実験報告書様式（一般利用課題・成果公開利用）

（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 ）

CROSS TOKAI **Experimental Report** J-PARC	**承認日** Date of Approval 2015/1/4 **承認者**Approver Jun-ichi Suzuki **提出日**Date of Report 2014/5/28
課題番号 Project No. 2013B0136 実験課題名 Title of experiment Elucidation of Conformations of Rigid and Well-defined Polymers by Small Angle Neutron Scattering 実験責任者名 Name of principal investigator Sota SATO 所属 Affiliation WPI-AIMR, Tohoku University	装置責任者 Name of Instrument scientist 装置名 Name of Instrument/(BL No.) BL-15 実施日 Date of Experiment 1st – 5th, March, 2014

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）

Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Polyquinoxaline polymer samples including C, H, N, and O atoms dissolved in halogen solvents or non-halogen solvents. The polymerization degrees of the measured samples were 20 to 200 mer. These polymers self-organize into rod, spiral conformation in solvents.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Recent years, the principal investigator of the project has worked on the development of magnetically aligned molecules for the NMR structure determinations. In general, molecules dissolved in solvents tumble randomly in the applied strong NMR magnetic field, which cancels dipole couplings between NMR active nuclei. Only when molecules slightly aligned against the applied NMR magnetic field, the molecules tumble anisotropically to show residual dipole couplings, affording unique three-dimensional information on the molecular structures.

In this work, the structures of polymers with well-defined polymerization degree synthesized by living polymerization reactions of aromatic quinoxaline derivatives were analyzed by small angle neutron scattering. The polymers are good candidates for the fundamental frameworks to realize controllable magnetic alignment due to the well-defined numbers of aromatic moieties included in the polymers and expected rigid, rod conformations to maintain the alignment directions.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

For the polymer samples dissolved in organic solvents were measured at room temperature under an atmospheric pressure in air with a standard experiment layout for solution analytes with several nanometers. The polymers with different polymerization degrees from 20 to 200 mer were analyzed, which molecular weights were precisely controlled by living polymerization method and determined by high resolution mass spectrometry.

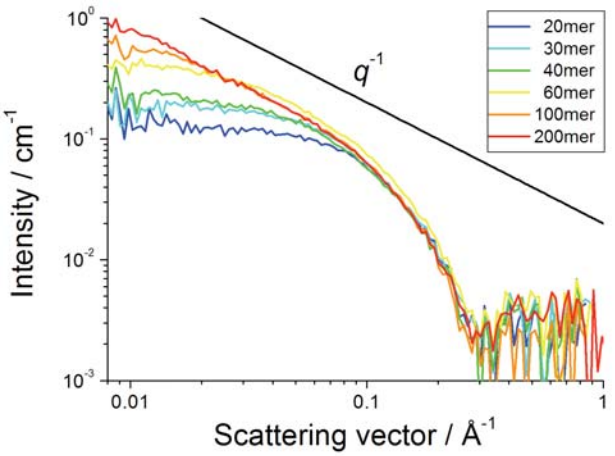

Figure 1. SANS profiles of polymers with different polymerization degrees dissolved in THF–d_8. The black straight line shows $I(q) \sim q^{-1}$, which represents the scattering profile of a thin rod in the low q–region.

The results of SANS profiles of the polymer samples showed different curves depending on the polymerization degrees (Figure 1). The most important finding was that a thin, rod conformation of the polymers was clearly revealed in a bulk solution state. The lengths of the polymers extend with larger polymerization degree, which is consistent to the stable, straight rod conformation of the polymers. More detailed analyses with Guinier plots afforded the information about the radius, R, of the rod polymers (Figure 2). The obtained R value of 11.4 ± 0.2 Å is well matched with the predicted structures by theoretical calculations. The rod conformation was basically maintained in different types of organic solvents, and the details are under analyses with the aid of theoretical methods to fully understand the solution-state behavior of the polymeres.

In conclusion, detailed solution conformation of the polymer samples were firmly determined to be straight rods. Due to the well–defined molecular weight estimated by mass spectrometry, three–dimensional molecular structures in solution state were visualized by the present SANS experiments. The understanding of the behavior will be reflected to the development of magnetic alignment materials.

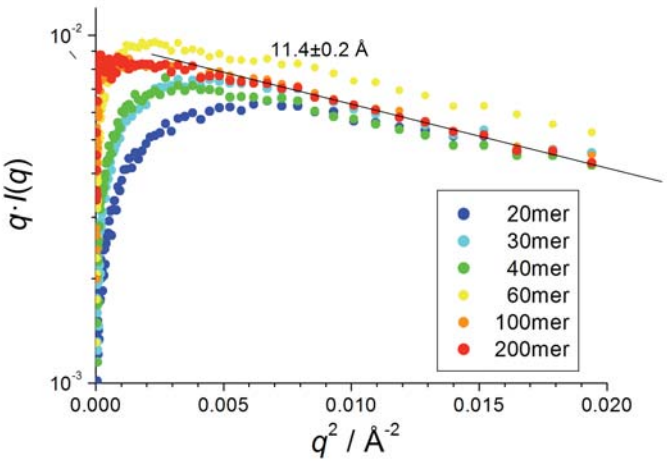

Figure 2. Cross sectional Guinier plots ($\log(qI(q))$ vs q^2) of polymers with different polymerization degrees dissolved in THF–d_8. The straight line shows that the sample is a thin rod shape, and the slope represents the radius of the rod, R, with the following manner; $\log(qI(q)) = A-(R^2q^2)/4$.