# **STARTING THE X-RAY**

# **SETUP**



How to start the TWO PC'S: Pilatus computer : 129.175.80.133 (access via the network) Images are saved in Images(\\129.175.80.133) P: Use the subfolder : 'PetitsAngles'

Session name : Detector login: det pswd: Pilatus2

Open a command window type : cd p2\_det Type : camonly



'ON' button is hidden here behind the black security trap !



PC for acquisition : SAXS-Cu (RIX-MW7110)

Password : aeiouy100%

#### - Start 'Autosampler' for acquisition



## **PILATUS detector, STEPS TO FOLLOW:**

- Check for the pressure in the compass.
  Pressure = 2 bars (dry air circulation).
- 1. Start the two PCs
- Wait for at least two hours with the detector OFF to ensure dry air inside.



 Turn on the PILATUS detector, ensure that it shows green light. Check for the humidity rate on the server using the 'THread' command. If the rate is still too high (> 2%), turn off the detector again and wait one more

hour before restarting the detector.







# **STARTING THE X-RAY**

SHUTTER

**SECTION** 



Turn ON the 'power' button on this box To turn On the X-RAY :

Turn 'IG ON' on the vacuum controller panel located behind the generator Wait for OPERATE to turn orange on the front vacuum panel.

(Vacuum system is assumed to be ready with the TMP pump running)

# ensure that the reading for the voltage of vacuum should be less than .090V

Press the ON switch for the TARGET (rotation of the anode) and after the ON switch for the X-RAY

(Power will automatically reach 20 kV and 10 mA)

To reach maximum power:

- Increase the voltage step by step to 40 kV

-Then increase current to 40 mA





Turn on the two vacuum pumps behind the generators :



#### **Open the shutter for X-RAY**

- Press the black switch of security loop it should turn blue (only when all the gates are closed ) If not, check that all gates are locked and the vacuum pump (located behind generator) is on.
- Press ON the switch for shutter you should be able to hear the sound of shutter. A small red light near to the shutter shows that it is open and we would get an orange light in the X- Ray path.

#### *To set up the PC attached to the detector:*

- 1. Start a terminal window
- 2. Write the code 'cd p2\_det'
- (it will change directory to p2\_det)
- 3. Write the code 'camonly'
- (control window for the detector will open)
- 4.read the temperature and humidity by writing the code 'THread'.
- 5. Ensure that humidity < 2%
- 6. Move to second PC

In case communication is lost between the two PCs, close the control window and type again 'camonly' or repeat the above procedure (points 1. to 6.)

#### Connections to the acquisition PC SAXS-Cu: beamstop

- PICO-X-2 : (1) IN : Intensity measured in the diode inside the beamstop;
  (2) OUT : towards the analogic/digital converter DT9816
- 2. analogic/digital converter DT9816 : (1) IN : from PICO-X 2 OUT; (2) USB OUT to the PC; (3) coaxial cable to the shutter.
- 3. Open the 'gestionnaire de périphériques' and make sure that DT9816 appears in 'DT Open Layers Data Acquisition Devices' an a subfolder named 'DT 9816'. If not, unplug/plug the USB connector again.
- 4. In Autosampler, in 'Current', select DT9816



#### **Connections to the acquisition PC of the 3 motors: Zaber : XZ sample holder**

#### + Z translation for Pilatus

- 1. The three motors are in serial connection on one power supply. Any of the three green slots can be used.
- 2. Plug the USB cable to the PC.
- 3. Open the 'gestionnaire de périphériques' and make sure that the USB serial port COM6 appears. If not, unplug/plug the USB connector again.
- 4. In 'Autosampler', in 'Motors', select 'Zaber'



Z motor



X motor

#### PILATUS Z translation



Power supply

USB to PC SAXS-Cu X motor

## Setup on second computer :

- 1. Start "AUTOSAMPLER" program
- 2. Put x-axis, z-axis, and detector to zero position by pressing 'home' button.
- (do it regularly and imperatively after restarting the setup)
- 3. Adjust height of detector to 40.
- 4. Adjusting fixed position in x and z
- (it corresponds to the reference position in air outside the sample)
- Press the tab 'option' in the first section , and then goes to 'current' tab
- Enter the value of fixed position



### **Autosampler window**

## **SCANNING OF SAMPLE:**

In the 'AUTOSAMPLER'

 Adjust the cursor to a particular position in X- direction (from where you want to scan the sample along Xdirection)

Press the 'SCAN' tab.

Press the 'STOP' tab to stop the scan

 Adjust the cursor to a particular position in Z- direction (from where you want to scan the sample along Zdirection)

Press the 'SCAN ' tab.

Press the 'STOP' tab to stop the scan

It will give scan of the sample. They can be saved and reloaded ('SAVE' and 'LOAD').



Scanning of the sample

# To set the temperature

- Activate a temperature controller (if not done) and restart autosampler.
- Temperature tabs in the upper section should be activated.
- Put the temperature you want to set for the sample and then press 'SET' tab

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<u>Temperature Tab</u>

# **OPTIONS UNDER DIFFERENT TABS**

## In 'Options' tab:

## 1. Current:

fixed position: Set the fixed position outside sample in air for your experiment

2. *Delay time* : set a delay time before acquisition. It is the time system will wait for taking measurements.



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Select for log file

Current Tab

In the middle section:

Add position: Used for adding current position of sample, so it can be used for analysis.

Select the position of sample and then press 'Add Current' tab

*Edit position*: The position and temperature of the sample can be modified using it

Press the edit position button, a dialog box appears –

Modify the position and temperature and then click on 'Modify' tab

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**Add Position** 



### Last section:

Add directory: Select the working directory

*Comment:* to add a comment line (ASCII code) to the log file

#its just to remember the details of the experiment (Make sure that log file creation is selected in the

current tab)

*File name*: Enter the name of file in this tab

*Exposure time* : add the time for which sample is to be exposed to X-rays.

*Frame*: it has to be equal to 1 with PILATUS detector. (Can be changed with CCD detector).

For taking measurement at a particular position:

Select the center of the sample by double tapping at the suitable position in both X and Z direction.

- 1. Add the position by placing 'ADD CURRENT 'tab.
- 2. Put suitable time for exposure of sample to X-RAY.
- 3. Choose suitable directory for saving your file by selecting 'working directory ' tab .
- 4. Choose suitable name for your file

eg: p123\_d2o\_1hr\_1pos\_0degree

(for several positions are in the list, a number will be added at the end of the name automatically)

5. Adjust the fixed position (outside sample) in the 'option' tab

- 6. Check the acquisition time for transmission measurements in the 'option' tab (usually equal to 20 s) (integ. Time)
- 7. Check PILATUS detector position is at 40

## Executing the sequence:

After having all the required changes Press the 'Acquire all' tab.

Measurements will get started !

(for all positions and temperatures in the list)

(if 'start' is selected instead 'acquire all', only the current position is measured and no log file is created; useful for test measurements)

It's done!