Journal of
Applied Crystallography
ISSN 0021-8898

Received 16 August 2006
Accepted 7 February 2007

Performance of the 36 m small-angle neutron scattering spectrometer at BATAN, Serpong, Indonesia

E. Giri Rachman Putra,* A. Ikram, E. Santoso and B. Bharoto

Neutron Scattering Laboratory, Centre for Technology of Nuclear Industrial Materials, National Nuclear Energy Agency – BATAN, Kawasan Puspiptek Serpong, Indonesia. Correspondence e-mail: giri@batan.go.id

A 36 m small-angle neutron scattering (SANS) spectrometer (SMARTer) has been installed at the end of a 49 m long neutron guide tube and located in the neutron guide hall at the Neutron Scattering Laboratory (NSL), Serpong, Indonesia. At present, this is the largest SANS spectrometer in the Asia–Pacific region and consists of an 18 m long tube collimation system and another 18 m long tube to accommodate a 128 × 128 ^{3}He two-dimensional position-sensitive detector. The detector can be moved continuously from 1.5 to 18 m and can be shifted laterally by 0.1 m to cover a large range of Q, where Q is the magnitude of the scattering vector given by $Q = (4\pi/\lambda)\sin(\theta/2)$, θ is the scattering angle and λ is the wavelength. By selecting the rotational speed of the velocity selector, the incident thermal neutron beam will have a wavelength λ in the range 3–6 Å and a Q range of 0.002–0.6 Å^{-1}. The maximum neutron flux at the sample position is 4×10^6 neutrons cm^{-2} s^{-1}. Measurements of some standard samples using SMARTer are reported for inter-laboratory comparisons that show, for the first time, how SMARTer's capabilities compare with those of other prominent SANS instruments.

1. Introduction

The small-angle neutron scattering (SANS) technique has many applications in the fields of polymer science, metallurgy, solid-state physics, colloids and biology (Hutchings & Windsor, 1987). Therefore, it is understandable that many SANS instruments are currently in operation all over the world and new ones are being planned and installed. Although the neutron scattering activity in Indonesia started with its first reactor, TRIGA Mark II located in Bandung, West Java, Indonesia and commissioned in the early 1960s, a SANS spectrometer was not built until 1990. The possibility of building a Neutron Scattering Laboratory (NSL) with more instruments, including a SANS spectrometer, became reality when the largest research reactor in Indonesia was commissioned in 1987.

The National Nuclear Energy Agency (BATAN) in Indonesia commissioned a 36 m SANS spectrometer (SMARTer) at the 30 MW multi-purpose reactor G. A. Siwabessy (RSG–GAS) and it was installed in the neutron guide hall at NSL, Serpong, Indonesia in 1992 (Marsongkohadi, 1996). A 49 m neutron guide tube is mounted on the radial beam port S5 of the reactor and bent to the left with a curvature of 3926 m. The tube consists of four optically flat Borkron glasses glued together to form a cross section of 33 × 90 mm. The inner surface is coated with a thin ^{58}Ni deposit. This guide tube, called the first neutron guide (NG1), is dedicated to the SANS spectrometer installed at its end. The layout of NSL Serpong, BATAN, Indonesia is shown in Fig. 1.

In the past several years, a lot of work, such as setting up and calibrating the instrument, upgrading and replacing some electronic parts and the control system, and developing a data reduction program, has been carried out to make SMARTer fully functional (Gunawan *et al.*, 1996; Ikram, 2005). In this paper, we report for the first time measurements of some standard samples to update the information on the performance of SMARTer compared to other world-class SANS facilities.

2. Instrument specifications and characteristics

SMARTer consists of an 18 m long tube for the collimation system and another 18 m long tube accommodating a 128 × 128 ^{3}He two-dimensional position-sensitive detector (2D-PSD) made by Risø

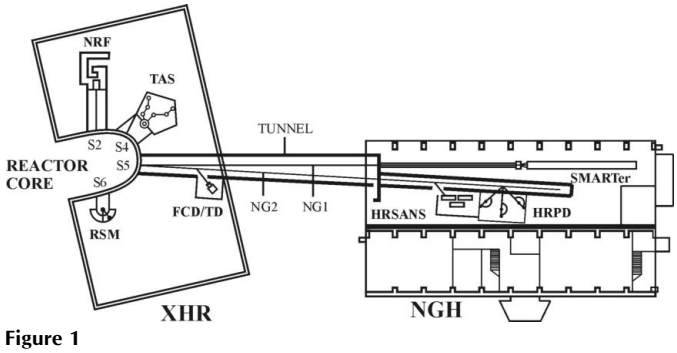

Figure 1
The layout of the neutron beam instruments at the Neutron Scattering Laboratory in Serpong, Indonesia. The instruments are installed in the experimental hall of the reactor (XHR) and in the neutron guide hall (NGH). SMARTer is located in the neutron guide hall. The other instruments are RSM (Residual Stress Measurement) Diffractometer, FCD/TD (Four Circle Diffractometer/Texture Diffractometer), TAS (Triple-Axis Spectrometer), NRF (Neutron Radiography Facility), HRPD (High Resolution Powder Diffractometer) and HRSANS (High Resolution Small Angle Neutron Scattering Spectrometer).

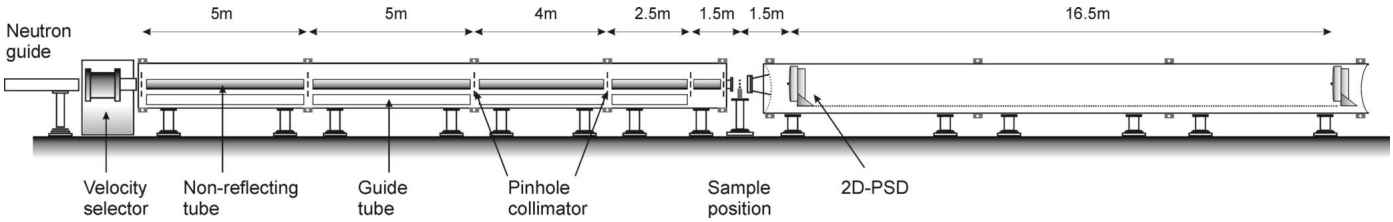

Figure 2
A schematic diagram of SMARTer.

National Laboratory, Roskilde, Denmark. The detector can be moved continuously from 1.5 to 18 m away from the sample position and can also be shifted laterally by 0.1 m from the centre beam to cover a high range of Q, where Q is magnitude of the scattering vector given by $Q = 4\pi/\lambda \sin(\theta/2)$, and λ and θ are the neutron wavelength and the scattering angle, respectively. The collimation system comprises four sections of movable guide tubes and one section of fixed non-reflecting tube (Fig. 2). The collimation is determined by adjustable apertures or pinholes at discrete distances of 1.5, 4, 8, 13 and 18 m from the sample. The variation of collimation length and sample-to-detector distance is fully computer-controlled.

The incident thermal neutron beam is monochromated using a mechanical multidisc velocity selector (Rosta, 1989) of type MDR-14-460-420. The performance of similar model MDR-13-410-420 multi-disc velocity selectors has been reported by Glinka *et al.* (1998). The minimum and maximum rotational speeds are 700 and 7000 r.p.m., respectively, to generate neutrons with λ in the range 3–6 Å. The expected performance of this type of velocity selector has been analysed by Hammouda (1992) and Copley (1993). Based on the instrument specifications of SMARTer, a theoretical Q range of 0.001–0.8 $Å^{-1}$ can be achieved. The neutron flux at the sample position was measured by means of gold foils for λ = 3.22 Å with a collimation length of 1.5 m and a reactor power of 22.5 MW. From this measurement, the maximum neutron flux is 4×10^6 neutrons cm^{-2} s^{-1}. Most of the time, the sample is measured under atmospheric conditions. Additional equipment for the sample environment includes a small heater for soft condensed matter experiments, provision for an external magnetic field up to 1 T, a furnace and a cryostat. The characteristics of SMARTer are summarized in Table 1.

Table 1
Specifications and characteristics of SMARTer.

Source	30 MW research reactor Neutron guide (NG1) 33 × 90 mm
Monochromator	Mechanical velocity selector with variable speed Tilting angle = 0°
Wavelength λ range	3–6 Å
Wavelength resolution ($\Delta\lambda/\lambda$)	10–20%
Source-to-sample distance	1.5, 4, 8, 13 and 18 m
Sample-to-detector distance	1.5 to 18 m continuously
Collimation	Circular pinholes with diameters 30, 20, 14, 10, 7 and 5 mm
Maximum flux at sample position	4×10^6 neutrons cm^{-2} s^{-1}
Sample size	> 5 mm in diameter
Effective Q range	0.002– 0.6 $Å^{-1}$
Size regime	10 to 3000 Å
Detector	128 × 128 ^{3}He position-sensitive proportional counter
Beam stop	50 mm in diameter, can be moved in X and Y directions
Pixel dimension (x, y)	4.64 × 4.99 mm
Ancillary equipment	Small heater up to 373 K, external magnetic field 1 T, furnace and cryostat

3. Experiments and instrument performance

An inter-laboratory comparison of SMARTer with SANS spectrometers at the Japan Atomic Energy Research Institute (JAERI), the former name of the Japan Atomic Energy Agency (JAEA), in Japan and the Australian Nuclear Science and Technology Organization (ANSTO) in Australia has been carried out by measuring a silver behenate (AgBE) standard sample. The result is presented in Fig. 3 showing a good agreement in Q calibration, since it produces a sharp 001 Bragg diffraction peak at 0.108 $Å^{-1}$ (Gilles *et al.*, 1998). This measurement suggested that the wavelength of SMARTer is 3.90 Å with $\Delta Q/Q$ = 0.135. However, the broadening of the scattering profile of SMARTer is most probably due to detector-edge effects and the fact that desmearing was not applied to the raw data.

By using the AgBE sample, we were also able to extend the calibration to some other characteristics of SMARTer, such as effective pixel dimension, wavelength, resolution *etc*. Wavelength variation as a function of rotational speed of the velocity selector, Q calibration as a function of sample-to-detector distances, and observation of second- and third-order Bragg peaks 002 and 003 have been accomplished.

Fig. 4 shows the two-dimensional corrected raw data patterns of AgBE, where a sharp Bragg diffraction peak of the first order is clearly seen under two different measurement conditions. It is shown

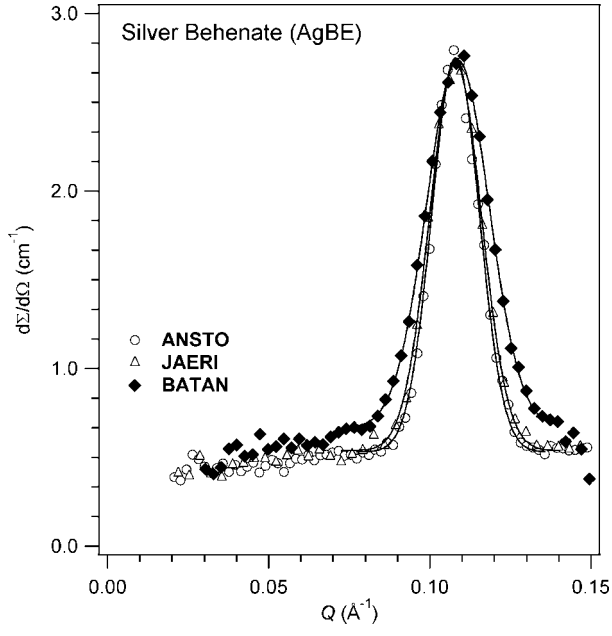

Figure 3
Inter-laboratory comparison of three SANS spectrometers, ANSTO, JAERI and BATAN, using an AgBE sample showing the 001 diffraction peak.

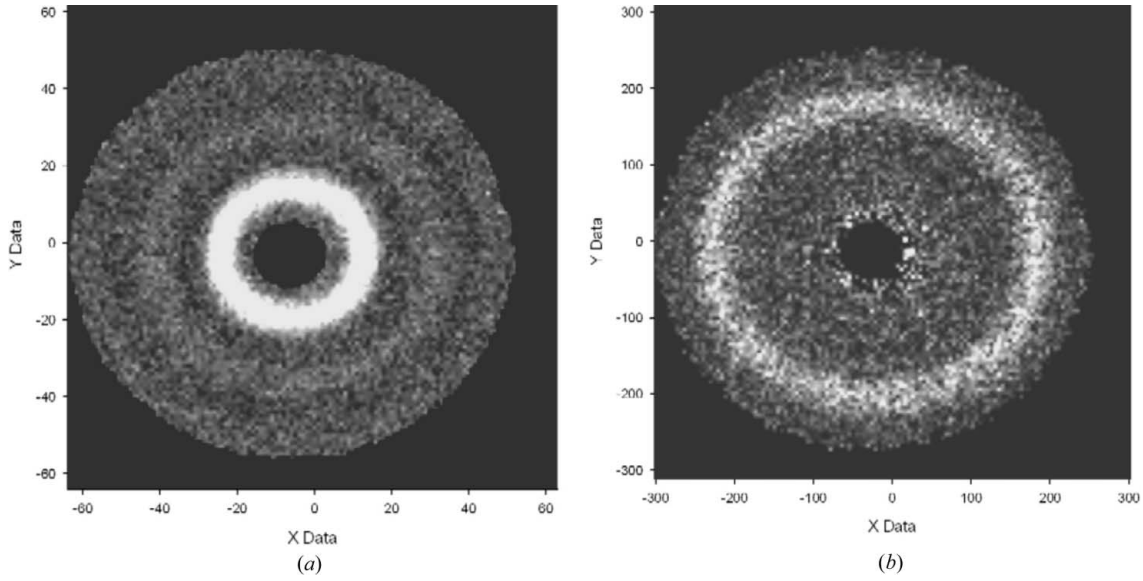

Figure 4
The two-dimensional corrected raw data patterns of AgBE for sample-to-detector distances of (a) 1.5 m with λ = 3.22 Å, showing the diffraction peaks 001, 002 and 003, and (b) 3 m with λ = 3.90 Å, showing the 001 diffraction peak.

that by changing the wavelength and sample-to-detector distance to shorter values, the second-order Bragg diffraction peak can be observed (Fig. 4a). However, the third-order peak is very weak and broad, and cannot be seen clearly in the two-dimensional pattern. Details of the experimental conditions for the measurement of the AgBE sample are presented in Table 2.

The wavelength variation as a function of rotational speed of the velocity selector was measured by positioning the detector 1.5 m from the sample. By using a new effective pixel size for the SMARTer data and a standard sample of AgBE, the neutron wavelength has been determined precisely from the graph of intensity I against scattering angle 2θ. The $\Delta Q/Q$ resolution, which is a total resolution of $\Delta\lambda/\lambda$ and $\Delta\theta/\theta$, was determined from the graph of I against Q. The *I versus Q* graphs of AgBE for various neutron wavelengths are shown in Fig. 5. All measurements show that the first-order Bragg diffraction peak is located at 0.108 Å^{-1}. These results proved that the instrument is well calibrated in Q. Fig. 5 also presents the second- and third-order Bragg diffraction peaks of AgBE. By using a wavelength shorter than 4.35 Å, the second-order peak at 0.217 Å^{-1} can be observed and it becomes more intense on decreasing the wavelength. Using the shortest wavelength of 2.74 Å, a weak and broad third-order peak at 0.325 Å^{-1} related to the lamellar structure of AgBE is observable.

A modified data reduction program based on a data reduction program from JAERI was used to correct the scattering pattern of the

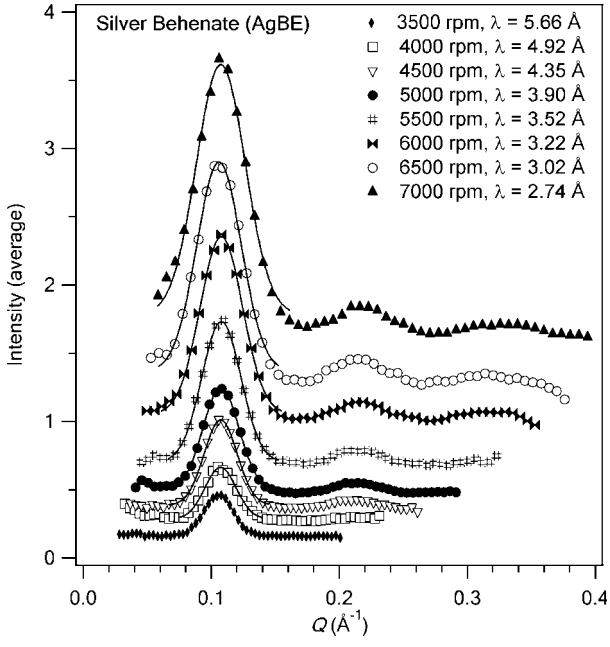

Figure 5
The I against Q profiles of an AgBE standard sample measured at a sample-to-detector distance of 1.5 m for various neutron wavelengths. The 001, 002 and 003 diffraction peaks are observed at 0.108, 0.217 and 0.325 Å^{-1}, respectively.

Table 2
Experimental settings for measurement of AgBE using SMARTer.

Sample-to-detector distance	1.5 and 3 m
Rotational speed of velocity selector	3500–7000 r.p.m.
Data collection time	AgBE 5 h
	Empty cell 5 h
	Electronic background 16 h
AgBE sample transmission	0.625 at 5000 r.p.m.
Sample cell transmission	0.91 at 5000 r.p.m.
Collimation length	4 m
Beam stop	60 mm in diameter
Pinhole setting	20 mm at source and 10 mm at sample

Table 3
Experimental settings for measurements of SDS and CTABr micellar solutions using SMARTer.

Sample-to-detector distance	1.5 and 3 or 4 m
Wavelength	3.90 Å
Data collection time	SDS micellar solution 1 h (5 mm thick)
	CTABr micellar solution 1 h (5 mm thick)
	Empty cell 1 h
	Electronic background 12 h
SDS sample transmission	0.625 at λ = 3.90 Å
CTABr sample transmission	0.63 at λ = 3.90 Å
Empty cell transmission	0.91 at λ = 3.90 Å
Collimation length	4 m
Beam stop	60 mm in diameter
Pinhole setting	20 mm at source and 10 mm at sample

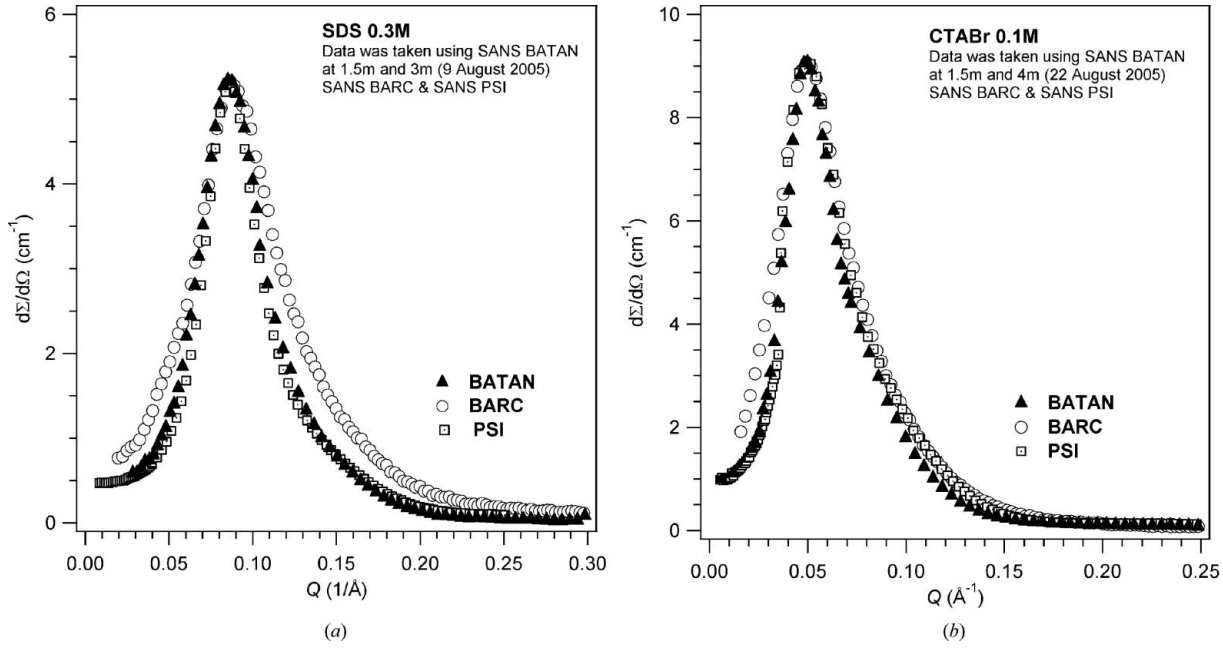

Figure 6
SANS profiles of (a) 0.3 *M* SDS and (b) 0.1 *M* CTABr micellar solutions obtained using three SANS spectrometers: at BATAN, Serpong, Indonesia, at Druva Reactor (BARC), India, and at SINQ (PSI), Switzerland.

sample from its backgrounds, such as empty cell, electronic background and detector efficiency. However, the program for the calculation of the absolute cross section from the scattering intensity (counting) is being developed and will be attached as a subprogram to the data reduction program of SMARTer in due time. The absolute cross sections of SANS profiles shown in Figs. 3 and 6 were scaled to fit with other results.

Another inter-laboratory comparison involving SMARTer at BATAN, Serpong, and SANS spectrometers at the Druva Reactor, Bhabba Atomic Research Centre (BARC), India, and at the Swiss Spallation Neutron Source (SINQ), Paul Scherrer Institute (PSI), Switzerland, has been carried out for the measurement of soft condensed matter samples. Fig. 6 shows measurement results from sodium dodecyl sulfate (SDS) and cetyltrimethylammonium bromide

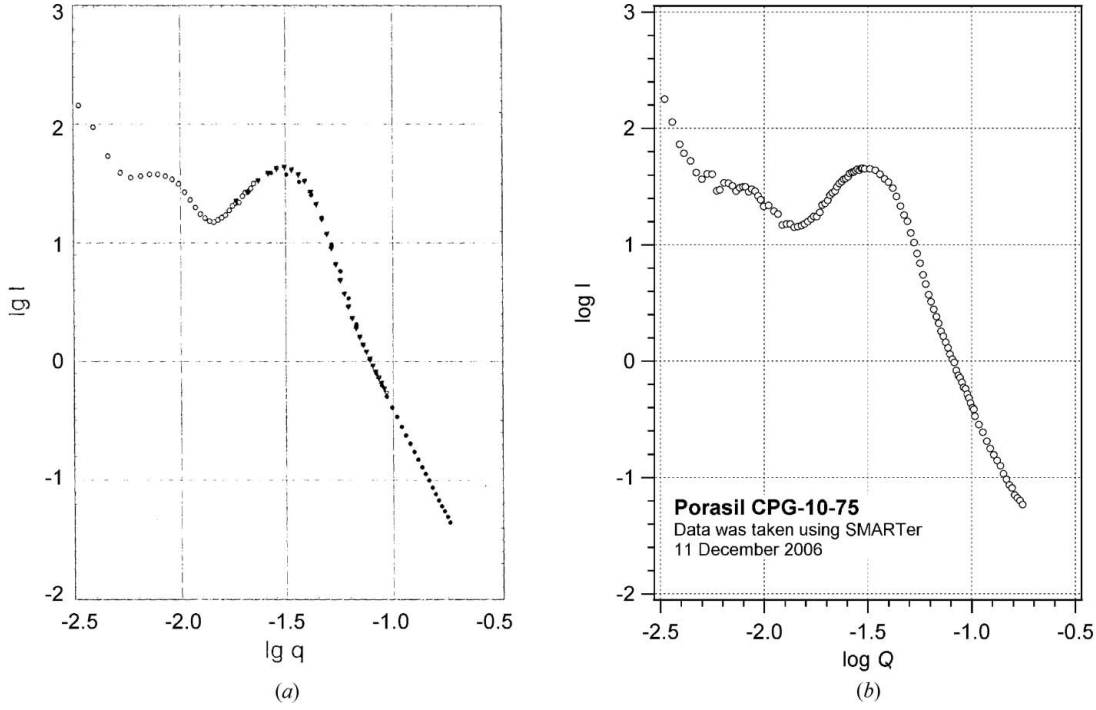

Figure 7
SANS profiles of a porous silica (Porasil) sample, CPG-10-75, measured by (a) SANS, HMI, Berlin, and (b) SMARTer.

(CTABr) solution samples. These results show that SMARTer has an excellent capability compared with other SANS spectrometers.

Data for 0.3 *M* SDS and 0.1 *M* CTABr micellar solutions have a peak at 0.08 and 0.05 Å^{-1}, respectively, and are definitely similar to the data from BARC, India, and PSI, Switzerland. Again, the corrected intensity from SMARTer in Fig. 6 was scaled to the other intensities for direct comparison. Details of those micellar solutions experiments are described elsewhere (Putra, 2007). The experimental settings of SMARTer for the measurements of SDS and CTABr micellar solutions are given in Table 3.

A small heater has been designed and built for temperature-dependent experiments. Details of the furnace/heater are given elsewhere (Santoso *et al.*, 2006). One of the most recent investigations using the heater is a study of the self-assembly of 5wt.% micellar solutions of the triblock copolymer PEO-PPO-PEO (Pluronics) in D_2O. At room temperature, this polymer exists as unimers and has a Gaussian coil structure. When the temperature increases from room temperature, the triblock copolymer PEO-PPO-PEO aggregates to form a a core–shell micelle structure (Mortensen, 1996, 2001). Beside the temperature-dependent measurements, we have also investigated whether the micelles can be entropically induced in the presence of salts at ambient temperature, instead of increasing the temperature. Micellar aggregation of the triblock copolymer PEO-PPO-PEO was the first *in situ* experiment using SMARTer. The inter-micellar interaction, size and thickness of the core–shell structure of the block copolymer PEO-PPO-PEO in D_2O using SMARTer have been discussed in detail elsewhere (Putra *et al.*, 2005; Putra, Ikram & Aswal, 2006).

SMARTer has also been used to investigate a Porod region using a porous silica (Porasil) sample. The SANS profile of a Porasil CPG-10-75 sample is shown in Fig. 7. This result is quite similar to the result, in Q as well as intensity, measured by the SANS spectrometer at the Hahn–Meitner Institute (HMI), Berlin. A slope of −3.8 at high Q for the Porasil sample indicates that it does not show Porod-law Q dependence as the sample surface is too rough. Meanwhile, an inter-correlation peak at 0.03 Å^{-1} indicates that the distance between the pores is about 200 Å. The experimental settings of SMARTer for measurements on this Porasil sample are given in Table 4.

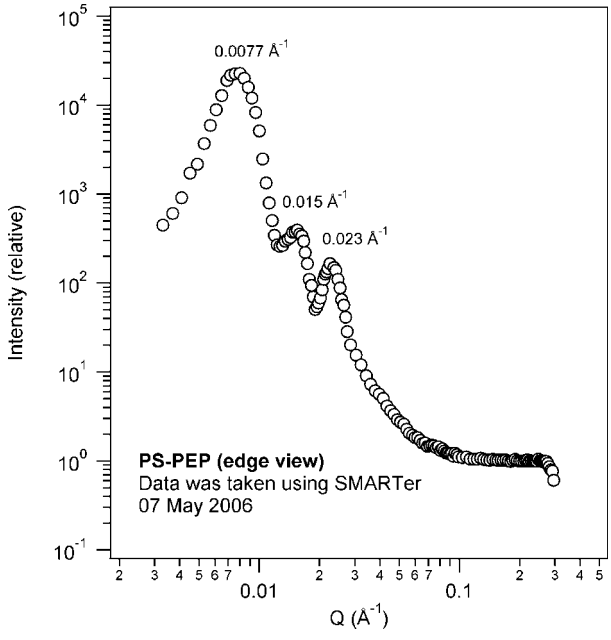

Figure 8
SANS profiles for the block copolymer PS-PEP (edge view) measured at sample-to-detector distances of 1.5 and 4 m for 2 h and at a sample-to-detector distance of 13 m for 8 h.

Table 4
Experimental settings for the Porasil sample using SMARTer.

Sample-to-detector distance	1.5, 4 and 18 m
Wavelength	3.90 and 5.66 Å
Data collection time	Porasil 2 h (1 mm thick)
	Empty cell 2 h
	Electronic background 14 h
Porasil sample transmission	0.83 at λ = 3.90 Å
Collimation length	4 and 18 m
Beam stop	50 mm in diameter
Pinhole setting	30 mm at source and 10 mm at sample

Table 5
Experimental settings for the deuterated block copolymer PS-PEP using SMARTer.

Sample-to-detector distance	1.5, 4 and 13 m
Wavelength	3.90 Å (1.5 & 4 m) and 5.66 Å (13 m)
Data collection time	PS/PEP 2 h at 1.5 & 4 m
	PS/PEP 8 h at 13 m
	Empty cell 2 h
	Electronic background 14 h
PS/PEP sample transmission	0.53 at λ = 3.90 Å
Collimation length	8 m
Beam stop	60 mm in diameter
Pinhole setting	30 mm at source and 7 mm at sample

Another type of sample which has been measured by SMARTer recently is an edge view of a thin film of the block copolymer PS-PEP [polystyrene-block-poly(ethylene-alt-propylene)]. This sample was measured at three different sample-to-detector distances, 1.5, 4 and 13 m, to cover the Q range $0.003 < Q < 0.3$ Å^{-1} using 3.90 and 5.66 Å neutrons. The results are shown in Fig. 8. Details of the experimental settings for the measurement of the block copolymer PS-PEP sample are presented in Table 5. The SANS profile shows several peaks at 0.023, 0.015 and 0.0077 Å^{-1} indicating the existence of lamellar structures. These peaks are correlated to a distance of about 82 Å due to phase separation of the two kinds of polymer chain blocks, polystyrene and poly(ethylene-alt-propylene). The second-order peak at 0.015 Å^{-1} can only be observed clearly by applying 20° double-fan-mode masking for subtracting the anisotropic scattering in the data reduction program.

4. Conclusions

Based on the experiment results, the performance of SMARTer is comparable to other, prominent SANS spectrometers. All types of samples, such as powders, liquids and polymer films, have been measured and investigated in an atmospheric environment, at high temperature (up to 373 K) and in a magnetic field (1 T) over the large Q range 0.002 to 0.3 Å^{-1}. The in-house modified data reduction program works successfully and produces corrected intensity data very well.

Some additional equipment, such as a drawing frame for morphological changes of polymer films and shear apparatus for soft condensed matter samples, will be designed and built to extend the range of materials behaviour studies that can be covered by SMARTer. More experiments with SMARTer are ready to be conducted. The spectrometer is available for users from overseas as well as from the region, since the neutron beam is available for 180 days throughout the year.

The authors acknowledge help and support from many colleagues, namely Dr J. Suzuki from JAERI (JAEA), Japan, for the data reduction program, Dr R. Knott from ANSTO, Australia, for the AgBE sample, Professor H. Hasegawa from Kyoto University, Japan, for supplying a block copolymer PS-PEP sample, Dr A. Wiedenmann from HMI, Berlin, Germany, for providing a Porasil sample, and also Dr V. K. Aswal from BARC, India, for providing SDS and CTABr data obtained using SANS BARC, India, and PSI, Switzerland.

References

Copley, J. R. D. (1993). *Nucl. Instrum. Methods Phys. Res. A*, **332**, 511–520.
Gilles, R., Keiderling, U. & Wiedenmann, A. J. (1998). *J. Appl. Cryst.* **31**, 957–959.
Glinka, C. J., Barker, J. G., Hammouda, B., Krueger, S., Moyer, J. J. & Orts, W. J. (1998). *J. Appl. Cryst.* **31**, 430–445.
Gunawan, G., Supriyanto, A. S., Mujamilah, M. & Kencono, A. W. (1996). *Progress Report Hamburan Neutron*, Neutron Scattering Laboratory, BATAN, Indonesia.
Hammouda, B. (1992). *Nucl. Instrum. Methods Phys. Res. A*, **321**, 275–283.
Hutchings, M. T. & Windsor, C. G. (1987). *Methods of Experimental Physics*, Vol. **23**, Part C. London: Academic Press.
Ikram, A. (2005). *Neutron Scattering Facilities in Serpong. Proceedings of the 6th National Seminar on Neutron and X-ray Scattering*, Serpong, Indonesia, 3 August 2005, pp. 9–16.
Marsongkohadi, M. (1996). *Neutron News*, **7**, 2.
Mortensen, K. (1996). *J. Phys. Condens. Matter*, **8A**, 103–124.
Mortensen, K. (2001). *Colloid Surf. A*, **183–185**, 277–299.
Putra, E. G. R., Bharoto, B., Santoso, E. & Mulyana, Y. A. (2007). *Neutron News*, **18**, 1.
Putra, E. G. R., Ikram, A. & Aswal, V. K. (2006). Private communication.
Putra, E. G. R., Ikram, A., Goyal, P. S. & Aswal, A. K. (2005). *Proceedings of the National Seminar and Congress of Indonesian Polymer Society*, Bandung, Indonesia, 20 November 2005. Indonesian Institute of Science.
Rosta, L. (1989). *Physica B*, **156–157**, 615–618.
Santoso, E., Wahyono, I. & Sairun, S. (2006). *Designing and Manufacturing an Automatic Sample Exchanger for SANS*, presented at the Materials Science and Technology Meeting, BATAN, Indonesia, 18 July 2006. National Nuclear Agency of Indonesia.