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User Guide for the Polaris Powder Diffractometer at ISIS

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POWDER DIFFRACTION AT ISIS

USER GUIDE FOR THE POLARIS POWDER DIFFRACTOMETER AT ISIS

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Version 2.1

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Preface To Version 2.1.

Since version 1.0 of the Polaris User Guide was written, in 1989, Polaris has undergone a series of modifications, each of which resulted in a significant improvement in instrument performance. This has meant that the original version 1.0 of the User Guide, as well as going out of print, has also become rather obsolete. The improvements to Polaris over the past three years commenced with the building of extended back scattering, 90 degrees and low angle detector banks, followed by evacuation of the entire Polaris beamline, and were completed with the installation of a new sample tank and 'get lost' pipe. The detectors are arranged to give approximately matched count rates in the three main detector banks, with the two "Brick" detectors, previously situated at $2\theta=90°$, relocated at very low angles to retain access to the high d-spacings (~20Å) of the old low angle detectors, albeit with a lower count rate than the other three detector banks.

Thus, it would appear to be timely to publish a revised User Guide documenting the current state of the Polaris diffractometer. This report, the result of the rewrite, is based very much on the original version 1.0 in terms of style and content. Minor changes have been made in the text where appropriate, new sections have been included and some obsolete sections deleted, but the layout has remained very much the same.

October 1994.

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1. INTRODUCTION

1.1 Overview

The POLARIS instrument at ISIS is scheduled as a high intensity, medium resolution powder diffractometer. The high incident neutron flux on POLARIS, combined with a large detector coverage, enables experiments to be performed with comparatively short counting times or where very little sample is available. This ability has been exploited during kinetic experiments of, for example, chemical reactions or phase transitions, where data are collected whilst a furnace or cryostat is heating or cooling the sample, and in diffraction from very small sample volumes, down to ~5mm^3. The provision of detector banks at 2θ~90° is particularly important for studies of powder samples at high pressures. The 90° scattering geometry has significant advantages for these experiments, as suitable collimation of the incident and diffracted beams eliminates contamination of the sample diffraction pattern with signals from the surrounding pressure transmitting apparatus.

This guide is intended to give a short description of the POLARIS diffractometer and to provide the basic information required to perform a powder diffraction experiment. To keep this guide as brief as possible the text contains several references to appropriate manuals, where more detailed information may be found.

1.2 Instrument Design

The POLARIS diffractometer is situated on beamline S1 at ISIS. It receives a polychromatic, 'white', beam of neutrons from a 295K H_2O moderator, poisoned with Gd at a depth of 2.0cm. The overall moderator to sample distance is 12.0m and the neutron beam dimensions at the POLARIS sample position are approximately 40mm high x 25mm wide. The incident and transmitted neutron flux is monitored by two low efficiency glass scintillator detectors, situated ~2m before the sample position and ~2.5m after it. During data collection, the sample tank is normally evacuated by a rotary pump, typically to a nominal 10^{-1}mbar. A turbo pump is also available which will reach the 10^{-4}mbar required to operate a closed cycle refrigerator. POLARIS has a total of 302 ^{3}He gas filled and ZnS scintillator detectors arranged in four separate banks: two at low angles, one at 2θ~90°, and one at back scattering angles. These are referred to as the A, B, E and C banks respectively. The details of these detector banks are given below, with the layout of the instrument shown schematically in figure 1.

Position:	Low angle	Low angle	Back scattering	90 degrees
type:	(^{3}He)	(ZnS)	(^{3}He)	(ZnS)
name:	A	B	C	E
no. of detectors:	80	40	38	144
2θ range:	28°<2θ<42°	13°<2θ<15°	135°<2θ<160°	83°<2θ<97°
Ω (ster):	0.046	0.003	0.16	0.99
Δd/d:	~1x10^{-2}	~2x10^{-2}	~5x10^{-3}	~7x10^{-3}
L_2 (m):	1.72-2.65	~2.2	0.65-1.10	~0.45
d-range (Å):	0.5-8.15	0.5-21.6	0.2-3.2	0.2-4.2
q-range (Å^{-1}):	0.8-12.6	0.3-12.6	2.0-31.4	1.5-31.4

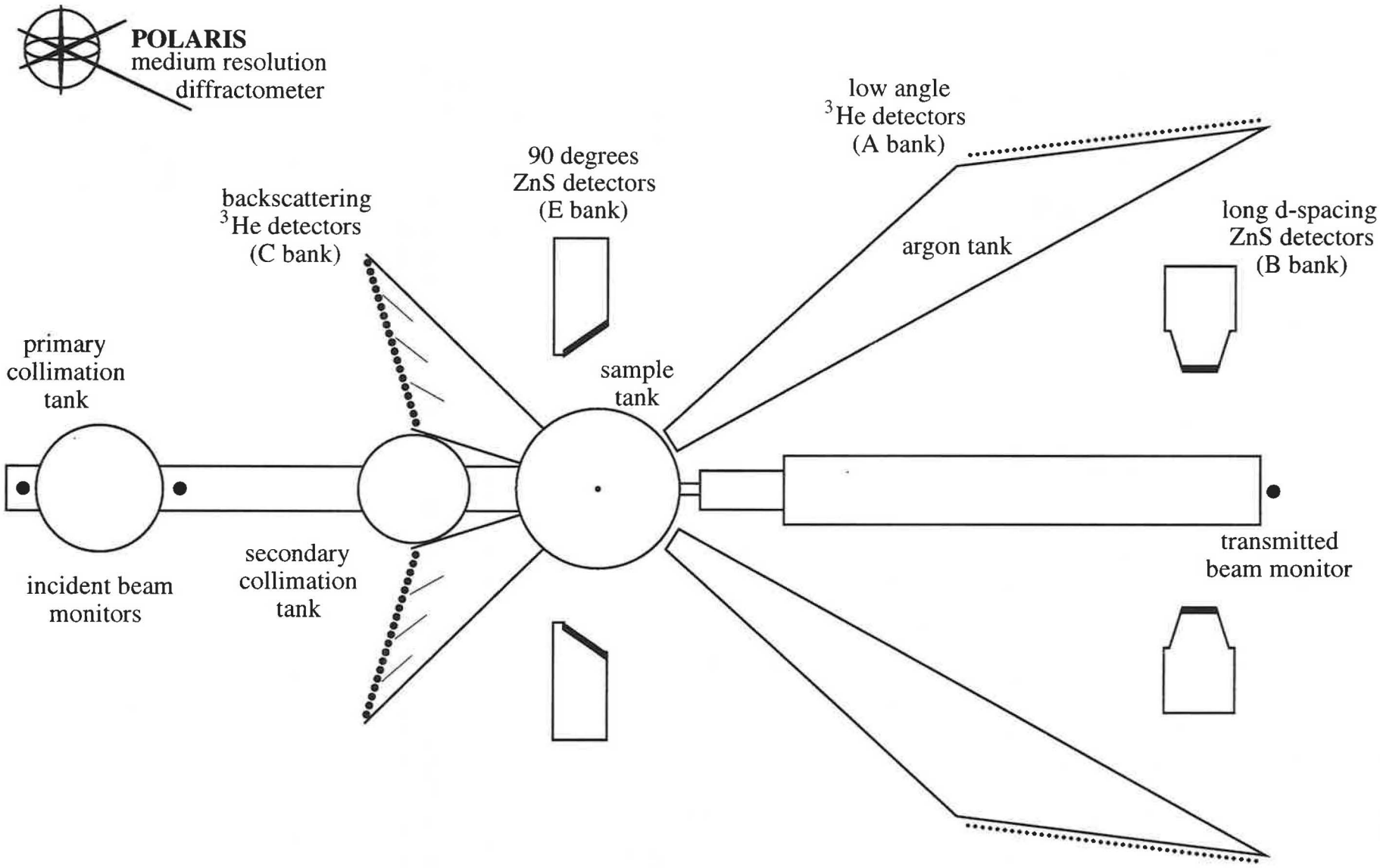


Figure 1. Schematic Layout Of The Polaris Diffractometer (June 1994).

For experiments where only small volumes of sample are available, for example high pressure studies, additional collimation located in the primary and secondary collimation tanks, figure 1, can be introduced to reduce the dimensions of the neutron beam at the sample position to 10mm high x 7mm wide. Moreover, the position of the collimator in the secondary collimation tank can be adjusted both horizontally and vertically, using stepper motors, allowing the neutron beam to be accurately aligned on the sample. More detailed information on the use of this collimation, when required, can be provided by the instrument scientists.

1.3 Principle of the Detector Arrangement

For a particular Bragg reflection,

$$2d\sin\theta = n\lambda = nht/(mL) \qquad (1)$$

where 2θ is the scattering angle, d is the interplanar spacing, λ is the neutron wavelength, t is the total time of flight, h is Planck's constant, m is the neutron mass and L the total flight path from moderator to sample to detector. From equation (1) it follows that the resolution, $\Delta d/d$, is:

$$R(d) = \Delta d/d = [\Delta\theta^2\cot^2\theta+\Delta t^2/t^2+\Delta L^2/L^2]^{1/2} \qquad (2)$$

It is clear from equation (2) 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 $2\theta=0°$, while it is zero at $2\theta=180°$.

POLARIS is designed to give approximately constant resolution in each of the detector elements within each bank. In the 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. At large scattering angles the contribution of the $\Delta\theta\cot\theta$ angular term is small and the C bank resolution is dominated by the time uncertainties. The back scattering detectors, arranged in a straight line inclined to the scattered neutron beam and all intersecting approximately the same fraction of the Debye-Scherrer cones, 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.

In the particular case of the A bank, the d-spacing and q values given in the above table refer to the ranges covered by the whole bank. An increased d-spacing range of up to ~11.1Å (corresponding to a minimum q of $0.5Å^{-1}$) can be accessed by considering only the detectors at lowest angles (~28°2θ) 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 counting statistics.

1.4 Data Acquisition

The POLARIS instrument and data acquisition are controlled by a MicroVAX 3200, known as the Front End Monitor (FEM), situated in the instrument cabin. The current instrument settings are contained in a file called the Current Run Parameter Table (CRPT) and whilst a run is in progress the diffraction data are temporarily stored in the Data Acquisition Electronics (DAE). At the end of the run the contents of the CRPT and the DAE are automatically written to a data file on the FEM, known as a RAW file. Shortly afterwards, this file is automatically archived onto optical disk. The FEM itself has a limited amount of disk storage space, so data files are usually deleted from the instrument disk at the end of each cycle of ISIS running. The files can, of course, be restored from optical disk so that analysis of the data can be performed.

ISIS users are given, on request, a user account on the central VAX 4500/4300 computer system, known as the 'HUB'. The HUB and the instrument FEM's are connected via an ethernet link to form a 'cluster'. One consequence of the cluster 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. However, the CRPT and DAE can only be accessed from the FEM.

2. PERFORMING AN EXPERIMENT ON POLARIS

2.1 Sample Safety Assessment

All users complete a 'Sample Record Sheet' as part of their application for beam time, the details on which are used to carry out a sample safety assessment. This will give comments (if any) from the ISIS Safety Section concerning possible chemical or radiological hazards associated with the sample; any recommendations 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 building R55 (the ISIS experimental hall), after which it should be displayed in the pocket on the door of the POLARIS blockhouse for the duration of the experiment.

2.2 Loading a Sample

For room temperature measurements a number of cylindrical V cans are available, with diameters between 5mm and 12mm. For use with air sensitive samples, these can be fitted with Cu 'O' ring seals for high temperature work, indium seals for low temperature work, or teflon 'O' rings for room temperature measurements. The sample cans are attached to a centre stick which is lowered into the sample tank. The distance between the centre of the POLARIS incident beam and the aluminium plate on the centre stick is 314mm.

After removal from the neutron beam, all samples which have become activated due to exposure to the neutron beam must be stored in a controlled area. For the POLARIS instrument, this controlled area is either the instrument blockhouse itself or the POLARIS active samples cupboard, situated at the back of the eVS instrument cabin. It should be noted at this point that the vanadium sample cans themselves become activated, emitting β radiation for approximately 1 hour after

removal from the POLARIS beamline. At the end of an experiment all samples which have been irradiated in the neutron beam, before being removed from R55, must be monitored by the ISIS Health Physics Group (x6696).

The POLARIS sample tank will accept all standard sample environment apparatus (cryostats, closed cycle refrigerators, furnaces, goniometers, pressure cells, etc.). Details of the sample loading procedure for each of these is beyond the scope of this guide and are available from the Sample Environment Group staff and instrument scientists. The CAMAC sample environment control system provides a means of monitoring and controlling sample environment parameters (e.g. temperature, etc.). Information on the CAMAC system is given in section 5.2 of the PuNCH user manual, a copy of which is kept in the POLARIS instrument cabin and which can also be obtained from the Computer Support office, room 1.38, building R3.

2.3 Sample Tank Evacuation

Air scattering of the neutron beam within the sample tank is reduced by evacuating the tank during data collection. An air admittance valve is situated to the right of the steps leading up to the POLARIS sample tank, below the argon tank. To evacuate the sample tank this valve should be fully closed (turned clockwise) and the rotary pump started by switching on the mains switch marked 'ROTARY PUMP' above the air admittance valve. The turbo pump need not be used unless pressures of $<10^{-3}$mbar are required, normally only when using a closed cycle refrigerator. To start the turbo pump the right hand switch on the control unit outside the blockhouse door should be depressed. The state of the sample tank vacuum can be monitored using the Pirani and Penning pressure gauges located on this control unit.

To let the sample tank up to atmospheric pressure the rotary and turbo pumps should both be stopped, where applicable, by switching off the appropriate switches. Air is allowed into the sample chamber by opening the air admittance valve. ***If the turbo pump has been in use it is extremely important that air is admitted into the sample tank slowly until the turbo has stopped spinning (the spinning of the turbo pump can be recognised as a faint 'whining' sound, which lowers in pitch as the rotation speed of the turbo pump blades decreases).***

Users are reminded that after removing irradiated samples from the neutron beam, they must follow the safety regulations concerning monitoring of induced β and γ activity, transferral of powders from cans, etc. as indicated on the sample safety assessment sheet.

2.4 Opening the Beamline Shutter

Before attempting to open the POLARIS beamline shutter the instrument blockhouse must be cleared of personnel and then locked. Having cleared the blockhouse a search button, located on the wall above the back scattering detectors, must be pressed. This allows approximately 30 seconds to leave the blockhouse and close the entrance door, during which time a 'buzzer' will sound. Once the blockhouse door is closed and bolted, the interlock key (labelled an 'S' key) can be removed from the lock and inserted into the vacant position in the uppermost key rack (item A, figure 2), to the left of the blockhouse door.

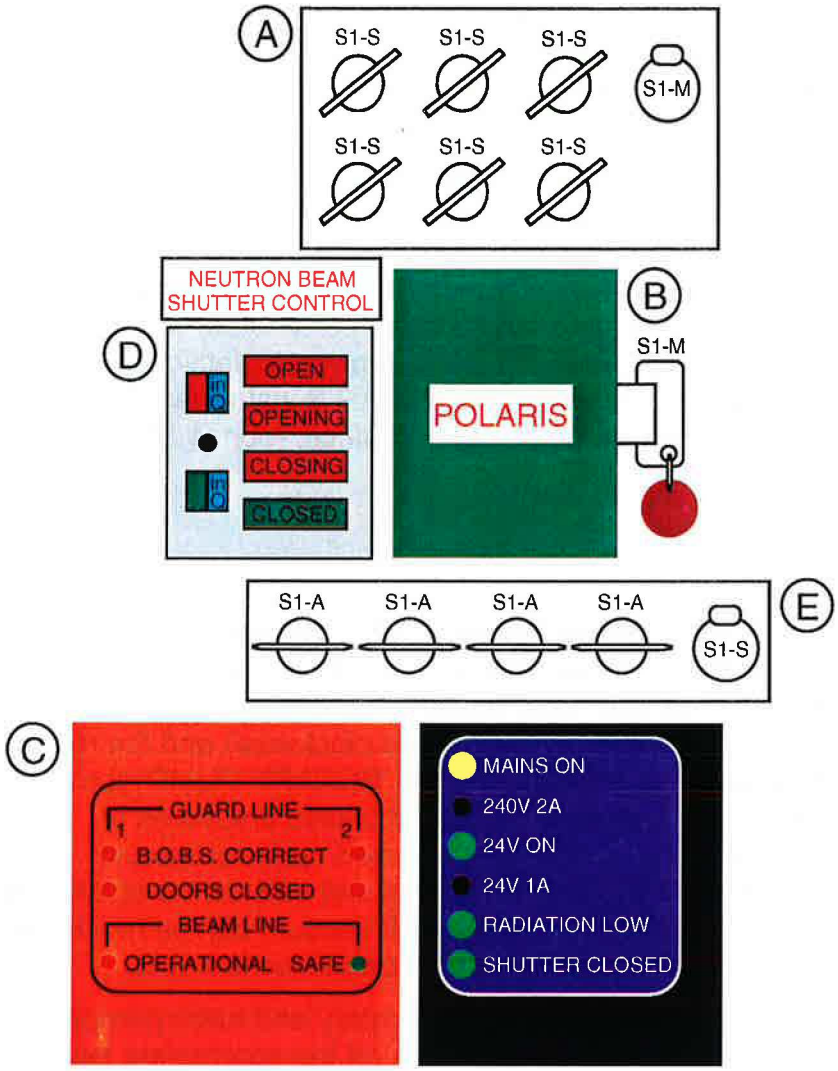


Figure 2. The Polaris beamline shutter control and interlock system

When all six 'S' keys are present in this key rack, the master ('M') key (with the dark red tag) may be removed and inserted into the green shutter interlock box (item B, figure 2), where it is turned clockwise through 90° so that it is vertical. It is not possible to open the shutter unless the master key is correctly positioned in this box. Before attempting to open the shutter, users must first check that the upper four red LED's on the orange interlock indicator box (item C) are lit, indicating that all the interlocks and micro switches are closed. Any attempt to open the shutter without all four LED's being lit will immediately shut down ISIS itself.

The beamline shutter is then opened by pressing the 'OPEN' button on the shutter control unit (item D), situated to the left of the green interlock box. The operation of opening the shutter takes approximately one minute. When the shutter is open the master key cannot be removed from the shutter interlock box and there is no access to the interlocked areas.

The sequence for closing the shutter is the reverse, with the 'CLOSE' button on the shutter control unit being pressed instead. The current position of the shutter is shown by one of the four "open","opening","closing","closed" indicators on this unit.

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 (x6789).

The interlock keys in the lower rack (item E, figure 2) are used to access the interlocked areas via other routes, for example when changing a sample in a cryostat through the port in the blockhouse top hatch. To release these 'A' keys it is necessary first to take an 'S' key from the upper rack and insert it into the vacant (right hand) position in the lower rack.

2.5 The POLARIS FEM and Workstation

The POLARIS Front End Monitor (FEM) controls both instrument and sample environment parameters and data collection. The FEM, situated in the POLARIS cabin, is a MicroVAX 3200 and has two terminals attached directly to it, both located in the cabin. Its node name within the ISIS VAX cluster is POL.

The right hand terminal, the 'workstation', running the 'Motif' windows environment, generally is used to control the instrument, inspect the experimental data, perform preliminary analysis of the data, etc. Commands to start and stop data collection and change the instrument configuration should normally be typed to the "Supervisor" window on the workstation. The left hand PERICOM terminal in the cabin provides additional access to the POL FEM.

The workstation offers the possibility of generating several software 'windows' on the terminal screen, each effectively logged onto the FEM independently and performing a different task. Full instructions on the use of the features available are given in the workstation manual, a copy of which is available in the instrument cabin. To open a new terminal session window on the workstation, use the left hand button on the mouse to pull down the 'Applications' menu on the 'Session Manager' window, then move the pointer arrow over the 'DECTerm' option in this menu. 'Clicking' the left hand button on the mouse will initiate the generation of the software window, which will automatically be logged on to the POL FEM as username POL.

Although it is possible to have different tasks running in different windows, the keyboard can only be connected to one window at time. The window with the dark grey frame is the one to which input from the keyboard is directed and anything typed at the keyboard will appear in this window. To make a window 'active' simply move the mouse so that the pointer is positioned somewhere on the required window and then press the left hand button on the mouse. If the window is partially hidden behind other windows it will automatically be brought to the front.

The 'Menu' icon at the top left hand corner of each window offers various options concerning the window itself. These are accessed by moving the pointer to the menu icon of the chosen window and then pressing the left hand button on the mouse to pull down the menu. To execute the required option move the mouse to position the pointer over the option and 'click' the left hand button on the mouse. The 'Delete' option will erase the window from the screen and automatically log off the associated process.

Two other options, "shuffle up" and "shuffle down", in a menu accessed by pressing the left hand mouse button while the pointer is in a background region of the screen,

allow windows to be pushed behind others or pulled to the front without changing the window to which keyboard input is directed.

To change the shape of the window move the pointer to one of the corner or side bars of the window, in which case the pointer changes to an arrow attached to a corner or edge, then press and hold the left hand button on the mouse. The window outline appears as a contrasting colour and becomes a stretchy box. The pointer can be moved until the box is of the required size and shape and then the button released.

If a window is not required for a short period, it may be shrunk to an 'icon'. This is done either by selecting the 'minimise' option from the menu or by 'clicking' the left hand mouse button on the square containing the small 'dot' at the top right hand corner of the window. The icon appears as a small box containing a picture of a terminal and keyboard, usually at the lower left hand corner of the screen. The window can be restored to its former size either by double clicking on the icon itself or by moving the pointer onto the icon, pressing the left hand button on the mouse and selecting the 'Restore' option.

3. DATA COLLECTION

3.1 The Instrument Control Program (ICP)

Data collection on ISIS instruments is controlled by the Instrument Control Program (ICP). This program is used to start and stop data collection, but it also allows data collection to be suspended temporarily while, for example, the helium flow in a cryostat is adjusted - an operation which on POLARIS may require entry into the blockhouse and for which the beamline shutter must be closed. Also, the ICP can be set (through the CAMAC sample environment control program) to suspend data collection automatically if, for example, the temperature of a sample in a furnace or cryostat drifts away from user defined limits. The available ICP commands, which are typed to the POL FEM, and their functions are:

BEGIN	Clears DAE memory (section 3.2) Sets parameters in DAE to those specified by CRPT (section 3.3) Instructs DAE to start data collection Sets DAE state to RUNNING
END	Stops data collection by DAE Copies contents of DAE memory and CRPT to file 'POLnnnnn.RAW' Increments the run number 'nnnnn' Sets DAE state to SETUP
PAUSE	Suspends data collection by DAE Sets DAE state to PAUSED
RESUME	Resumes data collection by DAE Sets DAE state to RUNNING
ABORT	Stops data collection by DAE Sets DAE state to SETUP
CHANGE	Enables the contents of the CRPT to be modified

3.2 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 number of spectra, each spectrum containing a histogram of neutron counts versus time-of-flight from a single detector. At the end of the run the contents of the DAE are automatically copied to a file on the FEM named POLnnnnn.RAW, where 'nnnnn' is a five figure run number which is incremented automatically at the end of each run. Shortly after creation, this RAW file is also archived onto optical disk. The DAE has four possible modes:

SETUP	Data are not currently being collected. The instrument parameters may be changed if required before starting a new run (section 3.3).
RUNNING	Data are currently being collected and stored in the DAE.
PAUSED	Data collection is temporarily suspended by the user.
WAITING	Data collection is temporarily suspended by the FEM. This may occur, for example, if a furnace or cryostat drifts outside user defined limits in the CAMAC sample environment control system, or before it reaches a requested temperature.

The current DAE mode and run status are displayed on the dashboard (section 3.4).

3.3 The CRPT

The Current Run Parameter Table (CRPT) contains additional information on the current run, and is copied, along with the contents of the DAE, to the RAW file on the FEM at the end of the run. It contains information on the current experiment (sample title, users' names, etc.) plus details of the instrument configuration and settings (the times-of-flight of the time channel boundaries, the detector used as the monitor spectrum, etc.).

At the beginning of an experiment it may be necessary to change some of the contents of the CRPT. This should only be done in consultation with an instrument scientist. The relevant command to type is:

$ CHANGE<cr>

at the "Supervisor" window when the DAE is in setup mode. The screen will then display the first page of the present 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. To move from one field to another the 'up' and 'down' cursor arrow keys are used. When the field prompts with 'toggle data type' the '.' key on the right hand keypad should be pressed until the field displays the required option. All other fields are altered by typing the appropriate numbers or characters into the field, the 'left' and 'right' cursor arrow keys and the 'delete' key allow corrections to be made.

To exit from the editor and overwrite the CRPT with the new values the user should press the 'F1' key on the right hand keypad, then press the key 'E'. If you wish to quit and leave the CRPT as it was originally then press 'F1' followed by 'Q'.

During the course of an experiment some simple alterations to the CRPT can be made without using the DEFT editor. These can be typed at the keyboard or given from a command file, regardless of the DAE state. For example:

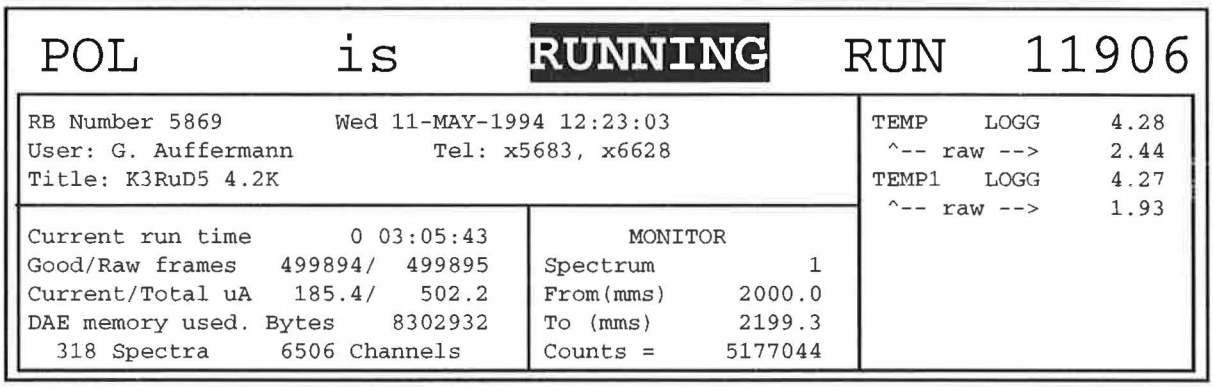
$ CHANGE TITLE C2(CN)4_at_18kbar_293K<cr>

Note that the title must be a continuous string of characters, hence the "_" symbol connecting different words. Also, note that all characters are 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 """C2(CN)4 at 18kbar 293K"""<cr>

3.4 The Dashboard



The POLARIS instrument dashboard, above, shows details of the current instrument configuration and experiment and will normally be displayed in the "Supervisor" window on the workstation. If the dashboard is not present it can be displayed on any terminal logged onto the POL FEM (as username POL) by typing:

$ STATUS ON<cr>

To turn the dashboard display off simply type:

$ STATUS OFF<cr>

At the top of the dashboard display the current DAE state (RUNNING, SETUP, etc.) 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 displays information on any sample environment parameters being monitored by CAMAC.

4. DATA ASSESSMENT

4.1 The PuNCH system

The PuNCH VAX cluster at ISIS provides links between the FEM's and the HUB (a VAX 4500/4300 system, cluster node name ISISE) and allows these computers to be accessed from the terminals situated in building R3. In addition, the HUB may also be accessed from outside RAL via JANET, Internet and DECnet.

POLARIS RAW data files on the FEM are named 'POLnnnnn.RAW', where 'nnnnn' is a five figure run number. As mentioned in sections 1.4 and 3.2, this data file is archived automatically by copying it to optical disk. The number of data files which can be stored on the FEM at any one time is limited by the amount of disk space

available on the computer. For this reason old RAW files are deleted periodically from the FEM disk, usually at the end of each ISIS experimental cycle. However, these data files can always be restored from the optical disk for subsequent data analysis, as described in section 4.6.

4.2 Accessing POLARIS data

During the course of an experiment it is desirable to be able to inspect the diffraction pattern whilst a run is still in progress. The data from the current run are stored in the POLARIS DAE which can ONLY be read by the POL FEM and thus can only be inspected from sessions logged on to POL. This can be done from the workstation or PERICOM terminal in the POLARIS cabin, or by using one of the terminals elsewhere (such as those in R3 or the DAC), as described below.

First the 'RETURN' key on the terminal keyboard should be pressed several times, until the prompt 'Vista >' (or similar) appears, then type:

Vista > **CONNECT POL<cr>** to connect to the POL FEM.

After pressing <cr> the terminal will display a bold 'POL' header and then prompt for a username. Now type:

Username: **POL<cr>** followed by
Password: (the password is not echoed to the screen)

Users can obtain the current POL password from one of the instrument scientists. Note that the PERICOM terminal in the POLARIS cabin is connected directly to the FEM, thus there is no requirement to connect through the Vista terminal server: one need only give a username and password.

The procedures for routine inspection of POLARIS data will be described in the following sections (4.3 and 4.4). Computationally more intensive procedures, such as data analysis and structure refinement, should not be carried out on the instrument FEM's, but should be performed on more powerful machines, such as the HUB (cluster node name ISISE). For this users will require a user account on the HUB, which can be arranged by one of the instrument scientists.

The user account will be set up so that users can access POLARIS data stored on the POL FEM, run the POLARIS data normalisation routines, retrieve data archived on the optical disk, etc. Users attempting to log onto the POL FEM using their own account are liable to be denied access, to prevent the FEM from becoming overloaded. Those users who have performed previous experiments on other ISIS instruments may already have an account set up with default settings to access data from the other instrument. If so, an instrument scientist can arrange for this to be amended.

To log on to the HUB computer from terminals in the Data Assessment Centre (DAC) in R55 or those in R3 the procedure described above for connecting to the POL FEM should be followed, except that:

Vista > **CONNECT ISISE<cr>**

should be typed instead of CONNECT POL. The user should then enter their own username and password.

Connections to the HUB can also be made through any of the sessions running on the POL FEM using the SET HOST command:

$ SET HOST ISISE<cr>

Again, the user should then enter their own username and password.

In order to ensure that all the commands necessary to analyse POLARIS data are set up when logging in, users should include in their LOGIN.COM file the command line:

$ @POL$DUA0:[POLMGR.COMMAND]LOGIN.COM

This will define the appropriate symbols and logical names used in data analysis, including the GENIEINIT initialisation command file which instructs GENIE to examine data from POLARIS, and the POL_COMMAND, POL_DATA and POL_CALIB_SILICON logical names referred to later in this manual.

4.3 GENIE

GENIE is a data display and manipulation program which runs on the HUB and the instrument FEM's. Full details of the commands available within GENIE are given in the GENIE manual and Appendix 2 of the PuNCH manual. Only those commands necessary to perform preliminary assessment of the data will be described here. To run GENIE simply type:

$ GENIE<cr>

The terminal will display the GENIE header at the top of the screen, print several lines of text and then display the prompt ">>". On the workstation a "GKS" window will be created (as an icon) where graphics created will be displayed. GENIE allows the user to inspect and modify time-of-flight spectra from individual detectors, sum these together, convert from time-of-flight to lattice d-spacing, etc. The data from run number 2189, for example, are accessed by typing:

>> ASSIGN 2189<cr>

When logged onto the POL FEM it is also possible to type:

>> ASSIGN DAE<cr>

to examine the data currently being collected and stored in the Data Acquisition Electronics (DAE).

Diffraction data on POLARIS are collected as histograms of neutron counts versus time-of-flight in which the time-of-flight axes are split into a large number of 'bins' bounded by time channel boundaries (TCB's), the positions of which are defined in the CRPT. Each of the histograms, which corresponds to the data collected in a single detector, are defined in GENIE as spectra, S'n', where n currently ranges from 1 to 306. The correspondence between the spectrum number 'n' and the position of the detector on POLARIS is determined by the wiring of the electronics and by data contained in the CRPT. The current parameters for each detector are listed in Appendix A.

To display the data contained in spectrum 33, for example, simply type:

>> DISPLAY S33<cr>

On the workstation this will cause the GKS window to be opened and the spectrum displayed. On a PERICOM terminal the screen will toggle to graphics mode before displaying the graphic. To exit from the graphics display on a PERICOM terminal and return to the >> prompt, hit 'RETURN'.

Also defined in GENIE are a number of workspaces, called W'n', which may be used to store spectra or, more commonly, to store the data resulting from some arithmetic operation involving several spectra. For example, if you wish to display the sum of the contents of spectra 17 and 35 divided by spectrum 1, the commands would be:

>> **W1 = (S17 + S35)/S1<cr>** followed by
>> **DISPLAY W1<cr>**

Here, workspace W1 has been used to store the calculated spectrum before displaying it. 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. The user is referred to the manuals mentioned above for details of these. To exit from GENIE type:

>> **EXIT<cr>**

4.4 Focusing the Data

The POLARIS detector banks are designed so that all the individual detectors within a particular bank have approximately the same resolution function. This allows the many detectors within each bank to be summed (or focused) into only four spectra (corresponding to the A, B, C and E detector banks), with a significant improvement in the counting statistics.

Since the detectors within each bank have differing values of neutron flight path ($L = L_1+L_2$) and scattering angle (2θ) they cannot simply be summed directly. Four GENIE command files (AFOCUS, BFOCUS, CFOCUS and EFOCUS) perform the required summations, using values for L and 2θ obtained from a calibration of the POLARIS instrument using an NBS standard silicon sample (SRM 640b).

These command files sum together the detectors within each bank by converting the spectra collected by the individual detectors from neutron time-of-flight to lattice d-spacing before adding them together, dividing by the incident monitor counts to normalise on counting time and converting back to time-of-flight. The summed data then have an instrumental background spectrum subtracted and are divided by the spectrum collected from a vanadium rod. The vanadium spectrum, which is corrected for attenuation and multiple scattering effects, normalises the data to the incident neutron flux distribution and also corrects for the variation in detector efficiency with neutron wavelength.

Vanadium and background normalisation spectra are recorded at the start of each cycle for use in all experiments. In order to focus POLARIS data, there are two different sets of command files depending upon whether the data are from the current cycle or a previous cycle. For old data the number of the silicon standard calibration run appropriate to the ISIS run cycle during which the data were collected must be known by the user. The command files are then run in GENIE, as follows.

4.4.1 Data from the Current ISIS Cycle

The vanadium and background data sets collected at the start of the current ISIS cycle are used to normalise the data for that cycle. The commands used to focus a single bank of detectors are:

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

after which the user is prompted first for the run number and then for the GENIE workspace into which the final spectrum will be placed. For example, to focus the data from run number 11916 into workspace 1.

>> Enter run number **11916<cr>**
>> Enter workspace number (not 15) **1<cr>**

To view the data currently being collected (ONLY possible when logged onto the FEM) type 'DAE' instead of a run number. Note that workspace number 15 is used by the focusing command files and cannot be selected to store the focused data set. The focused workspace may be viewed as described in the previous section, i.e.:

>> **DISPLAY W1**

With the large numbers of detectors present in the four POLARIS detector banks, the above focusing routines may take some time to complete, which may not be convenient for some experiments. An alternative routine is available to sum rapidly the data from the individual detectors, with the normalisation still using GENIE routines. This routine is accessed with the "FASTFOC" command in GENIE. This requires similar information to the AFOCUS, etc. routines described above, but in addition also prompts for the detector bank which is to be focused.

>> **FASTFOC**
>> Enter run no. of data : **11916<cr>** (or **DAE<cr>**)
>> Give detector bank (A,B,C or E) : **A<cr>**
>> Give workspace (not 15) : **1<cr>**

It should be noted, however, that the fast focusing routine does make some approximations during the summation and normalisation such that the resulting normalised spectrum may not be rigorously correct. It is advised, therefore, that the FASTFOC routine be used to assess qualitatively the data, but to use the AFOCUS, etc. commands when creating data files for profile refinement purposes.

4.4.2 Data from Previous ISIS Cycles

The procedure is similar to that given above except that 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, i.e.:

>> **@POL_COMMAND:AFnnnnn.COM**
(or **BFnnnnn**, **CFnnnnn** or **EFnnnnn**)

where 'nnnnn' is the four or five figure number of the silicon calibration run collected at the start of the cycle the old data were collected. A listing of available 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.

The normalised, focused data can then be written to a GENIE format data file by using the GENIE command WRITE, e.g.:

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

In this case, the contents of workspace 1 are written to the file AL2O3.NOR. The "WRITE/CLOSE" command is necessary to ensure that all the data are written and the file closed so that GENIE can subsequently read the data. To read the data back into a workspace, type:

>> **READ W2 Al2O3.NOR<cr>**

To write the contents of a workspace to an ASCII file, for example for subsequent transport to another computing installation, the GENIE command SHOW DATA may be used:

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

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

Once a spectrum has been focused it is possible to have hard copy output sent to one of the laser printers. To do this type:

>> **HC<cr>**

(On a PERICOM terminal it will be necessary first to leave the graphics screen by hitting <cr> before typing the HC command.)

This creates a postscript format file called "DEC_POSTSCRIPT.DAT" and submits it to the desired printer queue to be printed. The user is asked for their choice of printer from those situated in rooms 1.38 (No.0) and 2.26 (No.1) in building R3, or in the Data Acquisition Centre (No.2), the LAD cabin (No.3), or the SXD cabin (No.8) in R55.

If an alternative graphics format is required (e.g. HPGL, for inclusion into Word documents), this can be specified using the command:

>> **SET HARD device<cr>**

where 'device' is the code word appropriate to the desired format. A list of available device code words recognised by GENIE can be obtained through ISIS Computer Support. With non-postscript devices it is then necessary to type

>> **KEEP/HARD**

in order to save the graphic to the DEC_device.DAT file, which can then be printed, incorporated into documents, transferred to remote sites, etc. in the usual way.

4.5 Attenuation Corrections

In order to run the routine which corrects a diffraction pattern for sample attenuation, the weight of sample in the sample can must be determined. It is also necessary to measure both the diameter and height of the sample in the can and look up the

atomic masses and absorption and scattering cross sections of the elements present in the sample. An example for alumina, Al_2O_3, is given.

Weight of can = 19.98 g
Weight of can + sample = 31.63g
Weight of sample = 31.63 - 19.98 = 11.65g
Diameter of can (d) = 0.952 cm
Radius of can (r) = d/2 = 0.476 cm
Height of sample (h) = 5.5 cm

Atom	mass (amu)	σ_s	σ_a
Al	27	1.506	0.231
O	16	4.234	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 2200 ms^{-1}. Both these values can be found in the tables on the wall of the POLARIS cabin.

First calculate the mass of one Al_2O_3 scattering unit in grams:
Mass = $(27x2+16x3)x1.67x10^{-24} = 1.703x10^{-22}g$

Next calculate the sample volume:
volume = $\pi r^2 h = 3.141x0.476^2x5.5 = 3.914cm^2$

Then calculate the number of scattering units/cm^3:
N/V = (weight of sample)/(mass of scattering unit x volume)
= $11.65/(1.703x10^{-22}$ x3.914) = $1.748x10^{22}cm^{-3}$

Finally, calculate the total scattering cross section per scattering unit:
σ_{sc} = 2x1.506+3x4.234 = 15.71 barns

and the absorption cross section per scattering unit:
σ_{ab} = 2x0.231+3x0.0002 = 0.4626 barns at a neutron velocity of 2200 ms^{-1}

Assuming that a focused, normalised spectrum from a POLARIS detector bank is in a GENIE workspace, e.g. C bank in W1, the attenuation correction program is run by typing:

>> **CORRECT<cr>**

The program prompts for workspace numbers,
Enter workspace containing uncorrected data: **1<cr>**
Enter workspace to contain corrected data: **2<cr>**

the radius of the can and the number of scattering units per cm^3,
Enter cylindrical radius (cm): **0.476<cr>**
Enter number density of scattering units (cm^{-3}): **1.748E22<cr>**

and finally the total scattering and absorption cross sections per scattering unit.
Enter scattering cross section (barns): **15.71<cr>**
Enter absorption cross section (barns @ 2200m/sec): **0.4626<cr>**

The corrected spectrum will be written to W2.

4.6 Retrieving Archived POLARIS Data

Owing to the shortage of disk space on the POL FEM, RAW data files are normally deleted from the instrument disk at the end of each cycle. Users may restore these files from the optical disk archive system to the scratch disk, provided that there is sufficient space available on the disk to accommodate it. This is achieved by typing:

```
$ 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
Enter run number for first file =>: 11917
Enter run number for last file =>: 11919

**************************************************************
A restore process has been spawned. The files should be restored
to SCRATCH$DISK:[POLMGR.RESTORE] within about 10 minutes. Do not
log out until the process is completed. To check your process
type: $ SHOW PROC/SUBPROC
**************************************************************

Subprocess user01_1 has completed
```

As the directory SCRATCH$DISK:[POLMGR.RESTORE] is included in the definition of the logical area POL_DATA, all restored files can be read by GENIE without any further user intervention. The restored files will usually return within 10 minutes, but be warned - files on the scratch disk have only a limited lifetime and will be deleted again after ~1 week.

4.7 Creating Data Files for Profile Refinement

Details of the time-of-flight profile refinement programs available at ISIS are given in the RAL report RAL-92-032, "Profile Analysis Of Neutron Powder Diffraction Data At ISIS", copies of which can be obtained through the instrument scientists. The program written for the refinement of Polaris data, TF14LS, requires two input data files. The first file contains the normalised neutron diffraction profile, in a suitable ASCII format, the second file contains information from which the refinement program constructs the calculated diffraction profile.

For POLARIS data, the file containing the normalised neutron diffraction profile is created by typing:

```
>> TF_FILE<cr>
```

when running GENIE. The user is prompted for the number of the workspace containing the normalised diffraction pattern, followed by a name for the data file to be created and the time-of-flight limits required for profile refinement. The data in the workspace are assumed to have been generated by one of the POLARIS focusing routines (AFOCUS, etc.).

The second input file to TF14LS requires the user to know the values of certain instrument parameters, such as total flight path, zero point and peak shape parameters, for the detector bank in question. These are determined by Rietveld refinement of data collected from an NBS standard reference silicon sample. The refined parameters are stored in the file POL_CALIB_SILICON:Xnnnnn.CCL, where 'X' is the detector bank (A, B, C or E) and 'nnnnn' is the run number. The number of the most recent silicon calibration run should be displayed near the POLARIS cabin, but can be obtained from an instrument scientist. Users should copy this file and edit it so that it contains the unit cell and structural parameters corresponding to their own sample.

When analysing old data sets the appropriate .CCL file should be determined in a similar same way to that described in section 4.4.2 for focusing data from old raw files. From a DIRECTORY of POL_CALIB_SILICON:X*.CCL (X=A, B, C or E), use the run number closest to but lower than the run number of your own data.

4.8 Miscellaneous POLARIS Routines

This section lists other POLARIS routines which are run by typing the relevant command. Useful GENIE commands are:

GEC

This can be used to fit a single peak in a spectrum. The functional form is a convolution of a Gaussian (which models angular terms in the resolution function) and an exponential decay term (which models the time of flight contribution to the resolution).

>> **GEC<cr>**
Enter xmin **2.3<cr>**
Enter xmax **2.6<cr>**

Alternatively 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.

GAUSS

This is identical to GEC except that a Gaussian function is fitted to the peak. In general GEC is to be preferred, although GAUSS can be used for fitting to B bank reflections, where the resolution function of the instrument is dominated by uncertainties in the scattering angle and, consequently, is well approximated by a Gaussian function.

NDET

This normalises a single detector spectrum to the incident beam monitor. It is used when a comparison of the counts in a single detector is required for two different runs.

>> **NDET<cr>**
Enter run number **2844<cr>**
Enter spectrum number **33<cr>**
Enter workspace number **1<cr>**

PLOT2 (Also available **PLOT3**, **PLOT4**)

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

>> **PLOT2<cr>**
Enter workspace number **1<cr>**
Enter lower x limit **1.0<cr>**
Enter upper x limit **2.0<cr>**
Enter lower y limit **0.0<cr>**
Enter upper y limit **4.0<cr>**
Binning in groups of? **1<cr>**
Enter second workspace number **2<cr>**
Enter lower y limit **0.0<cr>**
Enter upper y limit **4.0<cr>**

If <cr> is entered in response to any of these prompts, default values are taken. (After the first plot, <cr> must be entered when using a PERICOM terminal to return the screen from graphics to text 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.

TPLOT

This plots the temperature log file either for a given run number or for the current run.

>> **TPLOT<cr>**
Give run number: **2518<cr>** (or **DAE <cr>**)
Give workspace: **1<cr>**
Give start date/time: **<cr>**
Give finish time: **<cr>**
Give SE block name: **TEMP<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, of the y axis on the plot. The log column refers to either the raw mV reading (column 2) or the converted temperature (column 3).

Useful DCL Commands.

POLPARS

This command interrogates raw data files and returns selected run information. It is called from DCL and can send output either to the screen or to a user defined file.

```
$ POLPARS

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

Output to a file (default is the screen) (Y/N) : Y
Enter name for output file : summary.dat
Enter first run number : 10226
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
```

APPENDIX A. INSTRUMENT PARAMETERS

A.1 Table of Approximate Detector Parameters (October 1994)

Detector	Spectrum	L1(m)	L2(m)	2θ(°)	Lsinθ(m)*
M1	S1	10.0	-	-	-
M2	S2 (U/S)	10.5	-	-	-
M3	S3	13.5	-	-	-
DAE diag.	S4	-	-	-	-
A1	S43	12.0	1.69	41.3	4.824
.	.	.	.	.	.
.	.	.	.	.	.
A40	S82	12.0	2.60	28.4	3.581
A41	S83	12.0	1.70	40.1	4.692
.	.	.	.	.	.
.	.	.	.	.	.
A80	S122	12.0	2.60	27.1	3.414
B1	S267	12.0	2.2	15	1.893
.	.	.	.	.	.
.	.	.	.	.	.
B20	S286	12.0	2.2	11	1.656
B21	S287	12.0	2.2	15	1.901
.	.	.	.	.	.
.	.	.	.	.	.
B40	S306	12.0	2.2	11	1.670
C1	S5	12.0	0.66	159.75	12.461
.	.	.	.	.	.
.	.	.	.	.	.
C19	S23	12.0	1.07	135.5	12.093
C20	S24	12.0	0.64	159.75	12.440
.	.	.	.	.	.
.	.	.	.	.	.
C38	S42	12.0	1.07	135.5	12.097
E1	S123	12.0	2.26	83.0	8.578
.	.	.	.	.	.
.	.	.	.	.	.
E36	S158	12.0	2.26	97.0	9.463
E37	S159	12.0	2.26	83.0	8.527
.	.	.	.	.	.
.	.	.	.	.	.
E72	S194	12.0	2.26	97.0	9.408
E73	S195	12.0	2.26	83.0	8.503
.	.	.	.	.	.
.	.	.	.	.	.
E108	S230	12.0	2.26	97.0	9.371
E109	S231	12.0	2.26	83.0	8.498
.	.	.	.	.	.
.	.	.	.	.	.
E144	S266	12.0	2.26	97.0	9.366

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

APPENDIX B. GENERAL INFORMATION

B.1 Useful Telephone Numbers

	Extension	Bleep
Instrument Scientists:		
Steve Hull	6628	
Ron Smith	5683	
Instrument Support:		
Jimmy Chauhan	5269	189
Other useful numbers		
POLARIS portakabin	6819	
ISIS Main Control Room	6789	
ISIS Health Physics	6696	
University Liaison	5592 (phone)	
	5103 (fax)	
Computer Support	3029 (mobile)	
	5414 (office)	174
Main Gate / Night Transport	5545	
Operator (daytime)	0	
Outside line	9	
EMERGENCY	2222	

To call a bleeper, dial 70, wait for the tone, dial the bleeper number followed by the number you wish them to call (i.e. 6819 for the POLARIS cabin) and wait for the tone once more before replacing the handset.

e.g. dial 70 (wait for tone) 189 6819 (wait for tone and replace handset) to page Jimmy Chauhan, asking him to call the POLARIS cabin.

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