Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supporting Information

Current-controlled ‘plug-and-play’ electrochemical atom transfer radical polymerization of acrylamides in water

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Supporting figures and tables

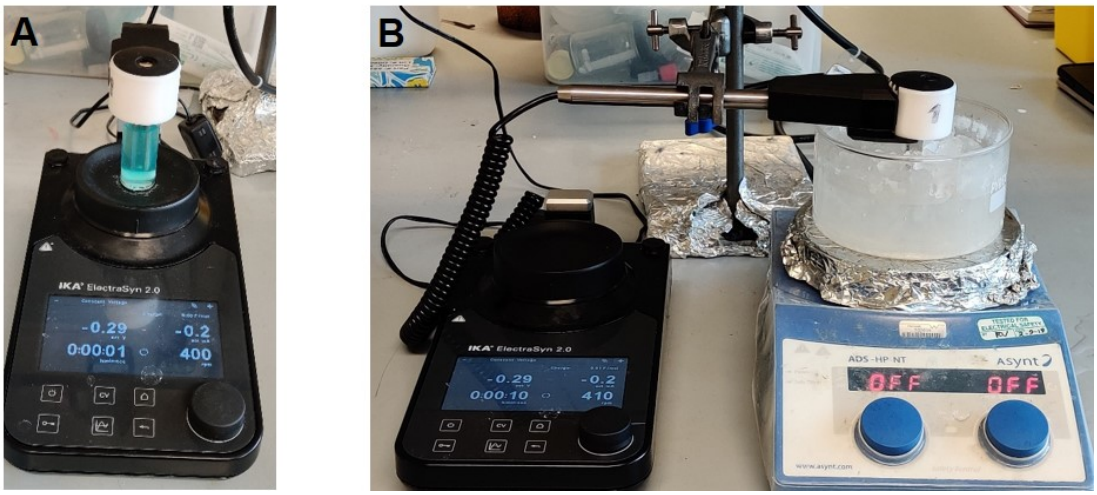

Fig S1. Example reaction configuration for seATRP at (A) room temperature; (B) 0 °C.

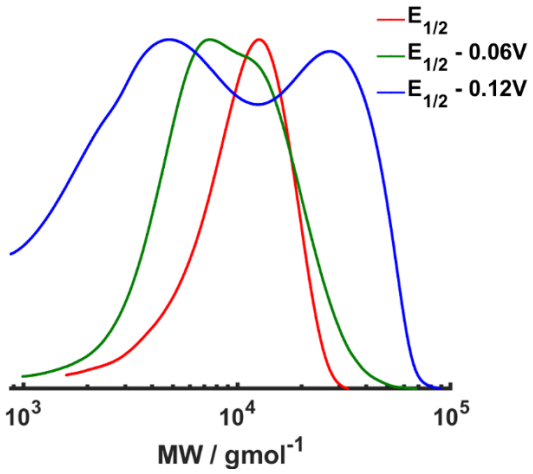

Fig S2. SEC in DMF for potentiostatic seATRP of 10 wt. % HEAm in H_2O + 0.1 M NaBr + 0.1 M potassium nitrate at room temperature performed at three different applied potentials. Conditions: [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.1] : [0.4] : [0.1].

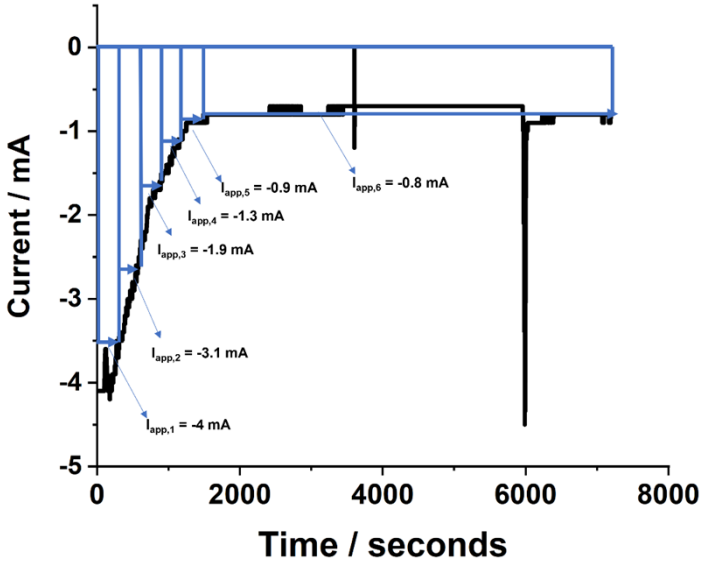

Fig S3. Current vs time plot recorded during the potentiosatic (E_{app} = $E_{1/2}$ = -0.13 V) seATRP of HEAm using [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.1] : [0.4] : [0.1] at room temperature.

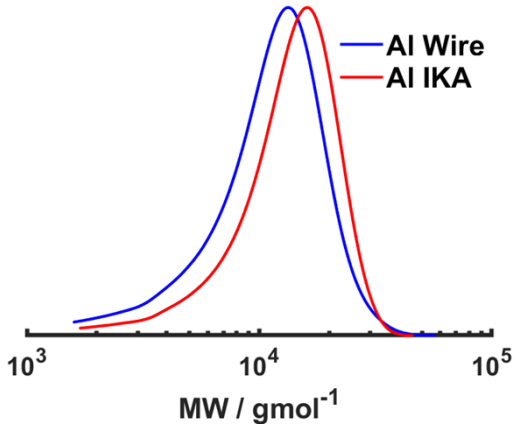

Fig S4. SEC of PHEAm from the current controlled seATRP of HEAm using [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.1] : [0.4] : [0.1] at room temperature. Reactions were conducted with an Al wire (blue, $M_{n,SEC}$ = 9000 g.mol^{-1}, $Đ_m$ = 1.39) and a commercial Al electrode from IKA (red, $M_{n,SEC}$ = 11000 g.mol^{-1}, $Đ_m$ = 1.32) as counter electrode.

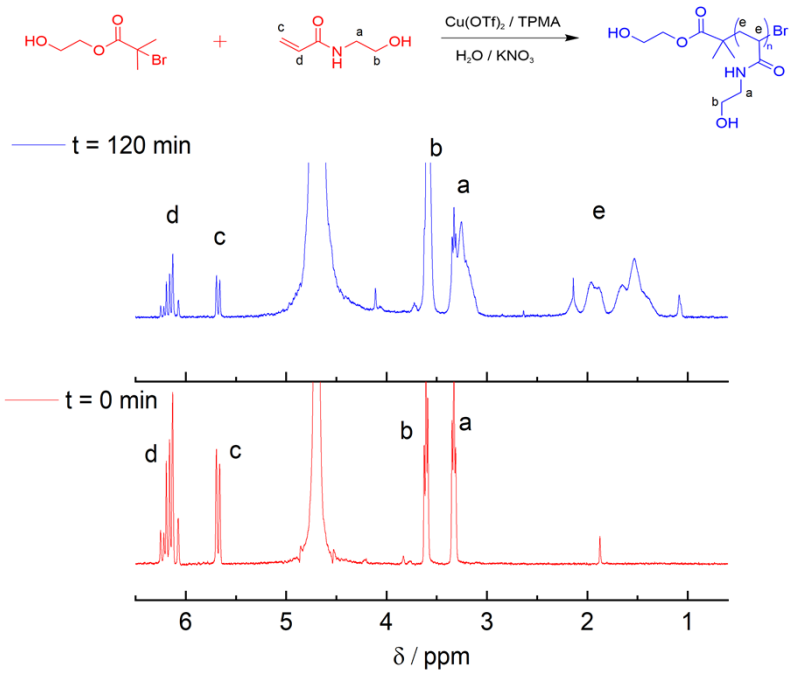

Fig S5. NMR of PHEAm from the current controlled seATRP of HEAm using [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.1] : [0.4] : [0.1] at room temperature. Conversion = 86 %, $M_{n,SEC}$ = 11000 g.mol^{-1}, $Đ_m$ = 1.31. Conversion calculated from the relative integrals of H_c and H_a

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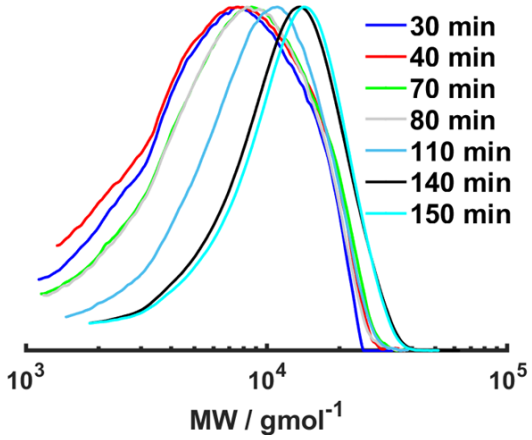

Fig S6. The evolution of the molecular weight distribution of PHEAm during the temporal control experiment conducting using [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.1] : [0.4] : [0.1] at room temperature. Between t = 0 and t = 30 min I_{app} = -4 mA (5 min), -3.1 mA (5 min), -1.9 mA (5 min), -1.3 mA (5 min), -0.9 mA (5 min) and -0.8 mA (5 min). Thereafter, during 10 min intervals I_{app} = 0 mA and during 30 min intervals I_{app} = -0.8 mA. For the final PHEAm $M_{n,SEC}$ = 9200 g.mol^{-1} and $Đ_m$ = 1.41.

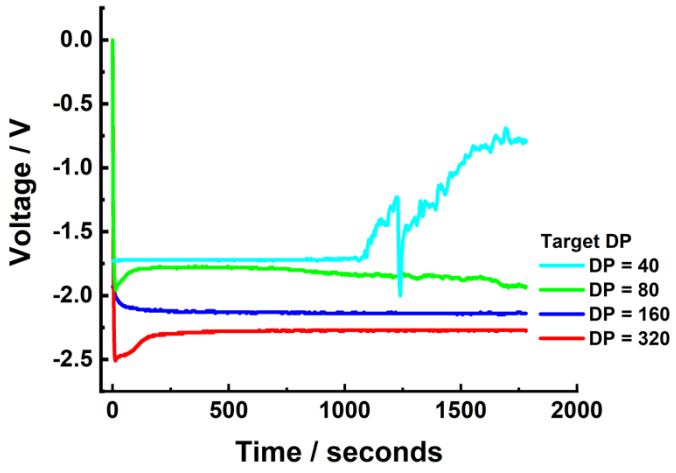

Fig S7. Voltage vs time plot generated during the first 30 minutes of the current controlled seATRP of HEAm as a function of [HEAm] / [HEBiB] at room temperature using the stepwise current profile; I_{app} = -4 mA (5 min), -3.1 mA (5 min), -1.9 mA (5 min), -1.3 mA (5 min), -0.9 mA (5 min) and -0.8 mA (5 min).

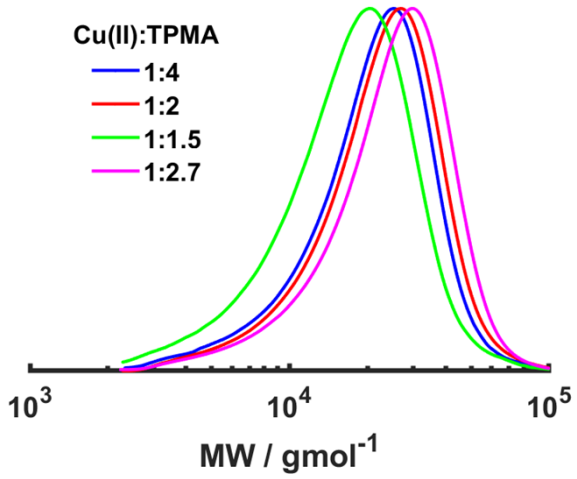

Fig S8. SEC of PHEAm from the current controlled seATRP of HEAm using [HEAm] / [HEBiB] = [160] at 0°C using [Cu^{II}] : [TPMA] = [1] : [4] (blue, $M_{n,SEC}$ = 15900 g.mol^{-1}, $Đ_m$ = 1.49); [1] : [2.7] (magenta, $M_{n,SEC}$ = 18700 g.mol^{-1}, $Đ_m$ = 1.47); [1] : [2] (red, $M_{n,SEC}$ = 17500 g.mol^{-1}, $Đ_m$ = 1.46); [1] : [1.5] (green, $M_{n,SEC}$ = 13200 g.mol^{-1}, $Đ_m$ = 1.51).

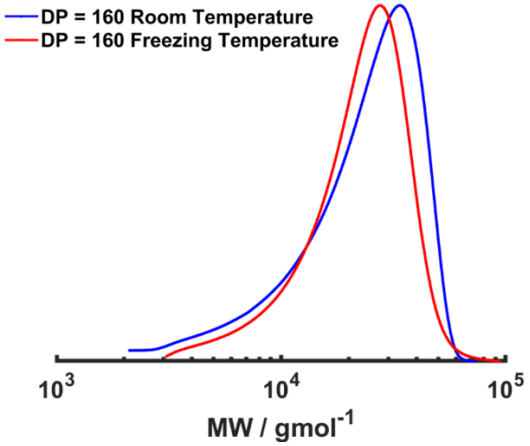

Fig S9. SEC of PHEAm from the current controlled seATRP of HEAm using [HEAm] / [HEBiB] = [160] at room temperature (blue, $M_{n,SEC}$ = 16500 g.mol^{-1}, $Đ_m$ = 1.55) and 0°C (red, $M_{n,SEC}$ = 17700 g.mol^{-1}, $Đ_m$ = 1.38).

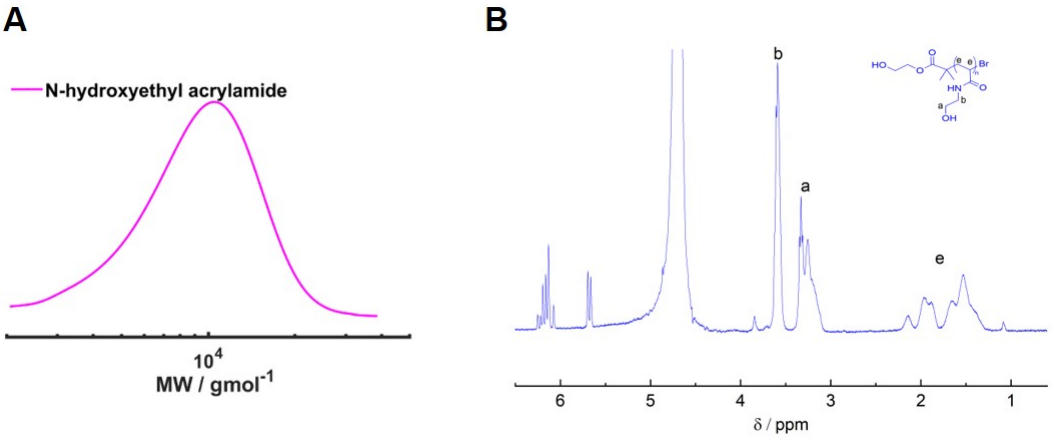

Fig S10. NMR and SEC of PHEAm from the current controlled seATRP of HEAm using [HEAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.3] : [0.8] : [0.1] at 0°C. Conversion = 83 %, $M_{n,SEC}$ = 8000 g.mol^{-1}, $Đ_m$ = 1.25.

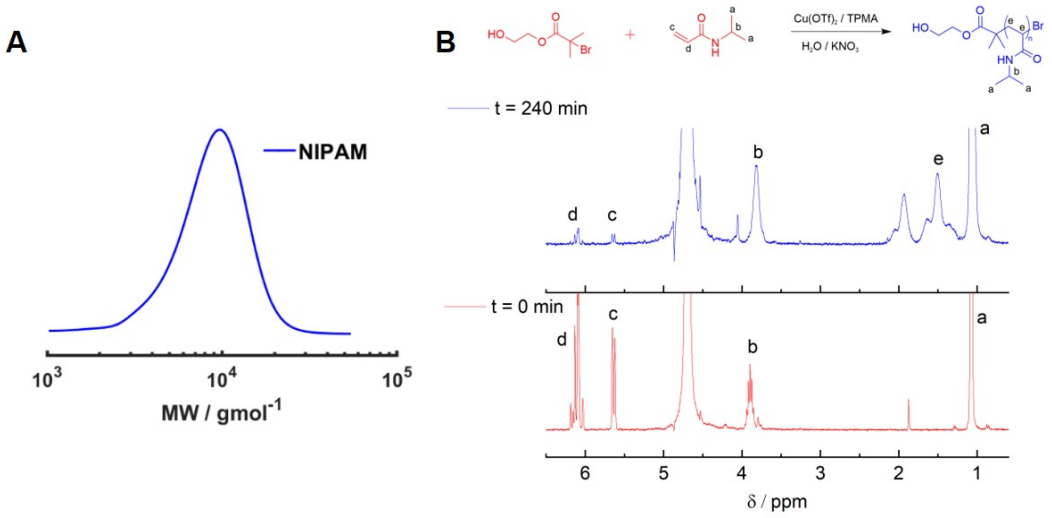

Fig S11. NMR and SEC of PNIPAm from the current controlled seATRP of NIPAm using [NIPAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.3] : [0.8] : [0.1] at 0°C. Conversion was calculated from the relative integrals of H_c and H_b. Conversion = 96 %, $M_{n,SEC}$ = 7300 g.mol^{-1}, $Đ_m$ = 1.31.

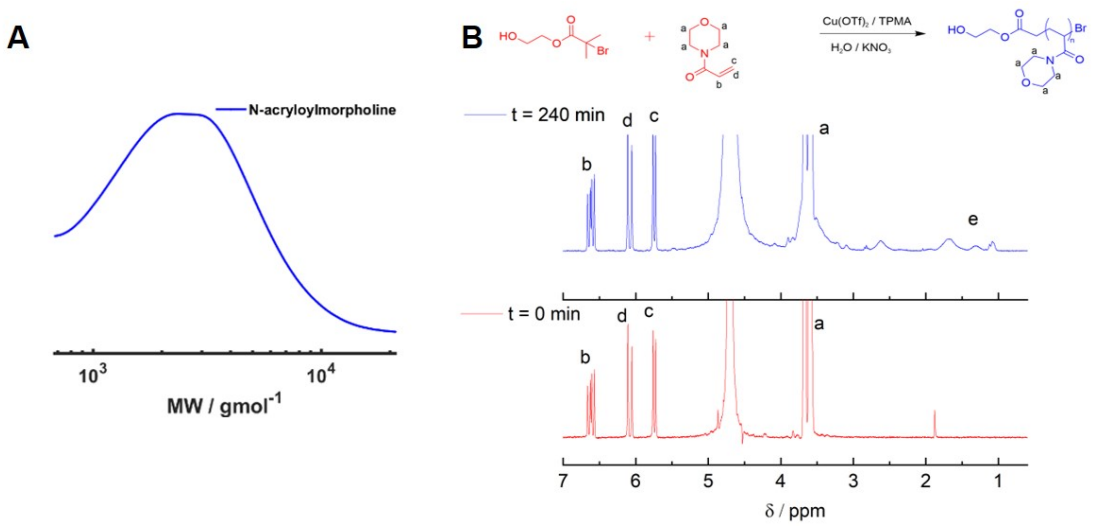

Fig S12. NMR and SEC of PNAM from the current controlled seATRP of NAM using [NAM] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.3] : [0.8] : [0.1] at 0°C. Conversion was calculated from the relative integrals of H_c and H_a. Conversion = 29 %, $M_{n,SEC}$ = 2000 g.mol^{-1}, $Đ_m$ = 1.60.

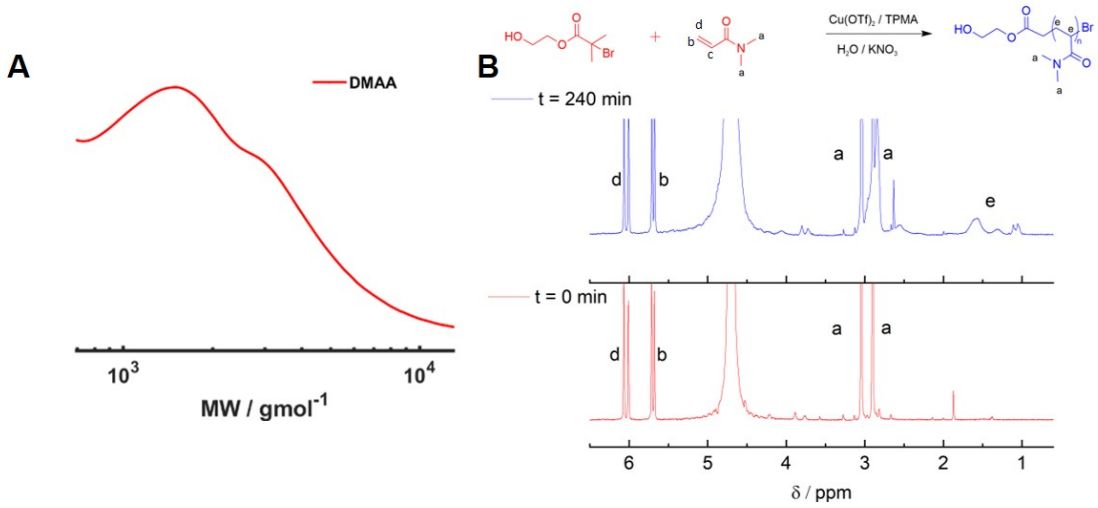

Fig S13. NMR and SEC of PDMAm from the current controlled seATRP of DMAm using [DMAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.3] : [0.8] : [0.1] at 0°C. Conversion was calculated from the relative integrals of $H_{b,}$ and H_a. Conversion = 49 %, $M_{n,SEC}$ = 1500 g.mol^{-1}, $Đ_m$ = 2.02.

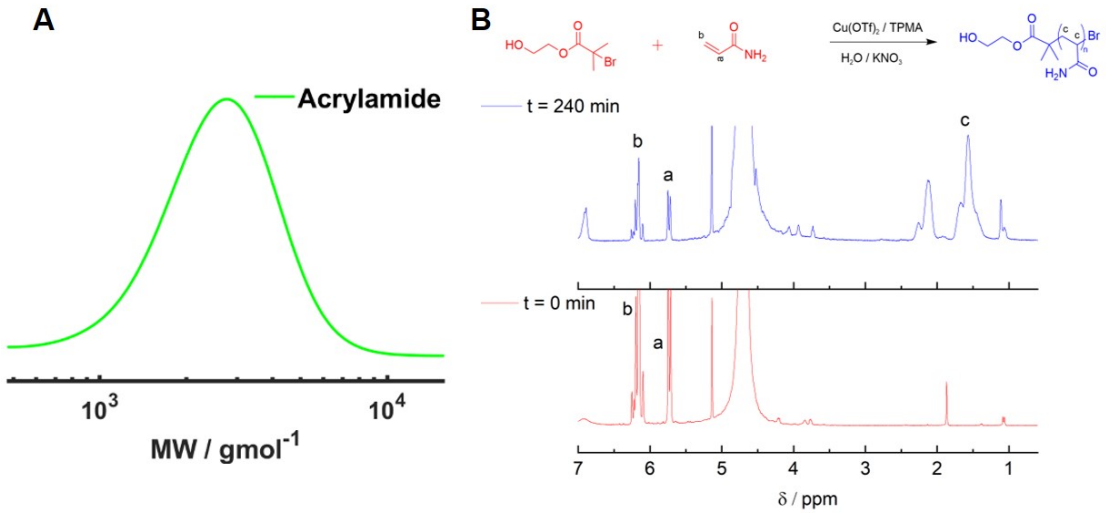

Fig S14. NMR and SEC of PAAm from the current controlled seATRP of AAm using [AAm] : [HEBiB] : [Cu^{II}] : [TPMA] : [NaBr] = [40] : [1] : [0.3] : [0.8] : [0.1] at 0°C. Conversion was calculated from the relative integrals of $H_{a,b}$ and H_c. Conversion = 83 %, $M_{n,SEC}$ = 2200 g.mol^{-1}, $Đ_m$ = 1.27.