CRISP Instrument Manual

C.J. Kinane, R.M. Dalgliesh, S. Langridge and D.G. Bucknall

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Chapter 1

Introduction

CRISP is one of several neutron reflectometers at ISIS. It is the original reflectometer built on Target Station 1 (TS1) and designed to be a general purpose reflectometer for investigation of a wide spectrum of interfaces and surfaces. The sample geometry is horizontal and therefore a wide range of samples can be investigated from air-liquid, liquid-solid to solid-solid cases in all disciplines of science. The instrument uses the pulsed neutron beam obtained from ISIS neutron facility and viewing a hydrogen moderator gives a normal neutron wavelength range of 0.5 - 6.5 Å. A large variety of sample environment equipment is available to users. The instrument unlike its sister SURF can also be run in a polarised mode allowing polarised neutron reflectivity (PNR) measurements as well being able to perform full polarisation analysis PNR (PA-PNR). The CRISP instrument is highly automated allowing precision reproducible measurements.

1.1 Location of instrument

The CRISP neutron reflectometer is found on the north side of the target station 2 experimental hall (building R55), between the LOQ small angle scattering instrument and the SURF reflectometer. See Figure 1.1. The instrument blockhouse is found on the ground floor and shares a common entrance area with SURF, with the instrument control cabin on the mezzanine level, immediately above the blockhouse.

1.2 Instrument scientists

The following are the instrument scientists who work on the CRISP instrument. The extension numbers apply to on-site telephones. If phoning from outside site then the full number is +[44] (0) 1235 44 + your extension number. Mobile short codes work within the lab only. For contact outside the lab the full number must be used.
Table 1.1:

<table>
<thead>
<tr>
<th>Person</th>
<th>Office</th>
<th>Mobile</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRISP responsible</td>
<td>Christy Kinane</td>
<td>6043</td>
</tr>
<tr>
<td>Instrument Scientists</td>
<td>John Webster</td>
<td>6116</td>
</tr>
<tr>
<td></td>
<td>Tim Charlton</td>
<td>6088</td>
</tr>
<tr>
<td></td>
<td>Robert Dalgliesh</td>
<td>5687</td>
</tr>
<tr>
<td></td>
<td>Arwel Hughes</td>
<td>6259</td>
</tr>
<tr>
<td></td>
<td>Max Skoda</td>
<td>5206</td>
</tr>
<tr>
<td>Group Leader</td>
<td>Sean Langridge</td>
<td>5269</td>
</tr>
</tbody>
</table>

1.3 Safety in the Experimental Hall

There are many things to remember when within the experimental hall in order to work safely. Many of these are the same safe practices used in any laboratory, but the presence of radiation means that we have several extra procedures which must be followed.

All new and returning users must check in with the user office situated in building R3 G11, before heading to the experimental hall located in building R55. On entering the experimental hall for the first time, it is *essential* that every new user and users returning after a 6 month absence undertakes the ISIS safety training course. This available on the web (http://www.isis.rl.ac.uk/usertraining/). Every user should print out the certificate from the test at the end of the test, this must be shown to the instrument responsible scientist to be allowed to start an experiment and must also be shown at the radiation safety office to obtain access to the hall. In addition, a safety package including all relevant information will be distributed to new users.

As part of the safety system, entrance to the experimental area is via swipe card doors. Every member of the experimental team must swipe their individual
cards when entering and leaving the hall. Cards for this swipe card system must be obtained on arriving at RAL after registration at the Main Control Room (MCR) radiation safety office at the main entrance to the Experimental hall. At the same time a radiation badge must also be collected and worn in a prominent position on your person at all times when in the experimental hall.

If there are any questions regarding any aspect of safety please contact your local contact in the first instance or the Main Control room on extension 6789.
Chapter 2
The CRISP Neutron Reflectometer

Figure 2.1: CRISP instrument.

The essence of a neutron reflection experiment is to measure the specular reflection as a function of the wave vector transfer, $Q$, perpendicular to the reflecting surface. This can be related to the neutron refractive index profile normal to the surface and interface, and is often simply related to the scattering length density. This yields information about the composition and density gradients at surfaces and interfaces.

The CRISP instrument neutron reflectometer uses a broad band neutron time-of-flight (TOF) method for determining the wavelength, $\lambda$ (and hence $Q$), at fixed
angles, $\theta$. A picture of the instrument is shown in Figure 2.1 with the magnet/flow cryostat assembly in place at the sample stage. Much of the instrument is highly automated allowing precision control and high degree of reproducibility.

The instrument views the 20K hydrogen moderator giving it an effective wavelength range of 0.5-6.5 Å at the source frequency of 40 Hz (For TS1 and TS2 simultaneous running) extending up to a maximum of 13 Å if operated at 25 Hz. The incident beam is well collimated by both coarse and adjustable fine collimating slits to give variable beam size and angular divergence, with typical dimensions of 40 mm wide (horizontal direction) and anything up to 10 mm in height (vertical direction). A variable aperture disc chopper defines the wavelength band, and prompt pulse suppression is achieved by a nimonic chopper. Additional frame overlap suppression is provided by the nickel coated silicon wafer frame overlap mirrors, which reflect out of the main beam wavelengths greater than 13 Å. The instrument has two types of detectors, a $^3$He single detector and a 1-D position sensitive multidetector. A schematic of the complete layout showing all the major components of the beamline is shown in figure 2.2.

The experimental arrangement is extremely flexible with the neutron beam inclined at 1.5° to the horizontal, which provides easy study of liquid surfaces. A schematic of the beamline in non-polarised mode is shown in Figure 2.3. For liquid surfaces angles less than 1.5° are achieved by insertion of a supermirror. The sample position is designed to be vibrationally isolated from the rest of the instrument and further aided by active anti-vibrational damping. Solid films and solid-liquid samples can be studied with ease, by use of a 2 arc, height adjustable sample position.

The CRISP reflectometer can easily be converted to a polarised neutron mode for the study of magnetic systems, a schematic of which is shown in Figure 2.5. This involves in the simplest case the use of a polarising mirror in a static field, a spin flipper and a static guide field. The sample is positioned either directly onto a goniometer or between the poles of a Magnet assembly capable of holding an Oxford instruments flow cryostat. Full polarisation analysis is possible by inclusion of a post sample analysing mirror and appropriate guide field.

A summary of the instrument is shown below:

Most of the sample environment equipment and beamline components are controlled via a PC based Labview system called SECI (Sample Environment Control Interface). A typical display from SECI is shown in Figure 2.4.

Movement of any motion is achieved either by manually inputting the required value within the Labview control and the clicking on the "Set" and "Move" buttons in sequence or by command line input via the OpenGenie window. The use of OpenGenie also allows for the use of scripts.
Figure 2.2: CRISP instrument.
Incident Wavelength | 0.5-6.5Å at 50 Hz  
|---------------------|------------------
|                     | (0.5-13.0 Å at 25 Hz)  
Optimal Q range | 0.005 - 1.1Å\(^{-1}\)  
Moderator | 20K \(H_2\)  
Maximum beam size | 40mm (H) \(\times\) 10mm (V)  
Detectors | \(^3\)He single detector (SD)  
| position sensitive multidetector (MD)  
Data acquisition | Custom made data acquisition electronics  
Moderator-sample distance | 10.25 m  
Sample-Detector distance | 1.87 m  
Choppers | Variable aperture disc chopper  
| Nimonic chopper  

Table 2.1:

2.1 Polarised Neutron Reflectivity (PNR) mode

To perform polarised neutron reflectivity measurements the reflectometer can be easily re-configured to give a polarised incident neutron beam and, if required, full polarisation analysis of the outgoing neutrons.

2.1.1 Converting to polarised operation

Assuming that the reflectometer is in the non-polarised mode then the polarised option should be checked in the motion control LabView panel on SECI. Clicking the polarised option and then the “Set” and “Move” buttons should move the polariser into the beam and likewise clicking the non-polarised option and then "Set" and "Move" should move the polariser out of the beam. To get the best polarisation set the "supermirror angle" axis to 0.4° (See Figure 2.4), this reduces the wavelength range to 1.5-6.5Å.

A schematic of the experimental set-up is shown in Figure 2.5 for full polarisation analysis. (For no polarisation analysis the supermirror analyser is removed). Your local contact will install the sample environment options you require (magnet, cryostat etc.), the guide fields and the analysis kit. The polarising mirror (PM1) and the spin flipper (SF1) are permanently installed but may be translated out of the beam in the case of PM1.

Before taking data there are several checks to be made to ensure that the polariser and spin flipper are correctly set-up:

1. Horizontal slits S1, S2, S3 and S4 are set to 40, 30, 30 and 40 mm respectively for liquid experiments and 30, 20, 20, 30 mm for solid state samples where the sample area is small.

2. Guide field is in the same direction as the field at the sample position. You
can only measure PNR with the magnet field aligned parallel to the guide field.

3. The power supply to SF1 (and the analysing mirror if installed) are switched on. Do not adjust these supplies.

4. Verify with your local contact that the DAE is set-up for multi-period data acquisition.

2.2 Sample Environment Equipment

The CRISP instrument has a standard set of sample environment equipment. Most of these are common to both the CRISP and SURF reflectometers except the polarised neutron equipment which are only available to the CRISP instrument. Most of these have been built in house or been bought specifically for the use on the beam lines. In addition, a number of special pieces of equipment built by university teams have also been used. A brief summary of the key pieces of sample environment equipment is given below, users should contact Christy Kinane or their named local contact if they are unsure which is most applicable to their experiment. In some circumstances, especially with new types of experiments special equipment may have to be developed which should be done in close association with the instrument scientists. This is to avoid safety issues and to allow for the best possible outcome of the proposed experiment. This section is not intended to be completely comprehensive, but to give an overview of what is available off the shelf.
Figure 2.4: Typical motion control screen display with genie window, motion control Vi and status bar.
2.2.1 Standard Goniometer Configuration

The standard normal reflection mode of the reflectometer has a 2 arc goniometer stack (φ and ψ motions) on top of a linear translation stage which is all mounted on a height table. The motions except the height table are standard Micro-control equipment. A drawing of the layout of the sample area configuration is shown in Figure 2.6. For liquid samples the goniometer stack is removed and an active anti-vibration table fitted.

The key parameters and load capacities of the sample position is listed below:

<table>
<thead>
<tr>
<th>Motion</th>
<th>Range</th>
<th>Resolution</th>
<th>Load capacity</th>
</tr>
</thead>
<tbody>
<tr>
<td>φ motion</td>
<td>± 5°</td>
<td>0.001°</td>
<td>12 kg</td>
</tr>
<tr>
<td>ψ motion</td>
<td>± 5°</td>
<td>0.001°</td>
<td>20 kg</td>
</tr>
<tr>
<td>Linear translator</td>
<td>600 mm</td>
<td>&lt;0.05 mm</td>
<td>120 kg</td>
</tr>
<tr>
<td>Height table</td>
<td>100 mm</td>
<td>&lt;0.05 mm</td>
<td>100 kg ‡</td>
</tr>
<tr>
<td>Anti-vibration table</td>
<td>n/a</td>
<td>n/a</td>
<td>150 kg</td>
</tr>
</tbody>
</table>

Table 2.2: ‡This is on top of the standard environment equipment.

The minimum distance from height table surface to beam centre is 236 mm and the maximum is 341 mm. If the translator is added these figures are modified to 121 mm (min) to 226 mm (max).

Sample mounting on the goniometer is via a kinematic plate. If users wish their equipment to fit onto these plates then they should contact Christy Kinane for full design drawings.
2.2.2 Liquid troughs

The multi-sample liquid troughs has the capacity for 5 liquid samples in individually sealed containers. Each unit contains a PTFE trough and is heated via heating mats. The units have the capability to be sealed to prevent evaporation of even volatile samples. Almost all of the construction is made from aluminium with quartz windows to allow laser alignment of the samples. All four units are also held within a temperature controlled box to reduce condensation effects. The whole unit mounts directly onto the active anti-vibration table which is itself mounted to the linear translator. This allows the possibility of measuring all five positions remotely under computer control. The summary of the liquid troughs is given below:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell temperature range</td>
<td>RT - 80° C</td>
</tr>
<tr>
<td>Outer unit temperature range</td>
<td>RT - 80° C</td>
</tr>
<tr>
<td>Sample volume (approx)</td>
<td>70 ml</td>
</tr>
</tbody>
</table>

Table 2.3:

The sample volume is assuming use of a water subphase which gives a good
meniscus which is a few millimeters higher than the lip of the PTFE trough.

2.2.3 Nima Langmuir Troughs

There are two Nima Langmuir troughs available for use on CRISP and SURF. Both have double moveable barriers allowing the measurement of reflectivity curves in-situ over a very wide area enclosed by the barriers. Both troughs are computer controlled with a user friendly interface allowing flexibility of use.

A majority of experiments will need to be carried out holding a fixed pressure over the length of the reflectivity measurement and this can easily be achieved as well as measuring more normal pressure-area curves. Full details of the use of the troughs can be found in the very helpful Nima handbook. The large Nima trough has moveable fixed barriers which allow the volume of subphase to be varied. The troughs both have perspex lids which incorporate neutron transparent quartz windows. This arrangement allows easy alignment of the liquid but prevents a majority of the airborne dust contaminants. This is not however a sealed unit and is therefore not vapour pressure tight. The troughs both have the possibility to link to a recirculating heater/cooling bath which has a maximum temperature range of -50 to 120°C. However, temperatures above 100°C are not recommended for the PTFE.

Care must be taken to ensure that the troughs are set up correctly and it is advised that users contact Christy Kinane or Luke Clifton in the ISIS Biolab before using the equipment. Calibration of the area enclosed by the barriers will be done before users will have access to the troughs although calibration of the pressure head will be the responsibility of the individual users. Again contact Christy Kinane, or the appropriate local contact if there are any problems.

A summary of the key features of the Nima troughs follows:

2.2.4 Solid Sample Changer

The four position sample holder mounts directly onto the standard goniometer stack will take samples which are typically silicon or quartz substrates, ranging from 10 to 100 mm diameter. Two modes of operation can be achieved by changing
the sample holding inserts. One insert allows for 4 independent temperatures on the 4 sample positions via heater cartridges giving temperature from RT to 350°C. The other insert is temperature controlled by connection to a recirculating bath giving a temperature range from -50 to 200°C. This cannot be achieved by each sample independently since the circulating fluid runs underneath all the samples. The samples are housed in a metal box with quartz windows allowing for laser alignment with a perspex lid. The unit is not sealable but allows for inert gas to be passed through it under a positive pressure (the unit does not allow the samples to be placed under vacuum).

### 2.2.5 Newport Electro-magnet

The sample stage accepts an electro-magnet which can be used to provide a continuous magnetic flux density up to 0.4T.

The magnet has the capability to hold an Oxford instruments He flow type cryostat (see section 2.2.6). The main characteristics of the magnet with and
Figure 2.8: The Solid Sample Changer

Figure 2.9: The Newport Magnet

Figure 2.10: The Oxford Flow Cryostat
without cryostat are detailed in table below.

<table>
<thead>
<tr>
<th>Magnet type</th>
<th>Max field at 20mm gap</th>
<th>90mm gap</th>
<th>Field direction *</th>
</tr>
</thead>
<tbody>
<tr>
<td>Newport</td>
<td>1.0 T</td>
<td>0.4 T</td>
<td>Longitudinal</td>
</tr>
<tr>
<td>Newport with cryostat</td>
<td>——</td>
<td>0.35 T</td>
<td>Longitudinal</td>
</tr>
</tbody>
</table>

Table 2.8: *Relative to the neutron beam direction.

The installation of the magnet is a complicated process and should not be undertaken by external users. If requested on the application form the magnet will be installed by your local contact at the beginning of the experimental run. Please discuss your magnetic field requirements with your local contact in advance of any experiment.

Measuring the magnetic field

A ”Hirst” teslameter is available in the CRISP cabin to measure the magnetic field strength of the magnet and the neutron guide fields. The hand held unit is simple to operate although care should be taken to ensure the correct Hall probe is used to measure either the longitudinal or transverse magnetic field.

Safety and the Electromagnet

The Electromagnets will be installed at the beginning of your experiment by the local contact but there are several safety issues which you should be familiar with before operating the magnet:

- The presence of an input voltage to the power supply is indicated by the ”Magnet On” sign in the blockhouse. If illuminated then there is the possibility that the magnet is energised and users must ascertain if the magnet is actually powered (measure the field or check the power supply).

- The power supply can be immediately shut off by tripping the interrupter switch which is clearly marked on the CRISP blockhouse wall.

- The electrical connection for the magnet is located in the CRISP blockhouse. It is essential that if the magnet needs to be connected/disconnected then the power must be shut-off either at the power supply or by tripping the interrupter switch.

- Cooling water must flow through the magnet at all times, even at low operating currents. When energised care should be taken to avoid introducing magnetic materials (e.g. watches, screwdrivers) near to the pole pieces.
2.2.6 Cryostats

Several cryostats are available on CRISP to provide operating temperature down to $\mu$1K. Only the Oxford Flow cryostat can be mounted in the Newport magnet in order to obtain fields of up to $\sim 5$ kOe. Temperatures less then 2k can be obtained by the use of a Heliox or dilution fridge. This however requires extra setup to be performed by the Cryogenics group.

**Oxford Flow Cryostat.**

Temperatures of 2.5K can be achieved rapidly ($\sim 2$ hrs) by the use of the Oxford Instruments Continuous Flow Cryostat. The need to have a helium dewar attached to the cryostat at all times means that it is especially important that users liaise with their local contact if they wish to make use of this equipment. Only one sample may be mounted at a time but rapid turn over may be achieved because of the fast cool down speed. The sample changing procedure is relatively simple but the correct procedures must be followed. Users should contact their local contact if they are unsure the method of correct operation. Samples may be up to 2.5cm x 2.5cm and in preference should have a surface area of the order of 1cm$^2$. In order to prevent slipping from the sample holder it is necessary to apply a small amount of vacuum grease to the bottom the sample. Control of temperature is best achieved by manual operation of the helium flow with automated heating. This method provides temperature stability to the order of $<0.1$K.

![Figure 2.11: The Oxford Flow Cryostat](image)
Chapter 3

Running the Instrument

3.1 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.

3.1.1 Laser alignment

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

<table>
<thead>
<tr>
<th>Motion</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>1.0 mm</td>
</tr>
<tr>
<td>S2</td>
<td>0.5 mm</td>
</tr>
<tr>
<td>S3</td>
<td>2.0 mm</td>
</tr>
<tr>
<td>S4</td>
<td>2.0 mm</td>
</tr>
<tr>
<td>THETA ($\theta$ = detector angle)</td>
<td>0.6°</td>
</tr>
</tbody>
</table>

Table 3.1:

1. Ensure that PHI ($\phi$ - 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 clicking on the Define OffSets button on the screen and entering the new value that you want phi to take then clicking apply. For example phi would normally be set so that Phi=Theta.

If using liquid surfaces (without the supermirror) the detector angle should be set to $1.5^\circ$. 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.

<table>
<thead>
<tr>
<th>Motion</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>4.0 mm</td>
</tr>
<tr>
<td>S2</td>
<td>2.5 mm</td>
</tr>
<tr>
<td>S3</td>
<td>3.0 mm</td>
</tr>
<tr>
<td>S4</td>
<td>4.0 mm</td>
</tr>
<tr>
<td>THETA ($\theta$ - detector angle)</td>
<td>$1.5^\circ$</td>
</tr>
</tbody>
</table>

Table 3.2: Liquid Surface Parameters

If using the magnet then these motions may also be used in the same way. The cryostat’s have both height and angle adjustments ($\phi$). In both cases your local contact will be able to demonstrate the use of this equipment. However, the method of alignment is the same:

1. Ensure the sample is horizontal, orthogonal to the beam direction.

2. Adjust the $\phi$ angle of the goniometer so that the sample surface is approximately parallel to the laser beam.

3. Adjust the height of the sample so that the laser beam height is half its original value. This can be measured by placing a ruler in front of S3.

4. Iterate between 2 and 3 to give a sample half-cutting and parallel to the beam.

5. Set the detector to the angle of interest, e.g. $0.6^\circ$ and adjust the $\phi$ angle so that the laser beam is aligned in the detector aperture and is symmetrically cut by slits S4.
Further iteration of the height and $\phi$ adjustments may be necessary to optimise
the reflected intensity. It is worth noting that at small $\phi$ the height adjustment is
well defined and conversely at large $\phi$ the alignment is relatively insensitive to the
height but the $\phi$ angle is well defined.

**Aligning the polarisation analyser**

The polarisation analyser is mounted on a second stage which has been installed
on the post-sample section of the instrument. Alignment of the second polariser is
made with an aligned sample and the reflected laser beam. In principle should you
require PA-PNR then your local contact will ensure that the analyser is installed
and aligned.

### 3.1.2 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>
<thead>
<tr>
<th>Motion</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>1.0 mm</td>
</tr>
<tr>
<td>S2</td>
<td>0.5 mm</td>
</tr>
<tr>
<td>S3</td>
<td>2.0 mm</td>
</tr>
<tr>
<td>S4</td>
<td>2.0 mm</td>
</tr>
<tr>
<td>THETA</td>
<td>0.0°</td>
</tr>
<tr>
<td>PHI</td>
<td>0.0°</td>
</tr>
</tbody>
</table>

Table 3.3: Neutronic alignment starting parameters.

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 $\text{THETA} = \text{PHI} = 0.0^\circ$ 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 $\text{THETA} = 0.0^\circ$ and $\text{HEIGHT}$ at half height Determines
   the zero position of the PHI motion on a slightly coarse scale.

3. PHI alignment at $\text{THETA} = 0.25^\circ$ 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=1μA) per step must be entered. 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 or xy 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. you must click in the OpenGENIE graphics window in-order for this to work.

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>
<thead>
<tr>
<th>Scan</th>
<th>Motion</th>
<th>Start Value</th>
<th>End Value</th>
<th>No. of Steps</th>
</tr>
</thead>
<tbody>
<tr>
<td>Height</td>
<td>HEIGHT</td>
<td>-0.5</td>
<td>0.5</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>PHI</td>
<td>-0.5</td>
<td>0.5</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>THETA</td>
<td>= 0.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Angle</td>
<td>PHI</td>
<td>-0.5</td>
<td>0.5</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>THETA</td>
<td>= 0.25</td>
<td>0.3</td>
<td>11</td>
</tr>
</tbody>
</table>

Table 3.4: Neutronic alignment scan starting parameters.

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 using the define offsets button 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.

3.2 Dash Board Commands

A typical dash board display is shown below (see Figure 3.1). This screen shows
the current status of the instrument. If this control is not present on the con-
trol machine double click on the SECI icon on the desktop to launch the SECI
instrument control program.

![Figure 3.1: A Typical dashboard display.](image)

Commands for manual running of the instrument should only be submitted
onto the dash board instrument control computer. Through an OpenGENIE win-
dow. 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 3.5
<table>
<thead>
<tr>
<th>Command</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BEGIN</td>
<td>Starts data collection, data is held in the DAE. The dash board will indicate RUNNING above the header block.</td>
</tr>
<tr>
<td>END</td>
<td>Ends the data collection and saves the data to file for archiving into a file CSP{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.</td>
</tr>
<tr>
<td>PAUSE</td>
<td>Pauses the data collection, the dash board will indicate a PAUSE mode. N.b this can cause scripts to finish prematurely.</td>
</tr>
<tr>
<td>RESUME</td>
<td>Resumes the data collection after the PAUSE statement has been issued and will return to the RUNNING mode.</td>
</tr>
<tr>
<td>ABORT</td>
<td>Ends the data run without saving the data. The DAE goes into SET-UP mode but the run number is not incremented.</td>
</tr>
<tr>
<td>mv (\text{theta, phi, height, s1, s2, s3, s4, sm_angle})</td>
<td>A command which must be used to move any of the instrument axes. It should be given in the following format: \text{mv}{\text{motion}} {\text{position}} where {\text{motion}} is the motion to move and {\text{position}} is the value to move to. An example is: \text{mv} \text{THETA} 0.5 which moves the detector to 0.5°.</td>
</tr>
<tr>
<td>POL_RUN</td>
<td>(u={X1}) (d={X2}) A command file for normal PNR running which begins a run and counts for {Xi} frames on each spin state before switching to the other spin state. Typically (X1=X2=5000) Since the command file does not contain any slit control information the beam line must be set up as required before running. This command file will continue until manually ended by {CTRL} C followed by END or ABORT.</td>
</tr>
<tr>
<td>POL_RUN uu={X1} ud={X2} du={X3} dd={X4}</td>
<td>A version of the POL_RUN which runs the full polarisation analysis control. Typically (X1=x2=x3=x4=5000)</td>
</tr>
<tr>
<td>do &quot;filename.gcl&quot;</td>
<td>Runs a command file with the name filename.gcl which must be in the current directory that contains a procedure called runscript (See below)</td>
</tr>
<tr>
<td>UPDATE</td>
<td>Transfers the DAE data into the CRPT memory on the PC system.</td>
</tr>
<tr>
<td>STORE</td>
<td>Writes the contents of the CRPT into a temporary file with a {run number}.SAV file extension.</td>
</tr>
<tr>
<td>load &quot;filename.gcl&quot;</td>
<td>loads a script file into OpenGENIE. This should be used if you wish to use a script with a procedure name other than runscript (See Section 3.2.1 for further details.)</td>
</tr>
</tbody>
</table>

Table 3.5:
3.2.1 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 an non polarised measurement 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:

```plaintext
LOOP
.
.
.
.
.
ENDLOOP
```

Loops may be added using makescript. An example of a polarised script is given below:

```plaintext
PROCEDURE Ni58
  # Script Generate by MakeScript
  # Setting the number of periods in the DAE
  single_detector periods=2
  change nperiods=2
  mv height=0.0
  waitformove
  LOOP
  mv s1=0.74 s2=0.35 s3=2.000 s4=2.000
  waitformove
  mv theta=0.4 phi=0.4
  waitformove
  change title="Ni58 th=0.4 dQ/Q=3% I=0 amps"
  pol
  run u=5000 d=5000 total=40000
  mv s1=1.48 s2=0.7 s3=3.000 s4=3.000
  waitformove
  mv theta=0.800 phi=0.800
  waitformove
  change title="Ni58 th=0.8 dQ/Q=3% I=0 amps"
  pol
  run u=5000 d=5000 total=180000
  ENDLOOP
ENDPROCEDURE
```

### 3.3 Running in Polarised Neutron (PNR) Mode

#### 3.3.1 Operating the Newport magnet power supply

The Newport magnet is powered by a "Danfysik Systems 7000 bi-polar" dc power supply. The power supply is located on top of the CRISP blockhouse roof. Your
local contact should set up the magnet for you prior to your experiment. However, please ensure that the followings steps are checked.

1. Check that the magnet is connected and that the cooling water is flowing.

2. Check that the emergency off button in the CRISP blockhouse on the right hand wall is out. (Ask your local contact to point this out if you are not sure)

3. Power the unit by switching the 3 phase power switch behind the cabinet to on.

4. Ensure that all faults on the front panel are cleared by pressing the RESET button.

5. Turn the unit on using the front panel ON button.

6. Increase the current using the CRISP instrument labview control. (If necessary the from panel set controls may be used but this is not recommended)

7. In case of emergency please press the magnet emergency stop button to shut off power to the magnet.

It is essential that the magnet is not operated above 10A. This will cause excessive heating in the magnet and may ultimately damage the magnet or power supply. The Danfysik power supply is not capable of supplying more than 10A.

![Figure 3.2: A photo of the Danfysik 7000 bi-polar supply front panel](image)

### 3.3.2 The Oxford Flow Cryostat

Temperatures of 4K can be achieved rapidly (2 hrs) by the use of the Oxford Instruments Continuous Flow Cryostat. The need to have a helium dewar attached to the cryostat at all times means that it is especially important that users liaise with their local contact if they wish to make use of this equipment.

Only one sample may be mounted at a time but rapid turn over may be achieved because of the fast cool down speed. The sample changing procedure is relatively simple but the correct procedures must be followed. Users should contact their local contact if they are unsure the method of correct operation.
Samples may be up to 2.5cm x 2.5cm and in preference should have a surface area of the order of 1cm$^2$. In order to prevent slipping from the sample holder it is necessary to apply a small amount of vacuum grease to the bottom the sample.

Control of temperature is best achieved by manual operation of the helium flow with automated heating. This method provides temperature stability to the order of <0.1K.
Chapter 4

Looking at your data

4.1 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

It should be noted that ISIS is in the middle of a conversion to the new MAN-TID Data analysis software.

Which can be found at:

http://www.mantidproject.org/Main_Page
4.2 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.

4.2.1 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 as a histogram.

`>> p/m s(2)` Plots on top of previous graph spectrum from monitor 2 using Markers.

`>> d/l s(3)` Displays on a new graph the spectrum from the detector as a line plot.

The raw data from monitors 1 and 2 have characteristic shapes as shown in Figure 4.1. 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. Your local contact will advise you.

![Figure 4.1: Typical plots of S1 (line) and S2(points) spectra raw data.](image)

When in polarised mode there are either two or four periods, one for each spin...
state depending whether PA is used. The data from all the periods are all held in spectrum within one specific run number. The spectra number for the PNR modes are shown below:

<table>
<thead>
<tr>
<th>Monitor</th>
<th>Normal</th>
<th>PA-PNR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monitor 1</td>
<td>S1 &amp; S6</td>
<td>S1, S6, S11, S16</td>
</tr>
<tr>
<td>Monitor 2</td>
<td>S2 &amp; S7</td>
<td>S2, S7, S12, S17</td>
</tr>
<tr>
<td>Detector</td>
<td>S3 &amp; S8</td>
<td>S3, S8, S13, S18</td>
</tr>
</tbody>
</table>

Table 4.1:

### 4.2.2 Looking at Raw Linear detector Data

In order to look at data from the CRISP linear detector a series of Matlab routines have been written that enable visualisation in both "raw" wavelength vs. Angle form or $Q_x$-$Q_z$ maps.

If you need to use the linear detector please consult your local contact for further instructions in how to use the software.

### 4.3 Reducing Raw Data to Reflectivity

Reflectivity is simply the ratio of reflected intensity divided by incident intensity ($I/I_0$) as a function of wavelength or more often $Q$. The reduction programs used on CRISP 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 depending on the detector used and whether using non-polarised or polarised beams. If in doubt just type the reduction command into the Genie window and follow the instructions displayed on screen:
<table>
<thead>
<tr>
<th>OpenGENIE Command</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>QUICK</td>
<td>Reduces single detector data, requires input of data location (ie DAE or run number) and angle of detector.</td>
</tr>
<tr>
<td>SOL</td>
<td>A version of QUICK for samples which uses a transmission file, ie in solid/liquid experiments, see section 4.3.3</td>
</tr>
<tr>
<td>POL</td>
<td>Reduces polarised neutron data giving the spin up and down in separate workspaces.</td>
</tr>
<tr>
<td>PA2</td>
<td>Version of POL for full polarisation analysis, with 4 different spin states.</td>
</tr>
</tbody>
</table>

Table 4.2:

4.3.1 Reducing non-polarised data to an ascii xye file

An example of a QUICK routine for reducing 3 angles is shown below, this is for non polarised data only:

```plaintext
# Load and reduce data sets

quick
3
1
72736 # Run number (0 for the dae)
0.13 # Angle
d/l wq 0.15 1e-7 0.1 # Plot data with scale defined.
p/e # show error bars
ww1=wq # Save the output to another variable

quick
3
1
72737
0.2
p1/l wq $blue
p1/e wq $blue
ww2=wq

quick
3
1
```

32
72748
0.55
p1/l wq $cyan
p1/e wq $cyan
ww3=wq

# Rebin and stitch data together
rebin ww1 ww1.x[2] 0.0085 # rebin data
rebin ww2 ww2.x[2] 0.013
rebin ww3 ww3.x[2] 0.047

wcomb1=stitch:l:v(ww1,ww2,ww3) 0.02 # Stitch the data sets together

# Normalised Crit edge to 1
wcomb1=wcomb1/max(wcomb1.y)
t/logx # Toggle the logarithmic x axis on or off.
d/l wcomb1
p/e

# Write data to a file
writexye wcomb1 "filename.dat"

4.3.2 Reducing polarised data to ascii xye files
An example of a reduction script for polarised neutron data is also given below along with how to calculate the spin asymmetry:

# Load and reduce data sets
pol
3
2
84931
84932
0.4
7
polplot
wpol
ww1=wpol
pol
3
2
84933
84934
0.81
7
polplot wpol
ww2=wpol

pol
3
5
84935
84936
84937
84938
84939
1.7
7
polplot wpol
ww3=wpol

# Rebin the polarised data
rebinpoldata ww1 ww1.u.x[2] 0.045
rebinpoldata ww2 ww2.u.x[2] 0.08
rebinpoldata ww3 ww3.u.x[2] 0.14
polcomb ww1 ww2 ww3
1
0.0005

# Normalised Crit edge to 1
wcomb.u=wcomb.u/max(wcomb.u.y)
wcomb.d=wcomb.d/max(wcomb.d.y)
polplot wcomb

# Calculate the spin asymmetry
asym=((wcomb.u-wcomb.d)/(wcomb.u+wcomb.d))
t/logy/off
d/l asym - -1 1 p/e t/logy/on

# Save the two spin states and spin asymmetry as ascii files
writexye wcomb.u ”filename_up.dat”
writexye wcomb.d ”filename_down.dat”
4.3.3 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 $S_1 = S_2 = s_3 = s_4 = 0.5$. Set the detector and $\phi$ goniometer to $0^\circ$ 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 \( \text{transmission} \) program with OpenGENIE that is then used by sol. The following illustrates a typical running of the transmission program

$$\text{transmission}$$

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

4.3.4 Rebinning Data

The reduced data can be tidied up and truncated using the rebin command. This has two modes of use:
- either
  $$>> \text{REBIN } \log Wx \{qmin\} \{step\} \{qmax\}$$
  or
  $$>> \text{REBIN } Wx \{qmin\} \{qmax\}$$
- In both cases Wx is the workspace where the data is held. \{qmin\} and \{qmax\} are the minimum and maximum Q values for truncating the data and \{step\} the percentage of Q data values for combining. Typical examples are:
  $$\text{REBIN } /\log W10 0.008 0.02 0.06$$
  or
  $$\text{REBIN W10 0.008 0.06}$$
In both cases the data from workspace w10 is taken from Q=0.008 - 0.06 and rebinned. In the first case with a 2% rebin and in the second case without altering the data set within these limits. When 2 or more data sets have been collected the Q range must be chosen so that there is overlap between them.

4.3.5 Combining Data Sets

Once the rebinning process has been completed the data sets must be adjusted so that each overlaps exactly the others. This is achieved (if necessary) by a simple Y-axis translation, ie

$$w10 = w10 \times 1.5$$

Once all the scaling has been carried out the data sets can be combined using COMB. This program takes the data in the workspaces and adds them into a single workspace. It will also autoscale data for you.

$$\text{COMB } w1 \text{ w2 w3}$$

The workspace w1,w2 and w3 should be the workspaces to be combined in ascending order of incident angle. A typical comb session is shown below:

$$\text{comb w405 w406 w407}$$

Found ".COMBINE" -- > ".COMBINE@8" in function cache of "combine_g3.so"

MODULE: beginning execution of "combine" from "combine_g3.so"

DATA READ IN xmin step xmax
Q range 1 0.1179E-01 steps in dx/x 0.1223E-02 0.4994E-01
Q range 2 0.2358E-01 steps in dx/x 0.1223E-02 0.7970E-01
Q range 3 0.4043E-01 steps in dx/x 0.1223E-02 0.4767E+00

Step in x (1) or dx/x (2) : 2

Enter the new step length : 0.03

No. of output bins will be 126

Is this likely to overfill the workspace?

Select new step length ? y or < CR >n

scale factor for data set 2 is 1.037251
scale factor for data set 3 is 1.070218
Scale data to last data set rather than first ? y or < CR >n
Comb asks a number of questions to the first always answer 2 if unsure. The second determines the bin size of the final data set in the same way as a rebin command. The 0.03 above results in a 3% bin size. The final two questions are checks. Firstly comb checks if you are happy with your selected bin size then it asks if you wish to scale the data to the first or last data sets. In the case of liquid-air interface experiments where the absolute scaling of the reflectivity is determined by fitting a D$_2$O data set answer yes here otherwise answer no.

It should also be noted that the stitch command can also be used. This does all of the above but also scales the data for you: wcomb=stitch:l:v(ww1,ww2,ww3)

The stitch command will ask all the same questions as the comb command.

4.4 Putting Data on an Absolute Scale

The reduced reflectivity data obtained from these reduction procedures is not on an absolute reflectivity scale. There are essentially 3 methods of achieving this and depend on the sample being measured.

- **If a total reflectivity region exists** By definition the total reflection region has unit reflectivity. The data is therefore simply scaled so this region is on $R = 1$.

- **If using a solid/liquid cell** In obtaining a cell transmission file (see section 4.3.3), the air transmission is obtained. The high l asymptotic value of $R$ obtained from the output of QUICK of this air transmission measurement will be the scale factor required to put the output of ”SOL” (including the transmission file reduction) on an absolute scale. This can of course be checked if a total reduction region is observed.

- **If using a air/liquids** Although the total reflection region can be observed for many liquids, in cases where one does not exist and/or where measurements are performed well away from this region a D2O calibration is required. The scale factor obtained to a fit to a D2O run will put all data onto an absolute scale. It if therefore essential to collect a good D2O calibration run at the beginning of the experiment.

4.5 Writing data to file

Since the data used within GENIE is in binary format it can be saved easily as binary format using the command.

```bash
>>> w Wx {filename}.Q
```
To convert to ascii and save to file the following commands should be used.

```
>> ascii_out Wx
Enter output file name==> {filename}.asc
Value of xmin for output==> {qmin}
Value of xmax for output==> {qmax}
Data written to ASCII file >>
```

This program writes data to file and saves as an ascii format. Files created in this way may be read back into a Genie II workspace using the command:

```
>> ascii_in {filename}.asc
```

Further details of running any of these routines can be obtained from your local contact.

To save workspaces and variables directly to an ascii format the writexye command can be used an example of which is given below:

```
writexye wcomb "filename.dat"
```

Where wcomb is the final workspace to be saved containing the data. This will produce and xye data file.
Chapter 5

Troubleshooting Problems

Before asking your local contact for help, it may be useful to check the list of problems below which are some of the most commonly occurring.

5.1 No neutrons

- Is the shutter open? Check shutter control box, open shutter if closed.
- Are the choppers running? Check chopper control box in inner part of cabin, restart chopper at cortina if necessary.
- Is monitor 2 in place? Check spectrum, drive monitor 2 into running position.
- Is main neutron beam on? Check beam on displays in ISIS hall.

5.2 Instrument Control PC Problems

- No dashboard Run SECI from the icon on the desktop
- Is the ”CRISP - User front Panel” window shown and running. Restart SECI and then run the front panel by clicking the arrow in the top left hand corner of the window.
- Motions have reset to all zero? Instrument needs resetting, seek assistance.
- Windows are hidden behind the dashboard. Check and uncheck the ”Keep on top” check box in the dashboard.
5.3 OpenGENIE Problems

- OpenGENIE has locked up. Close the window and open a new session using the icon on the start menu or quick launch menu.

- OpenGENIE has crashed. Start another window using the icon on the start menu or quick launch menu.