

## Supporting Information

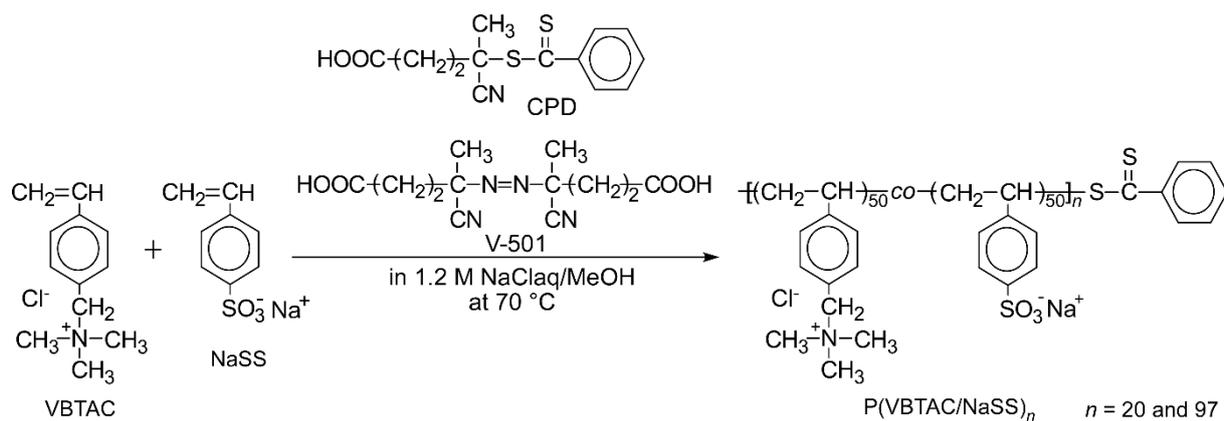
### Upper Critical Solution Temperature (UCST) Behavior of Polystyrene-based Polyampholytes in Aqueous Solution

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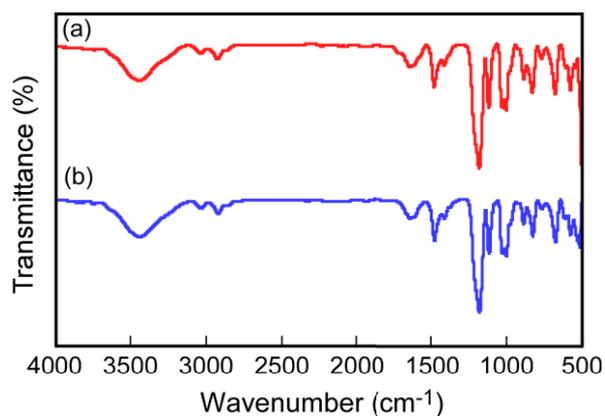
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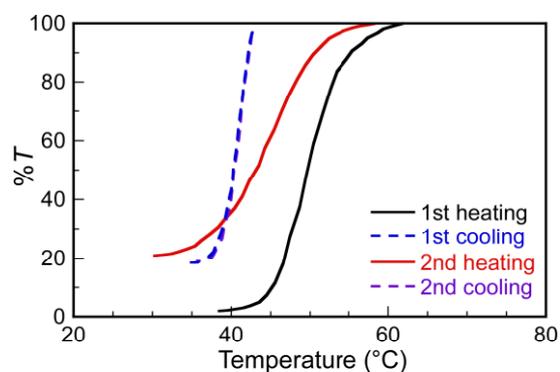
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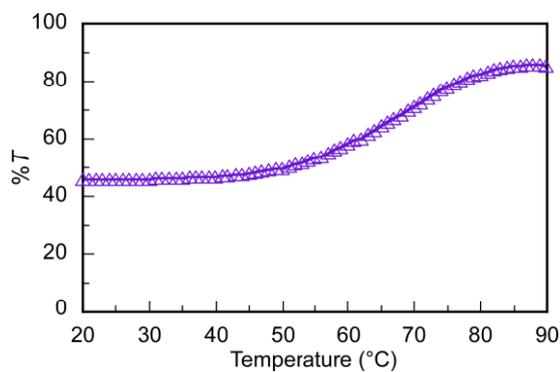
**Figure S1.** Synthesis scheme of P(VBTAC/NaSS)<sub>n</sub>.



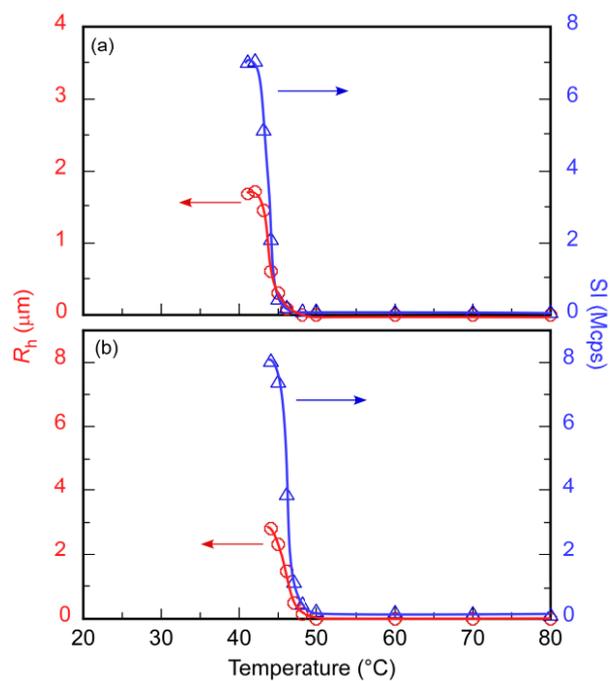
**Figure S2.** ATR-IR spectra for (a) P(VBTAC/NaSS)<sub>20</sub> and (b) P(VBTAC/NaSS)<sub>97</sub>.



**Figure S3.** Percent transmittance (%*T*) at 700 nm for 0.1 M NaCl aqueous P(VBTAC/NaSS)<sub>20</sub> at  $C_p = 1.0$  g/L as a function of temperature upon heating and cooling.



**Figure S4.** Percent transmittance (%*T*) at 700 nm for P(VBTAC/NaSS)<sub>97</sub> at  $C_p = 2.0$  g/L in pure water as a function of temperature upon cooling.



**Figure S5.** Hydrodynamic radii ( $R_h$ ,  $\circ$ ) and light scattering intensities (SI,  $\triangle$ ) for (a) P(VBTAC/NaSS)<sub>20</sub> at [NaCl] = 0.1 M and (b) P(VBTAC/NaSS)<sub>97</sub> at [NaCl] = 1.0 M with  $C_p = 2.0$  g/L as a function of temperature.

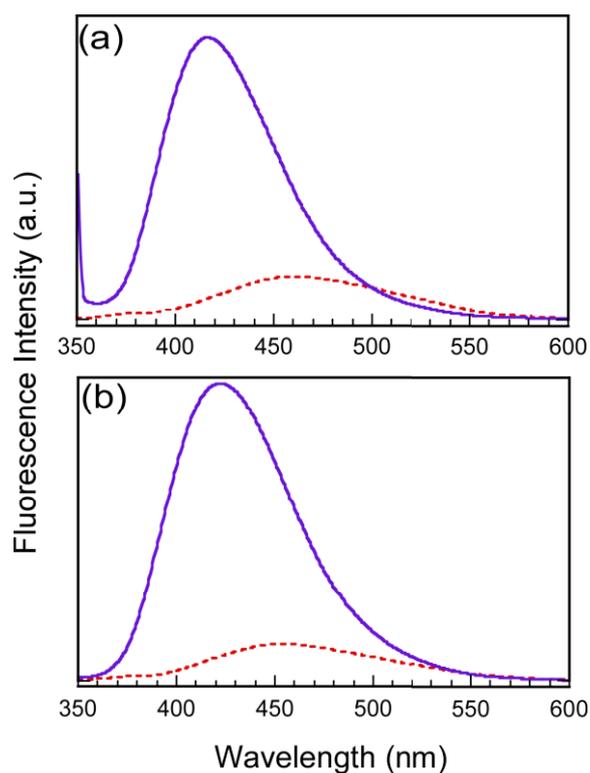


**Figure S6.** (a) Dispersion of P(VBTAC/NaSS)<sub>97</sub> just below the UCST and (b) flocculation of the polymer below the UCST upon standing for 2 h.

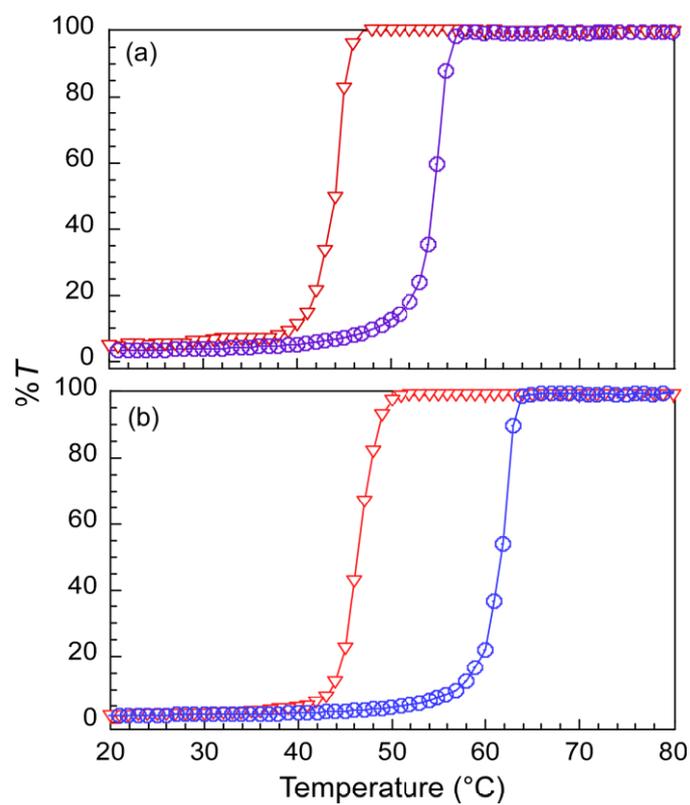
**Table S1.** Maximum fluorescence wavelength ( $\lambda_{\max}$ ) of PNA in the absence and presence of P(VBTAC/NaSS) $_n$  in aqueous solutions at 20 and 70 °C

Sample	without polymer <sup>a</sup>		P(VBTAC/NaSS) <sub>20</sub> <sup>b</sup>		P(VBTAC/NaSS) <sub>97</sub> <sup>c</sup>	
Temperature ( °C)	20	70	20	70	20	70
$\lambda_{\max}$ (nm)	463	452	412	417	417	423

<sup>a</sup>In pure water. <sup>b</sup>At  $C_p = 2.0$  g/L in 0.1 M NaCl solutions. <sup>c</sup>At  $C_p = 2.0$  g/L in 1.0 M NaCl aqueous solutions.



**Figure S7.** Typical examples of fluorescence emission spectra for PNA in the absence (---) and presence of P(VBTAC/NaSS)<sub>97</sub> (—) at  $C_p = 2$  g/L in 1.0 M NaCl aqueous solutions at (a) 20 and (b) 70 °C.



**Figure S8.** Percent transmittance (% $T$ ) at 700 nm for H<sub>2</sub>O ( $\nabla$ ) and D<sub>2</sub>O ( $\circ$ ) solutions of (a) P(VBTAC/NaSS)<sub>20</sub> at [NaCl] = 0.1 M and (b) P(VBTAC/NaSS)<sub>97</sub> at [NaCl] = 1.0 M with  $C_p = 2$  g/L as a function of temperature.