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Electronic Supplementary Material

for

Controlling the enthalpy-entropy competition in supramolecular fullerene liquid crystals via flexible chain length

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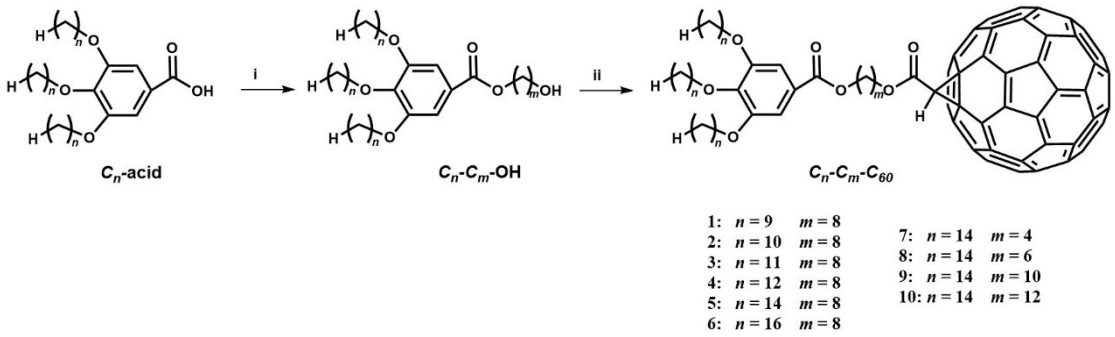
1. Materials and Methods

Reagents: *p*-toluenesulfonic acid (PTSA, 99 %) was purchased from J&K Chemical and used as received. 1-Bromononane (98 %), 1-bromodecane (98 %), 1-bromoundecane (98 %), 1-bromododecane (98 %), 1-bromotetradecane (98 %), 1-bromohexadecane (98 %), methyl 3,4,5-trihydroxybenzoate (98 %), 1,4-butanediol (chemically pure), 1,6-hexanediol (96.5 %), 1,8-octanediol (98 %), 1,10-decanediol (98 %), 1,12-dodecanediol (98 %), 4-(dimethylamino)pyridine (DMAP, 99 %), *N*,*N*-diisopropylcarbodiimide (DIPC, 99 %) and 1,2-dichlorobenzene (99 %) were purchased from Alfa Aesar and used as received. Trifluoroacetic acid (chemically pure), *N*,*N*-dimethylformamide (DMF, analytical reagent) were purchased from Sinopharm Chemical Reagent Co., Ltd, China, and used as received. [60]Fullerene (99.9 %) was purchased from Yongxin Technology Co., Ltd (Henan, China).

Instruments: NMR spectra were obtained on a Varian Mercury-400 MHz NMR spectrometer (400 MHz ^{1}H and 75 MHz ^{13}C Larmor frequency). ^{1}H NMR spectra were referenced to the solvent peak of $CDCl_3$ at δ 7.27 ppm. ^{13}C NMR spectra were referenced to $CDCl_3$ at δ 77.00 ppm. Elemental analysis experiments were performed on Vario EL cube. Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectra were acquired on a Bruker Ultraflex-Treme TOF/TOF mass spectrometer (Bruker Daltonics, Inc., Billerica, MA) equipped with a Nd:YAG laser (355 nm). All spectra were measured in positive reflection mode. The mass scale was calibrated externally using the peaks obtained from peptide standard at the molecular weight range under consideration. Trans-2-[3-(4-tert-butylphenyl)-2-methyl-2-propenylidenemalononitrile (DCTB) served as matrix and was prepared in $CHCl_3$ at a concentration of 20 mg/mL. Sodium trifluoroacetate (CF_3COONa) or silver trifluoroacetate (CF_3COOAg) were served as cationizing agent and prepared in methanol at a concentration of 10 mg/mL. Data analysis was conducted with Bruker's Flex Analysis software. Thermal gravimetric analysis (TGA) was carried out on a Discovery TGA with a heating rate of 10 °C/min in nitrogen atmosphere and about 1.0 mg of samples was used. Differential scanning calorimetry (DSC) measurements were performed on a TA DSC 2000 instrument over a temperature range of -50-120 °C at a scanning rate of 10 °C/min under a continuous nitrogen flow of 50 mL/min. About 5.0 mg of samples were used and sealed in the aluminum pans. The reference and sample pan weights were kept constant with a precision of ± 0.0001 mg. The phase transitions and their corresponding phase structural transformations were monitored by DSC experiment in the following three thermal processes. The samples were firstly heated up to 120 ºC (above the isotropic temperature) to eliminate any previous thermal history, and then subsequent cooling and heating experiments at a scan rate of 10 ºC/min were then recorded. Small angle X-ray scattering (SAXS) experiments were conducted on a X-ray scattering instrument (SAXSess mc^2, Anton Paar) equipped with line collimation and a 2200 W sealed-tube X-ray generator (Cu-Kα, λ = 0.154 nm), the samples were kept under vacuum during irradiation, and the irradiation time was 20 min at certain temperature, imaging plate (IP) was used to recorded the scattering pattern. Silver behenate was used as the calibration substance to calibrate the peak positions. Samples were prepared by melting on an aluminum substrate to obtain an about 0.5 mm thick film, and then first heated to 120 ºC to enter the isotropic state, subsequently cooled to the temperature range of liquid crystal at a scan rate of 1 ºC/min and annealed at a certain temperature for 12 hours, followed by cooling to 30 ºC. Polarizing optical microscopy (POM) observations were carried out on a polarizing microscope (Olympus Corporation, BX51-P), which was coupled with a computer-controlled video camera for obtaining pictures and a dual hot stage (Linkam THMS600) for controlling the temperature. The samples were placed between two glass slides, and firstly heated up to 120 ºC to remove their thermal history, then subsequently cooled to the temperature range of liquid crystal phase, and annealed at the corresponding temperature.

2. Synthetic Procedures and Characterization Data



Scheme S1. Synthetic rout of fullerene derivatives C_n-C_m-C_{60} (**1-10**).
Reagents and Conditions: (i) $HO(CH_2)_mOH$, PTSA, toluene, reflux, 20 hr; (ii) methano[60]fullerene carboxylic acid, DMAP, DIPC, PTSA, 1,2-dichlorbenzene, DMF, r.t., 2 hr.

Ten fullerene derivatives C_n-C_m-C_{60} (**1-10**) were synthesized following the procedure as listed in scheme S1. Here *n* denotes the alkyl chain length while *m* the flexible chain length. Firstly, 3,4,5-tri(alkyloxy)benzoic acids (C_n-acids) were reacted with excess α,ω-alkanediols using Steglich esterification reaction. Compound C_n-C_m-OH further underwent esterification reaction with methano[60]fullerene carboxylic acid, which was synthesized through the hydrolysis of t-butyl[60]fullerenoacetate, to give the final products of C_n-C_m-C_{60} with 60 % to 82 % yield after purification.

Methano[60]fullerene carboxylic acid was synthesized following reference procedure.[1, 2]

C_n-acid compounds were synthesized by a literature procedure.[3, 4]

Typical procedure for the synthesis of C_n-C_m-OH:

To a solution of C_9-acid (2.7 mmol) in toluene (80 mL) was added 1,8-octanediol (10.8 mmol) and PTSA (0.11 g) with stirring for 20 h at 140 °C. The mixture was poured into toluene (150 mL), filtered and the solution was washed with 5 % Na_2CO_3 twice, deionized water three times, dried with anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: ethyl acetate/petroleum ether = 1/8), and was recrystallized from acetone to give a white solid.

C_9-C_8-OH: ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.28-1.57 (m, 46H), 1.71-1.84 (m, 8H), 3.84 (t, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.25 (s, 2H). Yield: 82 %.

C_{11}-C_8-OH: ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.26-1.49 (m, 58H), 1.71-1.82 (m, 8H), 3.84 (t, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.24 (s, 2H). Yield: 80 %.

C_{16}-C_8-OH: ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.23-1.62 (m, 88H), 1.71-1.86 (m, 8H), 3.64 (t, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.25 (s, 2H). Yield: 82 %.

C_{14}-C_6-OH: ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.21-1.52 (m, 72H), 1.69-1.87(m, 8H), 3.64 (t, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.25 (s, 2H). Yield: 88 %.

C_{14}-C_{10}-OH: ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.18-1.53 (m, 80H), 1.67-1.87 (m, 8H), 3.61-3.7 (q, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.25 (s, 2H). Yield: 85 %.

C_{14}-C_{12}-OH: ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.18-1.54 (m, 84H), 1.69-1.87 (m, 8H), 3.61-3.7 (q, 2H), 4.01 (t, 6H), 4.28 (t, 2H), 7.25 (s, 2H). Yield: 83 %.

Typical procedure for the synthesis of C_n-C_m-C_{60}:

Compounds **1-10** were synthesized by the esterification of compound C_n-C_m-OH with methano[60]fullerene carboxylic acid. For example, a solution of C_9-C_8-OH (0.26 mmol) in a small amount of toluene was added to a mixture of [60]fullerene carboxylic acid (0.26 mmol) in 1,2-dichlorobenzene and DMF, then DMAP (0.26 mmol), PTSA (0.26 mmol) and DIPC (0.33 mmol) was added. The mixture was stirred for 2 h at r.t., then filtered and evaporated by a rotary evaporator under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: toluene/petroleum ether = 1/3). The obtained dark brown product was re-precipitated into methanol to obtain a brown solid.

1: Yield: 82 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.17-1.68 (m, 44H), 1.7-1.90 (m, 10H), 4.01 (t, 6H), 4.29 (t, 2H), 4.47 (t, 2H), 4.79 (s, 1H), 7.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.14, 22.70, 25.94, 26.06, 26.13, 28.69, 28.77, 29.20, 29.24, 29.31, 29.34, 29.37, 29.43, 29.58, 29.69, 30.34, 31.91, 31.94, 39.13, 65.04, 66.53, 69.20, 70.61, 73.50, 76.71, 77.03, 77.23, 77.35, 108.08, 125.03, 136.36, 140.48, 140.91, 141.11, 142.06, 142.07, 142.19, 142.38, 142.41, 142.80, 142.96, 142.98, 143.08, 143.25, 143.71, 143.93, 144.40, 144.57, 144.65, 144.67, 145.05, 145.06, 145.16, 145.20, 145.25, 145.56, 145.81, 148.26, 152.79, 166.43, 166.48. MALDI-TOF, Calc. for $C_{104}H_{76}O_7$: $(M)^+$ 1436.559. Found: 1437.185. Anal. Calc. for $C_{104}H_{76}O_7$ (1437.71): C, 86.88; H, 5.33. Found: C, 86.56; H, 5.52.

3: Yield: 65 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.20-1.57 (m, 56H), 1.67-1.90 (m, 10H), 4.01 (t, 6H), 4.29 (t, 2H), 4.46 (t, 2H), 4.79 (s, 1H), 7.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.15, 22.71, 25.94, 26.07, 26.13, 28.69, 28.77, 29.19, 29.24, 29.34, 29.38, 29.43, 29.59, 29.67, 29.71, 29.74, 30.34, 31.93, 31.94, 39.13, 65.04, 66.53, 69.19, 70.60, 73.51, 76.71, 77.03, 77.23, 77.35, 108.07, 125.03, 136.36, 140.47, 140.91, 141.11, 142.06, 142.07, 142.19, 142.37, 142.41, 142.80, 142.80, 142.96, 142.98, 143.08, 143.25, 143.71, 143.93, 144.40, 144.57, 144.65, 144.67, 145.05, 145.06, 145.16, 145.20, 145.25, 145.56, 145.81, 148.26, 152.79, 166.43, 166.48. MALDI-TOF, Calc. for $C_{110}H_{88}O_7$: $(M)^+$ 1520.653. Found: 1521.229. Anal. Calc. for $C_{110}H_{88}O_7$ (1521.87): C, 86.81; H, 5.83. Found: C, 86.51; H, 6.01.

6: Yield: 70 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.19-1.60 (m, 86H), 1.70-1.92 (m, 10H), 4.01 (t, 6H), 4.29 (t, 2H), 4.46 (t, 2H), 4.78 (s, 1H), 7.25 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.10, 22.65, 25.89, 26.01, 26.09, 28.63, 28.72, 29.16, 29.19, 29.28, 29.33, 29.38, 29.53, 29.63, 29.69, 30.28, 31.87, 39.01, 64.95, 66.43, 69.07, 70.52, 73.39, 107.96, 124.95, 136.25, 140.43, 140.78, 140.98, 141.94, 142.07, 142.28, 142.68, 142.86, 142.96, 143.13, 143.59, 143.81, 144.28, 144.45, 144.53, 144.56, 144.61, 144.94, 145.03, 145.07, 145.12, 145.45, 145.50, 145.68, 148.15, 152.69, 166.27, 166.33. MALDI-TOF, Calc. for $C_{125}H_{118}O_7Na$: $(M•Na)^+$ 1753.878. Found: 1753.901. Anal. Calc. for $C_{125}H_{118}O_7$ (1732.27): C, 86.67; H, 6.87. Found: C, 86.27; H, 6.80.

8: Yield: 62 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.21-1.52 (m, 70H), 1.69-1.95 (m, 10H), 4.01 (t, 6H), 4.33 (t, 2H), 4.49 (t, 2H), 4.78 (s, 1H), 7.25 (s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 14.14, 22.71, 25.72, 25.85, 26.08, 26.15, 28.62, 28.76, 29.36, 29.39, 29.45, 29.60, 29.68, 29.69, 29.70, 29.74, 29.76, 30.35, 31.94, 39.11, 64.82, 66.35, 69.23, 70.58, 73.51, 76.81, 77.02, 77.23, 108.10, 124.94, 136.37, 140.43, 140.91, 141.11, 142.03, 142.06, 142.18, 142.40, 142.45, 142.79, 142.94, 142.96, 142.98, 143.07, 143.24, 143.71, 143.92, 144.40, 144.57, 144.65, 144.67, 145.04, 145.07, 145.16, 145.18, 145.25, 145.54, 145.7, 148.23, 152.81, 166.39, 166.45. MALDI-TOF, Calc. for $C_{117}H_{102}O_7Na$: $(M•Na)^+$ 1641.752. Found: 1642.257. Anal. Calc. for $C_{117}H_{102}O_7$ (1620.06): C, 86.74; H, 6.35. Found: C, 87.01; H, 6.36.

9: Yield: 63 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.19-1.52 (m, 78H), 1.69-1.91 (m, 10H), 4.01 (t, 6H), 4.28 (t, 2H), 4.47 (t, 2H), 4.79 (s, 1H), 7.24 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.12, 22.67, 25.98, 26.03, 26.09, 26.14, 28.67, 28.73, 29.19, 29.28, 29.35, 29.39, 29.45, 29.50, 29.55, 29.65, 29.69, 29.73, 29.75, 30.34, 31.94, 39.18,

65.15, 66.62, 69.19, 70.63, 73.50, 76.81, 77.01, 77.23, 108.05, 125.07, 136.35, 140.47, 140.91, 141.11, 142.07, 142.18, 142.34, 142.41, 142.79, 142.95, 143.00, 143.07, 143.25, 143.70, 143.93, 144.39, 144.57, 144.65, 144.71, 145.06, 145.15, 145.20, 145.24, 145.57, 145.83, 148.28, 152.78, 166.42, 166.48. MALDI-TOF, Calc. for $C_{121}H_{110}O_7Na$: $(M•Na)^+$ 1697.825. Found: 1697.804. Anal. Calc. for $C_{121}H_{110}O_7$ (1676.16): C, 86.70; H, 6.61. Found: C, 86.61; H, 6.48.

10: Yield: 60 %. ^1H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, 9H), 1.20-1.55 (m, 82H), 1.69-1.92 (m, 10H), 4.01 (t, 6H), 4.28 (t, 2H), 4.47 (t, 2H), 4.79 (s, 1H), 7.25 (s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 14.14, 22.71, 26.05, 26.08, 26.13, 28.72, 28.78, 29.31, 29.34, 29.39, 29.43, 29.59, 29.60, 29.67, 29.70, 29.74, 29.76, 29.77, 30.29, 31.90, 39.07, 65.08, 66.54, 69.10, 70.55, 73.44, 76.81, 77.02, 77.23, 107.94, 125.00, 136.29, 140.43, 140.83, 141.03, 141.93, 142.11, 142.22, 142.33, 142.40, 142.79, 142.94, 142.95, 143.24, 143.71, 143.92, 144.39, 144.57, 144.65, 144.67, 145.04, 145.06, 145.16, 145.19, 145.24, 145.55, 145.81, 148.20, 152.71, 166.37, 166.42. MALDI-TOF, Calc. for $C_{123}H_{114}O_7$ Na: $(M•Na)^+$ 1725.856. Found: 1725.861. Anal. Calc. for $C_{123}H_{114}O_7$ (1704.22): C, 86.69; H, 6.74. Found: C, 86.91; H, 6.53.

The characterization data of compound **C_{10}-C_8-OH, C_{12}-C_8-OH, C_{14}-C_8-OH** and dyads **2, 4, 5** and **7** have been reported previously.[5]

3. Supporting Table

Table S1. Phase behavior of C_n-C_m-C_{60} (**1-10**).

Sample [a]	*n* [b]	*m* [c]	Phase behavior [d]	Lamella thickness in S_mC (nm) [e]
1	9	8	S_mC 117.3 [8.4] I	5.28
2	10	8	Cr -41.5 [1.61] S_mC 104 [8.49] I	5.37
3	11	8	Cr -22.1 [6.40] S_mC 99.6 [8.60] I	5.46
4	12	8	Cr -4.8 [12.5] S_mC 87.6 [8.04] I	5.73
5	14	8	Cr 16.6 [36.2] S_mC 76.8 [8.34] I	6.16
6	16	8	Cr 40.6 [51.3] S_mC 68.6 [8.31] I	6.34
7	14	4	Cr 12.5 [9.10] I	-
8	14	6	Cr 14.5 [32.5] S_mC 61.1 [6.96] I	5.76
9	14	10	Cr 20 [38.1] S_mC 90.3 [16.74] I	8.09
10	14	12	Cr 23.3 [45.4] S_mC 81.5 [13.35] I	7.83

[a] The data of dyads **2**, **4**, **5** and **7** from reference[5]; [b] number of carbon atoms of each alkyl tail; [c] number of carbon atoms of flexible spacer; [d] transition temperatures determined by DSC: peak temperatures in the DSC thermograms during the second heating cycle (at 10 °C/min), transition enthalpies in kJ/mol in brackets; Cr = crystallite, S_mC = smectic C phase, I = isotropic liquid phase; [e] lamella thickness determined by SAXS.

4. Supporting Figures

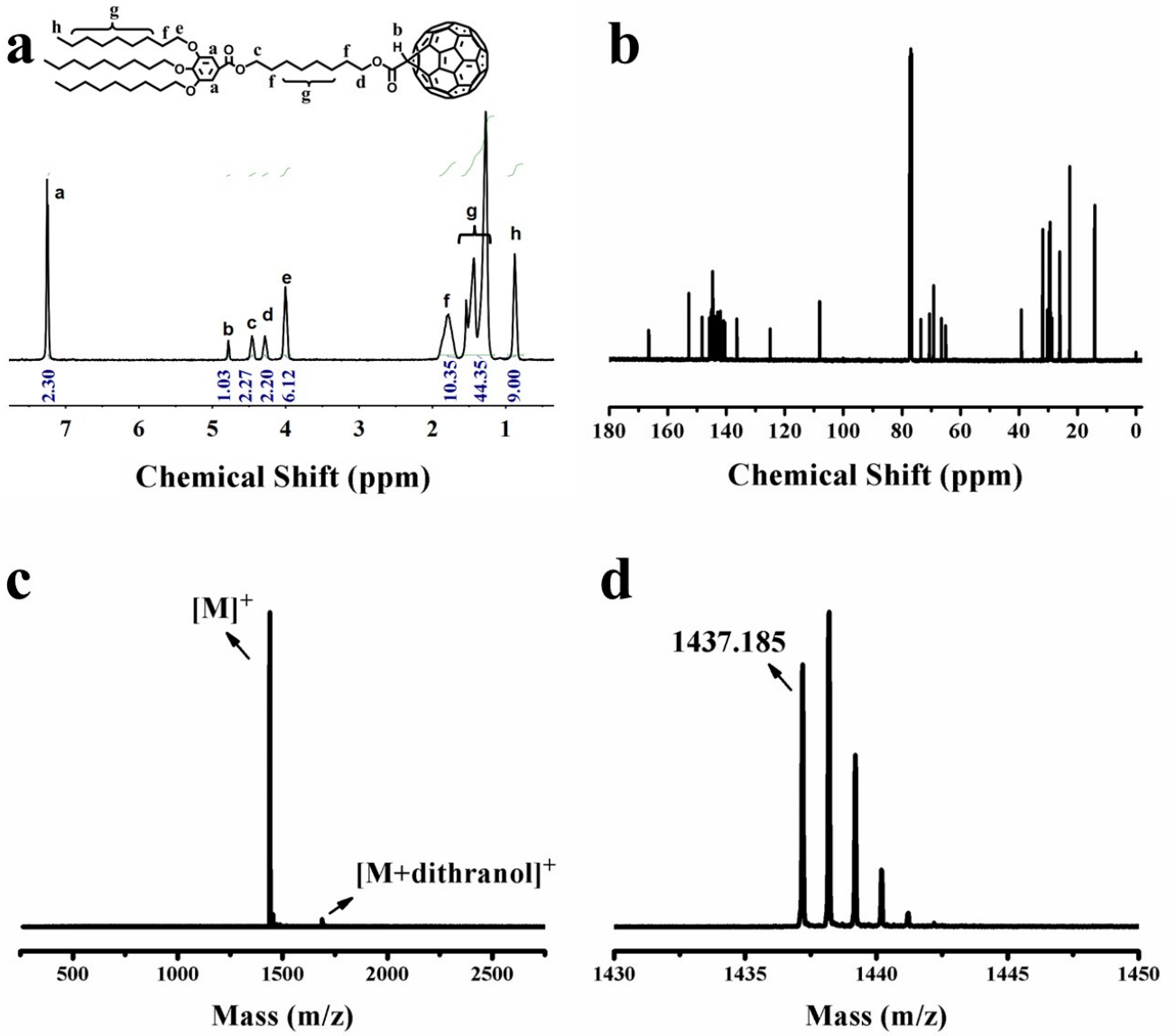


Fig. S1 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **1**, d) the zoom-in view of c.

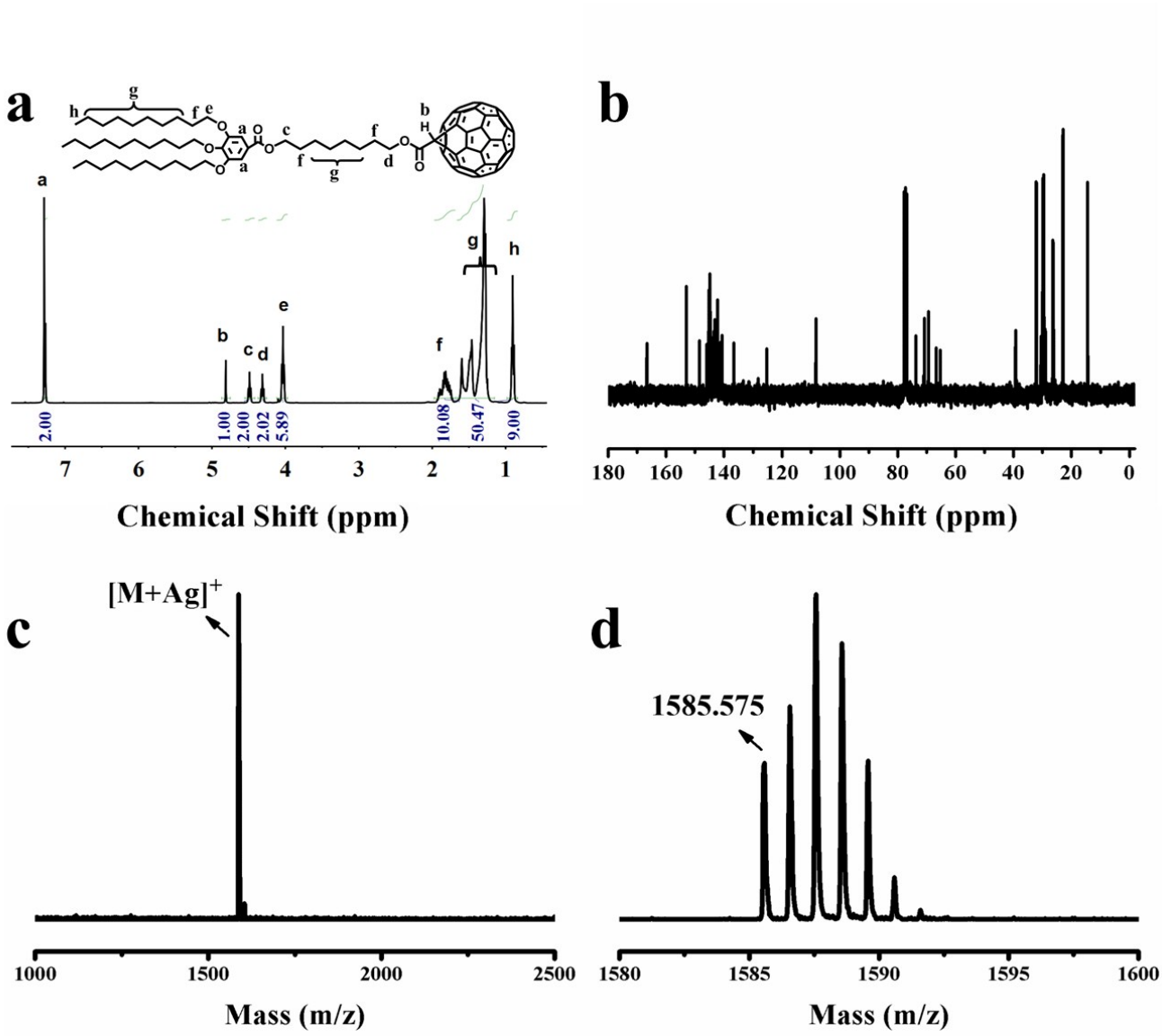


Fig. S2 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **2**, d) the zoom-in view of c.

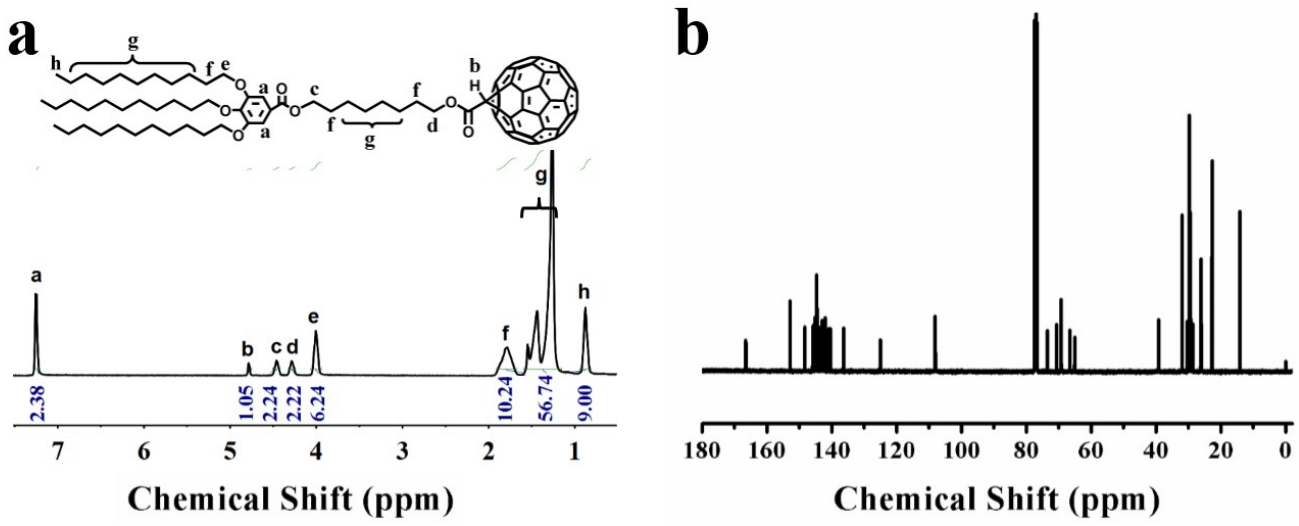


Fig. S3 a) ^{1}H NMR, b) ^{13}C NMR spectra of dyad **3**.

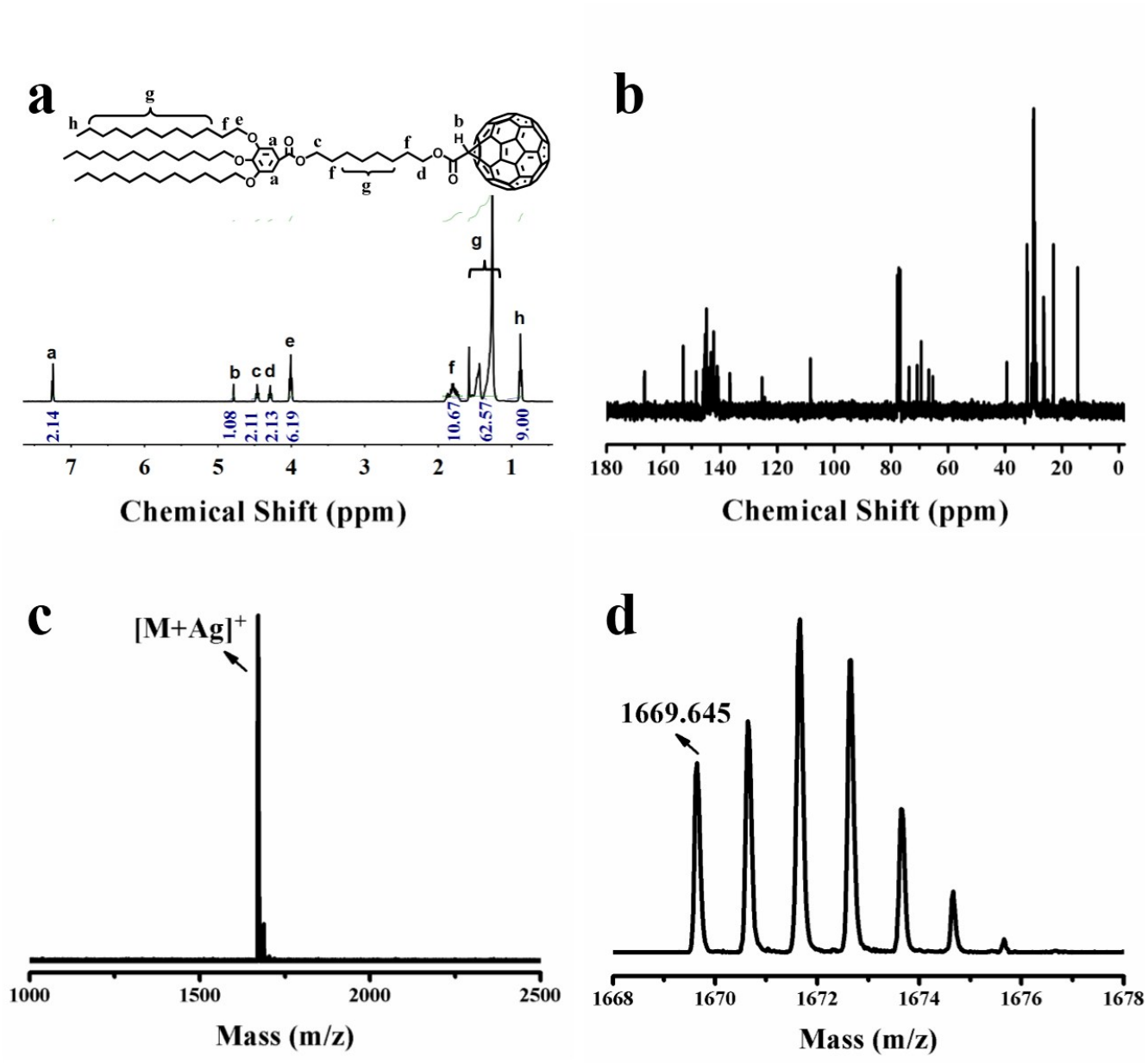


Fig. S4 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **4**, d) the zoom-in view of c.

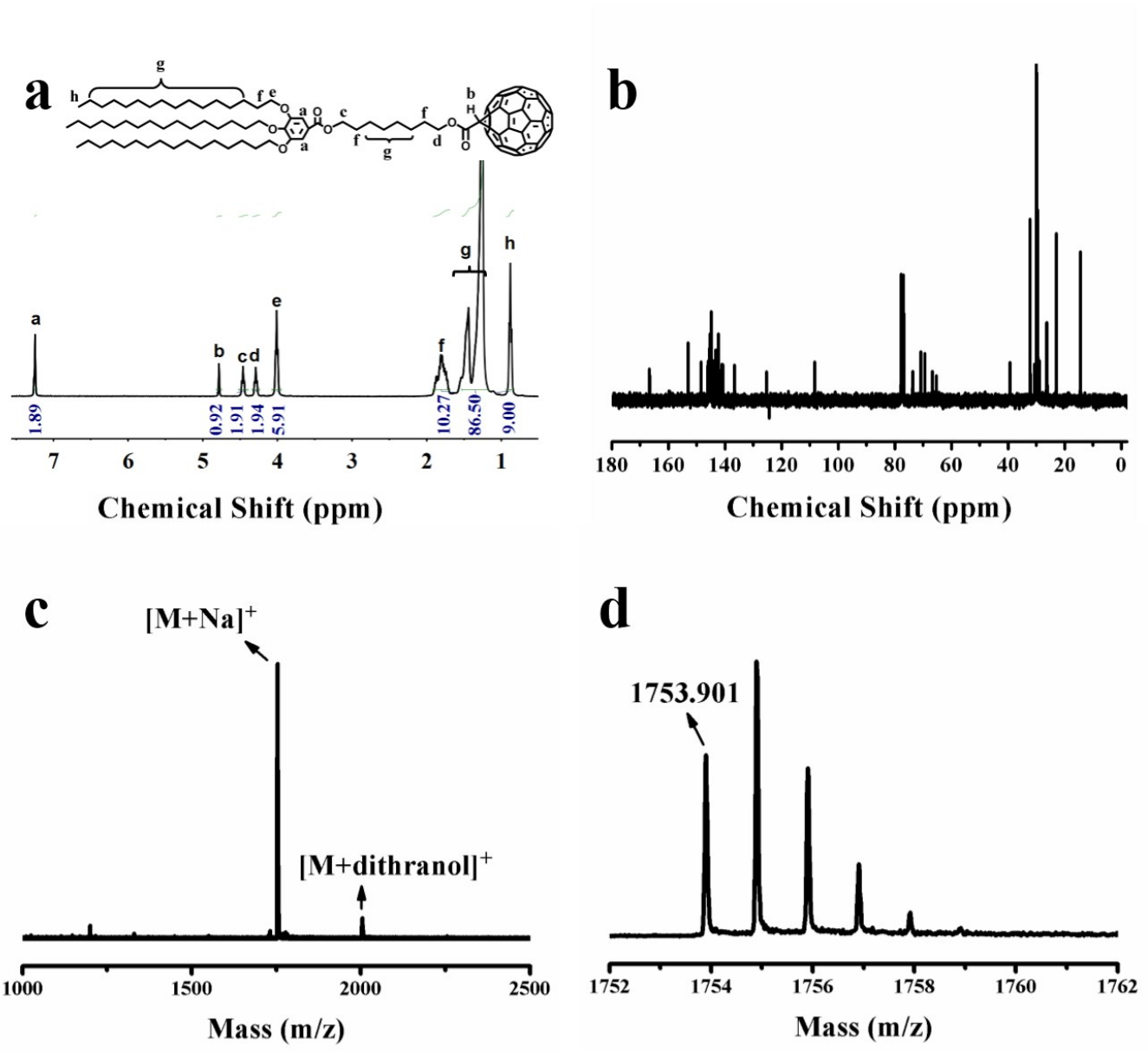


Fig. S5 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **6**, d) the zoom-in view of c.

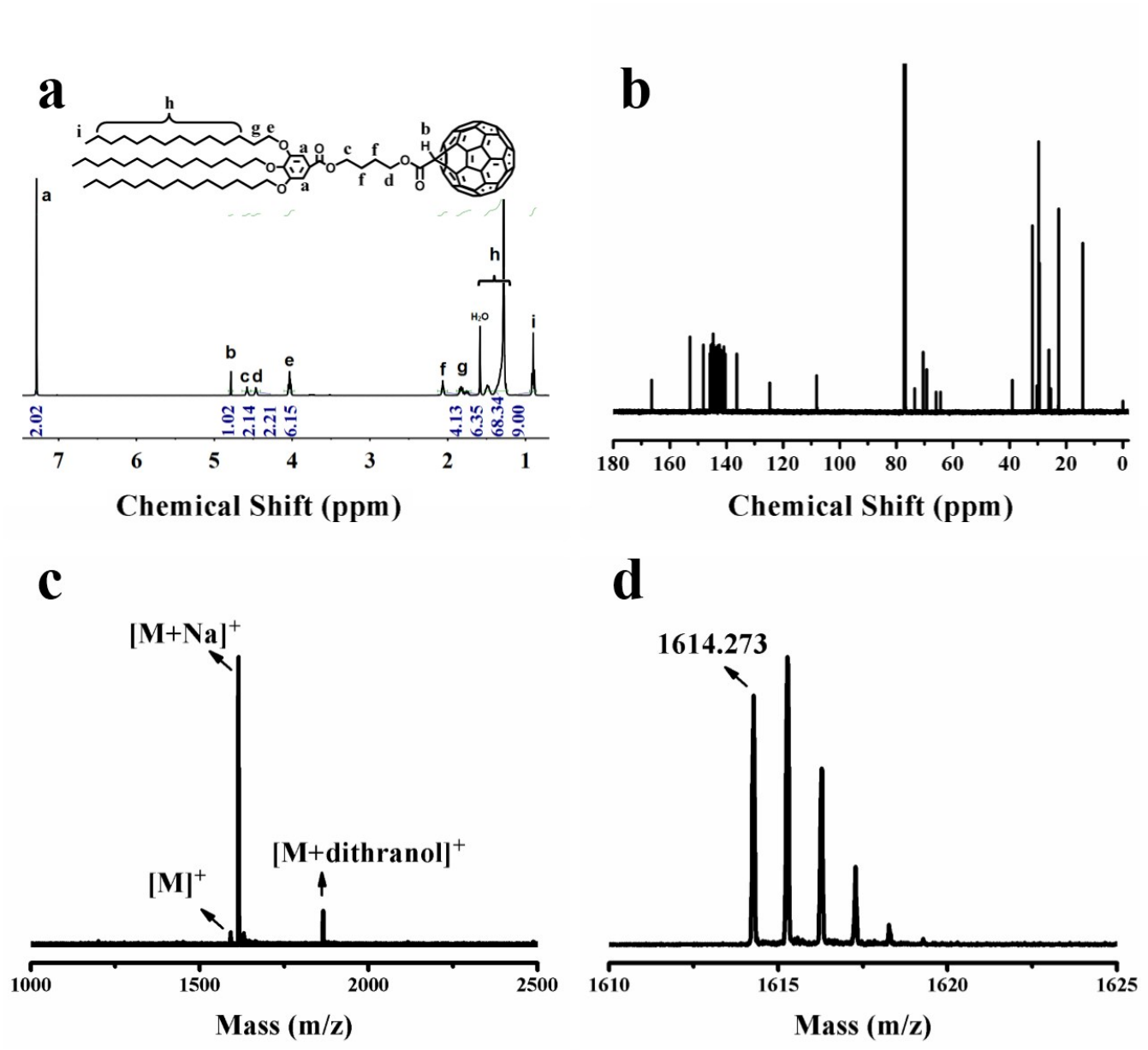


Fig. S6 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **7**, d) the zoom-in view of c.

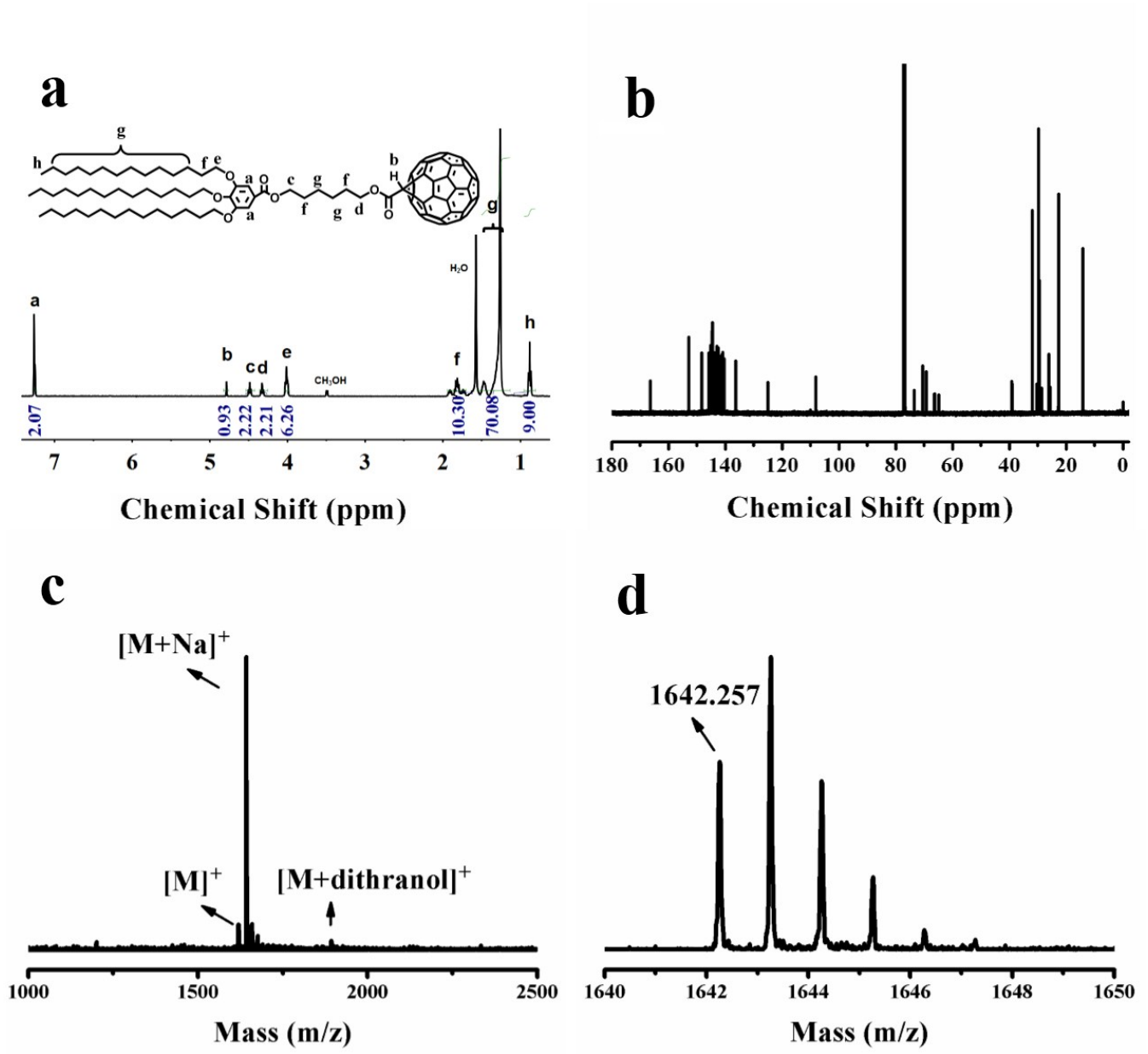


Fig. S7 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **8**, d) the zoom-in view of c.

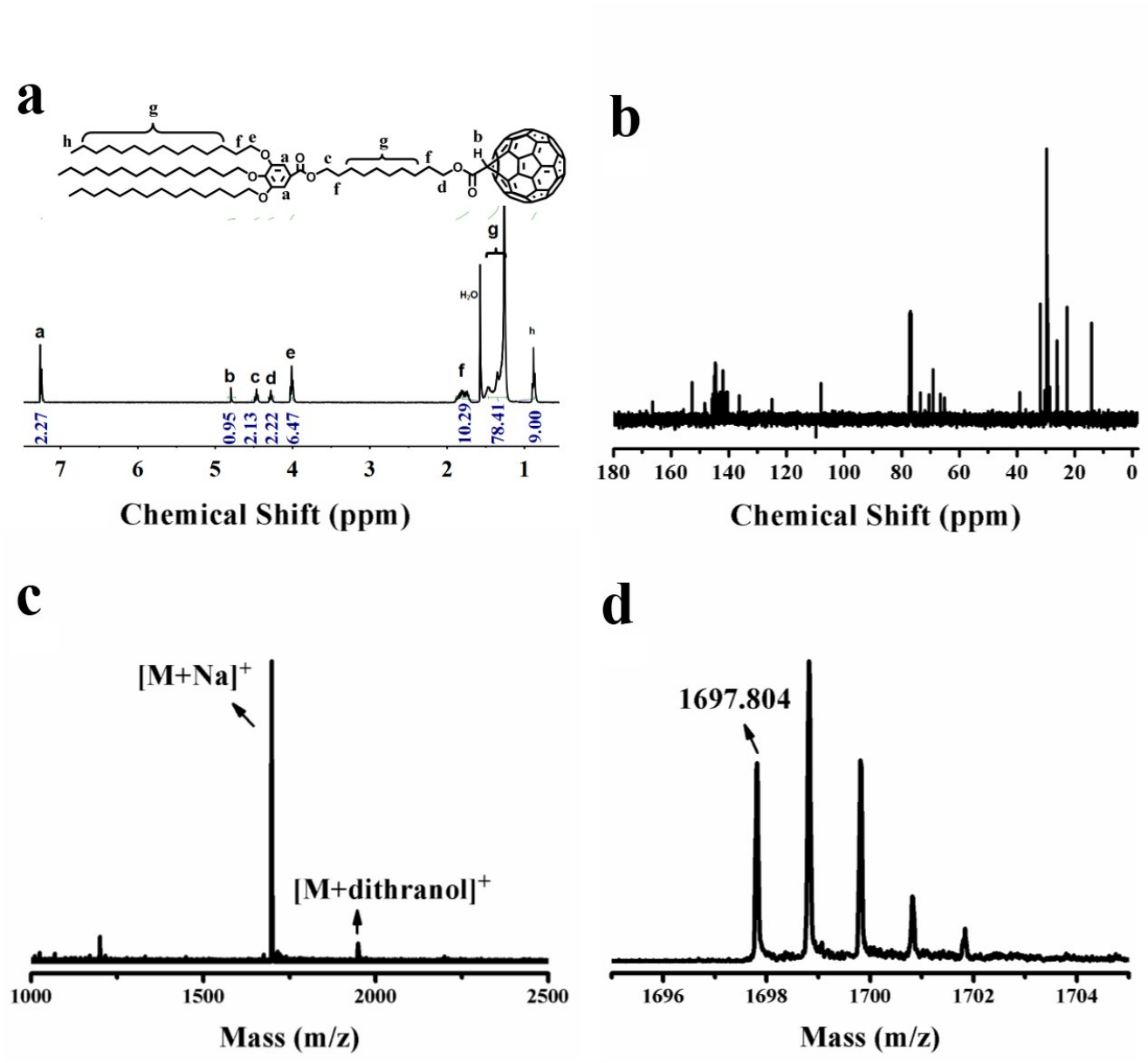


Fig. S8 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **9**, d) the zoom-in view of c.

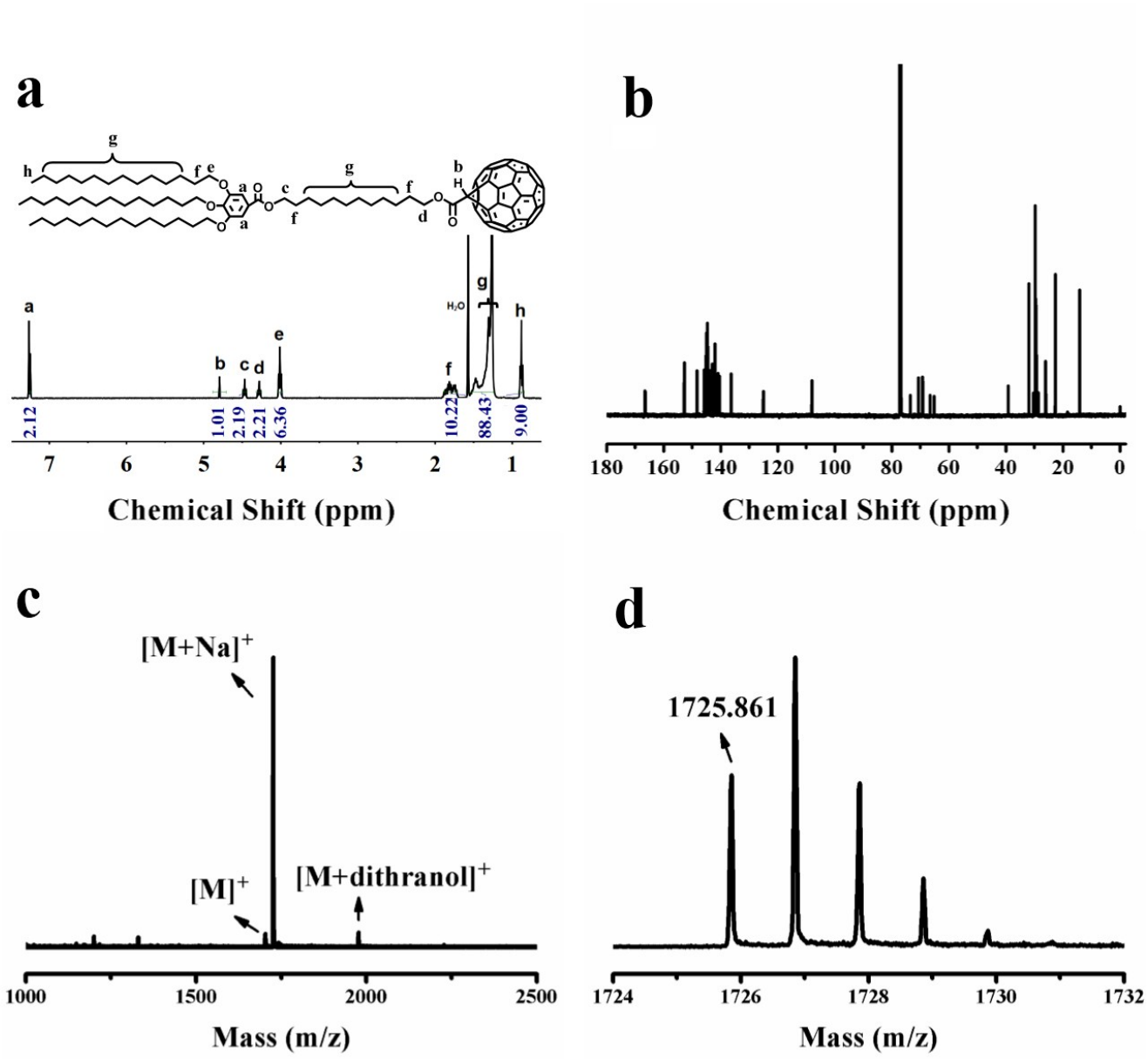


Fig. S9 a) ^{1}H NMR, b) ^{13}C NMR and c) MALDI-TOF mass spectra of dyad **10**, d) the zoom-in view of c.

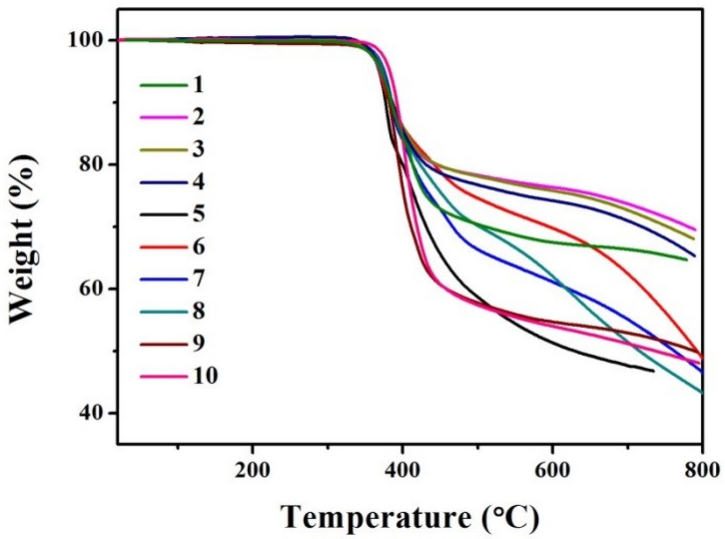


Fig. S10 TGA curves of dyads **1-10** at a heating rate of 10 °C/min under N_2.

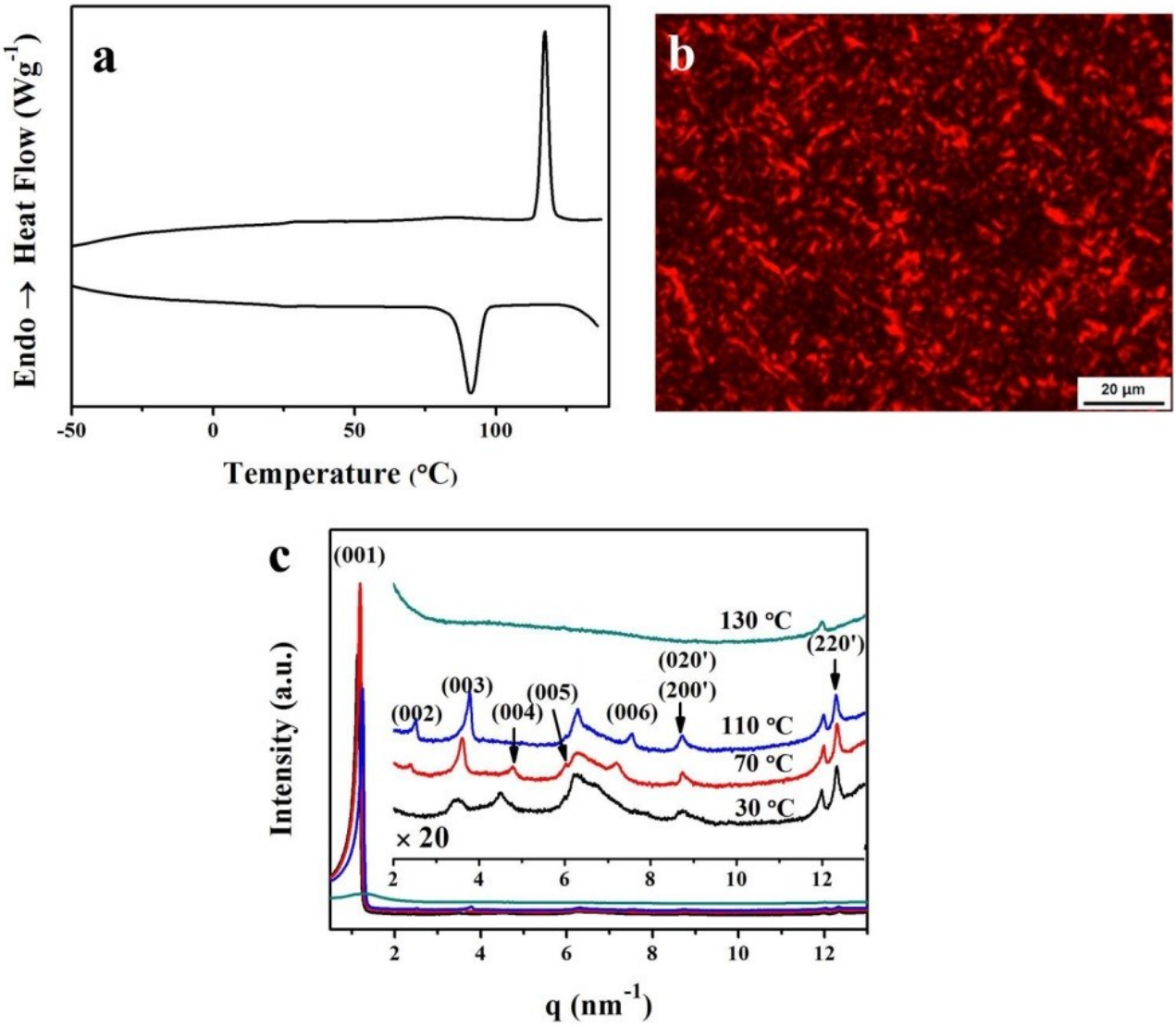


Fig. S11 a) DSC curve of dyad **1** at a scanning rate of 10 °C/min under N_2. b) POM picture of dyad **1** at 45 °C. c) Set of SAXS patterns of dyad **1** in the heating process after annealing at 100 °C for 12 h.

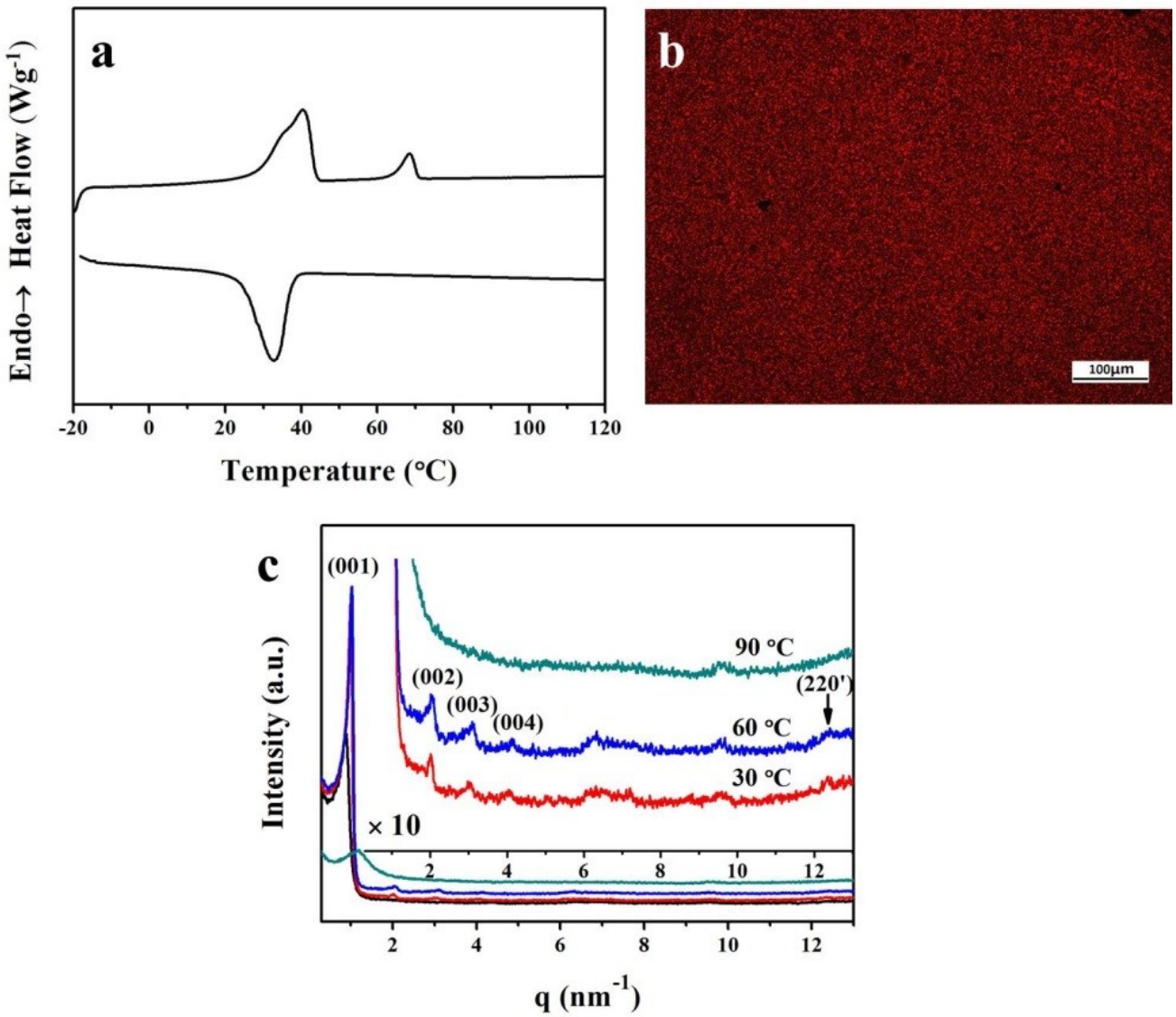


Fig. S12 a) DSC curve of dyad **6** at a scanning rate of 10 °C/min under N_2. b) POM picture of dyad **6** at 55 °C. c) Set of SAXS patterns of dyad **6** in the heating process after annealing at 50 °C for 12 h.

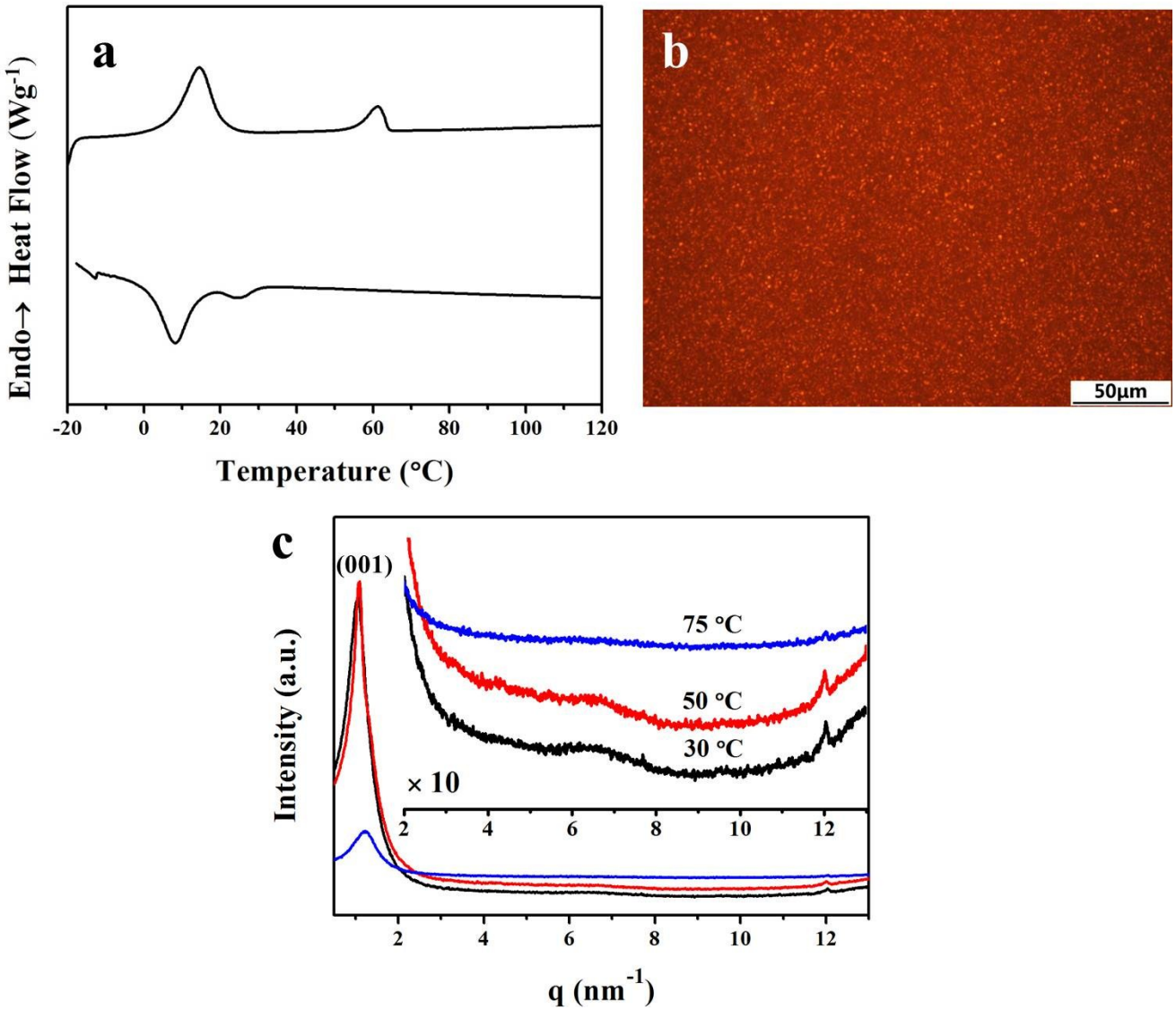


Fig. S13 a) DSC curve of dyad **8** at a scanning rate of 10 °C/min under N_2. b) POM picture of dyad **8** at 40 °C. c) Set of SAXS patterns of dyad **8** in the heating process after annealing at 45 °C for 12 h.

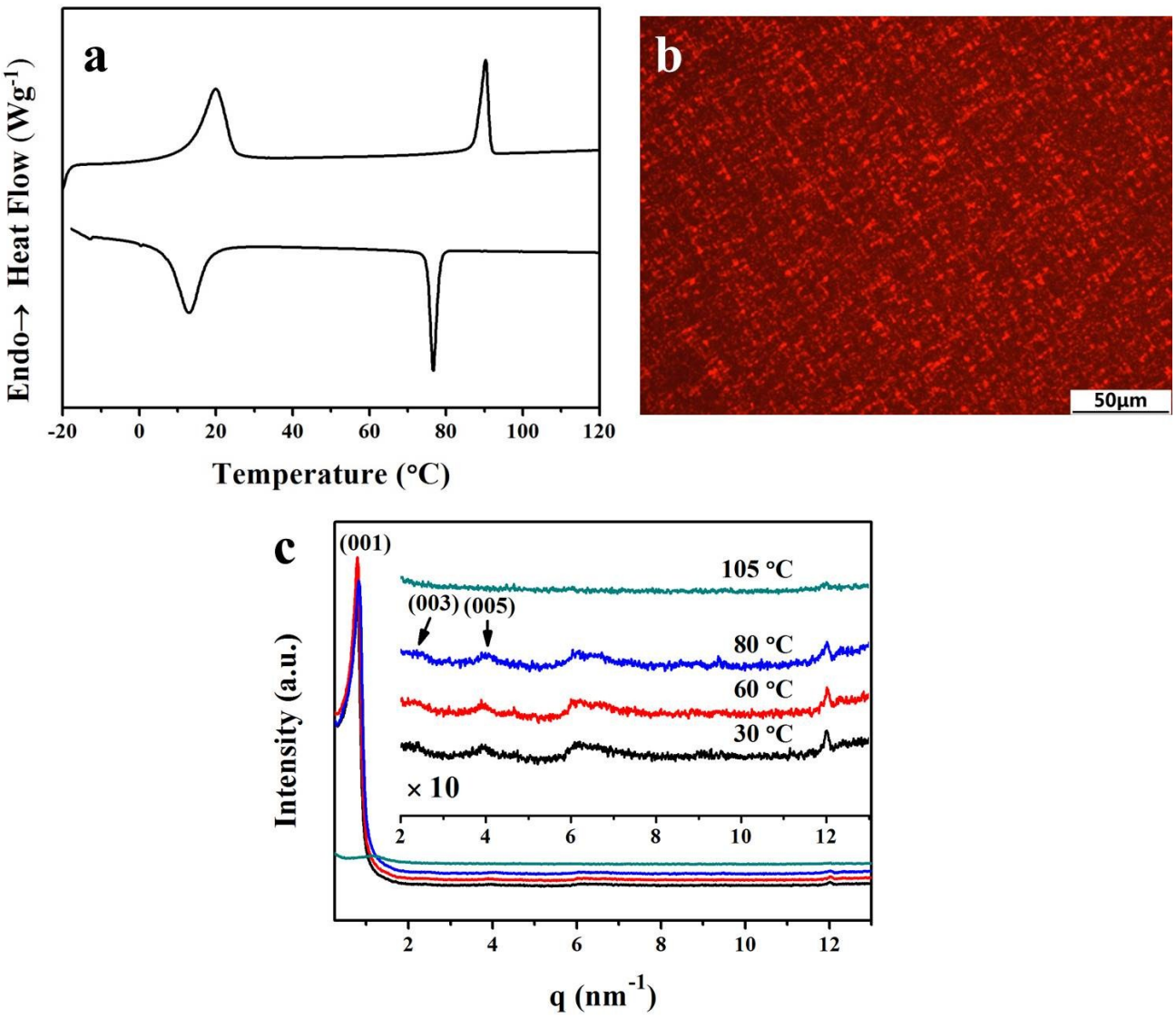


Fig. S14 a) DSC curve of dyad **9** at a scanning rate of 10 °C/min under N_2. b) POM picture of dyad **9** at 60 °C. c) Set of SAXS patterns of dyad **9** in the heating process after annealing at 80 °C for 12 h.

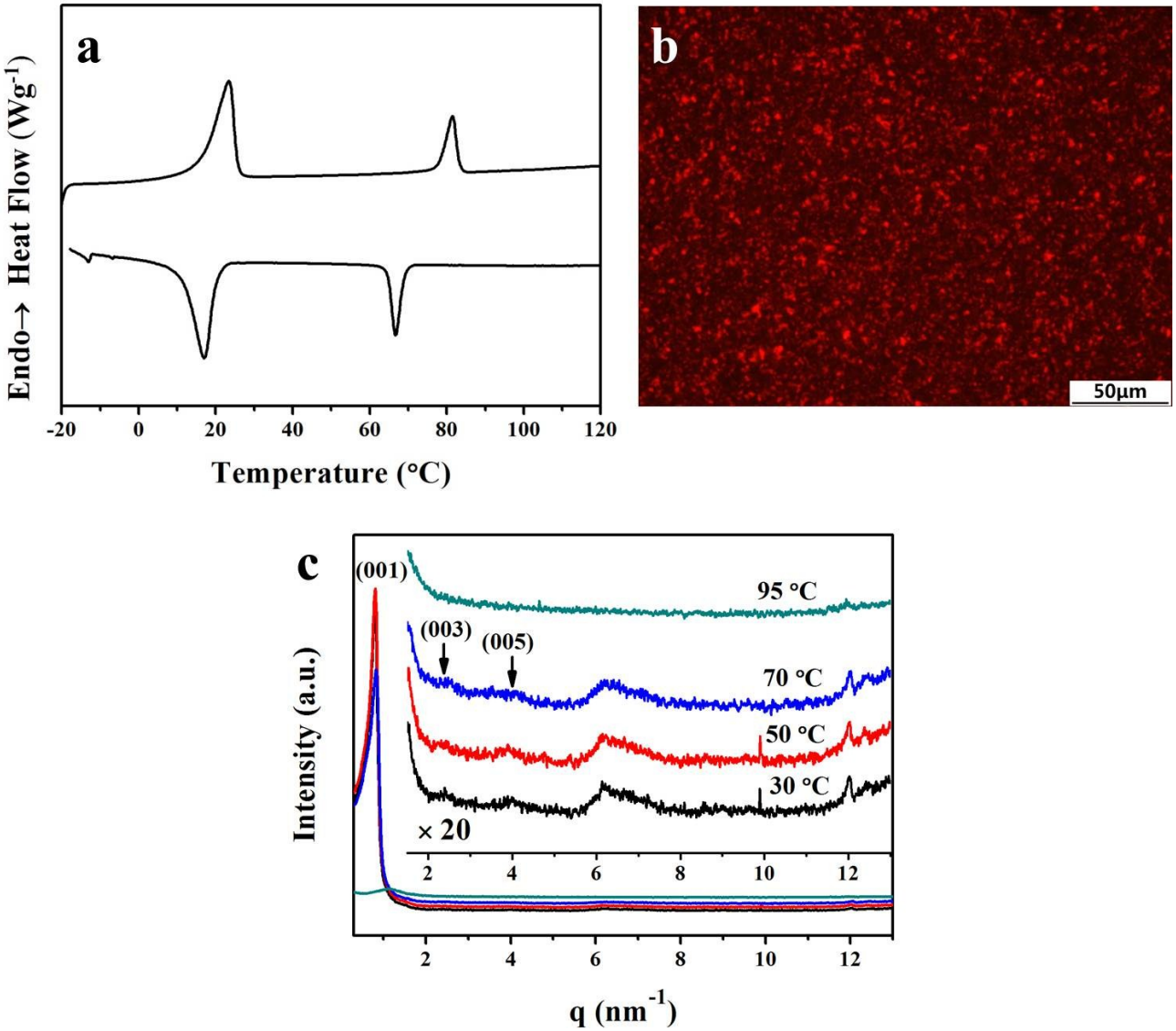


Fig. S15 a) DSC curve of dyad **10** at a scanning rate of 10 °C/min under N_2. b) POM picture of dyad **10** at 50 °C. c) Set of SAXS patterns of dyad **10** in the heating process after annealing at 70 °C for 12 h.

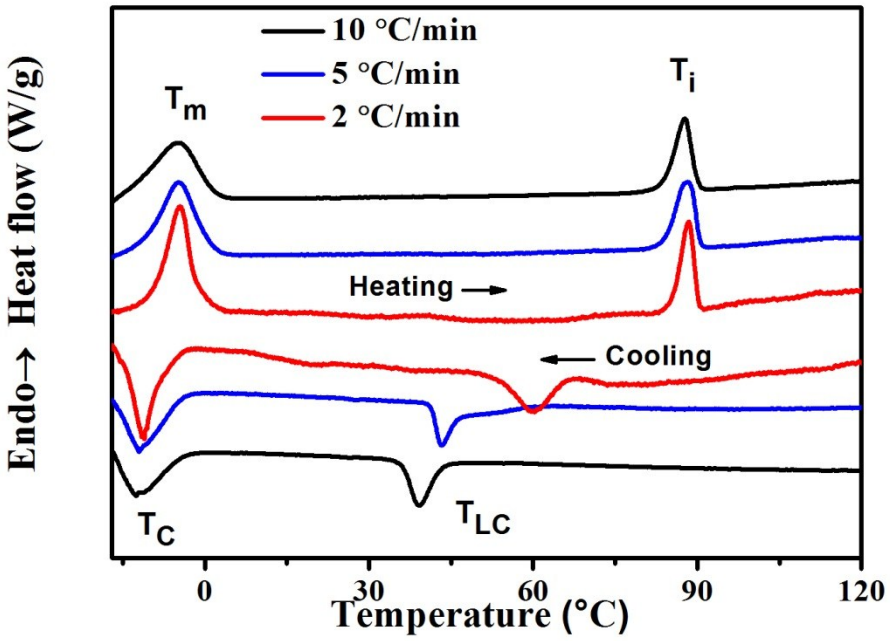


Fig. S16 DSC curves of dyad **4** at diffferent scanning rates.

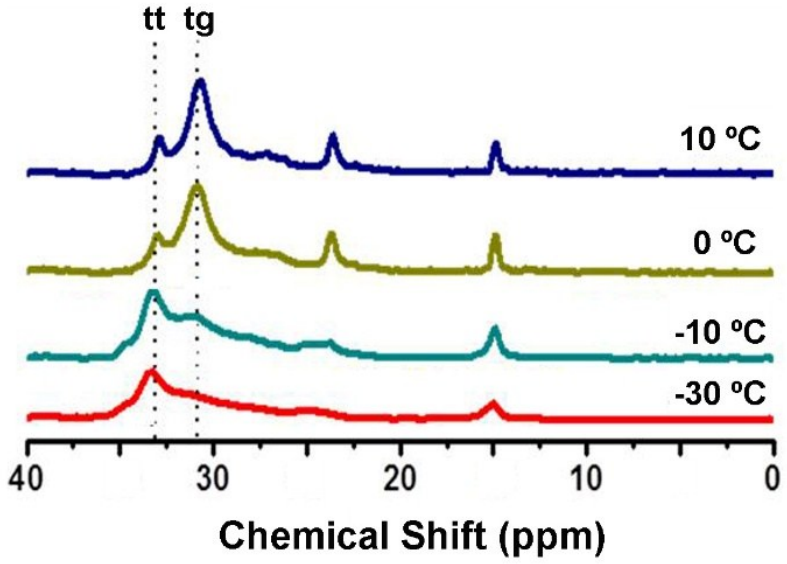


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Fig. S17 Bloch decay data of solid-state ^{13}C NMR spectra of dyad **4** during cooling from 10 °C to -30 °C.

5. References

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