PRISMA

Single Crystal Cold Neutron Spectrometer & Diffractometer

User Manual

SECOND EDITION

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ISIS

CONTENTS

Preface to the First Edition

This manual is intended as an easy-reference guide to help you perform an experiment on PRISMA and analyse the results. In this sense it does not attempt to be an exhaustive guide, and more detailed information may be found in the various publications and reports listed in the Introduction and in the more specific manuals which describe the operation and analysis programs. Copies of all of these are in the PRISMA cabin. The additional manuals are:

- The PRISMA Operating Program Manual
- The PRSCAL Manual
- The PRISMA GENIE Data Analysis Manual
- The PRSPLOT Manual
- The PUNCH Manual
- Various RAL reports describing cryostat operation

Preface to the Second Edition

PRISMA has undergone a complete rebuild since the First Edition of this manual, resulting in a rather different instrument to that of January 1995. Now that the development of the instrument has reached a quiescent stage, this new edition of the manual describes the operation of PRISMA for the foreseeable future.

Beginning an Experiment at ISIS

Before starting your experiment you must:

- report to the Main Control Room (MCR). You will be issued with a swipe card, which will let you in and out of the Experimental Hall (R55).
- pick up a film badge from the Health Physics Office opposite the MCR.
- watch the safety video either in the coffee room adjacent to the DAC, or in the coffee room on the top floor of R3; sign a card to register that you have done so.
- get the sample record sheet from the DAC and make sure that you understand the sample handling instructions
- acquaint yourself with the operation of the safety interlock system. Your local contact will explain this to you.

1. Introduction

PRISMA (=**PR**ogetto dell'**I**stituto di **S**trutura della **MA**teria del CNR) is the result of a collaboration between the Italian Consiglio Nazionale delle Ricerche (CNR) and the British Engineering and Physical Sciences Research Council (EPSRC). A brief introduction to how PRISMA works is given here, but detailed accounts may be found in the following publications, which are available from your local contact:

"The development of the PRISMA spectrometer at ISIS", Steigenberger *et al.* (1991) *Nuclear Instruments and Methods in Physics Research* **B53**, 87-96;

"The PRISMA Spectrometer at ISIS", Steigenberger *et al.* (1990) *Rutherford Report* RAL-90-004.

"A novel double-crystal analyser system for the PRISMA spectrometer at ISIS", Bull *et al.* (1997) *Physica* **B234-236**, 1061-1063;

The resolution characteristics of PRISMA are discussed in

"New considerations on a multi-analyser spectrometer resolution function", Hagen and Steigenberger (1992) *Nuclear Instruments and Methods in Physics Research* **B72**, 239-254.

In addition, there is a great deal of information about the instrument and HTML versions of the manuals on the World Wide Web at:

```
http://www.isis.rl.ac.uk/excitations/prisma/prisma.htm
```

PRISMA has a dual role as both a neutron diffractometer and spectrometer. In diffraction mode, it is identical in principle to the other single crystal diffractometer at ISIS, SXD. This principle is described on the following page. When operated as a spectrometer, it is perhaps understood best by comparison with a conventional triple-axis spectrometer. Both machines use Bragg reflection from analyser crystals to select the neutrons scattered by the sample that will ultimately be detected. However, unlike a triple-axis spectrometer, PRISMA has an array of independent analyser-detector arms instead of just one. Also, instead of having a monochromator crystal to define the incident neutron energy, PRISMA utilises the time-of-flight technique. This is the natural way of defining neutron energies at a spallation source such as ISIS, since it produces a pulse of polychromatic neutrons every 20 milliseconds which is roughly 30 microseconds wide. The spallation technique produces very high energy neutrons, and these must first of all be moderated to thermal energies before they can be of any use. This is accomplished by forcing the neutrons to pass through a dense hydrogenous material (usually liquid methane at a

temperature of about 100 K for PRISMA) before reaching the instrument. Since thermal neutrons travel at relatively slow velocities (for instance, a thermal neutron with an energy of 10 meV has a speed of about 1400 ms^{-1}), their energies may be realistically measured from their flight times between the moderator and instrument.

As mentioned above, PRISMA can also be configured as a high-resolution, low-background diffractometer. This is achieved simply by removing the analyser crystals. In fact, by using the single analyser detector module (known locally as PRISMA-2), it is possible to perform inelastic and diffraction runs simultaneously. A schematic view of the operation of this module is shown in Fig. 1, with one detector in the module set up for inelastic scattering and the other for diffraction. The left-most analyser-detector arm in the array is always referred to with the index 1, and the scattering angle of the whole array, ϕ, is measured with respect to this arm. Also, each analyser-detector arm may be considered to have its own individual value of ϕ, and since the separation of each arm from its neighbours in this module is 2° in ϕ, then if $\phi_1=-90°$, then $\phi_{16}=-60°$, for instance.
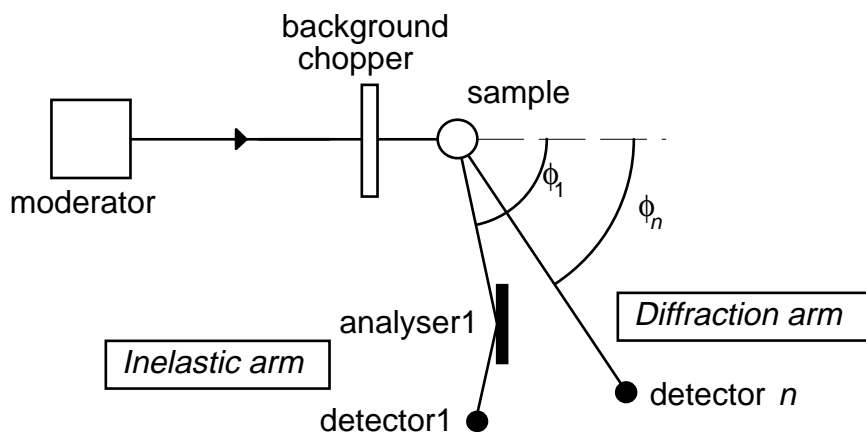
Fig. 1. A schematic plot of PRISMA showing the setup for simultaneous inelastic and diffraction runs.

In inelastic mode, the neutron counts are measured as a function of the time-of-flight, so that the signal in each detector corresponds to a scan along a parabolic **Q**,E path through $S(\mathbf{Q},E)$, the dynamic structure factor. Since there may be a total of 16 analyser-detector systems in all, each of which measures along a parabolic path, PRISMA allows for a two-dimensional slice through (**Q**,E) space for each ISIS pulse, in a single setting of the instrument and sample.

In the case of a diffraction measurement, each detector integrates over all energies of the scattered neutrons, so that the result is a radial elastic scan in reciprocal space. The vector diagram Fig. 2 shows this for a single detector.
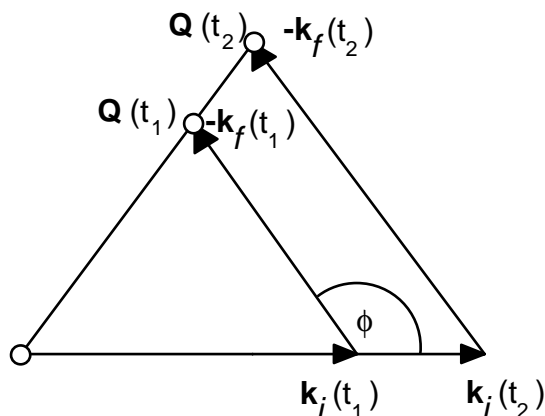
Fig. 2. A diagram showing the scattering vectors for
two times-of-flight t_1 and t_2 in a diffraction
experiment using a single detector. **Q** is the
wavevector transfer, $\mathbf{k}_i$ and $\mathbf{k}_f$ are the incident and
final wavevectors, respectively, and ϕ is the
scattering angle, i.e. 2θ

PRISMA has a number of detector modules that are used for different types of
experiments. These modules are frequently referred to locally as "PRISMA-n", where
$n = 2,3,$ or 4. They are detailed in Table 1.

Detector module	Local name	Number of analyser - detector systems	Range in energy transfer (meV)	Horizontal collimation after sample (minutes)
General-purpose diffraction/inelastic	PRISMA-2	16	0-100	60
High-resolution inelastic	PRISMA-3	5	0-40	30 or 60
Diffraction	PRISMA-4	16	N.A.	10 or 30

Table 1
Characteristics of the three detector modules.

The sections that follow give a basic outline of the layout of the spectrometer "on the
ground" and (perhaps more importantly for the user) the different functions of the various
terminals and computers in the PRISMA cabin. This is because unlike most ISIS
instruments (but more in common with a conventional triple-axis spectrometer),
PRISMA has a significant number of independently moving parts, and hence operation is
a somewhat involved process which requires the use of a purpose-written program (called
PRISMA), as well as the usual commands to the DAE and CAMAC.

1.1. The PRISMA cabin

The PRISMA cabin contains a number of terminals and a PC that are used for different (and specific) jobs. The layout is shown in the diagram below. You have a choice of two terminals on which to run the instrument: if you require hardcopy output during your experiment, then ask your local contact to set up the hardcopy LA120 terminal. If not, then you will be using a window on the PRISMA workstation. A word of warning is appropriate: the PC to the left of the LA120 hard-copy terminal should NEVER be touched, and you should NEVER exit from the PRISMA control program, whether it is running on the LA120 or a window on the PRISMA workstation. These two devices control the whole instrument, and should not be tampered with. Occasionally it may be necessary to restart the control program if the workstation has crashed. This is simply achieved by logging the workstation back on using the account name "PRISMA" (the password is obtainable from your local contact), and then running the control program by typing

```
$ PRISMA
```

in a DECterm. However, this will hopefully not be necessary during the course of your experiment, and, unless a scan or job file is running, the control window will always show the prompt

```
PRS>
```

indicating that PRISMA is idle and is waiting for a command.
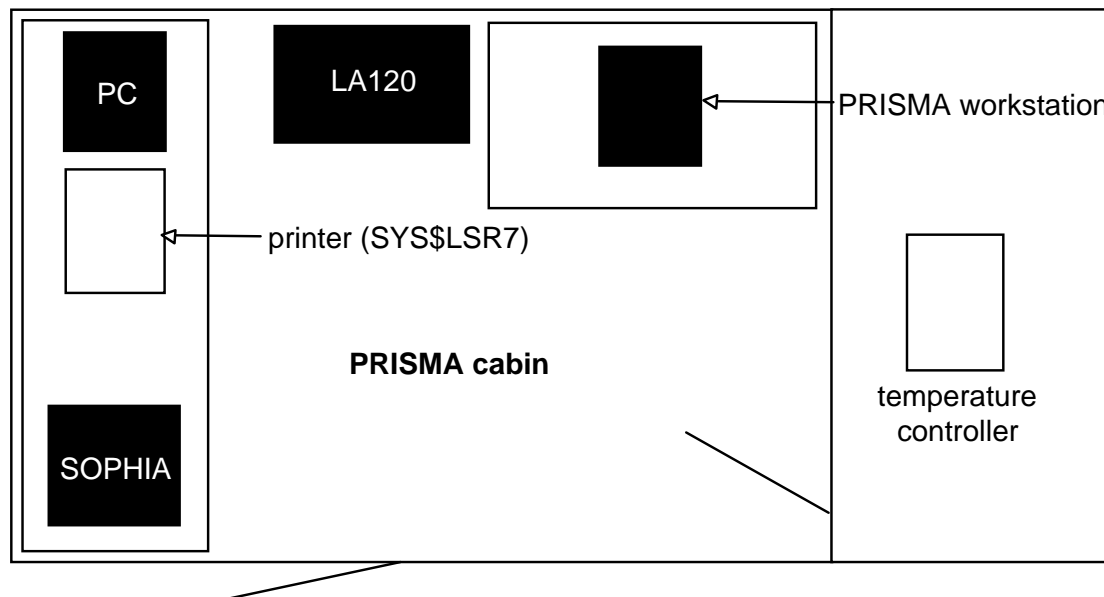
Fig. 3. The PRISMA cabin.

1.2. The PRISMA Dashboard

Before going on to describe the hardware of the spectrometer itself, there is one more feature of the control operations which should be mentioned, and that is the instrument dashboard. This usually runs in the top left-hand window of the PRISMA workstation, and displays most of the current instrument and sample environment parameters. We shall refer to this as the *supervisor* window from now on. Note, that while the dashboard is running in this window, it should only be used for certain commands. The dashboard may be turned off by typing

```
prisma/prisma: stat off
```

and turned on again by typing

```
prisma/prisma: stat on
```

in the window.

We shall now describe the most important parameters displayed in the dashboard, which are indicated in this diagram:
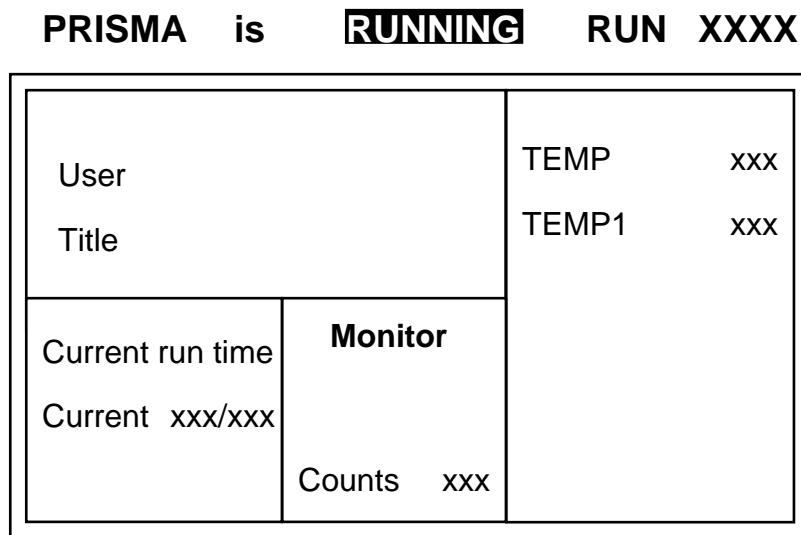
Fig. 4. The PRISMA dashboard, always displayed in
a window on the PRISMA workstation in the
Control area of the desktop.

In the top left-hand box of the dashboard are shown:

- the title of the current (or most recent) run,
- the users' initials,
- useful telephone numbers

The run title and user information may be changed with the **change** command, which is described at the start of Section 3.

In the lower left-hand box of the dashboard are shown:

- the duration of the current run in days, hours, minutes, and seconds,
- the ISIS proton current (in µA), and the total number of protons since the run started (in µA.hours)

The box to the right of this shows:

- the total counts measured in Monitor 1 (before the sample position)

In the right-hand most box are shown the following CAMAC parameters

- the value of the set-block TEMP, usually the controlling sensor
- the value of TEMP1, usually the temperature closest to the sample
- the value of LAKES if a Lakeshore temperature controller is being used

1.3. The Layout of the Spectrometer

The schematic diagram below shows how the detector modules and sample environment equipment are located in the PRISMA sample area with respect to the incident beam.
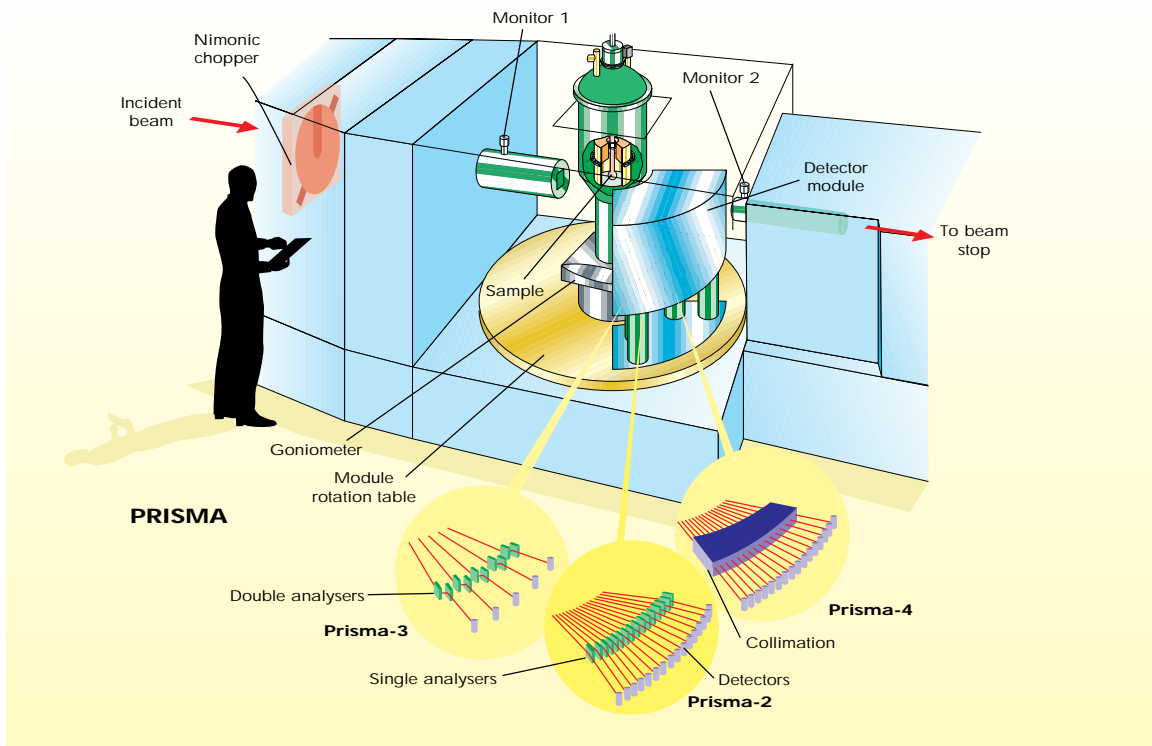
2. Installing a Sample on PRISMA

The vast majority of experiments performed on PRISMA use single crystal samples, but since crystals never come in standard sizes or shapes, an individual mount must always be made up for each sample. This can be a time-consuming process, and it is always best to do this well before the start of the experiment to minimise time spent in setting up. It is also recommended that the sample be well aligned in advance on its mount (usually with x-rays), because it can sometimes be a difficult job to do entirely from scratch on the spectrometer.

If the experiment is to be performed at room temperature, or in a furnace, the mount should end in a screw with an M8 thread. This is standard for all ISIS instruments. The sample should be supported by the mount on this thread, and the whole mount should not be more than about 10 cm long in total (including the thread).

2.1. Room Temperature Experiments

If the sample is not to be heated, then the best material to use for making a mount is aluminium. It is preferable to use a small strap of aluminium around the crystal, which is attached to an M8 screw thread. You should use as little aluminium as possible in the strap to avoid spurious scattering, and the rest of the mount should be shielded from the beam by wrapping cadmium sheet around it. The M8 thread on the mount is screwed into a PRISMA "candlestick", which is then attached to the large goniometer inside the instrument. This procedure must be carried out by your local contact, do not attempt to change samples yourself. To ensure that the sample will be fully in the beam, the height should be adjusted so that it is about 13 cm from the top surface of the candlestick.

2.2. Low Temperature Experiments Using the CCR

The majority of PRISMA experiments use the CCR since it should allow for temperatures between 10 and 300 K, and hence there are a number of aluminium sample cans available which fit directly onto the cold head of the PRISMA CCR. The sample is actually mounted inside the can on an aluminium rod, which should be cut so that the distance from the base of the sample can to the centre of the crystal is 48 mm, as shown in the diagram just overleaf. This ensures that the crystal sits at the correct height for the centre of the beam. The sample end of the rod should be filed to shape to ensure a good contact with the crystal. The crystal is then glued in place using Kwikfill, but it is important to reduce spurious scattering from the glue and the aluminium rod with a cadmium or gadolinium shield inside the can.

To ensure a good thermal contact from the sample to the cold head, a sample can should always be filled with helium exchange gas and sealed with a fresh piece of indium wire. Your local contact will show you how to prepare a sample in this way. Once the can is ready, it can simply be screwed directly onto the cold head of the CCR and the CCR

loaded into the instrument. This requires the Pelloby crane on the beamline, and must normally be done by the local contact unless you have a valid crane driver's licence!
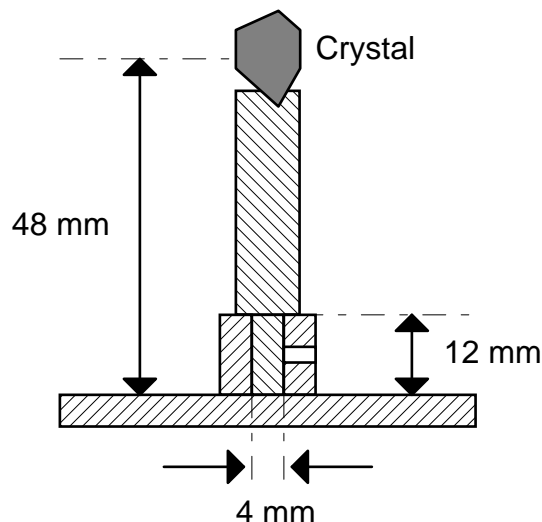
Fig. 5. A side-view of a crystal mounted for a CCR experiment.

2.3. Orange Cryostat Experiments

PRISMA has its own orange cryostat, with an internal bore of 50 mm. This means that samples larger than 50 mm in diameter will not fit inside it. The sample is mounted on the end of the cryostat centre-stick using a mounting arrangement similar to room temperature experiments (i.e. an aluminium strap attached to an M8 thread). Brief information is given below concerning the valve settings and flow rates. It is important to remember that both the warm and cold valves should only be finger tight. Over tightening them will cause damage. Much more detailed information is given in the RAL reports 93-006 and 92-041, copies of which are kept in the PRISMA cabin.

Cooling to >4K	Open the cold valve 1/2 turn. Open the warm valve until the flow observed on the gas recovery flow meter is 10L/min
Constant temperature >4K	Once the required temperature has been reached reduce the flow to 4L/min using the warm valve, and the temperature will be controlled by the Eurotherm and the cryostat heater, or, if you want the temperature to remain stable at 4K, switch the heater off.
Cooling to <4K	Close the warm valve, and open the cold valve 1/2 turn. Slowly open the Roots pump valve, never letting the pressure rise to above 10 torr. When the pump valve is fully open use the cold valve to set the flow to 0.5 to 1 L/min.

2.4. Furnace Experiments

When the sample is to be heated above room temperature (but not above about 700 K), then aluminium may still be used to make the sample mount. For temperatures above about 700 K, tantalum foil is generally suitable. It is still important to use as little material as possible around the crystal, so as to eliminate spurious scattering, and the rest of the mount should be shielded from the beam, as in a room temperature experiment. However, UNDER NO CIRCUMSTANCES should cadmium be used for shielding the mount when heating above room temperature, since it may melt or oxidise. Gadolinium foil is available for this purpose instead.

There is a choice of two furnaces for PRISMA experiments: the dedicated PRISMA furnace, or a RAL furnace. The disadvantage of the PRISMA furnace is that it cannot be used for temperatures above about 800 K, but it can hold a relatively large sample (up to about 60 mm in diameter). On the other hand, a RAL furnace will go up to about 1300 K, but is more restrictive, so that the sample must be 35 mm or smaller in diameter.

The sample mount is screwed directly into the centre stick of the furnace (both PRISMA and RAL types). The sample sensor should then be bent so that it is as close as possible to the sample without being directly at the beam height. As with the CCR and orange cryostat, it is assumed that the furnace has already been installed and set up by the local contact.

3. Setting Up an Experiment

First of all, the information displayed on the dashboard should be changed for your experiment and initials, since this is stored with the data you will collect. The **change** command allows you to edit the dashboard information and to modify the *icp* (*instrument control parameters*). In the supervisor window on the workstation (the one with the dashboard), type the command

`prisma/prisma:` **change**   (can be abbreviated to **cha**)

which initiates the dashboard editor. Move between areas using the cursor keys and over type or toggle as instructed. The first page contains title and user information, and only this should be changed. To exit press the "**[PF1(GOLD)]**" key, then an "**e**". The **PF1** key is on the keypad to the right of the keyboard. Note, that if you only want to change the run title in the dashboard, you can do this from the PRISMA control program by typing

`PRS>` **TI** *<your title>*

It is a good idea to include all details about temperature, type of scan, etc in the title. For example

`PRS>` **TI CsNiCrF6 at 9 K,phi1=-53,om=-10,ef1-8=18,d9-16=0.0**

If you intend to set a command file running to change scans or temperature automatically the experimental run title can be written directly to the dashboard by using the **TI** command in the command file. This is detailed in section 4.3.

3.1. PRISMA-2 Single analyser detector module

The PRISMA-2 detector module has 16 detector tubes, and is usually configured so that the first 12 have analyser crystals in front of them for inelastic scattering, while the last 4 have no analyser crystals, and so are used for diffraction. The 16 tubes cover a range in the ϕ angle of 30°, so if ϕ_1=-90, then ϕ_{16}=-60.

The operation of aligning a single crystal using PRISMA-2 is now described. Alignment of the sample is usually the most demanding part of a PRISMA experiment, which is why it is always best to have a prior alignment of your crystal in its mount before starting the experiment. Currently, an x-ray Laue camera and the ALF neutron alignment facility are periodically available in the Laboratory for this purpose, but if x-ray facilities exist at your home institute, you should use these.

To illustrate the alignment procedure, we will describe a single crystal alignment for a crystal of trigonal $NaNO_3$, where the scattering plane contains the two orthogonal symmetry directions **a*** and **c***. This procedure may easily be adapted for any other crystal by following the steps given here.

First of all, place your sample in the goniometer so that the **c*** direction is along the GY arc, as shown in the next diagram.
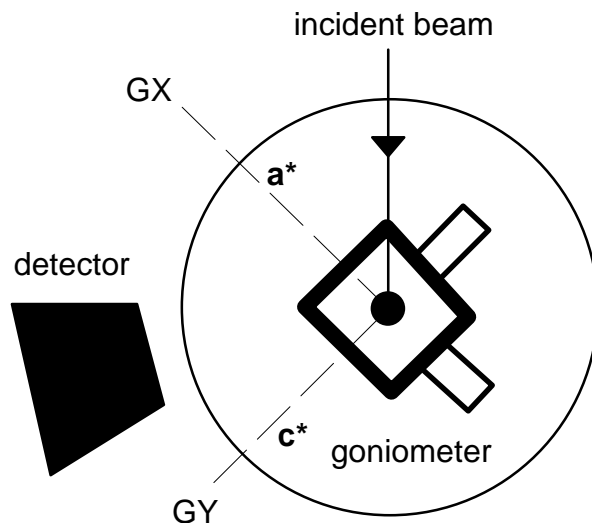
Fig. 6. A schematic view of PRISMA from above, showing the goniometer with the GX and GY arcs, and the sample orientation discussed in the text.

You are now ready to align the crystal on the instrument. Follow these steps, typing all of the relevant commands into the PRISMA control program running either on the workstation or the LA120:

(i) Open the shutter

(ii) Change the title on the dashboard by typing something like this

PRS> **TI ALIGNMENT OF NANO3 CRYSTAL AT RT**

(iii) Type in the lattice parameters, like so

PRS> **SE A=5.07 5.07 16.82 120 90 90**

These are the lattice parameters, in the order a, b, c, γ, α, β. The scattering plane is set like this:

PRS> **SE PX(1)=1 0 0 0 0 1**

which tells the spectrometer that the [1 0 0] reciprocal lattice vector (i.e. **a***) is along the GX arc, and that [0 0 1], i.e. **c***, is along GY. The convention is, of course, to use right-handed axes, so that if you were looking at a cubic crystal with the usual scattering plane containing the [1 1 0], [1 1 1] and [0 0 1] directions and the [1 -1 0] vertical, you would type

```
PRS> SE PX(1)=1 1 0 0 0 1
```

(iv) Change the time-channel boundaries by typing

```
PRS> SE START=1000 12000 10
PRS> SE MNT=1 MNS=1000
```

(v) Drive the arcs to zero if they are not already zeroed by typing

```
PRS> DR GX=0.0
PRS> DR GY=0.0
```

(vi) We now assume that the detector has been set up by your local contact in the standard way, with detectors 13 to 16 used for diffraction. Drive ϕ_{13} to -90° by typing

```
PRS> DR PHI13=-90
```

(vii) You are now ready to begin an alignment scan. You should start with

```
PRS> SC OM13-16=-46.5,0.1,NP=11
```

This command tells the spectrometer to measure the signal in detectors 13 to 16 while only Ω is scanned, where the crystal moves by 1° in total around Ω=-46.5, by an 11-point scan with a step size of 0.1°. Since we are using 4 detectors, the net Ω angle covered is 4° in this one scan. At each point in the scan, a printout of the integrated counts in each detector is given in the control program. When a large signal is seen in any of them (typically over 1000), then a Bragg peak is going into that detector. You can look at the intensity in each detector as a function of the sample d-spacing by using the program **PP** in GENIE. Follow these steps for using **PP**:

(a) Run GENIE in a window on the workstation, and type

```
>> pp
```

(b) Respond to the first question with the number of detectors being scanned, in our case it is **4**.

(c) Type in the absolute ϕ value for the first detector in the array, i.e. **90**.

(d) Put in the limits for display (in Angstroms) as something like **0.5** and **3.5**

(e) Input the bin size, usually **0.005**

(f) For the next question about the run number, answer "**dae**" if you want to look at a scan which is still running, or else type in the run number.

(g) Now the program asks for the detector spectrum and the point in the scan to be displayed. After this it will plot the spectrum of your choice.

(viii) If you cannot see any signal in the scan from the peaks corresponding to the **a*** direction, you should scan more widely in Ω with one of the following scans:

PRS> **SC OM13-16=-50.5,0.1,NP=11**

or

PRS> **SC OM13-16=-42.5,0.1,NP=11**

In this way you can cover a range of 4° in Ω with each scan. It may well be that you have to perform several of these scans before you find a Bragg peak that you can identify. For each successive scan you should increment (or decrement) the Ω value that you type in by 4°, since each scan covers a 4° range in Ω. However, sometimes the tilt of the crystal is misaligned by a few degrees out of the scattering plane such that the required fundamental Bragg peaks cannot be seen at all. If you have performed several Ω scans and still cannot see any recognisable Bragg peaks, then this is probably what has happened, and you should try scanning around the **c*** position instead, which may not be so badly misaligned in tilt. In this case try

PRS> **SC OM13-16=43.5,0.1,NP=11**

and then around Ω=39.5°, 47.5°, and so on.

The following steps assume that you have found **a***, but will work just as well if you have found **c*** instead, by substituting the appropriate Ω values. Remember that for a detector at ϕ=-90°, the **a*** position should be Ω=-45°, while the **c*** position should be Ω=45°.

(ix) When you have found the rough position of the **a*** direction by using the multidetector array, it is easiest to then complete the alignment by bringing around the **a*** reflections into detector 13 at ϕ=-90°. First we need to work out the value of Ω to bring **a*** into the new value of ϕ_{13}=-90°. If in the multidetector scan you had found the strongest signal for **a*** in detector 15 at Ω=-37.4°, say, then the following formula gives the value of Ω that you need as:

$$\Omega_{\phi=-90} = \Omega_{\phi} + \frac{1}{2}\left(90 - |\phi|\right)$$

where in our case Ω_{ϕ}=-37.4°. However, you need to know the value of the ϕ angle for detector 15. This may be obtained by the command

PRS> **PR ANGL**

and a complete list of all of the current spectrometer angles will be printed. For our example, we find that "`PHI15=-86`", which means that our new value of Ω is -35.4° using the above formula.

(x) You should now perform a fine scan around the new Ω value as a check:

PRS> **SC OM13=-29.4,0.2,NP=5**

You can see the data measured by the scan as a function of d-spacing by using **DD** in `GENIE`, which is similar to **PP** but is for a single detector scan.

(xi) Drive the Ω angle to the maximum of this scan, and scan around the tilt angle `GX`, by typing

PRS> **SC GX13=0.0,1.0,NP=9**

You may wish to do a finer scan after this to define the maximum in `GX` more precisely. You should then drive the `GX` angle to the peak by typing

PRS> **DR GX=**⟨*peak in GX*⟩

(xii) Now we are ready to set the zero of the Ω angle. Usually, we define Ω=-45° when the **a*** direction bisects the angle between the incoming beam and the detector at ϕ=-90°. This is the situation shown in the previous diagram, and is the alignment that we have just obtained. Print out the "zero angles" by typing

PRS> **PR ZERO**

We want to adjust the value of the parameter `ZOM` so that the Ω setting becomes -45°. We use the following equation:

$$\text{ZOM}_{\text{new}} = \text{ZOM}_{\text{old}} + \Omega + 45$$

Where ZOM_{old} is given on the printout, and Ω is the current angle. Reset `ZOM` by typing

PRS> **SE ZOM=**⟨*new value*⟩

The computer should reply by typing the value of `ZOM`, and that "`OM=-45.00`", or a number very close to it.

Note, in the general case, when you want to reset the Ω value from Ω_{old} to Ω_{new}, the formula you should use is

$$\text{ZOM}_{\text{new}} = \text{ZOM}_{\text{old}} + \Omega_{\text{old}} - \Omega_{\text{new}}$$

(xiii) We are now ready to align the **c*** arc. Drive Ω to 45° and do a GY scan by typing the following:

```
PRS> DR OM=45
PRS> SC GY13=0.0,1.0,NP=9
```

After you have optimised GY, the crystal is aligned and you are ready to begin collecting data, which is described in section 4.

3.2. PRISMA-3 Double analyser detector module

The PRISMA-3 detector module has 5 detector tubes, and is always configured so that each tube is behind a set of double-analysers. The detectors are separated by 10° in ϕ so if $\phi_1 = -90$, then $\phi_5 = -50$.

The operation of aligning a single crystal using PRISMA-3 is almost identical to that of PRISMA-2. The principal difference is that a rough alignment us usually performed using some diffraction tubes fixed to the side wall of the instrument. A strong reflection is then brought round into the double-analyser module for a careful alignment.

Begin by scanning for Bragg peaks in the fixed detectors. These will be set up to be detectors 6-10, while the PRISMA-3 detectors will be 1-5. For example,

```
PRS> SC OM6-10=-45,1,NP=11
```

When you have found a strong Bragg peak, maximise the intensity using the arcs, and then bring the peak into one of the PRISMA-3 detectors. First of all you need to know the appropriate ϕ angle. Let's say that one double-analyser arm has been set up with an analysing energy of 5 meV, and we want to look at the (1 0 4) peak. Go into PRSCAL and type the following:

```
CAL> bre 1 0 4 5 0 0
```

The program will respond with the appropriate Ω and ϕ angle you need to see the peak. You should do a small Ω scan to get the peak spot on.

3.3. PRISMA-4 Diffraction detector module

The PRISMA-4 detector module has 16 detector tubes, separated by 1° in ϕ so if $\phi_1 = -90$, then $\phi_{16} = -75$.

The operation of aligning a single crystal using PRISMA-4 is almost identical to that of PRISMA-2, except that all 16 tubes can now be used. For instance. you might begin with the following type of scan:

```
PRS> SC OM1-16=-45,0.1,NP=5
```

3.4. A Note of Caution

The procedure outlined in the previous section regarding single crystal alignment is really for the rare case of a completely trouble-free and easy alignment. In practice, numerous iterations around several of the steps may be necessary. Difficult alignments are usually a result of a poor prior alignment of the sample in its mount, particularly if the scattering plane of the crystal is tilted significantly from the horizontal in the mount. The arcs on the PRISMA goniometer can only be tilted by 15° in any direction and less if you are using a cryostat, which means that you may find that such a sample cannot be aligned during the first attempt on PRISMA. If this is the case, it must be removed from the beam and the excess tilt corrected on the mount itself. This can turn into an *extremely* time-consuming and tedious process, and it cannot be emphasised too strongly that you should align your sample as well as possible in its mount using x-rays before the start of the experiment. This should hopefully eliminate the possibility of one or more late nights at the start of your experiment!

3.5. Temperature Control

The temperature can be controlled either from the dashboard or using the PRISMA control program. It is conventional to turn the logging on (so that you can follow the temperature on the dashboard (the TEMP, TEMP1 or LAKES blocks)) within the control program using the following commands:

```
PRS> CSET TEMP/LOG
PRS> CSET TEMP1/LOG
PRS> CSET LAKES/LOG
```

Now change the temperature on the supervisor window of the workstation:

```
prisma/prisma: cset temp <your desired temperature>
prisma/prisma: cset temp1 <your desired temperature>
```

If the change involves heating, you may need to alter the power limits. Try

```
prisma/prisma: cset max_power <P%>
prisma/prisma: cset max_power1 <P%>
```

where P is a percentage of the total available, try 10 to start with. You may also need to change the operating voltage of the heater. This is controlled by a dial at the back of the Eurotherm crate, which sits in the rack in the side room of the PRISMA cabin.

The only other thing you may need to change are the PID parameters. The usual PID parameters for the PRISMA CCR are 1.0, 60.0, and 1.0, respectively. Values for other pieces of sample environment equipment are available in HELP, by typing

prisma/prisma: **help @temperature**

You can then change these parameters (prop, prop1, int, int1, deriv, deriv1) by typing, for instance

prisma/prisma: **cset deriv1 2.0**

At any time you may display a parameter by typing **cshow/enq**. For instance, if you type

prisma/prisma: **cshow/enq power1**

the current power will be returned.

If you wish to perform an alignment scan, it is easiest to turn the temperature logging off for the duration of the scan. This is because the logging slows the scan up. Type

PRS> **CSET TEMP/NOLOG**
PRS> **CSET TEMP1/NOLOG**
PRS> **CSET LAKES/NOLOG**

These commands simply mean that the dashboard no longer displays the current temperature, it does not mean that the temperature control itself is turned off.

In any case, you should have the temperature logging on while you are actually collecting data. You can change the set point from within the PRISMA control program by typing

PRS> **VX C=CSET TEMP** *<your desired temperature>*
PRS> **VX C=CSET TEMP1** *<your desired temperature>*

The **VX** command is used in command files for changing the temperature. This is explained in Section 4.5.

4. Collecting Data

4.1. Setting-Up the Time-Channel Boundaries

For both diffraction and inelastic scans, the time channel boundaries should be set up by typing the following:

```
PRS> SE START=15 19995 1
```

If you want to perform a scan (usually an inelastic scan for a single setting of the instrument) that will count indefinitely, i.e. until **end** is input to the supervisor window, type

```
PRS> SE MNT=0
```

Otherwise, type

```
PRS> SE MNT=1
```

which means that the DAE will count on ISIS frames. If you want to count for, say, 900000 frames (which is 5 hours), type:

```
PRS> SE MNS=900000
```

Remember that 50 frames=1 second.

4.2. Vanadium Calibration

It is usual to perform a calibration to account for the detector efficiencies before starting a diffraction or inelastic experiment. Do this by placing the vanadium rod in the beam (on a candlestick), and then run a scan that is effectively the same as the one that you will eventually use to collect data from your crystal. A calibration scan only requires about 40 minutes of beam, so you should type

```
PRS> SE START=15 19995 1 MNT=1 MNS=120000
```

before starting the scan. For calibrating diffraction scans which use detectors 13 to 16 you would type something like the following (after having driven ϕ_{13} to the appropriate value where you will collect data):

```
PRS> SC OM13-16=-45,0.0,NP=1
```

Obviously the Ω angle has no relevance for a vanadium calibration, and in the above command it was set to a nominal value and left there for the duration of the scan.

For calibrating an inelastic scan you have two choices, depending on the type of inelastic scan you will eventually use (see section 4.4). For a **CD** calibration, simply type the appropriate **CD** command on the LA120. For the more general type of inelastic scan, drive the ϕ angle and the analysing energies to their correct values and start the scan with

```
PRS> BEGIN
```

4.3. Diffraction Scans

When performing a diffraction scan you should consider how much of reciprocal space you need to cover in terms of the Ω angle. For a single setting of the spectrometer, each detector measures a radial scan in reciprocal space so that a set of detectors covers a fan-shaped area. Since the detectors are 1° apart in ϕ for the PRISMA-4 module, the separation corresponds to 0.5° in Ω. In the diagram below, we show a schematic view of reciprocal space for a crystal, with the fan corresponding to radial scans from 4 detectors.
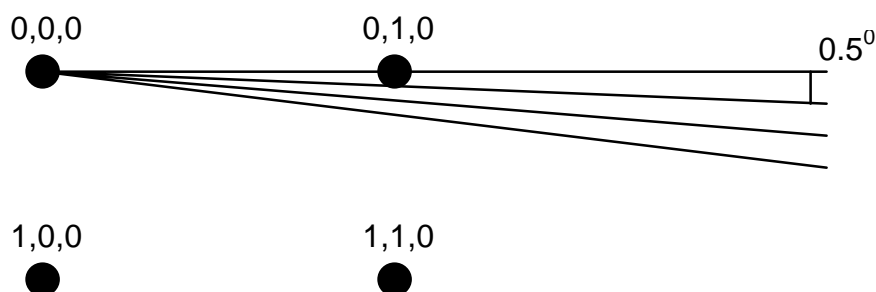
This corresponds to a single setting of the instrument. By rocking Ω, it is possible to build up a succession of the fans and cover a much larger portion of reciprocal space.

However, the first question to ask is, what sort of energy integration do you require in the part of reciprocal space in which you are interested? For instance, if you wanted to look at diffuse scattering at the zone boundary point (2.5 2.5 0) where there is a soft acoustic mode with an energy 8 meV, then you would require that the neutron energy for elastic scattering at (2.5 2.5 0) to be at least several times larger than 8 meV. This will ensure a sufficient integration over the soft mode, so that the quasi-static approximation is fulfilled. You can control this by changing the ϕ angle. Go into **PRSCAL** on the workstation, and use the **BR** command. For our example, using the default lattice parameters in PRSCAL, if you try

```
CAL> br 2.5 2.5 0 -90 0 0
```

where the fourth number is ϕ=-90°, PRSCAL gives the incident neutron energy for elastic scattering at (2.5 2.5 0) as E_i=31.96 meV. This is sufficient to integrate over the soft

mode, but it may be better to try an even higher energy to satisfy the quasi-static approximation more completely. In this case, a value of ϕ=-50° gives the much more satisfactory energy of E_i=89.46 meV. Since we are using 16 detectors, one of the detectors in the middle should be driven to this value of ϕ. To drive the scattering arm to the correct angle type

PRS> **DR PHI8=-50**

Note, it may be necessary at a later stage to change the value of ϕ slightly to eliminate problems with multiple scattering, since this is often highly dependent on the incident energy.

Now that you know the ϕ angles of the detectors you will work with, you can calculate the range in Ω for the area of reciprocal space you want to look at. To take yet another example, suppose you wanted to perform a fine scan around the (2 2 0) Bragg peak, using the ϕ angle that we have just determined. You could use two offset (but interleaving) scans with detectors 13 to 16. Since this makes four detectors in total, each setting covers a fan of 1.5° in Ω. Hence, by stepping the Ω angle, you can easily get a wide coverage of the area around (2 2 0). You could try two interleaving scans, each comprising five steps of 0.1°. To do this, you first need to find out the Ω angle corresponding to one of the middle detectors. In our example ϕ_{14}=-50°, and ϕ_{15}=-49°, so we could try

CAL> **br 2 0 0 -50 0 0**

which gives the Ω angle you need as -65° with the default lattice parameters. By using these two interleaving scans:

PRS> **sc om13-16=-64.8,0.1,np=5**
PRS> **sc om13-16=-65.3,0.1,np=5**

you can cover a range of nearly 4° in Ω, in steps of 0.1° very rapidly.

To cover a large portion of reciprocal space, interleaving scans are often unnecessary. So the scan

PRS> **sc om1-16=-65,16,np=3**

covers a much wider range by a fan with 14 "spokes" separated by 0.5°. The **rc** command in PRSCAL enables a plot of the scan trajectories in reciprocal space to be made. See the PRSCAL manual for full details.

In all cases, a diffraction run will be some sort of Ω scan, like those above. To set the number of frames that each point in the scan will be counted for, use the MNS parameter, after ensuring that MNT=1. See section 4.1

4.4. Inelastic Scans

Both the **CD** and the **BEGIN** commands described in the following two sections use a specified analyser-detector arm as their reference. This is the arm given by the parameter IANAL, and the default is IANAL=1. It also requires the parameters DMIN and DMAX to be set to the numbers of the arms being used for inelastic work. Usually, these values are 1 and 12 for PRISMA-2, and 1 and 5 for PRISMA-3, respectively. You can find out their current values with the command

PRS> **PR ANAL**

If the parameters are not correct, type

PRS> **SE IANAL=1 DMIN=1 DMAX=12**

and you answer "NO" (i.e. **N**) to the question about whether detectors 13 to 16 should be driven to their maximum angles.

4.4.1. The **CD** *Command*

This command enables an inelastic scan to be performed along a general direction in reciprocal space. It takes the form

CD *h k l* Δh Δk Δl *E*

These parameters are defined with reference to the analyser-detector arm given by IANAL. This arm measures a parabolic (**Q**,*E*)-trajectory about the (*h k l*) reciprocal lattice point, along the [Δh Δk Δl]* reciprocal lattice direction. *E* is the energy transfer where the trajectory crosses the (*h k l*) point. For instance, to perform a scan along the [1 1 0]* direction around the (3 -2 0) point you might type the following:

PRS> **CD 3 -2 0 1 1 0 -1.5**

You should always test out potential **CD** scans in PRSCAL, since there is a possibility that clashes of the analysers and detectors may occur, meaning that some configurations are physically impossible for the instrument. PRSCAL will tell you if your scan is possible or not, as well as giving you a plot of the trajectories in (**Q**,*E*)-space, in order to visualise the coverage. Note, because of the spectrometer geometry it is never possible to perform a **CD** scan with a pure transverse or longitudinal polarisation.

As an example, the diagram below shows typical scan trajectories (using PRSCAL), obtained with the command **CD 0 -6 6 0 0 1 2.5** for a crystal of KNO_3. The scattering plane was the **b*-c*** plane, so that the following commands were issued to the control program to set the sample parameters:

PRS> **SE A=5.425 9.120 6.228 90 90 90**

```
PRS> SE PX(1)= 0 1 0 0 0 1
```

The scan was performed simply by typing

```
PRS> CD 0 -6 6 0 0 1 2.5
```

after the time-channel boundaries had been set up, and values of `MNS` and `MNT` given. Also, don't forget to read section 4.6 on the **update** and **store** commands.

Q=(0.000 −6.000 6.000) DQ=(0.000 0.000 1.000) E0= 2.5
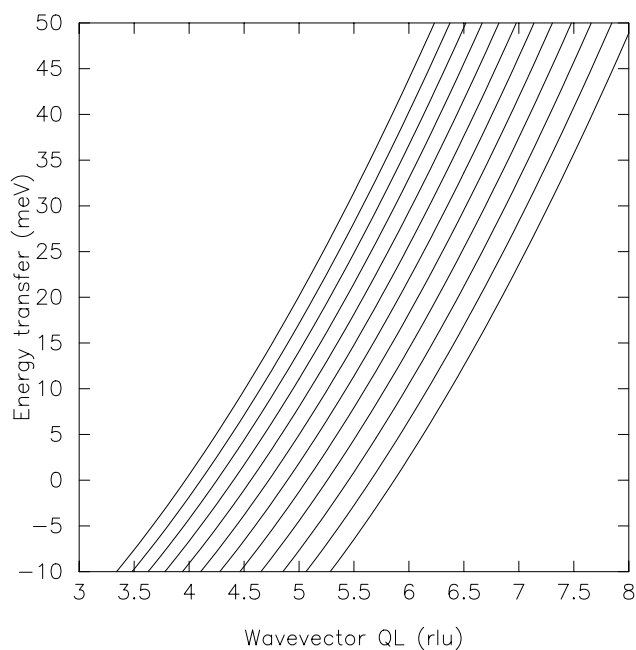
4.4.2. More General Inelastic Scans

It is possible to perform more general-purpose inelastic scans than are possible with the **CD** command. Here, you would drive each analyser-detector arm to a fixed analysing energy, and then position ϕ and Ω so that the scan trajectories cover the region of interest. `PRSCAL` is invaluable in considering this type of scan, because it will plot out the scan trajectories for you, and enable you to fine-tune all of the instrument parameters. The plots are made using the **rc** command in `PRSCAL`, and choosing the "CF path" option. The arms are driven to the desired analysing energies as in this example:

```
PRS> DR EF1-2=13.5 EF3=14
```

where arms 1 to 2 are driven to 13.5 meV, and arm 3 is driven to 14 meV. Note, that the lowest possible analysing energy you can use is about 13 meV with the PRISMA-2

general-purpose module and 3 meV with the PRISMA-3 double-analyser module. The scan is started by typing

PRS> **BEGIN**

after the appropriate time-channel boundaries, and values of `MNS` and `MNT` have been set. Also, don't forget to read section 4.6 on the **update** and **store** commands.

The diagram below shows the scan trajectories for arms 1 to 8, as used in an experiment looking at very broad inelastic scattering in the $(h\,h\,l)$ plane of a frustrated antiferromagnet. The parameters for the scan are Ω=-69°, ϕ_1=-53°, and `EF1-8`=18 meV. Each scan trajectory is a curve in the three-dimensional $(\mathbf{Q},E)$-space, and the diagram shows these trajectories projected onto the $\mathbf{Q}$-plane. The E-axis is thus out of the page. The slanting straight lines are the projections of the scan trajectories, and the curves that cut across these trajectories represent constant energy contours. We only show contours between -10 and 10 meV in this diagram, in steps of 5 meV.

OM=−69, PHI1=−53, EF1−8=18 meV
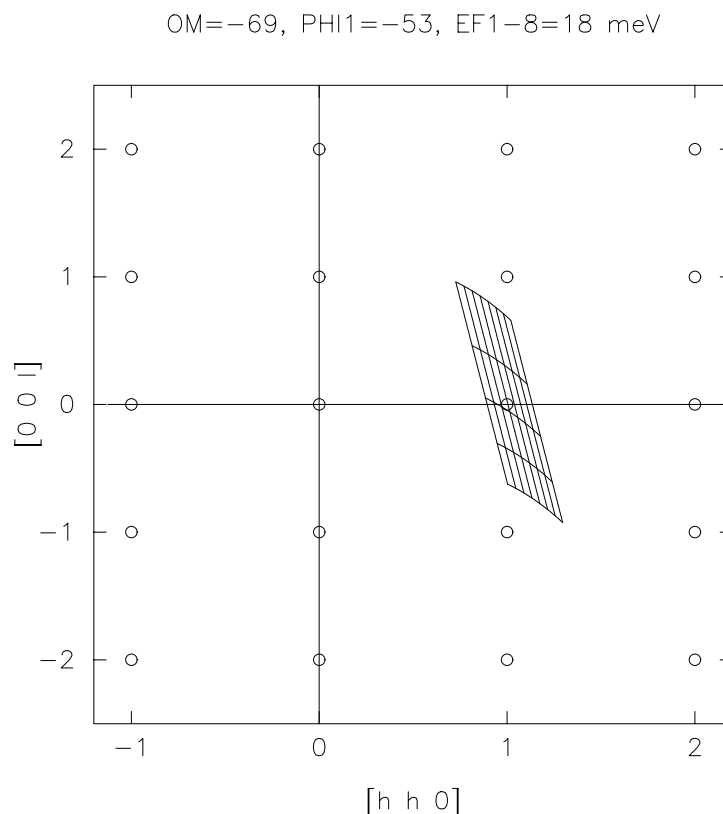
4.5. Using Command Files to Control PRISMA

Command files may be written to control the instrument, and are best edited on the workstation. The correct directory to use is `prisma$disk0:[prisma.run]`, and the command file should end with the extension "`.JOB`". An example command file (called "`EXAMPLE.JOB`") follows, illustrating a sequence of most of the commands you might need. Note, these commands may typed into the file directly without starting each line with a `$`, as you would in a usual VMS command file. It is very important to note that the command to change temperature has a slightly different format to the usual `CAMAC` command you would use on the workstation.

```
se start=15 19995 1            sets the time channel boundaries for a run
se mns=900000 mnt=1            sets the duration of the scan to be 900000
                               frames
cset temp/log                  sets TEMP logging on
cset temp1/log                 sets TEMP1 logging on
ti example : cd 0 -6 4 0 0 1 3.5   change the title of the scan to
                               "example : cd ..."
cd 0 -6 4 0 0 1 3.5            runs the scan, for 900000 frames
vx c=cset temp 250             sets TEMP to 250 K
vx c=cset temp1 250            sets TEMP1 to 250 K
dr phi9=-50                    drives the φ angle for detector 9 to 50°
se mns=3000                    sets the duration of each time period to be
                               3000 frames, i.e. one minute per point
sc om9-16=-30,0.1,np=11        omega scan using detectors 9 to 16
se mnt=0                       sets the duration of the scan to last until
                               operator intervention (i.e. END or ABORT
                               is typed in the supervisor window)
ti example at 250 K : inelastic scan    change the title of the scan
begin                          runs the scan, until operator intervention
```

When you have finished editing the file, turn the dashboard on again by typing

`prisma/prisma:` **stat on**

The command file may then be run by typing

`PRS>` **DO EXAMPLE.JOB**

in the control program. Execution of the whole command file may be stopped only when the dashboard shows that PRISMA is actually running, and this is done simply by typing **end** or **abort** in the supervisor window on the workstation.

4.6. Ending, Pausing, or Aborting a Run

The commands to end, pause, or abort a run should all be typed in the supervisor window, while the dashboard shows that "PRISMA is RUNNING". These commands are simply **end**, **pause**, or **abort**! **end** stops a run and stores the data in the DAE in the file prisma$disk0:[prsmgr.data]prs<*run no.*>.raw, **pause** stops the DAE from counting neutrons until **resume** is typed in the supervisor window, and **abort** ends a run without saving the data.

In addition, it is possible to save the DAE data to a file without ending the run. This means that you can look at a run periodically to see how it is getting on. The commands you need are **update** and **store**, typed in this order in the supervisor window. These store the current data in the DAE in the file

prisma$disk0:[prsmgr.data]prs<*run no.*>.sav.

If you leave PRISMA running for long periods of time (say, more than 12 hours), you should do this sort of backup anyway in case something goes wrong by running the command file time_upd.com . In the supervisor window, type

@time_upd <n>

where **<n>** is the number of hours between saves. The command file can be interrupted by typing **<CTRL>+Y.**

Here is a summary of all the commands you need to know from this section. All of the commands may be abbreviated to their first three letters.

update	Stores the data collected so far in the current run parameter table (*crpt*)
store	Stores the data collected up to the last **update** in the file prisma$disk0:[prsmgr.data]prs<*run no.*>.sav. The **store** command should always be preceded by an update
pause	Pauses data collection.
resume	Resumes data collection
abort	Aborts the current run without saving any data.
end	Ends the current run and stores the data in prisma$disk0:[prsmgr.data]prs<*run no.*>.raw

5. Data Analysis and Visualisation.

Several programs and utilities exist to help you analyse your data, all of which are explained more fully in the other manuals in the PRISMA cabin. The first stage of data analysis is always performed inside GENIE, and a brief introduction is given here. More detail may be obtained from the PUNCH Manual. To use the PRISMA data analysis routines from your own account, you should add the following line to the login.com file in your top level directory

$ @prisma$disk0:[prsmgr]login.com

Note, that if you are about to run GENIE or PRSPLOT in your own account on a window on the PRISMA workstation, type "**set disp/create/node=prisma**" before you start. This will ensure that the correct graphics windows are created.

5.1. **GENIE**

GENIE is the ISIS graphics software. To run GENIE from any ISIS computer with VMS simply type "**genie**". Note, unless you wish to look at data in the DAE itself (e.g. during a crystal alignment when the macros **DD** and **PP** are used), then you should run GENIE in your own account either on the workstation, or HORUS, or any other DEC Alpha machine. If you wish to look at the data currently stored in the DAE, you will only be able to do it while logged onto the instrument account with the userid PRISMA.

Individual *x*,*y*,*e*-datasets are held inside "workspaces" in GENIE, and it is possible to manipulate the *y*-values of each workspace by using the usual mathematical symbols +, -, *, / for add, subtract, multiply, or divide. For example, if you wanted to subtract the *y*-values in workspace 2 from those in 1, and then multiply the answer by 100000, you would type the following:

```
>> w3=w2-w1
>> w3=w3*1.0e5
```

where the result is stored in workspace 3. Note that for this to work, the *x*-values for the two workspaces need to be the same. If this is not so, then you will need to **rebin** the two workspaces first; see the GENIE section of the PUNCH manual. To perform more sophisticated manipulations of workspaces, the **function** and **transform** commands should be used, which are also explained in the PUNCH manual.

Now we will briefly describe the commands for viewing the data in a workspace. First you must decide whether you wish to plot your data as points or histograms. Toggle between the two modes by typing the following:

```
>> t m
```

The workspaces may be displayed with the command **d**. The plot command, **p**, allows you to plot the data as markers with error bars, or to plot one workspace on top of another. For example

>> **d/m w1 -10 90 0 10**   *plots intensity of workspace 1 as markers from 0 to 10 against energy transfer from -10 meV to 90 meV*

>> **p/e w1**   *adds error bars*
>> **p/l w2**   *overplots w2 as a series of lines joining up its points*

You may alter the binning of your data by

>> **a b 5**   *bins the data into groups of 5*

To get a hardcopy of a plot use the following two commands

>> **k/h**
>> **j "plaser**<*n*>**dec_postscript.dat"**

where the laser printer number, *n*, identifies the location of the printer according to the table below.

laser printer number	location
0	Computer support office, R3.
1	Coffee room, R3.
2	DAC, R55.
7	PRISMA cabin

To leave GENIE, type

>> **exit**

5.1.1. PRISMA GENIE *Data Analysis Programs*

Various GENIE macros and FORTRAN programs exist for analysing PRISMA data. The **PP** facility has already been introduced in section 3.1, and here we give a list of all of the macros you might need to use.

More detailed information is given in the PRISMA GENIE Data Analysis Manual. The first three macros in the following list (the "alignment macros") may be used to look at the current run simply by typing DAE when the run number is requested. In this case, you must be running GENIE on the PRISMA workstation. For the other data analysis macros, you must be running GENIE on a computer other than PRISMA, and if you wish to look

at the current run, you must first have done an **update** and **store** (see section 4.6), and within GENIE have typed

>> **set ext sav**

Also, when the particular macro asks the following question:

Enter file extension (default is RAW) ?

reply with "**sav**".

Macros useful during alignment (may be used for analysing the DAE):
>> **DD**   *Displays diffraction data for a single-detector scan*
>> **PP**   *Displays diffraction data for a multi-detector scan*
>> **AA**   *Integrates an Ω, φ, GX, or GY scan so that a counts vs. angle plot can be made*

Macros for analysing inelastic data:
>> **MRX**   *Transforms time-of-flight inelastic spectra to energy transfer spectra*
>> **CC**   *Transforms raw data to PRSPLOT format for contour plots*

Macros for analysing diffraction data:
>> **VCRS**   *Transforms raw data to PRSPLOT format for contour plots*

Macros for analysing temperature during a run:
>> **TP**   *Creates a temperature plot from a log file*
>> **TPC**   *Creates a temperature plot from the DAE*
>> **AT**   *Calculates the average temperature of a run*

Other useful macros:
>> **FF**   *Fits a workspace with Gaussian, Lorentzian,*
or   *Kropf or Reverse Kropf functions*
>> **TT**   *Transforms a workspace from time-of-flight to wavelength, incident energy, d-spacing or wavevector*
>> **ERR**   *Rescales error bars which are too small*

5.2. Inelastic Data

Inelastic spectra from individual detectors may be transformed into energy transfer using the **MRX** macro in GENIE, and then displayed. After typing

```
>> mrx
```

and giving the run number, etc., the macro asks whether any of the parameters (such as lattice parameter, analysing energy etc.) should be changed from what they were stored as when the data were saved. For instance, you might have decided that the lattice parameters that you typed into the PRISMA control program when you aligned the crystal were wrong, and so you can easily change them again at the data analysis stage. After that, it asks for which detectors are to be analysed, and the energy transfer required. The useful range is -20 to 100 meV for the general-purpose inelastic module. For the inelastic double-analyser module, the range of accessible energy transfers varies considerably depending on the analysing energy used.

However, it is usually more satisfying to create a colour contour plot of the data using PRSPLOT. Before this, the raw data must be processed using the **CC** macro in GENIE. The following list of operations is a step-by-step guide to processing an inelastic data set using **CC** and then plotting it with PRSPLOT.

(i) Create a "rebin file" outside of GENIE which the macro uses to bin your data as it is processed. This file should be in the GENIE command file format, and should have a relatively short name, such as "BIN.COM" for instance. The file consists of only one line which is a **rebin** command for workspace 3, so an example might be:

```
> reb w3 -10 (0.5) 20 (1) 40 (2) 60
```

and the data will be binned with the variable steps in the range -10 to 60 meV

(ii) Start GENIE, and type

```
>> cc
```

(iii) Answer the next question about monitor efficiency with a "**1**"

(iv) Enter the name of the rebin file

(v) Type in the run number, and answer the usual questions about which directory the data file is in, and whether it is a .RAW, or .SAV file

(vi) Answer the question about whether the sample parameters (i.e. lattice parameters, analysing energies, etc.) need to be altered

(vii) Type in the first and last detectors to be analysed, and then answer the prompts for individual detectors with a **0** or **1**.

(viii) When the run has been analysed, the macro asks whether another run should be processed. The output from all runs is written to a file called CONTOUR.OUT, and subsequent runs will be appended to it. When you have processed all of the runs required

for one data set, rename `CONTOUR.OUT`, so that any subsequent operations of **CC** with other data sets will create a new `CONTOUR.OUT`, rather than appending to the old one.

(ix) An optional step is to create a normalisation file at this point. It is not necessary to have one just for a rough look at data in `PRSPLOT`, but since the detector efficiencies may vary quite markedly, you will need one to produce good plots. The normalisation file has the following format: the first line is the run number, and the following 16 lines contain three columns, with the detector number, a scale factor, and background. After that, there may be another run number, and 16 more rows etc. if your `CONTOUR.OUT` file contains data from more than one run. The scale factors are obtained from the vanadium calibration that you should have performed at the start of your experiment. `PRSPLOT` will alter data according to the following formula:

modified data=scale factor*(original data-background)

Here is an example normalisation file:

```
8173
1                  1.235               0.0
2                  1.376               0.0
3                  0.971               0.0
4                  2.011               0.0
5                  1.037               0.0
6                  0.978               0.0
7                  1.349               0.0
8                  0.645               0.0
9                  1.301               0.0
10                 0.887               0.0
11                 0.992               0.0
12                 1.035               0.0
13                 1.0                 0.0
14                 1.0                 0.0
15                 1.0                 0.0
16                 1.0                 0.0
8174
1                  1.235               0.0
2                  1.376               0.0
3                  0.971               0.0
```

and so on...

(x) The next step is to start `PRSPLOT`, simply by typing "**prsplot**", answering the questions about terminal type ("`MX11`" if you are using a workstation, or a PC running an X-Windows emulator), hardcopy device (usually "`Colour postscript`"), and font ("**simp**", "**comp**", or "**cenb**" are three possibilities).

(xi) Type

> **l w1 contour.out**

or whatever the title is if you have renamed it.

(xii) Choose option **5** for the "FILE TYPE", and then either **1** or **2** for "CD" or "CEF" data (i.e. a general inelastic scan)

(xiii) The data are then read in, and you must choose the *x*-axis variable. For instance, if your data were collected with the command **CD 0 -6 6 0 0 1 2.5**, the *x*-axis variable would be L, and you would reply with a "**3**".

(xiv) Type in the name of your normalisation file if you have one, or give a carriage return if you don't

(xv) Type in the titles of the axes for the plot

(xvi) You are now ready to plot your data by typing

> **d/s w1**

for the whole data set, or

> **d/s w1** *<xmin> <xmax> <ymin> <ymax>*

for a section of it. You can also change the contour levels by typing

> **a/h i**

(xvii) To make a hardcopy of your plot, type

> **s**

and then leave PRSPLOT by typing

> **j/p**

and print the POST.DAT file that has been created. You can print it on the colour printer in the coffee room in R3 with the command:

$ **print/queue=colour$phaser0/notify post.dat**

5.3. Diffraction Data

The procedure for visualising diffraction data is similar to that for inelastic data. The raw data must first of all be processed in `GENIE`, to produce the file `CRITICAL.OUT`. The macro **VCRS** is used for this purpose. A step-by-step guide to processing diffraction data to produce a colour plot is given here:

(i) Create a "rebin file" which the **VCRS** macro uses to bin your data as it is processed. This file should be in the `GENIE` command file format, and should have a relatively short name, such as "`BIN.COM`" for instance. The file consists of only one line which is a rebin command for workspace 4, so an example might be:

```
> reb w4 0.5 (0.01) 1.5
```

The limits are the wavevector transfer in units of Å^{-1}, and the binning should be chosen so that there are no more than 500 bins in the workspace. In this example there are 100.

(ii) Go into `GENIE`, and type

```
>> vcrs
```

(iii) Enter the name of the rebin file

(iv) Answer "**1**" to calibrate your data with a vanadium run

(v) Type in the run number of the vanadium calibration, and answer the usual questions about the sample parameters, and the detectors to be analysed.

(vi) Answer the next question about smoothing the vanadium with a "**1**".

(vii) When the vanadium run has been analysed, the macro asks for the first run number of the data set to process. The macro will then process this, and come back asking for another. The output is written to a file called `CRITICAL.OUT`, and subsequent runs will be appended to it. When you have processed all of the runs required for one data set, rename `CRITICAL.OUT`, so that any subsequent operations of **VCRS** with other data sets will create a new `CRITICAL.OUT`, rather than appending to the old one.

(viii) The next step is to start `PRSPLOT`, simply by typing "**PRSPLOT**", answering the questions about terminal type ("`MX11`" if you are using a workstation, or a PC running an X-Windows emulator), hardcopy device (usually "`Colour postscript`"), and font ("**simp**", "**comp**", or "**cenb**" are three possibilities).

(ix) Type

```
> l w1 critical.out
```

or whatever you have renamed your file to.

(x) Choose option **6** for the `"FILE TYPE"` question

(xi) The data are then read in, and you must choose the variables along the *x* and *y*-axes, and the angle between the two axes

(xii) The next prompt is for the name of your normalisation file. If when you processed your data using **VCRS**, you included the vanadium run at the start, then the normalisation is automatically included in the data, and you can simply give a carriage return here. Otherwise, you may use a normalisation file, as for the inelastic data.

(xiii) Type in the titles of the axes for the plot

(xiv) You are now ready to plot your data by typing

> **d/s w1**

for the whole data set, or

> **d/s w1** *<xmin> <xmax> <ymin> <ymax>*

for a section of it. You can also change the contour levels by typing

> **a/h i**

(xv) To make a hardcopy of your plot, type

> **s**

and then leave `PRSPLOT` by typing

> **j/p**

and print the `POST.DAT` file that has been created. You can print it on the colour printer in the coffee room in R3 with the command:

$ **print/queue=colour$phaser0/notify post.dat**

6. SUMMARIES

6.1. Instrument Control

6.1.1. PRISMA Control

PRS> **SE START=1000 10000 10**	*Sets time-channel boundaries for crystal alignment*
PRS> **SE START=15 19995 1**	*Sets time-channel boundaries for full data collection*
PRS> **TI** *\<character string\>*	*Sets title*
PRS> **SE MNS=1000 MNT=1**	*Sets number of frames to be counted in each period to be a 1000 (this is usual for sample alignment)*
PRS> **SE MNT=0**	*Sets run to end only by operator intervention (i.e. an* **END** *or* **ABORT** *in the Supervisor window)*
PRS> **DR EF\<n_1\>-\<n_2\>=\<E\>**	*Drives analysers/detectors n_1-n_2 to the analysing energy E*
PRS> **BEGIN**	*Starts collecting data in current configuration*
PRS> **CD** *\<h\> \<k\> \<l\> \<u\> \<v\> \<w\> \<E_0\>*	
	Inelastic run through h, k, l along the u, v, w direction
PRS> **SC OM\<d_1\>-\<d_2\>=\<Ω\>,\<$\Delta\Omega$\>,NP=\<***no. of steps***\>**	
	Scan in omega angle, centred on Ω and collecting the data in detectors d_1-d_2, with a step size of $\Delta\Omega$
PRS> **CSET TEMP/LOG**	*Sets temperature* TEMP *logging on*
PRS> **CSET TEMP/NOLOG**	*Sets temperature* TEMP *logging off*
PRS> **VX C=CSET TEMP** *\<t\>*	*Changes the setpoint of* TEMP *to T*

6.1.2. PRISMA Supervisor Window

prisma/prisma: **pause**	*Pauses the current run, so that no data are collected*
prisma/prisma: **resume**	*Resumes data collection after a* **PAUSE**
prisma/prisma: **end**	*Ends the current run*
prisma/prisma: **abort**	*Aborts the current run*
prisma/prisma: **update**	*Stores the current data in the* CRPT
prisma/prisma: **store**	*Stores the* CRPT *in a* .sav *file*
prisma/prisma: **cset temp/log**	*Turns the temperature logging on*

```
prisma/prisma: cset max_power <P%>
```
Sets the maximum power to be used to P (as a percentage of the highest power available)

```
prisma/prisma: cset temp <t>
```
Sets TEMP *to temperature t*

6.2. Data Analysis and Visualisation

```
**********************************************************************
```
Note, if you are about to run GENIE or PRSPLOT in your own account on a window on the PRISMA workstation, type "**set disp/create/node=prisma**" before you start. This will ensure that the correct graphics windows are created.
```
**********************************************************************
```

6.2.1. GENIE

```
>> t m
```
Toggles between point-plotting and histogram-plotting mode

```
>> d/m w<workspace> <xmin> <xmax> <ymin> <ymax>
```
Displays the given workspace with markers (in point mode)

```
>> d/h w<workspace> <xmin> <xmax> <ymin> <ymax>
```
Displays the given workspace as a histogram (in histogram mode)

```
>> a b <bin>
```
Bins the data into groups of 'bin'

```
>> p/e w<workspace>
```
Plots error bars

```
>> p/l w<workspace>
```
Plots the workspace as a line over the top

```
>> k/h
```
Creates a hardcopy file

```
>> j "plaser7 dec_postscript.dat"
```
Prints on the PRISMA laser printer

```
>> j/p
```
Leaves GENIE *temporarily, type* "**log**" *to get back in*

```
>> exit
```
Leaves GENIE

6.2.2. Inelastic Data

```
>> mrx
```
GENIE *macro to transform raw data to energy transfer for quick visualisation*

```
>> cc
```
GENIE *macro to process raw data into full* PRSPLOT *format. Data written to* CONTOUR.OUT

6.2.3. Diffraction Data

>> **vcrs**   GENIE *macro to process raw*
                                                     data into full PRSPLOT *format.*
                                                     Data written to
                                                     CRITICAL.OUT

6.2.4. PRSPLOT

> **l w1** *<file name>*   *Loads data into* PRSPLOT
> **a/h i**   *Alters contour levels*
> **d/s w**<*workspace> <xmin> <xmax> <ymin> <ymax>*
                                                     Displays the given workspace as
                                                     a colour contour plot
> **t w1**   *Allows you to change the axis*
                                                     titles for a contour plot
> **a/n i**   *Alters normalisation (only use in*
                                                     the absence of a vanadium
                                                     normalisation)
> **x/p w**<*a free workspace> <xmin> <xmax> <ymin> <ymax>*
                                                     Cuts through the contour data set
                                                     between the given limits, and
                                                     puts the result in the specified
                                                     workspace
> **d/m w**<*workspace> <xmin> <xmax> <ymin> <ymax>*
                                                     Displays the given workspace
                                                     with markers (in point mode)
> **s**   *Writes the plot to a postscript*
                                                     file, called POST.DAT

6.3. A Final Checklist

Before you leave the experiment running for the night, quickly go through the following checklist:

- Interlocks complete
- Shutter open
- Heater on (if necessary)
- "PRISMA IS RUNNING" is shown at the top of the dashboard, and the "Counts" section (under "Monitor") of the dashboard is updating at about 16,000 counts every time. If the monitor is updating at a much lower (or higher) rate, then there are three possibilities: the first is that the proton beam current is low, the second is that the methane moderator is not working (you can find out about its status by phoning the MCR). Otherwise, the chopper has stopped, which is a job for the local contact.

7. Useful Phone Numbers

In the event of any problems with the instrument, computing, or sample environment, your first point of contact is your local contact, failing that any member of the PRISMA team. The names and phone numbers of the PRISMA team are

	RAL extension	**Home**
Mark Harris	6397	9-01865-724612
Martyn Bull	5805	9-01865-515520
Uschi Steigenberger	5145	9-813284

To dial a RAL extension from outside dial 01235 44, followed by the extension number. To make an external call from a RAL phone dial 9 before the number. To page somebody dial 70, choose option 1, enter the pager number, followed by an 11-digit message, usually the extension number you are calling from followed by sufficient zeros to fill the message. Wait for the tone before replacing the receiver.

Other numbers

	RAL extension	
ISIS Main Control Room	6789	
EMERGENCY	2222	
ISIS Health Physics	6696	
ISIS User Liaison Office	5592	5103 (fax)
Main Gate	5545	

8. Eating and Drinking

8.1. On-site

R22 Restaurant

	Mon - Fri	**Sat - Sun**
Breakfast	0730 - 0830	0800 - 0900
Lunch	1145 - 1345	1200 - 1300
Dinner	1715 - 1915	1800 - 1900

R1 coffee lounge hot drinks/snacks		0930 - 1130	(Monday-Friday)
		1200 - 1530	"
R22 coffee lounge		1200 - 1345	"

8.2. Pubs

Blewbury	The Red Lion
Chilton	Rose & Crown
East Hendred	The Plough,
	Wheatsheaf
East Ilsley	The Crown and Horns,
	The Swan
Steventon	The Cherry Tree
Wantage	The Lamb,
	The Swan
West Hendred	The Hare
West Ilsley	The Harrow

Appendix A
PRISMA Instrument Parameters

Beamline	N2
Moderator	95 K Methane poisoned at 2.25 cm
Incident flight path	9.035 m
Incident collimation (effective)	Horizontal: 15'
	Vertical: 23'
Beam size	Width: 30 mm
	Height: 50 mm
Beam height	Above sample table: 0.635 m
	Above goniometer: 0.246 m
Incident energy	3-250 meV
Monitor 1 position	-1.052 m
Monitor 2 position	1.037 m
Line-up detectors	5 ^{3}He 10 atm elliptical gas tubes
	at + 90°
	Final flight path: 1.056 m

PRISMA-2

Final flight paths	
Sample to analyser	0.573 m
Analyser to detector	0.170 m
Analysers	
Pyrolytic graphite (0 0 2)	Width: 45 mm
	Height: 55 mm
Germanium	Width: 10 mm
	Height: 40 mm
Mica	
Detectors	16 12.5 mm ^{3}He gas tubes (Reuter Stokes)
Angular separation of detector arms	2°
Final collimation	Horizontal: 60'
	Vertical: 5°
Final energy	12 meV < E_f < 30 meV
Angular range	-120° to +40°
Resolution	$dE_f/E_f = (0.04E_f + 4.58)$%
	(Germanium)

Notes:
1. The PRISMA-2 detector is normally operated with 12 inelastic detector arms and 4 diffraction detector arms (ie. analysers removed from inelastic arms).
2. It is not possible to operate PRISMA-2 and PRISMA-3 simultaneously.

PRISMA-3

Final flight paths

Sample to front analyser (LSF)	0.600 m
Front analyser to rear analyser (LFR)*	0.045 m
Rear analyser to detector (LRD)*	0.211 m

Analysers

Pyrolytic graphite (0 0 2)	Width: 45 mm
	Height: 55 mm
Detectors	5 1/2" 10 atm ^{3}He gas tubes
	(Reuter Stokes)
Angular separation of detector arms	10°
Final collimation	Horizontal: 30'-nil, 60'-nil, 30'-60'
	Vertical: 5°
Final energy	3 meV $< E_f <$ 12 meV
Resolution	30'-60' : $dE_f / E_f = 4\%$
	60'-nil : $dE_f / E_f = (2.3+0.5E_f)\%$

Notes:
1. Lengths marked * are defined when LFR is at 90° to LSF.
2. PRISMA-3 can be operated simultaneously with PRISMA-4. The minimum detector separation is 60°.
3. Pyrolytic graphite filters are available to remove second order contamination.
4. Beryllium filters are available for use with $E_f <$ 5 meV.

PRISMA-4

Final flight path	0.825 m
Detectors	16 ¼" ^{3}He gas tubes (Reuter Stokes)
Angular separation of detector arms	1°
Final collimation	Horizontal: 30', 60'
	Vertical: 1°- 3°

Notes:
1. PRISMA-4 can be operated simultaneously with PRISMA-3. Minimum detector spacing is 60º.

Appendix B
Useful Conversion Factors

Density: ρ (gcm^{-3}) = A (amu) x ρ (atoms Å^{-3}) x 1.66057
where A is atomic mass and ρ is density.

Neutron Energy:

$$E = \frac{\hbar^2 k^2}{2m} = \frac{h^2}{2m\lambda^2} = h\upsilon = \frac{m}{2}\frac{L^2}{t^2} = k_B T \qquad\qquad \lambda = 3955.41\frac{t}{L}$$

$$E = 2.0717 k^2 = \frac{81.787}{\lambda^2} = 4.1354\upsilon = 5.2276\times10^6\frac{L^2}{t^2} = 0.086165T$$

E[meV], λ[Å], υ [Thz], k[Å^{-1}], L[m], t[μsec], T[K]

Appendix C
Geometrical Relationships For Simple Crystal Structures

Interplanar spacings

Orthorhombic $\quad \dfrac{1}{d^2} = \dfrac{h^2}{a^2} + \dfrac{k^2}{b^2} + \dfrac{l^2}{c^2}$ $\qquad$ Tetragonal: a = b
$\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad$ Cubic: a = b = c

Hexagonal $\quad \dfrac{1}{d^2} = \dfrac{4}{3}\left(\dfrac{h^2 + k^2 + l^2}{a^2}\right) + \dfrac{l^2}{c^2}$

Rhombohedral $\quad \dfrac{1}{d^2} = \dfrac{\left(h^2 + k^2 + l^2\right)\sin^2\alpha + 2\left(hk + kl + hl\right)\left(\cos^2\alpha - \cos\alpha\right)}{\alpha^2\left(1 - 3\cos^2\alpha + 2\cos^3\alpha\right)}$

Monoclinic $\quad \dfrac{1}{d^2} = \dfrac{1}{\sin^2\beta}\left(\dfrac{h^2}{a^2} + \dfrac{k^2\sin^2\beta}{b^2} + \dfrac{l^2}{c^2} - \dfrac{2hl\cos\beta}{ac}\right)$

Volumes of Unit Cells

Orthorhombic $\quad$ V = abc $\qquad\qquad$ Tetragonal: a = b
$\qquad\qquad\qquad\qquad\qquad\qquad\qquad$ Cubic: a = b = c

Hexagonal $\quad V = \dfrac{\sqrt{3}}{2}a^2 c = 0.866a^2 c$

Rhombohedral $\quad V = a^3\sqrt{1 - 3\cos^2\alpha + 2\cos^3\alpha}$

Monoclinic $\quad V = abc\sin\beta$

Reflection Conditions

Simple Cubic (sc)	all reflections allowed
Face Centred Cubic (fcc)	h, k, l must be all even or all odd
Body Centred Cubic (bcc)	($h+k+l$) must be even
Hexagonal Close Packed (hcp)	allowed reflections must satisfy $l = 2n$ or h-k-1 = 3n or h-k-2 = 3n, where n is an integer