Supporting Information

for

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

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**Scheme S1.** Synthesis of Azo-t-boc.
Figure S1. $^1$H-NMR spectra of (a) Azo-OH, (b) Azo-t-boc, and (c) Azo-divinyl. (d) $^{13}$C 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.
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 µm), 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 on PNIPAM with AIEgen

| Polymer          | AIE unit                      | Fluorescence enhancement (%) upon heating | Ref. |
|------------------|-------------------------------|------------------------------------------|------|
| PNIPAM-b-P4VP    | 4-(9-(2-(4-Hydroxyphenyl)ethynyl)-7,10-diphenylfluoranthen-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 (%) = \( I/I_0 \times 100 \) (%)

where \( I_0 \) and \( I \) correspond to fluorescence intensity before and after heating