GILDA

USERS'

MANUAL

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INTRODUCTION

This manual describes the standard beamline equipment and procedures to be used to acquire absorption spectra.

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.

Your local contact will teach you all the procedures and control applications you will need to perform your experiment. Nevertheless, since it is difficult to learn how to control the beamline in the limited time of your experiment, this manual should serves as a reference guide to which you should refer when the local contact is not with you (night shifts, weekends…).

The manual should allows you to solve most of the problems you can encounter without calling your local contact, whom, outside working hours, should be available only for emergencies, such as major failures requiring procedures that are not reported in the manual.

Note that, since the beamline is continuously evolving, some of the issues described in the manual could be outdated.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

LOCAL CONTACT POLICY

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. The local contact will help you to perform your experiment. Nevertheless, remember that **your local contact is NOT there to perform the experiment for you.**
It is your obligation to provide sufficient staff to operate the experiment 24 hours a day (in any case a minimum of 2 people is strongly recommended). 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.
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 8.30 a.m. to 10.30 p.m.

You are required to have at least basic computer skills, experience in XAFS or XRD (depending on your experiment) techniques and the corresponding data analysis. In the case none of your research group members has 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 beam-line 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 collaboration,
- Include him/her as co-proposer,
- Include 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.
- 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.

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

We acknowledge the Italian CRG beam-line at ESRF (GILDA-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.

We remind you that we need your cooperation in keeping track of all publications resulting from research carried out at the GILDA 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.

GOLDEN RULES

LabView

1. NEVER run more than one Labview utility at a time(use the QUIT/EXIT button and leave the window open).

2. Quit ALL other Labview applications BEFORE acquisitions.

3. Never close Labview windows (except for graphic scan windows, which should be closed after a scan is completed).

Macros

1. Close ALL Labview applications BEFORE running a macro.

2. If you need to stop a macro, push the red button at the left top of the Labview window. Do not use the STOP buttons!

3. Move your acquired experimental files in a subfolder before running a macro. This way you will avoid the 'Overwrite file confirmation' warning message, which prevents the macro from going on.

Detector

1. Check once per day the nitrogen level (even if you are not using it) by checking that the green LED on the nitrogen level indicator (see page 55) is on.

2. If the 13-element detector stops working:

- Check that the 'Sample Holder' utility is quitted.
- Close and reopen the Xserver window on RITAPC1.

Front end

Remember to reset (by stopping/setting it) the AUTOMATIC MODE for the front end (see manual page 22) within 72 hours. This will prevent the front end from closing after 72 hours.

Servers

If you get a 'TCP error connection' warning message reset the servers as explained in the manual at page 81.

Sample preparation equipment

- Ball mill with two 5ml agate vials.
  Maximum accepted grain size: 2mm
  Minimum final size: 10microns
- Agate mortars 5, 6 and 10cm diameters
- Pellet dies (5 and 10mm diam.) and pellet press
- Two complete system for powder deposition on membranes (5 and 10mm)
- Oven, maximum temperature: 1100 C
- Atomic Force Microscope
- Optical microscope
- Glove box (inert atmosphere)
- Analytic balance
- Magnetic heater
- Chemical hut for volatile substances manipulation
- Ultrasound bath
- Pipettes, syringes, needles and cells to manipulate liquid samples
- Vacuum glass sample containers

Several fluids can be used for general purposes:

- Compressed air
- Industrial water
- Double demineralized water system
- Nitrogen Gas
- Gas evacuation line

Chemicals usually available in the lab:

- Boron Nitride, cellulose, graphite
- Poly-ethilene
- Ethanol, toluene, acetone

Should you need equipment not included in the above list, contact your local contact well before the experiment.

GENERAL DESCRIPTION

BEAMLINE SCHEME

OPTIC HUTCH
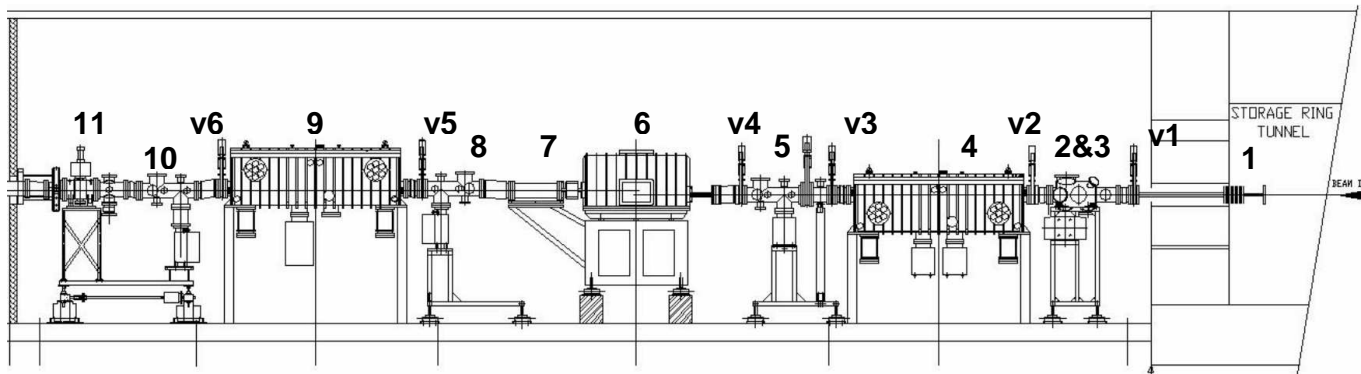
Figura e misure in mm

1.   FRONT END
2.   MAIN SLITS
3.   FILTERS AND BEAM MONITOR 0 (BM0)
4.   FIRST Pd/Pt HARMONIC REJECTION MAIN MIRROR
5.   BEAM MONITOR 1 (BM1)
6.   INPUT SLITS, MONOCHROMATOR, OUTPUT SLITS
7.   IONIZATION CHAMBER
8.   BEAM MONITOR 2 (BM2)
9.   SECOND Pd/Pt HARMONIC REJECTION MAIN MIRROR
10.   BEAM MONITOR 3 (BM3)
11.   EXAFS BEAM SHUTTER (B1)

vX ARE VACUUM VALVES.

EXPERIMENTAL HUTCH 1 (XAS)
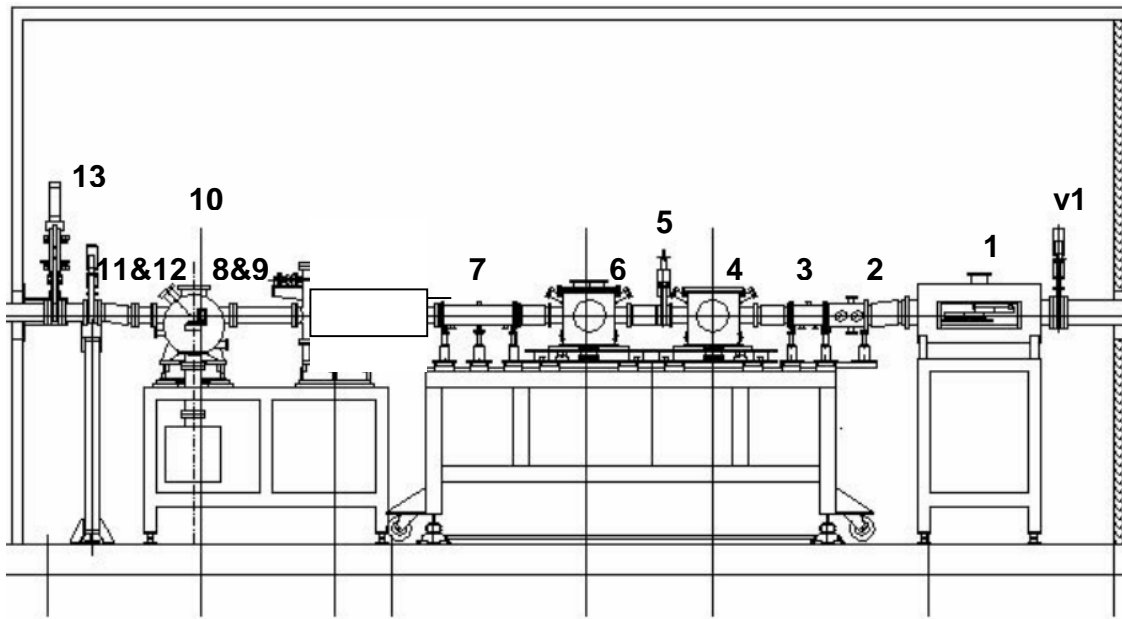
1. LOW ENERGY HARMONIC REJECTION MIRRORS
2. EXAFS SLITS
3. IONIZATION CHAMBER (I0)
4. EXAFS EXPERIMENTAL CHAMBER 1 (EXAFS CH1)
5. VAT VALVE
6. EXAFS EXPERIMENTAL CHAMBER 2 (EXAFS CH2)
7. IONIZATION CHAMBER (I1)
8. NORMALIZATION SLITS
9. IONIZATION CHAMBER (Inor.)
10. ReflEXAFS CHAMBER
11. REFLECTIVITY SLITS
12. IONIZATION CHAMBER (Irifl.)
13. DIFFRACTION BEAM SHUTTER (B2)

   vX ARE VACUUM VALVES.

EXPERIMENTAL HUTCH 2 (XRD)
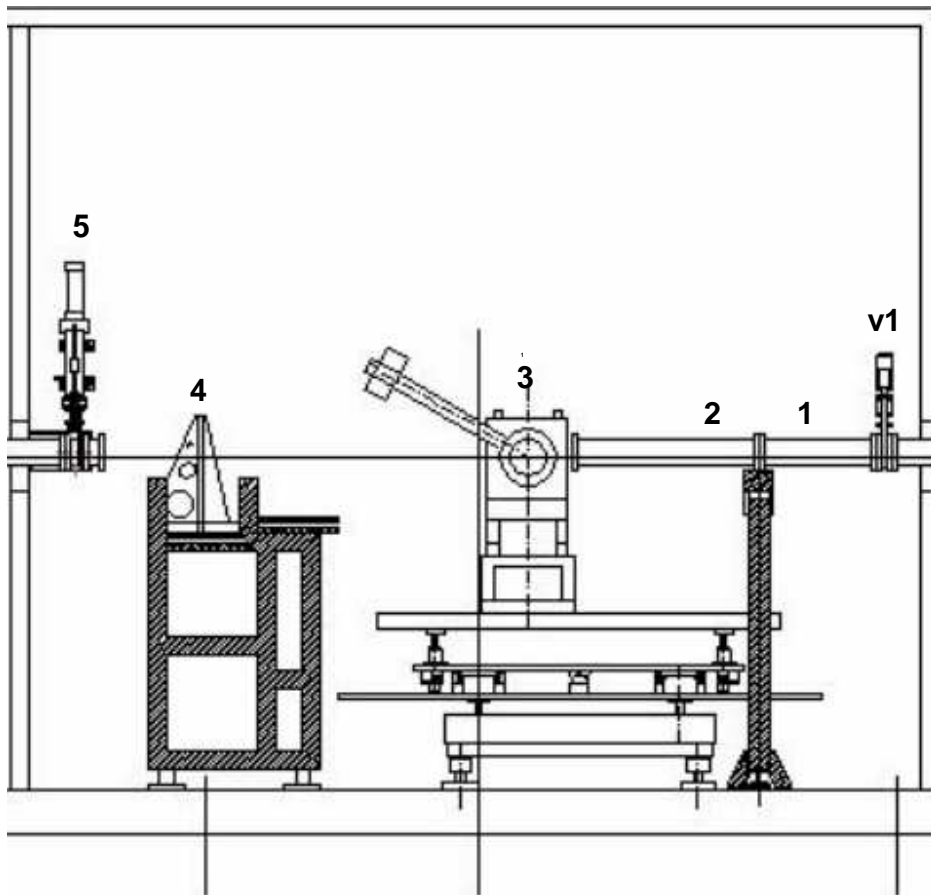
1. XRD SLITS
2. XRD IONIZATION CHAMBER
3. DIFFRACTOMETER
4. IMAGEPLATE
5. BEAMSHUTTER (B3)

vX ARE VACUUM VALVES.

CONTROL CABIN

GILDA's control cabin

BEAMLINE's PCs

OPERATION PCs

1. **RITAPCLINUX**
   OS: LINUX
   Location: control room

   This PC is dedicated to the acquisition and it runs the beamline control software (Labview) Acquired spectra are stored in a folder named EXAFS_USER_DATA, which has a shortcut on the desktop. A different subfolder is created for each experiment.

2. **RITAPCU1**
   OS: LINUX
   Location: control room

This PC is dedicated to the FRONT END and SECONDARY SHUTTERS operations.

3. **RITAPCU2**

OS: WINDOWS
Location: XAS experimental hutch

This PC runs the server for the 13-element detector (Xserver), controls ionization chambers operations (gas type and pressures, amplifications), vacuum operations, secondary slits operations and EXAFS CHAMBER 2 motion. This PC is usually accessed from the control room trough a display redirection (VNC) on RITAPC1 or on the acquisition PC.

4. **RITAPC1**

OS: WINDOWS
Location: control room

This PC it is usually only used to run the VNC connection with **RITAPCU2** (which is located inside the XAS exp. hutch).

WARNING The above PCs are intended for beamline operations ONLY! They run sensitive software and data and are critical for beamline operations.
Never install software or change any settings on those PCs.

1. **RITAPC** and **RITAPC3**
   OS: WINDOWS XP
   Location: control room

   These PCs are available for any users needing (internet connection, data analysis, entertainment...).
   They are equipped with the most common data analysis and graphic packages.
   Check with your local contact if what you need is already installed BEFORE installing new software.
   As a general rule you should create a folder in the USERS folder in order to limit the spreading of the files.
   Also remember that files left on the desktop will be periodically deleted.
   Both PCs have a network folder (EXAFS_USER_DATA_ON_RITAPCLINUX), shortcut in the taskbar at the top of the desktop, pointing to the user data folder on the acquisition PC, from which you can transfer the acquired spectra.

LABVIEW

Labview is the control software for beamline's operations and provides an intuitive graphical interface.
Each operation is controlled by a different Labview application ('.vi') linked in special folders on the desktop organized by tasks (EXAFS, REFLEXAFS, OPTIC HUTCH and MACROS). Once opened by double-clicking on the corresponding icon, applications must be launched clicking on the white arrow button at the top left side of the application window.
Since different applications may call the same server, more than one application active at a time can cause conflicts errors and crashes.

CONTROL PANELS

To avoid conflicts between applications, users will usually work with panels (applications launchers), which permit only one active application at a time.

WARNING

Except for the 'standalone' application (which control the 13-element detector) do not launch applications by doubl-clicking on the correspondent icons while a control panel is running!

Labview golden rules:

1) Labview applications are run by clicking the white arrow on the top left side of the application window. When working with panels applications are already run when loaded.

2) To close a application push the EXIT (or QUIT) button. Once exited, the window can be left open and another application can be launched.

If you cannot quit the application, click the red button at the top left of the application's window. Never close a running application by closing its window (except for graphic windows that pops up when performing scans).

EXPERIMENT LOGBOOK

The logbook is a fundamental part of the experiment. Even if you provide your own logbook, you are strongly encouraged to write down on the beamline logbook all the details of the experiment (beamline settings, sample environment, name and parameters of the spectra, samples description...). Remember that logbooks are permanently stored on GILDA, so you can access them at a following experiment or ask the GILDA staff to consult them for you at any moment.
It is also recommended to make a copy of the logbook before leaving the beamline.

CAMERA CONTROLLER

A 16 channels camera controller is located in the control room. Various cameras are placed in the hutches to see the sample holders, beam monitors and other equipment while the shutters are open.
There are three monitors: two CRTs placed above the acquisition PC and one connected to the video acquisition card installed on RITAPC. With the latter it is possible to record the video signal from a chosen camera in standard avi format.

USAGE

Choose the monitor using typing the corresponding monitor number on the numeric pad (1 for monitor number 1 and so on).

Choose the camera typing the corresponding camera number on the numeric pad. The following table summarizes all the existing cameras:

CHANNEL	CAMERA	DISPLAY NAME
1	EXP. CHAMBER 1	EXAFS CH1
2	EXP. CHAMBER 2	EXAFS CH1
3	ReflEXAFS 1	Irifl.
4	ReflEXAFS 2	Inor.
5	Free camera	FREE CAMERA
6	BEAM MONITOR 1	BM1
7	BEAM MONITOR 2	BM2
8	BEAM MONITOR 3	BM3
9	BEAM MONITOR 4	BM4
10	BEAM MONITOR 5	BM5
11	XRD 1	X-RAY DIFFR.
12	XRD 2	DIFFR. 1
13	XRD 3	DIFFR. 2
14	XRD 4	X-RAY EXAFS LIKE
15	XRD 5	EXAFS LIKE
16	XRD 6	EXAFS LIKE 2

OPERATION

FRONT END OPERATIONS

Control application: ID-APPLI
Location: RITAPCU1
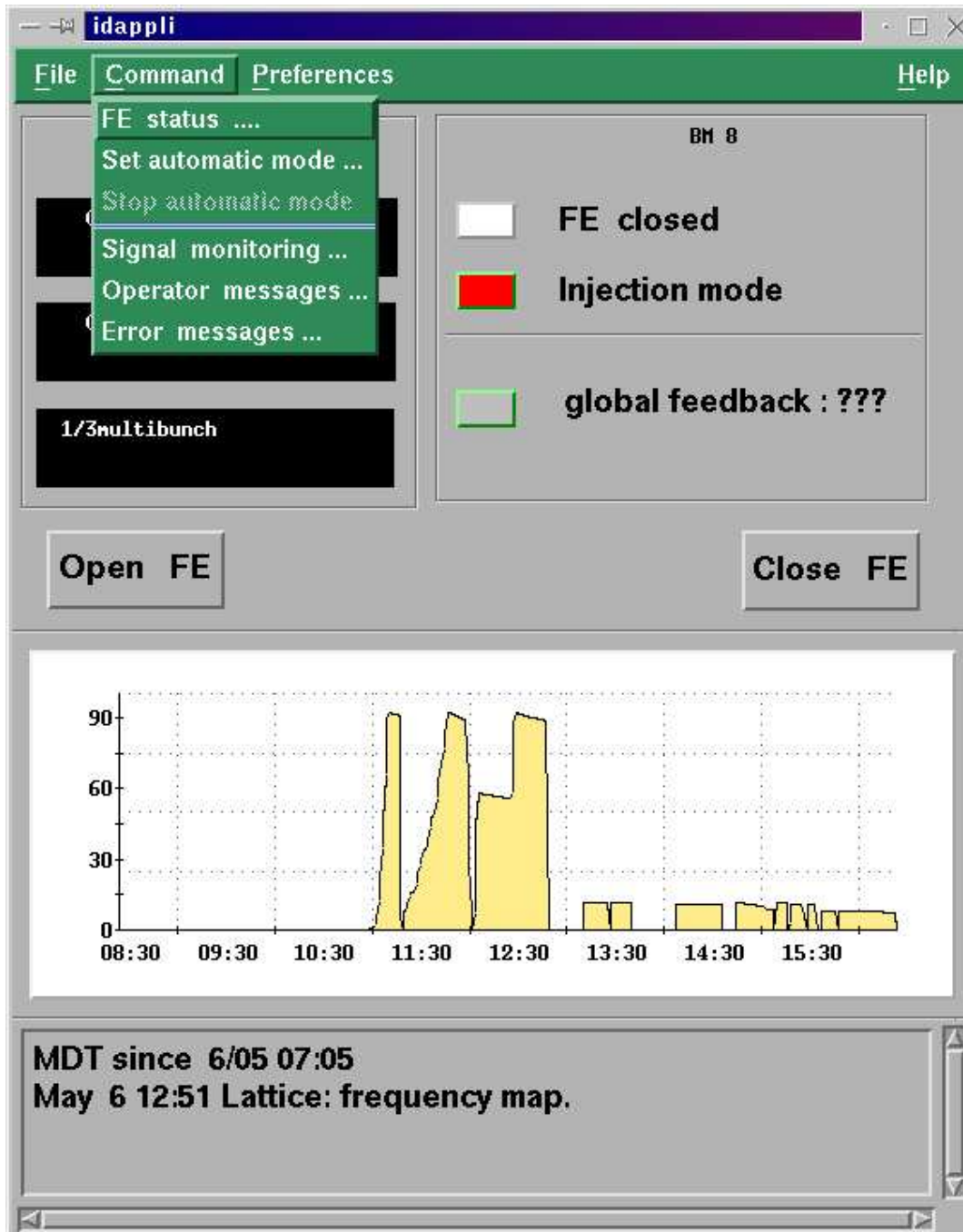
OPENING AND CLOSING THE FRONT END

The FRONT END (main beam shutter) is controlled from RITAPCU1. Before opening the FRONT END be sure that the optic hutch is correctly closed.

WARNING wait 30s before the searching procedure is completed before opening the FRONT END.
Before this delay of time the FRONT END won't open!

AUTOMATIC MODE

If something goes wrong with an injection or if the beam is lost, the FRONT END will automatically close. It will automatically reopen only if you have set AUTOMATIC MODE on id-appli.
To activate this mode, go on the settings menu and click on the SET AUTOMATIC MODE entry, then click on OK (sse figure).

WARNING AUTOMATIC MODE will last for 72 hours. After this delay of time the FRONT END will automatically close for security reasons.
Always remember to reset AUTOMATIC MODE within 72 hours from last activation.

TROUBLESHOOTING

If RITAPCU1 is off:

1) Switch it on and login using
   Username = opd08
   Password = tonic08

2) 'id-appli' and 'minicom' applications should start automatically when logged in. If not, double-click on the corresponding desktop

icons (FRONT END for 'id-appli' and SHUTTERS
for 'minicom').

3) Click on the 'OPEN' button on 'id-appli' to
open the FRONT END and on the 'CLOSE' button
to close it.

In case of failure of these operations call the
EXPERIMENTAL HALL OPERATOR (EHO) from the beamline
phone (2525).

SECONDARY SHUTTERS AND VACUUM VALVES OPERATIONS

Control application: MINICOM
Location: RITAPCU1
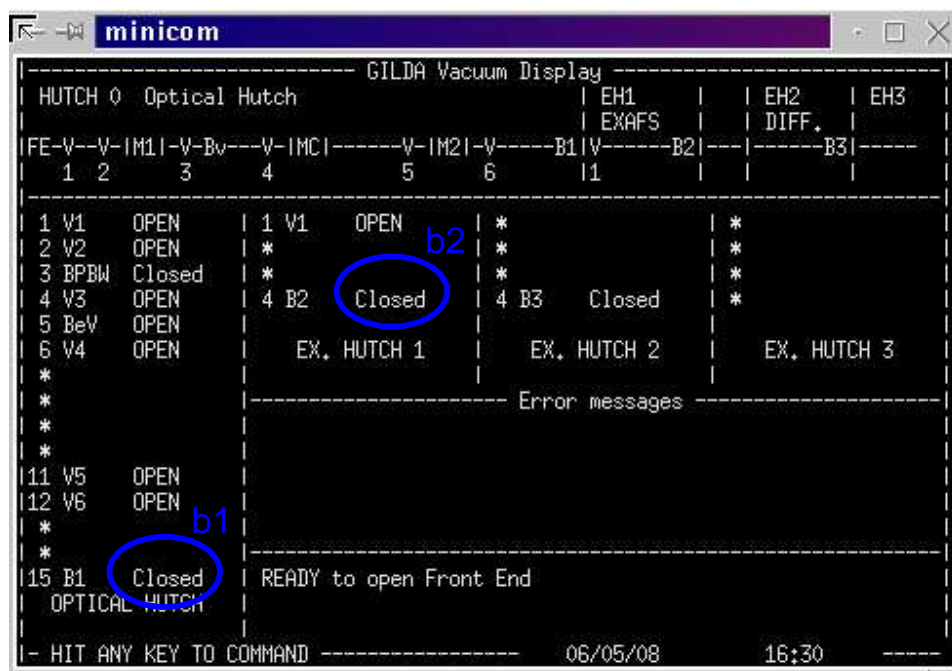
The 'minicom' application.

Secondary shutters and vacuum valves are controlled
from RITAPCU1.
An application ('minicom' figure above) shows the
current status the shutters and valves for each

hutch. An error area indicates possible failures. If you get a repeated error message call your Local Contact.

EXPERIMENTAL HUTCH SHUTTERS

Standard users operations

To enter the experimental hutch or to let the beam enter the hutch, you must close/open the corresponding hutch shutter ('B1' for XAS experimental hutch, 'B2' for XRD exp. hutch).
B1 is located between the optic hutch and the XAS hutch; B2 between the XAS and the XRD hutches.

To open/close the shutters, follow this procedure:

1) On RITAPCU1, focus on the shutters and vacuum application ('minicom', figure above) by clicking on its window.

2) Press the spacebar.

3) At the prompt 'Hutch number?' type 'b1' for XAS hutch shutter 'b2' for XRD hutch shutter.
The command inverts the former status of the device (opens if previously closed and vice-versa).

4) Check in the application window if the shutter status has changed (blue circles in the figure indicate the status of the XAS 'b1' and 'b2' shutters).

WARNING Once you start the hutch searching procedure you must wait 30s before you can open the shutter. During this period the open command is ignored.

Non-standard operations

VACUUM VALVES OPERATIONS

If there is a black-out or if the pressure of one of the vacuum sections rises above the set limit, one or more vacuum valves automatically close and an error message appears in the error area of the 'minicom' window (for example: 'vacuum error in test point #'). The following table reports the name and location of the existing vacuum valves:

VALVE NAME	HUTCH	HUTCH NUMBER
V1 *to* V6	OPTIC	1
V1	XAS	2

To open the valves manually follow this procedure:

1) On RITAPCU1, focus on 'minicom' clicking on its window.

2) Press any key to enter commands.

2) At the prompt 'Hutch number?' input the number of the hutch where the valve you want to open is referring to the above table and hit enter.

3) At the prompt 'Command?' type the valve name and hit enter. The command acts to invert the current status of the device (opens if previously closed and vice-versa).

EXAMPLE
Open valve number 3 (V3) in optic hutch (HUTCH 1):

    1) Press any key

2) Hutch number? type '1' and hit enter

3) Command?   type 'v3' and hit enter

4) Check if the status of the corresponding valve has changed in the 'minicom' window.

TROUBLESHOOTING

If RITAPCU1 is off follow the procedure reported in the TROUBLESHOOTING section in previous paragraph.

If you messed-up with typing commands press letter 'q' on the keyboard to reset the command prompt.

To reset the application display press any key, type the letter 'r' hit enter and wait.

SECONDARY SLITS (EXAFS SLITS) OPERATIONS

Control application EXAFS_TN
Location RITAPCU2 (Remote location RITAPCLINUX)
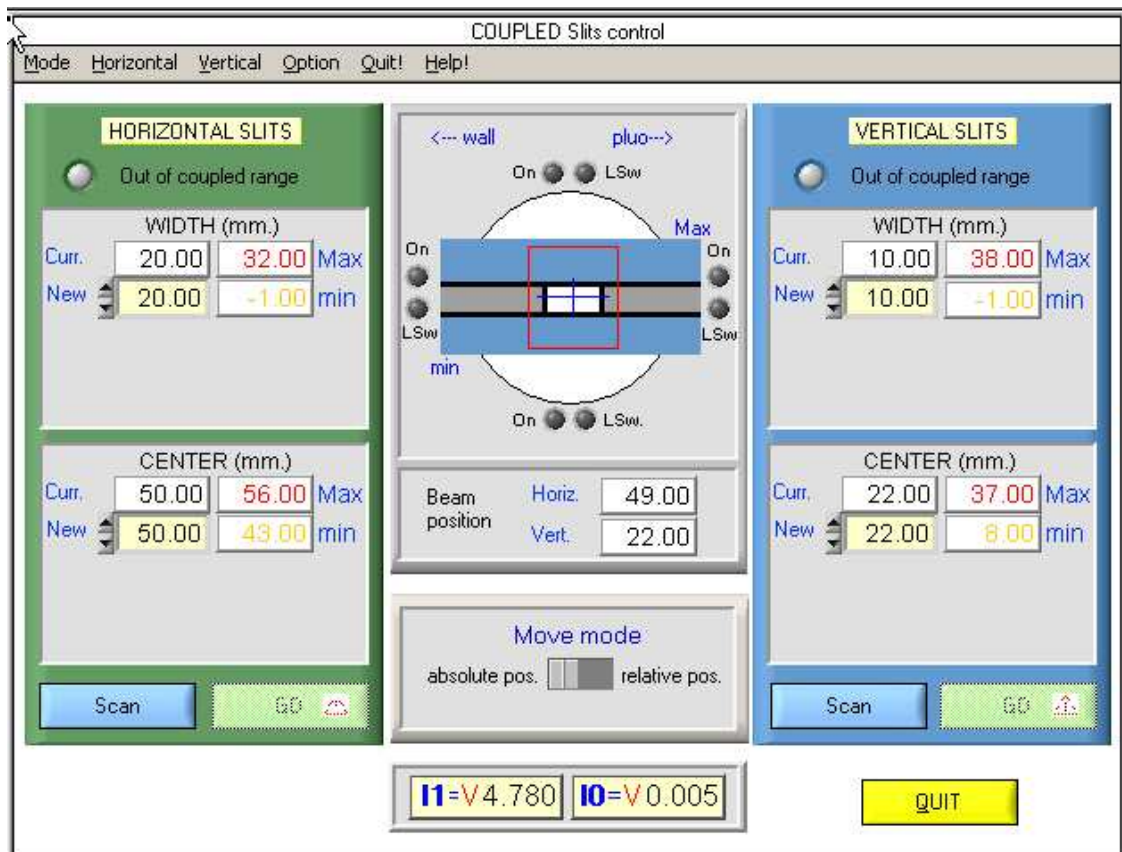
Secondary slits (EXAFS slits) are located before the first ionization chamber (I0) and can be adjusted using the EXAFS_TN application running on RITAPCU2 inside the XAS HUTCH but also accessible from the acquisition PC in the control room via a VNC connection (redirection of the RITAPCU2's display). If this connection is closed refer to the GENERAL TROUBLESHOOTING section at the end of this manual.

There are a couple of vertical and a couple of horizontal slits. Depending on your requirements

you can: change the width of the horizontal and vertical slits, act on a single slit or scan the center of the horizontal or vertical slits.

CHANGING WIDTH AND/OR CENTER OF THE EXAFS SLITS

To access the slits commands click on the SLITS button of the EXAFS_TN application.
To change the width or the center of the vertical or horizontal slits type the new value in the 'new' field and click the GO button.

SCANS
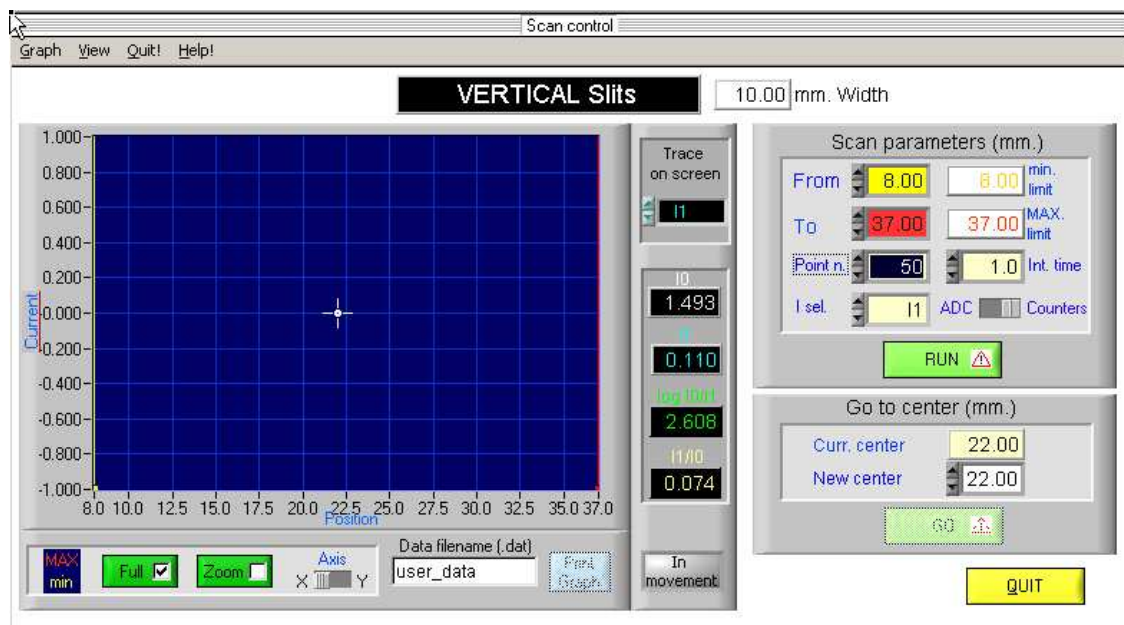
If you need to scan the center of the vertical or horizontal slits:

  1) Click on the SCAN button and insert the scan limits.

  2) Choose the number of points in order to have a suitable spacing between points (typically 0.1 mm, i.e. 10 points/mm).

3) Set the integration time (usually 0.1s is enough for a good statistics and a fast scan).

WARNING EXAFS_TN is not a Labview application and thus it does not need to be quit; nevertheless you must quit all Labview applications BEFORE performing a scan.

Scans are stored on RITAPCU2 and can be accessed trough a network folder (EXAFS_TN_SCANS) on the desktop of the acquisition PC.

IONIZATION CHAMBERS

There are two couples of ionization chambers in the XAS experimental hutch. The first are located before EXAFS CHAMBER 1 (I0) and after EXAFS CHAMBER 2 (I1) and are used for XAS experiments.
The other couple is used for gracing incident experiments (ReflEXAFS) and are located before (Inor.) and after (Irifl.) the REFLEXAFS CHAMBER.

WARNING

The voltage of an ionization chamber can be read from the red displays at the top of the electronics crate located at the right of the acquisition PC in the control room (see control cabin section).

CHANGING IONIZATION CHAMBERS PRESSURES AND GAS TYPE

Control application EXAFS_TN
Location RITAPCU2 (Remote location RITAPCLINUX)

It is possible to remotely change the gas pressure
and type in the I0 and I1 ionizations chambers from
the control room.
To change the pressure and/or gas type:
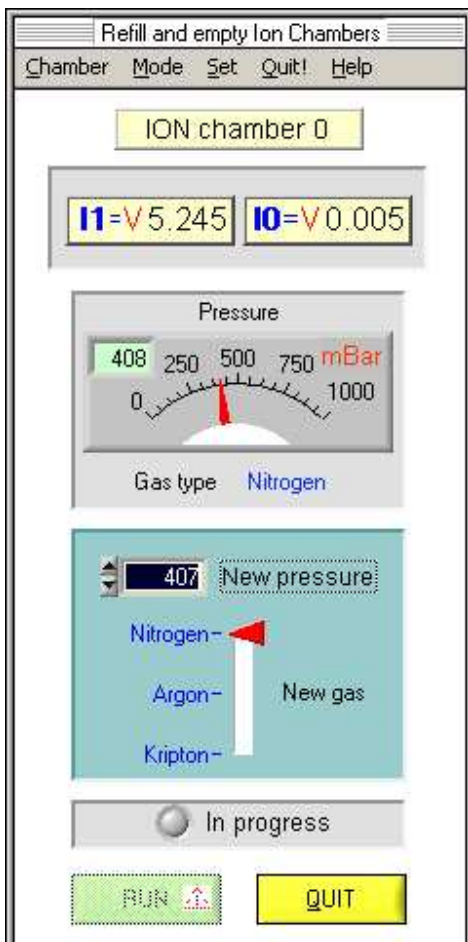
1) Click on the I0 or I1
   buttons in EXAFS_TN

2) Type the desired
   pressure and gas type

3) Hit the RUN button

To calculate the absorption and the photon flux you
can use the applications described in the SOME
USEFUL APPLICATIONS section or refer to the
following table.

NOTE: REFLEXAFS ionization chambers (Inor. and Irifl) have to be operated manually inside the XAS experimental hutch.

GAS PRESSURES

ION. CHAMBER	I_0	I_1
ABSORPTION	**10%**	**80%**

Energy KeV	Pressure mbar	Pressure mbar
4	0.10 N_2	0.50 N_2
5	0.20 N_2	1.00 N_2
6	0.30 N_2	0.10 Ar
7	0.50 N_2	0.10 Ar
8	1.00 N_2	0.20 Ar
9	1.00 N_2	0.30 Ar
10	0.05 Ar	0.40 Ar
11	0.10 Ar	0.50 Ar
12	0.10 Ar	0.60 Ar
13	0.15 Ar	0.70 Ar
14	0.20 Ar	1.00 Ar
15	0.20 Ar	0.10 Kr
16	0.25 Ar	0.10 Kr
17	0.30 Ar	0.15 Kr
18	0.35 Ar	0.15 Kr
19	0.40 Ar	0.20 Kr
20	0.50 Ar	0.20 Kr
21	0.60 Ar	0.20 Kr
22	0.65 Ar	0.25 Kr
23	0.75 Ar	0.30 Kr
24	0.85 Ar	0.30 Kr
25	1.00 Ar	0.35 Kr
26	0.10 Kr	0.40 Kr
27	0.10 Kr	0.45 Kr
28	0.10 Kr	0.50 Kr
29	0.10 Kr	0.55 Kr
30	0.10 Kr	0.60 Kr
31	0.10 Kr	0.65 Kr
32	0.10 Kr	0.70 Kr
33	0.15 Kr	0.80 Kr

CHANGING THE AMPLIFICATION

Control application: KEYTHLEY
Location: RITAPCU2 (Remote location RITAPCLINUX)
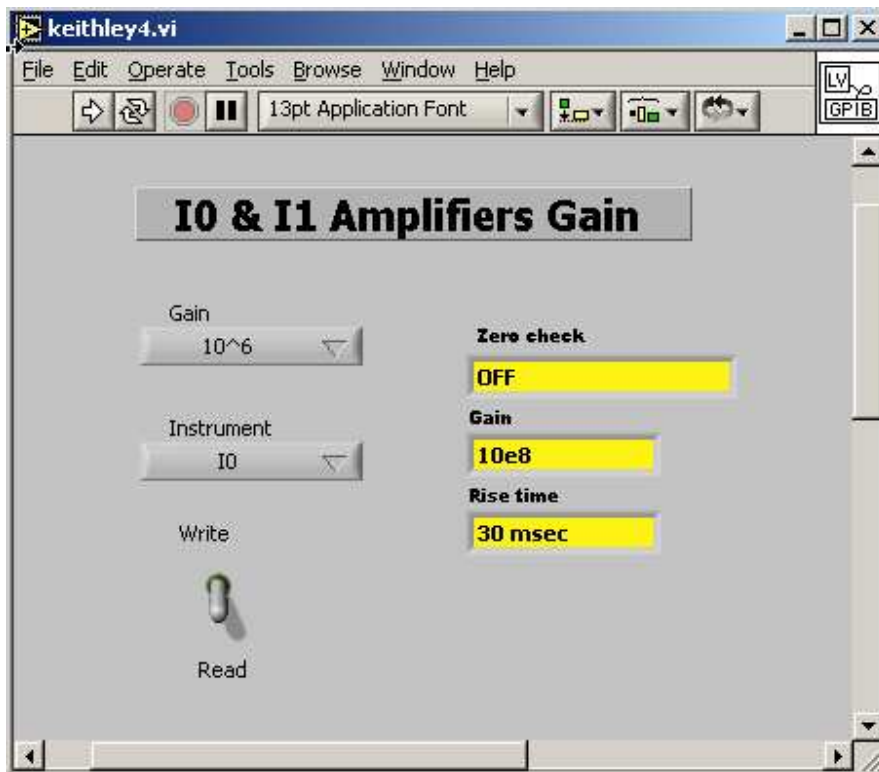
It is possible to remotely change or read the amplification of the I0 and I1 ionizations chambers from the control room.
To change/read the amplification, focus on the KEITHLEY Labview application on RITAPCU2 (or from the acquisition PC in the control room trough VNC connection).
BEFORE activating the application by clicking on the white arrow:

  1) Select write/read mode

  2) Select the ionization chamber (I0 or I1)

3) Select an amplification in the range 10^6-10^{11}

WARNING
You must set the amplification factor in order to keep the ionization chambers read-out voltage in the range **0.5 – 8.0 Volts.**
This is the linearity range for the VFCs (Voltage to Frequency Converters).
Outside this range the level of the noise will dramatically increase.

MOUNTING/CHANGING THE SAMPLES

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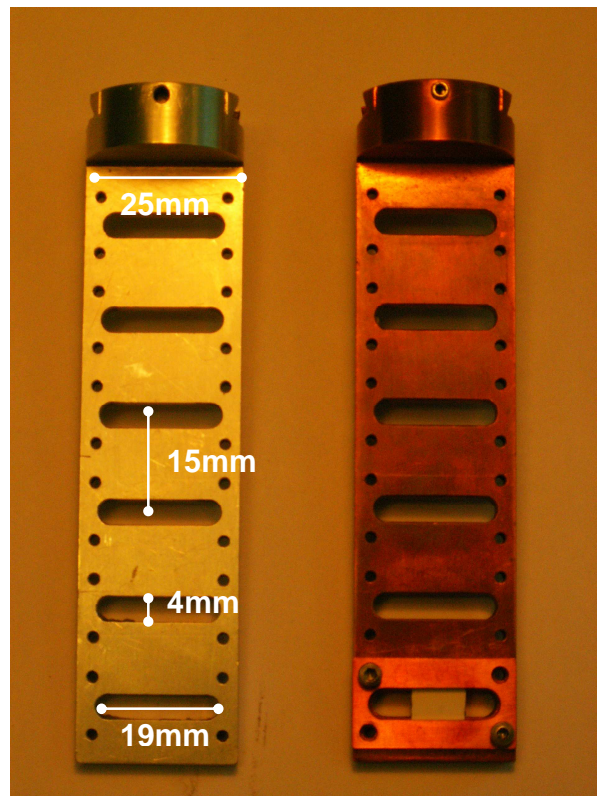
Sample holders

The sample holder can host up to 6 samples which size not exceeding 15 x 20 mm. 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.

VACUUM OPERATIONS

Control application: EXAFS_TN
Location: RITAPCU2 (Remote location RITAPCLINUX)

Once you have mounted the sampled on the sample holder in EXAFS CHAMBER 1 or 2, close the chamber and start the vacuum pumps. This can be done from the EXAFS_TN application. Follow this procedure:
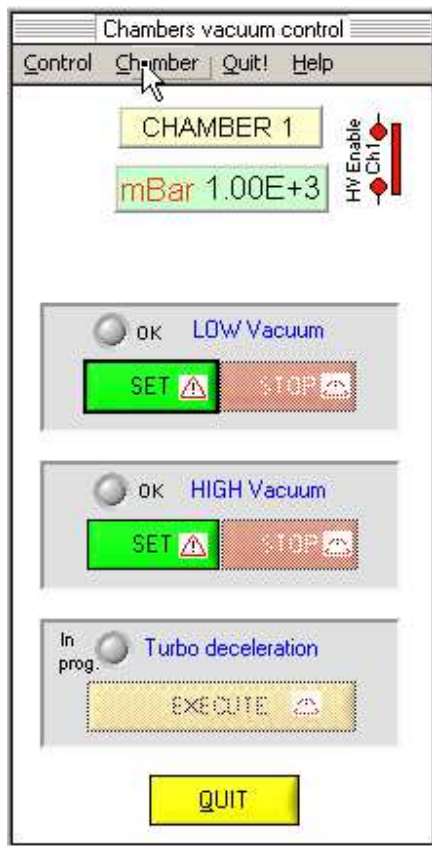
1) Close the exp. chamber porthole

2) **Do not forget to close the exp. chamber valve**

3) On the EXAFS_TN main window select 'vacuum' under the corresponding chamber menu and click on the CHAMBER 1 (or 2) button. The vacuum dialog window will pop up (figure).

4) Start the low vacuum by clicking the SET button under the 'low vacuum' section. If you require high vacuum (for example) for low temperatures)

start the turbo molecular pump by clicking the SET button under the 'high vacuum' section.

Stopping the pump and breaking the vacuum

1) To stop the low vacuum in exp. chamber 1 or 2 access the vacuum dialog window trough the EXAFS_TN application and click the STOP button.

2) If you want to stop the high vacuum (turbo molecular pump) click STOP under the high vacuum section and the TURBO DECELERATION button when enabled. A green indicator will light on showing that the deceleration is in progress. When it turns off, deceleration is completed.

3) Open the valve to break the vacuum. When the chamber is at atmospheric pressure after you can open the chamber's porthole.

4) Always remember to close back the exp. chamber valve!

WARNING

Once you have opened the exp. chamber 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.**

THE He/N CRYOSTAT

Control application: OB cryostat
Location: RITAPCU2 (Remote location RITAPCLINUX)
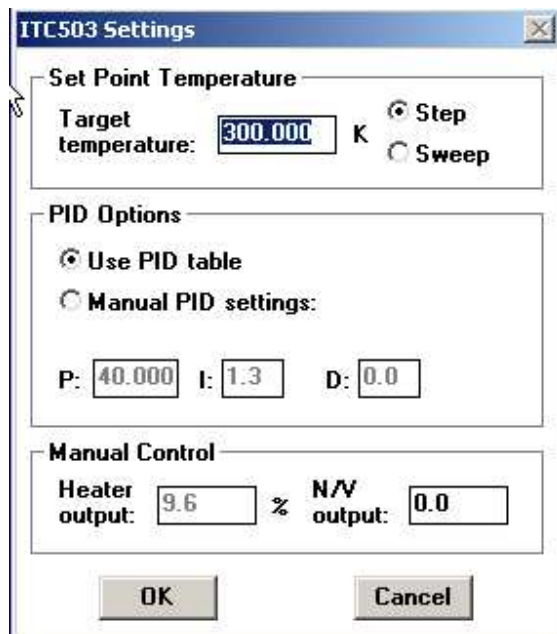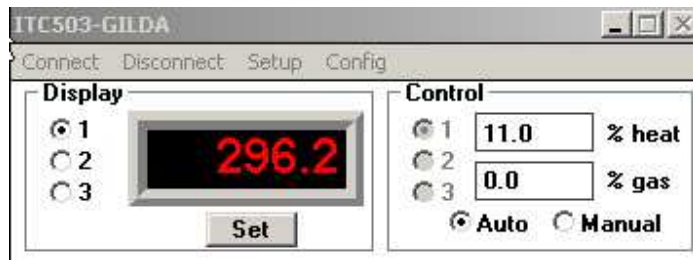
TEMPERATURE RANGE

REFRIGERATING LIQUID	MIN.(K)	MAX.(K)
Nitrogen	80	420
Helium	10	420

Cooling down the samples

1. Close EXAFS CHAMBER 1 and start the high vacuum following the procedure reported in the VACUUM OPERATIONS paragraph.

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

3. Once the turbo pump has reached his normal speed (green indicator on EXAFS_TN) start the He/N pumping and set the pumping level to 100% until the temperature reaches the set point.

4. In order to optimize the nitrogen consumption you can reduce the pumping level to down to a value where the heater power is not less than 10%. Typical working pumping level values are 70-100% for nitrogen, 30-40% for helium.

The He/N2 cryostat pump regulator.

NOTE

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%. At the same time, it should be the lower possible in order to reduce helium/nitrogen consumption. If the temperature rises above the set point increase the pumping level of a 5%-10%. If this does not solve the problem inform your local contact.

USEFUL INFORMATION

Duration of the helium/nitrogen reservoir

The helium reservoir should 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.
The liquid nitrogen dewar can last for a couple of days. Nevertheless, it is recommended that you refill it every day.

Cooling down time

Cooling down the sample takes about 45mn working with liquid nitrogen and about 15mn working with liquid helium. During the first 10mn the temperature won't lower because the refrigerating liquid is cooling down the cryostat sink.

Actual sample's temperature

Remember that, at 20K, the bottom of the sample holder is about 10 degrees warmer than the top. This shift decreases increasing the temperature. You can refer to the technical notes on the GILDA

WIKI page to have a complete characterization of the sample holder as a function of the temperature. If you need to monitor the temperature with higher accuracy (about 1K), you can ask your local contact to mount a temperature sensor close to the sample.

Heating up the samples

1. Stop the He/N pumping

2. Set the temperature to 300K on the cryostat application.

3. After the temperature reached the set point, wait at least 15mn that the sample holder warms up to room temperature.

4. Break the vacuum following the procedure reported in the VACUUM OPERATION paragraph.

WARNING Do not open the experimental chamber at low temperatures!

CHANGING THE ENERGY AND GETTING THE BEAM ON THE SAMPLES

Control application: SETENERGY
Location: EXAFS CONTROL PANEL or EXAFS folder on RITAPCLINUX
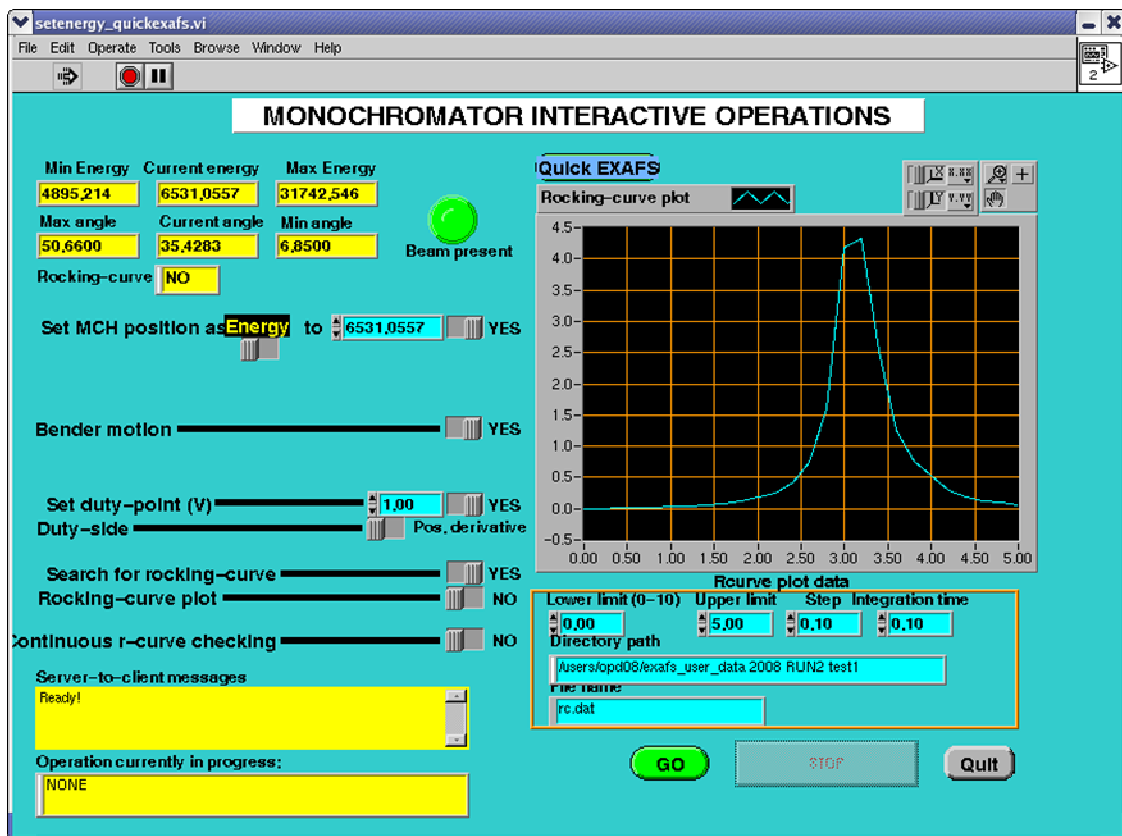
With this application you can supply a beam at the desired energy and intensity on the samples. It is also possible to plot the Rocking curve in order to know the maximum value of the beam intensity.

Procedure to set energy at a desired intensity:

With a curved crystal (focused beam)

1) Check the state of the following Boolean variables (and change if different):

Variable   State

Monochromator motion   Yes
Bender motion   Yes
Set duty point   Yes
Search for rocking-curve   Yes
Rocking curve plot   No
Continuous r-curve check   No

2) Input the desired energy in the 'energy' field. The default value is the current energy.

3) Input the desired output intensity in the 'Duty Point' field. Refer to the Duty Point section below to choose a suitable duty point value.

4) Input the rocking curve side (positive or negative derivative). See next section to choose the right value.

5) Push 'Go' button. Normally a green spot on the oscilloscope should start to 'float', its y voltage value corresponding to the duty-point value.
This means that the beam has been found and the desired duty-point is set. In case of fail the program will automatically look for the beam in a limited angle range. This procedure will last for about 30s.
Do not push the stop button while the application is looking for the beam. In case of a further fail

consult the TROUBLESHOOTING section in this paragraph or call the Local Contact.
<u>With a flat crystal (unfocused beam)</u>

Follow the procedure reported above for a curved crystal but check the state of the following Boolean variables (and change if different):

Variable	State
Bender motion	**No**
Set duty point	Yes
Search for rocking-curve	Yes
Rocking curve plot	No
Continuous r-curve check	No

Rocking curve and duty point

The Rocking curve is the reflectivity curve of the monochromator crystals as a function of the angle of the first crystal relative to the incident beam. When the crystals are parallel there is maximum reflected intensity (maximum of the Rocking curve).
The duty point is a chosen point on the Rocking curve and it represents the intensity of the beam.
You may usually need the maximum achievable intensity but, if you are acquiring low diluted samples with the germanium detector, you may also need to lower the beam intensity.
Working far from the maximum of the Rocking curve helps to avoid the contribution from higher harmonics.
The value of the duty must not exceed 80% of the maximum value of the Rocking curve.
Perform a plot of the Rocking curve in order to know its maximum value.

Rocking curve plot

To obtain a plot of the Rocking curve first be sure
that the beam is in the angular range reachable by
the application.
Set the monochromator energy with a duty-point
value of 0.1V.
If a green spot starts to float on the oscilloscope
then you can continue with the following procedure.

With a curved crystal

1) Check the state of the following boolean
variables (and change if different)

Variable   State

Monochromator motion   Yes
Bender motion   Yes
Set duty point   No
Search for rocking-curve   No
Rocking curve plot   Yes
Continuous r-curve check   No

2) If you want to save the plot of the rocking
curve input also a suitable file name.

3) Click 'Go' button. The plot will appear in the
window. For an EXAFS scan typical duty-point values
are 70-80% of the maximum of the curve.

With a flat crystal

Check the following Boolean variables (and change
if different) and follow the same procedure for a
curved crystal:

```
Variable                  State

Monochromator motion      Yes
Bender motion             No
Set duty point            No
Search for rocking-curve  No
Rocking curve plot        Yes
Continuous r-curve check  No
```

WARNING

1) Do not use the STOP button while the monochromator is moving to lower energies.
Doing it may cause to loose the energy calibration!

2) The value of the duty point must not exceed 70% - 80% of the maximum value of the Rocking curve. Exceeding this value the feedback system won't be able to keep the monochromator crystals parallel. This will result in zero beam intensity.

3) The QUICKEXAFS button is not intended for standard user operations. Do not use it!

TROUBLESHOOTING

Problem	Possible reason
'Setenergy' cannot find the beam	The ESRF current is zero.
	The front end is closed.
	The B1 shutter is closed.
	There is no gas in the ion chambers.
	The I0 amplification is wrong.

	The 'duty-point' is higher than 70%-80% of the rocking-curve maximum.
	The EXAFS slits are not correctly positioned.

Aligning the samples

Standard alignment

Control application: Sample_holder
Location: EXAFS CONTROL PANEL or EXAFS folder on RITAPCLINUX
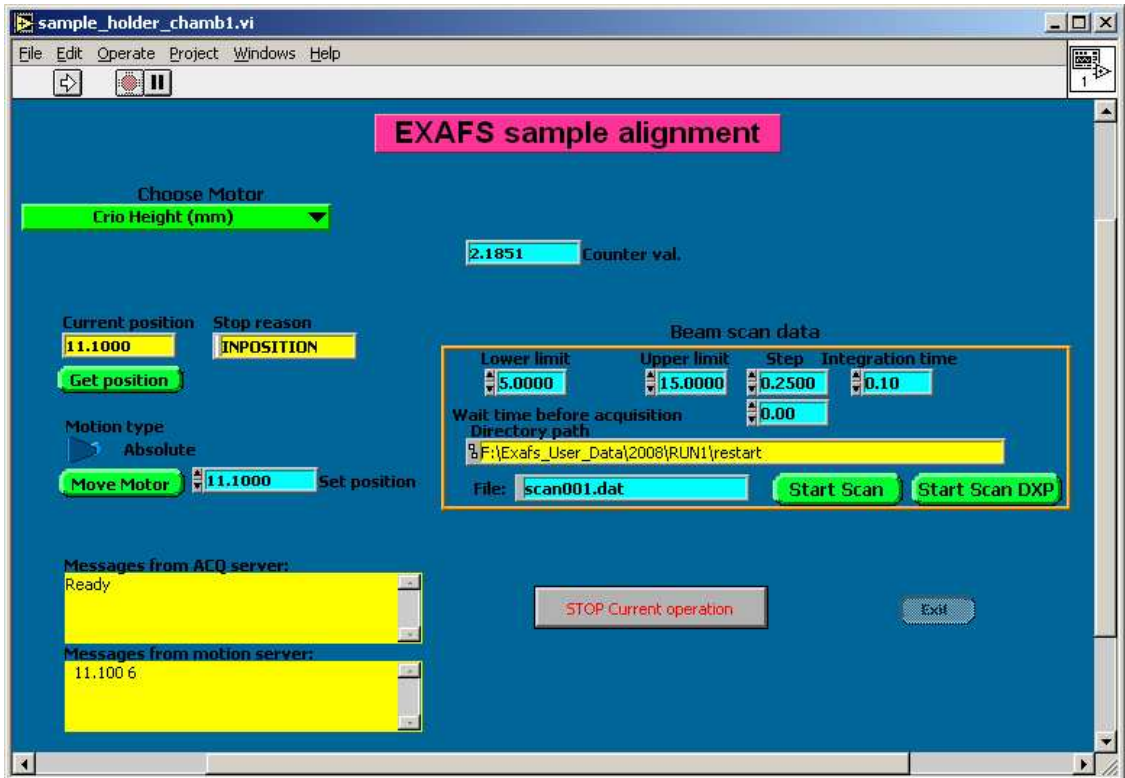
With this application you can:

1. Perform vertical translation of the sample holder and record scans of the transmitted or fluorescence yields.

2. Perform horizontal translations of the exp. chamber and record scans of the transmitted or fluorescence yields.

3. Rotate the sample holder and record scans of the transmitted or fluorescence yields.

4. Move the 13-element detector closer of farter the samples.

The following table reports the translations range for each motor (minimum motor step 0.006mm):

Traslation	MIN (mm)	MAX (mm)
Vertical	0 up	120 down
Horizontal	0 left*	42 right*
Rotation	-120° **	110° **
Multidetector	0 out	190 in

* Facing the incoming X-ray beam
** Positive values orient samples toward detector

USAGE

GET POSITION

1) Choose the translator you want to check the position under the 'Choose motor' menu.

2) Click on the GET POSITION button to retrieve the selected translator position.

MOVE

1) Choose the translator you want to move in the 'Choose movement' menu.

2) Click the GET POSITION field to know the current position.

3) Remember to check to motion type mode (ABSOLUTE/RELATIVE) indicator BEFORE starting the motion.

4) Type the new position in the 'Set position' field (cyan area).

5) Click 'Move Motor'. You can stop the motion at any time using the STOP button.

6) At the end of the operation the new position is loaded in the 'Current Position' field.

SCAN

To align your samples on the beam, you can perform a vertical and/or horizontal scan of the transmitted or fluorescence intensity. The procedure is the following:

1. 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 (refer to previous paragraph). If you have zero intensity, search the beam using Set_Energy application as explained in a previous section.

2. Choose the translator you want to scan in the 'Choose motor' menu.

3. In the SCAN area insert the scan. A spacing of 0.25 mm is typical value (0.006 mm is the minimum accepted value). The typical integration time for fast scans is 0.1s.

4. Change the default file name if you wish to keep the scan file.

5. Push the START SCAN button to perform a scan of the transmitted beam or the START FLUO button for fluorescence yield scan (fluorescence scan is performed acquiring the central detector channel (CH7) only).
In the fluorescent scan case be sure that cursors have been correctly set in the STANDALONE application and that this application is quitted (refer to the 13-ELEMENTS GERMANIUM DETECTOR section for details).

WARNING

Sample_holder application only acts on exp. chamber 1.
Exp. Chamber 2 is used for non standard setups and can be controlled by the EXAFS_TN application.

GIXAFS alignment

Control application: GIXAFS ALIGN
Location: EXAFS CONTROL PANEL or EXAFS folder on RITAPCLINUX
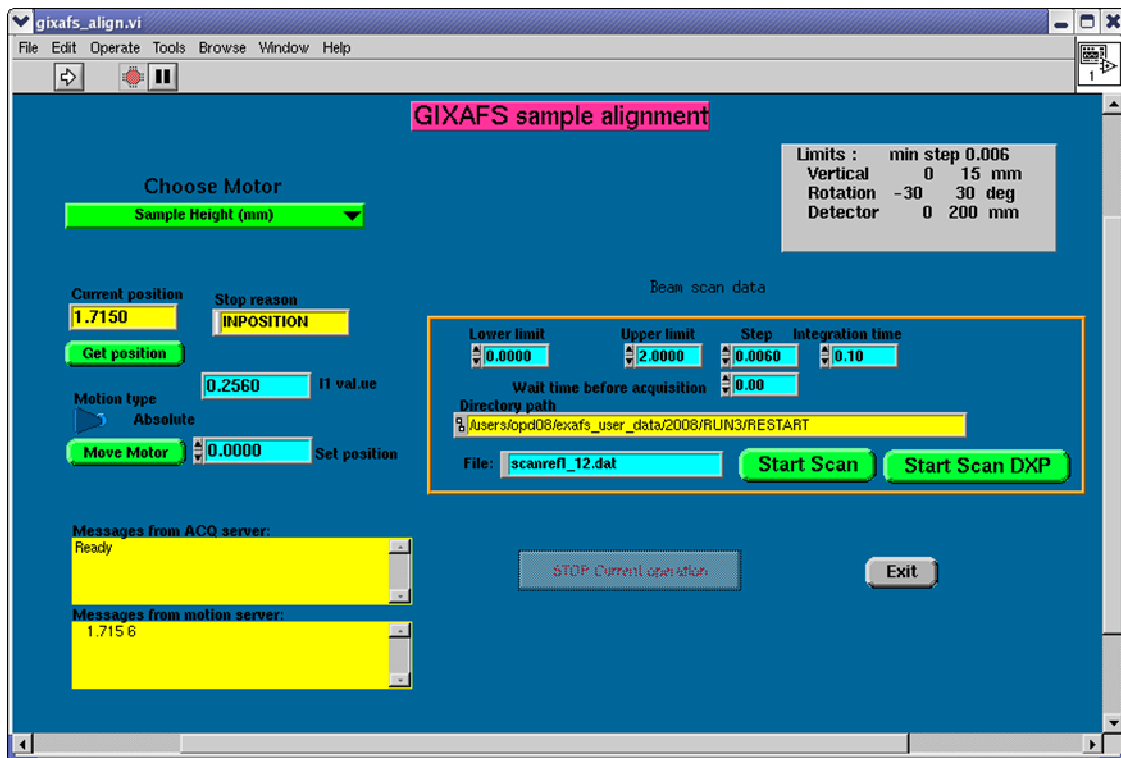
On GILDA it is possible to perform grazing incidence absorption experiments in the first EXAFS chamber.
The procedure for aligning the sample is similar to the alignment for ReflEXAFS experiment:

1) Define the height at which the sample cuts exactly half of the beam (z0). Move the height of the sample holder in order to cut half of the transmitted beam.

2) Scan the height (fig. 1a) of the sample in a range of about 1 mm across z0 (scan parameters:

steps 0.2, integration time 0.5s) and perform a derivative (fig. 1b) of the recorded spectra to obtain the exact position of the flex which corresponds to z0.

Fig. 1a

Fig. 1b
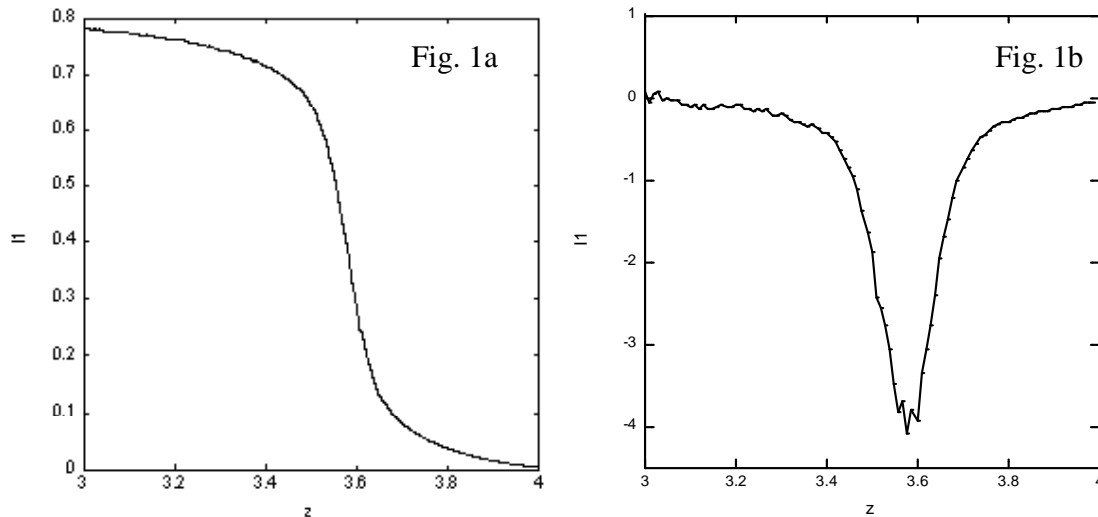
3) Define the angular position at which the sample surface is parallel to the beam ($\theta 0$). To individuate $\theta 0$ move the angular position to positive values until the transmitted beam goes to zero and do the same for negative values. $\theta 0$ is roughly the average between those to values.

4) Scan the angular position in a range of about one degree around $\theta 0$ (scan parameters: steps 0.2 degrees, integration time 0.5s). You should obtain a curve similar to that reported in figure 2. The peak of the spectra corresponds to $\theta 0$.

Fig. 2
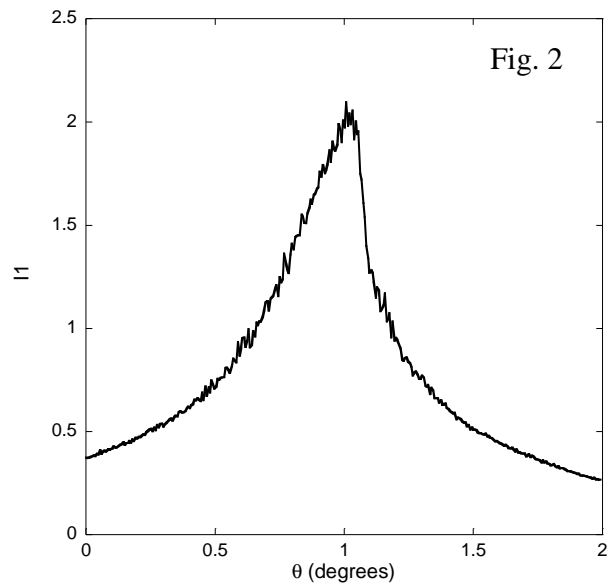
5) Repeat points 2 and 3 to refine the values of θ0 and z0.

6) To define the experimental working angle, add the obtained value for θ0 to your theoretical working angle.

13-ELEMENTS GERMANIUM DETECTOR

Control application: Standalone
Location: EXAFS folder on RITAPCLINUX
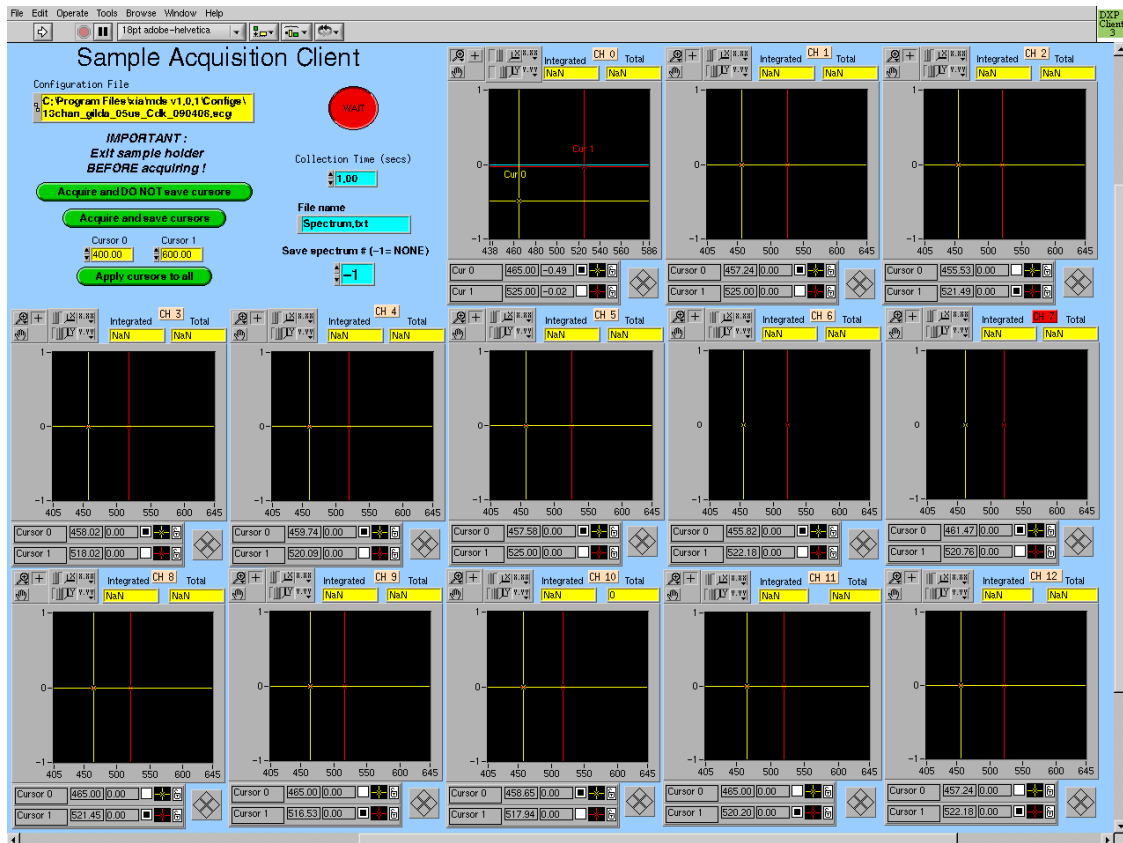
The 13-elements high purity Germanium detector is used for the collection of fluorescence spectra from diluted samples. The calibration of the detector will be done by your local contact.

Use the STANDALONE Labview application to choose the fluorescence line of interest.
STANDALONE shows windows for all the 13 channels of the Germanium detector. On the abscissa you have channels which are linked to the energy (ask your local contact for details about the energy calibration) and on the ordinate you have the counts divided by the integration time.

The procedure for setting the cursors (i.e. the integration limits) is the following:

1. Align the sample on the beam and rotate the sample holder toward the detector. The angular position of the sample holder is usually 45 degrees for standard acquisitions but it can be any value in the range 0 – 90 degrees.

2. Quit the SAMPLE_HOLDER application

3. Check that the beam intensity is not zero by looking at the I0 value on the red indicators or at the value of the point floating on the oscilloscope. In the case of zero beam intensity search the beam using SETENERGY application as explained in a previous section.

4. Launch the STANDALONE application by clicking on the white arrow on the top left of the window (the STANDALONE window should always remain open. Do not close it. If you cannot find it, open the EXAFS folder and double-click on the STANDALONE icon).

5. When prompted, choose the detector configuration file. Check the name of the configuration file and press click the CONTINUE button (if you are not sure about the name of the configuration file ask your local contact for details or read the TROUBLESHOOTING section in this paragraph).

6. Wait that the red 'WAIT' indicator turns to green ('QUIT').

7. CLick the 'ACQUIRE AND DO NOT SAVE CURSORS' button to acquire fluorescence spectrum of the sample.

8. Once located the K-alpha fluorescence line of the element of interest place the CURSORS sidewise the fluorescence line remembering that to **set Cursor 0 at lower channels than cursor 1**.
Cursors should be placed in zones of zero derivative to minimize the effects of slow drifts of the pulse analyzer.
The pulses between the cursors are integrated and displayed in the **'integrated**' field of each element window.
The '**total**' field shows the total count rate. Remember that **the total counts per second must not exceed 80.000; outside this range, the detector has a non-linear response!**
Repeat the CURSOR positioning for all the 13 channels.

9. Click the 'ACQUIRE AND SAVE CURSORS' button to store the final cursors settings and.

STORING THE TOTAL FLUORESCENCE LINES SPECTRA IN A FILE

1. Choose the file name in the 'file name' field.

2. Choose the detector element to record in the 'Save spectrum' field (type the number of the corresponding channel, the default value '-1' disables the recording of the fluorescence line spectra).

3. Click on the 'ACQUIRE AND DO NOT SAVE CURSORS' button.

WARNINGS:

1) Remember to check the liquid nitrogen level in the detector's dewar at least once per day.
A low liquid nitrogen level may result in detector shut down and more than 12 hours are necessary to recover. It is also likely the detector gets damaged!
The level indicator is located close to EXP. CHAMBER 1 (figure). If the green led is on the liquid nitrogen level its ok.
If the orange led (indicating that liquid nitrogen transfer is in progress) remains on for more than 30mn call your local contact.
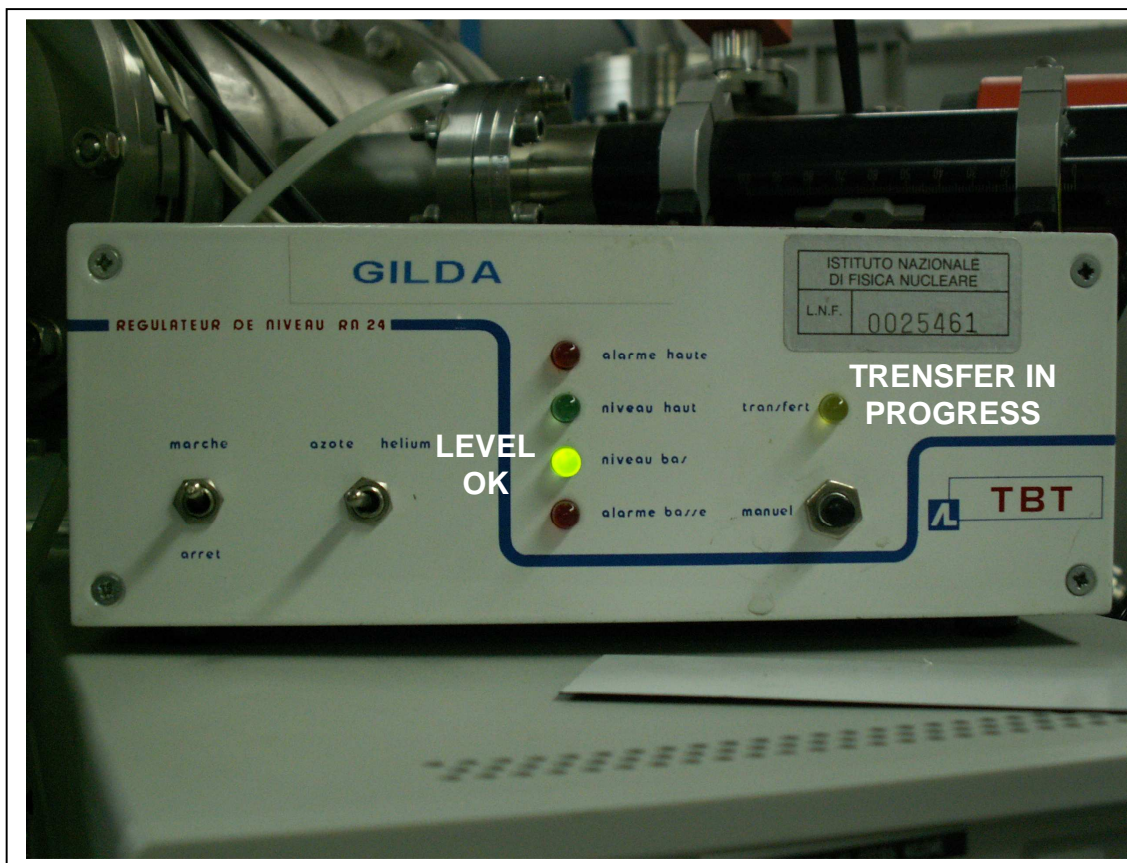
Liquid nitrogen level indicator

2) Do not keep the detector at count rates higher than 80.000 cps for long time!
During acquisitions, you must keep the total count rate below 80.000 cps. If you exceed this value try moving the detector farter (using SAMPLE_HOLDER) or reducing the duty point.

3) Remember to QUIT Sample_holder application before acquiring a fluorescence spectrum.

TROUBLESHOOTING

Problem	Solution
When pressing the ACQUIRE button I have zero or very low counts for all the channels	Quit Sample_holder Check that the beam is present Check that the sample is correctly aligned Check that you are using the right configuration file If you have checked all the above points and the detector is still not working: 1. Quit 'standalone' application 2. Close the Xserver window on RITAPC1 3. Reopen the Xserver window and run 'standalone' again
I have zero integrated counts	Check that cursor 0 is at lower channels than

	cursor 1
The path and name for the configuration file is missing	Reinsert the right path and name (ask your local contact)
Fluorescence peaks appear shifted respect to the cursors you have set	Check that the total count rate is less than 80.000 cps for each channel
I cannot find the fluorescence line of the absorber element	You are acquiring at an energy below the absorption edge energy The concentration of the absorber is too low compared with other excited elements in your sample. Try to put filters to reduce the fluorescence of the other elements

ACQUIRING A XAS SPECTRA

Application: EXAFS_SCAN_SETUP
Location: EXAFS CONTROL PANEL or EXAFS folder on RITAPCLINUX
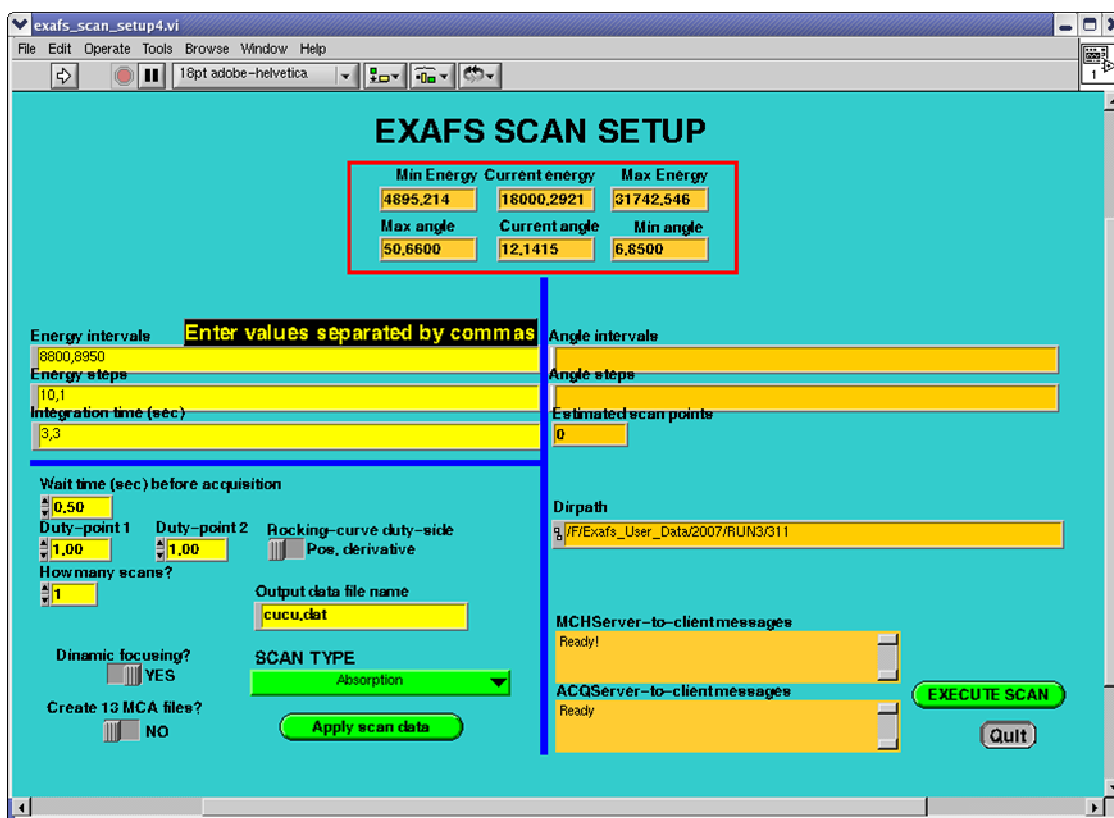
EXAFS_SCAN_SETUP permits to set the scan parameters for the acquisition of a XAS spectrum in different acquisition modes: transmission, fluorescence, multipurpose...).

USAGE:

1) Launch the application from the 'EXAFS CONTROL PANEL' on the desktop. If you are not using the 'EXAFS CONTROL PANEL' load the application from the 'EXAFS' folder on the desktop and click the white arrow at the top left side of the Labwiew window.

2) **Energy intervals**
Enter the energy intervals values of the spectrum.
Values must be separated by commas. You should
choose energy intervals in order to approximate the
energy to wave vector conversion curve reported in
the figure below with straight lines.
The last value corresponds to the extension of the
spectra. To convert the energy from eV to Å^{-1} (i.e.
energy to wave vector) use the following formula:

$$K_{max}(\text{Å}^{-1}) = \tfrac{1}{2}\ \text{sqrt}(E0-E_{max})$$

Energy to wave vector conversion

k (Å^{-1})	E (eV)
4	60
8	250
12	550
14	750
16	980
18	1250
20	1520
22	1850
24	2200
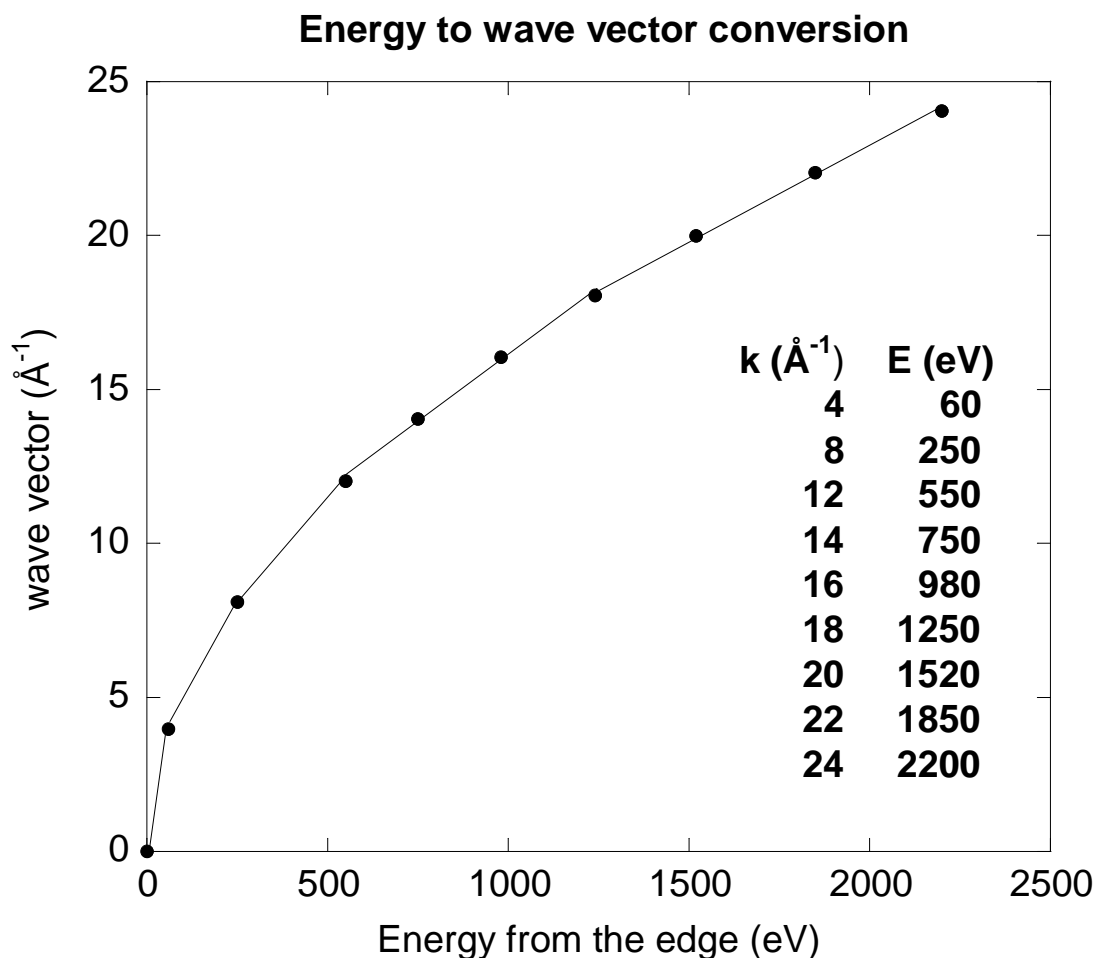
4) **Energy steps**
Enter the energy steps widths for each energy interval. Usually you may want to increase the energy resolution in the edge region. Minimum resolution values vary with energy and monochromator crystals.
Ask your local contact for the minimum energy resolution you can enter.
If you don't have this information check that the angular step for each interval is greater than 0.006 degrees.
You can find information on the angular intervals and steps in the right part of the application window.

5) **Integration times**
Enter the integration times for each energy interval. In transmission geometry an integration time of 3s is a reasonable value.
If you acquire in fluorescence mode you can calculate the integration time keeping in mind that the noise follows the Poisson law:

NOISE = 1/sqrt(cps*channels*integration time)

Where 'cps' is the number of counts per second and 'channels' are the detector elements.
Check the average counts per channel with the STANDALONE application.
For a good quality fluorescence spectrum the noise level should be less than equal to 0.001. If you cannot reach a good signal to noise level within 3 hours long spectra think about acquiring a reasonable number of repeated spectra instead of acquiring longer spectra. This way you can average several spectra enhancing the statistic. Further, if something goes wrong during the acquisition, you will lose less time by repeating a short spectrum than a long one.

If you want to estimate the time length of a spectrum, remember to take in to account 5 extra seconds for each energy point.

6) **Check that the number of integration time and step values corresponds to the number of energy intervals.**

7) **Wait time**
If different set to 1s.

8) **Duty-point**
Enter the initial and final duty-point. You should usually set identical initial and final values.
Different values can be used to maximize the photon flux when the beam decay time is shorter (16 or single bunch modes).

9) **Rocking curve side**
Choose the working Rocking curve side.
Usually set to **'Pos. derivative'**.

10) **Dynamical focusing**
Set to '**Yes**' if you are working with a curved crystal, '**No**' if you are working with a flat crystal. In case of doubt ask your local contact.

11) **13 MCA files**
Set to '**yes**' if you want to record the whole fluorescence signal for each detector element. This will create 13 extra files other than the standard file containing the energy scan of the integrated fluorescence line of the element of interest.

12) **Data file name**
Enter the file name with a suitable extension (ex. '.txt', '.dat'...). Data are recorded in ascii form. The application will add a numbering (01, 02...) before the extension to distinguish repeated spectra.

13) **How many scans?**
Enter the number of repeated spectra. For each spectrum the software will add a prefix before the extension (01, 02...) depending on the number of scans you have chosen.

14) **Scan type:**

'**absorption**'
For acquisitions in transmission mode.

'**multidetector Digital DXP**'
For fluorescence mode acquisitions.
You must have properly initialized the Multidetector and quitted the STANDALONE application before launching the scan (refer to the 13-element detector paragraph).

'**multipurpose**' Select this mode for custom acquisitions, for example if you are acquiring the electron signal (Total Electron Yield) or detectors other than the 13-element germanium.
You must select this mode also if you are acquiring in transmission mode with a reference compound in the Reflexafs chamber (usually recorded in the 7th column of the exp. file).

15) **Apply scan data**
Click this button to load parameters. An error message will pop up if the parameters are incorrect (i.e. non-monotonous energy intervals, missing steps or integration times...). In this case check the parameters and click again on **'Apply scan data'.**

16) **Execute scan**
Click this button to start the scan. A graphic window will pop up to show the current acquisition progress.

TROUBLESHOOTING

Problem	Possible reason
The transmission EXAFS scan is noisy	The sample is not correctly aligned
	The amplifier of the I1 chamber is saturating (i.e. read out voltage is > 8)
	The sample is extremely inhomogeneous.
Unusual background on the fluorescence EXAFS spectra with the Ge detector	'Cursors' are not correctly positioned.
	Presence of an emission line under the selected line.
	Total count rate too high compared with the peaking time.

WORKING WITH MACROS

Application: MACRO_TEMPLATE
Location: MACROS folder RITAPCLINUX's desktop
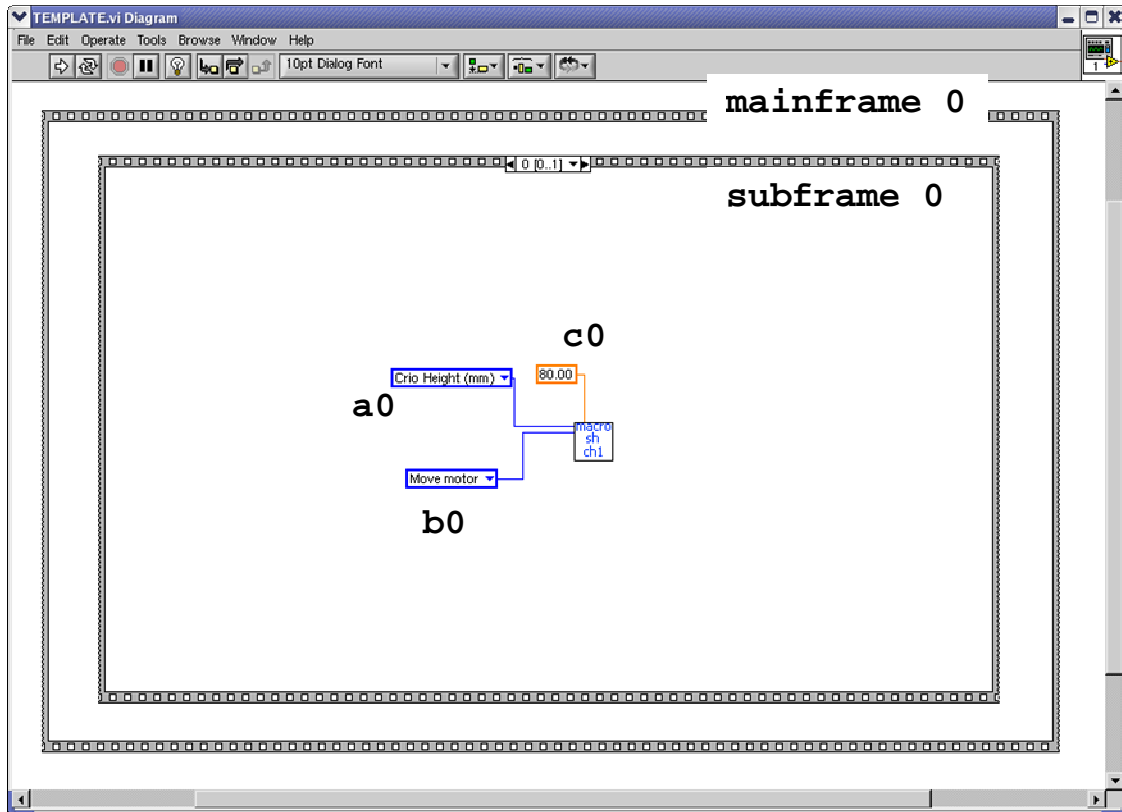
Macros are a powerful way to perform multiple
samples acquisitions without requiring your
presence on the beamline 24h/24h.
Nevertheless, since using macros you will not be
there checking that everything is running fine (for
example during night shifts), a great care must be
put in preparing and documenting each macro.

Macros are composed by frames and subframes, which
number and order is reported in a box at the top
center of the frames border.
Frame numbering starts from 0 and you can add as
many frames and/or subframes you need.
Frames are executed sequentially in the order given
by their numbering.
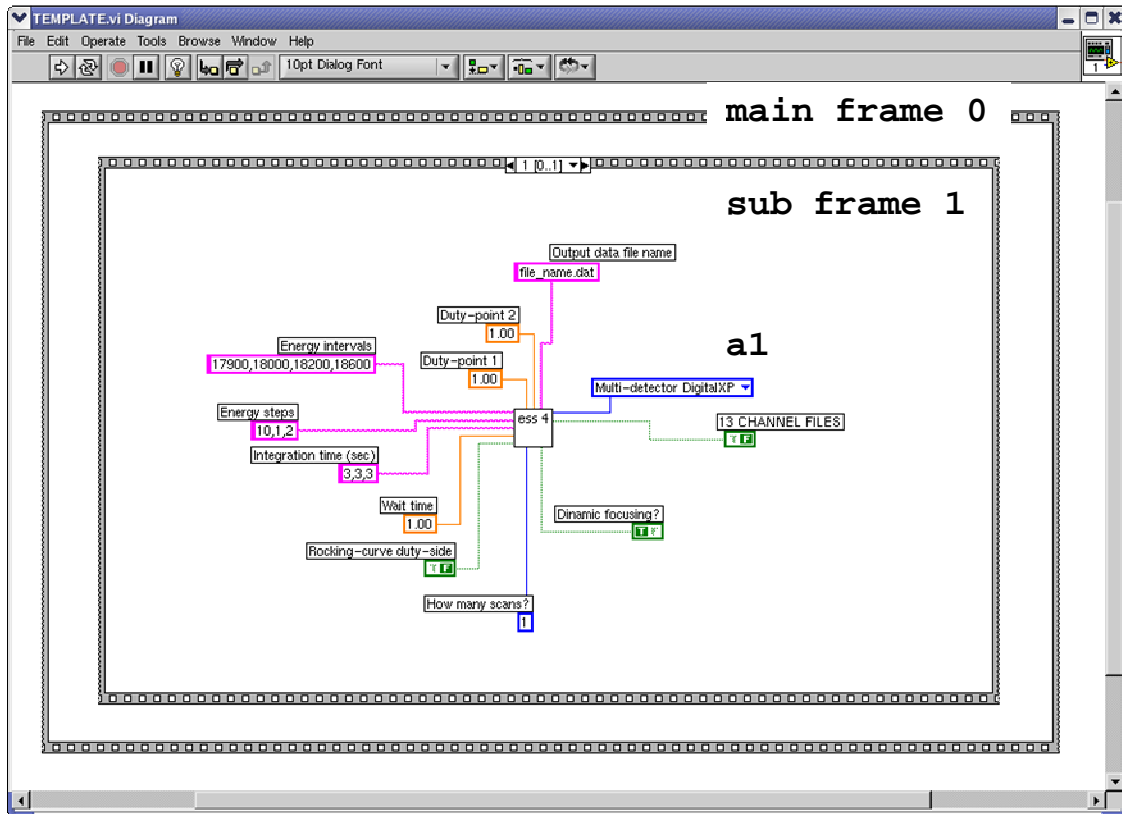
Main frames contain all the operation you want to perform on a specific sample while, each subframe corresponds to a single operation on a given sample.

There are two types of operations you can perform on a sample: the alignment (subframe 0 in the figure) and the acquisition of the spectra (subframe 1 in the figure).

The MACRO_TEMPLATE file comes with one main frame (one sample) and two subframes:
subframe 0 contain the sample holder vertical movement and subframe 1 the acquisition of a typical XAS spectrum with default scan parameters.

Add/removing frames

If you need to add a frame (or a subframe) you have to right-click on the frame border and choose the '**duplicate this frame**' entry from the menu.
This will create a copy of the frame placed right after the frame you have chose to duplicate. The numeration of the frames will automatically arrange to include the new frame.
If you want to delete a frame, right-click on the frame border and choose the '**delete this frame**' entry from the menu.

USAGE

0) BEFORE preparing the macro, you must retrieve the samples positions using the SAMPLE_HOLDER application as explained in the ALIGNING THE SAMPLES paragraph.

1) Open the MACRO_TEMPLATE.vi file located in the MACROS folder on the desktop.

2) From the menu bar, choose the '**show window diagram**' entry from the '**window**' menu. A window will pop up showing one main frame and two subframes: one sample movement (subframe 0) and one acquisition (subframe 1).

3) Likely you would like to add a horizontal movement of the sample holder and/or to set the detector distance from the sample.
Select the subframe 0 by clicking on the left or right arrows on the top center of the subframe border.
Right-click on the subframe border and choose the '**duplicate frame**' entry from the menu.

You have now added a frame (subframe 1) identical to subframe 0 and located right after (Note that former subframe 1 has changed to subframe 2).

4) In the subframe menu **(a0)** choose one of the following entries which act on the sample holder (or detector) movements:

Horizontal   horizontal position
Vertical   vertical position
Rotation   angular position
Multidetector   detector position

5) Set the menu **(b0)** to '**move**' entry, if different.

6) Set the desired position value in the box **(c0)**.

7) Using the arrows, move to the acquisition subframe (subframe 1 in the original Macro_template file) and fill the boxes with the appropriate scan parameters as discussed in the section AQUIRING A XAS SPECTRA.

8) Select the acquisition mode from menu **(a1)** and refer to the following table to set Boolean indicators (T true, F false):

DUTY SIDE	T = pos. derivative (default value)	F = neg. derivative
DYNAMIC FOCUSING	T = YES (default value)	F = No
13 MCA FILES	T = YES	F = No (Default value)

9) Type the data file name in the file name box.

5) If necessary, add a new main frame to operate on another sample:

right-click on the **main** frame border and choose the '**duplicate this frame**' entry. You have now a main frame (numbered 1) identical to frame 0.

6) Adjust the sample and detector position for the new sample by changing values in the subframes as explained in point 4.

7) Adjust scan parameters (if necessary) **and remember to change the file name!**

8) Repeat this procedure for all samples you want to measure (the sample holder can contain up to six samples).

9) Save the macro by choosing the '**save as**' entry in the '**file**' menu of the MACRO_TEMPLATE window. **You cannot overwrite MACRO_TEMPLATE file. Choose a different file name for your macro.**

10) Once saved, run the macro by clicking the white arrow at the top left of the macro window.
Remember to quit all other running Labview applications BEFORE running the macro!
It is also recommended that you wait for the acquisition of a few energy points BEFORE leaving the beamline.

WARNING

1) **Remember to choose a different file name for each sample**
If the macro finds a file with the same file name it will prompt for a 'overwrite file' confirmation and will wait until you answer.
To be sure to avoid this problem it is highly recommended moving all files in your experiment folder in a subfolder, so leaving the main folder empty.

2) **Carefully check the sample positions**
In the recorded files there is no information about the position of the samples.
Therefore, it is not possible to have trace of what went wrong (for example understand if you measured one sample for another or the same sample twice).
To help avoiding such mistakes it is recommended to carefully note the position and name of the sample on the logbook and to save a copy of the macro file in the experiment folder.

3) **Carefully check the scan parameters!**
An error in the definition of the parameters (for example missing energy steps or integration times) causes the macro to stop. The use of the 'duplicate this frame' entry should avoid these mistakes.

EXPERIMENTAL FILES

A different type of experimental file is recorded for each acquisition modes.
Exp. files are organized as follows:

Absorption (transmission acquisition)
5 columns

COLUMN	1	2	3	4	5
CONTENT	ENERGY	I0	I1	mu	ENERGY2

Where,
$mu=\ln(I0/I1)$ is the absorption and ENERGY2 is the energy red by an auxiliary encoder (to be ignored in normal operations)

Multidetector DXP digital (fluorescence acquisition
21 columns

```
COLUMN    1        2          3         4    5   ... 17
CONTENT   ENERGY   I0         I1        mu   ch1 ... ch13


COLUMN    18       19         20        21
CONTENT   ENERGY2  Inor       Irifl     mu2
```

Where,
ch1...ch13 are the 13 detector's elements Inor and Irifl the intensity red by the ionization chambers before and after the ReflEXAFS exp. chamber and mu2=ln(Inor/Irifl).

Multipurpose (custom acquisitions)
9 columns

```
COLUMN    1    2  3  4      5   6     7      8     9
CONTENT   E1   I0 I1 Ifluo  Ie  Inor  Irifl  Idet  E2
```

Where,
E1 and E2 are the Energy1 and Energy 2 columns.
Ifluo is the single element detector signal
Ie the TEY signal
Inor is the first ReflEXAFS ionization chamber
Irifl is the second ReflEXAFS ionization chamber. **This channel is usually used for the simultaneous acquisition of a reference compound spectrum.**
Idet is the signal from a generic detector.

TRANFERING EXPERIMENTAL FILES

Experimental files are located in a working folder 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:

EXAFS_USER_DATA/*current-year/current-run/your-experiment-folder*

You can find the EXAFS_USER_DATA folder on the desktop.
User's PCs (RITAPC and RITAPC3) have a network folder, located in the taskbar at the top of the display, which points to the EXAFS_USER_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 a user PC. Form User's PCs you can copy the files using a USB device or burn a CD/DVD if you prefer.
Do not connect USB devices to the acquisition PC!

PLOTTING EXPERIMENTAL FILES

Experimental files are described in detail in the EXPERIMENTAL FILES section above in this manual.

ABSORPTION files (Transmission mode)

Column 1 (energy)   = abscissa.
Column 4 (absorption)= ordinata.

MULTIPURPOSE files

Column 1 (energy) = abscissa.

To plot the absorption you have first to perform the logarithm of the ratio between column 1 (I0) and column 2 (I1).

To plot the absorption of a reference spectrum you have first to perform the logarithm of the ratio between column 2 (I1) and column 7 (Irifl).

To plot a custom signal you have to plot the ratio of the column containing the custom signal and column 1 (I0) in order to normalize the signal.

MULTIDETECTOR files

Column 1 (energy) = abscissa.

To plot the absorption you first have to check which detector channels (recorded in columns 5 to 17) have a reasonable noise (good channels).
Then plot the ratio of the sum of the good detector channels and column 1 (I0) divided by the number of the good channels:

mu_fluo = (ch1+...+ch13)/13*I0

You can use the **LabPlot** application (a standard GUI plotting application described in the next section) to plot exp. files on the acquisition PC.

WARNING

It is strongly recommended that you transfer the exp. files on the other beamline PCs for the analysis to avoid conflicts with the acquisition.

SOME USEFUL APPLICATIONS

ABSORBER, CHAMSAT, CHAMABS, FLUX
These applications are available at the GILDA web
page and are linked on the desktop of the
acquisition PC.

These applications serve to set correctly the
parameters of the ionization chambers (gas pressure
and type, desired absorption, saturation, flux
calculation).

The following table reports the length of each of
the four ionization chambers present in the XAS
exp. hutch:

	I0 (cm)	I1 (cm)	Inor (cm)	Irifl (cm)
Lenght	15	40	15	15

ABSORBER

Provides the absorption of an ion-chamber filled
with a given gas pressure.
It requires the input of the following parameters:

INPUT ENERGY   (KeV)
CHAMBER LENGTH   (cm)
GAS PRESSURE   (mbar)
GAS TYPE   (N, Ar or Kr)

CHAMABS

Provides the correct pressure to obtain a given
absorption. Requires the following parameters:

DESIRED ABSORPTION   (a value in the range [0,1])

```
INPUT ENERGY              (KeV)
CHAMBER LENGTH            (cm)
GAS TYPE                  (N, Ar or Kr)
```

FLUX

Provides the photon flux for a given detector current. Requires the following parameters:

```
READOUT VOLTAGE                  (V)
AMPLIFICATION                    (integer value)
INPUT ENERGY                     (KeV)
EFFICIENCY   (absorption)        (0...1)
ENERGY TO CREATE AN e-h PAIR     (eV)
(for ionization chambers leave to default value)
```

CHAMSAT

Provides the saturation pressure of an ion-chamber
Requires the following parameters:

```
INPUT ENERGY               (KeV)
CHAMBER LENGTH             (cm)
CHAMBER SPOURIOS LENGTH    (leave to default value)
GAS TYPE                   (N, Ar or Kr)
```

LiveESTRA AND **qESTRA**
These applications are installed on the acquisition PC.

LiveESTRA

This application performs an automated extraction of the EXAFS signal during the acquisition.
Once launched, the application reads the current acquisition experimental data file every 30s,

performs the extraction of the EXAFS signal and its
Fourier transform in the real space and shows the
result in a graphic window, without any external
intervention.
It also gives statistical information, such as
noise and standard deviation, and an estimation of
the edge step and the absorption edge (E0) values.

It can be useful to help judging if you acquiring
good quality spectra.

To start the application double-click on the
corresponding icon on the desktop. Adjust the size
of the graphic window for an optimal view.

To stop the application press control+c in the
terminal window (not the graphic one).

DETAILS

Live ESTRA noise estimation

Noise estimation is performed by subtracting high
degreee polynomials to the EXAFS signal. The
residual should be the statistical (white) noise.
This is not always true. For example in the near
edge region the amplitude of the EXAFS oscillation
is, in some cases, high enough to prevent the
polynomials from following the signal.
It has to be kept in mind that the calculated value
of the noise has not a real statistical value. It
is just an indication of the relative noise. For
good noise estimation you should wait a high k
region.
Further, the noise value should be multiplied for
the edge step to approach a real statistical value.
Keeping in mind the above considerations,
transmission spectra with a calculated noise less
than 0.0005 and fluorescence spectra with a noise

value less than 0.001 can be considered as good quality spectra.

qESTRA

qESTRA performs the same operations of LiveESTRA but can be used on files already acquired.
Further, this application permits the extraction of several files at the same time for a rapid comparison.

USAGE

1) Open a terminal window in the directory containing the exp. files.

2) Type 'qestra' to start the application.

3) Type the number of files you want to analyze and press enter.

4) Type the file names and press enter.

A graphic window will appear showing in sequence:

The raw absorption spectra
The pre-ege subctracted spectra
The EXAFS signals
The Fourier transforms
The noise estimations

WARNING

If you are using custom acquisition mode (multipurpose) and a non standard detector (scintillation, TEY, single element...) you will have to specify the column for the detector signal in the configuration file (see 'Advanced' section).

ADVANCED

LiveEstra and qESTRA have a common configuration file named '**settings.txt**'.
A shortcut to this file is on the desktop of the acquisition PC.
You can change many settings but, likely, you may only need to update the 'column settings' section reported below:

```
----------------------
columns settings
----------------------
1 E
2 i0
3 i1
0 (0: log(i0/i1), 1: i1/i0, 2: i1)
```

Variables reported above are the default one for a standard transmission acquisition:

COLUMN	CONTENT
1	energy
2	I0
3	I1
4	absorption = ln(I0/I1).

For custom acquisitions (multipurpose mode) using a non standard detector (scintillation, TEY, single element...):

1) Change the column number for I1 with the column number of the custom signal (see the EXPERIMENTAL FILES section).

2) Change the last row of the 'column settings' section accordingly with the type accordingly to the following table:

OPTION	OPERATION
0	ln(I0/I1)
1	I1/I0
2	I1

TROUBLESHOOTING

PROBLEM	SOLUTION
LiveESTRA or qESTRA does not start	Delete the configuration file 'setting.txt' located in the folder: 'EXAFS_USER_DATA/quickestra/' If you are performing non standard acquisitions update the configuration file as explained in the ADVANCED section.

ABSORPTION EDGES AND FLUORESCENCE LINES TABLE

A application from the IFEFFIT package (**Hephaestus**) is available for information on any element such as absorption edge and fluorescence line energies, density etc.

To start the application double-click on the icon on the desktop. This application is installed on any beamline PC except the acquisition PC.

XAFSMASS

With this application you can calculate the quantity (in mg) of powder needed to prepare

samples with an optimal absorption for transmission
measurements.
The application requires:

1)   Sample chemical formula
2)   Desired total absorption (optimal value 1.388)
3)   Sample area
4)   Absorption edge energy
5)   Sample density is required only if you want to
      know the sample thickness

GENERAL TROUBLESHOOTING

1. BEAMLINE'S SERVERS AND ERROR MESSAGES

Each beamline's hutch has one or more servers which bridge commands and information from the control cabin and viceversa.

The servers are listed in the following table:

SERVER LOCATION	SERVER NAME
OPTIC HUTCH (HUTCH 1)	ritavme2
XAS HUTCH (HUTCH 2)	ritamac10
XRD HUTCH (HUTCH 3)	ritavme3

If you get an error message from one of the servers, likely, you run more than one LABVIEW applications at the same time.
If this is the case try the following procedures one after the other until you solve the problem:

- QUIT/EXIT all running LABVIEW applications and run only the application you need.

- Restart LABVIEW by accessing the FILE menu of one of the LABVIEW application windows and choose the QUIT entry. Answer YES when prompted 'close all running vi's?'.
  If LABVIEW does not responds open a terminal window, type 'ps' and hit enter to locate the LABVIEW process ID number. Type 'kill -9' followed the LABVIEW ID number and hit enter.

- Restart the servers (see point 3.)

- Restart the acquisition PC (see point 4.)

3. RESTARTING THE SERVERS (VMEs)

If procedures listed above did not solve your problem or if your acquisition freezes (no increase of the energy points for a time much longer than the integration time chosen), you may have to restart both the OPTIC HUCTH SERVER (RITAVME2) **AND** one among the XAS (RITAMAC10) or the XRD (RITAVME3) servers, depending on your experiment.

To restart a server:

  a) Quit ALL LabView applications

  b) Open a terminal window on the acquisition PC
     and connect to the server by typing:

         ssh root@*servername*.esrf.fr

  d) login using the root password (scassaciufoli)

  e) type '**reboot**' and hit enter

  f) close the terminal window and wait 5 minutes
     before restarting LABVIEW applications.

2. RESTARTING A PC

If the PC has crashed turn it of by holding the power button pushed for at least 5 seconds. Push the power button again to restart the PC and wait the operating system has loaded.
Insert username and password.

All WINDOWS beamline's PCs have the following login parameters:

Username   opd08s
Password   tonic08

All LINUX beamline's PCs have the following login parameters:

Username   opd08
Password   tonic08

3. RESTARTING VNC CONNECTION WITH RITAPCU2

This connection redirects the display of RITAPCU2 which is located in XAS EXP. HUTCH and runs EXAFS_TN, the server for the 13-element detector, the He/N cryostat and the ionization chamber amplification application (KEITHLEY)   .

If this connection is down, double click on the VNC icon on the desktop and type the following login parameters in the dialog window:

Connect to   ritapcu2
Password   tonic08

4. RESTARTING EXAFS_TN

As explained in the body of this manual, this application controls the vacuum pumps of XAS EXP. CHAMBERS 1 and 2, the EXAFS slits and XAS EXP. CHAMBER 2 motors.

If you get an error message from this application ('TCP/IP connection error') do the following:

a) Click 'OK' buttons on the dialog windows until the application closes.

b) **From RITAPC1** connect to the EXAFS_TN server by opening a terminal window emulator (for example Putty) and connect to the following address using the TELNET protocol:

```
        telnet ritavme1.esrf.fr

        login using:

        username     giu_ro
        password     sof97
```

c) Type the command '**break**' and hit enter

d) Type **'logout',** close the terminal window and wait 3 minutes to restart EXAFS_TN by double-clicking on the corresponding icon.

5. RESTARTING LABVIEW

In some cases, when a application does not respond and you are not able to quit it, you will need to restart LABVIEW.
To do it go on the file menu on the application window and choose the quit entry. Answer YES while asked to confirm closing all the running applications.
To restart LABVIEW just start again the applications you need by double-clicking on the corresponding icons in the EXAFS, REFLEXAFS, XRD or MACRO folders or start the CONTROL PANEL you need, if you are working with panels.

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