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SMALL-ANGLE NEUTRON SCATTERING AT PULSED SOURCES COMPARED TO REACTOR SOURCES

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Abstract: Detailed comparisons of measurements made on small-angle neutron scattering instruments at pulsed spallation and reactor sources show that the results from the two types of instruments are comparable. It is further demonstrated that spallation instruments are preferable for measurements in the mid-momentum transfer domain or when a large domain is needed.

Introduction

Small-angle neutron scattering (SANS) instruments at pulsed sources use time-of-flight (TOF) to measure the magnitude of the momentum of each counted neutron, rather than monochromate the incident beam. This is necessary, as the time-averaged flux at pulsed spallation sources is low, and every available neutron must be used. The methods of data acquisition and reduction for TOF-SANS are new and some aspects are still under development, and we need to demonstrate that data obtained using these new techniques are reliable, and that measurements taken on the two classes of instruments are comparable. We present here some results of some measurements designed to meet this objective, in which detailed comparisons are done between measurements on standard samples at TOF instruments with SANS instruments at reactors. In this paper we will show that the answers obtained from the two types of instruments are the same, with the caveat that there are some minor differences that may be due to incoherent scattering, multiple scattering, and instrument resolution. We hope that as a result of this work experimenters will be aided in planning measurements and in making good choices as to which type of instrument might be more suitable for a particular measurement.

Results

A blend of d-polystyrene (48%) with h-polystyrene

Small-angle scattering from a blend of 48% deuterated polystyrene is expected to exhibit $I(Q) \sim Q^{-2}$, characteristic of a Gaussian coil. In Figure 1 we show the results of data taken on two TOF-SANS instruments: the Low-Q Neutron Diffractometer (LQD) at the Los Alamos Neutron Scattering Center, Los Alamos National Laboratory and the Small-angle Neutron Diffractometer (SAD) at Argonne National Laboratory; and two reactor-based instruments: the 30M SANS at Oak Ridge National Laboratory, and D-11 at the Institut Laue-Langevin.

Measurements on LQD and SAD were taken with instrument fixed geometry. The SAD measurement was taken with the detector off-axis. In each case data acquisition and reduction was done by standard methods and are described elsewhere [1-4]. Two geometries were used on the reactor instruments: 19 and 7 M sample to detector distances on the 30-M SANS, and 10 and 2.5 M on D-11. Measurements on the 30-M instrument were done by F. Bates and G. Wignall.

All data are placed in a scale of absolute differential scattering probability per unit solid angle, $dP/d\Omega$ (division by sample thickness will give $d\Sigma/d\Omega$ (cm^{-1}), the macroscopic differential cross section per unit volume per unit solid angle). This was done by determining a calibration constant for each instrument. For the reactor instruments, a primary standard, water, was used [5,6]. Secondary standards with cross sections derived from the reactor instruments were used to calibrate the TOF instruments. In the case of SAD this was an irradiated aluminum standard Al-4, the absolute intensity of which was determined on the Oak Ridge 30 M instrument [6]. For LQD calibration was done using the polystyrene blend discussed here and also a silica gel sample; the absolute intensity values for which were determined on D-11.

The absolute SANS intensities (shifted by factors of two for comparison in figure 1) from each measurement are identical, on the average. Since calibration of the TOF instruments was done using results from the reactor based instruments, there is no surprise that the intensity agrees. That the two TOF instruments agree very well, does show that the determination of a

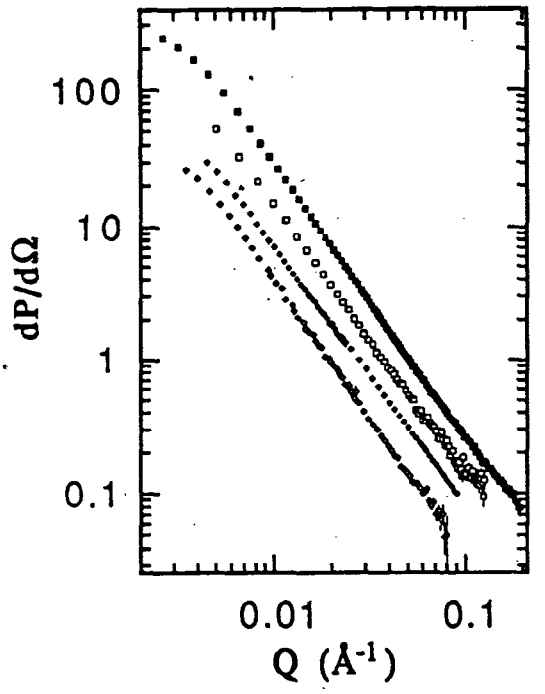

Figure 1. *Polystyrene Blend:* ■, LQD; □, SAD; ◆, D-11; ✧, 30-M SANS

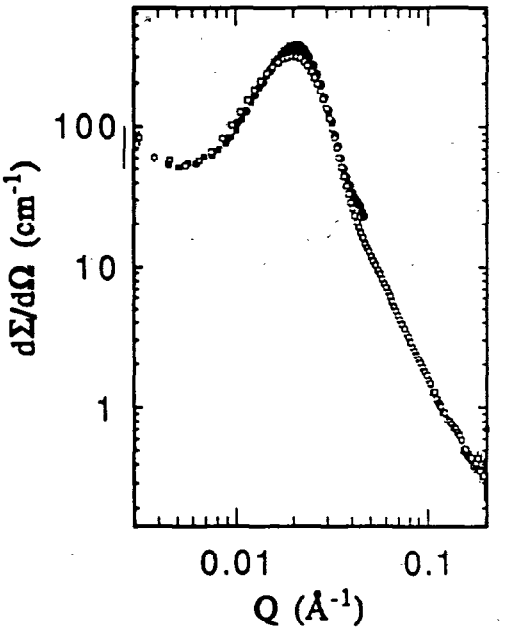

Figure 2. *Vycor Glass:* ■, D-11, 10M; ●, D-11, 5M; □ LQD

calibration constant for these instruments can be done in a straightforward manner, even though the two TOF instruments are very different in design and neutron source characteristics [1,7]

These data (figure. 1) show the large Q-domain accessible using TOF instruments on a single measurement—larger than that available on the reactor instruments, even with two camera settings. In principle, data can be obtained on LQD over the domain $0.002 < Q < 0.5$ Å^{-1}, and $0.005 < Q < 0.3$ on SAD. Realization of this range depends on a number of factors, including scattering intensity, counting times, incoherent scattering and the range of incident neutron wavelength used. These limit the domain of useable data in this instance to $0.003 \leq Q \leq 0.2$ Å^{-1} on LQD and $0.005 \leq Q$ 0.12 Å on SAD.

The slopes of the double log plots for the TOF data are -2 to within experimental error. The slopes of the lines for the reactor-SANS data are slightly smaller. We are not sure of the reasons for the difference but suspect that effects from incoherent scattering may play a role here. George Wignall (private communication) reports that a value of 0.4 cm^{-1} (0.04 in Figure 1) has been subtracted from the ORNL data to obtain a line on the double log plot with slope -2.

In the data taken one LQD there is a deviation form the $I(Q) \sim Q^{-2}$ power law at the lowest Q values . It is likely that in this domain $Q^{-1} \approx R_g$, the radius of gyration of the polymer; thus we are observing a cross-over into a Guinier region. There also may be some effects from multiple scattering [8] due to the large incident neutron wavelengths used to obtain very low Q data. This points out a potential disadvantage of TOF-SANS at very low Q, as limits in instrument length (if

a frame overlap chopper is not used) requires the use of long wavelength neutrons in order to obtain data in this region.

A Sample of Vycor glass:

The porosity of this vycor glass leads to a correlation peak at $Q \simeq 0.02$ Å^{-1}. In Figure 2 we compare data taken at D-11 and LQD. The D-11 data are taken at sample to detector distances of 10M and 5M using 10Å neutrons. Data on LQD were taken with a proton beam current on target of 60μA. The results give some further interesting comparisons between LQD and D-11.

Scattering measurements (expressed in figure 2 as $d\Sigma/d\Omega$ (cm^{-1})) taken on the two instruments are essentially the same, given that for a peaked function the instrument resolution becomes a factor in the result. In the example in Fig. 2 data, from LQD were reduced using all time channels. This gives the maximum Q-range for the measurement. When this procedure is used the precision in Q for LQD (sample to detector distance ≅ 4.3M) is similar to D11 at 5M, as evidenced by the similarity in curve shape and height. However, resolution of a TOF instrument is dependent on the TOF-channels used in data reduction [2]; thus, had only longer TOF-channels been included, a narrower peak in the region about $Q \approx 0.02^{-1}$ would have resulted.

The data acquisition time for this experiment and its transmission on LQD was 30 minutes. To achieve the same statistics (0.45%) in an equal width Q-bin (0.001 Å^{-1}) at a single value of Q (0.030 Å^{-1}) at D - 11 would take 9 minutes at the 5 - m position. Thus it took more than 3 times as long to get a data set at LQD, but the Q - range obtained is nearly 4 times as wide. In this example, the extended range allows determination of the power-law slope above $Q = 0.045$ Å^{-1} . When a constant (incoherent contribution) of 0.14 cm^{-1} is subtracted, the slope of -3.360±0.006 extends to the highest values of Q measured.

Conclusions:

From these examples, we conclude that the results of scattering measurements taken at TOF-SANS and reactor-based SANS instruments are comparable. Small differences can perhaps be better understood, and thus corrected. The current generation of small-angle instruments on pulsed spallation sources is competitive at moderate Q and may be faster when a wide range of Q is required. It appears, however, that present high flux reactor-based instruments are superior for measurements at very low-Q or over a narrow range of Q.

Acknowledgements

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Q(R.E.Lechner): Which kind of "incoherent back ground" were you referring to in your comparison of small angle scattering instruments, where you claim that this background is larger at reactors than at pulsed sources? Should not the ratio of coherent to incoherent scattering be independent of the instruments? Could it be that the compared instruments at pulsed sources are better shielded than at the reactors?

A(R.Pynn): We do not really understand yet why the pulsed source SANS data seem to be so much better than data from reactors. Perhaps it has to do with wavelength dependence of incoherent scattering. Remember that larger values of Q are measured with shorter wavelength neutrons at pulsed sources, so the incoherent contribution varies as a function of Q. We need to do more work to understand both the incoherent & inelastic scattering contributions to SANS for both reactors and pulsed sources.

C(N.Watanabe): I agree to your argument that simple statement "performance of SANS is proportional to average source flux" is too naive. My argument yesterday is just for simple comparison.

Q(N.Niimura): We should think that transmission and scattering measurement should be carried out simultaneously by developing the detector system for SANS.

A(R.Pynn): I agree with you. That means of course that we have to find a way of inter-calibrating the detector we use for transmission measurements and the one we use to measure scattering. Or perhaps we could develop a detector with a large enough dynamic range to measure both transmission and scattering.