Instructions for Experiment on the Two-Axis Diffractometer of BL04B2 for Disordered Materials.

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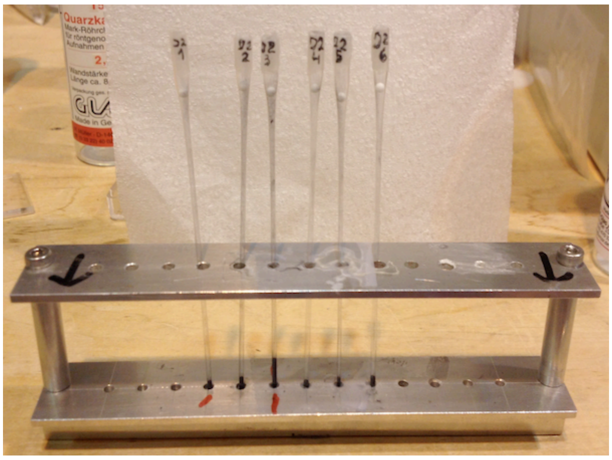
Sample preparation

1. Put the empty silica capillary tube (ϕ = 1 mm - 2mm) into the sample holder.
2. Use the folded powder paper to transfer the powder samples into the silica capillary carefully.
3. Use a plastic tube to secure the capillary, then shake up and down to minimize hollow space between powder grains.
4. Put a small white ball into the capillary and seal the top of the silica capillary tubes.

Note:

- Mark each capillary with the sample's name/reference number.
- Add an empty capillary next to the capillary with samples. Detected signal will be used for background substraction.
- Diameter of capillary and respective sample holder in use are dependent on the powder sample grain size.

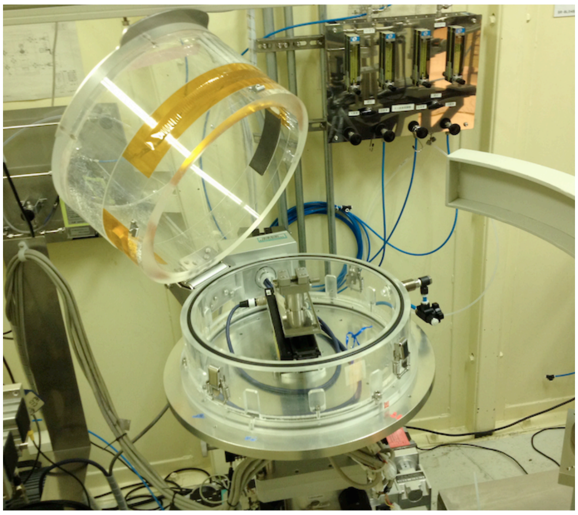


Powder sample inside capillary inserted into sample holder

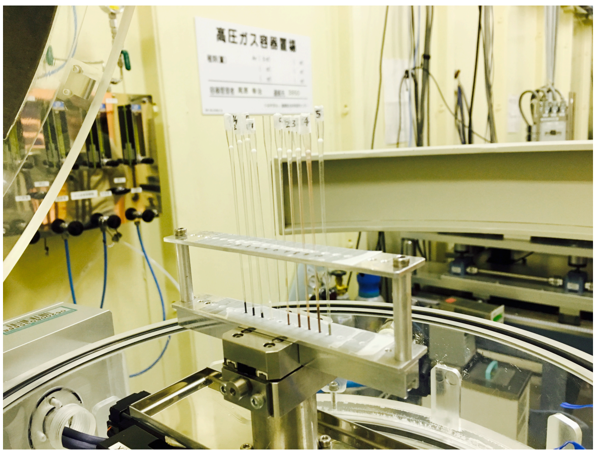
Sample loading

1. Powder samples in capillary inserted into sample holder used for measurements. If other than powder samples are measured, use respective sample holder. Ask beam-line scientist for help if needed.

2. Fix sample holder onto the positioning stage inside vacuum chamber on the diffractometer. Optimal configuration for sample mounting is $2\theta = 45^{o}$ for detector and sample X=0 for sample holder; diffractometer operation details are presented below.
   Be careful while in hutch: don't touch any detectors or respective cables!



Sample positioning stage inside vacuum chamber



Close-up of sample holder with capillaries fixed on the sample positioning stage

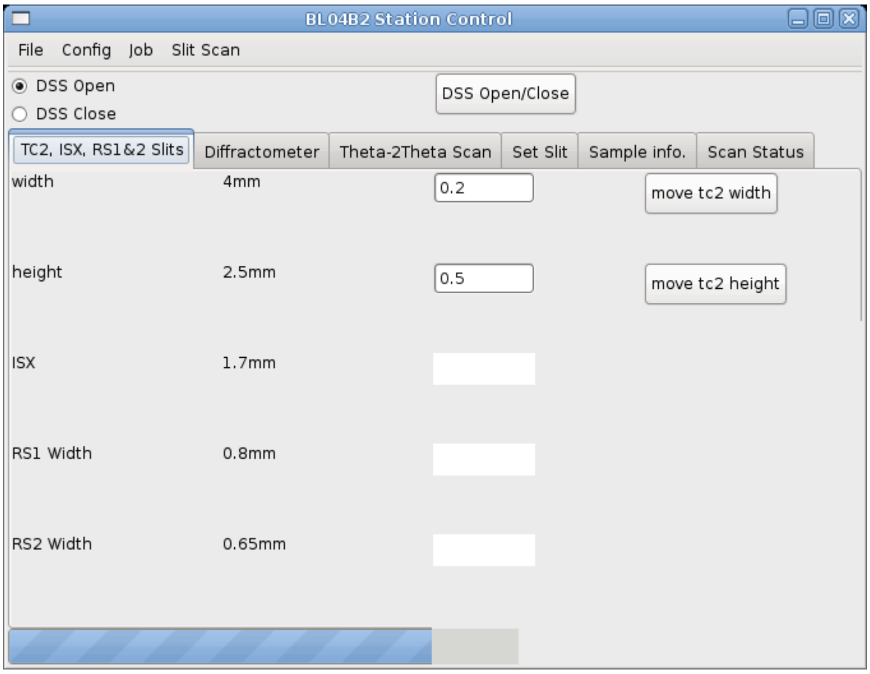
3. Close and fix the top cover of the vacuum chamber, switch-on vacuum pump and open vacuum valve to pump down chamber.

4. Exit the experimental hutch, push the **EXIT** bottom, close the hutch door by pushing **red button** (follow audible alarm signal), wait until the door interlock system is activated and turn key to **enable** position. On the touch screen push "**local only**" button, glowing red "DSS optics beam on".

Data Collection: X-ray exposure

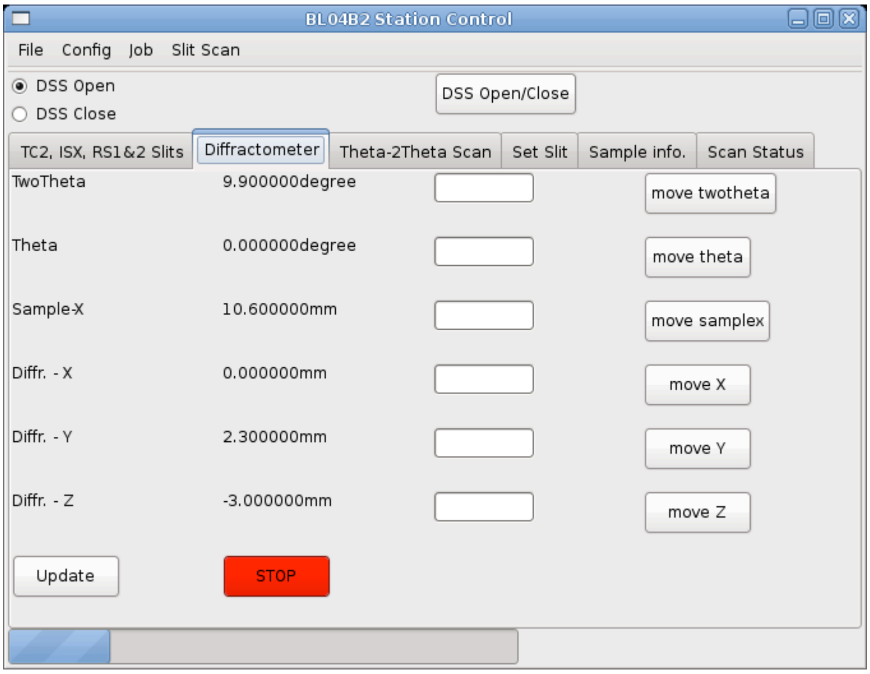
Collect data using station control program <bl04b22.py>. The icon can be found on the desktop of experimental PC.

1. Launch the control software <bl04B22.py>



In the **TC2, ISX, RSI1&2 Slits** tab check the current values of the TC2 and ISX slits. To protect AMPTEK detectors from strong X-ray flux, set the slit sizes to the values of: TC2width = 0.1, TC2height = 0.1, and ISX = 0.05.

Select the **Diffractometer** tab and move TwoTheta to the ~10^{o}



2. **DSS Open/Close**: beam on/off in the experimental hutch

   Press the **DSS Open/Close** button to be sure the Exp DSS open.

   The indicator DSS open on the control panel and glowing red BEAM ON button of the panel next to the experimental hutch door indicate the experimental hutch beam on state.

3. In **Diffractometer** tab find preliminary positions for sample: sample X and diff. Z, (on screen the beam position marked in red)

4. Perform QUICK scan in the Theta-2Theta Scan tab.

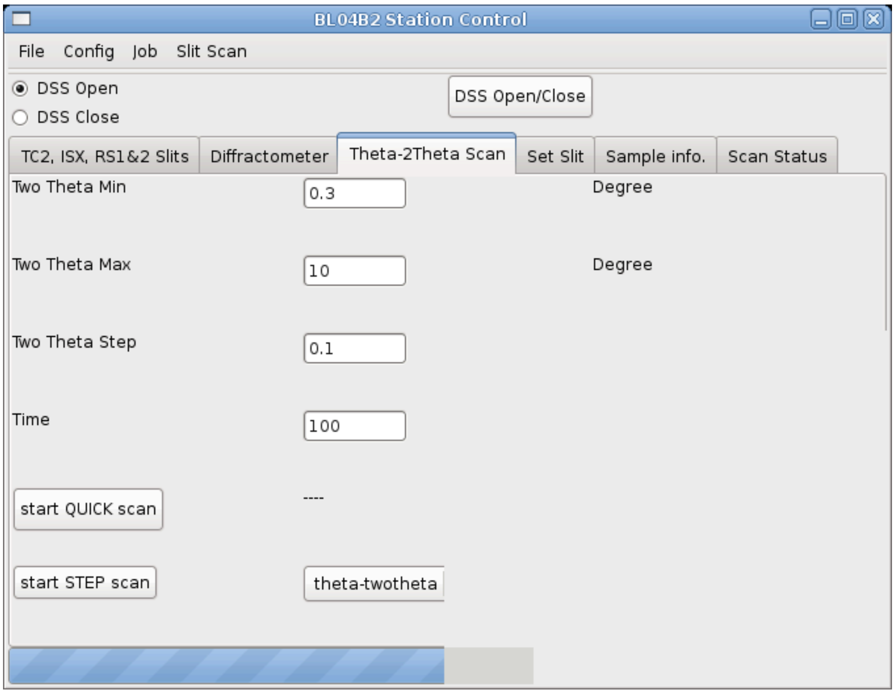
   A default range 2θ=0.3-10^{o}, step 0.1, dwell 100 msec click the start QUICK scan button and find 2theta position of the highest peak.

   Use for crystalline materials: tc2w=0.4, tc2h=0.2, isx=0.2

   for amorphous & nano ones: tc2w=2, tc2h=2.5, isx=1.0

   For empty quartz-capillary use 2θ = 2.8^{o} @ 61.4 keV

   2θ = 4.55^{o} @ 37.8 keV



For accurate position (in case of crystalline samples) perform **start STEP scan** around the peak position (step 0.01, time 1000 msec).

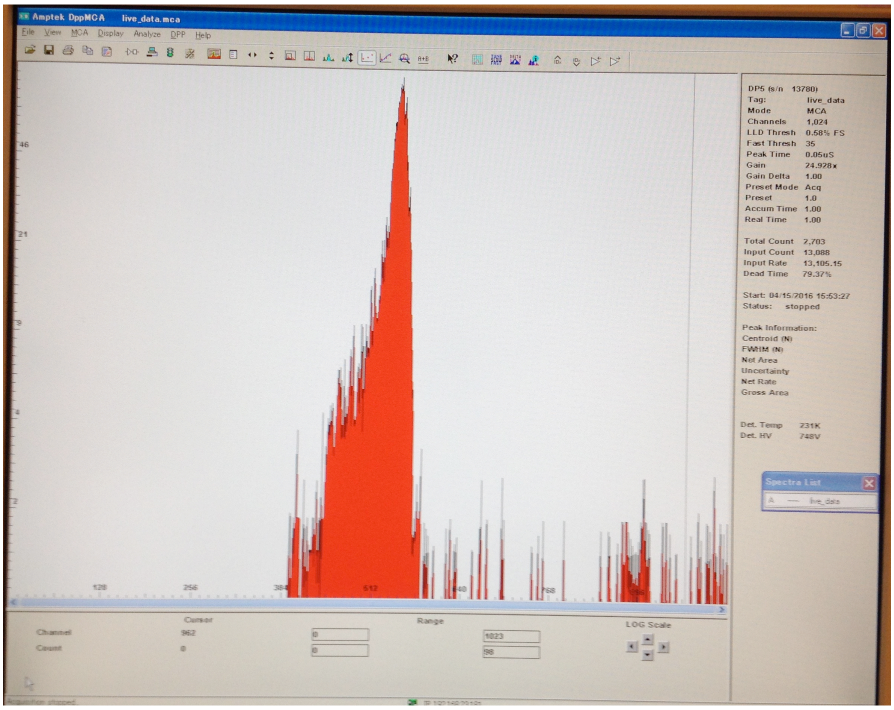
5. Confirm and optimize **sample X** position with scan around estimated position in range about twice the diameter of capillary (Δ=2ϕ), Step scan= 0.1, dwell= 1 sec, channel= 2

6. Move 2θ to 45^{o} and fine-tune the **sample Y** position by scanning from -4 to 4, step=0.2, dwell=1 sec, channel= 2

7. Return to the 2θ peak position of the first (highest XRD peak, e.g ~0.3-0.6^{o}) and optimize the peak intensity by altering slits size. For this tap to the **TC2, ISX, RS1&2 Slits** tab and change gradually slits size to get total count ~10, 000 (optimal) in 1 sec.
   Check the total count on the Amptek DppMCA screen monitor.
   Use A and F3 on the keyboard to display X-ray scattering in 1 sec.
   During optimization of the TC2 width, TC2 high, ICX width sizes hold relation: TC2 width : ICX width = 2 : 1
   The upper value (fully open) for tc2h is 2.5 mm, and for tc2w is 4.0 mm

(As an illustration: for crystalline   tc2w=0.4, tc2h=0.2, isx=0.2

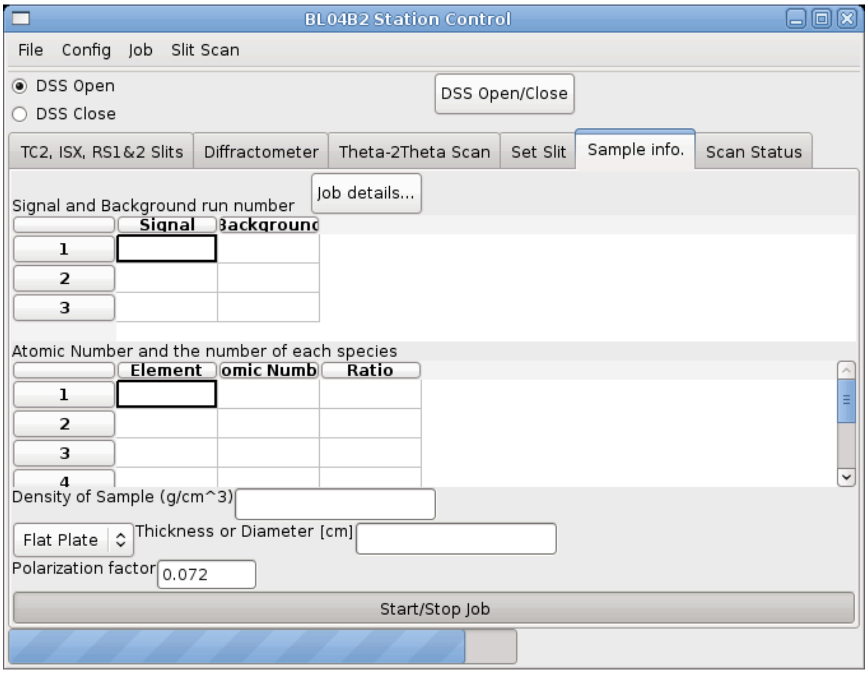
for amorphous   tc2w=2,   tc2h=2.5, isx=1)



AMPTEK detector CPS (abt. 10K)

8. If needed optimize sample measurement conditions:
   scan Z, scan sample X, scan Y

9. Place the experimental details into **Sample info**
   Tap the **Sample info** tab, press the **job details** button to <u>double-check</u> the set values for the job file in the stand-up window

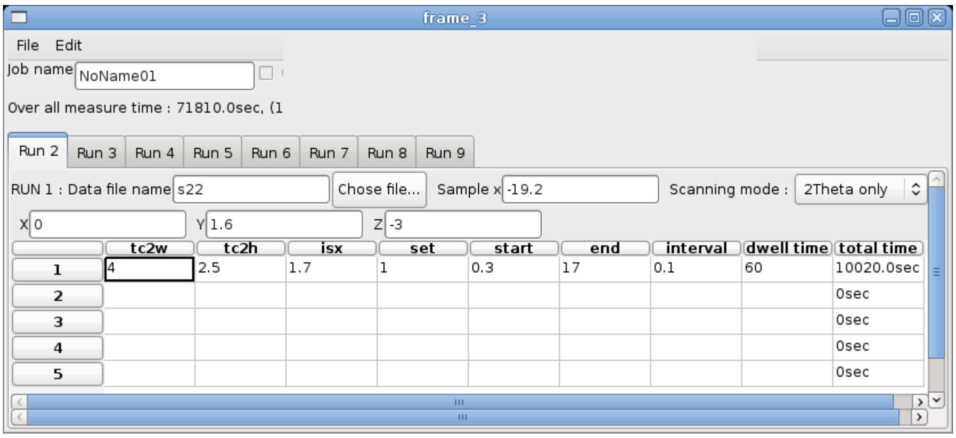


Data file name –– Sample X ––, Sample w 0 (fixed),

X 0 (fixed), Y ––, Z ––, Scanning mode 2Θ only or Θ/2Θ

tc2w –– , tc2h ––, isx ––, set 1 (fixed), start – (usually 0.3) ,

end –– (usually 17.001), interval –-, dwell time ––



Here **tc2w** is TC2 slit width, **tc2h** is TC2 slit height, and **isx** is incident slit width.

Usually, an interval (scan step) of ~0.1 for local and intermediate order amorphous or nano-materials and an interval of ~0.01 - 0.05 for long order crystalline samples are applicable.

In the current setting of horizontal two-axis diffractometer the tree detectors are away by 16^o, the angular range of the first detector and the second one is set to a value that partially overlapped. At value 2Θ end of 17^o the first and second detectors overlap $16\text{-}17^O$, while the second and third ones overlap at $32\text{-}33^o$.

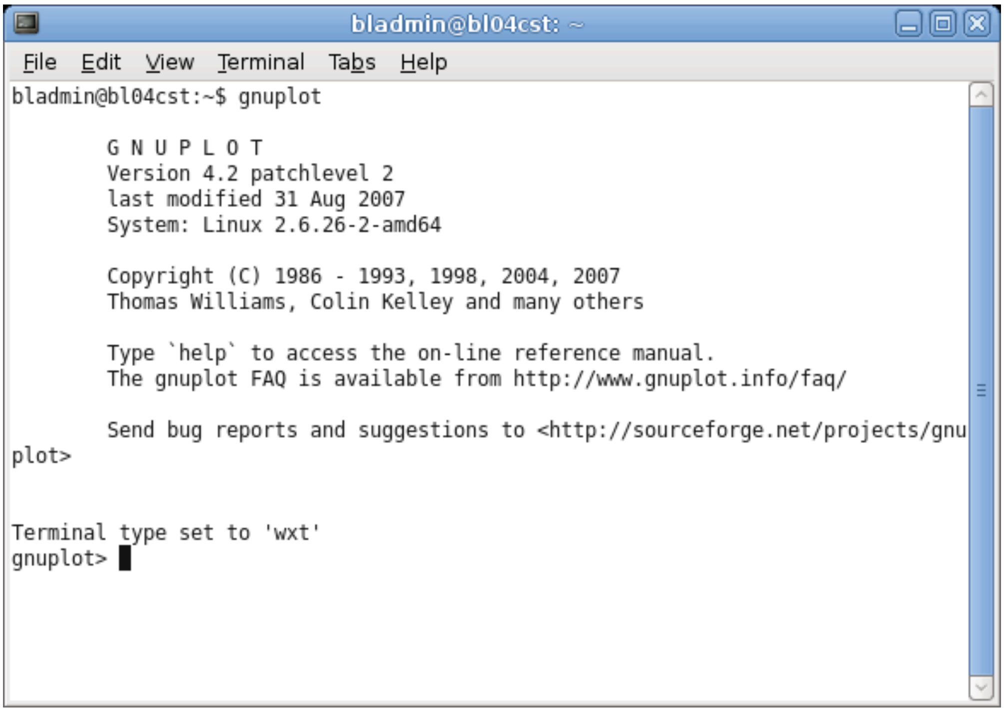
Before the job start check the values, modify not appropriate one and after revision be sure to run the File → Save → Replace guideline, notably the latest Save set secured in the job file.

10. After the set values have been confirmed press the **Start/Stop job** button and the measurements are started. The movement of the indicator under the button points to job in process, at scans completion it stopped. The job status (current 2Θ, X-sample, Y, Z, and slits positions) can be monitored by pressing the **Scan Status** tab.

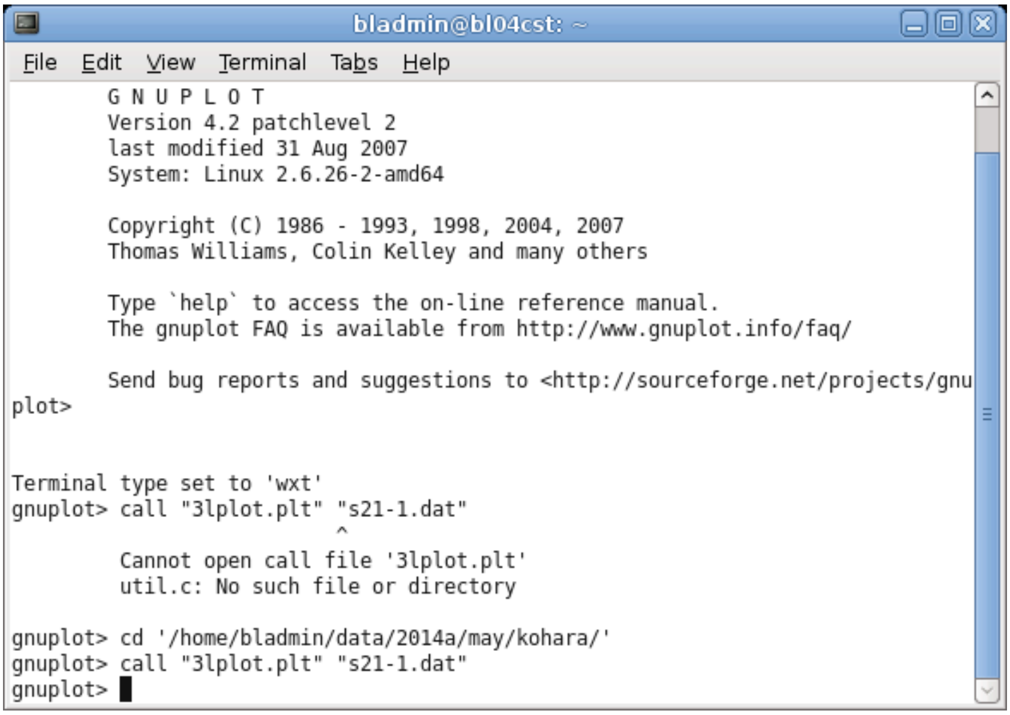
11. At scan-job completion, open the experimental hutch: DSS beam off, turn key in disable position, door open (green button). Open the leak valve of the vacuum camera and then open its top and take-out the sample changer.

Readout and plotting data

During the measurements the diffraction profile appear on the plot window. Plot and comparison of the acquired data (saved files) can be performed using the <gnuplot> program. Typing gnuplot on Linux terminal launch the plot software.



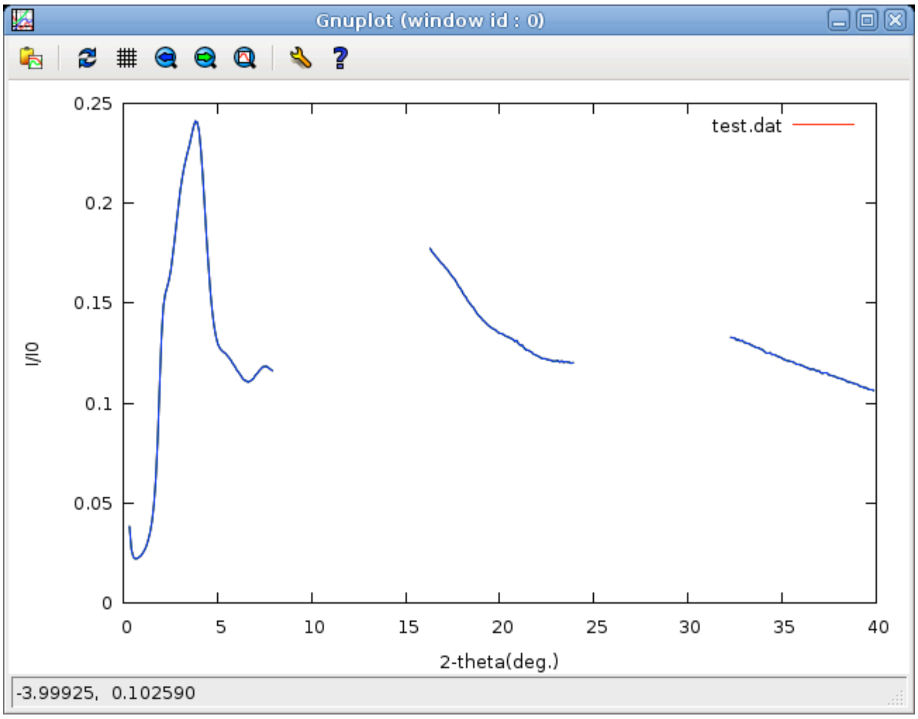
By using Linux commands <ls> (list all directories and files inside the current directory) and <cd> (change to any directory) move to the directory with acquired and saved files.



By using the <call> command load the p.plt followed by the input data file names that you want to plot.

gnuplot> call "p.plt" "name1.dat" "name2.dat" "name3.dat"

For comparison up to three files can be entered and superpose.
Since the data of three detectors per file are stored, it is plotted in three angular regions as shown bellow:



The plot of each file has its unique color, but be aware that the color of file and its legend line color may not be the same.

Copying & processing data: Files saved in "name.dat" format and can be copied for further processing.

Useful info about the beam-line

Things you need to know:

- 37.8 keV or 0.328 Å (Si 111)   0.10 - 22 (Q range, $Q=4\pi\sin\theta/\lambda$)
- 61.4 keV or 0.202 Å (Si 220)   0.16 - 36 (Q range)
- 113.3 kEV or 0.109 Å (Si 333)   0.30 - 40 (Q range)
- Photon flux @ 37.8 keV

  Flat: 2.2×10^{10} (photons/sec/1x 1mm^2 at 100mA)

  Bent: 7.1×10^{11}

  @ 61.4 keV

  Flat: 3.4×10^{9}

  Bent 9.2×10^{10}

At incident beam size: 0.2 (H) x 4 (W) mm^2:

Beam size   @ 37.8 keV: 0.220 mm

@ 61.4 keV: 0.375 mm

Energy resolution $\Delta E/E = 10^{-3}$