Station 2.3 Manual

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Abstract

The objective of this 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 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 hard copy 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 using both flat-plate geometry and capillary goniometry, and high flux surface reflectometry. The users are from academia and industry, 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 is maintained as a powerful microscopic tool for modern 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 such 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 experienced workers as well as new users.

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 2.3 enclosure

1.4 Getting Help

The following staff can be called upon for technical assistance:

Table 1. Technical and other supporting staff

Position	Name	Room	Phone	Pager
Station Scientist	Chiu Tang	B11	3744	117
Software Support	Mike Miller	C21	3220	218
Project Team Leader	Graham Bushnell-Wye	B53	3623	326
Technical Support (1)	Alfie Neild	S11	3159	282
Technical Support (2)	Trevor Rathbone	S8	3302	124
Electrical Support (1)	Andy Gallagher	D14	3548	160
Electrical Support (2)	Andy Wavish	D14A	3392	161
Former Station Scientist	David Laundy	B11	3238	228
Beamline Co-ordinator	Dave Bouch	S2	3147	290
Deputy Co-ordinator	Chiu Tang	B11	3744	117
Main Control Room (assistance during non-office hour)			3560	/

Table 2. Other useful telephone numbers

Function	**Phone**/*pager*
Station 2.3 (the control area and in the hutch)	3635
Station Enclosure (for Data Analysis)	3575
Safety Officer	3264

Table 2. Continued.

Radiation Protection Officer	3266
Hostel	3322
Nursing Sister (Occupational Health)	3234
Computer User Support (general inquiry)	3351
PC Support	3351
User Liaison Office	3223
General Administration	3224
Switch Board	0
Security Lodge (booking taxis in non-office hours)	3277
Liquid N_2 supply	*133*
Emergency	3333

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

2. Diffraction Geometries

2.1 Instrument Review

In the SRS, x-rays are produced by the acceleration of a 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 magnets 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 the beam is filtered by the station silicon monochromator. The monochromatic x-ray beam is finally incident on to the diffraction instrument as shown schematically in Fig. 1. The incident beam and diffracted signals are detected by scintillation counters (sections 4.4 and 4.5).
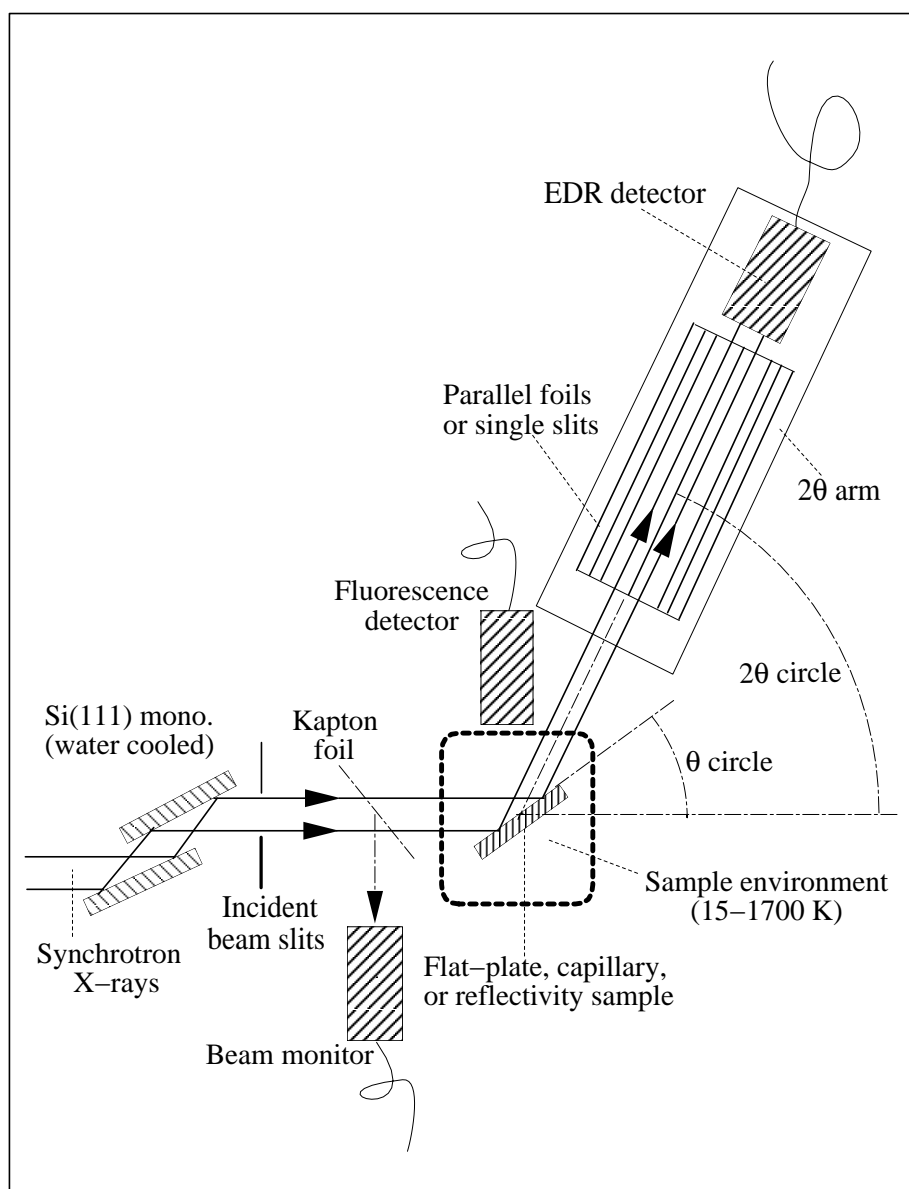
Fig. 1. The diffraction instrument on Station 2.3 in schematic.

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 experimental hutch. The instrument 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 and the different modes of operation are described in later sections. The technical summary of station is given in Table 3. Motor movements and data acquisition are performed by computers which are in the control gantry and in the newly constructed room (the 2.3 enclosure).

Fig. 2. The diffractometer inside the hutch with the cryostat mounted on the ω-circle. The photograph was taken from above the incident beam pipe.

2.2 Control Gantry and the Enclosure

The motorised diffractometer is controlled by electronic interfaces *via* a computer. The electronics and the computer are in the control gantry which is situated above the hutch as shown in Fig. 3. The photograph shows the computer (left), the electronics rack (center), the Mclennan Drive System (right), and the TV monitors (above the PC). The electronics and driver are described in section 4.5. Two of the monitors are linked to the cameras inside the hutch and one displays the SRS beam status.

Fig. 3. The control computer, electronic racks, Mclennan system and TV monitors on the gantry.

A new enclosure has recently been constructed for data analysis (Fig. 4). In the photograph, the centre computer is linked to the control computer, and it can be used to drive the diffractometer and to collect data. The work station (xrdws6) on the right and the PC (cctvig) on the left are for data analysis. Again, the enclosure has TV monitors which are linked to the cameras inside the hutch and to the SRS.

Fig. 4. The computers and camera-monitors in the 2.3 enclosure.

2.3 Powder Diffractometry

HRPD measurements can be performed in 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 improve the resolution without requiring the reduction of the beam size incident on the sample. In the flat-plate set-up, the positions of reflection peaks are insensitive to small changes in the sample height or sample 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 a flat plate or a 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 detail in section 3.4.1.

2.4 Surface Reflectometry

Taking advantage of the well collimated beam and high precision motors, the instrument can also be used as a surface probe [5]. In recent times, a large number of reflectivity studies have been performed successfully on this instrument. In this mode of operation, a large motorised goniometer (see section 3.4.2) is mounted on the ω-circle, and the parallel foils are replaced by a 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. Detailed experimental configurations can be found in Chapter 4 and reference [5].

2.5 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 [6,7]. There are many potential applications of this instrument including the use of high and low temperature devices. Recently, the cryogenic chamber (20-293 K) and the induction furnace (293-1700 K) have been successfully commissioned [8,9]. These apparatus can be used with the flat-plate geometry for non-ambient measurements.

Table 3. Function and technical descriptions of Station 2.3

Functional Description

Station 2.3 is a monochromatic station situated on a 1.2 T dipole magnet. The station has a two circle diffractometer, parallel foils, single slits assembly and motorised goniometer. The instrument is primarily intended for high resolution angular dispersive PD measurements and reflectivity studies.

(i) High resolution powder diffraction for Rietveld analysis of 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

Table 3. Continued.

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 window command interpreter, "**PINCER**". The software is constantly being improved to allow more versatility in the control of the diffractometer and data acquisition.

Technical Description

ω- or θ-circle : 0.2 mdeg. resolution

2θ-circle : 0.1 mdeg. resolution

Monochromator : monolithic 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.

<u>Benchmark</u> : Si powder (NBS 640b) at flat plate geometry. Using λ=1.4 Å and a 2×10 mm^2 beam at 230 mA, the (1 1 1) reflection peak count rate $\geq 35 \times 10^3$ counts per second with 2θ resolution of ~0.06°.

Typical experiment turn round time

2 hours for instrument reconfiguration, alignment and calibration of λ and 2θ. At least ½ day changeover time for the cryogenic apparatus or the induction furnace.

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

Software improvements, furnaces, cryogenic chamber and new reflectivity stage.

3. Instrumentation

3.1 Introduction

In this and the subsequent chapter, 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 descriptions of resolution slits, detectors and control hardware are given. 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 $\Delta\lambda/\lambda=1.7x10^{-4}$. To achieve thermal stability, the temperature of the monochromator is constantly maintained at $30.0\pm0.1°C$ using a closed-circuit water bath. The entire device is housed in the monochromator chamber which is evacuated by a rotary vacuum pump. Fig. 5 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.
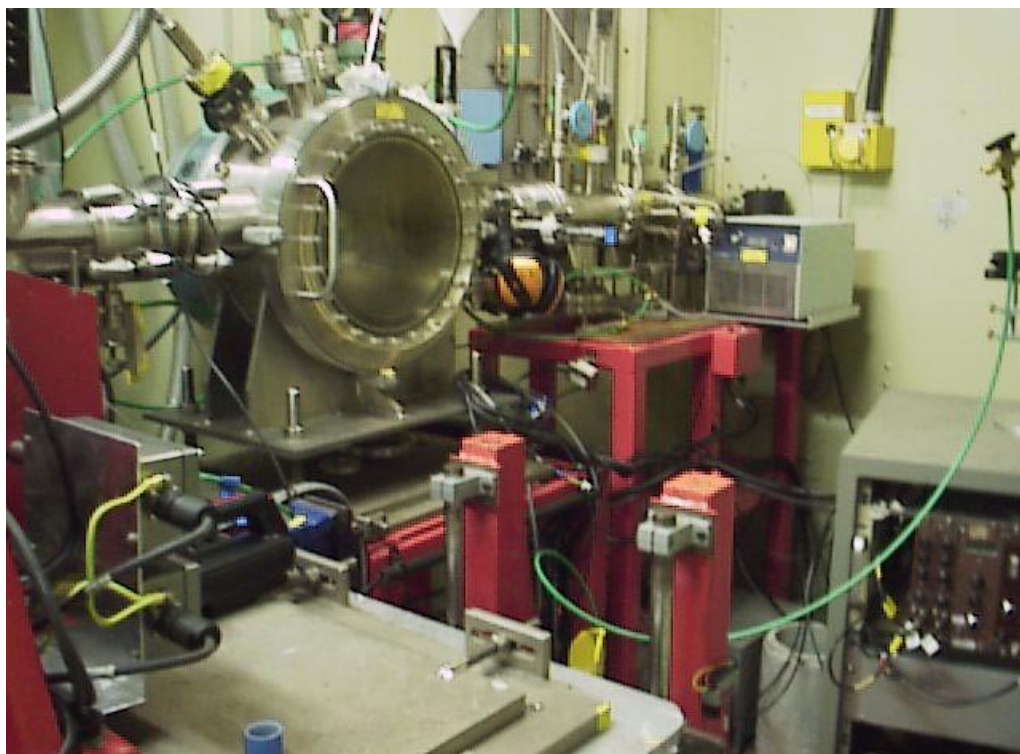
Fig. 5. Monochromator chamber and the beam pipe.

The emergent monochromatic beam is defined by centre opening slits before the sample position. The slits are housed inside a beam pipe linked to the monochromator chamber, and have an operational maximum and minimum aperture 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-rays are recorded by the detector on the 2θ arm. These detectors are described in sections 4.4 and 4.5. 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. 6 and the maximum flux is at wavelengths between 1.1 Å and 1.5 Å.
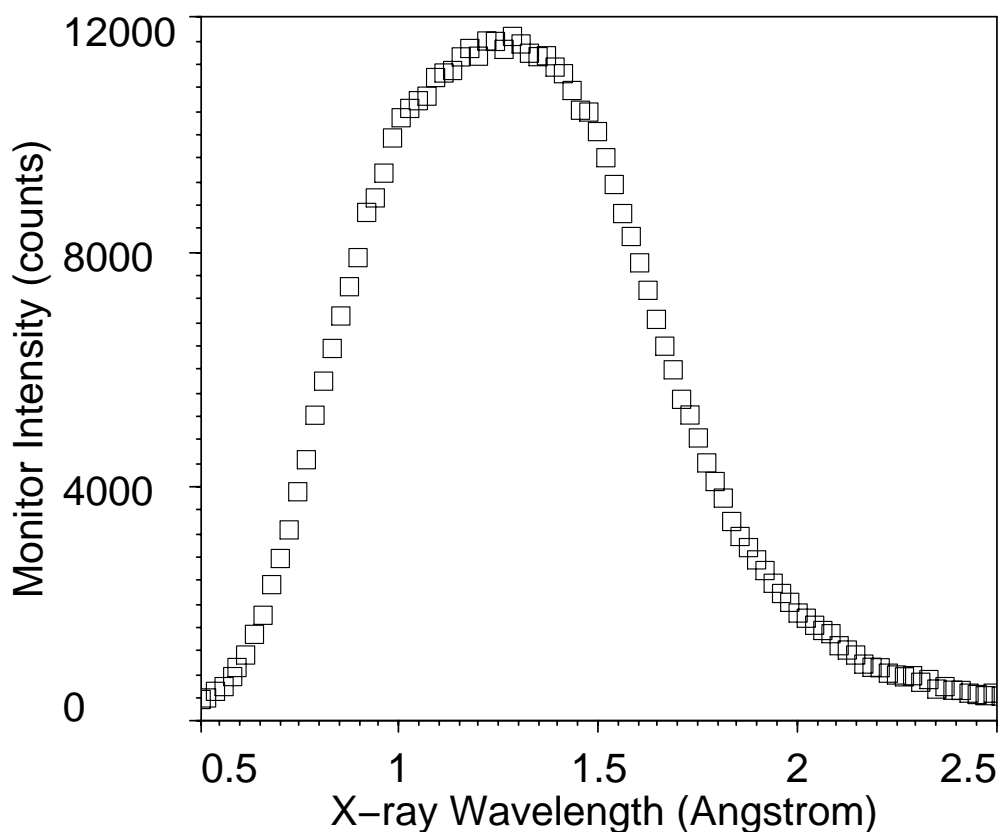
Fig. 6. Incident photon flux as a function of x-ray wavelength (λ)

measured using the beam monitor.

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3.3 Diffractometer

The diffractometer consists of two concentric Franke tables (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 high precision Heidenhain encoders and fed back to the rack to provide feed back to the drive system. The θ- and 2θ-circles are controlled independently with resolutions of 0.2 and 0.1 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, sometimes the drive system may lose track of the positions, and it is then necessary to re-calibrate the two axes. Usually when the positions have been lost, both circles will read zero. The upper and lower limits and other useful information about these motors are given in Table 4.

The diffractometer 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, particularly in reflectivity work, it is necessary to use a Huber goniometer (section 3.4.2). The arcs and translation (rot1, rot2 and trans) on the device 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	motor calibration	smallest step size	lower limit	upper limit
2θ	10 000/deg.	0.1 mdeg.	-30 deg.	130 deg.
ω(θ)	5 000/deg.	0.2 mdeg.	-180 deg.	180 deg.
mono	8 000/deg.	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	10 μm	-10 mm	10 mm
chi	10/deg	0.1°	-5°	95°
phi	100/deg	0.01°	0	180°

3.4 Sample Holders

3.4.1 Flat-Plate and Capillary Spinners

For flat-plate powder experiments, the sample is loaded to a disk holder (perspex) with three standard sizes; 15, 20 and 25 mm in diameter and 0.5-1.0 mm in depth. A limited number of holders are provided to a new user group. However, the holder design is available for user production 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 capillary mode, the flat-plate holder is replaced by the STOE goniometer spinner. With the aid of the telescope, the sample can be aligned with the spin axis at the centre of rotation. Again, the rate of spinning is controlled as 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. 7 and 8.
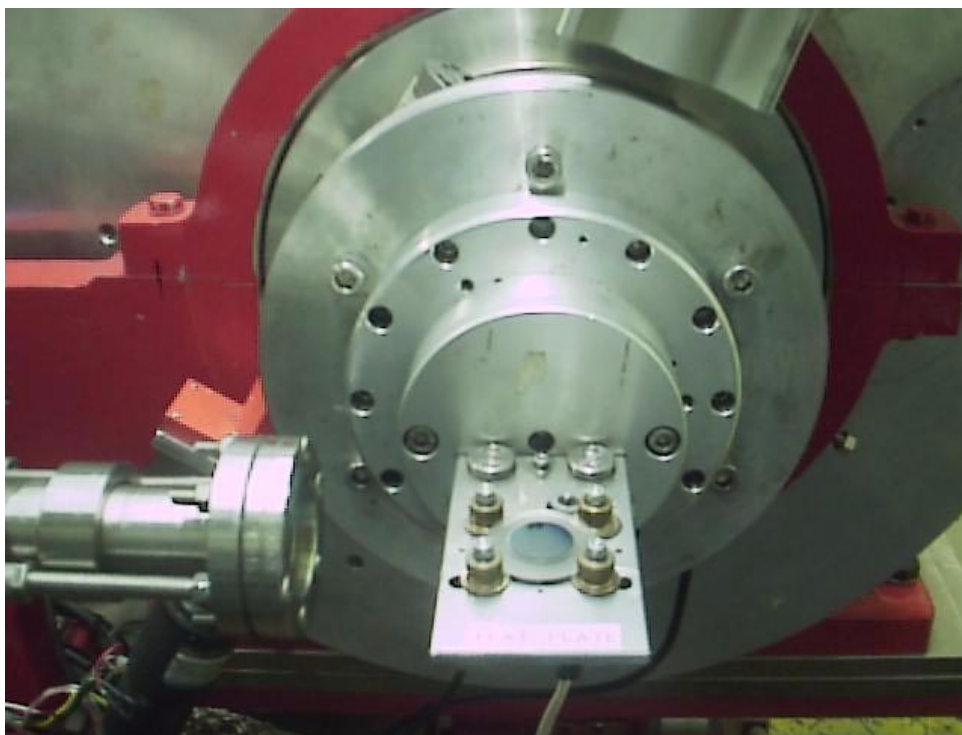
Fig. 7. Flat-plate sample spinner and the perspex holder.

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 20x25 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 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. 9 is a photograph of the device. In addition, the resolution is defined by single slits assemblies on the 2θ arm and these are described in detailed later (section 4.3) and in the paper by Tang et al. [5].
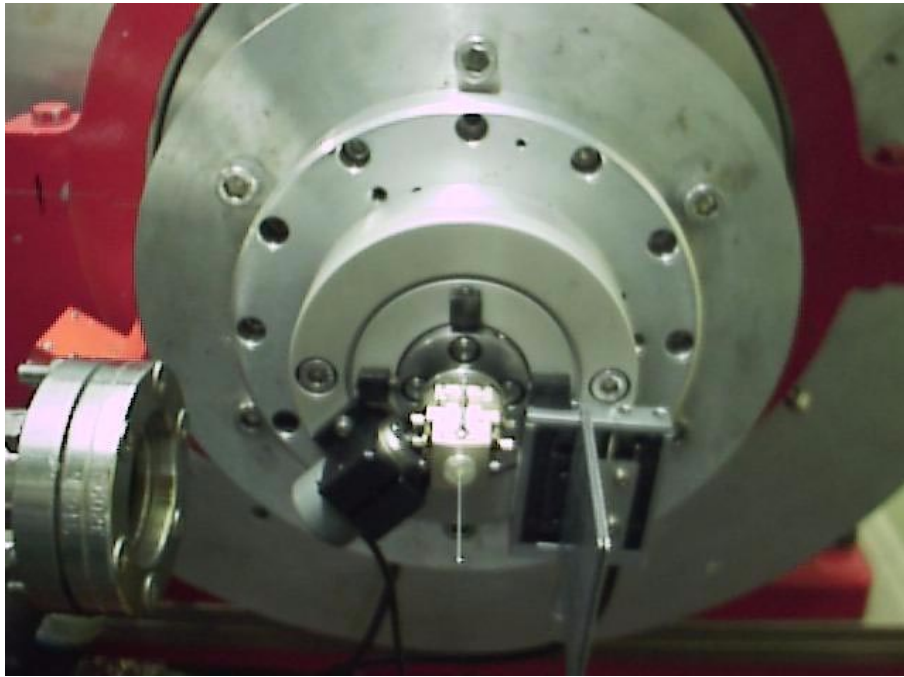
Fig. 8. A capillary specimen on the goniometer spinner.
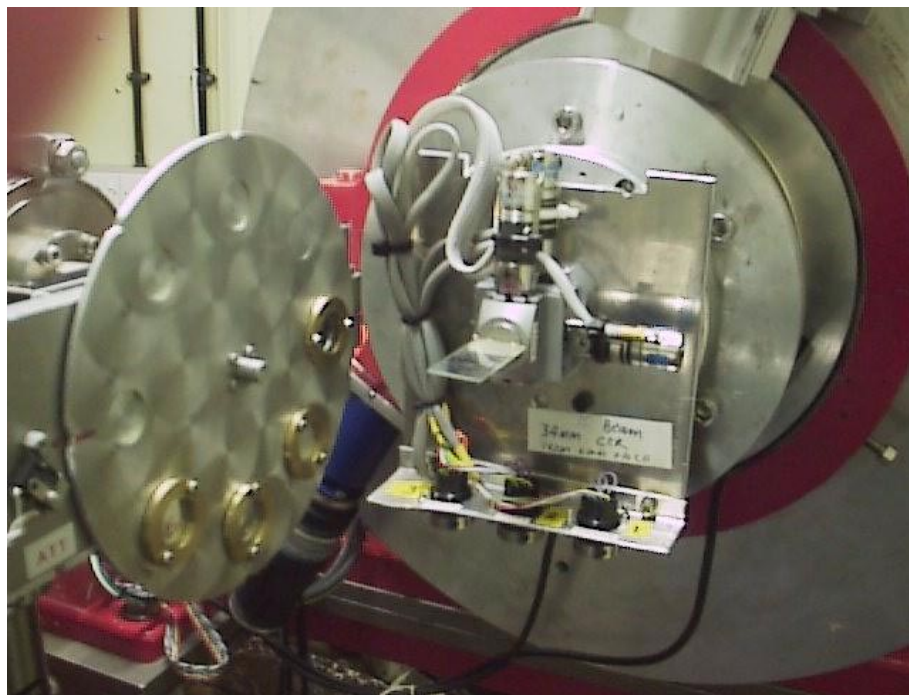
Fig. 9. Motorised goniometer stage for reflectivity sample

3.5 The Euler Cradle

A low resolution χ arc and a small ϕ circle on the Euler table can be mounted on the ω-circle to provide two more rotations. The χ arc, which has a range of 100°, can be used to rotate the ϕ circle (0-180°) from the horizontal plane to the vertical scattering plane. The χ circle is defined to be at 90° when the ϕ-axis is in the scattering plane. These rotations have been used in stress and texture measurements from bulk materials [7,10].

4. Resolution Slits and Detectors

4.1 The 2θ Arm

The arm mounted onto the 2θ-circle is an X95 optical bench (Fig. 2) which facilitates quick and user friendly changeover from one diffraction geometry to another. These geometries are described in Chapter 2. The resolution foils assembly, single slits, vacuum tube and detector (described later) are all fitted with standard X95 clamps. The aperture centre of each device has been accurately pre-aligned along the optical axis which is coincident at the centre of rotation of the diffractometer. Therefore, they can be readily mounted on the arm when required with a minimum re-alignment effort from the user.

4.2 Parallel Foils

The short vertical parallel foils (VPF) of 5 cm length is used for reducing axial beam divergence to improve the line shape at low angles. The VPF foils have an acceptance angle of 1.1° and they are housed in a vacuum vessel. Placed behind these slits are long parallel slits. There are two sets of long slits which are constructed from multiple foils of stainless steel. A new set of parallel foils (PF1) with 100 μm spacing (manufactured by Rigaku) are intended to be used to give an improved resolution. However, the original foil assembly (PF2) with 200 μm spacing (manufactured by the laboratory) giving a nominal resolution of 0.064°, are normally dedicated to the instrument for user application. Table 5 gives a selection of the foil assembly specifications, resulting in an enhanced nominal resolution of 0.03° for the PF1 slits.

Table 5. Specification for the VPF, PF1 and PF2 parallel foils available on Station 2.3.

	VPF foils	PF1 foils	PF2 foils
Foil Spacing (μm)	1000	100	200
Foil Length (mm)	50	365	355
Foil thickness (μm)	50	50	50
Foil flatness (μm)	~50	~10	~10
Aperture (mm^2)	20x20	20x20	20x20
Number of foils	~20	~130	~80
Foil material	Stainless steel	Stainless steel	Stainless steel
Angular aperture	1.1°	0.03°	0.06°

The PF1 or PF2 slits define the resolution of the instrument and they have an aperture of 20×20 mm^2. The resolution effect and collimated peak shape of these foils have been extensively studied by Roberts and Tang [11]. The angular resolution as a function of 2θ angle obtained from a high quality BaF$_2$ powder sample is shown in Fig. 10. The variation of the full-width at half-maximum (FWHM) of the Bragg reflections as a function of 2θ angle is plotted.
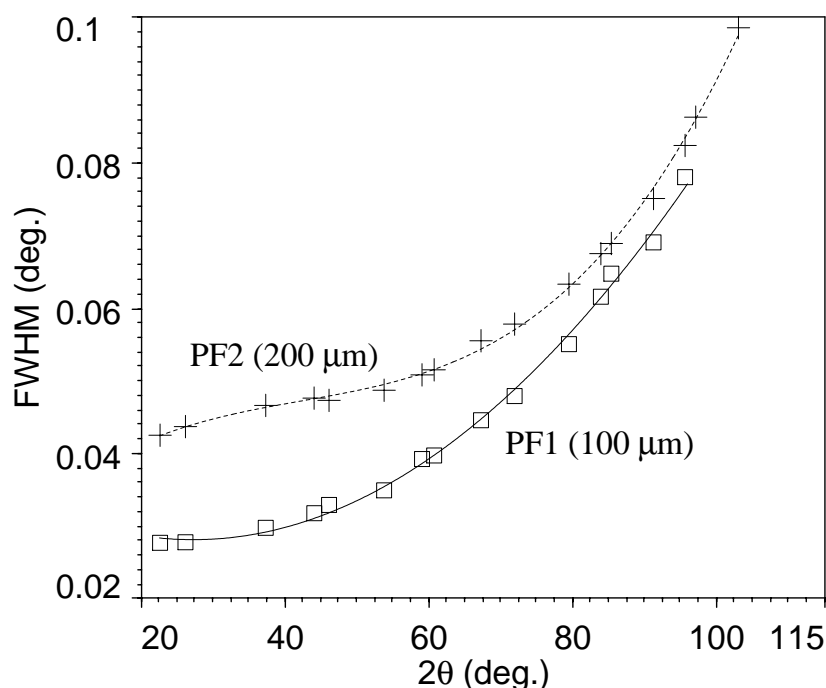
Fig. 10. FWHM as a function of 2θ angle measured from BaF$_2$ powder using the PF1 (data in open squares) and PF2 slits (data in crosses). Data were taken from reference [11].

Although the gain in resolution at low angle ($2\theta \leq 40°$) is 35% when the PF1 slits is used, a reduction in intensity of 57% is expected. In addition, the asymmetry in the peak profile and the tail features measured from the PF1 slits are more pronounced than those observed from the PF2 slits [11]. The parallel foils are housed in a vacuum vessel to reduce the background scattering level and the effects of air absorption. For most flat-plate powder measurements, the incident and diffraction intensity are recorded by the Harshaw scintillation counters and the enhanced dynamic range (EDR) detector. These detectors are described in sections 4.4 and 4.5. For a small capillary sample (diameter ≤ 1.0 mm), a single slits arrangement should be used instead of the parallel foils assemblies.

4.3 Huber Single Slits

In reflectivity measurements, a thin film or multilayer sample is placed on the large Huber goniometer (see section 3.4.2) which is mounted on the ω-circle. The instrumental resolution can be much improved by using the single slits arrangement on the 2θ arm. Two pairs of slits are used; the scatter and resolution slits, both of which have an adjustable centre-opening aperture. Typically, the vertical opening of these slits is set to 100 μm wide. Depending on the sample width, the horizontal opening can be set accordingly (up to 10 mm wide). They are mounted on the 2θ arm at 15 and 70 cm away from the sample. The scatter slits are placed nearer the sample position (15 cm), while the resolution slits are placed nearer to the detector location (70 cm away from the sample).

A Gaussian FWHM of 0.0081±0.0004° resolution measured from an attenuated incident beam of 100 μm x10 mm size has been recorded as shown in Fig. 11. Using an enhanced dynamic range (EDR) detector (section 4.5), reflectivity and diffuse scattering measurements can be performed. A detailed description of the reflectivity experimental set-up can be obtained in reference [5].
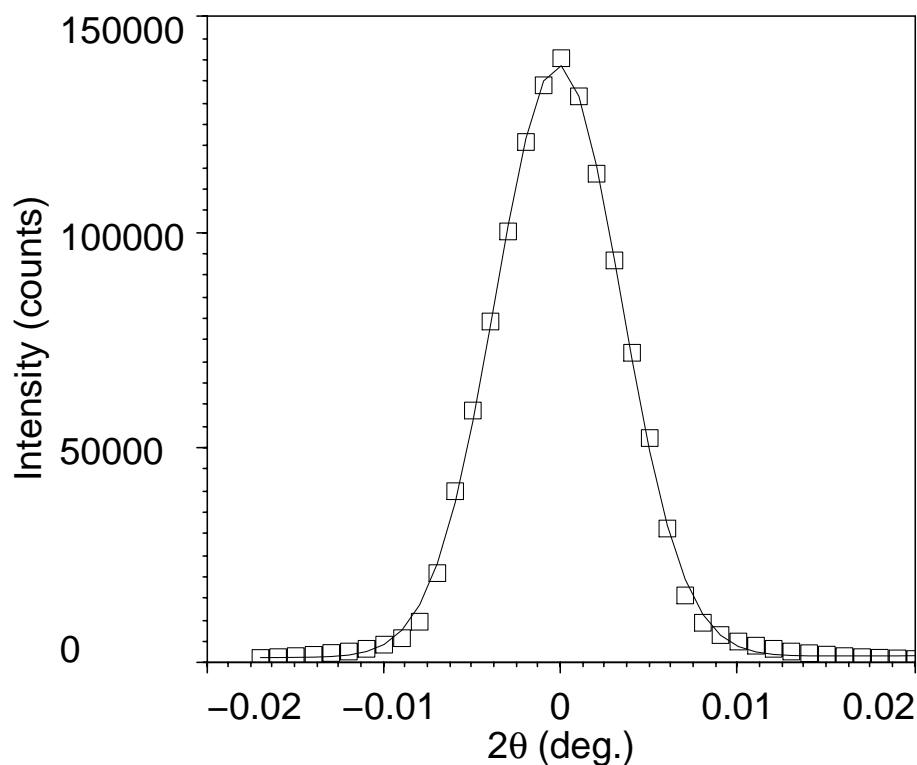
Fig. 11. The peak shape of the beam profile convoluted with the single slits arrangement. The intensity profile (open squares) can be described by a Gaussian function (solid line) [5].

4.4 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 4.6). The monitor is measuring the scattered monochromatic radiation from the kapton foil inside the beam (see section 3.2 and Fig. 1). The other detector can be mounted 90° above the sample to record the fluorescence signals for anomalous scattering studies. The saturation rate of both devices is about 4×10^5 counts per second (cps). It should be emphasised that dead-time correction is more necessary when the count rate is more than 20×10^4 cps.

On the back of each detector there are 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 plug with low voltage (±24V) supplies power 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 6.

Table 6. 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 ($\approx$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 connects 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 Meters (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 6.

4.5 Enhanced Dynamic Range Detector (EDR)

The scintillation counter on the 2θ arm is an EDR detector [12] which is manufactured by Bede Scientific Instruments. 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 (~0.5 cps) and is capable of detecting peak count rates of more than 450x10^3 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 a 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 placed in the electronic rack in the control area (above the hutch).

The three voltages displayed on the panel are listed in Table 7. 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 ($V_W \approx 1.5$ volt) above the threshold level, the high energy photons from the monochromator ($\lambda/3$ contamination) and cosmic rays can be discriminated.

Table 7. 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.

Below each indicator LED on the panel is a potentiometer. To change the levels simply choose the required voltage using the 'SELECT' button and turn the potentiometer accordingly. Note that the setting for V_{TH} and V_W in Table 7 were obtained using a photon beam of energy of 8.9 keV. More details about the detector can be found in the manufacturer's manual (listed in section 1.2) which is available in the station, and in reference [12].

4.6 Instrument Electronics and Control Computers

The control hardware and drive systems are situated on the gantry above the station hutch. There is an electronic rack, shown at about the centre of Fig. 3, which contains the detector electronics (amplifiers and ratemeters). The ORTEC 449-2 ratemeters are connected accordingly to indicate the analogue signals of the monitor and detector. These are quite useful for checking 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. The other rack (right-hand side of Fig. 3) contains the McLennan DC motor drive systems for the omega (or theta) and two theta circles, and the stepper motors described in section 3.3. The Viglen pentium PC named "Stn-vig" on the left-hand-side is linked to the McLennan systems and the stepper motor drive *via* the CAMAC multiplexer. The computer facilitates remote control of the instrument and data collections with the x-ray beam on. This machine is linked to the centre PC named "BL23vig" (Fig. 4) so that the computing tasks can be performed in the enclosure as well. By clicking the "Control 2.3 Station" icon on the BL23vig monitor screen, an invitation menu will be displayed for the password before the linkage is activated. The user name for both machines is "**xrd23**" on the "**dl**" domain. The software which drives the instrument and performs data collection is described in the next chapter.

5. Pincer Control Software

5.1 Software Configurations

The control system is based on the PINCER data acquisition software [13,14] which is now in widespread use on XRD stations. The software is based on a powerful command interpreter and provides a large suite of functions that can be combined in 'macro' command files. On Station 2.3, PINCER runs on the Viglen personal computer (pentium) which controls the instrument. The user name is "**xrd23**", password is "to be notified" and domain is "**dl**". If for some reason logging in has failed after several attempts, a direct access to the PC is possible without the entering of these identities. The PINCER software can still be used but the automatic transfer of data files (see section 5.8) will not be carried out.

The data acquisition software can be activated by clicking the PINCER icon and then following on screen instructions to bring up the windows shown in Fig. 12. Instructional commands have been collected into macros which run simply by typing the name of the macro at the prompt in the "Pincer Command Window". PD and other macros have been written to allow the user to perform nearly all the standard functions. Menu information will appear in the "Standard Input/Output Window". Note that the graphic window will only be permanently displayed after the first scan has been executed. For more details about PINCER, the reader is referred to the relevant documents listed in section 1.2.
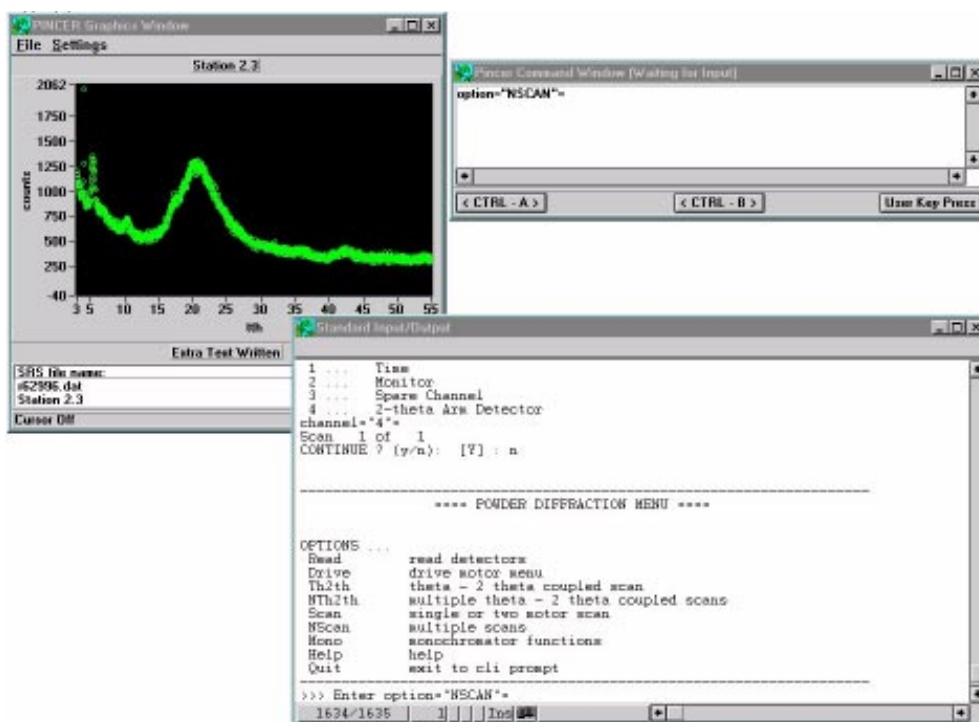
Fig. 12. The window display of PINCER software.

As Station 2.3 performs a wide range of research activities, the PINCER software allows the user to pre-set the experimental configurations. This operation will conveniently include or exclude certain command lines. 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 PDCONF (lower or upper case are acceptable), and the following menu will appear in the Standard Input/Output Window.

```
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, if data file is 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 powder diffraction macros are collected together, and by entering "PD" the following menu will appear on the window.

```
  ----------------------------------------------------------------
              ==== 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
 Help      help
 Quit      exit to cli prompt
  ----------------------------------------------------------------
>>> Enter option="QUIT"
```

The function of each option is summarised in Table 8. 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 (sections 5.8 and 10.3).

Table 8. 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 scans. The user is prompted for the scan range, step size, counting time and the number of times the scan is to be performed.
NTh2th	Allows the user to perform a number of theta/2 theta scans 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.
Mono	Allow the user to select or change or set the x-ray wavelength/energy.

5.3 Change of Wavelength (λ)

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

```
  --------------------------------------------------------------
                       ==== MONO MENU ====

Silicon Monochromator
Monochromator angle        ... 11.9688 degrees
Monochromator wavelength  ... 1.3004 Angstroms
Monochromator energy ... 9.5353 keV

OPTIONS ...
 Deg      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 three commands: Lam for wavelength, Kev for energy or D for angle, followed by the value. On screen instructions will appear whatever option has been chosen. Also the monochromator angle, wavelength, or energy can be set using SETANG, SETLAM or SETKEV respectively.

Warning : Normally, the monochromator is calibrated (section 5.4). The re-setting of Lam, Kev or Ang must not be performed unless the user is absolutely certain that the entered value is correct.

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 9 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 reason, they needed to be recalibrated. The procedures described in the Appendices (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 (± 0.0001 Å) and a small 2θ offset (≤ 0.1°). The values and the associated uncertainties are recorded in the station log book. See for example the calibration that appears in section 10.1.

5.5 To Move Motors

Reflectivity or single crystal experiments require the alignment of the sample at the centre of rotation of the diffractometer. The software allows individual motors to be driven when required. The "Drive" option enables the user to perform such operations. Type in "Drive" or "D" in the Command Window to run the motor driving macro and the following menu will display:

```
 --------------------------------------------------------------
           ==== DRIVE MOTOR MENU [Default Speed] ====
 DETECTOR ...
 channels-> time   1,  mon. 20102,   spare   0, detec.   512
 MOTORS ...
 1)   tth  twotheta : 20.0000 deg 2)   ome  omega(th): 5.0000 deg
 3)  mono  monochrom: 11.9688 deg 4)slits      slits :   0.01  mm
 5) table     table :  0.0000 mm  6) rot1 gon rot1 :   0.00 deg
 7)  rot2 gon rot2 :    0.00 deg 8)trans  gon tran :   0.76  mm
 9)   chi  crad chi :  90.000 deg 10)  phi  crad phi :  0.000 deg
 11) scat  sca slts :  0.0000 mm 12)spare         :      0 stp
type motor number or motor name in full
OPTIONS ...
Pos       drive to position    |  SET      redefine position
Inc       drive by increment   |  Help     help
Att       drive attenuator     |  Jog      1 motor jog
Quit
 --------------------------------------------------------------
>>> Enter option="QUIT"=pos <enter>
motor="tth"=ome <enter>
position=5=10.052 <enter>
```

The motors and definitions are listed in Table 9. The line immediately below the DETECTORS shows the counts in one second on the monitor and the detectors. Again, these readings are useful information. By using the pos command as shown in the above example, the user has moved the omega circle from 5° to 10.052°. When the move has been 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 increments (inc command) as well as absolute values (pos command). If the positions of these motors need to be redefined, type in command set and follow on screen instructions.

Caution : Do not reset a motor position unless it is absolutely necessary. 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. When the user is finished with this menu, type in `Q` to get back to the PD menu. All angular motors turn in degrees and linear translations move in millimetres.

The user can abort a driving motor by simply clicking the <CTRL-B> or <CTRL-A> button in the "Pincer Command Window". The first button will abort the drive command and return back to the menu, while the second button will return back to the Pincer prompt. The software will retain all motor positions. By restarting PD menu (see sections 5.1 and 5.2), all motor positions will be available.

Emergency stop: If all fails press an "EMERGENCY" button to cut off the power. This operation may lose the motor positions. Emergency buttons are on the diffractometer, Mclennan unit and in the control gantry.

Table 9. 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	Large arc on the Euler table
phi	Small circle on the Euler table
Att	Incident beam attenuation wheel with 0, 3, 2, 1, 0.7, 0.24, 0.14 mm thick aluminium absorbers.

5.6 Data Acquisition

After sample alignment (may not be necessary for powder work) and instrument calibration, data collection can commence using an appropriate beam size and x-ray wavelength. 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, if the user wishes to perform a $\theta/2\theta$ scan from 10° to 120° with step size of 0.01° and counting time of 2 second per point, the commands on screen (Command Window) will display as follows;

```
th <enter>
two theta start [10] : <enter>          (default value=10°)
two theta end [11] : 120 <enter>         (for 120°)
two theta step [0.01] :                 (default value=0.01°)
counting time seconds [1] : 2 <enter>    (for 2  seconds)
Number of scans with these parameters [1] : <enter>   (1 scan)
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>
```

The scan parameters, scan number and other information will be displayed on the Input/Output Window.

Warning : When the computer displays the following message:

```
Smain warning : Monitor counts are low.
- check X-ray shutter, ports and the SRS beam status!
```

The warning message means that there are no x-rays in the hutch beam pipe because, for example, the shutter is still closed. If there are 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 the plot of two-theta versus intensity will appear on the "PINCER Graphic Window". The data will be saved in a file labelled as "r12345.dat" that will appear at the lower-left of the window. 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 again. Examples of other scan options are given in the Appendices (section 10.2). At any time during a scan, it can be interrupted by pressing <CTRL-B> and waiting 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 ...
          use graphics Window to Rescale, Scale and Plot
          before printing graph, select the white background
Cli       temporary CLI prompt
Abort     abort the scan (the data will be save to file)
Quit      quit from this menu and continue scan
----------------------------------------------------------------
>>> Enter option="RESCALE"=
```

The menu above allows the user to change the plot scale or to abort the scan. The scan remains suspended until the user selects an option from the menu. The user may type in an option accordingly. By clicking the <CTRL-A> button, the scan will terminate immediately. Either operation will save the data in the files. When the scan is finished, another menu will appear as follow:

```
       --------------------------------------------------------------
                     ==== SCAN FINISHED MENU ====

OPTIONS ...
              use graphics Window to Rescale, Scale and Plot
              before printing graph, select the white background
Info          Display san info and statistics
Cli           temporary CLI prompt - terminate with 'return'
Quit          quit menu, and finish
       --------------------------------------------------------------
>>> Enter option="QUIT"=
```

For a hardcopy, the user should use the option in the graphics window and the plot is dumped on to the Lexmark laser printer placed in the gantry. When option Quit is entered, the scan is completely finished and the data file will be transferred automatically (section 5.8).

5.7 Beam Down Delay Options

With the beam down delay functions switched on (options 7, 20 and 21 in section 5.1), a running scan will pause when the x-ray beam is down as shown in the example below. Once the beam is back the count down routine will be activated automatically, and data collection will only resume when the specified delay time (e.g. 2700 sec) has been reached. The user can terminate the count down and continue the scan by clicking the <CTRL-B> button and enter option Term as shown below. Other options are also available.

```
Beam lost, counting down  2700 secs
SRS file name r12345.dat


  --------------------------------------------------------------
                  ==== BEAM MONITORING MENU ====

OPTIONS ...
              use graphics Window to Rescale, Scale and Plot
              before printing graph, select the white background
Cli           temporary CLI prompt - terminate with 'return'
Mon           continue monitoring beam
Abort         abort the scan (the data will be saved to file)
Term          terminate count down and continue scan
Quit          quit menu, and finish
       --------------------------------------------------------------
>>> Enter option="MON"=
```

5.8 SRS Data Format and File Transfer

Data files are named rxxxxx.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 previous 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 stored 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 the "/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 will only be there for a few days and they will be deleted in order to maintain sufficient disk space to accumulate new data files and 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. A Viglen pentium PC and a Silicon Graphics work station in the enclosure are for users to do their data analysis. Graphics and peak refinement programs are available on these computers. More programs are available in the CCP14 suite. Documentation on the software and the computers are listed in section 1.2. The work station can access the central computers for the PD programs described in this chapter. The PC has "PeakFit" (Jandel Scientific) 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 shape can be described (to a good approximation) 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 programs 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. To fully describe these programs is beyond the context of this manual, but brief introductions are given in the following sections.

6.2.1 Data Display (PLOT86)

The PLOT86 graphics 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. In that case only one set of observed, calculated and difference data can be plotted. A cursor can be used to measure 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 the current of the electron beam in the storage ring decays with time. To correct for the beam decay effect, the program PODSUM can be used to normalise the raw data against the monitor count. The software can only performed on the data file that is POD formatted (section 5.1). This program has an interactive help system and it is also useful for adding several data files together with normalisation. An upgraded version that will take various forms of SRS data format is currently being developed.

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, trial lattice parameters and symmetry of the unit cell. These values can be entered from a keyboard or from a file. The program performs refinement 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 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 SPG 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 for input to the 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 required 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 as good a 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 the case, 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 co-ordinate positions are refined. A profile is fitted to the PD data, and the structure factors are determined from the intensity of each reflection. The program MPREP is needed for preprofile preparation for MPROFIL.

6.3 Motplot

Although it is still at a development stage, "MOTPLOT" is now available in "xrdsv1" from the work station (xrdws6). This is a new window package which can be used to plot SRS data files. The software can be activated by typing in "motplot" and up to 100 data files can be read in using the "command" window. As the software is designed to be user friendly, a quick demonstration by the instrument scientist to a user should be sufficient for him/her to learn how to use it. On screen help is also available. Graphs in postscript form can be printed on the laser printer (Ps23psp) in the enclosure or they can be printed directly on any one the printers in the computer centre.

6.4 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 offer graphic routines such as "PLOTEK" and "PLOT86". Manuals can be obtained from the computer help desk.

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

7.1 Introduction

As safety is paramount, all users must watch periodically the laboratory safety video which is available on machines in the coffee lounge, the B-block foyer and the hostel. In order to let the 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 be 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 are 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 (on the gantry upstairs or in the enclosure), 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-activate the system and then **F2** to open shutter or **F1** to close shutter.

On screen instructions are displayed on the TANDY computer and sometimes it is necessary to press **F4** button to reset.

Attention: Quite often the shutter fails 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. On screen instructions are displayed on the TANDY computer to overcome other problems. If however a fault 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 is 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 down. If this has happened you must contact the personnel in the control room at once. The lever on the inside of the hutch door is designed 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.

7.4 Risk Assessment Form and Log Sheet

As an additional safety requirement, an experiment must have a **Risk Assessment** form displayed in the Main Control Room and on the board outside the hutch. Failure to comply could have result in experiments being stopped without prior notice. The user is required to fill in and sign a **SRS Station Log Sheet** when his/her experiment has been completed. The sheet should be left on the station or in the enclosure.

8. Trouble Shooting

Problem	Suggestion
No signal in the detector	Check if monitoring detector or detector HT is off
No signal in the monitor	Beam shutter not opened or detector HT is off.
Shutter opened but no beam	Port 2 not open (absent on TV monitor)
Port 2 not opened	Beamline vacuum problem (contact the control room or the station scientist)* or no SRS beam (wait for the beam).
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 (user ID: xrd23, domain:dl).
Stop motor driving	Click <CTRL-B> button to stop. If this failed try <CTRL-A> (see section 5.5).
To abort a scan	Click <CTRL-B> or <CTRL-A> button to abort (see section 5.6).
Auto data file transfer failed	Network is down. The transfer will resume automatically once the network is back.

The problem is serious and the port co-ordinator or the station scientist should be contacted at once.

**The problem is serious and the station scientist or the electrician should be contacted at once.*

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 two axes XRD diffractometers.

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 histogram 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

X-Ray Diffraction project at the SRS, headed by Dr. Graham Bushnell-Wye, including 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 flat-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 > prompt on the Pincer Command Window and the computer will ask the following questions.

```
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 (optional) <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.
- check X-ray shutter, ports and the SRS beam status!
```

This means that there are no x-rays in the hutch beam pipe because the shutter is still closed. If there are still no x-rays after the shutter is opened this means problems further upstream. The user 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 nine Si reflections 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] :
recalibrate two-theta axis
CONTINUE ? (y/n) : [N] :
recalibrate monochromator
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 theta axis when invited by the macro.

10.2 Scan Options

10.2.1 Theta-2Theta (Th2th)

```
Theta - 2 Theta scan
two theta start [10] :                    (default value=10˚)
two theta end [11] : 120                  (for 120˚)
counting time seconds [1] : 2             (for 2  seconds)
Number of scans with these parameters [1] : (default=1)

Number of data points . . . 11001
Predicted scan duration . . . 4.3 hours
Plot graph with LOG scale? (y/n) : [N] : (default=No)
CONTINUE ? (y/n): [Y] :                   (default to start scan)
>>> Enter title:                         (title may be typed in)
>>> Enter condition 1 :
>>> Enter condition 2 :
>>> Enter condition 3 :
Moving tth to start position 10.00000
Moving tth to start position  5.00000
Scan finished ... File name r12345.dat

SRS file name r12345.dat
```

The scan will start when the motors have reached the starting positions.

10.2.2 Ordinary Scan (Scan)

The example below is a scan of the scatter slits from -1 to 1 mm with a step size of 0.05 mm.

```
motor="tth"=scat
start1=0=-1
end1=1=1
step1=0.1=0.05
Type in motor2 or <return. for single motor scan [] :
Type in counting time per point in seconds [1] :
Type in channel to be plotted
1 ...Time
2 ...Monitor


3 ...Spare Channel
```

```
4 ...2-theta Arm Detector
channel="4"=                              (to plot 2-theta detector)
CONTINUE ? (y/n): [Y]:                    (No, if mistakes were made)
>>> Enter title:                          (title may be typed in)
>>> Enter condition 1 :
>>> Enter condition 2 :
>>> Enter condition 3 :
File name r12345.dat
Moving scat to start position -1.000
Scan finished ... file name r12345.dat
```

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

10.2.3 Multiple Theta-2Theta Scans (NTh2th)

```
******************************
Multiple Theta - 2 Theta Scans
******************************

scan parameters file(or <Enter> to type in scan parameters []:
total number of scans [2] : 3                    (for 3 scans)


***********
scan no.  1
***********
two theta start tthst [1]=10=
two theta end tthe[1]=11= 50
two theta step tths[1]=0.01=
counting time seconds sec[1]=1=


***********
scan no.  2
***********
two theta start tthst [2]=50= 50
two theta end tthe[2]=51= 90
two theta step tths[2]=0.01=
counting time seconds sec[2]=1= 5


***********
scan no.  3
***********
two theta start tthst [3]=90=
two theta end tthe[3]=91= 120
two theta step tths[3]=0.01=
counting time seconds sec[3]=1= 10
save each scan in a separate SRS file ? (y/n): [Y] :
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] :           (default to start scan)
>>> Enter title: <enter>          (title may be typed in)
>>> Enter condition 1 :

>>> Enter condition 2 :
>>> Enter condition 3 :
```
40

The scans will commence after the motors have reached the starting positions. The above example shows three θ-2θ scans that will be executed. The first scan starts at 10° 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 seconds for each point. The final scan starts at 90° and ends at 120°, with step size of 0.01° and 10 seconds per point. The three scan data sets will be saved in separate SRS files (section 10.3).

10.2.4 Multiple Ordinary Scans (NScan)

```
*************
Multiple Scans
*************


Motor to move between scans
**************************
motor (or <Enter> to ignore) [] :
nscan=1=10 <Enter>          (for 10 repeating scans)


Scan parameters
***************
Number of scans =  10
motor1="tth"=tth
start1=10=
end1=11=120
setp1=0.1=0.01
Type in motor2 or <return> for single motor scan [] :
Type in counting time per point in second [1] : 2

Increment parameters between scans
*******************************
increment counting time by [0] :
increment motor 1 start position by [0] :
increment motor 1 end position by [0] :
execute PINCER command before each scan [] :
execute PINCER command after each scan [] :
Type in channel to be plotted
1 ...Time
2 ...Monitor
3 ...Spare Channel
4 ...2-theta Arm Detector
channel="4"=                     (to plot two-theta detector)
Scan 1 of 10
CONTINUE ? (y/n): [Y]:
>>> Enter title:                 (title may be typed in)
>>> Enter condition 1 :
>>> Enter condition 2 :
>>> Enter condition 3 :
```

Here, the 2θ scan starts at 10° and ends at 120° with step size of 0.01° and 2 seconds per point. In total, 10 scans will 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 (section 10.3).

10.3 SRS Data File Formats

10.3.1 Theta-2theta 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.
       41950.     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 milliseconds. 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 degrees and seconds, 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 (counts). Note that for POD configuration (section 5.1), the angles and time will be in mdeg. and milliseconds, respectively.

11. Acknowledgements

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