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## **A Practical Neutron Training Course : Course Notes**

**S M Bennington A C Hannon and J B Forsyth**

December 1993

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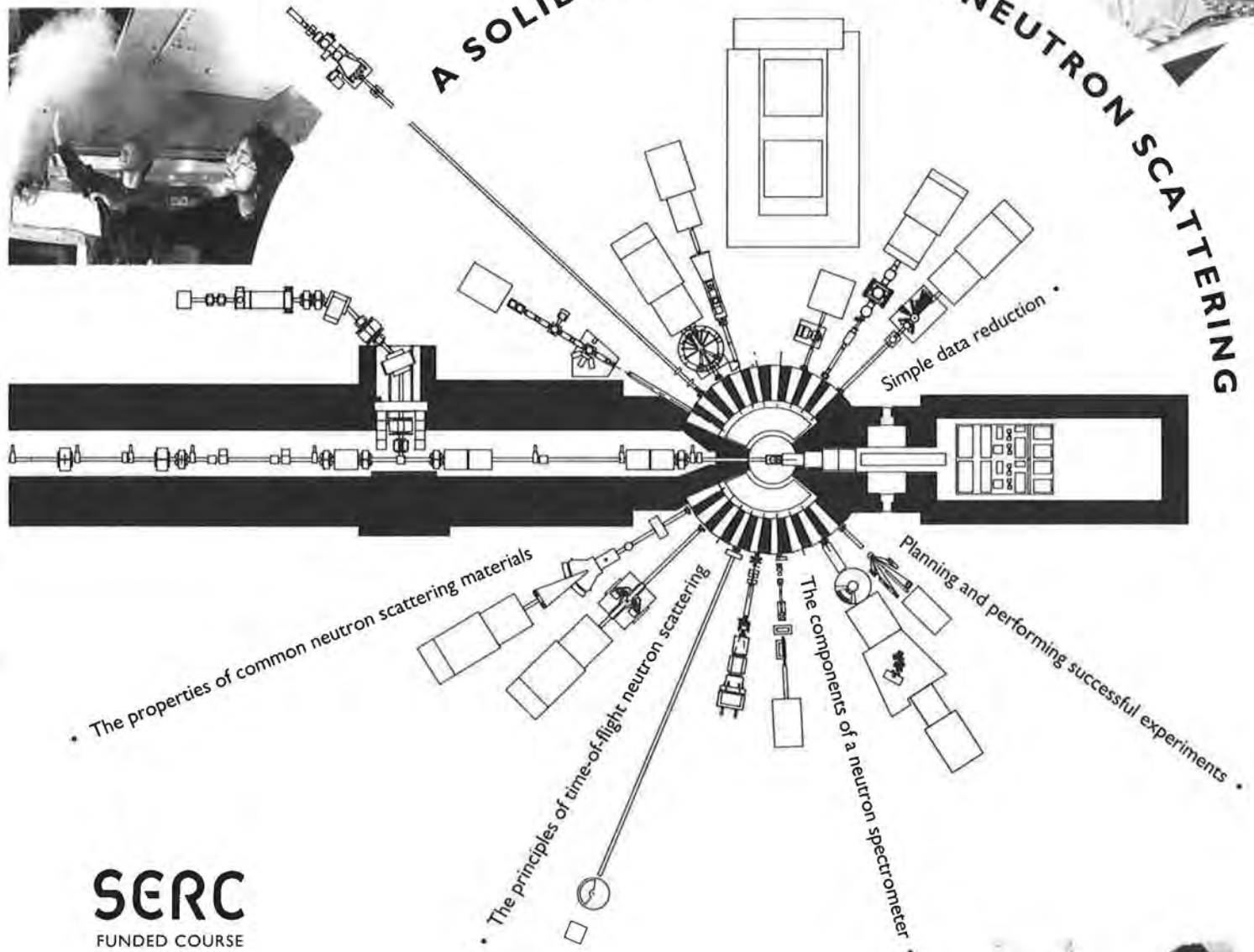
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For further information contact:  
S.M. Bennington (5193) or A.C. Hannon (5358)  
Tel: 0235 44 + extension Fax: 0235 445720  
The Rutherford Appleton Laboratory  
Chilton, Didcot, OXON OX11 0QX





**A Practical Neutron Training Course :  
Course Notes**

**S.M. Bennington, A.C. Hannon and J.B. Forsyth**



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## Introduction

The aim of this course is to provide students who are just starting work in neutron scattering with practical experience of running neutron scattering experiments. Beam time on both reactor and accelerator sources is very valuable and greatly sought after by scientists throughout the world. In such an intense situation a mistake that ruins an experiment cannot be afforded and hence there is little scope for the newcomer to the field to gain hands-on experience. This course is the only time in a neutron scattering career where it is possible to safely learn by making mistakes.

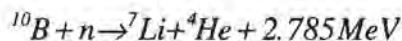
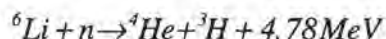
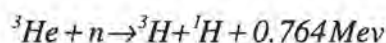
The course is constructed so as to provide an introduction to the practicalities of running neutron scattering experiments, especially those performed on a pulsed neutron source. It is intended to be similar in structure to a university undergraduate practical, and it is divided into a series of experiments which explore various aspects of neutron scattering. Although some guidance is given, the majority of the work is to be done alone or in small groups. Laboratory notes are provided as a guide to the experiments, and the information is presented in a logically consistent manner, with later sections making use of concepts introduced in earlier sections. The course is not meant to be a complete self-contained introduction to neutron scattering as this would take considerably longer than one week, but it attempts to provide foundations and to introduce ideas that will be built upon in subsequent practical work.



## 1. Detector Set up

Two different types of detector are used during the training course: The first is a  $^3\text{He}$  gas based detector, and the second is a scintillator made from a Zinc-Sulphide glass. Each have properties that suit them to different uses. The efficiency, gamma sensitivity, and intrinsic noise of a detector depends partly on its physical characteristics but also on the set-up of its electronics chain. In this section you will learn how to set-up these detectors. This may be the only time you will ever have to do this, but in the process we hope to instil in you a familiarity with properties of the two different types of detector and the function of the components in their electronics chain. Usually when doing a neutron scattering experiment it is not necessary to be aware of the details of the spectrometer. However, neutron instruments are complex pieces of scientific equipment and they occasionally malfunction. Although you may never be expected to correct an instrument fault, to be able to recognise that something is amiss is enormously valuable.

To detect a neutron it is necessary to convert its arrival at a particular time and place into an electrical pulse. Since neutrons are neutral it is not possible to do this directly as it is with a charged particle. The neutron must first be used to create charged particles in a nuclear reaction. Although many such reactions exist most are unsuitable for chemical reasons and only a few are commonly used in neutron detectors.



The recoil energy of the charged decay products causes further ionisation and it is this that is usually detected.

### 1.1. The $^3\text{He}$ Gas Detector

The gas detectors used in these experiments consist of a stainless steel tube filled with 10 bars of  $^3\text{He}$ . Stretched down the centre of the tube is a very thin tungsten wire charged to a high voltage. The ionised gas created by the nuclear reaction is accelerated by this voltage separating the electrons from their positive ions. The voltage on the central anode is maintained at a level high enough to create a cascade discharge when the electrons reach it, thus amplifying the original signal. If operated in the correct regime the voltage of the electrical pulse produced is directly proportional to the energy deposited by the nuclear reaction; hence the generic name for these devices - proportional detectors.

Despite the amplification within the detector the voltage pulse produced is only of the order of  $\mu\text{Vs}$  and would be lost in the noise pick of a cable of any reasonable length. To overcome this problem a pre-amplifier is used close to the detector to boost the signal so that it can reach the external electronics. The signal is then passed through a discriminator which discards pulses which are too large or small to be due to neutrons. It is then further amplified and converted into a square wave pulse which can be processed by more sophisticated digital electronics. On steady state source, such as a reactor this would be a scalar counter which simply increments each time a pulse arrives, but on a pulsed source the time of arrival of the neutron is also important and so more complicated electronics are needed to store both the time of arrival and the and position of each neutron.

The detector used in these experiments is encased in B<sub>4</sub>C shielding and mounted on a stand to raise it to the correct height. The pre-amplifier is the small metal box attached to the base of the stand.

Three cables are needed to run this particular type detector:

- 1) A co-axial cable to carry the high voltage supply to the gas tube.
- 2) A cable to supply 12V to power the pre-amplifier.
- 3) A small cable to carry the signal.

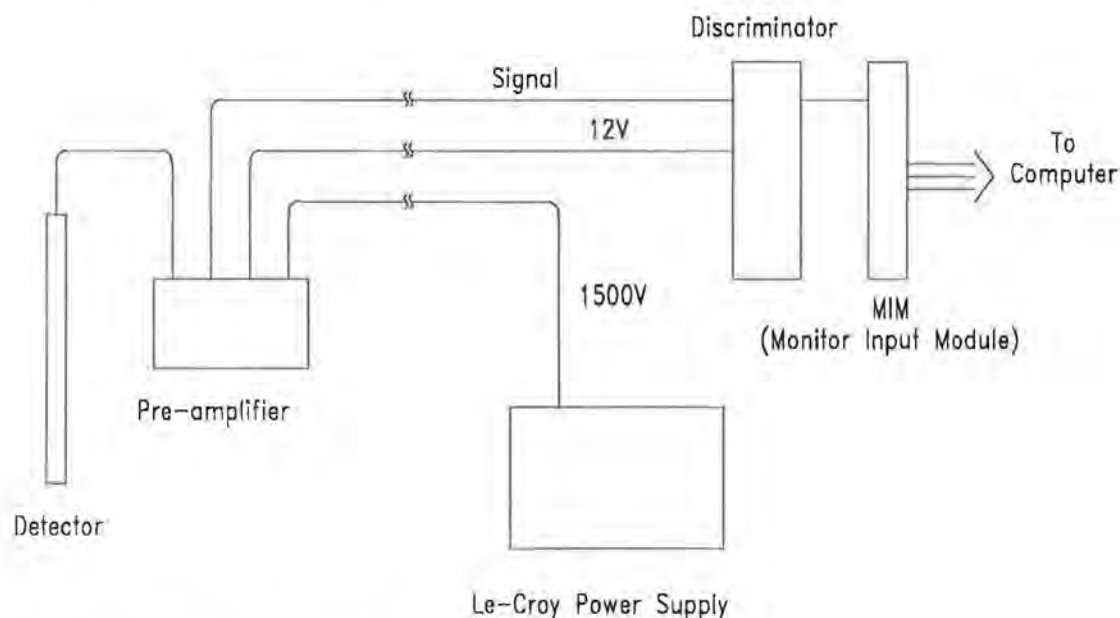


Figure 1 The schematic wiring diagram for a <sup>3</sup>He detector.

The discriminator and the digital electronics are found in the racks on the platform above the spectrometer. At ISIS the discrimination, further amplification and conversion to a square wave is all performed on the same card - which is misleadingly referred to as a discriminator.

The co-axial cable is connected to the back of the power supply. Once powered to 1500V the detector should be operational. The 12V pre-amplifier supply and the signal cable must both be connected to the front of the discriminator. Up to this point the electronics on all spectrometers are essentially the same, but the electronics that now convert the signal into histogrammed time-of-flight data are unique to ISIS.

The output of the discriminator must be connected to an input module; either a MIM (Monitor Input Module), or a DIM (Detector Input Module), This is entry port to a package of electronics known collectively as the DAE (Data Acquisition Electronics).

Setting up the <sup>3</sup>He detector:

1. Set-up the cabling as shown in figure 1 (The cables should all be in place, they should simply require connecting up). Raise the voltage on the detector to approximately 1500V, using the Le-Croy power supply. **Please do not touch the power supply without supervision**. It should now be operational

2. Place the vanadium sample in the beam, bolt the detector stand to the table and start a run. (See Appendix D for a more detailed description of how to start and run an experiment). Once the beam is on it should be possible to see the signal directly from the pre-amplifier on an oscilloscope (N.B. The pulse of the order of  $\mu\text{s}$  wide and a few mV high).
3. The first task is to set the correct amplifier gain in the discriminator. Connect the amplified detector signal (labelled MON) to an oscilloscope (N.B. width  $1\mu\text{s}$ , height 1V). This is controlled by a small potentiometer to be found on the side of the card. Using a small screwdriver to alter the gain to produce a pulse height of approximately 800mV. Check that the digital signal (labelled OUT) is also approximately 800 mV in amplitude. Setting the discriminator levels is vitally important in producing a quiet, gamma-ray insensitive detector.
4. To be able to trigger the digital electronics the square wave pulse must be longer than a microsecond. Like the gain, the length of the pulse is also controlled by a small potentiometer to be found on the side of the card. By looking at the output square wave pulse on the oscilloscope set the width to be approximately  $2\mu\text{s}$ .
5. Connect the output signal to one of the inputs of the Monitor Input Module (MIM).
6. The detector and its electronics chain are now ready. All that remains is to set up the software on the computer so that it knows the address of the detector. The computer uses two different files to locate the address of the given detector:

**WIRING.DAT** This gives the physical address of the detector input in the system crate. There are three components to the address:

- a) The Crate number. This is only important when there is more than one system crate, in this case it is simply 0.
- b) The Module number. This is the slot in which the MIM or DIM is plugged into in the system crate.
- c) The position. This is the number of the input in the MIM or DIM.

**SPECTRA.DAT** This links the detector number to the spectrum number that is used in GENIE.

Copies of these files are kept in `TEB$DISK0:[TRAINER.TABLES]`. Each time the detectors are set up it is necessary to rewrite these files. The directory and name of each of these files must be entered using the `CHANGE` command. Examples of these files are shown below.

**WIRING.DAT**

Number of detectors numbers of monitors

index	detector	time	reg	Crate	Module	Position	Monitor	M.prescale
1	1	1		0	0	0	1	0
2	2	1		0	0	1	1	0
3	3	1		0	0	2	0	0
4	4	1		0	0	3	0	0

**SPECTRA.DAT**

NDET:

DETECTOR	SPECTRUM
1	1
2	2
3	3
4	4

### 1.2. The Zinc Sulphide Detector

The zinc-sulphide detector (Zn-S) is an example of a scintillation device. The ionisation created by the nuclear reaction causes the emission of light. This light is collected and passed to a photomultiplier where it is amplified and converted into an electrical signal. In reality the glass is not pure Zn-S as this will not interact with neutrons but contains a few percent of  $B_2O_3$ . To understand how light is produced in the Zn-S detector it is necessary to invoke solid-state theory. The high velocity decay products from the nuclear reaction excite electrons from the valence band into the conduction band. In certain materials such as Zn-S electrons and holes can bind together to form an exciton which propagates through the lattice until it reaches and impurity atom where it traps and recombines giving out light. In a Zn-S detector the glass is seeded with silver particles that act as trapping centres.

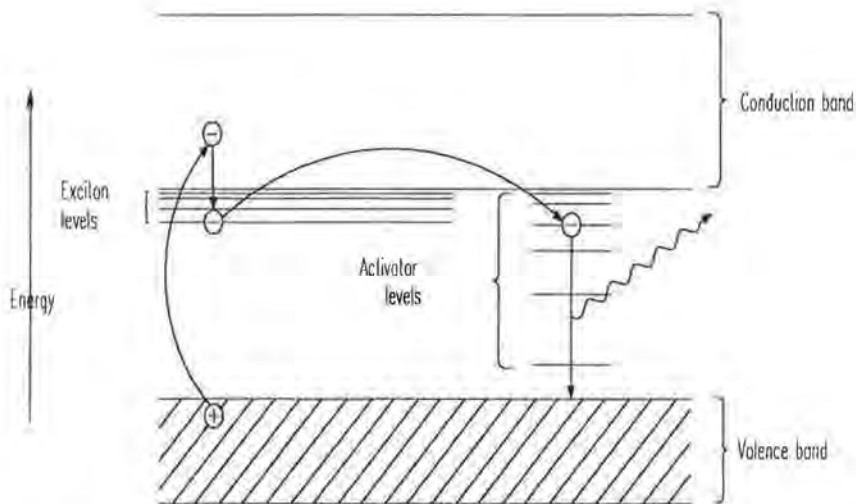


Figure 2. The band structure for an activated Zn-S detectors

Since the activator atoms have levels within the band gap of the glass the light emitted has a wavelength less than the band gap and the glass is transparent to it. Once the light has escaped from the glass it is collected with reflectors or a light guide and transmitted to a photomultiplier which converts the light into an electrical signal and amplifies it by about a factor of  $10^6$ . The amplification means that the pulse produced by the output of the photomultiplier is of the order of 1V and so no pre-amp is required.

The Zinc-sulphide detector used in this course is designed to have an active area similar to that of an  $^3\text{He}$  detector. Unless photomultipliers are cooled they produce noise themselves, because of this photomultipliers are normally used in coincidence. The light from the scintillator is channelled to two photomultipliers simultaneously, and their output is passed through a coincidence unit which only fires if both tubes produce a pulse at the same time. In general scintillator detectors are more sensitive to gamma radiation than gas detectors, but by careful set up of the discrimination and the coupling of the scintillator to the photomultipliers, it possible to produce a detector that has similar sensitivity.

Four cables are needed to set up the Zn-S detector:

- a) Two small co-axial cables to power the photo-multipliers.
- b) Two signal cables.

Zn-S detector set-up:

1. The co-axial cables must be connected to the Le-Croy. **Please do not touch the Le-Croy without supervision.** The two photo-multipliers have been optimised with detector 1 needing 1000 V and detector 2 requiring 1050 V. The two signal cables must be connected to the corresponding inputs in the discriminator. The signal cables must be connected to the amplifier on the platform on the top of the spectrometer. The amplifier is different from that used for the  $^3\text{He}$  detectors, it has three inputs although only two are needed for this detector
2. Place the vanadium sample in the beam, bolt the detector stand to the table and start a run. (*See Appendix D for a more detailed description of how to start and run an experiment*). Once the beam is on it should be possible to see the signal directly from the pre-amplifier on an oscilloscope (N.B. The pulse of the order of  $\mu\text{s}$  wide and a few mV high).
3. Connect up the cables to the discriminator. Check the input and output signals (labelled MON and OUT) for each of the photo-multipliers using the oscilloscope. (The pulse height is approximately 1V and the width is several hundred nano-seconds.)
4. Each individual output could be used straight away, but to reduce noise both are first passed through a coincidence unit. Connect up the outputs from the discriminator to the inputs of the four-fold logic unit and use the pins to switch off those inputs that are not being used. Select a two-fold coincidence using a peg and terminate any output that is not being used with a  $50\Omega$  terminator.
5. The output of the coincidence unit can be connected to the input module directly, but the pulse width is of the order of nano-seconds which might not register in the electronics. To stretch the pulse to the more normal  $1\ \mu\text{s}$ , it is possible to use the Quad discriminator. Remember to terminate the outputs not being used with a  $50\Omega$  terminator.
6. Connect the output from the Quad discriminator to the monitor input module (MIM) and adjust the WIRING.DAT and the SPECTRA.DAT.

## 2. Transmission-Related Properties of Materials

This section of the course involves neutron-related properties of materials, mostly relating to neutron transmission. Firstly the effect of resonances is shown and this is used to produce the all-important length scale calibration. The incoherent nature of vanadium scattering is introduced and is used to demonstrate the flux shape of the source, explaining its general features. An introduction is given to the transmission and shielding properties of important neutron scattering materials, and the transition from free to bound scattering cross-section is demonstrated.

### 2.1. Neutron Resonances and Calibration of Instrument Length

For the first part of this section of the course you are to observe the transmission of a resonant foil of tantalum. This will enable you to calibrate the total neutron flight path from moderator to detector which is essential for putting experimental results on a wavelength (or energy or d-spacing or...) scale. This is to be done by placing the gas detector at a scattering angle somewhere in the range 30° to 70° say, to observe scattering from the vanadium rod which should be placed in position 3 of the Test Beamline. Since you are going to calibrate the position you should ensure that the detector is carefully positioned and then not moved! You should use the extendable rule to measure a reasonably accurate value for the distance ( $L_2$ ) from sample position to the detector. The tantalum foil should be placed over the incoming neutron beam. It will be adequate to just fasten it to the inside of the tank by use of adhesive tape, but ensure that the foil covers the beam completely. You will also find it helpful to fix the second gas detector at a high scattering angle, say 150°, for later use in section 3 of the course.

With this setup do a run. You will find that at low times (a few hundred micro-seconds) there are 'bites' taken out of the spectrum recorded by the detector. These 'bites' are due to resonant absorption of neutrons by the foil; for certain incident neutron energies there is a very large cross-section for absorption of the incident neutrons by the nuclei of the foil. Of course the origins of this effect lie in the realm of nuclear physics, but it is of considerable use to us for calibrating the length scale of our neutron scattering instruments.

For the purposes of calibration we need to choose resonances which are well separated from other resonances. According to the literature (Mughabghab, 1984) the lowest energy a resonances of tantalum are at neutron energies of  $4.28 \pm 0.01 \text{eV}$ ,  $10.36 \pm 0.05 \text{eV}$ ,  $13.95 \text{eV}$ ,  $20.29 \text{eV}$ ,  $22.72 \text{eV}$ . Using the nominal value for the incident neutron flight path  $L_1$  from moderator to sample position given in appendix C (description of the test beamline), firstly identify these particular resonances in your data. Then measure the time-of-flight at which each resonance occurs as accurately as you can (either use the cursor in GENIE or the GENIE PEAK command to fit a Gaussian) and use these peak positions to calculate an accurate value for the total flight path ( $L_1 + L_2$ ) from moderator to detector. This may be done by a simple straight line plot, but you need to be aware that there is a  $t_0$  error in the timing due to time delay in the electronics.

(An alternative to tantalum is to use a foil of hafnium. This has three well-separated resonances at neutron energies of  $1.098 \pm 0.020 \text{eV}$ ,  $2.388 \pm 0.002 \text{eV}$  and  $6.60 \pm 0.02 \text{eV}$ . However, there are other resonances present as well so if you use the hafnium foil you should check your identification of the resonances with a course tutor, or you may get a bad calibration.)

For an absorbing material at neutron energies away from a resonance the absorption cross-section has the following dependence on neutron wavelength  $\lambda$ ;

$$\sigma_{abs}(\lambda) = \sigma_0 \times \lambda$$

Such a cross-section is proportional to  $1/v$  where  $v$  is the neutron speed.

## 2.2. Flux Shape

Now remove the resonant foil from the beam and do another run using the same gas detector to look at the neutron spectrum scattered by the vanadium rod. You will see that the spectrum from vanadium is essentially a smoothly varying function. The reason for this is that the scattering from vanadium is almost entirely incoherent. (The coherent scattering from vanadium is very small, but is not actually zero, and if you examine the vanadium spectrum very carefully you may just be able to discern very small Bragg peaks.) The fact that vanadium is to a good approximation a totally incoherent scatterer is a property which is widely used in neutron scattering. In fact we have already made use of this property in looking at the resonances from the resonant foil. Now we make use of the incoherence of the scattering from vanadium to look at the flux shape produced by the liquid methane moderator.

The term flux shape  $\Phi(E)$  is used to mean the number of neutrons in the beam per unit energy ie. the sample receives a flux of neutrons  $\Phi(E)dE$  with energy in the range  $(E, E+dE)$ . The flux shape from the source divides into two regions. At longer times (lower energies) the neutrons emerge from the moderator in thermal equilibrium with the moderator and in this range the flux shape can be described by a Maxwellian energy distribution. However, the higher energy neutrons which emerge earlier from the moderator have not suffered enough collisions in the moderator to achieve thermal equilibrium and conventionally these neutrons are termed epithermal neutrons.

Use your length calibration to convert your vanadium spectrum to an energy scale. The workspace containing this spectrum must be corrected for the energy-dependence of the efficiency of the gas detector and this may be done in GENIE by the command @TG:GASBYE. The Maxwellian region of the flux shape is given by;

$$\phi_{max}(E) = J \frac{E}{T^2} \exp\left(-\frac{E}{k_B T}\right)$$

where  $J$  is a constant and  $T$  is a characteristic temperature. Calculate the energy at which this function is a maximum and hence calculate the characteristic temperature. The temperature of the liquid methane moderator which is viewed by TEB is actually in the range 107-109K. Thus you will see that the value of  $T$  used in the equation is an effective temperature and is not the true temperature of the moderator. A full calculation of the flux shape from a moderator requires the use of large Monte Carlo codes (Taylor, 1984). However, the approach given here which is very widely used is basically a semi-empirical means of characterising the flux shape of a pulsed neutron source. This approach is very useful, for example in describing the peak shape seen in pulsed neutron powder diffraction.

In order to characterise the epithermal region of the flux shape use the GENIE transform TP:LOG\_LOG to take natural logs of both  $x$  and  $y$ . What is the general form of the flux shape  $\phi_{epi}(E)$  in the epithermal region? Use the REBIN command to remove irrelevant non-epithermal parts of the spectrum and then use the GENIE function TP:FIT\_LINE to characterise this behaviour numerically. When a generalised function is required for describing the flux shape over its whole energy range this is normally done by using a suitable joining function to combine the Maxwellian expression  $\phi_{max}(E)$  and the epithermal expression  $\phi_{epi}(E)$ .

## 2.3. Transmissions of Shielding Materials

Practically speaking the essence of a transmission measurement is to record the spectrum with the sample of interest in the beam and again with the sample removed. The transmission is then simply the ratio of the sample-in spectrum to the sample-out spectrum (both spectra having been normalised for length of run). If the sample has uniform thickness across the beam (a flat plate sample) then;

$$T = \exp(-\rho\sigma x)$$

Thus for a flat plate sample it is in principle a simple matter to extract the cross-section from a transmission measurement. In the case of a sample of non-uniform thickness across the beam (eg a cylinder) the transmission must be expressed as an integral of  $\exp(-\rho\sigma x)/A$  over the area  $A$  of the beam (assuming the beam to be uniform), with  $x$  varying across the beam. Hence for samples of non-uniform thickness it is no longer a simple matter to extract the sample's cross-section  $\sigma$  from the measured transmission. It is thus important for a transmission measurement to use a flat plate sample which covers the whole of the beam.

A useful consistent set of units is  $\rho$  in atoms  $\text{\AA}^{-3}$ ,  $\sigma$  in barns ( $1 \text{ barn} = 10^{-28} \text{ m}^2$ ),  $x$  in cm. Convince yourself that these are consistent units for calculating transmissions.

With the vanadium rod still in position 3, place a thick piece of cadmium over the incoming neutron beam and thus measure its transmission.

**Cadmium is toxic which means that whilst it may be handled you should always wash your hands afterwards, especially before handling food.**

{GENIE tips for transmission data:

When you have divided one workspace by another to obtain a ratio, you should not change the units of the result without first using the command SET YCODE Wn -1 on the workspace containing the ratio. (See Appendix A3, units conversion in GENIE.)

You may find it more straightforward to change the units of each of the two input workspaces separately, before one is divided by the other to yield the ratio.

The default x-axis limits selected by GENIE are not always very suitable when dealing with data on an energy scale so you will find it helpful to specify the x-limits when displaying a transmission versus energy curve in GENIE - you should be able to work out suitable values for the x-limits!!)

In order to normalise the sample-in and sample-out spectra to the length of the run you will need to use the INTEGRATE command in GENIE to integrate the incident monitor spectrum. You will find that to a good approximation the transmission of cadmium is either one or zero. Use your length scale calibration to determine the cut-off energy for cadmium. Whereas reactor neutron sources have negligible numbers of fast neutrons (ie. high energy neutrons), a spallation source produces enough fast epithermal neutrons to use in an experiment. Clearly an absorber such as cadmium is not a suitable shielding material for experiments using these high energy neutrons, but cadmium is excellent for the majority of reactor experiments (although not for instruments using ILL's hot source).

Now measure the transmission of the provided piece of 'crispy mix'. This black material is made from powdered  $\text{B}_4\text{C}$ , bound together with a small amount of resin (the reason for the name 'crispy mix' should be obvious!). As you will observe, this composite material is comparable to cadmium for stopping slow neutrons, but is much more effective for stopping high energy neutrons. The actual absorption of neutrons occurs as a result of the high absorption cross-section of the  $^{10}\text{B}$  isotope. This is a  $1/v$  cross-section and hence its effectiveness for absorbing neutrons falls off at higher neutron energies, as with cadmium. However, the high energy effectiveness of crispy mix is improved by the presence of light atoms (boron, carbon, hydrogen) which as a result of their scattering cross-sections have a moderating effect on the neutrons and slow them down so that they are more likely to be absorbed.

#### 2.4. *Transmission of Beryllium Filter*

Now place the Harwell beryllium filter over the incoming neutron beam in order to measure its transmission. We suggest using the lab-jack and pieces of polythene to position the filter in the beam as carefully as possible. This beryllium filter is composed of alternating layers of polycrystalline



beryllium and cadmium. Beryllium is potentially very toxic, but there should be no hazard since the filter is sealed. The filter should be placed in the beam so that the neutron direction is in the plane of the beryllium/cadmium layers, preferably with these layers vertical.

Calculate the transmission of the filter as a function of energy by use of your length scale calibration. The filter has a high transmission for neutrons of very low energy, but a low transmission for neutrons of a higher energy. What is the cut-off energy for this type of filter? {GENIE tip: you will need to specify a suitably low pair of x-limits when displaying the transmission of the Be filter in order to see the edge(s).} The beryllium filter will form the basis of a later part of the course where an indirect geometry (fixed final neutron energy  $E_f$ ) inelastic spectrometer is constructed. This spectrometer has the final neutron energy  $E_f$  fixed by the filter, so you will need the value you have obtained for the cut-off energy of the filter.

The sharp edge in the transmission of the beryllium filter is a Bragg edge; it is due to the Bragg cut-off which occurs when, as the neutron wavelength changes, the scattering angle required to observe a particular Bragg peak passes  $180^\circ$ . From Bragg's law it is clear that a Bragg peak with d-spacing  $d$  gives rise to a Bragg edge at a neutron wavelength  $\lambda=2d$ . If you examine your measured transmission carefully you may be able to observe a second smaller Bragg edge. If you can see this edge then...

Determine the cut-off energy for the second edge as well. Beryllium is hexagonal and the general formula for the d-spacing  $d_{hkl}$  of the  $(hkl)$  reflection of a hexagonal crystal with lattice parameters  $a$  and  $c$  is;

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

Given that the Bragg peak of the highest d-spacing is the (100) reflection and that the second highest d-spacing comes from the (002) reflection you should be able to determine the lattice parameters of beryllium.

The purpose of the cadmium in the beryllium filter should now be clear - it is to absorb the scattered neutrons and avoid them being scattered a second time back into the forward direction. You will have observed that the transmission of the beryllium filter is less than one for neutrons of energy less than the cut-off energy. For these neutrons Bragg scattering cannot occur and the reason why the transmission is still less than one is that these neutrons can still be absorbed, scattered incoherently or scattered inelastically. In fact the absorption and incoherent cross-sections of beryllium are both very low and thus the low energy transmission of the filter can be made to closer to a value of one by cooling it so as to remove the energy gain scattering from thermally excited phonons.

### 2.5. Free and Bound Cross-Sections

Measure the transmission of one of the provided sheets of polythene. Given that the chemical formula of polythene is  $(CH_2)_n$  and that the density of the polythene provided is  $0.908\text{gcm}^{-3}$ , extract from your measurement of the transmission the absolute cross-section  $\sigma(\lambda)$  as a function of wavelength  $\lambda$  (use the provided GENIE function TP:LOGE).

{GENIE tip: in this case the most reliable way to extract the transmission as a function of wavelength  $\lambda$  is probably as follows: 1) put the two workspaces on a  $\lambda$  scale, 2) use the REBIN command to put the data on a  $\lambda$  scale with a constant spacing in  $\lambda$  (say  $0.1\text{\AA}$ ), 3) finally take the ratio of the two workspaces.}

The relationship between the free (ie. low neutron wavelength) and the bound (ie. high neutron wavelength) scattering cross-sections for an element with atomic weight  $A$  is;

$$\sigma_{free} = \sigma_{bound} \left( \frac{A}{A+1} \right)^2$$

This effect is obviously largest for hydrogen which is why we have chosen polythene to demonstrate it. Use the provided copy of Sears' cross-section tables (Sears, 1992) to calculate both the free and bound scattering cross-sections for polythene. You should obtain reasonable agreement with your measurement. Note that the transition between the free and bound cross-sections of a hydrogenous material is actually quite similar in general form to the proportionality to wavelength shown by the absorption cross-section away from a resonance (see the first section of this part of the course). In the case of the scattering cross-section, the precise form of the transition between the free and bound values is governed by a double integral of the scattering function  $S(Q, \omega)$  over the physically accessible range of  $Q$  and  $\omega$ , since;

$$\frac{d^2\sigma}{d\Omega d\omega} = \frac{k_1}{k_0} \frac{\sigma}{4\pi} S(Q, \omega)$$

Thus the form of the transition between the free and bound scattering cross-sections depends upon the details of the structure and dynamics of the particular sample.

### 2.6. Demonstration of Recoil Scattering

Thus far it has been assumed that the neutrons scattered by the vanadium at position 3 have been scattered elastically with no change in neutron energy. Whilst true inelastic scattering may be neglected for this type of measurement, there is another effect known as recoil scattering which cannot be neglected. In order to demonstrate this effect you should repeat the original measurement with the resonant foil at position 2, only this time replace the vanadium rod with the provided perspex rod. You will find that the resonances have moved in time-of-flight. The reason for this is the effect known as recoil scattering. When the neutron arrives with very high energy (in these terms the energy of the resonances is high) then it scatters off a nucleus in the sample as though it is a free particle. Hence consider a particle of mass  $m$  and energy  $E_i$  (the neutron) colliding with a stationary particle of mass  $M$  (the nucleus) and use energy and momentum conservation to determine the relationship between the neutron's final energy  $E_f$  and the angle  $2\theta$  by which it is scattered. Show that for  $M=m$  (as for scattering off hydrogen - perspex is mostly hydrogen) this reduces to;

$$E_f = E_i \cos^2(2\theta)$$

In fact the use of a resonant foil in the incident beam has enabled us to construct a simple type of direct geometry (ie.  $E_i$  is fixed) inelastic spectrometer.

Backscattering (ie.  $2\theta > 90^\circ$ ) is impossible for hydrogen. You should be able to explain this.

From your first equation it should be clear that the recoil effect becomes smaller as the mass  $M$  of the nucleus involved increases above the neutron mass  $m$ . Hence assume that the effect is negligible for vanadium (atomic mass=51amu) and determine from the shifted position of the lowest energy resonance the value of  $E_f$  for the perspex. This value should agree roughly with that predicted by the equation for  $M=m$ , although agreement will not be exact due to ignoring recoil for the vanadium.

Calculate for vanadium the value of  $E_f$  for each of the resonances used in the calibration and hence calculate a corrected value for  $L_2$  taking into account recoil scattering by the vanadium.

### 3. Diffraction

#### 3.1. Making A Powder Diffraction Measurement

On a time-of-flight source doing a diffraction experiment requires little more than a sample and a detector. Even with the simple equipment available on TEB it should be possible to take excellent quality diffraction patterns if a little care is taken on the set-up of the detector and in performing the measurement.

In order to obtain a fully corrected diffraction pattern it is necessary to do four separate measurements:

- a) A run on the sample in a can.
- b) A run on the empty can.
- c) A run on a vanadium rod.
- d) A run on the empty instrument.

The empty instrument run enables the vanadium run and the sample+can run to be corrected for sample-independent background. The run on the empty can must be subtracted from the sample+can run using factors for attenuation and for multiple scattering. The vanadium run must also be corrected for attenuation and multiple scattering. The corrected sample data are then divided by the corrected vanadium data to remove the flux shape from the diffraction pattern.

However, for our current purposes we will dispense with the empty can and empty instrument runs since applying these corrections is time-consuming in terms of both experimental time and experimenter's effort, and does not actually help to illustrate the points we wish to convey in this exercise.

Place one detector at a high angle, say  $150^\circ$ , at position 3 and bolt it down. Similarly place a second detector at a lower angle, say  $40^\circ$ . With these angles do runs on first the nickel powder sample and then the vanadium rod. The final diffraction pattern will need to be of good statistical quality so that individual Bragg peaks can be fitted, so the runs on both nickel and on vanadium should be fairly long (we suggest for  $100\mu\text{amp-hours}$ , but you should examine the spectra critically in GENIE to decide for yourself).

Whilst the nickel powder run is in progress you can start to study the powder diffraction pattern of nickel. Firstly note the detailed shape of the powder peaks as seen at higher scattering angle - they are asymmetric (they have a sharp leading edge and a trailing edge which falls away more slowly) and this is a particular feature of neutron diffraction on a pulsed source.

#### 3.2. Indexing of Powder Diffraction Data, and Angle Calibration

The condition for a Bragg peak to be observed is of course given by Bragg's law;

$$\lambda = 2d \sin \theta$$

where  $\lambda$  is the wavelength of the scattered wave,  $d$  is the d-spacing of the reflection and  $2\theta$  is the scattering angle. Whilst the nickel run is in progress convert the data to d-spacing using the value for scattering angle  $2\theta$  marked on the detector table at position 3. Use the cursor command in GENIE to read off approximate positions of the first few (at least half a dozen or so) peaks, starting at highest d-spacing. You can now use these peak positions to index the pattern. For a cubic crystal the interplanar d-spacings are given by;

$$d = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}}$$

where  $h$ ,  $k$  and  $\ell$  are integers (0,1,2,3,...), and  $a$  is the lattice parameter. It follows from this that taking the square of the ratio of the d-spacing of two Bragg peaks will yield the ratio of two integers. Using the highest d-spacing Bragg peak as a reference peak, apply this operation to your measured Bragg peak positions and hence identify  $(hkl)$  for the first few peaks of highest d-spacing (nickel is cubic). Hence determine an approximate value of the nickel lattice parameter  $a$ . From the selection rules for systematic absences given in Appendix E, determine the structure type of nickel. This procedure is known as indexing and it can be extremely useful, for example in identifying unknown peaks in an experimental diffraction pattern, or alternatively as a first stage in determining the structure of a material.

Use the formulae given in Appendix B to relate neutron wavelength  $\lambda$  to time-of-flight  $t$  and flight path  $L$ . An important feature of the time-of-flight technique is that the wavelength of a neutron is proportional to its time-of-flight over a given flight path. Now use Bragg's law to obtain a relation between d-spacing and time-of-flight. You will find that d-spacing is also proportional to time-of-flight, but it is also important to note that the constant of proportionality depends on the product  $L \sin \theta$ , rather than on the flight path  $L$  alone. The Bragg peaks for a crystal of known lattice parameter may thus be used to calibrate  $L \sin \theta$  for a diffractometer, but cannot give a value for the total flight path  $L$ . In order to perform a complete calibration of a diffractometer it is necessary to obtain an independent value for  $L$ , for example by the use of a resonant foil as you have already done under section 2.1, and then a separate value for  $2\theta$  can be obtained.

Determine as accurately as you can the time-of-flight for one of the nickel Bragg peaks which you have identified. Given that the lattice parameter of nickel at 20°C is  $a=3.52387 \pm 0.00008 \text{ \AA}$ , calculate an experimental value for  $L \sin \theta$ . If the detector has not been moved since the flight path calibration run performed under section 2.1 then you can determine the flight path  $L$  for this detector and then get an accurate value for the scattering angle  $2\theta$  which may be compared with the nominal value marked on the detector table.

Measure the powder patterns of the two 'mystery' samples for long enough to identify the Bragg peak positions. Given that these samples are either some type of cubic or hexagonal, use the information in Appendix E to identify their structure type and calculate their lattice parameters. Given that these samples are elements use the information from Appendix F to identify these samples.

The intensity of the  $(hkl)$  reflection is proportional to the square modulus of the structure factor  $F_{hkl}$  defined by

$$F_{hkl} = \sum_j \bar{b}_j \exp(2\pi i(hx_j + ky_j + lz_j)) \exp(-B_j s^2)$$

where the  $j$  summations are over the atoms in the cell.  $(x_j, y_j, z_j)$  is the position of the  $j^{\text{th}}$  atom in unit cell coordinates (ie. in fractions of the lattice parameters),  $B_j$  is the isotropic temperature factor for the  $j^{\text{th}}$  atom and  $s = \sin \theta / \lambda$  for the  $hkl$  reflection. Make sure that you understand how the selection rules for systematic absences arise from the expression for the structure factor.

### 3.3. Powder Diffractometer Resolution and Powder Peak Shape

The shape of a Bragg peak measured on a time-of-flight diffractometer (and hence the resolution of the diffractometer) is due to a number of factors which can be divided into two types:

#### 1. Time

- *Histogramming.* The fact that the data are collected in histogram form has a broadening effect. However, provided the histogram bins are sufficiently narrow that an individual Bragg peak is covered by a reasonable number of bins (say 10 as a rule of thumb) this effect will not be significant.
- *Moderation time.* In converting the data to a d-spacing scale (for example) the data are treated as though all neutrons leave the source at the same instant in time. However, in practice the neutrons leave the source over a range of times due mainly to the moderation process. Hence, whereas it is assumed that neutrons arriving at the detector at a particular time have a precise velocity according to  $v=L/t$ , in reality they have a range of velocities in the region of this value.

#### 2. Geometry

- *Angle uncertainty.* The moderator, sample and detector all have finite sizes with the result that there is a spread in scattering angles  $2\theta$  which can result in a neutron being detected in the detector.
- *Flight path uncertainty.* Due to the finite sizes of the moderator, sample and detector there is a spread in neutron flight paths  $L$  from source to detector.

Generally the angle uncertainty gives the dominant contribution to the resolution of a practical diffractometer. Differentiate Bragg's law with respect to  $\theta$  (with constant  $\lambda$ ) to obtain a relation between the angular uncertainty  $\Delta\theta$  and the resultant d-spacing resolution width  $\Delta d$ . The resolution for a time-of-flight diffractometer is normally expressed in the form  $\Delta d/d = \dots$  since this is independent of  $d$  for a given detector. Show that  $\Delta d/d = \Delta t/t = \Delta Q/Q$ . How does the resolution vary with scattering angle  $2\theta$ ? For a fixed set of flight paths  $L_1$  and  $L_2$ , what two steps can be taken to minimise the resolution  $\Delta d/d$ ?

Estimate the angular width  $\Delta\theta$  arising from the finite detector width for your experimental setup. Using your formula calculate the d-spacing width in the form  $\Delta d/d$  for the two angles you have used to measure the powder diffraction pattern of nickel. Compare this width with what you measure experimentally.

The shape of a Bragg peak measured on a time-of-flight powder diffractometer is well described by the convolution of a Gaussian and an exponential decay. The peak broadening due to geometry is modelled by the Gaussian, whilst the exponential contribution comes from the shape of the pulse from the moderator. For your high scattering angle nickel spectrum you should fit this function to each of the non-overlapping peaks using the GEC function in GENIE. To do this you should put the spectrum into a workspace, display the workspace, and then use

PEAK TG:GEC  $\langle t_{min} \rangle$   $\langle t_{max} \rangle$

where  $\langle t_{min} \rangle$  and  $\langle t_{max} \rangle$  represent the minimum and maximum x-limits of the data to be used in the fit. If the fitting is successful then you will have two width parameters for each peak:  $\sigma$  the Gaussian width (standard deviation) and  $\tau$  the constant in the exponential decay.  $\tau$  is actually the mean time spent in the moderator by a neutron. Record these values along with the position of the peak. You should perform the fit for as many Bragg peaks as you can, being sure to have data over as wide a range of time-of-flight as achievable (be sure to have results extending to both sides of the Maxwellian peak in the flux shape). Eventually you will not be able to fit the peaks at the low time-of-flight end of the spectrum since they will start to overlap and also become less intense. With the large dynamic range available on a pulsed source it is inevitable that there will be overlapping peaks at the low time-of-flight (ie. low d-spacing) end of the spectrum. The experimental situation which can be

achieved within the scope of this course is somewhat limited in terms of count rate and counting time, and thus you will need to be quite careful to get good fits over as wide a range of time-of-flight as possible, and even then the results may tend to somewhat noisy. However, with care they should be good enough to illustrate the points which we wish to convey here.

For each fitted peak you can calculate the full width at half maximum (FWHM)  $\Delta t$  by running a second program. This program is a piece of FORTRAN code and must be run outside of GENIE. It can be invoked by typing

RUN TP:FWCALC

at the VMS prompt. Find  $\Delta t$  for each of the peaks. (Give a value of 0.5 for the fractional height, since it is the full width at half maximum which is to be calculated.)

Plot  $\Delta t/t$ ,  $\sigma$ ,  $\tau$ ,  $\sigma/t$  and  $v/t$  versus time-of-flight  $t$ . You should find that...

- The powder diffractometer resolution  $\Delta t/t$  ( $=\Delta d/d$ ) tends to increase somewhat with increasing time-of-flight  $t$ , with a slight maximum at the Maxwellian.
- The mean time spent in the moderator  $\tau$  increases steadily with increasing  $t$ , starting from a very low value. This is reasonable since neutrons detected at a larger time-of-flight have a lower energy and must on average have spent a longer time in the moderator to achieve this lower energy. At high time-of-flight  $\tau$  tends to a constant value. What is this value for the methane moderator from which the test beam obtains its neutrons? In fact this time contribution may be minimised by using very long flight paths (as in the High Resolution Powder Diffractometer at ISIS); since the time uncertainty  $\Delta t$  is a constant for a particular neutron wavelength the effect of increasing the flight path (and hence the time-of-flight  $t$ ) is to reduce  $\Delta t/t$ .
- The geometrical contribution to the resolution width  $\sigma/t$  is essentially constant, independent of  $t$ . (ie. the  $\sigma$  versus  $t$  curve is a straight line through the origin.) What is this constant value of  $\sigma/t$ ? The FWHM of a Gaussian is given by  $\sqrt{8 \log_e 2} \times \sigma = 2.355\sigma$  so you should multiply the constant value for  $\sigma/t$  by 2.355 to obtain the geometric contribution to the FWHM for the resolution function. Using the relation obtained earlier by differentiating Bragg's law, calculate the value of  $\Delta\theta$  which this corresponds to. Supposing that this  $\Delta\theta$  were due only to detector width  $w$ , what value of  $w$  does your  $\Delta\theta$  value correspond to? Compare this with the physical width of the detector. On your plot of  $\Delta t/t$  versus  $t$  draw a horizontal line corresponding to the geometric contribution to the FWHM for the resolution function, showing the relative magnitudes of the two contributions to  $\Delta t/t$  as a function of  $t$ .

### 3.4. Profile Refinement

It is possible to analyse powder diffraction data by relating the integrated peak intensities to the structure factors  $F_{hkl}$  as defined above. However, there is a large amount of information available in a powder diffraction pattern, and the best way to retrieve this information is to do a least-squares fit to the whole data set, known as a profile refinement. This has the advantage that it can get information from the positions, widths and intensities of all the peaks in the spectrum including those that are so close together as to be overlapping. With the large dynamic range available on a pulsed source it is inevitable that the data will have overlapping peaks at the low d-spacing end of the spectrum. Profile refinement is a very powerful technique, which can yield a large amount of physical information about a polycrystalline sample, and it would require a separate course to explain fully how to perform a profile refinement. Within the scope of the present course it is only possible to give an introductory demonstration of the technique.

- *Prepare experimental data file*

Normalise your high angle nickel powder diffraction pattern in GENIE by dividing it by the vanadium spectrum. If you examine the resultant normalised data it should be apparent to you that the flux shape has been normalised out, leaving an essentially flat background level between the Bragg peaks. Keeping the result on a time-of-flight x-axis (the ISIS profile refinement programs are written to work on data on a time-of-flight x-axis) you may produce a file suitable for input to the profile refinement program by typing

```
TR Wn TP:PROLSFILE Wn
```

in GENIE, where the normalised powder pattern is in workspace  $Wn$ . You will need to choose a name (*name*) for the output file of 6 characters or less which will be used to identify all files associated with the profile refinement. The experimental data file will then be called *name*.DAT. You will also need to specify the range in time-of-flight over which the data are to be output; you should specify the full range covering all the discernible Bragg peaks.

- *Prepare crystal data file*

The profile refinement program will also require an input file of extension .CCL which contains crystallographic information about your sample and instructions for performing the least squares fit. To obtain this file type (not in GENIE):

```
COPY TR:NIPOWD.CCL name.CCL
```

The .CCL file will have to be edited to contain the calibration information appropriate to your data:

- The value after L THE2 must be changed to be your own value of  $2\theta$ .
- The value after L PKCN must be changed to be your own total flight path value  $L_1+L_2$  (from section 2.1).
- The value after L ZERO must be changed to be your own  $t_0$  timing error (from section 2.1).

- *Perform refinement*

A batch job to perform the profile refinement may then be submitted by use of the command (not in GENIE) :

```
REFINE name queue
```

where *queue* is one of the following CRISP, HRPD, POL, SXD, LOQ, MARI - this specifies which computer will be used for the batch job. The refinement will produce a number of output files:

- *name*.OUT, *name*.LOG : Information about the refinement produced whilst the job is running. The .OUT file cannot be viewed until the batch job has completed, but the .LOG file can always be viewed by use of the command: TYPE *name*.LOG .
- *name*.HKL : The *hkl* indices of the reflections which lie within the range of time-of-flight to be fitted - produced only the first time the refinement is run.
- *name*.CCN : If the refinement has been successful then this file contains the updated values of the fitted parameters and may be used as the input crystal data file for subsequent refinements (in which case it must be renamed to *name*.CCL).
- *name*.PRO : This ASCII file contains the measured profile, the calculated profile and their difference.

- **Examining the profile fit**

Once the batch job has completed you may examine the results by typing (not in GENIE) :

```
PICTIC      name
```

This will generate several files which can be examined in GENIE named *name*.PIC, *name*.TIC and *name*.TAG. In GENIE the results can be examined by typing PLS which will display the experimental powder profile, the fitted profile and the residual. Once the data have been read in to GENIE by PLS they may be displayed over a different range in time-of-flight by use of the command PLC. Tick marks indicating the positions of the Bragg peaks may be added to the plot by use of the command TIC *ymin ymax* where *ymin* and *ymax* are the lower and upper ends of the tick marks.

The file TR:NIPOWD has been set up so that it should give a good fit to nickel data taken on Test Beam. However, if your refinement job fails or if the fit is not good you should first check the *name*.LOG and *name*.OUT files for helpful messages. If the fit is not good you should check whether you have correctly entered the right calibration values in the .CCL file. In practice a certain amount of skill and experience can be required to make the profile refinement run successfully so ask a course tutor for guidance if you have problems.

The general approach used in profile refinement is to start by only allowing a small number of simple variables (eg. scale factor) to be varied, and as the fit improves to gradually allow more variables to be varied in the refinement. The file TR:NIPOWD.CCL has been setup to vary the scale factor, the background parameters and the lattice parameter, which will allow for variations in experimental conditions between different runs on nickel. Other variables have been given values likely to lead to a good fit. There are separate texts and manuals on the profile refinement codes used at ISIS (David et al., 1992) and these should be consulted for further more detailed information on the subject.



### 3.5. Non-Crystalline Diffraction

Do a run on the SiO<sub>2</sub> glass rod with the detector at a scattering angle in the range 60 to 90°. For this measurement normalisation will be done using the run on the vanadium which you have already done with the detector at backward angle and so you must keep the detector at the same secondary flight path L<sub>2</sub> for the measurement on SiO<sub>2</sub>. This run will require fairly good statistics so it may be suitable for an overnight run.

Non-crystalline materials (gases, liquids or glasses) do not have a periodic atomic structure with the result that Bragg peaks are not observed in the diffraction pattern. Instead the diffraction pattern is a much smoother function. For such measurements it is conventional to plot the data against

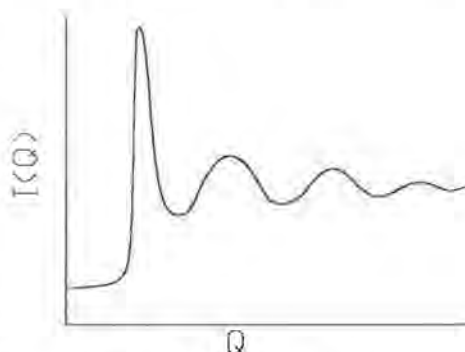


Figure 3 A typical diffraction pattern for a non-crystalline sample

momentum transfer  $Q$  rather than against  $d$ -spacing and the general form of the diffraction pattern  $I(Q)$  after normalisation and correction is typically as shown in the figure.

The approach used in the analysis of such data is very different to that used for analysing powder diffraction data from crystals. Firstly a constant is subtracted from  $I(Q)$  so that the result  $i(Q)$  oscillates about zero. A Fourier transform of  $i(Q)$  is then calculated according to;

$$D(r) = \frac{2}{\pi} \int_0^{\infty} M(Q) Q i(Q) \sin(rQ) dQ$$

The result of the Fourier transform  $D(r)$  gives information about the distances between atoms. A peak in  $D(r)$  indicates a distance which occurs frequently between atoms whilst a trough in  $D(r)$  indicates a distance which occurs infrequently or not at all.

In order to apply this approach to your SiO<sub>2</sub> glass data you should first normalise to vanadium and convert your data to a  $Q$ -scale. Then use the REBIN command so that the result is equally space in  $Q$  with a spacing of  $0.05\text{\AA}^{-1}$  and only covers the range in  $Q$  over which it has an overall appearance roughly like that in the figure. You will almost certainly find that at the low  $Q$  end your data become unreliable due to background and attenuation effects and your REBIN should delete this region. A reasonable value for the maximum  $Q$  might be  $20\text{\AA}^{-1}$ . The next stage is to determine a constant to subtract from your diffraction pattern so that the result  $i(Q)$  oscillates about zero. Once you have done this you can Fourier transform your data in GENIE by typing @TG:12D When using this program we recommend using a high value for the maximum  $Q$  and using  $10\text{\AA}$  and  $0.05\text{\AA}$  as values for  $r_{max}$  and  $\Delta r$ . The first peak in your  $D(r)$  will be at a physically unreasonably low value of order  $0.5\text{\AA}$ ; this peak is an error peak due to effects in the experimental data such as attenuation. The next two peaks arise from Si-O correlations and from O-O correlations. What is the Si-O distance, and the O-O distance? What is the O-Si-O bond angle? SiO<sub>2</sub> glass is generally believed to consist of a continuous random network of corner-sharing SiO<sub>4/2</sub> tetrahedra. The angle subtended at the centre of a tetrahedron by two vertices is  $109.47^\circ$  - compare your experimental result with this value.

## 4. Single Crystal Neutron Diffraction

This experiment should give the student experience in the Laue (stationary crystal, white beam) method for obtaining integrated intensities from a single crystal sample. The chosen material is manganous fluoride,  $\text{MnF}_2$  which is antiferromagnetic below its Néel temperature of 67 K. Measurements at ambient temperature are to be compared with similar measurements made at 20 K to determine the magnetic configurational symmetry, the direction of the  $\text{Mn}^{2+}$  moment and its magnitude. The effect of extinction may also be observed and its magnitude estimated.

1. Manganous fluoride is tetragonal with unit cell dimensions  $a=4.8736\text{\AA}$  and  $c=3.3101\text{\AA}$ . (A tetragonal lattice has parameters  $a=b\neq c$  and  $\alpha=\beta=\gamma=90^\circ$ .) The space group is  $P4_2/mnm$ . The unit cell contents are:

$$2 \text{ Mn at } 0, 0, 0 \text{ and } \frac{1}{2}, \frac{1}{2}, \frac{1}{2}$$

$$4 \text{ F at } \pm(\frac{1}{2}+x, \frac{1}{2}-x, \frac{1}{2}) \text{ and at } \pm(x, x, 0) \text{ with } x=0.3050$$

The sample has dimensions 8 x 5 x 3 mm and is elongated parallel to [001]. It is mounted about [010] (ie. its [010] direction is vertical) for attachment to a Cryocooler which can be rotated about a vertical axis. This geometry allows  $\{h0\ell\}$  reflections to be measured in detectors positioned in the horizontal plane.

(Remember that the vectors  $\underline{a}$ ,  $\underline{b}$ ,  $\underline{c}$  of the direct lattice are related to the reciprocal lattice vectors  $\underline{a}^*$ ,  $\underline{b}^*$ ,  $\underline{c}^*$  according to:

$$\underline{a}^* = \frac{2\pi}{V} \underline{b} \times \underline{c} \quad \underline{b}^* = \frac{2\pi}{V} \underline{c} \times \underline{a} \quad \underline{c}^* = \frac{2\pi}{V} \underline{a} \times \underline{b} \quad V = \underline{b} \times \underline{c} \cdot \underline{a}$$

$$\underline{a} = \frac{2\pi}{V^*} \underline{b}^* \times \underline{c}^* \quad \underline{b} = \frac{2\pi}{V^*} \underline{c}^* \times \underline{a}^* \quad \underline{c} = \frac{2\pi}{V^*} \underline{a}^* \times \underline{b}^* \quad V^* = \underline{b}^* \times \underline{c}^* \cdot \underline{a}^*$$

Bragg scattering can then occur for scattering vectors  $\underline{\kappa}$  which satisfy  $\underline{\kappa} = h\underline{a}^* + k\underline{b}^* + \ell\underline{c}^*$  where  $h, k$  and  $\ell$  are integers.

Note that for only this section of the course the symbol  $\underline{\kappa}$  is used to represent the scattering vector in order to avoid confusion with the magnetic interaction vector, see below.)

2. Station 3 of the TEST beam is used for the experiment. Mount two  $^3\text{He}$  gas detectors at scattering angles ( $2\theta$ ) of  $50^\circ$  and  $110^\circ$  respectively. Since the specimen is well-aligned about [010], the vertical aperture of the detectors can be limited by pieces of cadmium or  $\text{B}_4\text{C}$ , thus reducing the background and the risk of unwanted reflections whose scattering vectors are not in the horizontal plane. Assume that the specimen is aligned to better than  $0.5^\circ$  and adjust the detector apertures accordingly: note that the effect of misalignment is a function of detector angle. Record the distance of each detector from the specimen, the dimensions of its aperture and the angles the aperture subtends at the sample position.

**NB** It is a necessary condition for the correct measurement of an integrated intensity (which may then be related to the modulus of the corresponding structure factor) that the detector accept all the diffracted beam. For this reason, the detector aperture must not be reduced too much.

3. Note the alignment of the specimen's [001] direction relative to the outside of the orienter before installing the radiation shields and lowering the orienter into the specimen tank. The rotation of the crystal (the omega,  $\omega$ , axis) is measured by an incremental encoder and it can be set under computer control. It is first necessary to drive the circle under manual control until it reaches its negative limit (clockwise rotation when viewed from above). The reading of the scaler attached to the incremental encoder must then be set to zero.
4. To find the relationship between the crystal orientation in the  $\underline{a}^*-\underline{c}^*$  plane and the  $\omega$  axis angle it is necessary to centre a known  $h0l$  reflection in one of the detectors. Since orders  $nh$ ,  $nk$  and  $nl$  of any reflection  $hkl$  scatter into the same direction, it is sensible to look for an  $hkl$  with low indices so that the fundamental has a large d-spacing and  $n$  can take a large number of values for the usable range of incident wavelengths. Locate  $\underline{c}^*$  by observing the  $00l$  reflections in the  $110^\circ$  detector. First set [001] to an angle definitely on one side of the correct position (remember that the scattering vector  $\underline{\kappa}$  bisects the angle between the incident and diffracted beams). You should be able to get within  $5^\circ$  of the true  $\omega$  angle for scattering. Open the shutter and start taking data.
5. Use the computer to accumulate successive spectra, each of the same number of  $\mu\text{A}$ -hour accumulated beam, incrementing  $\omega$  between each measurement until you observe the  $00l$  series of diffraction peaks in the  $110^\circ$  detector. The  $\omega$  increment should be chosen as large as possible consistent with being sure not to miss the reflecting position (consult your detector aperture measurements relating to 2 above). When you have found the  $00l$  reflections, centre them in the detector by making further scans as a function of  $\omega$  and finding the angle midway between the angles corresponding to half the maximum intensity. Record the spectra at this  $\omega$  value for a small number of  $\mu\text{A}$ -hour. Compare the widths of the observed peaks with powder diffraction peak widths recorded with a similar scattering angle - can you explain the difference?
6. You are now in the position to set any other  $h0l$  row into the reflecting position for either detector. Draw a plan of the  $\underline{a}^*-\underline{c}^*$  reciprocal lattice to remind yourself how to do this. Measure the  $h00$  row in the  $50^\circ$  detector then, if time permits, one or two other rows in either detector (it might be interesting to measure the  $00l$  spectra in the  $50^\circ$  detector to compare the data obtained at different wavelengths).
7. Cool the sample to base temperature. Start collecting the same scans as in 6 above as soon as the temperature is below 20 K. Be sure to collect  $00l$  data in the  $110^\circ$  detector and the  $h00$  data in the  $50^\circ$  detector.
8. Use GENIE to extract integrated intensities from your scans. The integrated intensity,  $I_{obs}$ , observed at a wavelength in such patterns depends on the incident flux  $i_0(\lambda)$ , a geometrical factor and the sum of the squares of the moduli of the nuclear structure factor  $F$  and the magnetic interaction vector  $Q$ :

$$I_{obs} = \text{scalefactor} \times i_0(\lambda) \times \lambda^4 \times (1/\sin^2 \theta) \times (F^2 + Q^2)$$

where the nuclear and magnetic structure factors for the  $hkl$  reflections are

$$F_{hkl} = \sum_j \bar{b}_j \exp\left(2\pi i(hx_j + ky_j + lz_j)\right) \exp(-B_j s^2)$$

$$\underline{M}_{hkl} = \sum_j \underline{\mu}_j f_j(\underline{\kappa}) \exp\left(2\pi i(hx_j + ky_j + lz_j)\right)$$

and the  $j$  summations are over the atoms in the cell.  $(x_j, y_j, z_j)$  is the position of the  $j^{\text{th}}$  atom in unit cell coordinates (ie. in fractions of the lattice parameters).  $B_j$  is the isotropic temperature factor for

the  $j^{\text{th}}$  atom and  $s = \sin \theta / \lambda$  for the  $hkl$  reflection.  $\mu_j$  is the magnetic moment of the  $j^{\text{th}}$  atom and  $f_j(\mathbf{k})$  is the magnetic form factor of the  $j^{\text{th}}$  atom. The magnetic interaction vector is then given by

$$\underline{Q} = \hat{\mathbf{k}} \times [\underline{M} \times \hat{\mathbf{k}}]$$

Obtain an estimate of the scale factor in the experiment by comparing your calculated nuclear structure factors with the observed numbers of counts in the ambient temperature pattern. To first order, the  $i_n(\lambda)$  factor can be removed by dividing your spectra by the equivalent spectra from a vanadium sample. You may assume that both atomic species have an isotropic temperature factor  $B_j = 0.3 \text{ \AA}^{-2}$ . Compare the ambient temperature moduli of structure factors with a model calculation, given that the scattering lengths of Mn and F are  $-0.3730$  and  $0.5654 \times 10^{-12}$  cm respectively.

9. Which reflections in the low temperature spectra contain magnetic intensity? Deduce the magnetic structure from your observations.
10. Using your estimate of the scale factor, calculate the modulus of the magnetic interaction vector, and hence the magnetic structure factor, for each of the magnetic reflections. The scattering length equivalent of  $1\mu_B$  is  $0.2695 \times 10^{-12}$  cm. Plot these values as a function of  $\sin\theta/\lambda$  and estimate the magnetic moment on each  $\text{Mn}^{2+}$  ion given that the form factor for a 3d electron in the free ion has the values:

$\sin\theta/\lambda$ ( $\text{\AA}^{-1}$ )	0.00	0.05	0.10	0.15	0.20	0.25	0.30
form factor	1.000	0.9724	0.8955	0.7845	0.6579	0.5314	0.4159

## 5. Beryllium Filter Spectrometer

The Beryllium filter spectrometer is probably the simplest inelastic instrument it is possible to design and construct. The resolution is generally quite poor but it has the advantage of high intensity, and for this reason it is often used on sources where the flux is limited.

The instrument is of the indirect type where the analysing is done after the sample. In section 2 you should have measured the transmission of the Be-filter and seen that because of the Bragg scattering only neutrons of a wavelength longer than the  $3.97\text{\AA}$  (or energies less than  $5.2\text{meV}$ ) are transmitted. This confines the energy of the final neutrons to be between 0 and  $5.2\text{meV}$ , a coarse but effective energy analysis. If all the distances are known then it is possible to calculate the initial neutron energy from the total time-of-flight, assuming that the median final energy is close to the Bragg cut-off energy.

The two factors you should consider when building a spectrometer are the resolution and the intensity. The resolution of a Be-filter spectrometer has several main components (Windsor Chapter 9, page 355):

- (i) The most obvious contribution is the fixed error of  $5\text{meV}$  due to the spread in scattered energies.
- (ii) The spread of scattered neutron flight times over the secondary flight path causes a timing error with a FWHM of  $\Delta t \approx (2^{1/4} - 1)L_1(m/2E_c)^{1/2}$ . Where  $E_c$  is the Bragg cut-off energy and  $L_1$  is the scattered flight path.
- (iii) There is also a timing error due to the spread in secondary flight paths  $\Delta L_1$  due to the finite sizes of the sample and detector. This can be a very large effect due to the low energy of the final energy neutrons. For neutrons of energy  $E_d$ , the FWHM error is  $\Delta t \approx \Delta L_1(m/2E_d)^{1/2}$
- (iv) A further contribution comes from the timing error in the moderator. In the epi-thermal region this is  $\Delta t = BE_n^{-1/2}$  where B being a small constant value. This is generally much smaller than the other terms.

Convolving these terms together produces the complete resolution:

$$\Delta\hbar\omega = \left[ E_c^2 + \frac{E_0^3}{L_0^2} \left( \frac{(2^{1/4} - 1)^2 L_1^2}{E_c} + \frac{\Delta L_1^2}{E_d} + \frac{2 B^2}{m E_0} \right) \right]^{1/2}$$

Inelastic measurements take considerably longer than elastic measurements as the intensity of the scattering is so much smaller. A typical measurement may take more than 8 hours, which means that you will have to spend some time thinking about the design of the instruments before you start work.

The Beryllium used in these experiments consists of a 10cm long cylinder 5cm in diameter, wrapped in cadmium to absorb the scattered neutrons. This is placed inside a thermal flask and mounted on stand which raises it to the correct height and contains further shielding.

### 5.1. Designing the Beryllium Filter Machine

1. One of the first considerations should be to maximise the resolution. By using the equations given above pick the secondary and initial flight paths that produce the best resolution that are physically possible with the equipment available.
2. Resolution is irrelevant if there is no flux, and there is always a compromise to be made between resolution and intensity. The most important terms in the flux calculation are the lengths of the flight paths, the longer the flight path the lower the intensity. Find a sensible compromise between these two factors.

3. By using the conversion of energy to time given in the appendices, calculate the equation for the Energy as a function of time-of-flight.
4. Plot the momentum transfer as a function of energy transfer. (N.B. assume that the analysed energy is negligible). The form factor for magnetic scattering dies away quickly with momentum transfer (see Windsor p.g. 23). How could you redesign the spectrometer to look at magnetic scattering?
5. The background on an inelastic machine is far more critical than on diffraction machine. Indeed it can make all the difference between an excellent spectrometer and an unusable machine. So spend some time thinking about shielding and how to reduce the background of your design. The background in such machines usually consists of Bragg scattering from components in the beam making their way directly to the detector. These are weak and since they are not at the sample position they come at a different time to the elastic peak. For these reasons they are easy to confuse with real inelastic features. So be careful.
6. Choose the detector you intend to use with noise and background in mind and be aware that multiple scattering from the Beryllium can also find its way into the detector. This can be reduced by having some collimation after the filter.

### 5.2. *Building the Beryllium Filter Machine*

1. Before starting the actual measurement it is necessary to have values for the incident flight path and secondary flight paths. The total flight path can be measured without the beryllium filter by means of a resonant foil (see section 2), but secondary (and hence the primary) has to be found by measuring the sample to detector distance by hand..
2. There are several samples which you could look at but start with either the zirconium-hydride, or the ice sample. For inelastic scattering it is highly advantageous to cool the sample. This is because of detailed balance; at low temperatures the scattering system is in its ground state, which makes processes where the neutron can gain energy from an acoustic phonon and simultaneously lose it to the excitation of interest unlikely. The resulting spectrum is sharper and cleaner. A Closed Cycle Refrigerator (CCR) is supplied to cool the sample. Since it will be necessary to use the crane to lower the CCR into place, one of the tutors will have to help.
3. One of the major problems with the beryllium filter is frame overlap. The sample transmits all neutrons of energies below the cut-off and so if the sample has Bragg peaks at wavelengths so long that they reach the detector after the end of the frame, then frame-overlap is possible. It is difficult to guard against this completely but it should be possible to ensure that the scattering angle is low enough so that the diffraction pattern from the sample is entirely within the frame. A quick look at the diffraction pattern may be sensible.
4. Cooling the Beryllium filter is also advisable. Phonon scattering inside the beryllium can reduce the transmission of the sample at energies below the cut-off (0 to 5.2meV), hence reducing the intensity. Cooling the Beryllium from 300K to 70K by immersing it in liquid nitrogen can increase the flux by a factor of five. The beryllium used in these experiments is mounted inside a thermal flask which can be filled with liquid nitrogen. A dewar is also available with which it can be kept topped up.
5. By setting up a workspace correctly it is possible to use GENIE to convert from time of flight to energy transfer (see the appendices).

## 6. The Crystal Analyser Spectrometer

In a crystal analyser spectrometer one of the Bragg reflections from a single crystal is used to define either the primary or secondary wavelength (and hence energy). The time spread of the analysed neutrons is dependent on the crystal mosaic, which for most crystals will produce an energy spread far less than the 5.2 meV found in the Be-filter machine. Thus such machines have a much greater energy resolution, although at the penalty of less flux.

The constraints of the test beam make it much easier to look at the indirect geometry version of the crystal analyser spectrometer, and thus it is this type we will study. The energy resolution of an indirect geometry crystal-analyser spectrometer has several terms:

- (i) The mosaic of the crystal  $\Delta\theta_A$  gives a spread in the scattered wavevectors of  $\Delta|k_1| = k_1 \cot\theta_A \Delta\theta_A$ .
- (ii) The term due to the moderator time broadening in the epi-thermal region is given by  $\Delta t \approx K / k_0$  where K is a constant.
- (iii) The finite size of the sample and detector introduces a term due to a range in the actual analyser angle. This is obviously highly dependent on the specific geometry.
- (iv) There is also a path length timing error due to the range of possible secondary flight paths.

All the timing errors can be combined into an effective length  $\delta = \hbar k_0 \Delta t / m$ , and then added to the terms due the uncertainty in  $k_1$  to produce a resolution of:

$$\frac{\Delta\hbar\omega}{E_0} = 2 \left[ \left( \frac{\delta}{L_0} \right)^2 + \left\{ \frac{E_1}{E_0} \cot\theta_A \Delta\theta_A \left( 1 + \frac{L_1}{L_0} \left( \frac{E_0}{E_1} \right)^{3/2} \right) \right\}^2 \right]^{1/2}$$

In this section you will attempt to design and build a crystal analyser spectrometer. Inelastic scattering is technically more demanding than elastic scattering and great care must be taken in the design and assembly if any results are to be obtained. Do not be too disheartened if the results you obtain do not look like those you have seen from real spectrometers; it takes considerably longer than a couple of days to build such spectrometers.

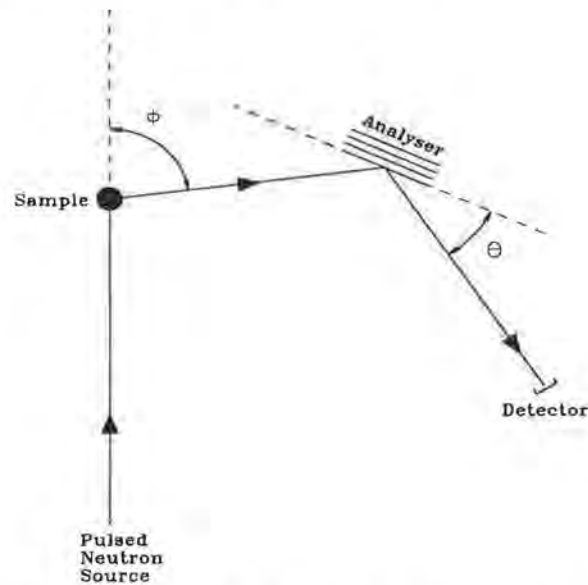


Figure 4. A schematic diagram of a crystal analyser spectrometer

### 6.1. The Design of the Crystal Analyser

1. The pyrolytic graphite (PG) analysers are arranged with the  $(00l)$  planes parallel to the surface. Thus when the analyser is used in reflection the  $(002)$ ,  $(004)$ ,  $(006)$ , etc. reflections are seen. Given that graphite is hexagonal with  $c=6.707\text{\AA}$  derive an expression that gives the analyser energy as a function of scattering angle. (Hint: convert Bragg's law to energy.)
2. Two positions are available to you: position 1 (the closest to the moderator) and position 3. With sensible estimates, or preferably measurements of the incident and secondary flight paths find the highest possible value of analyser angle (assume that the  $(002)$  reflection is used) given that the elastic line must reach the detector before the end of the frame.
3. The Beryllium filter is used to ensure that only the  $(002)$  reflection from the PG analyser is seen. Calculate the range of angles you would need ensure that the analyser's  $(002)$  reflection is below the filter's Bragg cut-off and that the  $(004)$  is above it.
4. The variation of momentum transfer ( $Q$ ) with energy transfer ( $E_T$ ) is dependent on the scattering angle (that is the angle  $\phi$  between the incident beam and the beam scattered by the sample). Choosing a sensible analyser angle that satisfies the conditions 1-3, plot the trajectory of the spectrometer in  $Q$ - $E$  space.
5. By taking into consideration all the above features pick analyser and scattering angles for a spectrometer that is suitable for:
  - a) Magnetic Scattering (low  $Q$ ).
  - b) Quasi-elastic scattering (Very high resolution).
  - c) Molecular Spectroscopy.
6. Design a spectrometer that can fit onto TEB for use in molecular spectroscopy. Take into account all the features you have investigated above and bear in mind that the spectrometer must physically fit onto either position 1 or 2 and make allowances for shielding. Remember the background on an inelastic spectrometer is far more critical than on a diffraction machine.



## 6.2. *Building the Spectrometer*

1. You should already know the primary flight path from earlier resonance foil experiments. The secondary flight paths and analyser angles will have to be carefully measured by hand.
2. There are several samples which you could look at but start with either the zirconium-hydride, or the ice sample. For both these materials it is highly advantageous to cool the sample. This is because of the detailed balance factor; at low temperatures the scattering system is in its ground state and neutron energy gain is impossible. This makes processes where the neutron can gain energy from the an acoustic phonon and simultaneously loose it to the excitation of interest unlikely, making the spectrum sharper and cleaner. A Closed Cycle Refrigerator (CCR) is supplied to cool the sample. Since it will be necessary to use the crane to lower the CCR into place, one of the tutors will have to help with the loading of the sample.
3. Whilst the sample is cooling assemble the components of the spectrometer according to your design. Try to set-up the analyser angle as accurately as possible, then do a series of short runs at different angles to maximise the reflected intensity.
4. Take great care and effort in trying to reduce the background on the spectrometer. This is vitally important as spurious peaks arriving at the wrong time can produce features that look inelastic. Without good shielding a spectromter cannot work.
5. When the spectrometer is assembled start a long run, preferably overnight.

## Appendices

### A. GENIE Basics

GENIE is a program written at ISIS and used extensively for display and manipulation of time-of-flight spectra at ISIS and at other neutron scattering centres worldwide. You will be given a manual of GENIE commands along with the other course documentation, but the purpose of this appendix is to actually get you started with using GENIE. To start the program type GENIE, or GEN for short. If you are using a terminal with windows a GKS icon will appear for the graphics. Once GENIE has started you should type commands after the >> prompt.

#### A1. *Spectrum Display*

To tell GENIE which run you want to display you use the ASSIGN command. For example ASS 3256 means that GENIE will then display spectra from run 3256. In order to enable GENIE to look at the current run before you have ended it you must type ASS DAE (DAE=Data Acquisition Electronics). To successfully access the current run you will also need to be logged on to the instrument computer.

Once you have used the ASSIGN command you can tell GENIE to plot on the terminal screen a particular spectrum for the assigned run by use of the DISPLAY command. For example D S2 will display on the screen spectrum number 2 for the currently assigned run. The limits to be used for the plot may be specified when giving a DISPLAY command, for example D S2 100 2000 will display spectrum number 2 from 100 to 2000 micro-seconds.

The spectra taken at ISIS are time-of-flight spectra and as such are stored as histogram information. When GENIE plots spectra it allows for histograms to be added together in groups. The user can control this by use of the ALTER BINNING command which will be found to have a large effect on the apparent noisiness of spectra. For example A B 5 tells GENIE to add the histograms together in groups of 5 before displaying. Note that this binning affects only the displayed data and not the data values stored for the run.

After doing a plot on the terminal screen you wish to plot a hardcopy on a laserprinter then first give the command K/H (keep hardcopy) to save the plot in a file (named DEC\_POSTSCRIPT.DAT). We have set up GENIE for the training course accounts so that issuing the command LAS3 in GENIE will send the plot to be plotted on the laserprinter in the LAD cabin whilst the command LAS2 will print on the DAC laserprinter.

#### A2. *Spectrum Manipulation*

In order to manipulate a spectrum it is first necessary to transfer the spectrum data values into a storage area within GENIE known as a workspace. For example the data for spectrum 2 of the currently assigned run may be put into workspace number 3 by typing W3=S2 . A workspace may be plotted on the screen by use of the DISPLAY command.

Simple arithmetic manipulations may be performed with workspaces. For example W3=2\*W1/W2 will multiply every y-value of workspace 1 by 2.0 and divide by the corresponding y-value of workspace 2, placing the result in workspace 3.

When using the monitor spectrum to determine a measure of the number of incident neutrons for a run you will need to make use of the command INTEGRATE. A spectrum must be placed in a workspace before this command can be used. For example workspace number 2 may be integrated from 5000µsec to 10000µsec by typing INT W2 5000 10000

Another particularly useful command in GENIE is the REBIN command. As already mentioned the time-of-flight data stored by GENIE are necessarily stored as histogram information. The REBIN command allows the counts recorded in each histogram of a workspace to be redistributed according to a new set of histogram bin boundaries. This can be very useful in producing a workspace whose x-values (ie. histogram bin boundaries) are suitable for further analysis. Not least of these uses is that the REBIN command can be used to delete unwanted data. For example, suppose that W3 contains data extending from 0.5 to 100 (the units are not important), but that we only want the data to cover the range from 1.5 to 11 with equally sized x-intervals of 0.05. This may be achieved by REB W3 1.5 (0.05) 11

More complicated arithmetical manipulations may be performed on a workspace by use of fortran programs written to be called from GENIE. For example we have provided a program TP:LOGE for taking the natural log of the y-values in a workspace (this is useful for extracting an absolute cross-section from transmission data); to perform this manipulation use the GENIE FUNCTION command by typing F W2 TP:LOGE W3 - this takes the y-values stored in W2 and puts in W3 the natural log of these y-values. In general the FUNCTION command is used when only the y-values of a workspace are to be changed whilst the TRANSFORM command is used when both x- and y-values are to be changed. The GENIE manual should be consulted for further information on the FUNCTION and TRANSFORM commands. We have provided the following programs (in the directory TP:);

TP:LOGE	Function	Take natural logs of the y-values
TP:LOG_LOG	Transform	Take natural logs of both x- and y-values
TP:FIT_LINE	Function	Fit a straight line to the data in the workspace

### A3. *Spectrum Units Conversion*

A GENIE workspace may have its x-axis units converted from time-of-flight in micro-seconds to more physically meaningful units as follows: Firstly use the SET PARAMETERS command to give GENIE the calibrated values relevant to the spectrum of interest. The format of the command is SET PAR Wn L1 L2 2θ Emode Efixed where:

Wn is the relevant workspace

L1 is the incident flight path in metres

L2 is the scattered flight path in metres

2θ is the scattering angle

Emode is the energy mode (0-elastic, 1-direct geometry, 2-indirect geometry)

Efixed is the fixed energy (it is recommended to set this to zero for elastic scattering)

All parameters must be specified, even if you intend to do a units conversion which does not require all of the parameters. For example if you are considering elastic scattering and wish to convert to an energy scale then the values given for both the scattering angle and the fixed energy are not relevant for the conversion, but must nevertheless be specified. In this case it is advisable to give reasonable plausible values, and a realistic value for the scattering angle and a fixed energy value of zero would suffice.

As an example SET PAR W3 17.4 0.46 50 0 0 tells GENIE that for the data in W3 the incident and scattered flight paths are 17.4m and 0.46m, the detector is at scattering angle 50°, the mode is for elastic scattering and that the fixed energy is zero.

Once the parameters have been set the units may be converted by use of the UNITS command. The options you are likely to use are as follows:

U/D W3	- put W3 on a d-spacing (Ångstroms) axis	(elastic scattering)
U/LAM W3	- put W3 on a wavelength (Ångstroms) axis	(elastic scattering)
U/Q W3	- put W3 on a Q (Å <sup>-1</sup> ) axis	(elastic scattering)
U/E W3	- put W3 on an energy (meV) axis	(elastic scattering)

U/W W3 - put W3 on an energy transfer (meV) axis (inelastic scattering)

Note that if the workspace whose units you are converting is a ratio (eg. sample divided vanadium) then before using the UNITS command you should first issue the command SET YCODE W3 -1 . (This is a subtlety required since GENIE normally plots histogram data as y-units per x-unit which is not suitable in the case of a ratio.)

## B. Useful Conversion Factors

Density:  $\rho(\text{g cm}^{-3}) = A(\text{amu}) \times \rho(\text{atoms } \text{\AA}^{-3}) \times 1.66057$  where A is atomic mass and  $\rho$  is density.

Neutron energy:

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

$$E = 2.0717k^2 = \frac{81.787}{\lambda^2} = 4.1354\nu = 5.2276 \times 10^6 \frac{L^2}{t^2} = 0.086165T$$

$E[\text{meV}]$ ,  $\lambda[\text{\AA}]$ ,  $\nu[\text{THz}]$ ,  $k[\text{\AA}^{-1}]$ ,  $L[\text{m}]$ ,  $t[\mu\text{sec}]$ ,  $T[^\circ\text{K}]$ .

## C. Description of the Test Beamline.

The Test Beamline, TEB, (see the figure at the end of these notes) is situated in the ISIS experimental hall on the south side of the target station (position S9). It views a liquid methane moderator inside the target station. Whereas the neutron beam on most ISIS instruments is largely inaccessible due to the presence of shielding material, the test beam has an open arrangement allowing easy access to the neutron beam. This design has been chosen so that many different applications can be performed on the same beamline, and makes it particularly suitable for our practical neutron scattering course. TEB has three sample positions at approximate distances from the moderator of 12.6m, 15.0m and 17.4m. Each of these positions has a standard ISIS sample environment tank, and there are slots between the tanks which can be used to hold collimation pieces for reducing the beam size. At position 3 (the one furthest from the moderator) we have provided a table for use in the training course to which the detector can be fixed at a variety of different marked scattering angles.

The size of the beam on the test beamline is defined by B<sub>4</sub>C collimating pieces which may be placed in the beam at various points. Typically a rectangular collimator which defines a beam of width 18mm and height 21mm is placed just before position 1. The actual size of the beam at each sample position is determined by the beam divergence which results from both the finite size of the collimator and the finite size of the moderator (a square 10cm on a side).

## D. Checklist on how to perform a run

### D1. Checklist for Beginning a Run

When the required experimental setup has been made a run may be done as follows:

- Press white search button at rear of blockhouse (having ensured blockhouse is empty!).
- Close door to blockhouse, slide and turn bolt in door, then remove slave key from bolt mechanism.
- Put slave key into grey interlock key box and remove master key.
- Put master key in green box and turn it clockwise (it's stiff!).
- Press button to open shutter, either by the blockhouse door or in the portacabin.

- Close air inlet valve and open pump valve on pump by blockhouse door.
- Change title on supervisor window on workstation in portacabin and then type BEGIN.

### D2. Checklist for Ending a Run

When the statistics of the run are adequate it may be ended as follows:

- Type END on supervisor window on workstation in portacabin.
- Press close shutter button.
- Close pump valve and open air inlet valve.
- When shutter has closed, remove master key from green box and put it in the grey interlock key box.
- Remove a slave key from the grey box, put it in the door bolt mechanism and release the door bolt.

## E. d-spacings and selection rules for simple crystal systems

### E1. Simple Cubic (sc)

$$d_{hkl} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}} \quad \text{all reflections allowed}$$

### E2. Face Centred Cubic (fcc)

$$d_{hkl} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}} \quad h, k \text{ and } \ell \text{ must be all even or all odd}$$

### E3. Body Centred Cubic (bcc)

$$d_{hkl} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}} \quad (h+k+\ell) \text{ must be even}$$

### E4. Hexagonal Close Packed (hcp)

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{\ell^2}{c^2} \quad \text{allowed reflections must satisfy } \ell=2n \text{ or } h-k-1=3n \text{ or } h-k-2=3n, \text{ where } n \text{ is an integer.}$$

## F. Crystal structures of some metallic elements

Scandium	Sc	hexagonal	a=3.31Å	c/a=1.594
Titanium	Ti	hexagonal	a=2.95Å	c/a=1.588
Vanadium	V	bcc	a=3.02Å	
Chromium	Cr	bcc	a=2.88Å	
Manganese	Mn	cubic	a=8.89Å	
Iron	Fe	bcc	a=2.87Å	
Cobalt	Co	hexagonal	a=2.51Å	c/a=1.622
Nickel	Ni	fcc	a=3.52Å	
Copper	Cu	fcc	a=3.61Å	
Zinc	Zn	hexagonal	a=2.66Å	c/a=1.856
Yttrium	Y	hexagonal	a=3.65Å	c/a=1.571
Zirconium	Zr	hexagonal	a=3.23Å	c/a=1.593
Niobium	Nb	bcc	a=3.30Å	

Molybdenum	Mo	bcc	a=3.15Å	
Technetium	Tc	hexagonal	a=2.74Å	c/a=1.604
Ruthenium	Ru	hexagonal	a=2.70Å	c/a=1.584
Rhodium	Rh	fcc	a=3.80Å	
Palladium	Pd	fcc	a=3.88	
Silver	Ag	fcc	a=4.09Å	
Cadmium	Cd	hexagonal	a=2.98Å	c/a=1.886
Lanthanum	La	hexagonal	a=3.75Å	c/a=1.619
Hafnium	Hf	hexagonal	a=3.20Å	c/a=1.582
Tantalum	Ta	bcc	a=3.31Å	
Tungsten	W	bcc	a=3.16Å	
Rhenium	Re	hexagonal	a=2.76Å	c/a=1.615
Osmium	Os	hexagonal	a=2.74Å	c/a=1.579
Iridium	Ir	fcc	a=3.84Å	
Platinum	Pt	fcc	a=3.92	
Gold	Au	fcc	a=4.08Å	
Mercury	Hg	(rhombohedral)	a=2.99Å	$\alpha=70.45^\circ$

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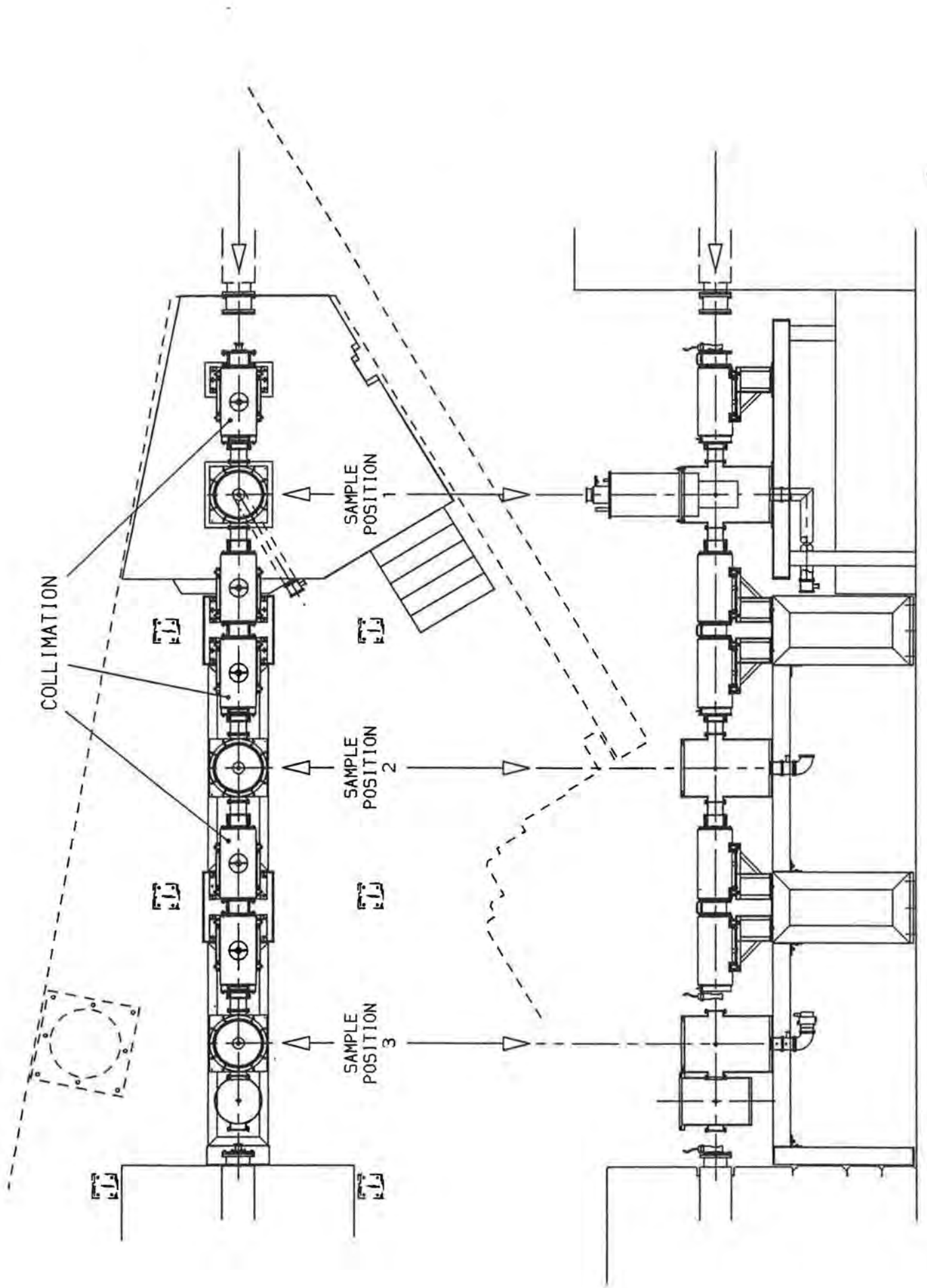


Figure 5 The Test Beamline, TEB.





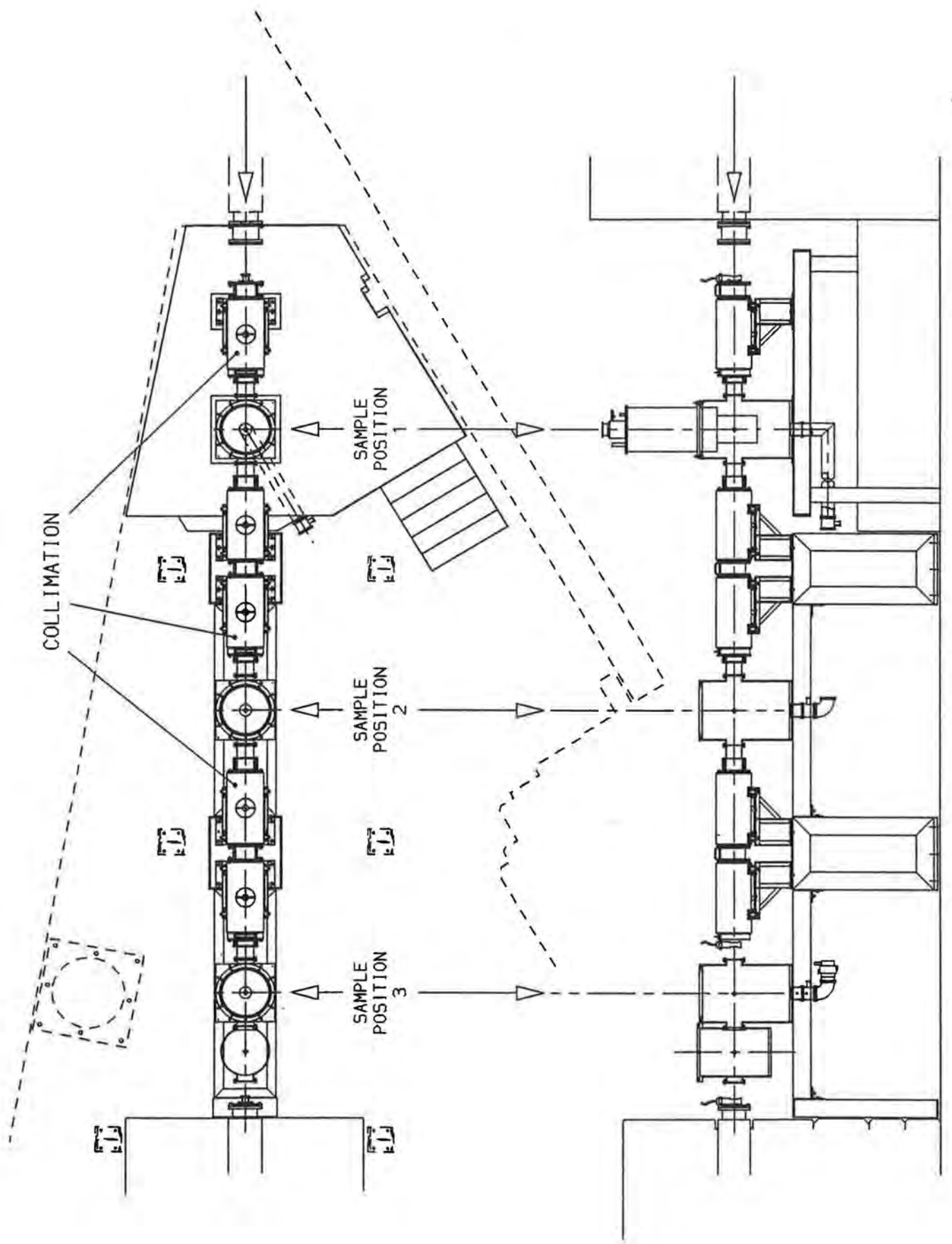


Figure 5 The Test Beamline, TEB.





...the patient's health and well-being...  
...the physician's duty to his patient...  
...the ethical principles of medicine...  
...the relationship between doctor and patient...  
...the importance of medical education...  
...the role of the medical profession...  
...the challenges of modern medicine...  
...the need for medical reform...  
...the future of the medical profession...