

# Quick-EXAFS users' guide

*Before any explanation on the useful Quick-EXAFS' commands, we remain you to read the Users' guide of SAMBA, because you will have to open/close the beamline, make the sample alignment, optimize the gain of the femtos etc...The Users' guide of SAMBA describe all the things you have to know first to use the beamline.*

## **I. Monochromator principle and useful commands**

The monochromator is equipped with a goniometer (motor MONO3-MT\_RX. 2) that allows positioning the crystal of the channel-cut to a near value of the Bragg THETA matches the energy that we want to work. The sweeping in energy is made by an oscillatory motion around this angle THETA, defined by an eccentric cam amplitude delta\_theta movement.

The energy of the monochromator is given by the command: [q3\\_energy](#) calculated by the Bragg formula ( $n\lambda = 2d \sin \theta$ )

$$E=12398.41857/(2*d*\sin \theta)$$

with  $d = 1.637472 \text{ \AA}$  for Si(311)

where  $d = 3.1356 \text{ \AA}$  for Si(111)

where the value of  $\theta$  integrates both the main goniometer position (motor MONO3-MT\_RX. 2) and position of the seesaw coded by a Renishaw encoder (mono3-cd-rx. 1). This angle is obtained by typing the command: [q3\\_theta](#).

The value of the angle defined by the goniometer is obtained by typing [q3\\_theta\\_alone](#).

The value of the swing angle is obtained by typing [q3\\_delta](#)

The angle [q3\\_theta](#) is equal to [q3\\_theta\\_alone](#) + [q3\\_delta](#).

Although this monochromator can operate normally in step by step mode in only moving the goniometer with fixed position of the swing, it is not recommended to use [q3\\_theta\\_alone](#) or [q3-energy](#) movements after that the settings have been optimized for quick-EXAFS mode.

Note: These online orders are part of Pysamba world. Therefore, launch a Pysamba session on the terminal. See explanation on the General leaflet of the beamline.

## **II. Acquisition in steps by step monochromator 's mode**

The acquisition of spectra in step by step mode, must be done with a **fixed position of Delta**.

The acquisition in energy in step by step mode, use the `q3_scan` command:

```
q3_scan E_start E_end step Integration_time name = 'toto'
```

This spectrum is acquired with a delay between the end of the movement and the beginning of the counting ions chambers of 0.25s. If we want to increase this delay, we can add to the end of the command line `delay = xx`.

If you want to make more acquisitions (e.g. 3), you must launch the command:

```
>for i in range(3)
```

```
> q3_scan E_start E_end step Integration_time name= 'toto' (return)
```

```
: (return)
```

```
: (return)
```

```
: (return)
```

```
(return)
```

The acquisition will begin and you see every step of the goniometer scrolled on the screen. A gnuplot window appears with the visualization of I1 in function of energy.

We move the energy to which you want to work by `q3_energy()`.

## **III. Calibration in energy in the step by step mode of the Monochromator**

1. Make a spectrum of reference with the monochromator in step-by-step configuration through the command:

```
q3_scan E_start E_end step Integration_time name = 'toto '
```

2. Locate on the graph and note the energy pointed by the threshold ( $E_{\text{struc}}$ ) structure

3. Move to this energy by the command:

```
mv q3_energy E_struc
```

4. Calculate the theoretical theta angle ( $\theta_{\text{The}}$ ) that corresponds to the energy threshold referenced in the booklet "References X - Ray Spectra" with the command:

```
e2theta (E_ref, d = q3_energy .d)
```

5. Make a DefinePosition on the theta pseudo-motor by typing the following command:

```
q3_theta.DefinePosition (theta_The).
```

Note : Don't forget to connect the right signal cables on the OKEN ion chambers.

### Example in the case of a calibration on the maximum of the Se K threshold

```
PySamba 152>mv q3_energy 12644.6  
17.422238
```

```
Out[152]: 12644.290541334905
```

```
PySamba 153>e2theta(12659.58,d=q3_energy.d)
```

```
Out[153]: 17.400524400303283
```

```
PySamba 154>q3_theta.DefinePosition(17.400524400303283)
```

```
Out[154]: 17.42271391530327
```

## **IV.The oscillator tuning**

### 1 List of specific commands of the oscillator:

`q3_cam.pos ()`: oscillation amplitude in degree

`q3_delta.speed ()`: oscillation speed in Hz

`q3_delta.start ()`: start the motor of the seesaw

`q3_delta.stop ()`: stop the motor of the seesaw

`q3_delta.state ()`: return status (standby or moving)

`q3_delta.pos ()`: number of revolutions of the cam's motor

The amplitude of oscillation is defined by the position of the translation cam. The `q3_cam.pos ()` command permit to force in degree the amplitude of oscillation.

**Attention with the current set-up, the limit value of the cam we can impose is 3°.**

**Do not go beyond this value.**

### 2 Adjustment of the position of the medium theta

The theta refinement is done in the QEXAFS\_MANAGER device with the swing moving of the seesaw.

First go to Pysamba to position the experiment at an energy **E** which is part of the spectral studied domain and return cam **C** and speed **V** values you want by using the following commands:

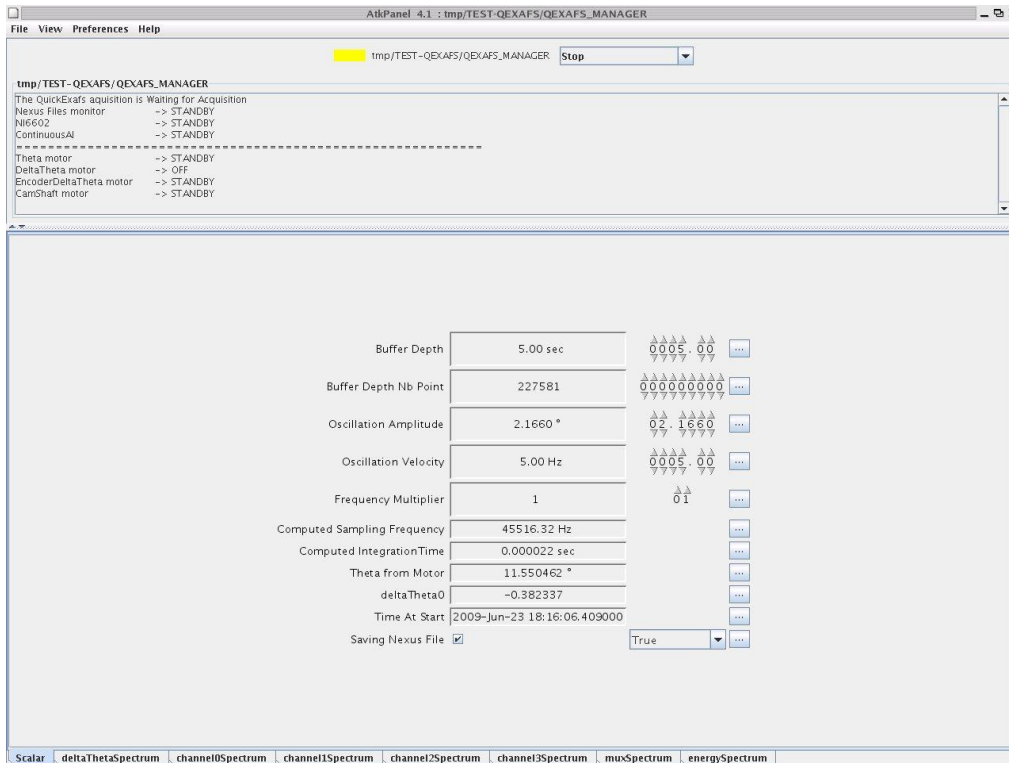
```
mv q3_energy E
```

```
q3_cam.pos(C)
```

```
q3_delta.speed(V)
```

In the window **QEXAFS\_MANAGER** (to open the device, open jive, and choose the Device/Tmp/TEST-QEXAFS/QEXAFS\_MANAGER) open the **Scalar** tab and return values of **C** and **V** imposed above (2.166 ° and 5.00 Hz in the example below).

Check that the value of the sampling frequency of the counter NI6602 card (**Frequency Multiplier**) is equal to 1. This is important because it reduces the size of the buffer allocated for data allowing to easily adjust the optimal acquisition in quick mode, namely: `q3_theta_alone`, `q3_cam` in the beginning of the experience. This option is also useful when you change the samples to adjust correct femtos gains (see "tuning the femtos gains in page 5").

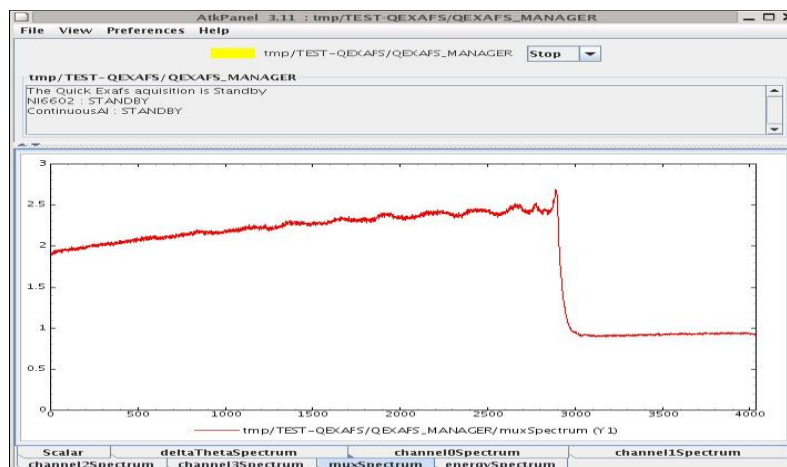


To launch the acquisition in quick mode, you have to select **start** in the scroll menu at the top of the window.

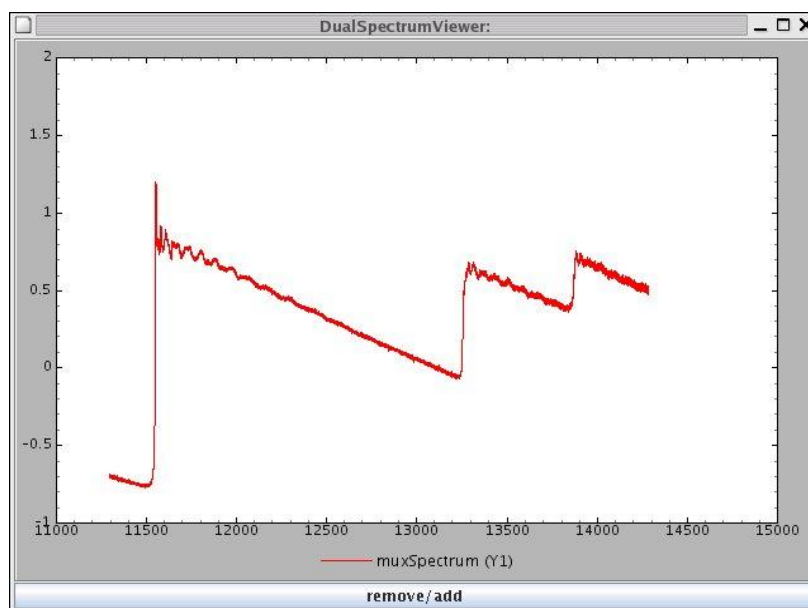
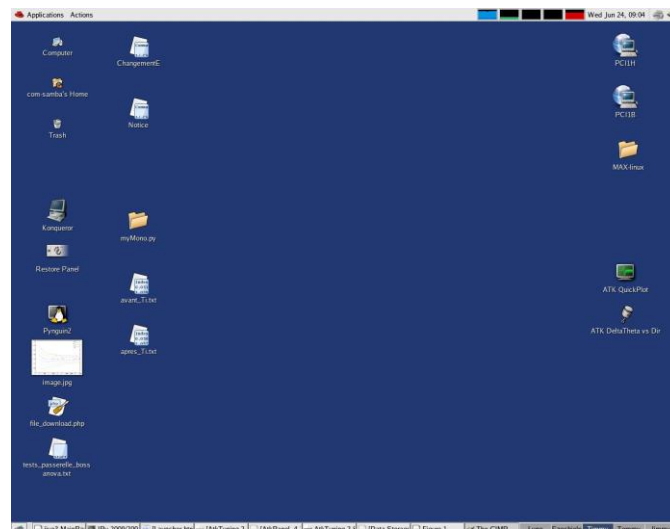
Note: If a pop up window comes with a message referring to a "time out", you release the pop up window and then go to preferences, choose set time-out device, and impose a time out value (which by default is 3000 ms) to 6000 msec.

To stop the acquisition in the quick mode, you have to choose : **stop**. Then you can continue only after that the QEXAFS-MANAGER buffer is emptied on the Ruche .

For adjustments it is convenient to see the spectrum in course of acquisition (tab at the bottom of the window **muxSpectrum**) or acquisition channels (channel0spectrum = I0 channel1spectrum = I1, channel2spectrum =I2). After twice the time needed to fill the buffer first, the first spectrum appears on the screen. The displayed spectrum is in reverse "normal acquisition", i.e intensity in function of decreasing energy. The x axis appears on the screen with a scale in number of points, so it is not linear in energy.



A useful way to see the spectrum in normal configuration (ie with a scale which increases in energy) is to use a tool available on the desktop called ATK QuickPlot. When you double-click on it, a window opens. On this window you can see spectra which are refreshing during the progress of the acquisitions.



Inside this window where spectra are drawn in energy, we will adjust the position of the medium theta in order to have the desired energy domain. Compared with the displayed spectrum,

-If you want more points in high energy, you need to shift the energies of the positive value of (100 eV for example) by the command

```
mvr q3_energy 100
```

-If you want more points to low energy (before threshold), you need to shift the energies of the negative value of (-100 eV for example) by the command

```
mvr q3_energy -100
```

Wait for the refresh of the screen (the buffer time) to see the result of the relative motion. This operation affects the energy taken in the fly of the seesaw oscillation.

Repeat the appropriate operation as many times as necessary to obtain the desired energy domain.

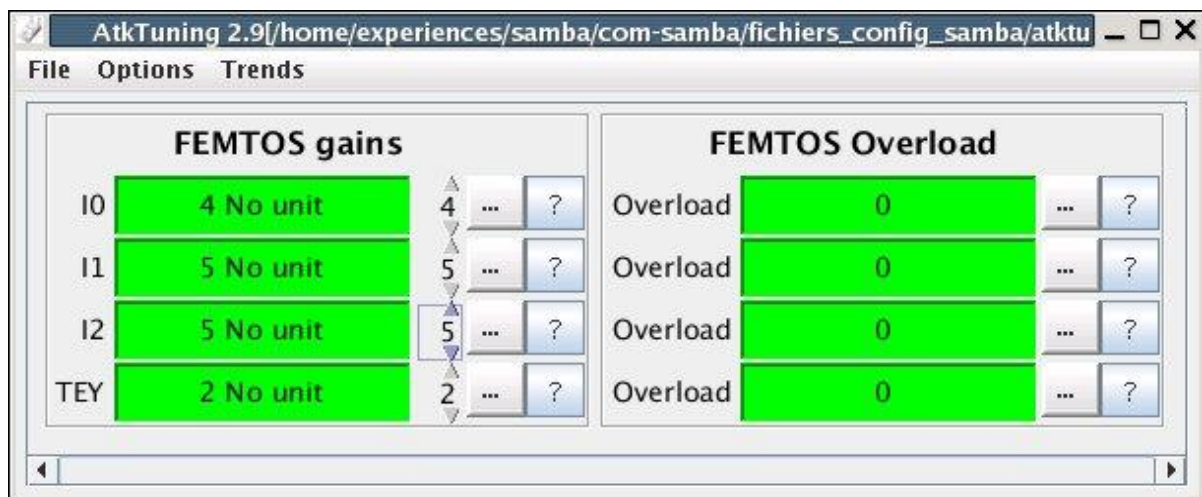
**IMPORTANT:** During the tuning of the seesaw oscillation, you should untick the Saving Nexus file checkbox at the bottom left of the QuickEXAFS\_Manager window.

## **V. What to do before launch kinetics ?**

### **1 Optimizing the femtos gains:**

In a mode "qualified adjustment" - that is to have a **Frequency Multiplier** = 1 and uncheck "Saving Nexus File" box which is at the bottom left of the window of the QEXAFS\_MANAGER- device, we launch a series of acquisition (start) and look alternately at the different counting channels (channel0spectrum = I0; channel1spectrum = I1, channel2spectrum=I2).

Maximum number of counts on the counting card is 16384. If the number of counts exceeds this value, the channel is overload and we see a straight line on the graph. In parallel we see on the ATK Tuning for femtos, the color of the channel relative to this ion chamber changed periodically from green to orange. In this case, we should reduce the gain applied on the femtos in order to have in green color all the channels, meaning that they are all adjusted below of 16384 counts.



### **2. Optimizing of the Frequency Multiplier parameter :**

Before launch the acquisition, we need also choose the right frequency of sampling for the counter card (**Frequency Multiplier**). Adopting a Frequency Multiplier > 1 improves the rate Signal/Noise, especially at high k value. Nevertheless we should take care by changing this sampling frequency factor not to exceed 190 kHz in Computed Sampling Frequency.

Indeed, the NI6602 card stops responding in excess of this sampling frequency and we could risk having a QEXAFS\_MANAGER device in a red state due to an "overrun". In case of red device, see section at the end of this notice for device troubleshooting.

The Frequency Multiplier value to impose depends both on the cam and the frequency of oscillation amplitude. So be careful if you change the frequency of oscillation during an experience also change the sampling frequency

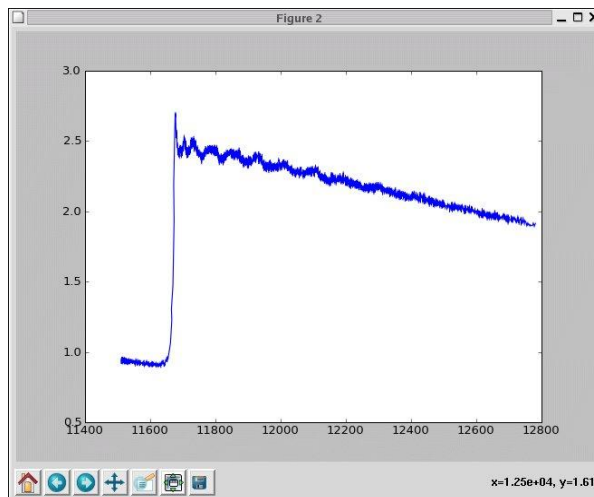
## VI. Display of the spectra during the acquisition

We saw previously that it is possible to see the spectrum during the acquisition with ATK QuickPlot which displays **mux** versus the **energy**.

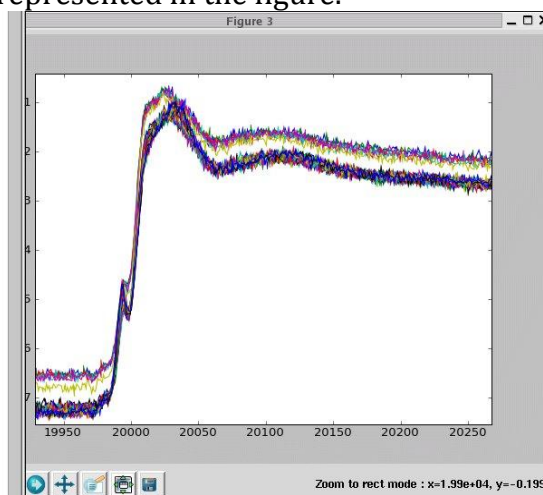
If you want to superimpose different spectra, we have an application in **Pysamba**. Type the command `quickplot`.

This command is a shortcut for the following sequence which consists of putting in a temporary file the current spectrum of the QEXAFS\_MANAGER (1) and displays it on a graphic screen (2):

```
(1): | PySamba 55>qm=DeviceProxy("tmp/TEST-QEXAFS/QEXAFS_MANAGER")
(2): | PySamba 56>plot(qm.energySpectrum,qm.muxSpectrum)
      | Out[56]: [<matplotlib.lines.Line2D object at 0xb7c97d6c>]
```



Whenever we remind the `quickplot` command, we superimpose the new spectrum to the previous spectra already represented in the figure.



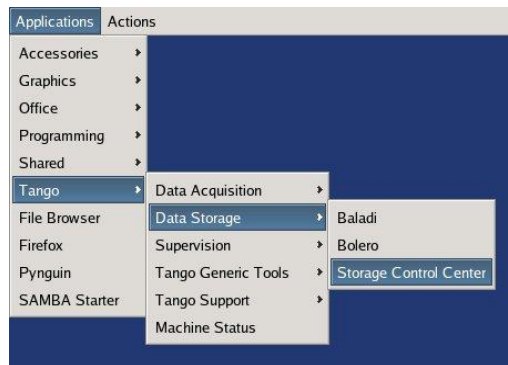
**Note:** to see only the current spectrum, we can open a new window by the command: `figure`



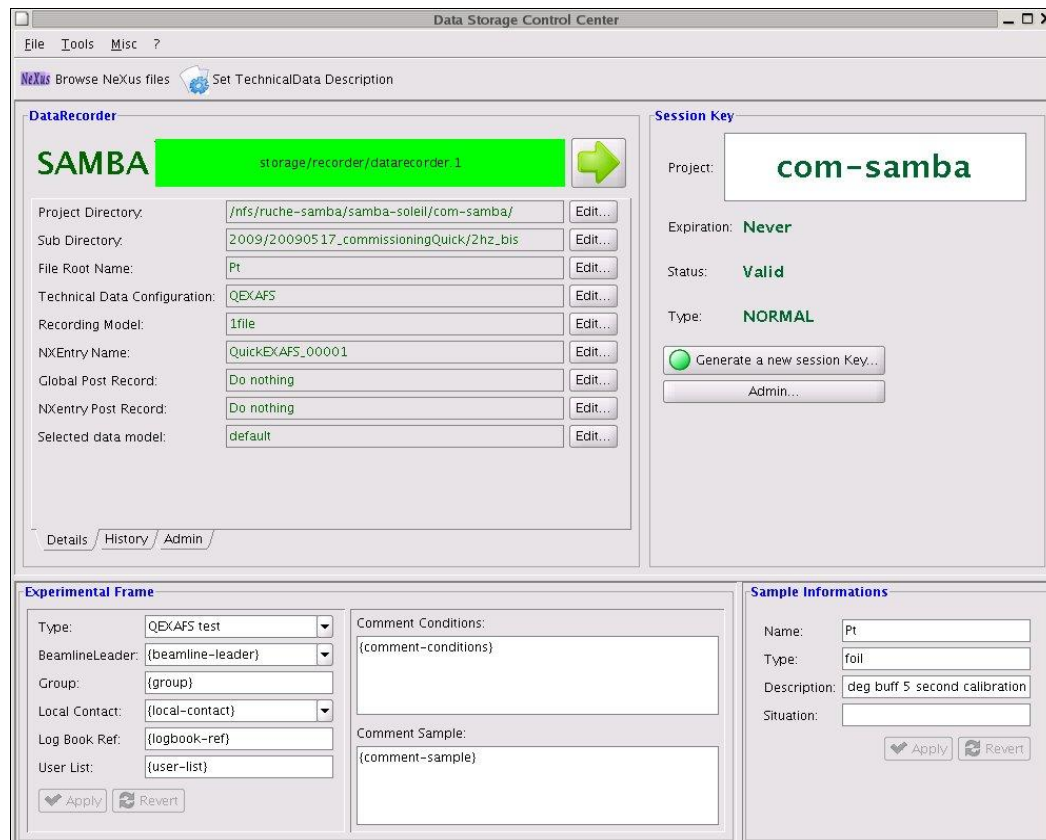
## VII. Acquisition QuickEXAFS mode

Once we have found the right parameters of the oscillator (amplitude, speed, and position in energy) and of the acquisition (frequency multiplier and gains of the femtos), we are ready to launch the acquisition of kinetics. We just have to fill the Storage Control Center to specify the place of storage, the generic name of the file of the kinetics, and the comments.

Opening of Data Storage Control Center:



The following window appears:



### IMPORTANT RECOMMENDATIONS

- 1 / Check the Technical Data Configuration is QEXAFS
- 2 / The Sub-Directory tree must be strictly organized.



We must specify a directory:

year/yearmonthday [\\_username](#)

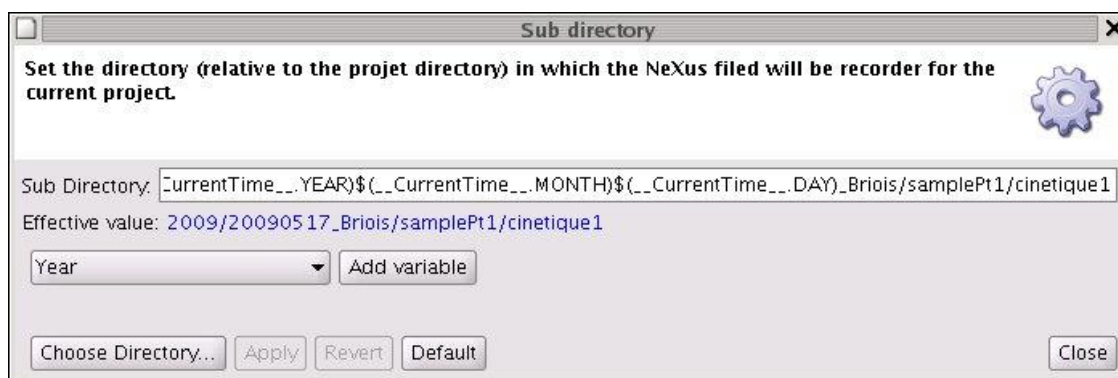
We create a subdirectory for each sample. The tree becomes:

year/yearmonthday [\\_username/sample\\_1](#)

and for each change of experimental conditions, we combine a new subdirectory:

year/yearmonthday [\\_username/sample\\_1/cond\\_exp\\_1](#)

To do this, click on Edit in the Sub Directory and we have the following window that appears :



After you fill the Sub Directory, don't forget to validate by a click on Apply.

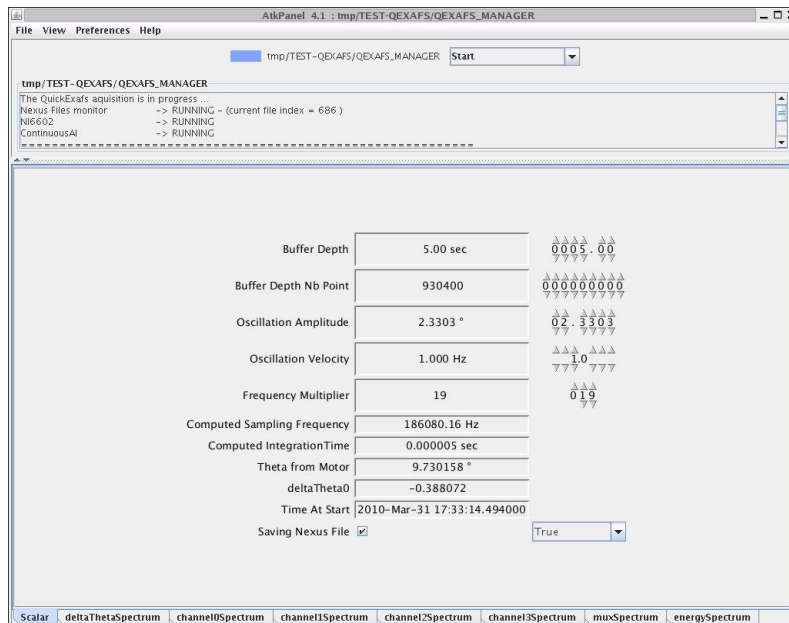
3 / The name of the file that contains scans done between a start and a stop of the QEXAFS\_MANAGER is finally set in File Root Name box.

Note: We may in Sample info box or Comment box at the bottom, add comments as you wish.

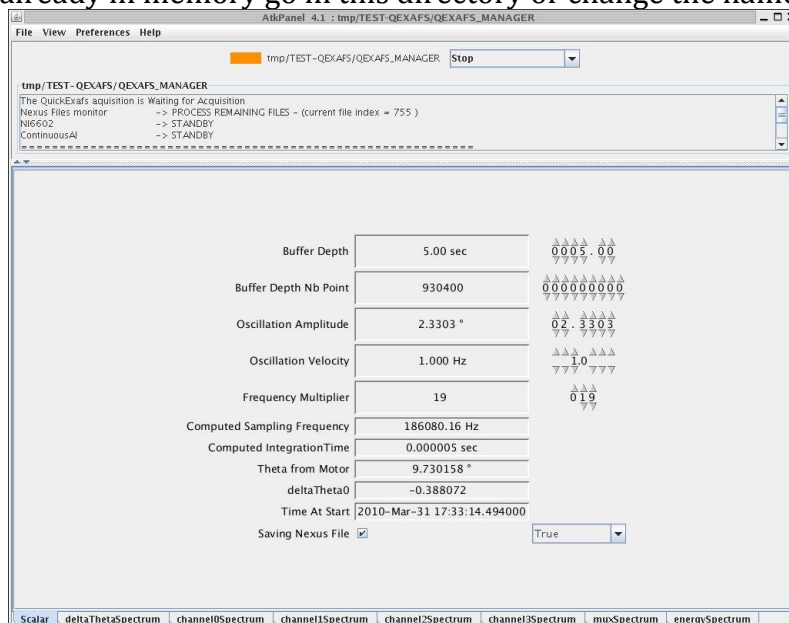
Kinetics will start as soon as we have done a [start](#) in the QEXAFS\_MANAGER device. To save the files we must also have check the "[Saving Nexus file](#)" box at the bottom left of the QEXAFS\_MANAGER window.

### **VIII. Tips, tricks, and problem solving.**

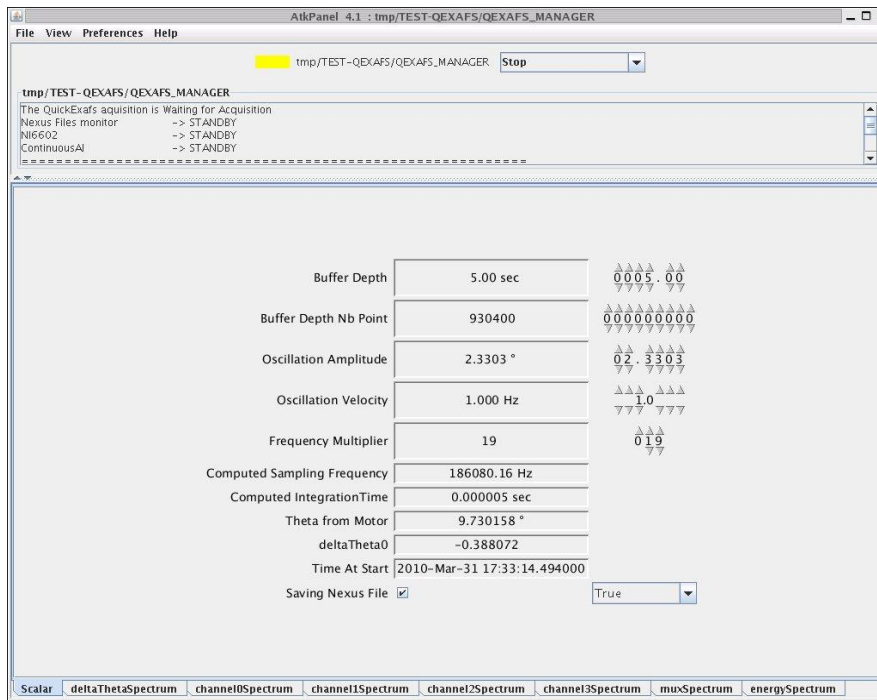
1 / When the QEXAFS\_MANAGER Device is in working mode, its state (color of the label before its name) is blue. If we save the nexus files by the Data Storage Control Center, we can also see the color of this device, changing from green to blue during all the time where the QEXAFS\_MANAGER device is running (see image below)



When we stop the acquisition, then the QEXAFS\_MANAGER device status changes in orange (see picture above) which means that the device is still busy to dump the memory. **Until the orange color persists, made especially nothing.** In particular, do not change in data Storage control Center names file or the directory because you may see the latest data already in memory go in this directory or change the name.



The QEXAFS\_MANAGER device is ready to accept a new start only when its status is became "standby" which corresponds to the yellow color.



## 2 / What to do when the QEXAFS\_MANAGER device status is red?

First we have to know the right origin of the problem. So, go up in the dialog box of the device and see which device is in false or error.

If NI6602 is FAULT, we should open jive and choose the Device/Tmp/TEST-QEXAFS tab, / and make a double click onto the CTP2 device. Usually an "init" of the device permit to turn the CTP2 card in a yellow status.

If ContinuousAI is FAULT, you should open jive and choose the Device/Tmp/TEST-QEXAFS tab, and make a double click on the SAI device. Usually an "init" of the device permit to deliver the SAI card in a yellow status.

If Nexus Files Monitor is FAULT, call the coordinators (on 97 97) who by an "Astor" proceeding will be able in principle to fix the problem by killing the DATA recorder device.

Once the issue is resolved on NI6602, SAI or Nexus Files Monitor, then make an init on the QEXAFS\_MANAGER device that switches back to STANDBY mode (yellow color).

## **IX. Processing to change nexus file in ascii file**

There is a function in Pysamba which permit to transform nexus files in ascii file with a post-treatment to have data sorted by the direction of the seesaw, averaged relative to the number of point defined in Frequency Multiplier with a calculation of the energy, of the mu and mu\_ref. This function is called "moulinex", it has been written by Emiliano Fonda.

**moulinex** transforms into a sub-directory (/nexus2ascii) all nxs files in a given directory as ascii files. In principle the ascii files are named 0000xx.txt, unless we specify a name that is in addition to this string (cf syntax below). This command is time consuming. It takes time to process a folder containing many hundreds of nexus files. Typically to treat

608 nexus files (registration at 0.5 Hz and 1 °) it takes more than one hour (and 1552 ascii files will be created). When we are in the nexus file directory of which we want to convert the files, we just have to write the following command:

`moulinex('name',d=__d111,_AngleUpward=True)` If we convert spectra recorded with Si (111)

`moulinex('name',d=__d311,_AngleUpward=True)` If we convert spectra recorded with Si (311)

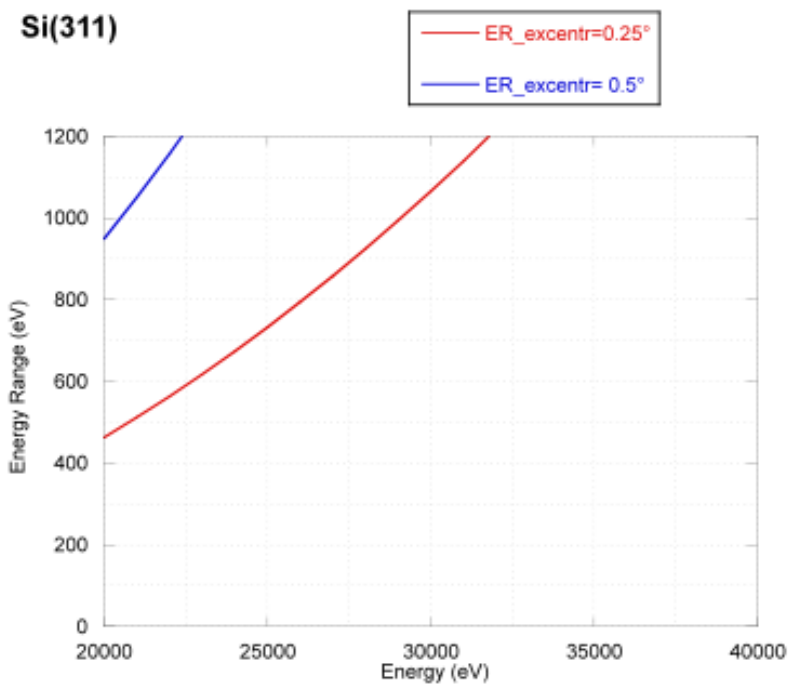
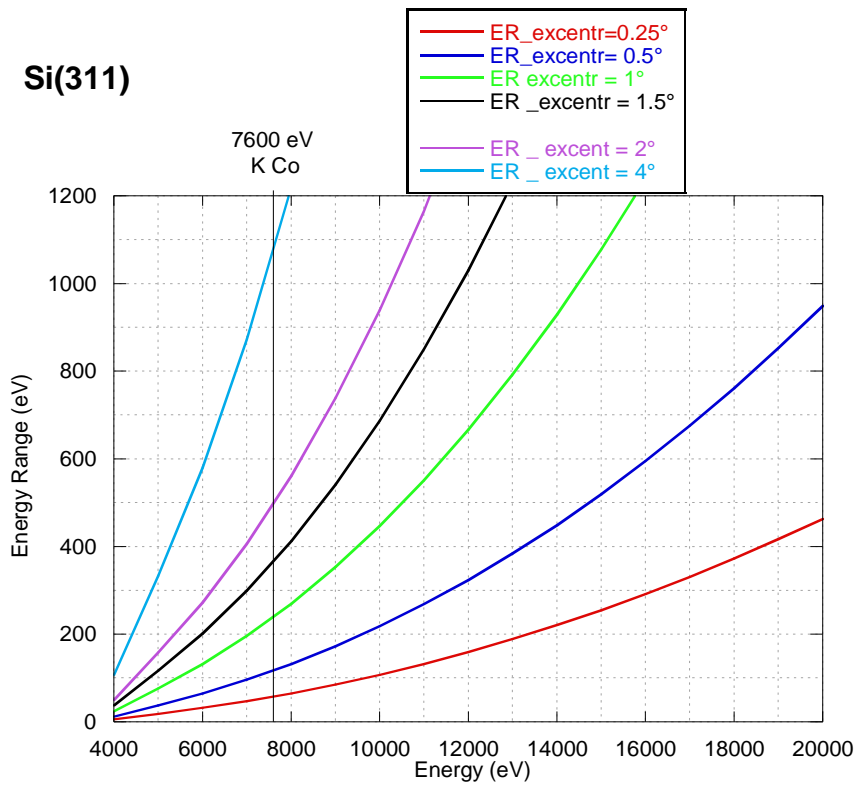
Note: the direction of the seesaw chosen for the extraction is the one that matches acquisitions in ascending angle (`AngleUpward = True`) that is to say high to the low energy spectrum. If we want to analyze the different direction, we should write in syntax: `AngleUpward = false`

A leaflet in English written by Emiliano details all the magnificent "moulinex" tool options.

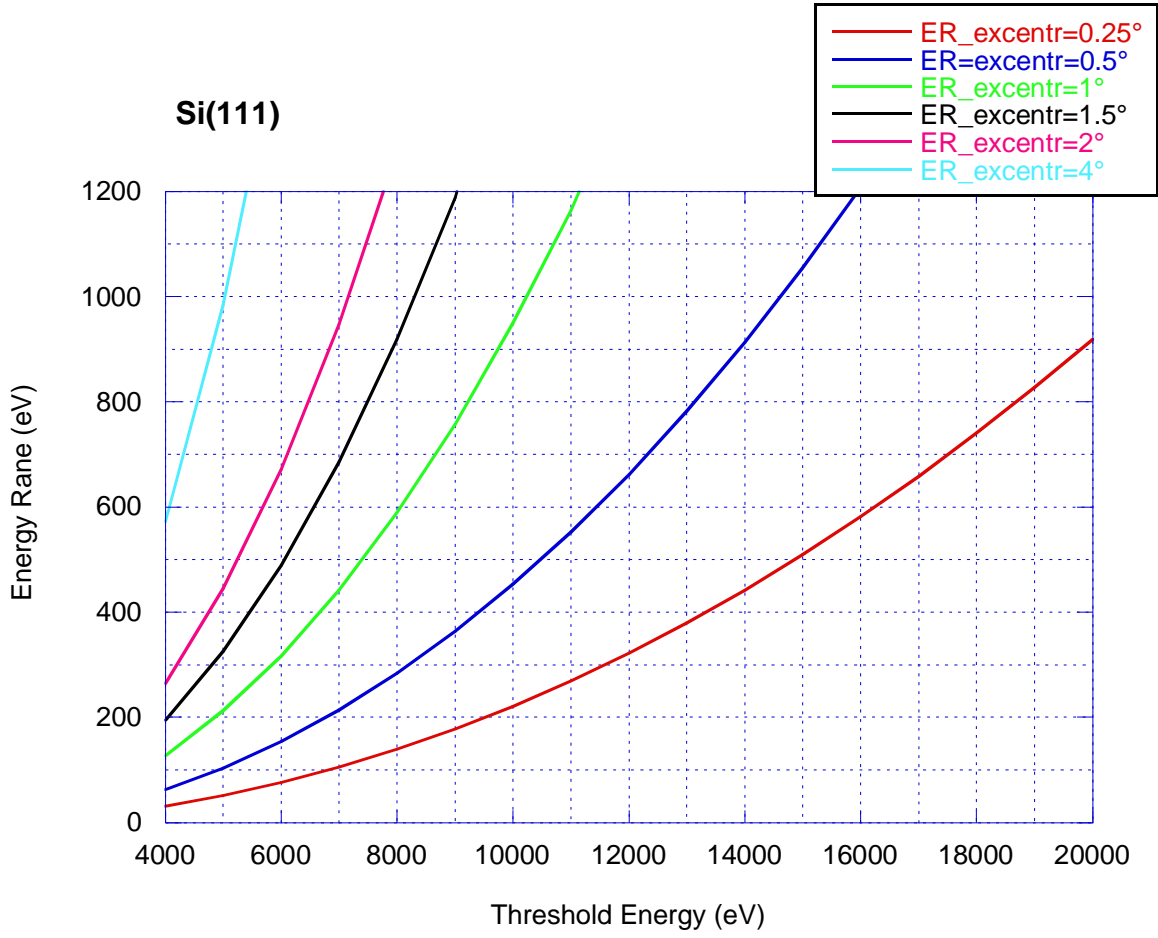
### **X. Choice of the amplitude of the cam versus the energy**

To determine the magnitude of the cam, required for recording of an EXAFS or XANES spectrum, we can refer to the graphs below. **Attention with the current setting, the limit of the cam is 3°. Do not go beyond this value.**

#### **Monochromator Si (311)**



### Monochromateur Si(111)



## **Change from the sagittale mode to Quick-EXAFS configuration**

Close the front-end,

1 / To remove the Oxford monochromator from the pink beam :

Sent rx1 to 0

tz1 in forward

tz2 in forward

2 / Bring the crystal of the Quick EXAF monochromator to the right height [q3\\_tz.pos \(\)](#). This requires looking at the beam on the imager after the Monochromators : we see when we cut the direct beam. By giving an angle to the channel cut [q3\\_rx2.pos \(\)](#), we see the reflected beam.

3 / Adjust the slits vgap2 in order to have a clean beam. We need to change vpos2 from the position they have with the OXFORD monochromator. Typically we are moving to vpos2.pos(4 or 7).

3 / Move down M2 mirror. Theoretically we should lower M2 about 25-H with  $H=2h\cos(\theta)$ . In this last formula : h is the gap of the channel-cut (h = 10 mm) and q is the Bragg angle of the channel-cut monochromator. We apply this law and we adjust the M2 position with imager 3, in order to have a constant beam size on the whole angular field explored during the experience. To do this, swing the seesaw ([q3 - delta .start \(\)](#)) and look at the imager 3.

In the EXAFS hutch, change the OXFORD by the OKEN ionization chambers. Small chamber for I0, and long ones for I1 and I2. Before that, don't forget to cut the high voltage, close the entry of gas valves and disconnect HT and signal cables.

To deliver the high-voltage in OKEN chambers we must remove the inhit cables from the ISEG drawers.