

# technical memorandum

Daresbury Laboratory

DL/SCI/TM35E

PROTEIN CRYSTALLOGRAPHY 7.2: WORKSTATION OPERATION

by

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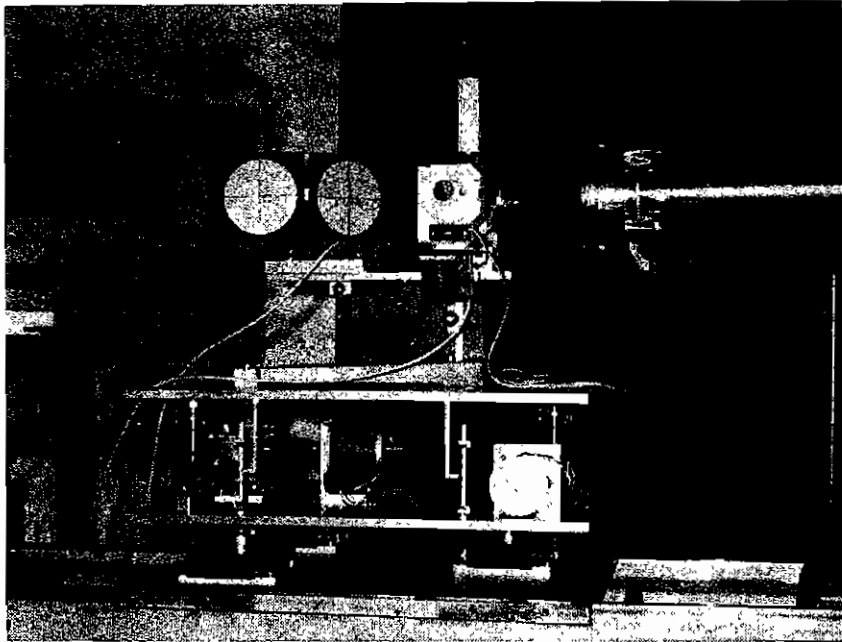
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ABSTRACT

This document gives a description of the routine operation of the P.X. workstation written with the station user in mind. It is only concerned with the practicalities of operation, and contains no detailed wiring or program information. The scanning and film processing facilities provided by Daresbury are also discussed.

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CLEARLY VISIBLE IN THE PHOTOGRAPH ARE:

- A) THE COOLING DEVICE.
- B) THE FLYING BEAM STOP (MK. 1 VERSION)
- C) THE IONISATION CHAMBER AND ITS CABLES
- D) THE LEAD SHIELD AND BEAM TUBE
- E) THE INGRESS PIPES AND PRESSURE GAUGE (ON THE FAR WALL OF THE HUTCH) FOR THE COOLER DRY AIR SUPPLY.
- F) THE NUMBERING ON CASSETTES, CASSETTES HAVE NOT BEEN ASSIGNED A NUMBERED POSITION ON THE CAROUSEL.

Fig.8

It must be clear that the keypad works in an on/off mode. If a button is pressed to start a motor, that motor will continue to drive until the same button is pressed again (i.e. the first operation of a button starts the motor, the second stops it). Operation of a different motor button while one motor is already driving will result in the PANIC function being called. All motors will stop, and the PANIC stop must be re-enabled in order to drive further motors. The only exception to this on/off type operation is the single step option key. When this is pressed, the single step motion is selected and any motor button pressed results in a single step operation of that motor. The single step button should be operated again in order to return to multiple step mode.

### 2.3 Ionisation chambers

The x-ray beam is monitored during the experiment by means of a miniature ionisation chamber, made up of an ebony horseshoe with pieces of copper clad circuit board forming the plates (see fig.3). The chamber is powered by an HT supply (shortly to be replaced by a smaller supply), at a potential of -100 V. The signal received on the second plate is collected by a Keithley 427 current amplifier, with gain normally set at  $10^9$  (i.e. for a 1 volt output, 1 nanoamp of ionisation current has been detected). Table 1 gives typical voltage readings and scale factors for machine energy for different wavelengths through a 0.3 mm collimator (the 1 . blue). Up to 10% error in these readings is acceptable - the alignment of the station depending on systematic errors such as the orthogonality of the beam and the mirror, any small change in beam orbit or steering etc.

This voltage measurement is fed into a voltage to frequency converter, and sampled by a CAMAC scaler. This gives an indication of exposure in terms of v/f counts (The data acquisition program using this method of determining exposures is documented elsewhere). The v/f converter operates between 0 and 10 V, as does the Keithley. Any attempt to read voltages/currents beyond the range of these instruments will result in damage and delay. Please watch your ion chamber reading and turn down the gain if the Keithley reads more than 10 volts.

### 2.4 Pre-monochromator slit setting

The slits are (in normal operation) adjusted to keep the beam off the mount and tip of the triangular monochromator, for reasons of heating and

radiation damage (to the mount). This is achieved by adjusting the slits until the flux reading on the ion chamber just starts to decrease, the slits being best moved on the FAST speed range (Don't forget to change to speed slow afterwards!). The slits marked HORIZONTAL refer to those whose edges are vertical, in other words the slits that control the HORIZONTAL aperture. Thus the slits required to protect the two ends of the monochromator are horizontal inner and horizontal outer.

For crystals with a large unit cell dimension, further sitting down reduces the beam cross fire angle, and hence improves order to order resolution. A reduced monochromator acceptance is also useful in eliminating the often observed monochromator warm up time. Attenuations of (respectively) 50% and 25% would be typical for these applications. However, longer exposure times (pro rata) must be expected.

The vertical slits may be adjusted in similar fashion, but these are not used to control the SR primary beam since the vertical source size and divergence (0.3 mm, 0.25 mRad) is acceptable. A second set of vertical slits exist before the focusing mirror.

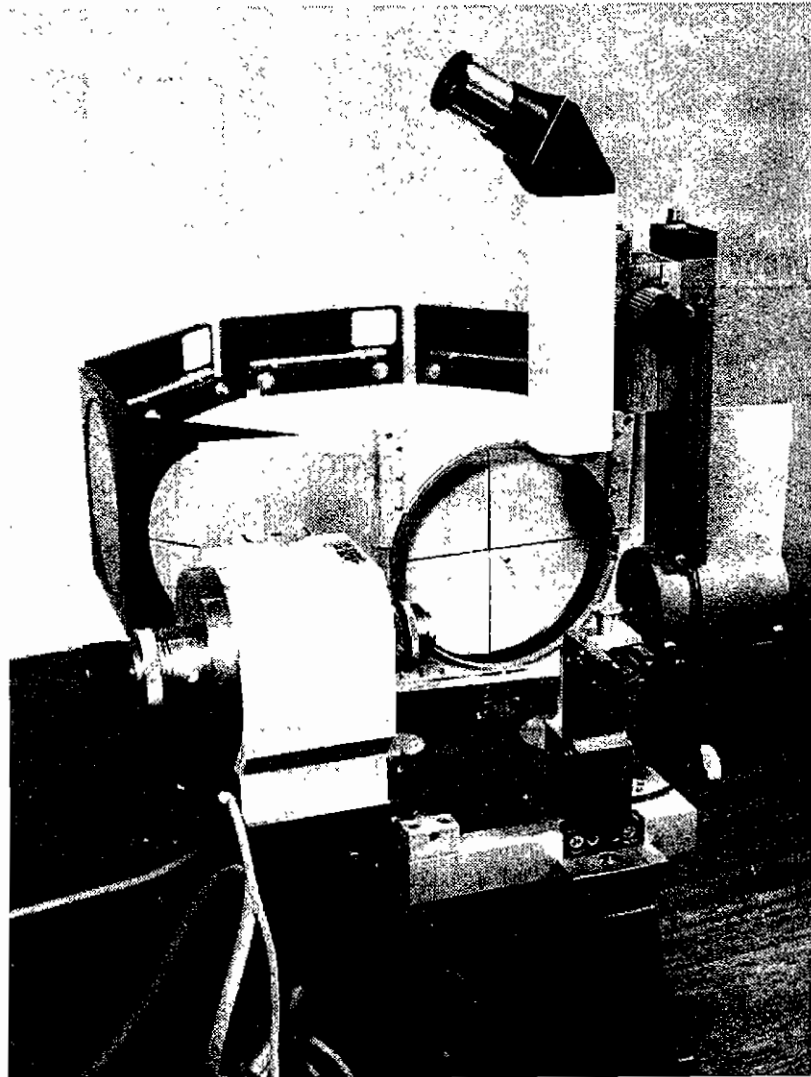
It is MOST IMPORTANT to record any movement of slits. Other users can not be expected to know how you have set the station up! Failure to note this will lead to loss of time for the group to follow. The information which must be recorded to maintain the smooth running of the station is given in Table 2.

### 2.5 Top carriage alignment

The Arndt-Wonacott camera fixes on to a 'top carriage' assembly which allows both vertical and horizontal translations and tilts for the alignment of the camera to the beam. The stepper motor key pad provides buttons for horizontal translation (two horizontal motors driven together), vertical translation (two vertical motors together), and the separate motions (vertical front, vertical back, horizontal front, horizontal back).

A typical alignment procedure (starting from scratch) would be as follows:

- a) Using plastic scintillator (yellow card) find the beam with respect to the collimator (mount to be roughly central to the beam).
- b) Fit the small circular screen to the front of the 4 . red collimator.



THE PHOTOGRAPH ILLUSTRATES THE 'ELEPHANT'S EARS' SCATTER GUARD ON THE ARNDT WONACOTT CAMERA. THE DEVICE WILL ONLY CUT OUT LOW ENERGY MULTIPLE SCATTERING, AND IF THE SCATTER PROBLEM IS BAD WILL PROBABLY NOT BE OF MUCH USE.

Fig.6

cassettes (except the one exposing) from low energy scatter (see photograph in fig.6).

### 3. THE COMPUTER

#### 3.1 General description

The LSI/11 used on the station is situated on the far right of the CAMAC crate in the computer rack. Also mounted in the rack are (below the crate) the dual floppy disk drive, and (at the bottom of the rack) the computer and CAMAC crate power supply. The computer derives its operating system from the SY: or system device, in our case a floppy disc in drive 0 (the right hand drive). The programmes the computer runs are taken from the left hand drive or DK: device.

In order to initialise (boot) the system, the correct floppy discs must first be placed in their drives. The P.X. Catex System disc is placed in drive 0 (r.h. drive), and the P.X. stepper motor drive disc in drive 1 (l.h. drive). The doors of the drives must be closed before the disc drive can read a disc.

On the LSI/11 there is a 3 position switch, the upper position of which is labelled HALT, the centre RUN and the lower INIT. To boot the computer (assuming the CAMAC crate power is on!) the switch is moved from RUN to INIT, and returned to RUN (The switch is of the pull and move type).

The computer will reply:

```
RT11MB (S) V04.00
etc
```

The dot is the prompt of the RT11 operating system. If the computer does not get this far, the following action should be taken:

a) Check the system disc is in the disc drive (0) with the door closed and the ready light up. Check the disc is in the right way round i.e. with the exposed rectangle of the magnetic surface parallel to the top edge of the drive.

b) Check the unit in CAMAC slot 5, (the floppy disc interface) which has two front panel connectors which occasionally pull out or get dirty.

c) Check that the terminal is connected to the LSI and switched on.

d) Check that the terminal 'NO SCROLL' button has not been pressed, by switching the terminal on and off.

e) Switch the LSI power supply off and on (bottom most in the computer rack).

f) Call in the crew/shift scientist.

If all else fails, contact a suitable member of the C.S.E. section through the Main Control Room. Most problems should be user solvable, however.

Once the dot prompt appears, the programme may be initiated by:

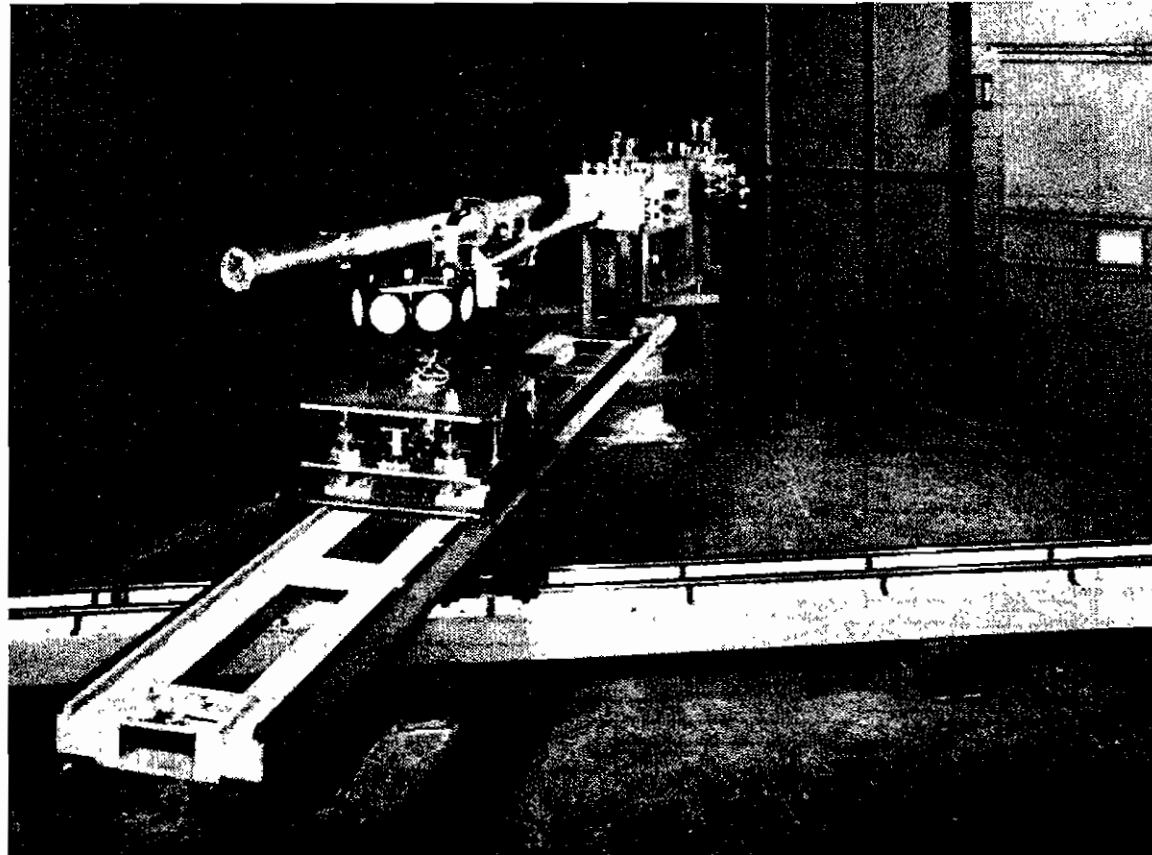
```
a) Type in      CAT(carriage return)
The computer will respond with :
                +CATEX VERSION(2) etc      ....
                +
```

```
b) Type in      IRD PXLOAD(cr)
The stepper motor program will now load and run.
The programme asks for a reference set of motor positions on starting
```

(1) is input to set all motor references to 10000 (note that this does not actually return any motor, but is an arbitrary number to which steps are added or subtracted as motors are moved), and

(2) input to read the last set of positions dumped to disc. Since these are nearly always out of date it is best to initialise to 10000.

The programme bases all motor positions on these numbers, and the current motor positions can be read out by typing in '?'. The gearing of the top carriage transitions is such that the front motor moves in steps of 5 micron, and the rear is 2.5 micron. The vertical translation button on the



THE ARNDT-WONACOTT CAMERA IS MOUNTED ON THE TOP PLATE OF THE ALIGNMENT CARRIAGE ASSEMBLY. TWO VERTICAL JACKS (FRONT AND BACK) DRIVEN BY STEPPER MOTORS RAISE AND LOWER (OR TILT) THE CAMERA. SIMILARLY, TWO SLIDERS PERFORM THE SAME FUNCTIONS IN THE HORIZONTAL. IN THE BACKGROUND, THE STEPPER MOTOR AMPLIFIERS CAN BE SEEN. THESE ARE CONNECTED TO THE CAMAC MULTIPLEXER (VIA THE HIM UNITS ON THE TOP OF THE RACK) BY TWO 15 WAY CANNON CABLES THROUGH THE 'LETTER BOX' CABLE EXIT. THE ENTIRE RACK IS POWERED FROM THE BLUE MARECHAL 3 PHASE SOCKET ON THE HUTCH HALL

Fig.4



in this limb (covering the beam pipe from the CVT valve/shutter to the two Be windows in the hutch) which is monitored on the ion gauge controller in the P.X. control rack.

One of the Be windows in the hutch separates high vacuum and atmosphere allowing the white, unreflected beam to enter the hutch (for alignment purposes). The other separates the high line vacuum from the rotary pump vacuum in the monochromator vessel.

Full instructions on pumping down or letting up the monochromator vessel are given on the pump mountings. NEITHER OPERATION SHOULD BE ATTEMPTED WITHOUT THE STATION MASTER. There are two separate rotary pumps in the P.X. hutch, one of which pumps the monochromator vessel and the other which pumps the beam pipe along the main arm. Since mylar suffers from radiation damage, windows on the beam pipe implode occasionally, but the separate pumping system prevents the monochromator vessel coming up to air at the same time. BEAM SHOULD NEVER BE LET ON TO THE MONOCHROMATOR UNLESS THE MONOCHROMATOR VESSEL IS FULLY PUMPED DOWN.

The monochromator vessel is isolated from the rotary pump by two valves, one just above the pump, and the other in the flexible vacuum pipe. These are normally open, and the vessel continually pumped.

The pump on the beam pipe is isolated in similar fashion, and continually pumped. Should a mylar window fall, however, or the vacuum in the pipe reach a critical upper limit, a pressure switch causes the rotary pump to trip off. To pumpdown again, the pump 'ON' button must be held down (for >1 min) until an adequate vacuum is achieved in the beam pipe. There is no pressure trip on the monochromator vessel vacuum, and this vacuum should be checked daily before light is let onto the monochromator. If vacuum is poor, (i.e. not right on the lower limit of the gauge) check that BOTH isolating valves are FULLY open. If this does not improve the vacuum, then the station master should be called.

## 6. HUTCH INTERLOCKS

The hutch is interlocked to both the pair of shutters admitting beam into the hutch, and the main port shutter admitting beam to all stations on line 7. The former shutters are referred to as LOCAL SHUTTERS, and are under direct control of the experimenter. The main port shutter is controllable only by the crew in the Main Control Room - if it is tripped for any reason, the crew must be asked to reopen it.

Beam cannot be allowed into a hutch until the hutch has been searched (and found to be empty!). The user is forced to look around the hutch whilst walking between push button switches that form the search points. The searcher is responsible for ensuring that the hutch is empty. A search is performed as follows:

- a) Take the key from the switch in the mask control module (situated in the P.X. control rack).
- b) Press the START SEARCH button on the grey search control box to the left of the main entrance to the hutch. The topmost (search started) lamp should light. Once this button has been pressed there remains approximately 40 seconds in which to search the hutch.
- c) Take the key to the first search point (directly behind the main arm on the far wall of the hutch), turn the key in the lock, press the button, turn the key back and remove the key. Note that the key is only removable in one position. Proceed to the second search point (in the left hand corner of the hutch).
- d) Press the button on the second search point.
- e) Press the button on the third search point (just inside the door of the hutch).
- f) Close the door GENTLY. Slamming the door often glitches the magnetic limit switches and drops the search.

# DISPLAY



Do not use

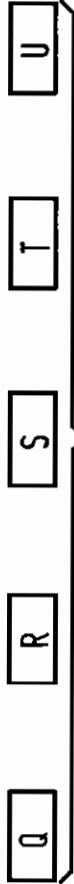
Vert. bottom pre-slit  
 Vert. top pre-slit  
 Horiz. inner pre-slit  
 Horiz. outer pre-slit  
 Vert. top post-slit

Vert. bottom post-slit  
 Horiz. outer post-slit  
 Horiz. inner post-slit  
 Mono X-axis  
 Mono Y-axis

B Mono Z-axis  
 C Mono rotation  
 D Mono B<sub>x</sub>  
 E Mono B<sub>y</sub>  
 F Not used

G Not used  
 H Horiz. front carriage  
 I Horiz. back carriage  
 J Vert. back carriage  
 K Vert. front carriage

L Horiz. translation carriage  
 M Horiz. rotation  
 N Vert. translation carriage  
 O Vert. rotation  
 P Not used



Not used

Q  
 R  
 S  
 T  
 U  
 V Abort programme  
 W Single/multiple step  
 X CLW/ACLW  
 Y Fast/slow  
 Z Panic

Fig. 2

## 7.2 The dark room

Darkroom practice will be well known to all readers of this document. However, some points are worth mentioning:

- a) Supplies of developer and fixer are available both in the darkroom, and in the sample prep. room (ancillary lab area). It is worth checking with the previous group on the age of the chemicals. Whilst Daresbury staff try to keep an eye on the state of chemicals, it is not always possible to find time to replace or replenish them.
- b) Keep the darkroom as clean as possible, and mop up all spillages.
- c) Film supplies are kept in the large refrigerators in the sample prep. room. Both CEA Reflex 25 and Kodak No Screen are available. Check the dates on the packages.
- d) Distilled water for film washing is available in the Chemistry lab.
- e) The film cassette lids and bodies are all adjusted via three PTFE screws to prevent film slippage after fiducials are exposed, and both cassette and lid marked with their own number. Please keep the relevant halves together for your own data's sake.
- f) Should the N<sub>2</sub> cylinder run out, contact Jim Sheldon (bleep 173).
- g) The rate of data acquisition is often limited by development time - if the fixer tank starts causing a bottleneck, repack exposed films and develop them at home. Occasional films may be developed to check for radiation damage to sample.
- h) A polaroid cassette holder (donated by North and Khorber) is available for sample setting on the synchrotron.
- i) Please record use of film and state of developer in the station log. (See Table 2).

## 7.3 Other equipment available

The following items are available for use:

- a) Four sets of Stoe goniometer arcs, and two Bragg-Nonius arcs.
- b) A micro manipulator (sample prep. room)
- c) An optical goniometer (sample prep. room)
- d) Two binocular microscopes (sample prep. room)
- e) Soldering iron for wax melting (sample prep. room)
- f) Table and angle poise lamp for sample mounting in cold room. (Cold room situated in Biological Support lab.)
- g) A 'flying' beam stop device is available, which can be used to expose the direct collimated beam on a film. Two power supplies are required to operate this device, and these are wired according to fig.9. The voltage on the solenoid and the timing potentiometer should be tweaked to give a suitable exposure time and beam stop displacement. A fuller description is given in ref.(6)
- h) A helium cone for the Arndt-Wonacott camera is available.

## 8. TROUBLESHOOTING GUIDE

### 8.1 Beam intensity incorrect

Should the beam intensity (as given in Table 1) be too low:-

- a) Tweak up carriage using motor buttons:

L	(horiz translation)
J	(vert back)
I	(horiz back)
N	(vert translation)
- & Cam focusing.
- b) Check BOTH vacuum gauges on the rotary pumps. If the vacuum is bad check all valves between pump and vessel, check pumps are switched on, check mylar windows are intact.

TABLE 3

Motor drive subroutines

PROGRAM SUBROUTINES LOADED ON COMMAND IRD PXLOAD :

KEYBD.CAT (MAIN PROG)

PANIC.CAT

CHKSTP.CAT

POSITN.CAT

PPRINT.CAT

MXDRV1.CAT

LOOKA.CAT

LOOKB.CAT

STEPS.CAT

PACKCN.REL

CFUNC.REL

FIGURE CAPTIONS

Fig.1 Station 7.2 general layout.

Fig.2 Portable terminal key allocations.

Fig.3 Ionisation chamber block diagram.

Fig.4 Photograph of workstation showing alignment carriage.

Fig.5 Monochromator assembly.

Fig.6 "Elephant's Ears" scatter guard fitted to Arndt-Wonacott camera.

Fig.7 Cooler block diagram.

Fig.8 Arndt-Wonacott camera on work station.

Fig.9 Wiring diagram of flying beam stop.

## REFERENCES

1. J.R. Helliwell, T.J. Greenhough, P.D. Carr, S.A. Rule, P.R. Moore, A.W. Thompson and J.S. Worgan, *J. Phys. E.* 15, (1982) 368-1372.
2. T.J. Greenhough and J.R. Helliwell, *JAC*, 15, (1982) 338-351.
3. T.J. Greenhough and J.R. Helliwell, *JAC*, 15, (1982) 493-508.
4. T.J. Greenhough, J.R. Helliwell and S.A. Rule, *JAC*, 16, (1983) 242-250.
5. T.J. Greenhough and J.R. Helliwell, *Progress in Biophysics & Molecular Biology*, 4, (1983) 67-123.
6. J.R. Helliwell and A.W. Thompson, *JAC Laboratory Note*, 16, (1983) 579.
7. T.J. Greenhough and P. Machin, *Daresbury Laboratory Internal Report*, DL/CSE/TM23, (1983)
8. T.J. Greenhough and P. Machin, *Daresbury Laboratory Internal Report*, DL/CSE/TM??, in preparation.
9. S.A. Kennerley, *Daresbury Laboratory Internal Report*, DL/CSE/TM24, (1983)

## TELEPHONE NUMBERS

J. Sheldon (Mechanical Services)	Bleep 173
D. Billing (Electrical Services)	Bleep 160
J. Helliwell (Station Master)	Bleep 217
J. Helliwell (Home Number)	0702 750802
Main Control Room + Crew	560,561,562

## REFERENCES

1. J.R. Helliwell, T.J. Greenhough, P.D. Carr, S.A. Rule, P.R. Moore, A.W. Thompson and J.S. Worgan, *J. Phys. E.* 15, (1982) 368-1372.
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4. T.J. Greenhough, J.R. Helliwell and S.A. Rule, *JAC*, 16, (1983) 242-250.
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- f) Should the N<sub>2</sub> cylinder run out, contact Jim Sheldon (bleep 173).
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- h) A helium cone for the Arndt-Wonacott camera is available.

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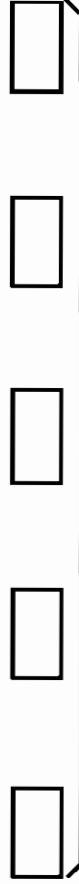
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N	(vert translation)
- & Cam focusing.
- b) Check BOTH vacuum gauges on the rotary pumps. If the vacuum is bad check all valves between pump and vessel, check pumps are switched on, check mylar windows are intact.



# DISPLAY



Do not use

Vert. bottom pre-slit  
 Vert. top pre-slit  
 Horiz. inner pre-slit  
 Horiz. outer pre-slit  
 Vert. top post-slit

Vert. bottom post-slit  
 Horiz. outer post-slit  
 Horiz. inner post-slit  
 Mono X-axis

B Mono Z-axis rotation  
 C Mono rotation  
 D Mono B<sub>x</sub>  
 E Mono B<sub>y</sub>

G Not used  
 H Horiz. front carriage  
 I Horiz. back carriage  
 J Vert. back carriage  
 K Vert. front carriage

L Horiz. translation carriage  
 M Horiz. rotation  
 N Vert. translation carriage  
 O Vert. rotation  
 P Not used

Q  
 R  
 S  
 T  
 U

Not used

V Abort programme  
 W Single/multiple step  
 X CLW/ACLW  
 Y Fast/slow  
 Z Panic

Fig. 2

in this limb (covering the beam pipe from the CVT valve/shutter to the two Be windows in the hutch) which is monitored on the ion gauge controller in the P.X. control rack.

One of the Be windows in the hutch separates high vacuum and atmosphere allowing the white, unreflected beam to enter the hutch (for alignment purposes). The other separates the high line vacuum from the rotary pump vacuum in the monochromator vessel.

Full instructions on pumping down or letting up the monochromator vessel are given on the pump mountings. NEITHER OPERATION SHOULD BE ATTEMPTED WITHOUT THE STATION MASTER. There are two separate rotary pumps in the P.X. hutch, one of which pumps the monochromator vessel and the other which pumps the beam pipe along the main arm. Since mylar suffers from radiation damage, windows on the beam pipe implode occasionally, but the separate pumping system prevents the monochromator vessel coming up to air at the same time. BEAM SHOULD NEVER BE LET ON TO THE MONOCHROMATOR UNLESS THE MONOCHROMATOR VESSEL IS FULLY PUMPED DOWN.

The monochromator vessel is isolated from the rotary pump by two valves, one just above the pump, and the other in the flexible vacuum pipe. These are normally open, and the vessel continually pumped.

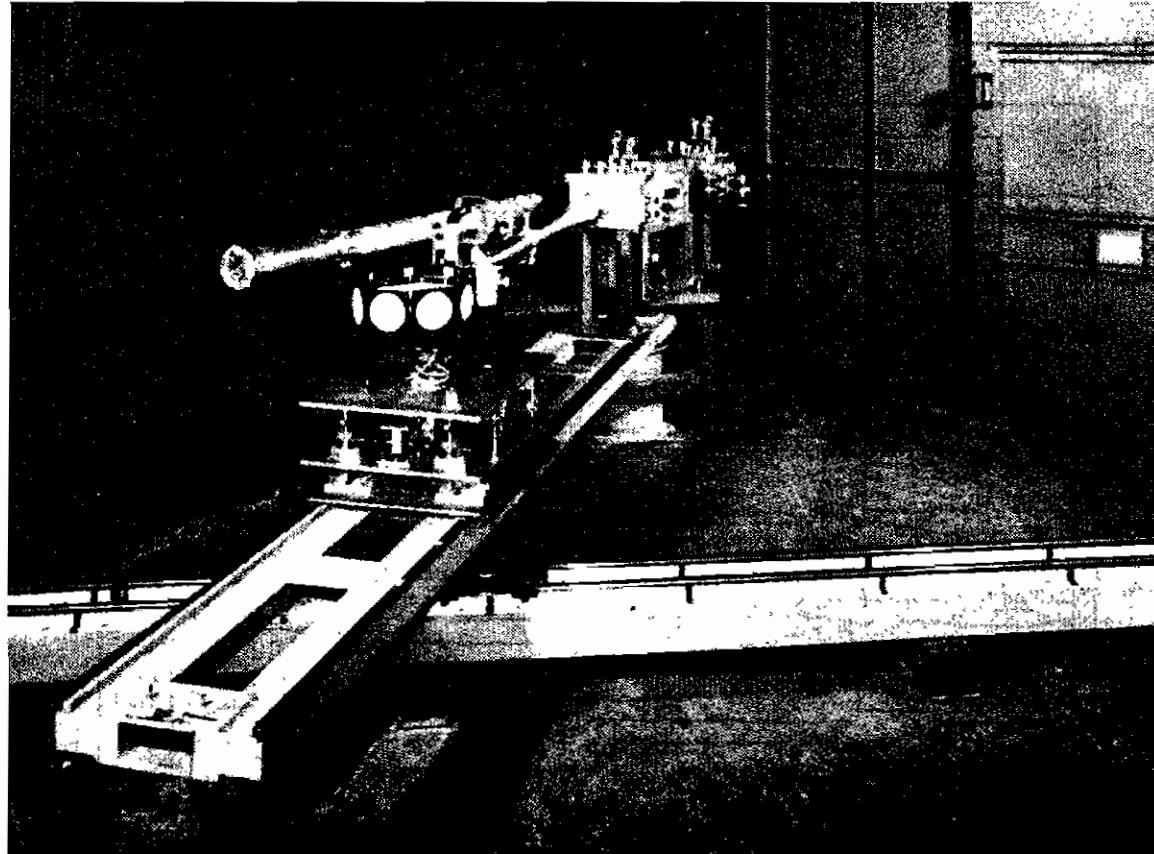
The pump on the beam pipe is isolated in similar fashion, and continually pumped. Should a mylar window fail, however, or the vacuum in the pipe reach a critical upper limit, a pressure switch causes the rotary pump to trip off. To pumpdown again, the pump 'ON' button must be held down (for >1 min) until an adequate vacuum is achieved in the beam pipe. There is no pressure trip on the monochromator vessel vacuum, and this vacuum should be checked daily before light is let onto the monochromator. If vacuum is poor, (i.e. not right on the lower limit of the gauge) check that BOTH isolating valves are FULLY open. If this does not improve the vacuum, then the station master should be called.

## 6. HUTCH INTERLOCKS

The hutch is interlocked to both the pair of shutters admitting beam into the hutch, and the main port shutter admitting beam to all stations on line 7. The former shutters are referred to as LOCAL SHUTTERS, and are under direct control of the experimenter. The main port shutter is controllable only by the crew in the Main Control Room - if it is tripped for any reason, the crew must be asked to reopen it.

Beam cannot be allowed into a hutch until the hutch has been searched (and found to be empty). The user is forced to look around the hutch whilst walking between push button switches that form the search points. The searcher is responsible for ensuring that the hutch is empty. A search is performed as follows:

- a) Take the key from the switch in the mask control module (situated in the P.X. control rack).
- b) Press the START SEARCH button on the grey search control box to the left of the main entrance to the hutch. The topmost (search started) lamp should light. Once this button has been pressed there remains approximately 40 seconds in which to search the hutch.
- c) Take the key to the first search point (directly behind the main arm on the far wall of the hutch), turn the key in the lock, press the button, turn the key back and remove the key. Note that the key is only removable in one position. Proceed to the second search point (in the left hand corner of the hutch).
- d) Press the button on the second search point.
- e) Press the button on the third search point (just inside the door of the hutch).
- f) Close the door GENTLY. Slamming the door often glitches the magnetic limit switches and drops the search.



THE ARNDT-WONACOTT CAMERA IS MOUNTED ON THE TOP PLATE OF THE ALIGNMENT CARRIAGE ASSEMBLY. TWO VERTICAL JACKS (FRONT AND BACK) DRIVEN BY STEPPER MOTORS RAISE AND LOWER (OR TILT) THE CAMERA. SIMILARLY, TWO SLIDERS PERFORM THE SAME FUNCTIONS IN THE HORIZONTAL. IN THE BACKGROUND, THE STEPPER MOTOR AMPLIFIERS CAN BE SEEN. THESE ARE CONNECTED TO THE CAMAC MULTIPLEXER (VIA THE HIM UNITS ON THE TOP OF THE RACK) BY TWO 15 WAY CANNON CABLES THROUGH THE 'LETTER BOX' CABLE EXIT. THE ENTIRE RACK IS POWERED FROM THE BLUE MARECHAL 3 PHASE SOCKET ON THE HUTCH HALL

Fig.4

cassettes (except the one exposing) from low energy scatter (see photograph in fig.6).

### 3. THE COMPUTER

#### 3.1 General description

The LSI/11 used on the station is situated on the far right of the CAMAC crate in the computer rack. Also mounted in the rack are (below the crate) the dual floppy disk drive, and (at the bottom of the rack) the computer and CAMAC crate power supply. The computer derives its operating system from the SY: or system device, in our case a floppy disc in drive 0 (the right hand drive). The programmes the computer runs are taken from the left hand drive or DK: device.

In order to initialise (boot) the system, the correct floppy discs must first be placed in their drives. The P.X. Catex System disc is placed in drive 0 (r.h. drive), and the P.X. stepper motor drive disc in drive 1 (l.h. drive). The doors of the drives must be closed before the disc drive can read a disc.

On the LSI/11 there is a 3 position switch, the upper position of which is labelled HALT, the centre RUN and the lower INIT. To boot the computer (assuming the CAMAC crate power is on) the switch is moved from RUN to INIT, and returned to RUN (The switch is of the pull and move type).

The computer will reply:

```
RT11FB (S) V04.00
etc
```

The dot is the prompt of the RT11 operating system. If the computer does not get this far, the following action should be taken:

a) Check the system disc is in the disc drive (0) with the door closed and the ready light up. Check the disc is in the right way round i.e. with the exposed rectangle of the magnetic surface parallel to the top edge of the drive.

b) Check the unit in CAMAC slot 5, (the floppy disc interface) which has two front panel connectors which occasionally pull out or get dirty.

c) Check that the terminal is connected to the LSI and switched on.

d) Check that the terminal 'NO SCROLL' button has not been pressed, by switching the terminal on and off.

e) Switch the LSI power supply off and on (bottom most in the computer rack).

f) Call in the crew/shift scientist.

If all else fails, contact a suitable member of the C.S.E. section through the Main Control Room. Most problems should be user solvable, however.

Once the dot prompt appears, the programme may be initiated by:

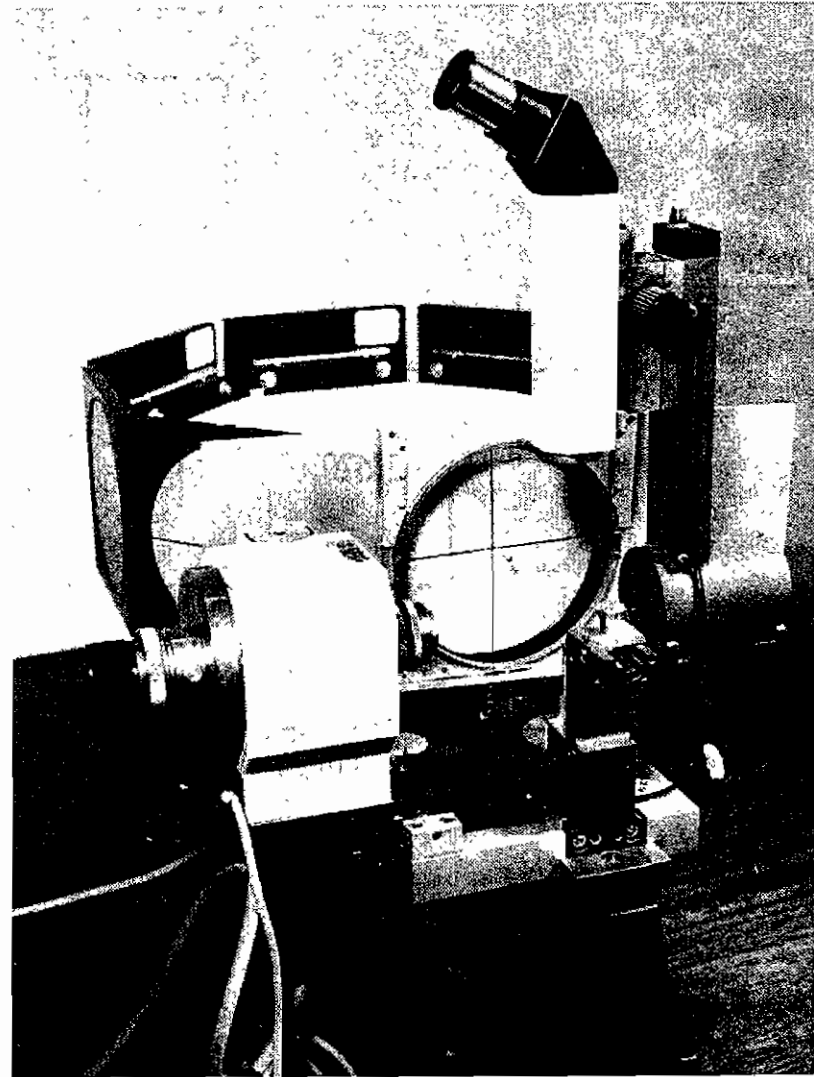
a) Type in CAT(carriage return)  
The computer will respond with :  
+CATEX VERSION(2) etc .....  
+

b) Type in IRD PXLOAD(cr)  
The stepper motor program will now load and run.  
The programme asks for a reference set of motor positions on starting

(1) is input to set all motor references to 10000 (note that this does not actually return any motor, but is an arbitrary number to which steps are added or subtracted as motors are moved), and

(2) input to read the last set of positions dumped to disc. Since these are nearly always out of date it is best to initialise to 10000.

The programme bases all motor positions on these numbers, and the current motor positions can be read out by typing in '?'. The gearing of the top carriage transitions is such that the front motor moves in steps of 5 micron, and the rear is 2.5 micron. The vertical translation button on the



THE PHOTOGRAPH ILLUSTRATES THE 'ELEPHANT'S EARS' SCATTER GUARD ON THE ARNDT WONACOTT CAMERA. THE DEVICE WILL ONLY CUT OUT LOW ENERGY MULTIPLE SCATTERING, AND IF THE SCATTER PROBLEM IS BAD WILL PROBABLY NOT BE OF MUCH USE.

Fig.6

It must be clear that the keypad works in an on/off mode. If a button is pressed to start a motor, that motor will continue to drive until the same button is pressed again (i.e. the first operation of a button starts the motor, the second stops it). Operation of a different motor button while one motor is already driving will result in the PANIC function being called. All motors will stop, and the PANIC stop must be re-enabled in order to drive further motors. The only exception to this on/off type operation is the single step option key. When this is pressed, the single step motion is selected and any motor button pressed results in a single step operation of that motor. The single step button should be operated again in order to return to multiple step mode.

### 2.3 Ionisation chambers

The x-ray beam is monitored during the experiment by means of a miniature ionisation chamber, made up of an ebony horseshoe with pieces of copper clad circuit board forming the plates (see fig.3). The chamber is powered by an HT supply (shortly to be replaced by a smaller supply), at a potential of -100 V. The signal received on the second plate is collected by a Keithley 427 current amplifier, with gain normally set at  $10^9$  (i.e. for a 1 volt output, 1 nanoamp of ionisation current has been detected). Table 1 gives typical voltage readings and scale factors for machine energy for different wavelengths through a 0.3 mm collimator (the 1 . blue). Up to 10% error in these readings is acceptable - the alignment of the station depending on systematic errors such as the orthogonality of the beam and the mirror, any small change in beam orbit or steering etc.

This voltage measurement is fed into a voltage to frequency converter, and sampled by a CAMAC scaler. This gives an indication of exposure in terms of v/f counts (The data acquisition program using this method of determining exposures is documented elsewhere). The v/f converter operates between 0 and 10 V, as does the Keithley. Any attempt to read voltages/currents beyond the range of these instruments will result in damage and delay. Please watch your ion chamber reading and turn down the gain if the Keithley reads more than 10 volts.

### 2.4 Pre-monochromator slit setting

The slits are (in normal operation) adjusted to keep the beam off the mount and tip of the triangular monochromator, for reasons of heating and

radiation damage (to the mount). This is achieved by adjusting the slits until the flux reading on the ion chamber just starts to decrease, the slits being best moved on the FAST speed range (Don't forget to change to speed slow afterwards!). The slits marked HORIZONTAL refer to those whose edges are vertical, in other words the slits that control the HORIZONTAL aperture. Thus the slits required to protect the two ends of the monochromator are horizontal inner and horizontal outer.

For crystals with a large unit cell dimension, further tilting down reduces the beam cross fire angle, and hence improves order to order resolution. A reduced monochromator acceptance is also useful in eliminating the often observed monochromator warm up time. Attenuations of (respectively) 50% and 25% would be typical for these applications. However, longer exposure times (pro rata) must be expected.

The vertical slits may be adjusted in similar fashion, but these are not used to control the SR primary beam since the vertical source size and divergence (0.3 mm, 0.25 mRad) is acceptable. A second set of vertical slits exist before the focusing mirror.

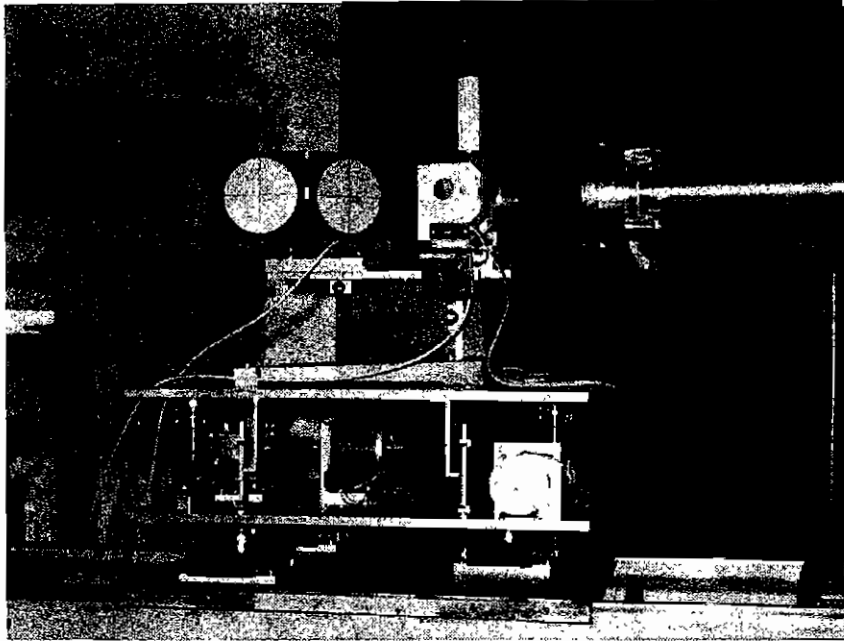
It is MOST IMPORTANT to record any movement of slits. Other users can not be expected to know how you have set the station up! Failure to note this will lead to loss of time for the group to follow. The information which must be recorded to maintain the smooth running of the station is given in Table 2.

### 2.5 Top carriage alignment

The Arndt-Wonacott camera fixes on to a 'top carriage' assembly which allows both vertical and horizontal translations and tilts for the alignment of the camera to the beam. The stepper motor key pad provides buttons for horizontal translation (two horizontal motors driven together), vertical translation (two vertical motors together), and the separate motions (vertical front, vertical back, horizontal front, horizontal back).

A typical alignment procedure (starting from scratch) would be as follows:

- a) Using plastic scintillator (yellow card) find the beam with respect to the collimator (mount to be roughly central to the beam).
- b) Fit the small circular screen to the front of the 4 . red collimator.



CLEARLY VISIBLE IN THE PHOTOGRAPH ARE:

- A) THE COOLING DEVICE.
- B) THE FLYING BEAM STOP (MK. 1 VERSION)
- C) THE IONISATION CHAMBER AND ITS CABLES
- D) THE LEAD SHIELD AND BEAM TUBE
- E) THE INGRESS PIPES AND PRESSURE GAUGE (ON THE FAR WALL OF THE HUTCH) FOR THE COOLER DRY AIR SUPPLY.
- F) THE NUMBERING ON CASSETTES, CASSETTES HAVE NOT BEEN ASSIGNED A NUMBERED POSITION ON THE CAROUSEL.

Fig.8

ABSTRACT

This document gives a description of the routine operation of the P.X. workstation written with the station user in mind. It is only concerned with the practicalities of operation, and contains no detailed wiring or program information. The scanning and film processing facilities provided by Daresbury are also discussed.

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# technical memorandum

# Daresbury Laboratory

DL/SCI/TM35E

PROTEIN CRYSTALLOGRAPHY 7.2: WORKSTATION OPERATION

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