Adapting N5, A Triple-axis Spectrometer, for Small Angle Neutron Scattering Measurements

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Small angle neutron scattering (SANS) is a powerful technique for the study of molecular structures and morphologies with length scales ranging from 10 Å to 1000 Å. Dedicated SANS instruments cover a scattering vector range (*q*-range) from 0.001 to 0.6 Å^{-1}, where q is defined as:

$$q = \frac{4\pi}{\lambda}\sin\left(\theta/2\right),$$

where and λ and θ are the neutron wavelength and scattering angle, respectively.

Presently, at the Canadian Neutron Beam Centre (CNBC) no cold source is available in the 120 MW National Research Universal (NRU) reactor. In order to adapt a triple-axis spectrometer for small angle measurements, the incident beam therefore needs to be highly collimated. Moreover, the use of a single crystal monochromator to select the wavelength of the incident neutrons ($\Delta\lambda/\lambda < 1\%$), significantly reduces neutron flux on the sample compared to the use of a velocity selector ($\Delta\lambda/\lambda \sim 10\%$). To increase incident neutron flux, while not increasing the beam size, we have employed multiple incident beams that converge at a spot on the detector, first proposed and tested by Nunes [1]. At CNBC, we have designed and implemented such a confocal Soller collimator (CSC, as shown in Figure 1). The CSC is 66 cm long and made up of 23 channels, whereby each channel is separated by 0.25 mm spring steel blades coated with Gd_2O_3. All channels converge on the same spot at the detector. Each individual channel has dimensions of 3.8 cm (high) by 0.13 cm (wide) at the monochromator end and 3.8 cm (high) by 0.10 cm at the end closest to the sample.

The experimental configuration for adapting the N5 triple-axis spectrometer to SANS measurements is shown in Figure 2. In order to cover a wide *q*-range, three values of λ (2.37, 4.00 and 5.23 Å) are used from the (002) crystal plane reflection of a PG monochromator at monochromator angles (θ_M) of 20.69°, 36.50° and 51.25°, respectively. However, the incident neutron beam can be contaminated by higher order harmonics of the fundamental neutron wavelength (i.e., $\lambda/2$, $\lambda/3$, etc.), which are reduced either through the use of a beryllium (Be) ($\lambda > 3.99$ Å) or pyrolytic graphite (PG) filter ($\lambda = 2.37$ Å). A sapphire filter is optionally used for reducing the presence of "fast neutrons" in the incident beam [2]. Therefore, depending on the chosen wavelength, either a sapphire ($\lambda \le 3.99$ Å), or Be ($\lambda > 3.99$ Å) filter (component 1 in Figure 2), cooled to liquid nitrogen temperature is placed upstream of the PG monochromator (component 2 in Figure 2). Moreover, due to the slit geometry of the incident beam, a 48 cm-long horizontal Soller collimator (HSC) with individual channels of 0.25 cm vertical opening is required on the scattered side (detector) in order to reduce the smearing due to vertical divergence. In some cases where high resolution is required, a 21.6 cm long HSC with 0.25 cm vertical opening of individual channels is also placed prior to the sample position.

Figure 3 shows a comparison of the total intensity (all channels opened) with that from different channels. It is clear that the CSC enhances the incident neutron intensity by a factor of 20 compared with that from each individual channel, without any noticeable effect on the projected beam size (on the detector) and the attainable minimal *q*, q_{min} is around 0.006 Å^{-1}. This development allows for the ubiquitous triple-axis spectrometer to have a capability of SANS, with little cost and effort.

Using this N5-SANS (Figure 2), we examined standard polystyrene microsphere samples with a diameter of 24 nm (PS02N, Bangs Laboratories). The microspheres arrived as 10 wt.% solutions and were subsequently diluted to 1 wt.% with D_2O (Atomic Energy of Canada Limited, Chalk River, Ontario, Canada). The samples were loaded in rectangular quartz cells having a 5 mm path length. The absolute intensities (i.e., $d\Sigma/d\Omega$) for the sample are obtained from the NG3-SANS located at National Institute of Standards and Technology (NIST), as shown in Figure 4a. To obtain the best-fit structural parameters, NG3-SANS data are fitted using a core-shell-sphere model convoluted with the instrumental resolution. The same model and structural parameters are used to rescale the scattering data of the sample obtained from N5-SANS, however, this time taking into consideration the N5-SANS instrumental resolution (Figure 4b). The N5-SANS data are rescaled for each *q*-range and then plotted on the same figure. Slight discontinuities at the overlapping regions of the rescaled data are observed, presumably due to different resolution functions of the individual *q*-ranges. We find good agreement between NG3-SANS and N5-SANS data (Figure 4b) proving the successful implementation of this SANS design. The total data collection time at the N5-SANS was ~ 4.5 hours (1½ hours for $\lambda = 5.23$ Å, 1 hour for $\lambda = 4$ Å and 1 hour for $\lambda = 2.37$ Å), which is longer than that at NG3-SANS (40 minutes). The statistics of the N5-SANS data are poorer than those obtained at the NG3-SANS data. The reasons for this are twofold: (1) the small detecting area of the 32-wire N5-SANS detector (12 cm x 6.5 cm) requires multiple detector locations to cover a given *q*-range (compared to the much larger 2-D detector at NG3 with a size of 65 cm x 65 cm), and (2) for weakly scattering samples, air scattering dominates at low-q, thus longer collecting times are needed to reduce the error bars when subtracting the air scattering from the sample scattering.

In this report, we have shown that the triple-axis spectrometer, N5, can be successfully used for SANS measurements with a simple installation of a CSC. Data (from 0.006 $Å^{-1}$ to 0.3 $Å^{-1}$) obtained from N5-SANS agree well with those collected from the well-established 30 m NG3-SANS. Fine adjustments such as the use of evacuated HSCs and multiple off-set monochromators can presumably improve the performance further.

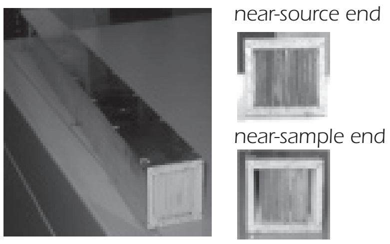

Fig. 1 *Photograph of the 23-channel CSC.*

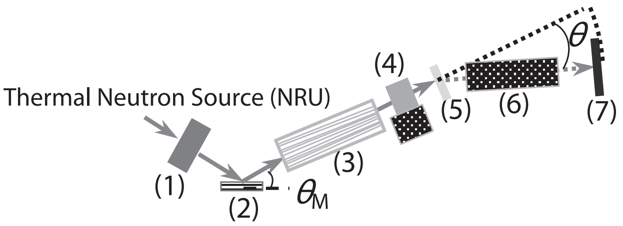

Fig. 2 *Schematic of the N5-SANS adapted from a triple-axis spectrometer. The components are as follows: (1) Sapphire or Be filter. (2) Monochromator. (3) 23-channel Converging Soller Collimator (CSC). (4) PG filter/21.6cm-long Horizontal Soller Collimator (HSC)/ open. (5) Sample. (6) 48cm long HSC. (7) 32-wire position sensitive detector.*

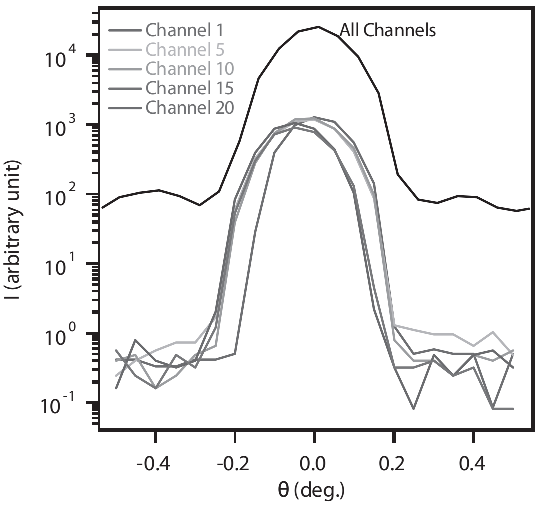

Fig. 3 *Comparison of incident beam intensity from individual channels (1, 5, 10, 15, 20) and all channels.*

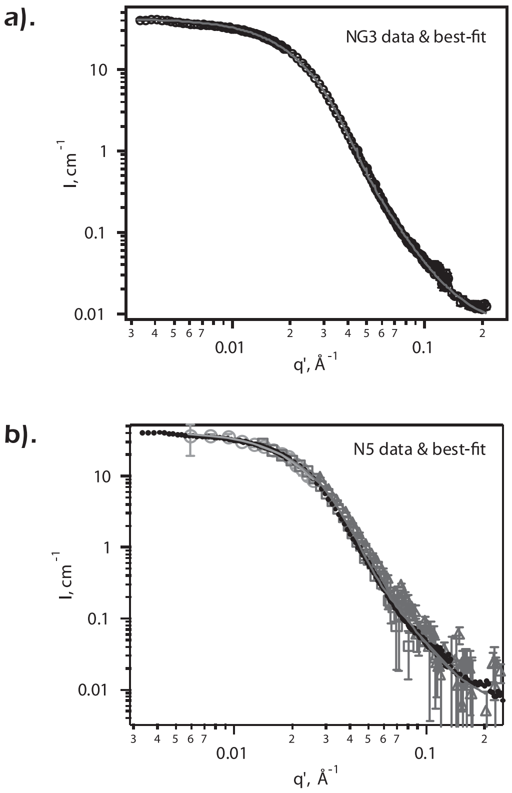

Fig. 4 *SANS data of 1 wt.% microspheres with a diameter of 24 nm obtained from (a) the NG3-SANS and (b) the N5-SANS. The best-fits using a core-shell-sphere model are shown as solid lines. N5-SANS data are rescaled using the same model and structural parameters as the NG3-SANS, but different instrumental resolution. Circles, squares and triangles in (b) represent the SANS configurations using λ = 5.23, 4, and 2.37 Å, respectively. NG3-SANS data (dots) are also plotted in (b) for comparison purpose.*

References

[1] C. Nunes, Nuclear Instr. Methods 119, 291 (1974).

[2] H. F. Nieman, D. C. Tennant, and G. Dolling, Rev. Sci. Instrum. 51, 1299 (1980).