1.18 PERFORMANCE OF BATAN-SANS INSTRUMENT

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ABSTRACT

SANS data from some standard samples have been obtained using BATAN-SANS instrument in Serpong. The experiments were performed for various experimental set-ups that involve different detector positions and collimator lengths. This paper describes the BATAN-SANS instrument briefly as well as the data taken from those experiments and followed with discussion of the results concerning the performance and calibration of the instrument. The standard samples utilized in these experiments include porous silica, polystyrene-poly isoprene, silver behenate, poly ball and polystyrene-poly (ethylene-alt-propylene). Even though the results show that BATAN-SANS instrument is in good shape, but rooms for improvements are still widely open especially for the velocity selector and its control system.

INTRODUCTION

The Small Angle Neutron Scattering (SANS) instrument in Serpong has already installed since 1991. Since then, even though there were already many efforts to run the instrument, some and various problems occurred. These problems pushed BATAN back a few steps before real experiments can be performed. In the first years of this workshop, attention was put mostly for the High Resolution Powder Diffractometer (HRPD) with 3 On the Job Training (OJT) held in Serpong. Since 1997, the attention was moved to the SANS machine. This movement was confirmed to be positive since in 1998 Indonesia was under economic crisis that affects the policy in the ministry of research and technology. The country now is asking the research sector to be more applicable and from the seven neutron instruments in Serpong, SANS machine seems to be promising.

The last four workshops have been focused on utilizing SANS method and machine in the area of polymer, especially Natural Rubber-Thermo Plastic Elastomer (NR-TPE). It is one of the requirements that we should have SANS machine which is working and able to perform a number of certain specific experiments. Polymer is not the main major of many of us in Serpong who are mostly physicists. So there is quite a gap in performing this task. This paper presents the results from various runs using several sample standards for various experimental conditions regarding the detector distances and collimation paths and pinholes.

BATAN - SANS INSTRUMENT

This instrument is installed at the end of a 58 m long neutron guide, situated in the neutron guide hall (NGH), to benefit from low background environment. The incident beam is monochromatized by a slot-type mechanical velocity selector having a minimum rotational speed of 700 rpm and a maximum rotational speed of 7000 rpm. The selector's tilting angle can be varied from –3.9° to +3.9°. By varying these rotational speed and tilting angle, neutron wavelengths of 2-5 Å and a Q range of $(0.001<Q< 0.6)$ $Å^{-1}$ can be achieved. Figure 1 shows the schematic diagram of the instrument.

The collimator is placed in an 18 m long tube, comprises of four sections of movable guide tube, and one section of a fixed collimator (non-reflecting) tube. Collimation is obtained by adjusting apertures (pinholes) at discrete distances of 1.5m, 4m, 8m, 13m and 18m from the sample position. The detector, which can be moved continuously from 1.5m to 18m in another 18m tube, is a 128 x 128 He-3 two dimensional position sensitive detector (2D-PSD) made by RISØ, with three beam stoppers of 40, 80 and 140 mm in diameter. The whole system, excluding the sample position is evacuated to 10^{-3} torr. Variations of collimation length and sample to detector distance are fully computer controlled. An automatic sample changer with six sample holders is provided.

In the last three years, many efforts have been carried out to bring this spectrometer back in action, including the SANS sub-workshops held together with the workshops. Some of its components have been replaced, but there are still many others need careful attention and repaired, such as the velocity selector, control motor for the guide tube and beam stopper to mention a few. The problem coming from a spurious peak has not been solved yet even after we tried to adopt the solution taken in JAERI by covering some part of the guide tube entrance after the velocity selector. It seemed in the beginning that it was effective, especially for experiments where the detector are placed at a distance which is less than 5 meters from sample position. Some inspections after that proved that actually the method is not working, especially for longer sample to detector distance experiments.

We also have checked some combinations of the pinholes and it seems that the neutron leakage is coming from the second guide tube in the collimation path. Right now we are running the experiment by placing neutron absorber in front of the sample to absorb neutron coming from other source. Even though this doesn't solve the problem completely, but for the time being, it works for some numbers of experimental settings.

POROUS SILICA

The Porasil (porous silica) sample is a test sample obtained from Oak Ridge National Laboratory. It has scattering cross section of (45 ± 1) cm^{-1} and correlation length of (21.5 ± 1.0) A [1]. SANS profiles were taken for several different sample-to-detector distances (SDD), i.e.: 4m, 5m, 6m, 7m, 8m, 9m, 10m and 11m with some different pinhole settings and collimation paths. The profiles taken at SDD of 4m and 11m do not include the whole peak since the q ranges are not proper for those profiles.

Figure 1 shows SANS profiles of the porasil sample taken at three different sample-to-detector distances. This figure shows that the peak position of the porasil sample appears at different q values for different SDD, even though the discrepancies are

negligible. This result confirms that BATAN-SANS machine is good enough for running experiment with q in the range of $0.1 < q < 0.7\ nm^{-1}$.

POLYSTYRENE-POLYISOPRENE

Polystyrene-poly isoprene (PS-PI) was obtained from the hydrogenation of poly-isoprene with a degree of more than 90%. This sample contained about 10000 PS-PI with ratio of styrene and isoprene about 60/40 wt%.

SANS profiles were taken from this sample for several different sample-to-detector distances, i.e.: 5m, 6m, 7m, 8m, 9m, 10m, 11m, 12m, 13m, 14m, 15m and 16m with some different pinhole settings and collimation paths. The profile moves from left hand side of the diagram to the right hand side according to the changing of q range covered for each SDD setting. It can be seen that profiles with SDD of 5m, 15m and 16 m are not fully covering the whole peak, so the peak position have bigger error bars. From each profile, peak position can be deduced and Figure 2 shows the peak positions obtained for different sample to detector distances. Even though the different is very small and can be neglected from the error bar, but it is clear that different peak position was obtained for different sample to detector distance for the same sample. This information can be used to analyze the data taken from any sample at various sample to detector distances, so the proper results can be obtained more precisely and guaranteed.

SILVER BEHENATE

The silver behenate [$CH_3(CH_2)_{20}COOAg$] (AgBE) sample was obtained from Dr. R. Knott (ANSTO) [2]. It is a standard for q calibration since it is one of the very few materials featuring Bragg reflections in the angular range accessible to SANS instruments. It provides a sharp diffraction peak at $1.08\ nm^{-1}$.

SANS profiles from this silver behenate were taken at several different SDD i.e.: 1.5m, 2m, 3m and 4m with some different pinhole settings and collimation paths. The profiles taken at SDD of 4m did not cover the whole Bragg Peak. Figure 3 presents some of SANS profiles that are taken at sample to detector distances of 1.5m, 2m and 3m. The data quality is not very good but Bragg peak position still can be determined. There is a systematic changing of the profiles that gives a hint for how the correction should be performed. It seems that the profiles taken at SDD of 1.5m has the Bragg peak value closer to the one from the literature. We are still trying to get more SANS profiles in this region with this sample to obtain better quality data.

POLY BALL

The poly ball sample was obtained from Prof. Hirokazu Hasegawa which was prepared by Prof. Hideki Matsuoka, both from Kyoto University, Japan. The SANS profiles were taken at SDD of 17m and 18m, and shown in Figure 4 . The 17m data were taken at two different wavelengths. The two sets of data taken at the same wavelength but different SDD gave similar profile that is very low in resolution. The other data taken at longer wavelength at SDD of 17m show some profile even though the structure does not

appear strong enough. We are still trying to increase the resolution in collecting more data so the structure of the profile can be observed clearly

POLYSTYRENE-POLY (ETHYLENE-ALT-PROPYLENE)

This polystyrene-block-poly (ethylene-alt-propylene) was obtained by hydrogenation of polystyrene-block-poly isoprene with a degree more than 99%. The ratio of styrene and ethylene/propylene was about 60/40 wt%. This sample in edge view configuration produces diffraction peak at q = 0.0762 nm^{-1} together with its derivatives. The SANS profiles from this sample were taken at SDD of 10m, 11m, 12m, 13m, 14m, 15m, 16m, 17m and 18m with some different pinhole settings and collimation paths. The second peak which is at about q = 0.1524 nm^{-1} is very difficult to observe since it has very shallow shape. Only the SANS profile taken at SDD of 18m can show this peak when the whole collimation system did not use any neutron guide tube.

Figure 5 shows four SANS profiles taken from this sample at SDD of 10m, 14m, 16m and 18m. The first figure can only show the third peak, while the first peak appears at SDD of 14m. In the profile with SDD of 16m, the second peak can be spotted even though it is very shallow. The last picture with SDD of 18m shows the three peaks. We are trying to find a better collimation and pinhole setting to obtain better quality data so the setting can be used for other samples.

CONCLUSION

It has been reported that even though not in perfect condition, BATAN-SANS machine is in good shape to produce SANS profiles for various samples, especially the ones related to polymer. Those various standard samples have been used to evaluate the condition of this SANS machine. Some discrepancies and shortcomings have been found and identified so more appropriate and proper SANS measurements can be conducted later for real samples in the FNCA project.

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REFERENCES

1. Gunawan et.al., Improvement of the SANS facility at BATAN, Short Communication, 1998
2. A. Ikram et.al., Inter-laboratory Project q Calibration of SANS Instruments using Silver Behenate, Experiment Report, Proceeding of The 1999 Workshop on the Utilization of Research Reactors, Tokai & Mito, Nov 25 – Dec2, 1999
3. A. Ikram, IAEA Regional (RCA) Workshop on Neutron Beam Research, Taejon, September 24 – 28, 2001.

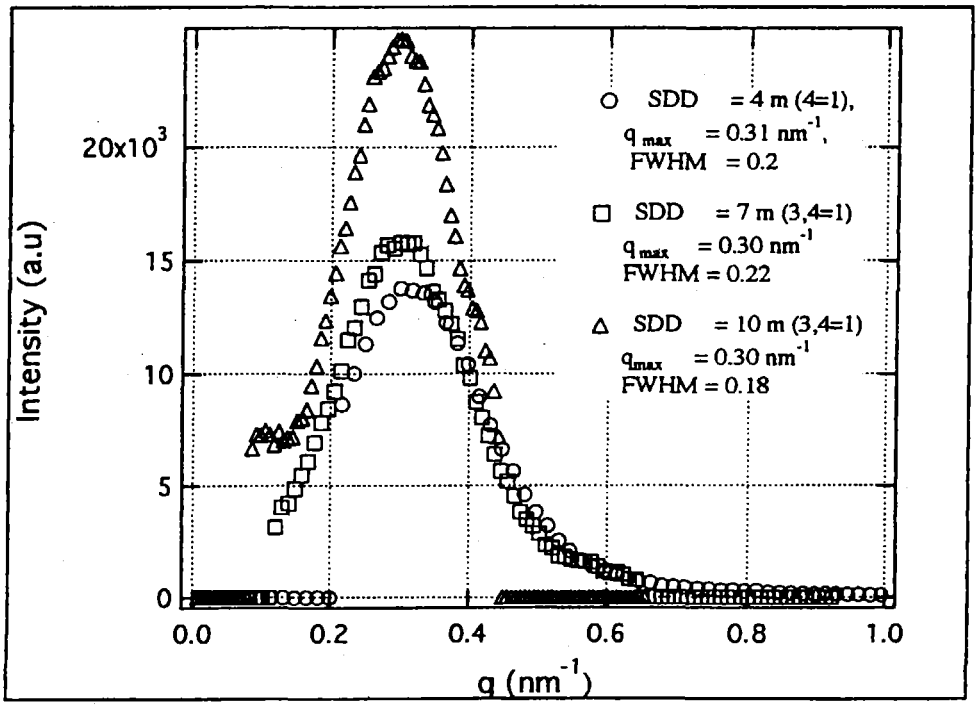

Figure 1. Porasil sample

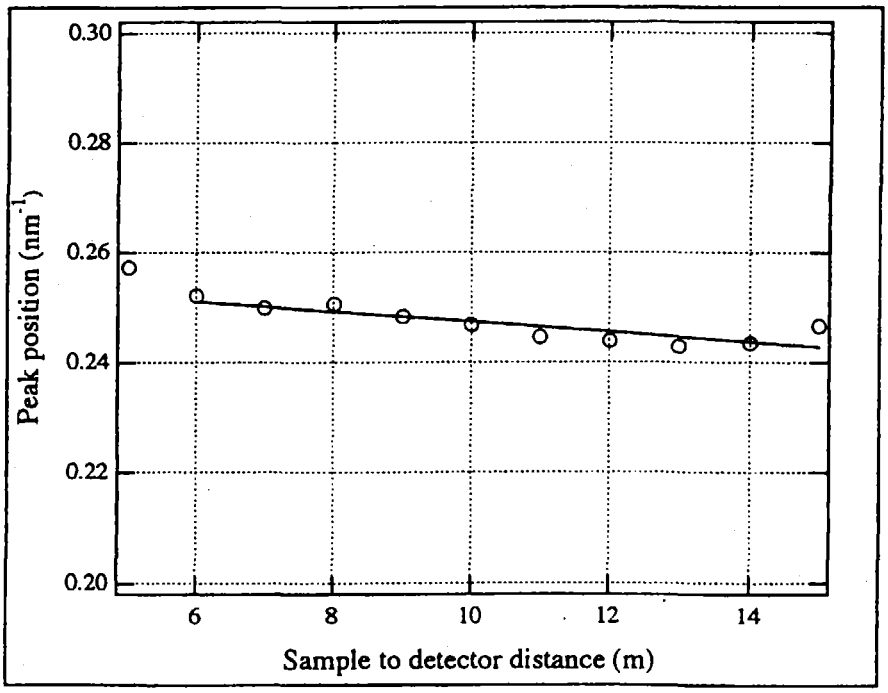

Figure 2. Polystyrene-polyisoprene sample

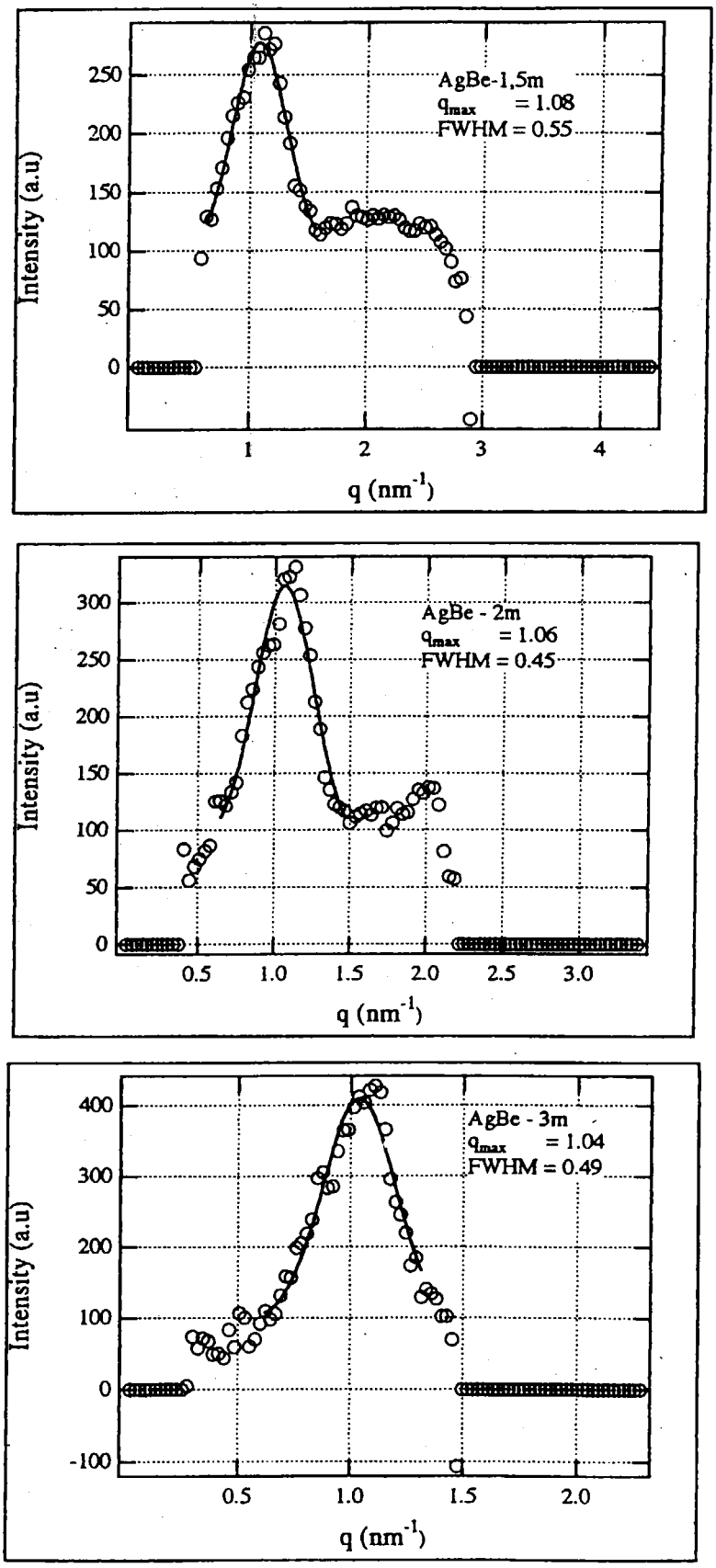

Figure 3. Silver behenate sample

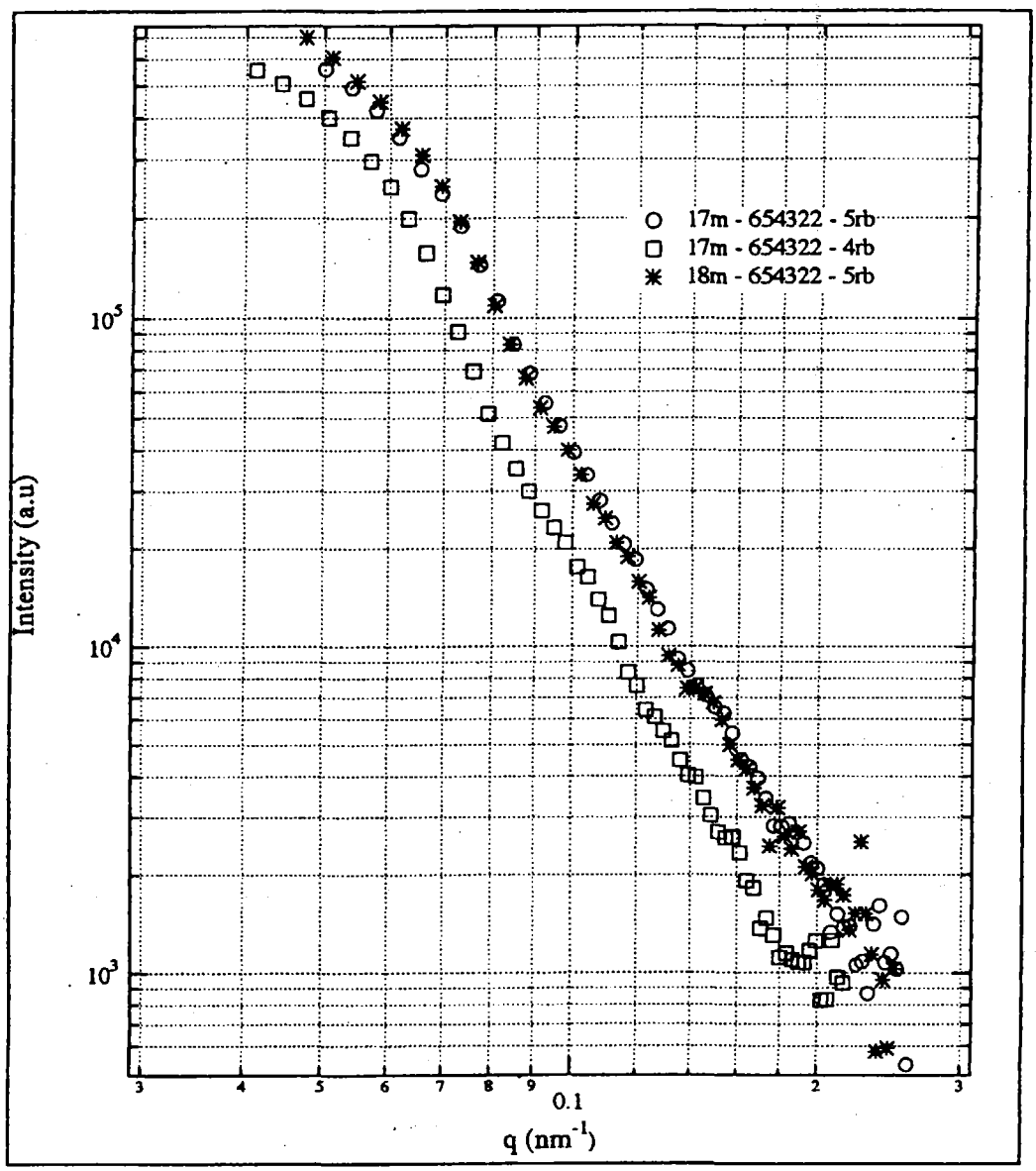

Figure 4. Polyball sample

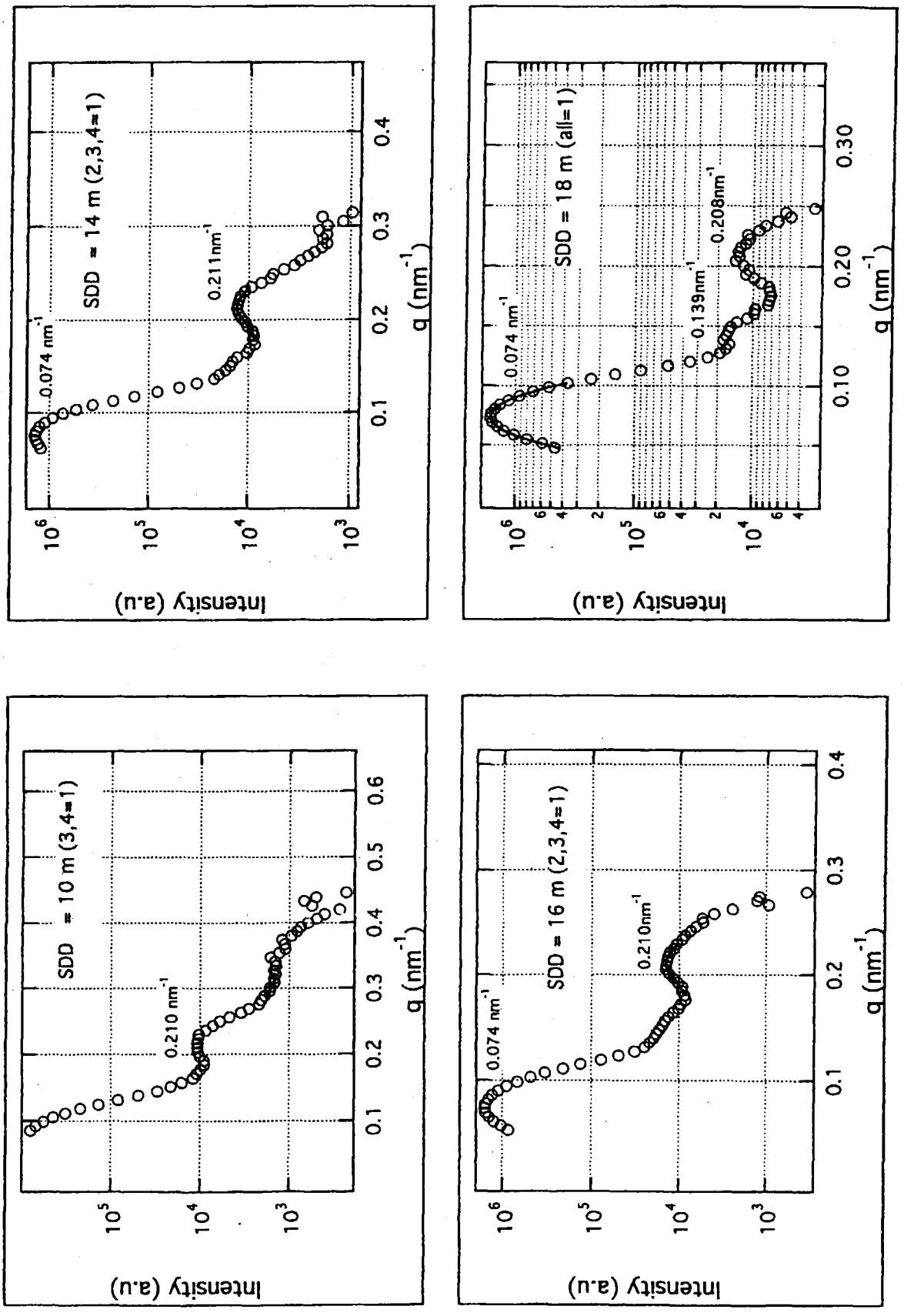

Figure 5. PS-PEP (EV) sample