Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Pd(II)-Catalyzed [3+2] Spiroannulation of α-Aryl-β-naphthols with Alkynes via a C-H Activation/Dearomatization Approach

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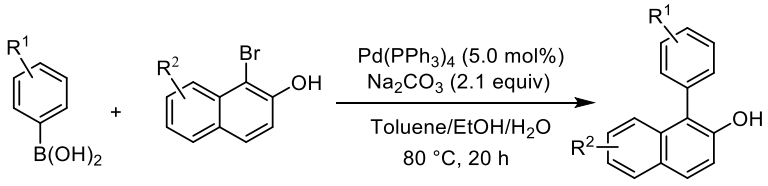
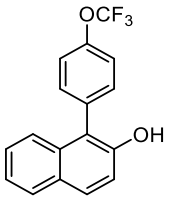
A. General information:

All reactions were carried out under an argon atmosphere using standard Schlenk-Lines or a glovebox (Innovative Technology). All reagents were used as received unless otherwise noted. CH_3CN, DMF and DME were dried over CaH_2. Toluene, and 1,4-dioxane were dried over sodium. Analytical thin-layer chromatography was performed with 0.25 mm coated commercial silica gel plates (TLC Silica Gel 60 F_{254}); visualization of the developed chromatogram was performed by fluorescence. Flash chromatography was performed with silica gel (300-400 mesh). Proton nuclear magnetic resonance (^{1}H NMR) data were acquired on Bruker Ascend 400 (400 MHz) spectrometer. Chemical shifts are reported in delta (δ) units, in parts per million (ppm) down field from tetramethylsilane. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; dd, doublet of doublets; td, triplet of doublets; m, multiplet, Coupling constants *J* are quoted in Hz. Carbon-13 nuclear magnetic resonance (^{13}C NMR) data were acquired at 100 MHz on Bruker Ascend 400 spectrometer Chemical shifts are reported in ppm relative to the center line of a triplet at 77.0 ppm for chloroform-*d* and the center line of a septet at 44.0 ppm for DMSO-d_6. Fluorine nuclear magnetic resonance (^{19}F NMR) data were acquired at 376 MHz on a Bruker Ascend 400 spectrometer, and chemical shifts are reported relative to inter standard $CFCl_3$ at 0.0 ppm. Infrared (IR) data were recorded as films on potassium bromide plates on a Bruker Tensor 27 FT-IR spectrometer. Absorbance frequencies are reported in reciprocal centimeters (cm^{-1}). Mass spectra were acquired on a Bruker Daltonics MicroTof-Q II mass spectrometer. HPLC analyses were performed on an Agilent Technologies 1260 Series using Daicel Chiralpak columns (IA, IB, IC,) in *n*-hexane/*i*-PrOH. Optical rotations were measured on a Rudolph Research Analytical Autopol II automatic polarimeter. Substrates **1a**[1], **1b**[2], **1c**[3], **1d**[2], **1e**[2], **1g**[2], **1h**[1], **1l**[1], **1q**[5], **1s**[6], **2a-m**[7-9], [D_1]-**1a**[2], **and** [D_5]-**1a**[2] were prepared according to the literature methods.

B. Preparation of substrates:

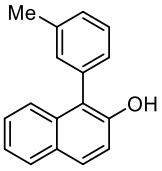


A 50 mL round bottom flask with a stir bar was fitted with a rubber septum and flame dried under high vacuum. The flask was purged with argon and charged with $Pd(PPh_3)_4$ (115.6 mg, 0.1 mmol), Na_2CO_3 (445.2 mg, 4.2 mmol), 1-bromonaphthalen-2-ol (446.1 mg, 2.0 mmol), arylboronic acid (4.0 mmol), 10.0 mL deoxygenated toluene, 2.0 mL deoxygenated ethanol, and 2.2 mL deoxygenated water. The mixture was stirred at 80 °C until the reaction was judged to be completed by TLC analysis. Water was added and extracted with EtOAc. The organic phase was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was then chromatographed on silica gel to afford the desired product **1**.



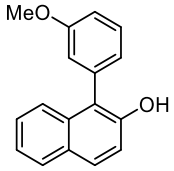
1-(4-(Trifluoromethoxy)phenyl)naphthalene (1f)

White solid (0.49 g, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.80 (m, 2H), 7.37 (q, *J* = 8.7 Hz, 4H), 7.35-7.32 (m, 3H), 7.26-7.24 (m, 1H), 4.91 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2, 149.3, 133.2, 133.0, 132.8, 129.9, 128.9, 128.2, 126.8, 124.3, 123.5, 121.9, 120.6 (d, *J* = 258.0 Hz), 119.6, 117.5. ^{19}F NMR (376 MHz, $CDCl_3$): δ -57.67 (s, 3F). IR (KBr): 3543, 3063, 2965, 1621, 1594, 1509, 1215, 1169, 814, 756, 748 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{11}F_3O_2Na$ $[M+Na]^+$ 327.0609, found 327.0613.



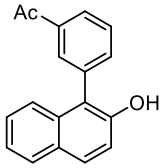
1-(*m*-Tolyl)naphthalen-2-ol (1i)

White solid (0.36 g, 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.82 (m, 2H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.47-7.45 (m, 1H), 7.39-7.34 (m, 3H), 7.31-7.25 (m, 3H), 5.20 (s, 1H), 2.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.1, 139.5, 134.1, 133.3, 131.7, 129.5, 129.4, 129.3, 128.9, 128.1, 128.0, 126.4, 124.7, 123.3, 121.1, 117.3, 21.5. IR (KBr): 3534, 2962, 2921, 2861, 2251, 1618, 1596, 1388, 1313, 1197, 1144, 909, 784, 743 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{14}ONa$ $[M+Na]^+$ 257.0942, found 257.0944.



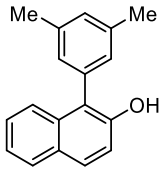
1-(3-Methoxyphenyl)naphthalen-2-ol (1j)

White solid (0.41 g, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82-7.79 (m, 2H), 7.52-7.48 (m, 1H), 7.46-7.44 (m, 1H), 7.37-7.31 (m, 2H), 7.27-7.25 (m, 1H), 7.06-7.03 (m, 1H), 7.04-7.00 (m, 1H), 6.96-6.95 (m, 1H), 5.21 (s, 1H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.6, 150.1, 135.5, 133.2, 130.8, 129.5, 128.9, 128.0, 126.5, 124.6, 123.3, 123.2, 120.8, 117.4, 116.3, 114.4, 55.4. IR (KBr): 3452, 3060, 3025, 2938, 2839, 1618, 1593, 1461, 1423, 813, 748 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{14}O_2Na$ $[M+Na]^+$ 273.0891, found 273.0852.



1-(3-(2-Hydroxynaphthalen-1-yl)phenyl)ethan-1-one (1k)

White solid (0.40 g, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (d, *J* = 7.6 Hz, 1H), 8.01 (s, 1H), 7.83 (d, *J* = 8.8 Hz, 2H), 7.71-7.63 (m, 2H), 7.36-7.30 (m, 3H), 7.26 (d, *J* = 8.9 Hz, 1H), 5.09 (s, 1H), 2.63 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.1, 150.3, 138.1, 136.1, 135.2, 133.2, 131.3, 129.9, 129.8, 128.7, 128.3, 128.2, 126.8, 124.3, 123.5, 120.3, 117.7, 26.8. IR (KBr): 3391, 3061, 2923, 2745, 2674, 1676, 1623, 1592, 1211, 1145, 909, 814, 698, 561 cm^{-1}. HRMS (ESI) m/z calculated for $C_{18}H_{14}O_2Na$ $[M+Na]^+$ 285.0891, found 285.0884.

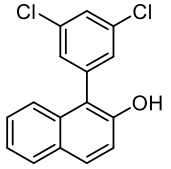


1-(3,5-Dimethylphenyl)naphthalen-2-ol (1m)

White solid (0.39 g, 78% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81-7.77 (m, 2H), 7.45-7.43 (m, 1H), 7.35-7.29 (m, 2H), 7.25 (d, *J* = 8.8 Hz, 1H), 7.12 (s, 1H), 7.03 (s, 2H), 5.21 (s, 1H), 2.39 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.1, 139.3, 133.9, 133.3, 130.1, 129.3, 128.9, 128.7, 128.0, 126.4, 124.8, 123.2, 121.3, 117.3, 21.4. IR (KBr): 3531, 3055, 2917, 2862, 1595, 1512, 1388, 1202, 1035, 855, 816, 746 cm^{-1}. HRMS (ESI) m/z calculated for $C_{18}H_{16}ONa$ $[M+Na]^+$ 271.1098, found 271.1085.

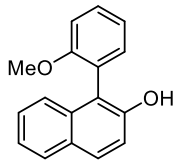
1-(3,5-Difluorophenyl)naphthalen-2-ol (1n)

Colorless oil (0.42 g, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.8 Hz, 2H), 7.31-7.26 (m, 3H), 7.15-7.13 (m, 1H), 6.90-6.86 (m, 3H), 4.93 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.8 (dd, *J* = 251.1, 13.0 Hz) 149.9, 137.9, 132.7, 130.4, 128.9, 128.2, 127.0, 124.1, 123.7, 118.9, 117.5, 114.3 (dd, *J* = 18.0, 6.9 Hz), 104.1 (t, *J* = 25.1 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ -107.91 (s, 2F). IR (KBr): 3544, 3063, 1620, 1590, 1427, 1331, 1143, 988, 862, 815 cm^{-1}. HRMS (ESI) m/z calculated for $C_{16}H_{11}F_2O$ $[M+H]^+$ 257.0778, found 257.0775.



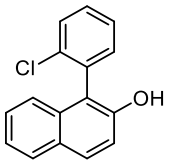
1-(3,5-Dichlorophenyl)naphthalen-2-ol (1o)

Colorless oil (0.36 g, 63% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.9 Hz, 2H), 7.42 (s, 1H), 7.31-7.24 (m, 5H), 7.13 (d, *J* = 8.9 Hz, 1H), 4.90 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.1, 137.8, 136.1, 132.8, 130.4, 129.7, 128.9, 128.6, 127.1, 125.7, 124.1, 123.7, 118.6, 117.5. IR (KBr): 3067, 1621, 1587, 1557, 1513, 1390, 1177, 1147, 859, 813, 748 cm^{-1}. HRMS (ESI) m/z calculated for $C_{16}H_{10}Cl_2ONa$ $[M+Na]^+$ 311.006, found 311.004.



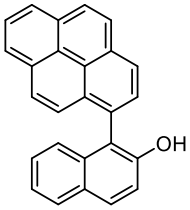
1-(2-Methoxyphenyl)naphthalen-2-ol (1p)

White solid (0.39 g, 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.77 (m, 2H), 7.49-7.45 (m, 1H), 7.34-7.28 (m, 4H), 7.23 (d, *J* = 4.1 Hz, 1H), 7.14-7.10 (m, 2H), 5.24 (s, 1H), 3.72 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.9, 150.7, 133.5, 133.4, 130.3, 129.5, 129.1, 128.1, 126.3, 124.9, 123.2, 122.6, 121.6, 117.9, 117.8, 111.9, 55.8. IR (KBr): 3057, 2938, 2837, 1621, 1594, 1495, 1461, 1179, 1023, 940, 861, 756 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{14}O_2Na$ $[M+Na]^+$ 273.0891, found 273.0888.



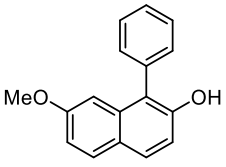
1-(2-Chlorophenyl)naphthalen-2-ol (1r)

Colorless oil (0.36 g, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.71 (m, 2H), 7.53 (dd, *J* = 5.8, 3.4 Hz, 1H), 7.36-7.32 (m, 2H), 7.30-7.24 (m, 3H), 7.16 (d, *J* = 8.9 Hz, 1H), 7.12-7.09 (m, 1H), 4.82 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3, 135.9, 133.3, 133.2, 133.0, 130.6, 130.3, 130.2, 128.9, 128.2, 127.8, 126.8, 124.3, 123.6, 118.6, 117.6. IR (KBr): 3539, 3069, 2962, 1621, 1585, 1513, 1390, 1265, 1177, 858, 813 cm^{-1}. HRMS (ESI) m/z calculated for $C_{16}H_{11}ClONa$ $[M+Na]^+$ 277.0396, found 277.0387.



1-(Pyren-1-yl)naphthalen-2-ol (1t)

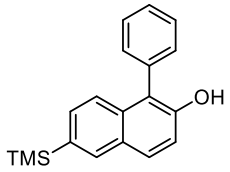
White solid (0.37 g, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (d, *J* = 7.8 Hz, 1H), 8.26 (d, *J* = 7.6 Hz, 1H), 8.19-8.17 (m, 3H), 8.07-8.02 (m, 2H), 7.96 (dd, *J* = 9.0, 3.0 Hz, 2H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.64 (d, *J* = 9.2 Hz, 1H), 7.40 (d, *J* = 8.9 Hz, 1H), 7.36-7.32 (m, 1H), 7.24-7.20 (m, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 4.93 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.3, 134.3, 131.9, 131.4, 131.2, 130.9, 130.2, 129.6, 129.2, 128.6, 128.6, 128.3, 128.2, 127.4, 126.8, 126.5, 125.7, 125.6, 125.5, 125.4, 125.2, 125.0, 124.8, 123.6, 119.4, 117.7. IR (KBr): 3045, 2925, 2852, 1619, 1597, 1515, 1463, 1390, 1298, 1195, 1145, 907, 848, 816, 732 cm^{-1}. HRMS (ESI) m/z calculated for $C_{26}H_{17}O$ $[M+H]^+$ 345.1279, found 345.1274.



7-Methoxy-1-phenylnaphthalen-2-ol (3u)

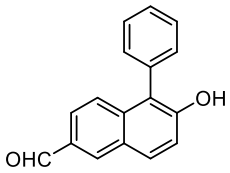
White solid (0.37 g, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (dd, *J* = 8.8, 4.6 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.43 (d, *J* = 7.0 Hz, 2H), 7.11 (d, *J* = 8.8 Hz, 1H), 7.00 (dd, *J* = 8.9, 2.4 Hz, 1H), 6.70 (d, *J* = 2.3 Hz, 1H), 5.09 (s, 1H), 3.69 (s, 3H). ^{13}C NMR (100 MHz,

CDC_{l3}): δ 158.3, 150.8, 134.6, 134.3, 131.1, 129.8, 129.6, 129.3, 128.5, 124.4, 120.3, 115.3, 114.8, 103.8, 55.1. IR (KBr): 3434, 2934, 2875, 1619, 1507, 1458, 1269, 1222, 1033, 833, 757, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{15}O_2$ $[M+H]^+$ 251.1072, found 251.1088.



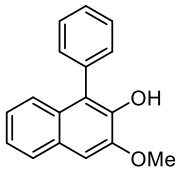
1-Phenyl-6-(trimethylsilyl)naphthalen-2-ol (1v)

Yellow solid (0.49 g, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (s, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.69-7.63 (m, 4H), 7.60-7.57 (m, 3H), 7.45 (d, *J* = 8.9 Hz, 1H), 5.49 (s, 1H), 0.55 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 135.6, 135.3, 134.9, 134.6, 132.2, 131.6, 130.8, 130.6, 129.5, 129.4, 124.8, 121.9, 118.4, 0.00. IR (KBr): 3438, 3057, 2926, 2855, 1618, 1594, 1439, 1219, 816, 726 cm^{-1}. HRMS (ESI) m/z calculated for $C_{19}H_{21}OSi$ $[M+H]^+$ 293.1362, found 293.1344.



6-Hydroxy-5-phenyl-2-naphthaldehyde (1w)

White solid (0.37 g, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.03 (s, 1H), 8.23 (d, *J* = 1.2 Hz, 1H), 7.89 (d, *J* = 8.9 Hz, 1H), 7.73 (dd, *J* = 8.8, 1.5 Hz, 1H), 7.54 (t, *J* = 7.3 Hz, 2H), 7.47 (dd, *J* = 8.6, 6.2 Hz, 1H), 7.41 (d, *J* = 8.8 Hz, 1H), 7.37-7.32 (m, 2H), 7.29 (d, *J* = 8.9 Hz, 1H), 5.35 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.0, 153.1, 136.7, 134.5, 133.2, 132.0, 131.3, 131.1, 129.9, 129.0, 128.0, 125.7, 123.7, 121.8, 118.6. IR (KBr): 3384, 3057, 2925, 1699, 1667, 1464, 1365, 1264, 1155, 748, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{13}O_2$ $[M+H]^+$ 249.0916, found 249.0910.

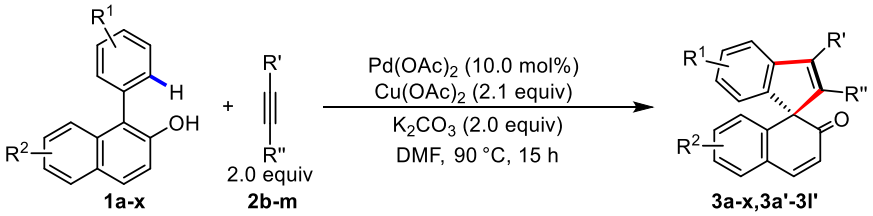


3-Methoxy-1-phenylnaphthalen-2-ol (1x)

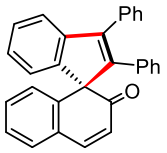
White solid (0.34 g, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 7.3 Hz, 2H), 7.41-7.33 (m, 4H), 7.28-7.22 (m, 1H), 7.17 (dd, *J* = 3.9, 2.5 Hz, 1H), 7.11 (s, 1H), 5.80 (s, 1H), 3.99 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.2, 142.5, 135.4, 130.9, 129.0, 128.9, 128.6,

127.6, 126.8, 126.5, 126.4, 124.9, 124.3, 124.0, 122.0, 109.5, 105.8, 105.5, 56.0. IR (KBr): 3055, 2939, 2832, 1629, 1601, 1511, 1345, 947, 881 cm^{-1}. HRMS (ESI) m/z calculated for $C_{17}H_{14}O_2Na$ $[M+Na]^+$ 273.0891, found 273.0891.

C. Catalytic results:

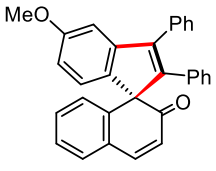


In a glovebox, a 5.0 mL vial equipped with a stir bar was charged with $Pd(OAc)_2$ (4.4 mg, 0.02 mmol), $Cu(OAc)_2$ (76.0 mg, 0.42 mmol), K_2CO_3 (55.2 mg, 0.40 mmol), 1-aryl-2-naphthol **1** (0.20 mmol), and alkyne **2** (0.40 mmol) followed by sequential addition of DMF (2.0 mL). The vial was sealed with a Teflon screw cap and then the reaction mixture was heated at 90 °C for 15 h. The crude reaction mixture was then subjected to a silica gel column to afford the desired product **3**.



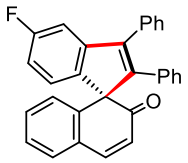
2,3-Diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3a)

Yellow solid (70.5 mg, 89% yield). PE/EA = 10:1, R_f = 0.21. The spectroscopic data is consistent with that reported in the literature[2] .^{1}H NMR (400 MHz, DMSO-d_6) δ 8.03 (d, *J* = 9.9 Hz, 1H), 7.67 (d, *J* = 7.5 Hz, 1H), 7.58-7.46 (m, 5H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.06-7.03 (m, 3H), 6.97 (d, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 7.7 Hz, 1H), 6.79-6.77 (m, 2H), 6.44 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 196.2, 147.8, 147.6, 145.4, 144.9, 144.7, 140.9, 135.2, 134.4, 131.3, 131.0, 129.9, 129.6, 129.5, 129.0, 128.7, 128.6 128.5, 128.3, 127.7, 127.0, 126.7, 126.3, 121.8, 121.7, 71.6.



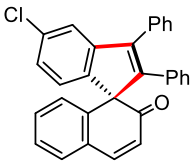
5-Methoxy-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3b)

Yellow solid (76.7 mg, 90% yield). PE/EA = 20:1, R_f = 0.34. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 9.9 Hz, 1H), 7.52-7.50 (m, 2H), 7.46-7.36 (m, 4H), 7.28-7.24 (m, 1H), 7.16 (td, *J* = 7.6, 1.4 Hz, 1H), 7.00-6.95 (m, 4H), 6.89 (d, *J* = 8.3 Hz, 1H), 6.83-6.80 (m, 3H), 6.60 (dd, *J* = 8.3, 2.5 Hz, 1H), 6.38 (d, *J* = 9.9 Hz, 1H), 3.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 159.8, 147.0, 146.3, 146.1, 144.5, 141.4, 139.9, 135.1, 134.4, 130.7, 129.9, 129.6, 129.5, 129.1, 128.9, 128.0, 127.9, 127.5, 127.0, 126.9, 126.5, 122.3, 111.9, 107.7, 71.1, 55.5. IR (KBr): 3056, 2941, 2835, 1662, 1599, 1471, 1441, 954, 741, 636 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}O_2Na$ $[M+Na]^+$ 449.1517, found 449.1514.



5-Fluoro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3c)

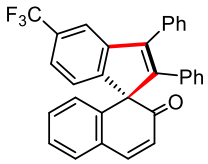
Yellow solid (71.2 mg, 86% yield). PE/EA = 20:1, R_f = 0.34. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 9.9 Hz, 1H), 7.50-7.48 (m, 2H), 7.46-7.39 (m, 4H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.02-6.90 (m, 6H), 6.82 (d, *J* = 7.2 Hz, 2H), 6.74 (td, *J* = 8.7, 2.3 Hz, 1H), 6.37 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 163.1 (d, *J* = 244.8 Hz), 147.8 (d, *J* = 8.8 Hz), 147.2, 146.4, 143.9, 143.2, 140.7, 134.6, 134.0, 130.8, 130.1, 129.7, 129.4, 129.1, 129.0, 128.2, 128.1, 127.8, 127.4, 126.9, 126.5, 122.8 (d, *J* = 9.2 Hz), 113.0 (d, *J* = 23.4 Hz), 109.2 (d, *J* = 24.2 Hz), 71.1. ^{19}F NMR (376 MHz, $CDCl_3$): δ -114.31 (s, 1F). IR (KBr): 3058, 2926, 1664, 1597, 1467, 1267, 1235, 1201, 872, 817, 740, 699 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{19}FONa$ $[M+Na]^+$ 437.1317, found 437.1337.



5-Chloro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3d)

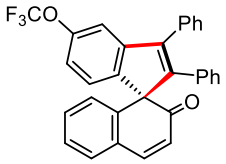
Yellow solid (70.5 mg, 82% yield). PE/EA = 20:1, R_f = 0.23. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 9.9 Hz, 1H), 7.57 (d, *J* = 7.1 Hz, 2H), 7.52-7.45 (m, 4H), 7.34-7.30 (m, 2H), 7.24-7.20 (m, 1H), 7.10-6.96 (m, 6H), 6.90 (d, *J* = 6.6 Hz, 2H), 6.44 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.1, 147.5, 146.9, 146.4, 146.0, 143.8, 140.4, 134.6, 134.0, 133.9, 130.9, 130.2, 129.7, 129.5,

129.2, 129.1, 128.2, 128.1, 127.9, 127.5, 126.9, 126.5, 126.3, 122.8, 122.0, 71.3. IR (KBr): 3057, 2925, 1664, 1618, 1489, 1455, 1263, 1160, 1122, 861, 748, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{20}ClO$ $[M+H]^+$ 431.1203, found 431.1213.



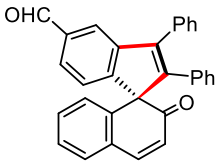
2,3-Diphenyl-5-(trifluoromethyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3e)

Yellow solid (87.3 mg, 94% yield). PE/EA = 5:1, R_f = 0.19.The spectroscopic data is consistent with that reported in the literature[4].



2,3-Diphenyl-5-(trifluoromethoxy)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3f)

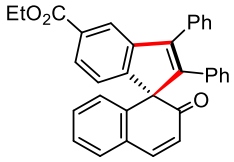
Yellow solid (88.3 mg, 92% yield). PE/EA = 20:1, R_f = 0.36. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 9.9 Hz, 1H), 7.46-7.35 (m, 6H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.08 (s, 1H), 7.01-6.86 (m, 6H), 6.80-6.78 (m, 2H), 6.35 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.1, 149.3, 147.6, 147.3, 146.4, 145.9, 143.7, 140.2, 134.4, 133.8, 130.8, 130.2, 129.7, 129.4, 129.1, 129.0, 128.2, 128.1, 127.9, 127.5, 126.9, 126.4, 122.6, 120.4 (d, *J* = 257.1 Hz), 118.7, 114.8, 71.2. ^{19}F NMR (376 MHz, $CDCl_3$): δ -57.72 (s, 3F). IR (KBr): 3059, 2927, 1666, 1597, 1564, 1467, 1263, 1165, 743, 699 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{19}F_3O_2Na$ $[M+Na]^+$ 503.1235, found 503,1220.



2'-Oxo-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalene]-5-carbaldehyde (3g)

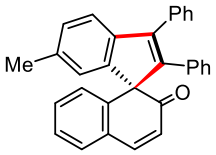
Yellow solid (71.3 mg, 84% yield). PE/EA = 10:1, R_f = 0.26. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.95 (s, 1H), 7.79 (d, *J* = 1.0 Hz, 1H), 7.71 (d, *J* = 9.9 Hz, 1H), 7.61 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.54-7.51 (m, 2H), 7.50-7.41 (m, 4H), 7.31 (td, *J* = 7.5, 1.2 Hz, 1H), 7.20 (td, *J* = 7.6, 1.4 Hz, 1H), 7.15 (d, *J* = 7.7 Hz, 1H), 6.86-6.97 (m, 3H), 6.94 (d, *J* = 7.8 Hz, 1H), 6.86-6.83 (m, 2H), 6.42 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 191.9, 153.8, 146.8, 146.7, 146.6, 143.7, 139.7, 136.4, 134.4,

133.7, 130.9, 130.3, 129.7, 129.4, 129.2, 129.1, 129.0, 128.3, 128.2, 128.1, 127.6, 126.9, 126.4, 122.3, 122.2, 71.8. IR (KBr): 3438, 3054, 2835, 2755, 1695, 1663, 1169, 878, 739, 698 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{21}O_2$ $[M+H]^+$ 425.1542, found 425.1532.



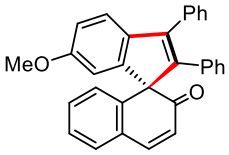
Ethyl-2'-oxo-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalene]-5-carboxylate (3h)

Yellow solid (79.0 mg, 87% yield). PE/EA = 10:1, R_f = 0.24.The spectroscopic data is consistent with that reported in the literature[4].



6-Methyl-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3i)

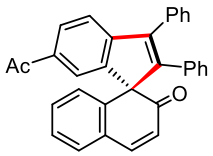
Yellow solid (68.1 mg, 83% yield). PE/EA = 20:1, R_f = 0.33. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 9.9 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.45-7.36 (m, 4H), 7.29-7.25 (m, 1H), 7.19-7.16 (m, 2H), 7.04 (d, *J* = 7.7 Hz, 1H), 6.99-6.95 (m, 4H), 6.83-6.80 (m, 3H), 6.40 (d, *J* = 9.9 Hz, 1H), 2.23 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 147.9, 146.2, 144.6, 144.2, 142.8, 141.4, 136.3, 135.4, 134.5, 130.7, 129.9, 129.6, 129.5, 129.0, 128.8, 128.5, 127.9, 127.8, 127.5, 127.0, 126.8, 126.6, 122.4, 121.6, 71.5, 21.5. IR (KBr): 3056, 3027, 1592, 2920, 2858, 1663, 1485, 1444, 1267, 1235, 1200, 741, 699 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}ONa$ $[M+Na]^+$ 433.1568, found 433.1567.



6-Methoxy-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3j)

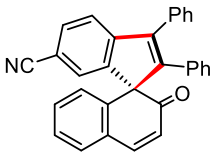
Yellow solid (69.0 mg, 81% yield). PE/EA = 20:1, R_f = 0.29. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 9.9 Hz, 1H), 7.53-7.50 (m, 2H), 7.43-7.35 (m, 4H), 7.28-7.24 (m, 1H), 7.20-7.14 (m, 2H), 6.99-6.93 (m, 4H), 6.82-6.79 (m, 2H), 6.76 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.58 (d, *J* = 2.3 Hz, 1H), 6.38 (d, *J* = 9.9 Hz, 1H), 3.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.8, 158.7, 149.3, 146.3, 144.3, 143.2, 141.3, 138.3, 135.4, 134.5, 130.8, 130.0, 129.6, 129.5, 128.9, 128.8, 127.9, 127.7, 127.6, 127.0,

126.8, 126.5, 122.5, 112.6, 108.7, 71.4, 55.5. IR (KBr): 3055, 2938, 2835, 1662, 1597, 1481, 1276, 1222, 1029, 741, 698 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}O_2Na$ $[M+Na]^+$ 449.1517, found 449.1510.



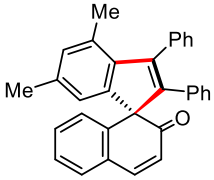
6-Acetyl-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3k)

Yellow solid (71.9 mg, 82% yield). PE/EA = 10:1, R_f = 0.35. ^1H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.72 (d, *J* = 9.9 Hz, 1H), 7.58 (d, *J* = 1.3 Hz, 1H), 7.52-7.39 (m, 6H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.29 (td, *J* = 7.5, 1.1 Hz, 1H), 7.17 (td, *J* = 7.6, 1.3 Hz, 1H), 7.06-6.97 (m, 3H), 6.91 (d, *J* = 7.8 Hz, 1H), 6.84-6.82 (m, 2H), 6.41 (d, *J* = 9.9 Hz, 1H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 196.1, 150.7, 149.0, 148.1, 146.8, 144.0, 139.9, 135.2, 134.5, 133.8, 130.8, 130.4, 129.8, 129.5, 129.2, 129.1, 129.0, 128.2, 128.1, 128.0, 127.7, 126.8, 126.4, 121.5, 121.2, 71.6, 26.7. IR (KBr): 3056, 1670, 1595, 1487, 1261, 1232, 1028, 834, 745, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{32}H_{22}O_2Na$ $[M+Na]^+$ 461.1517, found 461.1510.



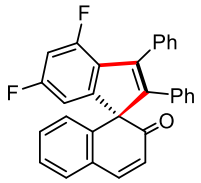
2'-Oxo-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalene]-6-carbonitrile (3l)

Yellow solid (65.7 mg, 78% yield). PE/EA = 10:1, R_f = 0.24. ^1H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 10.0 Hz, 1H), 7.54 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.50-7.41 (m, 6H), 7.36-7.32 (m, 2H), 7.24-7.19 (m, 2H), 7.08-6.99 (m, 3H), 6.89 (d, *J* = 7.7 Hz, 1H), 6.84 (d, *J* = 7.9 Hz, 2H), 6.42 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.2, 150.2, 148.9, 147.9, 146.8, 143.8, 139.3, 134.0, 133.3, 132.4, 131.0, 130.5, 129.6, 129.4, 129.2, 129.1, 128.5, 128.4, 128.2, 127.9, 126.7, 126.3, 125.1, 122.3, 119.2, 109.1, 71.5. IR (KBr): 3057, 2224, 1664, 1596, 1563, 1477, 1433, 1267, 1234, 1200, 839, 740, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{19}NONa$ $[M+Na]^+$ 444.1364, found 444.1350.



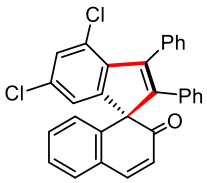
4,6-Dimethyl-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3m)

Yellow solid (74.7 mg, 88% yield). PE/EA = 20:1, R_f = 0.23.The spectroscopic data is consistent with that reported in the literature[10].



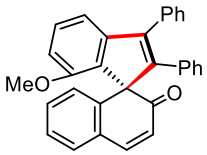
4,6-Difluoro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3n)

Yellow solid (69.1 mg, 80% yield). PE/EA = 10:1, R_f = 0.22. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 9.9 Hz, 1H), 7.53 (d, *J* = 6.8 Hz, 2H), 7.46 (d, *J* = 7.4 Hz, 1H), 7.41-7.33 (m, 4H), 7.28-7.25 (m, 1H), 7.04-6.97 (m, 4H), 6.77-6.68 (m, 3H), 6.60-6.58 (m, 1H), 6.40 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 161.7 (dd, *J* = 249.2, 10.6 Hz), 156.3 (dd, *J* = 256.0, 12.3 Hz), 151.2 (dd, *J* = 9.5, 6.4 Hz), 146.5, 145.7, 141.7, 139.7, 135.1, 133.7, 130.9, 130.2, 129.7, 129.6, 129.5, 129.1, 128.3, 128.2, 127.9, 127.8, 127.3, 127.1, 126.3, 106.2 (dd, *J* = 23.9, 3.9 Hz) 104.0 (t, *J* = 25.7 Hz). 71.8. ^{19}F NMR (376 MHz, $CDCl_3$): δ -119.52 (d, *J* = 5.8 Hz 1F). -112.77 (d, *J* = 5.8 Hz 1F). IR (KBr): 3057, 1664, 1605, 1471, 1295, 1236, 1113, 1031, 988, 846, 737, 698 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{18}F_2ONa$ $[M+Na]^+$ 455.1223, found 455.1223.



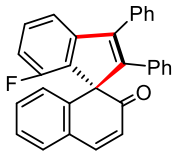
4,6-Dichloro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3o)

Yellow solid (72.4 mg, 78% yield). PE/EA = 10:1, R_f = 0.23. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 9.9 Hz, 1H), 7.52-7.40 (m, 6H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.28-7.21 (m, 2H), 7.03-7.00 (m, 2H), 6.98-6.94 (m, 3H), 6.75 (d, *J* = 7.4 Hz, 2H), 6.41 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.1, 150.6, 147.9, 146.5, 144.1, 139.9, 139.5, 135.6, 133.5, 132.2, 131.0, 130.3, 130.1, 129.9, 129.6, 129.1, 128.5, 128.4, 128.2, 128.1, 127.9, 127.4, 127.0, 126.3, 121.0, 71.1. IR (KBr): 3058, 2224, 1661, 1616, 1560, 1438, 1392, 1195, 1148, 752, 697 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{18}Cl_2ONa$ $[M+Na]^+$ 487.0632, found 487.0620.



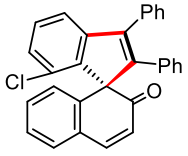
7-Methoxy-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3p)

Yellow solid (63.1 mg, 74% yield). PE/EA = 20:1, R_f = 0.28. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 9.9 Hz, 1H), 7.43-7.41 (m, 2H), 7.37-7.27 (m, 5H), 7.24-7.22 (m, 1H), 7.18 (td, *J* = 7.5, 1.5 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 2H), 6.94 (t, *J* = 7.4 Hz, 3H), 6.70-6.67 (m, 3H), 6.33 (d, *J* = 9.9 Hz, 1H), 3.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.1, 153.8, 148.7, 147.9, 145.3, 143.4, 139.4, 137.8, 134.7, 134.6, 130.9, 129.9, 129.6, 129.5, 129.2, 128.9, 128.5, 127.7, 127.6, 127.4, 127.2, 127.1, 126.6, 114.2, 110.0, 69.4, 55.5. IR (KBr): 3056, 2937, 2837, 1659, 1598, 1484, 1442, 1269, 1104, 769, 736 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}O_2Na$ $[M+Na]^+$ 449.1517, found 449.1515.



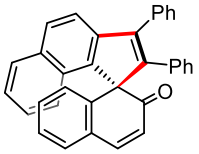
7-Fluoro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3q)

Yellow solid (65.4 mg, 79% yield). PE/EA = 20:1, R_f = 0.27. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 9.9 Hz, 1H), 7.44 (dd, *J* = 8.1, 1.6 Hz, 2H), 7.40-7.33 (m, 4H), 7.32-7.27 (m, 2H), 7.25-7.21 (m, 2H), 7.15 (d, *J* = 7.6 Hz, 1H), 7.05-7.01 (m, 1H), 6.98-6.94 (m, 3H), 6.80 (t, *J* = 8.7 Hz, 1H), 6.73-6.71 (m, 2H), 6.37 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 157.1 (d, *J* = 249.6 Hz), 149.2 (d, *J* = 5.4 Hz), 148.4, 146.1, 143.4, 138.5, 135.1, 134.9, 134.4, 133.9, 130.4, 130.0, 129.9, 129.8, 129.5, 128.9, 128.7, 127.9, 127.8, 127.5, 126.8, 117.4, 117.3, 113.9 (d, *J* = 20.2 Hz), 69.0. ^{19}F NMR (376 MHz, $CDCl_3$): δ -119.57 (s, 1F). IR (KBr): 3057, 2925, 2854, 1662, 1615, 1470, 1239, 1160, 765, 698, 636 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{20}FO$ $[M+H]^+$ 415.1498, found 415.1501.



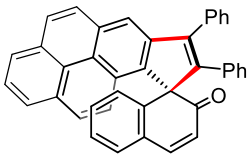
7-Chloro-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3r)

Yellow solid (54.2 mg, 63% yield). PE/EA = 20:1, R_f = 0.24. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 9.9 Hz, 1H), 7.39-7.28 (m, 10H), 7.19 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.08-7.06 (m, 1H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.91 (d, *J* = 7.6 Hz, 1H), 6.65 (d, *J* = 7.8 Hz, 2H), 6.34 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.9, 150.0, 148.9, 147.7, 145.8, 142.6, 137.8, 134.0, 133.9, 131.5, 130.3, 129.5, 129.4, 129.3, 129.0, 128.5, 128.0 127.9, 127.8, 127.6, 127.1, 126.7, 119.6, 71.2. IR (KBr): 3058, 2925, 1663, 1619, 1441, 1395, 1238, 1143, 1028, 796, 724, 635 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{20}ClO$ $[M+H]^+$ 431.1203, found 431.1187.



2,3-Diphenyl-2'H-spiro[cyclopenta[a]naphthalene-1,1'-naphthalen]-2'-one (3s)

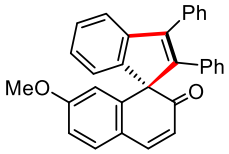
Yellow solid (80.3 mg, 90% yield). PE/EA = 10:1, R_f = 0.21. ^{1}H NMR (400 MHz, $CDCl_3$):δ 7.92 (dd, *J* = 12.9, 8.5 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.62 (d, *J* = 9.9 Hz, 1H), 7.51 (d, *J* = 7.0 Hz, 2H), 7.45-7.28 (m, 7H), 7.18 (t, *J* = 6.9 Hz, 2H), 7.13-7.09 (m, 1H), 7.06-7.02 (m, 2H), 6.85 (d, *J* = 7.6 Hz, 1H), 6.75 (d, *J* = 7.2 Hz, 2H), 6.44 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 150.2, 146.0, 145.9, 144.7, 143.5, 140.8, 134.6, 134.4, 133.3, 130.6, 130.4, 129.8, 129.7, 129.2, 129.1, 129.0, 128.5, 128.2, 127.8, 127.6, 127.4, 127.3, 127.1, 125.0, 123.4, 119.9, 71.6. IR (KBr): 3055, 2960, 1659, 1618, 1513, 1442, 1338, 1263, 1201, 820, 762, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{34}H_{22}ONa$ $[M+Na]^+$ 469.1568, found 469.1563.



7,8-Diphenyl-2'H-spiro[cyclopenta[a]pyrene-9,1'-naphthalen]-2'-one (3t)

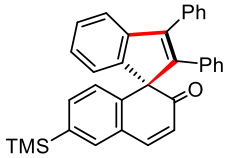
Yellow solid (67.6 mg, 65% yield). PE/EA = 5:1, R_f = 0.18. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (s, 1H), 8.15 (d, *J* = 7.5 Hz, 1H), 8.10-8.04 (m, 3H), 7.91 (t, *J* = 7.6 Hz, 1H), 7.84 (d, *J* = 9.1 Hz, 1H), 7.65 (d, *J* = 10.0 Hz, 1H), 7.56 (d, *J* = 7.0 Hz, 2H), 7.46-7.35 (m, 5H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.10 (dd, *J* = 15.2, 7.7 Hz, 2H), 7.01 (t, *J* = 7.5 Hz, 2H), 6.77 (d, *J* = 7.8 Hz, 1H), 6.73 (d, *J* = 7.3 Hz, 2H), 6.45 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 150.6, 146.0, 144.9, 143.7, 143.6, 140.7, 134.5, 134.4, 131.9, 131.1, 130.6, 130.5, 129.8, 129.7, 129.3, 128.9, 128.5, 128.0, 127.9, 127.8, 127.7,

127.6, 127.5, 127.4, 127.3, 125.6, 125.5, 125.4, 125.3, 125.1, 124.4, 123.0, 117.8, 71.4. IR (KBr): 3049, 2365, 1658, 1557, 1484, 1435, 1390, 1194, 1089, 877, 819, 694 cm^{-1}. HRMS (ESI) m/z calculated for $C_{40}H_{24}ONa$ $[M+Na]^+$ 543.1725, found 543.1690.



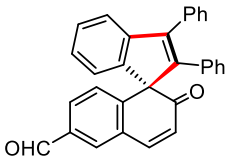
7'-Methoxy-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3u)

Yellow solid (69.9 mg, 82% yield). PE/EA = 10:1, R_f = 0.21. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, *J* = 9.9 Hz, 1H), 7.56 (d, *J* = 6.9 Hz, 2H), 7.47 (t, *J* = 7.3 Hz, 2H), 7.43-7.38 (m, 2H), 7.30 (s, 1H), 7.27-7.23 (m, 1H), 7.10-7.01 (m, 5H), 6.92-6.89 (m, 2H), 6.80 (dd, *J* = 8.4, 2.5 Hz, 1H), 6.53 (d, *J* = 2.4 Hz, 1H), 6.30 (d, *J* = 9.9 Hz, 1H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.6, 161.5, 147.9, 146.0, 145.3, 145.0, 144.7, 143.4, 135.3, 134.3, 131.5, 129.5, 129.2, 128.8, 128.0, 127.9, 127.7, 127.0, 126.3, 124.0, 123.0, 121.9, 121.5, 113.0, 112.6, 71.8, 55.2. IR (KBr): 3058, 2933, 2839, 1660, 1599, 1462, 1228, 1060, 1033, 872, 700, 645 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}O_2Na$ $[M+Na]^+$ 449.1517, found 449.1511.



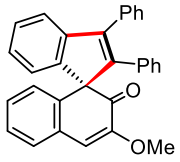
2,3-Diphenyl-6'-(trimethylsilyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3v)

Brown solid (79.6 mg, 85% yield). PE/EA = 10:1, R_f = 0.22. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 9.9 Hz, 1H), 7.52 (d, *J* = 6.0 Hz, 3H), 7.44 (t, *J* = 7.3 Hz, 2H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.30-7.27 (m, 2H), 7.24-7.20 (m 1H), 7.04-6.93 (m, 6H), 6.86 (d, *J* = 7.1 Hz, 2H), 6.39 (d, *J* = 9.9 Hz, 1H), 0.25 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.0, 148.8, 147.8, 146.6, 146.0, 145.9, 142.8, 141.1, 136.9, 136.5, 136.2, 135.6, 130.7, 130.4, 130.1, 130.0, 129.1, 129.0, 128.4, 128.1, 127.7, 127.5, 127.2, 123.0, 122.8, 72.9, 0.0. IR (KBr): 3058, 2955, 2898, 2856, 1665, 1614, 1409, 1246, 1200, 884, 752, 697, 584 cm^{-1}. HRMS (ESI) m/z calculated for $C_{33}H_{29}OSi$ $[M+H]^+$ 469.1988, found 469.1987.



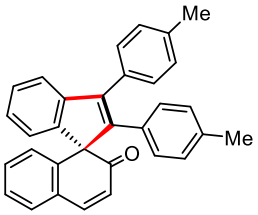
2'-Oxo-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalene]-6'-carbaldehyde (3w)

Yellow solid (43.3 mg, 51% yield). PE/EA = 5:1, R_f = 0.17. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.00 (s, 1H), 7.97 (s, 1H), 7.79 (d, *J* = 10.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 2H), 7.49-7.43 (m, 3H), 7.35-7.29 (m, 2H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.09 (d, *J* = 7.3 Hz, 1H), 7.03-7.01 (m, 4H), 6.85 (d, *J* = 7.3 Hz, 2H), 6.54 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.4, 191.0, 147.9, 146.7, 145.5, 145.3, 144.7, 144.5, 135.8, 134.8, 134.0, 131.5, 130.6, 130.5, 129.5, 129.0, 128.9, 128.3, 128.2, 128.1, 128.0, 127.8, 127.3, 126.6, 122.2, 121.7, 71.9. IR (KBr): 3058, 2955, 2898, 1657, 1612, 1263, 1163, 1029, 826, 700, 687 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{20}O_2Na$ $[M+Na]^+$ 447.1361, found 447.1359.



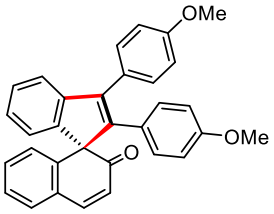
6'-((Tert-butyldimethylsilyl)oxy)-2,3-diphenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3x)

Yellow solid (68.2 mg, 80% yield). PE/EA = 10:1, R_f = 0.21. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.3 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 2H), 7.43 (d, *J* = 7.2 Hz, 1H), 7.37-7.33 (m, 2H), 7.20-7.24 (m, 2H), 7.10-7.01 (m, 6H), 6.98-6.91 (m, 3H), 6.80 (s, 1H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.4, 151.0, 147.6, 145.5, 144.9, 144.7, 136.8, 135.1, 134.4, 130.7, 130.1, 129.6, 129.2, 128.8, 128.3, 128.1, 128.0, 127.9, 127.8, 127.1, 126.5, 126.4, 121.9, 121.7, 116.6, 72.9, 55.8. IR (KBr): 3057, 3023, 2929, 1676, 1624, 1488, 1460, 1288, 1264, 1106, 757, 734, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}O_2Na$ $[M+Na]^+$ 449.1517, found 449.1519.



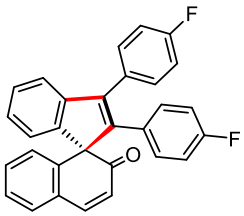
2,3-Di-p-tolyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3a')

Yellow solid (75.2 mg, 81% yield). PE/EA = 10:1, R_f = 0.23. The spectroscopic data is consistent with that reported in the literature[4].



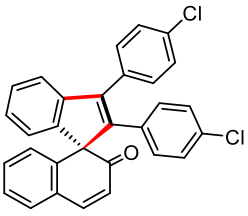
2,3-Bis(4-methoxyphenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3b')

Yellow solid (84.8 mg, 93% yield). PE/EA = 10:1, R_f = 0.19. The spectroscopic data is consistent with that reported in the literature[4].



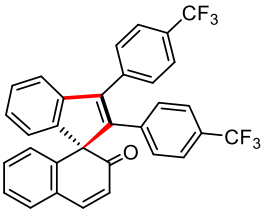
2,3-Bis(3-fluorophenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3c')

Yellow solid (74.3 mg, 86% yield). PE/EA = 20:1, R_f = 0.26. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 9.9 Hz, 1H), 7.45-7.39 (m, 2H), 7.31-7.25 (m, 4H), 7.22-7.16 (m, 2H), 7.10 (dd, *J* = 15.7, 8.0 Hz, 2H), 7.01 (d, *J* = 7.6 Hz, 1H), 6.97-6.94 (m, 1H), 6.91 (d, *J* = 7.8 Hz, 1H), 6.76-6.71 (m, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 6.53 (d, *J* = 10.7 Hz, 1H), 6.39 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.2, 163.2 (d, *J* = 246.9 Hz), 162.3 (d, *J* = 245.1 Hz), 147.6, 146.4, 144.6, 144.4, 140.4, 136.9 (d, *J* = 8.0 Hz), 136.3 (d, *J* = 8.0 Hz), 130.8, 130.7, 130.6, 130.2, 129.7, 129.6, 129.5, 128.0, 127.9, 126.9, 126.8, 126.5, 125.3 (d, *J* = 2.9 Hz), 124.9 (d, *J* = 2.8 Hz), 121.9, 121.8, 116.4 (d, *J* = 21.8 Hz), 115.7 (d, *J* = 22.6 Hz), 115.2 (d, *J* = 21.0 Hz), 114.3 (d, *J* = 21.2 Hz), 71.8. ^{19}F NMR (376 MHz, $CDCl_3$): δ -112.18 (s, 1F), -112.89 (s, 1F). IR (KBr): 3066, 2924, 2854, 1664, 1584, 1483, 1435, 1264, 1230, 920, 882, 788, 740, 700 cm^{-1}. HRMS (ESI) m/z calculated for $C_{30}H_{18}F_2ONa$ $[M+Na]^+$ 455.1223, found 455.1202.



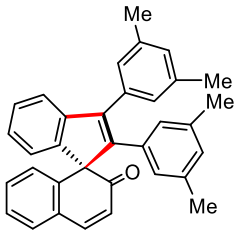
2,3-Bis(4-chlorophenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3d')

Yellow solid (84.5 mg, 91% yield). PE/EA = 20:1, R_f = 0.18. The spectroscopic data is consistent with that reported in the literature[9].



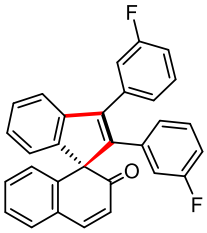
2,3-Bis(4-(trifluoromethyl)phenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3e')

Yellow solid (83.0 mg, 78% yield). PE/EA = 10:1, R_f = 0.21. The spectroscopic data is consistent with that reported in the literature[4].



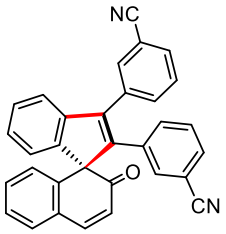
2,3-Bis(3,5-dimethylphenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3f')

Brown powder (70.5 mg, 78% yield). PE/EA = 20:1, R_f = 0.21. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 9.9 Hz, 1H), 7.35 (d, *J* = 7.4 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.20-7.15 (m, 4H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.00-6.93 (m, 4H), 6.60 (s, 1H), 6.48 (s, 2H), 6.38 (d, *J* = 9.9 Hz, 1H), 2.31 (s, 6H), 1.95 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 147.6, 146.1, 146.0, 144.5, 144.8, 141.7, 138.3, 137.0, 135.4, 134.1, 130.8, 129.9, 129.6, 129.5, 128.8, 127.8, 127.5, 127.1, 127.0, 126.8, 126.6, 126.2, 121.9, 121.5, 71.6, 21.4, 21.3. IR (KBr): 2918, 1665, 1598, 1458, 1393, 1233, 1199, 1233, 1038, 873, 844, 744, 602 cm^{-1}. HRMS (ESI) m/z calculated for $C_{34}H_{29}O$ $[M+H]^+$ 453.2218, found 453.2227.



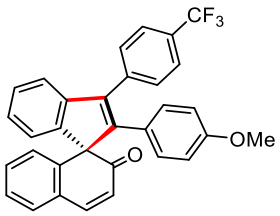
2,3-Bis(3-fluorophenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3g')

Yellow solid (62.2 mg, 72% yield). PE/EA = 10:1, R_f = 0.24. The spectroscopic data is consistent with that reported in the literature[4].

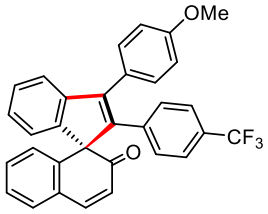


3,3'-(2'-Oxo-2'H-spiro[indene-1,1'-naphthalene]-2,3-diyl)dibenzonitrile (3h')

Brown solid (50.0 mg, 56% yield). PE/EA = 10:1, R_f = 0.25. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.78 (m, 1H), 7.72 (dd, *J* = 7.8, 1.6 Hz, 2H), 7.69 (d, *J* = 10.0 Hz, 1H), 7.60-7.56 (m, 1H), 7.47 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.37-7.29 (m, 3H), 7.24-7.20 (m, 2H), 7.17-7.12 (m, 2H), 7.06-7.03 (m, 3H), 6.87 (d, *J* = 7.7 Hz, 1H), 6.38 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 147.7, 146.6, 144.5, 144.4, 143.6, 139.2, 135.6, 135.3, 133.9, 133.4, 132.8, 132.2, 132.4, 131.2, 130.9, 130.5, 130.1, 129.8, 129.2, 128.4, 128.3, 127.6, 126.8, 126.4, 122.2, 121.8, 118.3, 113.5, 112.6, 72.1. IR (KBr): 3061, 2984, 2960, 2921, 2851, 2307, 2231, 1663, 1267, 1202, 1169, 803, 699, 647 cm^{-1}. HRMS (ESI) m/z calculated for $C_{32}H_{18}N_2ONa$ $[M+Na]^+$ 469.1317, found 469.1314.



3i'-1



3i'-2

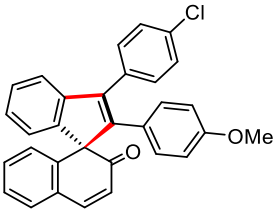
Compounds **3i'** was isolated as a regioisomeric mixture of **3i'-1** and **3i'-2** (rr = 1:1) in 95% yield (93.9 mg) by silica gel column chromatography with Petroleum ether/EtOAc (10/1).

2-(4-Methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3i'-1)

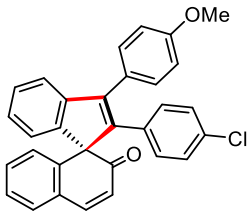
The spectroscopic data is consistent with that reported in the literature[4].

3-(4-Methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3i'-2)

The spectroscopic data is consistent with that reported in the literature[4].



3j'-1



3j'-2

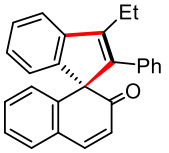
Compounds **3j'** was isolated as a regioisomeric mixture of **3j'-1** and **3j'-2** (rr = 1:1) in 92 yield (84.6 mg) by silica gel column chromatography with Petroleum ether/EtOAc (20/1). The pure analytical samples of **3j'-1** and **3j'-2** were obtained by preparative TLC on silica gel.

3-(4-Chlorophenyl)-2-(4-methoxyphenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3j'-1)

Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 9.9 Hz, 1H), 7.45 (dd, *J* = 18.3, 7.6 Hz, 5H), 7.29-7.25 (m, 1H), 7.22 (d, *J* = 3.8 Hz, 2H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.05-7.01 (m, 1H), 6.98 (d, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 7.7 Hz, 1H), 6.75 (d, *J* = 8.7 Hz, 2H), 6.54 (d, *J* = 8.7 Hz, 2H), 6.38 (d, *J* = 9.9 Hz, 1H), 3.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 158.6, 147.4, 146.2, 145.4, 145.2, 141.9, 141.1, 133.9, 133.6, 131.1, 130.7, 130.3, 129.9, 129.6, 129.2, 127.8, 127.6, 126.9, 126.5, 126.2, 121.7, 121.3, 113.6, 71.7, 55.0. IR (KBr): 3060, 2958, 2929, 2839, 1662, 1611, 1254, 1179, 824, 746 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}ClO_2$ $[M+H]^+$ 461.1308, found 461.1306.

2-(4-Chlorophenyl)-3-(4-methoxyphenyl)-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3j'-2)

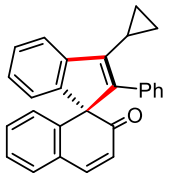
Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 9.9 Hz, 1H), 7.46 (d, *J* = 8.3 Hz, 3H), 7.34-7.30 (m, 2H), 7.28-7.25 (m, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 7.00 (t, *J* = 8.7 Hz, 5H), 6.94 (d, *J* = 7.8 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 2H), 6.41 (d, *J* = 9.9 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 159.1, 147.6, 146.3, 145.3, 144.9, 143.2, 140.9, 133.2, 132.7, 130.7, 130.6, 130.3, 130.0, 129.7, 128.2, 127.8, 127.7, 126.9, 126.6, 126.5, 122.0, 121.7, 114.4, 71.6, 55.3. IR (KBr): 3061, 2958, 2929, 2831, 1662, 1611, 1254, 1179, 822, 743 cm^{-1}. HRMS (ESI) m/z calculated for $C_{31}H_{22}ClO_2$ $[M+H]^+$ 461.1308, found 461.1303.



Compounds **3k'** was isolated as a regioisomeric mixture (rr = 10:1) in 84% yield (58.5 mg) by silica gel column chromatography with Petroleum ether/EtOAc (20/1). The pure analytical sample of the major regioisomer of **3k'** was obtained by preparative TLC on silica gel.

3-Ethyl-2-phenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3k')

Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 9.9 Hz, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.34-7.29 (m, 2H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.17-7.13 (m, 4H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.98-6.93 (m, 3H), 6.82 (d, *J* = 7.7 Hz, 1H), 6.28 (d, *J* = 9.9 Hz, 1H), 2.81-2.71 (m, 2H), 1.36 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 148.5, 146.1, 145.8, 145.6, 144.6, 140.8, 135.2, 130.3, 130.1, 129.7, 128.6, 128.1, 127.7, 127.5, 127.1, 127.2, 126.7, 126.1, 121.9, 120.7, 72.1, 19.7, 13.8. IR (KBr): 3059, 2968, 2928, 2871, 1662, 1619, 1461, 1235, 867, 820, 755, 698 cm^{-1}. HRMS (ESI) m/z calculated for $C_{26}H_{20}ONa$ $[M+Na]^+$ 371.1412, found 371.1414.

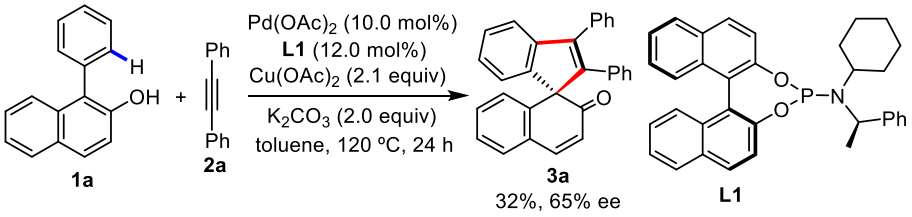


Compounds **3l'** was isolated as a regioisomeric mixture (rr = 8:1) in 66% yield (47.5 mg) by silica gel column chromatography with Petroleum ether/EtOAc (10/1).

3-Cyclopropyl-2-phenyl-2'H-spiro[indene-1,1'-naphthalen]-2'-one (3l')

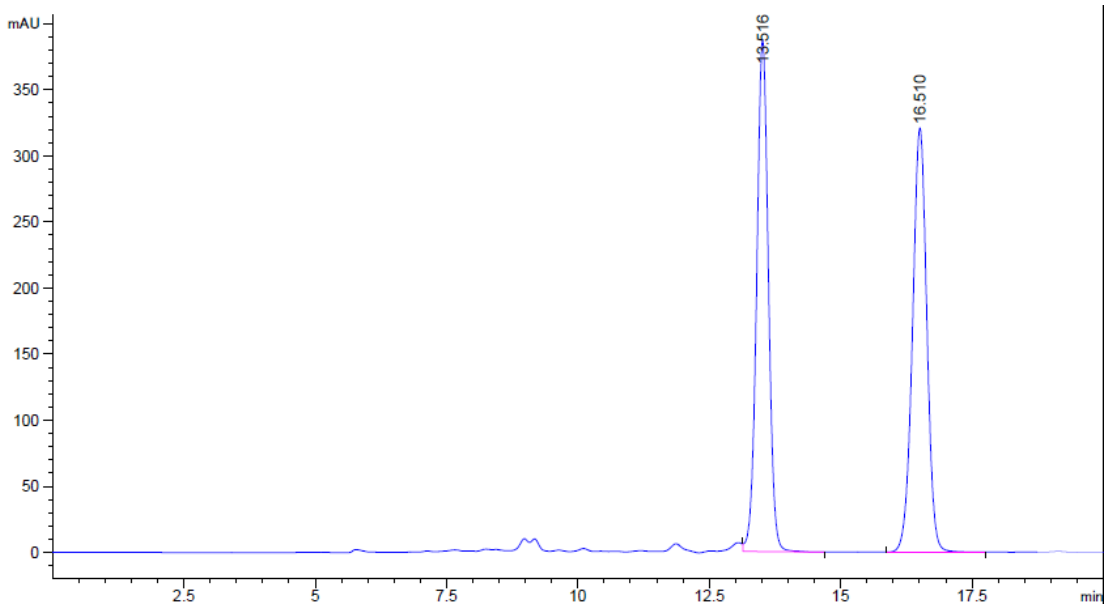
Yellow solid. The spectroscopic data is consistent with that reported in the literature[4].

D. Preliminary asymmetric studies:

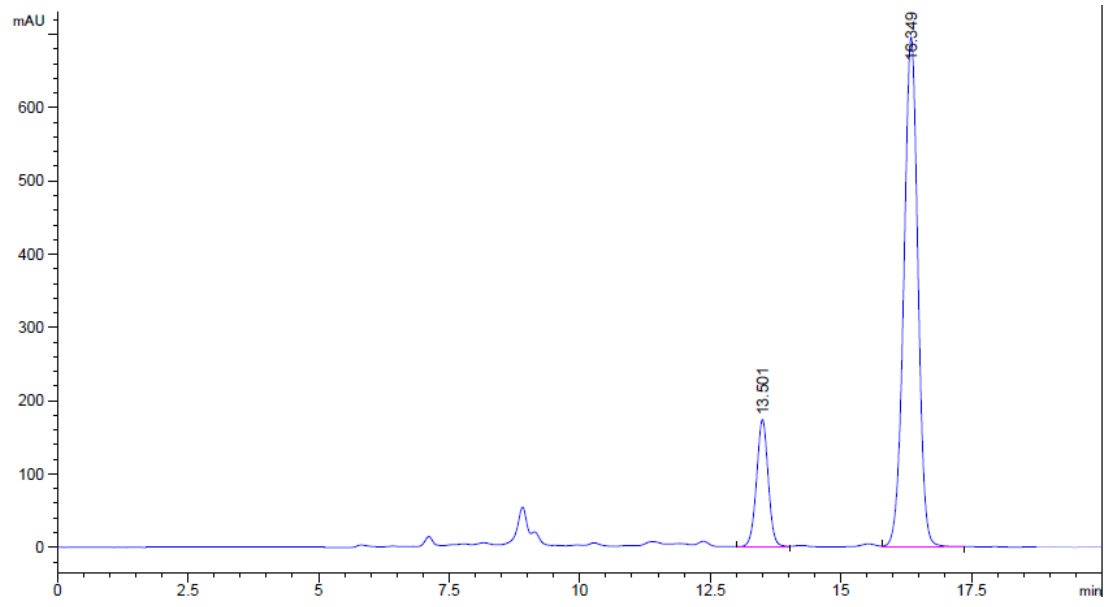


In a glovebox, a 5.0 mL vial equipped with a stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), phosphoramidite ligand **L1** (6.2 mg, 0.012 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and toluene (2.0 mL) was then added. After the catalyst mixture was stirred at room temperature for 30 minutes, substrate $Cu(OAc)_2$ (38 mg, 0.21 mmol), **1a** (22.0 mg, 0.1 mmol) and **2a** (35.6 mg, 0.2 mmol) were sequentially added. The vial was sealed with a Teflon screw cap and the reaction mixture was heated at 120 °C for 24 h. The crude reaction mixture was then subjected to a silica gel column to afford the desired product **3a**. 32% yield. 65% ee, $[\alpha]_D^{20}$ = -48 (c = 1.0, $CHCl_3$). The

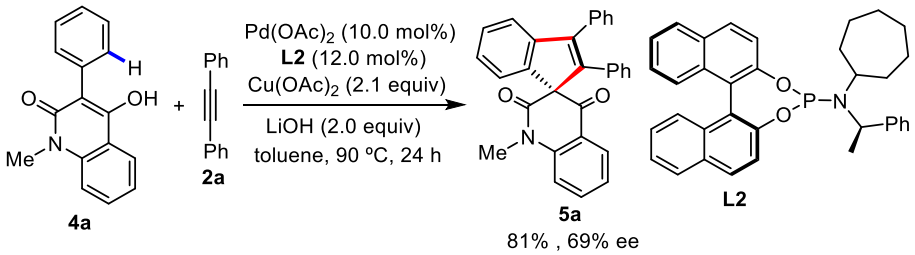
ee of compound **3a** was determined by HPLC using an IA column (Wavelength = 250 nm, *n*-hexane/*i*-PrOH = 85/15, flow rate = 0.5 mL/min, t_{major} = 16.3 min, t_{minor} = 13.5 min).



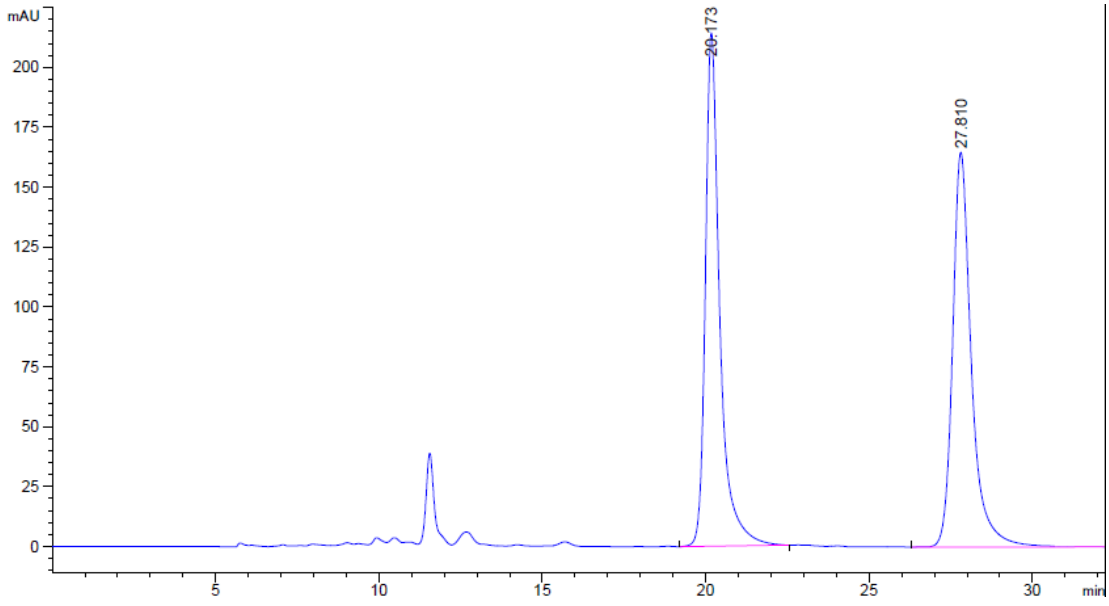
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.516	VB	0.2392	6012.60107	387.14816	49.7061
2	16.510	BB	0.2912	6083.69727	320.93021	50.2939



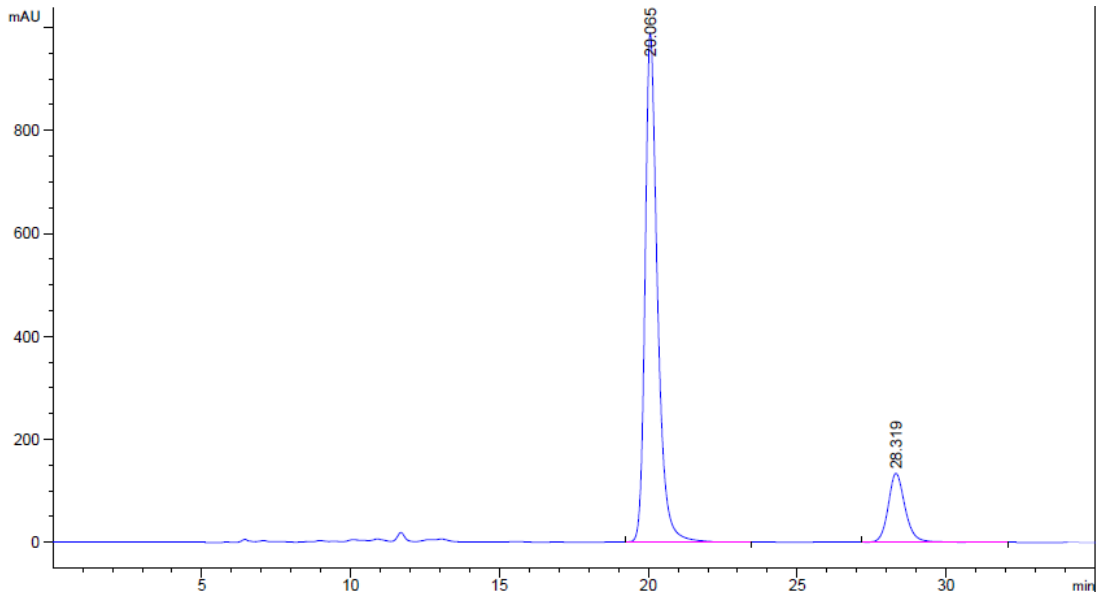
#	[min]		[min]	[mAU*s]	[mAU]	%
1	13.501	BV	0.2392	2696.22021	173.60493	17.1083
2	16.349	VB	0.2901	1.30635e4	695.45734	82.8917



In a glovebox, a 5.0 mL vial equipped with a stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), phosphoramidite ligand **L2** (6.4 mg, 0.012 mmol), LiOH (4.8 mg, 0.2 mmol) and toluene (1.0 mL) was then added. After the catalyst mixture was stirred at room temperature for 30 minutes, substrate $Cu(OAc)_2$ (38 mg, 0.21 mmol), **4a** (25.1 mg, 0.1 mmol) and **2a** (35.6 mg, 0.2 mmol) were sequentially added. The vial was sealed with a Teflon screw cap and the reaction mixture was heated at 90 °C for 24 h. The crude reaction mixture was then subjected to a silica gel column to afford the desired product **5a**[11] in 81% yield and 69% ee, $[\alpha]_D^{20}$ = +38.9 (c = 1.0, $CHCl_3$). The ee of compound **5a** was determined by HPLC using an IA column (Wavelength = 250 nm ,*n*-hexane/*i*-PrOH = 85/15, flow rate = 0.5 mL/min, t_{major} = 20.1 min, t_{minor} = 28.3 min).



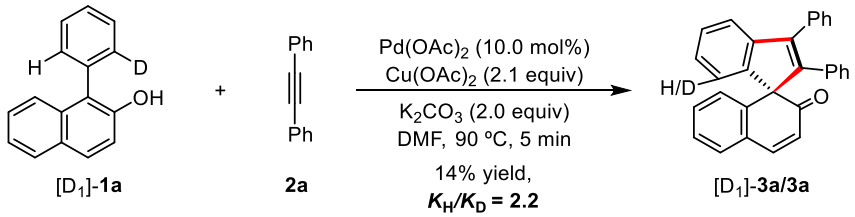
#	[min]		[min]	[mAU*s]	[mAU]	%
1	20.173	BB	0.4546	6585.68311	214.27861	49.9667
2	27.810	BBA	0.5950	6594.47168	164.75954	50.0333



#	[min]		[min]	[mAU*s]	[mAU]	%
1	20.065	BB	0.4350	2.86376e4	988.53662	84.4264
2	28.319	BB	0.5962	5282.58594	133.91763	15.5736

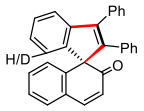
E. Deuterium-labeling experiments:

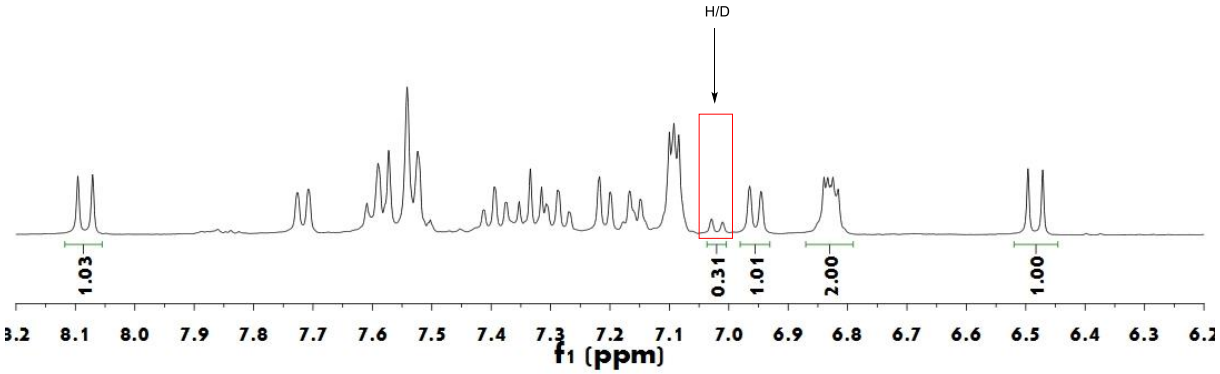
(a) Intramolecular kinetic isotope effect experiment



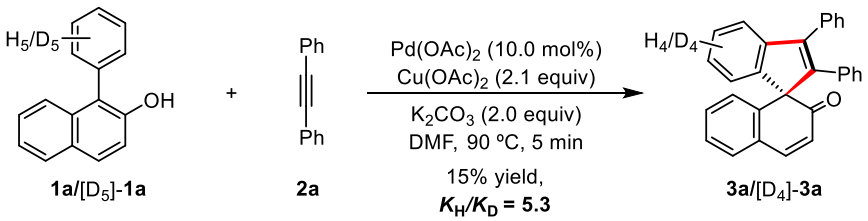
In a glovebox, a 5.0 mL vial equipped with a stir bar was charged with $Pd(OAc)_2$ (4.4 mg, 0.02 mmol), $Cu(OAc)_2$ (76 mg, 0.42 mmol), [D_1]-**1a** (44.2 mg, 0.20 mmol), K_2CO_3 (55.2 mg, 0.40 mmol) and **2a** (71.2 mg 0.40 mmol) followed by sequential addition of DMF (2.0 mL). The vial was sealed with a Teflon screw cap and then the reaction mixture was heated at 90 °C for 5 minutes. The crude product was then subjected to a silica gel column to afford a mixture of [D1]-**3a/3a** in 14% yield. The D/H incorporation in [D1]-**3a/3a** was determined by ^{1}H NMR spectroscopy. The kinetic isotopic effect of this reaction was determined to be k_H/k_D = 2.2.

n(D) = 0.31
n(H) = 1-0.31 = 0.69
KIE = n(H)/n(D) = 2.2





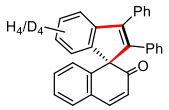
(b) Intermolecular kinetic isotope effect experiment

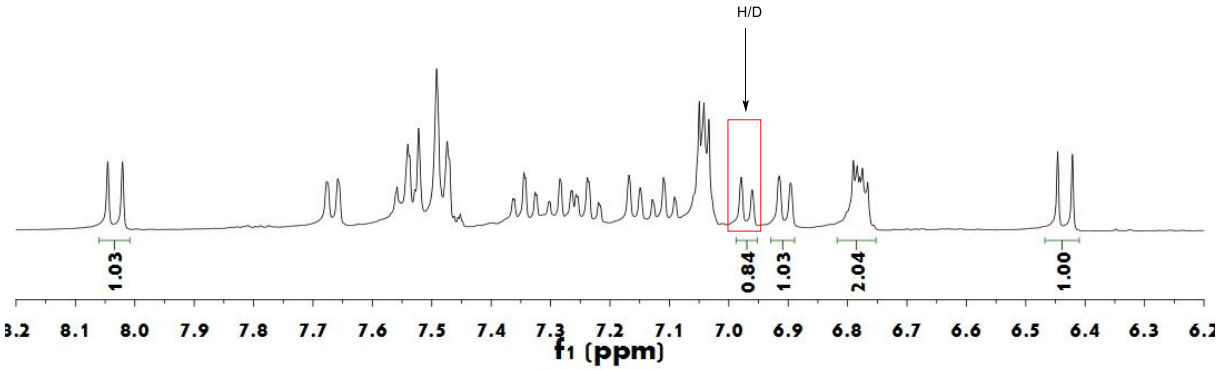


1a/[D_5]-**1a** **2a** **3a**/[D_4]-**3a**

In a glovebox, a 5.0 mL vial equipped with a stir bar was charged with $Pd(OAc)_2$ (4.4 mg, 0.02 mmol), $Cu(OAc)_2$ (76 mg, 0.42 mmol), [D_5]-**1a** (24.0 mg, 94% deuterated, 0.11 mmol) **+ 1a** (20.6 mg, 0.19 mmol), K_2CO_3 (55.2 mg, 0.40 mmol) and **2a** (71.2 mg, 0.40 mmol) followed by sequential addition of DMF (2.0 mL). The vial was sealed with a Teflon screw cap and then the reaction mixture was heated at 90 °C for 5 minutes. The crude product was then subjected to a silica gel column to afford a mixture of **3a/**[D_4]-**3a** in 15% yield. The D/H incorporation in **3a/**[D_4]-**3a** was determined by ^{1}H NMR spectroscopy. The kinetic isotopic effect of this reaction was determined to be k_H/k_D = 5.3.

n(H) = 0.84
n(D) = 1-0.84 = 0.16
KIE = n(H)/n(D) = 5.3





F. References

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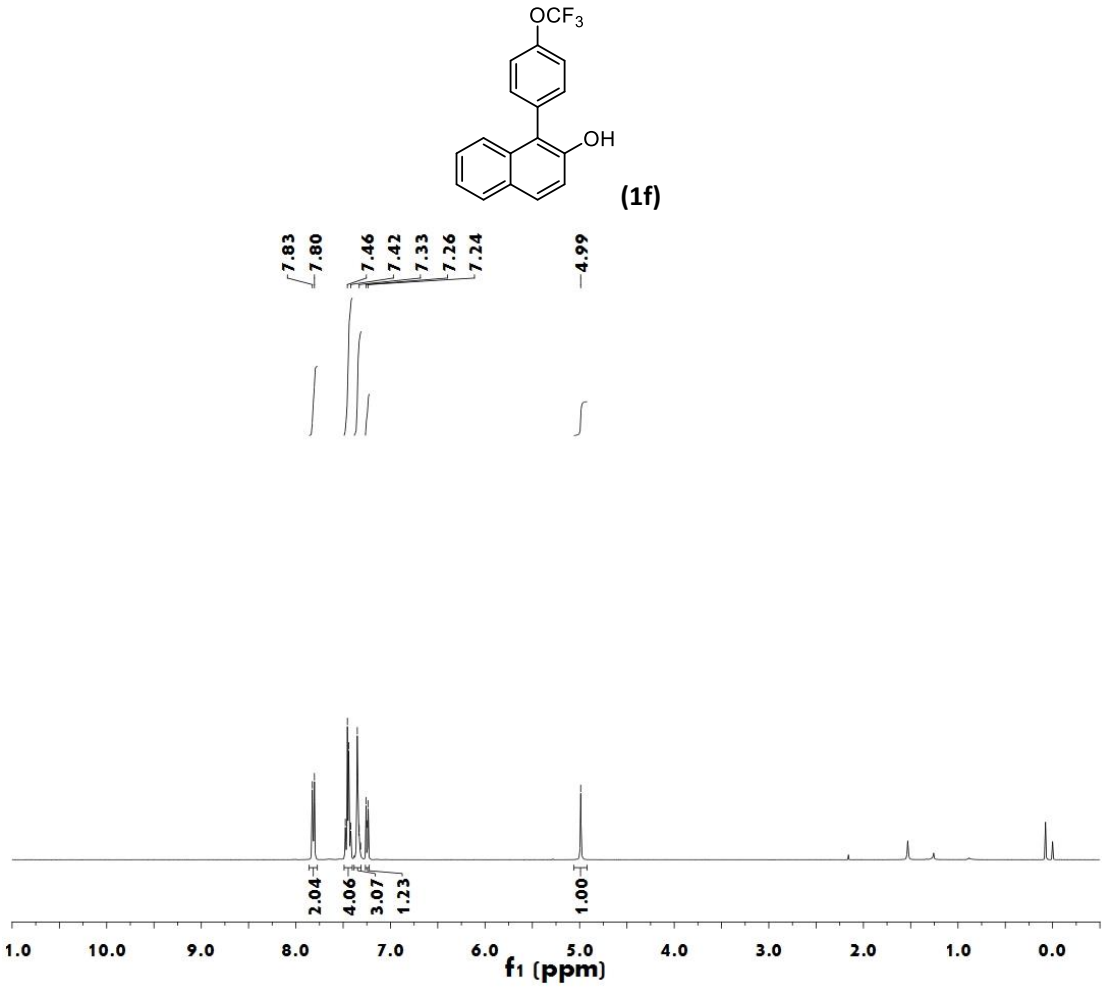
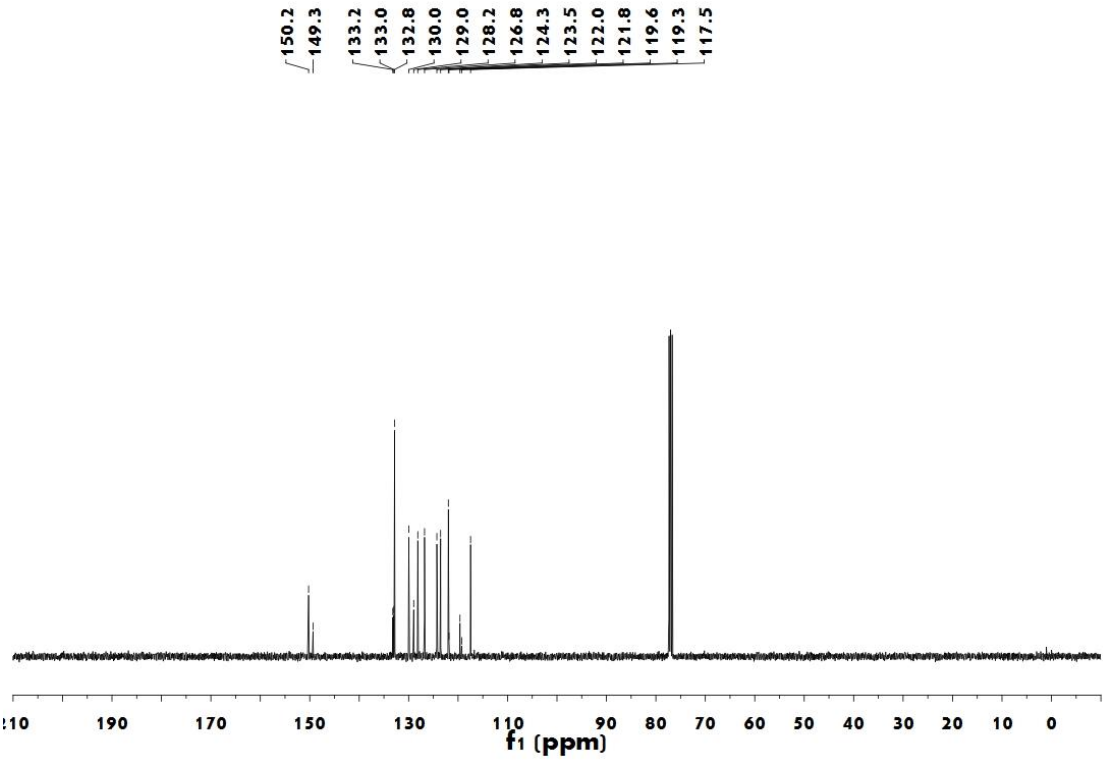
G. NMR spectra:

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124.3
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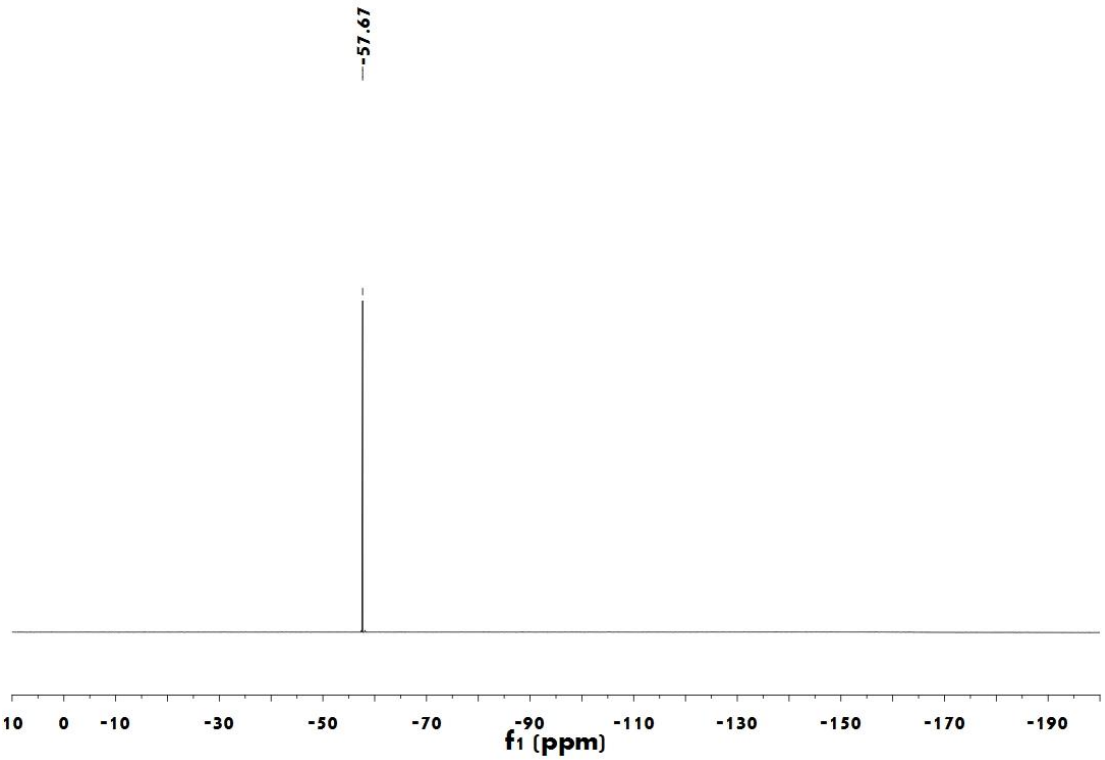
121.8
119.6
119.3
117.5
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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10
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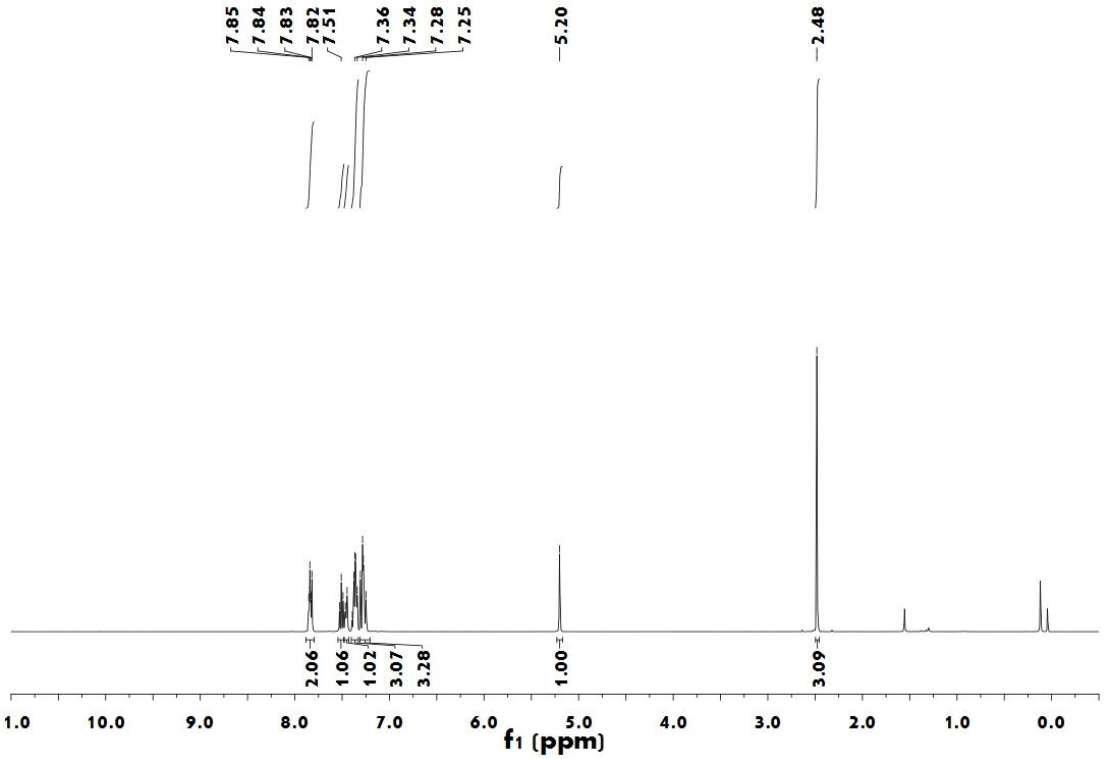
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
f1 (ppm)


Me
OH

(1i)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7.36
7.34
7.28
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5.20
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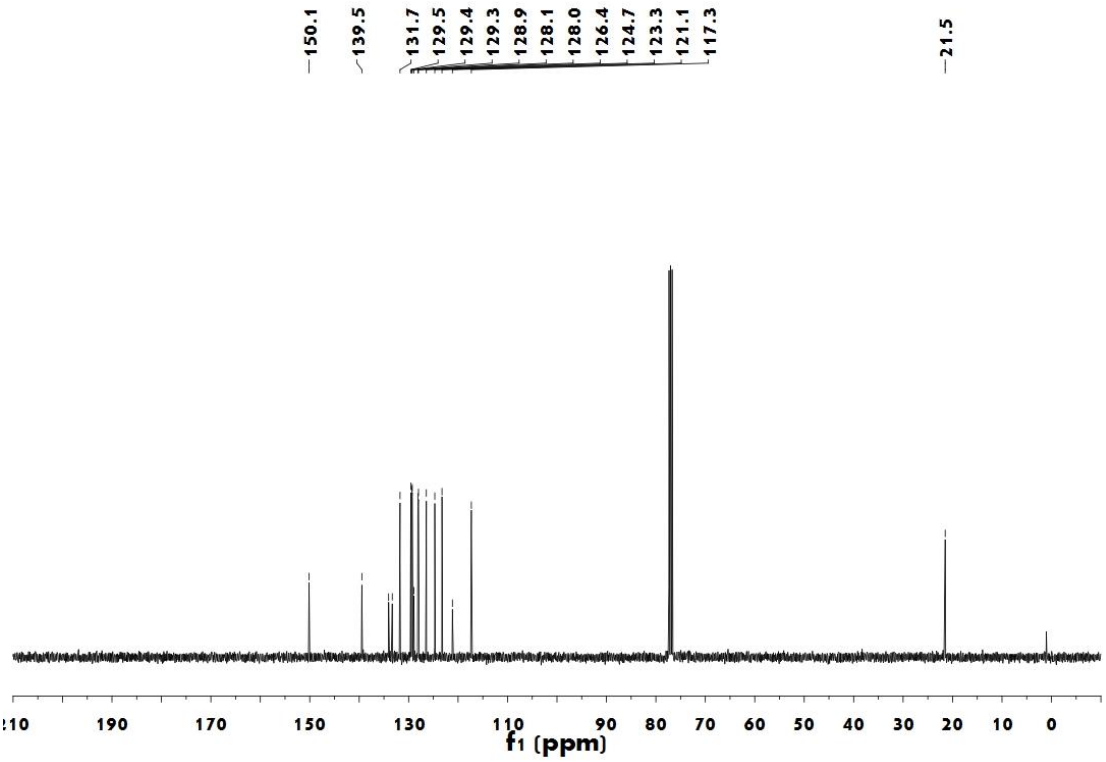
3.07
3.28
1.00
3.09
f1 (ppm)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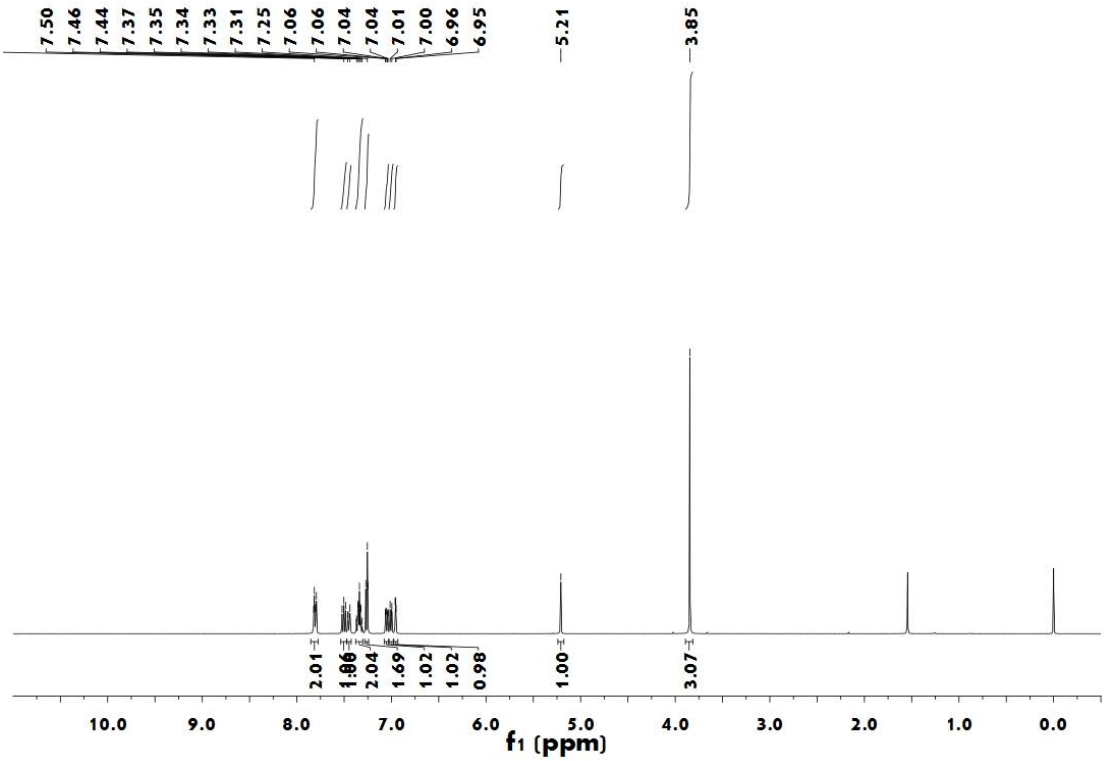
121.1
117.3
21.5
f1 (ppm)


MeO
OH

(1j)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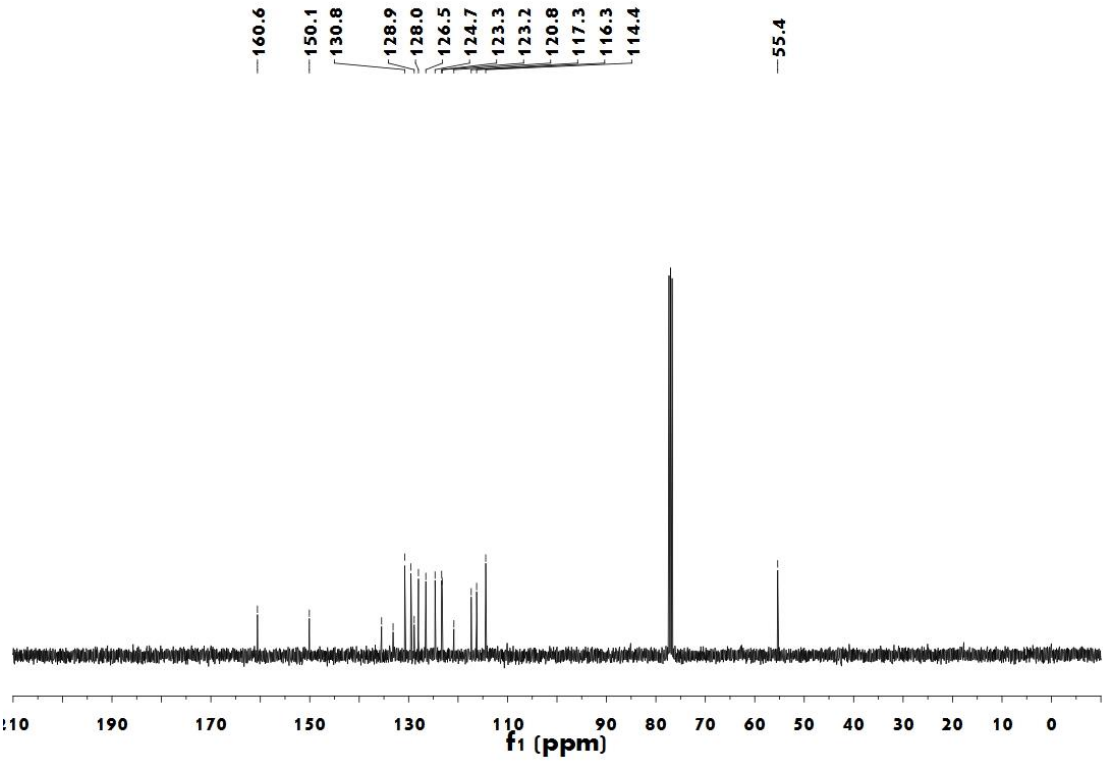
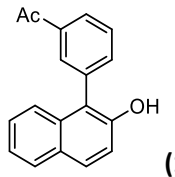
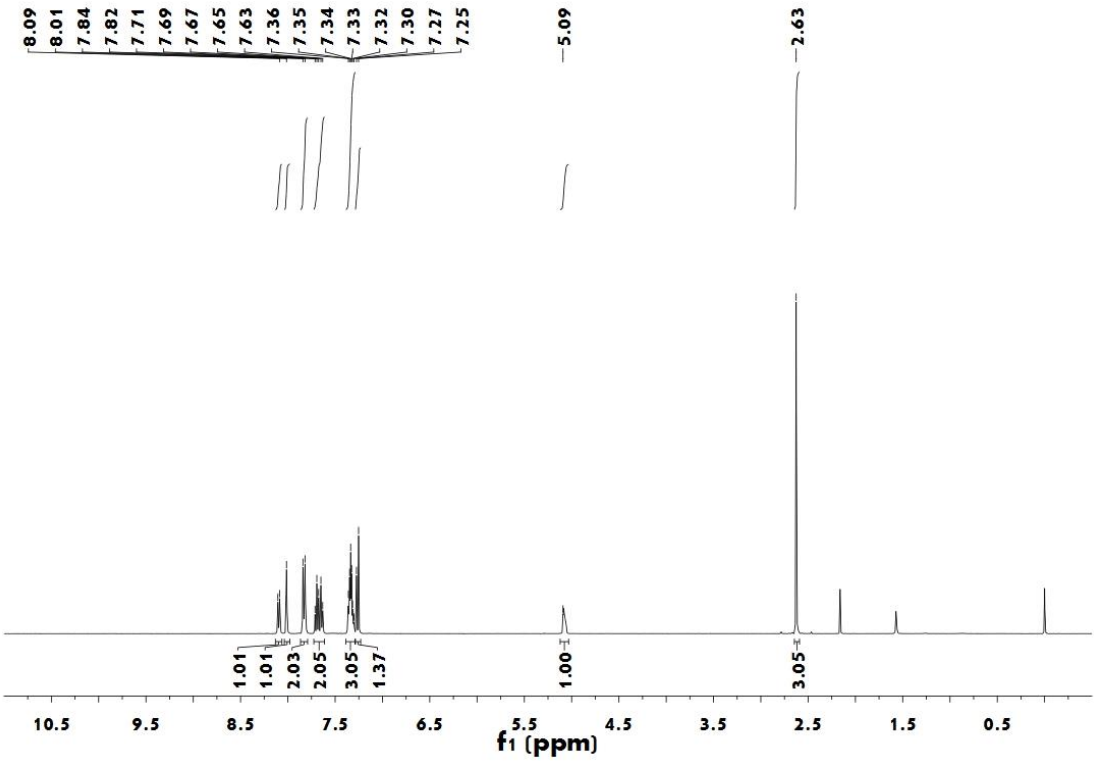
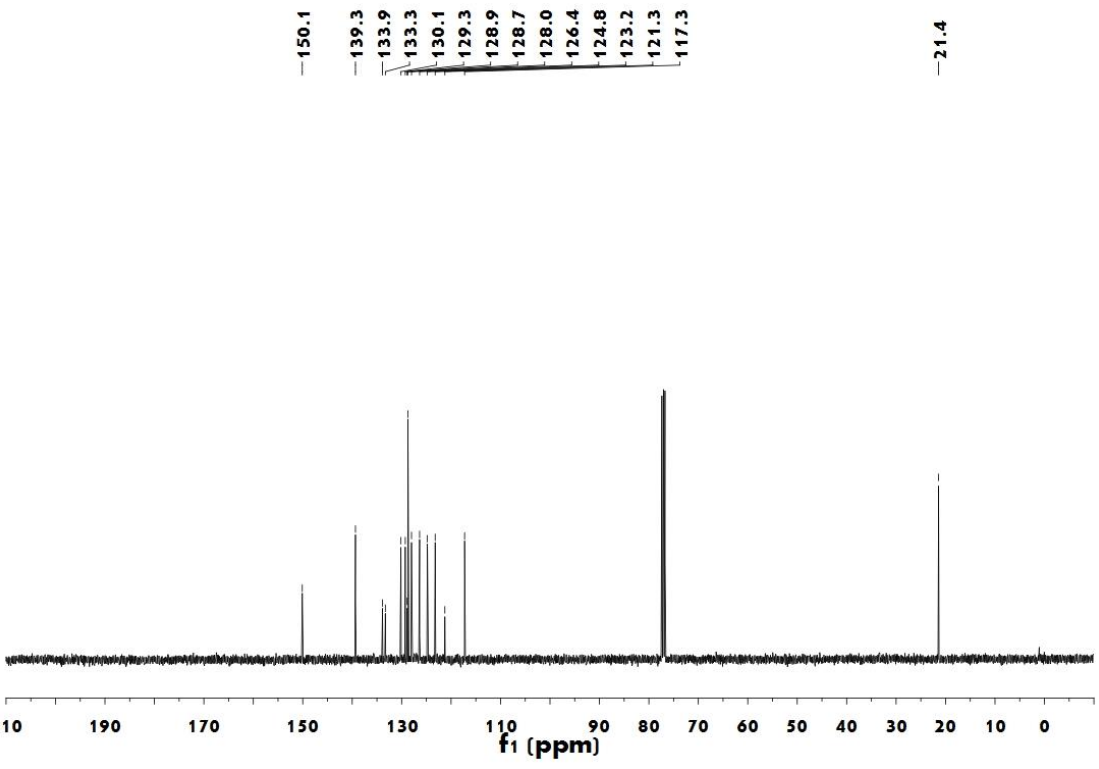
7.34
7.33
7.31
7.25
7.06
7.06
7.04
7.04
7.01
7.00
6.96
6.95
5.21
3.85
2.01
2.04
1.69
1.02
1.02
0.98
1.00
3.07
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

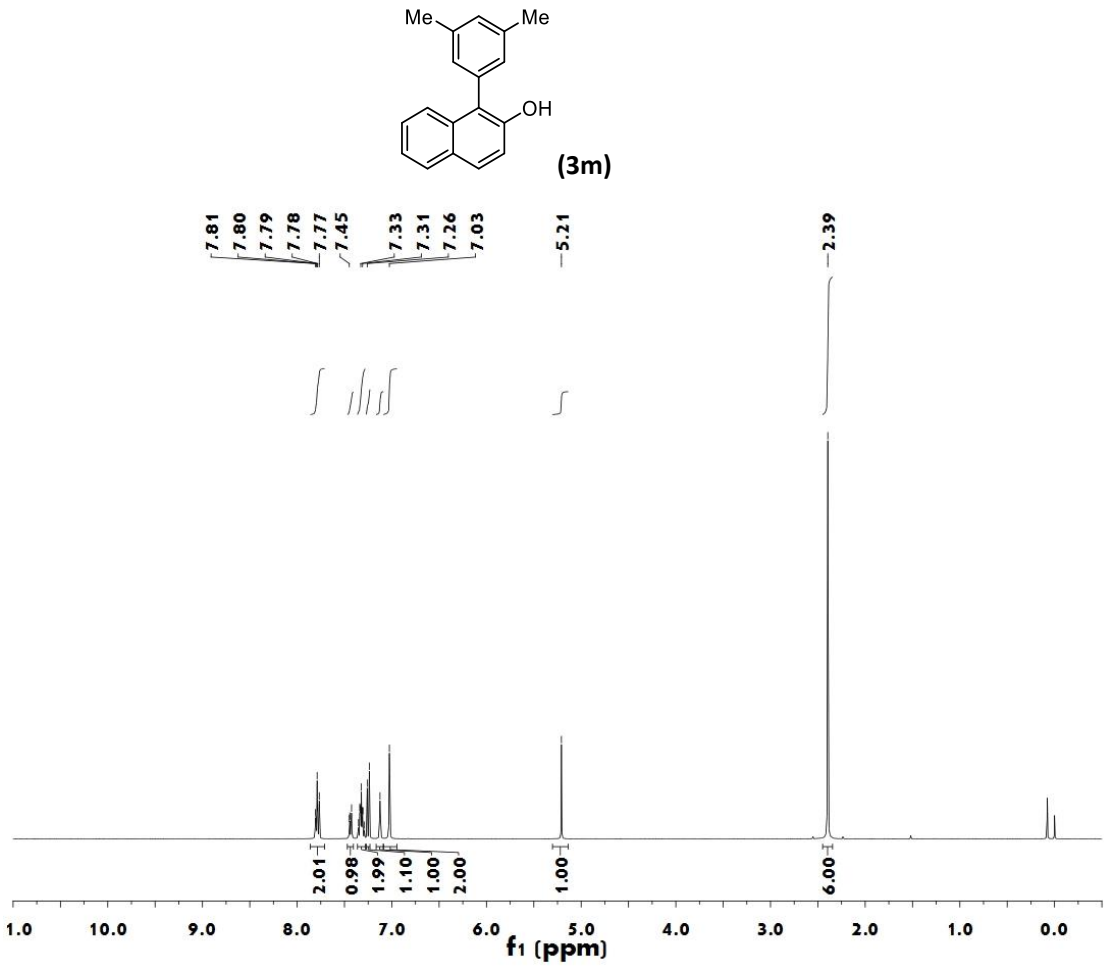

160.6
150.1
130.8
128.9
128.0
126.5
124.7
123.3
123.2
120.8
117.3
116.3
114.4
55.4
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

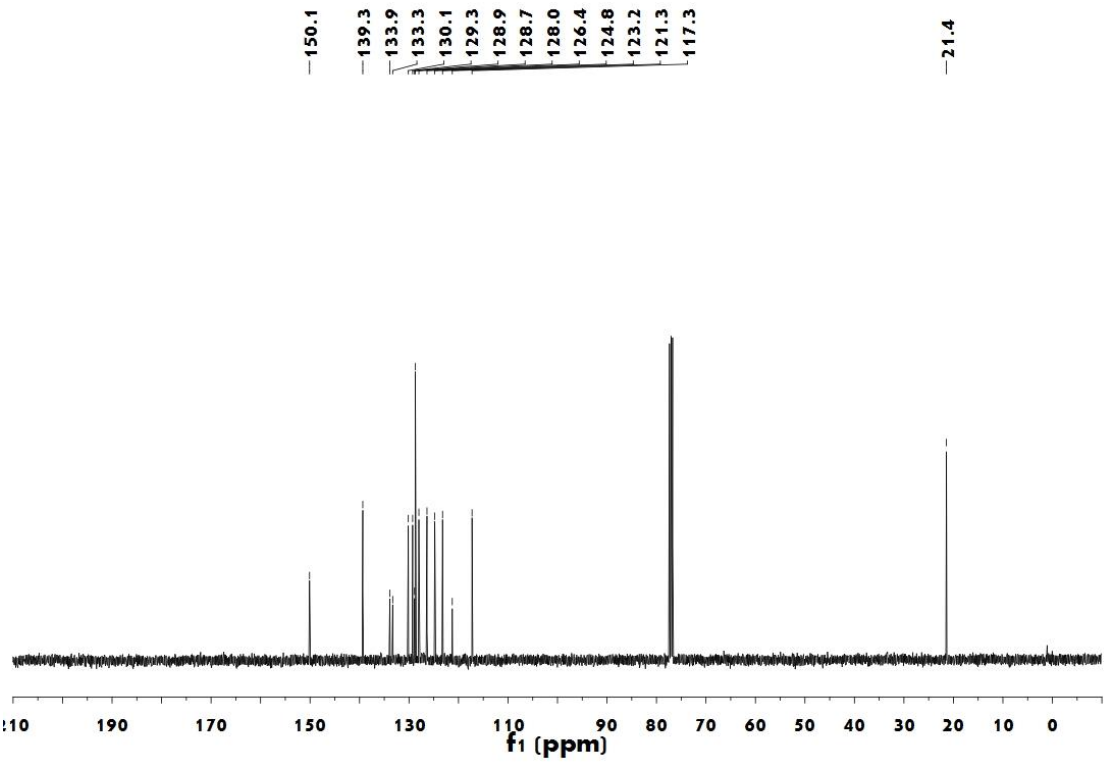

Ac
OH

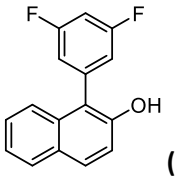
(1k)


8.09
8.01
7.84
7.82
7.71
7.69
7.67
7.65
7.63
7.36
7.35
7.34
7.33
7.32
7.30
7.27
7.25
5.09
2.63
1.01
1.01
2.03
2.05
3.05
1.37
1.00
3.05
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


150.1
139.3
133.9
133.3
130.1
129.3
128.9
128.7
128.0
126.4
124.8
123.2
121.3
117.3
21.4
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

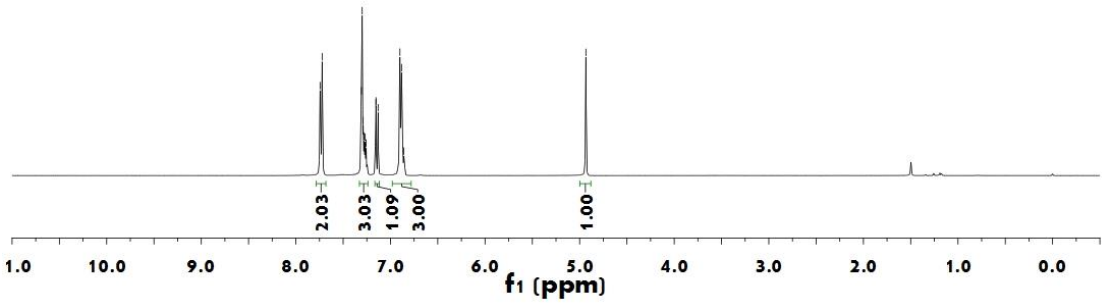

Me
Me
OH
(3m)
7.81
7.80
7.79
7.78
7.77
7.45
7.33
7.31
7.26
7.03
5.21
2.39
2.01
0.98
1.99
1.10
1.00
2.00
1.00
6.00
f1 (ppm)

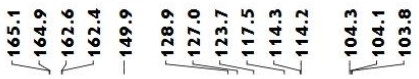

150.1
139.3
133.9
133.3
130.1
129.3
128.9
128.7
128.0
126.4
124.8
123.2
121.3
117.3
21.4
f1 (ppm)

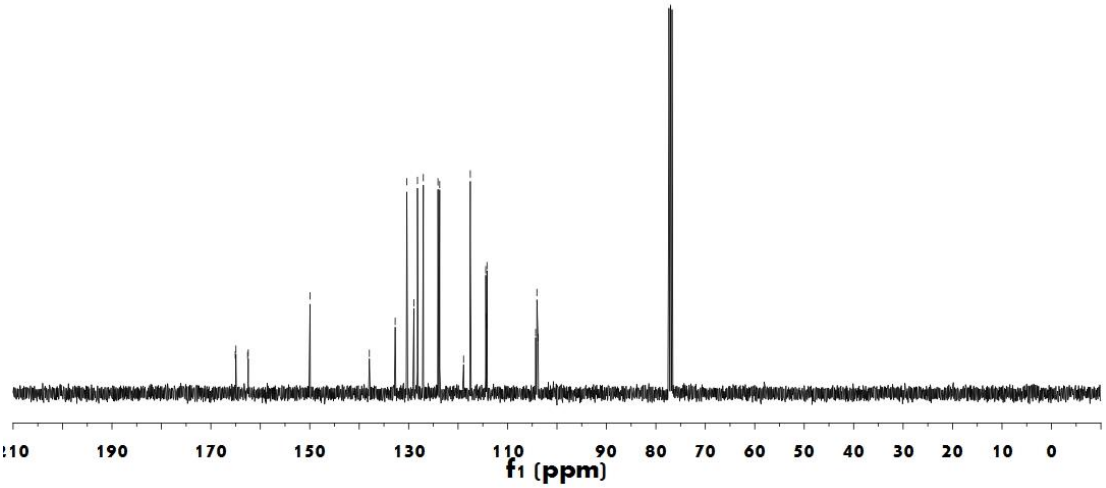

F
F
OH

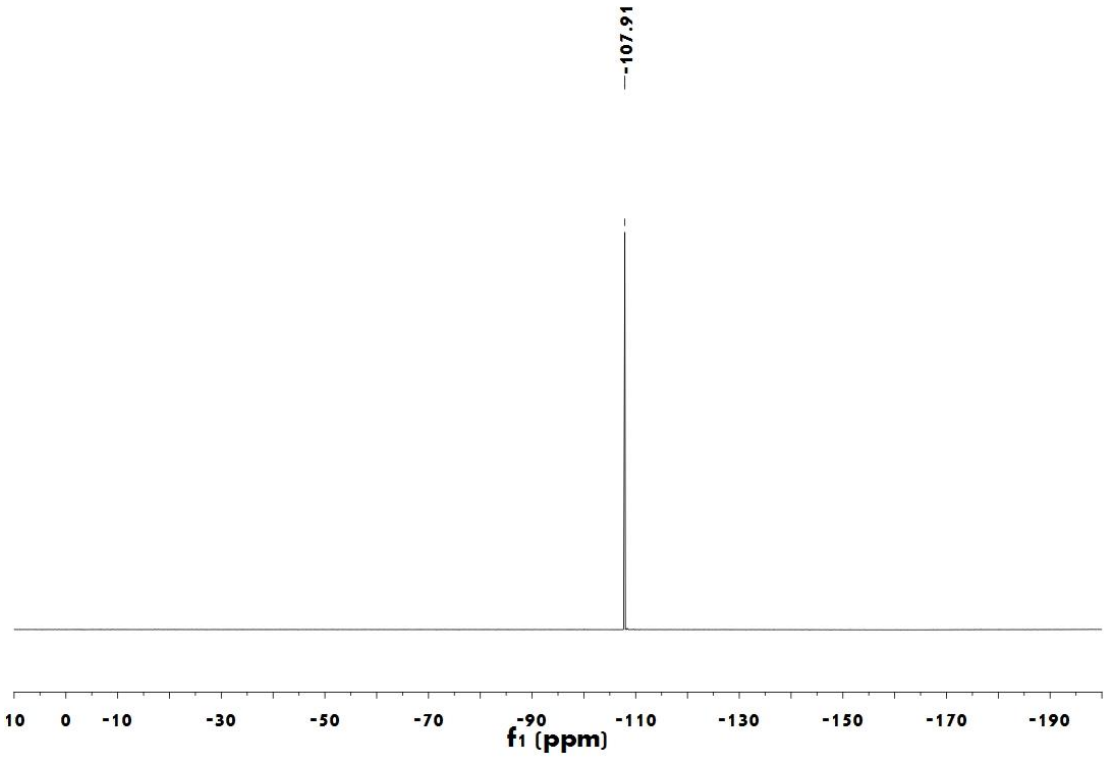
(1n)

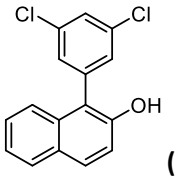

7.74
7.72
7.30
7.26
7.15
6.90
6.86
4.93


2.03
3.03
1.09
3.00
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

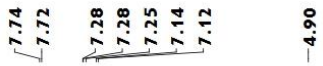

165.1
164.9
162.6
162.4
149.9
128.9
127.0
123.7
117.5
114.3
114.2
104.3
104.1
103.8

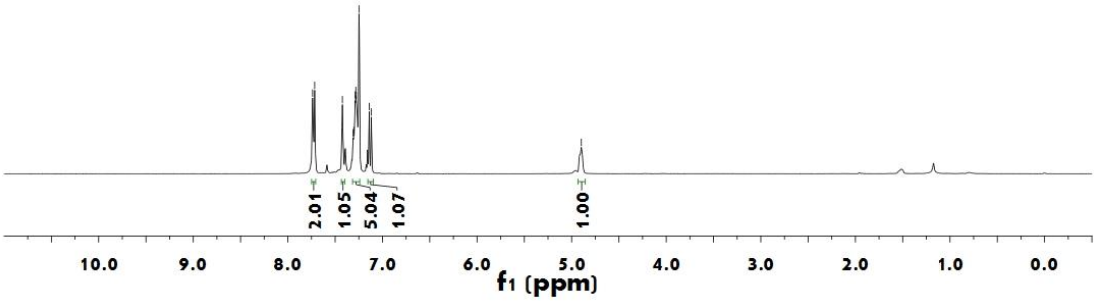

210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

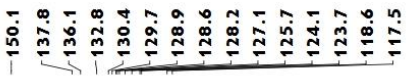

-107.91
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
f1 (ppm)

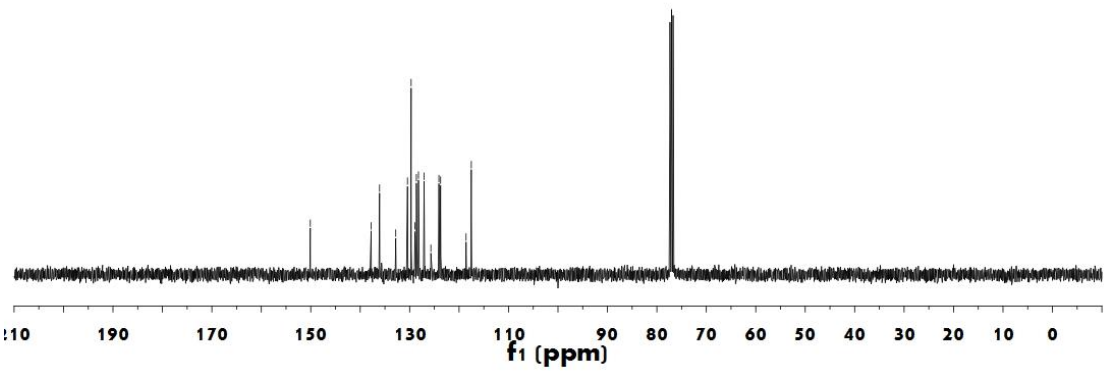

Cl
Cl
OH

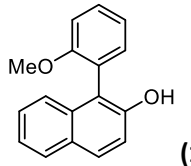
(1o)


7.74
7.72
7.28
7.28
7.25
7.14
7.12
4.90

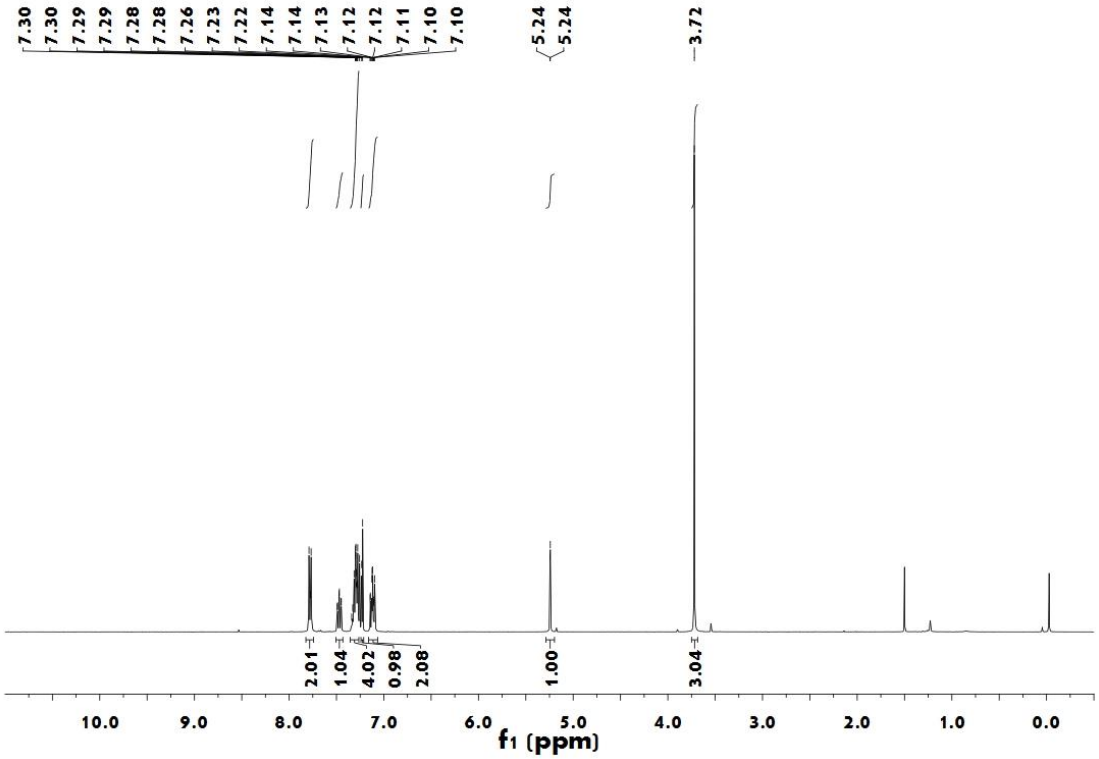

2.01
1.05
5.04
1.07
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

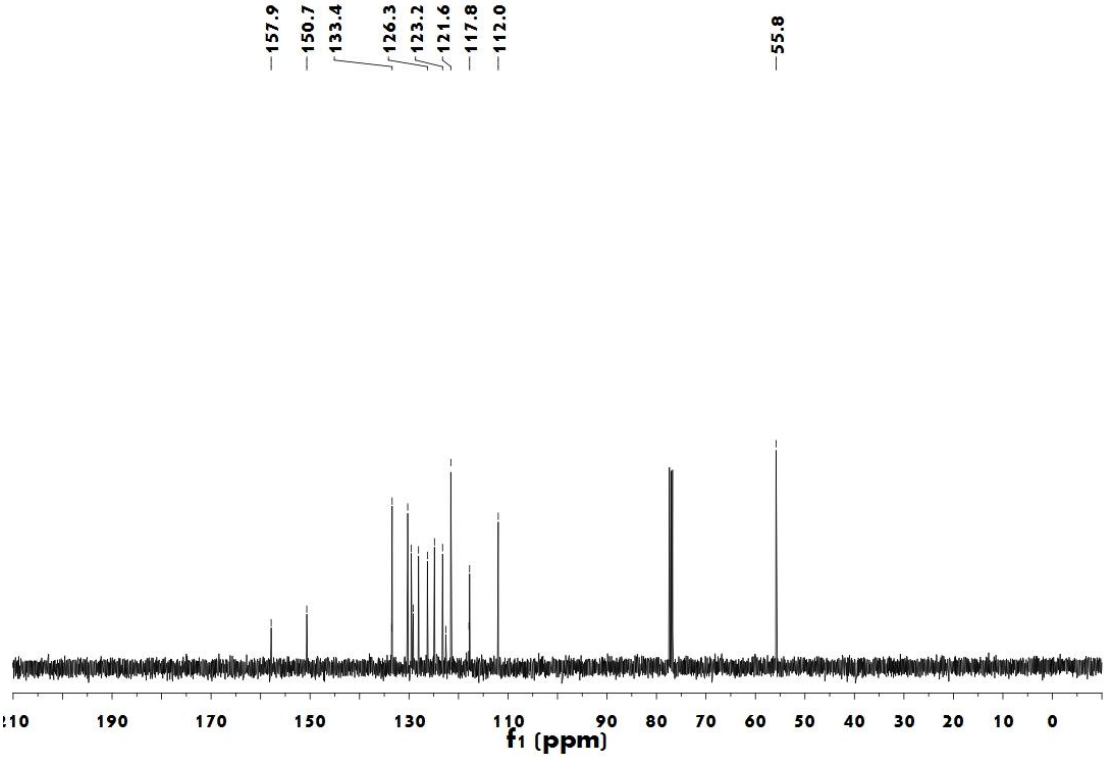

150.1
137.8
136.1
132.8
130.4
129.7
128.9
128.6
128.2
127.1
125.7
124.1
123.7
118.6
117.5


210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MeO
OH

(1p)

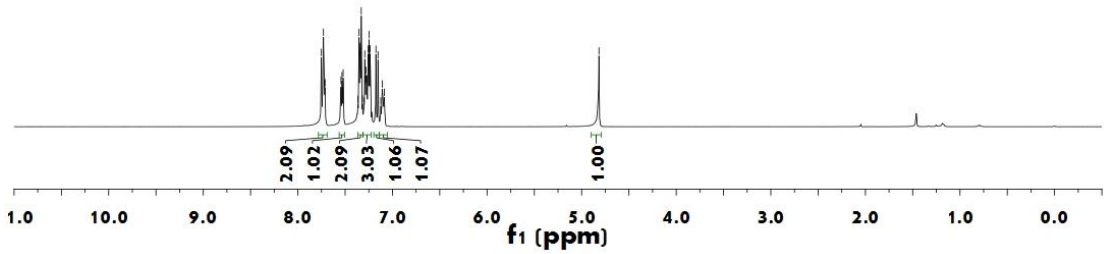

7.30
7.30
7.29
7.29
7.28
7.28
7.26
7.23
7.22
7.14
7.14
7.13
7.12
7.12
7.11
7.10
7.10
5.24
5.24
3.72
2.01
1.04
4.02
0.98
2.08
1.00
3.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

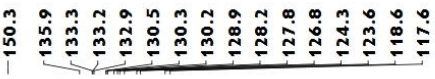

157.9
150.7
133.4
126.3
123.2
121.6
117.8
112.0
55.8
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

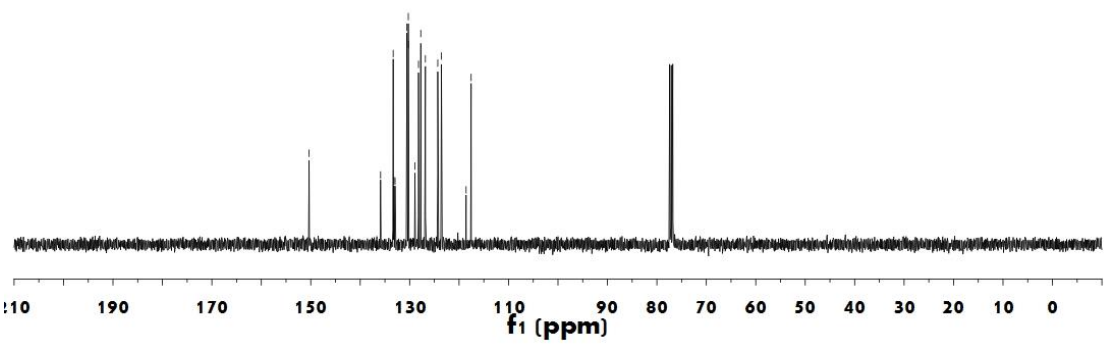

Cl
OH

(1r)


7.34
7.34
7.33
7.32
7.30
7.29
7.29
7.28
7.27
7.26
7.25
7.24
7.17
7.15
7.12
7.11
7.09
4.82


2.09
1.02
2.09
3.03
1.06
1.07
1.00
f1 (ppm)

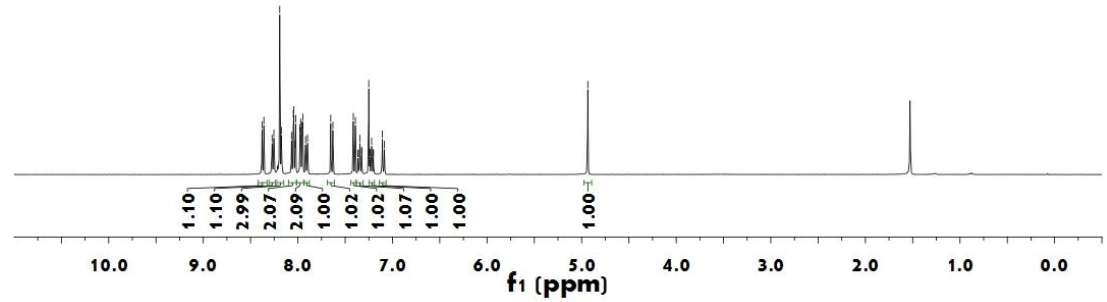

150.3
135.9
133.3
133.2
132.9
130.5
130.3
130.2
128.9
128.2
127.8
126.8
124.3
123.6
118.6
117.6

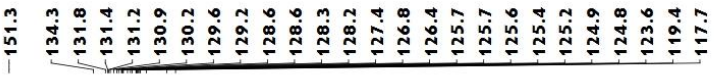

f1 (ppm)

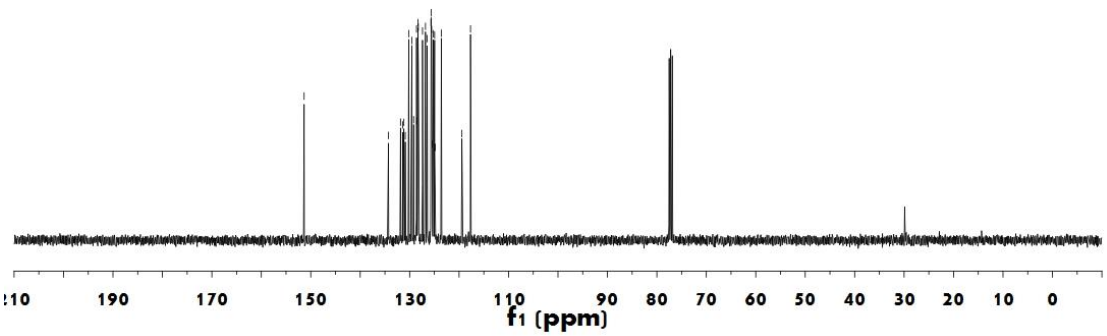

OH

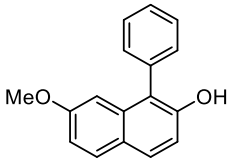
(1t)


7.65
7.63
7.41
7.39
7.36
7.36
7.34
7.33
7.32
7.25
7.24
7.24
7.22
7.20
7.20
7.11
7.09
4.93

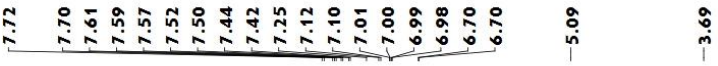

1.10
1.10
2.99
2.07
2.09
1.00
1.02
1.02
1.07
1.00
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

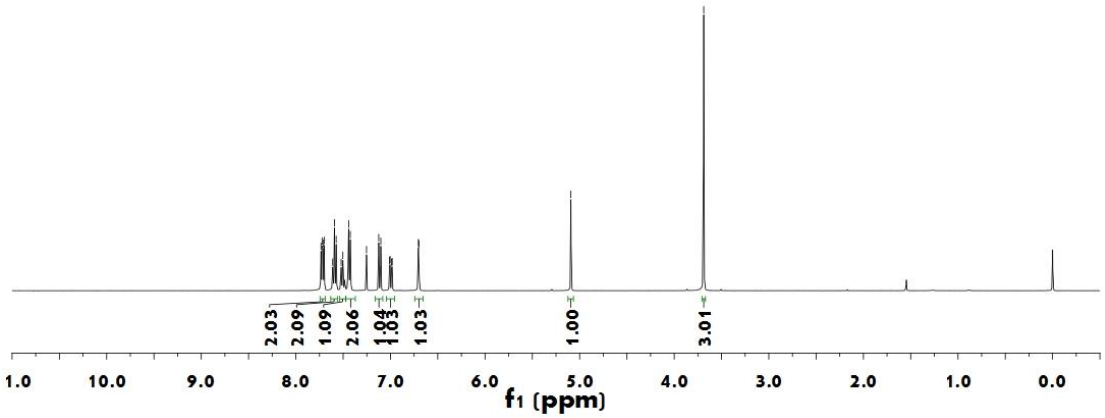

151.3
134.3
131.8
131.4
131.2
130.9
130.2
129.6
129.2
128.6
128.6
128.3
128.2
127.4
126.8
126.4
125.7
125.7
125.6
125.4
125.2
124.9
124.8
123.6
119.4
117.7

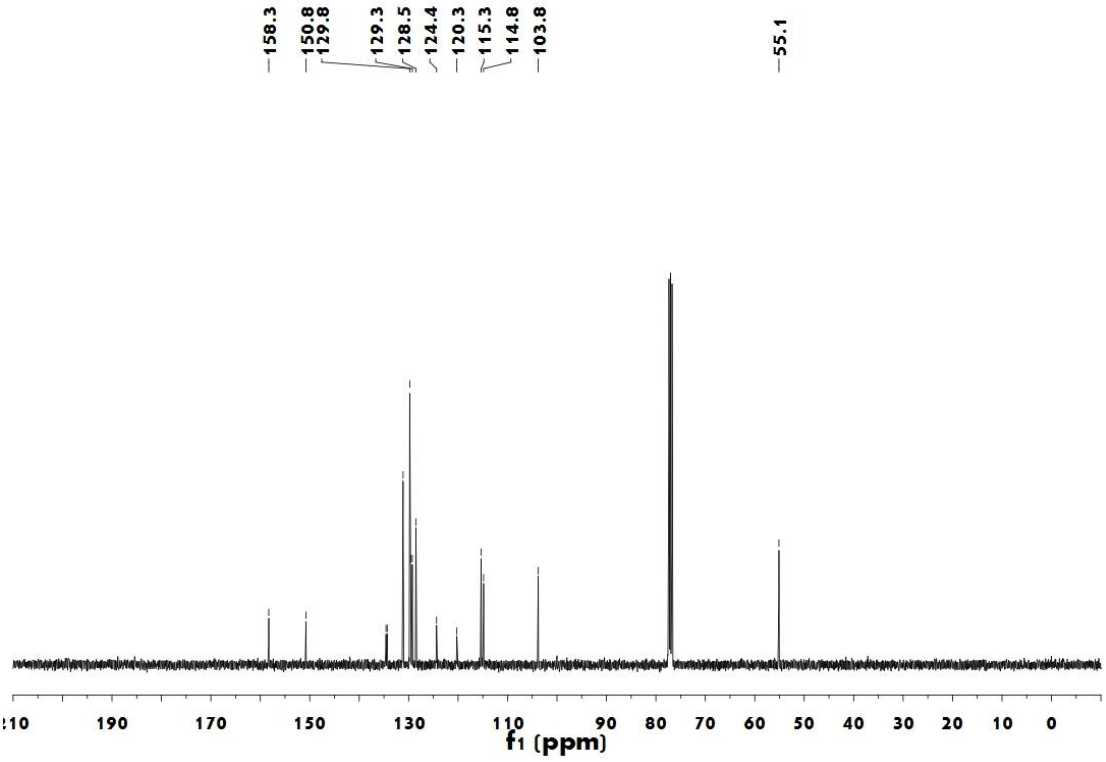

210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

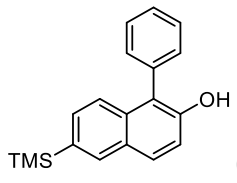

MeO
OH

(1u)

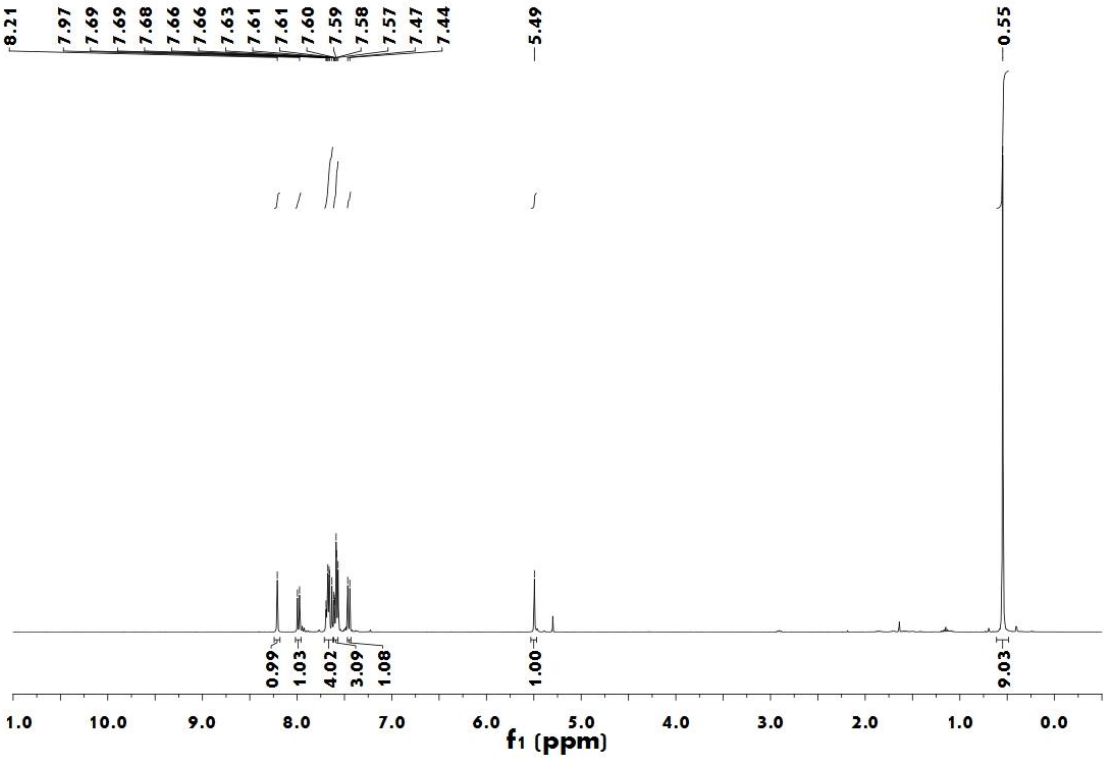

7.72
7.70
7.61
7.59
7.57
7.52
7.50
7.44
7.42
7.25
7.12
7.10
7.01
7.00
6.99
6.98
6.70
6.70
5.09
3.69

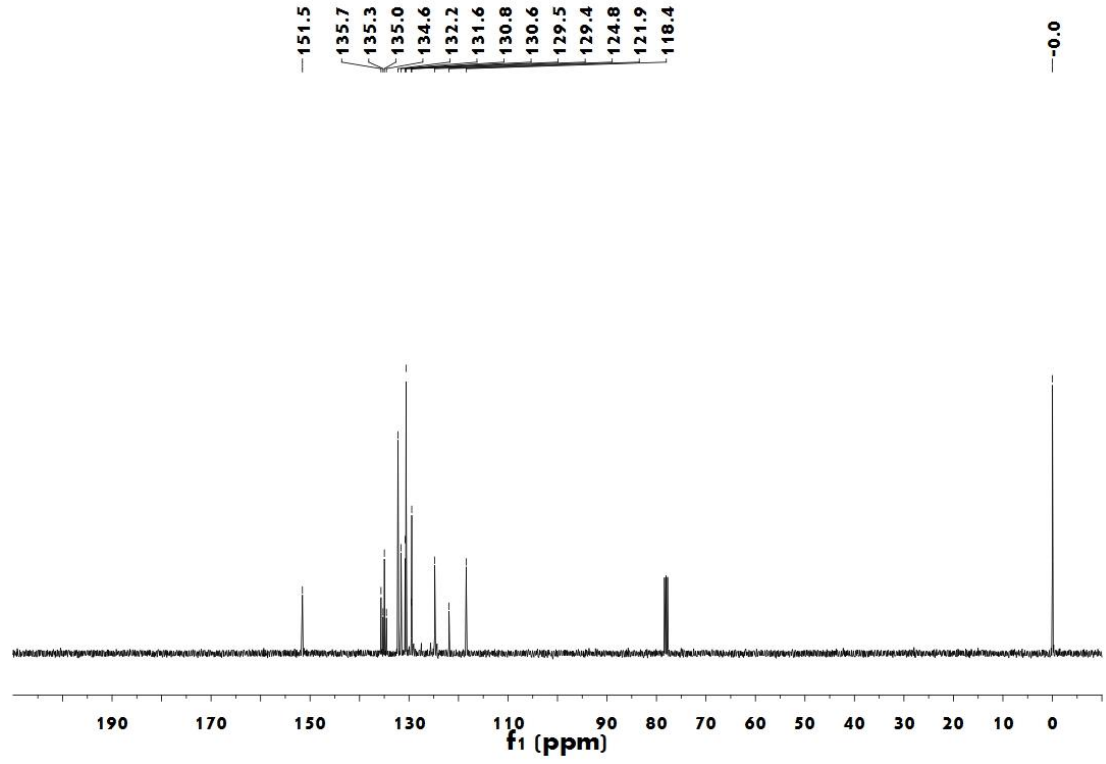

2.03
2.09
1.09
2.06
1.04
1.03
1.03
1.00
3.01
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

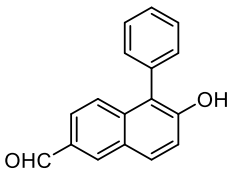

158.3
150.8
129.8
129.3
128.5
124.4
120.3
115.3
114.8
103.8
55.1
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OH
TMS

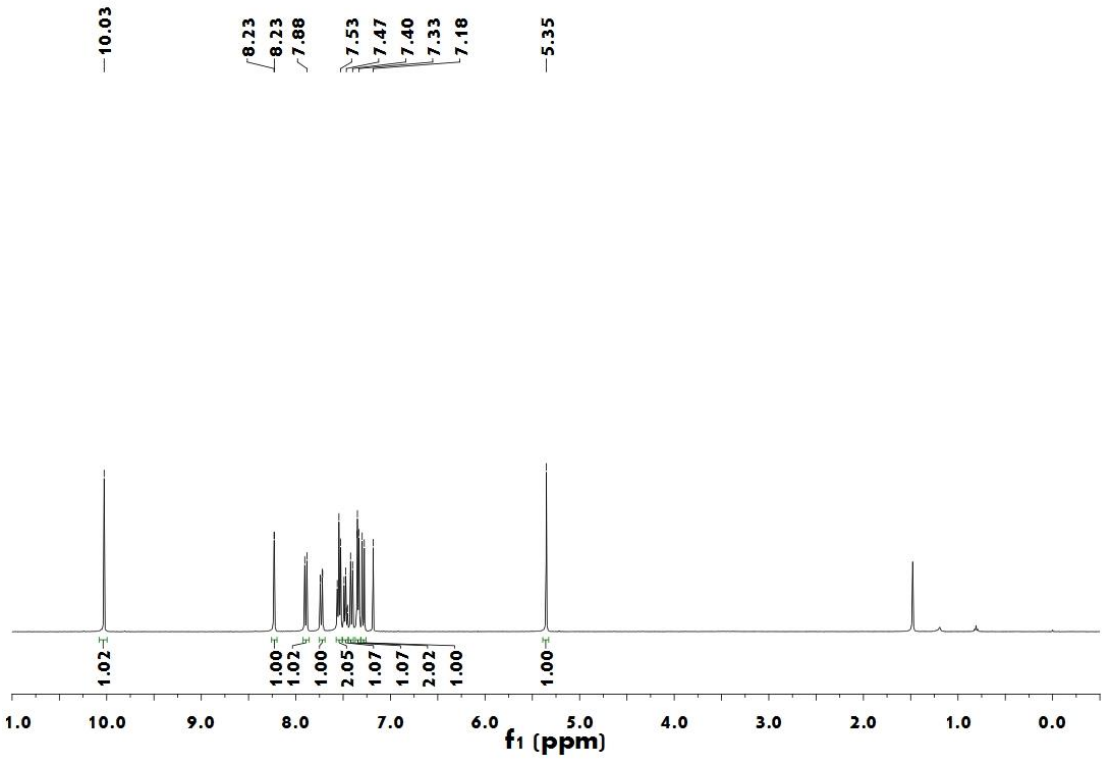
(1v)

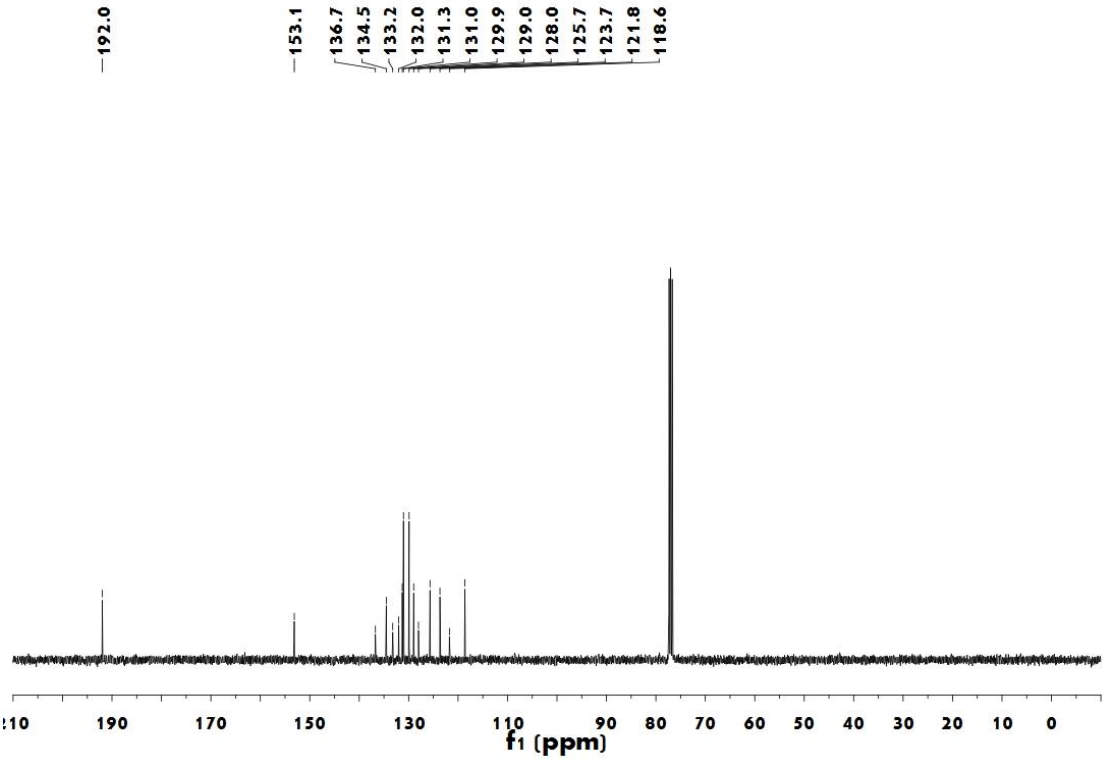

8.21
7.97
7.69
7.69
7.68
7.66
7.66
7.63
7.61
7.61
7.60
7.59
7.58
7.57
7.47
7.44
5.49
0.55
0.99
1.03
4.02
3.09
1.08
1.00
9.03
f1 (ppm)

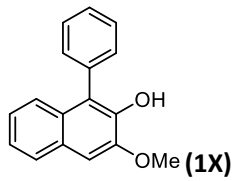

151.5
135.7
135.3
135.0
134.6
132.2
131.6
130.8
130.6
129.5
129.4
124.8
121.9
118.4
-0.0
f1 (ppm)


OH
OHC

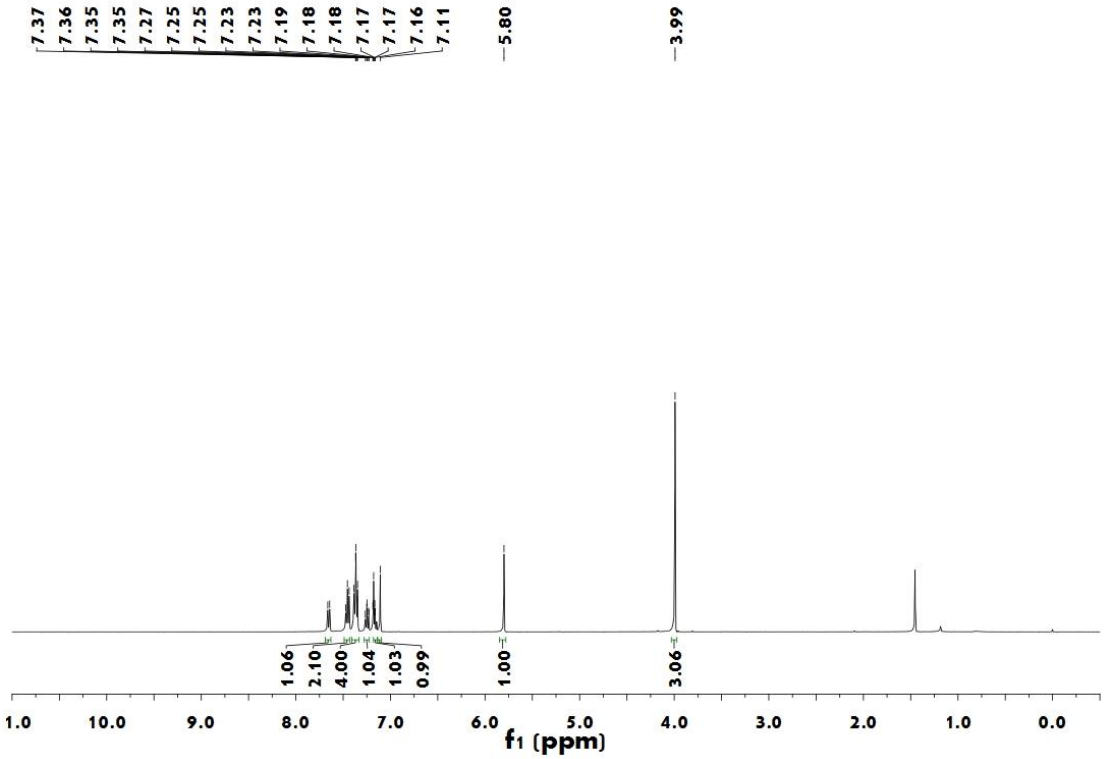
(1w)

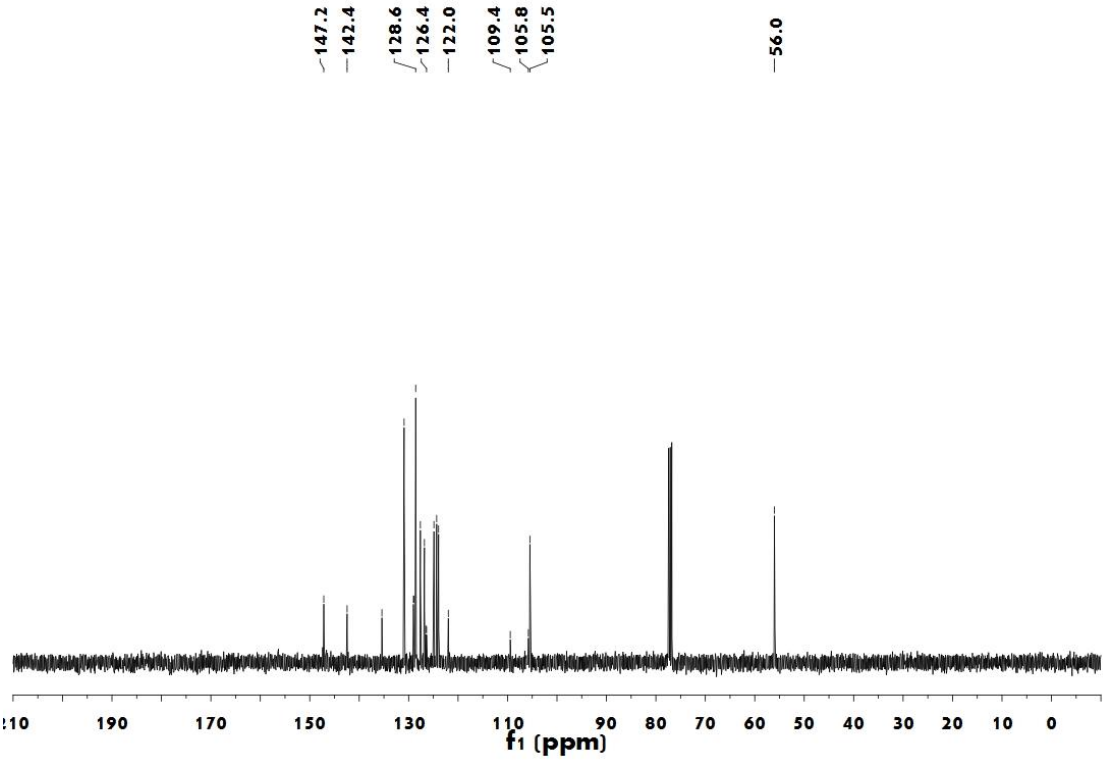

10.03
8.23
8.23
7.88
7.53
7.47
7.40
7.33
7.18
5.35
1.02
1.00
1.02
1.00
2.05
1.07
1.07
2.02
1.00
1.00
f1 (ppm)

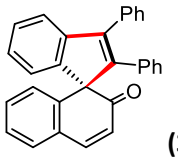

192.0
153.1
136.7
134.5
133.2
132.0
131.3
131.0
129.9
129.0
128.0
125.7
123.7
121.8
118.6
f1 (ppm)


OH
OMe

(1X)

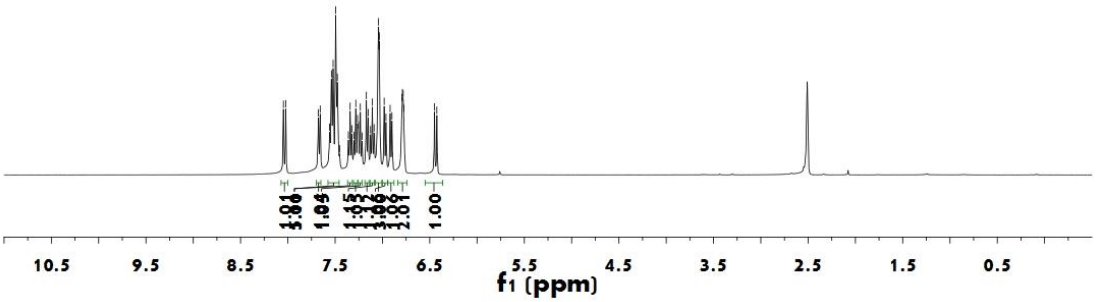

7.37
7.36
7.35
7.35
7.27
7.25
7.25
7.23
7.23
7.19
7.18
7.18
7.17
7.17
7.16
7.11
5.80
3.99
1.06
2.10
4.00
1.04
1.03
0.99
1.00
3.06
f1 (ppm)

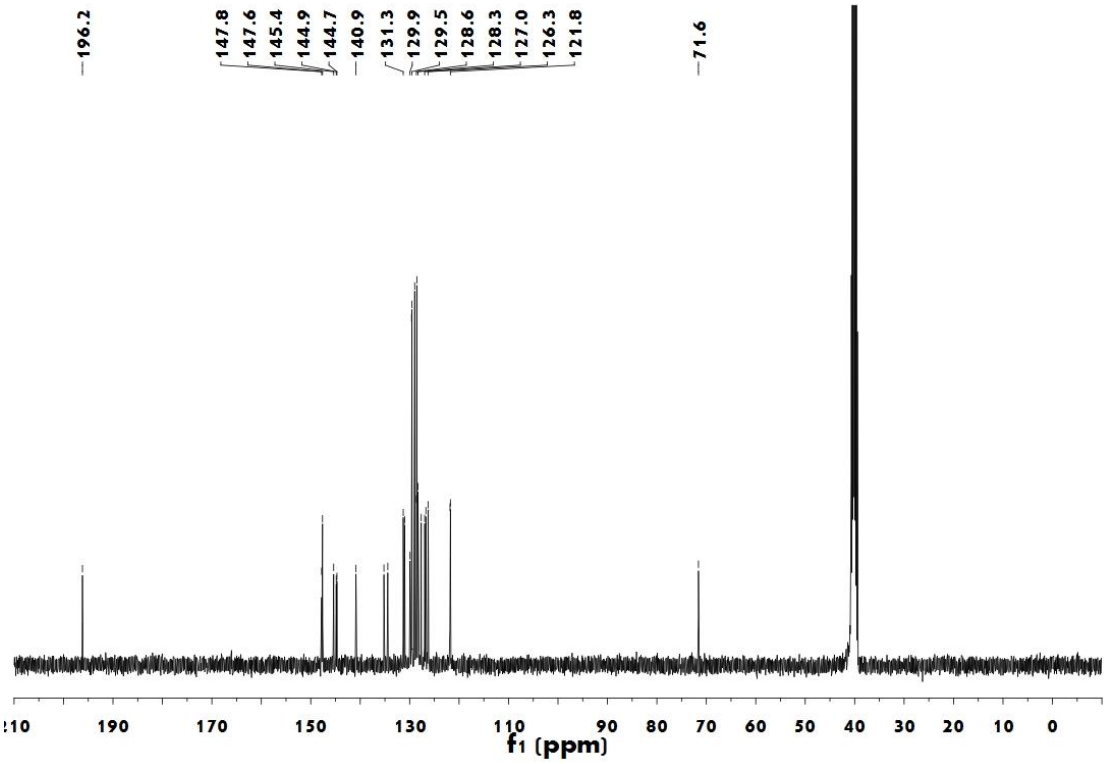

147.2
142.4
128.6
126.4
122.0
109.4
105.8
105.5
56.0
f1 (ppm)

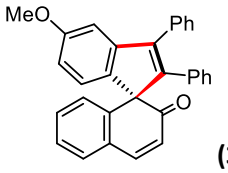

Ph
Ph
O

(3a)

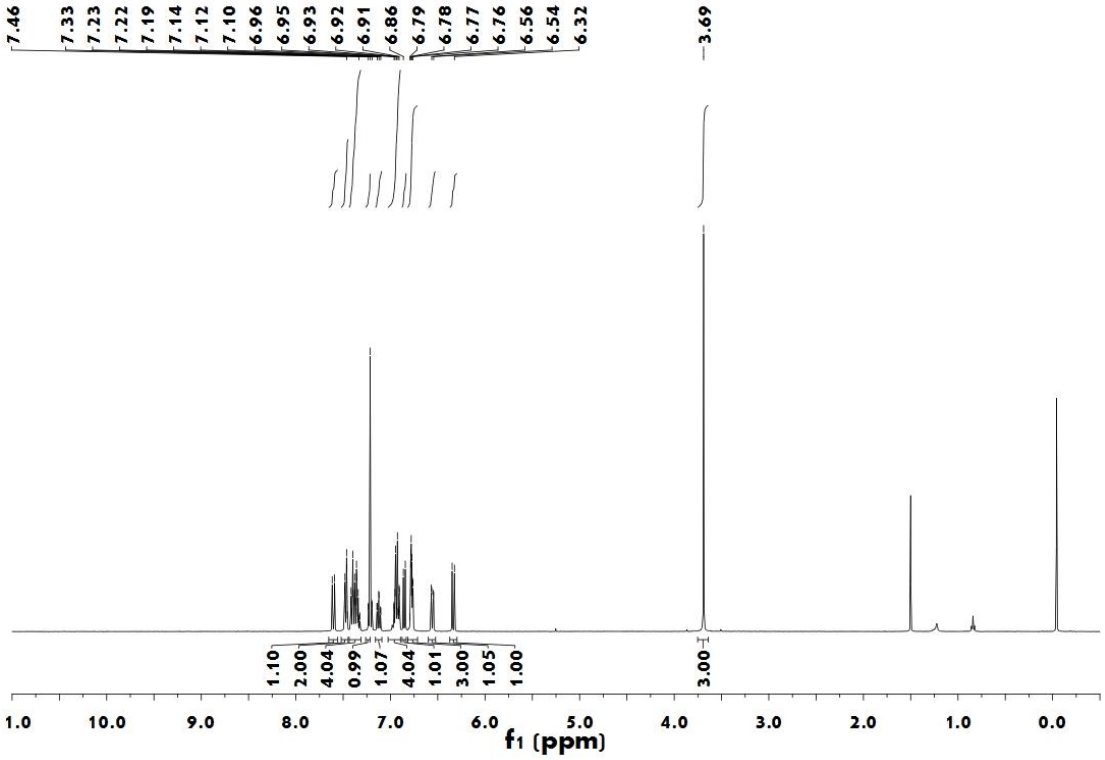

8.05
8.02
7.45
7.23
7.03
6.79
6.45
6.43

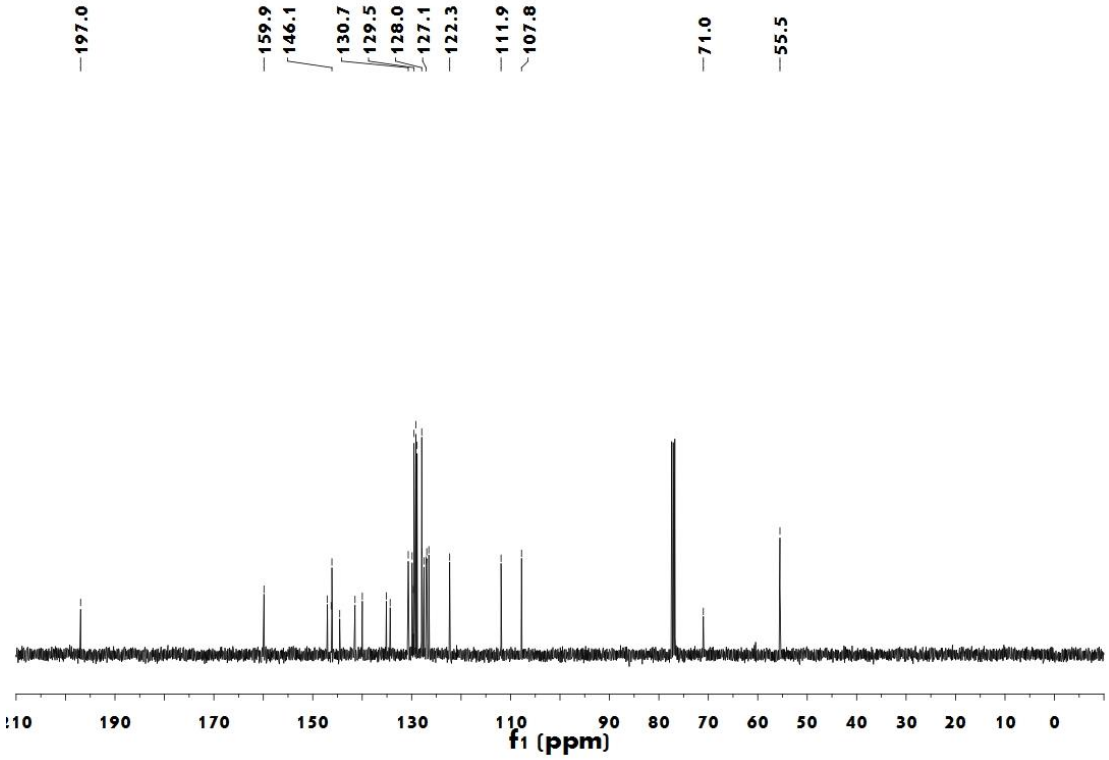

1.01
1.00
1.04
1.15
1.06
1.09
2.01
1.00
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

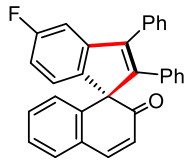

196.2
147.8
147.6
145.4
144.9
144.7
140.9
131.3
129.9
129.5
128.6
128.3
127.0
126.3
121.8
71.6
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MeO
Ph
Ph
O

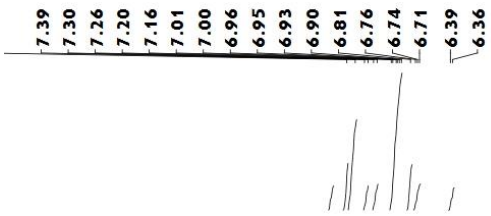
(3b)

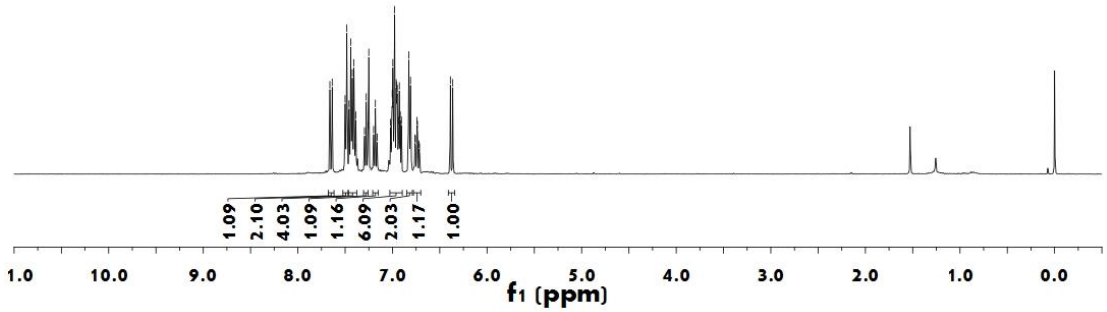

7.46
7.33
7.23
7.22
7.19
7.14
7.12
7.10
6.96
6.95
6.93
6.92
6.91
6.86
6.79
6.78
6.77
6.76
6.56
6.54
6.32
3.69
1.10
2.00
4.04
0.99
1.07
4.04
1.01
3.00
1.05
1.00
3.00
f1 (ppm)

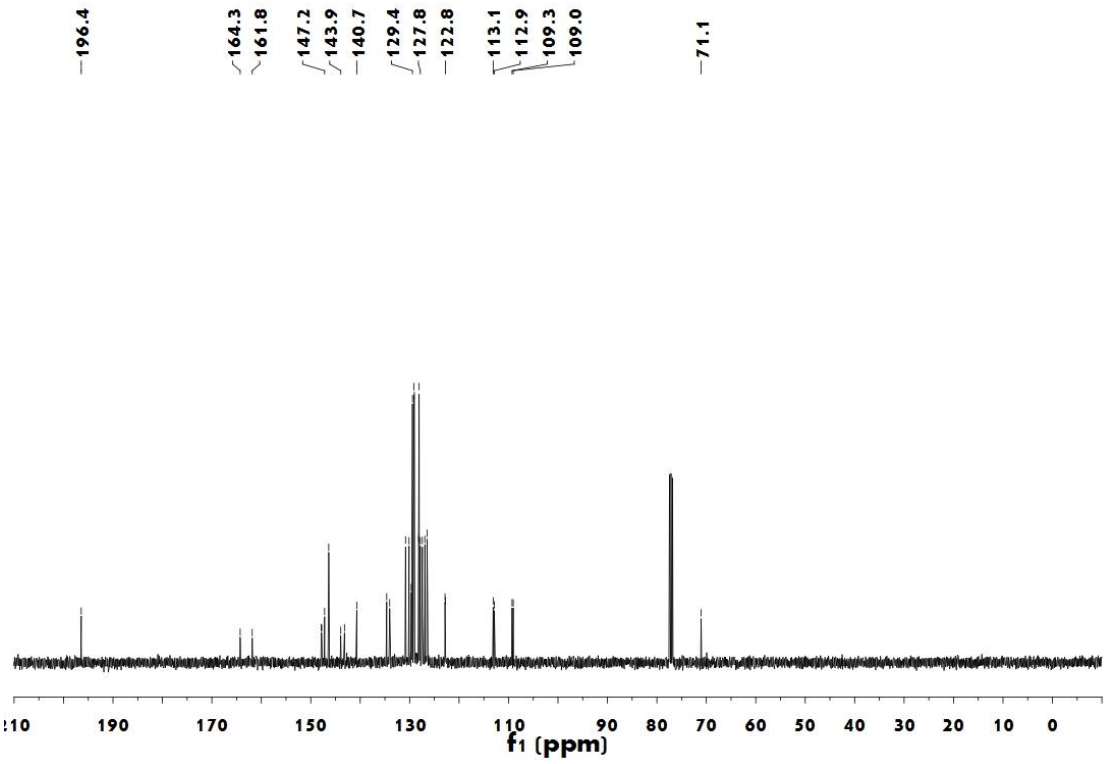

197.0
159.9
146.1
130.7
129.5
128.0
127.1
122.3
111.9
107.8
71.0
55.5
f1 (ppm)

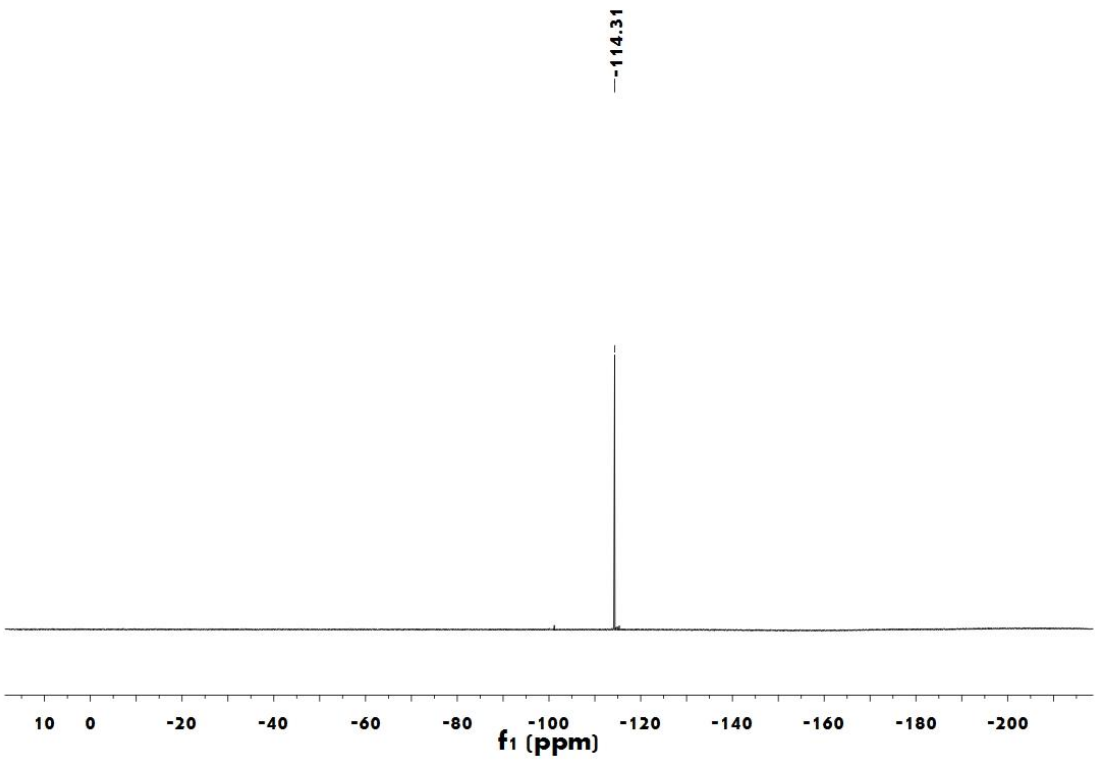

F
Ph
Ph
O

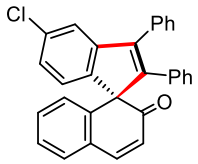
(3c)


7.39
7.30
7.26
7.20
7.16
7.01
7.00
6.96
6.95
6.93
6.90
6.81
6.76
6.74
6.71
6.39
6.36

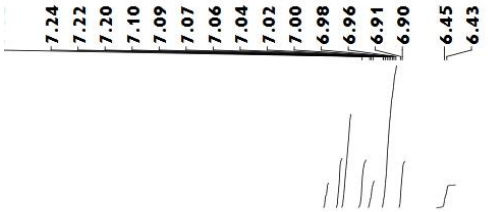

1.09
2.10
4.03
1.09
1.16
6.09
2.03
1.17
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

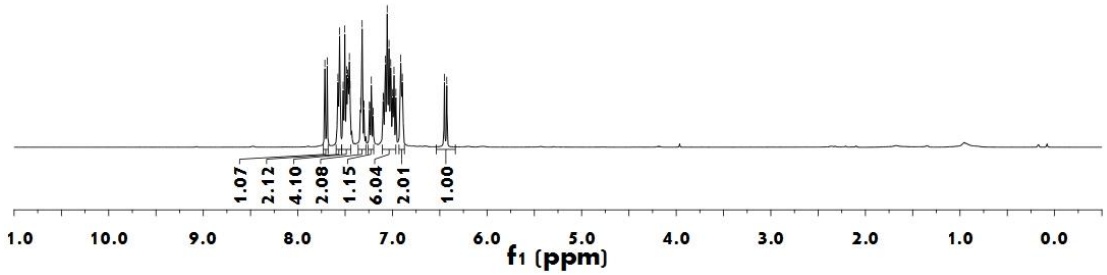

196.4
164.3
161.8
147.2
143.9
140.7
129.4
127.8
122.8
113.1
112.9
109.3
109.0
71.1
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

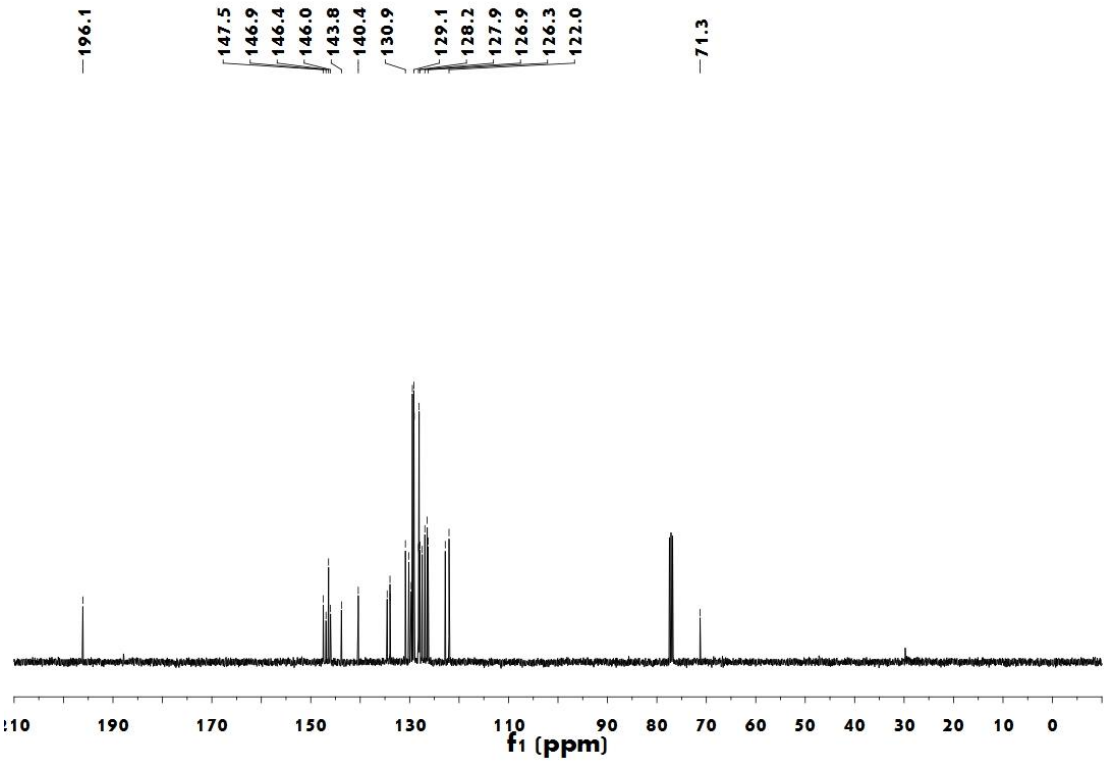

-114.31
10
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)

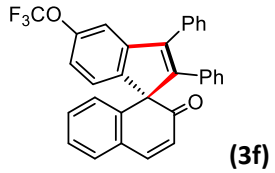

Cl
Ph
Ph
O

(3d)

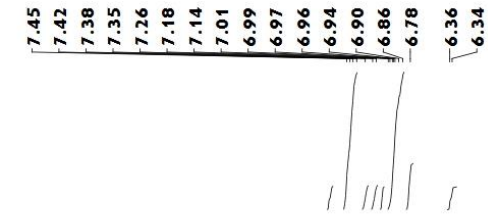

7.24
7.22
7.20
7.10
7.09
7.07
7.06
7.04
7.02
7.00
6.98
6.96
6.91
6.90
6.45
6.43

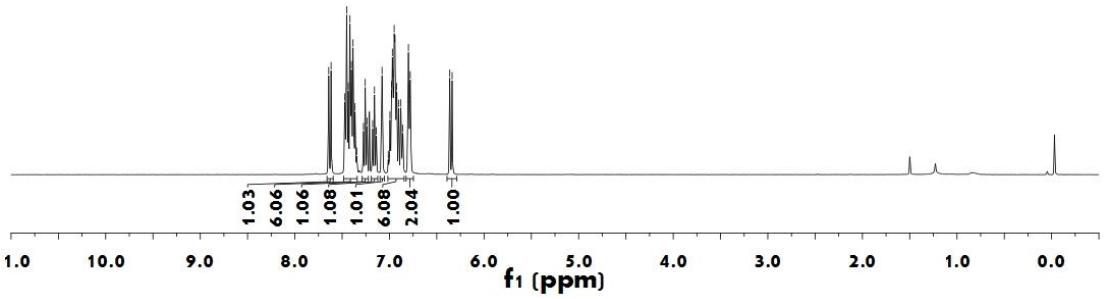

1.07
2.12
4.10
2.08
1.15
6.04
2.01
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

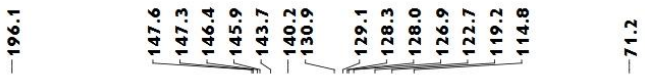

196.1
147.5
146.9
146.4
146.0
143.8
140.4
130.9
129.1
128.2
127.9
126.9
126.3
122.0
71.3
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

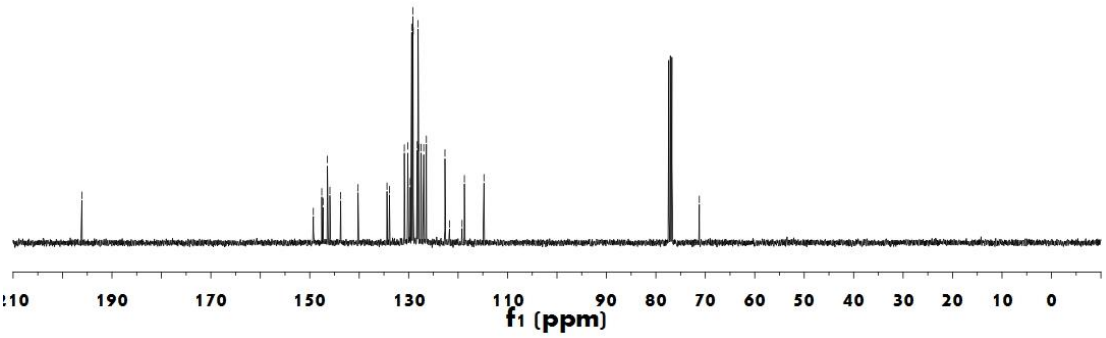

F3CO
Ph
Ph
O

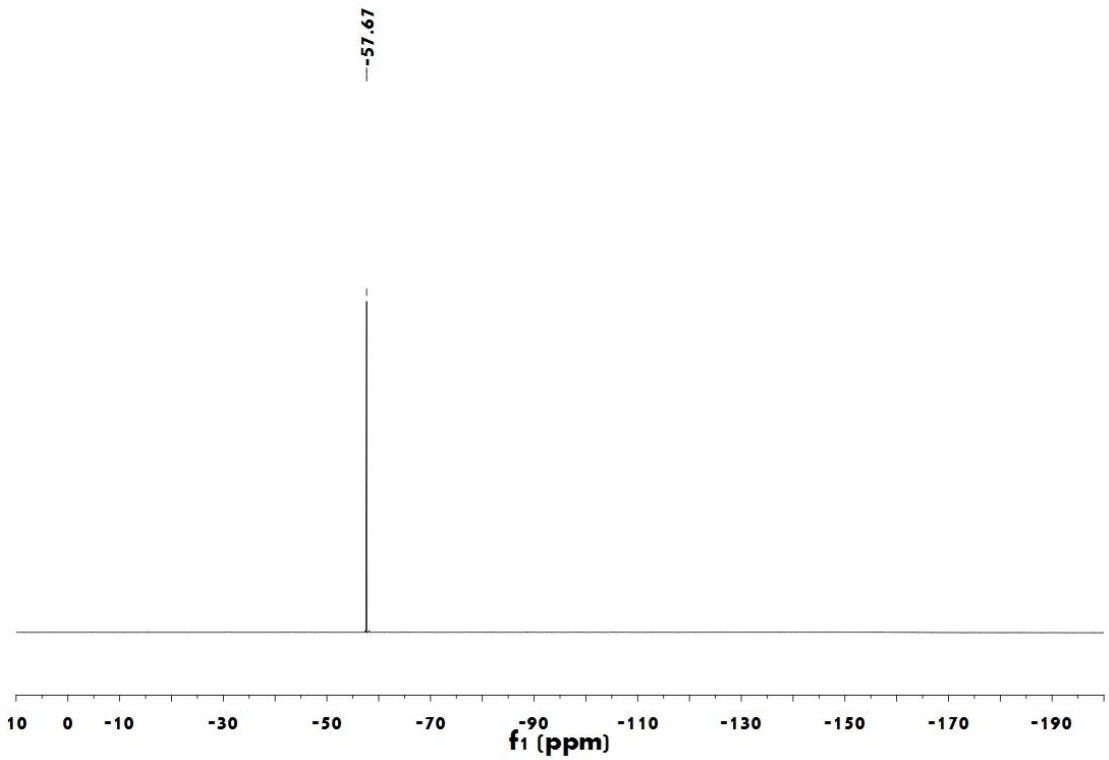
(3f)

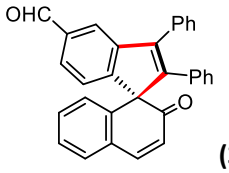

7.45
7.42
7.38
7.35
7.26
7.18
7.14
7.01
6.99
6.97
6.96
6.94
6.90
6.86
6.78
6.36
6.34


1.03
6.06
1.06
1.08
1.01
6.08
2.04
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

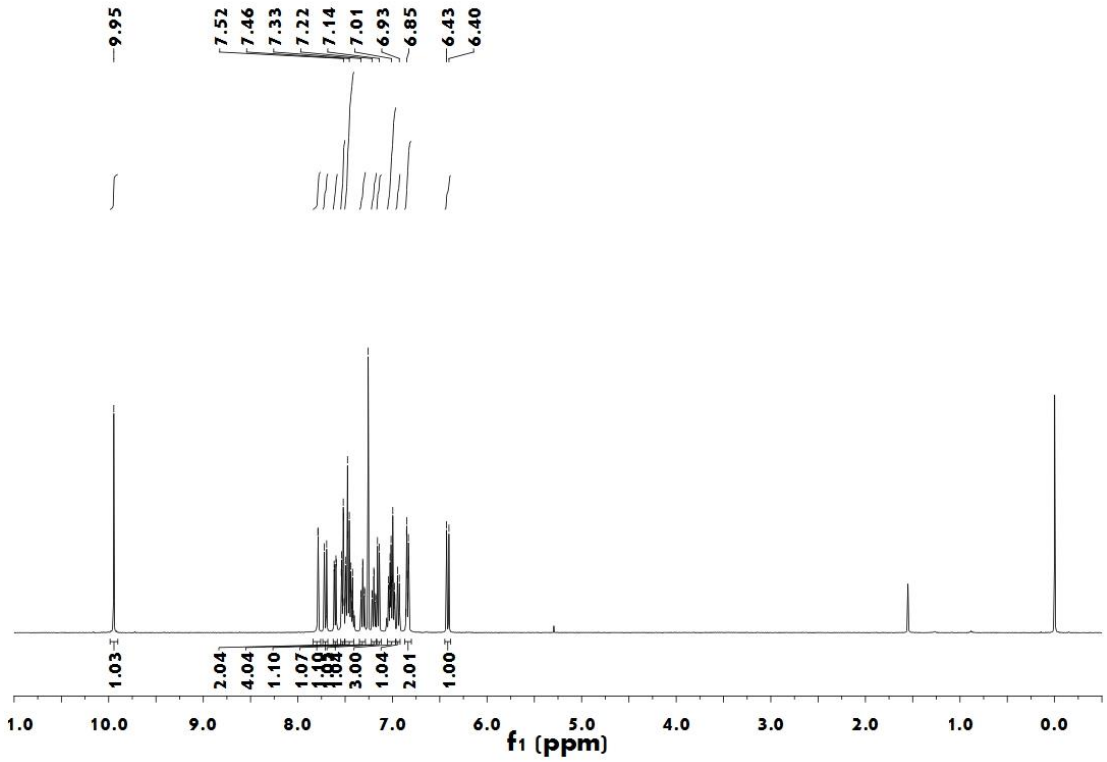

196.1
147.6
147.3
146.4
145.9
143.7
140.2
130.9
129.1
128.3
128.0
126.9
122.7
119.2
114.8
71.2

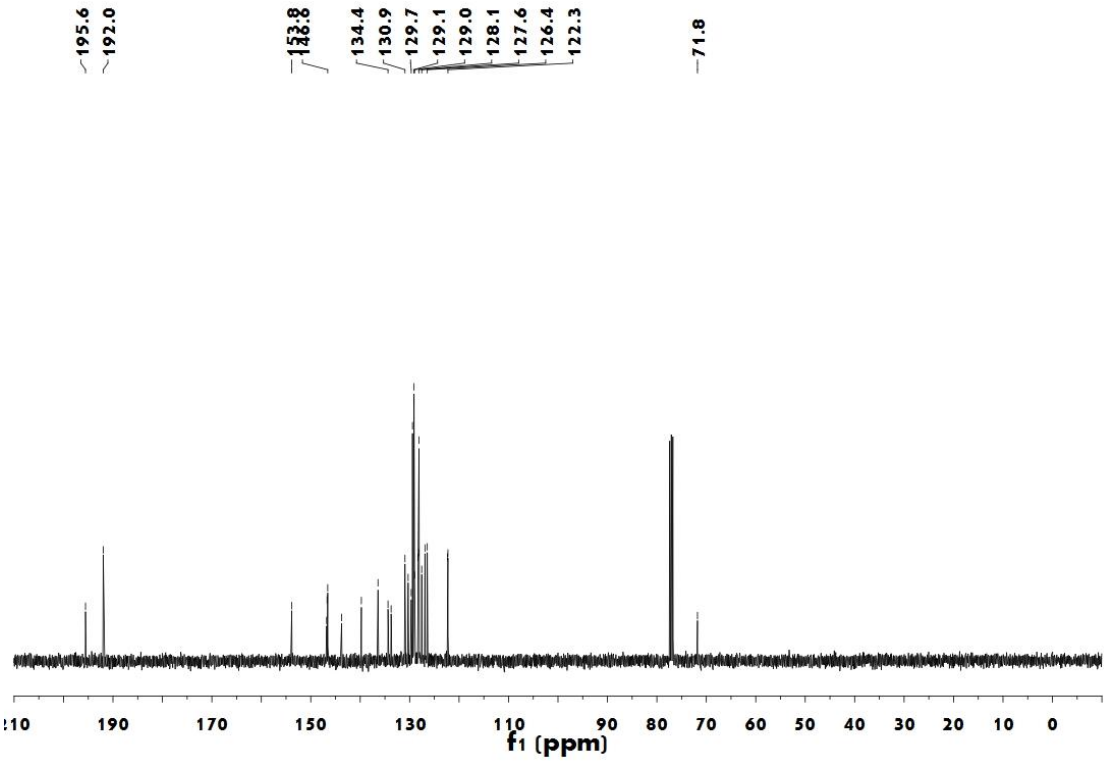

210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

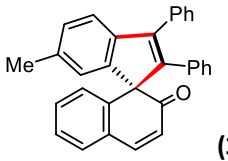

-57.67
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
f1 (ppm)


OHC
Ph
Ph
O

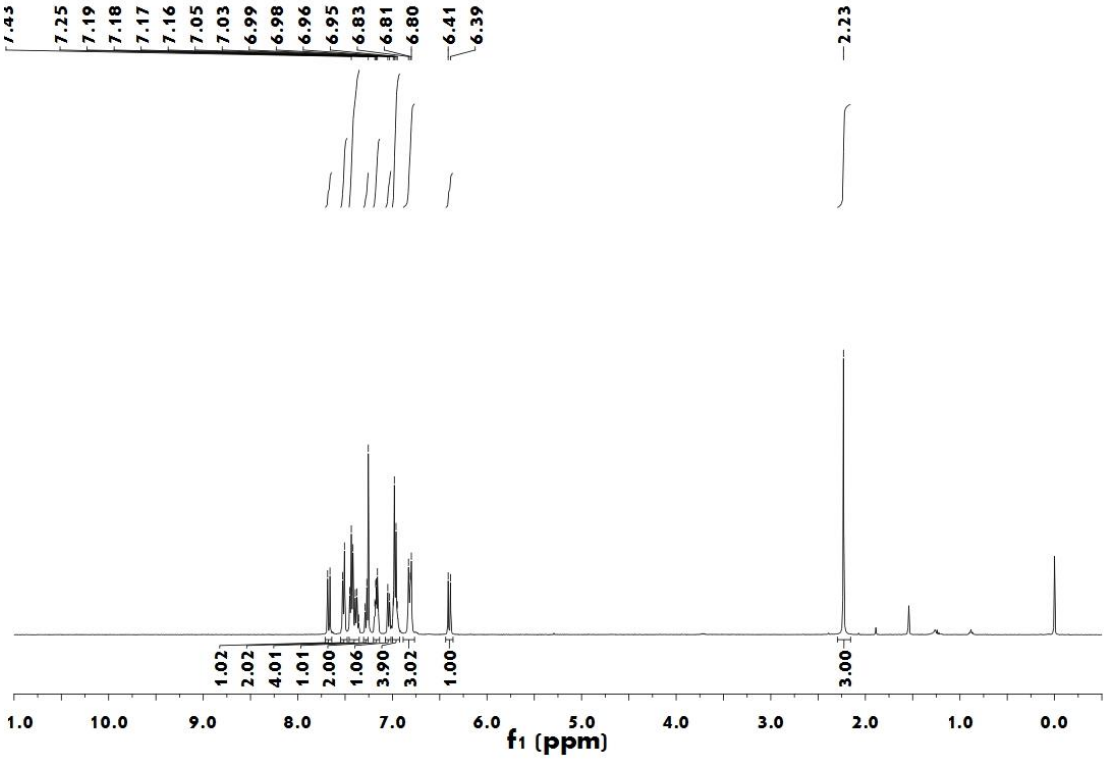
(3g)

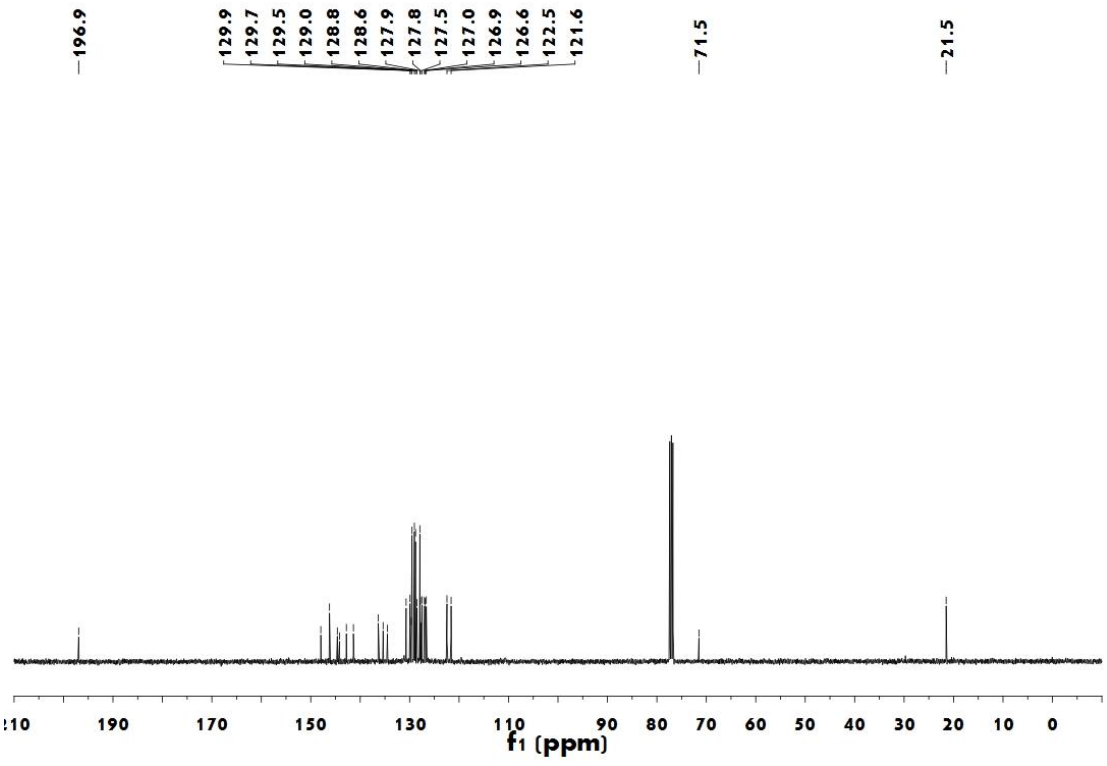

9.95
7.52
7.46
7.33
7.22
7.14
7.01
6.93
6.85
6.43
6.40
1.03
2.04
4.04
1.10
1.07
3.00
1.04
2.01
1.00
f1 (ppm)

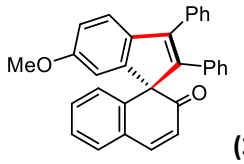

195.6
192.0
134.4
130.9
129.7
129.1
129.0
128.1
127.6
126.4
122.3
71.8
f1 (ppm)


Ph
Me
Ph
O

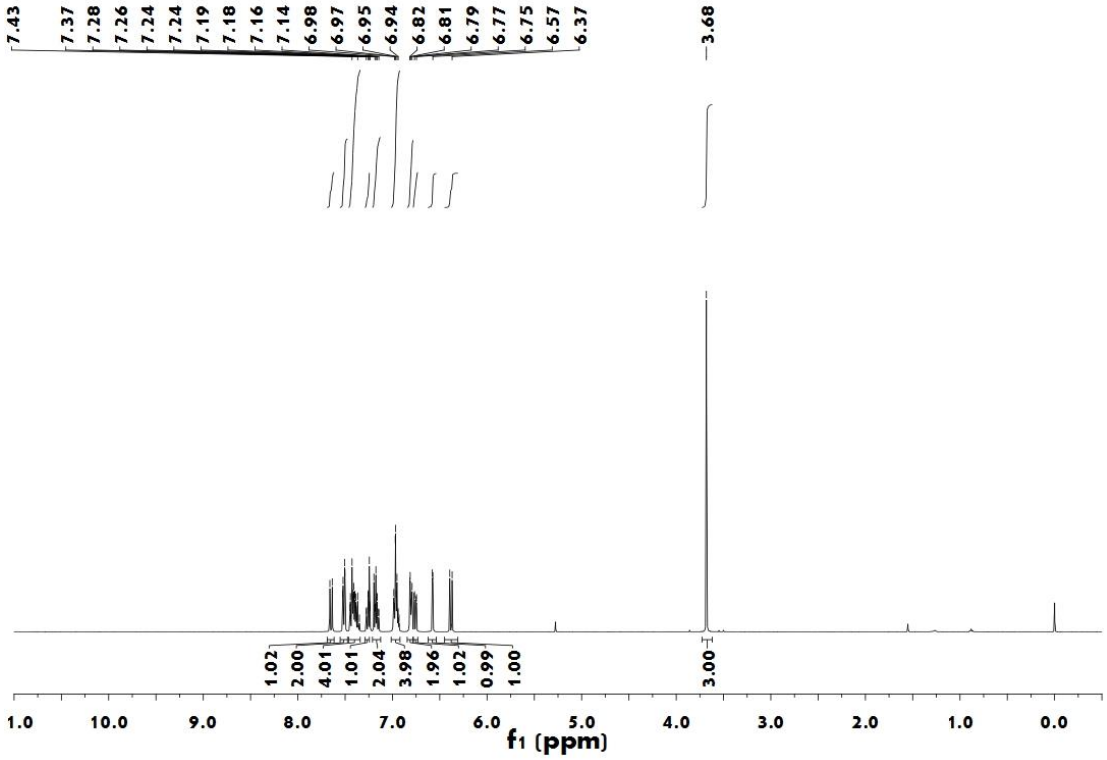
(3i)

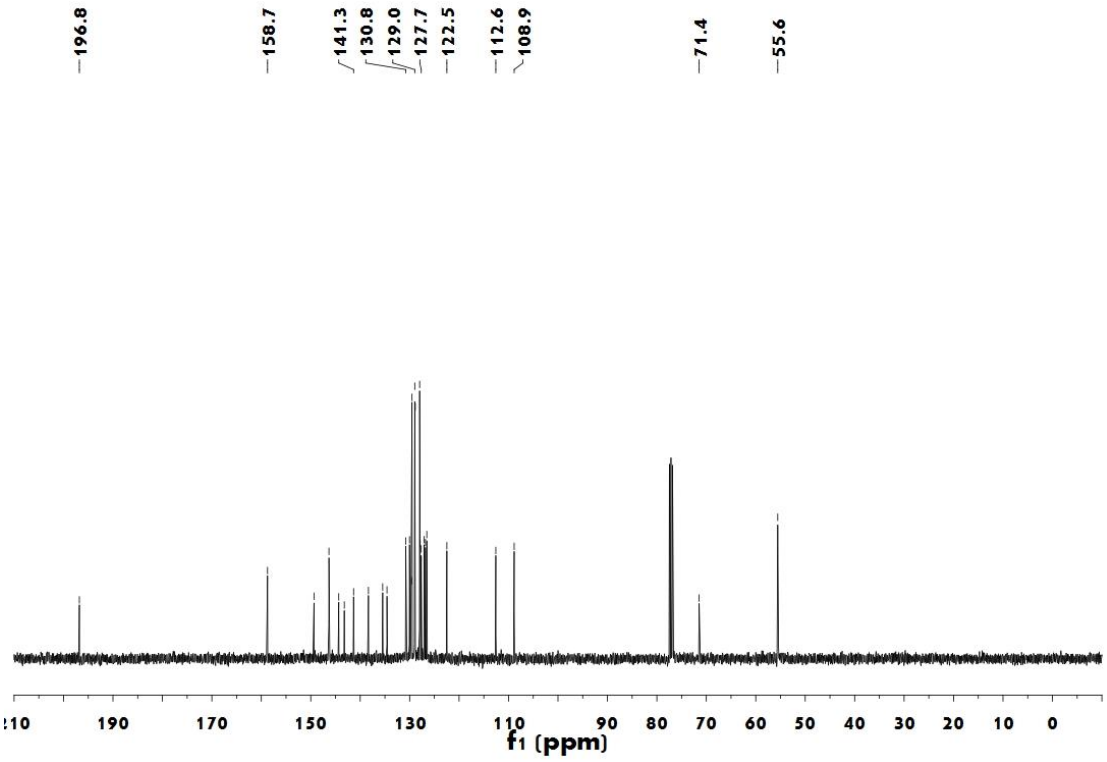

7.43
7.25
7.19
7.18
7.17
7.16
7.05
7.03
6.99
6.98
6.96
6.95
6.83
6.81
6.80
6.41
6.39
2.23
1.02
2.02
4.01
1.01
2.00
1.06
3.90
3.02
1.00
3.00
f1 (ppm)

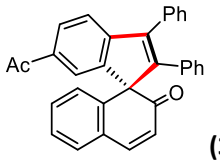

196.9
129.9
129.7
129.5
129.0
128.8
128.6
127.9
127.8
127.5
127.0
126.9
126.6
122.5
121.6
71.5
21.5
f1 (ppm)


Ph
MeO
Ph
O

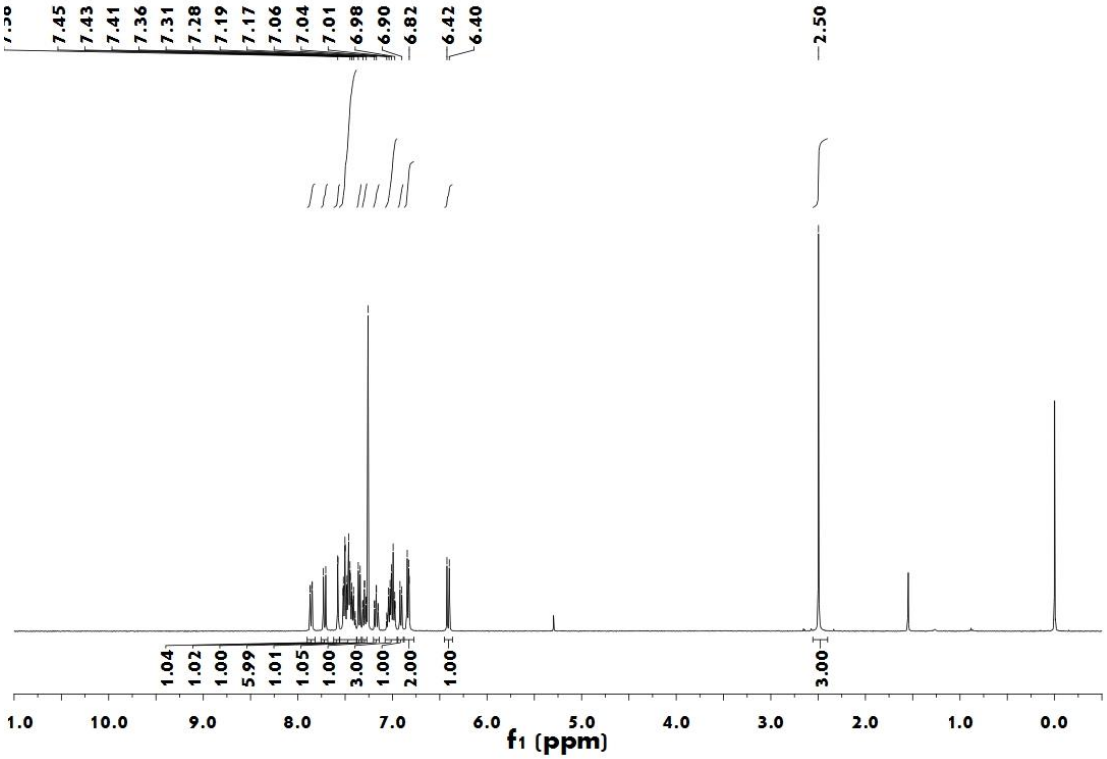
(3j)

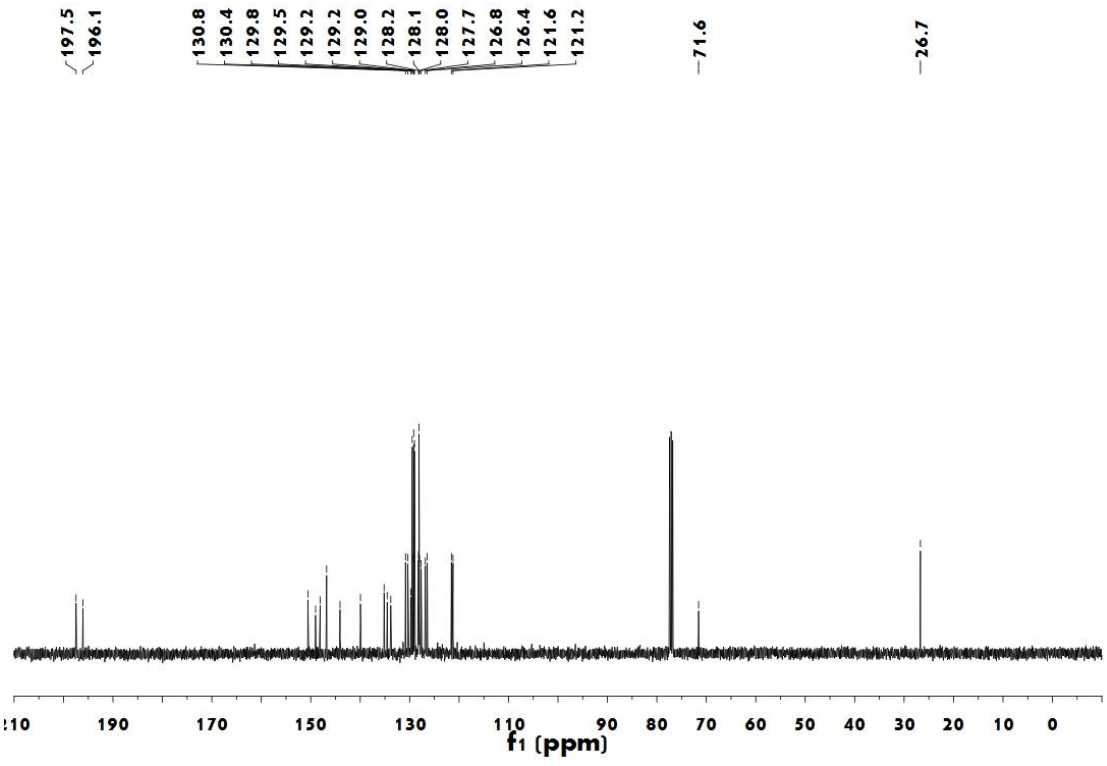

7.43
7.37
7.28
7.26
7.24
7.24
7.19
7.18
7.16
7.14
6.98
6.97
6.95
6.94
6.82
6.81
6.79
6.77
6.75
6.57
6.37
3.68
1.02
2.00
4.01
1.01
2.04
3.98
1.96
1.02
0.99
1.00
3.00
f1 (ppm)

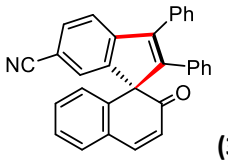

196.8
158.7
141.3
130.8
129.0
127.7
122.5
112.6
108.9
71.4
55.6
f1 (ppm)


Ph
Ac
Ph
O

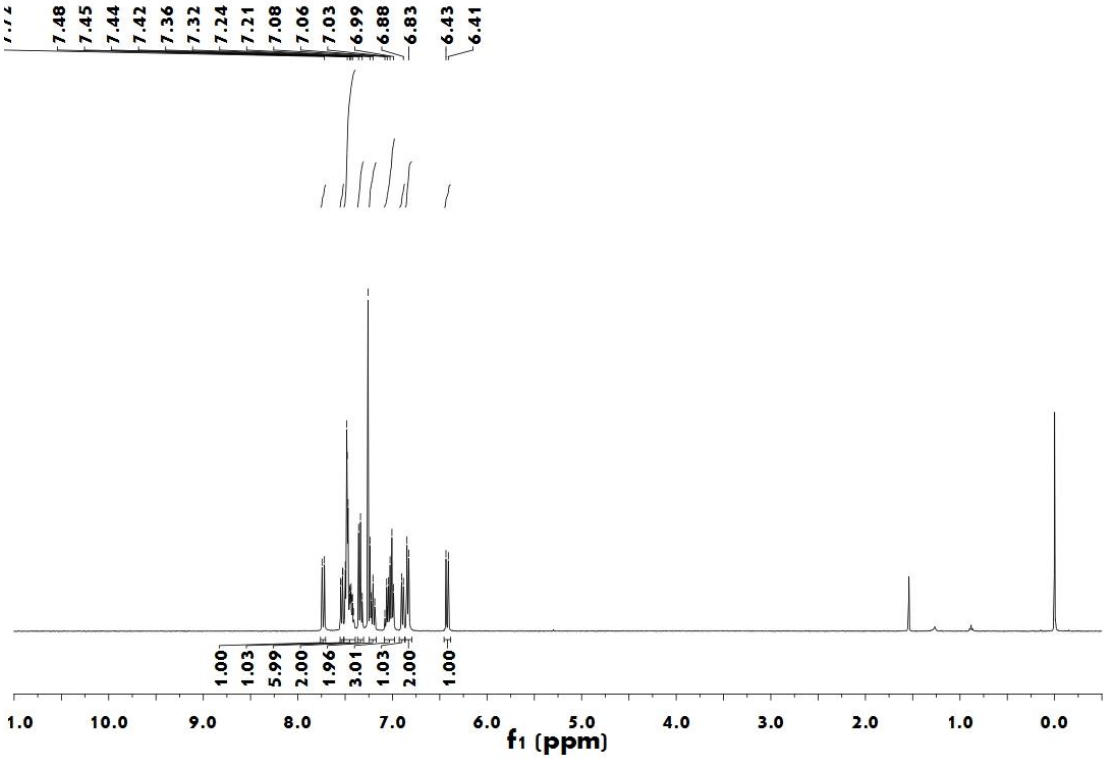
(3k)

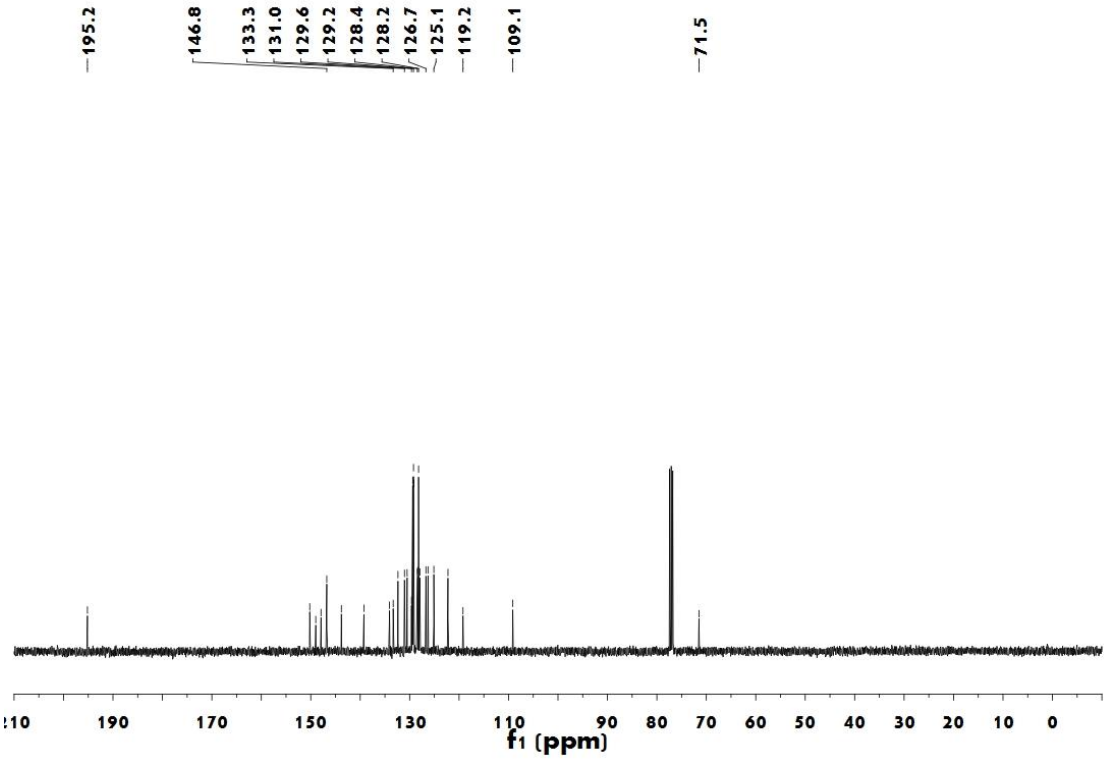

7.45
7.43
7.41
7.36
7.31
7.28
7.19
7.17
7.06
7.04
7.01
6.98
6.90
6.82
6.42
6.40
2.50
1.04
1.02
1.00
5.99
1.01
1.05
1.00
3.00
1.00
2.00
1.00
3.00
f1 (ppm)

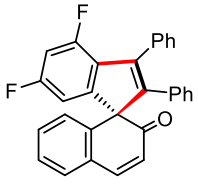

197.5
196.1
130.8
130.4
129.8
129.5
129.2
129.2
129.0
128.2
128.1
128.0
127.7
126.8
126.4
121.6
121.2
71.6
26.7
f1 (ppm)


Ph
NC
Ph
O

(3l)

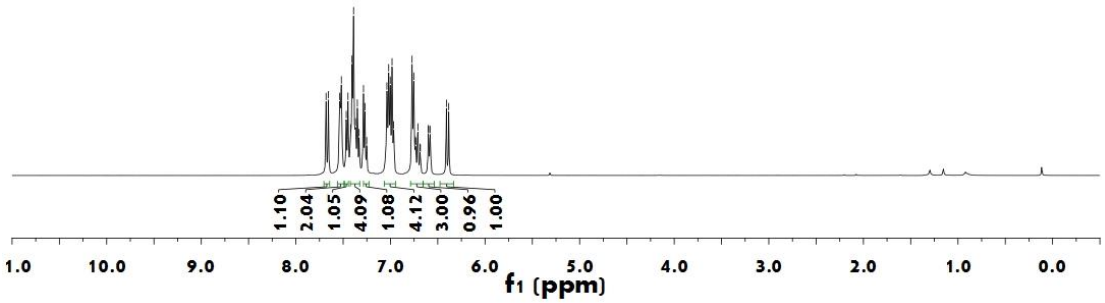

7.48
7.45
7.44
7.42
7.36
7.32
7.24
7.21
7.08
7.06
7.03
6.99
6.88
6.83
6.43
6.41
1.00
1.03
5.99
2.00
1.96
3.01
1.03
2.00
1.00
f1 (ppm)

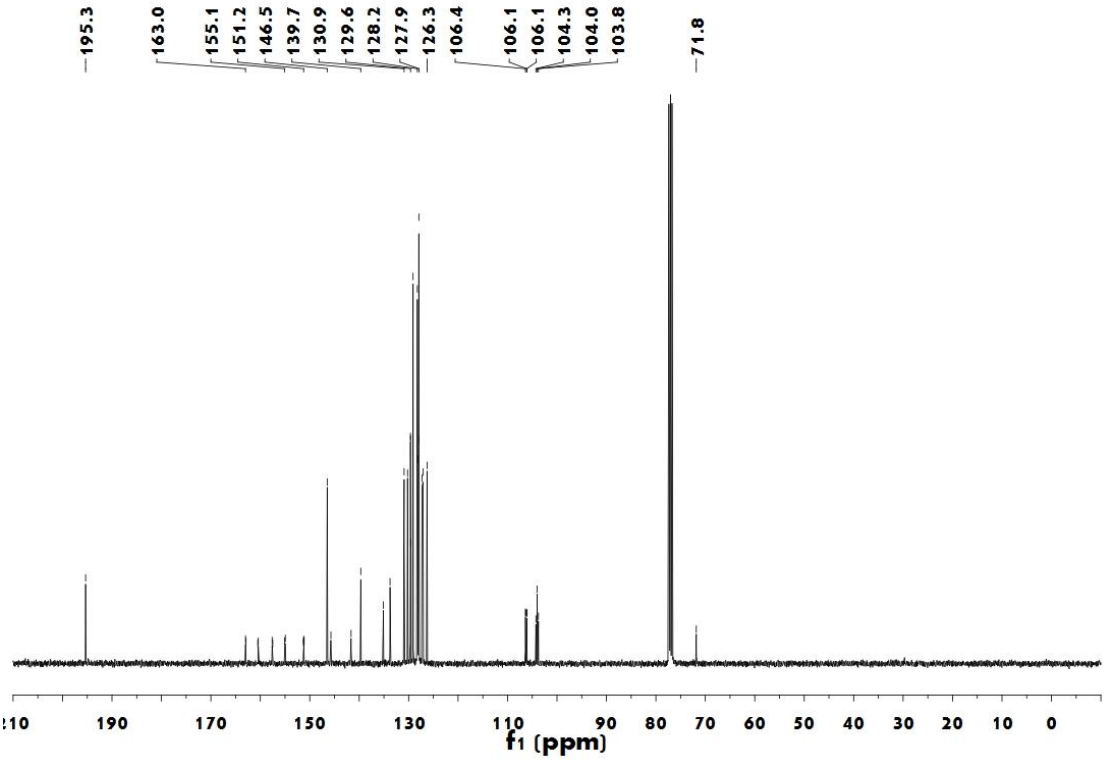

195.2
146.8
133.3
131.0
129.6
129.2
128.4
128.2
126.7
125.1
119.2
109.1
71.5
f1 (ppm)

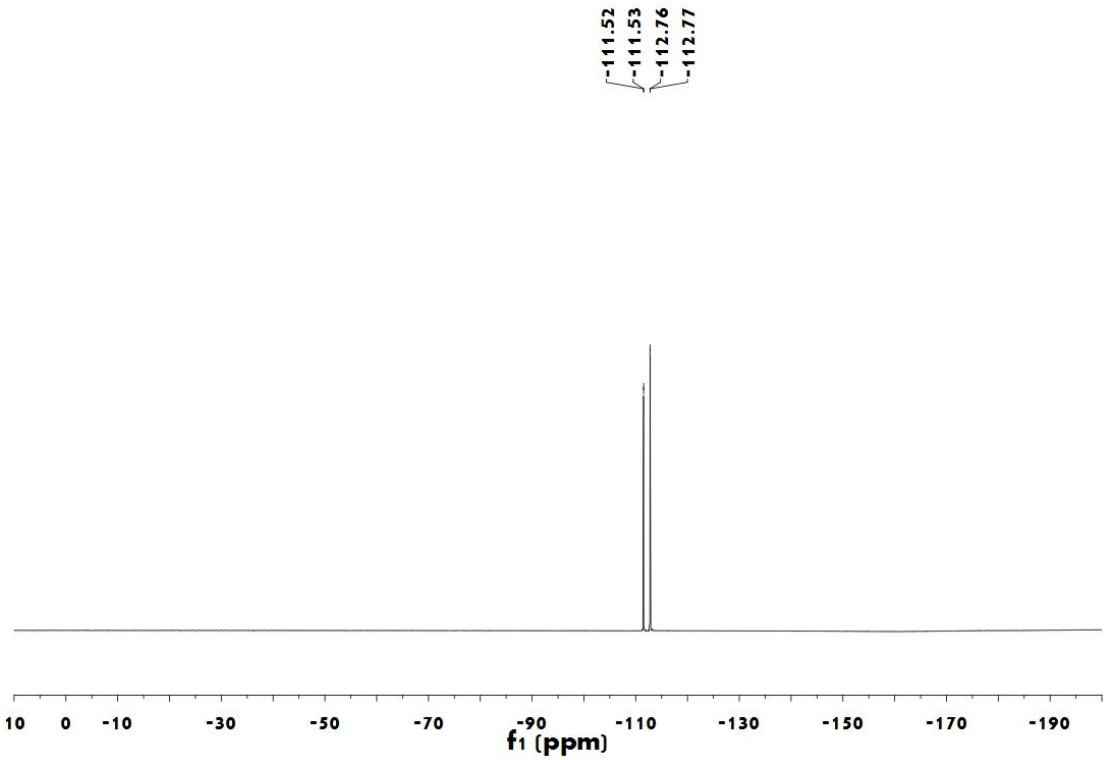

F
Ph
F
Ph
O

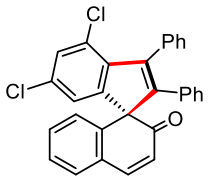
(3n)


7.68
7.66
7.54
7.52
7.47
7.45
7.42
7.41
7.39
7.37
7.35
7.33
7.28
7.27
7.25
7.04
7.02
7.00
6.98
6.97
6.77
6.75
6.74
6.73
6.71
6.69
6.68
6.60
6.60
6.58
6.41
6.38

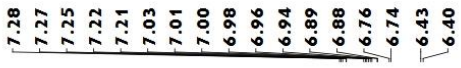

1.10
2.04
1.05
4.09
1.08
4.12
3.00
0.96
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

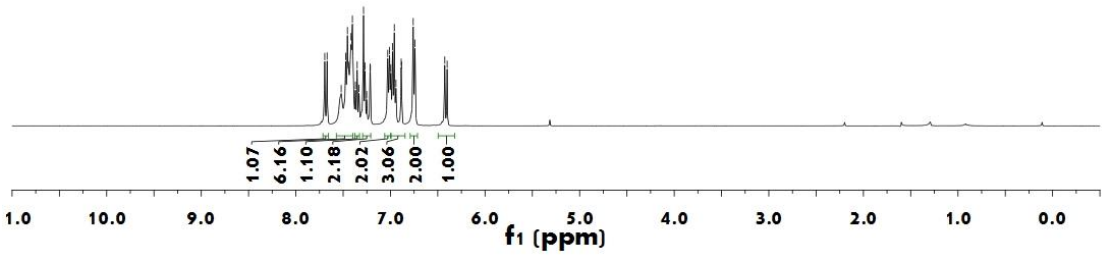

195.3
163.0
155.1
151.2
146.5
139.7
130.9
129.6
128.2
127.9
126.3
106.4
106.1
106.1
104.3
104.0
103.8
71.8
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

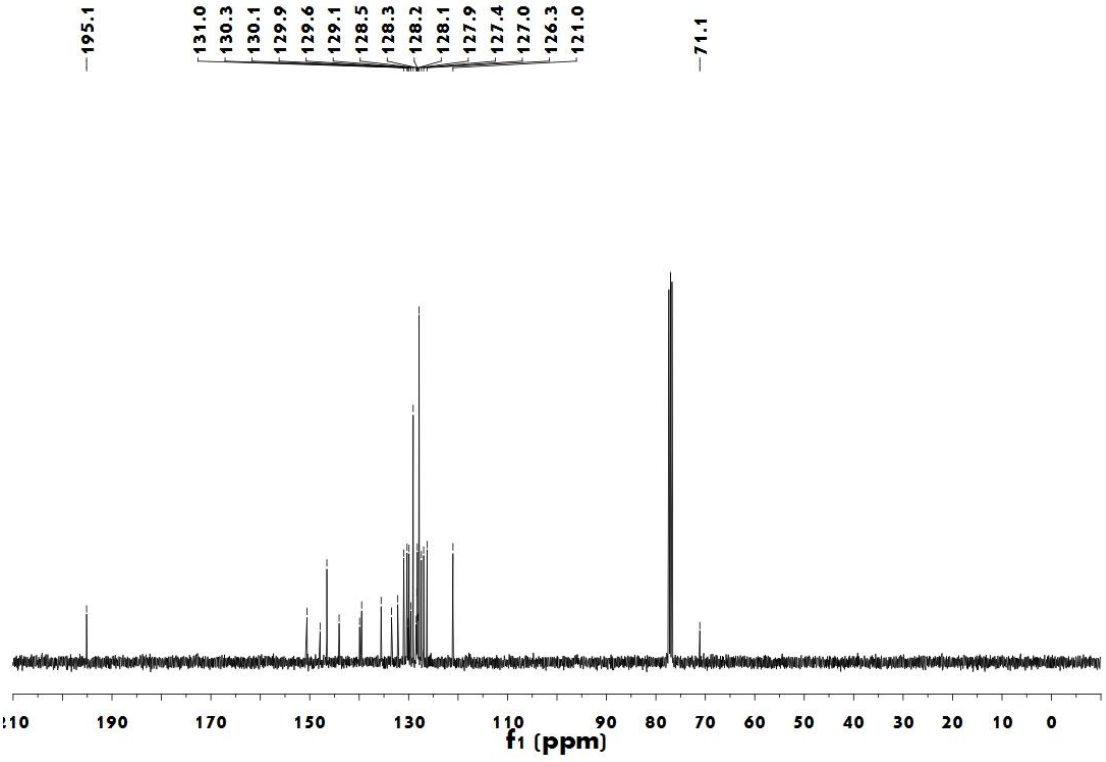

-111.52
-111.53
-112.76
-112.77
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
f1 (ppm)

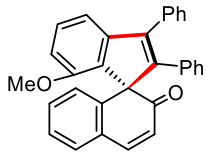

Cl
Ph
Cl
Ph
O

(3o)

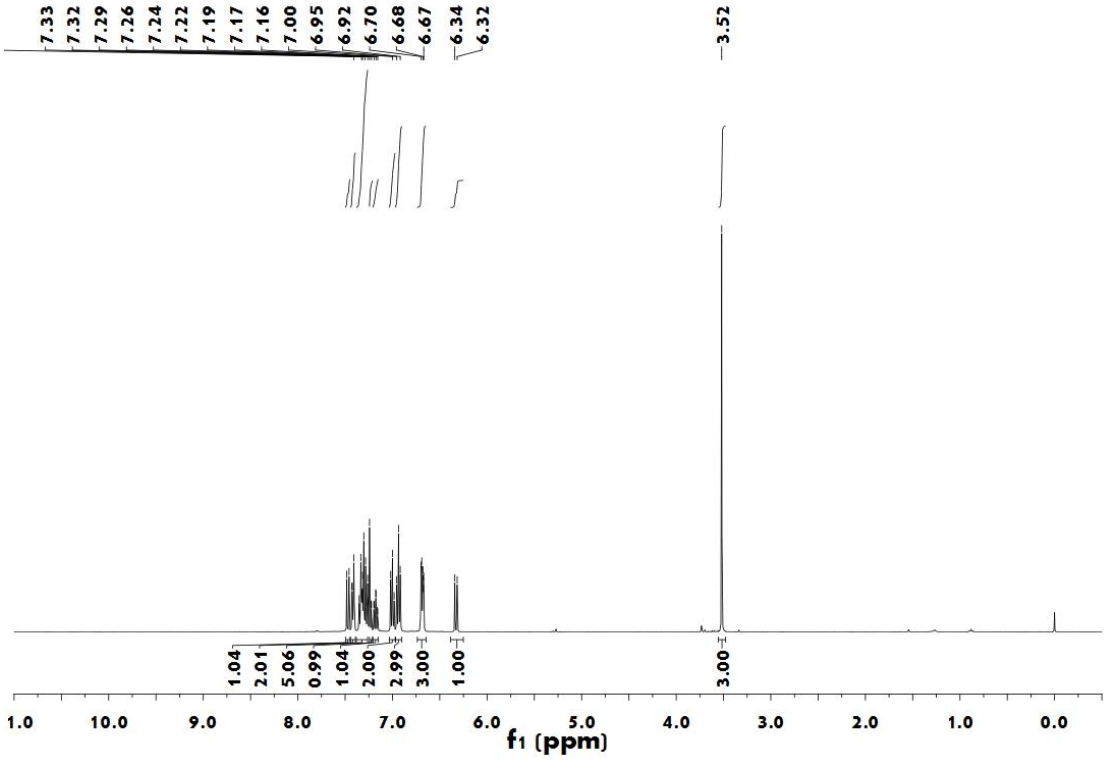

7.28
7.27
7.25
7.22
7.21
7.03
7.01
7.00
6.98
6.96
6.94
6.89
6.88
6.76
6.74
6.43
6.40

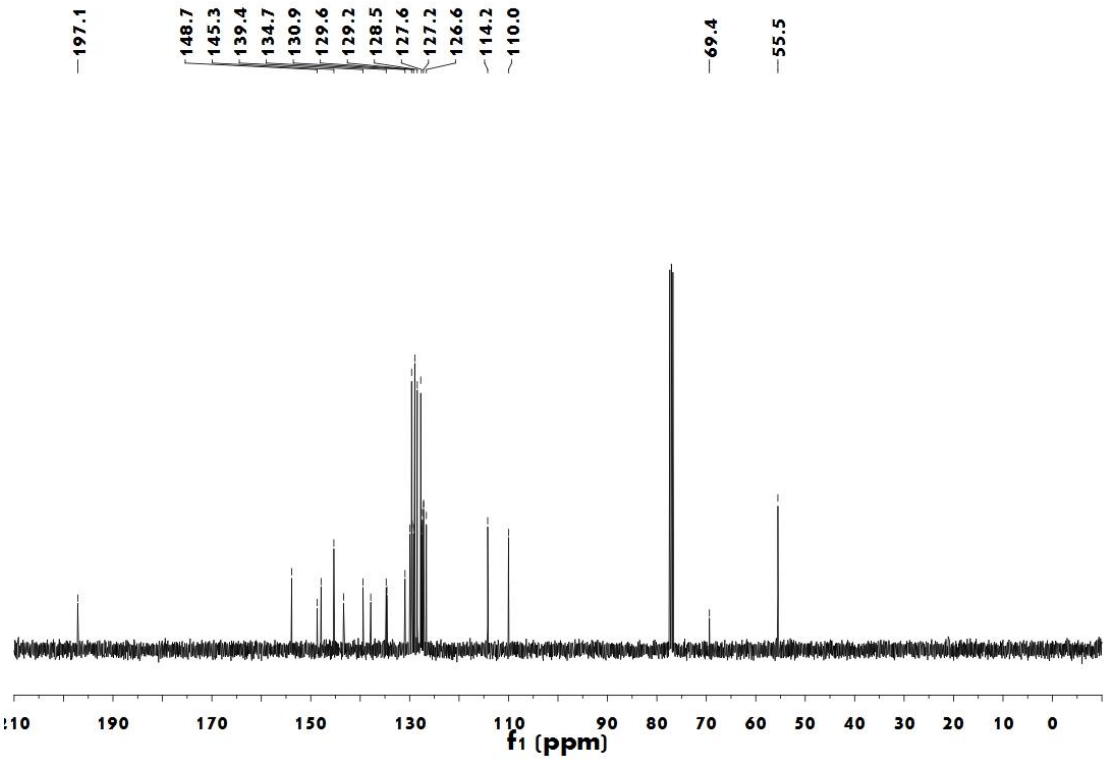

1.07
6.16
1.10
2.18
2.02
3.06
2.00
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

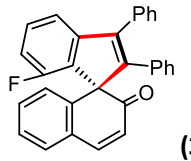

195.1
131.0
130.3
130.1
129.9
129.6
129.1
128.5
128.3
128.2
128.1
127.9
127.4
127.0
126.3
121.0
71.1
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Ph
Ph
MeO
O

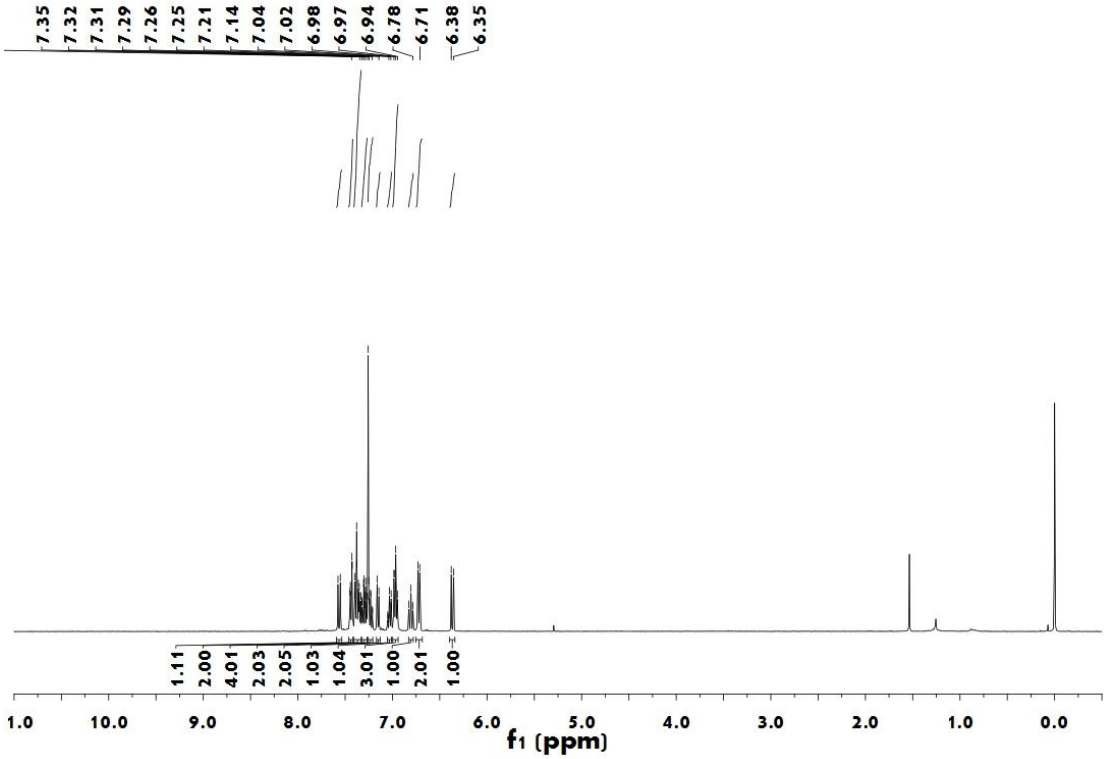
(3p)

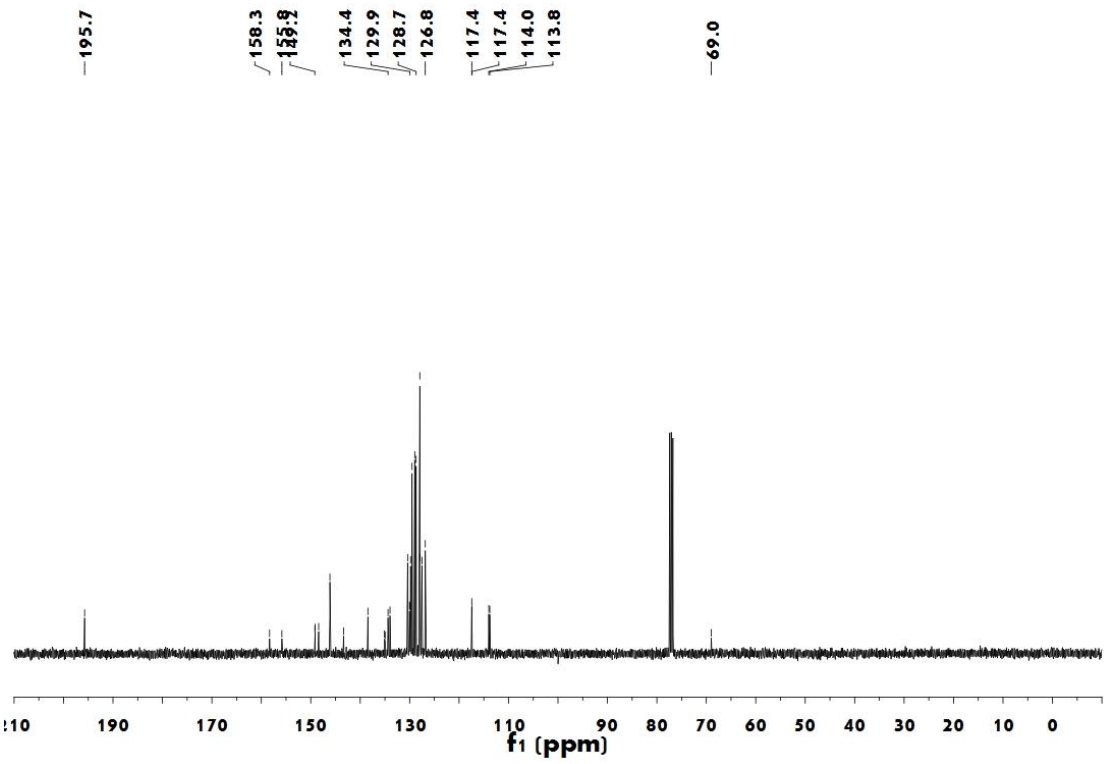

7.33
7.32
7.29
7.26
7.24
7.22
7.19
7.17
7.16
7.00
6.95
6.92
6.70
6.68
6.67
6.34
6.32
3.52
1.04
2.01
5.06
0.99
1.04
2.00
2.99
3.00
1.00
3.00
f1 (ppm)

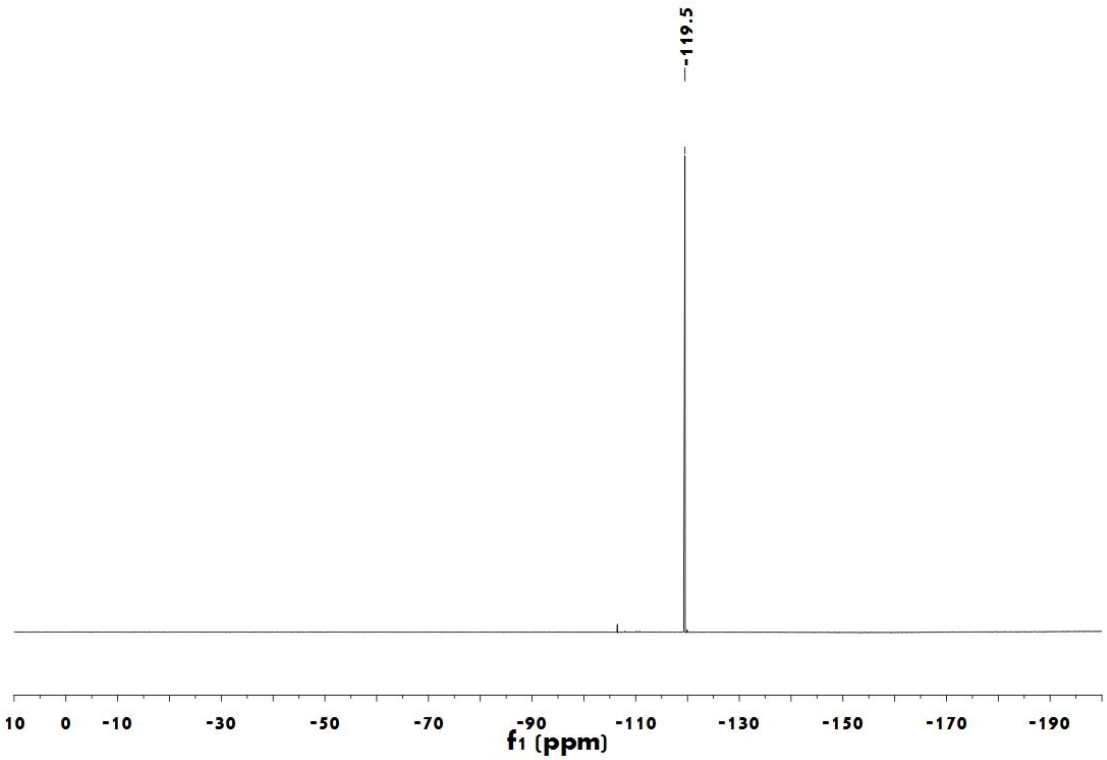

197.1
148.7
145.3
139.4
134.7
130.9
129.6
129.2
128.5
127.6
127.2
126.6
114.2
110.0
69.4
55.5
f1 (ppm)

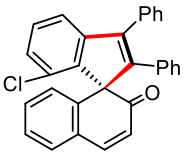

Ph
Ph
F
O

(3q)

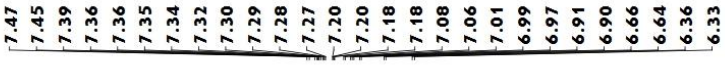

7.35
7.32
7.31
7.29
7.26
7.25
7.21
7.14
7.04
7.02
6.98
6.97
6.94
6.78
6.71
6.38
6.35
1.11
2.00
4.01
2.03
2.05
1.03
1.04
3.01
1.00
2.01
1.00
f1 (ppm)

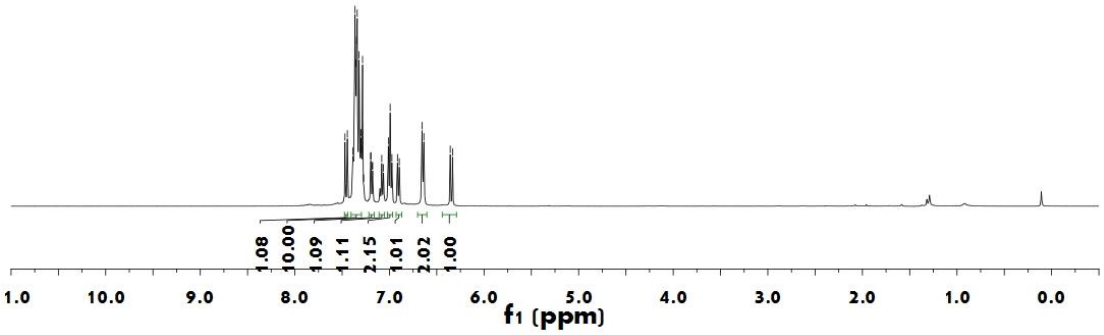

195.7
158.3
134.4
129.9
128.7
126.8
117.4
117.4
114.0
113.8
69.0
f1 (ppm)

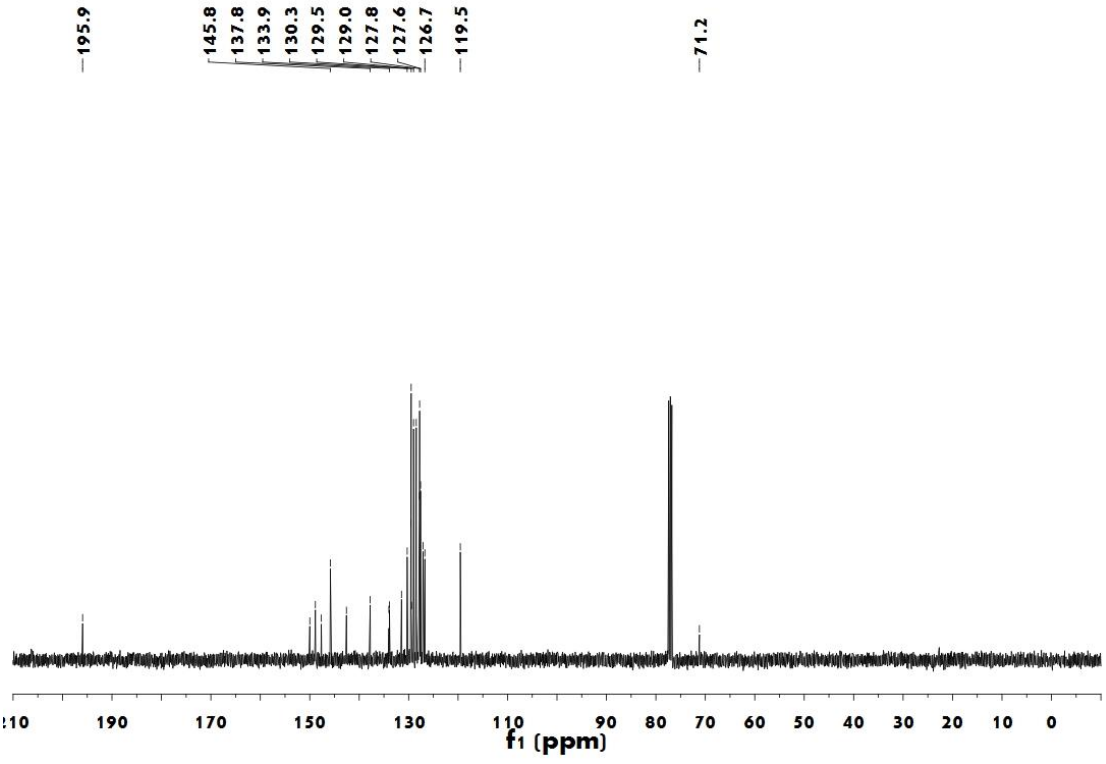

-119.5
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
f1 (ppm)

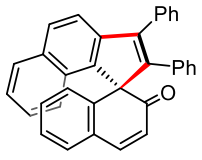

Ph
Ph
Cl
O

(3r)

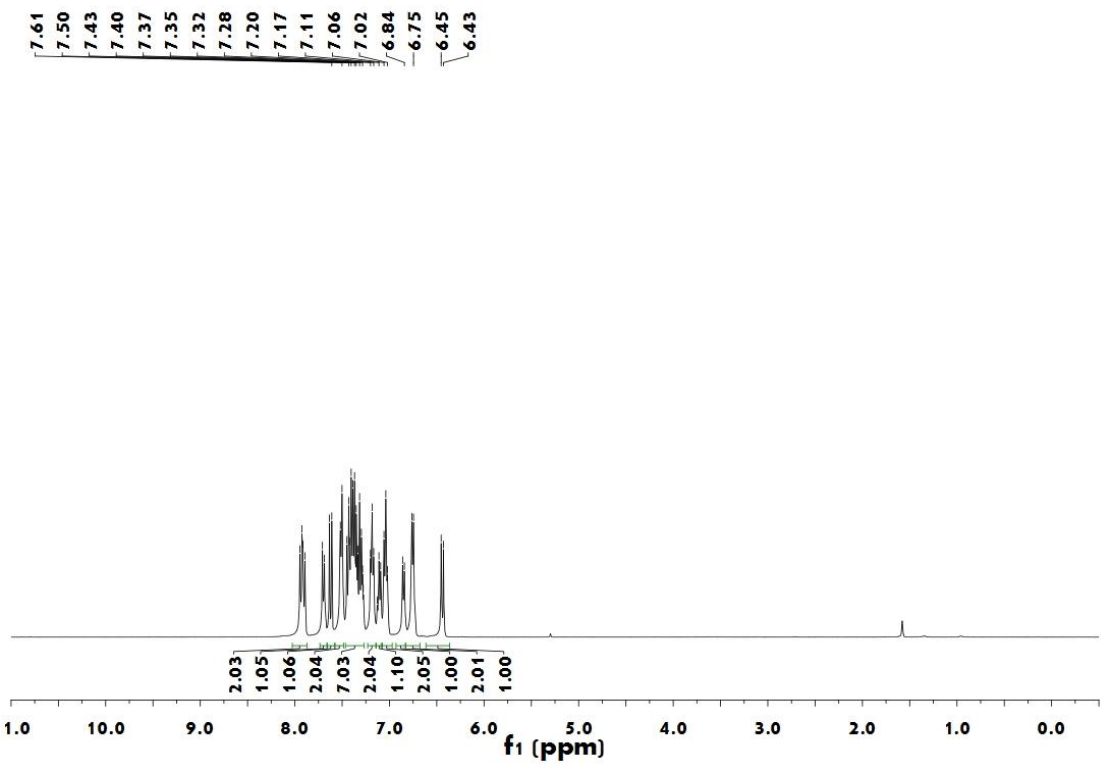

7.47
7.45
7.39
7.36
7.36
7.35
7.34
7.32
7.30
7.29
7.28
7.27
7.20
7.20
7.18
7.18
7.08
7.06
7.01
6.99
6.97
6.91
6.90
6.66
6.64
6.36
6.33

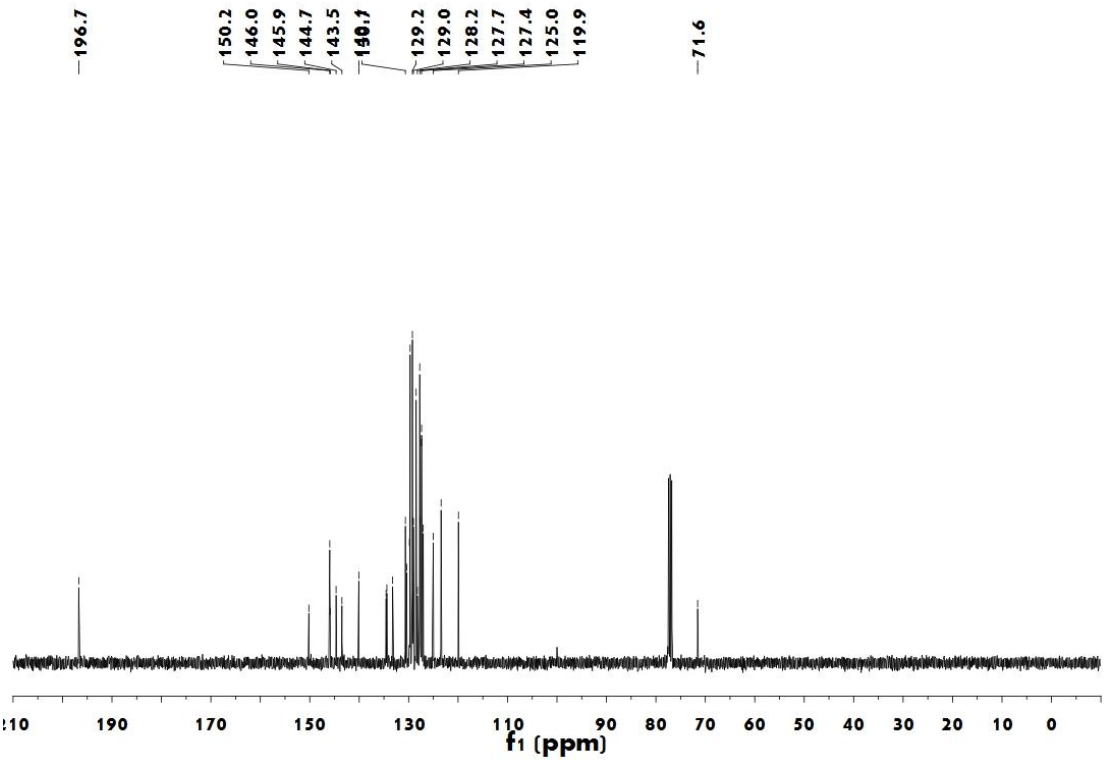

1.08
10.00
1.09
1.11
2.15
1.01
2.02
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

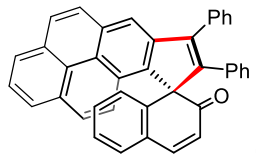

195.9
145.8
137.8
133.9
130.3
129.5
129.0
127.8
127.6
126.7
119.5
71.2
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Ph
Ph
O

(3s)

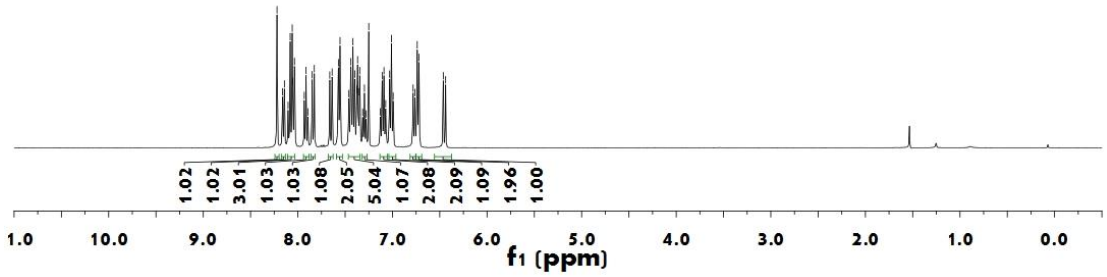

7.61
7.50
7.43
7.40
7.37
7.35
7.32
7.28
7.20
7.17
7.11
7.06
7.02
6.84
6.75
6.45
6.43
2.03
1.05
1.06
2.04
7.03
2.04
1.10
2.05
1.00
2.01
1.00
f1 (ppm)

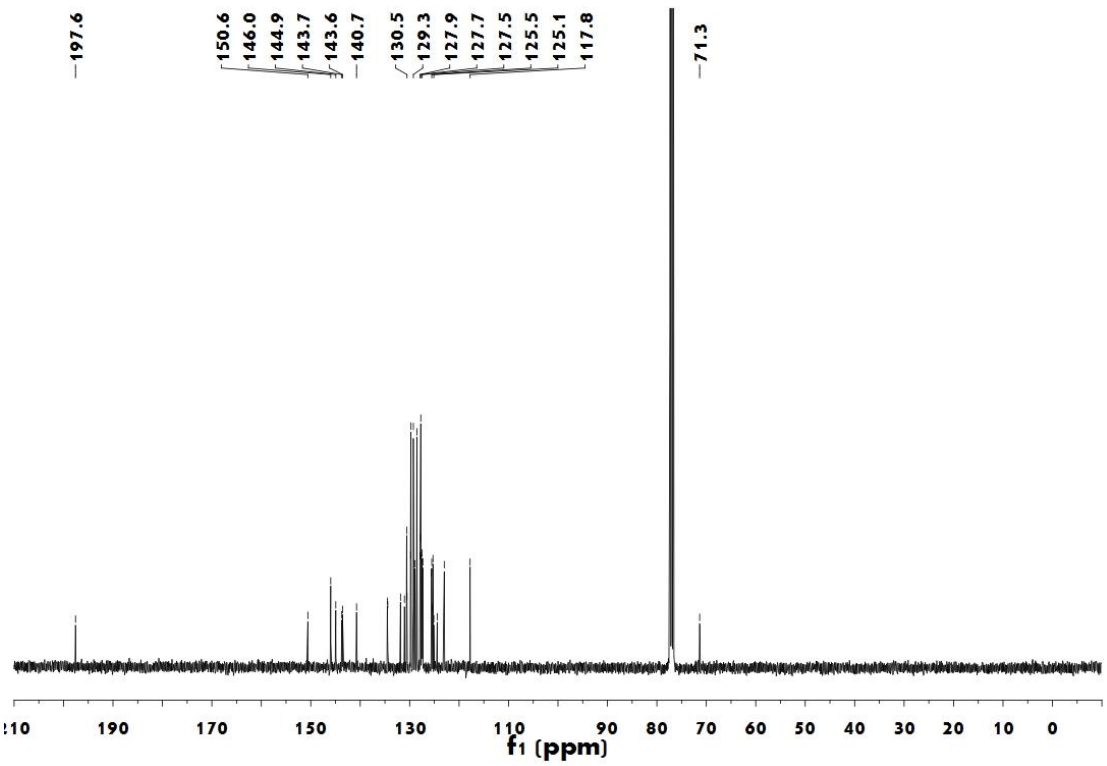

196.7
150.2
146.0
145.9
144.7
143.5
129.2
129.0
128.2
127.7
127.4
125.0
119.9
71.6
f1 (ppm)

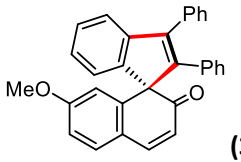

Ph
Ph
O

(3t)

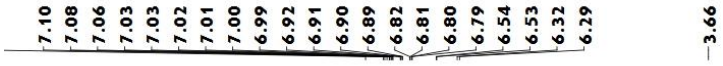

8.14
8.10
8.08
8.06
8.05
8.04
7.93
7.91
7.89
7.85
7.83
7.66
7.64
7.57
7.55
7.46
7.44
7.42
7.40
7.38
7.37
7.36
7.35
7.31
7.30
7.28
7.25
7.13
7.11
7.09
7.07
7.03
7.01
6.99
6.78
6.76
6.74
6.72
6.46
6.44


1.02
1.02
3.01
1.03
1.03
1.08
2.05
5.04
1.07
2.08
2.09
1.09
1.96
1.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


197.6
150.6
146.0
144.9
143.7
143.6
140.7
130.5
129.3
127.9
127.7
127.5
125.5
125.1
117.8
71.3
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Ph
Ph
MeO
O

(3u)


7.10
7.08
7.06
7.03
7.03
7.02
7.01
7.00
6.99
6.92
6.91
6.90
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6.82
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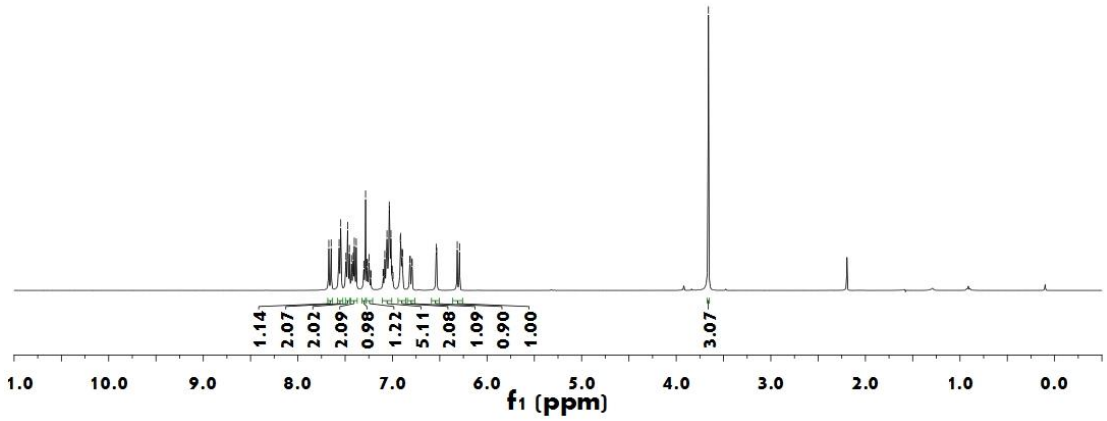
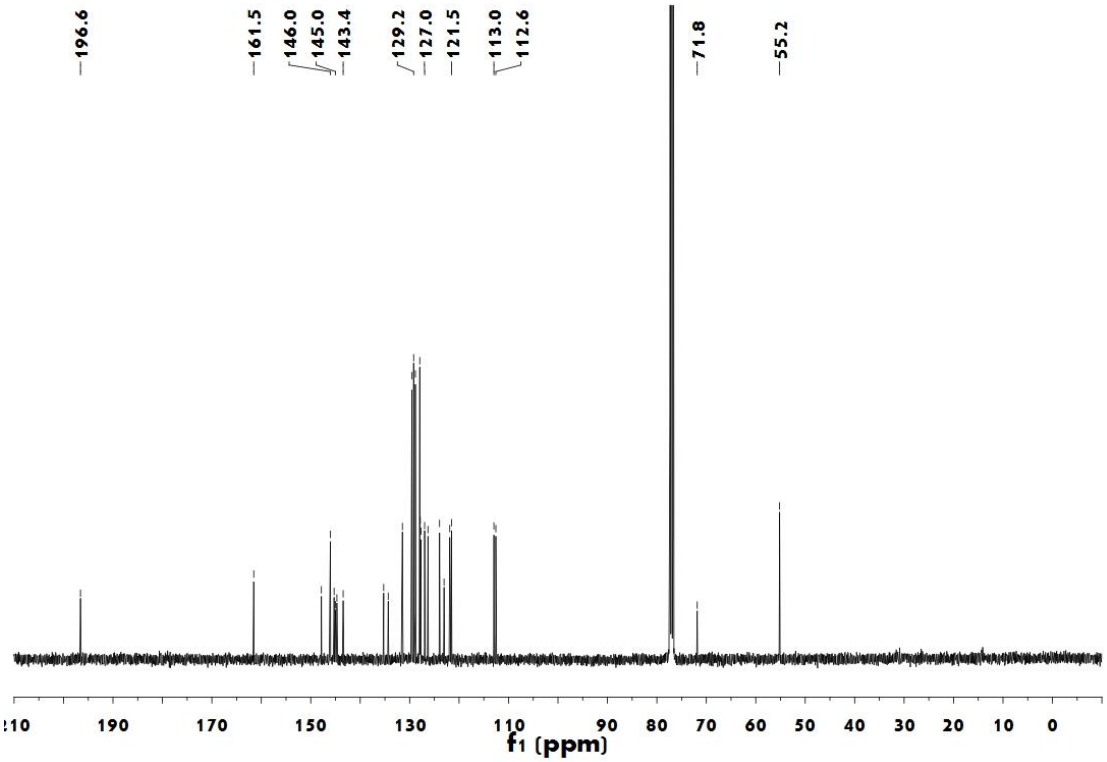
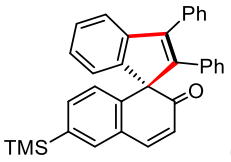
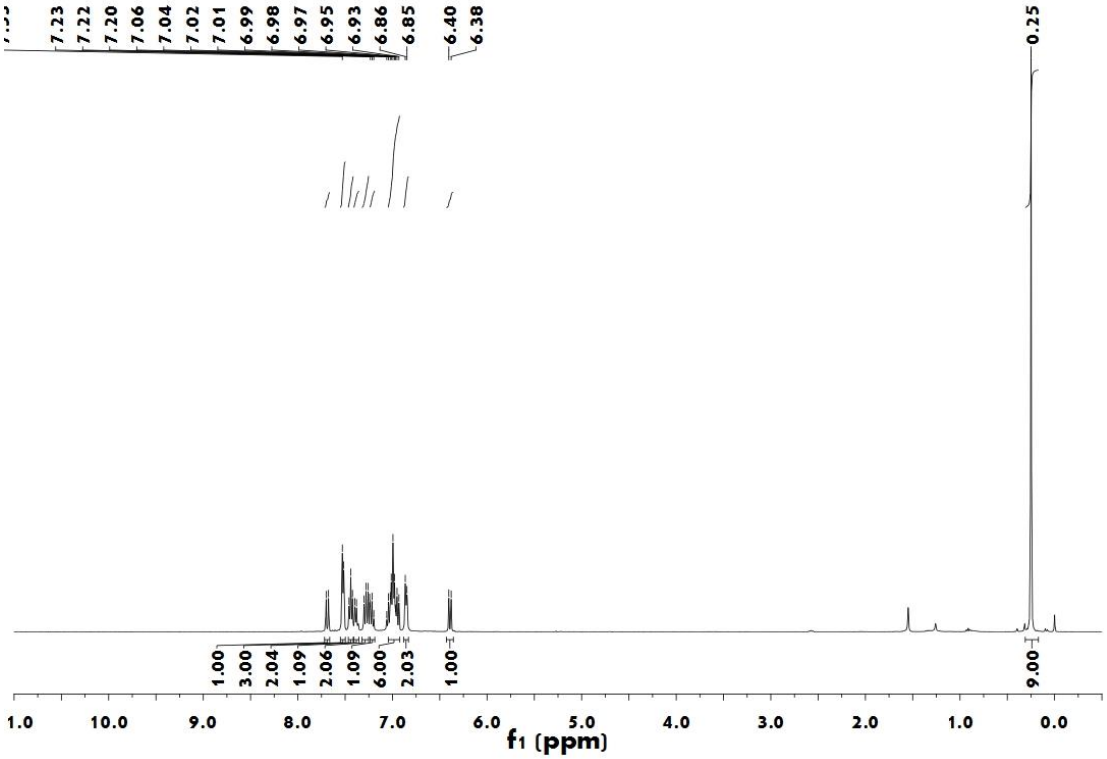
6.79
6.54
6.53
6.32
6.29
3.66

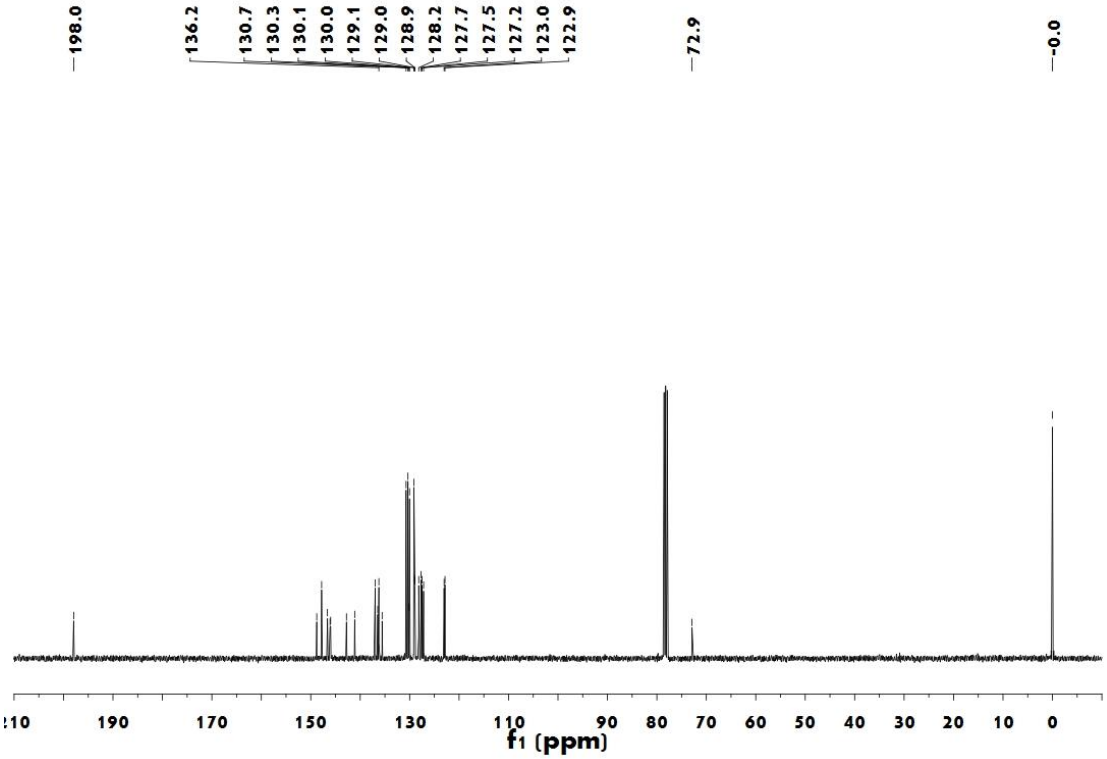

1.14
2.07
2.02
2.09
0.98
1.22
5.11
2.08
1.09
0.90
1.00
3.07
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

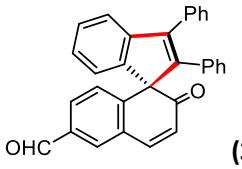

196.6
161.5
146.0
145.0
143.4
129.2
127.0
121.5
113.0
112.6
71.8
55.2
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Ph
Ph
O
TMS

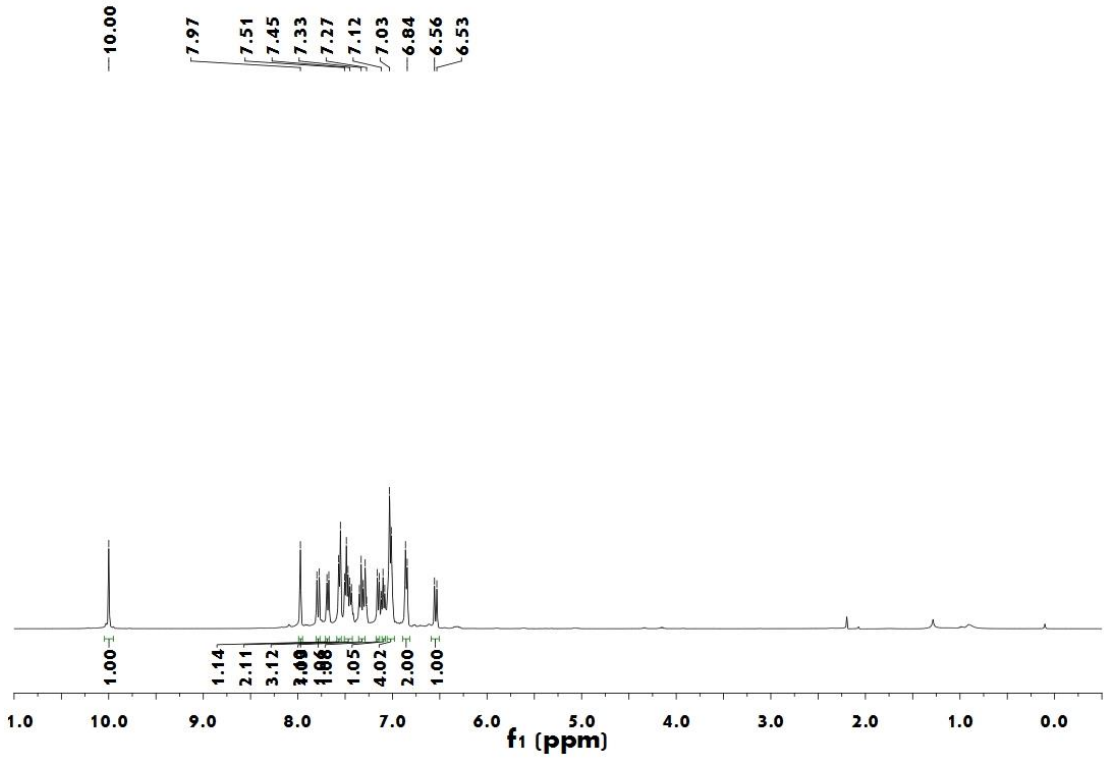
(3v)

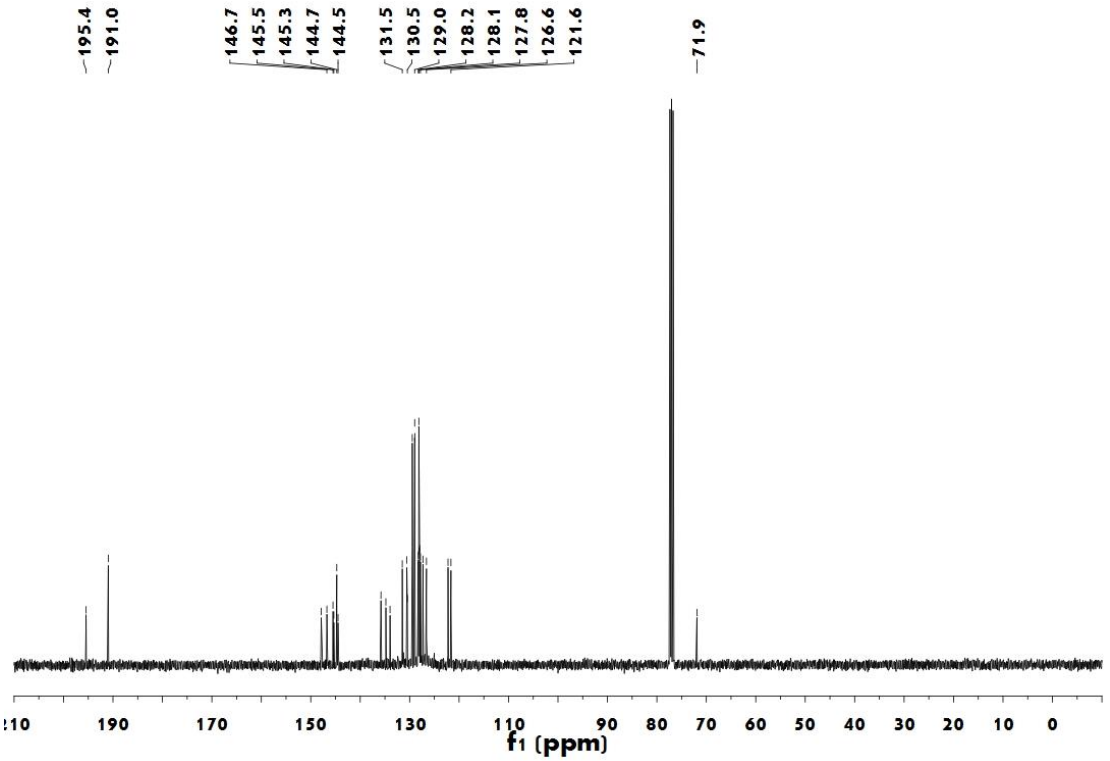

7.23
7.22
7.20
7.06
7.04
7.02
7.01
6.99
6.98
6.97
6.95
6.93
6.86
6.85
6.40
6.38
0.25
1.00
3.00
2.04
1.09
2.06
1.09
6.00
2.03
1.00
9.00
f1 (ppm)


198.0
136.2
130.7
130.3
130.1
130.0
129.1
129.0
128.9
128.2
127.7
127.5
127.2
123.0
122.9
72.9
0.0
f1 (ppm)


Ph
Ph
O
OHC

(3w)

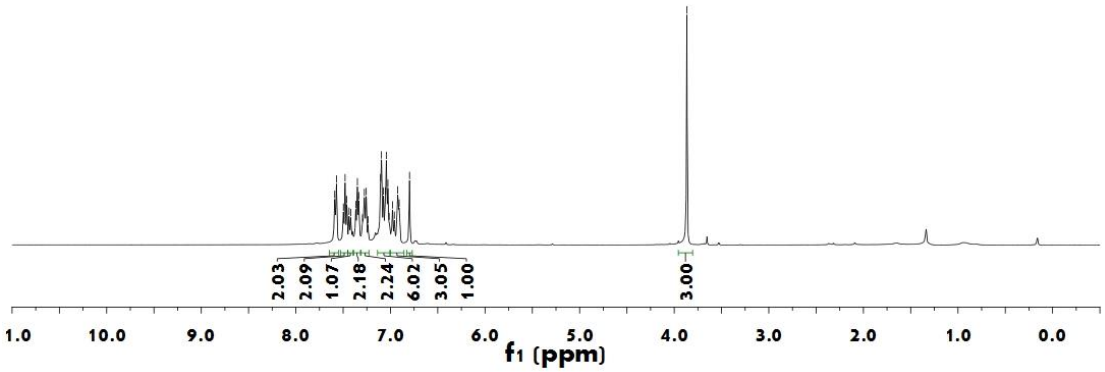

10.00
7.97
7.51
7.45
7.33
7.27
7.12
7.03
6.84
6.56
6.53
1.00
1.14
2.11
3.12
1.05
4.02
2.00
1.00
f1 (ppm)

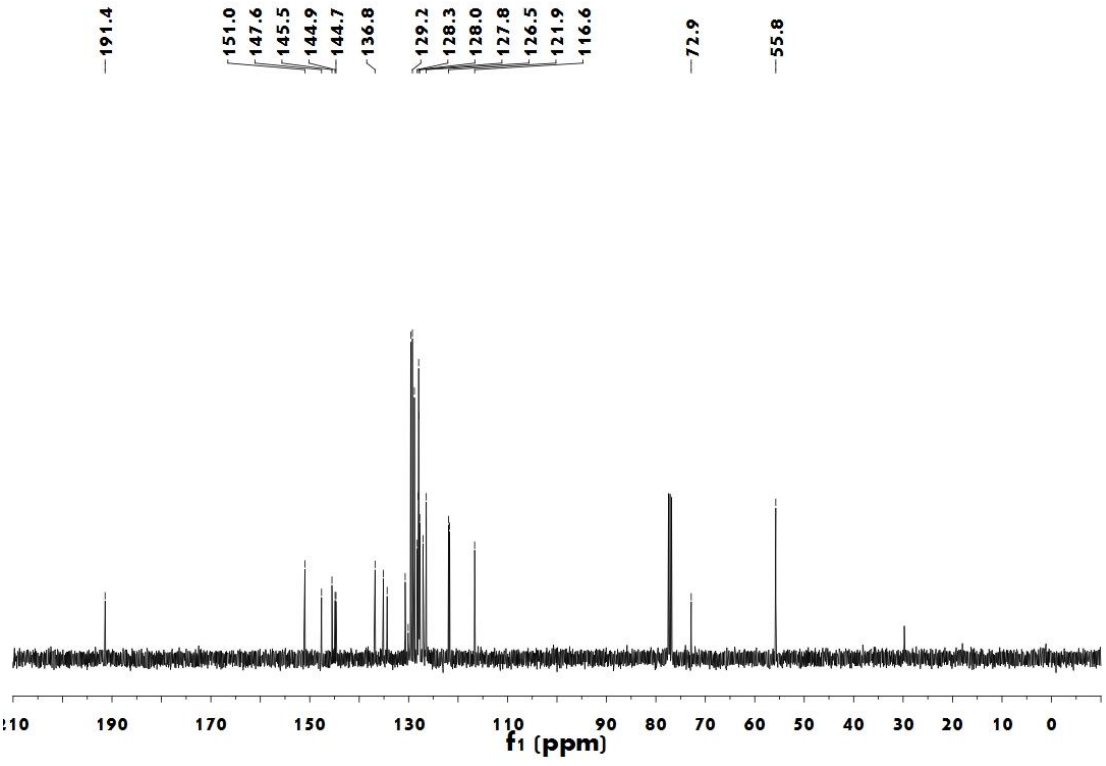

195.4
191.0
146.7
145.5
145.3
144.7
144.5
131.5
130.5
129.0
128.2
128.1
127.8
126.6
121.6
71.9
f1 (ppm)

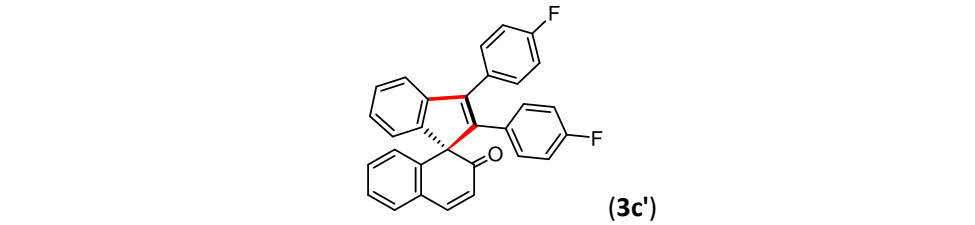

Ph
Ph
O
OMe

(3x)

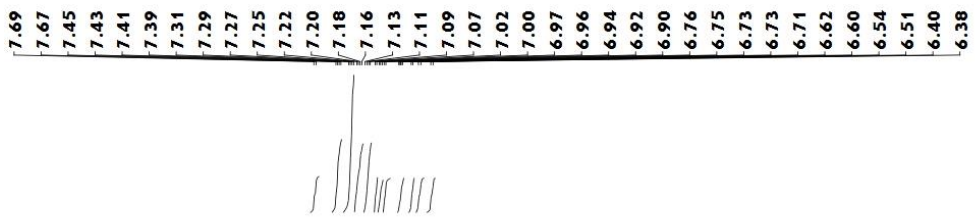

7.48
7.37
7.35
7.33
7.30
7.28
7.26
7.24
7.10
7.10
7.07
7.05
7.04
7.03
7.01
6.98
6.96
6.92
6.91
6.80
3.87

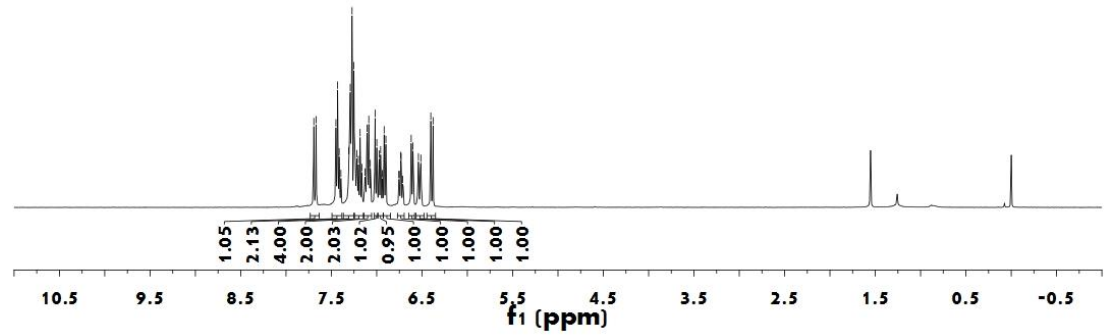

2.03
2.09
1.07
2.18
2.24
6.02
3.05
1.00
3.00
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

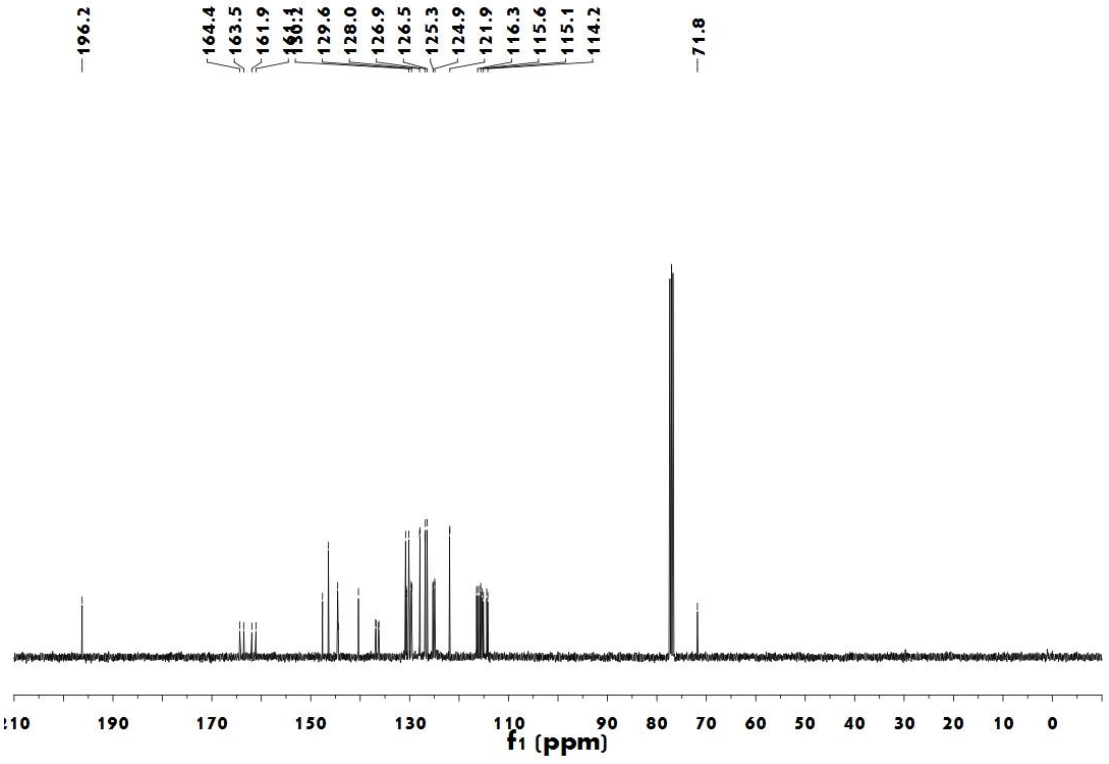

191.4
151.0
147.6
145.5
144.9
144.7
136.8
129.2
128.3
128.0
127.8
126.5
121.9
116.6
72.9
55.8
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

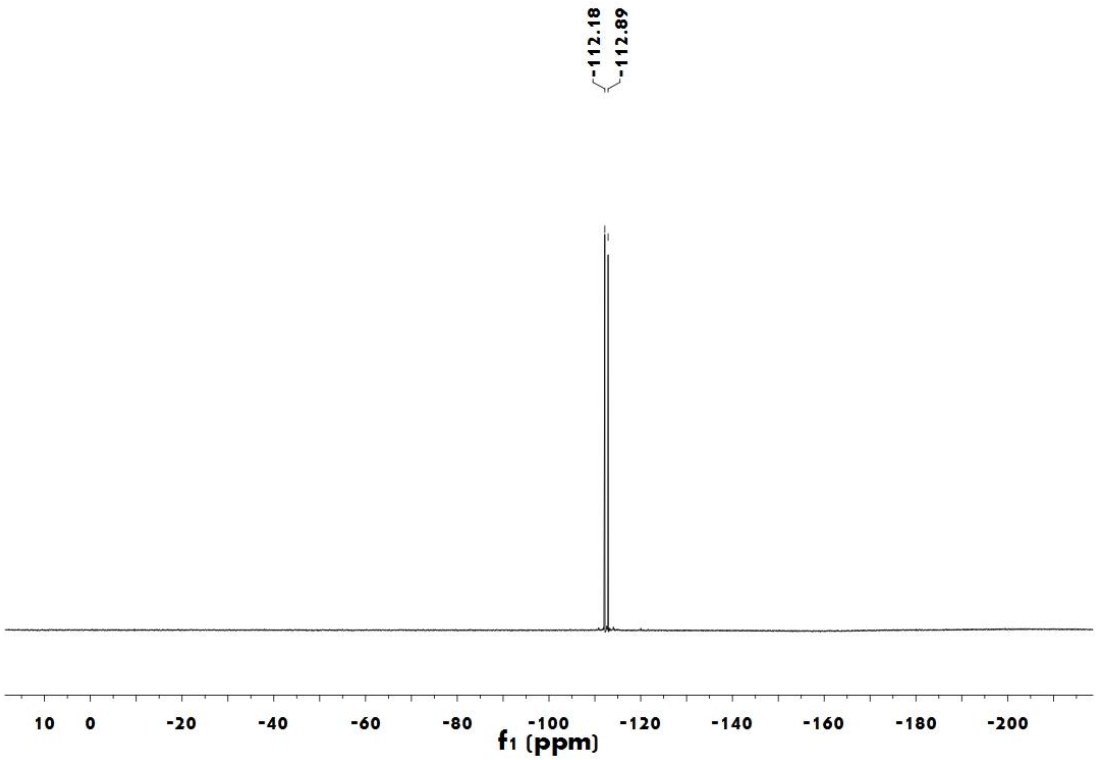

F
F
O

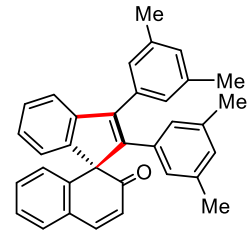
(3c')


7.69
7.67
7.45
7.43
7.41
7.39
7.31
7.29
7.27
7.25
7.22
7.20
7.18
7.16
7.13
7.11
7.09
7.07
7.02
7.00
6.97
6.96
6.94
6.92
6.90
6.76
6.75
6.73
6.73
6.71
6.62
6.60
6.54
6.51
6.40
6.38

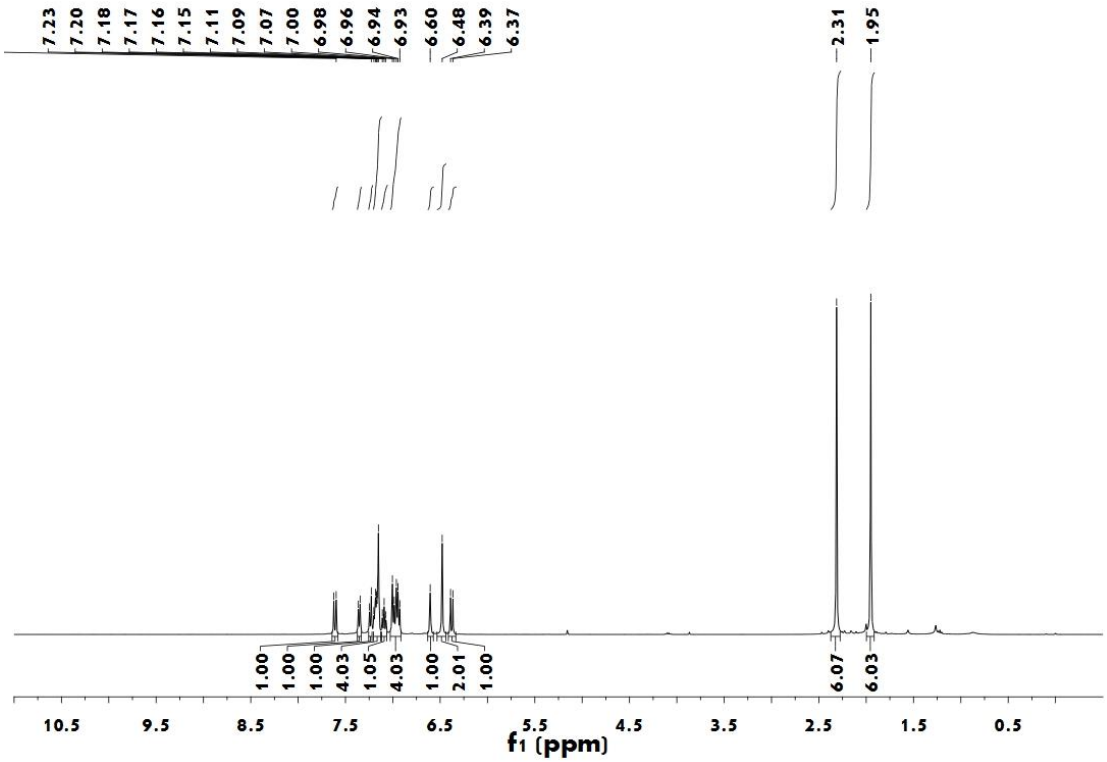

1.05
2.13
4.00
2.00
2.03
1.02
0.95
1.00
1.00
1.00
1.00
1.00
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

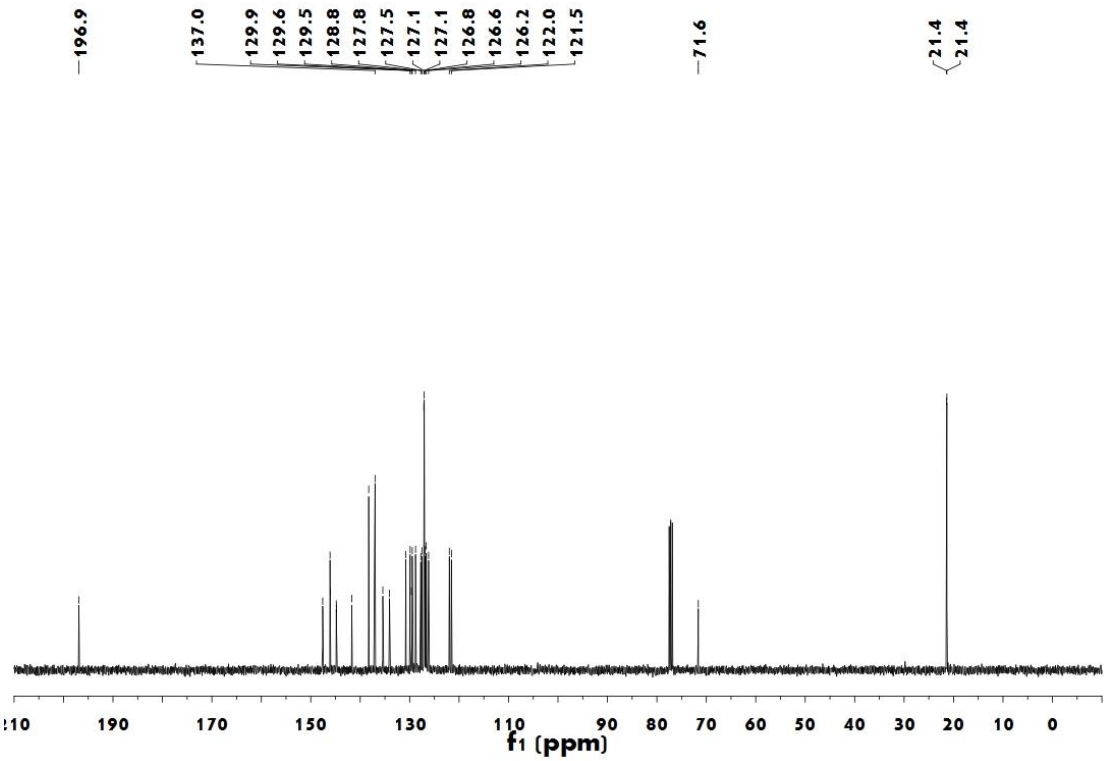

196.2
164.4
163.5
161.9
129.6
128.0
126.9
126.5
125.3
124.9
121.9
116.3
115.6
115.1
114.2
71.8
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

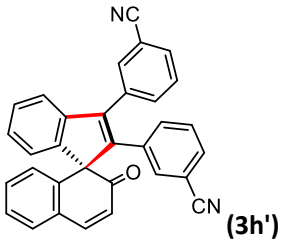

-112.18
-112.89
10
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)


Me
Me
Me
O
Me

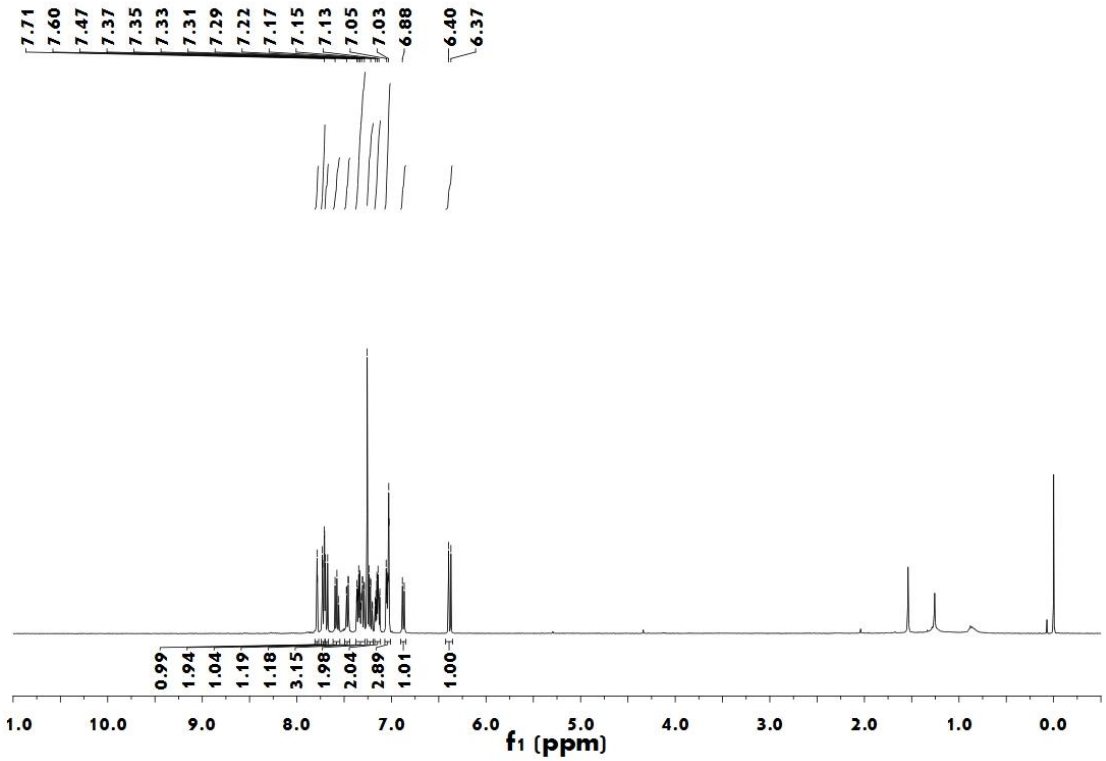
(3f')

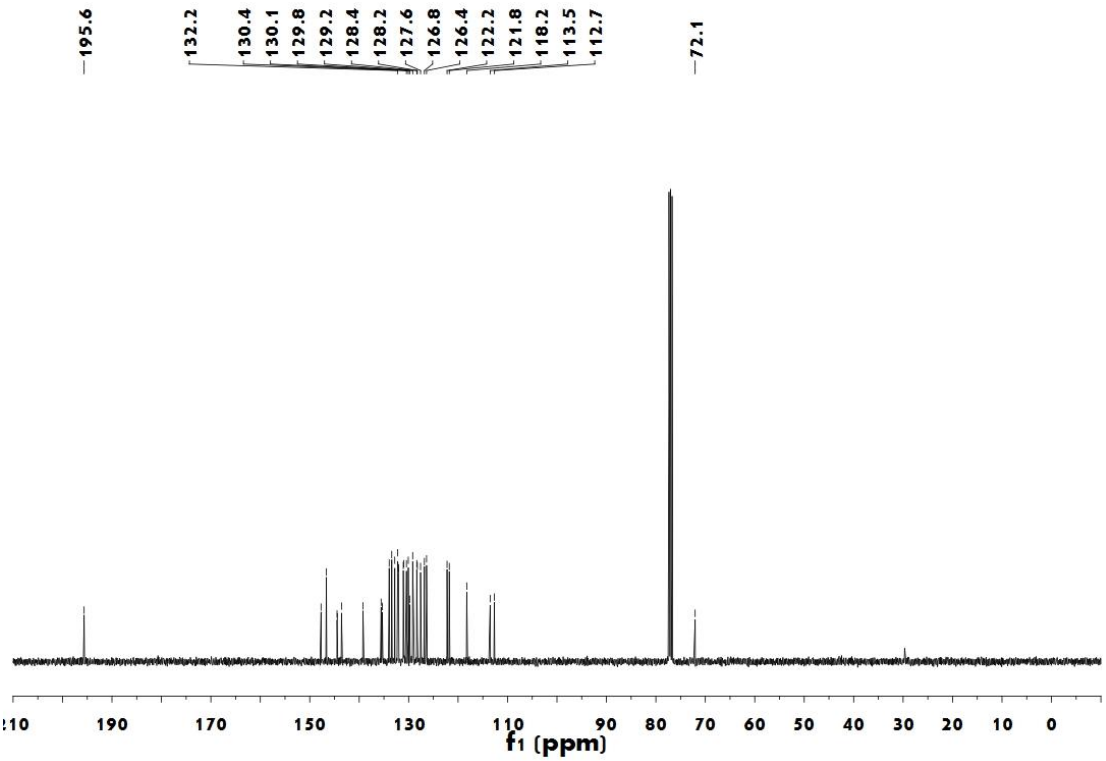

7.23
7.20
7.18
7.17
7.16
7.15
7.11
7.09
7.07
7.00
6.98
6.96
6.94
6.93
6.60
6.48
6.39
6.37
2.31
1.95
1.00
1.00
1.00
4.03
1.05
4.03
1.00
2.01
1.00
6.07
6.03
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

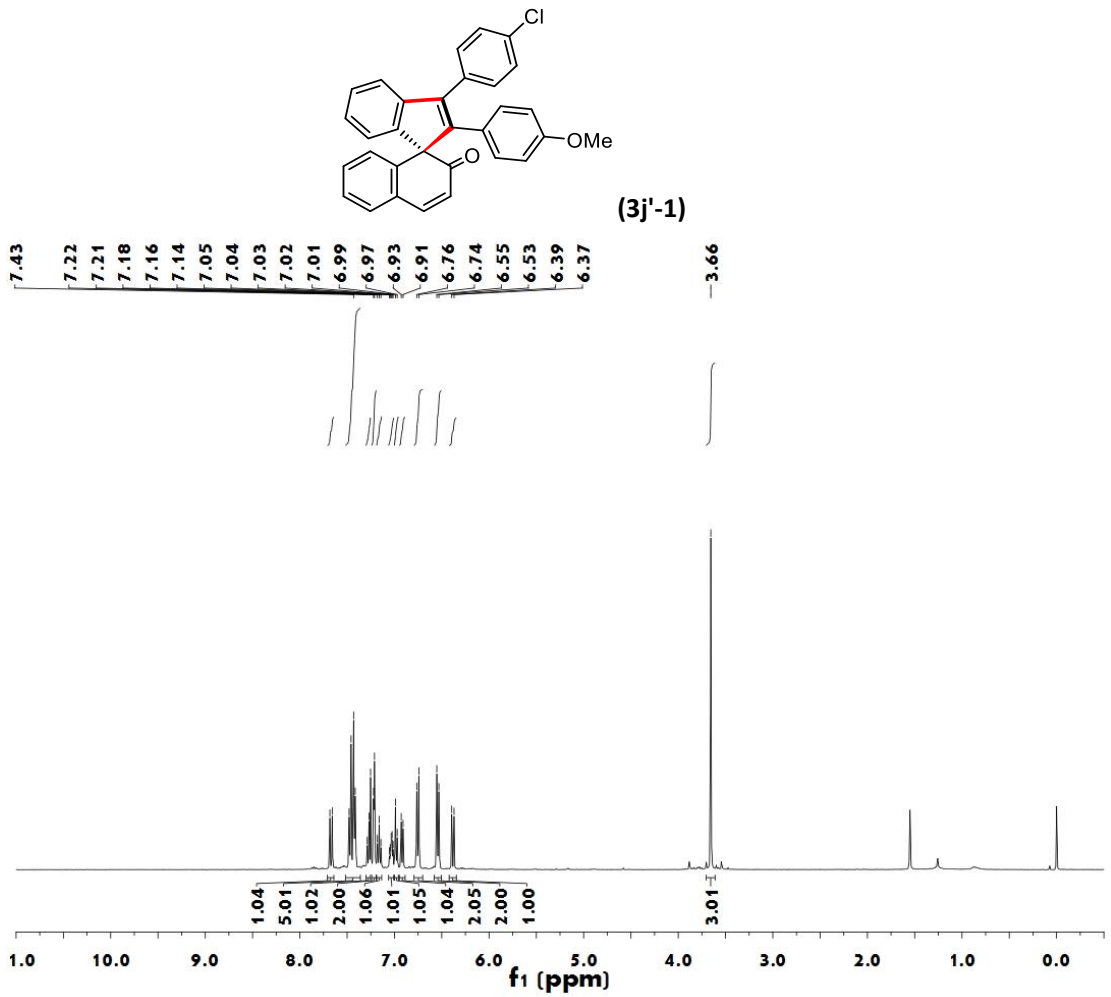

196.9
137.0
129.9
129.6
129.5
128.8
127.8
127.5
127.1
127.1
126.8
126.6
126.2
122.0
121.5
71.6
21.4
21.4
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

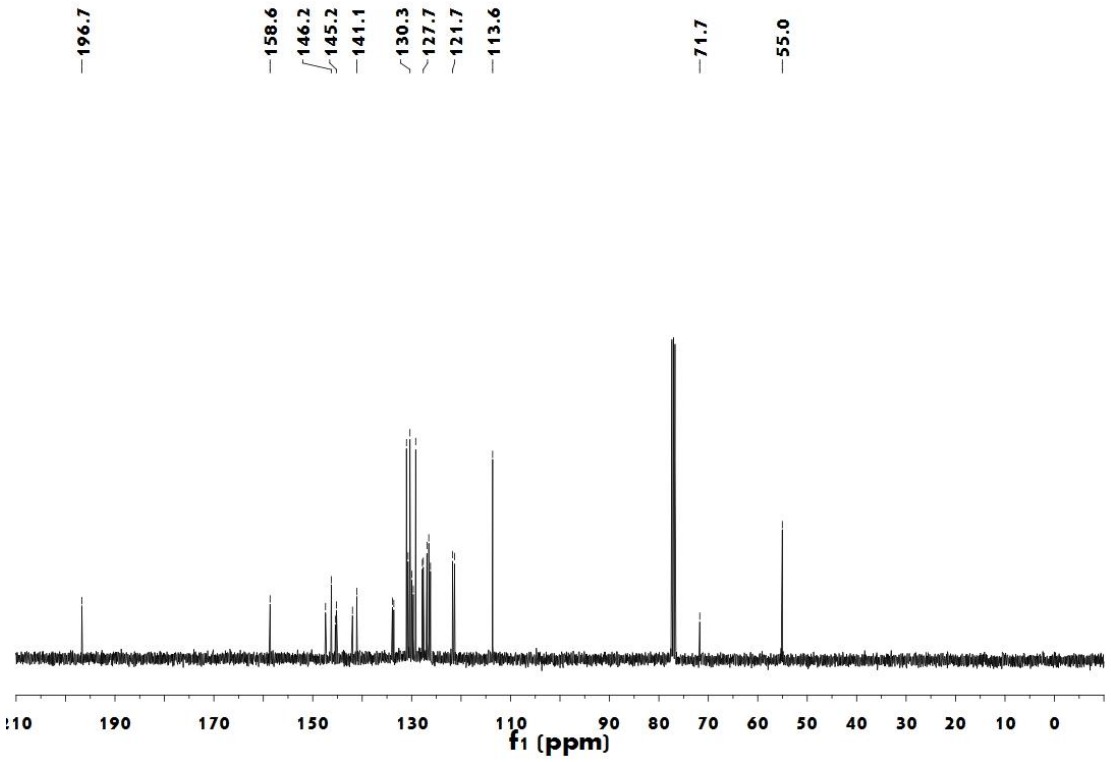

NC
O
CN

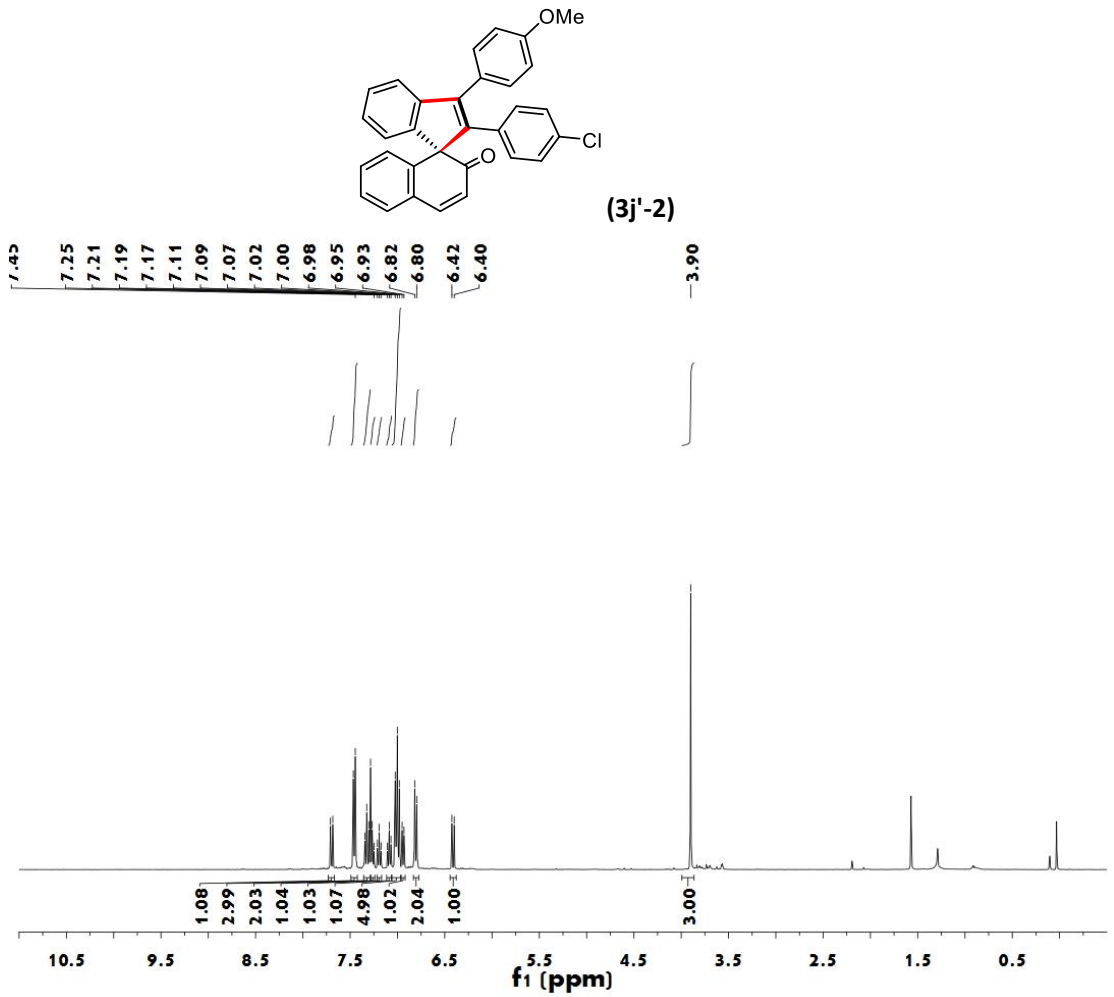
(3h')

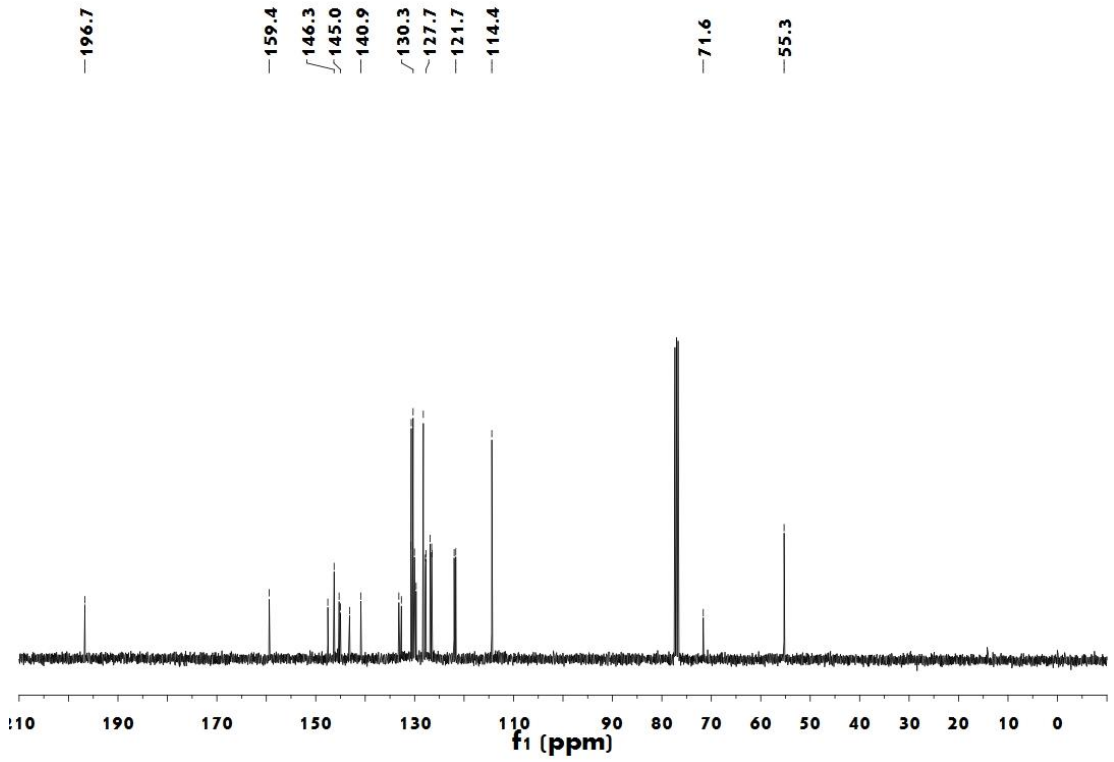

7.71
7.60
7.47
7.37
7.35
7.33
7.31
7.29
7.22
7.17
7.15
7.13
7.05
7.03
6.88
6.40
6.37
0.99
1.94
1.04
1.19
1.18
3.15
1.98
2.04
2.89
1.01
1.00
f1 (ppm)

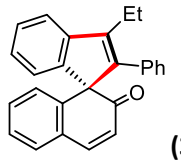

195.6
132.2
130.4
130.1
129.8
129.2
128.4
128.2
127.6
126.8
126.4
122.2
121.8
118.2
113.5
112.7
72.1
f1 (ppm)


Cl
OMe
O
(3j'-1)
7.43
7.22
7.21
7.18
7.16
7.14
7.05
7.04
7.03
7.02
7.01
6.99
6.97
6.93
6.91
6.76
6.74
6.55
6.53
6.39
6.37
3.66
1.04
5.01
1.02
2.00
1.06
1.01
1.05
1.04
2.05
2.00
1.00
3.01
f1 (ppm)

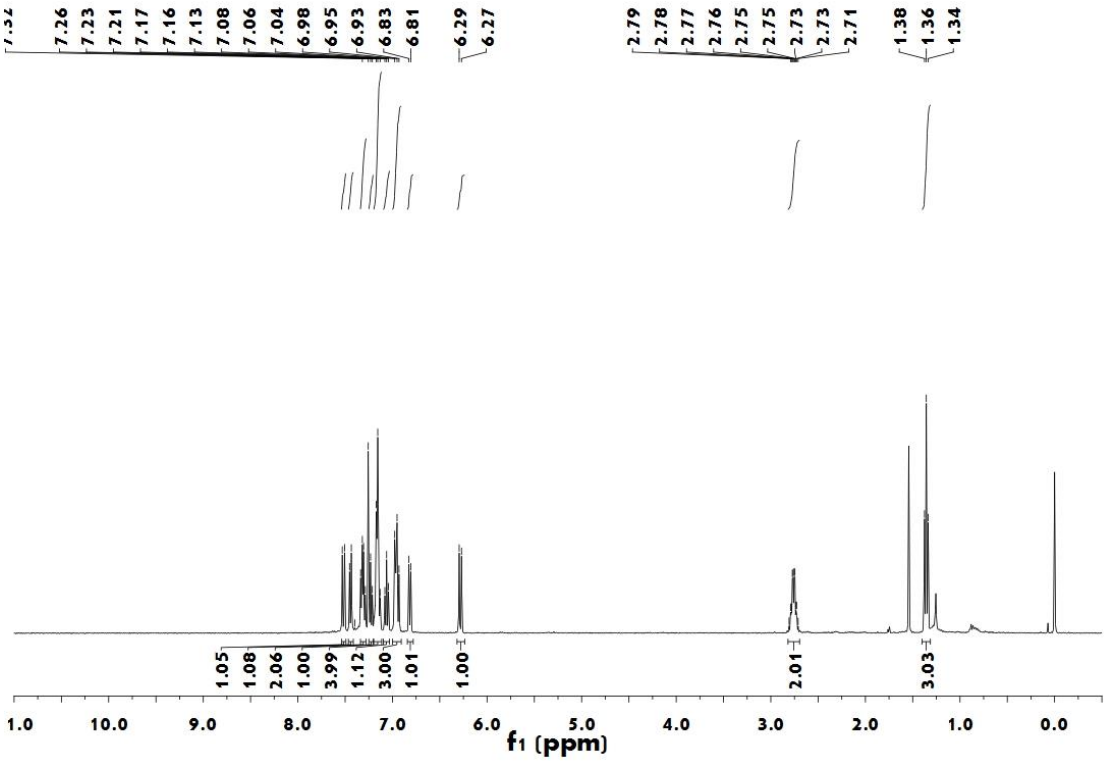

196.7
158.6
146.2
145.2
141.1
130.3
127.7
121.7
113.6
71.7
55.0
f1 (ppm)

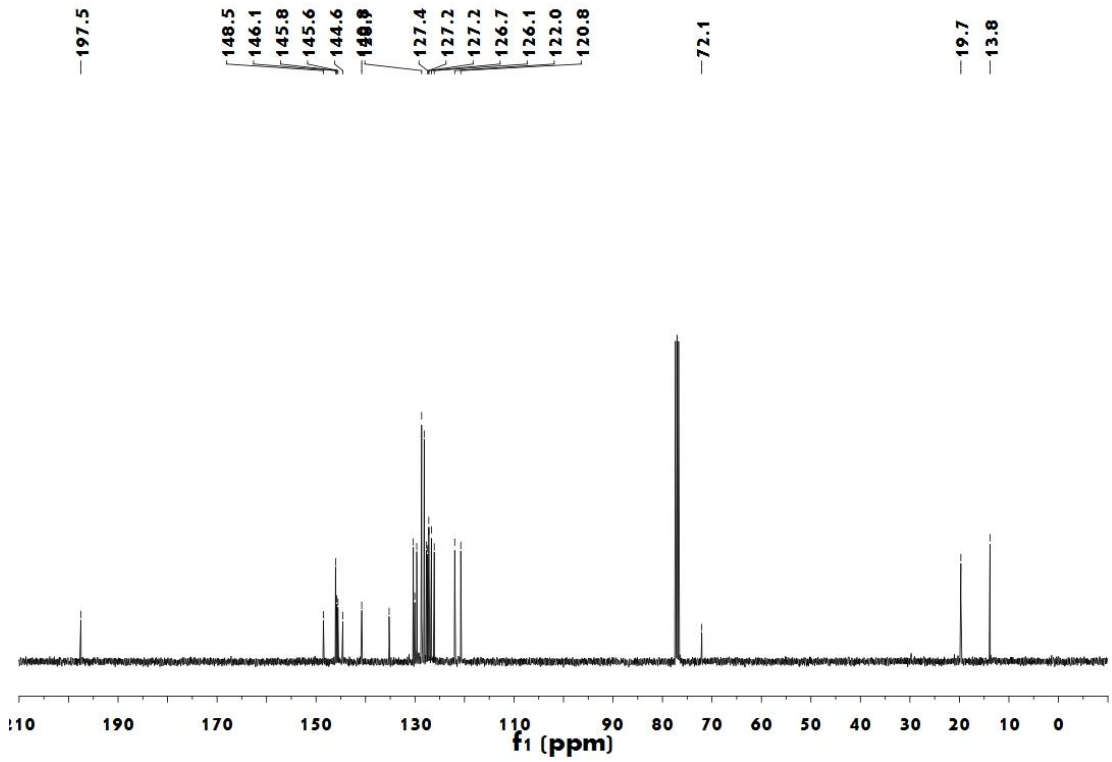

OMe
Cl
O
(3j'-2)
7.25
7.21
7.19
7.17
7.11
7.09
7.07
7.02
7.00
6.98
6.95
6.93
6.82
6.80
6.42
6.40
3.90
1.08
2.99
2.03
1.04
1.03
1.07
4.98
1.02
2.04
1.00
3.00
f1 (ppm)


196.7
159.4
146.3
145.0
140.9
130.3
127.7
121.7
114.4
71.6
55.3
f1 (ppm)


Et
Ph
O

(3k')


7.26
7.23
7.21
7.17
7.16
7.13
7.08
7.06
7.04
6.98
6.95
6.93
6.83
6.81
6.29
6.27
2.79
2.78
2.77
2.76
2.75
2.75
2.73
2.73
2.71
1.38
1.36
1.34
1.05
1.08
2.06
1.00
3.99
1.12
3.00
1.01
1.00
2.01
3.03
1.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


197.5
148.5
146.1
145.8
145.6
144.6
127.4
127.2
127.2
126.7
126.1
122.0
120.8
72.1
19.7
13.8
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)