The OSIRIS User Guide

1st Edition

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PREFACE

This user guide contains all the information necessary to perform a successful neutron scattering experiment on the OSIRIS spectrometer at ISIS, RAL, UK. Since OSIRIS is a continually evolving and improving instrument some information contained within this manual may become redundant. However, the basic instrument operating procedures should remain essentially unchanged. While updated manuals will be produced when appropriate, the most comprehensive source of information concerning OSIRIS is the Instrument Scientist /Local Contact. It would be appreciated, however, if this user guide were the first point of call should problems arise.

Acknowledgements

It is a pleasure to acknowledge all those who have contributed to the production of this user guide. In particular, past and present members of the Molecular Spectroscopy Group at the ISIS facility, UK, for fruitful discussion and comments.

CONTENTS

I. INTRODUCTION

This user guide contains all the information necessary to perform a successful neutron scattering experiment on the OSIRIS spectrometer at the ISIS Facility, RAL, UK. However, to ensure it is as concise as possible, other manuals and reports are referenced for specific details. Copies of all referenced material is either available in the instrument cabin or on the Internet at the given html address. Your Local Contact is also available for assistance and discussion regarding the precise details of the experiment.

This first section highlights the basic underlying physics of OSIRIS operating as a high-resolution quasi / in-elastic spectrometer and high-resolution long-wavelength diffractometer. Section 2, 'Performing an experiment on OSIRIS', details a typical experimental procedure. Finally, sections 3 and 4 discuss computer control as well as data analysis and visualisation.

1.1 THE INSTRUMENT

OSIRIS can be used as either a high-resolution, long-wavelength diffractometer or for high-resolution quasi / in - elastic neutron scattering spectroscopy. With regard spectroscopy, it is an inverted geometry instrument such that neutrons scattered by the sample are energy-analysed by means of Bragg scattering from large-area crystal-analyser array. In common with other instruments at a pulsed neutron-source, the time-of-flight technique is used for data analysis.

The instrument, situated on the N6(B) beam line at ISIS, views a liquid hydrogen moderator cooled to 25 K and consequently has access to a large flux of long-wavelength cold neutrons.

For the purpose of description, OSIRIS may be considered as consisting of two coupled spectrometer components.

i) THE ‘PRIMARY’ SPECTROMETER (BEAM TRANSPORT)

The ‘primary’ spectrometer is illustrated below in Figure 1.

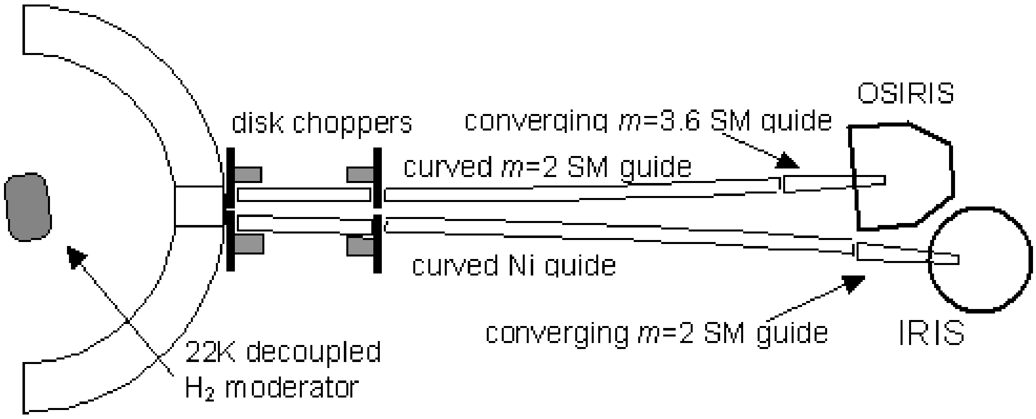

Figure 1 *The OSIRIS primary spectrometer*

Neutron beam transport, from moderator to sample position, is achieved using a curved neutron guide. Due to the curvature of the guide no neutron with a wavelength less than approx 1.5 Å is transported. While the majority of the guide section consists of accurately aligned m=2 super mirror sections (approx. 1m long and rectangular in cross-section), a 1.5m-long converging m=3 guide piece terminates the end. The tapered component helps focus the beam at the sample position (**44 mm (high) by 22 mm (wide)**) but also serves to increase flux. The incident neutron flux at the sample position is approximately **5.0 x 10^7 n cm^{-2} s^{-1}** (white beam at full ISIS intensity) with the wavelength intensity distribution at the sample position (up to 12Å) being illustrated in Figure 2. Note, however, that the flux at longer wavelengths (> 12 Å) is still sufficient to detect Bragg peaks with d-spacing close to 17.5 Å (corresponding to ~ 35Å neutrons !!) without significant frame overlap.

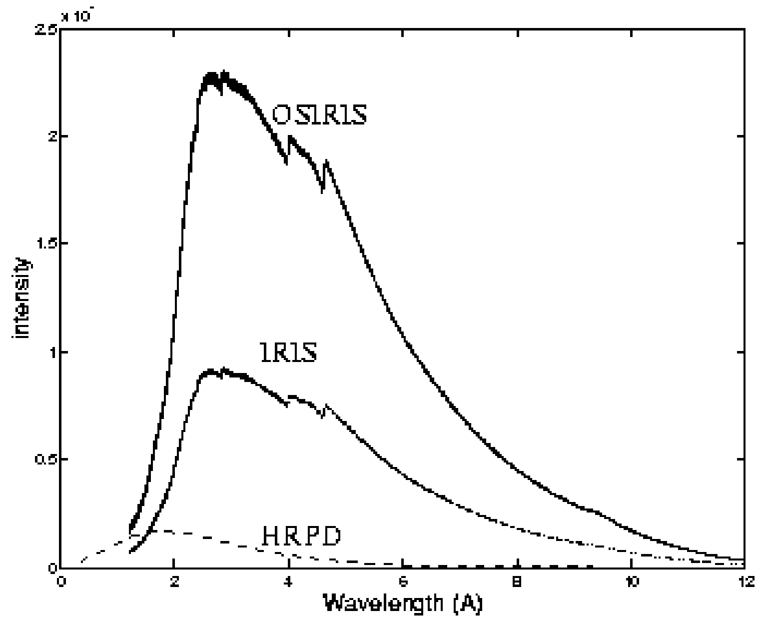

Figure 2 *Comparison of wavelength distributions on OSIRIS, IRIS and HRPD*

The wavelength profile on OSIRIS is illustrated in Figure 2. The increased in intensity on OSIRIS compared to that observed on IRIS is due to the use of the super mirror guide sections from moderator to sample - at present IRIS consists of accurately aligned nickel-plated glass tubes (approx. 1m long and rectangular in cross-section) terminated by a 2.5m-long converging nickel-titanium supermirror section. The flux on HRPD, though much lower for cold neutrons, extends to shorter wavelengths since HRPD views the liquid methane (CH_4) moderator at 100K, which gives an intensity maximum around 2Å.

In practice, the wavelength distribution illustrated above bears little resemblance to that observed in the incident beam monitor during an actual OSIRIS experiment since after leaving the moderator, and depending upon incident energy, each neutron either passes, or is absorbed by, one of two disc-choppers. In brief, the two choppers are used to define the range of neutron energies incident upon the sample during the experiment. Located at approx 6.3m (66 degree aperture) and 10m (98 degree aperture) from the moderator respectively, and operating at either 50, 25, 16.6 or 10 Hz, the choppers themselves are constructed from neutron absorbing material bar a small adjustable aperture through which the neutron may pass. The lower and upper limits of the incident wavelength band are therefore defined by adjusting the chopper phases, and hence opening times of each aperture, with respect to ‘t_0’ (the moment at which neutrons are produced in the target). Wavelength-band selection effectively defines the energy resolution and energy-transfer range (inelastic) or d-spacing range (elastic) covered during an experiment. Both choppers are synchronised to the ISIS operating frequency (50Hz) with the purpose of the 10m chopper being to avoid potentially problematic ‘frame’ overlap.

ii) THE ‘SECONDARY’ SPECTROMETER

The secondary spectrometer (Figure 3) consists of a 2m diameter vacuum vessel containing a pyrolytic graphite crystal analyser array, a 42-element 3He detector banks and a 8 module (962 ZnS tubes) diffraction detector bank oriented at $2\theta \sim 170^{o}$. The pyrolytic graphite analyser bank is cooled (installation of the cooling circuit is scheduled for mid 2003) close to liquid helium temperature to reduce background contributions from thermal diffuse scattering.

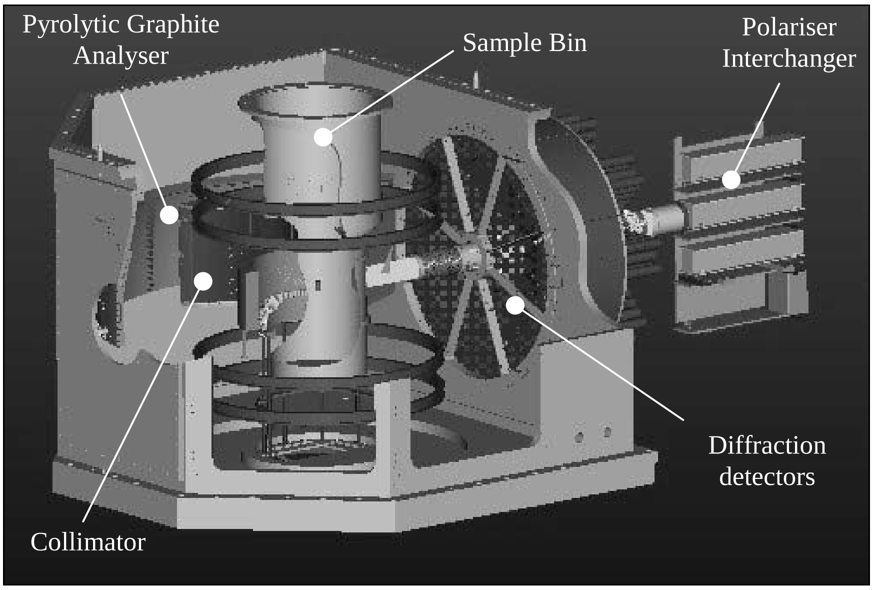

Figure 3 *The OSIRIS secondary spectrometer*

The incident beam monitor is placed immediately after the Polariser Interchanger. There is also a transmitted beam monitor in the 'get-lost' tube after the sample bin. Both are glass bead monitors. There are five beads horizontally and 6 vertically and the active component is ^{6}Lithium. The monitor efficiencies are wavelength-dependent, but always less than 1% over the range of wavelengths accessible on OSIRIS.

1.2. PRINCIPLE OF OPERATION

1.2.1. QUASI / IN - ELASTIC NEUTRON SCATTERING

In brief, during quasi / in-elastic neutron scattering experiments, the scattered neutrons are energy-analysed by means of Bragg-scattering from a large array of single crystals (pyrolytic graphite or mica). Only those neutrons with the appropriate wavelength/energy to satisfy the Bragg condition are directed towards the detector bank. By recording the time-of-arrival of each analysed neutron in a detector relative to t_0 , energy gain/loss processes occurring within the sample may be investigated. The quasi / in-elastic scattering process can be summarised mathematically as follows.

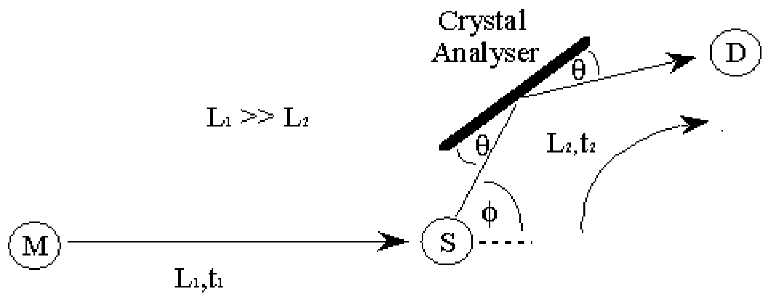

Figure 4 *An indirect-geometry inelastic neutron scattering spectrometer.*

During an OSIRIS experiment, the two disc choppers are used define the finite range of neutron energies incident upon the sample, S,

$$E = \frac{1}{2} m_n v^2 \quad \text{and} \quad p = m_n v = \frac{h}{\lambda} \quad \text{(de Broglie)} \qquad (1)$$

where m_n is the mass of the neutron. Consequently, the time-of-flight, t_1 , of each neutron along the primary flight path, L_1 , is variable. However, since only those neutrons with a final energy, E_2 , that satisfies the Bragg conditions,

$$\lambda = 2d\sin\theta \quad \text{(Bragg)} \qquad (2)$$

are scattered toward the detector bank, D, equations (1) and (2) can be re-formulated to give:

$$E_2 = \frac{1}{2} m_n \left(\frac{L_2}{t_2} \right)^2 = \frac{1}{2} m_n v^2 = \frac{p^2}{2m_n} = \frac{1}{2m_n} \left(\frac{h}{\lambda_a} \right)^2 = \frac{1}{2m_n} \left(\frac{h}{2d_a \sin\theta} \right)^2 \quad (3)$$

where d_a is the d-spacing of the analysing crystal.

The distance from the sample position to the detector bank (i.e. the secondary flight path, L_2) is accurately known. Consequently, the time, t_2, it takes for a detected neutron of energy E_2 to travel a distance L_2 can be calculated using,

$$t_2 = \frac{2m_n L_2 d_a \sin\theta}{h} \quad (4)$$

Should interactions within the sample lead to a loss/gain in neutron energy then a distribution of arrival times will result. By measuring the total time-of-flight, t (=t_1+t_2), and by having accurate knowledge of t_2, L_1 and L_2, the energy exchange within the sample can be determined:

$$\Delta E = E_1 - E_2 = \frac{1}{2} m_n \left[\left(\frac{L_1}{(t - t_2)} \right)^2 - \left(\frac{L_2}{t_2} \right)^2 \right] \quad (5)$$

1.2.2. DIFFRACTION

The diffraction detector bank on OSIRIS is used for either simultaneous measurement of structure vs. quasi / in-inelastic information or purely crystallographic determination during a diffraction experiment. Scattered neutrons reach the diffraction detectors directly and time-of-flight analysis is used to determine the d-spacing of the observed Bragg reflections. Here, the scattering geometry is simplified (Figure 5) with the scattering angle, 2θ, replacing the scattering angle, ϕ shown in the Figure 4.

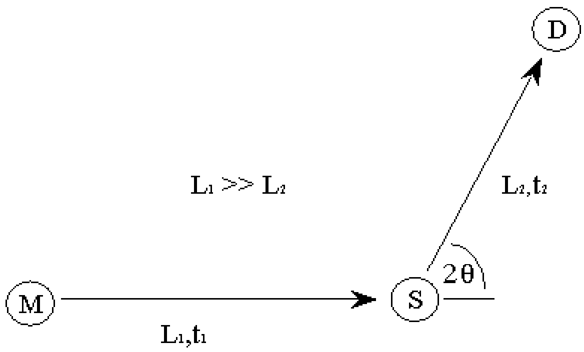

Figure 5 *A simple diffraction experiment*

From equations 1 and 2:

$$m_n\left(\frac{L}{t}\right)=\frac{h}{\lambda}=\frac{h}{2d_s sin\theta} \quad (6)$$

where L is the total flight-path, $L_1 + L_2$, t is the total flight-time, $t_1 + t_2$, and d_s represents the set of d-spacings measured,

$$d_s=\frac{ht}{2m_n L sin\theta} \quad (7)$$

II. PERFORMING AN EXPERIMENT ON OSIRIS

2.1. BEFORE ARRIVING AT OSIRIS

There are a number of administrative procedures that ***MUST*** be followed before arriving at the spectrometer. Failure to do so ***WILL*** delay the start of the experiment.

2.1.1. THE USER OFFICE, FILM BADGES AND SWIPE CARDS

Once at ISIS, the user should proceed directly to the User Office (U.O) in R3 to register his/her arrival. First time users will be given an information pack detailing all safety aspects at the facility. The user will also be required to watch the ISIS Safety Video. Once registration is complete, the user will then be directed to the ISIS Main Control room (MCR) in R 5.5 or perhaps the office of his/her Local Contact in R3. Outside office hours the MCR will hand out safety information but at the earliest available opportunity arrival should be registered at the U.O. Before entering building R55 a radiation badge and a 'swipe-card' (for entry into the experimental hall) must be obtained from the MCR.

2.1.2. SAMPLE SAFETY ASSESSMENT

As part of the beam time application procedure the ‘Principal Proposer’ will have submitted details concerning the chemical constitution of the sample(s) to be studied. This information is used to perform a sample safety assessment and subsequently generate a ‘sample safety assessment sheet’ detailing possible chemical or radiological hazards associated with the material. Recommended handling procedures after irradiation are also listed and ***MUST*** be followed. Before beginning the experiment the user should collect his/her sample safety assessment sheet from the filing cabinet in the Data Assessment Centre (D.A.C, building R55) and display it in the pocket beside the sample environment enclosure for the entire duration of the experiment. The user should have viewed the safety video and also read the safety handouts given to them when they arrived.

2.2. SELECTING SAMPLE CANS AND SCATTERING GEOMETRY

Sample can selection is usually determined by the type of experiment to be performed (i.e. diffraction or spectroscopy), form of the sample and/or the sample environment equipment to be used. Two geometries are available: cylindrical or flat plate.

2.2.1. FLAT PLATE CANS FOR QENS / INS EXPERIMENTS

The flat plate cans used on OSIRIS are made of aluminium and allow for a sample with a cross sectional area 40 x 40 mm but of variable thickness. The thickness itself is governed by the sample’s ability to scatter neutrons - a 10-15% scatterer is the ideal since multiple scattering is, in general, not a problem at this level. The optimal thickness of the sample can be roughly calculated using:

$$I = I_0 \exp(-n\sigma t) \quad \rightarrow \quad t = -\frac{1}{n\sigma}\ln\left(I/I_0 \right)$$

where I_0 is the incident intensity, I is the transmitted intensity, n is the number of scattering atoms per unit volume, σ is the 'average' scattering cross-section for the atoms in the sample and t is the thickness of the sample. For example, for a transmission of 85% (scattering of 15% ignoring absorption processes) then:

$$t = -\frac{1}{n\sigma}\ln(0.85)$$

More specifically, for polyatomic samples, $n\sigma = (n_1\sigma_1+n_2\sigma_2+n_3\sigma_3+...)$. However, in many cases all atoms bar hydrogen may be ignored since H has by far the largest incoherent scattering cross-section.

Flat plate sample cans are sealed using either indium (low temperature work, less than room temperature) or 'o'-rings (high temperature work) and may be used for liquids as well as powders. The advantage of using such cans is that the design specifically incorporates holes for cartridge heaters and temperature sensors enabling quick temperature changes and fine control. However, since the heaters and sensors have to be shielded (using cadmium) scattering in the plane of the sample will be greatly reduced and so sample orientation is important. In general, the sample can is oriented at ±45° relative to the incident neutron beam (straight-through is 0° with exact back scattering being 180° with angles on the graphite side of the instrument are defined as being positive and the angles on the mica side are negative). Which sample can orientation to use depends specifically upon the Q-range and energy-resolution required for the experiment. Cases to consider are:

i) *High-Q:* If high-Q values are required then reflection geometry is best (e.g. plane of sample at +45 such that the 'blind spot' occurs at low angles). Shielding the back of the sample with cadmium will reduce background scattering from the sample environment.

ii) *Low-Q*: If low-Q values are required then transmission geometry should be employed. A sample orientation of +135° is ideal for some magnetic scattering experiments in which the graphite 004 reflection is used (for its larger energy transfer range) but optimising the scattering on the lowest possible Q-values where the magnetic scattering is strongest. It should also be noted that spurious signals due to Bragg scattering would be reduced at low angles.

2.2.2. CYLINDRICAL CANS FOR QENS / INS EXPERIMENTS

The cylindrical sample cans used on OSIRIS are made of aluminium and are 50mm high by 20mm in diameter. For thin samples (0.5 to 2 mm), a hollow cylindrical insert may be placed inside resulting in an annular cross section (as viewed from above). The advantage of this sample geometry is that, unlike the flat plate cans, there are no edge effects and potentially problematic multiple scattering effects are reduced. In addition, sample can orientation is unimportant unless heaters and temperature sensors have been attached - without heaters/sensors there are no 'blind spots' on the analysers.

2.2.3. SAMPLE CANS FOR DIFFRACTION EXPERIMENTS

Cylindrical and flat plate aluminium sample cans can be used for diffraction experiments on OSIRIS. However, such cans should be used when the d spacing region of interest is greater than 2.2 Angstroms since any diffraction pattern collected below this value will exhibit a forest of strong Al Bragg reflections. Ideally, thin walled cylindrical vanadium sample cans should be used. 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.

2.3. LOADING A SAMPLE INTO THE NEUTRON BEAM

Most experiments on OSIRIS utilise the "Orange" cryostat - a card detailing its operation can be found in the pocket attached to the cryostat trolley. If one is not available, inform the Local Contact who will obtain a replacement and/or go through the operation of the cryostat and sample loading procedure. However, should a different piece sample environment equipment be requested (e.g. a CCR or Block heater) the Local Contact will provide assistance loading samples etc. Note: only personnel with a crane operator's licence (see Dennis Abbley for details, x 5455) are permitted to crane sample environment apparatus into and out of the beam line.

2.4. THE BEAM LINE SHUTTER INTERLOCK SYSTEM

The OSIRIS beam line shutter interlock system comprises of two coupled electronic/mechanical control systems; one to control the main shutter and which consequently affects both the IRIS and OSIRIS beam lines (N6A and N6B) and the other associated with only the OSIRIS intermediate shutter. There are very few occasions when it is necessary to open/close the main shutter and this should ***ONLY*** be done under the supervision of the Instrument Scientist or Local Contact. For information, main shutter controls can be found beside the IRIS cabin. The user may, however, operate the intermediate shutter control system after suitable instruction. The intermediate shutter control system, found on the instrument platform, consist of three boxes (shutter control, 'A' key and master key) and of a set of interlock keys (a master key (N6B-M) and three 'A'-keys labelled N6B-A) with corresponding locks.

The Local Contact will point out the location of these boxes and demonstrate how the interlock system operates. However, to summarise, the intermediate shutter cannot be opened unless all four keys are in their appropriate locks in the correct control boxes. Inserting and turning (clockwise) all the 'A-keys' in the 'A-key' box releases the master key (N6B-M). The master key can then be inserted into the lock in the side of the master key box. Once in position, and turned, the intermediate shutter can be opened by pressing the 'open' button on the shutter control box.

Upon pressing 'open' the master key is locked into position and cannot be removed until the intermediate shutter is closed. In principle, this means that all active areas on the OSIRIS beam line are inaccessible while the intermediate shutter is open. The area underneath the instrument platform, for will require access for some future instrument configurations, is only accessible with the main shutter has closed. Entry into this area is only allowed under the supervision of the Local Contact or Instrument Scientist.

Regaining access to an interlocked area (e.g. the sample environment enclosure) requires reversal of procedure outlined above. The shutter is closed, the master key is removed and inserted into the 'A-key' box which subsequently releases all three the A-keys for access to interlocked areas.

2.5. OSIRIS COMPUTING OVERVIEW

OSIRIS is controlled using a DEC Alpha Workstation. Commonly referred to as the OSIRIS FEM (Front End Minicomputer), the workstation makes use of the DEC OPENVMS operating system. In addition, there are two PC's available for use - while one is primarily for data analysis and file transfer the other (the machine by the door) should ***ONLY*** be used to control *'Ray Of Light'* software. The ALPHA workstation is configured to use the DECWINDOWS windows management system that has the advantage of automatically starting those windows necessary for instrument control and data analysis i.e. an OSIRIS DECTERM control window and/or the instrument 'Dashboard'. If the 'Dashboard' is not automatically launched upon logging in or not visible then type 'STAT ON' in any active DECTERM window. While all instrument control commands should be typed into the DECTERM window the 'Dashboard' simply provides information about the state of the instrument (section 3.1.4). Each user should work exclusively in his / her own directory. The directory name is given by the year of the experiment and the user's surname,

e.g. **osiris$disk0:[osiris.users.2003.jones]**

From here, the user can run the instrument and also analyse data. The local contact will create the directory at beginning of the experiment and you will automatically find yourself there when you open a new window. For those unfamiliar with VMS, below is a short table of equivalent UNIX/VMS commands:

Action	*UNIX*	*VMS*
Show current directory	pwd	show default
Change directory	cd	set default
List files in current directory	ls	directory
Delete file	rm	delete
Copy file	cp	copy
Move file	mv	rename

Most VMS system commands can be shortened to 3 or even 1 character. A more complete VMS help page (actually describes UNIX for VMS users) can be found at

http://cc.uoregon.edu/unixhelp/VMStoUNIX.html

Directories are written within square brackets and separated by full stops. To change directory you either give the full name or if you want to go down one level you need only type: ***SET DEF [.JONES]*** Alternatively, if you want to go up one step you can use: ***SET DEF [-]***. You can create a new directory by writing: ***CREATE/DIR [.NAME]***. Finally, ***NEDIT*** is a good, full-screen text editor, which is recommended for creating and editing text files e.g. command files.

2.6. SUITABLE INSTRUMENT SETTINGS

OSIRIS is easily configured to match the scientific problem under investigation. In brief, it is simply a matter of selecting an appropriate resolution and energy-transfer-range or, in the case of diffraction, the appropriate d-spacing range(s). For quasi/in-elastic scattering experiments different resolutions are associated with the different analyser reflections available. Selecting a particular analyser reflection (and hence resolution) and energy-transfer-range is achieved by defining:

a) the frequency and phases (time-delay settings relative to t_0) of the two disc-choppers and

b) the time-channel-boundaries (TCB's) for data acquisition.

The procedure is the same for selecting a particular d-spacing range when simply using the instrument as a diffractometer.

Standard instrument settings can be found in the Appendix along with corresponding chopper frequencies and phases. These settings are 'loaded' by typing single word commands (also given in the Appendix) in any active DECTERM window. However, occasion may arise when the nature of the problem under investigation warrants modified setting i.e. the standard settings are inappropriate because of the presence of spurious peaks. In this case seek advice from the Local Contact or Instrument Scientist.

2.7 DATA COLLECTION

2.7.1. CHANGE

Typing CHANGE <CR> in any DECTERM window will enter the DEFT screen editor (section 5.1.4 of the PUNCH manual [1]) and allow parameters within the Current Run Parameter Table (CRPT) (see section 3.1.2), such as the monitor range, time-channel-boundaries (TCB's) and the title of the experiment, to be modified. Key number ‘7’ on the right-hand keypad allows passage through the CRPT. To move from one data input field to another the 'up' and 'down' cursor arrow keys should be used. Should the prompt **'toggle data type'** appear press the **'.'** key on the right hand keypad until the field displays the required option. All other fields may be altered by typing appropriate numbers / characters. Corrections may be made using the 'left' and 'right' cursor arrow keys, the 'delete' key and the space bar. To exit the DEFT editor and save entries to the CRPT press the 'PF1' key on the right hand keypad followed by 'E' for Exit. If you wish to quit and leave the editor without overwriting the CRPT then press 'Q' for Quit. A successful edit will produce the following responses: ‘**Values written to INST.UPD’** and ‘**All parameters updated successfully’.**

In practice, single command words (see Appendices) are used to 'load' the different parameters for different instrument settings. Consequently, all that is required of the User is to enter an appropriate title, User names and experiment RB number. In addition, the User might wish to check that the CRPT lists the correct TCB's (in micro-seconds) and the correct monitor range (in micro-seconds) and, if going from an inelastic set-up to a diffraction set-up (or vice-versa), the correct spectra table (SPECTRA-JCR.DAT for inelastic experiments and SPECTRA.DAT for diffraction experiments). No other input is necessary although information such as type of sample can, orientation and scattering geometry can also be entered.

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 state of the DAE (section 3.1.1). For example:

CHANGE TITLE """An OSIRIS experiment""" <CR>

will alter the title of the current experiment. The treble quotes ensure that the title is reproduced literally with upper and lower case characters, spaces etc. A CHANGE command requires further explanation since it involves the use of the DEFT

2.7.2. BEGIN

To start a run type ‘BEGIN’ in any DECTERM window. After a few seconds the ‘Dashboard’ should indicate that OSIRIS is ‘RUNNING’ and the total number of micro-amps and the monitor counts will begin to increment.

2.7.3. DATA INSPECTION

To inspect diffraction or spectroscopy data sets as they are being collected use either the data visualisation program GENIE or OPENGENIE. Alternatively, diffraction data can be visualised in LAMP while spectroscopy data should be viewed using MOSES. These programs will aid a decision as to when it is appropriate to end a measurement. See section 3.2

2.7.4. END AND END OF EXPERIMENT

Once the data collected is of sufficient quality for subsequent detailed analysis, typing END will stop the run and store the data. The data is automatically archived after a few minutes onto an optical disk storage system. However, before the User leaves the beam line at the end of a scheduled experimental period, he/she ***MUST*** have all irradiated samples monitored for induced radioactivity. Assistance and advice in this matter may be sought from the ISIS Health Physics Office (6696) or the ISIS Main Control Room (6789).

How to treat radioactive samples (ISIS duty officer x6789) .	
>10mSv/hour	Phone ISIS duty officer leave sample within interlocked area
>0.1mSv/hour	Store sample in OSIRIS active sample cupboard with sample record sheet. The sample may NOT be removed from its container. For removal from ISIS contact the duty officer.
<0.1mSv/hour	The sample is not radioactive. For removal from ISIS contact the duty officer

If the sample is not active it should be removed from its can, the can cleaned ready for the next Users and the sample dealt with as according to the sample safety assessment (i.e. stored at ISIS, removed from ISIS or disposed of by ISIS staff). If removal of the sample from ISIS is required but not immediately possible due to the level of induced activity, arrangements should be made with the Local Contact to remove it at the earliest available opportunity. All active samples should be stored in the 'Active Sample' cupboard and ***MUST*** should be logged in (on storage) and out (upon removal) in the logbook located inside the cupboard. It is not guaranteed that samples will remain stored at ISIS indefinitely so do not forget do leave your e-mail address, so that we can contact you when the sample is safe for you to bring back.. It may be possible, with the assistance of Radiation Protection (6696), to package an active sample in such a way as to make its removal from ISIS safe. Before leaving, all film badges and swipe cards should be returned to the MCR.

III. OSIRIS COMPUTING

3.1. INSTRUMENT CONTROL

3.1.1. DATA ACQUISITION ELECTRONICS

During the course of a run, data is accumulated in the Data Acquisition Electronics (DAE) in a number of spectra, each spectrum corresponding to a particular detector. Each of these spectra contains a histogram of neutron counts versus time-of-flight. At the end of the run the contents of the DAE are automatically copied to a file on the FEM called OSI*****.RAW, where '*****' is a five figure run number incremented automatically at the end of each run. Shortly after creation, this RAW file is archived onto optical disk. The DAE has four possible states

SETUP	Data not collected. Instrument parameters may be changed.
RUNNING	Data is currently being collected and stored in the DAE
PAUSED	Data collection is temporarily suspended by the User
WAITING	Data collection is temporarily suspended for example, when a cryostat temperature is outside defined limits

The current DAE mode and run status are displayed on the 'Dashboard'.

3.1.2. THE C.R.P.T

The Current Run Parameter Table (CRPT) mentioned in section 2.8.1 contains information about 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 measurement. Relevant information includes the title of the experiment/run, user names, etc. plus details of the instrument configuration and settings (the TCB's, the detector used as the monitor spectrum, etc.).

3.1.3. THE I.C.P

The Instrument Control Program (I.C.P) controls data collection on most ISIS instruments. This program is used to start and stop data collection, but also allows data collection to be suspended temporarily to allow, for example, entry into an interlocked area. Data collection can also be suspended automatically if the CAMAC-based sample environment control system (section 3.1.5) indicates that, for example, the temperature has drifted outside of pre-defined limits. Commonly used I.C.P commands include:

CHANGE	Enables the contents of the CRPT to be modified (see section 2.8.1)
BEGIN	Clears the DAE memory, sets parameters in the DAE to those specified by the CRPT, instructs the DAE to start data collection. Sets DAE state to RUNNING
PAUSE	Suspends data collection by the DAE. Sets DAE state to PAUSED
RESUME	Resumes data collection by the DAE. Sets DAE state to RUNNING
UPDATE	Suspends data collection by the DAE. Copies the contents of the DAE to the CRPT. Restarts data collection by the DAE
STORE	Suspends data collection by the DAE. Copies the contents of the CRPT to the file IRS*****.SAV (***** = run number). Restarts data collection by the DAE.

ABORT Stops data collection by the DAE. Does NOT store data. Sets DAE state to SETUP.

END Stops data collection by the DAE. Copies the contents of the DAE memory and CRPT to file IRS*****.RAW. Increments the run number ‘*****’. Sets DAE state to SETUP.

The ABORT command does not store the accumulated data and so should only be used if it is certain that the data is not needed.

If ABORT is issued accidentally it is possible to recover the data using the program RECOVER, but only if a BEGIN command has not been issued since BEGIN clears the DAE memory. Within any active DECTERM window (or wherever the BEGIN and ABORT commands were issued) type at the prompt:

RUN SYS$PUBLIC:RECOVER

3.1.4. THE DASHBOARD

As mentioned in section 2.5, the OSIRIS ‘Dashboard’ (Figure 6) should be launched in a separate window on the FEM. It displays information concerning the current run such as current DAE state (RUNNING, SETUP, etc.) and run number.

In addition, information concerning the User, sample, run time, frame (proton pulse) count, present and accumulated proton beam current, the incident beam monitor counts and sample environment parameters being monitored by CAMAC is also displayed.

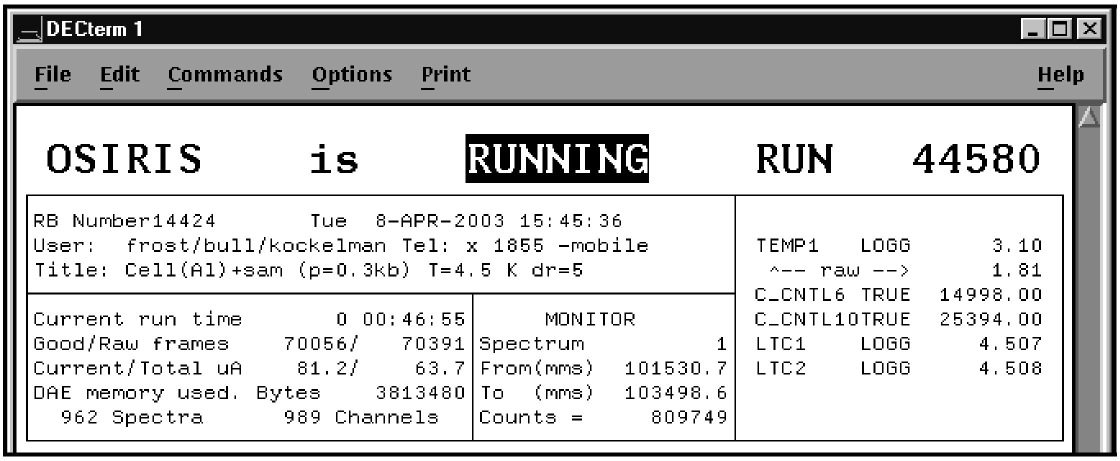

Figure 6 *The OSIRIS Dashboard*

A more advanced version of the OSIRIS 'Dashboard' is in development but of sufficient functionality that it is now available for general use. The new 'Dashboard' may be launched from any DECTERM window by typing '**TCP**' although it is advisable to close any other 'Dashboards' that may already be running. The new 'Dashboard' allows for instrument control via pull down menus rather than entering command lines in a DECTERM window. While most options are self-explanatory, the Local Contact will be available to provide further instruction.

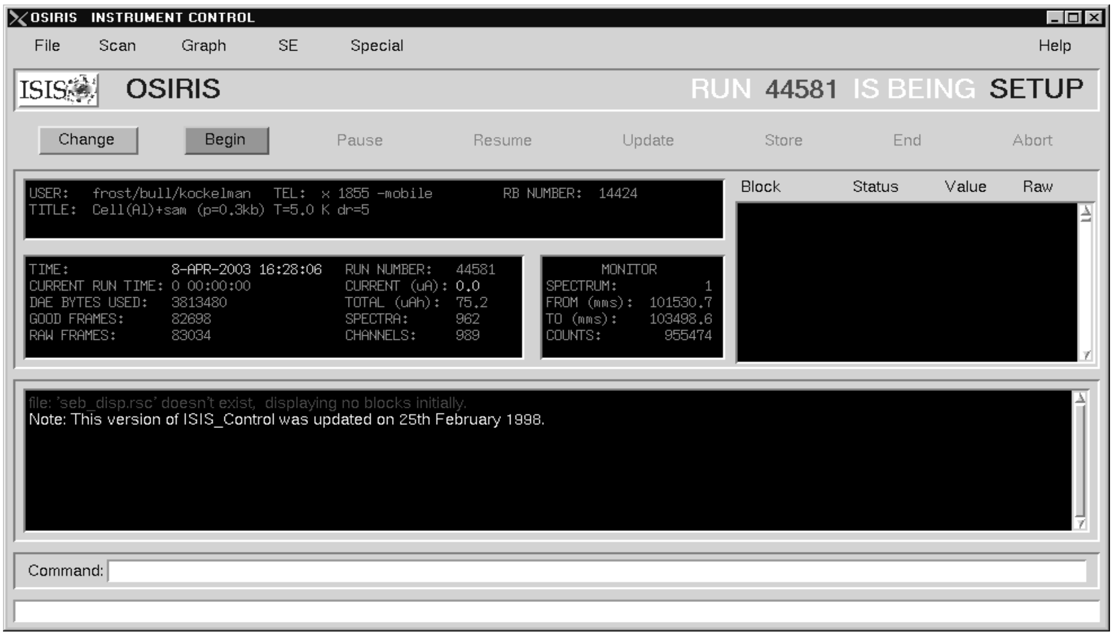

Figure 7 *The new OSIRIS Dashboard*

3.1.5. S.E.C.S, CAMAC AND EUROTHERM

A complete overview of the Sample Environment Control System (S.E.C.S) can be found in the PUNCH manual (ref. [2] section 5.2). However, those commands necessary for temperature control are outlined in the following section. The temperature of the sample and/or sample environment equipment can be set, as well as logged, from any computer terminal 'connected' to the OSIRIS FEM. In addition, data collection can be temporarily suspended when the temperature drifts outside of a specified range. There are essentially three aspects to the temperature control system. The FEM (for issuing the commands), the CAMAC unit (hardware/software interface) and the Eurotherm temperature controllers. The temperature controllers measure the millivolt output from resistance thermometers (Rh/Fe or Pt) or thermocouples (usually type-K) and control the temperature at a specified set point using a 3-term control algorithm (proportional band, integral time and derivative time - commonly referred to as PID control). The conversion from millivolts to K or C is achieved using 'look-up' tables held on the mainframe (each Rh/Fe sensor for example is calibrated at a number of points and has its own conversion table and identification number). While the unit of temperature (K or C) depends upon the sample environment equipment being used it would normally be Kelvin for a cryostat and Celsius for a furnace. The 'Dashboard' usually displays both the millivolt readings and the corresponding K or C value. TEMP1 and TEMP2 are examples of two software temperature control blocks in the S.E.C.S that correspond to the two EUROTHERM temperature controllers.

3.1.6. TEMPERATURE CONTROL

Listed below are the more useful commands in the S.E.C.S relating to the control of temperature:

CSET TEMP1 / LOG — Causes the temperature readings observed in temperature control block, TEMP1, to be logged. Each time an OSIRIS run is ended the temperature log file is closed and a new one opened. These files are called IRS*****.LOG where ' ***** ' is the run number. The .LOG file is stored on the FEM and archived to optical disk along with the RAW file.

CSET TEMP1/DEVSPEC=5864	Informs the S.E.C.S which calibration table control block TEMP1 should interrogate when converting mV to Kelvin. In this example, Rh/Fe sensor 5864 is to be used - the sensor numbers are written on the side of the sensors).
CSET TEMP1 / DEVSPEC = - 2	This command informs the control system that type-K thermocouple thermometry is being used.
CSET PROP1 2	This command sets the proportional band associated with temperature control block TEMP1 to 2% of the set point value **
CSET INT1 50	This command sets the integral time to 500 seconds **
CSET DERIV1 10	This command sets the derivative time to 100 seconds **

CSET TEMP1 15 / LOLIMIT = 10 / HILIMIT = 20 / CONTROL

This command issues a set point value of 15 (K or C) to temperature control block TEMP1. The controller attempts to maintain a temperature of 15 +/- 5 (K or (C) as denoted by the 'limits'. LOLIMIT and HILOIMIT are used by the ICP to inhibit data collection because of the /CONTROL prompt. If TEMP1 varies outside this range the ICP makes OSIRIS go into the WAITING state until the value returns into the range.

CSET TEMP1 / NOCONTROL	Data collection vetoing is disabled if TEMP1 falls outside HILIMIT or LOLIMIT,

CSHOW TEMP1

Displays information about the current status of TEMP1. The set point value and limits will be shown as well as an indication of whether this value is being logged and used as a control parameter (in the form T or F for true or false).

CSHOW CAMAC/OUT_OF_RANGE and CSHOW CAMAC/CONTROL

Indicates which temperature control blocks are causing data collection to be inhibited and those in use as control parameters, respectively.

*** suitable P.I.D values for the different pieces of sample environment apparatus used on OSIRIS are listed in Appendix IV*

3.1.7 COMMAND FILES

Automatic control of OSIRIS can be achieved using a simple command file written by the user. The command file is created using one of the VMS editors and should ideally have the extension *'.COM'*. An example .COM file is given below:

```
$! Measure at T=1.5K on d-range 1 and T=10K on d-range 2
$ cset temp1 1.5/hilimit=3.0/lolimit=1.0/control
$ drange 1
$ begin
$ change title """An OSIRIS experiment at T=1.5K d1"""
$ waitfor 50 uAmps
$ end
$ cset temp1 10/hilimit=12/lolimit=8/control
$ wait 00:30:00
$ drange 2
$ begin
$ change title """ An OSIRIS experiment at T=10K d2"""
$ waitfor 50 uAmps
$ end
```

Create command files in your own directory using ***NEDIT.*** After it has been saved, the syntax can then be checked using the '***checkcom***' program:

>checkcom
Enter name of command file :

If everything is OK, the program will return: ***Syntax check OK***

If you get any warning or error messages, you should go through the command file and correct them. Checkcom only spots the most obvious syntax and logical errors, so you still need to check your command files manually. After checking the syntax, *checkcom* will prompt you for the chopper frequency (hitting return will leave the program assuming it's 25Hz) and it will calculate the total running time of the command file.

Once OK, the command file can then be executed using the execute command:

EXECUTE TEST.COM or ***@TEST***.

Other useful commands when running command files are:

EXECUTION Shows which files are executing or waiting to execute

END_EXECUTION ends current run and stops the command file running

ABORT_EXECUTION aborts the current run and stops the command file

CLEAR_EXECUTION clears the batch queue and leaves the current run as it is

To view a read-only summary of all OSIRIS data sets collected type: ***JOURNAL***

3.2. DATA VISUALISATION AND ANALYSIS

Data visualisation, and subsequent analysis, software can be accessed from an account on a VMS machine by first modifying, and then re-running (by typing @login), the User’s ‘login.com’ file to include the following lines:

```
$ SET NOON
$ @OSIRIS$DISK0:[OSIRIS]USER_SETUP
$ IF f$mode().nes."BATCH" then goto cont
$ EXIT
$ CONT
```

3.2.1. INTERACTIVE PROGRAMS

- ### GENIE AND OPENGENIE

GENIE - "A Language for Spectrum Manipulation and Display" and the more advanced OPENGENIE software packages are common to all ISIS instruments and used for displaying and manipulating spectra and data sets. Detailed information can be found in the GENIE user manual available in the instrument cabin, from the ISIS computer support group or online at:

http://www.isis.rl.ac.uk/computing/Software/Genie2/genie2.htm

Alternatively, a comprehensive overview of OPENGENIE, is now available:

http://www.isis.rl.ac.uk/OpenGENIE/

To start GENIE: type '***GENIE***' in any DECTERM window.

To start OPENGENIE: type ***'OPENGENIE -L'*** in any DECTERM window.

Useful data visualisation commands for both packages include:

GENIE v2 Command	***Open GENIE Command***	***Description***
a b N (N=1,2,3,…)	a/b N	*alter binning*
a m N	a/m N	*alter markers*
ass	ass	*Assign*
d/h/l/m/e	d/h/l/m/e	*Display*
l	l	*Limits*
m	m	*Multiplot*
c/v/h	c/v/h	*Cursor*
ex	ex	*Exit*
j "OS command"	j "OS command"	*Jump*
k/h	k/h	*keep hardcopy*
p/h/l/m/e	p/h/l/m/e	*Plot*
reb	reb	*Rebin*
set disk my$disk	set/disk "my$disk:"	*set disk*
set dir [mydir]	set/dir "[mydir]"	*set directory*
set ext raw	set/ext "raw"	*set extension*
set inst ANY	set/inst "ANY"	*set instrument*

set par	setpar	*set wksp parameters*
set title w1	w1.title=	*set title*
sh data	sh/data	*show data*
sh par	sh/par	*show parameters*
sh def	sh/def	*show defaults*
u/?	u/?	*Units*
z	z	*Zoom*

Conversion of all OSIRIS data visualisation software to run under the OPENGENIE platform is currently underway.

- ## LAMP

LAMP is a package for the reduction of diffraction data collected on OSIRIS (i.e. reducing the data to GSAS, CCSL or some other portable format). LAMP runs under IDL with a convenient syntax for manipulating neutron data. For details about how to use LAMP see:

http://www.isis.rl.ac.uk/molecularspectroscopy/osiris/osiris_dataanalysis.htm

- ## MOSES

MOSES is a suite of programs for the full reduction and analysis of OSIRIS spectroscopy data which supersedes the IRIS data analysis software package, GUIDE. An online MOSES manual can be found at:

http://www.isis.rl.ac.uk/molecularspectroscopy/osiris/

A more in-depth descriptions of the individual analysis programs themselves can be in the GUIDE and IDA manuals. These can be found online at:

http://sutekh.nd.rl.ac.uk/wsh/index.html

MOSES can be launched by typing '**MOSES**' or '**OTKGENIE**' in any active DECTERM window.

IV REFERENCES

i) *PUNCH user guide*. R G Parry et al. RAL Report, RAL 88109 (1988).

ii) *GUIDE – OSIRIS Data Analysis* M.T.F.Telling and W.S.Howells RAL Report: RAL-TR-2000-004, Jan 2000 (http://www-dienst.rl.ac.uk/library/2000/tr/raltr-2000004.pdf)

iii) GSAS user guides, software and information: *http://public.lanl.gov/gsas/*

iv) Cambridge Crystallography Subroutine Library (CCSL) information: http://www.isis.rl.ac.uk/crystallography/documentation/CCSL/CCSLguideFramePage.htm

APPENDIX I – QUASI / IN ELASTIC SETTINGS.

Analyser reflection / relative flux intensity	Resolution (FWHM) at elastic line (μeV)	Δ E (meV)	Chopper operating frequency (Hz)	Time Channel Boundaries (μ s)	Monitor boundaries (μ s)	Phases (μS) $\theta_{6.3}$ / θ_{10}	Computer command
PG002 (1.0)	24.5	-0.5 to 0.5	50	52000 - 72000	63000 - 65000	9978 /15000	PG002
(1.0)	"	-0.3 to 1.4	50	44500 - 64500	63000 - 65000	8100 / 13250	PG002_OFFSET
PG004 (0.7)	54.5	-3.5 to 4.0	50	22500 - 42500	31000 - 33000	4500 / 6900	PG004

Table 1 *Standard Quasi / In - elastic settings*

Appendix II – Diffraction Settings.

The diffraction detectors are placed in a ring around the incident beam. They cover the full range of scattering angles 2θ from 150º to 171º, providing a total solid angle coverage of 0.67 steradians.

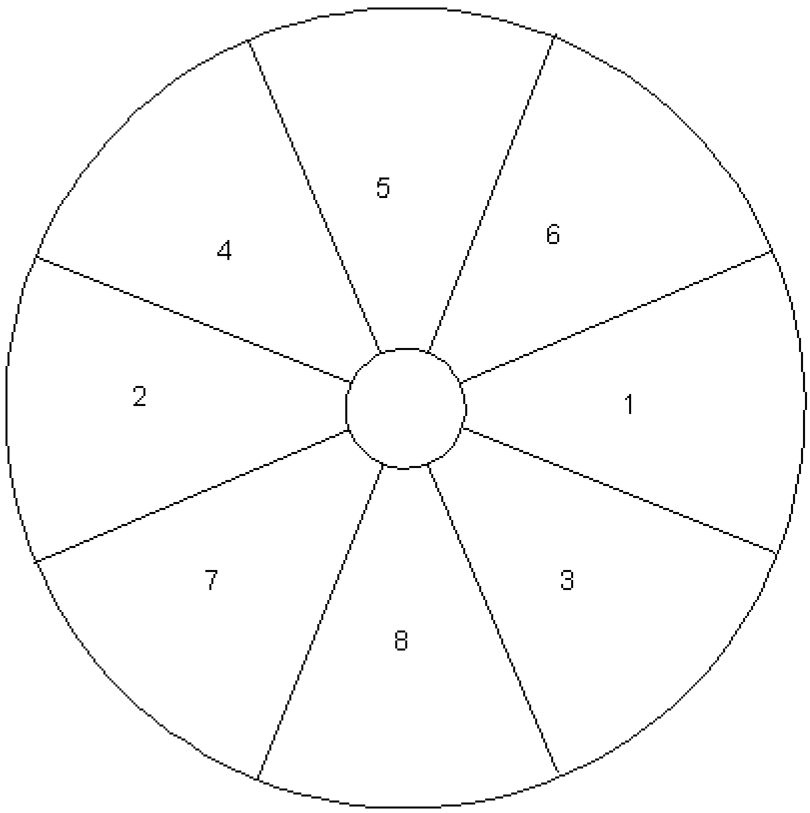

Figure 8: Diffraction modules as seen from the sample position

The detectors are shown schematically above and as seen from the sample position. They are scintillators and the full detector bank contains 8 modules, numbered according to the order in which they were installed. Each module consists of 120 detector elements. The first 20 are single detectors. Between 21 and 120 the even numbered detectors are still single but the odd numbered are physically composed of one detector above and one below the central strip, hardwired together, as shown for module 1 below.

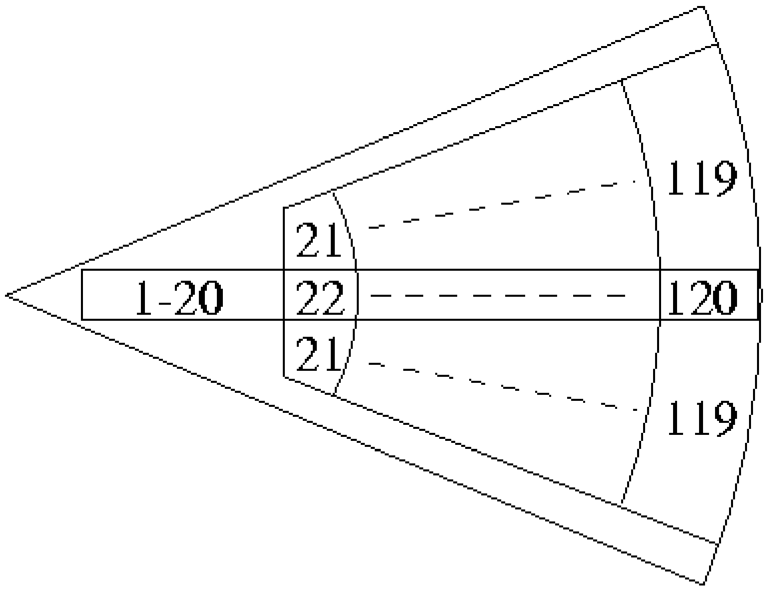

Figure 9: Location of the 120 diffraction detectors in module 1

The choppers typically run at a frequency of 25 Hz, which allows a 4 Å wide wavelength range to reach the sample with minimal contamination from other wavelengths. The d-spacing measured are given by Bragg's law: $\lambda=2d\sin\theta$ and since the diffraction detectors are close to backscattering ($\Rightarrow \sin\theta \approx 1$) the d-spacing is about half the neutron wavelength. The range of d-spacing measured with the choppers at 25Hz is thus about 2 Å wide. In order to measure a full range of d-spacing to properly characterise the sample, a series of runs are usually performed with an incremental increase of the chopper phasing. The data from these runs are then merged in software to create a data set that spans the full range of d-spacing of interest in a continuous manner.

The combination of a cold moderator and a super mirror guide provides a high flux of cold neutrons on OSIRIS. However, while neutron wavelengths of up to 70Å re accessible, 35Å is the experimental limit. The reduction in flux at long wavelengths is partly compensated for by the λ^4 form factor that applies to Bragg peak intensities. The use of a super mirror guide significantly enhances the neutron flux at all wavelengths, but at a price in beam divergence, which increases substantially as a function of wavelength. This is why the diffraction detectors on OSIRIS only cover the highest scattering angles, where beam divergence does not contribute greatly to the instrumental d-spacing resolution. At $\Delta d/d$ down to 2.5×10^{-3}, OSIRIS provides similar resolution to the HRPD 90° bank with the same counting rate as on GEM. OSIRIS can reach d-spacings in excess of 30 Å with high resolution, but cannot access d-spacing below about 0.9 Å, due to the curved guide cut off.

d-spacing range (Å)	Time-channel-boundaries (μ S)	Monitor boundaries (μS)	Phases (μS) $\theta_{6.3}$ / θ_{10}	TCB File	Computer command
0.50 to 2.90	11700 - 51700	30700 -32700	2394 / 3809	L02_2_2	drange 1
1.70 to 4.10	29400 - 69400	48400 - 50400	6382 / 10158	L04_2_2	drange 2
2.70 to 5.00	47100 - 87100	66100 - 68100	9573 / 15236	L06_2_2	drange 3
3.50 to 6.00	64800 - 104800	83800 - 85800	11807 / 20315	L08_2_2	drange 4
4.60 to 7.00	82500 - 122500	101500 - 103500	14998 / 25394	L10_2_2	drange 5
5.60 to 8.00	100200 - 140200	119200 - 121200	18189 / 30473	L12_2_2	drange 6
6.60 to 9.00	117900 - 157900	136900 - 138900	21380 / 35551	L14_2_2	drange 7
7.60 to 10.00	135500 - 175500	154500 - 156500	24571 / 630	L16_2_2	drange 8
8.60 to 11.00	153200 - 193200	172200 - 174200	27762 / 5709	L18_2_2	drange 9
9.60 to 12.00	170900 - 210900	189900 - 191900	30953 / 10788	L20_2_2	drange 10
10.60 to 13.00	188600 - 228600	207600 -209600	34144 / 15867	L22_2_2	drange 11
11.60 to 14.00	206300 - 246300	225300 - 227300	37336 / 20945	L24_2_2	drange 12
12.60 to 15.00	224000 - 264000	243000 - 245000	527 / 26024	L26_2_2	drange 13
13.60 to 16.00	241700 - 281700	260700 - 262700	3718 / 31103	L28_2_2	drange 14
14.80 to 17.35	259400 - 299400	278400 - 280400	7866 / 36182	L30_2_2	drange 15
15.80 to 18.40	237100 - 277100	256100 - 258100	11057 / 1260	L32_2_2	drange 16
15.80 to 18.40	254800 - 294800	273800 - 275800	14248 / 6339	L34_2_2	drange 17
15.80 to 18.40	232500 - 272500	251500 - 253500	17439/11418	L36_2_2	drange 18

Table 2 *Standard Diffraction Settings* ***at 25Hz***

- ***drange*** is used to change chopper phases and load new time channel boundaries. As an example look at [osiris.control]drange_2_2.com - the 2_2 in the filename refers to running both the choppers at 1/2 the ISIS frequency, i.e. at 25Hz. There are also files called drange_1_1.com and drange_5_5.com for use when running the choppers at 1/1 ISIS frequency (50Hz) and 1/5 ISIS frequency (10Hz) respectively. By default, ***drange*** runs the drange_2_2 command file.

APPENDIX III – INSTRUMENT PARAMETERS.

Operating vacuum:	5 x 10^{-5}mb (instrument tank)
	1 x 10^{-5}mb (sample environment bin)
Primary instrument flight-path:	L_1 = 34.00 m

<u>Inelastic:</u>

Analysing energy (for graphite at RT)	1.828 meV
Average secondary flight-path:	L_2 = 1.582 m
Angular coverage of ^{3}He detector bank:	11° < 2θ <150°
Spectrum numbers:	963 to 1004

<u>Diffraction:</u>

Average secondary flight path:	L_2 = 1.005m
Angular range of diffraction detectors:	167.1° < 2θ <172.4°
Resolution: .	$5\times10^{-3} < \Delta d/d < 6\times10^{-3}$
Solid angle:	0.67 steradians
d-spacing range:	0.8 Å < d < ~35 Å

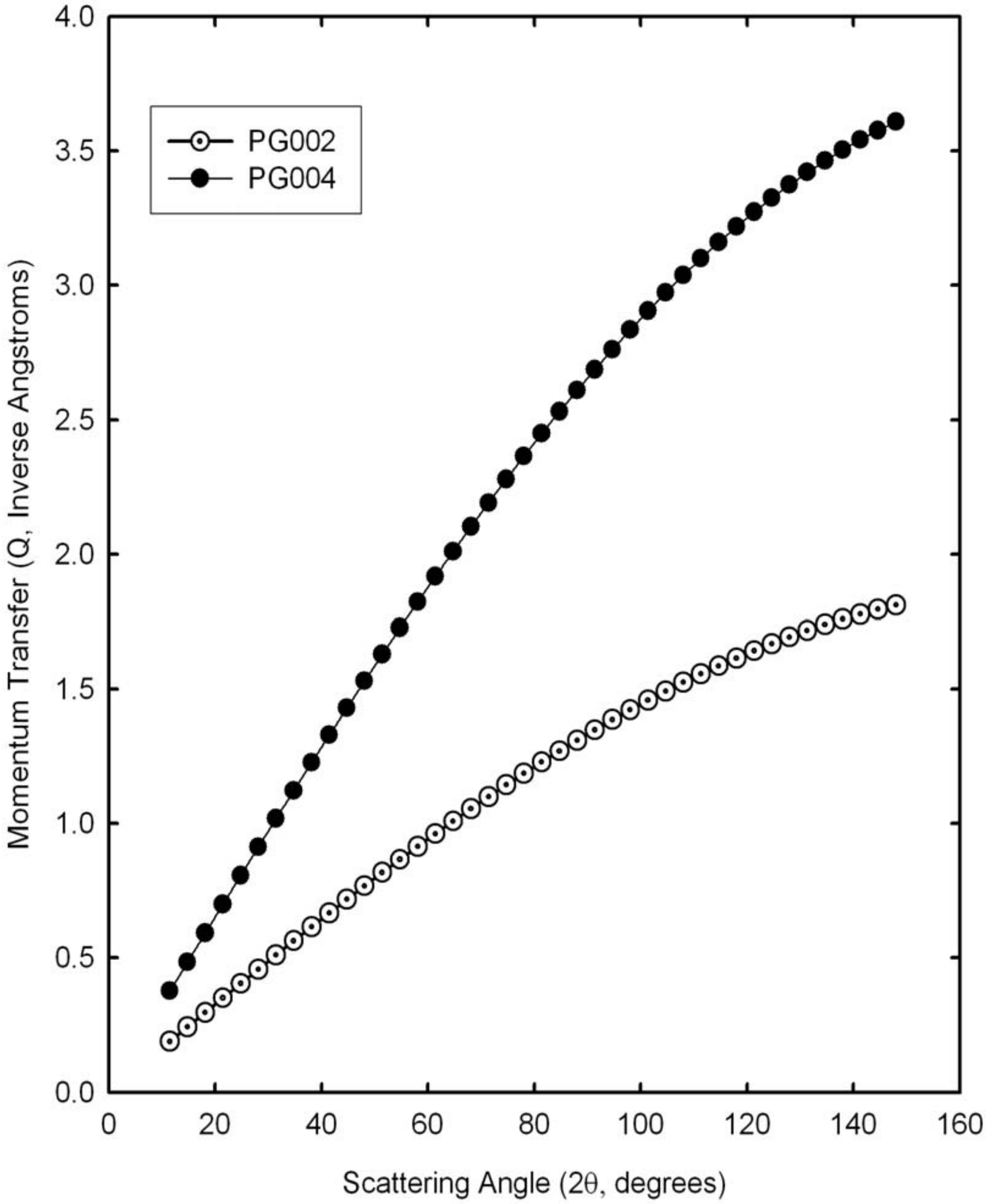

Figure 10: Momentum transfer vs. scattering angle for the OSIRIS pyrolytic graphite analyser bank. The Q values are those calculated at the elastic line i.e. $K_I = K_F$ Exact values are given in the table below

Detector No.	Spectra Number	2θ (degrees)	Q Value (002)	Q Value (004)
963	1004	148.00	1.81	3.60
964	1003	144.67	1.79	3.57
965	1002	141.34	1.77	3.54
966	1001	138.01	1.75	3.50
967	1000	134.68	1.73	3.46
968	999	131.35	1.71	3.42
969	998	128.02	1.69	3.39
970	997	124.69	1.66	3.32
971	996	121.36	1.64	3.27
972	995	118.03	1.61	3.21
973	994	114.70	1.58	3.16
974	993	111.37	1.55	3.10
975	992	108.04	1.52	3.03
976	991	104.71	1.49	2.97
977	990	101.30	1.45	2.90
978	989	98.06	1.42	2.83
979	988	94.73	1.38	2.76
980	987	91.40	1.34	2.68
981	986	88.07	1.30	2.60
982	985	84.74	1.26	2.52
983	984	81.41	1.22	2.44
984	984	78.08	1.18	2.36
985	982	74.75	1.14	2.27
986	981	71.42	1.09	2.19
987	980	68.09	1.05	2.10
988	979	64.76	1.00	2.01
989	978	61.43	0.96	1.91
990	977	58.10	0.91	1.82
991	976	54.78	0.86	1.72
992	975	51.45	0.81	1.62
993	974	48.12	0.76	1.53
994	973	44.79	0.71	1.43
995	972	41.46	0.66	1.32
996	971	38.17	0.61	1.22
997	970	34.80	0.56	1.12
998	969	31.47	0.51	1.01
999	968	28.14	0.45	0.91
1000	967	24.81	0.40	0.80
1001	966	21.48	0.35	0.69
1002	965	18.15	0.29	0.59
1003	964	14.82	0.24	0.48
1004	963	11.50	0.18	0.37

Appendix IV. – P.I.D Parameters

PROP = Proportional Band

INT = Integral Time

DERIV = Derivative Time

** as temperature increases 'Int' and 'Deriv' should be progressively decreased but keeping to a 6:1 ratio

Orange Cryostat

Temp (K)	Prop (%)	Int (s)	Deriv (s)
1 – 5	3	1	0.17
5 – 10	3	10	1.67
10 – 20	1	10	1.67
20 - 300	1	50	8.3

Orange Cryostat (control on the sample)

Temp (K)	Prop (%)	Int (s)	Deriv (s)
1 - 20	2	40	6.7
20 - 50	2	100	16.7
50 - 100	2	200	33.3
150 - 300	2	999	166.5

CCR

Temp (K)	Prop (%)	Int (s)	Deriv (s)
10 – 50	2	50	8.3
50 – 150	2	100	16.7
150 –300	2	200	33.3

RAL Furnace (Foil element)

Temp (Celcius)	Prop (%)	Int (s)	Deriv (s)
20 – 150	16	60	10
150 – 1000	16	30	5
1000 +	16	**	**

Appendix V. – Out Of Hours Support

Normal working hours for most ISIS staff (apart from the ISIS crew who are on shift duty) are from 08:30 to 17:00 (Mon to Fri). Outside these hours most local contacts at ISIS, including many members of the technical support groups, voluntarily agree to provide some form of out-of-hours User support. The first point of call (after this manual) should be the Local Contact for the experiment, assistance being available during ‘reasonable’ hours. The definition of ‘reasonable’ depends upon the individual concerned. However, as a general rule, for local contacts on OSIRIS and members of the technical support groups, the hours between 08:00 and 23:00 would probably be deemed reasonable. Unless it has been agreed that a person may be contacted outside of these hours then the following procedure should be adopted:

i) Check the manual for possible solutions and explanations.

ii) Investigate whether the problem can be put off until a more reasonable time e.g. can the experimental timetable can be adjusted by, perhaps, performing a background or a resolution measurement?

iii) Is a member of the ISIS crew able to assist with the problem?

iv) If none of the above apply ensure that the experimental set-up is safe (the ISIS duty officer in the MCR will advise if necessary) and wait until a more reasonable time. Loss of beam time due to ISIS/OSIRIS/Sample Environment problems is always dealt with sympathetically and, if appropriate, the lost beam time will be is rescheduled at a later date.

Appendix VI. – Useful Telephone Numbers

General:

Accident/Emergency/Fire	2222
Health Physics (Radiation)	6696
ISIS Main Control Room (MCR)	6789
OSIRIS Cabin	6896
Main gate (Security)	5545
Computer support	1763

Office numbers:

Dr Mark Telling	5529
Dr Spencer Howells	5680
Dr Tom Arnold	6088