

Table of Content

1. SETTING UP THE STATION ON ARRIVAL	4
1.1 Safety procedures	4
1.2 Turning on the X-ray beam.....	4
2. SAMPLES PREPARATION	5
2.1 Transmission measurements	5
2.2 Fluorescence measurements	6
3. DATA ACQUISITION SOFTWARE	6
4. POSITIONING YOUR SAMPLE	8
4.1 Moving the Sample stage	8
4.2 Use of the Cryostat	10
4.3 Transmission measurements	11
4.4 Fluorescence measurements	12
4.4.1 Saturation of the detector	14
4.4.2 Filling the detector with LN ₂	15
5. SCANNING	15
5.1 Editing the regions	16
5.1.1 k-space scanning	16
5.1.2 Angle/Energy-space scanning	18
5.2 Scanning	19
5.3 Quick EXAFS	20
Problems with QEXAFS	21
5.4 XRD measurements using the INEL detector	22
5.5 Plotting in Dubble7.....	22
6. DATA TRANSFER	24
6.1 Raw Data format	24
6.1.1 Transmission file	24
6.1.2 Fluorescence file	24
6.2 Transformation of the data in XY files	25

7. CALIBRATING THE MONOCHROMATOR.....	26
8. FILLING THE ION CHAMBERS**(EXPERIENCED USERS)	28
8.1 Ion Chambers Amplifiers	28
8.2 Filling the Ion Chambers	29
9. ALIGNING THE BEAM** (EXPERIENCED USERS)	33
9.1 Moving the monochromator.....	33
9.2 Setting the Monochromator crystals	34
9.2.1 Pitch of the monochromator m3	35
9.2.2 Linear positioning of the second crystal mtrans	35
9.4 Setting the Mirrors.....	36
9.4.1 Height of the mirror hgt	36
9.4.2 Strip of the mirror m4 m5	36
9.4.3 Bending of the mirror b1 b2	37
9.3 Setting the slits.....	37
9.3.1 Vertical slits.....	38
9.3.2 Horizontal slits	39
10. BASIC UNIX COMMANDS	40
11. SHORT SUMMARY.....	41
11.1. Turning on the X-ray beam.....	41
11.2. Starting data acquisition program.....	41
11.3. Moving the Sample stage	41
11.4. Quick edge step check.....	41
11.5. Starting a Scan.....	42
11.6. Quick optimization of the beam	42
11.7. Transformation of the data in XY files	42
12. TROUBLE SHOOTING	43
12.1. Acquisition Software	43
12.2. X-ray beam.....	43

1. Setting up the station on arrival

On arriving at the station at the start of a visit there are a number of procedures that must be undertaken before you can start your experiment. Most of these procedures **concern** the setting up of the station but first the safety requirements of the ESRF must be satisfied.

1.1 Safety procedures

Each User **must have passed the Safety Training course** on the ESRF intranet at <http://smis2.esrf.fr:8080/smis/servlet/SafetyTraining?startgst> **before starting the experiment**). Your Safety Approval Form (SAF) should be collected from the safety office and put on the notice board by the Beamline. If you are using hazardous chemicals/equipments you will be given a **Red experiment** SAF that includes special restrictions and obligations.

1.2 Turning on the X-ray beam

Once the search procedure using the PSS has been completed X-ray beam may be admitted to the hutch by using the control sub-window in **Shutter application S-line** window that is activated by double clicking on the valve 'safety shutter soft line' in the schematic diagram. **Shutter application S-line** can be opened via the S-line button on the right hand side of the menu bar at the bottom of **Dubble7** display.

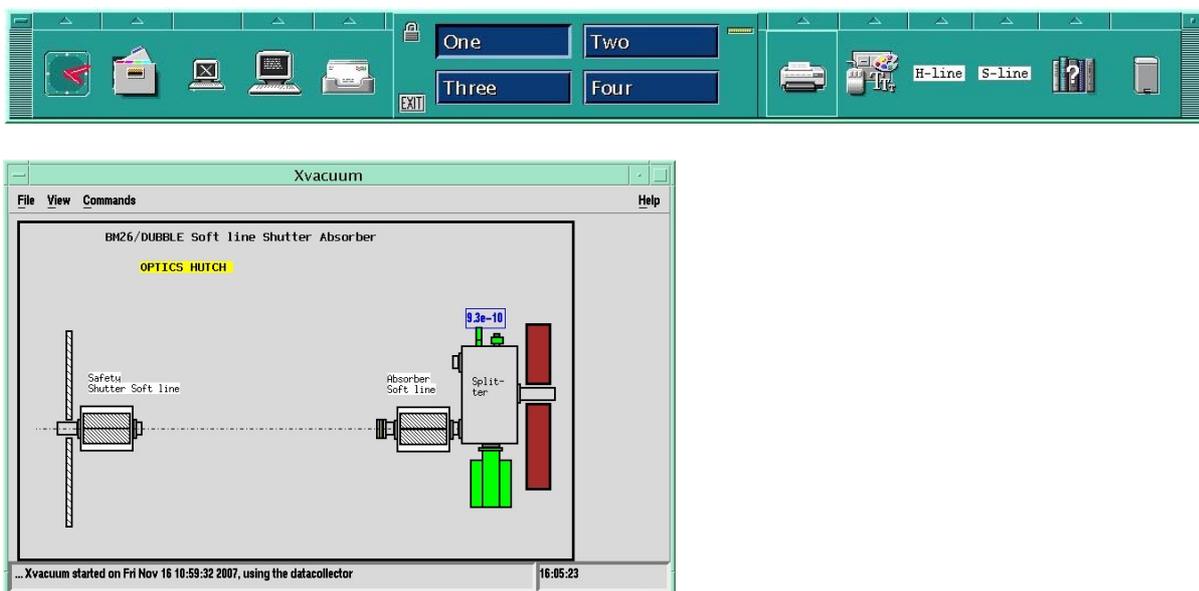
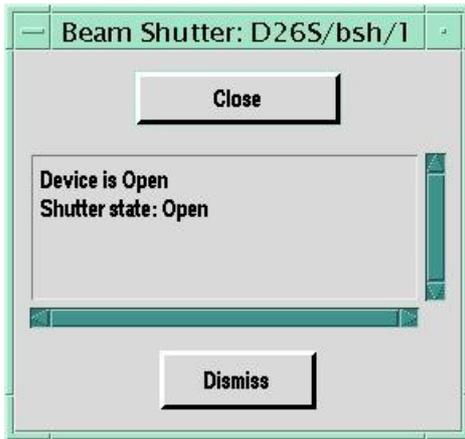


Figure 1



The valve will open if you select the '*Open*' button. To turn off the X-rays, select '*Close*' in the status window before opening the hutch.

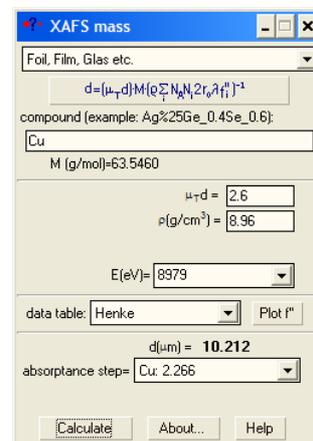
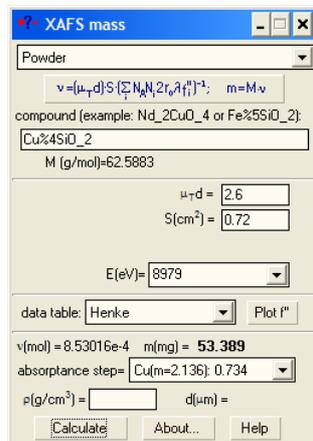
X In normal circumstances, only this valve needs be either opened or closed.

2. Samples Preparation

Sample preparation is a crucial step in an XAFS experiment. You will save a lot of time and get optimum quality data if you have already worked out the right concentration in scattering element.

2.1 Transmission measurements

The sample must have the right absorbance to produce a suitable jump. The optimum dilution obtained generally with BN or the thickness of the cell/sample can be determined for instance with the program *XAFSmass* that can be downloaded at <http://www.desy.de/~klmn/xafsmass.html>. Calculation of mass and absorption step for powder samples (left window) or calculation of thickness and absorption step for samples with known density (right window) can be performed.



2.2 Fluorescence measurements

The samples should have a dilution at least lower than the required dilution for the transmission measurements. Non-diluted samples will generate a strong self-absorption that will increase the intensity of the pre-edge region, XANES regions and decrease the intensity of the EXAFS oscillations. That will lead to unreliable XAFS results.

X To collect high quality XAFS data, concentration, homogeneity, grain size of your samples must always be optimised.

3. Data acquisition software

The Station is controlled through the program **exafs** running on the Sun workstation **Dubble7** connected to a data acquisition VME crate. The computer logon i.d. is **opd26** and the password will be given to you by your local contact.

First you need to load a 'settings file' by typing **exafs_interface** at a prompt in a local terminal window on the workstation. Two icons named "McLennan" and "Inel" should appear on the top left corner of the DUBBLE7 screen desktop.

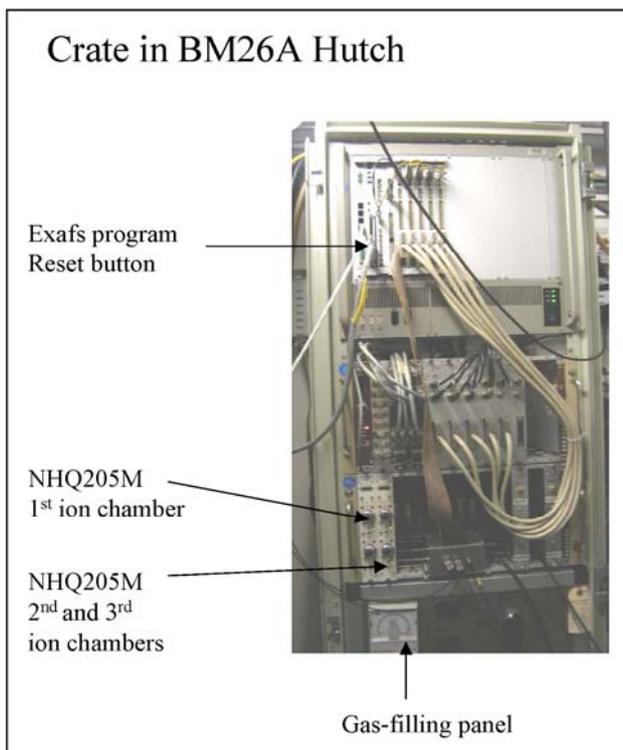
X These programs must be running before the EXAFS data acquisition program is started!

3 different windows **SOFT>** , **MONOS>** and **MIRRORS2>** will enable you to control respectively the sample position + slits, monochromator pitch and mirror height. For the first utilization these windows are opened up by typing **SOFT**, **MONOS** and **MIRRORS2** followed by enter in a terminal window.

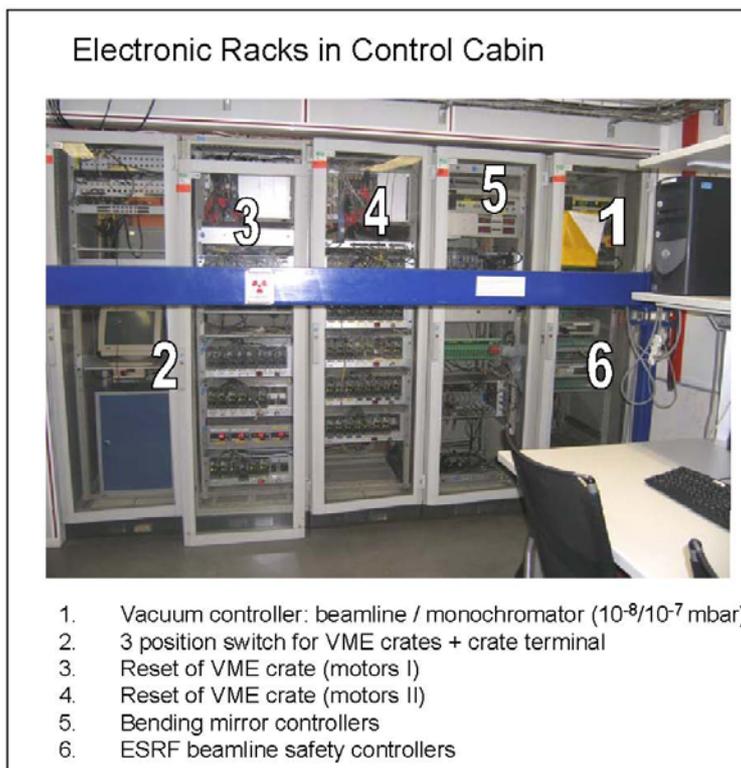
To open a terminal window click on the terminal icon on the menu bar at the bottom of the page. To start the acquisition program just type **exafs** that will bring up the main window EXAFS data acquisition program. It is a GUI program and most of the functions are fairly self-explanatory. (**Monitor** button opens the monitor window and pressing **Setup** button will open the experimental setup window).



If the program fails to start try typing **kill_exafs** in a window and then type **exafs**. If the program loses contact with the VME system then you may need to reset the VME. Before doing this quit the **exafs** program by clicking the **Exit** button on the main EXAFS window and re-enter it after the VME-crate has been reset. To reset the VME you need to enter the experimental hutch, locate the crate in the electronic rack (above the gas filling rig) and press the **reset** button 2 seconds (very small red button) on the MVME 162-522A unit at the LHS of the crate just below the VME system hard drive. After about 2 minutes you can start the **exafs** program on **Dubble7** again by typing **exafs** in a new window.



x Do not start a new **exafs** program if it is already running as this may cause severe interference problems!!



4. Positioning your sample

It is crucial for your sample (and the ion chambers) to be in the path of the beam in order to collect data. This alignment is made using fluorescent paper stick on the edge of a sample holder with a known distance from the sample.

4.1 Moving the Sample stage

The motors controlling the position of the sample stage as well as the Fluorescence detector are moved from the **SOFT**> window that is accessed by typing **soft** in Spec>

Sample stage motors settings:

- **sampley** = vertical movement (becomes **cryox** when the cryostat is installed)
- **samplex** = horizontal movement (becomes **cryoxy** when the cryostat is installed)
- **gonio** = rotational movement around y-axis (+ clockwise - anti-clockwise)
- **cradle** = vertical angle (not always implemented)
- **detect** = fluorescence detector movement towards and backwards relative to the sample

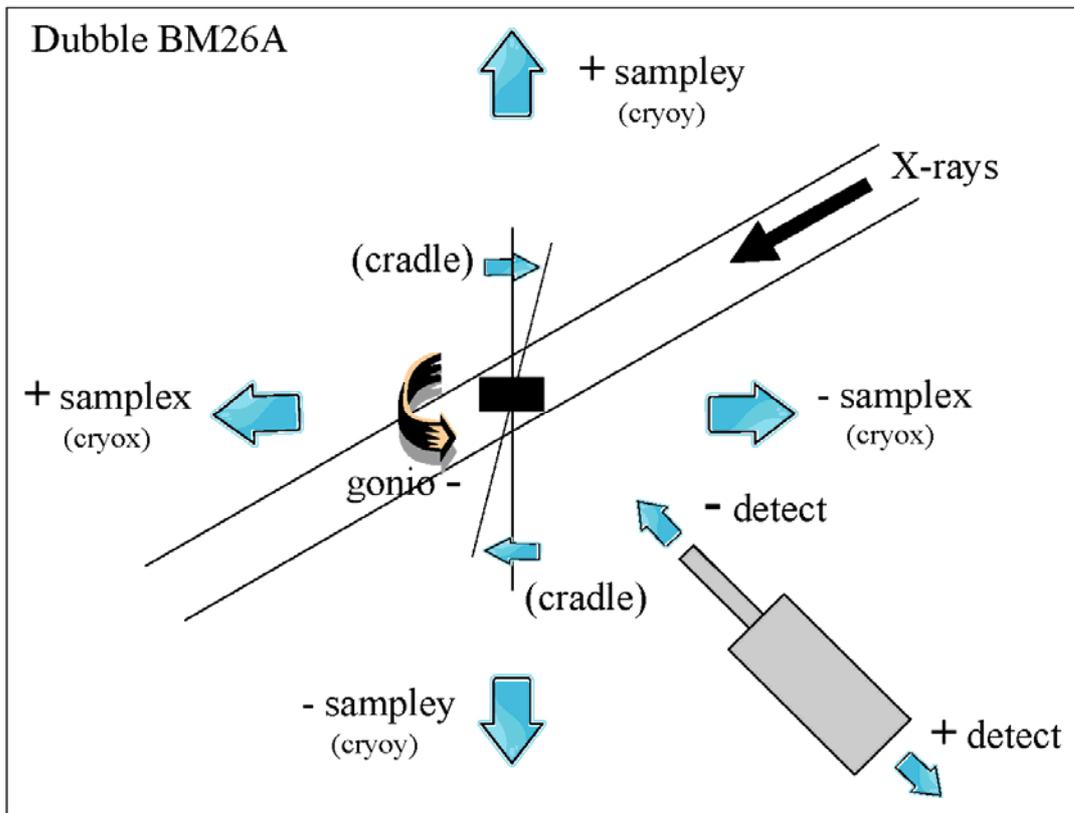
✘ Using negative *detect* values moves the detector towards the sample (also valid when using negative values of *samplex*).

► Command:

SOFT> **wm** [name of the motor] will give you the current positions of the corresponding motor.

```

Terminal
Window Edit Options Help
2709.SOFT> mvr sampley -0.5
2710.SOFT> mvr sampley 1
2711.SOFT> mvr sampley 4
2712.SOFT> mvr sampley 2
2713.SOFT> mvr sampley -1
2714.SOFT> mvr samplex 1
2715.SOFT> mvr samplex 1
2716.SOFT> mvr samplex 1
2717.SOFT> mvr samplex 1
2718.SOFT> mvr sampley -4
2719.SOFT>
    
```



► Command:

SOFT> **mv** (absolute movement) and **mvr** (relative movement)

change the current motor positions

mv [motor] [absolute value mm]

mv [motor] [relative value mm]

Example : **mvr sampley 0.1** (in mm)

✘ Be very careful in using the **mv** command since the sample stage may hit an object if you don't have in mind the absolute range of variation of the motors. Always prefer the **mvr** command.

► Command:

SOFT> **dscan** (relative scan)

This will plot generally the intensity of the 2nd ionisation chamber current as a function of the position of the selected motor. That will help you to position accurately your sample in the beam. Type “dscan” and press *enter* to see a tip how to use this SPEC macro.

dscan [motor] [relative starting point] [relative last point] [intervals between points] [measuring time second]

Example : **dscan samplex -3 3 60 1**

If the motor is exceeding its operational range you will get the message:

Warning: Motor 'x' didn't reach final position

★ To change the ionisation chamber, type **plotslect** at a prompt in SOFT> and select i1 or i3 instead of i2.

► Command:

SOFT> **newfile** to create a new file for the dscan data saving.

These scans can also be displayed and saved in the **Newplot** software if you have configured it before use. The **Newplot** program does not belong to the SOFT session. Just open a new terminal window and **Newplot** to start this program. (see section 5.5)

4.2 Use of the Cryostat

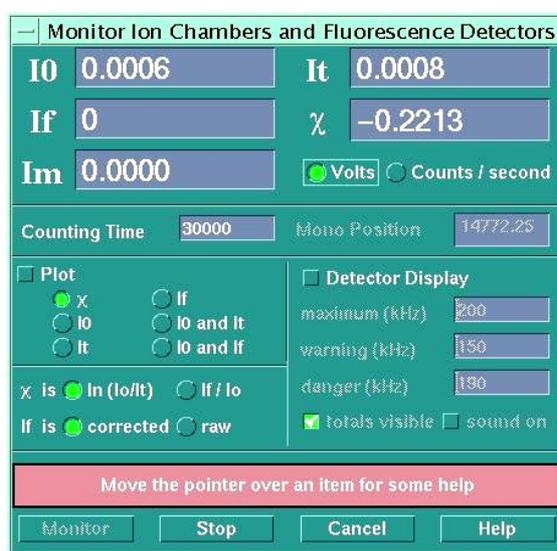
The cryostat can generate minimum temperatures of 12K. It is moved by using the commands **cryox** and **cryoy** (see drawing)

Samples must be by changed under constant He flushing to prevent air to enter the cryostat. If the sample rod gets stuck in the cryostat do not try to pull it out by force as it may be seriously damaged. In this case the cryostat should be warmed up to 80K above the melting point of Nitrogen (63 K) cryostat temperature controller until the ice has melted.

✘ Be very careful by changing samples as nitrogen ice could form around the sample and block the sample rod inside the cryostat.

4.3 Transmission measurements

1. If you wish to do a simple room temperature transmission measurement you position your sample in the beam using the fluorescent paper and the camera.
2. Interlock the hutch using the search procedure.
3. When the hutch is interlocked, turn on the X-ray beam.
4. Ensure the program is in Transmission mode.
5. Before starting a scan it is worth checking that the sample gives a reasonable edge step. Move the monochromator to about 50 eV below the edge (i.e. at a higher angle, lower energy than the edge) pressing start in the *Calibration* subwindow in the *Experimental Setup* window.
6. Choose the *monitor* button on the main program window.
7. Select χ as $\ln(I_0/I_t)$ and note its value and change the counting time to 500.



Monitor Ion Chambers and Fluorescence Detectors			
I0	0.0006	It	0.0008
If	0	χ	-0.2213
Im	0.0000	<input checked="" type="radio"/> Volts <input type="radio"/> Counts / second	
Counting Time	30000	Mono Position	14772.25
<input type="checkbox"/> Plot <input checked="" type="radio"/> χ <input type="radio"/> If <input type="radio"/> I0 <input type="radio"/> I0 and It <input type="radio"/> It <input type="radio"/> I0 and If		<input type="checkbox"/> Detector Display maximum (kHz) 200 warning (kHz) 150 danger (kHz) 100 <input checked="" type="checkbox"/> totals visible <input type="checkbox"/> sound on	
χ is <input checked="" type="radio"/> $\ln(I_0/I_t)$ <input type="radio"/> If / I0 If is <input checked="" type="radio"/> corrected <input type="radio"/> raw			
Move the pointer over an item for some help			
Monitor		Stop Cancel Help	

8. Similarly by pressing *End* move the monochromator to 50 eV after the edge.. The difference between the new value of χ and the old value is the edge step. Ideally for a transmission sample this should be near 1.0. It can be as low as 0.1 with an absorbing sample but should not exceed 1.5. If it is too big try reducing the amount of sample in the beam. If it is too small either put more sample in the beam or consider doing a fluorescence experiment. Also you should check the gains on the Novelec amplifiers. The DVM readings for I0 and It should lie between 0.1 and 10.0 V

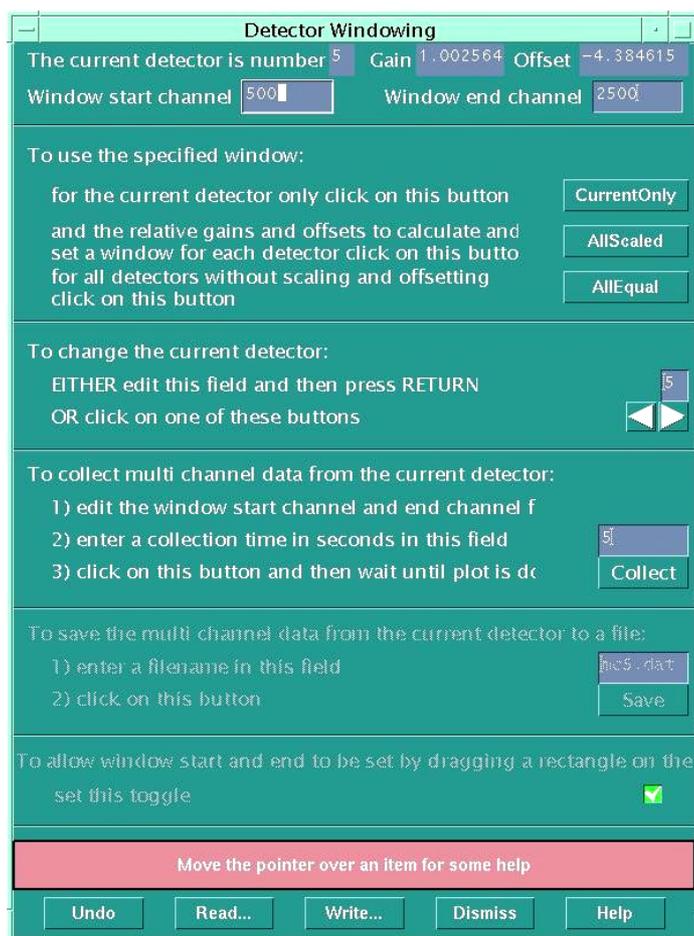
before and after the edge. If they are not, you either need to change the gain on the amplifier or remake the sample.

9. Once the edge step is satisfactory and the amplifiers are correctly set, (i.e. the values lie in the range **0.1 - 10.0 V** both before and after the edge – **1 V** is the optimum value) you are ready to set up the scan.

4.4 Fluorescence measurements

To do the fluorescence measurements the sample should usually be aligned at 45 degrees to the line of the beam, in order to maximize the solid angle seen by the detector. Furthermore the Ge solid-state detector must be tuned to the energy of the element you are studying. This is done in the *exafs* program. For setting up the detector it is useful to have a strong fluorescence signal; thus if your sample is very dilute in the element whose edge you wish to study, it may be advisable to use a sample that is more concentrated to set up the detector. A foil, however, will generally be too concentrated, but if this is all that is available the detector can be set-up using a foil. To avoid saturating the detector, ensure the foil is facing the beam, and that it is roughly parallel to the detector, i.e. minimizing the solid angle of the fluorescence that the detector can see.

1. Position your sample on the optical bench at a reasonable angle using the fluorescent paper and the camera.
2. Ensure the protective shield has been removed from the Ge detector and that it is pointing at the sample.
3. Once the detector is set-up if there is a lot of scatter present it may be worth placing a foil of atomic number $Z-1$, where Z is the atomic number of the element that is being studied, to reduce scatter from the sample.
4. Close up the hutch using the Search procedure and turn on the X-ray beam.
5. Now the detector must be set-up. Move the monochromator to about 50 eV before the edge (i.e. lower in energy) using the button **Start** in the main set-up window.
6. To window individually the 9 Ge detectors go into the menu **Hardware** of the main window of the acquisition programme and select **Detectors...** in the menu. You will obtain a new window **Detector windowing** in which you will be able to set the window start channel for each of the 9 channels (numbered 0 to 8)



7. First you need to enter the window start channel and the window end channel and click the *All equal* button to have all the detectors equals without scaling and offsets. The idea in setting up the detector is to window the fluorescence signal corresponding to the edge of interest, excluding the scatter peak. The entire range of the detector goes from 1 to 8192. For example a 1000-3000 window corresponds to the fluorescence excited by the Fe K-edge. For each separate detector the channels are linearly scaled with the energy but each detector is slightly offset from the other one. So each detector needs to be calibrated separately.

8. Pressing *Collect* with a collecting time of 5s will display the Fluorescence spectrum of your sample. Once the scatter peak is located, if the monochromator is moved to around 50 eV after the edge (higher in energy), near the scatter peak, the fluorescence peak should start to grow. If in doubt as to the identity of this peak, move back before the edge and then past it with the monochromator. The fluorescence peak should only start growing at a fast rate, when the monochromator is past the edge.

9. Having successfully identified the fluorescence peak, it is now time to set the window of each detector around it.

10. To window detector 0, you need to select detector 0 by changing the value in the current detector box by clicking on the right arrow in the section *To change the current detector*. Enter a Collecting time typically 5 s in the section *To collect multi channel...* and click the *Collect* button. Ensure that the toggle in the last section at the bottom of the program window is on, and move the vertical lines to set the limits of the fluorescence peak of interest.

11. Save the peak window settings by pressing *Current Only* in the section *To use the specified window* to save the new lower and upper limits of the current element of detector. When you have effectively saved the settings of the individual detector the background of the Window start channel will change from **brown to grey**.

✘ This has to be done for each detector individually!!

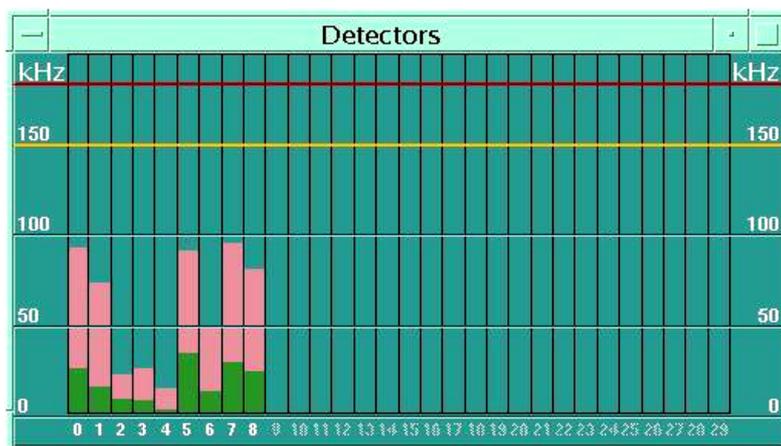
You have now successfully set up the fluorescence detector.

These detectors windows are saved when you write the Fluorescence measurement configuration file in the main set-up window. When you read a configuration file you can check the limits of channel 5 fluorescence peak limits to ensure that the detectors windowing is still valid. (this may have changed in case of a significant variation of the number of counts).

4.4.1 Saturation of the detector

Now it is worthwhile checking that the sample has an edge step and that the fluorescence detector is not being saturated before you commence scanning. Check on the saturation of the detectors.

- 1.** With the sample in position, the X-rays on and the program in Fluorescence mode, move the monochromator about 50 eV before the edge (*Start* position).
- 2.** Start the monitor routine, choosing monitor from the main program window.
- 3.** Change to counting time to **500 ms**.
- 4.** Define χ as I_f/I_0 , check its value. (I_f = fluorescence intensity; χ = contrast)
- 5.** In the *Experiment set-up* window move the monochromator to 50 eV past the edge
- 6.** Again check the value of the absorbance, the edge step will depend on the concentration of your sample but anything less than 0.005 will require many scans to obtain analysable data whereas the 0.05-0.5 range is more normal.
- 7.** By clicking on *Detector display* you will display a bar graph giving you the current intensity received by each one of the 9 detectors. The maximum count rate that can be measured without significant saturation is 150 kHz (1Hz = 1 Count/sec.). Above this value this value the detector is strongly non linear. It is recommended to operate at around 100 kHz. The green part of the bar represents the useful signal while the pink part above corresponds to the background fluorescence.
- 8.** If the count rate is too high, pull the detector away from the sample to reduce the solid angle seen using the **mvr detect** command.



9. If the count rate is low and the edge step is small, you can try moving the detector closer to the sample and optimising the sample position to increase these values.

10. Once the above steps have been completed satisfactorily you are ready to set up the scan.

11. The detectors do not normally need to be reset during the experiment provided the count rate is always constant (around 100 kHz). However if the count rate drops significantly they may need to be reset since the detector channel/energy linearity varies with the count rate. Checking one channel is enough to detect a variation in the fluorescence peak position.

✘ Be extremely careful as to never touch the sample with the detector window since it may break the Beryllium crystal and have serious consequences for you and the station.

4.4.2 Filling the detector with LN₂

The fluorescence detector should be refilled with liquid Nitrogen **on a daily basis** using the large movable Dewar located in the experimental hutch. This is filled at a LN₂ feeding point facility located in the main pathway 20 m from the station.

✘ Be sure you are filling the tank of the detector entirely (at this point Liquid N₂ should flow out of the detector onto the floor)

5. Scanning

Before you can begin to scan you must first set up the regions for the scan unless you already have a configuration file saved that you can read using the *read* button. These configuration files are generally saved in the *users\verylocal* directory.

5.1 Editing the regions

In the *Experiment set-up* window there is an automatic region selection, which provided the element / edge have been selected will produce default regions for calibrating the monochromator and running a k-space scan. These are the buttons marked:

- **calibration** - which produces a single region of about 100 eV centred on the edge position. This will find the edge, providing the calibration of the station is not too far out.
- **real use** - this produces a k-space scan with about 200 eV of pre-edge and a k-space region to 12 keV with a step of 0.05.

These buttons are useful as starting points but the regions they produce may need some adjustment for optimal data collection. The scan regions are edited on the *Experiment Set-up* window in the angle sub-menu.

There are 3 useful sub-windows:

- **Angle** for angle scanning,
- **K-space** for k-space scanning
- **Fast** for QEXAFS scanning.

Most EXAFS scans are now made using the scan k-space option, that enables you to use a fixed k-space step throughout the EXAFS region, however for XANES scans or some specific measurements, scanning is done by monochromator Bragg angle. If you are scanning by angle all the regions you wish to scan must be set up manually (the regions for an angle scan are to a large extent determined by the experiment, hence no details on what regions to use is offered here). For angle scans any number of regions can be used. k-space scans must have three regions and the edge must be in the second region. The method of editing each region is as described in below, if you are scanning in k-space the third region is defined by the k-space parameters. Due to the limitation of the diffraction conditions of the monochromator single crystals, the energy resolution decreases sharply at high energies.

5.1.1 k-space scanning

All editing is done in the *Experiment set-up* window:

Choose the type of scan (Transmission or Fluorescence) and the number of scans (repeats). Then for k-space scanning a default selection can be obtained using the **Real Use** button. (you may have to go up and down twice in the directory tree to actually read the file as the program may bug)

These can then be adjusted to suit the sample requirements, for dilute fluorescence samples you may wish to alter the first two regions, for all samples the k-space region will need optimising, and you may want to reduce the step size in the second region to give more points over the edge, especially if you are interested in the XANES region.

Edit the k-space values:

- Edge position in mDeg - *this can be altered from the defined value if you desire*
- Edge step in mDeg – *minimum 0.4*
- Start of k-space region - *normally a value between 2 and 4*
- End of k-space region - *normally between 10 and 20*
- The k-step - *the interval in k-space between each point: normally between 0.02 and 0.06*
- The k-weighting - *the time for which data is collected at each point is weighted with relation to the k value of that point, thus at higher k where the EXAFS oscillation is weaker more data is collected: normally 2 or 3*

- The time for which data is collected at the last point - *between 5000 and 30000 mSec*
- The time for which data is collected at the first point - *between 1000 and 5000 mSec*

The actual values can be seen by pressing **Apply** and then **Show**. The minimum step size is 0.4 mDeg as the monochromator will not move in smaller steps than this. At high energy thus the start of the k-region may need to be quite high to meet this condition. The corresponding total counting time does not include the dead time and is thus an underestimate.

You are now ready to scan. Press **Apply** and the green **Go** button will now be enabled on the main program window.

5.1.2 Angle/Energy-space scanning

For an angle scan there are four parameters for each region and some general parameters.

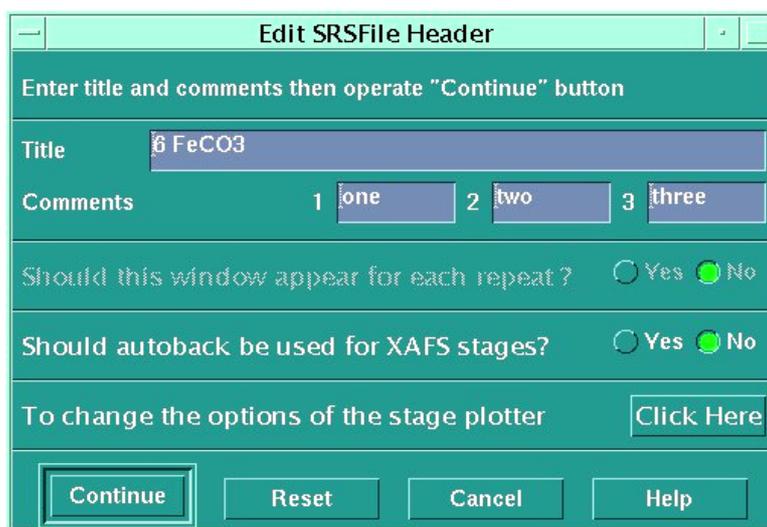


- Start of region in mDeg - *where the computer will start scanning in that region*
- End of the region in mDeg - *where the region ends*
- Step size in mDeg - *what the step is between each data point in the region (min 0.4 mDeg.)*
- Time limit in milliseconds - *how long the computer will collect data for at each point (If you have more than one region the end of each region is automatically defined as the start of the next.)*
- No. of regions - *the number of regions currently active in a scan*

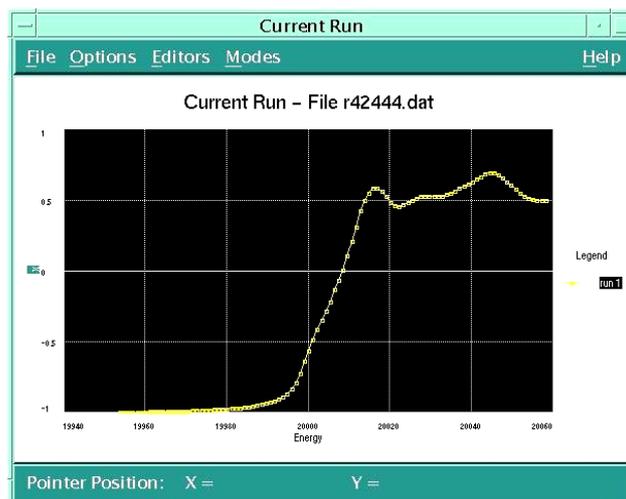
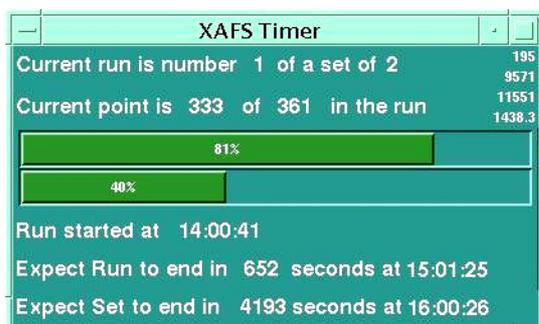
- No. of repeats - *the number of scans that will be done when the scan routine is run*
- Having entered these parameters and chosen **Transmission**, press the **Apply** button at the bottom of the window (the active background will change from brown to grey) and you are ready to scan.
- After a short time the green **Go** button on the main program window is enabled and you can begin to scan by depressing it.

5.2 Scanning

1. Press the Green **Go** button, a **Titles** window comes up.



2. You are prompted for titles in the title window, by pressing on 'Click Here', you will access to a window that will allow you to choose the x-axis as millidegrees or eV. eV is normal for data collection but millidegrees is useful when calibrating. After making your selection press **OK** and then **Continue** in the main titles window. The scan will start.



3. The measured graph in the *Current Run* window automatically rescales vertically during the scan.
 4. A *XAFS timer* window shows the approximate amount of time remaining in terms of data recording to go. This does not include dead time so is a slight underestimate.
- ✘ To abort the scan at any point select the red 'Stop' button on the main program window (It is necessary to apply the regions again after an aborted scan).

5.3 Quick EXAFS

Choose the fast sub-window in the main set-up window. You will see the regions are set automatically for you.

You have the choice of the length of scan between 60 and 1200 seconds, the k-range end (6 - 20 keV) and the k-space step down to 0.04 \AA^{-1} . These are changed using the sliders in the QEXAFS window.

Note that the time set is approximate only as the calculation changes it to get a suitable scan speed. Thus to find out the exact time your scan takes you should time it. As the program calculates theoretically the speed of the monochromator without any possibility to measure the actual angle, the edge position may not be always accurate especially for short scans. A reference foils should always be measured simultaneously. You can only stop a QuickEXAFS scan during the short period of time when the monochromator is idle at the end of a scan (the led next to angle display has a low intensity)

✘ It is not recommended that you use a scan time of less than 120 seconds as in shorter scans the accuracy of the energy scale is not guaranteed.

Then press *apply* and start a scan in the normal way.

✘ Do not abort a Qexafs run between scans or the monochromator will loose calibration. If you wish to abort wait until a scan has started.

Problems with QEXAFS

If in QEXAFS in a multiple scan run a scan fails to start, then abort the scan with the red *Stop* button. Look at the motor controller page or check the reading of the motor encoder on the monitor located on the shelf above the workstation.. When the Bragg motor stops moving it is safe to exit the program and then restart it and continue where you left off. If you issue a command before the monochromator stops, you will probably have to stop the Bragg motor manually.

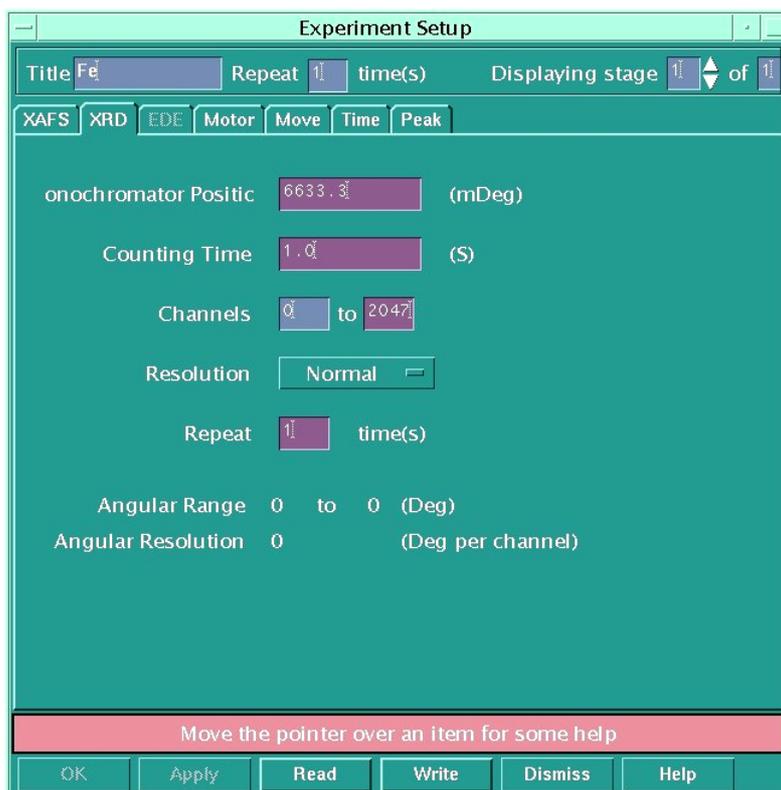
5.4 XRD measurements using the INEL detector

The *exafs* program can control directly the INEL detector program that runs on *Dubble8* to perform combined XAFS/XRD measurements. The detector has to be flushed with a special gas mixture before use. (Pressure 6.3 bar and voltage of 9.8 kV)

To set-up an XRD measurement, select the XRD sub-window in the main *Experiment set-up* window. You must choose the energy of the beam by entering the monochromator position in millidegrees, the counting time in seconds and the start end channels you want to use. The full channel range (0 to 2047) corresponds to a 2theta range of 0 to 90 degrees. The resolution corresponds to a read out step resolution of 0.01 degree by channel.

The sequence of measurement in case of a multi-technique experiment is defined by setting the corresponding value of the *Displaying stage* in the top right of the sub-window. Displaying stage 2 of 2 in the XRD sub-window defines a sequence XAFS–XRD measurements.

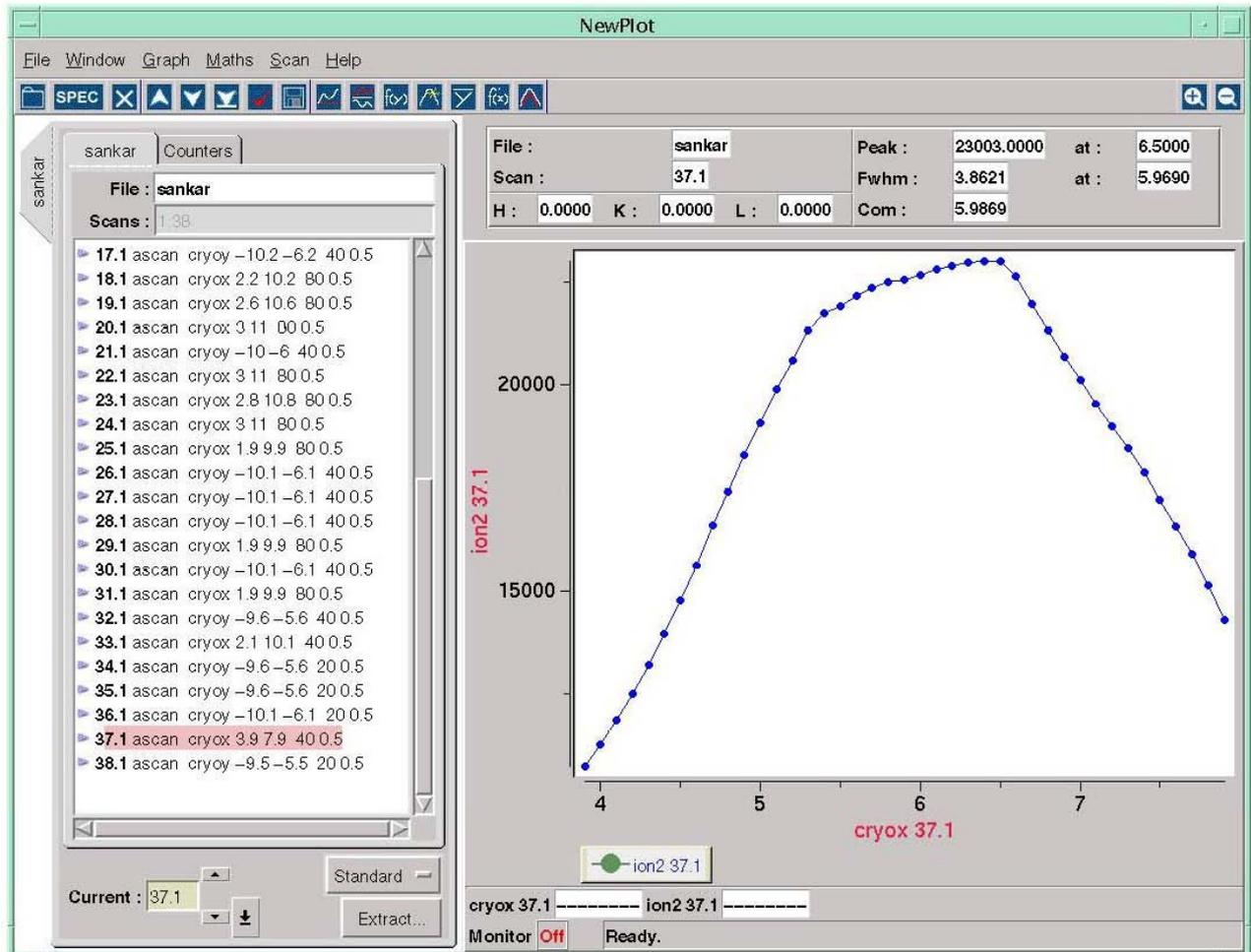
The XRD files named i00000.dat will be saved in the directory XRD in the Users/verylocal/DATA directory on Dubble7 with numbers corresponding to the alternating XRD/XAFS sequence of choice.



5.5 Plotting in Dubble7

For a direct graphical display in *Dubble7* you may use the program *Newplot*. *This* interface developed at the ESRF displays data from SPEC files. *Newplot* allows finding peaks in a scan. Fast, simple fitting routines are also available. To run *Newplot* you first need to create a new

filename by typing **newfile** in any directory in **SOFT>**. The program is then started by typing **newplot** at a prompt in an x-window of the workstation. It may be convenient to create a new directory in the **Users/verylocal/DATA** using the Unix command **mkdir**. It will save all the scans performed at the station in the same file.



6. Data transfer

Raw XAFS data files with file name r00000.dat are initially stored on the station computer *Dubble7* into the directory **users/verylocal/DATA**. In order to analyse them they must be transferred by ftp from the Windows PC *Dubble8* next to *Dubble7*. XRD files are normally stored in the directory users/verylocal/XRD of *Dubble8*. They have the format i00000.dat with the number matching the corresponding XAFS dataset when they are measured using the *exafs* program.

The acquisition program implemented on Dubble works in monochromator angles and not directly in Energy. The original files being only compatible with the *Excalib* program in the Daresbury software suite, must be transformed into xy files (Energy eV – absorbance χ).

6.1 Raw Data format

The raw files r00000.dat are saved in the 'Daresbury format'. Two types of files can be recorded: transmission and fluorescence files.

6.1.1 Transmission file

```
&SRS
SRSRUN=03351,SRSDAT=040130,SRSTIM=055843,
SRSTN='D26 ',SRSPRJ='dubble7 ',SRSEXP='exafs ',
SRSTLE='1CZS '
SRSCN1='He +H2 ',SRSCN2='RT -400C',SRSCN3='xanes ',
&END
12835.10 2000.00 325771.00 434483.00 0.00 0.00
12828.10 2000.00 326536.00 436973.00 0.00 0.00
12821.10 2000.00 327173.00 439753.00 0.00 0.00
12814.10 2000.00 328093.00 442769.00 0.00 0.00
12807.10 2000.00 325864.00 441641.00 0.00 0.00
```

.....

Monochromator angle / Counting time in ms / I0 / It / (Im)

6.1.2 Fluorescence file

```
&SRS
SRSRUN=02971,SRSDAT=031205,SRSTIM=011948,
SRSTN='D26 ',SRSPRJ='dubble7 ',SRSEXP='exafs ',
SRSTLE=' HDPCoMn13 '
SRSCN1='350 ',SRSCN2='He+H2 ',SRSCN3='after 1h',
&END
17750.50 3000.00 1298305.00 0.00 142260.00 0.00
5802.00 21654.00 10068.00 18432.00
28869.00 23949.00 8469.00 25017.00
0.00
17732.90 3000.00 1301152.00 0.00 144159.00 0.00
5889.00 21579.00 10377.00 18858.00
29148.00 24447.00 8469.00 25392.00
0.00
```

....

Monochromator angle / Counting time in ms / I0 / It / If / (Im) / n individual fluorescence detectors (8 or 9)

6.2 Transformation of the data in XY files

► Command

In the data directory:

> **transform file.dat 0 0** (Transmission files) file name r00000.dat.sample

> **transform file.dat 1 0** (Fluorescence files)

The XY datafiles can then be opened in *Viper* or *Origin* in *Dubble8* for a preliminary analysis.

✘ You are strongly advised to perform a full EXAFS analysis on some important datasets of your experiment to check that the quality of your data is sufficient, that no significant glitches are present and more generally that you are actually measuring what you are interested in.

7. Calibrating the monochromator

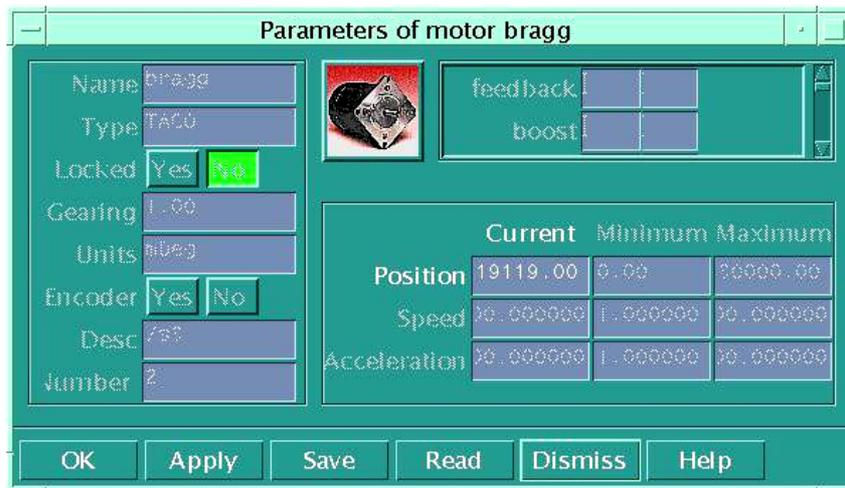
The monochromator has an encoder reading the angular position, and in normal circumstances is already calibrated. However, it is good practice to check whether this is the case, and sometimes you may find that the mono calibration is not correct. This may be caused by e.g. a power failure. The following describes how to calibrate the monochromator.

The real value of the monochromator position is displayed on the McLennan monochromator controller (monitor located on the shelf above the Sun station). The displayed value is generally larger by constant shift of **+1.66 degrees** than the value displayed in the *exafs* program as the real current Bragg angle.

1. Position a foil or a foil substitute (similar edge position) in a sample holder between the two ion chambers and with the beam hitting the central part of the sample. (The beam position is adjusted by using a fluorescent paper whose edge is at a known distance from the sample)
2. Interlock the hutch using the search procedure and turn on the X-rays.
3. In the *exafs* program move the monochromator to a position near the edge with which you are calibrating the monochromator using the *Experiment set-up* window
4. In this window you can use the automatic region selection that produces default regions for calibrating the monochromator and running a k-space scan according to the selected element's edge. The button marked *calibration* produces a single region of about 100 eV centred on the edge position. This will find the edge, providing the calibration of the station is not too far out. (*If the station is grossly miscalibrated, due to some unforeseen reasons just ask staff to fix the problem*)
5. During the scan try to identify where the edge is in mDeg as the scan proceeds. The edge position is often defined as the point of inflection on the absorption edge, but you have to decide where you think it is. By choosing the track pointer option from the *Modes* sub menu you can obtain the position of the cursor on the screen. For a better accuracy you can also transform the raw file into xy file and ftp it to *Dubble8* to open it into Viper and check the maximum of the derivative. (see section 6 *Data transfer*)
6. This energy can transform into a Bragg motor position in mDeg in the *Experimental Setup* window.
7. In the Hardware menu of the main window of the *exafs* program open *Motors* and choose *Bragg motor*.



8. In *Commands* menu open *edit motor* - type new angle value – click *apply* – dismiss this *edit motor* window – dismiss *motors* window – click *apply* in *Experimental Setup* window.



9. Check the calibration by recording a second scan over the edge (perhaps with a shorter range and smaller step size). If the edge is in the right place the station is calibrated, if not repeat the editing procedure after moving the monochromator to where the edge now is.

If you do not see an edge in your initial scan, there may either be a problem with the set-up on the optical bench or the station may be grossly out of calibration (contact staff).

8. Filling the ion chambers** (Experienced Users)

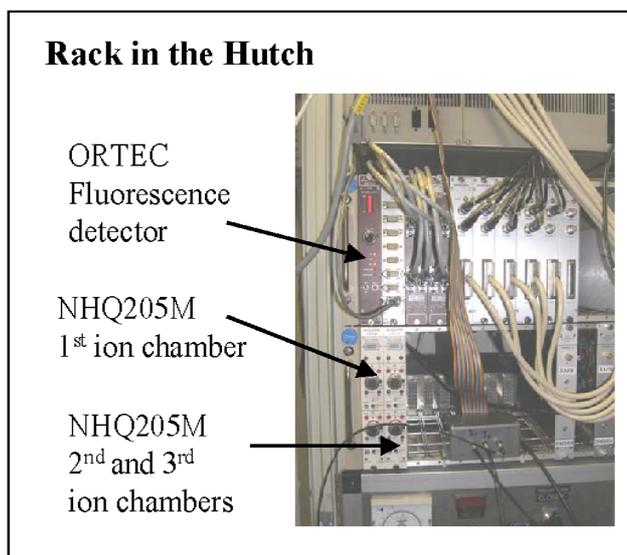
There are two commonly used modes of XAS spectroscopy on BM26A:

- **Transmission:** X-ray beam intensity is measured both before and after the sample using ion chambers;
- **Fluorescence:** X-ray beam intensity is measured before the sample and the intensity of fluorescence signal emitted from the sample is measured using a 9-elements germanium energy dispersive detector.

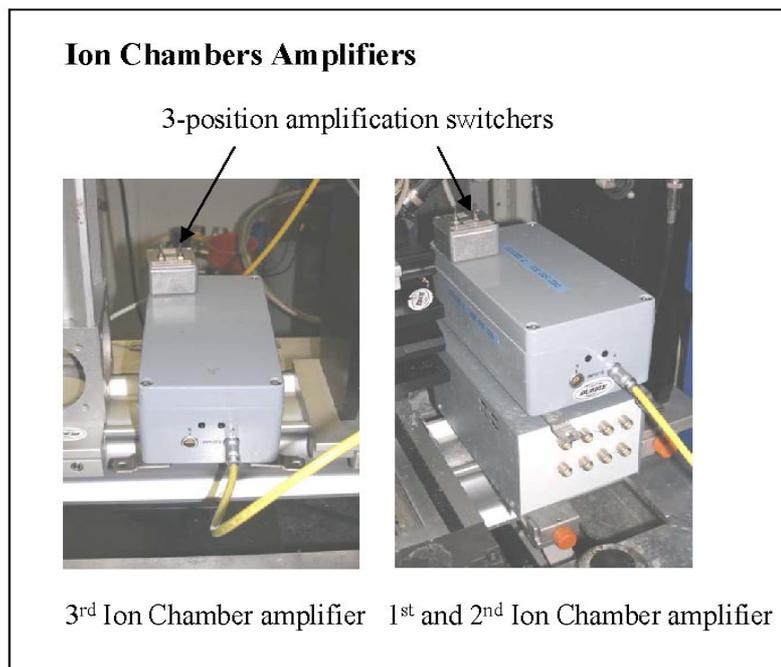
Transmission measurements require the use of two ion chambers I_0 and I_t whereas fluorescence measurements require only the first I_0 chamber. Synchronous “transmission/fluorescence” mode is also available since the *exafs* program reads out continuously the signals from all available detectors (I_0 , I_t , I_m , and I_f).

8.1 Ion Chambers Amplifiers

The ion chambers consist of two metallic plates with a potential difference of 800V between them supplied by a NHQ205N device. The chambers are filled with inert gases, which are ionised by the X-ray photons. The current intensity between the plates is amplified by a two-channel Novelec current amplifier positioned near the ion chambers. As a rule channel “A” is connected with to the output of the first ion chamber (I_0) and channel ”B” to the second ion chamber (I_t) (yellow cables).



A 3-position amplification switcher for each channel is located on top of each amplifier. The gain of the current amplifier can be changed by factor 10 to increase or decrease the output signal. The full signal range on the output of current amplifier is 0 – 10 V, recommended range is 0.1 – 1 V. If necessary the third ion chamber (I_m) can be connected to the second current amplifier. The amplified signal is fed into a voltage-to-frequency converter, whose digital output is sent to a scalar counter read by the station computer *Dubble7*.



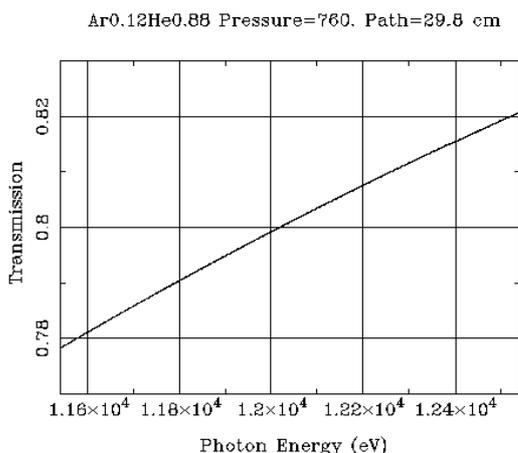
The ion chambers are filled with a mixture of gases that is prepared in the internal vessel of the gas rig located in the main rack in the experimental hutch.

8.2 Filling the Ion Chambers

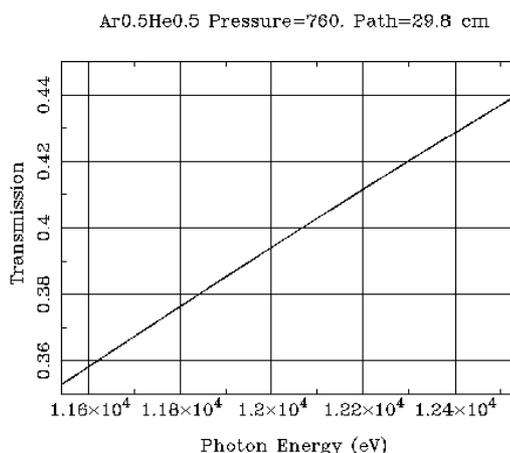
The first ion chamber (I_0) should typically absorb 10-20% (transmission) and the second (I_t) 50-80% of the X-rays according to the edge being measured. When used the third chamber (I_m) should absorb 80% of the X-rays intensity. The optimal gas mixtures for each ion chambers should give absorption curves centred on the required absorption value presenting the same linearity over the whole energy range of the Exafs measurements (typically 1000 eV).

These values are generally obtained by using Ar/He mixtures. Absorption curves can be plotted for instance on the web site http://henke.lbl.gov/optical_constants/gastrn2.html by entering a gas composition i.e. Ar0.3He0.7, using 29.8 cm as path Length (ion chamber length) and 760 Torr as pressure (atmospheric pressure). The curves below correspond to an I_0 absorption of ca. 20% (left) and an I_t absorption of 60% (right). These gas ratio values have to be multiplied by 4 when filling the gas rig internal vessel to obtain a sufficient amount of gas to fill in the ion chamber. These values can also be conveniently obtained by using the program *Hephaestus* from Bruce Ravel.

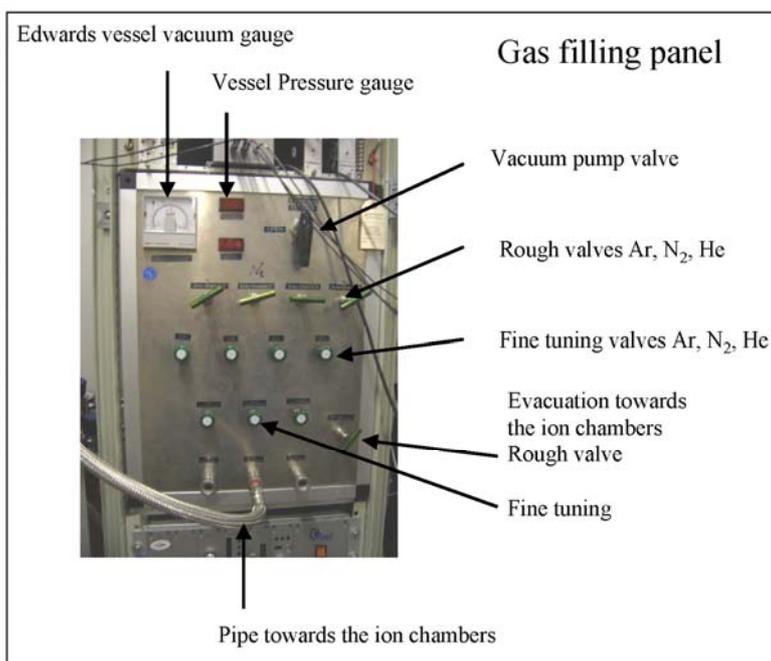
Gas Transmission



Gas Transmission



1. **Switch off** the ion chamber high voltage in the rack inside the hutch (NHQ205M power supply). Channel A on the left-hand side NHQ corresponds to first I_0 chamber, (channel B on this NHQ is not connected), channel A on the right-hand side NHQ corresponds to the second chamber whereas channel B corresponds the third (I_m) chamber.



2. The gas control panel has 4 rough valves (green bars) and 4 fine valves (green buttons) corresponding respectively to Ar, N_2 and He (the third valves starting from the left are not connected). There is another valve on the right-hand bottom part of the panel corresponding to the evacuation of the inner vessel towards the ion chambers. The rough valves must be open before opening the fine-tuning valves.

3. Check that the **all valves** on the gas rig panel are **closed**.

4. **Plug in the main power** supply (*black multiple plug on the floor*) for the vacuum Edwards and the digital pressure gauge (these two pieces of equipment are normally unplugged as they may provoke interferences in the measurements).
5. **Switch on** the vacuum pump on the floor under the rack and open the vacuum pump valve to pump down the intermediate gas vessel.
6. **Connect** the metallic filling pipe from the gas control panel to the ion chamber inlet using the automatic valve.
7. **Switch on** the electronic **Keller gauge** on the ion chamber by pressing the right button.
8. **Open** the ion chamber 2-position **valve**
9. **Open** completely the **vacuum pump** rough tuning **valve** on the gas control panel
10. Very slowly **open** the **fine tuning valve** to evacuate softly the ion chamber
11. Control the pressure at the Keller gauge to avoid too fast pumping. **Too fast pumping can result the breaking of Kapton windows of the ion chamber!** Recommended pumping speed at this stage is about <math><10\text{ mbar per second}</math>.
12. When you heard the click noise from the Kapton windows passing down under atmospheric pressure you can open the fine turning valve completely and wait till a pressure of **10^{-1}mbar** on the Edwards vacuum gauge is reached. It takes about 10 minutes.
13. **Close** the fine-tuning valve and the vacuum pump valve on the gas rig panel.
14. Prepare the required gas mixture in the gas rig intermediate vessel using the rough and fine-tuning valves and controlling the pressure up to a **final pressure of 4 bar** on the red electronic display of the Vessel Pressure Gauge (for example if you need to prepare 5% Ar + 95% He gas mixture you should fill the vessel first with 0.2 bar of Ar and complete it with He up to total pressure of 4 bar).
15. After the gas mixture is ready check that all the rough and fine-tuning gas valves are closed.
16. **Start filling the ion chamber** to a pressure of typically 1 bar. In some rare cases pressures up to a maximum of 2 bars may be admitted inside the ion chambers for some high-energy edges. Open very slowly the fine-tuning valve connected to the ion chamber. Control the pressure in the ion chamber at the Keller gauge to avoid too fast filling near atmospheric pressure. **Too fast filling can result the breaking of Kapton windows of the ion chamber!**
17. When you heard the click noise from Kapton windows passing up over the atmospheric pressure close the 2-position valve at the gas tube of the ion chamber and both the fine and rough-tuning valves on the gas rig panel.
18. Disconnect the metallic filling pipe.
19. **Switch** the 2/3 channel of NHQ 205M high voltage of the ion chamber supply back on.
20. Switch off the pump and unplug the power supplies of the vacuum and pressure gauges.

✘ Be very careful not to damage the kapton windows of the ion chambers when emptying or filling them. When the gas pressure inside the chamber approaches the atmospheric pressure the windows that are



flexible will move towards the outside on filling and towards the inside on emptying the chamber making a small click. Do fill or empty the chambers very slowly until you hear the windows click.

The ion chambers are now ready for use. If you wish to do experiments on more than one absorption edge, this procedure should be repeated when you change edges. If you are collecting data on two elements that are next to each other in the Periodic Table then you can use a fill mixture, which is averaged.

9. Aligning the beam** (Experienced Users)

When selecting a new edge energy, an optimization of the monochromator and mirror parameters has to be performed to realign the beam. The scale of this optimization increases as a function of the extent of the energy change.

The first step is to move the monochromator to a position corresponding to this new energy.

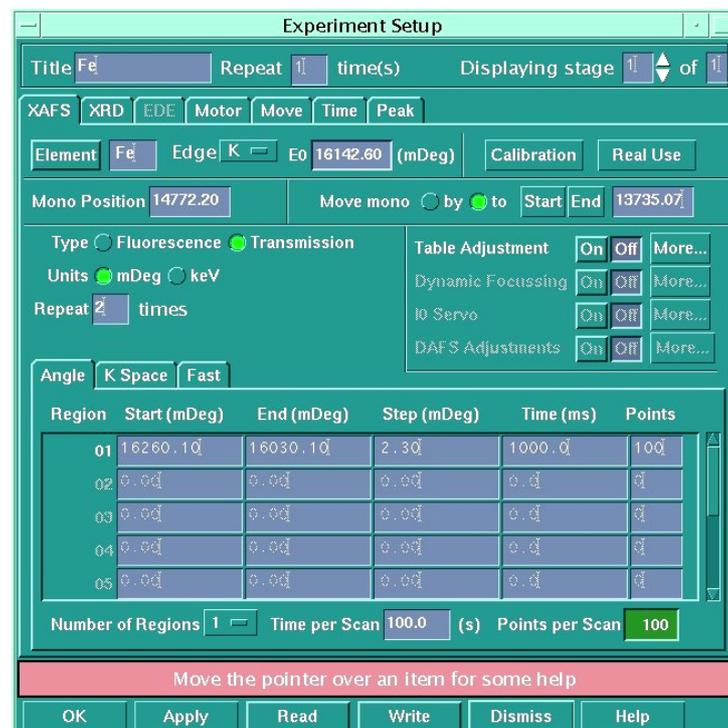
9.1 Moving the monochromator

As is explained in more detail in the scanning section, the monochromator is calibrated in millidegrees, which are related to X-ray energy by the Bragg equation:

$$n \cdot \lambda = 2 \cdot d \cdot \sin \theta$$

(where n is an integer, λ is the wavelength of the light, d is the lattice spacing of the crystal and θ is the angle of the crystal planes to the incident beam.)

1. In the *exafs* program select **Setup** from the main program window. You get the *Experiment set-up* window



2. If you select the element button, a window will come up with the periodic table in it. Choose the element of interest and then use the edge of interest K, LI, LII or LIII. The value of the edge in millidegrees will be listed in the E0 box.
3. The current position in millidegrees of the monochromator can be found by directly reading the value displayed on the Monochromator Optics Hutch monitor located on the shelf above the workstation (with a constant shift of +1.7 eV compared to exafs program reading)
4. You can move the monochromator to a defined position or by a number of millidegrees by selecting the relevant button in the *Experiment set-up* window. (Alternatively by selecting the Start or End button, the monochromator will automatically move to the end of the scan. Then type in a value in the *'Move Mono'* box and hit return and the monochromator should move.
5. Once it has finished moving the updated value for its position will appear in the *Mono Position* box.

✘ Always keep track of the beam during the energy change using a fluorescent paper. If you loose the beam it may be time consuming to find it back.

✘ When the monochromator is being moved by a large energy, it is recommended that you move it in steps of 2000 mDeg at a time.

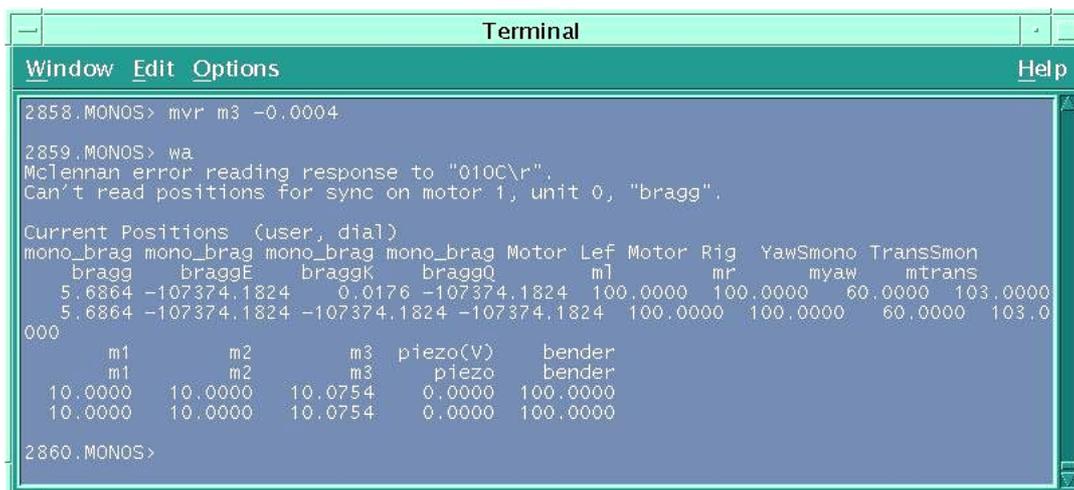
If the monochromator is moved by a large energy you will have to adjust several parameters related to the crystal monochromator and the mirrors.

9.2 Setting the Monochromator crystals

The monochromator settings are changed from the **MONOS>** window that is access by typing **monos** in **SPEC>**

► Command:

MONOS> wa will give you the current positions of all the monochromator motors with the display:



```

Terminal
Window Edit Options Help
2858.MONOS> mvr m3 -0.0004
2859.MONOS> wa
McLennan error reading response to "010C\r".
Can't read positions for sync on motor 1, unit 0, "bragg".

Current Positions (user, dial)
mono_brag mono_bragE mono_bragK mono_bragQ Motor Lef Motor Rig YawSmono TransSmon
bragg braggE braggK braggQ m1 mr myaw mtrans
5.6864 -107374.1824 0.0176 -107374.1824 100.0000 100.0000 60.0000 103.0000
5.6864 -107374.1824 -107374.1824 -107374.1824 100.0000 100.0000 60.0000 103,0
000
m1 m2 m3 piezo(V) bender
m1 m2 m3 piezo bender
10.0000 10.0000 10.0754 0.0000 100.0000
10.0000 10.0000 10.0754 0.0000 100.0000
2860.MONOS>
    
```

9.2.1 Pitch of the monochromator **m3**

The first parameter that needs some readjustment is the pitch that is the distance between the 2 mirrors usually controlled by the m3 motor (see figure below). The distance between the two crystals is usually set to 10.0 mm. Only small variations of m3 are necessary.

► Command:

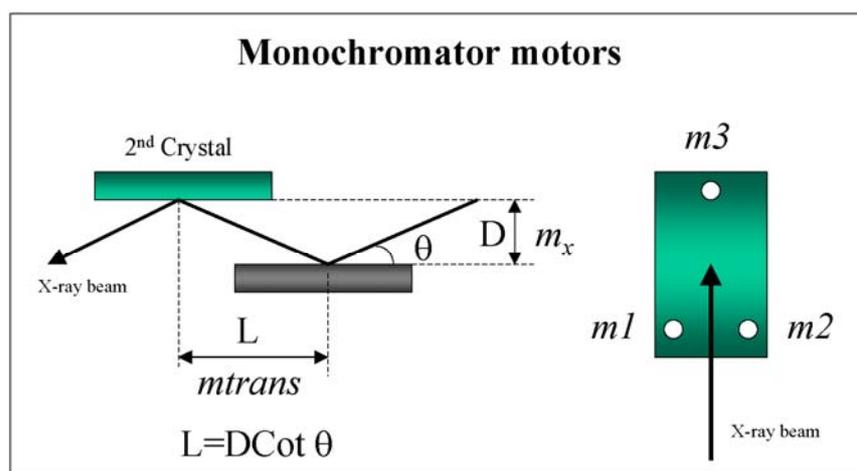
MONOS> mvr m3 0.001 (in mm) to adjust pitch of monochromator to find a maximal signal at the first ion chambers I_0 ; the typical step is 0.001; the steps should be smaller at high energy.

9.2.2 Linear positioning of the second crystal **mtrans**

The second parameter that needs to be changed is the mtrans the linear stage positioning of second crystal, which represents the relative translation of the second crystal with respect to the first one. This will ensure that the beam stays always in the centre of the second crystal during the energy scan. **mtrans** or L should be calculated from the expression $L=D \cdot \cot\theta$ with **D** representing the distance between the 2 crystals and θ the monochromator angle

► Command:

MONOS> mv mtrans 60 (in mm) will adjust linear stage positioning of second crystal to find a maximal signal at the first ion chambers I_0 ; this is a rare case when you should use the absolute movement. **mtrans** ranges from 30 at lowest energies to 130 at highest energies ($D = 10$ mm).



For non-standard experiment there are additional parameters that may also need to be adjusted like second crystal's bendersim (100), myaw (60), roll etc... but these stings should be only done by the beamline staff.

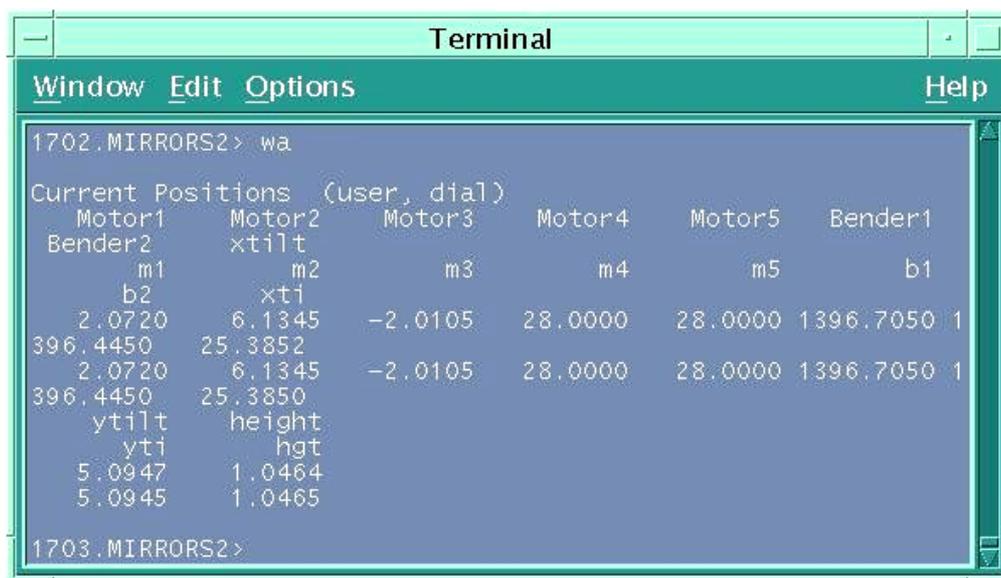
9.4 Setting the Mirrors

The mirrors are used to focus the X-ray beam and perform the harmonic rejection. They must be readjusted when the edge energy is changed.

The mirrors are moved from the **MIRRORS2>** window that is access by typing **mirrors2** in **SPEC>**

► Command:

MIRRORS2> wa will give you the current positions of all the mirrors motors with the display:



```

1702.MIRRORS2> wa
Current Positions (user, dial)
Motor1      Motor2      Motor3      Motor4      Motor5      Bender1
Bender2     xtilt
  m1         m2          m3          m4          m5          b1
  b2         xti
  2.0720     6.1345     -2.0105     28.0000     28.0000    1396.7050  1
396.4450    25.3852
  2.0720     6.1345     -2.0105     28.0000     28.0000    1396.7050  1
396.4450    25.3850
  ytilt     height
  yti       hgt
  5.0947    1.0464
  5.0945    1.0465

1703.MIRRORS2>
  
```

9.4.1 Height of the mirror **hgt**

Generally only changes of height are necessary: typical with steps of 0.1 mm. Only 4 steps in each direction are allowed.

► Command:

MIRRORS2> mvr hgt 0.01 (in mm) to adjust the height of the mirror relative to the beam position to find a maximal signal at the first ion chambers I_0 ; The typical step is 0.01;

9.4.2 Strip of the mirror **m4 m5**

A second parameter that may need optimisation is the nature of the mirror strip. The mirror consists in 2 strips: a pure Si strip and a Pt coated strip. With Energy >15 keV Pt strip should be used, for energies < 15 keV Si will give better results.

► Command:

MIRRORS2> tw2 m4 m5 (-)5 (-)5 (in mm)

Si strip corresponds to $m4 = m5 = 32$ mm whereas Pt strip ($E > 15$ keV) corresponds to $m4 = m5 = 20$ mm. To quit tw type q.

Additionally the horizontal and vertical tilt of the mirror controlled respectively by the virtual motors xtilt and ytilt corresponding to combinations of m1, m2 and m3 motors may also be altered.

9.4.3 Bending of the mirror **b1 b2**

In special occasions the vertical focusing of the beam may be altered by using the command:

► Command:

MIRRORS2> tw2 b1 b2 0.01 0.01 (in mm)

9.3 Setting the slits

It is important that the size of the beam going through the ion chamber is such that it covers most of your sample but is not larger than it. Hence the size of the beam may need to be modified with the use principally of the entrance Slits.

The resolution of the experiment, $\delta E/E$, is related to the vertical beam height, h , by:

$$\delta E/E = (h/L) \cot\theta$$

where θ is the Bragg angle and L is the distance of the monochromator from the tangent point (ca. 30 m for BM26A Dubble). The current energy resolution of Dubble Si 111 monochromator is :

$$\Delta E/E = 1.74 \cdot 10^{-4} \text{ or } \Delta E = 1.7 \text{ eV at } E = 9.659 \text{ eV.}$$

The size of the beam is generally modified by looking at the beam image on a fluorescent paper.

The slits are moved from the **SOFT>** window that is accessed by typing **soft** in **SPEC>**

► Command:

SOFT> slitshow will give you the current position of the current gaps and offsets of all slit sets with the display:



```

2722.SOFT> slitshow
Currently configured slitsets:
slit_params slitsets1
'slitsets1' Horiz. offset   Horiz. gap  Vert. offset  Vert. gap
(enabled)   -8.0381    28.3012    0.0238       0.7000

slit_params slitsets2
'slitsets2' Horiz. offset   Horiz. gap  Vert. offset  Vert. gap
(enabled)   -13.0181   63.2037    0.3831       16.3863

slit_params slitsets3
'slitsets3' Horiz. offset   Horiz. gap  Vert. offset  Vert. gap
(enabled)   -9.8000    58.1000   -4.1625       21.6750

slit_params slitsets4
'slitsets4' Horiz. offset   Horiz. gap  Vert. offset  Vert. gap
(enabled)    2.5125     4.0000   -15.0000      23.9750

2723.SOFT>

```

9.3.1 Vertical slits

The vertical beam size is the most important parameter since it is related directly to the energy resolution of your measurements. It is normally defined by the vertical gap *s1verg* and gap offset *s1vero* of the slits before the monochromator. The beam is usually focused vertically up to 300 μm . If necessary the vertical size can be increased by unbending the mirror.

After each change of edge energy the slits must be optimised. By adjusting slitsets1 you should aim at maximising the intensity of the beam that is measured by the intensity in the first ionisation chamber (I_0).

The recommended optimum values for Slitsets1 are:

Energies 5 to 9 keV vertical gap : **0.8 mm**

Energies 9 to 15 keV vertical gap : **0.5 mm**

► Command:

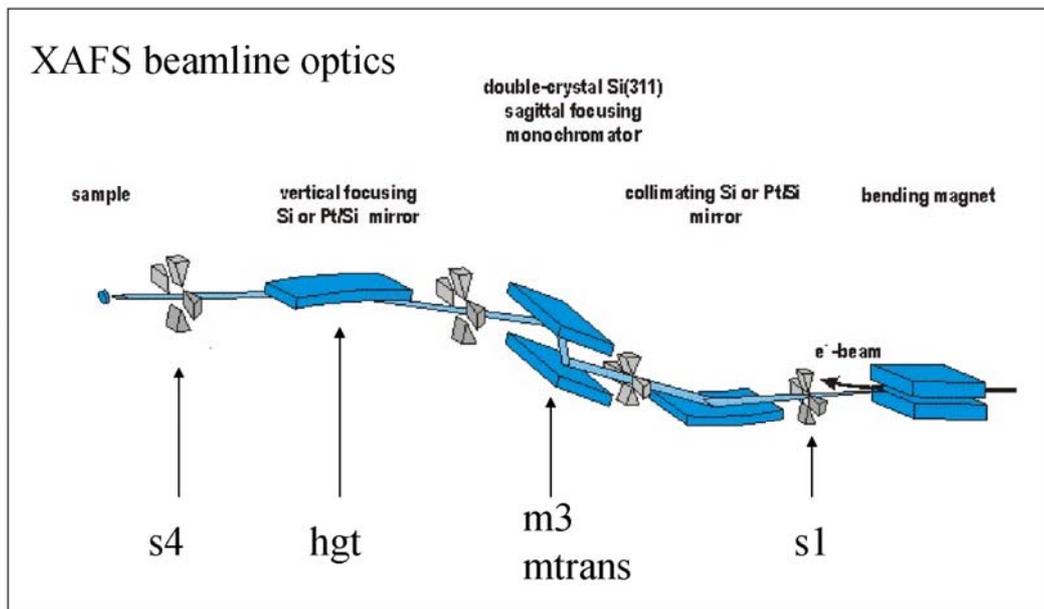
SOFT> mvr s1verg 0.1 (in mm) will change the gap of the vertical slits

s1vero and *s1verg* that control the vertical slit1 located before the monochromator are the most important ones to optimise since they control the energy resolution of your measurements.

9.3.2 Horizontal slits

The horizontal size of the beam is generally controlled by the slit gaps *s4horg*. The slits are centred on a zero position that can be moved using the respective offsets *s4horo*. Alternatively in a limited number of experiments s1 slit may also be modified. However, once the experiment has started don't modify s1horg since it may alter the beamline optics setting by modifying the thermal loads on the monochromator brought by the incoming flux.

The recommended optimum values for Slitsets1 Horizontal gap is as user wishes: **3 to 10 mm**



10. Basic Unix commands

• pwd

Use this command to find out what directory you are working in.

```
% pwd
/u/ma/jeremy
% cd homework
```

• cd

Use **cd** to change directory. Use [pwd](#) to see what directory you are in. **cd ..** to jump back one level.

```
% cd english
% pwd
/u/ma/jeremy/english
% ls
    novel poems
```

• cp

Use **cp** to copy files or directories.

```
% cp foo foo.2
```

This makes a copy of the file foo.

```
% cp ~/poems/jabber
```

This copies the file jabber in the directory poems to the current directory. The symbol "." stands for the current directory. The symbol "~" stands for the home directory.

• ls

Use **ls** to see what files you have. Your files are kept in something called a directory.

```
% ls
foo    letter2
    foobar    letter3
    letter1    maple-assignment1
%
```

Note that you have six files. There are some useful variants of the **ls** command:

```
% ls l*
letter1 letter2 letter3
```

• mv

Use this command to change the name of file and directories.

```
% mv foo foobar
```

The file that was named foo is now named foobar

• mkdir

Use this command to create a directory.

```
% mkdir essays
```

To get "into" this directory, do

```
% cd essays
```

To see what files are in essays, do this:

```
% ls
```

• rmdir

Use this command to remove a directory. For example, to remove a directory called "essays", do this:

```
% rmdir essays
```

A directory must be empty before it can be removed. To empty a directory, use rm.

11. Short summary

11.1. Turning on the X-ray beam

- Double click on the valve 'safety shutter soft line' in the schematic diagram. Press **open** in sub-window.

11.2. Starting data acquisition program

- type **exafs_interface** in an local terminal window to load a 'settings file' and then type **exafs**.

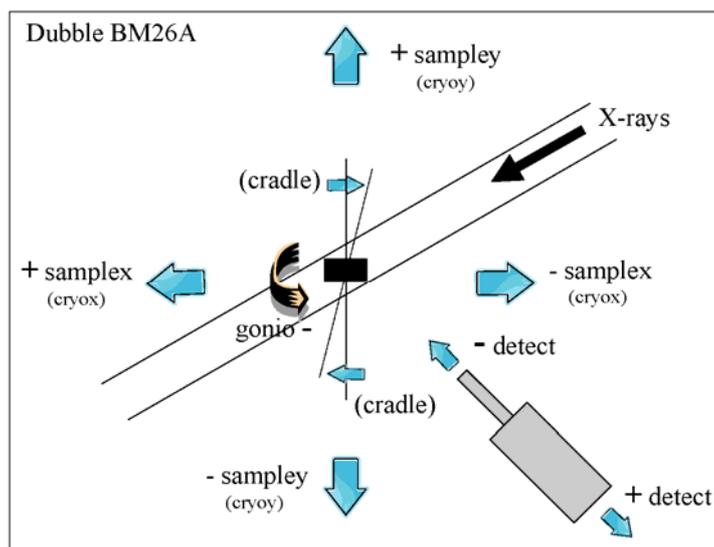
11.3. Moving the Sample stage

- Use the command **SOFT> mvr** (relative movement)

change the current motor positions : **mvr [motor] [relative value mm]**

Example : **mvr sampley 0.1**

- ◆ **sampley** = vertical movement (becomes **cryoy** when the cryostat is installed)
- ◆ **samplex** = horizontal movement (becomes **cryox** when the cryostat is installed)
- ◆ **gonio** = rotational movement around y-axis (+ clockwise - anti-clockwise)
- ◆ **detect** = fluorescence detector movement towards and backwards relative to the sample



11.4. Quick edge step check

- Press **calibration** in the **Experiment set-up** window automatically to produce a single region of about 100 eV centred on the edge position.
- Open the **Monitor** window and set a counting time of 500 ms.
- Check the absorption over the edge by comparing χ before and after the edge by moving the monochromator before (**Start**) and after the edge (**End**). In transmission $\chi = \ln(I_0/I_t)$ [around 1] , in Fluorescence $\chi = I_f/I_0$ [0.05-0.5]. Ion chambers gains should be adjusted to obtain I_0 and I_t values close to 1.

11.5. Starting a Scan

- If you already have a configuration file saved in the `users\verylocal\DATA` directory Press **Read** and then **Apply** (You may have to go up and down twice in the directory tree before it works)
- If not, you need to set up the regions for the scan:
 - ◆ Edge position in mDeg - *this can be altered from the defined value if you desire*
 - ◆ Start of k-space region - *normally a value between 2 and 4*
 - ◆ End of k-space region - *normally between 10 and 20*
 - ◆ The k-weighting - *the time for which data is collected at each point is weighted with relation to the k value of that point, thus at higher k where the EXAFS oscillation is weaker more data is collected: normally 2 or 3*
 - ◆ The time for which data is collected at the last point - *between 5000 and 30000 mSec*
 - ◆ The time for which data is collected at the first point - *between 1000 and 5000 mSec.*
 - ◆ The k-step - *the interval in k-space between each point: normally between 0.02 and 0.06*
- Press **Apply** and the green **Go** button will now be enabled on the main program window.
- Press the Green **Go** button, a **Titles** window comes up.
- Press **OK** and then **Continue** in the main titles window. The scan will start.

11.6. Quick optimization of the beam

- Pitch of the monochromator

MONOS> **mvr m3** 0.001 (in mm) to adjust pitch of monochromator to find a maximal signal at the first ion chambers I₀; the typical step is 0.001 (type **wa** to check the absolute position)

- Vertical gap of the s1 slit

SOFT> **mvr s1verg** 0.1 (in mm) will change the gap of the vertical slit. Large gap will increase the intensity of the beam and decrease the energy resolution.(type **slitshow** to check the absolute position)

- Height of the mirror

MIRRORS2> **mvr hgt** 0.01 (in mm) will adjust the height of the mirror relative to the beam position to find a maximal signal at the first ion chambers I₀; The typical step is 0.01; (type **wa** to check the absolute position)

11.7. Transformation of the data in XY files

- In the data directory type:

> **transform file.dat 0 0** (Transmission files) file name r00000.dat.sample
> **transform file.dat 1 0** (Fluorescence files)

12. Trouble shooting

12.1. Acquisition Software

● Crash of the exafs program

you may need to reset the VME. Before doing this quit the *exafs* program by clicking the *Exit* button on the main EXAFS window and re-enter it after the VME-crate has been reset. To reset the VME you need to enter the experimental hutch, locate the crate in the electronic rack (above the gas filling rig) and press the *reset* button 2 seconds (very small red button) on the MVME 162-522A unit at the LHS of the crate just below the VME system hard drive. After about 2 minutes you can start the *exafs* program on *Dubble7* again by typing *exafs* in a new window. (*see section 3*)

● Loss of the monochromator calibration

If in QEXAFS you stop the scan before it ends or in a multiple scan run a scan fails to start, then abort the scan with the red *Stop* button. When the Bragg motor stops moving it is safe to exit the program and then restart it. In the Hardware menu of the main window of the *exafs* program open *Motors* and choose *Bragg motor*. In *Commands* menu open *edit motor* - type the angle value displayed by the MacLennan controller on the Monitor **minus 1.66 Deg** – click *apply* – dismiss this *edit motor* window – dismiss *motors* window and click *apply* in the *Experimental Setup* window (with a Si 111 crystal 5 keV = ca. 25 Deg and 30 keV = ca. 3.5 Deg). You may need to recalibrate the monochromator with a metal foil corresponding to your edge.

12.2. X-ray beam

● No beam

No I₀ reading

- No beam*: check the storage ring status by clicking on the *Four* button in the bottom window of Dubble7
- The Front End shutter is closed*: open the FE by clicking on the button in the storage ring status window
- The S beamline shutter is not open*: open the shutter by clicking on the open button in the shutter window (see section 1)
- A slit is totally closed*: check the values of s1verg and s4horo by typing slitshow in a SOFT> window
- Not in Monitor mode*: the program is not in Monitor mode Press the Monitor button on the window

I₀ reading but No It reading

- The sample holder in the way of the beam*: move the sample stage position
- Not in Monitor mode*: the program is not in Monitor mode Press the Monitor button on the window