Electronic Supplementary Information for:

Cosolvent-free synthesis and characterisation of poly(phenyl-*co-n***-alkylsilsesquioxane) and poly(phenyl-***co***-vinylsilsesquioxane) glasses with low melting temperatures** Ryosuke Seto,^{*a*} Kiyoshi Kanamura,^{*a*} Satoshi Yoshida^{*b*} and Koichi Kajihara^{*a**}

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Fig. S1 Magnified liquid-state ¹H single-pulse NMR spectra of poly(Ph-*co*-R-SQ) with R = Me, Et, Pr, and Vi (300 MHz, CDCl₃) normalized to the maximum value.



Fig. S2 Results of least-squares fitting of ¹H NMR spectra shown in Figs. 3 and S1. Solid and dashed lines denote observed and simulated spectra, respectively, and dotted lines indicate spectra of decomposed peaks. Each peak was expressed by one or two pseudo-Voigt functions, and its intensity (area) was assumed to be proportional to the number of ¹H nuclei attributed to the peak.



Fig. S3 Full-width at half-maximum (FWHM) and centroid of peaks for alkyl and vinyl groups (top) and phenyl groups (bottom), evaluated by the peak fitting and decomposition of ¹H NMR spectra shown in Fig. S2. Centroid is plotted as the shift from the peak position of corresponding trimethoxide (monomer).



Fig. S4 Variation of average number of bridging oxygen atoms, $\langle m \rangle$, with the fraction of R-SQ units, $f_{\rm R}$, in poly(Ph-*co*-R-SQ).