Running the Instrument

Aligning samples

There are essentially two methods of sample alignment, either by laser or using neutrons. The laser beam travels along the same path as the neutrons. The choice of method depends on the sample. For solid films or liquids where the laser can be reflected off the sample then the use of the laser is preferable. If the use of complex sample environment is used where the laser can not transmit through the sample, then alignment may only be possible with neutrons.

Laser alignment

To start alignment of solid samples using the laser, the perpendicular goniometer motion (PSI) must be adjusted so that the surface of the sample is perfectly horizontal. Then the following slit and detector arrangement is recommended:

Table 1:

Motion	Setting
S1	1.0 mm
S2	0.5 mm
S3	2.0 mm
S4	3.0 mm
THETA (detector angle)	0.35

1. Ensure that PHI (sample angle) is non-zero with respect of the laser beam remembering that the beam is inclined at 1.5 to the horizontal.
2. Adjust HEIGHT so that the laser beam fully illuminates the sample surface and gives a maximum in the reflected beam intensity.
3. Adjust PHI until the laser beam equally illuminates the slit at the back of the single detector slit. (If using the multidetector then PHI should be adjusted until the laser beam hits the pencil mark indicated on the face of the detector).
4. Redefine PHI on the control PC. This is achieved by the command
   ```
   > define phi=0.35
   ```
   For example would normally be set so that PHI=THETA.

If using liquid surfaces (without the supermirror) the detector angle should be set to 1.5 . The slits are slightly different since the sample is larger and given below. Since no goniometer is used then the only adjustable motion is the height of the sample. This should be moved until the reflected laser beam sticks centrally on the detector slits.

Neutronic alignment

The alignment by neutrons is essential for samples which are opaque to light such as solid-liquid cells. In these cases the instrument must be set up as indicated below. The sample will need adjusting so that PHI is defined and set to 0.0 and therefore parallel to the neutron beam. PSI may need adjusting to ensure that the sample is horizontal in the perpendicular direction. The HEIGHT should be moved so that the laser beam strikes the cell slightly one side of the interface you are aligning on, ie the silicon - PTFE interface.

Table 2: Neutronic alignment starting parameters.

Motion	Setting
S1	1.0 mm
S2	0.5 mm
S3	2.0 mm
S4	3.0 mm
THETA	0.0
PHI	0.0

The sample is now ready for alignment and is achieved via a minimum of 3 alignment runs. A typical example set of alignments is given below:

1. Height alignment with THETA = PHI = 0.0 Defines the centre of the interface by finding the half height intensity from going from the neutron transparent silicon to the neutron opaque PTFE in this case.
2. PHI alignment at THETA = 0.0 and HEIGHT at half height determines the zero position of the PHI motion on a slightly coarse scale.
3. PHI alignment at THETA = 0.25 Determines the fine adjustment of the PHI motion.

These scans are performed using a program "SCAN" run within OpenGENIE on the dashboard computer. "SCAN" expects to be told whether the alignment scan is a height (adjusts HEIGHT motion) or a angle scan (adjusts PHI motion). Then the start value of the scan, last point of the scan, number of steps and number of frames (1200 frames=1A) per step must be entered:

```
> scan <axis> <from position> <to pos.> <number of steps> <number of frames>
```

Scans must be extensive enough so that the full range of the scan is achieved. The results of the scan will be plotted to the screen during the measurement.
Example commands for scans are shown below:

```
> scan height -0.5 0.5 11 250
```
or
```
> scan phi 0.3 0.4 11 250
```

After completion of the scan the option of saving the results to an ascii file is given. A number of functions are available to fit the data with Gaussian type functions giving fitted

positions for the maxima or half height depending on the type of scan. However, it is often preferable to simply use the cursor which is available from the program menu. By typing c into the OpenGENIE window the user may obtain a cursor that may be used to select the point thought to be the maximum or half height position on screen and obtain a value for this position by typing x. After selecting the point type e to exit the cursor routine.

It should be noted that the program does not move the instrument to the selected point automatically, it is up to the user to do this!

A guide to parameters used for each of the scans is:

**Table 3:** Neutronic alignment scan starting parameters.

Scan	Motion	Start Value	Step Size	No. of Steps
Height	HEIGHT	-0.5	0.1	11
PHI = THETA = 0.0				
Angle	PHI	-0.5	0.05	13
THETA = 0.0, HEIGHT = half height				
Angle	PHI	0.2	0.01	11
THETA = 0.25, HEIGHT = half height				

The angle scan about 0.0 need only be carried out once, in general, in order to obtain a rough position for subsequent angle scans. Fine adjustment should be carried out in a reflection geometry. The height and angle scans should be repeated iteratively until no change is observed in the positions of the peak maxima. *It is the decision of the user as to whether the motions should be redefined after each scan.* The correct positions could be set once a final positions have been obtained satisfactorily. For the case of a series of samples being measured on a sample changer redefinition should only be performed for the first sample and then subsequent positions recorded relative to the defined values.

Dash Board Commands

A typical dash board display is shown below (see **Figure 1**). This screen shows the current status of the instrument. If this control is not present on the control machine double click on the SECI icon on the desktop to launch the SECI instrument control program.

Figure 1
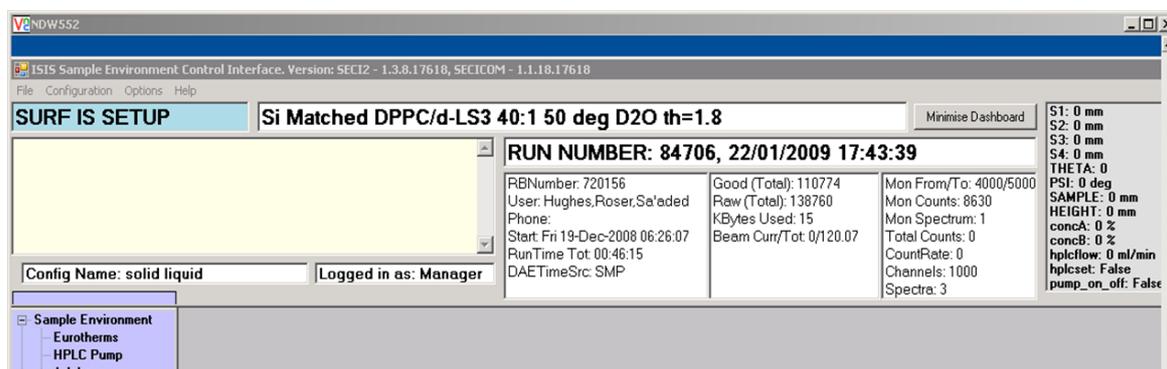
Commands for manual running of the instrument should only be submitted onto the dash board instrument control computer. Through an OpenGENIE window. Most commands are common to all instruments at ISIS although a number are specific to CRISP and SURF. A summary of the key commands are listed below in **Table 4.**

Table 4:

Command	Description
begin	Starts data collection, data is held in the DAE. The dash board will indicate RUNNING above the header block.
end	Ends the data collection and saves the data to file for archiving into a file SRF{run no}.RAW and therefore identifiable by the run number. The dash board run number will go into SETUP mode and increment the run number.
pause	Pauses the data collection, the dash board will indicate a PAUSE mode.
resume	Resumes the data collection after the PAUSE statement has been issued and will return to the RUNNING mode.
abort	Ends the data run without saving the data. The DAE goes into SET-UP mode but the run number is not incremented.
mv (theta, phi, height, s1, s2, s3, s4, sm_angle)	A command which must be used to move any of the instrument axes. It should be given in the following format: mv{motion} {position} where {motion} is the motion to move and {position} is the value to move to. An example is: mv THETA 0.5 which moves the detector to 0.5.
do "filename.gcl"	Runs a command file with the name filename.gcl which must be in the current directory that contains a procedure called runscript (See below)
load "filename.gcl"	loads a script file into OpenGENIE. This should be used if you wish to use a script with a procedure name other than runscript.

Writing Script Files

Script files are designed to allow multiple samples or angles (or both) to be run and therefore contain all the information required for changing slits, angles, and sample position. All the information for any automated control must be contained within the script. The commands are duplicates of everything that can be input manually into an OpenGENIE window on the control computer. A program called "Makescript" exists for generating script files automatically. This can be found on the side menu panel on the control computer. A typical file generated by MakeScript for looking at a sample at 3 angles is given below.

```
PROCEDURE runscript
#
# Script Generate by MakeScript
#
# Setting the number of periods in the DAE
change nperiods=1
#
mv s1=1.000 s2=0.500 s3=2.000 s4=2.000
mv theta=0.350 phi=0.350 height=0.000 sample=0.000
waitformove
change title="Sample 1 th=0.35"
begin
waitfor uamps=30.0
end
mv s1=2.000 s2=1.000 s3=3.000 s4=3.000
mv theta=0.800 phi=0.800 height=0.000 sample=0.000
waitformove
change title="Sample 1 th=0.8"
begin
waitfor uamps=60.0
end
mv s1=4.000 s2=2.500 s3=4.000 s4=4.000
mv theta=1.500 phi=1.500 height=0.000 sample=0.000
waitformove
change title="Sample 1 th=1.5"
begin
waitfor uamps=120.0
end
ENDPROCEDURE
```

Script files may be edited or written using notepad or any simple text editor but must be saved with a .gcl file extension.
For samples that may require repeated runs then the following must be added before and after the section which needs repeating:
```
LOOP
.
.
.
ENDLOOP
```

Loops may be added using makescript.

Looking at your data

Opengenie

OpenGenie is the current version of the ISIS data analysis software which is available for use on Windows, VMS, Linux and sgi from http://www.isis.rl.ac.uk/OpenGENIE/download.htm

Looking at Raw Data

Soon after a run starts the raw data should be checked to ensure that the monitor is in place and the sample looks sensible. unfortunately It is not encouraged to use the instrument control PC to analyse data so the other PCs in the CRISP cabin should be used.

Looking at Raw Single Detector Data

Within OpenGENIE the following commands should be typed:

> `ass $dae`   *Points the computer at the DAE (current data storage area).*
> `d/h s(1)`   *Displays the spectrum from monitor 1 in histogram mode h.*
> `p/m s(2)`   *Plots on top of previous graph spectrum from monitor 2.*
> `d/h s(3)`   *Displays on a new graph the spectrum from the detector.*

The raw data from monitors 1 and 2 have characteristic shapes as shown in **Figure 2**. The data from the detector varies depending on the sample but should be more intense on just background noise even after a few microamps of measurement.
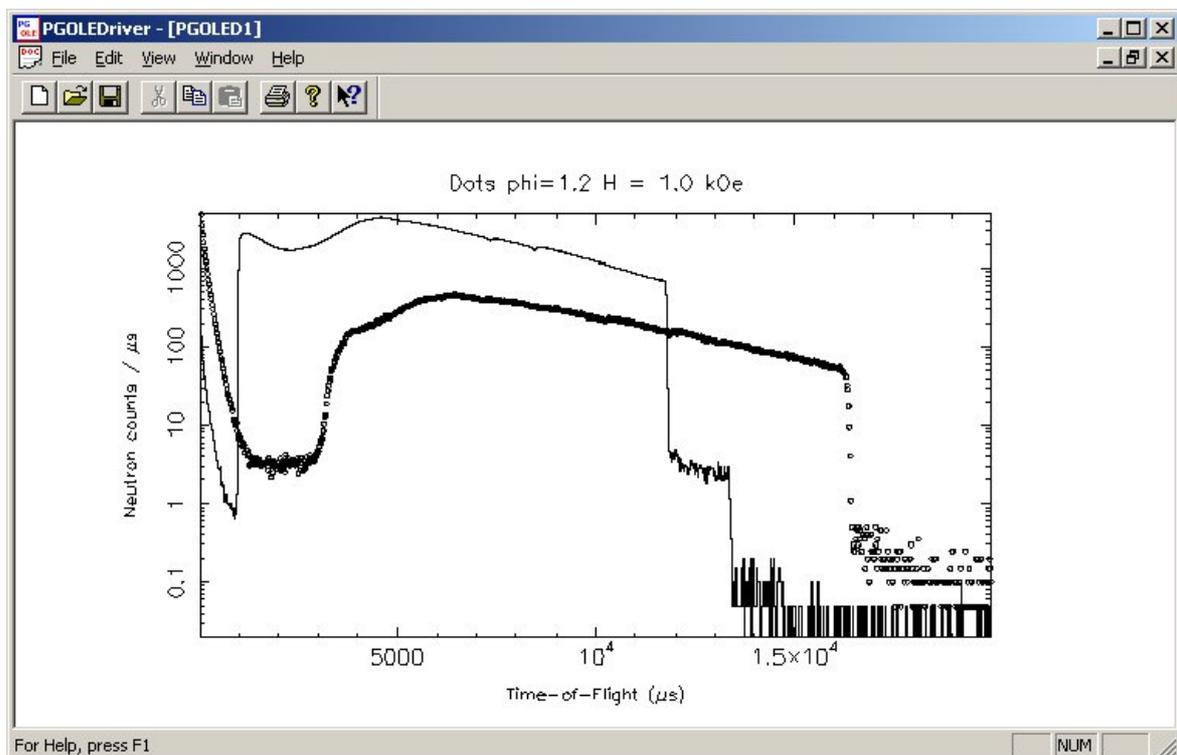
Figure 2: Typical plots of S1 (line) and S2(points) spectra raw data.

Reducing Raw Data to Reflectivity

Reflectivity is simply the ratio of reflected intensity divided by incident intensity as a function of wavelength or more often Q. The reduction programs used on SURF take the TOF data from the incident monitor and the detector data and convert these to reflectivity. The efficiency of the monitor and detector are also taken into account, the ratio performed and the conversion from time to wavelength and hence Q carried out.
Several reduction programs exist to do the reduction:

Table 4.2:

OpenGENIE Command	Description
`quick`	Reduces single detector data, requires input of data location (ie DAE or run number) and angle of detector.
`solid_liquid`	A version of `quick` for solid/liquid samples which uses a transmission file for the attenuation of the beam through the block.

Creating a Transmission File for Solid/Liquid Measurements.

To create a transmission file for the cell, the slits should be set up so that the single helium detector does not saturate e.g. approximately S1 = S2 = S3 = S4 =0.5. Set the detector and goniometer to 0 and move the sample down so that the neutron beam passes above the interface of interest by at least 1mm in order to measure the block transmission.

Two measurements are then required:
Block transmission, *ie* Si
Air transmission, *ie* no cell

Both these data sets are required by the <transmission> program with OpenGENIE that is then used by sol. The following illustrates a typical running of the transmission program

transmission
Enter run number for st tho block ==: 68977
Enter run number for st tho air == : 68978
Informational printing is ON
workspace 'trans' has transmission Vs wavelength.