



Technical Report
RAL-TR-97-038

User Guide for the Polaris Powder Diffractometer at ISIS

R I Smith and S Hull

September 1997

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ISSN 1358-6254

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POWDER DIFFRACTION AT ISIS

USER GUIDE FOR THE POLARIS POWDER DIFFRACTOMETER AT ISIS

R.I. Smith and S. Hull

Version 3.0

July 1997

Preface To Version 3.0.

Version 3.0 of the Polaris User Guide has been produced to coincide with the move of the instrument onto ISIS beamline N7. The new instrument has been built to an almost identical specification as the old, however the ability to start from scratch on a new beamline has allowed certain aspects of Polaris to be optimised.

Polaris on beamline N7 retains its four discrete detector banks, situated at backscattering angles, 90 degrees, low angles and very low angles. During the instrument move the Polaris detector complement was increased, with 20 ^3He tubes added to the backscattering detector bank and a further two ZnS scintillator modules added to the 90 degrees detector bank. This has approximately doubled the count rate of the backscattering bank, whereas in the 90 degrees bank the detectors have been moved further from the sample resulting in an improvement in resolution while maintaining the count rate. Finally, the geometry of the four ZnS scintillator detector modules in the very low angle bank was altered slightly to improve the performance of this bank.

The most significant change in the new Polaris, however, is the redesign of the instrument blockhouse. Access to the sample position is now at floor level, and no longer requires entry into the blockhouse. With only a small enclosure built around the sample position sample environment equipment, such as furnaces and cryostats, can be operated in a much more convenient and flexible manner.

May 1997.

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1. INTRODUCTION

1.1 Overview

The POLARIS instrument at ISIS is a high intensity, medium resolution powder diffractometer. The intense neutron flux on POLARIS combined with a large detector solid angle provides a high count rate, allowing experiments to be performed with short counting times or with very little sample. This has been exploited during kinetic experiments, e.g. studies of chemical reactions or phase transitions, where diffraction patterns are collected while a furnace or cryostat is heating or cooling the sample, and in diffraction from very small sample volumes, down to $\sim 1\text{mm}^3$. The detector banks at $2\theta \sim 90^\circ$ are particularly important for studies of samples contained in complex environments, such as under high pressures or in reaction vessels. By providing suitable collimation of the incident and scattered beams, contamination of the diffraction pattern from the sample with signals from the surrounding sample environment apparatus can be eliminated.

This User Guide gives a short description of the POLARIS diffractometer and provides the basic information required to perform a powder diffraction experiment. To keep the guide as brief as possible the text contains several references to appropriate manuals where more detailed information may be found.

1.2 Instrument Design

POLARIS is located on ISIS beamline N7 and receives a polychromatic, 'white', beam of neutrons from the ambient temperature water moderator. The sample position on POLARIS is 12.0m from the moderator, and the incident and transmitted neutron flux is monitored by two low efficiency glass scintillator detectors, positioned $\sim 4\text{m}$ before the sample position and $\sim 2.5\text{m}$ after it. The maximum beam dimensions at the sample position are 40mm high x 20mm wide, but two sets of stepper motor

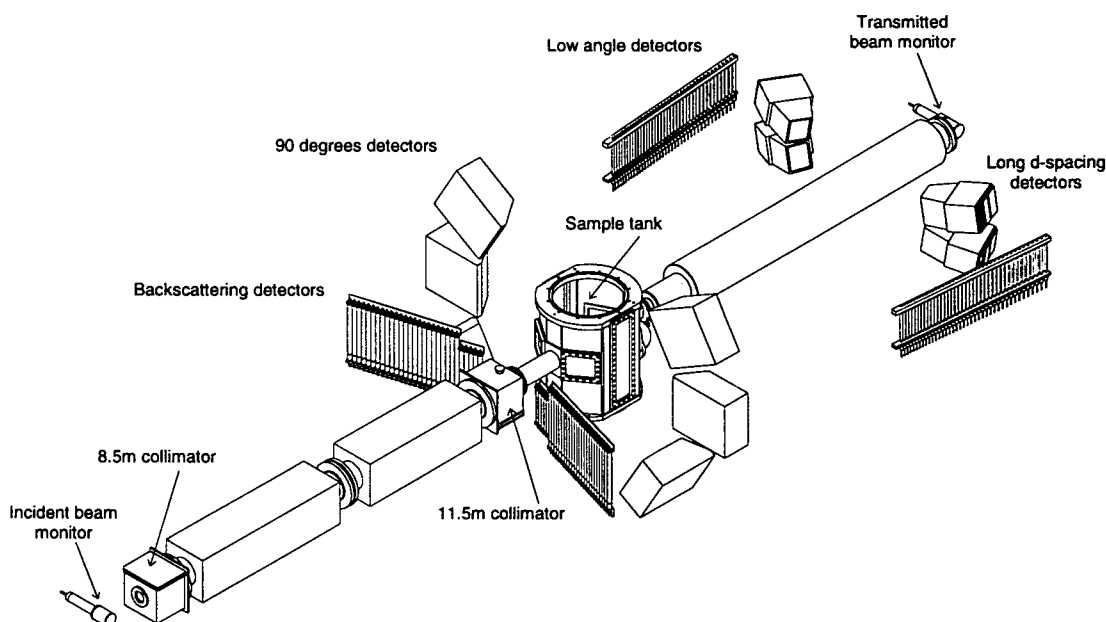


Figure 1. Schematic diagram of the POLARIS diffractometer

driven collimators in the incident beamline allow the beam size to be reduced, to match the sample size or to minimise the background from sample environment equipment. More detailed information on the use of this collimation can be provided by the instrument scientists (See Appendix D).

POLARIS has a total of 434 ^3He gas and ZnS scintillator detectors arranged into four separate banks: two at low angles, one at $2\theta \sim 90^\circ$ and one at backscattering angles. These are referred to as the A, B, E and C banks respectively. Details of these detector banks are given in Table 1, with a schematic diagram of POLARIS shown in figure 1.

Table 1. POLARIS detector configuration

	low angle (A)	low angle (B)	backscattering (C)	90 degrees (E)
detector type	^3He	ZnS	^3He	ZnS
no. of elements	$2 \times 40 = 80$	$4 \times 20 = 80$	$2 \times 29 = 58$	$6 \times 36 = 216$
L_2 (m)	1.72 - 2.65	~ 2.2	0.65 - 1.35	~ 0.80
2θ range	$28^\circ < 2\theta < 42^\circ$	$13^\circ < 2\theta < 15^\circ$	$130^\circ < 2\theta < 160^\circ$	$83^\circ < 2\theta < 97^\circ$
Ω (ster)	0.046	0.009	0.29	0.48
$\Delta d/d$	$\sim 1 \times 10^{-2}$	$\sim 3 \times 10^{-2}$	$\sim 5 \times 10^{-3}$	$\sim 7 \times 10^{-3}$
d -range (Å)	0.5 - 8.3	0.5 - 21.6	0.2 - 3.2	0.2 - 4.0
Q -range (Å $^{-1}$)	0.75 - 12.6	0.3 - 12.6	2.0 - 31.4	1.5 - 31.4

1.3 Principle of the Detector Arrangement

For a given Bragg reflection,

$$n\lambda = n \frac{h}{mv} = n \frac{h}{m} \frac{t}{L} = 2d \sin \theta \dots\dots\dots (1)$$

where λ is the neutron wavelength, h is Planck's constant, m is the neutron mass, t is the total time of flight, L is the total flight path from moderator to sample to detector, d is the interplanar spacing and 2θ is the Bragg scattering angle.

Substitution for h and m and conversion to appropriate units yields:

$$t(\mu\text{s}) = 505.56 L(\text{m}) \sin \theta d(\text{\AA}) \dots\dots\dots (2a)$$

$$\text{or} \quad d(\text{\AA}) = \frac{1.977 \times 10^{-3}}{L(\text{m}) \sin \theta} t(\mu\text{s}) \dots\dots\dots (2b)$$

From equation (2a) it can be seen that if the product $L \sin \theta$ is identical for all the detector elements in a bank, then neutrons scattered from a given crystallographic plane, hkl , will reach each of the detectors in that bank with exactly the same time-of-flight. Such an arrangement of detectors is said to be **time focused** (e.g., see C.G. Windsor, "Pulsed Neutron Scattering", Taylor & Francis, London, p237). On

POLARIS, however, the detectors are arranged into four discrete **resolution focused** banks, whereby all of the elements within each bank are arranged to have an approximately constant resolution. As a result, the individual detector elements are not time focused and this has important consequences in the data normalisation routines (section 4.4).

By partial differentiation of equation (2b) it is possible to derive an expression for the $\Delta d/d$ resolution of any detector.

$$R(d) = \Delta d/d = \left[\Delta\theta^2 \cot^2 \theta + \left(\frac{\Delta t}{t}\right)^2 + \left(\frac{\Delta L}{L}\right)^2 \right]^{1/2} \dots\dots\dots (3)$$

Inspection of equation (3) shows that for a given $\Delta\theta$, the contribution to $\Delta d/d$ from the angular uncertainties increases as the scattering angle decreases, becoming infinite at $2\theta=0^\circ$, while it is zero at $2\theta=180^\circ$.

In the very low angle B bank, the θ term dominates and constant resolution is obtained by arranging the detectors so that the $\Delta\theta \cot \theta$ term is constant. For detectors of identical size, this is achieved by positioning the detectors along a straight line, parallel to the incident neutron beam. In practice, however, the detector modules in the B bank are angled $\sim 10^\circ$ away from this ideal arrangement in order to increase the solid angle.

At large scattering angles the contribution of the $\Delta\theta \cot \theta$ term is small and the resolution is dominated by time uncertainties. The detectors in the backscattering C bank are arranged in a straight line inclined to the scattered neutron beam and all intersect approximately the same fraction of the Debye-Scherrer cones to give a constant resolution AND line shape across the bank. The poorer resolution of POLARIS compared with HRPD is due to larger $\Delta t/t$ values: Δt is approximately equal on the two instruments, but on HRPD t is a factor of 10 greater due to the larger flight path, giving a resolution 10 times better. However, POLARIS has the advantage of much higher intensities, particularly at short neutron wavelengths.

The d -spacing and Q ranges given in Table 1 refer to the average values covered by the whole bank. In the case of, for example, the A bank, longer d -spacings up to $\sim 11.1\text{\AA}$ (corresponding to a minimum Q of 0.5\AA^{-1}) can be accessed by considering only the detectors at lowest angles ($\sim 28^\circ 2\theta$) although obviously there will be a decrease in the counting statistics. However, it should be noted that these longer d -spacings will be recorded by the B bank detectors, albeit with poorer resolution and a lower count rate.

2. PERFORMING AN EXPERIMENT ON POLARIS

2.1 Sample Safety Assessment

All users complete a "Sample Record Sheet" as part of their application for ISIS beam time, the details on which are used to carry out a safety assessment. This will give comments (if any) from the ISIS Safety Section concerning possible chemical or radiological hazards associated with the sample. Any instructions concerning sample handling, etc. must be followed. Before the beginning of the experiment the user should collect their sample safety assessment sheet from the filing cabinet in the Data Assessment Centre (DAC) in the ISIS experimental hall (Building R55), after which it should be displayed in the pocket on the door to the POLARIS sample position for the duration of the experiment.

At the end of an experiment, before being removed from the ISIS experimental hall all samples which have been irradiated in the neutron beam **must** be monitored by the ISIS Health Physics Group (ext. 6696).

2.2 Loading a Sample

During data collection samples are normally held in thin walled cylindrical vanadium sample cans. These vary in length from 50mm to 75mm and have diameters ranging from 5mm to 11mm. When working with air sensitive samples they can be fitted with Teflon "O" rings for measurements at room temperature, Cu "O" rings for use in furnaces or indium seals for low temperature measurements. For data collection at room temperature, the sample cans are mounted on a "candlestick" so that the centre of the sample is 314mm from the lower face of the aluminium plate on the candlestick, figure 2.

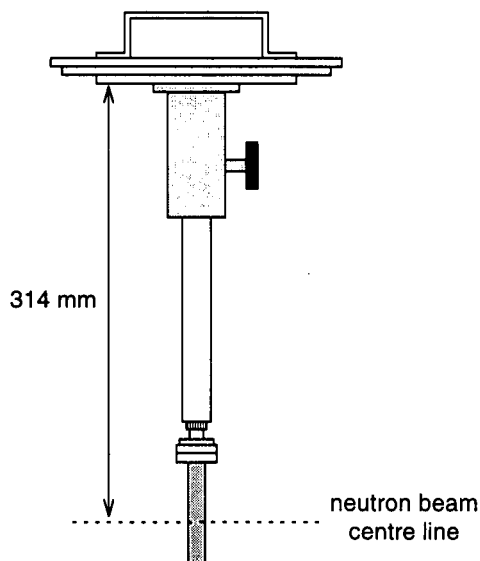


Figure 2. Sample alignment on POLARIS "candlestick".

Access to the POLARIS sample position is through the interlocked door at the front of the enclosure on top of the POLARIS blockhouse. When the POLARIS shutter is

closed (section 2.4), this door can be opened and the top of the sample tank is approximately 300mm below floor level.

The POLARIS sample tank accepts all standard ISIS sample environment apparatus such as cryostats, closed cycle refrigerators, furnaces, goniometers, pressure cells, etc. The sample mounting procedure for each of these is beyond the scope of this guide and details are available from ISIS User Support Group staff and instrument scientists.

After removal from the neutron beam all samples which have become activated due to neutron irradiation must be stored in a controlled area which, for POLARIS, is the POLARIS active samples cupboard situated close to the interlocked enclosure. Portable γ and β/γ radiation detectors are available on the beamline for monitoring samples after they have been removed from the beam. It should be noted at this point that the vanadium sample cans themselves become activated, emitting β radiation for approximately $\frac{1}{2}$ hour after removal from the neutron beam.

Handling of any sample having an induced activity $>75\mu\text{Sv/h}$ (including any activity arising from the sample can) must be carried out under the supervision of the ISIS duty officer (ext. 6789).

2.3 Sample Tank Evacuation

Attenuation of the incident and scattered neutron beam during data collection is reduced by evacuating the sample tank to a nominal pressure of ~ 0.1 mbar with a rotary pump. Also fitted is a turbo pump which will achieve a cryogenic vacuum of $\sim 10^{-4}$ mbar, necessary when operating a closed cycle refrigerator. The rotary pump is operated from the control unit located to the left of the shutter control system, beside the steps down to the instrument, figure 3. Two push button switches control the rotary pump and a solenoid operated vent valve while two warning lights indicate the running status of the rotary pump. Pirani and Penning gauges for monitoring the pressure in the sample tank and switches for operating the turbo pump are mounted in the rack above the vacuum control unit.

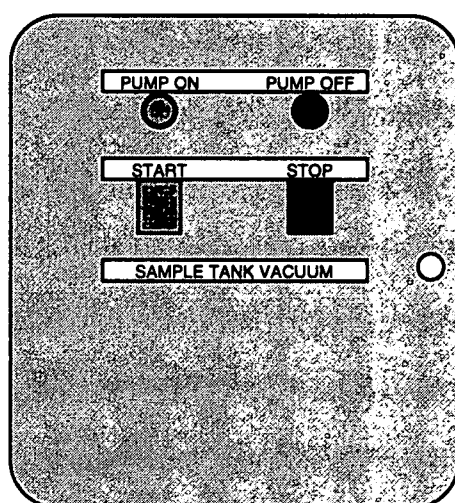


Figure 3. The POLARIS Sample Tank Vacuum Control

To evacuate the sample tank lift up the transparent cover and press the green switch labelled "START" on the control box. This closes the vent valve and starts the rotary pump. The green "PUMP ON" light on the control box will illuminate and the Pirani gauge will show the pressure in the sample tank dropping. After a short delay, the orange "VAC ON" warning lamp in the sample position enclosure will illuminate, providing an additional indication of the status of the sample tank vacuum.

The turbo pump need only be used when pressures of $<10^{-4}$ mbar are required, e.g. when using a closed cycle refrigerator, and is started by depressing the right hand switch on the panel between the Pirani and Penning pressure gauges.

To release the sample tank vacuum, lift up the transparent cover and press the red switch labelled "STOP" on the control unit. This stops the rotary pump and opens the vent valve. The red "PUMP OFF" light will illuminate and the Pirani gauge will show the pressure in the sample tank rising. If the turbo pump has been in use, it should first be switched off by pressing the right hand switch on the panel between the two pressure gauges. After 3-4 minutes, the pressure in the sample tank should reach atmospheric pressure, the "VAC ON" warning lamp in the sample position enclosure will extinguish and the sample "candlestick" can be removed.

In order to achieve a cryogenic vacuum in the sample tank, a thin window is fitted in the incident beam line, upstream of the sample tank. When changing samples users must ensure the sample tank has reached atmospheric pressure before attempting to remove the candlestick from the sample tank. Should any attempt be made to remove the candlestick while there is still a partial vacuum in the sample tank (indicated by the "VAC ON" warning lamp in the sample position enclosure) there is a risk that the pressure difference created across this window will cause it to tear.

2.4 Opening the POLARIS Beamline Shutter

Before the POLARIS beamline shutter can be opened, the interlocked door to the sample position enclosure must be closed and bolted. The interlock key (labelled "N7-A") then can be removed from the bolt lock and inserted into the vacant position in the shutter control system key rack (item A, figure 4), situated on the panel above the beam stop, beside the steps down to the POLARIS sample position enclosure.

When all seven keys labelled "N7-A" are present in this key rack, the key labelled "N7-M" (with a dark red tag attached) may be removed and inserted into the green shutter interlock box (item B, figure 4) where it is turned clockwise through 90° until it is horizontal. At this stage, users should check that the four red LED's labelled "GUARD LINE" on the orange interlock indicator box (item C, figure 4) are lit, indicating that all interlocks are in place and microswitches are closed. Any attempt to open the shutter without all four LED's being lit will immediately shut down the ISIS accelerator.

The POLARIS beamline shutter is then opened by lifting up the transparent cover and pressing the "OPEN" button on the neutron beam shutter control box (item D, figure 4). When the shutter is open the master key cannot be removed from the interlock box and there is no access to the interlocked areas.

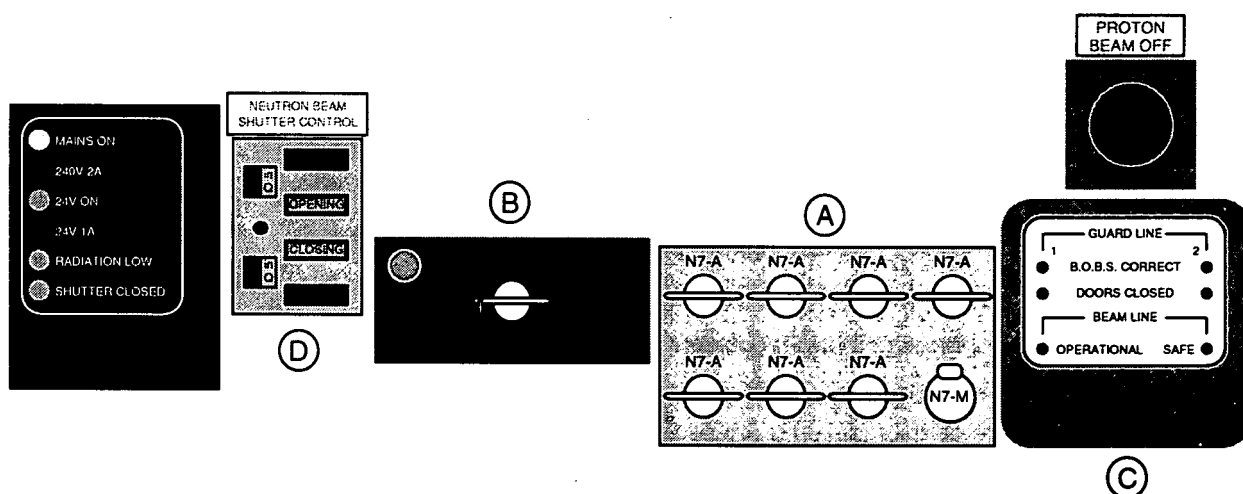


Figure 4. The POLARIS beamline shutter control and interlock system

To close the shutter lift up the transparent cover and press the "CLOSE" button on the shutter control unit. Once the shutter has closed the key labelled "N7 -M" can be removed from the interlock box and returned to the key rack, enabling one of the keys labelled "N7-A" to be removed to allow access to the sample position. The status of the POLARIS shutter is indicated by one of the four lamps on the neutron beam shutter control unit showing "OPEN", "OPENING", "CLOSING" or "CLOSED".

In common with all ISIS beamlines, POLARIS shutter open conditions are indicated by the following signals:

- (i) illumination of the "BEAM ON" lamps on top of the POLARIS sample position enclosure and on the target station wall, and
- (ii) the fluorescent lighting in the POLARIS blockhouse changing from white to blue and illumination of blue lighting in the vicinity of the sample position.

If any difficulties are encountered with the shutter or interlock system the user should immediately contact an instrument scientist or the ISIS Main Control Room (ext. 6789).

The remaining keys labelled "N7-A" in the interlock key rack allow access to other restricted areas along the beamline, for example the POLARIS blockhouse or chopper pit. Under normal circumstances, access to these areas is restricted to instrument scientists and ISIS User Support Group staff. In the event that users require access they must contact either an instrument scientist or the ISIS Main Control Room (ext. 6789)

In the unlikely event that any personnel become trapped inside an interlocked area, shutter open conditions will be indicated as described above. In such a case, the ISIS beam is switched off by pressing either of the Proton Beam Off buttons, which are located inside the POLARIS blockhouse on the beam stop wall and outside the blockhouse above the orange interlock indicator box on the beamline shutter control panel (figure 4).

3. DATA COLLECTION

3.1 Overview

Data collection on POLARIS is controlled by the Instrument Control Program (ICP) which runs on a computer known as the Front End Monitor (FEM), situated in the instrument cabin. The instrument settings pertaining to the run in progress are stored on the FEM in a file called the Current Run Parameter Table (CRPT), and during a run the diffraction data are accumulated in the Data Acquisition Electronics (DAE). At the end of each run the contents of the DAE are copied to the CRPT, which is then written to a file "POLnnnnn.RAW" on the FEM, known as a RAW file, where 'nnnnn' is a five figure run number. Shortly afterwards, the RAW file is archived onto optical disk. The FEM itself has a limited amount of disk space so RAW files are deleted periodically from the disk. These files can, of course, be restored from the optical disk so that data analysis can be performed at a later date.

The CAMAC control program runs on the FEM and provides a means of monitoring and changing sample environment parameters (e.g. temperature, etc.) by typing commands to the instrument computer. More detailed information on CAMAC is given in section 5.2 of the PuNCH User Manual, a copy of which is kept in the POLARIS cabin and which can also be obtained from the Computer Support office, Room 1.38, Building R3.

3.2 The POLARIS FEM

The POLARIS Front End Monitor (FEM) is used to control both data collection and sample environment parameters. The FEM is a Digital Alphastation 500/266 computer situated in the POLARIS cabin which runs the VMS operating system. Along with all the other ISIS instrument FEMs, it is linked to the central VAX 4500/4300 computer, which is known as the HUB, to form a "VMScluster". Within the VMScluster, the node name of the POLARIS FEM is "POL" and the node name of the HUB is "ISISE". One advantage of the VMScluster is that each computer has direct access to the disk storage area of any other computer in the cluster, so that users logged on to the HUB can access data files stored on the FEM. Note, however, that the POLARIS DAE can only be accessed from the POL FEM.

The *Motif* X-windows environment operating on the FEM allows users to run several sessions on the screen simultaneously, each logged onto POL independently and performing a different task, e.g. controlling the instrument, inspecting the experimental data, performing preliminary data analysis, etc. To open a new terminal window on the workstation, use the left mouse button to select the "DECTerm" option from the "Applications" pull-down menu in the "Session Manager" window. This will generate a new window which will automatically log on to the POL FEM with username POL.

Two useful options on the workstation, "shuffle up" and "shuffle down", are in a pull-down menu accessed by pressing the left hand mouse button while the pointer is in a background region of the screen. These allow windows to be pushed behind others or pulled to the front without changing the window to which keyboard input is directed.

3.3 The Dashboard

The POLARIS instrument dashboard, figure 4, shows details of the instrument configuration for the current experiment and will normally be displayed in the "Supervisor" window on the workstation (see section 3.4). If the dashboard is not present it can be displayed on any terminal logged onto the POL FEM (as username POL) with the STATUS command.

\$ **STAT ON<cr>** (to switch the dashboard off type **STAT OFF**)

At the top of the dashboard display the current DAE state (RUNNING, SETUP, etc., see section 3.5) and run number are shown. The remaining information on the dashboard gives information on the user, sample, run time, frame (proton pulse) count, present and accumulated proton beam current, the incident beam monitor counts and also displays values of any sample environment parameters being monitored by the CAMAC sample environment control program.

POL			is	RUNNING	RUN	16428
RB Number 0			Wed 27-NOV-1996 14:23:03			TEMP LOGG 4.28
User: Calibration			Tel: x5683, x6628			^-- raw --> 2.44
Title: NBS Silicon Detector Tests						TEMP1 LOGG 4.27
						^-- raw --> 1.93
Current run time 0 03:05:43			MONITOR			
Good/Raw frames 499894/ 499895			Spectrum 1			
Current/Total uA 195.4/ 502.2			From (mms) 2000.0			
DAE memory used. Bytes 8302932			To (mms) 2199.3			
318 Spectra 6506 Channels			Counts = 5177044			

Figure 5. The POLARIS Dashboard

3.4 The Instrument Control Program (ICP)

Data collection on POLARIS is controlled by the Instrument Control Program (ICP), which runs on the POL FEM. This program is used to start and stop data collection, modify instrument parameters and change sample environment conditions through the CAMAC control program. Communication between the ICP and CAMAC allows data collection to be suspended temporarily while a furnace or cryostat reaches its setpoint, and can also be used to suspend data collection automatically, e.g. if the temperature drifts away from limits defined by the user. The instrument control commands recognised by the ICP, along with their functions, are listed in Table 2.

To prevent unauthorised control of the instrument, the commands in Table 2 will only be accepted if issued from a session logged on the POL FEM with username POL and having the process name "supervisor". Normally, the window running this "supervisor" process will be displaying the POLARIS dashboard (section 3.3), will have its prompt displaying "Supervisor>" and will have "Supervisor" displayed in its title bar and icon title. Any attempt to control POLARIS from another process will result in the error message:

Failed to execute the command. STATUS= 148
Likely cause: Instrument under control of another terminal

Table 2. POLARIS ICP Commands.

BEGIN	Clears DAE memory (section 3.5) Sets parameters in DAE to those specified by CRPT (section 3.6) Instructs DAE to start data collection Sets DAE state to RUNNING
END	Stops data collection by DAE Copies data in DAE memory to CRPT Writes contents of CRPT to file 'POLnnnnn.RAW' Increments the run number 'nnnnn' Sets DAE state to SETUP
CHANGE	Enables the contents of the CRPT to be modified (section 3.6)
PAUSE	Suspends data collection by DAE Sets DAE state to PAUSED
RESUME	Resumes data collection by DAE Sets DAE state to RUNNING
UPDATE	Copies data in DAE memory to CRPT
STORE	Writes contents of CRPT to file 'POLnnnnn.SAV'
ABORT	Stops data collection by DAE Sets DAE state to SETUP

3.5 The Data Acquisition Electronics (DAE)

During the course of a run, diffraction data from POLARIS are accumulated in the Data Acquisition Electronics (DAE) as a series of spectra, each spectrum containing a histogram of neutron counts versus time-of-flight for a given detector. The DAE has four operating modes, listed in Table 3, and the current DAE mode is displayed at the top of the POLARIS dashboard (section 3.3). At the end of each run the data in the DAE are copied to the CRPT which is then written to a file named POLnnnnn.RAW on the POL FEM. At the end of each run the run number 'nnnnn' is incremented automatically. Shortly afterwards, each RAW file is automatically archived onto optical disk.

Table 3. POLARIS DAE Operating Modes.

SETUP	Data are not being collected and the CRPT may be changed before starting a new run (section 3.6)
RUNNING	Data are being collected and accumulated in the DAE
PAUSED	Data collection is temporarily suspended by the user
WAITING	Data collection is temporarily suspended by the FEM - this may occur if a furnace or cryostat drifts outside limits defined in the CAMAC sample environment control program, or while it heats or cools to a requested temperature

3.6 The Current Run Parameter Table (CRPT)

The Current Run Parameter Table (CRPT) is an area of memory in the ICP which holds a number of parameters associated with the current experiment, such as sample details, information on users (names and institutions) and the instrument configuration (times-of-flight of the time channel boundaries, monitor spectrum, mapping of individual POLARIS detectors into spectra in the GENIE data visualisation program (section 4.3), etc.). It also contains space into which the contents of the DAE can be copied at the end of, or during, a run. At the end of a run the contents of CRPT are written to a RAW file on the FEM.

At the beginning of an experiment it may be necessary to change some of the contents of the CRPT. This should be done in consultation with an instrument scientist. The command CHANGE should be typed at the "Supervisor" window when the DAE is in SETUP mode.

\$ CHANGE<cr>

The screen will clear and display the first of six pages which detail the current parameters in the POLARIS CRPT. The values in the various fields are altered using the DEFT screen editor, full details of which are contained in section 5.1.4 of the PuNCH manual. The "↑" and "↓" cursor keys are used to move from one field to another, while the "←" and "→" cursor keys and the "delete" key allow corrections to be made. The fields are altered simply by typing the appropriate characters or numbers into the field. Normally, users will only need to change details on the first page, and any changes to subsequent pages must be done only in consultation with an instrument scientist. If the field prompts with "toggle data type" the "." key on the right hand keypad should be pressed until the field displays the required option.

To exit from the editor and write the new parameters to the CRPT press the key marked "PF1" on the right hand keypad, then press "E" on the keyboard. If you wish to quit and leave the CRPT unchanged then press "PF1" followed by "Q".

During the course of a run it is possible to change the experiment title without using the DEFT editor. This is done with the CHANGE TITLE command, which can either be typed at the keyboard or given from a command file, regardless of the state of the DAE.

\$ CHANGE TITLE NiMgSiO4_1100_C_In_RALF1_Run_1<cr>

Note that the new title must be a continuous string of characters, hence the "_" symbol connecting different words. Note also that any lower case characters will be translated into upper case in the new title.

An alternative, which allows the use of spaces and lower case characters, uses triple sets of quotation marks (") at the beginning and end of the new title.

\$ CHANGE TITLE ""NiMgSiO4 1100 C in RAL F1 Run 1""<cr>

4. DATA ASSESSMENT

4.1 The PuNCH System

The PuNCH VMScluster at ISIS links all the instrument FEMs and the HUB (cluster node name "ISISE"), allowing these computers and their files to be accessed from the terminals situated in the DAC in the ISIS experimental hall (R55) and the offices in Building R3 as well as from the instrument FEMs. In addition, the HUB may also be reached from outside RAL via JANET, the Internet and DECnet.

4.2 Accessing POLARIS data

The procedures for routine inspection of POLARIS data will be described in the following sections (4.3 and 4.4). Except while carrying out an experiment on POLARIS, computationally intensive procedures such as data analysis and structure refinement should not be run on the POL FEM, but should be carried out on other machines in the VMScluster, such as the HUB (cluster node name "ISISE"). For this purpose users will require a user account, which can be arranged by one of the instrument scientists.

Users who have performed previous experiments at ISIS may already have their account set up with default settings to access data from another instrument. If so, an instrument scientist can arrange for these to be amended. In order to ensure that all the commands necessary to analyse POLARIS data are set up when logging in, users should edit their LOGIN.COM file so that it contains the command line:

\$ @POL\$DISK0:[POLMGR.COMMAND]LOGIN.COM

This will ensure the correct definition of the symbols and logical names used in POLARIS data analysis, including the GENIEINIT command file which initialises the GENIE data display and manipulation program (section 4.3) and sets up a series of symbols which point to command files used in data analysis. This LOGIN.COM file will also determine whether you have logged on to a VAX or an Alpha computer and set the logical name POL_PROGS to point to the directory containing the executable files for VAX or Alpha processors respectively.

Because the POLARIS DAE can only be accessed by the POL FEM, if you wish to examine the current run you will have to be logged on to POL. When working from your own user account the SET HOST command can be used to connect to the POL FEM.

\$ SET HOST POL<cr>

This will connect you to the POL FEM and prompt you to log on, which you should do with the username POL.

Username: **POL<cr>**

Password: **<cr>**

followed by

(Users can obtain the current POL password from an instrument scientist)

When using one of the workstations located in the DAC, another instrument cabin or building R3, it may be necessary to specify the terminal from which you are working in order that the GENIE graphics window is opened correctly. This is done with the SET DISPLAY command before starting up GENIE.

\$ SET DISPLAY /CREATE /NODE=nodename /TRANS=TCPIP<cr>,
or **\$ SET DISPLAY /CREATE /NODE=xxx_nnnnnnnn /TRANS=LAT<cr>**

where 'nodename' is either the VMS cluster node name of the workstation which you are using or a TCP address, xxx.xxx.xx.xx. The LAT name, xxx_nnnnnnnn, if required, will be displayed in the session manager window on the workstation which you are using.

To prevent the FEM from becoming overloaded, users attempting to log on to POL with their own username may find they are denied access. The SET HOST command can be used to connect to the HUB from any of the sessions running on the FEM.

\$ SET HOST ISISE<cr>

4.3 GENIE

During the course of an experiment it will be desirable to inspect the diffraction pattern whilst a run is still in progress, and also to compare it with the patterns from previous runs. This can all be done with the GENIE data display and manipulation program. Normally, GENIE will be running on one of the terminals in the POLARIS cabin, but it can also be run from any of the terminals in the DAC, or in the offices in building R3. Full details of the commands available in GENIE are given in the GENIE manual (Report RAL-86-102, "PuNCH Genie Manual") and Appendix 2 of the PuNCH manual. Only those commands necessary to perform a basic assessment of the data will be described here.

To start up GENIE simply type:

\$ GENIE <cr> (when working on a terminal other than the POL FEM,
it may be necessary to type "GENIE /DEV=MOTIF")

The terminal will clear, the GENIE header will be displayed at the top of the screen and several lines of text will scroll up the screen before the GENIE prompt ">>" is displayed. On a workstation, a window labelled "GKS" will be created (as an icon) where graphics will appear.

In order for GENIE to read and display ISIS data, its location (either in the DAE or from a RAW file) must be defined with the ASSIGN command followed by the data source. To look at the data in a RAW file, use the run number, e.g. for run 16361.

>> ASSIGN 16361<cr>

To look at the current run, use "DAE" in place of the run number.

>> ASSIGN DAE<cr>

As mentioned in sections 3.2 and 4.2, data from the current run are stored in the POLARIS DAE which can ONLY be accessed by the POL FEM. Therefore, ASSIGN DAE will only work from sessions logged on to POL.

Once the location of the data has been defined the contents of any spectrum, e.g. s33 (section 4.4), can be plotted with the DISPLAY command.

>> DISPLAY S33<cr>

On the workstation the data will be displayed in the GKS window as a plot of counts/microsecond against time-of-flight. On a PERICOM terminal the screen will toggle to graphics mode before displaying the data. To exit from the graphics display on the PERICOM terminal and return to the ">>" prompt, press the <Return> key.

In GENIE, a number of workspaces, denoted W'n', are defined which may be used to store a single spectrum or, more commonly, to store the result of some arithmetic operation involving several spectra. The current GENIE set-up on POLARIS allows users a choice of up to 26 workspaces. For example, to store the sum of spectrum 5 plus spectrum 34 divided by spectrum 1 in workspace 1 and then display the result, the commands would be:

```
>> W1=(S5+S34)/S1<cr> followed by  
>> DISPLAY W1<cr>
```

Further GENIE commands allow the user to modify the plot (alter the limits, change the units, etc.), fit functions to peaks in the data, etc. and full details of these are given in the GENIE manuals mentioned earlier. To exit from GENIE type:

```
>> EXIT<cr>
```

4.4 Normalising POLARIS Data

The data collected in each of the detectors on POLARIS are stored as a series of histograms of neutron counts versus total time-of-flight, in which the time-of-flight axes are split into a large number of 'bins' bounded by time channel boundaries (TCBs), defined by entries in the CRPT. Once in the DAE (and the RAW file at the end of the run), each histogram is referred to as a "spectrum". GENIE allows the user to inspect and manipulate these spectra, e.g. sum them together, convert from neutron time-of-flight to lattice d -spacing, etc.

The correspondence between the spectrum number 'n' and the position of the detector on POLARIS is determined by the wiring of the detector electronics and by data tables in the CRPT. The current parameters and spectrum numbers for each detector are listed in Appendix A.

As discussed in sections 1.2 and 1.3, the $\Delta d/d$ resolution of the POLARIS detector banks varies with scattering angle (2θ), and for this reason each bank is treated separately when normalising the data. Because each of the detectors in a particular bank has approximately the same resolution, all the detectors in that bank can be summed (or ***focused***) into a single GENIE workspace to give a significant improvement in the counting statistics. POLARIS data normalisation is carried out in three discrete steps through a set of GENIE command files, one command file for each detector bank (see also Appendix C).

First, the spectra from all the detectors in a given bank are focused into a single GENIE workspace, which is then divided by the incident monitor counts to normalise to counting time. Next, an instrument background spectrum is subtracted and finally the data are divided by a vanadium spectrum. The vanadium spectrum, which has been corrected for absorption and multiple scattering effects, normalises the data to the energy distribution of the incident neutron flux, taking into account the variation in detector efficiency with neutron energy. The background and vanadium data sets

used here are collected at the beginning of each ISIS cycle and are made available for use in normalising all POLARIS data during that cycle.

Because the detectors within each bank are not time focused (section 1.3), they cannot be summed directly in time-of-flight. Instead, the data are converted to a d -spacing scale (see equation (2b), section 1.3) as the spectra are summed in GENIE. The value of the product $L \sin \theta$ required for this conversion is obtained from a calibration of POLARIS using the NBS standard silicon sample (SRM 640b).

One of two procedures is used to normalise the data, depending upon whether the data were collected during the current ISIS cycle or during a previous cycle. For current data, only the run number is required, but for old data the name of the command file appropriate to the ISIS run cycle during which the data were collected must be determined.

4.4.1 Data from the Current ISIS Cycle

To normalise data from a given detector bank in GENIE, type the name of the focusing command file for that bank. The command file prompts first for the run number and then for the number of a GENIE workspace into which the final spectrum will be placed. For example, to focus and normalise the data from run number 11916 into workspace 1.

```
>> AFOCUS<cr> (or BFOCUS, or CFOCUS, or EFOCUS)
```

```
>> Enter run number : 11916<cr> (or DAE)
```

```
>> Enter workspace number (not 15) : 1<cr>
```

To normalise the data currently being collected type "DAE" instead of a run number (n.b. this is ONLY possible when logged onto the POL FEM). Note that workspace number 15 is used by the command file as a temporary store while focusing and normalising the data and should not be selected to contain the focused data. The data may then be viewed with the DISPLAY command, as described in the previous section.

```
>> DISPLAY W1<cr>
```

4.4.2 Data from Previous ISIS Cycles

First, before running GENIE, it is advisable first to check that the RAW file is still on the POL disk with the DIRECTORY command.

```
$ DIR POL_DATA:POLnnnnn.RAW<cr>
```

If the file does not appear in the directory listing, it can be retrieved from the optical disk archive system using the RESTPOL command, as described in section 4.7.

The data normalisation procedure is then similar to that given above for the current cycle, except that the full name of the command file which selects the normalisation runs (and, perhaps, a different detector configuration) appropriate to that cycle must be given explicitly by the user.

```
>> @POL_COMMAND:AFnnnnn.COM<cr> (or BFnnnnn, etc.)
```

In this case 'nnnnn' is the four or five figure number of the silicon calibration run collected during the same ISIS cycle as the old data. A listing of all the available GENIE command files can be found from a directory of POL_COMMAND:*F*.COM. In general, use the number which is closest to, but lower than, the run number of the data to be normalised. In case of any doubt, an instrument scientist will be able to advise.

4.4.3 Useful GENIE Commands

It is often desirable to save the information contained in a GENIE workspace to a file so that the data can be accessed quickly and efficiently at a later date. One option available allows the data to be written in binary format to a GENIE intermediate format file which stores not only the x, y, $\sigma(y)$ information, but also all the parameters associated with that workspace (experiment title, users' names, flight paths, scattering angle, etc.). This file is written using the WRITE command.

```
>> WRITE/OPEN W1 AL2O3.NOR<cr>      followed by  
>> WRITE/CLOSE<cr>
```

Here, the contents of GENIE workspace W1 are written to the file AL2O3.NOR. The "WRITE/CLOSE" command is necessary to ensure that all the data are written to the file and the file is closed. The GENIE intermediate format file is read back into GENIE with the READ command.

```
>> READ W2 AL2O3.NOR<cr>  
>> DISPLAY W2<cr>
```

Here, the file AL2O3.NOR is read back in to GENIE workspace W2 and, when plotted, all the information contained in the original workspace is preserved.

It is also possible to write the contents of a workspace to an ASCII file, e.g. for use in an alternative data analysis package or for transport to another computing facility, with the SHOW DATA command.

```
>> SHOW DATA Wn /OUT=filename<cr>
```

where 'n' is the number of the workspace containing the data. The "/OUT=filename" qualifier is optional and allows the name of the file to be specified. If omitted, a default filename of "FOR001.DAT" is used. Alternatively, use of "/OUT=TT" will result in the output listing being directed to the screen.

Once a normalised diffraction pattern (or, indeed, any other GENIE image) has been displayed on the graphics screen it is possible to write the image to a file which can then be printed on one of the laser printers, transferred to a remote site using FTP, etc. The file is written with the KEEP/HARD command and is given the filename DEC_device.DAT where 'device' is a code word for the file format.

```
>> KEEP/HARD<cr>
```

The default device is "POSTSCRIPT", but this can be changed either by adding the qualifier "/HARD=device" when starting GENIE or from within GENIE itself with the SET HARD command (used *before* typing "KEEP/HARD").

e.g. **\$ GENIE/HARD=HP7475<cr>**
and **>> SET HARD HP7475<cr>** are equivalent

This alternative device type, "HP7475", creates HPGL format files, which are particularly useful because they can be incorporated into Word documents and, furthermore, they can be viewed and edited in Word as a Microsoft Drawing or a Microsoft Word 6.0 Picture. A more detailed list of available device types can be obtained through ISIS Computer Support.

To print a POSTSCRIPT format file on one of the ISIS laser printers, the command PLASER is available.

\$ PLASERn DEC_POSTSCRIPT.DAT<cr>

where 'n' is the number of the laser printer.

A command file has been written which will save a graphic image and print it out on a laser printer automatically. Once the image has been created on the screen type HC (on a PERICOM terminal it will be necessary first to leave the graphics screen by hitting <cr> before typing HC).

>> HC<cr>

This prompts for a choice of printer from those situated in building R55 in the DAC (No 2) or the LAD cabin (No 3) or in building R3 in rooms 1-38 (No 0) or 2-9/10 (No 1), creates the postscript format file "DEC_POSTSCRIPT.DAT" and submits this file to the desired printer queue to be printed.

4.5 Absorption Corrections

Sample absorption effects are wavelength dependent and can be very significant for some elements and isotopes. A routine is available which will take a normalised POLARIS diffraction pattern stored in a GENIE workspace and correct the data for sample absorption. Before running the program, the sample mass and dimensions must be measured, the number density of scattering units in the sample calculated and the total scattering and total absorption (at a neutron velocity of 2200ms^{-1}) cross sections per scattering unit determined.

A worked example for alumina, Al_2O_3 , is given below.

Mass of can + sample:	31.63 g
Mass of empty can:	19.98 g
Mass of sample:	11.65 g
Diameter of sample can:	0.952 cm
Radius of can (r):	0.476 cm
Height of sample (h):	5.5 cm

Atom	mass (amu)	σ_s	σ_a
Al	27	1.503	0.231
O	16	4.232	0.0002

σ_s is the scattering cross section per atom in barns and σ_a is the absorption cross section per atom in barns at a neutron velocity of 2200ms^{-1} . Values for these parameters for most elements and isotopes can be found in the tables in the

POLARIS cabin, or in the article by Sears (V.F. Sears, "Neutron Scattering Lengths and Cross Sections", Neutron News, 1992, 3, 26).

First calculate the mass of one Al_2O_3 scattering unit in grams:

$$m = (26.982 \times 2 + 15.999 \times 3) / (6.022 \times 10^{23}) = 1.694 \times 10^{-22} \text{g}$$

Next calculate the sample volume:

$$V = \pi r^2 h = 3.141 \times 0.476^2 \times 5.5 = 3.914 \text{cm}^3$$

Then calculate the number of scattering units/cm³:

$$\begin{aligned} N/V &= (\text{weight of sample}) / (\text{mass of scattering unit} \times \text{volume}) \\ &= 11.65 / (1.694 \times 10^{-22} \times 3.914) = 1.757 \times 10^{22} \text{cm}^{-3} \end{aligned}$$

Finally, calculate the total scattering cross section per scattering unit:

$$\sigma_{\text{sc}} = 2 \times 1.503 + 3 \times 4.232 = 15.70 \text{ barns}$$

and the absorption cross section per scattering unit:

$$\sigma_{\text{ab}} = 2 \times 0.231 + 3 \times 0.0002 = 0.4626 \text{ barns at a neutron velocity of } 2200 \text{ms}^{-1}$$

The program is run from GENIE with the CORRECT command.

```
>> CORRECT<cr>
>> Enter workspace containing uncorrected data: 1<cr>
>> Enter workspace to contain corrected data: 2<cr>
Enter cylindrical radius (cm): 0.476<cr>
Enter number density of scattering units (cm-3): 1.757E22<cr>
Enter scattering cross section (barns): 15.70<cr>
Enter absorption cross section (barns @ 2200m/sec): 0.4626<cr>
```

The program determines the POLARIS detector bank in which the data were collected, calculates and applies the absorption correction for that scattering geometry and passes the corrected diffraction pattern back to the given GENIE workspace.

4.6 Profile Refinement

4.6.1 CCSL Based Software

Several programs based on the Cambridge Crystallography Subroutine Library (CCSL) have been written for the analysis of ISIS time-of-flight powder diffraction data. Full details on the use of these programs are given in the report RAL-92-032, "Profile Analysis Of Neutron Powder Diffraction Data At ISIS", copies of which can be obtained through an instrument scientist.

The CCSL program recommended for structure refinement using POLARIS data, TF14LS, requires two input files. The *diffraction data* file has the extension ".DAT" and contains the normalised experimental diffraction profile, corrected for sample absorption effects if necessary. The *crystal data* file has the extension ".CCL" and contains the structural (unit cell, space group, atomic co-ordinates, etc.), instrumental (total flight path, average scattering angle, zero point, line shape and line width) and least squares parameters required to construct the calculated profile

refinement. The instrument parameters in the crystal data file have been determined empirically using data collected from NBS standard samples.

The diffraction data file is in ASCII format and is generated from the binary data stored in a GENIE workspace with the TF_FILE command.

```
>> TF_FILE<cr>
>> Enter number of workspace containing the data : 1<cr>
Enter data filename (without extension) :C16628<cr>
Enter minimum t.o.f limit (usec) :2000<cr>
Enter maximum t.o.f limit (usec) :19550<cr>
```

The crystal data file is generated from one of a series of template files *Xnnnnn.CCL*, where 'X' is the Polaris detector bank (A, B, C or E) and 'nnnnn' corresponds to a silicon calibration run number, which can be found in the directory *pol_calib_silicon*. The appropriate silicon run number 'nnnnn' is determined in a manner similar to that described in section 4.4.2 for focusing data from previous ISIS cycles. Take a copy of the file *Xnnnnn.CCL* with run number closest to but lower than the run number of your own data.

To run the profile refinement program, simply type the program name followed by the input file name (without any extension) and an option for batch or interactive running.

```
$ TF14LS C16228 BATCH          (or "INTERACTIVE" for interactive running)
```

During structure refinement the appropriate extensions are assigned automatically to the input and output data file names. In general, three output files will be of interest: the *line printer* file (extension ".OUT"), the *profiles* file (extension ".PRO") and the *updated crystal data* file (extension ".CCN").

After examining the profile fit (section 4.6.2) and reading through the .OUT file, the next stage in the structure refinement procedure will be to introduce further variable parameters into the least squares. This can be done by taking the .CCN file containing the refined values of the variable parameters from the previous cycles of least squares, editing the least squares information and writing a new data file with the extension ".CCL". The REVI command is available to facilitate this.

```
$ REVI C16228<cr>
```

This starts the EDT editor and reads in the .CCN file. At the end of the editing session, typing <Ctrl-Z> exits from the editor and automatically writes the new file with the extension ".CCL".

4.6.2 Examining the Profile Fit

The .PRO output file contains the observed and calculated diffraction profiles, which can be inspected in GENIE in order to examine the profile fit. The PLS command loads the observed, calculated and difference profiles into GENIE workspaces 20, 21 and 22, the background profile into workspace 23, and the positions of reflection markers for up to three crystalline phases into workspaces 24, 25 and 26.


```
>> PLS<cr>
>> Enter filename (max 6 characters) : C16228<cr>
>> Enter lower and upper limits : <cr>
>> Enter binning : 1<cr>
```

Entering <cr> in response to the lower and upper time-of-flight limits causes default values corresponding to the minimum and maximum times of flight in the .DAT file to be used. Once the profiles have been read into GENIE using PLS, they can then be examined in smaller sections with the PLC command.

```
>> PLC<cr>
>> Enter lower and upper limits : 3000 5000<cr>
>> Enter binning : 1<cr>
```

If desired, the data in workspaces 20 to 26 can be converted to lattice d -spacing with the U/D command (units in d -spacing) and then the profiles displayed using PLC.

```
>> U/D W20<cr>    (and W21, W22, etc.)
```

To convert the data back to time-of-flight use the U/T command (units in total time-of-flight).

The positions of the allowed Bragg reflections can be displayed as vertical bars on the difference plot using the P/T command (plot using "tics").

```
>> P/T W24 ymin ymax<cr>    (or W25, or W26)
```

This will draw vertical bars between the given y-axis (intensity) limits at each of the x-axis (time-of flight, or d -spacing) positions

4.6.3 GSAS

Also available for profile refinement using ISIS data is GSAS (A.C. Larson and R.B. Von Dreele, GSAS General Structure Analysis System, Report LAUR 86-748, Los Alamos National Laboratory, 1994). Please note that this software is not supported by ISIS staff and that any problems should be addressed to the program authors. Also, no guarantee can be given that the instrument parameter file (below) is kept up-to-date.

The data files required by GSAS are in a different format to those used by the CCSL based routines. The ASCII format *GSAS standard powder data* file containing the normalised diffraction profile is generated from the data stored in a GENIE workspace with the GSAS_FILE command.

```
>> GSAS_FILE<cr>
```

This prompts for the number of the workspace containing the normalised diffraction profile before scrolling through several screens of information, asking for a detector bank number, time-of-flight limits, data file name, title, etc. In particular, the bank number relates to a series of entries in a file known as the *instrument parameter* file, which contains flight path, scattering angle and line shape parameters for the detector bank in which the data were collected. The POLARIS instrument parameter file, filename 3BANKS.POL, contains parameters appropriate to the A, C and E detector banks (bank numbers 3, 2 and 1 respectively) and can be found in the directory POL\$DISK0:[POL].

4.7 Retrieving Archived POLARIS Data

To restore RAW data files which have been deleted from the disk on the POL FEM onto the scratch disk the RESTPOL command can be used. Note, however, that this command may not execute correctly on VMScluster nodes other than ISISE.

\$ RESTPOL<cr>

Use to restore raw and log data files to SCRATCH\$DISK:[POLMGR.RESTORE]

Restored files are not guaranteed to stay for more than SEVEN days

Which file type? Enter RAW or LOG =>: **raw<cr>**

Enter run number for first file =>: **11917<cr>**

Enter run number for last file =>: **11919<cr>**

A restore subprocess has been spawned. DO NOT LOG OUT until this subprocess has completed.

Online restores should appear in SCRATCH\$DISK:[POLMGR.RESTORE] within about 10 minutes.

Offline restores may take much longer (up to 24 hours).

To check your process type: \$ SHOW PROC/SUBPROC

Subprocess user01_1 has completed

System responses to the restore requests are written to a file RESTLOG.LOG in the user's current default directory. Once the subprocess has completed, the restore requests will appear on one of the two archive restore queues, "online" or "offline". This can be checked with the MANODSR command.

\$ MANODSR /LIST=R<cr>

"Online" restores will be returned after approximately 10 minutes, although this can be as long as an hour or two if there are many files queued to be restored. "Offline" restores may take as long as one day to be returned. However, be warned - files on the scratch disk have only a limited lifetime and will be deleted again after ~2 weeks.

Because the directory SCRATCH\$DISK:[POLMGR.RESTORE] is included in the definition of the logical POL_DATA, all restored files can be read by GENIE without further user intervention.

4.8 Miscellaneous POLARIS Routines

4.8.1 Useful DCL Commands

(a) ISISNEWS

Periodically, the ISIS Main Control Room crew will broadcast messages giving information on the current operational status of ISIS to all terminals logged on the VMScluster, e.g. scheduled liquid transfers in the methane moderator, estimates of beam on times during repairs to ISIS, etc. As well as being broadcast to the terminals, these messages are also stored in a log file which can be inspected at

any time using the ISISNEWS command. Within the ISISNEWS routine there are four separate topics: Current_status, Notes, Summary and Unavailability. To view the messages in the log file use the Current_status topic option.

\$ ISISNEWS Current_status (can be abbreviated to **ISISNEWS C**)

(b) WAITFOR

This command is very useful when included in a command file. It interrogates the DAE and will cause the command file to wait at a particular point until the desired number of microAmp hours (or ISIS frames, or monitor counts) has been reached before continuing on to the next command line. The format of the command is:

\$ WAITFOR nnn UAMPS (or **nnn FRAMES**, or **nnn COUNTS**),

where 'nnn' is an appropriate integer.

(c) POLPARS

This command interrogates a single RAW file, or a series of RAW files, and returns selected run information either to the screen or to a user defined file.

\$ POLPARS<cr>

***** RUN PARAMETER SUMMARY FOR POLARIS DATA *****

Output to a file (default is the screen) (Y/N) : **Y<cr>**

Enter name for output file : **summary.dat<cr>**

Enter first run number : **10226<cr>**

Enter last run number : **<cr>** (default, <cr>, makes last run=first run)

SEARCHING FOR FILE : pol_data:pol10226.raw
FILE OPENED

The NBS Standard Silicon Sample

run number	10226
user was	Commissioning
start date/time	5-OCT-1993 14:39:20
finish date/time.....	6-OCT-1993 08:18:18
no. of spectra	318
acquisition mode	const T mode
number of time channels ..	6506
time channel increment.....	1.0000
TOF window (ms)	0.- 2.
GFC/RFC.....	2629178 2629179
Monitor counts	30205916
run duration (in hrs.)	17.65
run duration (in uA.Hr)	2704.5

4.8.2 Useful GENIE Commands

(a) PLOT2 (Also available PLOT3, PLOT4)

This plots two (or three or four) workspaces on a single graphics screen. It is useful where a comparison of two or more data sets is required.

```
>> PLOT2<cr>      (or PLOT3, or PLOT4)
>> Enter number of workspace at top : 1<cr>
>> Enter number of workspace at bottom : 1<cr>
>> Enter binning : 1<cr>
>> Enter lower x limit : 1.0<cr>
>> Enter upper x limit : 2.0<cr>
```

If <cr> is entered in response to any of these prompts, default values are taken (on a PERICOM terminal the <Return> key must be pressed after the first plot to return the screen to text mode from graphics mode). The y limits of the second plot are usually chosen to be identical to the default values for the first plot if a comparison between the relative intensities of spectra in two different workspaces is required.

(b) TPLOT

This plots the information contained in the sample environment log file, either for the current run or for a given run number.

```
>> TPLOT<cr>
>> Give run number: 16518<cr>      (or DAE<cr>)
>> Give workspace: 1<cr>
Give start date/time : <cr>
Give finish time : <cr>
Give Se block name : TEMP<cr>      (or TEMP1<cr>)
Please give units (K or C) : K<cr>
Which log column do you want, 2 or 3 (def 3)? <cr>
```

The SE (sample environment) block name usually refers to either TEMP or TEMP1. If more than two temperatures are being monitored then, e.g., TEMP2, TEMP3, etc. can also be plotted. The units determines the labelling, in K or C, on the y axis of the plot. The log column refers to either the raw mV reading (column 2) or the converted temperature in Kelvin or degrees Centigrade (column 3).

(c) GAUSS

This is used to fit a Gaussian function to a single peak displayed in GENIE. In general GEC is to be preferred (see below), although GAUSS can be used for fitting data from the B bank detectors, where the line shape is dominated by uncertainties in the scattering angle and, consequently, is well approximated by a Gaussian function.

```
>> GAUSS<cr>
>> Enter xmin : 2.3<cr>
>> Enter xmax : 2.6<cr>
```

If only <cr> is entered in response to the prompts for xmin and xmax, the cursor appears on the plot and the fitting region can be selected by moving the cursor to the left of the peak and typing L then moving the cursor to the right of the peak and typing R.

(d) GEC

This is identical to GAUSS except that the functional form used to fit the peak is a convolution of a Gaussian (which models angular contribution to the line shape) and an exponential decay term (which models the moderator contribution to the line shape).

>> GEC<cr>

Appendix A. POLARIS INSTRUMENT PARAMETERS

A.1 Table of Detector Parameters (June 1997)

Spectrum	Detector	L2 (m)	2 θ (°)	Lsin θ (m)*
S1	M1	-4.25	-	-
S2	M2	2.53	-	-
S3	not used	-	-	-
S4	DAE test	-	-	-
S5 - S33	C1 - C29	0.55 -1.35	160.0 -130.0	12.441 -12.069
S34 - S62	C30 - C58	0.55 -1.35	160.0 -130.0	12.428 -12.061
S63 - S102	A1 - A40	2.60 -1.70	28.4 - 41.3	3.489 - 4.787
S103 - S142	A41 - A80	2.60 -1.70	28.4 - 41.3	3.475 - 4.771
S143 - S178	E1 - E36	~0.86	95.0 - 85.0	9.292 - 8.685
S179 - S214	E37 - E72	~0.86	95.0 - 85.0	9.277 - 8.673
S215 - S250	E73 - E108	~0.80	85.0 - 95.0	8.706 - 9.329
S251 - S286	E109 - E144	~0.80	85.0 - 95.0	8.676 - 9.293
S287 - S322	E145 - E180	~0.80	85.0 - 95.0	8.760 - 9.372
S323 - S358	E181 - E216	~0.80	85.0 - 95.0	8.787 - 9.413
S359 - S378	B1 - B20	~2.2	15 -11	1.822 -1.604
S379 - S398	B21 - B40	~2.2	15 -11	1.818 -1.604
S399 - S418	B41 - B60	~2.2	15 -11	1.816 -1.605
S419 - S438	B61 - B80	~2.2	15 -11	1.820 -1.608

* Lsin θ value refined from NBS Silicon standard reference material (SRM 640b).

Note: To collect data in all four Brick detectors select file *inst_tables:wiring.4bricks* when changing the CRPT and connect the ribbon cables marked "BRICK 3" and "BRICK 4" to DIM2s in slots 9 and 10 respectively in instrument crate (normally used for detectors PC TEB1 and PC TEB2).

Appendix B. GENERAL INFORMATION

B.1 Useful Telephone Numbers

	Extension
Instrument Scientists:	
Steve Hull	6628
Ron Smith	5683
Other useful numbers:	
EMERGENCY	2222
POLARIS cabin	6819
ISIS Main Control Room	6789
ISIS Health Physics	6696
University Liaison	5592 (phone)
	5103 (fax)
Computer Support	3029 (mobile)
	5414 (office)
Main Gate	5545
Operator (daytime)	0
Outside line	9

For specific problems concerning sample environment equipment, computing, electronics etc., an up to date list of the relevant personnel and their telephone numbers is displayed in the POLARIS instrument cabin.

Appendix C. POLARIS DATA NORMALISATION

The information in this Appendix provides users with sufficient information about the POLARIS data normalisation procedures to enable them to take the standard GENIE command files and modify them to their own requirements. This may be necessary when customised sample environment equipment gives rise to a significant background signal or when unusual samples require the use of particular detectors within a bank.

C.1 Introduction

In order to improve the counting statistics, data collected by each of the detectors in a bank are summed (or ***focused***) into a single GENIE workspace before being normalised. Because the detector banks on POLARIS are not time focused, neutrons scattered from a given crystallographic plane, *hkl*, reach each of the detectors in a bank at different times-of-flight. Therefore, while carrying out the summation the spectra containing the data collected by each of the individual detectors in the bank are converted from time-of-flight to d -spacing before being accumulated into the sum.

After being converted back into units of time-of-flight, the focused data are divided by the monitor counts for that run (calculated by integrating the incident beam monitor spectrum over a pre-determined time-of-flight range) to normalise to the total number of neutrons incident on the sample. Finally, the data have an instrumental background subtracted (data collected from an empty sample tank) and are normalised to the POLARIS incident flux profile, determined using data collected from an incoherently scattering sample (a vanadium rod).

Focusing and normalisation of POLARIS data is carried out in GENIE using a series of command files, one for each detector bank, called AFOCUS.COM, BFOCUS.COM, CFOCUS.COM and EFOCUS.COM.

C.2 POLARIS Normalisation Data Sets

At the beginning of each ISIS cycle, three standard data sets are collected: from the NBS standard silicon sample, from a vanadium sample and from the empty POLARIS sample tank. From these data sets are produced a set of files which are made available to all POLARIS users for normalisation of their data and subsequent structure refinement.

In addition to the standard POLARIS focusing command files three additional GENIE routines are used to produce the normalisation data files.

ESPLINE:	smoothes the empty sample tank data
VSPLINE:	smoothes the vanadium rod data
VCORRECT:	corrects the vanadium rod data for absorption and multiple scattering effects

In order to run these routines, appropriate symbols need to be defined in GENIE with the SETUP command.

>> SETUP

C.3 Analysis of Normalisation Data Sets

In this section, the procedure for creating the background and vanadium data sets required by the AFOCUS.COM, etc. normalisation command files is described.

First, however, before starting GENIE copy the appropriate focusing command file (AFOCUS.COM, etc.) from the directory pol\$disk0:[polmgr.command.genie] to a suitable working directory, e.g. on the scratch disk. Then, edit this file and "comment out" the four command lines (which begin with ">") in each of the two sections headed "SUBTRACT INSTRUMENTAL BACKGROUND" and "NORMALISE TO VANADIUM SPECTRUM" near the end of the file by inserting a "!" into column 1 of the command line.

This modified command file is used in GENIE to focus the background and vanadium data sets, which are then smoothed and corrected before being written to GENIE intermediate format files. Finally, an updated focusing command file is written to use these new normalisation data sets. In the filenames which follow, 'nnnnn' refers to the run number of an appropriate background, vanadium or silicon normalisation/calibration run.

C.3.1 Empty Sample Tank Data

(1) Focus the data, using the modified AFOCUS, etc. command file.

>> **@AFOCUS** (or **@BFOCUS**, etc.) store focused data in, e.g., W1

(2) Smooth the focused data.

>> **ESPLINE**

>> Enter workspace containing raw data: **1**

>> Enter workspace to contain smoothed data: **2**

(3) Write the smoothed data to a GENIE intermediate format file.

>> **WRITE/OPEN W2 ABnnnnn.DAT** (or **BBnnnnn.DAT**, etc.)

>> **WRITE/CLOSE**

Note that by using data collected from an empty furnace or cryostat instead of the empty sample tank, a command file which subtracts the background from a furnace or cryostat is created. In this case it is suggested that both the background data and focusing command file names are amended to show this (e.g. are given extensions such as *.RALF1* or *.CRYO*). Also, if necessary, individual detectors (or groups of detectors) can be excluded from the summation by commenting out the appropriate sequence of 5 command lines which add that particular detector into the sum in the AFOCUS.COM, etc. GENIE command file.

C.3.2 Vanadium Rod Data

(1) Focus the data, using edited AFOCUS, etc. command file.

>> **@AFOCUS** (or **@BFOCUS**, etc.) store focused data in, e.g., W3

(2) Smooth the focused data.

>> **VSPLINE**

>> Enter workspace containing raw data: **3**

>> Enter workspace to contain smoothed data: **4**

(3) Subtract the smoothed instrument background.

>> **W5=W4-W2**

(4) Correct for absorption and multiple scattering.

>> **VCORRECT**

>> Enter workspace containing uncorrected data: **5**

>> Enter workspace to contain corrected data: **6**

(5) Write the smoothed, corrected data to a GENIE intermediate format file.

>> **WRITE/OPEN W6 AVnnnnn.DAT (or BVnnnnn.DAT, etc.)**

>> **WRITE/CLOSE**

(Note: if the ISIS target is uranium, an additional calculation is required between steps (4) and (5) above to correct for a time independent (or "delayed") neutron flux.

>> **TR W6 pol_progs:DELAY W7**

In this instance, in step (5) the contents of W7 should be written to the GENIE intermediate format file.)

Finally, edit the modified AFOCUS.COM, etc. command file and reinstate the "commented out" background subtraction and vanadium division command lines. At the same time, modify the command lines which read the background and vanadium data to give the new file location and run number details.

The new background, vanadium and focusing normalisation files, ABnnnnn.DAT, AVnnnnn.DAT and AFnnnnn.COM, etc. can then be copied to an appropriate directory on, e.g., the user disk.

\$ copy ABnnnnn.DAT user\$disk:[user01.work]ABnnnnn.DAT
(or **BBnnnnn.DAT**, etc.)

\$ copy AVnnnnn.DAT user\$disk:[user01.work]AVnnnnn.DAT
(or **BVnnnnn.DAT**, etc.)

\$ copy AFOCUS.COM user\$disk:[user01.work]AFnnnnn.COM
(or **BFnnnnn.COM**, etc.)

The incorporation of run numbers into these file names provides a convenient mechanism for normalising old POLARIS data, which may have been collected with a different instrument configuration. The only information required is the run number of the silicon data set collected at the beginning of that ISIS cycle and the appropriate command file is then run in GENIE by using the full file name, including its disk and directory specification if required.

>> **@user\$disk:[user01.work]AFnnnnn.COM (or BFnnnnn, etc.)**

Appendix D. OPERATION OF *Compumotor* JAWS CONTROLLER ON POLARIS

This Appendix describes the operation of the *Compumotor* stepper motor control units which operate the POLARIS beam-defining collimation. Changing the collimation settings can seriously affect the performance of the POLARIS data normalisation routines and must be done **only** in consultation with an instrument scientist.

D.1 Introduction

The maximum beam dimensions at the POLARIS sample position, defined by fixed collimation in the incident beam pipe, are 20mm wide x 40mm high. These are well suited to the majority of experiments, however under certain circumstances it may be desirable to reduce the beam size, for example when small samples are used or to reduce the background scattering from complex sample environment equipment, such as reaction vessels or pressure cells. For this purpose two sets of stepper motor driven collimators, or "jaws", have been built into the incident beamline and these are operated from two control units located in the POLARIS instrument cabin.

The front jaws are located in the chopper pit, ~8.5m from the moderator, and the back jaws are located in the POLARIS blockhouse, immediately in front of the sample tank, ~11.5m from the moderator. Each set of jaws has independently controlled horizontal and vertical motions, with the apertures centred on the beam centre-line. Potentiometers attached to the collimators allow the control units to determine each of the apertures by measuring a voltage and comparing it with a calibration table.

The construction of the jaws limits the maximum change in the aperture to 50mm and this has important consequences for the 8.5m jaws because these need to have a horizontal aperture of 55.0mm in order to collimate the incident beam properly. To allow for this a 5.0mm zero point offset has been built into the jaws' construction. This means that setting a beam height of 0.0mm on the control unit results in a vertical aperture of 5.0mm and setting a beam height of 50.0mm results in a vertical aperture of 55.0mm.

D.2 The JAWS Program

In order to calculate the optimum horizontal and vertical apertures of the front and back jaws for a given beam size at the sample position a FORTRAN program called JAWS has been written. The program is run by typing the command JAWS.

```
$ JAWS<cr>
```

```
_____JAWS_____
```

```
ENTER WIDTH IN MM:
```

```
10<cr>
```

```
ENTER HEIGHT IN MM:
```

```
25<cr>
```

 SET 8.5m JAWS TO: width=34.4mm, height= 39.2mm
 SET 11.5m JAWS TO: width=13.5mm, height= 27.7mm

The program compensates for the zero point offset built into the vertical aperture in the 8.5m jaws, discussed above, so that the values provided are those entered to the control units, as described in the following sections.

D.3 Control Unit Operation

The control unit for each set of jaws has a panel containing a keypad and LCD screen, which are used for automatic setting of the jaws' apertures, and a set of buttons which can be used in conjunction with the keypad and LCD screen for manual setting of the apertures.

To display the main menu on the LCD screen press the button labelled "MENU RECALL", to the left of the six function keys F1,F2,...,F6 situated below the screen. The display will read:

JOG MODE		POSITION MODE		CALIBRATION MODE		[screen A]
↑↑ F1	↑↑ F2	↑↑ F3	↑↑ F4	↑↑ F5	↑↑ F6	

D.3.1 Position Mode

In position mode the required jaws aperture is entered at the keypad. Press the F3 function key to select position mode. The display now reads:

SET BEAM WIDTH		SET BEAM HEIGHT		MOVE AXIS		[screen B]
↑↑ F1	↑↑ F2	↑↑ F3	↑↑ F4	↑↑ F5	↑↑ F6	

Press either F1 or F3 to select setting of horizontal or vertical aperture respectively (example given here for setting of beam width, F1). The display now reads:

CURRENT BEAM WIDTH IS 49.8 MM						[screen C]
ENTER NEW BEAM WIDTH IN MM (0 TO 50 MM)						
↑↑ F1	↑↑ F2	↑↑ F3	↑↑ F4	↑↑ F5	↑↑ F6	

Using the numeric keypad enter the jaws aperture desired, in millimetres. The value entered is echoed to the top left corner of the screen, and mistakes can be cleared by pressing "C/E" on the keypad. Once finished, press "ENTER". The display now reads:

						[screen D]
EXCEPT 34.4		MM		OR PUSH		
		YES	NO	MENU BUTTON		
↑	↑	↑	↑	↑	↑	
F1	F2	F3	F4	F5	F6	

To accept the set point press F3, otherwise press F4 to return to screen [C], above, and correct the mistake (or push the "MENU RECALL" button to abort the procedure and return to the main menu). The display now reads:

SET BEAM WIDTH		SET BEAM HEIGHT		MOVE AXIS		[screen B]
↑	↑	↑	↑	↑	↑	
F1	F2	F3	F4	F5	F6	

If required, press F3 to select the beam height option and follow the same procedure as for the beam width.

Once the required beam height and width have been entered press F6. The green "READY" light at the top, right hand corner of the control unit will now illuminate. The display now reads:

TO INITIATE MOTION PUSH START BUTTON						[screen E]
↑	↑	↑	↑	↑	↑	
F1	F2	F3	F4	F5	F6	

To operate the stepper motors which move the jaws press the green "START" button on the bottom row of buttons situated to the right of the screen and keypad unit. The green "READY" light will remain illuminated until the operation of moving the jaws has completed, at which point it goes out and the display then returns to screen [B], above.

D.3.2 Jog Mode

An alternative method of jaws operation involves "jogging" the stepper motors using the two sets of "open" and "close" buttons situated to the right of the screen and

keypad unit to move the jaws by arbitrary amounts. More importantly, however, this mode of operation can be used to determine the current jaws apertures, as follows. Press "MENU RECALL", if required, to return to screen [A] and then press F1 to select jog mode. The display will read:

JOG MODE			PUSH F5 TO		
EXIT			DISPLAY POSITIONS		
↑	↑	↑	↑	↑	↑
F1	F2	F3	F4	F5	F6

Press F5 to display the current jaws settings. The display now reads:

WIDTH IS 024.8 MM			F5		F6
HEIGHT IS 049.8 MM			UPDATE		EXIT
↑	↑	↑	↑	↑	↑
F1	F2	F3	F4	F5	F6

If it is suspected that the display is reading incorrectly, pressing F5 will cause the control unit to measure the potentiometer voltage and use this to determine the current jaws apertures. The screen will then display the updated jaws positions. To exit from jog mode and return to screen [A], above, press either F6 or "MENU RECALL".