Oxford School on Neutron Scattering (2022)

Practical Neutron Scattering (2 lectures)

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Recommended Books:

- Boothroyd, *Principles of Neutron Scattering from Condensed Matter*, Chapter 10
- Windsor, *Pulsed Neutron Scattering*

This is an excellent - and very complete - text on the practicalities of (pulsed) neutron scattering, which can nevertheless also be applied to continuous neutron scattering in many cases. Covers a lot of material that other texts often miss out concerning the practicalities of neutron scattering

- Berendsen, *Data and Error Analysis*

This is really a must for all experimental scientists! Explains fully the concepts of error presentation, estimation and propagation. Includes a full account of Poisson statistics.

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1. Before the Experiment: Optimising sample size & shape

Calculating and optimising the size and shape of your sample is most important in neutron scattering. Neutron beams worldwide are commonly low in flux - much much lower than x-ray beams for example. So most of the time it is important to maximise the number of neutrons scattered by your sample - and hence the size of your sample - and therefore the number of **scattering units**. This is not always the case however.

- Sometimes your sample may scatter too strongly for the detectors (dead-time)
- Perhaps you have absorbing materials in your sample - leading to **self-attenuation**
- Most modelling procedures make the assumption that the scattered neutrons have been scattered only once by the sample. This is never true - but we still want to try and minimise **multiple scattering** if we can

Correction routines do exist (e.g. in Mantid) in order to correct for sample self-attenuation and multiple scattering, but these aren't commonly used. This is because they are tricky and time-consuming calculations. So most of the time we rely on minimising these effects.

1.1 Scattering units

In principle we can define the sample scattering unit in any way we like. Often it is convenient to use the sample **chemical formula unit** for this. We then can find the cross-sections per formula unit.

For example, the table below shows the cross-sections and formulas weights for one formula unit of poly-ethylene.

	σ_{coh} (b)	σ_{inc} (b)	σ_{abs} (b)	M_f (amu)
C	5.55	0.001	0.3326	12.011
H	1.7568	80.26	0.0035	1.0079
C_2H_4	18.1272	321.042	0.6792	28.0536

σ_{coh} is the **coherent** cross-section
σ_{inc} is the **incoherent** cross-section
σ_{abs} is the **absorption** cross-section (*wavelength dependent)
M_f is the formula weight

The cross-sections per formula unit are just the sum over all the nuclei and can be found in tables on-line.

1.2 Number density and scattering length density

Number of scattering units in the sample (mass, m) is given by

$$N_s = \frac{m}{M_f} N_A \tag{1.1}$$

Where N_A = 6.022 x 10^{23} mol^{-1} is Avogadro's Number. Similarly the number density is given by

$$n_s = \frac{\rho}{M_f} N_A \tag{1.2}$$

where ρ is the density of the sample.

If we express N_A in units of $\times 10^{24}$ mol^{-1} then we get the number density in units of $\times 10^{24}$ cm^{-3} = (barn.cm)$^{-1}$. The reason for the choice of these units will become clear later on.

$$n_s = \frac{\rho}{1.661 M_f}$$
(1.3)

with ρ in units of g cm^{-3} M_f in g mol^{-1} and n_s in (barn.cm)$^{-1}$.

Example:
For poly-ethylene

ρ = 0.93 g cm^{-3}
M_f = 28.054 g mol^{-1}

so $n_s \sim$ 0.02 (barn.cm)$^{-1}$ or 2 x 10^{22} cm^{-3}

The **scattering length density** is defined as the total scattering length per unit volume.

$$\text{SLD} = \frac{\sum_i b_i}{V_m}$$
(1.4)

where the subscript i runs over the number of atoms in the volume of interest, V_m. The dimensions of the SLD are inverse area and given commonly in cm^{-2}. Since the volume V_m is arbitrary, it makes sense to recast it in terms of the mass of a formula unit, M_f, of the material divided by the density ρ.

$$V_m = \frac{M_f}{\rho N_A}$$
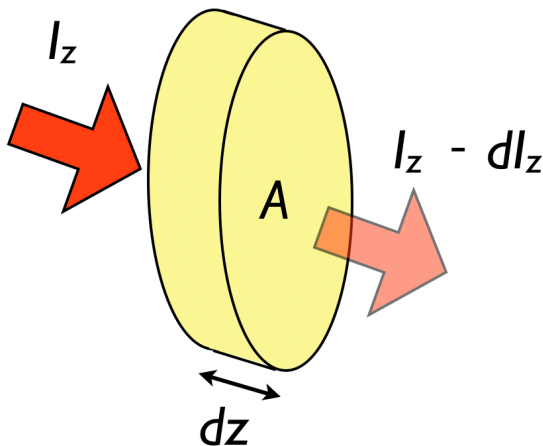(1.5)

The scattering length density can now be written as

$$\text{SLD} = \frac{\rho N_A}{M_f} \sum_i b_i = n_s \sum_i b_i$$
(1.6)

Example:
Poly-ethylene:

SLD $\sim$ 2 x 10^{22} x (2 x 6.65x10^{-13} + 4 x -3.73x10^{-13})
= -3.24x10^{-9} cm^{-2}

1.3 Transmission of neutrons

It is important to know that the chances of a neutron getting through your sample are high. To calculate the transmission of a thin slab-shaped sample, we refer to the figure below of a thin slab of area A, and thickness dz. The neutron intensity incident on the slab is I_z and the transmitted intensity is $I_z - dI_z$.

Transmission through a slab

The slab consists of molecules, with a total cross-section of σ_T per formula unit. Therefore, from the definition of the number density there are

$n_s A\, dz$

formula units in the slab. So the total cross-section of the material is

$\sigma_T\, n_s A\, dz.$

The fraction of untransmitted neutrons is the total absorption cross-section divided by the dimensional cross-section (the area A). Therefore,

$$\frac{dI_z}{I_z} = -\sigma_T n_s dz$$
(1.7)

Integrating both sides of 1.7 with respect to z,

$$\ln(I_z) = -\sigma_T n_s z + C \tag{1.8}$$

For a finite slab of total thickness, *t*, the limits of integration are z=0, *t* with I_0 at z=0 and I_1 at z=t. Therefore,

$$\ln(I_1) - \ln(I_0) = (-\sigma_T n_s t + C) - (-\sigma_T n_s 0 + C)$$
$$= -n_s \sigma_T t \tag{1.9}$$

Finally, we take the exponential of both sides to find the expression for the neutron transmission

$$T = \frac{I_1}{I_0} = \exp(-\sigma_T n_s t) \tag{1.10}$$

This is a form of the Beer-Lambert Law for optical attenuation. For neutrons, the product $\sigma_T n_s$ is termed the *attenuation coefficient* (sometimes denoted by *μ*) and commonly given in units of cm^{-1}.

Example:
Often it is desirable to limit the total scattered fraction of neutrons to less than 10%. This ensures that multiple scattering is a small additional component of the neutron counts.

To calculate how thick the 10% scattering sample should be we set Eq. 1.10 to

$$\exp(-\sigma_S n_s t) = 0.9$$
$$\therefore \sigma_S n_s t \simeq 0.1$$

Where σ_S denotes the total scattering cross-section, and we have assumed no absorption.

So. A 10% scattering poly-ethylene sample with
σ_S = 339.2 barns
n_s = 0.02 (barn cm)$^{-1}$ - we see now the usefulness of this strange unit

Has thickness   t = 0.1 / 339.2 / 0.02 = 0.015 cm

So only around 0.15 mm thickness of poly-ethylene scatters 10% of the beam. This indicates the strength of the scattering of neutrons from hydrogen

1.4 Absorbing materials - optimum sample thickness

There are a few isotopes of elements which prove especially challenging for performing neutron scattering measurements, due to their massive absorption cross-sections. They are - however - often very useful materials use in neutron instrument shielding and detectors.

element	σ_{abs}
Gd	49700 b
B	767 b
Cd	2520 b
Li	70 b
Ir	425 b
Dy	994 b
Sm	5922 b
^{3}He	5333 b

The table on the left shows examples of particularly highly absorbing materials (in their naturally occurring isotopic combinations). These large absorptions are due to nuclear resonances in these materials, and hence the absorption cross-sections are, in general, dependent on the incident neutron wavelength. The tabulated numbers on the left are the σ_{abs} values at a wavelength λ = 1.8 Å.

At low energies σ_{abs} is taken to be linear in wavelength,

$$\sigma_{abs}(\lambda) = \frac{\lambda \sigma_{abs}^{\lambda=1.8}}{1.8} \tag{1.11}$$

Sometimes it is possible to choose a non-absorbing isotope (e.g. B^{11} has a very low absorption cross-section σ_{abs} = 0.005 b).

Some of the above absorber materials, especially Cd and B, are used for shielding instruments and bits of your sample holder that you do not want to scatter from.

Example:
How much Cd is needed to absorb 99.9% of thermal neutrons?

n_s = 0.046 (barn.cm)$^{-1}$
σ_{abs} = 2520 barns

Therefore $\quad$ t = - ln(T) / σ_{abs} / n_s = - ln(0.001) / 115.9 = 0.6 mm

If a sample has some absorption then it's not quite so simple to work out the optimal thickness for scattering as it was for poly-ethylene using Eq. 1.10. In general, it will not be possible to get a 10% scatterer, because the sample will become too thick and the neutrons will not escape. If we assume a uniform shape to the sample (e.g. a slab-shaped sample for SANS or a cylinder for diffraction) then we can make a rough approximation that all neutrons traverse the sample with the same path length.

In that case we can write the fraction of neutrons scattered as

$$\Sigma = T_{\mathrm{abs}} * (1 - T_S) \tag{1.12}$$

Where T_{abs} is the absorption dependent sample transmission given by Eq. 1.10 with the absorption cross-section σ_{abs} and T_S is the scattering dependent transmission given by Eq. 1.10 with the (total) scattering cross-section σ_T. Expanding 1.12 we get

$$\begin{aligned} \Sigma &= T\left(1 - \exp[-n_s\sigma_T t]\right) \\ &= \exp[-n_s\sigma_{\mathrm{abs}}t] - \exp[-n_s(\sigma_{\mathrm{abs}} + \sigma_T)t] \end{aligned} \tag{1.13}$$

So - in order to maximise the scattered fraction of neutrons, we differentiate 1.13 and set to zero to find the turning point and therefore the optimal thickness of the absorbing sample

$$\frac{d\Sigma}{dt} = n_s(\sigma_{\mathrm{abs}} + \sigma_T)\exp[-n_s(\sigma_{\mathrm{abs}} + \sigma_T)t] - n_s\sigma_{\mathrm{abs}}\exp[-n_s\sigma_{\mathrm{abs}}t] = 0$$

$$\Rightarrow \quad \exp[-n_s\sigma_T t] = \frac{\sigma_{\mathrm{abs}}}{\sigma_{\mathrm{abs}} + \sigma_T}$$

$$\Rightarrow \quad t = \frac{\ln(\sigma_{\mathrm{abs}} + \sigma_T) - \ln(\sigma_{\mathrm{abs}})}{n_s\sigma_T} \tag{1.14}$$

Example:
Manganese metal has a moderately large absorption cross-section of 13.3 b @ 1.8 Å. Suppose we are doing an experiment on the IN5 time-of-flight spectrometer at the ILL with a incident neutron wavelength of λ = 5.1 Å. What in this case would be the optimum sample thickness?
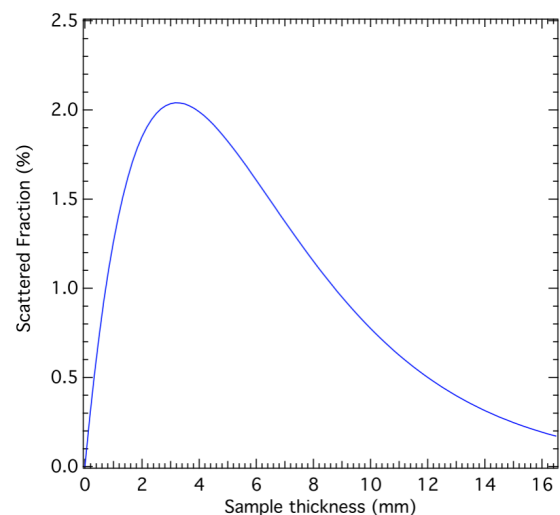
σ_{abs} = 13.3 * 5.1 / 1.8 = 37.7 barns
σ_T = 2.15 barns
n_s = 0.08 (barn cm)$^{-1}$

Therefore $\quad t = \dfrac{\ln(37.7 + 2.15) - \ln(37.7)}{0.08 * 2.15}$

$\qquad\qquad$ = 3.2 mm

This occurs at a sample transmission of 38%
For broad wavelength bands - optimise for maximum desired wavelength.

Scattered neutrons from Mn metal at λ = 5.1 Å

1.5 Optimising the sample shape

So far we have considered only the transmission of the direct beam at zero scattering angle. However what we *really* need to know is what is the transmission of the scattered neutrons *as a function of scattering angle.* In order to calculate that we need to know the shape of the sample accurately, and then average over all the possible scattering paths.

As an example (and the only example which is tractable) we take the case of a slab shaped sample (which we assume is much wider than it is thick).

The incident neutrons make an angle γ with the face of the slab, and the neutrons scatter from a thin layer of thickness dx at a depth x within the slab.

We can write the initial and final path lengths before and after scattering as

$$L_1 = \frac{x}{\sin\gamma}$$

$$L_2 = \frac{t - x}{\sin(\gamma - 2\theta)}$$

The incoming flux ϕ_0 is attenuated along L_1

$$\phi = \phi_0 \exp(-n_s\sigma \, L_l)$$

$$= \phi_0 \exp\left(\frac{-n_s\sigma x}{\sin\gamma}\right)$$

Scattering from an "infinite" slab

The outgoing beam is attenuated along L_2 (and here we assume that the wavelength doesn't change)

$$T_2 = \exp\left(-n_s\sigma\frac{t - x}{\sin(\gamma - 2\theta)}\right)$$

Outgoing number of neutrons is proportional to cross-section into solid angle $\Delta\Omega$, flux, n_s, thickness of slab Δt and is attenuated along L_2

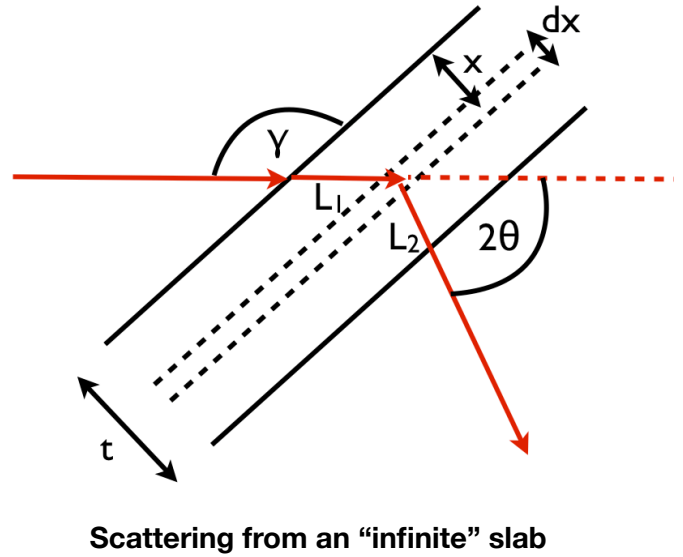$$\Delta N = \phi n_s \Delta t \left(\frac{d\sigma}{d\Omega}\right) T_2 \Delta\Omega$$

$$= \phi_0 n_s \exp\left(-\frac{n_s\sigma x}{\sin\gamma}\right)\frac{dx}{\sin\gamma}\left(\frac{d\sigma}{d\Omega}\right)\exp\left(-\frac{n_s\sigma(t - x)}{\sin(\gamma - 2\theta)}\right)\Delta\Omega$$

So total neutrons scattered is found by integrating above wrt. x between *x=0* and *x=t*, and then the transmission is given by dividing by the total neutrons scattered in the limit of zero-absorption, N_0. Finally we end up with
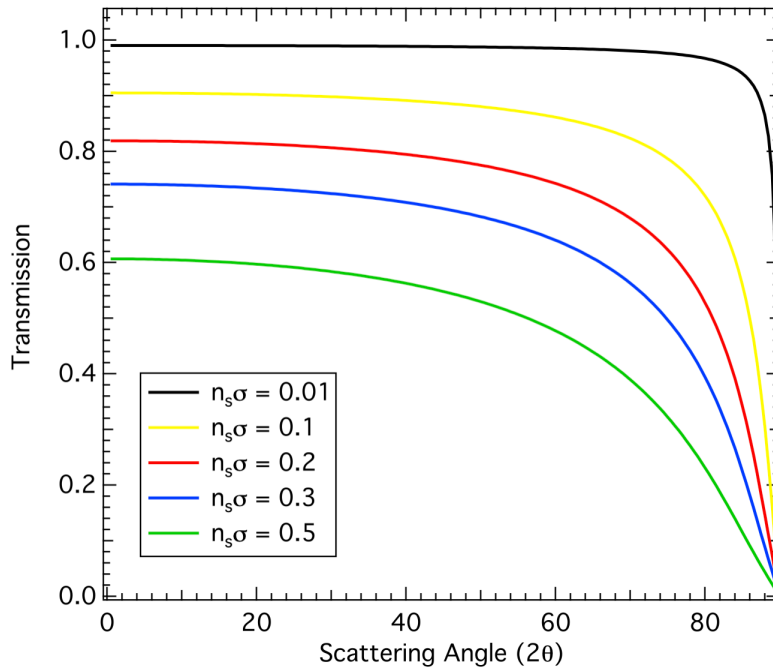
$$T = \frac{1}{n_s\sigma t(\operatorname{cosec}\gamma - \operatorname{cosec}(\gamma - 2\theta))}\left\{\exp\left(-\frac{n_s\sigma t}{\sin(\gamma - 2\theta)}\right) - \exp\left(-\frac{n_s\sigma t}{\sin\gamma}\right)\right\} \quad (1.14)$$

This is one of the very few solvable cases for transmission as a function of scattering angle. And, as you can see it is a complex expression. It also assumes that the slab is infinitely wide, which is of course not really the case.

Below is plotted the transmission given by expression 1.14 for the case of a monochromatic beam at normal incidence - i.e. $\gamma = 90°$ with a thickness of 1 cm, and a range of values of the attenuation coefficient of between 0.01 and 0.5 cm^{-1}.

Transmission of 1cm thick slab at normal incidence to the beam

At low angles the 2θ dependence is weak with the transmission being well approximated by the zero-angle transmission. However, as the scattering angle approaches 90° there is a very strong absorption. For this reason, slab shaped samples are popular in SANS experiments, and often in neutron spin-echo measurements. Beyond 2θ = 90° there is different expression for the transmission.

Many neutron instruments have large detector arrays which commonly surround the sample (in the equatorial plane), *e.g.* powder diffractometers, backscattering, time-of-flight spectrometers, etc. Cylindrical sample geometries are most adapted to these. Sometimes an annular cylinder is used to fill the beam, but minimise absorption. In these cases there is no analytical expression for the transmission as a function of angle - it must be calculated numerically (i.e. lookup tables or Monte-Carlo algorithms are commonly used)

In the case of a solid cylinder or sphere with an attenuation coefficient of < 1 cm⁻¹, a good approximation (better than 0.5% accuracy) is given by the expression

$$T = \exp\left\{-(a_1 + b_1 \sin^2 \theta)n_s\sigma R - (a_2 + b_2 \sin^2 \theta)(n_s\sigma R)^2\right\} \qquad (1.15)$$

Where R is the radius of the cylinder / sphere and the coefficients a_1, b_1, a_2 and b_2 have the
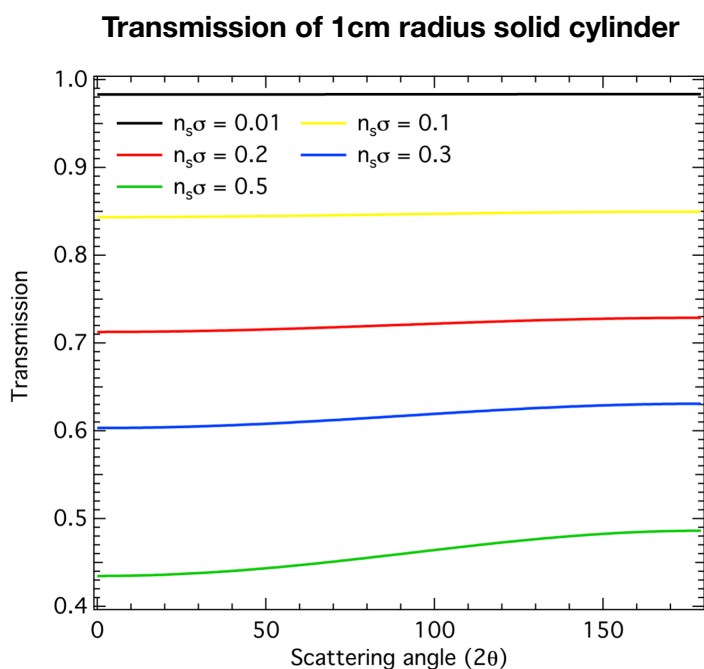
	a_1	b_1	a_2	b_2
cylinder	1.7133	-0.0368	-0.0927	-0.3750
sphere	1.5108	-0.0315	-0.0951	-0.2898

values,
See, A Hewat, Acta. Cryst. A **35** (1975) p248

This gives a much flatter transmission as a function of angle, and so is a good sample shape for neutron instruments which count neutrons over a wide angular range. Commonly, the sample transmission is assumed to be constant over the whole range of scattering angles in this

geometry. Below is the transmission through a 1cm radius cylinder for a range of attenuation coefficients between 0.01 and 0.5 cm^{-1}

Transmission of 1cm radius solid cylinder

1.6 Sample Holders

Choice of material of the sample holders / containers is crucial and depends on the type of neutron measurement being attempted. This also applies to external sample environments (such as cryostats, pressure cells, etc.)

Ideally the sample holders and sample environment should be made as thin as possible to reduce the mass of extraneous materials in the beam. They should also be shielded where possible using absorbing materials such as Cd or B_4C (boron carbide).

The table below shows a selection of sample holders for different applications.

	Material	Comment
Powder diffraction	vanadium	no Bragg peaks, large σ_{inc} some absorption
Inelastic scattering	Al / Cu	low scattering cross-sections, low incoherent scattering (Cu for low T)
Polarized neutrons	Al / Cu	
Liquids / glasses	vanadium / TiZr	no Bragg peaks TiZr is a "null-matrix"
SANS	Quartz	very low small angle scattering
Single crystals	Small pin (Al)	sample holds own shape

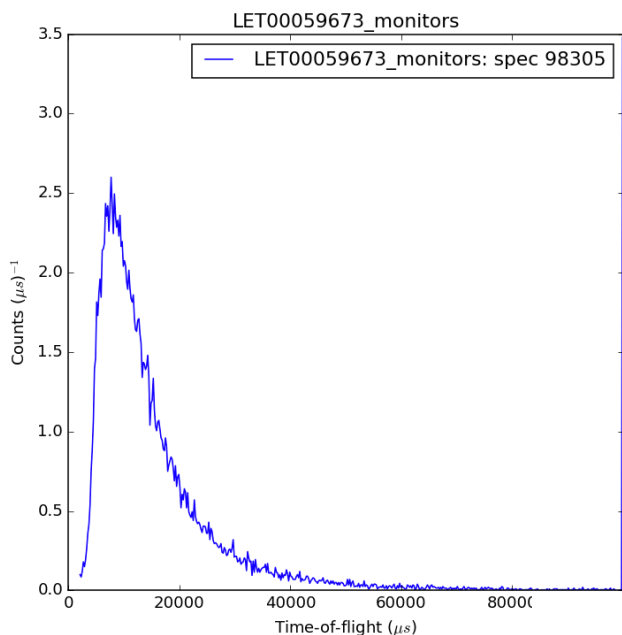# 2. During the experiment: Counting Neutrons

2.1 Neutron Histograms.

Neutron instruments include detectors which count neutrons as a function of scattering angle and/or neutron time-of-flight.   Note that the scattering angle is in general composed of two variables - in the case of spherical polar coordinates these are the in-plane and azimuthal angles θ and ϕ. In both of these cases the neutron data is stored - in memory and on disk - in the form of **a histogram**.

A histogram is a representation of data in which the data is sorted in increasing order as a function of some continuous variable, but which as been subdivided into predetermined intervals - often called **ranges** or **bins**. There is a mini-tutorial on histograms at:
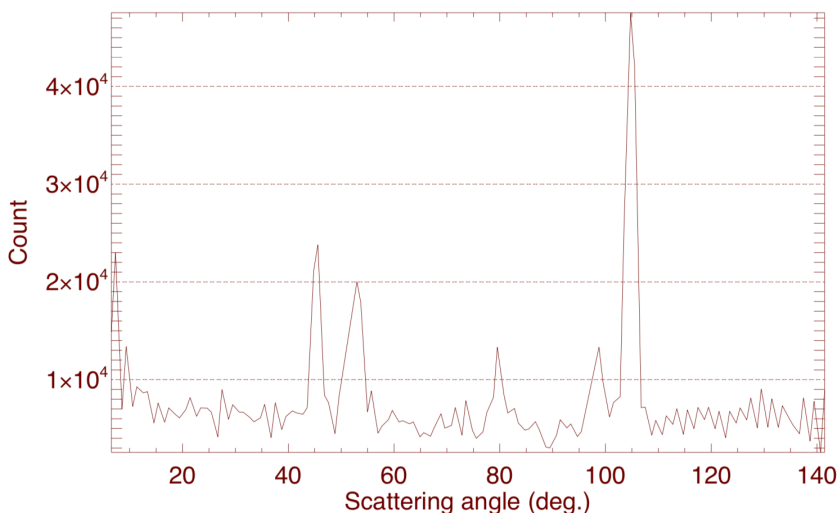https://www.mathsisfun.com/data/histograms.html

The detector solid angle element, dΩ, naturally defines a histogram bin width. Remember that the solid angle is just defined by an area (in this case, the detector window) divided by the squared distance between the sample and the detector.   For time-of-flight the histogram bins are determined by the counting electronics - and can generally be set as broad or as narrow as desired. There is no requirement that the histogram bin ranges be constant, though in practice they often are. If the bin-widths are not the same, then the neutron counts must be divided by the bin widths to create a **distribution function**.

LET00059673_monitors

This is a neutron time-of-flight distribution from LET (ISIS). It is a distribution because the neutron counts have been divided by the time-of-flight bin width (giving units of μs^{-1}). The bin width is 200 μs.

This is a neutron scattering angle histogram from D7 (ILL). The Y-axis is just raw neutron counts, and the scattering angle bin size is ~1°

2.2 Systematic error and random uncertainty

The measured histograms in a neutron experiment are subject to experimental error and uncertainty.

Systematic error can arise due to bad calibration and setup of the detectors (e.g. non-linearity), incorrect calibration of the detector angles or the time-of-flight counting electronics. It could even be said that it could be due to a bad sample…

The best way to check for systematic errors in your measurement **is to repeat it**. Perhaps on a different batch of sample and/or on a different instrument. Most rigorous is **independent corroboration** of the measurement. Unfortunately, independent corroboration and experimental repetition is very rare in the neutron scattering - since beam time is scarce and priority is given to experiments not already attempted.

Random uncertainty (often this is incorrectly called random error) is due to the unpredictability of the measurement. In the case of neutron scattering, we cannot predict in advance how many neutrons will be scattered into a detector in a certain time interval. We can only provide a probability that this will occur. The way to reduce random uncertainty in such a measurement is to repeat the measurement many times - and build up an average number of neutron counts per bin, and a spread in that value. The more times we repeat the measurement, the smaller the spread in the measured values. Multiple repetitions of a measurement build up a so-called *probability distribution* - of which the measured values are random samples.

2.3 Probability distributions - Expectation, Variance, Covariance

Neutrons arriving in a detector in a certain time interval (the *counting time*) can be considered as random samples of a probability distribution *p(n)*, where *n* is the count number and is therefore always an integer.

The **expectation of n** - also called the mean value of the probability distribution is given by

$$\mu = E[n] = \sum_{n=0}^{\infty} n p(n)$$

(2.1)

The **variance** of the distribution is defined as the expectation of the squared difference from the mean

$$\sigma^2 = E[(n-\mu)^2] = \sum_{n=0}^{\infty} (n-\mu)^2 p(n)$$

(2.2)

The square root of the variance of the distribution is called the **standard deviation** and it is this that is used as an estimate of the random uncertainty.

Lastly - imagine that we have performed two measurements and we want to add them up.

First measurement: mean: μ_1, variance: σ_1^2
Second measurement: mean: μ_2, variance: σ_2^2
Summed measurements: mean: μ_T, variance: σ_T^2

The expectation of the summed measurement is:

$$\mu_T = E[n_1 + n_2] = \sum_{n_1, n_2} (n_1 + n_2) p(n_{1,2})$$
$$= \sum_{n_1} n_1 p(n_1) + \sum_{n_2} n_2 p(n_2)$$
$$= \mu_1 + \mu_2$$

(2.3)

While the variance of the summed measurement is:

$$
\begin{aligned}
\sigma_T^2 = E[(n_T - \mu_T)^2] &= E[(n_1 - \mu_1 + n_2 - \mu_2)^2] \\
&= E[(n_1 - \mu_1)^2 + (n_2 - \mu_2)^2 - 2(n_1 - \mu_1)(n_2 - \mu_2)] \\
&= \sigma_1^2 + \sigma_2^2 + 2E[(n_1 - \mu_1)(n_2 - \mu_2)] \\
&= \sigma_1^2 + \sigma_2^2 + 2\,\mathrm{cov}(n_1, n_2)
\end{aligned}
\tag{2.4}
$$

where cov(n_1,n_2) is the called the covariance of n_1 and n_2.

We can simplify the expression for the covariance:

$$
\begin{aligned}
\mathrm{cov}(n_1, n_2) &= E[(n_1 - \mu_1)(n_2 - \mu_2)] \\
&= E[n_1 n_2 - n_1 \mu_2 - n_2 \mu_1 + \mu_1 \mu_2] \\
&= E[n_1 n_2] - E[n_1]E[n_2] - E[n_1]E[n_2] + E[n_1]E[n_2] \\
&= E[n_1 n_2] - E[n_1]E[n_2]
\end{aligned}
\tag{2.5}
$$

If the two measurements, n_1 and n_2 are independent, then the covariance is zero. This is because two variables are said to be independent if their joint expectation is equal to the product of their expectation. In this case;

$$
E[n_1 n_2] = E[n_1]E[n_2]
\tag{2.6}
$$

Following this, we can say, from the definition of the variance above, that if two variables are independent and are added (or subtracted), then their uncertainties **add in quadrature**

$$
\sigma_T^2 = \sigma_1^2 + \sigma_2^2
\tag{2.7}
$$

2.4 Combining measurements

In order to combine the results from various measurements in different ways, we start with the expression for the total differential.

$$
df = \sum_i \frac{\partial f}{\partial x_i} dx_i
\tag{2.8}
$$

where f is a function of variables x_1, x_2, ... In what follows we take f to be a combination of neutron measurements, each of which is independent from the other so that we can take the covariance to be zero. Under this assumption (and also, making the assumption that the uncertainties are small - for a first order expansion) we can take the square of Eq. 2.8 to obtain

$$
(df)^2 = \sigma_f^2 = \sum_i \left(\frac{\partial f}{\partial x_i} \right)^2 \sigma_{x_i}^2
\tag{2.9}
$$

2.4.1 Addition / Subtraction
This is just a check on what we did in the previous section. Two measurements n_1 and n_2 added together.

$$
\begin{aligned}
f &= n_1 \pm n_2 \\
\partial f / \partial n_1 &= 1 \\
\partial f / \partial n_2 &= 1 \\
\therefore \ \sigma_f^2 &= \sigma_{n_1}^2 + \sigma_{n_2}^2
\end{aligned}
\tag{2.10}
$$

2.4.1 Multiplication (or division) by a constant
This happens when we **normalise** data (against monitor counts or time for example; see §3.1).

$$
\begin{aligned}
f &= k * n_1 \\
df / dn_1 &= k \\
\therefore \ \sigma_f &= k * \sigma_{n_1}
\end{aligned}
\tag{2.11}
$$

2.4.2 Multiplication or division of measurements

Multiplication of neutron measurements is not a common operation… but division is very common. Neutron data is very commonly divided by a vanadium measurement. It is used for correcting the data for detector efficiency, solid angle and flux profile (see §3.2)

Let's deal with multiplication first.

$$f = n_1.n_2$$
$$\partial f / \partial n_1 = n_2$$
$$\partial f / \partial n_2 = n_1$$
$$\therefore \; \sigma_f^2 = n_2^2 \sigma_{n_1}^2 + n_1^2 \sigma_{n_2}^2$$

(2.12)

We can divide both sides by f^2 to obtain

$$\left(\frac{\sigma_f}{f} \right)^2 = \left(\frac{\sigma_{n_1}}{n_1} \right)^2 + \left(\frac{\sigma_{n_2}}{n_2} \right)^2$$

(2.13)

So, in the case of multiplied measurements the combined **fractional uncertainty** is the addition of the individual fractional uncertainties in quadrature.

For the case of division of data sets,

$$f = \frac{n_1}{n_2}$$
$$\partial f / \partial n_1 = 1/n_2$$
$$\partial f / \partial n_2 = -n_1/n_2^2$$
$$\therefore \; \sigma_f^2 = \frac{\sigma_{n_1}^2}{n_2^2} + \frac{n_1^2 \sigma_{n_2}^2}{n_2^4}$$

(2.14)

This looks a lot more complicated, until we again divide both sides by f^2 to give

$$\left(\frac{\sigma_f}{f} \right)^2 = \left(\frac{\sigma_{n_1}}{n_1} \right)^2 + \left(\frac{\sigma_{n_2}}{n_2} \right)^2$$

which is Eq. 2.14 again. So in the case of both multiplication and division of measurements, the **fractional uncertainties** are added in quadrature.

2.5 The Poisson distribution and error estimation

If we repeat a neutron measurement a few times how do we calculate the mean and the variance. Both of these depend on the shape of the probability distribution *p(n)* which (remember) is the probability of counting *n* neutrons as a function of *n*.

Whenever the measurement involves counting objects (like the number of flowers in a field per unit area, or the number of neutrons in a detector per unit time), then *p(n)* takes the form of the Poisson distribution.
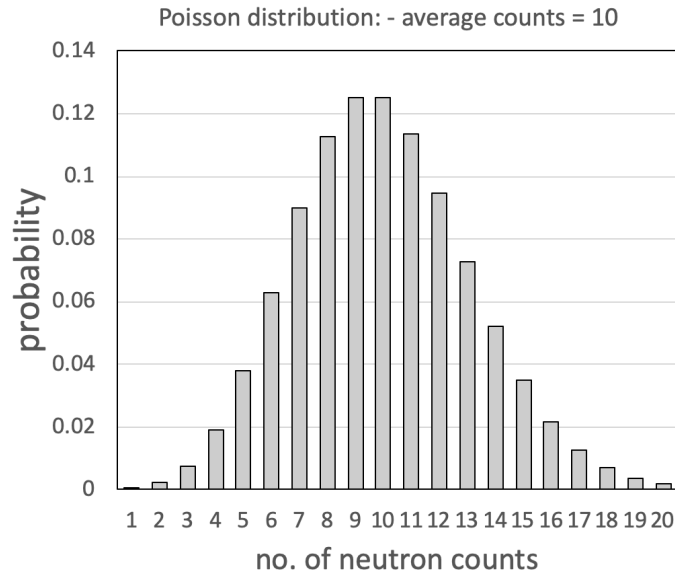
$$p_P(n) = \frac{\mu^n \exp(-\mu)}{n!}$$

(2.15)

Then integrating this according to Eqs. 2.1 and 2.2 we find the mean and variance,

$$\mu_P = \mu \quad \text{(by definition)}$$
$$\sigma_P^2 = \mu$$

(2.16)

This means that the standard deviation estimate of the uncertainty in neutron counts is just given by the square-root of the number of counts.

Below is a plot of the Poisson distribution giving the probability that exactly n neutrons are counted in a given time interval, assuming that the average count-rate is 10 neutrons per time interval.

Poisson distribution: - average counts = 10

In this case the estimated standard error would be $\pm\sqrt{10} = 3.16$ neutrons per time interval.

Obviously the longer we count (in other words, adding up consecutive time intervals) the larger the average number neutrons and uncertainty. However the fractional uncertainty, which for the Poisson distribution is given by

$$\frac{\sqrt{N}}{N} = \frac{1}{\sqrt{N}}$$

(2.18)

where N is the total (integrated) number of neutrons counted (i.e. the un-normalised mean over many small time intervals), decreases (slowly) as N increases.

Example:
Some useful numbers to remember about fractional uncertainty in neutron scattering....
If you want to count until the fractional uncertainty is 3% (0.03) then the number of counts required is

$$\frac{1}{\sqrt{N}} = 0.03$$
$$\therefore \ N \simeq 1100$$

Say now that we have decided we actually want to achieve a smaller uncertainty by a factor of 2, i.e. 1.5% (0.015). This means we need

$$\frac{1}{\sqrt{N}} = 0.015$$
$$\therefore \ N \simeq 4400$$

counts. *So we have to count 4 times longer to halve the experimental uncertainty*.

3. After the experiment: Data Correction

The correction of neutron data is primarily intended to deal with sources of systematic error. The aim is to turn the measured neutron intensities into measured neutron cross-sections - sometimes on an absolute scale. Do do this we need to know things like -
- how long did we count for?
- How big are our detectors
- what is our sample mass / thickness / volume?
- are there any sources of background counts?
- what is the sample transmission?

The expression for the total number of counts is,

$$N = \phi E(\lambda) \Delta\Omega n_s t \left(\frac{d\sigma}{d\Omega} \right) T \tag{3.1}$$

N		[neutrons]
ϕ	incident neutrons	[neutrons]
$E(\lambda)$	detector efficiency	[dimensionless]
$\Delta\Omega$	detector solid angle	[dimensionless]
n_s	number density	[e.g. (barn.cm)$^{-1}$]
t	sample thickness	[cm]
$d\sigma/d\Omega$	cross-section	[barns]
T	transmission	[dimensionless]

In this part of the lecture we will go through the very basics of correcting neutron data - both at pulsed and continuous sources. Much data correction is done using pre-made scripts in whatever analysis package is available (e.g. Mantid, LAMP, DAVE…).

3.1 Normalisation to monitor / time / current

The total number of neutrons counted in a measurement will depend on uninteresting factors. The least interesting of these is ϕ, the number of incident neutrons. Obviously the longer we count for, or the higher the incident flux, the more neutrons we will count. So we need to divide the measured neutron counts by a number, which indicates how many neutrons were available to be scattered.

Option 1: Counting Time
If the flux of the neutrons can be taken to be constant in time over the period of the measurement, then it is acceptable to divide the neutron counts by the counting time. However this is only really an option on continuous sources such as the ILL. On spallation sources, the neutron beam fluxes can be quite variable over the experiment.

Option 2: Integrated proton beam current
At a spallation source such as ISIS, a common method to normalise data is to use the integrated proton beam current to normalise the data. The beam current is generally recorded in units of µA or mA, and the time integral of this (which is the total proton charge) is recorded (units of µA.Hrs). Assuming that the proton monitors accurately reflect the current and integrated charge, then this can be used to normalise the data. This method is very commonly used at ISIS.

Option 3: Neutron Incident Beam Monitor
In most neutron spectrometers, there is a low efficiency detector placed upstream of everything else. This is called the incident beam monitor and it typically has a counting efficiency of around 1 neutron in every 10 000. On continuous sources this monitor will accurately reflect the integrated number of neutrons incident on the sample during the measurement. On pulsed sources, this monitor will - in addition - give the time-of-flight (wavelength) profile of the incident beam. Normalisation to monitor is arguably the most reliable method of the 3 choices, depending on the stability in time of the monitor.

3.2 Normalisation of the detector

The detector solid angle, $\Delta\Omega$, the wavelength dependent efficiency, $E(\lambda)$, and the wavelength dependent (and now normalised) intensity, $\phi_0(\lambda)$, can be removed from Eq. 3.1 by dividing by a flat scatterer; that is, a sample whose angle (and wavelength) dependent differential cross-section, $d\sigma/d\Omega$, is a constant.

$$N_{\mathrm{norm}} = \frac{\phi_0(\lambda)E(\lambda)\Delta\Omega n_s t \left(\frac{d\sigma}{d\Omega}\right) T}{\phi_0(\lambda)E(\lambda)\Delta\Omega * \mathrm{constant}}$$

$$= \frac{n_s t \left(\frac{d\sigma}{d\Omega}\right) T}{\mathrm{constant}} \tag{3.2}$$
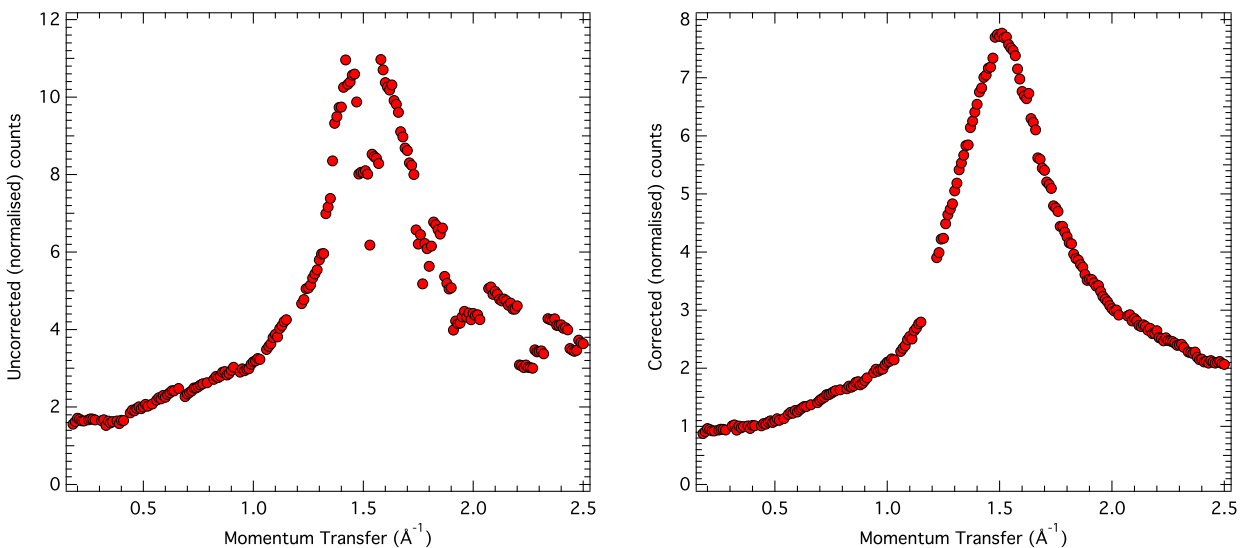
For most applications, the flat scatterer is chosen to be vanadium. As shown in the table below, the scattering from vanadium is dominated by incoherent scattering (due to the nuclear spin), and is therefore flat.

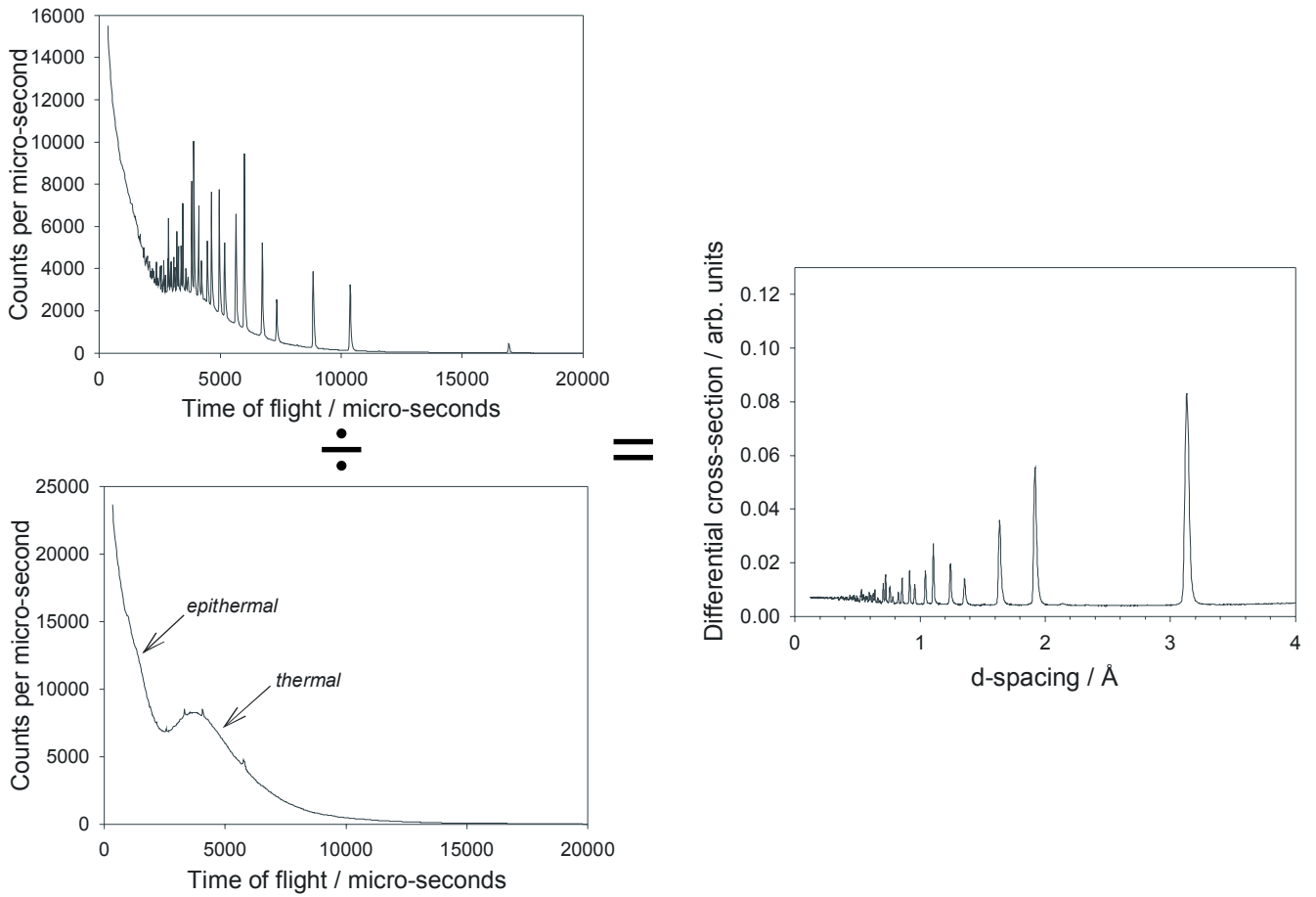|	abund. %	σ_{coh}	σ_{inc}	σ_{abs}
^{50}V	0.25	7.3	0.5	60
^{51}V	99.75	0.0203	5.07	4.9
V	-	0.038	5.06	5.04

Unfortunately, vanadium cannot be used to normalise SANS or reflectivity data since there is a lot of small-angle scattering from grains and oxide layers in metallic vanadium. For SANS instruments the scattering from water is sometimes used (flat at low Q), while for reflectivity, glassy carbon can be used.

Examples:
Vanadium normalisation on D7 at the ILL. Amorphous silica before and after division by a vanadium measurement. D7 is a monochromatic instrument, so the correction is mostly to do with differences in solid angle. Each individual detector has a different effective solid angle and efficiency - so the data before correction looks pretty ugly.

Vanadium normalisation on POLARIS at ISIS. Below we see the sample (silicon powder) and vanadium spectra as a function of time-of-flight. We are seeing only one detector spectrum (grouped). So this time the correction is mainly to do with energy efficiency and wavelength dependent neutron intensity. The corrected spectrum has been converted into d-spacing.

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3.3 Absolute normalisation

The cross-section and number density of vanadium is well known,

$(d\sigma/d\Omega)_V$ = 5.06 / 4π (barns st^{-1} f.u^{-1})

n_s = 0.0685 (barn cm)$^{-1}$

therefore we should be able to work out the constant term in Eq. 3.1 provided we know the vanadium thickness (and therefore the transmission).

$$\frac{N_{\text{sample}}}{N_{\text{vanadium}}} = \frac{n_s t \left(\frac{d\sigma}{d\Omega}\right) T}{0.0685 \ t_V \frac{5.06}{4\pi} T_V} \tag{3.2}$$

where T_V is the vanadium transmission and t_V is the vanadium thickness. Re-arranging we have,

$$\frac{d\sigma}{d\Omega} = \frac{N_{\text{sample}}}{N_{\text{vanadium}}} \frac{0.0685}{n_s} \frac{T_V}{T} \frac{t_V}{t} \frac{5.06}{4\pi} \tag{3.3}$$
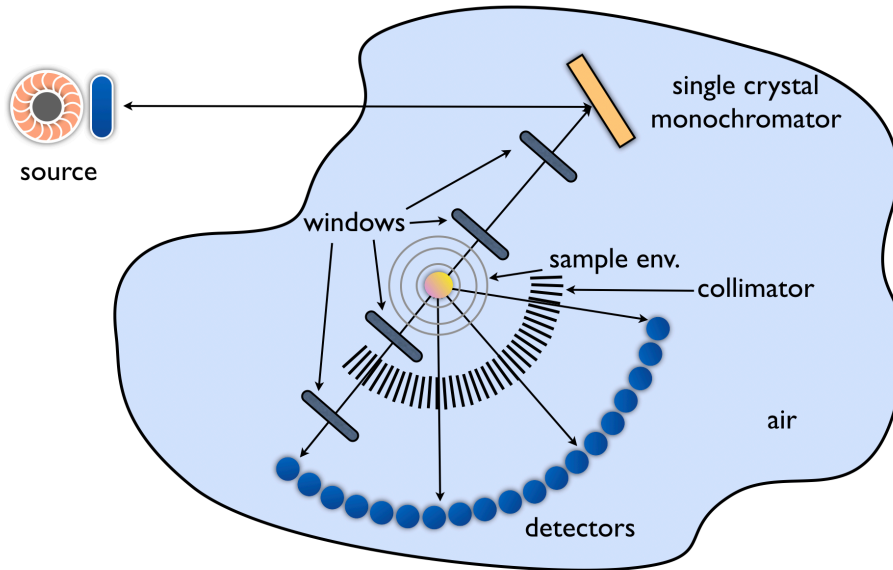
The ratio of sample and vanadium thicknesses and number-densities can be replaced by masses and formula weights if required.

3.4 Background Subtraction

There are many possible sources of background scattering in a typical neutron instrument. These can include; cosmic ray background scattering; neutrons escaping from the source; vacuum windows (often made of aluminium) in the beam path; sample environment such as cryostats, magnets, pressure-cells; sample holders; air in the beam path; …

These are depicted in the diagram below of a typical continuous source powder diffractometer.

Depiction of a typical continuous source powder diffractometer

One might expect that in order to subtract the background scattering, all you need to do is measure an **empty sample holder** in exactly the same conditions as the sample measurement (i.e. **filled sample holder**). This is in fact true for samples which have a very good neutron transmission.

In general though the measured neutron counts with a sample in the beam is a sum of three terms,

$$N_m = T_s N_s + T_s N_{bg_A} + N_{bg_B} \tag{3.4}$$

where, N_m is the measured count, N_s is the count due to sample scattering, N_{bgA} is the count due to background sources which have passed through the sample and is hence attenuated, and N_{bgB} is the count due to background sources which bypass the sample (unattenuated). Since N_s and N_{bgA} are attenuated by the sample they have to be multiplied by the sample transmission T_s. This is depicted in Fig. (a) below.
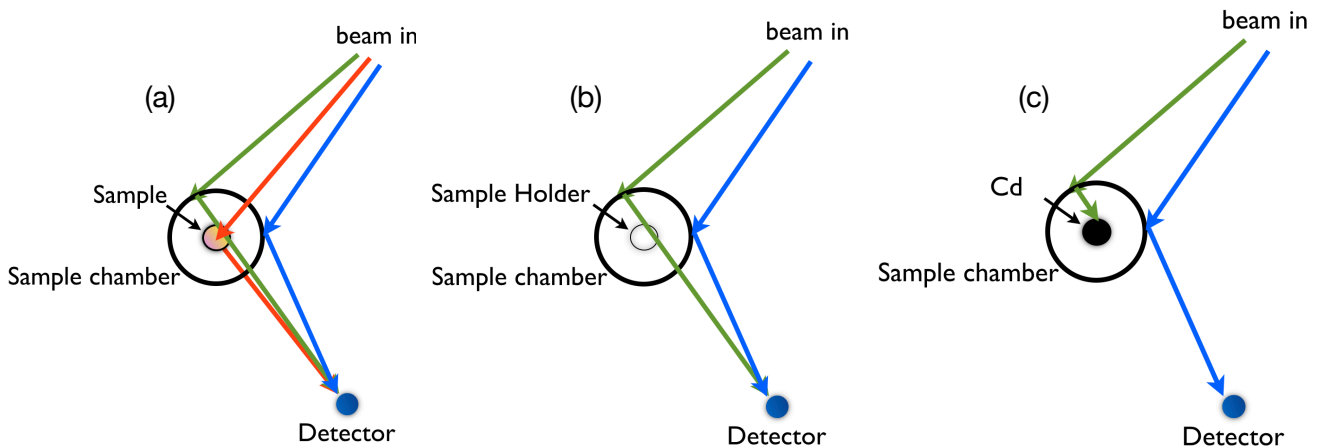

Fig. (b) depicts the situation for an empty sample holder measurement. In this case we are measuring both the N_{bgA} and N_{bgB} contributions - but the N_{bgA} term is no longer attenuated by the sample. Therefore the empty sample holder counts, N_E are;

$$N_E = N_{bg_A} + N_{bg_B} \tag{3.5}$$

Fig. (c) shows the situation when we measure a **totally absorbing** sample (such as Cd) in the sample holder. In this case, the N_{bgA} background term is blocked by the sample, giving;

$$N_{Cd} = N_{bg_B} \tag{3.6}$$

Combining Eqs. 3.5 and 3.6 we can write,

$$N_{bgA} = N_E - N_{Cd} \qquad (3.7)$$

And therefore re-arranging 3.4 and substituting 3.7 and 3.6 we get,

$$N_m = \frac{1}{T_s}(N_s - N_{cd}) - (N_E - N_{Cd}) \qquad (3.8)$$

Note that when the sample transmission is close to 1, then the N_{Cd} terms subtract, and all that is required is the empty sample holder measurement. Sometimes, N_{Cd} is quite close to zero (e.g. in SANS experiments) but it is still checked. In wide angle diffraction experiments N_{Cd} can be very large (almost as large as N_E).