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Correction: Tacticity, molecular weight, and temporal control by lanthanide triflate-catalyzed stereoselective radical polymerization of acrylamides with an organotellurium chain transfer agent

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rsc.li/polymersCorrection for 'Tacticity, molecular weight, and temporal control by lanthanide triflate-catalyzed stereoselective radical polymerization of acrylamides with an organotellurium chain transfer agent' by Yuji Imamura *et al.*, *Polym. Chem.*, 2020, DOI: 10.1039/d0py01280g.

After the paper was accepted, the authors recognized that some experimental procedures reported in the experimental section were erroneously described; while several reagents were added as a diluted solution, it was shown as if they were added as a neat form. The following are valid experimental procedures, and the corrected parts are highlighted as bold. The corrections do not affect at all on the experimental results as well as the conclusions of the paper.

General procedure of polymerization of DMAA in the presence of Lewis acid and the chain extension

A solution of **5** (50 μL , 0.25 mol L⁻¹ in methanol 0.013 mmol), DMAA (128 μL , 1.3 mmol), a solution of AIBN (40 μL , 0.078 mol L⁻¹ in methanol, 0.0031 mmol), α,α,α -trifluorotoluene (15 μL , 0.13 mmol, an internal standard), Y(OTf)₃ (134 mg, 0.25 mmol) in methanol (0.80 mL) in a dry Pyrex tube was heated at 60 °C under nitrogen atmosphere. An aliquot was withdrawn periodically and was dissolved in DMSO-*d*₆, and the monomer conversion was determined by ¹H NMR spectroscopy. After 70 min heating, the monomer conversion reached to 88%. The SEC analysis determined M_n (= 14 300) and D (= 1.20). The ¹H NMR analysis in DMSO-*d*₆ at 130 °C determined the diad selectivity (85% *m*). To the remaining polymerization mixture, DMAA (639 μL , 6.25 mmol) and a solution of AIBN (40 μL , 0.078 mol L⁻¹ in methanol, 0.0031 mmol) were added and the mixture was heated for further 4.5 h. The monomer conversion reached to 87% by the ¹H NMR analysis. Benzenethiol (1.6 μL , 0.016 mmol) was added and the resulting mixture was photoirradiated by a white LED (9.4 W) light for 1 h at room temperature.³⁹ The crude mixture was diluted with 1 mL of MeOH and the solution was poured into vigorously stirred diethylether (450 mL). The precipitated product was collected by suction filtration (0.60 g) and was dried under reduced pressure at 100 °C for overnight. The SEC analysis determined M_n (= 54 400) and D (= 1.52). The ¹H NMR analysis determined the diad selectivity (78% *m*). See Table S1 for the detailed data on the chain extension experiments.

General procedure of polymerization of DEAA in the presence of Lewis acid and the chain extension

A solution of **5** (4.4 μL , 0.025 mmol), DEAA (342 μL , 2.5 mmol), a solution of AIBN (30 μL , 0.20 mol L⁻¹ in methanol, 0.0061 mmol), α,α,α -trifluorotoluene (31 μL , 0.25 mmol, an internal standard), Y(OTf)₃ (268 mg, 0.50 mmol) in methanol (1.6 mL) in a dry Pyrex tube was heated at 60 °C under nitrogen atmosphere. An aliquot was withdrawn periodically and was dissolved in DMSO-*d*₆, and the monomer conversion was determined by ¹H NMR spectroscopy. After 70 min heating, the monomer conversion reached to 97%. The SEC analysis determined M_n (= 13 800) and D (= 1.23). The ¹H NMR analysis in DMSO-*d*₆ at 130 °C determined the diad selectivity (93% *m*). To the remaining polymerization mixture, DEAA (1.71 mL, 12.5 mmol) and a solution of AIBN (30 μL , 0.20 mol L⁻¹ in methanol, 0.0061 mmol) were added and the mixture was heated for further 2.5 h. The monomer conversion reached to 94% by the ¹H NMR analysis. Benzenethiol (3.1 μL , 0.030 mmol) and 1.8 mL of MeOH were

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added and the resulting mixture was photoirradiated by a white LED (9.4 W) light for 3 h at room temperature. The crude mixture was diluted with 3 mL of MeOH and the part of the solution was poured into vigorously stirred diethylether (200 mL). The precipitated product was collected by suction filtration and was dried under reduced pressure at 100 °C for overnight (0.61 g). The SEC analysis determined M_n (= 93 000) and D (= 1.09). The ^1H NMR analysis determined the diad selectivity (87% m). See Table S1 for the detailed data on the chain extension experiments.

Polymerization of DEAA under photo irradiation in the presence of Lewis acid

A solution of 5 (30 μL , 0.13 mol L^{-1} in methanol, 0.0038 mmol), DEAA (52 μL , 0.38 mmol), α,α,α -trifluorotoluene (4.7 μL , 0.038 mmol), $\text{Y}(\text{OTf})_3$ (40 mg, 0.075 mmol) in methanol (0.24 mL) in a dry Pyrex tube was photoirradiated by a white LED (9.4 W) light at 0 °C. The monomer conversion reached 85% after 2 h. Benzenethiol (0.47 μL , 0.0046 mmol) was added and photoirradiated by a white LED (9.4 W) light for 1 h at room temperature. The crude mixture was diluted with 0.5 mL of MeOH and the solution was poured into vigorously stirred diethylether (100 mL). The product collected by suction filtration was dried under reduced pressure at 100 °C for overnight. The SEC analysis determined M_n (= 12 000) and D (= 1.29). The ^1H NMR analysis determined the diad selectivity (93% m).

General procedure of polymerization of NIPAM in the presence of Lewis acid and the chain extension

A solution of 5 (100 μL , 0.13 mol L^{-1} in methanol, 0.013 mmol), NIPAM (141 mg, 1.3 mmol), a solution of AIBN (40 μL , 0.078 mol L^{-1} in methanol, 0.0031 mmol), α,α,α -trifluorotoluene (15 μL , 0.13 mmol, an internal standard), $\text{Y}(\text{OTf})_3$ (134 mg, 0.25 mmol) in methanol (0.80 mL) in a dry Pyrex tube was heated at 60 °C under nitrogen atmosphere. An aliquot was withdrawn periodically and was dissolved in $\text{DMSO-}d_6$, and the monomer conversion was determined by ^1H NMR spectroscopy. After 1.5 h heating, the monomer conversion reached to 86%. The SEC analysis determined M_n (= 13 000) and D (= 1.17). The ^1H NMR analysis in $\text{DMSO-}d_6$ at 130 °C determined the diad selectivity (86% m). To the remaining polymerization mixture, NIPAM (1.41 g, 12.5 mmol), a solution of AIBN (40 μL , 0.078 mol L^{-1} in methanol, 0.0031 mmol) and MeOH (1.0 mL) were added and the mixture was heated for further 5 h. The monomer conversion reached to 82% by the ^1H NMR analysis. Benzenethiol (1.5 μL , 0.016 mmol) was added and the resulting mixture was photoirradiated by a white LED (9.4 W) light for 1 h at room temperature. The crude mixture was diluted with 1 mL of MeOH and the part of the solution was poured into vigorously stirred diethylether (200 mL). The precipitated product was collected by suction filtration and was dried under reduced pressure at 100 °C for overnight (0.18 g). The SEC analysis determined M_n (= 56 800) and D (= 1.88). The ^1H NMR analysis determined the diad selectivity (71% m). See Table S1 for the detailed data on the chain extension experiments.

General procedure of polymerization of AM in the presence of Lewis acid

A solution of 5 (50 μL , 0.25 mol L^{-1} in methanol, 0.0125 mmol), AM (89 mg, 1.25 mmol), a solution of AIBN (40 μL , 0.078 mol L^{-1} in methanol, 0.0031 mmol), α,α,α -trifluorotoluene (15 μL , 0.125 mmol, an internal standard), $\text{Y}(\text{OTf})_3$ (134 mg, 0.25 mmol) in methanol (1.25 mL) in a dry Pyrex tube was heated at 60 °C under nitrogen atmosphere. An aliquot was withdrawn periodically and was dissolved in $\text{DMSO-}d_6$, and the monomer conversion was determined by ^1H NMR spectroscopy. After 2 h heating, the monomer conversion reached to 87%. Benzenethiol (1.5 μL , 0.015 mmol) was added and the resulting mixture was photoirradiated by a white LED (9.4 W) light for 1.5 h at room temperature. The SEC analysis determined M_n (= 4900) and D (= 1.10). The ^1H NMR analysis in $\text{DMSO-}d_6$ at 145 °C determined the diad selectivity (70% m). The polymer was purified by dialysis in water. The polymer was dried by evaporator (57 mg).

Synthesis of stereoblock PDEAA

A solution of 5 (4.4 μL , 0.025 mmol), DEAA (342 μL , 2.5 mmol), a solution of AIBN (80 μL , 0.078 mol L^{-1} in methanol, 0.0063 mmol) and α,α,α -trifluorotoluene (61 μL , 0.50 mmol) in methanol (1.6 mL) in a dry Pyrex tube was heated at 60 °C under nitrogen atmosphere. An aliquot was withdrawn periodically and was dissolved in $\text{DMSO-}d_6$, and the monomer conversion was determined by ^1H NMR spectroscopy. After 2 h heating, the monomer conversion reached to 47%. The SEC analysis determined M_n (= 3500) and D (= 1.08). The ^1H NMR analysis in $\text{DMSO-}d_6$ at 130 °C determined the diad selectivity (55% m). A slightly cloudy solution of $\text{Y}(\text{OTf})_3$ (134 mg, 0.25 mmol) in methanol (1.1 mL) was added and the resulting homogeneous solution was heated for 1 h. Overall monomer conversion reached 85%. Benzenethiol (3.1 μL , 0.030 mmol) was added and the resulting solution was photoirradiated by a white LED (9.4 W) light for 1 h at room temperature. The crude mixture was diluted with 1 mL of MeOH and the solution was poured into vigorously stirred diethylether (150 mL). The precipitated product was collected by suction filtration and was dried under reduced pressure at 100 °C for overnight (0.16 g). The SEC analysis determined M_n (= 12 100) and D (= 1.16). The ^1H NMR analysis determined the diad selectivity (71% m). The m diad selectivity of the second block was determined to be 91% m from the diad selectivity of the first block and the block copolymer together with the molar fraction of each block.



Typical experiment for the synthesis of low molecular weight PDEAA for TIMS-MS measurement

A solution of **5** (14 μl , 0.080 mmol), DEAA (164 μl , 1.2 mmol), a **solution of AIBN (48 μL , 0.42 mol L⁻¹ in methanol, 0.020 mmol)**, Y(OTf)₃ (19.2 mg, 0.036 mmol) and α,α,α -trifluorotoluene (29 μl , 0.24 mmol) in methanol (0.77 mL) in a dry Pyrex tube under nitrogen atmosphere was heated at 60 °C for 2 h. The monomer conversion reached to 99% by the ¹H NMR analysis. Benzenethiol (9.8 μl , 0.096 mmol) was added and the resulting solution was photoirradiated by a white LED (9.4 W) light for 1 h at room temperature. The crude mixture was diluted with 1 mL of MeOH and the solution was poured into vigorously stirred diethylether (150 mL). The product collected by suction filtration was dried under reduced pressure at 100 °C for overnight (0.11 g). The SEC analysis determined M_n (= 2500) and D (= 1.15), and the ¹H NMR analysis in DMSO-*d*₆ at 130 °C determined the diad selectivity (85% *m*).

The Royal Society of Chemistry apologises for these errors and any consequent inconvenience to authors and readers.

