

## SUPPORTING INFORMATION

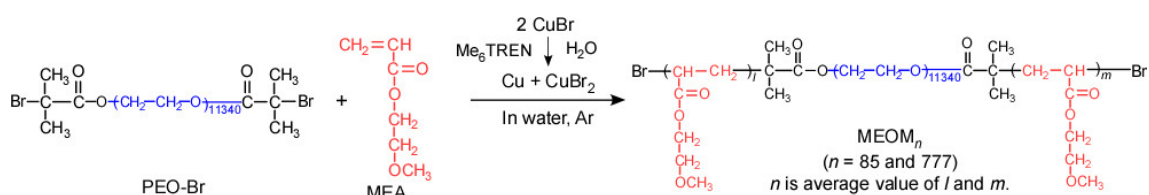
### Association Behavior of Amphiphilic ABA Triblock Copolymer Composed of Poly(2-methoxyethyl acrylate) (A) and Poly(ethylene oxide) (B) in Aqueous Solution

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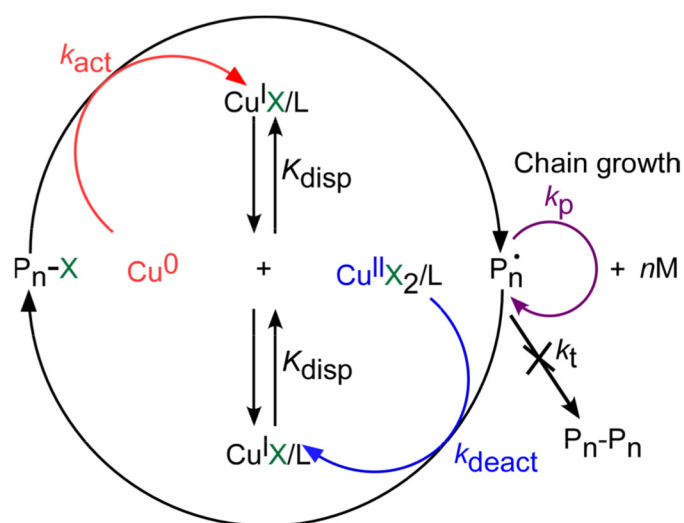
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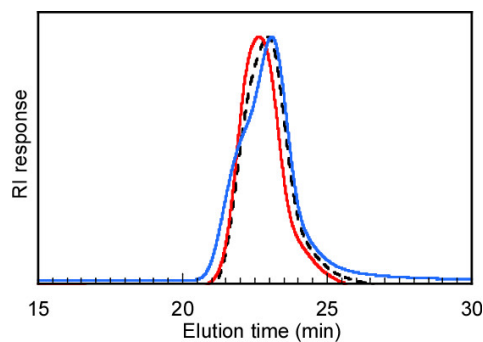
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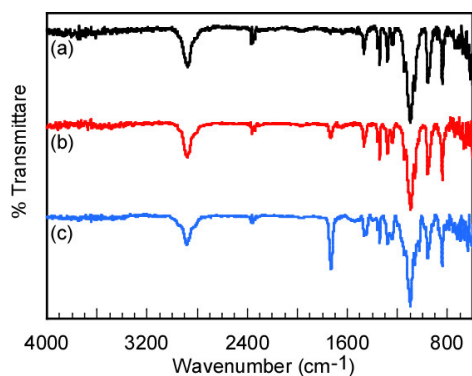
**Scheme S1.** Synthesis of MEOM<sub>n</sub> (n = 85 and 777).



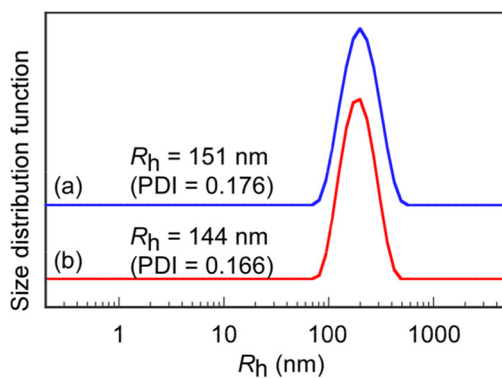
**Figure S1.** Mechanism of single-electron transfer-living radical polymerization (SET-LRP); M; monomer, P; polymer, Cu; copper, X; halogen, L; ligand,  $k_{\text{act}}$ ; activation,  $k_{\text{deact}}$ ; deactivation,  $k_p$ ; propagation,  $k_t$ ; termination.



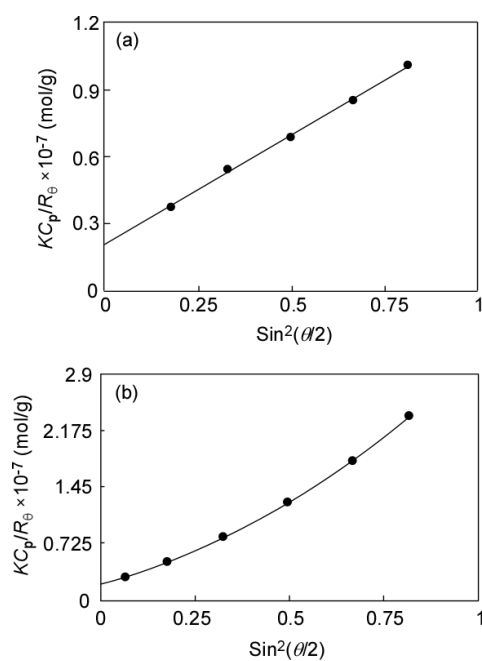
**Figure S2.** Gel-permeation chromatography (GPC) elution curves of PEO<sub>11340</sub>-Br (black line), MEOM<sub>85</sub> (red line), and MEOM<sub>777</sub> (blue line) using THF as an eluent with a flow rate of 1.0 mL/min at 40 °C.



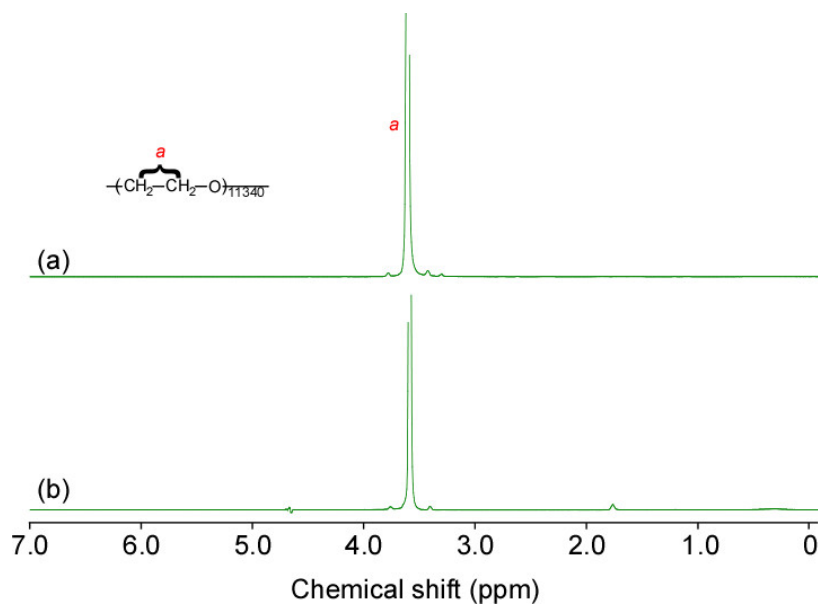
**Figure S3.** Attenuated total reflection (ATR) Fourier-transform infrared (FTIR) spectra of (a) PEO<sub>11340</sub>-Br, (b) MEOM<sub>85</sub>, and (c) MEOM<sub>777</sub>.



**Figure S4.** Hydrodynamic radius ( $R_h$ ) distributions of MEOM<sub>85</sub> aqueous solution prepared by directly dissolution (blue line) and by dialysis from THF solution against aqueous solution (red line); the final polymer concentration ( $C_p$ ) was adjusted to 0.1 g/L.



**Figure S5.** Debye plots for (a) MEOM<sub>85</sub> and (b) MEOM<sub>777</sub> in water.



**Figure S6.** Water suppression by gradient-tailored excitation (WATERGATE) <sup>1</sup>H NMR spectroscopy in D<sub>2</sub>O for (a) MEOM<sub>85</sub> and (b) MEOM<sub>777</sub> at 25 °C.