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**Supplementary Material**

**Key factors to prepare polyelectrolytes showing temperature-sensitive**

**LCST-type phase transition in water**

Yuki Kohno and Hiroyuki Ohno\*

\*Department of Biotechnology, Tokyo University of Agriculture and Technology,

2-24-16, Naka-cho, Koganei, Tokyo 184-8588, Japan

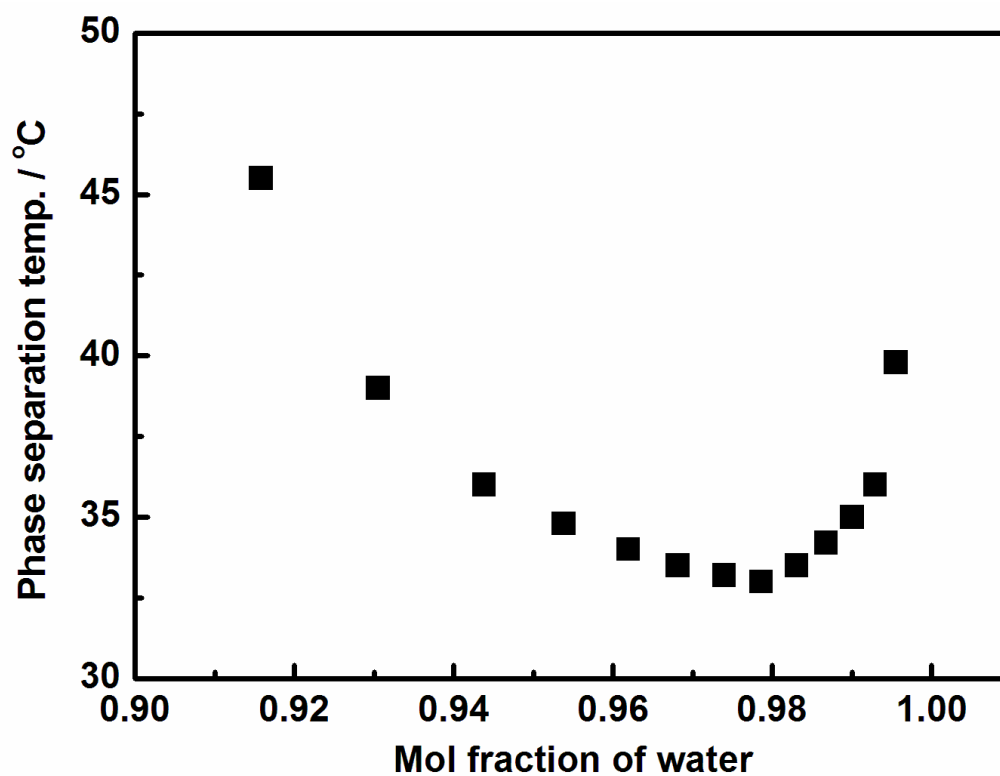
\*Corresponding Author: [ohnoh@cc.tuat.ac.jp](mailto:ohnoh@cc.tuat.ac.jp)

### Characterization of ILs

[P<sub>4444</sub>][SS]: <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, δ/ppm relative to TMS): 0.92(t, *J* 6.4, 12H), 1.43-1.44(m, 16H), 2.2(m, 8H), 5.2(d, *J* 10.5, 1H), 5.7(d, *J* 17.4, 1H), 6.7(q, *J* 28.4, 1H), 7.3(d, *J* 6.4, 2H), 7.8(t, *J* 7.8, 2H).

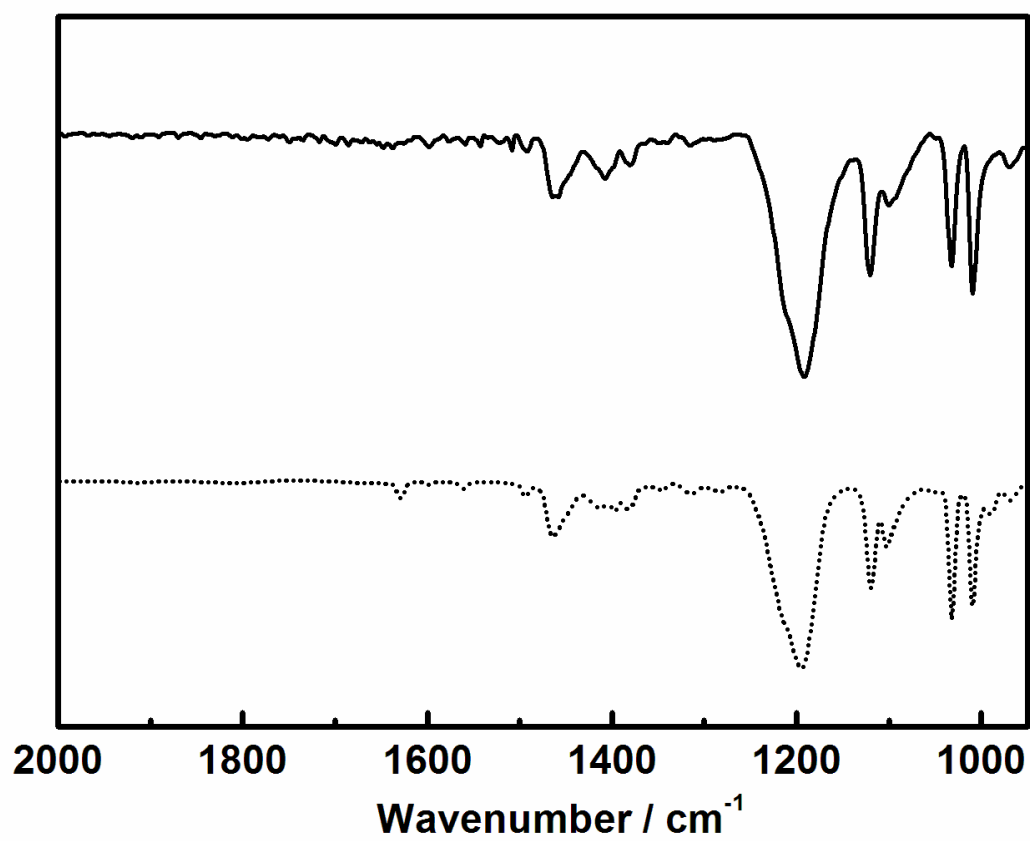
[P<sub>4446</sub>][SS]: <sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, δ/ppm relative to TMS): 0.85-0.93(m, 12H), 0.93-1.46(m, 20H), 2.18-2.23(m, 8H), 5.23(d, *J* 11.9, 1H), 5.72(d, *J* 18.3, 1H), 6.68(q, *J* 28.4, 1H), 7.34(d, *J* 7.8, 2H), 7.83(d, *J* 6.8, 2H).

Phase separation temperature of [P<sub>4444</sub>][SS]/water mixture

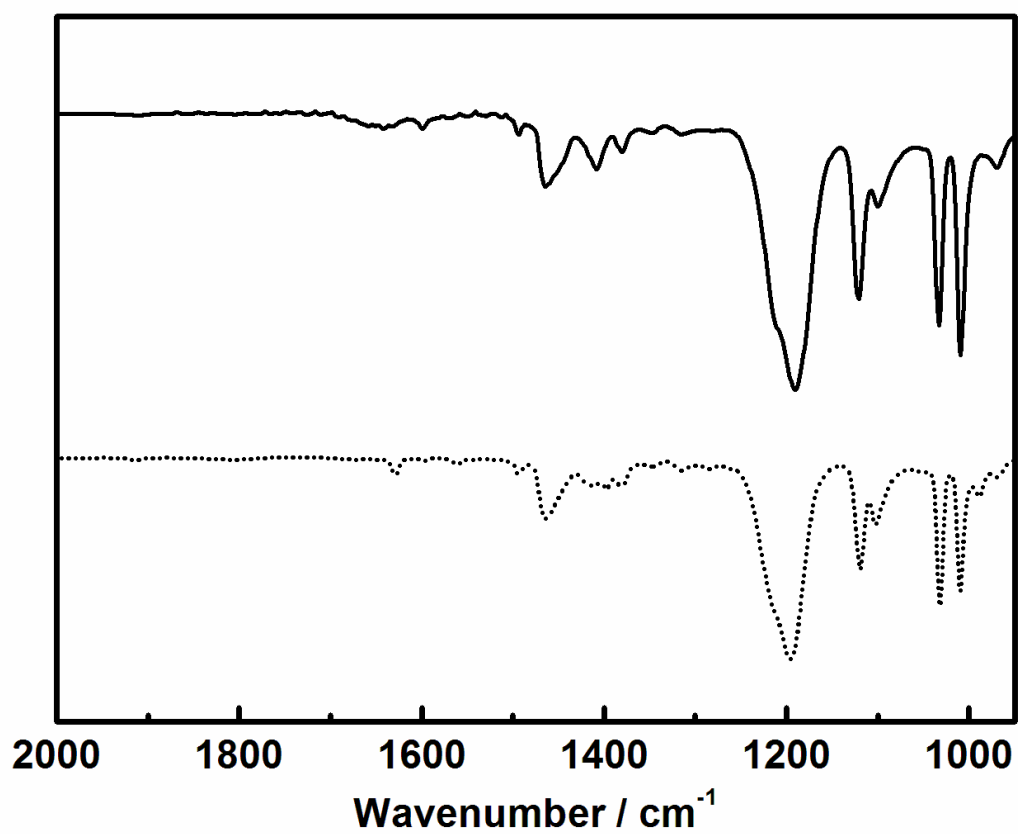


**Fig. S1.** Phase separation temperature of [P<sub>4444</sub>][SS] after mixing with different amount of water.

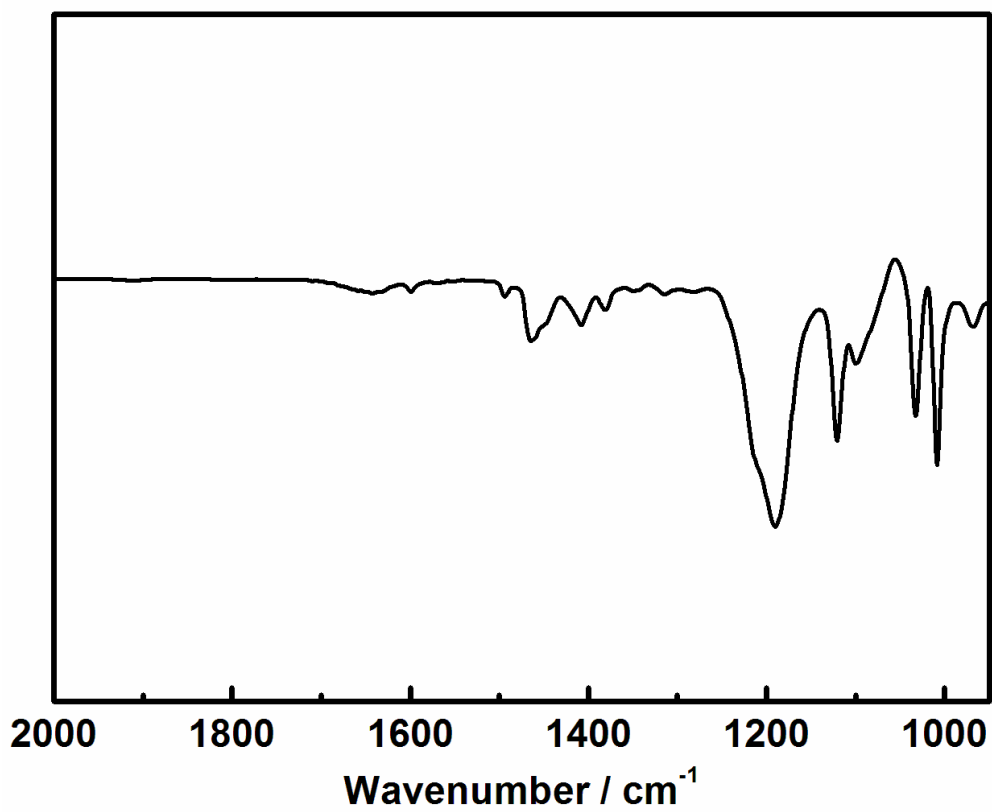
ATR-FTIR spectra of [P<sub>444n</sub>][SS] and their polymers



**Fig. S2.** ATR-FTIR spectra of [P<sub>444n</sub>][SS] (dotted line), and poly([P<sub>444n</sub>][SS]) (solid line).

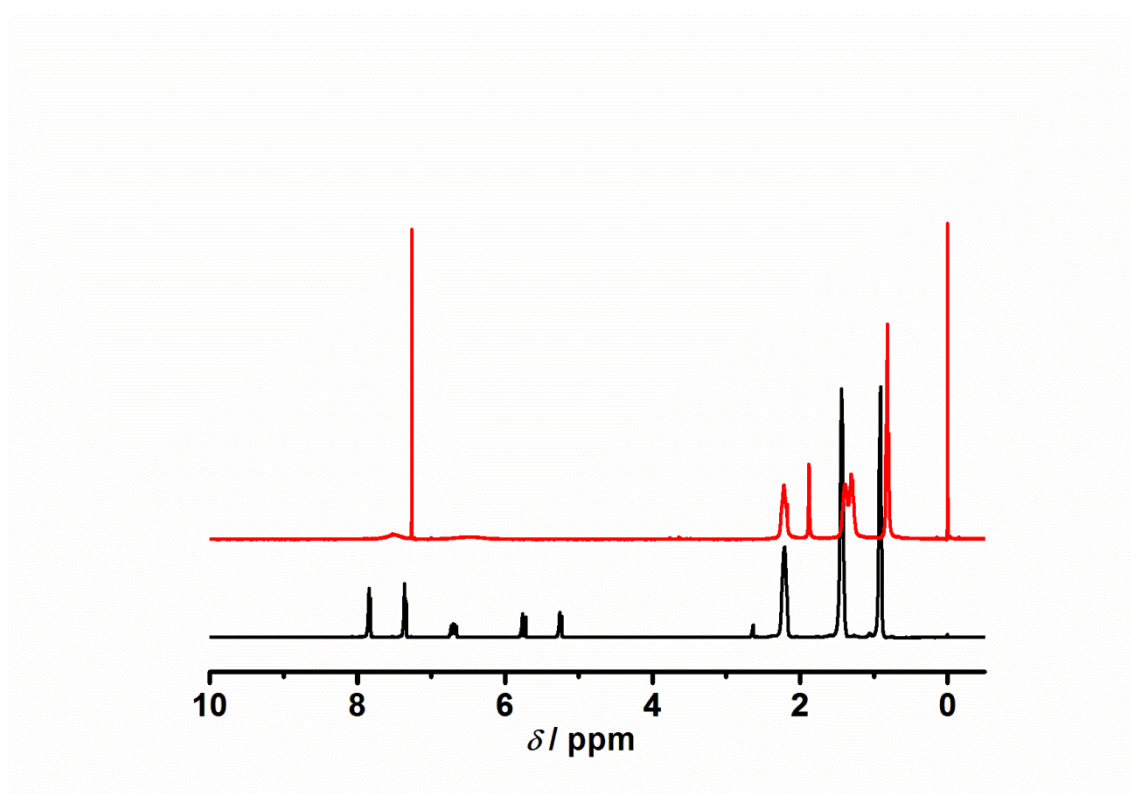


**Fig. S3.** ATR-FTIR spectra of [P<sub>4446</sub>][SS] (dotted line), and poly([P<sub>4446</sub>][SS]) (solid line).

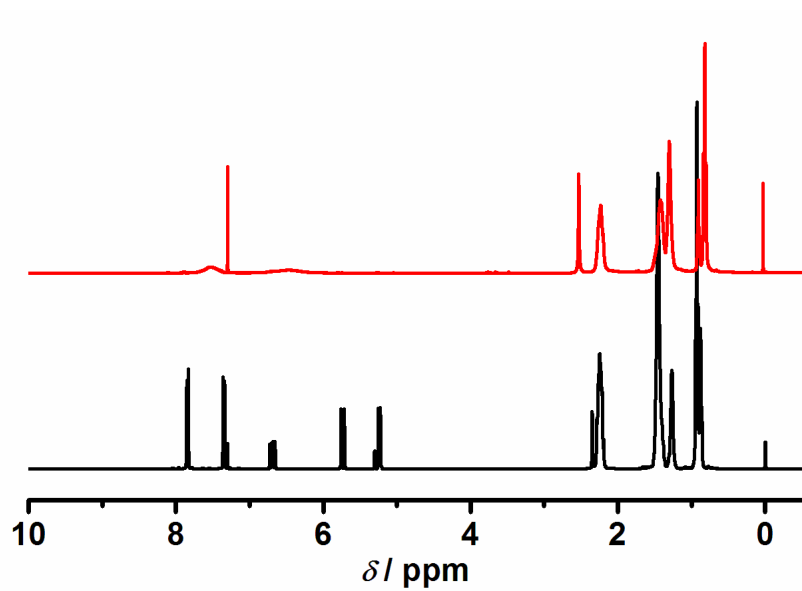


**Fig. S4.** ATR-FTIR spectra of poly([P<sub>4444</sub>][SS]<sub>0.7-co</sub>-[P<sub>4446</sub>][SS]<sub>0.3</sub>).

**$^1\text{H}$  NMR spectra of  $[\text{P}_{444n}][\text{SS}]$  and their polymers**

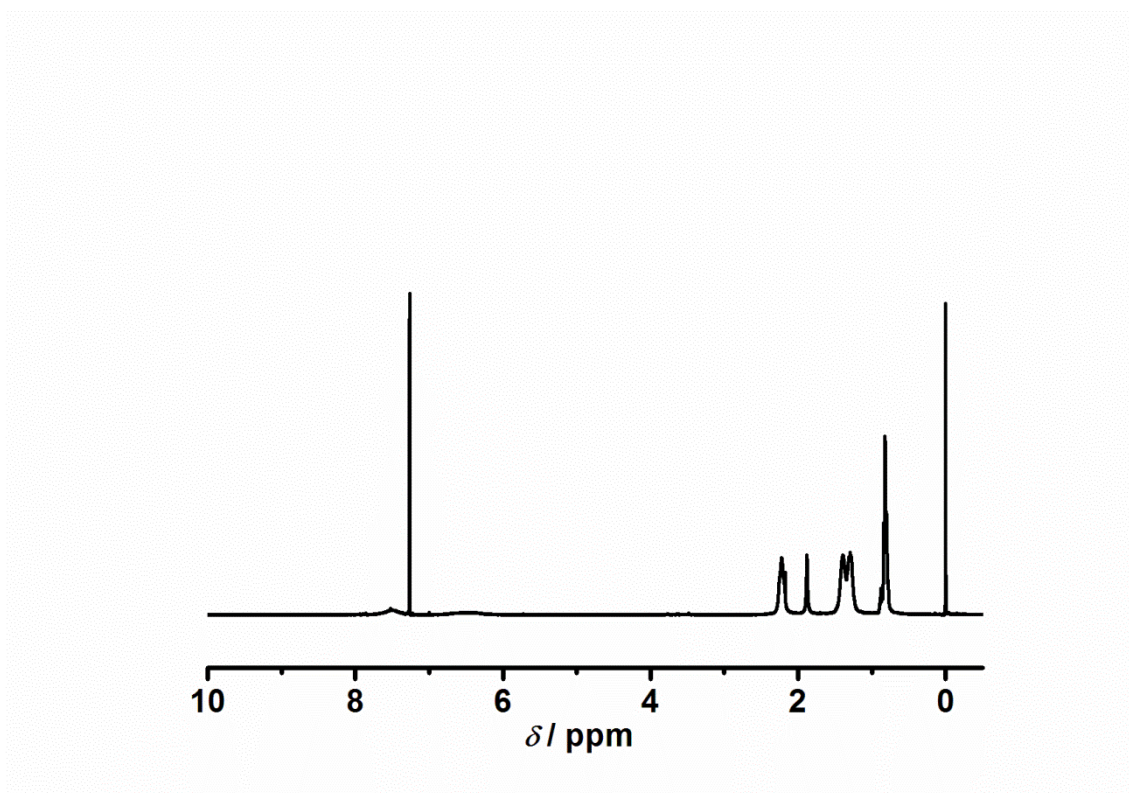


**Fig. S5.**  $^1\text{H}$  NMR spectra of  $[\text{P}_{444n}][\text{SS}]$  (black line), and poly( $[\text{P}_{444n}][\text{SS}]$ ) (red line).



**Fig. S6.** <sup>1</sup>H NMR spectra of [P<sub>4446</sub>][SS] (black line), and poly([P<sub>4446</sub>][SS]) (red line).





**Fig. S7.**  $^1\text{H}$  NMR spectra of poly( $[\text{P}_{4444}][\text{SS}]_{0.7}\text{-co-}[\text{P}_{4446}][\text{SS}]_{0.3}$ ).