Supporting Information

TBPB-initiated Cascade Cyclization of 3-Arylethynyl-[1,1'-biphenyl]-2-carbonitriles with Sulfinic Acids: Access to Sulfone-Containing Cyclopenta[gh]phenanthridines

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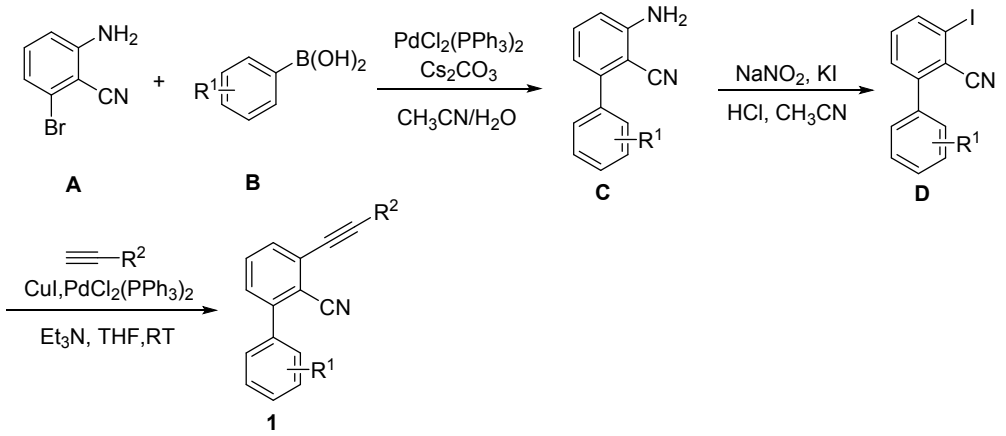
Table of Contents

General Information:

All reactions were carried out under Ar atmosphere unless otherwise noted. All catalysts and solvents were obtained from commercial suppliers. Reactions were monitored by TLC on silica gel plates (GF254), and the analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel plates. Sulfinic acids **2a−2g** were synthesized according to the literature. [1] ^{1}H NMR and ^{13}C NMR spectra were recorded on 500 MHz spectrometer at room temperature. ^{19}F NMR spectra was recorded on 400 MHz spectrometer at room temperature. Chemical shifts (δ) are reported in ppm downfield from tetramethylsilane. High resolution mass spectra were obtained on a high–resolution mass spectrometer in the ESI mode.

General Procedure for the Synthesis of 1.[2]

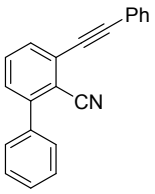


Step I: Substituted phenylboronic acid (6 mmol), 2-amino-6-bromobenzonitrile (5 mmol), $PdCl_2(PPh_3)_2$ (35.0 mg, 0.05 mmol) and Cs_2CO_3 (4.89 g, 15.0 mmol) were added in CH_3CN (20 mL) and H_2O (1.6 mL). The reaction mixture was stirred at 80 °C under argon atmosphere for 12 h. After the reaction was finished, the mixture was cooled to room temperature and extracted with EtOAc (40 × 3 mL). The combined organic phase was washed with brine (40 mL), dried over anhydrous $MgSO_4$. The solvent was removed under vacuum to give the crude products **C** without further purification.

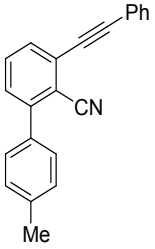
Step II: To a solution of **C**, $NaNO_2$ (3 equiv) and KI (3 equiv) in MeCN (30 mL) at 0 °C was added cold concd HCl (12 equiv) drop wise. The reaction mixture was stirred at 0 °C for 30 min. Then, the reaction was allowed to warm to room temperature and monitored by TLC. After the reaction was finished, the solvent was removed under vacuumto give the crude products **D** without further purification.

Step III: To a solution of **D**, $PdCl_2(PPh_3)_2$ (2 mol%), and CuI (2 mol%) in NEt_3 (0.25 M) was added

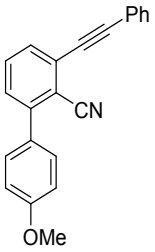
acetylene (1.2 equiv). The resulting mixture was heated under Ar atmosphere at 50 °C for 6-18 hours. After the reaction was finished, the crude mixture was purified by silica gel column chromatography to give the desired products **1**.



*3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1a**)*. Grey solid (57% yield for three steps); mp: 96-98 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.66-7.57 (m, 6H), 7.53-7.43 (m, 4H), 7.40-7.38 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.6, 138.4, 132.5, 131.1, 129.8, 129.6, 129.3, 129.2, 129.1, 128.8, 122.5, 117.5, 114.5, 96.3, 86.5 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}N$ [M+H$^+$]: 279.1048, found 279.1045.

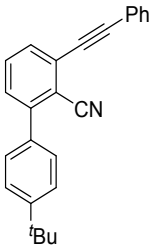


*4'-methyl-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1b**)*. Grey solid (55% yield for three steps); mp: 95-97 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.65-7.63 (m, 2H), 7.61-7.56 (m, 2H), 7.47 (d, J = 8.0 Hz, 2H), 7.44-7.42 (m, 1H), 7.40-7.37 (m, 3H), 7.31 (d, J = 8.0 Hz, 2H), 2.43 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.7, 139.3, 135.5, 132.5, 130.9, 129.9, 129.8, 129.6, 129.0, 128.833, 128.760, 122.6, 117.7, 114.4, 96.2, 86.6, 21.7 ppm. ESI-HRMS: m/z Calcd for $C_{22}H_{16}N$ [M+H$^+$]: 294.1277, found 294.1276.

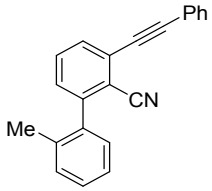


*4'-methoxy-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1c**)*. Grey solid (49% yield for three steps); mp: 88-89 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.65-7.63 (m, 2H), 7.59-7.56 (m, 2H), 7.54-7.51 (m, 2H), 7.42-7.41 (m, 1H), 7.39-7.37 (m, 3H), 7.04-7.02 (m, 2H), 3.87 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 160.6, 146.3, 132.450,

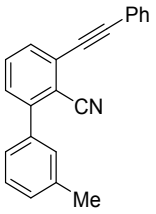
132.422, 130.7, 130.6, 130.4, 129.7, 129.6, 128.825, 128.775, 122.6, 117.8, 114.6, 114.2, 96.2, 86.6, 55.8 ppm. ESI-HRMS: m/z Calcd for $C_{22}H_{16}NO$ [M+H$^+$]: 310.1226, found 310.1228.



*4'-(tert-butyl)-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1d**)*. Grey solid (51% yield for three steps); mp: 96-98 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.66-7.64 (m, 2H), 7.60-7.56 (m, 2H), 7.54-7.51 (m, 4H), 7.45-7.43 (m, 1H), 7.40-7.36 (m, 3H), 1.38 (s, 9H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 152.4, 146.5, 133.4, 132.523, 132.465, 130.9, 129.9, 129.6, 128.8, 126.1, 122.6, 117.7, 114.2, 96.2, 86.7, 35.1, 31.7 ppm. ESI-HRMS: m/z Calcd for $C_{25}H_{22}N$ [M+H$^+$]: 336.1747, found 336.1744.

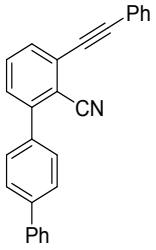


*2'-methyl-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1e**)*. Grey solid (49% yield for three steps); mp: 91-93 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.64-7.62 (m, 3H), 7.59 (d, J = 7.5 Hz, 1H), 7.34-7.28 (m, 3H), 7.23-7.21 (m, 1H), 2.22 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.9, 138.2, 136.0, 132.4, 132.2, 131.1, 130.9, 130.2, 129.7, 129.6, 129.3, 128.8, 128.1, 126.3, 122.5, 117.0, 116.0, 96.3, 86.4, 21.7 ppm. ESI-HRMS: m/z Calcd for $C_{22}H_{16}N$ [M+H$^+$]: 294.1277, found 294.1274.

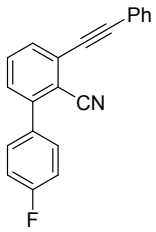


*3'-methyl-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1f**)*. Grey solid (46% yield for three steps); mp: 94-96 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.66-7.64 (m, 2H), 7.62-7.56 (m, 2H), 7.44-7.41 (m, 1H), 7.40-7.37 (m, 6H), 7.28 (d, J = 6.0 Hz, 1H), 2.45 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.8, 138.8, 138.3, 132.5, 132.4, 131.0, 129.9, 129.8, 129.6, 129.0, 128.840, 128.760, 126.3, 122.6, 117.6, 114.5, 96.3, 86.6 ppm. ESI-

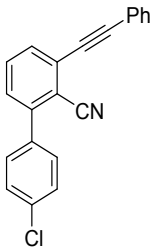
HRMS: m/z Calcd for $C_{22}H_{16}N$ [M+H$^+$]: 294.1277, found 294.1281.



*3-(phenylethynyl)-[1,1':4',1''-terphenyl]-2-carbonitrile (**1g**)*. Grey solid (46% yield for three steps); mp: 93-95 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.75-7.72 (m, 2H), 7.68-7.56 (m, 8H), 7.52-7.44 (m, 4H), 7.41-7.38 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.2, 142.2, 140.7, 137.2, 132.537, 132.479, 131.1, 129.8, 129.6, 129.3, 128.9, 128.1, 127.9, 127.6, 122.5, 117.6, 114.4, 96.4, 86.5 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{18}ClN$ [M+H$^+$]: 356.1434, found 356.1431.

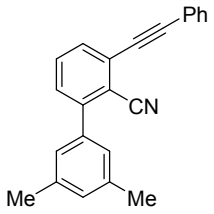


*4'-fluoro-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1h**)*. Grey solid (48% yield for three steps); mp: 92-94 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.65-7.58 (m, 4H), 7.56-7.50 (m, 2H), 7.42-7.36 (m, 4H), 7.22-7.17 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 165.9 (d, J = 249 Hz), 145.6, 134.4 (d, J = 4 Hz), 132.6, 132.5, 131.7, 131.0 (d, J = 9 Hz), 129.7, 128.905, 128.861, 122.4, 117.4, 116.2 (d, J = 42 Hz), 114.5, 96.5, 86.4 ppm. ^{19}F NMR (376 MHz, $CDCl_3$): δ -112.5 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}FN$ [M+H$^+$]: 298.1027, found 298.1031.

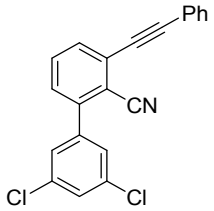


*4'-chloro-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1i**)*. Grey solid (53% yield for three steps); mp: 101-102 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.64-7.58 (m, 4H), 7.52-7.47 (m, 4H), 7.41-7.38 (m, 4H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 145.3, 136.7, 135.6, 132.6, 132.5, 131.4, 130.5, 129.7, 129.6, 129.4, 129.0, 128.9, 122.4, 117.3,

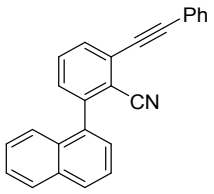
114.4, 96.6, 86.3 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}ClN$ [M+H$^+$]: 310.1226, found 310.1228.



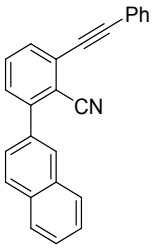
*3',5'-dimethyl-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1j**)*. Grey solid (53% yield for three steps); mp: 87-89 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.65-7.63 (m, 4H), 7.61-7.55 (m, 1H), 7.43-7.41 (m, 1H), 7.39-7.37 (m, 3H),7.18 (s, 2H), 7.10 (s, 1H), 2.40 (s, 6H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 147.0, 138.8, 138.3, 132.4, 132.3, 130.9, 130.8, 129.8, 129.6, 128.8, 128.7, 127.0, 122.6, 117.6, 114.5, 96.2, 86.7, 21.7 ppm. ESI-HRMS: m/z Calcd for $C_{23}H_{18}N$ [M+H$^+$]: 308.1434, found 308.1432.



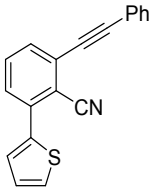
*3',5'-dichloro-3-(phenylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1k**)*. Grey solid (46% yield for three steps); mp: 105-106 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.67-7.60 (m, 4H), 7.47-7.46 (m, 1H), 7.44 (d, J = 1.5 Hz, 2H), 7.41-7.38 (m, 4H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 143.7, 141.1, 135.8, 132.7, 132.5, 132.0, 129.8, 129.422, 129.403, 129.2, 128.9, 127.7, 122.3, 116.8, 114.5, 97.1, 86.1 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{12}Cl_2N$ [M+H$^+$]: 347.0269, found 347.0266.



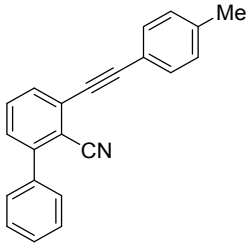
*2-(naphthalen-1-yl)-6-(phenylethynyl)benzonitrile (**1l**)*. Grey solid (52% yield for three steps); mp: 96-98 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.98-7.94 (m, 2H), 7.73-7.64 (m, 4H), 7.61-7.46 (m, 7H), 7.39-7.37 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 145.5, 136.0, 134.1, 132.5, 132.1, 131.7, 131.4, 131.2, 130.0, 129.8, 129.6, 129.0, 128.9, 128.5, 127.9, 127.1, 126.6, 125.6, 125.5, 122.5, 116.9, 116.8, 96.5, 86.4 ppm. ESI-HRMS: m/z Calcd for $C_{25}H_{16}N$ [M+H$^+$]: 330.1277, found 330.1278.



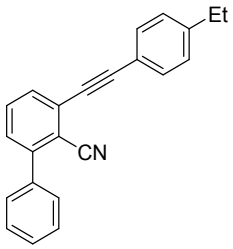
*2-(naphthalen-2-yl)-6-(phenylethynyl)benzonitrile (**1m**)*. Grey solid (52% yield for three steps); mp: 103-105 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.06-7.97 (m, 2H), 7.94-7.90 (m, 2H), 7.70-7.61 (m, 5H), 7.57-7.53 (m, 3H), 7.40-7.38 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.6, 135.7, 133.6, 133.5, 132.530, 132.486, 131.1, 130.0, 129.6, 129.0, 128.926, 128.854, 128.8, 128.2, 127.3, 127.1, 126.6, 122.5, 117.6, 114.7, 96.4, 86.6 ppm. ESI-HRMS: m/z Calcd for $C_{25}H_{16}N$ [M+H$^+$]: 330.1277, found 330.1275.



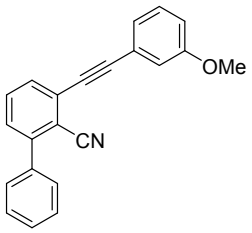
*2-(phenylethynyl)-6-(thiophen-2-yl)benzonitrile (**1n**)*. Grey solid (46% yield for three steps); mp: 95-96 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.69-7.68 (m, 1H), 7.66-7.64 (m, 2H), 7.58-7.54 (m, 3H), 7.45 (dd, J = 6 Hz, J = 1 Hz, 1H), 7.40-7.37 (m, 3H), 7.18 (dd, J = 5 Hz, J = 3.5 Hz, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 139.5, 138.7, 132.6, 132.50, 131.1, 129.7, 129.5, 129.4, 128.9, 128.7, 128.5, 127.9, 122.4, 117.7, 113.2, 96.7, 86.4 ppm. ESI-HRMS: m/z Calcd for $C_{19}H_{12}NS$ [M+H$^+$]: 286.0685, found 286.0687.



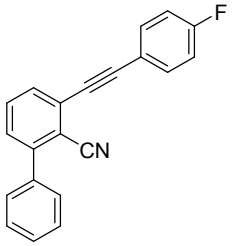
*3-(p-tolylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1o**)*. Grey solid (53% yield for three steps); mp: 94-96 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61-7.56 (m, 4H), 7.55-7.45 (m, 5H), 7.43-7.41 (m, 1H), 7.19 (d, J = 8.5 Hz, 2H), 2.38 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.6, 139.9, 138.4, 132.443, 132.378, 131.0, 129.6, 129.3, 129.2, 129.121, 129.064, 119.5, 117.6, 114.3, 96.7, 86.0, 20.0 ppm. ESI-HRMS: m/z Calcd for $C_{22}H_{16}N$ [M+H$^+$]: 294.1277, found 294.1279.



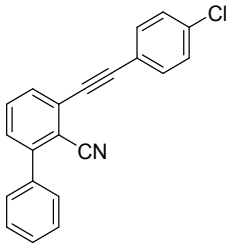
*3-((4-ethylphenyl)ethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1p**)*. Grey solid (56% yield for three steps); mp: 95-97 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61-7.55 (m, 6H), 7.52-7.46 (m, 3H), 7.43-7.41 (m, 1H), 7.21 (d, *J* = 8 Hz, 2H), 2.68 (q, *J* = 15.0 Hz, *J* = 7.5 Hz, 2H), 1.25 (t, *J* = 7.5 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.6, 146.2, 138.4, 132.5, 132.4, 131.0, 129.6, 129.3, 129.2, 129.121, 129.078, 128.4, 119.7, 117.6, 114.4, 96.7, 86.0, 29.3, 15.7 ppm. ESI-HRMS: m/z Calcd for $C_{23}H_{18}N$ [M+H$^+$]: 308.1434, found 308.1431.



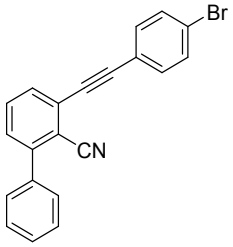
*3-((3-methoxyphenyl)ethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1q**)*. Grey solid (52% yield for three steps); mp: 99-101 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.63-7.56 (m, 4H), 7.52-7.43 (m, 4H), 7.30-7.23 (m, 2H), 7.16 (t, *J* = 8 Hz, 1H), 6.96-6.94 (m, 1H), 3.83 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 159.8, 146.6, 138.3, 132.5, 131.1, 129.930, 129.858, 129.3, 129.2, 129.1, 128.7, 125.1, 123.5, 117.5, 117.0, 116.4, 114.5, 96.3, 55.8 ppm. ESI-HRMS: m/z Calcd for $C_{22}H_{16}N$ [M+H$^+$]: 310.1226, found 310.1228.



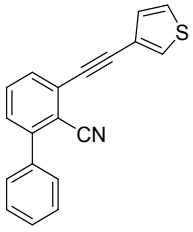
*3-((4-fluorophenyl)ethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1r**)*. Grey solid (43% yield for three steps); mp: 95-96 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.64-7.56 (m, 6H), 7.53-7.43 (m, 4H), 7.10-7.05 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 163.5 (d, *J* = 251 Hz), 146.7, 138.3, 134.5 (d, *J* = 8 Hz), 132.5, 131.0, 129.9, 129.3, 129.1, 128.7, 118.6 (d, *J* = 4 Hz), 117.5, 116.3, 116.1, 114.4, 95.3, 86.3 ppm. ^{19}F NMR (376 MHz, $CDCl_3$): *δ* -109.2 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}FN$ [M+H$^+$]: 298.1027, found 298.1025.



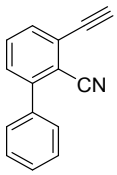
*3-((4-chlorophenyl)ethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1s**)*. Grey solid (46% yield for three steps); mp: 98-99 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61-7.56 (m, 6H), 7.52-7.44 (m, 4H), 7.35 (d, *J* = 8.5 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.7, 138.2, 135.8, 133.7, 132.6, 131.0, 130.1, 129.4, 129.244, 129.179, 129.1, 128.5, 121.0, 117.5, 114.5, 95.1, 87.5 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}ClN$ [M+H$^+$]: 314.0731, found 314.0728.



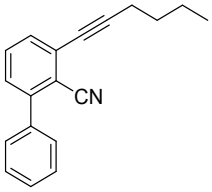
*3-((4-bromophenyl)ethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1t**)*. Grey solid (48% yield for three steps); mp: 101-103 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61-7.56 (m, 4H), 7.53-7.45 (m, 8H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.7, 138.2, 133.8, 132.5, 132.2, 131.0, 130.1, 129.4, 129.2, 129.1, 128.5, 124.1, 121.5, 117.5, 114.5, 95.1, 87.6 ppm. ESI-HRMS: m/z Calcd for $C_{21}H_{13}BrN$ [M+H$^+$]: 358.0226, found 358.0229.



*3-(thiophen-3-ylethynyl)-[1,1'-biphenyl]-2-carbonitrile (**1u**)*. Grey solid (36% yield for three steps); mp: 91-93 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.68-7.67 (m, 1H), 7.61-7.56 (m, 4H), 7.52-7.46 (m, 3H), 7.44-7.42 (m, 1H), 7.34-7.32 (d, *J* = 7.5 Hz, 1H), 7.29-7.28 (m, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.6, 138.3, 132.5, 131.0, 130.8, 130.4, 129.7, 129.3, 129.2, 129.1, 128.9, 126.0, 121.6, 117.5, 114.3, 91.5, 86.2 ppm. ESI-HRMS: m/z Calcd for $C_{19}H_{12}NS$ [M+H$^+$]: 286.0685, found 286.0687.



*3-ethynyl-[1,1'-biphenyl]-2-carbonitrile (**1v**).* Grey solid (42% yield for four steps); mp: 83-85 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61-7.57 (m, 2H), 7.55-7.53 (m, 2H), 7.51-7.44 (m, 4H), 3.50 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.7, 138.1, 132.5, 132.0, 130.6, 129.4, 129.2, 129.1, 127.5, 117.3, 115.0, 84.1, 80.5 ppm. ESI-HRMS: m/z Calcd for $C_{15}H_{10}N$ [M+H$^+$]: 204.0808, found 204.0805.

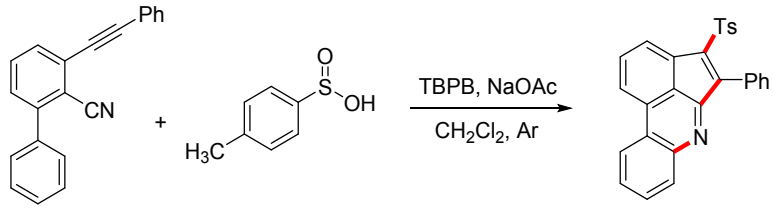


*3-(hex-1-yn-1-yl)-[1,1'-biphenyl]-2-carbonitrile (**1w**).* Grey solid (52% yield for three steps); mp: 85-86 °C; R_f = 0.52 (petroleum ether/ethyl acetate 10:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.55-7.56 (m, 3H), 7.50-7.43 (m, 4H), 7.36 (d, *J* = 7.5 Hz, 1H), 2.51 (t, *J* = 7.0 Hz, 2H), 1.69-1.63 (m, 2H), 1.57-1.50 (m, 2H), 0.96 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 146.4, 138.5, 132.4, 131.2, 129.7, 129.2, 129.150, 129.064, 117.8, 114.5, 98.4, 30.9, 22.4, 19.7, 14.0 ppm. ESI-HRMS: m/z Calcd for $C_{19}H_{18}N$ [M+H$^+$]: 260.1434, found 260.1435.

General Procedure for Synthesis of Products 3.

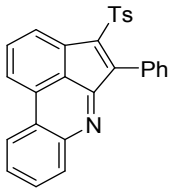
An oven-dried Schlenk tube (10 mL) was equipped with a magnetic stir bar, **1** (0.1 mmol), **2** (2 equiv, 0.2 mmol), NaOAc (2 equiv, 0.2 mmol). The flask was evacuated and backfilled with Ar for 3 times. Then 1 mL dichloromethane was added followed by TBPB (18 mg, 28 μL, 2.0 equiv). The tube was then sealed and the mixture was stirred for 24 h at 80 °C under Argon (1 atm). After the reaction was finished, the solvent was concentrated in vacuo and the residue was purified by chromatography on silica gel to afford the corresponding products **3**.

Experimental Procedure for Scale-up Reaction

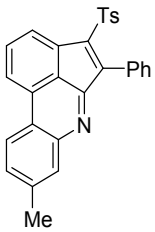


1a (1 mmol) **2a** (2 mmol) **3aa**

An oven-dried Schlenk tube (50 mL) was equipped with a magnetic stir bar, **1a** (1 mmol), **2a** (2 equiv, 2 mmol), NaOAc (2 equiv, 2 mmol). The flask was evacuated and backfilled with Ar for 3 times. Then 10 mL dichloromethane was added followed by TBPB (180 mg, 280 μL, 2.0 equiv). The tube was then sealed and the mixture was stirred for 24 h at 80 °C under Argon (1 atm). After the reaction was finished, the solvent was concentrated in vacuo and the residue was purified by chromatography on silica gel to afford the corresponding products **3aa** in 58% yield.

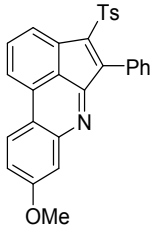


*5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3aa**)*. Yellow solid; (29.4 mg, 68%); mp: 195-197 °C; R_f = 0.36 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.53 (dd, *J* = 6.0 Hz, *J* = 3.0 Hz, 1H), 8.38-8.34 (m, 2H), 8.30 (dd, *J* = 6.5 Hz, *J* = 3.5 Hz, 1H), 7.88-7.85 (m, 1H), 7.76-7.73 (m, 2H), 7.68-7.66 (m, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.53-7.48 (m, 3H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.8, 148.7, 148.6, 144.7, 143.2, 138.9, 135.7, 133.3, 132.8, 131.6, 130.2, 129.9, 129.8, 129.4, 129.3, 129.1, 128.1, 128.0, 125.8, 124.9, 123.6, 123.3, 120.2, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}NO_2S$ [M+H$^+$]: 434.1209, found 434.1204.

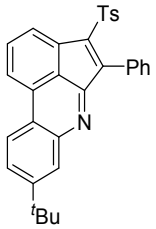


*8-methyl-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ba**)*. Yellow solid; (27.3 mg, 61%); mp: 184-186 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.42 (d, *J* = 8.0 Hz, 1H), 8.35-8.31 (m, 2H), 8.11 (s, 1H), 7.84 (t, *J* = 8.0 Hz, 2H), 7.66-7.65 (m, 2H), 7.59-7.56 (m, 3H), 7.52-7.46 (m, 3H), 7.11 (d, *J* = 8.5 Hz, 2H), 2.57 (s, 3H), 2.32 (s, 3H) ppm; ^{13}C NMR

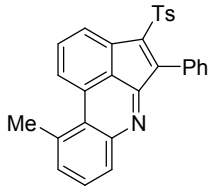
(126 MHz, $CDCl_3$): δ 161.7, 148.9, 148.7, 144.6, 143.0, 139.6, 139.0, 135.7, 133.2, 132.4, 131.5, 130.9, 130.3, 129.9, 129.8, 129.4, 128.046, 127.995, 125.4, 123.5, 123.0, 122.6, 119.9, 21.9 ppm. ESI-HRMS: m/z Calcd for $C_{26}H_{18}NO_2S_2$ [M+H$^+$]: 448.1366, found 448.1362.



*8-methoxy-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ca**)*. Yellow solid; (24.5 mg, 53%); mp: 188-190 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.40 (d, *J* = 9.0 Hz, 1H), 8.29-8.27 (m, 2H), 7.82 (t, *J* = 7.5 Hz, 1H), 7.70 (d, *J* = 2.5 Hz, 1H), 7.67-7.65 (m, 2H), 7.57 (d, *J* = 9.5 Hz, 2H), 7.52-7.48 (m, 3H), 7.39-7.36 (m, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 3.94 (s, 3H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 162.1, 160.5, 150.5, 148.6, 144.6, 142.9, 139.0, 135.6, 133.3, 131.5, 130.3, 129.9, 129.8, 129.5, 128.1, 128.0, 125.1, 124.2, 123.4, 120.4, 119.3, 119.1, 112.4, 56.0, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{29}H_{22}NO_3S$ [M+H$^+$]: 464.1315, found 464.1317.

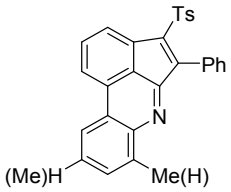


*8-(tert-butyl)-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3da**)*. Yellow solid; (26.9 mg, 55%); mp: 192-194 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.47 (d, *J* = 8.5 Hz, 1H), 8.37-8.30 (m, 3H), 7.87-7.83 (m, 2H), 7.69-7.67 (m, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 7.52-7.48 (m, 3H), 7.11 (d, *J* = 8.5 Hz, 2H), 2.32 (s, 3H), 1.44 (s, 9H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.7, 152.8, 148.9, 148.7, 144.6, 142.9, 139.0, 135.6, 133.2, 131.6, 130.3, 129.9, 129.8, 129.3, 128.9, 128.1, 128.0, 127.6, 125.4, 123.6, 122.9, 122.6, 120.1, 35.6, 31.7, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{32}H_{28}NO_2S$ [M+H$^+$]: 490.1835, found 490.1837.

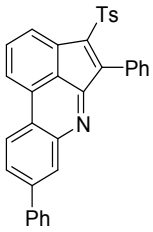


*10-methyl-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ea**)*. Yellow solid; (19.2 mg, 43%);

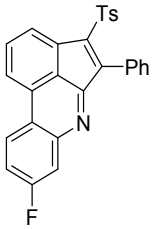
mp: 193-195 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.65 (d, *J* = 9.0 Hz, 1H), 8.40 (d, *J* = 7.0 Hz, 1H), 8.21 (d, *J* = 7.5 Hz, 1H), 7.87 (t, *J* = 8.0 Hz, 1H), 7.68-7.66 (m, 2H), 7.64 (t, *J* = 7.5 Hz, 2H), 7.59-7.57 (m, 3H), 7.52-7.48 (m, 3H), 7.11 (d, *J* = 8.5 Hz, 2H), 3.10 (s, 3H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.2, 149.8, 148.0, 144.6, 142.8, 139.0, 136.7, 135.7, 132.9, 132.7, 132.0, 131.7, 131.6, 130.6, 130.3, 129.8, 128.5, 128.2, 128.017, 127.966, 125.1, 124.2, 120.6, 25.9, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{29}H_{22}NO_2S$ [M+H$^+$]: 448.1366, found 448.1363.



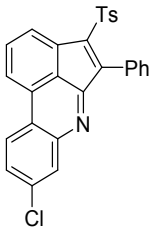
*7-methyl-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3fa**) and 9-methyl-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3fa'**)*. Yellow solid; (13.4 mg, 36%); mp: 181-183 °C; R_f = 0.36 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.41-8.30 (m, 3H), 8.19 (d, *J* = 8.0 Hz, 1H), 7.86-7.83 (m, 1H), 7.79-7.77 (m, 1H), 7.67-7.63 (m, 2H), 7.60-7.56 (m, 2H), 7.51-7.48 (m, 3H), 7.10 (d, *J* = 8.5 Hz, 2H), 2.72 (d, *J* = 72.5 Hz, 3H), 2.31 (d, *J* = 3.5 Hz, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 160.8, 148.7, 147.1, 144.6, 142.6, 139.7, 139.1, 139.0, 135.8, 135.6, 133.0, 132.5, 132.0, 131.6, 131.1, 130.4, 130.2, 129.9, 129.8, 129.1, 128.8, 128.046, 127.966, 127.9, 127.8, 125.6, 125.5, 124.9, 123.7, 123.5,123.0, 121.1, 120.3, 22.4, 21.9, 19.0 ppm. ESI-HRMS: m/z Calcd for $C_{29}H_{22}NO_2S$ [M+H$^+$]: 448.1366, found 448.1368.



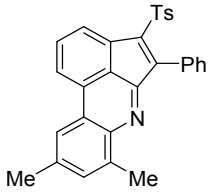
*5,8-diphenyl-4-tosylcyclopenta[gh]phenanthridine (**3ga**)*. Yellow solid; (23.4 mg, 46%); mp: 190-191 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.60-8.56 (m, 2H), 8.40-8.34 (m, 2H), 8.04-8.02 (m, 1H), 7.88 (t, *J* = 7.5 Hz, 1H), 7.76 (d, *J* = 7.5 Hz, 2H), 7.69-7.67 (m, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.52-7.47 (m, 5H), 7.41-7.38 (m, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 162.2, 149.1, 148.6, 144.7, 143.3, 142.1, 140.2, 138.9, 135.8, 133.4, 131.6, 130.6, 130.2, 130.0, 129.8, 129.4, 129.3, 128.327, 128.262, 128.1, 128.0, 127.8, 125.8, 123.9, 123.8, 123.7, 120.3, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{34}H_{24}NO_2S$ [M+H$^+$]: 510.1522, found 510.1525.



*8-fluoro-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ha**).* Yellow solid; (21.6 mg, 48%); mp: 178-179 °C; R_f = 0.34 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.51-8.48 (m, 1H), 8.34-8.30 (m, 2H), 7.97-7.95 (m, 1H), 7.86 (d, *J* = 7.5 Hz, 2H), 7.65 (d, *J* = 6.5 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.53-7.49 (m, 4H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 163.0, 162.9 (d, *J* = 249 Hz), 149.9 (d, *J* = 12 Hz), 148.3, 144.8, 143.7, 138.7, 135.8, 133.7, 131.5, 130.0, 129.8, 129.3, 128.1, 128.0, 125.8, 125.0 (d, *J* = 10 Hz), 123.5, 121.6, 119.8, 118.2 (d, *J* = 24 Hz), 117.3 (d, *J* = 21 Hz), 22.0 ppm; ^{19}F NMR (376 MHz, $CDCl_3$): *δ* -110.7 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}FNO_2S$ [M+H$^+$]: 452.1115, found 452.1117.

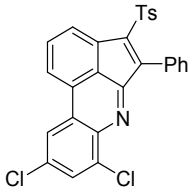


*8-chloro-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ia**).* Yellow solid; (25.7 mg, 55%); mp: 193-194 °C; R_f = 0.33 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.44 (d, *J* = 9.0 Hz, 1H), 8.34 (d, *J* = 7.0 Hz, 2H), 8.32-8.30 (m, 3H), 7.87 (t, *J* = 8.0 Hz, 1H), 7.71-7.68 (m, 1H), 7.65-7.63 (m, 2H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.52-7.47 (m, 3H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 162.9, 149.2, 148.3, 144.8, 143.8, 138.7, 135.8, 135.0, 133.8, 131.8, 131.5, 130.1, 130.0, 129.8, 129.5, 129.1, 128.1, 128.0, 126.2, 124.4, 123.5, 123.4, 120.2, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}ClNO_2S$ [M+H$^+$]: 468.0820, found 468.0823.

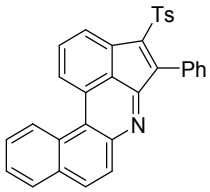


*7,9-dimethyl-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ja**).* Yellow solid; (23.0 mg, 50%); mp: 179-180 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.35-8.29 (m, 2H), 8.16 (s, 1H), 7.82 (t, *J* = 7.5 Hz, 1H), 7.79-7.77 (m, 2H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.49-7.46 (m, 4H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.74 (s, 3H), 2.59 (s, 3H), 2.31 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 159.1, 148.8, 145.9, 144.5, 141.6, 140.5, 139.2, 135.7, 132.7, 132.032,

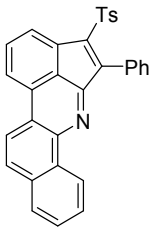
131.988, 130.6, 129.8, 129.7, 129.3, 127.9, 127.8, 125.4, 124.8, 123.6, 120.8, 120.0, 22.4, 21.9, 18.9 ppm. ESI-HRMS: m/z Calcd for $C_{30}H_{24}NO_2S$ [M+H$^+$]: 462.1522, found 462.1520.



*7,9-dichloro-5-phenyl-4-tosylcyclopenta[gh]phenanthridine (**3ka**)*. Yellow solid; (17.5 mg, 35%); mp: 208-209 ^{o}C; R_f = 0.34 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.40-8.37 (m, 2H), 8.27 (d, *J* = 8.5 Hz, 1H), 7.90-7.85 (m, 1H), 7.79-7.77 (m, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.51-7.46 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.31 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 162.2, 148.3, 144.9, 143.5, 143.4, 138.6, 138.1, 136.1, 134.7, 134.0, 132.0, 130.229, 130.205, 129.8, 129.7, 128.5, 128.0, 127.1, 127.0, 123.5, 121.9, 120.9, 21.9 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{17}Cl_2NO_2S$ [M+H$^+$]: 501.0357, found 501.0356.

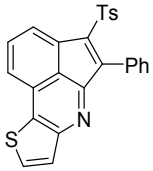


*5-phenyl-4-tosylbenzo[a]cyclopenta[gh]phenanthridine (**3la**)*. Yellow solid; (27.0 mg, 56%); mp: 187-188 ^{o}C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 9.15 (d, *J* = 8.5 Hz, 1H), 8.95 (d, *J* = 9.0 Hz, 1H), 8.44 (d, *J* = 7.0 Hz, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 8.04-8.01 (m, 2H), 7.93 (t, *J* = 7.5 Hz, 1H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.72-7.69 (m, 3H), 7.79 (d, *J* = 8.5 Hz, 1H), 7.51-7.51 (m, 3H), 7.12 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.1, 149.0, 148.5, 144.6, 141.8, 139.1, 136.1, 134.3, 133.5, 131.7, 131.0, 130.6, 130.313, 130.262, 129.9, 129.8, 129.4, 129.0, 128.7, 128.1, 128.0, 127.8, 127.6, 126.0, 122.6, 120.9, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{32}H_{22}NO_2S$ [M+H$^+$]: 484.1366, found 484.1368.

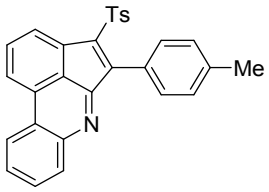


*5-phenyl-4-tosylbenzo[c]cyclopenta[gh]phenanthridine (**3ma**)*. Yellow solid; (25.1 mg, 52%); mp: 186-187 ^{o}C; R_f = 0.34 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 9.27-9.25 (m, 1H), 8.46-8.38 (m, 3H), 8.05 (d, *J* = 8.5 Hz, 1H), 7.96-7.92 (m, 1H), 7.88-7.83 (m, 3H),

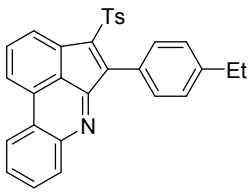
7.69-7.65 (m, 2H), 7.62 (d, *J* = 8.5 Hz, 2H), 7.58-7.55 (m, 3H), 7.12 (d, *J* = 8.5 Hz, 2H), 2.31 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 159.6, 149.1, 145.8, 144.6, 141.3, 139.2, 135.8, 133.6, 133.2, 133.1, 132.0, 130.6, 130.3, 129.9, 129.8, 129.4, 128.1, 128.0, 127.9, 127.7, 126.4, 125.6, 124.3, 122.7, 120.7, 120.3, 21.9 ppm. ESI-HRMS: m/z Calcd for $C_{32}H_{22}NO_2S$ [M+H$^+$]: 484.1366, found 484.1368.



*5-phenyl-4-tosylcyclopenta[ij]thieno[3,2-c]isoquinoline (**3na**).* Yellow solid; (16.7 mg, 38%); mp: 177-179 ^{o}C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.38 (d, *J* = 7.0 Hz, 1H), 8.05 (d, *J* = 8.5 Hz, 1H), 7.82 (t, *J* = 8.0 Hz, 1H), 7.79-7.72 (m, 2H), 7.63 (d, *J* = 7.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.51-7.47 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.31(s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 159.7, 155.9, 149.8, 144.5, 140.2, 139.2, 136.0, 133.4, 131.4, 131.1, 130.4, 130.0, 129.8, 128.2, 128.1, 127.9, 127.6, 127.4, 126.7, 124.5, 119.1, 21.9 ppm. ESI-HRMS: m/z Calcd for $C_{26}H_{16}NO_2S_2$ [M+H$^+$]: 440.0773, found 440.0776.

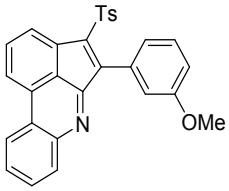


*5-(p-tolyl)-4-tosylcyclopenta[gh]phenanthridine (**3oa**).* Yellow solid; (30.4 mg, 68%); mp: 197-199 ^{o}C; R_f = 0.33 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.52-8.50 (m, 1H), 8.34-8.29 (m, 3H), 7.85-7.82 (m, 1H), 7.75-7.71 (m, 2H), 7.62 (d, *J* = 8.0 Hz, 4H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 2.48 (s, 1H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.9, 148.9, 148.6, 144.6, 142.4, 140.2, 139.1, 135.8, 133.2, 132.8, 131.6, 129.8, 129.4, 129.2, 129.1, 128.9, 128.0, 127.3, 125.5, 124.9, 123.4, 123.3, 120.3, 22.047, 21.967 ppm. ESI-HRMS: m/z Calcd for $C_{29}H_{22}NO_2S$ [M+H$^+$]: 448.1366, found 448.1365.

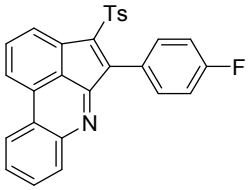


*5-(4-ethylphenyl)-4-tosylcyclopenta[gh]phenanthridine (**3pa**).* Yellow solid; (30.9 mg, 67%); mp: 195-196 ^{o}C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.57-

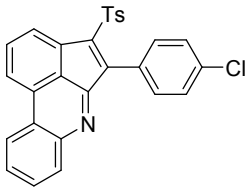
8.55 (m, 1H), 8.39-8.33 (m, 3H), 7.90-7.86 (m, 1H), 7.80-7.75 (m, 2H), 7.66-7.60 (m, 4H), 7.35 (d, J = 10.5 Hz, 2H), 7.13 (d, J = 10.5 Hz, 2H), 2.80 (q, J = 19.5 Hz, J = 9.5 Hz, 2H), 2.34 (s, 3H), 1.37 (t, J = 9.5 Hz , 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.9, 148.8, 148.7, 146.4, 144.5, 142.6, 139.0, 135.9, 133.3, 132.8, 131.7, 129.7, 129.4, 129.2, 129.1, 128.0, 127.7, 127.5, 125.6, 125.0, 123.4, 123.3, 120.2, 29.3, 21.9, 15.9 ppm. ESI-HRMS: m/z Calcd for $C_{30}H_{24}NO_2S$ [M+H$^+$]: 462.1522, found 462.1524.



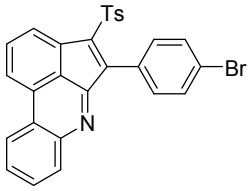
*5-(3-methoxyphenyl)-4-tosylcyclopenta[gh]phenanthridine (**3qa**)*. Yellow solid; (26.8 mg, 58%); mp: 192-193 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.53-8.51 (m, 1H), 8.37-8.35 (m, 2H), 8.32-8.30 (m, 1H), 7.86 (t, J = 7.5 Hz, 1H), 7.75-7.73 (m, 2H), 7.59 (d, J = 8.5 Hz, 2H), 7.39 (t, J = 8.0 Hz, 1H), 7.21 (d, J = 9.0 Hz, 2H), 7.12 (d, J = 8.5 Hz, 2H), 7.05 (d, J = 8.5 Hz, 1H), 3.86 (s, 3H), 2.32 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.7, 159.2, 148.6, 148.3, 144.7, 143.4, 138.8, 135.7, 133.3, 132.8, 131.4, 129.8, 129.4, 129.3, 129.2, 128.0, 125.9, 125.0, 124.0, 123.7, 123.3, 120.1, 116.6, 116.2, 55.7, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{29}H_{22}NO_3S$ [M+H$^+$]: 464.1315, found 464.1318.



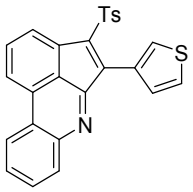
*5-(4-fluorophenyl)-4-tosylcyclopenta[gh]phenanthridine (**3ra**)*. Yellow solid; (25.3 mg, 56%); mp: 195-196 °C; R_f = 0.36 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.55-8.53 (m, 1H), 8.39-8.33 (m, 2H), 8.32-8.30 (m, 1H), 7.87 (t, J = 7.5 Hz, 1H), 7.78-7.76 (m, 2H), 7.73-7.70 (m, 2H), 7.58 (d, J = 8.0 Hz, 2H), 7.20 (t, J = 8.0 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 2.33 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 164.1 (d, J = 250 Hz), 161.5, 148.6, 147.4, 144.9, 143.2, 138.8, 133.7, 135.6, 133.8 (d, J = 8 Hz), 133.4, 132.7, 129.9, 129.5, 129.3 (d, J = 17 Hz), 127.9, 126.2, 125.8, 125.0, 123.7, 123.4, 120.1, 115.3 (d, J = 17 Hz), 22.0 ppm; ^{19}F NMR (376 MHz, $CDCl_3$): δ -111.0 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}FNO_2S$ [M+H$^+$]: 452.1115, found 452.1117.



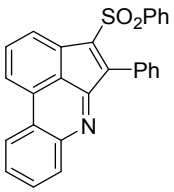
*5-(4-chlorophenyl)-4-tosylcyclopenta[gh]phenanthridine (**3sa**)*. Yellow solid; (24.7 mg, 53%); mp: 199-201 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.55-8.52 (m, 1H), 8.39-8.29 (m, 3H), 7.87 (t, *J* = 8.0 Hz, 1H), 7.77-7.75 (m, 2H), 7.64 (d, *J* = 7.0 Hz, 2H), 7.60 (d, *J* = 7.5 Hz, 2H), 7.47 (d, *J* = 7.0 Hz, 2H), 7.15 (d, *J* = 7.5 Hz, 2H), 2.33 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.4, 148.6, 147.1, 145.0, 143.4, 138.8, 136.3, 135.5, 133.4, 133.0, 132.7, 131.9, 130.8, 130.0, 129.4, 129.3, 128.4, 128.0, 125.9, 125.0, 123.8, 123.4, 120.1, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}ClNO_2S$ [M+H$^+$]: 468.0820, found 468.0818.



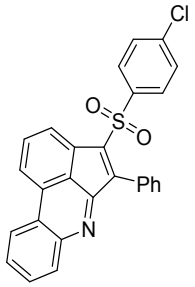
*5-(4-bromophenyl)-4-tosylcyclopenta[gh]phenanthridine (**3ta**)*. Yellow solid; (25.6 mg, 50%); mp: 206-207 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.55-8.53 (m, 1H), 8.39-8.32 (m, 2H), 8.31-8.29 (m, 1H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.77-7.75 (m, 2H), 7.64-7.56 (m, 6H), 7.15 (d, *J* = 7.5 Hz, 2H), 2.34 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.3, 148.6, 147.1, 145.0, 143.4, 138.8, 135.5, 133.4, 133.2, 132.7, 131.4, 130.0, 129.5, 129.4, 129.3, 129.1, 128.0, 125.9, 125.0, 124.7, 123.8, 123.4, 120.1, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{28}H_{19}BrNO_2S$ [M+H$^+$]: 512.0314, found 512.0317.



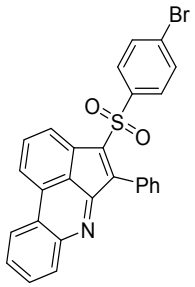
*5-(thiophen-3-yl)-4-tosylcyclopenta[gh]phenanthridine (**3ua**)*. Yellow solid; (21.1 mg, 48%); mp: 185-186 °C; R_f = 0.34 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.51-8.49 (m, 1H), 8.35-8.29 (m, 4H), 7.85-7.80 (m, 2H), 7.77-7.72 (m, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 7.44-7.42 (m, 1H), 7.14 (d, *J* = 8.5 Hz, 2H), 2.31 (s, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.6, 148.4, 144.7, 142.7, 140.9, 138.9, 136.2, 133.4, 132.7, 131.7, 131.2, 130.3, 129.9, 129.4, 129.3, 129.1, 127.6, 125.7, 125.0, 124.9, 123.3, 120.2, 22.0 ppm. ESI-HRMS: m/z Calcd for $C_{26}H_{18}NO_2S_2$ [M+H$^+$]: 440.0773, found 440.0776.



*5-phenyl-4-(phenylsulfonyl)cyclopenta[gh]phenanthridine (**3ab**)*. Yellow solid; (26.8 mg, 64%); mp: 193-194 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.55-8.51 (m, 1H), 8.38-8.36 (m, 2H), 8.32-8.28 (m, 1H), 7.87 (t, *J* = 8.0 Hz, 1H), 7.66-7.63 (m, 2H), 7.69-7.65 (m, 4H), 7.51-7.47 (m, 3H), 7.47-7.43 (m, 1H), 7.31 (t, *J* = 8.0 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.7, 149.0, 148.7, 142.9, 141.7, 135.7, 133.7, 133.3, 132.8, 131.6, 130.2, 130.0, 129.5, 129.331, 129.259, 129.2, 129.1, 128.1, 127.9, 125.8, 125.0, 123.7, 123.3, 120.1 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{18}NO_2S$ [M+H$^+$]: 420.1053, found 420.1057.

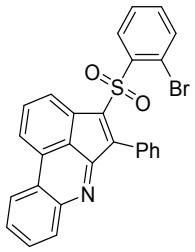


*4-((4-chlorophenyl)sulfonyl)-5-phenylcyclopenta[gh]phenanthridine (**3ac**)*. Yellow solid; (24.9 mg, 55%); mp: 183-185 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.57-8.55 (m, 1H), 8.41-8.35 (m, 2H), 8.33-8.31 (m, 1H), 7.89 (t, *J* = 8.0 Hz, 1H), 7.78-7.76 (m, 2H), 7.65-7.63 (m, 2H), 7.56 (d, *J* = 9.0 Hz, 2H), 7.53-7.48 (m, 3H), 7.25 (d, *J* = 9.0 Hz, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.4, 149.3, 148.7, 142.6, 140.3, 140.1, 135.4, 133.4, 132.9, 131.5, 130.2, 130.1, 129.6, 129.403, 129.367, 129.3, 128.2, 125.8, 125.0, 123.9, 123.4, 120.1 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{17}ClNO_2S$ [M+H$^+$]: 454.0663, found 454.0661.

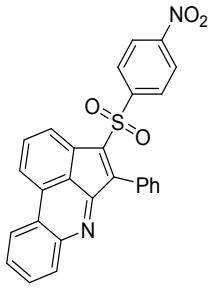


*4-((4-bromophenyl)sulfonyl)-5-phenylcyclopenta[gh]phenanthridine (**3ad**)*. Yellow solid; (26.3 mg, 53%); mp: 185-186 °C; R_f = 0.35 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.56-8.54 (m, 1H), 8.40-8.34 (m, 2H), 8.32-8.30 (m, 1H), 7.88 (d, *J* = 7.5 Hz, 1H), 7.78-7.75 (m, 2H), 7.65-7.63 (m, 2H), 7.55-7.47 (m, 5H), 7.43-7.41 (m, 2H) ppm; ^{13}C NMR (126

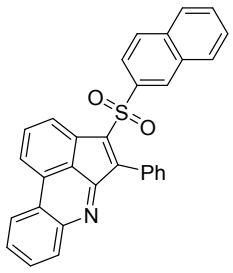
MHz, $CDCl_3$): δ 161.4, 149.3, 148.7, 142.5, 140.7, 135.4, 133.3, 132.8, 132.4, 131.5, 130.2, 130.0, 129.6, 129.4, 129.3, 129.0, 128.2, 125.8, 125.0, 123.9, 123.4, 120.1 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{17}BrNO_2S$ [M+H$^+$]: 498.0158, found 498.0155.



*4-((2-bromophenyl)sulfonyl)-5-phenylcyclopenta[gh]phenanthridine (**3ae**)*. Yellow solid; (23.7 mg, 48%); mp: 193-195 ^oC; R_f = 0.36 (petroleum ether/ethyl acetate 5:1); ^1H NMR (500 MHz, $CDCl_3$): δ = 8.59-8.57 (m, 1H), 8.41-8.36 (m, 2H), 8.33-8.31 (m, 1H), 7.89-7.86 (m, 2H), 7.78-7.76 (m, 2H), 7.55 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.30-7.27 (m, 3H), 7.20-7.13 (m, 2H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.4, 148.7, 147.9, 141.7, 139.8, 136.2, 135.0, 134.4, 133.4, 132.7, 131.8, 131.0, 129.9, 129.8, 129.5, 129.3, 129.2, 128.1, 127.4, 126.2, 125.1, 123.6, 123.4, 121.5, 120.1 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{17}NO_3S$ [M+H$^+$]: 498.0158, found 498.0155.



*4-((4-nitrophenyl)sulfonyl)-5-phenylcyclopenta[gh]phenanthridine (**3af**)*. Yellow solid; (16.7 mg, 36%); mp: 212-213 ^oC; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^1H NMR (500 MHz, $CDCl_3$): δ = 8.59-8.57 (m, 1H), 8.45-8.39 (m, 2H), 8.34-8.32 (m, 1H), 8.09 (d, *J* = 8.5 Hz, 2H), 7.94-7.91 (m, 1H), 7.82-7.77 (m, 4H), 7.65-7.63 (m, 2H), 7.56-7.48 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.0, 150.6, 150.4, 148.8, 147.1, 141.5, 135.1, 133.5, 133.0, 131.5, 129.8, 129.7, 129.612, 129.584, 129.2, 128.4, 125.9, 125.0, 124.211, 124.161, 123.4, 120.1 ppm. ESI-HRMS: m/z Calcd for $C_{27}H_{17}N_2O_4S$ [M+H$^+$]: 465.0904, found 465.0906.

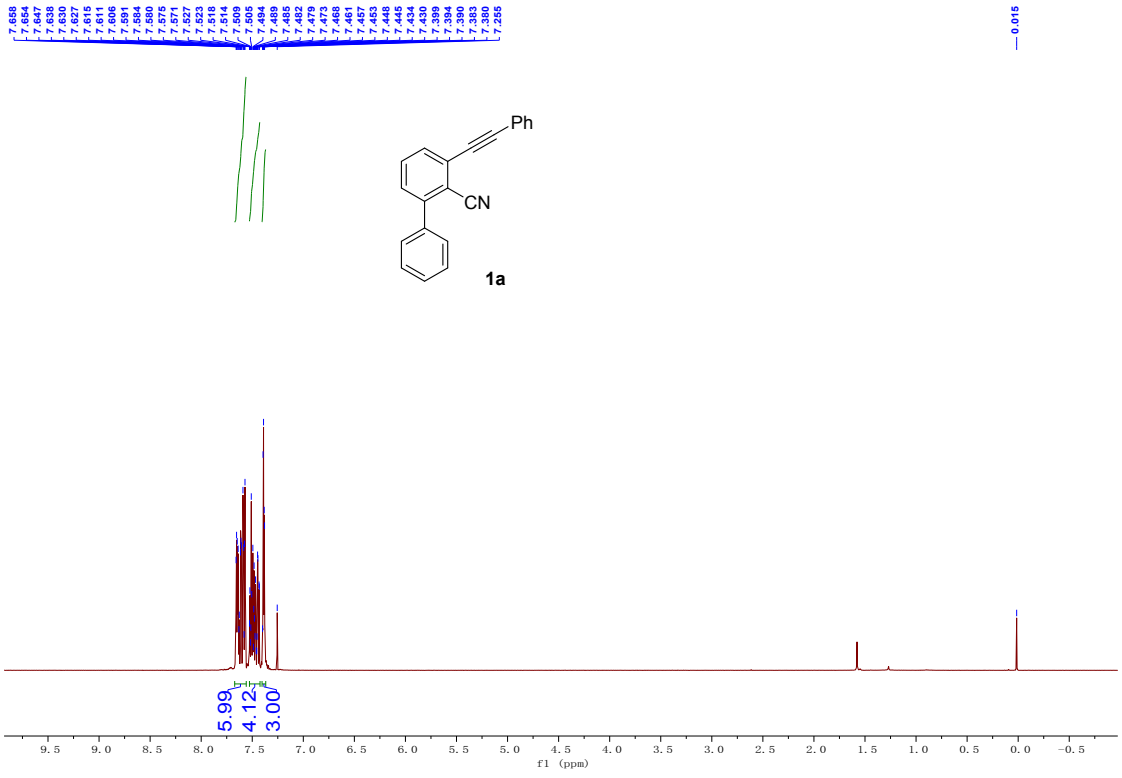


*4-(naphthalen-2-ylsulfonyl)-5-phenylcyclopenta[gh]phenanthridine (**3ag**).* Yellow solid; (22.0 mg, 47%); mp: 178-179 °C; R_f = 0.32 (petroleum ether/ethyl acetate 5:1); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.56-8.54 (m, 1H), 8.45-8.38 (m, 2H), 8.30-8.28 (m, 1H), 8.16 (s, 1H), 7.91-7.81 (m, 1H), 7.79-7.73 (m, 5H), 7.65-7.62 (m, 3H), 7.60-7.51 (m, 2H), 7.45-7.40 (m, 3H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.7, 149.1, 148.7, 143.0, 138.3, 135.7, 135.4, 133.4, 132.8, 132.2, 131.5, 130.2, 130.0, 129.9, 129.8, 129.5, 129.3, 129.2, 128.1, 128.0, 127.7, 125.9, 125.0, 123.7, 123.3, 122.7, 120.2 ppm. ESI-HRMS: m/z Calcd for $C_{31}H_{20}NO_2S_2$ [M+H$^+$]: 470.1209, found 470.1212.

Reference

[1] Yuan, Z.; Wang, H.; Mu, X.; Chen, P.; Guo, Y.; Liu, G. *J. Am. Chem. Soc.* 2015, **137**, 2468.

[2] (a) X. Li, X. Fang, S. Zhuang, P. Liu and P. Sun, *Org. Lett.* 2017, **19**, 3580; (b) J. B. Hynes, A. Pathak, C. H. Panes, C. C. Okeke, *J. Heterocyclic. Chem.* 1988, **25**, 1173; (c) X.-T. Zhu, Q.-L. Lu, X. Wang, T.-S. Zhang, W.-J. Hao, S.-J. Tu and B. Jiang, *J. Org. Chem.* 2018, **83**, 9890.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7.527
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7.514
7.509
7.505
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7.489
7.485
7.482
7.479
7.473
7.468
7.461
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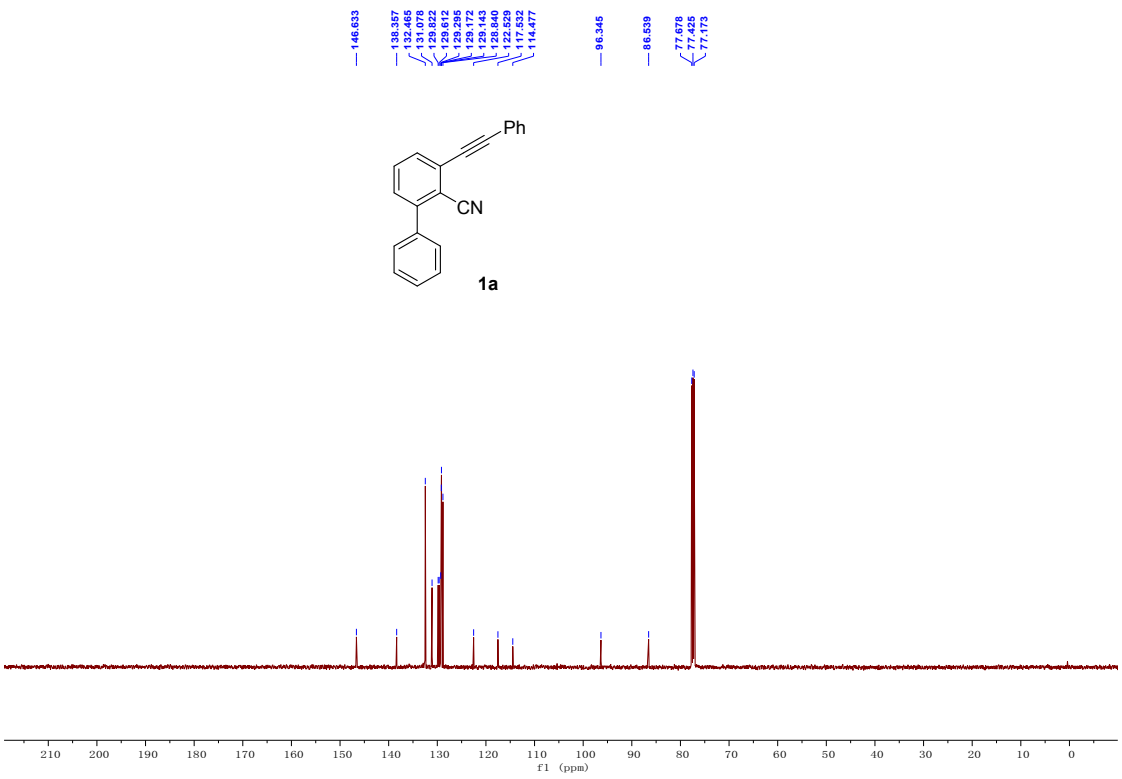
f1 (ppm)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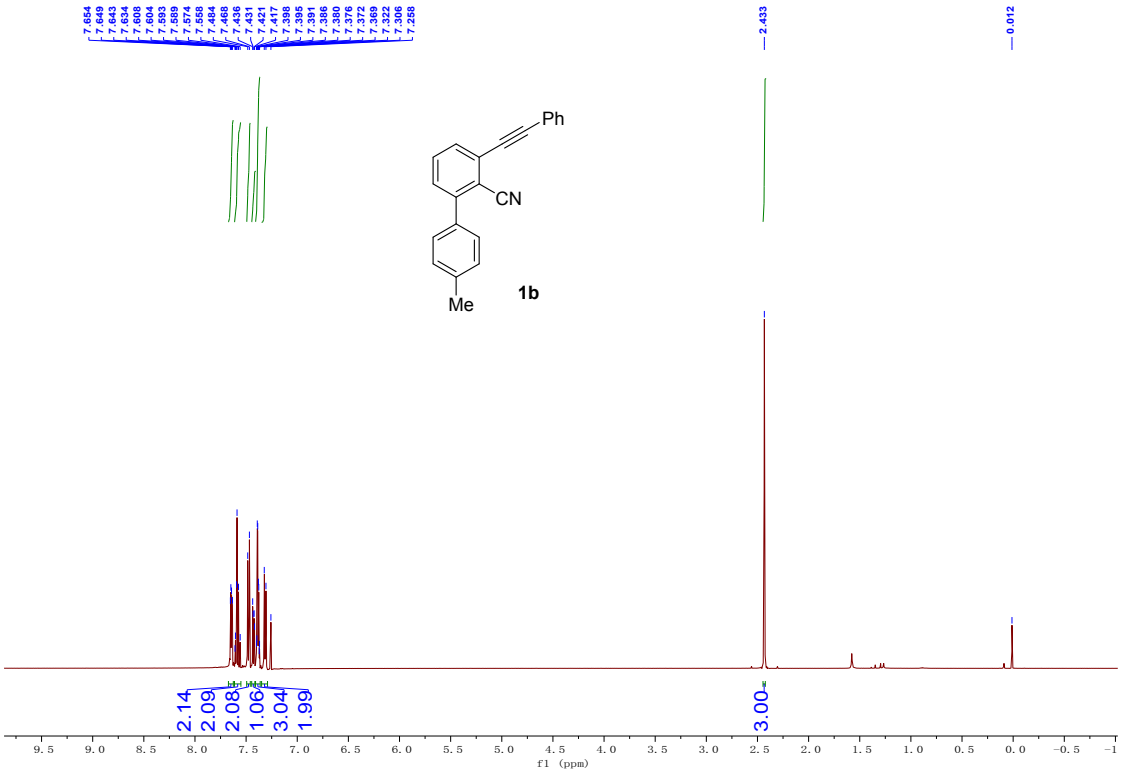
Ph
CN
1a
f1 (ppm)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7.436
7.431
7.421
7.417
7.398
7.395
7.391
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7.380
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7.372
7.369
7.322
7.306
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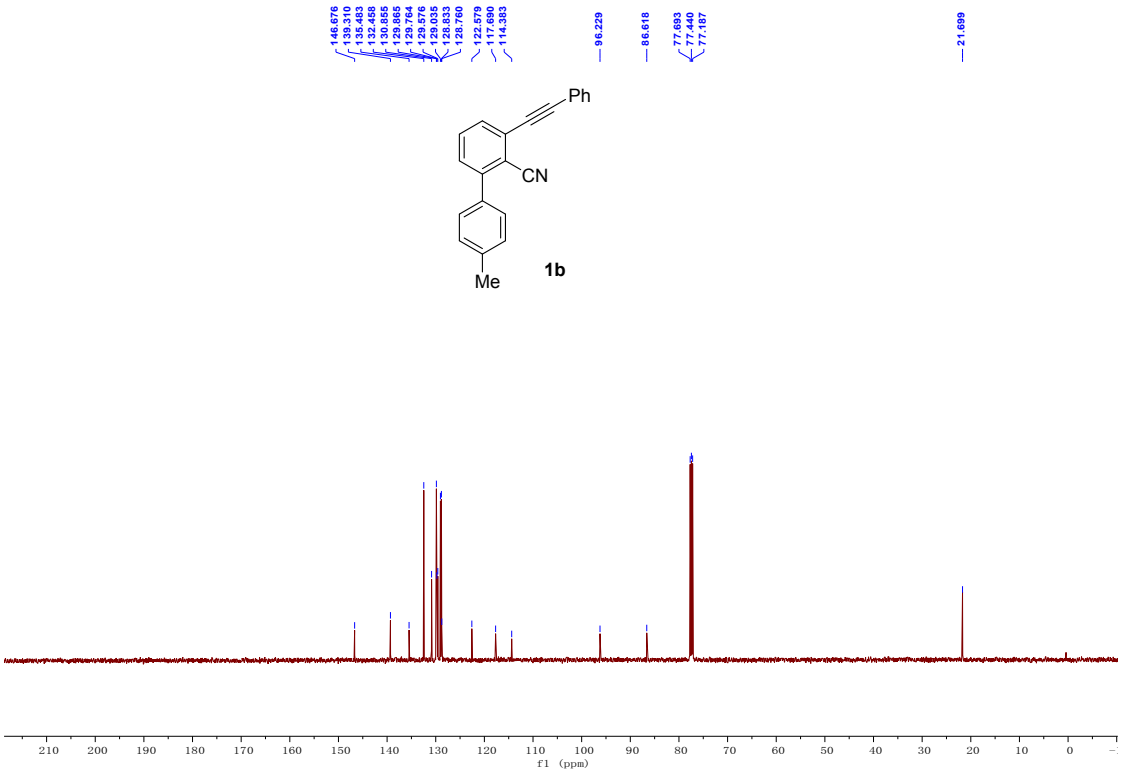
f1 (ppm)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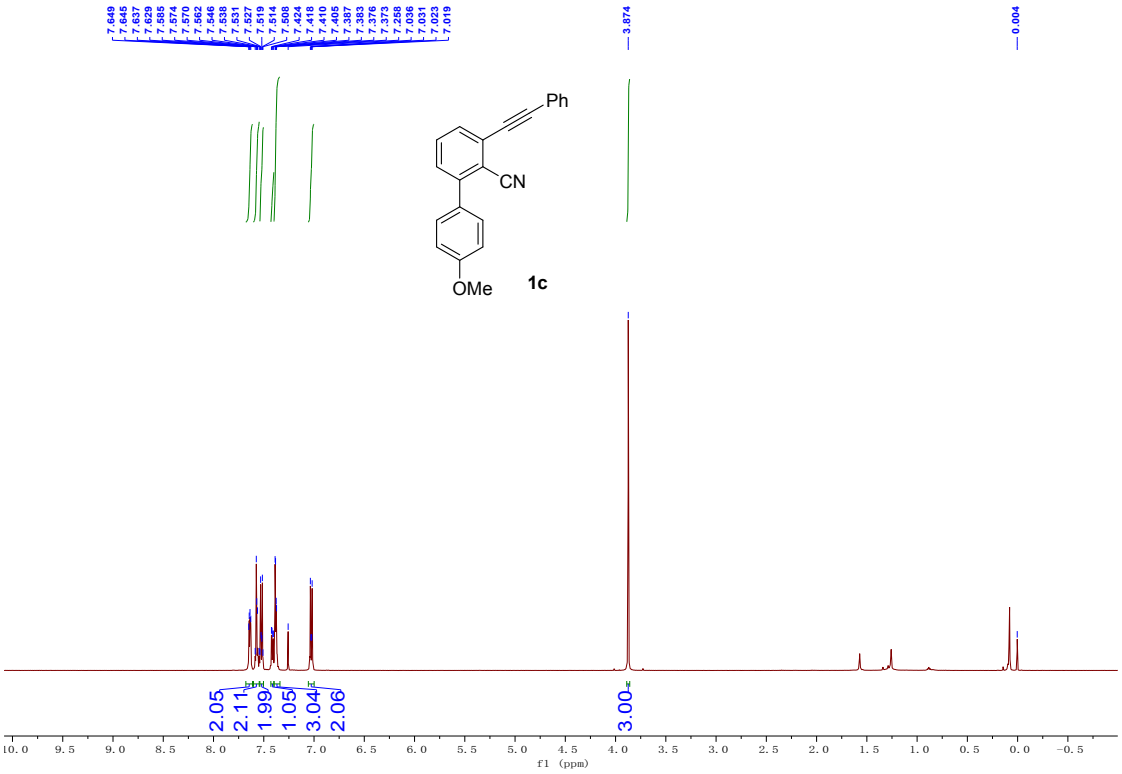
1b
f1 (ppm)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7.418
7.410
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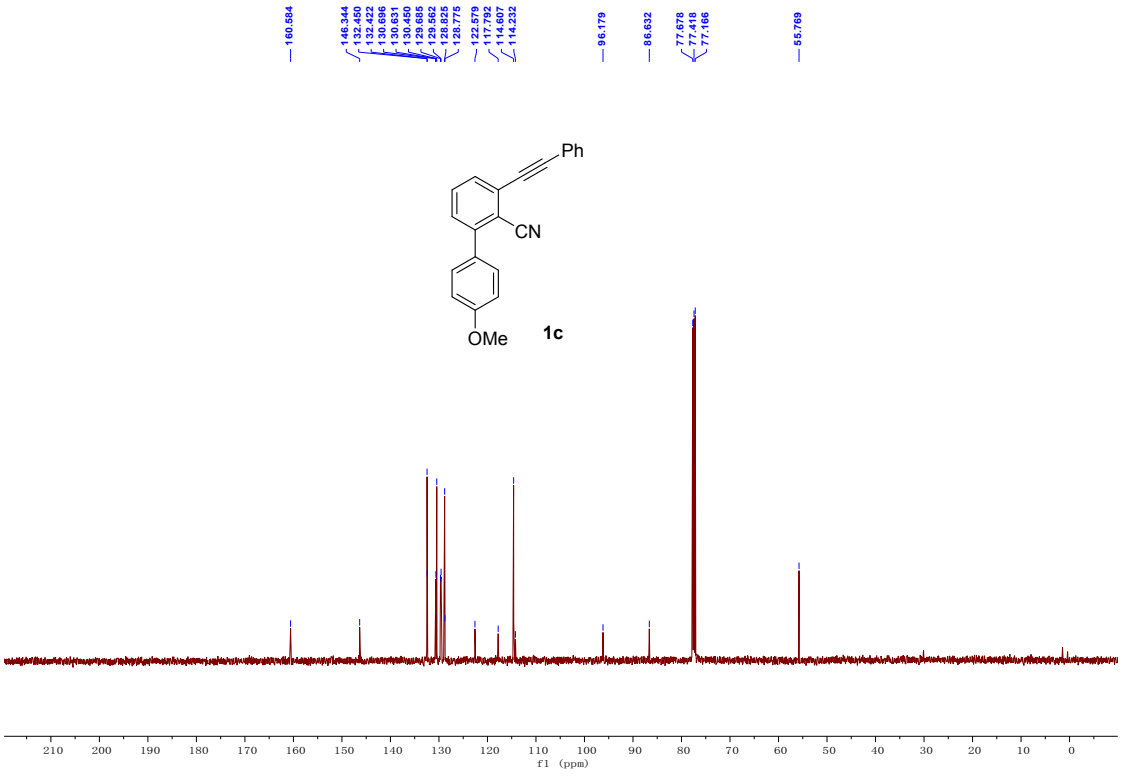
3.00
f1 (ppm)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86.632
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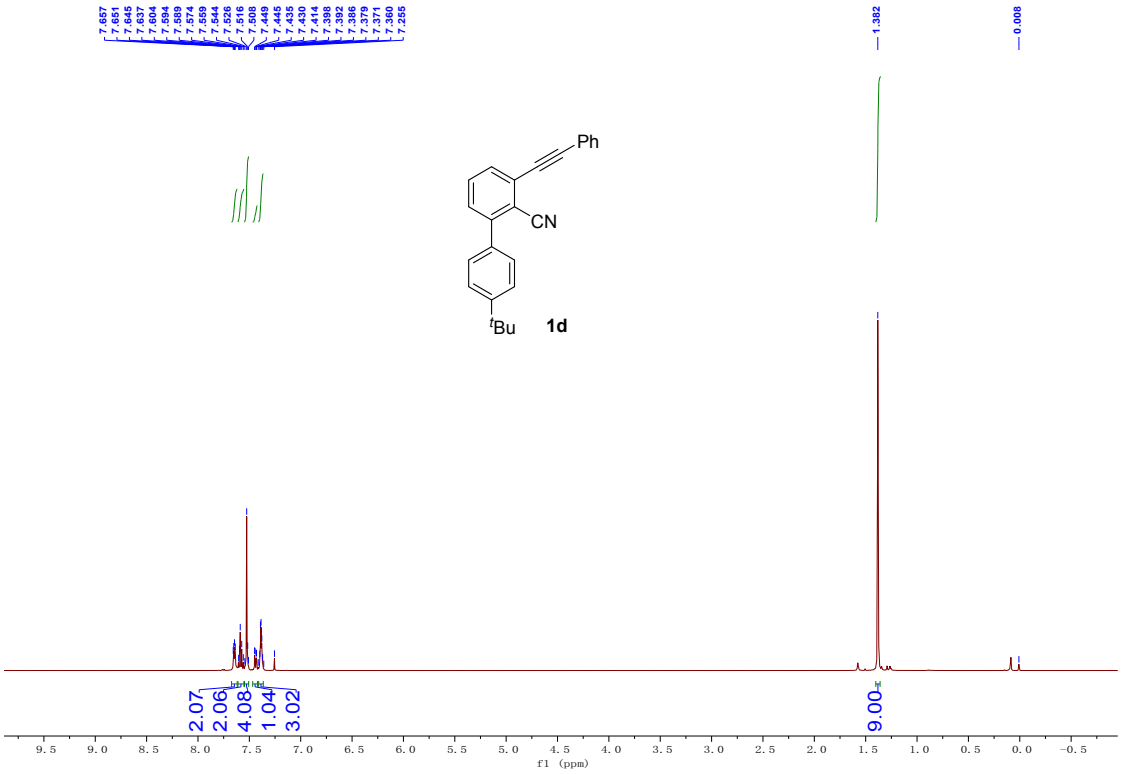
Ph
CN
OMe
1c
f1 (ppm)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7.445
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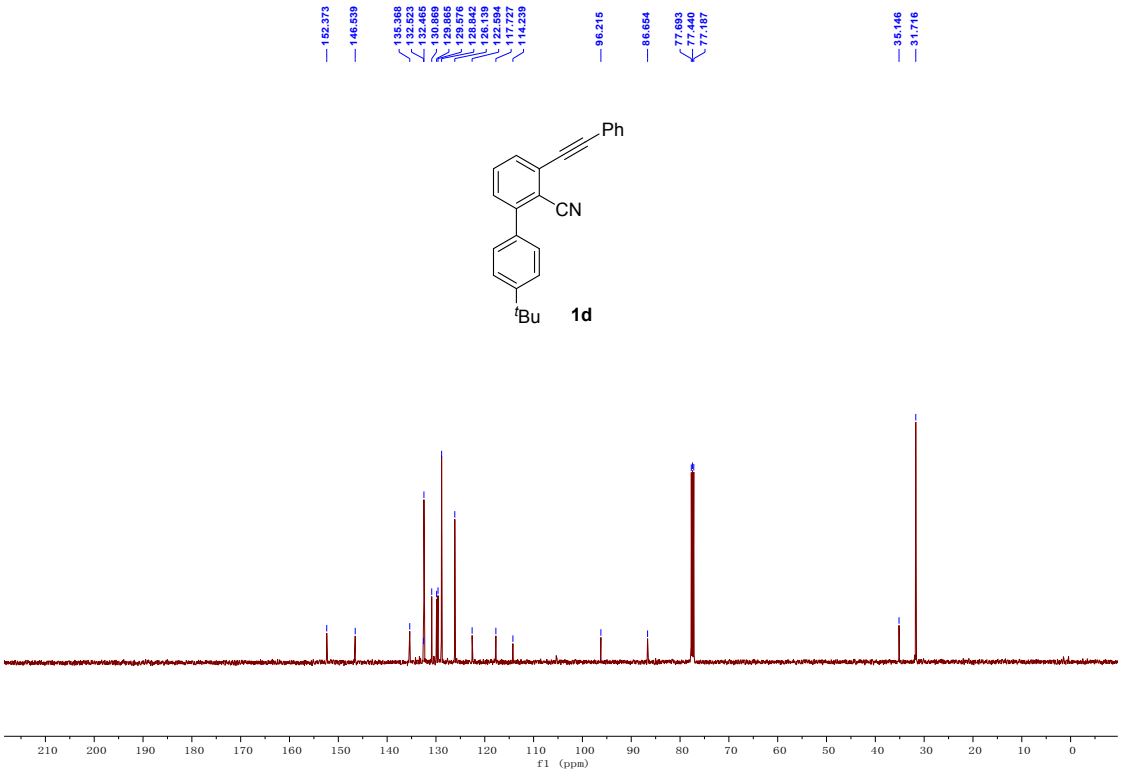
f1 (ppm)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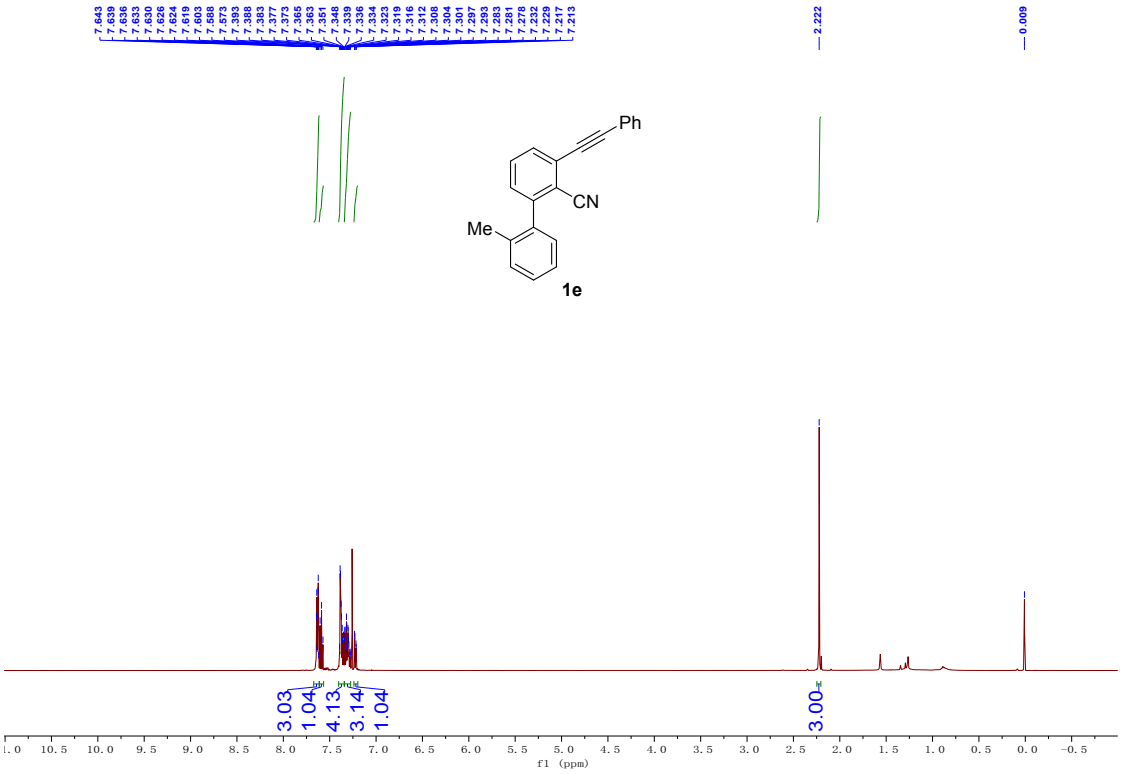
f1 (ppm)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7.383
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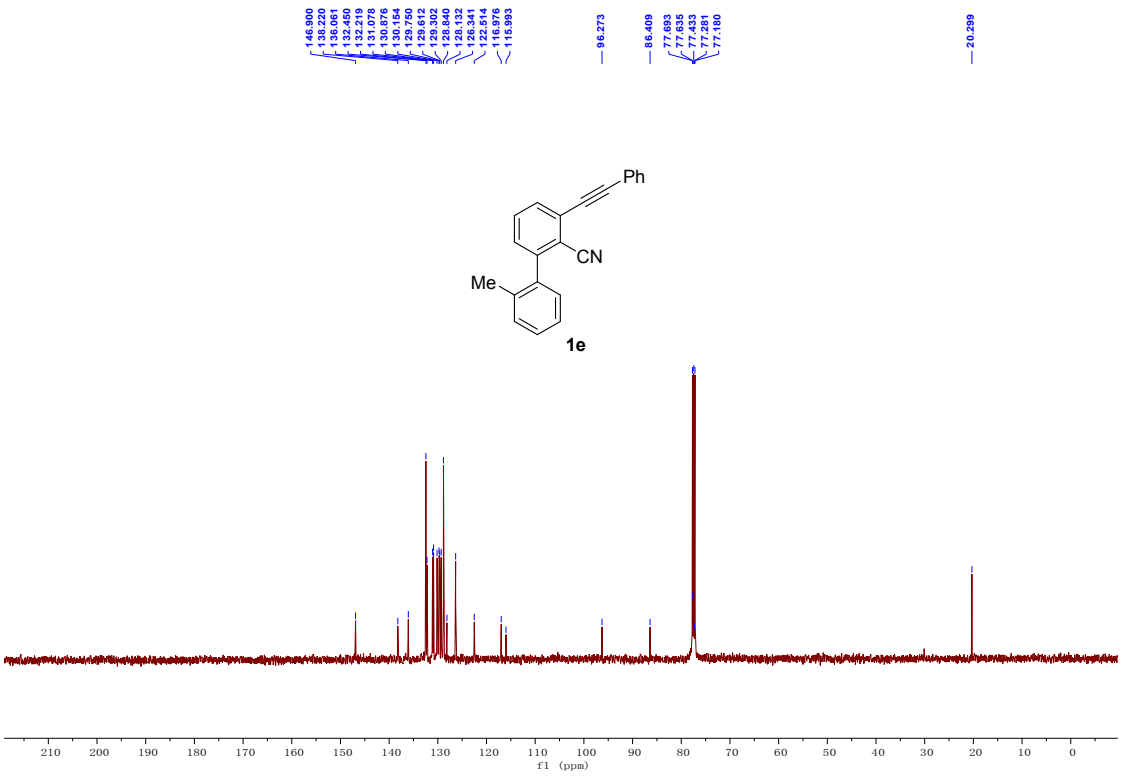
3.00
f1 (ppm)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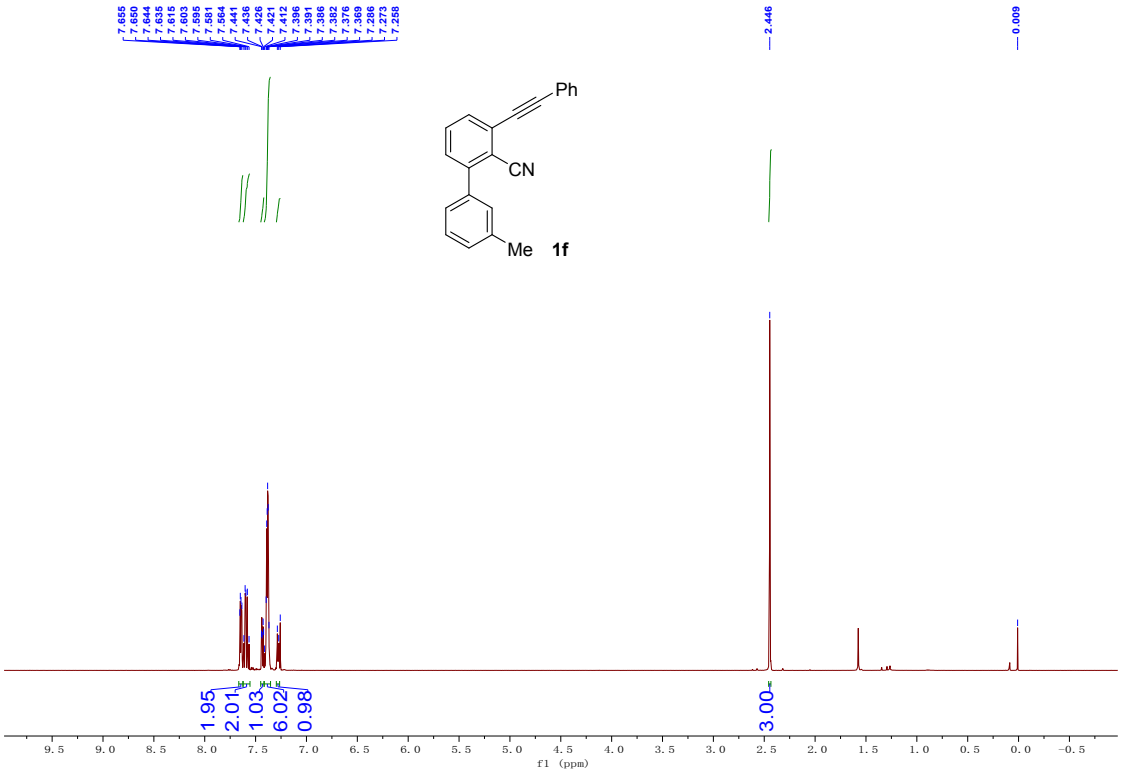
1e
f1 (ppm)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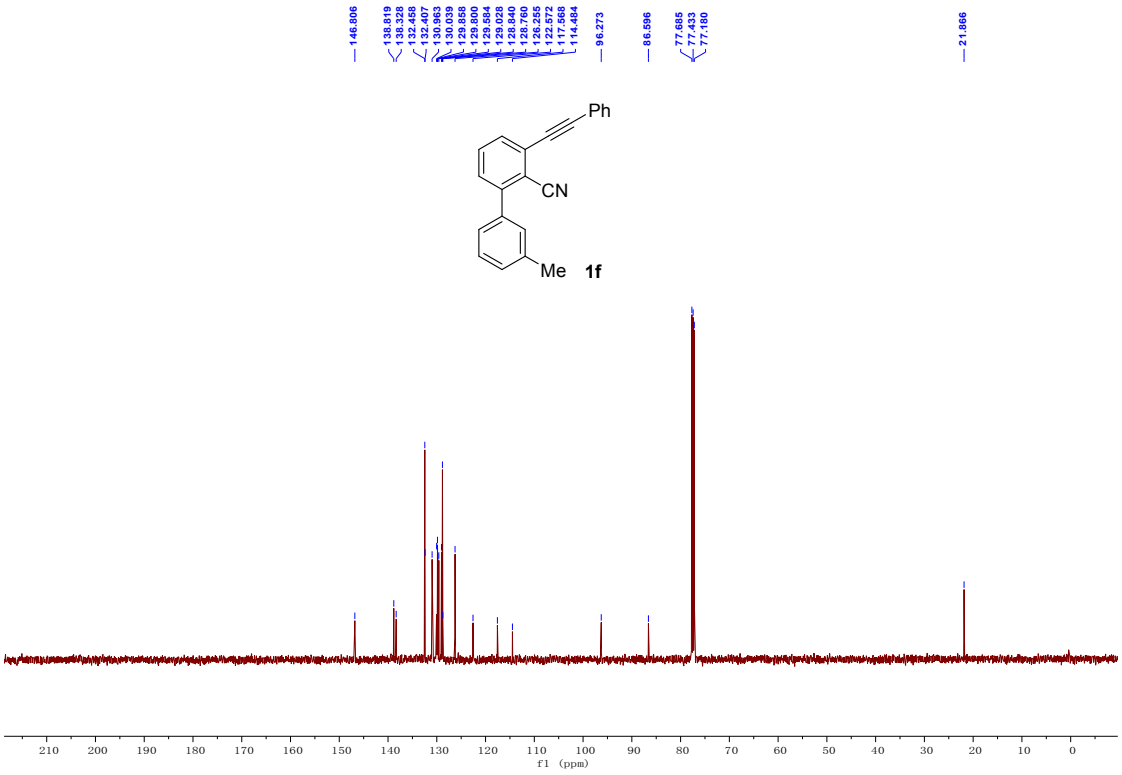
f1 (ppm)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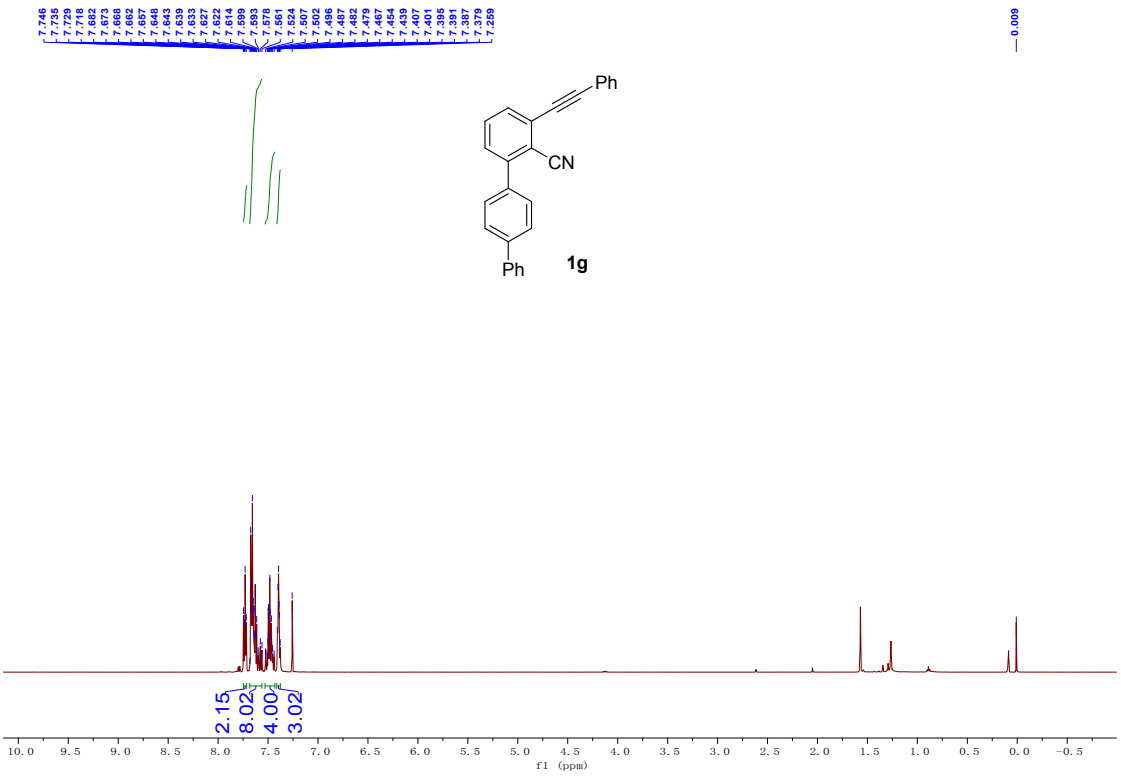
f1 (ppm)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Ph
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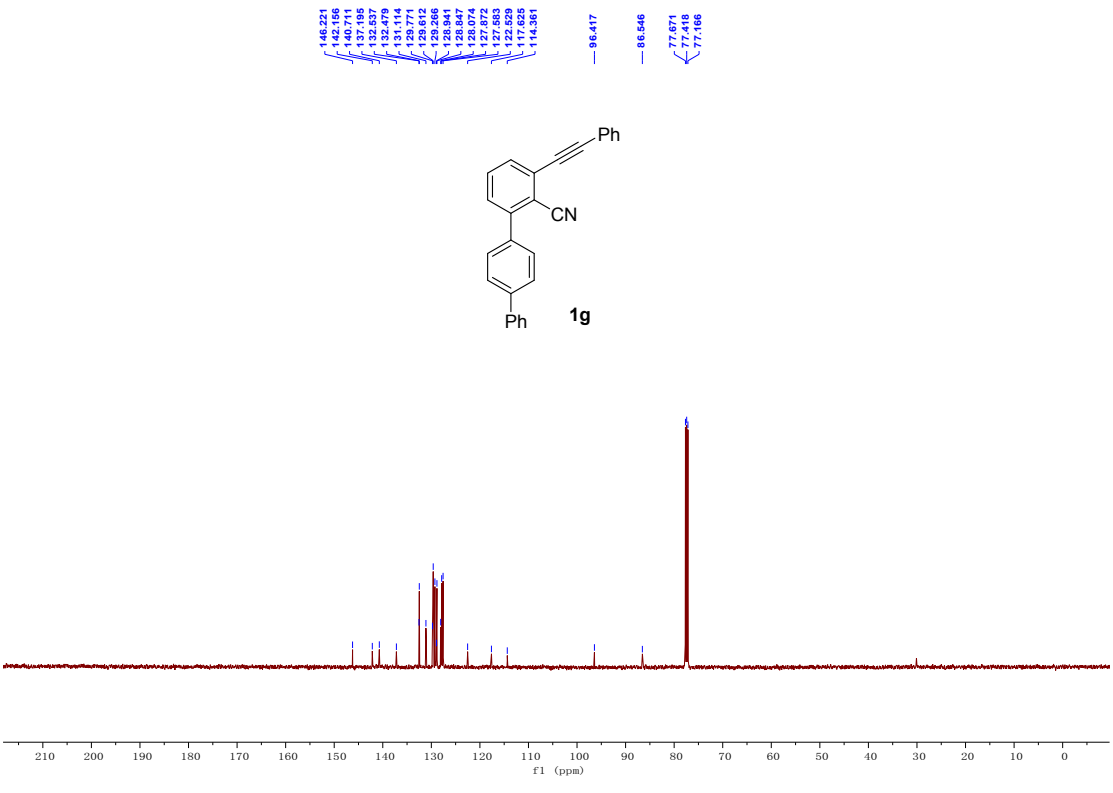
f1 (ppm)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Ph
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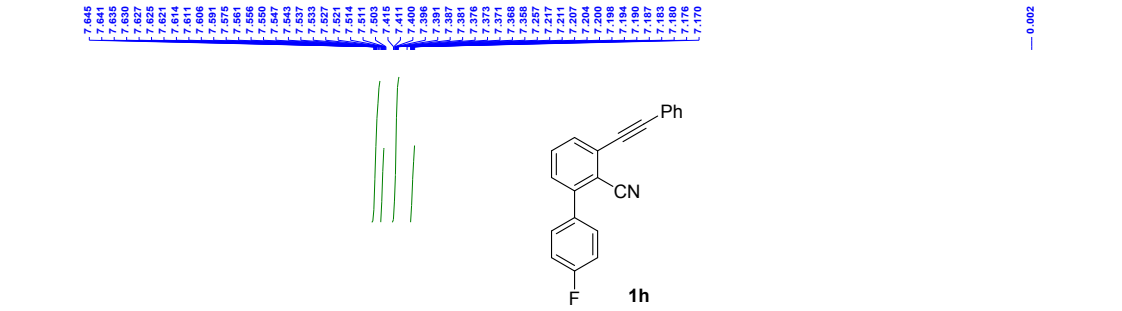
f1 (ppm)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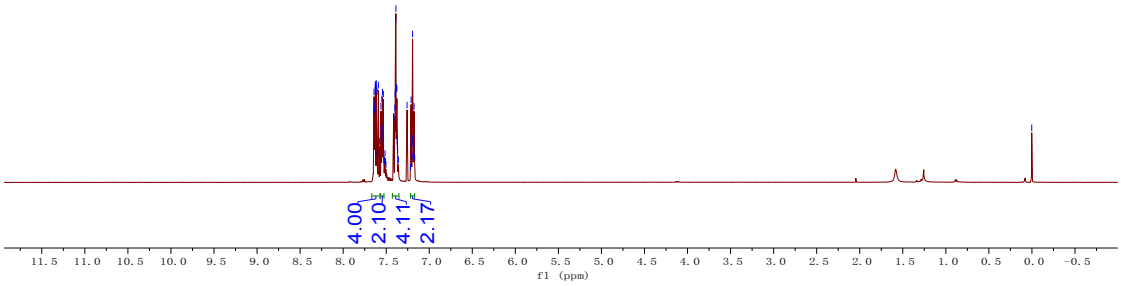
F
1h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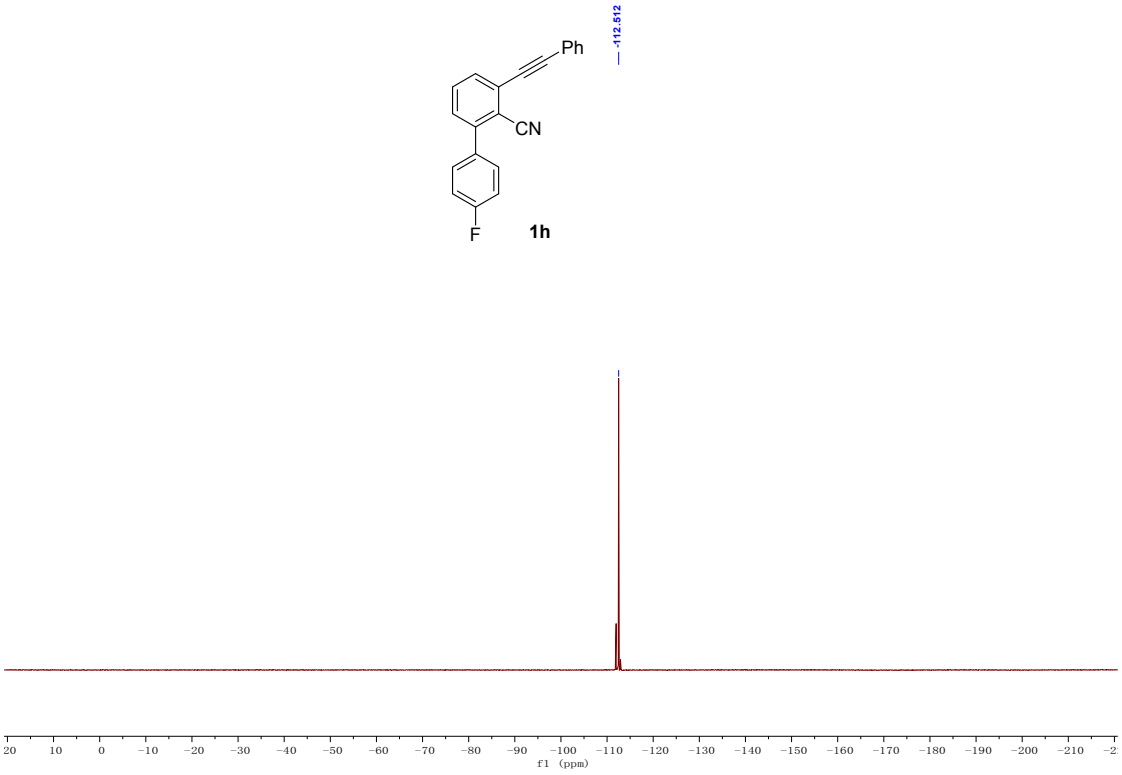
f1 (ppm)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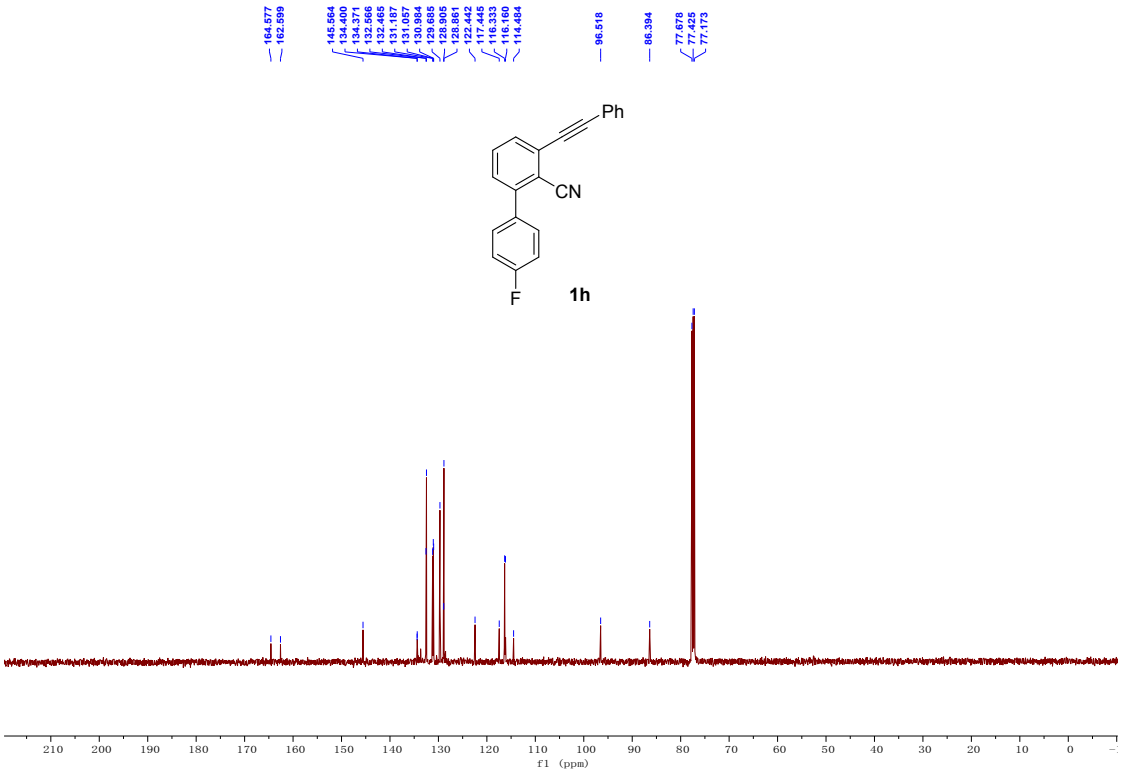
f1 (ppm)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131.697
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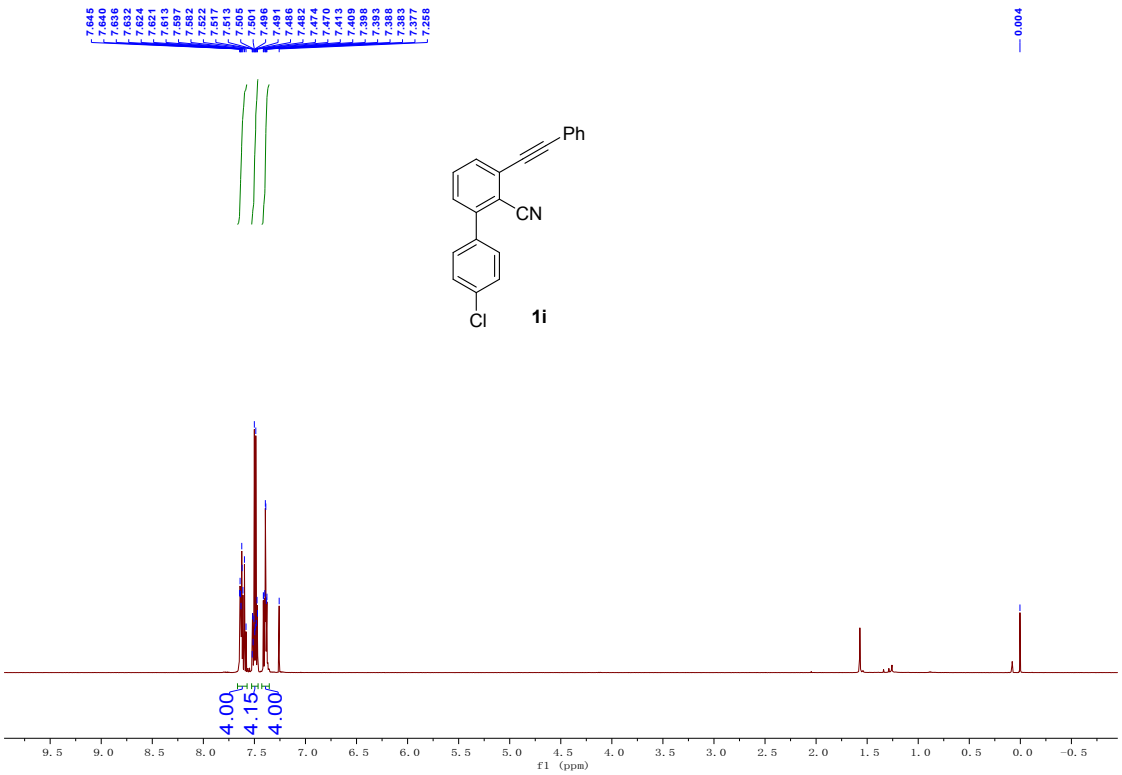
130.984
129.685
128.905
128.861
122.442
117.445
116.333
116.150
114.484
96.518
86.394
77.678
77.425
77.173
Ph
CN
F
1h
f1 (ppm)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7.613
7.597
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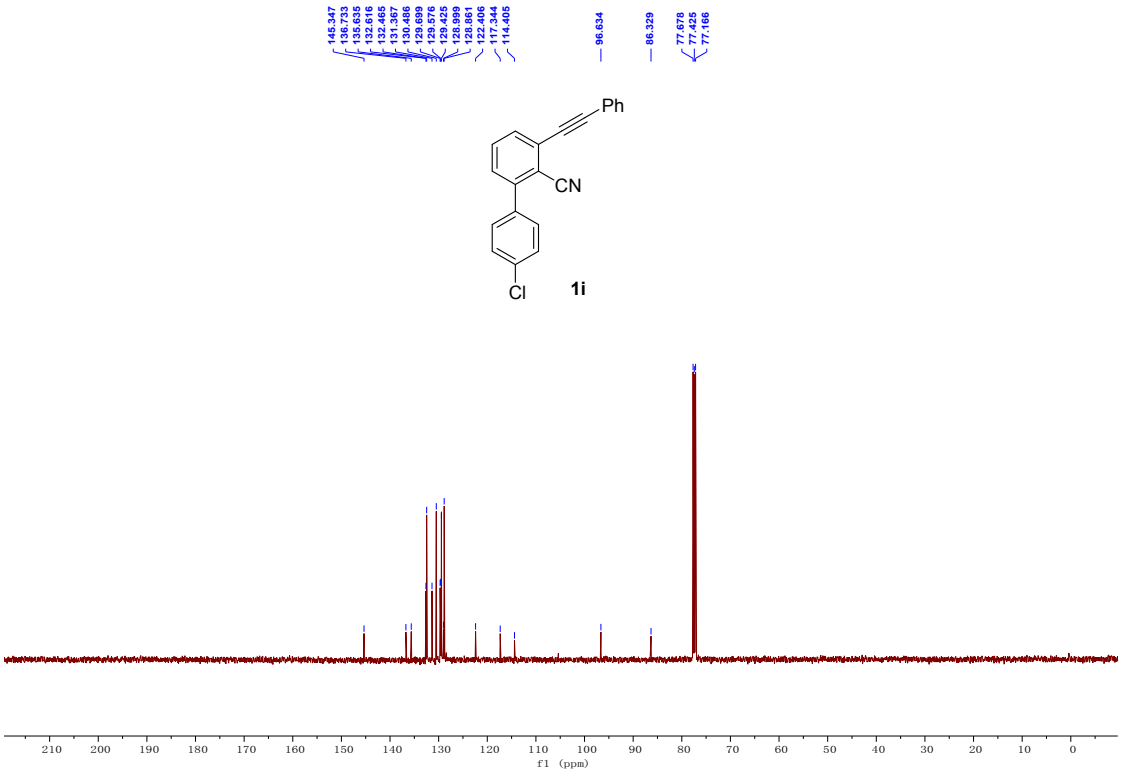
7.522
7.517
7.513
7.505
7.501
7.496
7.491
7.486
7.482
7.474
7.470
7.413
7.409
7.398
7.393
7.388
7.383
7.377
7.258
0.004
Ph
CN
Cl
1i
4.00
4.15
4.00
f1 (ppm)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136.733
135.635
132.616
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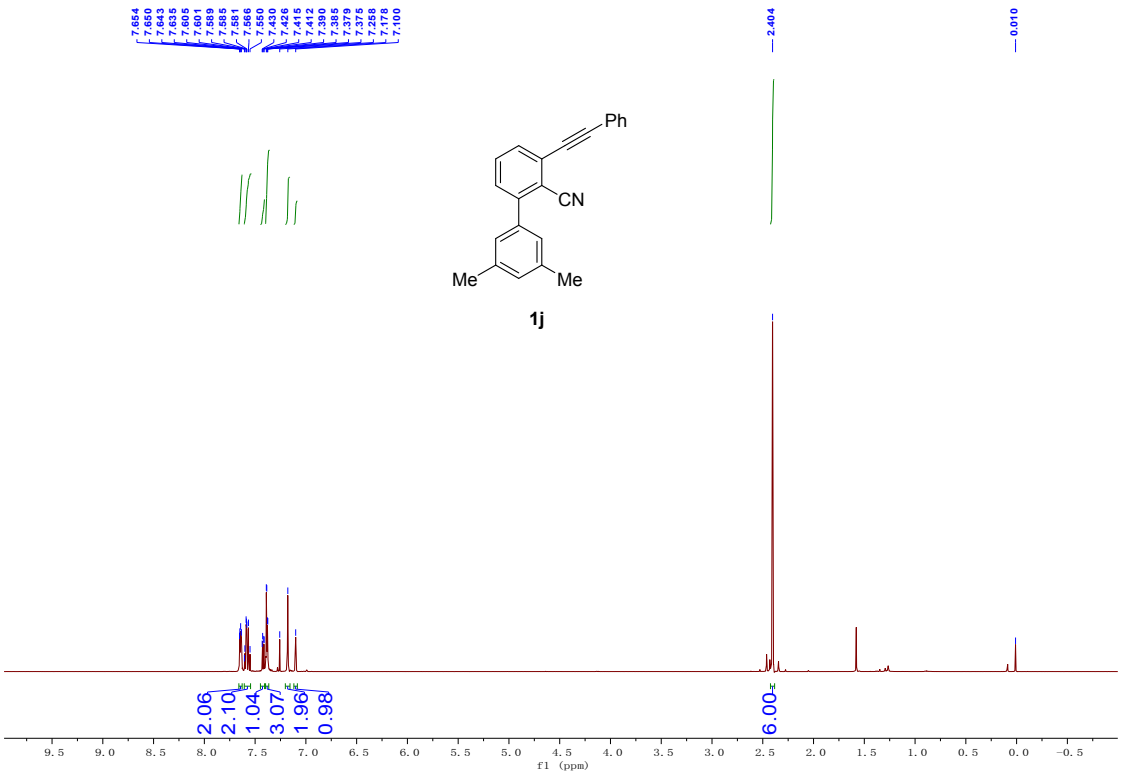
130.486
129.699
129.576
129.425
128.999
128.861
122.406
117.344
114.405
96.634
86.329
77.678
77.425
77.166
Ph
CN
Cl
1i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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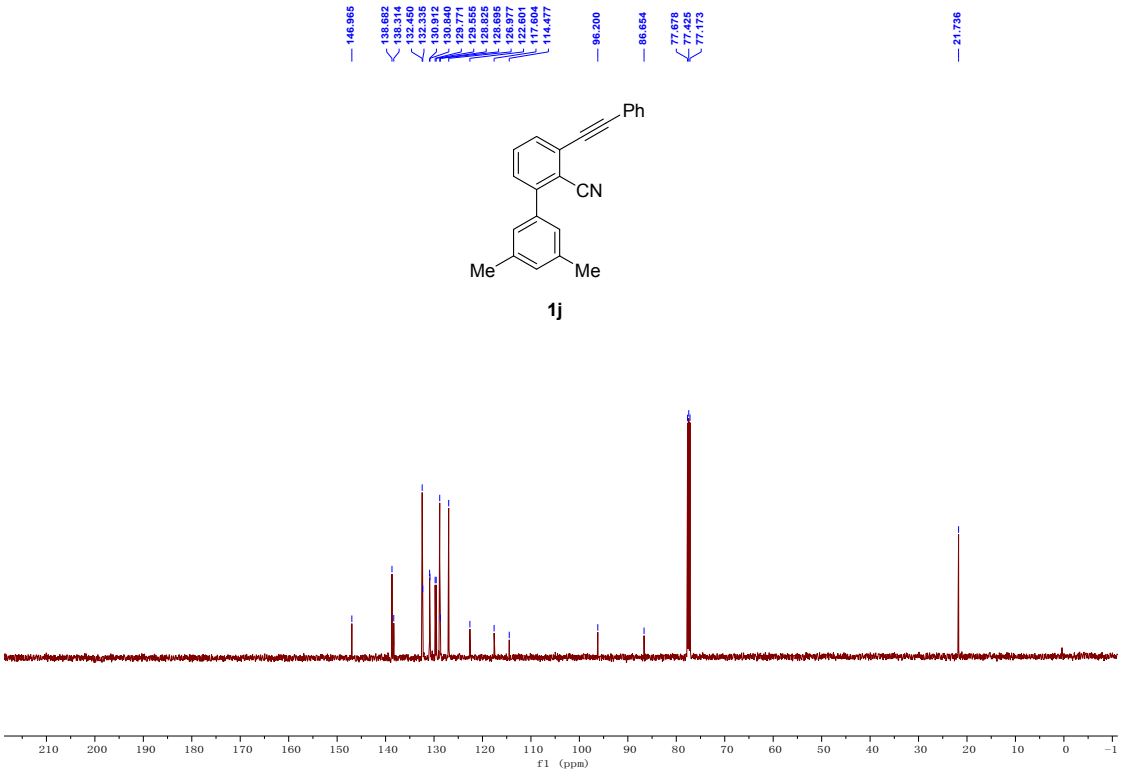
7.585
7.581
7.566
7.550
7.430
7.426
7.415
7.413
7.390
7.385
7.379
7.375
7.258
7.178
7.100
2.404
0.010
Ph
CN
Me
Me
1j
2.06
2.10
1.04
3.07
1.96
0.98
6.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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129.771
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128.825
128.695
126.977
122.601
117.604
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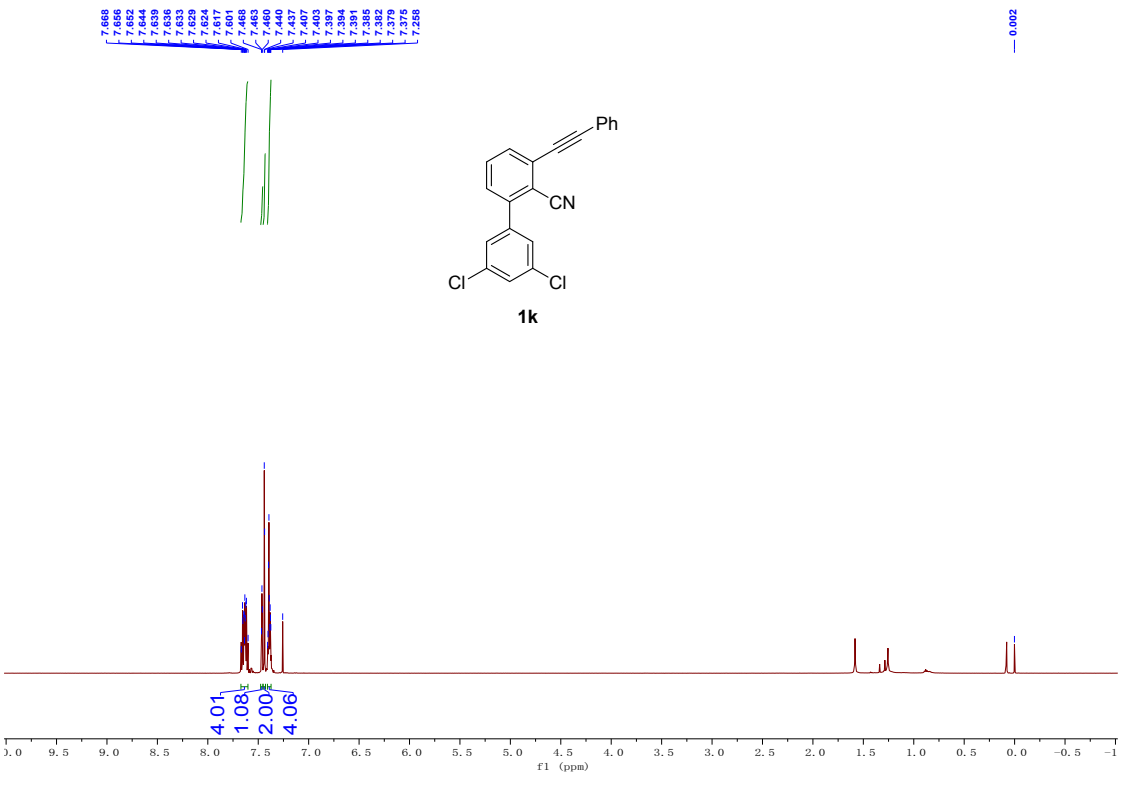
86.654
77.678
77.425
77.173
21.736
Ph
CN
Me
Me
f1 (ppm)

1j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7.629
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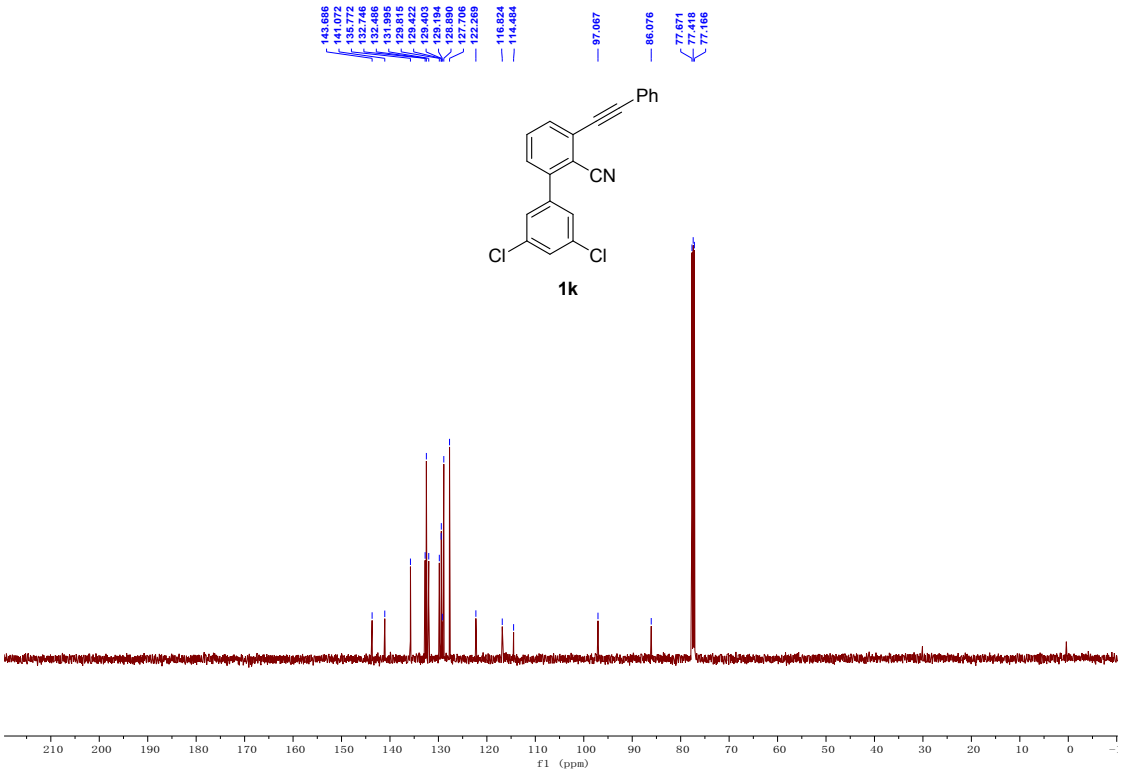
7.617
7.601
7.488
7.463
7.460
7.440
7.437
7.407
7.403
7.397
7.394
7.391
7.385
7.382
7.379
7.375
7.258
0.002
Ph
CN
Cl
Cl
4.01
1.08
2.00
4.06
f1 (ppm)

1k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129.422
129.403
129.194
128.890
127.706
122.269
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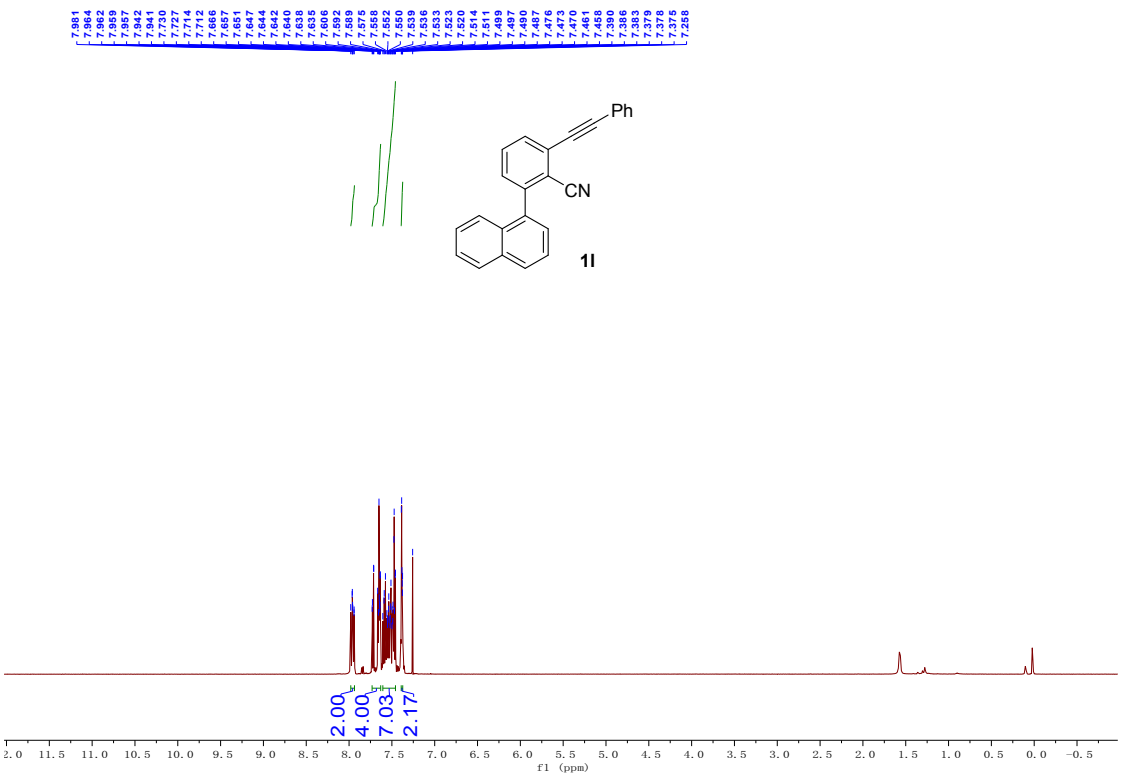
97.067
86.076
77.671
77.418
77.166
Ph
CN
Cl
Cl
1k
f1 (ppm)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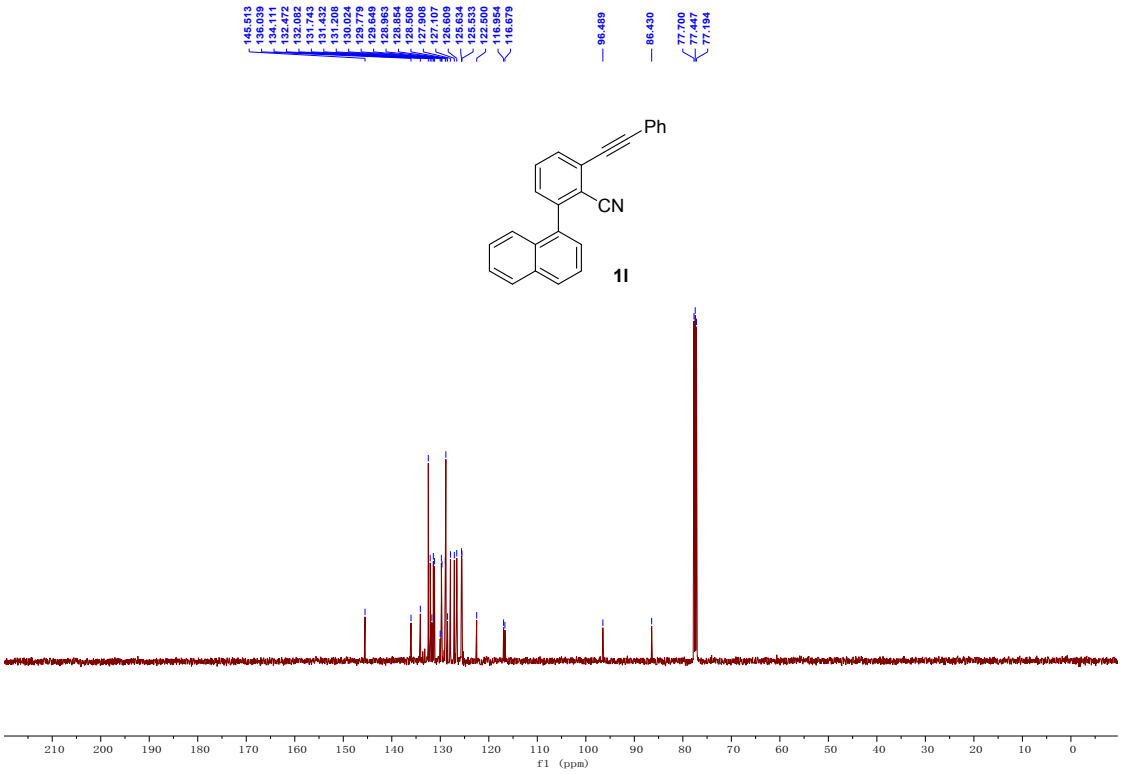
7.959
7.957
7.942
7.941
7.730
7.727
7.714
7.712
7.666
7.657
7.651
7.647
7.644
7.642
7.640
7.638
7.635
7.606
7.592
7.589
7.575
7.558
7.552
7.550
7.539
7.536
7.523
7.520
7.514
7.511
7.499
7.497
7.490
7.487
7.476
7.473
7.470
7.461
7.458
7.390
7.386
7.383
7.379
7.378
7.375
7.258
Ph
CN
1l
2.00
4.00
7.03
2.17
f1 (ppm)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136.039
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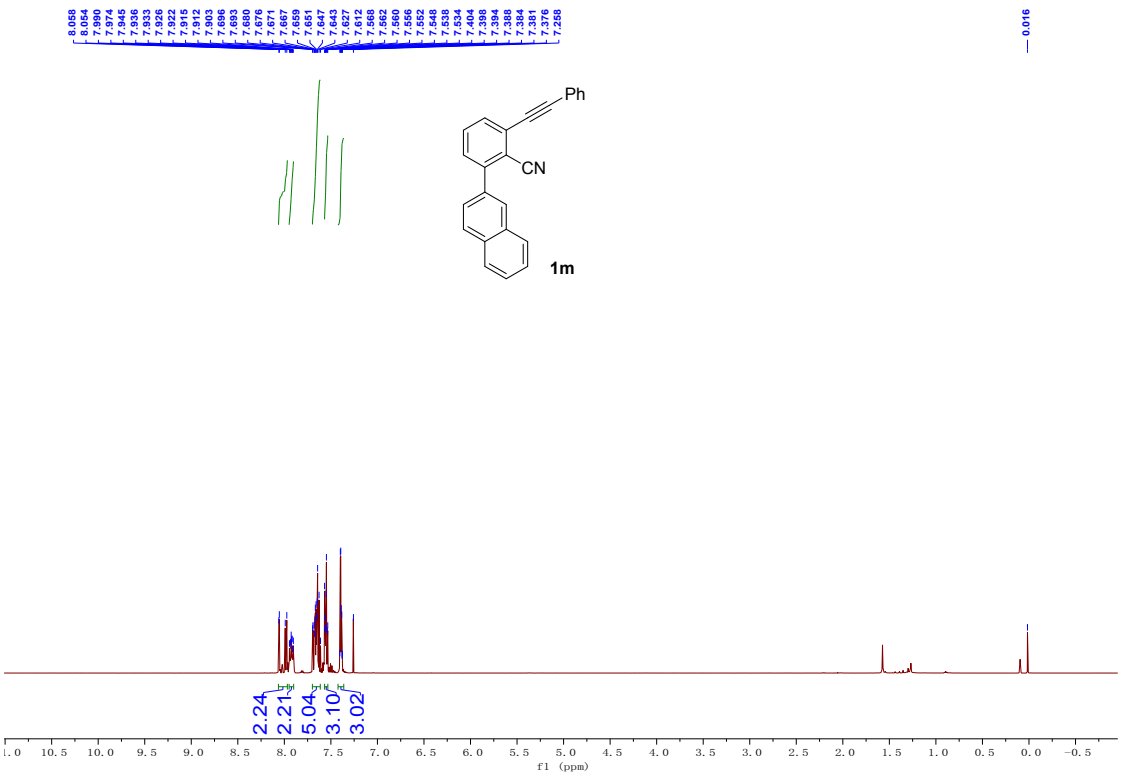
132.472
132.082
131.743
131.432
131.208
130.024
129.779
129.649
128.963
128.854
128.508
128.008
127.107
126.609
125.634
125.533
122.500
116.954
116.679
96.489
86.430
77.700
77.447
77.194
Ph
CN
1l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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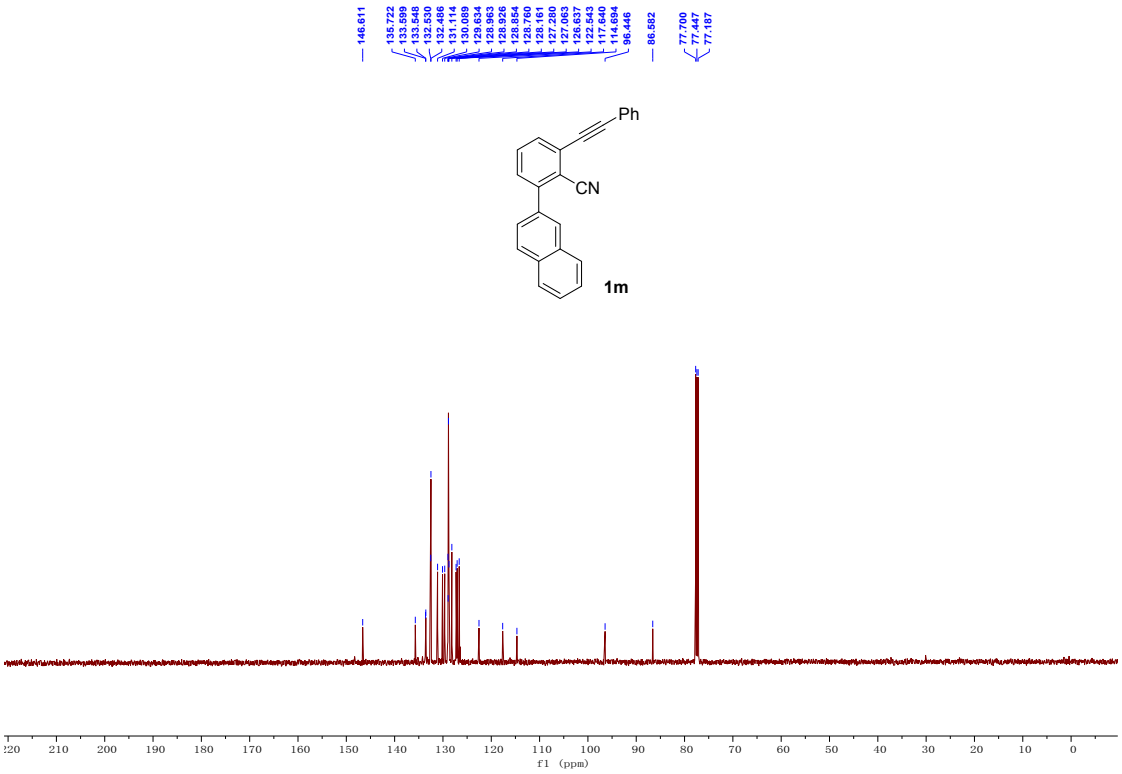
8.054
7.990
7.974
7.945
7.936
7.933
7.926
7.922
7.915
7.912
7.903
7.696
7.693
7.680
7.676
7.671
7.667
7.659
7.651
7.647
7.643
7.627
7.612
7.568
7.562
7.560
7.555
7.552
7.548
7.538
7.534
7.404
7.398
7.394
7.388
7.384
7.381
7.376
7.258
0.016
Ph
CN
1m
2.24
2.21
5.04
3.10
3.02
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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133.599
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130.089
129.614
128.963
128.926
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128.760
128.161
127.280
127.063
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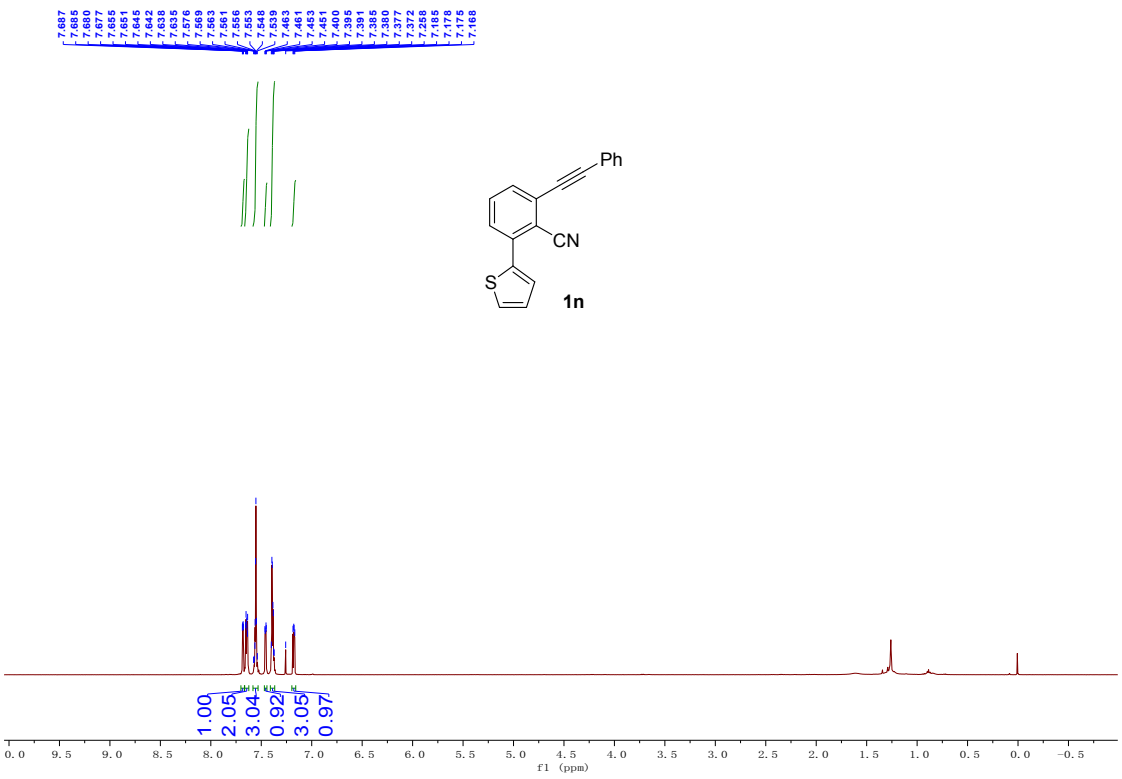
114.694
96.446
86.582
77.700
77.447
77.187
Ph
CN
1m
f1 (ppm)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7.685
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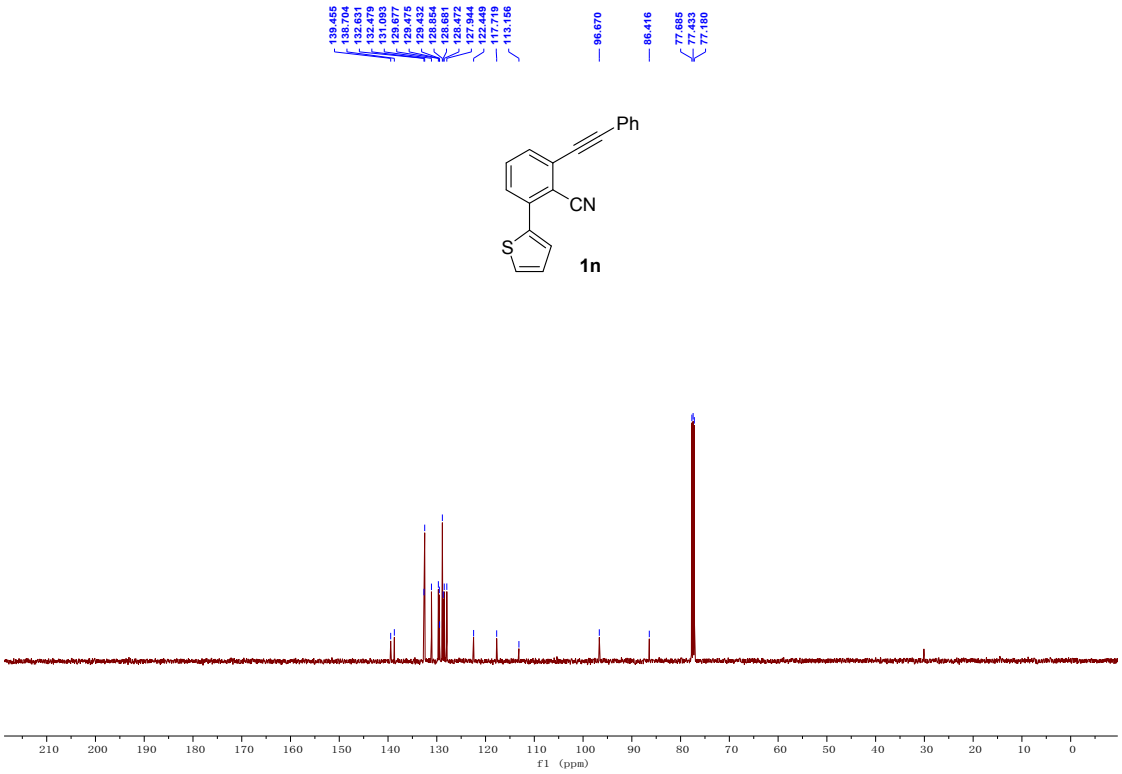
7.677
7.655
7.651
7.645
7.642
7.638
7.635
7.576
7.569
7.563
7.561
7.556
7.553
7.548
7.539
7.463
7.461
7.453
7.451
7.400
7.395
7.391
7.385
7.380
7.377
7.372
7.298
7.185
7.178
7.175
7.168
Ph
CN
S
1n
1.00
2.05
3.04
0.92
3.05
0.97
f1 (ppm)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132.631
132.479
131.093
129.677
129.475
129.432
128.854
128.681
128.472
127.944
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113.156
96.670
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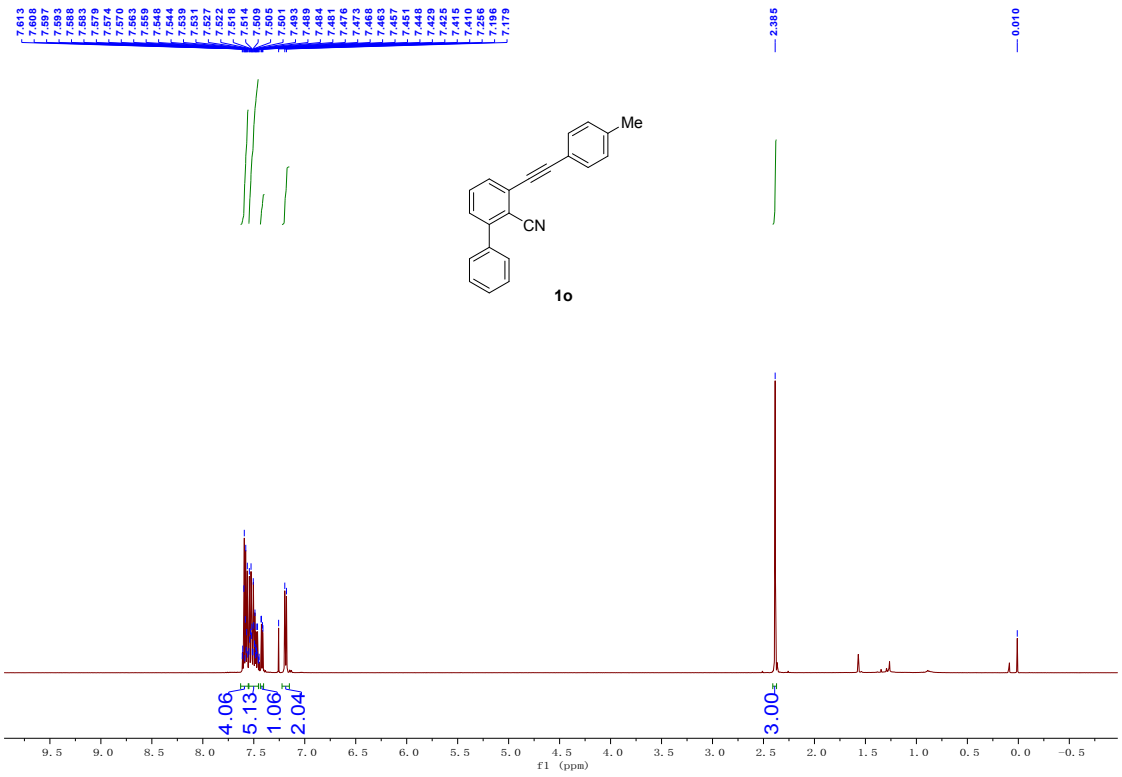
77.685
77.433
77.180
Ph
CN
S
1n
f1 (ppm)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7.597
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7.588
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7.579
7.574
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7.563
7.559
7.548
7.544
7.539
7.531
7.527
7.522
7.518
7.514
7.509
7.505
7.501
7.493
7.489
7.484
7.481
7.476
7.473
7.468
7.463
7.457
7.451
7.448
7.429
7.425
7.415
7.410
7.256
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Me
CN
1o
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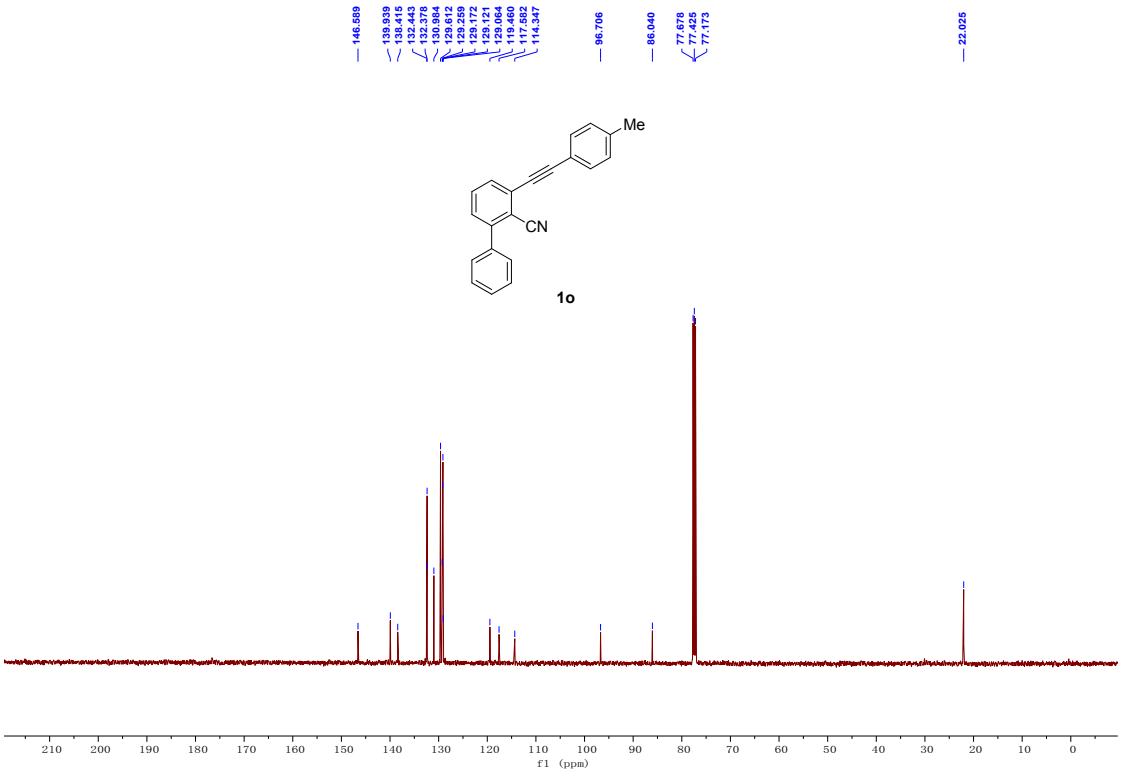
5.13
1.06
2.04
3.00
f1 (ppm)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129.612
129.259
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129.064
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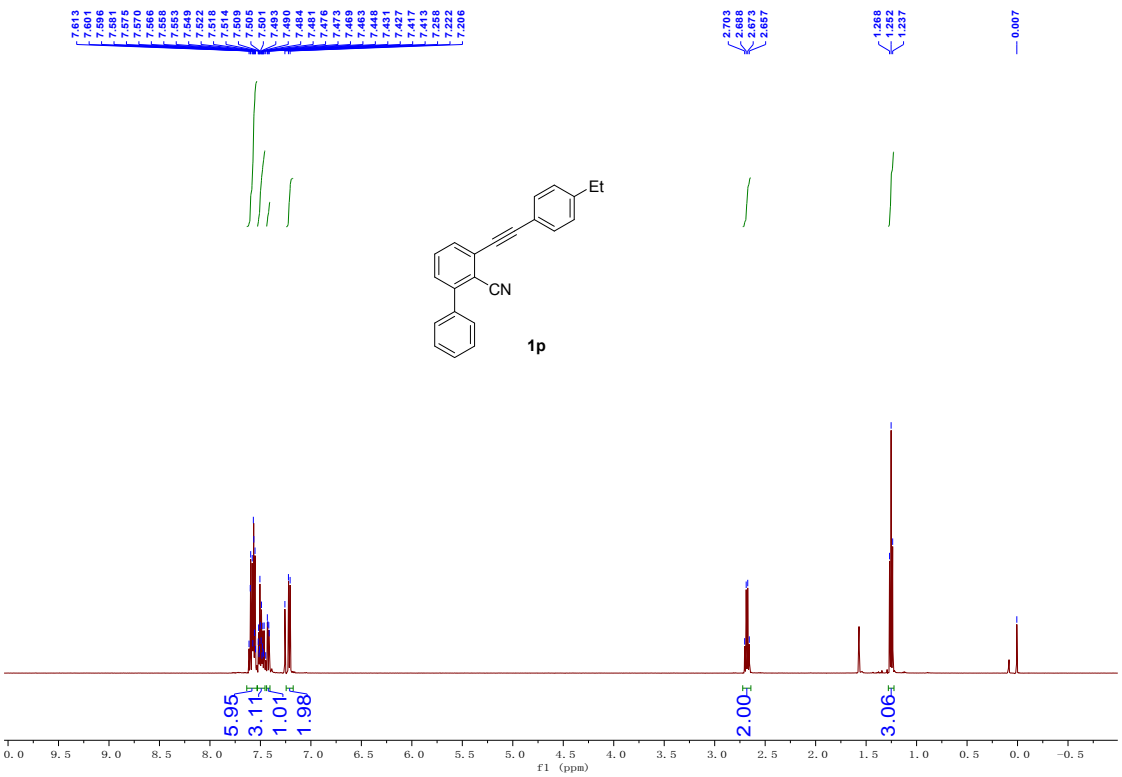
77.678
77.425
77.173
22.025
Me
CN
1o
f1 (ppm)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7.514
7.509
7.505
7.501
7.493
7.490
7.484
7.481
7.476
7.473
7.469
7.463
7.448
7.431
7.427
7.417
7.413
7.358
7.222
7.206
2.703
2.688
2.673
2.657
1.268
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CN
1p
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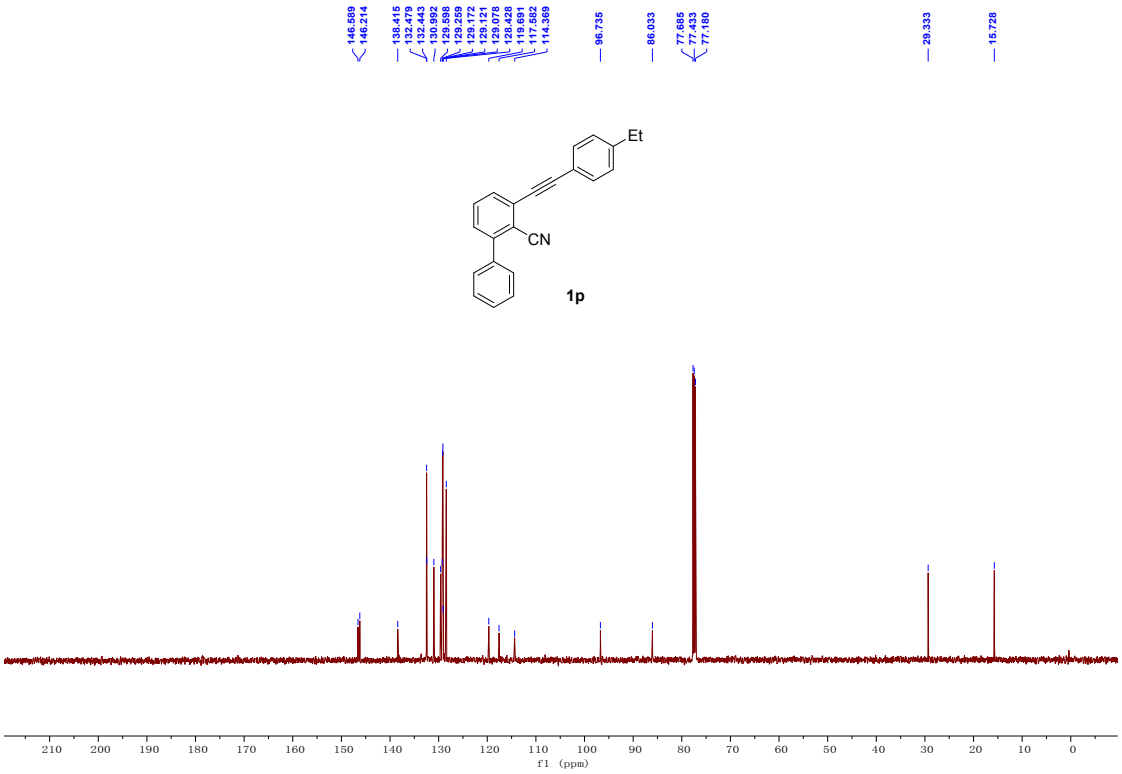
3.06
f1 (ppm)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128.428
119.691
117.592
114.369
96.735
86.033
77.685
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15.728
Et
CN
1p
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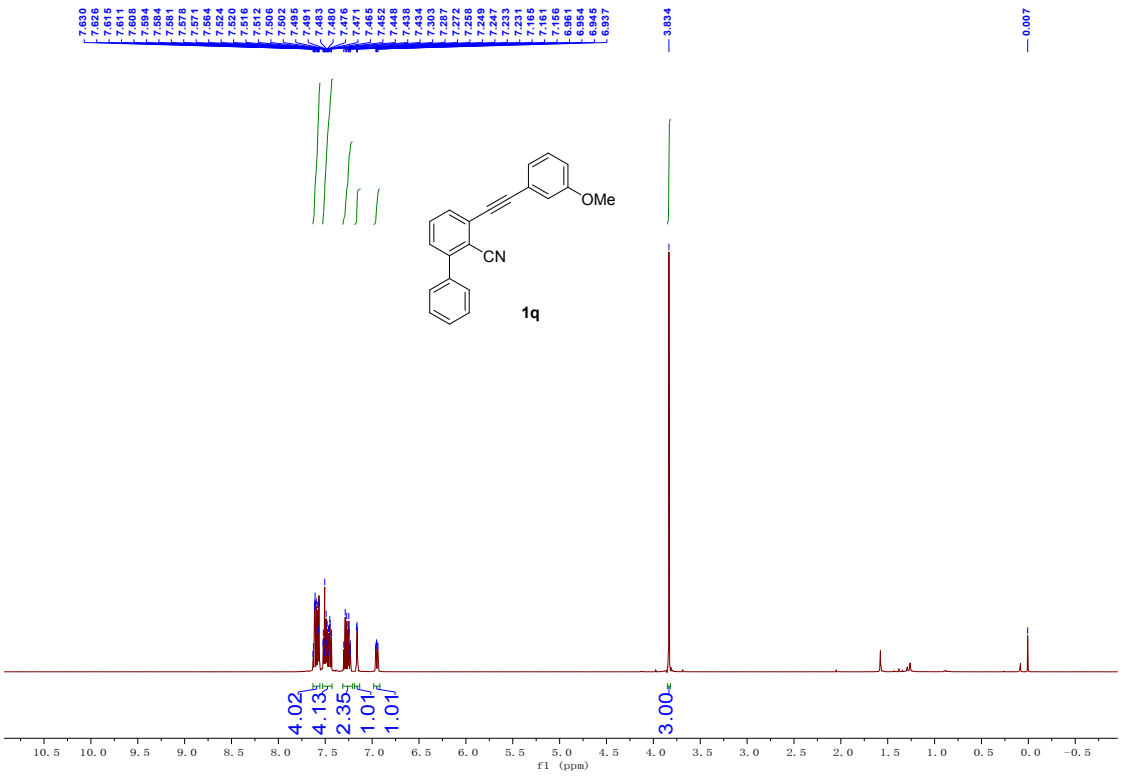
f1 (ppm)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7.594
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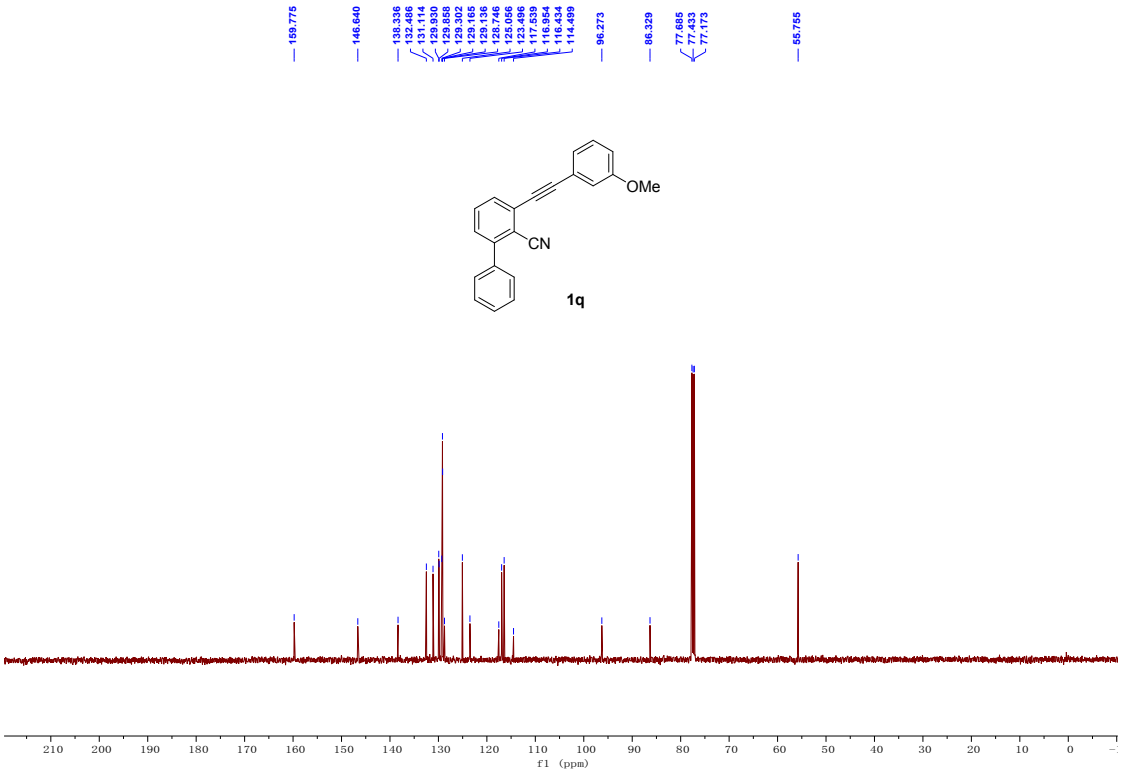
7.581
7.578
7.571
7.564
7.524
7.520
7.516
7.512
7.506
7.503
7.495
7.491
7.483
7.480
7.476
7.471
7.465
7.462
7.446
7.438
7.434
7.303
7.287
7.272
7.258
7.249
7.247
7.233
7.231
7.165
7.161
7.156
6.961
6.954
6.945
6.937
3.834
0.007
OMe
CN
1q
4.02
4.13
2.35
1.01
1.01
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)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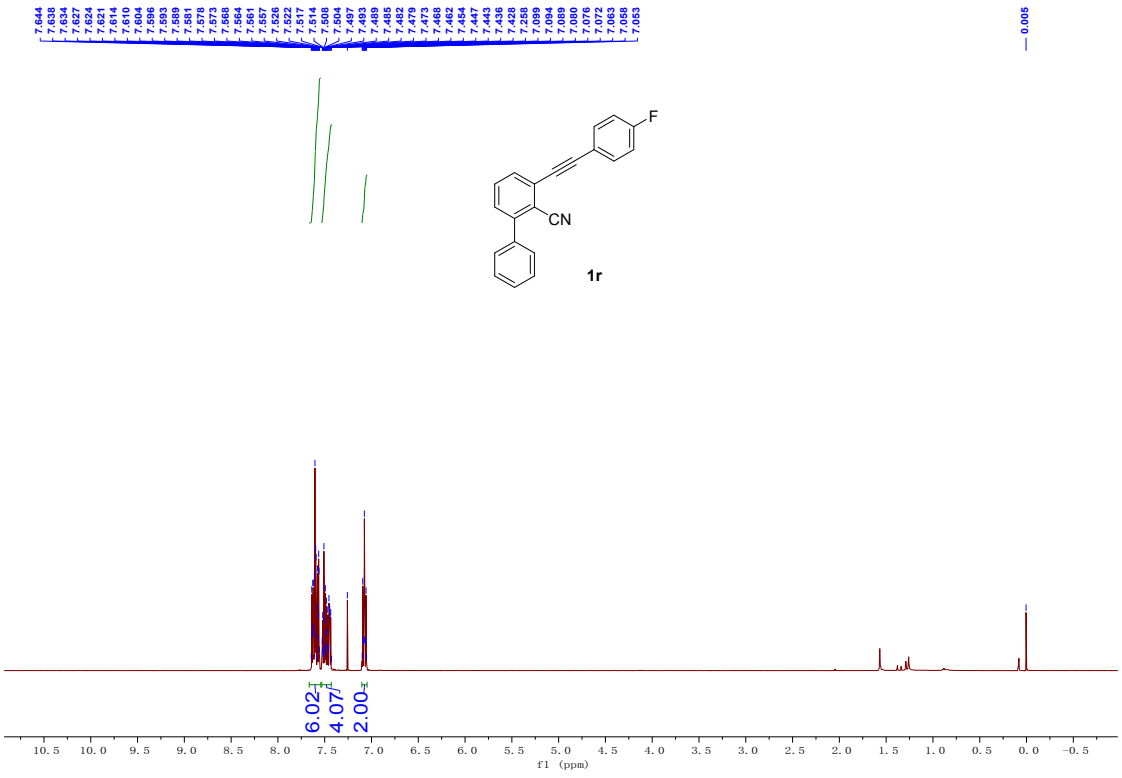
128.746
125.056
123.496
117.559
116.954
116.434
114.499
96.273
86.329
77.685
77.433
77.173
55.755
OMe
CN
1q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.578
7.573
7.568
7.564
7.561
7.557
7.526
7.522
7.517
7.514
7.508
7.504
7.497
7.493
7.489
7.485
7.482
7.479
7.473
7.468
7.462
7.454
7.447
7.443
7.436
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7.258
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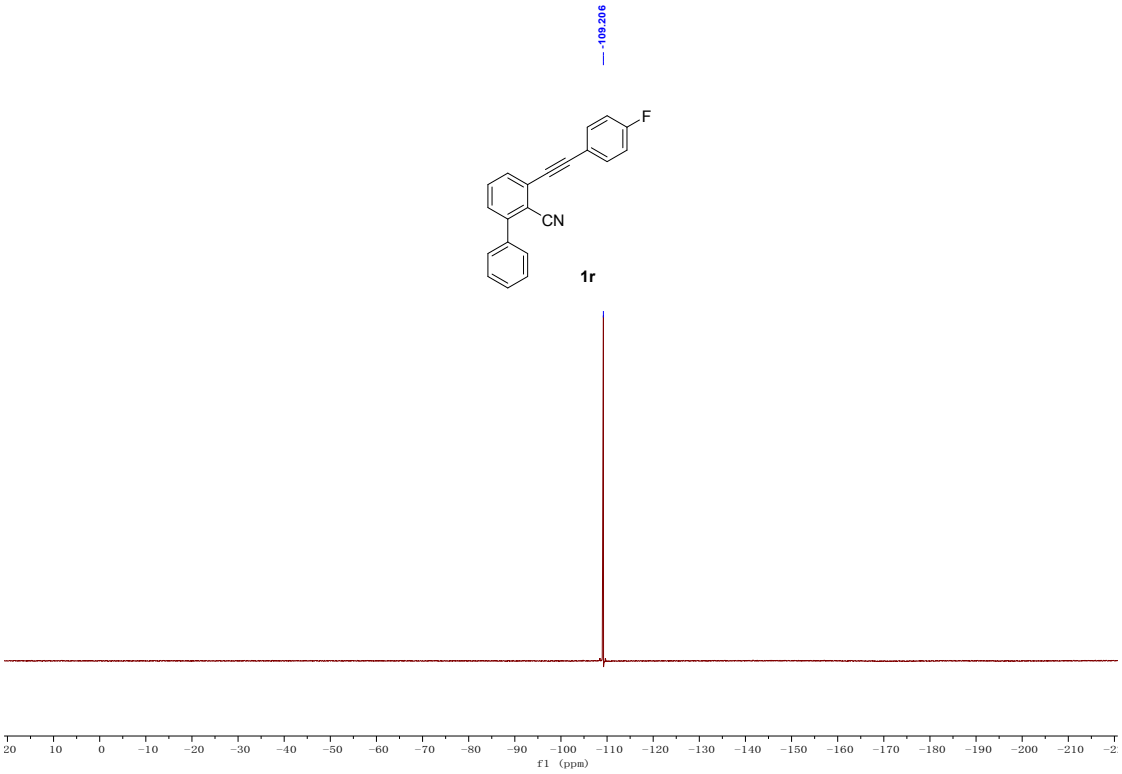
7.094
7.089
7.080
7.076
7.072
7.063
7.058
7.053
0.005
F
CN
1r
6.02
4.07
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


-109.206
F
CN
1r
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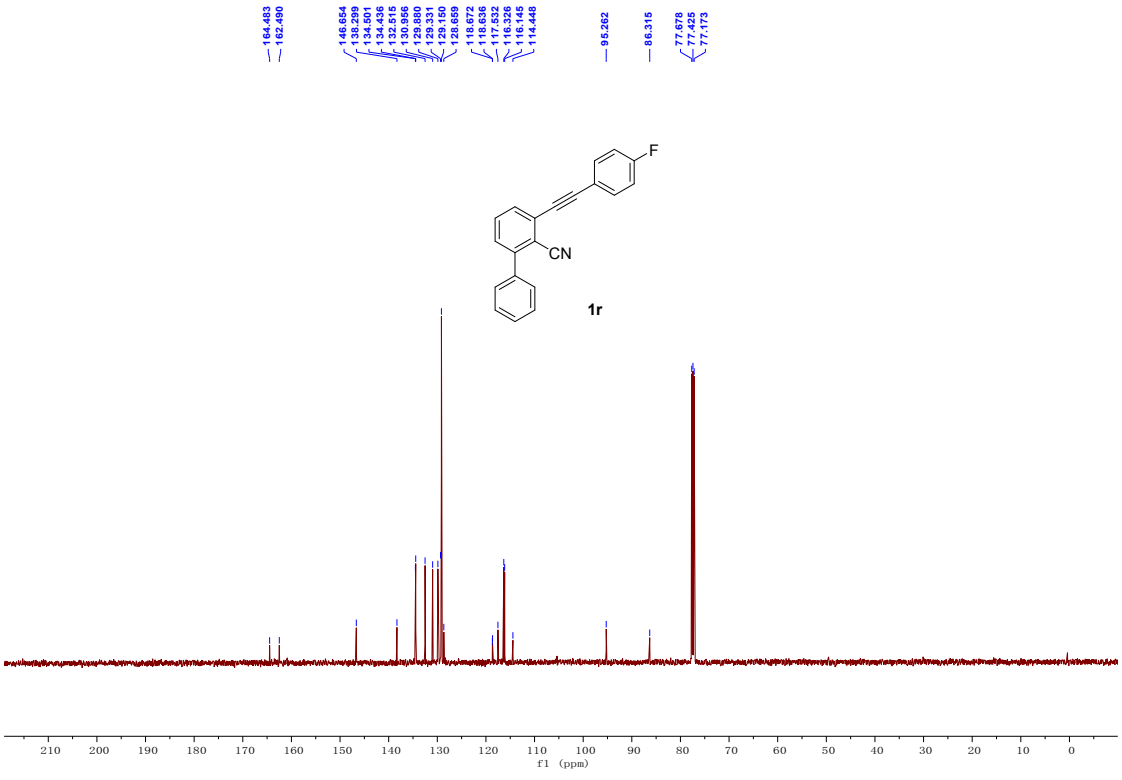
f1 (ppm)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129.150
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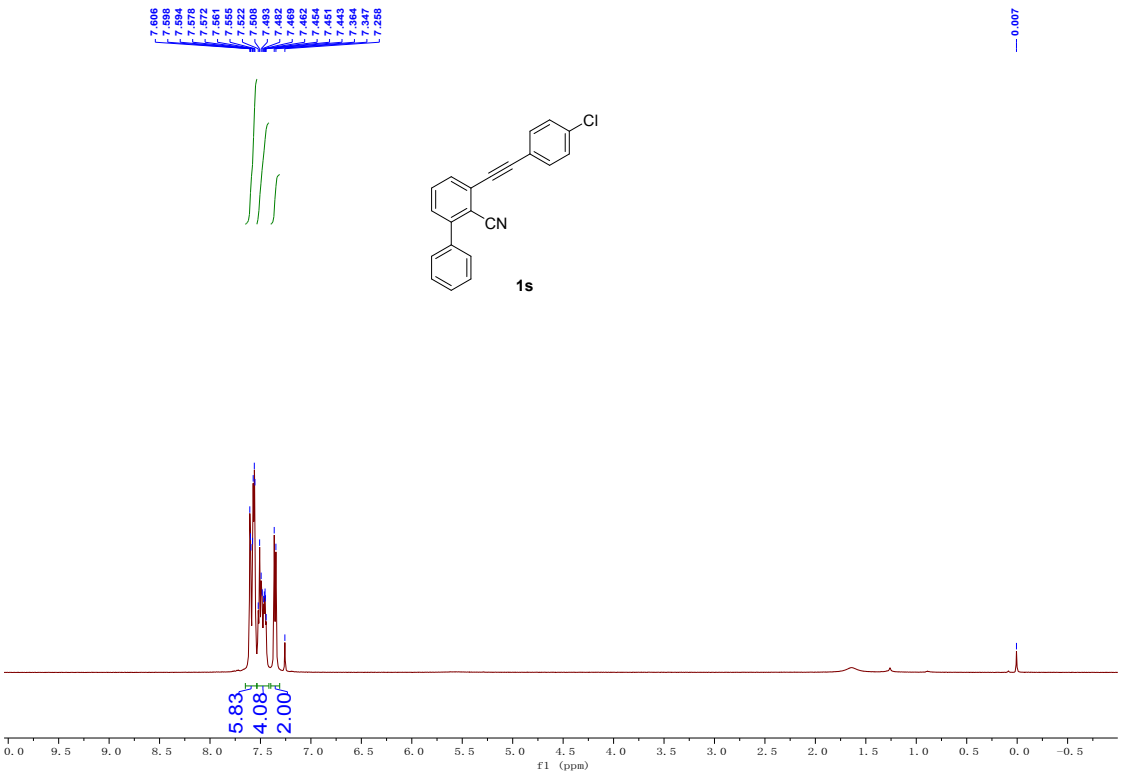
118.672
118.636
117.532
116.326
115.445
114.448
95.262
86.315
77.678
77.425
77.173
F
CN
1r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.462
7.454
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7.443
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7.347
7.258
0.007
Cl
CN
1s
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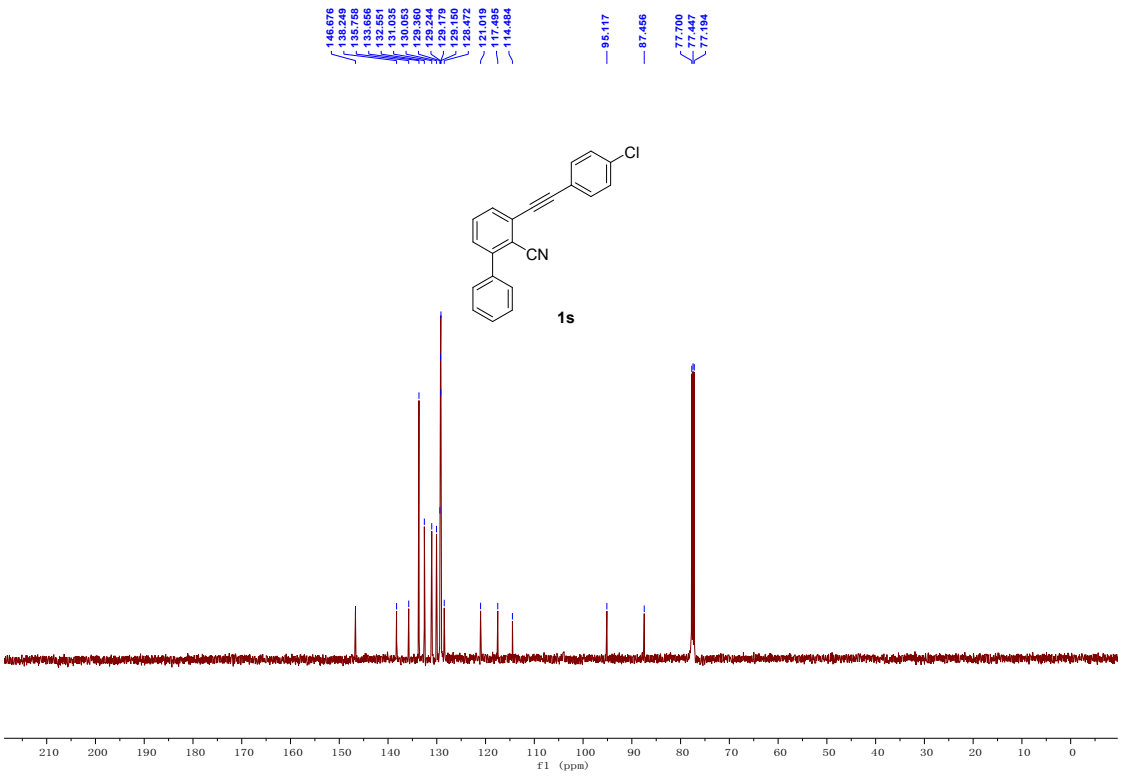
4.08
2.00
f1 (ppm)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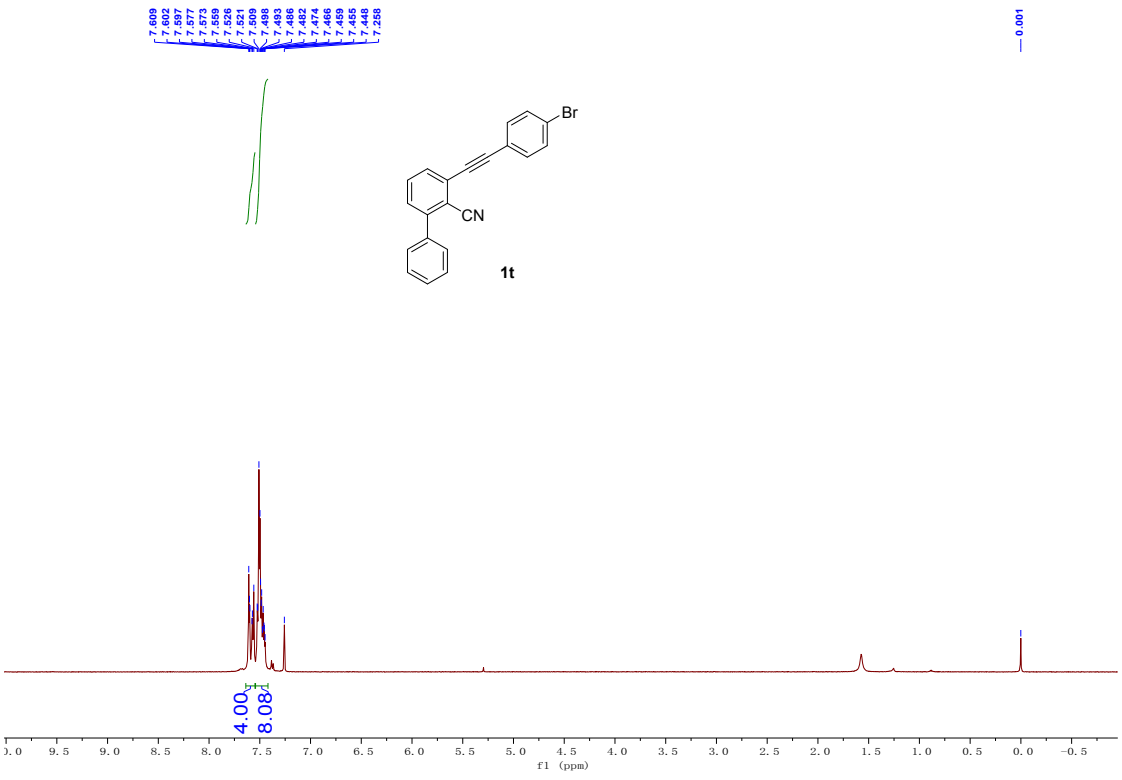
95.117
87.456
77.700
77.447
77.194
Cl
CN
1s
f1 (ppm)


7.609
7.602
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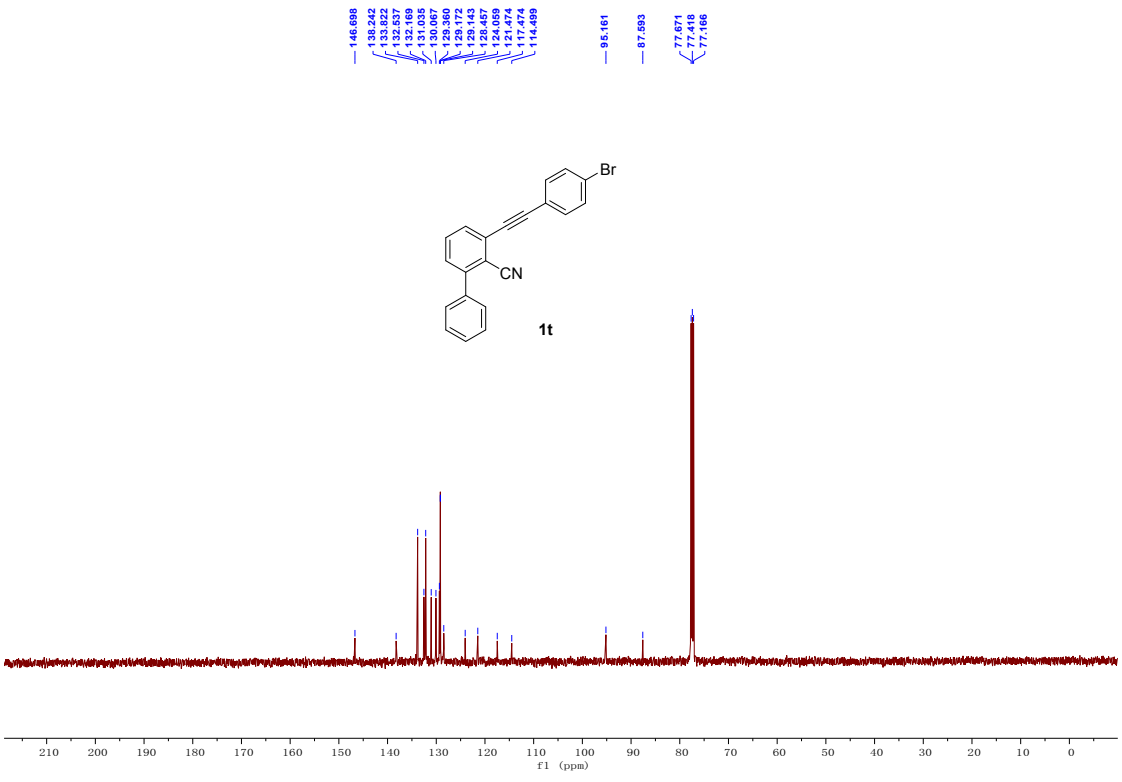
7.498
7.493
7.486
7.482
7.474
7.466
7.459
7.445
7.446
7.258
0.001
Br
CN
1t
4.00
8.08
f1 (ppm)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132.169
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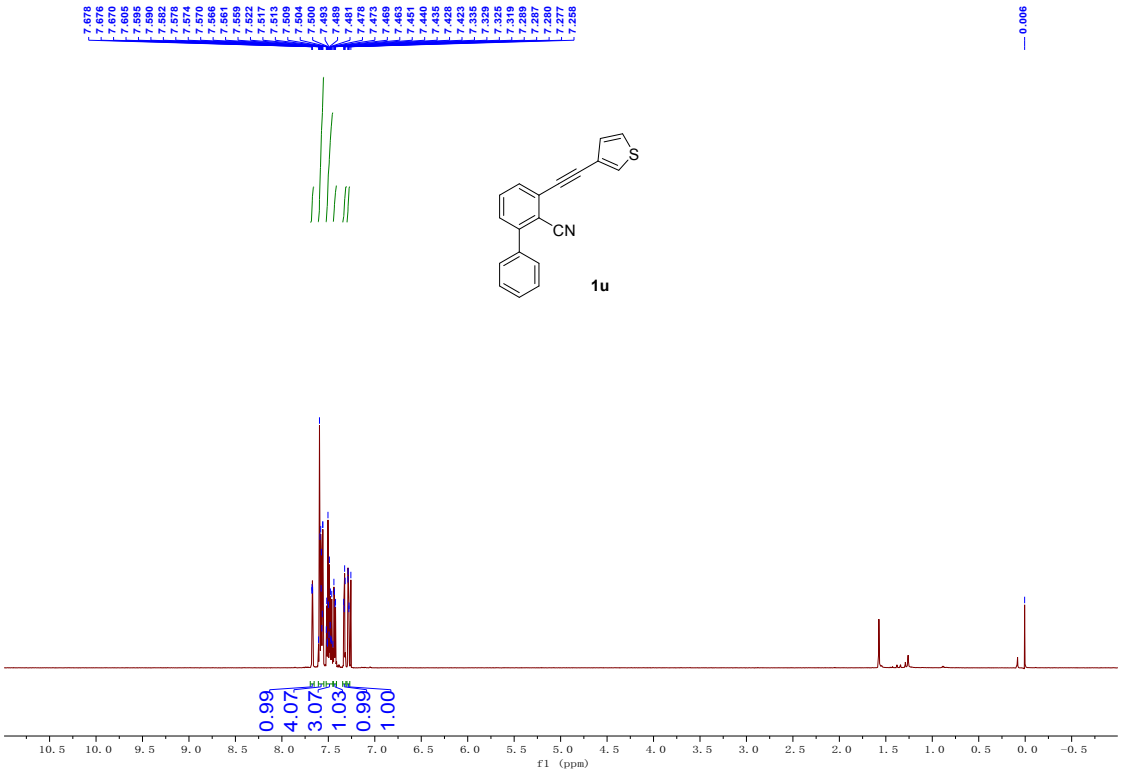
130.067
129.360
129.172
129.143
128.457
124.059
121.474
117.474
114.499
95.161
87.593
77.671
77.418
77.166
Br
CN
1t
f1 (ppm)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7.578
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7.566
7.561
7.559
7.522
7.517
7.513
7.509
7.504
7.500
7.493
7.489
7.481
7.478
7.473
7.469
7.463
7.451
7.440
7.435
7.426
7.423
7.335
7.329
7.325
7.319
7.289
7.287
7.280
7.277
7.258
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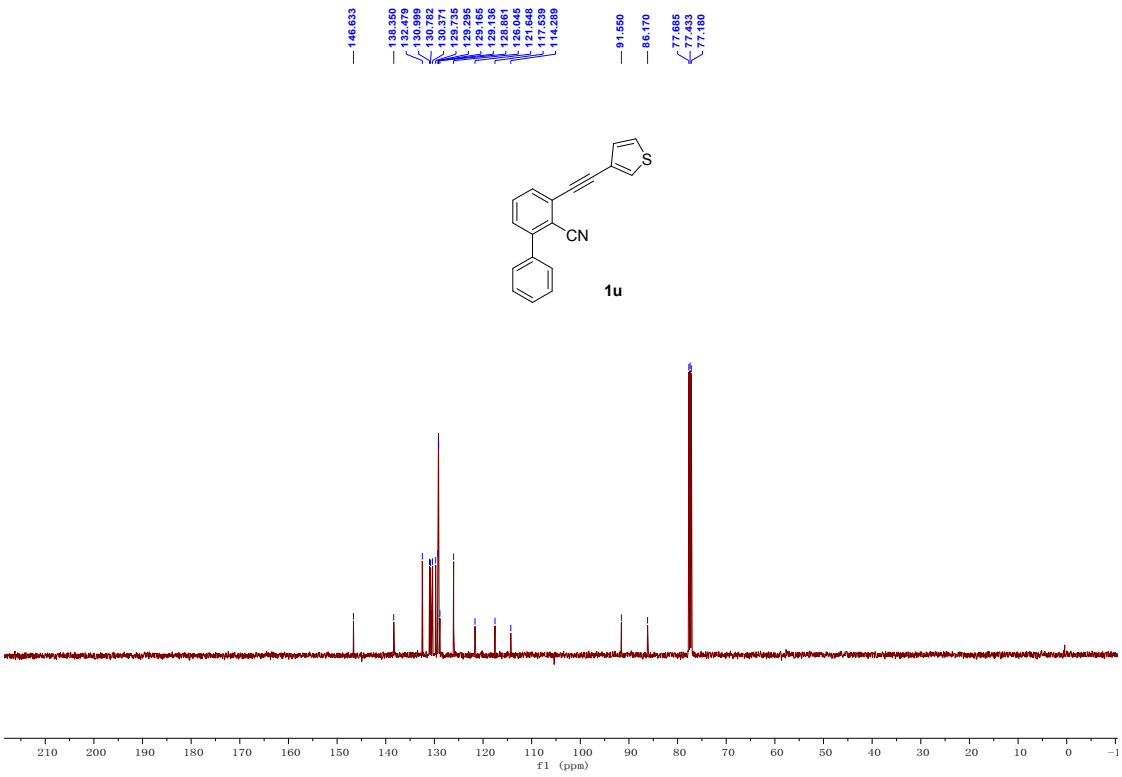
S
CN
1u
0.99
4.07
3.07
1.03
0.99
1.00
f1 (ppm)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129.165
128.556
128.861
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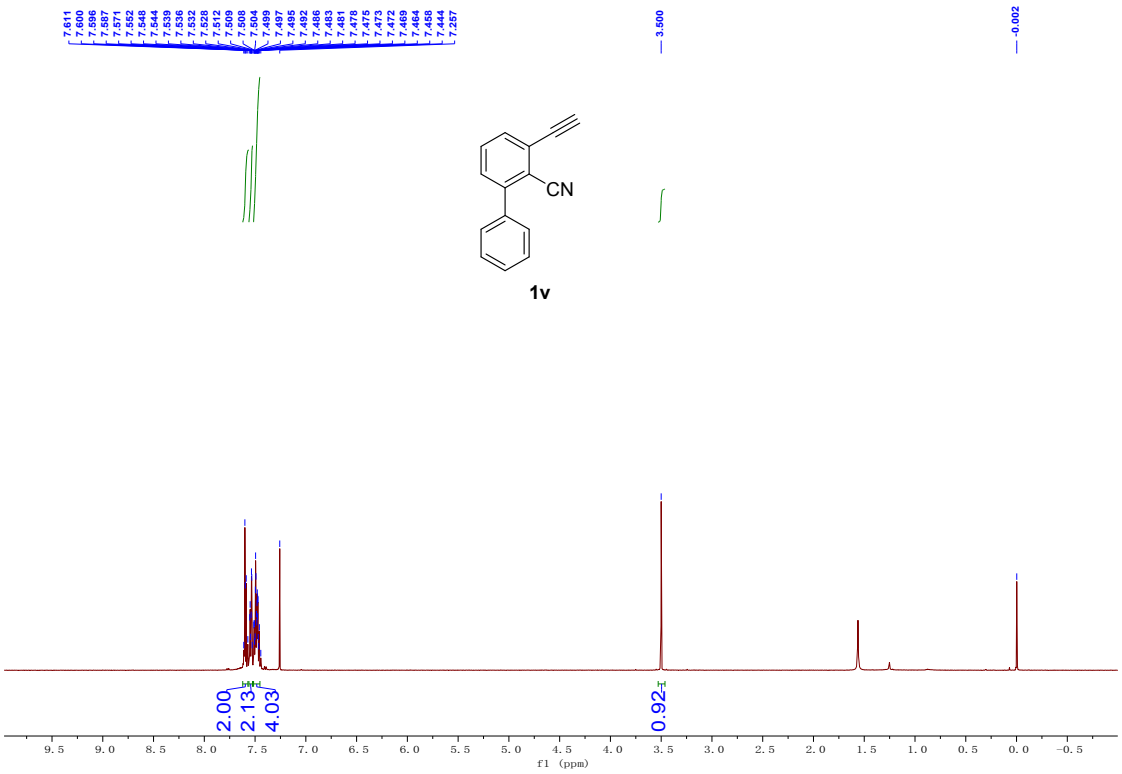
114.289
91.550
86.170
77.685
77.433
77.180
S
CN
1u
f1 (ppm)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7.587
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7.536
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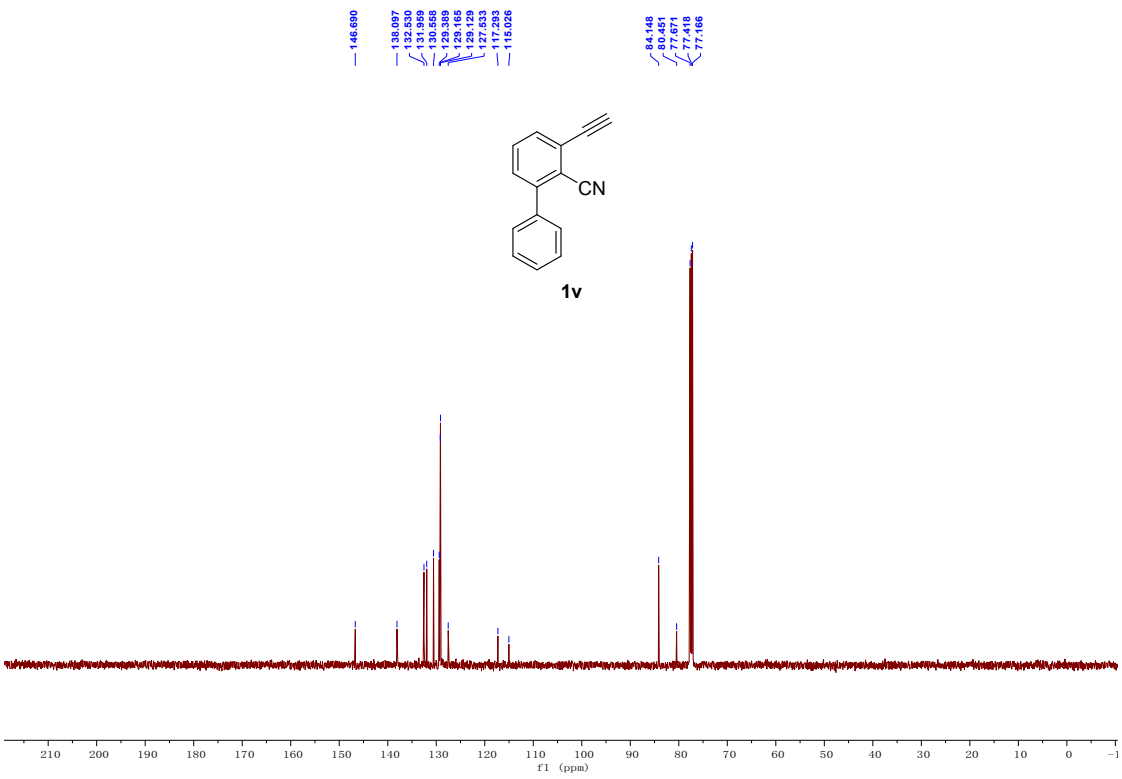
7.528
7.512
7.509
7.508
7.504
7.499
7.497
7.495
7.492
7.486
7.483
7.481
7.478
7.475
7.473
7.472
7.469
7.464
7.458
7.444
7.257
3.500
0.002
CN
1v
2.00
2.13
4.03
0.92
f1 (ppm)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129.165
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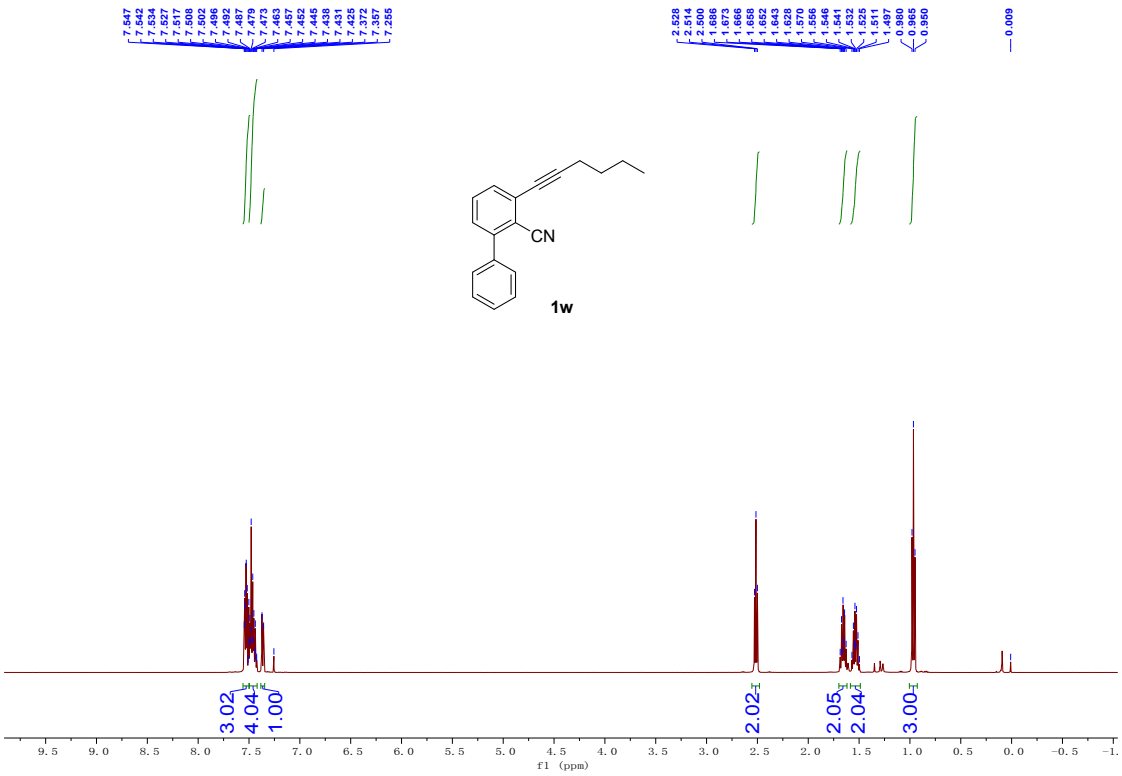
116.026
84.148
80.451
77.577
77.415
77.166
CN
1v
f1 (ppm)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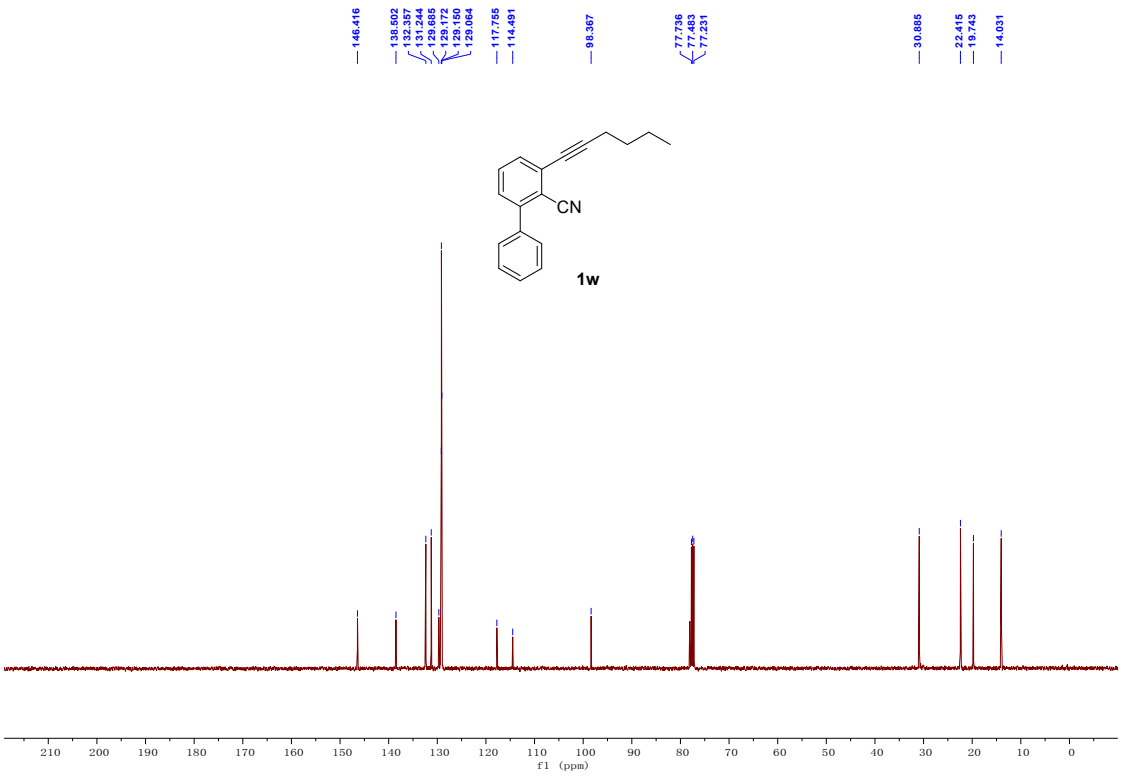
7.527
7.517
7.508
7.502
7.496
7.492
7.487
7.479
7.473
7.463
7.457
7.452
7.445
7.438
7.431
7.425
7.372
7.357
7.255
2.528
2.514
2.500
1.686
1.673
1.666
1.658
1.652
1.643
1.628
1.570
1.556
1.546
1.541
1.532
1.525
1.511
1.497
0.980
0.965
0.950
0.009
CN
1w
3.02
4.04
1.00
2.02
2.05
2.04
3.00
f1 (ppm)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129.172
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129.064
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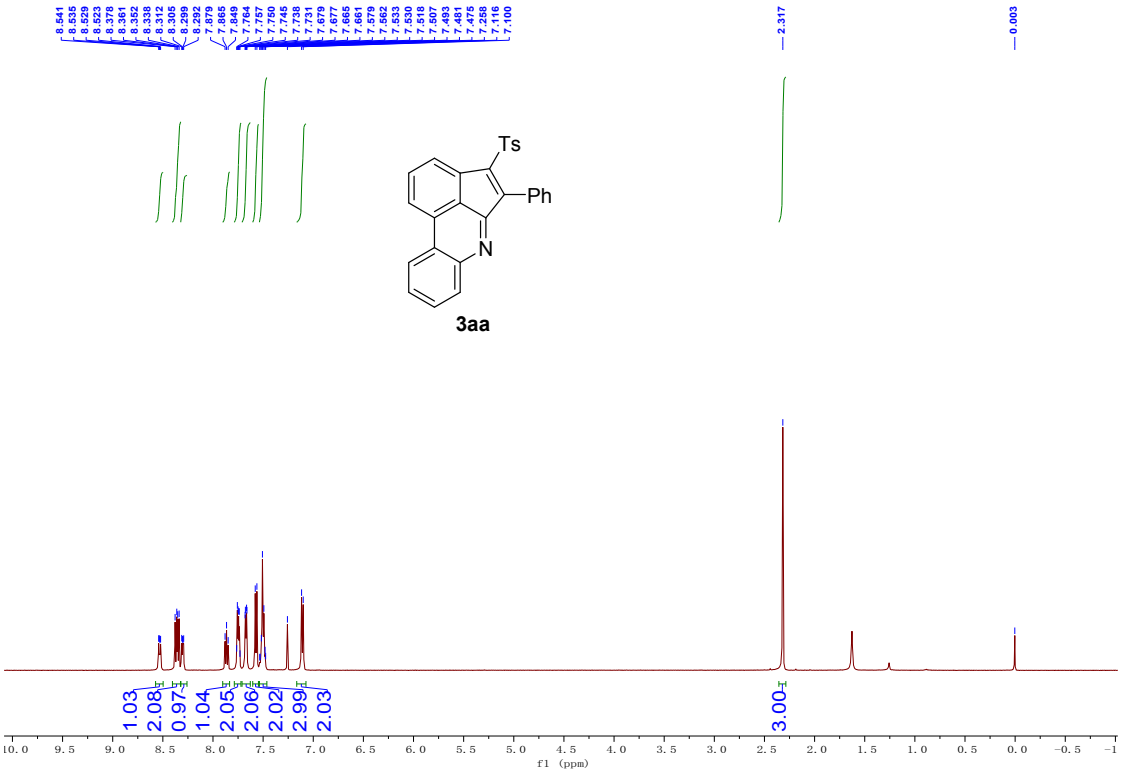
77.483
77.231
30.885
22.415
19.743
14.031
CN
1w
f1 (ppm)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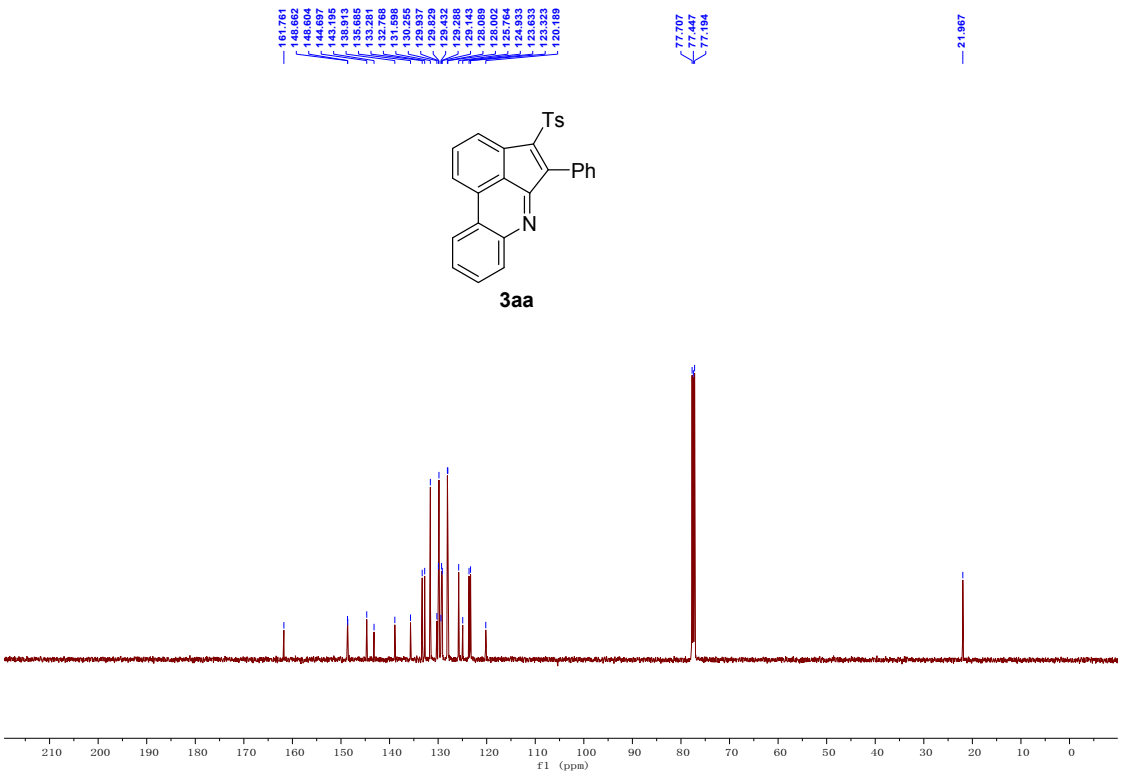
8.292
7.879
7.865
7.849
7.764
7.757
7.750
7.745
7.738
7.731
7.679
7.677
7.665
7.661
7.579
7.562
7.533
7.510
7.516
7.507
7.493
7.481
7.475
7.258
7.116
7.100
2.317
0.003
Ts
Ph
N
3aa
1.03
2.08
0.97
1.04
2.05
2.06
2.02
2.99
2.03
3.00
f1 (ppm)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138.913
135.685
133.281
132.768
131.698
130.255
129.937
129.829
129.432
129.288
129.143
128.089
128.002
125.764
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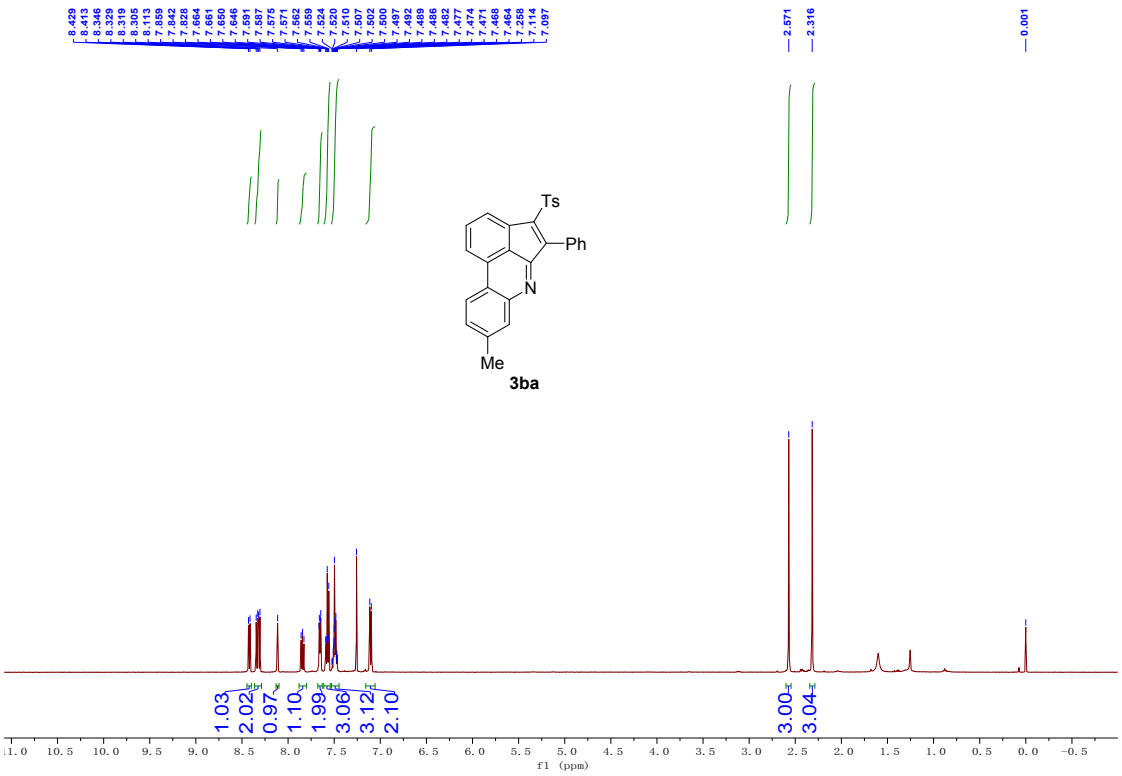
123.633
123.323
120.189
77.707
77.447
77.194
21.967
Ts
Ph
N
3aa
f1 (ppm)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8.319
8.305
8.113
7.859
7.842
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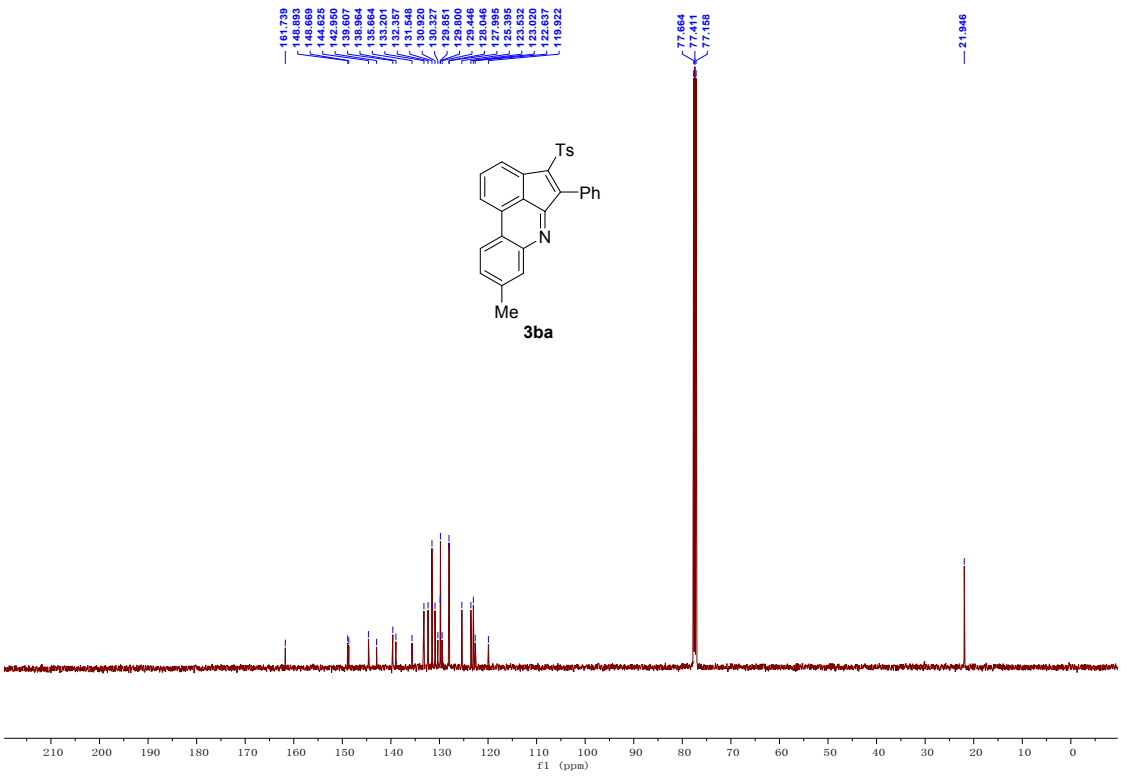
7.661
7.650
7.646
7.591
7.587
7.575
7.571
7.552
7.550
7.524
7.520
7.510
7.507
7.502
7.500
7.497
7.492
7.489
7.486
7.482
7.477
7.474
7.471
7.468
7.464
7.458
7.114
7.097
2.571
2.316
0.001
Ts
Ph
N
Me
3ba
1.03
2.02
0.97
1.10
1.99
3.06
3.12
2.10
3.00
3.04
f1 (ppm)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148.669
144.625
142.950
139.607
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133.201
132.357
131.548
130.920
130.327
129.851
129.800
129.446
128.046
127.995
126.395
123.532
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77.664
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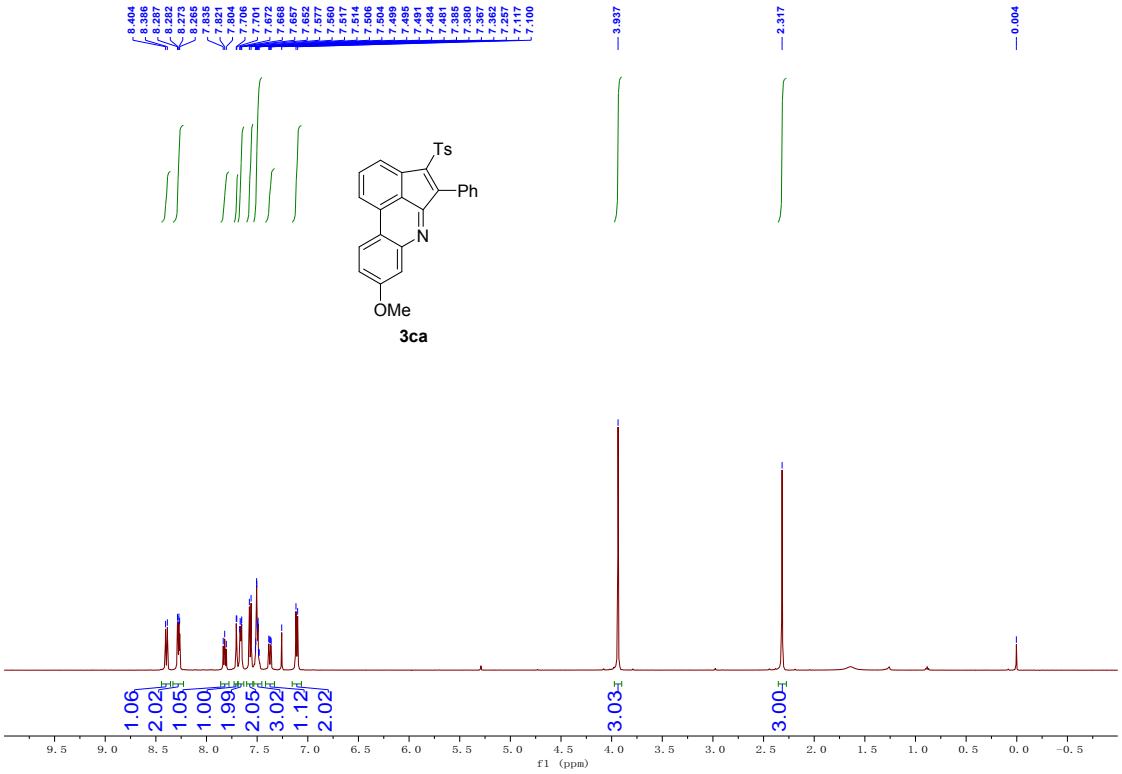
21.946
Ts
Ph
N
Me
3ba
f1 (ppm)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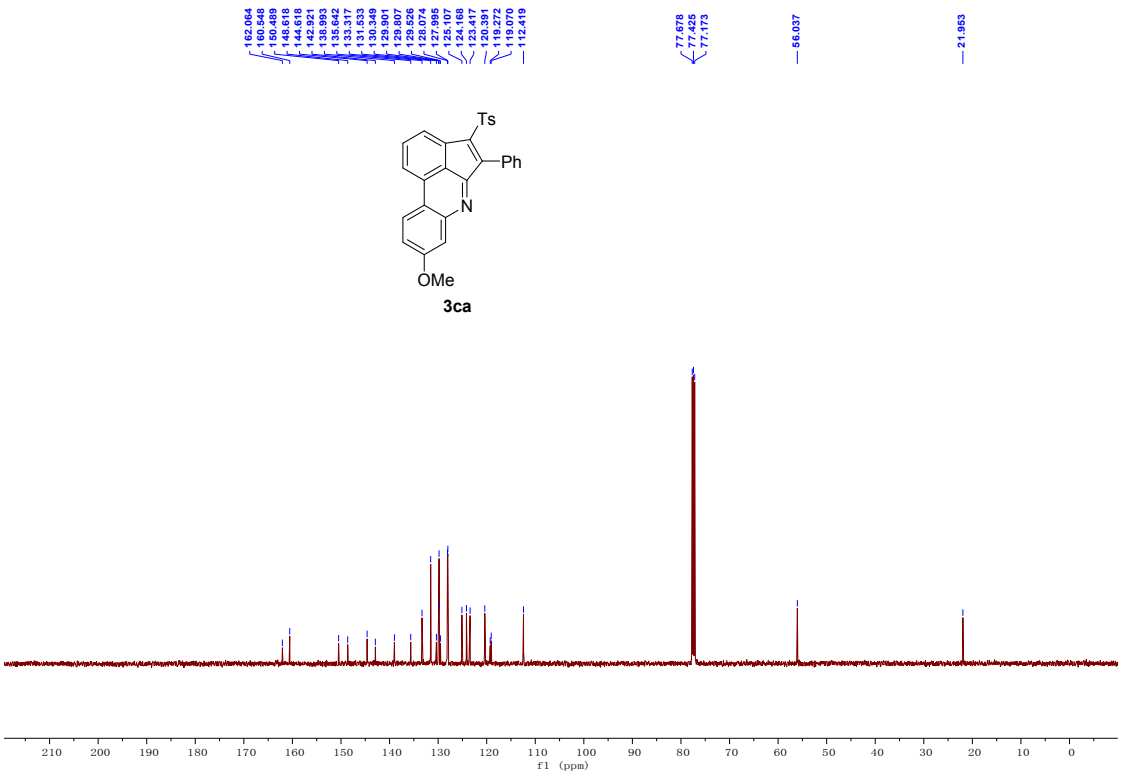
8.287
8.273
8.265
7.835
7.821
7.804
7.706
7.701
7.672
7.668
7.657
7.652
7.577
7.560
7.517
7.514
7.506
7.504
7.499
7.495
7.491
7.484
7.481
7.385
7.380
7.367
7.362
7.357
7.117
7.100
3.937
2.317
0.004
Ts
Ph
N
OMe
1.06
2.02
1.05
1.00
1.99
2.05
3.02
1.12
2.02
3.03
3.00
f1 (ppm)

3ca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144.618
142.921
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135.642
133.317
131.533
130.349
129.901
129.807
129.526
129.074
127.995
125.107
124.168
123.417
120.391
119.272
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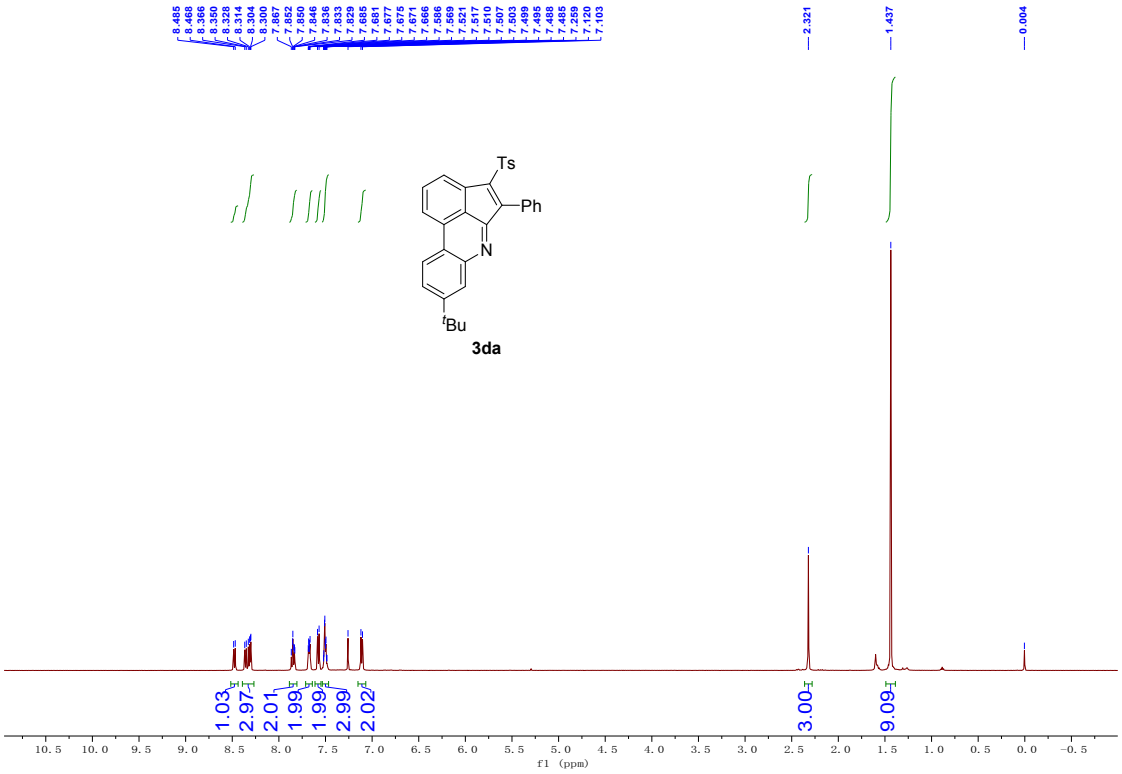
112.419
77.678
77.425
77.173
56.037
21.953
Ts
Ph
N
OMe
f1 (ppm)

3ca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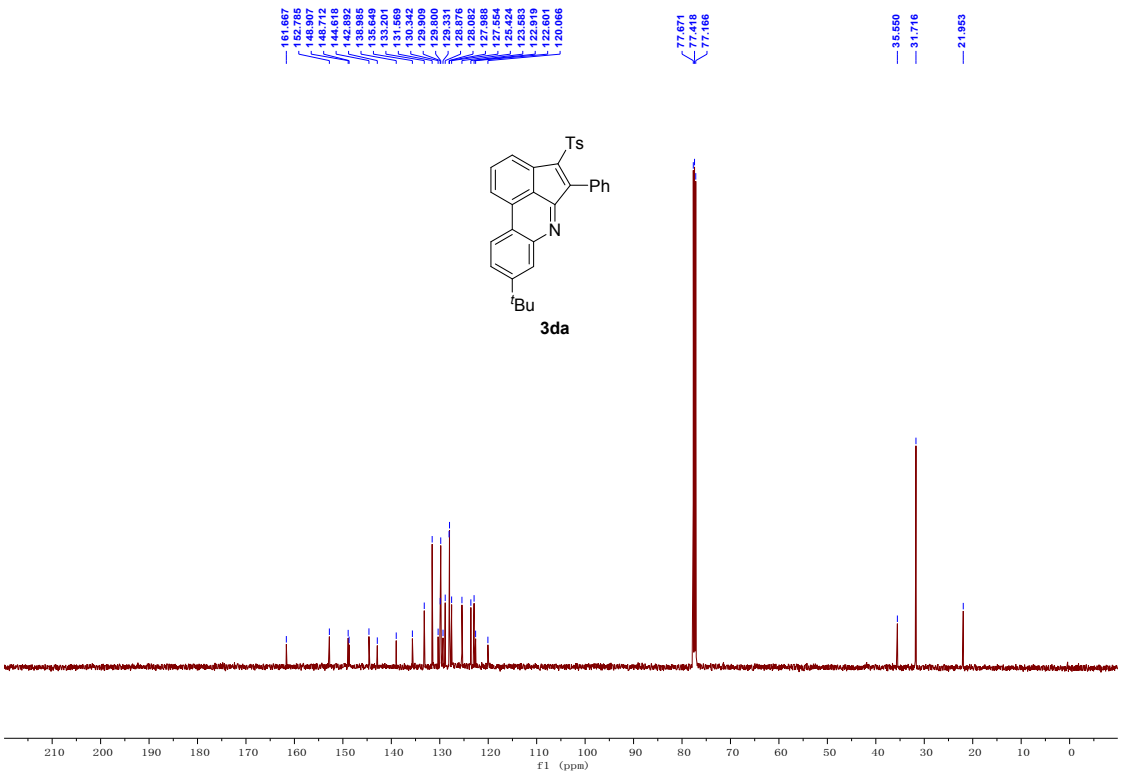
8.300
7.867
7.852
7.850
7.846
7.836
7.833
7.829
7.685
7.681
7.677
7.675
7.671
7.666
7.586
7.569
7.521
7.517
7.510
7.507
7.503
7.499
7.495
7.488
7.445
7.259
7.120
7.103
2.321
1.437
0.004
Ts
Ph
N
tBu
3da
1.03
2.97
2.01
1.99
1.99
2.99
2.02
3.00
9.09
f1 (ppm)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129.331
128.876
128.082
127.988
127.554
125.424
123.563
122.519
122.601
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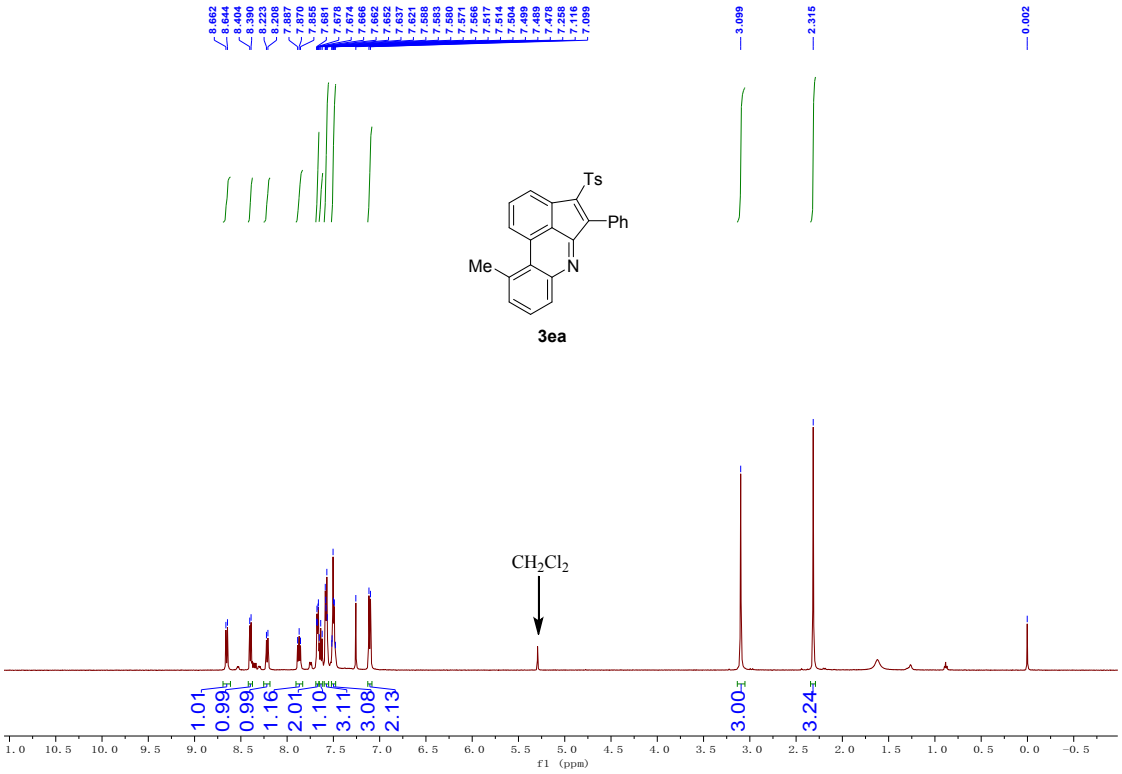
77.418
77.166
35.550
31.716
21.953
Ts
Ph
N
tBu
3da
f1 (ppm)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7.676
7.674
7.666
7.662
7.652
7.637
7.621
7.588
7.583
7.580
7.571
7.566
7.517
7.514
7.504
7.499
7.489
7.478
7.358
7.116
7.099
3.099
2.315
0.002
Ts
Ph
Me
N
3ea
CH2Cl2
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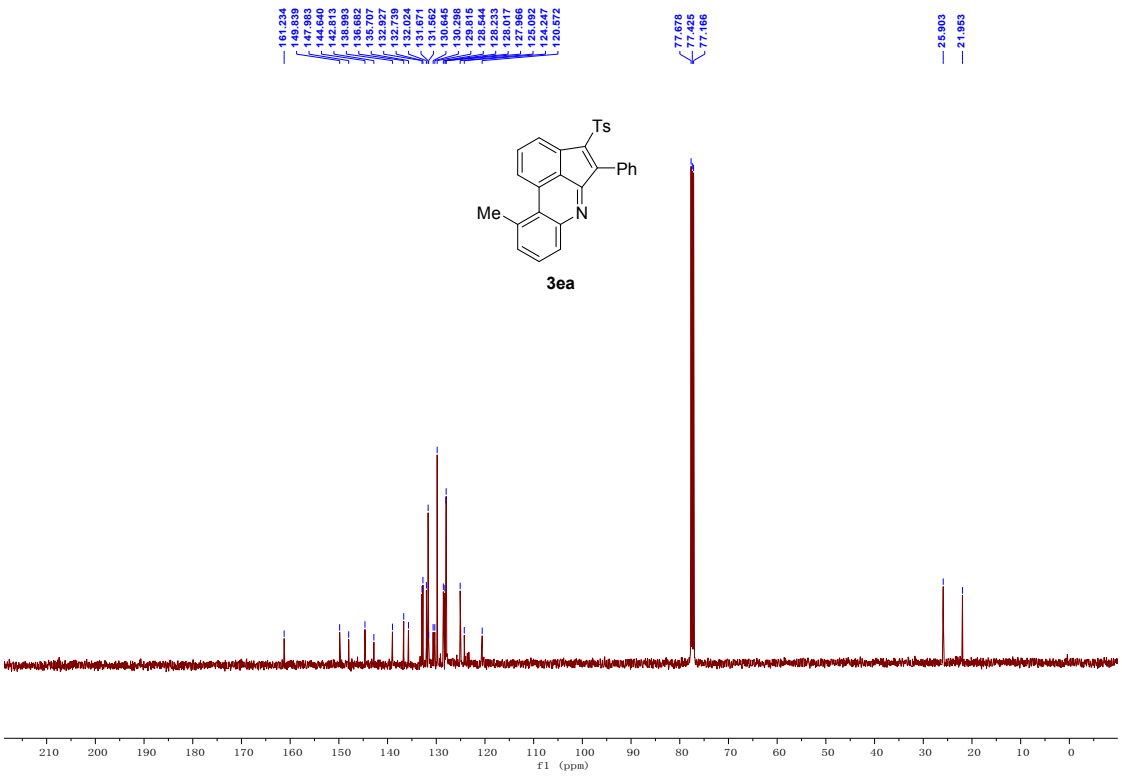
2.01
1.10
3.11
3.08
2.13
3.00
3.24
f1 (ppm)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130.645
130.298
129.815
128.544
128.233
128.017
127.966
125.092
124.247
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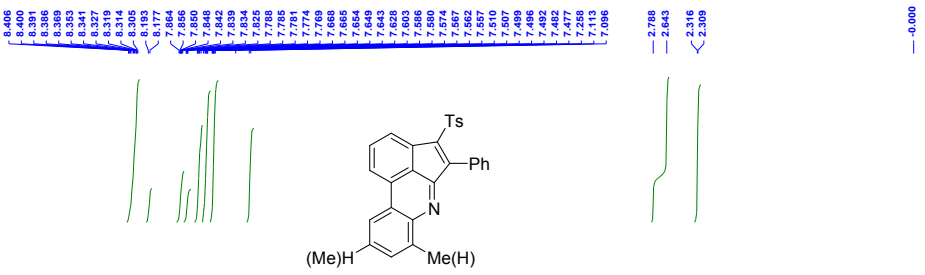
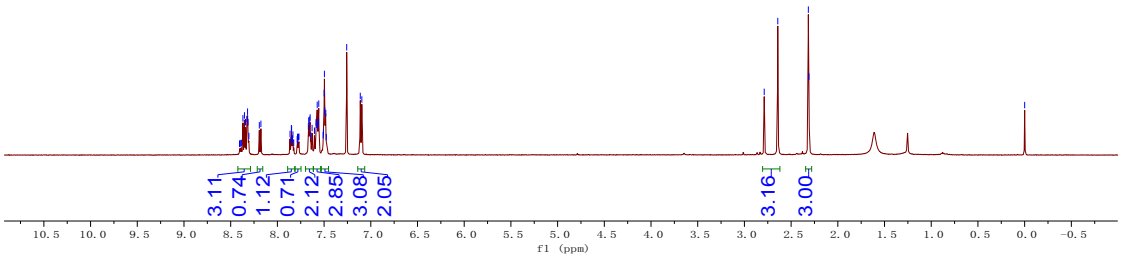
77.678
77.425
77.166
25.903
21.953
Ts
Ph
Me
N
3ea
f1 (ppm)



3fa+3fa'



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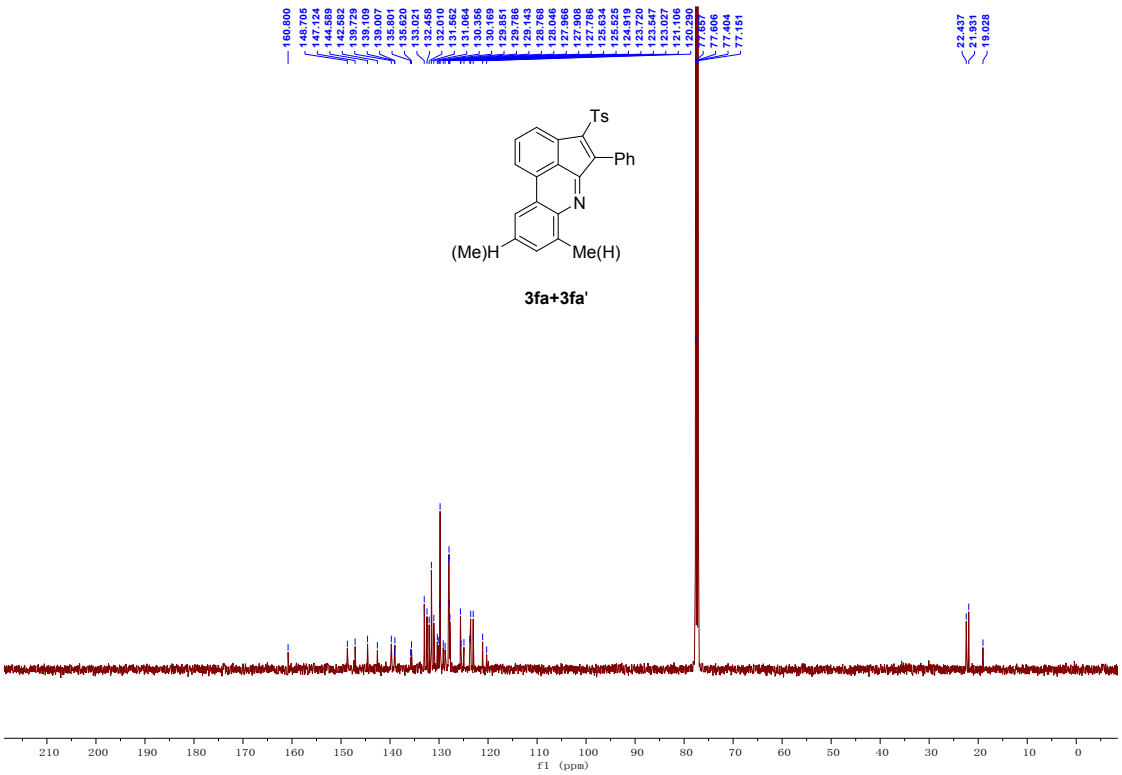

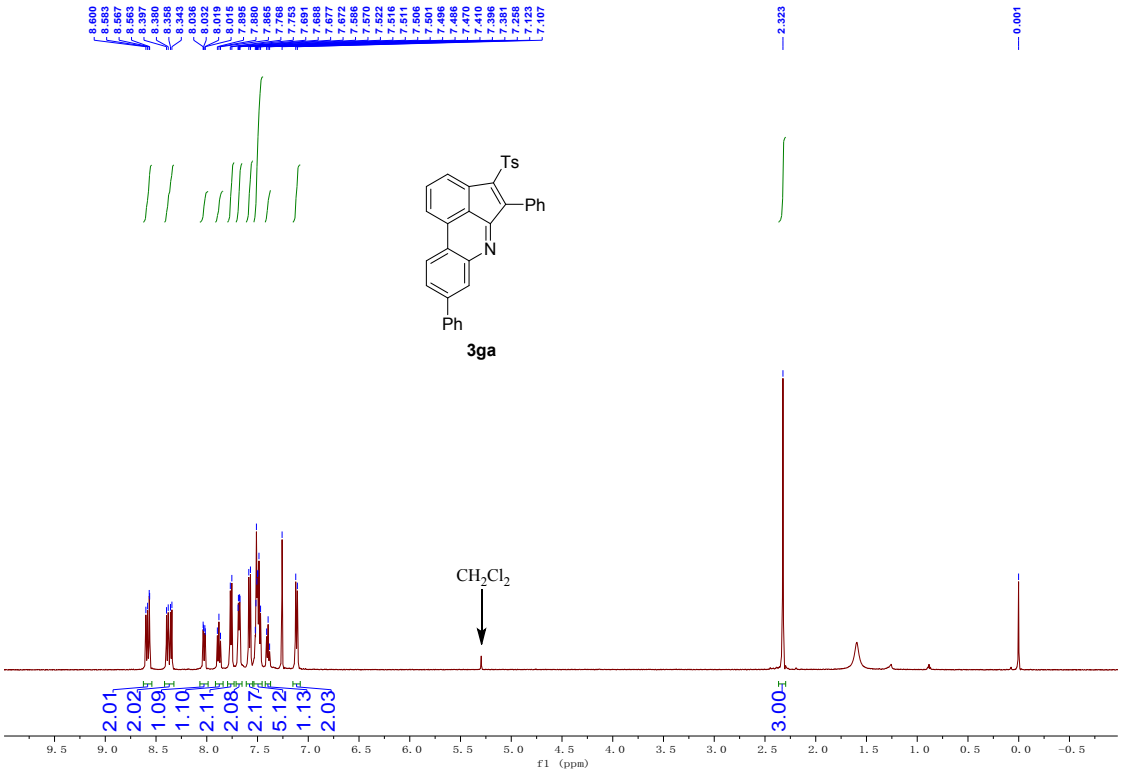
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7.880
7.865
7.768
7.753
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7.677
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7.586
7.570
7.522
7.516
7.511
7.506
7.501
7.496
7.486
7.470
7.410
7.395
7.381
7.258
7.123
7.107
2.323
0.001
Ts
Ph
N
Ph
3ga
CH2Cl2
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2.02
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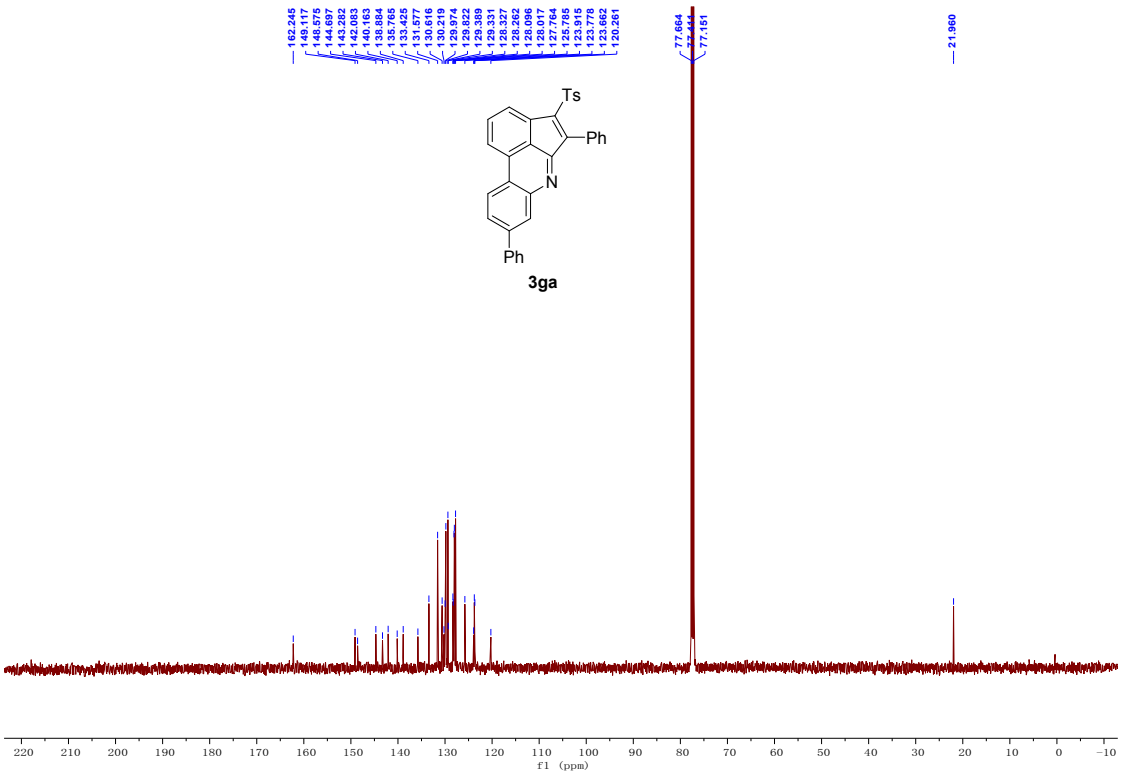
1.10
2.11
2.08
2.17
5.12
1.13
2.03
3.00
f1 (ppm)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130.219
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129.822
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128.327
128.262
128.096
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127.764
125.785
123.915
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77.444
77.151
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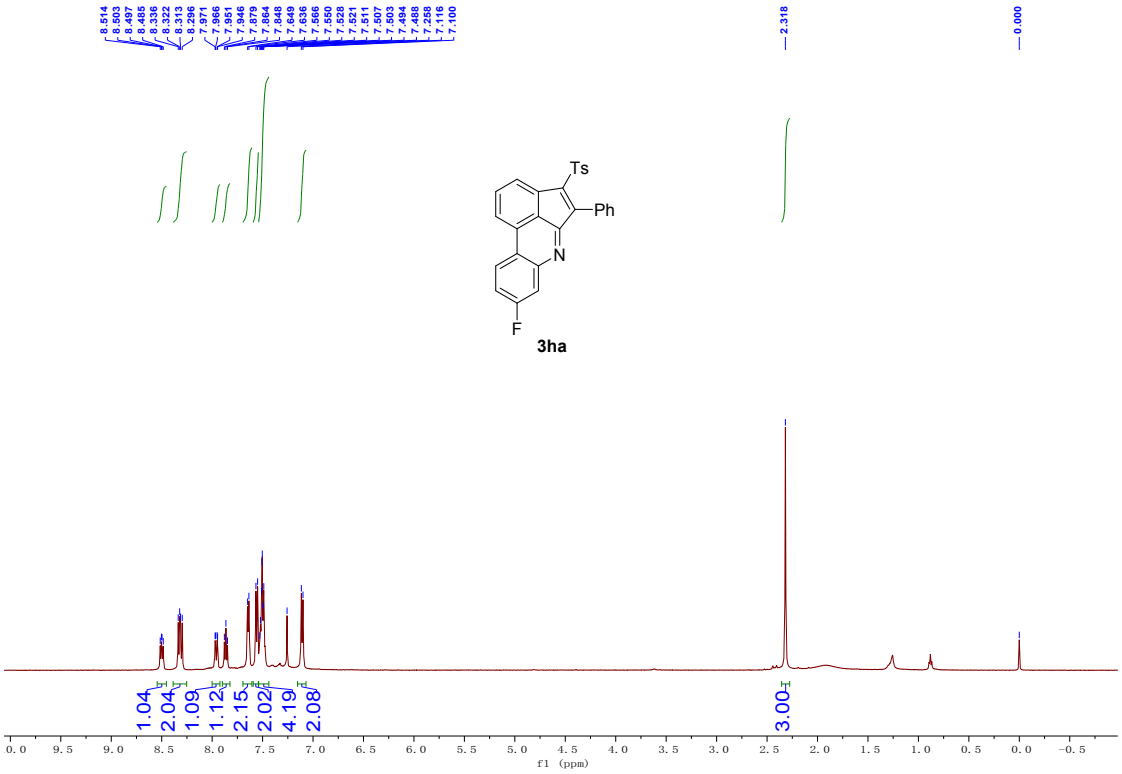
Ts
Ph
N
Ph
3ga
f1 (ppm)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7.966
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7.846
7.839
7.864
7.848
7.649
7.636
7.566
7.550
7.528
7.521
7.511
7.507
7.503
7.494
7.488
7.258
7.116
7.100
2.318
0.000
Ts
Ph
N
F
3ha
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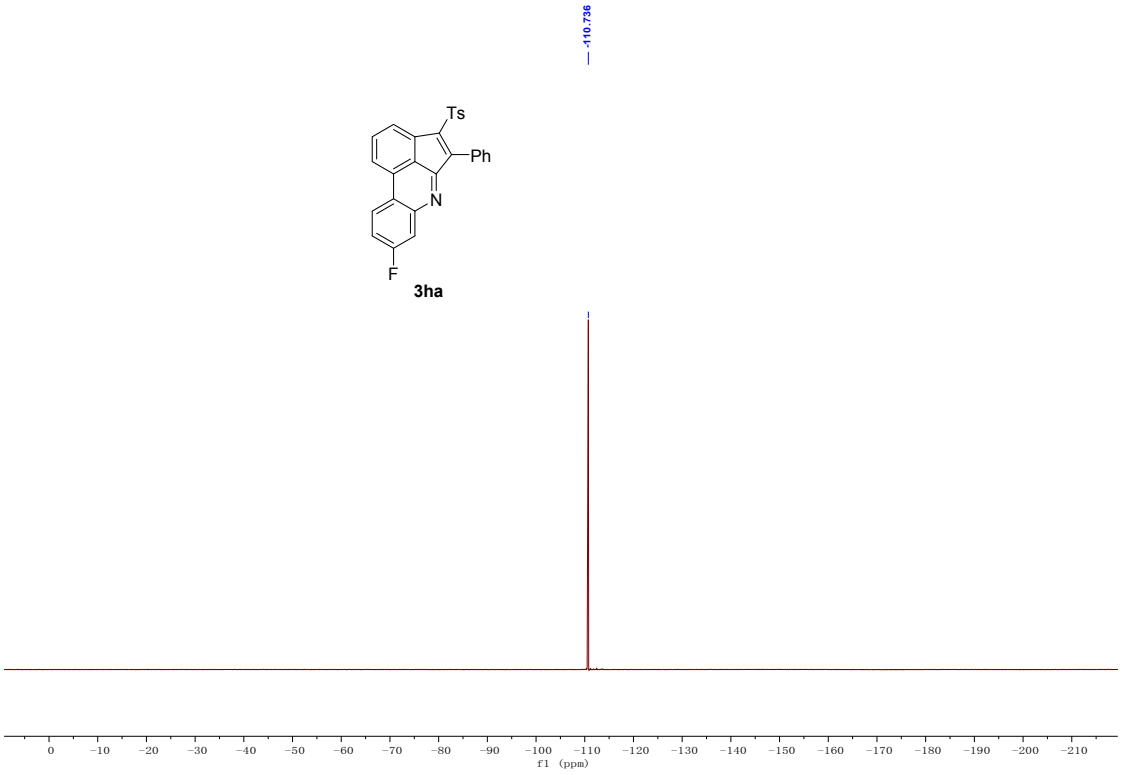
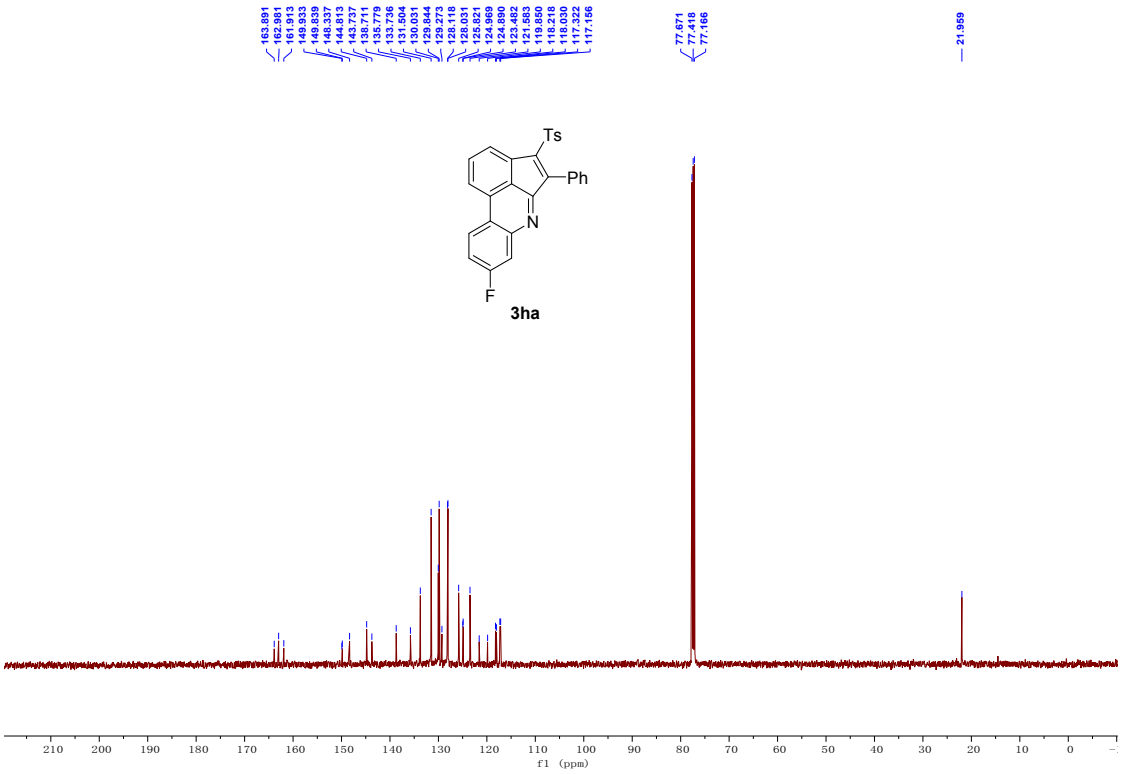
2.04
1.09
1.12
2.15
2.02
4.19
2.08
3.00
f1 (ppm)


-110.736
Ts
Ph
N
F
3ha
f1 (ppm)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130.031
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128.031
125.821
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124.890
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119.850
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118.030
117.722
117.166
77.671
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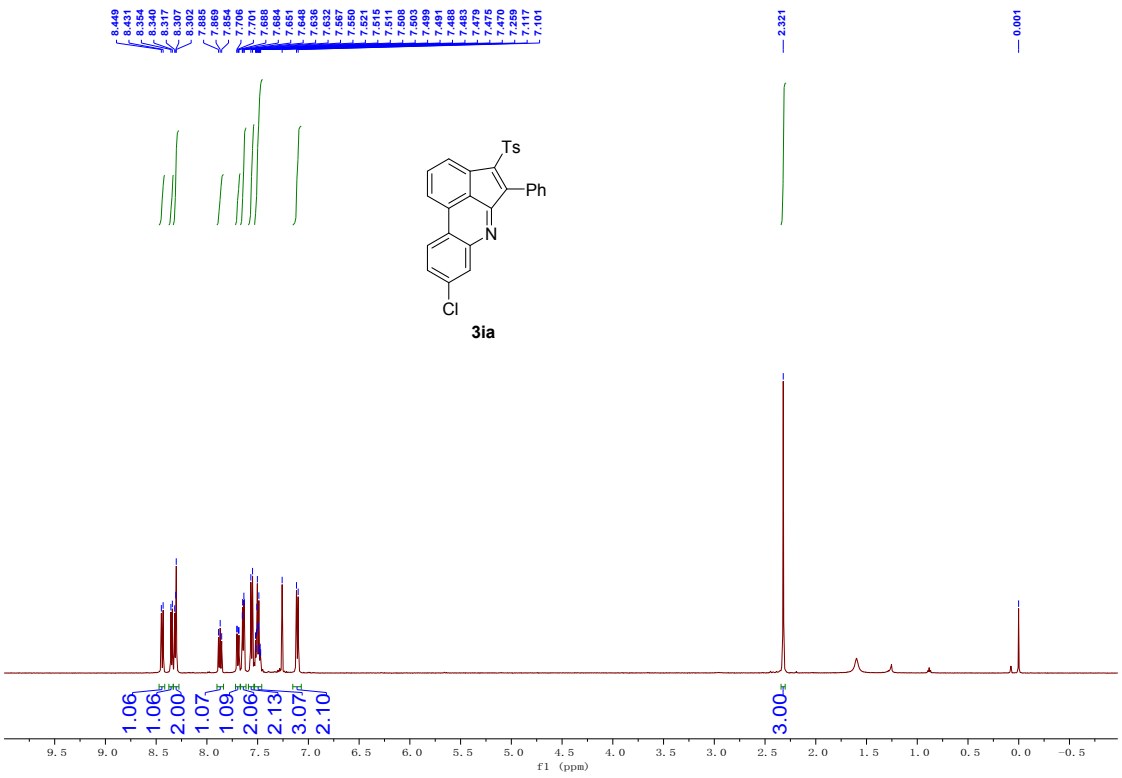
Ts
Ph
N
F
3ha
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.516
7.511
7.508
7.503
7.499
7.491
7.488
7.483
7.479
7.475
7.470
7.269
7.117
7.101
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0.001
Ts
Ph
N
Cl
3ia
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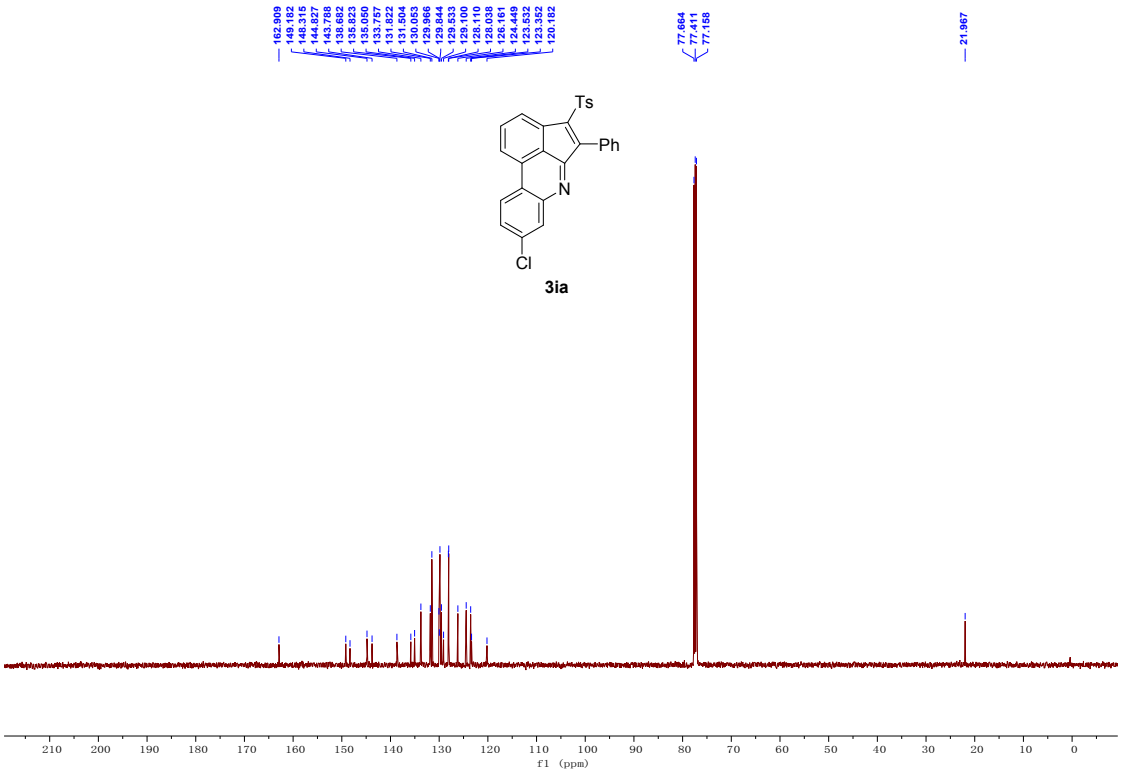
1.06
2.00
1.07
1.09
2.06
2.13
3.07
2.10
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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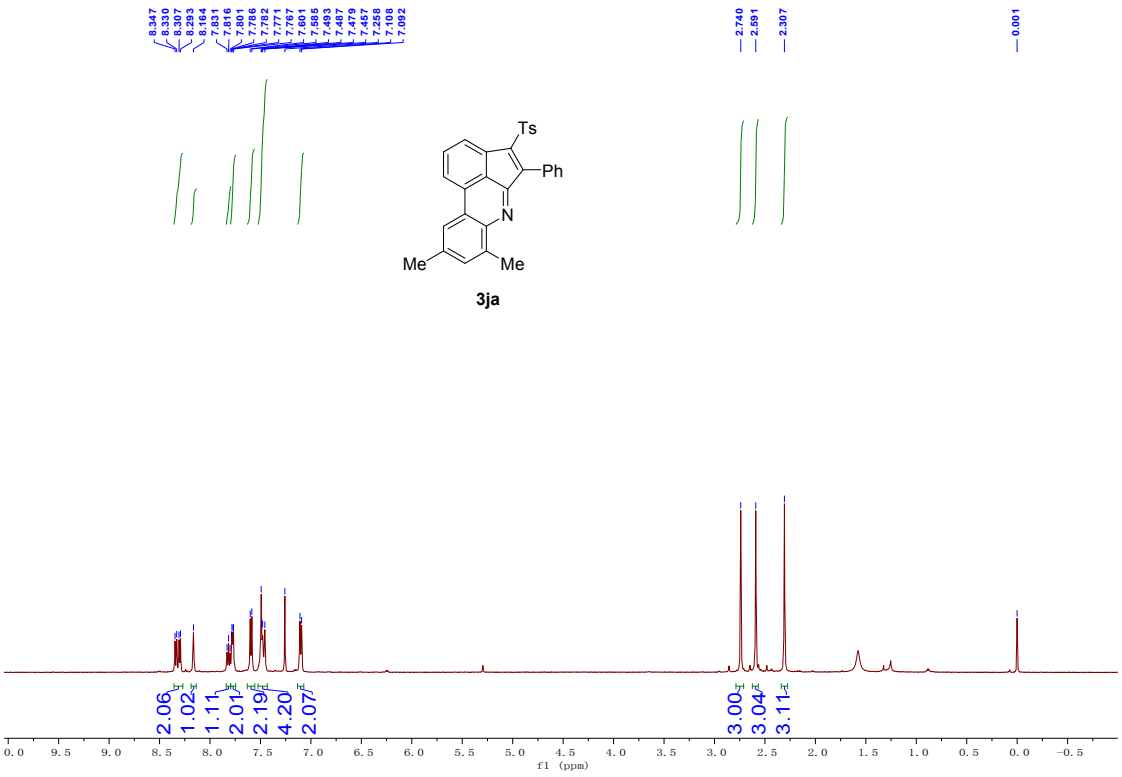
130.053
129.966
129.844
129.553
129.100
128.110
128.038
126.161
124.449
123.532
123.352
120.182
77.664
77.411
77.158
21.967
Ts
Ph
N
Cl
3ia
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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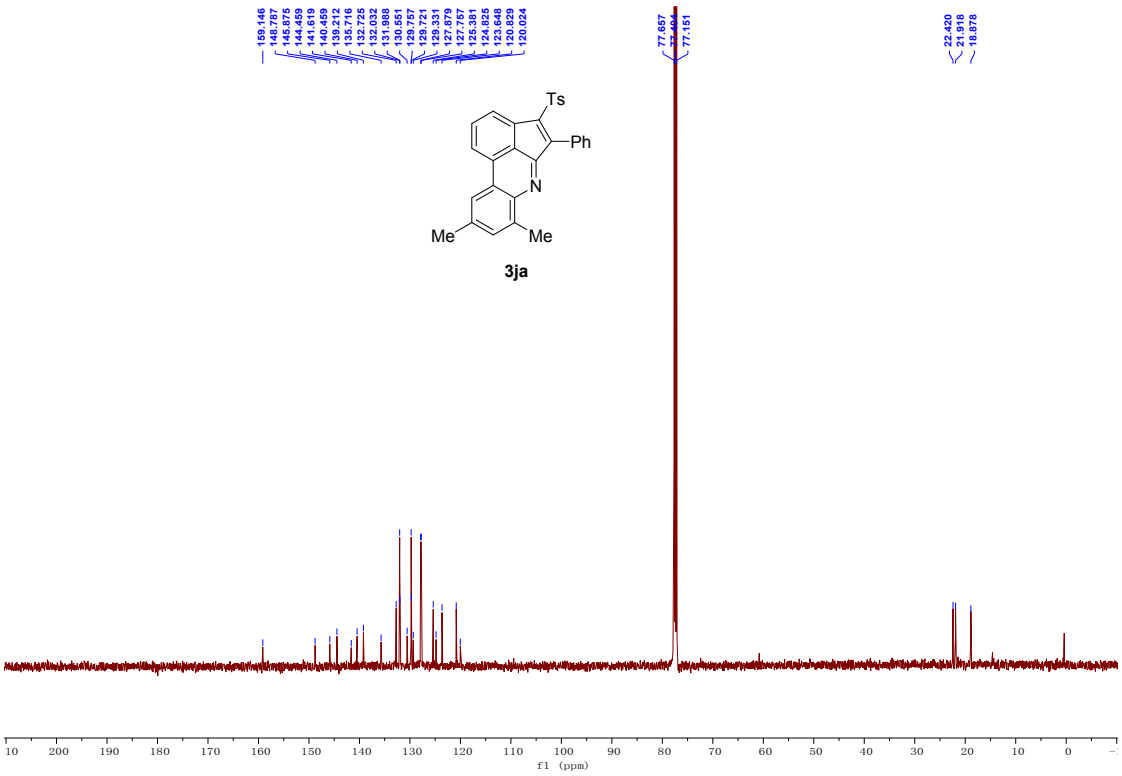
7.771
7.767
7.601
7.585
7.493
7.487
7.479
7.457
7.258
7.108
7.092
2.740
2.591
2.307
0.001
Ts
Ph
N
Me
Me
3ja
2.06
1.02
1.11
2.01
2.19
4.20
2.07
3.00
3.04
3.11
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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129.331
127.879
127.757
125.381
124.825
123.648
120.829
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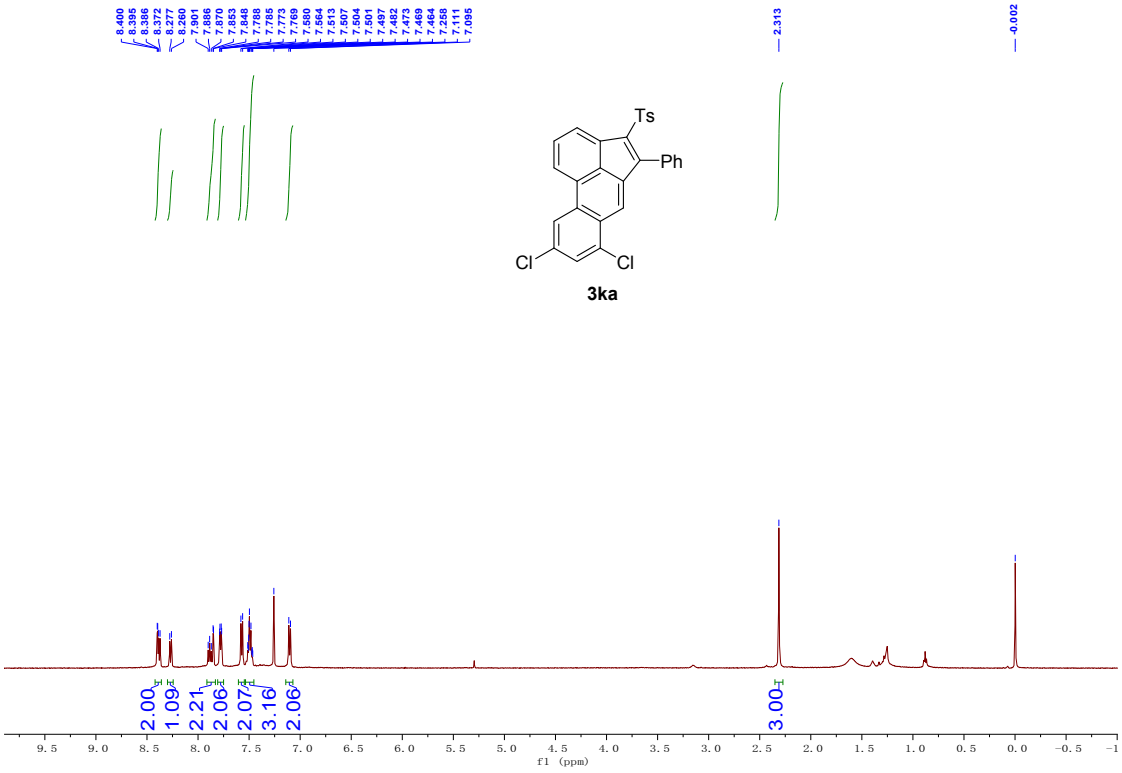
77.161
22.420
21.918
18.878
Ts
Ph
N
Me
Me
3ja
f1 (ppm)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7.901
7.886
7.870
7.853
7.848
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7.785
7.773
7.769
7.680
7.564
7.513
7.507
7.504
7.501
7.497
7.482
7.473
7.469
7.464
7.258
7.111
7.095
2.313
-0.002
Ts
Ph
Cl
Cl
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2.00
1.09
2.21
2.06
2.07
3.16
2.06
3.00
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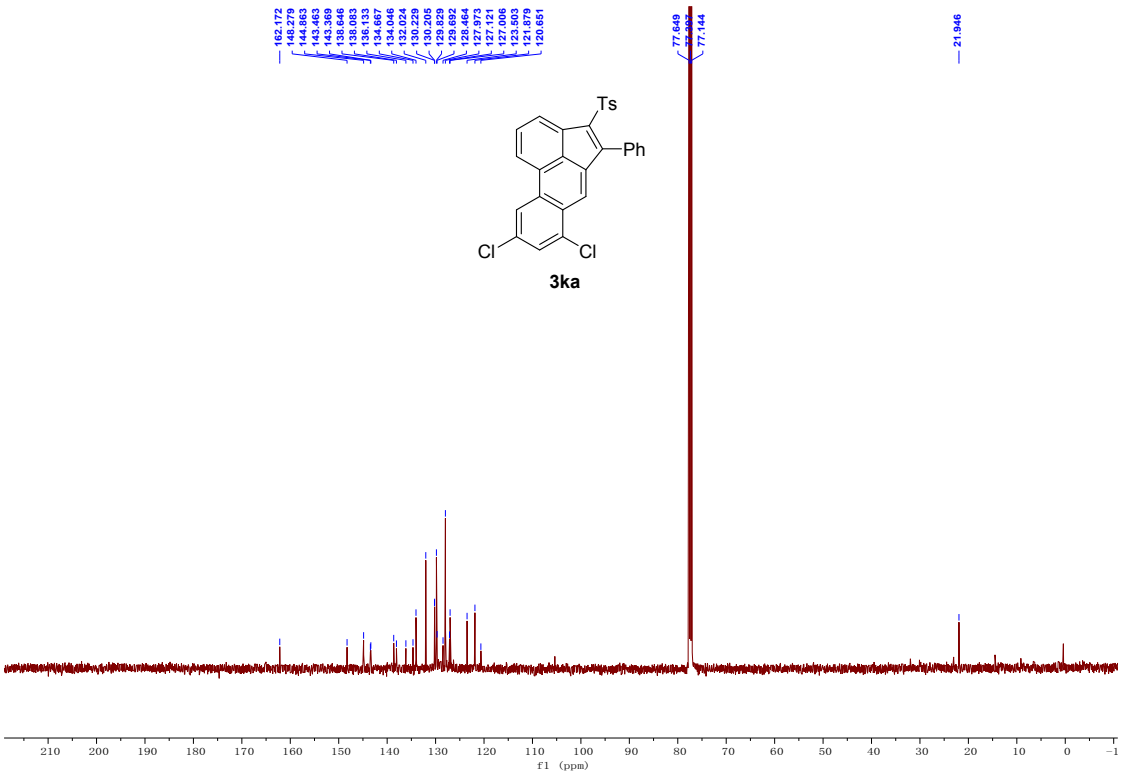
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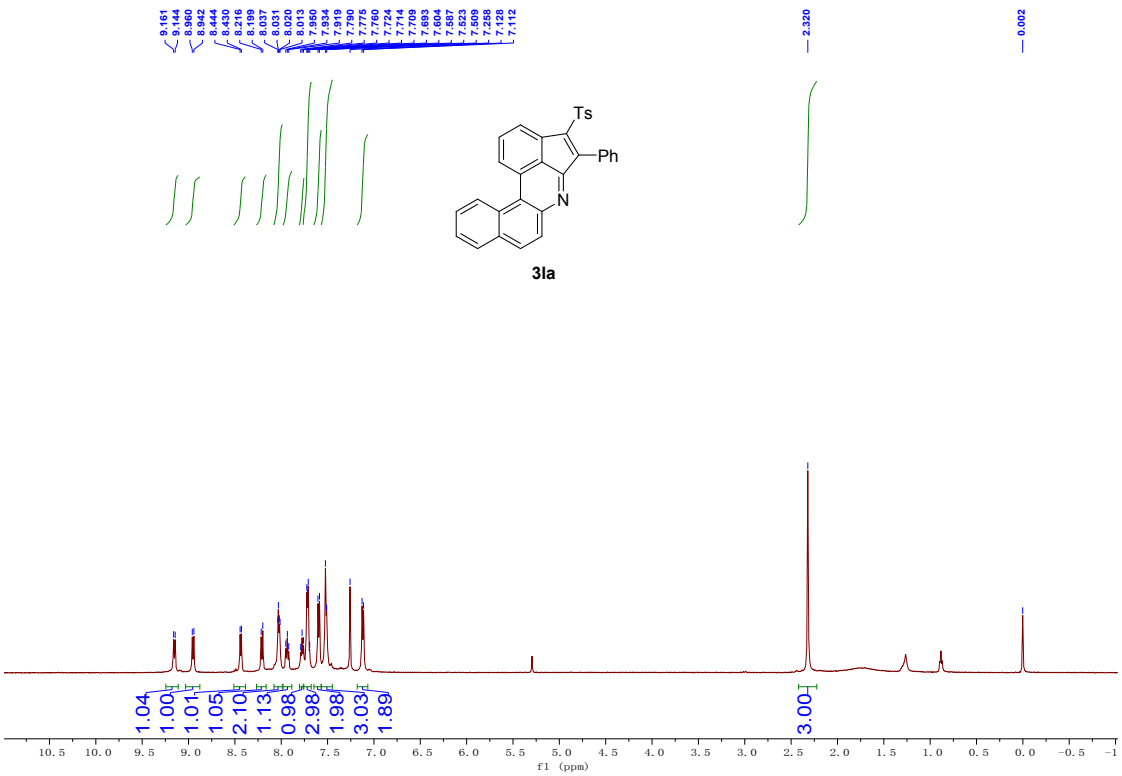
Ts
Ph
Cl
Cl
3ka
f1 (ppm)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8.037
8.031
8.020
8.013
7.950
7.934
7.916
7.790
7.775
7.760
7.724
7.714
7.709
7.693
7.604
7.587
7.521
7.509
7.258
7.128
7.112
2.320
0.002
Ts
Ph
N
3la
1.04
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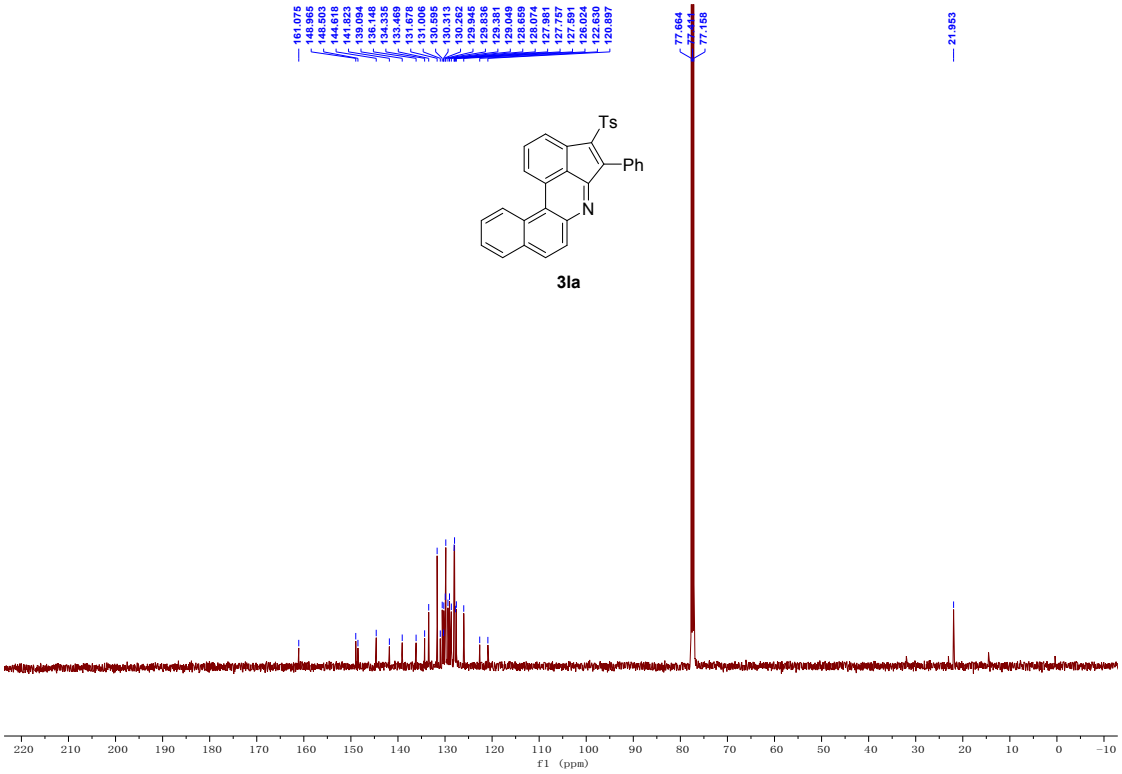
1.01
1.05
2.10
1.13
0.98
2.98
1.98
3.03
1.89
3.00
f1 (ppm)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130.262
129.945
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129.049
128.659
128.074
127.981
127.757
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120.897
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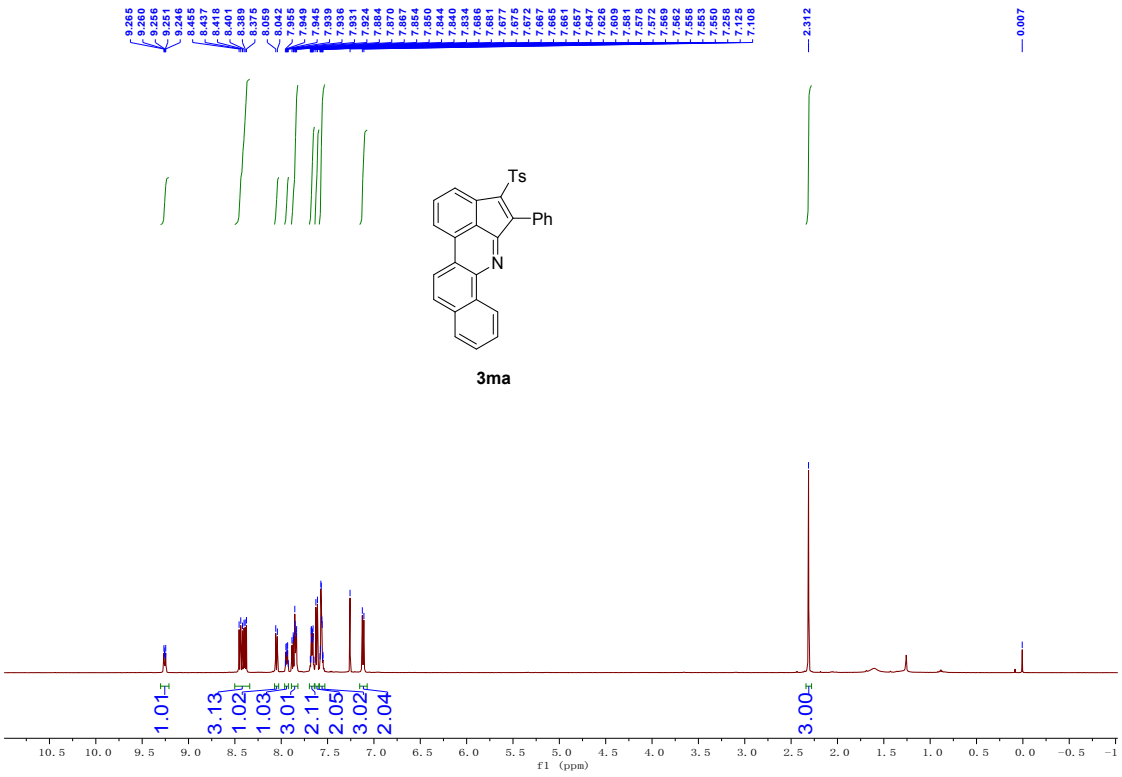
77.444
77.158
21.953
Ts
Ph
N
3la
f1 (ppm)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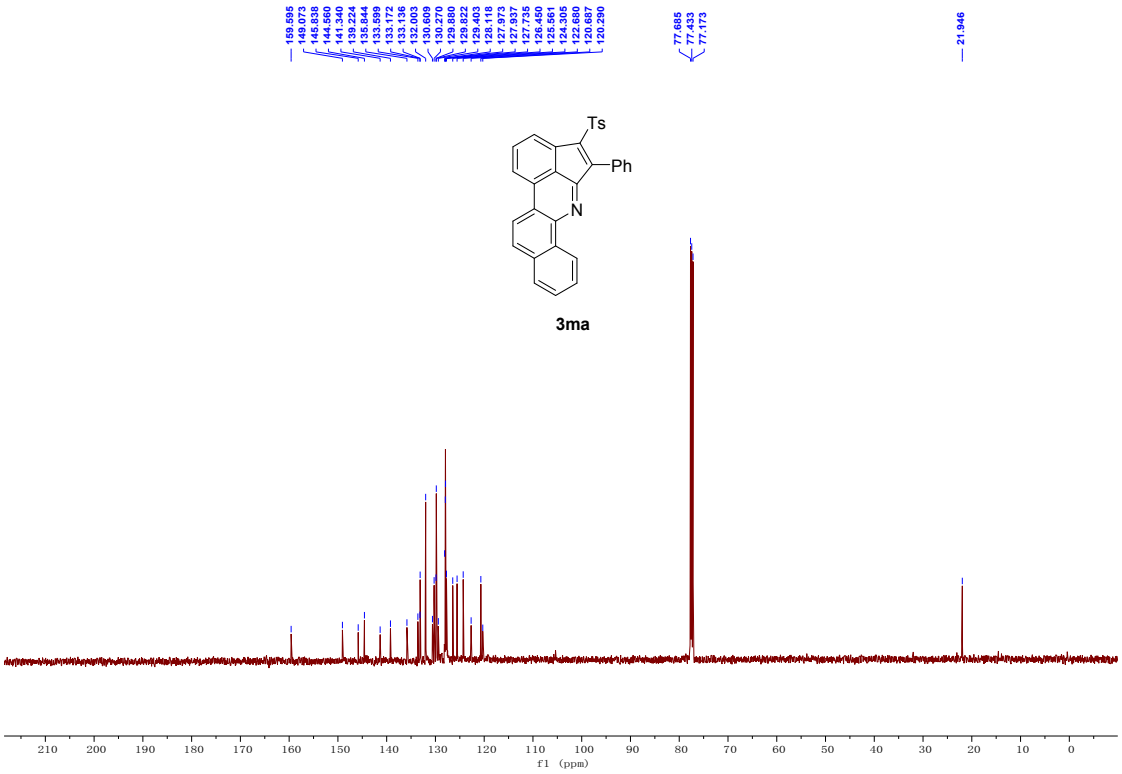
8.418
8.401
8.389
8.375
8.059
8.042
7.955
7.949
7.945
7.939
7.936
7.931
7.924
7.884
7.870
7.867
7.854
7.850
7.844
7.840
7.834
7.686
7.681
7.677
7.675
7.672
7.667
7.665
7.661
7.657
7.647
7.626
7.609
7.581
7.578
7.572
7.569
7.562
7.558
7.555
7.550
7.258
7.125
7.108
2.312
0.007
Ts
Ph
N
3ma
1.01
3.13
1.02
1.03
3.01
2.11
2.05
3.02
2.04
3.00
f1 (ppm)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132.003
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129.880
129.372
129.403
128.118
127.973
127.937
127.735
126.450
125.561
124.305
122.660
120.687
120.290
77.685
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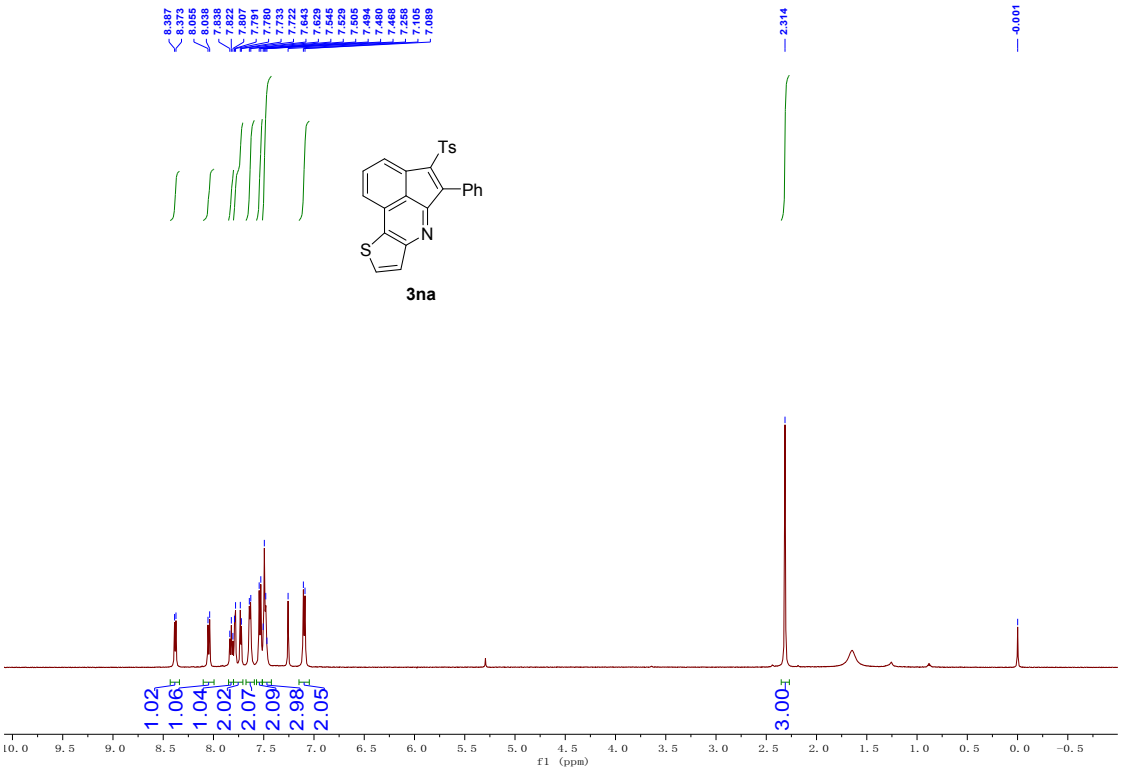
77.173
21.946
Ts
Ph
N
3ma
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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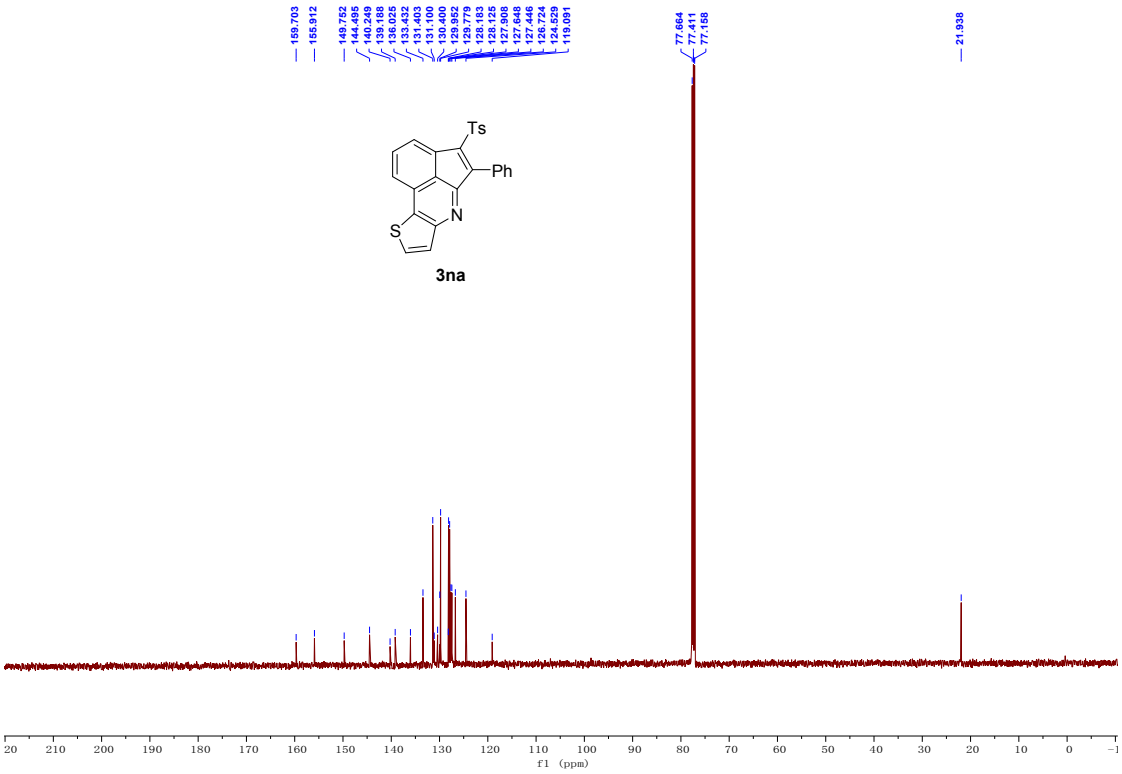
7.629
7.545
7.529
7.505
7.494
7.468
7.458
7.258
7.105
7.089
2.314
-0.001
Ts
Ph
N
S
3na
1.02
1.06
1.04
2.02
2.07
2.09
2.98
2.05
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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133.432
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129.952
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128.125
127.905
127.648
127.446
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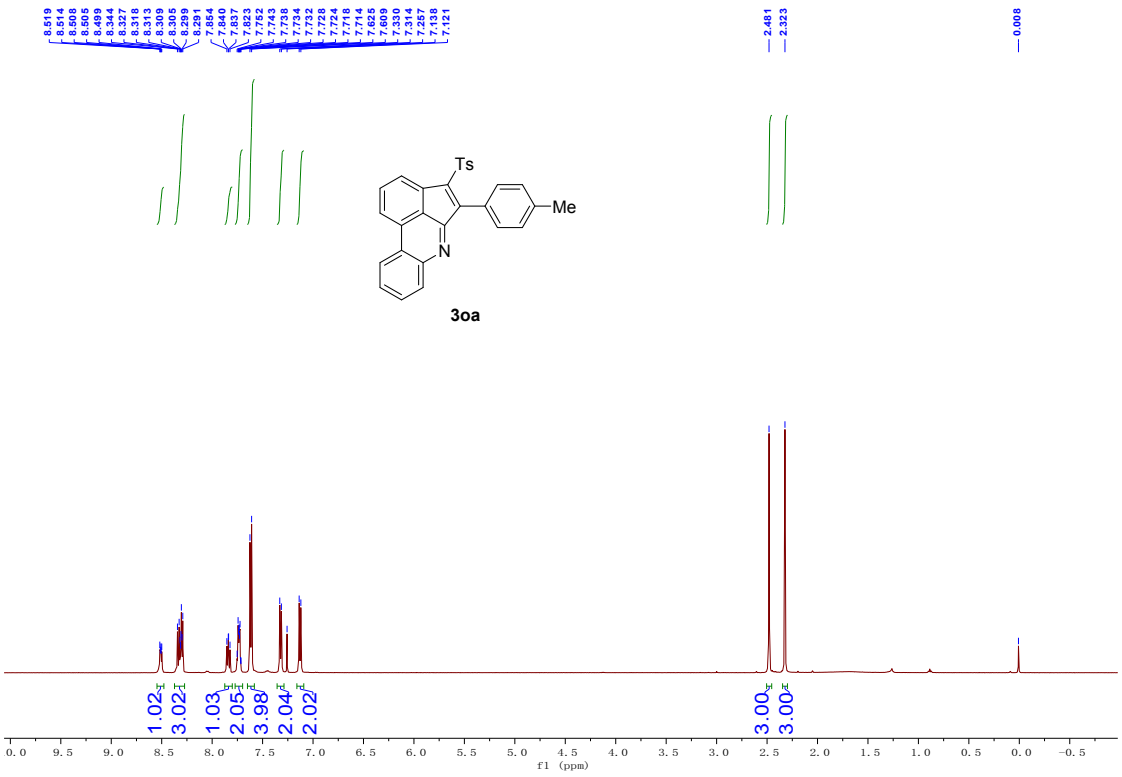
21.938
Ts
Ph
N
S
3na
f1 (ppm)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8.309
8.305
8.299
8.291
7.854
7.840
7.837
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7.743
7.738
7.734
7.732
7.728
7.724
7.718
7.714
7.625
7.609
7.330
7.314
7.297
7.138
7.121
2.481
2.323
0.008
Ts
Me
N
3oa
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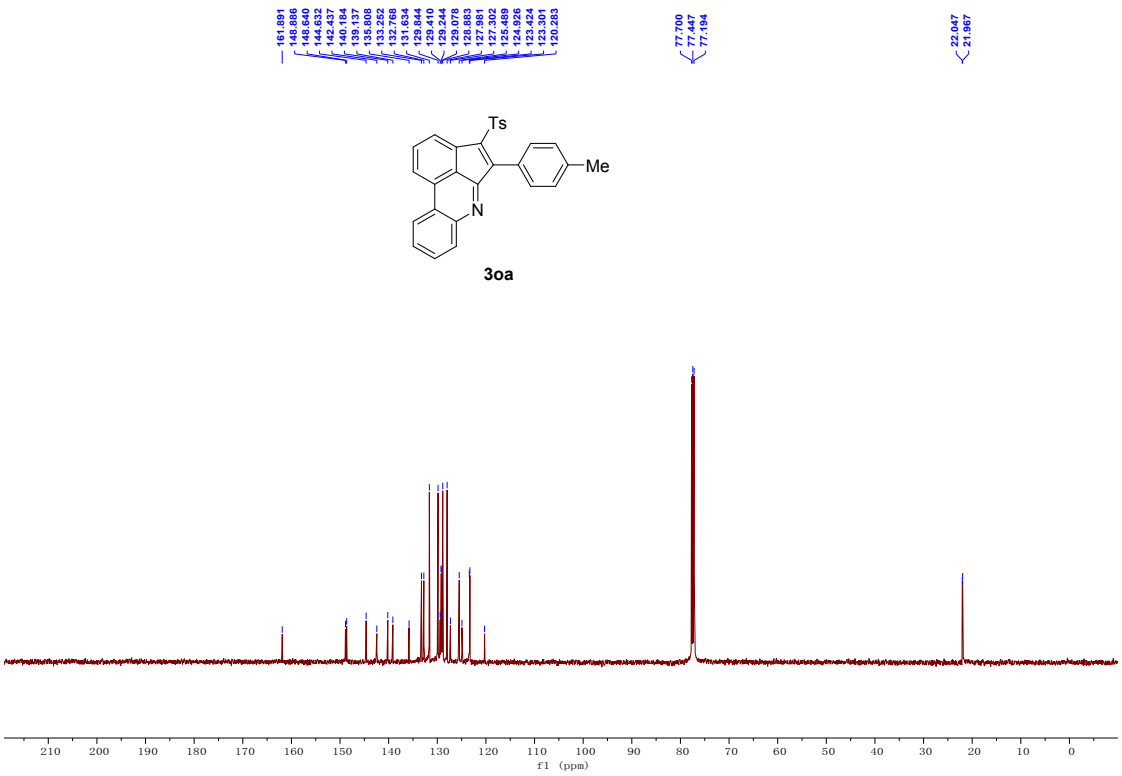
3.02
1.03
2.05
3.98
2.04
2.02
3.00
3.00
f1 (ppm)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135.808
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132.768
131.634
129.844
129.410
129.244
129.078
128.883
127.961
127.702
125.489
124.926
123.424
123.301
120.283
77.700
77.447
77.194
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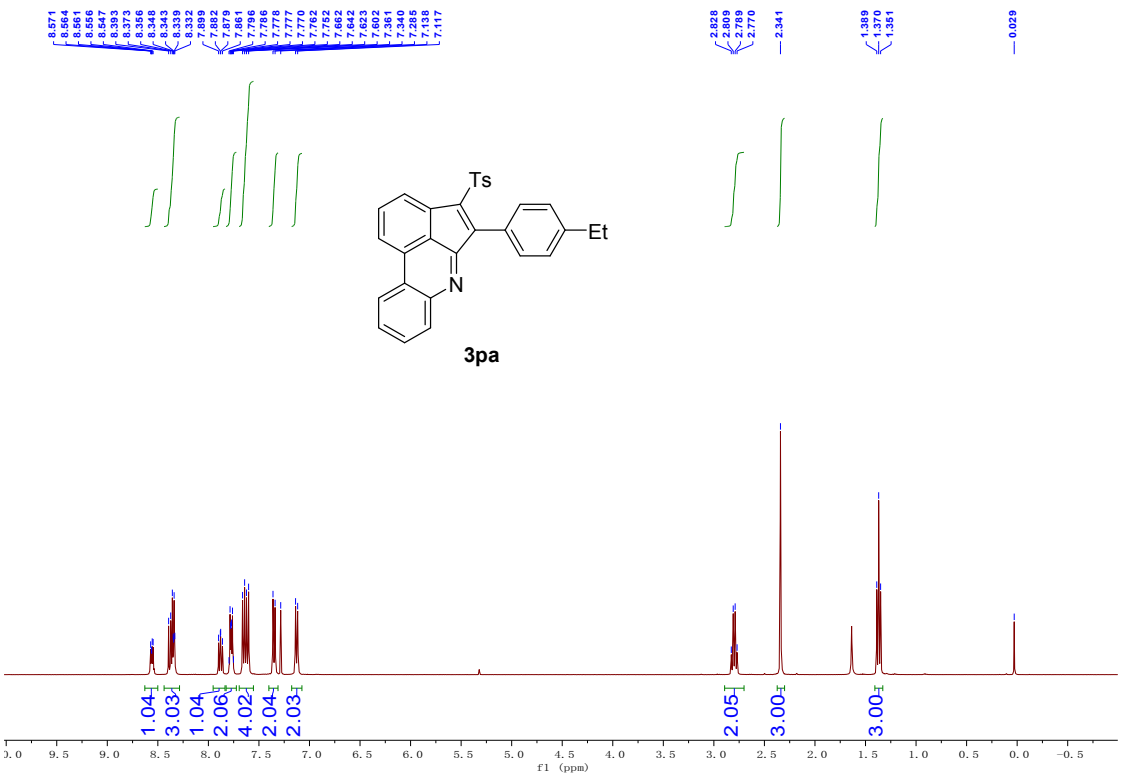
21.967
Ts
Me
N
3oa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.777
7.770
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7.752
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7.642
7.623
7.602
7.361
7.340
7.285
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2.809
2.789
2.770
2.341
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1.370
1.351
0.029
Ts
Et
N
3pa
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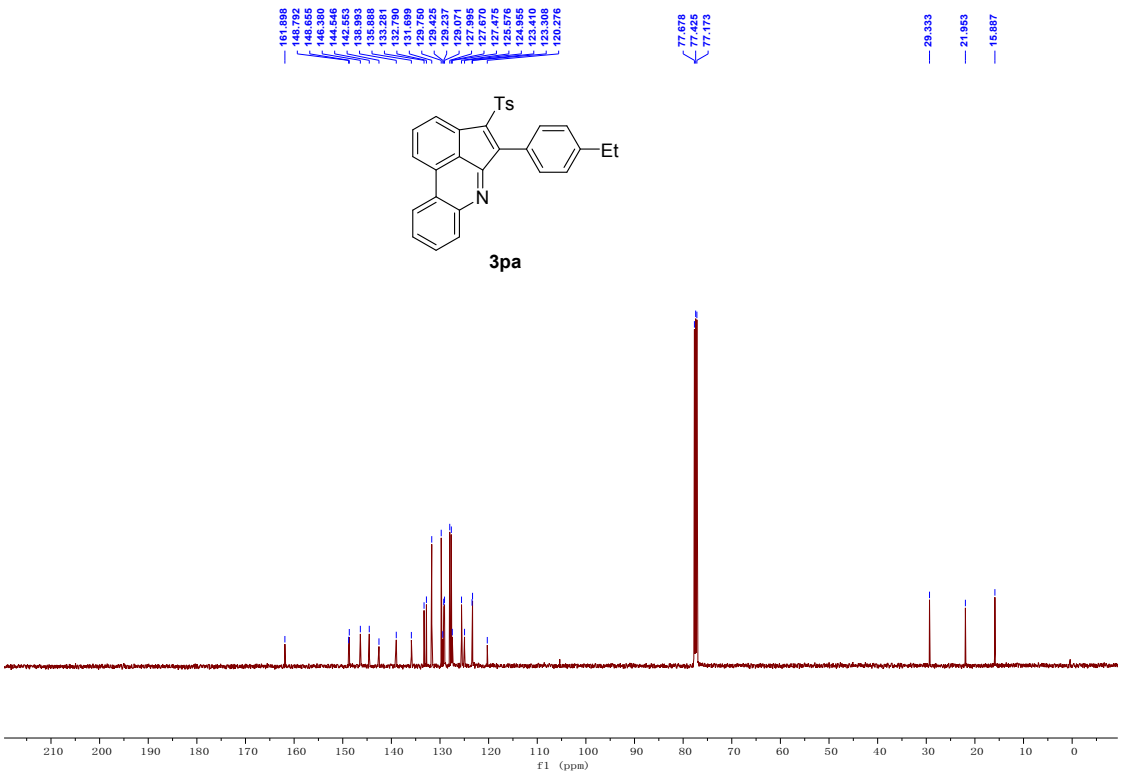
3.03
1.04
2.06
4.02
2.04
2.03
2.05
3.00
3.00
f1 (ppm)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129.425
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129.071
127.995
127.670
127.475
126.576
124.965
123.410
123.308
120.276
77.678
77.425
77.173
29.333
21.953
15.887
Ts
Et
N
3pa
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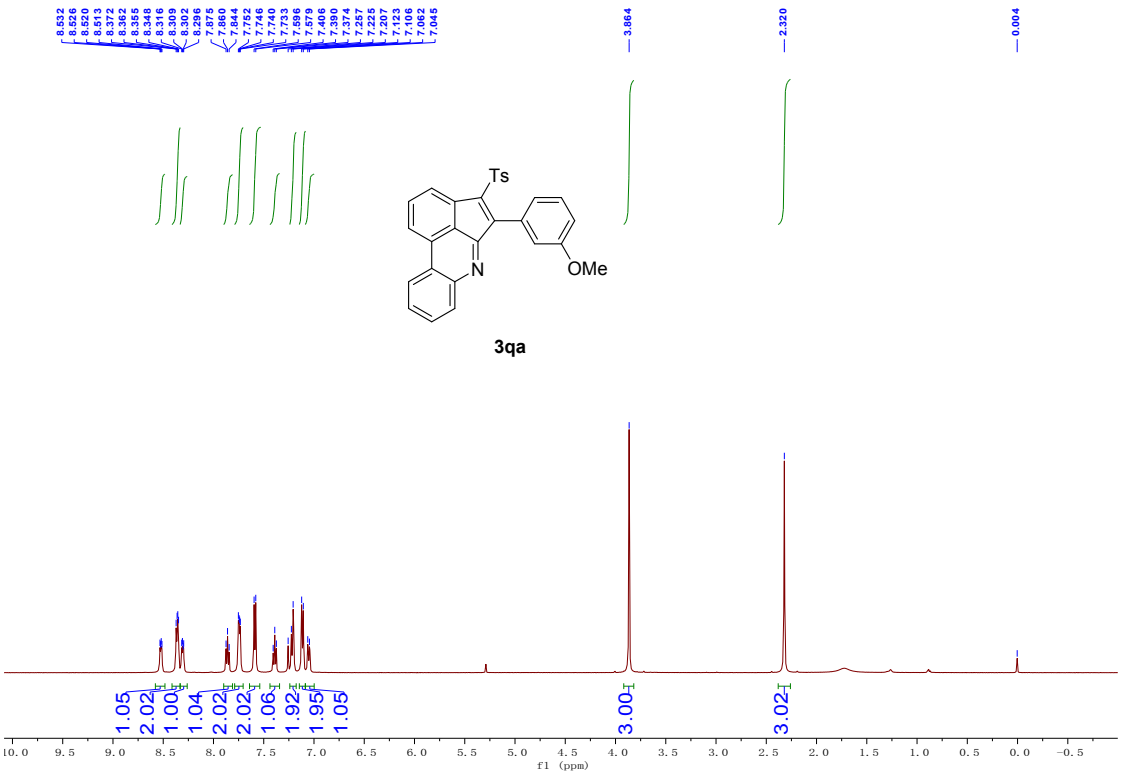
f1 (ppm)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8.309
8.302
8.296
7.875
7.860
7.844
7.752
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7.740
7.733
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7.679
7.406
7.390
7.374
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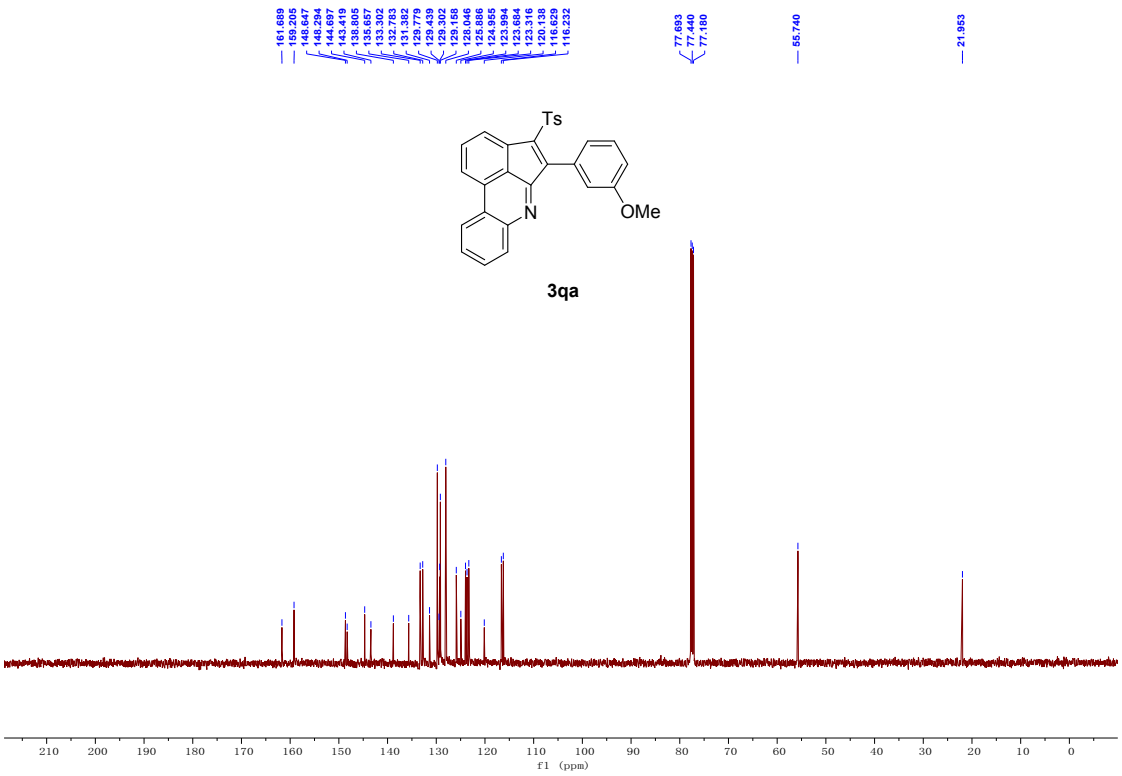
7.225
7.207
7.123
7.106
7.062
7.045
3.864
2.320
0.004
Ts
N
OMe
3qa
1.05
2.02
1.00
1.04
2.02
2.02
1.06
1.92
1.95
1.05
3.00
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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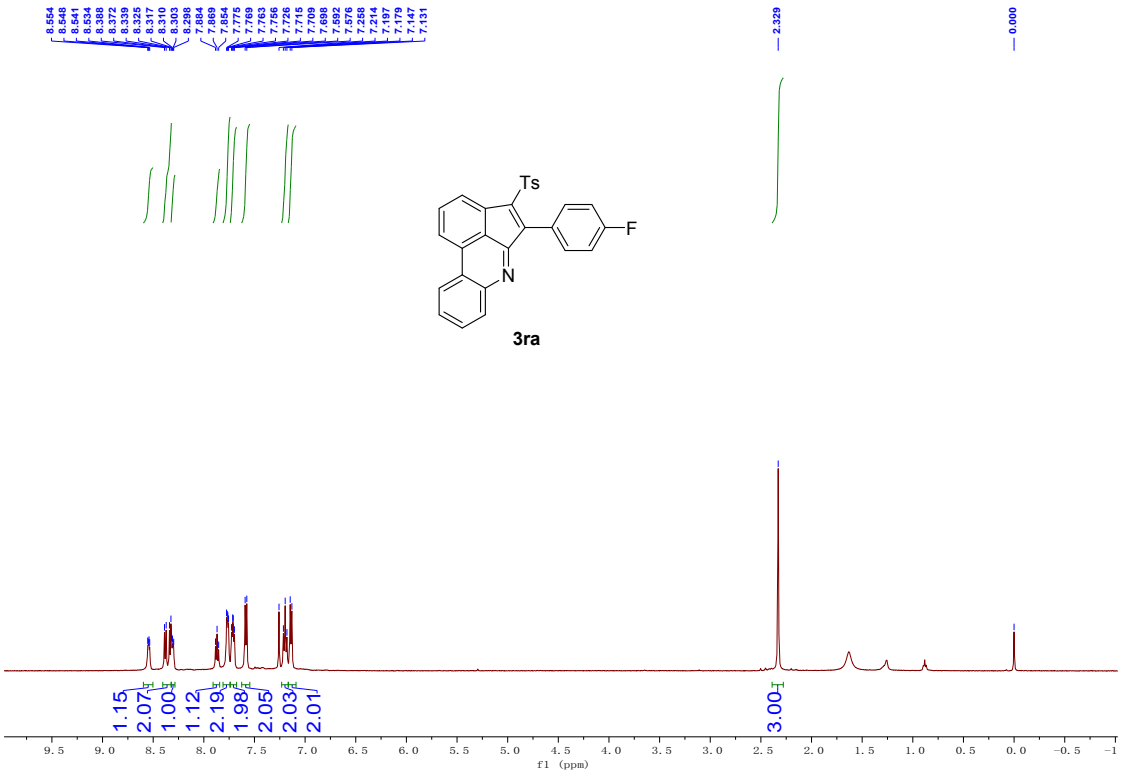
129.158
128.046
125.886
124.565
123.994
123.684
123.316
120.138
116.629
116.232
77.693
77.440
77.180
55.740
21.953
Ts
N
OMe
3qa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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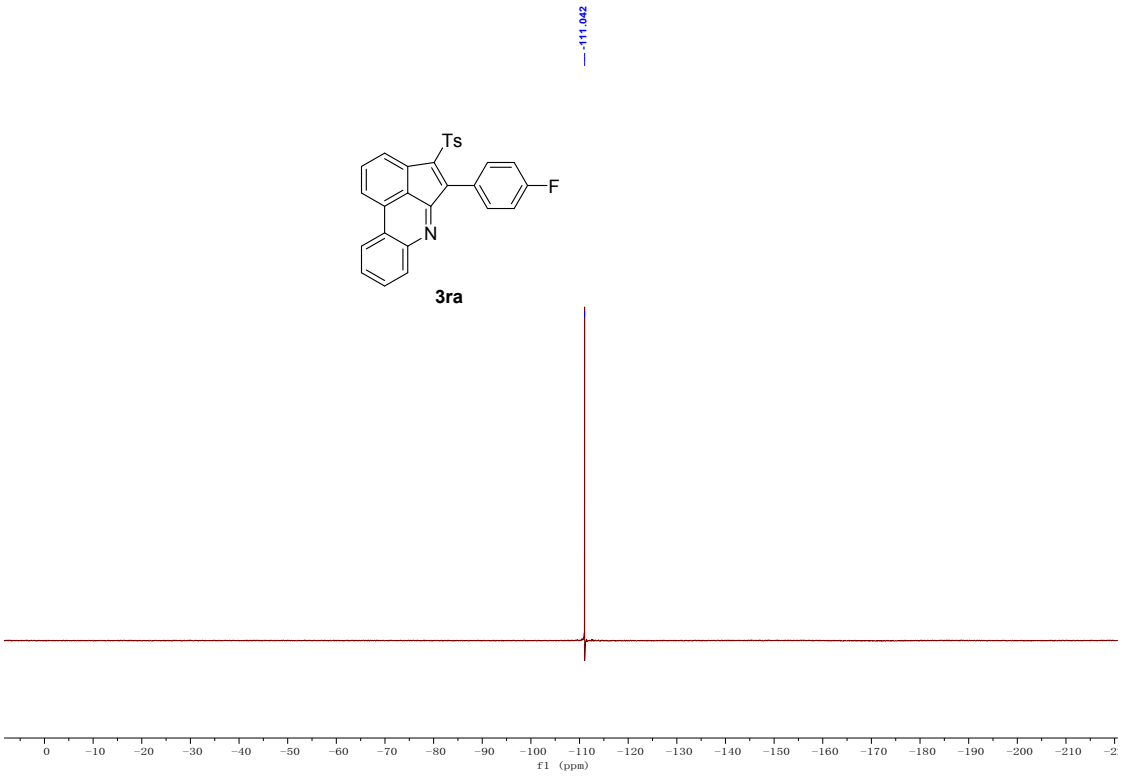
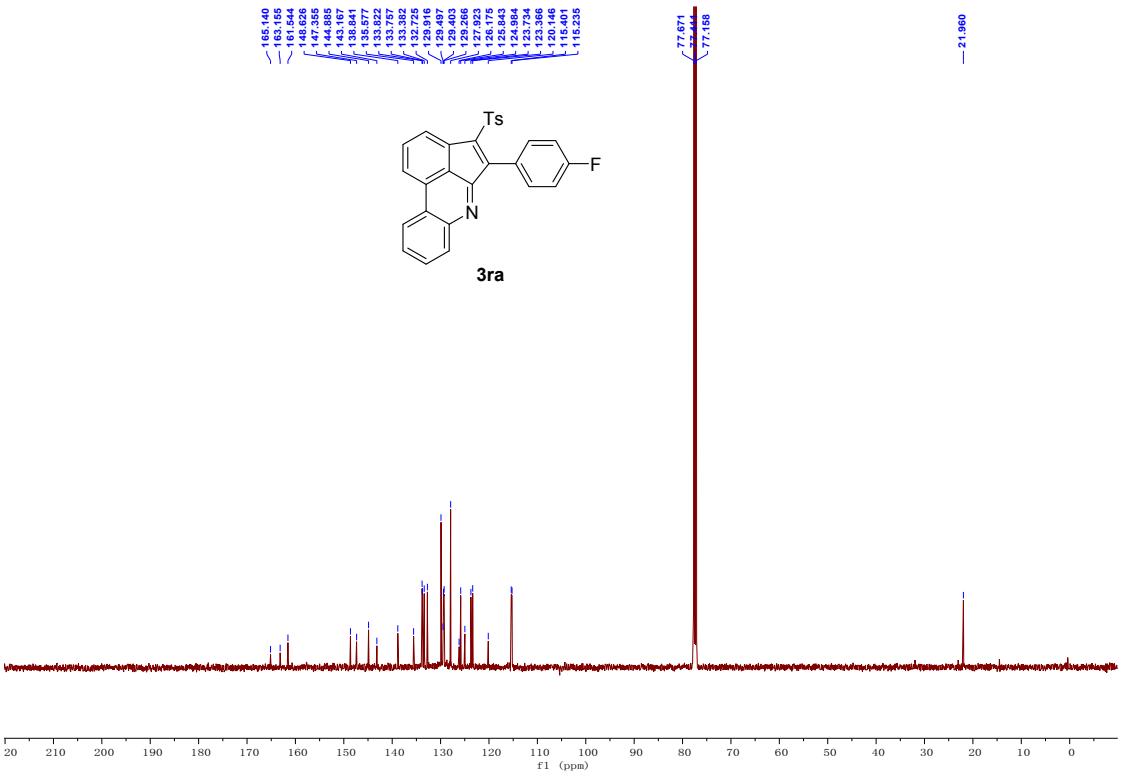
7.775
7.769
7.763
7.756
7.726
7.715
7.709
7.698
7.692
7.676
7.258
7.214
7.197
7.179
7.147
7.131
2.329
0.000
Ts
N
F
3ra
1.15
2.07
1.00
1.12
2.19
1.98
2.05
2.03
2.01
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


-111.042
Ts
F
N
3ra
0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210 −2
f1 (ppm)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120.146
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116.235
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Ts
F
N
3ra
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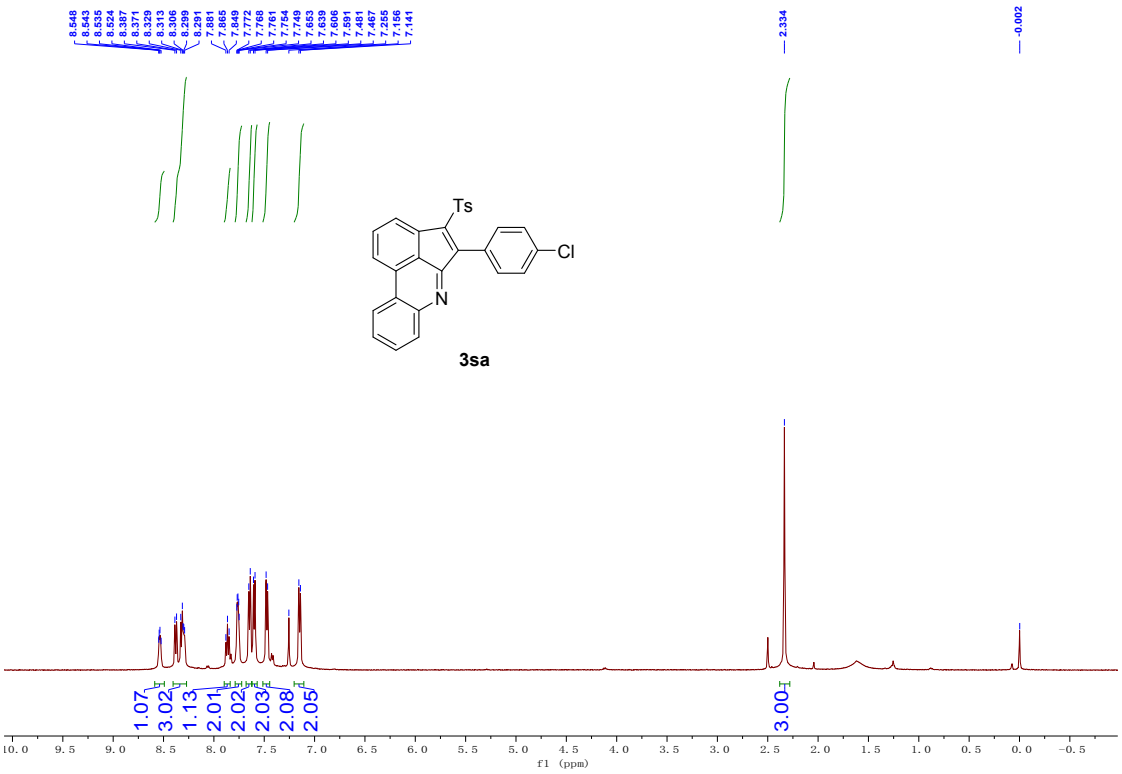
f1 (ppm)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7.849
7.772
7.768
7.761
7.754
7.749
7.655
7.639
7.606
7.591
7.481
7.467
7.255
7.166
7.141
2.334
-0.002
Ts
Cl
N
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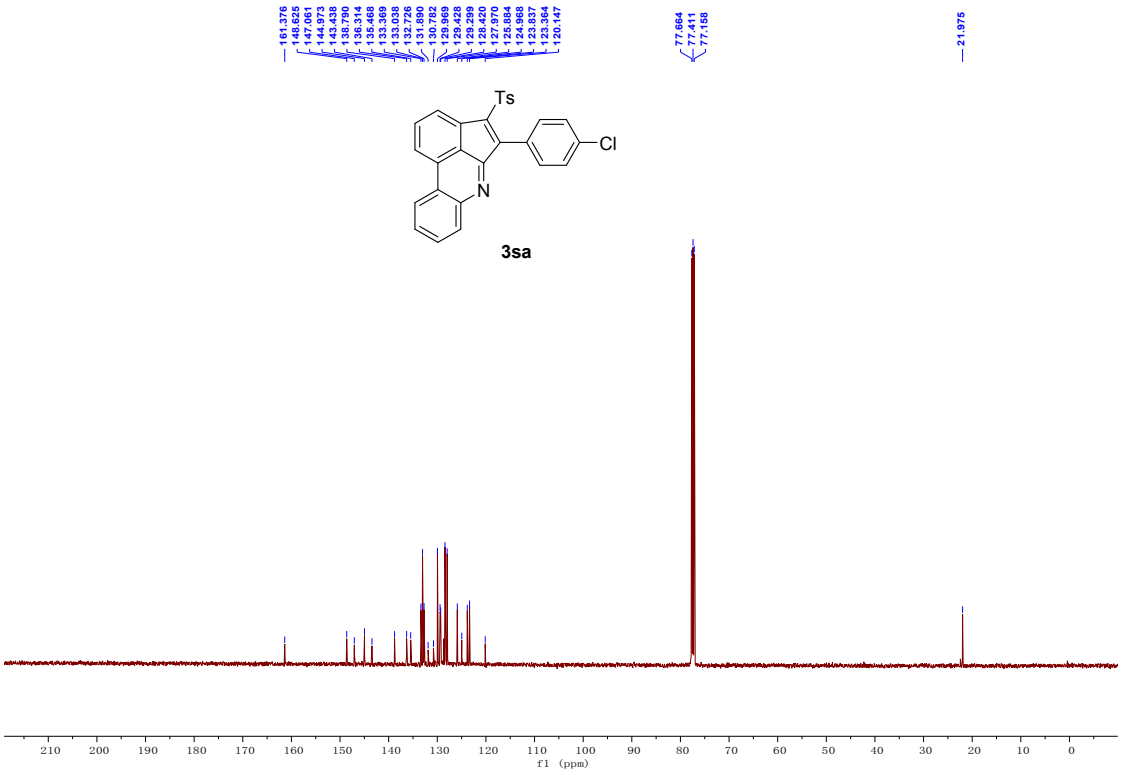
3.02
1.13
2.01
2.02
2.03
2.08
2.05
3.00
f1 (ppm)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130.782
129.969
129.428
129.299
128.420
127.970
126.864
124.998
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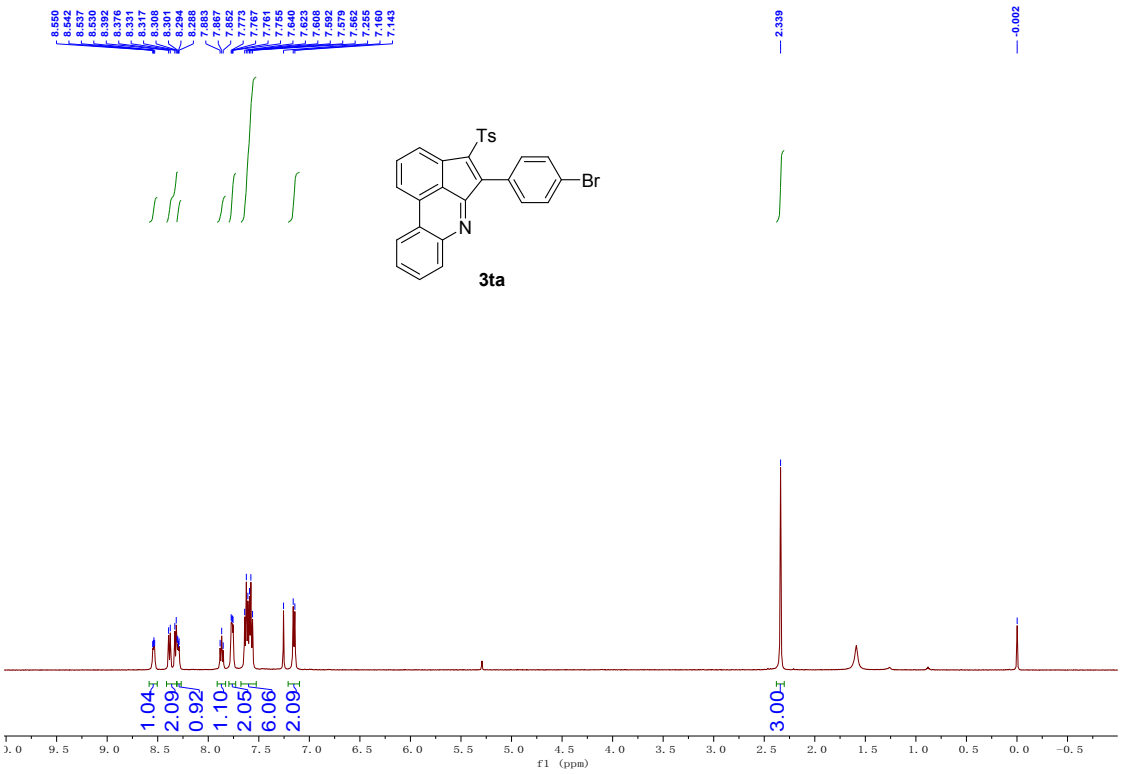
120.147
77.664
77.411
77.158
21.975
Ts
Cl
N
3sa
f1 (ppm)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7.608
7.592
7.579
7.562
7.555
7.160
7.143
2.339
-0.002
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Br
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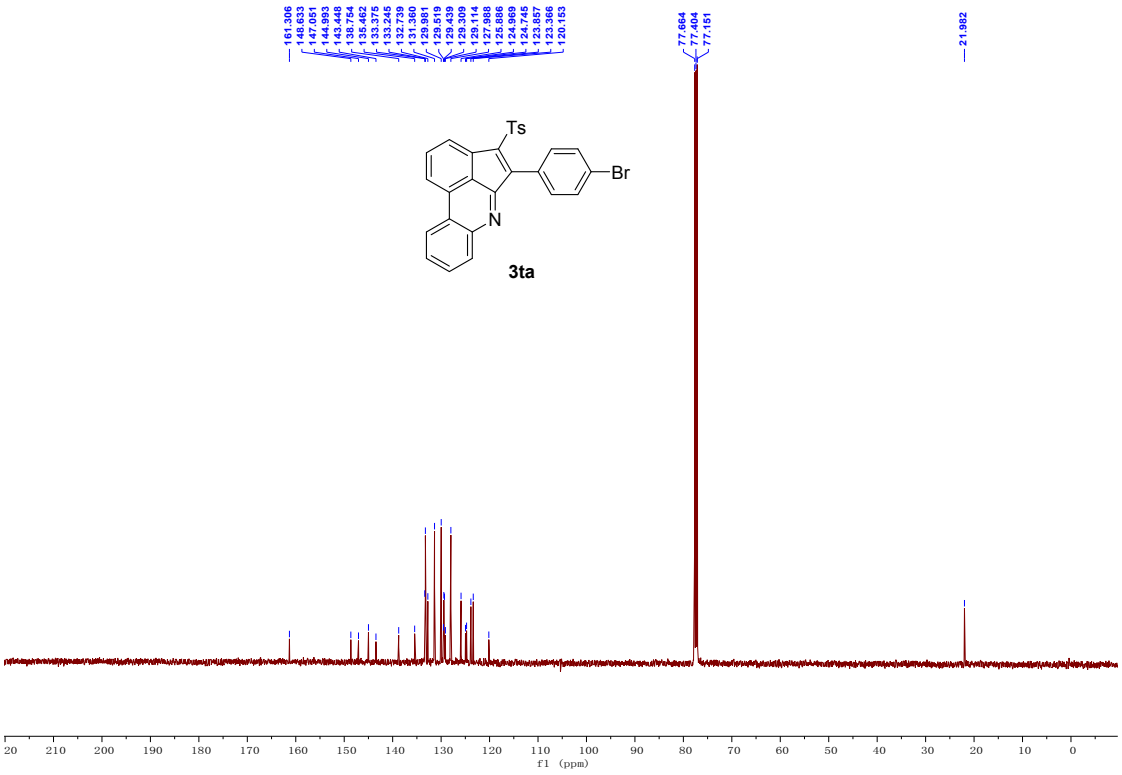
2.05
6.06
2.09
3.00
f1 (ppm)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132.739
131.360
129.981
129.519
129.430
129.309
129.114
127.988
125.886
124.969
124.745
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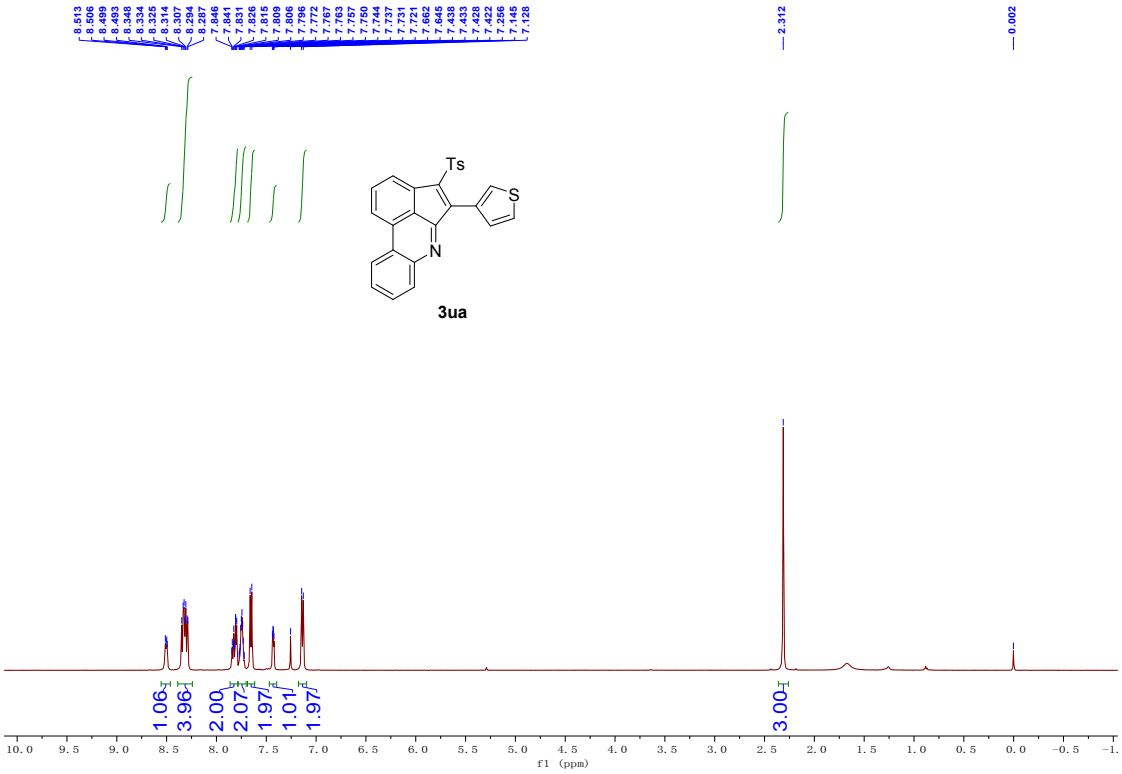
77.404
77.151
21.982
Ts
Br
N
3ta
f1 (ppm)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7.831
7.826
7.815
7.809
7.806
7.796
7.772
7.767
7.763
7.757
7.750
7.744
7.737
7.731
7.721
7.662
7.645
7.438
7.433
7.423
7.422
7.256
7.145
7.128
2.312
0.002
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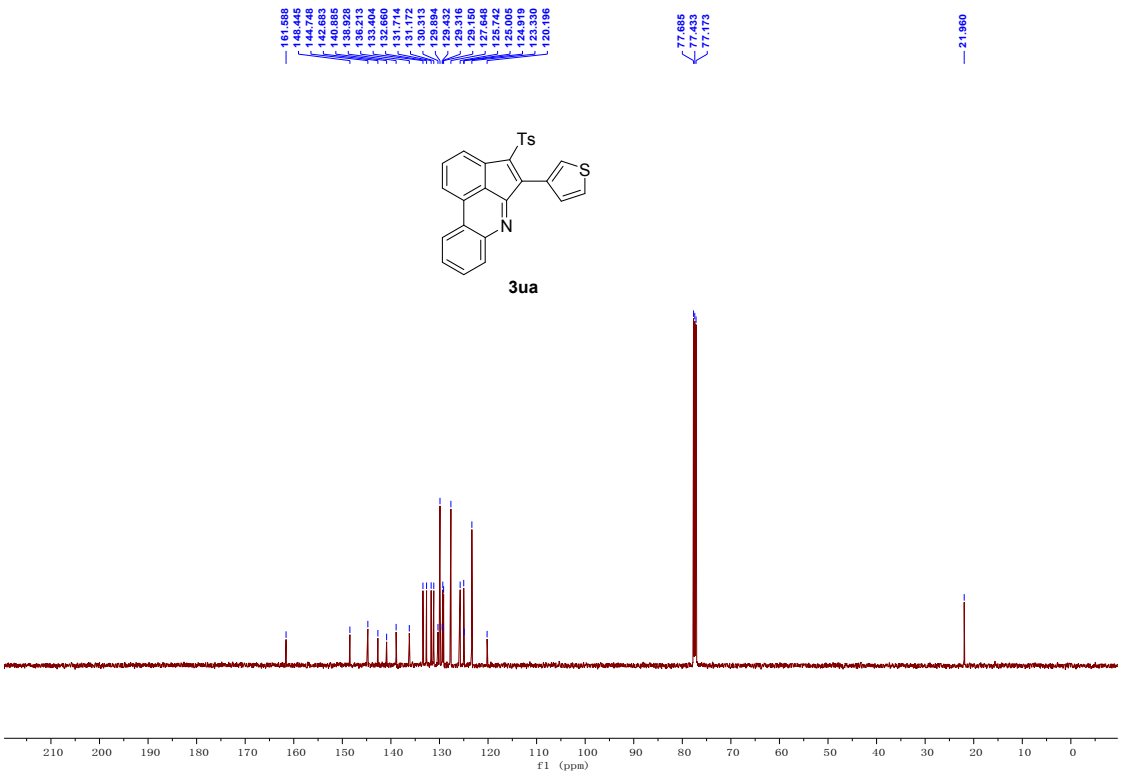
S
N
3ua
1.06
3.96
2.00
2.07
1.97
1.01
1.97
3.00
f1 (ppm)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130.353
129.894
129.432
129.316
129.150
127.648
125.742
125.005
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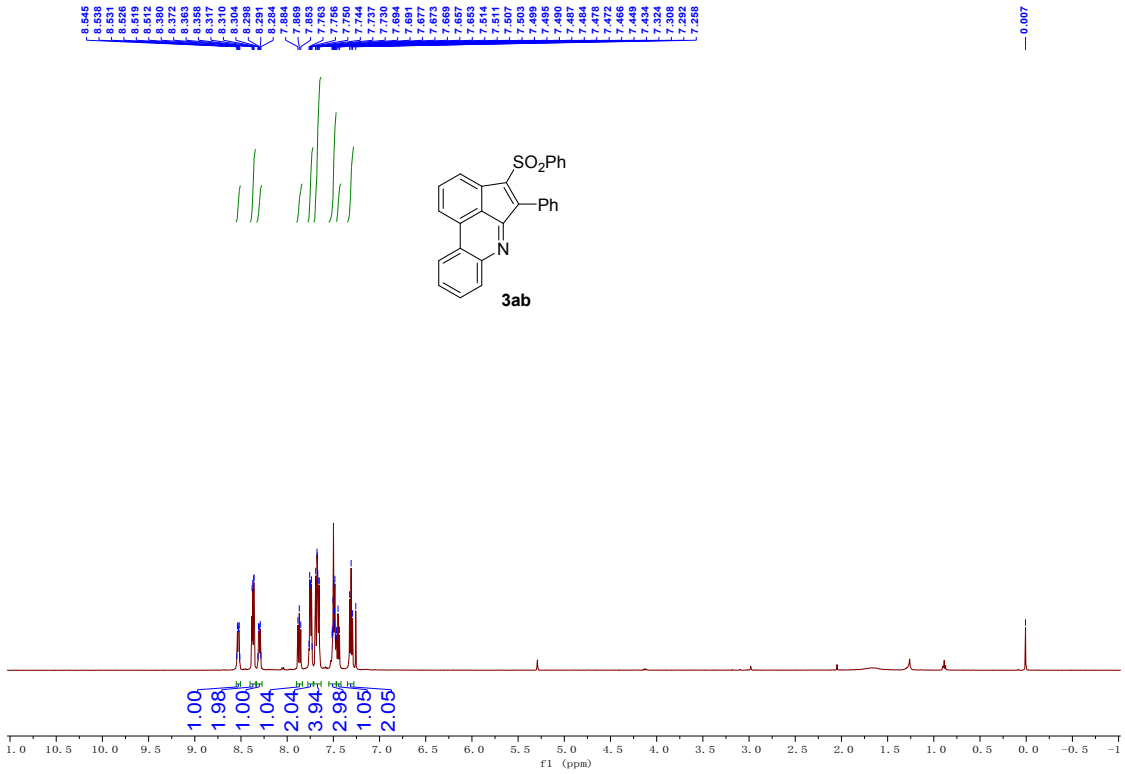
123.330
120.196
77.685
77.433
77.173
21.960
Ts
S
N
3ua
f1 (ppm)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8.372
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8.317
8.310
8.304
8.298
8.291
8.284
7.869
7.853
7.763
7.756
7.750
7.744
7.737
7.730
7.694
7.691
7.677
7.673
7.669
7.657
7.653
7.514
7.511
7.507
7.503
7.499
7.495
7.490
7.487
7.484
7.478
7.472
7.466
7.449
7.434
7.324
7.308
7.292
7.258
0.007
SO2Ph
Ph
N
3ab
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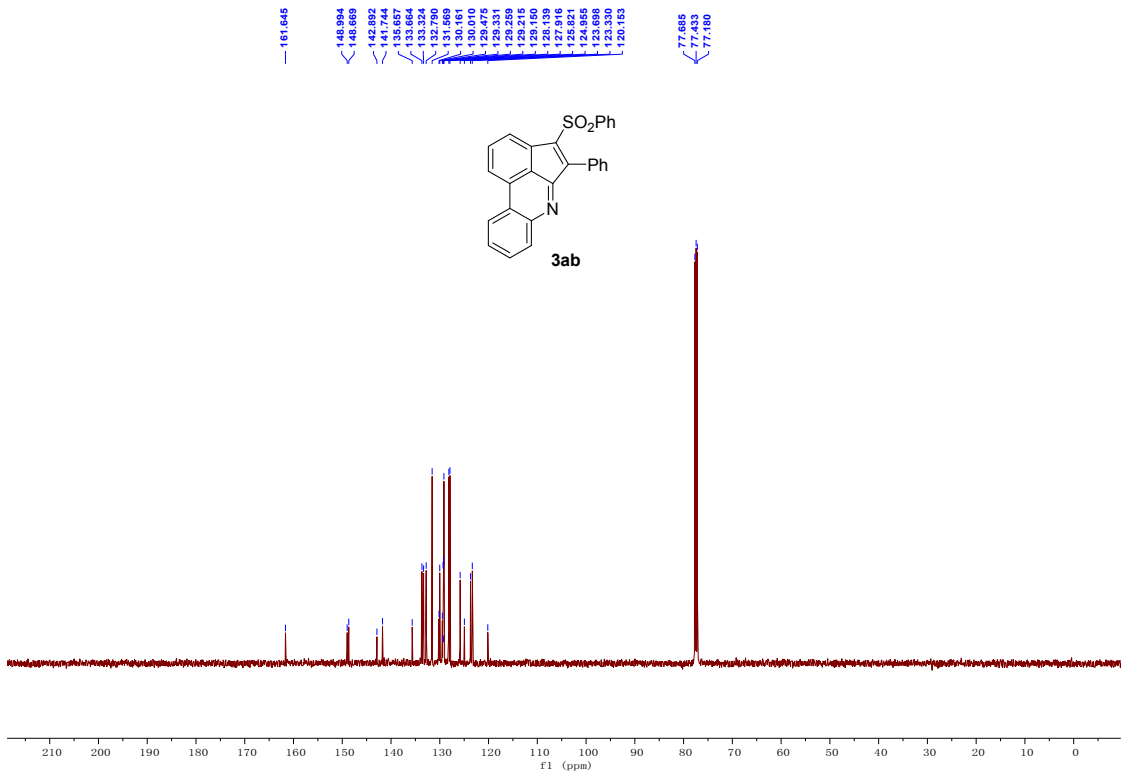
1.98
1.00
1.04
2.04
3.94
2.98
1.05
2.05
f1 (ppm)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129.475
129.331
129.209
129.215
129.150
128.139
127.916
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123.330
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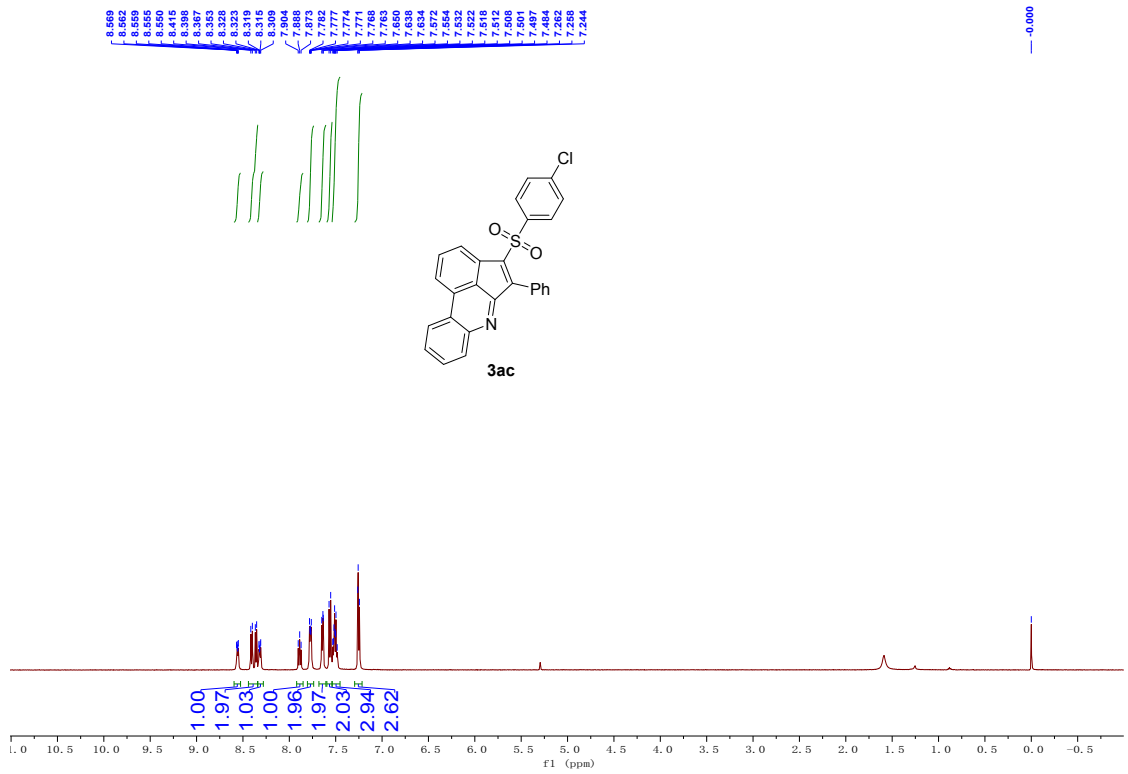
77.433
77.180
SO2Ph
Ph
N
3ab
f1 (ppm)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8.367
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8.323
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8.315
8.309
7.904
7.888
7.873
7.782
7.777
7.774
7.771
7.768
7.763
7.650
7.638
7.634
7.572
7.554
7.532
7.522
7.516
7.512
7.508
7.501
7.497
7.484
7.262
7.238
7.244
-0.000
Cl
O
S
O
Ph
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3ac
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1.03
1.00
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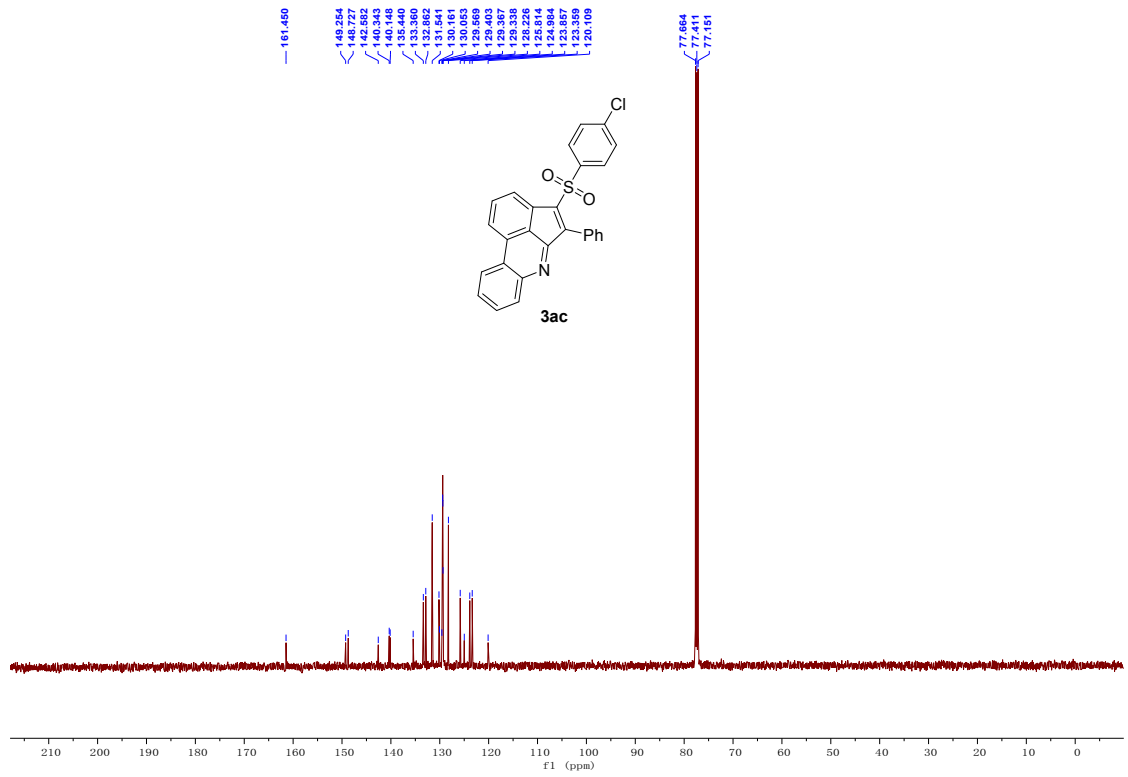
1.97
2.03
2.94
2.62
f1 (ppm)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130.161
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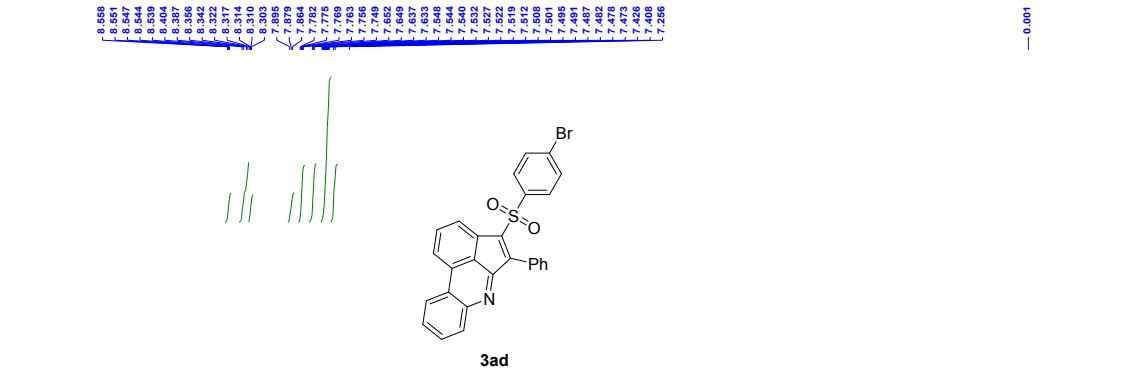
129.569
129.403
129.367
129.338
128.726
125.514
124.984
123.857
123.359
120.109
77.664
77.411
77.151
Cl
O
S
O
Ph
N
3ac
f1 (ppm)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8.317
8.314
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8.303
7.895
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7.864
7.782
7.775
7.769
7.763
7.756
7.749
7.652
7.649
7.637
7.633
7.548
7.544
7.540
7.532
7.527
7.522
7.519
7.512
7.508
7.501
7.495
7.491
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7.473
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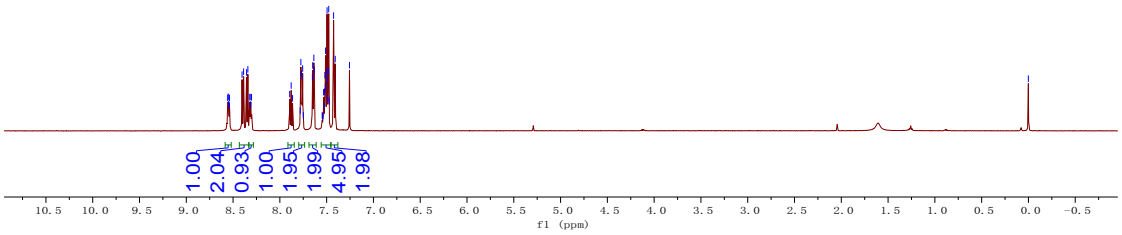
0.001
Br
O
S
O
Ph
N

3ad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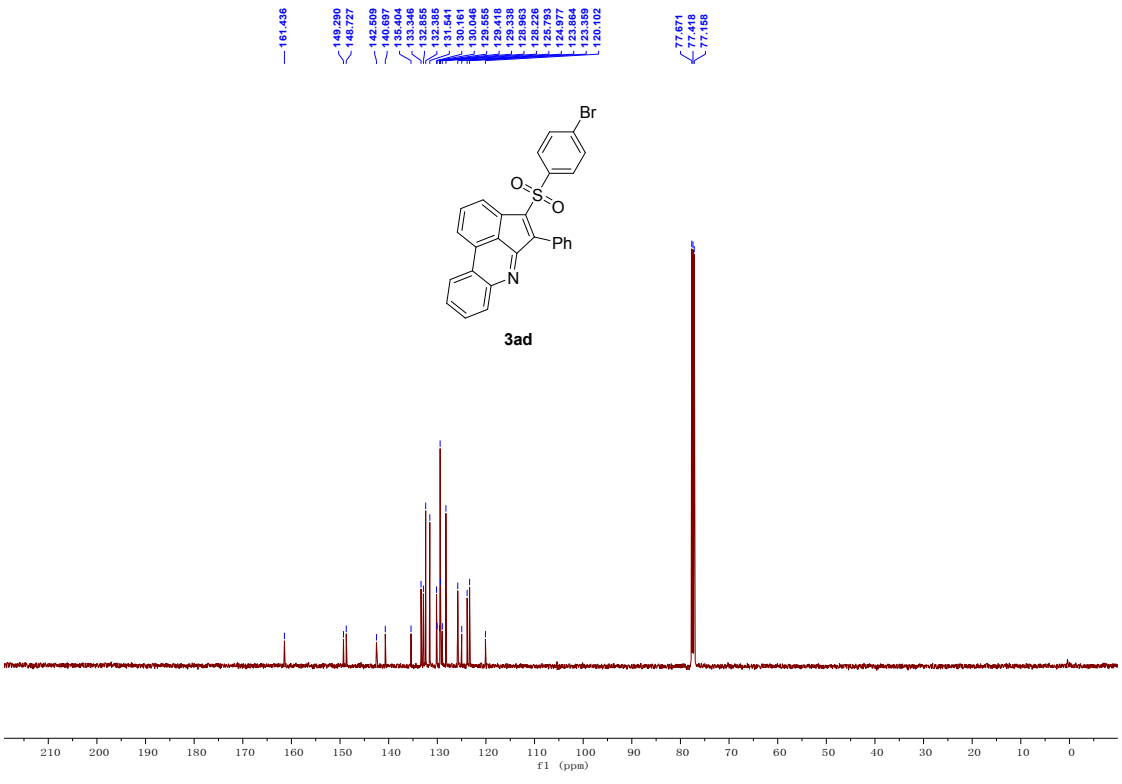
0.93
1.00
1.95
1.99
4.95
1.98
f1 (ppm)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129.418
129.338
128.963
128.226
125.793
124.977
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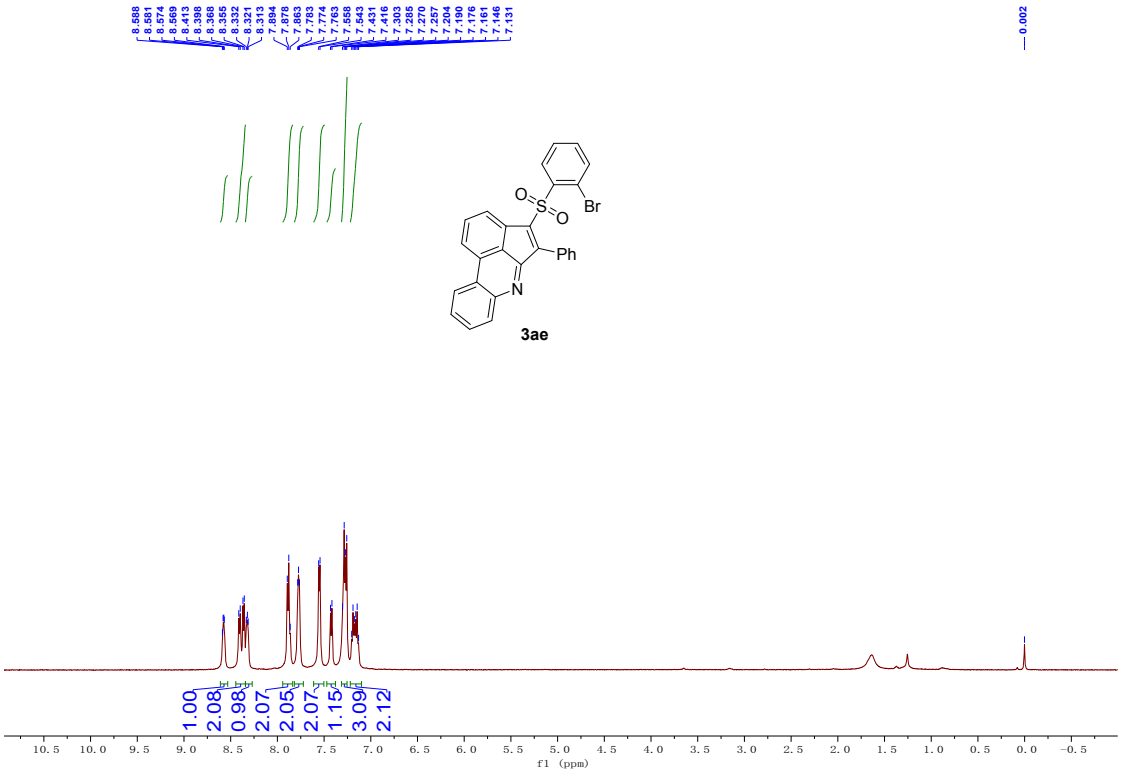
120.102
77.671
77.418
77.158
Br
O
S
O
Ph
N
f1 (ppm)

3ad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8.355
8.332
8.321
8.313
7.894
7.878
7.863
7.793
7.774
7.763
7.558
7.543
7.431
7.416
7.303
7.285
7.270
7.257
7.204
7.190
7.176
7.161
7.146
7.131
0.002
O
S
O
Br
Ph
N
3ae
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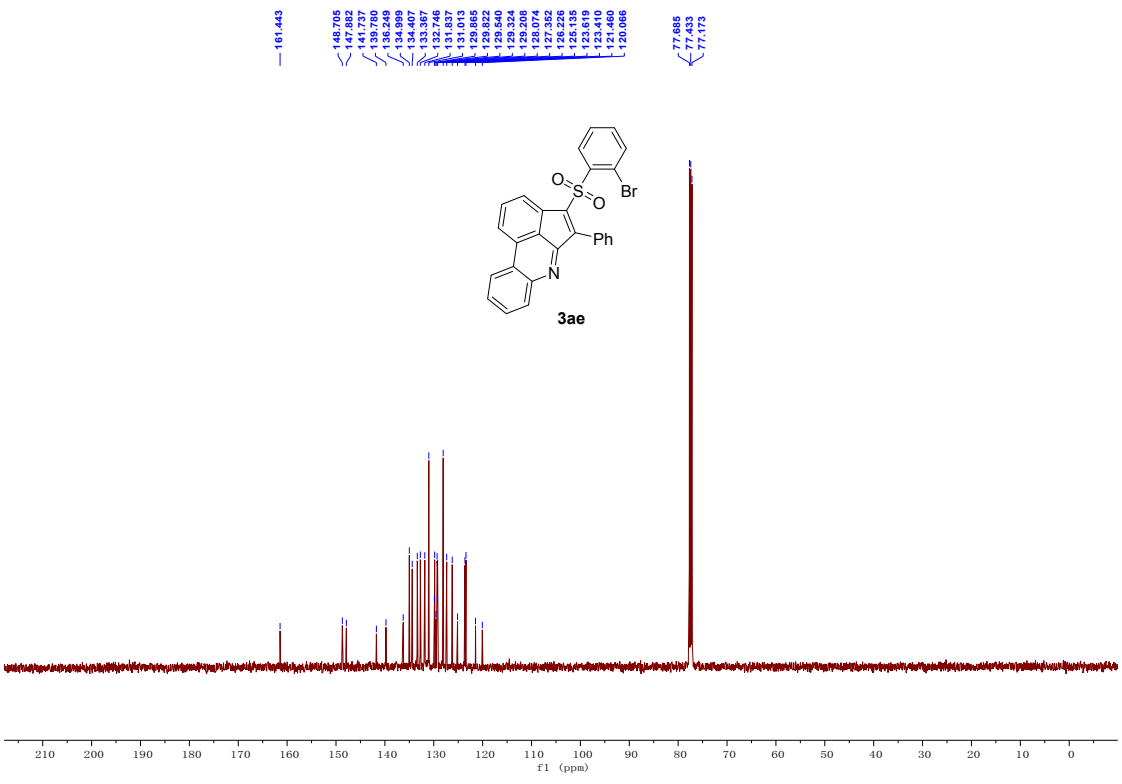
2.08
0.98
2.07
2.05
2.07
1.15
3.09
2.12
f1 (ppm)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134.999
134.407
133.367
133.246
131.837
131.013
129.865
129.822
129.540
129.324
129.208
128.074
127.252
126.226
125.135
123.619
123.410
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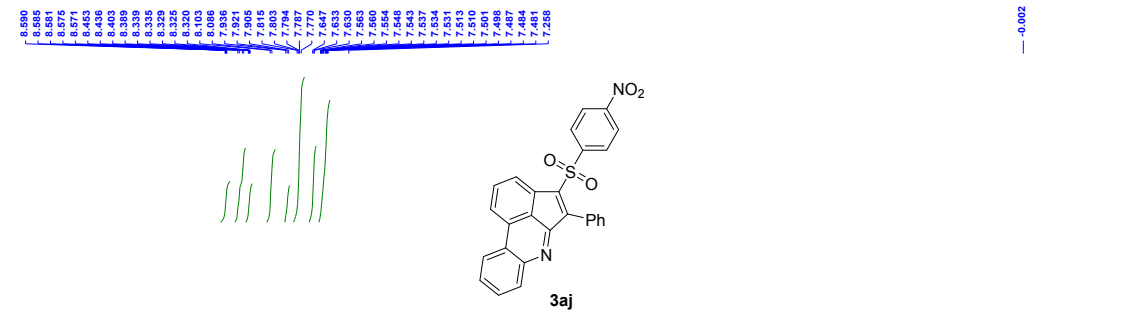
120.066
77.685
77.433
77.173
O
S
O
Br
Ph
N
3ae
f1 (ppm)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8.335
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8.325
8.320
8.103
8.086
7.936
7.921
7.905
7.815
7.803
7.794
7.787
7.770
7.647
7.633
7.630
7.563
7.560
7.554
7.548
7.543
7.537
7.534
7.531
7.513
7.510
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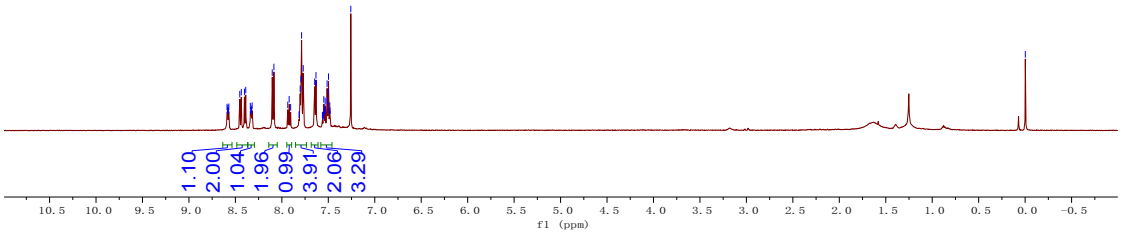
7.484
7.481
7.258
-0.002
NO2
O
S
O
Ph
N

3aj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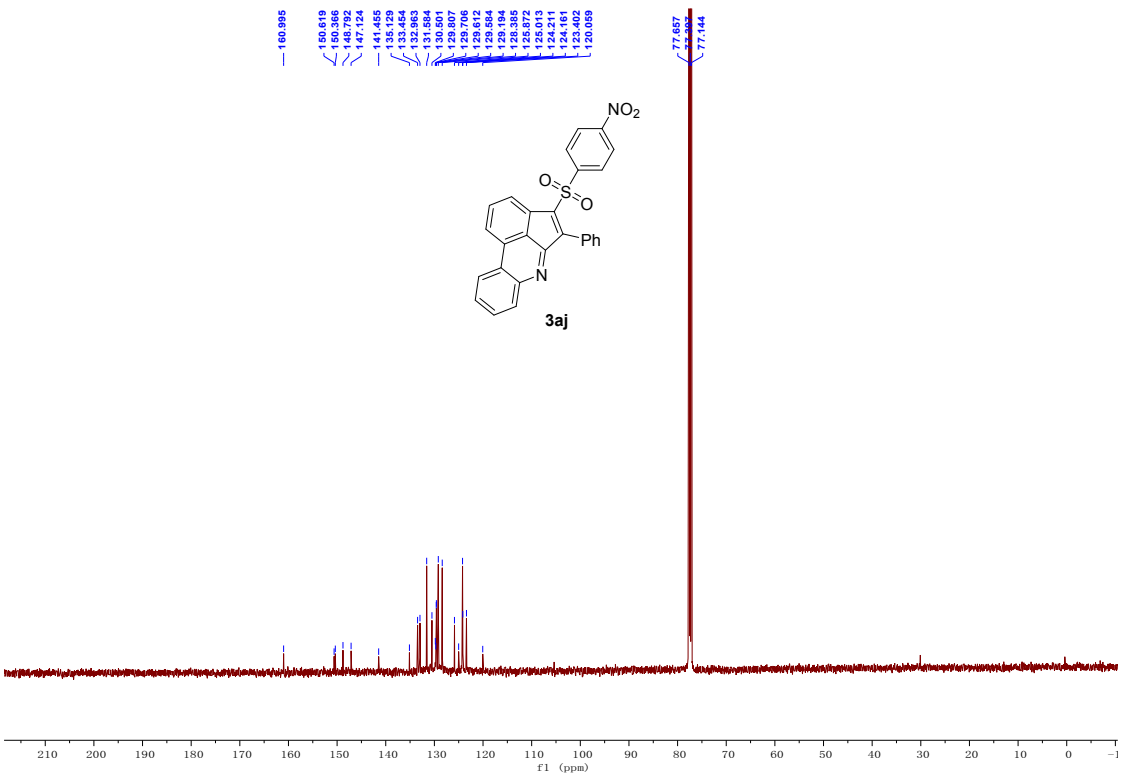
0.99
3.91
2.06
3.29
f1 (ppm)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129.613
129.554
129.194
128.385
125.872
125.013
124.211
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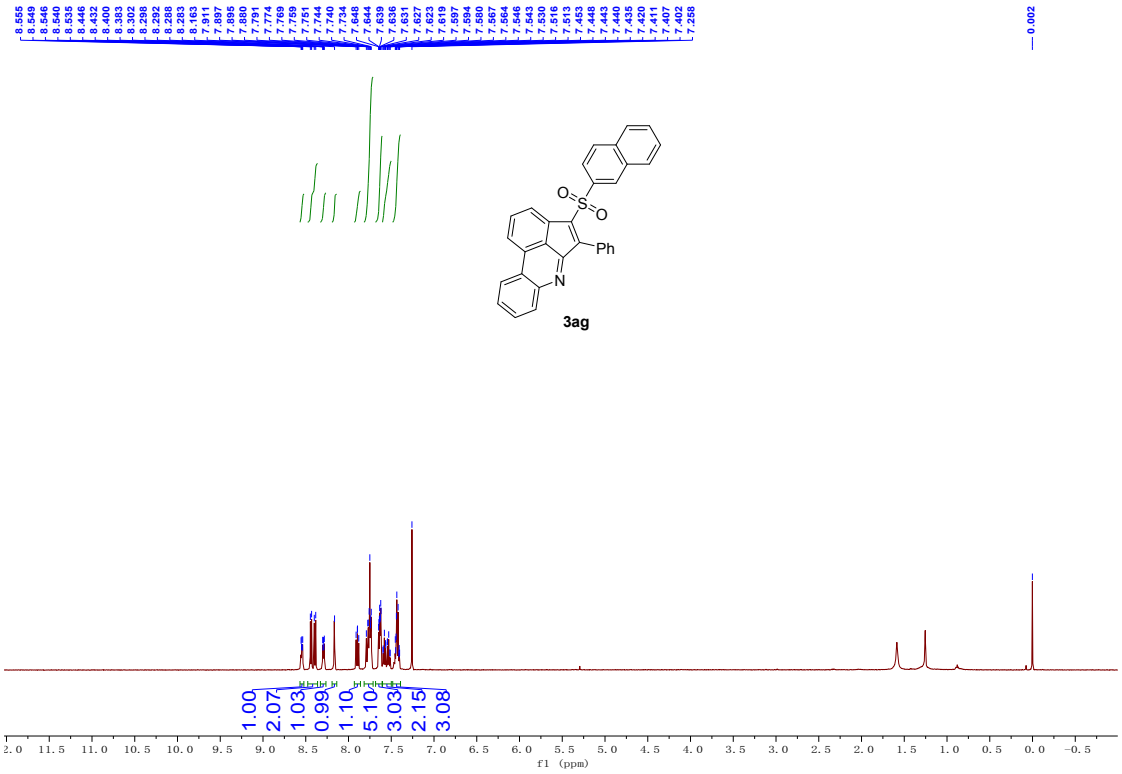
77.657
77.394
77.144
NO2
O
S
O
Ph
N
f1 (ppm)

3aj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8.283
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7.911
7.897
7.895
7.880
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7.774
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7.751
7.744
7.740
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7.644
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7.636
7.631
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7.623
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7.594
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7.554
7.546
7.543
7.530
7.516
7.513
7.453
7.448
7.443
7.440
7.435
7.420
7.411
7.407
7.402
7.258
0.002
O
S
O
Ph
N
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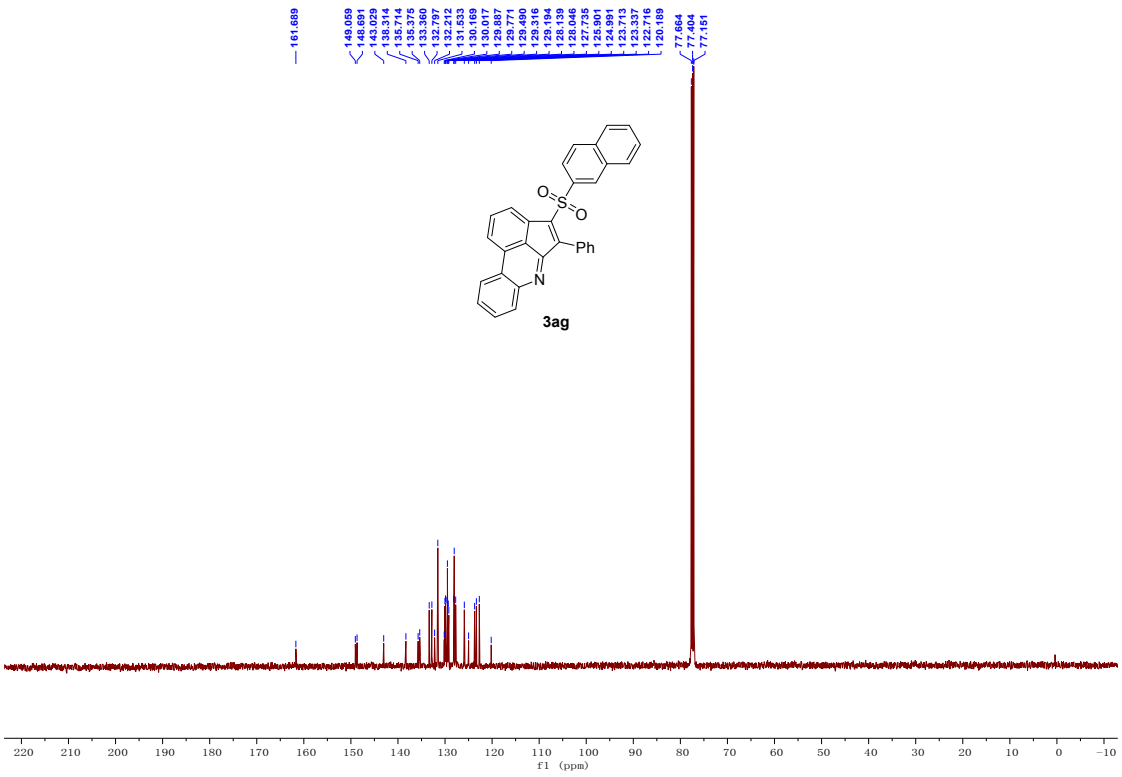
1.10
5.10
3.03
2.15
3.08
f1 (ppm)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129.771
129.490
129.316
129.164
128.139
128.046
127.735
125.901
124.991
123.713
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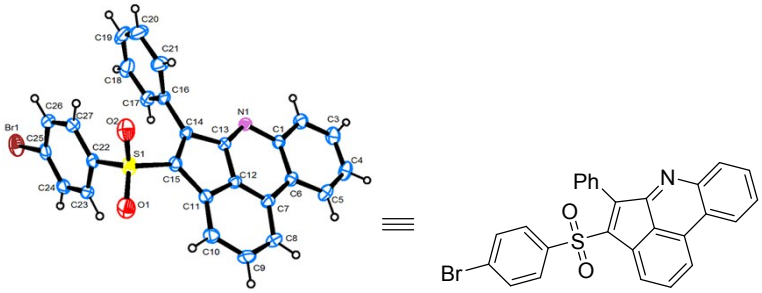
122.716
120.169
77.664
77.404
77.151
O
S
O
Ph
N
3ag
f1 (ppm)



X-ray crystal structure of **3ad**

X-ray Crystal Structure of **3ad**, CCDC 1952616. Thermal ellipsoids are drawn at 30% probability level.

Identification code	**3ad**
Empirical formula	C27 H16 Br N O2 S
Formula weight	498.38
Temperature	273(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $P2_1/n$
Unit cell dimensions	a = 7.9131 (3)Å alpha = 90 deg.
	b = 9.8841 (5), Å beta = 91.765(2) deg
	c = 26.9679 (12) Å gamma = 90 deg.
Volume	2108.26 (16) $Å^3$
Z, Calculated density	4, 1.570 g/cm^3
Absorption coefficient	2.075 mm^{-1}
F(000)	1008
Theta range for data collection	3.064 to 27.55 deg.
Limiting indices	-10<=h<=10, -12<=k<=12, -35<=l<=35
Absorption correction	-

Max. and min. transmission	0.9295 and 0.9115
Data / parameters	4819 /289
Goodness-of-fit on F^2	1.074
Final R indices [I>2sigma(I)]	R1 = 0.0441, wR2 = 0.1105
R indices (all data)	R1 = 0.0532, wR2 = 0. 1039
Largest diff. peak and hole	0.384 and -0.796 e. $Å^{-3}$