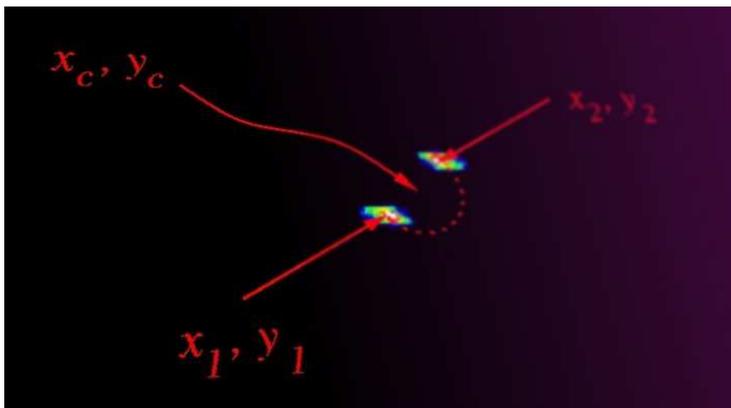


The “How To” Guide at the Surface Diffraction Station X04SA Materials Science beamline, Swiss Light Source

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Version 1.02

April 2007

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Chapter 1

Introduction

This document describes how one obtains x-radiation from the monochromator; diffractometer alignment and crystal orientation procedures commonly used at the Surface Diffraction station of the Materials Science beamline; plus a detailed listing of how to set up the Pilatus II pixel detector. The alignment procedures include positioning of the entire diffractometer relative to the direct x-ray beam; zeroing the detector movements; alignment of the sample surface; and procedures for efficiently and accurately obtaining the angular coordinates for Bragg reflections used to calculate the orientation matrix.

The procedures described in this document are “personal” – to an extent, they are simply “the way we do it”. Because the diffractometer is equipped with a novel area pixel detector, some procedures (particularly the alignment of the detector and sample motors and obtaining reflections for the orientation matrix) are novel to the seasoned crystallographer and may therefore seem strange. We believe, however, that they are far simpler and probably more reliable than they have been in the past using traditional point detectors.

That is not to say, however, that we cannot learn from experience. The author of this document (reachable under philip.willmott@psi.ch) would very much appreciate feedback regarding improvements, alternatives, and errors concerning all aspects of the contents.

Chapter 2

Getting beam

This chapter deals with finding and optimizing beam intensity from the double-crystal monochromator and other optics-hutch components. What follows is a step-by-step description of how to achieve this.

2.1 Preparatory operations

1. Check that the beamline scientist has closed the wiggler and opened the front end.
2. Set the front-end slits in the horizontal direction to the desired opening. At 32 mm, the full opening angle of the wiggler, equal to 2.5 mrad, is exposed to the beamline. Hence, closing the horizontal FE slits to 10 mm will produce a horizontal divergence of approximately 0.8 mrad, (a little less than 0.05°). Most of the power is in the center of the cone, hence closing down to 10 mm or thereabouts will only marginally affect the flux reaching the endstation.
3. Open all subsequent slits downstream, i.e., the EH1-, EH2-, and JJ-slits using the relevant EPICS widgets.
4. Using SPEC, raise the delta-arm of the surface diffractometer (spec command `mv del 35`) so that the x-ray eye (XRE) mounted on the far wall of EH2 can see the direct beam.
5. Ensure that the high-voltage supply for the ionization chamber (model NHQ 103M) is set to +1000 V and the current monitor/selector (SLS unit) is set to 10^{-8} mA (both are located on the main rack WSLA.37.0.66 in the EH2 experimental hutch, see Fig. 2.1).
6. Ensure that N_2 flows through the ionization chamber and that the flight tube is evacuated.
7. The “TORII” is an active element of the first crystal of the DCM that compensates for the thermal bump produced by absorption of the large majority of the incoming high-power white x-ray beam. By lowering this thermomechanical distortion, the transmitted flux to the second crystal is maximized. It is therefore very important to ensure that the TORII controller is turned on (it is located in the rack WSLA.37.0.52, situated behind the beamline). If the TORII controller is turned on, but is showing an error message or zero voltage, turn it off at the power switch at the back of the unit.

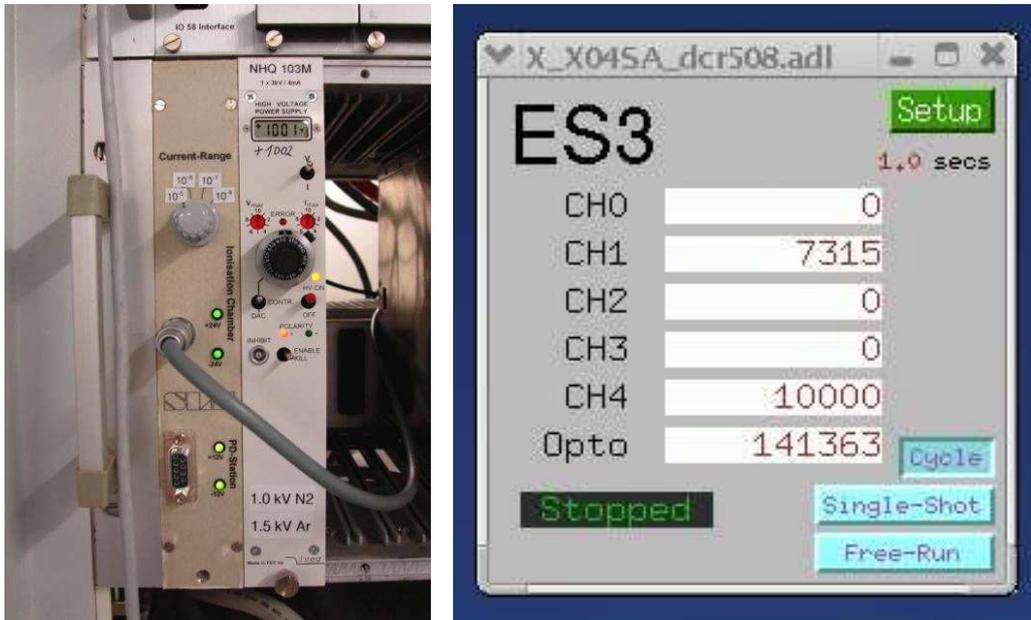


Figure 2.1: Left: the current selector/control and high-voltage supply for the ionization chamber. Right: the scaler EPICS widget. The `Opto` channel records the output from the ionization chamber. The scaler will continuously cycle if the `Cycle` button is clicked. The duration of each cycle can be set by clicking on the green `Setup` button. In this image, it has been set to 1 s.

8. Ensure that the fast photon shutter, the first component after the EH2 slits (see Fig. 2.2), has power and is open (this is opened via SPEC with the command `shop` and closed with the command `shc1`, or can be opened and closed by clicking on the F-PS button in the EPICS filter widget).

2.2 Optimizing the DCM motor positions and TORII settings

1. In the optics panel of the control computer pc5888, enter the desired photon energy (in keV) in the EPICS set-energy widget (see Fig. 2.3). This widget obtains the nominal motor and TORII settings for the desired energy from a look-up table.

Horizontal focussing of the beam is controlled by the sagittal focussing mechanism of the second crystal in the DCM, and vertical focussing is achieved by bending the second mirror. Both horizontal and vertical focussing can be independently set to `flat` (i.e., relaxed second crystal and second mirror), `parallel` (incident beam to the hutch has minimal divergence), `PD` (focussed at the powder station), or `ISSD` (focussed at the surface diffractometer).

Once the energy and focussing have been selected, press the upper red ! button, which causes a panel to appear, in which one should type `y` twice, in order that the optics motors move to the nominally set values. This typically takes up to 1 minute to complete.

2. The beam intensity is monitored using the ionization chamber via the `Opto` channel of the EPICS scaler

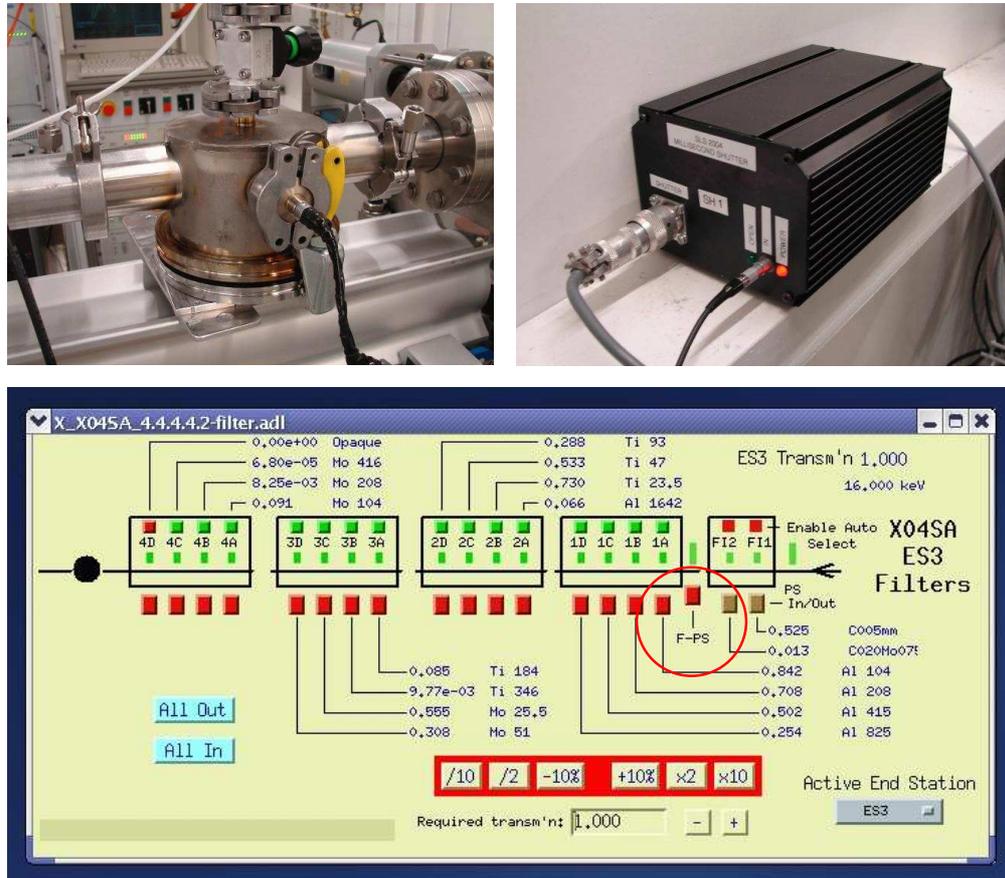


Figure 2.2: Top left: the fast photon shutter inside its vacuum housing. The shutter operates using an electromagnet and opens and closes within approximately 5 ms. Top right: the power supply to the fast photon shutter. Bottom: The toggle button for opening and closing the fast photon shutter on the EPICS filter widget has been highlighted with a red circle.

widget (see Fig. 2.1). Click on **Cycle** to continuously monitor the **Opto** signal. The cycle period can be changed by clicking on the green **Setup** button (see Fig. 2.1).

3. Now some fine-tuning is required to maximize the beam intensity. In most cases, the nominal motor positions are sufficiently close to their optimal values that the x-ray beam can be seen with the x-ray eye. If this is not the case, either the diffraction condition is poorly met by the second crystal in the DCM, or the beam is skewed so much that it does not hit the sensitive area of the XRE (ca. $6 \times 4 \text{ mm}^2$). The first problem is resolved by joggling the angle of the second crystal (ROX21, see Fig. 2.4), while the beam is shifted sideways using the ROX23 roll widget and up and down using the TR2C second-mirror tilt widget (see Fig. 2.4).
4. Once the beam has been located on the XRE screen, the signal will soon saturate the **Opto** scaler if the I0 current is left on the most sensitive scale of 10^{-8} mA. Change this to the least sensitive scale (10^{-5} mA, see Fig. 2.1) and continue tweaking ROX21 until no further improvement is found. This might also require

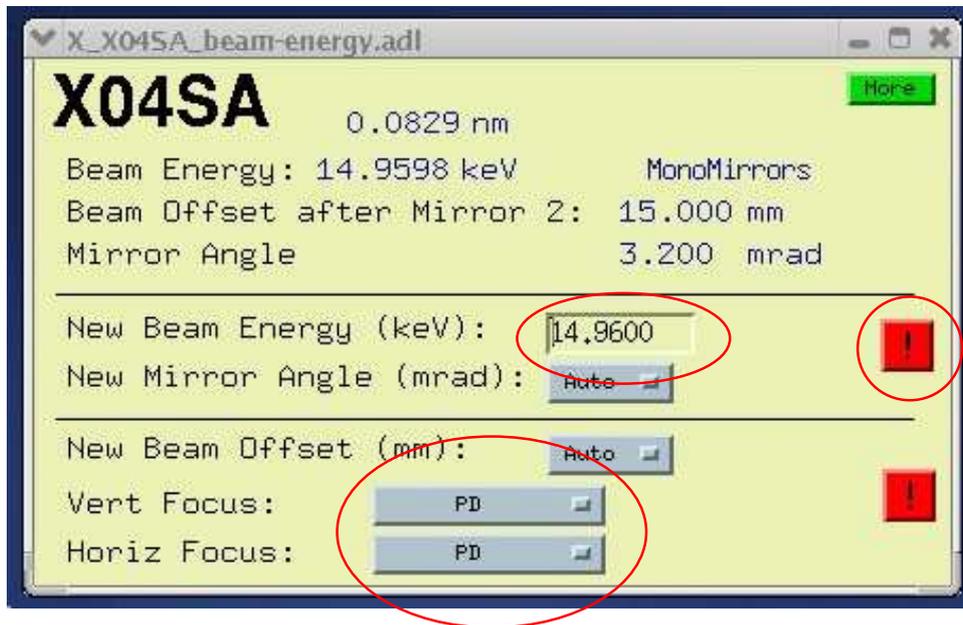


Figure 2.3: The EPICS set-energy widget. The desired energy is entered into the highlighted field, while horizontal and vertical focussing can be independently set using the pulldown menus also highlighted. Once the energy and focussing has been set, the motors will move to the nominal positions for this if the user clicks on the highlighted red ! button.

moving the beam a little using R0X23 and TR2C until the beam is approximately centered on the black dot of the XRE screen. **N.B. NEVER move the XRE from its present position on the bracket on the far wall of EH2!!! This has been calibrated to better than 0.1 mm!**

5. Now you want to optimize the TORII settings. The voltages can be changed using the TORII widget, visible in Fig. 2.4. They are automatically set to values close to the optimal by the EPICS set-energy widget, so normally adjustment is less than 0.1 V from these values. The values should be kept equal for both fields, and should never exceed 1.0 V.

A convenient tool for the TORII optimization is the python script `rock.py`. Open a shell and type in `rock.py --es3 rox21`. This will start a process in which R0X21 scans over a range of angles, the Opto counts are plotted, and the final curve is fit to a Gaussian and the width and amplitude of this are given. In this manner, the user can cyclically adjust the TORII voltages and run the python script, thereby obtain the optimal settings, for which the Gaussian is narrowest and most intense. Note that, after changing the TORII voltages, one should wait for ca. 20 s before running `rock.py`, in order to let the TORII mechanism thermalize.

In addition to optimizing R0X21, the vertical translation of the second crystal TRX22 should also be optimized. It might prove necessary to iteratively optimize both these motors two or three times.

6. Lastly, once the optimal settings have been found, the exact focussing conditions can be tailored for the specific needs of the experiment, using the motors highlighted in Fig. 2.4. For the horizontal (sagittal)

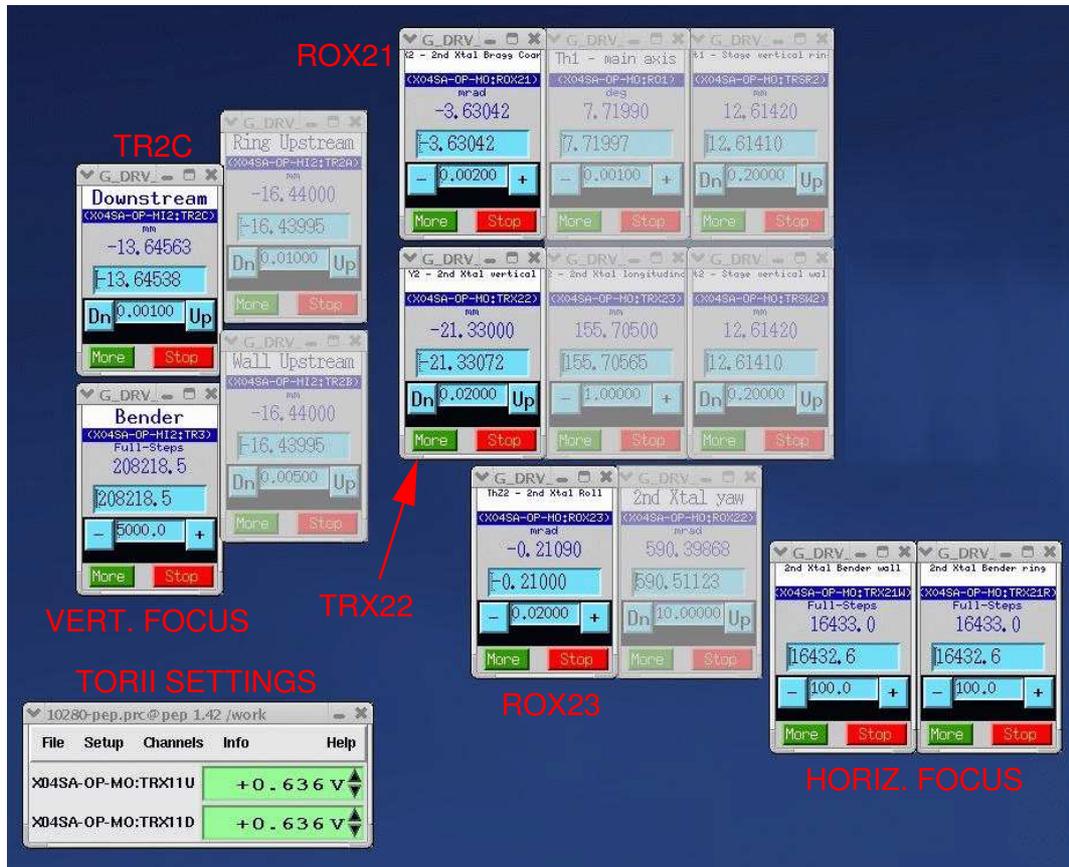


Figure 2.4: The important motors, etc., for optimizing the beam intensity. The motor widgets which have been faded out should in general not be changed.

focussing, make sure that both motors assume the same values, and that during moving, the difference in the settings is kept to less than approximately 1000 steps.

Chapter 3

Setting up the pixel detector and computer communications

This chapter deals with setting up the pixel detector computer with the control computer and the file server. What follows is a step-by-step description of how to set up.

3.1 General comments

There are three computers of import: the experimental control computer pc5758 (with a dual screen, located on the main work desk); the pixel control computer pc5888 (located in the experimental hutch EH2); and the pixel monitor computer, pc6025 (located to the left of pc5758).

One can operate the pixel detector in two modes – either locally and online using the program `tvx` on pc6025, or remotely (via SPEC) from pc5758. In order to make SPEC master, type `disconnect` in the `tvx` shell and click on `connect` on the trigger pulldown menu of the EPICS pixel control widget. When passing control from SPEC back to `tvx`, simply click on `disconnect` on the trigger pulldown menu of the EPICS pixel control widget and type `connect` in the `tvx` shell.

3.2 Setting up the file server and pixel computer

1. SPEC automatically creates a directory tree in the MS beamline file server for the experiment when the user invokes the customized SPEC command `sdStartup`. A typical experiment name that is used to label the root of the directory tree might be `LaA103_20061201`, and we will use this as an example from hereon. On the file server (Data1), we have to change the read/write permissions of the project directory created in `sdStartup`. In a shell on the control computer pc5758, type:

```
chmod -R a+w ~/sddir/expdata/LaA103_20061201/
```

The communication between the control computer and file server has been established. Now you must set up the pixel detector and computer.

2. Turn on the power supply to PILATUS II in the experimental hutch (flick of a single switch!).
3. Log on to the pixel monitor computer (pc6025) in the SD control hutch as `x04sa` (password MS).
4. On pc6025, remote login to the pixel computer pc5888 (which sits in the experimental hutch) by typing in a shell:

```
ssh det@pc5888 (Password: Pilatus2)
```

5. In the shell in which you have `ssh`'d to pc5888, type:

```
cd p2_1mod
```

This brings you into the directory of the pixel computer pc5888 where the relevant information and software for the single-module Pilatus II detector reside.

6. Type:

```
runtvx
```

This produces two shells, the so-called `camserver` shell and the `tvx` command-line shell. This takes a few seconds to load, and also includes the automatic calling up of a so-called “glossary” file, with a set of commands in it for setting up. The file is called `bl_4s.gl`.

7. In the `camserver` shell, type:

```
imgpath /sls/ES3/pixel/images
```

which sets the path to which images are initially sent on the file server (Data1). The final path is defined by the setup of `sdStartup` and the pixel control widget, which can be called up from the `X_X04SA LAUNCHER` (which itself appears if the SLS logo on the control panel is clicked). In more recent versions of `tvx`, this `imgpath` command is automatically called by the setting-up glossary `bl_4s.gl` used when starting up `tvx`.

8. You can set the energy below which x-ray signal is suppressed, which is essential in most cases to cut out fluorescence signal from the irradiated sample. This is called “trimming”. Think carefully which energy to choose. In the `camserver` command shell, type:

```
setth xxxg energyeV
```

whereby “xxxg” can be `lowg` (`vrf = -0.3 V`), `midg` (`vrf = -0.2 V`), or `highg` (`vrf = -0.15 V`), and `energyeV` is the threshold energy given in eV. An example might be `setth lowg 16000`. The reference voltage for the individual pixels, `vrf`, is normally set to either `-0.2 V` for lower trim energy settings (below ca. 15 keV) and longer dead times ($\tau = 194$ ns); or, more commonly (for us), `-0.3 V` for higher trim energy settings (above ca. 10 keV) and shorter dead times ($\tau = 112$ ns). The dead time setting corrects images for pile-up of pulses, which begins to become non-negligible after a count-rate of approximately 10^5 cts s⁻¹.

The program then selects the trimfile (from a set of trimfiles loaded into the pixel control computer) closest to the specified energy, and interpolates from this accordingly. You are now set up for trimmed data.

3.3 Looking at images

To look at images on the fly on pc6025, disconnect, if necessary, the control of the pixel detector from the control computer pc5758 by the clicking on the trigger field in the pixel control widget (X-X04SA-CCD.adl) and selecting `disconnect`. Then type in tvx:

```
connect,
```

and then type:

```
exposemb 1.1
```

To stop this, press, `ESC` or `k`

Before connecting to the control computer to perform scans using the pixel detector, you have to ensure that it is master. If the camserver was controlled by tvx until this point, type in tvx: `disconnect`. Then change from `disconnected` to `connected` for the trigger in the pixel widget on the control computer.

3.4 Resolving computer crashes

It can happen that after a crash of tvx, the control computer pc5758 does not communicate with the pixel computer. This is evident by the pixSNL dot at the top right hand corner of the pixel control widget (X-X04SA-CCD.adl) not continually hopping left and right (i.e., it is frozen). If this happens, you must reboot the ES3 VME crate, which is performed by typing in a shell on pc5758:

```
rmc X04SA-VME-ES3
```

This brings up a “remote minicom” shell (hence, “rmc”), in which you then type `CTRL-x`, which will reboot the VME crate. This takes a couple of minutes to complete. To exit the rmc, type `CTRL-a` then `q`, and then confirm you want to exit without resetting.

A second crash that sometimes arises is that as soon as SPEC tries to record an image, the pixel control widget shows a red panel with the message “NOT MASTER” on it, despite the fact that the camserver has been `disconnected` from tvx and `connected` to the EPICS interface of the control computer. If this happens, you have to restart tvx in full, using the procedure described above.

Chapter 4

Diffractometer alignment procedures

This chapter describes how to align the entire diffractometer so that the center of the diffractometer lies in the x-ray beam and the ALPHA, and GAMMA axes are vertical and the DELTA axis is horizontal. This involves adjustments of the diffractometer motors Y1, Y2, Y3, and TRX. Details of zeroing the diffractometer detector and sample motors is described in Chapter 5.

4.1 The diffractometer

The heart of the surface diffraction station is a large 5-circle diffractometer (2 sample movements, 3 detector movements). A schematic of the relevant motor movements of the diffractometer is given in Fig. 4.1. Movement directions of the hexapod are shown in Fig. 4.2.

The diffractometer can be configured in one of two sample geometries, i.e., either “vertical” geometry, in which the sample face is vertical (and hence the surface normal is horizontal) for which the hexapod is mounted with its z -axis horizontal on the ω_v (OMEGAV) rotation stage; or “horizontal” geometry, for which the sample surface is horizontal and the hexapod z -axis is nominally vertical and is mounted on the ϕ (PHI) rotation stage in the ω_h (OMEGAH) cradle. Hence, the sample movements for the vertical geometry are ω_v and α (ALPHA), while those for the horizontal geometry are ϕ and ω_h . The relevant detector motors are γ (GAMMA), δ (DELTA), and ν (NU).

4.2 Homing motors

All the motors shown in Fig. 4.1 have encoders and can be homed. Once a motor has been homed, its “dial” value (i.e., the one read by the encoder) can be compared to any previous value for that motor, as long as the previous value was also noted after the motor had been homed. Each motor has, in addition to the dial value, a “user” value – this is the one that the user can redefine and set at will to suit his/her experiment. Both values can be queried using the SPEC command `wm mot` (e.g., if the present value of DELTA is queried, type in `wm del`).

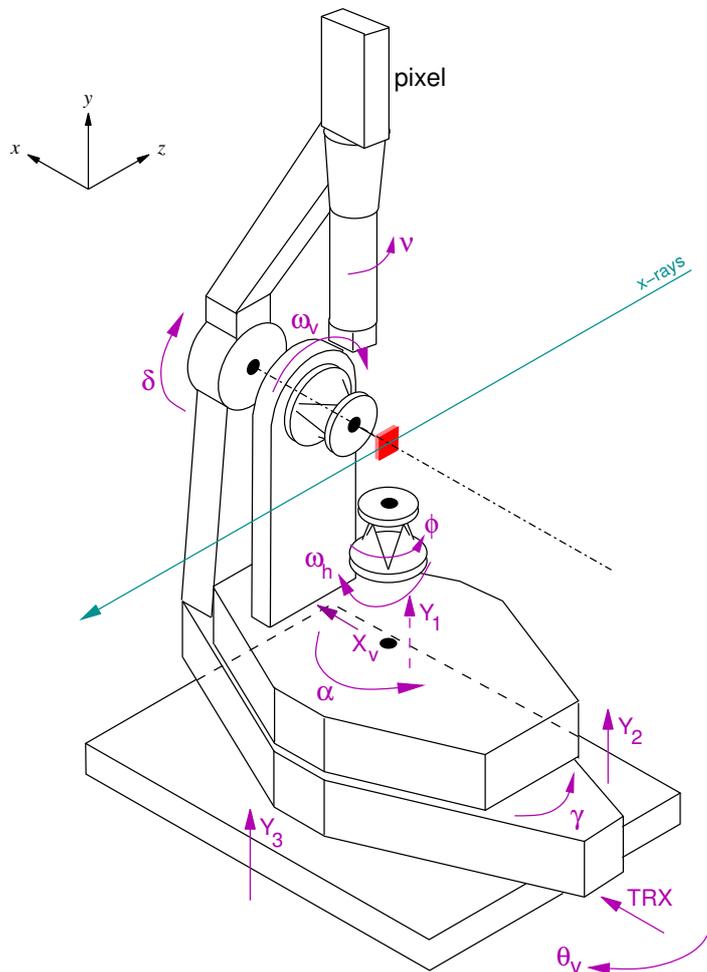


Figure 4.1: Motor movements of the 5-circle surface diffractometer. Positive directions are given by the arrow heads. Two geometries are available, either with the hexapod axis horizontal and the sample surface in the vertical plane (“vertical” geometry), or with the hexapod axis vertical and the sample surface in the horizontal plane (“horizontal” geometry).

Homing of the motors can be done directly via the MM4000 controller facility outside SPEC. If this is done, SPEC knows nothing about this, even if the motors had previously been homed in SPEC. Hence, **NEVER TRY TO HOME THE MOTORS OUTSIDE THE SPEC ENVIRONMENT!**

To home a motor from SPEC, type `home mot` (e.g., `home de1`). This normally takes a few seconds. Once completed, the dial value can be directly compared with any previously noted dial values for that motor. So, for instance, if the user knows that the desired dial value for Y1 after it has been homed is 10.233 mm, and the present dial value of Y1 directly after homing is 7.394 mm, the motor must be moved in a positive direction by 2.839 mm (`mvr Y1 2.839`).

Motor movements and values in SPEC are based (unless otherwise specified) on the user values (*not* the dial values), hence typing in `mv de1 10` will move DEL to a position where the user value is 10° . When aligning the diffractometer motors, it is normal to redefine the zeros of the user values, using the SPEC command `set mot`

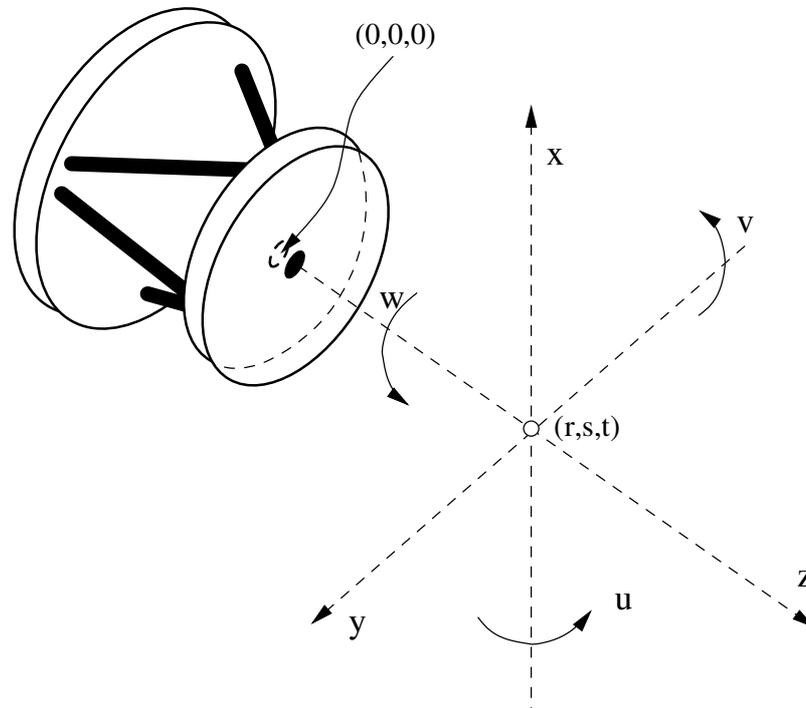


Figure 4.2: Motor movements of the hexapod. The point of rotation is given by entering the coordinates (r, s, t) , which are defined in a Cartesian coordinate system with its origin at the center of the back surface of the hexapod plate. Translational movements are in mm along x , y , and z , while rotations are given in degrees by u , v , and w .

0, which sets the present user position to 0. This has no effect on the dial value, which will remain unchanged.

4.3 Aligning without beam

The x-ray beam at the SD station enters the hutch horizontally at 1385 mm above the floor. Three marks in the hutch indicate this height. They are shown in Fig. 4.3.

Alignment without beam is performed using the theodolite available at the SD station. The theodolite should be adjusted so that the viewing optics rotate in the horizontal plane at 1385 mm. Horizontality to better than 0.3 seconds of arc ($1.5 \mu\text{rad}$) is easily achieved using the three adjustable feet and centering the gas bubble on the optics head (see Fig. 4.4). A quick check for horizontality can be made by aligning up the theodolite crosshairs and viewing each of the three marks shown in Fig. 4.4.

The center of the diffractometer should lie in the path of the x-ray beam. Sections 4.3.1 and 4.4.1 detail how this is performed.

4.3.1 Diffractometer height and tilt – Y1, Y2, and Y3

Firstly, the diffractometer height and tilt must be adjusted using motors Y1, Y2, and Y3, so that the ALPHA and GAMMA rotations are in the horizontal plane and the diffractometer center is at a height of 1385 mm.

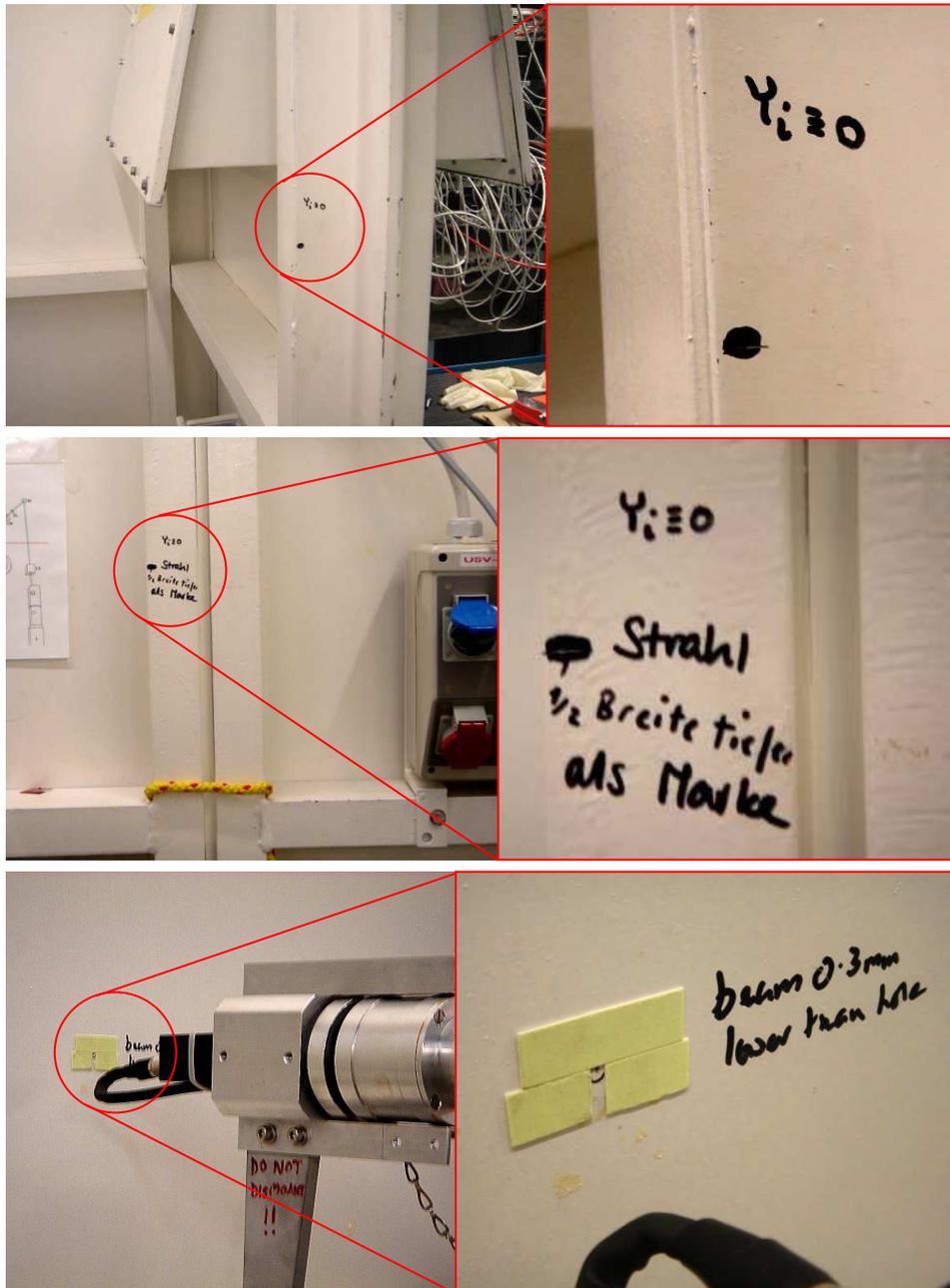


Figure 4.3: Three marks in the SD experimental hutch indicate the height of the incoming x-ray beam at 1385 mm. The upper pictures show the mark at the hutch door; the middle images that on the upstream wall (to the right of the EH2 slits); and the lower panel the position of the beam as it hits the downstream wall (behind the x-ray eye).

The first step is to zero the tilt – the absolute height of the diffractometer is performed after this.

In both the vertical and horizontal geometries, a 100 cm-rod is approximately horizontally fixed to the hexapod plate using mounts provided by the beamline, so that any rotation of the ALPHA table, on which the hexapod sits, will cause the rod to sweep out a large arc. With $\text{ALPHA} = 0^\circ$, focus the theodolite on the

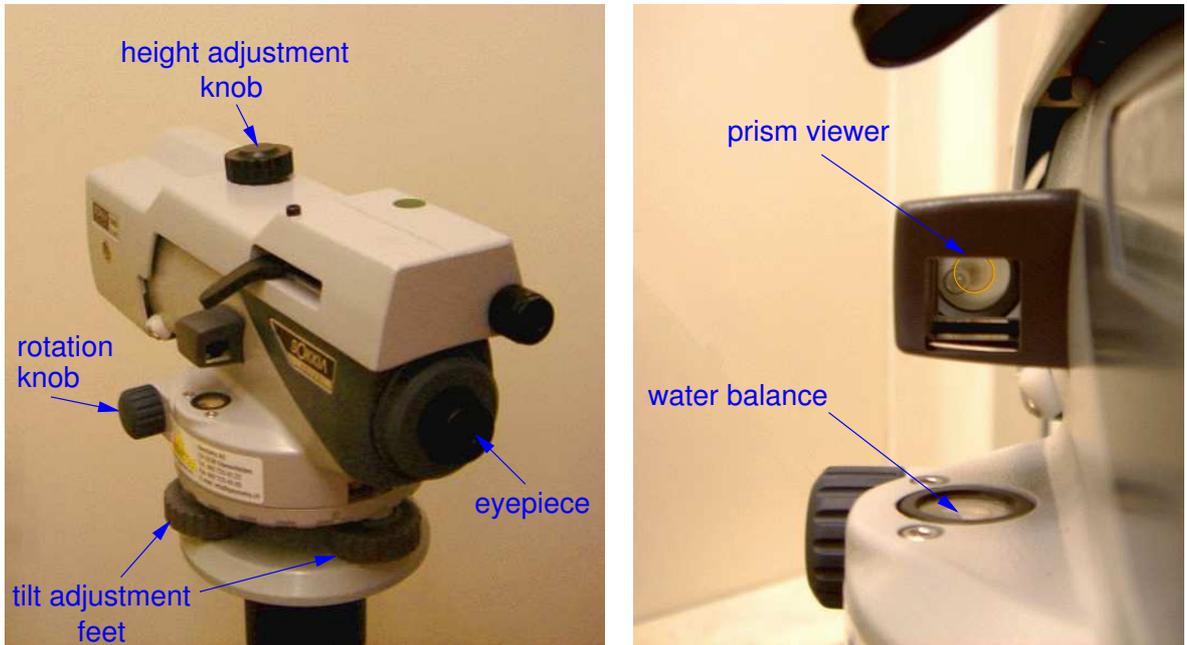


Figure 4.4: The Sokkia B20 theodolite. The horizontality of the theodolite can be adjusted using the three tilt adjustment feet until the air bubble of the water balance as viewed through the prism is centered. Note that in the right image, the bubble is not centered and that the front of the theodolite is tilting upwards and to the right. The theodolite can be swung by hand left and right, while fine adjustments are made with the rotation knob.

outside tip of the rod (you adjust the height of the theodolite with the height adjustment knob and the lateral direction with the rotation knob) so that the crosshair in the field of view of the theodolite intersects the tip of the rod, see Fig. 4.5).

Next, rotate ALPHA as far in the positive direction as possible. This will also require the rotation of the GAMMA stage to allow such large movements of the ALPHA table (BEWARE! If you move only the ALPHA stage, it will collide into the upright on the GAMMA table that supports the DELTA arm, see Fig. 4.1). If the diffractometer is not horizontal, the rod tip height will have changed and be observable through the theodolite. The tilt of the diffractometer can then be adjusted by moving motors Y1, Y2, and Y3 (see Fig. 4.1). In general, three positions of ALPHA (e.g., 0° , 90° , and as close as possible to 180°) are needed to compensate for the tilt (a minimum of three noncollinear points define a plane).

Once the tilt has been zeroed, the absolute height of the diffractometer must be adjusted so that the center is at a height of 1385 mm. To do this in the vertical geometry, remove the rod and mount the crosshairs provided by the beamline (not to be confused with the crosshairs of the theodolite), so that the two tungsten wires are in the vertical plane (see Fig. 4.6). Return ALPHA to 0° . Readjust the height of the beamline crosshair using the hexapod motors (SPEC command `hmvx` or `hmvx` in the vertical mode, `hmvz` in the horizontal mode), so that the horizontal hair is exactly at 1385 mm (i.e., it should overlap the horizontal crosshair of the theodolite). Now rotate OMEGAV by 180° (SPEC command `mvr ov 180` or `mvr ov -180`). Because there is no guarantee that the horizontal beamline crosshair lies on the rotation axis of OMEGAV (in fact, it is highly unlikely that

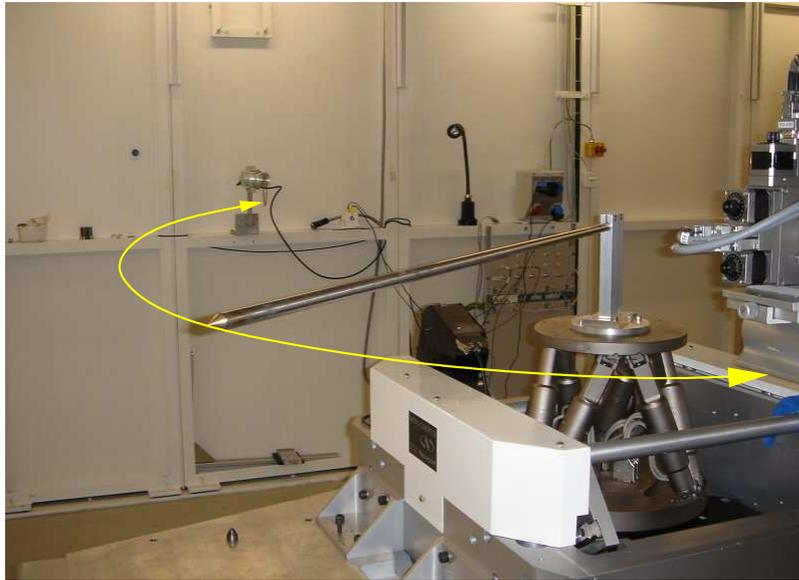


Figure 4.5: Image of the setup used in the horizontal geometry to zero the tilt of the surface diffractometer. By letting the tip of the rod sweep out a large arc as ALPHA is turned, the tilt of the diffractometer can be adjusted using the motors Y1, Y2, and Y3.

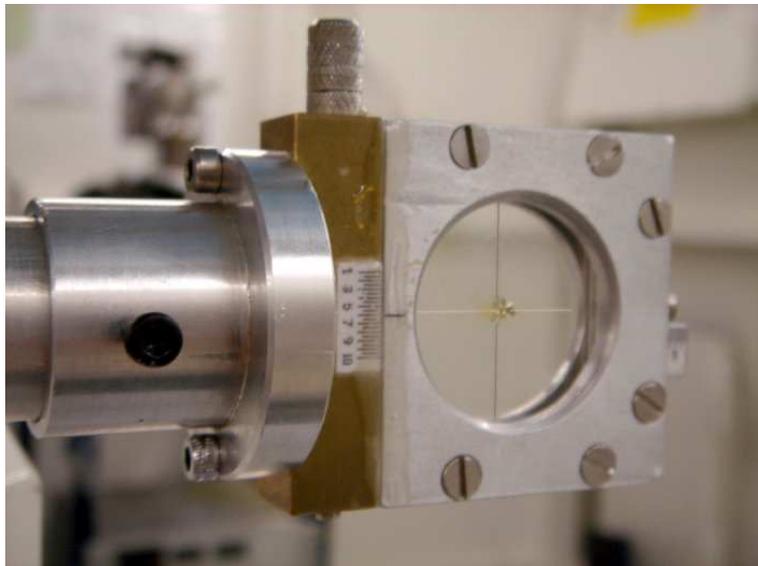


Figure 4.6: An image of the beamline diffractometer crosshairs.

it does), this rotation will most likely mean that it has shifted vertically. Adjust the heights of Y1, Y2, and Y3 by the same amount (using the SPEC command `mvyr`) so that the beamline horizontal crosshair (which moves with the diffractometer) moves half way back to the theodolite horizontal crosshair. Then readjust the hexapod motors so that the two crosshairs overlap. Repeat this procedure until rotation of OMEGAV by 180° results in

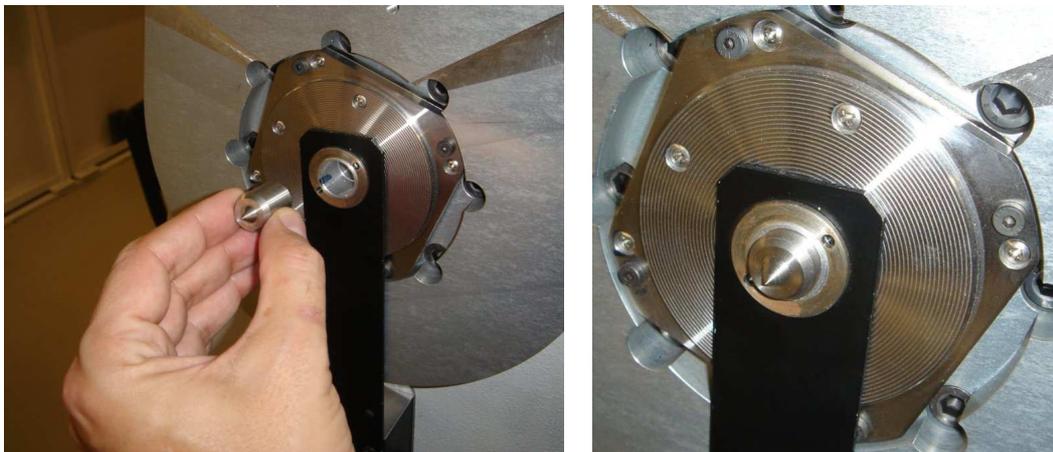


Figure 4.7: Images of the metal plug inserted in the central hole of the DELTA axis, used to adjust the absolute height of the diffractometer when used in the horizontal mode.

no change in the height of the theodolite horizontal crosshair.

In the horizontal geometry, there is no OMEGAV stage, and therefore adjustment of the absolute height of the diffractometer cannot be performed in the manner described above for the vertical geometry. Here, we simply insert a metal plug (provided by the beamline) into the central hole of the axis of the DELTA arm and adjust the height of the diffractometer until the central spot is at the horizontal theodolite crosshair (see Fig. 4.7). A second rough check can be performed using the rod holder, though this depends on the hexapod being exactly at its nominal zero position, i.e., with the top surface of the hexapod plate being exactly 170 mm below the diffractometer center and having no tilt, and the OMEGAH cradle being perfectly horizontal.

As a last point, a reliable set of values for the DIAL positions of Y1, Y2, and Y3 (viewable in SPEC using the command `wa`) has already been obtained by frequent calibration of the diffractometer by the SD beamline scientist (me). The values are only dependable if the user is sure that the motors Y1, Y2, and Y3 have been homed *in SPEC* (see also Section 4.2).

4.4 Aligning with beam

The first check is to ensure that the x-ray beam is indeed passing through the diffractometer at the nominal 1385 mm. Firstly, one has to optimize the beam for flux, focus, divergence, etc., then adjust the beam position using the roll motor of the second monochromator crystal of the double-crystal monochromator (lateral shifts) and the downstream motor of the second mirror (vertical adjustment) (highlighted GUI panels 1 and 2, respectively, in Fig. 4.8). This is done by observing the focussed beam in the control hutch on the x-ray eye monitor, using the x-ray eye mounted at the back of the experimental hutch. The video screen has a spot drawn on it, indicating the correct beam height and lateral position.

As a belt-and-braces check, once the beam has been positioned, the user can attach a piece of green x-ray paper on the exit of the flight tube and burn a spot there. With the theodolite, the height of this burn spot

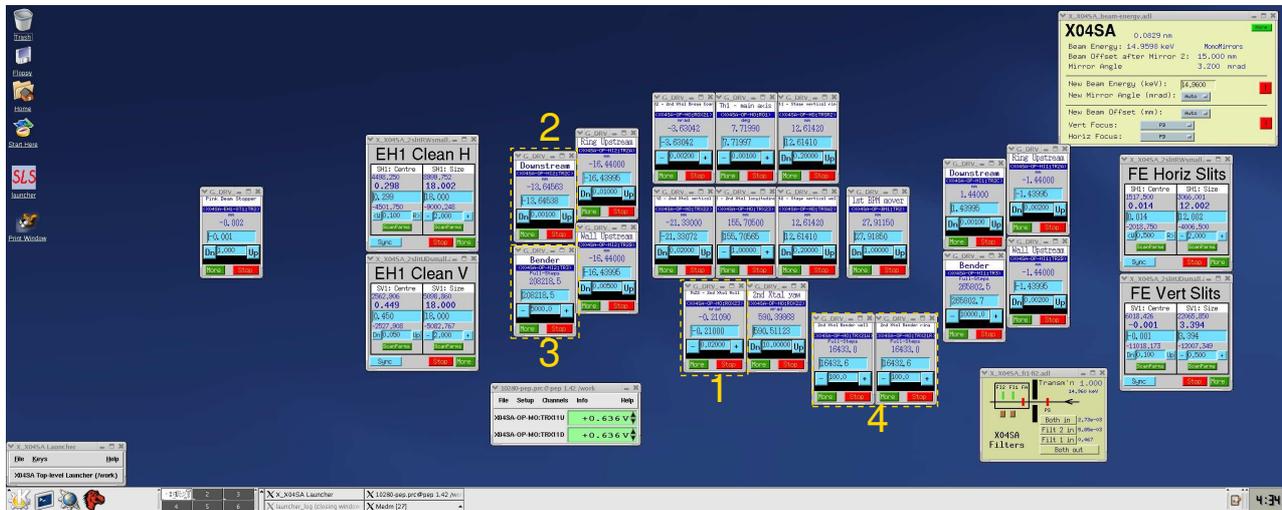


Figure 4.8: Screen shot of the optics display at the MS beamline. Four relevant GUI panels mentioned in the text have been highlighted.

can be determined and should be at 1385 mm.

4.4.1 Lateral displacement of the diffractometer – TRX

The remaining alignment of the position of the entire diffractometer is to bring the center of the diffractometer into the path of the x-ray beam in the horizontal plane. For this procedure we again use the beamline crosshairs, but now we use the x-ray eye instead of the theodolite. Because blooming is strong in the fluorescent screen of the x-ray eye, it is best to defocus the beam both vertically (relaxing the second mirror bender, GUI panel 3, Fig. 4.8) and horizontally (relaxing the sagittal focuser, GUI panels 4, Fig. 4.8) to carry out this operation, or otherwise the position of the tungsten wire crosshairs becomes washed out. This is particularly true for the vertical wire (the crosshair of interest for this particular alignment procedure), due to the $10\times$ larger horizontal divergence compared to that in the vertical direction.

First, make sure that the detector on the DELTA arm is out of the way from the field of view of the x-ray eye, by moving DELTA to 90° .

In the vertical geometry, the ALPHA table should be rotated between 0 and 180° (BEWARE! Again, make sure also to rotate GAMMA enough so that the ALPHA table does not collide into the upright), which turns the crosshairs also through 180° around the vertical axis of the diffractometer. The center of the diffractometer lies exactly in between the two positions of the vertical crosshair (as viewed on the x-ray eye monitor). The diffractometer is centered using the motor TRX (see Fig. 4.1) which moves the whole diffractometer horizontally. Fine adjustment of TRX is best made by recentering the vertical crosshair at each iteration using the diffractometer motor XV so that it is collinear with the ALPHA and GAMMA rotation axes.

The same procedure is carried out in principle for the horizontal geometry, only here the crosshairs are rotated between 0 and 180° using the PHI motor (with ALPHA held at 0°). Recentering of the crosshair at

each iteration is now performed using the hexapod motors.

Chapter 5

Detector and sample alignment procedures

This chapter describes how to zero the detector and sample using the diffractometer motors ALPHA, OMEGAH, DELTA, GAMMA and NU.

You have beam, the diffractometer has been correctly positioned, and you have mounted your sample on to the hexapod. Your next task is to align the motors ALPHA, DELTA, GAMMA and NU (for the vertical geometry) and OMEGAH, DELTA, GAMMA and NU (for the horizontal geometry). The azimuthal rotation motors OMEGAV (vertical geometry) and PHI (horizontal geometry) do not in principle have to be aligned (zeroed), as this is accommodated when obtaining the orientation matrix in the azimuthal miscut angle (see Chapter 6).

For all the below procedures, alignment is achieved using the PILATUS pixel detector. **Be sure to use appropriate filters – even a brief exposure of the detector to focussed direct beam will result in a permanent dead region where the pixel was exposed to x-rays¹. Work with extreme caution!!**

5.1 Aligning the detector motors - DELTA, GAMMA, and NU

The alignment procedures described in this section are common for both vertical and horizontal geometry.

The beam can be monitored by the pixel computer using the software package `tvx`. In `tvx`, type in the command `exposemb 1.1`². By surrounding the signal with a box using pull-down menus in the pixel image display panel, the integrated intensity, maximum intensity, average intensity, and center of mass of the signal

¹A focussed beam in the energy range spanning 8 to 25 keV photon energy will typically require a filter transmission of 10^{-6} in order for its signal to remain in the linear response region of the detector (below 10^5 cts/s/pixel). Increasing the transmission to 10^{-3} appears to cause no permanent damage to the detector, although this is obviously to be avoided.

²This command tells `tvx` to repeatedly perform 1.1 s-long exposures and allow statistics to be calculated on the region of interest defined by a box, which can be repositioned and resized as the user wants. Exposures of 1.0 s are avoided, as (for arcane reasons) in this mode, this can occasionally lead to the program crashing. In other words, exposure times in excess of 1.0 seconds should be chosen for the command `exposemb`.

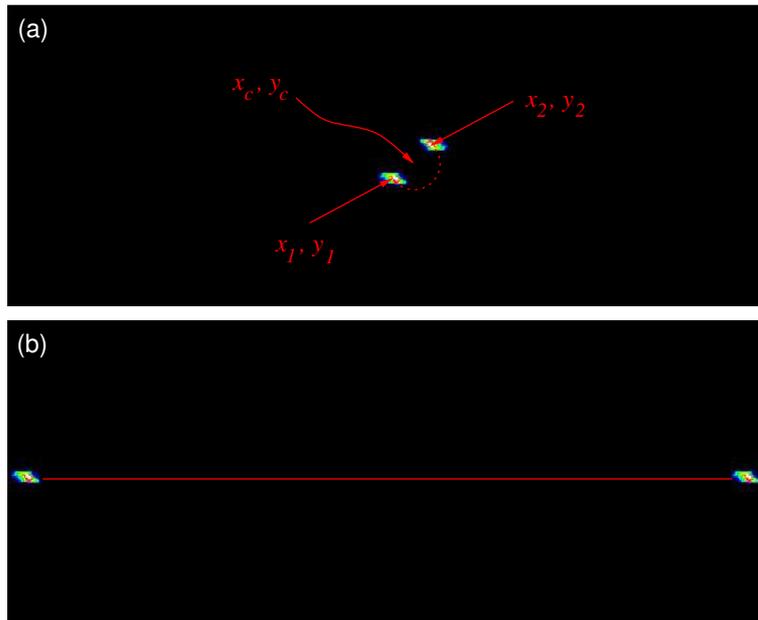


Figure 5.1: (a) The GAMMA and DELTA motors can be zeroed once the direct beam no longer describes a circle, i.e., it rotates about a fixed point, as NU is rotated. (b) By moving the detector to the left and right with the GAMMA motor, its horizontality can be checked by seeing if the straight line connecting the two extreme positions has a constant y -value on the detector display.

(“centroid”) can be constantly monitored.

First, ensure that the sample (if already mounted) is retracted out of the path of the direct beam. In the vertical geometry, this is achieved by moving XV in a positive direction (SPEC command `mvr xv`), while in the horizontal geometry, one must contract the hexapod (SPEC command `hmvz`). Next, introduce appropriate filters and bring the pixel detector into the direct beam by moving DELTA and GAMMA to zero (not forgetting that nominal zero might be far away from the direct beam, in which case you have to zero “by eye”, which is usually more than sufficient for this initial stage) and, if necessary, opening the fast photon shutter (SPEC command `shop`). By surrounding the signal with a box, the position (x_1, y_1) of the centroid on the pixel array can be monitored. Note the values of the beam centroid (typical values might be $x = 237.7$, $y = 89.1$).

Now rotate the detector around its own NU axis by 180° (SPEC command `mvr nu 180` or `mvr nu -180`) and observe how the direct beam rotates on the pixel display [see Fig. 5.1(a)]. Once the NU rotation is complete, note the new beam centroid positions (x_2, y_2) . The center of the NU rotation (x_c, y_c) is therefore at $(x_1 + x_2)/2$, $(y_1 + y_2)/2$. Adjust the GAMMA and DELTA positions using the SPEC commands `mvr gam` and `mvr del` until the direct beam centroid is at (x_c, y_c) . Zero the GAMMA and DELTA values by typing in SPEC `set gam 0` and `set del 0`.

The zero of NU is found by moving GAMMA until the direct beam is close to one side of the pixel display [See Fig. 5.1(b)]. In the present setup, this is about 2° away from `GAMMA = 0`. Note the y -value of the centroid, then move the direct beam to the opposite edge of the detector. The y -value of the centroid should

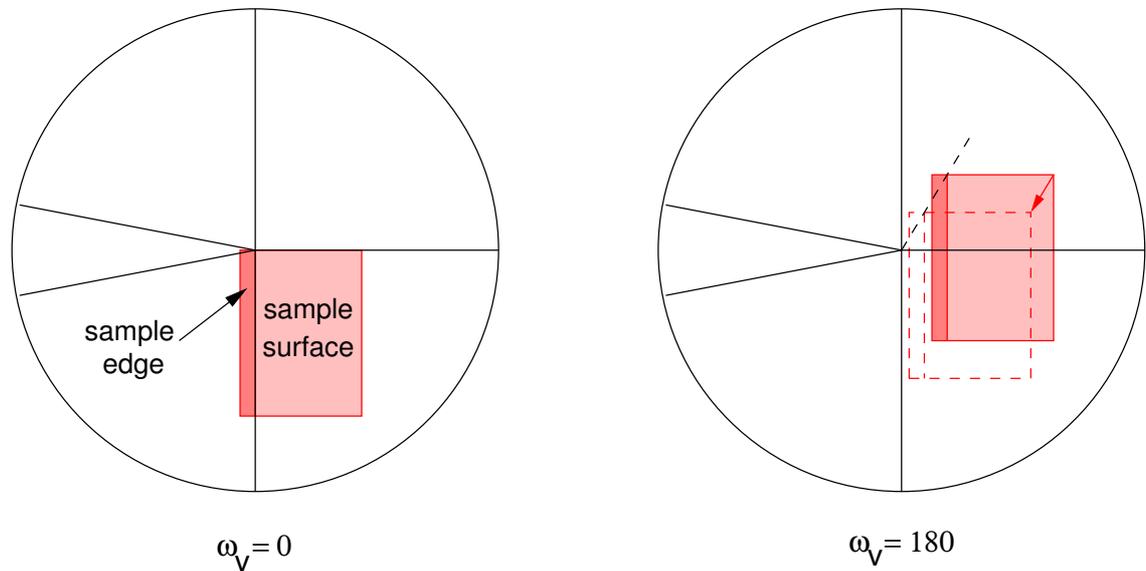


Figure 5.2: If the sample is visible using the theodolite (the view from which is shown here as a circle with crosshairs), the sample can be accurately aligned by eye. The position of a sample (here a flat square plate, viewed from an oblique angle) is seen to wobble as one rotates OMEGAV from 0 to 180°. By repositioning the sample to the position (shown here as a dashed box) mid in between the two end positions, the sample is centered.

not have changed (and indeed should also be equal to y_c if $\text{DELTA} = 0$). If there is any difference, rotate NU until the y -value of the centroid remains independent of GAMMA movements. Zero the NU rotation by typing in SPEC `set nu 0`.

5.2 Aligning the sample – hexapod, XV, ALPHA, and OMEGAH

Apart from the hexapod x - and y -centering, the alignment of the hexapod z -position (using XV or the hexapod z -movement), the angle of incidence (ALPHA in vertical geometry and OMEGAH in the horizontal geometry), and the alignment of the surface normal to the axis of sample rotation (OMEGAV in vertical geometry and PHI in horizontal geometry) all affect one another. Hence the procedures described below should be performed iteratively until they all agree.

5.2.1 x - and y -centering

The center of the front surface of the sample should be as accurately as possible at the center of the diffractometer and the surface plane should contain the direct beam. The first procedure is to get the sample centered, both in x and y , so that the direct beam passes across the middle of the surface; as well as in z , so that at zero incidence angle (ALPHA in the vertical mode and OMEGAH in the horizontal mode), half the beam width is occluded.

In the vertical geometry, the x and y -alignment can be monitored with the theodolite, as long as the sample

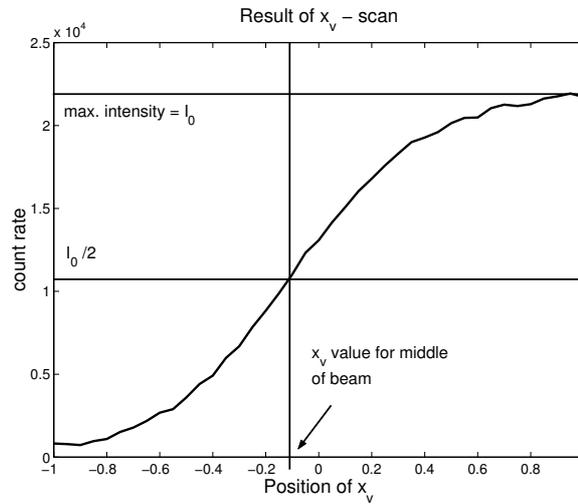


Figure 5.3: A scan across the direct beam in the horizontal direction using XV. The derivative of the curve yields the horizontal beam profile.

is visible (e.g., by removing a Be-dome, or through a vacuum viewport) and by rotating OMEGAV. By adjusting the hexapod x- and y-pseudo motors, the sample can be rapidly centered (see Fig.5.2).

In the horizontal mode, or if the sample cannot be seen visibly (as for samples in UHV Be-dome chambers), centering of the sample must be performed by occluding the direct beam using the hexapod x- and y-motors. The user should have a good idea of the dimensions of the sample and how it is mounted, to accurately interpret the occluding of the direct beam – for example, a sample holder might be misinterpreted as being the sample itself.

5.2.2 z-centering

The z -position of the sample should be adjusted so that at 0° incidence, half the beam is occluded. This can be monitored using the pixel detector and noting at which point the integrated intensity drops by 50%. In the vertical mode, this adjustment can be performed either with XV (see Fig. 5.3), or with the hexapod z -movement, while in the horizontal geometry, only the hexapod z -movement can be used.

5.2.3 Sample angular alignment – u and v

In the vertical geometry, the sample can be aligned using a laser pointer, as long as there is an unobstructed path for a specular reflection. The reflected laser spot on the wall (Fig. 5.4) is most conveniently monitored using the webcam installed in the SD Experimental Hutch (which is controllable from the Windows computer in the SD Control Hutch). If the sample normal is not coaxial with the OMEGAV rotation axis, the reflected beam will execute an elliptical path. Alignment of u and v (SPEC commands `hmvu` and `hmvv`, respectively) is achieved as follows. The position of the reflected beam is noted for one OMEGAV angle and again for an angle ± 180 from the original OMEGAV angle. The u and v hexapod pseudo motors are then adjusted to reposition

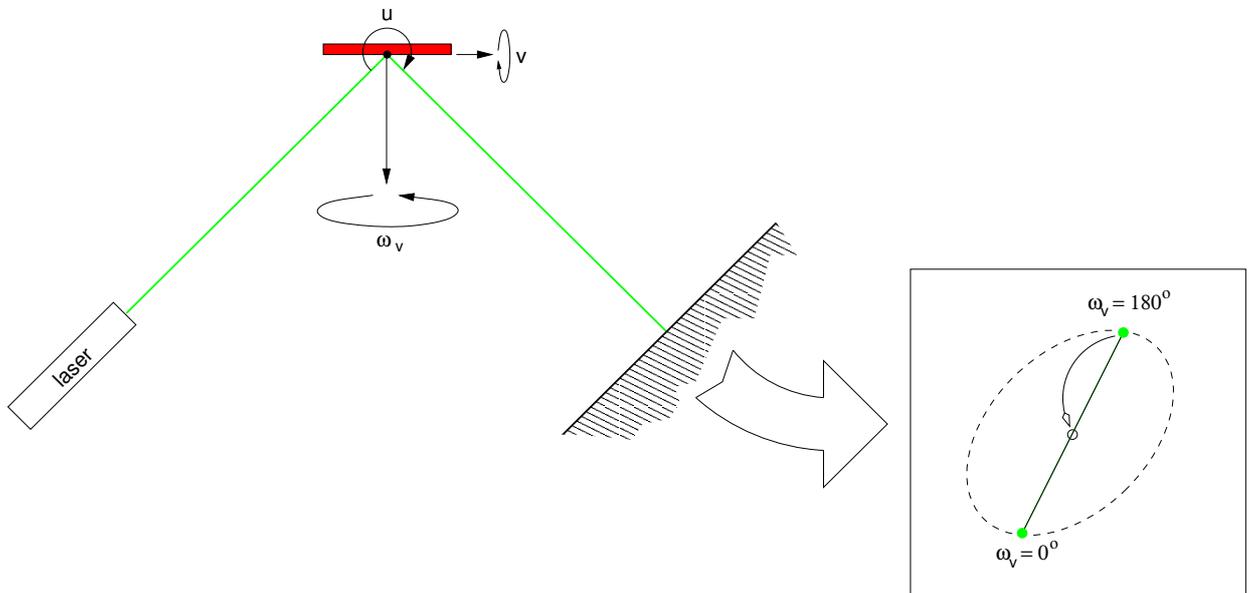


Figure 5.4: Alignment of u and v using a laser pointer.

the beam so that it is exactly in between the two marked points³.

Often, it is not possible to use the laser pointer and u and v alignment must instead be achieved by observing changes in the position of specularly reflected x-rays. The best incident angle (ALPHA in vertical, OMEGAH in horizontal geometry) is in general one sufficiently far away from the direct beam that this can be moved out of the field of view of the pixel detector, but should be kept to below the $(0, 0, 1/2)$ position of the specular rod. It lies beyond the scope of this manual to explain in detail why this should be so, but briefly, it has to do with sample miscut angles, which can often be of the order of magnitude of a few tenths of a degree. For typical energies of 12 keV and lattice constants of 4 Å, the $(0, 0, 1/2)$ position lies at about 3.5° .

The sample is turned into the beam using ALPHA or OMEGAH by the desired amount (not forgetting, however, that at this stage, these motors might not yet have been accurately zeroed). The sample is rotated around its nominal normal axis (OMEGAV in vertical, PHI in horizontal geometry) to an angle where the u -axis (x) is at right-angles to the incoming beam (see Fig. 4.2). The reflected beam is monitored on the pixel detector and the centroid of the signal is noted. Then the sample is rotated around OMEGAV or PHI by 180° , and the new position of the reflected beam is noted. Adjust u until the reflected signal on the pixel detector is exactly in between the two noted positions. Now turn the sample by 90° using OMEGAV or PHI (so that the v -axis of the hexapod, i.e., y) is at right-angles to the incoming beam. Repeat the above procedure for the hexapod v -axis.

³Note that if you discover that the reflected laser beam executes a figure of eight or “infinity” loop over 360° , this is a signature of a bent sample surface.

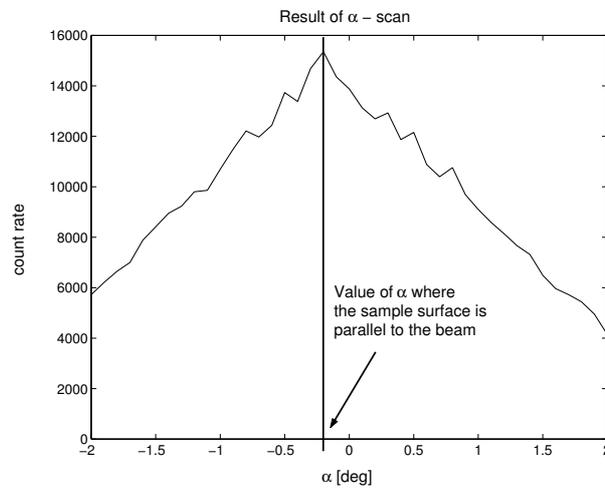


Figure 5.5: The signal maximum should occur when the sample is parallel to the incident beam.

5.2.4 Incoming angle - ALPHA or OMEGAH

Depending on the geometry, there are several ways to optimize the beam. The two easiest are explained here.

Once the u and v motors have been aligned (see the previous subsection), and also assuming that you have already aligned GAMMA and DELTA, you can move to a θ - 2θ position somewhere below the $(0, 0, 1/2)$ point on the specular rod. In the vertical mode, this involves the motors ALPHA and GAMMA, while in the horizontal mode, the motors are OMEGAH and DELTA. Now simply adjust ALPHA (OMEGA H) until the centroid of the reflected beam is seen to be exactly on the previously determined diffractometer center (x_c, y_c) (see Section 5.1). As an extra check, the position of the reflected beam should not change as one rotates OMEGA V (PHI).

The second method involves occluding the direct beam by tilting the sample with ALPHA (OMEGA H). In the ideal case, this produces a triangular plot when the intensity is plotted as a function of angle. The angle value for the apex of the plot is the zero of ALPHA (OMEGA H).

Chapter 6

Obtaining reflections for the orientation matrix

Obtaining the diffractometer angles for Bragg reflections is especially simple using the pixel detector. This is because, in contrast to peak searching using traditional point detectors, the local neighborhood in reciprocal space is also observable.

We consider a standard case, in which the nominal crystallographic orientation of the surface is known [e.g., the (110) surface of NdGaO_3], but the azimuthal angle (OMEGAV or PHI) is unknown. There will also be miscut angles, which can amount to a few tenths of a degree.

SPEC is set up to know the crystal parameters. For a given Bragg peak (hkl), SPEC can therefore work out the motor angles, though the value for OMEGAV (PHI) can be wrong by many degrees, especially if there is no information about the orientation of the crystal in the surface plane. Despite this, the values for GAMMA and DELTA (assuming zero miscut angles) are fixed. Finding the reflection is therefore a simple matter of rotating the sample around its normal until the Bragg peak is found on the pixel display. In addition, because the pixel detector “sees” the surrounding neighborhood in reciprocal space, the arrival of the Bragg peak can be predicted in advance by the appearance (stage left or stage right) of a crystal truncation rod signal as it moves towards the Bragg maximum. This is explained here in more detail.

The procedure is as follows. Move the motors with SPEC to a desired (hkl) out-of-plane Bragg peak (make sure it is not a systematic absence!) using the SPEC command `mvhk1` or `br`¹. If you are using surface coordinates (recommended!), this means $l \geq 1$. If you are sure that the direct beam is not in the frame of the pixel detector, remove all the filters. Start to turn OMEGAV (PHI) using the SPEC command `tw ov (tw phi)`. A step size of 0.5° or smaller is recommended to avoid jumping over the Bragg peak without noticing it. As you slowly move the motor, observe the pixel display in `tvx` using `exposemb 1.1`. At some time, a crystal truncation rod (CTR) signal will drift into the display, either from the left or right edges, depending on whether you are rotating the

¹Make sure that all the correct crystal parameters have been entered into SPEC using the command `setlat`, the x-ray energy is correctly set (using the SPEC command `setlambda`) and that the miscut angles are not wildly off (if working in surface coordinates, they should be close to zero).

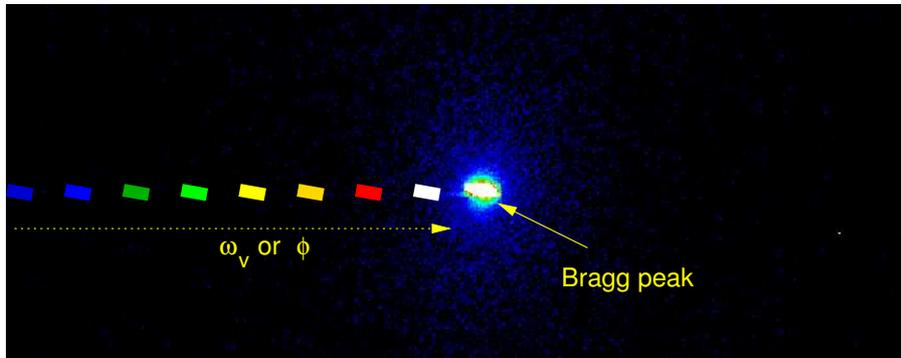


Figure 6.1: Finding the angular coordinates of a Bragg peak. First, optimize OMEGAV (PHI) so that the CTR/Bragg peak signal is maximized, which should happen when the CTR converges with the Bragg peak. Then reposition the pixel detector using GAMMA and DELTA until the centroid of the signal is exactly at (x_c, y_c) .

sample clockwise or anticlockwise (see Fig. 6.1). As the CTR signal approaches the Bragg peak, both of them will become more intense, and filters will have to be introduced, in order to avoid saturation (although it is impossible to damage the detector with Bragg peak signal).

Using the box functionality in `exposemb 1.1` in `tvx`, the integrated intensity can be monitored. As the CTR signal approaches the Bragg peak, the latter will also become more intense as the foot of it is intercepted by the Ewald sphere. The OMEGAV (PHI) step size will have to be decreased until the maximum intensity is known within typically 0.005° or less (depending, of course, on the quality of the sample). Ideally, the optimal OMEGAV (PHI) angle is where the CTR signal is directly on the Bragg peak, though often small movements of OMEGAV (PHI) can have a significant effect on the signal intensity without any detectable CTR movement on the display.

Once OMEGAV (PHI) has been optimized, the DELTA and GAMMA motors are also tweaked so that the Bragg peak is exactly at (x_c, y_c) (see Fig. 6.1). At this point, the Bragg peak can be added to the list of reflections used to obtain the orientation matrix.

Chapter 7

Creating flatfield files

This chapter describes the important procedure for obtaining the pixel sensitivity distribution (so-called “flat-field” file).

7.1 Setting up the diffractometer and scattering source

Tune the synchrotron radiation to the desired energy. The scattering sample is mounted on the hexapod holder¹ (whether in vertical or horizontal geometry) and can be positioned relative to the incoming focussed x-ray beam² by using the hexapod motors, the vertical position of the whole diffractometer (Y1, Y2, and Y3), or, in the vertical geometry, the motor XV (see Fig. 7.1), and OMEGAV or OMEGAH. The DELTA position of the pixel detector should also be optimized and typically lies between 60 and 120°. In general, ALPHA and GAMMA are left at 0°.

The adjustment of the sample and pixel positions is an iterative procedure, which might require several rounds before a satisfactory intensity has been achieved (more than 100 cts/s/pixel is desirable).

It is important, when recording flatfield data, that the angular distribution of the scattering intensity is constant across the field of view. One cannot merely check different regions of the detector to see if they deliver the same integrated intensities, as it this which is being calibrated. Instead, one should choose a small region of interest using the `box` pull down menu option of `tvx` and running `exposemb 1.1` on the terminal. The size of the box should be large enough to get integrated counts with sufficiently low statistical noise (of the order of 100 000 or more). Then, move DELTA and GAMMA by 1.6° and 3.7°, respectively (the angular extents of the detector’s field of vision), and see how the integrated intensity changes in the box. This should be less than the accuracy with which you want to record the flatfield data ($\sim 0.1\%$).

¹We have found lead glass to be a good elastic scatterer, although it should not be used at or above the L1 absorption edge at 15 858 eV.

²The degree of focussing, especially the horizontal (sagittal) focussing, can be adjusted to optimize the scattered flux once the pixel detector and sample positions have been more or less optimized.

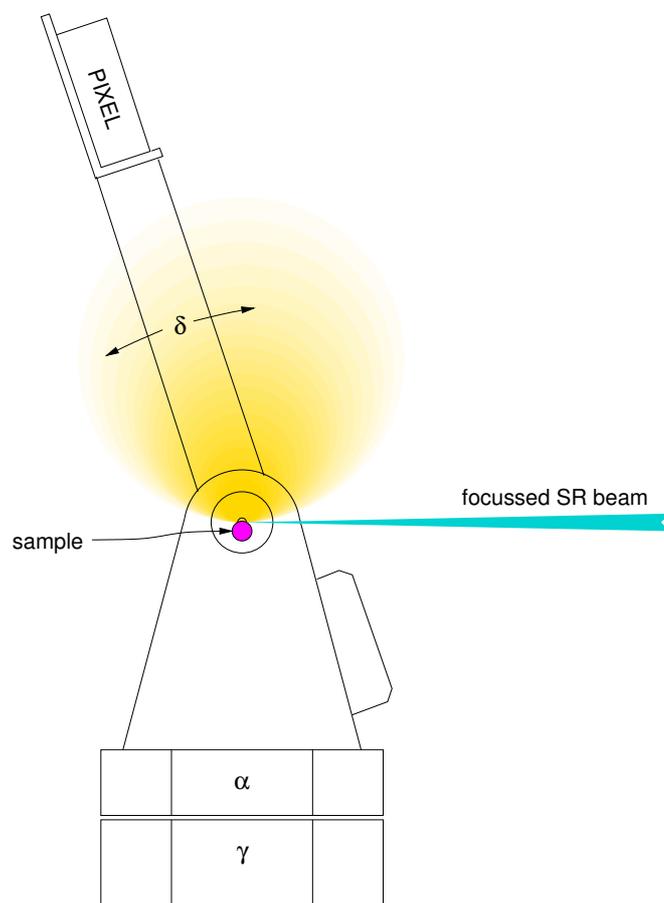


Figure 7.1: A schematic of how to set up the diffractometer to record flatfield files.

7.1.1 Choice of scattering sample

For flatfield correction files, the sample should be either amorphous (e.g., a liquid in a capillary), or a single crystal – polycrystalline material is to be avoided, as it is difficult to find detector positions which do not intercept one or more Debye Scherrer cones and produce brighter regions in the field of view. One wants to record *elastically* scattered radiation, hence the sample should not contain elements with strong absorption edges close to the wavelength being used, as then fluorescence signal with lower photon energy will dominate, and the pixels' responses may be different at this energy. Nonetheless, try to find a material with as high an atomic number, Z , as possible.

For example, the user wants to record at 16 keV. Strontium (Sr) has its K 1s absorption edge at 16 105 eV, hence one might be tempted to use, for example, a SrTiO₃ single-crystal substrate. However, the foot of this absorption edge extends to lower energies, which means that significant fluorescence will still be seen at 16 keV. Experience has shown us that one should move 500 eV or more below the nominal absorption edge to sufficiently suppress fluorescence signal. Hence, we should discount using SrTiO₃, and move up the periodic table to e.g., yttrium (Y) or zirconium (Zr). These two elements are found in yttrium stabilized zirconia (Y:ZrO₂), which is

available as a single crystal. Other crystals with even higher Z are LaAlO_3 ($Z_{\text{La}} = 57$) and NdGaO_3 ($Z_{\text{Nd}} = 60$).

7.2 Creating flatfield files

It is recommended that the user records at least one flatfield file for a typical beamtime of a few days duration. This can be performed either from SPEC using `ct`, or directly in `tvx` using the command `getffdata <frames> <exptime>`.