Supporting information

Click Chemistry Step Growth Polymerization of Novel α-Azide-ω-Alkyne monomers

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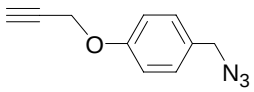
General

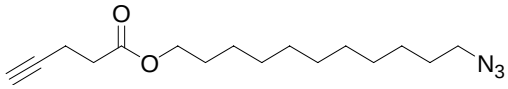
$Cu(PPh_3)_3Br$,[1] 11-azido-1-undecanol,[2] (**1**) and 1-(bromomethyl)-4-(2-propyn-1-yloxy)-benzene[3] were synthesized adapting synthetic procedures described previously. All other reactants were purchased from Aldrich and used as received. ^1H NMR spectra were recorded on a Bruker Advance II Spectrometer (250 MHz) in $CDCl_3$ or $(CD_3)_2SO$ at room temperature. ^{13}C NMR spectra (100 MHz) were performed on a Bruker DRX400 Spectrometer in $CDCl_3$. TMS was used as internal reference. SEC experiments were performed in $CHCl_3$ at 22 °C and a flow rate of 0.5 mL/min using a system equipped with a Waters 410 differential refractometer, PL Gel mixed C column (internal diameter 7.8 mm, length 30 cm). Number average molecular weights (M_n) were calculated using calibration curves obtained from polystyrene standards. Differential scanning calorimetry (DSC) was measured on a DSC Q100 (TA Instruments) at a heating rate of 10 °C/min under nitrogen atmosphere. The thermal gravimetric analysis measurements were performed with a TA Instruments TGA Q500 at a heating rate of 20 °C/min under nitrogen purge. ESI-TOF Mass Spectrometry experiments were carried out with a Micromass mass spectrometer (Waters) in positive mode. Maldi-TOF analyses were performed on an Applied Biosystems Voyager-DE STR equipment (nitrogen laser, 337 nm, accelerating potential of 25 kV) using dithranol as matrix. The samples were dissolved in $CHCl_3$ and MeOH. The mixture was directly infused in the source. FTIR spectra were taken on a Perkin-Elmer FT-IR spectrometer.

Synthesis of 4-(2-propynyloxy)-benzyl azide, 2.



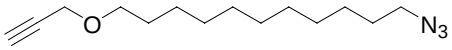
To sodium azide (2.87 g, 66.7 mmol) was added 1-(bromomethyl)-4-(2-propyn-1-yloxy)-benzene (5.00 g, 22.2 mmol) in a round-bottom flask containing 150 mL of DMF. The mixture was stirred in the dark for 20 hours at 45 °C. After filtration and addition of water, the product was extracted with dichloromethane, giving after evaporation of the solvents a white solid (3.97 g, 95.7%). ^{1}H NMR ($CDCl_3$) δ (ppm): 7.25 (m, *m*-Ar, 2H), 6.99 (m, *o*-Ar, 2H), 4.70 (d, OC**H**$_2$C≡CH, J = 3.8 Hz, 2H), 4.28 (s, C**H**$_2$N$_3$, 2H), 2.52 (t, OCH$_2$C≡C**H**, J = 3.8 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ (ppm): 157.60 (**C**OCH$_2$), 129.69 (*m*-Ar), 128.47 (*p*-Ar), 115.25 (*o*-Ar), 78.39 (CH$_2$**C**≡CH), 75.68 (CH$_2$C≡**C**H), 55.88 (**C**H$_2$C≡CH), 54.35 (**C**H$_2$N$_3$).

Synthesis of 3-butynoic acid 11-azido undecyl-ester, 3.



A solution of DCC (9.02 g, 47.8 mmol) in CH_2Cl_2 (20 mL) was added dropwise to a stirred solution of 11-azido-undecanol **2** (3.73 g, 17.5 mmol), 4-pentynoic acid (1.89 g, 19.3 mmol), DMAP (0.43 g, 3.50 mmol) and DPTS (0.51 g, 1.75 mmol) in CH_2Cl_2 (70 mL). The reaction mixture was stirred at room temperature for 24 hours, before being filtered and evaporated to dryness. The crude product was then purified by column chromatography eluting with a 98/2 mixture of petroleum ether and ethyl acetate, giving after evaporation of the solvents a slightly yellow liquid (4.85 g, 85.9%). ^{1}H NMR ($CDCl_3$) δ (ppm): 4.12 (t, C**H**$_2$O, J = 6.7 Hz, 2H), 3.25 (t, C**H**$_2$N$_3$, J = 6.9 Hz, 2H), 2.67-2.42 (m, O(C**H**$_2$)$_2$C≡CH, 4H), 1.97 (t, C≡C**H**, J = 2.4 Hz, 1H), 1.73-1.51 (m, C**H**$_2$CH$_2$O, C**H**$_2$CH$_2$N$_3$, 4H), 1.49-1.17 (m, CH$_2$CH$_2$(C**H**$_2$)$_7$CH$_2$CH$_2$, 14H). ^{13}C NMR ($CDCl_3$) δ (ppm): 171.82 (**C**OO), 82.56 (**C**≡CH), 68.97 (C≡**C**H), 64.89 (**C**H$_2$O), 51.51 (**C**H$_2$N$_3$), 33.44 (**C**H$_2$CH$_2$C≡CH), 29.45, 29.22, 29.15, 28.87, 28.63, 26.74, 25.90 (CH$_2$(**C**H$_2$)$_9$CH$_2$), 14.43 (**C**H$_2$C≡CH).

Synthesis of 1-azido-11-prop-2-ynyloxy-undecane, 4.

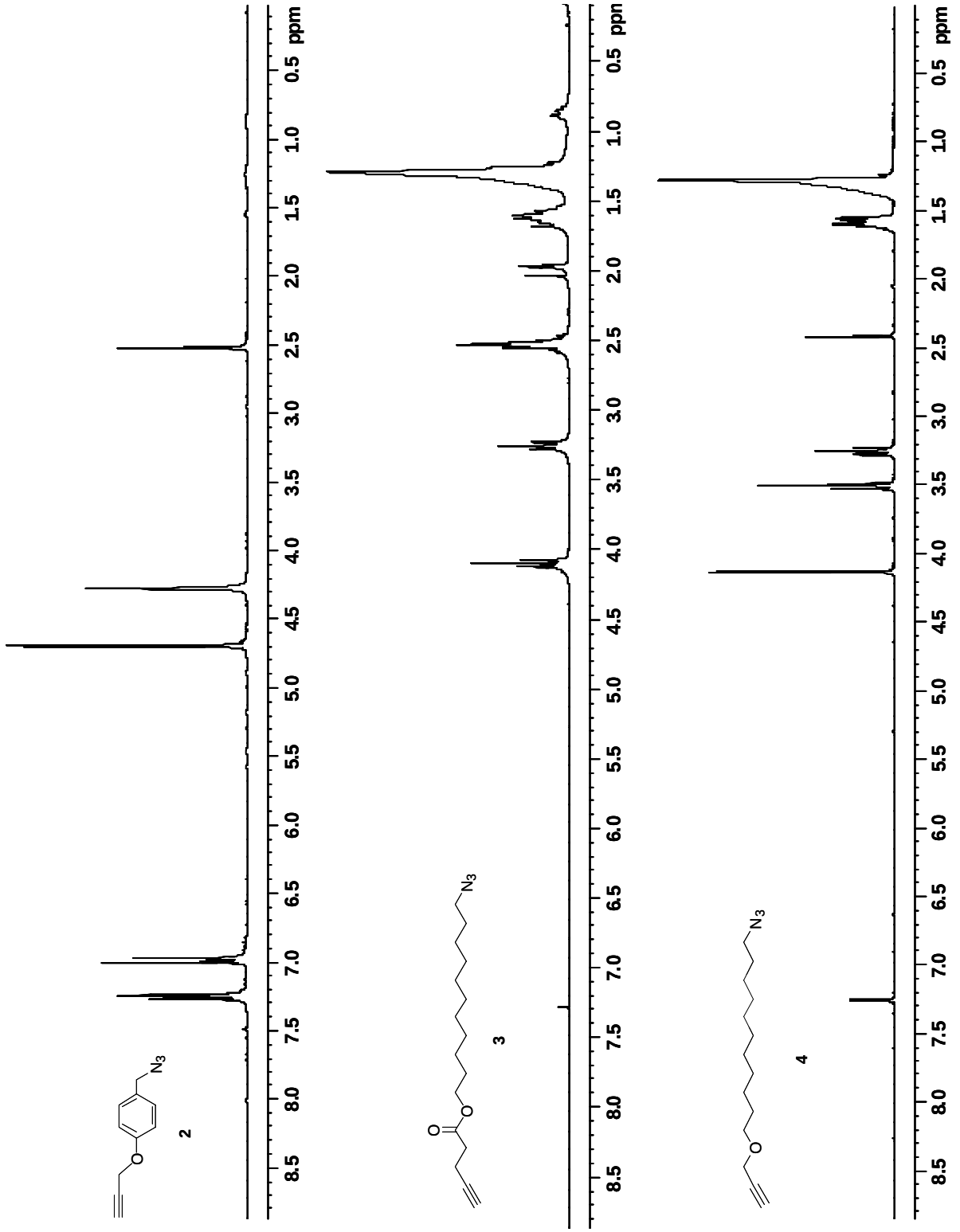


Sodium hydride (4.7 g, 117 mmol) was added in small portions to a stirred solution of 11-azido-1-undecanol **2** (10.0 g, 46.9 mmol) and 18-crown-6 (20 mg, 0.08 mmol) in dry THF (200 mL) maintained at 0 °C under argon. After hydrogen was entirely emitted, a solution of propargyl bromide (10.5 mL, 93.9 mmol) in dry THF (50 mL) was added dropwise. The mixture was stirred overnight at room temperature and after neutralization of residual NaH by distilled water (20 mL), the mixture was extracted with CH_2Cl_2 (2×100 mL). After drying the organic layer with $MgSO_4$ and evaporation of the solvents, the crude product was purified by column chromatography eluting with a 98/2 mixture of petroleum ether and ethyl acetate giving after evaporation of the solvents a slightly yellow liquid (9.87 g, 83.2 %). ^{1}H NMR ($CDCl_3$) δ (ppm): 4.12 (d, OC**H**$_2$C≡CH, J = 2.4 Hz, 2H), 3.51 (t, C**H**$_2$OCH$_2$C≡CH, J = 6.6 Hz, 2H), 3.25 (t, C**H**$_2$N$_3$, J = 6.9 Hz 2H), 2.41 (t, C≡C**H**, J = 2.4 Hz, 1H), 1.62-1.53 (m, C**H**$_2$CH$_2$O, C**H**$_2$CH$_2$N$_3$, 4H), 1.38-1.16 (m, CH$_2$CH$_2$(C**H**$_2$)$_7$CH$_2$CH$_2$, 14H). ^{13}C NMR ($CDCl_3$) δ (ppm): 80.11 (**C**≡CH), 74.00 (C≡**C**H), 70.31 (**C**H$_2$OCH$_2$C≡CH), 58.02 (**C**H$_2$C≡CH), 51.52 (**C**H$_2$N$_3$), 29.52, 29.46, 29.42, 29.15, 28.86, 26.73, 26.10 (CH$_2$(**C**H$_2$)$_9$CH$_2$).

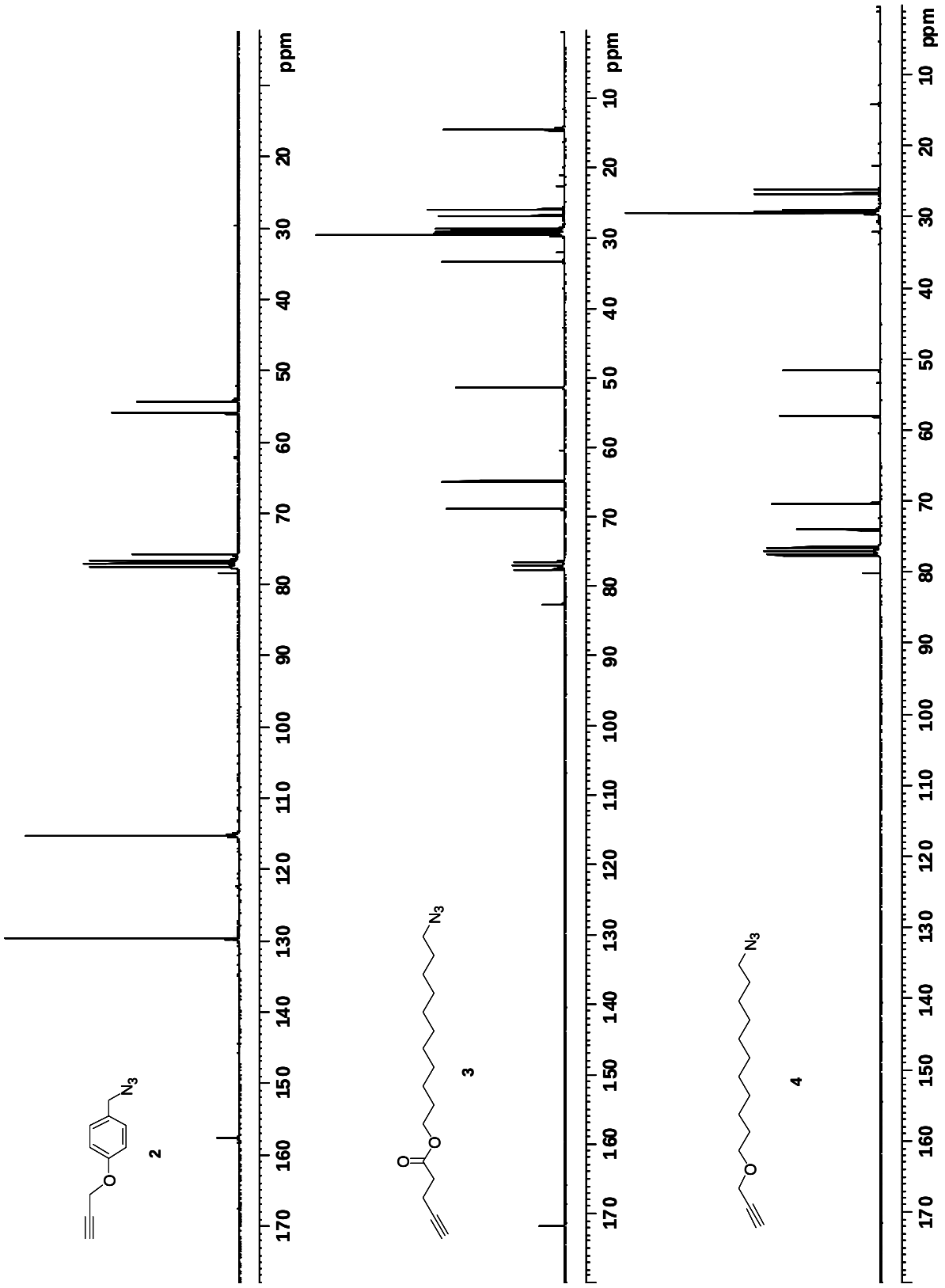
Mass Spectroscopy of monomers 2-4.

MS ESI-TOF analyses were performed on monomers **2-4**. However, oligomerization occurred during the experiments giving mainly di- and tri-adducts instead of the expected monomers. The same behaviour was observed during Maldi-TOF analyses. Therefore, we conclude that mass spectroscopy is not a relevant method for the characterization of heterofunctional α-azide-ω-alkyne monomers. To complete the characterization of monomers **2-4**, ^{1}H and ^{13}C NMR spectra are included herein.

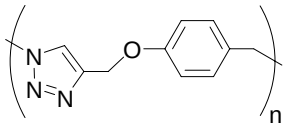
^{1}H NMR spectra of monomers 2-4



^{13}C NMR spectra of monomers 2-4

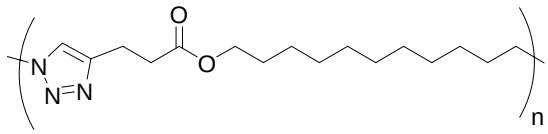


General procedure for click chemistry step growth homopolymerization, synthesis of polytriazole 5.



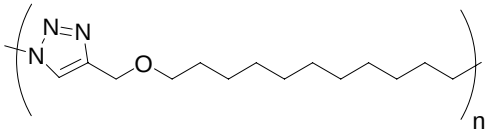
A solution of α-azide-ω-alkyne **2** (936 mg, 5.0 mmol), $Cu(PPh_3)_3Br$ (46 mg, 0.05 mmol) and DIPEA (1.94 g, 15.0 mol) in $CHCl_3$ (25 mL) was stirred overnight at 45 °C in the dark. After concentration under reduced pressure, the resulting brown solid was dissolved in DMSO and precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **5** as a white powder (745 mg, 79.6%). ^{1}H NMR ($(CD_3)_2SO$): δ (ppm) 7.97 (s, OCH_2C=C**H**, 1H), 7.07 (bd, *o*-Ar, 2H), 6.78 (bd, *m*-Ar, 2H), 5.46 (bs, Ph**CH**$_2$N, 2H), 5.14 (bs, PhO**CH**$_2$C=CH, 2H). ^{13}C NMR ($(CD_3)_2SO$) δ (ppm): 157.90 (**C**OCH_2), 142.81 (OCH_2**C**=CH), 129.46 (*m*-Ar), 128.62 (*p*-Ar), 124.30 (OCH_2C=**C**H), 114.87 (*o*-Ar), 61.11 (O**C**H_2C=CH), 52.33 (Ph**C**H_2N).

Synthesis of polytriazole 6.



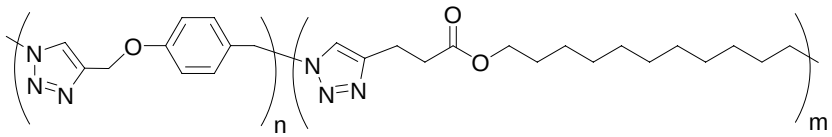
The same procedure as above was applied to **3** (1.47 g, 5 mmol). After concentration under reduced pressure, the polymerization mixture was precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **6** as a white powder (1.21 g, 82.3%). ^{1}H NMR ($CDCl_3$): δ (ppm) 7.36 (s, OCH_2C=C**H**, 1H), 4.29 (bt, NC**H**$_2CH_2CH_2$, 2H), 4.06 (bt, C**H**$_2$OCO, 2H), 3.03 (bt, C**H**$_2CH_2C$=CH, 2H), 2.72 (bt, C**H**$_2CH_2C$=CH, 2H), 1.87 (bs, C**H**$_2CH_2$N, 2H), 1.63 (bs, C**H**$_2CH_2$OCO, 2H), 1.26 (bs, NCH_2CH_2(C**H**$_2$)$_7$, 14H). ^{13}C NMR ($CDCl_3$) δ (ppm): 172.91 (**C**OO), 146.4 (CH_2**C**=CH), 121.10 (CH_2C=**C**H), 64.71 (CH_2**C**H_2OCO), 50.25 (CH_2**C**H_2N), 33.94 (O**C**H_2CH_2C=CH), 30.91, 30.31, 30.21, 29.70, 29.63, 28.90, 27.81, 26.51, 26.41 (CH_2(**C**H_2)$_9CH_2$), 21.05 (OCH_2**C**H_2C=CH).

Synthesis of polytriazole 7.



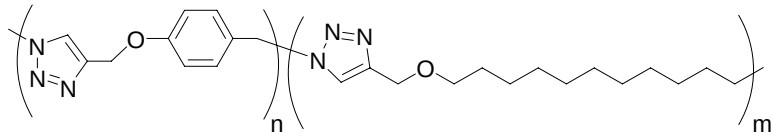
The same procedure as above was applied to **4** (1.26 g, 5 mmol). After concentration under reduced pressure, the polymerization mixture was precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **7** as a white powder (1.09 g, 86.3%). ^{1}H NMR ($CDCl_3$): δ (ppm) 7.52 (s, OCH_2C=C**H**, 1H), 4.62 (s, O**C**$\mathbf{H_2}$C=CH, 2H), 4.33 (bt, N**C**$\mathbf{H_2}$$CH_2CH_2$, 2H), 3.51 (bt, C**H**2$OCH_2C$=CH, 2H), 1.89 (bs, C$\mathbf{H_2}$$CH_2N$, 2H), 1.59 (bs, C$\mathbf{H_2}$$CH_2O$, 2H), 1.26 (bs, $NCH_2CH_2(C\mathbf{H_2})_7$, 14H). ^{13}C NMR ($CDCl_3$) δ (ppm): 145.42 ($CH_2$**C**=CH), 122.13 ($CH_2C$=**C**H), 70.88 (O**C**$H_2C$=CH), 64.41 (**C**$H_2OCH_2C$=CH), 50.36 ($CH_2$**C**$H_2N$), 30.31, 30.13, 29.66, 29.49, 29.36, 29.18, 26.36, 26.11, 24.75 ($CH_2(\mathbf{C}H_2)_9CH_2$).

General procedure for click chemistry step growth copolymerization, synthesis of polytriazole 8.



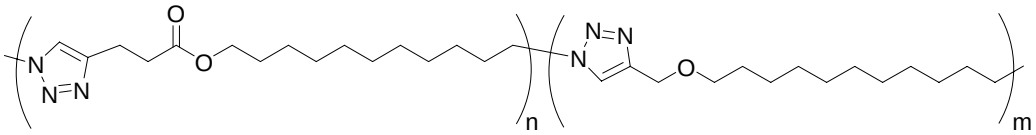
A solution of **2** (468 mg, 2.5 mmol), **3** (732 mg, 2.5 mmol), $Cu(PPh_3)_3Br$ (46 mg, 0.05 mmol) and DIPEA (1.94 g, 15.0 mol) in $CHCl_3$ (25 mL) was stirred overnight at 45 °C in the dark. After concentration under reduced pressure, the resulting brown solid was dissolved in DMSO and precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **8** as a white powder (999 mg, 83.3%). ^{1}H NMR ($(CD_3)_2SO$): δ (ppm) 8.35-7.50 (2bs, OCH_2C=C**H**, 2H), 7.26 (bt, *o*-Ar, 2H), 7.06 (bs, *m*-Ar, 2H), 5.54 (2bs, PhC$\mathbf{H_2}$N, 2H), 5.13 (bs, PhOC$\mathbf{H_2}$C=CH, 2H), 4.29 (bt, N**C**$\mathbf{H_2}$$CH_2CH_2$, 2H), 4.06 (bt, C$\mathbf{H_2}$OCO, 2H), 2.89 (bt, $OCH_2C\mathbf{H_2}C$=CH, 2H), 2.66 (bt, OC$\mathbf{H_2}$$CH_2C$=CH, 2H), 1.78 (bs, $NCH_2C\mathbf{H_2}CH_2$, 2H), 1.52 (bs, C$\mathbf{H_2}$$CH_2O$, 2H), 1.24 (bs, $NCH_2CH_2(C\mathbf{H_2})_7$, 14H). ^{13}C NMR ($(CD_3)_2SO$) δ (ppm): 172.02 ($CH_2$O**C**O), 157.90 (**C**$OCH_2$), 142.81 (PhO$CH_2$**C**=CH), 142.42 ($CH_2CH_2$**C**=CH), 129.46 (*m*-Ar), 128.62 (*p*-Ar), 124.30 (PhOCH_2C=**C**H), 121.79 (CH_2CH_2C=**C**H), 114.87 (*o*-Ar), 63.99 (**C**H_2OCO) 61.11 (PhO**C**H_2C=CH), 52.33 (Ph**C**H_2N), 49.32 (CH_2**C**H_2N), 33.07 (O**C**H_2CH_2C=CH), 29.66, 29.63, 28.81, 28.58, 28.32, 28.06, 28.29, 26.92, 26.16 ($CH_2(\mathbf{C}H_2)_9CH_2$), 20.66 (OCH_2**C**H_2C=CH).

Synthesis of polytriazole 9.



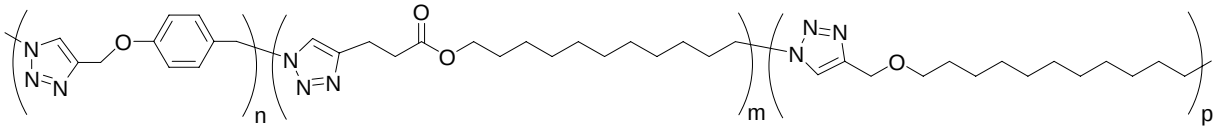
The same procedure as above was applied to a solution of **2** (468 mg, 2.5 mmol) and **4** (628 mg, 2.5 mmol). After concentration under reduced pressure, the resulting brown solid was dissolved in DMSO and precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **9** as a white powder (938 mg, 85.3%). ^{1}H NMR ($(CD_3)_2SO$): δ (ppm) 8.22-8.06 (2bs, $OCH_2C{=}C\mathbf{H}$, 2H), 7.26 (bt, *o*-Ar, 2H), 7.27 (bs, *o*-Ar, 2H), 7.01 (bs, *m*-Ar, 2H), 5.51 (2bs, $PhC\mathbf{H}_2N$, 2H), 5.10 (bs, $PhOC\mathbf{H}_2C{=}CH$, 2H), 4.44 (2s, $CH_2CH_2OC\mathbf{H}_2C{=}CH$, 2H), 4.32 (bs, $NC\mathbf{H}_2CH_2CH_2$, 2H), 3.41 ($C\mathbf{H}_2OCH_2C{=}CH$, 2H), 1.77 (bs, $NCH_2C\mathbf{H}_2CH_2$, 2H), 1.45 (bs, $C\mathbf{H}_2CH_2OCH_2C{=}CH$, 2H), 1.20 (bs, $NCH_2CH_2(C\mathbf{H}_2)_7$, 14H). ^{13}C NMR ($(CD_3)_2SO$) δ (ppm): 157.90 ($\mathbf{C}OCH_2$), 142.81 ($PhOCH_2\mathbf{C}{=}CH$), 142.62 ($CH_2OCH_2\mathbf{C}{=}CH$), 129.46 (*m*-Ar), 128.62 (*p*-Ar), 124.30 ($PhOCH_2C{=}\mathbf{C}H$), 123.70 ($CH_2OCH_2C{=}\mathbf{C}H$), 114.87 (*o*-Ar), 69.53 ($O\mathbf{C}H_2C{=}CH$), 63.26 ($\mathbf{C}H_2OCH_2C{=}CH$), 61.11 ($PhO\mathbf{C}H_2C{=}CH$), 52.33 ($Ph\mathbf{C}H_2N$), 49.32 ($CH_2\mathbf{C}H_2N$), 29.80, 29.62, 29.05, 28.89, 28.78, 28.76, 28.29, 25.76, 25.58 ($CH_2(\mathbf{C}H_2)_9CH_2$).

Synthesis of polytriazole 10.



The same procedure as above was applied to a solution of **3** (732 mg, 2.5 mmol) and **4** (628 mg, 2.5 mmol). After concentration under reduced pressure, the polymerization mixture was precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **9** as a white powder (1.14 g, 83.6%). ^{1}H NMR ($CDCl_3$): δ (ppm) 7.46-7.32 (2bs, $OCH_2C{=}C\mathbf{H}$, 2H), 4.62 (s, $OC\mathbf{H}_2C{=}CH$, 2H), 4.33 (bt, $NC\mathbf{H}_2CH_2CH_2$, 2H), 4.29 (bt, $NC\mathbf{H}_2CH_2CH_2$, 2H), 4.06 (bt, $C\mathbf{H}_2OCO$, 2H), 3.51 (bt, $C\mathbf{H}_2OCH2C{=}CH$, 2H), 3.03 (bt, $C\mathbf{H}_2CH_2C{=}CH$, 2H), 2.72 (bt, $C\mathbf{H}_2CH_2C{=}CH$, 2H), 1.89 (bs, $C\mathbf{H}_2CH_2N$, 2H), 1.87 (bs, $C\mathbf{H}_2CH_2N$, 2H), 1.63 (bs, $C\mathbf{H}_2CH_2OCO$, 2H), 1.59 (m, $C\mathbf{H}_2CH_2O$, 2H), 1.26 (m, $NCH_2CH_2(C\mathbf{H}_2)_7$, 28H). ^{13}C NMR ($CDCl_3$) δ (ppm): 172.91 ($\mathbf{C}OO$), 146.4 ($CH_2CH_2\mathbf{C}{=}CH$), 145.4 ($OCH_2\mathbf{C}{=}CH$), 122.13 ($OCH_2C{=}\mathbf{C}H$), 121.10 ($CH_2CH_2C{=}\mathbf{C}H$), 70.88 ($O\mathbf{C}H_2C{=}CH$), 64.71 ($CH_2\mathbf{C}H_2OCO$), 64.41 ($\mathbf{C}H_2OCH_2C{=}CH$), 50.36 ($CH_2\mathbf{C}H_2N$), 50.25 ($CH_2\mathbf{C}H_2N$), 33.94 ($O\mathbf{C}H_2CH_2C{=}CH$), 30.91-24.75 (18s, $CH_2(\mathbf{C}H_2)_9CH_2$), 21.05 ($OCH_2\mathbf{C}H_2C{=}CH$).

Synthesis of polytriazole 11.



The same procedure as above was applied to a solution of α-azide-ω-alkyne **2** (318 mg, 1.7 mmol), **3** (498 mg, 1.7 mmol), and **4** (427 mg, 1.7 mmol). After concentration under reduced pressure, the polymerization mixture was precipitated twice in diethyl ether, yielding after drying under reduced pressure polytriazole **9** as a white powder (1.14 g, 83.6%). ^{1}H NMR ($(CD_3)_2SO$): δ (ppm) 8.20-7.67 (3bs, OCH_2C=C**H**, 3H), 7.26 bd, *o*-Ar, 2H), 7.03 (bd, *m*-Ar, 2H), 5.51 (bs, Ph**C**$\mathbf{H}_2$N, 2H), 5.12 (bs, PhO**C**$\mathbf{H}_2$C=CH, 2H), 4.44 (2s, CH_2CH_2O**C**$\mathbf{H}_2$C=CH, 2H), 4.33 (m, N**C**$\mathbf{H}_2CH_2CH_2$, 4H), 3.97 (bt, **C**$\mathbf{H}_2$OCO, 2H), 3.41 (**C**$\mathbf{H}_2OCH_2$C=CH, 2H), 2.89 (bt, OCH_2**C**$\mathbf{H}_2$C=CH, 2H), 2.66 (bt, O**C**$\mathbf{H}_2CH_2$C=CH, 2H), 1.78 (m, NCH_2**C**$\mathbf{H}_2CH_2$, 4H), 1.52 (m, **C**$\mathbf{H}_2CH_2O$, 4H), 1.24 (m, NCH_2CH_2(C**H**$_2$)$_7$, 14H). ^{13}C NMR ($(CD_3)_2SO$) δ (ppm): 172.02 (CH_2O**C**O), 157.90 (**C**OCH_2), 142.81 (PhOCH_2**C**=CH), 142.62 (CH_2OCH_2**C**=CH), 142.42 (CH_2CH_2**C**=CH), 129.46 (*m*-Ar), 128.62 (*p*-Ar), 124.30 (PhOCH_2C=**C**H), 123.70 (CH_2OCH_2C=**C**H), 121.79 (CH_2CH_2C=**C**H), 114.87 (*o*-Ar), 63.99 (**C**H_2OCO), 69.53 (O**C**H_2C=CH), 63.26 (**C**H_2OCH_2C=CH), 61.11 (PhO**C**H_2C=CH), 52.33 (Ph**C**H_2N), 49.32 (CH_2**C**H_2N), 33.07 (O**C**H_2CH_2C=CH), 29.80-25.58 (18s, CH_2(**C**H_2)$_9CH_2$), 20.66 (OCH_2**C**H_2C=CH).

Influence of monomer concentration on the self-cyclization of monomer 4.

Synthesis of polytriazole **7** in $CHCl_3$ was reproduced using increasing monomer concentrations (0.1-50 wt% of monomer). ^1H NMR analyses of the crude mixtures were used to follow the percentage of monomer **4** self-cyclization among the polymerization mixture (Figure A). It appears that the amount of monocyclic species is directly related to the dilution.

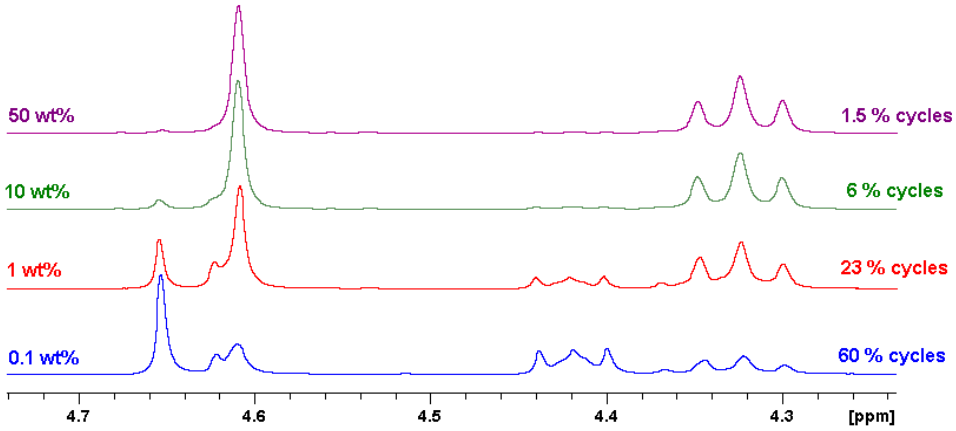


Figure A. ^1H NMR of crude polytriazole **7** for increasing concentrations of monomer 4 in $CHCl_3$.

The crude samples above were then characterized by SEC in $CHCl_3$, leading to similar conclusions. Indeed, Figure B shows a decrease of the peak corresponding to the cyclic derivative of monomer **4** (at an elution volume of 8.4 mL) while the concentration of monomer increases. Also, molar masses of the linear polytriazoles are shifted toward high values. Therefore the concentration of monomer in the reaction mixture allows to tune the molar mass and architecture distributions of the resulting polytriazoles.

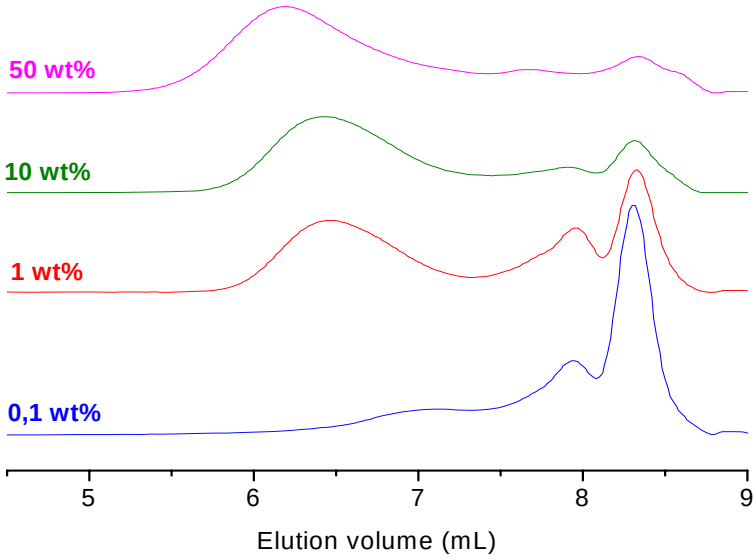
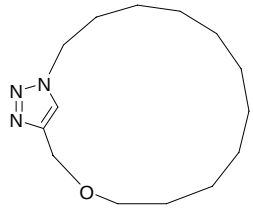


Figure B. SEC traces of crude polytriazole **7** for increasing concentrations of monomer **4** in $CHCl_3$.

The cycle issued from monomer **4** self-cyclization was isolated by recovering the filtrate after precipitation of the crude mixture in diethyl ether, and subsequent evaporation of the solvents.



^{1}H NMR ($CDCl_3$): δ (ppm) 7.56 (s, $OCH_2C{=}C\mathbf{H}$, 1H), 4.66 (s, $OC\mathbf{H}_2C{=}CH$, 2H), 4.42 (t, $NC\mathbf{H}_2CH_2CH_2$, J = 5.9 Hz, 2H), 3.51 (t, $C\mathbf{H}_2OCH2C{=}CH$, J = 6.4 Hz, 2H), 1.89 (m, $NCH_2C\mathbf{H}_2$, 2H), 1.62 (s, $C\mathbf{H}_2CH_2O$, 2H), 1.27 (s, $NCH_2CH_2(C\mathbf{H}_2)_7$, 14H). ^{13}C NMR ($CDCl_3$): δ (ppm) 146.72 ($CH_2\mathbf{C}{=}CH$), 122.82 ($CH_2C{=}\mathbf{C}H$), 69.33 ($O\mathbf{C}H_2C{=}CH$), 64.18 ($\mathbf{C}H_2OCH_2C{=}CH$), 50.59 ($CH_2\mathbf{C}H_2N$), 30.0.6, 29.72, 29.69, 29.38, 29.09, 28.55, 26.71, 26.50, 26.25 ($CH_2(\mathbf{C}H_2)_9CH_2$). MS (ESI-TOF): Signals at *m/z* 252.19 $[M+H]^+$, 51% and 274.19 $[M+Na]^+$, 100%.

FTIR study

In order to prove the contribution of triazole rings on the thermal behaviour of polytriazole **7**, FTIR spectra were recorded at temperatures ranging from 25 °C to 160 °C (Figure C). A decrease of the triazole signal at 3100 cm^{-1} is observed at temperatures above the melting temperature. In parallel, the shift of the C=C stretch band at 1640 cm^{-1} is observed. This was reversible upon cooling. These variations may be attributed to the presence of physical interactions between the triazole moieties, leading notably to a glass transition temperature higher than a C_{11} polyester analogue.

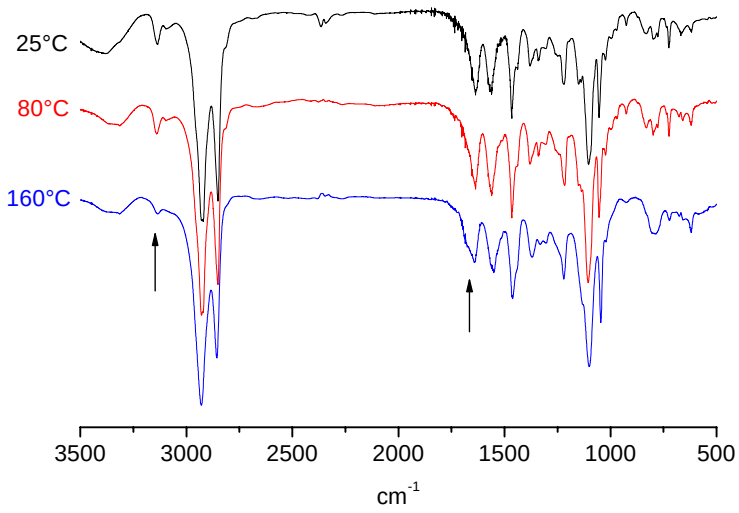


Figure C. FTIR of polytriazole **7** at 25, 80 and 160 °C

References

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