RAL-92-031

Science and Engineering Research Council
Rutherford Appleton Laboratory
Chilton DIDCOT Oxon OX11 0QX
RAL-92-031

The High Resolution Powder Diffractometer (HRPD) at ISIS - A User Guide

R M Ibberson W I F David and K S Knight

May 1992

THE HIGH RESOLUTION POWDER DIFFRACTOMETER (HRPD) AT ISIS - A USER GUIDE

R M Ibberson, W I F David and K S Knight

RAL-92-031
Version 2.1
May 1992

FOREWORD 2
1. INTRODUCTION 3
2. The High Resolution Powder Diffractometer (HRPD) 5
2.1 Resolution Considerations 5
2.2 HRPD Design 7
2.4 Data Acquisition 13
3. PERFORMING AN EXPERIMENT ON HRPD 14
3.1 Administration Requirements 14
3.2 Safety 14
3.3 Experimental running of HRPD 15
3.3.1 Sample Loading 15
3.3.2 Sample Environment Equipment 16
3.3.3 Sample Tank Evacuation 16
3.3.4 Opening the Beam Shutter 17
3.3.5 Chopper Settings and Control 17
4. DATA ACQUISITION AND INSTRUMENT CONTROL 19
4.1 The Dashboard 19
4.2 The DAE 20
4.3 Instrument Control Commands 21
4.4 The CRPT 22
4.4.1 The CRPT "DEFT" editor 22
5. DATA ASSESSMENT 24
5.1 Data Display and Manipulation - GENIE 24
5.1.1 Using GENIE 24
5.1.1 GENIE Keyboard Commands 25
5.1.2 The Peak Commands 26
6. DATA COLLATION AND REDUCTION PROGRAMS 28
6.1 The FOCUS Commands 28
6.1.1 FOCUS 28
6.1.2 FOC90 28
6.1.3 FOCLA 28
6.2 Normalisation Routines 29
REFERENCES 30
Appendix 1 - Temperature Control and PID Parameters 31
Appendix 2 - Example Instrument Control Command File 34
Appendix 3 - SPECTRA.DAT files 36

FOREWORD

This guide is intended to give a short description of the High Resolution Powder Diffractometer, HRPD, at ISIS and to provide the basic information required in order to perform a routine powder diffraction experiment. This manual supersedes the Introductory Users Guide[1] and for the sake of brevity refers to a number of other manuals where more detailed information may be found.

---

[1]David, W.I.F. *et al*, Rutherford Appleton Laboratory Report, RAL-88-103.

1. INTRODUCTION

The utility of neutrons as a crystallographic probe is a direct consequence of the relative weakness of the fundamental neutron-matter scattering. This and the compatibility of neutron wavelength with inter-atomic distances facilitates the determination of chemical and physical structural information, undistorted by the radiation, using neutron diffraction techniques. The neutron wavelength is related to its energy by the equation

$$\lambda = \left(\frac{h^2}{2mE}\right)^{½} = \frac{9.04}{E^{½}} \tag{1}$$

where λ is in Å and the energy E of the neutron is in meV. The neutron mass, m, is 1.675×10^{-27} kg.

At a pulsed source such as ISIS the universally applied method of determining λ is to measure the time of flight, T , of the neutron over a known flight path, L, from the source (T=0) to sample and subsequently over the scattering path to the detector.

The magnitude of neutron velocity, v, may be calculated as a function of energy and substituted in (1) to yield:

$$\lambda = 0.3955/v = 0.003955T/L \tag{2}$$

where λ is in Å, v is in mμs^{-1} , T is in μs and L in m. The neutron wavelength and its time of flight (TOF) have a linear relationship. The neutron beam at a pulsed source is polychromatic and, using the TOF method, the wavelengths are discriminated by their time of arrival at the detector, facilitating the measurement of different d-spacings at a fixed scattering angle. Bragg's Law may therefore be written as:

$$\lambda_{hkl} = 2d_{hkl} \sin\theta_0 \tag{3}$$

The combination of equations (2) and (3) gives the following relation in convenient units of time in μs and d-spacing in Å:

$$t_{hkl} = 505.55685(40)Ld_{hkl} \sin\theta_0 \tag{4}$$

Thus, for 10m and 100m instruments, a 1Å d-spacing will be detected in backscattering ($2\theta \approx 180°$) with a time of flight of approximately 5000μs and 50000μs respectively.

It should be emphasised that diffractometers, such as HRPD, at pulsed neutron sources operate in a fundamentally different manner from their conventional reactor-based counterparts. Instead of measuring Bragg reflections by scanning a detector from low to high 2θ scattering angles, HRPD uses the pulsed <u>white beam</u> nature of ISIS to measure Bragg reflections at <u>fixed</u> scattering angles, monitoring the time of arrival of the neutron after the initial neutron burst produced in the target.

2. THE HIGH RESOLUTION POWDER DIFFRACTOMETER (HRPD)

HRPD is the highest resolution neutron diffractometer of its type in the world, and is designed to achieve an optimal balance between the maximum attainable practical resolution and reasonable counting times.

2.1 Resolution Considerations

The resolution of a diffractometer, $\Delta d/d$, is a measure of the spread in the Bragg reflection for a given d-spacing, and is of paramount importance in determining the overall quality of a diffractometer. On a pulsed source it has three major contributions: a timing uncertainty, ΔT; an angular uncertainty, $\Delta\theta$ and flight path uncertainties ΔL; which may be combined (approximately) in quadrature.

$$\frac{\Delta d}{d} = \left[\left(\frac{\Delta T}{T}\right)^2 + (\cot\theta\Delta\theta)^2 + \left(\frac{\Delta L}{L}\right)^2\right]^{\frac{1}{2}} \tag{5}$$

The main contribution to ΔT is the moderation time of the neutron. The mechanism of neutron production and moderation at a spallation source is a complicated process and thus it is not surprising that the pulse shape is of a complex nature (Ikeda & Carpenter, 1985). However the finite time width of the initial neutron pulse is clearly independent of T; neutrons of a particular wavelength will propagate non-dispersively (because wavelength and hence velocity are constant) and so the ratio ($\Delta T/T$) is decreased simply by increasing the flight time or total neutron flight path. The error in flight path is similarly minimised and arises chiefly because of the finite moderator thickness (neutrons may be "born" from its front or back) and to a lesser extent due to finite sample and detector sizes. The uncertainty in (half) scattering angle is a consequence of neutron beam divergence and is simply represented by the differentiation of Bragg's Law. For the purposes of this discussion the contribution to the resolution function due to vertical beam divergence have been ignored. In consequence it can be seen that resolution increases linearly with flight path, and the maximum resolution is obtained in backscattering geometry with $\theta=90°$ i.e. $\cot\theta \rightarrow 0$. Moreover, for a given flight path the resolution for a time-of-flight diffractometer is almost constant. This resolution characteristic is extremely important since the extraction of the maximum resolution information is not favoured at one, restricted, high resolution region of the diffraction pattern. The study of phase transitions is greatly facilitated in that all orders of reflection splitting may be observed with equal clarity across the diffraction pattern. Careful consideration of these factors in the design of HRPD, *vide infra*, has realised an instrumental resolution (at backscattering) better than 10^{-3}.

Although the highest resolution is obtained at backscattering where the geometrical resolution contribution, $\Delta d/d = \Delta\theta\cot\theta$, is minimised, in many areas of study it is highly desirable to have substantial detection capacity at lower angles. The recent upgrade of HRPD with the addition of detector banks at 90° and low angles has gone some way to satisfying this requirement.

For constrained sample environments, such as high pressure cells, 90° scattering is optimal: with suitable collimation a diffraction pattern may be obtained without contamination from the cell components. This is illustrated in Figure 1. It should be emphasised that the polychromatic nature of the neutron beam on a time-of-flight diffractometer facilitates a complete diffraction pattern to be obtained at the fixed 90° angle.

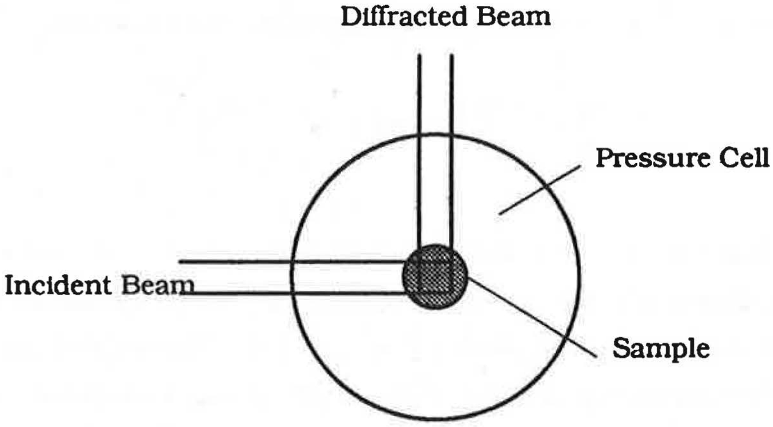


Figure 1 - Optimal collimation for 90° scattering.

Scattering at low angles (20° < 2θ < 30°) allows long d-spacings to be measured: for the same wavelength, the d-spacing measured at 20° is ~6 times larger than that measured in backscattering. The extension to the limit of accessible d-spacings is crucial for cell indexing purposes, the first step in *ab initio* structure determination.

The design of 90° and low-angle (20° < 2θ < 30°) detector banks for HRPD aimed to follow two basic criteria. Firstly, the resolution of both banks should be as high as technically feasible and, secondly, that the count-rates of backscattering, 90° and low-angle banks should be comparable.

At lower scattering angles the resolution becomes dominated by the geometric term, $\Delta\theta\cot\theta$. Both incident and scattered beam contribute to the angular divergence; naturally the resolution is thus optimised by minimising the angular divergence of both incident and scattered beams. On HRPD, the beam incident upon the sample has an angular divergence, $\Delta\theta=0.0017\lambda$, that is determined by the reflectivity properties of the neutron guide. Although for short wavelengths the beam divergence is acceptably small, Soller collimation is ideally required for the longer wavelengths and the use of the low angle bank. The installation of 5' and 10' Soller collimation is planned.

2.2 HRPD Design

The high resolution powder diffractometer, HRPD, at ISIS is approximately 100m in length. The correct moderator design is vital since the ultimate limit to resolution is set by the neutron pulse width $\Delta t(\lambda)$ and neutron flight-time $t(\lambda)$. Preserving the intrinsically sharp proton burst (approximately 0.4μs), is crucial to the maintenance of a "tight" neutron pulse structure, and is achieved on HRPD with the utility of a thin hydrogenous (liquid methane) moderator. The moderator is cooled to 90K to further modify the time structure of the neutron pulse by delaying the onset of thermalisation to energies less than 50meV. Needless to say, the moderator design determines directly the characteristic incident flux available and instrumental peak shape. The action of the moderator is to slow the fast spallation neutrons to suitable energies (wavelengths) for diffraction. The process is complex and involves two disparate physical processes (Taylor; 1982; 1984). Neutrons which escape the moderator in the epithermal region (typically λ<1Å) undergo a slowing-down process without the subsequent thermalisation which produces wavelengths in excess of 1Å. The slowing down and attainment of thermal equilibrium is a dynamic process that involves the collision of neutrons with the hydrogen atoms of the CH_4. The high scattering cross section of ^1H for neutron interactions mean that in such a collision the neutron is likely to lose energy. This mechanism is quasi-continuous and if unchecked would fail to produce a sharp neutron pulse structure as a function of time. At the point of thermalisation a neutron in collision with a hydrogen atom has as much chance of gaining as losing energy. The thermalisation process is controlled by the use of neutron absorbing gadolinium foil in the moderator design. The "poison depth" of the foil determines the time constant beyond which neutrons are absorbed instead of leaving the moderator ultimately for diffraction purposes.

The resultant flux distribution is illustrated in Figure 2. The HRPD peak flux is in the thermal region for neutrons of wavelength approximately 2Å, which corresponds to a d-spacing of about 1Å when observed in backscattering geometry. Whilst this region of a diffraction pattern is of paramount importance for structural studies it is clear that a significant epithermal neutron flux is also available which gives access to sub-Å Bragg reflections. This latter information is crucial in obtaining the high degree of accuracy in structural parameters reported later. Indeed this epithermal neutron flux, coupled with the constant resolution function already described, are perhaps the two main characteristics of a spallation source diffractometer which combine to produce a very potent technique for accurate and precise structural studies.

The 100m flight path of the instrument is necessary to negate the small flight path uncertainty (approximately 5cm) introduced primarily because of the finite moderator depth. However under normal circumstances flux intensity follows a simple inverse-square fall-off with distance, which clearly would reduce the neutron flux to an unacceptable degree over such an extended flight path. The instrument therefore incorporates a neutron guide which has been shown (Carlile *et al*, 1979) to produce a flux comparable to an effective flightpath of approximately 29/λ. Indeed the effectiveness of the guide can be appreciated in that at 1Å and 2Å the HRPD flux is roughly equivalent to 40m and 18m machines respectively. The comparative flux intensities with and without the neutron guide are illustrated in Figure 2. The guide itself is nickel plated glass of cross-section 2.5cm wide and 8cm high along which neutrons travel by total external reflection from the inside walls of the guide. The guide incorporates a curved section (of radius 18km), thus eliminating the direct line of sight from sample to the target so as to exclude potentially problematic high energy short-time γ and fast neutrons which occur every 20ms almost instantaneously with the proton pulse. The extremely shallow radius of curvature permits the transmission of sub-Å neutrons down to approximately λ= 0.5Å.

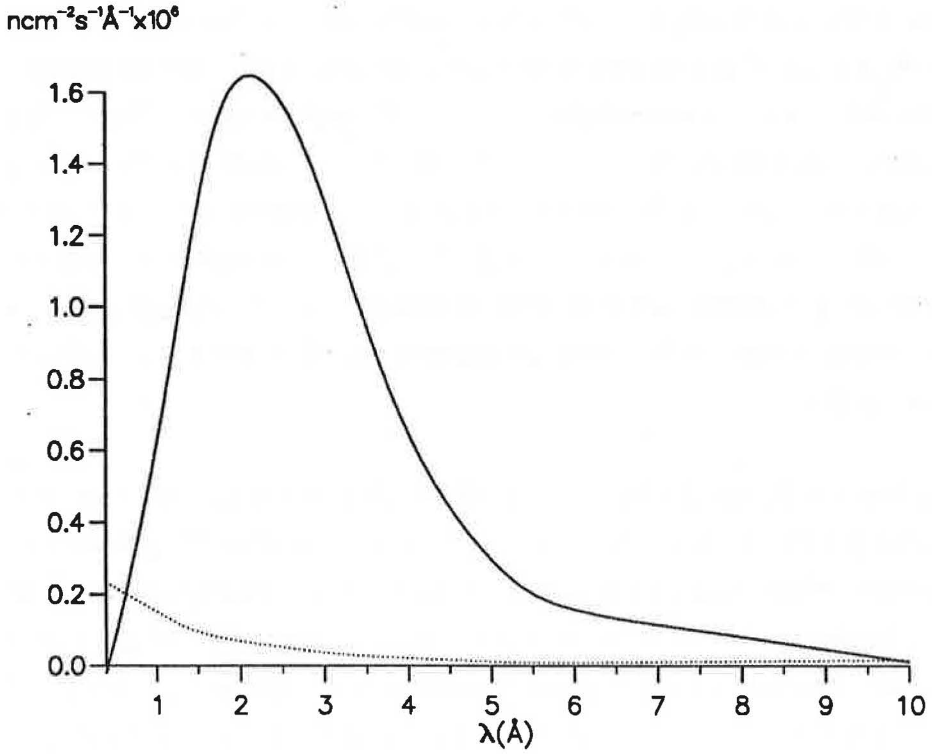


Figure 2 - The incident flux on HRPD and (dotted) the predicted flux without a neutron guide.

Unfortunately despite the ability to time sort a polychromatic beam the whole spectral range of neutron wavelengths illustrated in Figure 2 cannot be utilised simultaneously for a powder diffraction experiment. The long flightpath of HRPD sited on a 50Hz source introduces the problem of "frame overlap" of successive neutron pusles. This is illustrated in Figure 3 by a distance-time diagram describing the dispersion of successive neutron pulses. The pulses occur at A, B, O and C, with OO'-OO" representing the transmitted beam. It should be noted that chopper C2 stops rays AA'-AA", BB'-BB" and CC'-CC" in addition to the first harmonics from A and B (shown as dotted lines). In fact at 50Hz, without the use of such choppers, a wavelength band of only 0.2Å extent could be successfully used over such a flight path.

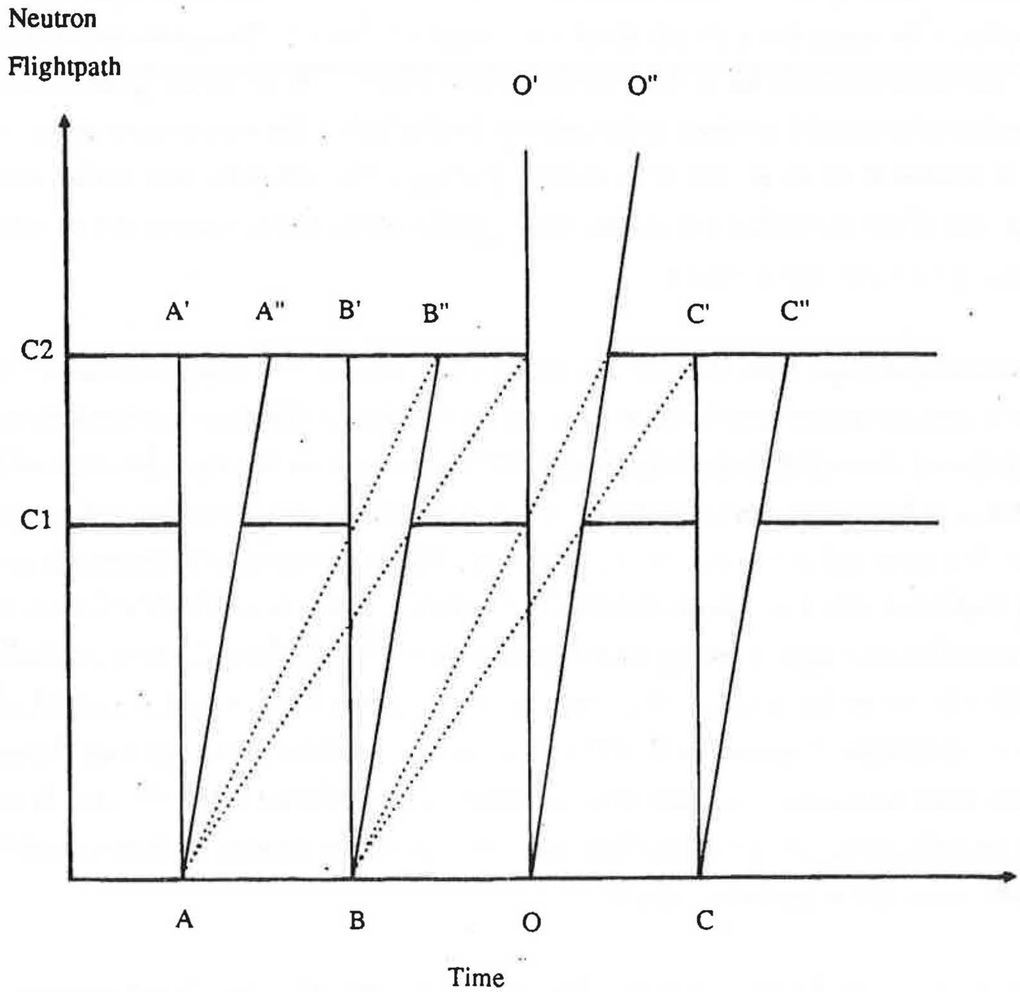


Figure 3 - The elimination of frame overlap by use of beam choppers

The problem of frame overlap is eliminated, as shown in the Figure, by the use of two disk choppers situated at 6(.135)m and 9(.200)m from the moderators. The 6m chopper rotates at ISIS frequency and operates as a wavelength selector: the offset time relative to T=0 may be varied to determine when the disk aperture is open to the beam. The 9m chopper spins at (50/n)Hz (where n=1,2,3,5 & 10) and closes appropriately to prevent frame overlap occurring. Thus the extent of the wavelength window may be increased at a cost of relative intensity (repetition frequency). HRPD typically operates at 5Hz or 10Hz with wavelength "windows" of 4Å and 2Å respectively, corresponding to a d-spacing range in backscattering of 2Å and 1Å respectively. The bounds of this window may intersect the available flux spectrum, as defined by the moderator, in any region.

The diffracted neutron beam on HRPD is currently detected in one of three fixed angle banks: in backscattering; at 90° and at low angles, as shown schematically in Figure 4. The sample may be loaded in one of two positions, either 1m or 2m from the backward bank. Use of the 1m position enables diffraction data to be recorded simultaneously in all three detector banks. The maximum resolution, $\Delta d/d = 4\times10^{-4}$, is obtained at the 2m position in backscattering, $2\theta_{ave} = 174°$,where the $\Delta\theta$ resolution term is minimised. Use of the 1m position provides the experimentalist with a slightly lower resolution, $\Delta d/d = 8\times10^{-4}$, but a four-fold increase in intensity.

The backscattering detector bank comprises six octants of Ce-activated ^{6}Li-doped scintillators. The individual octants are separated into 20 radial strips - thus in total there are 120 discrete detector elements which may be used, de-coupled, as a radially pixelated PSD. By convention, however, these elements are software linked to form twenty (incomplete) rings, designed to mirror the Debye-Scherrer rings produced by powder diffraction, and minimise geometric aberration. The data obtained in backscattering are of inherently high resolution; however the detector has limitations, namely an unwanted sensitivity to γ radiation and an effective upper d-spacing limit of approximately 5Å. This limit is a direct consequence of the incident flux of the diffractometer which peaks at a wavelength of 2Å, but shows a rapid fall-off to a maximum wavelength of approximately 10Å (see Figure 2). In order to measure long d-spacing information, which is crucial in cases such as cell indexing, detectors at lower angles are vital. In these detectors, for a given d spacing the Bragg's Law equation is satisfied by neutrons of shorter wavelength and therefore, on HRPD, of generally higher flux.

The 90° detector utilises ^{6}Li-doped ZnS scintillator which by virtue of its peak height response can discriminate between neutrons and γ radiation. This insensitivity to γ rays is significant when compared to the backscattering detector. The 90° bank comprises 6 modules each with 66 (3mm wide by 200mm high) elements. Each module is positioned on a constant radius from the 1m sample position in the azimuthal plane. As with the backscattering detector, diffraction data may be collected in each of the 396 discrete elements, but more usually the detector is software configured into 66 radial segments.

At low angles the relaxing of resolution requirements permits the use of $\frac{1}{2}$" He^3 tubes. The HRPD low angle bank, $2\theta_{ave}$=30°, currently houses 72 tubes which lie on a constant radius parallel to the through beam direction, and are configured in 3 rows of 24 tubes. Again similar software linking strategies may be applied. The long secondary flightpath of the low angle bank, necessary in order to minimise angular divergence, has required the large tank housing the detector to be filled with Ar gas. The tank is therefore discrete from the other sample and detector tanks which are evacuated during diffraction measurements.

The incident and transmitted beam intensity is monitored by two Davidson (1985) monitors situated at 93.50m and 96.74m from the moderator.

The characteristics of each detector bank are summarised below in Table 1.

	Backscattering	90°	Low Angle
Detector Specification	^{6}Li-doped glass scintillator	ZnS scintillator	$\frac{1}{2}$" 10atm He^3 tubes
Geometry	20 rings: $7 \leq r \leq 37$cm 6 Octants - 3110cm^2	Slab: 20x20cm 66x3mm elements 6 Modules - 2400cm^2	72 tubes (20cm active) 8 tubes/module 9 Modules - 1800cm^2
Fixed Scattering Angle	$160° \leq 2\theta \leq 176°$ (1m) $170° \leq 2\theta \leq 178°$ (2m)	$87° \leq 2\theta \leq 93°$	$28° \leq 2\theta \leq 32°$
Solid Angle (Ω)	0.31 ster (1m) 0.08 ster (2m)	0.08 ster	0.01 ster
Resolution ($\Delta d/d$)	$\approx 8 \times 10^{-4}$ $\approx 4 \times 10^{-4}$	$\approx 2 \times 10^{-3}$	$\approx 2 \times 10^{-2}$
d-spacing range (30-230ms)	≈0.6 - 4.6Å	≈0.9 - 6.6Å	≈2.2 - 16.5Å

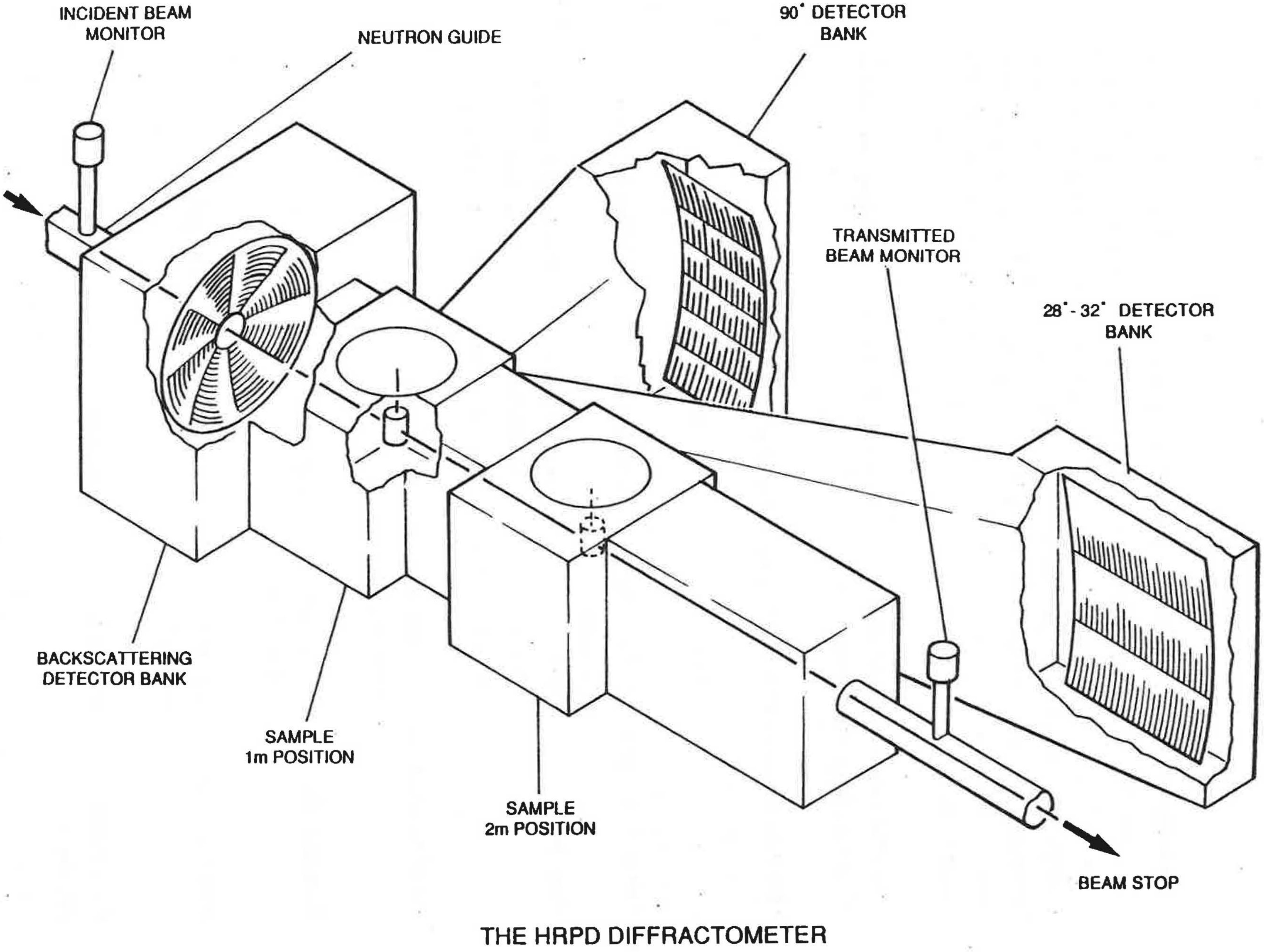


Figure 4 - Schematic view of the HRPD detector configuration

2.4 Data Acquisition

The HRPD instrument control and data acquisition are performed by a Micro VAX 3200 workstation, known as the Front End Microcomputer (FEM). The current instrument settings are contained in a file known as the Current Run Parameter Table (CRPT) and whilst a run is in progress the data is temporarily stored in the Data Acquisition Electronics (DAE). On ending a run the contents of the CRPT and DAE are written to a data file on the FEM which is automatically archived to optical disk, part of the central VAX8650 (HUB) computer system. Ease of file transfer is facilitated by a cluster system which joins the FEM and the HUB via an ETHERNET link.

3. PERFORMING AN EXPERIMENT ON HRPD

3.1 Administration Requirements

HRPD is located in R69, outside and south of R55, the main experimental Hall. On arrival users should first fulfil the following requirements before commencing their experiment:

- Register with the University Liaison Secretariat, UG3 R3.

- Register with the ISIS Main Control Room (MCR) in R55 to receive temporary film badges and a "Permit to Work". Both R69 and R55 are restricted radiation areas and therefore Health and Safety requirements necessitate the wearing of film badges.

- Get in touch with the Local Contact and obtain the experiment "Sample Record Sheet" (part of the original experiment proposal). The Sample Record Sheet contains a safety assessment of the experiment which cannot be started until the record sheet has been obtained.

After the experiment all users are required to complete an Experiment Report. The A3 report should be completed within three months of the experiment. Report forms are available from the University Liaison Secretariat or Instrument Scientists.

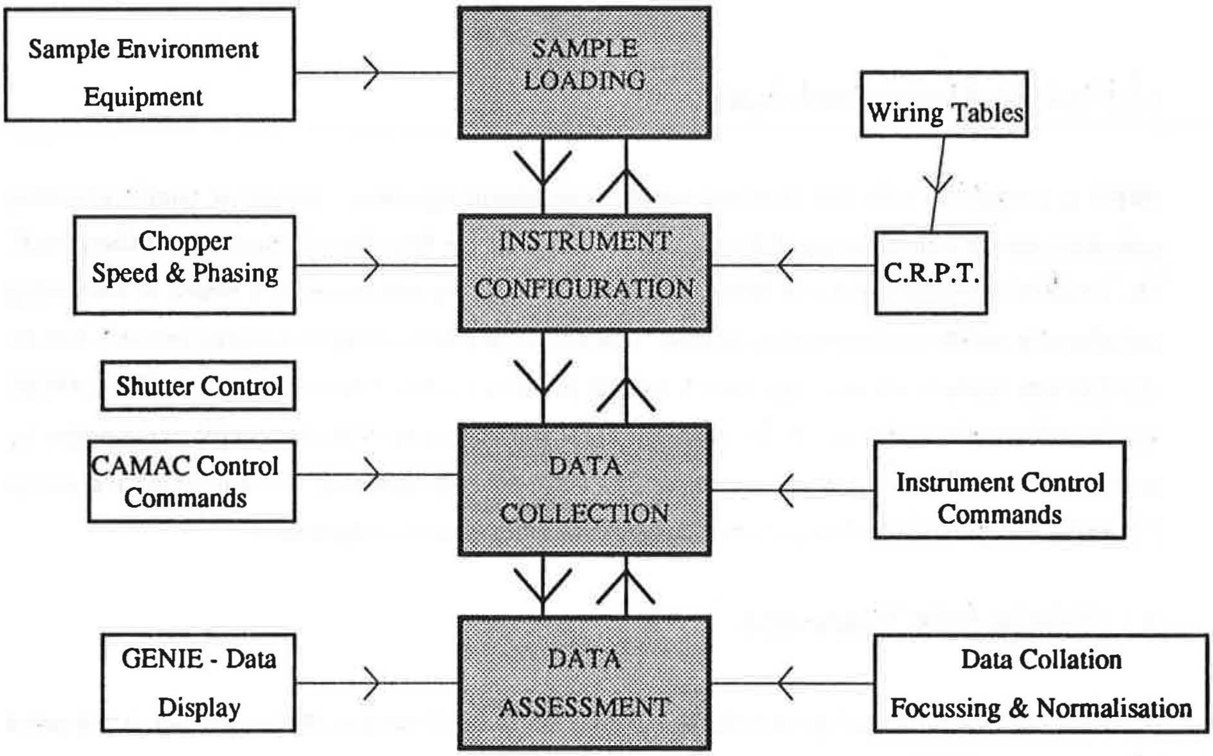
3.2 Safety

The University Liaison Secretariat will issue all users with a number of safety documents on registration. These must be read before beginning an experiment. In addition, all users must ensure they discuss with an instrument scientist in advance the particular hazards associated with the instrument and the approved procedure for performing the experiment.

The appropriate sample record sheet should be displayed on the door of the HRPD interlocked area for the duration of the experiment. The form contains infromation on chemical and radiological hazards associated with the sample as assessed by the RAL Safety Section. Any recommendations concerning sample handling both before and after irradiation must be adhered to. All samples run on HRPD must be monitored by the ISIS Health Physics Group (x6696) before being removed from R69.

3.3 Experimental running of HRPD

The following sections deal with the various aspects of instrument configuration and data collection strategy for the majority of powder diffraction experiments. The main stages are summarised below:



3.3.1 Sample Loading

The advent of multi-angle detector banks on HRPD has led to standardisation in favour of cylindrical vanadium sample cans. These "deep drawn" all vanadium cans have standard diameters of 5, 8, 12 and 15mm and may be hermetically sealed. The beam height is typically 25mm. Cans of slab geometry, with volumes of 1 to 6cm^3 may also be used. These cans consist of an aluminium body with thin vanadium windows attached by aluminium window frames and are not easily sealed. It is also necessary to mask the aluminium portion of the can using Gd foil.

Since an increase in sample volume will, in general, decrease the required counting time, as large a sample can as possible should be used. The exceptions to this general rule are with hydrogenous samples, where sample can area should be increased at the expense of thickness, or with strong neutron absorbing samples.

Sample cans are attached to the centre stick via an M4 fitting. The beam height is 300mm from the base of the top plate ('Tomkinson flange') of each sample position. The distance from the lowest aluminium flange on the ambient temperature centre stick lid to the centre of the sample can (ie beam centre) may be adjusted to 320mm (allowing 20mm for the top plate thickness) using the precision setting jig.

3.3.2 Sample Environment Equipment

HRPD is compatible with ISIS standard sample environment apparatus. Details of sample mounting procedures are outwith the scope of this manual and are available from Sample Environment Group staff. The CAMAC (Computer Assisted Measurement And Control) system provides a means of monitoring and adjusting sample environment parameters. Full details on CAMAC can be found in section 5.2 of the PUNCH user manual, see also Appendix 1 for PID parameters for temperature control. The CAMAC system enables data collection to be automatically controlled along with the sample environment by means of DCL (Direct Command Lanuage) files. An example command file illustrating the use of CAMAC in conjunction with instrument control commands is given in Appendix 2.

3.3.3 Sample Tank Evacuation

In order to reduce air-scattering, and usually with all sample environments, the sample tank is evacuated before commencing data collection. Valves are located at the far side of the raised platform alongside the 90° detector tank. A rotary and 'booster' pump are situated inside the guide tunnel that run continuously; on-off switching is not required. To evacuate the sample tank, fully close the valve labelled "back-to-air" then slowly open the valve above it connected to the pump. After some 30s this valve may be opened fully. To let the tank up to air the latter valve is first closed before slowly opening the "back-to-air" valve. The detectors may be damaged by rapid changes in pressure inside the sample tank. Letting up to atmosphere takes some twenty minutes. The state of the sample tank vacuum can be checked using the Pirani and Penning vacuum gauges located in the rack near the instrument console. The turbo pump situated alongside the sample tank should remain switched off unless pressures of $<10^{-6}$ torr are required, normally only when using a closed cycle refrigerator (CCR).

No attempt should be made to remove samples until the sample tank is at atmospheric pressure. Users are reminded that when removing samples after irradiation they must follow the safety regulations concerning monitoring of induced β and γ activity and the transferral of powders from cans.

The low angle detector tank is Ar filled and gas continually enters this tank from pressurised cylinders.

3.3.4 Opening the Beam Shutter

An interlock mechanism is in operation to prevent the shutter from being opened while the door that gives access to the instrument, and thus potentially the neutron beam, is open. Under normal operations only the interlocked area surrounding the instrument platform need be considered. On leaving the area the door is closed and the key (an "S" key) removed and inserted in the uppermost key press to the right of the door. Assuming this completes the full complement of 8 S keys, the tagged master ("M") key can be removed and located in the green shutter marshalling box alongside. The master key should be inserted and turned through 90° in a clockwise direction. The user should then check the four red LEDs on the control box are lit, indicating all interlocks are closed. Only when this state is reached may the shutter be opened via the remote control box. If interlock problems are encountered the user should immediately contact an instrument scientist or the ISIS control room (x6789).

The beam shutter is operated by pressing the appropriate "OPEN" and "CLOSE" buttons on the control unit, both operations take approximately one minute. Once the shutter is open the master key cannot be removed from the control box and there is no access to the interlocked area. On closing the shutter the key sequence is reversed to gain access to the instrument.

3.3.5 Chopper Settings and Control

Two disk choppers are situated at 6m and 9m from the moderator. The chopper settings may entered using the CAMAC system, however values can also be entered manually on the crates in the appropriate rack beneath the instrument platform.

Setting of the disk rotation speeds determines the "width" of the wavelength pulse incident on the sample. HRPD conventionally runs at either 10Hz or 5Hz to give the widest wavelength range in the diffraction pattern. Permitted settings are tabulated below:

Chopper Speed (Hz)		Pulse Width		Relative Beam
6m	9m	(ms)	(Å)*	Intensity
25	5	200	4.0	0.1
50	10	100	2.0	0.2
50	25	40	0.8	0.5
50	50	20	0.4	1.0

* - d-spacing

Rotation speeds can only be changed once the choppers are at rest. Stop and start buttons on the control rack allowsmanual resetting. When stationary, as shown by the rotor speed indicator, the desired speed may be selected using the dial on module 1 and then pressing the "enter data" button. It can take up to 15 minutes for the choppers to correctly phase (the LED error lights will go out) upon restarting.

The chopper phasing determines the starting time of flight of flux "pulse width" selected above. The required time offsets are set using CAMAC. The commands are:

$ CPHS100 <start_time>

and

$ CPHS200 <start_time>

for a 100ms and 200ms pulse widths respectively.

4. DATA ACQUISITION AND INSTRUMENT CONTROL

HRPD is controlled using the Vax 3200 workstation; instrument commands are issued from a "supervisor" process (or window) from which the instrument "dashboard" is normally displayed. The supervisor process is usually run from the [HRPD] account.

4.1 The Dashboard

The instrument dashboard shows details of the current run and displays the DAE mode (see section 4.2). The commands

$ STAT ON

and

$ STAT OFF

turn the dashboard display on and off.

The dashboard information is generally self-explanatory. As data are collected both "frame" count figures, μA.hr total and the monitor count value should all increase. It should be noted that the figure given for the instantaneous proton current corresponds to the effective current for the current HRPD chopper configuration. Therefore an ISIS current of 200μA will be shown as 40μA when running the instrument at 10Hz.

A frame is the period between ISIS neutron pulses. On HRPD the beginning of each frame is defined by two synchronisation (synch) pulses, one from the protons from the synchrotron and one from the 6m chopper. A "good" frame, during which data is added to the DAE, requires the trigger from both these pulses. On HRPD a "raw" frame is defined solely by the chopper synch pulse, so that an incrementing raw frame count serves only to indicate that the 6m chopper is running. Data collection is suspended if the frame is not good. Frame vetoes may arise due to a number of reasons:

i) ISIS beam off

ii) HRPD beam choppers stopped or not at requested phase or speed

iii) Methane moderator temperature outside pre-set limits

iv) DAE faults

The command

$ VETO

may be invoked to interrogate the data acquisition system as to which vetoes are causing the suspension of data collection. The following vetoes are currently enabled:

i) FRAME, PPFM and FIFO which refer to the data acquisition electronics

ii) CHOPPER which represents the ISIS synchronisation pulse and thus will register when the beam trips off

iii) EXTNVET0 for the chopper synchronisation pulse

iv) EXTNVET1 which checks for the moderator temperature

The displayed monitor count normally refers to the upstream monitor. The counts figure is the integral between the limits shown.

4.2 The DAE

The DAE (Data Acquisition Electronics) governs data collection for each individual run. It correlates parameters from a number of files but it should be necessary only to edit the CRPT (see section 4.4) before commencing a run.

There are four possible modes of the DAE :

SETUP	No data recording taking place, parameters may be changed prior to beginning a run.
RUNNING	Data recording in progress.
PAUSED	Data recording temporarily suspended due to an interactive user issued command.
WAITING	Data recording temporarily suspended due to an out of range parameter in a CAMAC sample environment block.

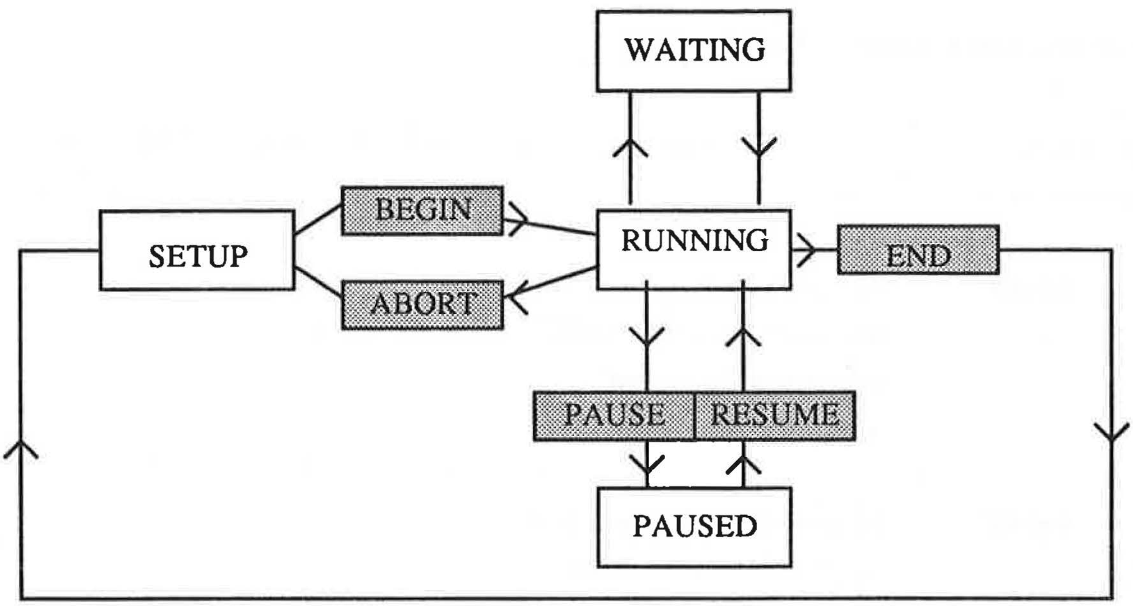
DAE mode and the current run status are displayed on the dashboard.

4.3 Instrument Control Commands

Data collection is controlled using five basic instrument commands which must be issued from the "supervisor" process.

BEGIN	sets DAE memory to zero sets parameters in DAE to those specified by CRPT sets DAE to acquire data sets DAE mode to RUNNING
PAUSE	suspends data collection by DAE sets DAE mode to PAUSED
RESUME	resumes data collection by DAE from a PAUSED state sets DAE mode to RUNNING
END	stops data collection copies DAE memory and CRPT to HRP"Run no.".RAW dashboard run number is incremented sets DAE mode to SETUP
ABORT	stops data collection no data is saved sets DAE mode to SETUP

The inter-relationship between the DAE modes and instrument commands is illustrated in the flow-diagram below. (Instrument commands are shown shaded).



4.4 The CRPT

The CRPT (Current Run Parameter Table) file acts as an intermediary storage file between the DAE and the final stored raw data file. In addition, it contains the settings of various run parameters. It is normally necessary to edit the run parameter section of the CRPT prior to each experiment. The CRPT may be edited using the command CHANGE when the DAE is in SETUP mode.

$ CHANGE <CR>

presents the user with the appropriate pages of the current CRPT, values are altered using full screen editing techniques (DEFT), full details of which are contained in section 5.1.4 of the PUNCH manual. The cursor keys (⇑ ⇓) allow movement between the various fields, when the field prompts with "toggle data type" the "." key on the right hand key-pad should be used. All other fields are altered by typing appropriate numbers or characters into the field. The cursor keys (⇐ ⇒) allow corrections to be made by overstrike. To exit and update the CRPT with the new values hit <GOLD> E. To quit and leave the CRPT as it was, hit <GOLD> Q. The <GOLD> key is "F1" on the right-hand keypad.

4.4.1 The CRPT "DEFT" editor

The information relating to run parameters in each field is shown in reverse-video. Only the first three pages of the file should be routinely altered.

Page 1 : All fields are self-explanatory.

Page 2 : The range of the spectrum is set to correspond to the chopper settings. The method of deriving the time channel boundaries is usually to fill in the fields shown on the page. If there is insufficient space, for example with a very complex spectrum construction, a separate file, TCB.DAT which defines the boundaries, is set up. By entering the file name on the first line, the time channel boundaries are set by reading the file only. Steps of 0.0001 in mode dT/T=C give logarithmically varying time channel boundaries of instrument resolution. This may be assumed as the default setting for the highest resolution studies. Many samples however are intrinsically line broadened and so a value of 0.0002 or greater could equally well be used, giving the advantages of a reduced memory requirement and increased speed of data manipulation. The constraints imposed by 8Mb maximum memory can be encountered when using all three detector banks.

The Status Display Monitor corresponds with the monitor count information shown on the dashboard. Spectrum 21 corresponds to the upstream monitor and is used in most configurations. The displayed number of counts on the dashboard is the integral between the two limits set in the CRPT. This region is generally placed somewhere mid-way in the spectrum.

Page 3 : All fields are self-explanatory.

The remaining pages under normal circumstances remain unchanged. The software detector configuration is specified on page 5 by the SPECTRA.DAT file. The available options are given in Appendix 3.

During the course of the experiment some simple alterations to the CRPT can be made without using the DEFT editor. These are typed to the supervisor process when the DAE is in SETUP mode. Most useful is:

$ CHANGE TITLE C6D6_2m_T=5K

Note that the title must be a continuous character string and that some characters such as +, (,), / are not accepted.

5. DATA ASSESSMENT

Full analysis of HRPD data is possible using either the HRPD 3200 or the HUB computer from a local or remote terminal. It should be noted that inspection of data of a current run is only permitted if the user is logged on as [HRPD] on the FEM computer. Outside of this special case, data can be inspected from the user's own account following the installation of a suitable login procedure. All data display and reduction programs simply use the run number for reference. The raw data files must, however, be on disk. Recent data sets are stored in HRPD$DISK0:[HRPMGR.DATA] whereas older data may need to be restored from optical disk using the command:

$ RESTHRPD

The program prompts for run number, year of collection and cycle number. Data are automatically restored to SCRATCH$DISK:[HRPMGR.RESTORE]. All programs search for the required run number in both these areas, which are defined by the logical name HRPD_DATA.

5.1 Data Display and Manipulation - GENIE

GENIE is a data display and manipulation software package that is standard for all instruments at ISIS. A full explanation of GENIE is available in the Punch User Guide or the Punch GENIE Manual Version 2.3. The GENIE program allows modification of experimental spectra, such as the addition of spectra and major features such as model fitting and external user-definable functions which are of particular use with HRPD data This section deals primarily with the display and initial stages of analysis of HRPD data. Keyboard commands typed to the screen will be written in full, with the shortest abbreviation shown in bold and capitalised.

5.1.1 Using GENIE

GENIE may be accessed using the HRPD FEM or the HUB computer. The GENIE program is run as follows :

$ GENIE
>>.........(from this prompt GENIE commands may be issued)

The program contains a number of general WORKSPACES, a GRAPHICS WORKSPACE and a number of BUFFERS. In each work-space data relating to a single spectrum may be held. Data are read as arrays of x, y and e (ordinate, observation and error) values plus other parameters necessary for data interpretation and manipulation.

Running the GENIE program initialises a series of default settings concerning the location and subsequent retrieval of raw data files for inspection. Thus to assign a file to all work-spaces only the relevant run number and not its full location need be entered, for example

>> **ASS**ign **999**

It should be noted that only when logged on to the FEM may :

>> **ASS**ign **DAE**

and

>> **ASS**ign **CRPT**

be used to allow direct access to the current run data in the data acquisition electronics (DAE) and the current run parameter table (CRPT). An individual spectrum from such locations is added to a workspace simply with the command:

>> **Wn = Sn**

Alternatively data from suitable produced GENIE "intermediate" files, for example .FST, .NOR and .COR files described below, may be read into a workspace:

>> **RE**ad/**OP**en **Wn <filename>** <CR>
>> **RE**ad/**CL**ose <CR>

5.1.1 GENIE Keyboard Commands

For quick reference some of the basic GENIE commands are summarised below. The command is written in full with the shortest abbreviation in bold capitals. In each case hit <CR> to return to a ">>" prompt from the graphics screen.

Alter Binning: A bin grouping of 1 results in the display of every data point. (The default value is 10.)
>> **A B 5** only every fifth data point is displayed.

Cursor[2]: Switches on graphics cursor, hit **E** to exit. When the cursor is in operation (use ⇒⇐⇑⇓ keys) single character commands may be typed. **X** displays the x-co-ordinate, **Y** displays the y-co-ordinate and **P** displays x- and y-co-ordinates.

Display: Displays spectrum in a specified workspace.

HC: For laser print hard copy.

EXit : To leave the GENIE program.

Limits: To set new limits for the graph displayed.
L/X : To set x-limits eg. >> **L/X 50000 70000**
L/Y: To set y-limits eg. >> **L/Y 0 250**
L/D : Resets limits to xmin, xmax, ymin and ymax. (**L** <CR> will default to these settings).

Unit : To modify the units of a specified workspace.
U/D : converts to d spacing in Å.

Zoom: To blow up a selected portion of the plot. Opposite corners of the required area are defined by positioning the cursor and hitting any character key[3].

5.1.2 The Peak Commands

A variety of peak fitting routines is available, all of which are run using the PEAK command. The command may only be used following the display command:

>> **PEak Xmin Xmax** <CR>

---

[2]When using a workstation the cursor commands are menu driven using the mouse.

[3]On a workstation opposite corners of the area are defined using the mouse and clicking the left hand button.

The Xmin, Xmax limits may be set on the command line, however the default is to return the graphics cursor and allow on screen selection by hitting first <CR> then "L" or "U" to mark lower and upper limits respectively. When using a workstation the limit selection becomes menu driven and the mouse is used. The default peak command fits a linear background returning 0th, 1st and 2nd moments of the remainder. To use another routine simply insert the program name and its location on the command line, for example:

>> PEak G:GEC Xmin Xmax <CR>

Notes on the programs available are given below in Table 3.

Program Name	Modelled Peak Function
G:GEC	Gaussian + exponential
G:CFVAN	Ikeda-Carpenter + Voigt
G:CGVAN	Ikeda-Carpenter + Gaussian
G:VOIGTVAN	Voigt

Table 3

It should be noted that the peak fitting routines which include the Ikeda-Carpenter function may only be used on data displayed as a function of time of flight.

6. DATA COLLATION AND REDUCTION PROGRAMS

6.1 The FOCUS Commands

Command files are set up to focus HRPD data, that is to produce one average summed spectrum from the individual elements of each detector bank configuration. The programs may be run from GENIE or outside in the normal VAX/VMS environment. Running the focus programs creates GENIE intermediate files of the name HRP'Run_number' with extensions .FST, .FST90_n (where n is a module number) or .FSTLA depending on whether the data are from backscattering, 90° or the low angle detector bank respectively. Focused data from the DAE is written to a file of name HRP00000.*, where * is FST, FST90_n or FSTLA.

6.1.1 FOCUS

FOCUS is run to sum the 20 backscattering spectra. The program prompts for a run number, whether the 1m or 2m position is used and for a GENIE workspace number. DAEFOCUS is used to look at the current run.

6.1.2 FOC90

FOC90 is run to sum the spectra collected at 90°. The program prompts for a run number, 90° bank module number and workspace number. If all six modules are software linked, giving a total of 66 spectra at 90°, module number 1 should be used. DAEFOC90 is used to look at the current run.

6.1.3 FOCLA

FOCLA is run to sum the spectra collected in the low angle detector bank. The program prompts for a run number, workspace number and the number of discrete low angle spectra and first spectrum number of the low angle data. Conventionally, 24 individual low angle spectra are collected beginning with spectrum number 89 (see Appendix 3). DAEFOCLA is used to look at the current run.

6.2 Normalisation Routines

Standardised normalisation routines are currently available only for data collected in backscattering and at 90°. The procedure involves two stages:

i) correction for the incident flux distribution by division of each backscattering or 90° spectrum with a suitable monitor spectrum;

ii) correction for detector efficiency by division of a corrected and flux normalised vanadium spectrum.

The first step is achieved running program NORM.

$ NORM

The program incorporates options to allow manual or automatic "flat background" subtraction (the result of detector quiet counts and γ response) and similarly for the final spectrum limits. Users are reminded that data at the extremities of the raw spectrum should not normally be included. In these regions the chopper apertures defining the window are not fully open, thus the data is necessarily of a lower statistical quality. The program individually treats all 20 spectra in backscattering, summing them to the file HRP'run_number'.NOR, which is of GENIE intermediate file format.

Correction for detector efficiency is achieved using program VA_COR:

$ VA_COR

The program prompts for the run number of the normalised file and a file containing the corrected vanadium spectrum information. The files GD:VAN1M.SPL, GD:VAN2M.SPL, GD:VAN90.SPL and GD:VANLA.SPL should be used for 1m, 2m position 90° andlow angle data respectively. The program output is to file HRP'run_number'.COR which is of GENIE intermediate file format. The .COR file may be read into GENIE and written out in ASCII format for use with profile refinement programs described elsewhere.

REFERENCES

Carlile C J, Johnson M W, Williams W G (1979) Neutron Guides on Pulsed Sources. Rutherford Appleton Laboratory Report 79-084.

Davidson P L (1985) Thermal neutron beam line monitor. Rutherford Appleton Laboratory Report 85-032

Ikeda S., and Carpenter J.M., Nucl. Instrum. Methods A**239**, (1985), 536-544.

Parry R G ed. (1990) PUNCH User Manual, Rutherford Appleton Laboratory Report 79-084.

Taylor A D (1982) Moderated neutron pulse shapes. Argonne National Laboratory Report 82-80.

Taylor A D (1984) Moderator performance predictions. Rutherford Appleton Laboratory Report 84-120.

Appendix 1 - Temperature Control and PID Parameters

Of the many parameters that can be varied in the Eurotherm temperature controller, five are important to enable efficient temperature control. In order of importance they are Proportional Band (P), Integral Time (I), Derivative Time (D), Cycle Time and Power output. A description of their functions is beyond the scope of this manual, therefore only brief notes and some values of PID for certain common situations are given.

It is suggested that the cycle time remain at its minimum setting of 0.1s. It is also generally recommended that the ratio of I:D should be ≈5:1 and that P be in the range ≈1.5-5% . The required PID values are best entered using the CAMAC system as described elsewhere, but it is also possible to enter values direct to the Eurotherm. The format of the command to change a parameter is:

$ CSET <parameter_name> <value>

The values that can be changed are given below. Values are qualified with "1" to specify address 1 of the Eurotherm rather than address zero which is default.

Parameter name	Description	Units
PROP(1)	proportional band	percentage points
INT(1)	integral time	seconds
DERIV(1)	derivative time	seconds
MAX_POWER(1)	maximum output power	percentage points
CYCLE	cycle time	seconds
TEMP(1)	set-point value	K, °C or mV

The subsequent PID values serve as guide-lines - the values are strongly sample and "setup" dependent. The heater power setting is also critical. The MAX_POWER setting should be used to avoid overshooting a required set point particularly, at the low temperature range of either a cryostat or furnace.

Orange Cryostat - directly heated sample

T(K)	P(%)	I(s)	D(s)
1-5	3	1	0.2
5-10	3	10	2
10-20	1	10	2
20-300	1	50	10

Orange Cryostat - cryostat heating

T(K)	P(%)	I(s)	D(s)
1-5	3	1	0.2
5-10	3	10	2
10-20	1	10	2
20-300	1	50	10

CCR (bottom loading)

T(K)	P(%)	I(s)	D(s)
20-50	2	50	10
50-150	2	100	20
150-300	2	200	40

RAL Furnace

T(°C)	P(%)	I(s)	D(s)
20-150	16	60	12
150-1000	16	30	6
> 1000	16	*	*

* as the temperature increases the time constants should be progressively decreased

Appendix 2 - Example Instrument Control Command File

The example shown below illustrates the use of a command file to control a sequence of runs as a function of temperature. The command file is implemented as in standard DCL by typing:

@ FILE_NAME.COM

The command file control is halted by typing "<cntrl> y " (interrupt) which will return an interactive prompt leaving the instrument in its current state. On restarting a command procedure the file should first be edited to remove completed steps: the command sequence does not begin from the point last reached!

An exclamation mark (!) may be used to comment the command file and specifically to comment out those commands already executed.

```
! EXAMPLE.COM
$ WSO:==WRITE SYS$OUTPUT                       ! define WSO to write commentary to screen
$ CSET PROP 1
$ CSET INT 50
$ CSET DERIV 10
!
$ ITEMP=50                                     ! set variable ITEMP
$ LOOP:                                        ! Label (note ":")
$ CSET TEMP/RANGE=2/CONTROL 'ITEMP'            !request set point as "ITEMP"±1K
$ WSO "Waiting 5 minutes for temperature equilibration"    !commentary
$ WAIT 00:05:00
$ CHANGE TITLE Sample_name_T='ITEMP'K
$ BEGIN
$ WSO "Waiting for 100uAhr T=",'ITEMP'," K"
$ WAITFOR 100 UAMP                             ! run duration set as 100µAhr
$ END
$ ITEMP=ITEMP+5                                ! increment ITEMP by 5
$ GOTO LOOP                                    ! continuous run loop set up
```

It may be required to change the time of flight range over which a spectrum is collected; this may also be performed automatically. The chopper phasing may be set from within the command file using the commands described in section 3.3.5. The CRPT may be automatically updated by loading an appropriate file created using the command NEXTRUN:

$ NEXTRUN FILE_NAME.DEF

The command invokes the CHANGE command, however the updated values are written to a file. This file may be loaded to the CRPT at the appropriate stage of a command procedure. For example:

$ WAITFOR 100 UAMP	! run duration set as 100μAhr
$ END	
$ CPHS100 90	!change chopper phasing to begin at 90ms
$ WAIT 00:02:00	! wait 2 minutes for re-phasing
$ LOAD FILE_NAME.DEF	!load file created using NEXTRUN
$ BEGIN	

Appendix 3 - SPECTRA.DAT files

The software linkage and configuration of the numerous detector elements which currently comprise the three detector banks are defined and specified in the CRPT by three files:

i) WIRING.DAT
ii) DETECTOR.DAT
iii) SPECTRA.DAT

The wiring and detector files **should not** be changed. These files determine an index for each of the detector elements. Currently in use are the files:

WIRING.DAT_710
DETECTOR.DAT_710

The 710 elements consist of: 240 in backscattering; 2 beam monitors; 396 at 90°; 72 in the low angle bank. Each of these elements is assigned to a spectrum number in the SPECTRA.DAT file. A number of commonly used configurations are described below.

SPECTRA.DAT_22

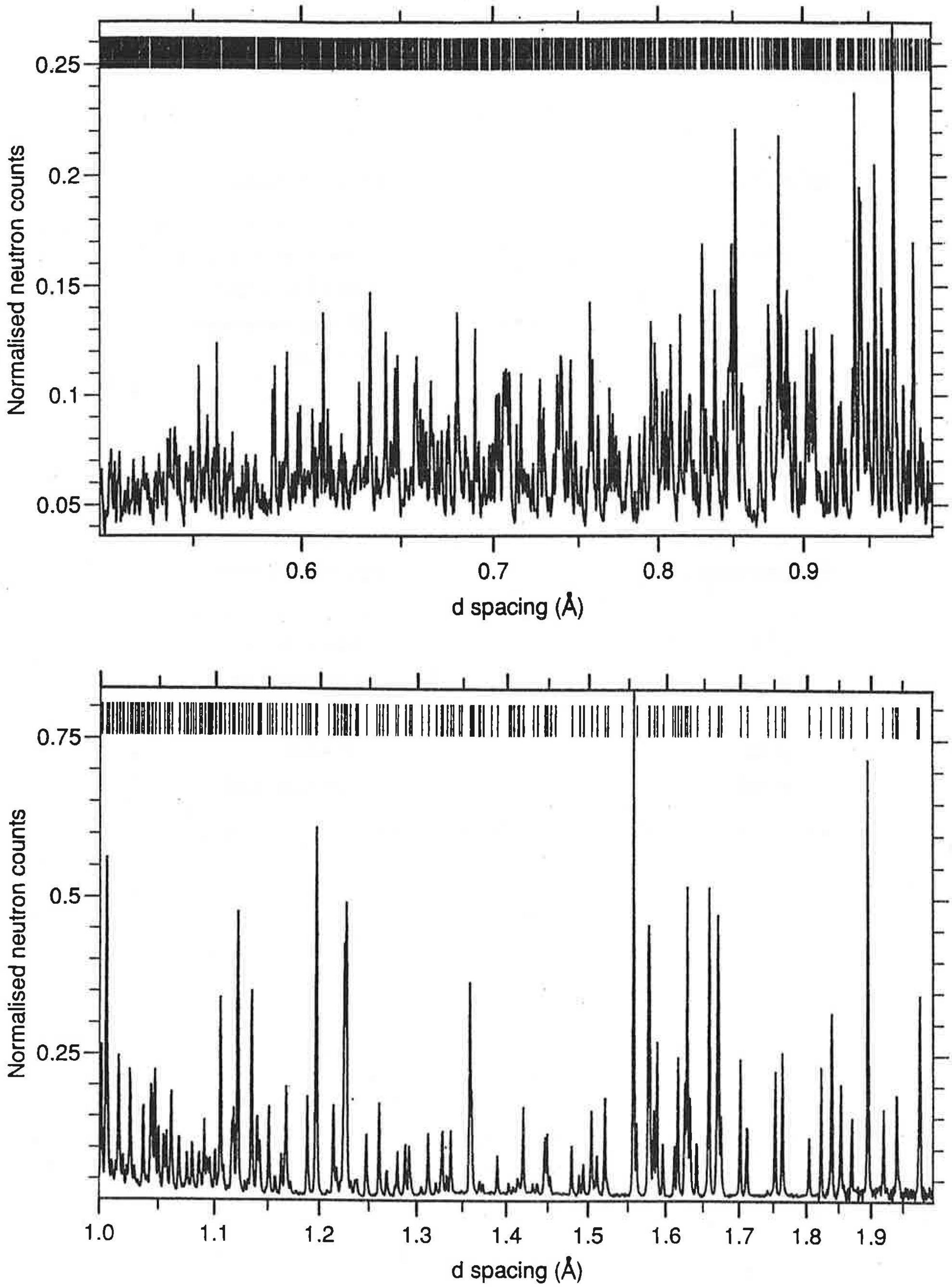
Spectrum Number	Detector Assignment
1-20	Backscattering
21	Upstream monitor
22	Downstream monitor

SPECTRA.DAT_88

Spectrum Number	Detector Assignment
1-20	Backscattering
21	Upstream monitor
22	Downstream monitor
23-88	90° Bank

SPECTRA.DAT_112

Spectrum Number	Detector Assignment
1-20	Backscattering
21	Upstream monitor
22	Downstream monitor
23-88	90° Bank
89-112	Low Angle Bank



An example HRPD diffraction pattern: Benzene (C_6D_6). (Data were collected over the TOF range 12-112ms which correspeonds to ≈ 0.24-2.24Å)