Manual for GISAXS setup

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1 X-ray source

The most important part of GISAXS setup is X-ray source (IμS, Incoatec, Germany) (Fig. 1). The X-ray source generates CuK_α radiation (0.154 nm; 8.04 keV). It consists of X-ray tube and optics housing with focusing Montel optics. The recommended settings for Cu anode are **45 kV** and **650 μA**. The required power is ~30 W. The flux in vacuum is $3.3x10^8$ photons/s.

Caution: Exit window of optics housing is made from Be. Be window has to be handled with great care and special precautions must be taken when carrying out any activity which could result in the release of beryllium dust.

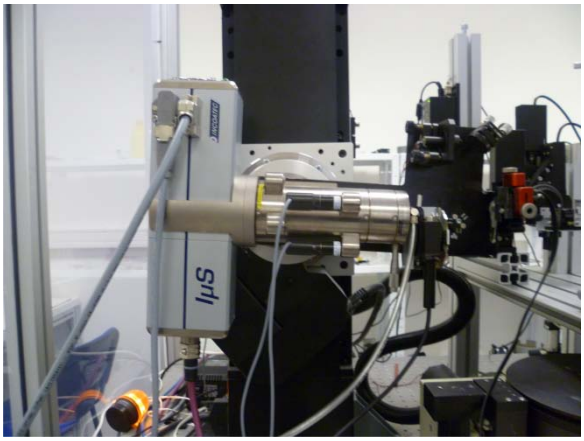


Figure 1. X-ray source (IμS, Incoatec, Germany).

1.1 Turn on/off X-ray source

In order to turn X-ray source on, turn **Main power key ON**. Press **LOC button** to operate X-ray source in the local mode. **LOC indicator** will light. Press **High Voltage button** to operate X-ray source in high voltage mode. **High voltage indicator** will light. Turn **kV** and **μA knobs** to set operating voltage and current in filament. All buttons and knobs necessary to operate high voltage generator can be found on the front panel of X-ray source (Fig. 2). In order to heat X-ray tube filament, please follow instructions in **Tube conditioning** and **Tube Warm-up sections**.

In order to turn X-ray source off, turn **Main power key OFF**. You can also use **EMERGENCY STOP button**.

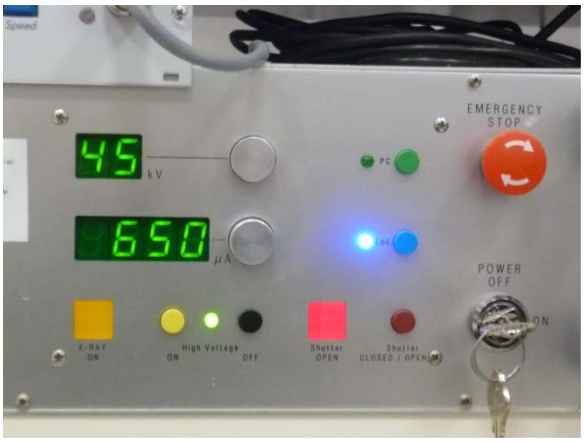


Figure 2. Front panel of the Voltage controller shows all buttons necessary to operate X-ray source.

1.2 Emergency stop

In case of emergency press the **EMERGENCY STOP button** of the generator to switch off the main power (Fig. 3).

To unlock **EMERGENCY STOP button** turn the knob clockwise as indicated by the arrows. Start operating system as desired.

Figure 3. EMERGENCY STOP button.

1.3 Manual/Remote control

The X-ray source can be controlled manually or remotely. The knobs and buttons for manual control can be found on the front panel of the high voltage generator (Fig. 4). The instructions for **manual control** can be seen below:

- Go to front panel of the high voltage generator.
- Press local control button/**LOC button**. **LOC indicator** will light.
- Press **High Voltage ON button. High Voltage indicator** will light.
- Use **high voltage knob** to increase voltage in kV.
- Use **current knob** to increase current in filament in μA.

Figure 4. Image shows High Voltage ON/OFF buttons and PC/LOC buttons.

The instructions for **remote co**ntrol can be seen below (Fig. 5):

- Go to rare panel (back side) of the high voltage generator.
- Find USB connector.
- Connect USB connector in rear panel of the high voltage generator with GISAXS computer. Use USB cable (short one, 20 cm) + amplified USB extension cable. *Caution*: It will not work without amplified USB extension cable (USB repeater).
- Go to front panel of the high voltage generator.
- Press remote control button/**PC button**. **PC indicator** will light.
- Press **High Voltage button. High Voltage indicator** will light.
- Go to computer and click on **IμS Control** program.

- Go to **Device → COM Port**.
- Set COM Port to COM 5 and Baud rate to 115200.
- Go to **Device** menu → **Connect.**

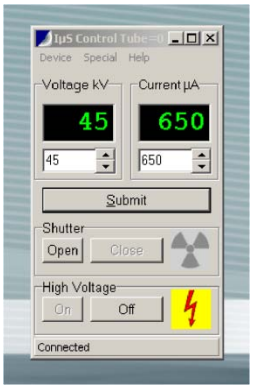


Figure 5. IμS Control program.

1.4 Tube conditioning

If the tube has not been used for **more than 2 months**, a manual tube conditioning as shown below has to be carried out. Tube conditioning will raise the high voltage from 0 kV to 50kV with a preheated filament at nearly no beam current, then return to 25 kV, power up to 550 μA and raise it to 50 kV again at maximum power. The generator will automatically shut down when finished. This task lasts approx. 2h.

Set generator to	**and wait for at least**
5 kV/ 0 μA	30 sec
10 kV/ 0 μA	30 sec
15 kV/ 0 μA	30 sec
20 kV/ 0 μA	30 sec
25 kV/ 0 μA	10 min
30 kV/ 0 μA	10 min
35 kV/ 0 μA	10 min
40 kV/ 0 μA	10 min
45 kV/ 0 μA	10 min
50 kV/ 0 μA	10 min
25 kV/ 550 μA	10 min
30 kV/ 680 μA	10 min
35 kV/ 800 μA	10 min
40 kV/ 700 μA	10 min
45 kV/ 650 μA	10 min
50 kV/ 600 μA	10 min
then back to the recommended settings	
45 kV/ 650 μA	

The tube conditioning can be done through **remote control** (Fig. 6):

- Open **IμS Control** program.
- Go to **Special → Tube conditioning**.
- Tube conditioning command and remaining time appears in the blue field at the bottom of the **IμS Control** window.

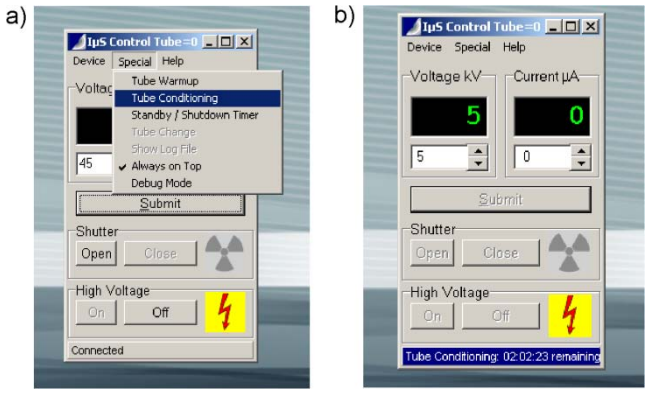


Figure 6. a) Tube conditioning option available in Special menu. b) The remaining time of tube conditioning appears in the blue field at the bottom of the IμS Control window.

1.5 Tube warm-up

If the tube was has not been used for more than 24h, the high voltage rate of increase has to be restricted to protect the tube. Any allowed current settings according to the table above can be used during warm-up:

Setpoint	**and wait for at least**
up to 25 kV	No restrictions
25 kV and above	max. 10 kV steps/30 sec

The warm-up can be done through remote control (Fig. 7). Go to **IμS Control** program. If high voltage generator operates in remote control or PC mode, the “Warm-up needed” will be displayed if an existing tube has not been used for more than 2 days. Then go to menu **Special** menu and click on the command **Tube Warm-up**. It will raise the high-voltage

slowly to 45 kV at 80 % of the maximum current for each voltage. The generator will not shut down immediately when finished. This task lasts approx. 8 minutes and is intended to drive the tube to operating power carefully. The progress (remaining time) will be displayed in the status bar similar to the tube conditioning program.

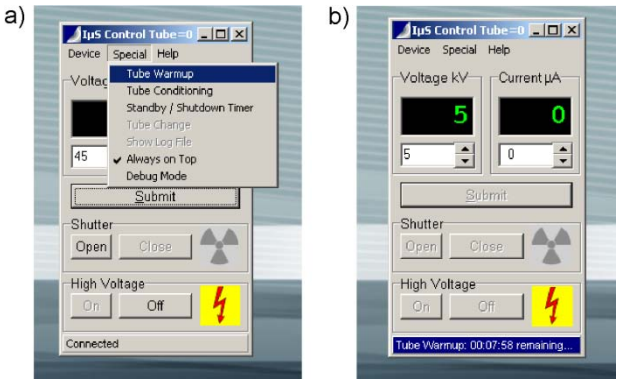


Figure 7. a) Tube warm-up option available in Special menu. b) The remaining time of tube warm-up appears in the blue field at the bottom of the IμS Control window.

1.6 Shutter

Between X-ray anode and multilayer optics, shutter is installed. If shutter is closed, X-ray beam is stopped and provides safety. Shutter can be controlled manually or remotely. In order to open shutter manually, the generator has to be in the local mode, high voltage must be present and has to be higher than 6 keV. To open shutter please press **Shutter CLOSED/OPEN button**. It causes that **Shutter OPEN indicator** will light as well as two red lamps on the top of the tube housing (Fig. 8).

Shutter can be also opened in **remote mode** (Fig. 5). Please follow instructions:

- Open **IμS Control** program.
- Press **Shutter Open**. It will open shutter.
- Press **Shutter Close**. It will close shutter.

Figure 8. Shutter of X-ray source. **Shutter OPEN indicator** lights what indicates opened shutter.

1.7 Vacuum alarm

If your IμS is equipped with an integrated vacuum control please note the small switch on the left-hand side of the front panel (Fig. 9a). Make sure the vacuum alarm switch is set to the ON position (the OFF is not visible) during normal operation.

Operating the system with an open shutter and poor vacuum will damage the optics!

- Never operate the system without sufficient vacuum.
- The controller records the time periods during which the shutter is open when the vacuum poor. Operating the system with poor vacuum for too long with breach warranty.

We recommend a vacuum pressure of approx. 5 mbar or better inside the optics housing (Figs. 9b-c). The vacuum switch has two setpoints:

- If the vacuum is poorer than 20 mbar the vacuum alarm switch will start flashing red. When set to the ON position there is an acoustic alarm. The acoustic can be switched off by turning the switch to the OFF position.
- If the vacuum is below 50 mbar and the shutter is open, the controller records the time period.

Figure 9. (a) Vacuum alarm button (in the OFF position). (b) Vacuum connector of optics housing. (c) Diaphragm pump for evacuating optics housing.

1.8 Motorized optics housing

Optics housing allows high precision alignment stage for the optics, providing up to four movements of the optics are actuated by eccentrics, so that no stop positions exist. The alignment of optics with respect to the primary X-ray beam is adjustable by two manual screws at the housing entry and two motorized screws at the housing exit (Fig. 10). The positions of the screws at the housing entry were pre-aligned by optics manufacturer and it is recommended to not move them unless necessary. If there is any problem with screws at the housing entry, please call Incoatec company (Hamburg, Germany).

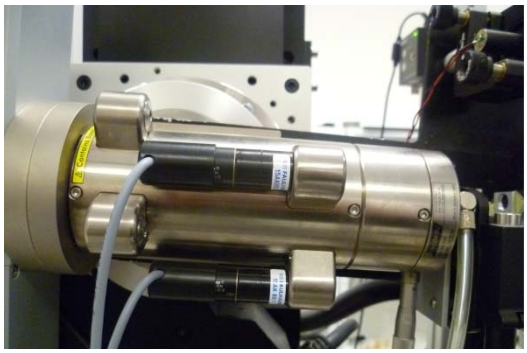


Figure10. Manual screws at the housing entry and motorized screws at the housing exit.

The positions of the screws at the housing exit can be controlled remotely by control unit and its joystick (Fig. 11). The Ethernet cables (grey) on the Fig. 10 are connected to the X1 and Y1 connectors at the back side of the control unit (Fig. 11a). The joystick (Fig. 11b) is connected to the front panel of the control unit. The joystick has two round buttons that are responsible for setting step and selection of motor connectors (e.g. X1, Y1).

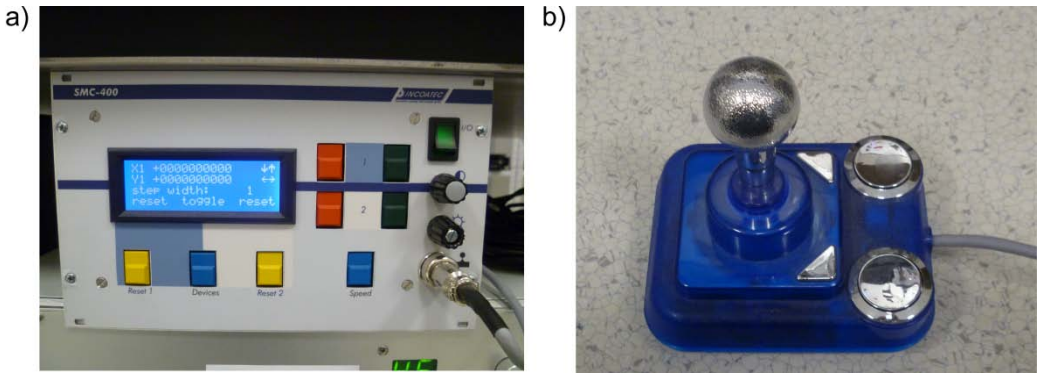


Figure11. Image shows a) control unit of optics housing and b) its joystick.

For finding the beam and coarse alignment of the optics, you can use X-ray sensitive camera (Fig. 12a). To convert analog signal of MarCam to digital signal, video grabber (Belkin, F5U228) is necessary (Fig. 12b).. It is connected to the BNC connector of MarCam and USB port of GISAXS computer.

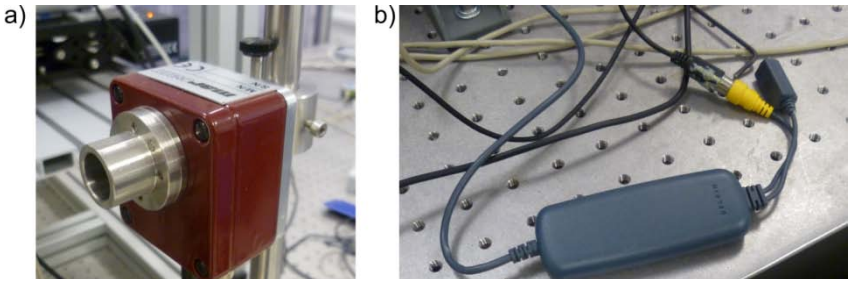


Figure12. Image shows a) X-ray sensitive camera MarCam and b) its video grabber.

To see X-ray beam, please follow instructions (Fig. 13):

- Go to **Labview → Mar_cam → Mar_cam.vi.**
- Run **Mar_cam.vi**. Please press run button  of LabView system. The program will open communication with Hi-Speed USB DVD camera (MarCam+video grabber).
- **Hi-Speed USB DVD Creator Properties window** opens two times. Press **OK button** in both cases.
- Image of X-ray beam appears in **MAR_CAMERA window**.
- Zoom in X-ray beam by blue and red cursors. It will appear in **FOCUS window**.
- The total intensity in **FOCUS window** appears in **Meter** and **Intensity box**.
- Press **Zero button** above Meter. It will reset the Meter needle position.
- The program will automatically integrate X-ray spot along vertical (**Hor_scan**) and horizontal axes (**Ver_scan**).
- The horizontal (**Hor_scan**) and vertical (**Ver_scan**) intensity profiles will be automatically fitted with Gaussian function.
- FWHM of X-ray beam in horizontal and vertical directions appear respectively in **FWHM_Hor** and **FWHM_Ver** boxes in μm.
- To **stop Mar_cam.vi.** Press **Stop Camera button.**

Caution 1: Measured intensity is in arbitrary units and is not physically relevant (counts and photons/second). Mar_cam.vi converts image from MarCam to the 2D array of unsigned 32 bit numbers. This is the reason that the total intensity appearing in the intensity box is too large number (~10^{10}).

Caution 2: To communicate with MarCam in LabView system, you need NI_IMAQ driver for USB Cameras. It allows you to configure any DirectShow imaging device and acquire images into LabView. It can be downloaded from NI Developer Zone.

Caution 3: Never use **Stop button**  of LabView system to stop Mar_cam.vi. It will cause crash of LabView system. Mar_cam.vi operates in Top-level application window that doesn't show **Stop button**. Path to Top-level application window in LabView system: File → VI Properties → Window Appearance → Top-level application window.

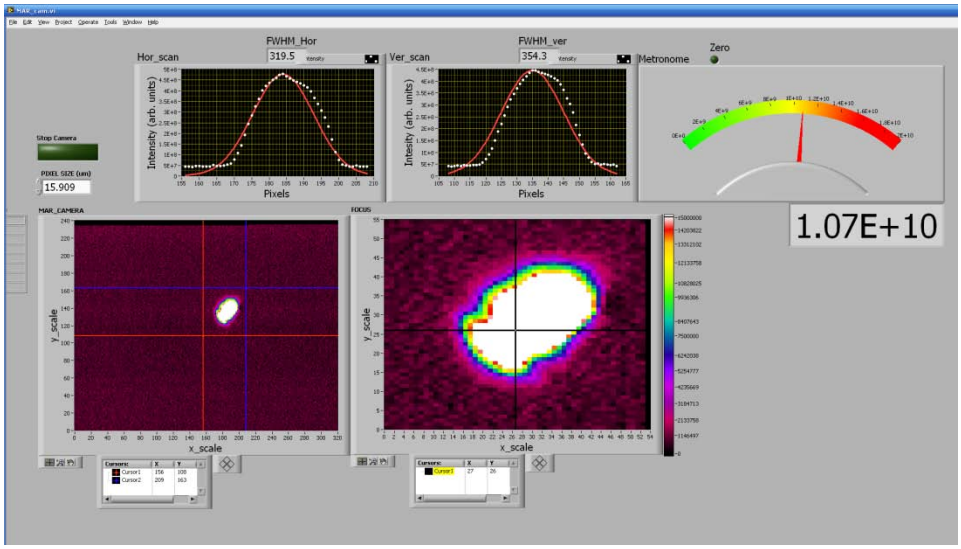


Figure13. Image shows Front panel of Mar_cam.vi.

1.9 X-ray beam profile

X-ray sensitive camera allows you to visualize beam profile. In order to understand beam profile, it is necessary to describe focusing Montel optics. Montel optics consists of two elliptically shaped multilayer mirrors arranged side-by-side (Fig. 14a). The primary X-ray beam can be then singly diffracted by single mirror or doubly diffracted by both mirrors (Fig. 14b). The part of direct beam is not diffracted and passes through multilayer optics. The singly diffracted beams appear as strips (Fig. 14b). Doubly diffracted beam converges to the focal spot (divergence 5 mrad). The focal length of Montel optics is 56 cm with focal spot size 270 μm (FWHM). SAXS/GISAXS measurements require removing of direct and singly

diffracted beams. For this purpose, you can use square pinhole built-in optics housing (see next section) and additional slits.

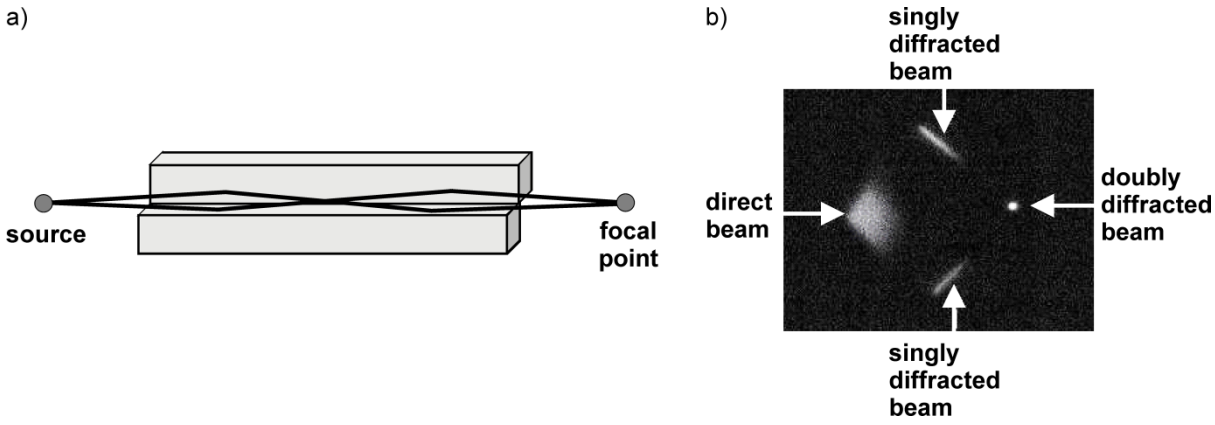


Figure 14. a) Scheme of Montel optics. b) Beam profile in the focal point.

1.10 Square pinhole built-in optics housing

Before exit Be window of optics housing, square pinhole is placed. It has shape of rotated square (like Renault symbol). It is useful to remove single diffracted strips as well as primary beam. The position of square pinhole in vertical direction is adjustable with micrometer screw (Fig. 15a). In lateral direction, the pinhole is positioned by hexagon socket screw. The access to the hexagon socket screw is on the back side of black plate (Fig. 15b).

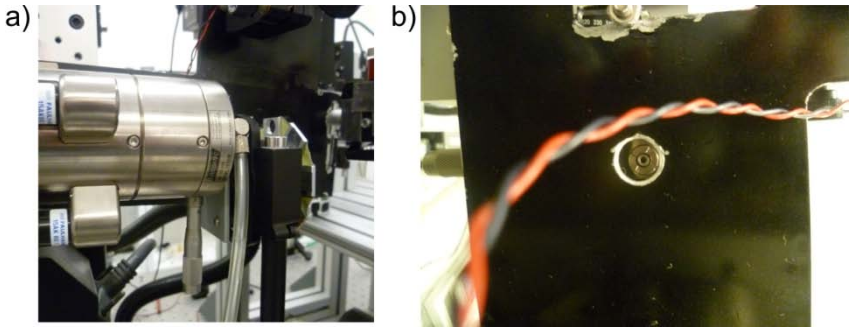


Figure 15. a) Micrometer screw of square pinhole that allows its adjustment in vertical direction. b) Hexagon socket screw of square pinhole that allows its adjustment in lateral direction (back side of the black plate).

2 X-ray detector – Pilatus 100K

Pilatus 100 K is a high-performance 2D X-ray detector (Fig. 16). It is based on hybrid pixel technology that combines pixilated silicon sensors with CMOS-based readout electronics. Technical specification of X-ray detector can be seen below:

Number of modules	**1**
Sensor	**Reverse-biased silicon diode array**
Sensor thickness	**320 µm**
Pixel size	**172 x 172 µm^2**
Format	**487 x 195 = 94 965 pixels**
Area	**83.8 x 33.5 mm^2**
Dynamic range	**20 bits (1: 1 048 576)**
Counting rate per pixel	**>2 x 10^6 X-ray/s**
Energy range	**5-30 keV**
Quantum efficiency (calculated)	**3 keV: 80 %**
	8 keV: 99 %
	15 keV: 55 %
Energy resolution	**500 eV**
Adjustable threshold range	**3 - 20 keV**
Threshold dispersion	**50 eV**
Readout time	**2.7 ms**
Framing rate	**200 Hz**
Point-spread function	**1 pixel**
Data formats	**Raw data, TIF , EDF, CBF**
External triggered/gate	**5V TTL, 3 different modes**
Software interface	**Through socket connection; clients for EPICS, SPEC and stand-alone operation are available**
Cooling	**Air cooled**
Dimensions (WHD)	**180 x 143 x 180 mm^3**
Weight Approx.	**Approx. 2 kg**

GISAXS setup includes microfocus X-ray source (IμS, Incoatec, Germany) that generates CuK_α radiation (8.04 keV, 0.154 nm). Quantum efficiency of silicon sensor for 8 keV is 99 %.

Counting rate per pixel of Pilatus detector is > 2 x 10^6 photons/s. In order to avoid overexposure of pixels of Pilatus detector, Mo filter with thickness of 25 μm has to be inserted into the X-ray beam path. The attenuation factor of Mo filter is ~2 %. The total flux of microfocus X-ray source in vacuum is ~3 x 10^8 photons/s. It means that attenuated intensity at the filter output is ~10^6 photons/s.

In addition, the total intensity is affected by intensity losses due to scattering in the air. The linear absorption coefficient for air is ~0.01 cm^{-1} at 1 atm and at 20 °C with CuK_α radiation. It means that intensity decreases to 37 % of the original value at a distance 100 cm due to scattering in the air. In GISAXS setup, distance between exit Be window of optics housing and sample is ~30 cm. It means that intensity impinging on the sample is ~74 % of the original value. For Be window-detector distance 100 cm and Mo filter, the total intensity in primary beam is ~3x 10^8 x 0.02 x 0.37=2.2 x 10^6 photons/s.

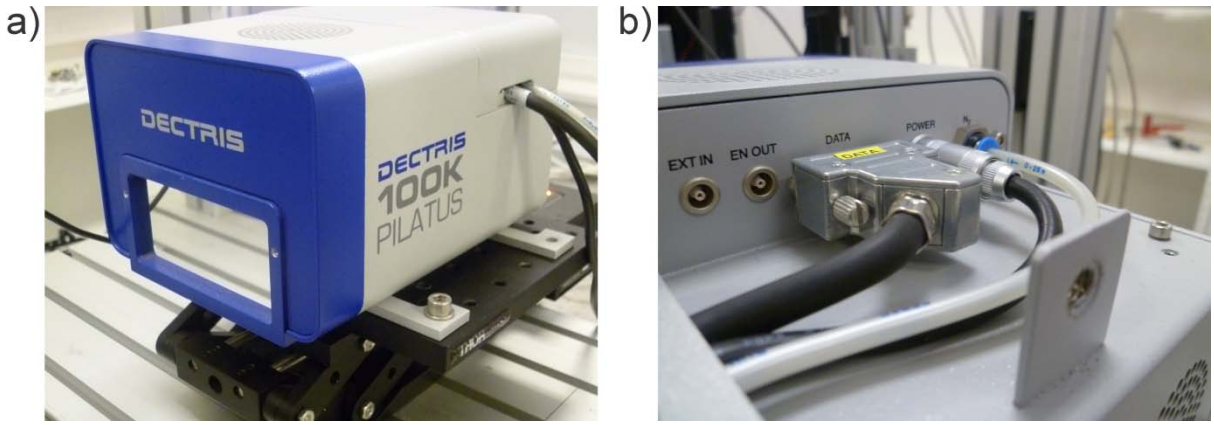


Figure 16. Images show a) front and b) back side of Pilatus detector.

The silicon sensor is placed behind a 20 μm thick aluminized mylar foil (Fig. 16a). The power supply is a compact switching power supply that can be connected to all common suplly voltages (100 – 240 VAC, 50/60 Hz, 700 mA) (Fig. 17a). The output of power supply

is +12 V DC/2.5A. The output connector of power supply is push-pull LEMO connector that plugs into the POWER connector on the back side Pilatus detector (Fig. 16b).

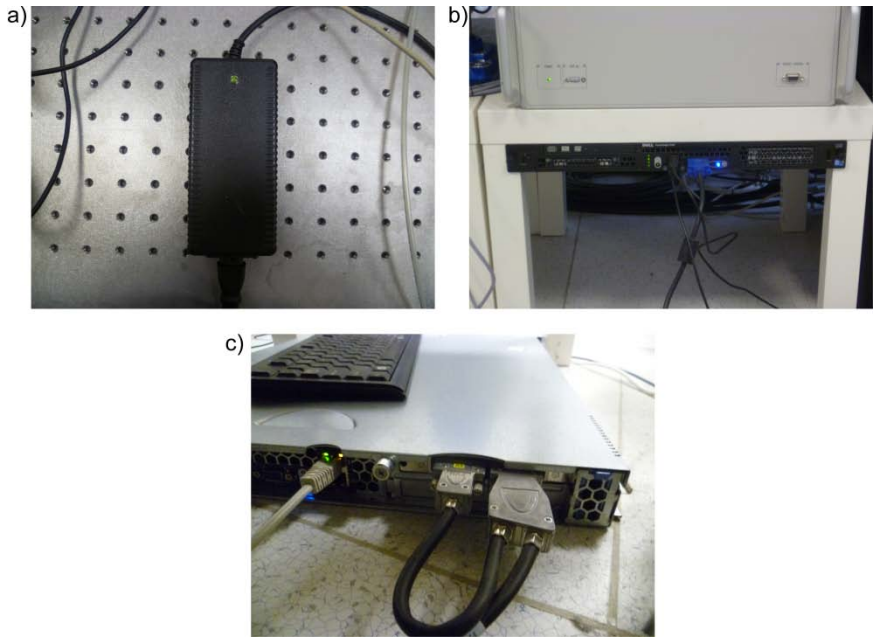


Figure 17. a) Power supply unit of Pilatus detector. b) Front side of Pilatus computer. c) Data acquisition card with TX and RX connectors on the back side of Pilatus computer.

The Pilatus computer (Fig. 17b) is a standard PC with customized LINUX OS and a proprietary data acquisition card to communicate with the detector. The connectors on the PC data acquisition card are RS232 connectors: TX (transmit data) and RX (receive data) (Fig. 17c).

The PILATUS detector has to be connected to a nitrogen flow to avoid humidity. Recommended continuous flow of nitrogen is 1 L/h. Use gas valve on the wall of X-ray lab to ensure flow of nitrogen gas into the Pilatus detector (Figs. 18a-b). The control of flow of nitrogen is possible due to nitrogen flow meter on the optical table of GISAXS setup (Fig. 18c). Please set flow rate to 1 L/h with knob on the front side of the flow meter (Fig. 18c). The input connector of Pilatus detector is Swagelok quick connector. It is labeled as N_2 connector at the back side of Pilatus detector (Fig. 16b).

Caution 1. The data cable should be pulled onto the computer connectors with the screws, rather than forcefully pushed on (Fig. 17c).

Caution 2. The operating system is optimized for high speed data acquisition and has a custom kernel: Therefore do not permit any software upgrades on the kernel.

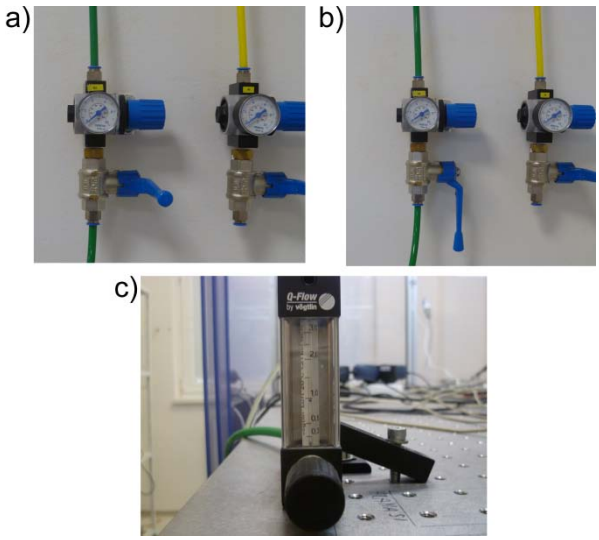


Figure 18. Gas valve in the a) OFF and b) ON positions. c) Nitrogen flow meter on the optical table. The flow rate of nitrogen is set to 1 L/h.

2.1 Detector operation under Linux OS

In the first step, log in to the Pilatus computer.

Normal log in procedure:

- User: **det**
- Password: **Pilatus2**

To change IP address of Linux OS, please follow instructions:

- Go to **Start Menu of Linux OS → Computer → Administrator Settings (YaST).**
- **Run as root – KDE su window** appears. Type Superuser/Root password: **!dec124!.**

- **YaST Control Center** of Pilatus detector appears**.**
- Go to **Network devices → Network Card → Overview menu** (**of Network Settings)**.
- Press **Configure button** and change Statically assigned IP address.
- Press **Next** and **Finish buttons**. It will establish new IP address.

Caution: Default gateway (147.213.112.1) in Linux OS can be set in **Routing menu of Network Settings**. Hostname/DNS servers can be set in **Hostname/DNS menu of Network Settings**. Name servers are: 147.213.1.1, 147.213.1.34. Check **Write Hostname to /etc/hosts box** in Hostname/DNS menu and press **Finish button**.

Disabling Firewall Service in Linux OS is important as Firewall can block TCP/IP communication between Pilatus computer (Linux OS) and GISAXS computer (WinXP). Go to **Security and Users → Firewall → Disable Firewall Automatic Starting**. Press **Next** and **Finish buttons**.

To communicate with Pilatus detector in Linux OS, you have to open **Camserver window**. Please follow instructions:

- Go to **Start Menu** of Linux OS → **Terminal Program** Konsole. It will open **Shell-Konsole of Linux OS** (Fig. 19a).
- Type **cd p2_det**. It will go to p2_det directory.
- Type **ls**. It will show all files in the current directory.
- Type **camonly**. It will open **Camserver** of Pilatus detector (Fig. 19b).
- To **exit Camserver**, type **exit** command.

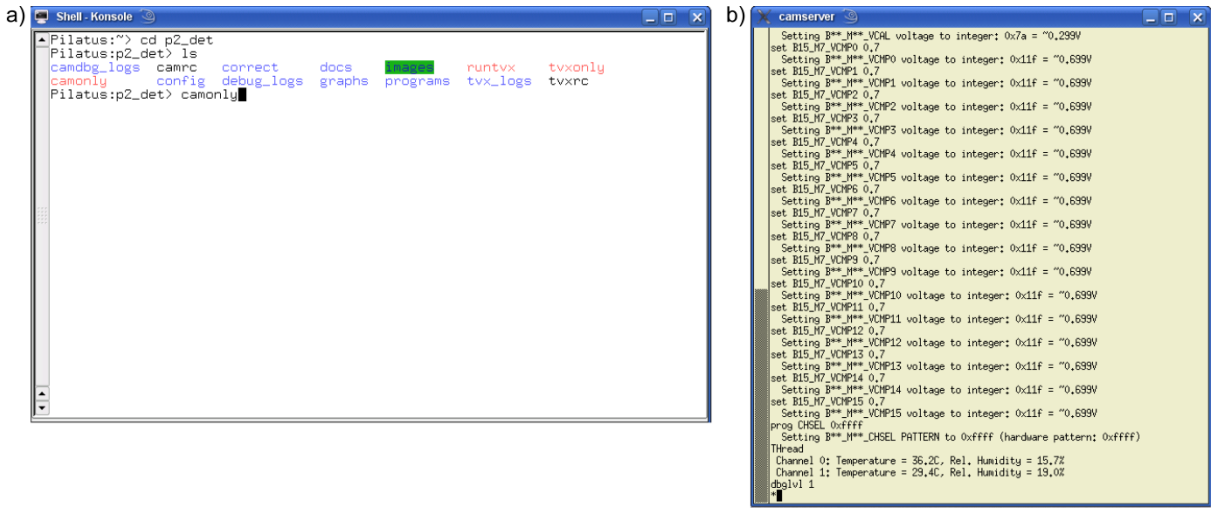


Figure 19. a) Shell-Konsole and b) Camserver windows of Pilatus detector.

To set threshold energy, please follow instructions:

- Open **Camserver** of Pilatus detector as described above.
- Type **setthreshold midg 5000**. It will set gain and threshold energy of Pilatus detector to midg and 5000 eV, respectively. The above settings are recommended for CuK_{α} radiation (8.04 keV, 0.154 nm).

To take image with Camserver, please follow instructions:

- Open **Camserver** of Pilatus detector and set energy threshold as described above.
- Type **ni N** or **nimages N**. Number of images in sequence will be set to N. E.g. ni 10 will set number of images in sequence to 10 images.
- Type **expt X** or **exptime X**. Exposure time will be set to X s. E.g. expt 1 will set exposure time to 1 s.
- Type **expp Y** or **expperiod Y**. Exposure period will be set to Y s. E.g. expp 1.1 will set exposure time to 1.1 s. The exposure period must be at least 3 ms longer than the exposure time.

- Type **expo filename.tif** or **exposure filename.tif**. Make an exposure with specified name. E.g. expo filename.tif will make exposure with name: filename.tif. Except tif format, Pilatus detector supports following formats: raw, edf, cbf.
- To **exit Camserver**, type **exit** command.

To communicate with Pilatus detector in Linux OS, you can also use **TVX** in combination with **Camserver** (Fig.20). Please follow instructions:

- Go to **Start Menu** of Linux OS → **Terminal Program** Konsole. It will open **Shell-Konsole of Linux OS**.
- Type **cd p2_det**. It will go to p2_det directory.
- Type **ls**. It will show all files in the current directory.
- Type **runtvx**. It will open **TVX + Camserver + Image windows** (Fig. 20).
- Set threshold energy in Camserver window. Type **setthreshold midg 5000.**
- To **exit Camserver** and **TVX**, type **exit** command into the both windows. It will close them.

I recommend to use only Camserver to take images. Runtvx is e.g. useful to make continuous exposure. Please type following command:

- Type **exposem X** into the TVX window. It will activate continuous camera mode with exposure time X without saving images. It will take images until any key is pressed. The last image is stored in temp.tif. E.g. exposem 1 will make continuous image sequence with exposure time 1 s.
- Type **expose X** into the TVX window**.** It will take an image with specified exposure time X. E.g. expose 1 will take an image with exposure time 1 s.

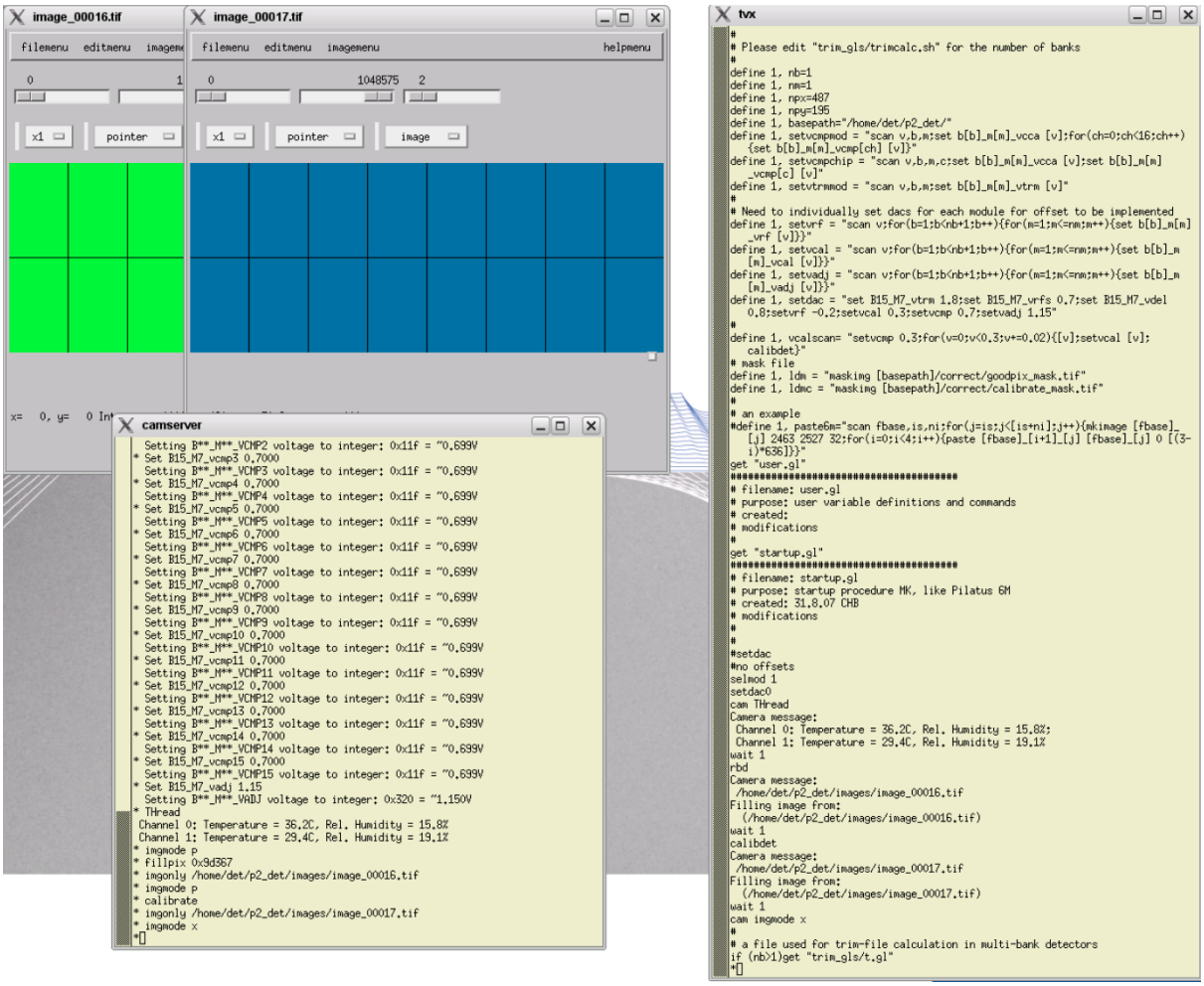


Figure 20. TVX, Camserver and Image windows of Pilatus detector.

2.2 Detector operation under Win XP

The Pilatus detector can be remotely controlled by GISAXS computer (Fig. 21). It is possible thanks to X-Win32 program (X Windows emulator) that allows remote display performance of LINUX desktop on the LAN (Fig. 22a). To start communication with Pilatus computer through X-Win32 program, please follow instructions:

- Open **Start Menu of Win XP**.
- Go to **Programs → XWin32** (Fig. 22a).
- **X-win32 logo** appears in the notification area of WinXP system (Fig. 22b).
- Right click on **X-win32 logo**. The X-win32 menu appears (Fig. 22c).

- Go to **My Sessions** and select **Pilatus session** (Fig. 22c).
- **Xterm window** appears on the screen. It is a mirror of Shell-Konsole in LINUX OS (Fig. 23).
- Type **cd p2_det.** It will go to p2_det directory).
- Type **ls**. It will show all files in the current directory.
- Type **camonly**. It will start **Camserver**.
- Type **setthreshold midg 5000**. It will set gain and threshold energy of Pilatus detector to midg and 5000 eV, respectively. The above settings are recommended for CuK_{α} radiation (8.04 keV, 0.154 nm).
- To take image with **Camserver**, please see previous section: **Detector operation under LINUX OS**. There is explained in-detail how to operate Pilatus detector with Camserver.

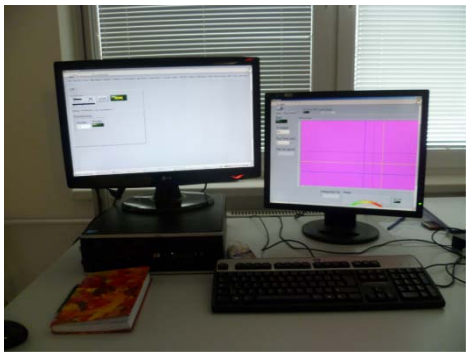


Figure 21. GISAXS computer.

Pilatus session has following parameters that can be set in **X-config (X-Win32):**

- **Session Name: Pilatus** (session type: StarNetSSH).
- **Host: 147.213.112.19** (IP address of Pilatus computer).
- **Port: 22** (port of Pilatus computer).
- **Login: det** (login of Pilatus computer).
- **Command: xterm** (it will start shell in SUSE).
- **Password: Pilatus2**
- **Confirm Password: Pilatus2**

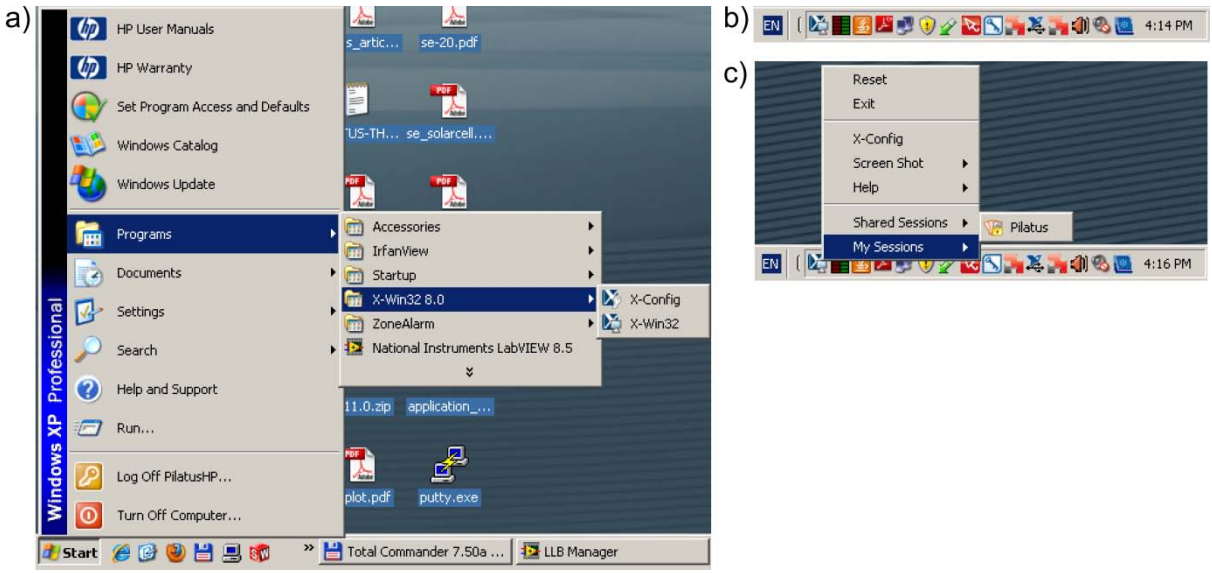


Figure 22. a) X-Win32 program in Programs menu. Notification area of Win XP with **X**-win32 logo. c) X-Win32 menu with Pilatus session that appears above notification area of Win XP.

Caution 1: Disable Firewall of Win XP as Firewall can block TCP/IP communication between Pilatus computer (Linux OS) and GISAXS computer (WinXP).

Caution 2: On the GISAXS computer, Zone Alarm Free Firewall is installed. To start Zone Alarm, go to **Start Menu of Win XP → Programs → Zone Alarm → Zone Alarm Security → Zones** (Fig. 24). Find IP address of Pilatus computer: 147.213.112.19. If IP address of Pilatus computer is not in the list, please add IP address of Pilatus computer. Select Trusted Zone.

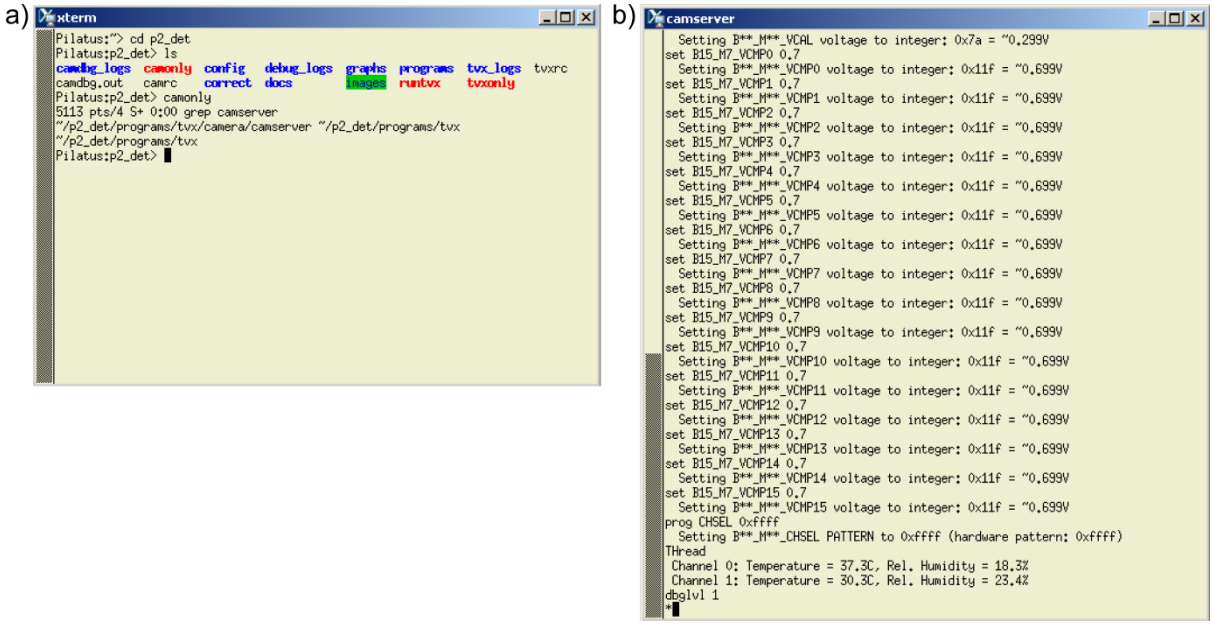


Figure 23. a) Xterm and b) Camserver windows in WinXP.

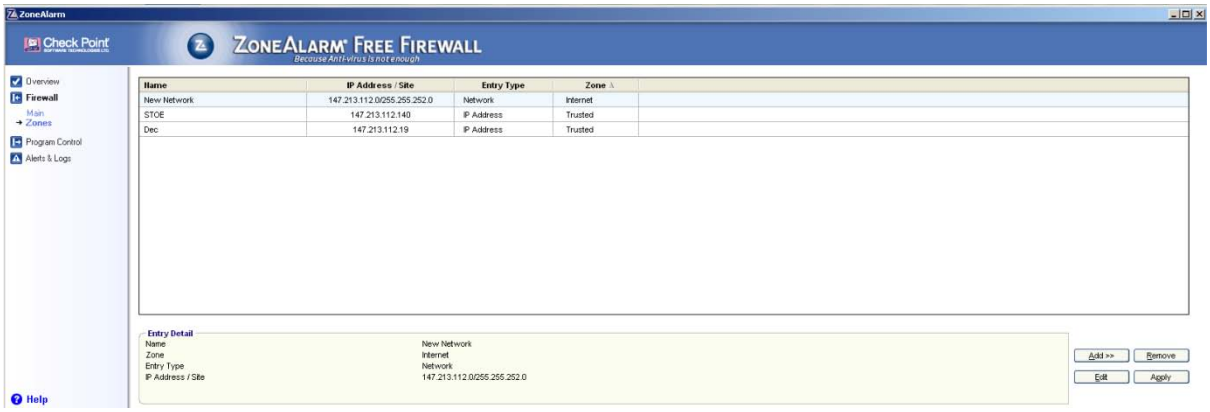


Figure 24. Zone Alarm window in Win XP.

3 Motors

Motors can be divided into 5 groups:

- **Newport motors**
- **Thorlabs motors**
- **Newfocus motor**
- **AFM motor**

3.1 Newport motors

GISAXS setup includes two Newport motors: **linear stage** (M-IMS300V) and **rotation stage** (RV120PP-F). Linear and rotation stages are responsible for positioning of X-ray source (Fig. 25). Rotation stage adjusts angle of incidence in GISAXS experiment on liquid surfaces.

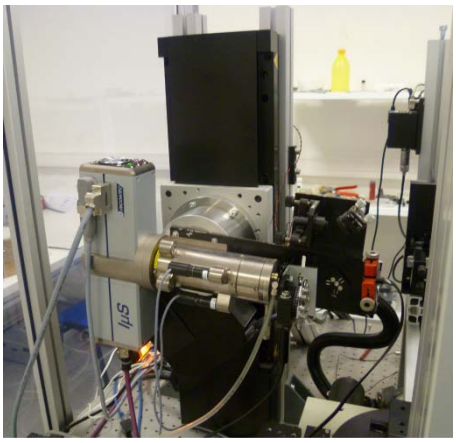


Figure 25. X-ray source mounted on rotation and vertical stage.

Newport motors are controlled via XPS controller and two driver cards installed on the back side of the XPS controller (Fig. 26a-b). XPS controller is connected to the GISAXS computer via Ethernet cable. Two connectors are available on the back side of XPS controller: HOST and REMOTE (Fig. 26c). I recommend direct connection between GISAXS computer and HOST connector. Please use cross-over cable.

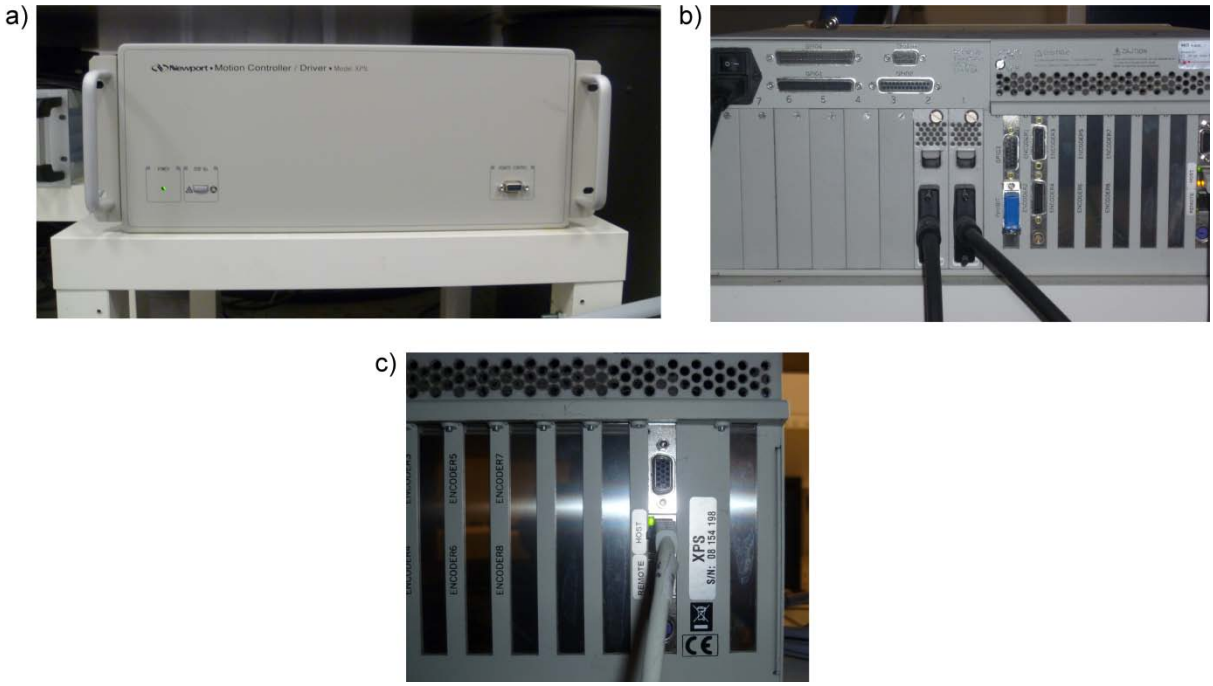


Figure 26. a) Front panel of the XPS controller. b) Rare panel of the XPS controller with two driver cards. c) Zoom in on HOST and REMOTE connectors on the rear panel of XPS controller.

TCP/IP configuration of XPS controller:

- Static IP address: **147.213.112.49**
- NetMask value: **255.255.252.0**
- Gateway IP address: **147.213.112.1**

In order to be able to communicate with the controller, please follow instructions:

- Open web browser (Internet Explorer, Mozilla Firefox) and connect to **http://147.213.112.49**
- The welcome web page of XPS controller appears (Fig. 27a).
- Fill the following fields: **Name**: Administrator; **Password**: Administrator (they are valid for both user and administrator rights)
- Press **Enter**

Once when you entered to the XPS webpage, click on the **FRONT PANEL** and then on **Move** (Fig. 27b). On the move page, you will see two positioner names: **GROUP1.POSITIONER** and **GROUP2.POSITIONER**. **GROUP1.POSITIONER** refers to the **linear stage**. **GROUP2.POSITIONER** refers to the **rotation stage**. The move page also provides access to basic group functions like initialize, home, or motor disable, and allows executing relative and absolute moves.

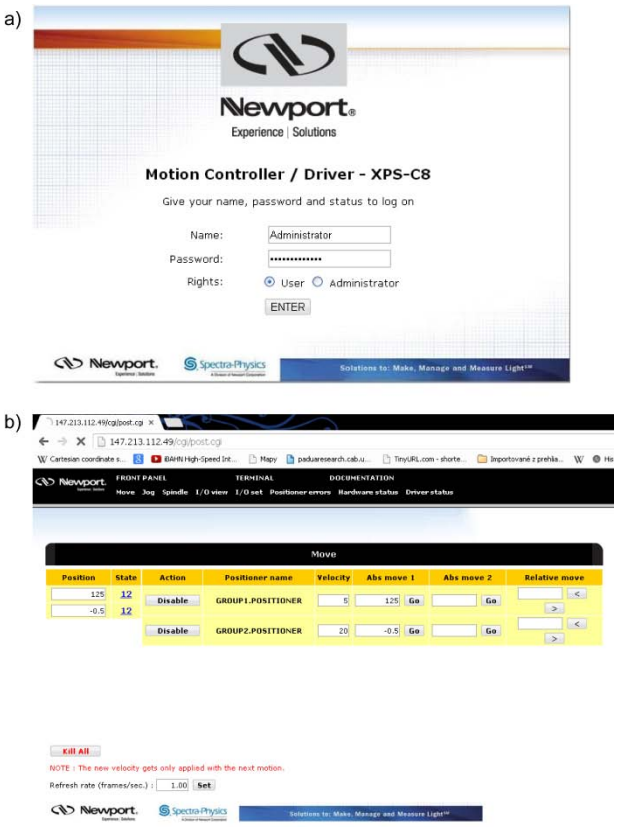


Figure 27. a) Welcome page and b) Move page of XPS controller.

To stop all motors push **STOP ALL button** on the front panel of the XPS controller (Fig. 28). It is used for the **Emergency Stop** of the system and will immediately shut down all the motors. Once when the motors were stopped, it is necessary to restart them.

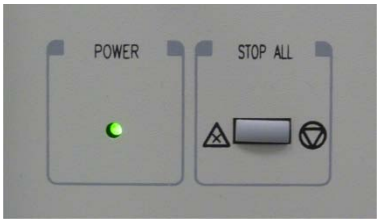


Figure 28. STOP ALL button and LED on the front panel of XPS controller.

To restart Newport motors turn off and turn on XPS controller using **POWER ON/OFF** switch on the back side of XPS controller. The LED on the front panel of the XPS controller will turn green. There is an initial beep when the XPS controller is switched on. Once the controller completes booting up there is a second beep. It can take 1 to 2 minutes for the controller to boot up. Then log in to the XPS webpage as described above. Go to **FRONT PANEL→Move**. Select positioner name you want to initialize. Press **Initialize** and then **Home** button.

3.2 Thorlabs motors

Thorlabs motors are the most important motors used in GISAXS setup. Thorlabs motors can be divided into (Fig. 29):

- **MTS25/M-Z8** (linear stage, travel range 25 mm)
- **L490MZ/M** (LabJack, linear stage, travel range 50 mm)
- **PRM1/MZ8** (rotation stage)
- **CR1/M-Z7** (rotation stage)

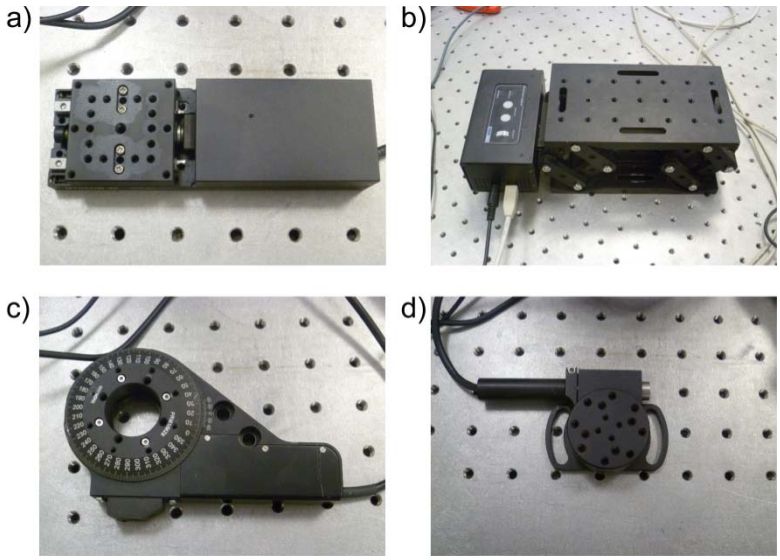


Figure 29. Images of linear stage a) MTS25/M-Z8 and b) heavy-load motor L490MZ/M. Images of rotation stages: c) PRM1/MZ8 and d) CR1/M-Z7.

MTS25/M-Z8, PRM1/MZ8, CR1/M-Z7 are controlled by **T-Cube** DC Servo Motor Controller: **TDC001** (Figs. 30a-b). It is single–channel controller that enables manual and remote control of DC servo motor. On the front panel of T-cube, USB and DC POWER connectors are placed (Fig. 30a). On the rear panel of T-cube, D-type (15 pin) connector is placed (Fig. 30b). Installation of TDC001 controller:

- Connect the stage to the D-type (15 pin) connector of TDC001 controller.
- Connect TDC001 controller (DC connector) to the main supply and switch on.
- Connect TDC001 controller (USB connector) to the USB hub of GISAXS computer (Fig. 30c).

Caution: The distance between USB hub and GISAXS computer exceeds 3 meters. To avoid signal decay, amplified USB extension cables/USB repeaters have to be used (Fig. 30d).

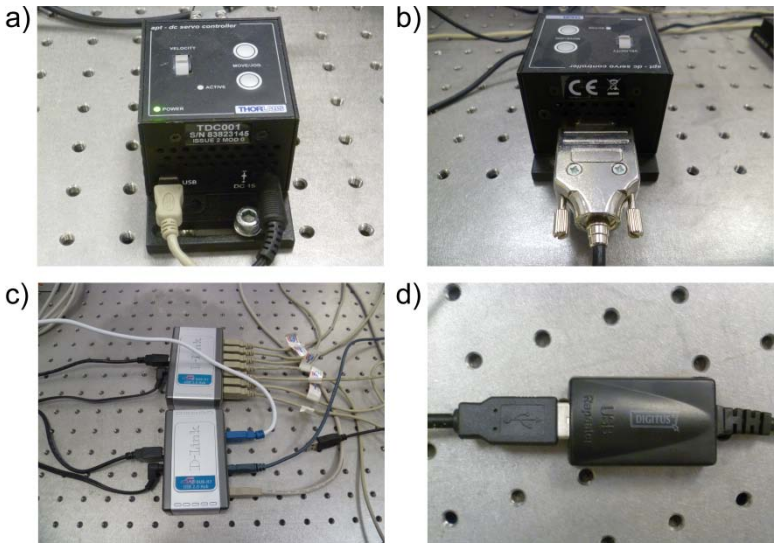


Figure 30. a) Front panel of T-cube TDC001 with USB and DC POWER connectors. b) Rear panel of T-cube TDC001 with D-type (15 pin) connector. c) D-link 7-Port USB hub. d) USB repeater DIGITUS.

To move Thorlabs motors manually, please use Velocity Potentiometer and Move/Jog buttons on the top panel of T-cube (Fig. 31). Move/Jog buttons allow to jog the motor and make discrete position increments in either direction. Velocity Potentiometer allows to drive the motor at a varying speed in either forward or reverse directions for fast and easy motor control.

Figure 31. Top-view on T-cube TDC001 controller.

Once the motors were connected to the GISAXS computer, it is necessary to **configure** them. Please follow instructions below:

- Open **Start Menu**.
- Go to **Programs → Thorlabs → APT → APT Config**.
- In the **ATP Config**, the list of available motors can be seen (Fig. 32). Each Thorlabs motor connected to the GISAXS computer is identified by serial number of its T-cube controller.
- In the **Serial No. field**, select the serial number of your motor.
- In the **Stage field**, select type of motor (e.g. MTS25/M-Z8).
- Press **Add/Change Stage Association button**. In this way, motor with serial number can be added to the list in the main window.
- To remove stage from the list of motors press **Remove Stage Association button**.
- Control the list of motors in the main window if it contains all motors.
- Press **Exit** button to leave **APT Config**.

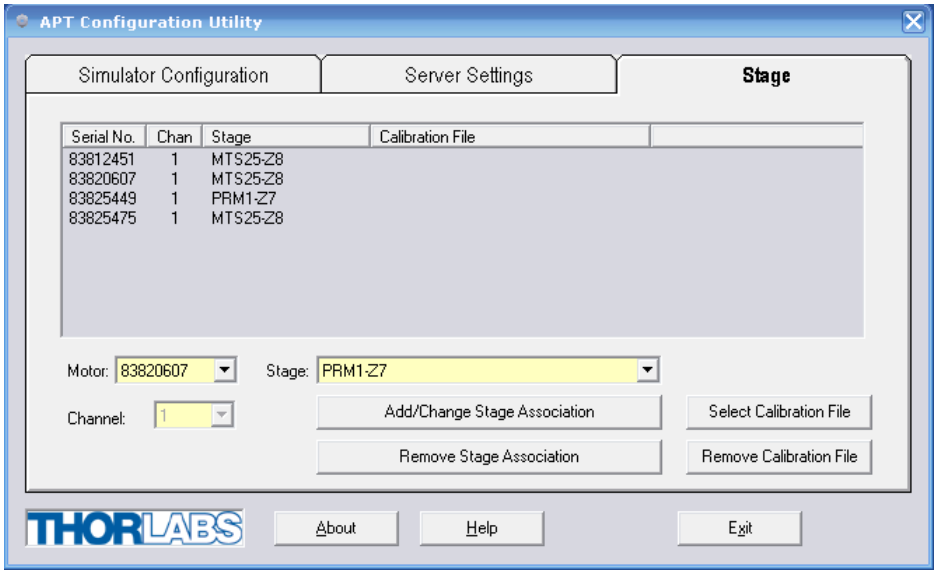


Figure 32. GUI of APT Configuration Utility program.

To control Thorlabs motors from GISAXS computer, you have to use APT User software (Fig. 33). Please follow instructions:

- Open **Start Menu**.
- Go to **Programs → Thorlabs → APT → APT User**.
- In the **APT User**, you can home motor and execute relative/absolute moves (Fig. 33).

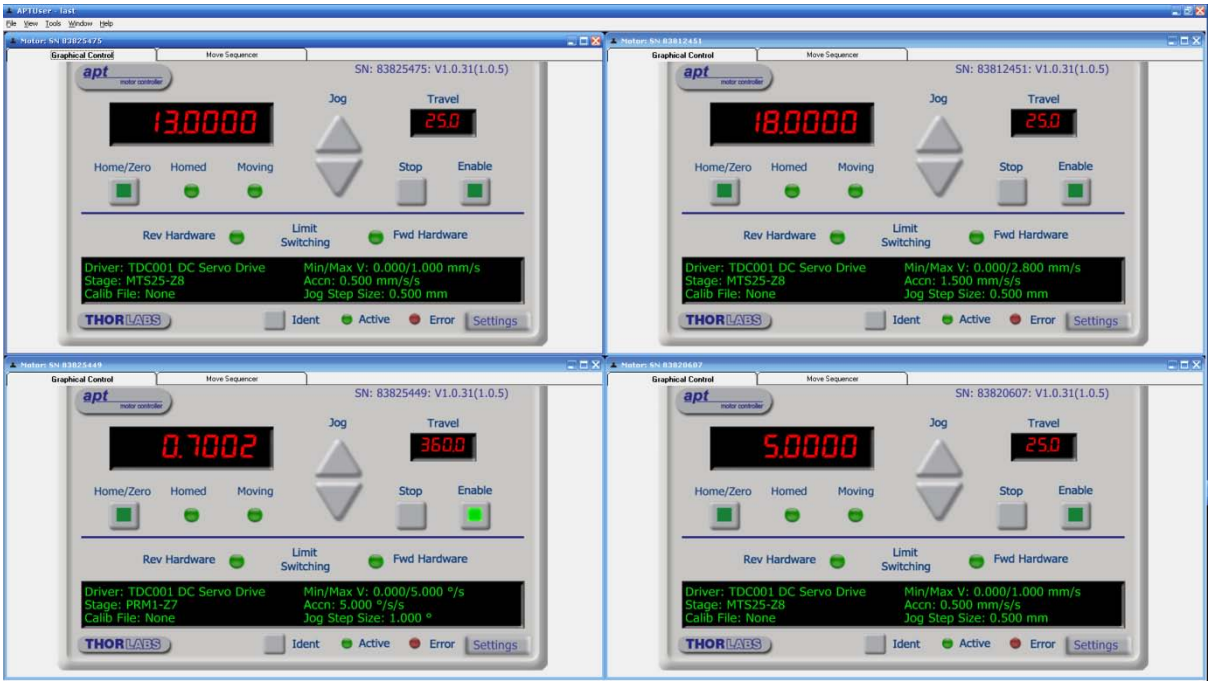


Figure 33. GUI of APT User program.

3.3 Newfocus motors

Newfocus picomotor (8051-M) works on the IP address (147.213.112.152) and port (23). It allows XY positioning of the first external pinhole in GISAXS setup (Fig. 34a). The picomotor is connected to the Picomotor Ethernet Controller (Model 8753) (Fig. 34b). The picomotor can be driven manually or remotely. The manual control is possible due to the Hand Terminal (Model 8757) that is plugged directly into the picomotor controller (Fig. 34c). GISAXS computer is connected to the picomotor controller via Ethernet cable that allows remote control.

Figure 34. Image shows a) Newfocus Picomotor, b) Picomotor Ethernet Controller and c) Handle Terminal.

The hand terminal (Model 8757) (Fig. 34c) allows you to select type of motor, adjust speed of movement and send continuous step. Please follow instructions below:

- Press **Motor button** on the hand terminal. It will **select motor** (Mot A, Mot B, Mot C). **Mot A** is responsible for movement in the **horizontal (X) direction**. **Motor B** is responsible for movement in the **vertical (Y) direction**. Mot C is not relevant.
- Press **Hi/Lo button**. It allows you to change speed. The hand terminal can work at two speed levels: **Coarse** (2000 Hz) and **Fine** (250 Hz). Coarse is high speed mode. Fine is low speed mode.
- Press **–JOG/+JOG (-STEP/+STEP) button.** It will move motor in negative/positive direction.
- Press **STOP button** to stop continuous movement of motor.

The picomotor can be also driven remotely from GISAXS computer (MainQueue.vi).

TCP/IP configuration of picomotor controller:

- Mode: **Static**
- Hostname: **C1**
- IP address: **147.213.112.152**
- Subnet Mask: **255.255.252.0**
- Default Gateway: **147.213.112.1**

Open web browser (Internet Explorer, Mozilla Firefox) and connect to the IP address of picomotor: **http://147.213.112.152**. On the NEW FOCUS webpage, the current IP settings of picomotor controller can be found.

3.4 AFM motor

AFM motor is a Newport motor (M-MTM100CC.1) (Fig. 35a). It can bear up to 100 kg with an exceptional high axial load capacity up to 200 N. The high normal load capacity (1000 N) was necessary as motor should bear commercial AFM (Dimension Edge, Bruker AXS) and STOE diffractometer. AFM motor is driven by Single-Axis Motion Controller (SMC100CC) (Fig. 35b). On the front panel of SMC100CC controller box, you can find RS232C communication port for computer communication. GISAXS computer has no RS232 communication port. Therefore, RS232-USB converter is used to ensure communication between SMC100CC controller box and computer (Fig. 35c). On the back side of SMC100CC controller box, you can find power supply input (DC IN) and 25 pin motor connection (MOTOR).

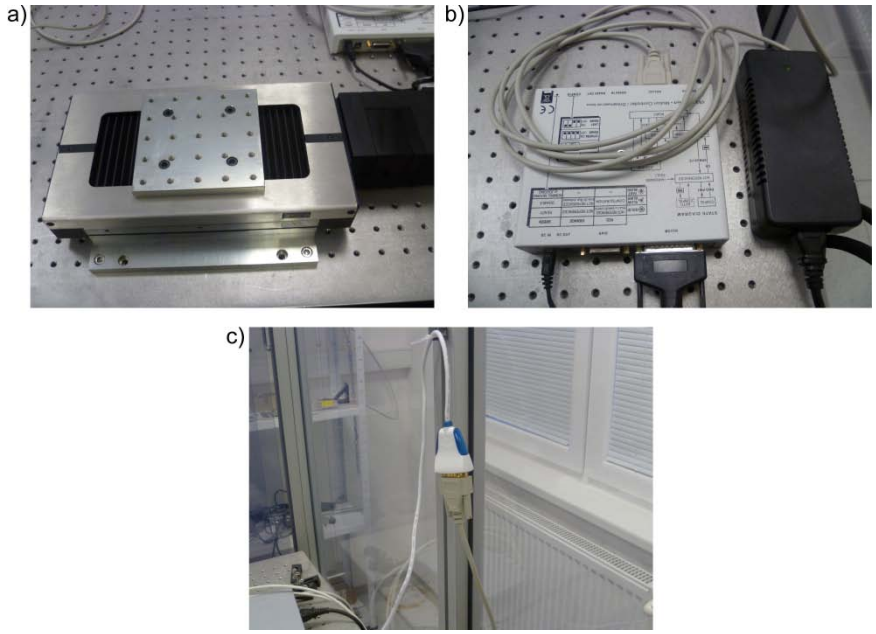
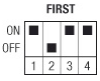


Figure 35. Image shows a) AFM motor, b) SMC100 controller box with power supply unit and RS232-USB converter.

Motor connection:

- Set the dip switches on the SMC100CC to FIRST 

.
- Connect Newport motor (M-MTM100CC.1) to MOTOR connector of SMC100CC controller box.
- Connect RS232-USB converter to RS232C communication port of SMC100CC controller box.
- Connect the power supply to the SMC100CC controller box. The LED on the SMC100CC turns RED.

AFM motor cannot be driven manually as handle terminal (SMC-RC) was not purchased.

AFM motor can be driven remotely from GISAXS computer. Pleases follow instructions:

- Go to **Newport → SMC100Tools → SMC100 User Tools**.

- Open the SMC100CC utility program **SMC100 User Tool** that is located on the Desktop of your GISAXS computer (Fig. 36).

- Press **Configuration button**.
- Press **Communication settings button**.
- Select **Port** (COM 4).
- Press **Open** button. It will open communication.
- Go back to the main menu and press **Motion portal button**. Here you can set values of absolute/relative moves. The travel range of motor is 100 mm. It can move from -50 mm to +50 mm.

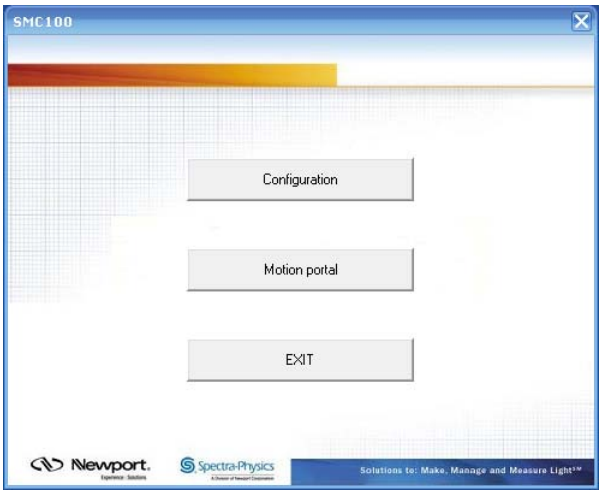


Figure 36. GUI of SMC100 User Tool program.

4 Operation of GISAXS setup in Lab View – GISAXS.llb

All programs and sub-programs necessary to operate GISAXS setup are in **GISAXS.llb** (GISAXS library) (Fig. 37)**.** To open **GISAXS.llb**, please follow instructions:

- Go to **Desktop of Win XP**
- Open **GISAXS_main folder**.
- Open **GISAXS.llb** (GISAXS library).

The top level programs (under line) in GISAXS library are (Fig. 37):

- **CamGISAXS.vi**
- **MainQueue.vi**
- **Pilatus_live.vi**
- **Pilatus_setthreshold_main.vi**
- **Pilatus_snap_main.vi**

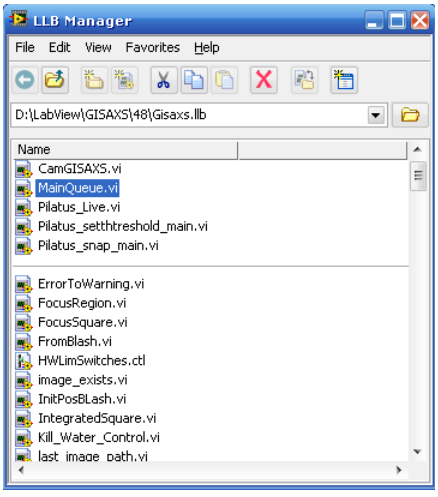


Figure 37. Picture shows GISAXS.llb. The programs under line are top level programs.

MainQueue is the main program that allows you to control motors and X-ray detector.

X-ray detector can be also controlled by separate programs:

- **Pilatus_snap_main.vi**
- **Pilatus_setthreshold_main.vi**
- **Pilatus_live.vi**

Basic notes to Lab View:

- Double click on *.vi, you want to operate (e.g.Pilatus_snap_main.vi).
- To **run *.vi** click on **run button** on the front panel toolbar (Fig. 38a).
- To **stop *.vi** click on **stop button** on the front panel toolbar (Fig. 38b).

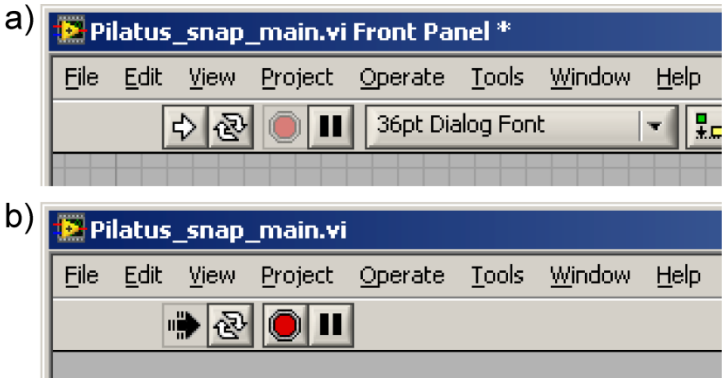


Figure 38. Front panel toolbar of a) not running vi and b) running vi. Not running vi shows run button . Running vi shows stop button .

5 Detector operation in Lab View – GISAXS.llb

To operate detector on GISAXS computer, **Camserver** has to be started in Win XP. The procedure how to start Camserver on GISAXS computer is in-detail explained in the chapter X-ray detector–Pilatus 100K in the section Detector operation under Win XP. The communication with Pilatus detector works through Telnet communication in Lab View.

Caution: Telnet communication is not included in LabView. Therefore, if you reinstall LabView, NI LabView Internet toolkit has to be additionally installed on GISAXS computer. Otherwise, Telnet communication with detector will not work.

The following programs

- **MainQueue.vi**
- **Pilatus_live.vi**
- **Pilatus_setthreshold_main.vi**
- **Pilatus_snap_main.vi**

open a Telnet Connection using the IP address (147.213.112.19) and remote port (41234) of Pilatus detector.

GISAXS computer has following IP address:

- IP address: **147.213.112.243**
- Subnet mask: **255.255.252.0**
- Default gateway: **147.213.112.1**
- Preferred DNS server:**147.213.1.1/147.213.1.34** (use one of two options)
- Alternate DNS server: **208.67.220.220**

Pilatus computer has following IP address:

- IP address: **147.213.112.19**
- Subnet mask: **255.255.252.0**
- Default gateway: **147.213.112.1**
- Preferred DNS server:**147.213.1.1/147.213.1.34** (use one of two options)
- Alternate DNS server: **208.67.220.220**

5.1 Set threshold - Pilatus_setthreshold_main.vi

Program **Pilatus_setthreshold_main.vi** allows you to set threshold energy of Pilatus detector (Fig. 39). Before doing any exposure, you have to set threshold energy. In normal operation, the threshold energy should always be set to 50 % of the energy of the incoming X-rays (CuK_α radiation; 8.04 keV). The recommended settings for CuK_α radiation are **MidGain** and threshold energy set to **5000 eV**. To operate Pilatus_setthreshold_main.vi, please follow instructions:

- Open and run **Pilatus_setthreshold_main.vi.**
- Select **midG** (middle gain) in the **Set threshold scroll menu**.
- Type threshold energy **5000 eV** in **Energy (eV) box.**
- Press **Send button**. It will set threshold energy of Pilatus detector.

The program sends following command to the **Camserver** of Pilatus detector:

- **setthreshold midg 5000**

It will set gain and threshold energy of Pilatus detector to midg and 5000 eV, respectively. The above settings are recommended for CuK_α radiation (8.04 keV, 0.154 nm).

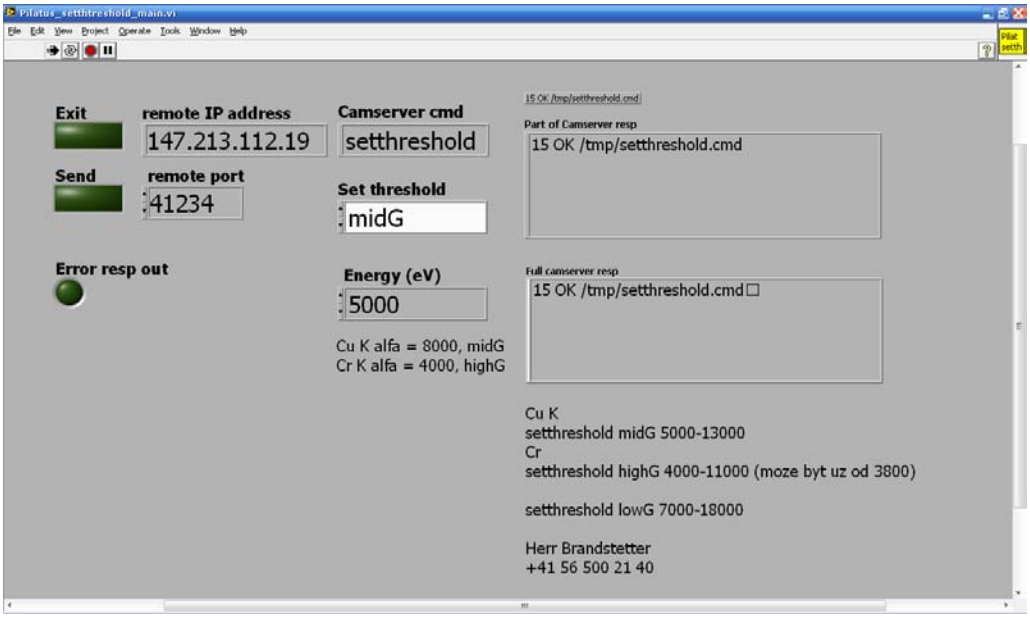


Figure 39. Front panel of Pilatus_setthreshold_main.vi. Gain of Pilatus detector is set to midG and threshold energy is set to 5000 eV.

5.2 Bad pixels of Pilatus detector

For CuK_{α} radiation (8.04 keV, 0.154 nm), Pilatus detector has two bad pixels: (445,143) and (167, 76). The bad pixels have to be removed and set to zero value. It is done automatically in *.vi programs communicating with Camserver of Pilatus detector.

5.3 Take image with Pilatus detector - Pilatus_snap_main.vi

Program **Pilatus_snap_main.vi** allows to take N images in sequence with specified exposure time and name (Fig. 40). To operate Pilatus_snap_main.vi, please follow instructions:

- Open and run **Pilatus_snap_main.vi.**
- Type number of images into the **N images box**.
- Type exposure time into the **Exp Time (sec) box.**
- Type exposure period into the **Exp Period (sec) box**. The exposure period must be at least 3 ms longer than the exposure time.

- Type image name into the **Image Name box**. Never add suffix **(*.tif)** behind the image name. It will be done automatically. E.g., if you type image name "**image**", the program will send to the **Camserver** of Pilatus detector "**image_x.tif**". The suffix "**_x**" prevents wrong numbering.
- Press **Snap button**. It will make **N** images with specified exposure time and name.

The program sends following commands to the **Camserver** of Pilatus detector:

- **ni N** (number of images in sequence is X, e.g. ni 10)
- **expt X** (exposure time is X s, e.g. expt 1 s)
- **expp Y** (exposure period is Y s, e.g. expp 1.01 s)
- **expo image.tif** (expose images with specified name and .tif suffix)

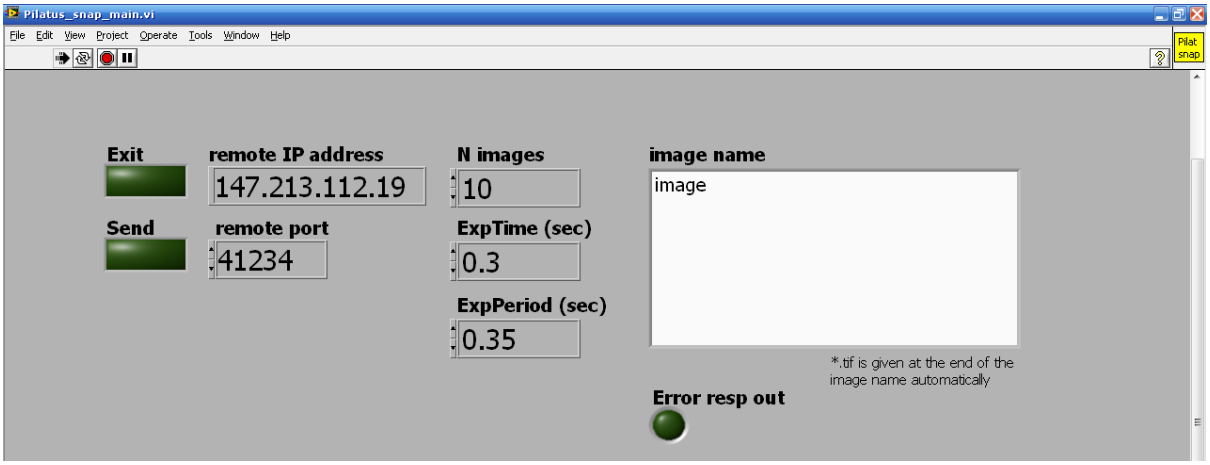


Figure 40. Front panel of Pilatus_snap_main.vi. Numeber of images in sequence, exposure time and exposure period are 10, 0.3 s and 0.35 s, respectively.

5.4 Pilatus detector in live mode

Program **Pilatus_Live.vi** allows to take N images with specified exposure time in live mode (Fig. 41). It means that sequence of images will show as a movie. To operate Pilatus_Live.vi, please follow instructions:

- Open and run **Pilatus_Live.vi.**

- Go to **Snap tab**.
- Type number of images into the **N images box**.
- Type exposure time into the **Exp Time (sec)** box**.**
- **Exposure period** will be automatically set to the value that is 5 ms larger than exposure time.
- Image name will be automatically set to **live.tif**.
- Press **Start button**. It will take **N** images in sequence with specified expsoure time. During exposure, **Start button** is disabled. After finishing exposure, **Start button** is enabled.
- To stop program operation, press **Stop button**  on the front panel toolbar or **Exit button** on the front panel of **Pilatus_Live.vi**. I prefer to use **Stop button**.

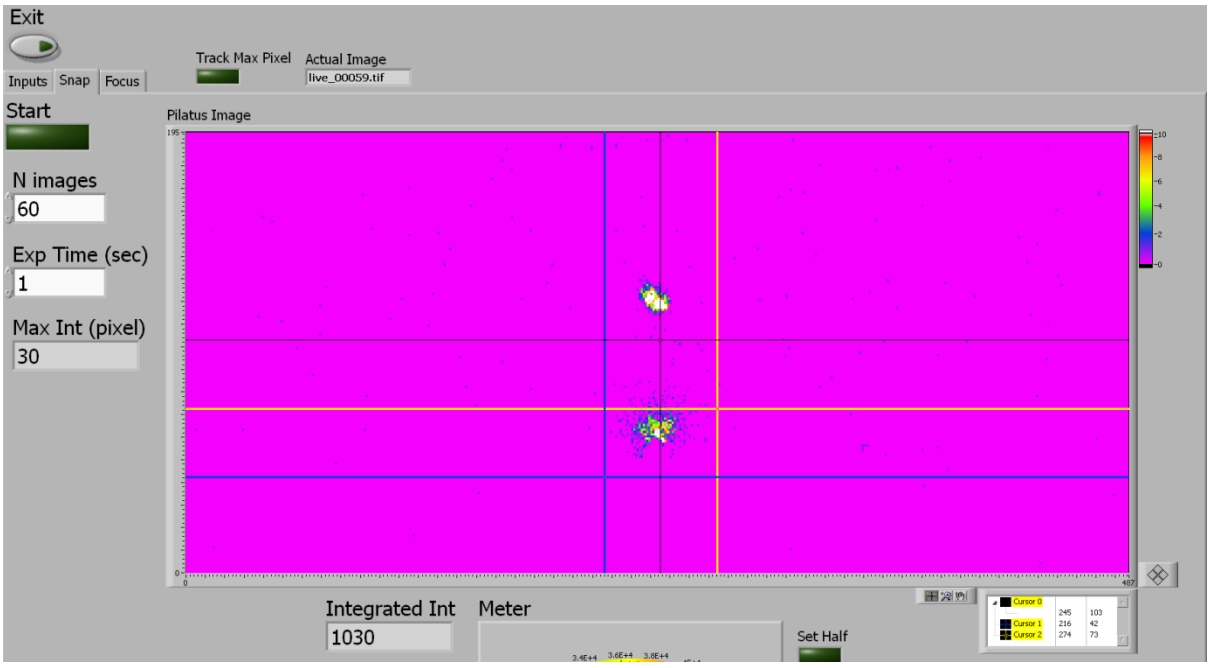


Figure 41. Snap tab of Pilatus_Live.vi.

The program sends following commands to the **Camserver** of Pilatus detector:

- **ni N** (number of images in sequence is N to 100, N=100)
- **expt X** (exposure time is X s, e.g. expt 1 s)

- **expp Y** (exposure period is Y s, e.g. expp 1.01 s)
- **expo live.tif** (expose images with following name live.tif)

The images appear in the folder: **/home/det/p2_det/images/.** The images from **/home/det/p2_det/images/** folder are copied into the shared **z:/** space on the hard drive of GISAXS computer. The program **Pilatus_Live.vi** opens the last created image (live_*.tif) and shows it in its window. The name of last opened image appears in **Actual Image box**.

You can zoom in on particular X-ray feature appearing on Pilatus detector. Please drag blue and yellow cursors in **Snap tab** to select your region of interest. The selected area appears in **Focus window** in **Focus tab** (Fig. 42). Press **Fit button** in **Fit tab**. It will integrate your feature along horizontal and vertical axes. The horizontal and vertical intensity profiles will be fitted with Gaussian function. The FWHM of horizontal and vertical intensity profiles appear in **FWHM-Hor (µm)** and **FWHM-Ver (µm) boxes**, respectively. It can be used e.g. for fast characterization of primary X-ray beam.

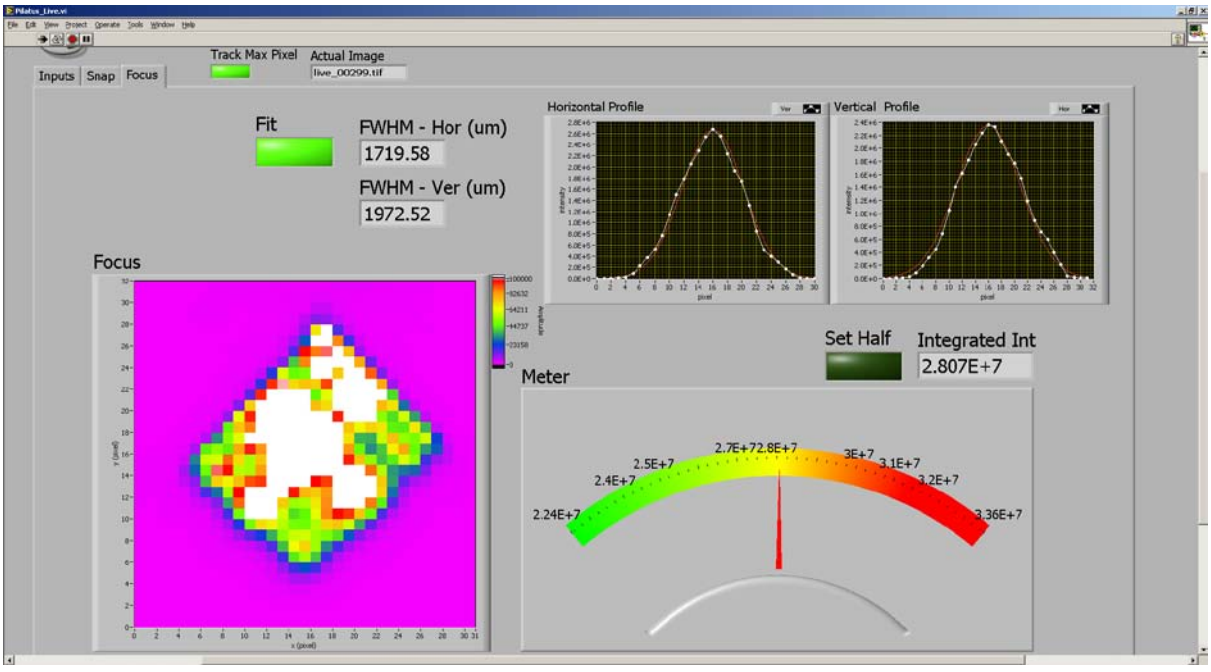


Figure 42. Fit tab of Pilatus_Live.vi.

The total intensity in zoomed area appears in **Integrated Int box** and **Meter** (Fig. 43). The total intensity is visible in both **Snap** and **Focus tab**. Press **Set Half button** (Fig. 43). It will reset the meter needle position.

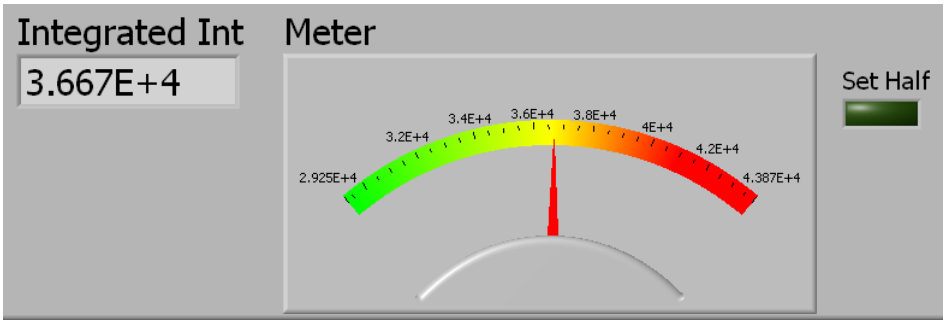


Figure 43. Integrated Int box and Meter in Snap tab of Pilatus_Live.vi.

Press **Track Max Pixel button** (Fig. 44). Maximum intensity in pixel appears in **Max Int (pixel) box** in **Snap tab** (Fig. 44).

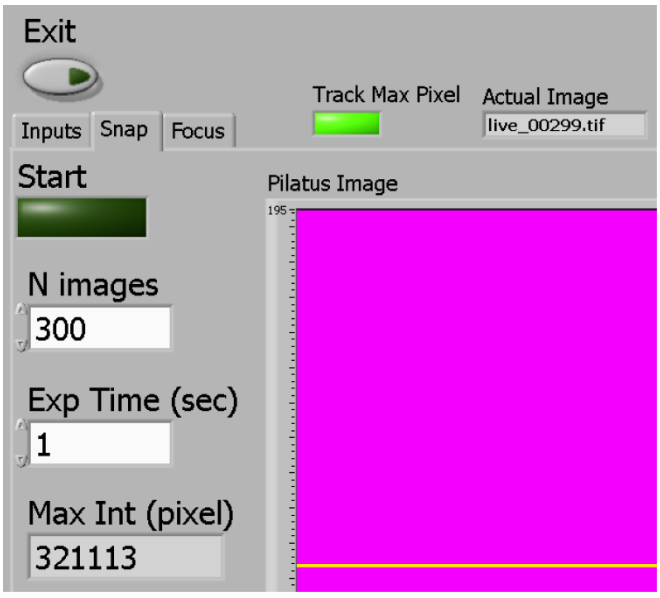


Figure 44. Image shows activated Track Max Pixel button and Max Int (pixel) box.

5.5 Detector operation - MainQueue.vi

The previously introduced programs (**Pilatus_snap_main.vi, Pilatus_setthreshold_main.vi)** are integrated into the **MainQueue.vi**. To operate MainQueue.vi, please follow instructions:

- Open and run **MainQueue.vi**.
- Go to **Pilatus tab**.
- The following sub-tabs appear: **Snap, Set threshold, Bad pixels.**
- **Snap tab** is a copy of **Pilatus_snap_main.vi.**
- **Set threshold tab** is a copy of **Pilatus_setthreshold_main.vi.**
- **Bad pixels tab** includes bad pixels of Pilatus detector.

IP address (147.213.112.19) and Port (41234) of Pilatus detector are set in S/N tab of MainQueue.vi.

6 Positioning of X-ray source – MainQueue.vi

Position of X-ray source (IμS, Incoatec) is controlled by two Newport motors: linear stage (GROUP1.POSITIONER) and rotation stage (GROUP2.POSITIONER) (Fig. 25). **Linear stage** moves X-ray source in vertical direction. Therefore, the name of linear stage is **LAMP VERTICAL**. Travel range of linear stage is 300 mm. **Rotation stage** tilts X-ray source and is responsible for adjustment of angle of incidence in GISAXS experiment on liquid surfaces. Therefore, the name of rotation stage is **LAMP ROTATION**.

TCP/IP communication settings of Newport motors can be found in **S/N tab** in **Newport box** of MainQueue.vi (Fig. 45):

- **IP address**: 147.213.112.49
- **User login**: Administrator
- **User password**: Administrator

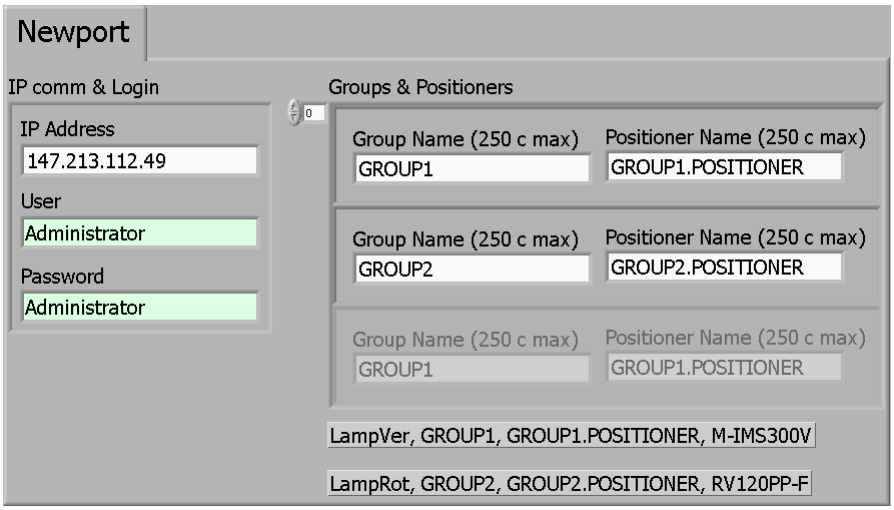


Figure 45. Newport settings in S/N tab of MainQueue.vi.

Linear stage is represented by GROUP1 and positioner name GROUP1.POSITIONER. Rotation stage is represented by GROUP2 and positioner name GROUP2.POSITIONER.

To operate linear stage, go to **Lamp tab → Lamp Vertical**. The current position of linear stage appears in **Current Position (mm) box** (Fig. 46).

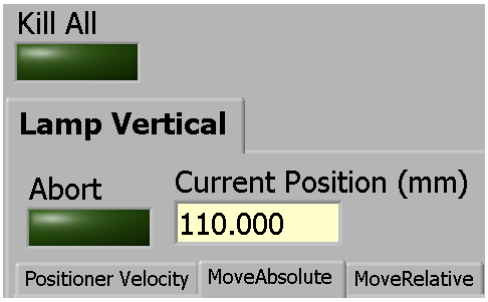


Figure 46. Current Position (mm) box in the Lamp Vertical tab.

To execute **absolute move** of **linear stage,** please follow instructions (Fig. 47a):

- Go to **MoveAbsolute tab**.
- Type absolute position into the **Move Abs box**.

- Press **Move Abs button.** It will execute absolute movement.

To execute **relative move** of **linear stage,** please follow instructions (Fig. 47b):

- Go to **Lamp tab → Lamp Rotation**.
- Go to **MoveRelative tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in positive direction, press **PLUS button**.
- To move in negative direction, press **MINUS button**.

To **set velocity** of **linear stage** follow instructions (Fig. 47c):

- Go to **Positioner Velocity tab**. Type new positioner velocity into the **Set Velocity box**. Typical velocity of linear stage is 5.
- To change current velocity, press **Set Vel button.**
- New velocity appears in the **Get Velocity box**.

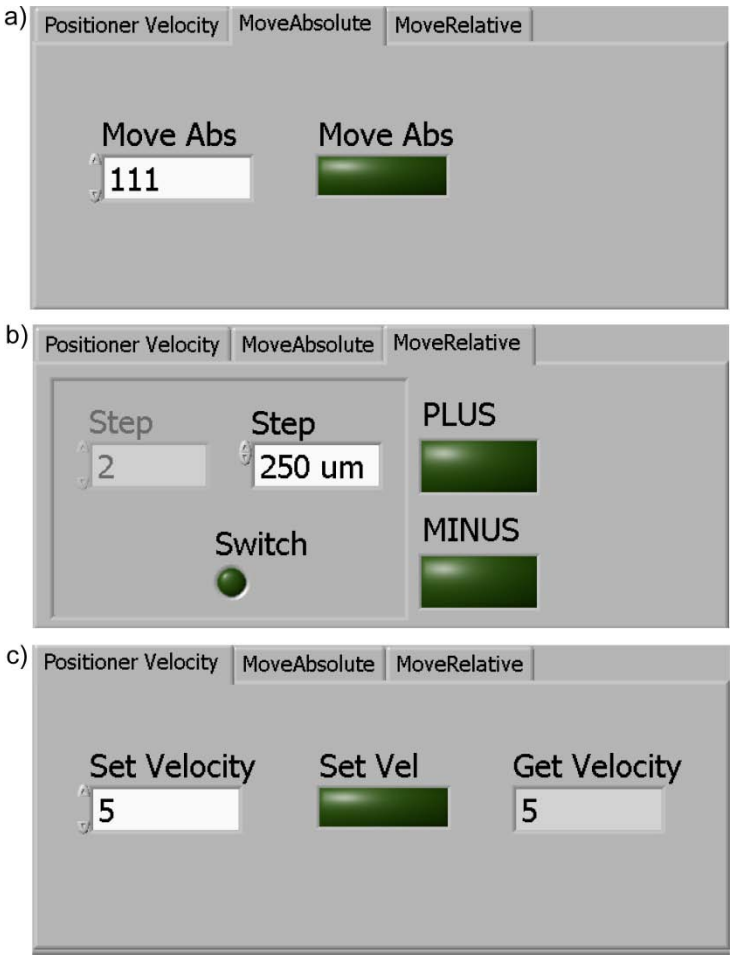


Figure 47. a) Move absolute tab . b) Move relative tab. c) Positioner Velocity tab of vertical stage.

To operate rotation stage go to **Lamp tab → Lamp Rotation**. The current position of rotation stage appears in **Current Position (deg) box** (Fig. 48).

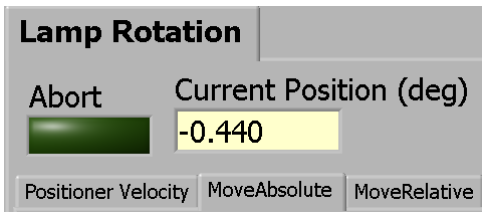


Figure 48. Current Position (deg) box in the Lamp rotation tab.

To execute **absolute move** of **rotation stage,** please follow instructions (Fig. 49a):

- Go to **MoveAbsolute tab**.
- Type absolute position into the **Move Abs box**.
- Press **Move Abs button.** It will execute absolute movement.

To execute **relative move** of **rotation stage,** please follow instructions (Fig. 49b):

- Go to **MoveRelative tab**.
- Set step of your scan in **Step** box. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode click on **Switch** button.
- To move in positive direction, press **PLUS button**.
- To move in negative direction, press **MINUS button**.

To **set velocity** of **rotation stage,** please follow instructions (Fig. 49c):

- Go to **Positioner Velocity tab**.
- Type new positioner velocity into the **Set Velocity box**. Typical velocity of rotation stage is 20.
- To change current velocity press **Set Vel button.**
- New velocity appears in the **Get Velocity box**.

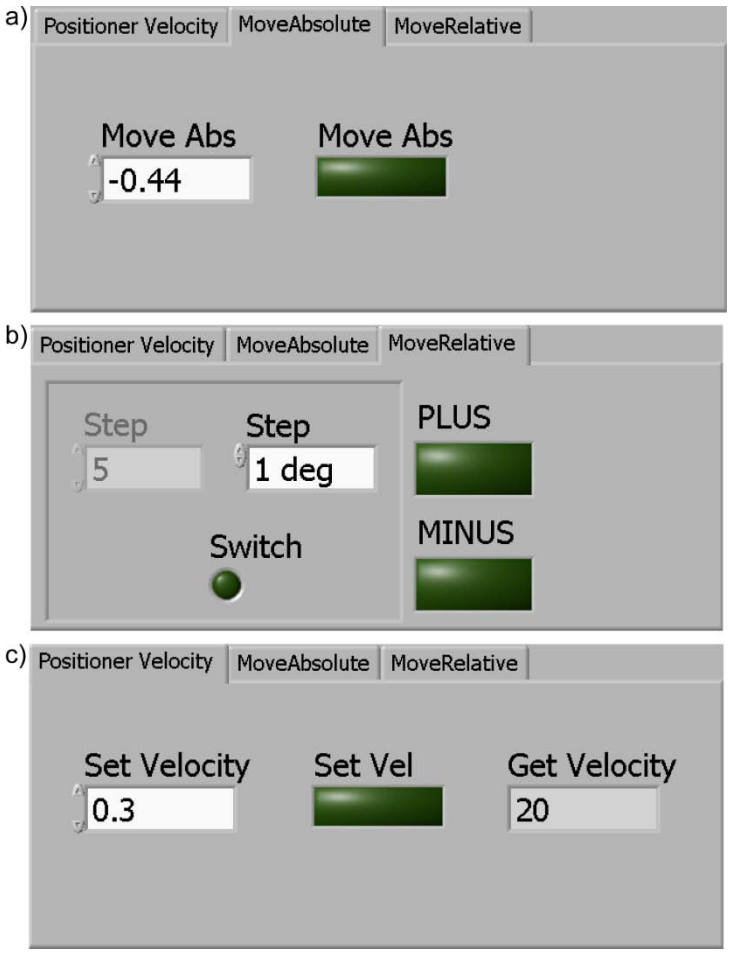


Figure 49. a) Move absolute tab . b) Move relative tab. c) Positioner Velocity tab of rotation stage.

If connection between XPS controller of Newport motors and GISAXS computer is broken, it is necessary to initialize and home motors, respectively. To do so please follow the instructions:

- Press **Kill button**. It will kill and reset the group (Fig. 50a).
- Press **Init/Home button**. It will initialize motor and move it into home position (Fig. 50b).

To kill and reset GROUP1.POISTIONER and GROUP2.POISTIONER please press **KILL ALL button** (Fig. 50c). To initialize and home motors please follow instructions above.

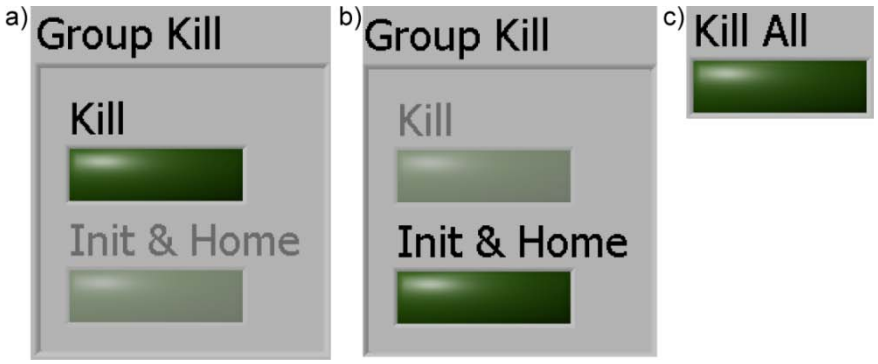


Figure 50. a) Enabled Kill button in Group Kill box. b) Enabled Init&Home button in Group Kill box. c) Kill All button.

7 Filter wheel – MainQueue.vi

The Filter Wheel allows you to attenuate or completely stop primary X-ray beam (Fig. 51). The attenuation of X-ray beam is necessary to avoid overexposure of pixels of X-ray detector. It is placed between exit Be window of X-ray source and the first external pinhole. It includes following attenuation modes: **Shutter, Air, Mo, 2Cu, Cu**. Shutter is made of lead foil that completely stops X-ray beam. Air is characterized by 100 % transmission coefficient. Mo filter is made of Mo foil with thickness of 25 μm. The transmission coefficient of Mo foil is ~2.2 %. 2Cu filter is made of two Cu sheets with total thickness of 50 μm. The transmission coefficient of 2Cu is ~10.5 %. The last filter is Cu filter with thickness of 25 μm. The transmission coefficient of Cu is 32.5 %.

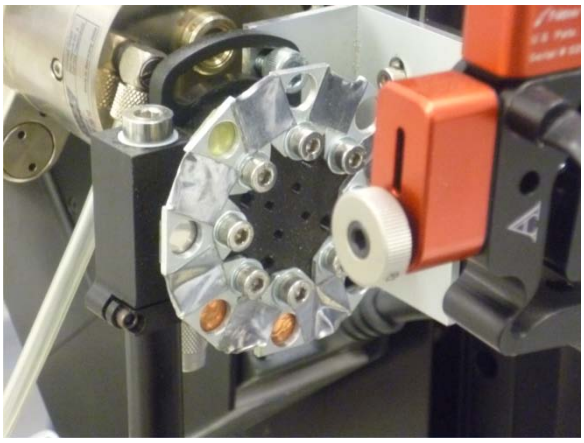


Figure 51. Photo of filter wheel of GISAXS setup. The filter wheel is in Air position.

To operate filter wheel in MainQueue.vi, you have to enable it in Thorlabs S/N array box (Fig. 52). Please follow instructions below:

- Go to **S/N tab** in MainQueue.vi.
- Go to **Thorlabs S/N array box**.
- Type 13 in numerical box of Thorlabs S/N array box**.**
- Input data of motor 13 are (Fig. 52):
  - name of motor: **Filter Wheel**
  - serial number (S/N) according to T-cube: **83823145**
- To make Filter wheel active, press **Active button**.

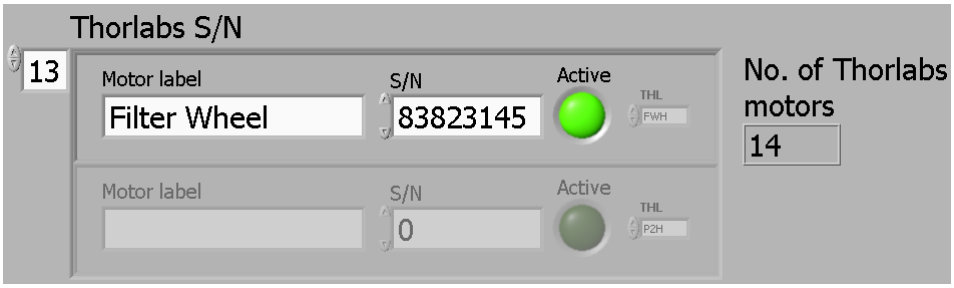


Figure 52. Image shows 13th position of Thorlabs S/N array box. The 13th position corresponds to the Filter Wheel motor.

The filters are placed on the rotation stage (Thorlabs, CR1/M-Z7). Following angles correspond to the different attenuation modes of filter wheel (Fig. 53):

- **Angle of 0 deg** corresponds to the **Shutter** (transmission coefficient 0 %).
- **Angle of 45 deg** corresponds to the **Air** (transmission coefficient 100 %).
- **Angle of 315 deg** corresponds to the **Mo** (transmission coefficient 2.2 %).
- **Angle of 270 deg** corresponds to the **2Cu** (transmission coefficient 10.5 %).
- **Angle of 225 deg** corresponds to the **Cu** (transmission coefficient 32.5 %).

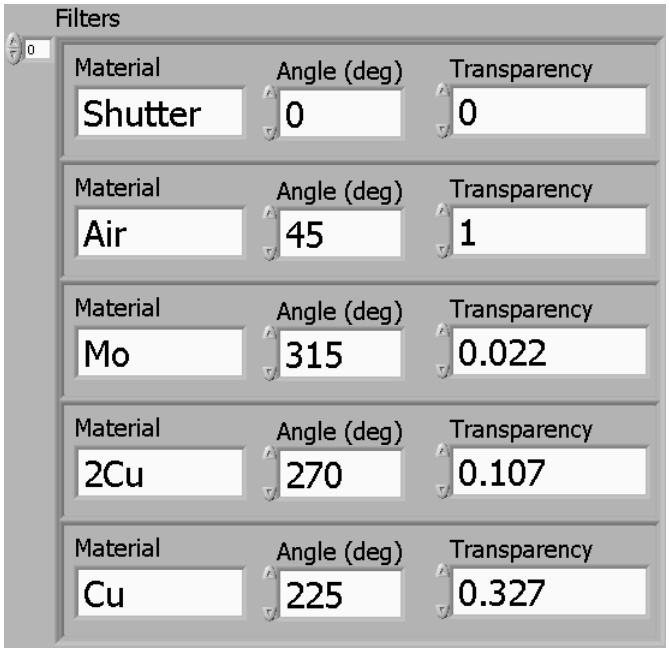


Figure 53. Image shows list of attenuation modes of filter wheel in Filter Wheel tab. Each attenuation mode corresponds to the specific angle of rotation stage CR1/M-Z7 and is characterized by its transmission coefficient.

To set filter please follow instructions (Fig. 54):

- Go to **Filter Wheel tab → Filters tab**.
- Select appropriate filter from **Choose Filter menu**.
- Press **Set Filter button**. It will set filter position.

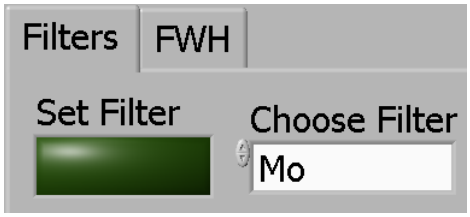


Figure 54. Image shows Choose Filter menu and Set Filter button in Filters tab.

If communication with filter wheel motor breaks, it is not possible to simply reset motor, home it and continue its use. It is due to the fact that motor has no home switch. The home function will change current position to zero position. In order to set Shutter to zero position, you have to manually move Shutter into the path of X-ray beam and then press Home button. In this way, Shutter position will be set to zero position and you can continue your work. Please be careful with X-ray radiation and always use shutter.

To zero current position please follow instructions (Fig. 55):

- Go to **Filter Wheel tab → FWH tab**.
- Move Shutter manually into the path of X-ray beam (Fig. 55a).
- Press **HOME button**. It will set current position to zero value (Fig. 55b).

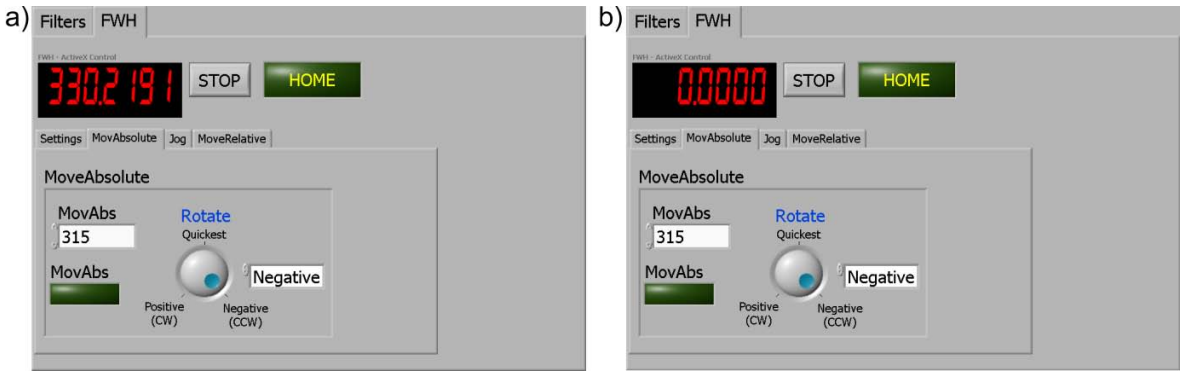


Figure 55. FWH tab a) before and b) after homing.

8 First external pinhole - MainQueue.vi

The position of the first external pinhole is controlled by **Newfocus picomotor (8051-M)** and **Picomotor Ethernet Controller (Model 8753)** (Fig. 34). TCP/IP communication settings of Newfocus picomotor can be found in **S/N tab** in the **Newfocus box** of MainQueue.vi (Fig. 56):

- **IP address**: 147.213.112.152
- **Port**: 23

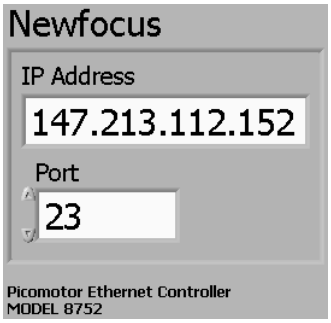


Figure 56. Newfocus box in S/N tab of MainQueue.vi.

Newfocus picomotor (8051-M) is dual-axis positioner that allows pinhole movement in horizontal and vertical direction.

To move first pinhole in **horizontal (X) direction,** please follow instructions:

- Go to **Pinhole 1 tab → P1X (MOT A) tab** (Fig. 57a).
- Select speed in **HI/LO SPEED menu**. You can select two speed modes: **RES COARSE** and **RES FINE**. RES COARSE is fast movement and RES FINE is slow movement. The frequency of RES COARSE is 2000 Hz. The frequency of RES FINE is 250 Hz.
- To move screw in positive or negative directions, press **PLUS/MINUS button**.

To move first pinhole in **vertical (Y) direction**, go to **Pinhole 1 tab → P1Y (MOT B) tab** (Fig. 57b). The instructions are identical with those for pinhole adjustment in horizontal direction.

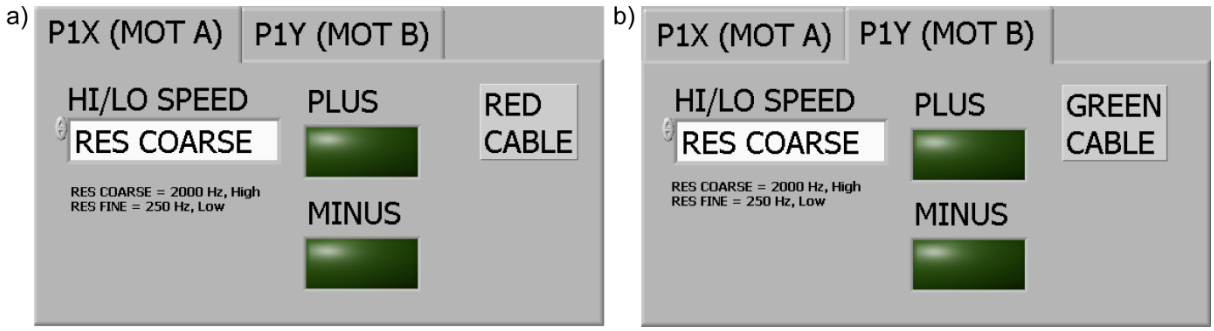


Figure 57. a) P1X (MOT A) tab allows pinhole movement in horizontal direction. b) P1Y (MOT B) tab allows pinhole movement in vertical direction.

The first external pinhole defines size of beam (Fig. 58). It can be round or square. The optional sizes of first external pinhole are 50 μm (round), 100 μm (round), 556 μm (round) and 441 μm (square) (Fig. 58). The pinholes were made of tungsten and were fabricated by laser-drilling technology.

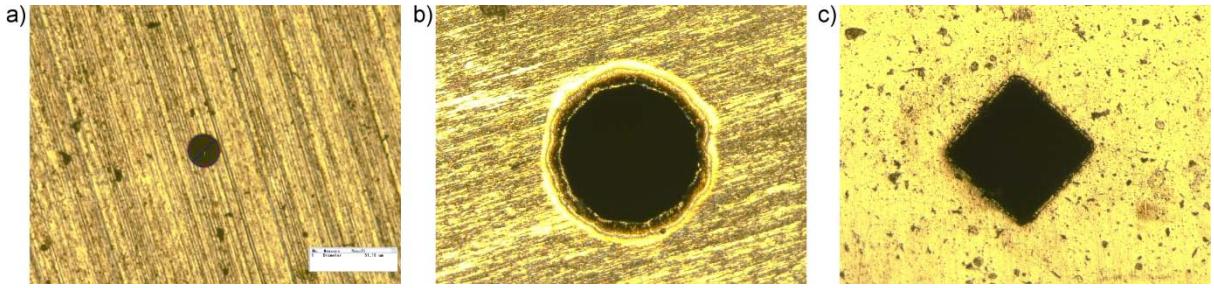


Figure 58. Various pinholes that can be inserted into Newfocus stage: round pinhole with diameter 51 μm, round pinhole with diameter 556 μm and c) square pinhole with size of the side 441 μm. The images were taken with magnifications: a) 500x, b) 200x and c) 200x.

The pinholes with different sizes can be glued on the top of aluminum tube (Fig. 59a-b) that can be inserted into the Newfocus stage (Fig. 60a). On the bottom of Newfocus stage, there is

1/16” hexagon screw. Please use 1/16” hexagon wrench to fix Pinhole tube in Newfocus stage (Fig. 60b).

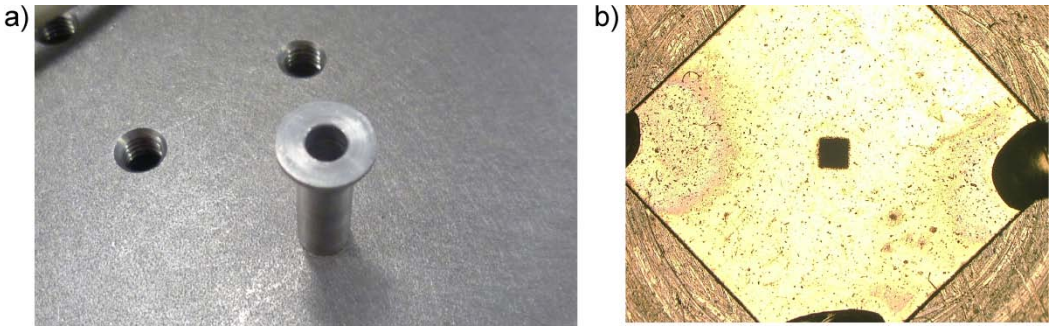


Figure 59. a) Tube without pinhole. b) Pinhole glued on the top of aluminum tube.

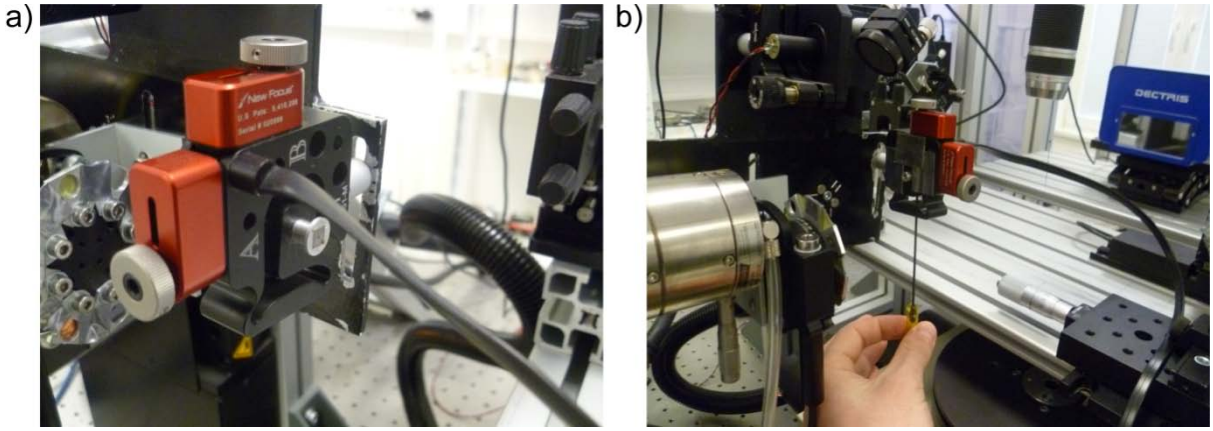


Figure 60. a) Insertion of Pinhole tube into the Newfocus stage. b) Fixation of Pinhole tube with 1/16” hexagon wrench.

9 The second external pinhole

The second external pinhole is placed immediately behind the first external pinhole (Fig. 61a). The pinhole has shape of rotated square with size of the side 1300 μm and serves as anti-scatter slit. The main function of anti-scatter slit is to stop air scattering. The second external pinhole is glued on the moveable lamella of optical element (SP40, OWIS) (Fig. 61a). SP 40 has four independently adjustable lamellas made of copper. The second external pinhole is attached to the XY positioning system that consists of two motorized stages (MTS25/M-Z8, Thorlabs) (Fig. 61b).

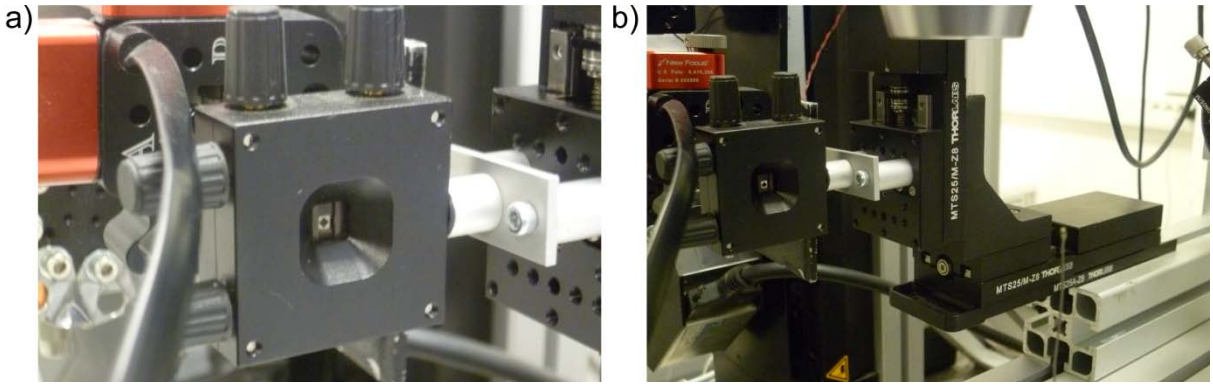


Figure 61. Images show a) second external pinhole glued on moveable copper lamella and b) its XY positioning system.

To operate the **second external pinhole** in MainQueue.vi, you have to enable:

- horizontal stage: **Pinhole 2-Hor** (P2H)
- vertical stage: **Pinhole 2-Ver** (P2V)

Please follow instructions below:

- Go to **S/N tab** in MainQueue.vi.
- Go to **Thorlabs S/N array box** (Fig. 62).
- Type 0 in numerical box of Thorlabs S/N array box**.**
- Input data of motors 0 and 1 appear.
- Input data of motor 0 are (Fig. 62):
  - name of motor: **Pinhole 2-Hor**
  - serial number (S/N) according to T-cube: **83825857**
- Input data of motor 1 are (Fig. 62):
  - name of motor: **Pinhole 2-Ver**
  - serial number (S/N) according to T-cube: **83825475**
- To make **Pinhole 2-Hor** and **Pinhole 2-Ver** active, press their **Active buttons**.

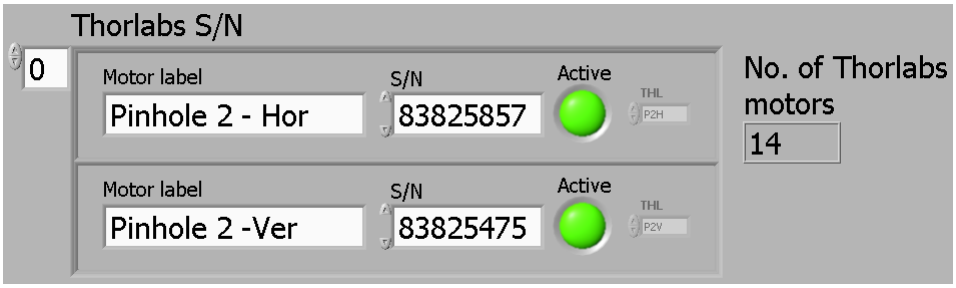


Figure 62. Image shows motor 0 (horizontal stage) and 1 (vertical stage) in Thorlabs S/N array box.

To execute absolute move of the second external pinhole in horizontal/vertical direction, please follow instructions below (Fig. 63a):

- Go to **Pinhole 2 tab → Horizontal/Vertical tab → MoveAbsolute tab**.
- Type new absolute position in **MovAbs box**.
- To execute absolute move, press **MovAbs button**.

To execute relative move/jog second external pinhole in horizontal/vertical direction, please follow instructions below (Fig. 63b-c):

- Go to **Pinhole 2 tab → Horizontal/Vertical tab → MoveRelative/Jog tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in positive direction, press **PLUS button**.
- To move in negative direction, press **MINUS button**.

To stop move of your motors, press **STOP button** (Fig. 63).

To home motors, press **HOME button** (Fig. 63).

Figure 63. Tab of the second external pinhole in MainQueue.vi enables to execute a) absolute, b) relative moves and c) jog.

10 Sample holder

Sample holder consists of rotation, vertical and horizontal stages. The horizontal stage is optional, is not included in standard solution and allows sample movement in horizontal direction. The sample holder motors are Thorlabs motors and includes:

- vertical stage (MTS25/M-Z8)
- rotation stage (PRM1/M-Z8)
- horizontal stage (optional, MTS25/M-Z8)

To rotate sample and move sample in vertical and horizontal directions, you have to enable:

- rotation stage: **Solid Sample-Rot** (SSR)
- vertical stage: **Solid Sample-Ver** (SSV)
- horizontal stage: **Solid Sample-Hor** (SSH)

Please follow instructions below:

- Go to **S/N tab** in MainQueue.vi.
- Go to **Thorlabs S/N array box** (Fig. 64).
- Type 2 in numerical box of Thorlabs S/N array box**.**
- Input data of motors 2 and 3 appear.
- Input data of motor 2 are (Fig. 64):
    - name of motor: **Solid Sample - Rot**
    - serial number (S/N) according to T-cube: **83825449**
- Input data of motor 3 are (Fig. 64):
    - name of motor: **Solid Sample – Ver**
    - serial number (S/N) according to T-cube: **83812451**
- Input data of motor 4 are (Fig. 64):
    - name of motor: **Solid Sample – Hor**
    - serial number (S/N) according to T-cube: **not used**
- To make motors active, press their **Active buttons**.

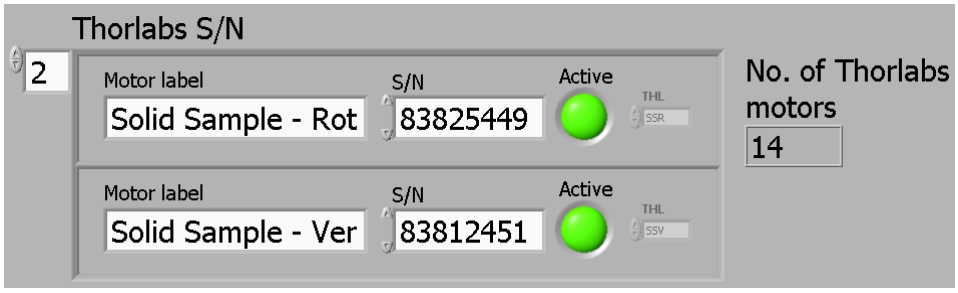


Figure 64. Image shows motor 2 (rotation stage) and 3 (vertical stage) in Thorlabs S/N array box.

To execute absolute move of the rotation stage, please follow instructions below (Fig. 65a):

- Go to **Solid Sample tab → SSR tab → MoveAbsolute tab**.
- Select **clockwise (CW)** or **counter clockwise (CCW)** direction of movement.
- Type new absolute position in **MovAbs box**.
- To execute absolute move, press **MovAbs button**.

To execute relative move/jog of the rotation stage, please follow instructions below (Fig. 65b-c):

- Go to **Solid Sample tab → SSR tab → MoveRelative tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in clockwise direction, press **PLUS (CW) button**.
- To move in counter clockwise direction, press **MINUS (CCW) button**.

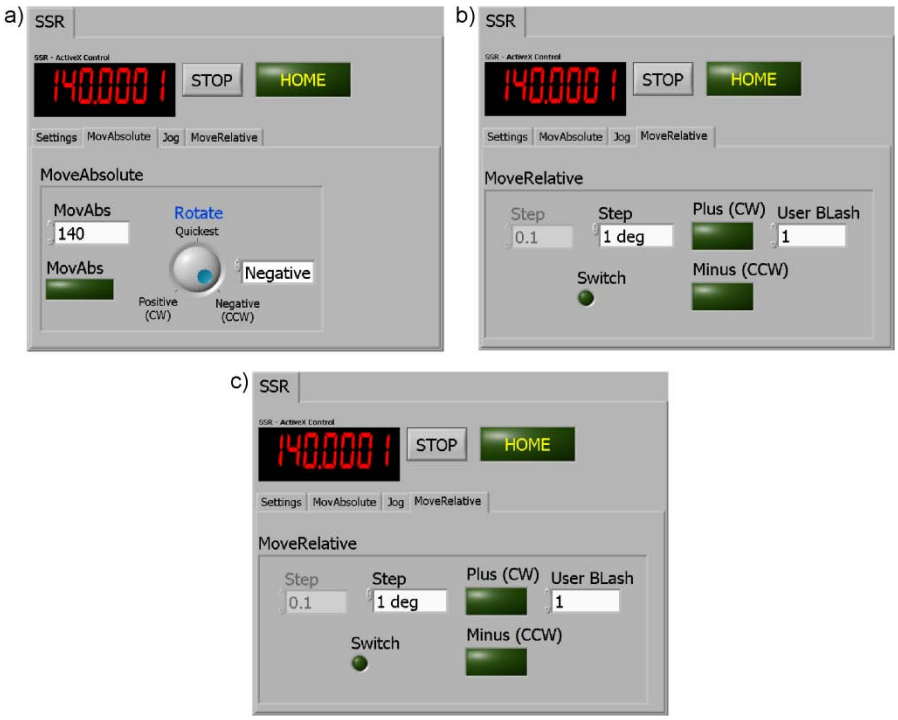


Figure 65. Tab of the rotation stage in MainQueue.vi enables to execute a) absolute, b) relative moves and c) jog.

To execute absolute move of the sample in vertical/horizontal direction, please follow instructions below (Fig. 66a):

- Go to **Solid Sample tab → SSV/SSH tab → MoveAbsolute tab**.
- Type new absolute position in **MovAbs box**.
- To execute absolute move, press **MovAbs button**.

To execute relative move/jog of the sample in horizontal/vertical direction, please follow instructions below (Fig. 66b-c):

- Go to **Solid Sample tab → SSV/SSH tab → MoveRelative tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in positive direction, press **PLUS button**.

- To move in negative direction, press **MINUS button**.

To stop move of your motors, press **STOP button** (Figs. 65-66).

To home motors, press **HOME button** (Fig. 65-66).

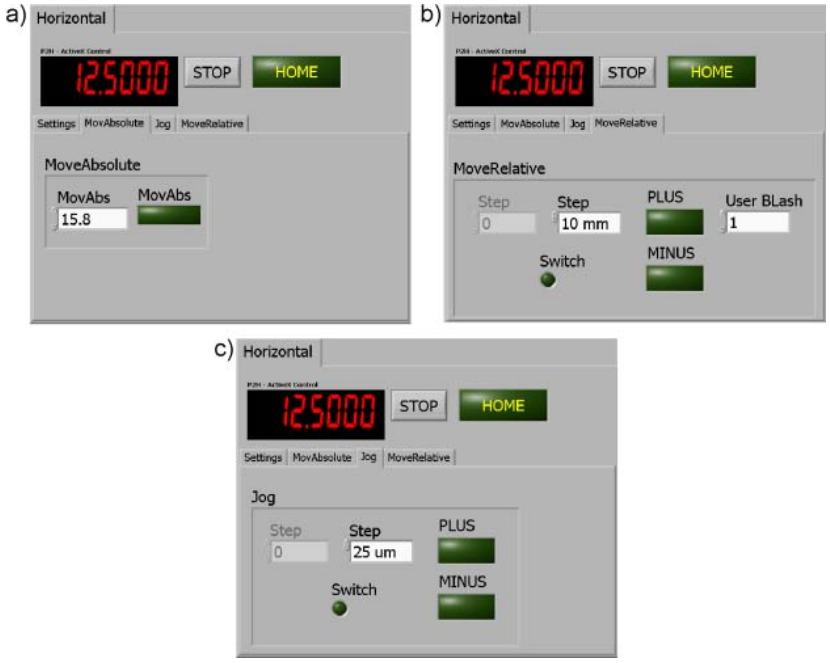


Figure 66. Tab of the vertical stage in MainQueue.vi enables to execute a) absolute, b) relative moves and c) jog.

The sample movement in horizontal direction is not standard move and **Solid Sample – Hor** motor is not standard used. Enabling of SSH motor will lead to activation of **SSH Scan tab** (Fig. 67). The **SSH Scan tab** allows you to scan sample in horizontal direction. To scan sample in horizontal direction, please follow instructions.

- Align your sample with rocking curve algorithm (**Rocking Curve tab**). It will set zero angle of incidence.
- Set appropriate **angle of incidence**. Go to **Solid Sample tab → MoveRelative tab**. Press **Switch button**. Type angle of incidence in **Step box**. Press **Minus(CW) button**.

- Type new folder path into the **Folder Path box**. If folder already exists, the scan will not be executed.
- Set number of images, you want to expose in sequence for one horizontal position. Type number of images into the **N images box**.
- Set exposure time in **Exp Time (sec) box** for one image.
- Set exposure period in **Exp Period (sec) box** for one image.
- Set scanning range in horizontal direction. Use **From (-)** and **To (+)** boxes. The scanning goes from smaller to larger value.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- Once the scanning range and step of your scan are set, number of steps appears in **No. of Steps box**.
- Press **Start Scan button**. It will start scanning procedure in horizontal direction. Indicator **SSH Scan** is turned on and lights. The initial position of **SSH motor** is entered into the **Initial Pos box**. The current position of **SSH motor** is entered into the **Current Pos box**.
- Once the scanning procedure is finished. Indicator **SSH Scan** is turned off.
- The images are copied to the path, you defined in the **Folder Path box**.

To stop scanning procedure, press **STOP button**.

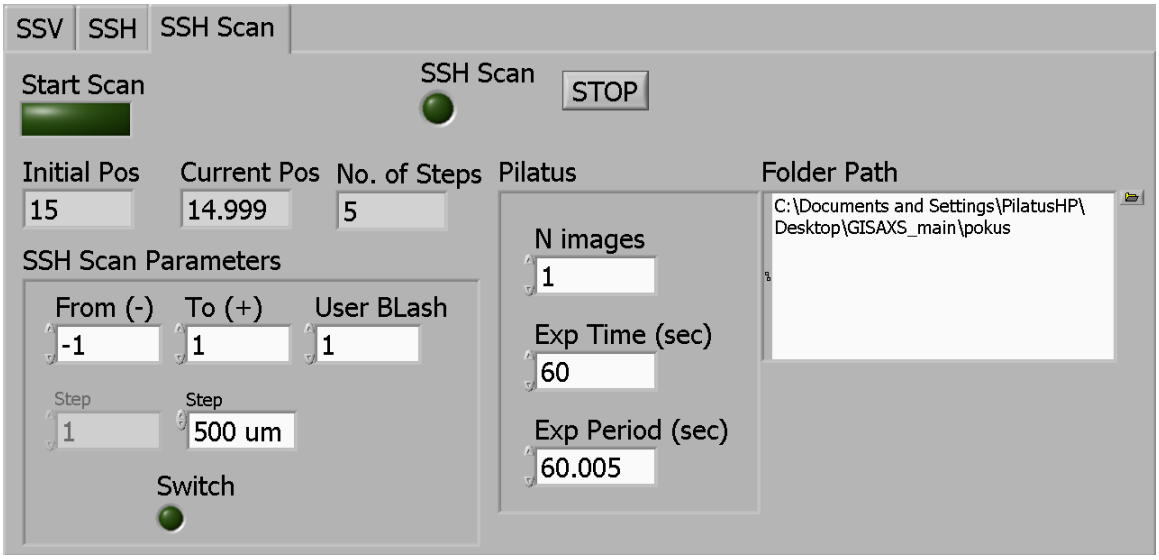


Figure 67. SSH scan tab of MainQueue.vi.

11 Sample alignment - MainQueue.vi

In the first step, go to **Filter Wheel tab** and select **Mo filter** from **Choose Filter** menu to attenuate X-ray primary beam. Before you proceed to the **Rocking Curve** tab, make sure that your primary beam has an entirely free path toward the detector (e.g. is not shaded by your sample). It can be done by laser pre-aligned into the direction of X-ray beam. In order to fix it, go to **Solid Sample tab → SSV tab** and move your sample in vertical direction to uncover X-ray beam.

In the next step, go to **Rocking Curve tab** (Fig. 68). Place your mouse cursor on **Get One Image box**. Type exposure time in seconds into to the **Expt (sec) box** (Fig. 68). Recommended exposure time for **Mo filter** is **1 s**. Then press **Get button** (Fig. 68). It will cause that following set of commands will be sent into the Pilatus detector:

- ni 1 (do one image)
- expt **X** (it will set exposure time to value entered into the **Expt (sec) box**)
- expp **X**+0.005 (exposure period will be automatically set to value 5 ms larger than exposure time)

- expo RIC_imag.tif (it will take image with specified name)

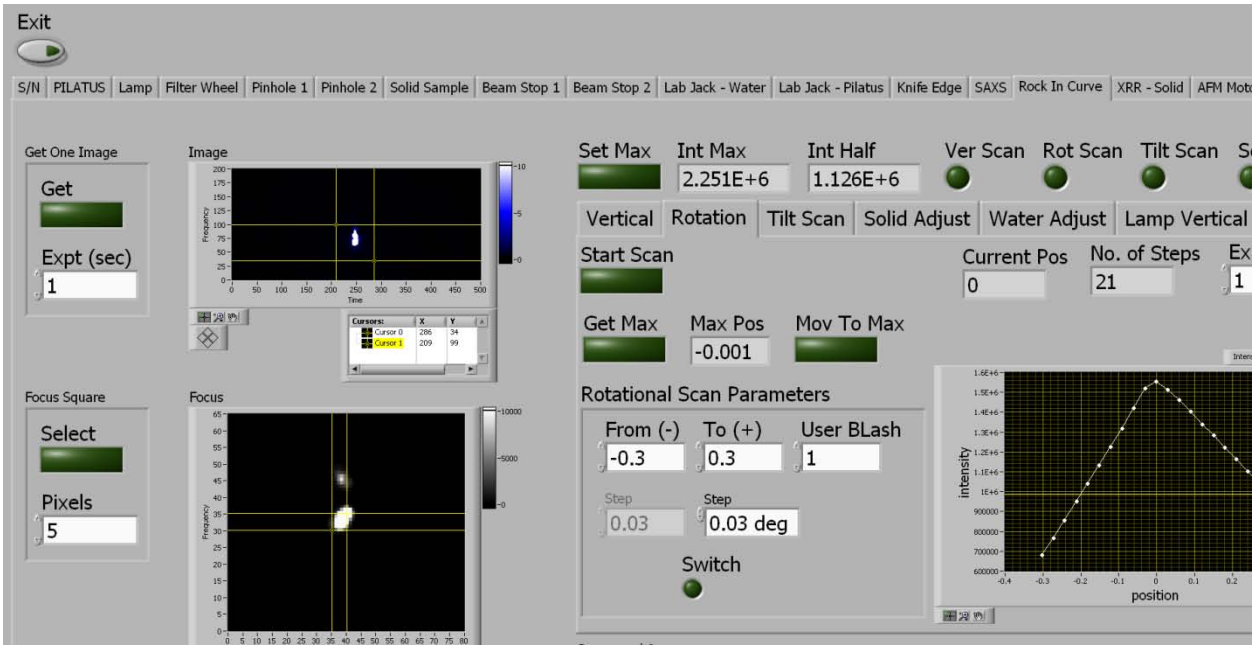


Figure 68. Rocking Curve tab in Main Queue.vi

The new image appears in the **Image window** (Fig 69). Zoom in your region of interest by horizontal and vertical **yellow cursors**. The zoom image appears in the **Focus window** (Fig. 70).

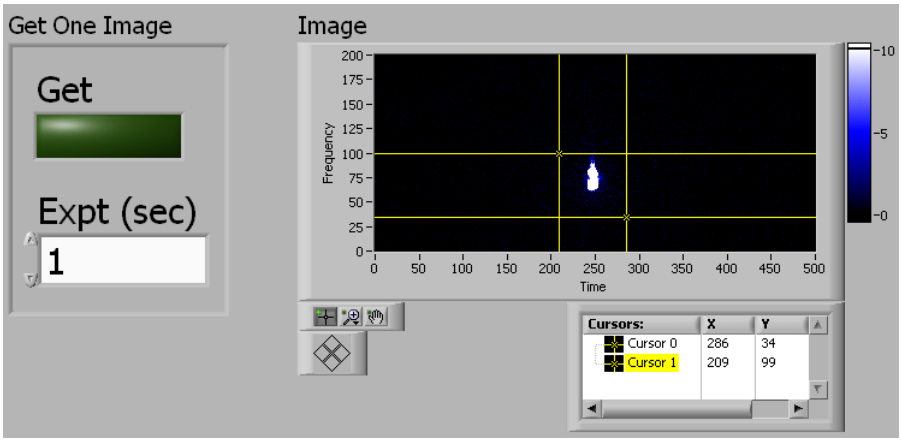


Figure 69. Image window in Main Queue.vi

Type number of pixels into the **Pixels box** (Fig. 70). In this way, you will adjust region of interest in the **Focus window** (Fig. 70). The region of interest has a square shape and is bounded by yellow cursors. You can use the mouse to drag yellow square to select **Integration Area** of the primary X-ray beam. Then press **Select button** (Fig. 70)**.** It will define borders of your **Integration Area**. The maximum intensity in pixel is shown in the **Int Max box** (Fig. 70).

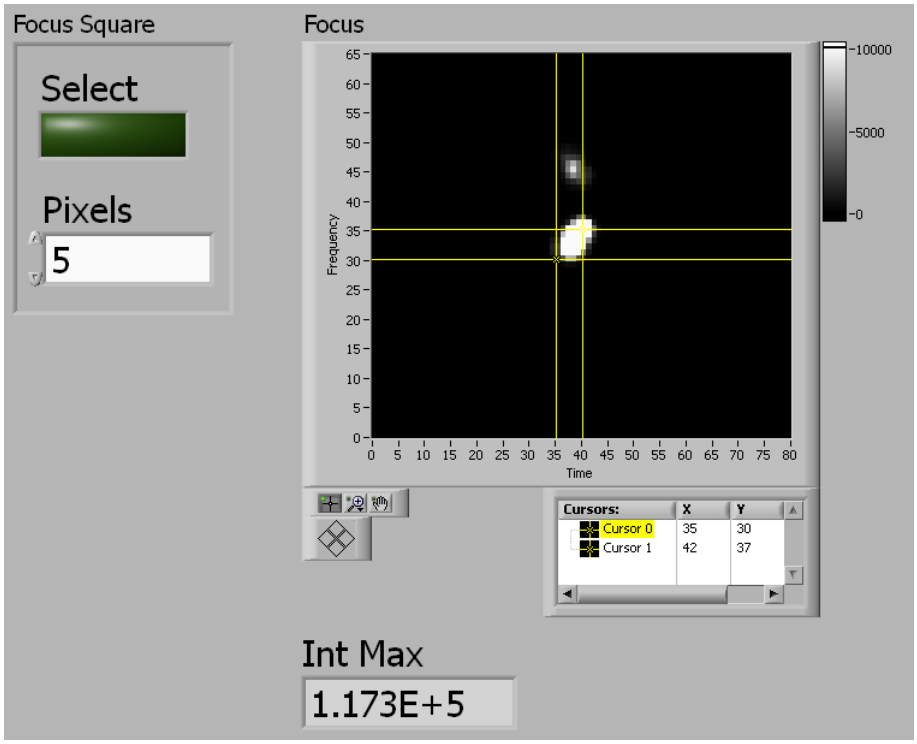


Figure 70. Focus window and Int Max box of MainQueue.vi.

Press **Set Max button** in **Rocking Curve tab** (Fig. 71). It will lead to integration of your region of interest (yellow square) in the **Focus window** (Fig. 70). The **total intensity** in the yellow square appears in the **Int Max box** placed next to **Set Max button** (Fig. 71). The half of the total intensity appears in the **Int Half box** placed next to **Int Max box** (Fig. 71). We are interested in half of the total intensity which is important for vertical alignment of your sample.

The alignment procedure described below is also known as **Rocking Curve.** Rocking curve algorithm contains vertical and rotational alignment of your sample. At the end of rocking curve procedure, you will find zero angle of incidence.

Before applying rocking curve, please follow instructions:

- Move your sample to position in which it will cover primary X-ray beam. Go to **Solid Sample tab** and use **SSV motor**.
- Move your sample to zero position just by the eye by means of the pre-aligned laser. Go to **Solid Sample tab** and use **SSR motor**.
- Make sure that **Hardware Limit Switches** of SSR motor are set to **HWLIMSW_IGNORE** value.

The **vertical alignment** of the sample includes following steps (Fig. 71):

- Go to **Rocking Curve tab → Vertical tab**.
- Set scanning range in vertical direction. Use **From (-)** and **To (+)** boxes. The scanning goes from **down** (larger value) to **up** (smaller value).
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- Once the scanning range and step of your scan are set, number of steps automatically appears in the **No. of Steps box**.
- Type exposure time for each image in **Expt (sec) box**. Recommended value is 1 s.
- Press **Start Scan button**. It will start scanning procedure in vertical direction. Indicator **Ver Scan** is turned on and lights. The initial position of **SSV motor** is entered into the **Initial Pos box**. The scanning procedure can be seen on the fly in the **Graph 1** and **Graph 2**. **Graph 1** shows intensity as a function of motor position. **Graph 2** shows $\mathbf{1/(I-I_{half})^2}$ as a function of motor position where **I** is the actual measured intensity and $\mathbf{I_{half}}$ is the half of the total intensity of the primary X-ray beam.
- Once the scanning procedure is finished. Indicator **Ver Scan** is turned off.

- Press **Get Half button**. The position appears in **Half Pos box** in mm. In **Half Pos**, the absolute value of intensity difference between measured intensity (**I**) and half of the total intensity ($\mathbf{I_{half}}$) is minimal. Contrary to this, $\mathbf{1/(I-I_{half})^2}$ has maximum in **Half Pos.**
- Press **Mov To Half**. The vertical motor (SSV motor) moves to **Half Pos.** New position appears in the **Current Pos box**.

Caution: **User BLash box** includes preselected value **1 mm**. This value compensates motor backlash in one direction (up→down).

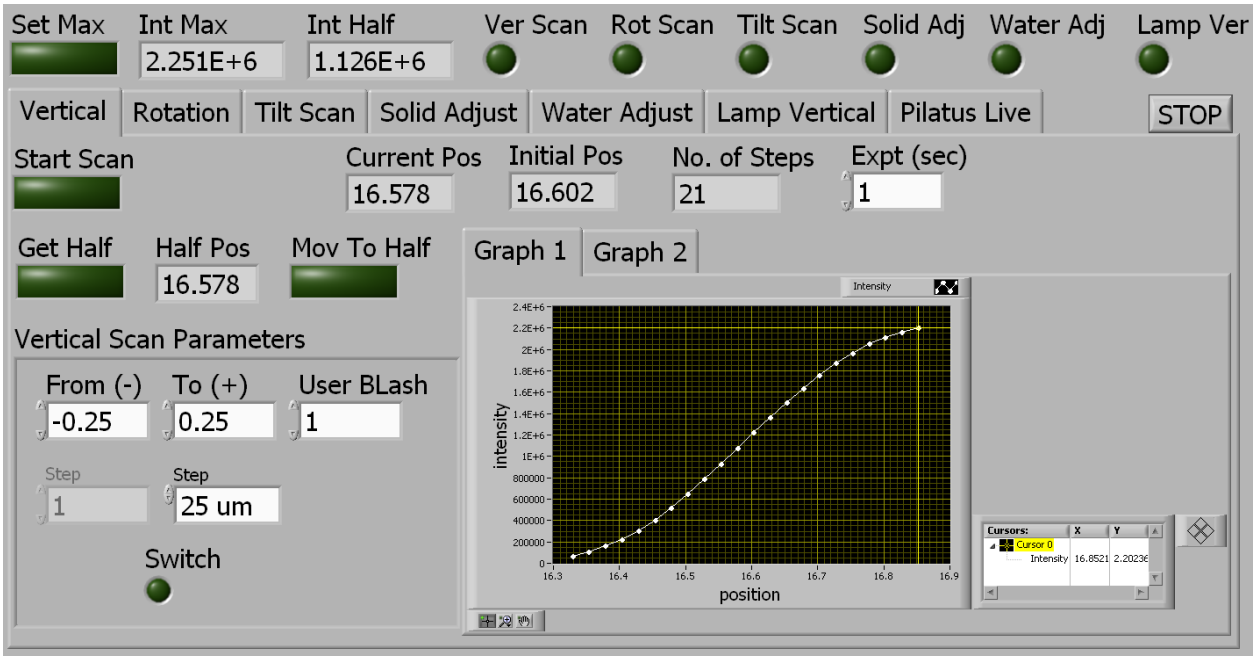


Figure 71. Vertical tab in Rocking Curve tab of MainQueue.vi.

The **rotation alignment** of the sample includes following steps (Fig. 72):

- Go to **Rocking Curve tab → Rotation tab**.
- Set scanning range. Use **From (-)** and **To (+) boxes**. The scanning goes from smaller to larger value (left→right).
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.

- Once the scanning range and step of your scan are set, number of steps appears in the **No. of Steps box**.
- Type exposure time for each image in **Expt (sec) box**. Recommended value is 1 s.
- Press **Start Scan button**. It will start rotational scanning procedure. Indicator **Rot Scan** is turned on and lights. The initial position of SSR motor is set to zero value. The scanning procedure can be seen on the fly in the **Graph**. **Graph** shows intensity as a function of the motor position.
- Once the scanning procedure is finished. Indicator **Rot Scan** is turned off. Press **Get Max button**. The position appears in **Max Pos box** in deg. In **Max Pos**, the measured intensity reached maximum value.
- Press **Mov To Max**. The rotation motor (**SSR motor**) moves to **Max Pos.** New position appears in the **Current Pos box** and is immediately set to zero.

Caution: **User BLash** box includes preselected value **1 deg**. This value compensates motor backlash in one direction (right→left, detector→source).

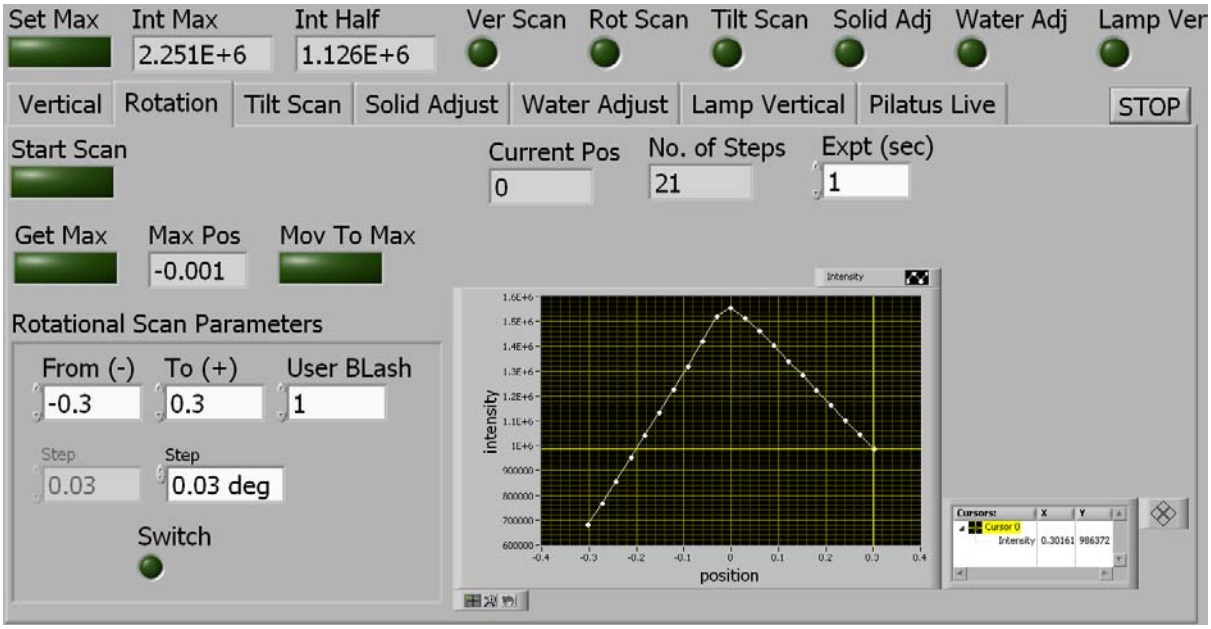


Figure 72. Rotation tab in Rocking Curve tab of MainQueue.vi.

Rocking curve procedure is standard done in three steps:

1-st step:

- From (-)= -1 mm; To (+)= 1 mm; Step=0.1 mm, No. of Steps=21
- From (-)= -1 deg; To (+)= 1 deg; Step=0.1 deg, No. of Steps=21

2-nd step:

- From (-)= -0.5 mm; To (+)= 0.5 mm; Step=0.05 mm, No. of Steps=21
- From (-)= -0.5 deg; To (+)= 0.5 deg; Step=0.05 deg, No. of Steps=21

3-rd step:

- From (-)=-0.25 mm; To (+)= 0.25 mm; Step=0.025 mm, No. of Steps=21
- From (-)=-0.3 deg; To (+)= 0.3 deg; Step=0.03 deg, No. of Steps=21

Vertical motor (SSV, Solid Sample Vertical):

- Travel Range: 25 mm
- Bidirectional Repeatability: 1.6 μm.

Rotation motor (SSR, Solid Sample Rotation):

- Bidirectional Repeatability: ±0.1 deg.
- Repeatable Incremental Motion (Max)=0.03 deg.

To set angle of incidence, please follow instructions below (Fig. 73):

- Go to **Solid Sample tab → SSR tab**.
- Go to **Move Relative tab.**

- Light **Switch button**.
- Type **angle of incidence** into the **Step box**.
- Press **Minus button**. Remember angle of incidence is increased in the minus direction. In the plus direction, angle of incidence is decreased.
- **SSR motor** will move to negative direction and angle of incidence will be set.

For example: Set angle of incidence to 0.3 deg. It means that you will set angle 360–0.3 deg=359.7 deg.

Figure 73. SSR tab in Solid Sample tab of MainQueue.vi.

12 Additional scanning procedures

The Rocking Curve tab includes Tilt Scan, Solid Adjust, Water Adjust, Lamp Vertical and Pilatus Live tab.

Tilt Scan allows you to scan tilt of sample. The scanning is performed by SSH motor. Please enable SSH motor in Thorlabs S/N array box.

Solid Adjust allows you to find maximum reflected intensity from solid surface. The scanning is performed by SSV motor.

Water Adjust allows you to find maximum reflected intensity from water surface. The scanning is performed by Lab Jack-Water motor.

Lamp Vertical allows you to find maximum reflected intensity from both solid and liquid surfaces. The scanning procedure is performed by Lamp Vertical motor where X-ray source is mounted.

Pilatus live shows image taken during scanning procedure.

13 Beamstop

The MainQueue.vi includes two beamstops: **Beam Stop 1** and **Beam Stop 2**. The both beamstops are used only in the case if evacuated flight tube is inserted between sample and detector. The flight tube is evacuated by diaphragm pump to 5 mbar. The input and output windows of flight tube are made of mylar foil. The first beamstop is placed before input mylar window of evacuated flight tube. It is important as it decreases scattering from the mylar foil. The first beamstop should be used carefully as distance between sample and beamstop is too short.

Standard, GISAXS setup works without evacuated flight tube and only one beamstop (**Beam Stop 1**) is used. To operate the **Beam Stop 1** in MainQueue.vi, you have to enable:

- horizontal stage: **Beam Stop 1-Hor** (BS1H)
- vertical stage: **Beam Stop 1-Ver** (BS1V)

Please follow instructions below:

- Go to **S/N tab** in MainQueue.vi.
- Go to **Thorlabs S/N array box** (Fig. 74).
- Type 5 in numerical box of Thorlabs S/N array box**.**
- Input data of motors 5 and 6 appear.
- Input data of motor 5 are (Fig. 74):

- name of motor: **Beam Stop 1-Hor**
- serial number (S/N) according to T-cube: **83820632**

- Input data of motor 6 are (Fig. 74):
  - name of motor: **Beam Stop 1-Ver**
  - serial number (S/N) according to T-cube: **83823542**
- To make **Beam Stop 1-Hor** and **Beam Stop 2-Ver** active, press their **Active buttons**.

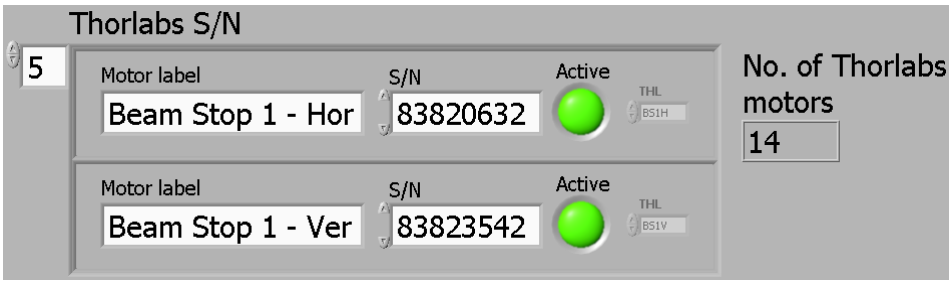


Figure 74. Image shows motor 5 (horizontal stage) and 6 (vertical stage) in Thorlabs S/N array box.

To execute absolute move of the beamstop in horizontal/vertical direction, please follow instructions below (Fig. 75a):

- Go to **Beam Stop 1 tab → Horizontal/Vertical tab → MoveAbsolute tab**.
- Type new absolute position in **MovAbs box**.
- To execute absolute move, press **MovAbs button**.

To execute relative move/jog second external pinhole in horizontal/vertical direction, please follow instructions below (Fig. 75b-c):

- Go to **Beam Stop 1 tab → Horizontal/Vertical tab → MoveRelative/Jog tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in positive direction, press **PLUS button**.
- To move in negative direction, press **MINUS button**.

To stop move of your motors, press **STOP button** (Fig. 75).

To home motors, press **HOME button** (Fig. 75).

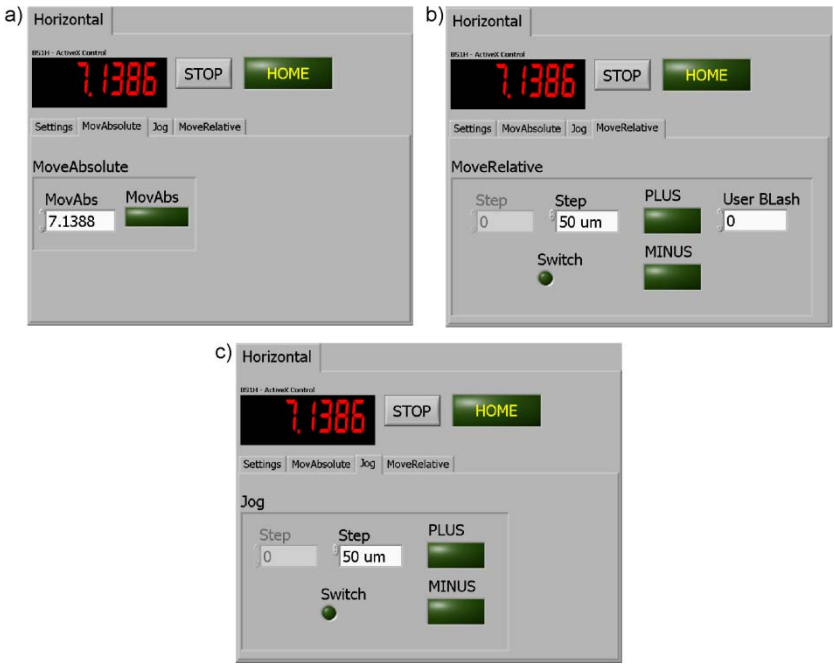


Figure 75. Tab of the bemastop in MainQueue.vi enables to execute a) absolute, b) relative moves and c) jog.

14 Motorized detector movement

To move 2D X-ray detector in vertical direction, you can use LabJack motor (L490MZ/M, Thorlabs). The travel range of LabJack is 50 mm.

Please follow instructions below:

- Go to **S/N tab** in MainQueue.vi.
- Go to **Thorlabs S/N array box** (Fig. 76).
- Type 10 in numerical box of Thorlabs S/N array box**.**
- Input data of motor 10 appears.
- Input data of motor 10 are (Fig. 76):

- name of motor: **Lab Jack-Pilatus**
- serial number (S/N) according to T-cube: **46824880**

- To make **Lab Jack-Pilatus** active, press **Active button**.

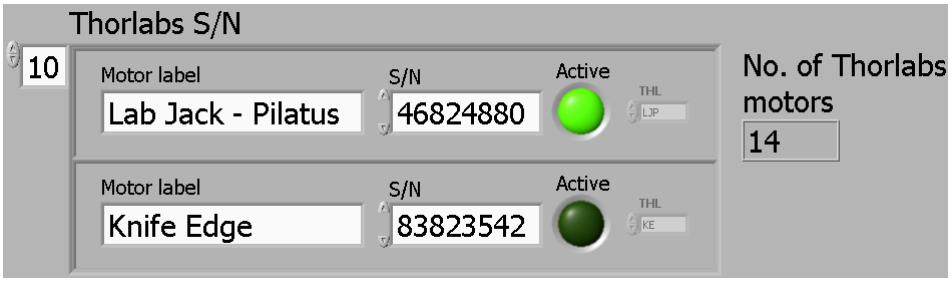


Figure 76. Image shows motor 10 in Thorlabs S/N array box.

To execute absolute move of the LabJack in vertical direction, please follow instructions below (Fig. 77a):

- Go to **LabJack-Pilatus tab → LJP tab → MoveAbsolute tab**.
- Type new absolute position in **MovAbs box**.
- To execute absolute move, press **MovAbs button**.

To execute relative move/jog second external pinhole in horizontal/vertical direction, please follow instructions below (Fig. 77b-c):

- Go to **LabJack-Pilatus tab → LJP tab → MoveRelative/Jog tab**.
- Set step of your scan in **Step box**. The value can be entered manually or you can use preselected values. To switch between manual and preselected mode, press **Switch button**.
- To move in positive direction, press **PLUS button**.
- To move in negative direction, press **MINUS button**.

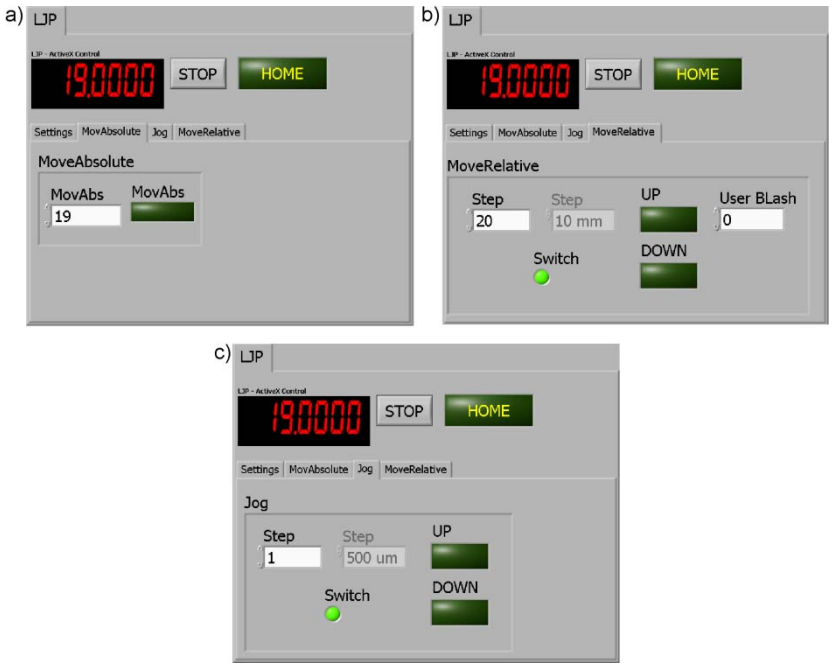


Figure 77. Tab of the LabJack-Water in MainQueue.vi enables to execute a) absolute, b) relative moves and c) jog.

15 Pre-aligned laser beam

Laser beam pre-aligned in the direction of X-ray beam allows you to visualize X-ray path toward sample. The laser system consists of laser diode (CPS196, Thorlabs), protected silver mirror (PF10-03-P01, Thorlabs) and pellicle beam-splitter (BP145B1, Thorlabs) (Fig. 78a). The beamsplitter is placed on magnetically coupled mounting base which enables its removing before the starting of measurement (Fig. 78b).

The laser has to be aligned to two points. The first point is given by position of the first external pinhole (Fig. 79a) and the second point is given by position of the beamstop (Fig. 79c). To align laser beam into the path of X-ray beam, please use knobs on the laser, mirror and beamsplitter holder (Fig. 78a).

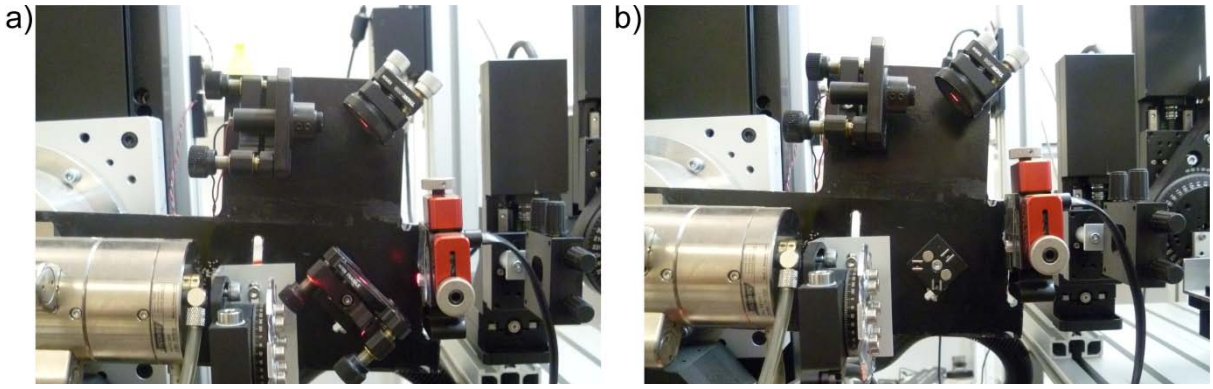


Figure 78. a) Laser point adjustment system includes focus laser diode, mirror and removable beamsplitter. b) Laser system without beamsplitter.

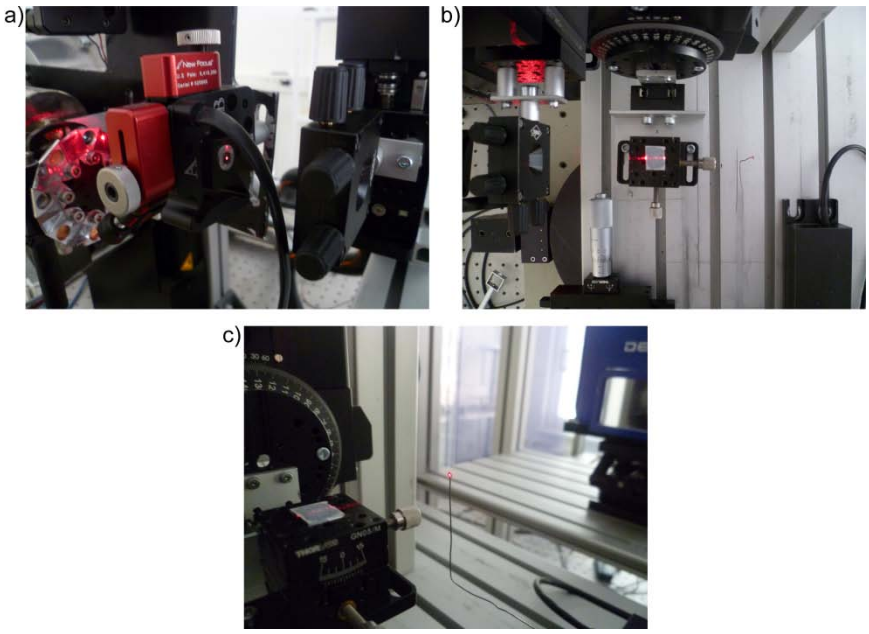


Figure 79. a) Laser beam pass through the first external pinhole. b) Laser spot on the sample surface. c) Laser spot on the beamstop.

16 SAXS sample holder

SAXS sample holder consists of translation stage with micrometer screw (PT1/M, Thorlabs) (Fig. 80a), dowetail rails (RLA075/M and RLA150/M, Thorlabs) (Fig. 80a) and sample holder (Fig. 80b). Translation stage allows to translate sample holder with glass capillary into

the path of X-ray beam. Sample holder has five insertion apertures designed for capillaries with outside-diameter 0.7 mm (Fig. 80c).

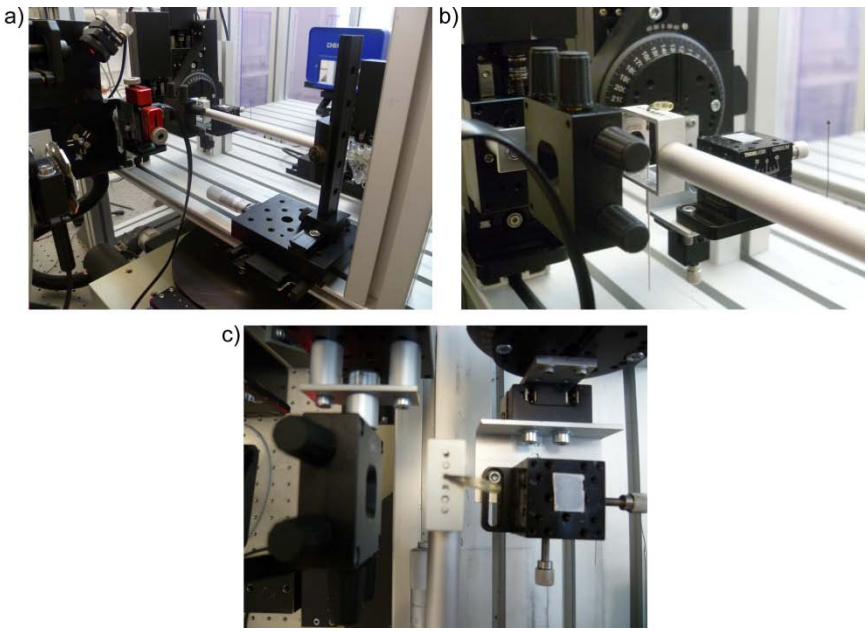


Figure 80. a) Photo of manual SAXS system. b) Glass capillary with powder sample in SAXS sample holder. c) Top view of sample holder with 5 insertion apertures.