LISA BEAMLINE USERS GUIDE

The complete guide to the beamline

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A comprehensive guide to LISA

How not to feel alone when the local contact is unreachable

This document is oriented to users for their standard activity at the beamline. It contains info about the instrumentation and how to carry out the experiments.

The manual is not intended as an exhaustive guide for the X-ray Absorption Spectroscopy. Users should have at least a basic knowledge of XAS spectroscopy and on the related data analysis before performing the experiment.

Users are invited to read this document before coming at the beamline and to keep it with them during their experimental session.

Note that, since the beamline is continuously evolving, some of the issues described in the manual could be outdated.

It is important that you help the beamline staff keeping this manual useful by giving your feedback and suggestions.

💬 | Advice
If it ain't broke don't fix it.

1. General description of LISA

The LISA beamline

LISA is a beamline dedicated to X-ray Absorption Spectroscopy and related techniques.
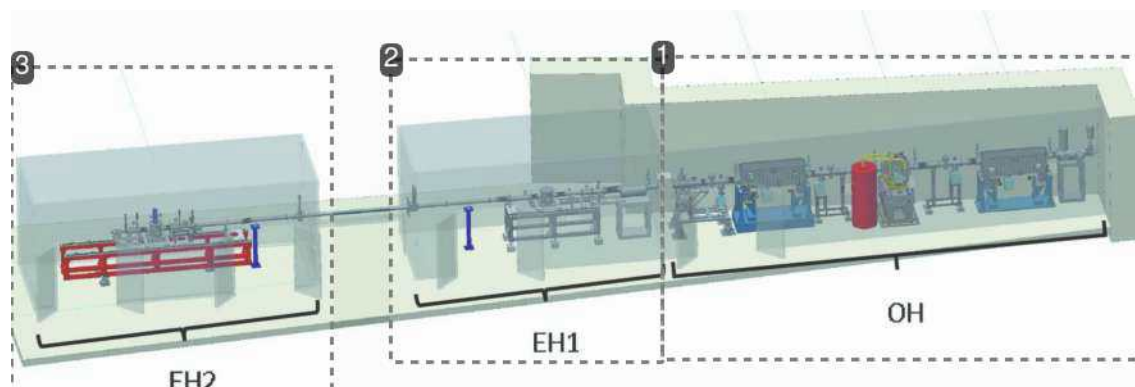
The X-ray optics consists in a collimating mirror, a fixed exit monochromator and a focusing mirror.

The main parameters are:

|	
Energy Range	4.0 - 90 KeV
Energy resolution (ΔE/E)	$1.3 * 10^{-4}$ eV for Si(111) $1.9 * 10^{-5}$ eV for Si(311)
Spot Size On Sample	50 μm (H) × 100 μm (V) FWHM
Max flux at sample	$2 * 10^{11}$ ph/s @ 10000 eV Si(111)

2. Beamline layout

LISA consists of three lead hutches: the optic hutch (OH) containing the main optical elements, the first experimental hutch (EH1) with the instrumentation for experiments with a non-focused beam and low-energy harmonic rejection mirrors, the second experimental hutch (EH2) containing all the instrumentation for experiments with a focused beam [F. d'Acapito et al., J. Synchrotron Rad. (2019). **26**, 551-558].

1. Optic Hutch, OH

2. First experimental hutch, EH1

3. Second experimental hutch, EH2

2.1. Optic Hutch

The X-ray optics consist of two mirrors and one monochromator. The first cylindrical mirror (positioned before the monochromator) collimates the beam in the vertical plane. The double crystal monochromator (DCM) allows the access of a wide energy range (4−90 keV), which offers the possibility to probe the K- and L- edges of most of the elements heavier than calcium (Ca). The DCM contains two liquid nitrogen cooled crystal pairs: Si(311) for experiments needing high energy resolution, and Si(111) for experiments needing high photon flux. The second mirror (toroidal, placed after the DCM) focalizes the beam about 17 m downstream on a roughly circular spot of diameter ≤100 µm. Both mirrors are made of a single crystal Si substrate with two optical regions: one exposing the silicon, and the other coated with Pt. The mirrors operate at a working angle of 2 mrads, with resulting energy cut-off of about 15 and 40 keV for the Si and Pt stripes, respectively [F. d'Acapito et al., J. Synchrotron Rad. (2019). **26**, 551-558].
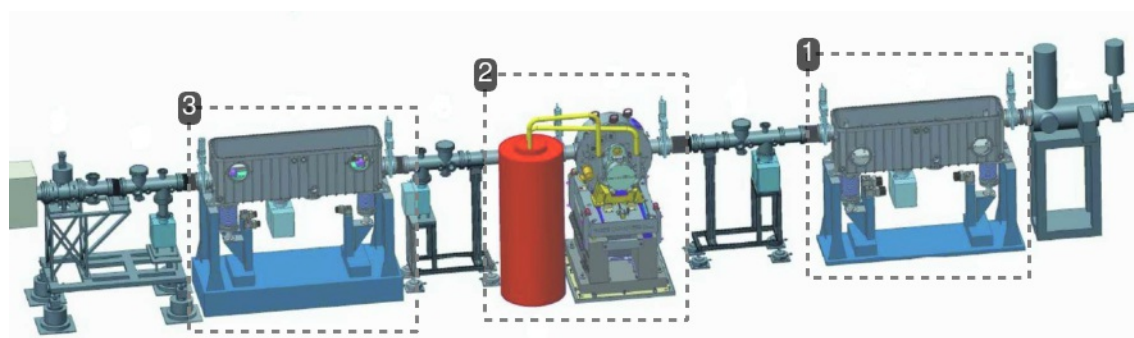
1. cylindrical collimating mirror

2. monochromator and LN2 cooling system

3. toroidal focusing mirror

> ⚠ | Warning
> The user is not allowed, nor requested, to access the optic hutch. For any intervention here you must contact the LISA staff or call the Experimental Hall Operator (2525).

2.2. Experimental Hutch 1

The first experimental hutch EH1 is dedicated to experiments where a non-focused beam (dimensions of ~1 mm × 2 mm) is preferable, mainly EXAFS in transmission mode. The hutch contains a vacuum chamber with a manipulator for the sample preceded and followed by ion chambers [length 150 mm (I_0) and 400 mm (I_1)]. These detectors are parallel plate chambers that can be filled with N_2, Ar or Kr at a pressure varying from 0.1 to 1 bar. A manual distribution of gases is available for the users. The experimental chamber is equipped with a cold finger LN2 manipulator 'Trento' (100 K < T < 525 K), which can be exchanged with a bellow manipulator or a second cold finger He/LN2 manipulator 'Oxford', usually mounted in EH2 (fig. 1).
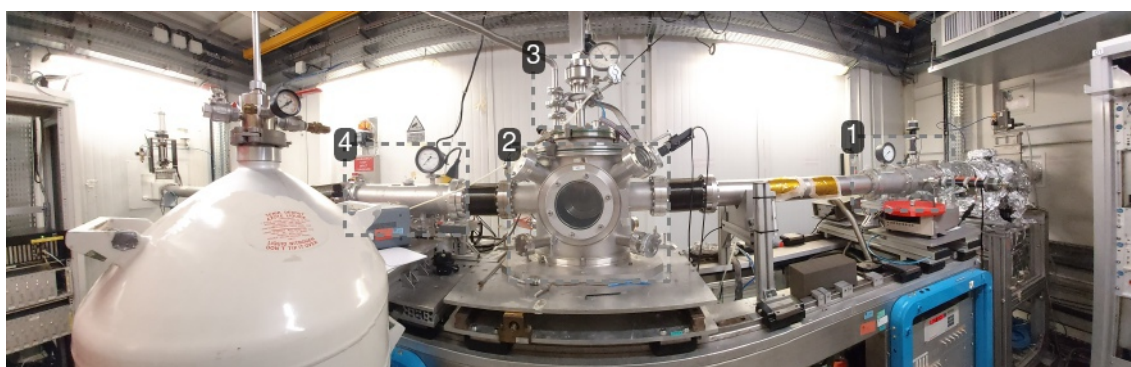
1. Ionization chamber *I0_eh1*

Measure of the incoming beam intensity *I_0*

2. Sample chamber

3. Cold finger manipulator

4. Ionization chamber *I1_eh1*

Measure of the intensity transmitted after the sample *I_1*

The principal motors in EH1 (fig. 1) are:

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

If the Trento manipulator is installed, *cryotr1* moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

If the bellow manipulator is installed, *vbell* moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

If the Oxford manipulator is installed, *vcof1* moves the sample holder vertically. Positive (negative) values move the sample holder up (down).
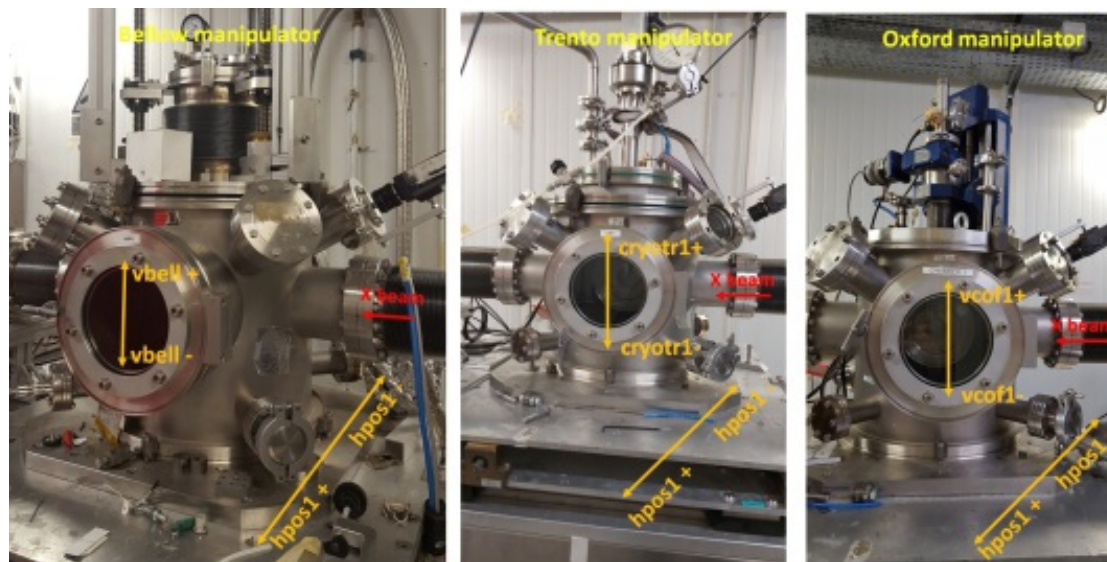

Figure 1: Sample chamber in EH1. Left): Bellow manipulator mounted. Center): Trento manipulator mounted. Right): Oxford manipulator mounted.

2.3. Experimental Hutch 2

The second experimental hutch EH2 is dedicated to experiments with focused beam. The instrumentation for data collection in EH2 consists of three ion chambers of length 100 mm similar to the ones previously described. Also here the gas type and pressure regulation is realized via a manually operated gas distribution line. For the collection of fluorescence from the samples, two high-purity germanium detector arrays (one with 12 elements, the other with 13 elements) are available for high energies (E > 15 keV) whereas in the lower-energy range a four-channel silicon drift detector (SDD) is used (ARDESIA project; [I. Hafizh *et al*., *JINST* **14** P06027 (2019)]). Experiments are carried out in vacuum chambers with manipulators hosting cold finger cryostats (temperature range from room temperature to 20 K) or a high-temperature reactor cell (MICROTOMO; [Bellet et al., J. Appl. Cryst. **36**, 366−367 (2003)]) up to 1300 K.
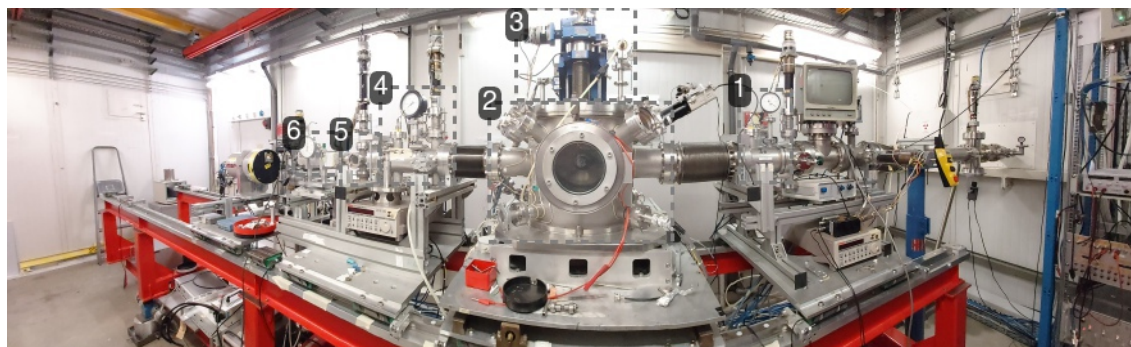
1. Ionization chamber *I0_eh2*

Measure of the incoming beam intensity I_0

2. Sample chamber

3. Cold finger cryostat

4. Ionization chamber *I1_eh2*

Measure of the intensity transmitted after the sample I_1

5. Chamber for reference standard

6. Ionization chamber *IX_eh2*

Measure of the intensity transmitted after the reference sample I_x

Below are listed the principal motors in EH2 (fig. 2):

If the Oxford manipulator is installed, **vcof** moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

If the Trento manipulator is installed, **vcryo** moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

If the bellow manipulator is installed, **vbell2** moves the sample holder vertically. Positive (negative) values move the sample holder up (down).

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

rot2 (only when the Oxford manipulator is mounted) rotates the sample holder. Positive angles orient samples towards the detector. *0* value corresponds to the beam perpendicular to the sample holder.

ydet moves the fluorescence detector mounted behind the sample chamber closer or farther the samples, values range from *0* to *200* 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).
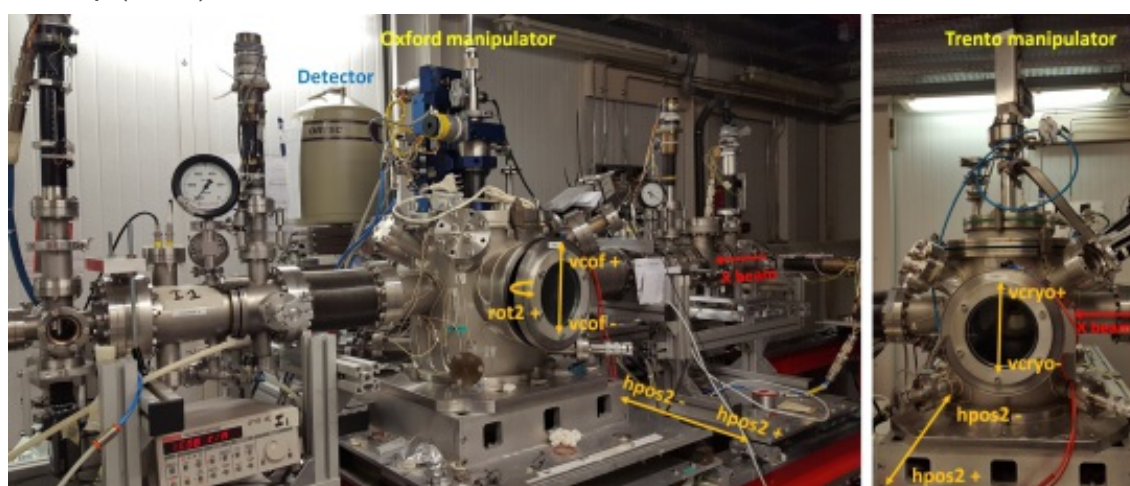
Figure 2: Main motors in second experimental hutch EH2. Left): Oxford manipulator mounted. Right): Trento manipulator mounted.

3. Beamline Control

Control room

The control of the whole experimental setup is made via **SPEC** with graphical user interfaces for easier operation.

1. ld082 PC

Control PC

This PC runs the software (SPEC, python) for the control of beamline optics, motors and for data collection. It contains tailor made apps with GUI for easier operation.

2. wbm08pc1 PC

Users PC

PC dedicated to user needs (internet connection, data analysis, …). It is equipped with the most common data analysis and graphic packages. You can transfer the acquired spectra with the program WinSCP installed in the Wbm08pc1 PC, which allows you to connect to the user data folder on the acquisition PC.

3. Camera monitors

4. Voltmeters

They display a voltage proportional to the intensity of the X ray beam.

4. Detectors

The LISA beamline is equipped with several types of detectors used for different techniques (fluorescence, XEOL, TEY). Below a brief description of each detector is presented.

4.1. HP-Ge detectors

For the collection of fluorescence from the samples two High Purity Germanium (HPGe) detector arrays, one with 12 elements 'Ruby', the other with 13 elements 'Nicole', are available. In both cases the readout is made by digital analysis of the output pulses using the XIA-DXP system. The detectors can be easily installed on a platform behind the sample chamber in EH2 and can be moved closer or farther the sample via the motor *ydet* (see section 2.3 and figure 3).

Figure 3: *12 element HP-Ge fluorescence detector*

💡 | A tip
   | before sending the beam on the sample for the first time push back Ruby at *ydet*=50mm

⚠️ | Warning
   | The count rate should never exceed 80 kcps. Higher count rates could damage the detector.

4.2. SDD detector

A 4 channels Silicon Drift Detector (SDD) is available (ARDESIA project, [I. Hafizh *et al*., *JINST* **14** P06027 (2019)]). This detector is particularly effective for fluorescence experiments at E < 15 keV, offering high count rates (up to 1Mcps/channel) and high energy resolution (down to 150 eV FWHM). The detection module consists of 2 × 2-pixel monolithic SDD coupled with a 4-channel version of the CUBE CMOS preamplifier. The instrument has been realized to fit inside the sample chamber with a finger-like structure and allows to operate in vacuum. It can be moved closer or farther the sample via the motor **ydet** (see section 2.3). A built-in Peltier system grants proper cooling (-30 °C). The readout is made by digital analysis of the output pulses using the XIA-DXP system.

Figure 4: the 4 elements SDD detector.

⚠️ Warning

The count rate should never exceed 80 kcps. Higher count rates could damage the detector. The detector should not go warmer than -17 °C.

4.3. Total Electron Yield detector

The Total Electron Yield apparatus allows to probe up to six samples, with the possibility to collect simultaneously florescence and TEY spectra. The main characteristics are:

- Secondary electrons collector, anode at V ≈ 45 V
- He gas electron multiplier
- Multi-sample holder (up to six samples)

The device is placed in the measurement chamber filled by He gas (0.25 bar) for signal amplification. The samples are placed onto the carbon tapes (Fig. 5) for a better connection to ground. The incident X-ray beam (red dashed line) passes through the front window and impinges directly upon the sample. The Auger electrons emitted from the sample surface ionize the He atoms creating a cascade. The total electron yield (Auger and secondary electrons) is collected by the anode (right-hand side of Fig.5) kept at a moderate voltage (≈ 45 V in the present case). The resulting current is converted to a voltage signal via

a Keithley amplifier. Due to the short mean free path of the electrons, TEY measurements allow to probe a material close to its surface, this is useful in case of thin films on a substrate, thick samples or when fluorescence measurements are not possible because of the large emission background from the matrix or spurious Bragg reflections.

Figure 5: Total Electron Yeld device.

4.4. XEOL

A system for the collection of XAS-XEOL data is also available: it consists in a lens (BK7 glass) collecting the emission from the sample coupled to a silica optic fiber. The system is mounted inside the measurement chamber and can operate in vacuum and with the sample at LNT. The fiber runs out of the chamber via a feedthrough and it shines a fast photomultiplier tube (PMT, model HAMAMATSU H3164-10) sensitive in the range 300-650 nm. Band-pass optical filters (bandwidth 10nm) can be mounted between the tip of the fiber and the PMT in order to select the desired optical emission band. When working in 16b or 4b mode the PMT pulses can be windowed by a fast gate (Philips mod 744) synchronized with the x ray flashes, thus realizing a time resolved data collection. Samples for XAS-XEOL are prepared in pellets with a moderate absorption ($\Delta \mu \approx 0.1$) in order to obtain a good optic signal and at the same time a spectrum in transmission mode.

4.5. Ionization chambers

The ionization chamber consist of a gas-filled chamber with two parallel plates electrodes.

An high voltage is applied between the electrodes to create an electric field in the fill gas. When gas atoms or molecules between the electrodes are ionized by incident X rays, ion-pairs are created and the resultant positive ions and dissociated electrons move to the electrodes of the opposite polarity under the influence of the electric field. This generates an ionization current in the region of nanoamperes to microamperes, which is fed to a Keithley current amplifier and converted into a voltage proportional to the beam intensity.

Depending of the energy of the X ray beam, different gasses are used to fill the chamber to achieve an optimal absorption: N_2 for the energy range 4 - 15 KeV; Ar for 15 -35 KeV ; Kr above 35 KeV.

There are 5 ionization chambers at LISA: *I0_eh1* and *I1_eh1* located in EH1; *I0_eh2, I1_eh2, IX_eh2, located* in EH2. They are described in sections Experimental Hutch 1 and Experimental Hutch 2.

⚠ Warning

The gas filling has to be done manually. Unless you are an experienced user, ask the local contact to set up the ion chambers for you.

⚠ Warning

Before changing the ion chamber gas, always switch off the corresponding Keitheley and set the high voltage to zero

4.5.1. How to change the Keithley gain

You must set the Keithley current amplifiers gain in order to keep the ionization chambers read-out voltage in the linearity range **0.5 - 8.0 Volts**. Outside this range the level of the noise will dramatically increase. Moreover above 8 V the chambers response becomes strongly non linear, resulting in serious artifacts in the collected spectra.

It is possible to remotely change or read the gain of the I0_eh2, I1_eh2 and IX_eh2 Keithley from the control room.

To read the gain of all the Keithley, use the command *kshow* in the eh2 SPEC session (light yellow terminal).

To change the gain of a Keithley, type *k428set_gain <Keithley_name> <value>*

As an example to set the gain of I0_eh2 to 10^7 type:

eh2> k428set_gain I0_eh2 7

The gain value can be any integer between 5 and 10.

To make the zero compensation (after changing the gain):

eh2*> shclose*

eh2*> k428zero_corr <Keithley_name>*

eh2*> shopen*

The Keithley I0_eh1 and I1_eh1, located in EH1 have to be operated manually. To change the gain rotate the gain wheel.

5. Experiments

This section covers the basic information necessary to perform the XAS measurements.

5.1. Mounting the samples

You can mount samples (pellets, membranes, films...) on a standard copper or aluminum sample holder (see figure 6). The top of the holder has a dovetail shape to fit inside the experimental chamber.
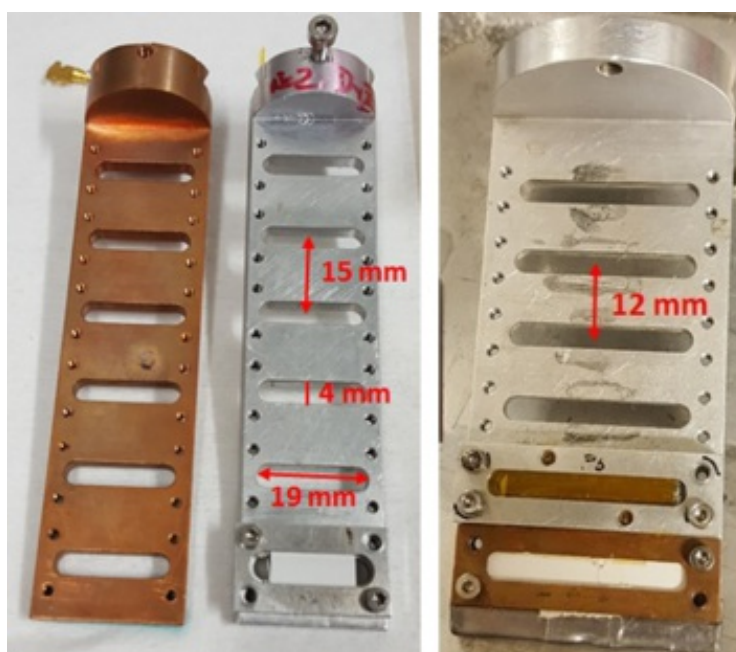

Figure 6: Sample holders for EH2 (left) and for EH1 (right).

The sample holder can host up to 6 samples which size not exceeding 15 mm in height. 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.

5.2. Vacuum operations

Low vacuum operations in EH1 (few mbar) (only for room temperature measurements)

Pump the sample chamber in EH1

1. close the experimental chamber port

2. close the experimental chamber venting valve (placed under the bench behind the respective turbo pump)

3. On the panel on the right of the hutch press the black button marked ROT CH 1 to start the pump

4. Move the black toggle switch marked ELEC V CH 1 in position On, to open the electro valve

5. Wait that the vacuum is reached (few mbar or less)

Stop the low vacuum in EH1 sample chamber

1. On the panel on the right of the hutch move the black toggle switch marked ELEC V CH 1 in position Off, to close the electro valve

2. Press the red button marked ROT CH 1 to stop the pump

3. Open slowly the experimental chamber venting valve.

4. When the chamber is at atmospheric pressure you can open the port

High Vacuum operations in EH1 sample chamber (necessary to work at low temperature)

Pump the sample chamber in EH1 to high vacuum

- Follow the steps 1 to 5 for pumping the chamber to low vacuum

- When the pressure inside the chamber is few mbar or less, press the start button of the turbo pump controller

- Wait that the pump controller is in normal operation.

Stop the high vacuum in EH1 sample chamber

- Press the stop button on the turbo pump controller

- Follow the steps 1 and 2 for stopping the low vacuum

- Wait about 5 minutes, then open **very 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

1.

Venting valve

2.

Rotative pump start and stop buttons
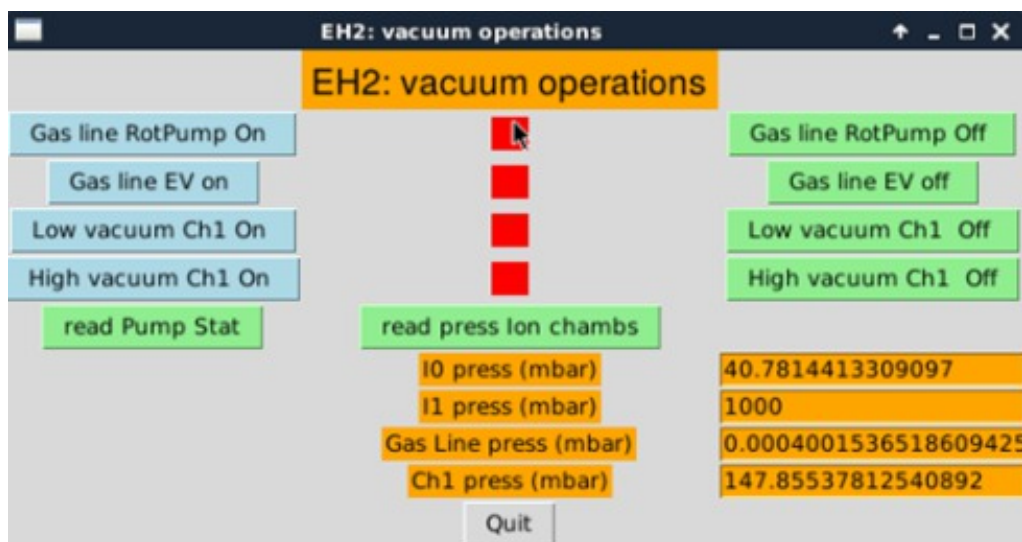
3.

Electro valve toggle switch

4.

Chamber pressure gauge (channel 6)

5.

Turbo pump controller

Vacuum operations in EH2

In EH2 the vacuum operations can be done remotely with the dedicated GUI *'EH2: vacuum operations'* on the ld082 PC.

EH2 vacuum operations main window

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

Pump the experimental chamber in EH2

- close the experimental chamber port
- close the experimental chamber venting valve (placed under the bench behind the respective turbo pump)
- press the button **Low vacuum Ch1 On**
- the program will start the rotative pump and will prompt when the chamber is in vacuum.

Stop the low vacuum in EH2 sample chamber

- press the button **Low vacuum Ch1 Off**
- open slowly the experimental chamber venting valve.
- when the chamber is at atmospheric pressure you can open the port.

High Vacuum (necessary to work at low temperature)

Pumping the experimental chamber in EH2

- Close the experimental chamber port
- close the experimental chamber venting valves (placed under the bench behind the respective turbo pumps)

- press the button **High vacuum Ch1 On**
- 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.

Stop the high vacuum in the experimental chamber in EH2

- press the button **High vacuum Ch1 Off**
- after about 5 minutes, open **very 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
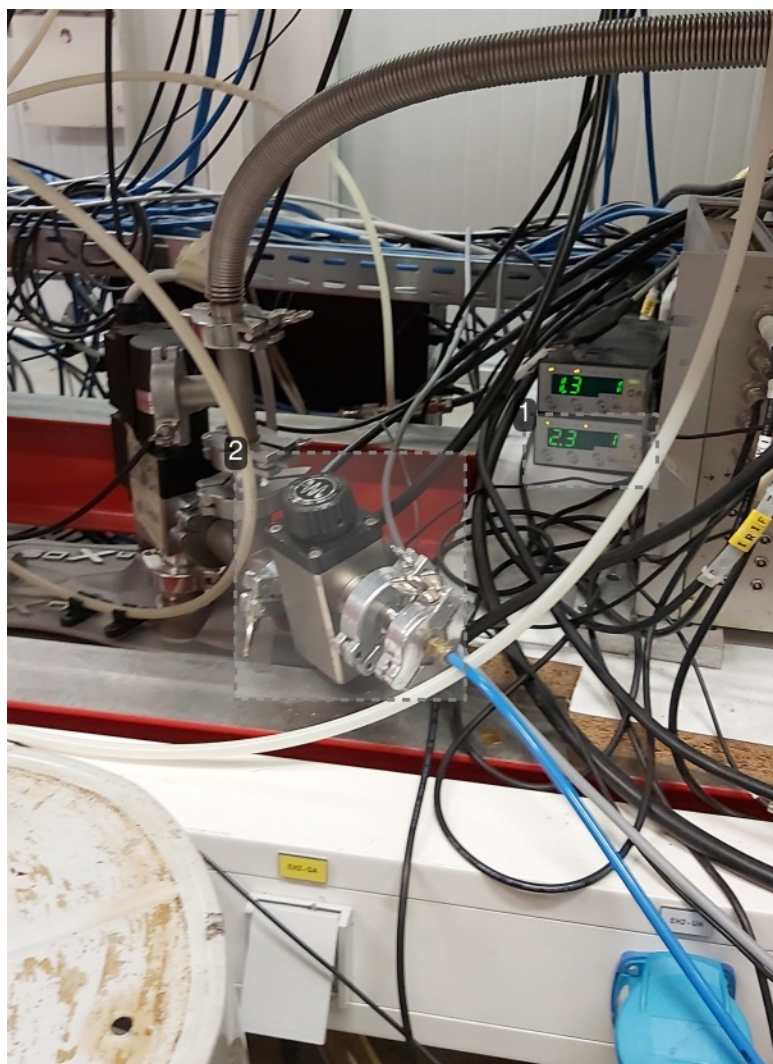
1.

Chamber pressure gauge

2.

Venting valve

> ⚠️ **Warning**
>
> If the turbo pump was on you will have to stop it and wait that the deceleration is complete before opening the venting valve. Always open the valve very slowly to avoid damaging the turbo pump. **Opening the valve with the turbo pump on will damage the turbo pump.**

> ⚠️ **Warning**
>
> NEVER try to force open the sample chamber when it is under vacuum. Always wait that the chamber is at atmospheric pressure before opening the port.

5.3. Cooling the samples

Cooling the samples with Trento manipulator (usually in EH1)

1. Close the sample chamber and start the high vacuum following the procedure described in section ***Vacuum Operations***

2. Set the desired temperature remotely with the command ***umv euro_sp*** <value> on the light green window XAS, or by adjusting manually the setpoint on the eurotherm controller.

3. Start the N2 pumping by switching on the cryostat pump and opening gradually the valve until the pressure gauge reaches the higher depression (about -0.6 -0.8 bars). The temperature should start to decrease (typical rate 0.1 K/s).

4. When the set temperature is reached, in order to optimize the nitrogen consumption you must reduce the depression by gradually closing the pumping valve until the depression stabilizes to a value around -0.2 bars.

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

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

Heating the samples with Trento manipulator to room temperature

1. Close the pumping valve

2. Stop the N2 pumping by switching off the cryostat pump.

3. Set the temperature to 295 K with the command ***umv euro_sp 295*** on the light green window ***XAS***, or by adjusting manually the setpoint on the eurotherm controller.

4. 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).

5. Once the samples are at room temperature, you can stop the high vacuum and vent the chamber (see section vacuum operation). **NEVER open the venting valve at low temperature!**

1.

Eurotherm controller

2.

Pump valve

3.

Pressure gauge

4.

LN2 pump

Cooling the samples with Oxford manipulator (usually in EH2)

1. Close the sample chamber and start the high vacuum following the procedure described in section *Vacuum Operations*

2. Set the desired temperature remotely with the command *umv temp <value>* on the light green window XAS, or by adjusting manually the setpoint on the oxford controller.

3. Start the He2/N2 pumping by switching on the cryostat pump and opening gradually the valve until the pressure gauge reaches the higher depression (about -0.6 -0.8 bars). The temperature should start to decrease (typical rate 0.1 K/s).

4. When the set temperature is reached, in order to optimize the nitrogen consumption you must reduce the depression by gradually closing the pumping valve until the depression stabilizes to a value around -0.2 bars.

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

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

Heating the samples with Oxford manipulator to room temperature

1. Close the valve indicated by the arrow in figure .

2. Stop the He2/N2 pumping by switching off the cryostat pump.

3. Set the temperature to 295 K with the command ***umv temp 295*** on the light green window ***XAS***, or by adjusting manually the setpoint on the oxford controller.

4. 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).

5. Once the samples are at room temperature, you can stop the high vacuum and vent the chamber (see section vacuum operation). **NEVER open the venting valve at low temperature!**

1.

Oxford controller

2.

LN2/He pump

3.

Pump valve

4.

Pressure gauge

💬 | ***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…).

5.4. How to align the samples

Setting the energy and checking the beam intensity

The monochromator energy is a SPEC pseudo-motor with a position defined in keV. As an example, under XAS window 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 voltmeter. If you have zero intensity, check the machine status on the workspace ***Machine and Vacuum Status*** and control that the beamline shutters are open.

> ✎ Note
>
> **The voltage reading must be 0.5 < V < 8**. Beyond these values the Ion chambers loose their linearity, and you risk to collect distorted spectra.

5.4.1. XAS

To align the samples with the beam, do a position scan. The scan are done from eh1 or eh2 window for the hutch EH1 and EH2, respectively.

EH1 motors:

- cryotr1 (or vcof1 or vbell depending on the installed manipulator) for vertical position
- hpos1 for horizontal position

EH2 motors:

- vcof (or vcryo or vbell2 depending on the installed manipulator) for vertical position
- hpos2 for horizontal position
- rot2 for rotation (only for the Oxford manipulator)

> 👁 Position scan in EH1 (EH2)
>
> For vertical position scan in **eh1 (eh2)** use the command:
>
> ***dscan cryotr1 (vcof) -5 5 20 1***
>
> For horizontal scan use:
>
> ***dscan hpos1 (hpos2) -5 5 20 1***
>
> First two parameters are the position interval relative to the starting position in mm, 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 is 1 s.
>
> You can also perform a scan between two absolute positions with the command ***ascan***, for example
>
> ***ascan vcof 3 18 40 1***
>
> Performs a vertical scan between absolute position 3 and position 18.

5.4.2. GIXAS

Vertical geometry

1. Rotate the sample holder parallel to the X-ray beam **(rot2** motor position **90)**, and make sure that the holder is out of the beam.

2. read the value of the voltmeter **I1**

3. Move motor **hpos2** until you read half of the **I1** initial value

4. Do a rotation scan (motor **rot2**) and move to the value corresponding to the maximum of the curve.

5. Repeat steps 2 to 4 until the rotation scan yields a cusp. The cusp marks the zero of the angular scan.

6. Move **rot2** to the value corresponding to the desired angle (usually 2°)

Horizontal geometry (GIXAS sample holder)

1. With the sample holder out of the X-ray beam, move the **rgixas** motor to position **0**

2. read the value of the voltmeter **I1**

3. Move motor **zgixas** until you read half of the **I1** initial value

4. Do a rotation scan (motor **rgixas**) and move to the value corresponding to the maximum of the curve.

5. Repeat steps 2 to 4 until the rotation scan yields a cusp. The cusp marks the zero of the angular scan.

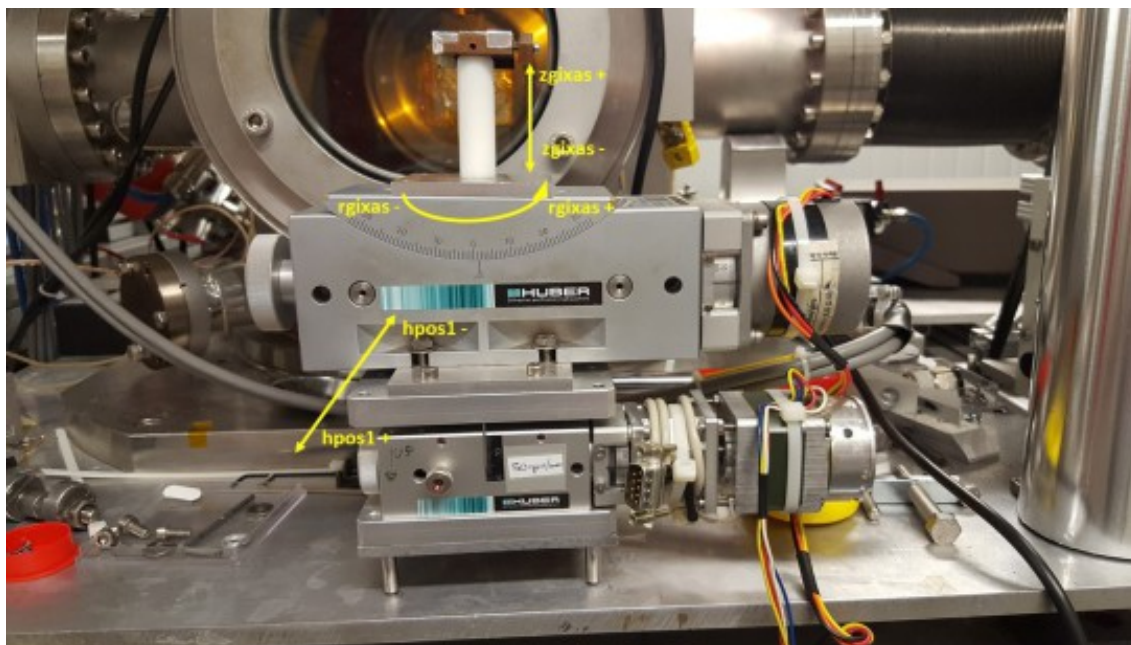6. Move **rot2** to the value corresponding to the desired angle (usually 2°)

Figure 7: The GIXAS sample holder

5.4.3. XEOL or optical P&P

1. Look at the sample camera monitor and check the X-ray beam position on the fluorescent screen.

2. Using the hpos2 and vcof motors align the X-ray beam spot on the mark on the fluorescent screen.

3. Enter the EH2 hutch and adjust the distance of the lenses until you obtain a nice light spot on the mark on the fluorescent screen.

4. Go back in the control room, open the shutter. You should see the light and X-ray spots at the same place on the fluorescent screen.
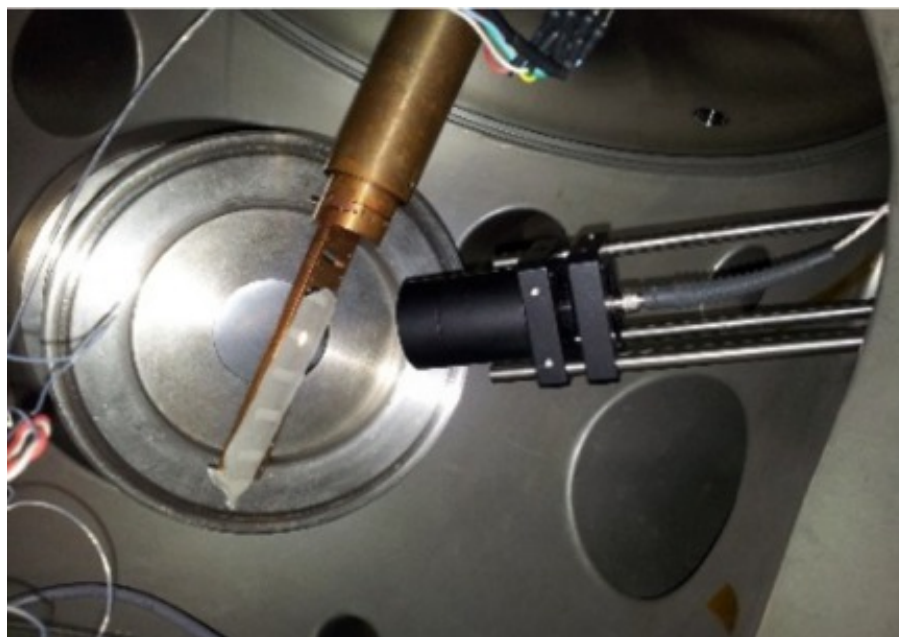

*Figure 8:* Sample holder and optics setup for XEOL

5.5. 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:

5.5.1. Kscan

The Kscan utility is used to acquire spectra in step mode, i.e. by moving to an energy value and counting a given amount of time, then moving to the next energy value and so on. This utility allows multiple scan regions to be set up, with different parameters in each, as well as scan regions where the scan mesh is equally spaced are in 'k space' rather than in energy.

You can choose your scan preferences with the kscan setup GUI:
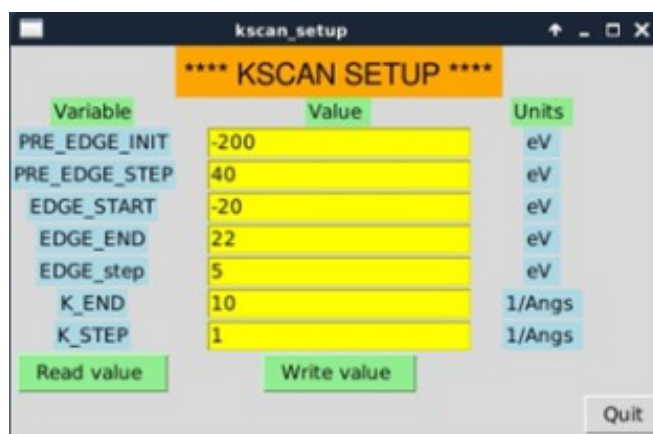
Kscan setup main window

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, press the button 'Write value'.

Now you can use the Kscan utility to collect the spectra.

Kscan data acquisition main window

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. **No longer used, leave it to -1**

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. **No longer used, leave it to -1**

Chamber. 1 or 2 select EXAFS Hutch EH1 or EH2

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.
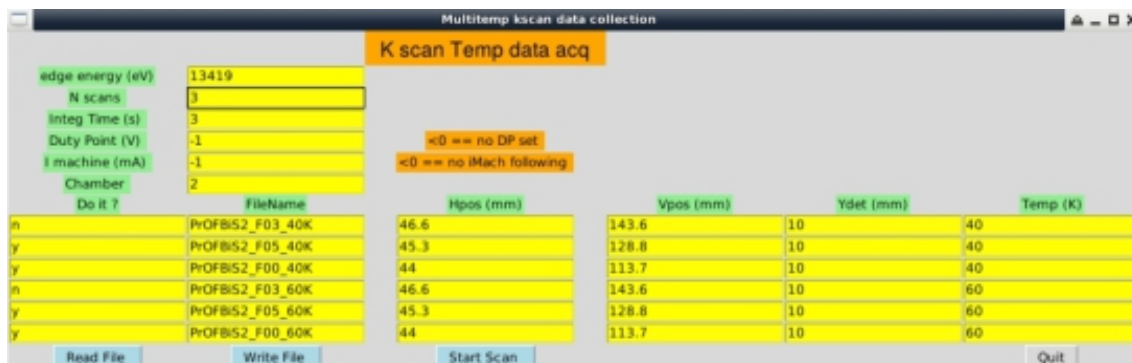
> ⚠️ **Warning**
>
> The filename must not contain spaces or special characters. Only _ or - are allowed. Otherwise the acquisition will not start.

> ⚠️ **Warning**
>
> If N scan >1 the program will repeat the scan automatically adding a _2, _3, _# at the and of the filename. However if you restart an acquisition always remember to change the filename, otherwise you will end with multiple spectra in the same data file.

5.5.2. Multitemp kscan

It is possible to acquire spectra at different temperatures in automatic way, in the range 20 K to 400 K. A SPEC routine has been developed, which can change temperature, check temperature stability and perform the scans automatically. A user GUI is available, which can collect spectra at up to six different temperatures.


Do it ?	FileName	Hpos (mm)	Vpos (mm)		Ydet (mm)	Temp (K)
n	PrOFBiS2_F03_40K	46.6	143.6	10	40	40
y	PrOFBiS2_F05_40K	45.3	128.8	10	40	40
y	PrOFBiS2_F00_40K	44	113.7	10	40	40
n	PrOFBiS2_F03_60K	46.6	143.6	10	60	60
y	PrOFBiS2_F05_60K	45.3	128.8	10	60	60
y	PrOFBiS2_F00_60K	44	113.7	10	60	60

Multi temperatures Kscan main window

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

5.5.3. ContScan

Spectra acquisition in quick mode is available at LISA, through a SPEC routine where the monochromator Bragg angle is moved continuously, while fast counting the ion chambers. A dedicated user GUI *'Cont scan'* is also available which allows to choose the energy scan interval and the duration of scan. A complete EXAFS spectrum can be acquired in few minutes.
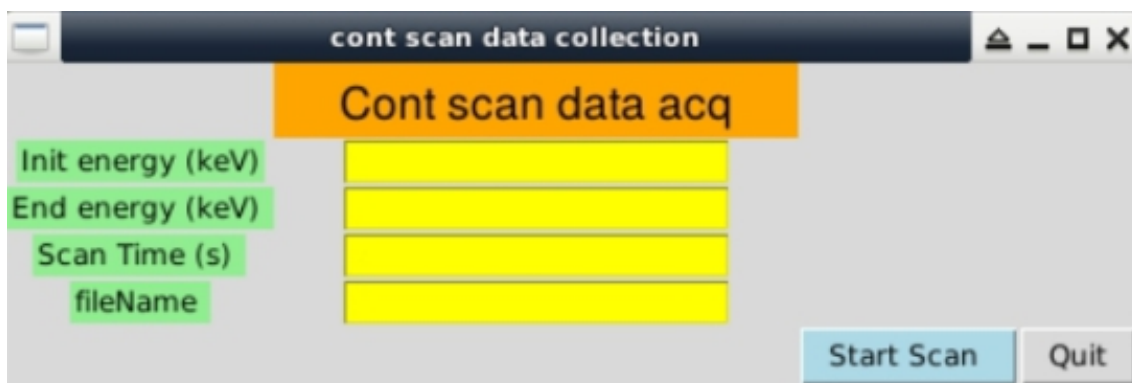
Contscan main window

Init energy: Starting energy value in keV

End energy: final energy value in keV

Scan Time: time for the whole spectrum acquisition in seconds (typical values are 240 s)

Filename: name of your data file.

⚠ Warning

Switch off XIA with the command *xiaoff* on the XAS terminal before using contscan

5.5.4. P&P

A dedicated macro for pump & probe measurements has been implemented at LISA. It allows time-gated point-by-point data acquisition triggering an external stimulus in order to compensate eventual drifts in the acquisition setup over long-term measurements. The macro is launched from the **XAS** terminal and is called **LISAKscan2pp.**

Usage: LISAKscan2pp <energy_motor> <edge_energy> <time_factor> <dutyPoint> <initCurrent>
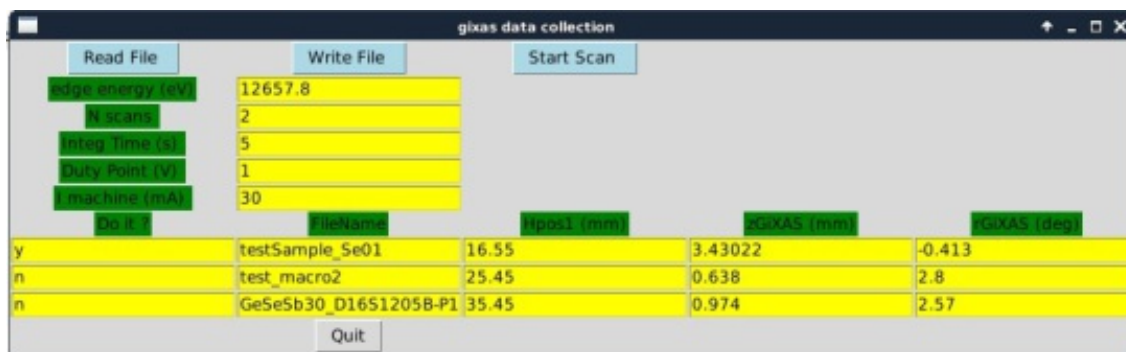Example: **XAS> LISAKscan2pp energy 7709 10 -1 -1**

5.5.5. GIXAS

Vertical geometry

Once the sample is aligned for GIXAS (see section How to align the samples) the acquisition can be done using the standard Kscan acquisition program.

Horizontal geometry (GIXAS sample holder)

In this case you can use the **gixas data collection** utility.

GIXAS data collection main window

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.

5.5.6. Pause or stop an acquisition

When the Kscan GUI is used, you can pause or interrupt the spectra during the acquisition using the buttons on the right cupboard in the control room. 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.
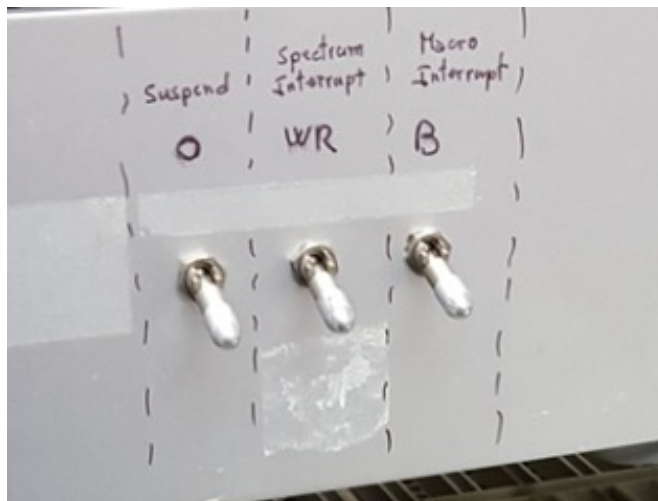
*Figure 9: Buttons for pausing/stopping the acquisition*

Alternatively you can press Ctrl+C to stop the entire macro.

5.6. Plotting 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*, **I0_eh1(2)** as *Y* and **I1_eh1(2)** as *Mon* for the spectra acquired in EH1(2) with the Kscan utility.

In the case of fluorescence spectra select as *Y* the data columns corresponding to the detector channels, named **fluo01, fluo02… fluo12** and **I0_eh2** as *Mon.*

If you want to plot the reference select the columns **I1_eh2** as *Y* and **IX_eh2** as *MON*
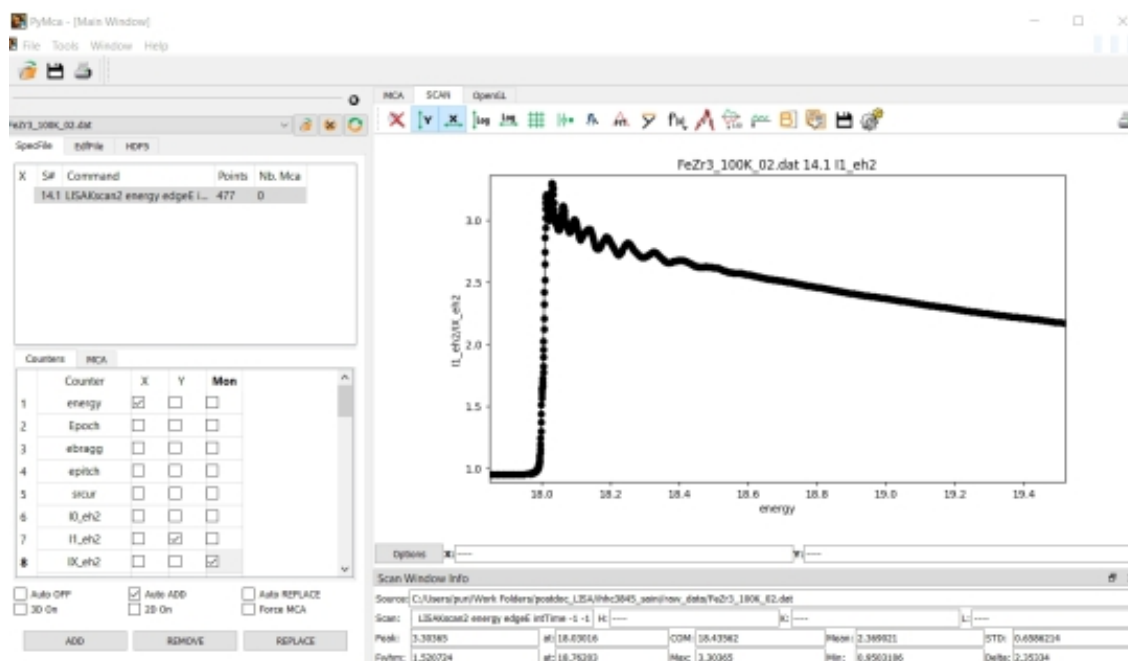
PyMCA main window

6. Data treatment

How to transfer experimental files

Experimental files are located in a working folder in the acquisition PC created for your experiment. Your working folder should have the name of the experiment code you have been assigned. The folder path is the following:

/mnt/multipath-shares/data/visitor/your-experiment-code/bm08/s1/s1_ds1

The user PC Wbm08pc1 has installed a program called WinSCP, which allows you to connect remotely to the data folder on the acquisition PC. Navigate to your folder referring to the above path and transfer your files into a working folder you created in USERS folder on the user PC. From the user PC you can copy the files using a USB stick or upload them on your cloud drive.

How to pretreat and reduce data

Raw XAS data can be reduced to a simpler user friendly format using the following python codes:

dconv_multiV2.py for transmission or fluorescence data.

dconv_multi_ppV3.py for pump and probe data.

Double click on the *.py* file to start the program

The program takes a *.txt* configuration file named **dconv_config.txt** which contains users parameters.

The output is a file with the structure:

Calculated Energy (eV), I0, I1, mu, (fluo channels if applicable), experimental energy (ev)

That can be easily treated with standard data extraction codes.

Configuration file dconv_config.txt

7. Final remarks

Local Contact Policy

1.One staff 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.

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 during regular working hours in case of a problem (8:00-17:00 on weekdays) and outside regular working hours (17:00-22:00 on weekdays, 08:00-22:00 on weekends and holidays),**

·**provide specific help in certain cases (including outside regular working hours) through contacts with the Hall Operators.**

In case of major difficulty, and if your Local Contact is not present on the beamline, you should contact the **Hall Operator**, who will decide what kind of action can be taken.

4. You are required to have at least basic experience in information technology, XAFS technique, 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. Publications resulting from work at the BM08 beam-line should 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. By applying for and accepting beam time at the ESRF you accept to properly acknowledge all ESRF data in your publications. According to the ESRF data policy you must therefore cite the DOIs of the data in all publications which use this data, even if the data is still under embargo at the time of publication.

8. 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**.

See also https://www.esrf.fr/UsersAndScience/UserGuide/Preparing/LocalContact

How to contact LISA staff

Internal numbers

Name	Phone	Skype	Mattermost
F. d'Acapito	2426	dacapitodac	@dacapito
A. Puri	2859	alessandro_puri	@alessandropuri
LISA lab	2743	LISA@ESRF	@opd08
Beamline	2085		

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