Neutron interactions– practical

Introduction
These practical exercises act as an introduction to the running of the ISIS instruments and software as well as an introduction to some of the basic interactions of neutrons with matter. By the end of this practical you should be familiar with:

1) the interlocks on the instrument
2) controlling the shutter
3) starting and ending runs
4) using the OpenGenie/MANTID software to look at the raw data and to do simple data manipulations
5) the neutron cross-sections - coherent/incoherent and absorption.
6) what coherent and incoherent scattering do to your data
7) the basics of time-of-flight neutron diffraction

We are going to introduce these ideas by recreating a classic experiment first performed by Shull, Wollan and co-workers at the dawn of neutron scattering in 1948. The results of this experiment were initially puzzling, but its resolution was an important milestone in the development of the technique.

Background
The neutron was discovered by Chadwick at the Cavendish laboratory in Cambridge in 1932, and although some demonstration experiments were done in 1936 real neutron scattering experiments were not done until the first reactors were built for the Manhattan project during the Second World War. The first dedicated instrument for this kind of work was a crude 2-axis instrument built in 1943 by Ernest Wollan at the reactor at Oak Ridge National Laboratory in Tennessee, dubbed the Clinton Pile. Over the next ten years he and Clifford Shull developed the instrument and the technique[1], created a library of scattering lengths[2] and performed many classic experiments such as determining the structure of ice[3] and other hydrogen containing systems[4], and provided the first direct evidence for antiferromagnetism[5]. They also developed polarised neutron scattering[6] and were able to separate magnetic and nuclear scattering in ferromagnetic materials[7]. Clifford Shull received a Nobel Prize for this work in 1994. Ernest Wollan only failed to receive the same honour because of his death in 1984.

Worksheet
A) Transmission measurements
After Enrico Fermi and others developed the Rn-Be radioactive source of neutrons there was a concerted attempt to measure the total cross-sections of various materials by teams throughout the world[8,9]. This ultimately led to Fermi getting the Nobel Prize in 1938 for his discovery that some elements transmute when bombarded with neutrons. The cross-sections were found using transmission measurements i.e. taking the ratio of the transmitted intensity with some material in the beam to that with nothing in the beam. This is a relatively easy process and we are going to attempt to recreate these measurements using the ISIS instruments.
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 integrated incident intensity). If the sample has uniform thickness across the beam (a flat plate sample) then:

\[ T = \exp(-\rho \sigma x) \]

Where \( T \) is the transmission, \( \rho \) is the density, \( \sigma \) is the cross-section and \( x \) is the thickness. 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 (e.g. 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 thickness 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 and consistent set of units is \( \rho \) in atoms Å\(^{-3}\), \( \sigma \) in barns (1 barn=10\(^{-28}\) m\(^2\)), \( x \) in cm. Convince yourself that these are consistent units for calculating transmissions.

**Practical**

- None of the instruments have suitable transmission monitor, so we will perform our measurements in a different way, using the unusual scattering properties of vanadium. Put the vanadium sample in the beam and collect a data set. *(N.B. Vanadium can get active in a neutron beam, please monitor it carefully)*
- Leave the vanadium the beam and put a sheet of cadmium somewhere in the incident beam. Collect a second data set. *(N.B. Cadmium is toxic, wear gloves at all time and be careful when removing the cadmium; it may be active)*

Using the information in Appendix 3 and estimates of the flight paths on the instrument create a plot of the transmission of cadmium.
- Make a similar measurement using one of the polythene sheets
- Finally, make the same measurement with a thin foil of tantalum metal in the beam

**Questions**

1. Explain the shape of the vanadium spectrum that you see and estimate the temperature of the moderator (see Appendix 1)
2. Plot the transmission of cadmium as a function of energy. What is the transmission at 25meV and above 300meV?
3. Given that the chemical formula of polythene is \((\text{CH}_2)_n\) and that the density of the polythene provided is 0.908 gcm\(^{-3}\), extract from your measurement the absolute cross-section as a function of wavelength. If you assume all the scattering is from hydrogen make an estimate for the total scattering cross-section of hydrogen at 25meV.
4. Knowing that the nuclear neutron absorption resonances of Ta are at energies of: 4.28, 10.36, 13.95, 20.29 and 22.72 eV, calibrate the total flight-path between the moderator and the detector (NB, there may be a \( t_0 \) offset due to timing electronics)
B) Neutron diffraction measurements

Solving the structure of the Alkali Hydrides

In 1947 and 1948 Shull and Wollan were looking at hydrogen containing materials, such as ice and metal-hydrides, and trying to determine their structures using neutron diffraction. Although quite a lot was already known, x-rays were not able to measure the positions of the hydrogen atoms. We are going to recreate their early measurements on NaH and NaD and use these results to illustrate some important points about the interactions of neutrons with matter.

The alkali metal hydrides LiH, NaH, KH and CsH were known to have an fcc structure. In the case of LiH the x-ray scattering powers of lithium and hydrogen are sufficiently similar for the hydrogen positions to have been determined and the structure revealed to be of the NaCl type with the hydrogen replacing the chlorine. Although it was suspected that the others were the same there was no proof. Using unit cell size arguments it was determined that there were only two possible fcc structures that they could have: either the NaCl type or the ZnS type.

The NaCl type structure has two atoms in each basis separated by half the body diagonal of the cubic unit cell. There are four NaCl units in each unit cube:

<table>
<thead>
<tr>
<th>Na</th>
<th>0 0 0</th>
<th>½ ½ 0</th>
<th>½ 0 ½</th>
<th>0 ½ ½</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl</td>
<td>½ ½ ½</td>
<td>0 0 ½</td>
<td>0 ½ 0</td>
<td>½ 0 0</td>
</tr>
</tbody>
</table>

The ZnS structure can be seen as two interpenetrating fcc structures displaced by one-quarter of a body diagonal. In the conventional cubic cell the atom co-ordinates are:

<table>
<thead>
<tr>
<th>Zn</th>
<th>0 0 0</th>
<th>½ ½ 0</th>
<th>½ 0 ½</th>
<th>0 ½ ½</th>
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<tr>
<td>S</td>
<td>¼ ¼ ¼</td>
<td>¼ ¾ ¾</td>
<td>¾ ¾ ¾</td>
<td>¾ ¾ ¾</td>
</tr>
</tbody>
</table>

Performing a diffraction experiment on a pulsed neutron source requires little more than a sample and a detector. On a pulsed source, wavelength analysis of the "white" incident neutron beam by time-of-flight methods means a complete diffraction pattern can be collected in a single, fixed detector.
In order to minimise the time taken to collect a diffraction pattern of sufficient quality, multiple detectors are used, grouped into banks. Each detector bank is optimised to provide the best compromise between resolution, count rate and the \( d \)-spacing range accessible in the time frame between successive neutron pulses from the source, which at ISIS is 20ms between the 50Hz pulses.

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

a) the sample in a can.
b) the empty can.
c) a vanadium rod.
d) the empty instrument.

The empty instrument data enable the vanadium run and the sample + can data sets to be corrected for sample-independent background. The data from the empty can must be subtracted from the sample can run using factors for attenuation and for multiple scattering. The vanadium data must also be corrected for attenuation and multiple scattering. Finally, the corrected sample data are divided by the corrected vanadium data to remove the flux shape from the diffraction pattern (i.e. to take into account the variation in intensity of the incident neutron beam with wavelength).

For our current purposes, however, we will dispense with the empty can and empty instrument runs because 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.

**Practical**

- Perform diffraction measurements on NaD and NaH: these are already in pre-sealed cans (N.B. do not open the cans; the samples are very sensitive to air.)
- Examine the data collected in each of the detector banks on the diffractometer. The final data sets patterns will need to be of good statistical quality so that individual Bragg peaks can be fitted, so the runs on both NaH(D) and on vanadium should be fairly long, but you should examine the spectra critically in Mantid to decide for yourself. When examining the diffraction patterns, look first at the summed data from the detectors in a bank.
- Whilst the data collection is in progress you can start to study the powder diffraction pattern. Firstly note the detailed shape of the powder peaks as seen in the detectors at higher scattering angles - 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.
- Note also how the positions and widths of the reflections from the NaH(D) sample vary from one detector bank to another.

**Questions**

5. Using Appendix 4 calculate the \( d \)-spacing of the first few major reflections of NaH given that the lattice parameter is 4.88 Å and that it is cubic.

6. Use the relationships in Appendix 3 to derive an expression between time-of-flight and \( d \)-spacing. Use this to calculate where in time you would expect the 111 and 200 reflections to occur.
7. Using Mantid display the spectra in time-of-flight and identify the major reflections – convert to d-spacing to check.

8. What do you notice about the general shape of the pattern? Where is the detected intensity greatest? Where is the detected intensity least?

9. Now look at the diffraction pattern after it has been normalised (i.e. it has been corrected using the vanadium, as mentioned in the introduction).

10. Compare the spectrum at low and high angles:
   i. Where is the detector bank with the highest resolution (narrowest reflections) positioned?
   ii. Where is the detector bank with the lowest resolution (broadest reflections) positioned?
   iii. By differentiating Bragg’s law see if you can understand why

11. Which detector bank measures the longest d-spacings?

12. Calculate expressions for the structure factors of the NaCl and ZnS structures and using these expressions calculate the structure factors for the major peaks for both candidate structures.

13. Using Mantid find the integrated intensity under first few peaks.

14. Using your measured intensities and your expressions for the structure factors determine the correct structure for NaH(D)

15. Given a scattering length of 3.45 fm for Na estimate the scattering length of hydrogen

16. Using the transmission data that you measured earlier calculate the coherent and incoherent cross-sections for hydrogen.

17. What is the origin of this incoherence?

18. If there is time you may also be able to calculate the triplet and singlet scattering lengths for hydrogen.
Appendix 1  The incident spectrum

The term flux shape $\phi(E)$ is used to denote the number of neutrons in the beam per unit energy i.e. 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.

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

where $J$ is a constant and $T$ is a characteristic temperature.

Appendix 2  Coherent and incoherent scattering

Neutron cross-sections are defined as effective areas and are conventionally given in barns ($10^{-24} \text{ cm}^2$). They are related to a quantity $b$ known as the scattering length by the relation

$$\sigma = 4\pi b^2$$

The scattering length can be positive or negative and sometimes imaginary numbers are used to denote neutron absorption.

Unlike x-rays which scattering from the electrons around an atom, neutrons interact with the nucleus. This means that different isotopes or nuclear spin states can have different scattering cross-sections. Let us consider a system where the scattering length varies and scattering length $b_i$ occurs with a relative frequency of $f_i$ $(\sum f_i = 1)$. The average scattering length is then

$$\bar{b} = \sum f_i b_i$$

and the average value of $b^2$ is

$$\bar{b^2} = \sum f_i b_i^2$$

Neutron scattering cross-sections are normally split into coherent and incoherent parts which expressions given by

$$\sigma_{\text{coh}} = 4\pi \bar{b}^2 \quad \sigma_{\text{inc}} = 4\pi \left\{\bar{b^2} - (\bar{b})^2\right\}$$

The coherent scattering is just the average value and gives information about the correlation between different atoms and so it is the cross-section used to describe interference effects such as diffraction. The incoherent scattering is that due to the randomness in the system and has no equivalence in x-ray scattering. It only shows
interference effects between the same atom at different times, the scattering from different atoms has random phases and cancels out.

To calculate these values we must assume that there is no correlation between the values of $b$ for the different nuclei then we can write

$$b_j b_j = \overline{b^2} \quad j' = j$$

$$b_j b_j = \overline{b^2} \quad j' = j$$

**Spin and Isotopic Incoherence**

There are two types of incoherence in neutron scattering: that due to different isotopes and that due to different nuclear spins. Suppose the system consists of a single isotope of nuclear spin $I$. The spin of the nucleus-nucleon system has the values $I + \frac{1}{2}$ or $I - \frac{1}{2}$ (denoted by $b^+$ and $b^-$). The number of states associated with each spin state is

$$2(I + \frac{1}{2}) + 1 = 2I + 2 \quad \text{for} \quad I + \frac{1}{2}$$

$$2(I - \frac{1}{2}) + 1 = 2I \quad \text{for} \quad I - \frac{1}{2}$$

If the neutrons are not polarised and the nuclear spins have random directions, then the probabilities that the neutrons will be scattered for each spin-state are

$$p^+ = \frac{2I + 2}{4I + 2} = \frac{I + 1}{2I + 1} \quad \text{for} \quad b^+$$

$$p^- = \frac{2I}{4I + 2} = \frac{I}{2I + 1} \quad \text{for} \quad b^-$$

giving an average cross-section of

$$\overline{b} = \frac{1}{2I + 1} \{ (I + 1)b^+ + I b^- \}$$

If there are several isotopes within a material then for each isotope the frequencies $p^+$ and $p^-$ must be multiplied by the relative abundance of the isotope.

$$\overline{b} = \sum_{\xi} \frac{c_{\xi}}{2I_{\xi} + 1} \{ (I_{\xi} + 1)b^+_{\xi} + I_{\xi} b^-_{\xi} \}$$

where $c_{\xi}$ is the relative abundance of the $\xi$th isotope and $I_{\xi}$ is its nuclear spin. $\overline{b}$ is known as the coherent scattering length.
Free and bound cross-sections

The relationship between the **free** (i.e. short neutron wavelength) and the **bound** (i.e. long neutron wavelength) scattering cross-sections for an element with atomic weight $A$ is;

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

This effect is obviously largest for hydrogen which is why we have chosen polythene to demonstrate it. Use the copy of Sears’ cross-section tables (Sears, 1992) to calculate both the free and bound cross-section of polythene. 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.

**Neutron Diffraction**

Neutron diffraction is a coherent elastic scattering process governed by an equation of the form:

$$ \frac{d\sigma}{d\Omega} = N \frac{(2\pi)^3}{v_0} \sum |F_N(\kappa)|^2 $$

where $F_N(\kappa)$ is known as the nuclear unit-cell structure factor, $N$ is the number of unit cells in the sample and $v_0$ is the volume of the unit cell.

The main equation is made up of a summation of delta functions whose magnitude is given by the square of the structure factor. The delta function’s occur when the scattering vector $\kappa$ equals a reciprocal lattice vector $\tau$,

$$ \kappa = k_i - k_f = \tau $$

which is graphically represented in Figure 1.

![Figure 1: Bragg's law in reciprocal space. Coherent scattering only occurs when $\kappa = \tau$.](image-url)
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When the vector $\boldsymbol{\tau}$ is perpendicular to a crystal plane, its magnitude is

$$\tau = n \frac{2\pi}{d}$$

where $d$ is the spacing of the plane. Since $k = \frac{2\pi}{\lambda}$ where $\lambda$ is the wavelength of the neutrons we can derive the more familiar expression for Bragg’s law

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

The intensity of the scattering is given by nuclear structure factor which is given by a summation over the atoms in the unit cell

$$F_N(\mathbf{K}) = \sum_d \bar{b}_d \exp(i\mathbf{K} \cdot \mathbf{r}_d) \exp(-W_d)$$

$\mathbf{r}_d$ is the position of the $d^{th}$ atom in unit cell and $W_d$ is the Debye-Waller factor. Breaking the vectors into their Cartesian components for and using assuming we are at the Bragg condition $\mathbf{K} = \boldsymbol{\tau}$

$$\mathbf{\tau} \cdot \mathbf{r}_d = (h\mathbf{a} + k\mathbf{b} + l\mathbf{c}) \cdot (x_d\mathbf{a} + y_d\mathbf{b} + z_d\mathbf{c}) = 2\pi(hx_d + ky_d + lz_d)$$

for the $(hkl)$ reflection. This formula gives the rules for the non-zero diffraction peaks. For example a bcc lattice has atoms at $(0,0,0)$ and $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ which give a structure factor of

$$F_N(hkl) = b\left[1 + \exp[i\pi(h + k + l)]\right]$$

since the exponential is -1 if $h + k + l$ is odd and +1 if it is even, we have

$$F_N = 0 \quad \text{if } h + k + l \text{ is odd}$$

and $$F_N = 2b \quad \text{if } h + k + l \text{ is even}$$
Appendix 3  Useful Conversion Factors

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

Neutron energy:

\[
E = \frac{h^2 k^2}{2m} = \frac{h^2}{2m \lambda^2} = h \nu = \frac{m L^2}{2 \tau^2} = k_B T
\]

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

\( E[\text{meV}], \; \lambda[\text{Å}], \; \nu[\text{THz}], \; k[\text{Å}^{-1}], \; L[\text{m}], \; \tau[\text{µsec}], \; T[\text{K}] \).

Appendix 4  \( d \)-spacings and selection rules for simple crystal systems

1. Simple Cubic (sc)

\[
d_{hk\ell} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}}
\]

all reflections allowed

2. Face Centred Cubic (fcc)

\[
d_{hk\ell} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}}
\]

\( h, k \) and \( \ell \) must be all even or all odd

3. Body Centred Cubic (bcc)

\[
d_{hk\ell} = \frac{a}{\sqrt{(h^2 + k^2 + \ell^2)}}
\]

\( h+k+\ell \) must be even

4. Hexagonal Close Packed (hcp)

\[
\frac{1}{d_{hk\ell}^2} = 4\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{\ell^2}{c^2}
\]

reflections must satisfy \( 0\ell; \; \ell=2n \) or \( hk0; \; h-k=3n \)

or \( hk0; \; h-k=3n, \) where \( n \) is an integer.
## Appendix 5  Crystal structures of some metallic elements

<table>
<thead>
<tr>
<th>Element</th>
<th>Symbol</th>
<th>Structure</th>
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<th>c/a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scandium</td>
<td>Sc</td>
<td>hexagonal</td>
<td>3.31Å</td>
<td>1.594</td>
</tr>
<tr>
<td>Titanium</td>
<td>Ti</td>
<td>hexagonal</td>
<td>2.95Å</td>
<td>1.588</td>
</tr>
<tr>
<td>Vanadium</td>
<td>V</td>
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<td></td>
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<tr>
<td>Chromium</td>
<td>Cr</td>
<td>bcc</td>
<td>2.88Å</td>
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<tr>
<td>Manganese</td>
<td>Mn</td>
<td>cubic</td>
<td>8.89Å</td>
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<tr>
<td>Iron</td>
<td>Fe</td>
<td>bcc</td>
<td>2.87Å</td>
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<td>Cobalt</td>
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<td>1.622</td>
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<td>fcc</td>
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<td>Tantalum</td>
<td>Ta</td>
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<tr>
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<td>3.92</td>
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<tr>
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<tr>
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<td>Hg</td>
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## References