Supporting information

Electrocatalytic Linear Coupling of Alkenes via Radical Anion under Mild Conditions

Jingao Xiao, Feng Long, Sheng Yi, Haifang Luo, Changqun Cai, Hang Gong*

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Supplemental Experimental Procedures

General information

All the reagents were purchased from commercial suppliers and used without further purification unless otherwise noted. All the reactions were carried out under air in oven-dried glassware with magnetic stirring. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400, 100 MHz, respectively) at ambient temperature. HRMS were made by means of ESI with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. IR spectra were recorded on a new Fourier transform infrared spectroscopy. Unless otherwise noted, all reagents were weighed and handled in air. Oil bath was used for reactions that require heating. The melting points were measured on a micro melting point apparatus.

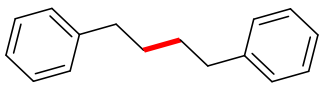
Reaction apparatus diagram

Figure S1. Diagram of reaction device

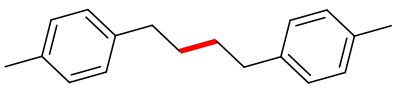
Syntheses and Characterizations of Products

General Procedure of the Coupling Reactions

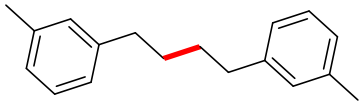
Styrene derivatives (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), and DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. The current intensity was stable at 30-35 mA during the whole reaction. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. Using the mixture of petroleum ether (PE) and ethyl acetate (EA) as a developing agent, the mixture was purified by column chromatography or PTLC to obtain the target product.



1,4-diphenylbutane (2a)[1]: Purified by PTLC; R_f = 0.6 (Pure PE); Yellow oil; from styrene, Isolated yield 66% (13.9 mg); from 1-fluoro-4-vinylbenzene, Isolated yield 43% (9.0 mg); from 1-chloro-4-vinylbenzene, Isolated yield 49% (10.3 mg); from 1-bromo-4-vinylbenzene, Isolated yield 51% (10.8 mg); from ethynylbenzene, Isolated yield 34% (7.2 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.29-7.24 (m, 4H), 7.19-7.15 (m, 6H), 2.63 (t, J = 6.8 Hz 4H), 1.66 (p, J = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 142.7, 128.6, 128.4, 125.8, 36.0, 31.2.

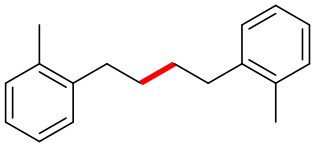


1,4-di-p-tolylbutane (2b)[2]: Purified by PTLC; R_f = 0.5 (Pure PE); White solid; M.p. = 73-75 °C; Isolated yield 72% (17.1 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.07 (dd, J = 11.2 Hz, 8.4 Hz, 4H), 2.59 (t, J = 2.8 Hz, 4H), 2.32 (s, 6H), 1.64 (p, J = 3.6 Hz, 4H);^{13}C NMR (101 MHz, Chloroform-d) δ 139.7, 135.2, 129.1, 128.4, 35.5, 31.4, 21.1.

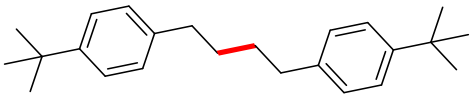


1,4-di-m-tolylbutane (2c)[3]: Purified by PTLC; R_f = 0.5 (Pure PE); Yellow oil; from

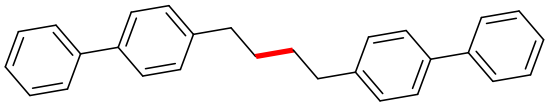
1-methyl-3-vinylbenzene, Isolated yield 75% (17.8 mg); from 1-(trifluoromethyl)-3-vinylbenzene, Isolated yield 18% (4.3 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.17 (t, *J* = 7.6 Hz, 2H), 7.01-6.97 (m, 6H), 2.61 (t, *J* = 6.8 Hz, 4H), 2.33 (s, 6H), 1.67 (t, *J* = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 142.7, 137.9, 129.4, 128.3, 126.5, 125.6, 35.9, 31.3, 21.6.



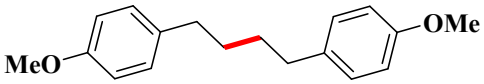
1,4-di-o-tolylbutane (2d): Purified by PTLC; R_f = 0.5 (Pure PE); Yellow oil; Isolated yield 80% (19.0 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.16-7.11 (m, 8H), 2.65 (t, *J* = 7.2 Hz, 4H), 2.32 (s, 6H), 1.68 (p, *J* = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 140.9, 136.0, 130.2, 128.9, 126.0, 125.9, 33.4, 30.3, 19.5. IR: 2930, 2859, 2360, 2340, 1491, 1458, 1259, 1095, 1028, 802, 737, 669; HRMS (ESI): m/z $[M+K]^+$ calcd for $C_{18}H_{22}K$: 277.1353, found 277.1348.



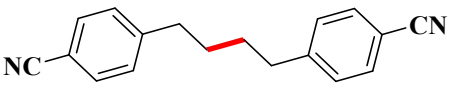
1,4-bis(4-(tert-butyl)phenyl)butane (2e)[4]: Purified by PTLC; R_f = 0.5 (Pure PE); White solid; Isolated yield 80% (25.7 mg); M.p. = 160-162 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.30 (d, *J* = 8.4 Hz, 4H), 7.12 (d, *J* = 8.4 Hz, 4H), 2.61 (t, *J* = 6.4 Hz, 4H), 1.67 (p, *J* = 3.6 Hz, 4H), 1.31 (s, 18H); ^{13}C NMR (101 MHz, Chloroform-d) δ 148.5, 139.7, 128.2, 125.3, 35.4, 34.5, 31.6, 31.3.



1,4-di([1,1'-biphenyl]-4-yl)butane (2f)[2]: Purified by column chromatography; R_f = 0.3 (Pure PE); White solid; M.p. = 262-264 °C ; Isolated yield 97% (35.2 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.62 (d, *J* = 7.6 Hz, 4H), 7.55 (d, *J* = 8.0 Hz, 4H), 7.46 (t, *J* = 7.6 Hz, 4H), 7.36 (t, *J* = 7.2 Hz, 2H), 7.29 (d, *J* = 7.6 Hz, 4H), 2.73 (t, *J* = 6.8 Hz, 4H), 1.77 (p, *J* = 3.2 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 141.8, 141.3, 138.8, 129.0, 128.8, 127.2, 127.1, 35.6, 31.2.

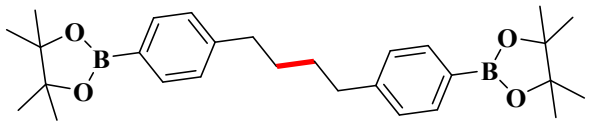


1,4-bis(4-methoxyphenyl)butane (2g)[2]: Purified by PTLC; R_f = 0.3 (PE : EA = 40:1); White solid; M.p. = 78-79 °C; Isolated yield 30% (8.2 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.09 (d, *J* = 8.4 Hz, 4H), 6.82 (d, *J* = 8.0 Hz, 4H), 3.79 (s, 6H), 2.57 (t, *J* = 6.8 Hz, 4H), 1.62 (p, *J* = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 157.7, 134.9, 129.4, 113.8, 55.4, 35.0, 31.4.

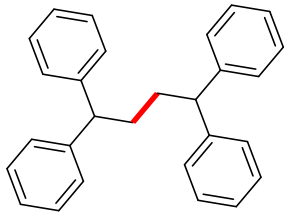


4-(4-(4-isocyanophenyl)butyl)benzonitrile (2h)[5]: Purified by PTLC; R_f = 0.4 (PE :

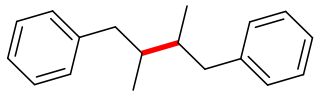
EA = 4:1); White solid; M.p. = 132-134 °C; Isolated yield 45% (11.7 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.57 (d, *J* = 8.0 Hz, 4H), 7.26 (d, *J* = 8.0 Hz, 4H), 2.69 (d, *J* = 6.4 Hz, 4H), 1.67 (p, *J* = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 147.9, 132.3, 129.3, 119.2, 109.8, 36.0, 30.5.



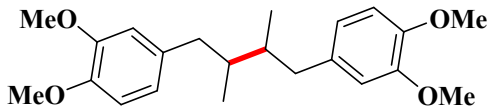
1,4-bis(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butane (2i)[2]: Purified by PTLC; R_f = 0.3 (PE : EA = 20:1); Yellow oil; Isolated yield 55% (25.2 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.72 (d, *J* = 7.6 Hz, 4H), 7.17 (d, *J* = 7.6 Hz, 4H), 2.63 (t, *J* = 6.8 Hz, 4H), 1.65 (p, *J* = 3.2 Hz, 4H), 1.34 (s, 24H); ^{13}C NMR (101 MHz, Chloroform-d) δ 146.1, 135.0, 128.0, 83.8, 36.1, 31.0, 25.0.



1,1,4,4-tetraphenylbutane (2j): Purified by PTLC; R_f = 0.3 (Pure PE); White solid; M.p. = 126-127 °C; Isolated yield 63% (22.7 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.28-7.25 (m, 8H), 7.20-7.15 (m, 12H), 3.93 (t, *J* = 5.4 Hz, 2H), 2.04 (t, *J* = 3.2 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 145.0, 128.5, 127.9, 126.2, 51.6, 34.2. IR: 3335, 2976, 2360, 2340, 1649, 1454, 1383, 1085, 1043, 877, 669, 603; HRMS (ESI): m/z $[M+Na]^+$ calcd for $C_{28}H_{26}Na$: 385.1927, found 385.1934.

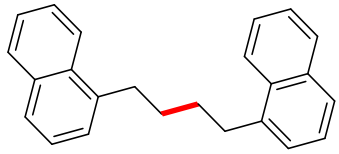


2.3-dimethyl-1,4-diphenylbutan (2k): Purified by PTLC; R_f = 0.6 (Pure PE); Yellow oil; Isolated yield 74% (17.5 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.32-7.26 (m, 2H), 7.22-7.17 (m, 2H), 7.12 (d, *J* = 7.6 Hz, 3H), 2.85 (dd, *J* = 13.2, 4.8 Hz, 0.4H), 2.68 (dd, *J* = 13.6, 6.0 Hz, 1.6H), 2.45 (dd, *J* = 13.6, 8.4 Hz, 1.6H), 2.38 (dd, *J* = 13.2, 9.2 Hz, 0.4H), 1.87-1.79 (m, 2H), 0.86 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (101 MHz, Chloroform-d) δ 142.0, 141.8, 129.3, 129.2, 128.3, 128.3, 125.8, 125.7, 41.5, 39.5, 39.5, 38.3, 16.3, 14.1. IR: 3025, 2958, 2925, 2872, 2360, 2340, 1601, 1493, 1453, 1378, 1259, 1093, 1063, 1029, 804, 740, 696; HRMS (ESI): m/z $[M+NH_4]^+$ calcd for $C_{18}H_{26}N$: 256.2060, found 256.2059.

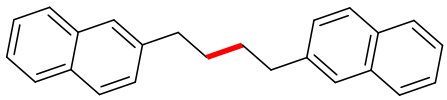


1,4-bis(3,4-dimethoxyphenyl)-2,3-dimethylbutane (2l): Purified by PTLC; R_f = 0.2 (PE : EA = 20:1); Yellow oil; Isolated yield 40% (14.3 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 6.76 (d, J = 8.4 Hz, 2H), 6.71-6.58 (m, 4H), 3.83 (d, *J* = 14.4 Hz, 12H), 2.75 (dd, *J* = 13.2, 5.2 Hz, 0.3H), 2.55 (dd, *J* = 14, 7.2 Hz, 1.7H), 2.40 (dd, *J* = 13.6, 7.6 Hz, 0.3H), 2.40 (dd, *J* = 13.6, 9.2 Hz, 1.7H), 1.78-1.74 (m, 2H), 0.83 (d, *J* = 6.4 Hz,

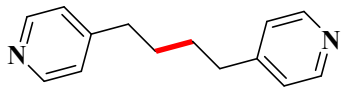
6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 148.7, 147.0, 134.3, 120.9, 112.1, 110.9, 56.0, 55.8, 41.1, 39.3, 38.9, 37.7, 16.3, 13.9. IR: 2995, 2954, 2931, 2872, 2833, 2360, 2340, 1605, 1588, 1512, 1459, 1416, 1377, 1327, 1258, 1233, 1190, 1153, 1139, 1027, 942, 846, 804, 764, 743, 669, 644; HRMS (ESI): m/z $[M+Na]^+$ calcd for $C_{20}H_{30}O_4Na$: 381.2036, found 381.2048.



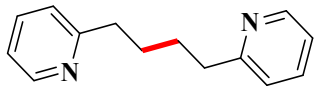
1,4-di(naphthalen-1-yl)butane (2m): Purified by column chromatography; R_f = 0.2 (Pure PE); White solid; M.p. = 106-107 °C; Isolated yield 74% (22.8 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 8.06 (d, *J* = 7.6 Hz, 2H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.54-7.47 (m, 4H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.33 (d, *J* = 7.2 Hz, 2H), 3.14 (t, *J* = 6.4 Hz, 4H), 1.93 (p, *J* = 3.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 138.7, 134.0, 132.0, 128.9, 126.6, 126.1, 125.8, 125.7, 125.5, 124.0, 33.1, 31.0. IR: 3734, 2360, 2340, 669, 651; HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{24}H_{23}$: 311.1794, found 311.1792.



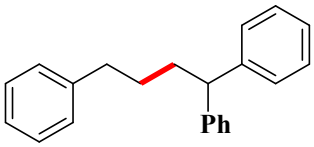
1,4-di(naphthalen-2-yl)butane (2n): Purified by column chromatography; R_f = 0.3 (Pure PE); White solid; M.p. = 164-165 °C; Isolated yield 98% (30.4 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.80 (dd, *J* = 16.4, 7.2 Hz, 6H), 7.62 (s, 2H), 7.48-7.41 (m, 4H), 7.34 (d, *J* = 8.4 Hz, 2H), 2.83 (t, *J* = 6.4 Hz, 4H), 1.81 (p, *J* = 3.2 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 140.2, 133.7, 132.1, 127.9, 127.7, 127.5, 126.5, 126.0, 125.2, 36.1, 31.1. IR: 3734, 2360, 2340, 669, 651; HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{24}H_{23}$: 311.1794, found 311.1792.



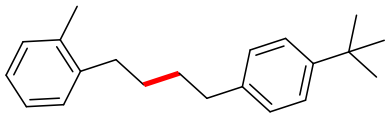
1,4-di(pyridin-4-yl)butane (2o)[2]: Purified by PTLC; R_f = 0.3 (PE : EA = 3:1); Yellow oil; Isolated yield 41% (8.7 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 8.48 (d, *J* = 6.4 Hz, 4H), 7.08 (d, *J* = 6.0 Hz, 4H), 2.63 (t, *J* = 7.2 Hz, 4H), 1.67 (p, *J* = 3.6 Hz, 4H).^{13}C NMR (101 MHz, Chloroform-d) δ 151.2, 149.8, 124.0, 35.1, 29.8.



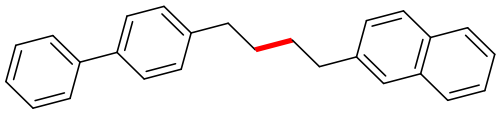
1,4-di(pyridin-2-yl)butane (2p) [2]: Purified by column chromatography; R_f = 0.2 (PE : EA = 3:1); Yellow oil; Isolated yield 33% (7.0 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 8.50 (d, *J* = 6.0 Hz, 2H), 7.56 (t, *J* = 7.6 Hz, 2H), 7.13-7.06 (m, 4H), 2.82 (t, *J* = 7.6 Hz, 4H), 1.80 (p, *J* = 7.6 Hz, 4H); ^{13}C NMR (101 MHz, Chloroform-d) δ 162.2, 149.3, 136.5, 122.9, 121.1, 38.3, 29.7.



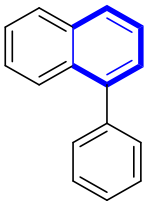
butane-1,1,4-triyltribenzene (2q)[6]: Purified by PTLC; R_f = 0.4 (Pure PE); White solid; M.p. = 78-79 °C; Isolated yield 60% (34.3 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.11 (m, 15H), 3.93 (td, *J* = 7.8, 2.5 Hz, 1H), 2.70 – 2.60 (m, 2H), 2.10 (dd, *J* = 7.7, 2.8 Hz, 2H), 1.62 (td, *J* = 7.4, 3.0 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-d) δ 145.1, 142.4, 128.5, 128.4, 128.3, 127.9, 126.1, 125.7, 51.3, 35.9, 35.3, 29.9.



1-(4-(4-(tert-butyl)phenyl)butyl)-2-methylbenzene (2r): Purified by PTLC; R_f = 0.4 (Pure PE); Yellow oil; Isolated yield 54% (30.2 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33-7.26 (m, 2H), 7.12 (td, *J* = 4.9, 2.6 Hz, 6H), 2.65-2.58 (m, 4H), 2.29 (d, *J* = 5.6 Hz, 3H), 1.72-1.61 (m, 4H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-d) δ 148.4, 140.8, 139.5, 135.9, 130.2, 128.8, 128.1, 125.9, 125.8, 125.2, 35.4, 34.4, 33.3, 31.5, 30.3, 30.0, 19.4. IR: 3720, 2930, 2859, 1492, 1460; 827, 730; HRMS (ESI): m/z [M+2H_2O-H]$^-$ calcd for $C_{21}H_{31}O_2$: 315.2330, found 315.2350.

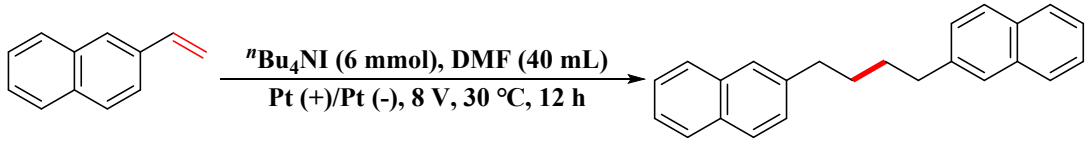


2-(4-([1,1'-biphenyl]-4-yl)butyl)naphthalene (2s): Purified by PTLC; R_f = 0.4 (Pure PE); Yellow oil; Isolated yield 65% (43.7 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.74 (m, 2H), 7.60 (d, *J* = 7.8 Hz, 4H), 7.53 (dd, *J* = 8.0, 3.7 Hz, 3H), 7.44 (t, *J* = 7.5 Hz, 4H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.27 (dd, *J* = 6.4, 3.8 Hz, 1H), 2.84 (t, *J* = 7.1 Hz, 1H), 2.76 – 2.67 (m, 3H), 1.78 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.7, 141.2, 140.1, 138.7, 133.6, 132.0, 128.9, 128.7, 127.8, 127.6, 127.4, 127.1, 127.0, 126.4, 125.9, 125.1, 36.0, 35.5, 31.1, 31.0. IR: 3742, 3680, 3031, 2923, 2852, 1598, 1487, 1006, 756, 731, 690; HRMS (ESI): m/z [M+Li]$^+$ calcd for $C_{26}H_{24}Li$: 343.2033, found 343.2056.



1-phenylnaphthalene (3)[7]: Purified by PTLC; R_f = 0.6 (Pure PE); White solid; M.p. = 45-46 °C; Isolated yield 70% (28.5 mg); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.56-7.42 (m, 9H). ^{13}C NMR (101 MHz, Chloroform-d) δ 140.8, 140.4, 133.9, 131.7, 130.2, 128.4, 127.8, 127.4, 127.1, 126.2, 125.9, 125.5.

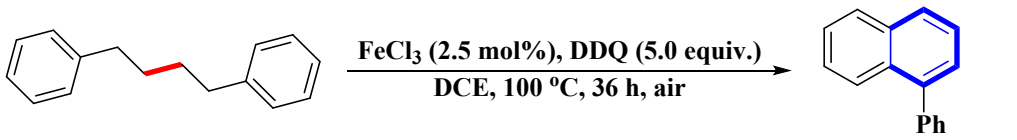
Scaled-up preparation of 2n



1n (6 mmol, 0.92 g) **2n (78%, 724 mg)**

2-vinylnaphthalene (6 mmol), tetrabutylammonium iodide (6 mmol, 1 equiv.), and DMF (40 mL) were added to a three necked flask (100 mL), and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution. The flask was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 12 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 50 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 50 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 50 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. Using the mixture of PE and EA as a developing agent, the mixture was purified by column chromatography to obtain the target product.

Transformation of 2a



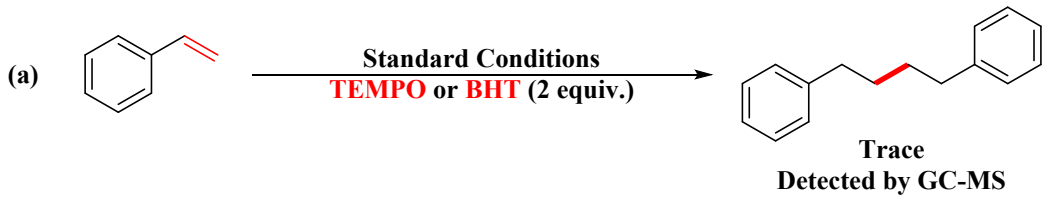
2a **3 (70%) Anti-cancer**

1,4-Diphenylbutane (0.2 mmol), ferric chloride (2.5 mol%), DDQ (1 mmol, 5.0 equiv.), and DCE (2 mL) were added to a 10 mL reaction tube. The sealed reaction tube was placed in an oil bath at 100 ℃, and the reaction was carried out at constant pressure for 36 hours under magnetic stirring. After the reaction is completed, the electrode was taken out after cooling to room temperature, and the reaction solution was filtered with diatomaceous earth. The filtrate was transferred to a separatory funnel and washed three times with saturated saline (10 mL). The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude

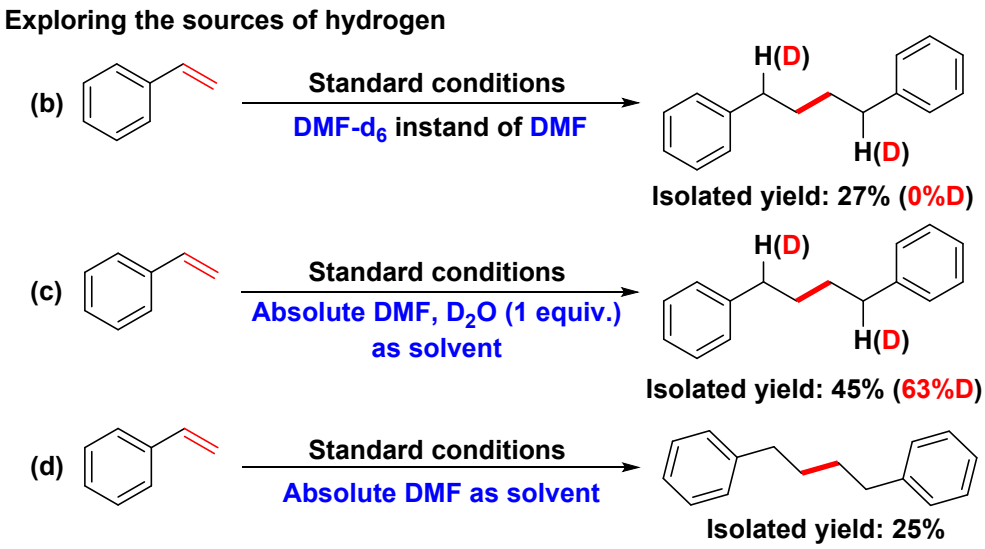
product. Using the mixture of PE and EA as a developing agent, the mixture was purified by PTLC to obtain the target product.

Control experiments

Radical inhibition experiment



Styrene (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), TEMPO (0.4 mmol, 2 equiv.) or BHT (0.4 mmol, equiv.) and DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. The obtained mixture was detected by GC-MS.



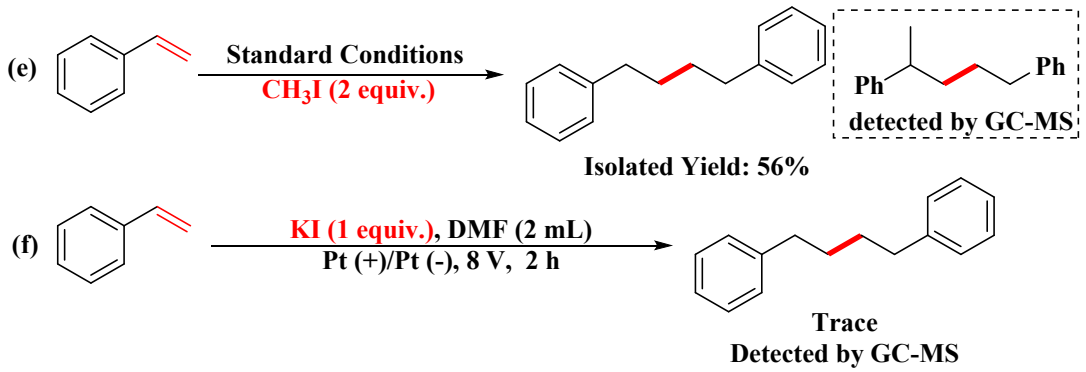
For (eq. b): Styrene (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), and DMF-d_6 (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. Using the mixture of PE and EA as a developing agent, the mixture was purified by PTLC to obtain the target product.

For (eq. c): Styrene (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), D_2O (0.2 mmol), and dry DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed into the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated

saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. Using the mixture of PE and EA as a developing agent, the mixture was purified by PTLC to obtain the target product.

For (eq. d): Styrene (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), and dry DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed into the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. Using the mixture of PE and EA as a developing agent, the mixture was purified by PTLC to obtain the target product.

Exploring the hydrogenation process



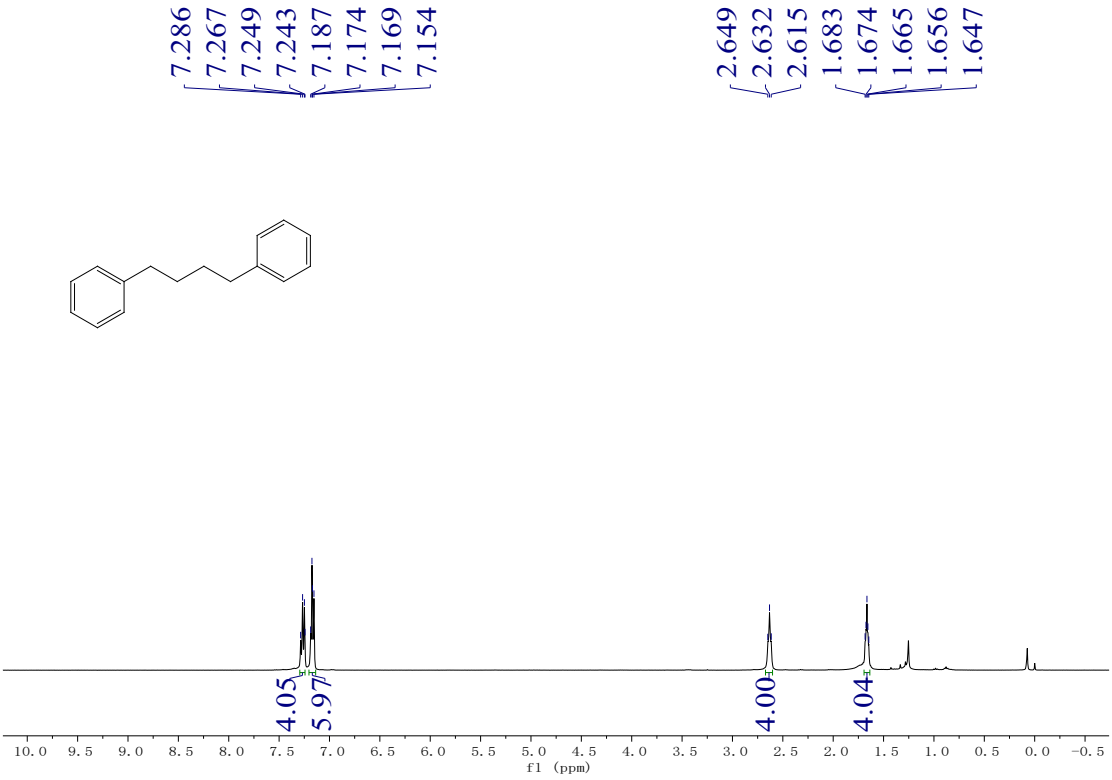
For (eq. e): Styrene (0.2 mmol), tetrabutylammonium iodide (0.2 mmol, 1 equiv.), CH_3I (0.4 mmol), and DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution.

The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. The obtained mixture was detected by GC-MS. Then, using the mixture of PE and EA as a developing agent, the mixture was purified by PTLC to obtain the target product.

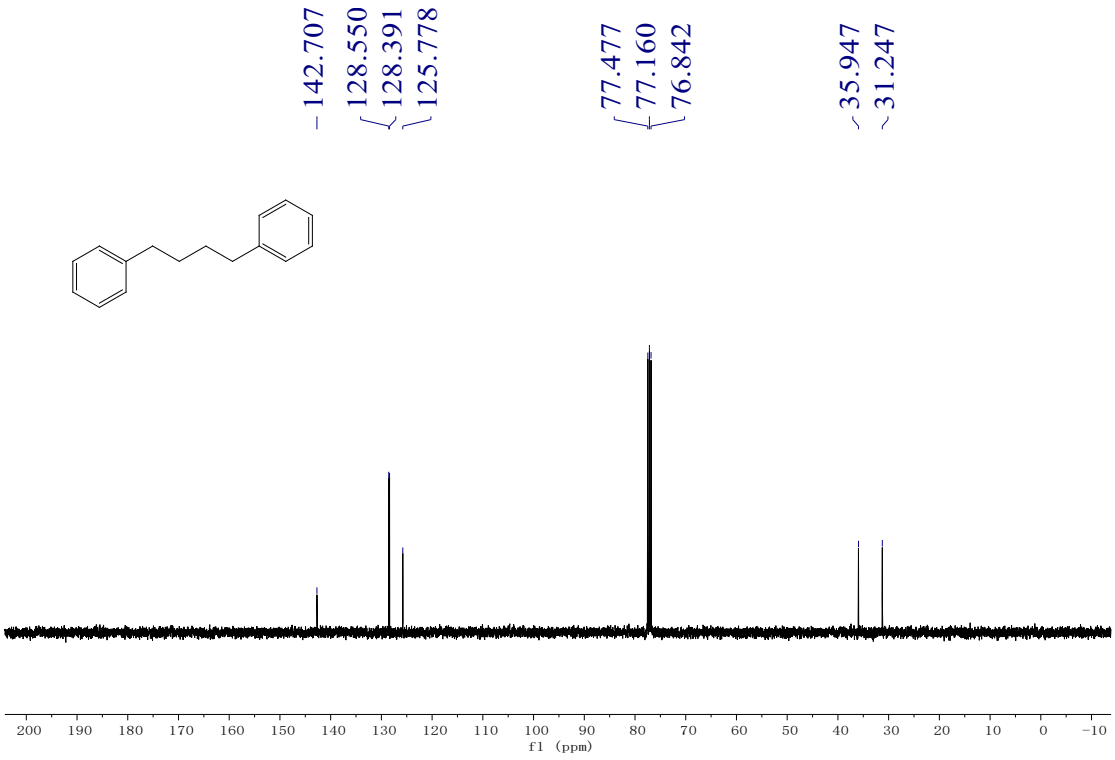
For (eq. f): Styrene (0.2 mmol), KI (0.2 mmol, 1 equiv.), and DMF (2 mL) were added to a 10 mL reaction tube, and two platinum electrodes (10 mm x 10 mm x 0.1 mm) were immersed in the reaction solution. The sealed reaction tube was placed in an oil bath at 30 ℃, and the reaction was carried out at constant pressure for 2 hours under magnetic stirring. After the reaction was completed, the electrode was taken out after cooling to room temperature, and the reaction solution is transferred to a separatory funnel. Then, 10 mL of saturated saline was added to the mixture, and the extraction was repeated three times with 10 mL of ethyl acetate, and the obtained organic phase were combined. After that, the organic phase was washed with 10 mL of saturated saline solution. The separated organic phase was dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum to obtain the crude product. The obtained mixture was detected by GC-MS.

Copies of ^{1}H and ^{13}C NMR spectra

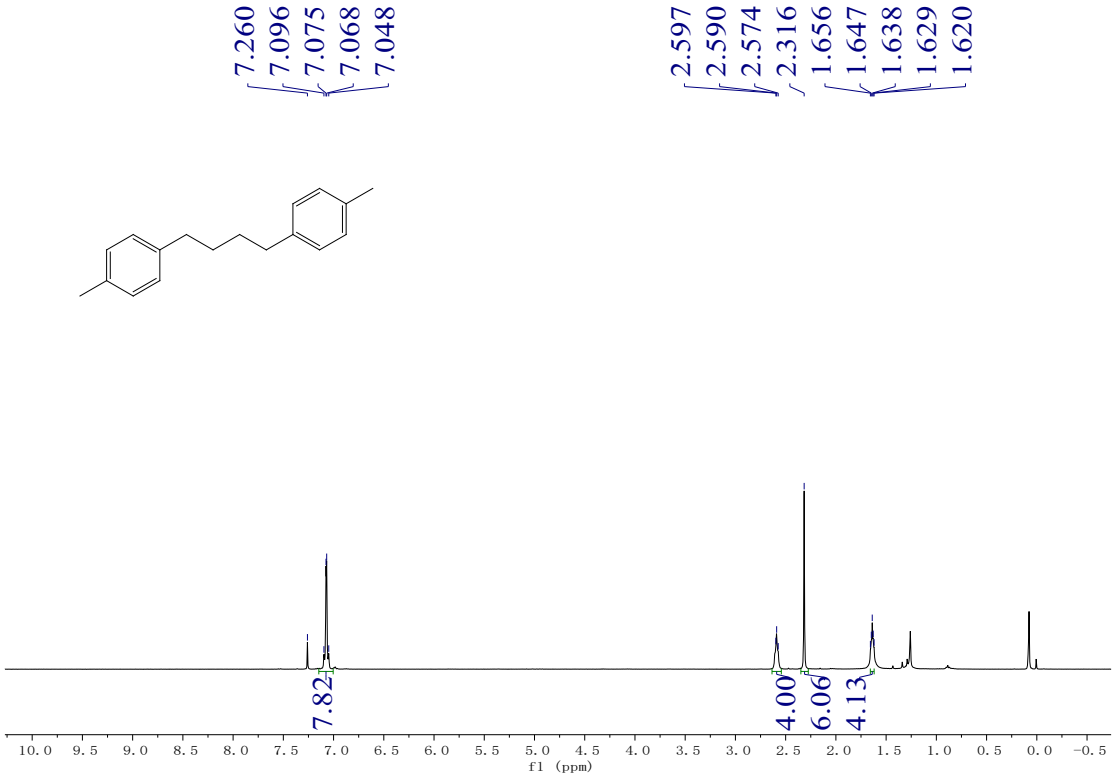
^{1}H NMR spectrum of 2a (400 MHz, $CDCl_3$)



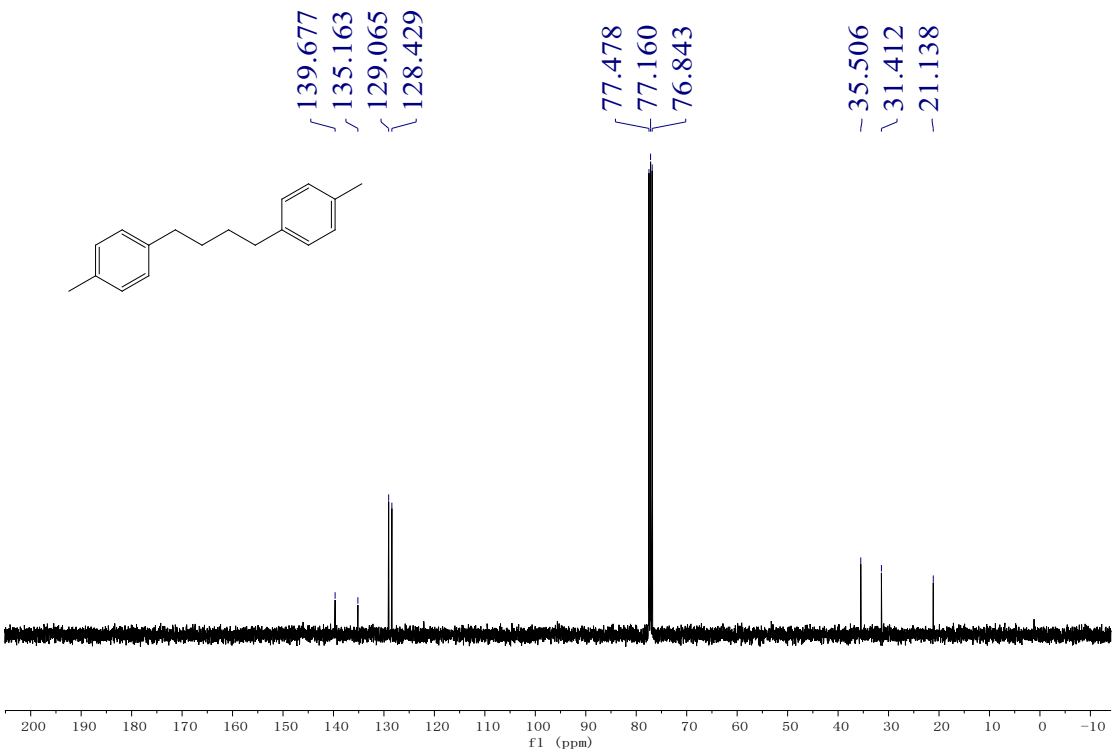
^{13}C NMR spectrum of 2a (100 MHz, $CDCl_3$)



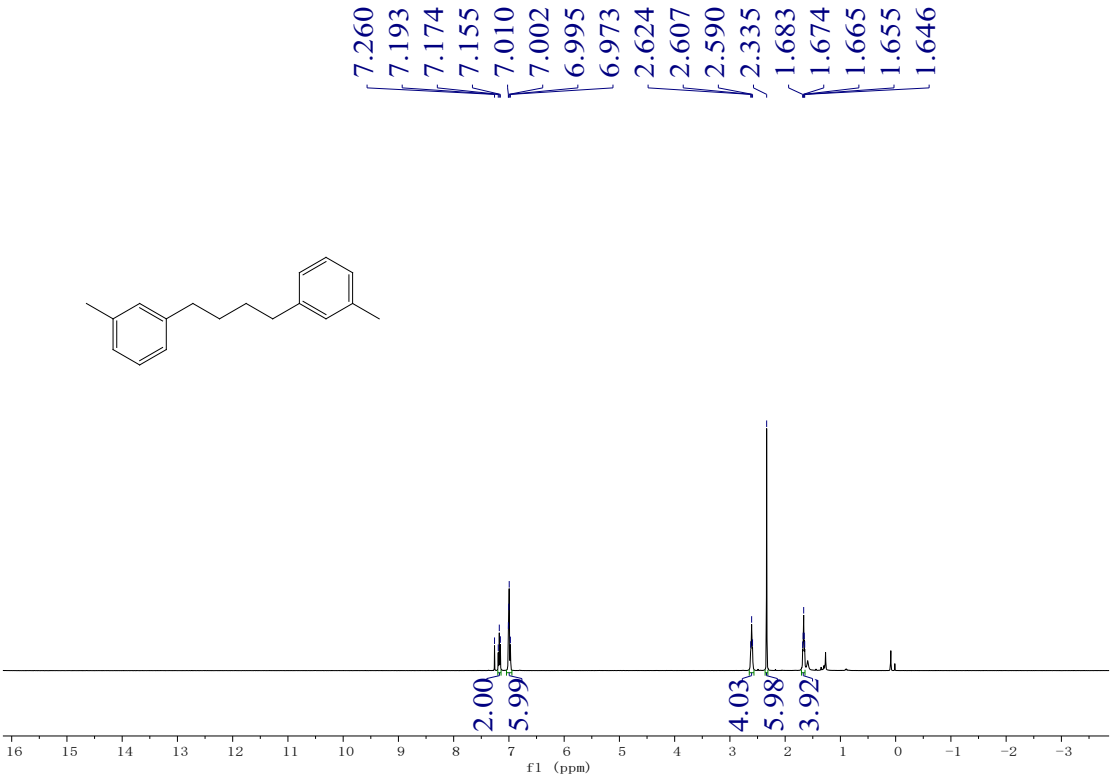
^{1}H NMR spectrum of 2b (400 MHz, $CDCl_3$)



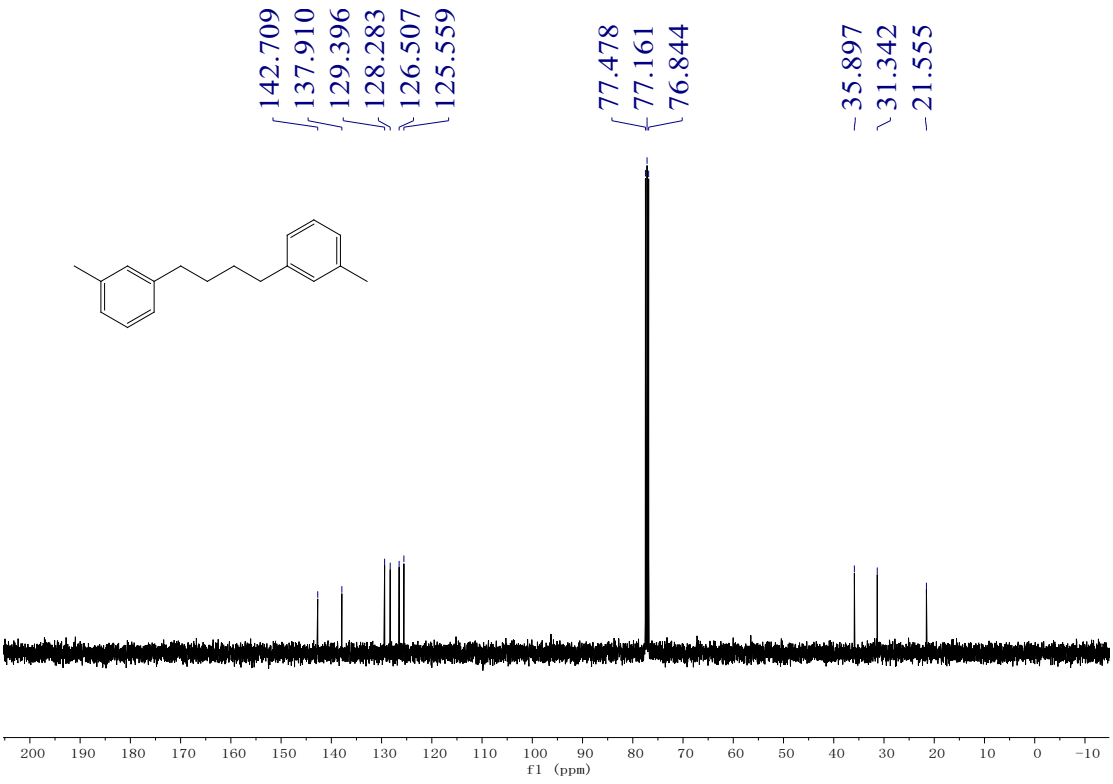
^{13}C NMR spectrum of 2b (100 MHz, $CDCl_3$)


139.677
135.163
129.065
128.429
77.478
77.160
76.843
35.506
31.412
21.138
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

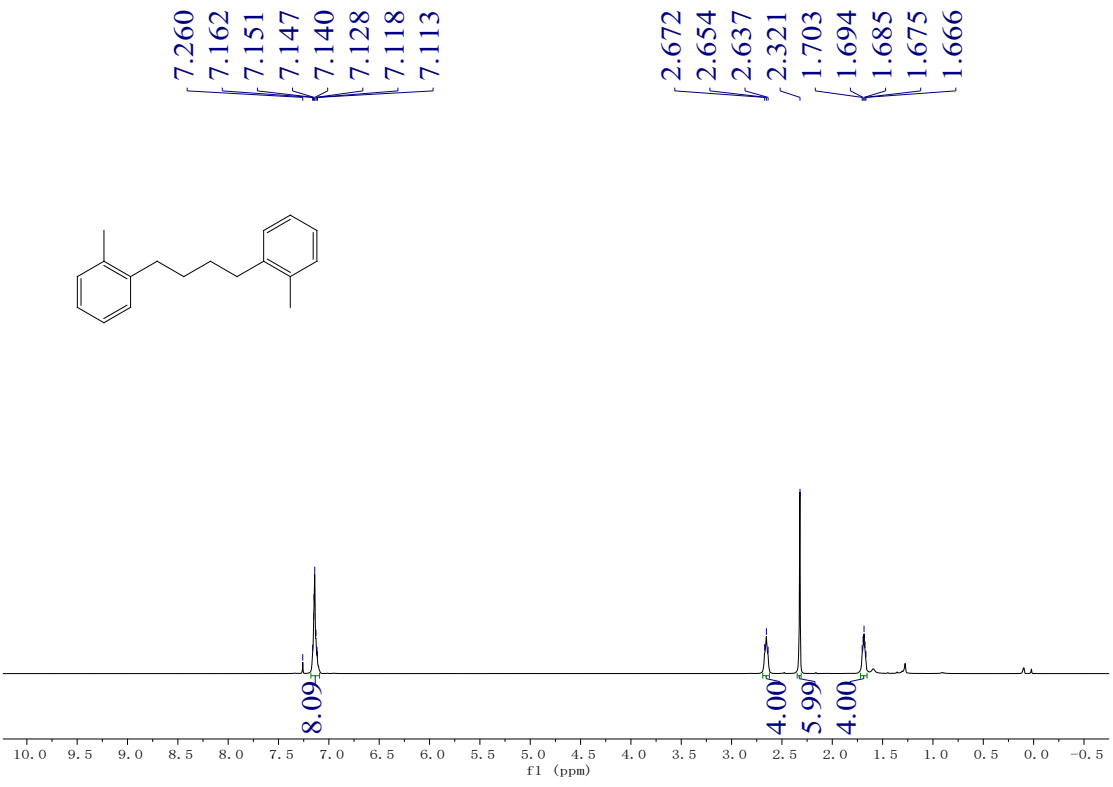
^{1}H NMR spectrum of 2c (400 MHz, $CDCl_3$)



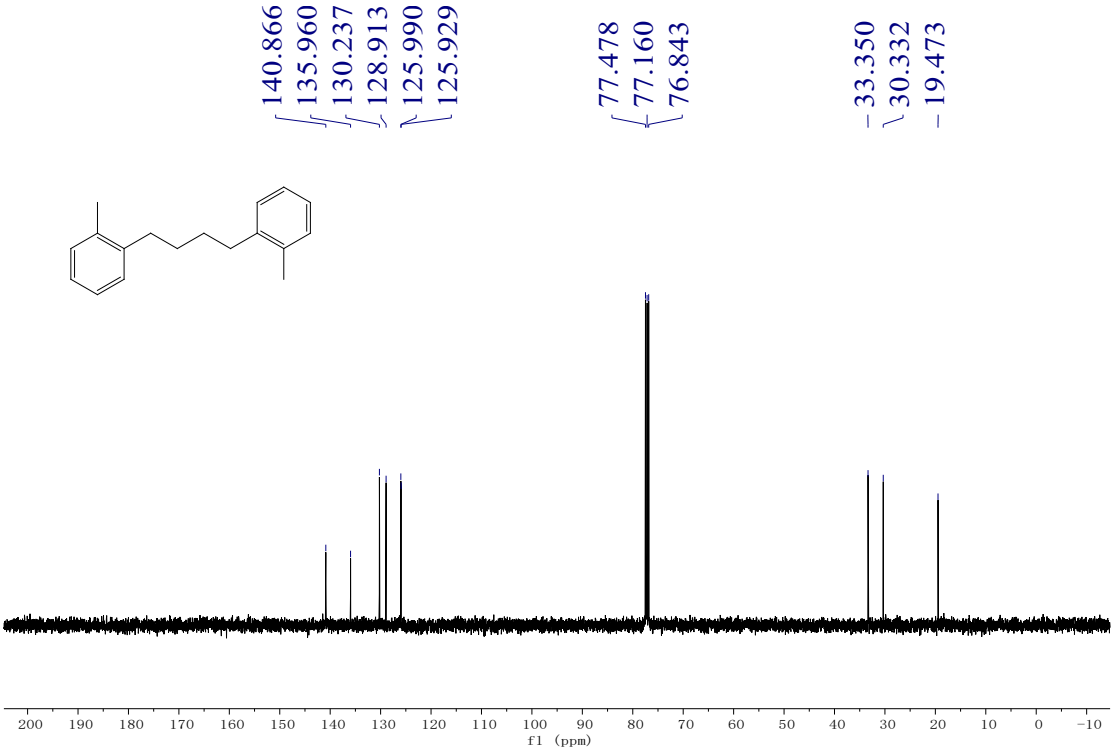
^{13}C NMR spectrum of 2c (100 MHz, $CDCl_3$)



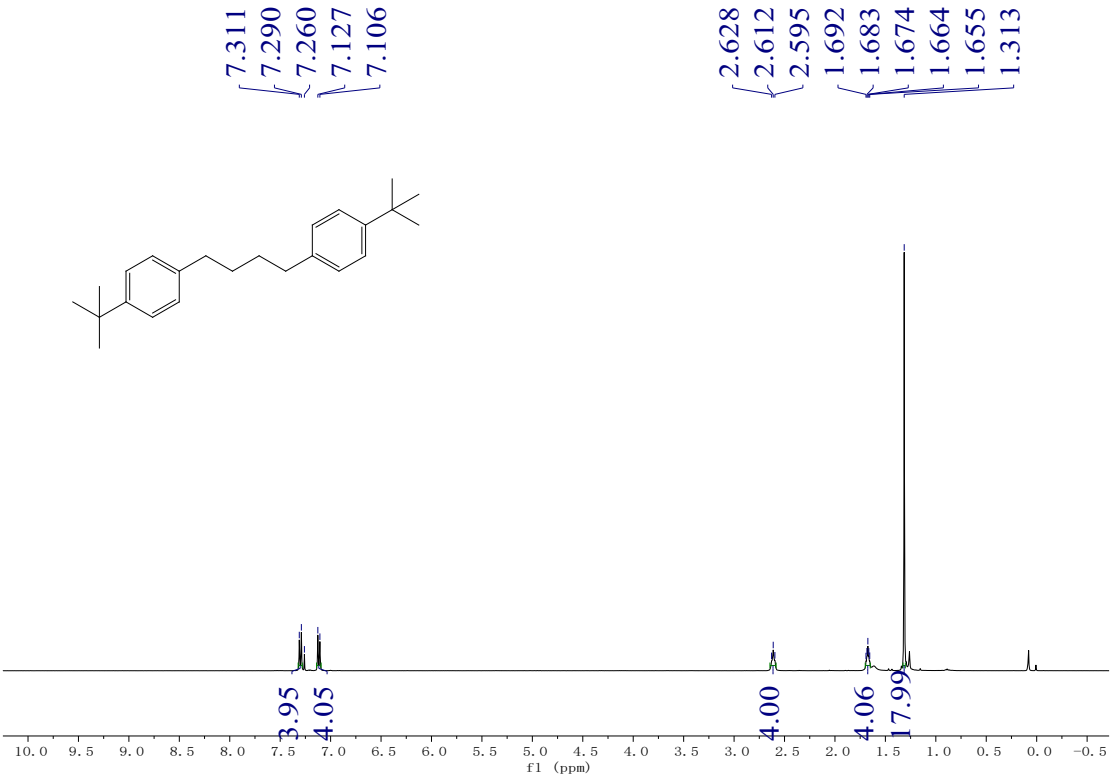
^{1}H NMR spectrum of 2d (400 MHz, $CDCl_3$)



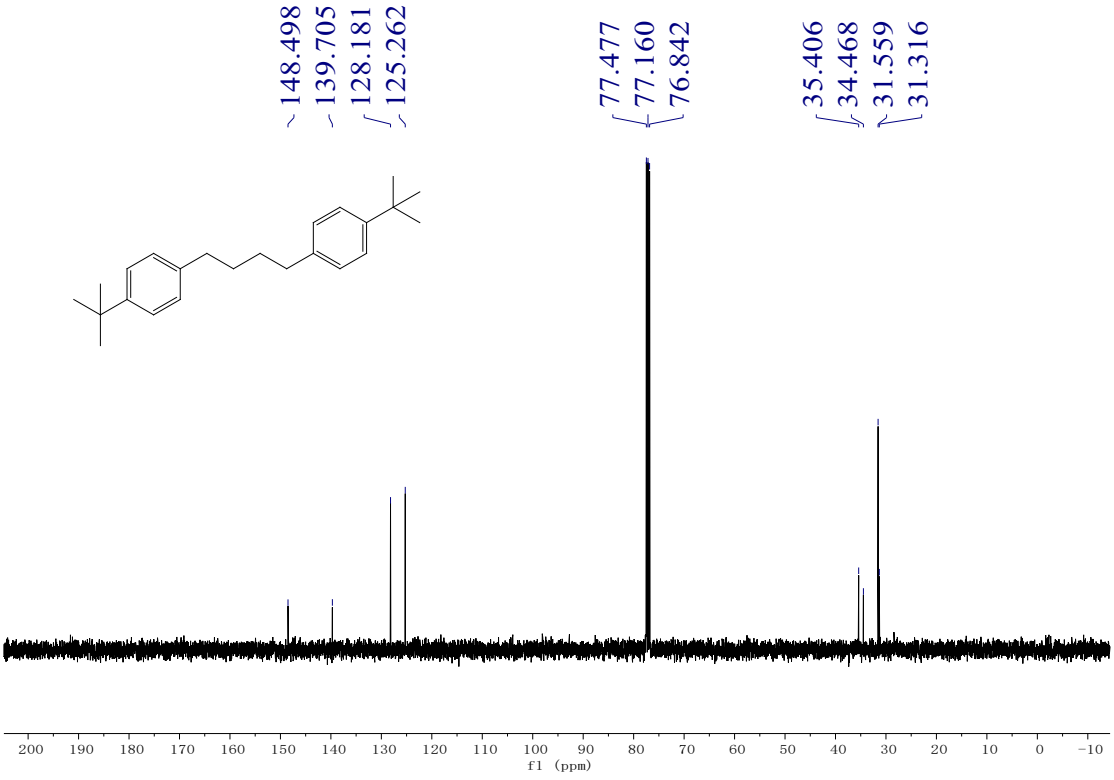
^{13}C NMR spectrum of 2d (100 MHz, $CDCl_3$)



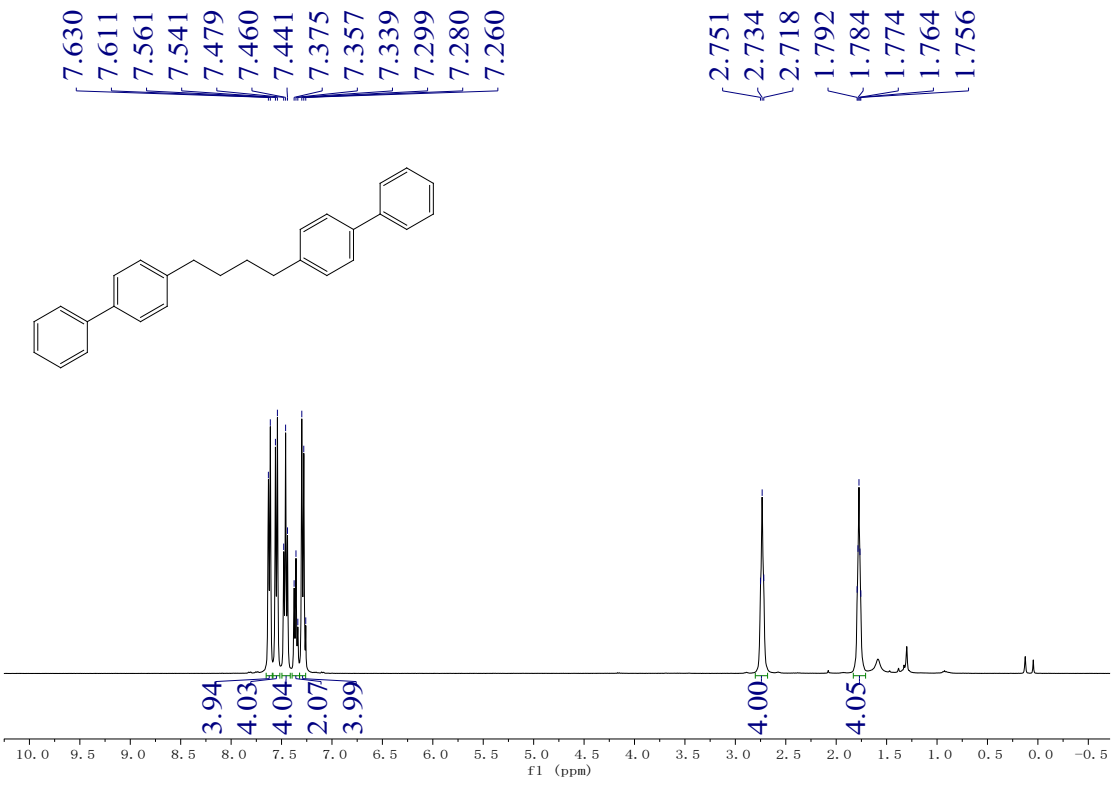
^{1}H NMR spectrum of 2e (400 MHz, $CDCl_3$)



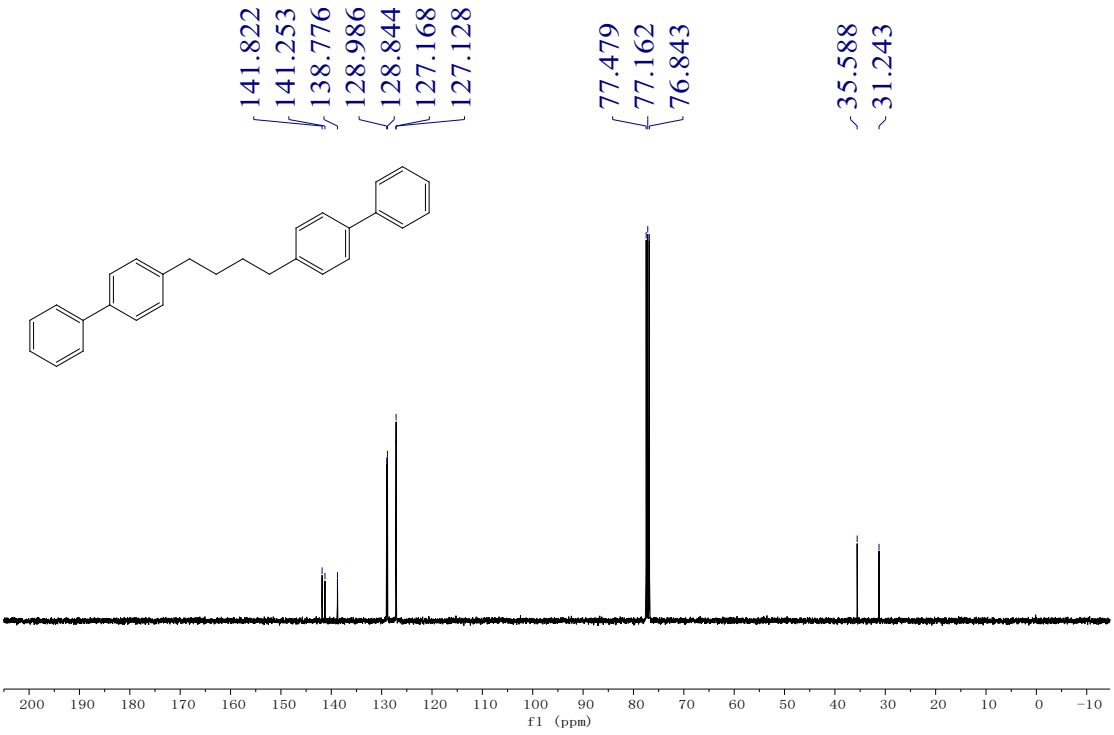
^{13}C NMR spectrum of 2e (100 MHz, $CDCl_3$)



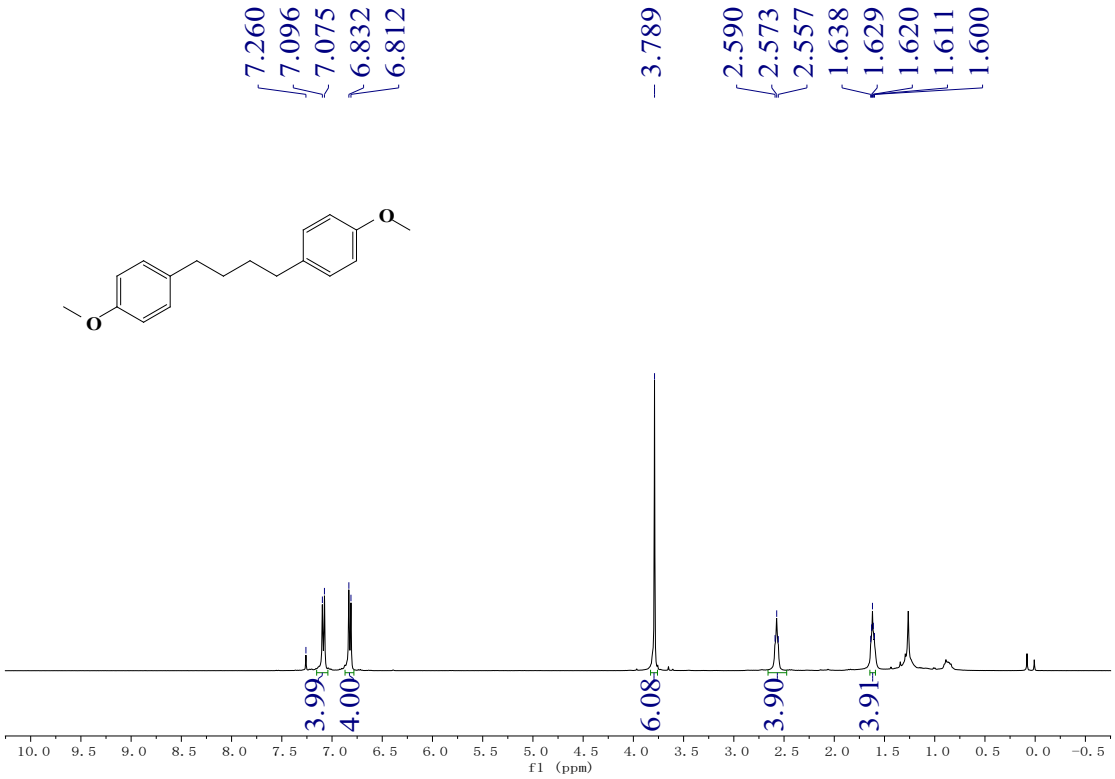
^{1}H NMR spectrum of 2f (400 MHz, $CDCl_3$)



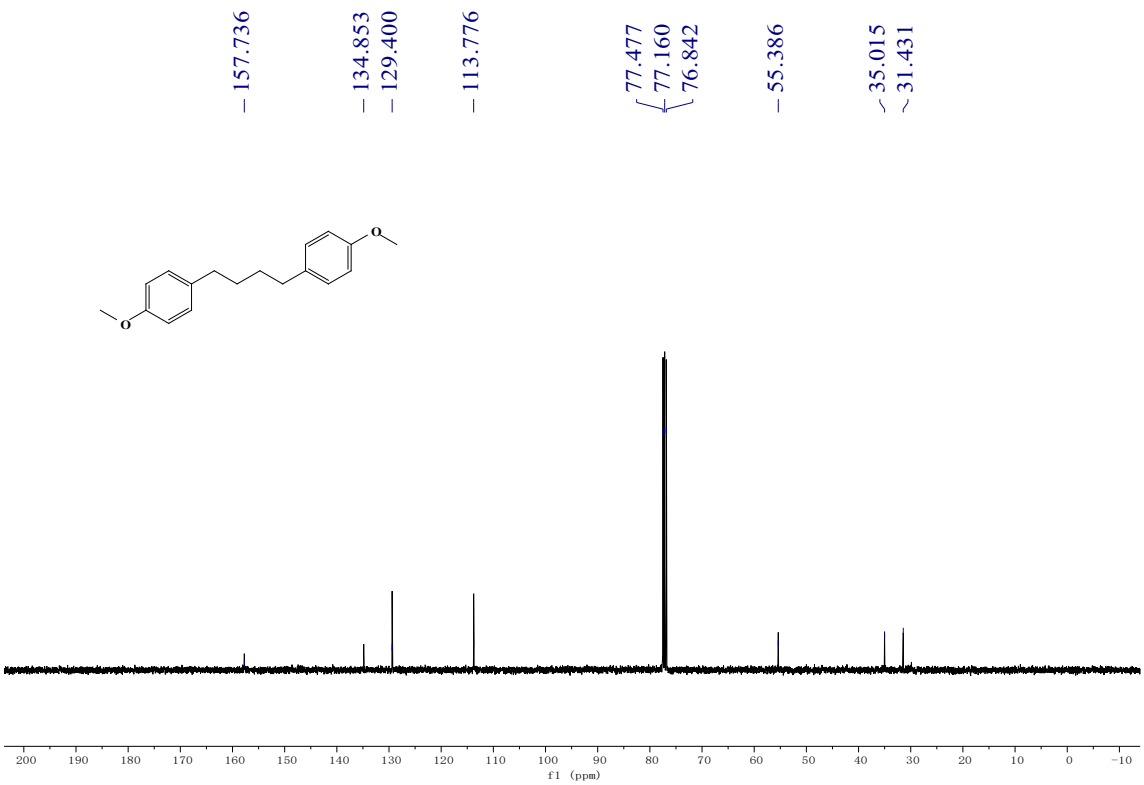
^{13}C NMR spectrum of 2f (100 MHz, $CDCl_3$)



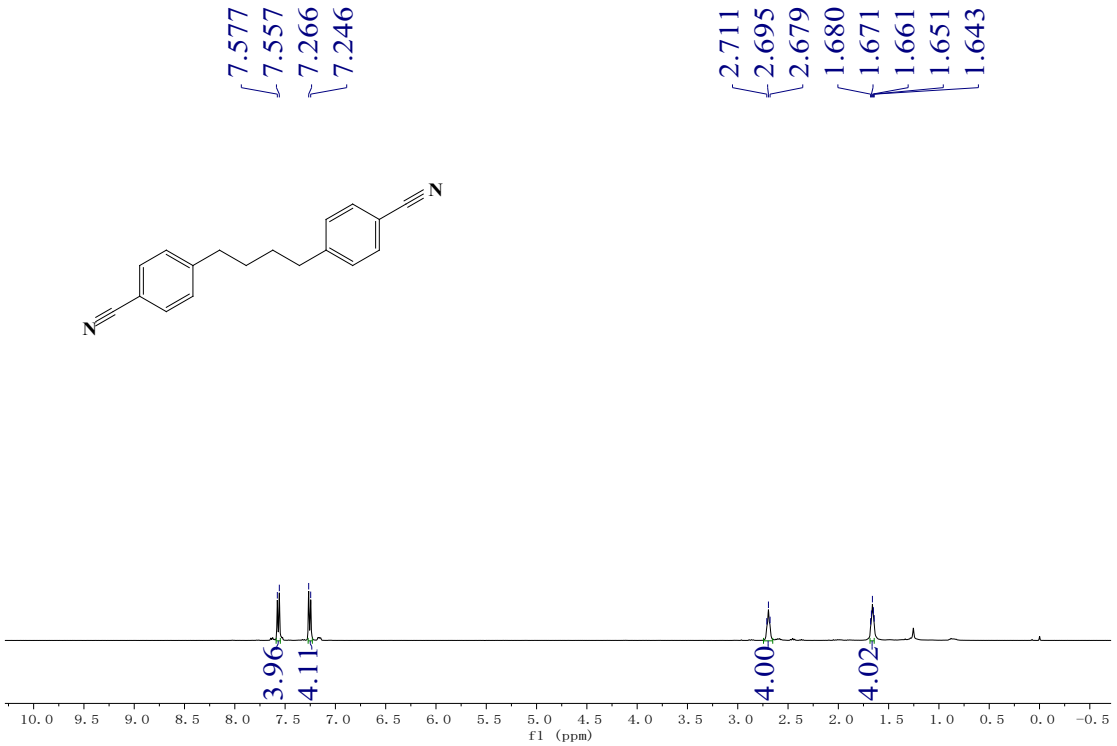
^{1}H NMR spectrum of 2g (400 MHz, $CDCl_3$)



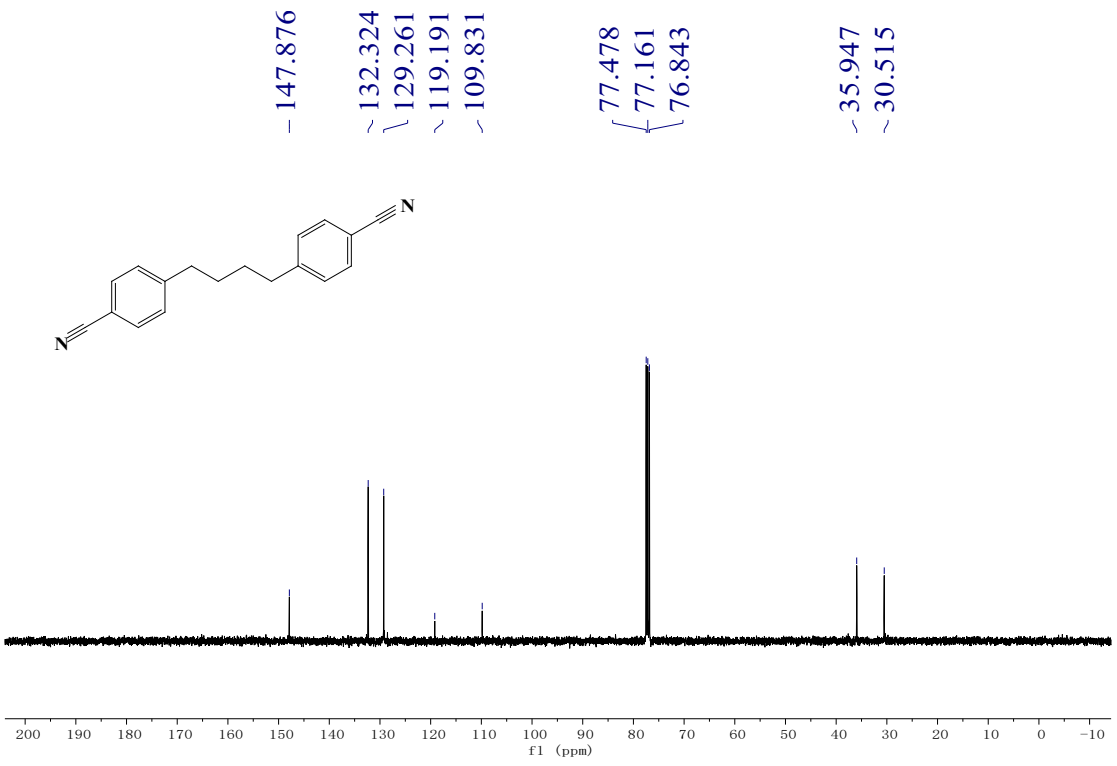
^{13}C NMR spectrum of 2g (100 MHz, $CDCl_3$)



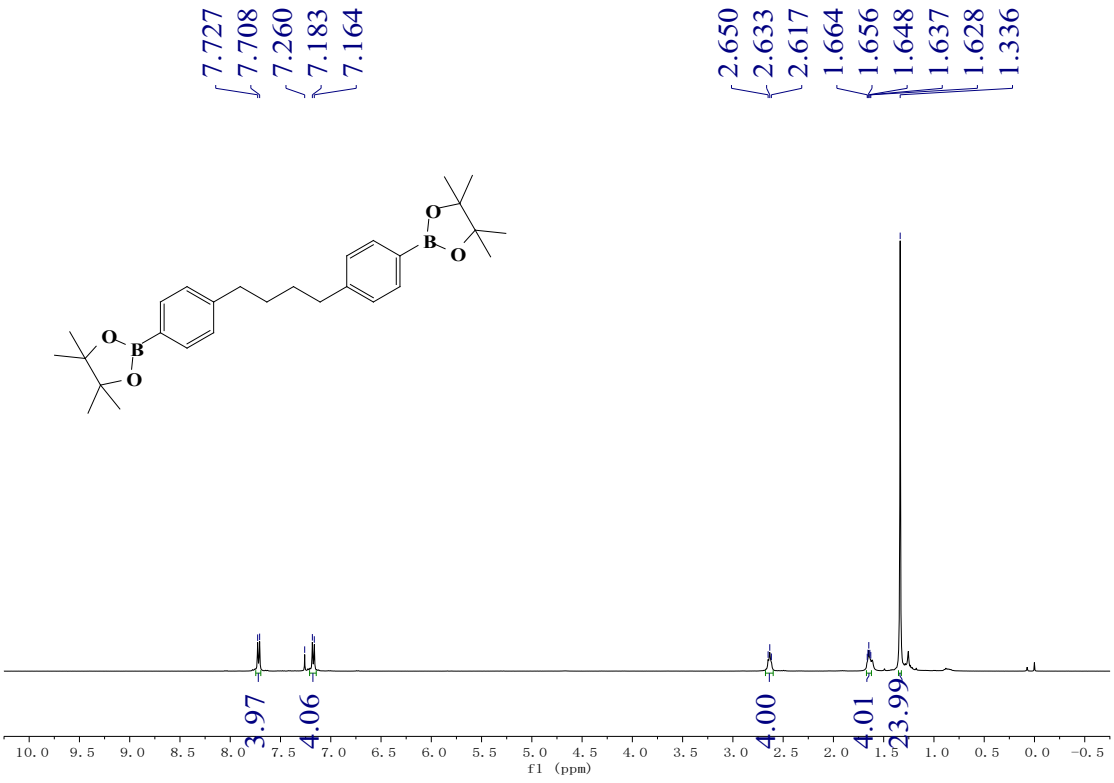
^{1}H NMR spectrum of 2h (400 MHz, $CDCl_3$)



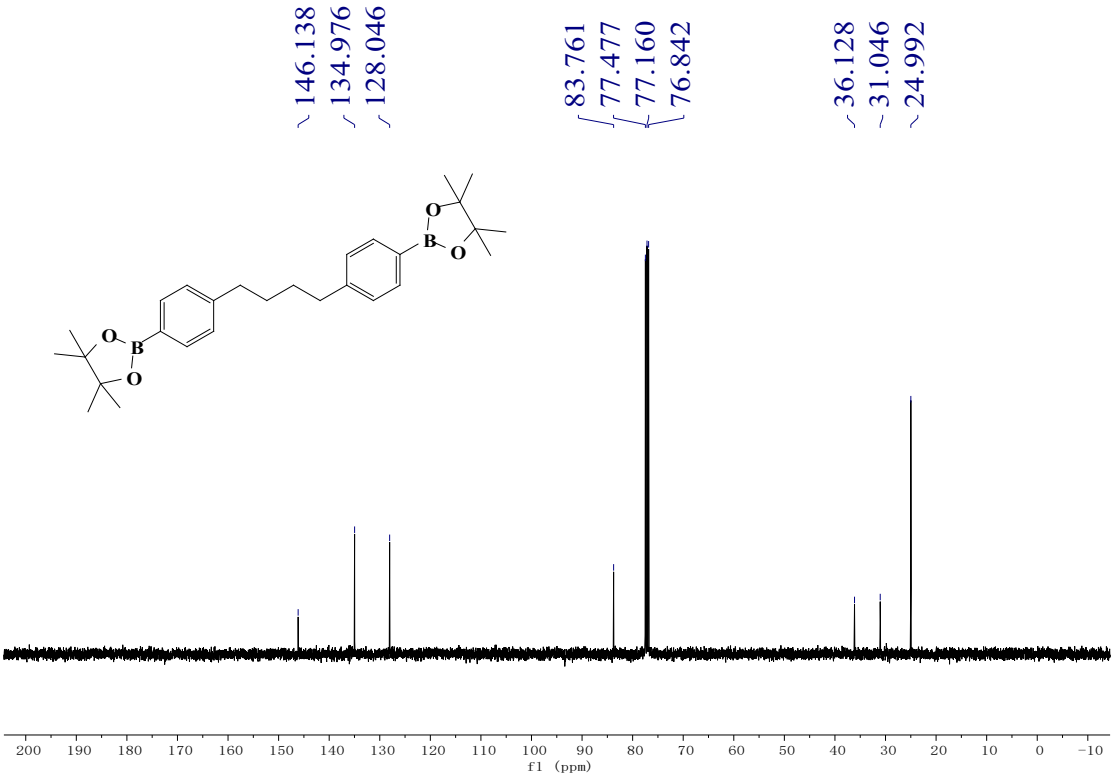
^{13}C NMR spectrum of 2h (100 MHz, $CDCl_3$)



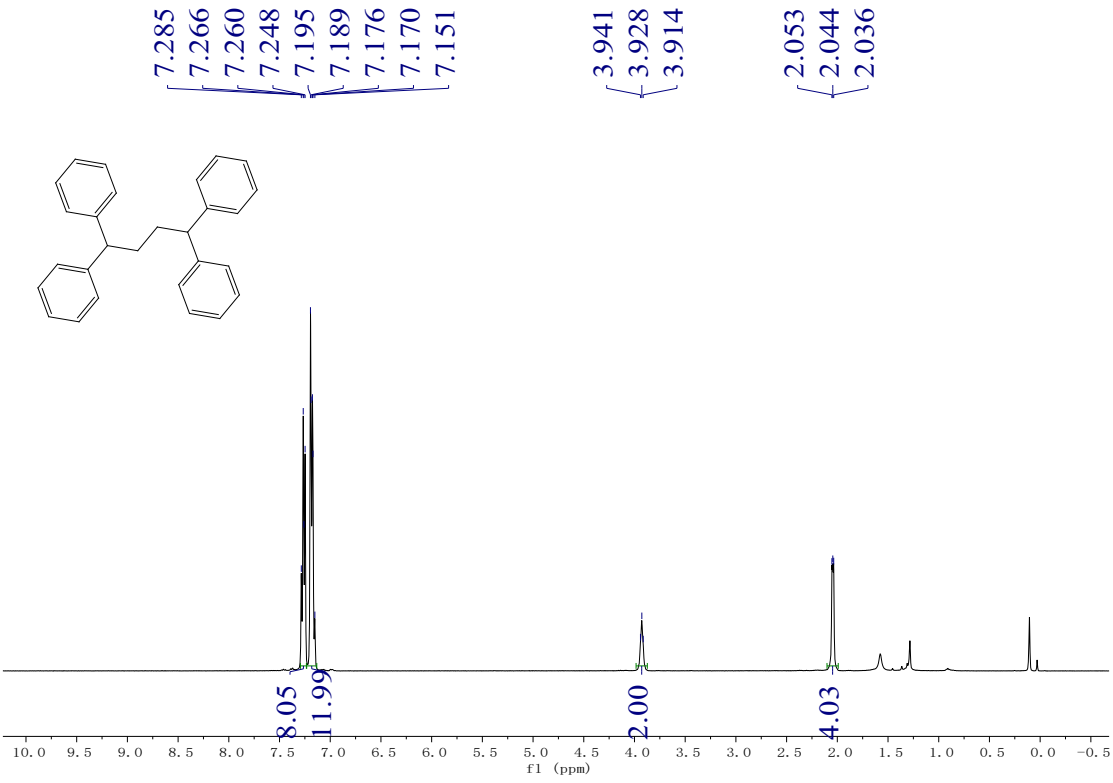
^{1}H NMR spectrum of 2i (400 MHz, $CDCl_3$)



^{13}C NMR spectrum of 2i (100 MHz, $CDCl_3$)



^{1}H NMR spectrum of 2j (400 MHz, $CDCl_3$)



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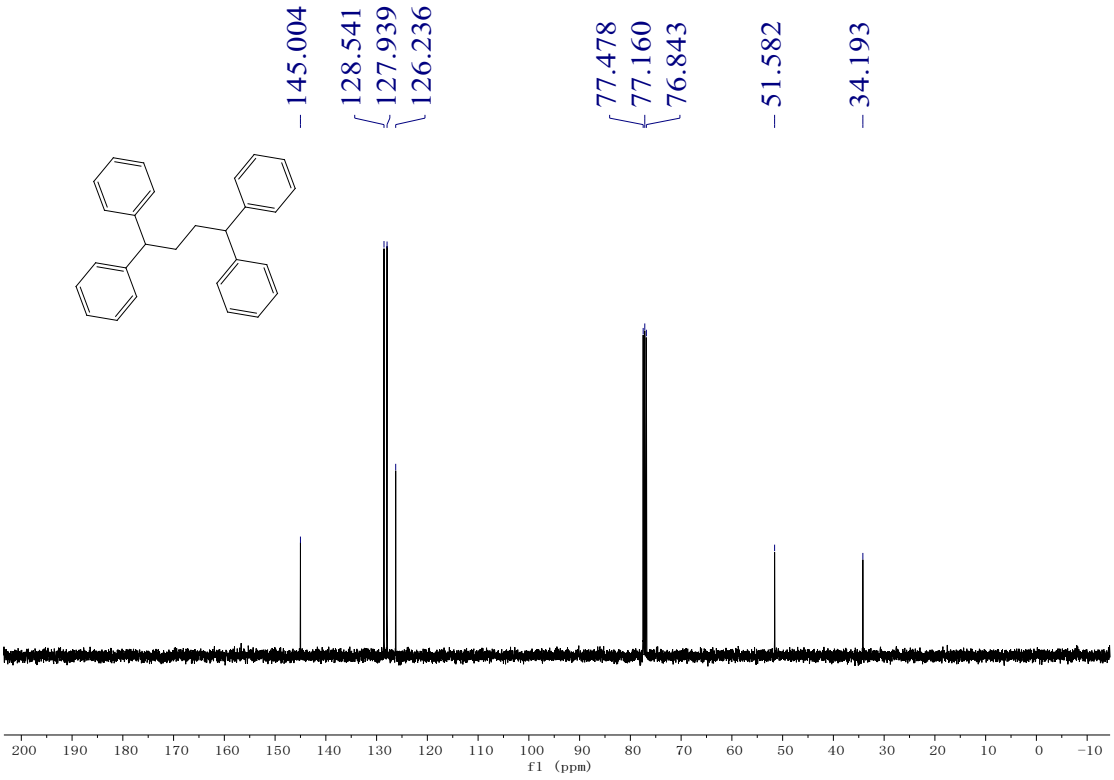
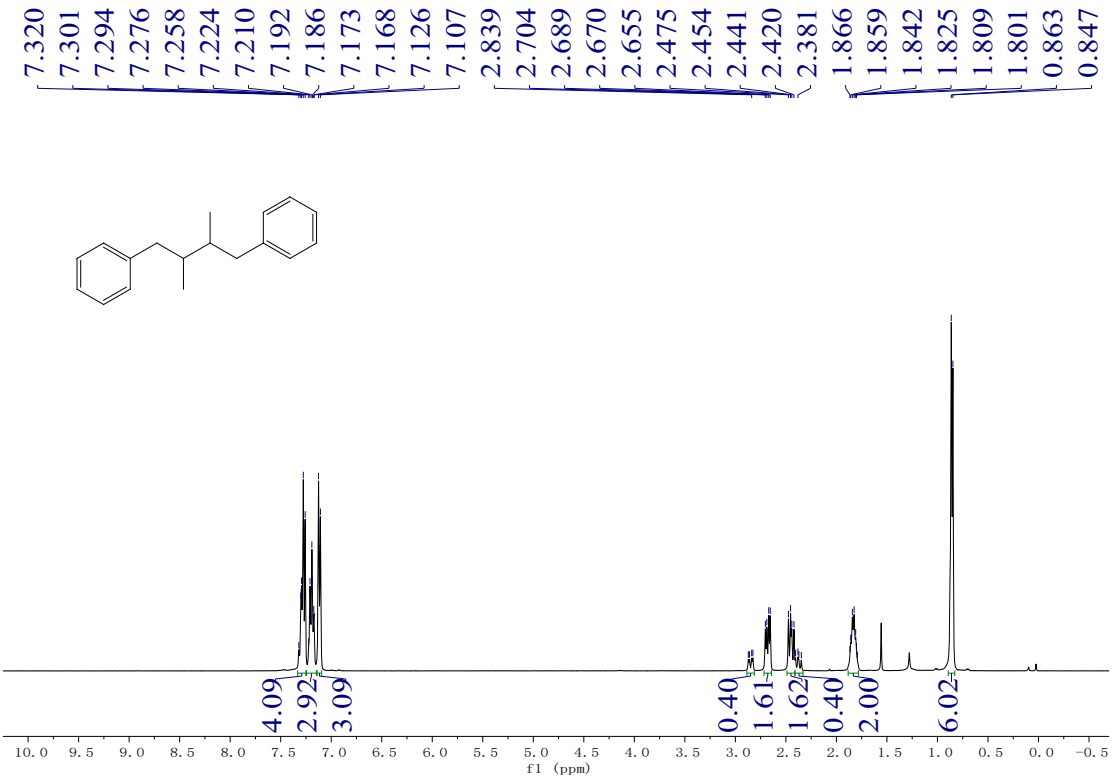
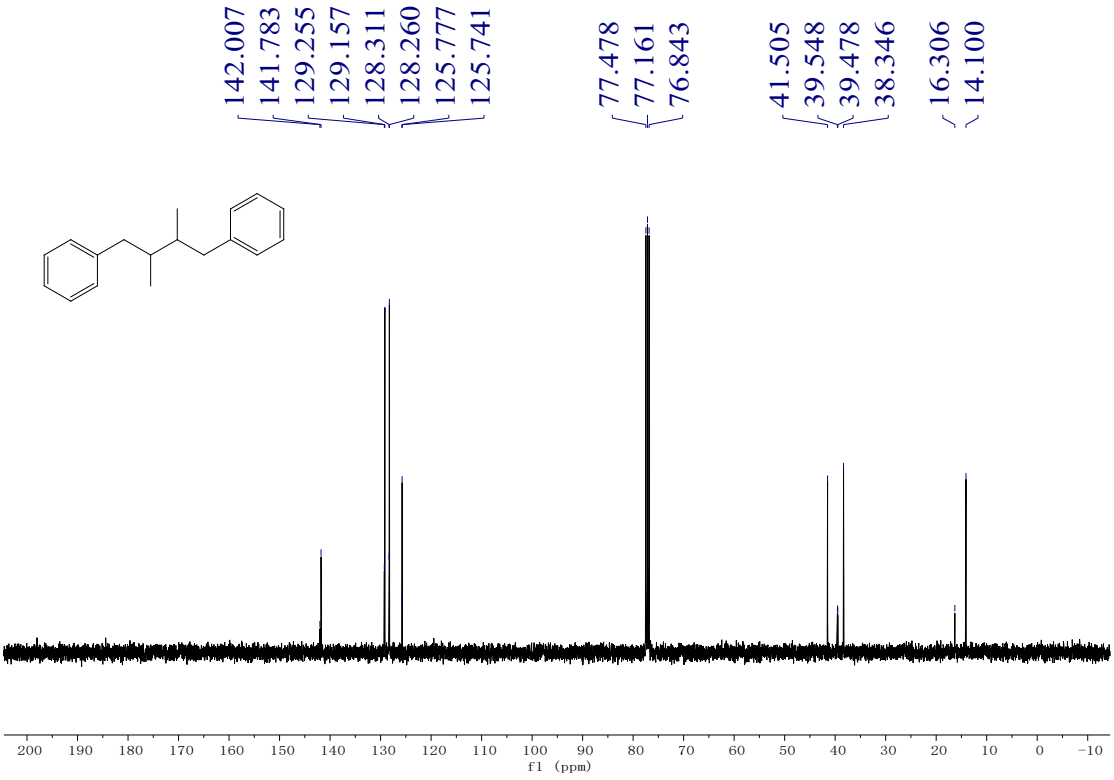


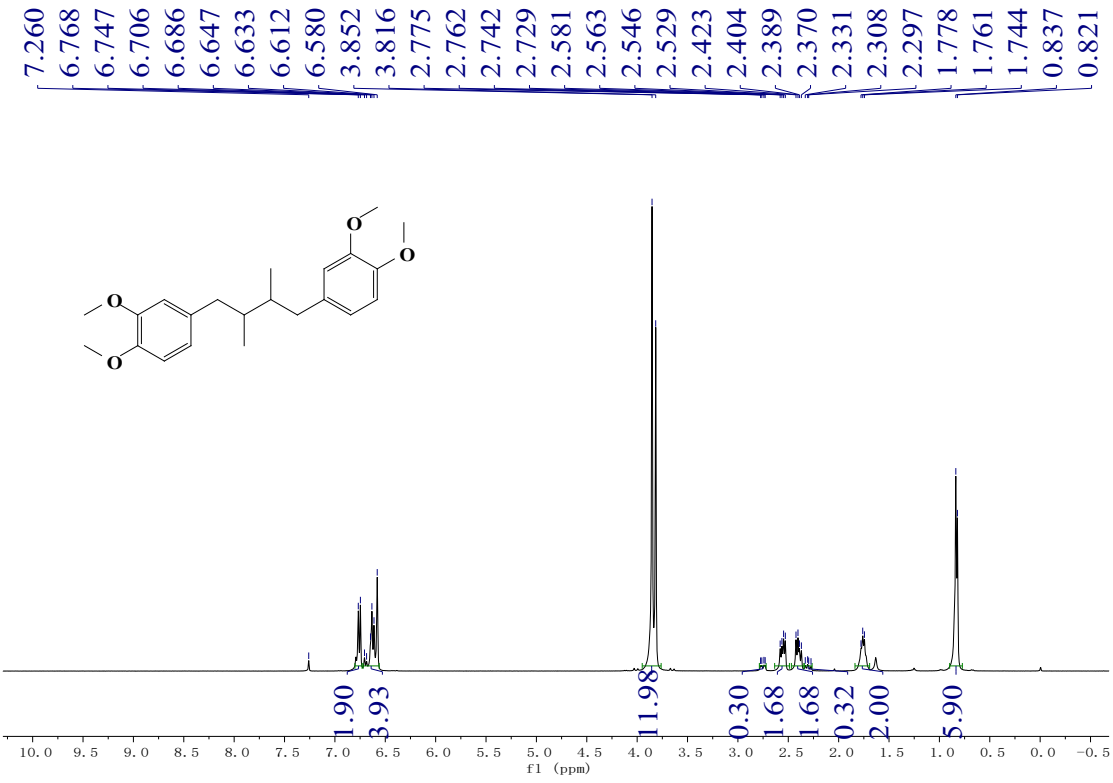
^{1}H NMR spectrum of 2k (400 MHz, $CDCl_3$)



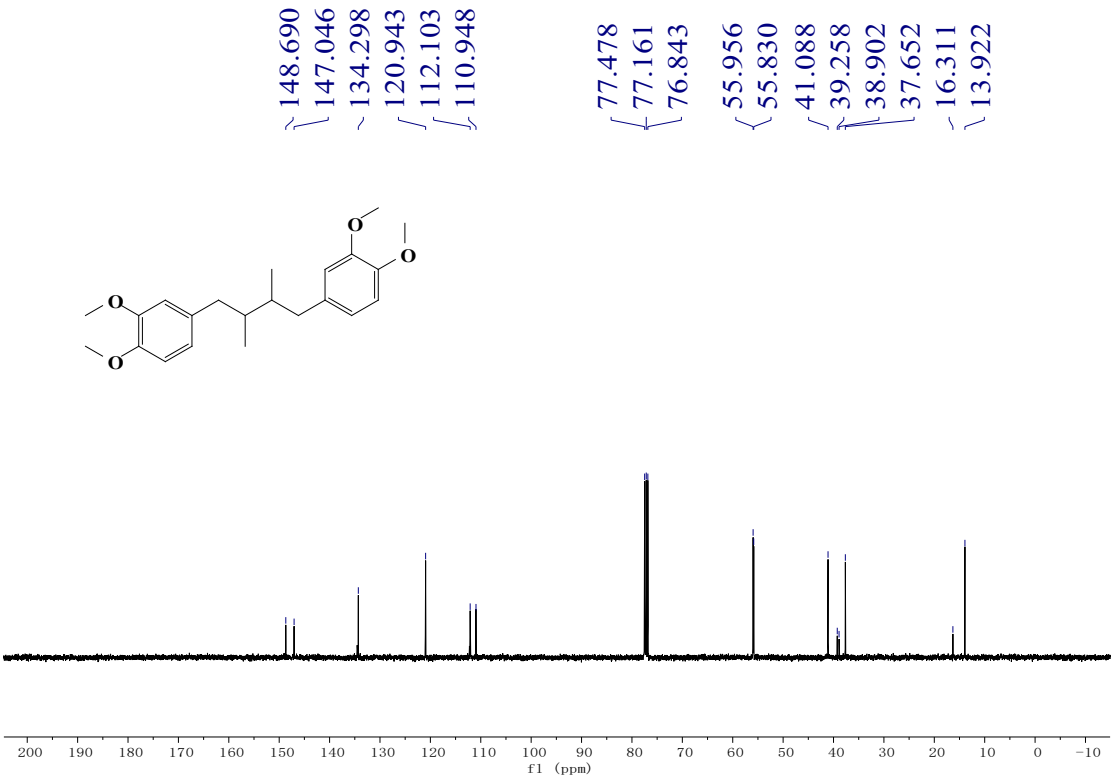
^{13}C NMR spectrum of 2k (100 MHz, $CDCl_3$)



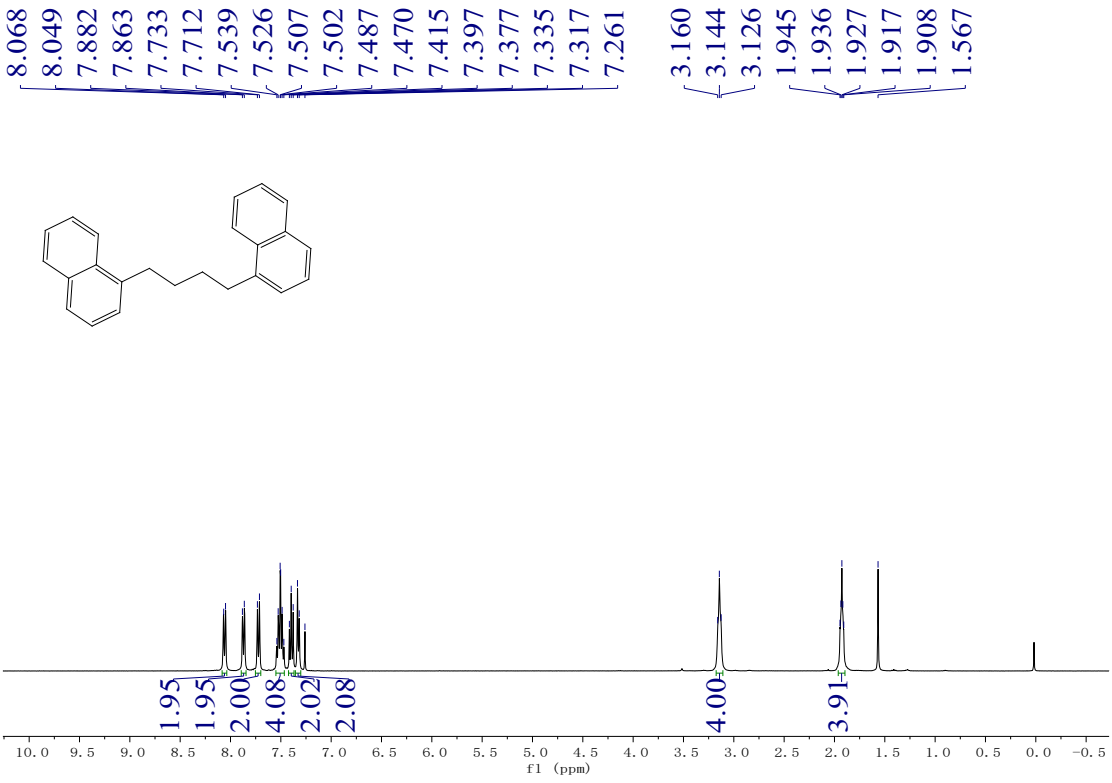
^{1}H NMR spectrum of 2l (400 MHz, $CDCl_3$)



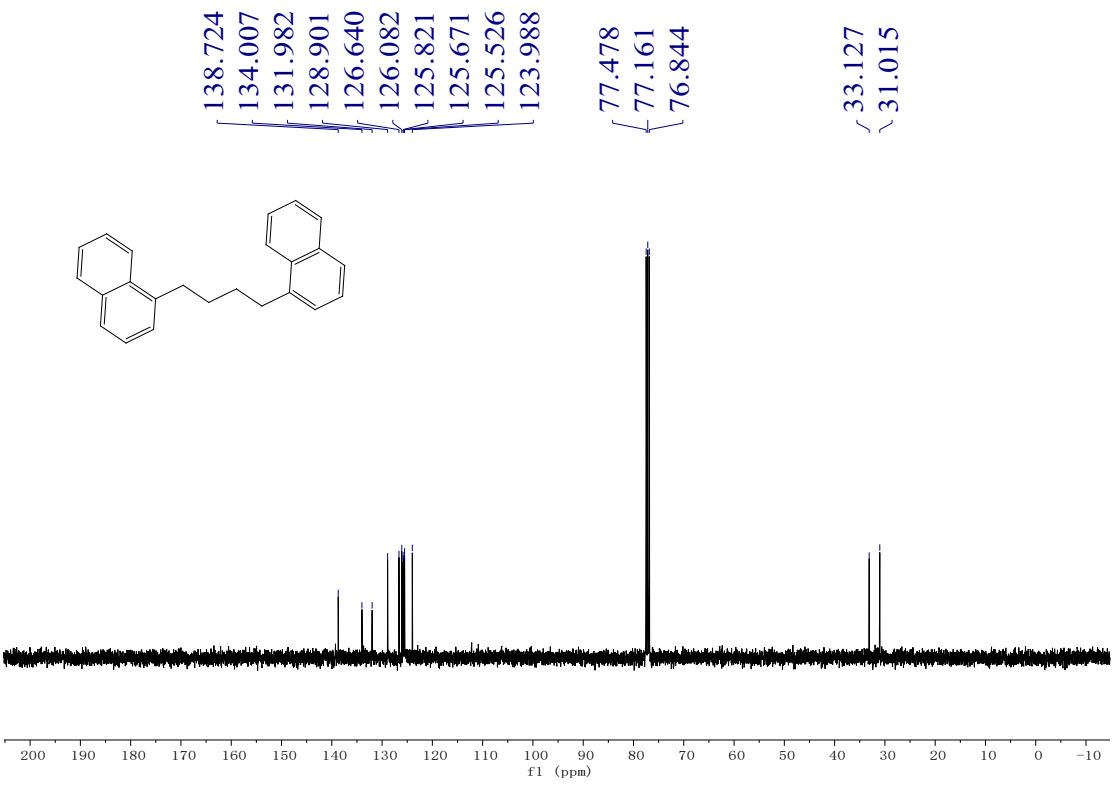
^{13}C NMR spectrum of 2l (100 MHz, $CDCl_3$)



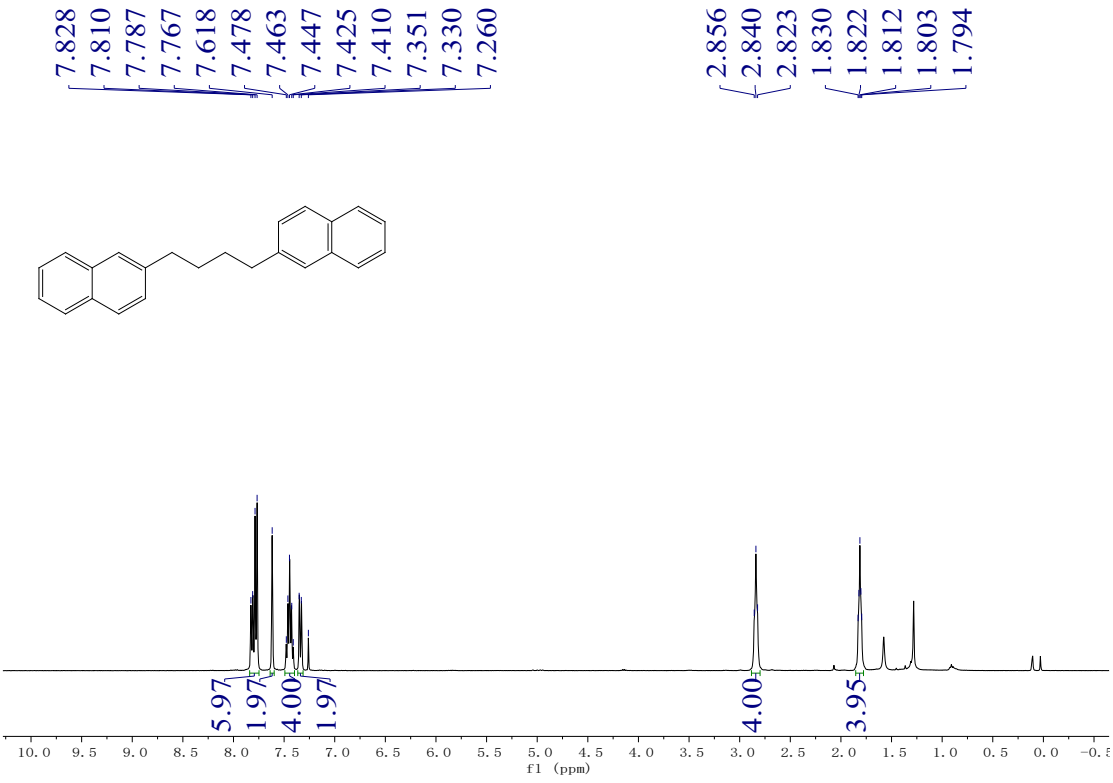
^{1}H NMR spectrum of 2m (400 MHz, $CDCl_3$)



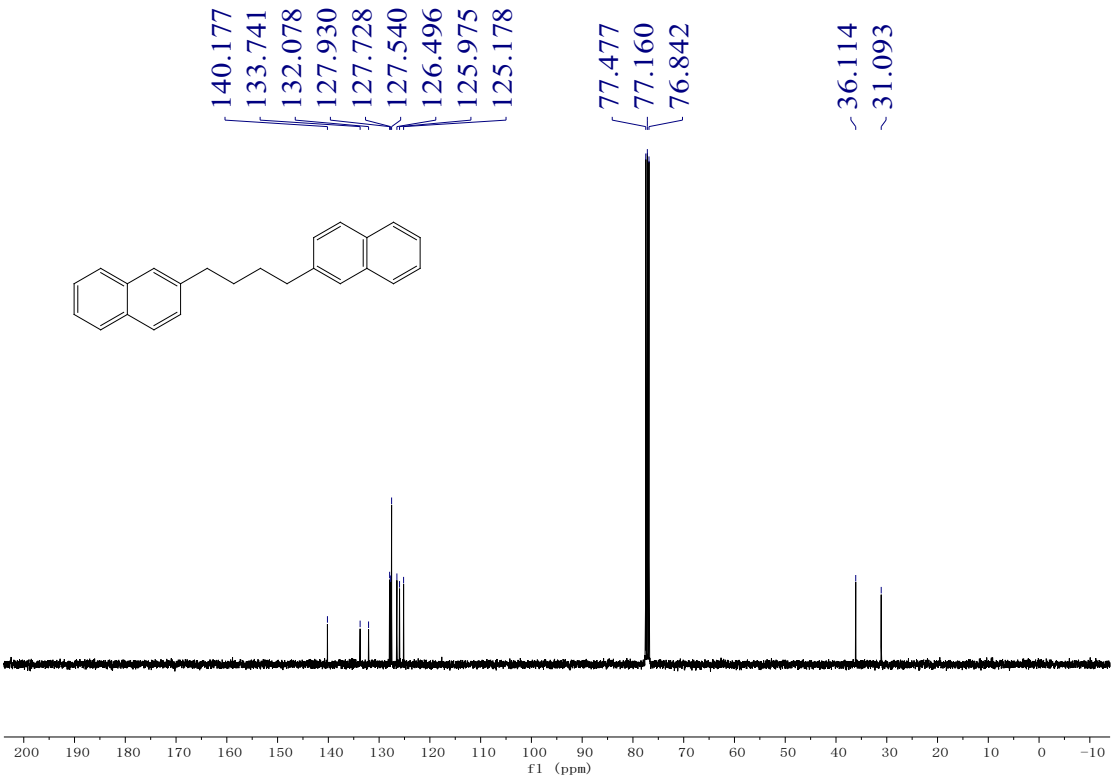
^{13}C NMR spectrum of 2m (100 MHz, $CDCl_3$)



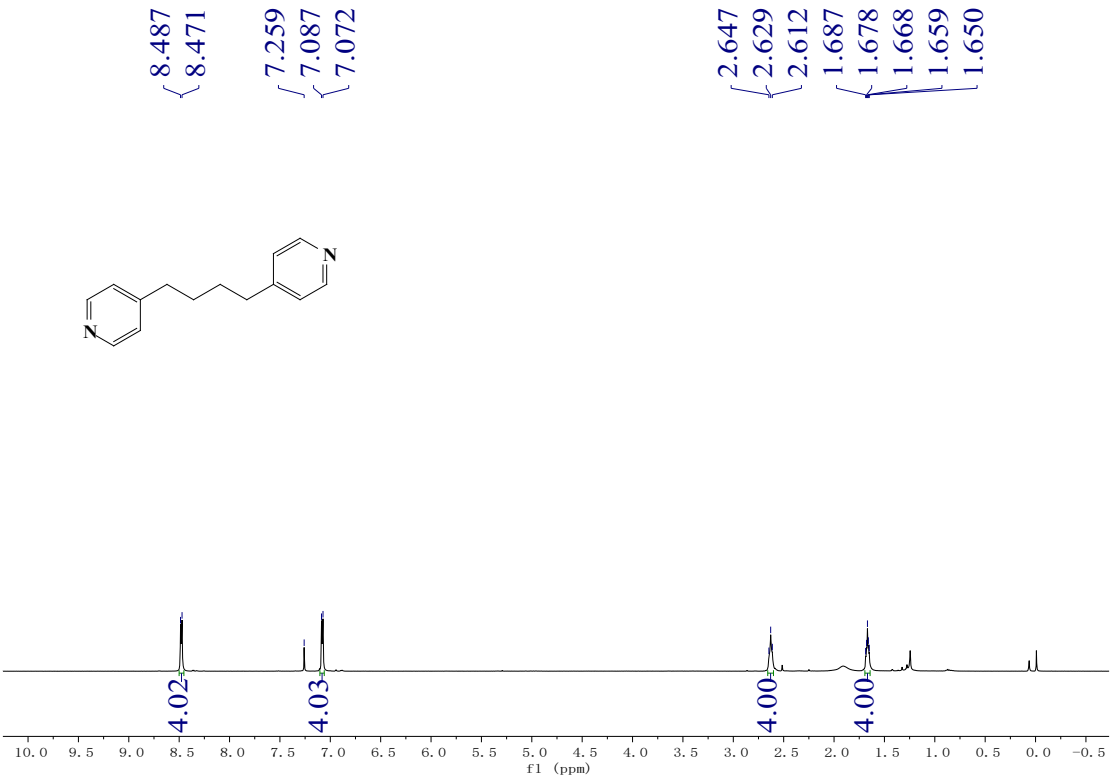
^{1}H NMR spectrum of 2n (400 MHz, $CDCl_3$)



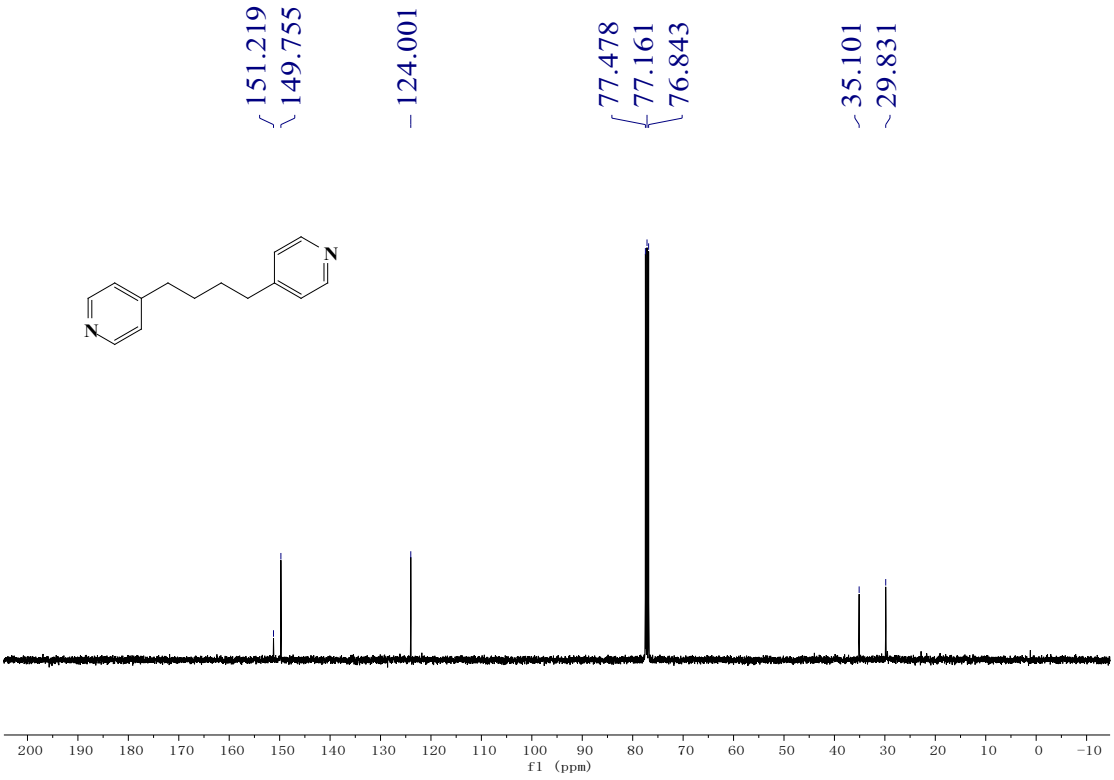
^{13}C NMR spectrum of 2n (100 MHz, $CDCl_3$)



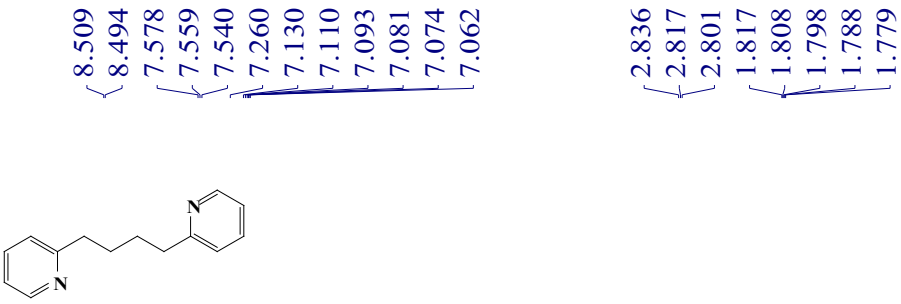
^{1}H NMR spectrum of 2o (400 MHz, $CDCl_3$)

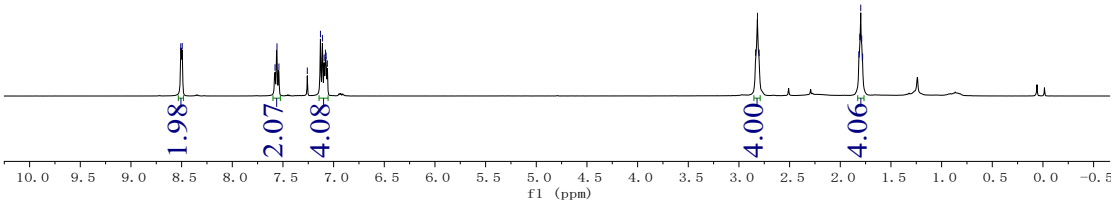


^{13}C NMR spectrum of 2o (100 MHz, $CDCl_3$)

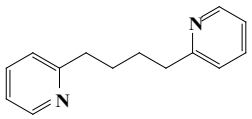


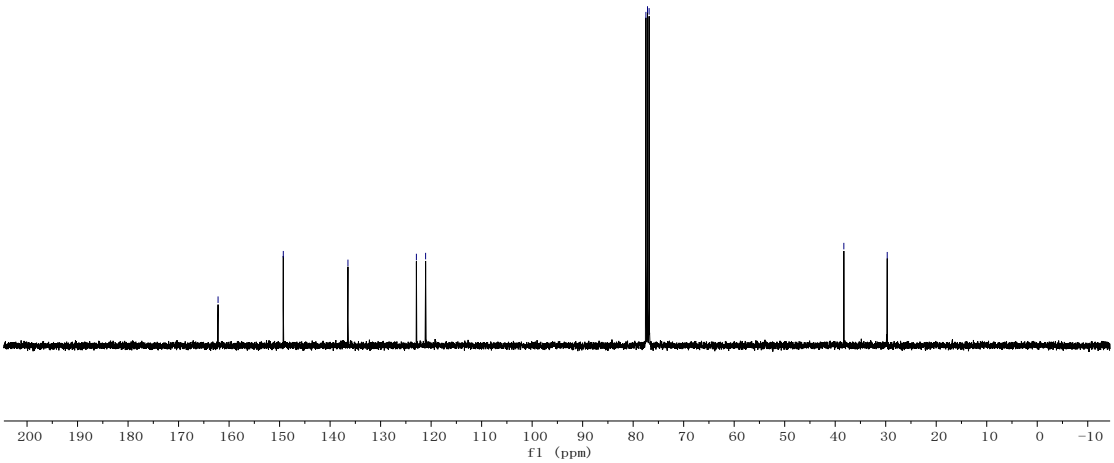
^{1}H NMR spectrum of 2p (400 MHz, $CDCl_3$)



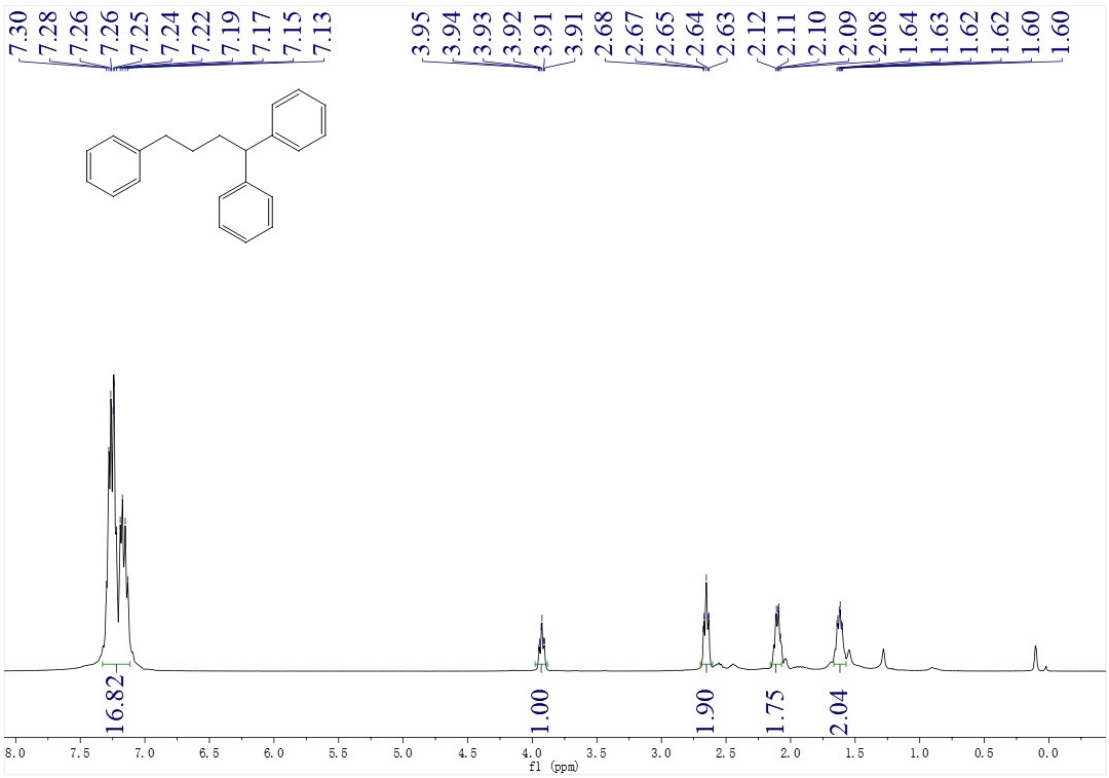


^{13}C NMR spectrum of 2p (100 MHz, $CDCl_3$)

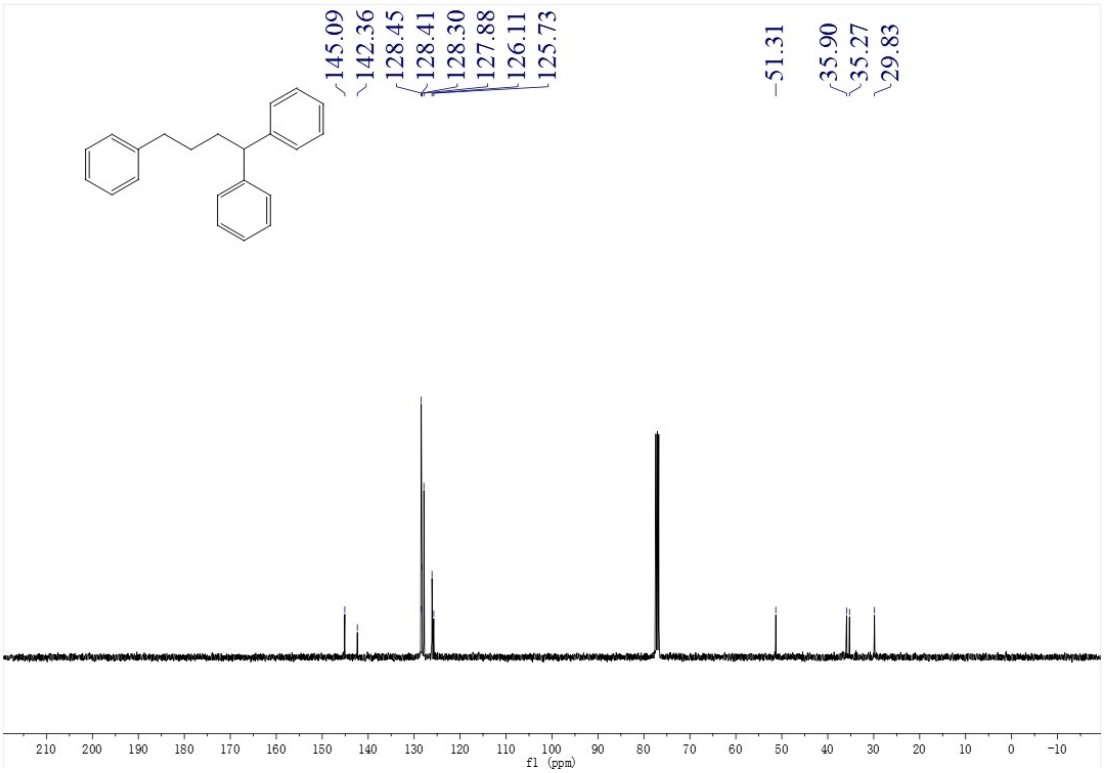




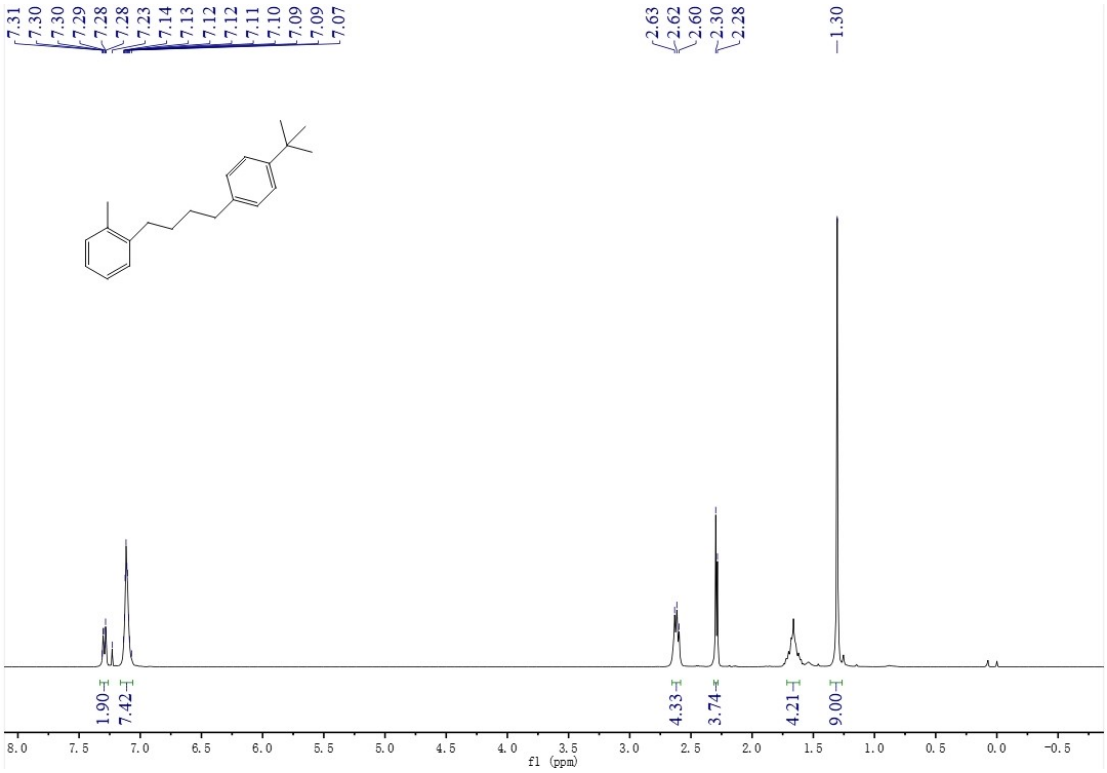
^{1}H NMR spectrum of 2q (400 MHz, $CDCl_3$)



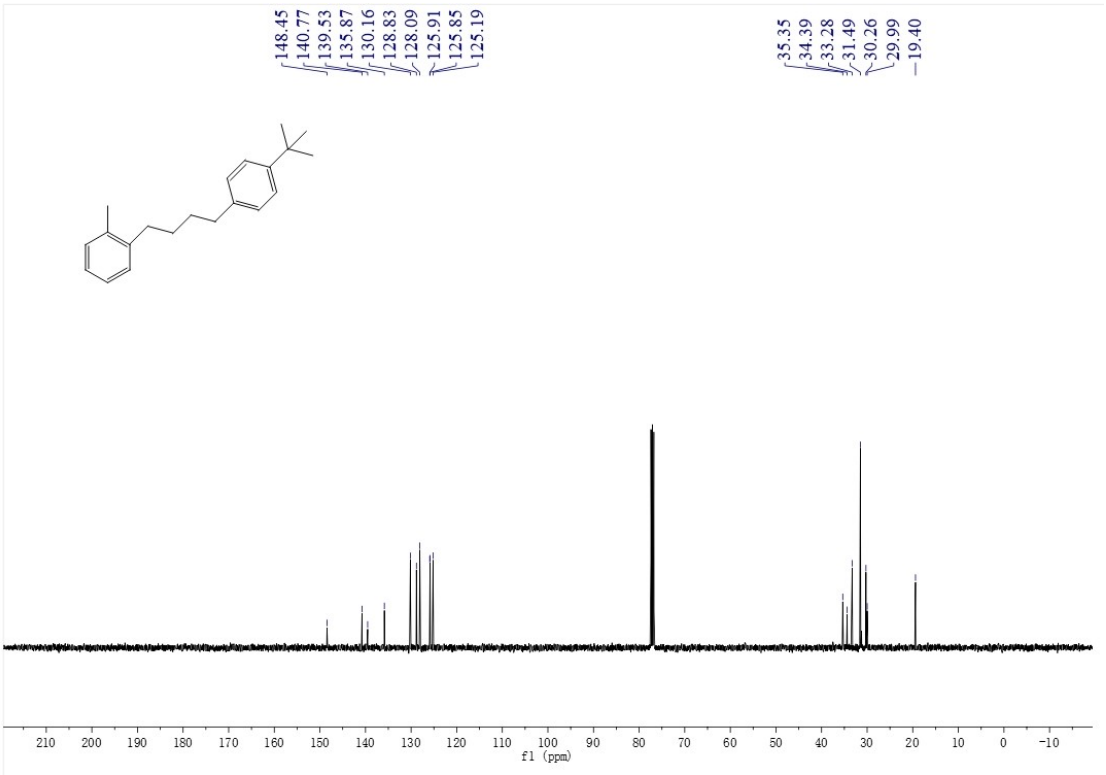
^{13}C NMR spectrum of 2q (100 MHz, $CDCl_3$)



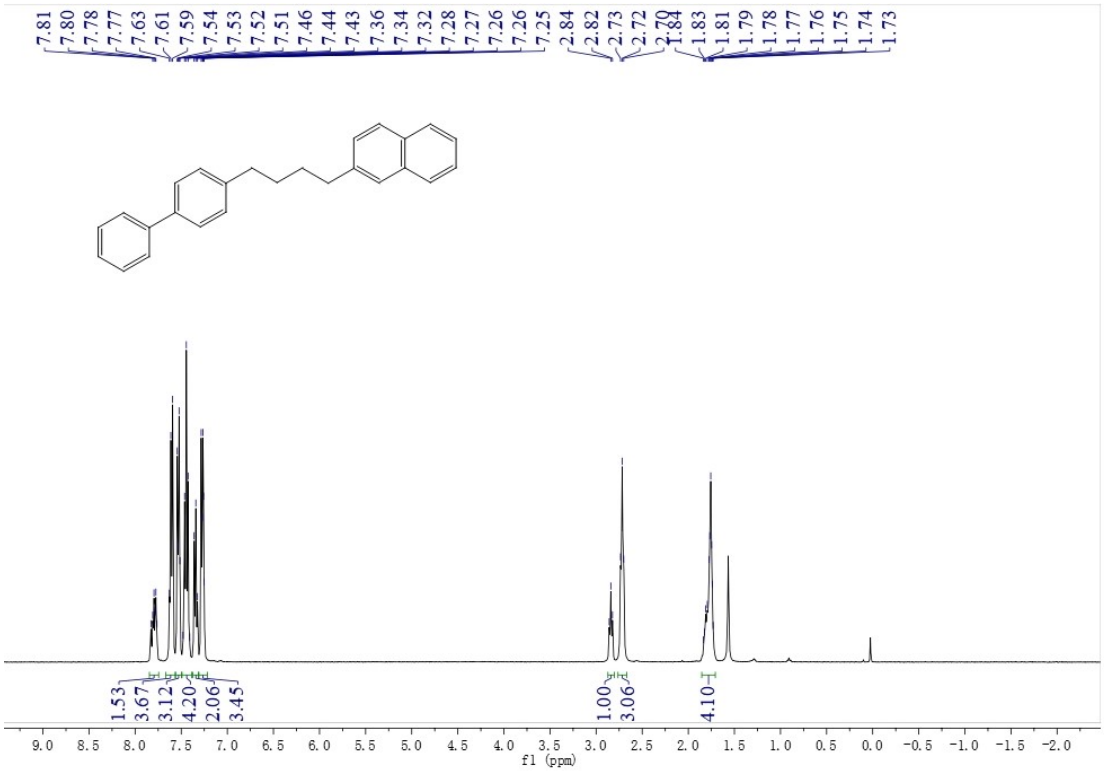
^{1}H NMR spectrum of 2r (400 MHz, $CDCl_3$)



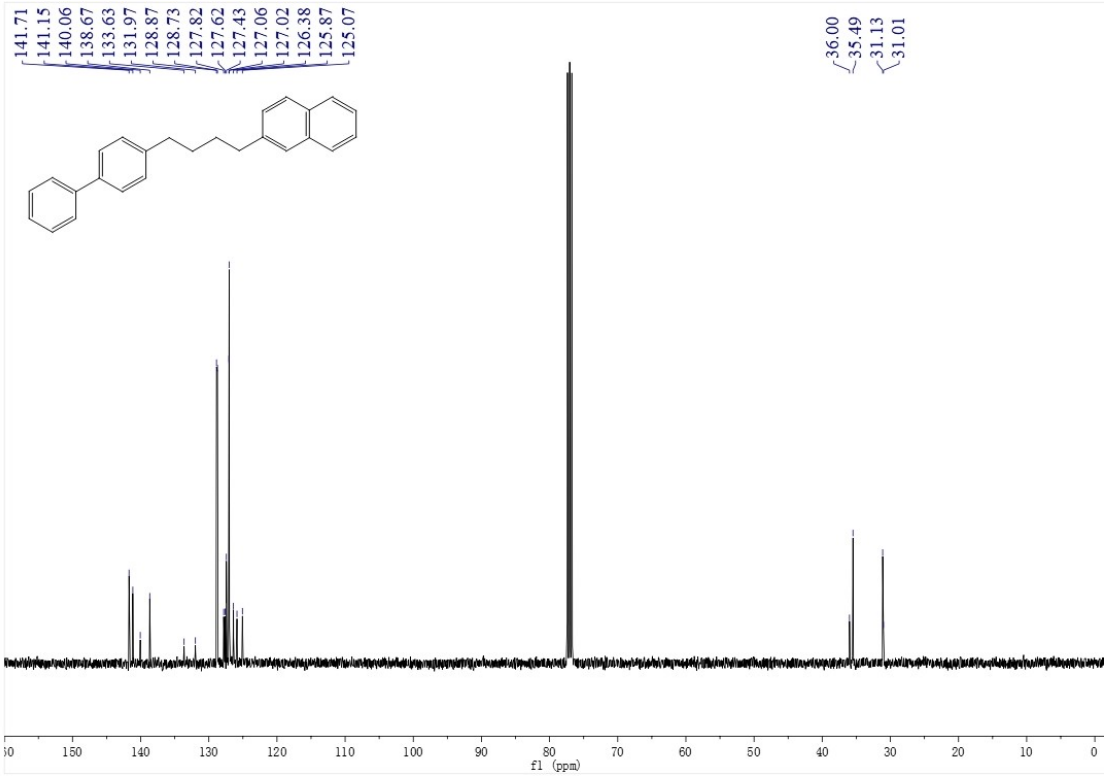
^{13}C NMR spectrum of 2r (100 MHz, $CDCl_3$)



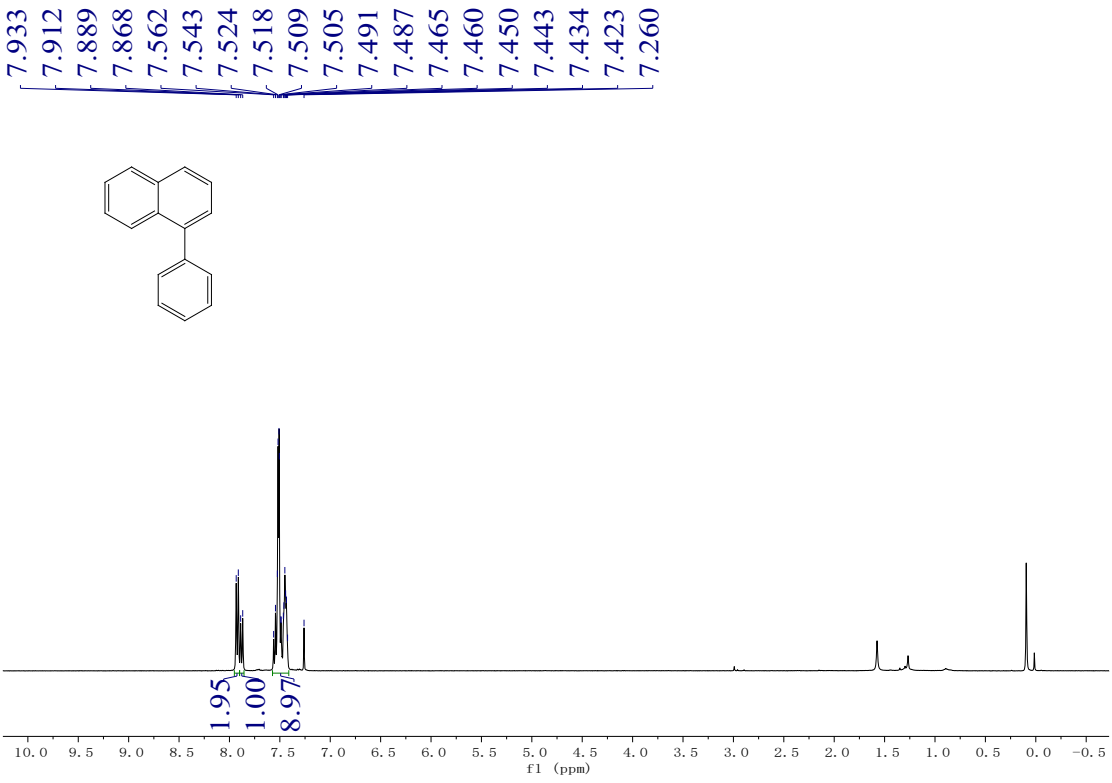
^{1}H NMR spectrum of 2s (400 MHz, $CDCl_3$)



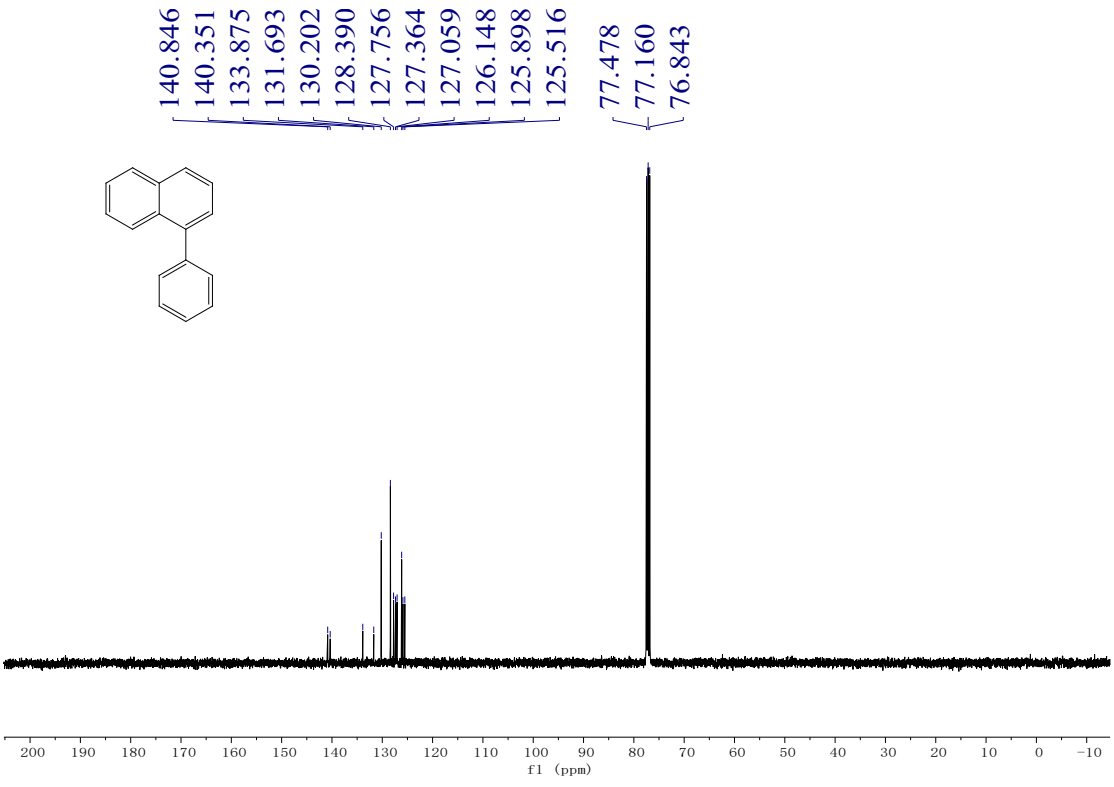
^{13}C NMR spectrum of 2s (100 MHz, $CDCl_3$)



^{1}H NMR spectrum of 3 (400 MHz, $CDCl_3$)



^{13}C NMR spectrum of 3s (100 MHz, $CDCl_3$)



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