13BMD

Diamond Anvil Cell Operation
Notes on 13BMD - DAC operation
May 2008

This document is aimed to be an introduction to first time users and to be of help for troubles you might encounter while operating. Beamline scientist will be always available for further help. Your comments and bugs reports would help us improve and keep the manual updated.

In order support the beamline experiments, at GSECARS is available a sample preparation lab which includes microscopes, an EDM drilling machine and a mechanical drilling machine. There is also a Laser Lab for laser drilling, Raman spectroscopy and measurement of the ruby fluorescence. Moreover a Gas Loading System (GLS) is available at GSECARS to load He, Ne and Ar. We are also capable to load Kr and Xe, however we don’t have these gases at the moment, so please contact us if you need to load such gases. Ar can be also cryoloaded.

GSECARS does not provide DACs or diamonds.

Please remember to fill out the end of the experiment form!

Login: dac_user

Password: Daagbf
Acknowledgement requirements

When work is conducted at GSECARS Sector 13, APS, and is published in journals, books, conference proceedings, or other printed technical media, the following acknowledgment statement should be included in the manuscript:

http://gsecars.org/acknowledge.htm

[Portions of this work were]/[This work was] performed at GeoSoilEnviroCARS (Sector 13), Advanced Photon Source (APS), Argonne National Laboratory. GeoSoilEnviroCARS is supported by the National Science Foundation - Earth Sciences (EAR-0622171), Department of Energy - Geosciences (DE-FG02-94ER14466) and the State of Illinois. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

Please send copy of each published manuscript to GSECARS (Building 434A, Argonne National Laboratory, 9700 South Cass Ave., Argonne Il 60439) and to the APS User Office (Building 401, Room B1154, 9700 South Cass Ave., Argonne, IL 60439) as soon as possible.
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1. Main Station Control

The bending magnet station D is controlled by three computers located in the control area.

The beamline motors are controlled through an MEDM window, which also monitors the beam status. To open the standard window for DAC experiments, double click on the MEDM shortcut on the desktop, the main menu will open:

Click on the BMD experiments tab, and then select 13 BMD DAC in the pull down menu. The main DAC control window will pop up:
All motor positions in this window are expressed in mm. In order to change a value of the motor position click on arrows or + and - buttons (the motor position is changed according to the step size) or type a value on the light blue field (you have to have the mouse positioned on the box!) and press enter.

Motor position in mm
the pointer has to be on this field to change the value, press enter to make the change effective

Click here to change the motor position by the step size

MOTOR NUMBER
the full motor name is 13BMD:m89

Step size, change by entering the desired value

Three useful step sizes

GSECARS beamlines adopt the same right handed coordinate system used for the ring calculations, i.e. the Z direction is along the beam, positive direction downstream, Y is vertical, positive direction up, X is then automatically defined: in the horizontal direction perpendicular to the beam.

In the setup we make sure that the center of rotation of omega is aligned along the beam, position (0,0,0). The microscope, looking along the beam, is positioned so that the focal plane contains the (0, 0, 0) position.

Go to Appendix 1 for a detailed description of the MEDM 13 BMD DAC window controls.
2. Scan & View

*Software for sample and pinhole centering based on absorption profiles*

1. Run new IDL window by double-clicking on the IDL shortcut on the desktop.
2. The software can be started by entering the string `scanw` in a command line of IDL window.

Please note that an IDL window should run only one application!

1) Enter the correct motor name in the text widget, e.g.:

- BMD: 13BMD:m90, vertical sample stage motor
- 13BMD:m89, horizontal motor, perpendicular to the beam direction for $\omega=-90^\circ$
- 13BMD:m49, horizontal pinhole motor
- 13BMD:m50, vertical pinhole motor

and hit ‘Enter’.
When the motor name is entered, the actual motor position is automatically read. Define the scan range, the step size and the exposure time per step (typically 0.3 second).

2) In the text widget 2, type the address of the diode counter, the scaler name:
   - for BMD type 13BMD:scaler1

Click the button Scan, the photodiode will be inserted automatically and the shutter will open automatically too. If you are using MAR345, make sure its shield covers the detector.

In the screenshot below is a typical absorption scan of the gasket hole. You can find the center by clicking on the button Center and then by clicking on left and right positions defining the limits of the object you want to center. By clicking on the button move, the motor will be driven in the center position.

Scan&View Troubleshooting:

*The program is not responding.*

In most cases the program is just waiting for a position to be defined (after you intentionally or accidentally hit the buttons Position and Center), just click randomly in the plot. If it doesn’t unfreeze the program, please restart it.

Make sure you didn’t use the IDL window supporting scanw application for other purposes.
3. X-ray detector

3.1 Start MAR3450 IP

The detector is controlled by its own LINUX computer. The detector will be set up for you by the beamline staff, however, if for some reason you need to restart the program, follow these instructions:

The program that controls the detector is called mar345dtb. A shortcut to start it is on the taskbar.

In the main MEDM window, on the MAR-IP sub window, click on More:

- set the mode to 3450: 3450x0.10
  If necessary change the host and folder paths, e.g. setting the strings
  Folder: UC
will write diffraction images on the server folder with the following address:

T:\dac_user\2008\BMD_2008-2\UC
• Click on the restart button, the “Uninitialized!” warning will appear on the bottom, while the connection with the Linux computer is reestablished (may take as long as 2 minutes!). When the “Done” message appears, close the window.
• You are now ready to use the “Collect” button on the screen to take diffraction patterns.

3.2 Perkin-Elmer detector setup

3.3 Detector calibration

The calibration of the X-ray detector is made using CeO₂ as standard material. Then using the built-in features in Fit2D (Appendix 3), the tilt and sample-detector distance can be determined.

• Put the holder with CeO₂ standard. Positions are stored by the program “remember me” (shortcut on the desktop).
• Move microscope "IN", turn light “ON”. You should see the CeO₂ standard. Adjust focus and sample position if necessary.
• Move microscope "OUT".
• Scan the clean-up slits. (see section C 3.)
• Erase the IP detector before taking the standard exposure (button on MEDM window or on linux machine). Typically 120 seconds is enough for CeO₂.
• Use Fit2D for image loading and detector calibration (see Appendix3).

The X-ray energy is calibrated independently, by scanning the absorption edge of a Ba compound, which is usually done by beam-line staff. Contact beamline staff for more information (accuracy, etc.) about the X-ray energy.

4. Sample centering

4.1. Mounting the dac
Before mounting the DAC with the holder on the sample stage, drive the omega motor to the -90° position. Mount the DAC holder on the sample stage, pay attention to avoid collisions with the pinhole or any other hardware. If the pinhole position is too close to allow the DAC rotation, move the pinhole out.

Click on microscope [In] button on the 13 BMD DAC window, click on “Unzoom”. Search and focus the gasket hole position (you better use 0.100 mm step movements),
center the gasket hole on video, then zoom in and center it again (now with 10μm step size or less). Bring the sample into focus.

4.2. Correction for diamond refraction

The focus position of the sample determined with the microscope is actually quite far from the real focus position because of the diamond refraction effect. In order to find the true sample position, open the IDL 6.4 and type “bmd_stage” in the command line. The following window will pop up:

Choose whether the direction towards microscope is positive or negative (default one). In the window ‘Diamond thickness’ type the approximate thickness of your diamonds.

Second, click ‘Sample’ button. The program reads the sample position and activates ‘Diamond Table’ button. Click on the ‘Diamond Table’ button, the stage will move and shall focus on the table of your diamond. If the image is not in focus, adjust the focus position by moving the motor m91 (focus). Once the image is in focus, click ‘Update’ button. This will re-calculate the correct diamond thickness and also will compute appropriate diamond correction.

Finally, click on the ‘Diamond Correction’ button. The sample is moved automatically into the correct position and the microscope position is adjusted accordingly, so that you still can observe your sample. Take the microscope...
4.3. Centering based on absorption

You can now proceed to a finer sample positioning by collecting absorption profiles of the sample with the program Scan&View. For a detailed description of this program refer to the relative instruction. Insert the diode using the button on the MEDM 13 BMD DAC window. Start scanning the pinhole in horizontal direction (motor 13BMD:m49). When the scan is finished, center the absorption profile and move to this position. Repeat the operation for the vertical pinhole motor entering the motor name 13BMD:m50. The pinhole position drift should not exceed the value ±50μm, contact GSECARS staff if you are getting a larger drift of the pinhole position.

Now scan the gasket hole by typing the motor names 13BMD:m89 or 13BMD:m90. Change the scan range according to the gasket hole size, typically ±0.1 to ±0.2mm. If your experiment requires some extent of omega rotation or you need very precise structure determination, it is better to refine the center of the sample position along the beam. This is performed collecting horizontal absorption scans at different omega angles. If the sample is perfectly centered along the beam, the horizontal position of the gasket hole do not changes; if you observe a change in such position (Δy) you can use the value to calculate the displacement along the beam direction Δx. In the same program, which was used for diamond correction, record three horizontal positions of the gasket center, one is in position perpendicular to the x-ray beam and two others at some angle from the central position. You can have control as to by how much the sample shall be rotated. Type in the appropriate ‘-omega’ or ‘+omega’ angles in the appropriate windows. The program shall guide you through the process. Once all three positions are recorded, click on the ‘Focus Correction’ button. The stage is moved according to the calculated correction and the microscope position is adjusted accordingly.

If you want to use the microscope to pick the probing spot, close the table shutter and insert the microscope. You can determine the exact “position of the beam on the microscope” (marked with a cross on tape on the video) by loading the horizontal and vertical scans and driving the motors to the sides of the gasket hole. After picking the spot you want to investigate, take the microscope out.

5. The beam

5.1. Beam status control

The status of the ring can be checked on the “TV” located in the IDC control area and in the user area as well as on the web at http://www.aps.anl.gov/aod/blops/status/smallHistory.html

The bending magnet station works with monochromatic radiation, for diffraction experiments the energy range used is 20 to 50 keV. The beam for DAC operation is usually focused to ~5μm in the horizontal direction and 10μ in the vertical. The status of the beamline is shown in the main MEDM window. On top left corner is the storage ring current, and the X-ray energy and wavelength at the station. On top right corner is the status of downstream stations. Stations A, B and D must be searched (green status) in order to have beam in BMD. On bottom right corner is a plot of the beam intensity at the ion chamber in the station versus time. The red horizontal line shows the target intensity.
Refer to the troubleshooting section if you don’t have beam at the station or if the feedback system cannot maintain steady the target intensity.

### 5.2. Change the pinhole tip

The size of the pinhole can be changed according to your experimental requirements. Ask your beamline scientist for the pinhole box with a set of pinholes of several sizes. There is a mark on them, align such mark with the mark on the cleanup pipe. Scan and center the pinhole position with the beam, you may have to use a large enough scan range (± 0.100mm) to find the pinhole.

- If you cannot see the pinhole profile (this might happen after an accidental bump of the pinhole), replace the tip of the pinhole with the larger (first one) of the pinhole box, which has a very large hole. Scan and center the pinhole positions with increased scan width (up to 2mm), replace the working tip and repeat the scan.

### 5.3. Change the beam energy

to be updated

### 6. Angle dispersive powder diffraction

#### 6.1. Single exposure

The collection of single exposures is controlled directly on the main MEDM window; simply set the exposure time, the File path (e.g. /CARS5/Data/dac_user/UC/), the Base name, move out the IP shield and hit the Collect button. Don’t use long Base name.

- If the Data Collection looks frozen, click on More button, Click on the Restart button and wait ~2minutes to reestablish the connection with the detector.

You can use the software **GSE_Shell** for quick powder data evaluation and processing. You can find a link to the software as well as a manual on the desktop.

#### 6.2. 2-D scan

The collection of a 2 dimensional scan is rarely performed at the bending magnet because of the relatively long exposure time usually required.

a) Setting the vertical scan

- In the main DAC window, click “FULL CONTROL” (lower-left corner), go to SCAN > scan1
• Type in the PV name of the vertical motor under “positioners” as shown in the example.

• Input the desired values for the scan start, center, end, and the step size
• Select the LINEAR scan mode, use the RELATIVE scale, select PRIOR POS in the after scan tab.

• Under “Detector triggers” subwindow, enter the scaler desired for the scan. The example (13MARIP:Collect) is the scaler name of the MAR345 IP detector.

b) Setting the horizontal scan

• In the main DAC window, click “FULL CONTROL” (lower-left corner), go to SCAN > scan2
• Fill in the PV name of the horizontal motor under positioners as shown in the example.

• Under Detector triggers specify this time you will specify the “address” of the Scan1.

• Select the LINEAR scan mode, use the RELATIVE scale, select PRIOR POS in the after scan tab.

• Input the desired values for the scan start, center, end, and step size

• Click the button “scan” to start the measurement

7. Monochromatic single crystal diffraction

The program for collecting diffraction patterns with omega rotation is MAR345_DC, open the link to the MAR345_DC.sav file on the desktop.
1. The Linux directory has to be specified in the first box. Such directory is found in the EPICS MAR-IP window, clicking on the “More” button, the rows host+folder make the directory path
   !! end the directory path with /
   !! what looks like spaces in EPICS window are actually underscore (_)
2. Specify the windows directory where to store the images
   !! end with /
3. Click on the EPICS Config
   - Clik the “connect” button
4. Click on TCP/IP Config, set the proper **port number** (usually 5002) and **164.54.160.128** as IP number

**A** To collect a single exposure with omega oscillation, set the proper starting value for Omega (start), set the desired exposure time and the range (upper box), then click on Single image button. With the parameters set as in the screenshot below, an image will be collected starting from -110 degrees and ending at -70 degrees in 180 s.

![MAR345 Image](image)

**Attention:** Remember, that the maximum speed of the rotation motor is 5 degrees per second! NEVER EVER exceed this speed, because it may cause the damage to the stage and hardware inside!

**B** To collect a sequence of images with step omega scans, set the omega starting value, the exposure time, the range for a single exposure in the upper Range box, and the total range in the below one, set the number of steps and finally click on **Start scan**
If the program crashes, you first have to change the port number in the Linux computer, then you can restart MAR345 DC.

In the LINUX machine
Go in “tables” subdirectory by typing
`cd tables`
the command
`emacs config.270`
edit the file containing the connection information. In the next to last row is the port number, which has to be specified in configuration of MAR345_DC

If the connection is lost, this number has to be changed (any number), save changes and start mar345dtb

7. Brillouin Spectroscopy

Brillouin system will be set up to your liking by the beamline scientist. After the plate spacing is set and the stabilization is in place, you are ready to go.

1) Double-click the Ghost shortcut on the desktop. Spectra collecting software for the Brillouin system will be run.
2) Double-click on the Brillouin Win1024 icon on the desktop. Software for the analysis of the spectra will be run by this program.

In the Ghost, set the number of sweeps to some large number (usually 999999). If error message appears, click Cancel.
In order to set up the Brillouin Win1024 software, go to ‘File’, then choose ‘Calibration’ from the sub-menu. ‘Calibration Info’ window will appear (see all window screenshots below). Choose ‘This is current run-time calibration’, enter the plate spacing and scanning amplitude (usually set to 580). ‘Calibration’ window will open, along with ‘Calibration Ghost Calculator’ window. Go to ‘File’, pick up your calibration file and then click ‘Accept/Add to average’. If you collected more than one calibration file at the same conditions, repeat for other calibration files.
When done, click on ‘Accept’ button, then ‘Close’ in ‘Calibration Ghost Calculator’ window. Click ‘Exit’ in the ‘Calibration’ window. ‘Win1024’ program is ready to analyze your data!

Please open calibration spectrum
Changing the plate spacing

When you need to change the plate spacing you have to switch off the stabilization of the system.

**First**, switch off “Stabilisation” and make “Shutter off” on the front panel of the Interferometer Control:

![Interferometer Control Panel](image1)

**Second (very important!!!),**

Turn off all the lights illuminating your sample (the microscope light and the “Low Lamp”).

Open the “13BMD panel #9” on the top-level (CAREFUL! Do not hurl the door of the panel):

![13BMD Panel #9](image2)

And move the right toggle-switch to the “Align” position:
The picture on the oscilloscope will change to:

Upper panel: Rotate the knob from “OFF” to the Z-position:

Now you can change the “Z” (which is actually a plate spacing) using the toggle-switch “ADJUST”.

The progress of the changes should be closely observed on a round scale to the right of the toggle-switch.
* Be **CAREFULL (!!!)**: the arrow moves slowly in the beginning, but increases the speed when you keep pushing the “ADJUST” toggle-switch.

The best way is to push the toggle-switch to move the arrow on the round scale for about a quarter of the circle, then release the toggle-switch.

At the same time you have to observe the Interferometer: See the green spot near the “Z”-knob. Adjust the knob if the spot moves far from the central position and especially if it turns red.

When reached the desired plate spacing, close the lid of the round scale and rotate the knob from the “Z” to the “OFF”-position on the upper panel.

Continue the procedure of setting the stabilization from the ☼ sign in the appropriate instructions below.

**Brillouin System Stabilization**

The system is stabilized and everything is OK:

When the high peak in the center of the oscilloscope does not appear, the stabilization is lost.
**First,** switch off “Stabilisation” (1) and make “Shutter off” (2) on the front panel of the Interferometer Control (see all pictures above)

**Second (very important!!!),**

Turn off all the lights illuminating your sample (the microscope light and the “Low Lamp”).

Open the “13BMD panel #9” on the top-level (CAREFUL! Do not hurl the door of the panel) and move the right toggle-switch to the “Align” position.

The picture on the oscilloscope will change to:

![Oscilloscope Picture](image)

☀️ **Start from here on after changing the plate spacing**

The goal is to make both negative central peaks as long as possible and bring them together to the center. Use knobs on the front panel of the Interferometer Control:

![Interferometer Control Knobs](image)

The “Z”-knob moves both of the peaks (in the center of the oscilloscope) simultaneously; the “ΔZ”-knob moves only the **second** peak; both the “X1” and the “Y1”-knobs help to adjust the **first** peak; “X2”, “Y2” – the **second**.
1. Distinguish between the **first** and the **second** peak, using “ΔZ” knob.
2. Using “Z” knob, bring the **first** peak to the center of the oscilloscope and try to make it longer (slightly rotating “X1” and “Y1” knobs). The lower oscilloscope with running green cursor helps to make fine adjustments.
4. Move the **first** peak from the center position and bring there the **second** one. Using “X2” and “Y2” knobs make it as long as possible.
5. Bring the **first peak** to the center (use the “Z” knob). Then use the “ΔZ”-knob to bring the **second** one to the center. They should match together as good as possible.
6. Open the panel (#9) on the top-level (CAREFUL! Do not hurl the door of the panel) and switch the right toggle-switch to the “Tandem” position.

On the upper oscilloscope you should observe a small positive peak in the center. Using all the knobs (first try the “ΔZ”) try to make the peak about one division higher than the background in the “Align” mode.

When its done, switch on the “Stabilisation” and move the right toggle-switch to the “Window” position.
Sample positioning and laser focusing.

When the Brillouin system is set up and the stabilization is on, Brillouin spectra can be collected.

1) Put the sample on the kinematic stage and bring it to the microscope focus as described in the ‘Sample Centering’ part before. If you do not need x-ray diffraction, you may skip diamond correction part.

2) Screw the red filter onto the microscope tube.

3) Move ‘Microscope Z’ position in the main DAC control window by about 640 μm in the negative direction. This will bring the image back to focus. Fine adjust if necessary.

4) In the main DAC control window click on the green ‘Brillouin’ button. The following window will appear:
5) Brillouin is operated by the buttons in the two purple blocks. Left part is for the laser beam operation and focusing, right part is the collection part. Bring both parts vertically down to about zero position, the same for the horizontal part.

6) Click on the ‘Laser Shutter’ button (the hutch shall be closed and Laser Enabled button pressed!).

7) By moving the left part vertically and horizontally (use 0.1 mm steps) bring the laser spot onto the desired portion of the sample. Adjust the focus to minimize beam size.

8) Bring the low lamp in, turn it on (CAREFUL!! MAKE SURE THAT NO SPECTRA IS BEING COLLECTED, OTHERWISE PERMANENT DAMAGE TO THE DETECTOR COULD RESULT!), then repeat the previous steps for the collecting part of the system. You may observe the image on the pinhole at the entrance to the interferometer by choosing one of the monitors and selecting ‘pin-hole’ as the desired option. Adjust the focus. If you see two bright spots, put the pin-hole in between them.

9) Go into the hutch and insert the mask in front of the collecting lens. Do not forget to disable the laser before going in!

10) Return outside, close the hutch, enable laser and then click on the ‘Laser Shutter’ button on the Brillouin control window.

11) Click on the ‘Start acquisition’ button in the ‘Ghost’ program and observe the signal. You shall see something like this:
The number of peaks and their position will, of course, vary. However, if nothing but the central peak and ‘ghosts’ are observed, try to pick up a different spot on the sample or check whether you are collecting the signal from your sample and not any stray reflection. Monitor the stabilization signal, sometimes it is lost and this explains why no signal is observed. If nothing helps, ask your beamline scientist for assistance.

**Brillouin system calibration**

Brillouin system calibration is done with quartz glass. The longitudinal and shear velocities for this glass shall be $V_p = 5950\text{ m/s}$ and $V_s = 3760\text{ m/s}$, respectively. Beamline scientist usually checks the system before the run starts, but you may repeat calibration if desired. The deviations of about 100 m/s (2-3%) are considered normal.
Ruby fluorescence and Raman measurements

In order to take the ruby fluorescence or Raman measurements, you need to do the following steps:

1) Run WinSpec program.
2) Go to the Brillouin main control window and click on the small button ‘Brill’ in between purple blocks controlling Brillouin system. The button shall turn black and read ‘Ruby’.
3) Direct the laser beam at the desired spot, using the stage motors, m89 and m90.
4) Go to the WinSpec program, choose ‘Spectrograph’ option then choose the appropriate grating and then pick up the position of the center of the grating used.
5) Go to ‘Acquisition’, set up the desired exposure time, file name, and then press button with little green circle with ACQ in it.
6) When done, switch back to the Brillouin system by clicking on the button between two purple blocks. It shall turn from ‘Ruby’ back into ‘Brill’.

ATTENTION: For the ruby fluorescence measurements, you may put one or two filters in to reduce the intensity of the laser, otherwise the signal will be too big.
A1. Troubleshooting

1. No x-ray beam

- Check if the storage ring is supplying light, make sure stations A, B and D are searched

- Is the main shutter open? If yes, check the window at the lower-right corner of the DAC window. If current reading is 0(zero), contact beam-line staff.

- If the current reading changes over time as shown below, this means that the “Setpoint” is a little too high for the feedback to keep the top number. Reset the “Setpoint” value (5% below the maximum).

- If you have a good reading from Current, but no x-ray beam from photodiode, then check the following:
  - Is the table-top shutter OPEN?
  - Is clean-up slit at the right (“IN”) position?
  - Is the photodiode IN?
  - If yes to all above, but still there is no beam, contact beamline staff.
A2. Detailed description of the 13 BMD DAC MEDM control window

The controls of the station motors is roughly arranged as the hardware in the table setup. From the right to the left, upstream to downstream:

A. beam and shutter status
B. clean-up slits control
C: the sample stage
D: microscope
E: photodiode
F: X-Ray detector
G: Beam energy
H: Control of IP plate exposures
A. beam and shutter status

-Ion Chamber: clicking on the blue buttons would let you change the either the scaling as swell or the unit. You don’t usually need to change these values during standard experiments.

- Table shutter: by clicking on the buttons “Open” and “Close”, you will open or close the table shutter, red color means that the shutter is closed, green means open.

- The graph is a plot of the beam intensity at the ion chamber before the table mirror, the bar at the bottom is the control for the feedback motor that keeps the intensity constant. The feedback is usually kept “On” and it supposed to keep the intensity of the beam close to the set point, which is represented by the horizontal red line on the graph. If, for some reason, the target intensity cannot be reached, you may need to lower it. To do that, turn the feedback off, change the set point (one or two units) then turn the feedback on again.

⚠️ It’s better to keep the shutter closed while you are not using the beam; also, it’s recommended to erase the detector after the shutter has been left open without the shield covering the detector.
B. clean-up slits control

The clean-up slit is aimed to cut the beam tails. You can mount different pinholes, with different aperture to change the beam size. The pinhole has to be perfectly aligned with the beam and it should be positioned as close as possible to the sample. The clean-up slit position is controlled through three motors. The X and Y motors are used to center the pinhole with respect to the beam (see Scan&View section) while the Z motor controls the distance to the sample.

Tips: The X and Y motors should be scanned anytime Z is changed before collecting data.

Avoid collisions of the clean-up slit, they might cause pretty large shifts, and long time to center back the pin-hole.
C: the sample stage

At the bottom of the sample holder there are 5 motors allowing the sample positioning and alignment.

- **m36**: is the first of the sandwich, at the bottom. It is used to align the center of rotation of omega with the beam. Don’t use this motor to align the sample.

- **Omega**: it performs rotation around the vertical axis (Y). You can run rotations by simply clicking on the buttons with the omega values. The value -90 corresponds to the DAC perpendicular to the beam.

**Tip**: make sure your rotation won’t cause any collision!

- **m89**: performs shifting in the direction perpendicular to the beam at ω=0
- **m90**: performs shifting in the vertical
- **focus**: performs shifting in the direction parallel to the beam at ω=0
- **chi**: the χ rotation (around the Z axis, parallel with the beam at ω=0) is performed with a special stage.
**D: microscope**

By clicking on the button ‘In’, the microscope is positioned with its optical axis parallel to the beam and the focus at the center of rotation of $\omega$ (sample position), when the microscope $Z=0$. Due to the diamond refraction, it is necessary to apply a correction to the microscope $Z$, which is calculated with Refraction calculator program. In order to quickly locate the sample, click the “unzoom” button.

**E: photodiode**

The photodiode detector is used to collect X-ray absorption profiles. It can be moved in the beam path and off by clicking the corresponding buttons. The sensitivity is changed by clicking on the blue left button.
F: X-ray Detector

Depending on the configuration set by the beamline staff, the detector can be moved either closer and further or side to side with respect to the sample by changing the value on the blue box. The detector shield is moved in and out by clicking on the orange button.
Tip: you need to collect a pattern for the detector calibration for each detector-sample position.

G: Monitor camera

You can change the views on the monitors over the desk by clicking on the blue buttons and choosing among the listed options.

H: Beam energy

The current energy at the storage ring and the wavelength of the beam at the station are shown in this field.
J: Control of IP detector exposures

You can define in these fields the location in the server where to write the collected images (see section 5), their names, the series number and the data collection time. Hit “collect” to start the acquisition, don’t forget to take out the shield first! Click on “More” to open windows for the 2D scan control, click on “Erase” to clean the detector, particularly after the detector has been left unshielded with the table shutter open.
A3. Getting calibration parameters with FIT2D from powder pattern

Fit2D is a software by A.P. Hammersley, on the web you can find a detailed manual. Here we report the standard steps we are performing to get the calibration parameters.
1) read pattern in FIT2D (3450x3450 arrays)
2) click "powder" diffraction
3) input the diffraction pattern
4) click "calibrant", choose CeO2
5) set a reasonable sample detector distance and the correct wavelength

![FIT2D Graphics Window](image)

**CALIBRANT PATTERN REFINEMENT**

**OF DISTANCE WAVELENGTH ETC.**

<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>VALUE</th>
<th>CHANGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample to detector</td>
<td>350.00</td>
<td></td>
</tr>
<tr>
<td>Wavelength (Angstroms)</td>
<td>0.3344</td>
<td></td>
</tr>
<tr>
<td>Size of horizontal pixels (pixels)</td>
<td>100.00</td>
<td></td>
</tr>
<tr>
<td>Size of vertical pixels (pixels)</td>
<td>100.00</td>
<td></td>
</tr>
<tr>
<td>Number of additional sections</td>
<td>360</td>
<td></td>
</tr>
<tr>
<td>Reject outliers</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Reject nominal</td>
<td>4.0000</td>
<td></td>
</tr>
<tr>
<td>Output full information</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Refine X/Y beam centre</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Refine sample to detector distance</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Refine X-ray wavelength</td>
<td>NO</td>
<td></td>
</tr>
<tr>
<td>Refine detector</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Non-orthogonality</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>Enthalpically number of rings</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Click on variable to change, or 'O.K.'
6) Check the "TWO CLICK" option (right bottom corner) input coordinates of inner ring.
7) click on CAKE button, select “circle coordinates”, manually enter the second ring coordinates.

Enter the default 0.0 coordinate for start azimuth, 360 for end azimuth, beam centre for inner limit, finally pick a spot on the image rim.

Click on Integrate button, the parameters of the next two screenshots should be OK.
At this point, in general, powder lines are not yet completely straight, but rather look like this:

![Image of powder lines]

Click on “Exchange” button, you should get the original pattern back. Click on Exit, then “TILT” button, the first two screenshot parameters should be fine (OK, OK). Select Circle coordinates, Input coordinates of the inner ring, click on a spot out the ring (not too close) that would define the width for ring search, select other well separated rings.

8) click CAKE>Integrate, OK, OK. The powder lined should now be straight, check better zooming in
9) Click again on Calibrant button, change to “NO” the status of “refine X/Y BEAM CENTRE” and of “REFINE ORTHOGONALITY”, you will only refine the sample to detector distance. >> OK >> OK

10) select again the coordinated for the inner ring, click on “Integrate”, the window will show the calibration parameters needed to analyze the data
    click OK>>OK>>
You now have the plot intensity vs 2-theta, you may want to compare this pattern with the d-spacings for the standard to further confirm the correctness of your calibration