

Supporting Information

Reductive Coupling of Azonaphthalenes for the Synthesis of BINAMs via a Diboron-Enabled [5,5]-Sigmatropic Rearrangement

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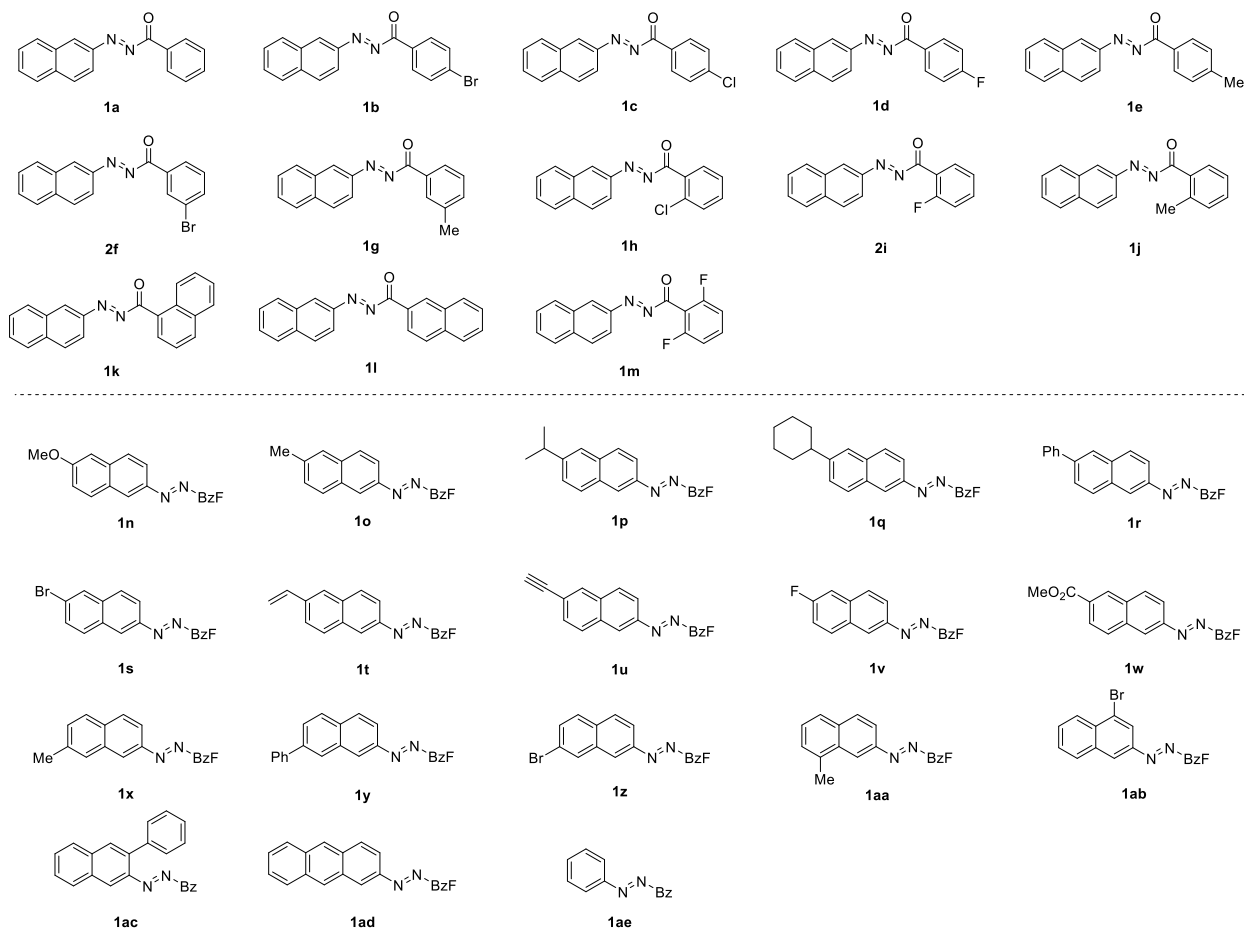
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General Information

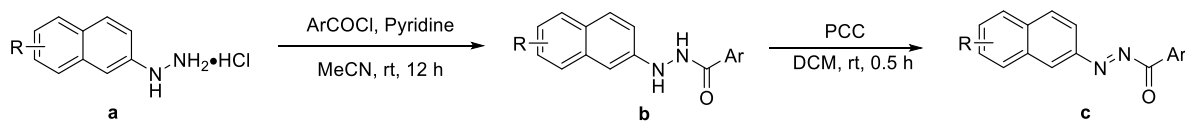
Unless otherwise stated, all reagents and solvents were purchased from reputable commercial sources and used without further purification. Flash column chromatography was performed on silica gel with a particle size range of 200-300 mesh. Analytical thin-layer chromatography was performed on precoated silica gel plates (60F-254) and visualized with a UV light source emitting at 254 nm. Both ^1H and ^{13}C NMR spectra were acquired on a Bruker spectrometer, operating at either 400 MHz or 500 MHz and were referenced to tetramethylsilane as the internal standard. Chemical shifts were reported as parts per million (ppm) relative to the signals of CDCl_3 and $\text{DMSO-}d_6$. Additionally, chemical constants were expressed in Hertz (Hz), measured downfield from tetramethylsilane. Anhydrous 1,2-dimethoxyethane was purchased from Sigma-Aldrich and used in its unaltered state. High-resolution mass spectrometric analysis reported as the mass-to-charge ratio (m/z), was performed using Agilent 7200 QTOF and Bruker MicroTOF-QII spectrometers.

Substrates explored in the manuscript



General procedure for the synthesis of azonaphthalenes

Azonaphthalenes were prepared according to the reported literature.¹⁻³

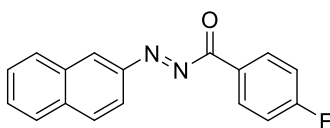


To a solution of 2-naphthylhydrazine hydrochloride (10 mmol) in acetonitrile (20 mL), benzoyl chloride (11 mmol) and pyridine (1.8 mL, 22 mmol) were added and stirred overnight at room temperature. After completion of the reaction, the solvent was concentrated in vacuo and diluted with ethyl acetate (50 mL). The organic phase was carefully washed with saturated aqueous sodium bicarbonate (50 mL) and extracted with ethyl acetate (3 x 50 mL). The combined organic phase was washed with brine, dried over sodium sulphate. After removal of the solvent, the crude

product was then purified by flash chromatography on silica gel eluted with *n*-hexane: ethyl acetate (5:1 to 2:1).

Pyridinium chlorochromate (PCC) (5 mmol) was added to a solution of naphthyl hydrazide (5 mmol) in dichloromethane (20 mL) and the reaction mixture was stirred at room temperature until the complete consumption of the starting material. The mixture was filtered, concentrated and purified by flash chromatography eluting with *n*-hexane: ethyl acetate (10:1).

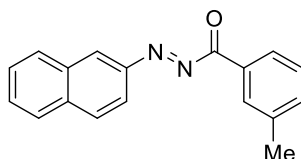
(*E*)-(4-fluorophenyl)(naphthalen-2-yl diazenyl) methanone (1d)



1d

Red solid, 55% yield; R_f 0.61 (*n*-hexane: ethyl acetate = 10:1). **$^1\text{H NMR}$ (500 MHz, CDCl_3)** δ 8.68 (s, 1H), 8.25 – 8.16 (m, 2H), 8.09 (d, $J = 8.0$ Hz, 1H), 8.02 (dd, $J = 8.9, 1.9$ Hz, 1H), 7.96 (dd, $J = 8.4, 3.6$ Hz, 2H), 7.70 – 7.62 (m, 2H), 7.24 (t, $J = 8.6$ Hz, 2H). **$^{13}\text{C NMR}$ (126 MHz, CDCl_3)** δ 180.4, 166.6 (d, $J = 257.3$ Hz), 149.8, 136.0, 133.5 (2C) (d, $J = 9.6$ Hz), 133.2, 132.3, 130.0, 129.6, 129.1, 128.1, 127.7 (d, $J = 3.1$ Hz), 127.3, 116.3 (2C) (d, $J = 22.1$ Hz), 115.4. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}$, m/z : 279.0930, found: 279.0928.

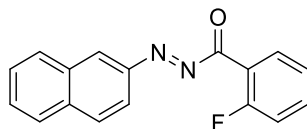
(*E*)-(naphthalen-2-yl diazenyl) (m-tolyl)methanone (1g)



1g

Red solid, 47% yield; R_f 0.50 (*n*-hexane: ethyl acetate = 10:1). **$^1\text{H NMR}$ (500 MHz, CDCl_3)** δ 8.67 (d, $J = 1.5$ Hz, 1H), 8.08 (d, $J = 7.9$ Hz, 1H), 8.04 (dd, $J = 8.9, 1.9$ Hz, 1H), 7.99 – 7.92 (m, 4H), 7.69 – 7.61 (m, 2H), 7.50 (d, $J = 7.6$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 1H), 2.46 (s, 3H). **$^{13}\text{C NMR}$ (126 MHz, CDCl_3)** δ 182.2, 149.8, 138.8, 135.9, 135.4, 133.2, 131.8, 131.1, 131.0, 129.9, 129.2, 128.9, 128.8, 128.1, 127.9, 127.2, 115.5, 21.4. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{15}\text{N}_2\text{O}$, m/z : 275.1179, found: 275.1181.

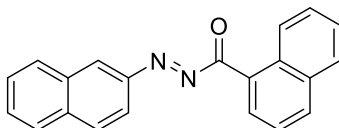
(*E*)-(2-fluorophenyl) (naphthalen-2-yl diazenyl) methanone (1i)



1i

Red solid, 51% yield; R_f 0.53 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.61 (d, $J = 1.9$ Hz, 1H), 8.19 – 8.12 (m, 1H), 8.09 – 8.02 (m, 1H), 8.01 (d, $J = 1.9$ Hz, 1H), 7.98 – 7.92 (m, 2H), 7.71 – 7.59 (m, 3H), 7.39 – 7.33 (m, 1H), 7.24 – 7.16 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.8, 162.5 (d, $J = 261.1$ Hz), 149.8, 136.3 (d, $J = 9.1$ Hz), 135.9, 133.2, 132.6, 131.5, 129.9, 129.5, 128.9, 128.1, 127.2, 124.6 (d, $J = 3.6$ Hz), 119.6 (d, $J = 11.4$ Hz), 117.2 (d, $J = 22.1$ Hz), 115.7. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{12}\text{FN}_2\text{O}$, m/z : 279.0928, found: 279.0928.

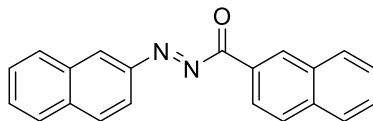
(E)-naphthalen-1-yl(naphthalen-2-yl) diazenyl methanone (1k)



1k

Bright orange solid, 45% yield; R_f 0.55 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.24 (dd, $J = 8.7, 1.0$ Hz, 1H), 8.65 (d, $J = 1.9$ Hz, 1H), 8.36 (dd, $J = 7.3, 1.3$ Hz, 1H), 8.14 (d, $J = 8.3$ Hz, 1H), 8.08 – 8.02 (m, 2H), 7.94 (t, $J = 8.3$ Hz, 3H), 7.77 – 7.70 (m, 1H), 7.67 – 7.58 (m, 3H), 7.58 – 7.53 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 183.2, 149.9, 136.0, 135.6, 134.2, 133.6, 133.4, 131.8 (2C), 130.0, 129.6, 129.1, 129.0, 128.9, 128.2, 127.5, 127.3, 127.0, 126.3, 124.5, 115.8. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}$, m/z : 311.1179, found: 311.1182.

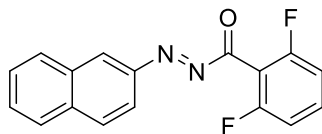
(E)-naphthalen-2-yl(naphthalen-2-yl) diazenyl methanone (1l)



1l

Bright orange solid, 39% yield; R_f 0.44 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.68 (d, $J = 10.1$ Hz, 2H), 8.18 (dd, $J = 8.6, 1.6$ Hz, 1H), 8.11 – 8.06 (m, 2H), 8.01 – 7.90 (m, 5H), 7.67 – 7.55 (m, 4H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 182.2, 150.3, 136.6, 136.3, 133.7, 133.6, 132.8, 132.5, 130.3, 130.2, 129.9, 129.7, 129.4, 129.3, 128.9, 128.5, 128.4, 127.7, 127.4, 125.6, 116.0. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}$, m/z : 311.1179, found: 311.1182.

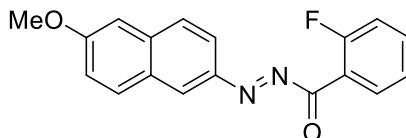
(E)-(2,6-difluorophenyl) (naphthalen-2-yl) diazenyl methanone (1m)



1m

Bright orange solid, 54% yield, R_f 0.47 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.58 (s, 1H), 8.05 – 8.00 (m, 2H), 7.93 (d, $J = 9.3$ Hz, 2H), 7.71 – 7.52 (m, 3H), 7.05 (t, $J = 8.4$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 177.7, 161.3 (2C) (d, $J = 258.2$ Hz), 149.5, 136.3, 134.4, 134.3 (t, $J = 10.6$ Hz), 133.1, 132.8, 130.1, 129.6, 129.3, 128.1, 127.3, 115.7, 112.3 (2C) (dd, $J = 21.6, 3.8$ Hz). **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{11}\text{N}_2\text{O}$, m/z : 297.0834, found: 297.0837.

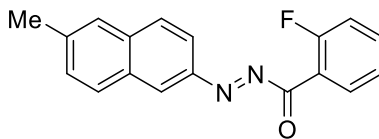
(E)-(2-fluorophenyl) ((6-methoxynaphthalen-2-yl) diazenyl) methanone (1n)



1n

Red solid, 45% yield; R_f 0.26 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.53 (d, $J = 2.0$ Hz, 1H), 8.16 – 8.11 (m, 1H), 8.00 (dd, $J = 8.9, 2.0$ Hz, 1H), 7.95 (d, $J = 8.9$ Hz, 1H), 7.82 (d, $J = 8.9$ Hz, 1H), 7.68 – 7.64 (m, 1H), 7.37 – 7.33 (m, 1H), 7.28 – 7.24 (m, 1H), 7.24 – 7.22 (m, 1H), 7.22 – 7.18 (m, 1H), 4.00 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.9$ Hz), 162.5 (d, $J = 260.9$ Hz), 160.2, 148.5, 137.8, 136.1 (d, $J = 9.0$ Hz), 132.6 (2C), 131.6, 128.4, 128.2, 126.5, 124.5 (d, $J = 3.5$ Hz), 119.9, 117.1 (d, $J = 22.2$ Hz), 116.5, 106.4, 55.5. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{14}\text{FN}_2\text{O}_2$, m/z : 309.1035, found: 309.1035.

(E)-(2-fluorophenyl) ((6-methylnaphthalen-2-yl) diazenyl) methanone (1o)

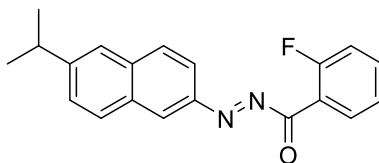


1o

Red orange solid, 57% yield; R_f 0.53 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.54 (d, $J = 1.9$ Hz, 1H), 8.19 – 8.08 (m, 1H), 8.01 – 7.90 (m, 2H), 7.82 (d, $J = 8.9$ Hz, 1H), 7.68

(s, 1H), 7.67 – 7.60 (m, 1H), 7.42 (dd, $J = 8.4, 1.7$ Hz, 1H), 7.35 – 7.29 (m, 1H), 7.17 (dd, $J = 10.0, 8.6$ Hz, 1H), 2.56 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.3$ Hz), 162.5 (d, $J = 261.0$ Hz), 149.3, 139.3 (2C), 136.2, 132.6, 131.6, 131.4, 129.7, 129.5, 128.8, 127.2, 124.6 (d, $J = 3.5$ Hz), 119.8 (d, $J = 11.4$ Hz), 117.2 (d, $J = 22.2$ Hz), 115.8, 22.0. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{14}\text{FN}_2\text{O}$, m/z : 293.1085, found: 293.1085.

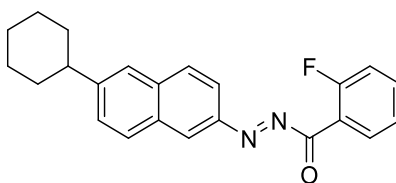
(E)-(2-fluorophenyl) ((6-isopropynaphthalen-2-yl)diazenyl) methanone (1p)



1p

Red solid, 38% yield; R_f 0.59 (*n*-hexane: ethyl acetate = 10:1). ^1H NMR (400 MHz, CDCl_3) δ 8.55 (d, $J = 1.9$ Hz, 1H), 8.17 – 8.09 (m, 1H), 7.97 (dd, $J = 8.8, 2.1$ Hz, 2H), 7.87 (d, $J = 8.9$ Hz, 1H), 7.72 (s, 1H), 7.69 – 7.60 (m, 1H), 7.50 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.33 (t, $J = 7.6$ Hz, 1H), 7.22 – 7.13 (m, 1H), 3.17 – 3.08 (m, 1H), 1.37 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.9$ Hz), 162.5 (d, $J = 261.0$ Hz), 150.1, 149.4, 136.3, 136.2 (d, $J = 9.0$ Hz), 132.6, 131.7, 131.6, 129.9, 129.2, 127.1, 124.6 (d, $J = 3.7$ Hz), 124.5, 119.8 (d, $J = 11.6$ Hz), 117.2 (d, $J = 22.2$ Hz), 115.7, 34.5, 23.8 (2C). HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{20}\text{H}_{18}\text{FN}_2\text{O}$, m/z : 321.1398, found: 321.1398.

(E)-((6-cyclohexylnaphthalen-2-yl)diazenyl) (2-fluorophenyl) methanone (1q)

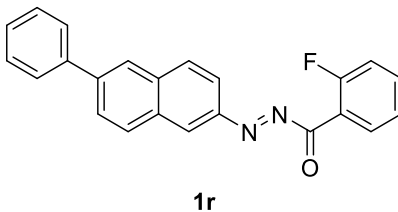


1q

Red solid, 61% yield; R_f 0.48 (*n*-hexane: ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3) δ 8.54 (d, $J = 2.0$ Hz, 1H), 8.15 – 8.10 (m, 1H), 7.99 – 7.95 (m, 2H), 7.86 (d, $J = 8.9$ Hz, 1H), 7.71 (s, 1H), 7.67 – 7.61 (m, 1H), 7.48 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.36 – 7.31 (m, 1H), 7.22 – 7.15 (m, 1H), 2.76 – 2.67 (m, 1H), 2.03 – 1.97 (m, 2H), 1.91 (d, $J = 12.8$ Hz, 2H), 1.81 (d, $J = 12.7$ Hz, 1H), 1.60 – 1.40 (m, 5H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.7$ Hz), 162.5 (d, $J = 261.2$ Hz), 149.4, 149.3, 136.3, 136.2 (d, $J = 9.0$ Hz), 132.6, 131.7, 131.6, 129.8, 129.2, 127.6, 124.9,

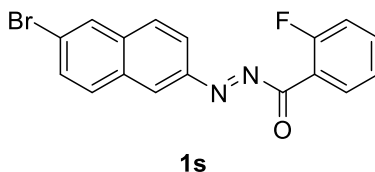
124.5 (d, $J = 3.7$ Hz), 119.8 (d, $J = 11.3$ Hz), 117.2 (d, $J = 22.2$ Hz), 115.6, 44.9, 34.3 (2C), 26.9 (2C), 26.1. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{23}H_{22}FN_2O$, m/z : 361.1711, found: 361.1712.

(E)-(2-fluorophenyl)((6-phenylnaphthalen-2-yl)diazenyl)methanone (1r)



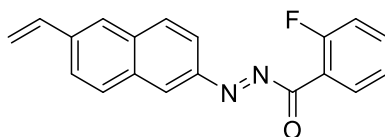
Red solid, 49% yield; R_f 0.45 (*n*-hexane: ethyl acetate = 10:1). **1H NMR (500 MHz, $CDCl_3$)** δ 8.63 (d, $J = 1.9$ Hz, 1H), 8.18 – 8.10 (m, 3H), 8.08 – 8.02 (m, 1H), 8.00 (d, $J = 8.9$ Hz, 1H), 7.88 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.78 (d, $J = 7.0$ Hz, 2H), 7.70 – 7.65 (m, 1H), 7.55 (t, $J = 7.7$ Hz, 2H), 7.45 (t, $J = 7.4$ Hz, 1H), 7.37 (t, $J = 7.6$ Hz, 1H), 7.21 (dd, $J = 10.4, 8.5$ Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 180.8 (d, $J = 5.8$ Hz), 162.5 (d, $J = 261.0$ Hz), 149.8, 141.7, 140.4, 136.3 (d, $J = 9.2$ Hz), 132.6, 132.3, 131.3, 130.5, 129.8, 129.1 (2C), 128.9, 128.1, 127.5 (2C), 126.9, 125.9, 124.6 (d, $J = 3.5$ Hz), 119.7 (d, $J = 11.1$ Hz), 117.2 (d, $J = 22.2$ Hz), 116.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{23}H_{16}FN_2O$, m/z : 355.1241, found: 355.1244.

(E)-((6-bromonaphthalen-2-yl)diazenyl)(2-fluorophenyl) methanone (1s)



Red solid, 53% yield, R_f 0.54 (*n*-hexane: ethyl acetate = 10:1). **1H NMR (400 MHz, $CDCl_3$)** δ 8.45 (d, $J = 1.9$ Hz, 1H), 8.08 – 8.01 (m, 1H), 8.00 (d, $J = 1.9$ Hz, 1H), 7.95 – 7.90 (m, 1H), 7.82 (d, $J = 8.7$ Hz, 1H), 7.76 (d, $J = 8.9$ Hz, 1H), 7.61 – 7.55 (m, 2H), 7.30 – 7.23 (m, 1H), 7.13 – 7.06 (m, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 180.8 (d, $J = 5.6$ Hz), 162.5 (d, $J = 260.9$ Hz), 149.8, 136.7, 136.4 (d, $J = 9.1$ Hz), 132.6, 131.6, 131.3, 130.8, 130.7, 130.3, 128.6, 124.7 (d, $J = 3.6$ Hz), 123.3, 119.4 (d, $J = 10.7$ Hz), 117.2 (d, $J = 22.6$ Hz), 117.1. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{17}H_{11}BrFN_2O$, m/z : 357.0033, found: 357.0033.

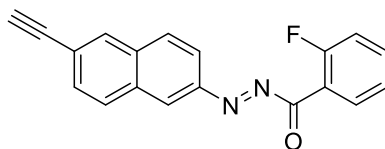
(E)-(2-fluorophenyl)((6-vinylnaphthalen-2-yl)diazenyl) methanone (1t)



1t

Red solid, 38% yield; R_f 0.50 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.53 (d, $J = 2.0$ Hz, 1H), 8.16 – 8.09 (m, 1H), 8.02 – 7.96 (m, 2H), 7.89 (d, $J = 8.9$ Hz, 1H), 7.82 (d, $J = 1.6$ Hz, 1H), 7.73 (dd, $J = 8.5, 1.7$ Hz, 1H), 7.65 (d, $J = 8.4$ Hz, 1H), 7.38 – 7.31 (m, 1H), 7.21 – 7.15 (m, 1H), 6.91 (dd, $J = 17.6, 10.9$ Hz, 1H), 5.97 (d, $J = 17.6$ Hz, 1H), 5.45 (d, $J = 10.9$ Hz, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.8$ Hz), 162.5 (d, $J = 261.1$ Hz), 149.8, 138.0, 136.5, 136.3 (d, $J = 9.1$ Hz), 136.2, 132.8, 132.6, 131.1, 130.3, 129.6, 126.3, 124.6 (2C), 119.7 (d, $J = 11.1$ Hz), 117.2 (d, $J = 22.3$ Hz), 116.3, 116.1. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{19}\text{H}_{14}\text{FN}_2\text{O}$, m/z : 305.1085, found: 305.1090.

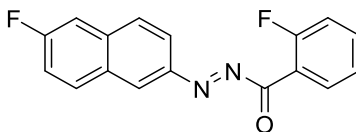
(E)-((6-ethylnaphthalen-2-yl)diazenyl)(2-fluorophenyl) methanone (1u)



1u

Red solid, 65% yield; R_f 0.46 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.56 (d, $J = 2.0$ Hz, 1H), 8.18 – 8.11 (m, 1H), 8.09 (d, $J = 1.4$ Hz, 1H), 8.05 – 7.97 (m, 2H), 7.90 (d, $J = 8.9$ Hz, 1H), 7.71 – 7.62 (m, 2H), 7.38 – 7.32 (m, 1H), 7.20 (dd, $J = 10.0, 8.6$ Hz, 1H), 3.28 (s, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.8 (d, $J = 6.0$ Hz), 162.5 (d, $J = 260.9$ Hz), 150.2, 136.4 (d, $J = 8.9$ Hz), 135.3, 132.8 (2C), 132.6, 132.2, 130.7, 129.9 (d, $J = 2.6$ Hz), 129.4, 124.7 (d, $J = 3.6$ Hz), 122.5, 119.4 (d, $J = 11.2$ Hz), 117.2 (d, $J = 22.2$ Hz), 116.8, 83.5, 79.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{19}\text{H}_{12}\text{FN}_2\text{O}$, m/z : 303.0928, found: 303.0929.

(E)-((6-fluoronaphthalen-2-yl)diazenyl)(2-fluorophenyl) methanone (1v)

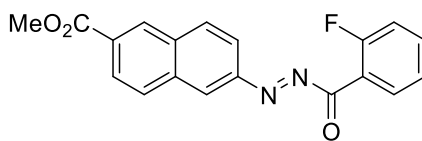


1v

Bright orange solid, 54% yield; R_f 0.50 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.56 (d, $J = 1.9$ Hz, 1H), 8.16 – 8.10 (m, 1H), 8.06 – 8.00 (m, 2H), 7.86 (d, $J = 8.9$ Hz,

1H), 7.69 – 7.62 (m, 1H), 7.53 (dd, $J = 9.6, 2.5$ Hz, 1H), 7.39 – 7.31 (m, 2H), 7.20 – 7.15 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.8$ Hz), 162.5 (d, $J = 261.0$ Hz), 162.4 (d, $J = 251.2$ Hz), 149.4 (d, $J = 2.5$ Hz), 137.0 (d, $J = 9.6$ Hz), 136.4 (d, $J = 9.1$ Hz), 132.6, 132.5 (d, $J = 9.4$ Hz), 131.1, 130.1, 128.8 (d, $J = 5.3$ Hz), 124.6 (d, $J = 3.7$ Hz), 119.5 (d, $J = 11.3$ Hz), 117.6 (d, $J = 25.3$ Hz), 117.2 (d, $J = 22.2$ Hz), 117.0, 111.7 (d, $J = 20.9$ Hz). HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{11}\text{F}_2\text{N}_2\text{O}$, m/z : 297.0834, found: 297.0837.

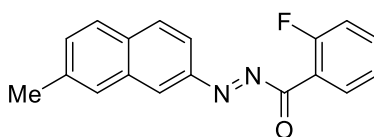
Methyl (*E*)-6-((2-fluorobenzoyl)diazenyl)-2-naphthoate (1w)



1w

Red solid, 63% yield; R_f 0.29 (*n*-hexane: ethyl acetate = 10:1). ^1H NMR (500 MHz, CDCl_3) δ 8.67 (s, 1H), 8.60 (d, $J = 1.5$ Hz, 1H), 8.20 – 8.12 (m, 2H), 8.11 – 8.07 (m, 1H), 8.07 – 8.04 (m, 2H), 7.72 – 7.59 (m, 1H), 7.41 – 7.33 (m, 1H), 7.23 – 7.15 (m, 1H), 4.03 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.8 (d, $J = 6.0$ Hz), 166.7, 162.6 (d, $J = 261.1$ Hz), 150.9, 136.5 (d, $J = 9.1$ Hz), 135.4, 134.9, 132.8, 132.5, 130.9 (d, $J = 14.6$ Hz), 130.2, 130.0, 129.9, 126.6, 124.7 (d, $J = 3.6$ Hz), 119.3 (d, $J = 11.3$ Hz), 117.2 (d, $J = 22.2$ Hz), 116.8, 52.5. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{19}\text{H}_{14}\text{FN}_2\text{O}_3$, m/z : 337.0983, found: 337.0985.

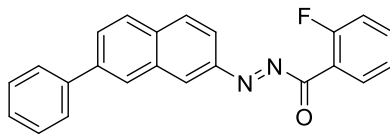
(*E*)-(2-fluorophenyl)((7-methylnaphthalen-2-yl)diazenyl) methanone (1x)



1x

Red solid, 59% yield, R_f 0.55 (*n*-hexane: ethyl acetate = 10:1). ^1H NMR (400 MHz, CDCl_3) δ 8.50 (d, $J = 1.9$ Hz, 1H), 8.15 – 8.09 (m, 1H), 7.92 (dd, $J = 8.9, 1.9$ Hz, 1H), 7.87 (d, $J = 8.9$ Hz, 1H), 7.81 (d, $J = 8.4$ Hz, 2H), 7.67 – 7.60 (m, 1H), 7.46 (dd, $J = 8.3, 1.8$ Hz, 1H), 7.36 – 7.30 (m, 1H), 7.21 – 7.15 (m, 1H), 2.55 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 180.9 (d, $J = 5.7$ Hz), 162.5 (d, $J = 261.1$ Hz), 149.9, 137.1, 136.3 (d, $J = 8.9$ Hz), 134.2, 133.4, 132.6, 131.2, 131.1, 129.2, 128.9, 127.9, 124.6 (d, $J = 3.6$ Hz), 119.7 (d, $J = 10.9$ Hz), 117.2 (d, $J = 22.2$ Hz), 114.8, 21.7. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{14}\text{FN}_2\text{O}$, m/z : 293.1086, found: 293.1085.

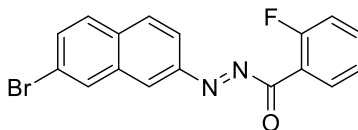
(E)-(2-fluorophenyl)((7-phenylnaphthalen-2-yl)diazenyl) methanone (1y)



1y

Red solid, 51% yield; R_f 0.50 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.55 (d, $J = 1.8$ Hz, 1H), 8.13 (d, $J = 1.8$ Hz, 1H), 8.07 – 8.03 (m, 1H), 7.92 – 7.84 (m, 3H), 7.81 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.68 – 7.64 (m, 2H), 7.59 – 7.54 (m, 1H), 7.46 – 7.40 (m, 2H), 7.36 – 7.31 (m, 1H), 7.28 – 7.23 (m, 1H), 7.12 – 7.07 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.9 (d, $J = 5.8$ Hz), 162.6 (d, $J = 261.0$ Hz), 150.1, 140.3, 140.0, 136.4 (d, $J = 9.0$ Hz), 135.0, 133.5, 132.6, 132.1, 131.7, 129.8, 129.6 (d, $J = 15.7$ Hz), 129.3, 129.0, 128.6, 127.9, 127.6, 127.5, 124.6 (d, $J = 3.5$ Hz), 117.2 (d, $J = 22.2$ Hz), 116.4, 115.8. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{23}\text{H}_{16}\text{FN}_2\text{O}$, m/z : 355.1241, found: 355.1241.

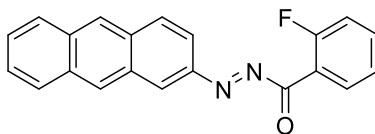
(E)-((7-bromonaphthalen-2-yl)diazenyl)(2-fluorophenyl) methanone (1z)



1z

Red solid, 57% yield, R_f 0.58 (*n*-hexane: ethyl acetate = 10:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.61 (d, $J = 1.9$ Hz, 1H), 8.20 – 8.10 (m, 2H), 8.07 – 8.01 (m, 1H), 7.93 (d, $J = 8.8$ Hz, 1H), 7.87 (d, $J = 8.9$ Hz, 1H), 7.75 – 7.66 (m, 2H), 7.60 – 7.52 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 180.8 (d, $J = 5.9$ Hz), 162.6 (d, $J = 261.1$ Hz), 150.2, 136.5 (d, $J = 9.1$ Hz), 134.3, 134.2, 132.5, 132.1, 131.7, 129.8, 129.7, 129.5, 124.7 (d, $J = 3.6$ Hz), 121.3, 119.3, 117.2 (d, $J = 22.2$ Hz), 116.4. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{11}\text{BrFN}_2\text{O}$, m/z : 357.0033, found: 357.0032.

(E)-(anthracen-2-yl)diazenyl (2-fluorophenyl)methanone (1ad)

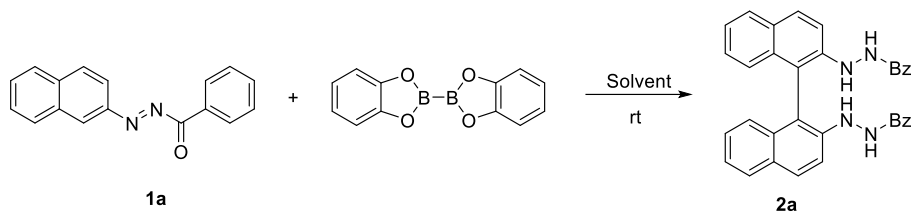


1ad

Orange solid, 29%; R_f 0.55 (*n*-hexane: ethyl acetate = 10:1). **^1H NMR (500 MHz, CDCl_3)** δ 8.80 (s, 1H), 8.65 (s, 1H), 8.47 (s, 1H), 8.17 – 8.12 (m, 1H), 8.10 – 8.03 (m, 3H), 8.00 – 7.94 (m, 1H), 7.65 (dd, $J = 13.4, 7.2$ Hz, 1H), 7.61 – 7.52 (m, 2H), 7.35 (t, $J = 7.7$ Hz, 1H), 7.23 – 7.16 (m, 1H). **^{13}C NMR (126 MHz, CDCl_3)** δ 180.8 (d, $J = 5.8$ Hz), 162.5 (d, $J = 261.0$ Hz), 149.6, 136.2 (d, $J = 9.1$ Hz), 135.3, 133.5, 132.9, 132.6, 132.3, 130.9, 130.1, 129.7, 128.5 (d, $J = 28.9$ Hz), 128.2, 127.1, 126.8, 126.4, 125.4, 124.6 (d, $J = 3.7$ Hz), 117.2 (d, $J = 22.3$ Hz), 114.7. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{20}\text{H}_{18}\text{FN}_2\text{O}$, m/z : 321.1398, found: 321.1398.

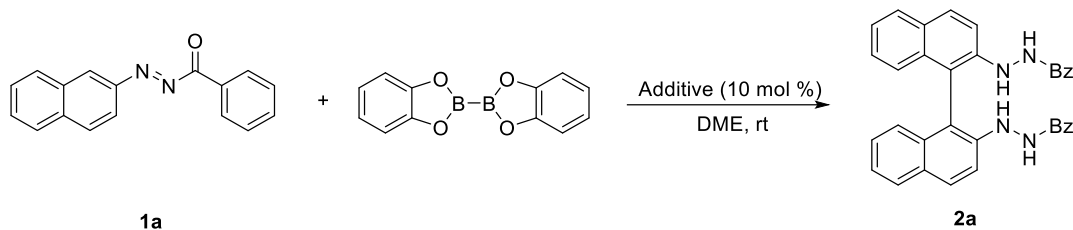
Reaction development and optimization

Supplementary Table 1: Optimization of reaction conditions in terms of solvent^a



entry	Solvent	Yield (%) ^b
1	1, 2-dimethoxyethane	51
2	1, 4 dioxane	32
3	MTBE	44
4	THF	40
5	Dichloromethane	33
6	Methanol	0
7	Toluene	16
8	DMF	12
9	DMSO	9
10	Acetone	<i>n.d</i>
11	Ethyl acetate	25
12	Acetonitrile	50
13^c	1, 2-dimethoxyethane	73

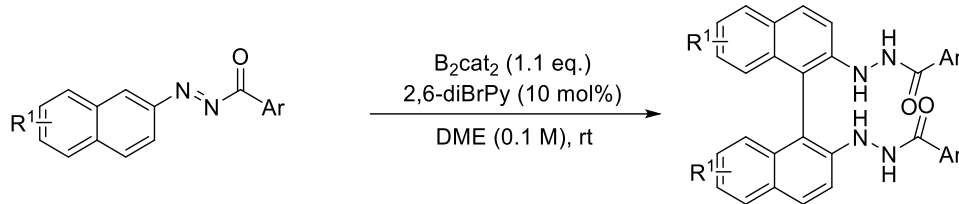
^aUnless otherwise indicated, reactions were performed using **1a** azonaphthalene (0.1 mmol), and diboron (0.055 mmol) in 1.0 mL solvent at room temperature for 2 h. ^b Yields were determined by crude ¹H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. ^c 0.11 mmol of B₂Cat₂ was used. *n.d* not detected.

Supplementary Table 2: Optimization of reaction conditions in terms of additives

entry	Additive	Yield (%) ^a
1	-	73%
2	CS_2CO_3	81%
3	PPh_3	67%
4	Triethylamine	68%
5	DABCO	63%
6	DBU	76%
7	DMAP	81%
8	2,6-lutidine	63%
9	2,6-dibromopyridine	89%
10	2,4,6-tribromopyridine	72%
11	4-iodopyridine	74%
12	3-bromopyridine	71%
13	2-cyanopyridine	66%
14	4-methoxy pyridine	63%
15	4-benzyl pyridine	58%
16	4-acetylpyridine	67%

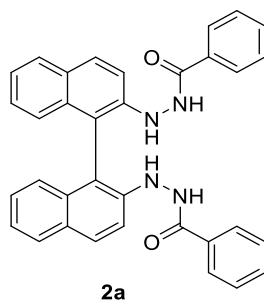
^aUnless otherwise indicated, reactions were performed using **1a** azonaphthalene (0.1 mmol), B_2cat_2 (0.11 mmol) and additive (0.01 mmol) in 1.0 mL DME at room temperature for 2 h. ^b Yields were determined by crude 1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard.

Synthesis of BINAM derivatives via [5,5]-sigmatropic rearrangements



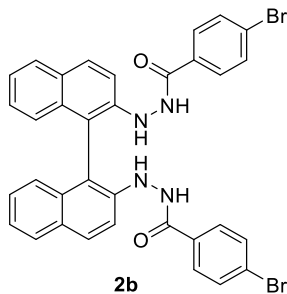
A dry 4 mL vial equipped with a Teflon-coated magnetic stirring bar was charged with azonaphthalene (0.2 mmol), B₂cat₂ (0.22 mmol) and 2,6-dibromopyridine (0.02 mmol). Anhydrous 1,2-dimethoxyethane (2.0 mL) was added and stirred at room temperature. Upon reaction completion, the mixture was concentrated under reduced pressure to give a residue which was then purified by washing with warm methanol to give the desired product.

N',N'''-([1,1'-binaphthalene]-2,2'-diyl)di(benzohydrazide) (**2a**)



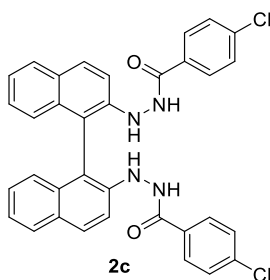
Following the general protocol, compound **2a** was obtained in 89% yield as a white solid. R_f 0.39 (*n*-hexane: ethyl acetate = 2.5:1). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.66 (s, 2H), 8.00 – 7.75 (m, 8H), 7.60 – 7.42 (m, 10H), 7.28 (t, *J* = 7.4 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 2H), 6.80 (d, *J* = 8.5 Hz, 2H). ¹³C NMR (126 MHz, DMSO-*d*₆) δ 168.0, 145.1, 134.0, 133.2, 132.3, 130.1, 129.2, 128.9 (2C), 128.6, 127.7 (2C), 127.0, 124.5, 123.3, 115.5, 112.2. HRMS (ESI) calcd for [M+H]⁺ C₃₄H₂₇N₄O₂ *m/z* 523.2129, found 523.2130.

N',N'''-([1,1'-binaphthalene]-2,2'-diyl)bis(4-bromobenzohydrazide) (**2b**)



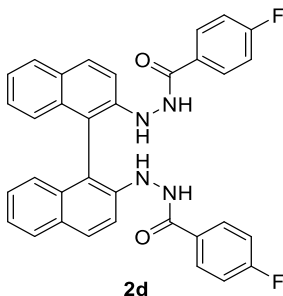
Following the general protocol, compound **2b** was obtained in 76% yield as a white solid. R_f 0.57 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.71 (d, $J = 2.0$ Hz, 2H), 7.94 (d, $J = 9.1$ Hz, 2H), 7.87 (d, $J = 6.8$ Hz, 2H), 7.78 (d, $J = 8.6$ Hz, 4H), 7.54 (d, $J = 8.6$ Hz, 4H), 7.48 (s, 2H), 7.41 (d, $J = 9.0$ Hz, 2H), 7.31 – 7.24 (m, 2H), 7.21-7.15 (m, 2H), 6.77 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 167.0, 144.8, 137.1, 134.0, 131.9, 130.2, 129.6 (2C), 129.2, 129.1 (2C), 128.6, 127.0, 124.5, 123.4, 115.4, 112.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{25}\text{Br}_2\text{N}_4\text{O}_2$, m/z : 679.0339, found: 679.0338.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(4-chlorobenzohydrazide) (2c)**



Following the general protocol, compound **2c** was obtained in 84% yield as a white solid. R_f 0.54 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.71 (d, $J = 2.0$ Hz, 2H), 7.94 (d, $J = 9.1$ Hz, 2H), 7.87 (d, $J = 6.8$ Hz, 2H), 7.78 (d, $J = 8.6$ Hz, 4H), 7.54 (d, $J = 8.6$ Hz, 4H), 7.48 (s, 2H), 7.41 (d, $J = 9.0$ Hz, 2H), 7.31 – 7.24 (m, 2H), 7.21-7.15 (m, 2H), 6.77 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 167.0, 144.8, 137.1, 134.0, 131.9, 130.2, 129.6 (2C), 129.2, 129.1 (2C), 128.6, 127.0, 124.5, 123.4, 115.4, 112.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{25}\text{Cl}_2\text{N}_4\text{O}_2$, m/z : 591.1349, found: 591.1347.

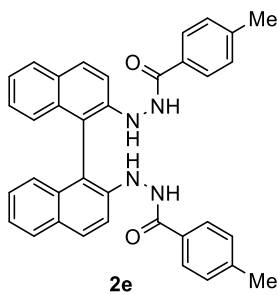
***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(4-fluorobenzohydrazide) (2d)**



Following the general protocol, compound **2d** was obtained in 79% yield as a white solid. R_f 0.53 (*n*-hexane: ethyl acetate = 2.5:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.67 (s, 2H), 7.94 (d, $J = 9.0$ Hz, 2H), 7.89 – 7.80 (m, 6H), 7.51 (s, 2H), 7.42 (d, $J = 9.0$ Hz, 2H), 7.34 – 7.23 (m, 6H), 7.20

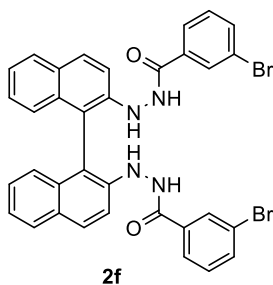
– 7.15 (m, 2H), 6.77 (d, $J = 8.5$ Hz, 2H). ^{13}C NMR (126 MHz, DMSO) δ 166.9, 164.6 (d, $J = 249.1$ Hz), 144.9, 134.0, 130.4 (2C) (d, $J = 9.1$ Hz), 130.1, 129.6 (d, $J = 3.0$ Hz), 129.2, 128.6, 127.0, 124.5, 123.3, 116.0 (2C) (d, $J = 22.0$ Hz), 115.4, 112.2. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{25}\text{F}_2\text{N}_4\text{O}_2$, m/z : 559.1940, found: 559.1946.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(4-methylbenzohydrazide) (2e)**



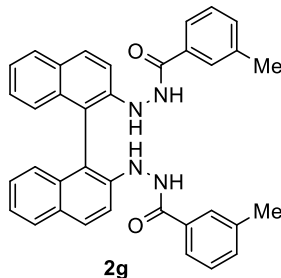
Following the general protocol, compound **2e** was obtained in 89% yield as a white solid. R_f 0.34 (*n*-hexane: ethyl acetate = 3:1). ^1H NMR (500 MHz, DMSO- d_6) δ 10.55 (d, $J = 2.1$ Hz, 2H), 7.94 (d, $J = 9.1$ Hz, 2H), 7.86 (d, $J = 8.6$ Hz, 2H), 7.67 (d, $J = 7.9$ Hz, 4H), 7.55 (s, 2H), 7.40 (d, $J = 9.1$ Hz, 2H), 7.31 – 7.22 (m, 6H), 7.17 (t, $J = 7.7$ Hz, 2H), 6.77 (d, $J = 8.5$ Hz, 2H), 2.34 (s, 6H). ^{13}C NMR (126 MHz, DMSO- d_6) δ 167.8, 145.2, 142.2, 134.0, 130.4, 130.0, 129.4 (2C), 129.2, 128.5, 127.7 (2C), 126.9, 124.5, 123.3, 115.4, 112.2, 21.5. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{31}\text{N}_4\text{O}_2$, m/z : 551.2442, found: 551.2438.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(3-bromobenzohydrazide) (2f)**



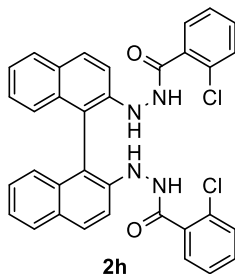
Following the general protocol, compound **2f** was obtained in 69% yield as a white solid. R_f 0.57 (*n*-hexane: ethyl acetate = 2.5:1). ^1H NMR (400 MHz, DMSO- d_6) δ 10.72 (s, 2H), 7.95 (d, $J = 9.0$ Hz, 2H), 7.90 – 7.84 (m, 4H), 7.80 – 7.72 (m, 4H), 7.47 – 7.40 (m, 6H), 7.28 (t, $J = 6.8$ Hz, 2H), 7.19 (t, $J = 6.9$ Hz, 2H), 6.77 (d, $J = 8.4$ Hz, 2H). ^{13}C NMR (126 MHz, DMSO- d_6) δ 166.7, 144.7, 135.4, 135.0, 134.0, 131.3, 130.3, 130.2, 129.3, 128.6, 127.0, 126.7, 124.5, 123.4, 122.3, 115.5, 112.2. HRMS (ESI) calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{25}\text{Br}_2\text{N}_4\text{O}_2$, m/z : 679.0339, found: 679.0337.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(3-methylbenzohydrazide) (2g)**



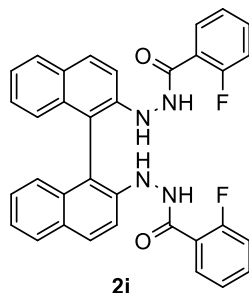
Following the general protocol, compound **2g** was obtained in 83% yield as a white solid. R_f 0.43 (*n*-hexane: ethyl acetate = 3:1). **¹H NMR (400 MHz, DMSO-*d*₆)** δ 10.57 (s, 2H), 7.91 (dd, J = 30.9, 8.7 Hz, 4H), 7.55 (s, 6H), 7.45 – 7.09 (m, 10H), 6.78 (d, J = 8.5 Hz, 2H), 2.31 (s, 6H). **¹³C NMR (126 MHz, DMSO-*d*₆)** δ 168.2, 145.1, 138.2, 134.0, 133.3, 132.8, 130.1, 129.2, 128.8, 128.6, 128.2, 127.0, 124.7, 124.5, 123.3, 115.5, 112.1, 21.3. **HRMS (ESI)** calcd for $[M+H]^+$ C₃₆H₃₁N₄O₂ m/z : 551.2442, found: 551.2441.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(2-chlorobenzohydrazide) (2h)**



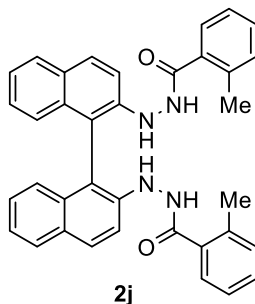
Following the general protocol, compound **2h** was obtained in 72% yield as a white solid. R_f 0.29 (*n*-hexane: ethyl acetate = 2.5:1). **¹H NMR (400 MHz, DMSO-*d*₆)** δ 10.56 (d, J = 2.1 Hz, 2H), 8.03 (d, J = 9.1 Hz, 2H), 7.91 (d, J = 7.5 Hz, 2H), 7.59 (d, J = 9.0 Hz, 2H), 7.55 (d, J = 8.0 Hz, 2H), 7.51 – 7.45 (m, 2H), 7.39 (d, J = 3.8 Hz, 6H), 7.29 (t, J = 7.1 Hz, 2H), 7.19 (t, J = 6.9 Hz, 2H), 6.77 (d, 2H). **¹³C NMR (126 MHz, DMSO-*d*₆)** δ 167.7, 144.6, 134.9, 133.9, 132.0, 130.9, 130.4, 130.1, 129.7, 129.3, 128.6, 127.6, 127.1, 124.2, 123.5, 115.7, 112.2. **HRMS (ESI)** calcd for $[M+H]^+$ C₃₄H₂₅Cl₂N₄O₂, m/z : 591.1349, found: 591.1348.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2i)**



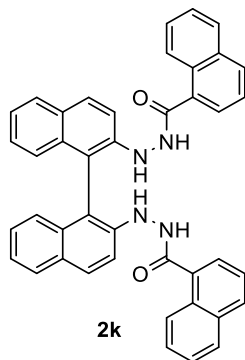
Following the general protocol, compound **2i** was obtained in 87% yield as a white solid. R_f 0.33 (*n*-hexane: ethyl acetate = 2.5:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.52 (s, 2H), 8.01 (d, $J = 9.0$ Hz, 2H), 7.90 (d, $J = 8.0$ Hz, 2H), 7.57 (t, $J = 7.3$ Hz, 4H), 7.50 (d, $J = 8.9$ Hz, 4H), 7.39 – 7.26 (m, 6H), 7.22 – 7.17 (m, 2H), 6.77 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.5, 159.7 (d, $J = 250.2$ Hz), 144.7, 134.0, 133.5 (d, $J = 8.3$ Hz), 130.4 (d, $J = 2.8$ Hz), 130.2, 129.3, 128.6, 125.1 (d, $J = 3.2$ Hz), 125.1, 124.5, 123.4, 122.7 (d, $J = 15.0$ Hz), 116.8 (d, $J = 21.7$ Hz), 115.4, 112.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{25}\text{F}_2\text{N}_4\text{O}_2$, m/z : 559.1940, found: 559.1939.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(2-methylbenzohydrazide) (**2j**)**



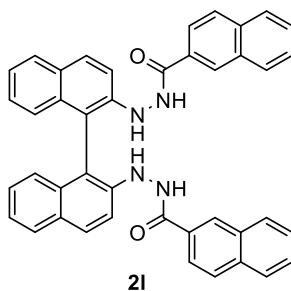
Following the general protocol, compound **2j** was obtained in 65% yield as a white solid. R_f 0.49 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.39 (d, $J = 2.2$ Hz, 2H), 8.02 (d, $J = 9.1$ Hz, 2H), 7.90 (d, $J = 6.9$ Hz, 2H), 7.56 (s, 2H), 7.49 (d, $J = 8.9$ Hz, 2H), 7.41 (d, $J = 7.5$ Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 2H), 7.31 – 7.22 (m, 6H), 7.18 (t, $J = 7.3$ Hz, 2H), 6.75 (d, $J = 8.0$ Hz, 2H), 2.24 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 170.4, 145.0, 136.5, 134.8, 134.0, 131.1, 130.5, 130.1, 129.2, 128.6, 127.9, 127.0, 126.0, 124.5, 123.4, 115.4, 112.2, 19.8. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{31}\text{N}_4\text{O}_2$, m/z : 551.2442, found: 551.2444.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(1-naphthohydrazide) (**2k**)**



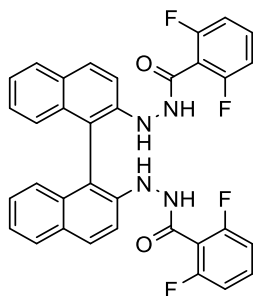
Following the general protocol, compound **2k** was obtained in 73% yield as a white solid. R_f 0.46 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.80 (s, 2H), 8.16 – 8.06 (m, 6H), 7.96 (d, $J = 8.1$ Hz, 4H), 7.89 – 7.80 (m, 4H), 7.64 (d, $J = 8.6$ Hz, 4H), 7.46 (t, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.5$ Hz, 2H), 7.23 (t, $J = 7.7$ Hz, 2H), 7.05 (t, $J = 7.7$ Hz, 2H), 6.82 (d, $J = 8.3$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 170.2, 145.0, 134.1, 133.6, 132.3, 131.2, 130.9, 130.4, 130.3, 129.3, 128.7, 127.1 (2C), 126.8, 126.5, 125.7, 125.4, 124.5, 123.5, 115.3, 112.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{42}\text{H}_{31}\text{N}_4\text{O}_2$ m/z : 623.2440, found: 623.2442.

***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(2-naphthohydrazide) (**2l**)**



Following the general protocol, compound **2l** was obtained in 82% yield as a white solid. R_f 0.40 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.83 (s, 2H), 8.43 (d, $J = 4.8$ Hz, 2H), 7.96 (dd, $J = 10.8, 4.1$ Hz, 8H), 7.89 (d, $J = 8.1$ Hz, 2H), 7.81 (d, $J = 6.7$ Hz, 2H), 7.67 (d, $J = 6.4$ Hz, 2H), 7.64 – 7.50 (m, 6H), 7.29 (t, $J = 7.5$ Hz, 2H), 7.24 – 7.18 (m, 2H), 6.83 (t, $J = 6.7$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 168.2, 145.1, 134.8, 134.1, 132.5, 130.6, 130.1, 129.3 (2C), 128.6 (2C), 128.3, 128.2, 128.1, 127.4, 127.0, 124.6, 124.3, 123.3, 115.6, 112.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{42}\text{H}_{31}\text{N}_4\text{O}_2$, m/z : 623.2442, found: 623.2445.

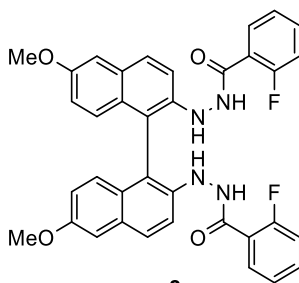
***N',N'''*-([1,1'-binaphthalene]-2,2'-diyl)bis(2,6-difluorobenzohydrazide) (**2m**)**



2m

Following the general protocol, compound **2m** was obtained in 89% yield as a white solid. R_f 0.37 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.76 (d, $J = 2.3$ Hz, 2H), 8.04 (d, $J = 9.1$ Hz, 2H), 7.91 (d, $J = 8.0$ Hz, 2H), 7.61 – 7.53 (m, 2H), 7.49 (d, $J = 9.0$ Hz, 2H), 7.29 (t, $J = 6.6$ Hz, 4H), 7.25 – 7.16 (m, 6H), 6.77 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 161.1, 159.8 (d, $J = 247.4$ Hz), 159.8 (d, $J = 250.8$ Hz), 144.4, 133.9, 133.0, 130.2, 129.3, 128.6, 127.1, 124.5, 123.6, 115.3, 113.5 (t, $J = 22.2$ Hz), 112.6 (d, $J = 4.3$ Hz), 112.40 (2C). **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{23}\text{F}_4\text{N}_4\text{O}_2$, m/z 595.1752, found: 595.1758.

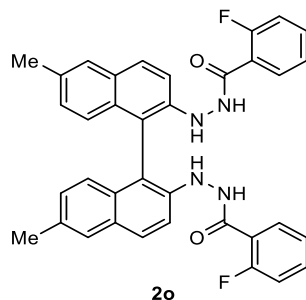
***N',N''*-(6,6'-dimethoxy-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2n)**



2n

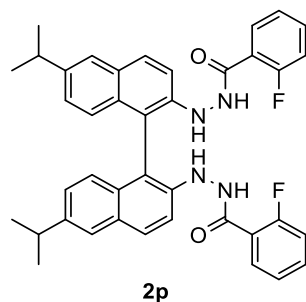
Following the general protocol, compound **2n** was obtained in 87% yield as a white solid. R_f 0.21 (*n*-hexane: ethyl acetate = 2.5:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.46 (s, 2H), 7.91 (s, 2H), 7.44 (d, $J = 95.7$ Hz, 14H), 6.89 (s, 2H), 6.68 (s, 2H), 3.83 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.7 (d, $J = 250.1$ Hz), 155.8, 142.8, 133.4 (d, $J = 8.4$ Hz), 130.4 (2C), 130.2, 129.2, 129.1 (d, $J = 24.1$ Hz), 125.0 (d, $J = 3.2$ Hz), 122.8 (d, $J = 15.1$ Hz), 119.3, 116.7 (d, $J = 21.9$ Hz), 115.9, 112.9, 107.2, 55.6. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_4$ m/z : 619.2151, found: 619.2154.

***N',N'''*-(6,6'-dimethyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2o)**



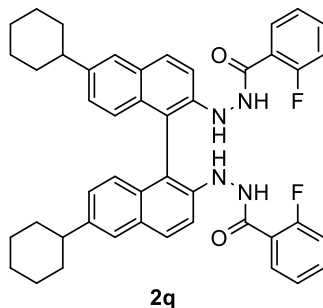
Following the general protocol, compound **2o** was obtained in 83% yield as a white solid. R_f 0.34 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.48 (s, 2H), 7.90 (d, $J = 9.0$ Hz, 2H), 7.65 (s, 2H), 7.55 (t, $J = 6.8$ Hz, 4H), 7.47 – 7.40 (m, 4H), 7.36 – 7.25 (m, 4H), 7.03 (dd, $J = 8.8, 1.8$ Hz, 2H), 6.67 (d, $J = 8.7$ Hz, 2H), 2.38 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.7 (d, $J = 250.1$ Hz), 143.9, 133.5 (d, $J = 8.3$ Hz), 132.4, 132.2, 130.4, 129.4 (2C), 129.1, 127.5, 125.0 (d, $J = 2.6$ Hz), 124.6, 122.8 (d, $J = 15.0$ Hz), 116.8 (d, $J = 21.7$ Hz), 115.4, 112.42, 21.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_2$, m/z : 587.2253, found: 587.2256.

***N',N'''*-(6,6'-diisopropyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2p)**



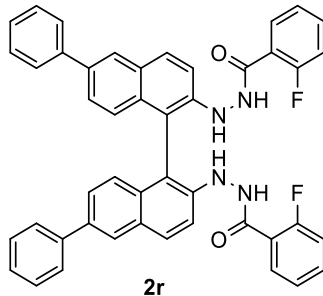
Following the general protocol, compound **2p** was obtained in 85% yield as a white solid. R_f 0.37 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.46 (s, 2H), 7.93 (d, $J = 9.1$ Hz, 2H), 7.69 (d, $J = 1.9$ Hz, 2H), 7.61 – 7.52 (m, 4H), 7.44 (d, $J = 9.0$ Hz, 2H), 7.40 – 7.38 (m, 2H), 7.37 – 7.23 (m, 4H), 7.13 (dd, $J = 8.8, 1.9$ Hz, 2H), 6.72 (d, $J = 8.7$ Hz, 2H), 2.97 (p, $J = 6.8$ Hz, 2H), 1.25 (dd, $J = 6.9, 3.4$ Hz, 12H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.7 (d, $J = 249.9$ Hz), 144.0, 143.3, 133.4 (d, $J = 8.4$ Hz), 132.6, 130.3 (d, $J = 2.6$ Hz), 129.8, 129.4, 126.8, 125.1, 125.0, 124.7, 122.8 (d, $J = 15.0$ Hz), 116.7 (d, $J = 21.8$ Hz), 115.3, 112.4, 33.6, 24.4, 24.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{40}\text{H}_{37}\text{F}_2\text{N}_4\text{O}_2$, m/z : 643.2879, found: 643.2880.

***N',N'''*-(6,6'-dicyclohexyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2q)**



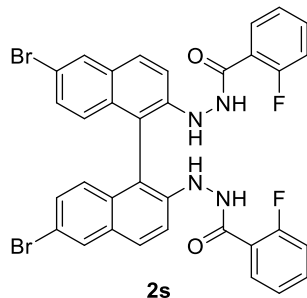
Following the general protocol, compound **2q** was obtained in 87% yield as a white solid. R_f 0.40 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.65 (d, $J = 10.8$ Hz, 2H), 7.77 (d, $J = 8.8$ Hz, 4H), 7.53 (s, 2H), 7.36 (d, $J = 9.0$ Hz, 4H), 7.12 – 6.96 (m, 8H), 2.58 – 2.46 (m, 2H), 1.84 (d, $J = 11.4$ Hz, 4H), 1.76 (d, $J = 8.6$ Hz, 4H), 1.66 (d, $J = 12.2$ Hz, 2H), 1.44 – 1.26 (m, 8H), 1.24 – 1.15 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 164.4 (d, $J = 3.7$ Hz), 160.6 (d, $J = 248.7$ Hz), 143.4, 143.0, 133.7 (d, $J = 9.0$ Hz), 132.5, 131.9 (d, $J = 2.6$ Hz), 123.0, 129.8, 127.5, 124.8 (2C), 124.7, 119.8 (d, $J = 13.3$ Hz), 116.1 (d, $J = 24.0$ Hz), 115.5, 113.9, 44.3, 34.5, 34.3, 27.0 (2C), 26.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{46}\text{H}_{45}\text{F}_2\text{N}_4\text{O}_2$, m/z : 723.3505, found: 723.3507.

***N',N'''*-(6,6'-diphenyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2r)**



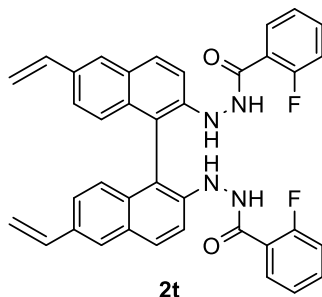
Following the general protocol, compound **2r** was obtained in 75% yield as a white solid. R_f 0.23 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.56 (s, 2H), 8.22 (s, 2H), 8.12 (d, $J = 9.1$ Hz, 2H), 7.74 (d, $J = 7.7$ Hz, 4H), 7.61 – 7.51 (m, 10H), 7.47 (t, $J = 7.6$ Hz, 4H), 7.39 – 7.28 (m, 6H), 6.92 (d, $J = 8.8$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.5, 159.8 (d, $J = 250.3$ Hz), 145.0, 140.5, 135.1, 133.5 (d, $J = 8.6$ Hz), 133.3, 130.8, 130.4 (2C), 129.6, 129.4 (2C), 127.6, 127.1 (2C), 126.2 (d, $J = 12.5$ Hz), 125.3, 125.1 (d, $J = 3.3$ Hz), 122.7 (d, $J = 14.9$ Hz), 116.8 (d, $J = 21.8$ Hz), 115.9, 112.0. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{46}\text{H}_{33}\text{F}_2\text{N}_4\text{O}_2$, m/z : 711.2566, found: 711.2576.

***N',N'''*-(6,6'-dibromo-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2s)**



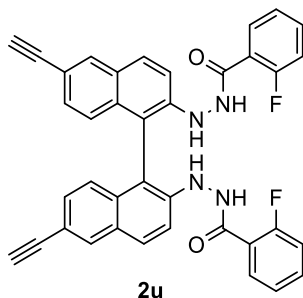
Following the general protocol, compound **2s** was obtained in 92% yield as a white solid. R_f 0.29 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.55 (s, 2H), 8.16 (d, $J = 2.2$ Hz, 2H), 8.01 (d, $J = 9.1$ Hz, 2H), 7.62 – 7.53 (m, 4H), 7.51 (d, $J = 9.1$ Hz, 2H), 7.46 (s, 2H), 7.36 – 7.27 (m, 6H), 6.68 (d, $J = 9.0$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.5, 159.7 (d, $J = 250.3$ Hz), 145.3, 133.6 (d, $J = 8.3$ Hz), 132.5, 130.5, 130.4 (2C), 130.1, 129.7, 126.6, 125.1 (d, $J = 3.2$ Hz), 122.5 (d, $J = 14.9$ Hz), 116.8 (d, $J = 21.6$ Hz), 116.6, 116.3, 111.6. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{23}\text{Br}_2\text{F}_2\text{N}_4\text{O}_2$, m/z : 715.0150, found: 715.0147.

***N',N'''*-(6,6'-divinyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2t**)**



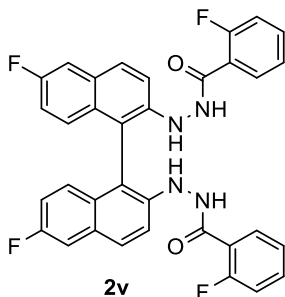
Following the general protocol, compound **2t** was obtained in 58% yield. R_f 0.39 (*n*-hexane: ethyl acetate = 2.5:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.54 (d, $J = 12.9$ Hz, 2H), 8.16 (td, $J = 7.7, 1.8$ Hz, 2H), 7.81 – 7.73 (m, 4H), 7.72 – 7.60 (m, 4H), 7.45 – 7.37 (m, 2H), 7.38 – 7.29 (m, 4H), 7.28 – 7.21 (m, 4H), 7.17 (dd, $J = 8.8, 2.3$ Hz, 2H), 5.82 (d, $J = 17.6$ Hz, 2H), 5.28 (d, $J = 11.4$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 165.5, 160.8 (d, $J = 245.8$ Hz), 145.7, 137.0, 134.2 (d, $J = 9.3$ Hz), 132.3, 129.5, 129.4, 127.7, 127.0, 126.6, 126.5, 126.3, 125.2 (d, $J = 2.5$ Hz), 123.9, 116.4 (d, $J = 20.0$ Hz), 116.2 (d, $J = 14.3$ Hz), 113.0, 108.1. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{38}\text{H}_{29}\text{F}_2\text{O}_2$, m/z : 611.2253, found: 611.2254.

***N',N'''*-(6,6'-diethynyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2u**)**



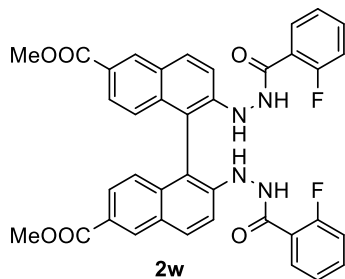
Following the general protocol, compound **2u** was obtained in 83% yield as a white solid. R_f 0.24 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.57 (s, 2H), 8.10 (s, 2H), 8.04 (d, $J = 9.1$ Hz, 2H), 7.60 – 7.49 (m, 8H), 7.39 – 7.20 (m, 6H), 6.74 (d, $J = 8.8$ Hz, 2H), 4.17 (s, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.7 (d, $J = 250.4$ Hz), 145.9, 133.6 (2C), 132.6, 130.4 (2C), 129.6, 128.7, 125.1 (d, $J = 2.7$ Hz), 124.8, 122.5 (d, $J = 14.9$ Hz), 116.8 (d, $J = 21.7$ Hz), 116.37, 116.19, 111.5, 84.4, 80.8. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{38}\text{H}_{25}\text{F}_2\text{N}_4\text{O}_2$, m/z : 607.1940 found: 607.1946.

***N',N'''*-(6,6'-difluoro-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2v)**



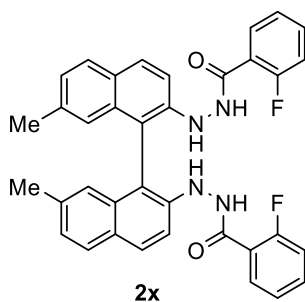
Following the general protocol, compound **2v** was obtained in 92% yield as a white solid. R_f 0.27 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.53 (s, 2H), 8.00 (s, 2H), 7.70 (dd, $J = 9.9, 2.8$ Hz, 2H), 7.54 (s, 6H), 7.45 (d, $J = 2.1$ Hz, 2H), 7.36 – 7.27 (m, 4H), 7.14 (d, $J = 2.8$ Hz, 2H), 6.77 (d, $J = 3.7$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.7 (d, $J = 250.2$ Hz), 158.9 (d, $J = 240.7$ Hz), 144.4, 133.5 (d, $J = 8.4$ Hz), 130.9, 130.4, 129.7 (2C), 127.0 (d, $J = 8.6$ Hz), 125.1 (d, $J = 2.7$ Hz), 122.6 (d, $J = 14.9$ Hz), 117.1 (d, $J = 24.9$ Hz), 116.8 (d, $J = 9.4$ Hz), 116.7, 112.2, 111.7 (d, $J = 20.4$ Hz). **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{23}\text{F}_4\text{N}_4\text{O}_2$, m/z : 595.1752, found: 595.1753.

Dimethyl 2,2'-bis(2-(2-fluorobenzoyl)hydrazineyl)-[1,1'-binaphthalene]-6,6'-dicarboxylate (2w)



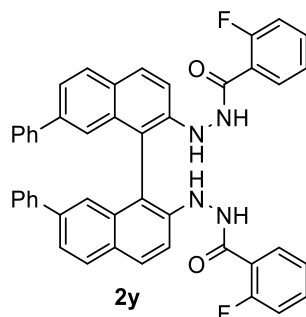
Following the general protocol, compound **2w** was obtained in 88% yield as a white solid. R_f 0.13 (*n*-hexane: ethyl acetate = 2.5:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.61 (s, 2H), 8.62 (s, 2H), 8.25 (d, $J = 9.1$ Hz, 2H), 7.70 (dd, $J = 9.0, 1.9$ Hz, 2H), 7.61 – 7.54 (m, 8H), 7.39 – 7.25 (m, 4H), 6.86 (d, $J = 8.8$ Hz, 2H), 3.87 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 166.9, 165.4, 159.8 (d, $J = 250.4$ Hz), 147.2, 136.3, 133.7 (d, $J = 7.9$ Hz), 132.2, 131.6, 130.4, 128.2, 126.3, 125.1 (d, $J = 2.7$ Hz), 124.6, 124.3, 122.4 (d, $J = 14.7$ Hz), 116.8 (d, $J = 21.8$ Hz), 116.3, 111.3, 52.5. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{38}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_6$, m/z : 675.2050, found: 675.2051.

***N',N'''*-(7,7'-dimethyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2x**)**



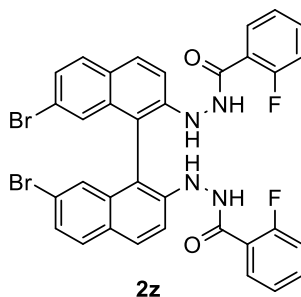
Following the general protocol, compound **2x** was obtained in 83% yield as a white solid. R_f 0.28 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.94 (d, $J = 9.0$ Hz, 2H), 7.79 (d, $J = 8.3$ Hz, 2H), 7.63 – 7.50 (m, 4H), 7.39 (d, $J = 8.9$ Hz, 2H), 7.37 – 7.23 (m, 6H), 7.13 (dd, $J = 8.3, 1.7$ Hz, 2H), 6.57 (s, 2H), 2.15 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.3, 159.7 (d, $J = 250.0$ Hz), 144.7, 136.1, 134.1, 133.5 (d, $J = 8.4$ Hz), 130.4, 129.9, 128.5, 127.5, 125.7, 125.1 (d, $J = 3.5$ Hz), 123.3, 122.7 (d, $J = 14.9$ Hz), 116.8 (d, $J = 21.7$ Hz), 114.4, 111.9, 22.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_2$, m/z : 587.2253, found: 587.2249.

***N',N'''*-(7,7'-diphenyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2y**)**



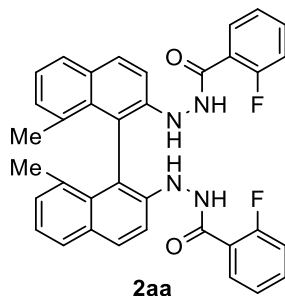
Following the general protocol, compound **2y** was obtained in 84% yield as a white solid. R_f 0.25 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.55 (s, 2H), 8.03 (dd, $J = 31.9, 8.7$ Hz, 4H), 7.63 – 7.50 (m, 8H), 7.46 (s, 2H), 7.37 – 7.31 (m, 4H), 7.31 – 7.26 (m, 8H), 7.25 – 7.20 (m, 2H), 7.07 (s, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.4, 159.8 (d, $J = 250.1$ Hz), 145.3, 141.1, 138.8, 134.1, 133.5 (d, $J = 8.2$ Hz), 130.4, 130.1, 129.4 (3C), 128.5, 127.8, 127.1 (2C), 125.1 (d, $J = 3.4$ Hz), 122.9, 122.7 (d, $J = 14.8$ Hz), 122.2, 116.8 (d, $J = 21.8$ Hz), 115.6, 112.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{46}\text{H}_{33}\text{F}_2\text{N}_4\text{O}_6$, m/z : 711.2566, found: 711.2567.

***N',N'''*-(7,7'-dibromo-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2z**)**



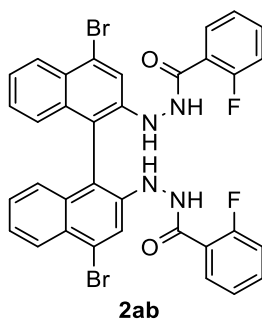
Following the general protocol, compound **2z** was obtained in 93% yield as a white solid. R_f 0.24 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.54 (s, 2H), 8.06 (d, $J = 9.1$ Hz, 2H), 7.89 (d, $J = 8.6$ Hz, 2H), 7.61 – 7.47 (m, 6H), 7.45 – 7.38 (m, 4H), 7.37 – 7.26 (m, 4H), 6.88 (s, 2H). $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 165.3, 159.8 (d, $J = 250.3$ Hz), 146.0, 135.1, 133.6 (d, $J = 8.5$ Hz), 131.2, 130.8, 130.4 (d, $J = 2.7$ Hz), 127.7, 126.5, 125.7, 125.1 (d, $J = 3.3$ Hz), 122.4 (d, $J = 14.8$ Hz), 121.1, 116.8 (d, $J = 21.7$ Hz), 116.0, 110.2. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{23}\text{Br}_2\text{F}_2\text{N}_4\text{O}_2$, m/z : 715.0150, found: 715.0151.

***N',N'''*-(8,8'-dimethyl-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (**2aa**)**



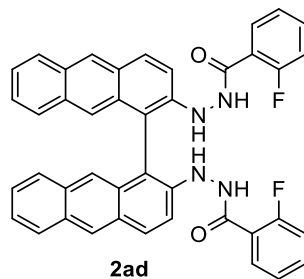
Following the general protocol, compound **2aa** was obtained in 82% yield as a white solid. R_f 0.35 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.47 (d, $J = 11.3$ Hz, 2H), 8.04 – 7.95 (m, 2H), 7.64 (d, $J = 8.7$ Hz, 2H), 7.51 (d, $J = 7.9$ Hz, 2H), 7.49 – 7.41 (m, 2H), 7.28 (d, $J = 2.3$ Hz, 2H), 7.21 (t, $J = 7.4$ Hz, 2H), 7.17 – 7.08 (m, 6H), 7.08 – 7.03 (m, 2H), 2.48 (s, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 164.0 (d, $J = 3.7$ Hz), 160.7 (d, $J = 248.2$ Hz), 145.4, 134.1 (d, $J = 9.3$ Hz), 133.5, 132.9, 132.1 (d, $J = 2.5$ Hz), 130.0, 129.6, 127.2, 126.2, 125.2 (d, $J = 3.1$ Hz), 123.3, 116.2 (d, $J = 24.4$ Hz), 115.6, 115.3, 105.0, 19.4. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{36}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_2$, m/z 587.2253 found: 587.2252.

***N',N'''*-(4,4'-dibromo-[1,1'-binaphthalene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2ab)**



Following the general protocol, compound **2ab** was obtained in 72% yield as a white solid. R_f 0.43 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 10.55 (s, 2H), 8.11 (d, $J = 8.4$ Hz, 2H), 7.78 (s, 2H), 7.61 – 7.56 (m, 2H), 7.51 (t, $J = 6.5$ Hz, 2H), 7.47 – 7.42 (m, 4H), 7.39 – 7.33 (m, 2H), 7.31 (t, $J = 7.5$ Hz, 4H), 6.86 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, DMSO) δ 165.4, 159.7 (d, $J = 249.7$ Hz), 145.2, 134.9, 133.7 (d, $J = 8.7$ Hz), 130.3, 130.2, 128.3, 127.3, 125.2 (2C), 125.1, 124.5, 122.4 (d, $J = 15.1$ Hz), 119.3, 116.8 (d, $J = 21.7$ Hz), 111.7. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{34}\text{H}_{23}\text{Br}_2\text{F}_2\text{N}_4\text{O}_2$, m/z : 715.015, found: 715.0145.

***N',N'''*-([1,1'-bianthracene]-2,2'-diyl)bis(2-fluorobenzohydrazide) (2ad)**

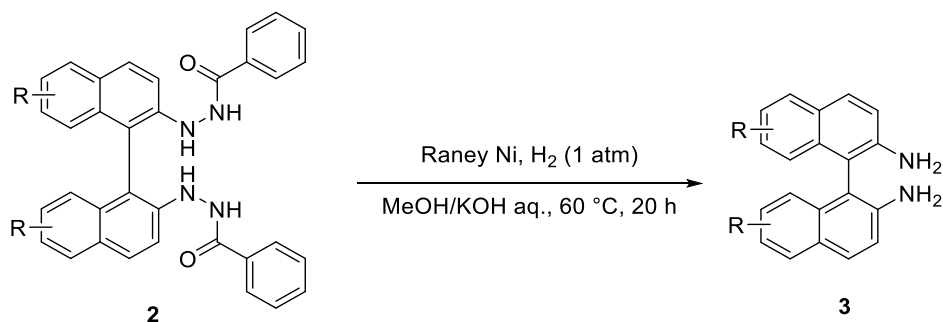


Following the general protocol, compound **2ad** was obtained in 58% yield as a white solid. R_f 0.44 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.57 (d, $J = 12.4$ Hz, 2H), 8.31 (s, 2H), 8.19 (s, 2H), 8.14 (dd, $J = 7.7, 1.9$ Hz, 2H), 7.97 – 7.86 (m, 6H), 7.43 – 7.30 (m, 8H), 7.28 – 7.24 (m, 4H), 7.16 (dd, $J = 9.0, 2.2$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 163.7, 160.8 (d, $J = 248.0$ Hz), 144.7, 134.2 (d, $J = 9.3$ Hz), 132.3 (d, $J = 2.3$ Hz), 130.8, 130.4, 129.9, 128.8, 128.3, 127.7, 126.3, 125.5, 125.2 (d, $J = 3.2$ Hz), 124.4, 124.2, 122.9, 117.7, 116.6, 116.3 (d, $J = 24.5$ Hz), 106.0. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{42}\text{H}_{29}\text{F}_2\text{N}_4\text{O}_2$, m/z : 659.2253, found: 659.2254.

Gram scale synthesis of BINAM derivatives

Azonaphthalene (15 g, 57.7 mmol), B_2cat_2 (15 g, 63.5 mmol) and 2,6-dibromopyridine (1.4 g, 5.77 mmol) were added to a 500 mL dry round-bottomed flask equipped with a Teflon-coated magnetic stirring bar. Anhydrous 1,2-dimethoxyethane (280 mL) was added and the reaction was stirred at room temperature for 1 hour. The reaction mixture was then concentrated to dryness and purified by washing with warm methanol to give 13.7 g of **2a** (91% yield).

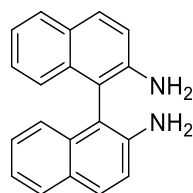
Cleavage of *N-N* Bond — Accessing free BINAM



A Schlenk tube containing approximately 100 mg of Raney nickel and equipped with a Teflon-coated magnetic stir bar was washed with methanol (5 x 2 mL). Methanol (1 mL), BINAM derivative **2** (0.1 mmol, 1 equiv.), and aqueous potassium hydroxide solution (0.5 mmol, 0.2 M) were added. The reaction mixture was degassed, backfilled with hydrogen gas (H_2 balloon), and

stirred at 60 °C until complete consumption of the starting material. The reaction was allowed to cool to room temperature and filtered carefully whilst washing with ethyl acetate, and the filtrate was then concentrated to dryness. The resulting residue was dissolved in dichloromethane, washed with brine, and dried over sodium sulfate. The organic phase was filtered off, evaporated to dryness, and purified by flash chromatography using (n-hexane: ethyl acetate = 6:1 to 4:1) to obtain **3** as a white solid.

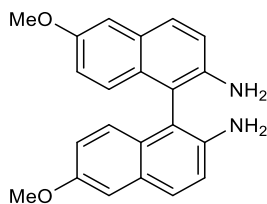
[1,1'-binaphthalene]-2,2'-diamine (**3a**)



3a

Following the general protocol, compound **3a** was obtained in 90% yield; R_f 0.41 (n-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.74 – 7.67 (m, 4H), 7.17 – 7.08 (m, 4H), 7.05 (d, $J = 8.7$ Hz, 2H), 6.99 (d, $J = 8.5$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 142.5, 133.7, 129.6, 128.6, 128.2, 126.9, 124.0, 122.5, 118.4, 112.8. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{20}\text{H}_{17}\text{N}_2$, m/z : 285.1386, found: 285.1384.

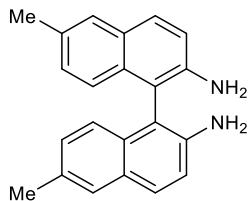
6,6'-dimethoxy-[1,1'-binaphthalene]-2,2'-diamine (**3n**)



3n

Following the general protocol, compound **3n** was obtained in 83% yield as a white solid. R_f 0.24 (n-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.70 (d, $J = 8.7$ Hz, 2H), 7.14 (dd, $J = 5.7, 3.0$ Hz, 4H), 6.99 (d, $J = 9.1$ Hz, 2H), 6.90 (dd, $J = 9.2, 2.6$ Hz, 2H), 3.88 (s, 6H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 155.5, 140.6, 129.4, 129.0, 128.2, 125.7, 119.1, 119.0, 113.8, 106.8, 55.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{22}\text{H}_{21}\text{N}_2\text{O}_2$, m/z : 345.1598, found: 345.1596.

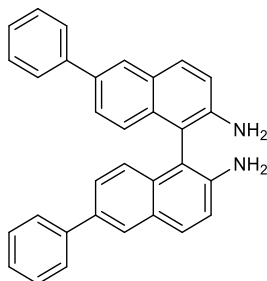
6,6'-dimethyl-[1,1'-binaphthalene]-2,2'-diamine (**3o**)



3o

Following the general protocol, compound **3o** was obtained in 89% yield as a white solid. R_f 0.47 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.75 (d, $J = 8.8$ Hz, 2H), 7.60 (s, 2H), 7.15 (d, $J = 8.7$ Hz, 2H), 7.07 (d, $J = 8.7$ Hz, 2H), 7.01 (d, $J = 8.6$ Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 141.8, 131.9, 131.8, 129.0, 128.8, 127.3, 124.0, 118.5, 113.1, 21.3. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{22}\text{H}_{21}\text{N}_2$, m/z : 313.1699, found: 313.1699.

6,6'-diphenyl-[1,1'-binaphthalene]-2,2'-diamine (**3r**)

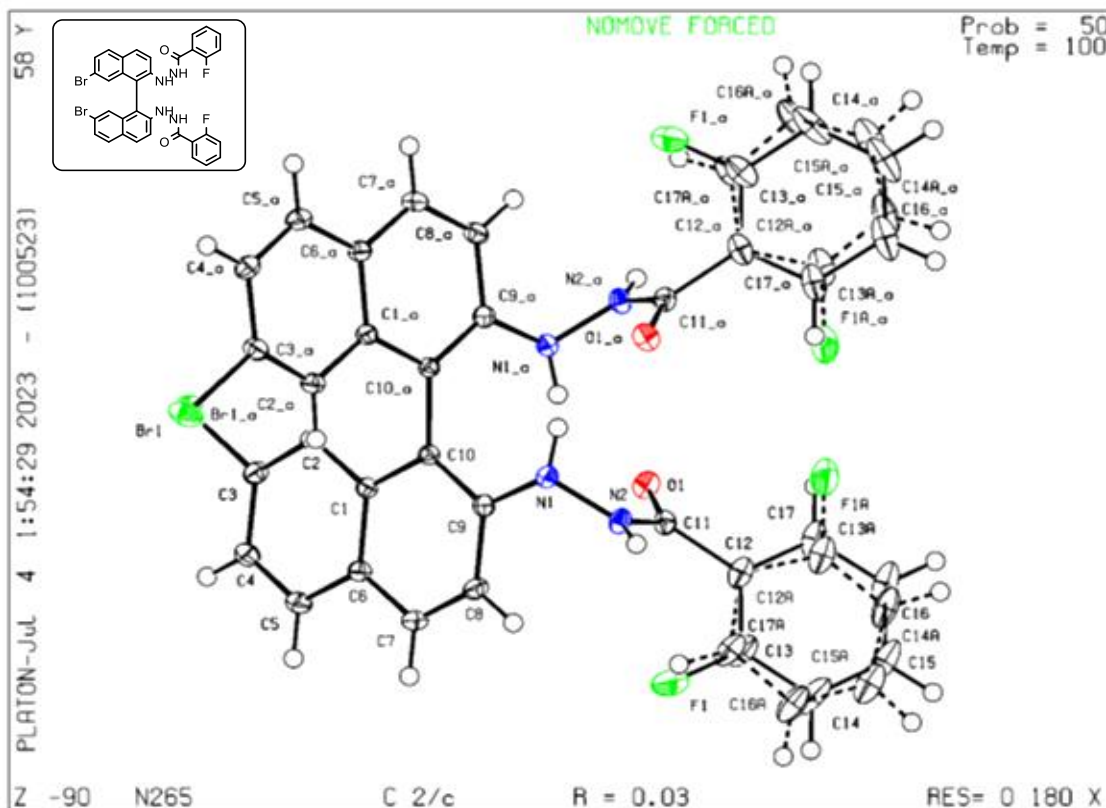


3r

Following the general protocol, compound **3r** was obtained in 85% yield as a white solid. R_f 0.36 (*n*-hexane: ethyl acetate = 3:1). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.05 (d, $J = 1.9$ Hz, 2H), 7.91 (d, $J = 8.8$ Hz, 2H), 7.69 (d, $J = 6.9$ Hz, 4H), 7.53 (dd, $J = 8.7, 2.0$ Hz, 2H), 7.47 (t, $J = 7.7$ Hz, 4H), 7.35 (t, $J = 7.4$ Hz, 2H), 7.23 (dd, $J = 8.7, 3.7$ Hz, 4H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 142.9, 141.3, 135.3, 132.9, 123.0, 128.8 (3C), 127.1 (2C), 126.9, 126.5, 126.2, 124.3, 118.8, 112.4. **HRMS (ESI)** calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{32}\text{H}_{25}\text{N}_2$, m/z : 437.2012, found: 437.2011.

Crystal Structure of **2z**

The Cambridge Crystallographic Data Centre (CCDC) has received the crystallographic data for compound **2z** (CCDC 2294982) which is accessible at no cost via www.ccdc.cam.ac.uk/conts/retrieving.html.



Single Crystal Structure X-ray Analysis

Sample Code:	N265
Sample ID:	et-7br-bm
Student/Researcher:	Emmanuella Twumasi
Supervisor:	Lu Yixin
CDCC:	2294982
Date:	4-07-2023

Note: The crystal is monoclinic, space group C2/c. The asymmetric unit contains half a molecule of the compound $C_{34}H_{22}Br_2F_2N_4O_2$. The 1-fluoro-benzene group was disordered by flipping over with occupancy ratio =87:13.

Restraints in bond lengths and thermal parameters were applied to the disordered atoms. Hydrogens on the N atoms were located from different map. Final R values are R1=0.0266 and wR2=0.0665 for 2- theta up to 59°.

Supplementary Table 3. Crystal data and structure refinement for N265.

Identification code	N265	
Empirical formula	C34 H22 Br2 F2 N4 O2	
Formula weight	716.37	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 13.8725(9) Å	a = 90°.
	b = 29.1188(18) Å	b = 127.298(2)°.
	c = 9.3620(6) Å	g = 90°.
Volume	3008.4(3) Å ³	
Z	4	
Density (calculated)	1.582 Mg/m ³	
Absorption coefficient	2.746 mm ⁻¹	
F(000)	1432	
Crystal size	0.299 x 0.294 x 0.124 mm ³	
Theta range for data collection	2.293 to 29.579°.	
Index ranges	-19<=h<=19, -37<=k<=40, -13<=l<=12	
Reflections collected	52265	
Independent reflections	4220 [R(int) = 0.0480]	
Completeness to theta = 25.242°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7459 and 0.6142	
Refinement method	Full-matrix least-squares on F ²	
Data/restraints /parameters	4220 / 250 / 262	
Goodness-of-fit on F ²	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0266, wR2 = 0.0640	

R indices (all data)	R1 = 0.0313, wR2 = 0.0665
Extinction coefficient	n/a
Largest diff. peak and hole	0.984 and -0.902 e.Å ⁻³

Supplementary Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for N265. U(eq) is defined as one-third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	3911(1)	2274(1)	10129(1)	26(1)
O(1)	3656(1)	4855(1)	6716(1)	19(1)
N(1)	4470(1)	4300(1)	5285(2)	15(1)
N(2)	3809(1)	4709(1)	4487(2)	15(1)
C(1)	3590(1)	3290(1)	6722(2)	13(1)
C(2)	4057(1)	2987(1)	8197(2)	15(1)
C(3)	3305(1)	2668(1)	8138(2)	17(1)
C(4)	2081(1)	2620(1)	6662(2)	18(1)
C(5)	1618(1)	2908(1)	5227(2)	18(1)
C(6)	2342(1)	3249(1)	5222(2)	15(1)
C(7)	1858(1)	3559(1)	3766(2)	18(1)
C(8)	2552(1)	3900(1)	3825(2)	17(1)
C(9)	3795(1)	3946(1)	5332(2)	14(1)
C(10)	4333(1)	3638(1)	6746(2)	12(1)
C(11)	3433(1)	4960(1)	5266(2)	16(1)
C(12)	2724(2)	5384(1)	4273(2)	21(1)
C(13)	1729(5)	5388(1)	2490(5)	28(1)
C(14)	1063(4)	5784(1)	1629(5)	40(1)
C(15)	1418(3)	6186(1)	2595(4)	40(1)
C(16)	2412(3)	6195(1)	4394(4)	36(1)

C(17)	3061(3)	5794(1)	5242(4)	27(1)
F(1)	1400(1)	4988(1)	1557(2)	36(1)
C(12A)	2724(2)	5384(1)	4273(2)	21(1)
C(13A)	2870(30)	5839(6)	4860(20)	30(3)
C(14A)	2140(20)	6197(6)	3790(30)	31(3)
C(15A)	1160(20)	6107(8)	2070(30)	33(3)
C(16A)	1000(30)	5679(8)	1370(30)	28(2)
C(17A)	1790(30)	5325(9)	2390(30)	22(2)
F(1A)	3867(9)	5855(3)	6622(13)	41(2)

Supplementary Table 5. Bond lengths [\AA] and angles [$^\circ$] for N265.

Br(1)-C(3)	1.8962(14)
O(1)-C(11)	1.2342(18)
N(1)-C(9)	1.4106(18)
N(1)-N(2)	1.4110(16)
N(1)-H(1)	0.86(2)
N(2)-C(11)	1.3406(19)
N(2)-H(2A)	0.84(2)
C(1)-C(2)	1.4220(19)
C(1)-C(6)	1.4270(18)
C(1)-C(10)	1.4359(19)
C(2)-C(3)	1.372(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.404(2)
C(4)-C(5)	1.369(2)
C(4)-H(4)	0.9500
C(5)-C(6)	1.415(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.419(2)

C(7)-C(8)	1.361(2)
C(7)-H(7)	0.9500
C(8)-C(9)	1.4260(19)
C(8)-H(8)	0.9500
C(9)-C(10)	1.3848(18)
C(10)-C(10)#1	1.501(3)
C(11)-C(12A)	1.499(2)
C(11)-C(12)	1.499(2)
C(12)-C(13)	1.378(4)
C(12)-C(17)	1.399(3)
C(13)-F(1)	1.357(4)
C(13)-C(14)	1.388(3)
C(14)-C(15)	1.377(4)
C(14)-H(14)	0.9500
C(15)-C(16)	1.387(4)
C(15)-H(15)	0.9500
C(16)-C(17)	1.393(3)
C(16)-H(16)	0.9500
C(17)-H(17)	0.9500
C(12A)-C(13A)	1.402(16)
C(12A)-C(17A)	1.429(17)
C(13A)-F(1A)	1.367(15)
C(13A)-C(14A)	1.377(16)
C(14A)-C(15A)	1.362(16)
C(14A)-H(14A)	0.9500
C(15A)-C(16A)	1.363(16)
C(15A)-H(15A)	0.9500
C(16A)-C(17A)	1.378(17)
C(16A)-H(16A)	0.9500
C(17A)-H(17A)	0.9500

C(9)-N(1)-N(2)	113.75(11)
C(9)-N(1)-H(1)	114.0(14)
N(2)-N(1)-H(1)	109.8(14)
C(11)-N(2)-N(1)	120.95(12)
C(11)-N(2)-H(2A)	121.2(14)
N(1)-N(2)-H(2A)	117.8(14)
C(2)-C(1)-C(6)	118.12(12)
C(2)-C(1)-C(10)	121.52(12)
C(6)-C(1)-C(10)	120.33(12)
C(3)-C(2)-C(1)	119.53(13)
C(3)-C(2)-H(2)	120.2
C(1)-C(2)-H(2)	120.2
C(2)-C(3)-C(4)	122.98(13)
C(2)-C(3)-Br(1)	119.73(11)
C(4)-C(3)-Br(1)	117.28(11)
C(5)-C(4)-C(3)	118.20(13)
C(5)-C(4)-H(4)	120.9
C(3)-C(4)-H(4)	120.9
C(4)-C(5)-C(6)	121.48(13)
C(4)-C(5)-H(5)	119.3
C(6)-C(5)-H(5)	119.3
C(5)-C(6)-C(7)	121.73(13)
C(5)-C(6)-C(1)	119.67(13)
C(7)-C(6)-C(1)	118.60(13)
C(8)-C(7)-C(6)	120.88(13)
C(8)-C(7)-H(7)	119.6
C(6)-C(7)-H(7)	119.6
C(7)-C(8)-C(9)	120.70(13)
C(7)-C(8)-H(8)	119.7
C(9)-C(8)-H(8)	119.7
C(10)-C(9)-N(1)	121.31(12)

C(10)-C(9)-C(8)	120.85(13)
N(1)-C(9)-C(8)	117.76(12)
C(9)-C(10)-C(1)	118.48(12)
C(9)-C(10)-C(10)#1	122.85(11)
C(1)-C(10)-C(10)#1	118.61(11)
O(1)-C(11)-N(2)	122.89(13)
O(1)-C(11)-C(12A)	121.26(13)
N(2)-C(11)-C(12A)	115.85(13)
O(1)-C(11)-C(12)	121.26(13)
N(2)-C(11)-C(12)	115.85(13)
C(13)-C(12)-C(17)	118.3(2)
C(13)-C(12)-C(11)	123.92(19)
C(17)-C(12)-C(11)	117.74(16)
F(1)-C(13)-C(12)	118.1(2)
F(1)-C(13)-C(14)	119.4(3)
C(12)-C(13)-C(14)	122.5(3)
C(15)-C(14)-C(13)	118.5(3)
C(15)-C(14)-H(14)	120.7
C(13)-C(14)-H(14)	120.7
C(14)-C(15)-C(16)	120.7(2)
C(14)-C(15)-H(15)	119.6
C(16)-C(15)-H(15)	119.6
C(15)-C(16)-C(17)	120.0(3)
C(15)-C(16)-H(16)	120.0
C(17)-C(16)-H(16)	120.0
C(16)-C(17)-C(12)	120.0(2)
C(16)-C(17)-H(17)	120.0
C(12)-C(17)-H(17)	120.0
C(13A)-C(12A)-C(17A)	113.8(11)
C(13A)-C(12A)-C(11)	130.5(7)
C(17A)-C(12A)-C(11)	115.5(9)

F(1A)-C(13A)-C(14A)	127.8(15)
F(1A)-C(13A)-C(12A)	107.9(11)
C(14A)-C(13A)-C(12A)	124.3(13)
C(15A)-C(14A)-C(13A)	118.8(17)
C(15A)-C(14A)-H(14A)	120.6
C(13A)-C(14A)-H(14A)	120.6
C(14A)-C(15A)-C(16A)	120.1(16)
C(14A)-C(15A)-H(15A)	119.9
C(16A)-C(15A)-H(15A)	119.9
C(15A)-C(16A)-C(17A)	121.2(18)
C(15A)-C(16A)-H(16A)	119.4
C(17A)-C(16A)-H(16A)	119.4
C(16A)-C(17A)-C(12A)	120.9(17)
C(16A)-C(17A)-H(17A)	119.5
C(12A)-C(17A)-H(17A)	119.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+3/2

Supplementary Table 6. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for N265. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^*2U^{11} + \dots + 2hka^*b^*U^{12}]$

	U11	U22	U33	U23	U13	U12
Br(1)	17(1)	33(1)	25(1)	14(1)	10(1)	1(1)
O(1)	24(1)	20(1)	16(1)	2(1)	14(1)	4(1)
N(1)	14(1)	13(1)	15(1)	1(1)	8(1)	1(1)
N(2)	20(1)	14(1)	15(1)	3(1)	12(1)	2(1)
C(1)	12(1)	13(1)	14(1)	-1(1)	7(1)	1(1)
C(2)	12(1)	17(1)	14(1)	1(1)	6(1)	1(1)

C(3)	15(1)	18(1)	16(1)	3(1)	9(1)	2(1)
C(4)	15(1)	18(1)	20(1)	-1(1)	9(1)	-4(1)
C(5)	13(1)	20(1)	17(1)	-1(1)	7(1)	-2(1)
C(6)	12(1)	15(1)	14(1)	-1(1)	7(1)	0(1)
C(7)	12(1)	20(1)	14(1)	0(1)	5(1)	1(1)
C(8)	16(1)	18(1)	13(1)	2(1)	7(1)	2(1)
C(9)	14(1)	14(1)	14(1)	-1(1)	9(1)	1(1)
C(10)	11(1)	12(1)	13(1)	-1(1)	7(1)	1(1)
C(11)	17(1)	14(1)	18(1)	0(1)	11(1)	-1(1)
C(12)	25(1)	18(1)	27(1)	6(1)	21(1)	6(1)
C(13)	28(1)	29(1)	31(1)	9(1)	19(1)	12(1)
C(14)	42(1)	40(2)	40(1)	22(1)	26(1)	26(1)
C(15)	54(2)	32(1)	54(2)	25(1)	42(2)	26(1)
C(16)	53(2)	20(1)	54(2)	12(1)	43(1)	13(1)
C(17)	38(2)	17(1)	38(1)	5(1)	30(1)	7(1)
F(1)	26(1)	39(1)	25(1)	1(1)	5(1)	5(1)
C(12A)	25(1)	18(1)	27(1)	6(1)	21(1)	6(1)
C(13A)	39(3)	20(3)	38(3)	6(2)	28(3)	8(3)
C(14A)	45(4)	21(3)	44(3)	9(3)	36(3)	15(3)
C(15A)	45(3)	27(3)	41(3)	15(3)	33(3)	15(3)
C(16A)	35(3)	29(3)	36(3)	13(3)	29(2)	19(3)
C(17A)	25(3)	26(3)	28(2)	12(3)	23(2)	13(3)
F(1A)	50(4)	20(3)	40(3)	-2(2)	21(3)	5(3)

Supplementary Table 7. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for N265.

	x	y	z	U(eq)
H(1)	5144(19)	4359(7)	6310(30)	22(5)
H(2A)	3703(19)	4800(7)	3550(30)	24(5)
H(2)	4883	3005	9217	18
H(4)	1586	2393	6658	22
H(5)	793	2877	4212	21
H(7)	1038	3528	2736	21
H(8)	2205	4110	2853	20
H(14)	378	5777	400	48
H(15)	978	6461	2023	48
H(16)	2650	6475	5047	43
H(17)	3732	5799	6480	32
H(14A)	2304	6502	4245	38
H(15A)	591	6342	1355	39
H(16A)	339	5623	149	34
H(17A)	1704	5039	1843	27

Supplementary Table 8. Torsion angles [°] for N265.

C(9)-N(1)-N(2)-C(11)	66.65(17)
C(6)-C(1)-C(2)-C(3)	0.1(2)
C(10)-C(1)-C(2)-C(3)	178.17(13)
C(1)-C(2)-C(3)-C(4)	0.8(2)
C(1)-C(2)-C(3)-Br(1)	-178.20(10)
C(2)-C(3)-C(4)-C(5)	-0.6(2)
Br(1)-C(3)-C(4)-C(5)	178.49(12)
C(3)-C(4)-C(5)-C(6)	-0.7(2)
C(4)-C(5)-C(6)-C(7)	-177.84(14)
C(4)-C(5)-C(6)-C(1)	1.6(2)
C(2)-C(1)-C(6)-C(5)	-1.3(2)
C(10)-C(1)-C(6)-C(5)	-179.36(13)
C(2)-C(1)-C(6)-C(7)	178.17(13)
C(10)-C(1)-C(6)-C(7)	0.1(2)
C(5)-C(6)-C(7)-C(8)	176.66(14)
C(1)-C(6)-C(7)-C(8)	-2.8(2)
C(6)-C(7)-C(8)-C(9)	1.9(2)
N(2)-N(1)-C(9)-C(10)	-145.63(13)
N(2)-N(1)-C(9)-C(8)	37.75(17)
C(7)-C(8)-C(9)-C(10)	1.7(2)
C(7)-C(8)-C(9)-N(1)	178.39(13)
N(1)-C(9)-C(10)-C(1)	179.14(12)
C(8)-C(9)-C(10)-C(1)	-4.3(2)
N(1)-C(9)-C(10)-C(10)#1	-3.9(2)
C(8)-C(9)-C(10)-C(10)#1	172.66(13)
C(2)-C(1)-C(10)-C(9)	-174.60(13)
C(6)-C(1)-C(10)-C(9)	3.41(19)
C(2)-C(1)-C(10)-C(10)#1	8.3(2)
C(6)-C(1)-C(10)-C(10)#1	-173.71(13)

N(1)-N(2)-C(11)-O(1)	1.3(2)
N(1)-N(2)-C(11)-C(12A)	-179.15(12)
N(1)-N(2)-C(11)-C(12)	-179.15(12)
O(1)-C(11)-C(12)-C(13)	-128.7(4)
N(2)-C(11)-C(12)-C(13)	51.8(4)
O(1)-C(11)-C(12)-C(17)	48.0(3)
N(2)-C(11)-C(12)-C(17)	-131.6(3)
C(17)-C(12)-C(13)-F(1)	-179.5(4)
C(11)-C(12)-C(13)-F(1)	-2.9(8)
C(17)-C(12)-C(13)-C(14)	0.7(8)
C(11)-C(12)-C(13)-C(14)	177.4(4)
F(1)-C(13)-C(14)-C(15)	-179.4(5)
C(12)-C(13)-C(14)-C(15)	0.3(9)
C(13)-C(14)-C(15)-C(16)	-0.7(7)
C(14)-C(15)-C(16)-C(17)	-0.1(6)
C(15)-C(16)-C(17)-C(12)	1.2(6)
C(13)-C(12)-C(17)-C(16)	-1.5(6)
C(11)-C(12)-C(17)-C(16)	-178.3(3)
O(1)-C(11)-C(12A)-C(13A)	50(2)
N(2)-C(11)-C(12A)-C(13A)	-129(2)
O(1)-C(11)-C(12A)-C(17A)	-134(2)
N(2)-C(11)-C(12A)-C(17A)	47(2)
C(17A)-C(12A)-C(13A)-F(1A)	-174(3)
C(11)-C(12A)-C(13A)-F(1A)	2(4)
C(17A)-C(12A)-C(13A)-C(14A)	5(5)
C(11)-C(12A)-C(13A)-C(14A)	-179(2)
F(1A)-C(13A)-C(14A)-C(15A)	-178(3)
C(12A)-C(13A)-C(14A)-C(15A)	3(5)
C(13A)-C(14A)-C(15A)-C(16A)	-7(5)
C(14A)-C(15A)-C(16A)-C(17A)	3(5)
C(15A)-C(16A)-C(17A)-C(12A)	6(6)

C(13A)-C(12A)-C(17A)-C(16A)	-10(5)
C(11)-C(12A)-C(17A)-C(16A)	174(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+3/2

Supplementary Table 9. Hydrogen bonds for N265 [\AA and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
N(1)-H(1)...O(1)#1	0.86(2)	2.13(2)	2.9000(16)	148.8(18)
N(2)-H(2A)...O(1)#2	0.84(2)	1.96(2)	2.7781(16)	166(2)

Symmetry transformations used to generate equivalent atoms:

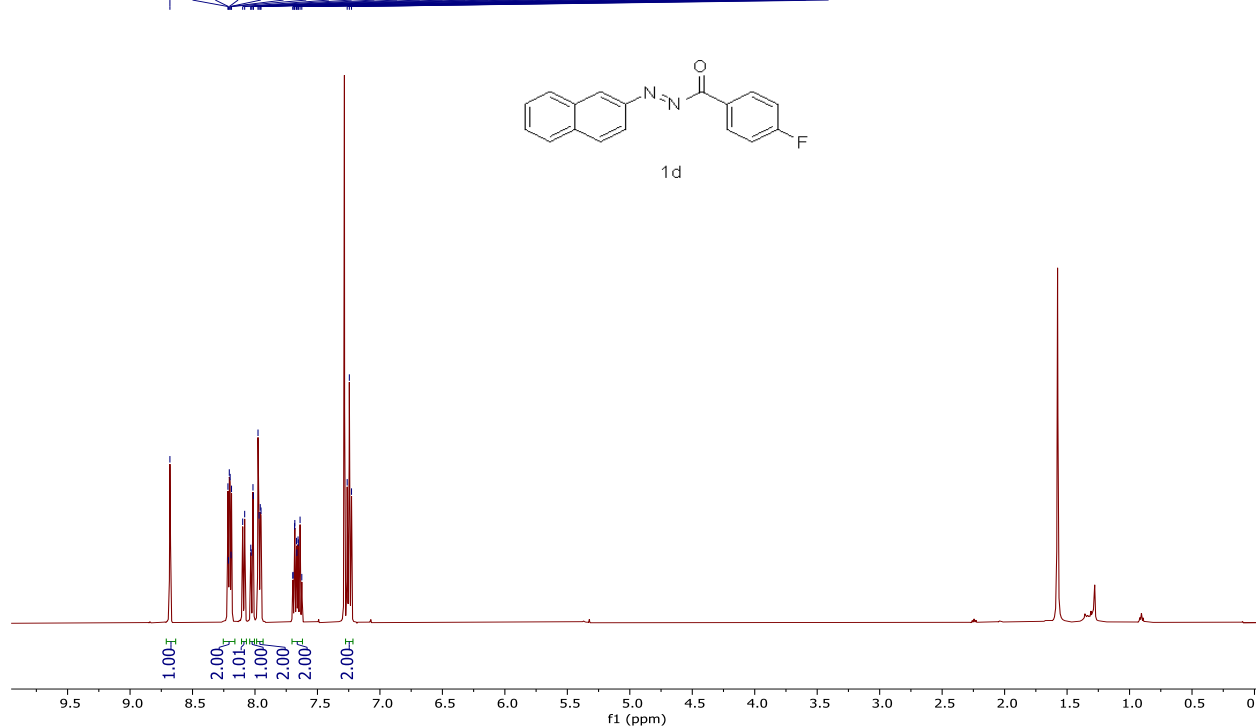
#1 -x+1,y,-z+3/2 #2 x,-y+1,z-1/2

References

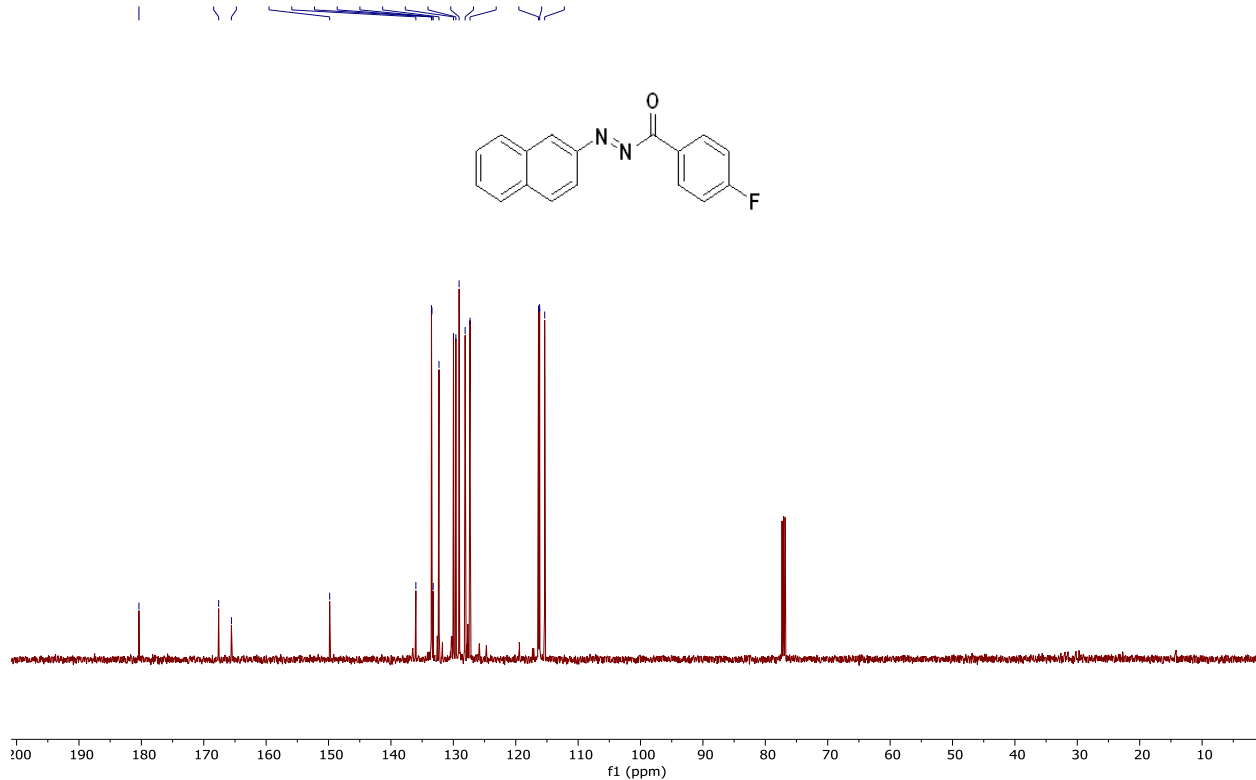
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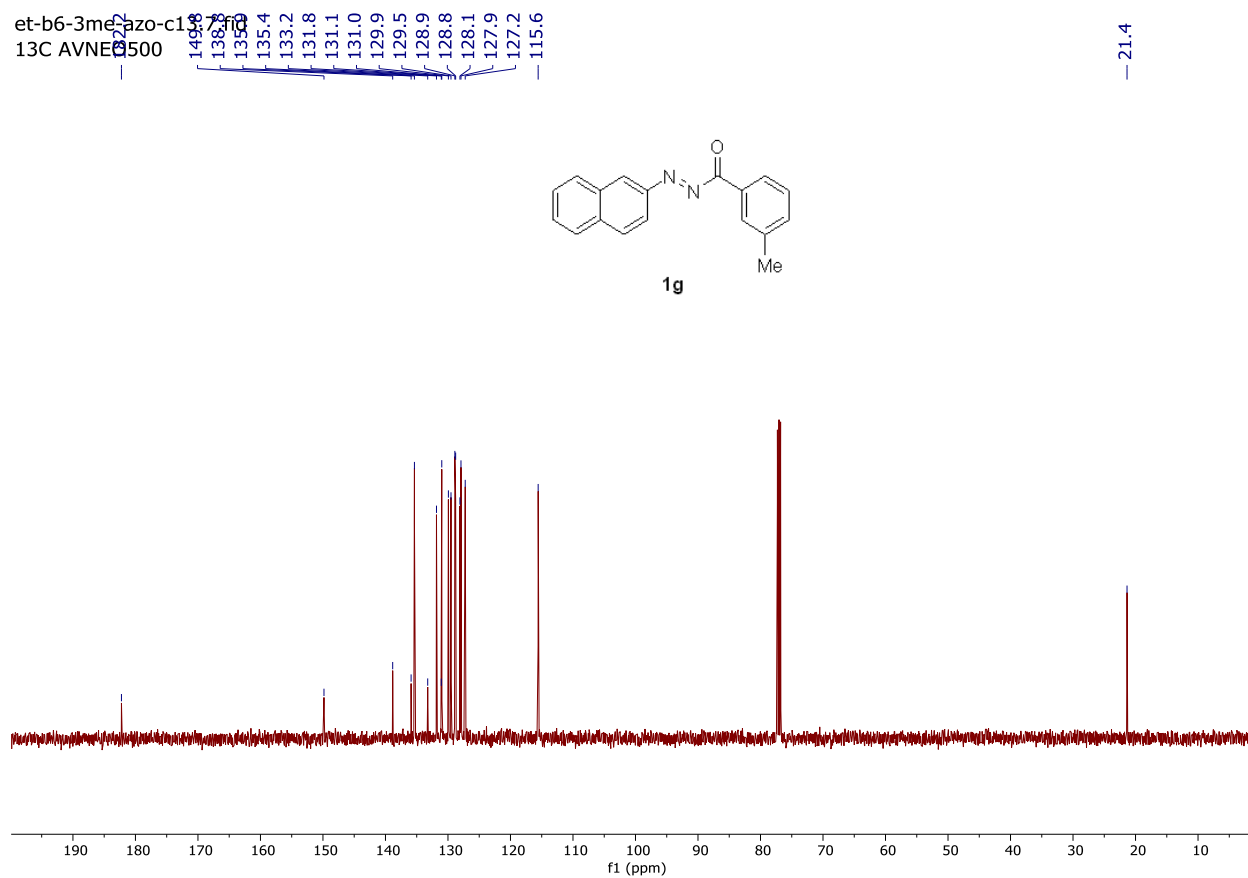
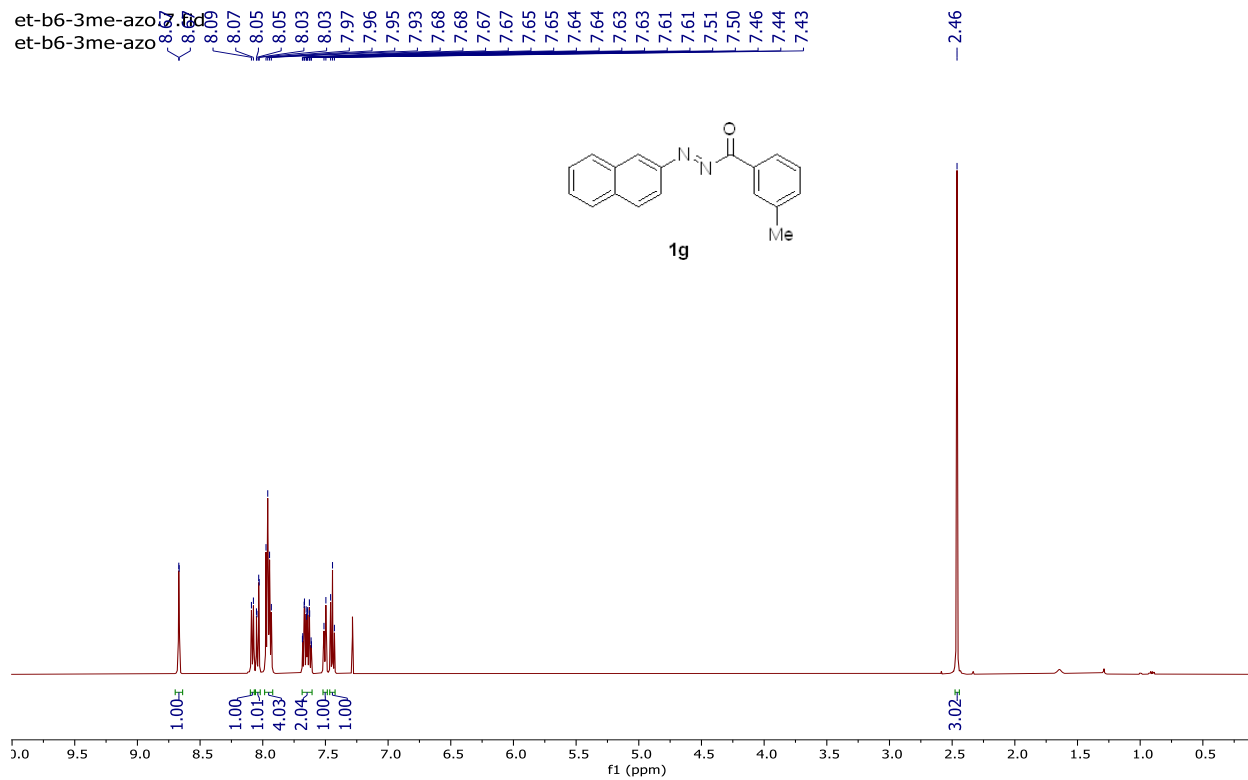
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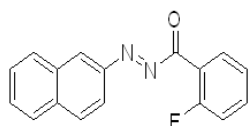


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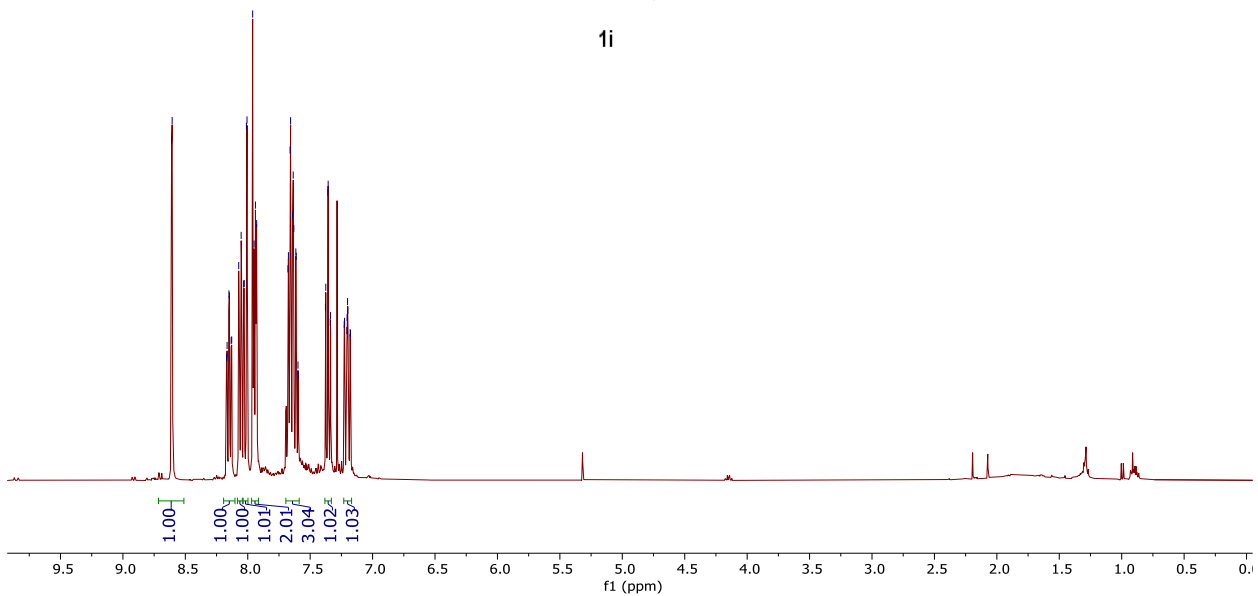




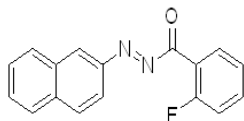
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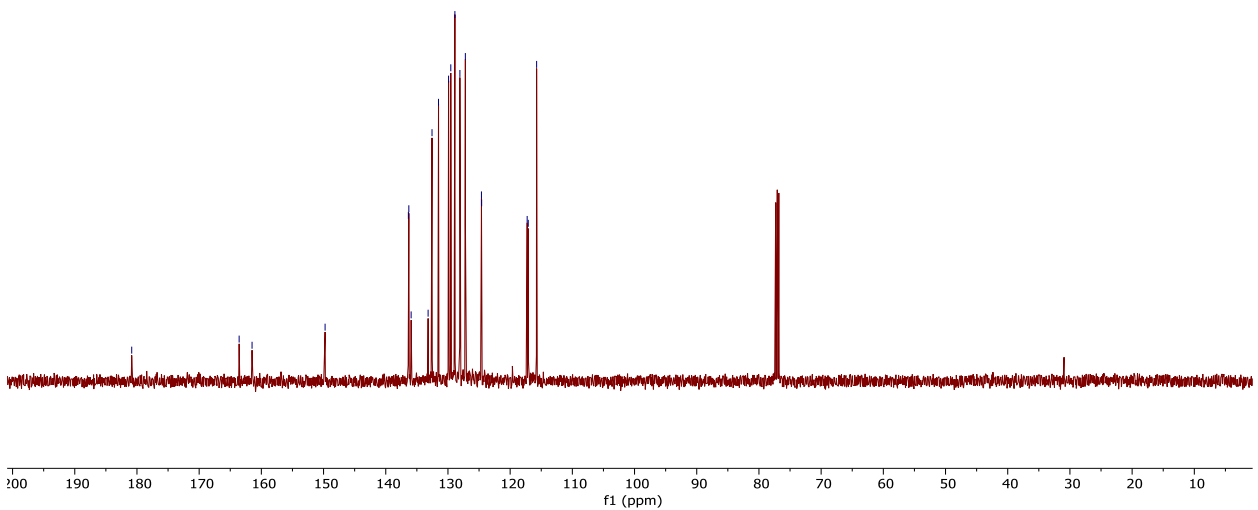
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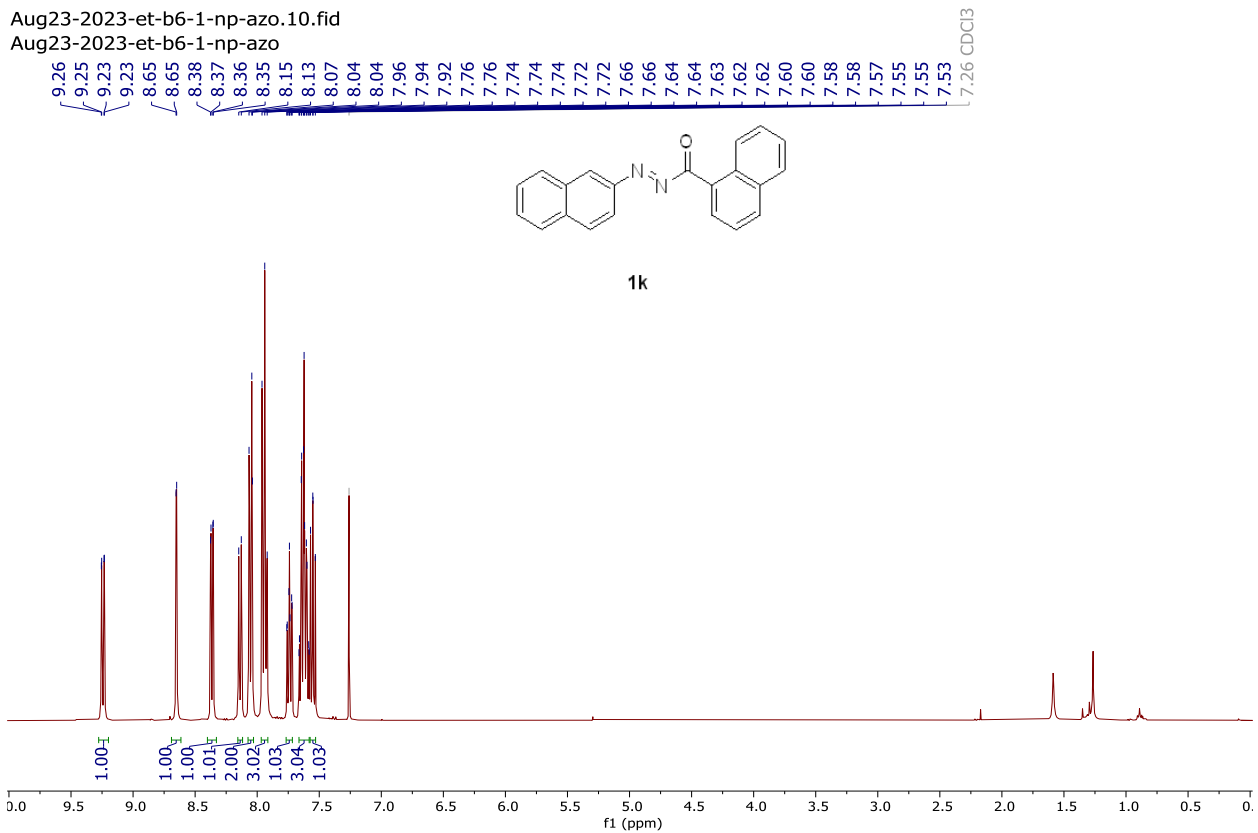


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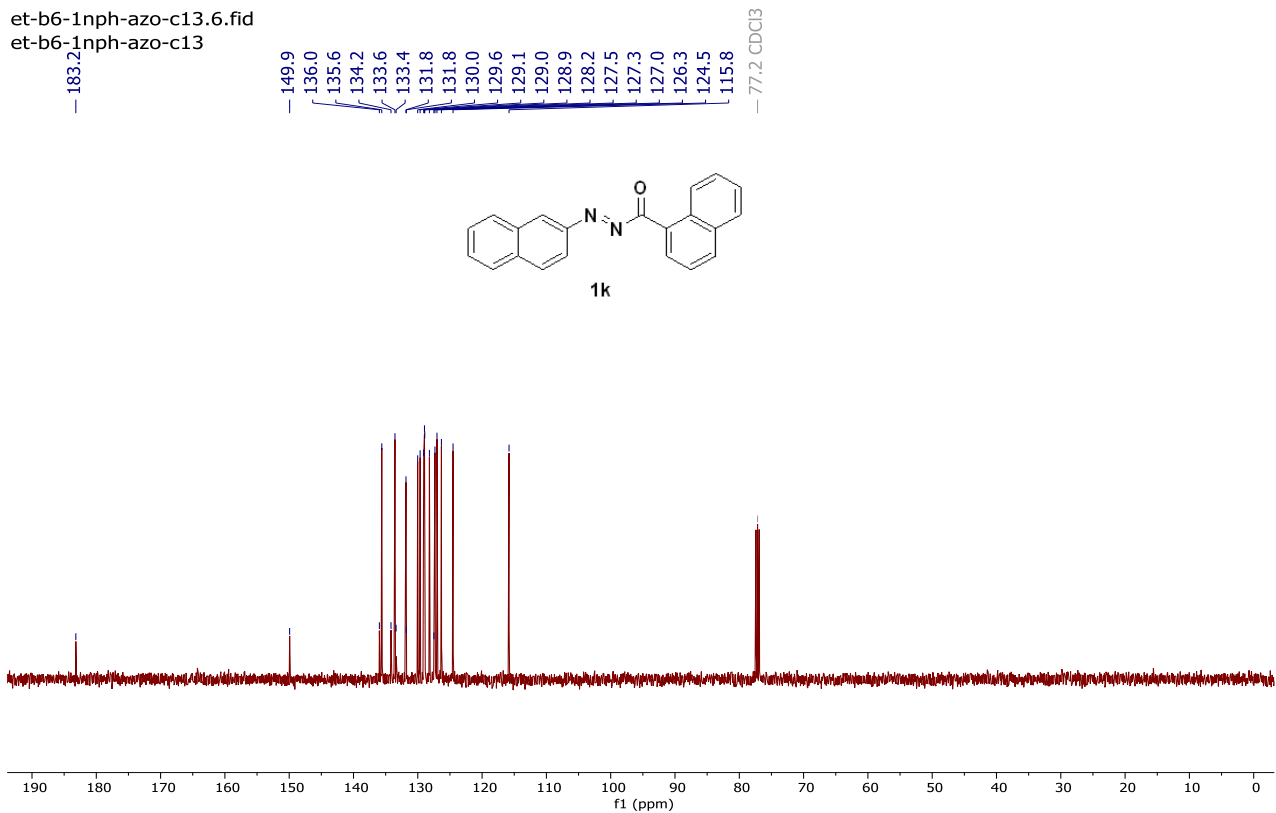
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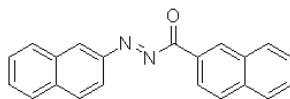
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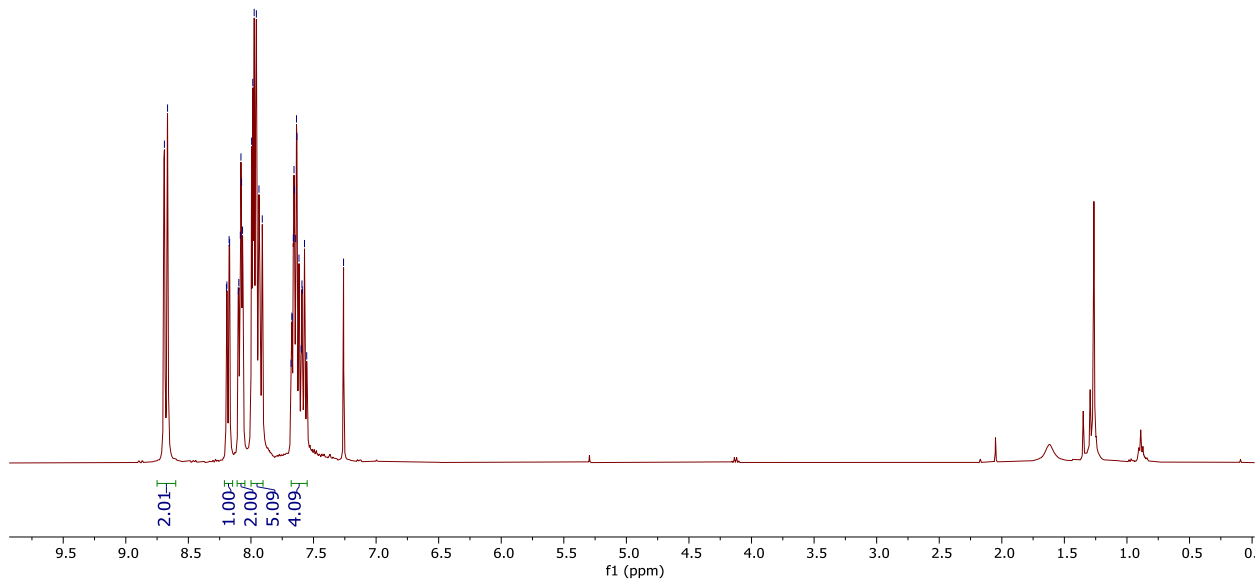


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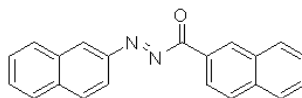


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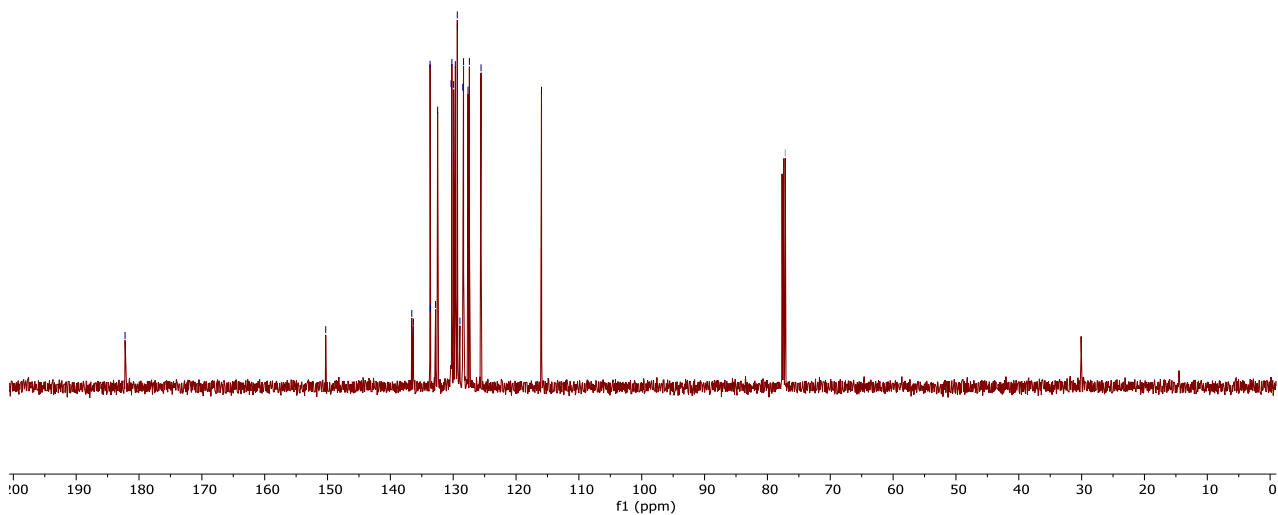


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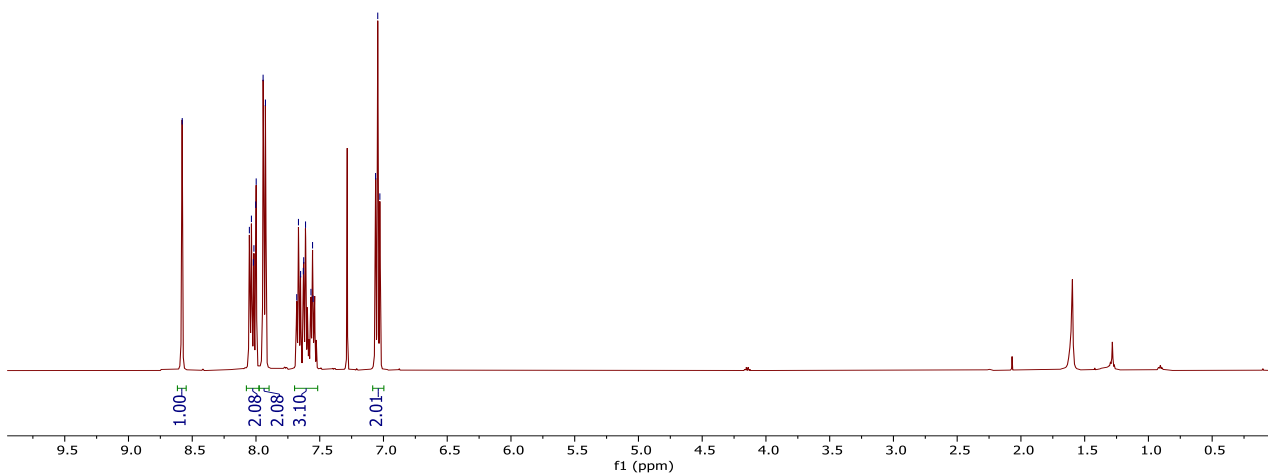
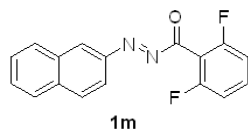
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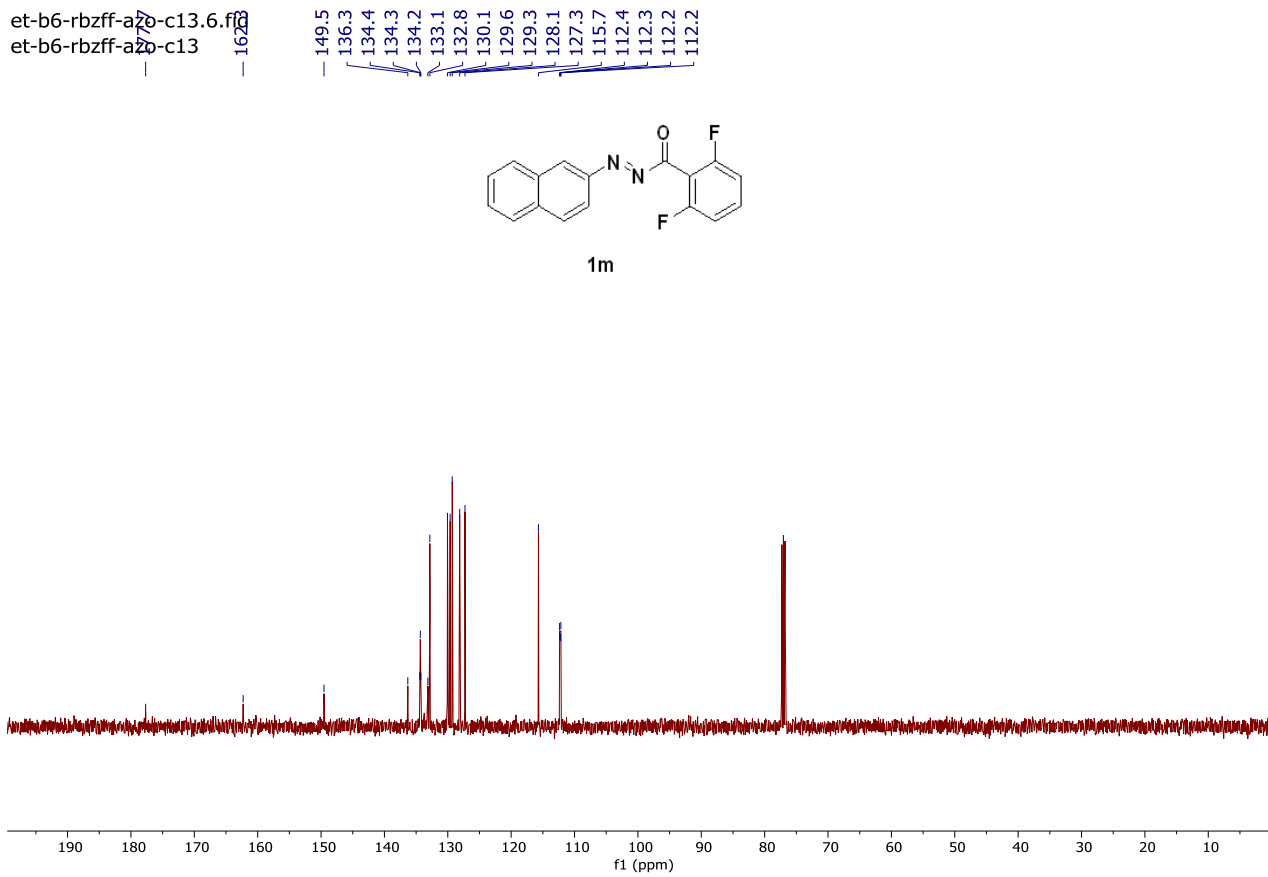
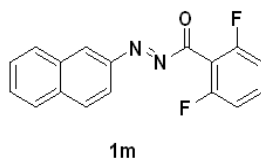
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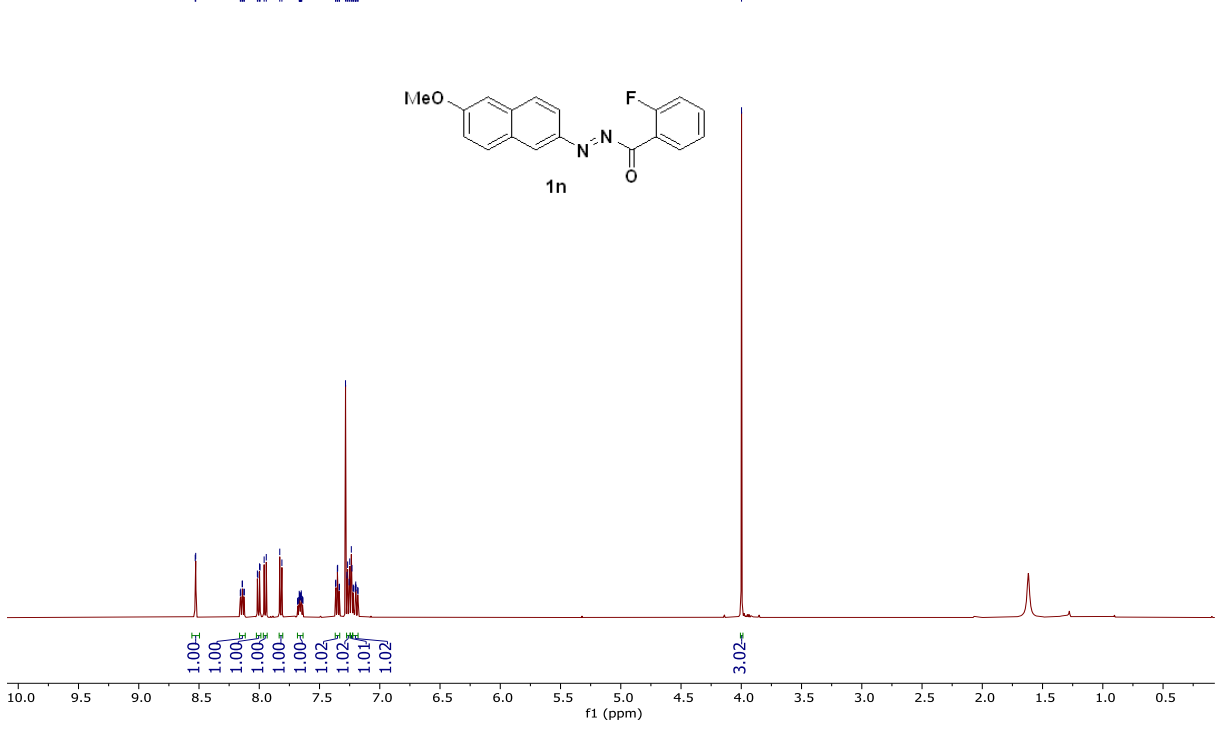
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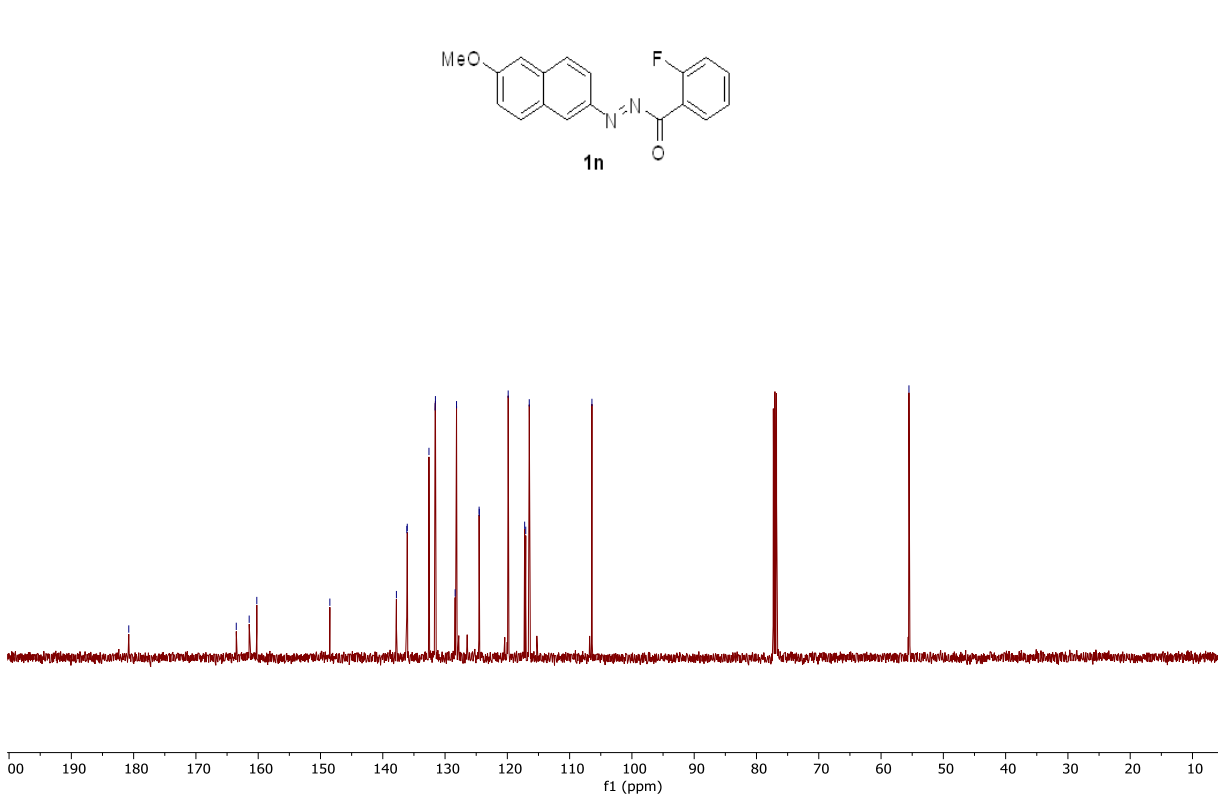
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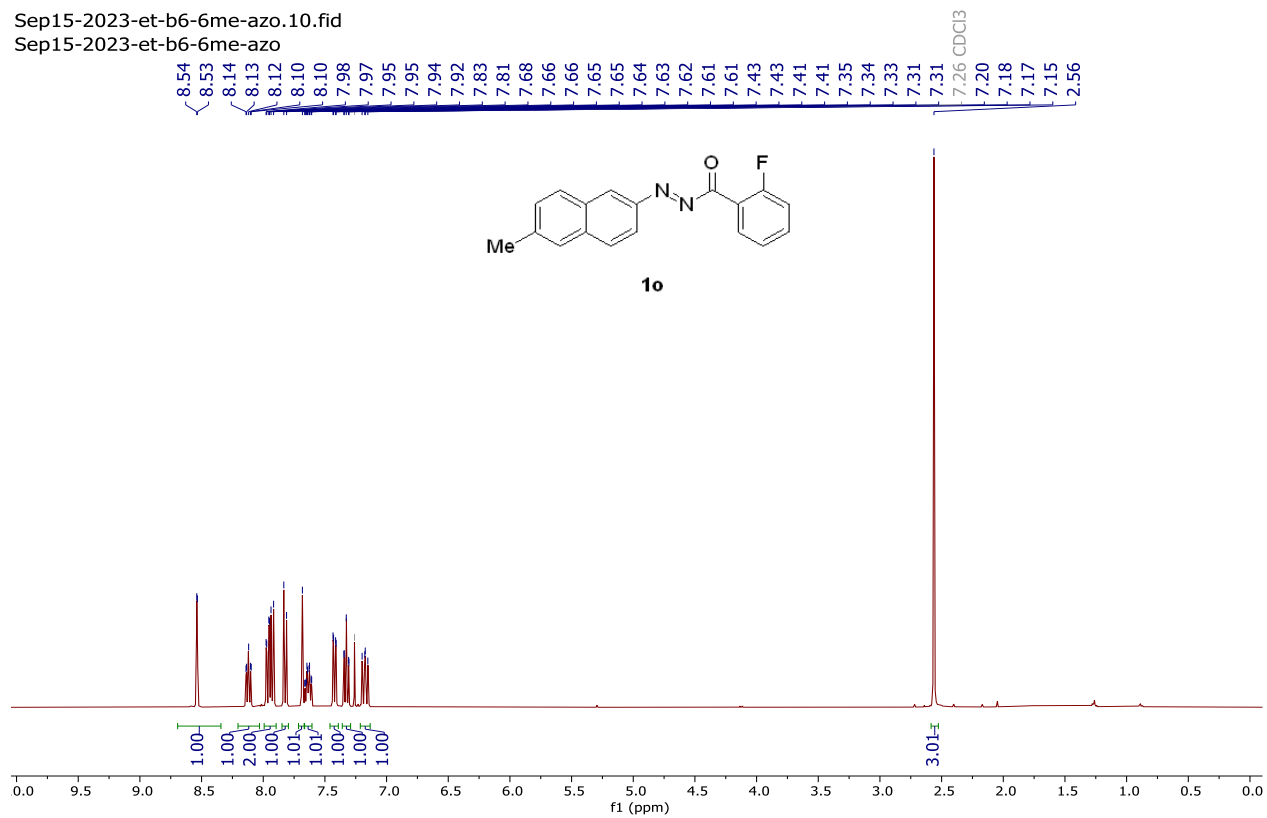


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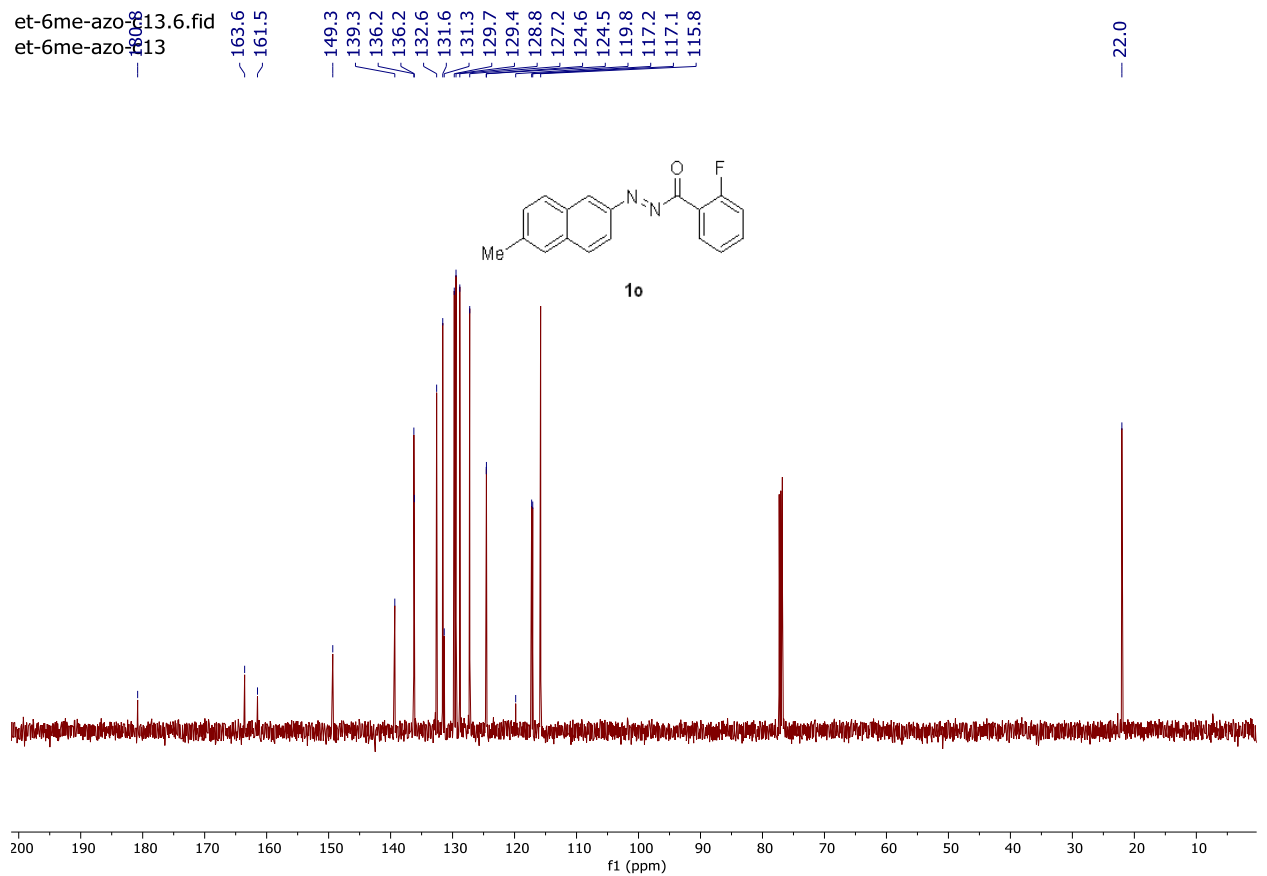
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Sep15-2023-et-b6-6me-azo

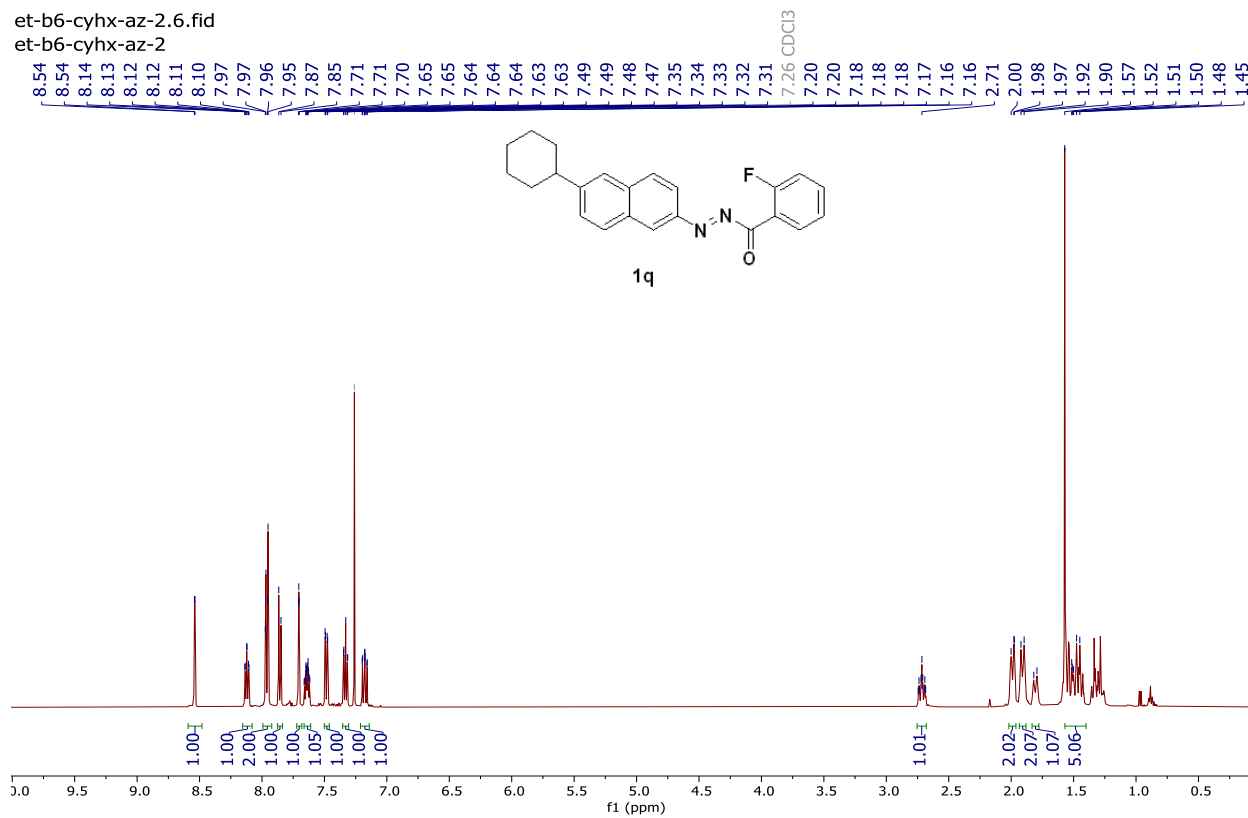


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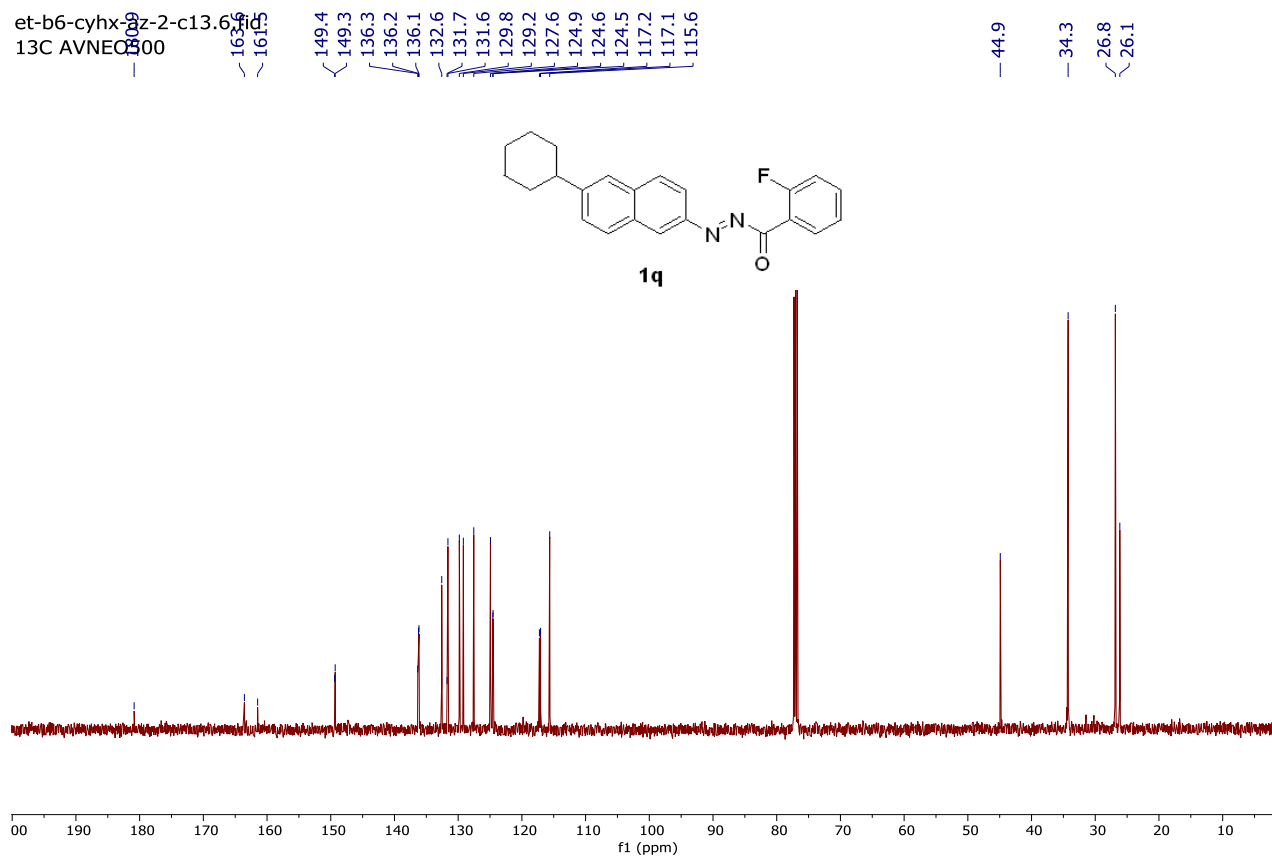
et-6me-azo 13



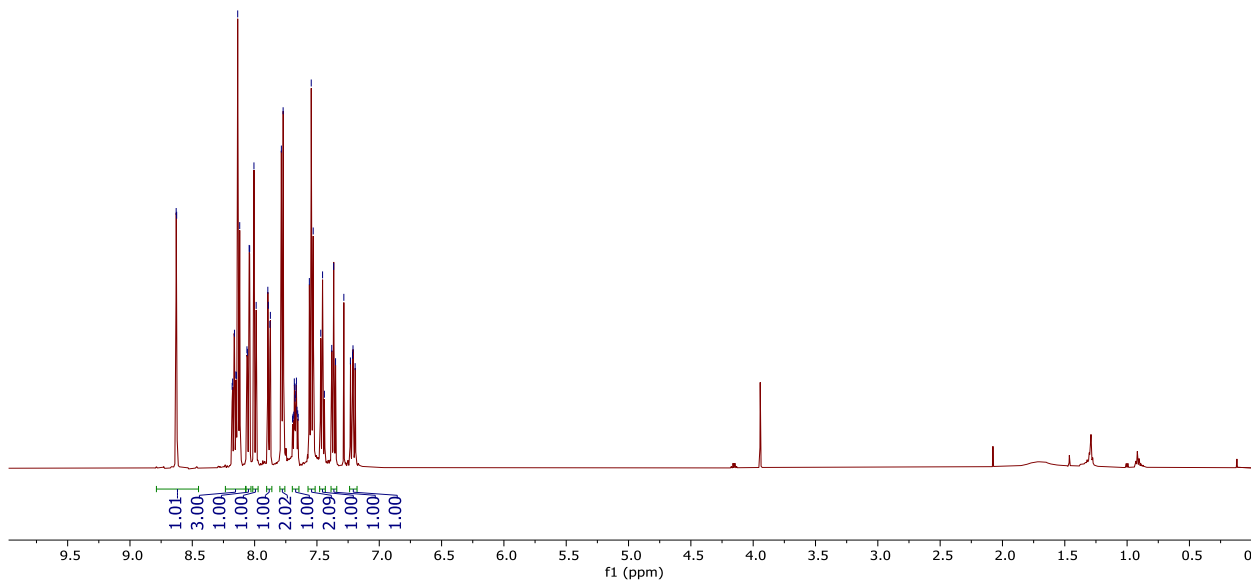
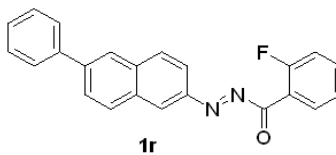
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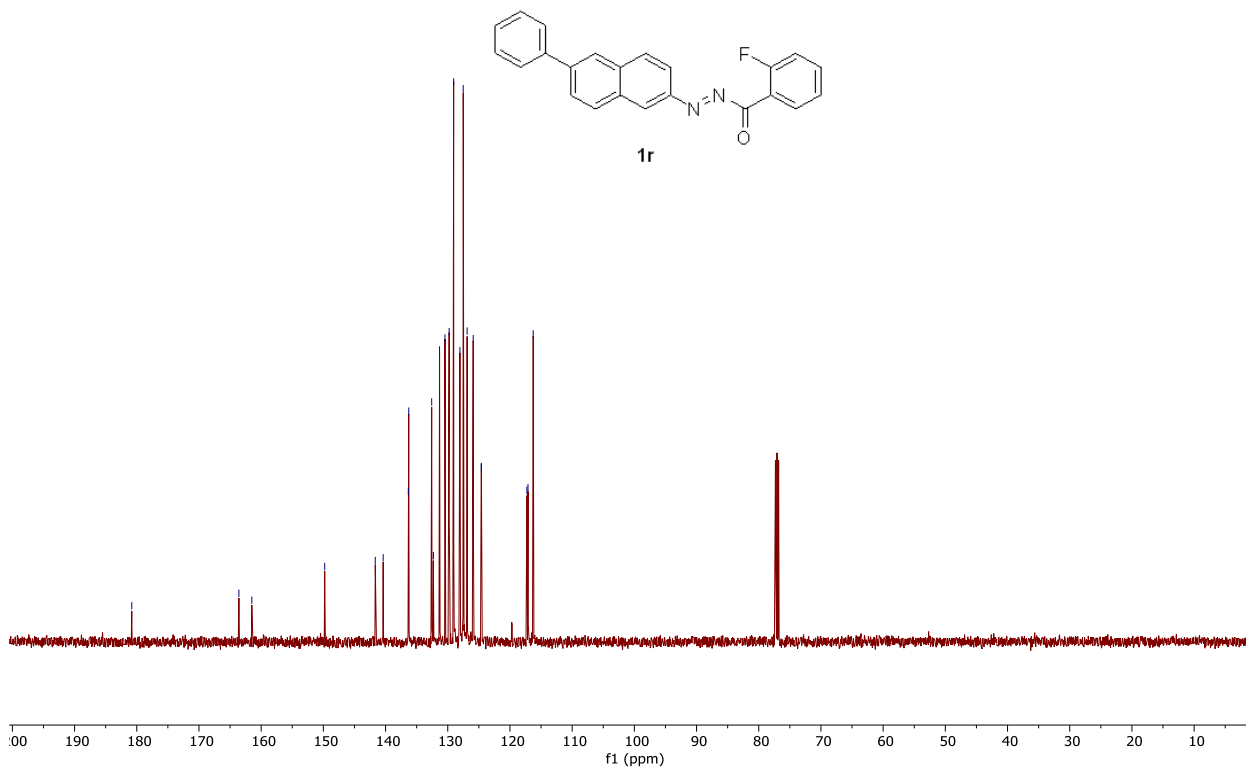
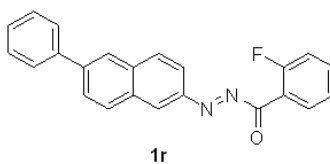
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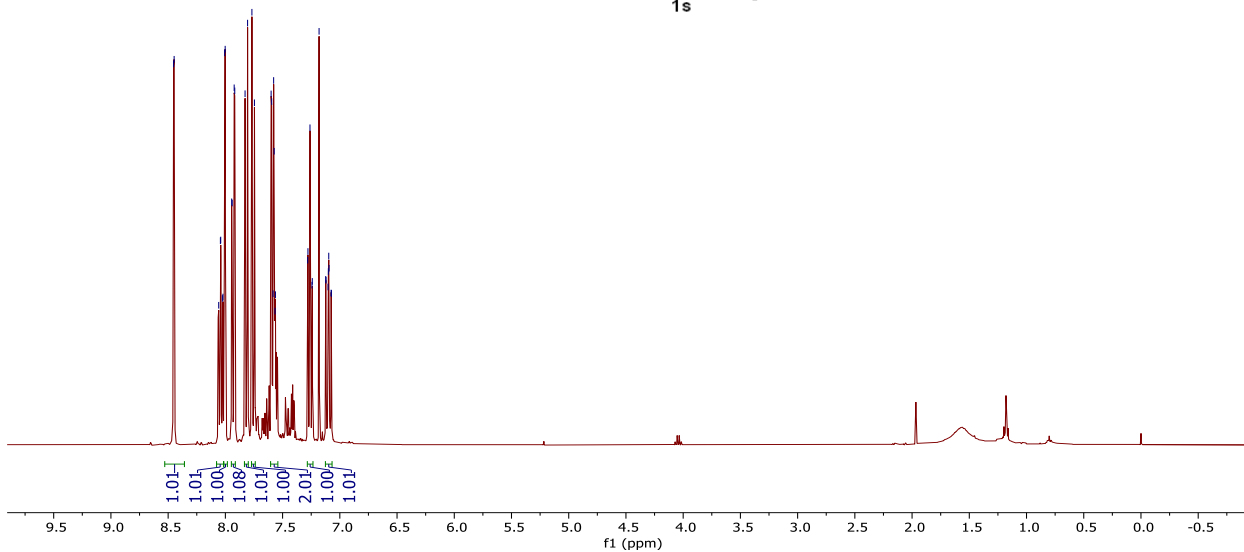
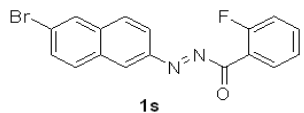
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et-b6-6ph-azo 8.61
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8.04
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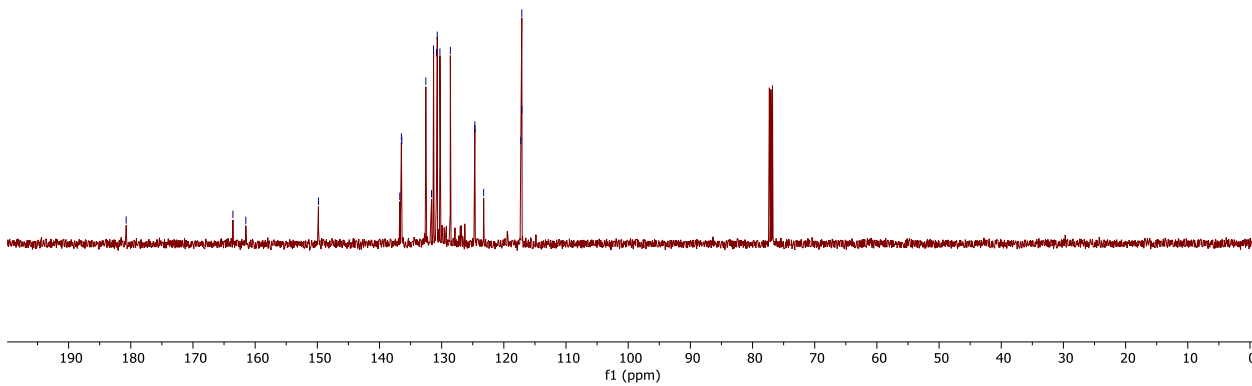
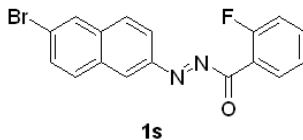
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et-b6-6ph-azo-c13 141.7
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136.3
132.6
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131.3
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127.5
126.9
125.9
124.6
124.6
117.3
117.1
116.3



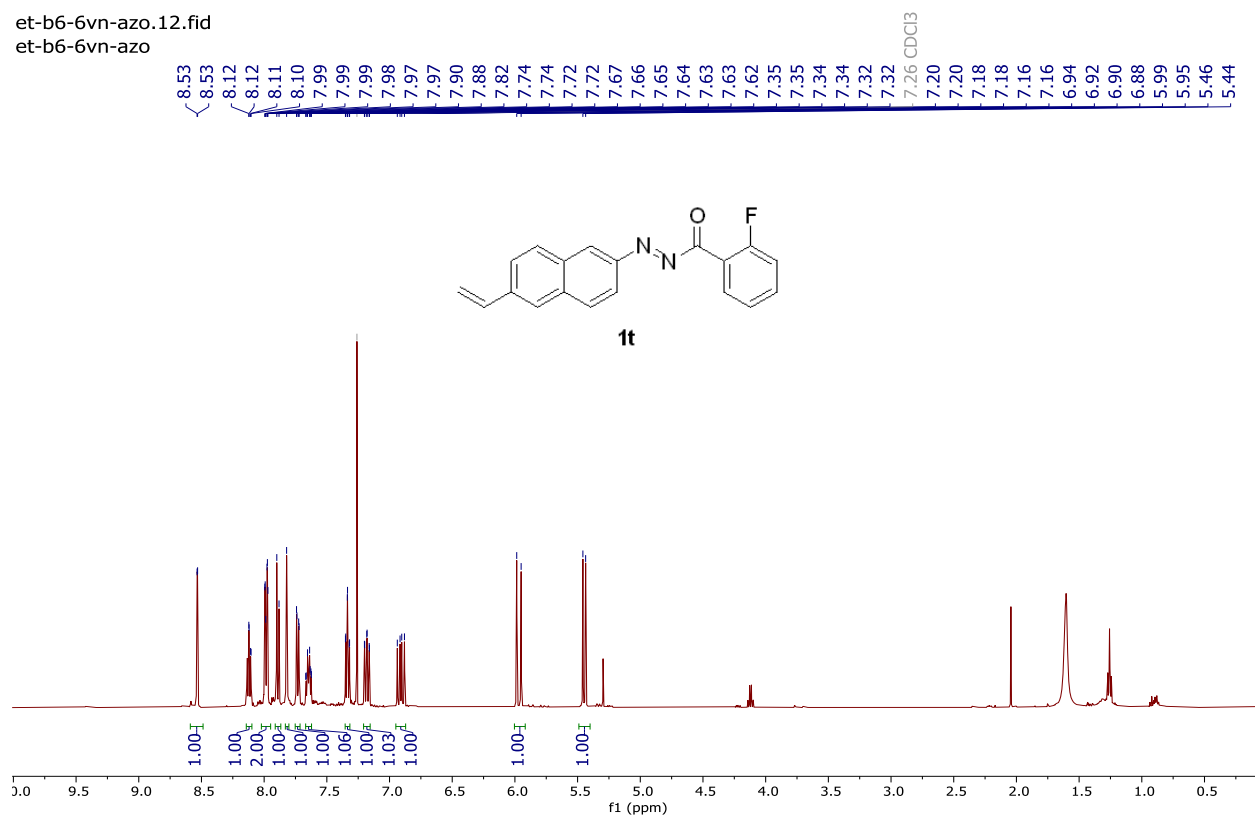
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7.07



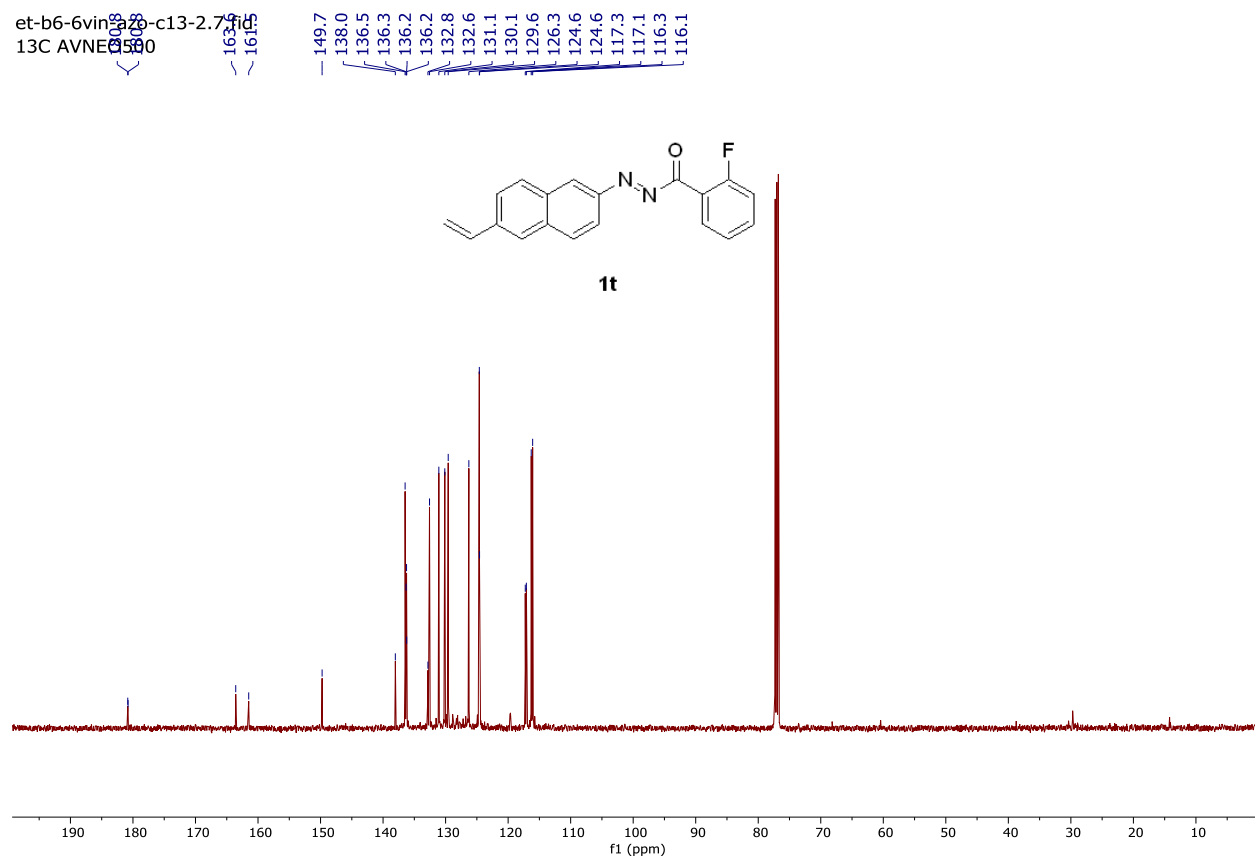
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117.1
117.1



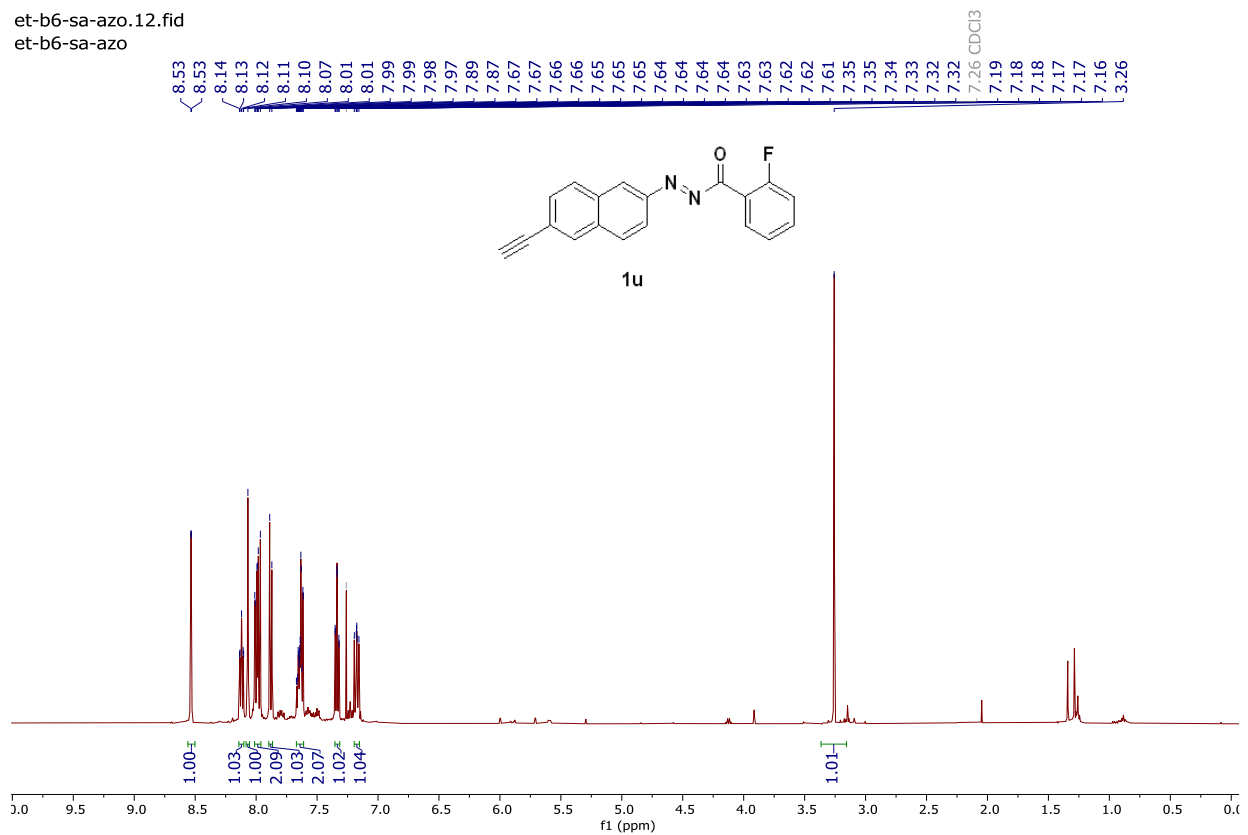
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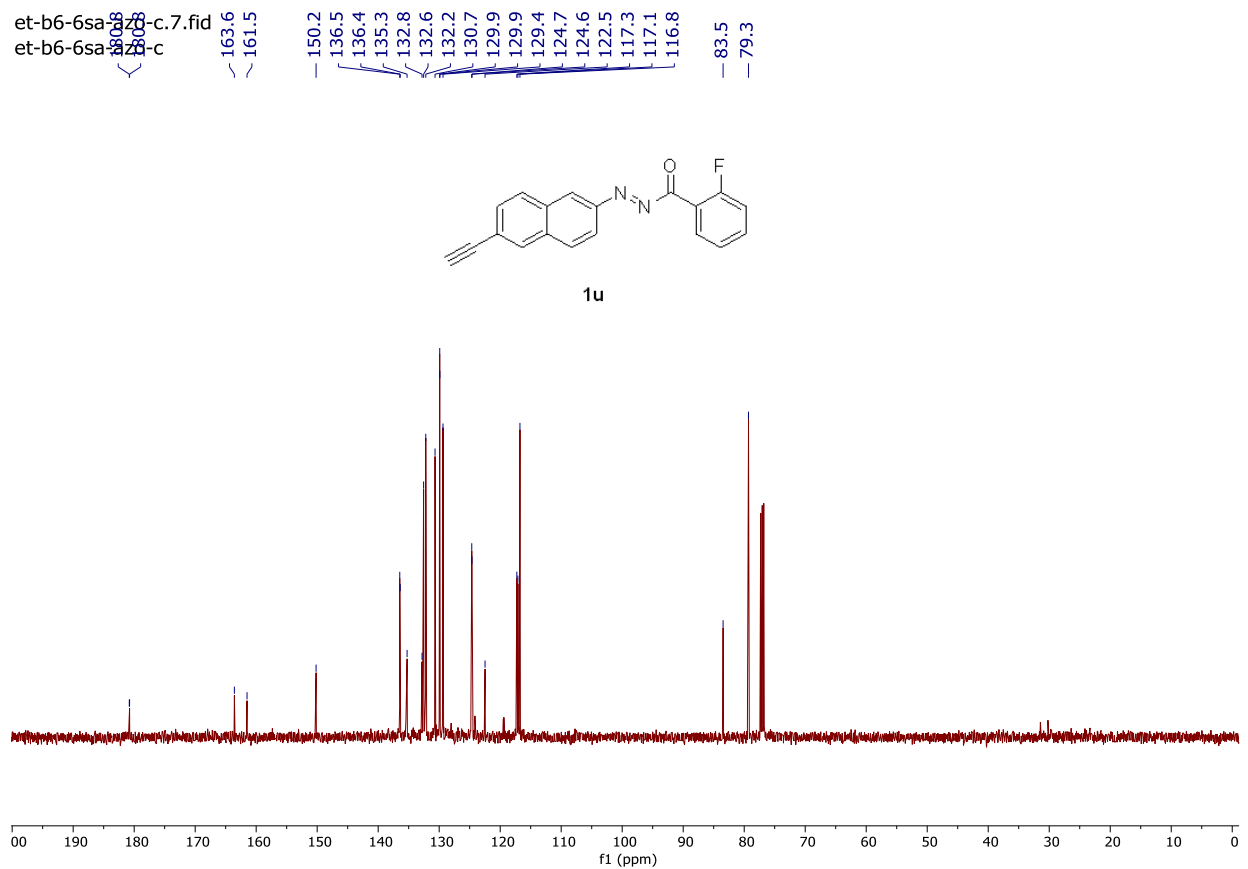
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13C AVNE 1500



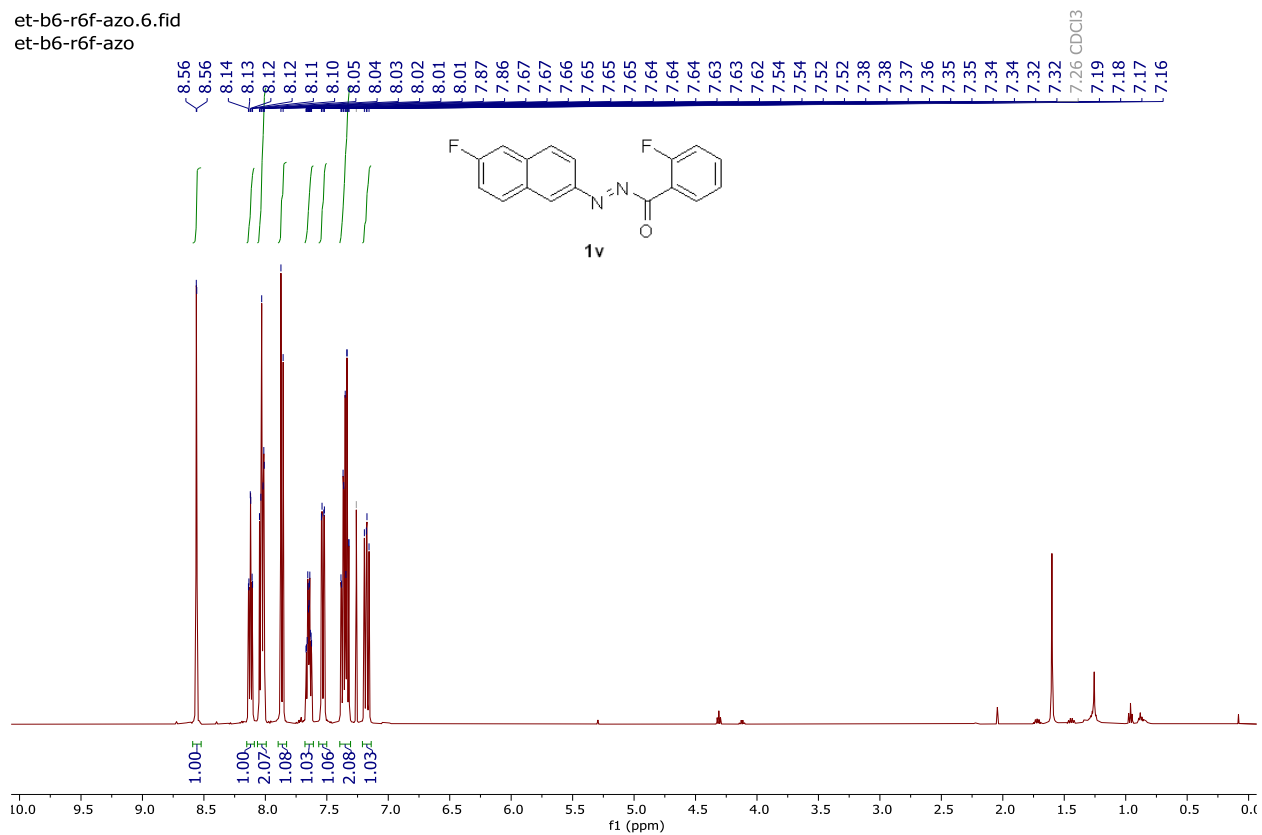
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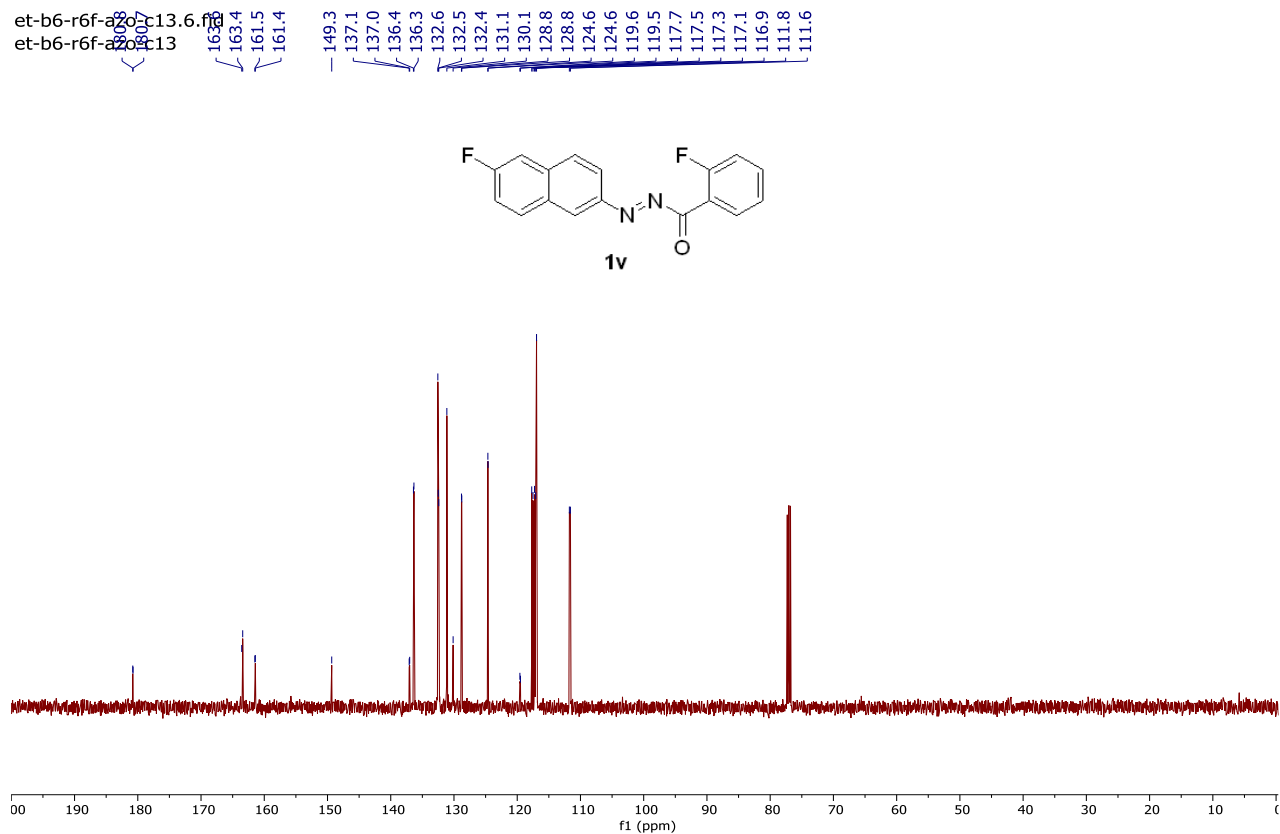
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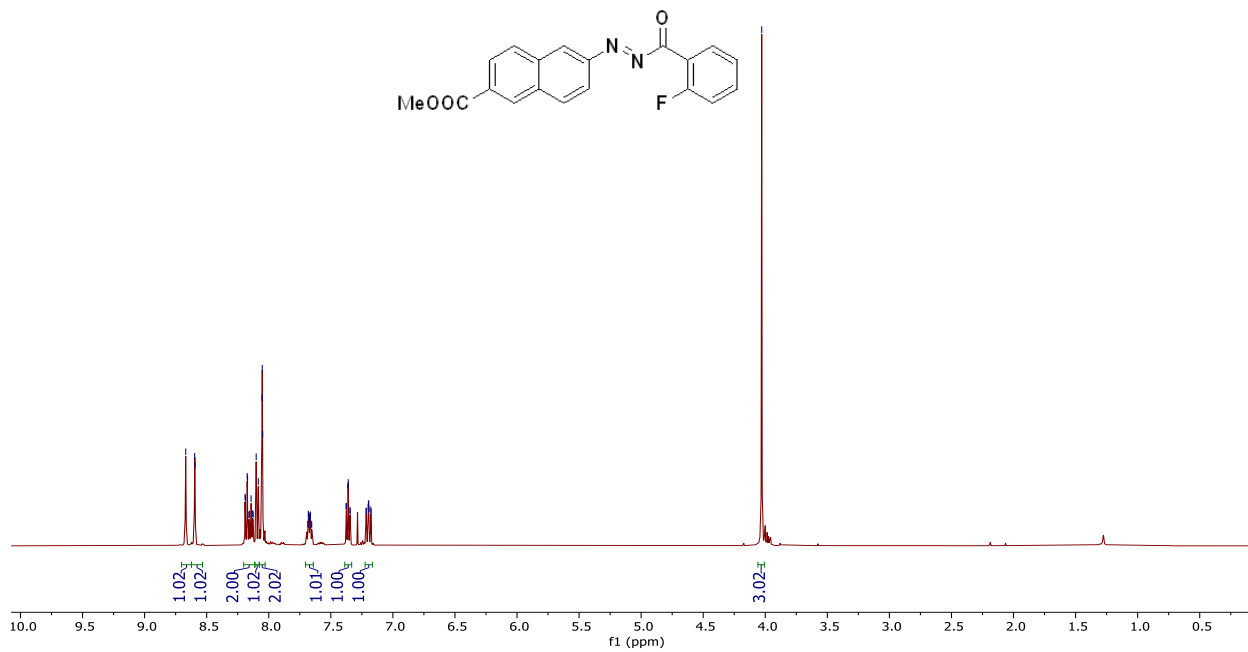
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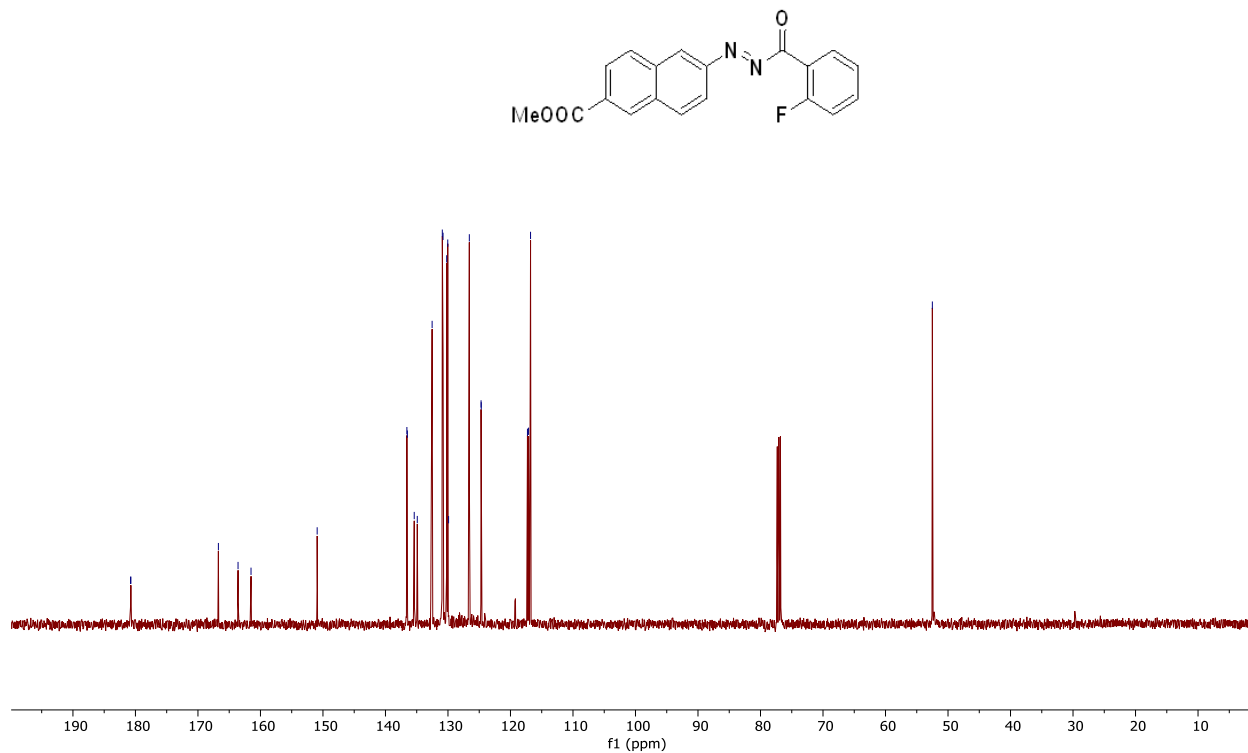
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et-b6-4co2me-azo-x-16
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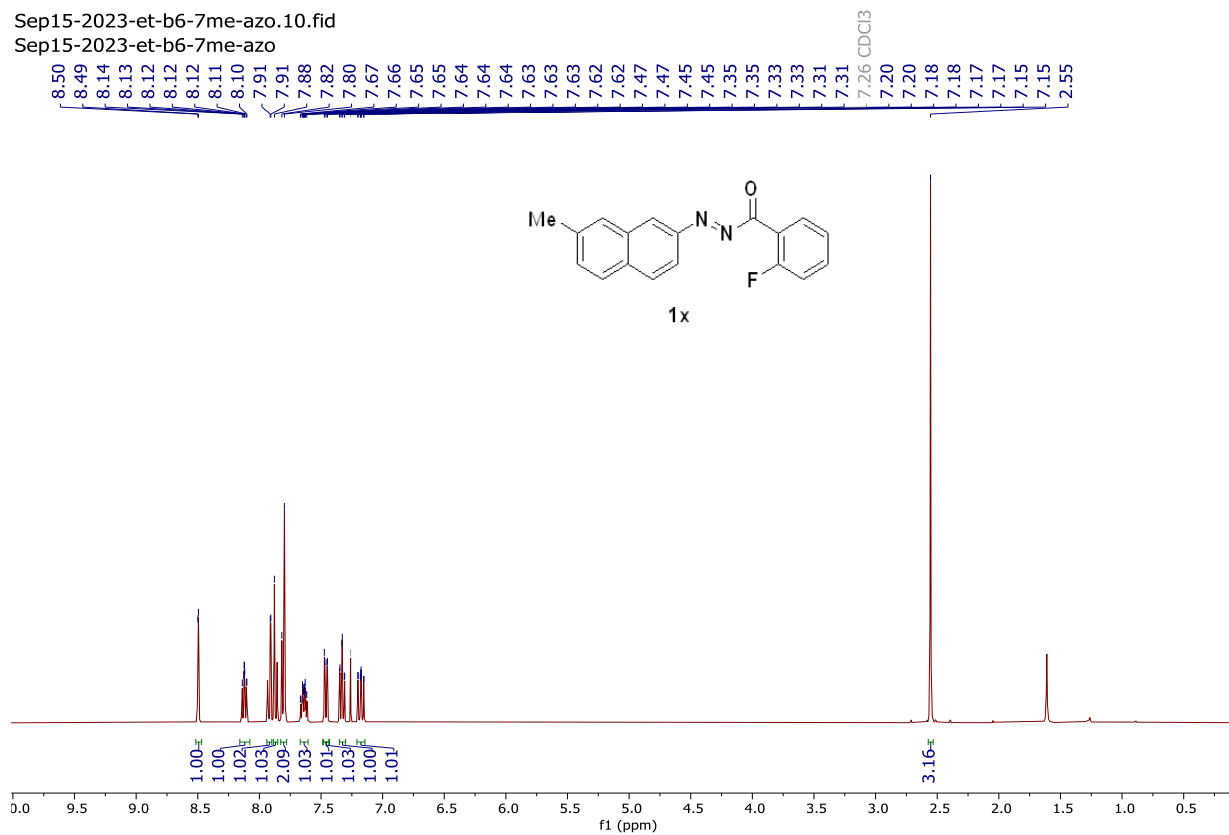


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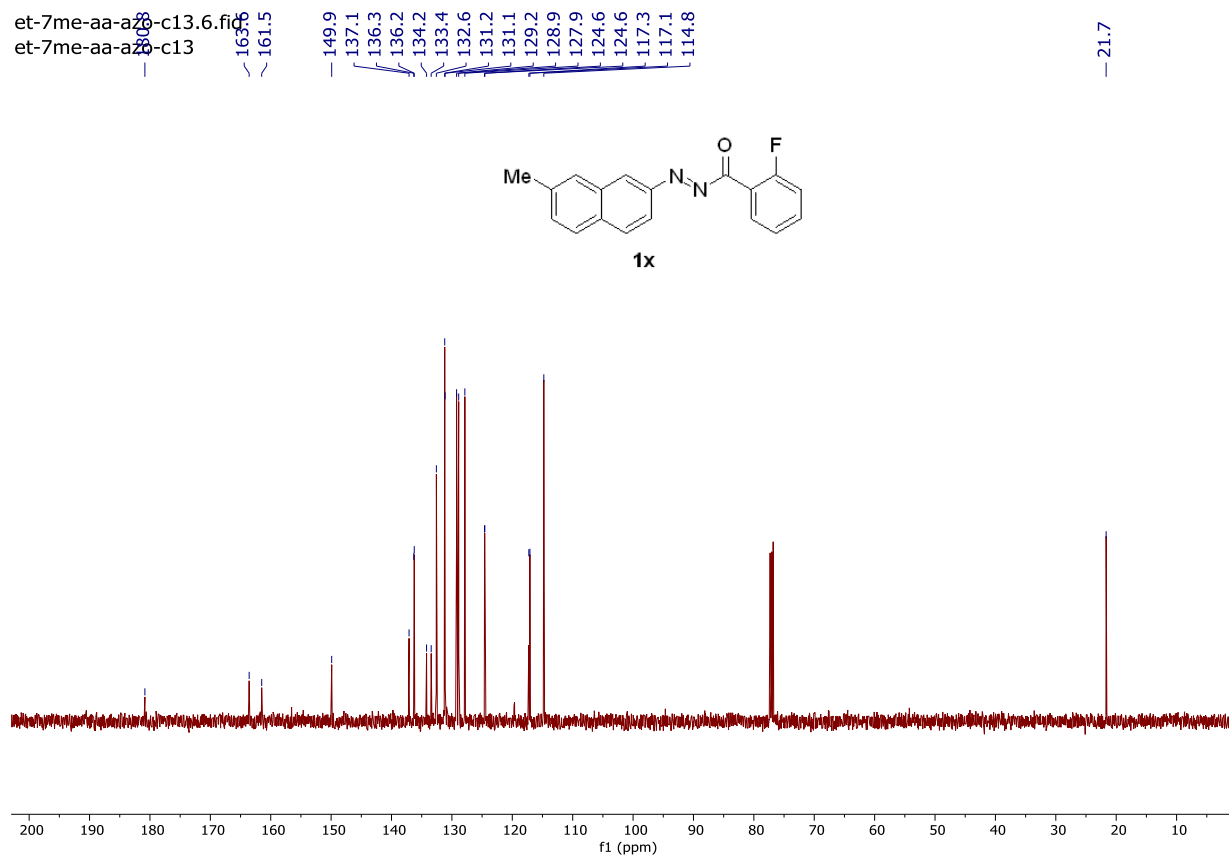
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Sep15-2023-et-b6-7me-azo

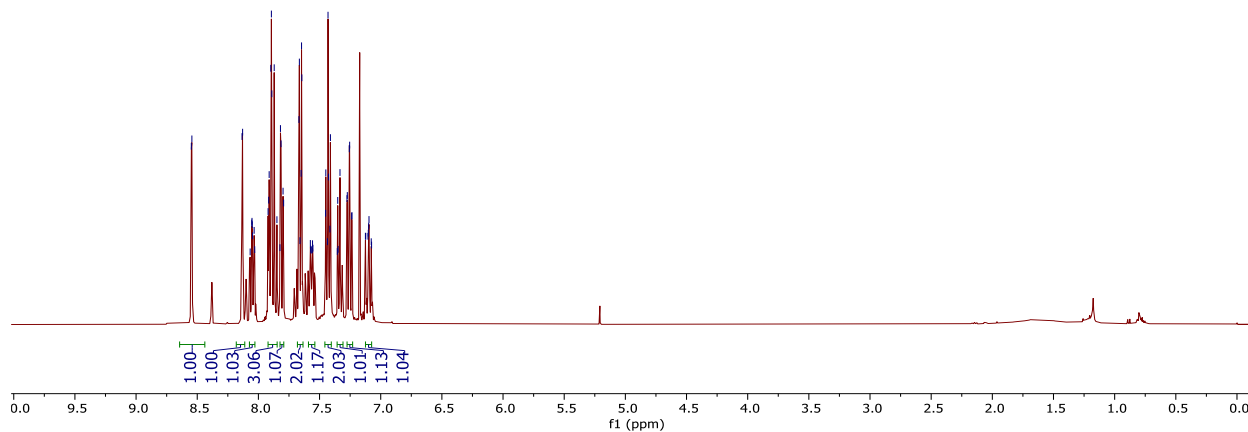
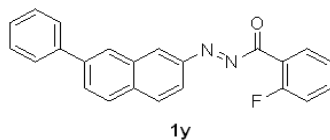


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et-7me-aa-azo-c13

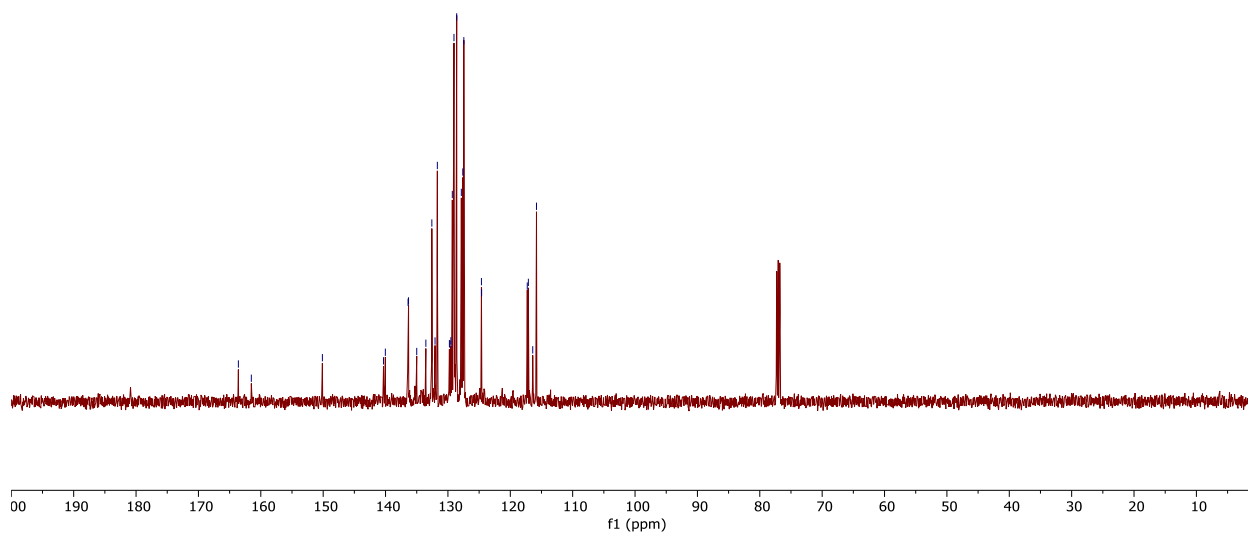
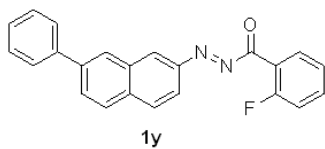


Sep 15 2023 et-b6-7ph-az-c13.6.13
 Sep 15 2023 et-b6-7ph-az

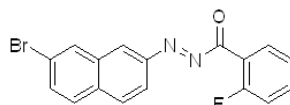


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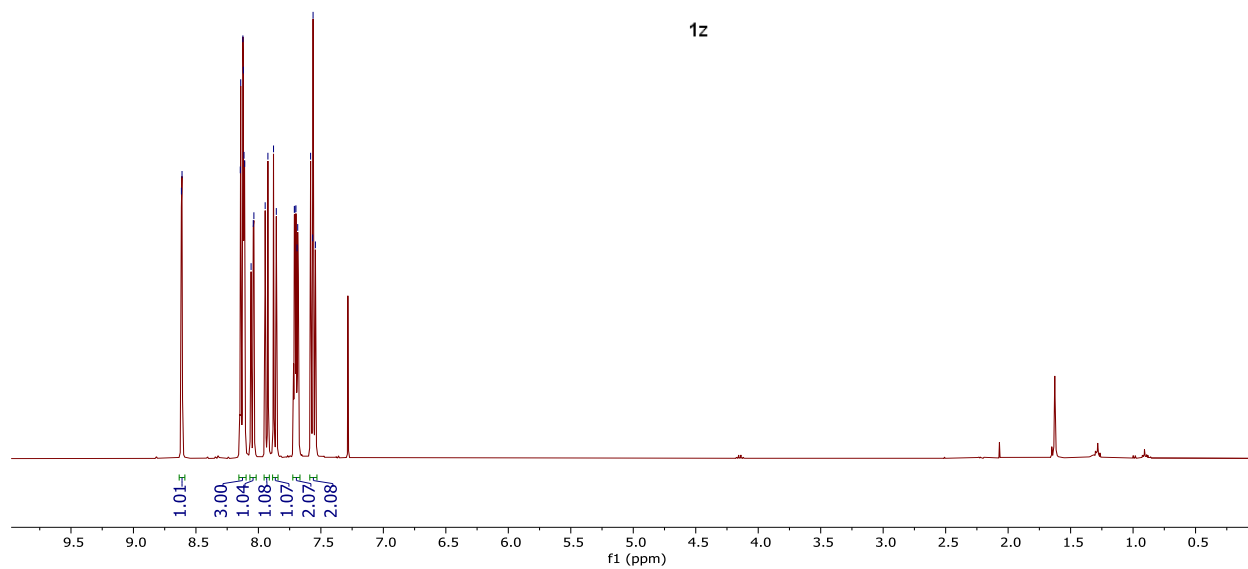
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 116.4
 115.8



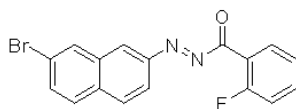
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Dec25-2023-et-b6-7br-a2o-c13



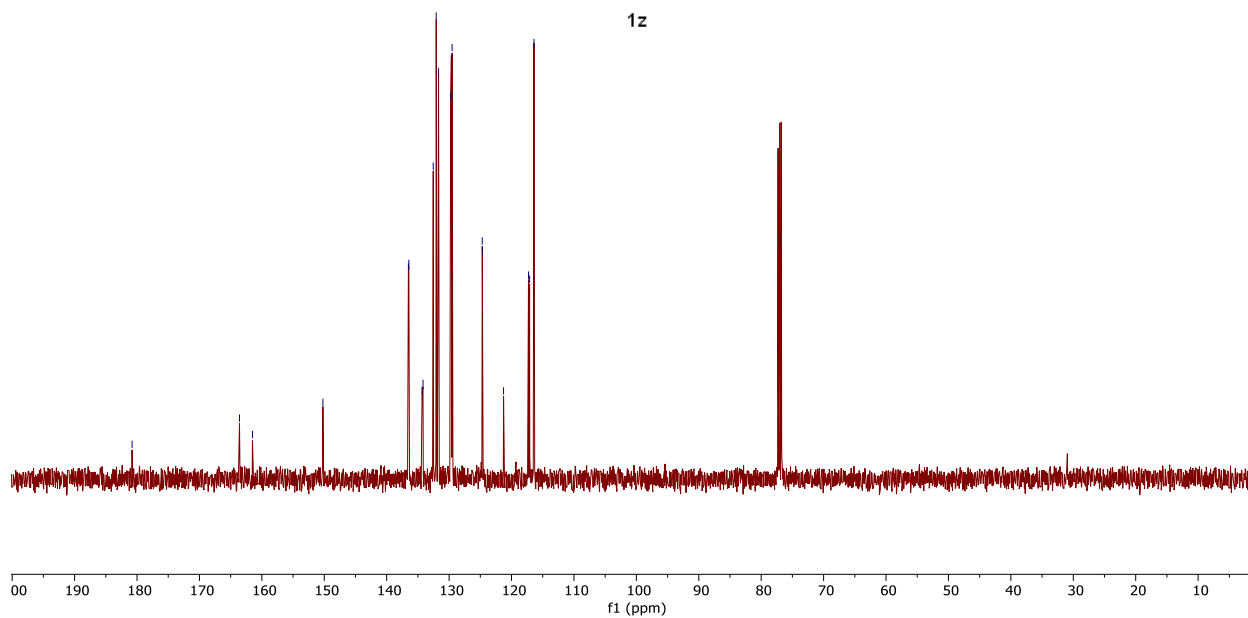
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et-b6-7br-a2o-c13.6
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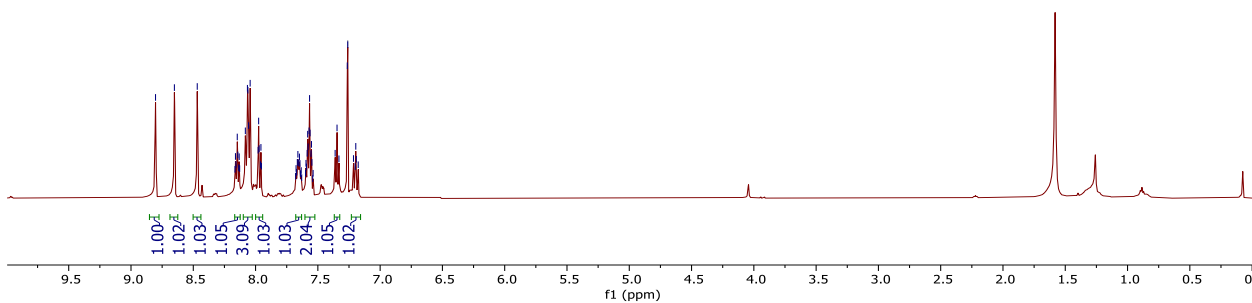
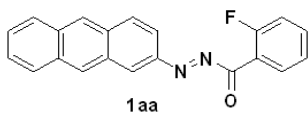


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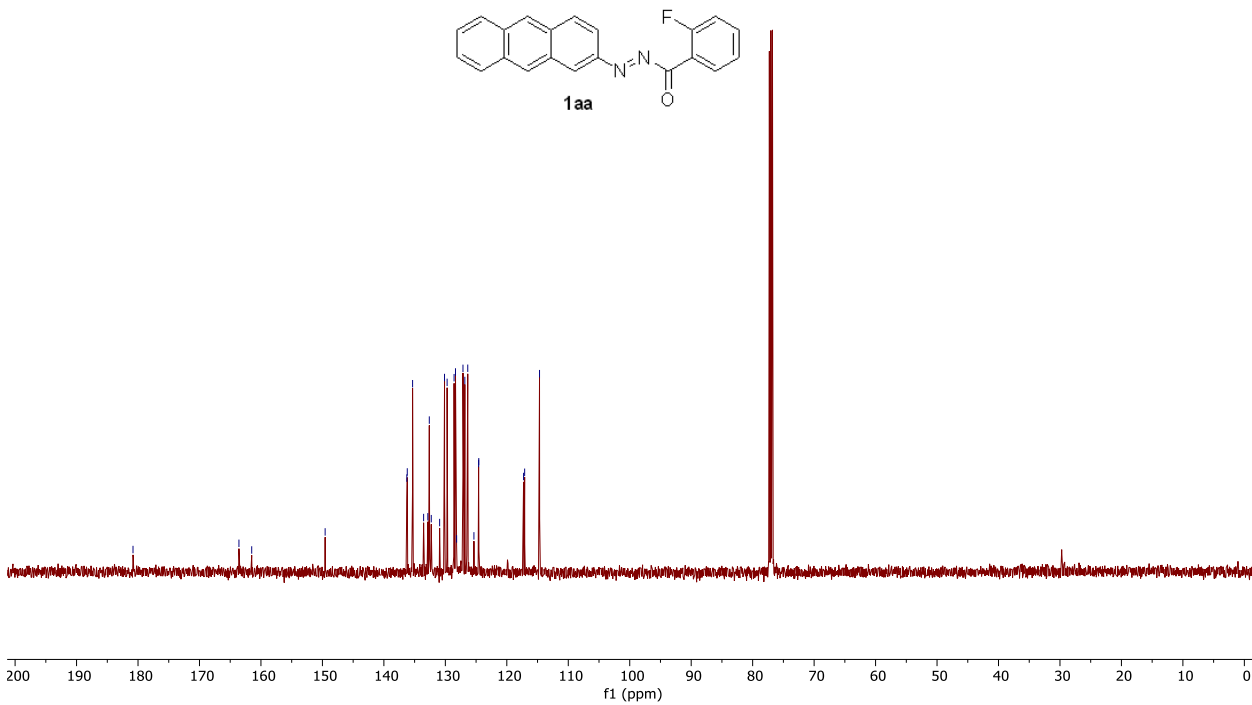
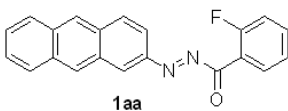
et-b6-anc-az.7.fid
et-b6-anc-az

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7.18



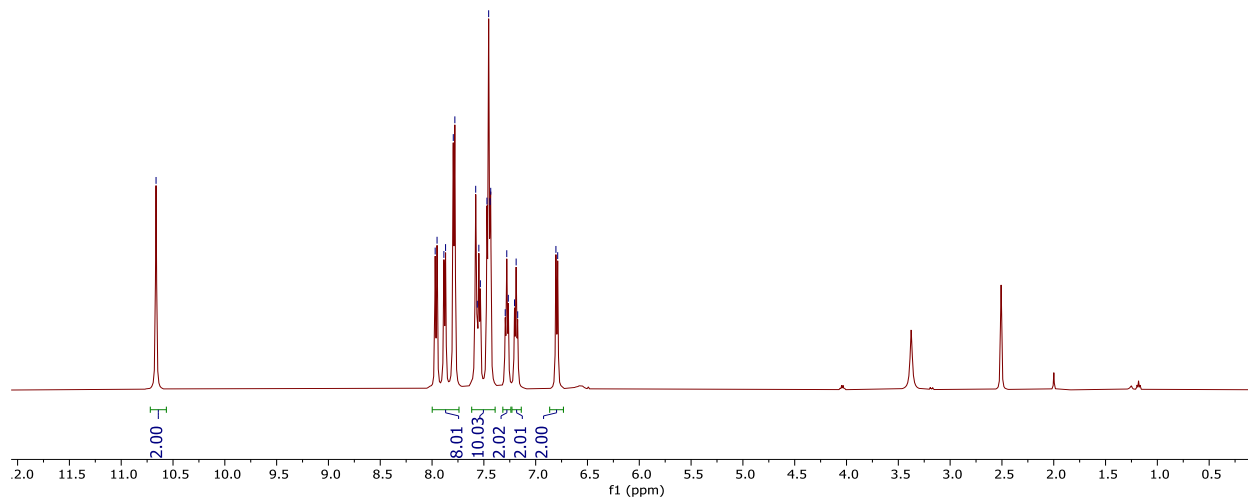
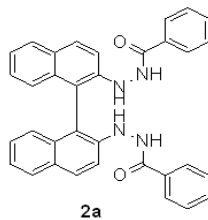
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et-b6-anc-az.c13

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161.5
149.6
136.3
136.2
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133.5
132.8
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132.3
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129.7
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117.1
114.7



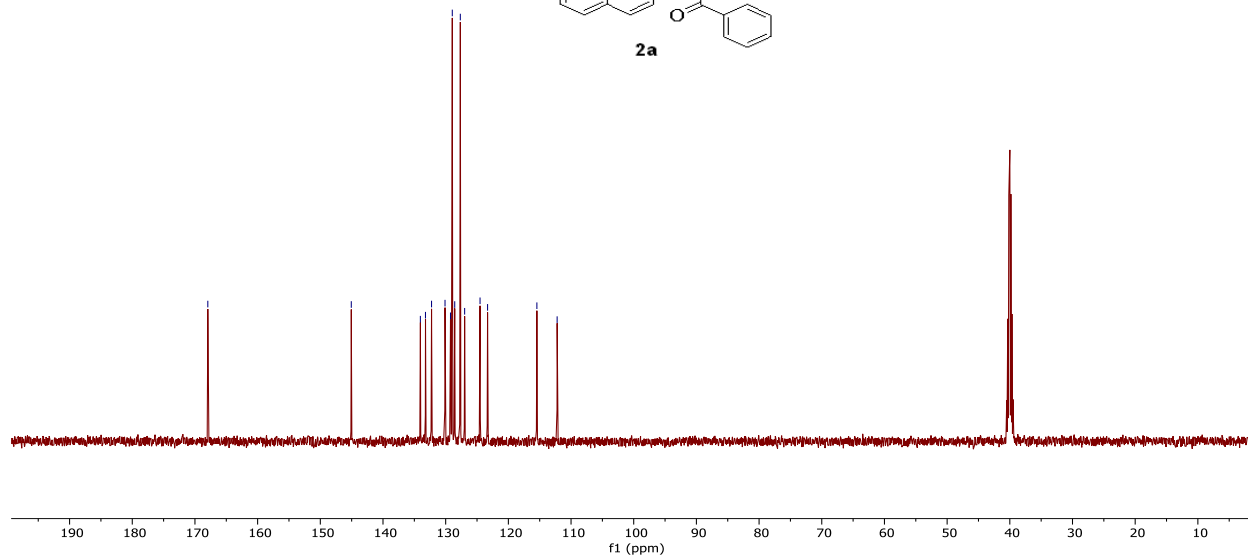
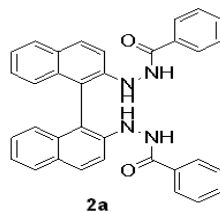
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et-b6-bz-bnm-13.6

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7.55
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6.79

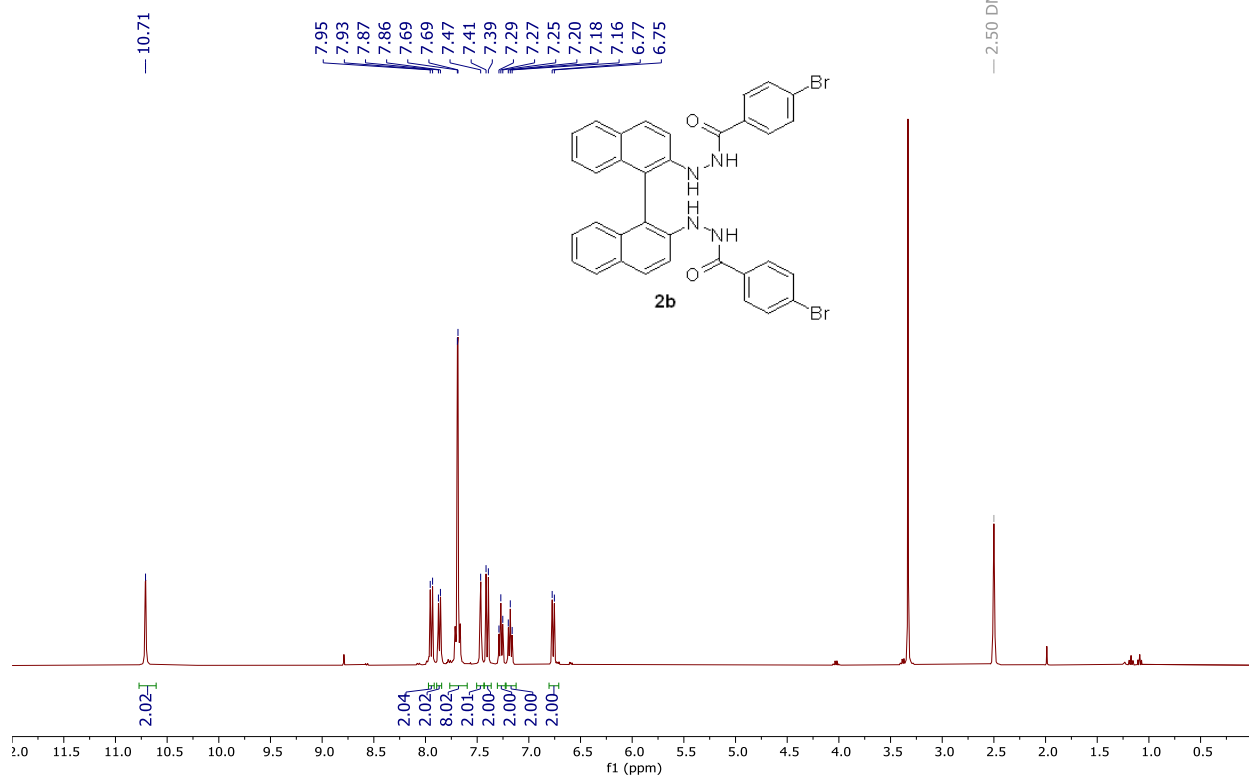


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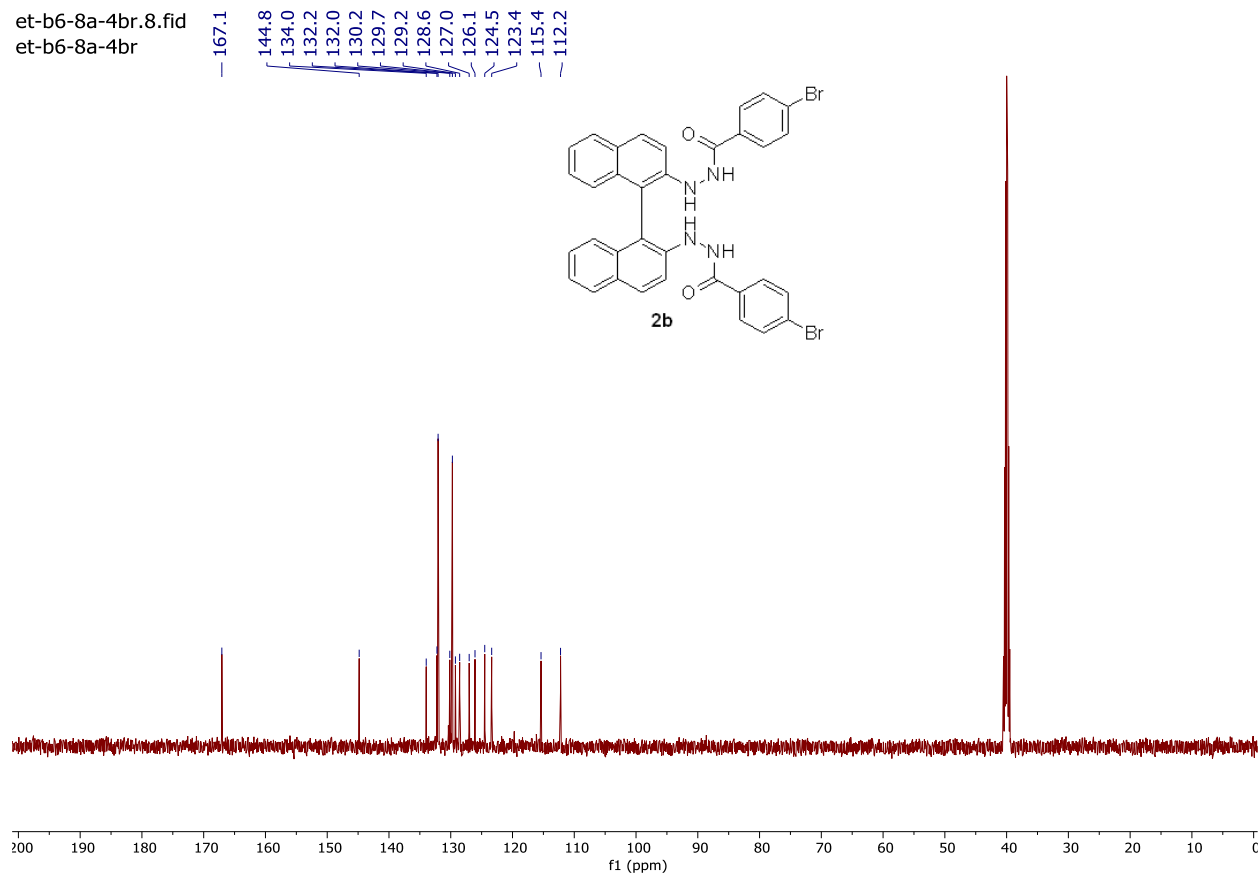
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127.7
127.0
124.5
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115.5
112.2



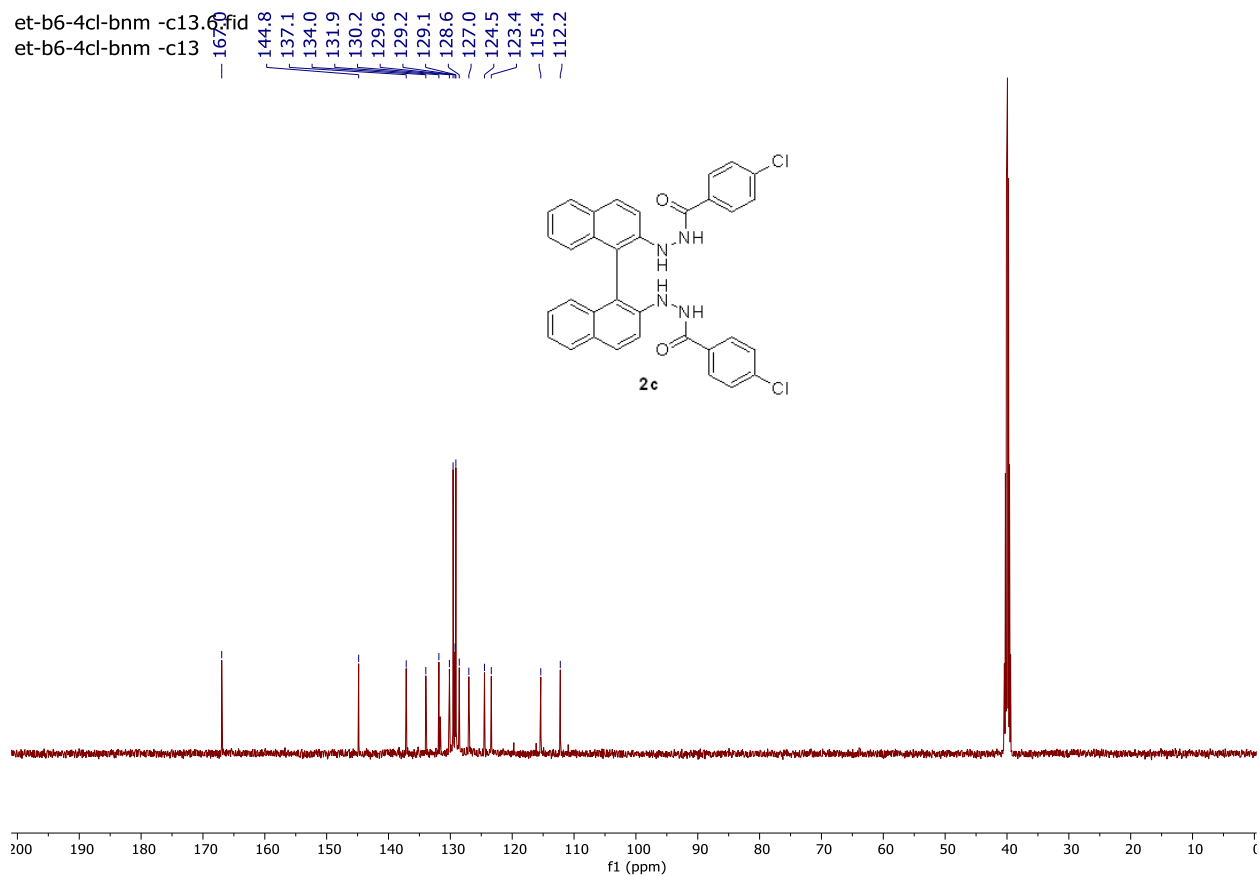
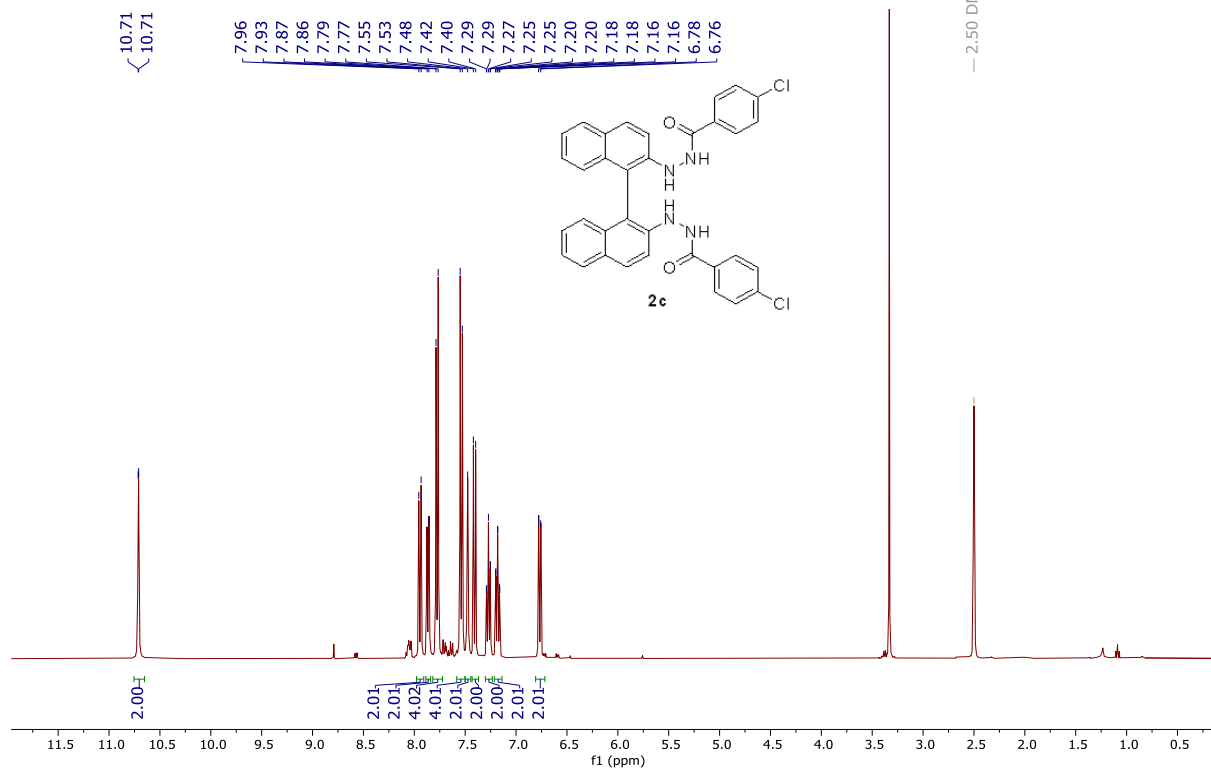
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May18-2023-et-b6-8a-4br



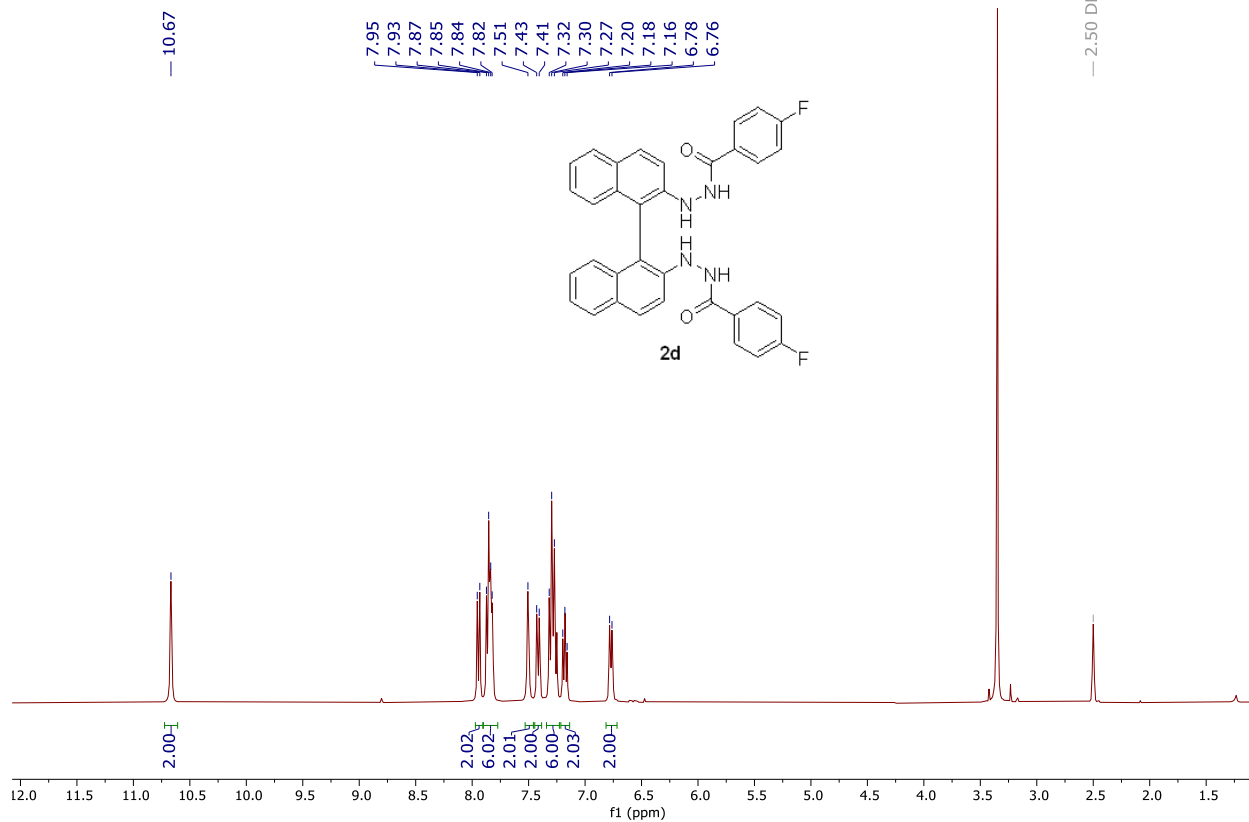
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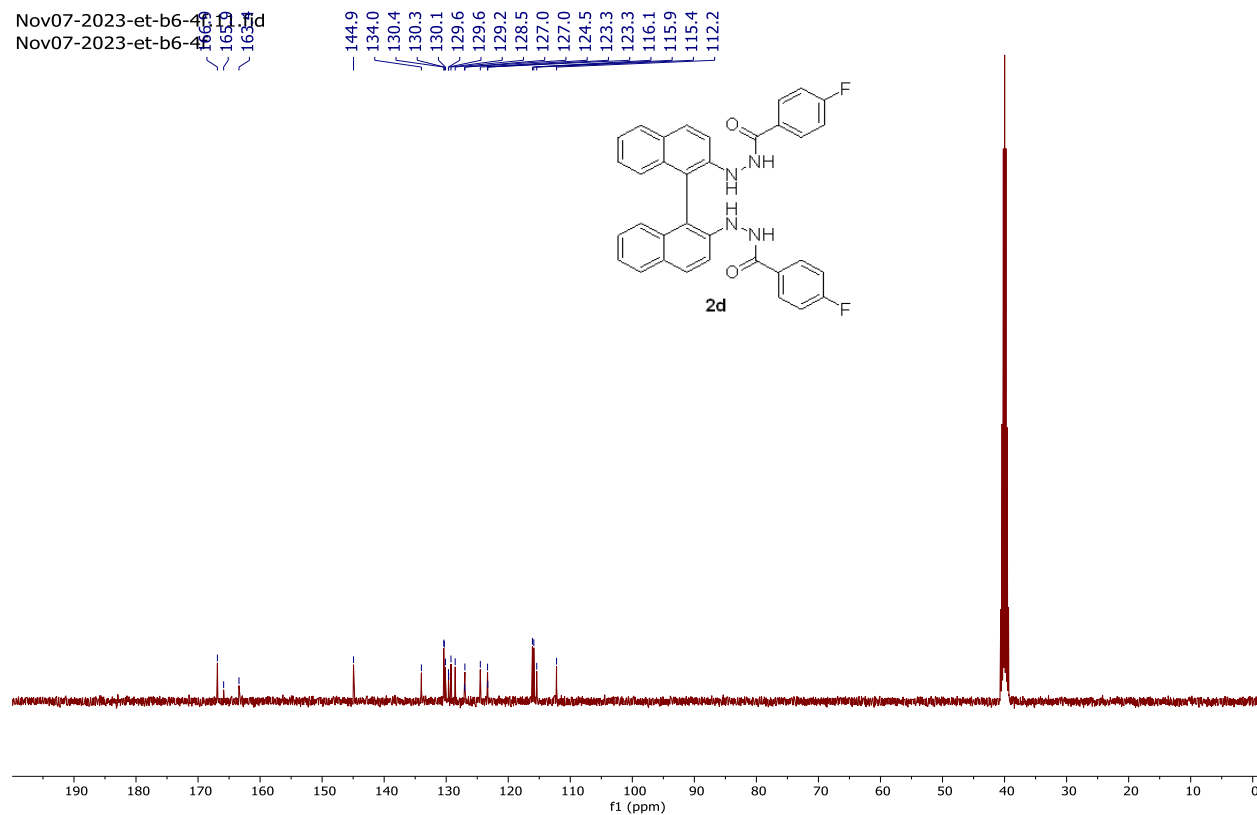
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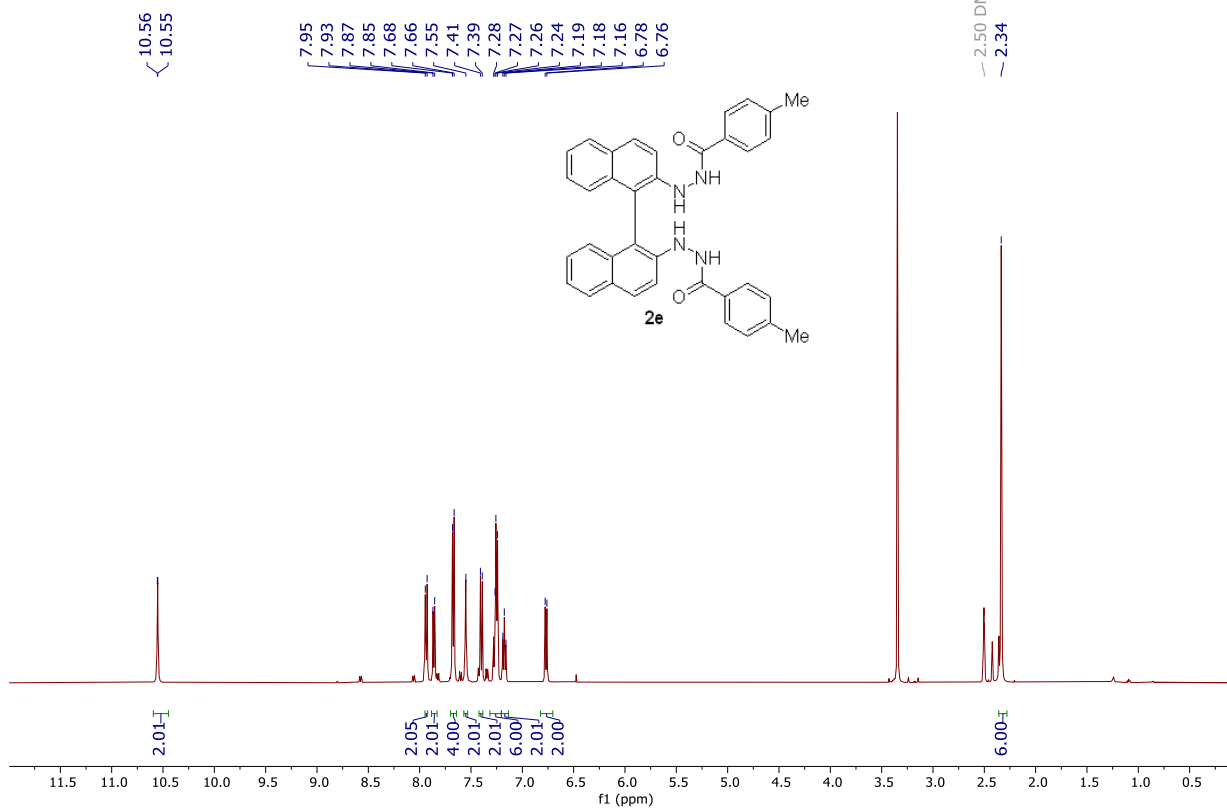
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Nov07-2023-et-b6-4f



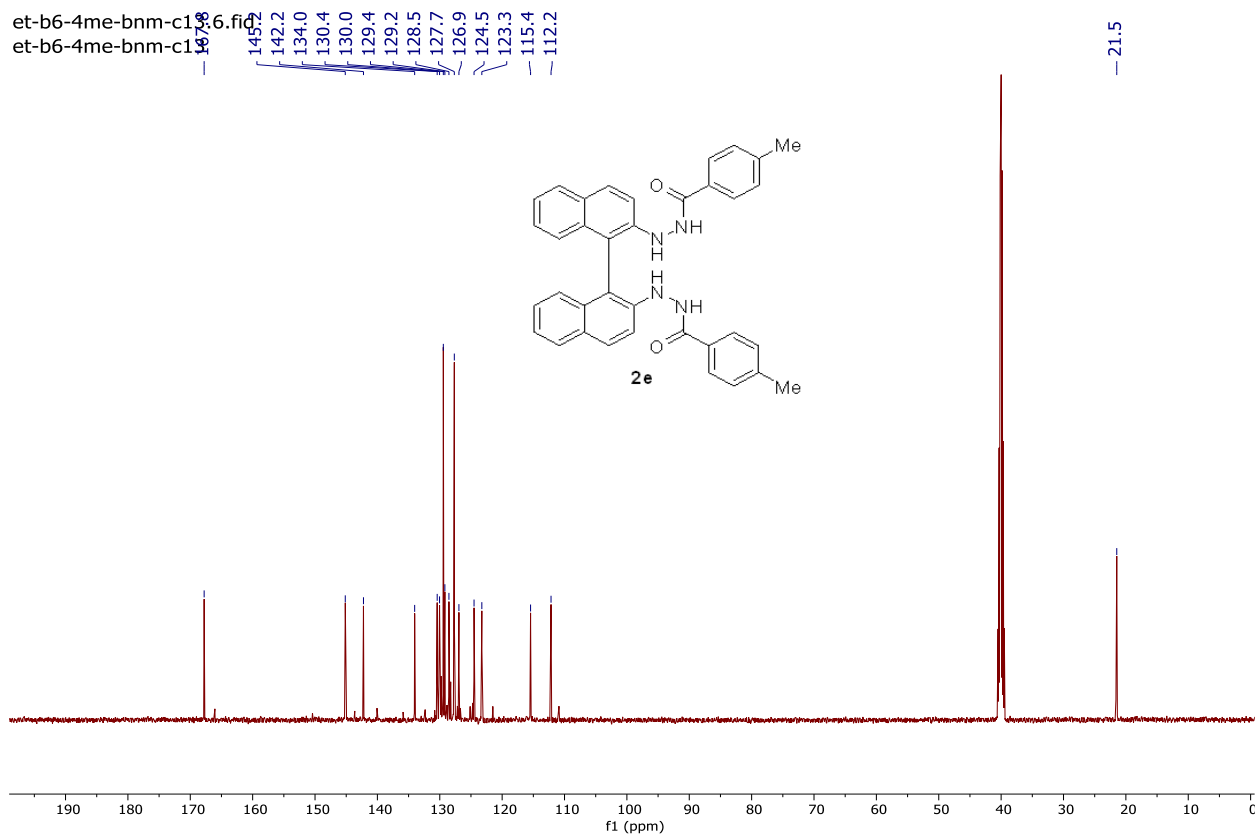
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Nov07-2023-et-b6-4f



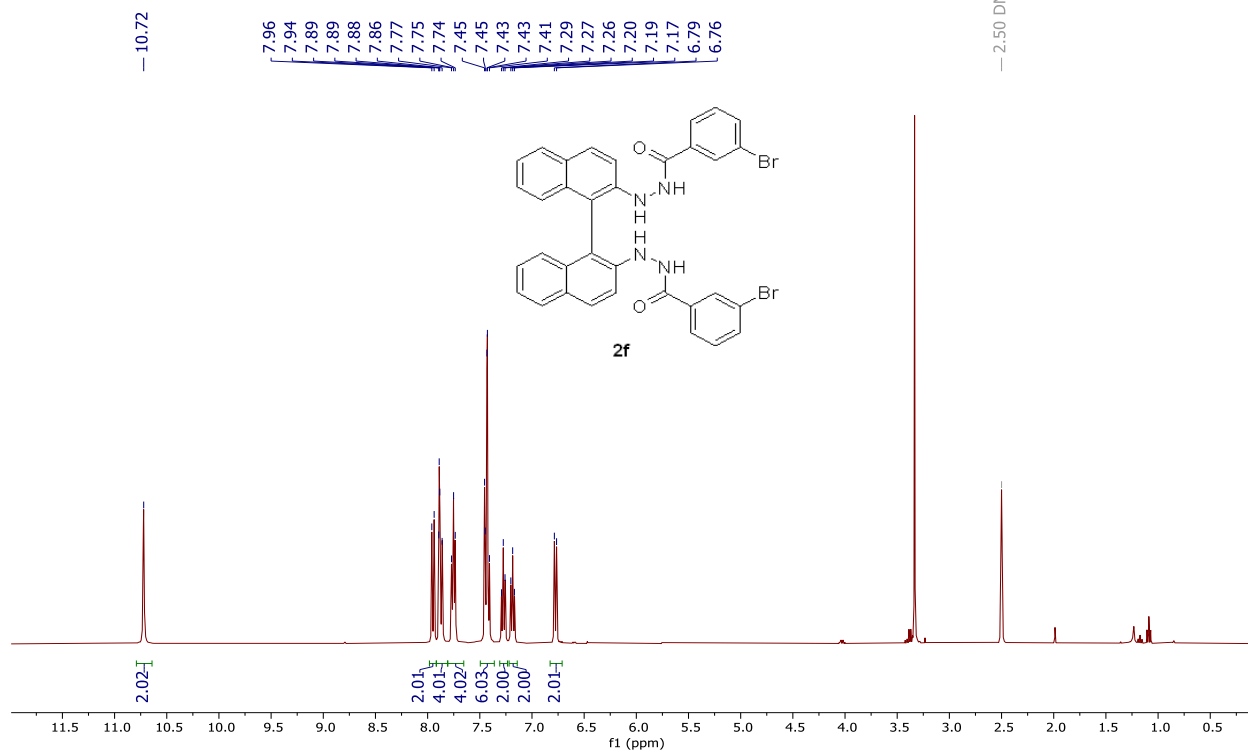
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et-b6-4-me-bnm



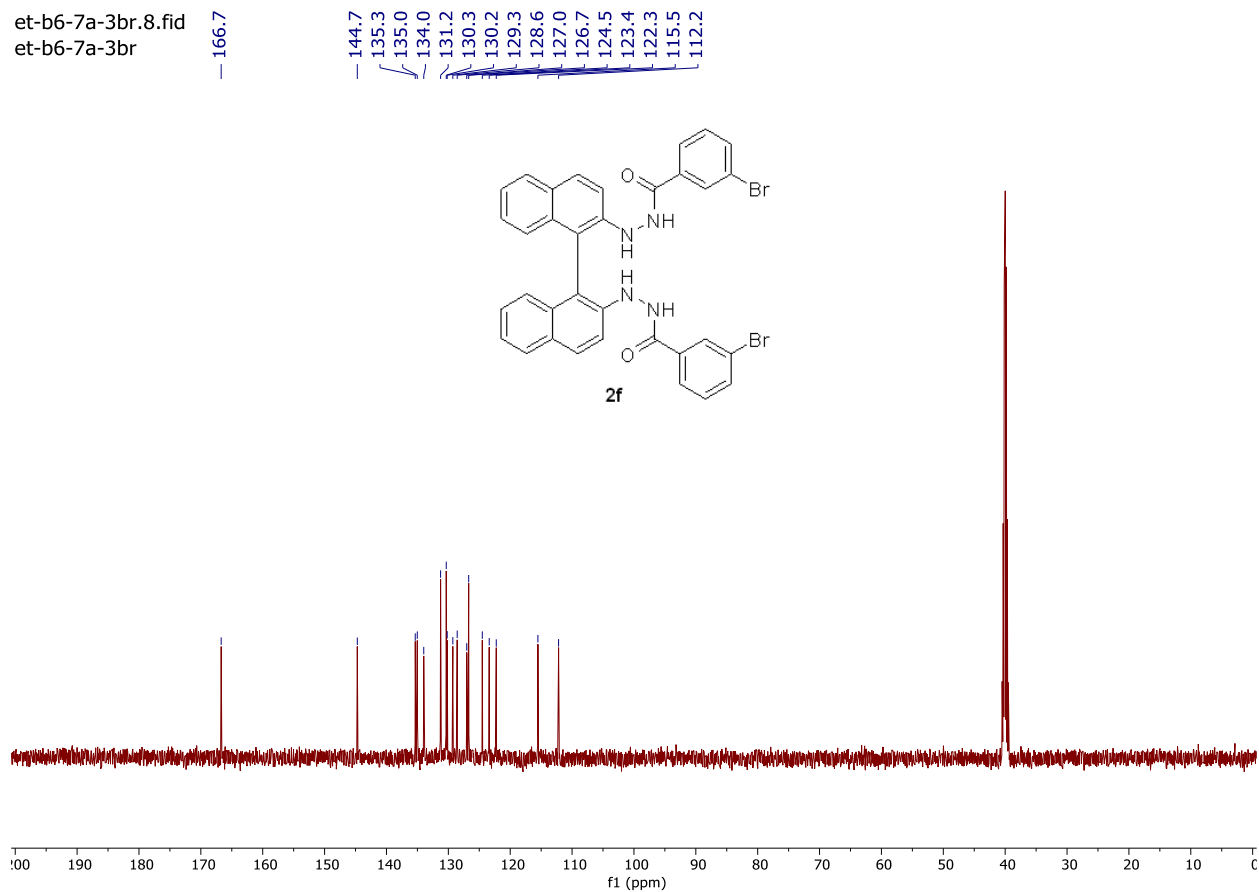
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May18-2023-et-b6-7-3br.10.fid
May18-2023-et-b6-7-3br



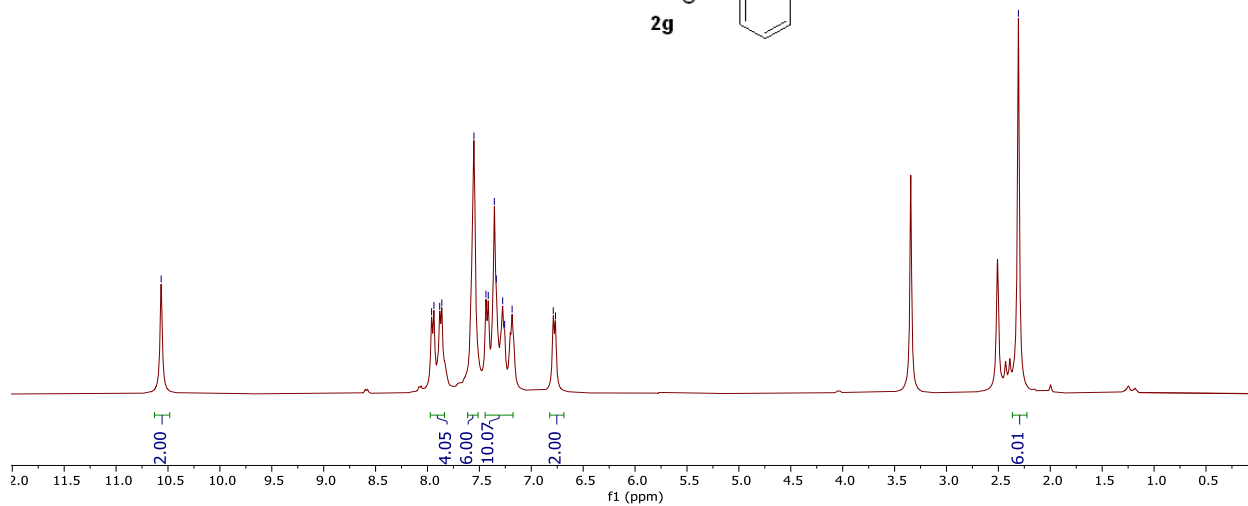
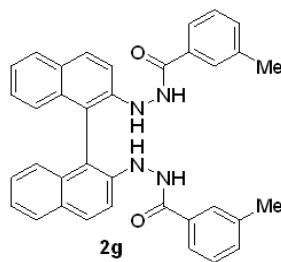
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et-b6-7a-3br



May18-2023-et-b6-11b-3me.10.fid
May18-2023-et-b6-11b-3me

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7.94
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6.77

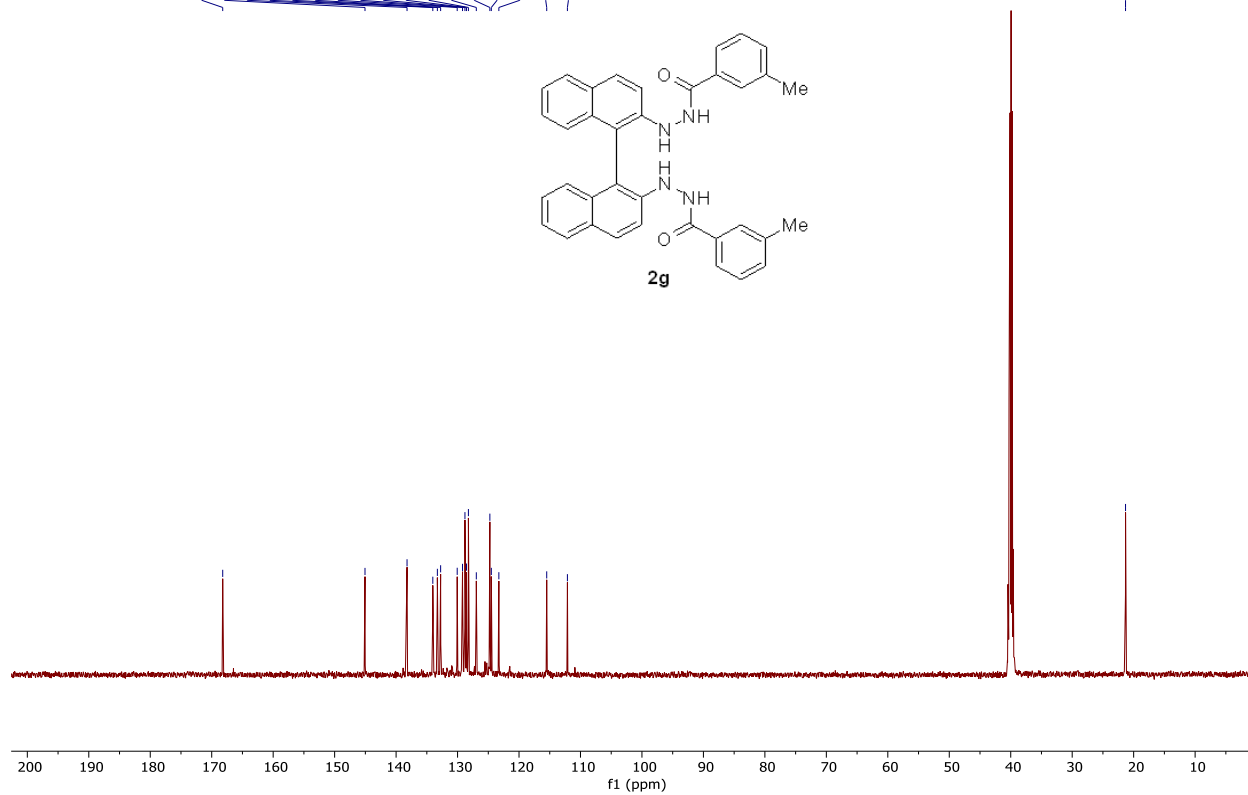
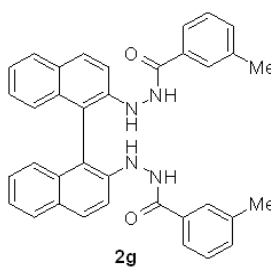
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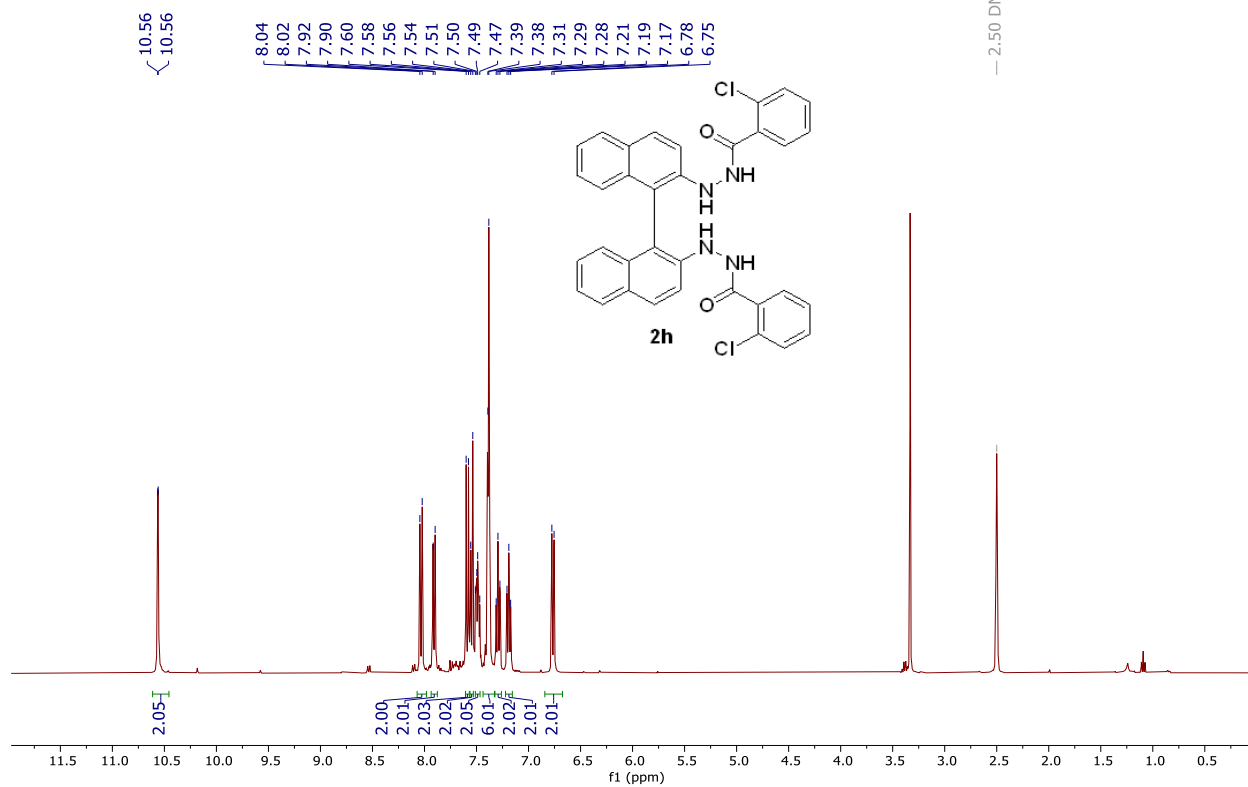
et-b6-3me-bnm-cl
et-b6-3me-bnm-cl

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115.5
112.1

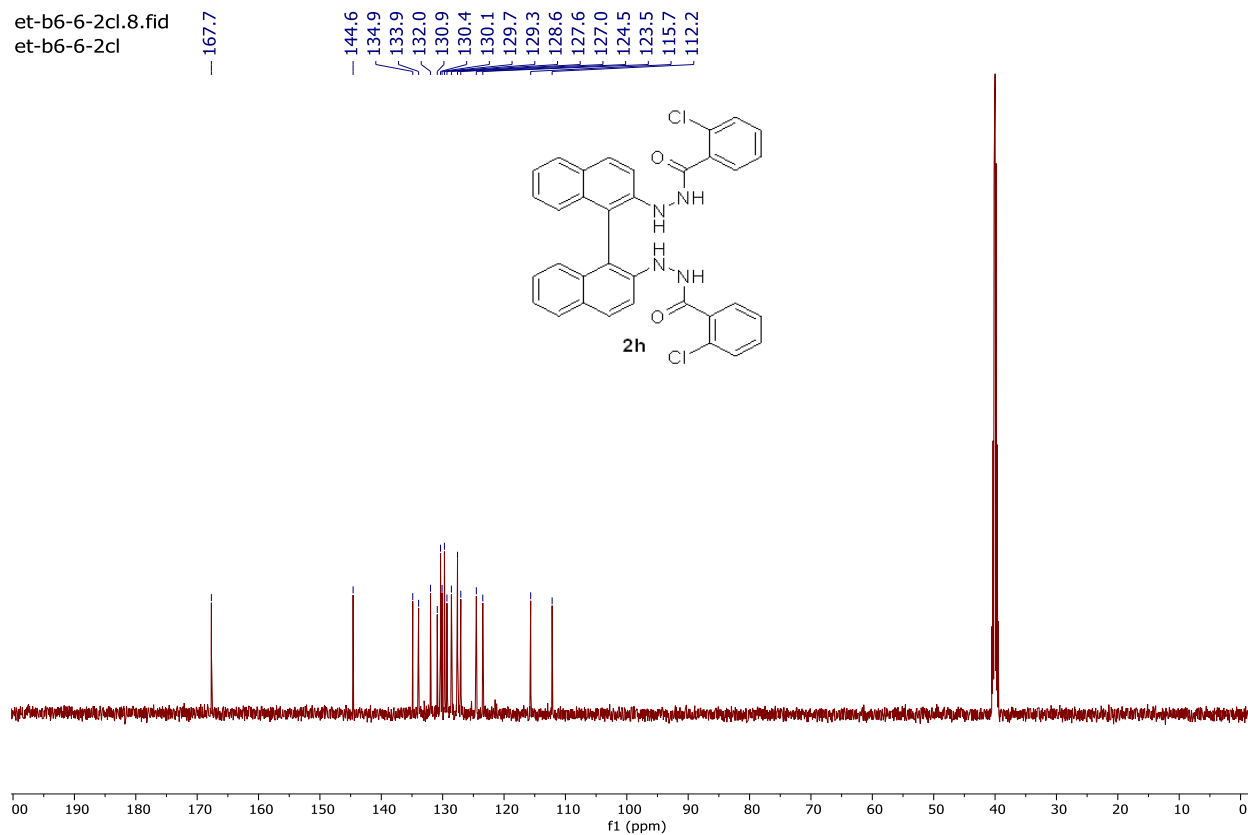
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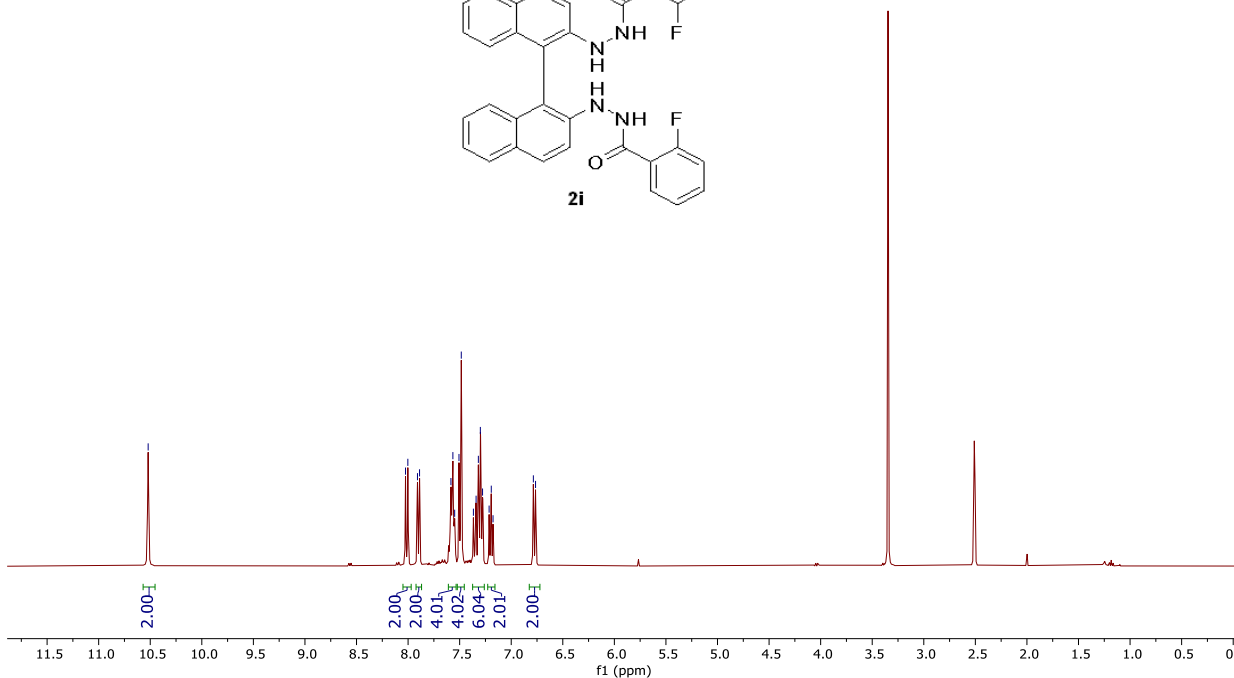
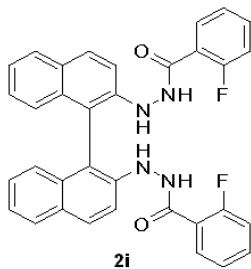


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et-b6-6-2cl



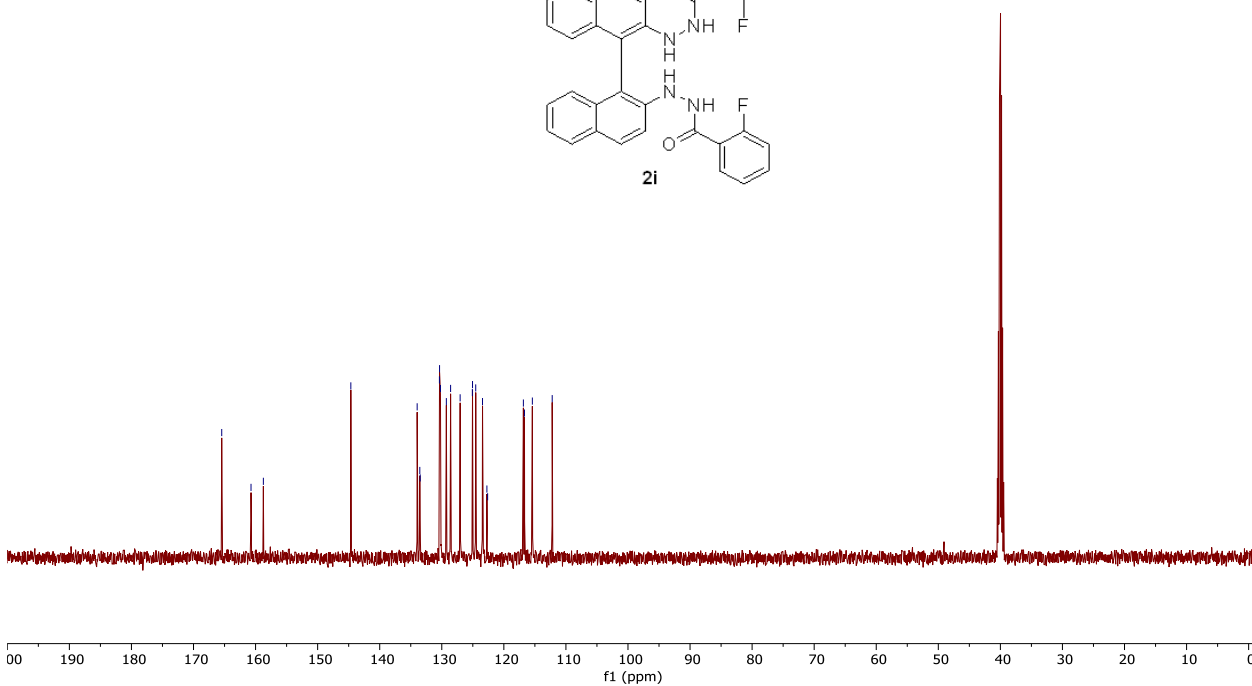
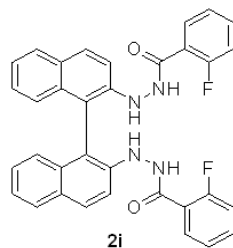
May18-2023-et-b6-3-bzf.10.fid
May18-2023-et-b6-3-bzf

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6.76

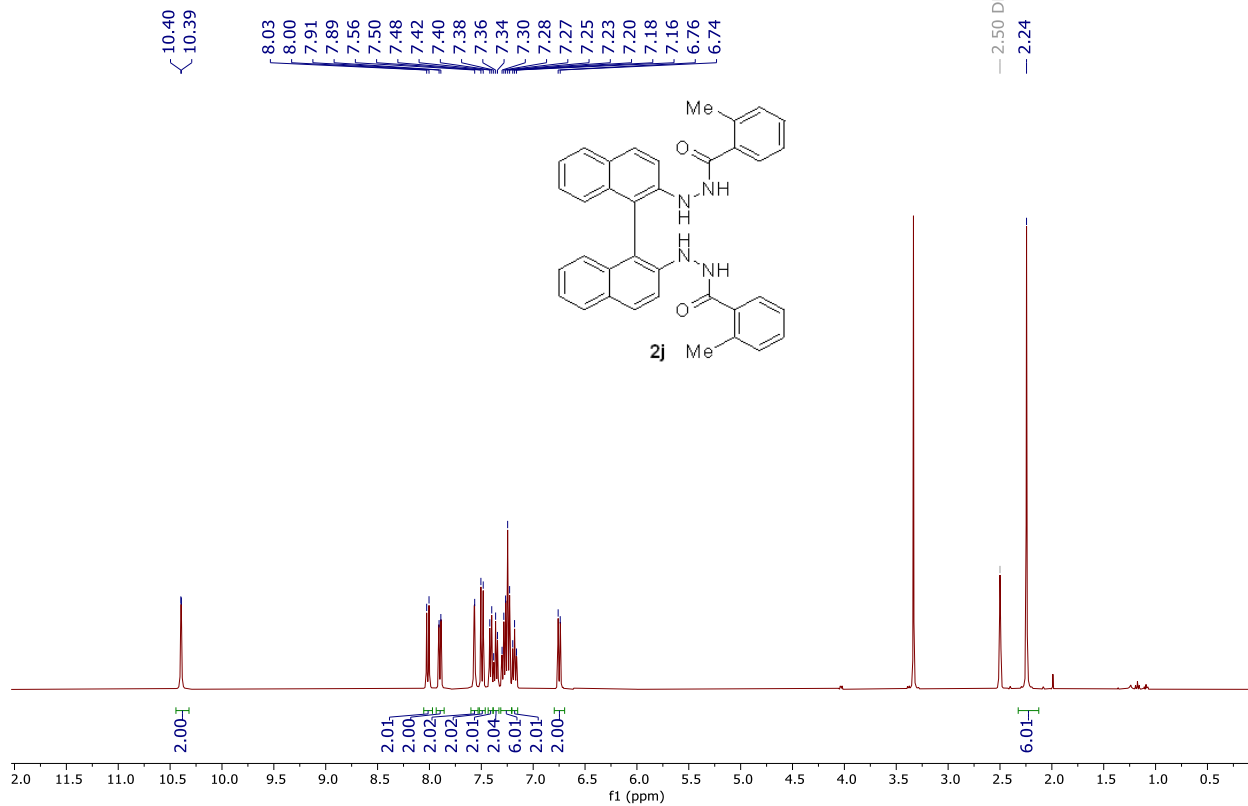


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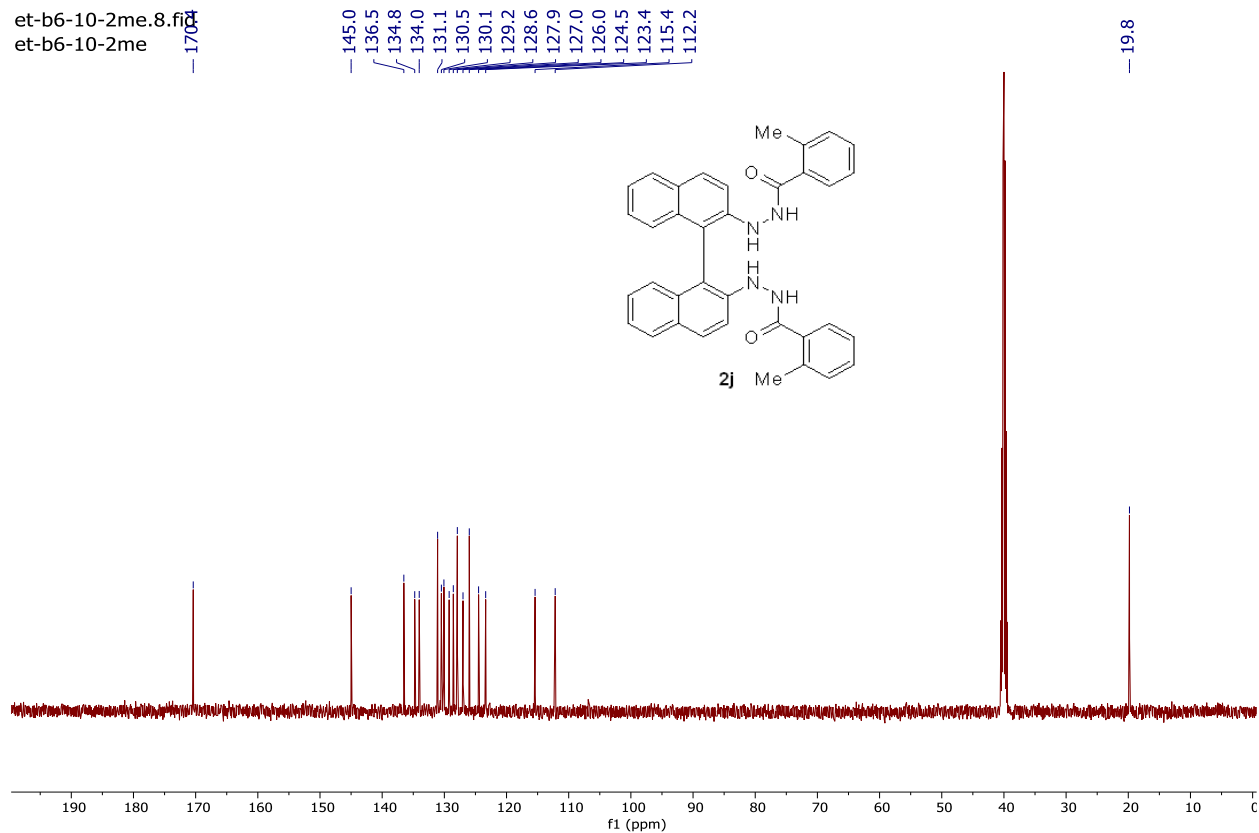
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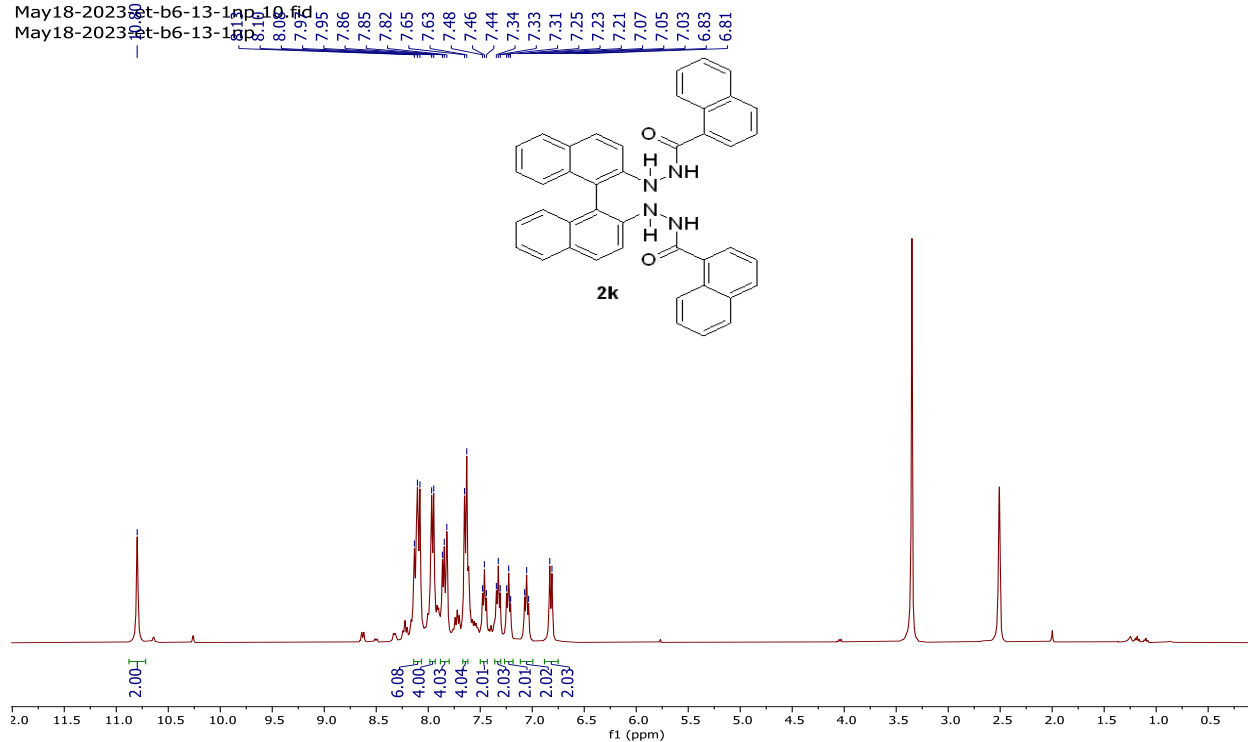
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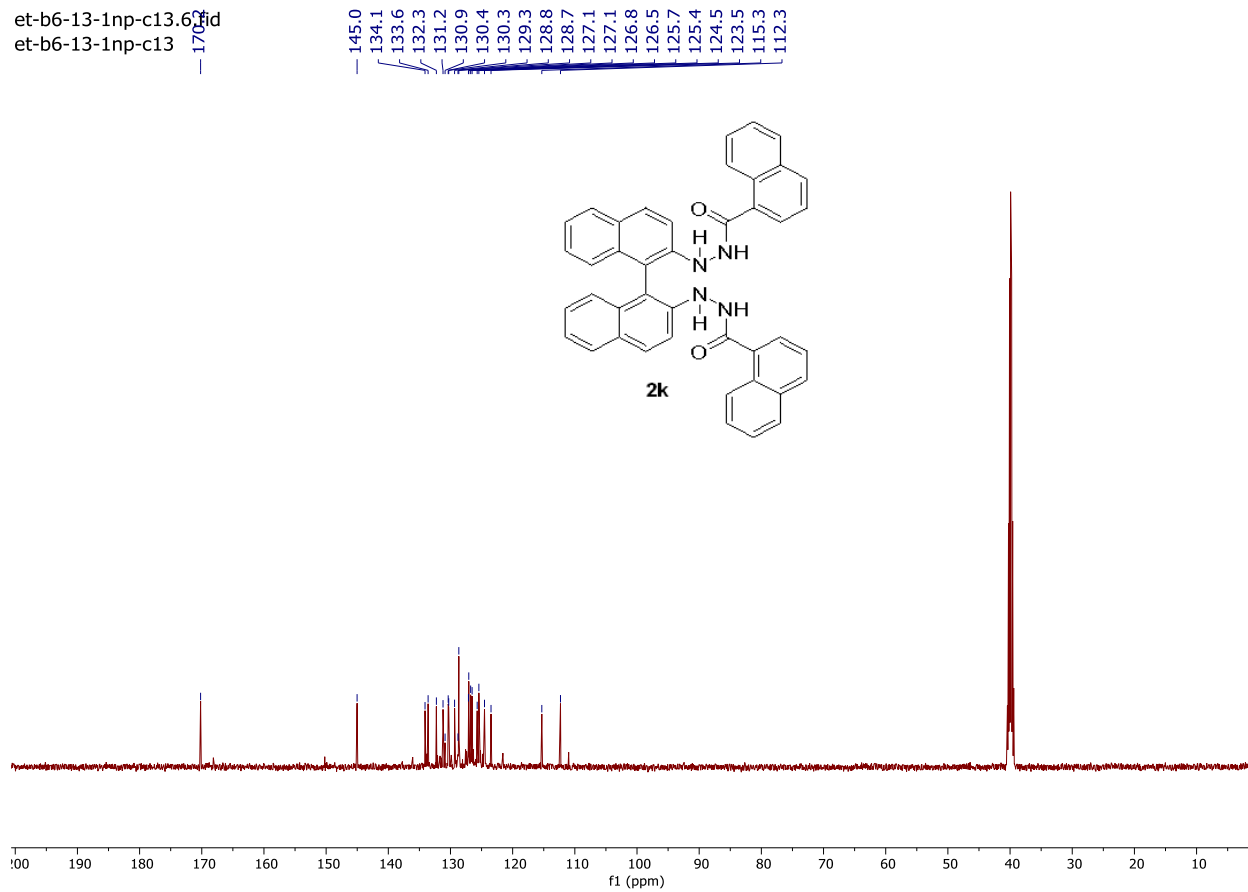
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et-b6-10-2me



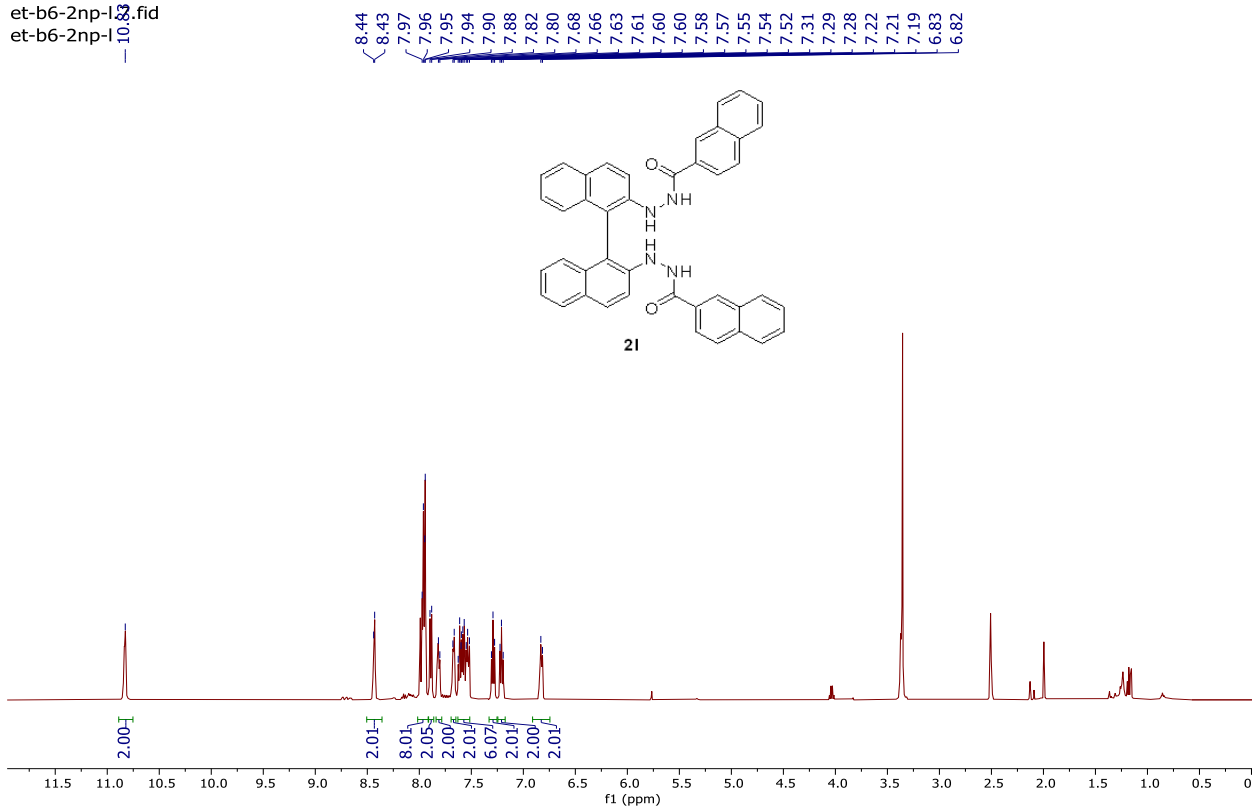
May18-2023 et-b6-13-1np-c13.63fid
May18-2023 et-b6-13-1np-c13



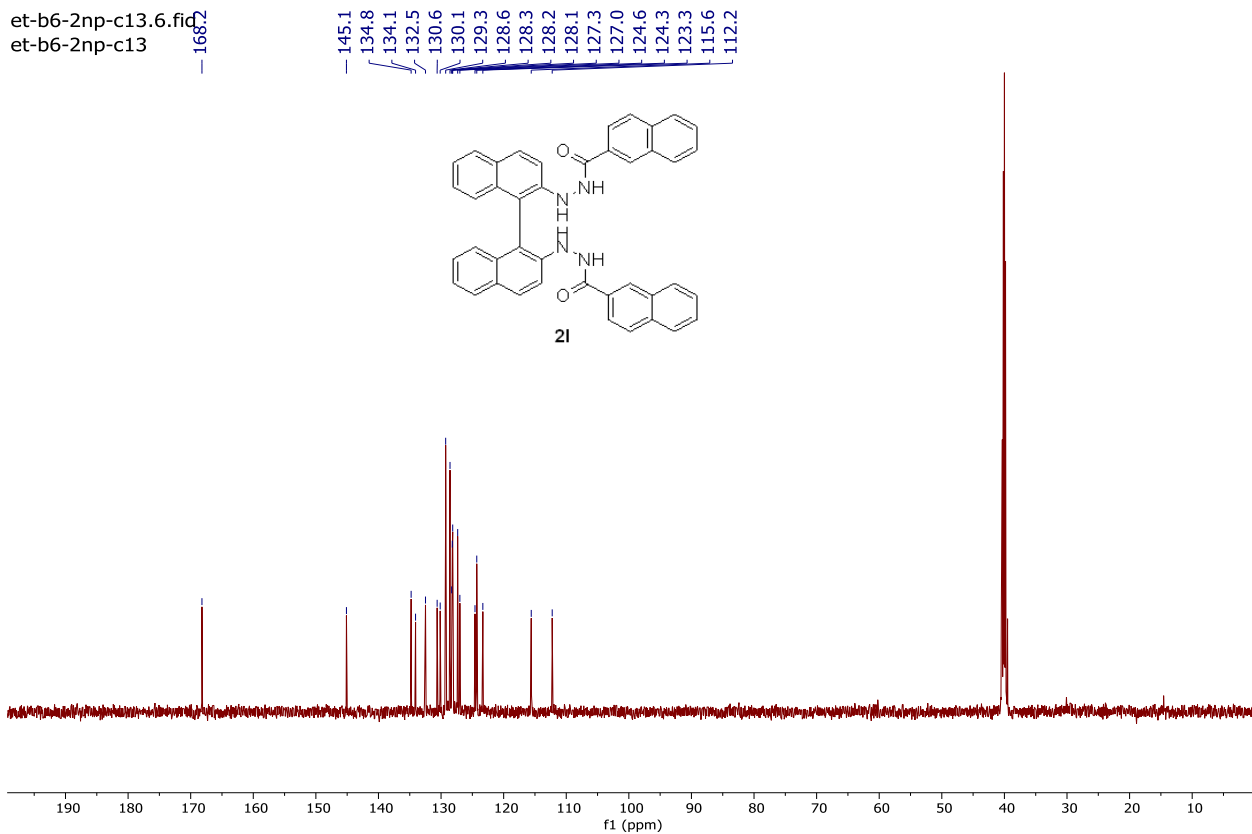
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et-b6-13-1np-c13



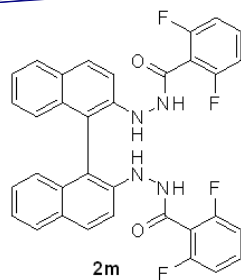
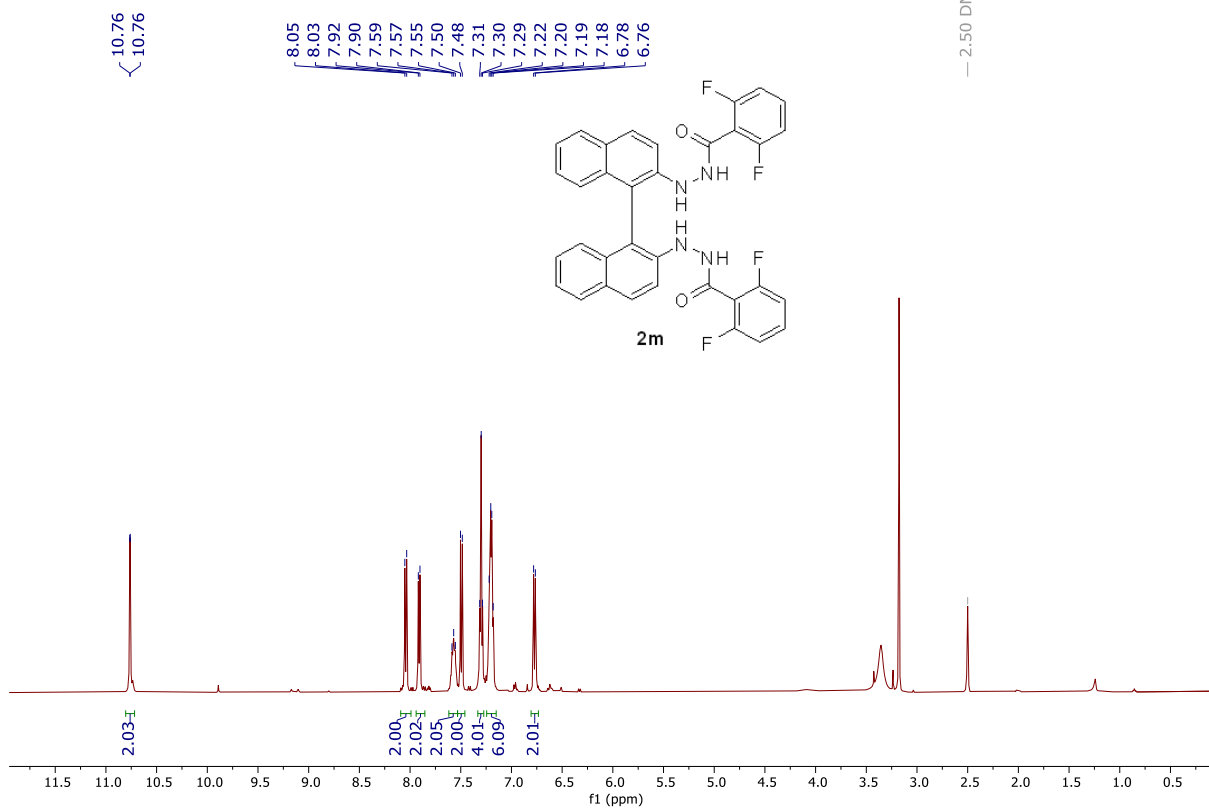
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et-b6-2np-l



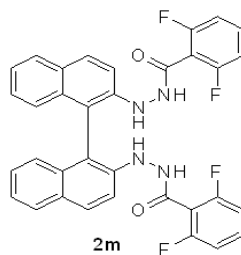
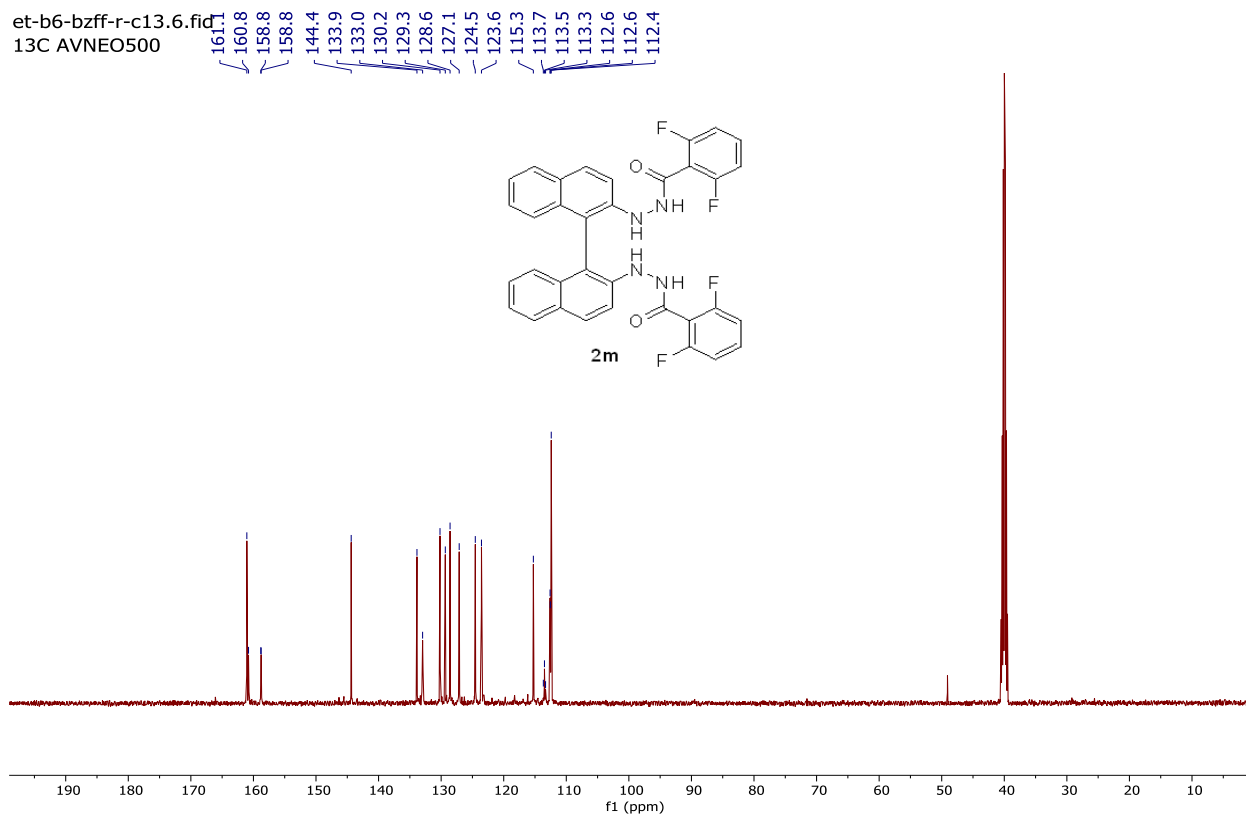
et-b6-2np-c13.6.fid
et-b6-2np-c13



et-b6-bzff-r.7.fid
et-b6-bzff-r

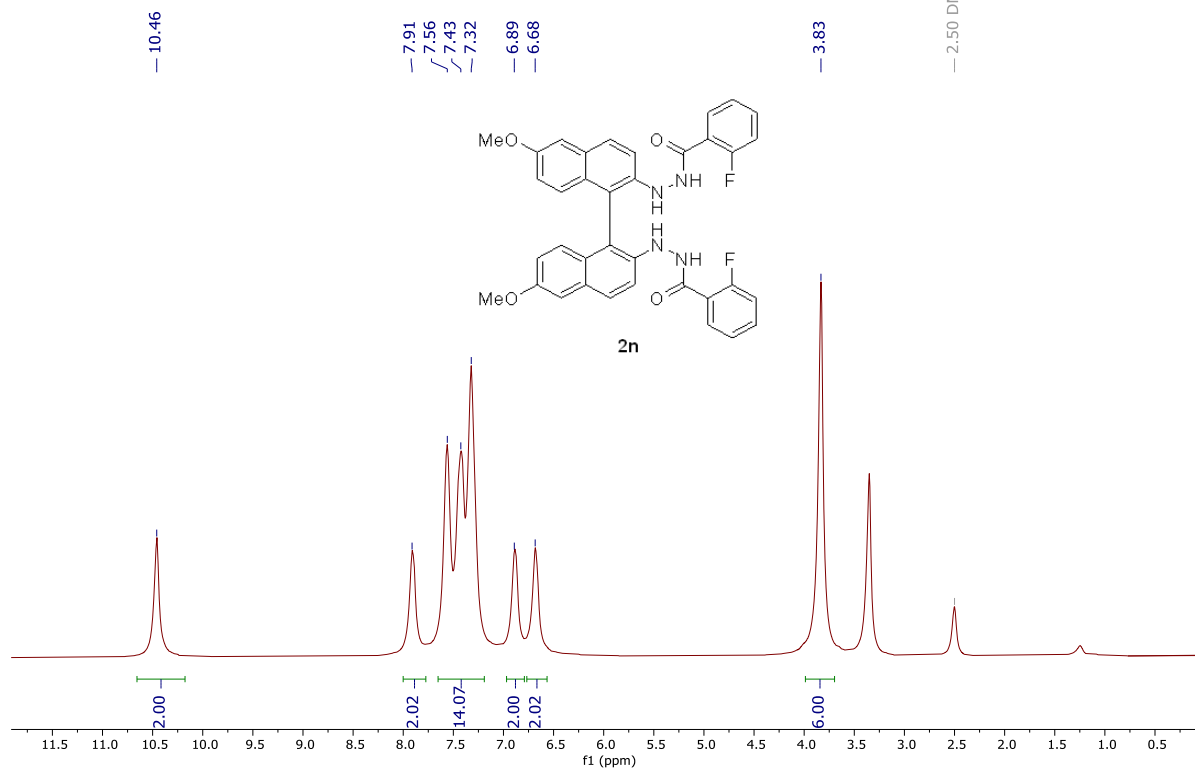


et-b6-bzff-r-c13.6.fid
13C AVNEO500

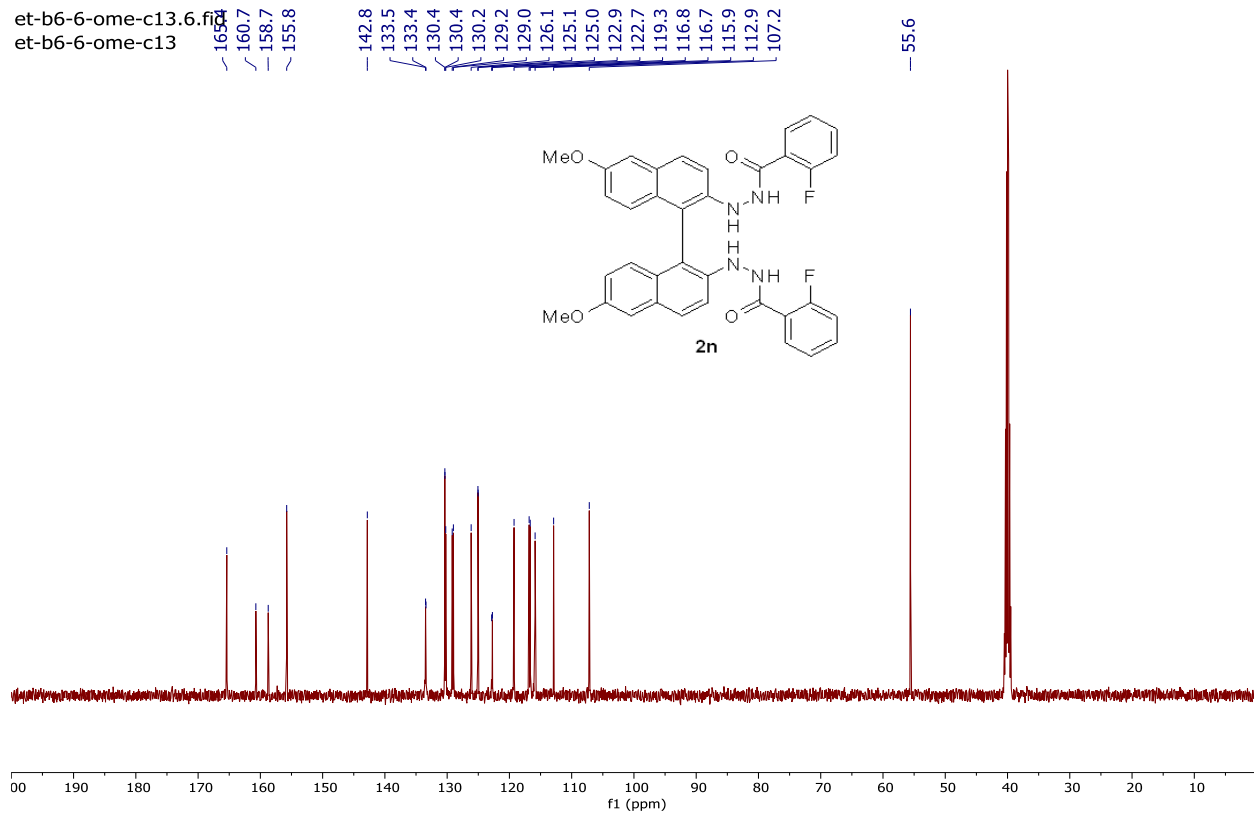


Nov02-2023-et-b6-6ome-bm-h.10.fid
Nov02-2023-et-b6-6ome-bm-h

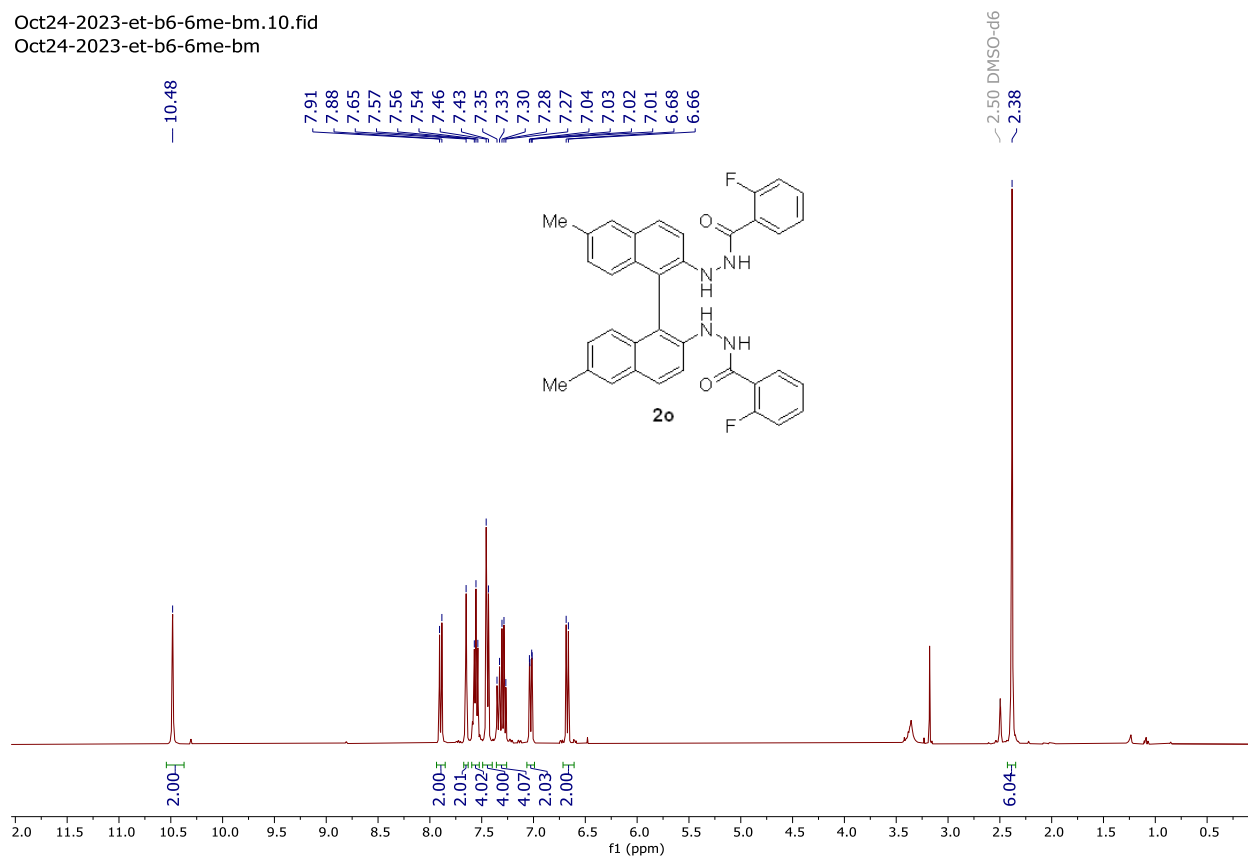
— 2.50 DMSO-d6



et-b6-6-ome-c13.6.fid
et-b6-6-ome-c13



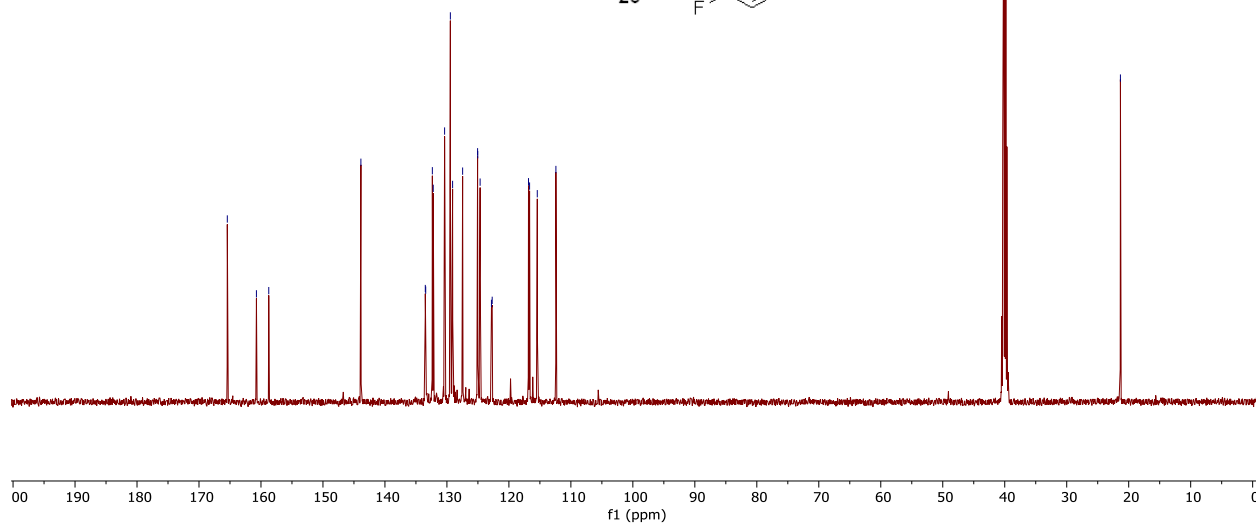
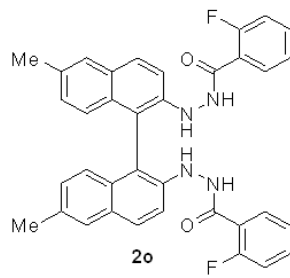
Oct24-2023-et-b6-6me-bm.10.fid
Oct24-2023-et-b6-6me-bm



et-b6-r6me-bnm-c13
et-b6-r6me-bnm-c13

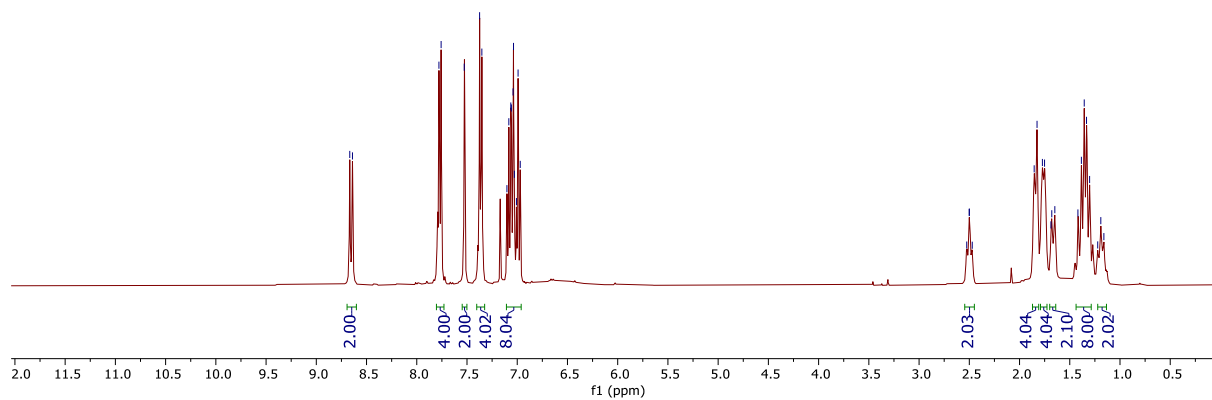
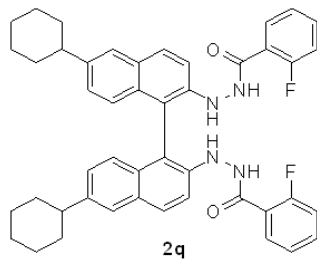
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133.5
133.4
132.3
132.2
130.4
129.4
129.1
127.5
125.1
125.0
124.6
122.8
122.7
116.8
116.7
115.4
112.4

21.3



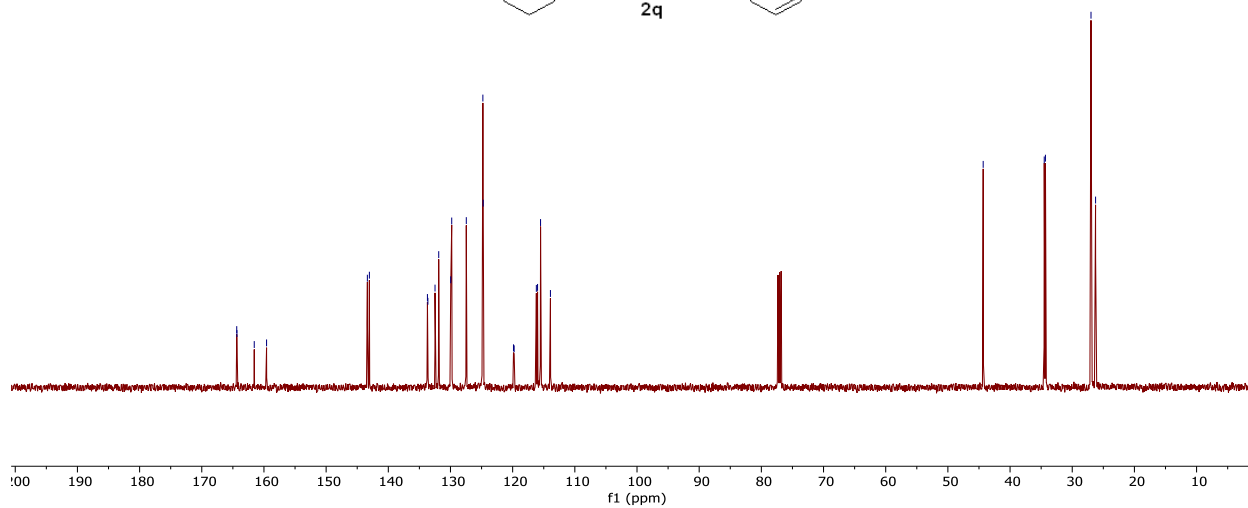
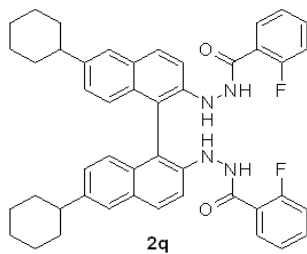
Nov02-2023-et-b6-6cyx-bm-h.10.fid
Nov02-2023-et-b6-6cyx-bm-h

8.67 7.78 7.76 7.53 7.37 7.35 7.10 7.08 7.06 7.06 7.04 7.03 7.01 6.99 6.97 2.53 2.50 2.50 2.47 1.85 1.83 1.77 1.75 1.69 1.68 1.65 1.42 1.38 1.36 1.33 1.30 1.22 1.19 1.16

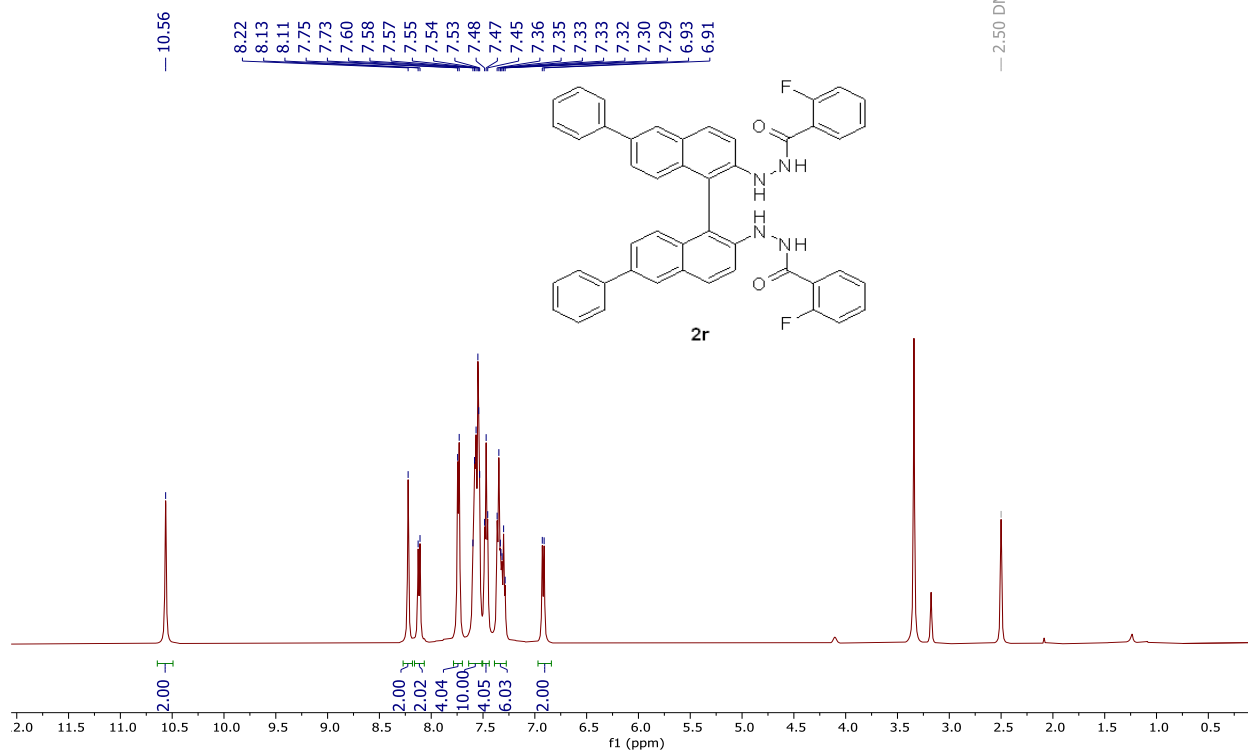


et-b6-cyx-c13.6.fid
et-b6-cyx-c13

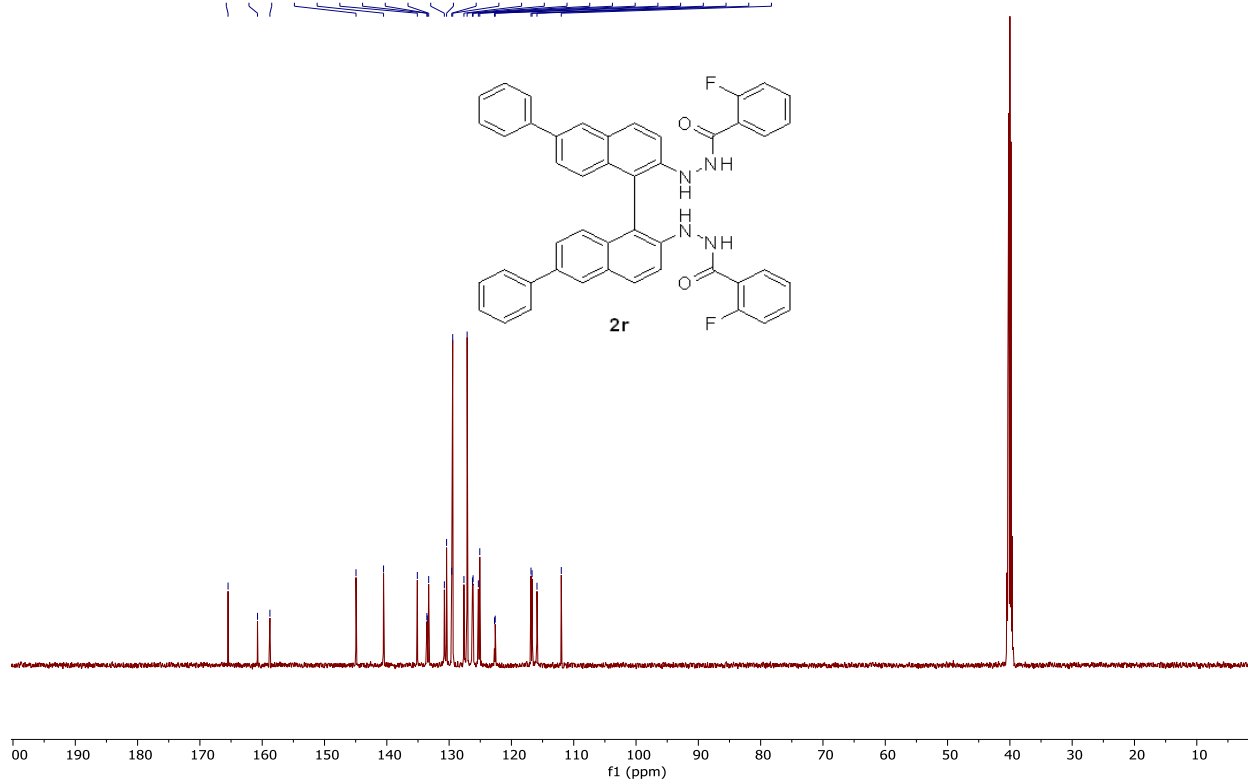
164.4 164.4 161.6 159.6 143.4 143.0 133.7 133.6 132.5 131.9 130.0 129.8 127.5 124.8 124.7 119.9 119.8 116.2 116.0 115.5 113.9 44.3 34.5 34.3 27.0 26.2



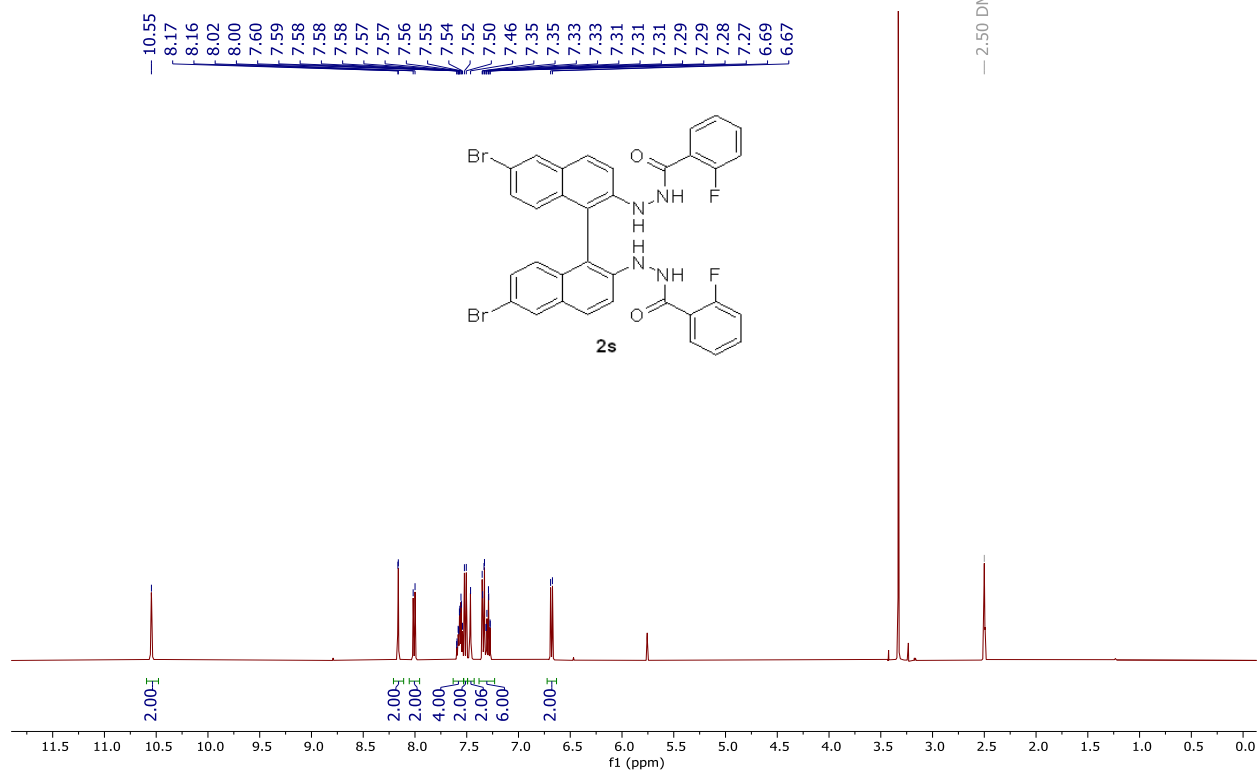
et-b6-6-ph-bnm.7.fid
et-b6-6-ph-bnm



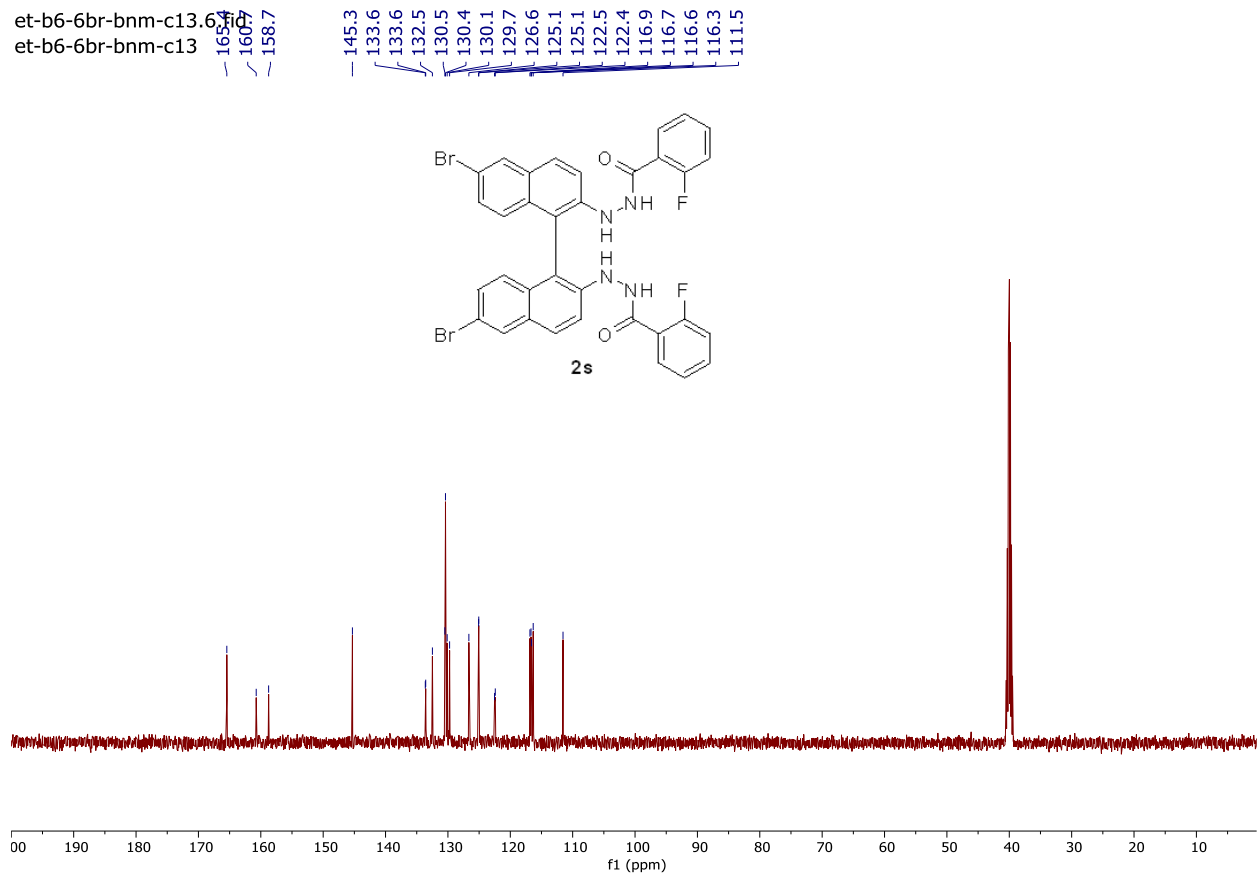
et-b6-6-ph-bnm-c13-5
et-b6-6-ph-bnm-c13-5



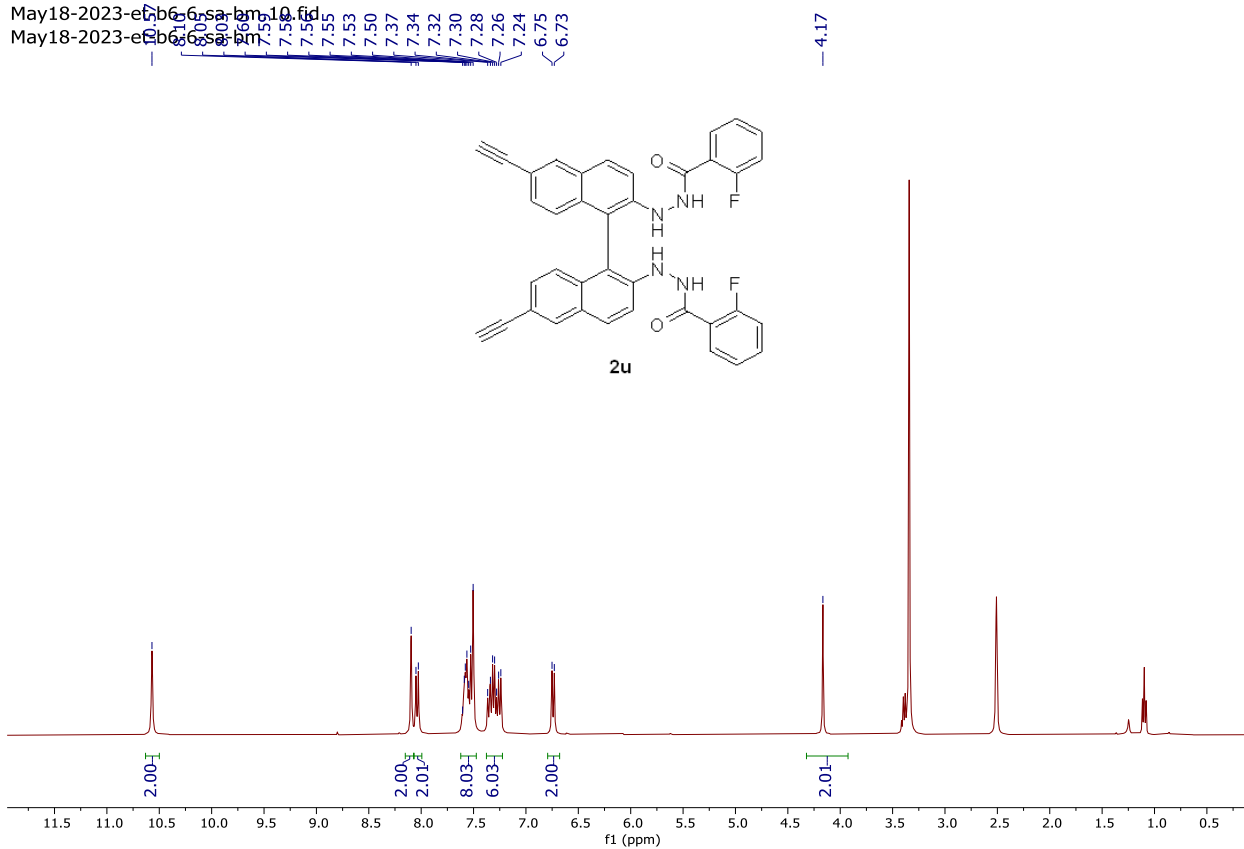
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et-b6-6br-bnm



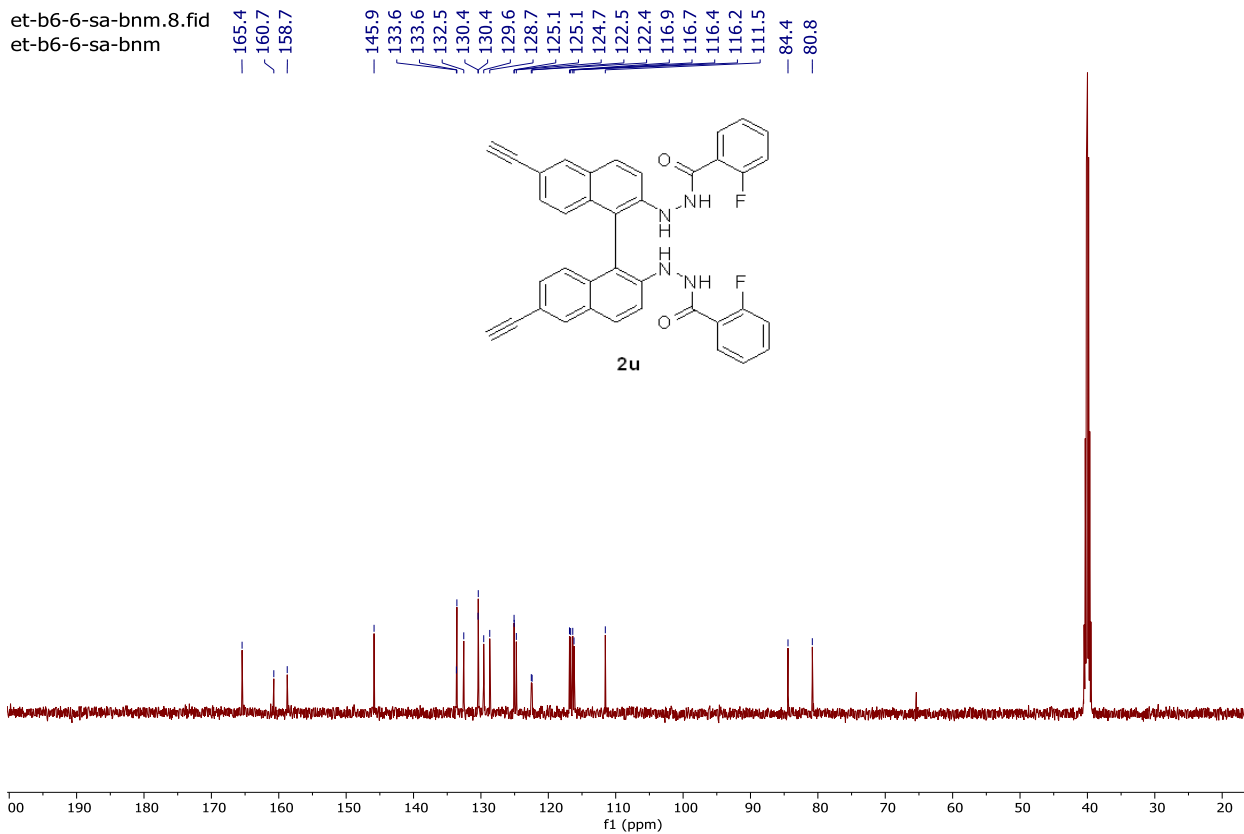
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et-b6-6br-bnm-c13



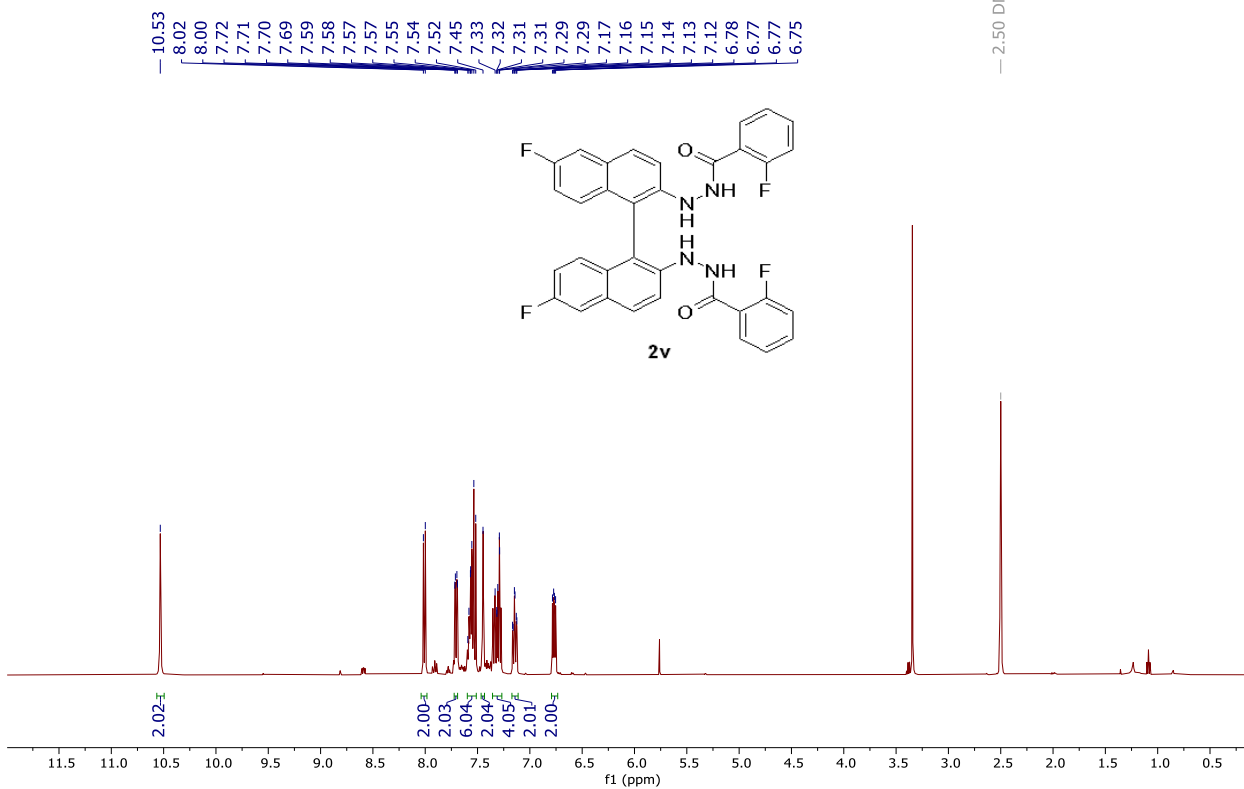
May18-2023-et-b6-6-sa-bnm.10.fid
May18-2023-et-b6-6-sa-bnm



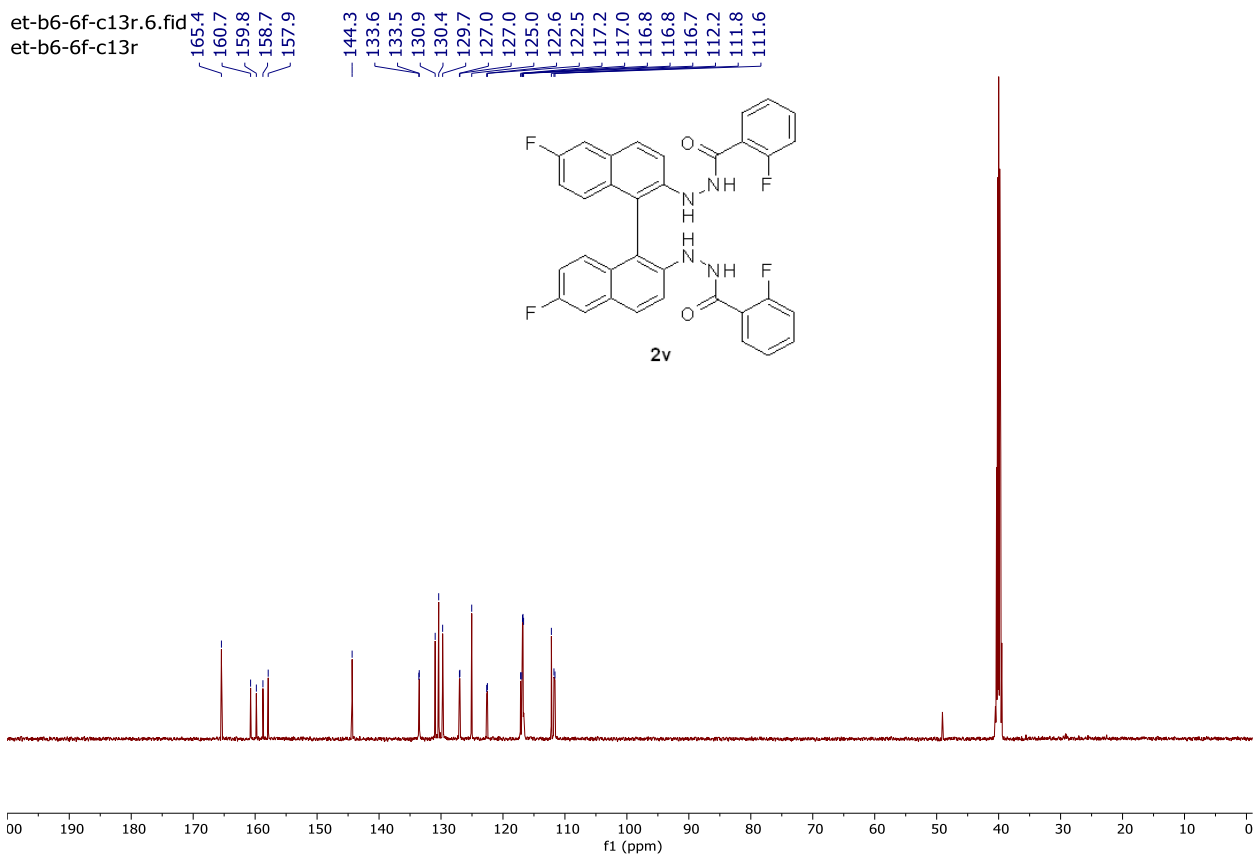
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et-b6-6-sa-bnm



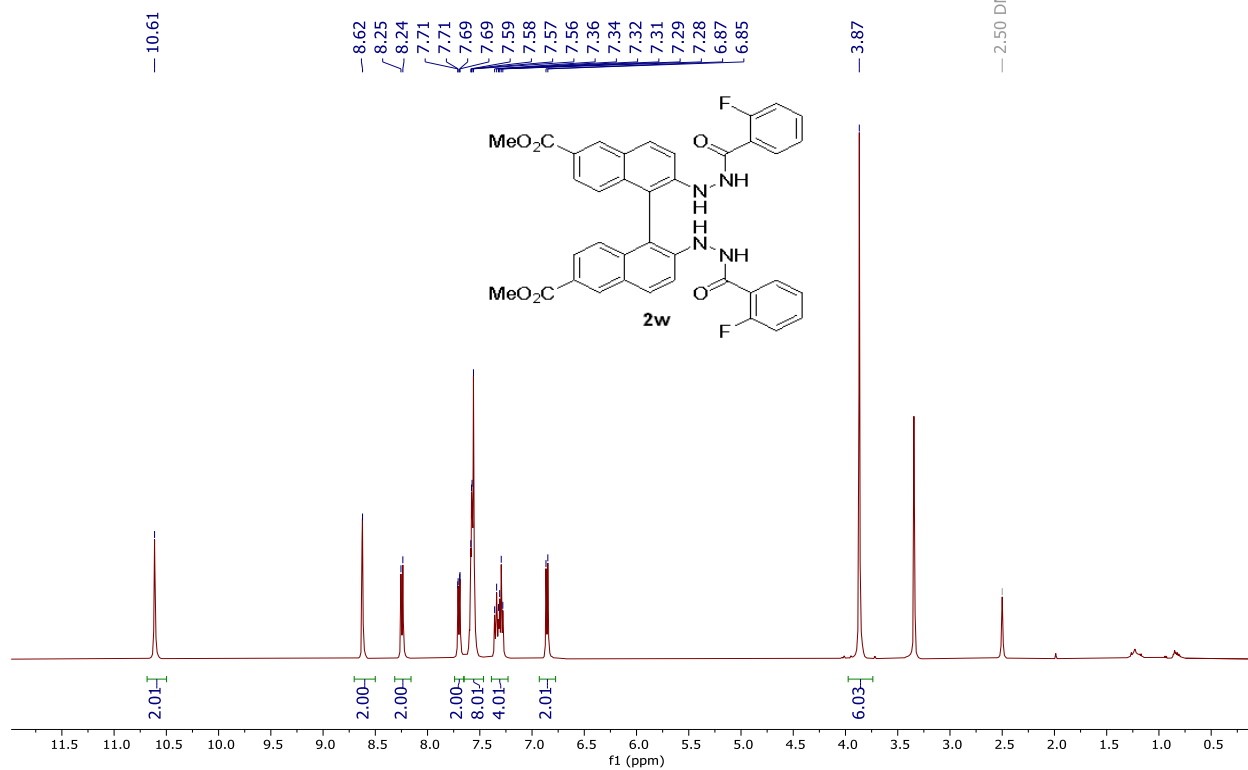
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et-f-bnm



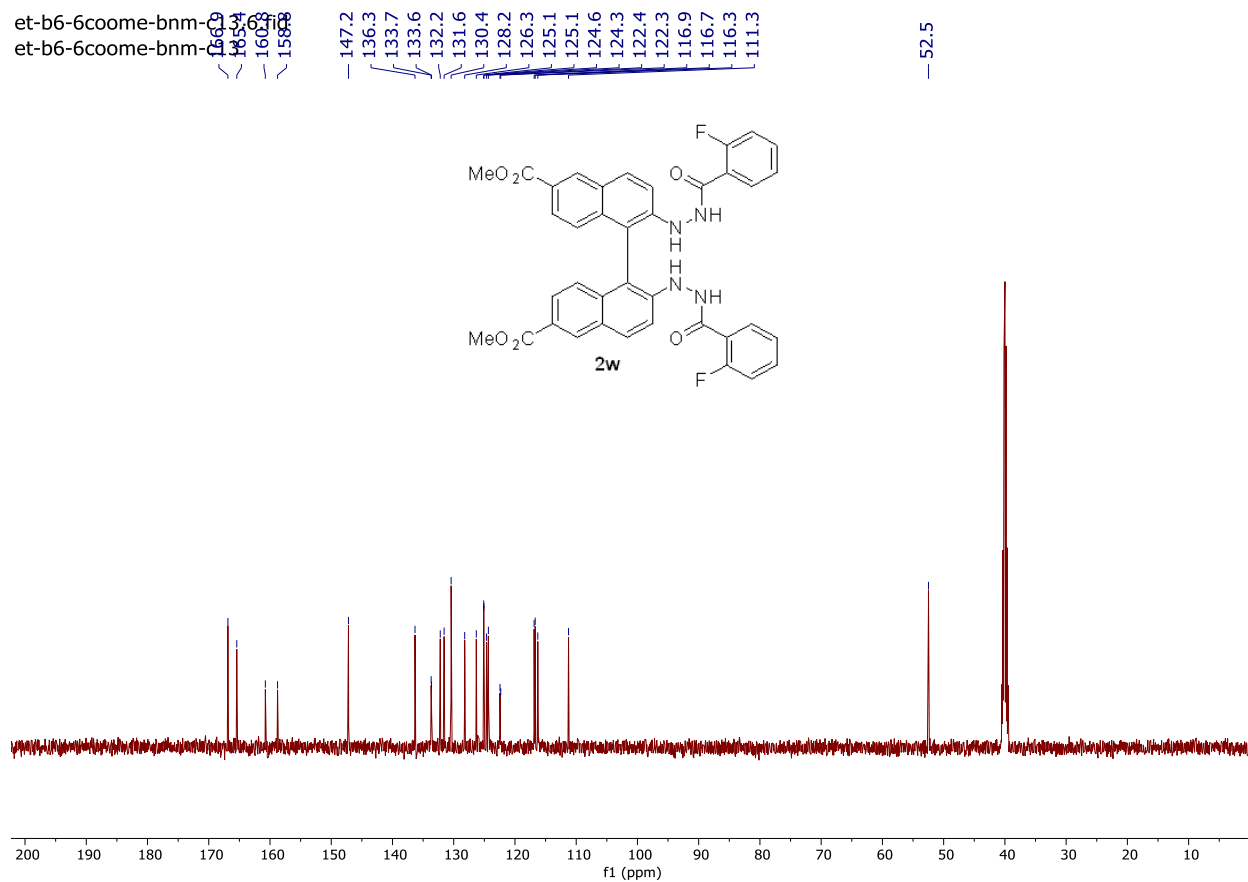
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et-b6-6f-c13r



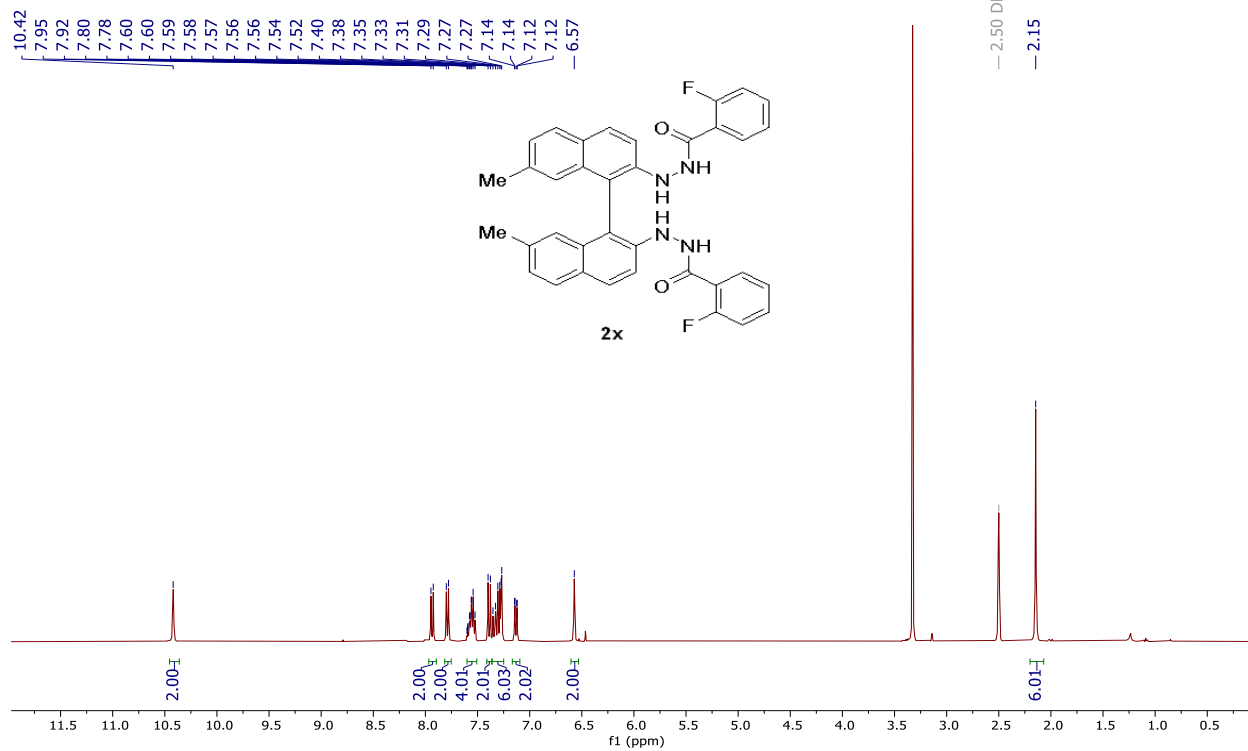
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et-b6-6coome-bnm



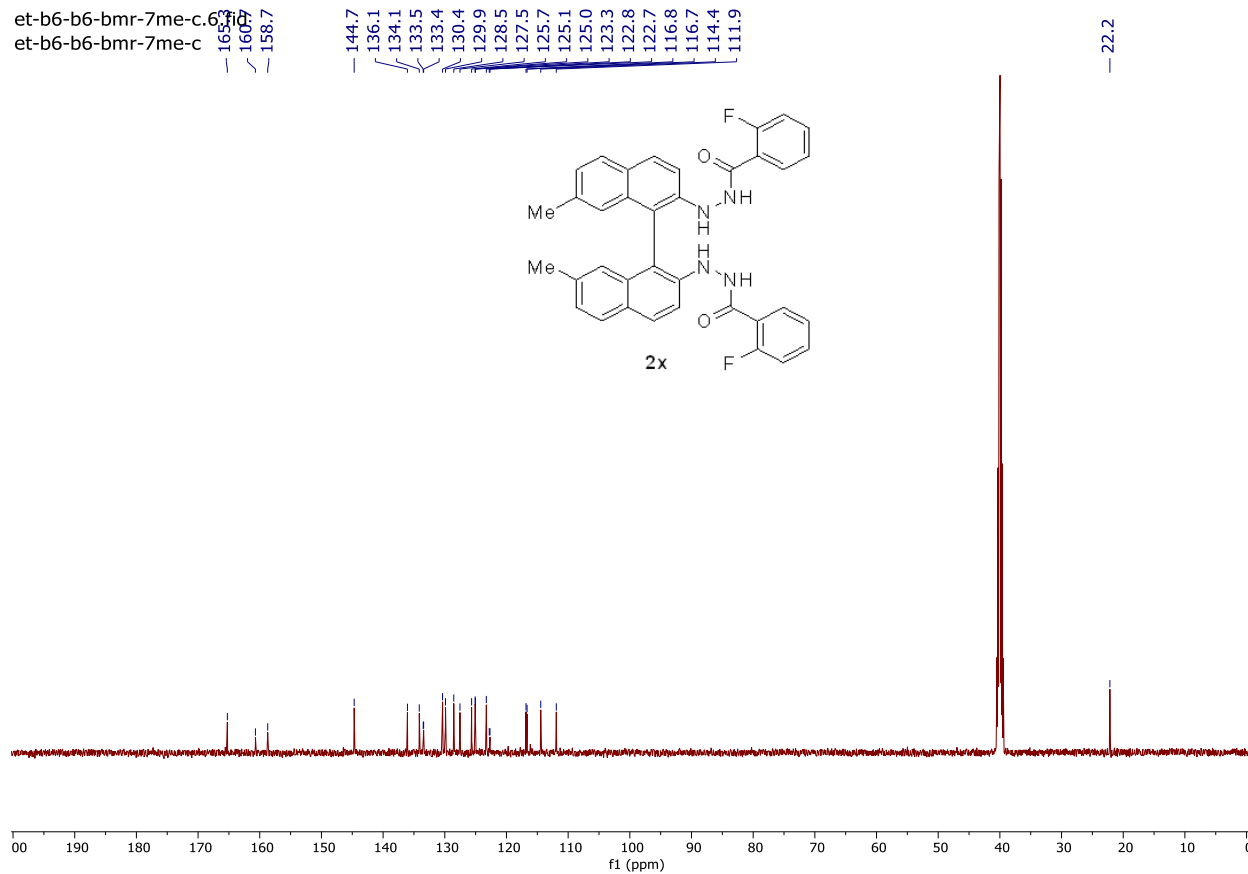
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et-b6-6coome-bnm-1654
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et-b6-6coome-bnm-1588



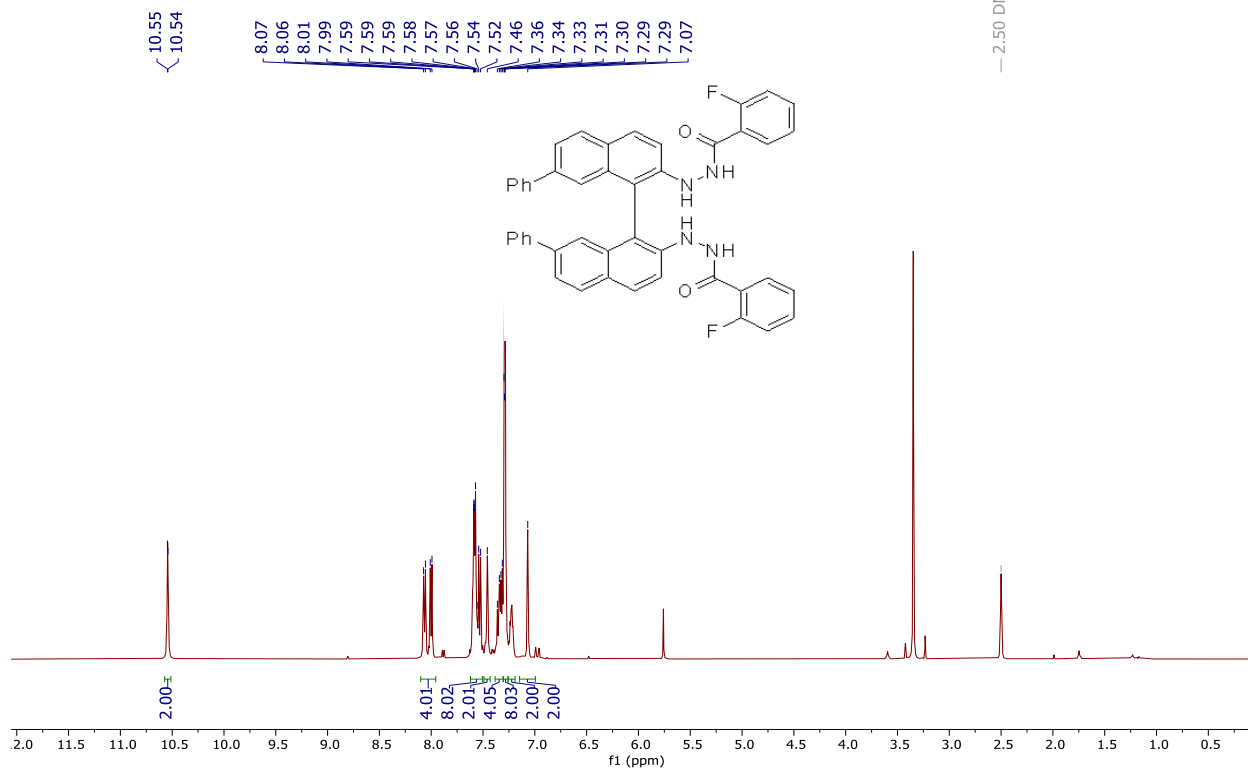
Sep23-2023-et-7me-bm.10.fid
Sep23-2023-et-7me-bm



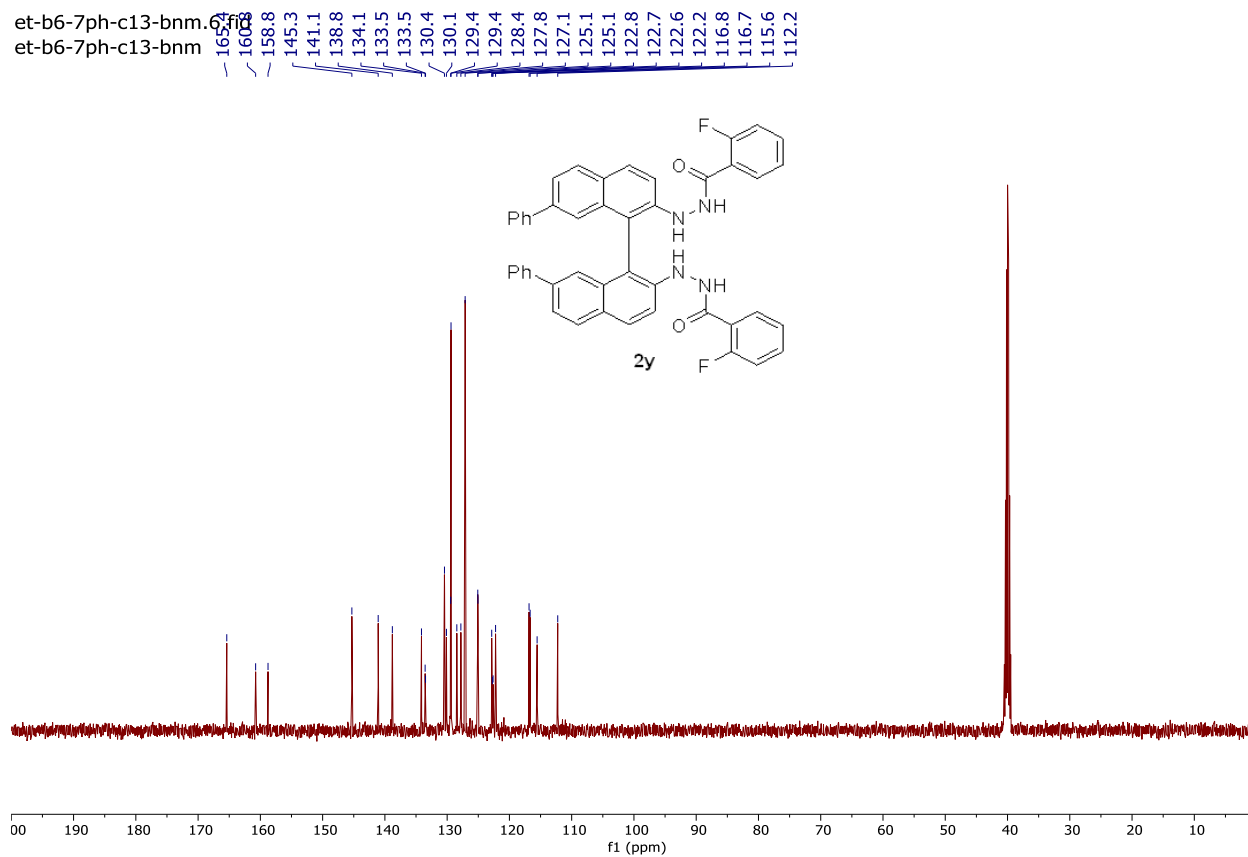
et-b6-b6-bmr-7me-c.63
et-b6-b6-bmr-7me-c



et-b6-7ph-bnm.7.fid
et-b6-7ph-bnm

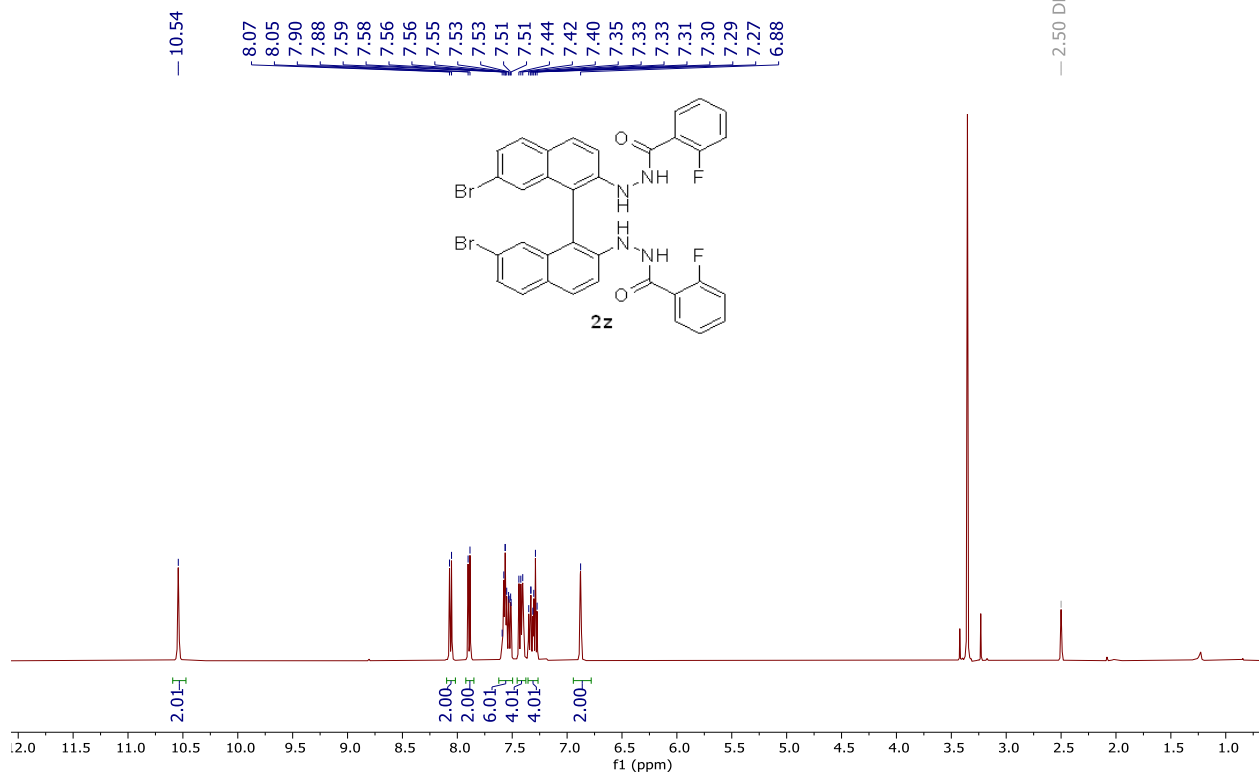


et-b6-7ph-c13-bnm.6.fid
et-b6-7ph-c13-bnm

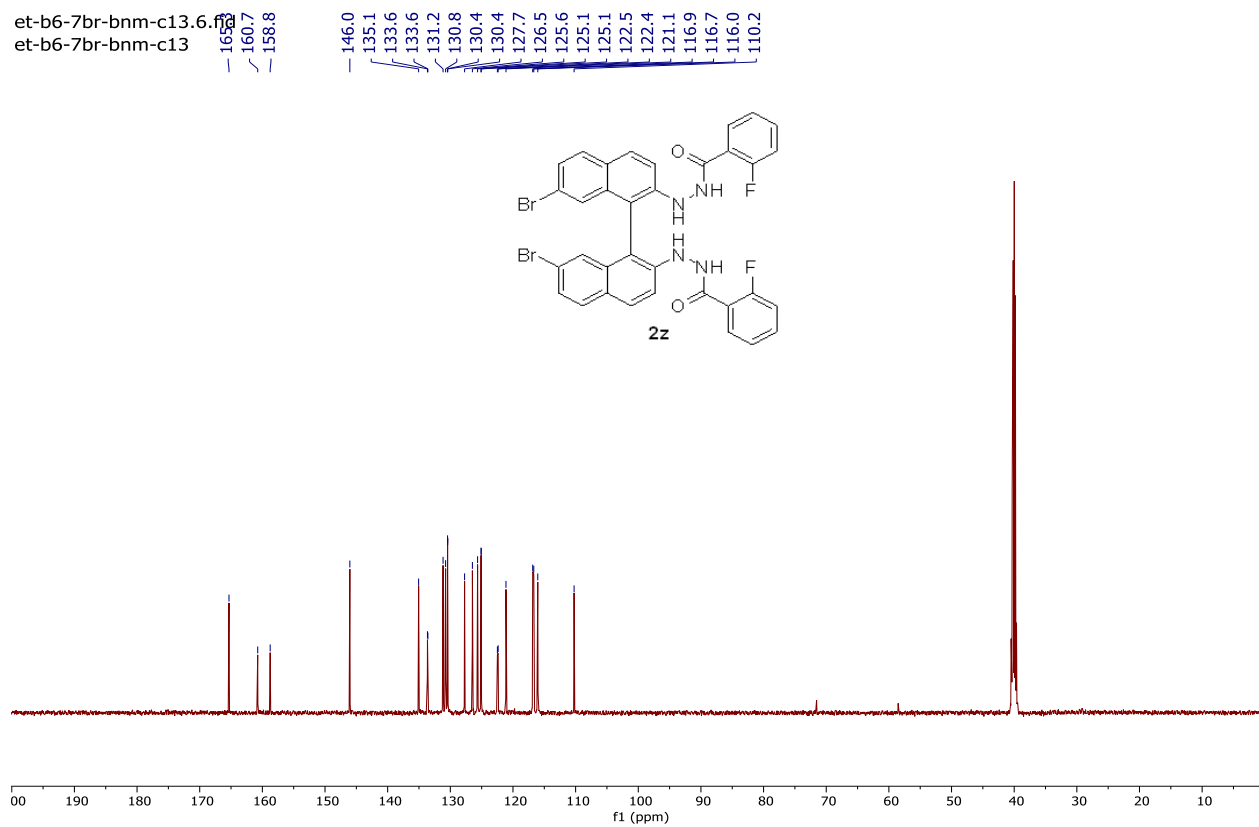


et-b6-7br-bnm-r.7.fid
et-b6-7br-bnm

—2.50 DMSO-d6



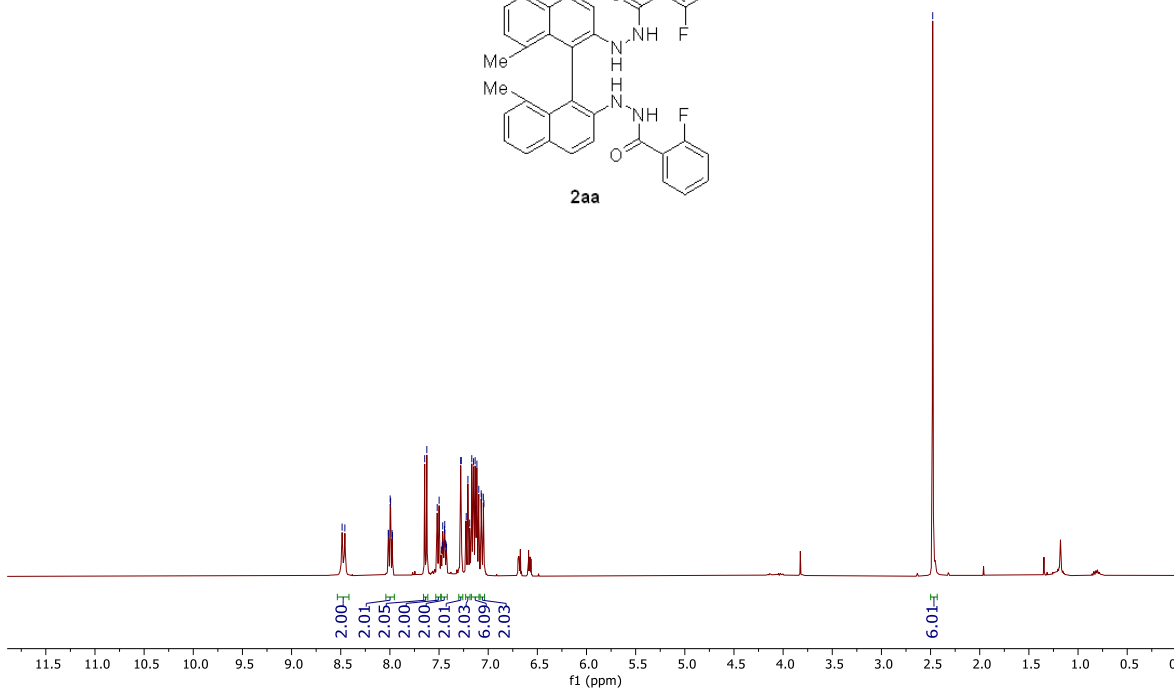
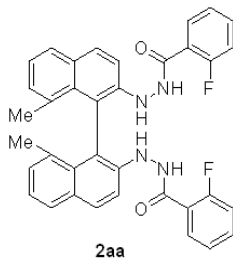
et-b6-7br-bnm-c13.6.fid
et-b6-7br-bnm-c13



S

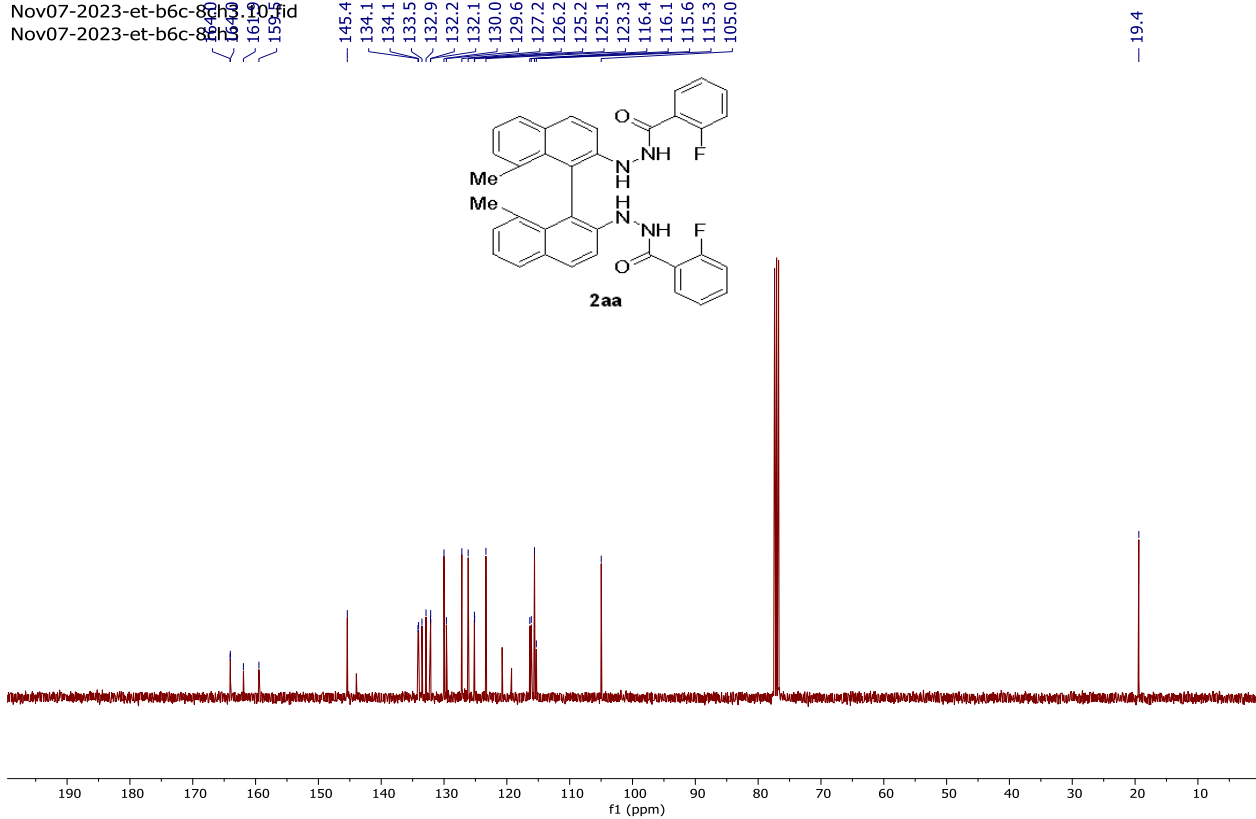
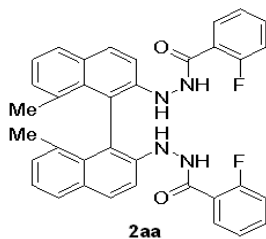
Nov06-2023-et-b6-8ch3-bm.10.fid
Nov06-2023-et-b6-8ch3-bm

8.49
8.46
8.02
8.01
8.00
7.99
7.98
7.97
7.65
7.62
7.52
7.50
7.48
7.47
7.44
7.44
7.44
7.43
7.42
7.28
7.27
7.22
7.21
7.19
7.17
7.15
7.13
7.11
7.10
7.07
7.07
7.05
7.04
2.48

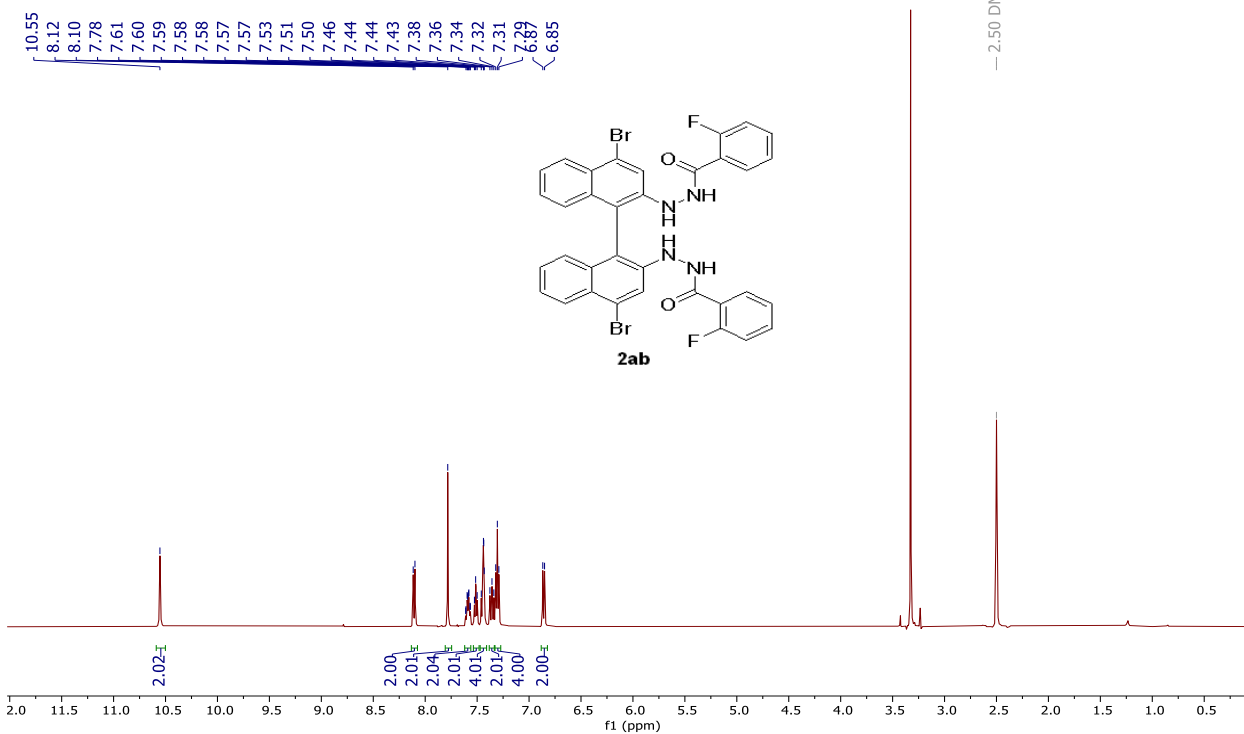


Nov07-2023-et-b6c-8ch3-10.fid
Nov07-2023-et-b6c-8ch3-10.fid

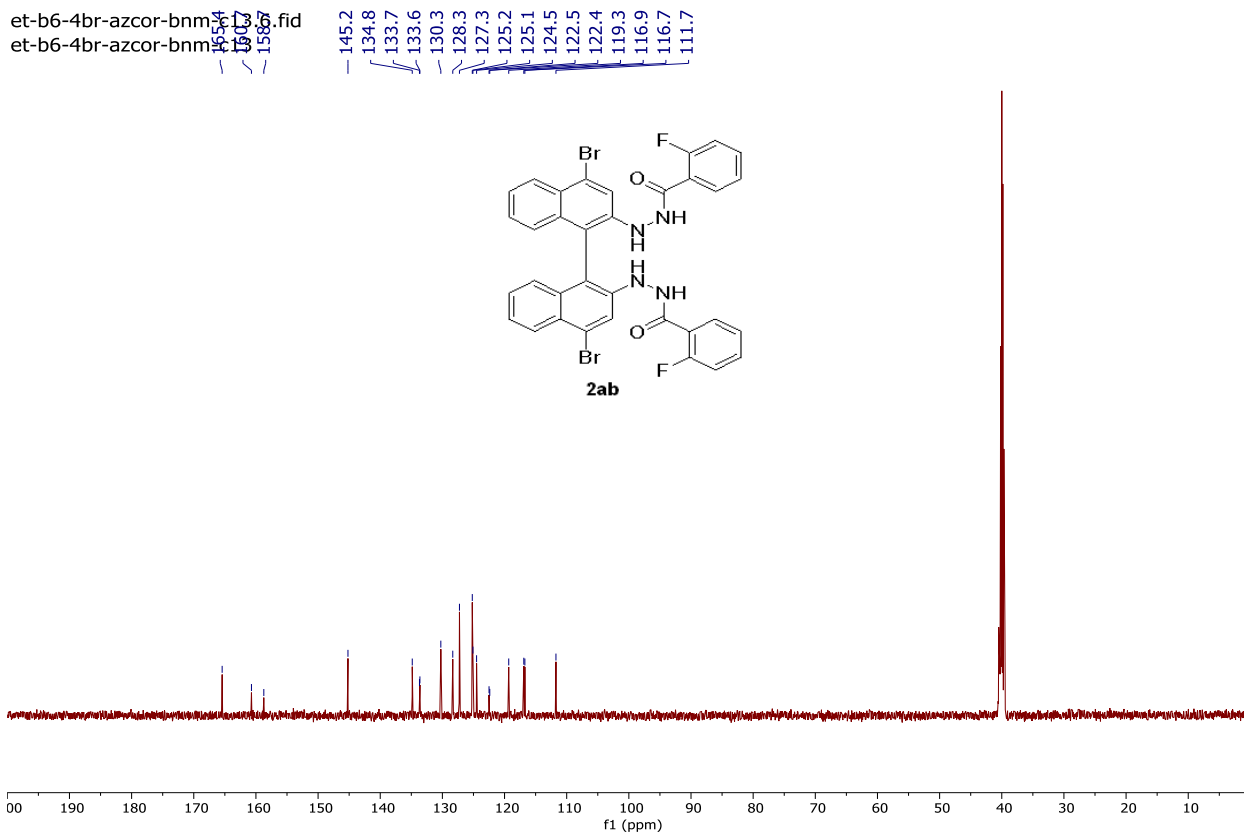
145.4
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134.1
133.5
132.9
132.2
132.1
130.0
129.6
127.2
126.2
125.2
125.1
123.3
116.4
116.1
115.6
115.3
105.0



et-b6-4br-azcor-bnm-h.6.fid
et-b6-4br-azcor-bnm-h

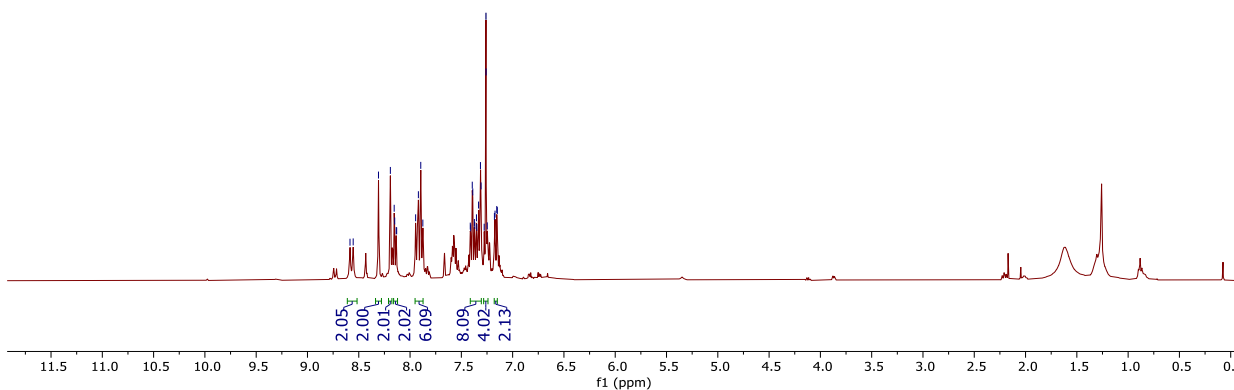
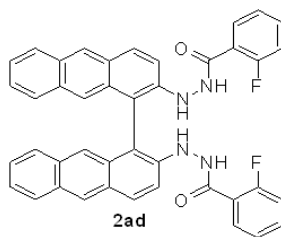


et-b6-4br-azcor-bnm-h.6.fid
et-b6-4br-azcor-bnm-h



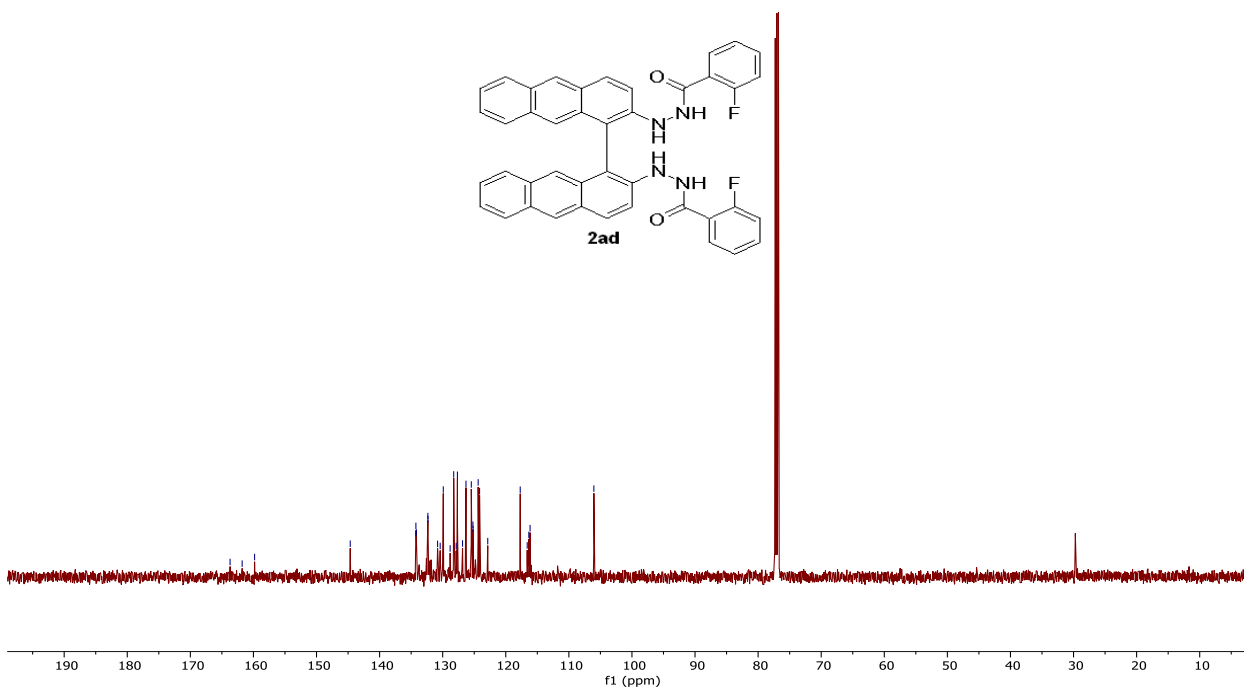
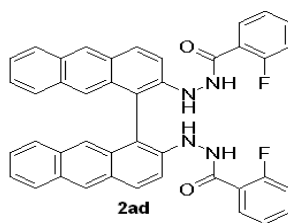
Nov06-2023-et-b6-ant-bm.10.fid
 1H NMR (400 MHz, Chloroform-d) 8.57 (d, J = 12.4 Hz, 2H), 8.31 (s, 2H), 8.19 (s, 2H), 8.14 (dd, 2H), 8.09 (dd, 2H), 7.43 - 7.30 (m, 6H), 7.28 - 7.24 (m, 5H), 7.18 (dd, 2H).

8.59
8.55
8.31
8.19
8.15
8.15
8.13
7.95
7.92
7.90
7.88
7.41
7.41
7.39
7.39
7.37
7.37
7.35
7.35
7.33
7.33
7.31
7.31
7.28
7.26 CDCl3
7.26
7.25
7.18
7.17
7.15



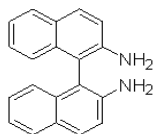
et-b6-ant-c13.6.fid
 et-b6-ant-c13

163.7
161.8
159.8
144.7
134.2
134.2
132.3
132.3
130.8
130.4
129.9
128.8
128.3
127.9
127.7
126.9
126.3
125.5
125.2
124.4
124.2
122.9
117.7
116.6
116.3
116.1
106.0

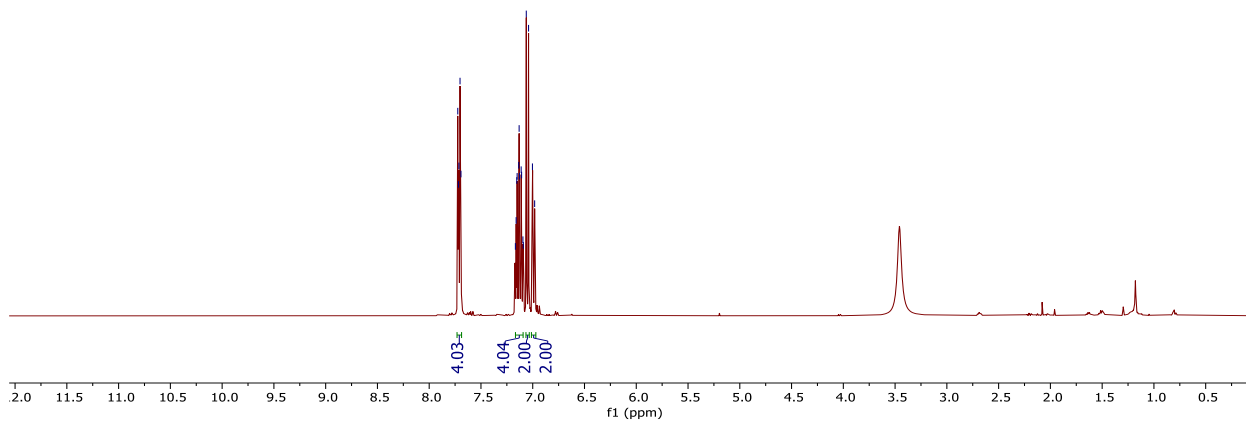


Aug23-2023-et-b6-bnmclv.10.fid
Aug23-2023-et-b6-bnmclv

7.72
7.72
7.71
7.70
7.69
7.17
7.16
7.15
7.15
7.14
7.13
7.13
7.11
7.11
7.09
7.09
7.06
7.04
7.00
6.98

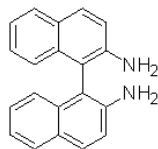


3a

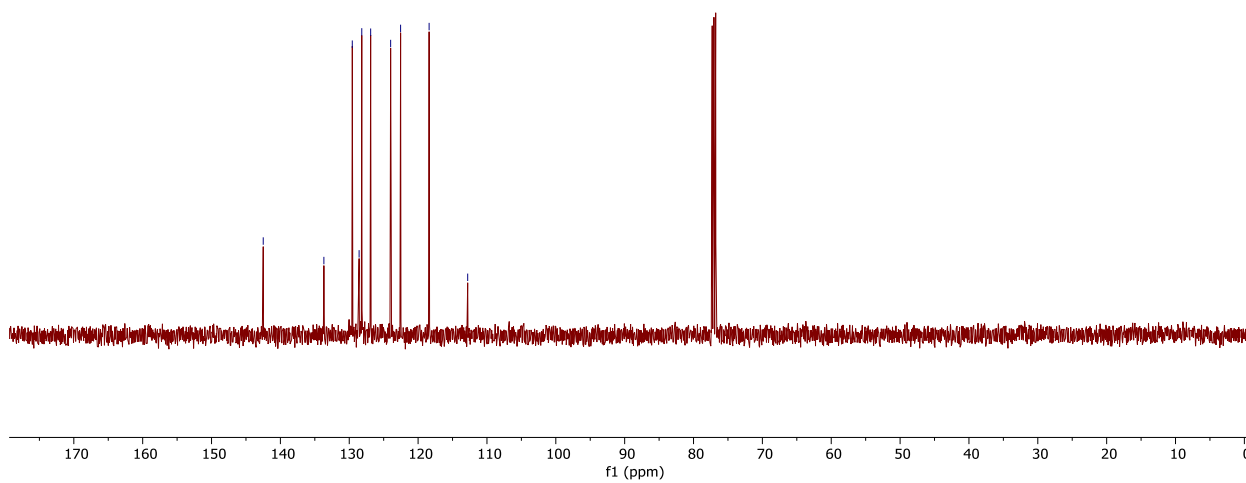


et-b6-bz-c13-clv.6.fid
et-b6-bz-c13-clv

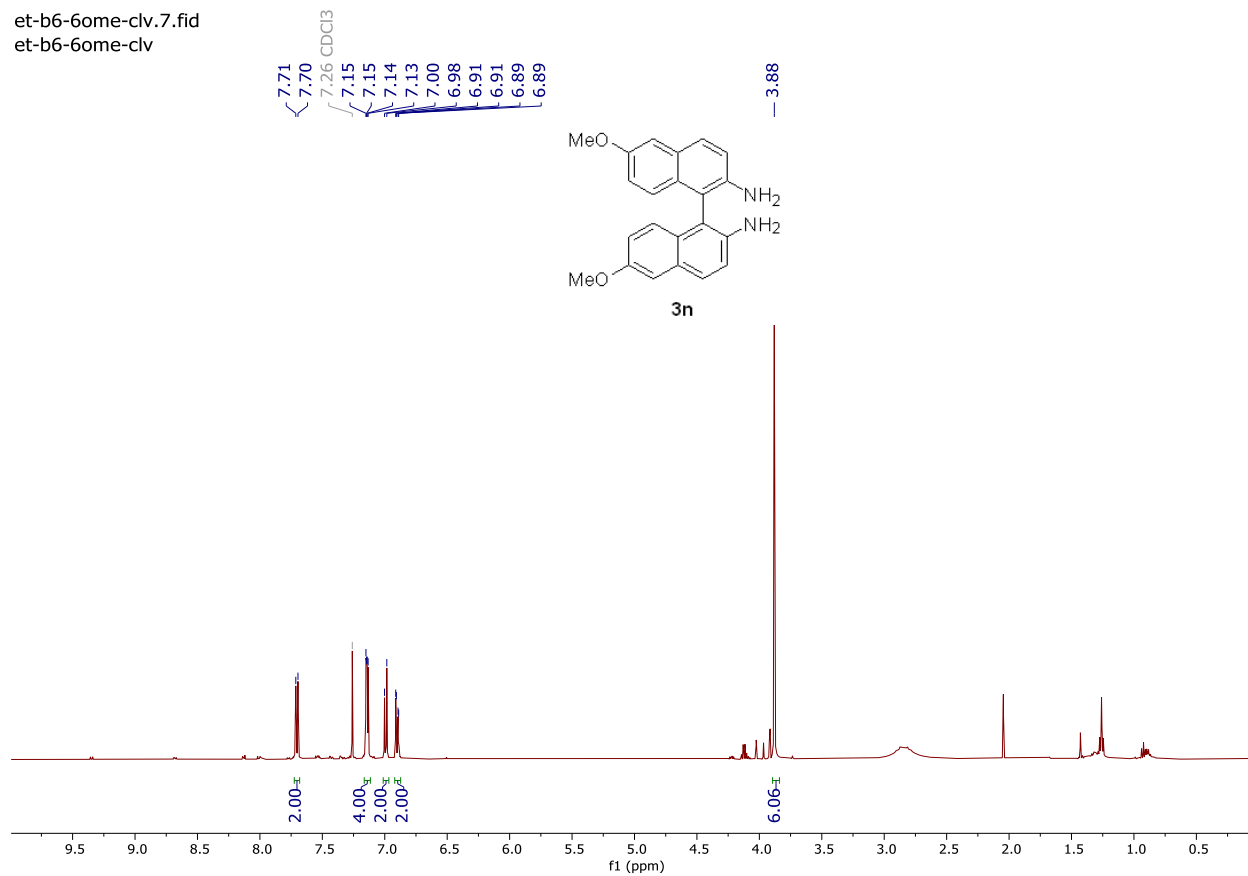
142.5
133.7
129.6
128.6
128.2
126.9
124.0
122.5
118.4
112.8



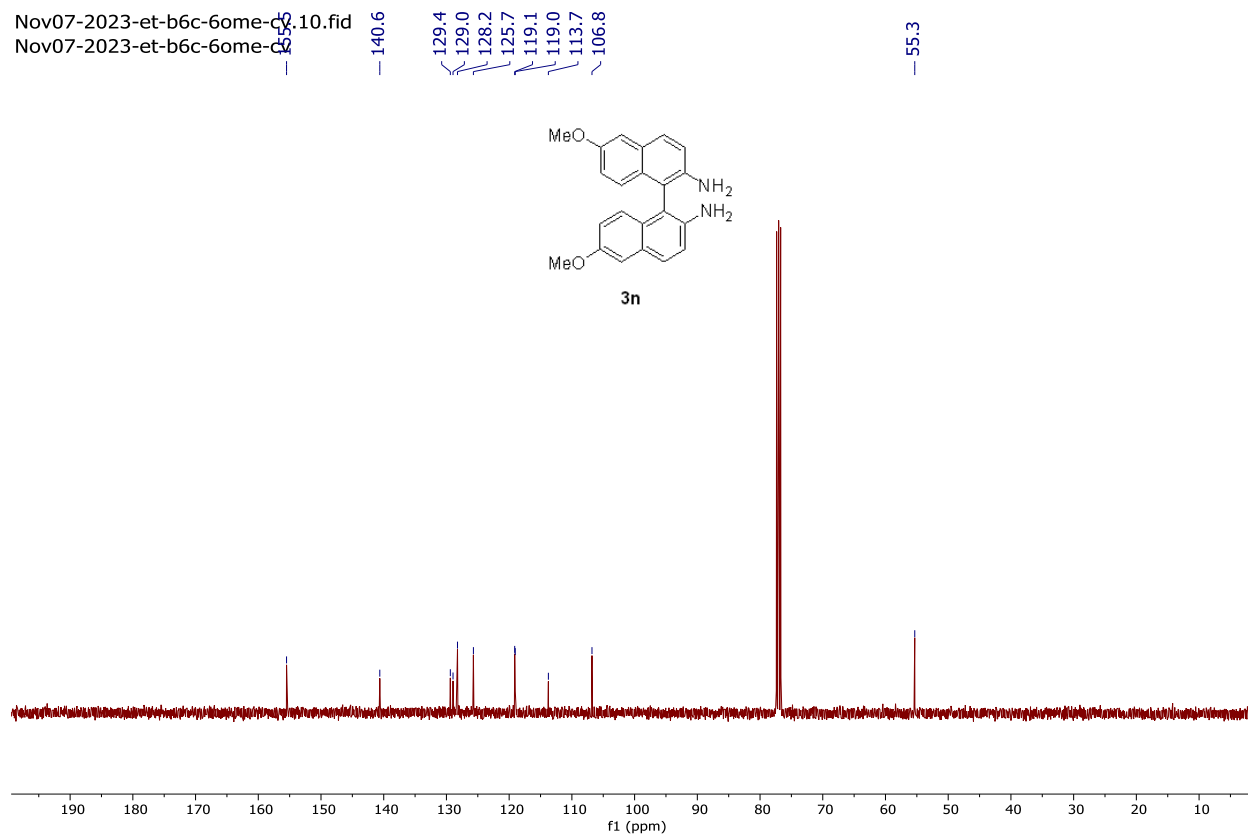
3a



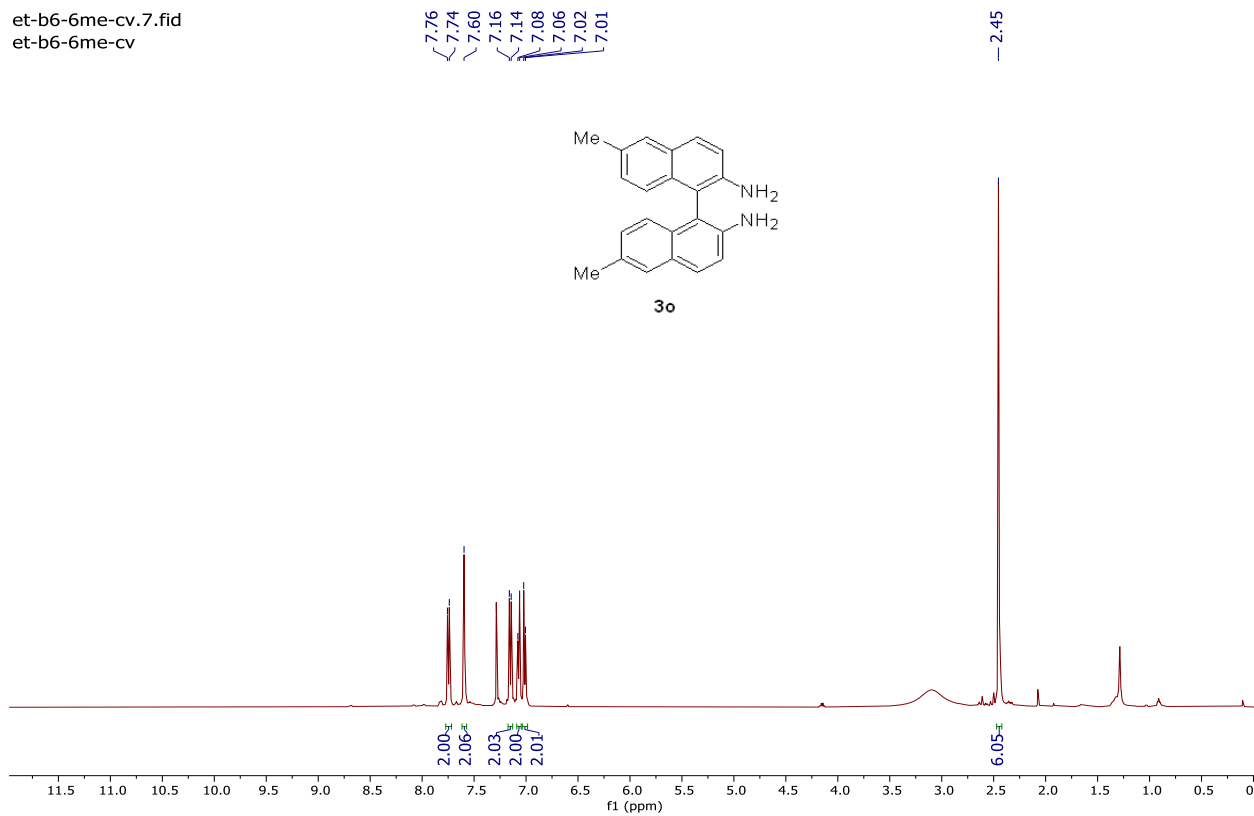
et-b6-6ome-clv.7.fid
et-b6-6ome-clv



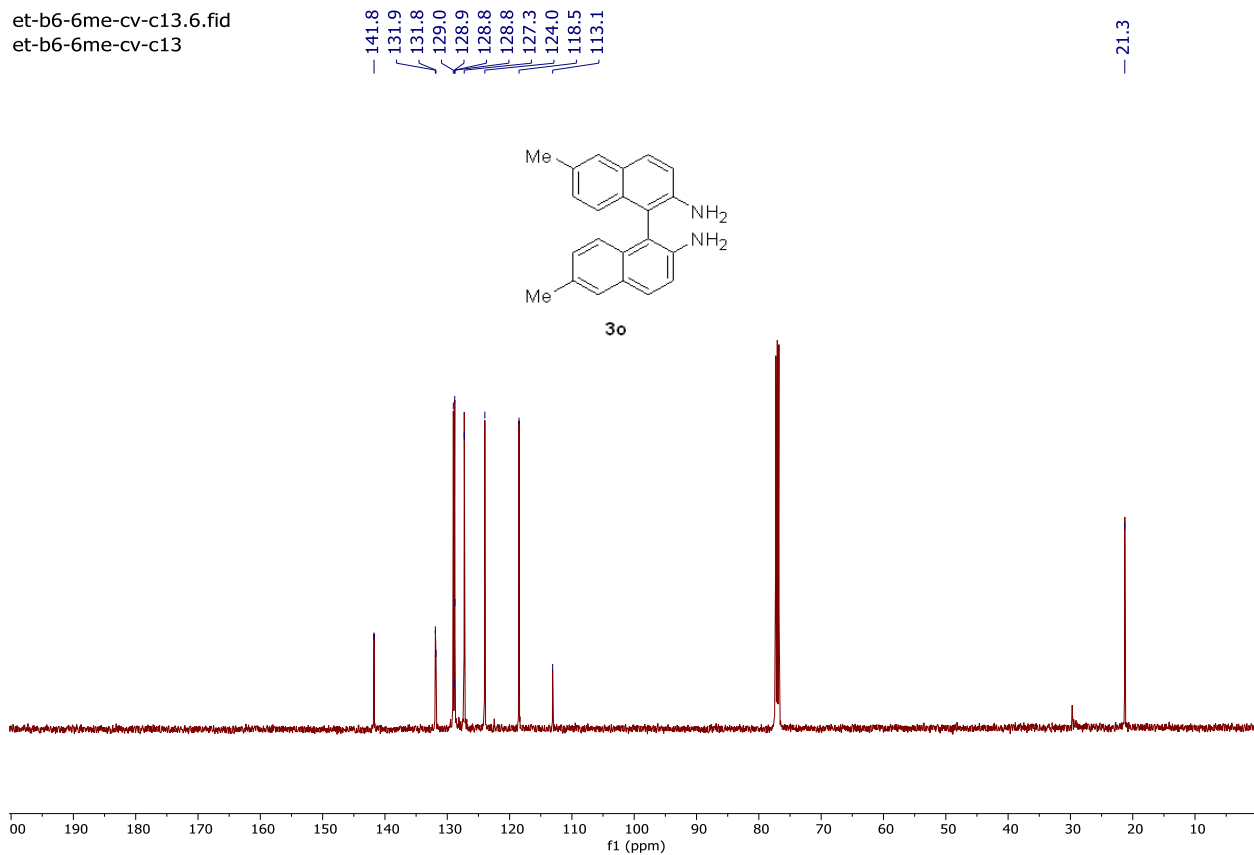
Nov07-2023-et-b6c-6ome-clv.10.fid
Nov07-2023-et-b6c-6ome-clv



et-b6-6me-cv.7.fid
et-b6-6me-cv

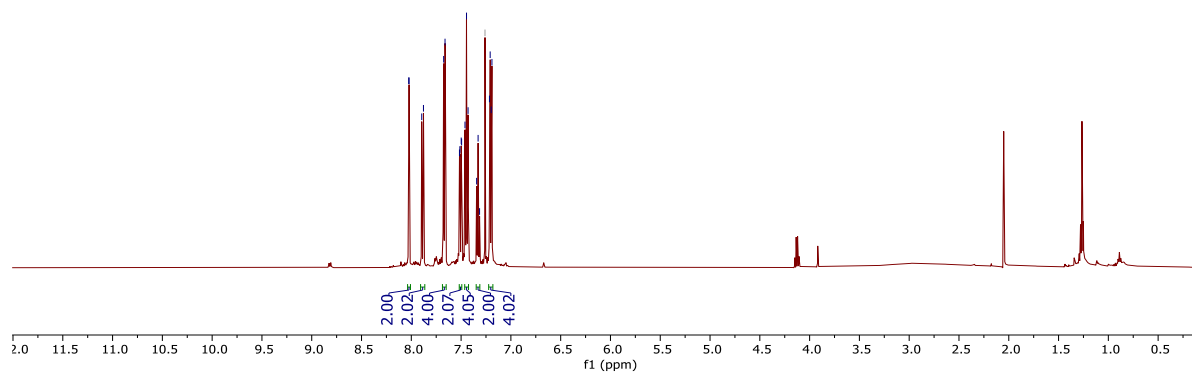
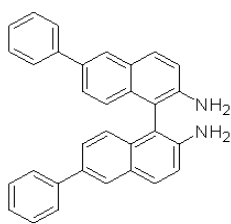


et-b6-6me-cv-c13.6.fid
et-b6-6me-cv-c13



et-b6-6ph-clv.7.fid
et-b6-6ph-clv

8.03
7.90
7.88
7.68
7.66
7.52
7.51
7.50
7.49
7.46
7.45
7.43
7.34
7.33
7.31
7.26 CDCl₃
7.22
7.21
7.20
7.19



et-b6-6ph-cv-c13.6.fid
et-b6-6ph-cv-c13

142.8
141.3
135.3
132.9
130.0
128.8
127.1
126.9
126.5
126.2
124.5
118.8
112.4

