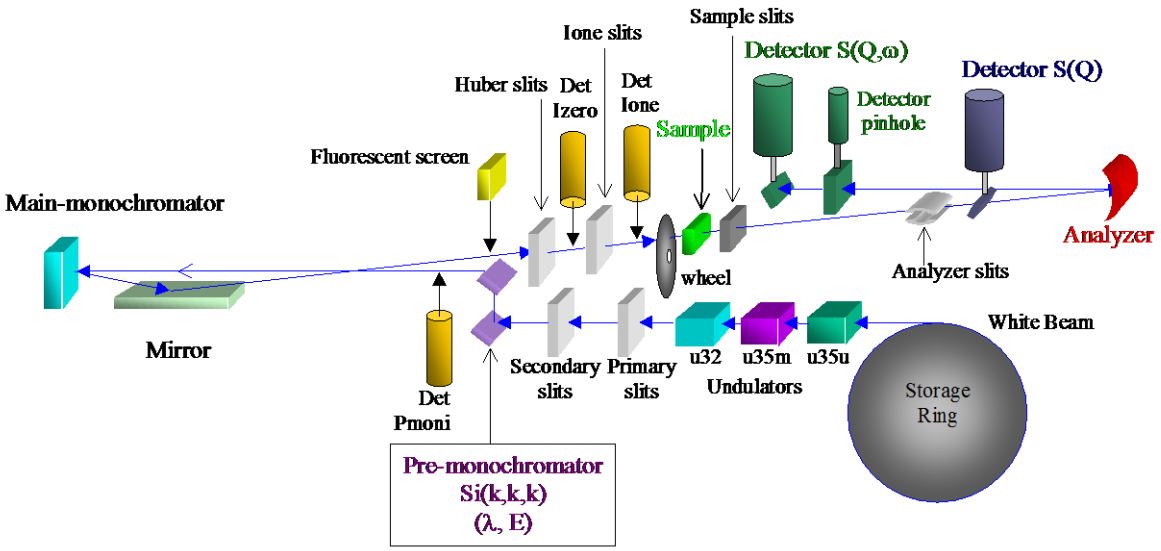
Inelastic X-ray Scattering Beam-line ID28



Version: 27 August 2015

1. FAST CONFIGURATION CHANGE (TORUS&ML)

Assuming that we always work with postmonochromator, to quickly change the energy/resolution configuration, follow this procedure. **N.B.:** This method may not work for the beginning of the beam schedule/after start-up. Primary beam position must be verified first.

1. Put in the small CCD and take the reference of the beam position. Don't move the CCD horizontal/vertical position after this.
2. It is generally preferred to change the energy in the torus configuration. So if you want to switch both from the multilayer to the torus configuration and to change the energy, switch to torus first (before changing energy).
3. Before changing anything print the current reference positions (FOURC, KB, HX2, SLITS) and put them in the "reference positions logbook". Check in the "reference positions logbook" for the set-up (motor positions from the Fourc, KB, HX2 and SLITS windows) that you would like to change to. **Use the latest.**
4. Change the positions of the undulators to the values given in the reference logbook. For the Si (9 9 9) reflection use the u176 undulators, for all other Si reflections use the u32 undulators. Also if you use the ID application to move undulators remember to type in "**reconfig**" in the FOURC window after (see p.7 and use the latest "reference positions logbook" reference). Move tth away from 0 (e.g. umv tth 3). CHECK **tthsetup** if ML mode was changed (with **tth≠0**).
5. Move the following motors to the positions given in the logbook:

FOURC: mono, pmth, pmz, ty, mihexz, miroty, I1h, I2h

KB: hfmth, moniy, hfmy, mlgap; check the remaining and don't touch the benders

HX2: bender

SLITS: cly, clz **(CLOSE THE FRONTEND)**

6. Scan the undulators gaps with pmoni. If you will also scan the primary/secondary slits (see bullet No. 7 below) then first scan mono (with pmoni) and move the premono to 2/3 of the intensity maximum of the scan (see p.8).

7. If the ring current/mode was changed then the primary/secondary slits should be realigned. Follow the procedure of p.5 of the main manual or use the macro “slitscan ()” (keep mono to 2/3).

8. Scan the cly, clz, slits (see p. 10-11). Put mono back to the MAX (see bullet No. 6).

9. Optimize premonochromator: plotselect pmoni; dscan pi1 -0.5 0.5 20 1; umv pi1 MAX (see p. 19 of the main manual).

10. Open i1shg and i1svg to 3 mm.

11. Move the mirror manually, if you switch between ML and torus.

12. umv pi2 0. Check to see if there's a signal in Imirr, if so go to step 12 (N.B. For signal in Imirr DON’T FORGET TO OPEN THE SHUTTER). If not, try to tweak pmth by 0.01 step, looking for some intensity on the LEDs. Otherwise, plotselect pomoni and scan pmth (can be as far as 0.1). Then go to the peak and scan: plotselect Imirr; d2scan mono -0.01 0.01 pmth -0.024 0.024 100 (use 250 for 12 12 12) 1. Check pomoni during the scan. Go to the peak with **two** motors, scan mono and pi2. If pi2 is larger than 0.5, tweak pmth.

N.B.: pmth position is badly reproducible

13. Optimize the spot with small CCD (if needed):

<u>For beam position:</u> tweak miroty, ty (in ML) (see also p. 23 in the main manual), or miroty, mihexy/mchi (in Torus) (see also p.31 of the main manual).

<u>For beam focus:</u> tweak mihexz, mirotz and hfmb/hfmb1/hfmb2 (in ML) (see also p. 27 in the main manual), or mirotz, mihexz (in Torus) (see also

p. 33 of the main manual).

14. Check to see if there's a signal in Ione, if so great! Run the almomi () macro. If not, one needs to optimize the beam position through the I1 slits. This can be done by following the last optimization produced given on p. 21/29/34 of the main manual. Remember to place the kapton foils when working on the (11 11 11) and (12 12 12) configuration.

15. CHECKS: tthsetup (REMEMBER with **tth≠0**), beam check routines, th motor value – adjust it according to ty (with **tth≠0**).

2. STANDARD ALIGNMENT PROCEDURE

A) Prepare Premono for slits alignment

FOURC (u32a,u32b,u32c) or (u176a,u176b,u176c) or IDappli → ID control

Close undulators to their correct value for the Si(n,n,n) reflection.

In October 2012, the undulator device server was updated. As a consequence, the motors were renamed, and the "u32tap" pseudo-motor was replaced by pseudo-motors for each magnetic array. Furthermore, the nomenclature changed:
u32u -> u32a; u32m -> u32b; u32d -> u32c

Nota bene that for the silicon (9,9,9) reflection the short period (17.6 mm) undulators have to be used

The table below only provides approximate values, the undulator gaps should be moved to the latest reference positions and checked by short scans (see below)!

	Mono	**u32c u176c**	**u32b u176b**	**u32a u176a**
n=8	7.0414	14.06	14.00	14.12
n=9	6.2398	11.11	11.37	11.34
n=11	5.0751	17.81	17.89	17.92
		12.425	12.28	12.33
n=12	4.6403	13.1	13.08	13.16
n=13	4.2546	13.868	13.883	13.9*

* only estimated values, correct values need to be checked by large scans.

Undulators can be moved either via the ID application or within FOURC

1) ID application
   - Open a window on leonov and type: jidappli
   - Go to the "View"menu and put the last known reference positions for the undulator gaps
   - Type **Reconfig** in FOURC. Check the tthsetup if ML mode was changed (with tth≠0)

- Put the Be collimating lenses (see p.6-7) and the mono to the correct positions (see last known reference positions and don't forget to **close the front** end before changing the Be lenses)
- Scan the undulators (±0.1) with pmoni

2) In FOURC

FOURC: ID_switch_undulator <undulator mnemonic>:
allows to switch between the magnetic arrays of a given undulator

FOURC: wid ! shows gap of active undulators
FOURC: ID_show ! shows status of undulators and specifies which ones are active (ON).

Scan premono in order to locate maximum of harmonics.
FOURC: plotselect pmoni
FOURC: dscan mono –0.15 0.15 30 1
Put mono to maximum of harmonics.

Scan pi1-motor and put it to maximum intensity.
FOURC: dscan pi1 –0.5 0.5 20 1
Based on the previous mono scan, move premono to 2/3 of the intensity maximum on the steep side of the harmonics (towards smaller Bragg angles).Keep mono to 2/3 for the scans of the primary/secondary slits and of the Be lenses

Nota bene: If the pmoni detector shows a constant value of ~106, or after a power cut, the gain of the femto amplifier needs to be reduced:
femto_gain fpmoni 1exx (e.g.: femto_gain fpmoni 1e08)
For more details about the femto amplifiers and their control, see Femto current amplifiers

B) Alignment of primary and secondary slits.

Plot initial settings for later comparison
Macro command for the primary/secondary slits alignment: **slitscan ()**

SLITS > umv pvg 0.2 phg 4; umv svg 4; umv shg 8

1) plotselect pmoni
2) dscan pvo –1 1 40 1; put pvo to its COM-value.
3) umv phg 0.2; umv pvg 0.6
4) dscan pho –2 2 40 1; put pho to its COM-value.
5) umv svg 0.2; umv phg 4; umv pvg 4
   (always close at least one slit to avoid excess heat load)
6) dscan svo –1 1 40 1; put svo to its COM-value.
7) umv shg 0.2; umv svg 1.0
8) dscan sho –2 2 40 1; put sho to its COM-value.
9) umv shg 2.4
10) umv pvg 0.6 phg 1.6

Nota bene:
There are two settings of the slits, depending whether the postmono is in or out.

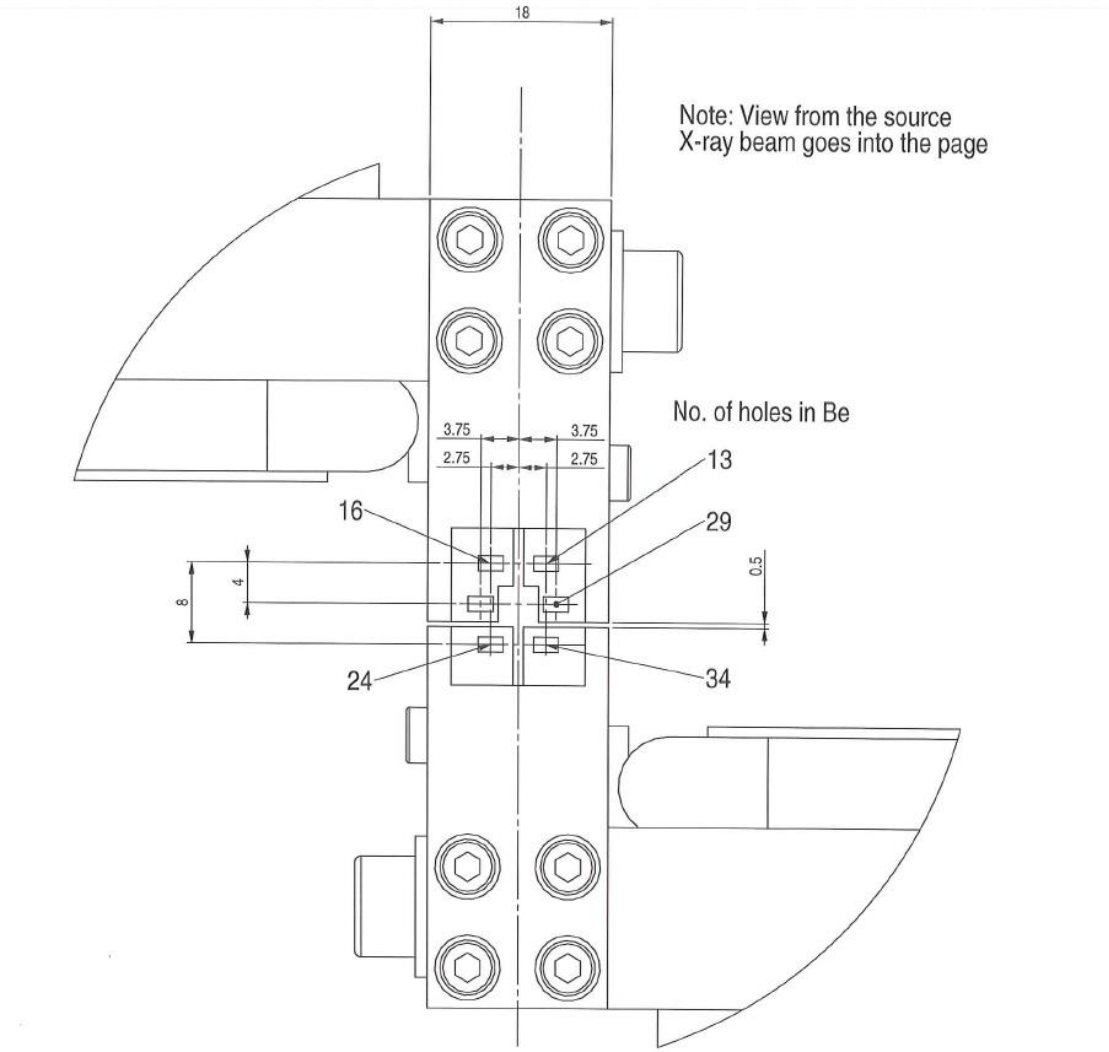
	phg	**pvg**	**shg**	**svg**
Postmono out	1.6	0.6	2.4	1.0
Postmono in	2.2	1.0	2.6	1.2

In comparing these newly determined values for pvo, pho, svo and sho with the initial ones, the angular movement in the horizontal and vertical plane (under the assumption that the source point did not move) can be determined:

$\Delta sy' = \Delta pho/27000$ mm; $\Delta sz' = \Delta pvo/27000$ mm (for primary slits)
$\Delta sy' = \Delta sho/49000$ mm; $\Delta sz' = \Delta svo/49000$ mm (for secondary slits)

pho, sho in positive direction: towards ring tunnel
pvo, svo in positive direction: upwards

C) Collimating lens



The lenses are primarily used in conjunction with the post-monochromator. It is recommended to close the front end during the initial positioning of the lens in order to avoid overheating of the collimating lens unit. The indicated positions are only approximate and need to be refined each time. It is further advisable to check the primary slits beforehand.

	n=8	**n=9**	**n=11**	**n=12**	**n=13**	**out**
cly	2.75	-2.75	-2.75	+3.75	+2.75	0
clz	-4.0	-4.0	+4.0	0	+4.0	0

1) **CLOSE THE FRONT END** before moving the lenses
2) Move cly, clz according to the previous table
3) Close the primary slits gaps umv pvg 0.3 phg 0.5
4) open the front end
5) plotselect pmoni
6) dscan clz -1 1 40 1
7) umv clz CEN
8) dscan cly -1 1 40 1
9) umv cly CEN
10) dscan clz -0.4 0.4 20 1
11) umv clz CEN
12) Reopen the primary slits gaps: umv pvg 1 phg 2.2
13) Put mono back to the MAX (see p.4)

If the scan is done too large, it can happen that the front end closes due to the temperature interlock of the collimating lenses (thus preventing overheating). For resetting the system you have to type in the spec application *slits*:

intlck reset wcid28a

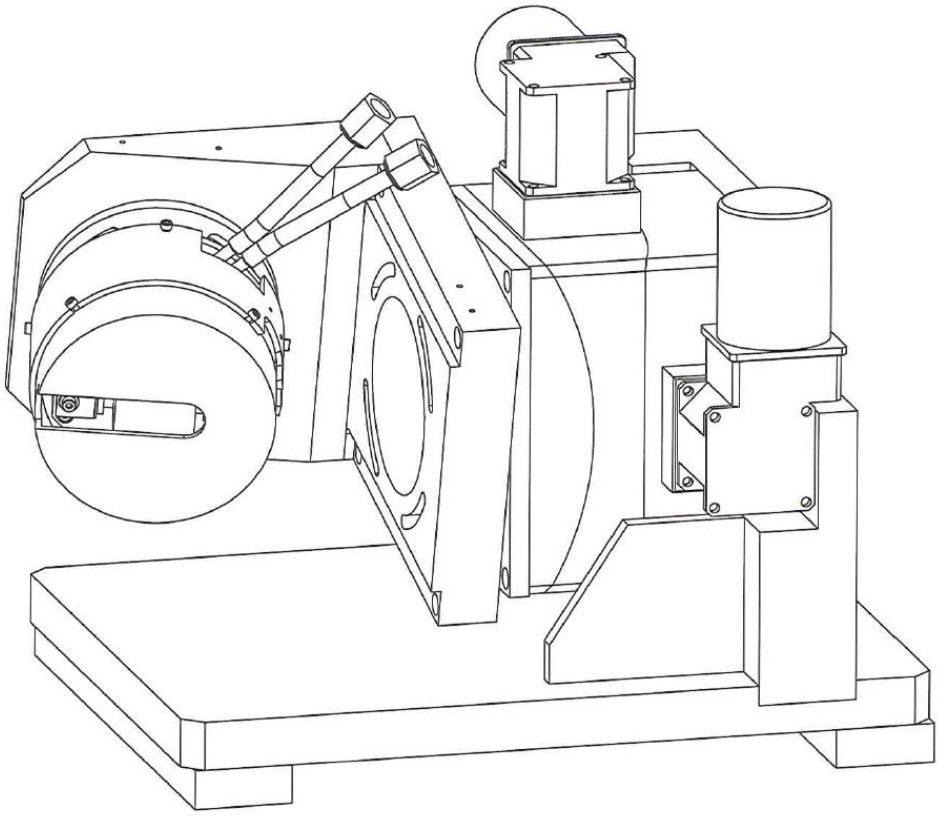
Note: curs (+/-) could be a useful command here to select cly/clz.

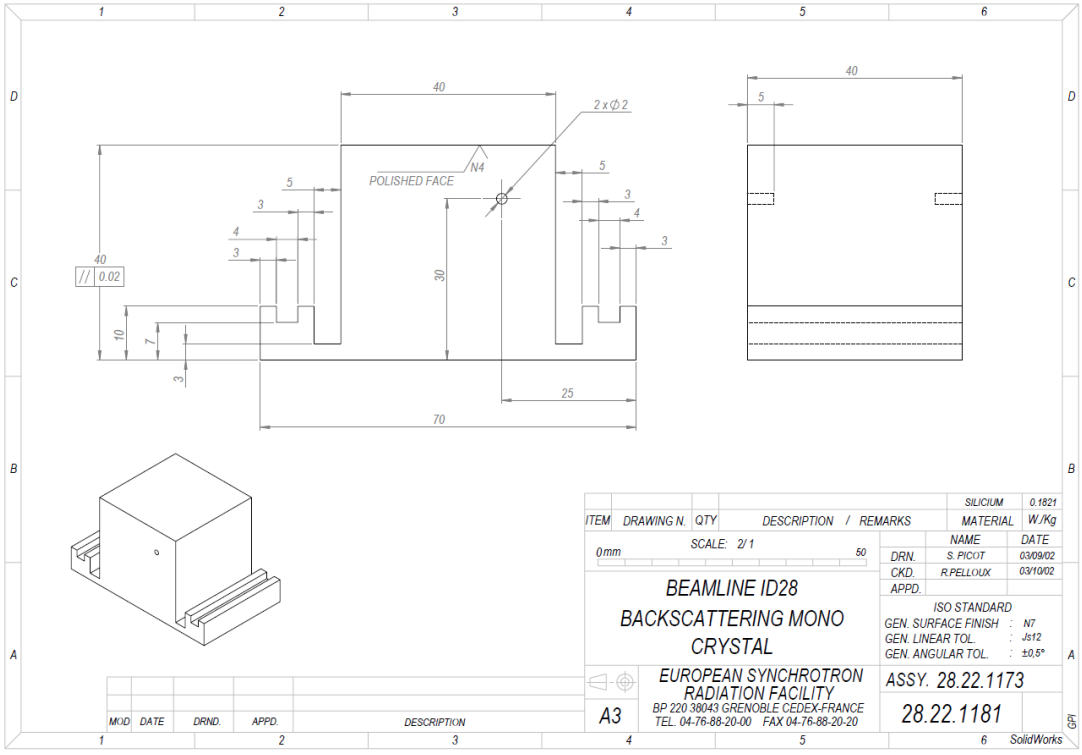
D) Correction of Premono position

If there is a significant difference in the beam position, the position of the premono should be corrected via the hexapode (spec program hx1). The approximate correction values to be employed are:
Δy (mohexy) = Δsy'* 51000 mm
Δz (mohexz) = Δsz'* 51000 mm

hexy in positive direction: towards ring tunnel
hexz in positive direction: upwards

E) Alignment of the main mono, unfocused beam





Reflection order	Reflectivity
7 7 7	0.81
888	0.85
999	0.75
11 11 11	0.69
12 12 12	0.73
13 13 13	0.59

This is the procedure after a long shutdown or a modification on the main mono itself. In this case, it is attempted to first put the unfocused beam through the standard alignment pinhole.

If there has been a large angular movement in the horizontal or vertical position (as determined after the (re-)alignment of the primary and secondary slits), one should correct the main mono height and its sideways position so that the x-ray beam is nicely centred on the asymmetrically cut main mono.

Δz (zmono) = Δsz' 73000 mm*
zmono in positive direction: upwards

Δy (ymono) = Δsy' 73000 mm*
ymono in positive direction: towards ring tunnel

1) Put premono to its nominal angular position for the desired reflection.
2) Open the motorized Huber slits and the Ione slit unit to 9 mm x 9 mm.
3) Open the horizontal slits in front of the main mono:
   umv mxgap 12
4) Find reflection with the mono screen (mscr roughly around 42.0 mm).
   This value for mscr is only approximative. The important point is to position the screen such that the "premono"-beam passes through the slot.
5) Eventually you have to slightly tweak the premono to find/maximize the reflection.
6) Steer the backscattered beam just above the slot in the screen.
7) Put mscr out of the beam: umv mscr 2
8) Move big fluo screen into the beam:
   FOURC> fluorins 1
   and move mchi until you see beam on screen.
9) Optimize mchi by determining the two positions where the

reflected beam is cut by the various Be windows, and put mchi to the center between these two previously determined values.
At the end of the procedure, move the big fluo screen out.
FOURC> fluorext 1

10) Installation of the Huber standard alignment pinhole with pin-diode detector behind. (sax = 0; say = +0.1; saz = 0; th=0; chi=0; phi=0)
11) Move tth to an uncritical value, since the pin diode is almost transparent for x-rays.
12) Center beam through alignment pinhole with mth and mchi.

The resulting absolute Bragg angle should is dependent on the reflection order and whether the postmonochromator is in the beam or not. The following table gives a summary of the corresponding angles:
mth(unfocused)
umvr mth
In the focused position the Bragg angle is always 89.98 degrees.

i) configuration without postmonochromator

	n=8	**n=9**	**n=11**	**n=12**
mth [deg]	89.8880	89.8880	89.8881	89.8881
umvr mth* [deg]	0.1320	0.1320	0.1320	0.1319

i) configuration with postmonochromator

	n=8	**n=9**	**n=11**	**n=12**
mth [deg]	89.8993	89.8995	89.8997	89.8997
umvr* mth [deg]	0.1206	0.1205	0.1203	0.1203

*refers to relative movement of mth with respect to the motor position in the focused beam configuration.

F) Alignment of main mono in focused beam position

If there has been no major intervention, it is not necessary to realign the main mono in the unfocused beam, but one has to correct for the eventual changes in the angular position of the x-ray beam.

If there has been a large angular movement in the horizontal or vertical position (as determined after the (re-)alignment of the primary and secondary slits), one should correct the main mono height so that the x-ray beam is nicely centred on the asymmetrically cut main mono.

Δz (zmono) = Δsz'* 73000 mm
zmono in positive direction: upwards

Δy (ymono) = Δsy'* 73000 mm
ymono in positive direction: towards ring tunnel

To double-check a scan in ymono should be done. zmono is less critical, since the crystal is 40 mm high.

Nota bene! If you move either zmono or ymono an alignment of the main monochromator in the unfocused beam position is needed !!! This can be done after having done a rough alignment in the focussed beam.

Make sure that mxgap is large open, for example 20 mm in order to avoid that the beam is cut by the slit blades!

1) plotselect imirr
2) dscan ymono –7 0 35 1

Determine the position where ½ of the total intensity is lost. From this position (ymono[pos]), you have to move relative by + (37.32*sin(15))/2. *37.32 corresponds to the length of the flat part of the main monochromator, and 15 degrees is the angle of incidence of the X-rays.*

3) umv ymono ymono[pos] + 4.83

4) type hexaoff in fourc to avoid interference with the hexapode spec versions.
5) If there has been a large angular movement in the horizontal or vertical position (as determined after the (re-)alignment of the primary and secondary slits), one should correct the mirror hexapode positions mihexz and mihexy.

6) Correction of mirror position with mirror hexapode (spec program hx2):
   Δhexy = Δsy' * 69000 mm
   Δmihexz = Δsz' * 69000 mm

   mihexy in positive direction: towards ring tunnel
   mihexz in positive direction: upwards

In order to fine position the mirror height, perform a scan with mihexz:

7) plotselect imirr
8) dscan mihexz –2 2 40 1 (the FWHM of the profile should be about 2.9 mm)
9) umv mihexz to the center of the profile
10) dscan miroty –0.02 0.02 40 1
11) umv miroty to the center of the profile
12) repeat steps 5-8 until there is no longer a shift of the center value.

This iterative procedure is necessary, since a wrong initial miroty position will give a wrong center in mihexz. Watch out when using imirr. For poor alignment the beam will hit the aluminum frame which will give a stronger signal than the scattering from the Kapton foil,. In case of doubt, use izero.

Align focused beam through standard alignment pinhole with mchi and miroty. This can be done either with deta2 and tth at about 0 degrees (this implies that the spectrometer is more or less correctly aligned), or with the newdet detector. The procedure below refers to the alignment with the newdet pinhole detector.
Before mounting the pinhole, check the exact tth-position for the newdet detector.

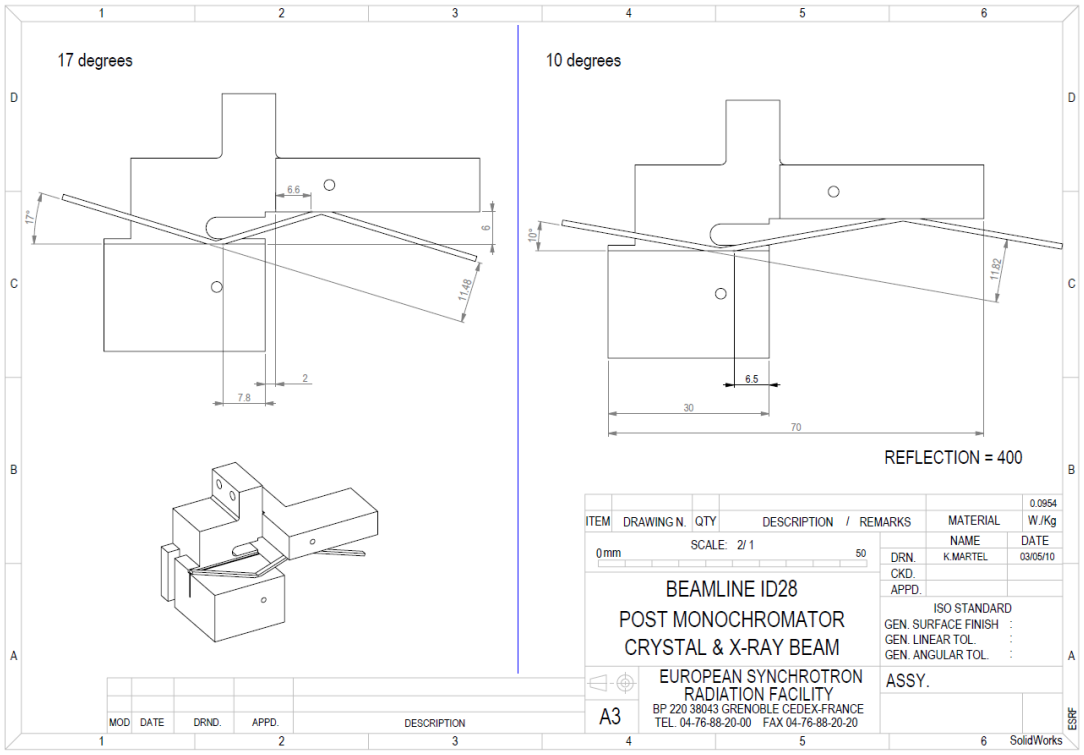
13) umv tth 0.6; znewdet -55 (znewdet will hit the limit before, but this is OK)
14) plotselect newdet
15) dscan tth 0.1 -0.1 40 1 center tth on newdet detector
16) umv sax 0 saz 0 say 0.1 th 0 chi 0 phi 0

    Mount standard alignment pinhole

make small tweaks and scans with mihexy and miroty in order to optimise the signal through the pinhole.

G) Postmonochromator

The insertion of the postmono leads to a slight change of the beam height at the analysers of -2.7 mm (-2.9 mm) at 6.5 m (7m). This is a marginally small value compared to the Q-resolution of the spectrometer.



Si (4 0 0); $\Delta E/E = 2.53 \times 10^{-5}$

reflection order	Bragg angle [deg]	Darwin width [μrad]
8	16.78	7.63
9	14.868	6.72
11	12.119	5.43
12	11.096	4.96
13	10.217	4.56

n	ω_D (theo) [deg]	ω_D (exp) [deg]	Refl (exp)
8	4.37×10^{-4}		
9	3.85×10^{-4}	4.2×10^{-4}	0.85
11	3.11×10^{-4}	2.6×10^{-4}	0.88
12	2.84×10^{-4}	2.9×10^{-4}	0.99 ???
13	2.61×10^{-4}		

The starting point is typically that the reflection of the main backscattering mono has been found already, so that we know the correct angle of "mono".

The first step is to call the collimating lens routine to put the correct lens in. This lens is needed in order that the transmission of the postmono is OK (Its spectral angular acceptance is significantly smaller than the vertical beam divergence, so we would lose photons, if we do not use the coll. lens.)

a) execute collimating lens routine (see p. 6-7)
b) plotselect pmoni
c) dscan pi1 -0.5 0.5 20 1
d) plotselect imirr
e) dscan mono -0.0015 0.0015 20 3
f) umv mono CEN (3-4x)
g) move zmono to the correct position for the postmono-configuration. This needs to be done step-by-step (1 mm at the time, and follow the backscattered beam either with Imirr or the small alignment CCD. This procedure allows to put the main mono already at the correct height without losing the beam.
h) Put postmono into the beam, and correct the other optics and motor positions accordingly (see table below)

		n=8	**n=9**	**n=11**	**n=12**	**n=13(old)**	**out**
Mono	abs	7.0413	6.2398	5.0751	4.64	4.2546	
pmscr2	abs	38.5	38.5	38.5	38.5	38.5	2
pmy	abs	0	0	0	0	0	10
pmz	abs	1.0	1.25	1.8	2.0		-
pmchi	abs	0.75	0.25	0.25	0.25		
pmth	abs	17.084	15.156	12.385	11.353		
zmono	relative	11.489	11.598	11.733	11.776	11.810	-
mihexz	relative	11.489	11.598	11.733	11.776	11.810	-
miroty	relative	0.254	0.254	0.254	0.254	0.254	-
ratio mono/ pomono		2.4878		2.4734	2.48		

As the beam position changes from run to run the movements of zmono, mihexz, and miroty are relative movements !!!! It is furthermore advisable to check the correct positions of the undulators. The values for the postmono angles are only approximate, and the reflection must be found each time again! Furthermore it is advisable to check pmz, using pomoni and verifying with Imirr that indeed the flux from the main backscattering monochromator is optimised.

Nota bene: Usually, zmono is not corrected. If you think pmchi needs to be checked, keep in mind, that it does not move reproducibly! pmono2, the weak link between the two reflecting surfaces of the postmono crystal, is usually OK, should only be checked, if the flux after the main mono (imirr) is too low.

Optimise postmono with respect to premono

i) umv pi2 0
j) plotselect pomoni
k) dscan pmth 0.04 -0.04 80 1. Count after every pmth scan to check whether there is signal.
l) umv pmth CEN
m) dscan pmth 0.003 -0.003 30 1
n) dscan pmchi -0.15 0.15 30 1 (**not reproducible**)
o) umv pmchi CEN
p) dscan pmth -0.003 0.003 30 1
q) umv pmth CEN
r) dscan pmz -0.5 0.5 20 1
s) put pmz to the optimum position (flat part of the profile)
t) dscan pmono2
u) umv pmono2 CEN

This should be already sufficient to have some signal on the Imirr detector. The fine tuning needs to be done iteratively.

Optimise premono and postmono with respect to main backscattering mono

v) plotselect imirr
w) dscan pmth 0.003 -0.003 30 1
x) umv pmth CEN
y) dscan mono -0.004 0.004 40 1
z) umv mono CEN (3-5 times)
aa) dscan pi2 -0.4 0.4 40 1
bb) umv pi2 CEN

cc) dscan mono -0.001 0.001 20 1
dd) umv mono CEN (3-5 times)

Note: In order to get signal in Imirr, it is often more useful to do a combined d2scan:

d2scan mono -0.005 0.005 pmth -0.012 0.012 80 1

(and then pi2: dscan pi2 -0.4 0.4 40 1; umv pi2 CEN)

Optimise beam position through I1 slits

ee) plotselect ione
ff) umv i1svg 0.2 i1shg 0.5
gg) dscan miroty -0.02 0.02 40 3
hh) umv miroty CEN
ii) dscan i1sho -0.5 0.5 25 3
jj) Make a warning, if the CEN value is off by more than 100 microns with respect to the initial value of i1sho.

Standard i1svg, i1shg working positions:

	Multilayer	**Torus**
I1shg	0.9	0.5
I1svg	0.1	0.2

H) Multilayer alignment

The (horizontally focusing) multilayer and the (vertically focusing) mirror are now in the SPEC programme KB; motors have been renamed:

hfmb1, hfmb2, hfmb:	multilayer bender motors, hfmb moves both motors simultaneously. + larger bending radius
hfmth:	multilayer theta - positive angles
hfmtz:	real motor, used to make y movement
hfmz1, hfmz2, hfmz:	z-motion, hfmz moves both motors simultaneously + upwards
hfmtx:	tilt around x-axis (chi) by opposite movement of hfmz1 and hfmz2.
hfmy:	horizontal movement of multilayer + towards EXPH
hfmpi:	HFM piezo for fine tuning of multilayer angle
vfmb1, vfmb2, vfmb:	mirror bender motors, vfmb moves both motors simultaneously. + larger bending radius
vfmth:	mirror theta - positive angles
hfmtz:	real motor, used to make y movement
vfmz1, vfmz2, vfmz:	z-motion, vfmz moves both motors simultaneously + upwards
vfmpi:	VFM piezo for fine tuning of mirror angle
vfmmoni:	y-translation of scattering foil for monitor detector between VFM and HFM.
minisl:	100 μm vertical slit; 5 out of the beam, approx. working position: 22.667 torus configuration 27.555 multilayer configuration 13.67 KB configuration
moniy:	y-translation of slit-ion chamber unit - towards EXPH

mlleft, mlright:	horizontal slits in front of the VFM and HFM
mloff, mlgap:	gap and offset of these slits

Nota bene: the piezos are driven by the wago box. Range of voltage from 0 to 10 V.
!!! Make sure that piezo is at its default value of 5 before the alignment.

moniy problem: issue of non-reproducibility

It seems that only during the first movements steps are lost, but then things are OK. One can always recover the situation by renaming the motor position after a check of the upper hardware limits. This should be: upper limit: +20.76

Note: the problem shall soon be fixed. We bought a new stronger motor, which should avoid the above problem.

Prior to changing to multilayer configuration, check correct beam position with standard (50 µm) alignment pinhole.

Alignment pinholes:
OLD: 100*100 µm and say 0.1
NEW: 50*50 µm and say 0

umv tth 0.60 (more precisely:actual position of newdet in direct beam)
umv sax 0; umv saz 0; umv say 0.1; umv th 0; umv chi 0; umv phi 0
plotselect newdet
dscan say 0.5 -0.5 25 1
dscan saz –0.3 0.3 30 1

or

Check the position of the spot with the small bassler camera. Put the camera on the goniometer, then:
umv sax 0; say 0, saz 0 chi 0

The spot should then be visible at the reference X- and Y positions.
See note above beamline control computer for most recent value.

If say is off by more than +/- 0.05 mm, the beam position has to be corrected by mihexy/ty!
If saz is off by more than +/- 0.05 mm, the beam position has to be corrected by miroty!

Check the horizontal beam position in front of the multilayer

1) umv mlgap 1
2) plotselect imirr
3) dscan mloff –2 2 40 1
4) center mloff
5) umv mlgap 2

Multilayer should be by default parallel to the incident beam direction. Check parallelism and approximate transverse position of multilayer.

6) umv i1shg 0.1; umv i1svg 1; plotselect ione
7) ascan hfmy –1.5 0 30 1
8) umv hfmy to ½ height on the right side of the profile
9) dscan hfmth –0.2 0.2 40 1
10) umv hfmth to center of profile

Refinement after optimization of hfmth

11) dscan hfmy –0.3 0.3 30 1
12) umv hfmy to ½ height on the right side of the profile
13) dscan hfmth –0.05 0.05 40 1

Move manually the x-ray mirror sideways to the cylindrical position and correct for the height in HX2 (close the beam shutter)

14) hexaon
15) umvr mihexz –2.0

Nota bene! The exact cylindrical radius of the mirror depends on the configuration (torus, cylinder, use of collimating lens). The following table indicates the correct values to which *bender* needs to be moved in *HX2*.

	Torus w/o lens	**Torus with lens**	**Cylinder w/o lens**	**Cylinder with lens**
bender	237	147.5	279.5	187

umv bender to the appropriate value according to the previous table
dscan mihexz -2 2 30 1

Set to correct multilayer modus in FOURC with tthsetup !!!!
Move tth to a save value (tth≠0), i.e. 3 degrees

FOURC> tthsetup (opens a little menu offering several choices)

"0" or "Out of beam" to calculate tth without taking into account the ML.
"1" or "888" tth with the ML in the Si (8,8,8) configuration.
"2" or "999" tth with the ML in the Si (9,9,9) configuration.
"3" or "111111" tth with the ML in the Si(11,11,11) configuration.
"4" or "121212" tth with the ML in the Si(12,12,12) configuration.

Or, shorter: tthsetup + Parameter +{return}
tthsetup 0, tthsetup Out of beam
tthsetup 888, tthsetup 1
tthsetup 999, tthsetup 2
tthsetup 111111, tthsetup 3
tthsetup 121212, tthsetup 4

Any other parameter value/text with either not change anything if **tthsetup** is used in the short version, or will just repeat the little menu, until a correct value/answer is given.
CTRL-C in **tthsetup** will leave the settings unchanged.

(ii) Once **tthsetup** is set, wa or every wm of any motor will give an additional line of comment indicating the present tthsetup-setting.
(iii) The first use of **tthsetup** is in the start-up of fourc or in the setup/resetup/config/reconfig within fourc. By default the setting chosen at the most recent closing of fourc (CTRL-D) is used then. In the worst case (e.g. after a severe crash), the default-setting is "0", assuming, the ML is not used.
(iv) Once **tthsetup** is set, each scan in the SPEC-datafile will have a comment line "#C …." Indicating the chosen setting.
(v) As it is programmed so far, the real motor **tthm** cannot be scanned/moved directly!!!, since the value of the pseudo-motor **tth** is re-calculated when **tthm** is changed, but with **tth** changed, tthm is again re-calculated etc….
(vi) For BLISS/ID28: The macro containing the tthsetup and the tth/tthm calculations is in the file "tth_multiplayer.mac".

Move goniometer tower to the correct positions for the chosen multilayer configuration

16) umv wheel 315
17) umv th "$2*\theta_{ML}$" (see table in the next page)
18) set th 0 (do not change the dial value of th)
19) umv ty "multilayer value" (see table in the next page)

Correction of the slit unit offsets

20) umv moniy “multilayer value”, umvr hfmth “multilayer value”
21) umv i1sho “multilayer value”
22) umv i1shg 3.0
23) umv i1svg 3.0

n	θ_{ML} [°]	umvr hfmth [°]	ty [mm]	moniy [mm]	th [°]
ML out	-	-	-	0	0
8	0.89116	-0.89116	47.787	-35.62	1.7823
9	0.7921	-0.7921	42.40	-31.59	1.5842
11	0.6481	-0.6481	34.51	-25.85	1.2962
12	0.6056	-0.6056	31.31	-23.76	1.2112

N.B. The hard limit of ty is at -2.402

Approximate real motor positions; these should be used when changing configuration

n	I1h [mm]	I1v [mm]	I2h [mm]	I2v [mm]	i1sho [mm]	i1svo [mm]	i1shg [mm]	i1svg [mm]
no ML	+0.6	-0.047	+3.0	+0.46	-0.065	+0.19	0.5	0.2
8 (ML)	-4.59	+3.23	+0.56	+3.04	-0.12	+2.38		
8 (KB)	-4.6	+3.28	+1.07	+2.99	+0.43	+2.38	1.1	0.1
9 ML)	-3.52	-0.047	+0.67	+0.46	0	+0.26		
9 (KB)	-3.52	+2.89	+0.60	+3.04	0	+2.44		
11 (ML)	-2.83	-0.047	+1.51	+0.46	+0.3	+0.24	0.9	
12 (ML)								
12 (KB)	-3.01	+3.22	+1.28	+3.04	0			

Check beam position with mloff-scan

24) plotselect imirr
25) umv mlgap 1
26) dscan mloff –4 4 20 1

If mloff is off with respect to "toroidal" position, correct with mchi-scan until the mloff-value is OK.

27) dscan mchi –0.003 0.003 30 1
28) umv mchi CEN
29) umv mlgap 4

Rocking curve of multilayer

30) umv huxg 5; umv i1shg 5; umv huzg 3; umv i1svg 3
31) plotselect izero
32) dscan hfmth –0.02 0.02 40 1; umv hfmth CEN
33) Drive beam to appropriate pixel position of small bassler camera with ty and mihexz/miroty

Sometimes it might be useful to make a mihexz-scan to check whether the beam is approximately centred on the mirror.

Plotselect imirr; dscan mihexz -1.5 1.5 30

Check and optimise focus with small Bassler camera:

34) Cylindrical mirror (in HX2): "bender" is not particularly sensitive, first attempt to optimise should be done with "mihexz" (typical range +/- 0.3 mm), and then "mirotz" (typical range +/- 0.03). If improvement is visible, a few iterations might be needed.
35) Multilayer (in MIRROR): for coarse improvement of the focus, user "hfmb" (±0.05), for fine tuning "hfmb1" and "hfmb2" need to be moved seperately until the best focus is achieved.

Recheck rocking curve of multilayer:
dscan hfmth ±0.02

Check direct beam position on deta2

36) take out the bassler camera (or the alignment pinhole if used instead)

37) umv wheel 270 (135;225;315)
38) plotselect deta2
39) umv a2hgap 5
40) umv tth 0
41) dscan tth 0.08 –0.08 40 1
42) umv tth CEN
43) umv a2hgap 20

Mount standard alignment pinhole and check beam position at sample

44) plotselect deta2
45) umv wheel 135 (or thicker)
46) umv sax 0; umv saz 0; umv say 0;
47) umv th "multilayer value"; umv chi 0; umv phi 0
48) dscan ty 0.15 -0.15 30 1; umv ty CEN
49) dscan saz –0.2 0.2 40 1

If saz is off by more than +/- 0.05 mm, the beam position has to be corrected by miroty!

Refine tth = 0 position

50) umv wheel 270 (225;315)
51) plotselect deta2
52) umv a2hgap 5
53) umv tth 0
54) dscan tth 0.08 –0.08 40 1
55) umv tth CEN
56) umv a2hgap 20

If there is a significant offset, the angular scale of tth has to be corrected.

57) umv tth 0
58) note down value of tthm
59) umv tth (determined center value)
60) set tthm (value determined in step 59)
61) wm tth (should be at 0 after step 61)
62) umv a2hgap 20

The reason for this non-straightforward procedure is that tth is a pseudo-motor, involving a mathematical expression to convert the physical position of the real motor tthm (motor driving the lead screw) into the value for the scattering angle. If an angular correction is needed, one has therefore to act on the real motor tthm.

Check slit positions

63) umv huxg 2; umv huzg 2
64) plotselect izero
65) dscan huxo –2 2 20 1
66) umv huxo CEN
67) dscan huzo –2 2 20 1
68) umv huzo CEN

N.B. Normally we never move the Izero slits

69) umv i1shg 0.9
70) plotselect ione
71) dscan i1sho –1 1 20 1
72) umv i1sho CEN
73) umv i1svg 0.1
74) dscan i1svo –0.3 0.3 30 1
75) umv i1svo CEN
76) put i1svg to appropriate opening for the experiment (umv i1shg 0.9, i1svg 0.1 for ML)

Find the beam reference position with the PRL

- Put the PRL in.
- Find the pinhole position by moving the PRL and keep this as a reference beam position.

Remove the pinhole and the PRL and mount the plexiglass. Continue with the alignment of the spectrometer analyzers as in page 34.

Automised Optimisation of focal size and shape

(Routine is not yet operational)

This is done within the KB spec session.

kbf_setup_limits <hor gap> <hor offset> <vert gap> <vert offset>
kbf_focus <bender increment> <hor/vert> <one, both> <rel scan start> <rel scan stop> <# intervalls> <counting time>

Typical values are:

kbf_setup_limits 0.1 1 0 49
kbf_focus 0.005 hor both -1.5 1.5 40 0.0003
kbf_focus 0.005 vert both -0.3 0.3 40 0.0003

I) Switching from Multilayer to Torus configuration

Prior to changing to torus configuration, check correct beam position with standard (50 μm) alignment pinhole.

FOURC > umv tth 0
FOURC > umv sax 0 saz 0 say 0.1; umv th 0 chi 0 phi 0
FOURC > umv wheel 180 (or other appropriate value)
FOURC > plotselect deta2
FOURC > dscan say 0.1 -0.1 20 1
FOURC > dscan saz –0.3 0.3 30 1

or

Check the position of the spot with the small bassler camera. Put the camera on the goniometer, then:
FOURC > umv sax 0; say 0, saz 0 chi 0

The spot should then be visible at the reference X- and Y positions.

If say is off by more than +/- 0.05 mm, the beam position has to be corrected by mchi!
If saz is off by more than +/- 0.05 mm, the beam position has to be corrected by miroty!

Move to the values (relative or absolute) as indicated in the table below.
This puts the multilayer out and back parallel to the incident beam.

n	θ_{ML} [°]	umvr hfmth [°]	ty [mm]	moniy [mm]	hfmy [mm]	i1sho [mm]	umvr th [°]
8	0.89116	0.89116	0	0	-2	0	-1.7823
9	0.7921	0.7921	0	0	-2	0	-1.5842
11	0.6481	0.6481	0	0	-2	0	-1.2962
12	0.6056	0.6056	0	0	-2	0	-1.2112

Set the user value of th to 0. It should correspond to the dial zero-value as well.

Move manually the x-ray mirror to the torus position and correct for the height in HX2

77) *FOURC* > hexaon

78) *FOURC* > umvr mihexz 2.0

Nota bene! The exact cylindrical radius of the mirror depends on the configuration (torus, cylinder, use of collimating lens). The following table indicates the correct values to which *bender* needs to be moved in *HX2*.

	Torus w/o lens	**Torus with lens**	**Cylinder w/o lens**	**Cylinder with lens**
bender	237	147.5	279.5	187

Set to torus modus in FOURC with tthsetup !!!!

Move tth to a save value, i.e. 3 degrees

FOURC> tthsetup (opens a little menu offering several choices)

"0" or "Out of beam" to calculate tth without taking into account the ML.

Or, shorter: tthsetup + Parameter +{return}
tthsetup 0, tthsetup Out of beam

Any other parameter value/text with either not change anything if **tthsetup** is used in the short version, or will just repeat the little menu, until a correct value/answer is given.
CTRL-C in **tthsetup** will leave the settings unchanged.

(vii) Once **tthsetup** is set, wa or every wm of any motor will give an additional line of comment indicating the present tthsetup-setting.

(viii) The first use of **tthsetup** is in the start-up of fourc or in the setup/resetup/config/reconfig within fourc. By default the setting chosen at the most recent closing of fourc (CTRL-D) is used then. In the worst case (e.g. after a severe crash), the default-setting is "0", assuming, the ML is not used.

(ix) Once **tthsetup** is set, each scan in the SPEC-datafile will have a comment line "#C …." Indicating the chosen setting.

(x) As it is programmed so far, the real motor **tthm** cannot be scanned/moved directly!!!, since the value of the pseudo-motor **tth** is re-calculated when **tthm** is changed, but with **tth** changed, tthm is again re-calculated etc….

(xi) For BLISS/ID28: The macro containing the tthsetup and the tth/tthm calculations is in the file "tth_multiplayer.mac".

Open the I0 and I1 slits in order to catch the beam on the Bassler camera
N.B. The I0 slits are always kept open and normally we don't move them.

79) *FOURC* > umv huxg 8 huzg 8 i1shg 4 i1svg 4
80) make a coarse scan or manual movement of "miroty" and “ty”until you find the beam on the Bassler camera. Drive the beam on the right spot with miroty and mchi.

Once the beam is roughly at the correct position, make a scan of the mirror height to centre the beam approximately on the mirror.

81) *FOURC* > plotselect imirr
82) *FOURC* > dscan mihexz -2 2 20 1
83) *FOURC* > umv mihexz CEN

Check and optimise focus with small Bassler camera:

84) First attempt to optimise should be done with “mihexz” (typical range +/- 0.3 mm), and then “mirotz” (typical range +/- 0.03). If improvement is visible, a few iterations might be needed.

Check direct beam position on deta2

85) take out bassler camera
86) umv wheel 270 (135;225;315)
87) plotselect deta2
88) umv a2hgap 5
89) umv tth 0
90) dscan tth 0.08 –0.08 40 1
91) umv tth CEN
92) umv a2hgap 20

Mount standard alignment pinhole and check beam position at sample

93) plotselect deta2
94) umv wheel 135 (or thicker)
95) umv sax 0; umv saz 0; umv say 0;
96) umv th 0; umv chi 0; umv phi 0
97) dscan say 0.6 -0.6 30 1
98) dscan saz –0.2 0.2 40 1

If say is off by more than +/- 0.1 mm and/or saz is off by more than +/- 0.05 mm, the beam position has to be corrected by mihexy and miroty, resp.!

Refine tth = 0 position

99) umv wheel 270 (225;315)
100) plotselect deta2
101) umv a2hgap 5
102) umv tth 0
103) dscan tth 0.08 –0.08 40 1
104) umv tth CEN
105) umv a2hgap 20

If there is a significant offset, the angular scale of tth has to be corrected.

106) umv tth 0
107) note down value of tthm
108) umv tth (determined center value)
109) set tthm (value determined in step 59)
110) wm tth (should be at 0 after step 61)
111) umv a2hgap 20

The reason for this non-straightforward procedure is that tth is a pseudo-motor, involving a mathematical expression to convert the physical position of the real motor tthm (motor driving the lead screw) into the value for the scattering angle. If an angular correction is needed, one has therefore to act on the real motor tthm.

Check slit positions

N.B. The I0 slits are always kept open and normally we don't move them.

112) umv huxg 2; umv huzg 2
113) plotselect izero
114) dscan huxo –2 2 20 1
115) umv huxo CEN
116) dscan huzo –2 2 20 1
117) umv huzo CEN
118) umv i1shg 0.5
119) plotselect ione
120) dscan i1sho –1 1 20 1
121) umv i1sho CEN
122) umv i1svg 0.2
123) dscan i1svo –0.3 0.3 30 1
124) umv i1svo CEN
125) put i1svg to appropriate opening for the experiment (umv i1shg 0.5, i1svg 0.2 for torus)
126)

J) Switching from Multilayer to KB configuration

- The starting point is a fully optimised optics/setup in the multilayer mode.
- While still in the multilayer configuration, mount the bassler camera and record the X, Y positions as a reference.

Further elements of importance are:

1. Minislit

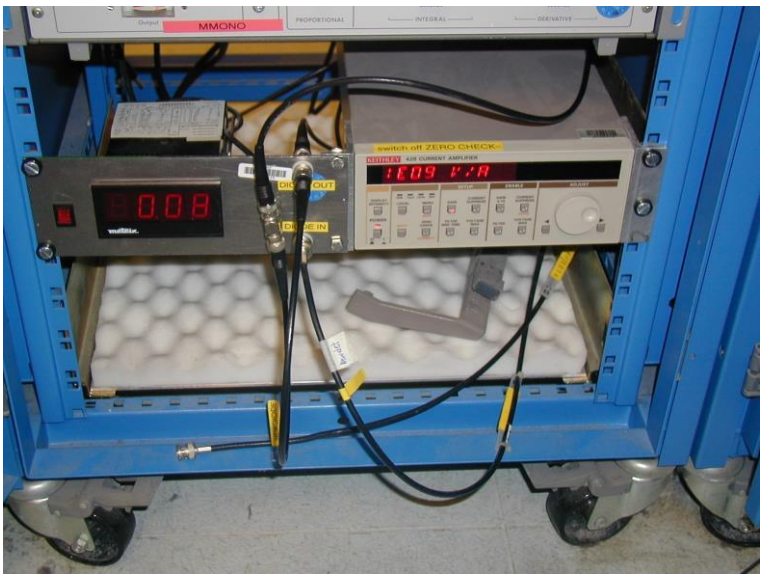
The minislit is located in front of the vertically focusing mirror (behind the mlgap and the Imirr monitor) and consists of a 100 μm fixed vertical slit. It shall be used for the automatic beamfocusing procedure. Since moving mth is non reproducible, we tweak mth and use minislit scans until we get the beam in the right position (centered in the minislit scan). The minislit is positioned about 25.64 m away from the main mono (3.36 m in front of the sample position).

Approximate minislit positions:

ML optics: 27.5 mm
KB optics: 13.72 mm (in full KB configuration)
OFF the beam: 2 mm

2. Alignment diode

This diode is installed underneath analyzer 2 and slightly off in angle. In order to activate it, you have to swap the indicated newdet and newdiode cables (see photo below) in the Keithley.



Move the newdet and the bassler camera so that the beam hits the newdiode (newdet is in/out for znewdet -49/5 respectively).
tth diode position: +0.477 degrees
umv tth 0.477
Check for signal in the newdiode. If not, then scan tth.
Nota bene!!! If you determine another value for the tth diode position, you need to reset tthm and tth such that at the intensity profile of newdet/newdiode is nicely centered at 0.477 degrees.

Positioning the beam at the correct height

In the KB console:
1) plotselect imirr
2) umv minisl 27.5 (nominal position in the ML configuration)
3) dscan minisl -0.2 0.2 40 1
4) umv minisl CEN (keep this as a reference position to get back to when you switch back to the ML configuration)
5) umv minisl 13.72 (nominal position in the KB configuration)

6) Manual intervention in EH1: move manually Imirr Kapton foil to position 35.5 which is the nominal position for the KB configuration (the nominal position of the Imirr Kapton foil for the ML and Torus configuration is 17.5)
7) umvr mth 0.133
8) dscan mth -0.004 0.004 40 1
N.B.1: Since mth is very non reproducible, instead of scanning mth it is preferable to tweak mth (+/- raises/lowers the beam respectively, e.g. ±0.0002) and then dscan minisl -0.4 0.4 20 1. Tweak mth until the beam is nicely centered on the minislit. We expect more than 1000 ct/s on Imirr.

Once mth is optimised, the mchi position needs to be checked as well. To this end a mloff scan (dscan mloff -2 2 20 1) and an eventual correction of mchi needs to be performed.
VALUE FOR mlgap FOR THE SCANS?
9) Repeat tweak/scans until you are nicely centered through the minislit (for the vertical position) and mlgap (for the horizontal position).
10) VALUE FOR minisl/mlgap FOR THE MEASUREMENTS?
umv minisl 2 mlgap 2

Optimising the vertical focusing mirror height and the angle of reflection

11) Move the quadrupole chamber motors I1h, I1v, I2h, and I2v as well as i1svo to the tabulated values for the corresponding reflection order (see p. 22 and the latest reference positions on the logbook). Put the bassler camera back in the beam position. Open the Ione slit wide in order to make sure that the beam hits the bassler camera.

12) umv hfmz 0
The movement is from the ML configuration position which is -7.5. This is the nominal position of the ML chamber. As the vertical opening of the bender is 8 mm, there is no need for a precise height positioning.

13) plotselect ione
14) dscan vfmz -1 1 40 1
15) umv vfmz CEN
16) optimise the vertical beam position by tweaking vfmth (±0.01).
17) optimise the horizontal beam position by tweaking hfmth/ty
18) optimize the beam focus on the bassler camera using hfmb/hfmb1/hfmb2 and vfmb/vfmb1/vfmb2.
19) check/optimise the nominal beam position using the standard pinhole (at sax=0, say=0, saz=0) and the pin diode at tth=0.477.

Real value of the scattering angle:

$$2\Theta = (\mathrm{tth}[^\circ]^2 + 0.397^2[^\circ])^{1/2}$$

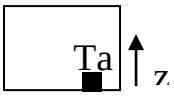
K) Switching from Conventional Torus to Torus - KB configuration

NB: the purpose of exercise is to get tight vertical focus (10-15 mkm) keeping low horizontal divergence (and width typical for torus configuration). Potentially interesting for thick films.

- Starting point: fully optimized torus configuration
- Put the CCD, move say and saz to move the beam to the center
- Unbend the mirror completely :
    - HX2: umv bender 0 #The beam dimension increases vertically !
- Verify if the beam is falls to the center of mirror:
    - FOURC: plotselect imirr; dscan mihexz -1.5 1.5 20 1; umv mihexz [good position]
    - mind the soft limits and the vertical projection of mirror of ~2.5 mm (This means the FWHM of the profile is ~2.5 mm => find the low limit and move mihexz to **edge + 1.25** mm)
- Put the minislit in (ML nominal position):
    - KB: plotselect imirr; umv minisl **27.5**; dscan minisl -0.5 0.5 20 1
    - You should get profile with few peaks but roughly centered at 27.5
- Move minislit
    - KB: umv minisl 13.72 (or better move -13.78 relative to the ML position that you found)
- Move manually imirr Kapton foil to position **35.5** (EH1)
- Move beam up by miroty
    - FOURC: umvr miroty -0.32
- Adjust the angle

- FOURC: plotselect imirr; dscan miroty -0.02 0.02 20 1

- Remove the minislit
  - KB: umv minisl **2**
- Move I1v, I2v to the reference position
  - FOURC: umv I1v [good] I2v [good] (e.g for **999: I1v = 2.89, I2v = 3.04**; see p. 22 of the manual)
- Open slits
  - FOURC: umv i1svo 2.5; umv i1svg 3 i1shg 3
- Move ML chamber out of the beam:
  - KB: umv hfmz 0
  - Bring the beam to the center of CCD by tweaking vfmth (horizontal direction on the screen).
  - Bring the beam to the center of CCD by tweaking mchi (vertical direction on the screen)
  - Mind the bad reproducibility of mchi, go with small steps (~**0.0002** degrees)
  - Optimize the focus tweaking hfmb (0.01)
- Remove CCD, put standard plexiglas with a piece of Ta in front



  - Move tth to Q = 10 nm^{-1} (for 999: tth = 6.357, check page 102 in the manual)
  - umv say 2
  - Locate the Ta piece by making a scan across the Ta edge in the z and the y directions (say and saz).
  - Put the Ta sheet in the center of rotation: umvr sax -2 (the thickness of the plexiglass + Ta sheet)
  - FOURC: plotselect deta2; dscan saz -0.1 0.1 40 1

        - Minimize the FWHM: take the derivative by diff command in newplot (or Pmca), use a Pseudo-Voigt or a Lorentzian function to fit the profile. Hint: reverse the curve (curve x (-1))
        - Tweak vfmb by 0.001 until you get the best focus/minimize the FWHM.
- Remove plexiglas, put new pinhole with a piece of plastic behind
    - FOURC: umv say 0 saz 0 sax 0 (if old pinhole: say = 0.1)
- Optimize beam position with **vfmth** and **mchi** (tweak them), check with say/saz scans (until they are centered)
- Close the slits and recenter them around the beam:
    - FOURC: plotselect ione
        - umv i1svg 0.1; dscan i1svo -0.3 0.3 20 1; umv i1svo CEN
        - umv i1shg 0.1; dscan i1sho -1 1 20 1; umv i1sho CEN
        - umv i1shg 0.5
- If precise offset knowledge is needed, proceed with silver behenate

Switching back to conventional Torus configuration

- Put the CCD, move say and saz to move the beam to the center
- Open slits
    - FOURC: umv i1svo 0; umv i1svg 3 i1shg 3
- Move ML chamber to the right height
    - KB: umv hfmz -7.5
- Move miroty to the reference position
- Move minisl

    - KB: umv minisl 27.5
- Move manually imirr Kapton foil (EH1) to 17.5
- Move I1v and I2v to the reference position (page 22)
- Move bender and mihexz to the reference position
- Tweak mchi to bring the spot to the center (vertical displacement on the screen)
- Move minisl
    - KB: umv minisl 27.5
- Check the beam position
    - KB: plotselect imirr; dscan minisl -0.2 0.2 20 1
    - Adjust mth if needed
- Remove minislit
    - KB: umv minisl 2
- If you will stay in torus configuration, you may need to align the beam with pinhole
- If you switch to the ML, follow the standard procedure, using the reference positions
- NB: new pinhole is used with say 0, old pinhole is used with say = 0.1

L) Alignment of the Spectrometer

This is typically done using the Si(9,9,9) reflection. For the first phase of the alignment the sample pinhole should be removed, as well as the alignment pinhole. Furthermore the detector pinholes need to be placed out of the beam, this corresponds to the beam passing through the large gap in the centre of the pinhole array (see the drawing below):
dsz = 8.5
dsr = 5.6
dsz1 = -7.0
dsr1 = 2.0

1) Put an appropriate attenuation of the x-rays. (wheel 225 or 245 in the beginning).
2) umv tth 0.618 (check for the latest most correct value for newdet).
3) umv znewdet -48.6
4) plotselect newdet
5) Check direct beam is centred on newdet (dscan tth 0.1 -0.1 20 1 & dscan znewdet -0.4 0.4 20 1)
6) move dz downwards (negative values) until beam is cut completely.
7) dscan dz 0 1 40 1; determine the ½-height of the profile, and set this dz-value to 0.(both set- and dial-values).
8) umv dz 1.1
9) umv dy 0
10) dscan dy –1 1 40 1; determine the ½-height of the profile, and set this dy-value to 0.(both set- and dial-values).
11) umv dy 7.41
12) umv dsz 0
13) umv dsr 0
14) dscan dsr –2 2 20 1; move dsr to 1.5mm from the ½-height of the profile.
    Nota bene, the dsr position is only temporary, since it has been determined with the newdet detector and not with the analyser beam.
15) dscan dsz –2 2 20 1; move dsz to the COM of the profile.
16) set and set_dial dsz 0
17) umv dsz 8.5
18) umv dsz1 -15.25 (= approximate position of 3x3 pinhole in direct beam.
19) dscan dsr1 –2 2 20 1; move dsr to -1.5mm from the ½-height of the profile.

Nota bene, the dsr1 position is only temporary, since it has been determined with the newdet detector and not with the analyser beam.

20) dscan dsz1 -2 2 20 1; move dsz1 to the COM of the profile.
21) umvr dsz1 7.95
22) umv znewdet -5

Check of the zero-position of the spectrometer arm. This procedure should be performed each time the spectrometer is aligned "from scratch" or the focusing set-up is modified (torus <-> cylinder+multilayer, KB configuration).

23) umv wheel 225/270/315
24) umv tth 0
25) plotselect deta2
26) umv a2hgap 5
27) umv a2vgap 60

If the spectrometer has been put in air, the analysers might not be optimised on the detector. A proper tth=0 procedure therefore demands adjustment of analyser 2.
Warning: ath1,achi1,ath5, and achi5 are no pseudomotors. Therefore, one can use them directly. ath2,achi2,ath3,achi3,ath4, and achi4 are pseudomotors. Therefore one should use athp(i) and achip(i) instead.

28) dscan athp2 –0.015 0.015 30 1
29) umv athp2 CEN
30) dscan achip2 –0.015 0.015 30 1
31) umv achip2 CEN
32) dscan tth 0.1 –0.1 20 1
33) determine the center value
127) umv tth (determined center value)
128) set tthm (value at which tth=0)
129) wm tth (should be at 0 after step 68)

The reason for this non-straightforward procedure is that tth is a pseudo-motor, involving a mathematical expression to convert the physical position of the real motor tthm (motor driving the lead screw) into the value for the scattering angle. If an angular correction is needed, one has therefore to act on the real motor tthm.

Mount standard plexiglass scatterer in order to proceed with the alignment of the crystal analysers and umv say 2

Alignment of analysers #1 to #5

34) Move tth to Q=10 nm^{-1} for analyser #3
35) Close all the analyser slits but the ones for analyser #3
~~36) umv dsz 9~~
~~37) umv dsz1 24~~
(We did not move these motors last time we did it)
38) dscan achip3 –0.02 0.02 40 1; umv achip3 CEN
39) dscan athp3 –0.02 0.02 40 1; umv athp3 CEN
40) umv dsr 0 (horizontal translation)
41) umv dsz 4.1 (put dsz in its operational position). For experiments, not using the Si (12 12 12) configuration, the dsz-value can be "relaxed" to 4.4 mm.
42) dscan dsr –2.5 2.5 25 1
Put dsr at the correct position.
43) recheck athp3 and achip3.
44) Align analysers 1 to 5.
45) Double-check that all analysers are properly aligned by doing a dsr scan. THIS IS NOT REPRODUCIBLE.

Alignment of analysers #6 to #9

46) plotselect deta7
47) umv dsz1 position in step 21
48) dscan ath7 -0.015 0.015 20 1, umv ath7 CEN
49) dscan achi7 -0.015 0.015 20 1, umv achi7 CEN
50) umv dsz1 23.2 (working position of 3.5x3.5 mm pinhole). This value needs to be carefully checked in order to avoid flux losses. In fact, with respect to the height position determined in the direct beam, the pinhole needs to be moved relative by (+15.25 – 4.26 mm). The exact value needs to be checked carefully each time.
51) dscan dsr1 -2.5 2.5 25 1, umv dsr1 CEN. If this does not give a conclusive profile, use the smaller pinhole to center it: umv dsz1 = 12.21-4.26, and redo the dsr1 scan. For experiments, not using the Si (12 12 12) configuration, the dsz1-value can be "relaxed" to 23.4 mm.
52) align analysers 6,8, and 9
53) Double-check that all analysers are properly aligned by doing a dsr1 scan. THIS IS NOT REPRODUCIBLE

Macro for analysers alignment: anaali ()

Alignment of the sample pinhole

- Aligning the ssz position of the sample pinhole

54) Mount the sample pinhole, umv say 0, plotselect deta2, umv tth 0, umv wheel 180, dscan ssz –1.5 1.5 30 1; umv ssz CEN.

- Aligning the ssy position of the sample pinhole

The ssy-position of the sample pinhole is best optimised with the scattered beam by making a scan and checking the intensity on analyser #1 and #5. It should nevertheless be positioned approximately OK (by eye).

55) Mount plexiglass, umv say 2, go to Q=10 nm^{-1} for analyser #3, dscan ssy -0.75 0.75 20 1 (broad), run almomi (), anaali ()

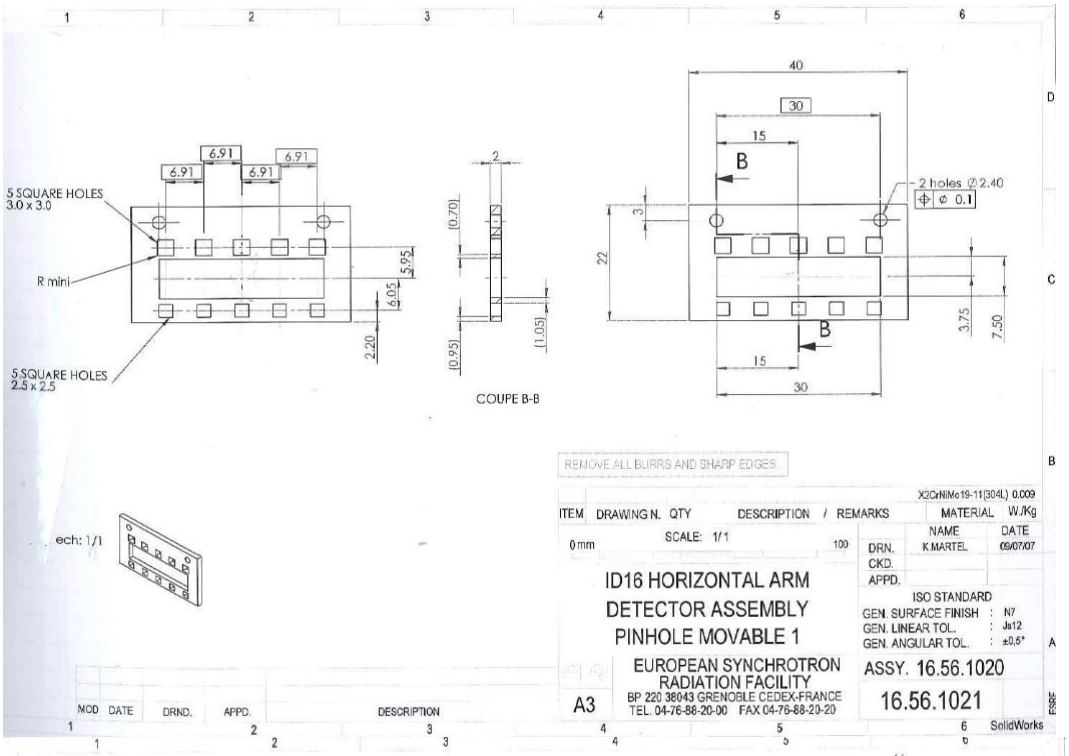
Realignment when changing from small/large to large/small scattering angles

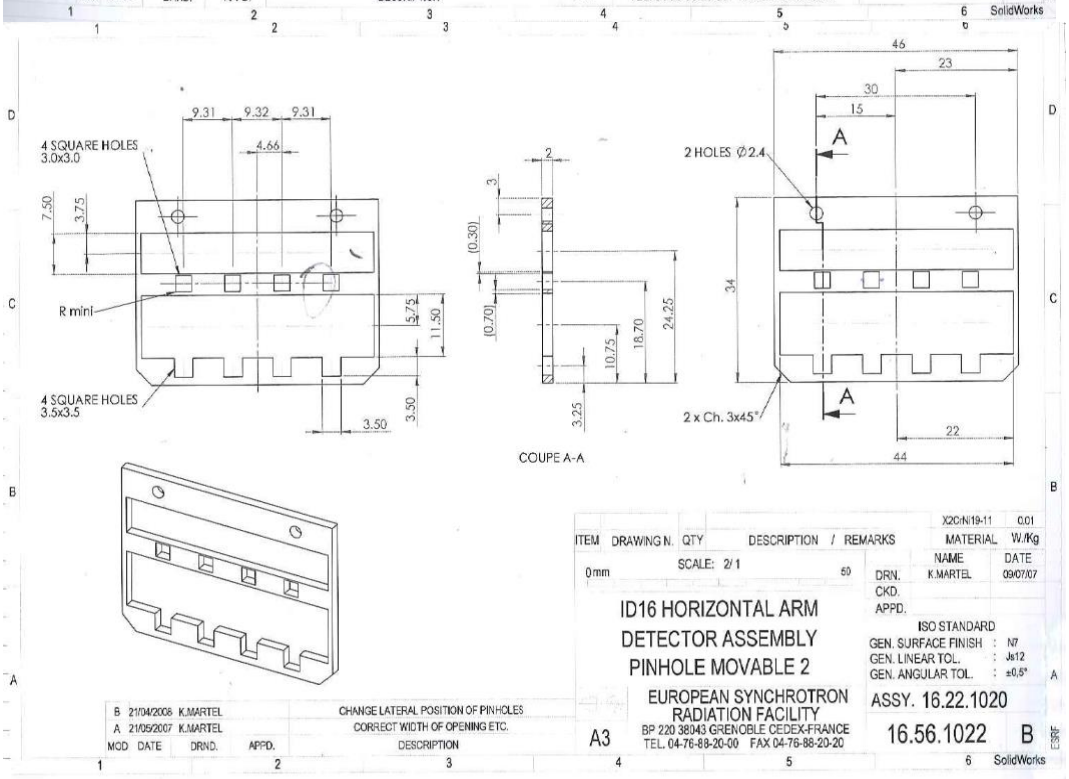
- There is no need for corrections for tth range from 0 to 25 degrees.
- For larger angles analysers 1-7 stay within less than 10% signal loss, ath8 and ath9 need to be tweaked.

Current operational position of the detector pinholes and chamber

dz:	1.1	dy:	7.41
dsz:	4.4	dsz1:	-4.4
dsr:	6.1	dsr1:	3.25

Drawings of the detector pinholes (these are the old ID16 pin-holes).





3. ADDITIONAL ALIGNMENT ROUTINES AND BEAMLINE ELEMENTS

A) Kappa goniostat

Verified on 01/06/2015 after ICEPAP migration.

Kappa name	phi	kappa	omega
DRAP name	dchi	deflz2	deflth
cable	372	373	374
Config	106	107	108
Steps per degree/mm	2000	2000	2000
Sign of user dial	-1	+1	-1
Backlash (steps)	-500	500	-500
Steady state rate (Hz)	400	800	800
Base rate (Hz)	100	200	200
Acceleration time (ms)	200	250	250
Current (A)	0.9	0.9	2

limit switches: OFF

Rotations:
omega - CW (bird's view)
phi - CW (bird's view) @ kappa = 0
kappa – CW (sight toward ring's wall) @ omega = 0

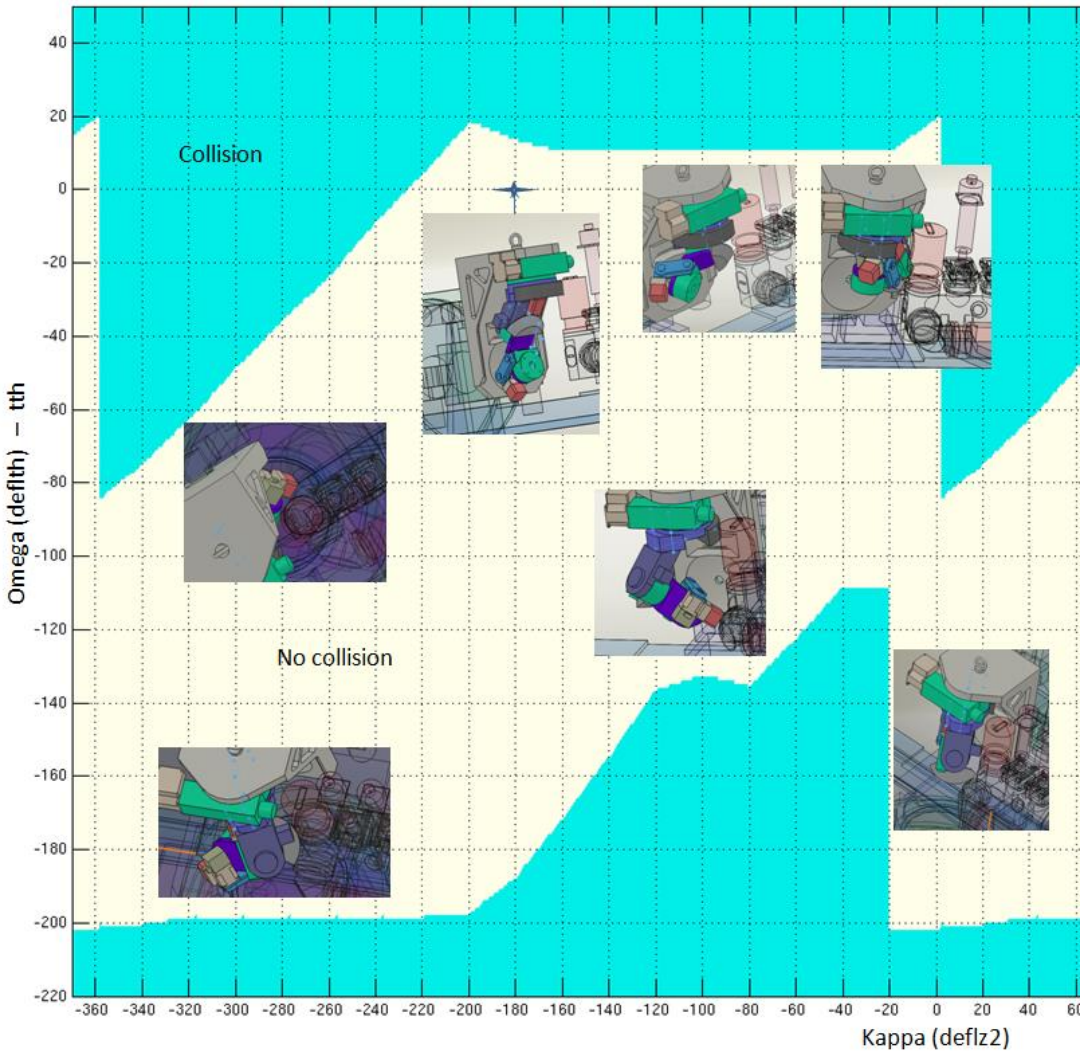
The following table was done presumably by Alexey, long ago.
available omega for variable kappa

kappa	collision-free	shadow
0	-200 +35	-
24	-205 +35	-
30	-115 +30	-
60	-120 +30	-
90	-135 +30	-60 0
120	-150 +30	-90 -10
150	-175 +30	-115 -30
180	-200 +35	-120 -45
195	-190 +20	-140 -60
210	-190 +10	-150 -65
195	-190 0	-160 -70
240	-190 -15	-170 -80
255	-190 -30	-180 -90

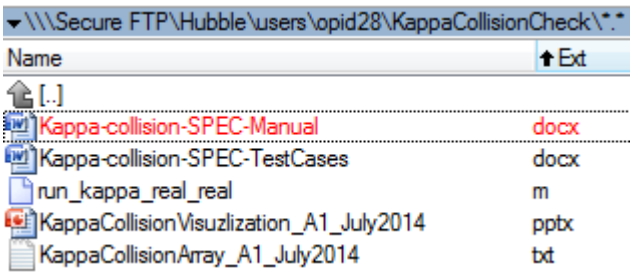
270	-190 -35	-185 -110
300	-190 -35	-
330	-190 -55	-

tth tested up to 20 deg. – detector connector has to be modified – needs to be shorter than 25 mm (from detector cover)
shift of collision-free omega range by tth

The following collision map was done more recently, using SolidWorks model. Last verification by moving real motors done 01/06/2015.



There is a software solution “Kappa Collision Prevention System”, which, when enabled, prevents the user from breaking the equipment due to collisions. Refer the documentation in the following folder on hubble:


\\\Secure FTP\Hubble\users\opid28\KappaCollisionCheck*.*
Name
Ext
[..]
Kappa-collision-SPEC-Manual
docx
Kappa-collision-SPEC-TestCases
docx
run_kappa_real_real
m
KappaCollisionVisuzlization_A1_July2014
pptx
KappaCollisionArray_A1_July2014
txt

Characteristics of temporary motors

	Motor settings	Steps [deg/mm]	Sign	Back-lash	Steady state rate	Base rate	Accel time	Rotation/ translation direction
adth	6/6/6	5000	-1	-400	2000	200	2000	+ cw
achi	6/6/6	500	1	50	250	200	500	+ cw looking towards source
LLNL achi	6/6/6	200	1	50	100	50	2000	+cw towards source
deflector diode z	7/7/7	-400	1	100	200	200	200	+ upwards
dchi	7/7/7	400	1	100	50	100	250	+ccw looking towards source
deflz2	7/7/7	-400	1	50	200	200	125	+ upwards
deflth	6/6/6	-4000	1	500	1000	200	250	+cw looking towards source
deflz1	4/4/4	20000	1	2000	1000	200	125	+ upwards
deflector wedge	7/7/7	-150000	1	10000	1000	200	250	+ upwards
entrance slit Florence	0/0/0	1600	1	800	800	200	125	+ towards ring wall
beamstop Florence	0/0/0	1600	1	800	800	200	125	+ towards ring wall
hobj2	8/8/0	20500	1	0	1000	200	125	
wobj2	8/8/0	1600	-1	0	2000	200	125	
stopy	1/1/0	800	-1	0	1000	200	125	+ towards EXPH
stopz	1/1/0	800	-1	0	1000	200	125	+ upwards
piny	9/9/0	20480	-1	0	2000	100	1000	
pinz	9/9/0	20480	-1	0	2000	100	1000	

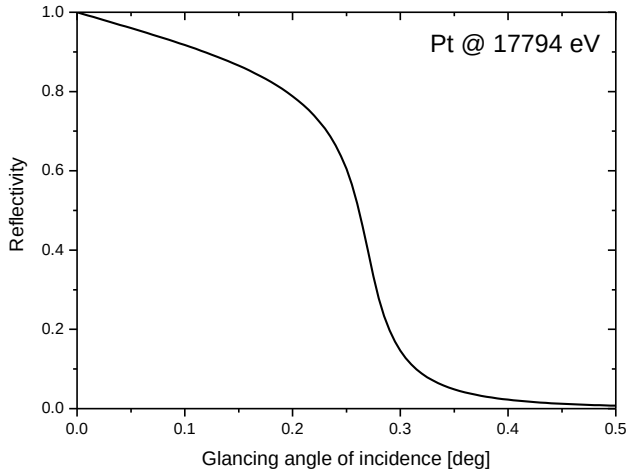
Motor and cable assignment for the temporary motor rack

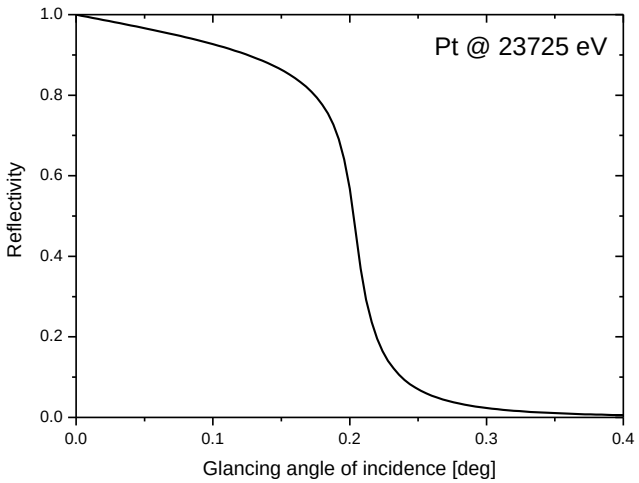
	1	2	3	4	5	**6**	**7**	**8**
Motor	333	372	373	374	375	**376**	**391**	**???**
Limit	341	380	381	382	383	**384**	**392**	**???**
Typical motors	adth, achi, diodez	dchi, hobj2, wedge, florence cell in	deflz2, wobj2, florence cell out	deflth, stopy	deflz1, stopz	**znewdet**	**pmono2**	**ash9**

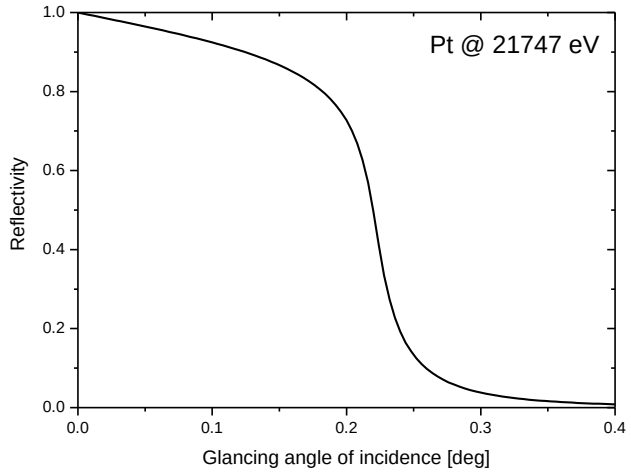
Simple diagnostics of icepap motors

1) Open a terminal on beamline control computer
2) su- blissadm (password: spec92)
3) type: icepapcms (this opens the graphical interface)
4) click on icepapid281
5) follow hopefully self-explanatory menus.
6) One of the key commands to quest for motor status: 16:?vstatus

B) Grazing Incidence Setup (refine routine next time)







Standard beamline configuration

Nominal height of detectors: dz_0 = 0.75 mm
Incident angle of beam: α_1 = 4.7 mrad = +0.2693º
Distance toroidal mirror to sample: D = 25 m
Distance deflector mirror – sample: d = 200 mm
Distance sample-detectors entrance: d' = 85 mm

Main motors:

dchi: + counterclockwise (looking towards spectrometer)
deflth: + counterclockwise (looking at the goniometer)
deflz1: + upwards (deflector height)
deflz2: + upwards (*deflector support height*)
slitz: check next time

1.- Deflector mirror alignment:

a) Slit/Beam position: i1svo, i1svg

We need to know the operating angle of grazing incidence in the sample, α_s.

Example: $TaSe_2$
Critical angle, α_s **= 0.14 degrees = 2.4435 mrad**

The beam shall be deflected from +4.7 mrad to –2.44 mrad in order to impinge the sample in critical incidence:

$\Delta\alpha$ = 7.1435 mrad ⟶ θ_{Pt} = -3.572 mrad = -0.2047 degrees

There will be a change in the height of the beam at the sample position given by
200 x tan($\Delta\alpha$) = -1.4288 mm = Δh
which needs to be corrected for by shifting the vertical slits by the same amount:

umvr i1svo 1.4288

This will be the first approximation to get the beam impinging the sample in the critical angle. If we want to take into account the change of the height Δh in a second iteration, the initial angle would be 4.73 mrad and **$\Delta\alpha$ would be 7.172 mrad**. **θ_{Pt} = -3.586 mrad** and the new value for **Δh = -1.434 mm**.

So instead of the previous command, a more accurate one would be
umvr i1svo 1.434

b) Deflecting mirror:

- Mount slitz and pico in front of detector entrance: use the tripod to mount slitz parallel to entrance slit

- Move the deflecting mirror out from the beam path

- ***Plotselect pico1***; scan with **dz** until locating the beam position in the 100 micron slit of **slitz** (approx. dz'=dz_0 + 1.5 mm, where 1.5 is Δh)

- Move deflector height until it intersects the beam path: **umvr deflz1 -1**

- Make scans in **deflth**, **deflz1**, and **dchi**, until getting a symmetric triangular shape (mirror aligned parallel to beam), centering dfeflz1 at half width of the maximum intensity, and deflth and dchi in the maximum of the triangle.

With this process, the deflecting mirror is aligned parallel to the beam. By making

umvr deflth θ_{Pt} (negative angle)

the beam will be deflected to the sample position with an angle equal to the critical angle. To check this we can search the deflected beam by making a scan in **dz** (approx. dz''=dz_0-2.111, where 2.111 = (208+85 mm) x 7.204 mrad)

2.- Sample alignment:

- Take reference with tripod of **sax=0** position (use pinhole)
- At **sax=say=saz=0**, center your sample with the aid of the tripod and **move phi**, **chi**, **saz** and **sax** until the sample appears clearly centered, flat and with the surface at the right height from the telescope.
- **Scan in saz** to get the right height; move saz to half the maximum intensity.

- **Scan in phi** to set sample parallel to beam (symmetric triangular shape) and move to peak of triangle. Iterate with the previous step.
- **Scan in chi** to see how surface roughness affects the alignment (chi can be used to correct partially for the slope of the sample surface and the roughness).
- **Scan in say** to center sample in y-direction: check surface roughness.

Once the sample has been properly aligned parallel to the beam, it is enough to move phi equal to the critical angle:
umvr phi 1.4 (positive angle)

We can then check the critical angle by measuring the reflectivity with ***pico*** or with one of the detectors, for example ***deta2***.

3.- Sample orientation:

- Use the CCD camera to get the position of the right reflection and correct in **chi** and **th** to get the reflection in the appropriate spot of the CCD:
  y-position of the detectors = **40 ??**
- Use the program **name??** to calculate the appropriate corrections.

C) Beam check routines

1) michk (michkon, michkoff)

This check stops the scan, if the machine sets the injection flag. During the injection, even when the front-end stays open, no data are collected. Once the injection procedure is finished, the flag is reset, and the data collection continues.
Syntax: michkon <time> (>10s, default 15s)
We normally never touch this check.

2) monochk (monochksetup, onmonochk, offmonochk)

This check is always activated. It checks whether there is beam (via pmoni) and whether the premono is optimised with respect to the main mono. If this is not the case, the angle of the premono will be slightly corrected.

```
                          MONOCHECK SETUP

1  MONO monitor mnemonic <imirr>
2              threshold <90> %
3                   type <analog>
4  BEAM monitor mnemonic <pmoni>
5              threshold <20> %
6                   type <analog>
7  time for temperature stabilization after open refill <180>
sec.
8  time for temperature stabilization after beam loss <180> sec.
9    MONO motor mnemonic <mono>
10            tweak value <0.0003>
11        scan magnitude <0.001>
(lineup mono -0.001 0.001 20 1)
12        scan intervals <20>
13 scan integration time <1> sec.
14            line up at <CEN>
15           plot filter <1>
16 pre-scan magnitude after injection <0.002>
(lineup mono -0.002 0.002 40 1)
17 pre-scan intervals after injection <40>

Enter wanted option, or 0 to quit (0)?
```

The tweak value (#10) may need to be changed depending on the energy configuration. To get the right tweak value, look at the last mono scan of the almomi (with pmoni), find the maximum value, calculate the 90% threshold value and determine the suitable mono tweak.

3) *mirrorchk (mirrorchksetup, onmirrorchk, offmirrorchk)*

This check is typically used, if relatively large samples are used, corresponding to Ione-slit sizes larger than 100 microns. If the ratio Ione/izero is no longer correct, a small angular correction of the gold mirror is performed (miroty). An option allows as well to correct for an eventual (horizontal) movement of the beam. This can be activated within the set-up.

```
                        MIRRORCHECK SETUP

1  MIRROR monitor mnemonic <i10>
2                threshold <80> %
3                     type <analog>
4  readjust MIRROR after MONO correction <no>
5  check lateral beam centering <no>
6  check vertical beam centering <no>
7  MIRROR motor mnemonic <miroty>
(lineup miroty -0.005 0.005 30 1)
8            tweak value <-0.001>
9  scan counter mnemonic <ione>
10         scan magnitude <0.005>
11         scan intervals <30>
12  scan integration time <1>
13             line up at <CEN>

Enter wanted option, or 0 to quit (0)?
```

The tweak value (#8) may need to be changed depending on the energy configuration. To get the right tweak value, look at the last miroty scan of the almomi (with Ione), find the maximum value, calculate the 80% threshold value and determine the suitable miroty tweak.

4) ***Postmonocheck (postmonochksetup, onpostmonochk, offpostmonochk)***

This check has to be activiated, if the postmono has been inserted between the premono and the main mono. If the ratio Imirr/pmoni is below the pre-defined threshold, the routine first attempts to recover the situation with a premono tweak, then with a postmono tweak, and if this is not sufficient, the check routine will make alignment scans on the premono (mono-scan), on the postmono (pi2-scan), and again on the premono (mono-scan).

```
                        POST MONO CHECK SETUP

1 POST MONO motor mnemonic <pi2>
2            tweak value <0.065>
3    scan counter mnemonic <imirr>
4           scan magnitude <0.4>
(lineup pi2 -0.4 0.4 25 1)
5           scan intervals <25>
6    scan integration time <1> sec.
7               line up at <CEN>
8             plot filter <2>
```

```
Enter wanted option, or 0 to quit (0)?
```

The tweak value (#2) may need to be changed depending on the energy configuration. To get the right tweak value, look at the last pi2 scan of the almomi (with Imirr), find the maximum value, calculate the 80% threshold value and determine the suitable pi2 tweak.

Tweak value at Si (9 9 9) and 80% threshold: -0.048

5) ***qpchk (qpchksetup, onqpchk, offqpchk)***

monitor mnemonic:	ihorpos
threshold:	0.003
type:	analog
piezo mnemonic:	hfmpi
correction ratio:	see value in relevant table
min voltage:	1
max voltage:	9
mirror motor mne :	mirrot
correction ratio :	see value in relevant table

We normally never touch this check.

D) Alignment of the monitor slits

There are three slit units.
The imirr slit unit consists of only two blades (mlleft and mlright, pseudomotors: mlgap and mloff) which define the horizontal beam size. They are only used, when the multilayer is in operation.

The Huber slits are positioned behind the multilayer and in front of izero. They consist of four blades: huxl, huxr, huzd and huzu. Pseudomotors allow to move the offsets and gaps: huxo, huzo, huxg and huzg.

The ione slits are positioned in front of the ione detector. They are used to define the beam size impinging on the sample: i1sho, i1svo, i1shg and i1svg.

The alignment of the above slit units is only mandatory, if something strange is going on, or if the beamline is aligned "from scratch", i.e. after a long shutdown. Depending on the set-up (with or without multilayer), part of the procedure can be skipped.

In addition there is another pair of horizontal blades in front of the main mono (mxl and mxr with pseudomotors mxgap and mxoff). These are only activated for alignment purposes and checking beam movements. They should be set by default to 10 mm.

I) Imirr slits

1) plotselect imirr
2) umv mlgap 1
3) dscan mloff –3 3 30 1
4) umv mloff CEN
5) umv mlgap 4

The correct value of mlgap depends on the experiment. There might be cases where one would put the slits tighter, meaning that a small fraction of the edge of the beam is cut.

II) Huber slits

Procedure for setup without multilayer
This procedure might be shortened by simple performing offset scans with the nominal gap openings

6) plotselect izero
7) umv huxg 0.5
8) dscan huxo –2 2 20 1
9) umv huxo CEN
10) umv huzg 0.2
11) dscan huzo –2 2 20 1
12) umv huzo CEN

Otherwise, perform knife-edge scans to center properly each blade.

13) umv huxg 10
14) umv huzg 10
15) ascan huxl –1 1 40 1
16) determine ½ height of knife-edge scan and move huxl to this value.
17) set huxl 0
18) umv huxl 5
Repeat procedure for huxr, huzd and huzu
19) umv huxg 2
20) umv huzg 2

III) Ione slits

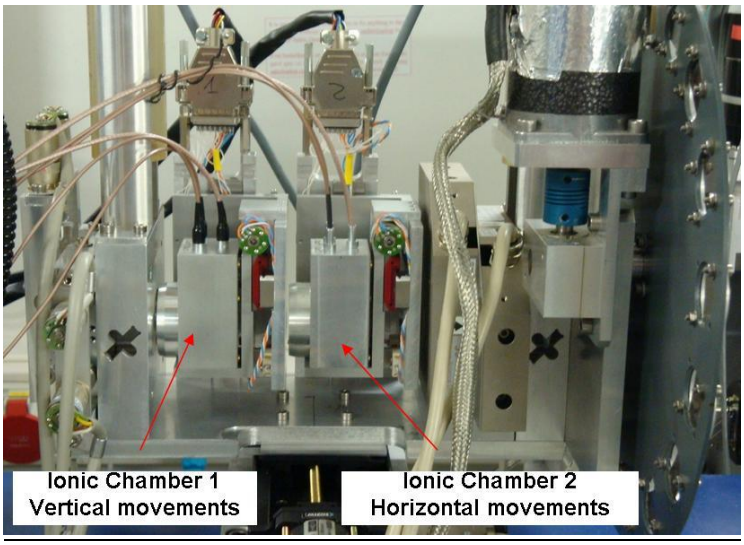
Procedure for setup without multilayer

21) plotselect ione
22) umv i1svg 0.5
23) umv i1shg 0.2
24) dscan i1sho –0.6 0.6 30 1
25) umv i1sho CEN
26) umv i1shg 0.5
27) umv i1svg 0.02
28) dscan i1svo –0.15 0.15 30 1
29) umv i1svo CEN
30) umv i1svg 0.2

Standard values of i1shg and i1svg are for standard samples:
shg: 0.5 mm; svg: 0.2 mm

E) Alignment of the Quadrupole Ion Chambers

The quadrupole ion chambers (QIC) are located in-between the Izero and Ione slits, and can be activated if a small beam needs to be stabilised within +/5 μm at the sample position.
The diameter (clearance for x-ray beam) is about 6.7 mm.



Voltage = 1800V;

IC	motors [mm]	Directions	counters	V560 channel n.	
IC 1	I1v	+ up	iver1	12	
	I1h	+ towards EXPH	iver2	13	
			iverpos = (iverr1-iver2)/(iver1+iver2)		
IC 2	I2v	+ up	ihor1	14	
	I2h	+ towards EXPH	ihor2	15	
			ihorpos = (ihor1-ihor2)/(ihor1+ihor2)		

limits of the relevant motors are indicated as reference (as of 23.8.2010)

	low limit	**high limit**
i1sho	-7.438	7.379
I1h	-5.795	4.368
I2h	-2.653	7.361

Position of ion chambers

(approximate) distances involved:
moniy – sample: 450 mm
Ion1 unit – sample: 500 mm
Ion2 unit – sample: 435 mm
I1 unit – sample: 370 mm

Relative movements of the various components
(just indicative to have an idea; these are not real motor positions)

n	I1h [mm]	ymoni [mm]	I2h [mm]	i1sho [mm]
no ML	0	0	0	0
8	31.12	32.67	33.14	35.16
9	27.66	29.04	29.45	31.25
11	22.63	23.76	24.10	25.57
12	21.14	22.20	22.52	23.89

calibration in torus configuration:

I1h: iverpos(I1h) = -1.75×10^{-5} μm^{-1}
I1v: iverpos(I1v) = -2.36×10^{-3} μm^{-1}
I2h: ihorpos(I2h) = 8.70×10^{-4} μm^{-1}
I2v: ihorpos(I2v) = -1.90×10^{-5} μm^{-1}

pi1: iverpos(pi1) = 2.578×10^{-3} μm^{-1}
!!!Nota bene: this only works if there is no postmono in the beam!

calibration in multilayer configuration

I1h: iverpos(I1h) = -2.55×10^{-5} μm^{-1}
I1v: iverpos(I1v) = -3.78×10^{-3} μm^{-1}

I2h: ihorpos(I2h) = 7.43×10^{-4} μm^{-1}
I2v: ihorpos(I2v) = -1.9×10^{-5} μm^{-1}
Piezo: ihorpos(hfmpi) = 0.0076 V^{-1} (in check routine: +0.0076)
hfmth: ihorpos(hfmth) = 25.407 deg^{-1} (in check routine: -25.407)

calibration in KB configuration

I1h: iverpos(I1h) = -1.47×10^{-5} μm^{-1}
I1v: iverpos(I1v) = -1.915×10^{-3} μm^{-1}
Piezo: iverpos(vfmpi) = -0.046 V^{-1} (in check routine: +0.046 ???)
vfmth: iverpos(vfmth) = +162.666 deg^{-1}

I2h: iverpos(I2h) = 7.37×10^{-4} μm^{-1}
I2v: ihorpos(I2v) = -1.75×10^{-5} μm^{-1}
Piezo: ihorpos(hfmpi) = 0.0081 V^{-1} (in check routine: +0.0081)
hfmth: ihorpos(hfmth) = 25.407 deg^{-1} (in check routine: -25.407)

F) Bassler “focus quality” camera

Position:

FOURC: umv say 0 saz 0

Starting camera:
1) connect to leonov from gagarin2 via the xterminal application
2) Open firefox
3) Click on BPM monitor icon which is located on the right of the top tool bar.
4) the rest is self-explanatory. Note that the pixel position and the FWHM of the focal spot are indicated as well in FOURC.

Start-up problem:
If the camera does not start up correctly, most likely it is a server problem. To fix this, you need to open a new terminal window (not inside a SPEC session):

Login as blissadm: su blissadm
Password: spec92
type: dserver –gui&

This shows the status of all the device servers, and by clicking on the appropriate icon, you can stop and restart the device server.

If this does not work, you have to perform a hardware reset of the camera, which is simply done by unplugging the power cord, and the replug it. You then should restart the server, and the application in firefox.

Pixel size: 3.57 μm/pixel
y and z are inverted

G) Premono and Mirror Hexapode control

The premono and the mirror are installed on Hexapodes which allow to perform very fine movements off all three translations and rotations.

mihexx (mm) :	translation along the beam direction in the horizontal plane
mihexy (mm):	translation perpendicular to the beam direction in the horizontal plane.
mihexz (mm):	translation perpendicular to the beam in the vertical plane.
mirotx (mrad):	rotation around x-axis.
miroty (mrad):	rotation around y-axis.
mirotz (mrad):	rotation around z-axis.

The movements are controlled by two different SPEC versions *HX1* (premono) and *HX2* (mirror). Furthermore, the two most important movements of the mirror hexapode can as well be found in *FOURC* (mihexz and miroty).
NOTA BENE !!!
In order to avoid a conflict when using the same motor in two different applications, one has to deactivate the respective motor in the application which is not used.

Deactivation of hexapode motors in *HX2* and activation in *FOURC*:
HX2> hexaoff
FOURC> hexaon

The various functions of the hexapode are called by the hexapode menu. For example in *HX2*:

1 – Show Hexapode	Indicates the status of the hexapode
2 – STOP	Stops hexapode movement
3 – Soft reset	This only resets error flag, and might be necessary, if the VPAP has been switched off, or the computer had to be rebooted.
4 – Set Reference Position	**DO NOT USE**
5 – Move hexapode legs	allows to move individual legs of hexapode. This is not of particular interest.
6 – Move absolute to pos	allows to nove to a specific position.
7 – Switch mode	This allows to make simulations of movements. In simulation mode, the hexapode does not move.

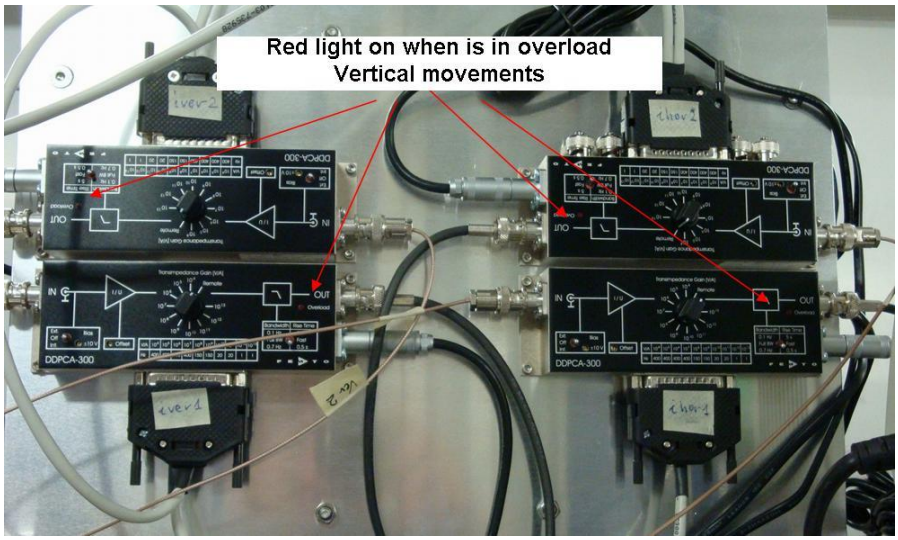
8 – Set new Lengths Values	**DO NOT USE**
9 – Search Home switches	**Only for Expert Personnel**
10 – Full reset	**Only for Expert Personnel**
11 – Calc and Check positions	

H) Femto Current Amplifier Adjustment

Indication which devices are active
FOURC> femtogain

Indication of actual amplification:
FOURC> femto_gain <device name>

Change of amplification:
FOURC> femto_gain <device name> xxx xxx = 1e07, 1e08,…….,1e13



You should check if the current amplifier is in overload with the web camera (red light on)

I) Setup of the Keithley Amplifiers

1) Keithley in Control Cabin CC1

This current amplifier amplifies the (current) signal from a silicon pin diode, located inside the spectrometer arm. This detector is used for alignment purposes. Especially after a shutdown or an electrical breakdown, it needs to be properly reinitialised. Detector name: newdet

1) Deactivate the zero check.
2) Increase the filter rise time to 300 msec.
3) Increase the gain to 10^8.
4) Switch off the light in the hutch and eliminate any other source of light.
5) Do a zero check correction by pushing the “shift”-button and the “correct” button.

2) Picoamperemeter (in EH1)

setup: picoammeter_range pico1
specify ranges which are available
picozero: makes zero check for chosen range. This implies that there is no beam on the diode and that the light is off in the hutch!

picoammeter_on pico1 enables pico1
picoammeter_off pico1 disables pico1

J) Florence HP vacuum chamber – special issues

Maximum value of tth at sax = 0 and th = 0: 16 degrees

dchi: entrance slit in hole. It has three slots, two of them which are operational: a 80 μm and 180 μm one. The 80 μm one is the one used for standard measurements.

deflth2: exit beam stop. It is about 1mm wide.

dchi and deflz2 positions with sax = 0, say = +0.1, saz = 0

dchi:	upper limit:	+17.8912
	½ cutting beam:	+3.03
	180 μm slit:	+8.06
	80 μm slit:	+13.06
deflz2:	lower limit:	-14.31
	centered on beam:	+0.21

K) Analyser temperature adjustments

This is typically done at the Si (9,9,9).

a) dscan monot T0+0.15 T0-0.15 20 20
b) Determine all the CEN values
c) Take the analyser #3 temperature as a reference, and calculate all the temperature differences.
d) Change the set points of analysers, if the temperature difference is larger than 0.01 deg C.

See separate chapter for the change of the analyser temperatures

L) Analyser temperature control

The analyzers temperature control is performed in the SPEC program f300

1) Reading of the temperature setpoints
   f300> *f300*
   The display should show:
   Number of F300 Temperature controllers: 9

	GPIB	*Display*	*Celsius*	*Setpoint*		*Enter new Setpoint*
1	*15*	*+0.000000B*	*22.5840*	*22.5840*	*>*	
2	*16*	*-0.000000B*	*22.6781*	*22.6781*	*>*	
3	*17*	*+0.000000B*	*22.5850*	*22.5850*	*>*	
4	*18*	*+0.000000B*	*22.5990*	*22.5990*	*>*	
5	*19*	*+0.000000B*	*22.5791*	*22.5791*	*>*	
6	*22*	*+0.000000B*	*22.6200*	*22.6200*	*>*	
7	*21*	*-0.000000B*	*22.6310*	*22.6310*	*>*	
8	*20*	*+0.000000B*	*22.5600*	*22.5600*	*>*	
9	*23*	*+0.000000B*	*22.6750*	*22.6750*	*>*	

2) Change of temperature setpoint
   When the prompt goes in the column “Enter new setpoint” you are allowed to change the value
   →write the new value in the correspondent row and type return

   N.B. The setpoints are saved in the file:../blissadm/local/spec/userconf/f300.f300.setpoints

3) Change the settings
   Press the “c” button and the display should show:
   Number of F300 Temperature controllers: 9
   Use arrow keys to move cursor, "c" to change settings, "w" for saving, "q" to quit

	GPIB	*Sensitivity*	*mA*	*Mode*	*Unit*	*Reference*
1	*15*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
2	*16*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
3	*17*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
4	*18*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
5	*19*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
6	*22*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*

7	*21*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
8	*20*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*
9	*23*	*High*	*1.0*	*Deviation*	*Ratio*	*Internal*

These values are saved in the file:
../blissadm/local/spec/userconf/f300.f300.config

Starting from scratch

- Switch on all the ASL;
- Run the f300 program;
- then type f300 to see if the setpoints are correct; N.B. if the setpoints are not correct is better to insert them by hand
  (For example if you start > f300 –f → all the setpoints go to zero)

M) Read-out of Primary Slits Thermocouples (in SLITS)

If the server is not running: wagosetup wcid28a
Reading the T's: intlck show
Reset of the TC parameters: intlck reset

N) GPIB adresses on ID28

Gpibid28b – 160.103.48.96
IP address exists, but the controller is nowhere addressed

GPIB0 – gpibid28a – 160.103.48.95

pma_112	prem_ad1:12	Commented out
15	F300	Ana1
16	F300	Ana2
17	F300	Ana3
18	F300	Ana4
19	F300	Ana5
20	F300	Ana6
21	F300	Ana7
22	F300	Ana8
23	F300	Ana9

GPIB1 – gpibid28c – 160.103.48.98

address	instrument	Destination
2	PI controller	premono
5	PREMA	Main mono
6	PREMA	Ana1
7	PREMA	Ana2
8	PREMA	Ana3
9	PREMA	Ana4
10	PREMA	Ana5
11	PREMA	NTC main mono 2. probe
12	PREMA	Ana7
13	PREMA	Ana6
14	PREMA	Ana8
15	PREMA	Ana9

GPIB2 – gpibid28d – 160.103.48.18

address	instrument	Destination
3	F700	Main mono

GPIB3 – gpibid28e – 160.103.48.19

address	instrument	Destination
2	ST15 cryostat	EH1
22	Keithley pico.	EH1

check and update

O) Detector thresholds and signal heights

5-Element detector (new 5x(3x8) monolith)
7245 Amplifier
Coarse gain: 200; fine gain: 10; shaping time: 4 μs; unipolar mode; multiplier: x1; resistive diode; polaritiy: positive; output: uni

Reflection	#1	#2	#3	#4	#5
(7,7,7)					?/? (?)
(8,8,8)	2.0/2.6 (2.3)	1.9/2.5 (2.2)	1.9/2.5 (2.2)	1.9/2.5 (2.2)	2.0/2.6 (2.3)
(9,9,9)	2.3/2.9 (2.6)	2.1/2.7 (2.4)	2.05/2.65 (2.35)	2.1/2.7 (2.4)	2.3/2.9 (2.6)
(11,11,11)	2.8/3.4 (3.1)	2.6/3.2 (2.9)	2.5/3.1 (2.8)	2.6/3.2 (2.9)	2.8/3.4 (3.1)
(12,12,12)	(3.4)	(3.0)	(3.0)	(3.0)	(3.0)
(13,13,13)					

4-Element detector
Amplifier to be defined

Reflection	#6	#7	#8	#9
(7,7,7)				
(8,8,8)				
(9,9,9)				
(11,11,11)				
(12,12,12)	(3.4)	(2.5)	(2.5)	(3.0)
(13,13,13)				

Monitor detectors
Izero/Ione: 7243 Amplifier
Coarse gain: 50; fine gain: 10; shaping time: 4 μs; auto; ASYM; input: positive; output: unipolar
S(Q): 7243 Amplifier
to be defined

Reflection	Izero	Ione	S(Q)
(7,7,7)			
(8,8,8)	1.7/4.0 (2.6)	1.7/6.0 (2.4)	
(9,9,9)	1.7/4.0 (2.8)	1.7/6.0 (2.6)	
(11,11,11)	1.7/4.0 (3.6)	1.7/6.0 (3.0)	
(12,12,12)			
(13,13,13)			

P) Temperature Setpoints of Hutches

OH1: T_{set} = 23° C, GV

OH2: T_{set} = 22° C, GV

OH3: T_{set} = 22° C, MV

EH1: T_{set} = 20.8° C, MV

CC1: T_{set} = 24° C

Q) Approximate count rates on the monitor detectors

(in Hz and normalised to 200 mA ring current)

Detector	**n=8**	**n=9**	**n=11**	**n=12**
pmoni ($1x10^8$)	$1.43x10^5$	$2.51x10^5$	72 000	52 574
pomoni ($1x10^8$)	$6.41x10^4$	$1.11x10^5$	33 921	25 105
imirr	17570	12 724	1 528	1008
izero	5070	2 154	624*	422*
(ione**	13042	6980	4 400*	1170*)

* with Kapton scattering foils
** depends on ione slit size

Relative count rates (for same sample)

YBCO (February 2012): n=9 versus n=11 factor 11 (on deta2)
YBCO (February 2012): n=9 versus n=12 factor 11 (on deta2)

R) Relative tth between analyzers

	#6	#2	#7	#3	#8	#4	#9	#5
#1	0.741°	1.520°	2.267°	3.047°	3.79	4.569°	5.30	6.097°
#6		0.780°	1.527°	2.307°	3.04	3.829°	4.56	5.357°
#2			0.747°	1.527°	2.26	3.049°	3.78	4.577°
#7				0.780°	1.52	2.302°	3.04	3.830°
#3					0.74	1.522°	2.26	3.050°
#8						0.781	1.52	2.31
#4							0.737	1.53
#9								0.789

S) Column list from TEMPERATURE SCANS as of Jan. 30, 2003

Col#	Counter name	Counter meaning	Inv.#
1	Temperature	monot setpoint	-40
2	H		-39
3	K		-38
4	L		-37
5	Epoch		-36
6	Sec	integration time	-35
7	Pindiode		-34
8	izero OH2	pmoni	-33
9	izero EH1	izero (mon. after ML)	-32
10	Ione/Izero		-31
11	Sec		-30
12	Prema_ad5	NTC mono in Ω	-29
13	Prema_ad6	NTC analyser #1	-28
14	Prema_ad7	NTC analyser #2	-27
15	Prema_ad8	NTC analyser #3	-26
16	Prema_ad9	NTC analyser #4	-25
17	Prema_ad10	NTC analyser #5	-24
18	Prema_ad11	NTC analyser #6	-23
19	Prema_ad12	NTC analyser #7	-22
20	Prema_ad13	NTC analyser #8	-21
21	Prema_ad14	NTC analyser #9	-20
22	Prema_ad16	NTC EH1	-19
23	Pico		-18
24	deta1		-17
25	deta2		-16
26	deta3		-15
27	deta4		-14
28	deta5		-13
29	SR current		-12
30	NTC_mono	mono temp. in °C	-11
31	Cryostat	T2(sample) in displex	-10
32	Newdet	S(Q)-det near Ana2	-9

33	prema_ad15	PT100 chamber 7m	-8
34	Ione	monitor after slits	-7
35	Detsq	S(Q) near sample	-6
36	deta6		-5
37	deta7		-4
38	deta8		-3
39	deta9		-2
40	Imirr	monitor before ML	-1

T) Scanner Prema

(17.10.2002)

Assignment of channels

Channel #	Cable #	destination	sensor	unit	type
1	100	Crystal-front	Pt100	T	4-pole
2	101	Crystal back	Pt100	T	4-pole
3			Pt100	T	4-pole
4			Pt100	T	4-pole
21	102	x-heat ex. Out	Pt100	Ohm	2-pole
22	98	Exchang 2 out	Pt100	Ohm	2-pole
23	98	Cold head out	Pt100	Ohm	2-pole
24	99	Exchang 1 out	Pt100	Ohm	2-pole
25	99	Exch 2 in (ret)	Pt100	Ohm	2-pole
26	107	Supp. Above	TC, type K	mV	2-pole
27	108	Supp. Below	TC, type K	mV	2-pole

Nota bene: For the 2-pole resistance values one has to subtract 3.3 Ohm due to the cable resistance.

Monitoring of mono temperatures:

- Start the spec session “cool”
- newsession etc.
- plotselect ci with i=1,9 (channels 1 to 27)
- program is written manualy (=temporarily) in the setup of “cool”.
- Reading of each channel takes 2 secs, therefore it is better to use a an interval reading time larger than 20 secs to be safe.

U)Temperature Scans

There are two pseudocounters: T-monot and E_monot, they are the temperature and the energy setpoint of the main backscattering monochromator.
Thus, if you use either monot or deltae for your scans, you have the complementary information in the counter list.

Furthermore, there is a command for ixsscans which automatically moves the wheel to zero, znewdet and zdetsq1 out of the way, and activates the premas. The command is as follows:
ixsscan monot T0+0.5 T0-0.5 50 60

or

ixsscan deltae -20 20 40 60

4. LOW/HIGH TEMPERATURE EQUIPMENT

A) Joule-Thomson low temperature displex

User manual
By L. Paolasini an T. Forrest for ID28

PRINCIPLE

The principle of operation of a Joule-Thomson is to inject the ^{4}He under high pressure (about 10 bar) across a carbon trap immersed in liquid nitrogen (at 70 K) in order to remove the impurities and purify the gas.

This gas is then thermalized on the first stage at 50 K, and then on the second stage at 10 K with a cold head of a closed cycle Gifford-McMahon. This gas, the temperature of which is now 10 K, flows across a contra-flow exchanger, and then across impendence gate in which the gas is pressure reduced from 10 to 1 bar, lowering its temperature. The gas then renters the exchanger in a contra-flow and cools the gas which flows into the impendence gate. In such a way we reach a temperature close to that of liquid Helium, 4.2K.

If we lower the pressure behind the impendence gate with the primary pump, from 1 bar to a few mbar, this lowers the temperature to 1.8 K.

SAMPLE MOUNTING

In order to reach the lowest temperature, the sample must be surrounded by Be dome, which contains a He exchange gas. The Be dome in Fig.1(4) is sealed with indium wire (1.5 mm diameter) on the copper block. The exchange gas is injected using a He gas bag (Fig.1(1)), provided with a 3-way valve (Fig.1(3)), which allows the pumping and flushing of the exchange gas injection line (Fig1.(2)). Finally, a thermal screen dome (Fig.1(5)) is screwed on the displex thermal screens.
PLEASE, USE THE GLOVES WHEN MANIPULATING THE BE DOMES!!!.

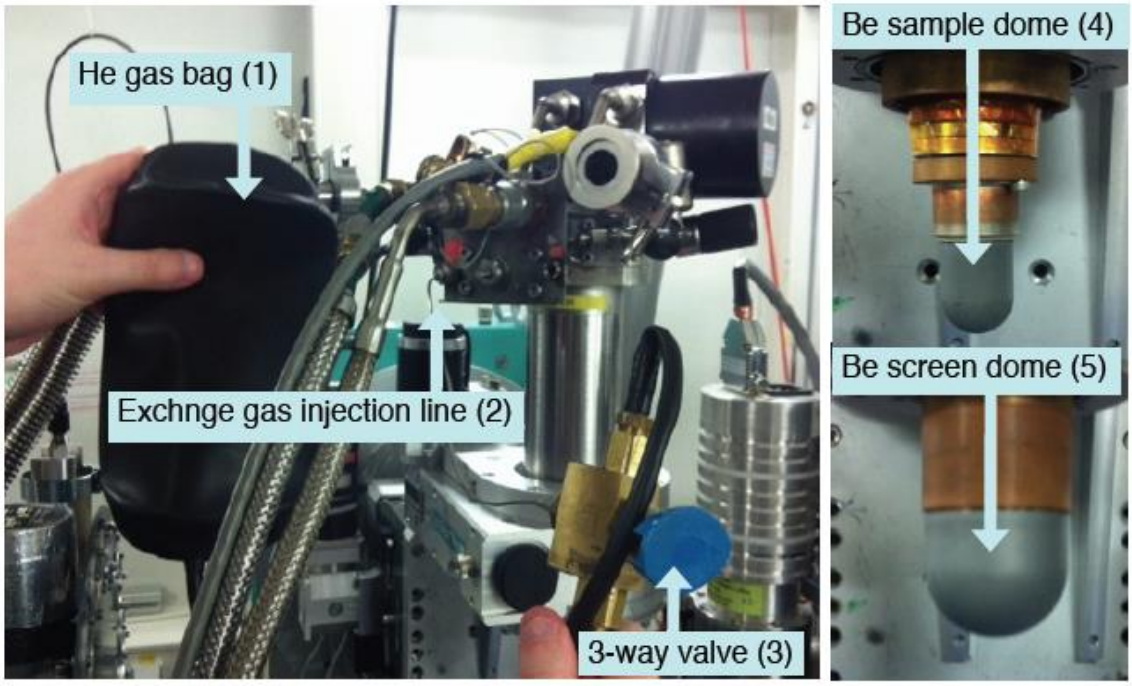


Fig.1 General picture of the displex head connections.

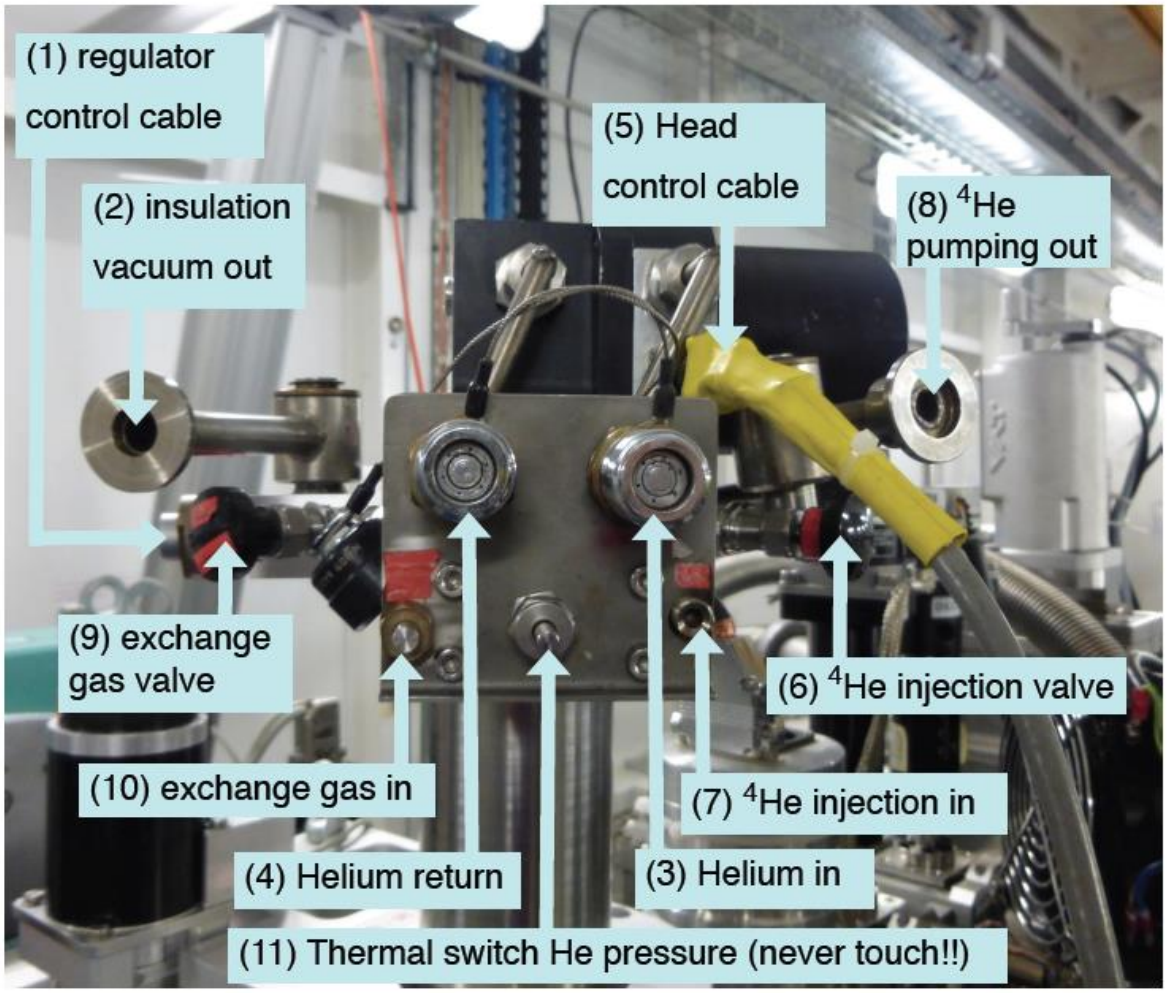


Fig.2 General picture of the displex head connections.

INSTALLATION

- Connect the 340 lakeshore temperature controller to the cryostat, Fig.2(1).
- Pump the insulation vacuum in the displex, Fig.2(2).
- Connect the chilled water pipes to the compressor, taking care about the in and out taps.
- Connect the cold head with the compressor's 2 Helium flux lines, taking care about the helium in (green, Fig.2(3)) and the return (red, Fig.2(4)). Connect also the controller cable on the diplex head, Fig.2(5).
- Connect the carbon trap to the 4He gas bottle, which is connected to the correct 40 bar regulator. Notice that for the moment the carbon trap is still at room temperature.
- Connect to other end of the carbon trap to the cryostat without tightening (we allow a small leakage).

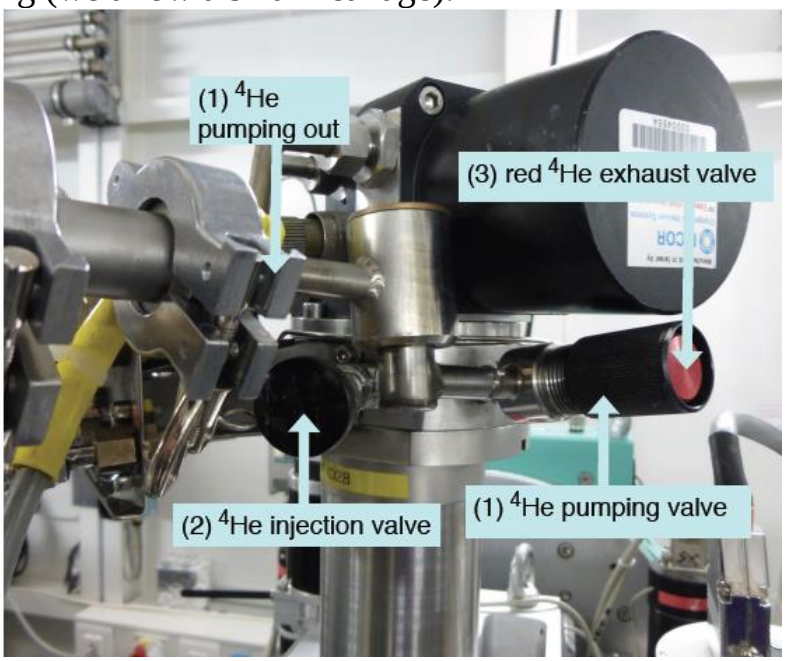


Fig. 3 4He injection line scheme.

- Be sure that the injection valve of the 4He is closed on the cryostat, Fig.3(2). Start the circulation of the 4He gas; this will purge the injection line and the carbon trap. Continue this purge for at least 1 minute, with the 4He pressure of a 1-2 bar (as determined by the 4He regulator on the bottle).

- Slowly place the carbon trap into the filled liquid nitrogen dewar, Fig. 4, and leave the 4He gas to circulate for an additional minute.

- Tighten the connectors of the injection line on the cryostat's sides. Check for leakages on the whole injection line, pay particular attention to the connections and places that have been wielded or soldered (use the gas leakage detector fluid "Mille bulle".
- Purge the cryostat's internal injection line. This is done by first opening the 4He injection valve Fig.2(2) on the cryostat, and then starting to pump the flexible helium tube. When the tube is purged, open the helium pump valve, Fig. 2(1). Continue to purge the system for at least 1 minute and then close the helium pump valve. Break the vacuum inside the helium tube.
- Switch on the compressor and set the injection pressure to 20/25 bars with the helium bottle's regulator.

OPERATION

Once the compressor has been switch on the cryostat will start to cool.

The complete cooling operation takes approximately 3 hours. After 2 ½ hours the displex reaches a plateau region between 20 K & 15 K, and after about half an hour the temperature drops rapidly to about 4.5 K. At this stage the injection pressure must be reduced to 8 bar, this is the normal pressure of operation.

We have two modes of operation :

T>4.3K:

It is not necessary to pump on the Joule-Thomson evaporation stage, and we leave the He pumping valve close (Fig.3(1)). The gas flows through the red evacuation valve which is positioned in the bottom of the handwheel. In this configuration we can work between 4.3 K and 300 K.

T<4.3K:

We lower the pressure behind the Joule-Thomson gate by using the primary pump (single stage, 12 m^3/h minimum). With this configuration we can reach a base temperature of 1.8 K. In principle it is possible to lower the temperature to 1.5 K, but only for a duration of 2 hours. This is done by lowering the injection pressure to 2 bar. After this time the temperature will increase to 10 K, and it will be necessary to increase the injection pressure to 8 bar, in order to lower the temperature again.

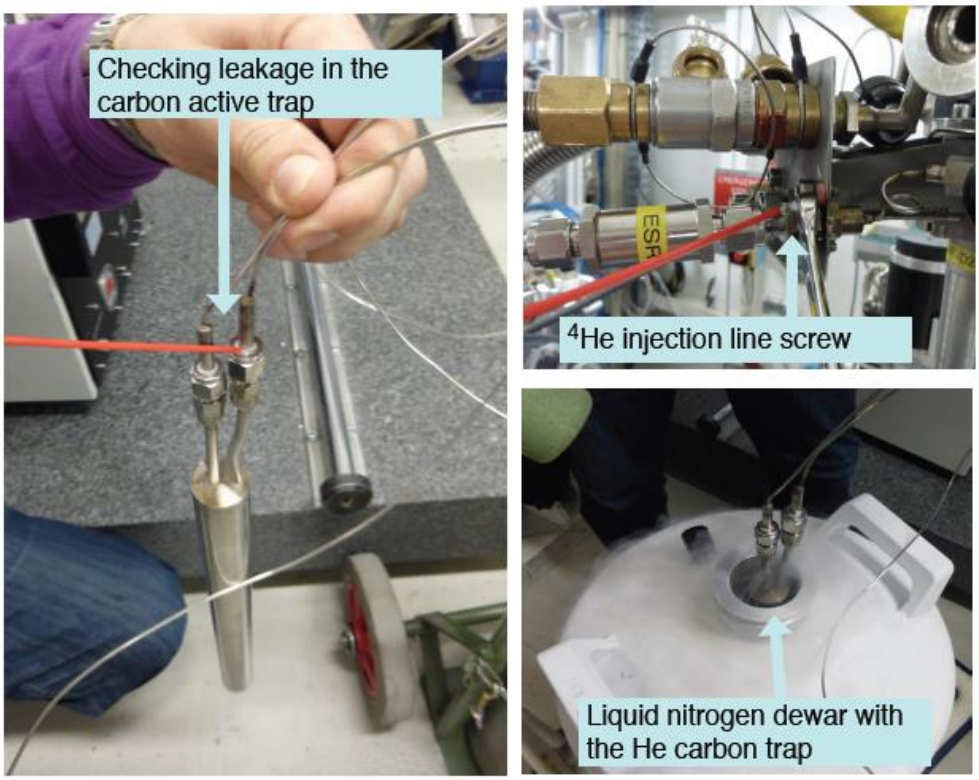


Fig. 4 Helium injection line setup.

MAINTENANCE OF CARBON TRAP.

Every 2 weeks, or in the case of a long shutdown, it is necessary to clean the cold carbon trap (see fig 4). The operation is as follows:

1. Close the 4He injection valve on the cryostat and close the 4He bottle.
2. Losen the injection line on the cryostat in order to de-pressurize and avoid a pressure increase during the baking of the carbon trap. DO NOT RETIGTEN THE INJECTION LINE.
3. Remove the carbon trap from the dewar, and heat with a hot air gun at 200-300ºC. Continue to heat for 2-3 minutes.
4. Open the 4He bottle and allow the Helium to flush the hot carbon trap, continue to flush for 2-3 minutes.
5. Place the carbon trap back inside the liquid nitrogen dewar. Wait for 1 minute and then retighten the injection line on the cryostat. Then open the 4He injection valve, Fig.3(2).

If for any reason you need to de-connect the injection line, you must remove the carbon trap from the dewar and repeat the heating operation that is described above. In fact, if the circuit is open to air the carbon is rapidly saturated and will become inactive.

You must fill the liquid nitrogen dewar in order to keep the carbon trap at a low temperature.

CONTROLLING TEMPERATURE VIA THE SPEC SESSION

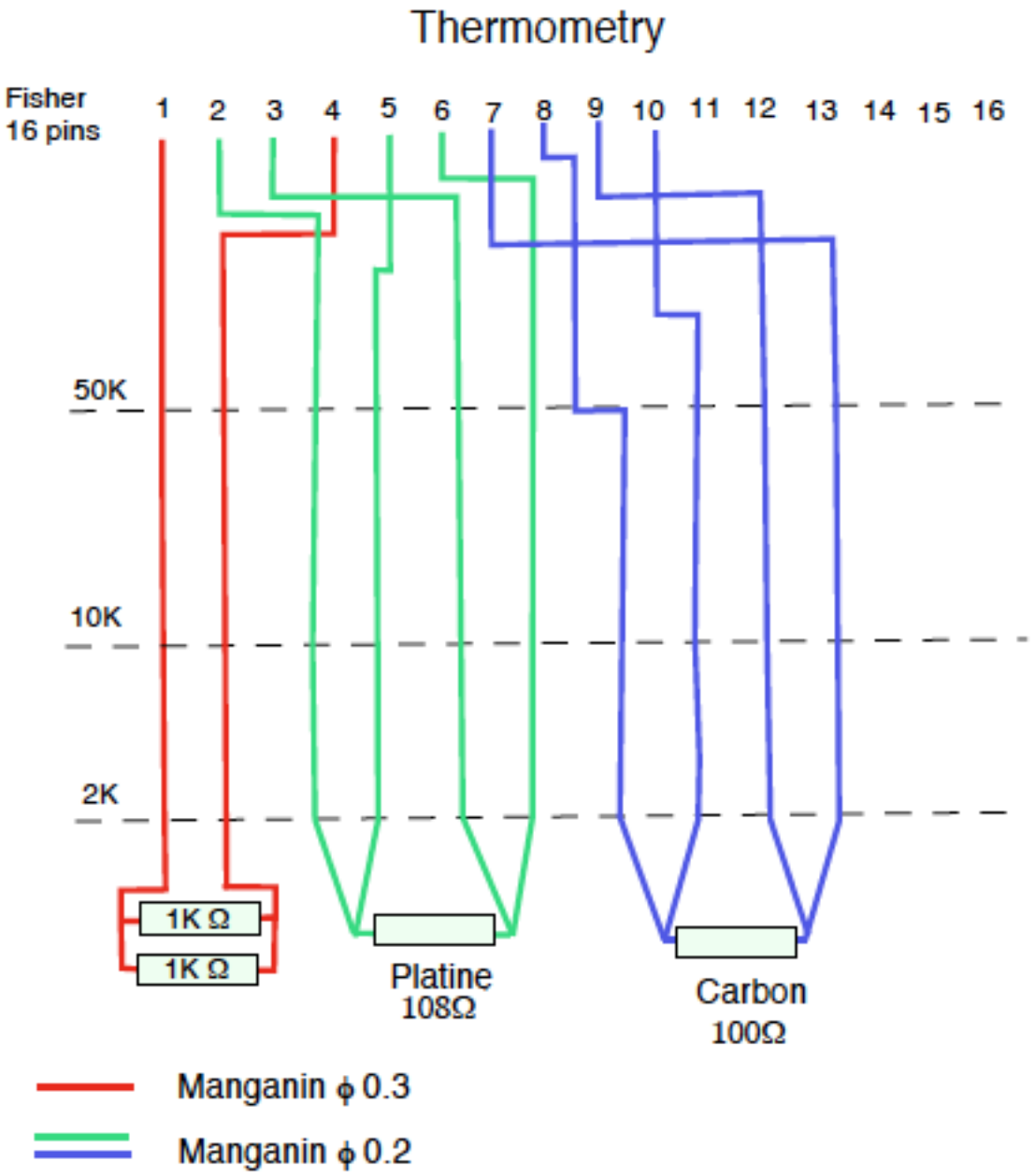
The temperature of the sample can be controlled in the spec fourc session, via a lakeshore 340 device.
The cryostat has two temperature sensors.
Temp A: a high temperature (>50 K) PT-100 type sensor.
Temp B: a low temperature (<50 K) carbon/glass type sensor.
The values of these sensors can be seen with the “ct” (last values on the list) or “te” spec commands.
The lakeshore 340 has several useful spec macros, a list and brief description of these macros are displayed with the ”lshelp” command. A fuller description of the macros follows.
“lsontemp/lsofftemp” This switches on/off the temperature logging in the spec session.
“lsonreg/lsoffreg” This switches on/off the temperature regulation by the heater.
“te [setpoint]” The command by itself will display the temperature & resistance of the two sensors and the setpoint. ANYTHING ELSE? The command followed by a number will change the setpoint of the cryostat. Note that if ramping is turned on, the setpoint displayed on the screen will correspond to the ramp setpoint not the final one.
“lsshow” Displays all parameters of the controller. WHAT ARE THE PARAMETERS?
“lsramp [rate]’’ Sets the setpoint ramp rate in degrees Kelvin per minute.
“lsonramp/lsofframp” Switches on/off the setpoint ramping option.
“”lsheater [range]” Sets heater range, 0=off, 1=1.8 K to ?, 2=? to ? 3= ? to ?, 4= ? to ?, 5=170 K to 300 K.
“lsmode [mode]” Sets the PID control mode. Use only 2=zone or 4=auto modes. FYI, if using auto mode, makes sure that heater has been switched on and is in the correct range “lsheater [range]”.
“lscontrol [*A* or *B*]” Changes the control sensor. Use A (TempA) for T>50 K and B (for TempB) for T<50K, which correspond to the Pt100 and the Carbon glass, respectively. Notice that the calibration curves must be entered manually on the lake shore, and they corresponds to the ILL-01 and ILL-02, respectively.
“lscontrast [contrast]” sets the contrast on the lakeshore display.
“lslistallcurves” Reads and displays calibration curves of the temperature sensors. DO NOT CHANGE.
“Lswritecurve [crv] [file]” Writes curve to instrument from file. DO NOT CHANGE.

WARMING THE CRYOSTAT.

The following section gives instructions for warming the cryostat to room temperature. This is done before changing samples, or at the end of the experiment.

- Turn off the compressor, and then set the temperature to 300 K on the A sensor channel (Pt 100 sensor).
- Release the pressure in the 4He bottle's regulator. When the pressure is below few bars, you can safely close the 4He injection valve (Fig.3(2)) and the 4He pump valve (Fig3(3)). Afterwards you may disconnect the 4He injection line from the displex.
- Wait until cryostat has reached room temperature with the aid of the heater. This normally takes 1hour.
- Turn off the vacuum pump, open the vacuum to air (Fig.2(2)) and carefully remove the Berllium domes, using gloves.

TERMOMETRY


Thermometry
Fisher
16 pins
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
50K
10K
2K
1K Ω
1K Ω
Platine
108Ω
Carbon
100Ω
Manganin ϕ 0.3
Manganin ϕ 0.2

B) ST15 cryostat

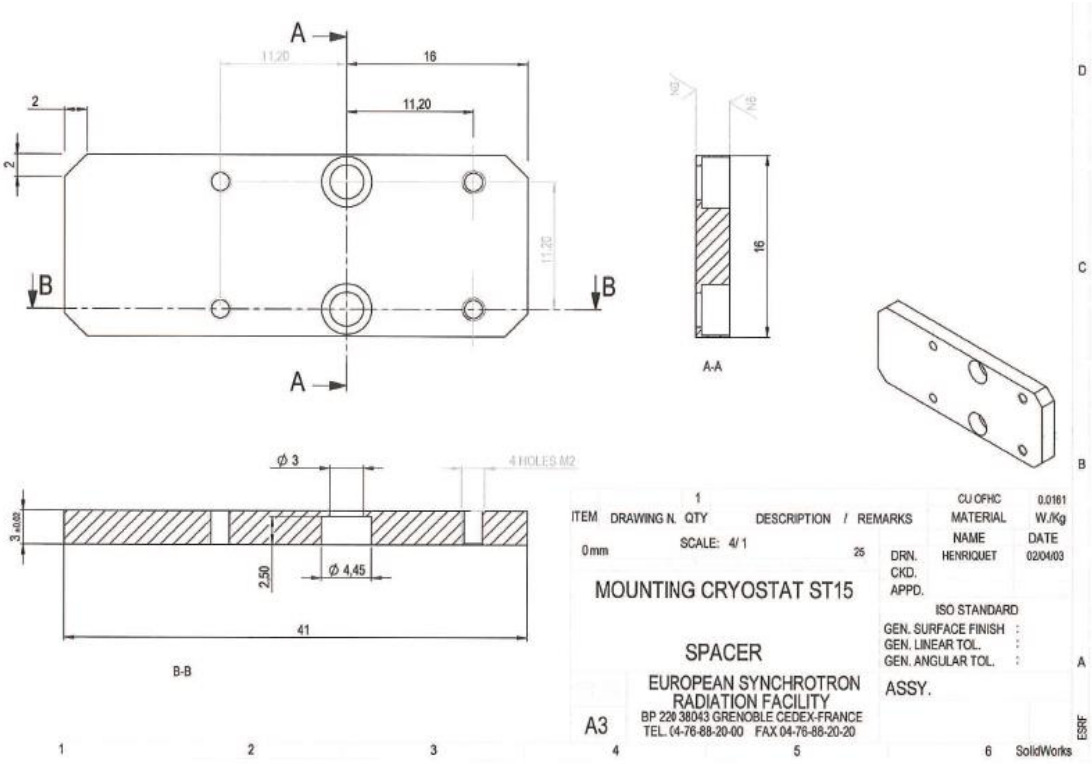
Hardware

The device is owned by ID28. It is controlled via GPIB, and in order to work properly, the control unit (located in the second electronics rack in EH1) needs to be switched on.

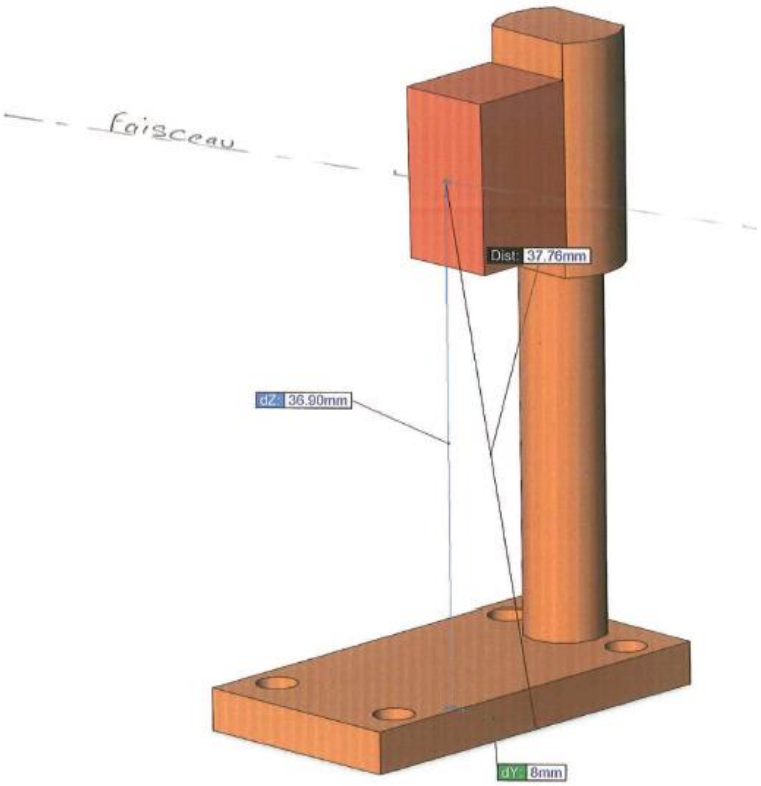
The base temperature on the sample is in the order of 13 -14 K, with a thermal screen it could be as low as 10 K.

The cold head induces vibrations, which are in the order of 25 μm x 7 μm (hor. x vert.)

There are numerous mounts, a proto-typical example is given below.



Technical drawing of the cold head base plate, in which the sample holder needs to be mounted.



Example of a sample holder (nota bene, depicted upside down), to be mounted on the cold head base plate (see above). The distance between the base plate and the center of the sample should be ***36.90 mm.***

Control in FOURC

Pseudo-motor for temperature:	cryo [Kelvin]
Counters:	T1 cryo (base plate copper finger)
	T2 cryo (sample temperature)

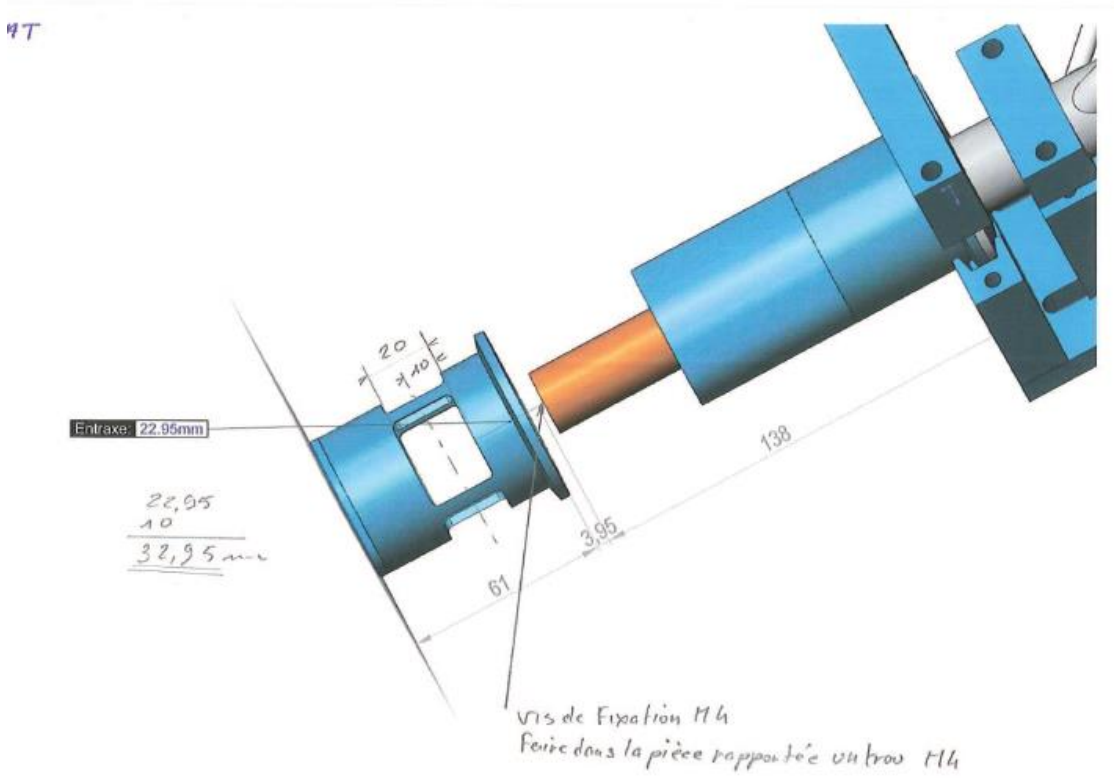
C) Miniflow cryostat

The device needs to be reserved from the Sample Environment Service.

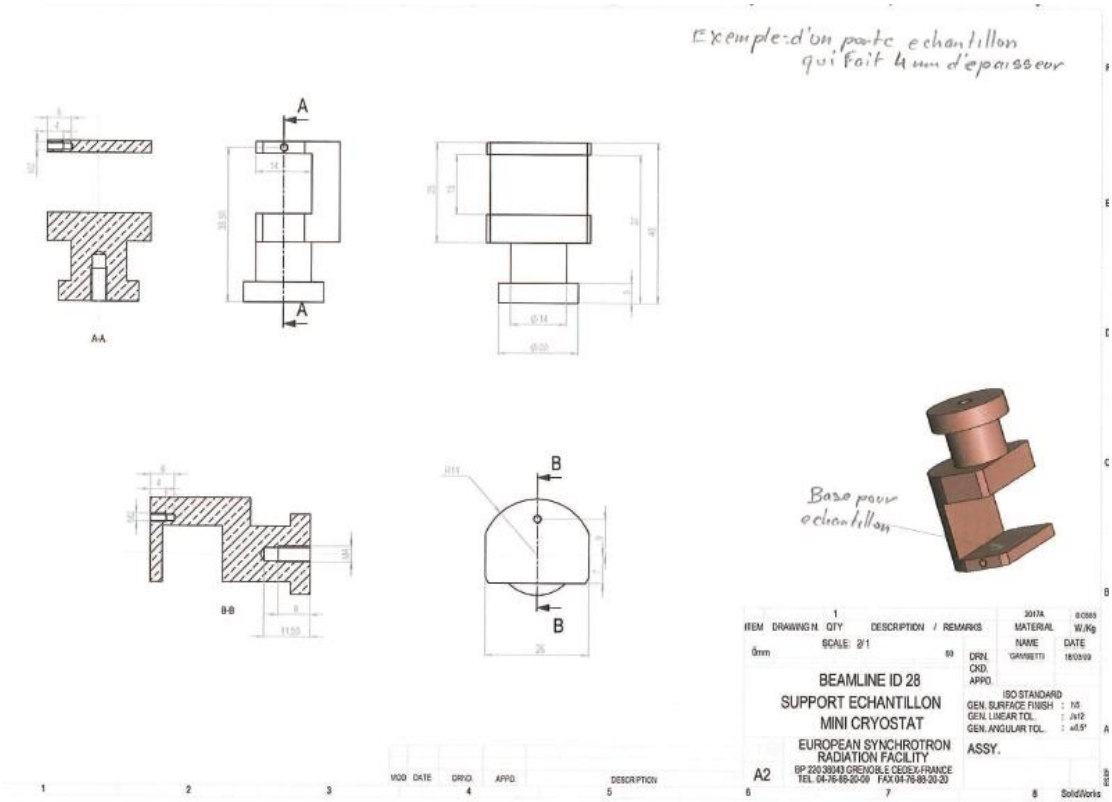
Hardware

The device can be controlled via ……

There are numerous mounts, a proto-typical example is given below.



Perspective view of the miniflow cryostat with some relevant distances. The distance between the copper base plate and the center of the sample should be ***32.95 mm.***



Example of a sample holder for the miniflow cryostat. The sample holder needs to have a threaded M4 hole in order to be mounted onto the cold finger

.

D) Eurotherm

Most of the temperature devices are controlled via the Rocket port interface.
Channnels 1 – 8: RS422
Channels 9 – 16: RS232

pseudo motor	pseudo counter	rocket port #	interface cable #	temperature device
teuro1	teur1pv	1	15	Id28 Eurotherm in black rack
teuro2	teur2pv	2	16	
teuro3	teur3pv	9	19	
teuro4	teur4pv	11	20	

FOURC> umv teuro1 to 300 set the setpoint to 300 (K or degrees – internal setup)

Setup to be defined for crysotream, heat blower, miniflow cryostat, and low T high P cryostat

E) Oxford Cryostream

Usage hints
Before using switch off the ventilator, that cools down the detector array.

Hardware
Cable #20, connected to port 11 in the RS232/422 interface rack in CC1.

Temperature control

FOURC> stream_enable	enables operation of cryostream
FOURC> stream_disable	disables operation of cryostream
FOURC> newstreamramp <setpoint/K> [<rate_K_per_hour>]	Heating, second option specifies the rate
FOURC> newstreamcool <setpoint/K>	lowers setpoint temperature
FOURC> newstreampurge	returns device to room temperature
FOURC> newstreamhold	keeps device at desired set point
FOURC> newstreamstatus	indicates status of device
FOURC> stream_save_temp	saves the T in the spec file

Counter: CryoStream

F) Heat Blower

Hardware
Cable #19, connected to port 9 in the RS232/422 interface rack in CC1.

Software inside FOURC
There are commands to enable/disable the motor and sensor corresponding to the mini gas blower. They can be either updated/modified by typing ‘setup’ and then editing the setup file, or by line command in FOURC.

```
# Gas Blower
#
# enable(0)/disable(1) motor
motor_par(teuro3, "disable", 0)
# enable(0)/disable(1) counter
counter_par(teur3pv, "disable", 0)
```

Motor: teuro3 ! umv teuro3 xxx [Celsius]
Counter: teuro3 PV

G) Lakeshore temperature controller

Motor: ls336 ! umv ls336 xxx [K]
Counters: LS336 c0 & LS336 c1

FOURC> lsontemp ! activates Lakkeshore controller
FOURC> lsofftemp ! de-activates Lakkeshore controller

After installing the Lakeshore 340 (which is connected to the GPIBID28E box), SPEC should be setup to communicate with Lakeshore. If this is not done yet, you cannot read the temperature by **ct** or **te** command in FOURC.

To setup Lakeshore:

+ Open a new sheel (terminal)
+ Login as blissadm:
  %su - blissadm
  %password: spec92
+ Then go to:
  %cd /users/blissadm/local/spec/spec.d/fourc
+ edit the setup file:
  %nedit setup

Find the Lakeshore 340 device, comment/uncomment the commands below it and save it. Close the file.

Type: **resetup** in FOURC. Done.

5. SINGLE CRYSTAL PRE-REQUISITES, PREPARATION AND ORIENTATION

A) **Pre-requisites**

- crystal structure is known

  ABSOLUTE MUST within the basic structure
  poorly known superstructure, modulation, etc. can be tolerated

- crystal is of good quality

  - untwinned – should be checked with x-ray tube and avoided whenever possible
  - with reasonable rocking curve (up to 1 degree is tolerated, depending on the experiment)
  - without surface impurities (any kinds of glue, damaged layer, etc.) – washing and etching are prescribed

- crystal is of the appropriate size

  - for the transmission experiment the signal is maximum when the sample thickness is equal to the absorption length for the given wavelength – use http://www.cxro.lbl.gov and check that the density is correct.

  - for the reflection experiment the beam footprint and/or focal depth should merge with the reflecting surface size

- crystal is of the appropriate orientation

  - the orientation should be known at least approximately (axis of needle crystal, two faces of regularly shaped sample, etc.)

  - for the experiment in reflection the facets should be compatible with the reflection geometry for the selected phonon branches

- crystal is properly mounted

  - high pressure – refer to the DAC constrains

  - low temperature with cryostat – refer to the ST15 or mini-flow cryostat mounts

- “conventional” single crystal work with cryostream or heat blower – use standard brass holders (http://hamptonresearch.com/product_detail.aspx?cid=19&sid=115&pid=338) 1/2" length and 1/8” diameter with glass fibers (ambient and low temperature) or quartz capillaries (heating)

hints:
- to minimize the vibrations in the cryostream flux, use the conical fibers

- use the thermally stable glues/ciments when you expect to heat up

- keep the holder-to-sample distance within 6 mm

- if you want to keep the sample far from the goniometer head (very high temperature? ice formation danger? Geometrical constrains?), take 49 mm head without the extender and mount the sample at slightly below 21 mm. Sample should be of roughly correct orientation, otherwise when the arcs are moved, the internal translations may not bring the sample back to the spectrometer center

B) Preparation: Sample Pre-alignment

Basic mount (heavy load theta) – goniometer head axis is horisontal

put the alignment pinhole

umv th 0 chi 0 sax 0 say 0.1 saz 0

put the PRL system (when available) and focus it on the pinhole

replace the pinhole by the goniometer head with the sample

if the sample is thick, umvr sax –(thickness/2)

use the big motorised head translations to bring the sample surface to the focal point

if the sample is thick, umvr sax thickness/2

Additional theta mount ADTH – goniometer head axis is vertical

put the alignment pinhole

umv th 0 chi 0 sax 0 say 0.1 saz 0

put the PRL system (when available) and focus it on the pinhole

replace the pinhole by the adth mount, put the goniometer head with the centering pin

move the adth axis to the center of rotation of spectrometer

umv sax -0.25 say -0.15 (or use better estimate)

uncouple the motor from the rotation stage

mark the position of pin for adth = “0” and “180” in the head frame

use the head translation to bring the pin to the middle

mark the position of pin for adth = “90” and “270” in the head frame

use the head translation to bring the pin to the middle

use sax translation to get a sharp image of the pin

use say translation to bring the pin to the pinhole reference position

check the absence of precession and repeat, if needed

replace the goniometer head with the centering pin by the head with sample

if the sample is thick, umvr sax –(thickness/2)

use the head translations to bring the sample surface to the focal point, check the precession and refine as for centering pin, if necessary

if the sample is thick, umvr sax thickness/2

couple the circle to the motor in the convenient position

switch off the adth power

attach the cable

turn on the adth power

make sure that adth motor (47) is properly configured

Additional chi mount (use with DAC)

<u>Preliminary info:</u> The rotation stage in which the DAC is mounted, allows for manual translations in order to bring the sample into the center of rotation of the "additional chi" mount. To this end, you have to slighly unscrew the inner circle, holding the cell. Four small screws without head, radially to the outer circle then allow translating the DAC. Note that there is some slack, and once close to the ideal position, you need to start tightening the screws of the inner circle. Be patient, it takes a while to d the fine positioning.

umv th 0 chi 0 sax 5 say 0.1 saz 0 (standard membrane cell)

mount the rotation stage and the sample, check the movement range, safe for the capillary, if applicable

put the PRL system and focus it on the sample

untight the screws blocking the translation

uncouple the motor from the rotation stage

mark the positions of the sample for the rotations "0", "90", "180" and "270" (relative, convenient to refer to the centering screws)

move the sample to the center of the square with the help of the small screws without head.

check the precession, repeat if necessary

tighten the screws blocking the translation

couple the motor to the rotation stage

switch off the adth power

attach the cable

turn on the adth power

make sure that adth motor (47) is properly configured

Alignment in the direct beam

Preliminary info: the alignment in the direct beam is rarely necessary when the optical alignment was successful, except for the DACs.
You can use either newdet or deta2 - refer to the user manual for the newdet; to use the deta2, make sure that the absorber (wheel) is at large enough value and tth is at 0 (otherwise umv tth 0)
Alignment is efficient only if the sample is absorbing and the beam size is comparable with the sample size (otherwise umv i1shg (horizontal size) i1svg (vertical size) to reduce dimensions of the incident x-ray beam onto the sample)
If the sample is poorly absorbing, use fluorescence detectionwith silicon diode (see user manual)

localize the sample using say and saz scans (a is positive number)

dscan say a –a N time

dscan saz -a a N time

umv th -5

get say1 sample position

umv th 5

get say2 sample position

umvr sax (say2-say1)/(2sin(5 deg))

C) Orientation Matrix

NB: for the use of th the orientation should be known in advance, the accessible angular range is very small
find the data on the crystal structure (CarIne is useful program for further use)

1. Alignment with CCD

mount the CCD camera, if tth is at 0 (otherwise umv tth 0)

switch on the camera (CamWare64 V3.13)

activate the software in "live" mode with 0.25-0.5 s acquisition time

remember the collision-free range of motor movement (no limits for adth unless you have additional equipment in, -15 to +15 th, capillary issues for additional chi)

start CCD.exe with proper parameters

OR

start "**SensiCam_Calibration**":

You can use this program to get the tth and chi values corresponding to every pixel of the CCD (Si powder patterns were used for the CCD calibration).

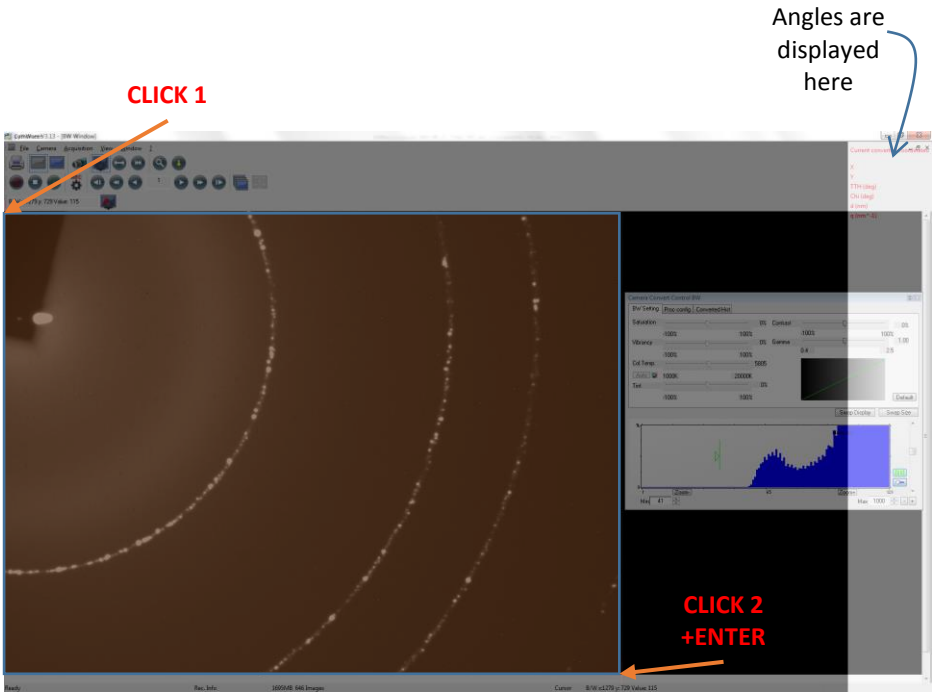
2. Start the SCC.py and choose the right beam configuration (torus, multilayer etc).

3. Press F5 to open a transparent window and overlay it to the CCD camera window (CamWare V3.13).

4. Define the CCD pixel limits by clicking (single click) 2 points at the upper left and lower right corners of the CCD window, as shown in the next picture. Press ENTER.

5. The chi and tth values corresponding to the cursor pixel are shown on the top right of the window.

Additional theta mount ADTH – goniometer head axis is vertical
for convenience (if no counterindications):
set adth 0; set_dial adth 0; set_lm adth -300 300



Overlay of the CCD CamWare64 V3.13 window with the "SensiCam_Calibration" transaparent window.

low complexity task: alignment of the sample with known vertical axis

rotate the adth in positive direction until the spot of interest appears in the scattering plane

refine adth, note the deviation from the scattering plane (CCD.exe OR with SensiCam_Calibration)

rotate the adth in positive direction until the second spot of interest appears in the scattering plane, ideally orthogonal to the first one

refine adth, note the deviation from the scattering plane (CCD.exe)

If the ambiguity with the indices can be present (different intensities for non-equivalent spots with the same |Q|, monoclinic cell, two spots not lying in high-symmetry planes, etc.), use any software to check the consistency

If the deviations from the scattering plane are too large, correct with the head arcs and head translations, then repeat the spot search in the proximity of the reference positions

If you are not sure, what is |Q| for a given reflection (few reflections with nearly the same |Q|, badly calibrated CCD, …), use for the cross-check the adth spacing between two reflections (can be calculated for known structure with the help of CarIne or any other software); HKL, 2H2K2L, 3H3K3L series can be helpful

high complexity task: alignment of the sample with unknown orientation

try to find the reflections with small |Q| first, there is les ambiguity with the HKL identification

check if the configuration is suitable for the measurement

re-glue the sample or take another one if needed

repeat the exercise until you get two basic reflections close to the scattering plane

Additional chi mount (DAC)

for the convenience (if no counterindications):
set adth 0; set_dial adth 0; set_lm adth -300 300

for given chi (adth motor) move th within the available range until you get the spot of interest

if unsuccessful, move adth by 45 deg and move th again within the available range

when you see the spot of interest, bring it to the scattering plane, correcting the th

note the values

find another spot, ideally orthogonal (adth ±90 deg), note the values

if the ambiguity with the indices can be present (different intensities for non-equivalent spots with the same |Q|, monoclinic cell, two spots not lying in high-symmetry planes, etc.), use any software to check the consistency

2. Alignment with the analyzers

make sure that the absorber (wheel) is at large enough value, monot is at T0 (otherwise umv monot T0)

umv tth (calculated 2theta/ estimated with CCD) adth (estimated with CCD) chi (estimated with CCD)

umv a2hgap 60 a2vgap 60

move chi ±1 looking to the LED signal of the discriminator module

if there is no signal, move chi ±2 looking to the LED signal or reduce the absorber thickness

if there is still no signal, move chi ±3 or reduce the absorber thickness or try to move slightly adth and tth

when the intensity is high (LED does not blink) umv a2vgap 40

if signal disappears umvr chi ±0.3

when the intensity is high (LED does not blink) umv a2vgap 20

if signal disappears umvr chi ±0.3

when the intensity is high (LED does not blink) umv a2vgap 10

if signal disappears umvr chi ±0.3

umv a2vgap 60

dscan adth –a a N time umv adth (center)

dscan say a –a N time umv say (center)

dscan saz –a a N time umv saz (center)

umv a2hgap 5

if signal disappears umvr tth ±0.1

dscan tth a –a N time umv tth (center)

if the rocking curve looks strange and the sample is large at least in one dimension, try to explore different points of the sample

make final rocking curve

dscan adth –a a N time

print the positions

pon; wm sax say saz tth adth chi; poff

go to the second spot, repeat the alignment

refine the lattice parameters and check the compatibility of two angular positions with available software (Mathcad or ID28 Macro Maker or Spec; see the following sections)

if OK, consider the possible ambiguity in the HKL identification; if it is possible, take into account the intensities and/or CCD diffraction patterns and/or the calculated position of third diffraction spot

if say/saz for two reflections are substantially different, implement the precession correction software (Mathcad or ID28 Macro Maker; see the following sections)

follow the same guidelines when additional chi is installed

D) Crystal orientation with Mathcad

Basic procedure

1. Create a folder with an appropriate name on the PC Gagarin2 (to the right of the control desk). Copy there three files:

orientation_template.xmcd
rotation_definitions.xmcd
elastic

and then you can change the name of the 1st file to whatever you like, as the three files above are permanently stored in My Documents/Orientation/Template

2. Try to find two reflections

3. Enter the global parameters into the orientation script

- lattice parameters [Angstroem, degrees]
- wavelength (0.7839, 0.6968, 0.5701, 0.522)
- temperature [K]
- analyser slit opening [mm]
- distance to analyser [RA in mm]
- file name for elastic tensor reading ["elastic" by default]
- for each reflection (h k l) and angles (th/adth), tth, chi
- phi value, constant phi mode only
- define geometry: standard or additional th (adth)

4. Orientation matrix refinement

Play with the initial values of solver until refined (h k l) and angles for the two basic reflections get close to the initial ones.
1st set usually works fine with seros
2nd set try thinp first (0.1, 1, -0.1, -1 – typical set)
If absolutely unsuccessful, think about the correctness of given (h k l).

5. Precession implementation:

If/when you have found the good sample position (sax, say , saz), you can try to keep this position with the x-ray beam by using the precession subroutine.

- for given angles (direct beam or Bragg reflection) provide the position
- when using additional theta, provide the adth rotation center (sax and say)

- click on (=activate) the “precession” switch, then sample position will be stored in macro files.

6. If everything looks satisfying, enter the desired (h k l)

Check whether the refinement did work: compare the refined (h k l) with the initial one and check whether the angles are not strange.

You can read now:

- angles / stored automatically in *.prn file
- optimal sample position (if you use precession) / stored in the same file
- list of (hkl) for all analysers / stored automatically in *.hkl file
- list of qx, qy, qz, |q| for 5 analysers / not stored

7. You can write the macro semi-automatically

- select the energy of the center of the scan, half-width [meV] and time per point [s]
- click “Add scan” twice to add the scan to the macro “macro.prn” by default

…you will see that the word “SCAN” changes colour from black to red – when you change parameters of scan, it must be red, otherwise you will store a lot of intermediate “scans”

- do not forget to remove all the “ from macro.prn before running it in FOURC

Example: open file with MSWord and press “Ctrl+Shift+A”

<u>Sometimes useful options</u>

1. Spot identification (useful with CCD) or checking the quality of orientation matrix refinement

- just enter angles and read (h k l)

2. If you prefer to work with th/tth geometry (no adth!) with constant phi and chi, you can use proposed phi/chi offsets.

3. If you want to visualise the resolution in reciprocal space

- check the analyser opening
- check if the closest Bragg spot is well chosen

Now you can rotate the obects with the mouse. If the Bragg spot is too large/small compared to the resolution rectangle, play with SR parameter.

4. If you are mainly interested in the elasticity and you have some ideas about the values of the elastic moduli, you can get approximate energies and relative intensities of acoustic phonons.

- check if closest Bragg spot is well chosen
- check the temperature
- check the content of "elastic" file
  - 1st line: density (g/cm^3) / number of moduli / 0
- following lines: i / j/ C_{ij} [GPa]

Read the energies and relative intensities of phonons

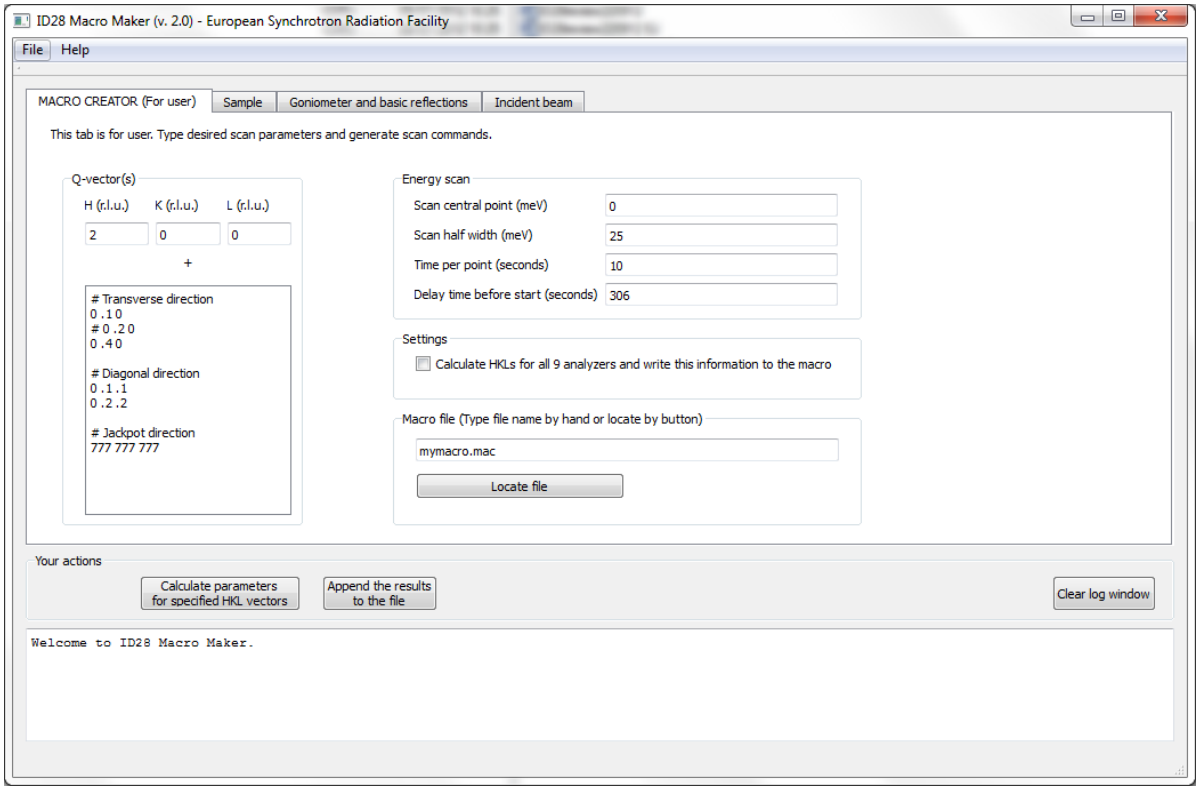
E) ID28 Macro Maker Manual

Abstract

The program "ID28 Macro Maker" helps you to create macros for the IXS experiment. It has a very simple and intuitive interface allowing non-experienced user to easily create macros. Experienced users (local contacts) can configure the program to operate with a variety of goniometer configurations.

How it works (for user)

The program is already configured by you with assistance of the local contact for this particular experiment. If it is not – see next sections. If you accidentally close the window, just open it again – all settings will be restored.

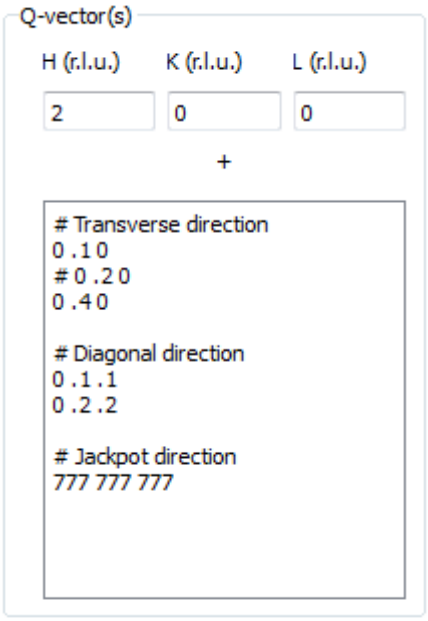


Macro Creator tab

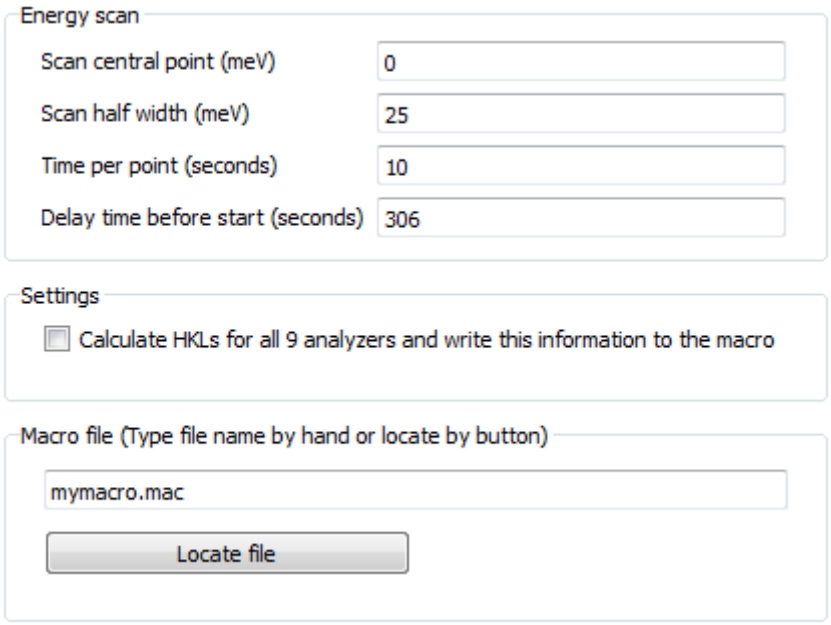
In tab "MACRO CREATOR (For user)" you specify 4 things you need:

1. The set of Q-vectors (in hkl) for which you want to measure IXS.
2. The parameters of energy scan.
3. The settings (just one for now).
4. The file, to which the macros will be appended.

The first panel is box “Q-vectors”. In the larger text edit box you specify a list of HKL-points. They will be summed up with the main point (usually a reciprocal lattice node) to form a list of points in which to make IXS. In the example below the measurements are supposed to happen in (2 0.1 0), (0 0.4 0), (0 0.1 0.1), (0 0.2 0.2) and (777 777 777) points. Point (0 0.2 0) is skipped by “#” sign. The list of HKL points can be copy-pasted.



In the right-side panels you specify the parameters of energy scan (self-explanatory). The checkbox in “Settings” panel allows you to decide if you want the HKL-positions of all 9 analyzers to be shown in log file and dumped to macro as comments. This is a matter of taste. In the bottom-right panel you select the file, to which you want the macros to be appended.



Actions

Below the “Marco Creator” tab resides a panel with buttons. Even more below is the log window. You may clear it by one of the buttons, if there is too much information.

Button “Calculate parameters ”

When you press this button the program does the following:

1. Calculates UB matrix on the basis of 2 basic reflections (all the parameters for that are in other tabs). If these parameters are inconsistent with reality, the program points to that and stops, until you make these parameters reasonably reasonable. If you specified to use “Precession correction”, the program also calculates the off-centering of the crystal relative to the goniometer center for further corrections.
2. Calculates, for each HKL vector, the rotation angles and checks, if they are within software limits. All relevant information is written to the log.
3. Prepares a summary on the calculations made and saves the results internally. After you may ask the program to compose a macro on their basis.

After calculations you see in the very end of the log a summary.

```
RESULTS:
     H     K     L Geom. solutions   Sol. in range    Status
     2   0.1     0                2               1    OK, ready to write a macro
     2   0.4     0                2               0    Error: no solutions within software limits
     2   0.1   0.1                2               1    OK, ready to write a macro
     2   0.2   0.2                2               1    OK, ready to write a macro
   779   777   777                0               0    Error: no solutions

SUMMARY:
3 of 5 calculations were succesful and can be used for writing macro.
```

Here we see, that not all the calculations were successful. There were 2 errors. For one HKL it was impossible to find a solution that is within software limits, for other – there were no solutions at all. If we scroll the log up we see the more detailed description of what happened.

```
===== Computing angles for (H K L) = (  2 0.1   0) =====

  Found 2 solutions:
            tth        adth         chi         sax         say         saz
  0:     16.2704     54.8903   -0.465733   -0.139312   -0.447006     -0.0134  ALL in range
  1:     16.2704    -141.381    -179.534   -0.159421   -0.455869     -0.0134  NOT in range: chi

===== Computing angles for (H K L) = (  2 0.4   0) =====

  Found 2 solutions:
            tth        adth         chi         sax         say         saz
  0:     16.4262     55.1055    -6.61254   -0.139301   -0.447046     -0.0134  NOT in range: chi
  1:     16.4262    -141.429    -173.387   -0.159416   -0.455877     -0.0134  NOT in range: chi

===== Computing angles for (H K L) = (  2 0.1 0.1) =====

  Found 2 solutions:
            tth        adth         chi         sax         say         saz
  0:     16.2922     57.8504   -0.424502   -0.139172   -0.447562     -0.0134  ALL in range
  1:     16.2922    -138.442    -179.575   -0.159709   -0.455378     -0.0134  NOT in range: chi

===== Computing angles for (H K L) = (  2 0.2 0.2) =====

  Found 2 solutions:
            tth        adth         chi         sax         say         saz
  0:     16.3884     60.8611    -2.43248   -0.139059   -0.448134     -0.0134  ALL in range
  1:     16.3884    -135.542    -177.568   -0.159969    -0.45488     -0.0134  NOT in range: chi

===== Computing angles for (H K L) = (779 777 777) =====

  NO solution for 2Theta - Q is too large.
```

Button “Append to file”

This button works only if you have previously managed to produce calculations without errors. If the calculations were ALL okay, then after pressing, the program will append a set of macros to the file specified.
An example:

```
226  # ------------- Thu 4. Oct 16:20:07 2012 ----------------
227
228  # Analyzer 2 at (2 0 0)
229  #   1.8140  2.0000  2.1853  2.3710  2.5546  1.9046  2.0933  2.2753  2.4593
230  #   0.0003  0.0000 -0.0004 -0.0009 -0.0014  0.0002 -0.0002 -0.0006 -0.0011
231  #   0.0233 -0.0000 -0.0281 -0.0612 -0.0988  0.0126 -0.0135 -0.0435 -0.0787
232  # Energy scan from -25 meV to 25 meV with 0.683371 energy step
233  umv tth 16.26 adth 54.86 chi 1.6 sax -0.139314 say -0.447 saz -0.0134
234  umv monot T0+0.555
235  sleep(306)
236  ascan monot T0+0.555 T0-0.555 74 10
237
238  # Analyzer 2 at (0 0 2)
239  #  -0.0249 -0.0000  0.0298  0.0647  0.1044 -0.0134  0.0144  0.0461  0.0832
240  #  -0.0010 -0.0000  0.0012  0.0027  0.0043 -0.0006  0.0006  0.0019  0.0035
241  #   1.8197  2.0000  2.1796  2.3596  2.5374  1.9076  2.0904  2.2668  2.4451
242  # Energy scan from -25 meV to 25 meV with 0.683371 energy step
243  umv tth 16.77 adth 145.13 chi 0.79 sax -0.14705 say -0.4607 saz -0.0134
244  umv monot T0+0.555
245  sleep(306)
246  ascan monot T0+0.555 T0-0.555 74 10
```

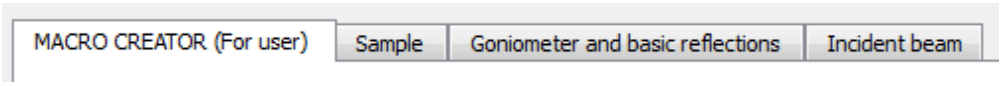
You may avoid having here all the positions of 9 analyzers by un-checking the corresponding checkbox.

How it works (configuration)

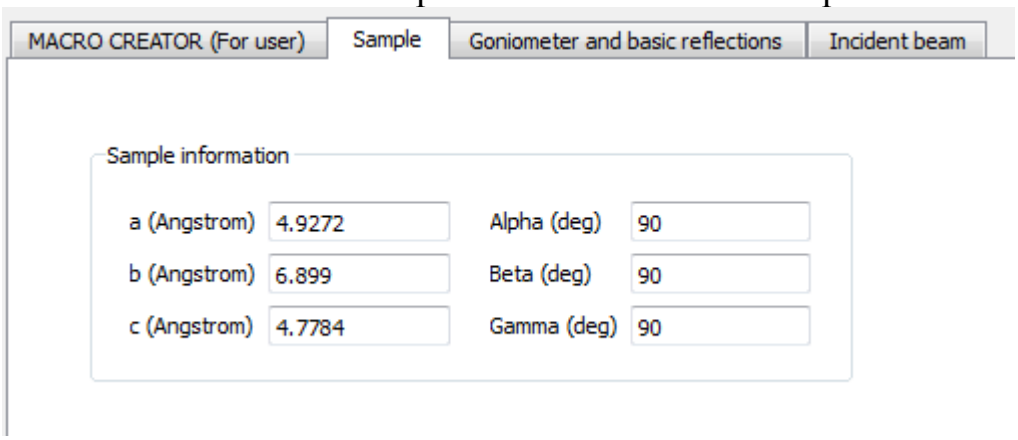
Previously, we have seen, how the program works when properly configured. Here we will see, how to properly configure the program.

Configuration windows

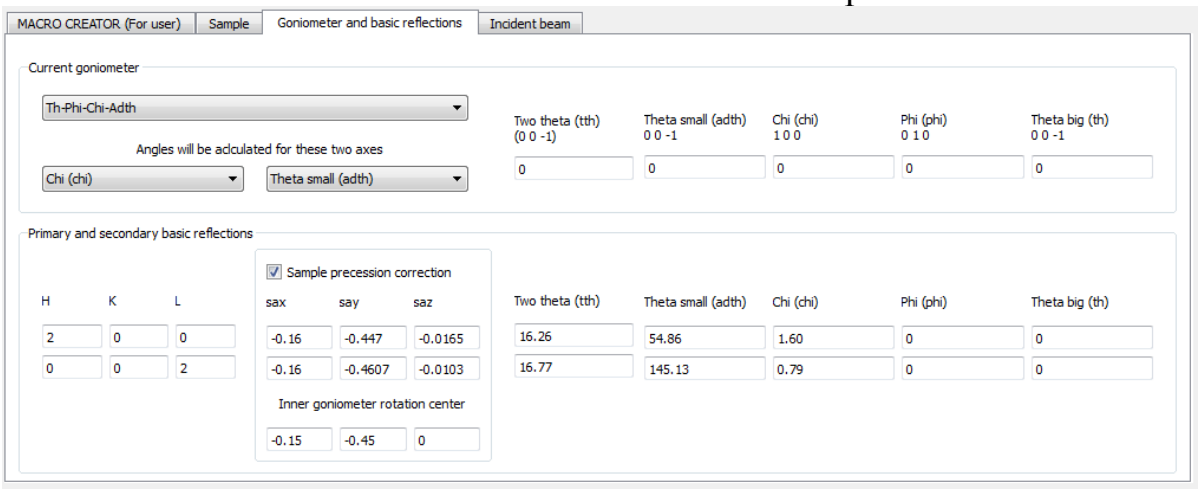
Before using "Macro Creator" tab, several tabs on the rigth have to be filled:



The information on the sample has to be filled in "Sample" tab:

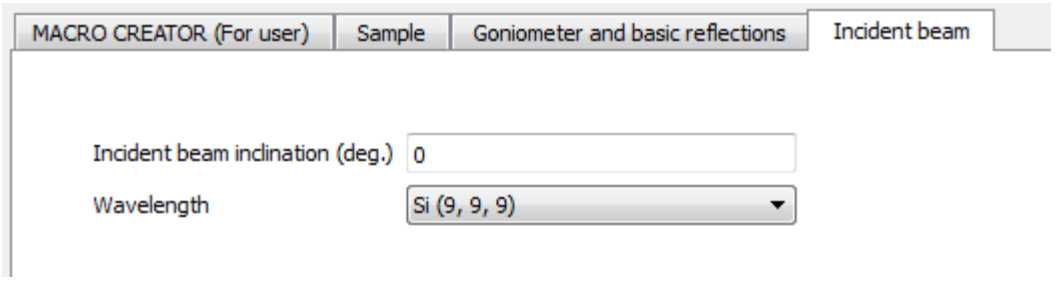


The tab "Goniometer and basic reflections" is more complex:



Here you select one of the goniometers, previously specified in XML file. Also you select the axes, for which calculations to be performed. All the goniometer axes are represented on the right side.
In the bottom part you enter the information for basic reflections. Optionally, you enter the positions of the translation box for these reflections. If the check-box "Sample precession correction" is checked, they will be used for corresponding calculations. For these calculations to

be correct you have also to specify the position of the inner goniometer relative to the outer one.
Additional settings are in the tab “Incident beam”:



Configuration files

First of all, which files are used for configuration:

1. XML file – for settings that are changed seldom.
2. INI files – they are used to save the contents of forms.

XML file

The settings, that are not assumed to change frequently are in XML file. By default it is “instrument_geometry.xml”, it is located in the same directory as the program and is loaded and analyzed on the program start. The file contains several sections. First section is dedicated to the description of goniometers, that can be used for calculations. An example is shown below.

```
<goniometer name="Th-Phi-Chi-Adth" >
    <element name="adth" nameDescriptive="Theta small">
        <angle> 0 </angle>
        <axis> 0 0 -1 </axis>
        <angle_constraint_min> -180 </angle_constraint_min>
        <angle_constraint_max> +180 </angle_constraint_max>
    </element>
    <element name="chi" nameDescriptive="Chi">
        <angle> 0 </angle>
        <axis> 1 0 0 </axis>
        <angle_constraint_min> -6 </angle_constraint_min>
        <angle_constraint_max> +6 </angle_constraint_max>
    </element>
    <element name="phi" nameDescriptive="Phi">
        <angle> 0 </angle>
        <axis> 0 1 0 </axis>
        <angle_constraint_min> -5 </angle_constraint_min>
        <angle_constraint_max> +5 </angle_constraint_max>
    </element>
    <element name="th" nameDescriptive="Theta big">
        <angle> 0 </angle>
        <axis> 0 0 -1 </axis>
        <angle_constraint_min> -15 </angle_constraint_min>
        <angle_constraint_max> +30 </angle_constraint_max>
    </element>

    <translation-box>
        <positioned-before> chi </positioned-before>
        <sax0> 0 </sax0>
        <say0> 0.1 </say0>
        <saz0> 0 </saz0>
        <sax_min> -10 </sax_min>    <sax_max> 10 </sax_max>
        <say_min> -10 </say_min>    <say_max> 10 </say_max>
        <saz_min> -10 </saz_min>    <saz_max> 10 </saz_max>
    </translation-box>

</goniometer>
```

For each of the goniometers, that you plan to use, you create a section <goniometer>. For each of the rotation axes of goniometer you write a section <element> with name, equal to the angle name in SPEC. Parameter nameDescriptive is only for nice-looking interface. After the <element>s you make a section <translation-box>. You specify to which axis the translation box is connected by tag <positioned-before>. For each moveable element, including translations, we specify the constraints. You may setup here as many goniometers, as you like, with arbitrary oriented rotation axes, including kappa geometry.

By a special section you specify the limits for 2 theta rotation:

```
79      <limits>
80          <tth>
81              <min> 0 </min>
82              <max> 45 </max>
83          </tth>
84      </limits>
```

There is an importans section with tags names starting with "human_taste_limit".

```
86      <setup>
87          <human_taste_limit_q_hkl_basic_reflections> 1e-3 </human_taste_limit_q_hkl_basic_reflections>
88
89          <human_taste_limit_u_determinant> 1e-3 </human_taste_limit_u_determinant>
90
91          <human_taste_limit_u_adjoint_inverse> 1e-3 </human_taste_limit_u_adjoint_inverse>
92
93      </setup>
```

They are used to specify the sensibility of the program to injustice. Particularly:

1. The program halts, if it discovers, that the length of basic reflection's Q-vector, calculated on the basis of sample parameters and HKL indices, is sufficiently different from the corresponding length, calculated by 2-theta angle and wavelength. The corresponding value of sensibility is in <human_taste_limit_q_hkl_basic_reflections>.
2. The program halts, if it discovers, that the U-matrix (left part of UB-matrix), calculated on the basis of 2 reflections, fails to be strict rotation matrix ($|U|=1$, $U^{T}\text{-}U^{-1} = 0$). The level of sensibility is controlled by the remaining 2 parameters.

The last section is controlling the conversion between energy and temperature for different reflections.

<u>INI files</u>

All the information, that is in control windows of the program is stored to INI files, to be recovered after program's restart. When you close the program, it saves all the content to "ID28MacroMaker.ini", and restores it, when you start the program again.

Optionally, and quite usefully, you can save and restore the content of the windows to and from arbitrary file. It is useful to store the parameters for a particular experiment to later recall all the situation.

You should not modify INI files by hand, they are only for program's internal use.

F) Crystal orientation with Spec

It is possible to create a UB matrix, and hence determine the crystal orientation within spec. This can be done with the big theta (*th*) or additional theta (*adth*) configurations, however these two motors cannot be used in conjunction. In order to switch between the two configurations, the *phi* motor (which is now a pseudo motor) has to be linked to another (real) motor. (The motor which rotates the phi angle has been renamed as *sphi*.)
When using the big theta, the phi pseudo motor should be connected to sphi motor.
When using the adth motor, the phi pseudo motor should be connected to adth motor.

One can link the motors by following the instructions below:

Open the configuration menu in the fourc window (config).
Go to the "phi" motor at no. 155, this is the last motor on the list (one place to the left). Scroll down to the Name field and press the p key.
Use the arrow keys to highlight the motor name and press the ' key.
Type the name of the motor that you wish to link to phi, either *adth* or *sphi*, then press the enter key.
To exit this screen press the p key.
Change the settings of phi motor to those of adth or sphi.
Write the changes to the configuration file (w key) and then exit (ctrl+c keys).
When the fource window is reconfigured, one should see a message stating that the phi motor has been linked to *adth* or *sphi*.

Once the correct motors have been linked, one needs to set the mode in which spec calculates the UB matrix. This is done by entering the *setmode* command in the fourc window.
For the big theta motor, use mode fixed phi mode (#3).
For the adth motor, use the fixed theta mode (#1).

Useful commands for calculating the UB matrix in spec:
setlat: sets lattice parameters of the crystal
or0 or *or1*: sets the primary or secondary reflection to the current angles of the spectrometer.
setor0 or *setor1*: sets the primary or secondary reflection without having to move the spectrometer.
or_swap: swaps the primary and secondary reflections.

pa: shows the lattice parameters and the positions of the two reflections.
wh: displays the current *H K L* coordinates and positions of the primary spectrometer motors.
ca H K L: calculates the motor angles for a reciprocal space position defined by the coordinates *H K L*.
ubr H K L: drives the spectrometer to the reciprocal space position given by the coordinates *H K L*.
hklscan H_{start} H_{end} K_{start} K_{end} L_{start} L_{end} *intervals time*: hklscan is a linear scan in reciprocal space over the three coordinates *H K L* which range from *Hstart*, *Kstart* and *Lstart* to *Hend*, *Kend* and *Lend*, respectively.

A) **addIXS**

A MatLab based user interface for converting and adding together inelastic x-ray scattering (IXS) spectra at ID28

Andrew Walters October 2010

Introduction to addIXS

addIXS is a MatLab based program which takes the raw inelastic x-ray scattering (IXS) data from a SPEC file and converts it into an xye file, where:

x is the energy transferred to the sample
y is the normalized intensity (normalized to monitor ‘ione’)
e is the square-root error on the intensity

Step 1: Starting addIXS

1. Open a Konsole on Leonov at ID28 (this can be done remotely)
2. Create the directory in which you will save your IXS spectra. Your directory should be a subdirectory of the /users/opid28/experiments/ directory. The unix command to do this is mkdir /users/opid28/experiments/my_experiment
3. Type “matlab” in the terminal window
4. Choose the directory you have just created as your working directory within MatLab (the path given at the top of the screen). This is the folder in which your IXS spectra will be saved
5. Type “addIXS” at the MatLab prompt
6. The addIXS user interface will appear:

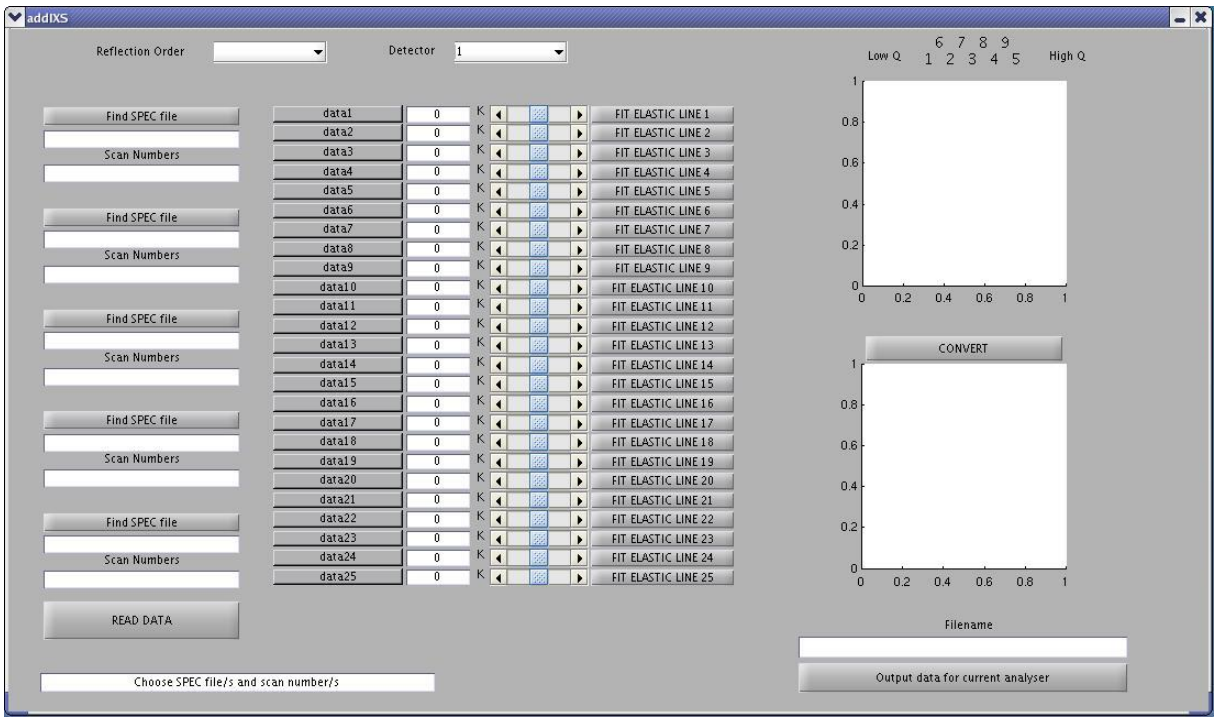


Figure 1: addIXS at startup

Step 2: Loading your SPEC data into addIXS

1. Choose the Si reflection order of the monochromator which was used for the IXS scans. If you are not sure which Si reflection was used, ask your local contact. The Si reflection is chosen via the drop-down menu labeled "Reflection Order". The Si reflection order is used in the calculation which converts the temperature of the monochromator into the energy of the monochromated beam
2. To select the SPEC file/s which contain/s the data to be converted or summed together, click on the top button which says "Find SPEC file".

The data can be found in /data/id28/archive/galilee/

3. Ask your local contact which subdirectory your data are in. When you have selected the SPEC file, the name of the SPEC file will appear below the "Find SPEC file" button. The other "Find SPEC file" buttons can be used to read from other SPEC files.

Please note that a maximum of
5 SPEC files can be read simultaneously

4. Below the name of the SPEC file there is a window labeled "Scan numbers". Enter the scan number/s which you wish to convert and/or sum. Note that normal MatLab array conventions can be used (e.g. 212:214 will load scans 212, 213 and 214).

Note that a maximum of 25 scans
can be read simultaneously

5. Press the "READ DATA" button. This can take a few seconds.
6. When the scans are loaded, the intensities measured by Detector 1 will appear at the top right, and a set of buttons

corresponding to each scan will appear in the centre of the user interface panel.

By default when the scans are first loaded, all of the scans are selected (the toggle buttons on the left are all down). To deselect any scan, simply press on the corresponding toggle button. The button will appear to be up, and no data measured in that detector will be plotted. Data measured in different detectors can be accessed using the drop-down menu labeled "Detector". addIXS should now look something like Figure 2.

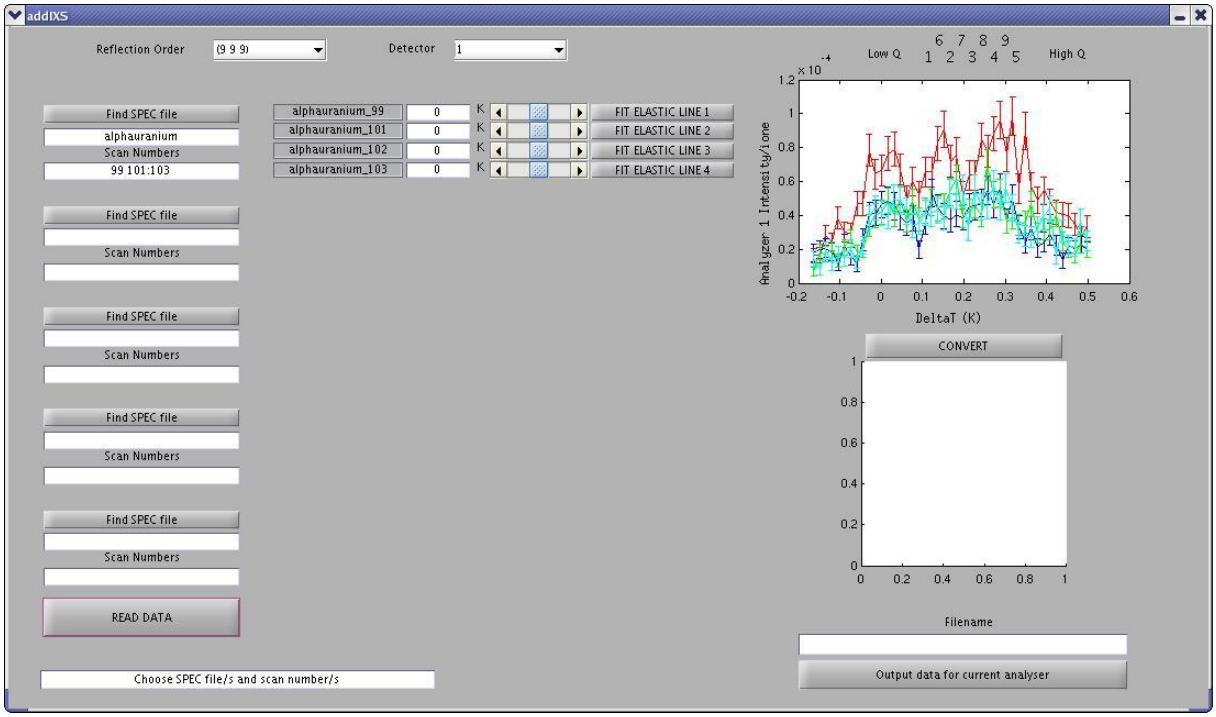


Figure 2: addIXS screen after clicking "LOAD DATA"

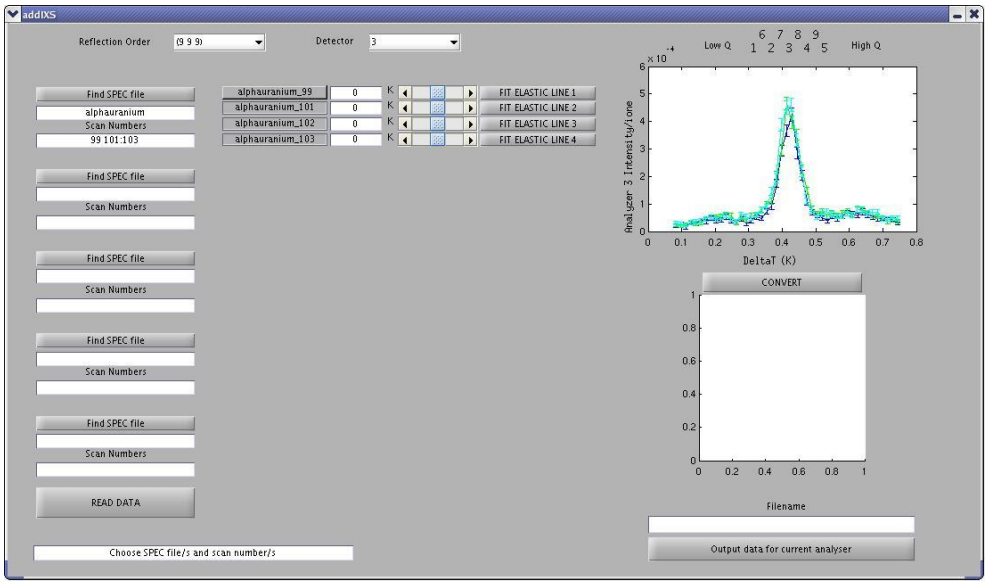
Step 3: Finding the temperature offsets for each analyzer crystal

The next step is to find the appropriate temperature offset for each analyser crystal. Although the temperatures of the analysers are kept as stable as possible, there can be some drift over a week-long experiment. In addition the measured temperatures of the analysers are very different to each other, due to the different contact resistances on the thermistors. Depending on the type of IXS data being measured, the temperature offset can be found in different ways.

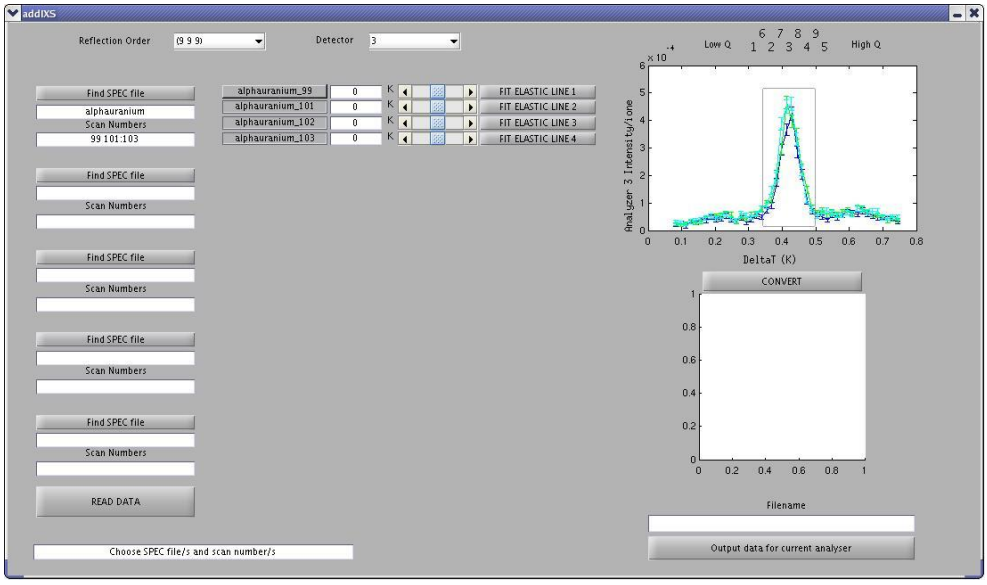
Perhaps the most common way is to include an elastic line in the IXS scan and then fit the elastic line in the immediate vicinity of the elastic line (so that any low energy phonons do not affect the fit). The elastic line can be fitted easily in addIXS, as can be seen in the following example.

Finding the temperature offsets for an analyzer: an example

1. We would like to sum the data measured in detector 3 in scans alphauranium_101, alphauranium_102 and alphauranium_103, as shown above. Note that alphauranium_99 has been deselected.

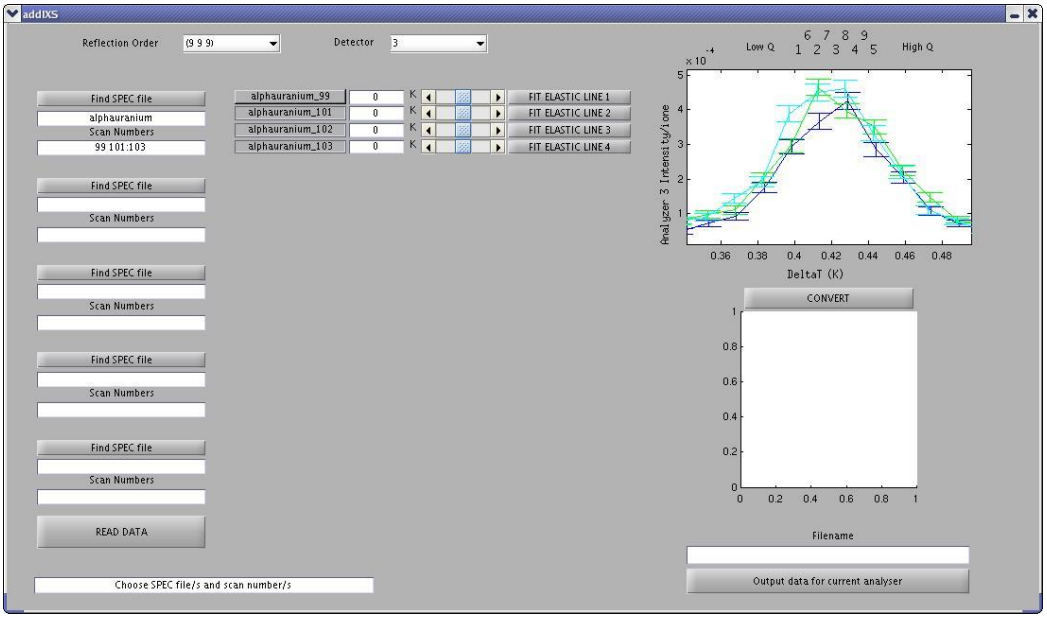


2. Select the range of temperatures over which the elastic line will be fitted for alphauranium_101. This is done by simply dragging a box around the region of interest. The scale can be put back to the default values by clicking the right mouse button and selecting “Reset to Original View”

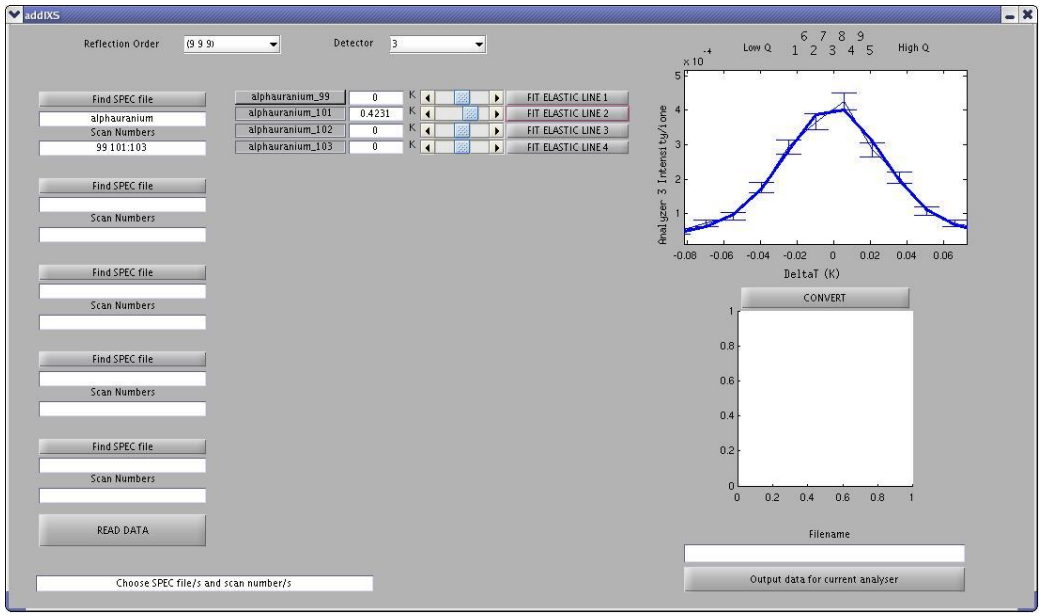


Note that it may be easier in terms of visibility if you select only the scan of interest, although this is not necessary

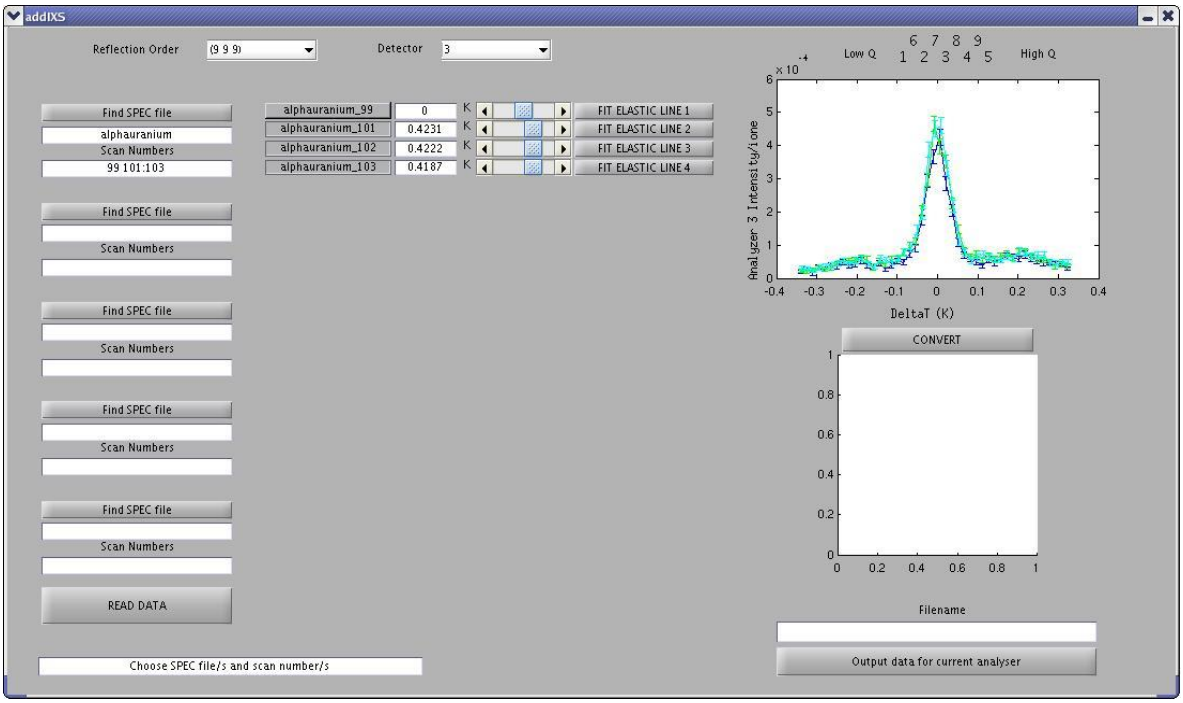
3. Click “FIT ELASTIC LINE X”, taking care to press the button which corresponds to the scan you are interested in.



4. The fitted value of the centre of the elastic line will appear in the box next to the scan of interest. The fit to the elastic line will also appear in the plot window. addIXS automatically subtracts this temperature offset from the plotted data. So if you have found the offset correctly, the elastic line should be at precisely 0.



5. This process is then repeated for the other two scans (alphauranium_102 and alphauranium_103).



In addition to the fitting option described above, the zero temperature offset can be typed in by hand (press the Enter key to update the value) or tweaked with the aid of a slider button.

Step 4: Converting the data onto an energy transfer scale

Once you are satisfied with the temperature offset/s, select all the files which you would like to sum, and press the "CONVERT" button to convert the selected datasets onto an energy transfer scale. When the convert button is pressed, all of the selected datasets for the chosen detector are converted onto an energy transfer scale and summed. The result is plotted in the panel at the bottom right of the addIXS user interface.

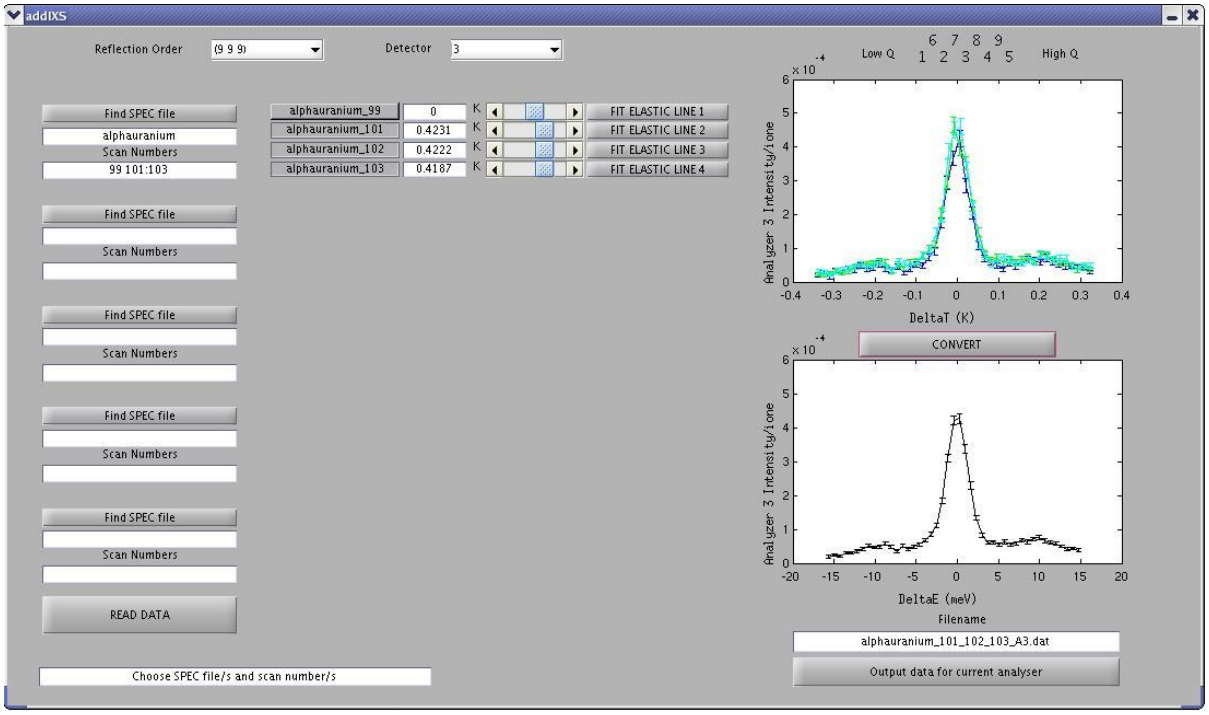


Figure 4: addIXS screen after clicking "CONVERT"

The name for the output datafile is automatically chosen in the format

<SPEC filename> _ <scan number> _A <detector number> .dat

This name for the output file can be changed if preferred by simply clicking on the filename and retyping. The output file will be saved into the MatLab working directory.

Steps 3 and 4 have to be repeated for each detector, as the temperature of the corresponding analyser will be different in each case.

B) MatLab User Interface: analyzer_resolutions

1. Ensure that the relevant analyzer resolution files in the analyzer_resolutions folder have the following syntax:

a**x**res**n_mode**<***user_defined_string***>**.**exp

where:
x = analyzer number (from 1 to 9)
n = main mono reflection order (7, 8, 9, 11, 12, 13)
mode = experimental conditions for the measurement, including:

- 16b_st (16 bunches, analyzer slits 20x60)
- 16b_60 (16 bunches, analyzer slits 60x60)
- um_st (200mA, analyzer slits 20x60)
- um_60 (200mA, analyzer slits 60x60)
- pm (postmonochromator)

<***user_defined_string***> = a user defined string of letters which can identify the specific analyzer resolution files selected, for example the program can read in files labelled as aXres9_pm_01042011.exp if this string is defined as '_01042011'. If preferred this can be left blank.

2. Open matlab in the appropriate directory of leonov.

3. Type **analyzer_resolutions** in MatLab. The user interface should appear.

4. Define the folder that contains the analyzer resolution files by clicking 'Select data directory'

5. Select the correct reflection order, the experimental mode the data were collected in and select the analyzers which were used in the experiment (automatically set as all).

6. Import the data.

7. Select the fit function you wish to use. The choices are:

- Pseudovoigt 1: Lorentzian and Gaussian widths are the same
- Pseudovoigt 2: Lorentzian and Gaussian widths are independent
- Voigt: Lorentzian and Gaussian widths are again independent
- Lorentzian
- Gaussian

8. Click on the appropriate button to fit the data.

9. Clicking "Export results" creates several ascii files:
The files with suffix ".fit" contain energy and normalized fitted intensity for the fit to the data for each of the 9 analysers.
The files with suffix ".nor" contain energy, normalized fitted intensity, and normalized errors for the fit to the data for each of the 9 analysers.
The file with suffix ".txt" summarizes the fit parameters.
A file with suffix ".param" is created if the resolution functions are fitted with "Pseudo-Voigt 1 " or "Pseudo-Voigt 2". This file is written in the syntax of ixs_fitter so it can be directly used as configuration file for the fit routine.

Note: The parameter *resnorm* in the .txt file is essentially an unnormalised χ^2 for the fit, and consequently can only be used in a relative fashion: i.e. if the value of resnorm is smaller for a Gaussian fit than for a Lorentzian fit, then the Gaussian is a better fit.

7. LIST OF MOTORS

A) FOURC list of motors

Motors for four-circle operation and additional motors

tth	2-theta arm of spectrometer
th	sample rotation around vertical axis; + cw (birds view)
chi	sample rotation around incident beam axis for th = 0; + cw looking towards undulator source
phi	sample rotation perpendicular to incident beam axis at th = 0; + cw looking towards ring wall
sax	sample translation along beam direction for th = 0; + away from spectrometer
say	sample translation perp. to beam direction in horizontal plane; + towards ring wall
saz	sample translation vertical; + upwards
tiltz	sample rotation around vertical axis for chi = phi = 0, redundant with th; + cw (birds view)
ty	translation of goniometer tower; + towards experimental hall
tthm	real motor of 2-theta arm of spectrometer, dimension in mm
adth	huber 410, sample rotation around vertical axis, if chi = phi = 0.
bmstop	beamstop to be installed behind sample chamber, + towards experimental hall.

Undulators

u32a	gap of 32 mm linear undulator (upstream)
u32b	gap of 32 mm linear undulator (middle)
u32c	gap of 32 mm linear undulator (downstream)
u176a	gap of 17.6 mm linear undulator (upstream)
u176b	gap of 17.6 mm linear undulator (middle)
u176c	gap of 17.6 mm linear undulator (downstream)

High heatload premonochromator

mono	main theta axis of channel-cut premonochromator
pi1	linear pusher to fine tune 2.crystal surface with respect to first one. scan not larger than +/- 2 mm
pmscr	diagnostics module behind premono with Kapton scattering foil and fluo screen; default position: approx. 66 mm; + towards inside vacuum; out of beam: 46

Postmonochromator

pmy	y-translation(hor. and perpend. to incident beam); + towards EXPH +10: out of beam; 0: crystal centred on beam. 20000 steps/mm = 0.05 μm; stroke: +0,-10 mm; 2.5 A
pmz	z-translation; + upwards 40000 steps/mm = 0.025 μm; stroke: +/- 5 mm; 5 A

pmth coarse theta of postmono; + ccw looking from window flange = larger Bragg angles
20000 steps/deg = 0.87 µrad; stroke: +/- 4 deg; 5 A

pmchi chi of postmono; + ccw looking from incident beam direction
20000 steps/deg = 0.87 µrad; stroke: +/- 1 deg; 2.5 A

pi2 linear pusher to fine tune postmono angle theta.+ pushing
400 steps/µrad = 2.5 nrad; stroke: +/- 50 µrad

pmscr2 diagnostics module behind postmono with Kapton scattering foil; default position: 41 mm; + towards inside vacuum; out of beam: 21
400 steps/mm = 2.5 µm; stroke: +/- 20 mm; 2 A

pmono2 weak link between first and second crystal of postmono

oh3mony Motorised translation to insert diode into pre-/postmono beam.

Main backscattering mono

ymono y-translation of mono chamber; + towards ring tunnel
1600 steps/mm = 0.625 µm; stroke: +/- 5 mm; 1.2 A

zmono mono chamber height; + upwards

mth theta angle; + deflects the beam upwards

mchi chi angle; + cw (birds view)

mxl left blade of main mono hor. Slit; ½ cutting at 0; default position: -3 + towards inside vacuum

mxr right blade of main mono hor. Slit; ½ cutting at 0; default position: -3 + towards inside vacuum

mxoff offset of main mono slits

mxgap gap of main mono slits.

mscr Fluo screen for detection of backscattered beam; default position: 1; operation position: approx. 40.5; + towards inside vacuum

monot temperature setpoint of main mono

deltae energy transfer of main mono, needs to be set-up properly.

Mirror unit

miroty theta angle of gold mirror; needed for onmirrorchk; + defectsd beam upwards

mihexy horizontal translation of gold mirror

mihexz vertical translation of gold mirror; + upwards

Focusing lens unit

fly y-translation of lens; + towards ring tunnel; +12 (out), +6 (n=8); 0 (n=9), -6 (n=11)
1000 steps/mm = 1 µm; stroke: +/- 15 mm; 1.2 A

flz z-translation of lens; + upwards
20000 steps/mm = 0.05 µm; stroke: -2/+13 mm; 1.2 A

flth theta of lens (rotation axis hor. + perpend. to x-ray beam; + cw looking at the window flange
20000 steps/deg = 0.87 µrad; stroke: +/- 1 deg

KB focusing unit

hfmy horizontally focusing mulitlayer, y-translation, + towards EXPH

hfmth horizontally focusing mulitlayer, - positive reflection angles
hfmpi HFM piezo for fine-tuning of theta
vfmth vertically focusing mirror, - positive reflection angle
vfmpi VFM piezo for fine-tuning of theta
minisl 100 μm vertical slit(5: out of the beam, 20: approximate working pos.)

Entrance slit unit

Izero slit unit

huxl left blade of motorised Huber slits
huxr right blade of motorised Huber slits
huzu top blade of motorised Huber slits
huzd bottom blade of motorised Huber slits
huxo horizontal offset of Huber slits
huxg horizontal gap of Huber slits
huzo vertical offset of Huber slits
huzg vertical gap of Huber slits

Ione slit unit

i1sho horizontal offset of JJ slits
i1shg horizontal gap of JJ slits
i1svo vertical offset of JJ slits
i1svg vertical gap of JJ slits

wheel Attenuation wheel, increasing attenuation with increasing motor position: n*22.5 degrees (n=0-15). No attenuation: wheel 0.

Quadrupole Ion Chamber Unit

I1h vert.. polarised ion chamber, y-movement, + towards EXPH
I1v vert.. polarised ion chamber, z-movement, + upwards
I2h hor. polarised ion chamber, y-movement, + towards EXPH
I2v hor. polarised ion chamber, z-movement, + upwards

7 m spectrometer

ssy horizontal translation spectrometer entrance pinhole. It has to be aligned carefully before each experiment; - towards experimental hall.
ssz vertical translation of entrance pinhole; + upwards.

dy horizontal translation of detector chamber; + towards ring wall.
dz vertical translation of detector chamber; + upwards
dsz vertical translation of detector pinhole ana#1-#5
dsr horizontal translation of pinhole ana#1-#5; + towards experimental hall
dsz1 vertical translation of pinhole ana#6-#7
dsr1 horizontal translation of pinhole ana#6-#7; + towards experimental hall

zdetsq1 vertical translation of close-by S(Q) detector; + downwards.
znewdet vertical translation for alignment diode: in -55, out -5

a(i)hgap	horizontal gap of analyser slits for ana#i i=1-5
a(i)vgap	vertical gap of analyser slits for ana#i
a(i)hrot	horizontal rotation of slit drum #i
a(i)vrot	vertical rotation of slit drum #i

achi1	chi angle of ana #1; + deflecting beam towards tunnel
ath1	theta angle of ana #1; + head direction upwards
ath2(3,4)	pseudo motor theta angle ana #2 (3,4); + deflecting beam upwards
achi2(3,4)	pseudo motor chi angle ana #2 (3,4); + deflecting beam towards tunnel
athp2(3,4)	real motor ath for ana #2 (3,4), moves at 45° to vertical plane: + head direction upwards
achip2(3,4)	real motor achi for ana #2 (3,4), moves at -45° degrees to vertical plane. + head direction upwards.
ath5	theta angle of ana #5; + deflecting beam upwards
achi5	chi angle of ana #5; + deflecting beam deflecting beam towards tunnel
ath(6-9)	theta angle of ana #6 - #9; + deflecting beam upwards
achi(6-9)	chi angle of ana #6 - #9; + deflecting beam deflecting beam towards tunnel

Multi-purpose motors, (mostly used for deflecting mirror set-up for surface phonon measurements)

adth	additional theta for single crystal work
deflz1	+ upwards *(0.95A, 20.000 /mm)*
deflz2	+ upwards *(1.8A, 400 /mm)*
deflth	+ ccw, looking towards ring tunnel *(1.2A, 4.000 /mm)*
dchi	+ ccw, looking towards source *(1.8A, 400 /mm)*

Others

cryo	temperature setpoint of ST15 cryostat
bmstop	motorised beamstop to be mounted on transfer line.
mary	y translation of area detector support (MAR or Pilatus)
marz	z-translation of area detector support (MAR or Pilatus)

teuro1	temperature setpoint Eurotherm 1
teuro2	temperature setpoint Eurotherm 2

ICEPAP diagnostics

- open a window on leonov
- login is bliss adminstrator
- type: su -blissadm; password: spec92
- type: icepapcms

B) SLITS LIST OF MOTORS

Primary slits

pl	left blade; specify directions
pr	right blade
pu	upper blade
pd	down blade
pho	horizontal offset
phg	horizontal gap
pvo	vertical offset
pvg	vertical gap

Secondary slits

sl	left blade
sr	right blade
su	upper blade
sd	down blade
sho	horizontal offset
shg	horizontal gap
svo	vertical offset
svg	vertical gap

Collimating lens system

cly	horizontal translation
clz	vertical translation

C) HX1 (Premono hexapode) LIST OF MOTORS

mohexx	translation along x-direction; specify directions
mohexy	translation along y-direction
mohexz	translation along z-direction; + upwards
morotx	rotation around x-axis
moroty	rotation around y-axis
morotz	rotation around z-axis

D) HX2 (mirror hexapode) LIST OF MOTORS

mihexx	translation along x-direction; specify directions
mihexy	translation along y-direction
mihexz	translation along z-direction; + upwards
mirotx	rotation around x-axis
miroty	rotation around y-axis
mirotz	rotation around z-axis

bender	bender motor for variable meridional radius

E) KB LIST OF MOTORS

hfmb1, hfmb2, hfmb:	multilayer bender motors, hfmb moves both motors simultaneously. + larger bending radius
hfmth:	multilayer theta - positive angles
hfmtz:	real motor, used to make y movement
hfmz1, hfmz2, hfmz:	z-motion, hfmz moves both motors simultaneously + upwards
hfmtx:	tilt around x-axis (chi) by opposite movement of hfmz1 and hfmz2.
hfmy:	horizontal movement of multilayer + towards EXPH
hfmpi:	HFM piezo for fine tuning of multilayer angle
vfmb1, vfmb2, vfmb:	mirror bender motors, vfmb moves both motors simultaneously. + larger bending radius
vfmth:	mirror theta - positive angles
hfmtz:	real motor, used to make y movement
vfmz1, vfmz2, vfmz:	z-motion, vfmz moves both motors simultaneously + upwards
vfmpi:	VFM piezo for fine tuning of mirror angle
vfmmoni:	y-translation of scattering foil for monitor detector between VFM and HFM.
moniy:	y-translation of slit-ion chamber unit - towards EXPH
mlleft, mlright:	horizontal slits in front of the VFM and HFM
mloff, mlgap:	gap and offset of these slits

8. ID28 MEMBERS AND PHONELIST

(27.08.2015)

ID28 ---------- 20.61 / 21.04

ID28 staff:

Alexei Bossak ----------2652 --------- 04.76.42.38.99 ------ 06.84.38.98.31
Denis Gambetti ----------28.75 / 21.04 04.76.13.03.13
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Tom Forrest ----------2319
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Associated members :

Herve Gonzalez ------ 28.08 / 25.44
Marie-Claire Lagier --22.59
Keith Martel ---------- 23.59

IXS laboratory (near ID16): 68.92

9. APPENDIX

S(Q) detector (detsq) at tth ≈-0.3°

zdetsq1 = 36.65
zdetsq1 out = 13.3

PLEXIGLAS

Si(8 8 8)

Q [nm^{-1}] = 4π/λ * sin(2θ/2) = 160.3 nm^{-1} *sin(2θ/2)

2θ (10 nm^{-1}) = 7.153° λ = 0.7839 Å
E = 15.816 keV

Analyzer No°	tth [°]
1	8.673°
	7.933°
2	7.153°
	6.406°
3	5.626°
4	4.104°
5	2.576°

Si(9 9 9)

Q=180.35*sin(2θ/2)

2θ (10 nm^{-1}) = 6.357° λ = 0.6968 Å
E = 17.794 keV

Analyzer No°	tth [°]
1	7.877°
6	7.137°
2	6.357°
7	5.610°
3	4.830°
4	3.308°
5	1.780°

Si(11 11 11)

Q=220.414*sin(2θ/2)

$2\theta(10\ nm^{-1}) = 5.200°$ $\lambda = 0.5701$ Å

E = 21.7477 keV

Analyzer No°	tth [°]
1	6.720°
6	5.980°
2	5.200°
7	4.453°
3	3.673°
4	2.151°
5	0.623°

Si(12 12 12)

Q=240.459*sin(2θ/2)

$2\theta(10\ nm^{-1}) = 4.767°$ $\lambda = 0.5226$ Å

E = 23.725 keV

Analyzer No°	tth [°]
1	6.287°
6	5.547°
2	4.767°
7	4.020°
3	3.240°
4	1.718°
5	0.190°