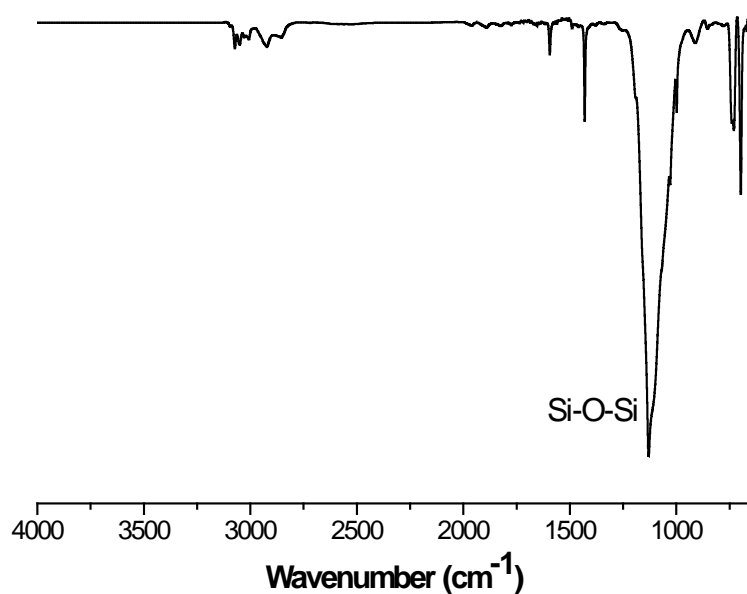


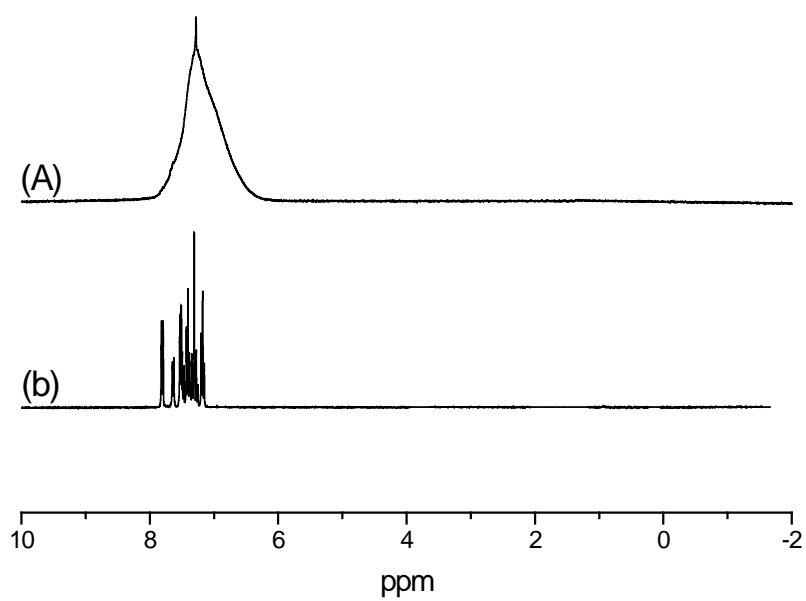
Supporting Information for

Structural Control of Fully Condensed Polysilsesquioxanes: Ladderlike vs. Cage Structured Polyphenylsilsesquioxanes

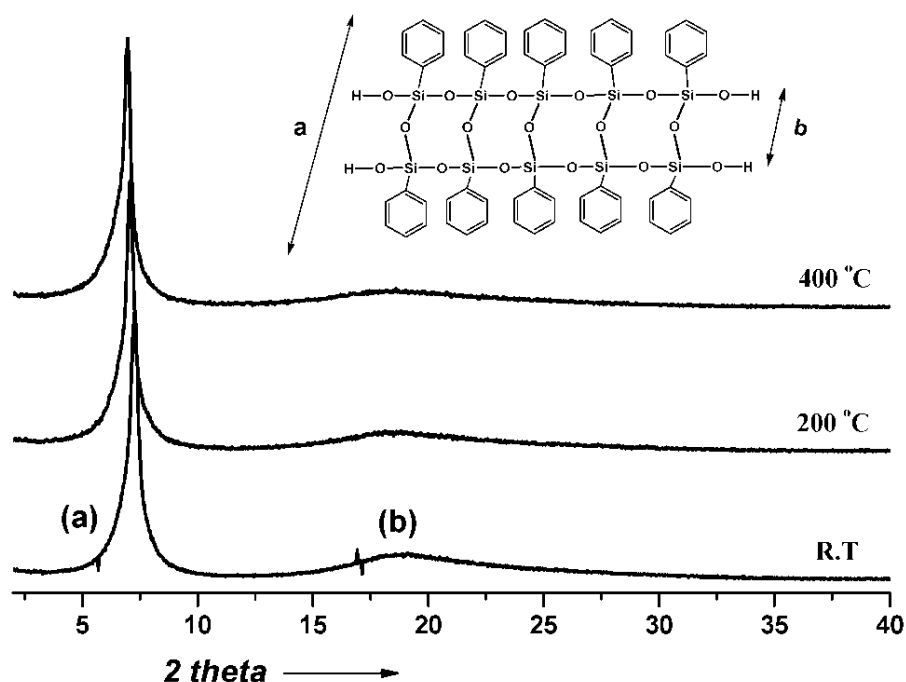
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Supporting Figure S1. FT-IR Spectra for T₁₂-Phenyl POSS



Supporting Figure S2. ^1H NMR Spectra of (A) LPPSQ and (B) T_{12} -Phenyl POSS



Supporting Figure S3. XRD pattern of LPPSQ due to temperature: periodic chain-to-chain distance (a) and thickness (b).

Supporting Figure S3 shows the X-ray diffraction (XRD) pattern of the obtained LPPSQ. Ladder-structured polysilsesquioxane typically shows two characteristic diffraction peaks in XRD patterns at various temperatures. The first sharp peak at 7.3° originated ($d_1 = 12.1 \text{ \AA}$) from the intramolecular periodic chain-to-chain distance (a) in the ladderlike structured LPPSQ. With the second diffusing peak at 18.8° ($d_2 = 4.7 \text{ \AA}$), a wide range of diffraction angles showed the average thickness (b) of the ladder-structured LPPSQ. In addition, XRD patterns of LPPSQ confirmed that thermal variation from room temperature to high temperature (400 °C) did not significantly alter the structure. This meant that synthetic LPPSQ was stable without thermal decomposition defects such as methoxy or intermediate hydroxyl groups.