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 	承認日 Date of Approval 2017/12/9 承認者 Approver Jun-ichi Suzuki 提出日 Date of Report 2017/4/11
課題番号 Project No. 2016B0170 実験課題名 Title of experiment Analysis of structural homogeneity of syndiotactic polypropylene gels using small-angle neutron scattering 実験責任者名 Name of principal investigator Tomoki Maeda 所属 Affiliation Keio University	装置責任者 Name of responsible person Hiroki Iwase 装置名 Name of Instrument/(BL No.) TAIKAN/BL15 実施日 Date of Experiment 2016/2/21-2/22

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. Name of samples: Syndiotactic polypropylene (sPP)/deuterated-decalin (d-decalin) gel Chemical formula of sPP: $(C_3H_6)_n$ Chemical formula of d-decalin: $C_{10}D_{18}$ Compositions including physical form: sPP/(sPP+d-decalin) = 0 – 0.1, Gel (solid)
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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. <b>【Experimental method】</b> The sPP was dissolved into d-decalin at 160°C. The sPP/d-decalin solutions were cooled slowly at room temperature (RT) or quenched using liquid nitrogen (LN) to form sPP/d-decalin gels. The sPP/d-decalin gels slowly cooled at RT were named as GEL-RT, whereas the gels quenched using LN were named as GEL-LN. Small-angle neutron scattering (SANS) measurements were performed on TAIKAN instrument. A quartz cells with the thickness of 1 mm were used. The measurement time was set to 1 hr for the pure solvent of decalin-d and 1.5 hr for the sPP/d-decalin gels, respectively. Furthermore, very small angle neutron scattering (V-SANS) measurements were also performed by using RPMT detector to obtain the profiles in the Q range below $0.006 \text{ \AA}^{-1}$ . The measurement time was set to 2.5 hr.
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## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

### 【Results】

The SANS profiles of GEL-RT and GEL-LN were shown in Figure. When focusing on the profiles in the range of  $Q < 0.003 \text{ \AA}^{-1}$ , which were measured by using RPMT detector, the scattering intensity of GEL-RT showed strong upturn whereas the intensity of GEL-LN showed a moderate upturn. The upturn observed in the SANS profiles could be due to the heterogeneity in the sPP/d-decalin gel. Therefore, it was indicated that the heterogeneity of PP gel was suppressed for GEL-LN as compared with GEL-RT.

As for the profile of GEL-LN in the range of  $0.01 \text{ \AA}^{-1} < Q < 0.1 \text{ \AA}^{-1}$ , the scattering intensity decreased according to the negative power law ( $Q^{-1}$ ), and then decreased according to the negative fourth power law ( $Q^{-4}$ ). The scattering intensity of GEL-RT also decreased according to the negative power law ( $Q^{-1}$ ), and then decreased according to the negative fourth power law ( $Q^{-4}$ ). The  $Q$  value where the slope changed from  $Q^{-1}$  to  $Q^{-4}$  for GEL-LN ( $Q = 0.04 \text{ \AA}^{-1}$ ) was larger than the  $Q$  values for GEL-RT ( $Q = 0.022 \text{ \AA}^{-1}$ ), indicating that the structure in GEL-LN was smaller than the structure in GEL-RT.

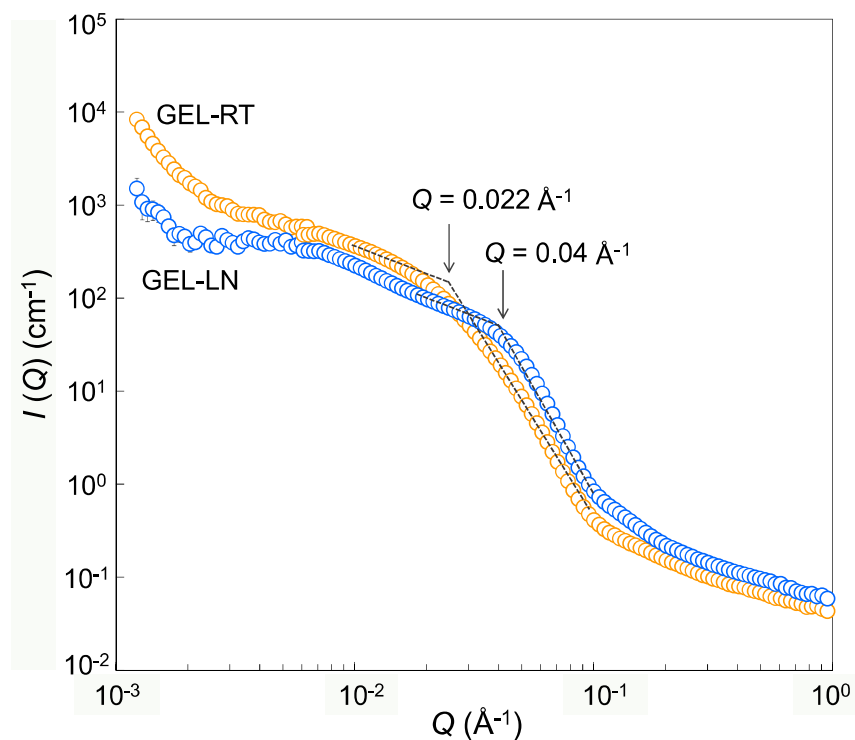


Figure. The scattering intensity profiles of GEL-LN and GEL-RT.