

Gold-catalyzed efficient tandem assembly of terminal alkynes and arynes

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Supporting Information

Content

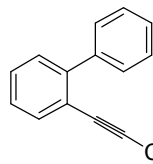
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General All reactions were carried out under the nitrogen atmosphere in oven-dried flask. CH₃CN and toluene were distilled from Na using benzophenone as the indicator. Benzyne precursors were prepared according to the method of reference 1. Other materials were purchased from common commercial sources and used without additional purification. ¹H NMR spectra were recorded at 400 MHz or 500 MHz using TMS as internal standard. ¹³C NMR spectra were recorded at 100 MHz using TMS as internal standard. Mass spectroscopy data of the reaction product were collected on an HRMS-EI instrument.

Representative procedure of the gold-catalyzed coupling reaction of terminal alkynes and arynes : 1-ethynyl-benzene (31 mg, 0.3 mmol), CuI (6 mg, 0.03 mmol), AuClPPh₃ (15 mg, 0.03 mmol) and CsF (276 mg, 1.8 mmol) were charged into an oven-dried flask, followed by the addition of MeCN (2 mL) under the protection of nitrogen atmosphere to form a suspension. The solution of 2-(trimethylsilyl)phenyl triflate (180 mg, 0.6 mmol) in MeCN (3 mL) was added into the above suspension by syringe under N₂, and the reaction mixture was then put into a 40 °C oil bath to reaction for an hour. After the completion of reaction, the reaction mixture was filtered through a pad of cellite, and the solvent was then removed under reduced pressure. The residue was then separated on a silica gel column by using petroleum ether as eluent and the final product was obtained as colorless oil.

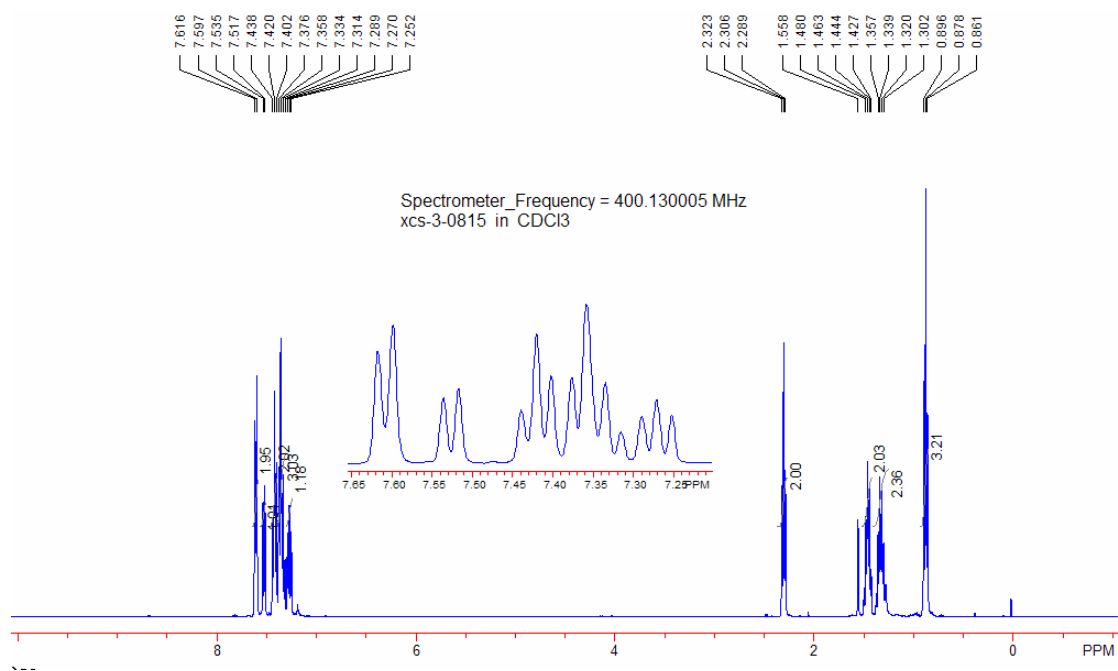
Representative procedure of the intramolecular hydroarylation reaction: Alkynylated biphenyl (127 mg, 0.5 mmol), AuClPPh₃ (25 mg, 0.05 mmol) and AgSbF₆ (15 mg, 0.05 mmol) were placed into an oven-dried flask, and then distilled toluene (5 mL) was charged into the flask under N₂. The mixture was allowed to react at 110 °C for 12 h to finish the reaction. After the completion of the reaction, the mixture was filtered to remove the insoluble powder, and the resulting filtrate was condensed under vacuum. The final product was obtained by flash chromatograph on a silica gel column as a white powder.

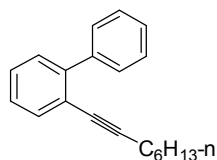
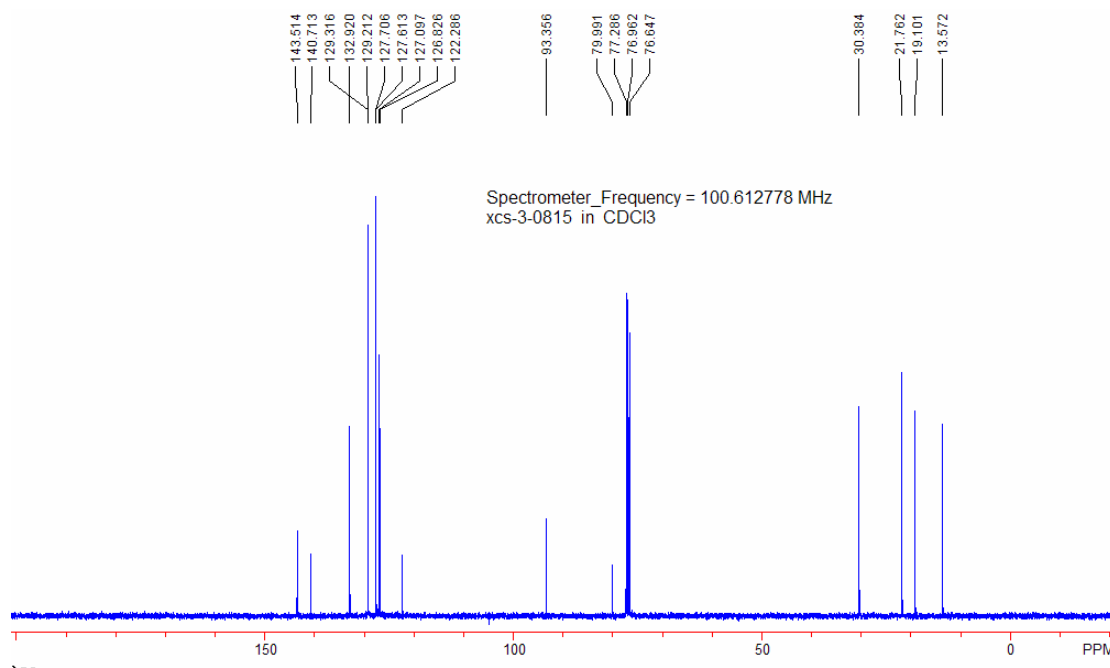
Characterization data of the product



$\text{C}_4\text{H}_9\text{-n}$ T2-1, 851901-90-1, Ref. 2

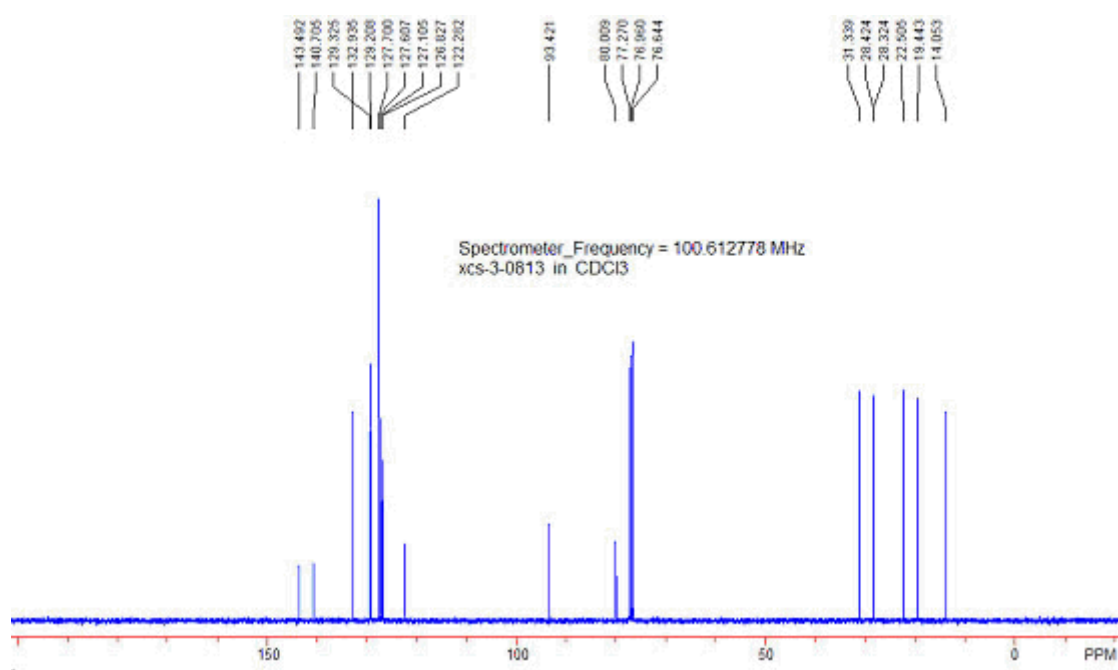
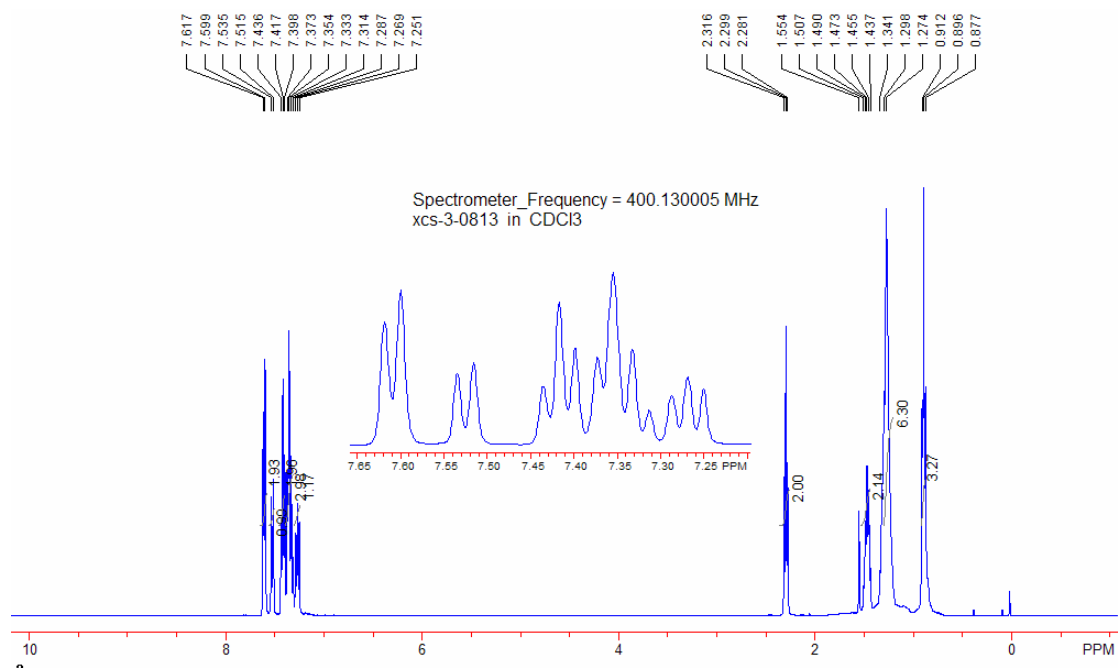
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.61 (d, $J = 7.6$ Hz, 2 H), 7.53 (d, $J = 7.2$ Hz, 1 H), 7.42 (t, $J = 7.6$ Hz, 2 H), 7.31-7.38 (m, 3 H), 7.27 (t, $J = 7.4$ Hz, 1 H), 2.31 (t, $J = 6.8$ Hz, 2 H), 1.43-1.50 (m, 2 H), 1.28-1.38 (m, 2 H), 0.88 (t, $J = 7.0$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.5, 140.7, 132.9, 129.3, 129.2, 127.7, 127.6, 127.1, 126.8, 122.3, 93.4, 80.0, 30.4, 21.8, 19.1, 12.6. HRMS (EI) Calcd for $\text{C}_{18}\text{H}_{18}$: $[\text{M}]^+$ 234.1409; Found, 234.1413.

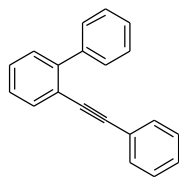




T2-2, 263017-26-1, Ref. 3

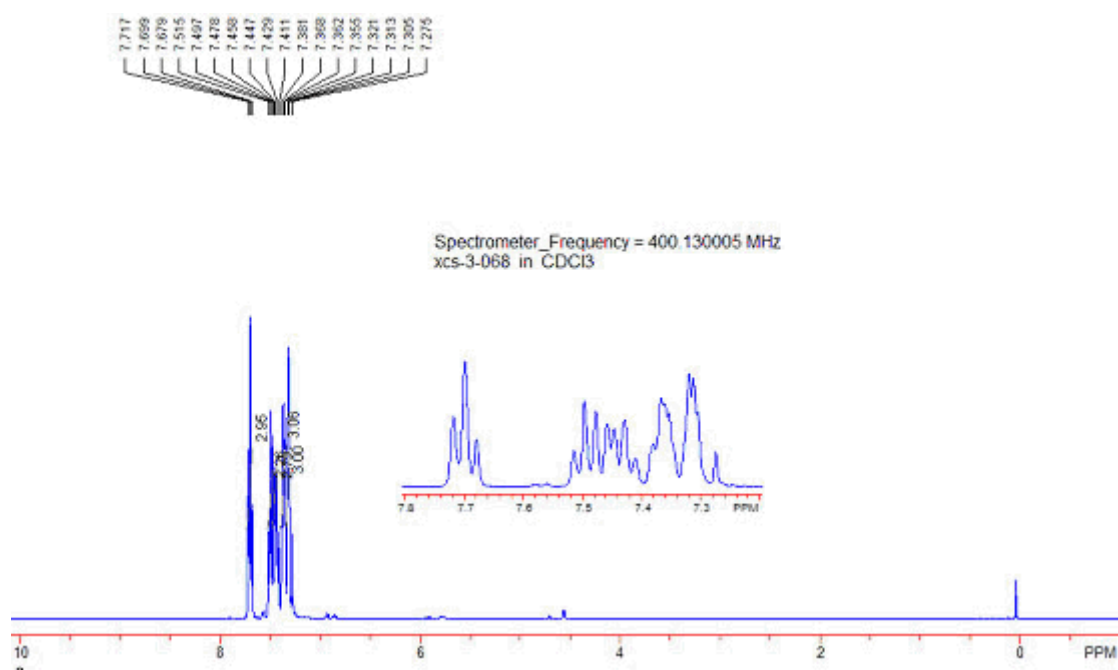
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.61 (d, *J* = 7.2 Hz, 2 H), 7.53 (d, *J* = 8.0 Hz, 1 H), 7.42 (t, *J* = 7.6 Hz, 2 H), 7.31-7.37 (m, 3 H), 7.27 (t, *J* = 7.2 Hz, 1 H), 2.30 (t, *J* = 7.0 Hz, 2 H), 1.44-1.51 (m, 2 H), 1.27-1.34 (m, 6 H), 0.90 (t, *J* = 7.0 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 143.5, 140.7, 132.9, 129.3, 129.2, 127.7, 127.6, 127.1, 126.8, 122.3, 93.4, 80.0, 31.3, 28.4, 28.3, 22.5, 19.4, 14.0. HRMS (EI) Calcd for C₂₀H₂₂: [M]⁺ 262.1722; Found, 262.1723.

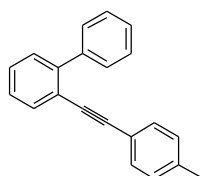
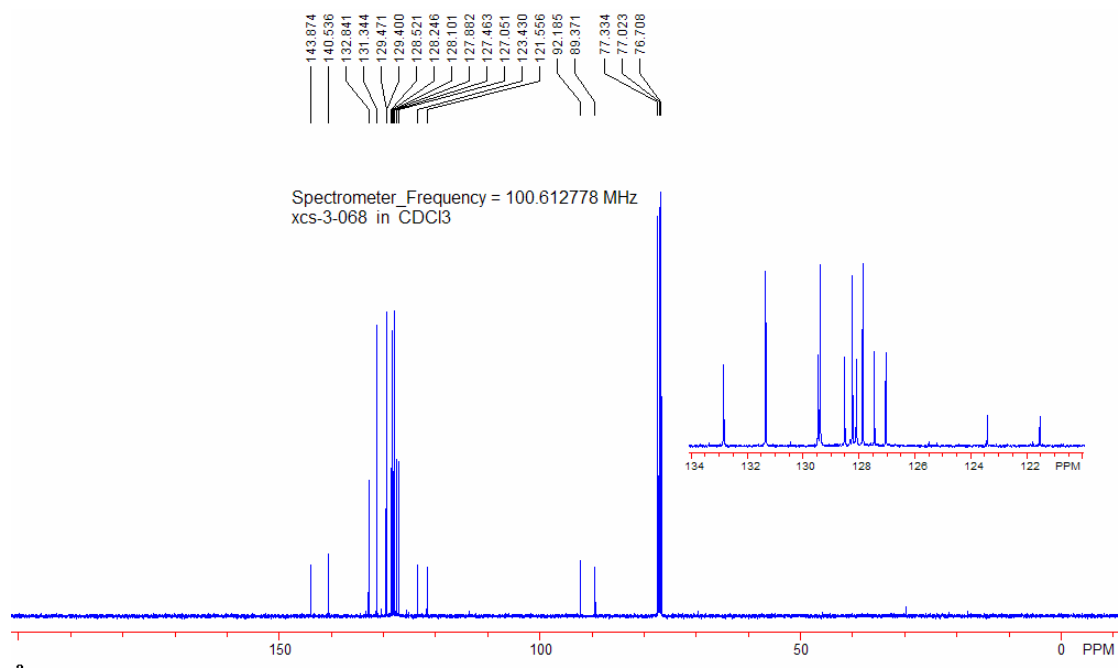




T2-3, 10271-65-5, Ref. 4

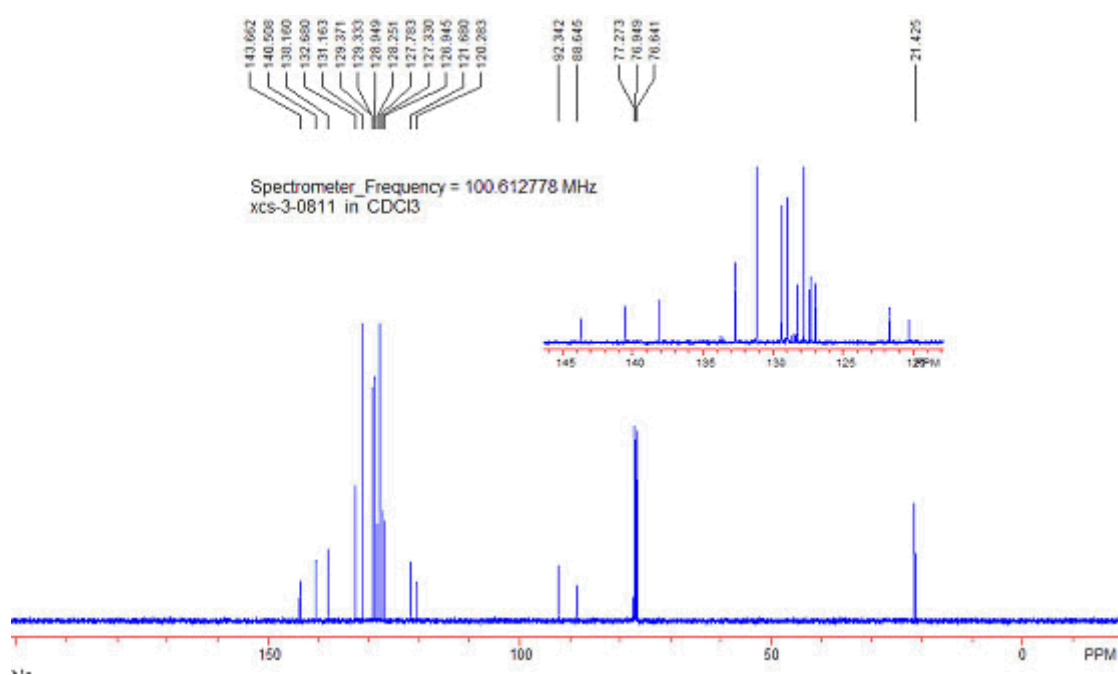
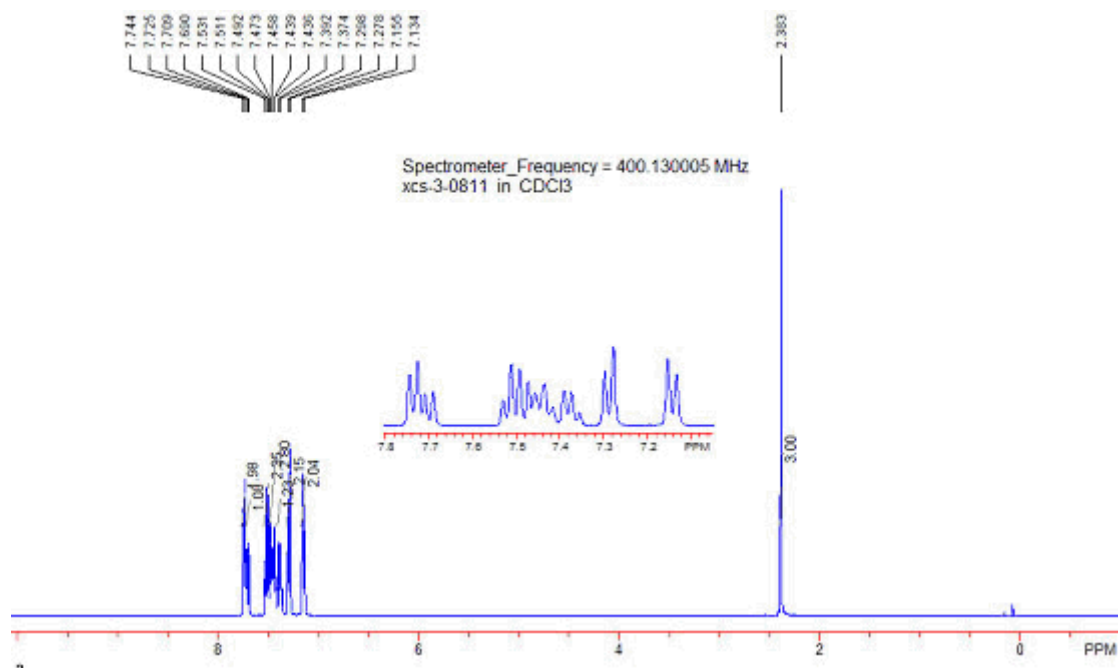
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.68-7.72 (m, 3 H), 7.50 (t, $J = 7.4$ Hz, 2 H), 7.41-7.46 (m, 3 H), 7.26-7.38 (m, 3 H), 7.30-7.32 (m, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.9, 140.5, 132.8, 131.3, 129.5, 129.4, 128.5, 128.2, 128.1, 127.9, 127.5, 127.0, 123.4, 121.6, 92.2, 89.4. HRMS (EI) Calcd for $\text{C}_{20}\text{H}_{14}$: $[\text{M}]^+$ 254.1096; Found, 254.1090.

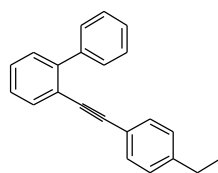




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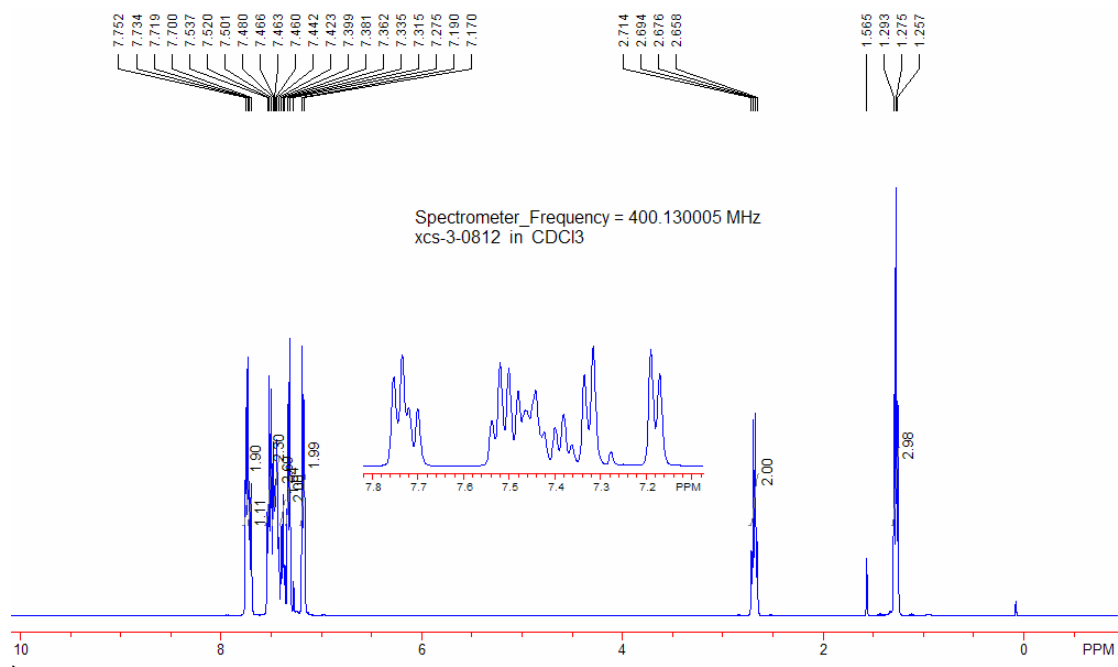
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.73 (d, J = 7.6 Hz, 2 H), 7.70 (d, J = 7.6 Hz, 1 H), 7.51 (t, J = 7.8 Hz, 2 H), 7.42-7.47 (m, 3 H), 7.37 (t, J = 7.2 Hz, 1 H), 7.29 (d, J = 8.0 Hz, 2 H), 7.14 (d, J = 8.4 Hz, 2 H), 2.38 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.7, 140.5, 138.2, 132.7, 131.2, 129.4, 129.3, 128.9, 128.3, 127.8, 127.3, 126.9, 121.7, 120.3, 92.3, 88.6, 21.4. HRMS (EI) Calcd for $\text{C}_{21}\text{H}_{16}$: $[\text{M}]^+$ 268.1252; Found, 268.1251.

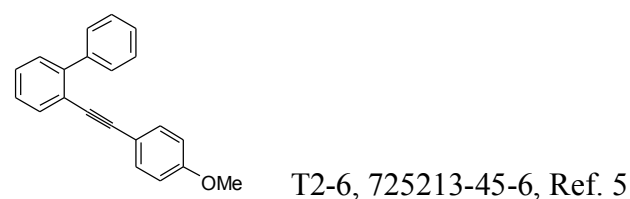
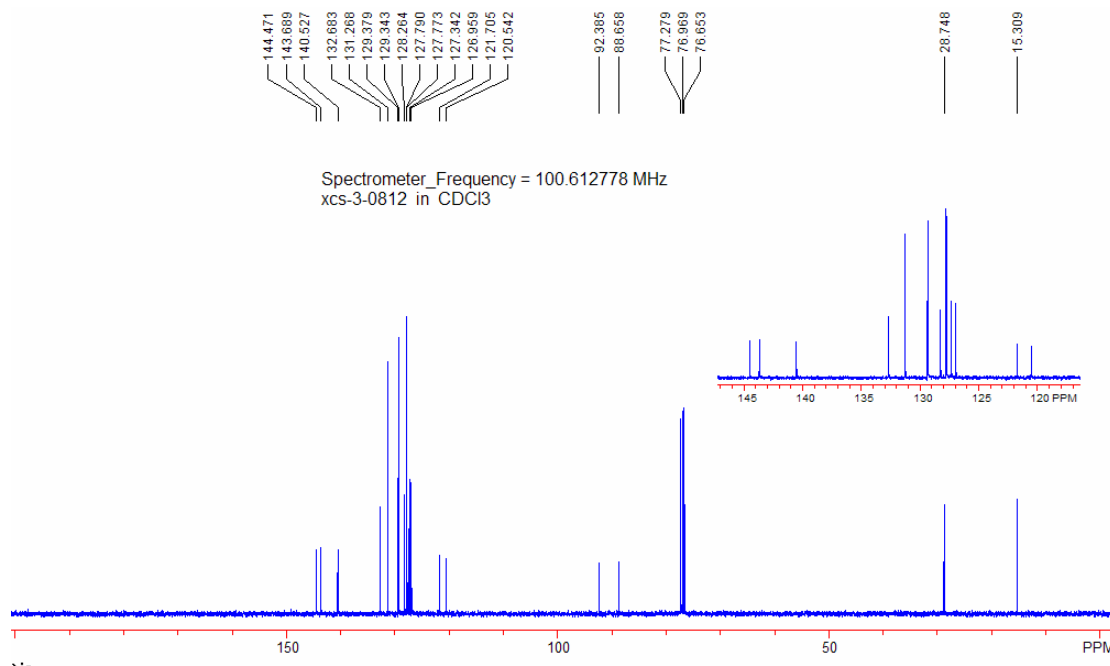




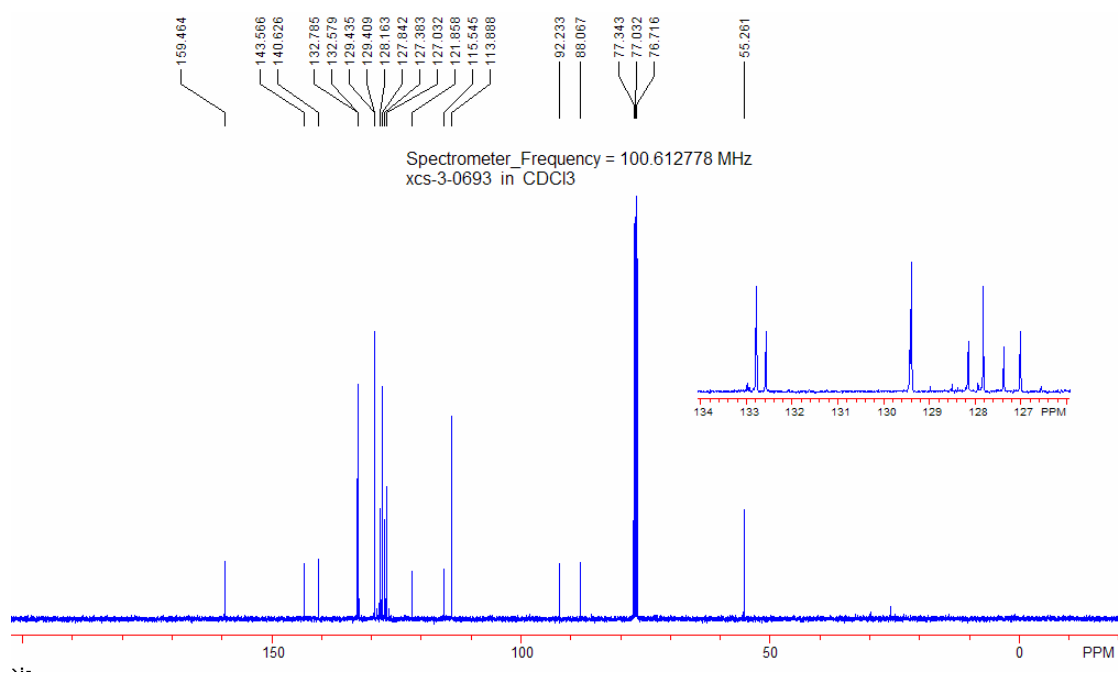
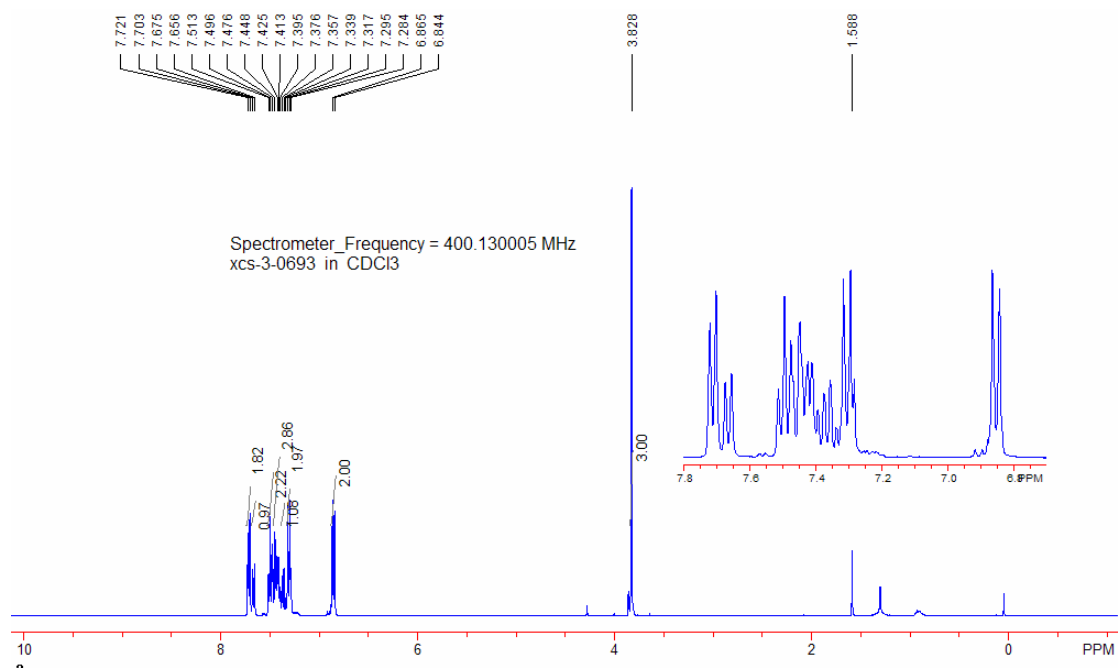
T2-5, new compound

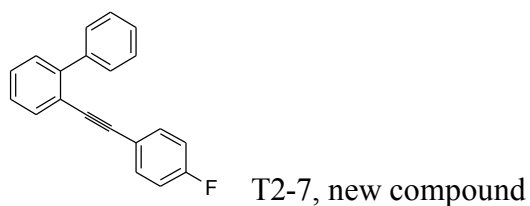
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.74 (d, $J = 7.6$ Hz, 2 H), 7.71 (d, $J = 7.6$ Hz, 1 H), 7.52 (t, $J = 7.2$ Hz, 2 H), 7.44-7.48 (m, 3 H), 7.38 (t, $J = 7.4$ Hz, 1 H), 7.33 (d, $J = 8.0$ Hz, 2 H), 7.18 (d, $J = 8.0$ Hz, 2 H), 2.68 (q, $J = 7.4$ Hz, 2 H), 1.28 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.5, 143.7, 140.5, 132.7, 131.3, 129.4, 129.3, 128.3, 127.8, 127.8, 127.3, 126.9, 121.7, 120.5, 92.4, 88.6, 28.7, 15.3. HRMS (EI) Calcd for $\text{C}_{22}\text{H}_{18}$: $[\text{M}]^+$ 282.1409; Found, 282.1409.



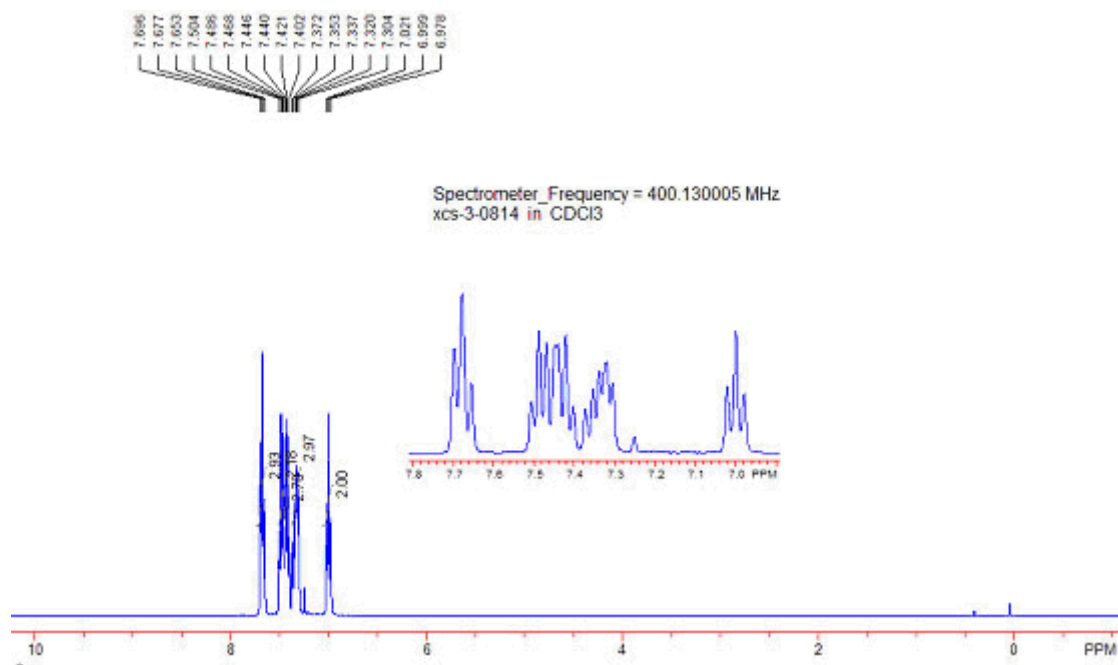


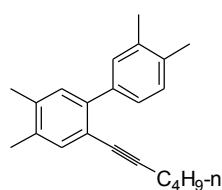
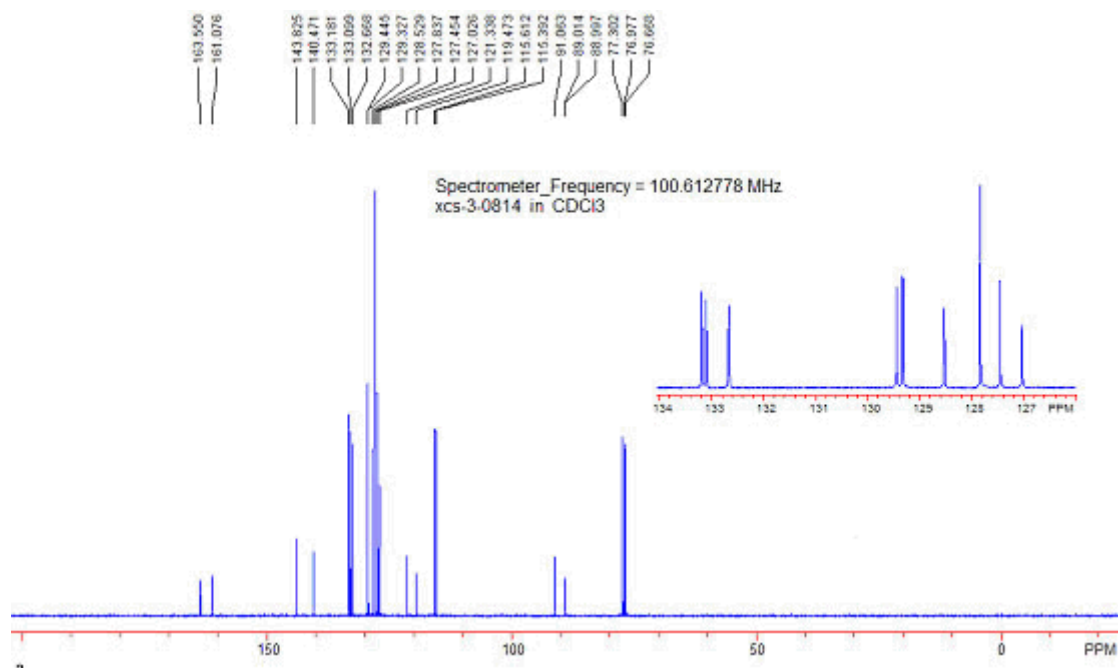
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.71 (d, *J* = 7.2 Hz, 2 H), 7.67 (d, *J* = 7.6 Hz, 1 H), 7.50 (t, *J* = 7.4 Hz, 2 H), 7.40-7.45 (m, 3 H), 7.34 (t, *J* = 8.0 Hz, 1 H), 7.29 (d, *J* = 8.4 Hz, 2 H), 6.85 (d, *J* = 8.4 Hz, 2 H), 3.83 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 159.5, 143.6, 140.6, 132.8, 132.6, 129.44, 129.41, 128.2, 127.8, 127.4, 127.0, 121.8, 115.5, 113.9, 92.2, 88.1, 55.3. HRMS (EI) Calcd for C₂₁H₁₆O: [M]⁺ 284.1201; Found, 284.1203.





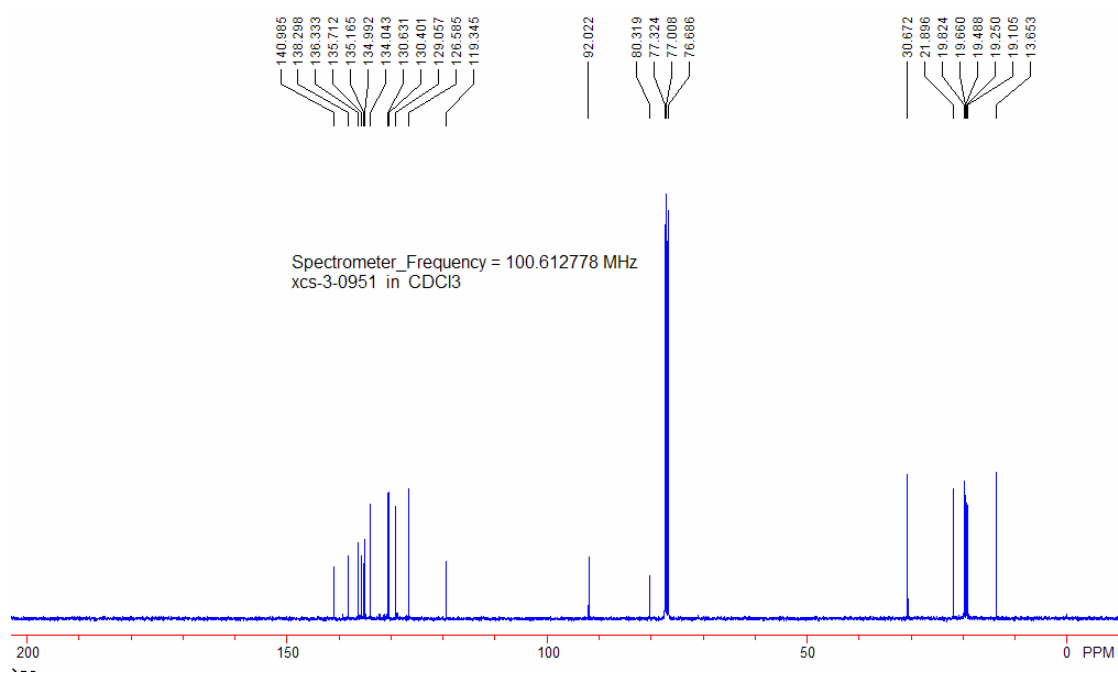
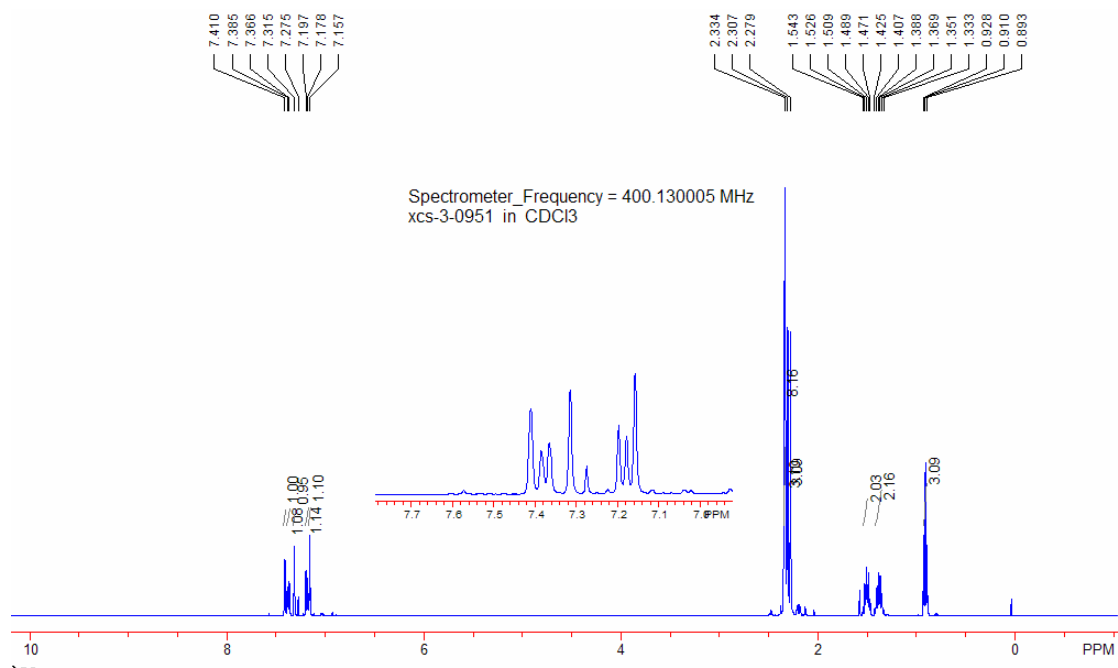
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.65-7.70 (m, 3 H), 7.49 (t, $J = 7.2$ Hz, 2 H), 7.40-7.45 (m, 3 H), 7.30-7.37 (m, 3 H), 7.00 (t, $J = 8.6$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.3 (d, $J = 247.4$ Hz), 143.8, 140.5, 133.1 (d, $J = 8.2$ Hz), 132.7, 129.4, 129.3, 128.5, 127.8, 127.4, 127.0, 121.3, 119.4 (d, $J = 3.9$ Hz), 115.5 (d, $J = 22.0$ Hz), 91.1, 89.0 (d, $J = 1.7$ Hz). HRMS (EI) Calcd for $\text{C}_{20}\text{H}_{13}\text{F}$: $[\text{M}]^+$ 272.1001; Found, 272.1003.

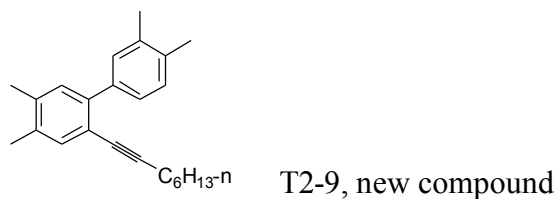




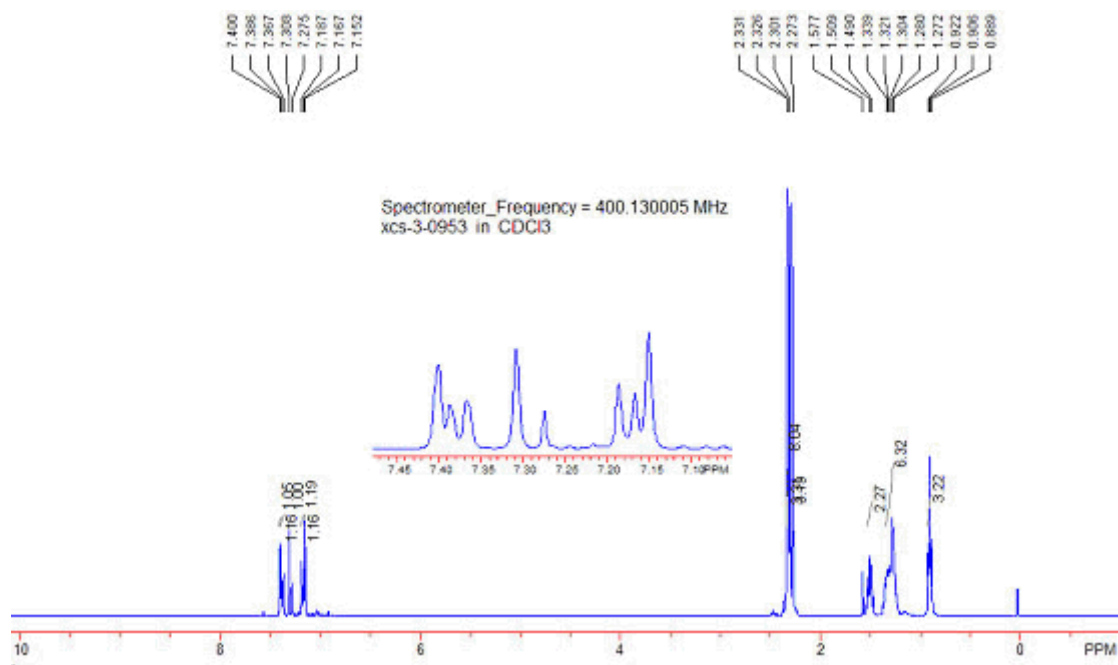
T2-8, new compound

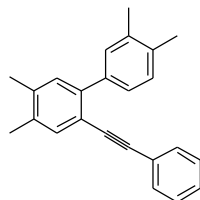
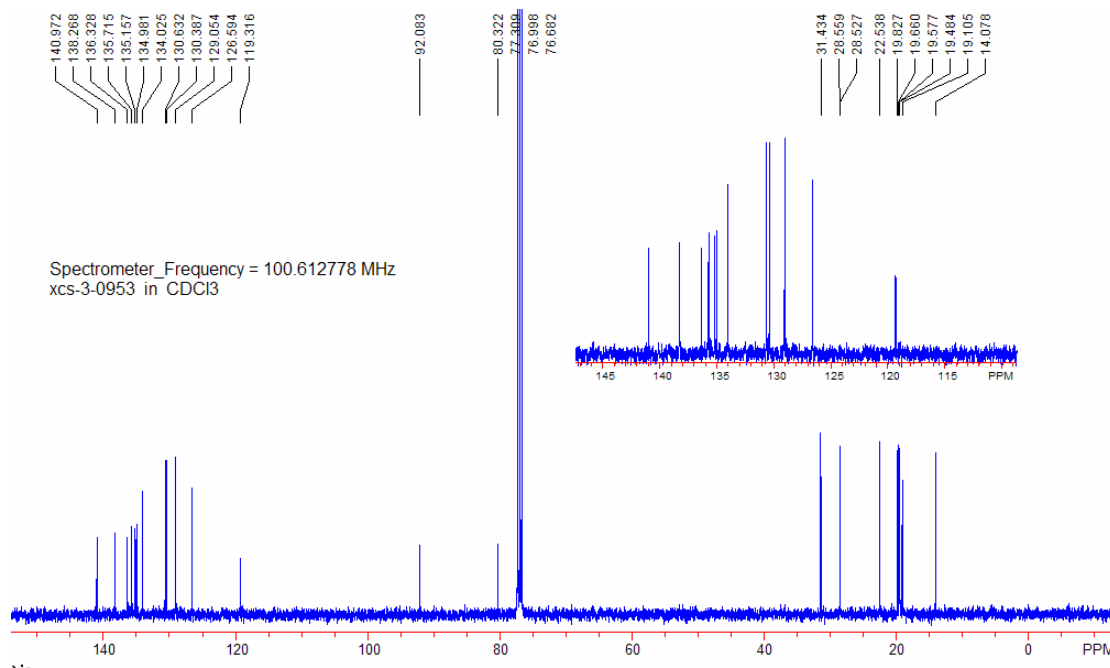
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.41 (s, 1 H), 7.37 (d, $J = 7.6$ Hz, 1 H), 7.32 (s, 1 H), 7.19 (d, $J = 7.6$ Hz, 1 H), 7.16 (s, 1 H), 2.31-2.35 (m, 8 H), 2.31 (s, 3 H), 2.28 (s, 3 H), 1.47-1.54 (m, 2 H), 1.33-1.42 (m, 2 H), 0.91 (t, $J = 7.0$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.9, 138.3, 136.3, 135.7, 135.2, 135.0, 134.0, 130.6, 130.4, 129.0, 126.6, 119.3, 92.0, 80.3, 30.7, 21.9, 19.8, 19.7, 19.5, 19.2, 19.1, 13.6. HRMS (EI) Calcd for $\text{C}_{22}\text{H}_{26}$: $[\text{M}]^+$ 290.2035; Found, 290.2037.





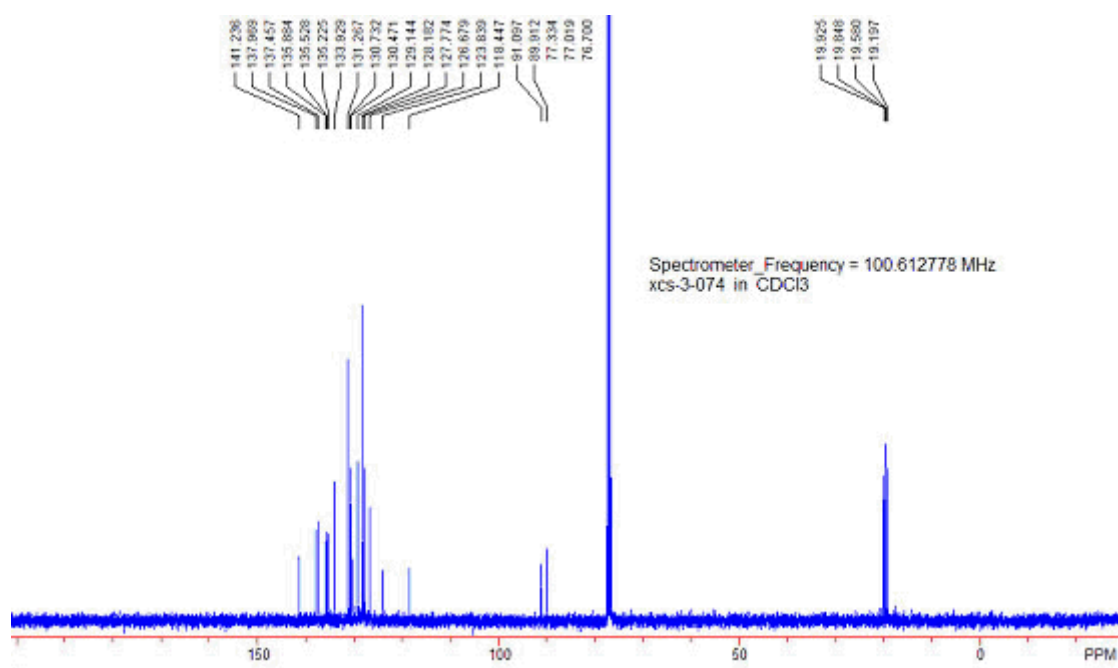
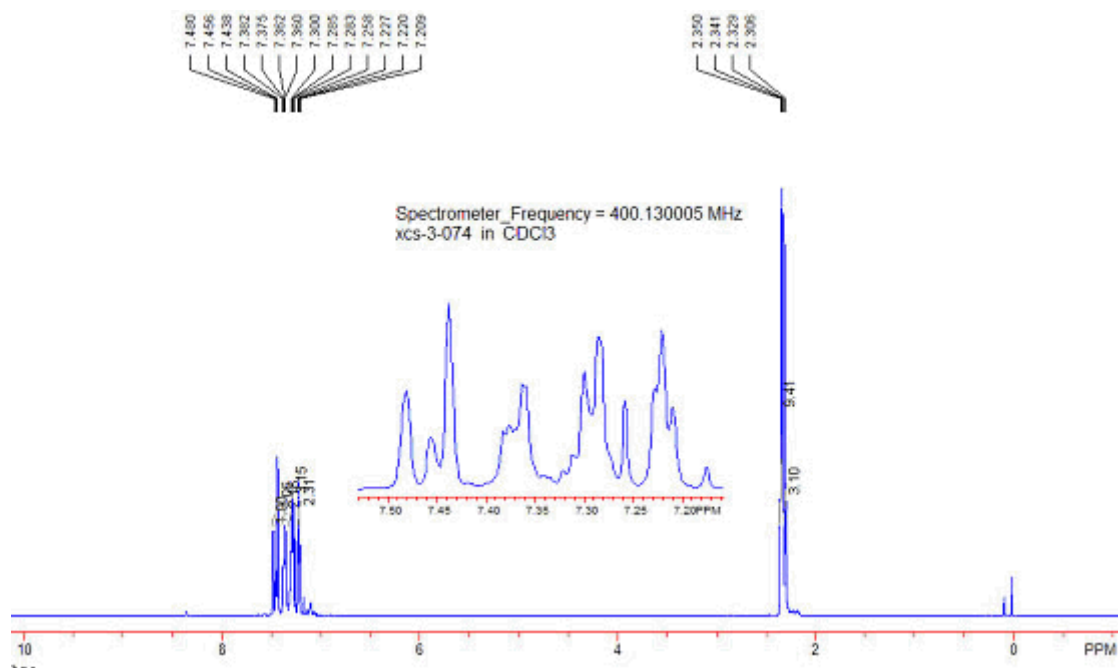
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.40 (s, 1 H), 7.38 (d, $J = 7.6$ Hz, 1 H), 7.31 (s, 1 H), 7.18 (d, $J = 8.0$ Hz, 1 H), 7.15 (s, 1 H), 2.32-2.33 (m, 8 H), 2.30 (s, 3 H), 2.27 (s, 3 H), 1.47-1.54 (m, 2 H), 1.27-1.38 (m, 6 H), 0.91 (t, $J = 6.6$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.9, 138.3, 136.3, 135.7, 135.2, 134.9, 134.0, 130.6, 130.4, 129.0, 126.6, 119.3, 92.1, 80.3, 31.4, 28.6, 28.5, 22.5, 19.8, 19.7, 19.6, 19.5, 19.1, 14.1. HRMS (EI) Calcd for $\text{C}_{24}\text{H}_{30}$: $[\text{M}]^+$ 318.2348; Found, 318.2350.

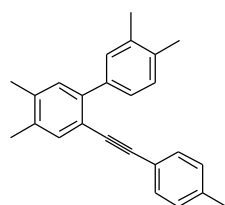




T2-10, new compound

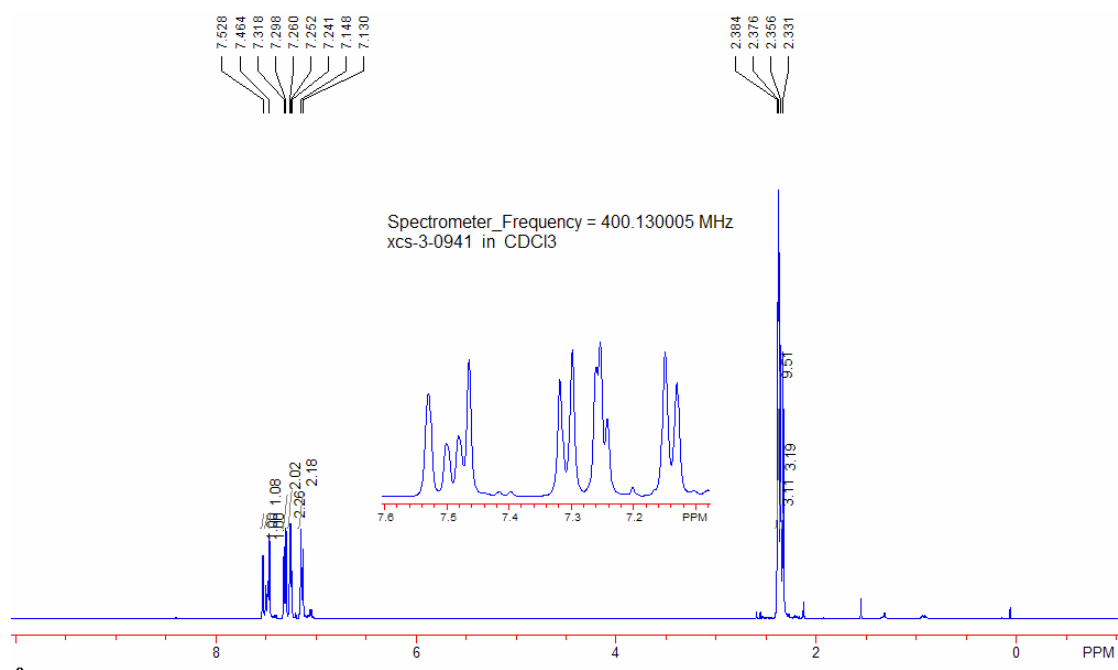
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.48 (s, 1 H), 7.45 (d, *J* = 7.2 Hz, 1 H), 7.44 (s, 1 H), 7.36-7.38 (m, 2 H), 7.28-7.30 (m, 3 H), 7.21-7.23 (m, 2 H), 2.35 (s, 3 H), 2.34 (s, 3 H), 2.33 (s, 3 H), 2.31 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 141.2, 138.0, 137.4, 135.9, 135.5, 135.2, 133.9, 131.3, 130.7, 130.5, 129.1, 128.2, 127.8, 126.7, 123.8, 118.4, 91.1, 89.9, 19.9, 19.8, 19.6, 19.2. HRMS (EI) Calcd for C₂₄H₂₂: [M]⁺ 310.1722; Found, 310.1724.

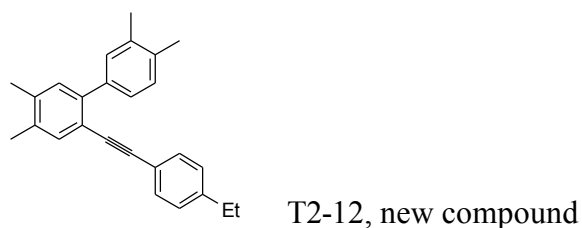
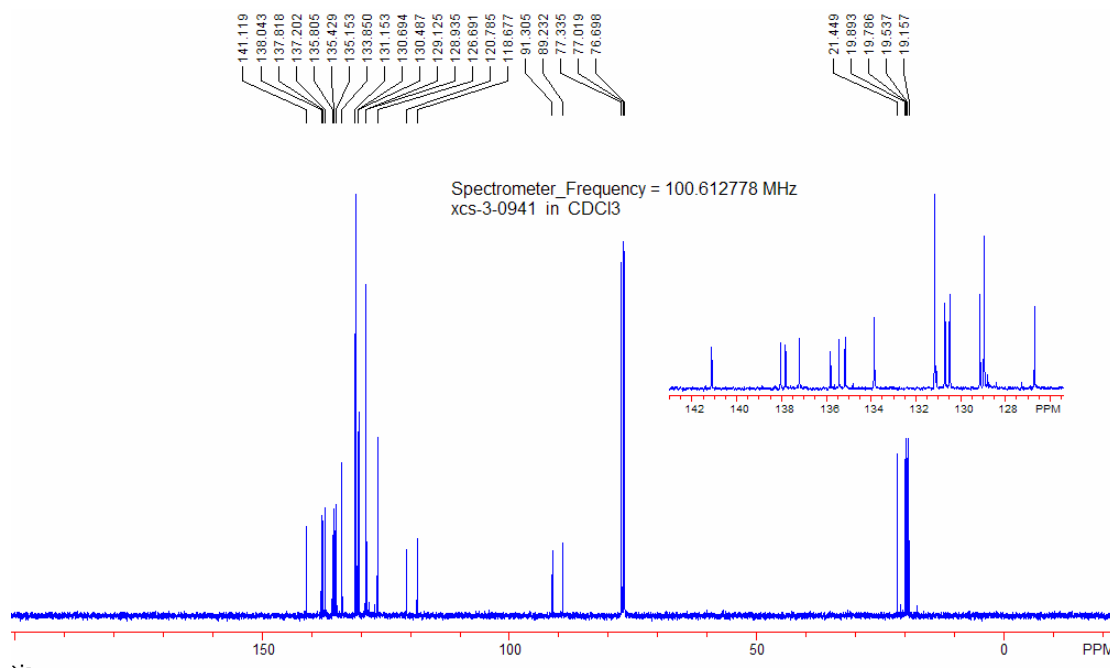




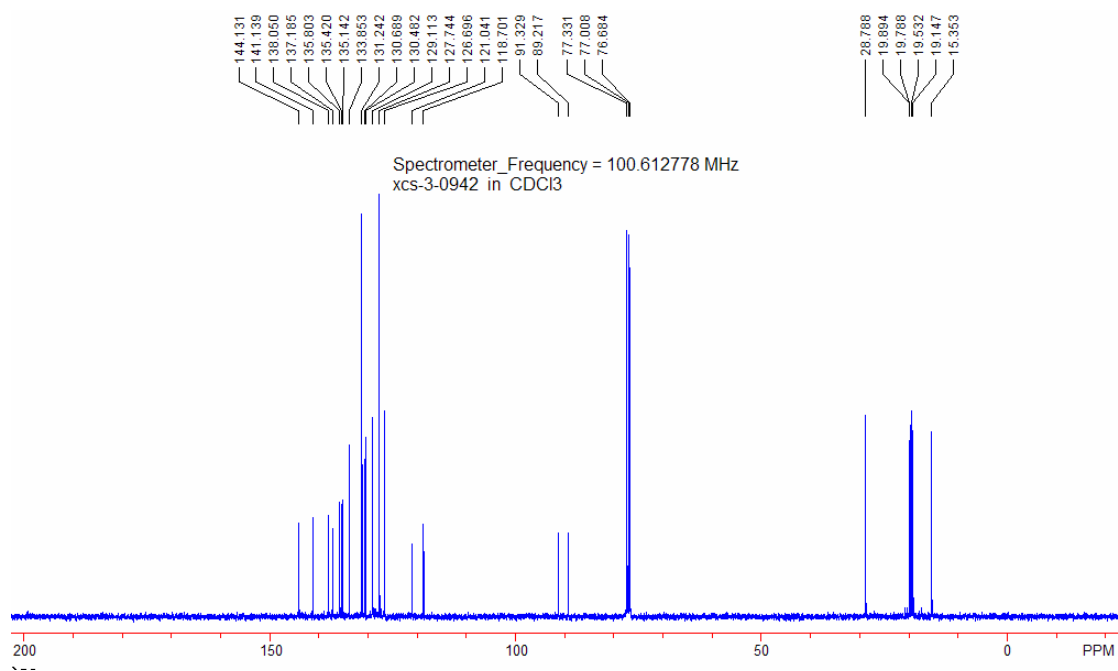
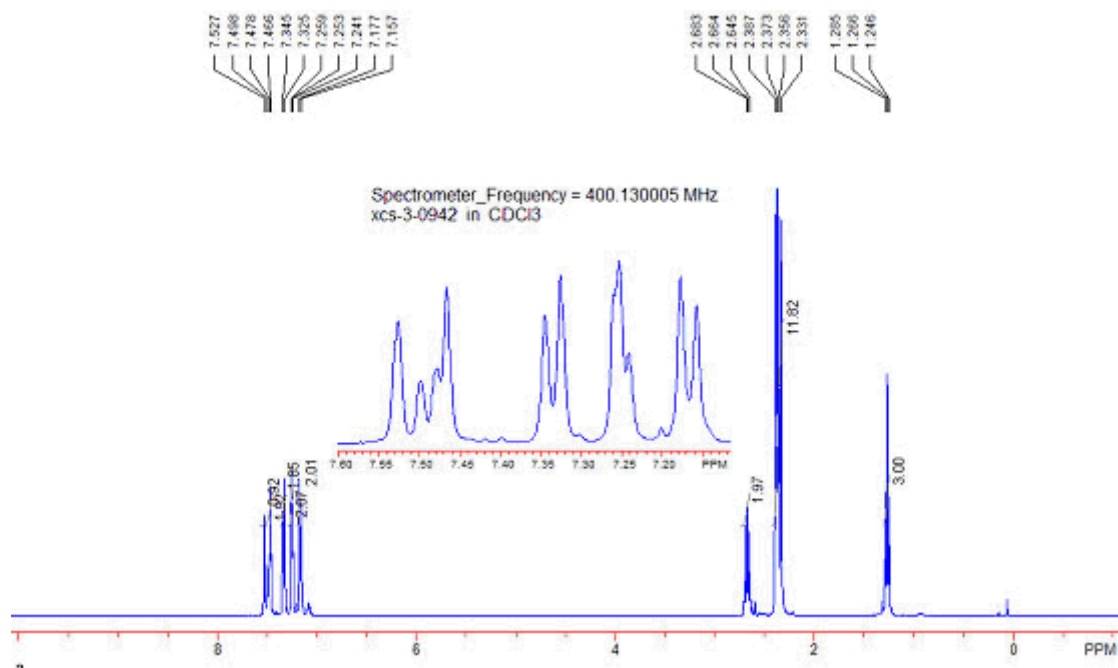
T2-11, new compound

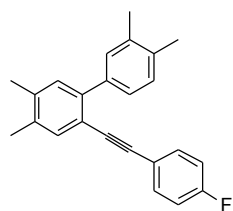
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.53 (s, 1 H), 7.49 (d, $J = 7.6$ Hz, 1 H), 7.46 (s, 1 H), 7.31 (d, $J = 8.0$ Hz, 2 H), 7.24-7.26 (m, 2 H), 7.14 (d, $J = 7.2$ Hz, 2 H), 2.38-2.37 (m, 9 H), 2.36 (s, 3 H), 2.33 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 141.1, 138.0, 137.8, 137.2, 135.8, 135.4, 135.2, 133.8, 131.2, 130.7, 130.5, 129.1, 128.9, 126.7, 120.8, 118.7, 91.3, 89.2, 21.4, 19.9, 19.8, 19.5, 19.2. HRMS (EI) Calcd for $\text{C}_{25}\text{H}_{24}$: $[\text{M}]^+$ 324.1878; Found, 324.1876.





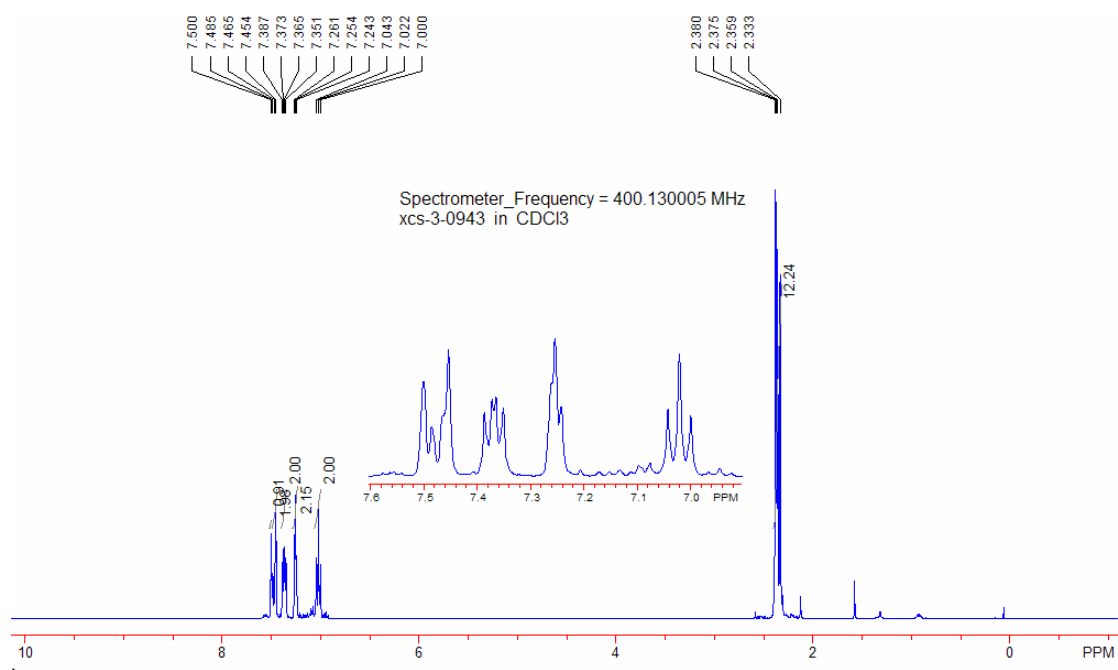
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.53 (s, 1 H), 7.49 (d, *J* = 8.0 Hz, 1 H), 7.47 (s, 1 H), 7.34 (d, *J* = 8.0 Hz, 2 H), 7.24-7.26 (m, 2 H), 7.17 (d, *J* = 8.0 Hz, 2 H), 2.67 (q, *J* = 7.6 Hz, 2 H), 2.39 (s, 3 H), 2.37 (s, 3 H), 2.36 (s, 3 H), 2.33 (s, 3 H), 1.27 (t, *J* = 7.8 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 144.1, 141.1, 138.0, 137.2, 135.8, 135.4, 135.1, 133.8, 131.2, 130.7, 130.5, 129.1, 127.7, 126.7, 121.0, 118.7, 91.3, 89.2, 28.8, 19.9, 19.8, 19.5, 19.1, 15.3. HRMS (EI) Calcd for C₂₆H₂₆: [M]⁺ 338.2035; Found, 338.2030.

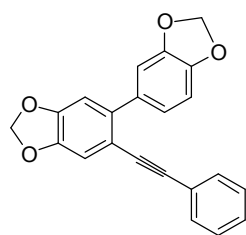
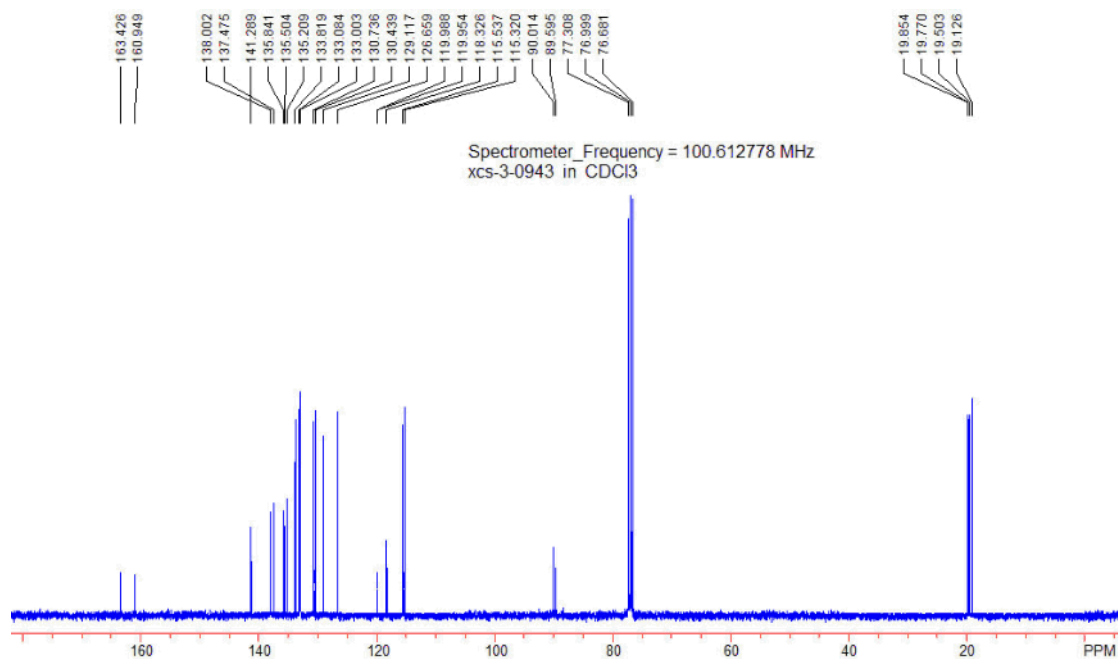




T2-13, new component

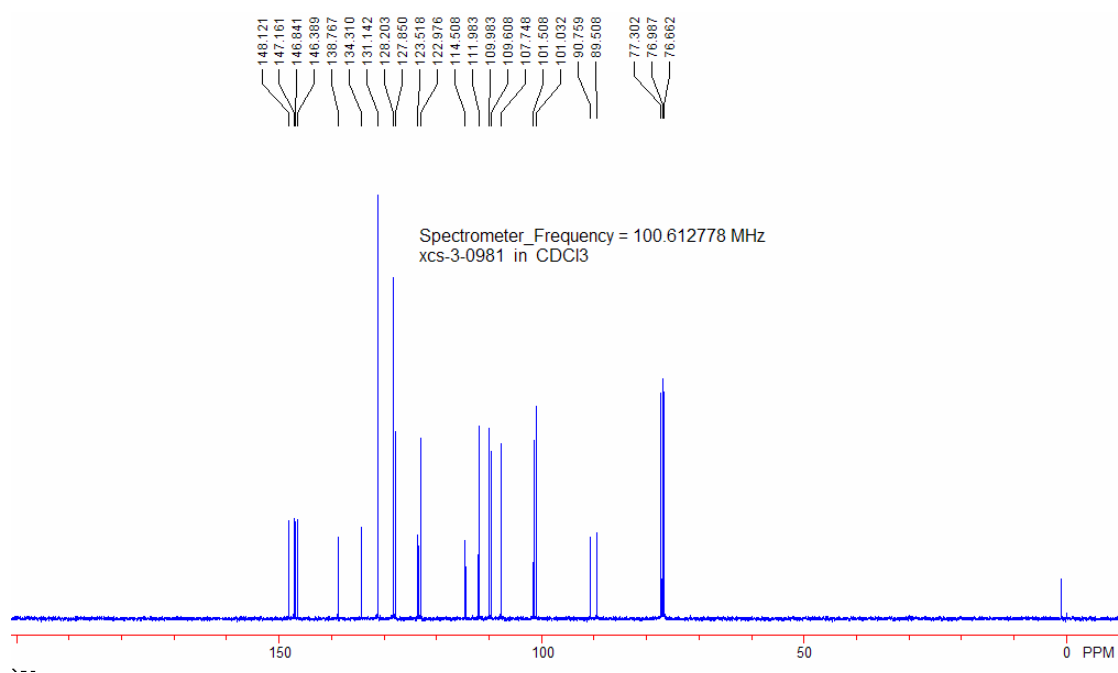
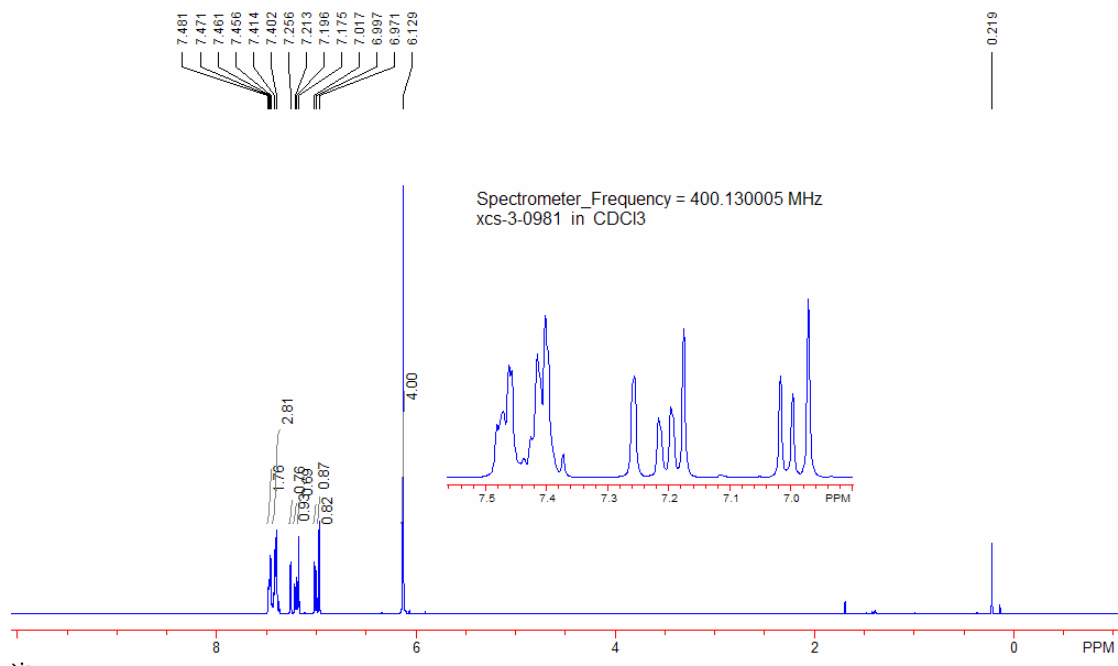
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.50 (s, 1 H), 7.45-7.48 (m, 2 H), 7.35-7.39 (m, 2 H), 7.24-7.26 (m, 2 H), 7.02 (t, $J = 8.6$ Hz, 2 H), 2.38 (s, 3 H), 2.37 (s, 3 H), 2.36 (s, 3 H), 2.33 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.2 (d, $J = 247.7$ Hz), 141.3, 138.0, 137.5, 135.8, 135.5, 135.2, 133.8, 133.0 (d, $J = 8.1$ Hz), 130.7, 130.4, 129.1, 126.6, 119.9 (d, $J = 3.4$ Hz), 118.3, 115.4 (d, $J = 21.7$ Hz), 90.0, 89.6, 19.9, 19.8, 19.5, 19.1. HRMS (EI) Calcd for $\text{C}_{24}\text{H}_{21}\text{F}$: $[\text{M}]^+$ 328.1627; Found, 328.1621.

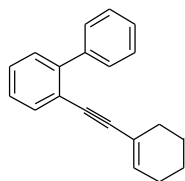




T2-14, new compound

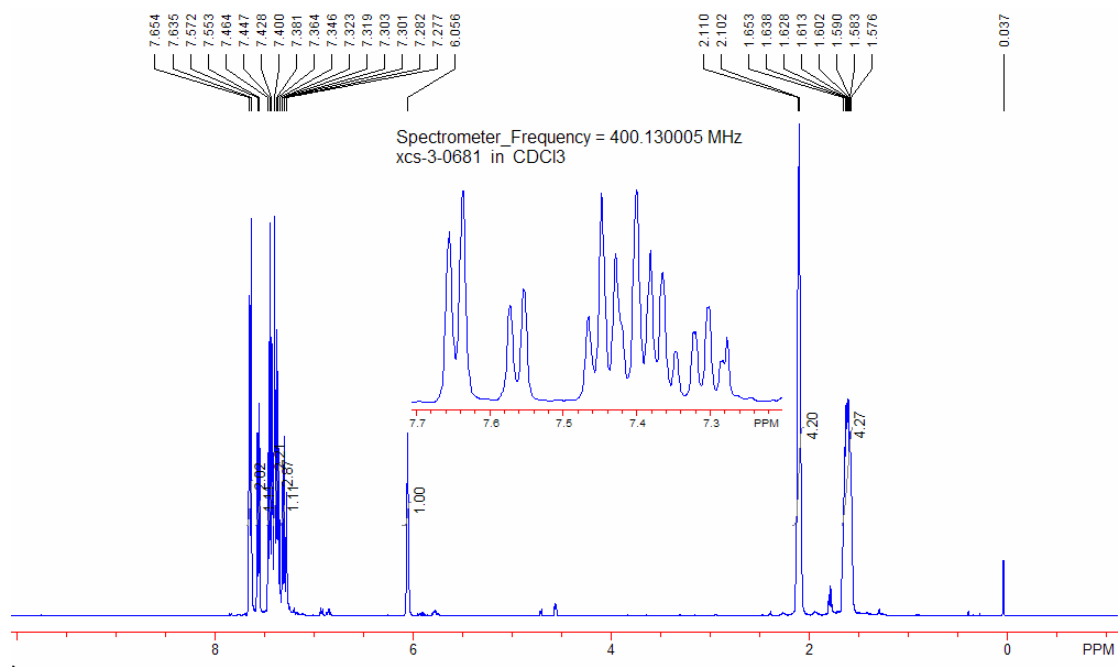
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.46-7.48 (m, 2 H), 7.40-7.42 (m, 3 H), 7.26 (s, 1 H), 7.20 (d, *J* = 6.8 Hz, 1 H), 7.18 (s, 1 H), 7.01 (d, *J* = 8.0 Hz, 1 H), 6.97 (s, 1 H), 6.13 (s, 4 H); ¹³C NMR (100 MHz, CDCl₃) δ 148.1, 147.2, 146.8, 146.4, 138.8, 134.3, 131.1, 128.2, 127.8, 123.5, 123.0, 114.5, 112.0, 110.0, 109.6, 107.7, 101.5, 101.0, 90.8, 89.5. HRMS (EI) Calcd for C₂₂H₁₄O₄: [M]⁺ 342.0892; Found, 342.0898.

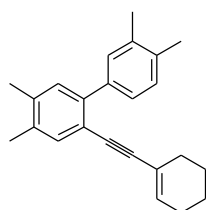
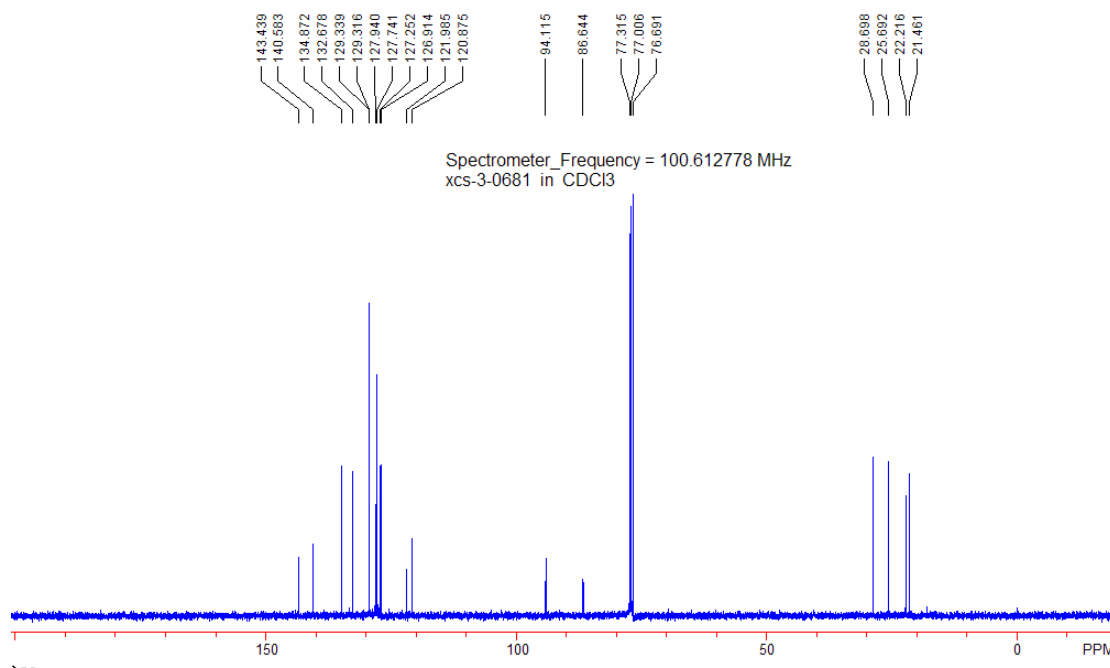




T2-15, 851901-88-7, Ref. 5

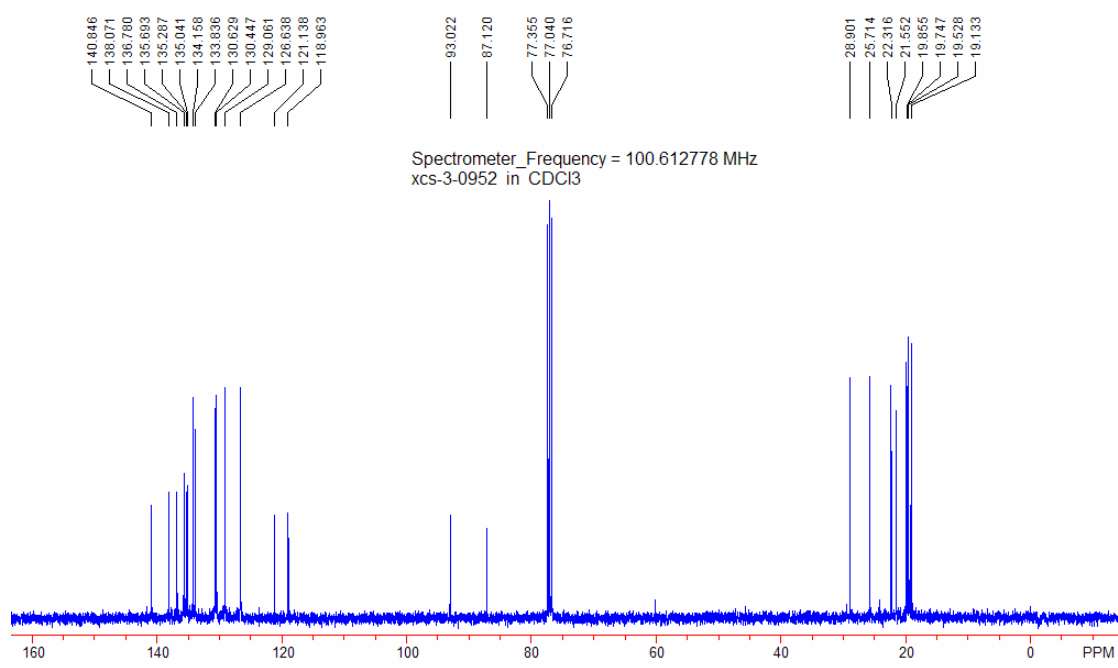
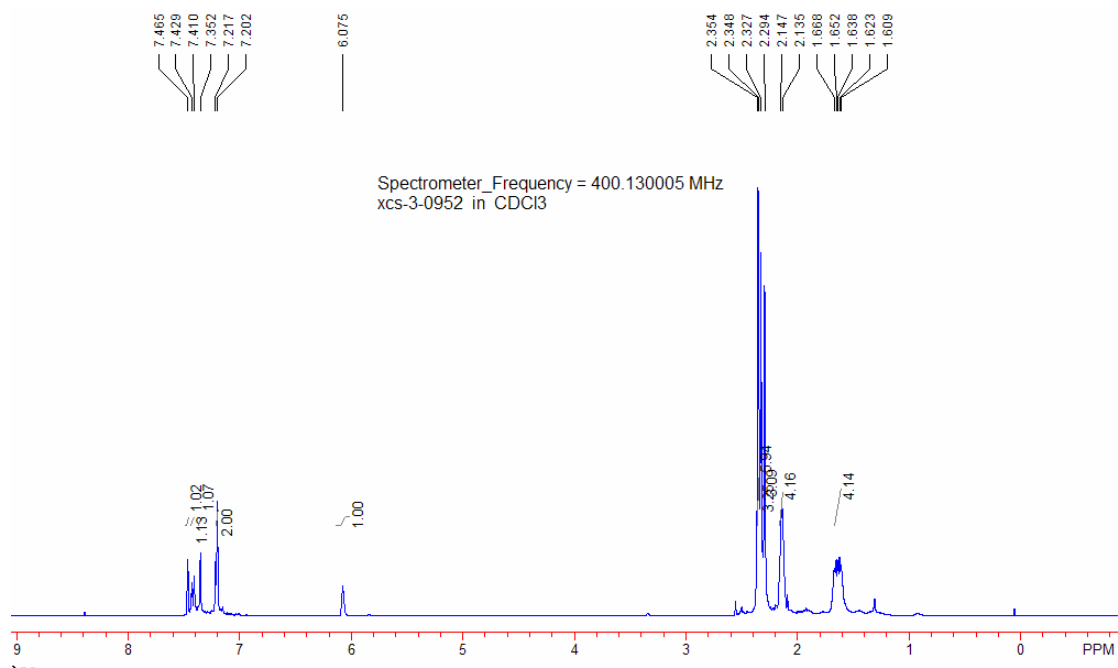
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.64 (d, $J = 7.6$ Hz, 2 H), 7.56 (d, $J = 7.6$ Hz, 1 H), 7.45 (t, $J = 7.2$ Hz, 2 H), 7.35-7.40 (m, 3 H), 7.30 (t, $J = 8.2$ Hz, 1 H), 6.06 (s, 1 H), 2.10-2.11 (m, 4 H), 1.58-1.65 (m, 4 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.4, 140.6, 134.9, 132.7, 129.34, 129.32, 127.9, 127.7, 127.2, 126.9, 122.0, 120.9, 94.1, 86.6, 28.7, 26.7, 22.2, 21.5. HRMS (EI) Calcd for $\text{C}_{20}\text{H}_{18}$: $[\text{M}]^+$ 258.1409; Found, 258.1405.

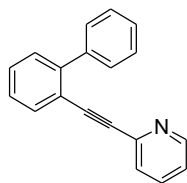




T2-16, new compound

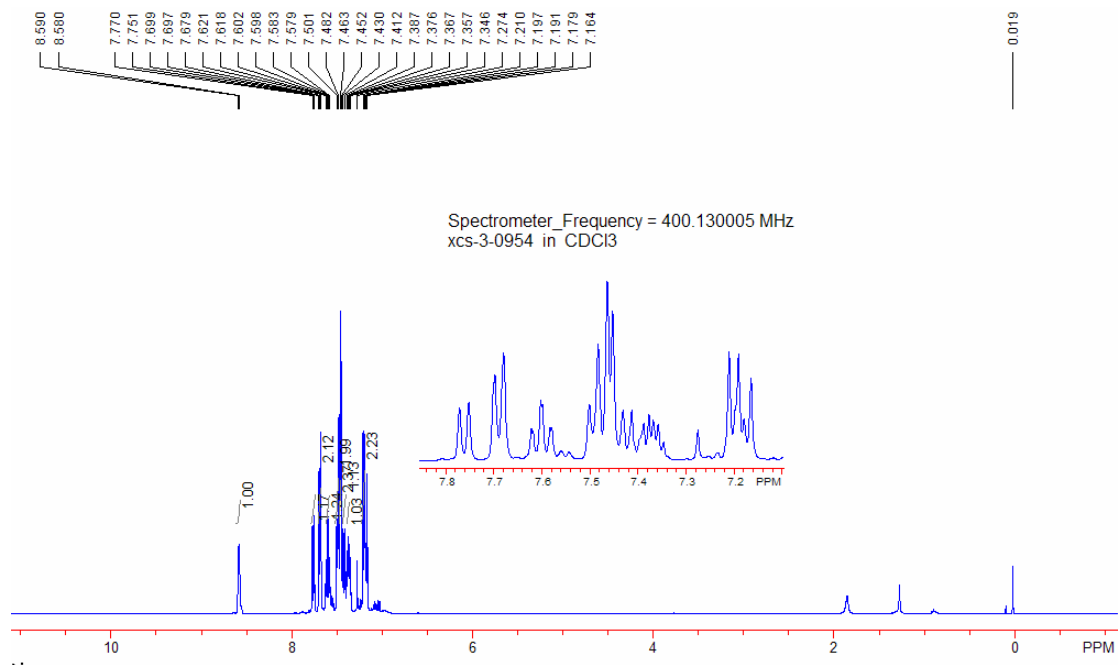
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.46 (s, 1 H), 7.42 (d, *J* = 7.6 Hz, 1 H), 7.35 (s, 1 H), 7.21 (d, *J* = 6.0 Hz, 1 H), 7.20 (s, 1 H), 6.08 (s, 1 H), 2.35 (s, 3 H), 2.34 (s, 3 H), 2.33 (s, 3 H), 2.29 (s, 3 H), 2.14-2.15 (m, 4 H), 1.61-1.67 (m, 4 H); ¹³C NMR (100 MHz, CDCl₃) δ 140.8, 138.1, 136.8, 135.7, 135.3, 135.0, 134.2, 133.8, 130.6, 130.4, 129.1, 126.6, 121.1, 118.9, 93.0, 87.1, 28.9, 25.7, 22.3, 21.6, 19.8, 19.7, 19.5, 19.1. HRMS (EI) Calcd for C₂₄H₂₆: [M]⁺ 314.2035; Found, 314.2034.

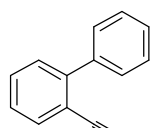
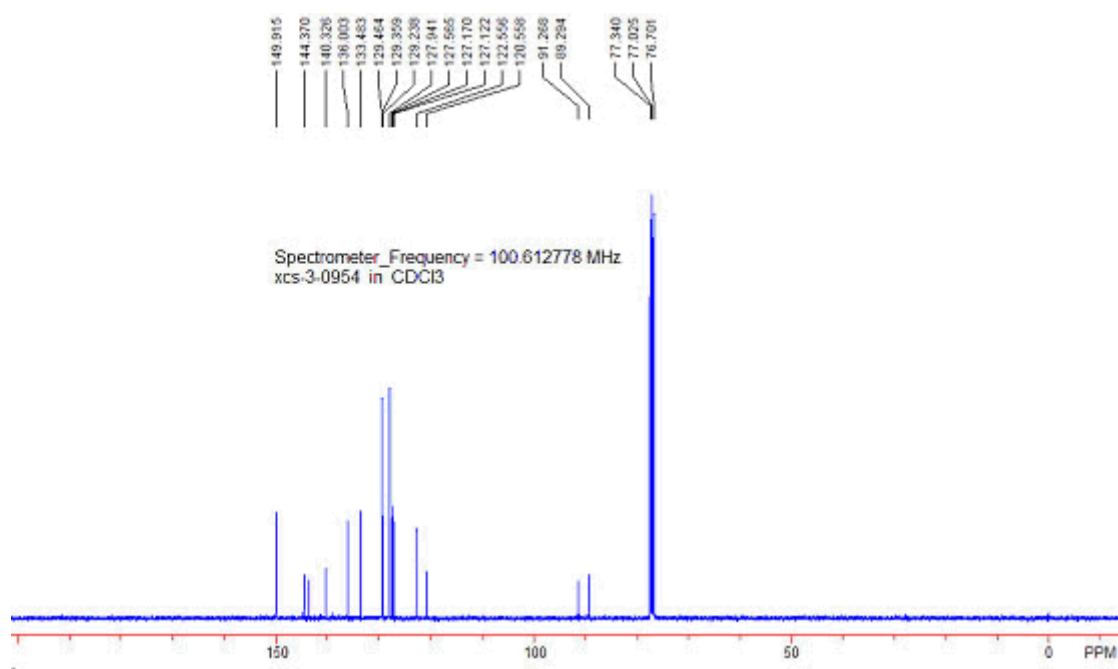




T2-17, new compound

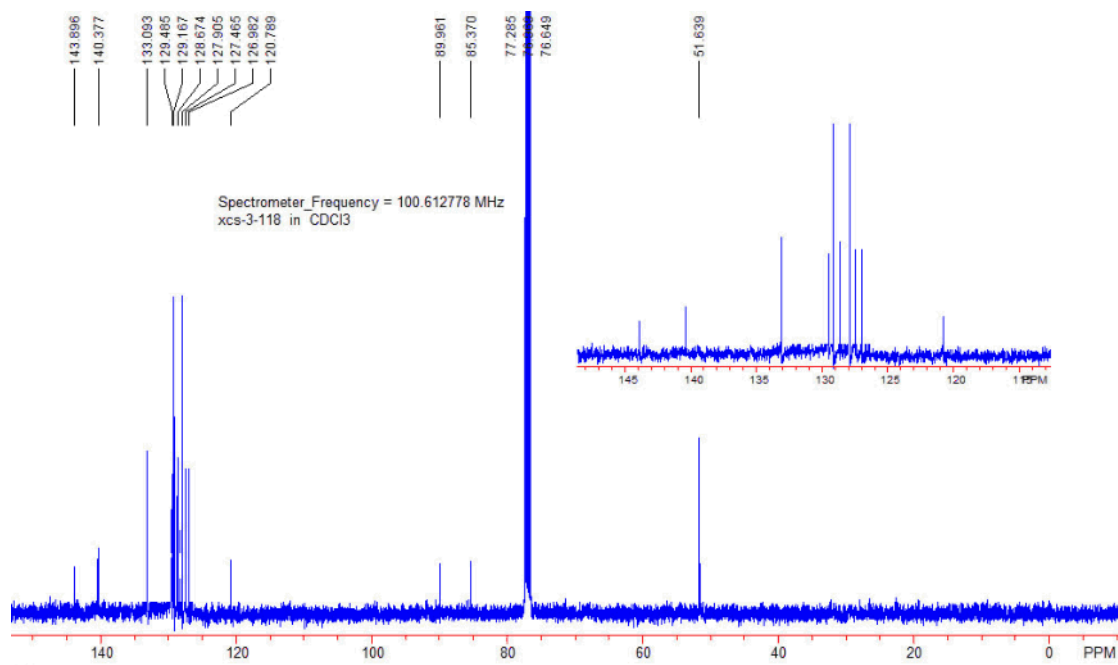
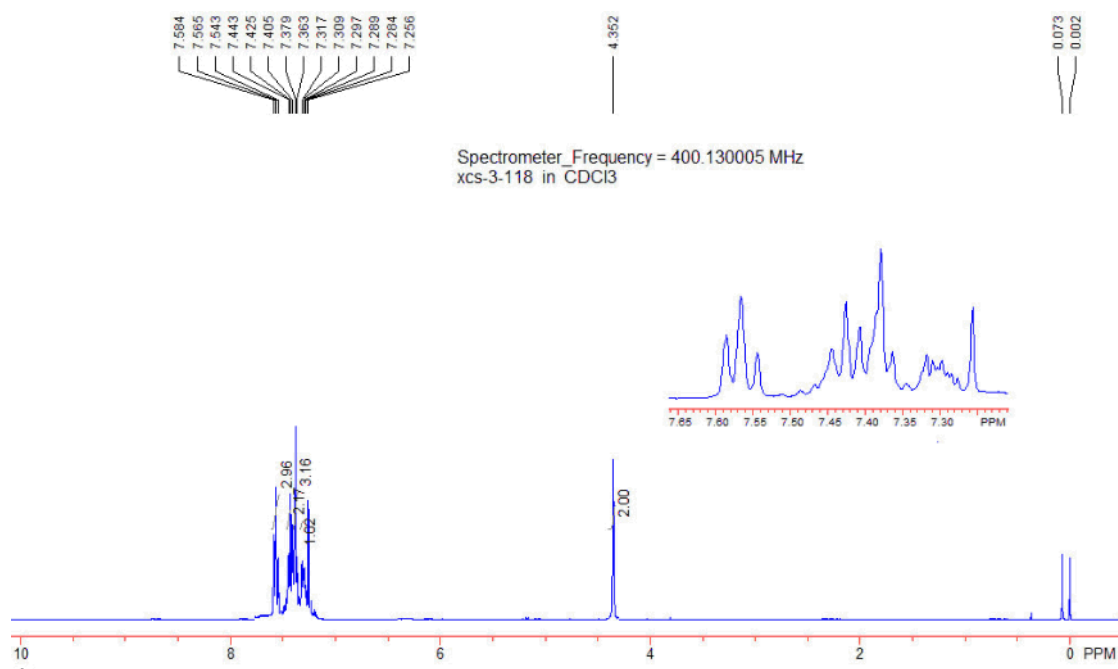
^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.58 (d, $J = 4.0$ Hz, 1 H), 7.76 (d, $J = 7.6$ Hz, 1 H), 7.69 (d, $J = 7.2$ Hz, 2 H), 7.60 (t, $J = 7.6$ Hz, 1 H), 7.49 (d, $J = 7.6$ Hz, 2 H), 7.45 (d, $J = 4.4$ Hz, 2 H), 7.42 (d, $J = 7.2$ Hz, 1 H), 7.37 (m, 1 H), 7.20 (d, $J = 7.6$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.9, 144.4, 140.3, 136.0, 133.5, 129.5, 129.4, 129.2, 127.9, 127.6, 127.2, 127.1, 122.6, 120.6, 91.3, 89.3. HRMS (EI) Calcd for $\text{C}_{19}\text{H}_{13}\text{N}$: $[\text{M}]^+$ 255.1048; Found, 255.1041.

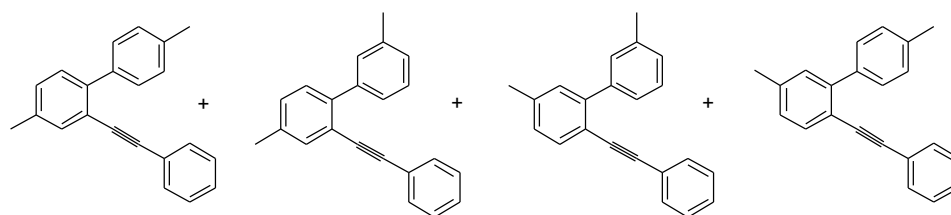




OH T2-18, 1001920-52-0, Ref. 6

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.57 (d, J = 7.6 Hz, 2 H), 7.55 (d, J = 8.8 Hz, 1 H), 7.43 (t, J = 7.6 Hz, 2 H), 7.36-7.40 (m, 3 H), 7.30 (m, 1 H), 4.35 (s, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.9, 140.4, 133.1, 129.5, 129.2, 128.7, 127.9, 127.5, 126.9, 120.8, 89.9, 85.4, 51.6. MS (EI): m/z : 208 [M^+], 190, 178, 165, 152, 94.

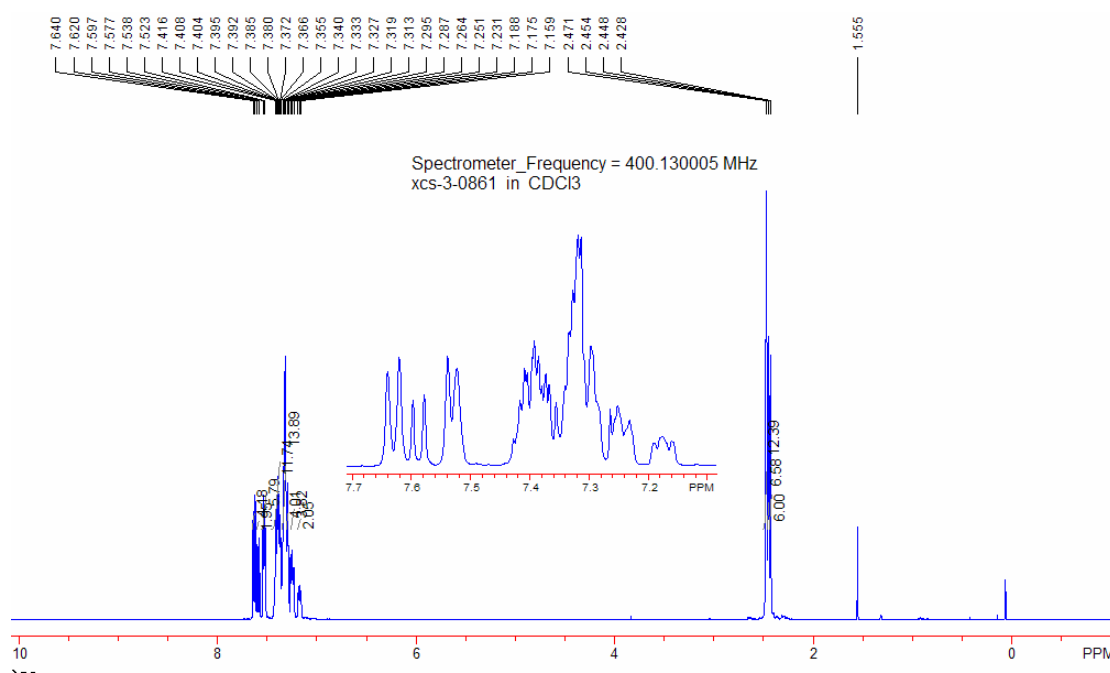


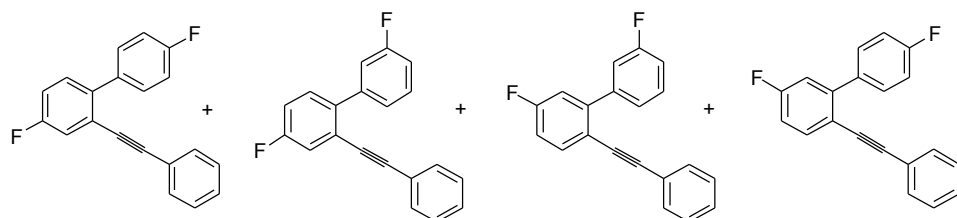
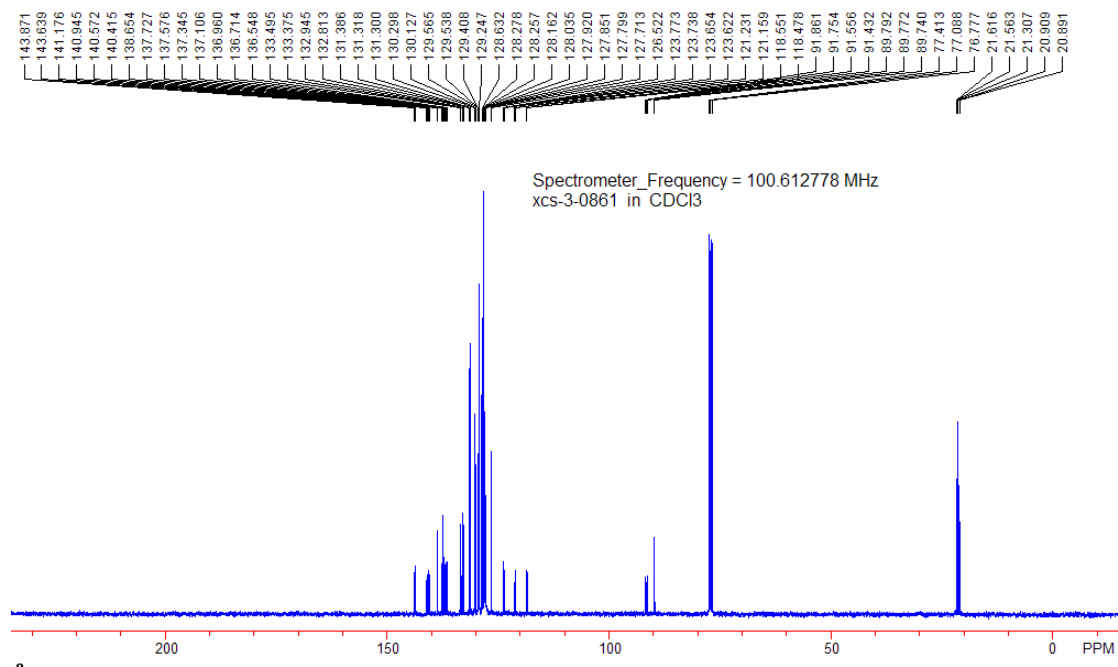


Scheme 3-1

223114-17-8, Ref. 7 new compound new compound 223114-14-5, Ref. 7

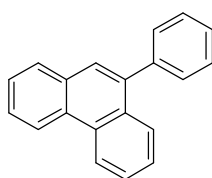
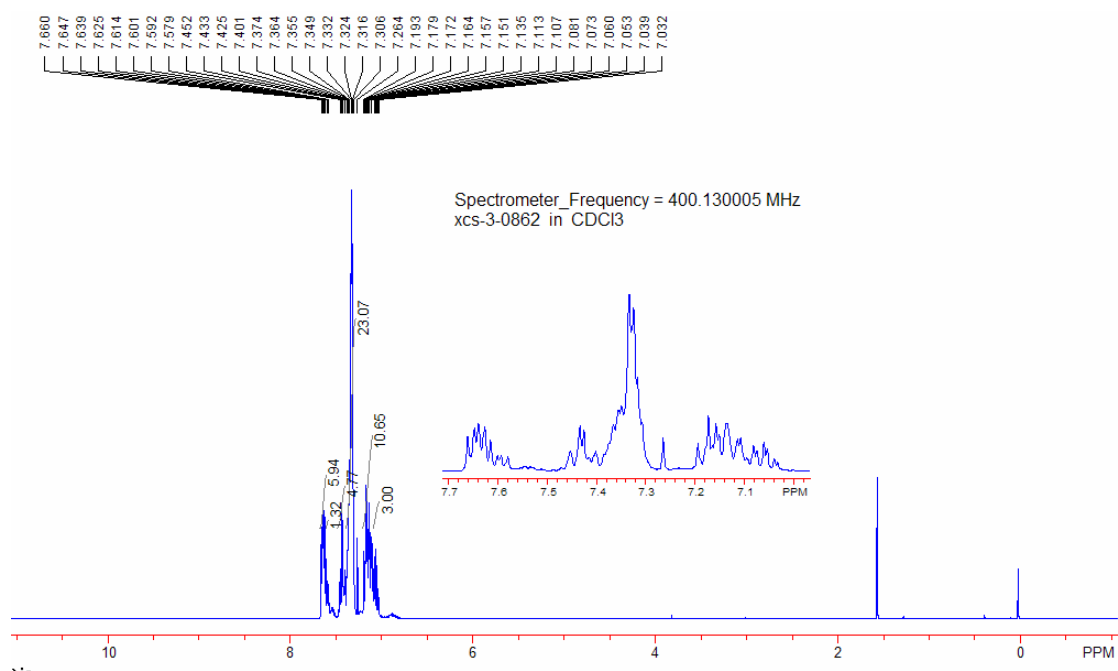
^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.63 (d, $J = 8.0$ Hz, 4 H), 7.59 (d, $J = 8.0$ Hz, 2 H), 7.52-7.54 (m, 6 H), 7.37-7.43 (m, 12 H), 7.29-7.34 (m, 14 H), 7.26-7.29 (m, 4 H), 7.23-7.26 (m, 4 H), 7.16-7.19 (m, 2 H), 2.46-2.47 (m, 12 H), 2.45-2.46 (m, 6 H), 2.43-2.44 (m, 6 H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.9, 143.6, 141.2, 140.9, 140.6, 140.4, 138.6, 137.7, 137.6, 137.3, 137.1, 137.0, 136.7, 136.5, 133.5, 133.4, 132.9, 132.8, 131.4, 131.3, 130.3, 130.1, 129.6, 129.5, 129.4, 129.2, 128.6, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 126.5, 123.8, 123.7, 123.6, 121.2, 121.1, 118.6, 118.5, 91.9, 91.8, 91.6, 91.4, 89.8, 89.7, 21.6, 21.5, 21.3, 20.9, 20.8. HRMS (EI) Calcd for $\text{C}_{22}\text{H}_{18}$: $[\text{M}]^+$ 282.1409; Found, 282.1406.





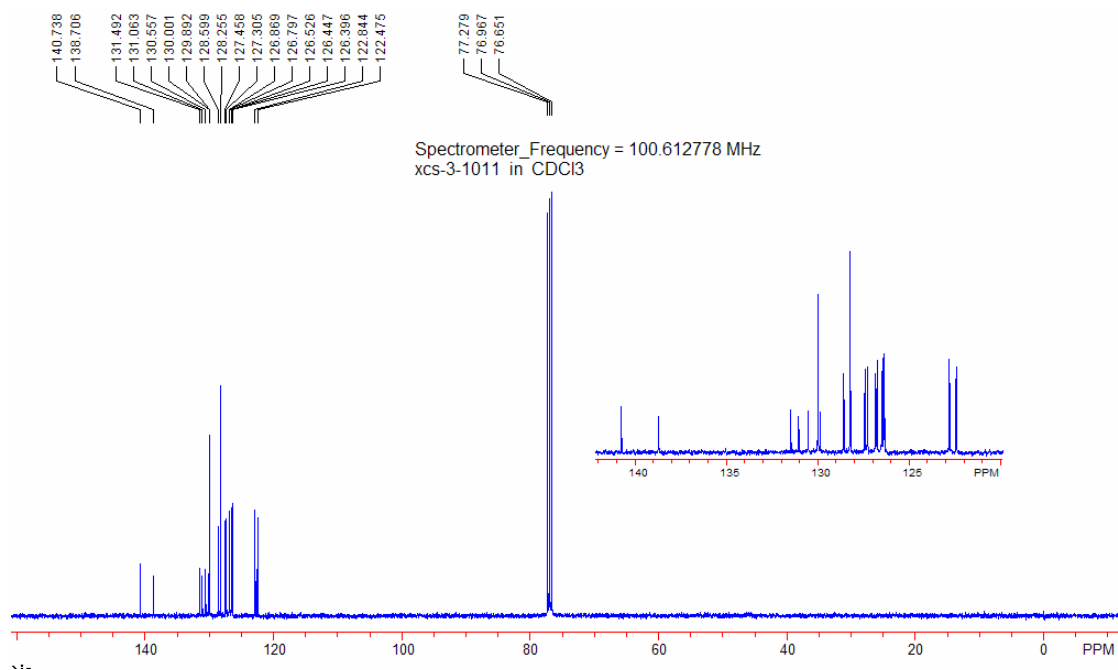
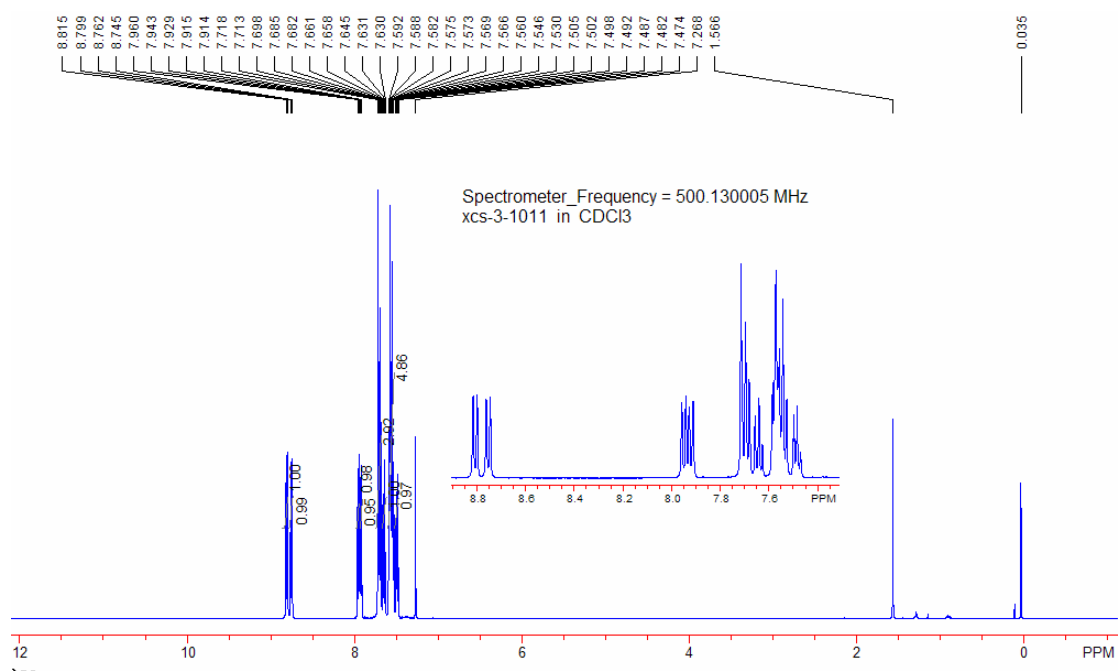
Scheme 3-2, new compounds

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.61-7.66 (m, 6 H), 7.59 (d, $J = 7.6$ Hz, 1 H), 7.40-7.45 (m, 5 H), 7.31-7.37 (m, 23 H), 7.11-7.19 (m, 10 H), 7.03-7.08 (m, 3 H); HRMS (EI) Calcd for $\text{C}_{20}\text{H}_{12}\text{F}_2$: $[\text{M}]^+$ 290.0907; Found, 290.0899.



Scheme 4, 844-20-2, Ref. 4

¹H NMR (500 MHz, CDCl₃, TMS) δ 8.80 (d, *J* = 8.0 Hz, 1 H), 8.75 (d, *J* = 8.5 Hz, 1 H), 7.95 (d, *J* = 8.5 Hz, 1 H), 7.92 (d, *J* = 7.5 Hz, 1 H), 7.68-7.72 (m, 3 H), 7.64 (t, *J* = 7.5 Hz, 1 H), 7.53-7.59 (m, 5 H), 7.49 (t, *J* = 7.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 140.7, 138.7, 131.5, 131.1, 130.6, 130.0, 129.9, 128.6, 128.2, 127.4, 127.3, 126.9, 126.8, 126.5, 126.4, 126.3, 122.8, 122.5. MS (EI): *m/z* (%): 254 (100) [M⁺], 126 (30), 113 (15).



References

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