

Supplementary Information for

Synthesis of Pyrrole-based Poly(arylenevinylene)s via Rh-Catalyzed

Dehydrogenative Direct Alkenylation

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1. Materials

All reagents from commercial sources were used without further purification, unless otherwise noted. (Pentamethylcyclopentadienyl)rhodium(III) dichloride dimer ($[\text{Cp}^*\text{RhCl}_2]_2$) and 2-phenylpyridine was purchased from Sigma-Aldrich. $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ was purchased from Wako Pure Chemical Industries. The anhydrous solvents were purchased from Kanto Chemical. 2-Phenylpyrimidine was purchased from Tokyo Chemical Industry and purified by recrystallization from anhydrous hexane under nitrogen atmosphere. 1-(2-Pyrimidinyl)pyrrole (**1**) was prepared according to the literature method^{S1} and purified by recrystallization from hexane. 1-(2-Pyridinyl)pyrrole was prepared according to the literature method^{S2} and purified by High Performance Liquid Chromatography (HPLC). Tris(acetonitrile)(pentamethylcyclopentadienyl)rhodium bis(hexafluoroantimonate) ($[\text{Cp}^*\text{Rh}(\text{CH}_3\text{CN})_3][\text{SbF}_6]_2$) was prepared according to the literature method.^{S3} 2,7-Diethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene^{S4} and 1,4-bis(2-ethylhexyloxy)-2,5-ethenylbenzene^{S4a, S5} were synthesized referring to previous reports.

2. General methods

¹H and ¹³C{¹H} spectra were recorded on Bruker AVANCE-400 or AVANCE-600 NMR spectrometers. ¹H and ¹³C{¹H} NMR spectra were measured with tetramethylsilane (TMS) as an internal standard. Gel permeation chromatography (GPC) measurements were carried out on a SHIMADZU prominence GPC system equipped with polystyrene gel columns, using DMF solution of LiCl (0.01 M) as an eluent after calibration with polystyrene standards. The absorption spectra were recorded on a JASCO V-630 spectrometer. MALDI-TOF-MS spectra were recorded on an AB SCIEX MALDI TOF/TOF 5800 using *trans*-2-[3-(4-*tert*-Butylphenyl)-2-methyl-2-propenylidene]malononitrile (DCTB) as matrix. The highest occupied molecular orbital (HOMO) levels were estimated by photoelectron yield spectroscopy (PYS)

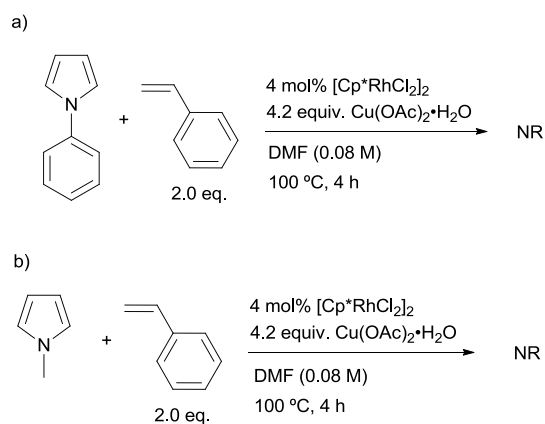
using an AC-3 spectrometer (Riken Kieiki). Elemental analyses were carried out with a Perkin-Elmer 2400 CHN Elemental Analyzer. Thermogravimetric analyses (TGA) were carried out using TG/DTA7300 systems from Seiko Instruments Inc.

3. Model reaction

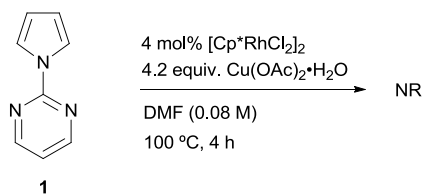
3.1 Synthesis of compound **2**⁵⁶

A mixture of $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (168 mg, 0.84 mmol), $[\text{Cp}^*\text{RhCl}_2]_2$ (4.94 mg, 8.0 μmol), **1** (29.0 mg, 0.20 mmol), and styrene (45.8 μL , 0.40 mmol) was stirred in anhydrous DMF (2.4 mL) for 4 h at 100 °C under nitrogen atmosphere in the dark. Then the reaction mixture was cooled to room temperature. To the mixture was added dichloromethane (40 mL) and ethylenediamine (1.6 mL). The organic layer was washed with distilled water (40 mL x 3) and dried over sodium sulfate. The product was isolated by column chromatography on silica gel using chloroform as an eluent. 2-[2,5-Bis(*E*-2-phenylethenyl)-1*H*-pyrrol-1-yl]pyrimidine (**2**) was obtained as a yellow solid (63.2mg, 91%). ¹H NMR (400 MHz, CDCl_3): δ 8.92 (d, $J = 4.8$ Hz, 2H), 7.37-7.35 (m, 5H), 7.29 (t, $J = 7.7$ Hz, 4H), 7.18 (t, $J = 7.3$ Hz, 2H), 7.14 (d, $J = 16.1$ Hz, 2H), 6.91 (d, $J = 16.1$ Hz, 2H), 6.73 (s, 2H). The ¹H NMR spectrum essentially agrees with those in the previous report.⁵⁷

3.2 Control experiments of model reaction



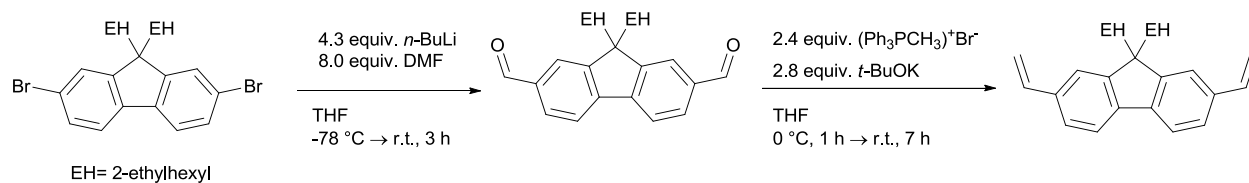
Scheme S1 Control experiments using (a) 1-phenylpyrrole and (b) 1-methylpyrrole.



Scheme S2 Control experiment in the absence of styrene.

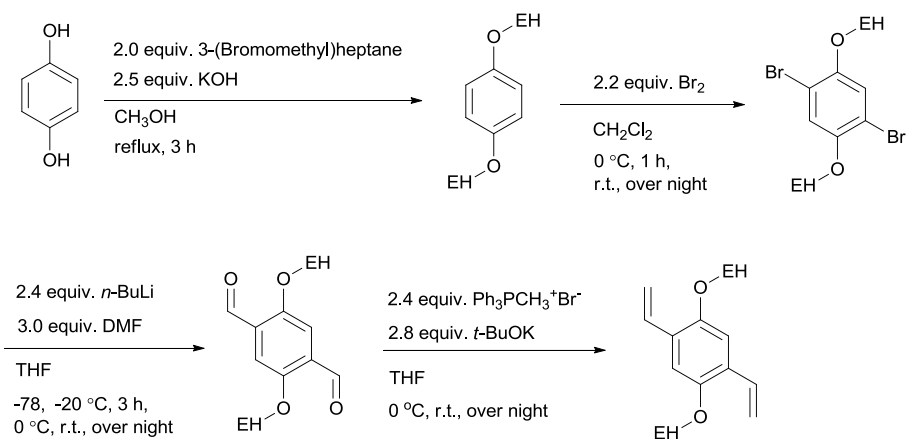
4. Synthesis of monomers

4.1 Synthesis of 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene (Scheme S3)^{S4}



Scheme S3 Synthesis of 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene

4.2 Synthesis of 1,4-diethenyl-2,5-bis[(2-ethylhexyl)oxy]benzene (Scheme S4)^{S4a, S5}



Scheme S4 Synthesis of 1,4-bis(2-ethylhexyloxy)-2,5-ethenylbenzene

5. Rh-catalyzed dehydrogenative direct alkenylation polycondensation

5.1 General procedure for Rh-catalyzed dehydrogenative direct alkenylation polycondensation

A mixture of $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (168 mg, 0.84 mmol), $[\text{Cp}^*\text{RhCl}_2]_2$ (4.94 mg, 8.0 μmol), directing-group-containing arene monomer (0.20 mmol), and divinyl monomer (0.20 mmol) was stirred in anhydrous DMF (2.4 mL) at a prescribed temperature for a prescribed time under nitrogen atmosphere in the dark. Then the reaction mixture was poured into a vigorously stirred mixture of distilled water (40 mL) and ethylenediamine (1.6 mL). The suspension was stirred overnight in the dark. The precipitate was separated by filtration and washed with the following solutions and solvents: aqueous solution of ethylenediaminetetraacetic acid disodium salt (0.1 M, pH = 8), aqueous solution of sodium hydroxide (0.1 M), distilled water, methanol, and hexane. The precipitate was dissolved in chloroform and the

solution was filtered through a Celite plug. A reprecipitation from chloroform/methanol gave a polymeric product.

5.2 Synthesis of Polymer 1 (Scheme 2, Table 1, entry 2)

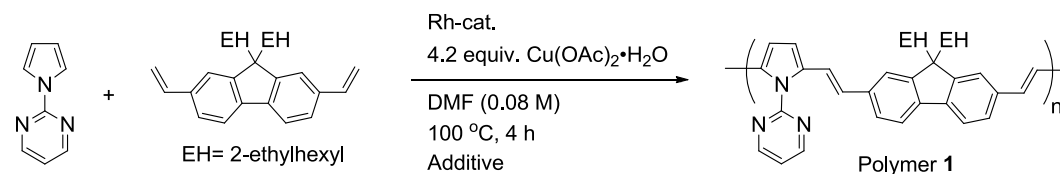
1 (29.0 mg, 0.20 mmol) and 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene (88.5 mg, 0.20 mmol) were used as the monomers. The reaction was carried out at 100 °C for 4 h, giving poly[[1-(2-pyrimidinyl)-1H-pyrrole-2,5-diyl]-1,2-ethenediyl[9,9-bis(2-ethylhexyl)-9H-fluorene-2,7-diyl]-1,2-ethenediyl] (Polymer **1**) as an orange solid in 81% yield (94.9 mg, $M_n = 22400$, PDI = 5.6). ^1H NMR (400 MHz, CDCl_3): δ . 8.94 (d, $J = 4.8$ Hz, 2H), 7.55 (d, $J = 8.0$ Hz, 2H), 7.38 (t, $J = 4.8$ Hz, 1H), 7.34-7.31 (br, 4H), 7.19-7.15 (br m, 2H), 6.99 (d, $J = 16.8$ Hz, 2H), 6.76 (s, 2H), 1.95 (br s, 4H), 0.88-0.48 (br m, 30H). $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.6, 157.7, 151.1, 140.4, 136.3, 134.9, 127.8, 125.1, 125.0, 124.9, 122.0, 121.8, 121.6, 119.5, 119.0, 117.7, 109.7, 54.5, 44.6, 34.5, 33.7, 28.1, 28.0, 26.9, 22.7, 14.0, 10.3. Anal. calcd. for $\text{C}_{41}\text{H}_{49}\text{N}_3$: C 84.34, H 8.46, N 7.20; found C 83.42, H 8.44, N 6.93. Anal. calcd. for $\text{C}_{41}\text{H}_{49}\text{N}_3$: C 84.34, H 8.46, N 7.20; found C 82.51, H 8.24, N 7.04.

A part of the product (30 mg) was further purified. Ethylenediamine (30 μL) was added to a solution of the polymer in THF. The solution was stirred for 1 h and the polymer was reprecipitated in distilled water. The separated polymer was washed with distilled water and methanol. A reprecipitation from THF/distilled water gave the purified polymer (21 mg, 70%). Anal. calcd. for $\text{C}_{41}\text{H}_{49}\text{N}_3$: C 84.34, H 8.46, N 7.20; found C 83.42, H 8.44, N 6.93.

This purification step improves the result of elemental analysis but the physical properties of the parent polymer and the purified polymer were essentially same.

5.3 Effect of catalyst and additive on direct alkenylation polycondensation

Table S1. Direct alkenylation polycondensation of **1** with 2,7-ethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene under various conditions ^a

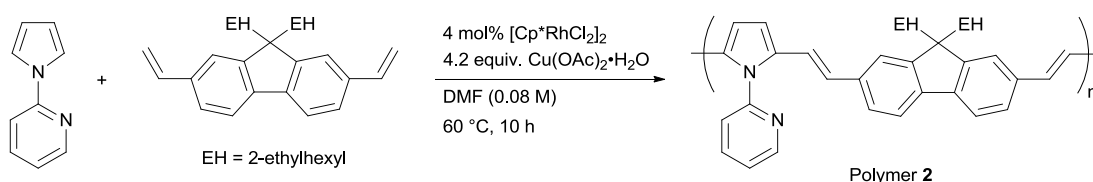


Entry	Rh-cat. (mol%)	Additive (equiv.)	Yield / %	$M_n \times 10^{-3}$	M_w/M_n
S1	$[\text{Cp}^*\text{Rh}(\text{MeCN})_3](\text{SbF}_6)_2$ (8)	-	70	10.7	3.8
S2	$[\text{Cp}^*\text{RhCl}_2]_2$ (2)	-	37	5.5	2.0
S3 ^b	$[\text{Cp}^*\text{RhCl}_2]_2$ (2)	-	68	9.4	2.3
S4	$[\text{Cp}^*\text{RhCl}_2]_2$ (4)	K_2CO_3 (4.2)	0	-	-
S5	$[\text{Cp}^*\text{RhCl}_2]_2$ (4)	1-AdCOOH (4.2)	86	14.3	2.9

^a Reactions were conducted using Rh-catalyst and $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (4.2 equiv.) in DMF (0.08 M) at 100 °C for 4 h. ^b Reaction was conducted for 8 h.

5.4 Synthesis of Polymer 2 (Scheme S5)

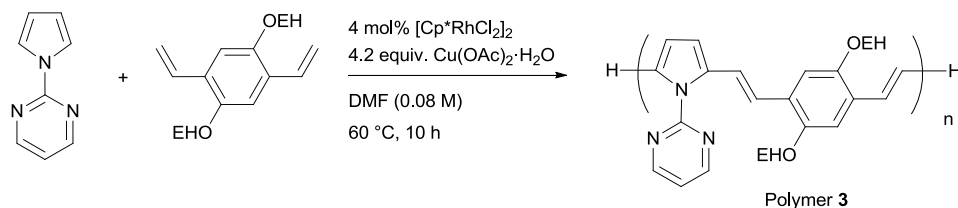
1-(2-Pyridinyl)pyrrole (25.9 μL , 0.20 mmol) and 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9H-fluorene (88.5 mg, 0.20 mmol) were used as the monomers. The reaction was carried out at 60 $^{\circ}\text{C}$ for 10 h, giving poly[[1-(2-pyridinyl)-1H-pyrrole-2,5-diyl]-1,2-ethenediyl[9,9-bis(2-ethylhexyl)-9H-fluorene-2,7-diyl]-1,2-ethenediyl] (Polymer 2) as an orange solid in 85% yield (99.5 mg, $M_n = 55800$, PDI = 4.2). ^1H NMR (400 MHz, CDCl_3): 8.77 (s, 1H), 7.92-7.88 (m, 1H), 7.51-7.46 (m, 3H), 7.36 (d, $J = 7.8$ Hz, 1H), 7.22 (br, 4H), 6.93-6.87 (br m, 2H), 6.76-6.73 (br m, 4H), 1.90 (s, 4H), 0.86-0.62 (m, 22H), 0.51-0.44 (m, 8H). $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 151.3, 151.1, 149.6, 140.3, 138.1, 136.1, 134.6, 127.4, 125.0, 124.9, 124.8, 123.6, 123.1, 121.8, 121.7, 121.4, 119.5, 116.8, 108.7, 54.5, 44.5, 34.5, 33.7, 28.0, 26.9, 22.7, 14.0, 10.3. Anal. calcd. for $\text{C}_{42}\text{H}_{50}\text{N}_2$: C 86.55, H 8.65, N 4.81; found C 86.17, H 8.73, N 4.61.



Scheme S5 Synthesis of Polymer 2 via Rh-catalyzed dehydrogenative direct alkenylation

5.5 Synthesis of Polymer 3 (Scheme S6)

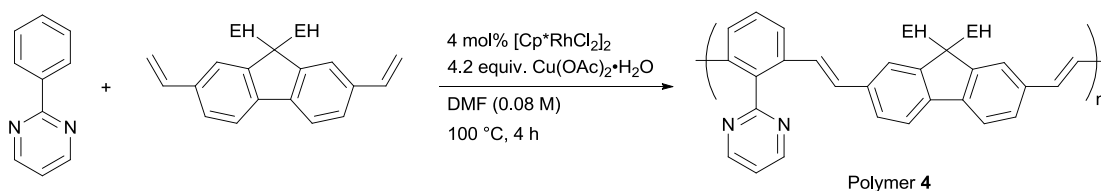
1 (29.0 mg, 0.20 mmol) and 1,4-diethenyl-2,5-bis[(2-ethylhexyl)oxy]benzene (77.3 mg, 0.20 mmol) were used as the monomers. The reaction was carried out at 60 $^{\circ}\text{C}$ for 10 h, giving poly[[1-(2-pyrimidinyl)-1H-pyrrole-2,5-diyl]-1,2-ethenediyl[2,5-bis(2-ethylhexyl)-1,4-phenylene]-1,2-ethenediyl] (Polymer 3) as a dark red solid in 78% yield (82.8 mg, $M_n = 23800$, PDI = 2.9). ^1H NMR (400 MHz, CDCl_3): δ 8.88 (2H, d, $J = 4.8$ Hz), 7.31 (1H, t, $J = 4.9$ Hz), 7.19 (2H, d, $J = 16.1$ Hz), 7.11 (2H, d, $J = 16.1$ Hz), 6.87 (2H, s), 6.71 (2H, s), 3.79 (4H, d, $J = 5.3$ Hz), 1.72-1.66 (2H, m), 1.50-1.32 (17H, m), 0.91 (12H, t, $J = 7.4$ Hz). $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.5, 157.8, 150.9, 135.4, 127.0, 121.9, 118.9, 118.3, 110.3, 110.0, 71.8, 39.6, 30.7, 29.1, 24.0, 23.1, 14.1, 11.2.



Scheme S6 Synthesis of Polymer 3 via Rh-catalyzed dehydrogenative direct alkenylation

5.6 Synthesis of Polymer 4 (Scheme S7)

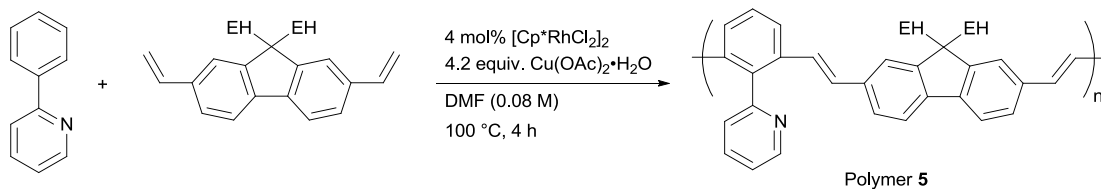
2-Phenylpyrimidine (31.0 mg, 0.20 mmol) and 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9*H*-fluorene (88.5 mg, 0.20 mmol) were used as the monomers. The reaction was carried out at 100 °C for 4 h, giving poly[[9,9-bis(2-ethylhexyl)-9*H*-fluorene-2,7-diyl]-1,2-ethenediyl[2-(2-pyrimidinyl)-1,3-phenylene]-1,2-ethenediyl] (Polymer 4) as pale yellow solid in 79% yield (93.9 mg, $M_n = 10100$, PDI = 1.9). ^1H NMR (400 MHz, CDCl_3): δ . 8.95 (d, $J = 4.9$ Hz, 2H), 7.72-7.71 (m, 2H), 7.52 (d, $J = 7.9$ Hz, 2H), 7.48 (t, $J = 7.7$ Hz, 1H), 7.35-7.34 (m, 1H), 7.25-7.22 (m, 4H), 7.04 (dt, $J = 15.6, 6.6$ Hz, 2H), 6.80-6.74 (m, 2H), 1.94-1.84 (m, 4H), 0.85-0.62 (m, 23H), 0.47-0.44 (m, 8H). $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.9, 157.0, 151.0, 140.7, 137.1, 136.7, 136.0, 131.5, 129.0, 125.9, 125.6, 125.4, 124.9, 122.5, 122.1, 121.9, 121.6, 119.6, 119.0, 54.6, 44.5, 34.6, 34.5, 33.7, 28.0, 27.1, 26.9, 22.7, 22.6, 14.0, 10.4, 10.3, 10.2.



Scheme S7 Synthesis of Polymer 4 via Rh-catalyzed dehydrogenative direct alkenylation

5.7 Synthesis of Polymer 5 (Scheme S8)

2-Phenylpyridine (28.5 μL , 0.20 mmol) and 2,7-diethenyl-9,9-bis(2-ethylhexyl)-9*H*-fluorene (88.5 mg, 0.20 mmol) were used as the monomers. The reaction was carried out at 100 °C for 4 h, giving poly[[9,9-bis(2-ethylhexyl)-9*H*-fluorene-2,7-diyl]-1,2-ethenediyl[2-(2-pyridinyl)-1,3-phenylene]-1,2-ethenediyl] (Polymer 5) as pale yellow solid in 82% yield (96.9 mg, $M_n = 21300$, PDI = 2.5). ^1H NMR (400 MHz, CDCl_3): δ . 8.84 (s, 1H), 7.80-7.70 (m, 3H), 7.52 (d, $J = 8.0$ Hz, 2H), 7.46 (t, $J = 7.8$ Hz, 1H), 7.36 (d, $J = 7.5$ Hz, 2H), 7.22 (m, 4H), 7.05 (dt, $J = 16.1, 4.1$ Hz, 2H), 6.78 (d, $J = 15.8$ Hz, 2H), 1.87 (br, 4H), 0.85-0.61 (br m, 22H), 0.48-0.43 (br m, 8H). $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.6, 151.1, 149.5, 140.7, 138.5, 136.8, 136.0, 135.9, 130.8, 128.5, 126.5, 126.4, 125.9, 125.7, 125.4, 124.6, 122.4, 122.0, 121.7, 119.6, 54.5, 44.4, 34.5, 33.7, 28.0, 26.9, 22.6, 14.0, 10.3.



Scheme S8 Synthesis of Polymer 5 via Rh-catalyzed dehydrogenative direct alkenylation

6. NMR spectra data and MALDI-TOF-MS spectra data

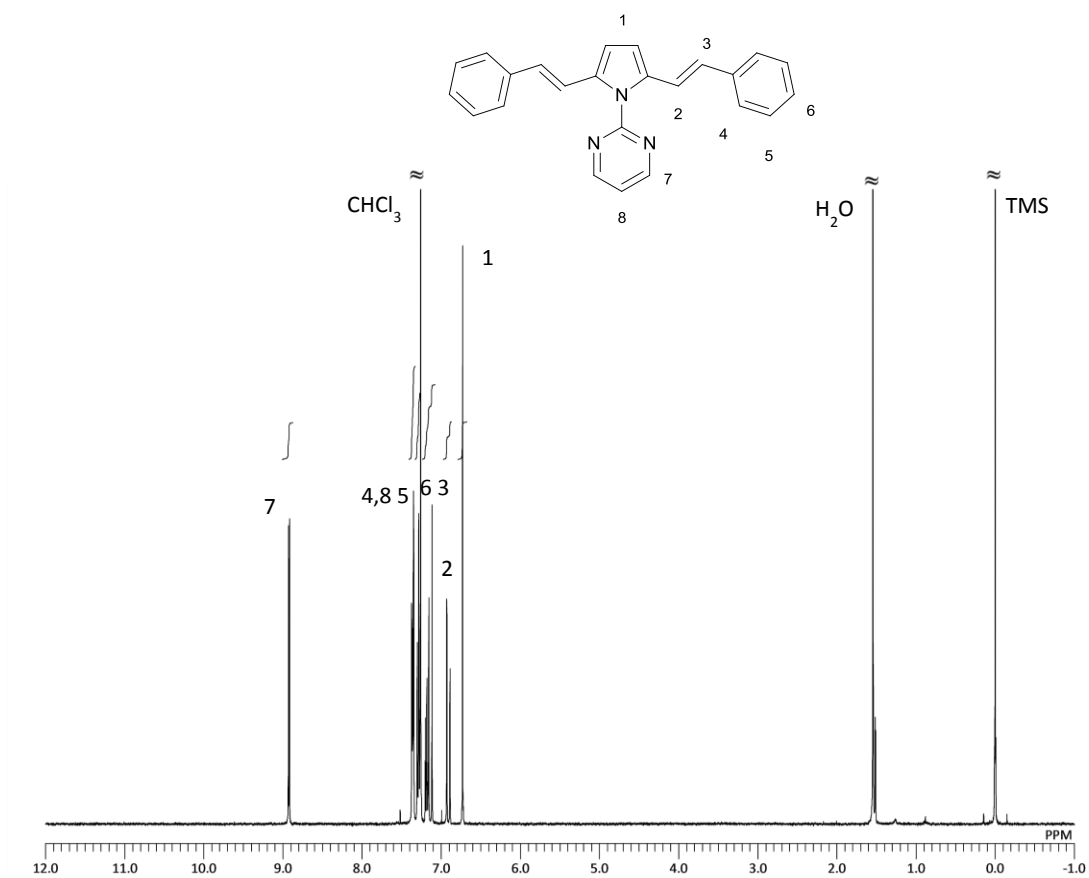


Fig. S1 ^1H NMR spectrum of **2** (CDCl_3 , 400 MHz).

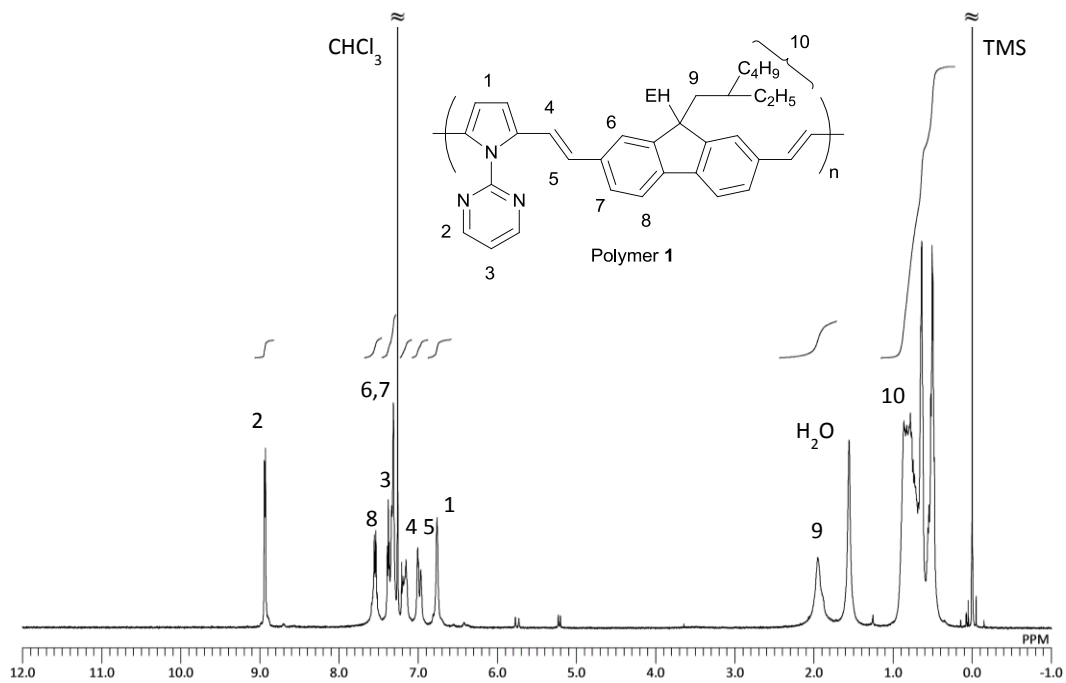


Fig. S2 ^1H NMR spectrum of Polymer 1 (CDCl_3 , 400 MHz).

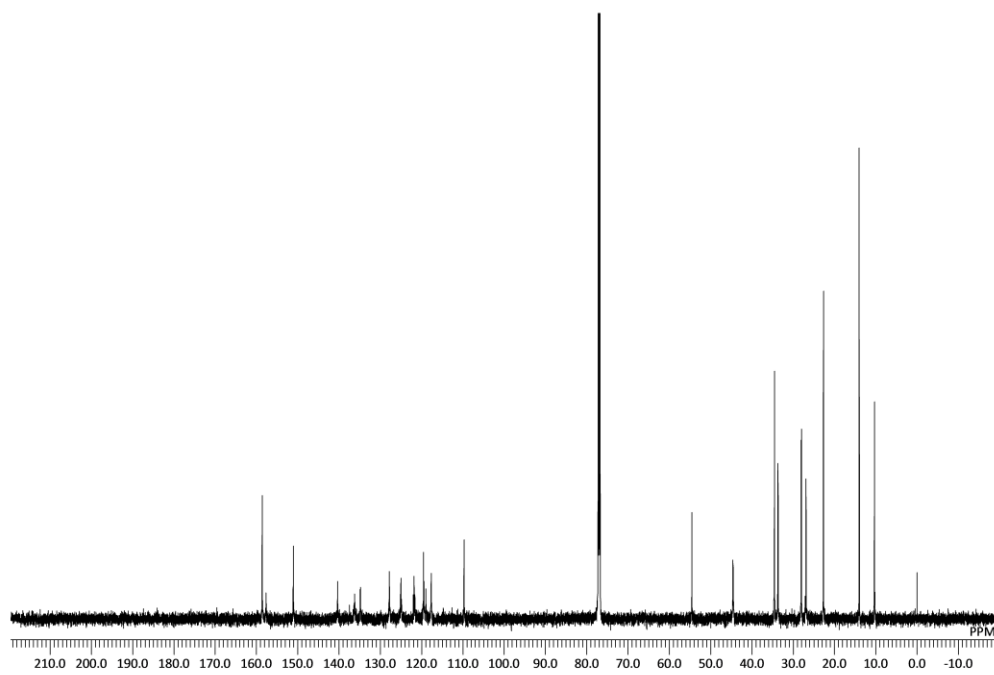


Fig. S3 $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of Polymer 1 (CDCl_3 , 150 MHz).

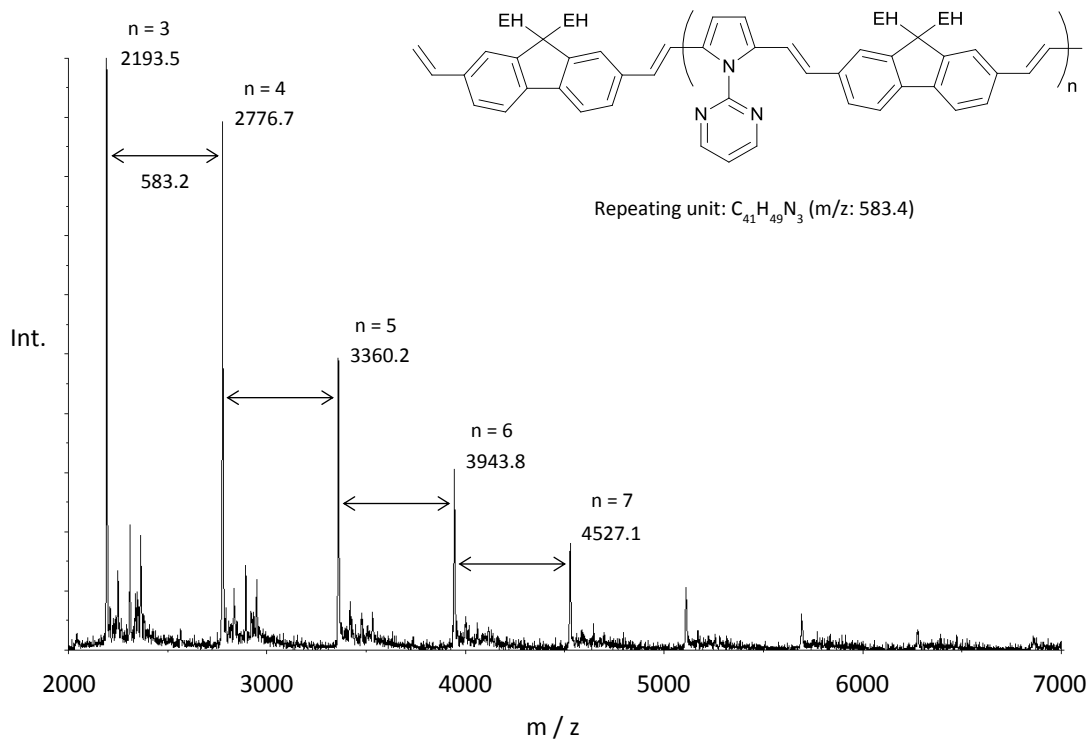


Fig. S4 MALDI-TOF-MS spectrum of Polymer 1.

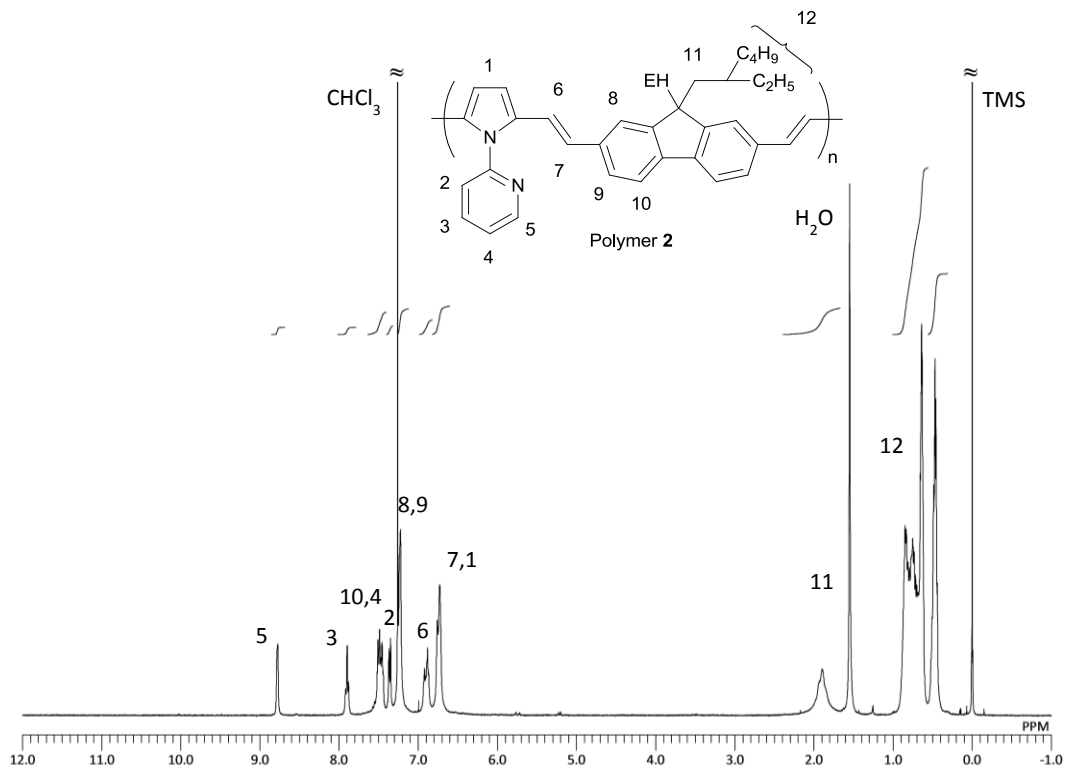


Fig. S5 ¹H NMR spectrum of Polymer 2 (CDCl₃, 400 MHz).

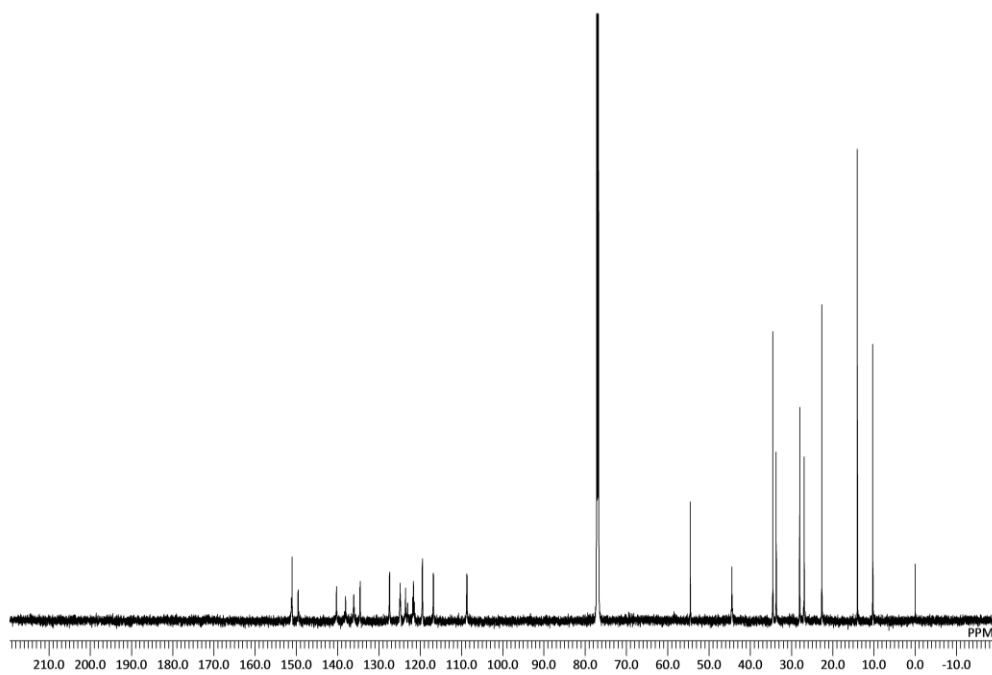


Fig. S6 ¹³C{¹H} NMR spectrum of Polymer 2 (CDCl₃, 150 MHz).

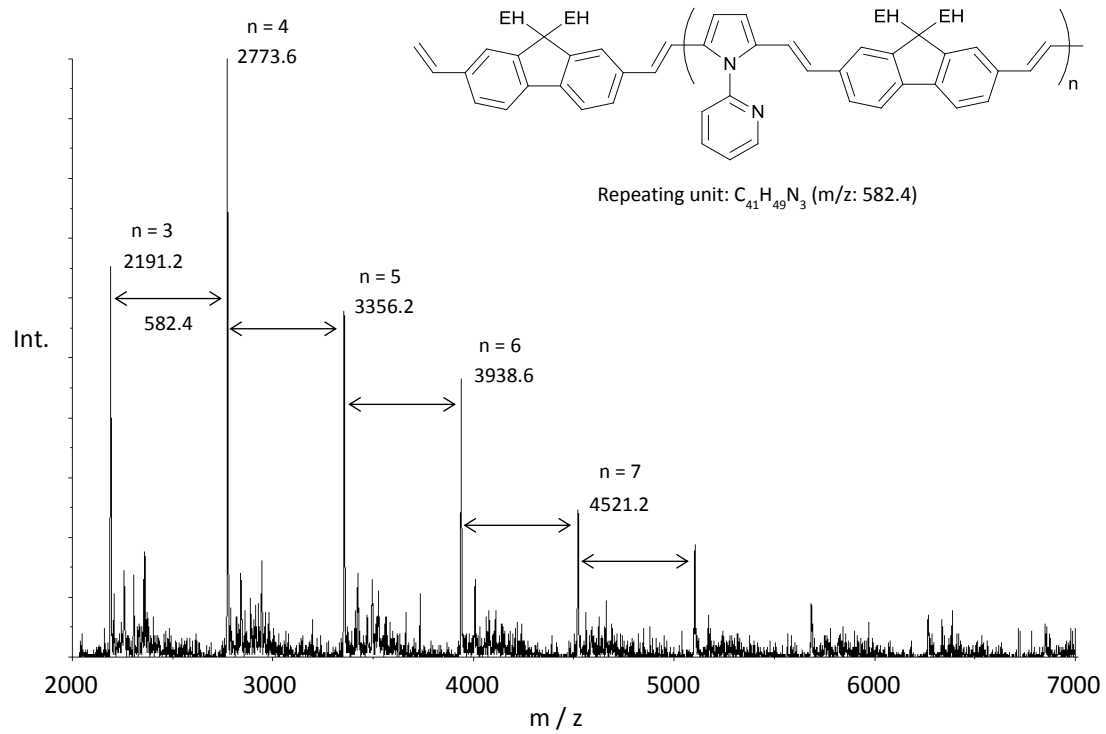


Fig. S7 MALDI-TOF-MS spectrum of Polymer 2.

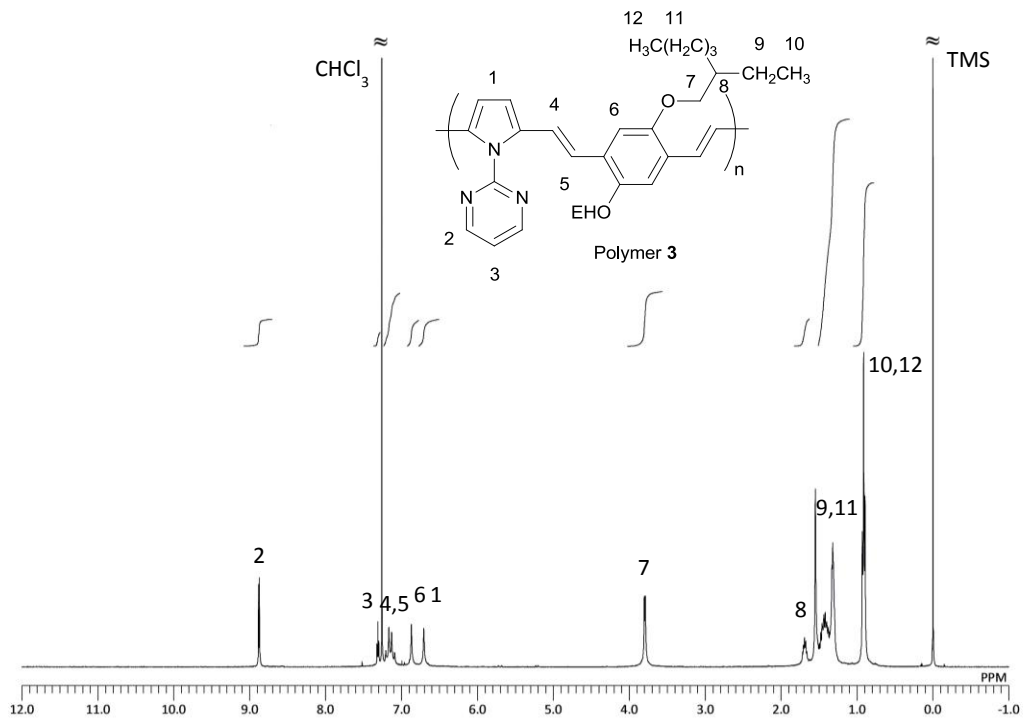


Fig. S8 ^1H NMR spectrum of Polymer 3 (CDCl_3 , 400 MHz).

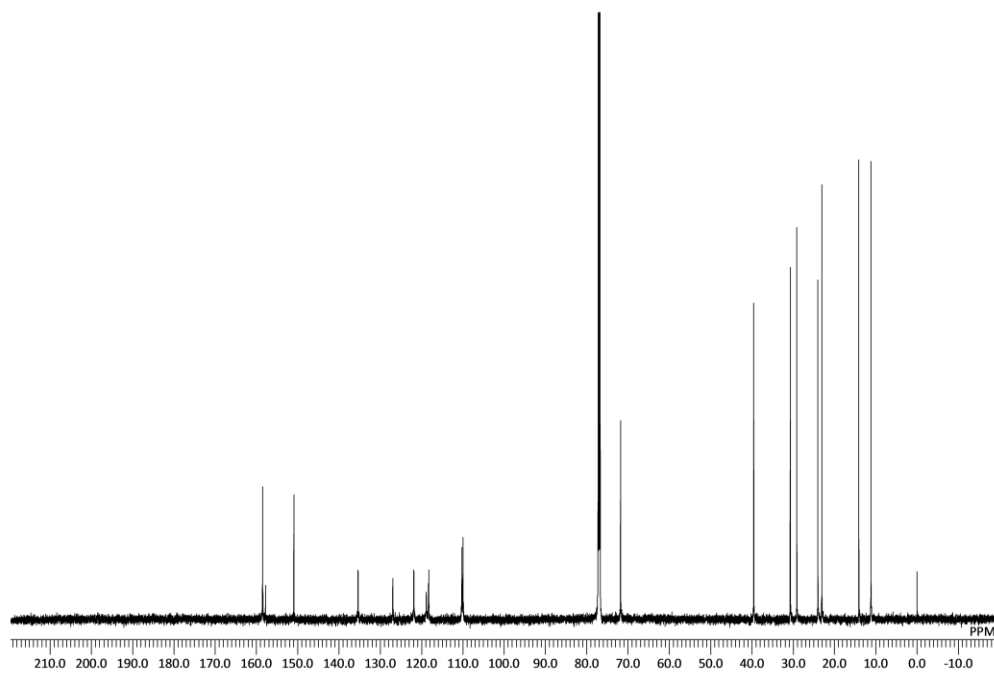


Fig. S9 $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of Polymer 3 (CDCl_3 , 150 MHz).

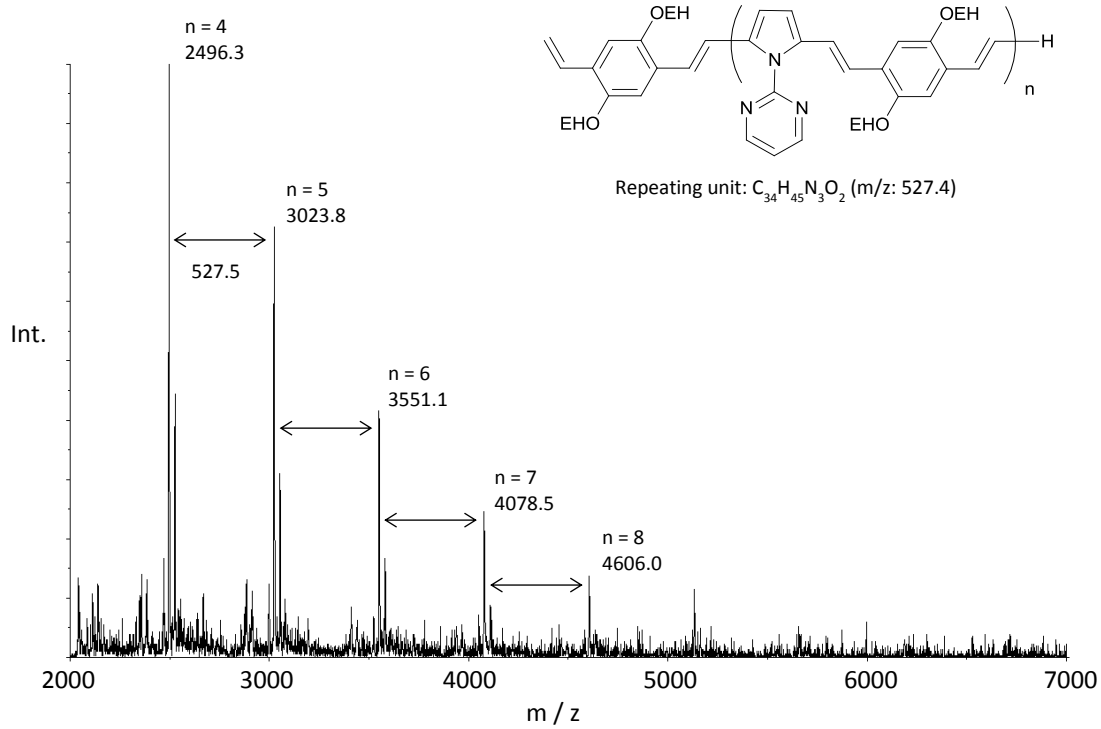


Fig. S10 MALDI-TOF-MS spectrum of Polymer 3.

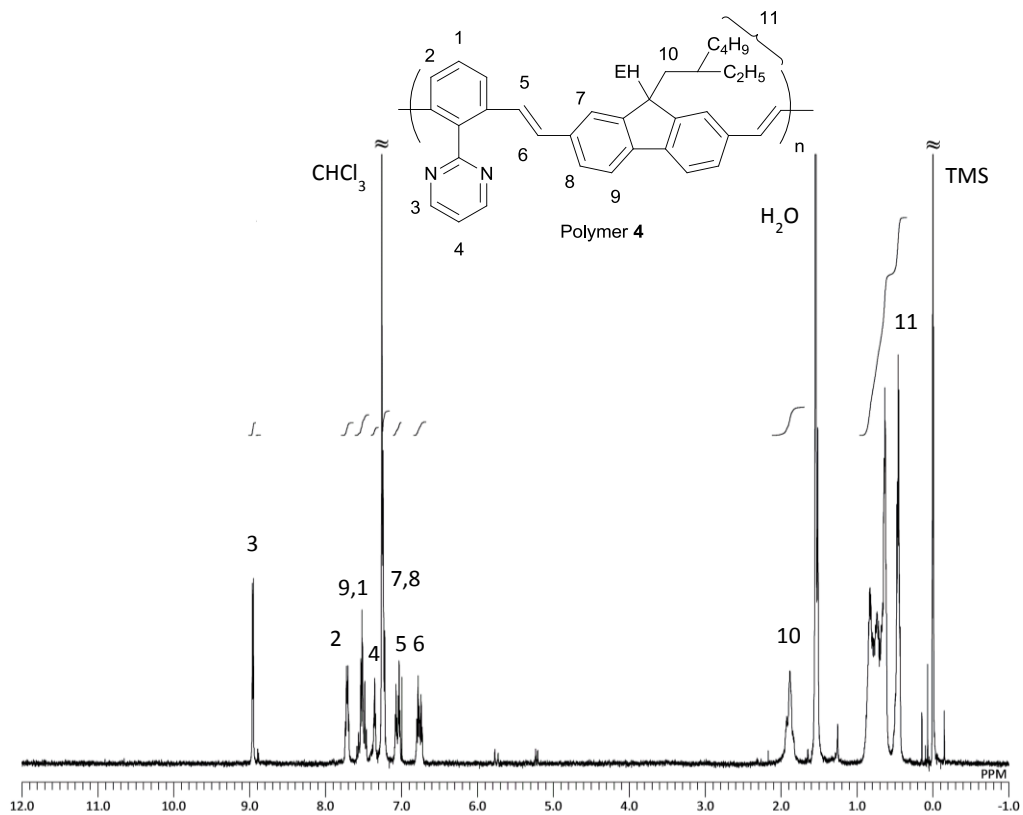


Fig. S11 ^1H NMR spectrum of Polymer 4 (CDCl_3 , 400 MHz).

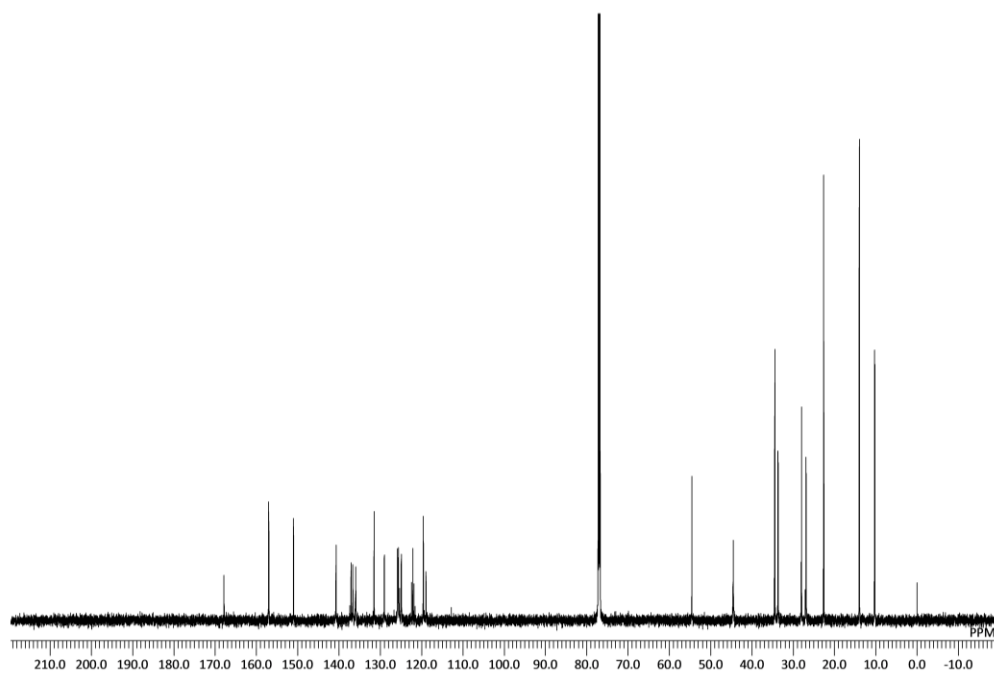


Fig. S12 $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of Polymer 4 (CDCl_3 , 150 MHz).

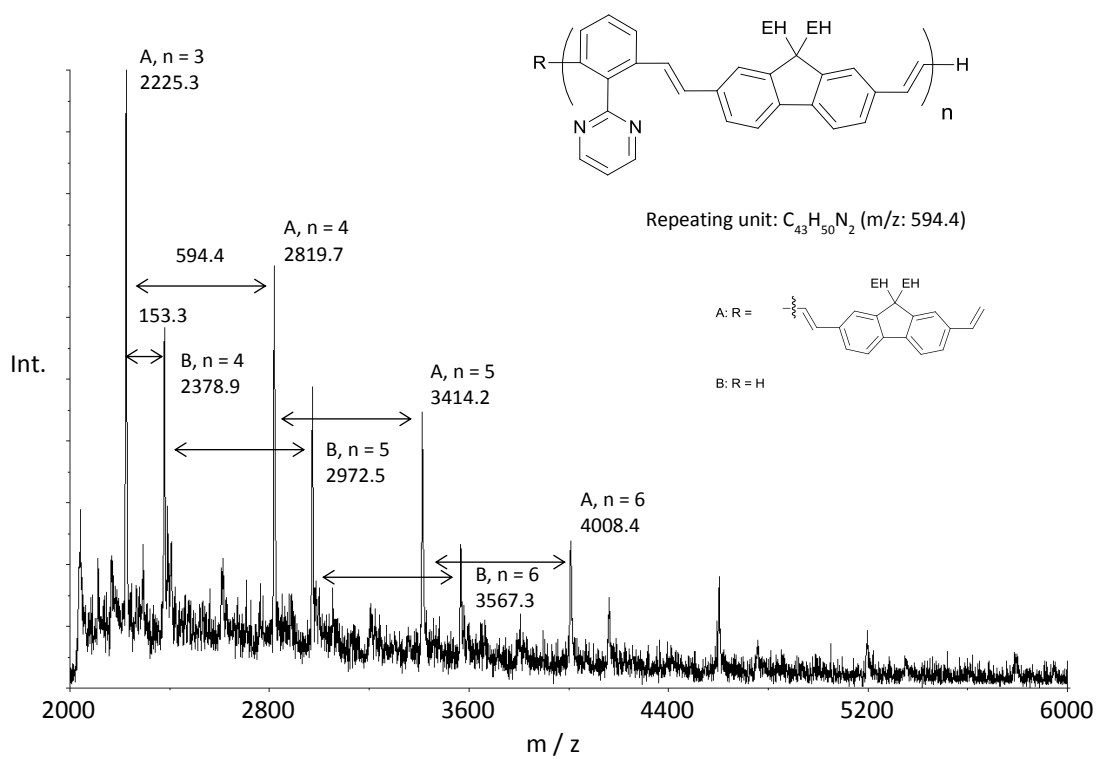


Fig. S13 MALDI-TOF-MS spectrum of Polymer 4.

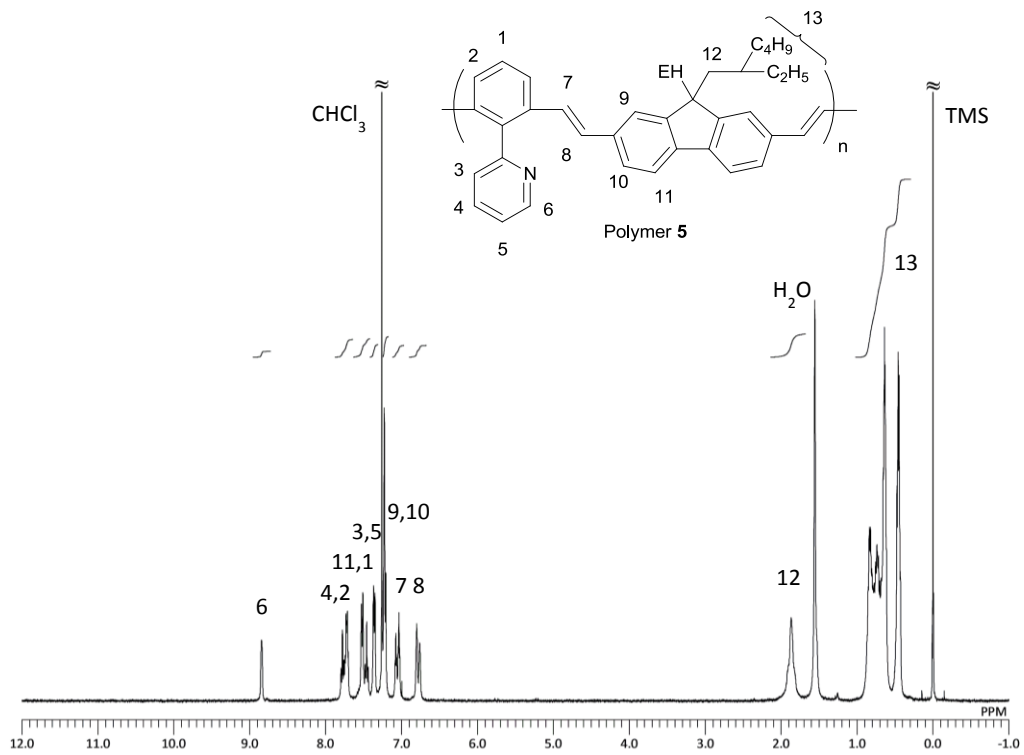


Fig. S14 ¹H NMR spectrum of Polymer 5 (CDCl₃, 400 MHz).

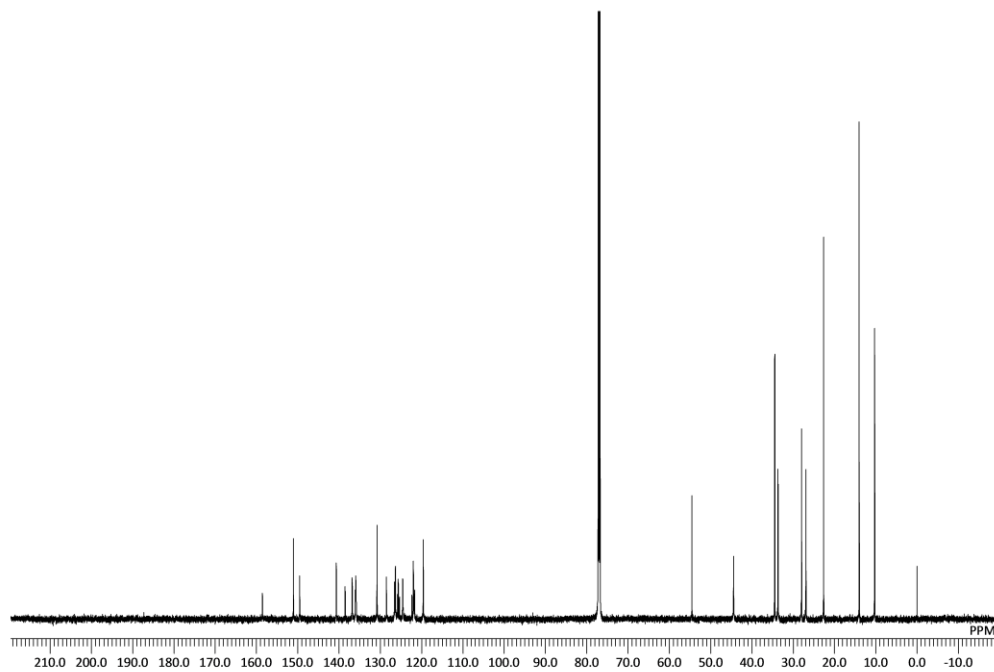


Fig. S15 ¹³C{¹H} NMR spectrum of Polymer 5 (CDCl₃, 150 MHz).

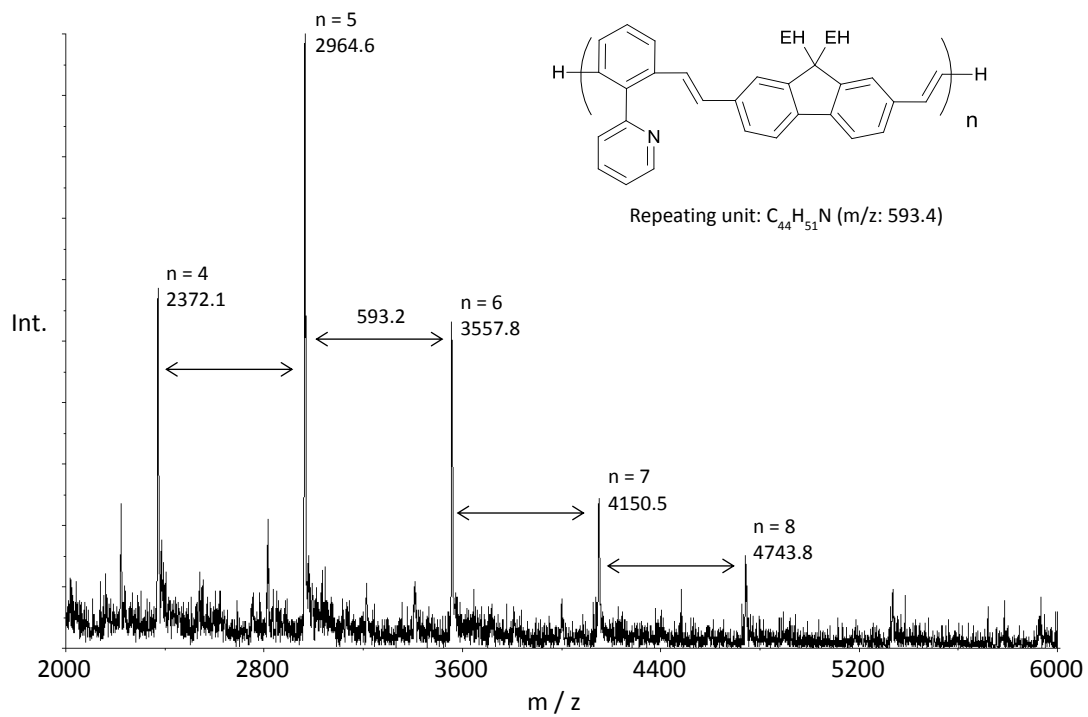


Fig. S16 MALDI-TOF-MS spectrum of Polymer 5.

7. Fabrication and characterization of FETs

To estimate the hole mobilities of the Polymers **1,2**, OFETs with a top-contact geometry were fabricated and characterized as follows. A glass/Au gate electrode/Parylene-C insulator substrate was prepared according to the previously reported methods.⁵⁸ The Polymers **1,2** were spin-coated from toluene solution onto the Parylene-C layer. The coated substrate was then transferred to a N₂-filled glove box where it was dried for 10 min at 110 °C. Au (40 nm) source-drain electrodes were thermally evaporated onto the substrates through shadow masks. The channel length and width were fixed at 75 μm and 5 mm, respectively. The OFET measurements were conducted using a Keithley 2636A System Source Meter under vacuum.

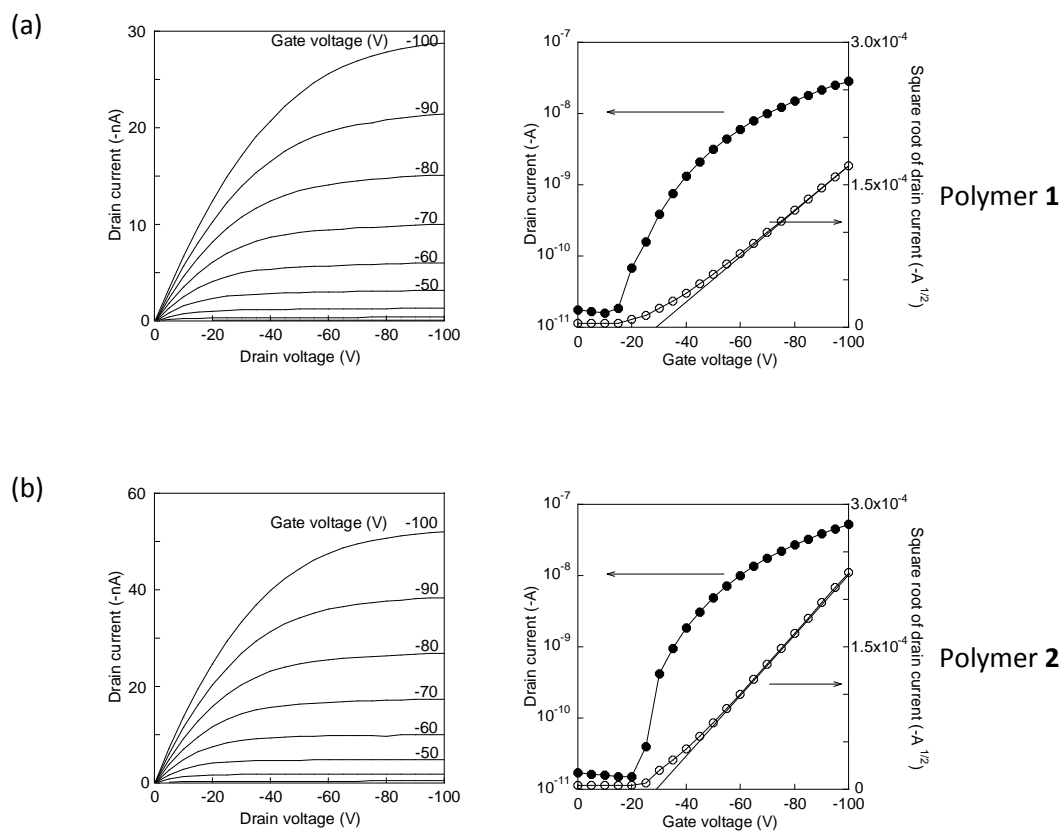


Fig. S17 Typical characteristics of the top-contact FETs for (a) Polymer **1** and (b) Polymer **2** films. Left: output curves at different gate Voltage; right: transfer curves at $V_{sd} = -100$ V

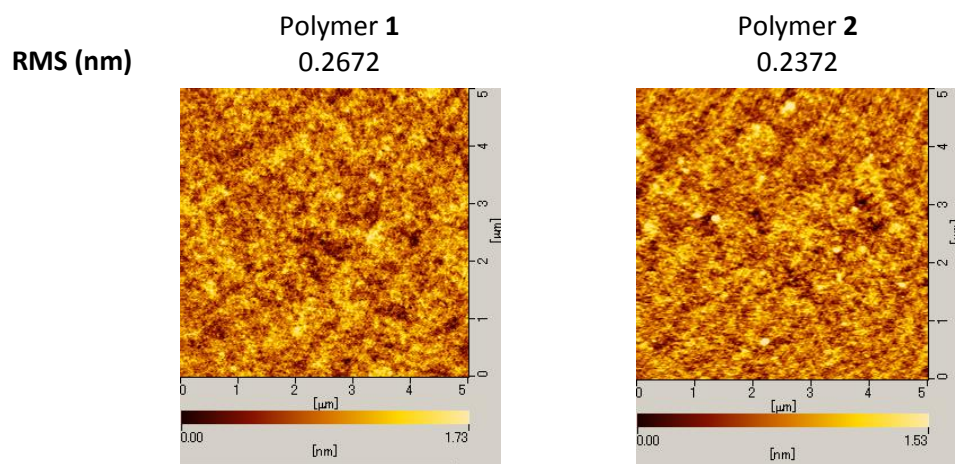
Table S2. OFET characteristics ^a

Polymer	μ_h^b [$\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$]	on / off ratio	V_{th}^c [V]
Polymer 1	7.13×10^{-5}	1.26×10^3	-28.4
Polymer 1	7.85×10^{-5}	1.80×10^3	-27.2
Polymer 1	8.33×10^{-5}	1.85×10^3	-26.1
Polymer 1	6.74×10^{-5}	1.34×10^3	-31.1
Ave	$7.5 \pm 0.7 \times 10^{-5}$	$1.6 \pm 0.3 \times 10^3$	-28 ± 2
Polymer 2	1.89×10^{-4}	1.24×10^3	-34.9
Polymer 2	1.63×10^{-4}	3.43×10^3	-29.0
Polymer 2	1.34×10^{-4}	2.76×10^3	-30.4
Polymer 2	2.25×10^{-4}	4.11×10^3	-29.2
Ave	$1.8 \pm 0.4 \times 10^{-4}$	$3 \pm 1 \times 10^3$	-31 ± 3

^a OFET configuration; Glass / Au gate electrode / Parylene-C insulator / Polymer / Au source-drain electrodes.

^b Field-effect hole mobility.

^c Threshold voltage.

**Fig. S18** AFM images ($5 \times 5 \mu\text{m}^2$) of Polymers **1** and **2**.

8. Thermal properties

Table S3. Temperatures at 5% weight-loss

Polymer	Polymer 1 ^a	Polymer 2
Temp. (°C)	376	395

^a Table 1 Entry 2.

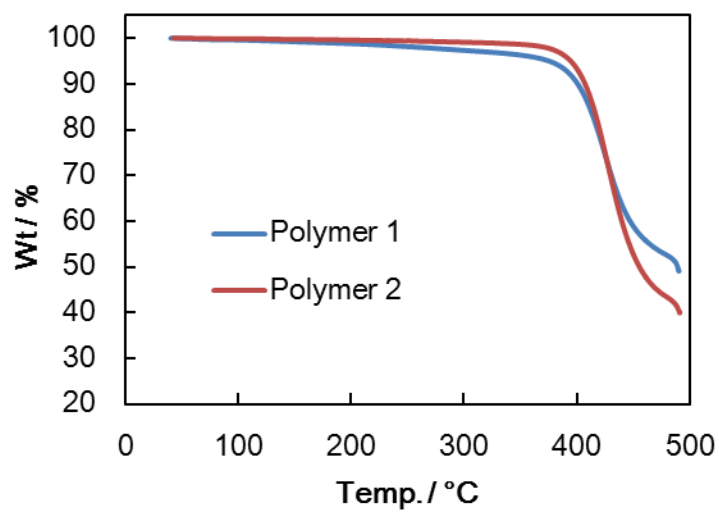


Fig. S19 TGA curves of Polymers 1 and 2 at a heating rate of 10 °C min⁻¹ under Ar.

10. References

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