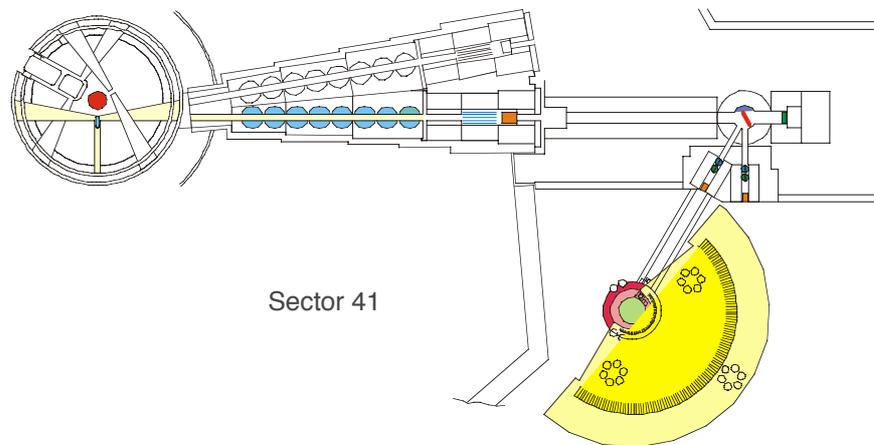


USER GUIDE HRPT:

HIGH RESOLUTION POWDER

DIFFRACTOMETER FOR THERMAL NEUTRONS

AT THE SINQ TARGET STATION



Caution: Sensitive detector - Avoid shocks/vibrations !
(Do not place auxiliary equipment onto the detector shielding !)

version September 2002

A) GENERAL PROPERTIES AND PARAMETERS	p.
A1. Instrument design	3
A2. Polychromatic neutron beam	5
A3. Monochromators	5
A4. Secondary collimation	11
A5. Sample, sample table, beam stop	11
A6. Detector bank	11
A7. Multidetector and effective collimation	11
A8. Detector electronics	12
A9. Resolution Functions	13
A10. Standard samples	15
A11. Examples of powder measurements and profile refinements	15
A12. References	17
B) HRPT COMPUTER CONTROL	18
How to...	
B1. log into the HRPT computer	18
B2. start the instrument control program SICS	18
B3. start the powderstatus display	19
B4. start a measurement on HRPT: the SICS commands	22
B5. edit the command file	24
B6. perform scans	24
B7. add, subtract and transform data files	25
B8. view data files	26
B9. use PC and Mac terminals of HRPT	26
Appendix	
B10. Data evaluation, profile refinement, plots	27
B11. Unix and further SICS commands	30
B12. Troubleshooting	30
B13. Summary of HRPT motors	31
B14. Check list HRPT	32
B15. Contact persons	33
C) SAFETY PROCEDURE AT SINQ	34

A) GENERAL PROPERTIES AND PARAMETERS

A1. Instrument design

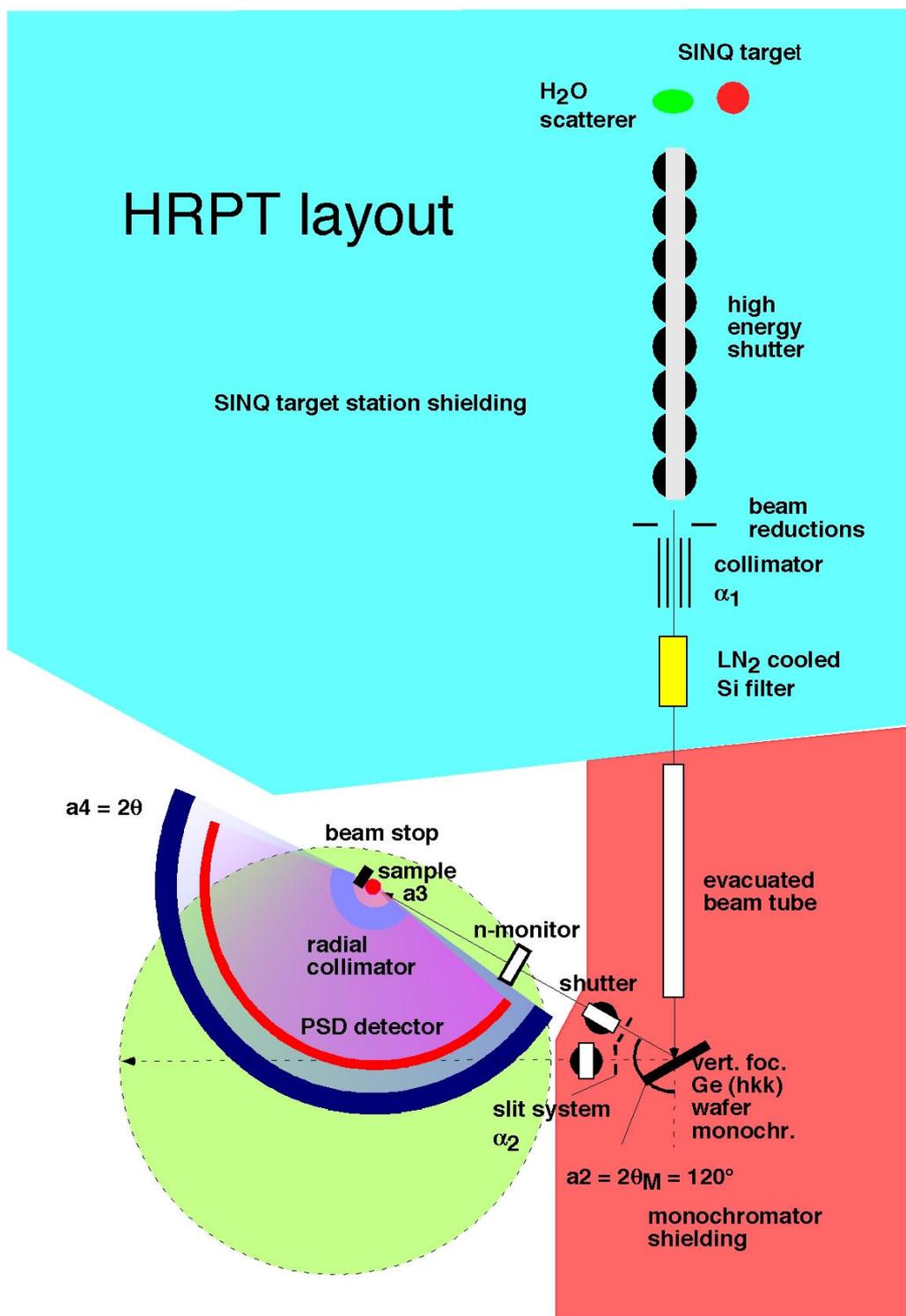


Fig. 1a: HRPT concept

Complementary to DMC, the multidetector diffractometer HRPT is designed as flexible instrument for efficient neutron powder diffraction studies - also for small sample sizes. High resolution is achieved by thermal neutrons, large scattering angles of the monochromator and of the sample (up to 165°). By means of primary collimators, a secondary slit system and by appropriate choice of the

sample diameter, resolution and intensity may be optimised. Due to the use of a large position sensitive (PSD) ^3He detector, simultaneous measurements are possible within a scattering angle range of 159.9° with angular step 0.1 degree (e.g. for real-time experiments). Depending on the desired point density and resolution up to $\delta d/d < (0.002, 0.001)$ [in the high intensity (HI) and high resolution (HR) modes of operation, respectively for monochromator take-off-angle $2\theta_M = 120^\circ$; $d =$ interplanar spacing], the detector may be positioned on air cushions also at intermediate positions. Standard measurements are performed by means of an evacuated aluminium pot equipped with a cooling machine, which can be oscillated to reduce preferred orientation effects of the sample. An oscillating radial collimator suppresses Bragg peaks from the sample environment such as for cryostats or furnaces. The instrument is controlled via the SICS software (clients written in JAVA, TCP/IP network, <http://Ins00.psi.ch>) of PSI by an UNIX work station, permitting rapid online refinements. The data transfer from the fast Cerca electronics (developed by E. Berruyer) to a PSI user interface and histogram memory (developed by N. Schlumpf et al.) is made via a central PC data exchange system and optical cables. More instrumental details may be found in the references [1,2].

Complementary to synchrotron X-ray powder diffraction studies, the applications of HRPT are high-resolution refinements of chemical and magnetic structures as well as phase analysis of novel materials. Because of the generally weak absorption, neutrons yield information on bulk properties (average over large sample volumes) and permit in a nondestructive way the detection of lattice distortions, defects and internal microstrains. Also real-time (insitu) investigations of chemical or structural changes and of magnetic phase transitions in crystalline, quasicrystalline, amorphous and liquid samples including technically interesting new materials are possible. Examples are precise localization of light elements (isotopes) such as hydrogen (deuterium) in metal deuterides (metal hydrogen storage systems) or zeolites, oxygen in high- T_c and CMR systems and other ceramics or ^7Li in ionic conductors etc. Another advantage may be the distinction between neighbouring elements in the periodic table such as Mn (negative neutron scattering amplitude b) and Fe ($b > 0$). Moreover, nuclear neutron scattering yields in case of a sufficiently large range of momentum transfer accurate information on average thermal motion (harmonic and anharmonic temperature factors). These applications are important in crystallography, condensed matter physics, chemistry and material science. In particular, profile analysis as a function of external parameters such as temperature (e.g. 10 K - 300 K with CTI cooling machines) is standard in neutron diffraction.

**Accessible temperature range: 120 mK – 1300 K,
vertical magnetic field up to 4 T,
hydrostatic pressure up to 8 kbar (Ti-Zr zero matrix pressure cell).**

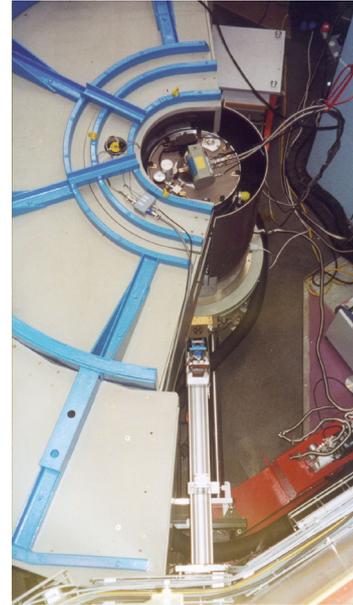
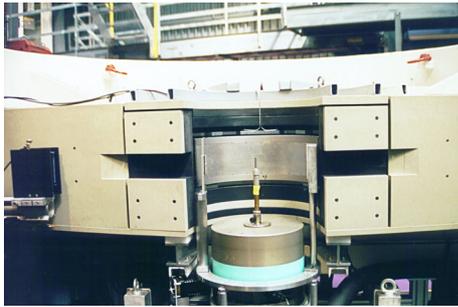


Fig. 1b: Instrument views at SINQ target station. Picture on the right: Evacuated Al pot with oscillating closed-cycle He refrigerator. Oscillation may reduce preferred orientation effects.

A2. Polychromatic neutron beam

Thermal neutron beam R41 from H₂O scatterer close to the target
 Primary beam width: 58, 44 or 29 mm (+ beam closed), beam height 150 mm within the plug

PRIMARY COLLIMATION:

Al alloy-Gd-O Soller collimators with

1. $\alpha_1 = 6', 12'$ (high resolution),
2. $\alpha_1 = -$ ($\approx 40'$, high intensity)

Si FILTER:

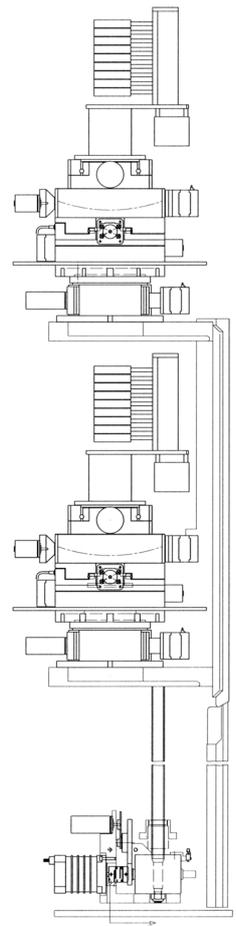
Length 20 cm, liquid N₂ cooled, check temperature

EVACUATED BEAM TUBE:

Between Si filter and monochromator, vacuum to be checked



Fig. 2: HRPT monochromator lift.



A3. Monochromators

MONOCHROMATOR SCATTERING ANGLES $2\theta_M$: 90° or 120°

MONOCHROMATOR 1 (lower lift position):

Ge (hkk), wafer type, vertically focusing (for ω not very different from 0, -> intensity gain up to a factor of 4, curvature variable), see Figs. 3 and 4

Planes (511) parallel to the monochromator surface plane. Other (hkk) planes may be used by slight rotation (angle ω) of the monochromator around the vertical axis coinciding with a $[01\bar{1}]$ axis of the monochromator, see table 1. Due to the nonzero structure factor of Ge reflections (2h2k2l) for even indices (hkl) higher order contamination $\lambda/2$ is more likely than in case of odd (hkl), but the intensity may be higher !

Table 1: Possible choice of neutron wavelength λ [Å].

Ge		$2\theta_M = 90^\circ$			$2\theta_M = 120^\circ$			$I_{rel.}(V, HI)$	$10^{-6}I(n\text{-mon.})/(h, 1250\mu A)$
(hkk)	ω_M^{511} [°]	$\lambda_{cal.}$	$\lambda_{obs.}$	det. eff.	$\lambda_{cal.}$	$\lambda_{obs.}$			
311	-9.45	2.413	2.410*	0.79	2.955	2.953* ^s	1.5	3.3	
400	15.79	2.000		0.75	2.450	2.449*#	0.8	2.8	
133	-60.94	1.836	1.832	0.73	2.248	2.246* ^s	0.5	1.0	
511	0.00	1.540	1.539(1)	0.68	1.886	1.886	4.6	6.3	
533	-24.52	1.220	1.219	0.61	1.495	1.494	4.1	5.1	
711	4.37	1.121		0.58	1.372	1.372	3.3	3.5	
733	-15.43	0.978		0.54	1.197	1.197	2.8	2.7	
822	3.68	0.943		0.53	1.155	1.154	3.3	3.2	

* PG(C) filter; # 1.6 Å contamination due to double Bragg scattering in the monochromator; mgpl is set to 3.25 and mcvl to 0.5 to avoid this $(2/3)\lambda$ contribution; ^s $\lambda/3$ contamination

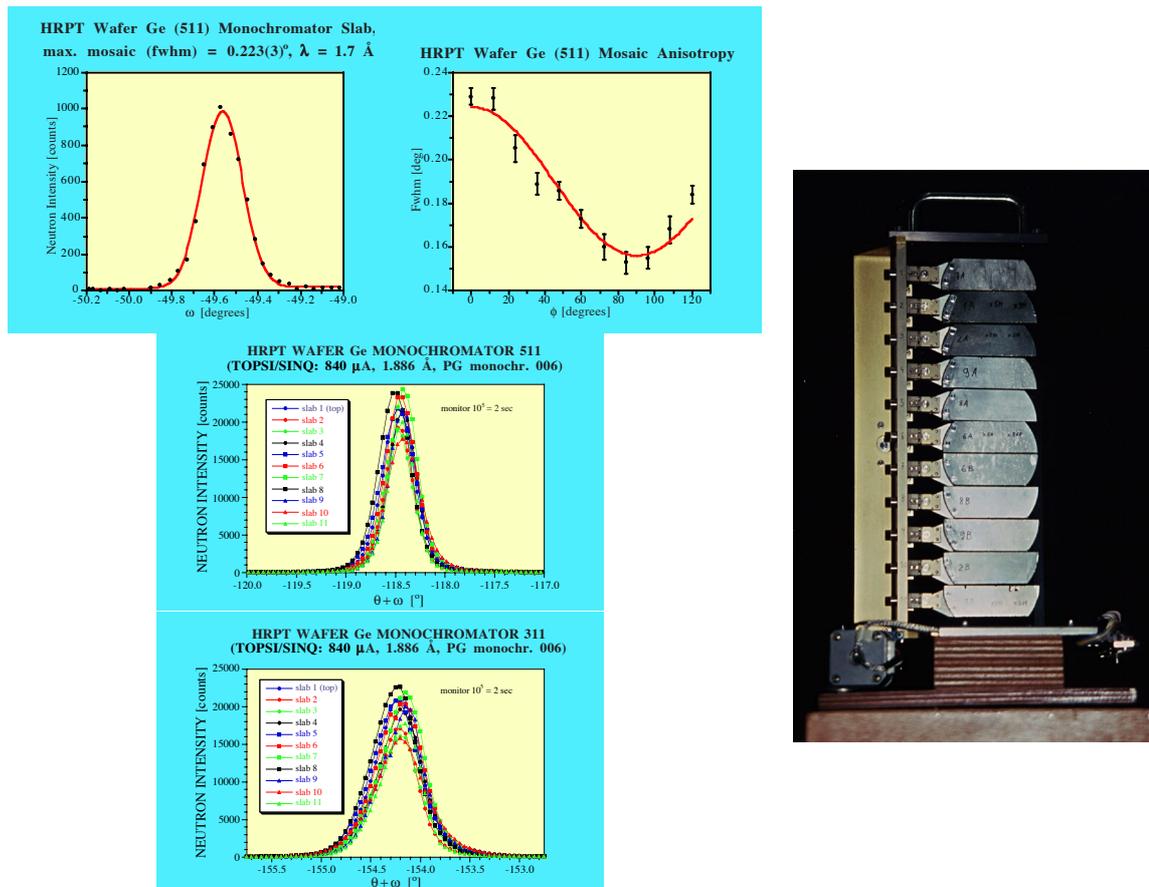


Fig. 3: Vertically focusing wafer type Ge monochromator of HRPT, 28.5 cm high, slabs of ≈ 7 cm width, 2.5 cm height and 9.6 mm thickness, total mosaic halfwidth $\beta \approx 15'$.

Tab. 2a: Primary beam and monochromator parameters (values in degrees).

Variable	Control no.	SICS name, short	SICS name, long
Collimator drum A (inner) Beam size: Closed, Width58xheight150 mm ² , Width44xheight150 mm ² , Width29xheight150 mm ²	b01	a17	cex1: 0, 44.6, 152.4, 310.1
Collimator drum B (outer) Beam size: Closed, None, width55xheight150 mm ² , 6', width55xheight150 mm ² , 12', width55xheight150 mm ²	b02	a18	cex2: 0, 85.9, 206.9, 261.9
Rotation ω Upper translation \perp cryst. [mm] Lower translation \parallel cryst. [mm] Upper tilt \perp crystal [°] Lower tilt \parallel crystal [°] Focusing curvature [°] Upper lift position [mm]	c09 c11 c10 c05 c12 c06 d01	a1 a12 a13 a14 a15 a16 a37	momu \approx -1.14 (333) Mtvu \approx 0 mtpu \approx 0 mgvu \approx 0.2 mgpu \approx -2.0 mcvu \approx 0 mexz = 0
Rotation ω Upper translation \perp cryst. [mm] Lower translation \parallel cryst. [mm] Upper tilt \perp crystal [°] Lower tilt \parallel crystal [°] Focusing curvature [°]	c07 c01 c08 c03 c02 c04	b1 a22 a23 a24 a25 a26	moml mtvl mtpl mgvl mgpl mcvl: -0.010 = flat, min. -2.568, max. 9.516 mexz = 668
Lower lift position [mm]	d01	a37	mexz = 668
Monochr. scattering angle $2\theta_M$	-	a2 *)	mtt = 90 or 120

*) The value of a2 may defined by typing **a2 90** or **a2 120**.

Tab. 2b: Further monochromator parameters for a2 = 90°.

hkl	moml _{av.}	moml _{max.}	mgvl	mgpl	mcvl	rel. intensity (without focusing) to (511)	foc. gain (2 cm high beam at mon.)
311	-9.82	-9.95	-0.1	0	2.5	0.88	3.4
400	15.36	15.2	-0.12	-0.16	3.2	1.30	3.1
133	-61.36	-61.45	-0.1	-0.14	3.7	0.75	1.9
511	-0.45	-0.46	-0.1	0	2.5	1.00	4.0
533	-24.97	-25.05	-0.11	-0.16	2.6	0.46	3.2
711	3.93	3.84	-0.12	0	3.0	0.33	3.7
733	-15.89	-15.96	-0.1	0	3.3	0.17	3.4

Tab. 2c: Monochromator parameters for $\alpha_2 = 120^\circ$.

hkl	moml_{max}	mgvl	mgpl	mcvl
311	-25.04	-0.1	0	2.1
400	0.20	-0.12	3.25*	0.5*
133	-76.55	-0.1	-0.14	1.55
511	-15.6	-0.1	0	2.0
533	-40.1	-0.1	0	2.0
711	-11.19	-0.12	0	2.0
733	-31.0	-0.1	0	2.1
822	-19.25	-0.15	-0.1	2.06

* avoids the $(2/3)\lambda$ contamination (mgpl = -0.16 and mcvl = 2.3 with $(2/3)\lambda$)

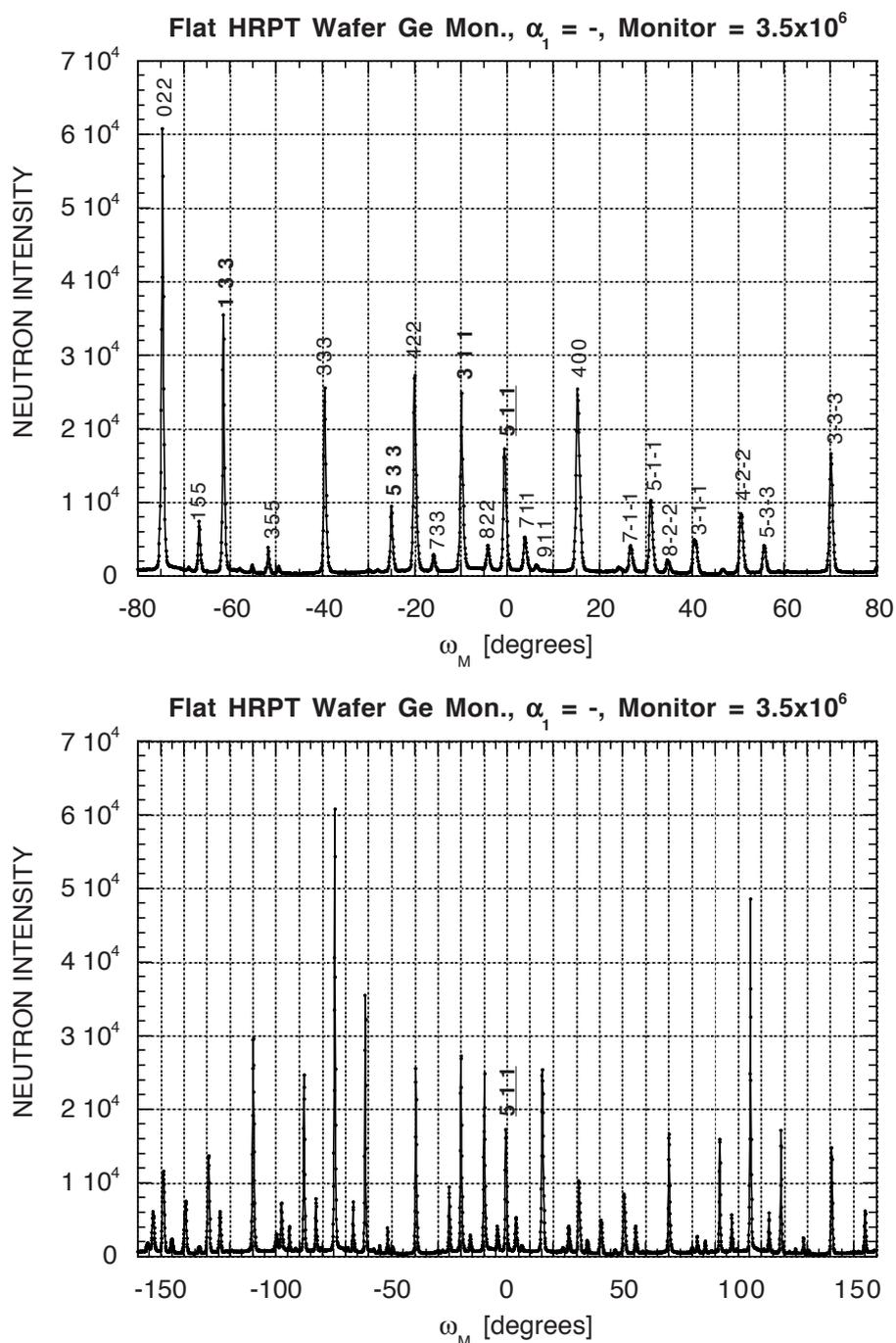


Fig. 4a: Neutron intensity from flat wafer Ge monochromator as a function of monochromator rotation angle ω for $2\theta_M = 90^\circ$. By using a primary collimation $\alpha_1 = 12'$ or $6'$, the intensity (511) is reduced by factors of approximately 2.6 and 5, respectively.

MONOCHROMATOR 2 (upper lift position):

Presently a perfect Ge (hkk) plate of diameter 3" and thickness 9 mm, (111) parallel to the plate surface, [01 1] \approx vertical, may be used e. g. for crystal tests, mosaic determination.

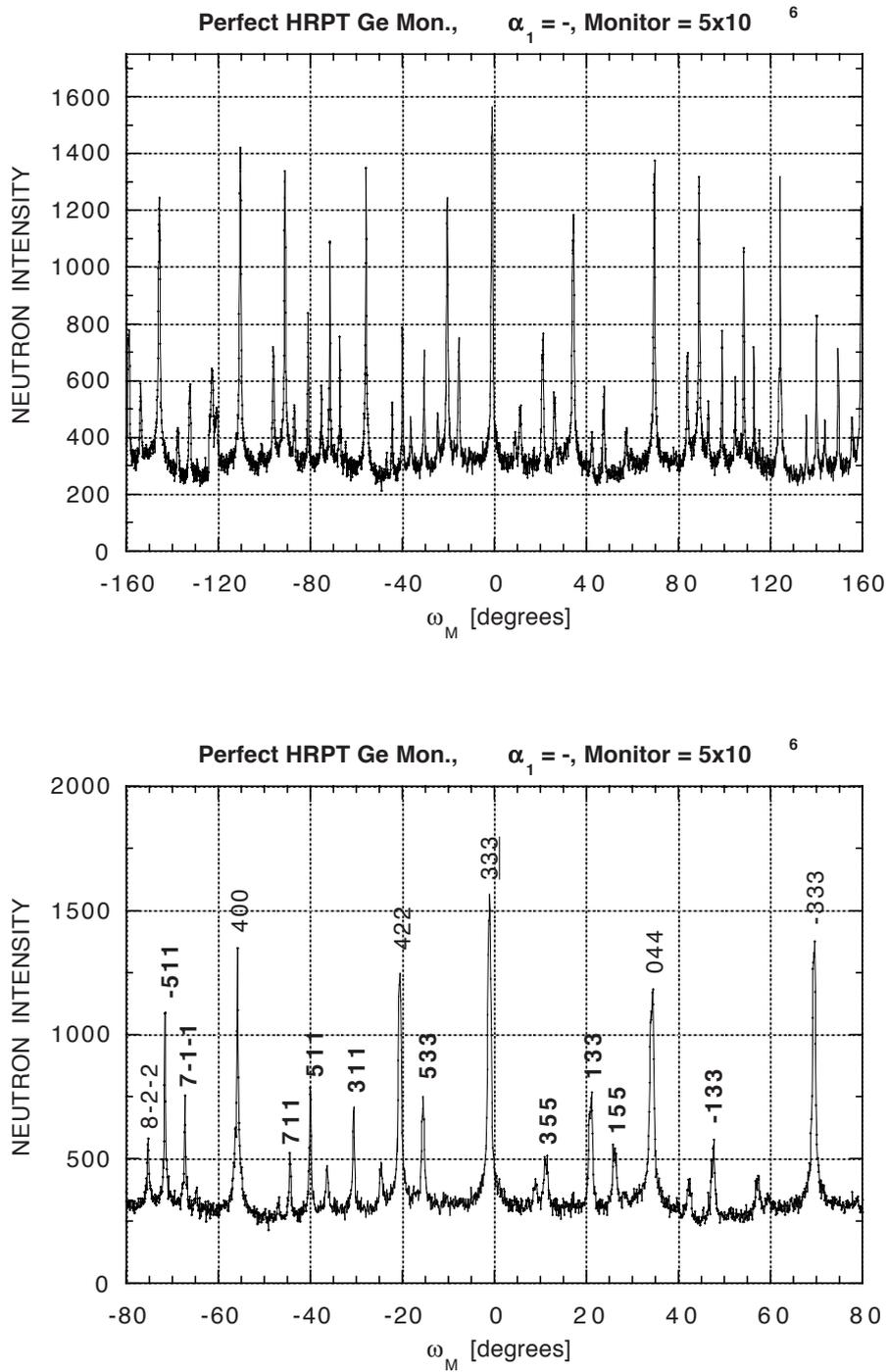
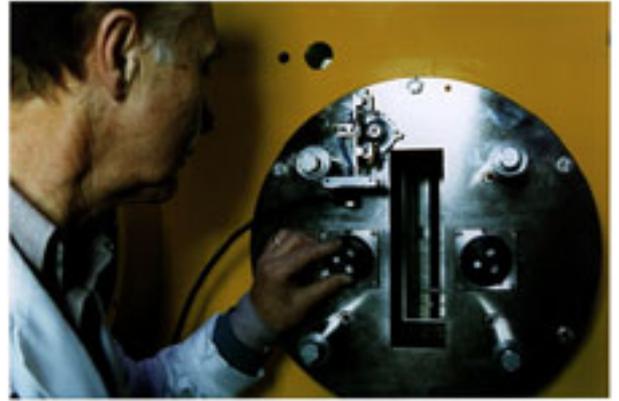


Fig. 5: Neutron intensity from the perfect Ge monochromator as a function of monochromator rotation angle ω_M for $2\theta_M = 90^\circ$.

A4. Secondary collimation

α_2 : \leq approximately $40'$ due to vertical slit system, **beam width w horizontally variable: (0 ÷ 60) mm**, at a distance of 72 cm from the monochromator and 54 cm from the plug end towards the sample, permits to tune the resolution according to the needs



A5. Sample, sample table, beam stop

Cylindrical samples with height up to ≈ 5 cm, distance sample table – neutron beam center = 270 mm (also 400 mm possible), radius of sample table = 20 cm

Fig. 6: Horizontal slit system for the monochromatic neutron beam.

Sample table angle: $-164^\circ < \alpha_3 = \text{som} = \text{om}(\text{ega}) < 164^\circ$

Caution: Be careful to not let the direct beam onto the detector – make sure that the beam stop ($1^\circ \approx 4.7$ mm) is properly placed !

A6. Detector bank

Range of scattering angles $\alpha_4 = \text{stt} = \text{th} = 2\theta \approx 3^\circ \div 5^\circ (+ 159.9^\circ)$ with a precision $\approx \pm 0.001^\circ$

Radial (1°) mylar-Gd-O collimator to eliminate Bragg peaks from sample environment such as from cryostat or furnace tails and to reduce background, see Fig. 7

□

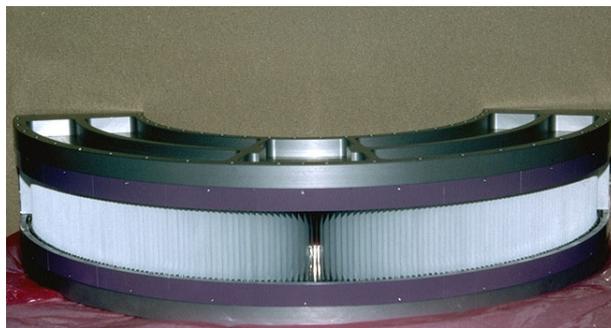


Fig. 7: Radial collimator of HRPT with individually stretched mylar Gd-O foils, constructed and manufactured at Risø by J. Linderholm, JJ X-Ray.

A7. Multidetector and effective collimation

Position sensitive detector (PSD), produced by Cerca, Romans, see Fig. 8, **25x64 = 1600 wires** (W, Au plated, **150 mm high**, diameter $\text{Ø} \approx 22 \mu$),

angular spacing 0.1° , radius $R = 1500 \text{ mm}^*$ -> distance 2.62 mm^{} between the wires**

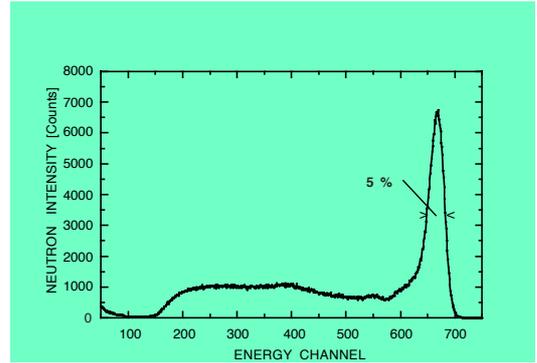
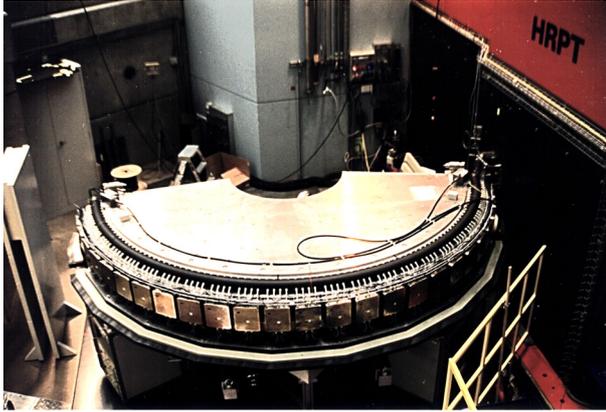
Detector gas: ^3He (3.6 bar) + CF_4 (1.1 bar), **effective detection length 3.5 cm**

[distance front cathode-anode (2 mm Al) = 15 mm, distance anode-back cathode = 20 mm, 10 mm thick Al window, 1.5 mm thick teflon in the 2 mm gap between Al window and front cathode]

(cf. Tab. 1 for the high det. efficiency.)

For high resolution work with the PSD it is important to optimise the collimations α_1 , α_2 and α_3 . A recommended value is $\alpha_2 = 2\alpha_1$. The corresponding slit width w may be calculated from the distance slit – sample ≈ 258 cm as: $w [\text{mm}] \approx 5160 \text{tg}\alpha_2 - D$. Max. $w = 6$ cm (30/30 [mm]) for high intensity mode. α_3 depends on the sample diameter D , on * and **: $\text{tg}\alpha_3 = (D[\text{mm}] + 2.62)/3000$, where D is the diameter of a cylindrical sample (Table 3). **Optimum D : 5 or 8 mm.**

□



□

Fig. 8: HRPT detector with excellent detector characteristics (figure on the right for $\lambda = 1.54 \text{ \AA}$ and neutron beam width 2 mm) at SINQ target station.

Table 3: Dependence of the effective collimation α_3 on the sample diameter.

D [mm]	α_3 [°]	α_3 [']
2.62	0.10	6.0
5	0.15	8.7
6	0.16	9.9
8	0.20	12.0
9	0.22	13.3
10	0.24	14.5

A8. Detector electronics

The Cerca detector electronics consists of 25 modules with 64 wires per module. A central data exchange system SCE (industrial PC with special card, using Windows 3.11) communicates (ihm program) with an external PC as ‘Cerca quick look facility’ (via ethernet link, working under Windows 95, e. g. for detector tests) and in parallel with the mainly used PSI interface and histogram memory (developped by N. Schlumpf et al.) for normal data collection – see **special manual**. Picture length 1 s (or longer) down to 20 ms may be used. The module memory capacity is limited to $2^{10} = 1024$ counts per picture and channel.

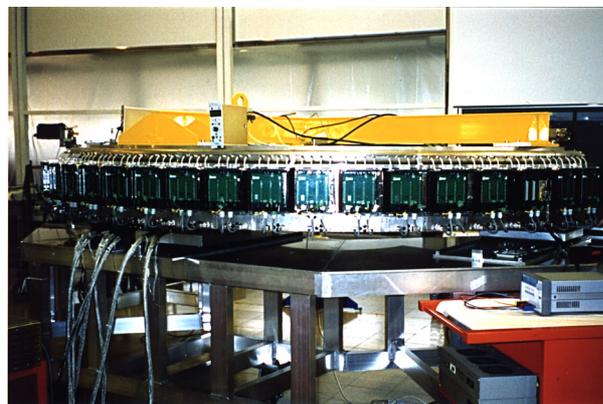


Fig. 9: Detector electronics, developed by E. Berruyer, Cerca, F-26104 Romans.

□□A9. Resolution functions

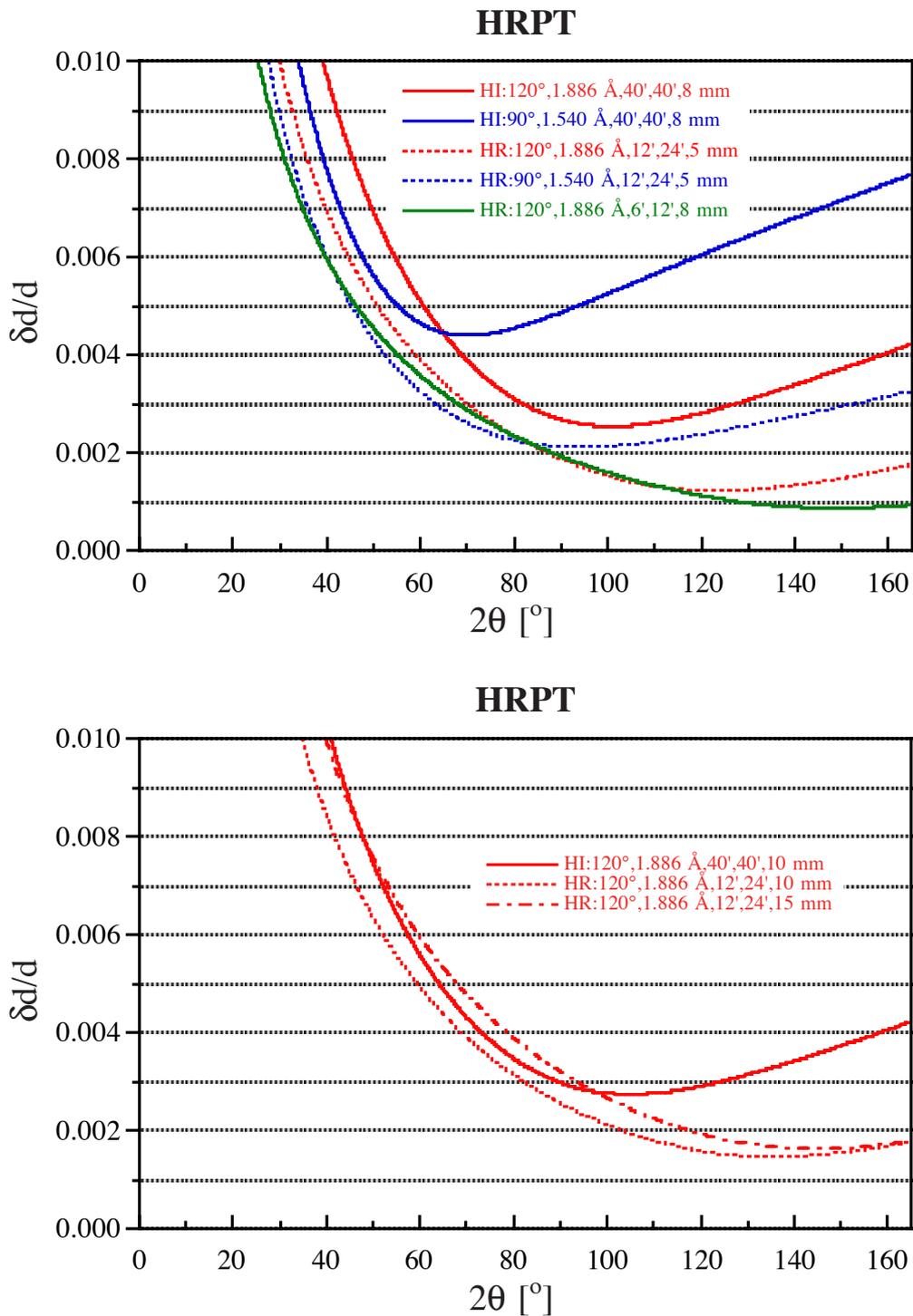


Fig. 10a: Calculated resolution functions of HRPT. d = lattice spacing. The legend indicates high intensity (HI) and high resolution (HR) modes of operation, $2\theta_M$, neutron wavelength λ , collimations α_1 , α_2 and the sample diameter.

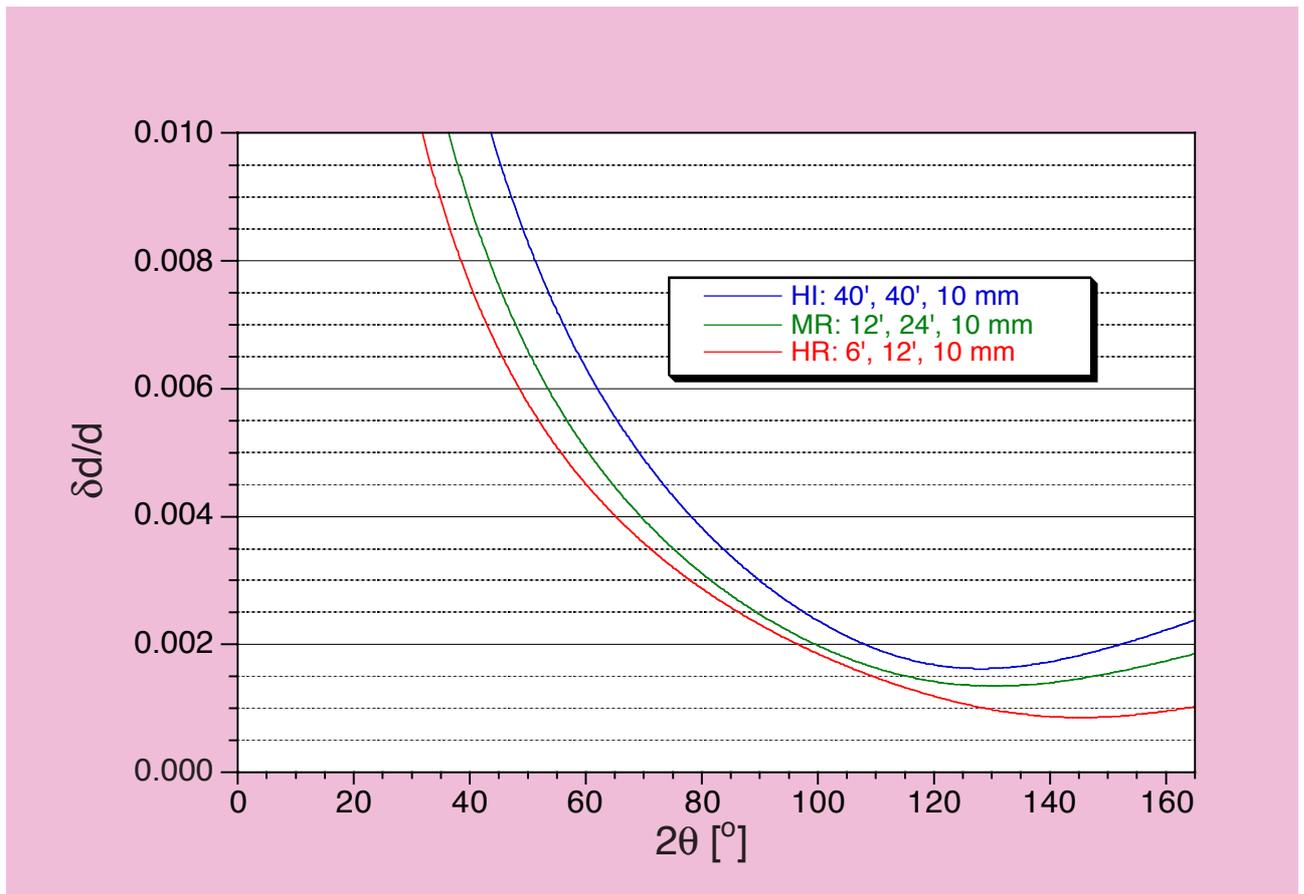


Fig. 10b: Observed resolution functions of HRPT for $2\theta_M = 120^\circ$ (based on FWHM values).

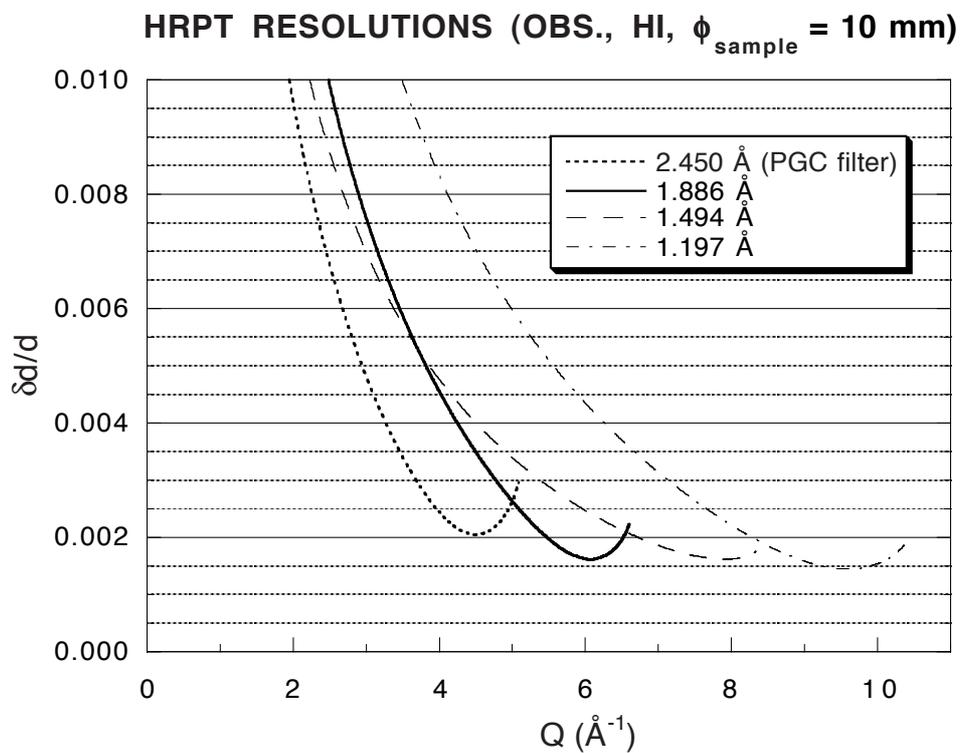


Fig. 10c: Observed high intensity resolution functions of HRPT for $2\theta_M = 120^\circ$ as a function of momentum transfer Q and neutron wavelength λ .

As indicated in Fig. 10, one may tune the resolution of HRPT to a large extent by choosing the appropriate instrumental parameters such as collimations and sample diameter, provided that sufficient neutron intensity remains at SINQ. Program PRESOL based on refs. [3, 4] may help in this choice.

A10. Standard samples

1. **Si standard** reference material 640b from National Bureau of Standards, Gaithersburg, U.S.A.: $a = 5.43094(4) \text{ \AA}$ at 298 K – **for neutron wavelength calibration**,
2. Y_2O_3 : $a = 10.6038 \text{ \AA}$, space group I a 3,
3. $\text{Na}_2\text{Ca}_3\text{Al}_{12}\text{F}_{14}$ (+ a trace of CaF_2): $a \approx 10.249 \text{ \AA}$, space group I 2₁ 3,
4. Al_2O_3

A11. Examples of powder measurements and profile refinements

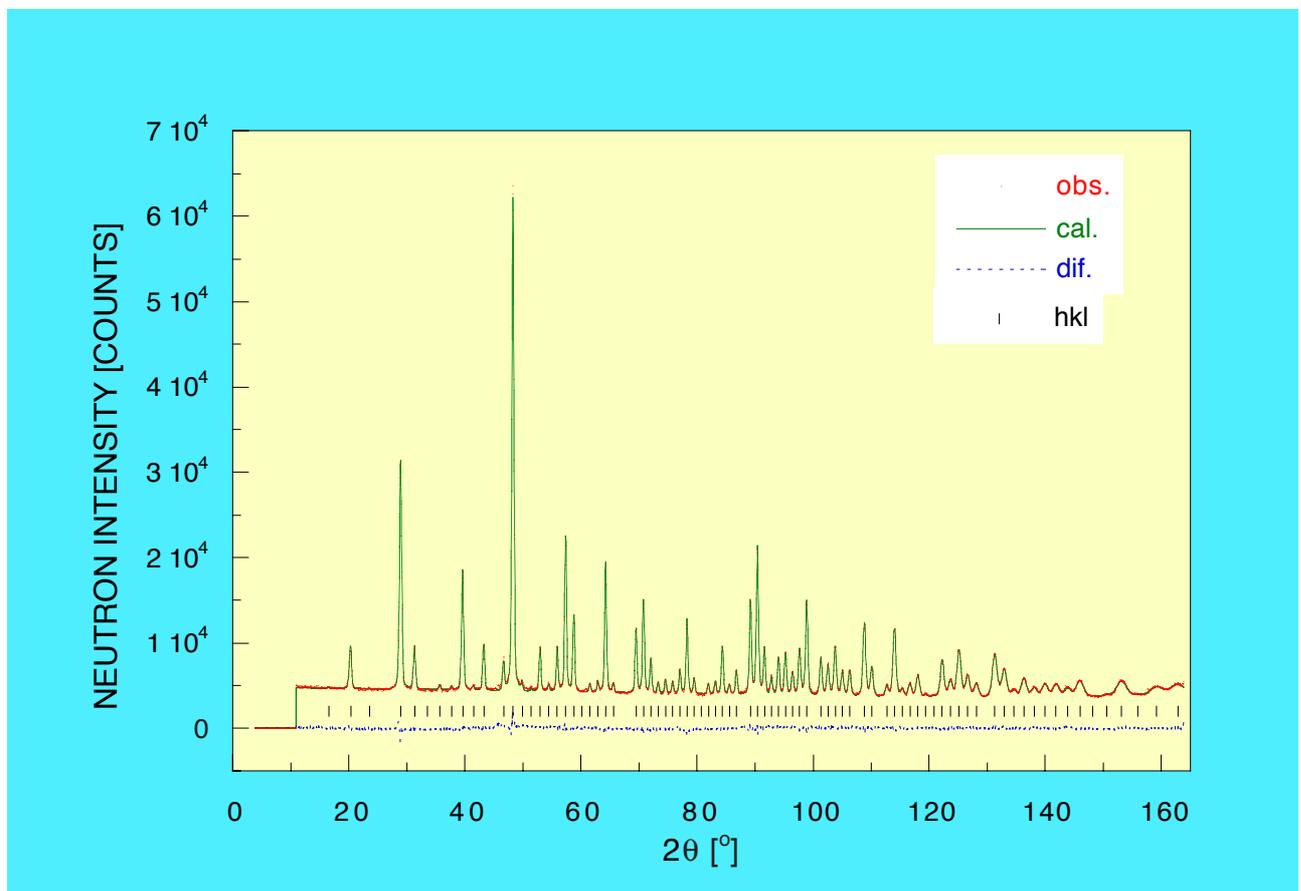


Fig. 11: Observed, calculated and difference neutron diffraction pattern of Y_2O_3 at $T = 293 \text{ K}$ (sample diameter 8 mm) with $\delta 2\theta = 0.05^\circ$ for $2\theta_M = 90^\circ$, $\lambda = 1.539 \text{ \AA}$, $\alpha_1 = 12'$ and $\alpha_2 \approx 24'$.



Fig. 12a: Observed, calculated and difference neutron diffraction pattern of $\text{Na}_2\text{Ca}_3\text{Al}_{12}\text{F}_{14} + \text{CaF}_2 + \text{V}$ from the sample container (sample diameter 5 mm) with $\delta 2\theta = 0.05^\circ$ for $2\theta_M = 120^\circ$, $\lambda = 1.197 \text{ \AA}$, $\alpha_1 = 12'$ and $\alpha_2 \approx 24'$.

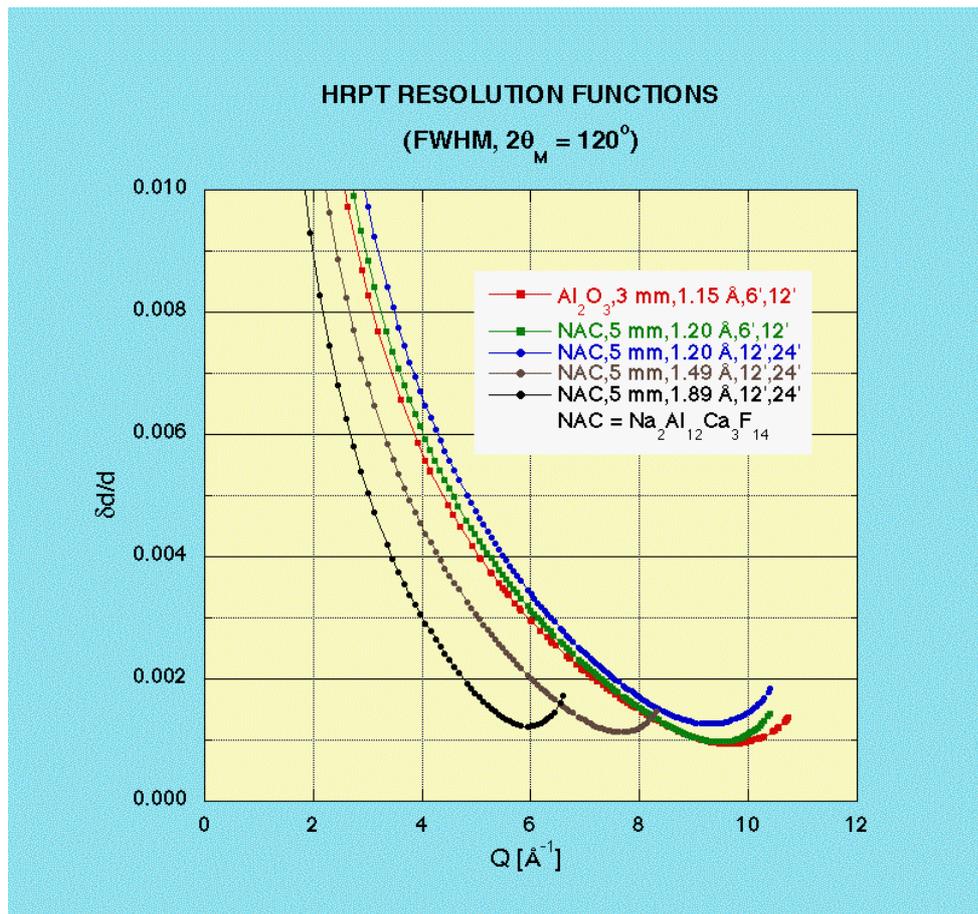


Fig. 12b: HRPT resolution functions as a function of momentum transfer Q , based on the total FWHM of Bragg peaks as obtained from FullProf.

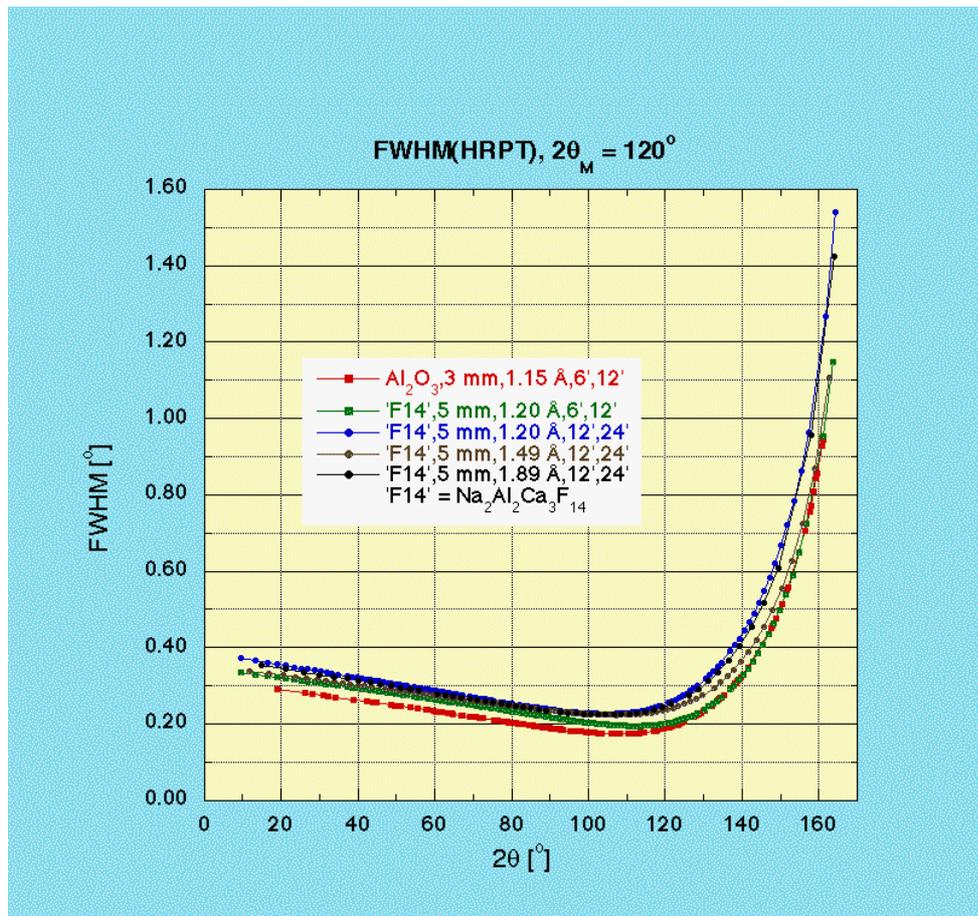


Fig. 12c: FWHM(HRPT) as a function of scattering angle, as obtained from FullProf.

A12. References

- [1] P. Fischer, G. Frey, M. Koch, M. Könnecke, V. Pomjakushin, J. Schefer, R. Thut, N. Schlumpf, R. Bürge, U. Greuter, S. Bondt, and E. Berruyer, High-resolution powder diffractometer HRPT for thermal neutrons at SINQ, *Physica B* **276**, 146-147 (2000).
- [2] P. Fischer, L. Keller, J. Schefer, and J. Kohlbrecher, Neutron Diffraction at SINQ, *Neutron News* **11**, No. 3, 19-21 (2000).
- [3] A. Dönni, P. Fischer, L. Keller, V. Pomjakushin, Y. Nemoto, T. Goto, and S. Kunii, The Cubic to Trigonal Phase Transition in HoB₆ Measured on the New Powder Neutron Diffractometer HRPT at SINQ, *J. Phys. Soc. Japan.* **70**, Suppl. A, 448-450 (2001).
- [4] A. W. Hewat, *Nucl. Instr. and Meth.* **127**, 361 (1975).
- [5] G. Caglioti, A. Paoletti, and F. P. Rici, *Nucl. Instr.* **3**, 223 (1958).

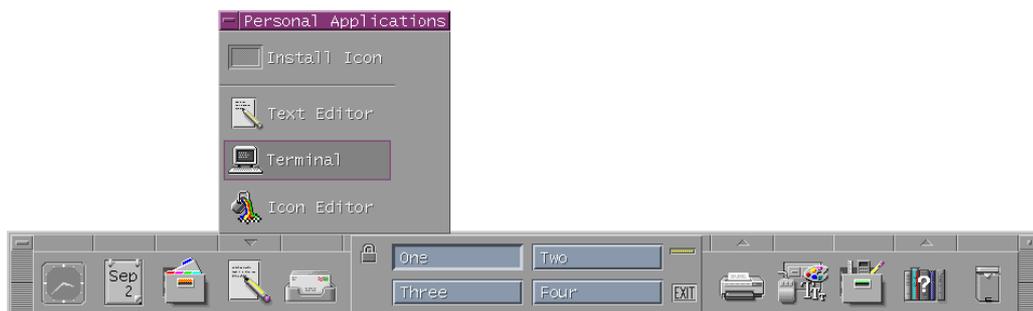
B) HRPT COMPUTER CONTROL

INSTRUMENT COMPUTER: Unix alpha workstation: `lnsa11.psi.ch`,
internet address `129.129.138.21`

INSTRUMENT CONTROL SOFTWARE: SICS, written by M. Könnecke,
see <http://lns00.psi.ch/> DMC and HRPT

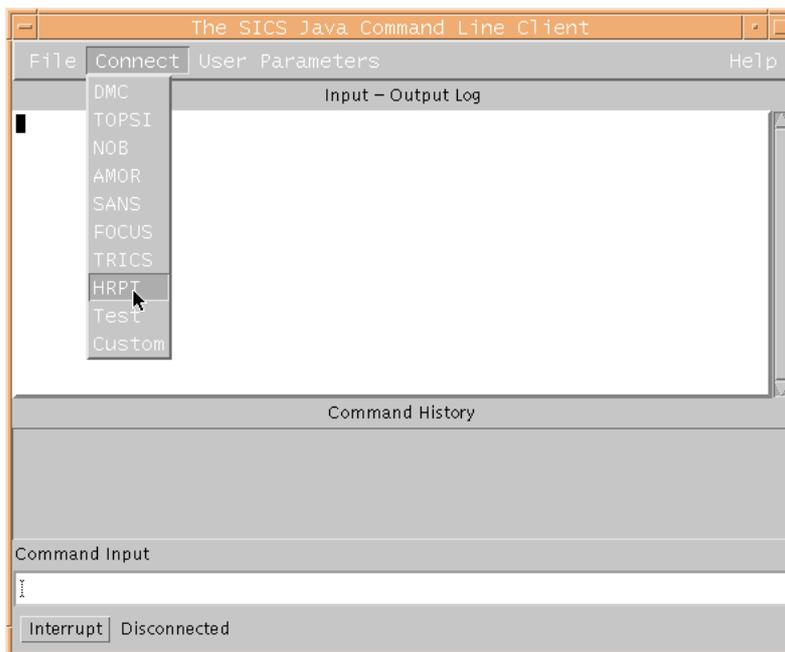
B1. How to log into the HRPT computer

After starting up the workstation click on the *XDMCP* button and choose *LNSA11.PSI.CH*. Log in with username *lnsg* and password `*****` (case sensitive, this is a Unix machine). Wait for the Common Desktop Environment to start up. Open a terminal window (*dterm*) as shown below. You will be asked for your last name and if this is your first login a subdirectory `/data/lnsg/<last name>/` will be created.



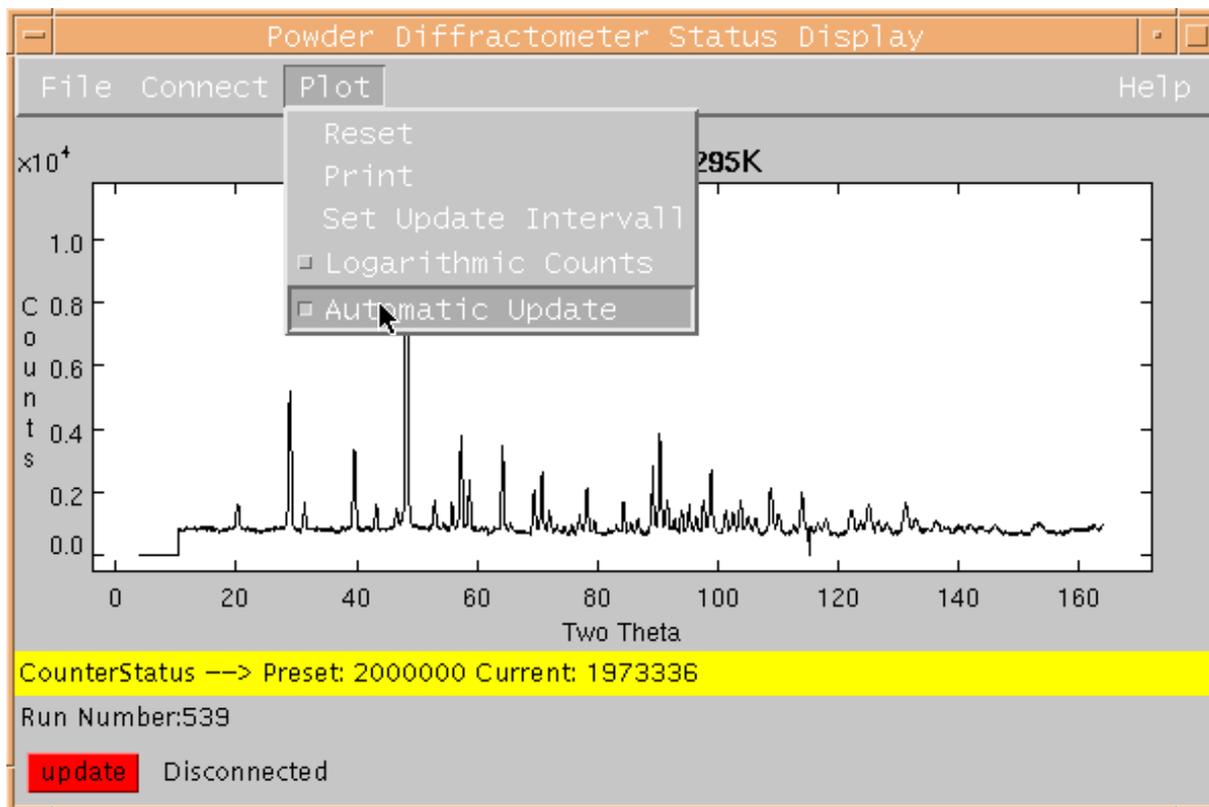
B2. How to start the instrument control program SICS

Activate the window *dterm* and type the command `sics&` (& means that the program runs in the background.) at the prompt. After a few seconds the *SICS Java Command Line Client* appears on the screen. Connect to HRPT.

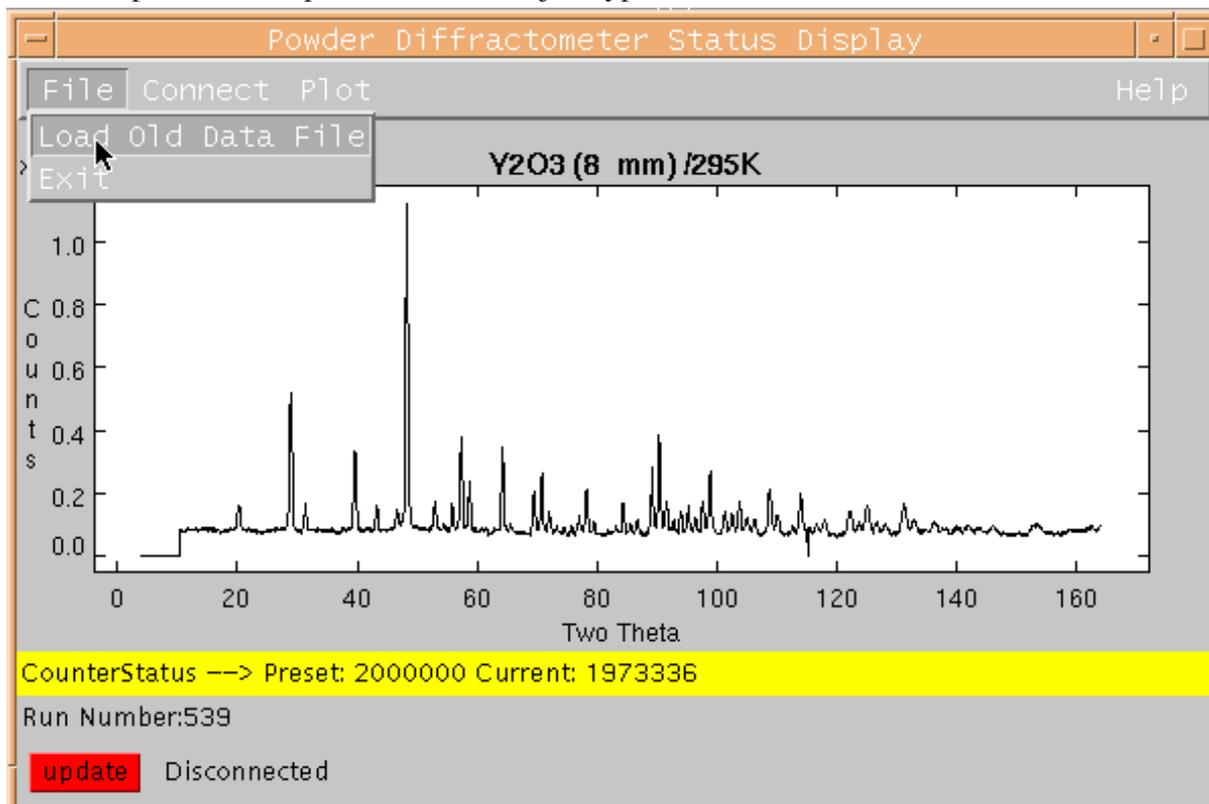


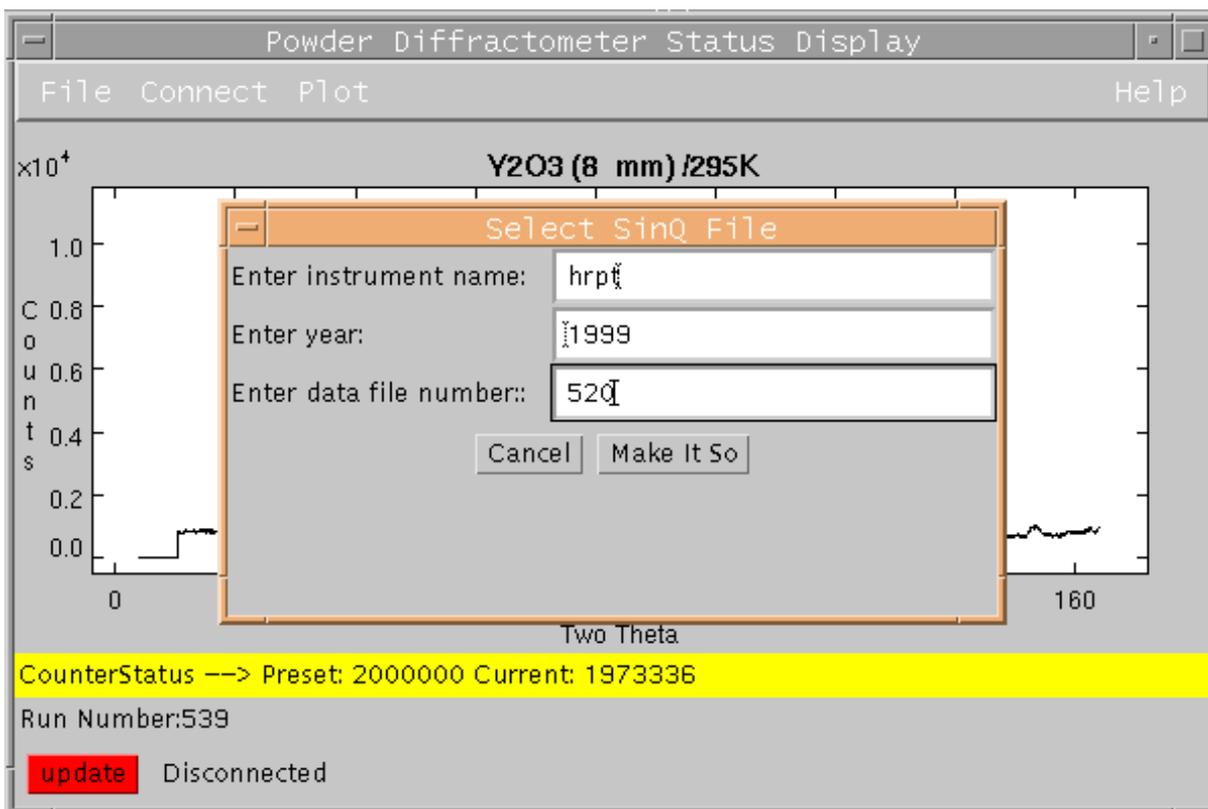
After connecting to HRPT you are able to read out instrument parameters like motor positions and temperatures, but you do not have the privilege to move the instrument or start the data collection, i.e. you have passive control of the experiment only. Only on the terminals in the HRPT cabin or on the one close to HRPT you are also allowed to have active control of the instrument. To get the privilege select *Set Rights* from the *User Parameters* menu.

The status display shows the content of the histogramming memory. If the measurement is running, you can update the display either by hitting the red *update* button in left bottom corner of the window or by activating the *Automatic Update* function of the *Plot* menu.

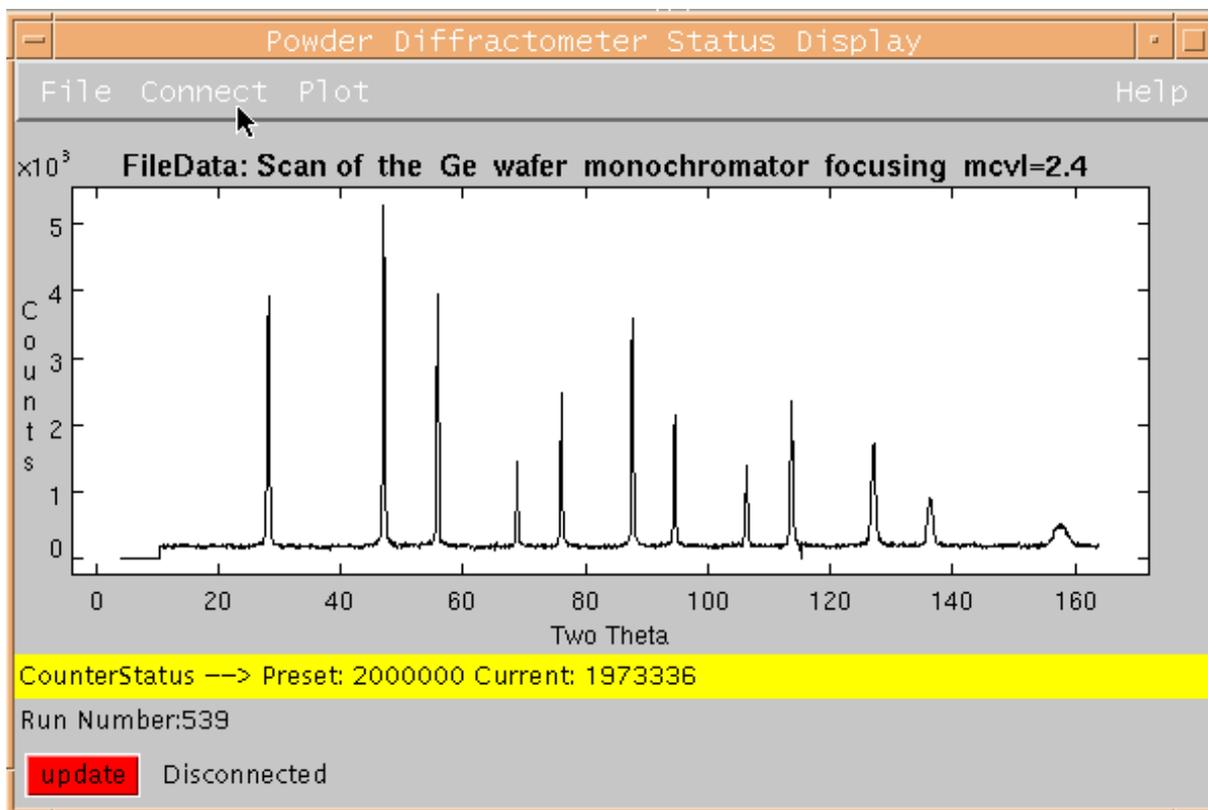


You can also use the status display to view old data files. To see previous measurements, select *Load Old Data Files* in the *File* menu. You will be asked for the file number of the data file. To view for example the file hrpt005201999.hdf, just type 520.





By hitting the red *update* button the display will return to the current measurement.



For more information click on *Help* in the top right corner of the *Powder Diffractometer Status Display*.

B4. How to start a measurement on HRPT: The SICS commands

HRPT can be controlled by typing SICS commands into the *Command Input* field of the *SICS Java Command Line Client*. A detailed description of the SICS command language and all SICS commands concerning HRPT can be found on the world wide web addresses <http://lns00.psi.ch/>. For most experiments the following few commands are sufficient.

<parameter>

Displays the current value of <parameter>. For example the command **temperature** or **tt** displays the current temperature, the command **a4** shows the current position of angle 4. There are two angles that users frequently use:
a3: position of sample table,
a4: position of detector bank, i.e. scattering angle 2Θ of the first detector.
Examples: **temperature,a4,a3**

drive <motor> <new value>

Drives the selected motor including **beam reductions cex1**, **primary collimators cex2**, **monochromator angle $\omega_M = moml$** and **focusing mcv1** to the new position. **When rotating the sample table or moving the detector bank, be very careful and watch out for anything that might obstruct the movement. The movement can be stopped by clicking on the *interrupt* button in the left bottom corner of the *SICS Java Command Line Client* window or in case of danger by pressing a **black emergency button**.** Never change an angle of the instrument without being instructed by the instrument responsible.
Note: a4 corresponds to the first of 1600 detectors of HRPT. Usual range $2^\circ \leq a4 \leq 5^\circ$!
Examples: **drive a4 5**; **drive temperature 20** (waits until the temperature is within tolerance limits !)

run temperature <value>

Sends new set temperature to the temperature controller.
Example: **run temperature 2.5**

wait <seconds>

No action during the given time in seconds.
Example: **wait 300**

<variable> <new value>

Each variable can be set by typing its name followed by the new value. **The variables *user*, *sample* and *title* must be set before every measurement.**
Examples: **user name**,
lambda 1.886,
sample xxx_8mmx50mm
title sample_293K_1.539A_12'_14/14mm

counter setmode <mode>

Sets the default preset to mode, where <mode> is either **monitor** or **timer**

count <mode> <preset>

Starts the counting (, if the monitor counts exceed a **threshold value**). For timer mode the preset is given in seconds. After reaching the preset value the data will be automatically stored.
Examples: **count monitor 1000000**, **count timer 10**

repeat <n> <mode> <preset>

Repeats the count command n times, n files will be stored.
Example: **repeat 4 monitor 1000000**

fileeval </path/filename>

Runs a command file. The command file may contain any SICS command.
This is the regular way to perform a measurement on HRPT.
Example: **fileeval /data/lnsg/username/user.run**

Temperature control:

If the SICS command **temperature** or **tt** does not read out the temperature, then type **tecs on** in the *Command Input* field of the *SICS Java Command Line Client*. Stop of temperature readout by means of instruction **tecs off**.

Unix instruction **tecs** and afterwards **plot** (or **varwatch&** with option log to file in file menu) may be used to control the time dependence of the temperature.

To start the measurement use the **count, **repeat** or **fileeval** command. To stop the measurement click on the **interrupt** button in the left bottom corner of the *SICS Java Command Line Client* window.**

Command files such as 1p49.run **to set the lambda in the high intensity mode** may be copied at lnsa11 from /home/HRPT/lambda/.

***** Do not measure longer than about an hour per run because of possible problems during a measurement.** Program **addit** permits you to sum up individual files to a merged name.dat file ! **Put in a maximum of information on the experimental parameters in title etc. ! Be careful to position a4 (= position of first anode wire !) during measurements only in a small range such as for an intermediate step. Do not forget the shielding around the sample opposite to the detector ! *****

Example of a simple command file, to be executed by fileeval /data/lnsg/name/Y2O3.run:

```
user name
sample Y2O3_8mmx50mm
temperature tolerance 3
drive temperature 100
wait 300
drive a4 3.95
title Y2O3_8mmx50mm_100K_1.53A_12'_14/14mm_a4=3.95
count monitor 3000000
drive a4 4.0
title Y2O3_8mmx50mm_100K_1.53A_12'_14/14mm_a4=4.00
count monitor 3000000
...
```

More elegant command file example.run (may be copied at lnsa11 from /home/HRPT/lambda/):
(Step 0.35 is used instead of 0.05 for safer a4 positioning and in case of varying detector channels.)

```
user name
lambda 1.494

set preset 1000000
set step 0.35

tt tolerance 50
tt ramp 15
drive tt 500
wait 300

for {set i 1} { $i <= 500} { set i [ expr $i +1]} {

for {set a 4.65} {$a < 5.01} { set a [ expr $a + $step]} {
  catch {drive a4 $a;} msg
  title xxx_1.494A_HI_furnace_T=500K,a4=$a,N$i
  sample xxx_V6x25mm_in_furnace,a4=$a,N$i
```

```

count monitor $preset;
}
}

```

fileeval /data/lnsg/name/name.run

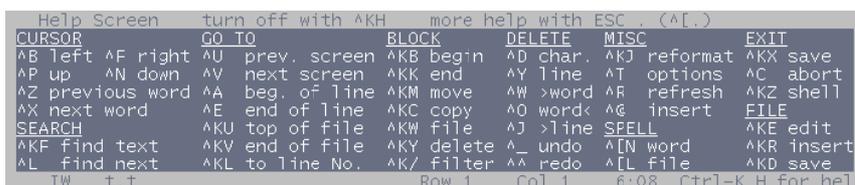
B5. How to edit the command file

There are several ways to create and/or edit a command file:

(i) You may use the most convenient **nedit** or you can click on the **editor icon** (pencil) of the Common Desktop Environment control bar and the built in editor will start up.



(ii) Activate the window *dterm* and type **joe <filename>** at the prompt and the joe editor will open the file <filename>. If the file doesn't exist, joe creates a new file. To get the help feature of the joe editor with a description of the editor commands, use **<ctr>k <ctr>h** in the editor window. Caution: Occasional editor problems due to resizing of the window !



(iii) Or you can activate the window *dterm* and type **emacs <filename>** at the prompt.

Inside emacs: (**ctl** means control button; **esc** is equivalent to the **alt** button at some terminals.)

- **ctrlxctlc** exit (if the file was not saved, you will be asked: Save file ?),
- **ctrlspace or ctrl@** to set the mark for cut or paste, move the cursor to the end of the desired region,
- **ctrlw** for cut,
- **escw** to copy,
- **ctrlp** to paste,
- **ctrlk** to kill a line,
- **ctrlp** to paste a line,
- **escd** to delete a word to the right,
- **ctrlld** to delete a character to the right,
- **ctrlg** universal cancel (if you get lost use it several times),
- **ctrlxu** undo,
- **ctrlhctrlh** help.

B6. Scans of parameters

(generally only for instrument responsables)

For scans of e. g. monochromator angles etc. with the proton current as monitor:

(cables of p current monitor and of neutron monitor to be interchanged)

Instructions:

```

counter setexponent 0,
scan mode monitor,

```

scan setchannel 4 (to count with the neutron monitor),
sscan var1 start end npoints preset and
topsistatus& for control and plot,
in menu Connect: **HRPT** and
at the end in menu File: **Exit**.
(Later topsistatus will be called scanstatus.)

Files such as hrpt001371999.**dat** are produced from such measurements.

These files may be copied also during the measurement from Insa11, **/home/HRPT/data/** .

It should be noted that the **file numbers** are **unique**, i. e. name.dat and name.hdf files cannot exist with the same number.

In this context useful command: **peak** -> peak center etc.

B7. How to add, subtract and transform data files

The HRPT raw data files *hrptxxxxyear.hdf* (e.g. hrpt008251998.hdf) are stored in the NeXus format. The programs **addit**, **subit** add or subtract such files, correct the data for differences of efficiency of the 1600 counters of HRPT's position sensitive detector (V single crystal calibration) and transform the result into an ascii file name.dat with the well known DMC data format:

```
HRPT, Name_V_302K_1.494A_HI,a4=5.0,N25
lambda= 1.49400, T= 0.000, dT= 0.000, Date=2002-07-25 07:28:57
 4.650 0.050 164.900 66500000., sample="LiBD4_V_9/7mmx50mm_osc.,a4=5.0,N25"
1053. 1087. 1122. 1258. 1393. 1522. 1651. 1783. 2049. 2148.
2361. 2441. 2609. 2748. 2983. 2897. 3181. 3383. 3468. 3498.
..
 30.8 32.5 34.1 36.0 37.9 39.6 41.4 40.3 46.2 47.4
 50.2 50.3 52.4 53.6 55.3 55.2 57.2 60.3 59.6 60.9
..
 86.6 85.9 85.9 86.0 84.2 84.7 82.8 84.6 77.5 82.4
 80.3 78.2 77.5 76.8 73.4 70.0
Filelist='hrpt/2002/4976-5040,5042-5091'
a4=4.65 5.; Numor=4976 5091; Npkt=1596; owner='X.YYY'
bMon=0.; pMon=39572812. 167147680.; muR=0.; sMon=5.E5 2000000.
```

The DMC data format header consists of a three lines:

First line: HRPT, title,

second line: neutron wavelength, temperature information and date,

third line: start value of scattering angle 2Θ , 2Θ step, 2Θ end value, monitor and sample.

The header is followed by the block of intensities and the block of statistical errors of the corresponding intensities, evt. followed by other instrument parameters.

Examples of files to be used in **addit**: 2968-3037,3085-3180 or name1.dat,name2.dat

B8. How to view data files

To view raw data files `hrptxxxxyear.hdf`, use the `powderstatus` display or activate the `dterm` window and type `fit` at the prompt. You will be asked for the run number. To view e. g. the file `hrpt001252002.dat`, type `HRPT/125`. Use in `fit` bars `no` and connect `yes` for diffraction patterns.

To view converted files with DMC format, activate the `dterm` window and type `fit` at the prompt. You will be asked for the filename. Follow the instructions given by the program. At the `fit` prompt type `help` for instructions about the plot and fit features of the program.

In particular program `fit` may be used with instruction `bgedit` to determine visually background values stored in a file `name.bg`.

With `fit` and instruction `plin number(real)` or `plog number` one may easily plot in linear or logarithmic scale a series of files, e.g. as a function of run number or temperature, on a single plot with constant vertical shift `number` between the files.

Instruction `trans` in `fit` permits to change from scattering angle to momentum transfer Q or interplanar spacing d .

B9. How to use PC and Mac terminals of HRPT

In addition to the main workstation terminal in the experiment cabin of HRPT which is running under Unix, another PC terminal close to the instrument may be used for easy access and quick measurements. The PC operates under Linux. For details see special instruction sheet close to the terminal.

A second PC in the experimental cabin runs under WindowsNT. This PC may be also used as Unix terminal via JavaWebStart for the SICS and `powderstatus` clients.

Use username `Insguest` and password `XXXXXX`.

Moreover for primary data evaluation or online refinements PC versions of `FullProf`, `GSAS`^{*)}, `Jana2000`, `Fox`, `Atoms` etc. may be used. `Netscape`^{*)} permits to see the accelerator status. Users should create their own folders in folder `Users`, but should not change the programs etc. From the PC data may be also stored to floppy discs or CD-s.

Also a Mac is available in the HRPT cabin for `Netscape`^{**)}, special graphics, text editing etc.

For user laptops, an internet connection is provided in the HRPT cabin.

From and to PSI, direct ftp connections are only possible via the special ftp server `ftp.psi.ch` with user name `anonymous` and the user e-mail address as password. There are directories `PSI_incoming` and `PSI_outgoing` available for user subdirectories.

^{*)} Program `fullp#8-gsas filein fileout` transforms data with DMC format to GSAS format.

^{**)} In particular the inorganic data file ICSD is online available via the LNS diffraction homepage.

Appendix

B10. Data evaluation, profile refinement, plot

After finishing the measurements on HRPT, users should log onto **lnsa15.psi.ch** under account **lnsg** with password *********.

Various unix programs such as **fit**, **poly**, indexing programs, **latcon**, **lazy**, **fp2k (FullProf)**, **jana2000** (Needs instruction **unlimit** before program start.) and **expo** (for direct structure determination) may be used, see </data/lnslib/src/diffraction/manuals/unixprograms.readme> and http://www1.psi.ch/www_sing_hn/SINO/instr/diffraction/diffraction_links.html. The FullProf manual is accessible under http://www1.psi.ch/www_sing_hn/SINO/doc/fullprof.html and for the newest **fp2k2** version as FullProf_Manual.pdf in the directory </data/lnslib/src/diffraction/manuals/>. Starting values for the fit of the instrumental parameters are:

Observed resolution parameters for Si standard sample with diameter 10 mm.

$2\theta_M$ [°]	λ [Å]	α_1 [']	α_2 [']	U	V	W
90	1.538	40	40	0.392	-0.595	0.331
90	1.538	12	24	0.224	-0.373	0.219
90	1.537	6	12	0.094	-0.183	0.149
120	1.886	40	40	0.109	-0.308	0.317
120	1.886	12	24	0.065	-0.179	0.194
120	1.885	6	12	0.022	-0.080	0.128
120	2.450	40	40	0.160	-0.384	0.352

$\lambda = 1.538$ Å, $\alpha_1 = 12'$, $\alpha_2 = 24'$: NPROF = 7, $y = 0.095$, $asympt1 = 0.062$, $asympt2 = 0.014$, $2\theta_0 = -0.16$.

Observed resolution parameters for Si standard sample with diameter 5 mm.

$2\theta_M$ [°]	λ [Å]	α_1 [']	α_2 [']	U	V	W
120	1.886	40	40	0.101	-0.284	0.276

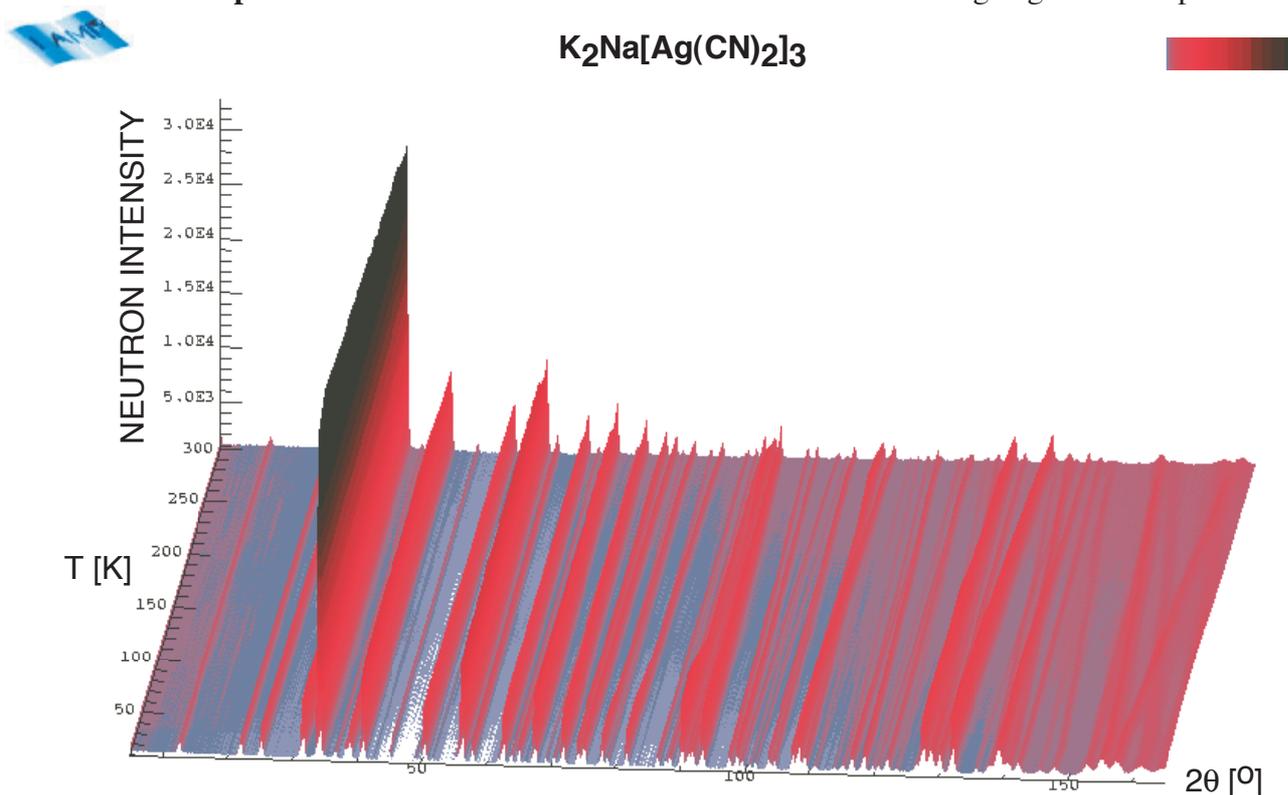
Observed resolution parameters for Na₂Al₂Ca₃F₁₄ and Al₂O₃# standard samples ($2\theta_M = 120^\circ$, asymmetry limit 50 to 60 degrees).

Diam. [mm]	λ [Å]	α_1 [']	α_2 [']	U	V	W	y	Asy1	Asy2
3#	1.154	6	12	0.033580	-0.096768	0.091108	0.028910	0.03942	0.02083
5	1.197	6	12	0.033255	-0.105217	0.107496	0.037424	0.05037	0.01750
5	1.197	12	24	0.053243	-0.145157	0.135151	0.038867	0.001	0
5	1.197	40	40	0.075247	-0.212993	0.190286	0.085888	0.08391	0.03293
10	1.197	12	24	0.073257	-0.205282	0.219835	0.031848	0.03330	0.00572
10	1.197	40	40	0.085745	-0.23353	0.24811	0.069809	0.07078	0.02386
5	1.494	12	24	0.038696	-0.108848	0.118303	0.021007	0	0
5	1.494	40	40	0.052947	-0.140426	0.165178	0.050730	0.03480	0.02283
10	1.494	12	24	0.050066	-0.151779	0.192683	0.021872	0	0
10	1.494	40	40	0.060263	-0.148955	0.212500	0.040985	0.02441	0.01299
5	1.886	12	24	0.052979	-0.140267	0.136927	0.015596	0	0
5	1.886	40	40	0.101640	-0.287520	0.278734	0.035677	0.09396	0.05031
10	1.886	12	24	0.071761	-0.204817	0.226112	0.016048	0	0
10	1.886	40	40	0.104489	-0.293687	0.328811	0.028520	0.04750	0.02477

Ref. for FullProf: J. Rodriguez-Carvajal, Physica B **192**, 55 (1993).

For plotting name.plt files from lazy and for profile refinement plots, program fit may be used (with 'prf' parameter 3 in FullProf).

For 2-D and 3-D plots of of diffraction data as functions of both scattering angle and temperature



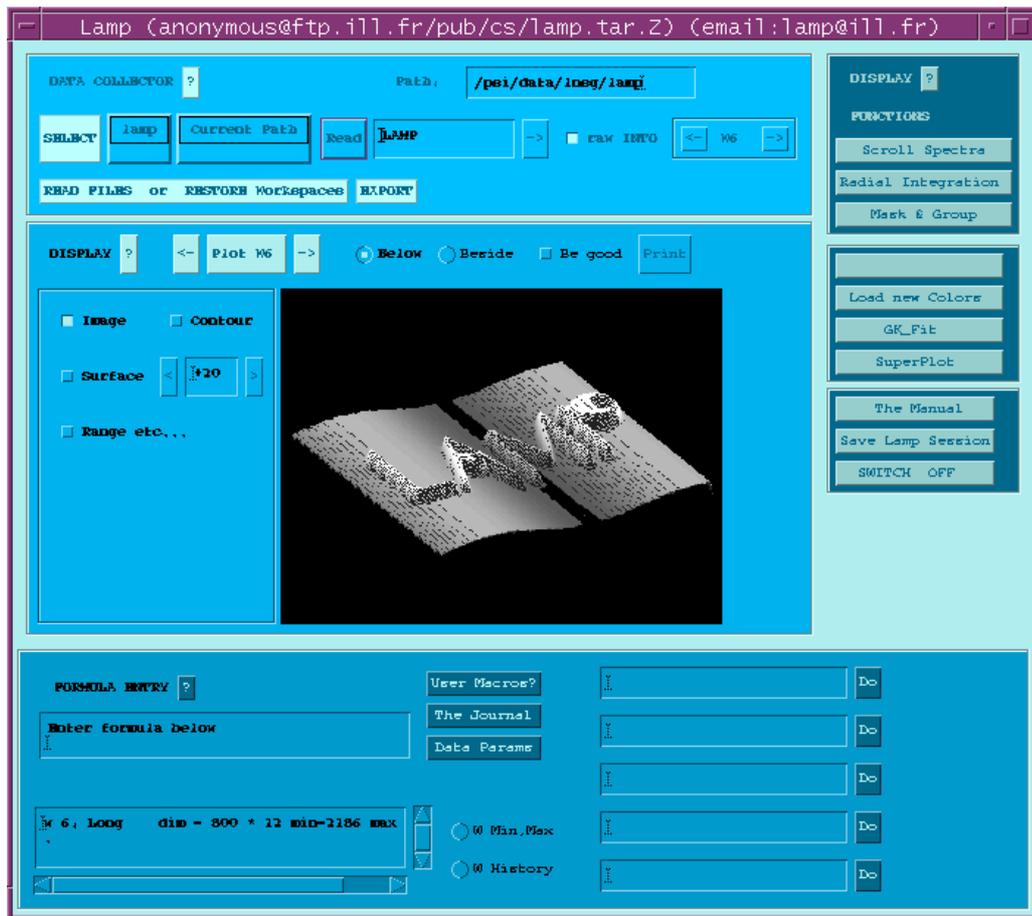
Example of a 3-D plot of HRPT data.

program lamp may be used, see subsequent instructions.

To create "three-dimensional" plots of your diffraction data (e.g. temperature dependence of the diffraction pattern), the data sets must be converted into a special data format using the program *clamp* (convert to *lamp* format). To start **clamp** activate the window *dterm* and type the command **clamp** at the prompt. The program will guide you interactively. **clamp** is able to convert the same file formats that are recognized by the program **fit**, in particular raw data format as well as DMC format. Scattering angle range and monitor are expected to be the same for all data files. Two output files are created: **LAMP** and **LAMPascii**.

mclamp may be similarly used in case of real-time data to merge the data into smaller groups.

To start – on Insa15.psi.ch - the program **lamp**, activate the window *dterm* and type first the command **idlinit** and then **lamp** at the prompt. Now hit the **START LAMP** button in the lamp desktop window. After the main lamp window has opened, click on **DATA Access**. Choose **lamp** in the file format list and type in **LAMP** to read in the files created by the conversion program **clamp**. After hitting the **Read** button the data is read into a workspace and can be plotted.



Click on **The Manual** for details of the possibilities of **lamp**. Here are the most important features:
 Click on **Range etc...** to set the plot range; always select **Regular Grid** !
 Click on **Be good** to control several plot options like shading of the surface, plot angle, title etc.
 Select **Below** to view the plot in the main window, **Print** for a postscript file of this plot.
 Select **Beside** to view the plot in a separate window, to change the color settings and to create postscript files for prints on e. g. laser color printers.
 Select **Surface** for three-dimensional surface plots (+20 = view angle, may be changed).
 Select **Contour** for contour plots, i.e. the projection onto the xy-plane (in this example: the 2θ -Temperature plane).

B11. Useful unix and further SICS commands

cat file	yields print of file on screen
cd name	change directory to directory name (blank -> home directory)
cd ..	move up in the directory tree
cp file1 file2	copy file1 to file 2
commandlog interval	to modify (as lnsmanager) e.g. time stamps, def. = 60 min.
counter setthreshold 1 50	yields monitor counter threshold 50 (needs lnsmanager privilege)
(counter setthreshold 1 0	yields monitor counter threshold 0 to count without neutrons in timer mode)
counter stop	stop of counting
graphit change	moves graphite filter (up or down)
lp -d SINQ_THO_1 file	prints file on laser printer close to HRPT hutch (see CUPS details)
ls	list of files
mkdir name	make directory name
nedit&	yields a menu driven editor
par list	yields information on parameter with name par, e. g. limits
pwd	print working directory
ps -a	show all active jobs
rACS&	yields accelerator status running in the background
rm file	remove file
sumvar n1-n2	yields HRPT measurement history between n1 and n2 as run numbers
or ownerhrpt n1 n2	
One may change the sumvar output by changing setenv sumvars, e.g. setenv sumvars "Numor:5,Date,Title,Temp,Counts,sMon,pMon,sample", to get in particular also the Sum count over all detectors.	
sicstail <n>	yields on lns11.psi.ch the end of the log file, n = number of lines shown
who	show all users
whoami	show on which account you are logged in
xcalc&	yields a calculator

For more detailed basic information on unix see: <http://lns00.psi.ch/misc/unixbasic.pdf>.

B12. Troubleshooting

If possible, look with unix command **sicstail** at the log file information. Then try with sics instruction **counter stop**.

If the SICS server does not respond, do the following:

- Open a new terminal window or use **su - HRPT** or
- login to lns11 as user HRPT and password ##### by using the command **login**,
- type **killsics**,
- type **killtecs** in case of temperature control,
- type **startsics**,
- type **logout**.

Further possibility to restart SICS from the SICS Java Command Line Client:

- **sics_exitus** (as lnsmanager),
- connect again to HRPT.

For more trouble shooting hints concerning the SICS software check out the www page <http://lns00.psi.ch/sics/trouble.htm>.

B13. Summary of HRPT motors

cex1, a17:

Inner collimator drum, beam reductions

cex2, a18:

Outer collimator drum, collimation α_1

momu, a1:

Omega rotation of upper monochromator crystal [perfect Ge(hkk)]

mtvu, a12:

Translation vertical to the upper monochromator crystal (main surface plane)

mtpu, a13:

Translation parallel to the upper monochromator crystal

mgvu, a14:

Tilt goniometer vertical to upper monochromator crystal

mgpu, a15:

Tilt goniometer parallel to upper monochromator crystal

mcvu, a16:

Vertical focusing curvature of upper monochromator crystal [perfect Ge (hkk) plate]

moml, b1:

Omega rotation angle of lower monochromator crystal [**wafer type Ge (hkk)**]

mtvl, a22:

Translation vertical to the lower monochromator crystal

mtpl, a23:

Translation parallel to the lower monochromator crystal

mgvl, a24:

Tilt goniometer vertical to lower monochromator crystal

mgpl, a25:

Tilt goniometer parallel to lower monochromator crystal

mcvl, a26:

Vertical focusing curvature of lower monochromator crystal

mexz, a37:

Monochromator lift position

mtt, a2:

2 θ monochromator, fixed to 120 or 90 degrees

som, om(ega), a3:

Omega rotation angle of sample table [degrees]

stt, th, a4:

2 θ of the first of 1600 detectors [degrees]

B14. CHECK LIST HRPT

- **Optimum neutron wavelength** chosen, i.e. adapted to the sample/aspect to be studied ?
 - **Optimum resolution (depending on sample diameter)/intensity** ? Collimations, beam reductions such as monochromatic neutron beam width (α_2) and evt. filters set right ?
 - **Direct beam stop okay** ?
 - **Additional shielding around sample opposite to the detector** mounted ? (**To be removed before moving a4 in a larger range !**)
 - **A3 and a4 softlimits okay** ?
 - Measurement file okay, i. e. monitor values/**measurement time per single run not longer than about 1 hour** ?
 - **Sample temperature/vacuum etc. okay** ?
 - **Radial collimator oscillating** ?
 - Detector cooling system working well ? Maximum detector temperature not exceeded* – see temperatures or warnings indicated at the back of the detector shielding (below) ?
 - LN₂ filter cold ?
 - Vacuum in beam tube before monochromator okay (to be seen on the TriCS side) ?
 - Cerca-PC working and display light reduced – to save the screen ? (In case of zero counts from the histogram memory the cause may be: “SCE is not connected”. This message may appear on the screen of the Cerca-PC. Restart of the Cerca program by G. Frey will be necessary.)
 - Evt. sample transmission to be measured.
 - **Log book information**, experimental report **properly completed** ?
- * In such a case G. Frey should be contacted.

B15. Contact persons

Instrument responsables

Peter Fischer, Vladimir Pomjakushin, Denis Sheptyakov,	WHGA/133, WHGA/133, WHGA/133,	Tel. 2094 Tel. 2094 (from 1.11.2002) Tel. 3070
--	-------------------------------------	--

SICS software

Mark Könnecke,	WHGA/247,	Tel. 2512
----------------	-----------	-----------

Electronics

Roman Bürge, Gerhard Frey,	WMHA/C22, WLGA/B19,	Tel. 4299 Tel. 4311 or 079-4747982
-------------------------------	------------------------	---------------------------------------

Mechanics

Peter Keller, Rudolf Thut, Roger Schneider	WHGA/245, WLGA/D26, WPGA/6	Tel. 2052 Tel. 2465 Tel. 4352
--	----------------------------------	-------------------------------------

Sample environment (cryostats, furnaces, liquid gases)

Markus Zolliker, Stephan Fischer,	WHGA/135, WLGA/D28,	Tel. 2089 Tel. 4118
--------------------------------------	------------------------	------------------------

C) SAFETY PROCEDURE AT SINQ

Before the Experiment:

Safety (<http://lns00.psi.ch/safety/safety.htm>) sheet has to be signed and posted at the experiment.

NOTE:

All active material has to be shipped according to international law using specialized companies to:
Paul Scherrer Institute, Att. SU (Active Samples), Bldg. OSUA, CH-5232 Villigen PSI

You are not allowed to ship such items to your local contact You are or to bring them in by yourself.

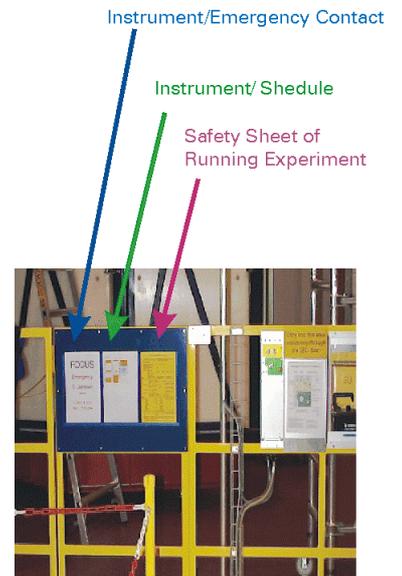
After the Experiment:

http://lns00.psi.ch/safety/samples_at_sinq.pdf

Note:

All active material has to be shipped according to international law using the SAR-company (for questions: Contact H.U. Aebersold, Tel. 4294)

You are not allowed to remove active material from PSI on your own.



Entrance Safety Table CDMPG20060409

All material brought into the SINQ-halls (TH/LH) is **considered to be active** as long as not stated inactive by a safety official (SU) from PSI. The instrument responsible is not allowed to make this safety procedure for you.

Samples must be stored temporarily in the special cabinet in the guide hall WNLA opposite of TA (every instrument responsible has a key). You are not allowed to take them to the hutches or to offices.

Store the sample in the transparent plastic box (-> one sample per box!) as shown to the right and include a label with the following information:

- Name, Adress/Phone/FAX, E-Mail
- Date, Experiment Number
- Spectrometer
- Material, Amount [g]
- Local Contact

Activity will be measured by SU.

Please **distinguish** between samples and materials:

- to stay at PSI (West and East)
- to exit PSI
- to exit Switzerland



