Supporting Information

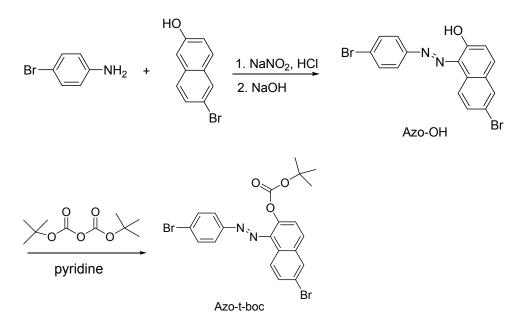
for

Synthesis of poly(N-isopropylacrylamide) polymer crosslinked with an AIE-active azonaphthol for thermoreversible fluorescence

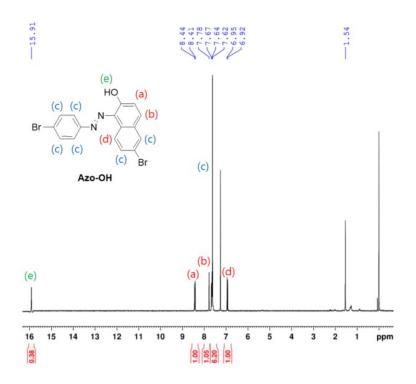
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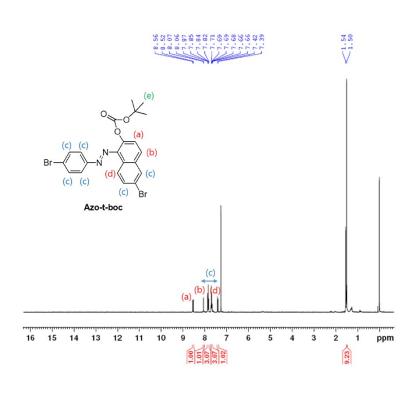
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Scheme S1. Synthesis of Azo-t-boc.



(a)



(b)

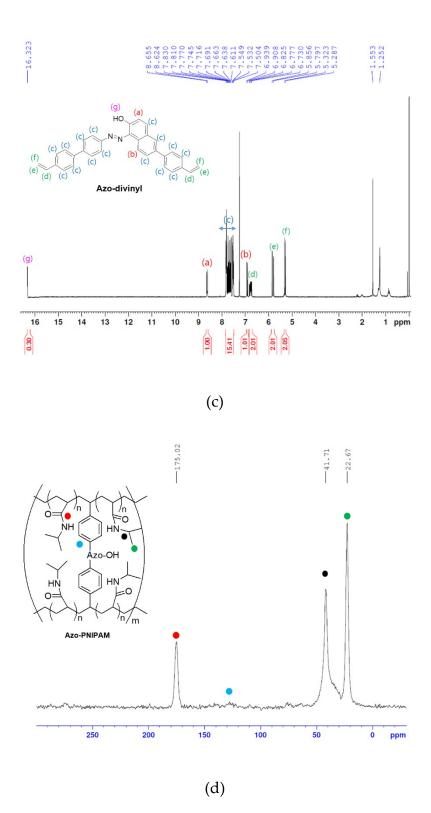


Figure S1. ¹H-NMR spectra of (a) Azo-OH, (b) Azo-t-boc, and (c) Azo-divinyl. (d) ¹³C solid-state NMR spectrum of Azo-PNIPAM.

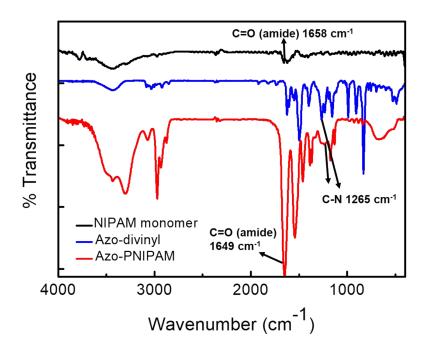


Figure S2. FT-IR spectra of NIPAM, Azo-divinyl, and Azo-PNIPAM.

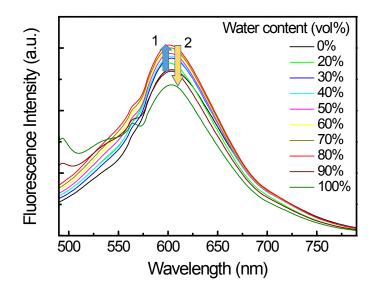
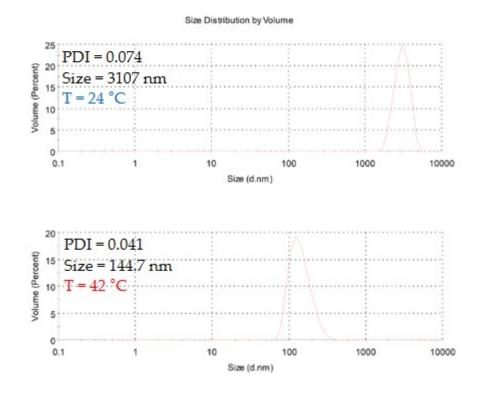
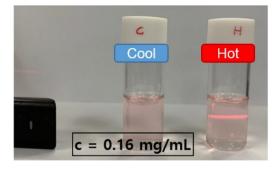


Figure S3. Changes in the fluorescence spectra of Azo-PNIPAM in THF solution (3 mg/mL) with increasing water content. Excitation wavelength 450 nm.



(a)



(b)

Figure S4. (a) Particle size distributions of Azo-PNIPAM in aqueous solution (1.1 mg/mL) (b) at 24 °C (above) and at 42 °C (below). (b) Visualization of deaggregation (left) and aggregation (right) of Azo-PNIPAM in aqueous solution (0.16 mg/mL) using a laser pointer (0.83 μ m), showing a beam path only in the right vial.

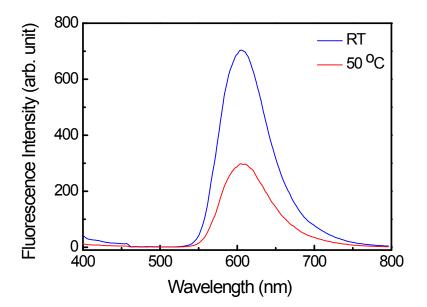


Figure S5. Changes in the fluorescence spectra of Azo-OH film at different temperatures.

Table S1. Summary of Literature Reports on Thermoresponsive Polymers Based onPNIPAM with AIEgen

Polymer	AIE unit	Fluorescence enhancement (%) upon heating	Ref.
PNIPAM-b-P4VP	4-(9-(2-(4-Hydroxyphenyl)ethynyl)- 7,10- diphenylfuoranthen-8-yl)phenol	210	39
PNIPAM-b-PDPA	TPE (Tetraphenylethylene)	250	52
PNIPAM-b-PMMA	TPE	240	67
PNIPAM	ANTH (Anthracene)	200	69
PNIPAM	TPE	150	68
PNIPAM	TPE	137	66
PNIPAM	Tetraphenylthiophene	107	70
PNIPAM	Azo-naphthol	130	This work

Fluorescence enhancement (%) = I/I $_{o}$ x 100 (%)

where I₀ and I correspond to fluorescence intensity before and after heating