（ ※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 2016/6/8
承認者 Approver Jun-ichi Suzuki
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課題番号 Project No.
2014B0051

実験課題名 Title of experiment
Investigation of the Mechanism of the Solvent-dependent Helix Inversion of Poly(quinoxaline-2, 3-diyl)s by Small Angle Neutron Scattering

実験責任者名 Name of principal investigator
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Jun-ichi Suzuki

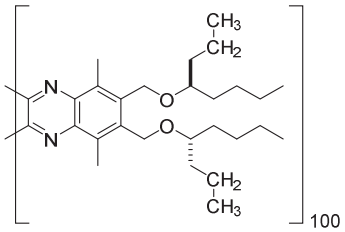
装置名 Name of Instrument/(BL No.)
Taikan / BL15

実施日 Date of Experiment
3/19(土) 10:00 – 3/23(水) 9:00

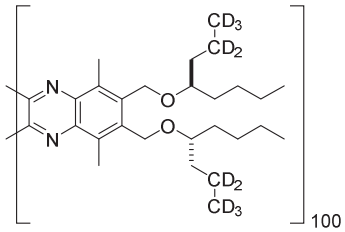
試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

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

Poly(quinoxaline-2,3-diyl)s bearing normal or partially deuterated 4-octyloxymethyl side chains (**4-Oct-H** and **4-Oct-D**, see below). These polymers were dissolved in tetrahydrofurane-d_8, *n*-octane-d_{18}, or cyclohexane-d_{12}.



4-Oct-H



4-Oct-D

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.

Recently we found that poly(quinoxaline-2,3-diyl)s bearing 4-octyloxymethyl side chains exhibit solvent-dependent helix inversion between *n*-octane and cyclohexane. In order to reveal a detailed mechanism of the helix inversion of poly(quinoxaline-2,3-diyl)s with 4-octyloxymethyl side chains, we carried out the small-angle neutron scattering (SANS) experiments of the polymer in tetrahydrofurane-d_8, *n*-octane-d_{18}, or cyclohexane-d_{12}. We prepared poly(quinoxaline-2,3-diyl)s bearing normal and partially deuterated 4-octyloxymethyl side chains (**4-Oct-H** and **4-Oct-D**) to distinguish between the propyl moiety and the butyl moiety in the chiral side chains. We also prepared quartz cells with a high vacuum greaseless stopcocks to suppress evaporation of volatile organic solvents (Figure 1). The sealability of the quartz cell was confirmed by enclosing chloroform before use (the weight loss was less than 0.02% after 24 hours at room temperature).

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

Figure 1. A quartz cell with a high vacuum greaseless stopcock. The path length was 2.0 mm.

4-Oct-H and **4-Oct-D** (ca. 3 mg) were dissolved in 700 μL of tetrahydrofurane-d_8, *n*-octane-d_{18}, or cyclohexane-d_{12}, and the solutions were enclosed within the quarts cells. We then carried out the SANS experiments of these solutions at 20 °C with BL15 "Taikan" diffractometer at MLF, J-PARC (Figure 2). In the low q region (q < 0.2 Å^{-1}), the SANS patterns indicated that **4-Oct-H** and **4-Oct-D** had rigid rod-like main chains, which were in good agreement with our previous report (*Macromolecules*, **2015**, *48*, 7983). In the high q region (0.2 Å^{-1} < q < 0.7 Å^{-1}), the SANS patters of **4-Oct-H** and **4-Oct-D** showed small peaks, arising from the chiral side chains. It should be noted that the small peaks of **4-Oct-H** and **4-Oct-D** were different, reflecting the conformation of the chiral side chains. We are now trying to reveal the detailed conformations of **4-Oct-H** and **4-Oct-D** in these solutions, by using theoretical calculations and SANS simulations.

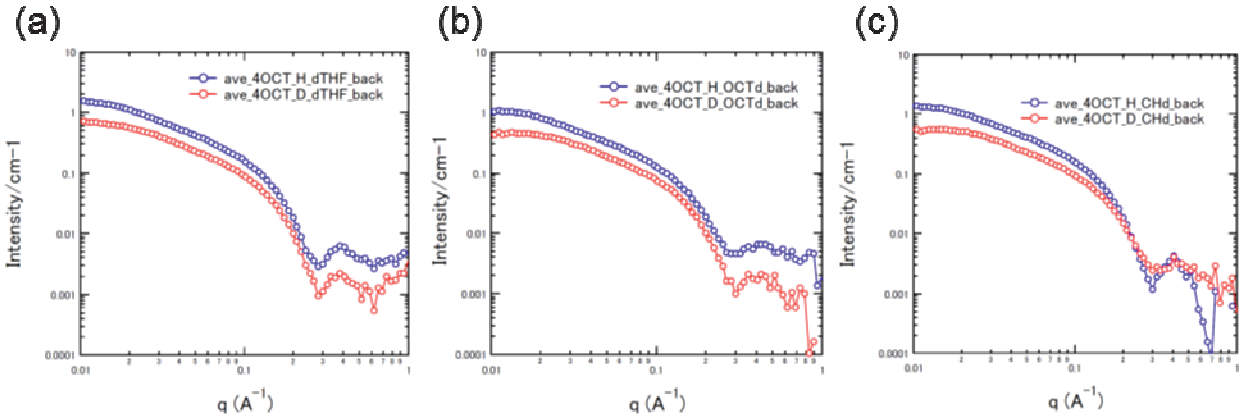


Figure 2. SANS patterns of **4-Oct-H** and **4-Oct-D** in (a) tetrahydrofurane-d_8, (b) *n*-octane-d_{18}, and (c) cyclohexane-d_{12}.