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SRS Station Guide

Station 2.3 Manual

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SRS Station Guide

Station 2.3 Manual

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Abstract

The objective of the manual is to effectively provide assistance to users so that they can perform successful experiments at station 2.3 during their visits. In order to compile a comprehensive document, the functions of the instrument hardware and software are described in detail. Where appropriate it also contains useful information and other documentation for help and/or reference. In addition, suggestions and instructions are available to overcome problems which inevitably face the users as the instrument is quite advanced in the performing of complex experimental tasks. This document can provide help as part of the overall user support facility and it is therefore intended that the manual is readily available in hardcopy as well as in electronic form.

1. General Introduction

1.1 Introductory Remarks

The instrument described in this manual is one of many machines which are available for users in the laboratory. The main modes of operation in station 2.3 are high resolution powder diffraction and high flux surface reflectometry. The users are from academia and industry who are working in many fields of science including chemistry, engineering, earth science, physics, material science, pharmacy and industry. The technology of the instrument is constantly improved so that it can maintain a powerful microscopic tool for modem scientific studies. This document is therefore produced as a part of an integrated program to provide effective support to our diverse community of users.

The layout of this manual is that the remainder of the present chapter provides a list of related documents and the support personnel. The technical introductory and operational modes or diffraction geometries are presented in chapter 2. The instrument hardware is in chapter 3 and also in chapter 4. The description of the control software and the instructions of data acquisition are in chapter 5. A brief account of the data analysis facilities is given in chapter 6. Chapter 7 provides important instructions of experimental hutch interlocking and opening of the x-ray shutter. Finally, other useful informative details such as trouble shooting, glossary, references and appendices are also included.

1.2 Intended Readership

New users of station 2.3 are strongly recommended to read the manual before the actual experimental time and refer to it whenever they have difficulties with the instrument. The contents are often updated since the hardware and software of the instrument is constantly improved or upgraded. As a result, the technical information in this document can provide helpful guidance to new users as well as the experienced workers.

1.3 Related Documents

- *Manual for Multipattern Rietveld Refinement Program,* A.N. Fitch and A.D. Murray, December 1989.
- *A Two Circle Powder Diffractometer for Synchrotron Radiation with a Closed Loop Encoder Feedback System.* R.J. Cernik, P.K. Murray, P. Pattison and A.N. Fitch, December 1989.
- *User's Guide to Daresbury X Terminal Server,* R. Whittington. August 1993.
- *Getting Started on xrdsv1.* C.E. Dean and M.C. Miller, November 1993.

- *PINCER Data Acquisition System Guide*, M.C.Miller, August 1995.
- *PINCER Data Acquisition User Guide*, M.C. Miller and C. Marshall, September 1995.
- *PINCER Configuration Guide*, M.C. Miller, November 1995.
- *System Information for SM9497 (Mclennan Servo Supplies Ltd)*
- *Enhanced Dynamic Range (EDR) Detector (Bede Scientific Instruments Ltd)*
- *CCP14 Manual -Version 2.0*, A.J. Holland, March 1996.
- *Instructions to Experimenters for Operating the Beamline Control Terminals.* C. Morrell
- The above documents are available in the station

1.4 Getting Help

The following staff can be called upon for technical assistance:

Table 1. Supporting Staff

Position	Name	Room	Phone	Bleep
Station Scientist	Chiu Tang	B4	3225	117
Software Support	Mike Miller	C21	3220	218
Project Team Leader	Bob Cernik	B11	3238	254
Technical Support (1)	Alfie Neild	S11	3159	282
Technical Support (2)	Trevor Rathbone	S8	3302	124
Electrical Support	Andy Gallager	D14	3548	160
Former Station Scientist	David Laundy	N10	3666	/
Beamline Co-ordinator	Dave Bouch	S2	3147	290
Control Room (assistance during non-office hour)			3560	/

Table 2. Other Useful Telephone Numbers

Function	Phone
Station 2.3 (in control area and hutch)	3635

Hostel	3322
Computer User Support (general inquiry)	3351
User Liaison Office	3223
General Administration	3224
Security Lodge (booking taxis in non-office hours)	3277
Emergency	**3333**

A copy of the laboratory phone directory is available in the station.

2. Diffraction Geometries

2.1 Instrument Review

In the SRS x-rays are produced by the acceleration of the 2 GeV electron beam in the storage ring. The beam current is about 250 mA at the beginning of the fill and it decays down to about 120 mA at the end of the fill. The beam status is displayed on TV monitors. Station 2.3 is situated about 15 m tangentially from a pair of 1.2 T dipole magnet where x-ray wavelengths of 0.5-3 Å are available. The white beam from the synchrotron is intrinsically well collimated (~1 mrad vertical divergence) and then the beam is filtered by the station silicon monochromator. The monochromatic x-ray is finally incident on to the diffraction instrument as shown schematically in Fig. 1. The incident beam and diffracted signals are detected by the two scintillation counters (section 4.3).

The diffractometer is a two circle machine with a sample ω or θ axis and a detector 2θ axis. Fig. 2 is a photograph of the diffractometer inside the hutch. The motorised circles and other motors are controlled by the electronics and computer. The control centre is situated in the work area above the hutch as shown in Fig. 3. The diffractometer was initially designed for high resolution powder diffraction (HRPD) studies [1,2]. The beam optics (Fig. 1) are based on the design described in reference [3]. The precision design of the instrument is also useful for low angle and other high resolution applications. Details of the instrument are described in later sections. The technical summary of station is given in Table 3. First, it is necessary to briefly describe the different modes of operation.

2.2 Powder Diffractometry

HRPD measurements can be performed on either the Hart-Parrish (flat-plate) [4] or capillary diffraction geometry. A sample spinner described in section 3.4 is used in each geometry. Parallel foils mounted on the detector arm improves the resolution without requiring the reduction of the beam size incident on the sample. In the flat-plate set-up, the positions of reflection peak are insensitive to small changes in the sample height or surface or to sample transparency. In the capillary geometry, a spinning goniometer is used instead. If the capillary sample is small, two pairs of Huber single slits should be used instead of the parallel foils. For a highly absorbing material, it is advisable to use flat plate or very thin capillary sample. If the sample suffers preferred orientation, it is advisable to use the capillary geometry instead. The two sample holders are described in details in section 3.4.1.

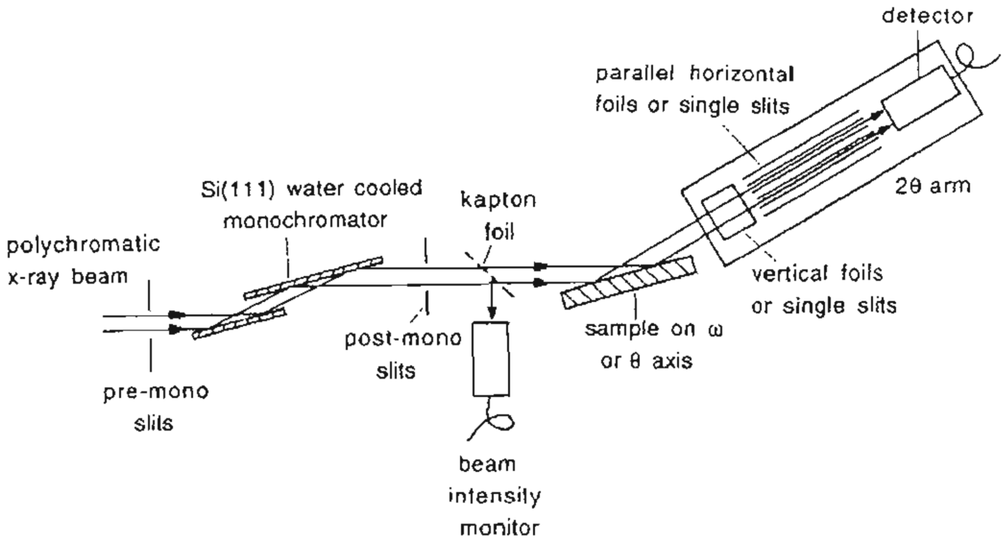

Fig. 1. Schematic of station 2.3

2.3 Surface Reflectometry

Taking advantage of the well collimated beam and high precision motors, the instrument can also be used as a surface probe. In recent time, a large number of reflectivity studies had been performed successfully on this instrument. In this operation mode, the detector arm is moved 5 cm further back to accommodate a large motorised goniometer (see section 3.4.2). The goniometer is placed on the ω-circle and the parallel foils are replaced by single slits arrangement. To improve the signal dynamic range, an enhanced detector (see section 4.4) is used on the 2θ arm instead of the standard scintillation counter.

2.4 Other Applications

Detailed investigations of lattice dynamics using single crystals have been performed on the instrument. Exploiting the parallel beam x-ray optics and high photon flux, diffraction measurements of stress from bulk and thin film polycrystalline materials have been successfully carried out. There are many potential applications using this instrument including the use of high and low temperature devices. Currently, the cryogenic chamber (20-293K) and the induction furnace (293-1800K) are in development.

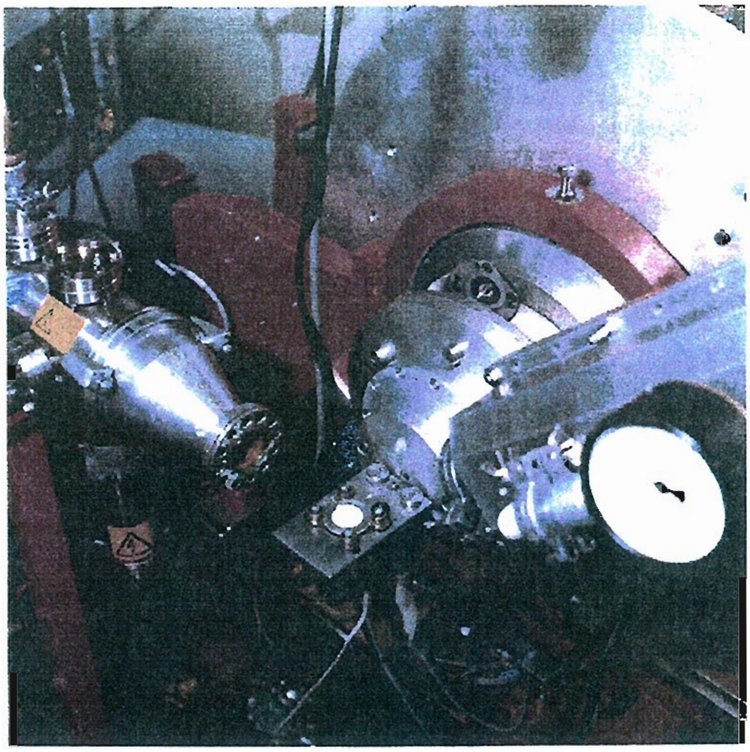

Fig. 2. The diffractometer inside the hutch

Fig. 3. Control centre on the gantry

Table 3. Function and technical descriptions of station 2.3

Functional Description

Station 2.3 is a monochromatic station situated on a dipole magnet with a two circle diffractometer, primarily intended for high resolution angular dispersive powder diffraction measurements.

Current projects include:

(i) High resolution powder diffraction for Rietveld analysis of the data leading to accurate structure determination.

(ii) Structural studies of phase transition at low temperatures.

(iii) Using anomalous dispersion to solve difficult structural problems.

(iv) Reflectivity and diffuse scattering measurements

Recent Developments

While the bulk of the work continues to be powder diffraction, there has been an increasing demand for the use of the instrument for reflectivity, low and high temperature studies.

The instrument software is a system based on a command line interpreter, "**PINCER**". The software is constantly being improved to allow more versatility in the control of the diffractometer and data acquisition.

Technical Description

θ–circle : 0.2 mdeg. resolution

2θ-circle : 0.1 mdeg. resolution

Monochromator : Si(111) water cooled channel cut, 0.1 mdeg. resolution ($\Delta\lambda/\lambda \sim 1.7 \times 10^{-4}$).

Diffraction geometry : Hart-Parrish (flat plate) or capillary

Reflectometry : motorised goniometer, pairs of single slits.

Benchmark : Si powder (NBS 640b) at flat plate geometry. Using λ=1.4 Å and 2x10 mm^2 beam of 230 mA, the (1 1 1) reflection peak count rate = 30×10^3 counts per second with 2θ resolution of 0.05 deg.

Typical experiment turn round time

2 - 4 hours for instrument reconfiguration, alignment and calibration.

Target station efficiency

90%

5% estimated down time - station hardware and software failure.

5% estimated down time - beam line, mainly vacuum and shutter problems.

Target for station improvements

1. Software improvements.
2. In-situ reflectivity sample stages
3. Induction furnace and cryogenic chamber.

3. Hardware

3.1 Introduction

In this and the subsequent chapters, the hardware is described in detail. The text structure is arranged so that the front section (monochromator and incident beam slits) of the instrument monochromatic are presented first. Then the middle sections (the diffractometer and sample holders) are described. Subsequently, the resolution slits, detectors and control hardware are given respectively. Finally, the descriptions of the instrument computer and electronics are presented.

3.2 Monochromator and Incident Beam

A polychromatic beam of just more than 2x15 mm^2 is incident on to the channel-cut Si (111) monochromator which is a monolithic double-bounce single crystal. The wavelength resolution is defined by the Darwin width of the crystal which is $\Delta\lambda/\lambda=1.7\times10^{-4}$. To achieve thermal stability, the temperature of the monochromator is constantly maintained at 30.0•0.1°C with a closed-circuit water bath. The entire device is housed in the monochromator chamber which is evacuated by a rotary vacuum pump. Fig. 4 is a photograph showing the chamber and the beam pipe. It should be emphasized that the user must not let x-rays into the chamber if the vacuum is poor. The tuning and calibration of wavelength are described in sections 5.3 and 5.4 respectively

The emergent monochromatic beam is defined by a centre opening slits before the sample position. The slits is housed inside hutch beam pipe linked to the monochromator chamber, which has an operational maximum and minimum apertures of 2x10 mm^2 and 50x50 μm^2, respectively. The vertical position of the slits can be precisely controlled since they are driven by a stepper motor. To manually set the aperture, the rotary pump is valved off and vented with N_2 gas to atmospheric pressure. The whole slit unit can then be lifted out for adjustments.

The defined beam passes through a 25 μm kapton foil mounted at 45°. The downward scattered radiation is monitored by the scintillation counter (monitor) positioned between the sample and the slits as shown in Fig. 1. The beam finally emerges from the pipe and is incident on to the sample. The diffracted x-ray is recorded by the detector on the 2θ arm. These detectors are described in section 4.3. The minimum motor step size and other hardware information of the monochromator and the slits are given in Table 4. Using an x-ray beam of 2x10 mm^2 produced by an electron current of 220 mA, the photon flux scattered by the kapton foil can be measured by the monitor as a function of wavelength. The results are shown in Fig. 5 and the maximum flux is at wavelengths between 1.1 Å and 1.5 Å.

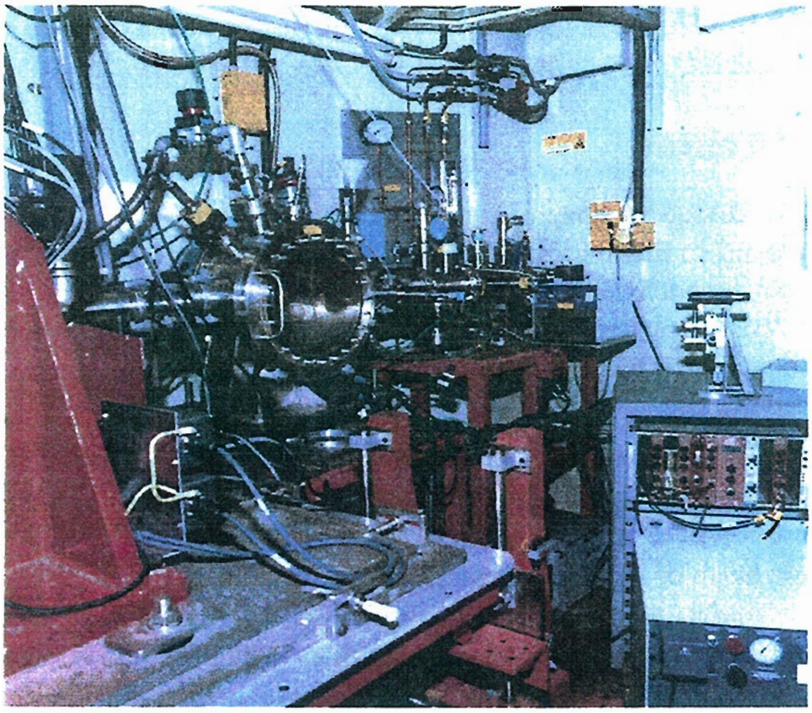

Fig. 4. Monochromator chamber and beam pipe

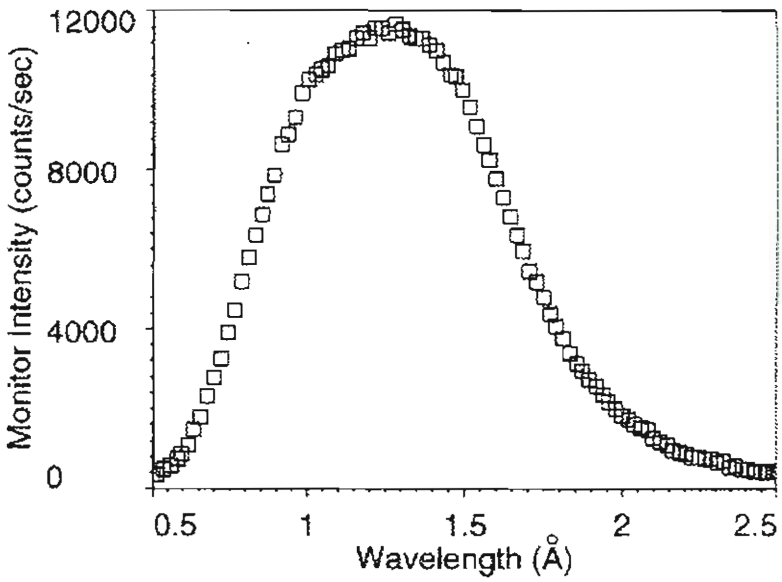

Fig. 5. Photon flux as a function of wavelength

3.3 Diffractometer

The diffractometer consists of two concentric Franke table (type MDM 401 and MDM 251) mounted with the rotational axis in the plane of the synchrotron orbit (horizontal). The two axes are driven, independently or coupled, by DC motors. These motors are encoded and interfaced to the SM9487 Mclennan Servo Drive (see the system manual for details). The movement of each axis is measured by a high precision Heidenhain encoders and fed back to the rack to provide feed back to the drive system. The θ and 2θ are controlled independently with resolutions of 0.1 and 0.2 mdeg. respectively. Backlash is largely eliminated in the servo systems and in the software.

The encoders are not able to provide the absolute positions of each axis but do provide very accurate incremental encoding. Once the axis has been calibrated (section 5.4), subsequent positions of either axis will be reliable. In the event of power failure or computer crash, the encoded positions of the axes will be maintained by the emergency power supply. However, sometime the drive system may lose track of the positions, and it is then necessary to recalibrate the two axes. Usually when the position have been lost, both circles will read zero. The upper and lower limits and other useful information of these motors are given in Table 4.

The diffracometer table height is motorised and it can be precisely controlled. The incident beam profile can be scanned using the slits motor. For precise sample alignment, in particularly in reflectivity work, it is necessary to use a Huber goniometer (section 3.4.2). The arcs and translation (rot1, rot2 and trans) of this goniometer are motorised. The parameters of these motors are given in Table 4. Motor commands and functions are described in section 5.5.

Table 4. Hardware information of the instrument motors

motor	step/deg.	smallest step size	lower limit	upper limit
2θ	10 000	0.1 mdeg.	-20 deg.	130 deg.
$\omega(\theta)$	5 000	0.2 mdeg.	0 deg.	180 deg.
mono	8 000	0.1 mdeg.	4 deg. (λ=0.5 Å)	45 deg. (λ=4.4 Å)
table	-16667/mm	0.1 μm	-5 cm	5 cm
slits	80/mm	0.01 mm	-4 mm	4 mm
rot1	-1204/deg	10 mdeg.	-15 deg.	15 deg.
rot2	-4000/deg	10 mdeg.	-15 deg.	15 deg.
trans	-1204/mm	10μm	-10 mm	10 mm

3.4 Sample Holders

3.4.1 Flat Plate and Capillary Spinners

For flat-plate powder experiment, the sample is loaded to a disk holder with three standard sizes; 15, 20 and 25 mm in diameter and 0.5-1.0 mm in depth. A limited number of holder is provided to each user group. However, the holder design for user production is available if a large number is required. The disk can be slipped onto the STOE spinner mounted on the ω circle. The spinning rate can be controlled by adjusting the potentiometer of the power supply unit mounted on the wall.

In the capillary mode, the flat-plat holder is replaced by the STOE goniometer spinner. With the aid of the telescope, the sample can be align with the spin axis at the centre of rotation. Again, the rate of spinning is controlled as that described for the flat-plate holder. The sample length should be about 25 mm and the maximum and minimum capillary diameters are 2.0 and 0.2 mm, respectively. The two sample holders are shown in Figs. 2 and 6 respectively.

3.4.2 Large Huber Goniometer

For the reflectivity set-up, the sample is placed on the glass or perspex holder of the large Huber goniometer. The holder surface is about 25x25 mm^2 for either the glass or perspex plate. The goniometer, which has two translations and two arcs, is mounted horizontally on the ω circle. The vertical and both rotational movements are motorised, and they can be driven remotely (see section 5.5). This facilitates the precision sample centring which is necessary when a very small beam is used for this kind of work. The motor parameters are listed on Table 4. Fig. 7 is a photograph of the device.

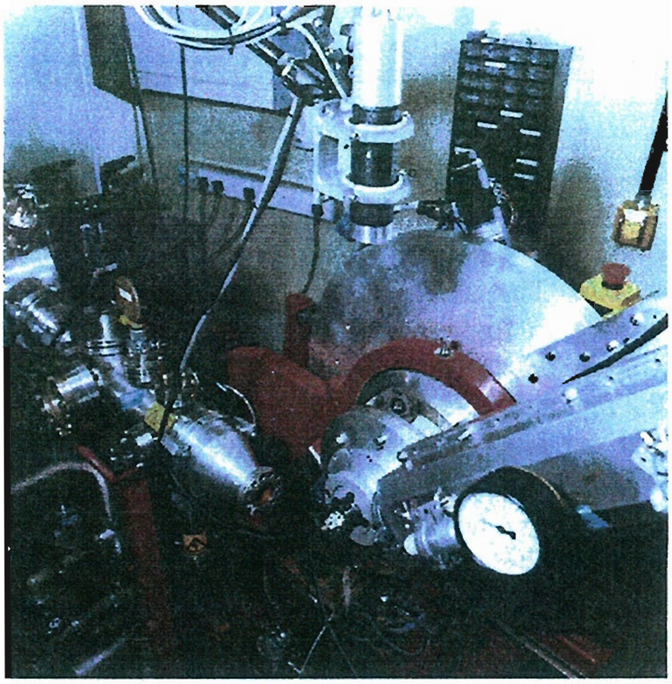

Fig. 6. Capillary sample holder with fluorescence detector at 90 degrees

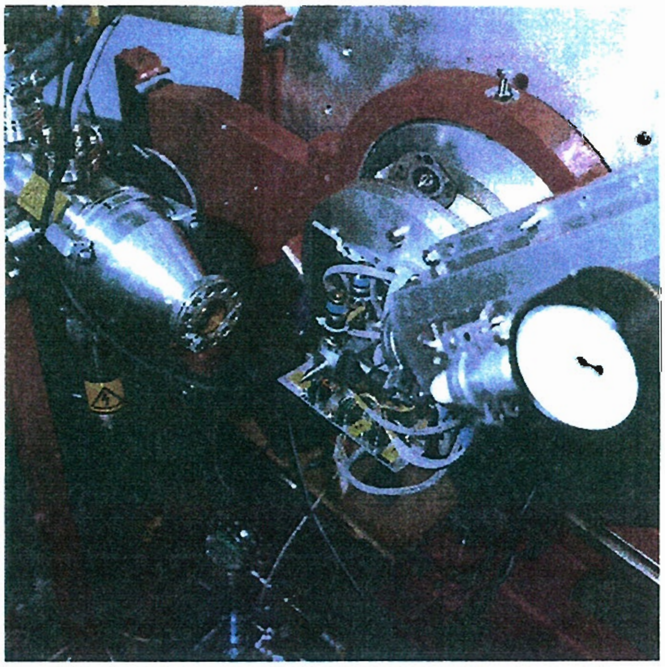

Fig. 7. Motorised sample stage for reflectivity

4. Resolution Slits and Detectors

4.1 Parallel Foils

There are two sets parallel slits which are essentially constructed from multiple foils of molybdenum-stainless steel. The short slits of 5 cm length is used for limit the horizontal axial divergence to improve the line shape at low angles and have an acceptance angle of 1.2 degree. The long collimating foils of 0.05 mm thick, 355 mm in length and spaced 0.2 mm apart, have a nominal angular acceptance of 0.06°. These define the resolution of the instrument and they have an overall aperture of about 20x20 mm^2. The angular resolution as a function of 2θ angle using a flat plate silicon powder [2] is shown in Fig. 8. The parallel foils are housed inside the cover on the detector arm. To reduce air attenuation, the cover is evacuated by a rotary vacuum pump. For most flat-plate powder measurements, the incident and diffraction intensity are recorded by the Harshaw scintillation counters and sometime the enhanced dynamic range (EDR) detector may be used. These detectors are described in sections 4.3 and 4.4. For small capillary sample (less than 1.0 mm in diameter), single slits arrangement should be used instead of the parallel foils.

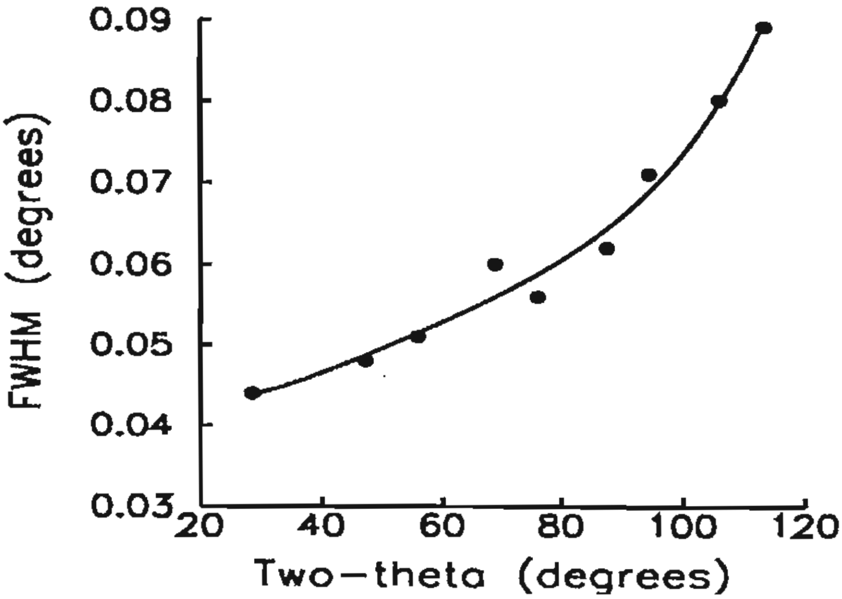

Fig. 8. FWHM as a funtion of two-theta [2]

4.2 Huber Single Slits

In reflectivity measurements, the resolution can be much improved by using the single slits arrangement on the detector arm. Two pairs of slits are used; the scatter and resolution slits which both have an adjustable centre-opening aperture. Typically, the vertical opening of these slits are set to 0.05 to 0.1 mm wide. They can be mounted on the 2θ arm with the cover removed. The first pair is placed nearer the sample position, while the resolution slits is placed nearer to the detector location. The thin film sample is placed on the large Huber goniometer which is mounted on the ω-circle. Using an enhanced dynamic range (EDR) detector (section 4.4), reflectivity and diffused scattering measurements can be performed.

4.3 Scintillation Counters

On the instrument, two Harshaw scintillation detectors are connected to the NIM electronics in the rack in the work area above the hutch (section 4.5). The monitor is viewing the scattered monochromatic radiation from the kapton foil inside the beam (see Fig. 1). The other detector can be mounted on the detector arm to record diffraction signal. However, this detector often mounted 90 above the sample to record the fluorescence signals for anomalous scattering studies. The saturation rate of both devices is about $4x10^5$ counts per second (cps). It should be emphasised that deadtime correction is more necessary when the count rate is more that $50x10^3$ cps.

On the back of each detector, there are three connectors which are connected to the electronic rack inside the hutch. The first is the HT socket connected to the ORTEC 556 High Voltage (0-3 kV) Power Supply unit. The 9 way D type connector that carries low voltage (±24V) supplies to the preamplifier of the photomultiplier tube. This cable is connected to the PREAMP socket on the back of an ORTEC 474 Timing Filter Amplifier. The BNC signal cable connects to the input socket of the amplifier. The HT voltage and amplifier settings for the detector and monitor are summarised in Table 5.

Table 5. Electronic parameters for the detector and monitor

	HT ORTEC 556	Amplifier ORTEC 474	SCA ORTEC 551	Energy Range (resolution)
Detector	0.9 kV	Gain=10 Integrate=50 ns Diff=50 ns	Lower Level=1.0* Upper Level=5.0* (NOR,0.1-1.1msec)	3.5 - 20 keV (≈40%)
Monitor	0.9 kV	Gain=20 Integrate=20 ns Diff=20 ns	Lower Level=1.5 Upper Level=8.0 (NOR,0.1-1.1msec)	As above
Saturation rate = $500x10^3$ cps				

* Levels were set using an x-ray beam of λ=1.4 Å.

Warning: Apart from the SCA levels, no adjustments of electronic parameters should be made without consulting the instrument scientist.

Caution : The Beryllium windows on the detectors are potentially hazardous. Care should be taken to avoid contact with the windows.

The output signal cable connect to the single channel analyser (SCA) in the electronic rack above the hutch (ORTEC Timing SCA 551). The output count rate of the SCA can be monitored on one of the LOG/LIN Rate Meter (ORTEC 449-2). Initially, the SCA should set to "NORMAL" and the "UPPER LEVEL" turned to maximum with the beam off. The "LOWER LEVEL" should be turned up from zero until the count rate meter drops to a few counts per second. With this setting, the SCA will have a wide window and should therefore respond to signal pulses, but not the electronic noise. The output signal trace is monitored by an oscilloscope. When an X-ray reflection has been found, the levels can be adjusted again to improve the discrimination of the SCA against background radiation and higher order reflections. Using the pulse trace of the signals on the scope, the electronic window can be set appropriately. The electronic settings for the two counters are also summarised in Table 5.

4.4 Enhanced Dynamic Range Detector (EDR)

As mention earlier, the scintillation counter on the 2θ arm can be replaced with the EDR detector which is manufactured by Bede Scientific Instrument Ltd. The detector is suitable for detection of X-rays in the approximate energy range of 4-25 keV. The design basically contains a scintillator, photomultiplier tube and high performance control electronics. The detector gives a very low background level (~0.2 cps) and it is capable of detecting peak count rates of more than 450×10^3 cps. However, the correction of deadtime is necessary for count rates above this level.

The head-amplifier box is made up of three stages: the scintillator, the photomultiplier tube and the amplification circuitry. There are three sockets on the detector headamp box, which are the BNC signal, radial E_{HT} and four-core power connections. These are wired to the back panel of the EDR Interface housed in the electronic rack inside the hutch. The signal from the detector is "cleaned" and amplified within the interface module. It is then discriminated and converted to TTL signal which is transmitted to the computer via the NIM crate on the electronics rack above the hutch.

The signal pulse discrimination is performed on the front panel which is housed inside the electronic rack in the control area (above the hutch). The three voltages displayed on the panel are listed in Table 6. The E_{HT} level is the voltage across the collector and the photocathode of the tube. This can be altered to change the gain of the head-amp but the current setting is optimum and should not be adjusted. The threshold voltage (V_{TH}) represents the signal lower cut-off level to eliminate background, the dark current of the detector and other electrical noise. With a small window voltage ($V_W \approx 2$ volt) above the threshold level, the high energy photons from the monochromator (λ/3 contamination) and of cosmic rays can be discriminated.

Table 6. Front panel voltage settings (EDR)

Function	Display	Voltage (V)*
Bias Voltage	E_{HT}	905
Threshold	V_{TH}	0.20
Window	V_W	1.50

Photon Energy Range = 4 - 20 keV

* Levels were set using an x-ray beam of λ=1.4 Å.

Warning: Do not adjust the E_{HT} setting.

Caution : The Beryllium windows on the detectors are potentially hazardous. Care should be taken to avoid contact with the windows.

Below each indicator LED on the panel is a potentiometer. To change the levels simply choose the require voltage using the 'SELECT' button and turn the potentiometer using a small screw driver. Note that the setting for V_{TH} and V_W in Table 6 were obtained using a photon beam of energy of 8.9 keV. More details about the detector can be found in the manufacturer manual (listed in section 1.2) which is available in the station.

4.5 Electronics and Control Computer

The station control hardware and work area are situated on the gantry on top of station hutch. There is an electronic rack contains the detector electronic (amplifiers and ratemeters), the instrument computer (Viglen PC) and a CAMAC crate which allows the computer to read the detectors and control the motors. The other rack contains the McLennan DC motor drive systems for the omega (or theta) and two theta circles, and the stepper drivers for the motors described in section 3.3. The computer communicates through RS232 ports to the McLennan systems and via the CAMAC multiplexer with the stepping motor drivers. The PC and electronics components are shown in Fig. 9.

The ORTEC 449-2 ratemeters are connected accordingly to indicate the analogue signals of the monitor and detector. These are quite useful to check the count rate to give the user some idea of the strength of the signals. In addition, the 2θ and EDR detectors are each connected to a cathode-ray-oscilloscope so the signal pulse shape (NIM and TTL) can be monitored.

Fig. 9. View from left to right of control computer, electronics rack and motor rack (McLennan DC drives above and steppers below)

5. Pincer Control Software

5.1 Software Configurations

The control system is based on the PINCER data acquisition [5,6] which is now in widespread use on several SRS stations. The software is based on a powerful command interpreter and provided a large suite of functions that can be combined in 'macro' command files. On station 2.3, PINCER runs on the Viglen personal computer which controls the instrument. The commands can be entered directly from the keyboard or they can be collected into macros which run simply by typing the name of the macro at the prompt. Macros have been written to allow the user to perform nearly all functions normally required. With advance notifications to the appropriate staff customised macros can be written. For more details about PINCER, the reader is referred to the relevant documents listed in section 1.2.

The PINCER software is executed from the DOS prompt (at any directory) by typing:

PINCER <enter> (<enter> means press the Enter key and this command and subsequent commands can be entered in lower or upper case)

As the 2.3 users perform a wide range of research activities, the instrument has therefore several configurations and mode of operations in order to conveniently accommodate them. Before any data collection it is strongly recommended that the configuration software options should be properly set up. This is done by running the macros STDCONF and PDCONF from the PINCER prompt>. Type in STDCONF the following menu will appear.

```
0. exit

1. Flat plate with STOE sample spinner (powder diffraction)

2. Cryostat

3. Reflectivity measurements with Huber goniometer and long detector arm

4. Reflectivity measurements with Huber goniometer and short detector arm

5. Single crystal with large goniometer and long detector arm (disabled)

option=0=
```

Enter command accordingly.

Type in PDCONF the following menu will appear.

```
1. Graph plotting during theta/2 theta scan    ON

2. ctrl B action during theta/2 theta scans    MENU
```

```
3. Graph plotting during ordinary scans   ON

4. ctrl B action during ordinary scans   MENU

5. Graph plotting during time scans   ON

6. ctrl B action during ordinary scans   MENU

7. beam down delay (sec) theta/2 theta scan (0=disable)   2700

8. SRS scan file format is   POD

9. cryostat being used   ON

10. LOG scale on graph   DISABLE

11. Auto-rescale during theta/2 theta scans   ON

12. Auto-rescale during ordinary scans   ON

13. Auto-rescale during time scans   ON

14 use read motor position in theta/2 theta scans   YES

15. collision detection NONE

16. Save theta/2 theta data in SRS file   YES

17. Save scan data in SRS file   YES

18. Save read detector data in SRS file   YES

19. Allow turbo mode in theta/2 theta scan   NO

20. beam down delay (secs) ordinary scans (0=disable)   0

21. monitor rate limit for pausing scans (cps)   50

22. write MATLAB strings to output file   YES

23. automatically transfer SRS file   YES

24. read MCA data   NO

select option to change or 0 to exit   opt=8=
```

Enter number and change option accordingly. However, data file intended for Rietveld refinement you must enter `POD` for option 8; angles will be saved in mdeg., and `NO` for option 22; no `MATLAB` strings will be written in data files. If `USUAL` is entered for option 8; angles will be saved in degree.

5.2 PD Menu

The main diffraction macros are collected together into a menuing system. These are run from the PINCER prompt by typing:

PD <enter>

The following menu will then appear on screen.

```
------------------------------------------------------------

                  = = = POWDER DIFFRACTION MENU = = =

OPTIONS ...

 Read        Read detectors

 Drive       drive motor menu

 Th2th       theta-2theta couple scan

 NTh2th      multiple theta-2theta couple scans

 Scan        single or two motor scan

 NScan       multiple scans

 Mono        monochromator functions

 Print       print out the 'Brief Guide to PD Measurements'

 View        view on graphic screen an old SRS scan file

 Help        help

 Quit        exit to cli prompt

------------------------------------------------------------

>>> Enter option="QUIT"
```

The function of each option is summarised in Table 7. The option required can be typed in using the upper case character(s) denoted for the command. For `read` and other scan options, the data sets are stored in SRS file format (section 5.7).

Table 7. PD commands and functions

Option	Function
Read	Plot out the count rate in one of the three detector channels as function of time.
Drive	Allows the user to select one of the motors which may be driven to a new

	position or may have its position redefined to a new value.
Th2th	Performs one or more coupled theta/2 theta scan. The user is prompted for the scan range, step size, counting time and the number of times the scan (scan parameters) is to be performed.
NTh2th	Allows the user to perform a number of theta/2 theta scan but the scanning parameters can be set-up individually for each scan.
Scan	Perform a scan of one or two of the motors. The user is prompted for the scan parameters for each motor.
Nscan	As in Scan but this option allows the scan parameters to be entered individually for each scan.

5.3 Change of Wavelength

To change the wavelength, the user simply types in "Mono" on the PD options and the following menu will appear:

```
-------------------------------------------------------------

                          = = = MONO MENU = = =

Silicon Monochromator

Monochromator angle         ... 10.3561 degrees

Monochromator wavelength    ... 1.1273 Angstroms

Monochromator energy        ... 11.0000 keV


OPTIONS ...

 Degree        drive mono to angle (in degrees)

 Lam           drive mono to wave-length (in angstroms)

 Kev           drive mono to energy (in keV)

 SETANG        redefine mono angle

 SETLAM        redefine mono wave-length

 SETKEV        redefine mono energy

 Quit          exit to cli prompt
-------------------------------------------------------------

>>> Enter option="QUIT"
```

The incident radiation can be selected by typing in either one of the there commands: Lam for wavelength, Kev for energy or D for angle and follow by the value. On screen instructions will appear whatever has been chosen. Also the monochromator angle, wavelength, or energy can be set using SETANG, SETLAM or SETKEV respectively.

Warning : Normally, the monochromator is well calibrated (section 5.4). The re-setting of Lam, Kev and Ang must not be performed unless the user is absolutely certain that the calibration is out.

5.4 Wavelength and Two-Theta Calibrations

Normally the calibrations of λ and 2θ are done by the instrument scientist whenever it is applicable using up to 11 reflections from an NBS640b silicon standard powder. When the positions are lost due to computer crash or power failure or motor crash or any other reasons, they needed to be recalibrated. The procedures described in the Appendix (section 10.1) should only be attempted with the help of the instrument scientist or with someone who has the experience. A proper calibration should yield a very accurate wavelength scale (< 0.0001 A) and a very small 2θ offset (< a few mdeg). The values and the associated uncertainties are recorded in the station log book. See for example the calibration in section 10.1.

5.5 To Move Motors

Reflectivity or single crystal experiments require the alignment of sample at the centre of rotation of the diffractometer. The "Drive" option enables the user to perform such an operation. Type in D at the PD menu to run the motor driving macro and the following menu will display:

```
-------------------------------------------------------------

                  = = = DRIVE MOTOR MENU = = =

DETECTOR ...

channels -> time    1, mon. 5102, spare    0, detec.   12

MOTORS ...

tth          two-theta :  10.0000      ome   omega(theta)  :    5.0000

mono     monochromator :  10.3561    slits          slits  :      0.00

table            table :   0.0000     rot1      gonio rot1 :      0.00

rot2        gonoi rot2 :     0.00    trans     gonio trans :      0.00

chi   large gonio rot  :    0.000      phi large gonio rot :     0.000

 type motor name in full
```

```
OPTIONS ...

Pos             drive to position

Inc             drive by increment

SET             redefine position

Help            help

Quit
------------------------------------------------------------

>>> Enter option="QUIT"=pos <enter>

motor="tth"=ome <enter>

position=5=10.052
```

The motors and definitions are listed in Table 8. The line immediately below the DETECTORS shows the counts in one second on the monitor and the detectors. Again, these readings are useful information readily available. By typing the pos command as the above example have shown, the user has moved the omega circle from 5 to 10.052 .

When the move has completed the computer will update the motor position in the drive menu, and then invite the user for the next operation. Movements can be performed by increment (inc command) as well as absolute (pos command). If the positions of these motors needed to redefined type in command set and follow on screen instructions. Repetitive increment positioning can be performed using the Jog function. Type in the command at the drive menu and follow the instructions on the screen. Note that this option is not displayed on the menu. Also note that motors rot1, rot2, trans, phi and chi do appear on the menu if powder diffraction mode has been be set using "stdconf" described in section 5.1. When the user finished with this menu type in Q to get back at PD menu.

The user can abort a driving motor by simply pressing <Ctrl>B, i.e. pressing the Ctrl and B keys simultaneously. For some reason if this operation failed, <Ctrl>A should be used. This will jump out of "PINCER" entirely and abort the drive command immediately. However, the software will record all motor positions before aborting. By restarting Pincer and PD menu (see sections 5.1 and 5.2), the correct motor positions will be available.

Caution : Do not reset a motor position unless it is absolutely required.

Emergency : If all failed press the "EMERGENCY" button to cut off the power of the Mclennan drive unit. This operation may lose the motor positions.

Table 8. Drive motor commands and functions

Motor	Definition and Function
tth	2θ axis - allows the user to drive the two-theta motor
ome	$\omega(\theta)$ axis - allows the user to drive the omega motor
mono	θ_m axis - to move monochromator (in angle only)
slits	Incident beam slits vertical translation
table	Diffractometer vertical translation
rot1	Goniometer arc - axis moves about the vertical plane
rot2	Goniometer arc - axis moves about the horizontal plane
trans	Goniometer transitional stage- vertical movement
chi	Not available
phi	Not available

All angular motors turn in degree and linear translations move in millimetre.

5.6 Data Acquisition

With an appropriate beam size and wavelength, data collection can commence when instrument is calibrated and the sample is aligned. By simply typing in Th2th, Nth2th, Scan or NScan option on the PD menu (section 5.2), the computer will invite the user for the scan parameters; scan regions, scan range, step size, counting time and number of scans. As an example, the user wishes to performed $\theta/2\theta$ scan from 10 to 120 with step size of 0.01 and counting time of 2 second per point. The command on screen will display as follow:

```
>>> Enter option="Quit"= th <enter>

Theta - 2 Theta scan

two theta start [10] : <enter>                          (default value=10 deg.)

two theta end [11] : 120 <enter>                        (for 120 deg.)

counting time seconds [1] : 2 <enter>                   (for 2 seconds)

Number of scans with these parameters [1] : <enter>     (default value=1 second)

Number of data points . . . 11001
```

```
Predicted scan duration . . . 4.3 hours

Plot graph with LOG scale? (y/n) : [N] : <enter>        (default=No)

CONTINUE ? (y/n): [Y] : <enter>                         (default to start scan)*

>>> Enter title: <enter>                                (title may be typed in)

>>> Enter condition 1 : <enter>

>>> Enter condition 2 : <enter>

>>> Enter condition 3 : <enter>

File name r12345.dat

Moving tth to start position  10.0000

Moving ome to start position   5.0000
```

Warning : When the computer displays the following message:

```
Smain warning : Monitor counts are low.
```

This means that there is no x-rays in the hutch beam pipe because, for example, the shutter is still closed. If there is still no x-rays after the shutter is opened this means problems further upstream. In that case, the user should contact the station scientist or personnel in the Main Control Room for help.

The scan commences when the motors have reached their starting positions, and a plot of two-theta versus intensity will appear on the screen. If mistakes were made during the setting up of the scan, type in N (No) at line indicated by the * above and the menu will invite the user to re-enter the scan parameters. Examples of other scan options are given in the Appendix (section 10.2). During a scan the screen will change from a text mode to a graphics and accumulating data will be displayed in a plot. At any time during a scan, it can be interrupted by pressing <Ctrl>B (holding down Ctrl and pressing B keys) and wait for the last scan point to be completed. This will temporarily suspend the scan and bring up a new menu as follow:

```
---------------------------------------------------------

              = = SCAN IN PROGRESS MENU = = =

OPTIONS ...

Rescale         auto rescale graph

Scale           input new scale for graph

Cli             temporary CLI prompt

Quit            quit from this menu and continue scan

Plot            plot graph
```

```
Abort           abort the scan (the data will be save to file)
-------------------------------------------------------------

>>> Enter option="RESCALE"=
```

The menu above allows the user to change the plot scale, dump plot to the printer or abort the scan. The scan remains suspended until the user selects or <enter> from the menu. The user may type in option accordingly. When the scan is finished, another menu will appear as follow:

```
-------------------------------------------------------------

                = = SCAN FINISHED MENU = = =

OPTIONS ...

Scale           input new scale for graph

Cli             temporary CLI prompt - terminate with 'return'

Quit            quit menu, and finish

Plot            plot graph on printer

Info            Display scan info and statistics

Display         redisplay graph
-------------------------------------------------------------

>>> Enter option="QUIT"=
```

For a hardcopy, the user should type in `plot` and the graph on the screen is dumped on to the Epson printer. When option "Quit" is typed in, the scan is completely finished and the data file will be transferred automatically (see section 5.7).

5.7 SRS Data Format and File Transfer

Data files are named rxxxx.dat where xxxxx refers to a unique file number given to each SRS file on the station. Each successive SRS file created has a number one larger than the pervious file allowing the data files to be identified. All data files have the standard SRS header including the run number, date, time, motor positions and other relevant information. The angle(s), time, monitor and detector readings are store in columns. The end of the data file is marked by "END OF DATA". The data format of theta/2-theta and ordinary scans are presented in the Appendices (section 10.3).

After each scan, the data file is transferred automatically over the network to "/srsdata/pd23" directory of xrdsv1 main computer. Sometime the computer network is down and error messages will appear on screen. These messages are informative and no action should be taken because the automatic transfer process will resume once the network is restored. After the files have been transferred, the data is still present on the instrument PC but the files are renamed with an extension .con, for example, r12345.dat would become r12345.con after the transfer. However, the .con files are deleted every now and then in order to maintain sufficient disk space to run the instrument.

Eventually, the transferred files in "/srsdata/pd23" will be automatically archived to optical disks. To retrieve these files, log on xrdsv1 and type in **nsrsget pd23 r12345.dat**. This will retrieve that particular data file. For a range of files, type in **nsrsget pd23 r12345.dat-r12350.dat**. For more information type in **man nsrsget**.

6. Data Analysis Software

6.1 Introduction

The analysis of the data must be carried out on other terminals and not on the instrument computer. There are two computers in the control area for users to do their data analysis. Graphic and peak refinement programs are available in these computers. More programs are available in the CCP14 suite. Documentation on the software and the computers are listed in section 1.2. The X-terminal can access to the central computers for the PD programs described in this chapter. The other is the second Viglen PC which has MATLAB software for data analysis. The user may load his/her own software into this PC to perform data analysis. Note that the work must be deleted after the completion of the experiment.

6.2 PD Data

When diffraction patterns are taken using the parallel foils geometry, the reflection peak shaped can be well described by a pseudo-Voigt function. Single or multiple patterns can be analysed using Rietveld refinement software which is available on the "xrdsv1" system. Most of these program have been collected together into the Powder Diffraction Program Library (PDPL). The command PDHELP accesses a help system for these programs which gives notes and examples. The manual of the software listed in section 1.2 is available in the station. A personal copy can be obtained from the computer centre.

6.2.1 Data Display (PLOT86)

The PLOT86 graphic program can be used to display PD data on a terminal or to output data to various printers and plotters. Up to five data sets can be plotted unless difference data sets, e.g. MPROF or PKFIT, are to be displayed then only one set of observed, calculated and difference data can be plotted. A cursor can be used to measured peak and background intensities. This program has no interactive help system.

6.2.2 Data Normalisation (PODSUM)

The X-ray flux is not constant as to the current of the electron beam in the storage ring decays. To correct for this beam decay the program PODSUM normalised the raw data against the monitor count. But the software can only performed on the data file which is POD formated (section 5.1) The program which has an interactive help system is also useful to join data sets together and normalised them.

6.2.3 Lattice Parameter Refinement (REFCEL)

The REFCEL program is used for lattice parameter determination. Miller indices and two-theta angles are needed as input together with 2θ zero point, λ, trail lattice parameters and symmetry of the unit cell. These values can be entered from a keyboard or from a file. The program performs refinement of minimisation to determine the cell parameters and the zero point. The routine has interactive help systems.

6.2.4 Reflection Position Determination (DRAGON)

This program produces a list of the positions of Bragg reflections and the Miller indices. The refined cell parameters, zero point, λ and space group symmetry are needed as input quantities. This program can also produce a file called "hkl.hkl" which is needed for the MPREP preprofile preparation program.

6.2.5 Symmetry Operations (SPG)

The SPD routine gives a list of symmetry operations for a space group, the space group number or symbol is required as input. The symmetry operations can be output to a file which is input to MPROF profile (Rietveld) refinement program. All symmetry operations are listed apart from (x,y,z) and centrosymmetric operations.

6.2.6 Pattern Prediction (LAZY, LAZGEN and DATGEN)

The program LAZY is used to calculate the structure factors for a trial crystal structure. The cell parameters, λ, atomic coordinates, temperature factors and site occupation factors are requires as input. This data can be either be input from the keyboard or from a file generated using LAZGEN program. A file of structure factors named "fout.dat" is produced. This file us used as input for the program DATGEN which predicts PD patterns from the structure factors. Peak shape and halfwidth parameters as also required as input.

6.2.7 Rietveld Profile Refinement (MPREP and MPROF)

The MPREP (preprofile preparation) program must be run before the MPROF (profile refinement) program. The first program is used to subtract a background from the PD data and to inform the second program the position of the reflections. MPROF is a least-squares refinement program which calculates a PD pattern from trial structural parameters and compares this pattern to the observed data. The refinement process varies the trial parameters to get a good match as possible to the observed data. Note that the trial structure must be a reasonable match to the actual structure. If this is not so the program may not refine to a sensible result.

6.2.8 Ab-initio Structure Factor Determination (MPROFIL)

The program is used to determine structure factors for ab-initio structure determination. This program is similar to MPROF but no atomic coordinate positions are refined. A profile is fitted to the PD data, the structure factors are determined form the intensity of each reflection. The program MPREP is needed for preprofile preparation for MPROFIL.

6.3 Other Software

The program CRYSTAL is also available to perform structural refinement. Individual peak shape analysis can be carried out using PKFIT which is also available on "xrdsv1". A user guide listed in section 1.2 is available in the station. The "xrdsv1" and "xserv1" central computers also offers graphic routines such as "PLOTEK" and "PLOT86". Manuals can be obtained from the computer help desk. At a development stage is "MOTPLOT" which will be available in the near future.

7. Interlocking the Hutch and Opening the X-ray Shutter

7.1 Introduction

As safety is paramount all new and experienced users must watch the laboratory safety video which is available on machines in the coffee lounge, the B-block foyer and the hostel. In order to let x-ray beam into the hutch, it is necessary to first interlock the hutch. In this video, the interlocking procedures are shown. The purpose of doing this is to ensure that there can be nobody inside exposed to the x-ray radiation. All users must follow the procedures described in section 7.2 and aware of the safety features described in section 7.3. New users must ask the station scientist to demonstrate the procedures.

7.2 Interlocking Procedures

1. Turn the switch just outside the hutch door to the START SEARCH position.

2. Press the START SEARCH green button - a warning bleep will sound inside the hutch to alert anyone still inside and they must now exit the hutch.

3. Walk inside the hutch and check there is no other personnel inside the hutch. Press the green button marked SEARCH POINT which is located at the far end.

4. Exit from the hutch, close the door and press SEARCH COMPLETED button. A solenoid will operate to lock the door.

5. Wait for the hutch to interlock (timer). During this time, blue warning lights will come on inside the hutch. When the timer finishes (takes about 30 seconds), the blue lights will go out and the illuminated panel above the hutch door marked HUTCH INTERLOCKED will come on.

6. On the TANDY computer (outside the hutch or upstairs), press F2 button to open the shutter to let x-rays into the hutch*.

7. To re-enter the hutch press F1 button on the Tandy computer* and turn the switch on the hutch door to the OFF position. Wait for the timer to unlock. When it is completed you may enter the hutch.

For more information about the TANDY terminal, users should read the menu, "Instruction to Experimenters for Operating the Beamline Control Terminals", which is available on the station. Any problems encountered with using the terminal should be reported as soon as possible to the station scientist or to the Main Control Room.

When the computer has been idle for a while, it is necessary to press the <space bar> key to re-activated the system and then **F2 to open shutter or **F1** to close shutter.*

Attention: Quite often the shutter failed to open after pressing **F2** because the set-up or parameters have been lost. To re-activate the system, key in

X2STOP03

and then press **F2** to open shutter.

In most cases, this operation should resolve the problem. If however the problem persists, the user should contact the station scientist during office hours or the Main Control Room in non-office hours.

7.3 Safety Features

There are two large yellow round buttons inside the hutch for emergency shutdown. One on the far wall next to the green search button and the other is on the right hand side wall adjacent to the hutch door. When either one of these buttons is pressed the entire beam line will be shut. If this has happened you must contact the instrument scientist of personnel in the control room. The lever on the inside of the hutch door is design for emergency exit if necessary. If the switch on the hutch door is turned OFF with the x-ray beam on, the Tandy computer will close the shutter before allowing the door to be opened.

8. Trouble Shooting

Problem	Suggestion
No signal in the detector	Check monitoring detector
No signal in the monitor	Beam shutter not opened
Shutter Opened but no beam	Port 2 not open (absented on TV monitor)
Port 2 not opened	Beamline vacuum problem or SRS beam lost.
Motor not moving	Power mains is turned off
	Tracking problem (red light on McLennan Drive unit) - motor has crashed or overloading
	Or hardware limits are reached.
Control computer stuck	Reboot PC by pressing <ctrl/Alt/Delete> keys
Stop motor driving	<Ctrl/B> to stop. If this failed try <Ctrl/A>
To abort a scan	<Ctrl/B> and then press <A> key to abort.

9. Glossary

CAMAC	An ageing but useful 24-bit instrumentation bus which does not constitute a computing bus. IEEE-583.
DC Servo motor	A motor control system which instead of producing fixed current steps as a stepper motor does, provides varying current to match the load in a feedback system. It is normally found with high resolution encoders for the most demanding detector positioning. It is used routinely at Daresbury on twotheta axes of diffractometers for example.
HRPD	High resolution powder diffraction.
MACROS	Pincer command (batch) files containing lists of Pincer commands and maths functions with full flow control, local variables and with extension *.mac*.
MCA	Multichannel Analyser. Consists of an ADC to convert analogue input voltages from a detector to a digital value and a histogramming memory unit which is incremented at the address proportional to the ADC output value.
OMEGA	Sample rotation circle also known as theta when coupled with two-theta.
PATH	In DOS refers to both the full location name of a file and to the environment variable which defines the search order when a command is typed.
SCA	Produces integrated TTL counts from an MCA region of interest (may be the whole spectrum).
TTL	Digital signal widely used in computers and CAMAC and frequently implemented using LEMO cables.
THETA	Sample rotation circle also known as omega if not coupled with two-theta.
TWOTHETA	Detector arm which scans diffraction peaks.
XRD	Inorganic X-Ray Diffraction Project at the SRS, headed by Dr. Bob Cernik. Includes Powder Diffraction, Topography and Single Crystal Diffraction Stations.

10. Appendices

10.1 Calibrations of λ and 2θ

1. Put on the Si standard powder (already loaded on a flate-plate holder)

2. Using the jog box inside the hutch drive the omega and two-theta axes to their zero position with the aid of a spirit level.

3. On the instrument computer set omega and two-theta axes to zero.

4. Set McLennan LED units to zero for both theta and two-theta displays (optional).

5. With the beam on, type in SICALIB at Pincer ">" prompt and the computer will invite you for the following questions.

```
>sicalib

counting time seconds [1] : <Enter>

mono angle error (degree) [1] : <Enter>

two theta error (degree) [1] : <Enter>

Number of reflections to scan [9] : <Enter>

Electron beam current (mA) [ ] : 200 <Enter>

Predicted calibration duration ... 16.1 Minutes


hit key to continue

CONTINUE ? (y/n) : <Enter>

>>> Enter condition 1 <Enter>

>>> Enter condition 2: <Enter>

>>> Enter condition 3: <Enter>

File r12345.dat
```

Warning : When the computer displays the following message:

```
Smain warning : Monitor counts are low.
```

This means that there is no x-rays in the hutch beam pipe because the shutter is still closed. If there is still no x-rays after the shutter is opened this means problems further upstream. You should contact the station scientist or personnel in the control room for help.

With the x-ray beam on, the diffractometer will scan the first of the nine Si reflections and follow by the rest when the motors have moved to the starting positions. When the scans are finished, the following table will be displayed:

```
Refining wavelength and zero-point

iter  lambda (angstroms)  two-theta zero

****************************************

  1       1.399612   0.007329

  2       1.399612   0.007329

****************************************



****************************************

  h   k   l   fitted  discrepancy

  1   1   1   25.7922  -0.0007

  2   2   0   42.7485    0.0003

  3   1   1   50.6009    0.0006

  4   0   0   62.0513   -0.0002

  3   3   1   68.3424    0.0004

  4   2   2   78.2863   -0.0001

  3   3   3   84.0650    0.0005

  4   4   0   93.5912   -0.0006

  5   3   1   99.3381   -0.0004

****************************************

hardcopy

CONTINUE ? (y/n) : [N] :
```

The discrepancies in λ and 2θ should be small as shown in the table. Finally, the user needs to answer Y (yes) to calibrate the wavelength and two axes when invited by the software macro.

10.2 Scan Options

10.2.1 Theta-2Theta (Th2th)

```
Theta - 2 Theta scan

two theta start [10] : <Enter>                               (default value=10 )

two theta end [11] : 120 <Enter>                             (for 120  )

counting time seconds [1] : 1 <Enter>                        (for 2  seconds)

Number of scans with these parameters [1] : <Enter>          (default value=1 second)

Number of data points . . . 11001

Predicted scan duration . . . 4.3 hours

Plot graph with LOG scale? (y/n) : [N] : <Enter>             (default=No)

CONTINUE ? (y/n): [Y] : <Enter>                              (default to start scan)

>>> Enter title: <Enter>                                     (title may be typed in)

>>> Enter condition 1 : <Enter>

>>> Enter condition 2 : <Enter>

>>> Enter condition 3 : <Enter>

File name rxxxxx.dat
```

The scan should starts when the motors have reached the starting positions.

10.2.2 Ordinary Scan (Scan)

```
------------------------------------------------------------

                 = = = MOTOR POSITIONS = = =


MOTORS ...

 tth        two-theta :  10.0000      ome   omega(theta) :     5.0000

mono    monochromator :  10.3561    slits          slits :       0.00
```

table	table :	0.0000	rot1	gonio rot1 :	0.00
rot2	gonoi rot2 :	0.00	trans	gonio trans :	0.00
chi	large gonio rot :	0.000	phi	large gonio rot :	0.000

---

motor="tth"=table <Enter>

start1=0=-1 <Enter>

end1=1=<Enter>

step1=0.1=0.05 <Enter>

Type in motor2 or <return. for single motor scan [] :

Type in counting time per point in seconds [1] :

Type in channel to be plotted

T ... Time

M ... Monitor

S ... Spare Channel

D .. 2-theta Arm Detector

channel="D"= <Enter> *(to plot 2-theta detector)*

CONTINUE ? (y/n): [Y]: <Enter> *(No, if mistakes were made)*

>>> Enter title: <Enter> *(title may be typed in)*

>>> Enter condition 1 : <Enter>

>>> Enter condition 2 : <Enter>

>>> Enter condition 3 : <Enter>

File name r12345.dat

The scan should starts when the motors have reached the starting positions.

10.2.3 Multiple Theta-2 Theta Scans (Nth2th)

****

Multiple Theta - 2 Theta Scans

****

```
scan parameters file (or <Enter> to type in scan parameters [] :

total number of scans [2] : 3 <Enter>


***********

scan no.  1

***********

two theta start tthst [1]=10.000= <Enter>

two theta end tthe[1]=11= 50 <Enter>

two theta step tths[1]=0.01= <Enter>

counting time seconds [1]=1= <Enter>


***********

scan no.  2

***********

two theta start tthst [2]=50.01= 50 <Enter>

two theta end tthe[2]=90= 90 <Enter>

two theta step tths[1]=0.01= <Enter>

counting time seconds[2]=1= 5 <Enter>


***********

scan no.  3

***********

two theta start tthst [2]=90.01= 90 <Enter>

two theta end tthe[2]=130.01= 130 <Enter>

two theta step tths[1]=0.01= <Enter>

counting time seconds[2]=1= 10 <Enter>

Number of scans =  3
```

```
  tth start   tth end       tth step      count time  (secs)

     10.000    50.000          0.010          1.000

     50.000    90.000          0.010          5.000

     90.000   120.000          0.010         10.000

Predicted scan duration ... 19.3 Hours

CONTINUE ? (y/n): [Y] : <Enter>                          (default to start scan)

>>> Enter title: <enter>                                 (title may be typed in)

>>> Enter condition 1 : <Enter>

>>> Enter condition 2 : <Enter>

>>> Enter condition 3 : <Enter>

File name r12345.dat
```

The scans will commence after the motors have reached the starting positions. The above example shows three θ-2θ scans have been executed. The first scan starts at 10 deg and ends at 50 . with step size of 0.01 and counting time of 1 second for each point. The second scan starts at 50 and ends at 90 with step size of 0.01 and 5 second for each point. The final scan starts at 90 and ends at 130 . with step size of 0.01 and 10 second per point. The three scan data sets will be saved in separate SRS files.

10.2.4 Multiple Ordinary Scans

```
----------------------------------------------------------

                  = = = MOTOR POSITIONS = = =


 MOTORS ...

  tth        two-theta  :  10.0000      ome    omega(theta)  :     5.0000

 mono    monochromator  :  10.3561    slits           slits  :       0.00

table            table  :   0.0000     rot1      gonio rot1  :       0.00

 rot2       gonoi rot2  :     0.00    trans     gonio trans  :       0.00

 chi   large gonio rot  :    0.000      phi large gonio rot  :      0.000

----------------------------------------------------------


**************
```

Multiple Scans

****

Motor to move between scans

****

motor (or <Enter> to ignore) [] :

nscan=1=5 <Enter> *(for 5 repeating scans)*

Scan parameters

****

Number of scans = 10

motor1="tth"

start1=10= <Enter>

end1=11=120

setp1=0.1=0.01

Type in motor2 or <return> for single motor scan [] : <Enter>

Type in counting time per point in second [1] : 2 <Enter>

Increment parameters between scans

****

increment counting time by [0] : <Enter>

increment motor 1 start position by [0] : <Enter>

increment motor 1 end position by [0] : <Enter>

execute PINCER command before each scan [] : <Enter>

execute PINCER command after each scan [] : <Enter>

Type in channel to be plotted

T ... Time

```
M ...  Monitor

S ...  Spare Channel

D ..   2-theta Arm Detector

channel="D"= <enter>                         (to plot 2-theta detector)

CONTINUE ? (y/n): [Y]: <enter>

>>> Enter title: <enter>                     (title may be typed in)

>>> Enter condition 1 : <enter>

>>> Enter condition 2 : <enter>

>>> Enter condition 3 : <enter>

File name r12345.dat
```

Here, the 2θ scan starts at 10 and ends at 120 with step size of 0.01 and 2 second per point. In total 5 scans have be executed with the same scan parameters. The first scan will start when the two-theta motor has reached the starting position. The data of these scans will be saved in separate SRS files.

10.3 SRS Data Files

10.3.1 Theta-2 theta file in POD configuration

The following data file is an example of a theta/2-theta scan.

```
&SRS
 SRSRUN=25066,SRSDAT=950719,SRSTIM=124048,
 SRSSTN='PD23',SRSPRJ='POWDERDF',SRSEXP='12345432',
 SRSTLE='                                                                    ',
 SRSCN1='        ',SRSCN2='        ',SRSCN3='        ',
 &END
    tth =    44.5000,
    ome =     0.0000,
   mono =    13.7963,
  slits =       0.00,
  table =     0.0000,
   rot1 =       0.29,
   rot2 =      -0.27,
  trans =       0.00,
    chi =      0.000,
    phi =      0.000,
 Unused =          0,
 Unused =          0,
    TWOTHETA    THETA        TIME       CHAN2       CHAN3       CHAN4
    41600.      20800.       5000.      6497.          0.         66.
    41650.      20825.       5000.      6506.          0.         66.
    41700.      20850.       5000.      6622.          0.         49.
    41750.      20875.       5000.      6479.          0.         64.
    41800.      20900.       5000.      6623.          0.         69.
    41850.      20925.       5000.      6454.          0.         67.
    41900.      20950.       5000.      6503.          0.         67.
    41951.      20975.       5000.      6536.          0.         76.
    42000.      21000.       5000.      6704.          0.         74.
    42050.      21025.       5000.      6564.          0.         60.
    42100.      21050.       5000.      6780.          0.         90.
```

```
    42150.      21075.       5000.      6630.        0.       113.
    42200.      21100.       5000.      6583.        0.       117.
    42250.      21125.       5000.      6622.        0.       115.
    42300.      21150.       5000.      6680.        0.       121.
    42350.      21175.       5000.      6717.        0.       128.
    42400.      21200.       5000.      6797.        0.       111.
    42451.      21225.       5000.      6605.        0.       102.
    42500.      21250.       5000.      6583.        0.        83.
    42550.      21275.       5000.      6566.        0.        76.
    42600.      21300.       5000.      6433.        0.        63.
    42650.      21325.       5000.      6559.        0.        65.
    42700.      21350.       5000.      6690.        0.        59.
    42749.      21375.       5000.      6622.        0.        65.
    42800.      21400.       5000.      6588.        0.        51.
    42850.      21425.       5000.      6675.        0.        60.
    42900.      21450.       5000.      6570.        0.        62.
    42950.      21475.       5000.      6497.        0.        57.
    43000.      21500.       5000.      6690.        0.        54.
END OF DATA
```

The first two columns in the above file are the 2θ and θ values in mdeg. The subsequent column is collection time (5 sec) per point in millisecond . The remaining columns are the counts on the monitor, spare (fluorescence) detector and the 2θ detector. Note that for USUAL configuration (section 5.1), the angles and time will be in degree and second, respectively.

10.3.2 Ordinary Scan in USUAL configuration

The following data file is an example of an omega scan.

```
&SRS
 SRSRUN=25065,SRSDAT=950719,SRSTIM=005609,
 SRSSTN='PD23',SRSPRJ='POWDERDF',SRSEXP='12345432',
 SRSTLE='                                                         ',
 SRSCN1='        ',SRSCN2='        ',SRSCN3='        ',
&END
    tth =     4.1161,
    ome =     2.0982,
   mono =    14.8801,
  slits =       0.00,
  table =     0.0000,
   rot1 =       0.00,
   rot2 =       0.00,
  trans =       0.25,
    chi =      0.000,
    phi =      0.000,
 Unused =          0,
 Unused =          0,
      ome          Time      Chan2      Chan3      Chan4
    2.0400      1.0000      479.      2301.      269.
    2.0410      1.0000      455.      2393.      280.
    2.0422      1.0000      452.      2270.      265.
    2.0428      1.0000      480.      2349.      269.
    2.0442      1.0000      455.      2397.      285.
    2.0452      1.0000      475.      2371.      278.
    2.0462      1.0000      458.      2303.      249.
    2.0470      1.0000      463.      2377.      292.
    2.0482      1.0000      464.      2300.      305.
    2.0492      1.0000      446.      2334.      296.
    2.0500      1.0000      488.      2259.      311.
    2.0512      1.0000      492.      2247.      338.
    2.0522      1.0000      469.      2389.      427.
    2.0530      1.0000      466.      2347.      442.
    2.0540      1.0000      482.      2256.      477.
    2.0552      1.0000      491.      2334.      597.
    2.0560      1.0000      501.      2264.      571.
    2.0570      1.0000      484.      2210.      599.
    2.0582      1.0000      510.      2203.      572.
```

```
    2.0592       1.0000       454.       2321.        581.
    2.0600       1.0000       478.       2243.        481.
END OF DATA
```

The data columns are omega (deg.), time (sec), monitor, spare detector and 2θ detector readings. Note that for POD configuration (section 5.1), the angles and time will be in mdeg. and milliseconds, respectively.

10.4 Photographs

Originals of the photographs in this manual are stored in the Information Centre of the laboratory. Here are the reference numbers.

Fig. No.	Photo. No.	Fig. No.	Photo. No.
2	DL96/220/5	6	DL96/220/7
3	DL96/220/2	7	DL96/220/8
4	DL96/220/17	9	DL96/220/1

10.5 Acknowledgements

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11. References

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