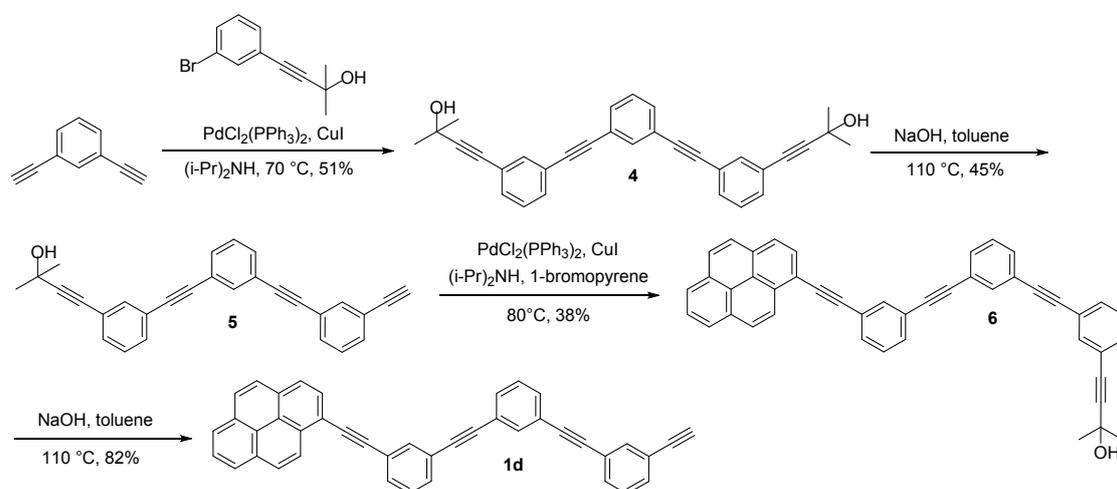


Materials and Instruments. Unless otherwise noted, all starting materials were obtained from commercial suppliers and used without further purification. All solvents were dried before use following standard procedures. Column chromatography was carried out with silica gel (300-400 mesh), and thin-layer chromatography (TLC) was performed on precoated silica gel plates (0.4-0.5 mm thick, GF254, Huanghai, China) and observed under UV light. The ^1H NMR spectra were recorded on 400 MHz and ^{13}C NMR were recorded on 100 MHz spectrometers in the indicated solvents at room temperature (298 K). Chemical shifts were reported in parts per million (δ) using residual solvent protons as the internal standard. High resolution mass spectra (ESI, EI, MALDI) were obtained on microTOF II (Bruker), Thermo Fisher Scientific LTQ FT Ultra and Waters Micromass GCT Premier spectrometers. Fluorescence spectra were recorded in a 10- or 1-mm quartz cell on a Cary Eclipse (VARIAN) spectrophotometer. UV-vis and Circular dichroism (CD) spectra were measured in a 1-cm quartz cell on a Lambda 750 S (Perkin Elmer) spectrophotometer and a MOS-450 spectropolarimeter, respectively. Scanning electron microscopy (SEM) was performed on Ultra55 (Zeiss) and Pro X (Phenom). Transmission electron microscopy (TEM) images were obtained on CM200FEG (Philips). Rheological characterisation of organogels was performed on HAAKE MARS III (Thermo Fisher, America).

Methods for the preparation of gels. A typical procedure for the gel formation is as follows: a known weight of the oligomer to be tested and a measured volume of the selected solvent were placed in a sealed test tube. The system was heated at 75 °C until the solid was completely dissolved, then the solution was allowed to cool down to room temperature under ultrasound aged for 1.5 min and finally a partly transparent gel was formed.



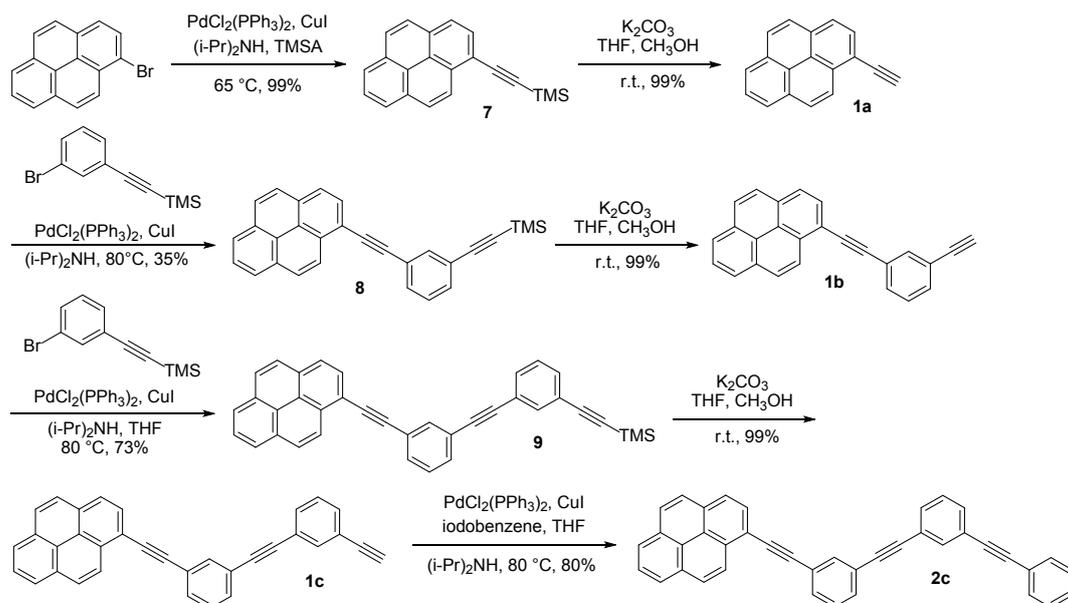
Compound 4. In a 50-mL three-necked round-bottomed flask, a mixture of 2-methyl-4-(3-bromophenyl)-3-butyn-2-ol (3.01 g, 12.6 mmol) and diisopropylamine (30 mL) was degassed and back-filled three times with N_2 . Bis(triphenylphosphine)palladium (II) dichloride (0.34 g, 0.5 mmol) and copper (I) iodide (0.05 g, 0.3 mmol) were then added to the flask. The mixture was subjected to more vacuum/ N_2 cycles. After stirring for 30 minutes, 1,3-diethynylbenzene (0.78 mL, 6.0 mmol) was introduced into the flask via a syringe. The resulting mixture was stirred at 70 °C for 18 hours and filtered. The filtrate

was concentrated by rotary evaporation and the residue was purified by column chromatography (ethyl acetate/hexane = 1/5) to give **4** as a white solid (1.34 g, 51%). Mp. 148°C. ¹H NMR (400 MHz, CDCl₃): δ 7.69 (t, *J* = 1.2Hz, 1H), 7.61 (t, *J* = 1.2Hz, 2H), 7.49-7.45 (m, 4H), 7.39 (dt, *J* = 7.6, 1.2Hz, 2H), 7.36-7.28 (m, 3H), 1.63 (s, 12H), 1.61 (s, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 134.68, 134.58, 131.46, 131.42, 131.26, 128.50, 128.38, 123.34, 123.18, 123.09, 94.46, 89.10, 88.98, 81.24, 65.55, 31.38. HRMS (ESI): *m/z* Calcd for C₃₂H₂₆NaO₂ [M+Na]⁺: 465.1830. Found: 465.1814.

Compound 5. To a solution of **4** (0.22 g, 0.5mmol) in toluene (10 mL) was added ground NaOH (0.02 g, 0.5 mmol) and the mixture was heated at 110 °C. After being stirred for 3 hours, the reaction mixture was filtered and evaporated. The residue was chromatographed on silica gel (ethyl acetate/hexane = 1/20 to 1/1) to afford the titled compound **5** as a yellow solid (0.09 g, 45%). Mp. 146-148 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.69 (t, *J* = 1.2Hz, 1H), 7.67 (t, *J* = 1.2Hz, 1H), 7.61 (t, *J* = 1.2Hz, 1H), 7.52-7.45 (m, 5H), 7.39 (dt, *J* = 8, 1.2Hz, 1H), 7.37-7.28 (m, 3H), 3.10 (s, 1H), 1.65 (s, 1H), 1.63 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 135.12, 134.72, 134.64, 131.98, 131.86, 131.50, 131.31, 128.54, 128.47, 128.41, 123.39, 123.33, 123.23, 123.12, 122.51, 94.43, 89.13, 88.99, 88.95, 82.69, 81.29, 77.87, 65.60, 31.42. HRMS (ESI): *m/z* Calcd for C₂₉H₂₀NaO [M+Na]⁺: 407.1412. Found: 407.1398.

Compound 6. In a 100-mL three-necked round-bottomed flask, a mixture of **5** (0.48 g, 1.3 mmol), 1-bromopyrene (0.37 g, 1.3 mmol) and diisopropylamine (50 mL) was degassed and back-filled three times with N₂. Bis(triphenylphosphine)palladium(II) dichloride (0.04 g, 0.05 mmol) and copper(I) iodide (0.006 g, 0.03 mmol) were then added to the flask. The mixture was subjected to more vacuum/N₂cycles and stirred at 80 °C for 48 hours and filtered. The filtrate was concentrated by rotary evaporation and the residue was purified by column chromatography (ethyl acetate/hexane = 1/8 to 1/4) to give **6** as a yellow solid (0.28 g, 38%). Mp. 156-158 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.68 (d, *J* = 9.2Hz, 1H), 8.26-8.03 (m, 8H), 7.92 (t, *J* = 1.2Hz, 1H), 7.75 (t, *J* = 1.2Hz, 1H), 7.70 (dt, *J* = 7.6, 1.2Hz, 1H), 7.62 (t, *J* = 1.2Hz, 1H), 7.57-7.35 (m, 7H), 7.31 (t, *J* = 8Hz, 1H), 1.63 (s, 6H), 1.62 (s, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 134.74, 134.70, 132.00, 131.51, 131.42, 131.33, 131.25, 131.07, 129.65, 128.65, 128.56, 128.45, 128.42, 128.28, 127.23, 126.26, 125.71, 125.65, 125.46, 124.53, 124.31, 123.98, 123.49, 123.45, 123.28, 123.15, 117.43, 94.46, 94.18, 89.38, 89.27, 89.16, 89.05, 81.31, 65.60, 31.44. HRMS (ESI): *m/z* Calcd for C₄₅H₂₈NaO [M+Na]⁺: 607.2038. Found: 607.2045.

Compound 1d. The titled compound was prepared according to the procedure similar to that described for the preparation of **5** except that **6** (0.27 g, 0.5mmol) was used in place of **4** to give **1d** as a yellow solid (0.20 g, 82%). Mp. 154-156 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.67 (d, *J* = 9.2 Hz, 1H), 8.26-8.02 (m, 8H), 7.92 (s, 1H), 7.76 (s, 1H), 7.71-7.69 (m, 2H), 7.57-7.31 (m, 8H), 3.11 (s, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 135.15, 134.71, 131.99, 131.88, 131.55, 131.42, 131.25, 131.08, 129.65, 128.64, 128.56, 128.47, 128.27, 127.23, 126.26, 125.70, 125.64, 125.47, 124.53, 124.32, 123.99, 123.49, 123.39, 122.55, 117.44, 94.18, 89.38, 89.28, 89.18, 88.99, 82.72, 77.86. HRMS (MALDI-DHB): *m/z* Calcd for C₄₂H₂₂ [M]⁺: 526.1722. Found: 526.1715.



Compound 7. In a 250-mL three-necked round-bottomed flask, a mixture of 1-bromopyrene (3.93 g, 14.0 mmol) and diisopropylamine (180 mL) was degassed and back-filled three times with N_2 . Bis(triphenylphosphine)palladium(II) dichloride (0.49 g, 0.7 mmol) and copper(I) iodide (0.13 g, 0.7 mmol) were then added to the flask. The mixture was subjected to more vacuum/ N_2 cycles. After stirring for 30 minutes, trimethylsilylacetylene (20.00 mL, 0.14 mol) was introduced into the flask via a syringe. The resulting mixture was stirred at room temperature for 24 hours and filtered. The filtrate was concentrated by rotary evaporation and the residue was purified by column using hexane as the eluent to give **7** as a yellow solid (4.50 g, 99%). ^1H NMR (400 MHz, CDCl_3): δ 8.57 (d, $J = 8\text{ Hz}$, 1H), 8.24-8.01 (m, 8H), 0.40 (s, 9H). HRMS (EI): m/z Calcd for $\text{C}_{21}\text{H}_{18}\text{Si}$ $[\text{M}]^+$: 298.1178. Found: 298.1173.

Compound 1a. To a solution of **7** (2.98 g, 10.0 mmol) in tetrahydrofuran (120 mL) and methanol (50 mL) was added K_2CO_3 (4.14 g, 30.0 mmol). The resulting mixture was filtered and evaporated after being stirred at room temperature for 12 hours. The residue was dissolved in dichloromethane (150 mL) and the solution was washed with H_2O (2×50 mL), brine (50 mL) and finally dried over anhydrous Na_2SO_4 to afford the titled compound **1a** as a white solid (2.22 g, 99%). ^1H NMR (400 MHz, CDCl_3): δ 8.60 (d, $J = 8\text{ Hz}$, 1H), 8.25-8.02 (m, 8H), 3.63 (s, 1H). HRMS (EI): m/z Calcd for $\text{C}_{18}\text{H}_{10}$ $[\text{M}]^+$: 226.0783. Found: 226.0787.

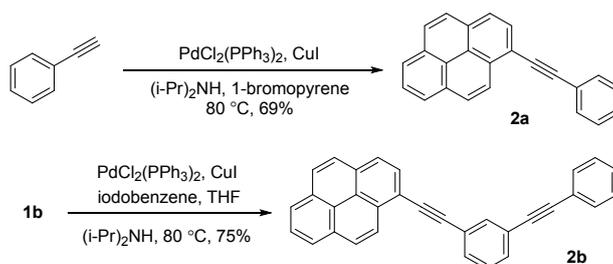
Compound 8. The titled compound was prepared according to the procedure similar to that described for the preparation of **6** except that **1a** (7.81 g, 34.5 mmol) and 1-bromo-3-trimethylsilylethynylbenzene (9.17 g, 36.2 mmol) were used in place of **5** and 1-bromopyrene, respectively, to give **8** as a bright yellow solid (4.82 g, 35%). ^1H NMR (400 MHz, CDCl_3): δ 8.65 (d, $J = 9.2\text{ Hz}$, 1H), 8.26-8.03 (m, 8H), 7.84 (t, $J = 1.6\text{ Hz}$, 1H), 7.66 (dt, $J = 8, 1.2\text{ Hz}$, 1H), 7.49 (dt, $J = 7.6, 1.6\text{ Hz}$, 1H), 7.37 (t, $J = 8\text{ Hz}$, 1H), 0.29 (s, 9H). HRMS (EI): m/z Calcd for $\text{C}_{29}\text{H}_{22}\text{Si}$ $[\text{M}]^+$: 398.1491. Found: 398.1487.

Compound 1b. The titled compound was prepared according to the procedure similar to that described for the preparation of **1a** except that **8** (1.00 g, 2.5 mmol) was used in place of **7** to give **1b** as a yellow solid (0.81 g, 99%). ¹H NMR (400 MHz, CDCl₃): δ 8.65 (d, *J* = 9.2 Hz, 1H), 8.25-8.02 (m, 8H), 7.86 (t, *J* = 1.6 Hz, 1H), 7.70 (dt, *J* = 8, 1.2 Hz, 1H), 7.52 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 3.15 (s, 1H). HRMS (EI): *m/z* Calcd for C₂₆H₁₄ [M]⁺: 326.1096. Found: 326.1102.

Compound 9. In a 250-mL three-necked round-bottomed flask, a mixture of **1b** (4.19 g, 12.8 mmol), 1-bromo-3-trimethylsilylethynylbenzene (3.41 g, 13.5 mmol), diisopropylamine (140 mL) and tetrahydrofuran (20 mL) was degassed and back-filled three times with N₂. Bis(triphenylphosphine)palladium(II) dichloride (0.45 g, 0.6 mmol) and copper(I) iodide (0.12 g, 0.6 mmol) were then added to the flask. The mixture was subjected to more vacuum/N₂ cycles and stirred at 80 °C for 48 hours and filtered. The filtrate was concentrated by rotary evaporation and the residue was purified by column chromatography (ethyl acetate/hexane = 1/20 to 1/12) to give **9** as a bright yellow solid (4.67 g, 73%). Mp. 141 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.66 (d, *J* = 9.2 Hz, 1H), 8.25-8.02 (m, 8H), 7.90 (t, *J* = 1.2 Hz, 1H), 7.72 (t, *J* = 1.6 Hz, 1H), 7.70 (dt, *J* = 8, 1.6 Hz, 1H), 7.55-7.51 (m, 2H), 7.46 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 0.29 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ 135.10, 134.64, 131.92, 131.76, 131.52, 131.48, 131.35, 131.17, 130.99, 129.58, 128.60, 128.38, 128.21, 127.17, 126.20, 125.65, 125.59, 125.39, 124.47, 124.40, 124.22, 123.93, 123.53, 123.46, 123.24, 117.36, 104.09, 95.08, 94.15, 89.37, 89.16, 0.08. HRMS (EI): *m/z* Calcd for C₃₇H₂₆Si [M]⁺: 498.1804. Found: 498.1809.

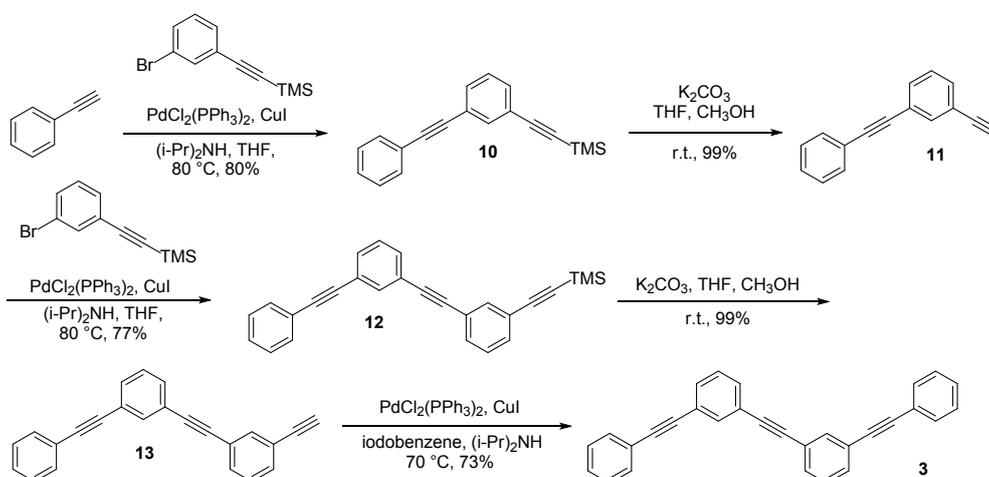
Compound 1c. The titled compound was prepared according to the procedure similar to that described for the preparation of **1a** except that **9** (3.49 g, 7.0 mmol) was used in place of **7** to give **1c** as a yellow solid (2.95 g, 99%). Mp. 139-141 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.67 (d, *J* = 9.2 Hz, 1H), 8.26-8.03 (m, 8H), 7.91 (t, *J* = 1.2 Hz, 1H), 7.71-7.69 (m, 2H), 7.56-7.54 (m, 2H), 7.48 (dt, *J* = 8, 1.6 Hz, 1H), 7.43 (t, *J* = 8 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 3.12 (s, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 135.17, 134.67, 131.99, 131.90, 131.56, 131.41, 131.23, 131.05, 129.64, 128.64, 128.49, 128.44, 128.27, 127.22, 126.26, 125.70, 125.64, 125.44, 124.53, 124.47, 124.29, 123.96, 123.41, 122.54, 117.40, 94.15, 89.38, 89.29, 89.01, 82.73, 77.88. HRMS (EI): *m/z* Calcd for C₃₄H₁₈ [M]⁺: 426.1409. Found: 426.1414.

Compound 2c. The titled compound was prepared according to the procedure similar to that described for the preparation of **9** except that **1c** (0.85 g, 2.0 mmol) and iodobenzene (0.42 g, 2.0 mmol) were used in place of **1b** and 1-bromo-3-trimethylsilylethynylbenzene, respectively, to give **2c** as a yellow solid (0.80 g, 80%). Mp. 154-155 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.67 (d, *J* = 9.2 Hz, 1H), 8.25-8.02 (m, 8H), 7.92 (t, *J* = 1.6 Hz, 1H), 7.78 (t, *J* = 1.6 Hz, 1H), 7.70 (dt, *J* = 8, 1.6 Hz, 1H), 7.57-7.52 (m, 5H), 7.44 (t, *J* = 8 Hz, 1H), 7.39-7.35 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 134.66, 131.92, 131.65, 131.47, 131.36, 131.31, 131.17, 130.99, 129.58, 128.60, 128.50, 128.44, 128.37, 128.20, 127.17, 126.19, 125.64, 125.58, 125.39, 124.47, 124.40, 124.22, 123.94, 123.69, 123.48, 123.36, 122.97, 117.36, 94.16, 90.08, 89.39, 89.25, 89.20, 88.51. HRMS (EI): *m/z* Calcd for C₄₀H₂₂ [M]⁺: 502.1722. Found: 502.1718.



Compound 2a. The titled compound was prepared according to the procedure similar to that described for the preparation of **6** except that ethynylbenzene (0.40 mL, 3.5 mmol) was used in place of **5** to give **2a** as a light green solid (0.73 g, 69%). ^1H NMR (400 MHz, CDCl_3): δ 8.68 (d, $J = 9.2$ Hz, 1H), 8.25-8.02 (m, 8H), 7.75-7.73 (m, 2H), 7.47-7.39 (m, 3H). HRMS (EI): m/z Calcd for $\text{C}_{24}\text{H}_{14}$ $[\text{M}]^+$: 302.1096. Found: 302.1091.

Compound 2b. The titled compound was prepared according to the procedure similar to that described for the preparation of **9** except that iodobenzene (0.51 g, 2.5 mmol) was used in place of 1-bromo-3-trimethylsilylethynylbenzene to give **2b** as a yellow solid (0.75 g, 75%). Mp. $136\text{-}137\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3): δ 8.67 (d, $J = 9.2$ Hz, 1H), 8.26-8.03 (m, 8H), 7.92 (t, $J = 1.6$ Hz, 1H), 7.69 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.60-7.55 (m, 3H), 7.43 (t, $J = 8$ Hz, 1H), 7.40-7.37 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 134.64, 131.98, 131.69, 131.39, 131.34, 131.24, 131.07, 129.64, 128.60, 128.47, 128.41, 128.25, 127.23, 126.25, 125.69, 125.62, 125.47, 124.52, 124.31, 123.91, 123.79, 123.04, 117.47, 94.26, 90.10, 89.28, 88.60. HRMS (EI): m/z Calcd for $\text{C}_{32}\text{H}_{18}$ $[\text{M}]^+$: 402.1409. Found: 402.1406.



Compound 10. The titled compound was prepared according to the procedure similar to that described for the preparation of **6** except that phenylacetylene (6.13 g, 60.0 mmol) and 1-bromo-3-trimethylsilylethynylbenzene (15.19 g, 60.0 mmol) were used in place of **5** and 1-bromopyrene, respectively, to give **10** as a colourless oil (13.18 g, 80%). ^1H NMR (400 MHz, CDCl_3): δ 7.66 (s, 1H), 7.53-7.51 (m, 2H), 7.47 (dt, $J = 8, 1.5$ Hz, 1H), 7.42 (dt, $J = 8, 1.6$ Hz, 1H), 7.36-7.34 (m, 3H), 7.28 (t, $J = 8$ Hz, 1H), 0.26 (s, 9H). HRMS (EI): m/z Calcd for $\text{C}_{19}\text{H}_{18}\text{Si}$ $[\text{M}]^+$: 274.1178. Found: 274.1179.

Compound 11. The titled compound was prepared according to the procedure similar to that described for the preparation of **1a** except that **10** (10.97 g, 40.0 mmol) was used in place of **7** to give **11** as a colourless oil (8.09 g, 99%). ¹H NMR (400 MHz, CDCl₃): δ 7.68 (s, 1H), 7.55-7.45 (m, 4H), 7.37-7.29 (m, 4H), 3.10 (s, 1H). HRMS (EI): *m/z* Calcd for C₁₆H₁₀ [M]⁺: 202.0783. Found: 202.0782.

Compound 12. The titled compound was prepared according to the procedure similar to that described for the preparation of **6** except that **11** (8.05 g, 39.8 mmol) and 1-bromo-3-trimethylsilylethynylbenzene (10.07 g, 39.8 mmol) were used in place of **5** and 1-bromopyrene, respectively, to give **12** as a white solid (11.47 g, 77%). ¹H NMR (400 MHz, CDCl₃): δ 7.72 (s, 1H), 7.67 (s, 1H), 7.55-7.42 (m, 6H), 7.38-7.28 (m, 5H), 0.26 (s, 9H). HRMS (EI): *m/z* Calcd for C₂₇H₁₂Si [M]⁺: 374.1491. Found: 374.1486.

Compound 13. The titled compound was prepared according to the procedure similar to that described for the preparation of **4** except that **12** (7.13 g, 19.0 mmol) was used in place of **7** to give **13** as a white solid (5.75 g, 99%). ¹H NMR (400 MHz, CDCl₃): δ 7.70 (s, 1H), 7.66 (s, 1H), 7.56-7.46 (m, 6H), 7.38-7.30 (m, 5H), 3.11 (s, 1H). HRMS (EI): *m/z* Calcd for C₂₄H₁₄ [M]⁺: 302.1096. Found: 302.1090.

Compound 3. The titled compound was prepared according to the procedure similar to that described for the preparation of **4** except that **13** (0.36 g, 1.2 mmol) and iodobenzene (0.24 g, 1.2 mmol) were used in place of 1,3-diethynylbenzene and 1-bromo-3-trimethylsilylethynylbenzene, respectively, to give **3** as a bright green solid (0.33 g, 73%). ¹H NMR (400 MHz, CDCl₃): δ 7.73 (s, 2H), 7.56-7.49 (m, 8H), 7.40-7.33 (m, 8H). HRMS (EI): *m/z* Calcd for C₃₀H₁₈ [M]⁺: 378.1409. Found: 378.1404.

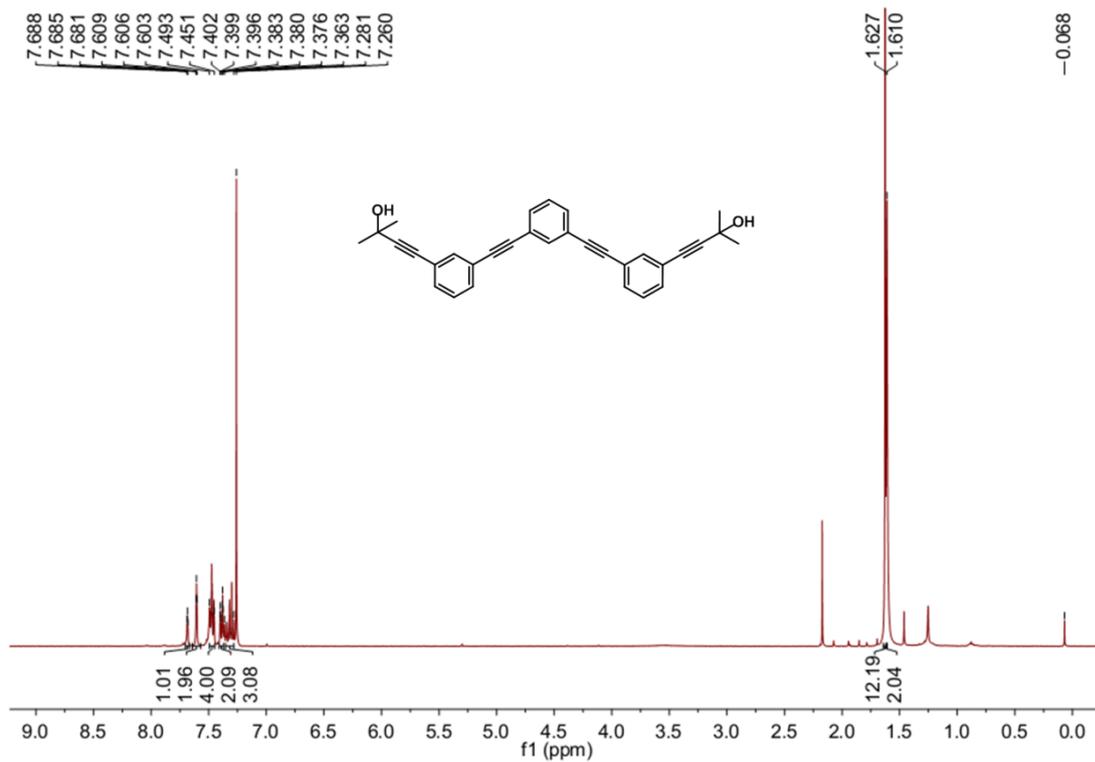


Fig. S1 ¹H NMR spectrum (400 MHz) of 4 in CDCl₃.

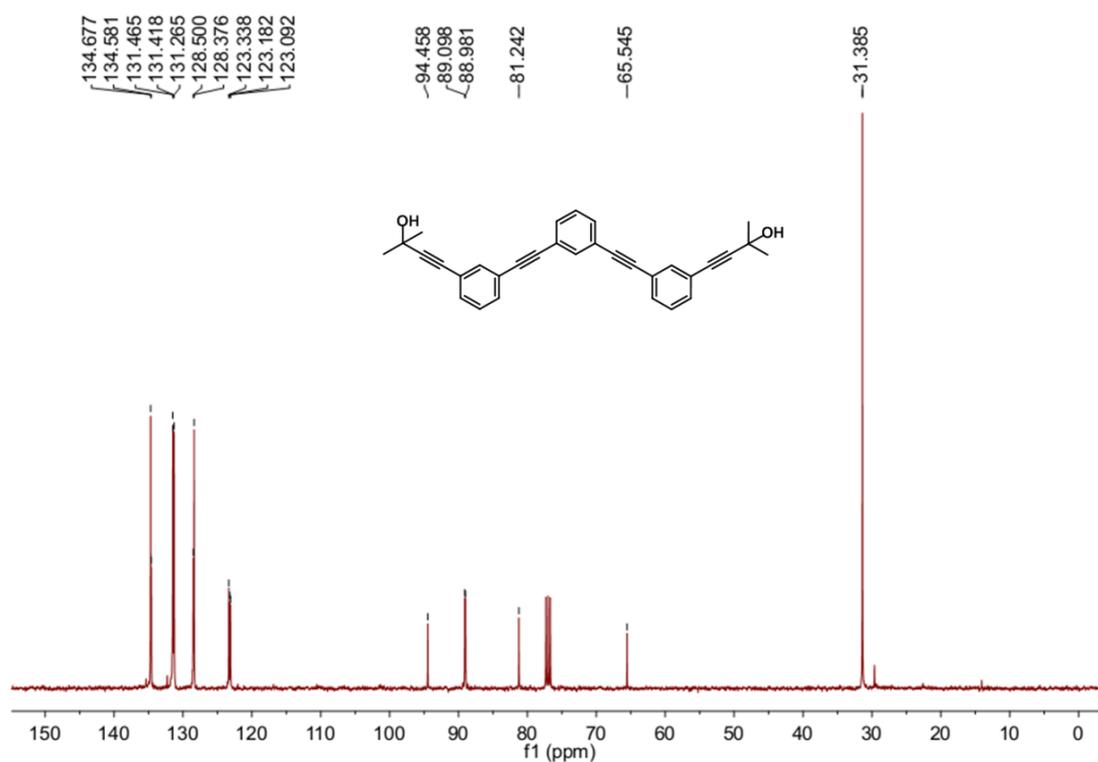


Fig. S2 ¹³C NMR spectrum (100 MHz) of 4 in CDCl₃.

Display Report

Analysis Info

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Sample Name cyy0402701
Comment

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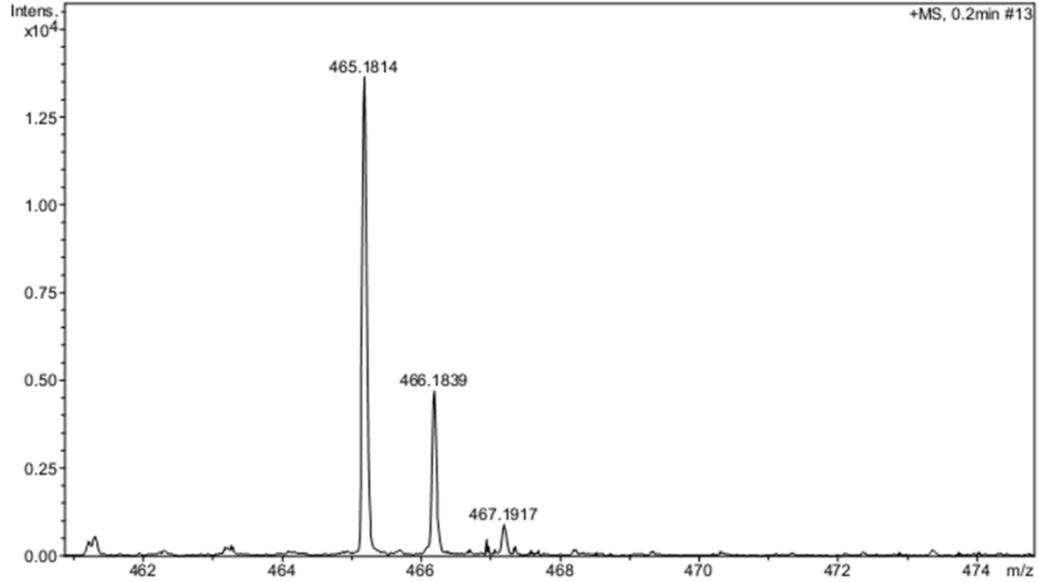
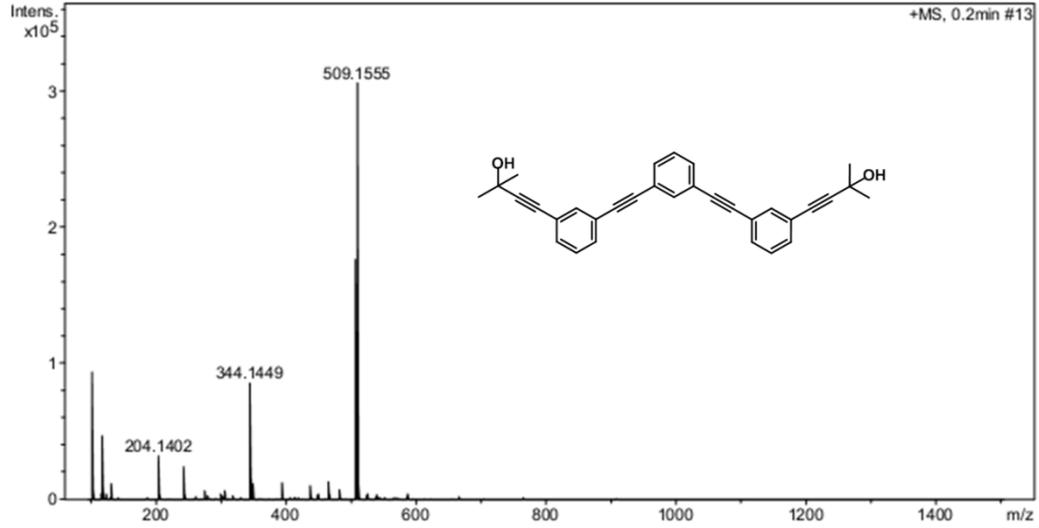


Fig. S3 High resolution mass spectra of 4.

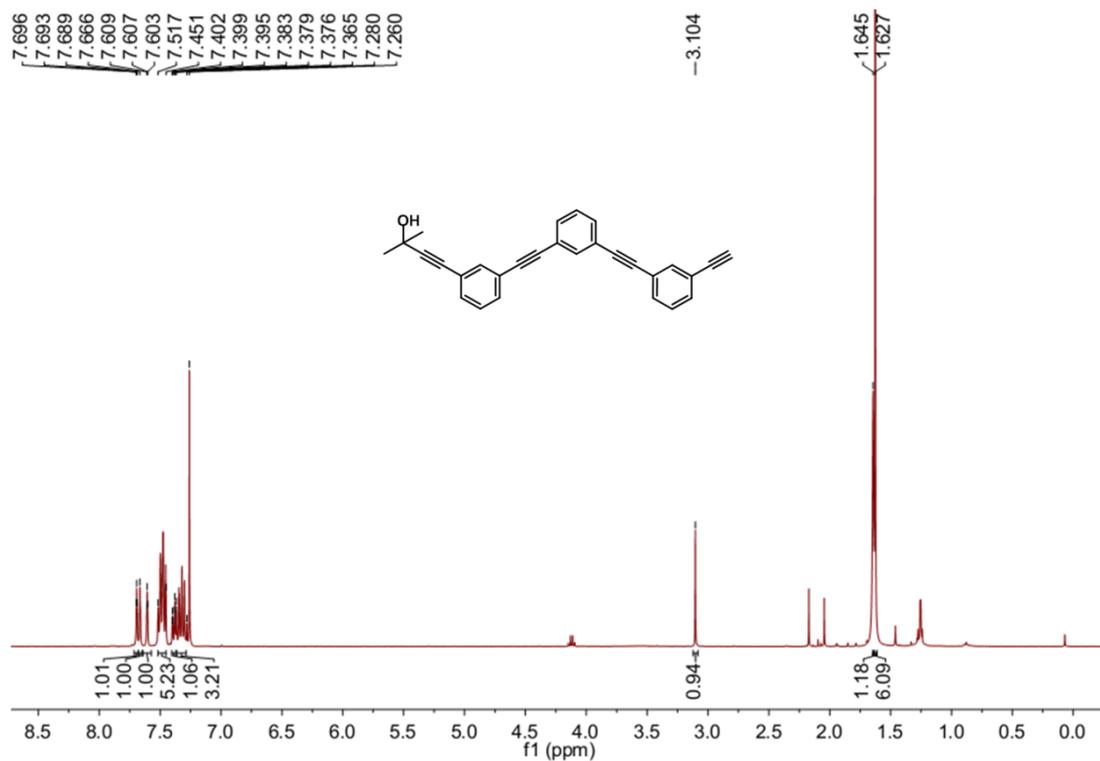


Fig. S4 ¹H NMR spectrum (400 MHz) of **5** in CDCl₃.

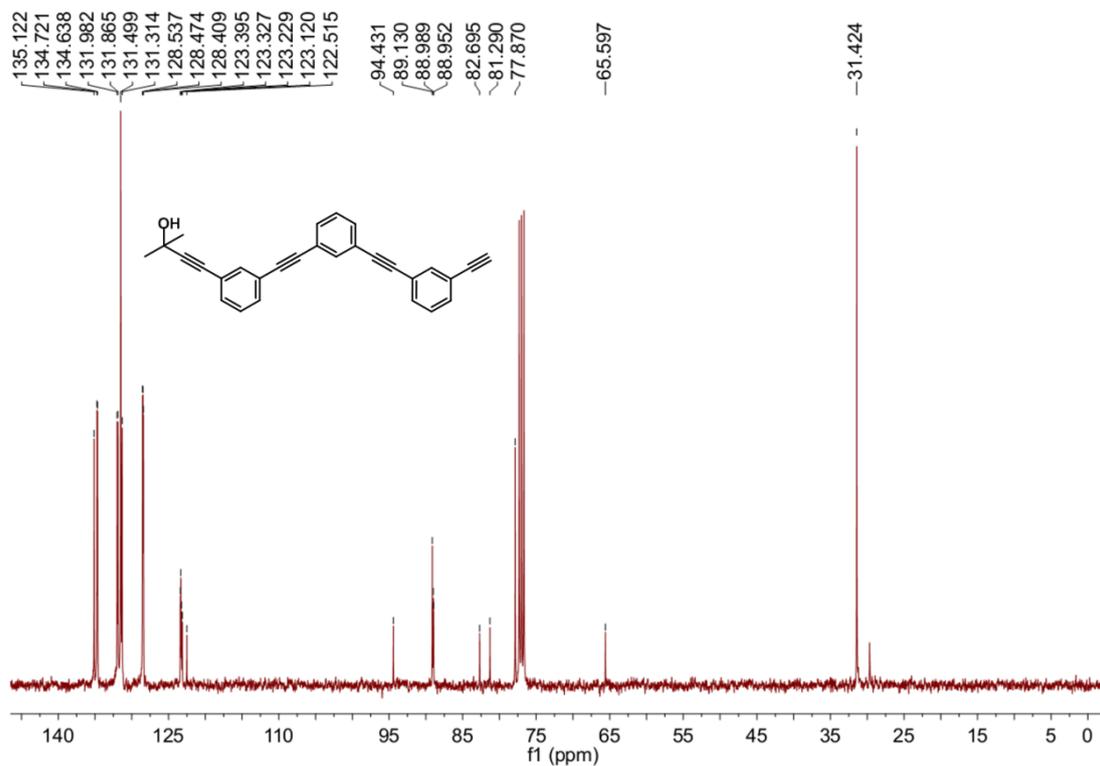


Fig. S5 ¹³C NMR spectrum (100 MHz) of **5** in CDCl₃.

Display Report

Analysis Info

Analysis Name D:\Data\MS\lzt\0902\cyy00402901_RA6_01_9720.d
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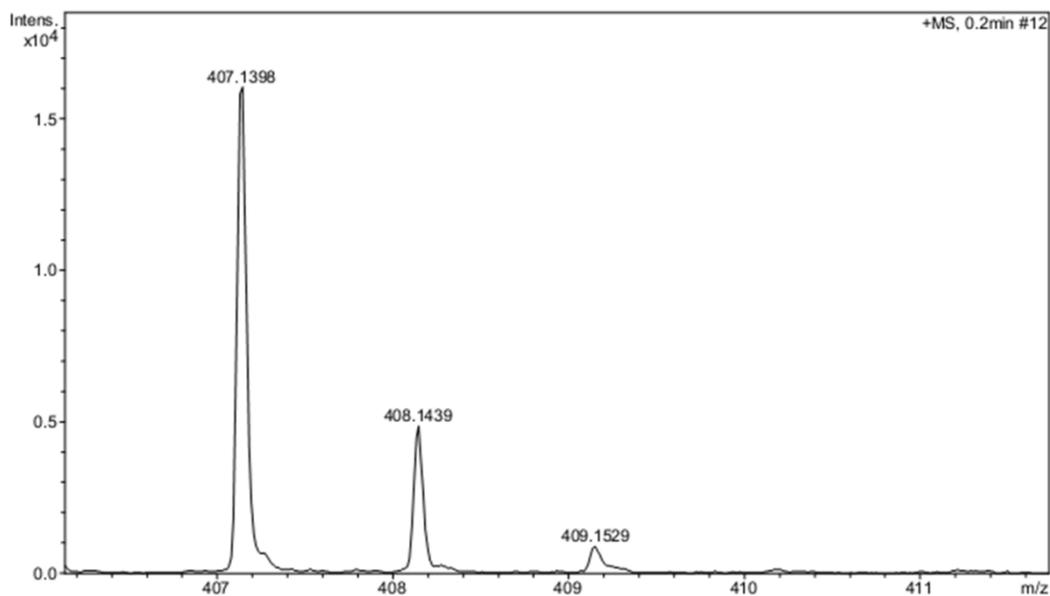
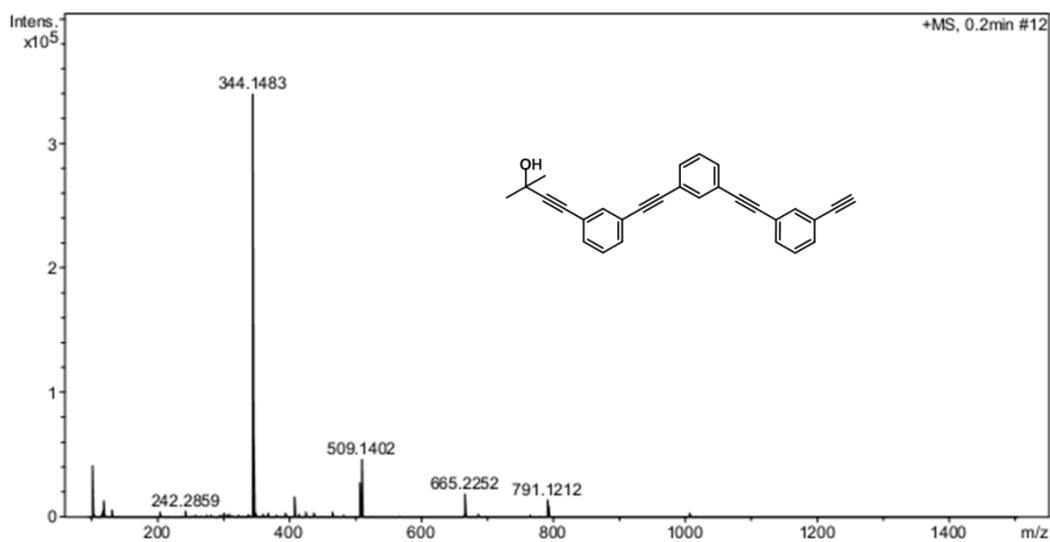


Fig. S6 High resolution mass spectra of **5**.

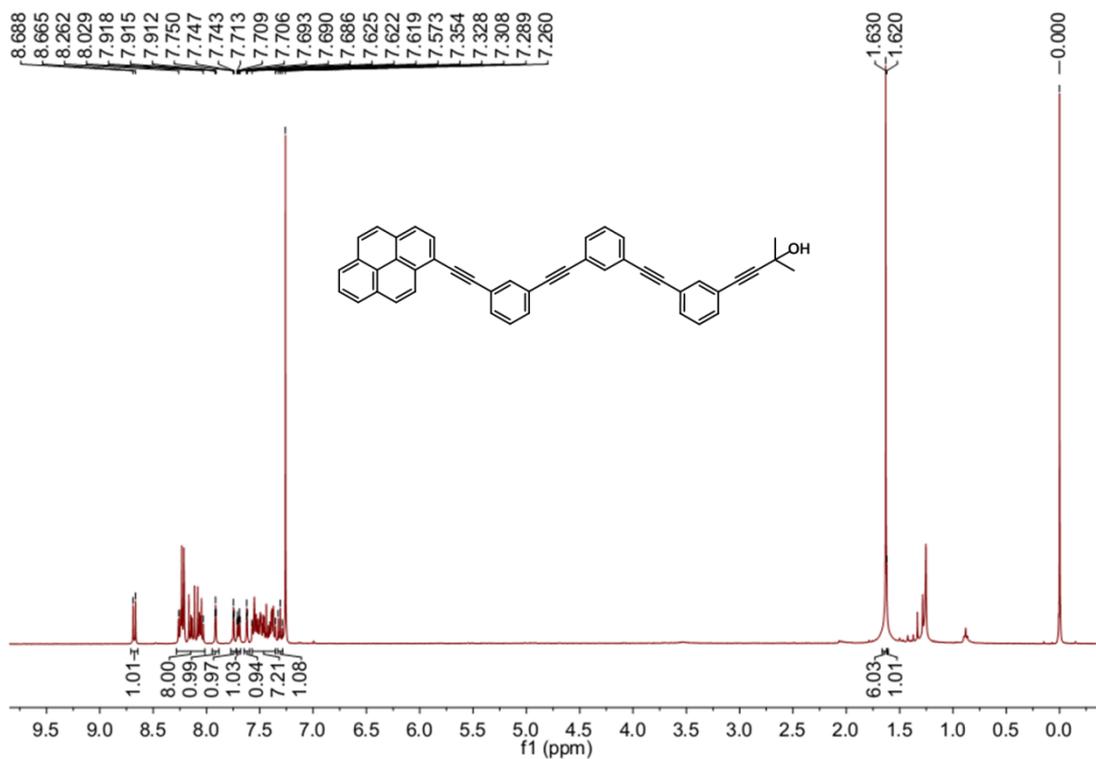


Fig. S7 ^1H NMR spectrum (400 MHz) of **6** in CDCl_3 .

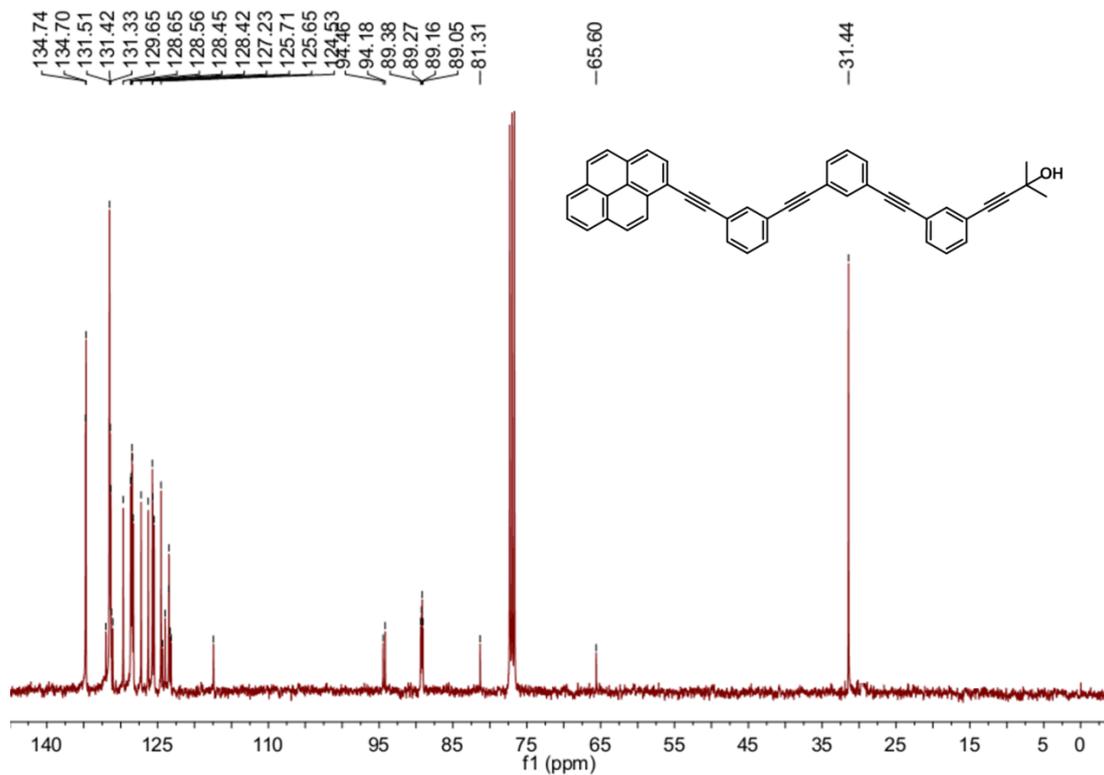


Fig. S8 ^{13}C NMR spectrum (100 MHz) of **6** in CDCl_3 .

Display Report

Analysis Info

Analysis Name D:\Data\MS\LZT\0617\CYY0512101_BA8_01_114.d
Method tune_200-800_hcoona-pos-2.5min.m
Sample Name CYY0512101
Comment

Acquisition Date 6/18/2014 7:51:17 AM

Operator gftang
Instrument / Ser# micrOTOF II 10257

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4000 V	Set Dry Gas	6.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

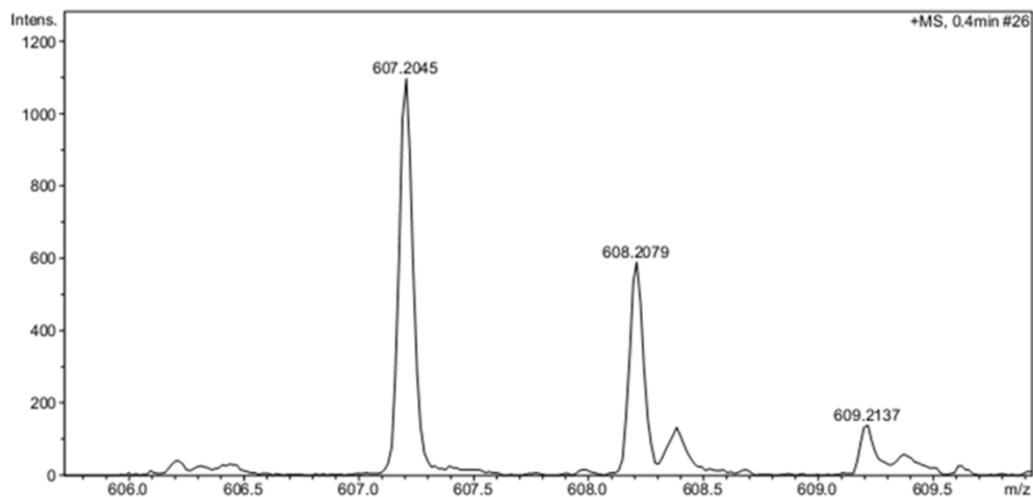
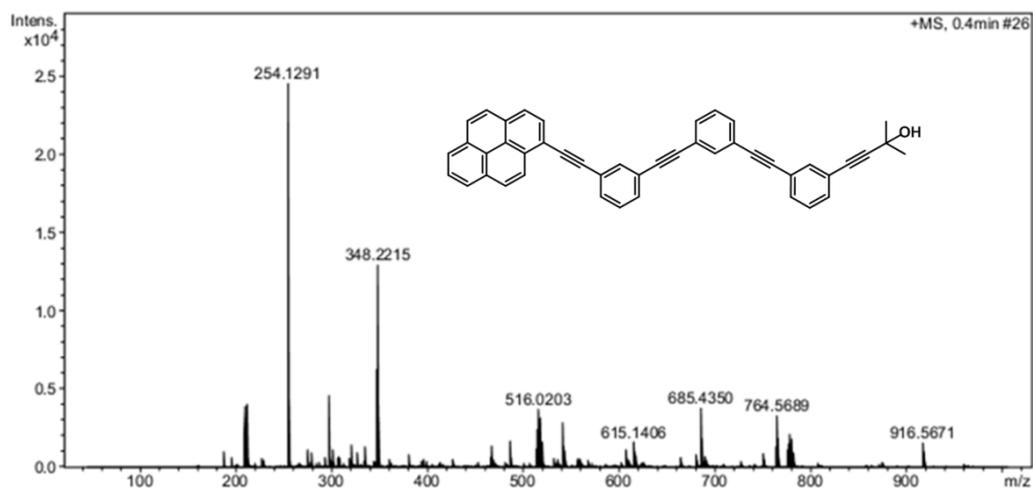


Fig. S9 High resolution mass spectra of 6.

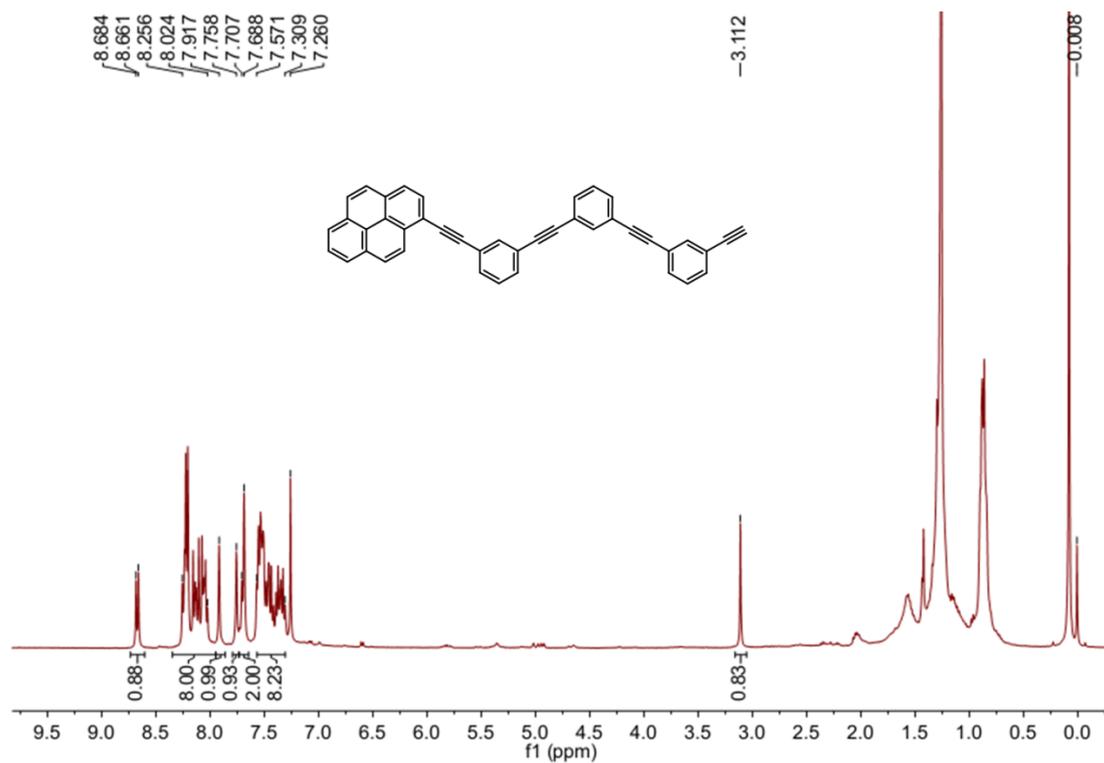


Fig. S10 ^1H NMR spectrum (400 MHz) of **1d** in CDCl_3 .

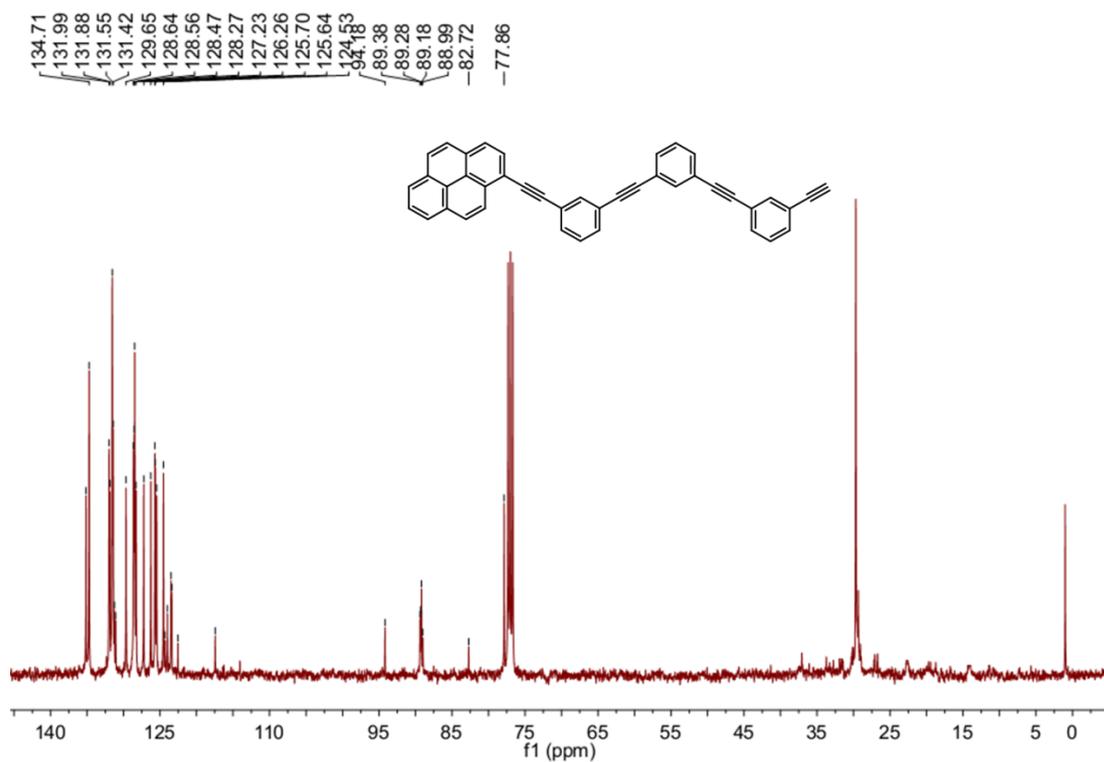


Fig. S11 ^{13}C NMR spectrum (100 MHz) of **1d** in CDCl_3 .

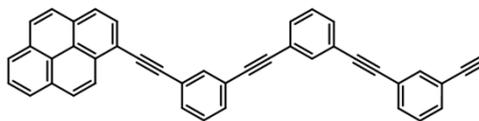
Instrument: Thermo Fisher Scientific LTQ FT Ultra

Card Serial Number : M150675

Sample Serial Number: CYY0607501

Operator : HUAQIN

Date: 2015/03/11



Operation Mode: MALDI_DHB

Elemental composition search on mass 526.17

m/z= 521.17-531.17

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
526.1715	526.1716	-0.23	32.0	C ₄₂ H ₂₂
	526.1721	-1.20	19.5	C ₂₈ H ₂₄ O ₆ N ₅
	526.1708	1.34	14.5	C ₂₇ H ₂₈ O ₁₀ N
	526.1708	1.35	20.0	C ₂₆ H ₂₂ O ₅ N ₈
	526.1734	-3.74	24.5	C ₂₉ H ₂₀ O ₂ N ₉
	526.1735	-3.75	19.0	C ₃₀ H ₂₆ O ₇ N ₂
	526.1694	3.90	15.0	C ₂₅ H ₂₆ O ₉ N ₄

Fig. S12 High resolution mass spectra of 1d.

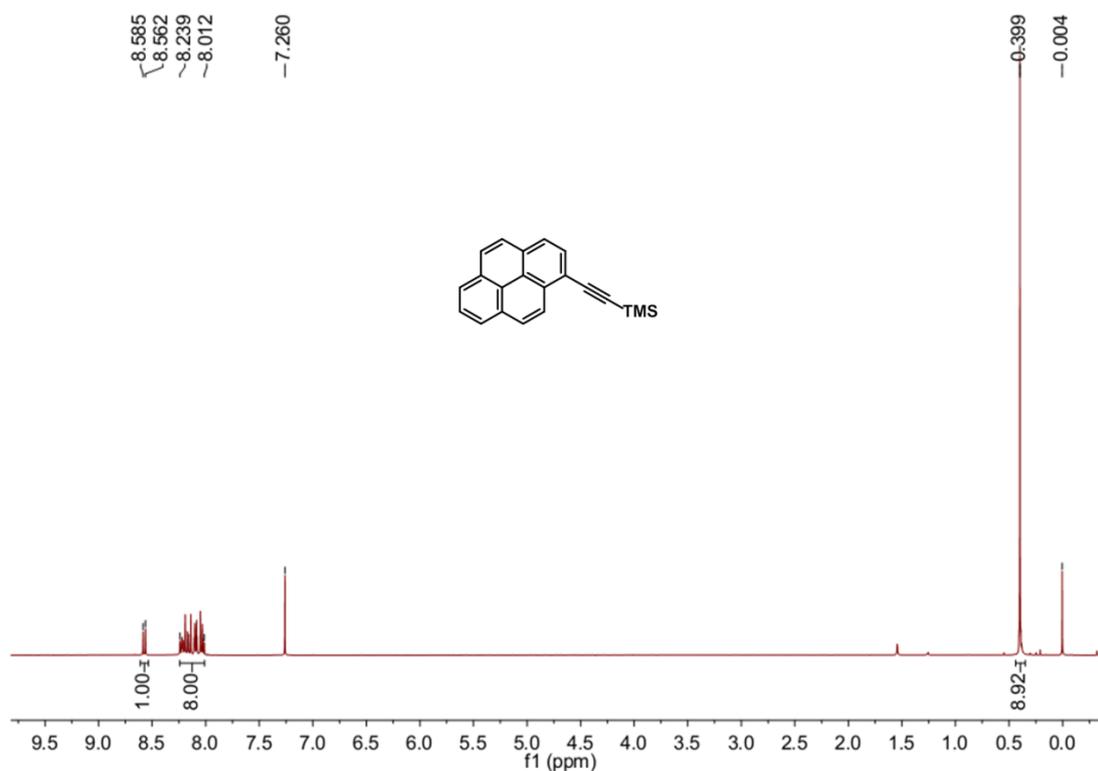


Fig. S13 ¹H NMR spectrum (400 MHz) of 7 in CDCl₃.



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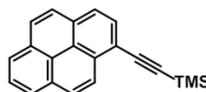
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0456

Sample Serial Number: CYY0608701

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

562 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 Si: 0-1 S: 0-1 Cl: 0-1

Minimum:

Maximum:

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
298.1173	298.1171	0.2	0.7	5.0	149.0	C13 H22 N2 O2 Si S
	298.1178	-0.5	-1.7	14.0	2.4	C21 H18 Si
	298.1165	0.8	2.7	6.0	343.8	C13 H18 N2 O6
	298.1183	-1.0	-3.4	1.0	2578.3	C12 H23 O6 Cl
	298.1158	1.5	5.0	5.0	2746.8	C16 H23 O S Cl

Fig. S14 High resolution mass spectra of 7.

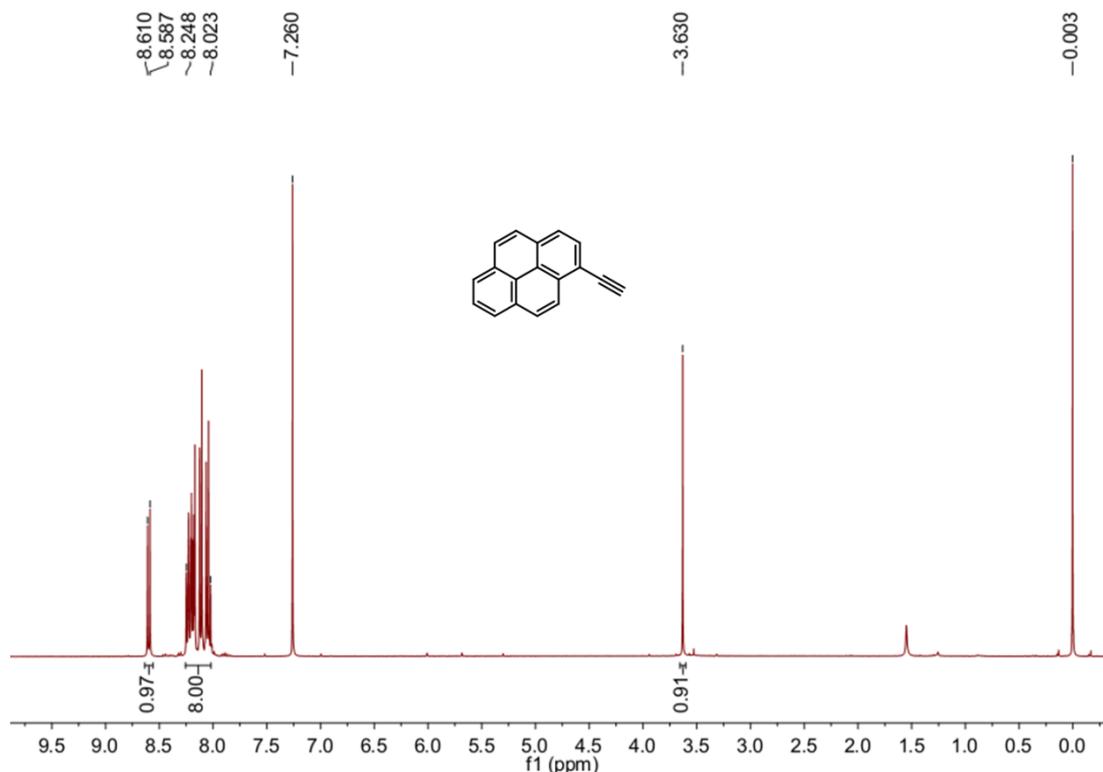


Fig. S15 ^1H NMR spectrum (400 MHz) of 1a in CDCl_3 .

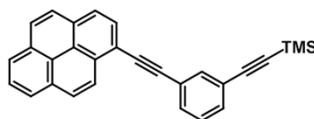
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0460

Sample Serial Number: CYY0609901

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

399 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 Si: 0-1 S: 0-1

Minimum:

Maximum:

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula	C21	H26	N2	O2	Si	S
398.1487	398.1484	0.3	0.8	11.0	3903.1	C21 H26 N2 O2 Si S						
	398.1491	-0.4	-1.0	20.0	24.2	C29 H22 Si						
	398.1478	0.9	2.3	12.0	13068.9	C21 H22 N2 O6						

Fig. S18 High resolution mass spectra of **8**.

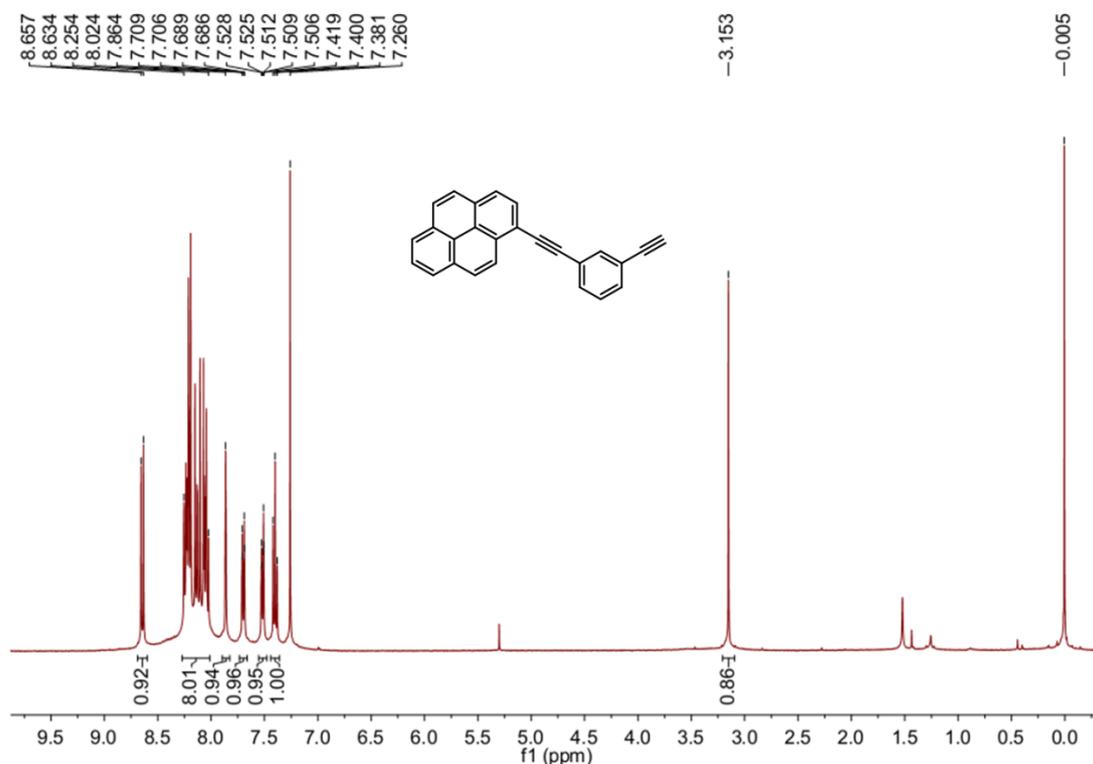


Fig. S19 ¹H NMR spectrum (400 MHz) of **1b** in CDCl₃.



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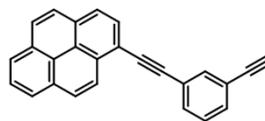
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0461

Sample Serial Number: CYY0610301

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

327 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 Si: 0-1 S: 0-1

Minimum:

Maximum:

Mass

326.1102

Calc. Mass

326.1096

326.1094

326.1089

326.1087

mDa

0.6

0.8

1.3

1.5

Si: 0-1

2.0

5.0

1.8

2.5

4.0

4.6

DBE

-1.5

50.0

20.0

1.5

11.0

11.0

i-FIT

45.1

7945.5

2228.1

1206.3

Formula

C26 H14

C11 H24 N O6 Si S

C18 H18 N2 O2 S

C17 H18 N2 O3 Si

Fig. S20 High resolution mass spectra of 1b.

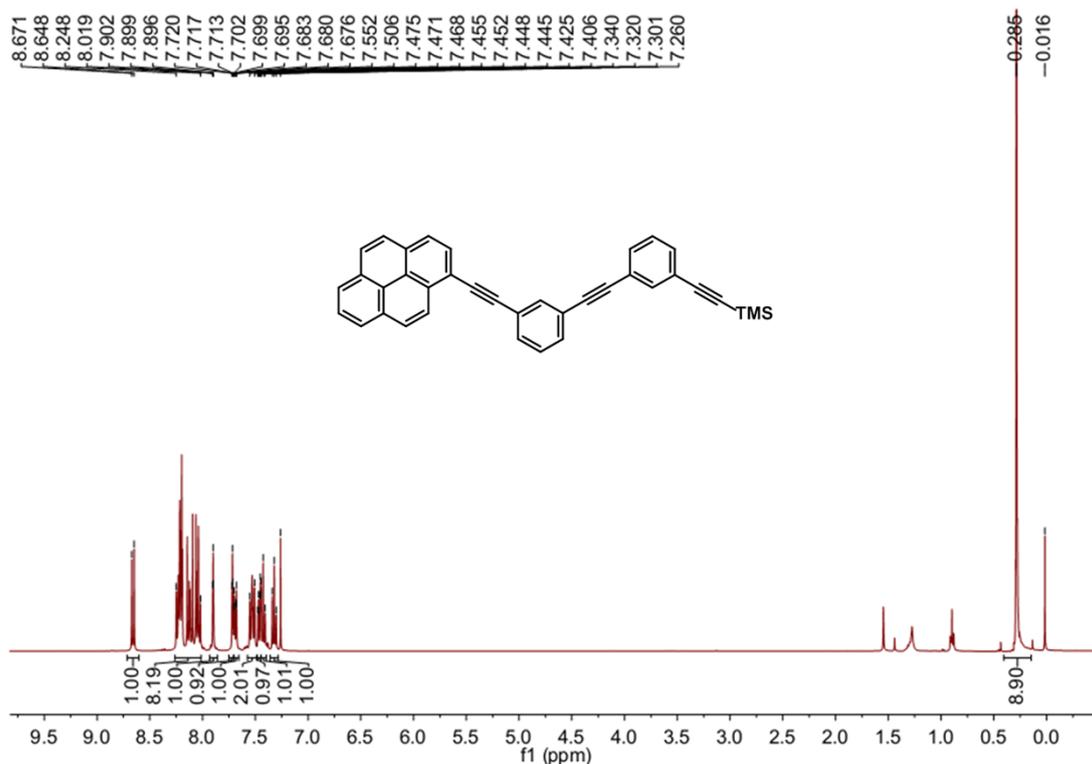


Fig. S21 ¹H NMR spectrum (400 MHz) of 9 in CDCl₃.

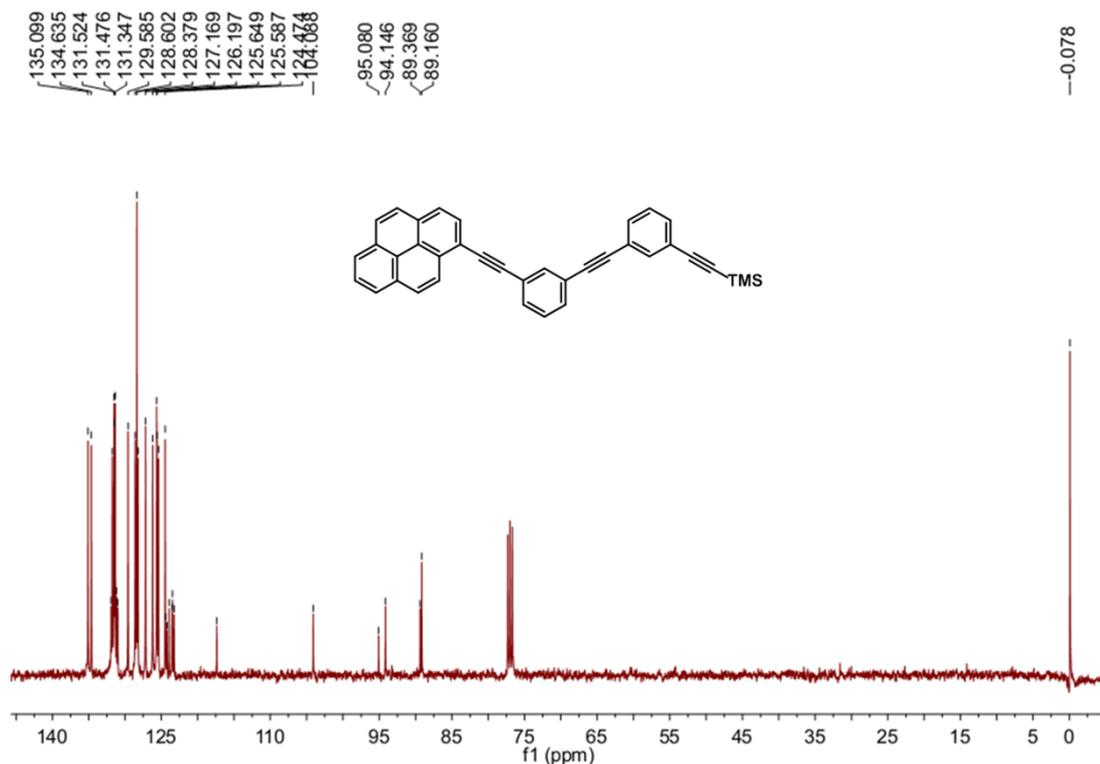


Fig. S22 ^{13}C NMR spectrum (100 MHz) of **9** in CDCl_3 .



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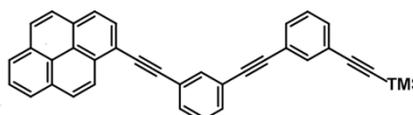
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0462

Sample Serial Number: CYY0610501

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
499 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 Si: 0-1 S: 0-1

Minimum:

Maximum:

Mass

Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
498.1804	0.5	1.0	26.0	5546053.0	C37 H26 Si
498.1797	1.2	2.4	17.0	5546052.0	C29 H30 N2 O2 Si S
498.1825	-1.6	-3.2	13.0	5546050.0	C26 H30 N2 O6 S
498.1791	1.8	3.6	18.0	5546050.5	C29 H26 N2 O6
498.1831	-2.2	-4.4	22.0	5546052.0	C34 H26 O4

Fig. S23 High resolution mass spectra of **9**.

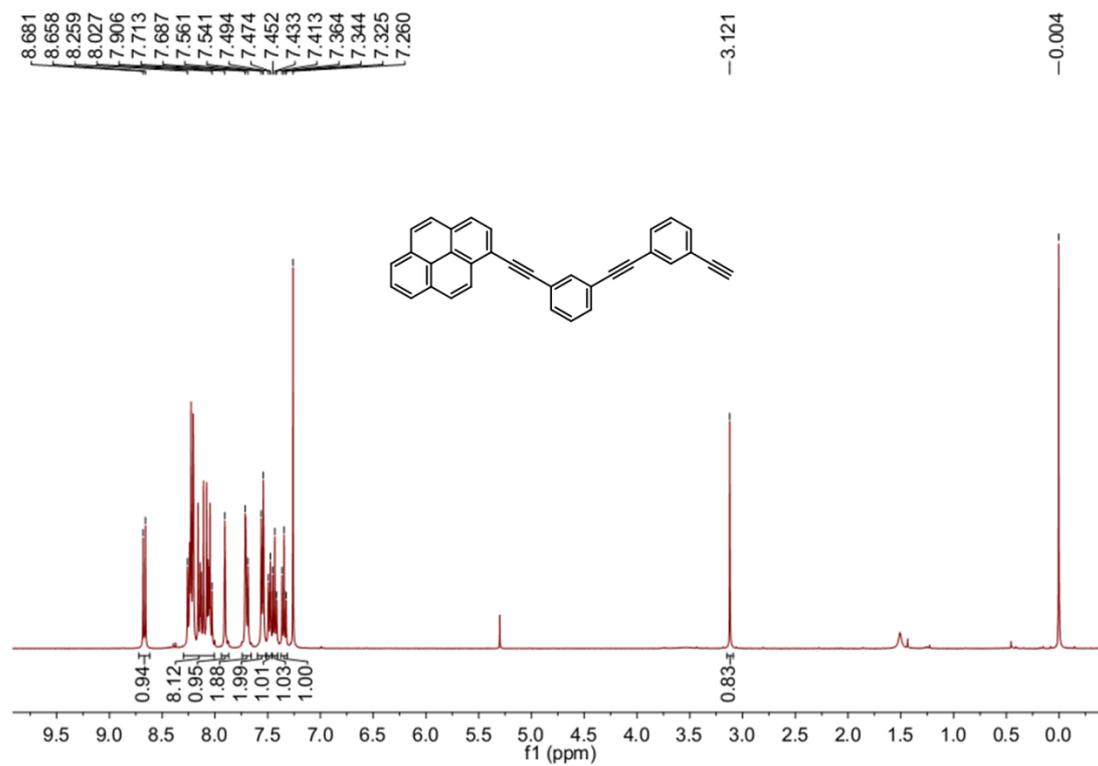


Fig. S24 ¹H NMR spectrum (400 MHz) of **1c** in CDCl₃.

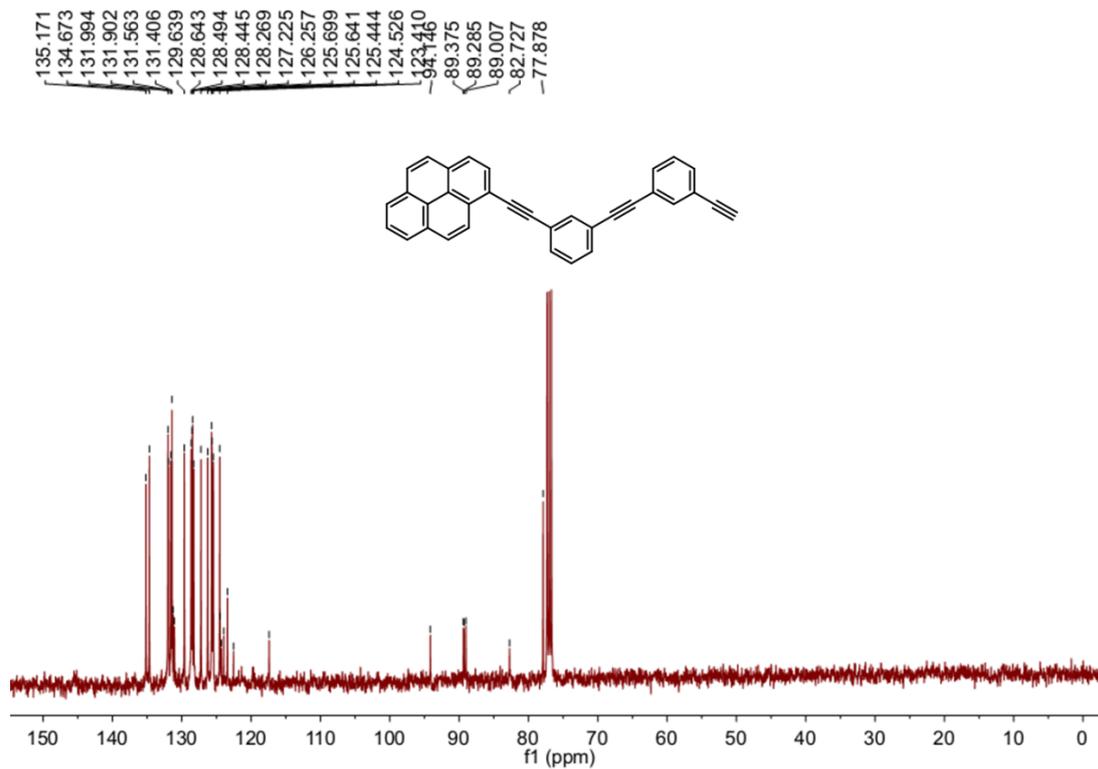


Fig. S25 ¹³C NMR spectrum (100 MHz) of **1c** in CDCl₃.



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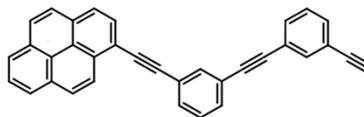
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0457

Sample Serial Number: CYY0610701

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
427 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
426.1414	426.1409	0.5	1.2	26.0	5546025.5	C34 H18
	426.1407	0.7	1.6	7.5	5546025.0	C19 H28 N O6 Si S
	426.1402	1.2	2.8	17.0	5546025.0	C26 H22 N2 O2 S
	426.1400	1.4	3.3	17.0	5546025.5	C25 H22 N2 O3 Si
	426.1433	-1.9	-4.5	12.0	5546025.0	C22 H26 N2 O3 Si S

Fig. S26 High resolution mass spectra of 1c.

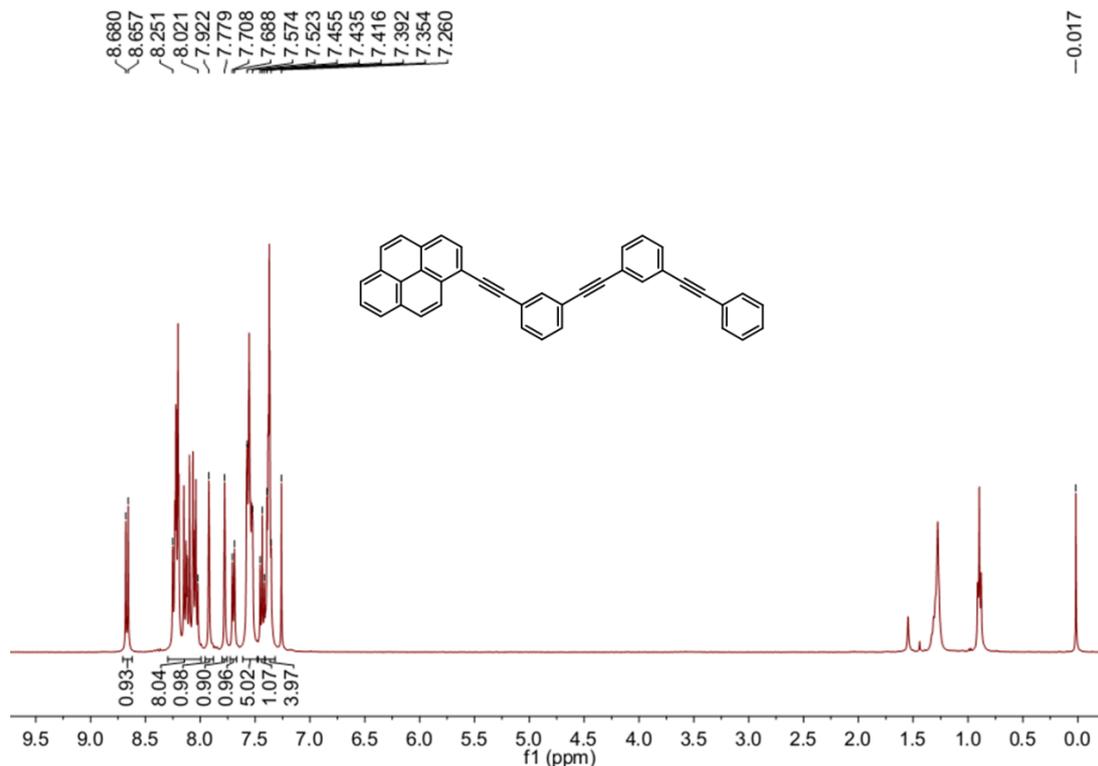


Fig. S27 ¹H NMR spectrum (400 MHz) of 2c in CDCl₃.

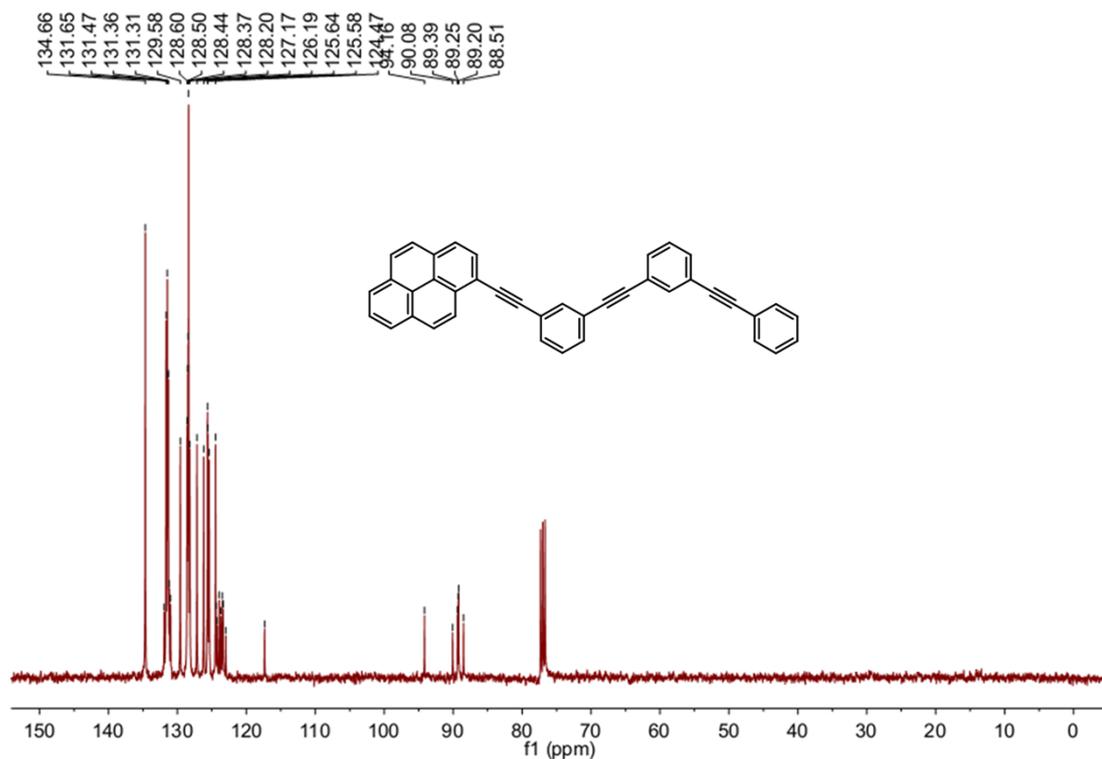


Fig. S28 ^{13}C NMR spectrum (100 MHz) of **2c** in CDCl_3 .



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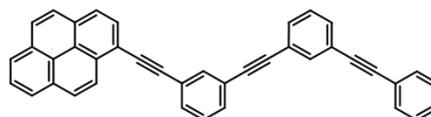
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-01-0458

Sample Serial Number: CYY0611101

Operator: Li

Date: 2014/01/26



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
501 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)
Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 Si: 0-1 S: 0-1

Minimum: -1.5

Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula	C	H	N	O	Si	S
502.1718	502.1720	-0.2	-0.4	11.5	5546025.5	C25 H32 N O6 Si S						
	502.1715	0.3	0.6	21.0	5546025.5	C32 H26 N2 O2 S						
	502.1722	-0.4	-0.8	30.0	5546026.0	C40 H22						
	502.1713	0.5	1.0	21.0	5546025.5	C31 H26 N2 O3 Si						

Fig. S29 High resolution mass spectra of **2c**.

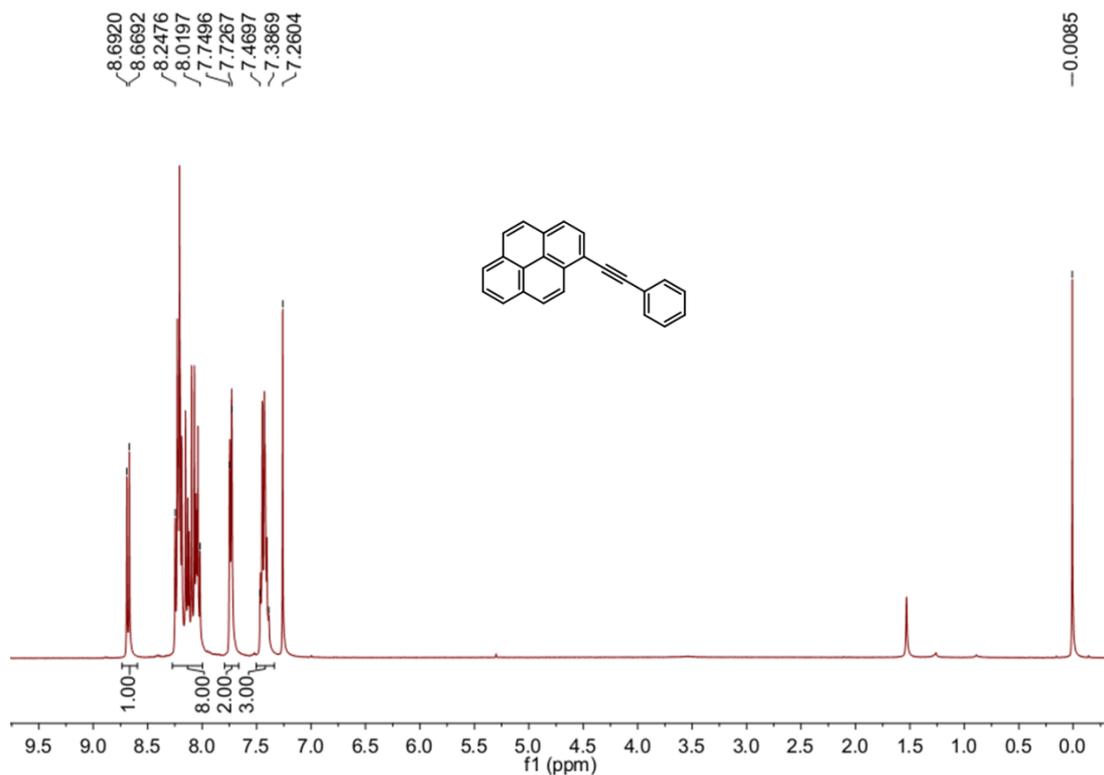


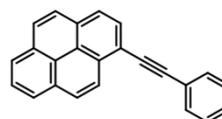
Fig. S30 ^1H NMR spectrum (400 MHz) of **2a** in CDCl_3 .

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Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1361
 Sample Serial Number: CYY0611701
 Operator: Li
 Date: 2014/04/09



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
 452 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
 Elements Used:
 C: 0-50 H: 0-80 N: 0-2 O: 0-6 S: 0-1 Cl: 0-1 I: 0-1

Minimum:		2.0	5.0	-1.5		
Maximum:				50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
302.1091	302.1089	0.2	0.7	9.0	5546050.0	C16 H18 N2 O2 S
	302.1096	-0.5	-1.7	18.0	5546054.5	C24 H14

Fig. S31 High resolution mass spectra of **2a**.

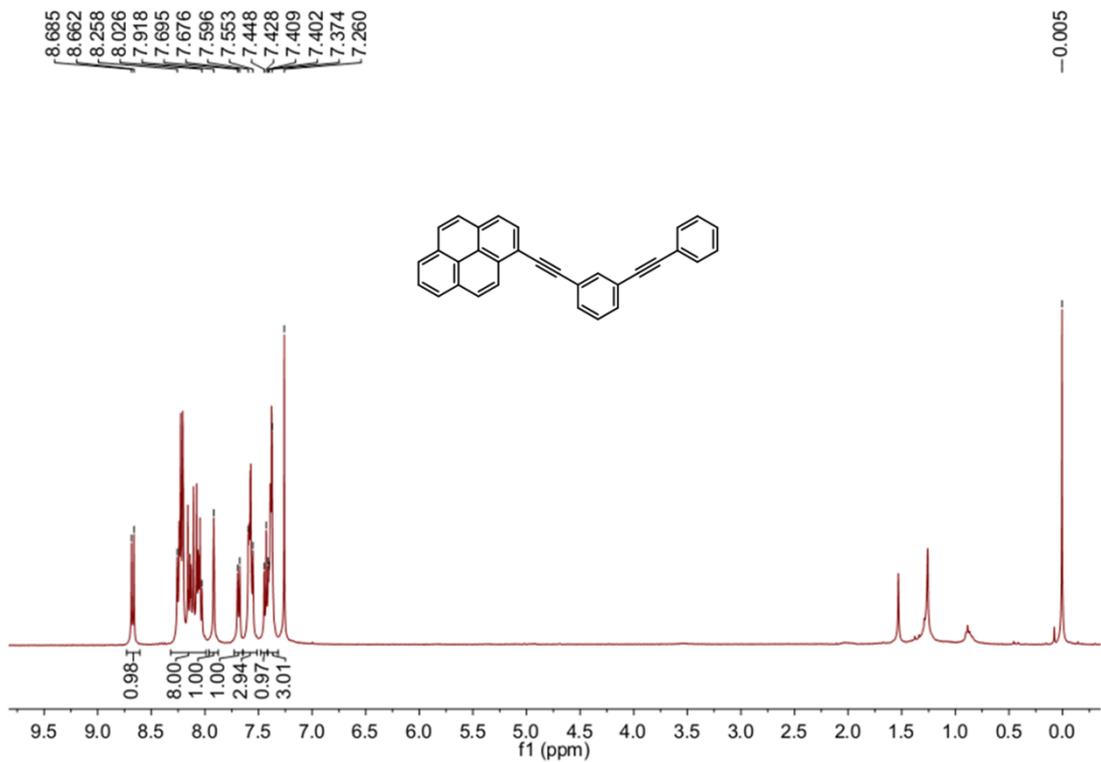


Fig. S32 ¹H NMR spectrum (400 MHz) of **2b** in CDCl₃.

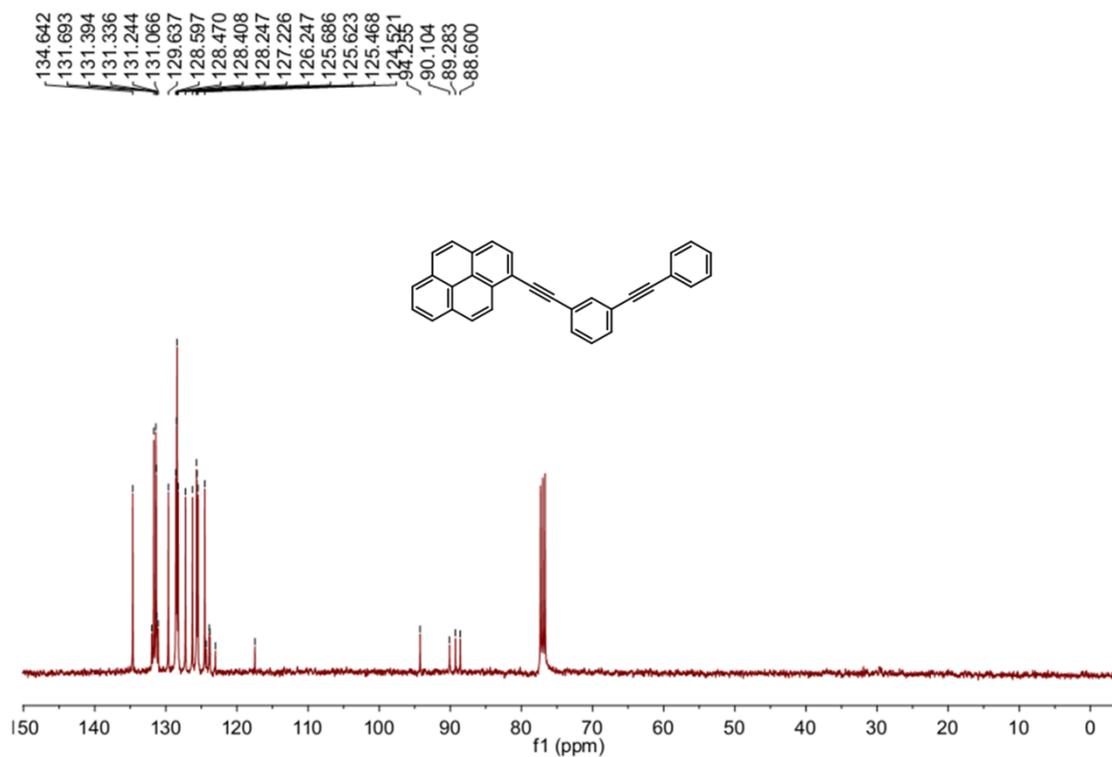


Fig. S33 ¹³C NMR spectrum (100 MHz) of **2b** in CDCl₃.

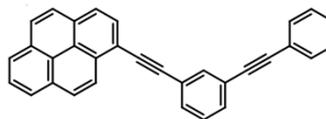
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1362

Sample Serial Number: CYY0611501

Operator: Li

Date: 2014/04/09



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
653 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-80 N: 0-2 O: 0-6 S: 0-1 Cl: 0-1 I: 0-1

Minimum:									
Maximum:		2.0	5.0	-1.5					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
402.1406	402.1409	-0.3	-0.7	24.0	5546025.5	C32	H18		
	402.1402	0.4	1.0	15.0	5546025.5	C24	H22	N2	O2 S
	402.1420	-1.4	-3.5	4.0	5546025.5	C19	H31	O	I
	402.1420	-1.4	-3.5	10.0	5546025.0	C23	H27	O2	S Cl
	402.1387	1.9	4.7	15.0	5546025.0	C26	H23	O2	Cl
	402.1425	-1.9	-4.7	-0.5	5546025.0	C16	H34	N	Cl I

Fig. S34 High resolution mass spectra of **2b**.

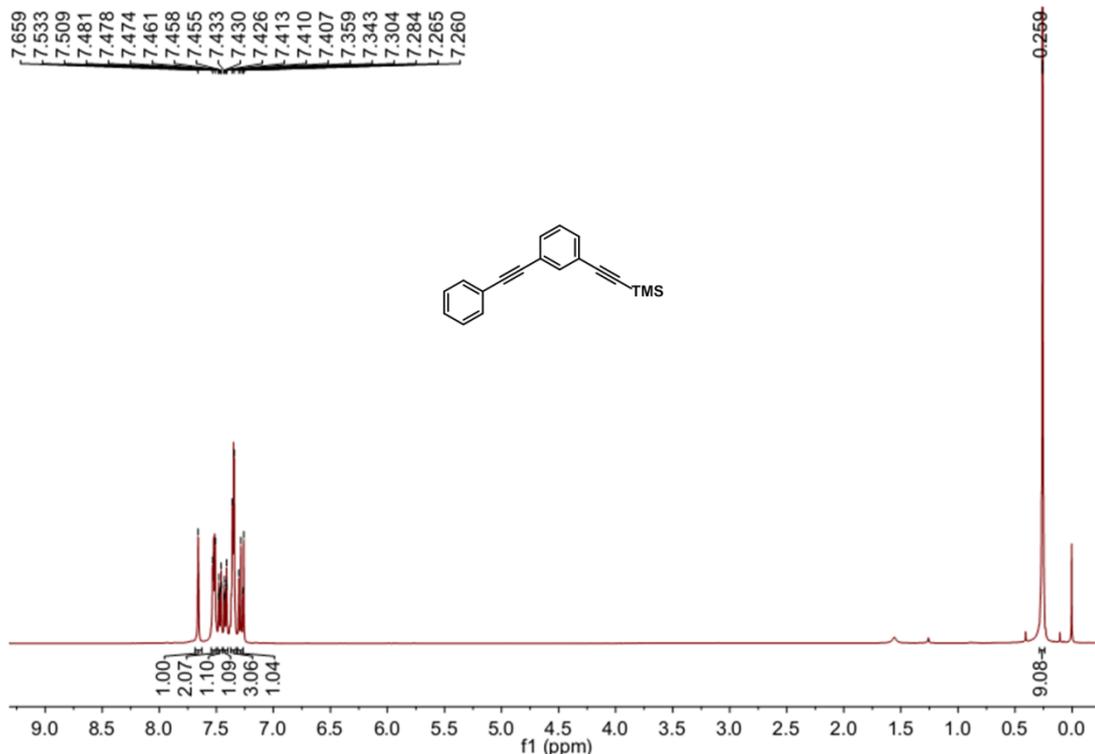


Fig. S35 ^1H NMR spectrum (400 MHz) of **10** in CDCl_3 .



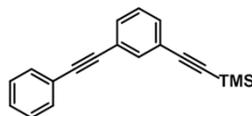
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1661

Sample Serial Number: CYY07000301

Operator: Li

Date: 2014/04/29



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
 394 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-45 H: 0-80 N: 0-2 O: 0-4 S: 0-1 Cl: 0-1 Si: 0-1

Minimum:		2.0	5.0	-1.5					
Maximum:				50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula			
274.1179	274.1178	0.1	0.4	12.0	13.6	C19 H18 Si			
	274.1171	0.8	2.9	3.0	1539.8	C11 H22 N2 O2 S Si			

Fig. S36 High resolution mass spectra of 10.

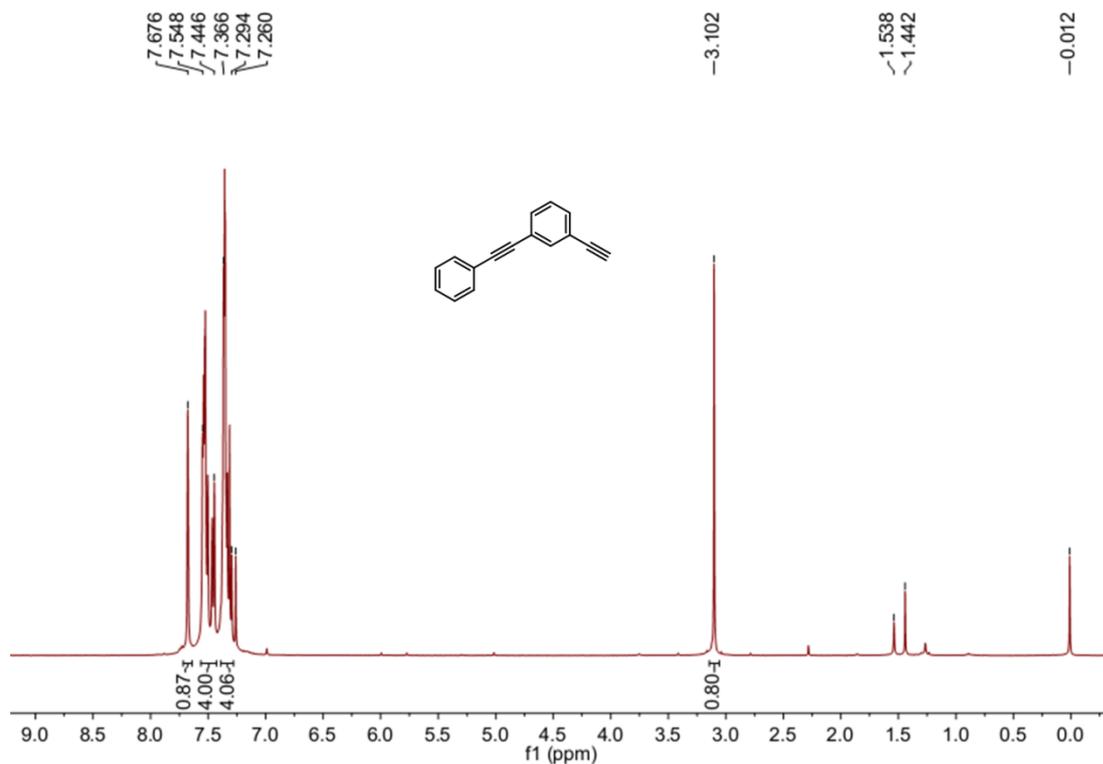


Fig. S37 ¹H NMR spectrum (400 MHz) of 11 in CDCl₃.

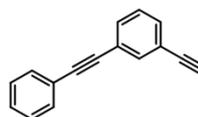
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1660

Sample Serial Number: CYY07000501

Operator: Li

Date: 2014/04/29



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
 291 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-45 H: 0-80 N: 0-2 O: 0-4 S: 0-1 Cl: 0-1 Si: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
202.0782	202.0783	-0.1	-0.5	12.0	14.4	C16 H10
202.0776	202.0776	0.6	3.0	3.0	2353.2	C8 H14 N2 O2 S
202.0774	202.0774	0.8	4.0	3.0	1555.8	C7 H14 N2 O3 Si

Fig. S38 High resolution mass spectra of 11.

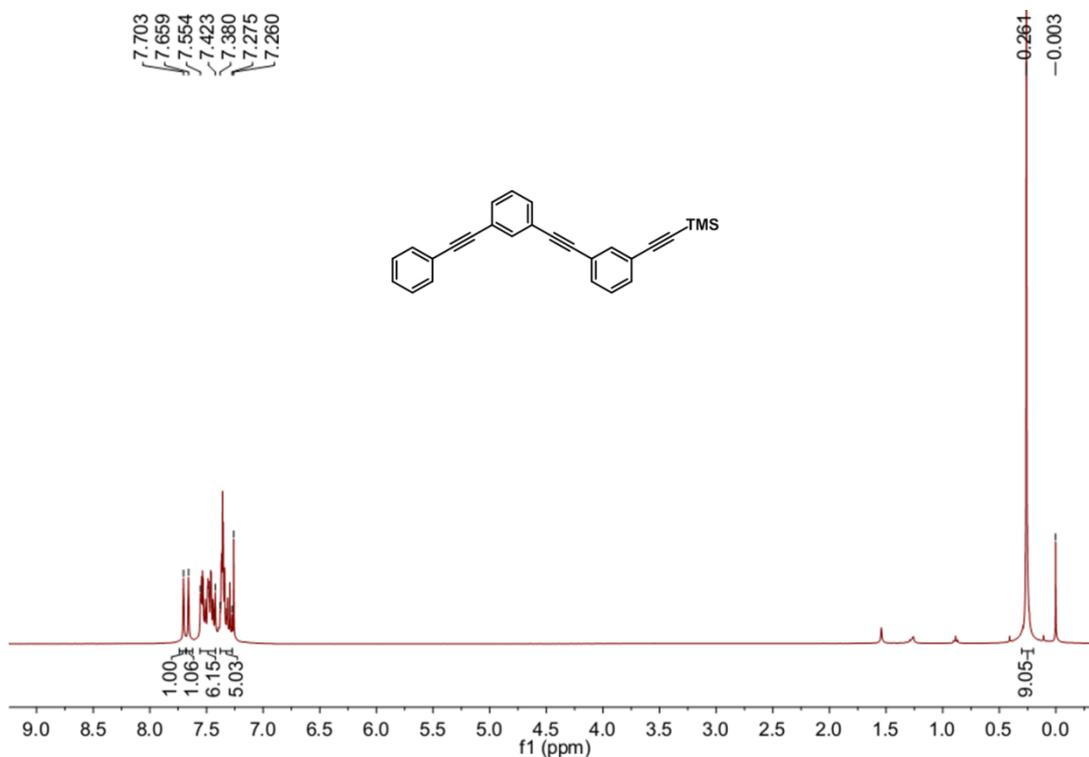


Fig. S39 ¹H NMR spectrum (400 MHz) of 12 in CDCl₃.

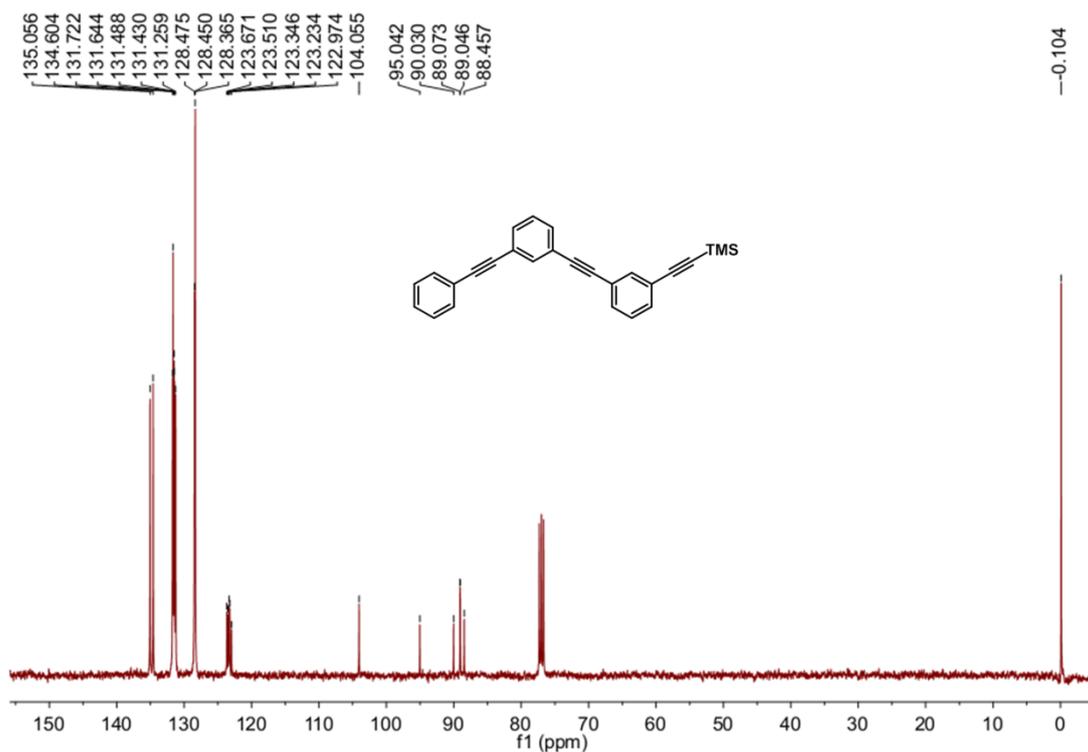


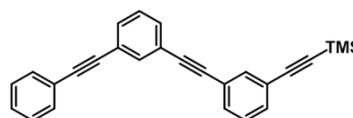
Fig. S40 ^{13}C NMR spectrum (100 MHz) of **12** in CDCl_3 .

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 High Resolution MS Data Report



Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1659
 Sample Serial Number: CYY07000701
 Operator: Li
 Date: 2014/04/29



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
 537 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
 Elements Used:
 C: 0-45 H: 0-80 N: 0-2 O: 0-4 S: 0-1 Cl: 0-1 Si: 0-1

Minimum:		2.0	5.0	-1.5		
Maximum:				50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
374.1486	374.1491	-0.5	-1.3	18.0	16.2	C27 H22 Si
	374.1484	0.2	0.5	9.0	185.1	C19 H26 N2 O2 S Si
	374.1469	1.7	4.5	9.0	3021.5	C21 H27 O2 Cl Si
	374.1471	1.5	4.0	9.0	3138.7	C22 H27 O S Cl
	374.1503	-1.7	-4.5	4.0	3428.9	C18 H31 O2 S Cl Si

Fig. S41 High resolution mass spectra of **12**.

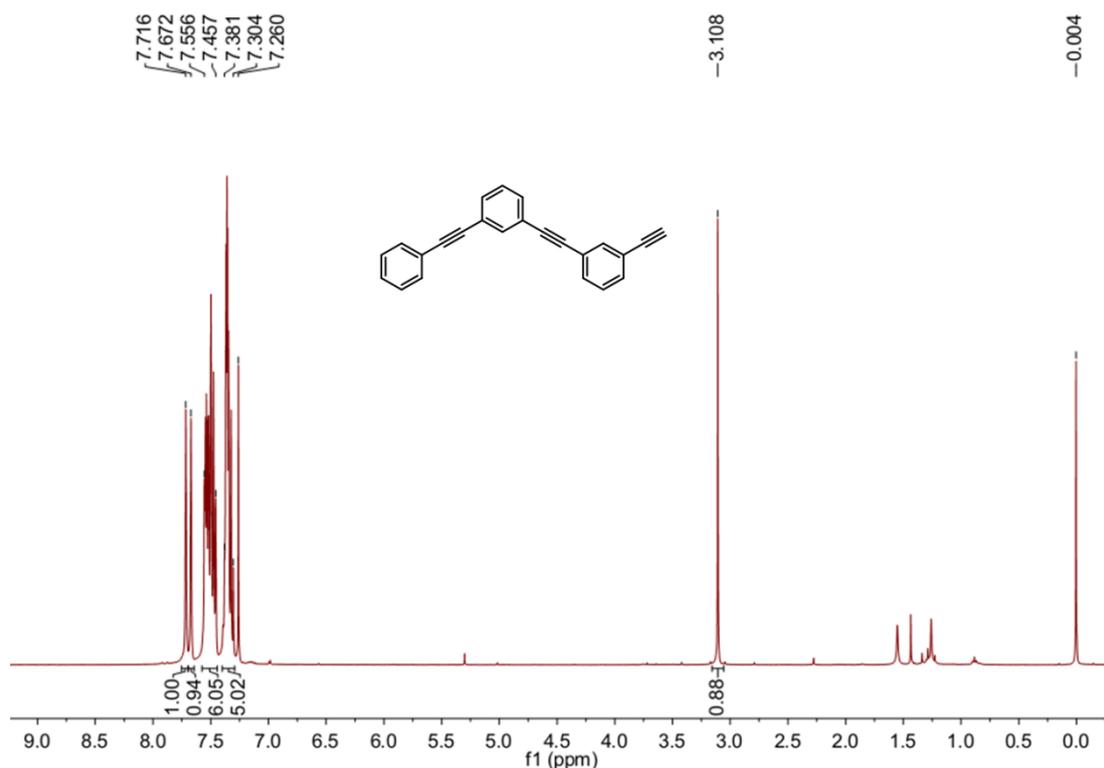


Fig. S42 ^1H NMR spectrum (400 MHz) of **13** in CDCl_3 .

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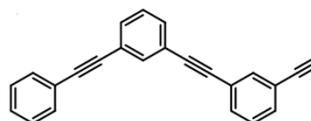
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1658

Sample Serial Number: CYY07000901

Operator: Li

Date: 2014/04/29



Elemental Composition Report

Single Mass Analysis
 Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
 420 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)
 Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
302.1090	302.1096	-0.6	-2.0	18.0	6.0	C24 H14
	302.1089	0.1	0.3	9.0	353.7	C16 H18 N2 O2 S
	302.1078	1.2	4.0	6.0	493.2	C13 H16 N2 O4 F2
	302.1100	-1.0	-3.3	5.0	506.4	C13 H19 N2 O3 F S

Fig. S43 High resolution mass spectra of **13**.

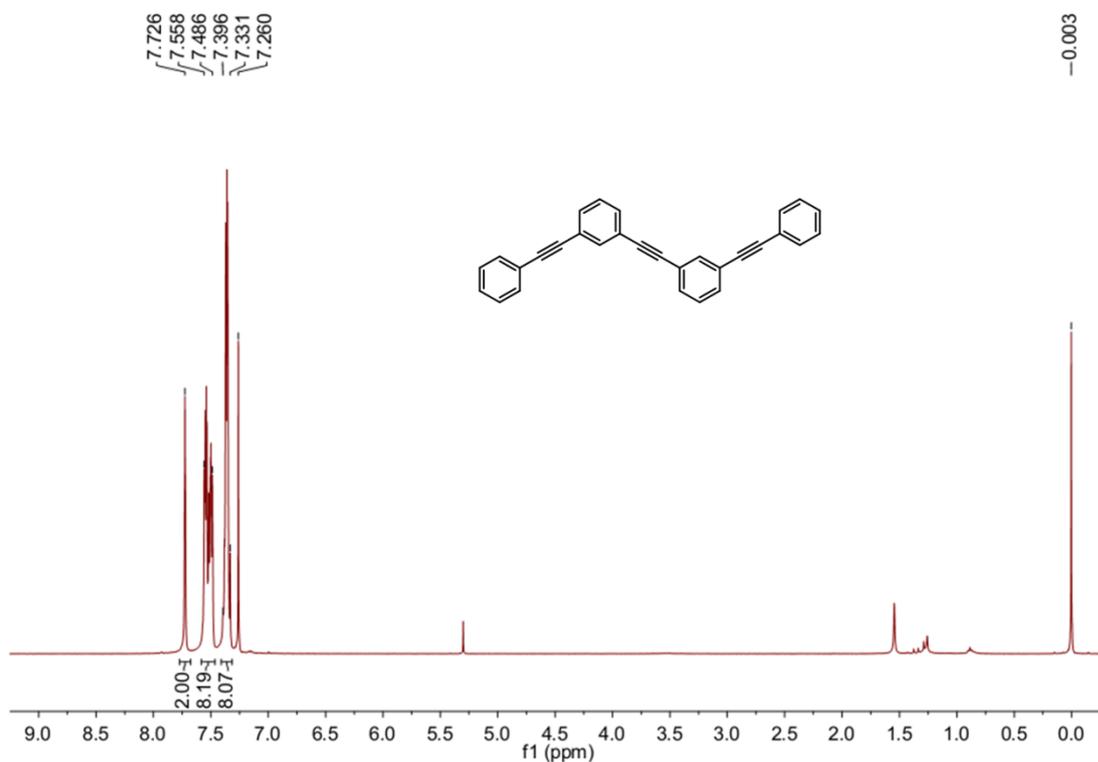


Fig. S44 ^1H NMR spectrum (400 MHz) of **3** in CDCl_3 .

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report



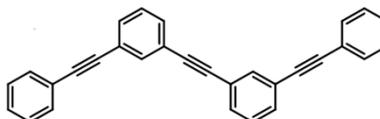
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T15-04-1657

Sample Serial Number: CYY07011101

Operator: Li

Date: 2014/04/29



Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

531 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-45 H: 0-80 N: 0-2 O: 0-4 F: 0-3 S: 0-1

Minimum: -1.5

Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
378.1404	378.1409	-0.5	-1.3	22.0	9.5	C30 H18
	378.1420	-1.6	-4.2	18.0	25.5	C27 H19 O F
	378.1402	0.2	0.5	13.0	413.5	C22 H22 N2 O2 S
	378.1391	1.3	3.4	10.0	532.9	C19 H20 N2 O4 F2
	378.1413	-0.9	-2.4	9.0	551.9	C19 H23 N2 O3 F S

Fig. S45 High resolution mass spectra of **3**.

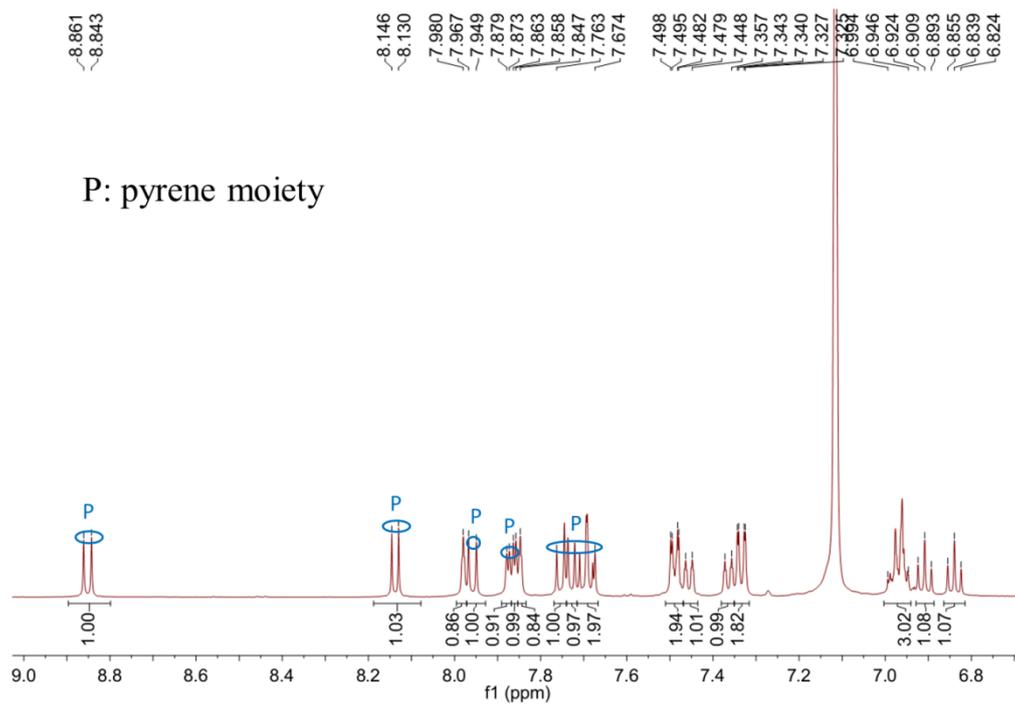


Fig. S46 ^1H NMR spectrum (500 MHz) of **2c** in benzene- d_6 .

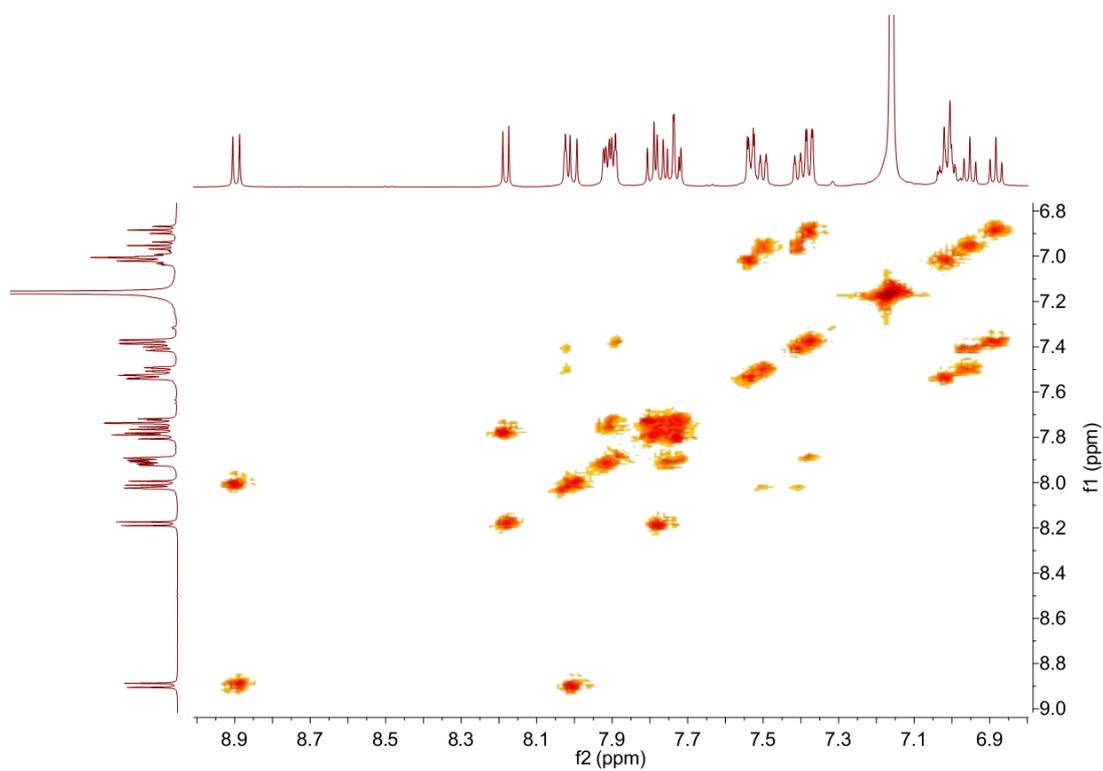


Fig. S47 ^1H - ^1H COSY spectrum (500 MHz) of **2c** in benzene- d_6 at 10 mM.

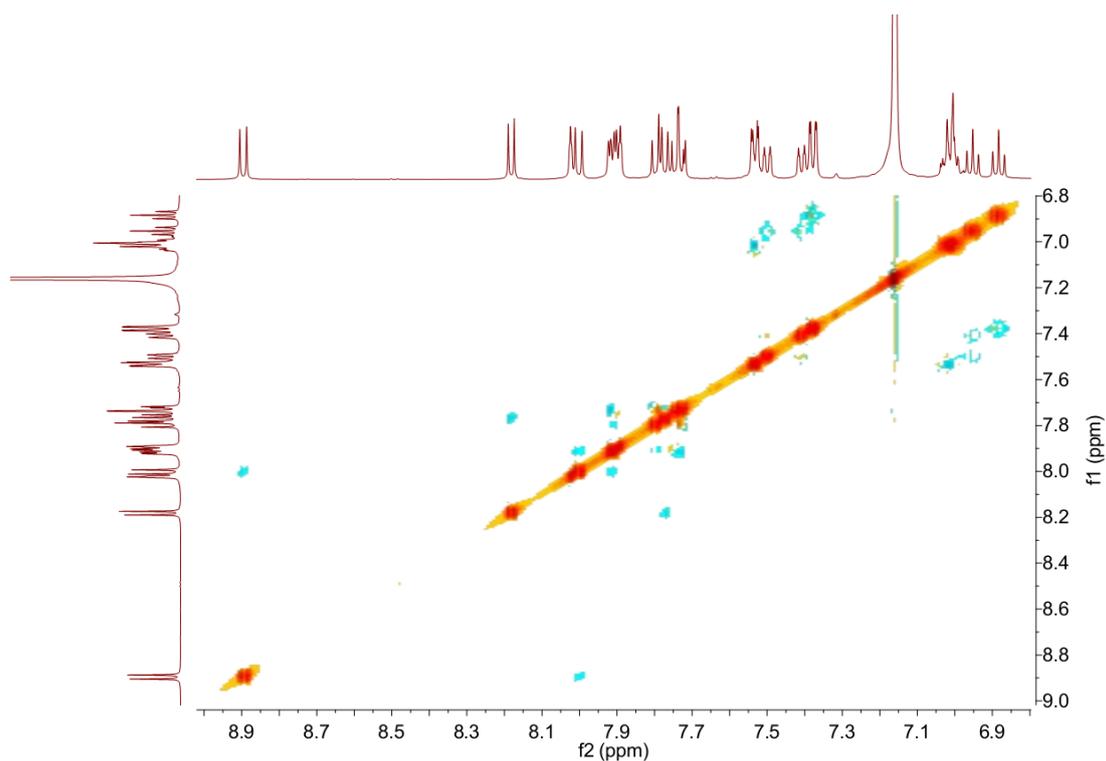


Fig. S48 NOESY spectrum (500 MHz) of **2c** in benzene- d_6 at 10 mM.

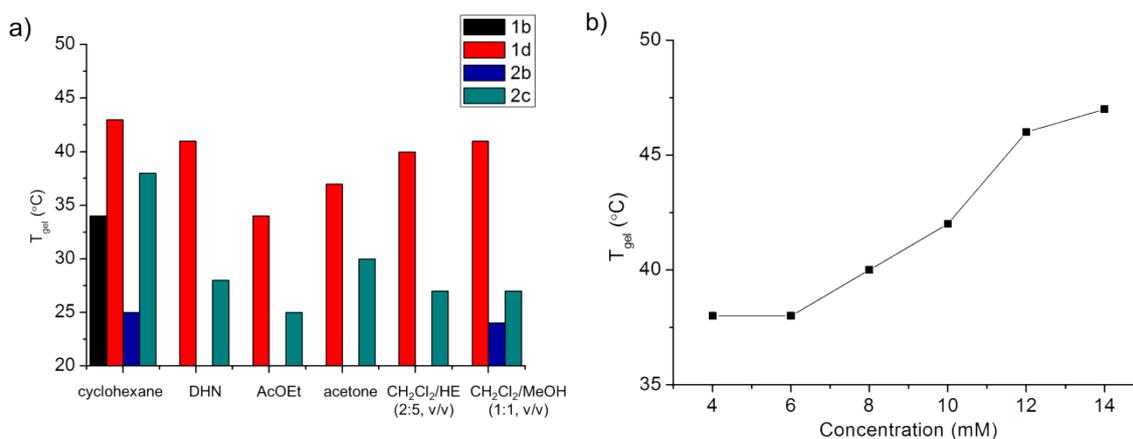


Fig. S49 a) Bar diagram showing the gel-sol transition temperatures (T_{gel}) of the gels of **1b** (black), **1d** (red), **2b** (blue) and **2c** (green) at their respective critical gelation concentrations (CGCs) in different solvents. b) Plot of T_{gel} versus concentration of **2c** in cyclohexane. T_{gel} was tested using the “stable-to-inversion of a test tube” method.

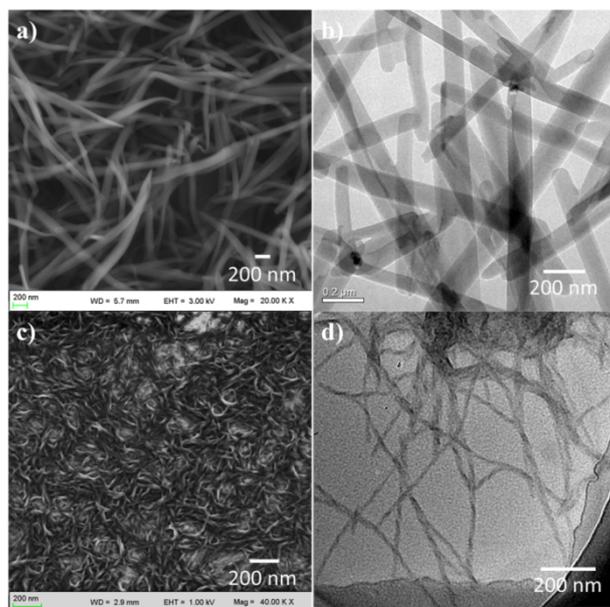


Fig. S50 SEM (a and c) and TEM (b and d) images of air-dried organogels formed in cyclohexane by **1b** (a,b), **1d** (c,d).

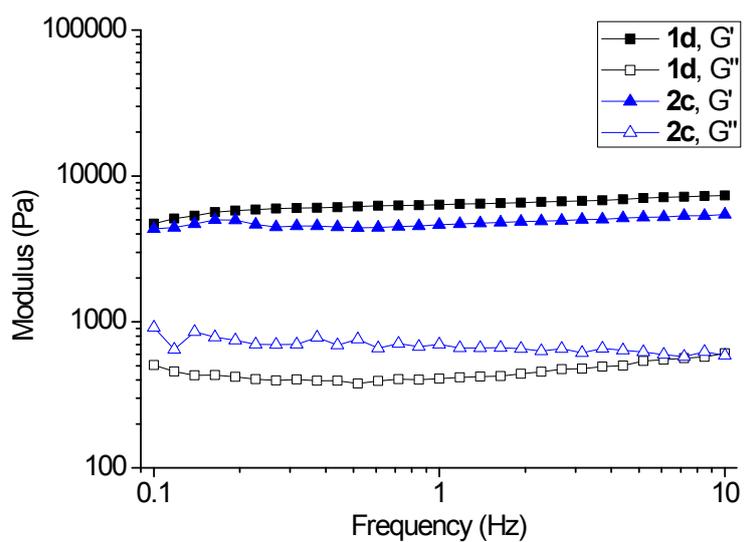


Fig. S51 Frequency-dependent storage modulus (G') and loss modulus (G'') for the organogels formed in cyclohexane by **1d** and **2c** (20 °C, $\gamma = 0.001$). The concentration for **1d** and **2c** was 7.0 mM.

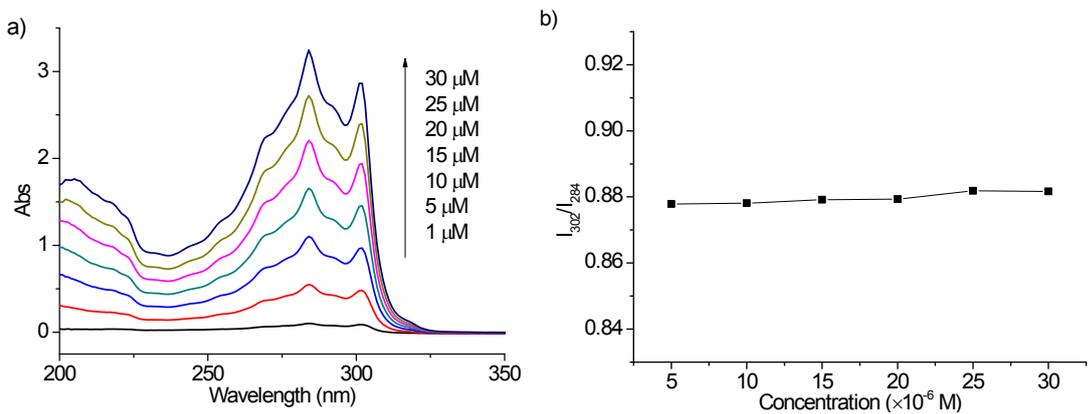


Fig. S52 UV-vis absorption spectra of **3** in cyclohexane at different concentrations.

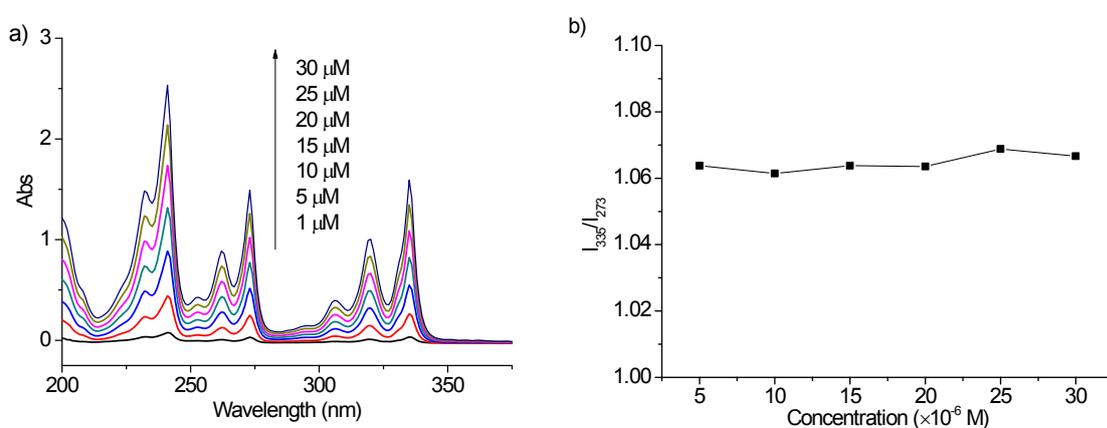


Fig. S53 UV-vis absorption spectra of pyrene in cyclohexane at different concentrations.

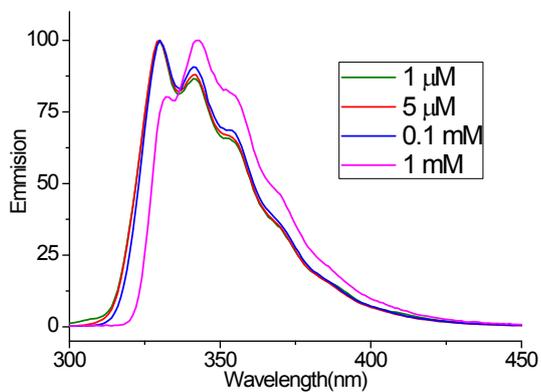


Fig. S54 Emission spectra of **3** in cyclohexane at different concentration.

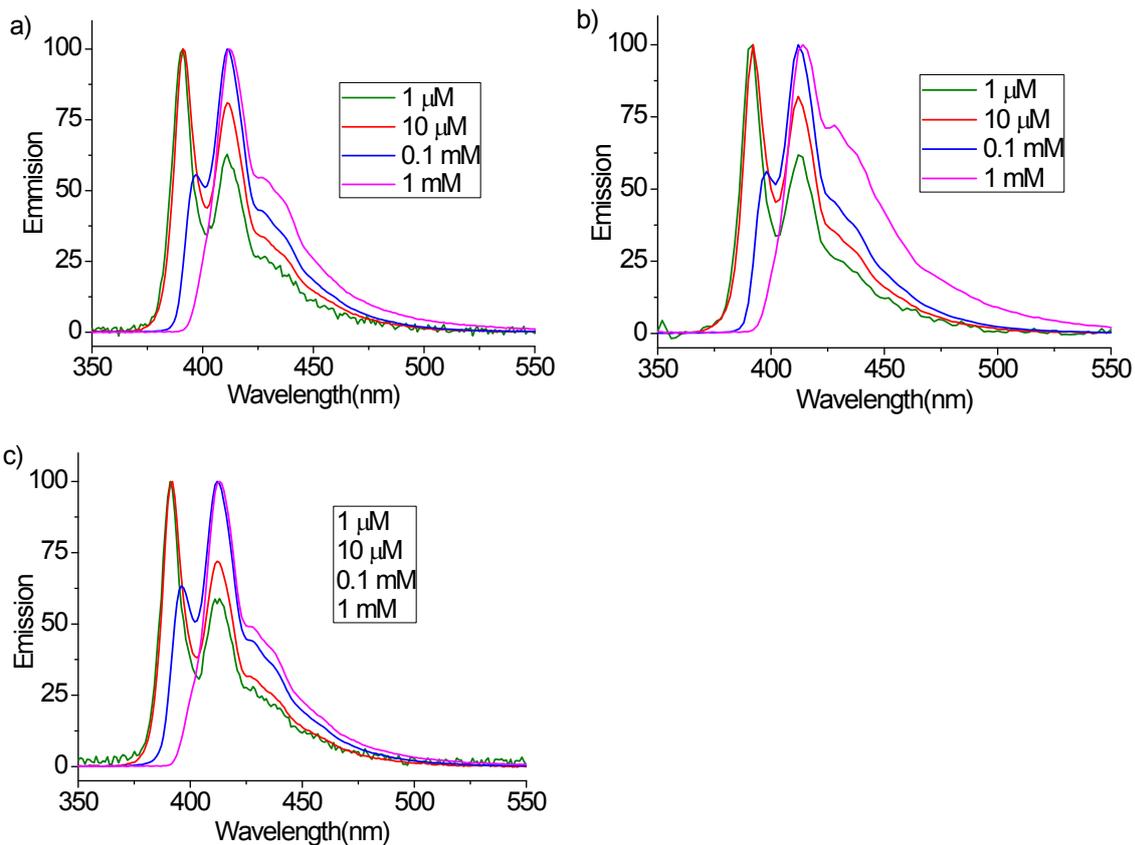


Fig. S55 Emission spectra of a) **1b**, b) **1c** and c) **1d** in cyclohexane with increasing concentration from 1×10^{-6} M to 1×10^{-3} M.

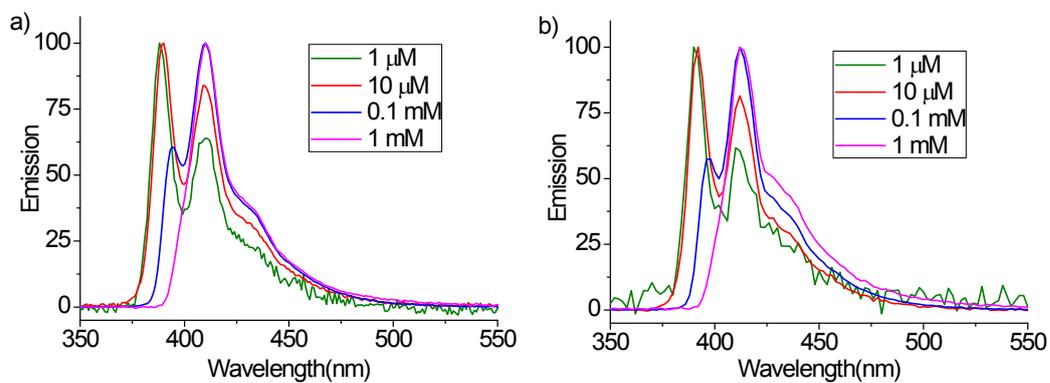


Fig. S56 Emission spectra of a) **2a**, b) **2b** in cyclohexane with increasing concentration from 1×10^{-6} M to 1×10^{-3} M.