USER MANUAL

Operation manual for users

May 2018

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1 First Experimental Hutch EH1

The first experimental hutch EH1 (fig. 1) is dedicated to experiments with unfocused beam, mainly EXAFS in transmission mode.

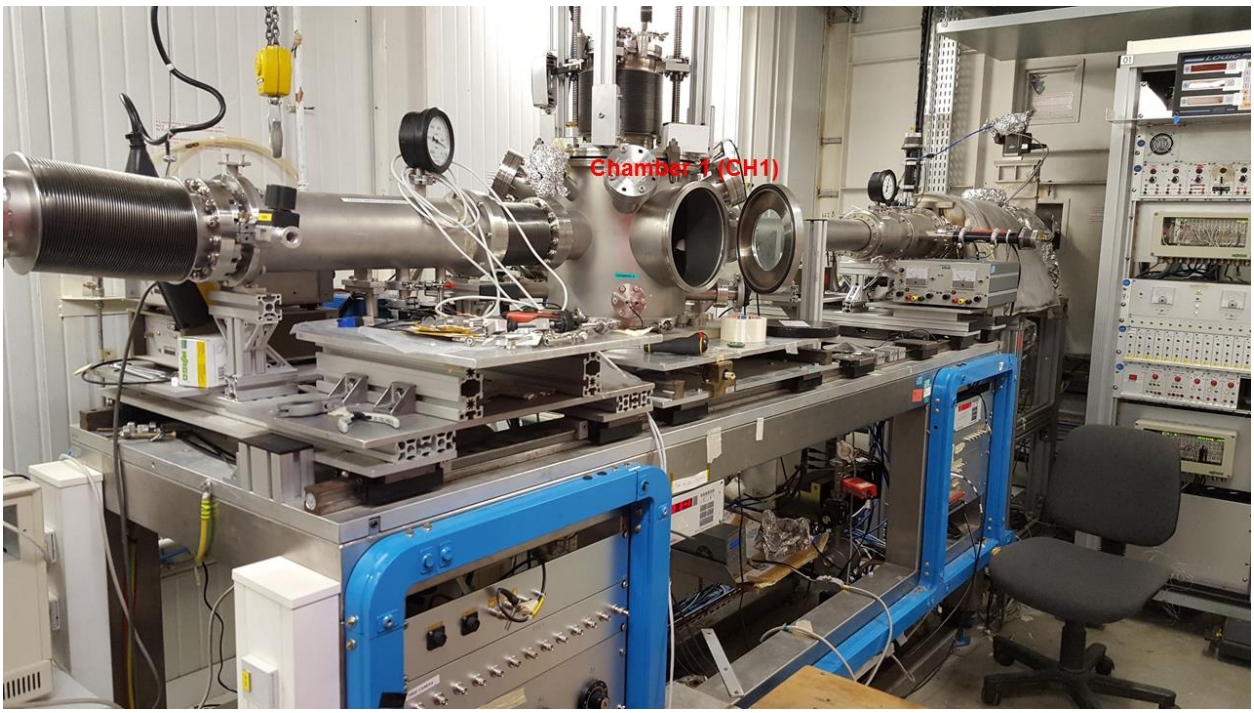


Figure 1: First experimental hutch EH1.

The principal movements in EXAFS chamber 1 (CH1) (fig. 2) are

hpos1 moves the EXAFS chamber 1 horizontally. Positive (negative) values move the chamber right (left) facing the incoming X-ray beam.

vbell moves the sample holder vertically in chamber 1. Positive (negative) values move the sample holder up (down).

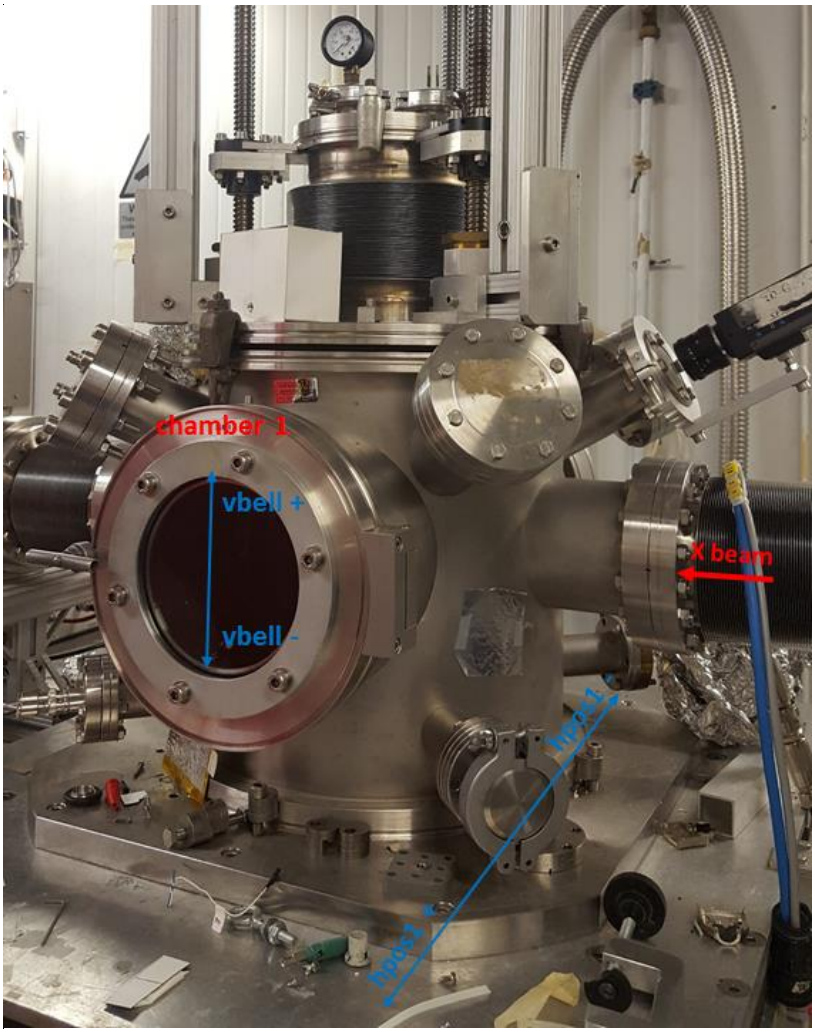


Figure 2: EXAFS chamber 1.

2 Second Experimental Hutch EH2

The second experimental hutch EH2 (fig. 3) is dedicated to experiments with focused beam (i.e. EXAFS in fluorescence mode, GIXAS, experiments with diluted samples).

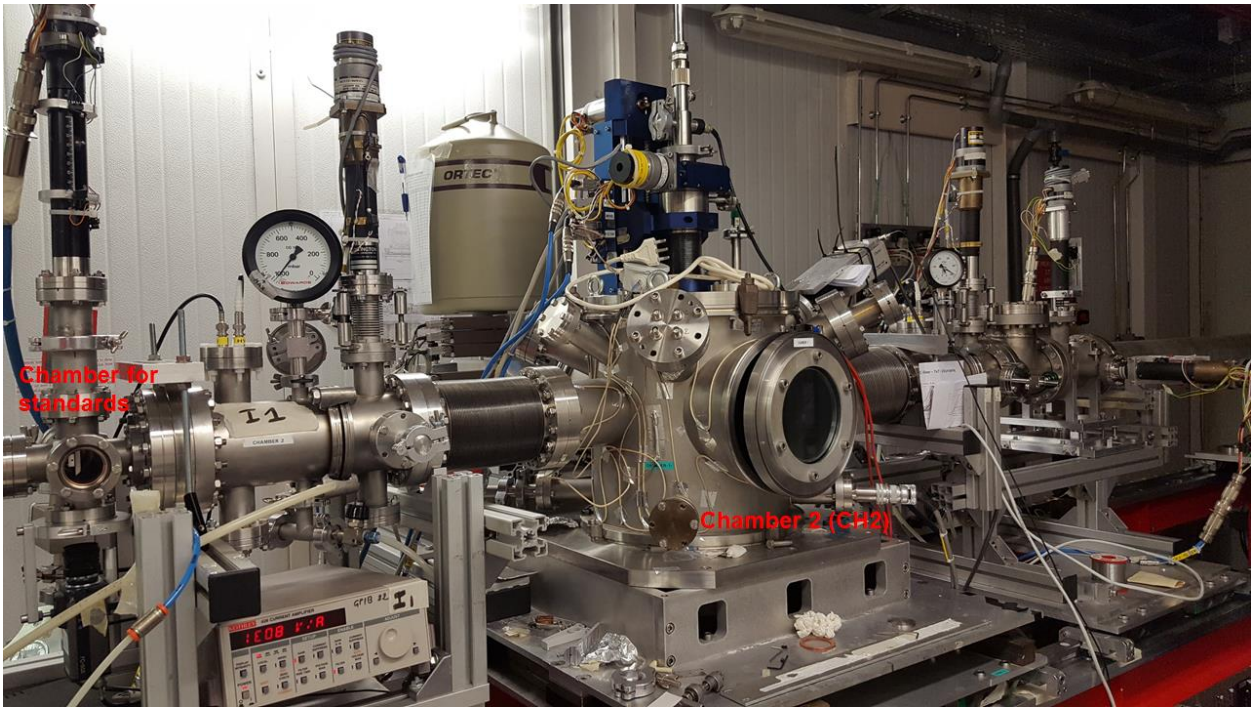


Figure 3: Second experimental hutch EH2.

Below are listed the principal motors in EH2 (fig. 4):

vcof moves the sample holder vertically in chamber 2. Positive (negative) values move the sample holder up (down).

hpos2 moves the EXAFS chamber 2 horizontally. Positive (negative) values move the chamber right (left) facing the incoming X-ray beam.

rot2 rotates the sample holder in chamber 2. Positive angles orient samples towards the detector. *0* value corresponds to the beam transversal to the sample holder.

ydet moves the fluorescence detector behind chamber 2 closer or farther the samples, values range from *0* to *210* corresponding to the closest distance to the sample.

vstd moves the sample holder vertically in the standards chamber. Positive (negative) values move the sample holder up (down).

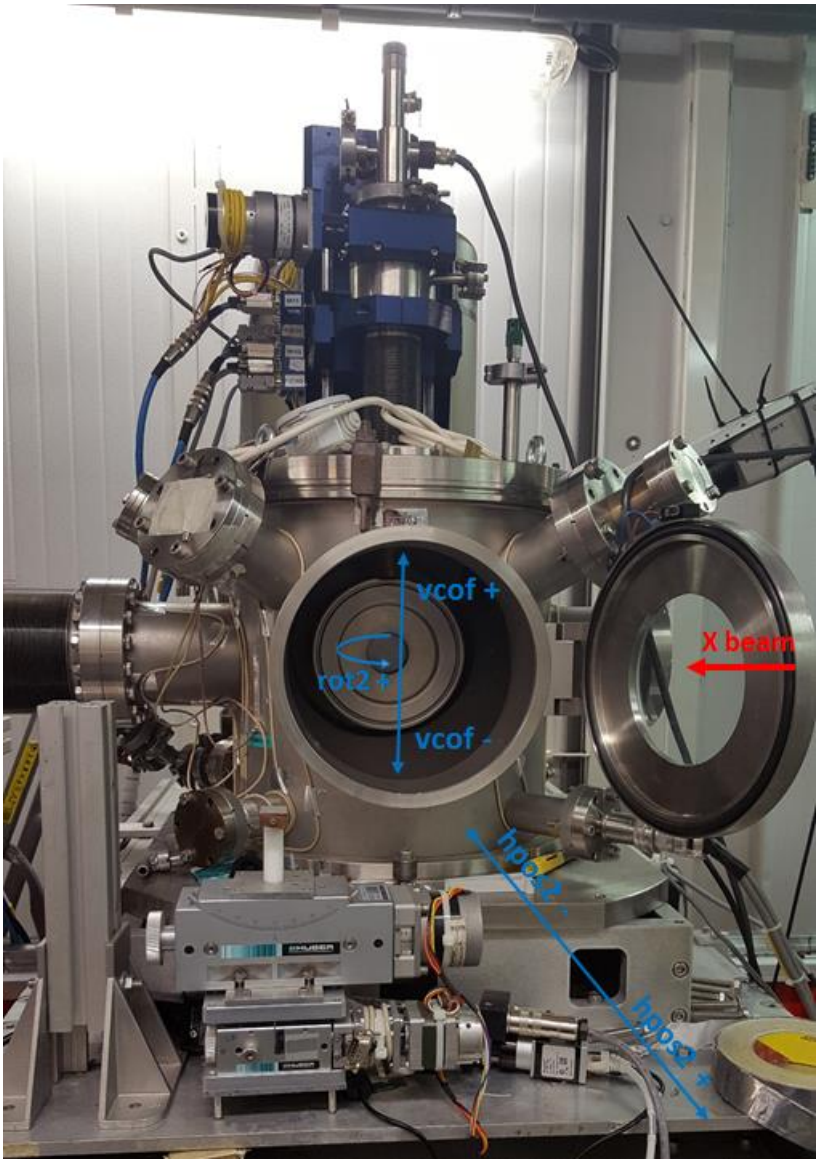


Figure 4: EXAFS chamber 2.

3 How to control the motors

All the motors are controlled from the main pc ***ld082*** of the beamline located in the control room via the spec sessions ***eh1***, ***eh2***, respectively for the first and second experimental hutch.
To move a motor relative to its position:
umvr motor_name xx (e.g. *umvr hpos1 10*)

Where *xx* is the amount you want to move in mm or degrees

To move a motor to a given position you can use the same commands but with *umv* replacing *umvr*.

To control the motors you can also use the utilities **eh1_motors** and **eh2_motors** located in the folder 'DATA-COLLECTION' on the desktop of the main pc.

eh1_motors and eh2_motors utilities

4 Mounting the samples

You can mount solid samples (pellets, membranes, films) on a standard copper or aluminium sample holder (see figure 5).

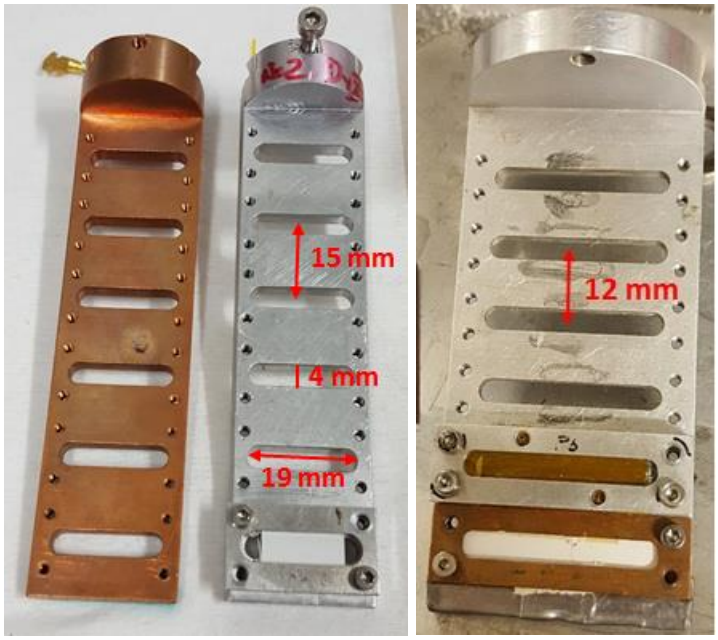
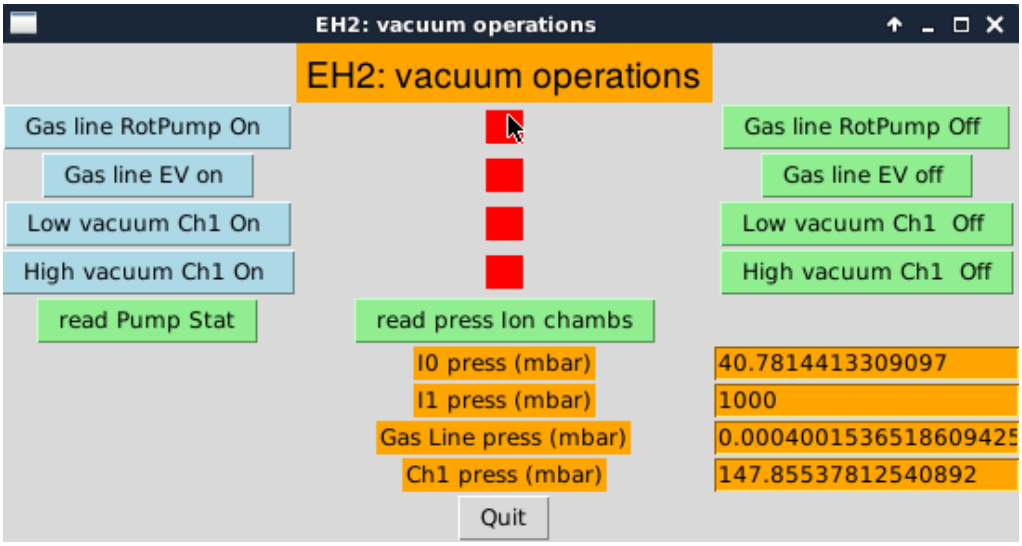


Figure 5: Sample holders chamber 1 (left) and chamber 2 (right).

The sample holder can host up to 6 samples which size not exceeding 15 mm in high. With larger samples you can mount a reduced number of samples.
Copper sample holder is more efficient for low temperatures purposes but you should avoid using it for fluorescence acquisition close to the Cu absorption edges.

5 Vacuum operations

Once you have mounted the sample on the sample holder in EXAFS CHAMBER 1 or 2, close the chamber port and start the corresponding vacuum pumps. This can be done remotely with the Vacuum utility or from the spec session **VACUUM** (pink command line terminal).



Vacuum utility

Low Vacuum (few mbar) (only for room temperature measurements)

1) Pumping the XAS experimental chamber in EH2

- close the experimental chamber port
- do not forget to close the experimental chamber venting valves (placed under the bench behind the respective turbo pumps)
- press the button **Low vacuum Ch1 On**
- alternatively on the VACUUM spec session, type: ***start_low_vacuum_ch2***
- the program will start the rotative pump and will prompt you when the chamber is in vacuum.

2) Stopping the pump and breaking the vacuum in the XAS experimental chamber in EH2

- press the button **Low vacuum Ch1 Off**
- alternatively on the VACUUM spec session, type: ***stop_low_vacuum_ch2***
- open slowly the experimental chamber venting valve.
- when the chamber is at atmospheric pressure you can open the port.

High Vacuum (10^{-6} mbar) (necessary to work at low temperature)

1) Pumping the XAS experimental chamber in EH2

- Close the experimental chamber port

- Do not forget to close the experimental chamber venting valves (placed under the bench behind the respective turbo pumps)
- press the button **High vacuum Ch1 On**
- alternatively, on the VACUUM spec session, type: ***start_high_vacuum_ch2***
- the program will automatically start the rotative pump and when a suitable pressure has been reached start the turbo pump. Wait until the end of the sequence and do not interrupt the program. The program will prompt you when the chamber is in high vacuum.

2) Stopping the pump and breaking the high vacuum in the XAS experimental chamber

- press the button **High vacuum Ch1 Off**
- alternatively on the VACUUM spec session, type: ***stop_high_vacuum_ch2***
- after about 5 minutes, open slowly the experimental chamber venting valve. This will slow down the turbo-molecular pump
- when the chamber is at atmospheric pressure you can open the port

NB: for the experimental chamber in EH1, you can follow the same procedure BUT with CH1 replacing CH2 using the spec session VACUUM.

WARNING
Once you have opened the exp. Chamber venting valve to break the vacuum REMEMBER TO CLOSE IT BACK. **Starting the pumps with the valve open prevents lowering the chamber pressure and can damage the pumps!**

If the turbo pump was on you will have to stop it and wait that the deceleration is complete BEFORE opening the valve and breaking the vacuum in the exp. chamber. **Opening the valve with the turbo pump on will damage the turbo pump.**

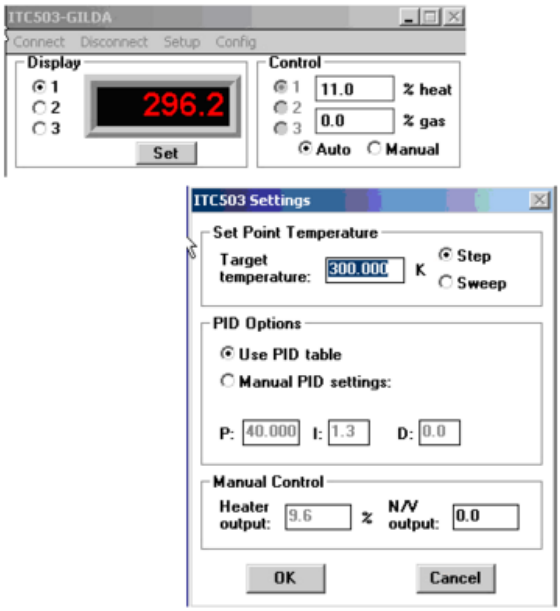
Check the Vacuum status in CH1/CH2
At any moment (except while pumping the chambers), you can check the status of the vacuum in the first and second experimental chambers, by typing: ***status_vacuum_ch1*** or ***status_vacuum_ch2***.
The software will tell you if the rotative and turbo pumps are running, and if there is vacuum (low or high) in the corresponding chambers.

6 Cooling the samples

1. Close EXAFS CHAMBER 2 and start the high vacuum following the procedure reported in the *Vacuum operations* section, until the turbo pump has reached his operative speed. A message on the VACUUM spec session will inform you when the chamber has reached high vacuum conditions.

2. Launch the Control application Cryostat on the pc inside the EXAFS hutch

3. Set the temperature on the cryostat application by clicking on the set button, typing the desired temperature and clicking the OK button.



Cryostat control application

4. Start the He2/N2 pumping by switching on the cryostat pump (figure 6 left) and opening gradually the valve indicated by the arrow in figure 6 right until the vacuum-meter (figure 6 right) reaches the higher depression (about -0.6 -0.8 bars). The temperature should start to decrease (typical rate 0.1 K/s).

5. When the set temperature is reached, in order to optimize the nitrogen consumption you must reduce the depression by gradually closing the valve indicated by the arrow in figure 6 right, until the depression stabilizes to a value around -0.2 bars. Note that the heater power reported in the Cryostat control window should not be less than 10-15%, otherwise the temperature can drift to higher temperature values and not higher than 30% to avoid excessive nitrogen consumption.

Figure 6: left: He/N cryostat pump. Right: cooling line to the sample holder.

6. Wait some minutes for the sample holder to thermalize because of its thermal inertia.

7. In the first minutes the depression can drift. Check it and adjust the vacuum valve accordingly.

Heating up the samples

1. Close the valve indicated by the arrow in figure 3 right and stop the He2/N2 pumping by switching off the cryostat pump.

2. Set the temperature to 295 K in the cryostat control window and hit the OK button.

3. After the temperature has reached the set point (this will take up to 30 minutes), wait some minutes for the sample holder to reach room temperature (because of its thermal inertia).

4. Once the samples are at room temperature, you can stop the high vacuum and vent the chamber. NEVER open the experimental chamber at low temperature!

Notes:

Working temperature: for good temperature stability the helium/nitrogen flux must be high enough to keep the cryostat heater on to a power that should not be less than 10 - 15%. At the same time, it should be as lower as possible in order to reduce helium/nitrogen consumption.

Duration of the nitrogen/helium reservoir:
The liquid nitrogen dewar can last for at least 3-4 days. It is recommended that you refill it regularly. The local contact will show you how to refill the liquid nitrogen dewar. The helium reservoir dewar last for about 5-6 days. Since changing the dewar takes time and requires the intervention of your local contact, always check the helium consumption to estimate the remaining level.

Cooling down time Cooling down the sample takes about 45-60 min. when working with liquid nitrogen and about 15-20 min. when working with liquid helium. During the first 5 min. the temperature will not change due to thermal inertia of the system (cryostat transfer pipe, heat sink, sample holder...).

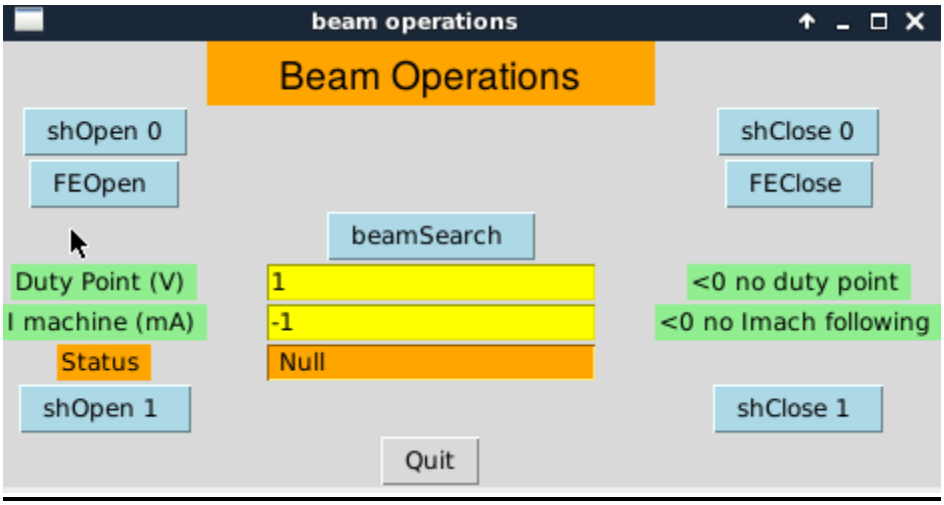
Real temperature on the samples: Remember that, the bottom of the sample holder is warmer than the top. The shift increases by decreasing the working temperature, reaching up to 10 degrees. If you need to monitor the temperature of your sample with higher accuracy (about 1K), you use a temperature sensor mounted close to the sample.

7 How to align the samples

Mount the samples in EXAFS chamber 1 or 2 (see fig. 1)

The monochromator energy is a SPEC pseudo-motor with a position defined in keV. Under XAS you can use the command
XAS> umv energy 13.0
To set the output at 13 keV.

Check that the beam intensity is not zero by looking at the I0 value on the red indicators or the value of the point floating on the oscilloscope. If you have zero intensity, search the beam using the utility **beam** located in the folder 'DATA-COLLECTION' on the desktop of the main pc.



Beam utility

Enter the duty point value and the machine current I and press beamSearch button. If you enter negative I values the duty point will not be rescaled according to the machine current, this is recommended for 16 bunch mode.

To align the samples with the beam, do a position scan. For example from spec session **eh1** or **eh2** use the command:
dscan vbell (or vcof) -5 5 20 1
For vertical scan and
dscan hpos1(2) -5 5 20 1
For horizontal scan.

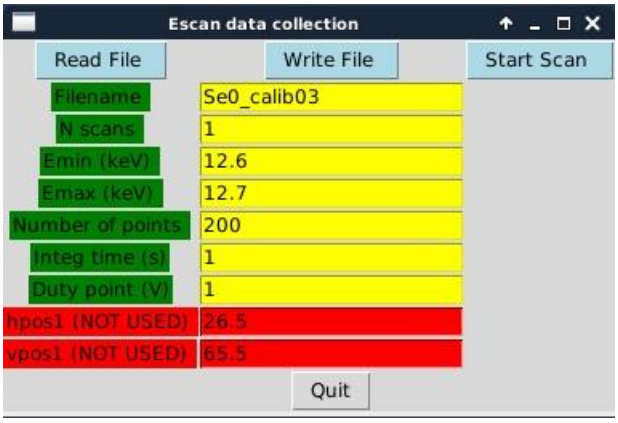
First two parameters are the position interval, third parameter is the number of position steps and the last one is the integration time in seconds. Typical intervals are *-4 4* for vertical scan and *-8 8* for horizontal scan. The typical integration time for fast scans is 0.5s.

You can also perform a scan between two definite positions with the command *ascan*, for example
ascan vbell 3 18 40 1
Performs a vertical scan between position 3 and position 18.

8 How to acquire spectra

Once the samples are well aligned with the beam you can start acquiring spectra. There are different data collection utilities available:

8.1 Escan



Escan utility

Escan acquires data at constant steps in energy for one scan region, the main parameters are:

Filename: name of your data file.
N scans: number of scans for sample.
Emin: initial energy in keV
Emax: final energy in keV.
Number of points: number of steps in the energy interval.
Integ time: integration time in seconds.
Duty point: beam duty point in volts.

Once you have entered the parameters click the Write File button and then the Start Scan button to start the acquisition.

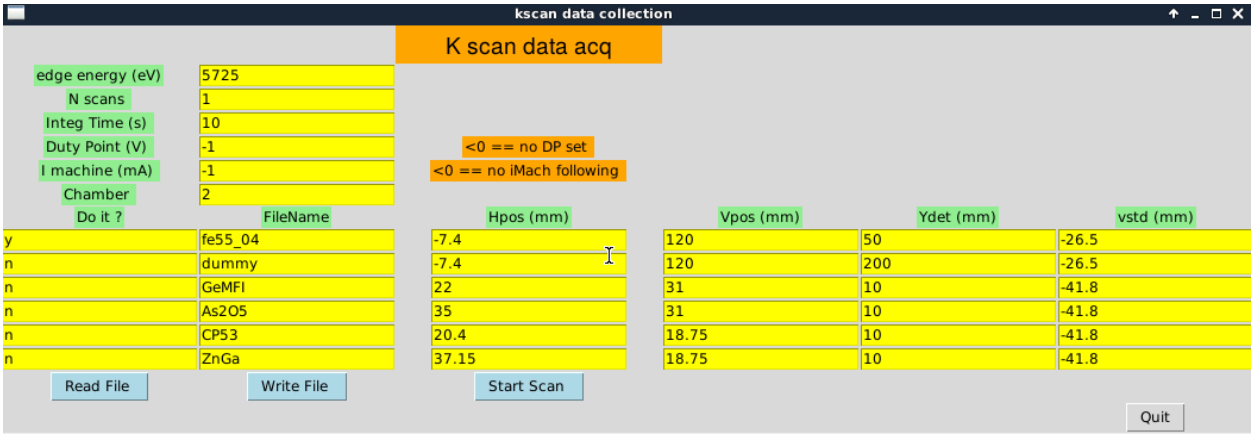
8.2 Kscan

Kscan utility allows multiple scan regions to be set up, with different parameters in each, as well as scan regions where the steps are in 'k' rather than energy.
You can choose your scan preferences with the kscan setup interface.

Kscan setup

PRE_EDGE_INIT: initial energy relative to edge in eV.
PRE_EDGE_STEP: energy increment in the pre edge region in eV.
EDGE_START: starting edge region energy relative to the edge in eV.
EDGE_END: final edge region energy relative to the edge in eV.
EDGE_STEP: energy increment in edge region in eV.
K_END: final k value.
K_STEP: k increment.

Once you have set up your scan preferences, you can use the Kscan utility to collect the spectra.



Kscan utility

edge energy: edge energy in eV
N scans: number of scans for each sample
Integ time: integration time for each step in seconds.
Duty point: beam duty point in volts. If < 0 no duty point is set.
I machine: machine current in mA. If < 0 the duty point will not be rescaled according to the machine current, this is recommended for 16 bunch mode.
Chamber: 1 or 2 select EXAFS chamber 1 or chamber 2
Do it?: y or n, if y scan is executed.
Filename: name of your data file.
Hpos, Vpos: Horizontal and vertical position of the sample.
Ydet: fluorescence detector distance from the sample, *0* is the maximum distance, *210* is the minimum distance.
vstd: vertical position of the reference compound

Once you have entered the parameters click the Write File button and then the Start Scan button to start the acquisition.

You can pause or interrupt the spectra during the acquisition using the buttons on the cupboard in the control room (fig. 7). Left one **O** is for pausing the acquisition, center one **WR** is for stopping a single spectrum, while the right one **B** is for stopping the entire macro application.

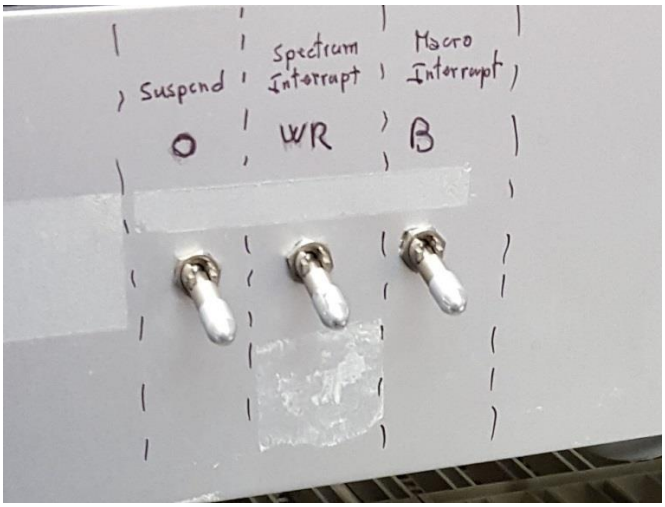
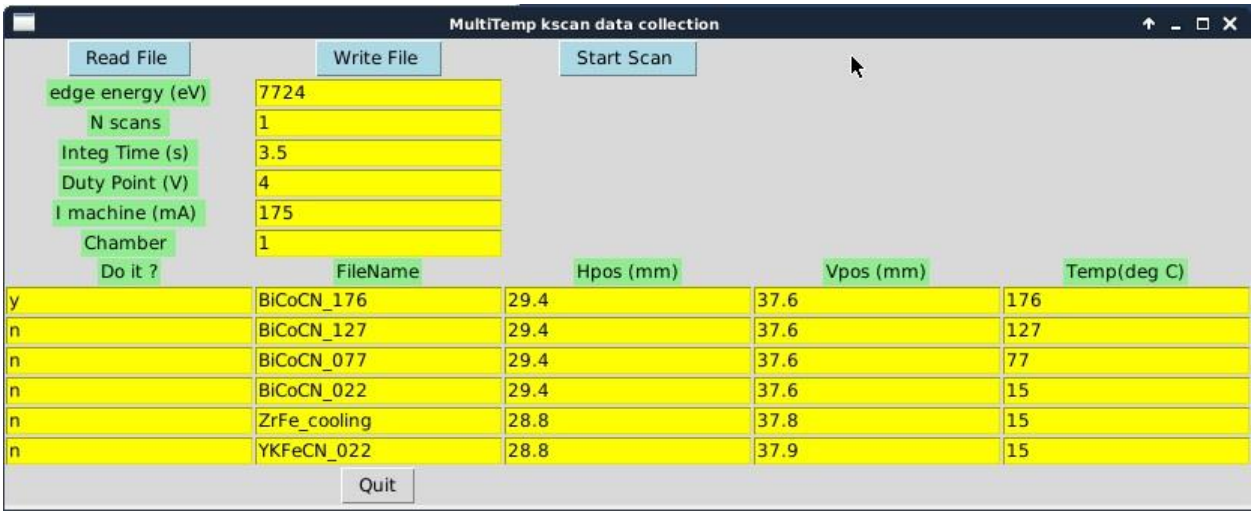


Figure 7: Buttons for pausing/stopping the acquisition

8.3 MultiTemp kscan (to be updated)

In experiments using the furnace form the sample environment pool, Multi temperature kscan allows k scan to be performed at different temperatures.

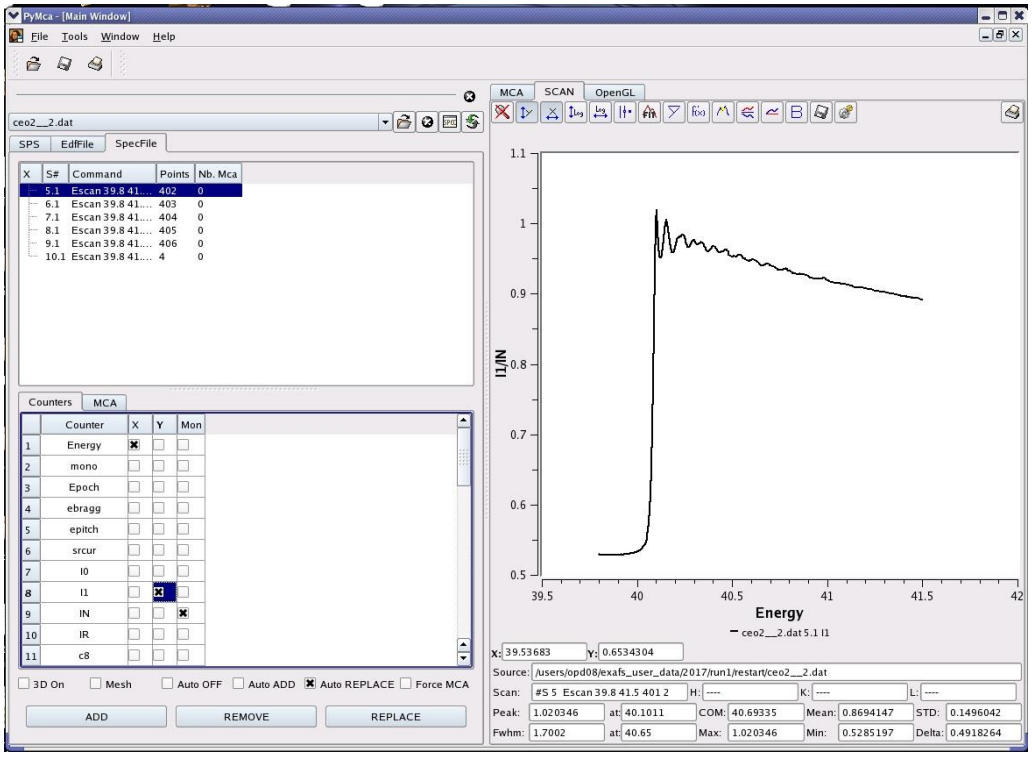


MultiTemp kscan utility

It takes the same parameters of kscan, with an extra column, where the temperature is entered.

8.4 Plotting the data.

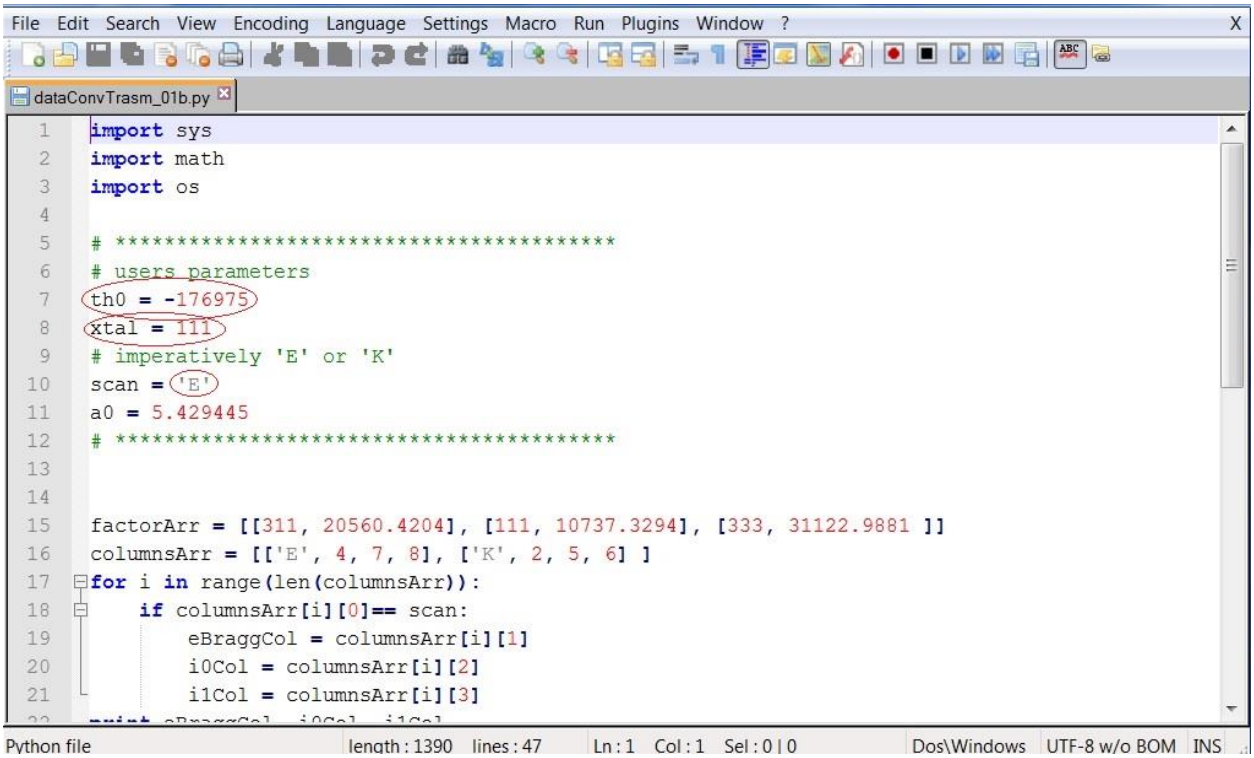
You can plot the spectra using the software PyMCA. Open the data file you want to plot and select the columns **Energy** as *X*, **I1** as *Y* and **IN** as *Mon* for the spectra acquired with the Escan utility. For the spectra acquired with the Kscan utility select the columns **I0** as *Y* and **I1** as *Mon*.
In the case of fluorescence spectra select as *Y* the data columns corresponding to the detector channels, named **fluo01, fluo02… fluo12**



PyMCA main window

9 Data treatment

Raw XAS data can be reduced to a simpler format using the following python codes:
dataConvTrasm_01b.py for transmission data.
dataConvFluo_01b.py for fluorescence data.
They are launched in the working directory with the command
python dataConvTrasm_01b.py 'dataFileName.ext'
In the first rows there is a section that must be checked before converting. It contains the zero angle ***th0*** for energy calibration, the crystal type ***xtal*** and the kind of data i.e. obtained by ***K*** or ***E*** scans.
The output is a file named *'dataFileName_r.ext'* with the structure
Energy (eV), I0, I1, mu, (fluo channels if applicable), secondary energy (ev)
That can be easily treated with standard data extraction codes.



Data treatment program with indicated the parameters to be checked.

10 GIXAS sample holder (to be updated)

In figure 8 the GIXAS sample holder is shown:

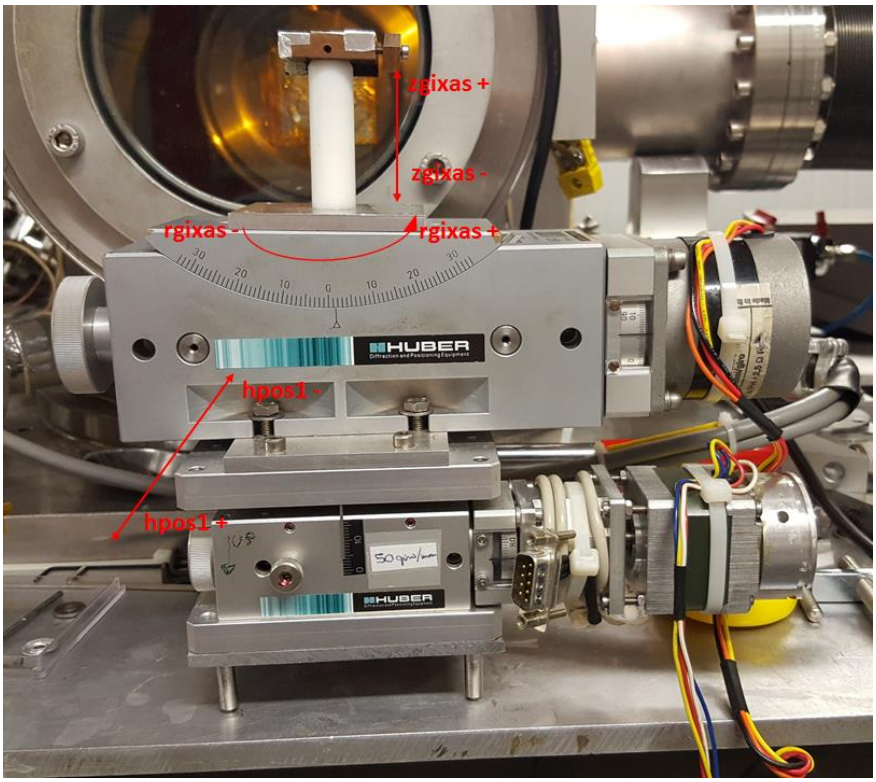


Figure 8: The GIXAS sample holder

Below are listed the principal motors of the GIXAS sample station.

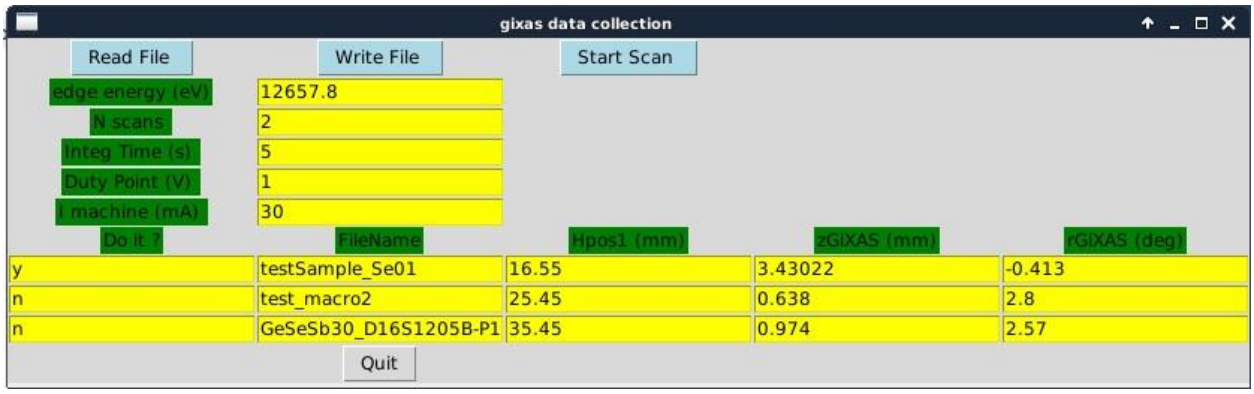
zgixas moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

rgixas rotates the sample holder respect to the incoming X beam. Positive (negative) values increase (decrease) the incidence angle.

hpos1 moves the sample holder horizontally. Positive (negative) values move the holder right (left) facing the incoming X-ray beam.

10.1 Acquiring GIXAS spectra.

To collect GIXAS spectra you can use the **gixas data collection** utility.



GIXAS data collection utility

It takes the same parameters of the kscan utility, plus the columns ***zGIXAS***, ***rGIXAS*** where the motor positions of the GIXAS holder are entered.
Once you have entered the parameters click the Write File button and then the Start Scan button to start the acquisition.
Remember to configure the scan using the utility **kscan_setup** before starting the acquisition.

11 Local Contact policy

1. One member of the beam-line team will be associated to your experiment as **LOCAL CONTACT**. You are encouraged to contact him/her as soon as possible, once the acceptance of your experiment has been notified to you.

2. The local contact will help you to perform your experiment. **He/She is NOT there to perform the complete experiment for you**. It is your obligation to provide sufficient staff to operate the experiment 24 hrs a day (in any case a minimum of 2 people is required). You need to ensure that the A-Form is completed early enough to ensure that all arrangements can be done properly. In addition, you and your staff have to pass the Safety Training course before starting the experiment.

3. Before your departure, you are asked to leave the beam-line control and experimental areas in the same conditions they were in at your arrival. Your departure time might have to be booked accordingly to allow for "clean up" time.

   The LOCAL CONTACT will:
   - **prepare the beam-line within the limits of a standard set-up**,
   - **introduce you to the operating of the beam-line**
   - **help you in setting up the sample environment**,
   - **be on call from 8.00 a.m. to 10.00 p.m**.

4. You are required to have at least basic experience in information technology, XAFS or XRD (depending on your experiment) techniques, as well as in the corresponding data analysis. In the case none of your research group have these prerequisites, collaborations between users and the beam-line staff should be arranged well before the experiment starts. Should you need help and support from the staff equivalent to that of a collaborator (custom setups, data analysis, full-time assistance...) you should offer them the **status of collaborators** formally. If the beamline scientist accepts, he/she will be willing to make an effort beyond his/her normal duties.

   The usual course of action would be to
   - address the issue,
   - agree on a collaboration,
   - include him/her as co-proposer,
   - include the him/her in further discussions on the interpretation of results,
   - award him/her co-authorship,
   - have him/her approve the final version of the submitted publication.

5. If you have not established a formal collaboration with a local contact, at the time you submit a proposal, you should not include his/her name as a co-proposer.

6. Irrespective of special arrangements such as those under point 5, publications resulting from work at the BM08 beam-line have to contain an acknowledgment according to the following pattern:

- *We acknowledge the Italian CRG beam-line at ESRF (LISA-BM08) and we would like to thank [the name of your local contact] for assistance in performing the experiment.*

A similar acknowledgment should be included at conference presentations, including the proceedings, and at other public presentations.

7. We remind you that we need your cooperation in keeping track of all publications resulting from research carried out at the LISA-BM08 beam-line. This can be achieved by sending an e-mail to your local contact or directly to the beam line responsible, containing details of the reference as soon as it has been published or accepted. **Your publication record will also be made available to the referee committees for future applications for beam-time**.

12 Lisa phone numbers

- Beamline **2085**
- Sample preparation Laboratory **2487**
- **local contact 06 88 38 69 94** from the beamline phone dial 0 before the number