Small Angle Neutron Scattering

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Introduction

Small angle scattering (SAS) is the collective name given to the techniques of small angle neutron (SANS), X-ray (SAXS) and light (SALS, or just LS) scattering. In each of these techniques radiation is elastically scattered by a sample and the resulting scattering pattern is analysed to provide information about the size, shape and orientation of some component of the sample.

The type of sample that can be studied by SAS, the sample environment that can be applied, the actual length scales that can be probed and the information that can ultimately be obtained, all depend on the nature of the radiation employed. For example, LS cannot be used to study optically opaque samples and SAXS cannot (easily) be employed to study thick samples or samples requiring complex containers, whilst SANS (and SAXS) probe different length scales to LS. Thus to a large extent these techniques are complementary. They do, however, also share several similarities. Perhaps the most important of these is the fact that, with minor adjustments to account for the different types of radiation, the same basic equations and "laws" (for example, those due to Guinier, Zimm, Kratky and Porod) can be used to analyse data from any of the three techniques. This is a tremendous advantage and one that has certainly eased the transition from one technique to another for thousands of students over the years.

In this review it is only going to be possible to give a brief overview of SANS. Sadly, the standard textbook on SANS has yet to be written, but many excellent texts on LS and SAXS do exist[1-5], as do texts on neutron scattering in general[6-12]. The interested reader is urged to consult these for further information. There are also several texts and papers dealing with the application of SANS in colloid and polymer science[13-20].

A much more comprehensive version of this article, complete with experimental protocols and data analysis methods is now in print as Chapter 7 in* Modern Techniques for Polymer Characterisation, *R A Pethrick & J V Dawkins (editors), John Wiley, 1999 (ISBN 0-471-96097-7). This book also contains similar chapters on Neutron Reflectivity and Inelastic Neutron Scattering. Though primarily aimed at polymer scientists the articles are sufficiently general that they should prove of use in other areas.

STOP PRESS: Those specifically interested in solution scattering from biological macromolecules may like to consider* Small Angle X-Ray and Neutron Scattering from Solutions of Biological Macromolecules, *D I Svergun, M H J Koch, P A Timmins & R P May, OUP, 2013 (ISBN 978-0-19-963953-3).

The Properties Of Neutron Radiation

Neutron radiation can be produced to cover a range of wavelengths; 0.01 - 3 nm would not be untypical. This range is comparable to that which may be obtained with X-rays (for example, the Cu-Ka line at 0.15 nm) but is orders of magnitude smaller than that of visible light (400 - 700 nm). The usefulness of SAS to colloid and polymer science becomes clear when one considers the length scales involved; bond lengths are typically around 0.1 nm, the radius of gyration of a polymer in solution is usually 1 - 10 nm, a surfactant micelle may be 10 - 100 nm in diameter, whilst latex particles and emulsion droplets are often 100 - 1000 nm in diameter.

In the case of electromagnetic radiation, energy, E, and wavelength, λ , are related through Planck's equation

$$E = hc / \lambda \quad \text{... (1)}$$

but because the neutron has a finite mass (m = 1.674′ 10^{-27} Kg) it is necessary to consider its kinetic energy instead, given by

$$E = h^2 / 2m\lambda^2 = mv^2 / 2 \quad \text{... (2)}$$

where E is in Joules, λ is in metres, v is the neutron velocity in ms^{-1}, c = 2.997′ 10^8 ms^{-1} and Planck's Constant, h = 6.626′ 10^{-34} Js. Thus, a neutron with a wavelength of 0.15 nm has an energy of 5.83′ 10^{-21} J or, in more practical units, 36.4 meV. By contrast, the energy of a 0.15 nm X-ray photon is ~ 8.2 keV, more than 200,000 times greater than the energy of the neutron. Quite apart from the effects that radiative heating may have, depositing this much energy in a sample can easily bring about serious molecular degradation; the C-C bond energy is only ~ 4 eV. Neutrons therefore have a particular advantage over X-rays in the study of sensitive samples, such as biological material for example. Substituting the kinetic energy of the neutron into Equation 1 and solving for λ yields an *equivalent wavenumber* for the 0.15 nm neutron of ~ 293 cm^{-1}. This value is comparable to those of typical IR/Raman vibrational modes and demonstrates that neutrons can also be used to probe the dynamics of a sample, an aspect of neutron scattering that will not be discussed further in this review but which is more than adequately covered by the earlier references.

The most fundamental difference between neutron and electromagnetic radiation is the mechanism by which the incident radiation interacts with matter. Light and X-rays are both scattered by the electrons surrounding atomic nuclei, but neutrons are scattered by the nucleus itself. This single fact has several important consequences.

In the case of light or X-rays, the scattering cross-section of an atom (a concept which may be likened to the collision cross-section encountered in the classical derivation of chemical kinetic theory) increases in direct proportion to the number of electrons present; that is, it increases with increasing atomic number, Z. However, the strength of the neutron-nucleus interaction varies completely irregularly with Z; not even isotopes of the same element have the same *neutron scattering cross-section*, σ . The most significant isotopic variation occurs when Z = 1. Hydrogen has a (coherent[21]) σ_{coh} of 1.75′ 10^{-24} cm^2 (or, in physicists units, 1.75 barns) which is roughly the same as that of manganese. On the other hand, for deuterium σ_{coh} = 5.6 barns, similar to the value for carbon-12. Thus, and unlike X-rays, not only can neutrons "see" hydrogen isotopes, but they can differentiate between them. As will be shown later, this simple fact is pivotal to getting the most out of a SANS experiment. A selection of neutron scattering cross-sections, and *neutron scattering lengths, b*, are given in Table 1.[22] Where no atomic number is given, natural isotopic abundance is assumed.

Because atomic nuclei are some 10^4 - 10^6 times smaller than typical neutron wavelengths, the nuclei effectively act as point scatterers. The result of this is that the nuclear scattering remains constant as the scattering angle increases, allowing scattering patterns to be collected over the full range from forward to backward angles. The scattering is spherically symmetric. This is in marked contrast to the case with X-rays where atomic diameters are only 0.1 - 10 times typical wavelengths, resulting in a decrease in the scattering with increasing angle.

The interaction of neutrons with matter is weak and the absorbtion of neutrons by most materials is correspondingly small. Neutron radiation is therefore very penetrating. For example, it would require X-rays with energies of some 10^5 eV to penetrate a sample (and its container) more than a millimetre or two thick. Neutrons, on the other hand, can be used to probe the bulk properties of samples with pathlengths of several centimetres or, alternatively, samples with somewhat shorter pathlengths but contained inside complex pieces of apparatus (cryostats, furnaces, pressure cells, shear apparatus, etc).

The neutron has a small magnetic moment. This can interact with the spin and orbital magnetic moments present in a sample containing atoms with unpaired electrons, giving rise to an additional scattering mechanism.[24]

Table 1

Atomic Nucleus	b_{coh} (fm)	σ_{coh} (barns)	σ_{inc} (barns)	σ_{abs}[23] (barns)
^{1}H	- 3.741	1.8	80.3	0.3
^{2}D	+ 6.671	5.6	2.1	0.0
B	+ 5.304	3.5	1.7	767.0
C	+ 6.646	5.6	0.0	0.0
N	+ 9.362	11.0	0.5	1.9
O	+ 5.803	4.2	0.0	0.0
Na	+ 3.580	1.6	1.7	0.5
Si	+ 4.153	2.2	0.0	0.2
P	+ 5.131	3.3	0.0	0.2
S	+ 2.847	1.0	0.0	0.5
Cl	+ 9.577	11.5	5.3	33.5
Ti	- 3.438	1.5	2.9	6.1
V	- 0.382	0.0	5.1	5.1
Cd	+ 5.130	3.3	2.5	2520.5
Gd	+ 6.550	29.4	151.2	49700.1

Among the similarities between electromagnetic and neutron radiation (conferred by the De Broglie/Schrödinger wave-particle duality principle) are that both may be polarised, both give rise to birefringence, both may have the plane of polarisation rotated by an "active" material (cf. optical activity) and both demonstrate the concept of refractive indices. Interestingly, the neutron refractive index of a material, *n*, given by

$$n = (1 - N b_{coh} \lambda^2 / 2\pi) \quad \ldots (3)$$

where *N* is the atomic number density is, typically, slightly less than unity. Optical refractive indices are, of course, greater than unity. This difference allows neutrons to be totally externally reflected from a surface (providing the angle of incidence is less than some critical angle), a property that has spawned an entire field of research - neutron reflectometry (NR).[25,26] This technique is proving to be a powerful means of investigating surfaces and interfaces and is discussed elsewhere in this review.

There are some drawbacks with using neutron radiation. An obvious one is that it is not a technique for the laboratory benchtop. Neutron sources, like synchrotron radiation sources (SRS), are very large, costly, facilities which are best constructed, operated and shared between several nations. A second drawback is that compared to light or X-rays, neutron sources are relatively weak (strictly, they are lacking in brilliance). The flux of neutrons on a SANS instrument at the most powerful of neutron sources is typically several orders of magnitude lower than the flux of photons on a SRS SAXS beamline, and not too dissimilar to the flux from a laboratory rotating anode X-ray generator. This is yet another aspect of the complementary nature of the different SAS techniques. It is also difficult to focus neutrons whereas it is comparatively easy to focus light or X-rays down to a few mm. In a SANS experiment the neutron beam is typically collimated to be a few mm in diameter, though this is usually at the expense of flux.

Neutron Sources

There are two means of producing neutrons in sufficient quantities for worthwhile experiments. The most obvious of these is to use a nuclear reactor. Here neutrons are released by the fission of uranium-235. Each fission event releases 2 - 3 neutrons, though one of these is needed to sustain the chain reaction. The most powerful of the reactor (or what are also termed "steady-state" or "continuous") neutron sources in the world today is the 55 MW HFR (High-Flux Reactor) at the Institute Max von Laue - Paul Langevin (usually shortened to just the "ILL") in Grenoble, France.[27] The ILL is jointly operated by France, Germany, the United Kingdom, and 12 other countries. The facility commenced operation in 1972.

The other approach to neutron production is that used in *spallation* neutron sources. These use particle accelerators and synchrotrons to generate intense, high-energy, proton beams which are, in turn, directed at a target composed of heavy nuclei. Provided that the protons have sufficient kinetic energy they are able to overcome the intrinsic long-range electrostatic and short-range nuclear forces they encounter and effectively blast the target nuclei apart. The word spallation is a quarrying term for "chipping away". The most successful spallation neutron source in the world is the ISIS Facility near Oxford in the United Kingdom.[28-30] It is based around a 200 μA, 800 MeV (ie, 160 kW), proton synchrotron operating at 50 Hz, and a tantalum target which releases approximately 12 neutrons for every incident proton. ISIS is operated by the United Kingdom but also receives funding from a variety of international partners. The facility commenced operation in late 1984. The most powerful spallation neutron source in the world is the 850 kW SNS at the Oak Ridge National Laboratory, USA.

Today there are some 37 neutron sources in 21 countries[31]; of these, 23 are in continental Europe (including Russia and Scandinavia), 9 are in North America (including Canada; IPNS closed in 2008), 2 are in Japan with 1 in each of Australia and India. Five of the sources are spallation sources, the remainder are generally rather aging reactors, though some, such as the ILL, have undergone recent refurbishment to extend their useful lifetimes.[32] The total number of SANS instruments at these sources is 36; of which 21 are to be found at the European facilities.[33] Despite this apparent glut of facilities and instruments, the demand for SANS beam time typically outstrips the beam time actually available by a factor of 2 or 3.

Small Angle Neutron Scattering

A re-derivation of the theory of SANS from first principles is beyond the scope of this review, as are instrumentation details. Instead, a more pragmatic, source-independent, approach is presented.

In any SAS experiment, a beam of collimated, though not necessarily monochromatic, radiation is directed at a sample, illuminating a small volume, V (= $A\ t_s$, where A is the cross-sectional area of the beam and t_s is the pathlength of the sample), typically < 0.5 cm^3 for solvated systems. Some of the incident radiation is transmitted by the sample, some is absorbed and some is scattered. A detector, or detector element, of dimensions $dx \times dy$ positioned at some distance, L_{sd} , and scattering angle, θ , from the sample then records the flux of radiation scattered into a solid angle element, Δ Ω (= $dx\ dy$ / L^2_{sd}). This flux, $I(\lambda, \theta)$, may be expressed in general terms in the following way

$$I(\lambda, \theta) = I_0(\lambda)\ \Delta\Omega\ \eta(\lambda)\ T\ V\ \frac{\partial \sigma}{\partial \Omega}(Q) \quad \text{... (4)}$$

where I_0 is the incident flux, η is the detector efficiency (sometimes called the response), T is the sample transmission and $(d\sigma / d\Omega)(Q)$ is a function known as the (*microscopic*) *differential cross-section*. Although this function is specific to SANS, analogous functions exist for light and X-rays. The first three terms of Equation 4 are clearly instrument-specific whilst the last three terms are sample-dependent.

The objective of a SANS experiment is to determine the differential cross-section, since it is this which contains all the information on the shape, size and interactions of the scattering bodies (assemblies of scattering centres) in the sample. The differential cross-section is given by

$$\frac{\partial \sigma}{\partial \Omega}(Q) = N_p\ V_p^2\ (\Delta\delta)^2\ P(Q)\ S(Q) + B_{inc} \quad \text{... (5)}$$

where N_p is the number concentration of scattering bodies (given the subscript "p" for "particles"), V_p is the volume of one scattering body, $(\Delta \delta)^2$ is the square of the difference in *neutron scattering length density* (what is more commonly called the *contrast* for convenience), *P(Q)* is a function known as the *form* or *shape factor*, *S(Q)* is the *interparticle structure factor*, *Q* is the modulus of the *scattering vector* and B_{inc} is the (isotropic) incoherent background signal. $(d\sigma / d\Omega)(Q)$ has dimensions of $(\text{length})^{-1}$ and is normally expressed in units of cm^{-1}.

In some texts the microscopic differential cross-section in Equation 4 is replaced by the product $N_p \times (d\sigma / d\Omega)(Q)$, a quantity known as the *macroscopic differential cross-section*, $(d\Sigma / d\Omega)(Q)$. In such cases Equation 5 is modified accordingly. It is also worth noting that the product $N_p \times V_p$ is the same as the volume fraction of scattering bodies, ϕ_p.

Neutron scattering is unfortunately a prime example of a field desperate for some form of systematic nomenclature. For example, it is very common to find $(d\sigma / d\Omega)(Q)$ or $(d\Sigma / d\Omega)(Q)$ written simply as *I(Q)*, whilst the form factor is variously expressed as *P(Q)*, *F(Q)* or even, and very confusingly, as *S(Q)*. Worse still, the scattering vector is to be found represented as *Q*, *q*, *h*, *k* and *s*. For the most part this mixture of nomenclature reflects the multi-disciplinary evolution of the field.

The principle terms in Equation 5 will now be considered individually.

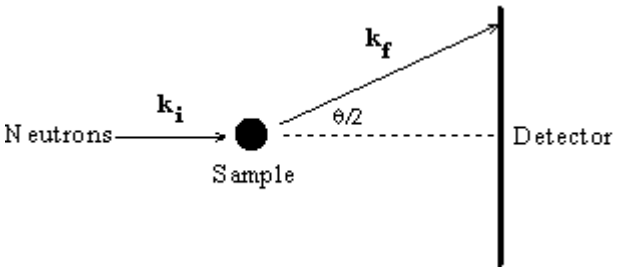
The Scattering Vector

The quantity colloquially referred to as "the scattering vector" (given the symbol *Q* throughout this review) is the modulus of the resultant between the incident, $\boldsymbol{k_i}$, and scattered, $\boldsymbol{k_s}$, wavevectors, see Figure 1, and is given by

$$Q = |\boldsymbol{Q}| = |\boldsymbol{k_f} - \boldsymbol{k_i}| = \frac{4\pi n}{\lambda} Sin(\theta / 2) \quad \text{... (6)}$$

although, as has already been shown, in neutron scattering $n \sim 1$. *Q* has dimensions of $(\text{length})^{-1}$; normally quoted in nm^{-1} or Å^{-1}.

Figure 1



Substituting Equation 6 into Bragg's Law of Diffraction

$$\lambda = 2d\, Sin(\theta / 2) \quad \text{... (7)}$$

yields a very useful expression

$$d = \frac{2\pi}{Q} \quad \text{... (8)}$$

where *d* is a distance. Equations 6 and 8 are central to SANS experiments because through their combined use it is possible to both configure an instrument (i.e., ensure that its "*Q*-range" allows you to see what you expect) and to quickly and rapidly "size" the

scattering bodies in a sample from the position of any diffraction peak in *Q*-space. For example, one of the SANS instruments at the ISIS Spallation Neutron Source, called *LOQ*, has a *Q*-range of ~ 0.06 - 10 nm^{-1}, allowing it to probe a range of length scales from ~ 0.6 - 100 nm.

Equation 6 also provides a way of highlighting the different approaches to SANS measurements at the different types of neutron source. At a steady-state source it is usual to vary *Q* by effectively scanning q at a pre-selected value of λ . On pulsed sources it is more common to employ a "fixed instrument geometry" (effectively a constant q mode) and to obtain a range of *Q* values by time-sorting different λ 's from a polychromatic incident beam as they arrive at the detector. One of the consequences of this approach is that pulsed source SANS instruments have a greater dynamic range in *Q* than their steady-state source counterparts, even though the full *Q*-range actually accessible by both types of instrument is generally quite similar. Fixed-geometry instruments are therefore ideal for studying systems where a range of length scales are involved, or possibly, where the length scales are uncertain. On the other hand, steady-state source instruments sometimes have the advantage that if the *Q*-range of interest is known then it may be possible to optimise the count rate by utilising a wavelength close to peak of the flux distribution.

Physics-based texts often refer to the scattering vector as the *momentum transfer*. This is because the product h *Q* is equal to the change in the momentum of the neutron on scattering.

The Contrast Term

The neutron scattering length density, δ , of a molecule of *i* atoms may be readily calculated from the simple expression

$$\delta = \sum_i b_i \cdot \frac{D N_A}{M_w} \quad \text{... (9)}$$

where *D* is the bulk density of the scattering body and M_w is its molecular weight. With polymers it is only necessary to calculate δ for one repeat unit. δ has dimensions of $(\text{length})^{-2}$ and can also be negative.

The contrast is simply the difference in δ values between that part of the sample of interest, δ_p , and the surrounding medium or matrix, δ_m , all squared; i.e., $(\Delta \delta)^2 = (\delta_p - \delta_m)^2$. Clearly, if $(\Delta \delta)^2$ is zero then Equations 5 and 4 are also zero and there is no SANS. When this condition is met the scattering bodies are said to be at *contrast match*. Since the SANS from a multi-component sample is essentially a contrast-weighted summation of the SANS from each individual component, the technique of contrast matching can be used to dramatically simplify the scattering pattern. For example, in the study of adsorbed layers it is quite common to contrast match the substrate to the dispersion medium, typically by mixing hydrogenous and deuterated forms of the medium in the appropriate ratio, so that the only SANS observed is that from the adsorbed layer alone. A selection of neutron scattering length densities for the fully hydrogenated and perdeuterated forms of common solvents and polymers are shown in Table 2. Scattering length densities of some common substrates are shown in Table 3.

The scattering length density of a molecule is remarkably sensitive to the value of the density used in its calculation and so a reliable knowledge of the latter is a pre-requisite for a successful contrast matching experiment.

In LS the contrast term arises out of the difference in refractive indices between the different components, whilst in SAXS it arises out of the differences in electron density.

Table 2

Solvent	d (h form) (´ 10^{10} cm^{-2})	d (d form) (´ 10^{10} cm^{-2})	Polymer	d (h form) (´ 10^{10} cm^{-2})	d (d form) (´ 10^{10} cm^{-2})
Water	- 0.56	+ 6.38	PB	- 0.47	+ 6.82
Octane	- 0.53	+ 6.43	PE	- 0.33	+ 8.24
Cyclohexane	- 0.28	+ 6.70	PS	+ 1.42	+ 6.42
Toluene	+ 0.94	+ 5.66	PEO	+ 0.64	+ 6.46
Chloroform	+ 2.39	+ 3.16	PDMS	+ 0.06	+ 4.66
Carbon Tet.	+ 2.81		PMMA	+ 1.10	+ 7.22

Table 3

Substrate	d (´ 10^{10} cm^{-2})	Substrate	d (´ 10^{10} cm^{-2})
Silicon	+ 2.07	SiO_2	+ 3.15
Quartz	+ 3.47	TiO_2	+ 2.57

The Form Factor

The form factor is a function that describes how $(d\sigma/d\Omega)(Q)$ is modulated by interference effects between radiation scattered by different parts of the same scattering body. Consequently it is very dependent on the shape of the scattering body. The general form of $P(Q)$ is given by Van de Hulst's equation[2]

$$P(Q) = \frac{1}{V_p^2}\left|\int_0^{V_p} \exp\left[i\, f(Q\alpha)\right]\, dV_p\right| \quad \ldots (10)$$

where α is a "shape parameter" that might represent a length or a radius of gyration, for example. Fortunately analytic expressions exist for most common shapes and expressions for more complex topologies (for example, concentric cylinders[34] or hinged rods[35]) can usually be deduced from these. A selection of form factors are shown in Table 4.

Table 4

Sphere of radius R_p	$P(Q) = \left[\frac{3\,(\,Sin\,(QR_p) - QR_p\ Cos\,(QR_p)\,)}{(QR_p)^3} \right]^2$
Disc of negligible thickness and radius R_p (J_1 is a first-order Bessel function)	$P(Q) = \frac{2}{(QR_p)^2}\left[1 - \frac{J_1(2QR_p)}{QR_p}\right]$
Rod of negligible cross-section and length L (S_i is the Sine integral function)	$P(Q) = \frac{2S_i(QL)}{QL} - \frac{Sin^2\,(QL/2)}{(QL/2)}$
Gaussian random coil with z-average radius of gyration R_g, polydispersity (Y+1) and $U = \frac{(QR_g)^2}{(1+2Y)}$	$P(Q) = \frac{2\left[(1+UY)^{-1/Y} + U - 1\right]}{(1+Y)\,U^2}$
Concentrated polymer solution with screening length x where $\xi = R_g\left(\frac{\phi}{\phi^*}\right)^{\frac{\nu}{(3\nu-1)}}$	$P(Q) = P(0)\left[\frac{1}{1+(Q\xi)^2}\right]$

The Structure Factor

The interparticle structure factor, given by

$$S(Q) = 1 + \frac{4\pi\ N_p}{QV}\int_0^\infty [g(r) - 1]\,r\ Sin\,(Qr)\,dr \quad \text{... (11)}$$

is a function that describes how $(d\sigma / d\Omega)(Q)$ is modulated by interference effects between radiation scattered by different scattering bodies. Consequently it is dependent on the degree of local order in the sample, such as might arise in an interacting system for example. The corollary of this of course, is that SAS can be used to gain information about the relative positions of the scattering bodies, usually through the *radial distribution function*

$$G(r) = \frac{4\pi\ N_p\ r^2}{V}\,g(r) \quad \text{... (12)}$$

where r is a radial distance outward from the centre of any scattering body in the sample, and $g(r)$ is obtained from Equation 11 by Fourier inversion. $G(r)$ is typically a damped, oscillating, density distribution function whose maxima correspond to the distance of each nearest-neighbour coordination shell. Moreover, $Ln\ g(r)$ is directly related to the potential energy function describing the interactions between the scattering bodies.

An alternative procedure involves using one of the many approximate forms of $S(Q)$ that have been developed over the years (to describe particular types of system) to calculate the expected shape of $(d\sigma\ /\ d\Omega)(Q)$.[36,37] This may then be model-fitted to the observed scattering data.

Unfortunately, as $N_p \rightarrow 0$ (i.e., as the concentration of scattering bodies becomes more dilute) so $S(Q) \rightarrow 1$, meaning that this type of insight into the microscopic structure can only be obtained in concentrated and/or strongly interacting samples.[13]

Useful Conversions

Scattering Vector 1 $Å^{-1} = 10\ nm^{-1}$

Cross-Section 1 barn = $10^{-24}\ cm^2$

Wavelength 1 fm = 10^{-6} nm = 10^{-15} m = 10^{-5} Å

Energy 1 eV = $1.60219' 10^{-19}$ J » 10^5 J mol^{-1} » $10^4\ cm^{-1}$

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21. Only *coherently* scattered neutrons, where phase is conserved, carry any structural information about the sample. All nuclei with non-zero spin also scatter neutrons *incoherently* (because different isotopes may be present in different amounts and/or have different nuclear spin states). In SANS incoherent scattering manifests itself as an isotropic background signal. This can be a problem if there is too much hydrogen in the sample since hydrogen has a much greater incoherent neutron scattering cross-section, σ_{inc}, than most other common nuclei, see Table 1.
22. For a more comprehensive compilation of nuclear scattering lengths and cross-sections, see V.F. Sears, *Neutron News*, (1992), 3, 26. The scattering cross-section and scattering length are related by $\sigma = 4\pi b^2$.
23. σ_{abs} is the absorbtion cross-section. Samples and sample containers should not contain large quantities of nuclei with high absorbtion cross-sections.
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