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Copper-Catalyzed Three Component Reaction of Aryl Acetylene, Sulfonyl Azide and Enaminone to Form Iminolactone via 6 π Electrocyclization

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A. General information

Unless otherwise specified, all reactions were carried out under a nitrogen atmosphere, with dry solvents in anhydrous conditions. THF, ethyl acetate (Ea), toluene, dichloromethane, 1,2-dichloroethane and acetonitrile were dried by activated molecular sieve (4 Å). All chemicals were used without further purification as commercially available unless otherwise noted. Thin-layer chromatography (TLC) was performed on silica gel plates (60F-254) using UV-light (254 and 365 nm). Flash chromatography was conducted on silica gel (200–300 mesh). Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV400 MHz spectrometer. Chemical shifts were reported in parts per million (ppm). Infrared spectra (IR) were recorded by an ATR module and absorption bands are given in wavenumbers (cm^{-1}). High resolution mass spectra (HRMS) were recorded on a Waters TOFMS GCT Premier using ESI ionization. Petroleum ether (PE) refers to the fraction with boiling point in the range 60–90 ^{o}C.

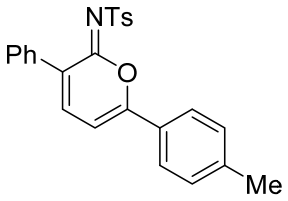
Enaminone **1** was prepared from corresponding ketone with N,N-dimethylformamide dimethyl acetal in toluene under reflux [1]. Sulfonyl azide **3** was prepared from corresponding sulfonyl chloride and sodium azide according to the reported procedure [2].

B. General procedure for the three-component reaction

To a flame dried round bottle flask with a magnetic stirring bar were added CuI (7.6 mg, 0.04 mmol, 10 mol %), PPh_3 (21.0 mg, 0.08 mmol, 20 mol %) and enaminone **1** (0.4 mmol), followed by the addition of 1,2-dichloroethane (2 mL) and TEA (28 μL, 0.2 mmol, 0.5 equiv.) under nitrogen atmosphere at room temperature. Aryl acetylene **2** (1 mmol, 2.5 equiv.) and sulfonyl azide **3** (1 mmol, 2.5 equiv.) was then added, and the resulting mixture was stirred overnight. After the completely consumption of enaminone 1 monitored by TLC, the solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel with PE/Ea as eluent to afford product **4** or **5**.

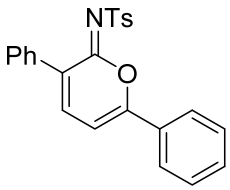
C. Analytical data of iminolactone 4 and 5

4-methyl-N-(3-phenyl-6-(p-tolyl)-2H-pyran-2-ylidene)benzenesulfonamide **4a**



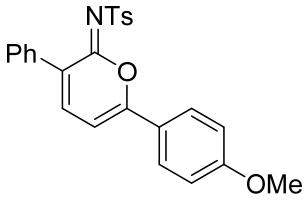
The title compound was prepared according to the general procedure to afford **4a** (118.0 mg, 71% yield) as a yellow powder. M.p. 138.1 – 142.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.65 – 7.57 (m, 2H), 7.50 (d, *J* = 7.4 Hz, 1H), 7.38 – 7.35 (m, 3H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 6.89 (d, *J* = 7.4 Hz, 1H), 2.43 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 158.9, 142.6, 142.1, 141.1, 139.9, 134.3, 130.0, 129.2, 128.8, 128.7, 128.2, 127.3, 127.3, 126.5, 126.0, 103.2, 21.6, 21.5 ppm; IR (KBr) ν 3085, 3030, 2958, 2860, 1639, 1511, 1446, 1301 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_3S$ $[M+H]^+$ = 416.1315, found = 416.1327.

N-(3,6-diphenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4b**



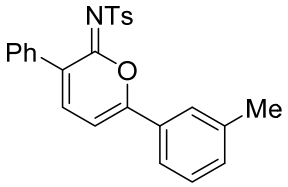
The title compound was prepared according to the general procedure to afford **4b** (126.7 mg, 79% yield) as a yellow powder. M.p. 168.6 – 172.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.00 (m, 2H), 7.90 (d, *J* = 8.1 Hz, 2H), 7.63 – 7.58 (m, 2H), 7.54 – 7.47 (m, 4H), 7.40 – 7.33 (m, 3H), 7.22 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 7.4 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 158.8, 142.6, 140.7, 139.8, 134.2, 131.4, 130.1, 129.2, 129.2, 128.8, 128.8, 128.2, 128.0, 126.5, 126.0, 103.6, 21.5 ppm; IR (KBr) ν 3108, 3065, 2953, 2850, 1641, 1510, 1446, 1300 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{20}NO_3S$ $[M+H]^+$ = 402.1158, found = 402.1161.

N-(6-(4-methoxyphenyl)-3-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4c**



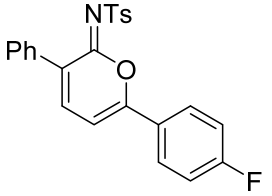
The title compound was prepared according to the general procedure to afford **4c** (110.5 mg, 64% yield) as a yellow powder. M.p. 129.2 – 132.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.7 Hz, 2H), 7.90 (d, *J* = 7.6 Hz, 2H), 7.62 – 7.57 (m, 2H), 7.47 (d, *J* = 7.4 Hz, 1H), 7.42 – 7.32 (m, 3H), 7.23 (d, *J* = 7.8 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 2H), 6.81 (d, *J* = 7.4 Hz, 1H), 3.86 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 160.1, 159.0, 142.5, 141.2, 140.0, 134.4, 129.2, 128.7, 128.6, 128.2, 127.9, 126.5, 122.6, 114.7, 102.3, 55.5, 21.5 ppm; IR (KBr) ν 3055, 3025, 2960, 2835, 1634, 1511, 1446, 1146 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_4S$ $[M+H]^+$ = 432.1264, found = 432.1277.

4-methyl-N-(3-phenyl-6-(m-tolyl)-2H-pyran-2-ylidene)benzenesulfonamide **4d**



The title compound was prepared according to the general procedure to afford **4d** (121.3 mg, 73% yield) as a yellow powder. M.p. 128.3 – 131.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* =8.2 Hz, 2H), 7.86 (d, *J* =7.8 Hz, 1H), 7.80 (s, 1H), 7.64 – 7.58 (m, 2H), 7.50 (d, *J* =7.4 Hz, 1H), 7.44 – 7.34 (m, 4H), 7.31 (d, *J* =7.6 Hz, 1H), 7.22 (d, *J* =8.1 Hz, 2H), 6.91 (d, *J* =7.4 Hz, 1H), 2.45 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 158.9, 142.5, 140.7, 139.9, 138.9, 134.3, 132.3, 130.0, 129.1, 128.8, 128.2, 127.8, 126.5, 123.4, 103.6, 21.6, 21.5 ppm; IR (KBr) ν 3098, 3045, 2915, 2853, 1636, 1514, 1446,1311 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_3S$ $[M+H]^+$ = 416.1315, found = 416.1330.

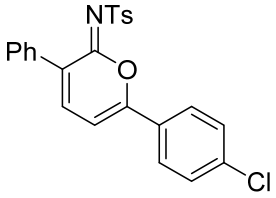
N-(6-(4-fluorophenyl)-3-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4e**



The title compound was prepared according to the general procedure to afford **4e** (72.1 mg, 43% yield) as a yellow

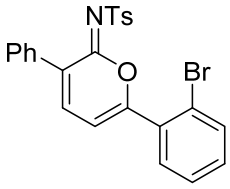
powder. M.p. 192.7 – 195.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* =7.9, 5.4 Hz, 2H), 7.91 (d, *J* =7.9 Hz, 2H), 7.58 (d, *J* =3.7 Hz, 2H), 7.48 (d, *J* =7.3 Hz, 1H), 7.40 – 7.34 (m, 3H), 7.25 (d, *J* =7.9 Hz, 2H), 7.19 (t, *J* =8.4 Hz, 2H), 6.88 (d, *J* =7.3 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.60 ($J_{C\text{-}F}$ = 253.4 Hz), 158.7, 158.7, 142.7, 140.7, 139.7, 134.1, 129.2, 128.9, 128.7, 128.3 ($J_{C\text{-}F}$ = 9.3 Hz), 128.2, 127.8, 126.4, 116.52 ($J_{C\text{-}F}$ = 22.2 Hz), 103.3, 21.5 ppm; IR (KBr) ν 3098, 3043, 2923, 2868, 1639, 1501, 1441, 1296 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{19}FNO_3S$ $[M+H]^+$ = 420.1064, found = 420.1075.

N-(6-(4-chlorophenyl)-3-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4f**



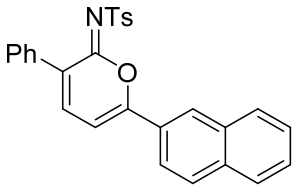
The title compound was prepared according to the general procedure to afford **4f** (87.2 mg, 50% yield) as a yellow powder. M.p. 169.8 – 173.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* =8.2 Hz, 2H), 7.88 (d, *J* =7.9 Hz, 2H), 7.62 – 7.55 (m, 2H), 7.51 – 7.44 (m, 3H), 7.40 – 7.33 (m, 3H), 7.24 (d, *J* =8.1 Hz, 2H), 6.90 (d, *J* =7.3 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 158.5, 142.8, 140.4, 139.7, 137.6, 134.0, 129.6, 129.2, 129.0, 128.7, 128.5, 128.3, 128.3, 127.2, 126.5, 103.7, 21.5 ppm; IR (KBr) ν 3088, 3065, 2950, 2865, 1636, 1519, 1446, 1294 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{19}ClNO_3S$ $[M+H]^+$ = 436.0769, found = 436.0770.

N-(6-(2-bromophenyl)-3-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4g**



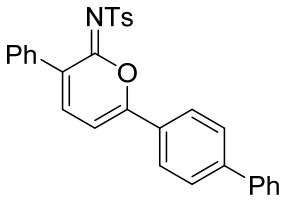
The title compound was prepared according to the general procedure to afford **4g** (99.9 mg, 52% yield) as a yellow powder. M.p. 158.8 – 161.9 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* =7.7 Hz, 1H), 7.81 (d, *J* =7.8 Hz, 2H), 7.70 (d, *J* =7.9 Hz, 1H), 7.64 (d, *J* = 4.9 Hz, 2H), 7.54 – 7.49 (m, 2H), 7.42 – 7.29 (m, 4H), 7.19 (d, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.7 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 157.8, 142.7, 139.5, 134.3, 134.0, 131.8, 131.2, 129.0, 128.9, 128.3, 128.2, 126.8, 121.0, 109.5, 21.4 ppm; IR (KBr) ν 3065, 3025, 2950, 2865, 1639, 1529, 1309, 1149 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{19}BrNO_3S$ $[M+H]^+$ = 480.0264, found = 480.0271.

4-methyl-N-(6-(naphthalen-2-yl)-3-phenyl-2H-pyran-2-ylidene)benzenesulfonamide **4h**



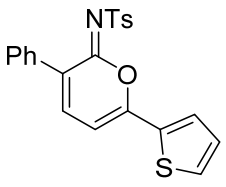
The title compound was prepared according to the general procedure to afford **4h** (92.1 mg, 51% yield) as a yellow powder. M.p. 220.0 – 222.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 8.06 – 8.02 (m, 1H), 8.00 – 7.93 (m, 4H), 7.90 – 7.85 (m, 1H), 7.65 – 7.63 (m, 2H), 7.60 – 7.53 (m, 3H), 7.41 – 7.38 (m, 3H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.4 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.9, 158.8, 142.6, 140.7, 139.8, 134.6, 134.2, 133.1, 129.5, 129.2, 129.1, 128.8, 128.3, 128.0, 127.8, 127.2, 127.1, 126.6, 122.0, 104.0, 21.5 ppm; IR (KBr) ν 3055, 3033, 2955, 2850, 1634, 1526, 1289, 1144 cm^{-1}; HRMS (ESI) m/z calcd for $C_{28}H_{22}NO_3S$ $[M+H]^+$ = 452.1315, found = 452.1322.

N-(6-([1,1'-biphenyl]-4-yl)-3-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4i**



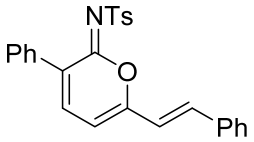
The title compound was prepared according to the general procedure to afford **4i** (108.9 mg, 57% yield) as a yellow powder. M.p. 209.5 – 211.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.4 Hz, 2H), 7.92 (d, *J* = 8.2 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.67 – 7.58 (m, 4H), 7.54 – 7.46 (m, 3H), 7.44 – 7.34 (m, 4H), 7.28 – 7.20 (m, 3H), 6.97 (d, *J* = 7.4 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 158.8, 144.1, 142.6, 140.7, 139.8, 134.2, 129.2, 129.0, 128.8, 128.2, 128.2, 127.8, 127.1, 126.5, 103.5, 21.5 ppm; IR (KBr) ν 3060, 3030, 2958, 2853, 1636, 1514, 1299, 1156 cm^{-1}; HRMS (ESI) m/z calcd for $C_{30}H_{24}NO_3S$ $[M+H]^+$ = 478.1471, found = 478.1475.

4-methyl-N-(3-phenyl-6-(thiophen-2-yl)-2H-pyran-2-ylidene)benzenesulfonamide **4j**



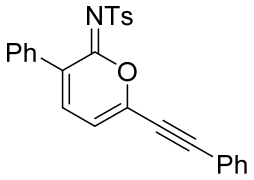
The title compound was prepared according to the general procedure to afford **4j** (97.8 mg, 60% yield) as a yellow powder. M.p. 65.6 – 68.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 3.3 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.62 – 7.56 (m, 2H), 7.51 (d, *J* = 4.9 Hz, 1H), 7.45 (d, *J* = 7.4 Hz, 1H), 7.39 – 7.34 (m, 3H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 4.3 Hz, 1H), 6.72 (d, *J* = 7.4 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 155.3, 142.6, 140.6, 139.9, 134.3, 133.4, 129.8, 129.6, 129.2, 128.9, 128.7, 128.2, 127.4, 126.5, 102.7, 21.5 ppm; IR (KBr) ν 3080, 3030, 2920, 2855, 1634, 1529, 1416, 1146 cm^{-1}; HRMS (ESI) m/z calcd for $C_{22}H_{18}NO_3S_2$ $[M+H]^+$ = 408.0723, found = 408.0732.

4-methyl-N-(3-phenyl-6-((E)-styryl)-2H-pyran-2-ylidene)benzenesulfonamide **4k**



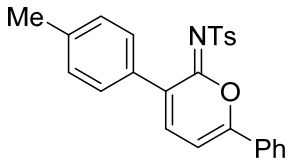
The title compound was prepared according to the general procedure to afford **4k** (104.3mg, 61% yield) as a yellow powder. M.p. 77.4 – 80.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.85 (d, *J* = 15.9 Hz, 1H), 7.60 – 7.57 (m, 4H), 7.44 – 7.31 (m, 7H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.68 (d, *J* = 15.9 Hz, 1H), 6.41 (d, *J* = 7.3 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 142.6, 140.6, 140.0, 137.8, 135.4, 134.4, 129.7, 129.2, 128.9, 128.8, 128.2, 128.0, 127.9, 126.5, 117.2, 107.1, 21.5 ppm; IR (KBr) ν 3060, 3028, 2950, 2858, 1644, 1524, 1446, 1301 cm^{-1}; HRMS (ESI) m/z calcd for $C_{26}H_{22}NO_3S$ $[M+H]^+$ = 428.1315, found = 428.1321.

4-methyl-N-(3-phenyl-6-(phenylethynyl)-2H-pyran-2-ylidene)benzenesulfonamide **4l**



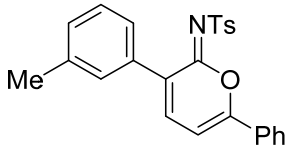
The title compound was prepared according to the general procedure to afford **4l** (69.8mg, 41% yield) as a yellow powder. M.p. 221.5 – 223.1 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.2 Hz, 2H), 7.66 – 7.56 (m, 4H), 7.51 – 7.42 (m, 3H), 7.41 – 7.36 (m, 3H), 7.34 (d, *J* = 7.3 Hz, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 6.68 (d, *J* = 7.3 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 143.2, 142.9, 139.0, 138.8, 133.9, 132.1, 130.5, 130.4, 129.2, 129.0, 128.8, 128.7, 128.3, 127.9, 120.4, 112.7, 98.3, 80.7, 21.5 ppm; IR (KBr) ν 3095, 3058, 3030, 2960, 2208, 1629, 1451, 1309 cm^{-1}; HRMS (ESI) m/z calcd for $C_{26}H_{20}NO_3S$ $[M+H]^+$ = 426.1158, found = 426.1152.

4-methyl-N-(6-phenyl-3-(p-tolyl)-2H-pyran-2-ylidene)benzenesulfonamide **4m**



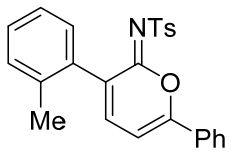
The title compound was prepared according to the general procedure to afford **4m** (86.4 mg, 52% yield) as a yellow powder. M.p. 193.5 – 196.7 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 7.5, 1.7 Hz, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.55 – 7.45 (m, 6H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 7.4 Hz, 1H), 2.37 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 158.9, 142.6, 140.3, 139.8, 138.9, 131.3, 131.2, 130.1, 129.2, 129.1, 128.9, 128.6, 127.9, 126.5, 125.9, 103.8, 21.5, 21.3 ppm; IR (KBr) ν 3090, 3052, 2952, 2851, 1637, 1494, 1300, 1141 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_3S$ $[M+H]^+$ = 416.1315, found = 416.1324.

4-methyl-N-(6-phenyl-3-(m-tolyl)-2H-pyran-2-ylidene)benzenesulfonamide **4n**



The title compound was prepared according to the general procedure to afford **4n** (89.7 mg, 54% yield) as a yellow powder. M.p. 150.8 – 154.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.00 (m, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.54 – 7.48 (m, 4H), 7.43 (s, 1H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.30 – 7.26 (m, 1H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 7.4 Hz, 1H), 2.38 (s, 3H), 2.35 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.8, 142.5, 140.4, 140.0, 137.8, 134.1, 131.4, 130.1, 129.6, 129.5, 129.2, 129.1, 128.1, 126.5, 126.0, 125.9, 103.5, 21.5, 21.4 ppm; IR (KBr) ν 3055, 3028, 2954, 2857, 1634, 1496, 1301, 1155 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_3S$ $[M+H]^+$ = 416.1315, found = 416.1321.

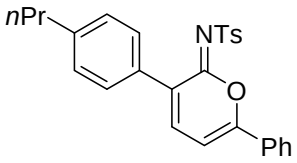
4-methyl-N-(6-phenyl-3-(o-tolyl)-2H-pyran-2-ylidene)benzenesulfonamide **4o**



The title compound was prepared according to the general procedure to afford **4o** (66.5 mg, 40% yield) as a yellow powder. M.p. 157.3 – 161.3 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.02 (m, 2H), 7.80 (d, *J* = 8.1 Hz, 2H), 7.56 –

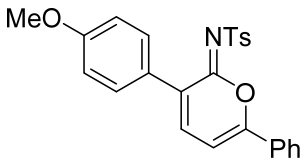
7.51 (m, 3H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.30 – 7.26 (m, 1H), 7.24 – 7.14 (m, 5H), 6.92 (d, *J* = 7.3 Hz, 1H), 2.34 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 158.8, 142.5, 142.1, 139.7, 136.8, 134.3, 131.5, 130.2, 130.1, 129.8, 129.4, 129.2, 129.1, 128.7, 126.5, 126.1, 125.8, 103.1, 21.5, 20.1 ppm; IR (KBr) ν 3080, 3063, 2962, 2863, 1636, 1506, 1449, 1294 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_3S$ $[M+H]^+$ = 416.1315, found = 416.1328.

4-methyl-N-(6-phenyl-3-(4-propylphenyl)-2H-pyran-2-ylidene)benzenesulfonamide **4p**



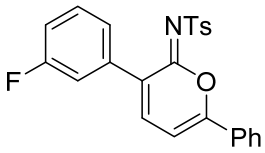
The title compound was prepared according to the general procedure to afford **4p** (97.5 mg, 55% yield) as a yellow powder. M.p. 126.5 – 128.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.55 – 7.48 (m, 4H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H),6.92 (d, *J* = 7.4 Hz, 1H), 2.66 – 2.54 (m, 2H), 2.38 (s, 3H), 1.1.73 – 1.60 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 158.9, 143.7, 142.5, 140.2, 139.9, 131.5, 131.3, 130.1, 129.2, 129.1, 128.6, 128.3, 128.0, 126.5, 125.9, 103.6, 24.4, 21.5, 13.9 ppm; IR (KBr) ν 3080, 3026, 2953, 2928, 2865, 1639, 1509, 1306 cm^{-1}; HRMS (ESI) m/z calcd for $C_{27}H_{26}NO_3S$ $[M+H]^+$ = 444.1628, found = 444.1639.

N-(3-(4-methoxyphenyl)-6-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4q**



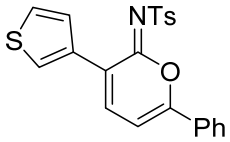
The title compound was prepared according to the general procedure to afford **4q** (81.1 mg, 47% yield) as a yellow powder. M.p. 160.2 – 162.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 6.3 Hz, 2H), 7.91 (d, *J* = 8.1 Hz, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.53 – 7.47 (m, 4H), 7.23 (d, *J* = 8.0 Hz, 2H), 6.92 – 6.88 (m, 2H), 3.82 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 159.2, 159.0, 142.6, 139.9, 139.6, 131.2, 130.2, 130.1, 129.2, 129.1, 127.6, 126.5, 125.9, 113.7, 103.7, 55.3, 21.5 ppm; IR (KBr) ν 3013, 3065, 2960, 2838, 1634, 1511, 1496, 1309 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{22}NO_4S$ $[M+H]^+$ = 432.1264, found = 432.1271.

N-(3-(3-fluorophenyl)-6-phenyl-2H-pyran-2-ylidene)-4-methylbenzenesulfonamide **4r**



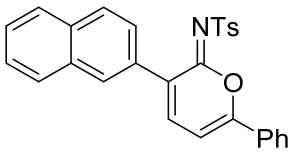
The title compound was prepared according to the general procedure to afford **4r** (105.7 mg, 63% yield) as a yellow powder. M.p. 159.9 – 162.9 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 6.6, 2.8 Hz, 2H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.58 – 7.50 (m, 4H), 7.42 – 7.30 (m, 3H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.06 (t, *J* = 7.9 Hz, 1H), 6.95 (d, *J* = 7.4 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (J_{C-F} = 245.7 Hz), 160.2 , 158.3 , 142.8 , 141.1 , 139.7 , 136.1 (J_{C-F} = 8.2 Hz) , 131.6 , 129.9 , 129.8 (J_{C-F} = 8.3 Hz), 129.3 , 126.5 , 126.1 , 124.4 (J_{C-F} = 2.2 Hz) , 115.9 (J_{C-F} = 17.2 Hz), 115.7 (J_{C-F} = 15.1 Hz), 103.5 , 21.5 ppm; IR (KBr) ν 3100, 3070, 2960, 2853, 1641, 1494, 1301, 1149 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{19}FNO_3S$ $[M+H]^+$ = 420.1064, found = 420.1076.

4-methyl-N-(6-phenyl-3-(thiophen-3-yl)-2H-pyran-2-ylidene)benzenesulfonamide **4s**



The title compound was prepared according to the general procedure to afford **4s** (99.4 mg, 61% yield) as a yellow powder. M.p. 206.1 – 208.3 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 2.2 Hz, 1H), 8.10 – 8.01 (m, 3H), 7.96 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.54 – 7.48 (m, 3H), 7.42 (d, *J* = 5.0 Hz, 1H), 7.34 – 7.30 (m, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 6.93 (d, *J* = 7.6 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 157.9, 142.8, 138.3, 133.4, 131.4, 130.0, 129.3, 129.3, 126.6, 126.5, 126.4, 126.4, 126.0, 125.4, 122.2, 103.4, 21.5 ppm; IR (KBr) ν 3143, 3093, 2955, 2848, 1634, 1496, 1301, 1151 cm^{-1}; HRMS (ESI) m/z calcd for $C_{22}H_{18}NO_3S_2$ $[M+H]^+$ = 408.0723, found = 408.0735.

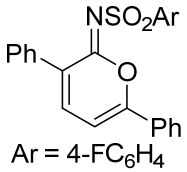
4-methyl-N-(3-(naphthalen-2-yl)-6-phenyl-2H-pyran-2-ylidene)benzenesulfonamide **4t**



The title compound was prepared according to the general procedure to afford **4t** (92.1 mg, 51% yield) as a yellow powder. M.p. 191.8 – 194.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.06 (m, 3H), 7.92 (d, *J* = 8.3 Hz, 2H), 7.85 –

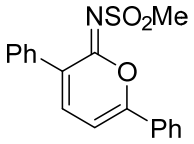
7.80 (m, 3H), 7.74 (dd, J = 8.6, 1.7 Hz, 1H), 7.65 (d, J = 7.4 Hz, 1H), 7.56 – 7.48 (m, 5H), 7.22 (d, J = 8.1 Hz, 2H), 6.99 (d, J = 7.4 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 158.8, 142.6, 140.8, 139.9, 133.3, 133.0, 131.7, 131.5, 130.1, 129.3, 129.2, 128.4, 127.9, 127.7, 127.6, 126.8, 126.5, 126.4, 126.2, 126.0, 103.6, 21.5 ppm; IR (KBr) ν 3065, 2958, 2925[1], 2856, 1636, 1449, 1301, 1146 cm^{-1}; HRMS (ESI) m/z calcd for $C_{28}H_{22}NO_3S$ $[M+H]^+$ = 452.1315, found = 452.1324.

N-(3,6-diphenyl-2H-pyran-2-ylidene)-4-fluorobenzenesulfonamide **4u**



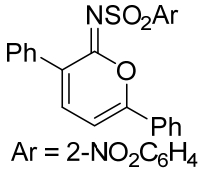
The title compound was prepared according to the general procedure to afford **4u** (92.1 mg, 51% yield) as a yellow powder. M.p. 168.2 – 170.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.97 (m, 4H), 7.58 (dd, J = 6.1, 2.7 Hz, 2H), 7.56 – 7.49 (m, 4H), 7.42 – 7.33 (m, 3H), 7.10 (t, J = 8.6 Hz, 2H), 6.97 (d, J = 7.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7 (J_{C-F} = 253.1 Hz), 159.9, 159.1, 141.0, 138.8, 134.1, 131.6, 130.0, 129.3, 129.2, 129.0 (J_{C-F} = 16.7 Hz), 128.7, 128.3, 127.9, 126.0, 115.7 (J_{C-F} = 22.5 Hz), 103.8 ppm; IR (KBr) ν 3103, 3083, 2963, 2925, 1636, 1511, 1444, 1311 cm^{-1}; HRMS (ESI) m/z calcd for $C_{23}H_{17}FNO_3S$ $[M+H]^+$ = 406.0908, found = 406.0915.

N-(3,6-diphenyl-2H-pyran-2-ylidene)methanesulfonamide **4v**



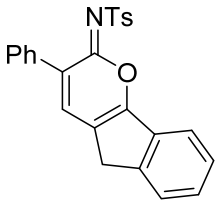
The title compound was prepared according to the general procedure to afford **4v** (83.3 mg, 64% yield) as a yellow powder. M.p. 160.7 – 163.1 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.65 – 7.60 (m, 2H), 7.51 (d, J = 3.0 Hz, 2H), 7.50 (d, J = 2.0 Hz, 2H), 7.46 – 7.38 (m, 3H), 6.94 (d, J = 7.4 Hz, 1H), 3.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.9, 140.6, 134.3, 131.4, 129.9, 129.3, 128.9, 128.8, 128.3, 127.7, 125.9, 103.2, 42.9 ppm; IR (KBr) ν 3025, 2955, 2925, 2858, 1636, 1451, 1301, 1122 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{16}NO_3S$ $[M+H]^+$ = 326.0845, found = 326.0860.

N-(3,6-diphenyl-2H-pyran-2-ylidene)-2-nitrobenzenesulfonamide **4w**



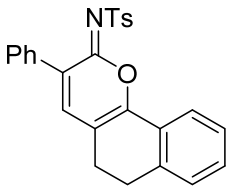
The title compound was prepared according to the general procedure to afford **4w** (107.2 mg, 62% yield) as a yellow powder. M.p. 148.8 – 151.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (dd, J = 6.0, 3.3 Hz, 1H), 8.04 – 7.98 (m, 2H), 7.75 (dd, J = 5.8, 3.4 Hz, 1H), 7.64 (dd, J = 5.9, 3.4 Hz, 2H), 7.58 (dd, J = 10.5, 5.0 Hz, 3H), 7.53 – 7.47 (m, 3H), 7.41 – 7.33 (m, 3H), 7.02 (d, J = 7.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 159.7, 147.8, 141.8, 135.7, 133.9, 132.9, 132.0, 131.7, 129.8, 129.8, 129.3, 128.9, 128.8, 128.4, 128.0, 126.0, 124.5, 104.0; IR (KBr) ν 3095, 3060, 2958, 2855, 1639, 1456, 1314, 1161 cm^{-1}; HRMS (ESI) m/z calcd for $C_{23}H_{17}N_2O_5S$ $[M+H]^+$ = 433.0853, found = 433.0862.

4-methyl-N-(3-phenylindeno[1,2-b]pyran-2(5H)-ylidene)benzenesulfonamide **5a**



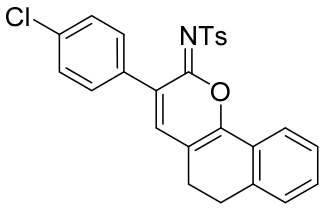
The title compound was prepared according to the general procedure to afford **5a** (122.4 mg, 74% yield) as a yellow powder. M.p. 226.1 – 228.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.84 (d, *J* = 7.5 Hz, 1H), 7.67 (s, 1H), 7.62 (d, *J* = 6.7 Hz, 2H), 7.55 (d, *J* = 7.1 Hz, 1H), 7.52 – 7.42 (m, 2H), 7.42 – 7.33 (m, 3H), 7.27 (d, *J* = 8.1 Hz, 2H), 3.70 (s, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 158.9, 143.0, 142.6, 139.3, 135.2, 133.9, 129.4, 129.2, 129.0, 128.6, 128.2, 127.9, 127.0, 126.7, 125.2, 120.6, 120.4, 33.3, 21.5 ppm; IR (KBr) ν 2965, 2928, 2858, 1626, 1494, 1459, 1306, 1154 cm^{-1}; HRMS (ESI) m/z calcd for $C_{25}H_{20}NO_3S$ $[M+H]^+$ = 414.1158, found = 414.1167.

4-methyl-N-(3-phenyl-5,6-dihydro-2H-benzo[h]chromen-2-ylidene)benzenesulfonamide **5b**



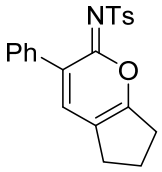
The title compound was prepared according to the general procedure to afford **5b** (128.2 mg, 75% yield) as a yellow powder. M.p. 218.5 – 221.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.3 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 2H), 7.62 (dd, *J* = 7.6, 1.7 Hz, 2H), 7.46 – 7.33 (m, 6H), 7.23 (t, *J* = 7.8 Hz, 3H), 2.97 (t, *J* = 7.8 Hz, 2H), 2.78 (t, *J* = 7.8 Hz, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 154.7, 142.6, 142.5, 140.1, 137.0, 134.5, 130.9, 129.2, 128.8, 128.7, 128.2, 127.8, 126.9, 126.5, 124.3, 114.9, 27.2, 24.6, 21.5 ppm; IR (KBr) ν 3058, 2960, 2920, 2888, 1644, 1504, 1449, 1301 cm^{-1}; HRMS (ESI) m/z calcd for $C_{26}H_{22}NO_3S$ $[M+H]^+$ = 428.1315, found = 428.1328.

N-(3-(4-chlorophenyl)-5,6-dihydro-2H-benzo[h]chromen-2-ylidene)-4-methylbenzenesulfonamide **5c**



The title compound was prepared according to the general procedure to afford **5c** (97.9 mg, 53% yield) as a yellow powder. M.p. 258.5 – 260.2^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.3 Hz, 1H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.46 – 7.31 (m, 5H), 7.29 – 7.18 (m, 3H), 2.97 (t, *J* = 7.8 Hz, 2H), 2.78 (t, *J* = 7.8 Hz, 2H), 2.39 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 155.0, 142.6, 142.4, 139.9, 137.1, 134.7, 132.9, 131.1, 130.1, 129.2, 128.4, 127.9, 126.8, 126.5, 124.4, 114.8, 27.2, 24.6, 21.5 ppm; IR (KBr) ν 3055, 2953, 2850, 2833, 1639, 1444, 1291, 1141 cm^{-1}; HRMS (ESI) m/z calcd for $C_{26}H_{21}ClNO_3S$ $[M+H]^+$ = 462.0925, found = 462.0932.

4-methyl-N-(3-phenyl-6,7-dihydrocyclopenta[b]pyran-2(5H)-ylidene)benzenesulfonamide **5d**



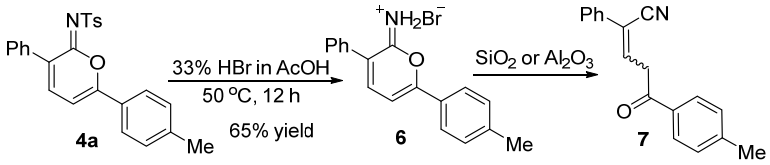
The title compound was prepared according to the general procedure to afford **5d** (67.2 mg, 46% yield) as a yellow powder. M.p. 194.9 – 197.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_{3)}$ δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 7.2 Hz, 2H), 7.35 – 7.30 (m, 4H), 7.22 (d, *J* = 8.0 Hz, 2H), 2.85 (t, *J* = 7.6 Hz, 2H), 2.69 (t, *J* = 7.1 Hz, 2H), 2.37 (s, 3H), 2.20 – 2.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 160.2, 142.6, 140.3, 139.6, 134.9, 129.0, 128.9, 128.5, 128.2, 127.3, 119.9, 30.4, 27.8, 21.5, 20.7 ppm; IR (KBr) ν 3033, 2958, 2865, 2840, 1649, 1446, 1291, 1149 cm^{-1}; HRMS (ESI) m/z

calcd for $C_{21}H_{20}NO_3S$ $[M+H]^+$ = 366.1158, found = 366.1163.

D. Gram scale synthesis of inminolactones 4b and 5b

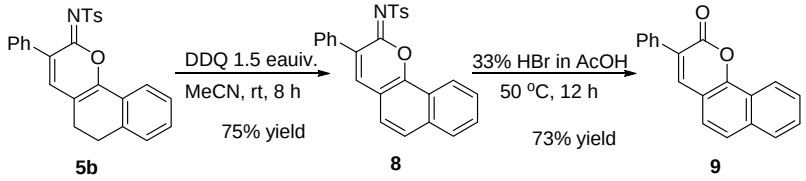
To a flame dried round bottle flask with a magnetic stirring bar were added CuI (76 mg, 0. 4 mmol, 10 mol %), PPh_3 (21.0 mg, 0. 8 mmol, 20 mol %) and enaminone **1b** (4 mmol, 701 mg) or 1n (4 mmol, 805 mg), followed by the addition of 1,2-dichloroethane (20 mL) and TEA (0.28 mL, 0.2 mmol, 0.5 equiv.) under nitrogen atmosphere at room temperature. Phenylacetylene **2a** (10 mmol, 1.1 mL, 2.5 equiv.) and p-toluenesulfonyl azide **3a** (10 mmol, 1.97g, 2.5 equiv.) was then added, and the resulting mixture was stirred overnight. After the completely consumption of enaminone **1** monitored by TLC, the solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel with PE/Ea = 5:1 as eluent to afford yellow solid **4b** (1.24g, 77% yield) or **5b** (1.21g, 71% yield).

E. Transformation of 4a and 5b



To the seal tube was added **4a** (1.2 mmol, 498.6 mg), followed by adding 5 mL 33% HBr in AcOH. The mixture was stirred at 50 °C and the solid was dissolved. Yellow solid was precipitated after overnight. The solid was filtered, washed by water and recrystallized by DCM to get pure **6** (266.9 mg, 65% yield) as a yellow powder. M.p. 198.5 – 201.1 °C; ^{1}H NMR (400 MHz, DMSO) δ 10.83 (s, 1H), 9.76 (s, 1H), 8.04 – 7.97 (m, 3H), 7.69 (d, J = 7.7 Hz, 1H), 7.61 – 7.52 (m, 5H), 7.45 (d, J = 8.1 Hz, 2H), 2.42 (s, 3H)；^{13}C NMR (100 MHz, DMSO) δ 166.3, 158.3, 147.0, 143.2, 132.4, 130.5, 130.1, 129.8, 129.2, 126.7, 126.4, 122.8, 106.0, 21.6；IR (KBr) ν 3425, 3315, 2920, 2855, 1651, 1446, 1314, 1191 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{16}NO$ $[M\text{-}Br^-]^+$ = 262.1226, found = 262.1237. During our purification of compound **6**, we found compound 6 was easily decomposed to compound **7** as a pale yellow solid. M.p. 87.2 – 90.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.1 Hz, 2H), 7.61 (d, J = 7.0 Hz, 2H), 7.45 – 7.37 (m, 3H), 7.34 – 7.27 (m,3H), 4.29 (d, J = 7.0 Hz, 2H), 2.44 (s, 3H)； ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 144.9, 138.9, 133.5, 132.7, 129.6, 129.4, 129.0, 128.4, 125.9, 118.3, 116.4, 40.9, 21.7；IR (KBr) ν 3445, 3050, 3035, 2863, 2226, 1686,

1499, 1449 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{14}NO$ $[M-H]^-$ = 260.1081, found = 260.1077.



To the solution of **5b** (0.2 mmol, 85.5 mg) in MeCN (2 mL) was added 2,3-dicyano-5,6-dichlorobenzoquinone, DDQ (0.3 mmol, 68.1 mg) at room temperature. After the completely consumption of **5b** monitored by TLC, the solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel with PE/Ea = 5:1 as eluent to afford yellow solid **8** (63.8 mg, 75% yield). M.p. 88.2 – 91.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (d, *J* = 8.2 Hz, 1H), 8.00 (d, *J* = 8.1 Hz, 2H), 7.93 (d, *J* = 8.2 Hz, 1H), 7.91 (s, 1H), 7.83 – 7.67 (m, 5H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.48 – 7.42 (m, 3H), 7.32 (d, *J* = 7.9 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 149.9, 142.7, 140.4, 139.8, 134.8, 134.5, 129.4, 129.2, 129.0, 128.9, 128.2, 128.1, 127.7, 126.6, 125.8, 123.3, 123.0, 122.8, 115.6, 21.5 ppm; IR (KBr) ν 3065, 3033, 2963, 2853, 1821, 1686, 1459, 1311 cm^{-1}; HRMS (ESI) m/z calcd for $C_{26}H_{20}NO_3S$ $[M+H]^+$ = 426.1158, found = 426.1171.

To the seal tube was added **8** (0.6 mmol, 255.3 mg), followed by adding 3 mL 33% HBr in AcOH. The mixture was stirred at 50 °C and the solid was dissolved. After the completely consumption of **8** monitored by TLC, the mixture was diluted by 20 mL water and extracted by DCM (2 × 20 mL). The combined organic phase was washed by water, dried by Na_2SO_4, filtered and concentrated under reduced pressure and the residue was purified by column chromatography on silica gel with PE/Ea = 5:1 as eluent to afford white powder **9** (119.2 mg, 73% yield). M.p. 174.2 – 176.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, J = 7.9 Hz, 1H), 8.20 (d, J = 8.1 Hz, 1H), 7.88 – 7.61 (m, 7H), 7.56 – 7.36 (m, 3H)； ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 149.9, 139.1, 134.4, 132.5, 129.7, 129.1, 128.6, 128.5, 128.4, 128.0, 127.4, 126.9, 123.8, 122.7, 118.0, 115.7 ppm；IR (KBr) ν 3417, 3065, 3000, 2855, 1726, 1636, 1509, 1449 cm^{-1}; HRMS (ESI) m/z calcd for $C_{19}H_{13}O_2$ $[M+H]^+$ = 273.0910, found = 273.0922.

F. X-Ray crystallographic analysis of 4b

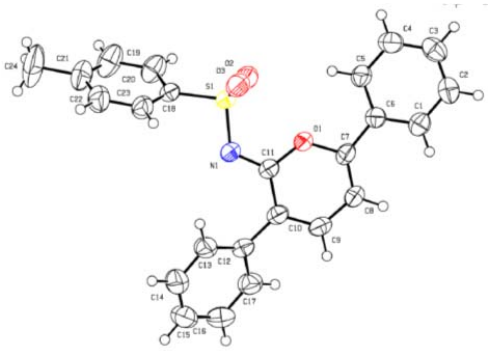


Figure 1. X-ray structure of **4b**

Table 1. Crystal data and structure refinement for **4b**

Empirical formula	C24 H19 N O3 S	
Formula weight	401.46	
Temperature	293K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 16.2229(11) Å	α= 90°.
	b = 10.6431(5) Å	β= 98.650°.
	c = 12.0294(7) Å	γ = 90°.
Volume	2053.4(2) $Å^3$	
Z	4	
Density (calculated)	1.299 Mg/m^3	
Absorption coefficient	0.183 mm^{-1}	
F(000)	840	
Crystal size	0.42 x 0.28 x 0.13 mm^3	
Theta range for data collection	2.977 to 25.347°.	
Index ranges	-19<=h<=19, -12<=k<=12, -14<=l<=9	

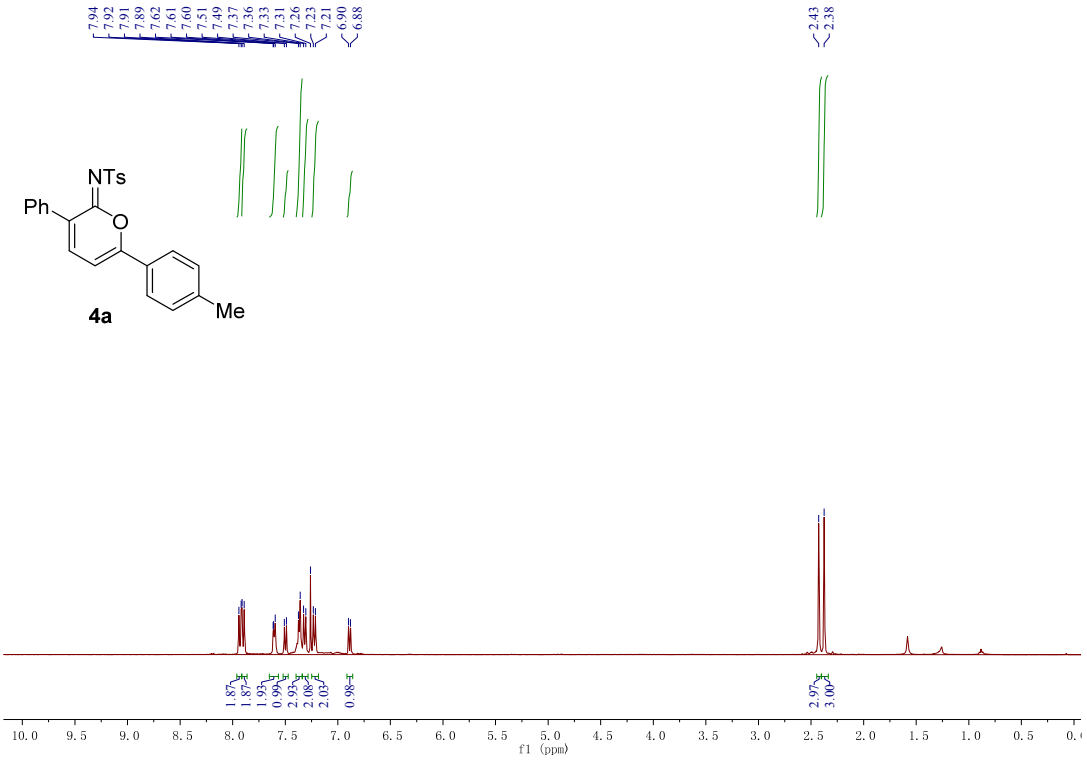
Reflections collected	7994
Independent reflections	3759 [R(int) = 0.0344]
Completeness to theta = 25.242°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.86145
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3759 / 0 / 263
Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0494, wR2 = 0.1161
R indices (all data)	R1 = 0.0747, wR2 = 0.1381
Absolute structure parameter	0.02(4)
Extinction coefficient	n/a
Largest diff. peak and hole	0.203 and -0.393 e.$Å^{-3}$

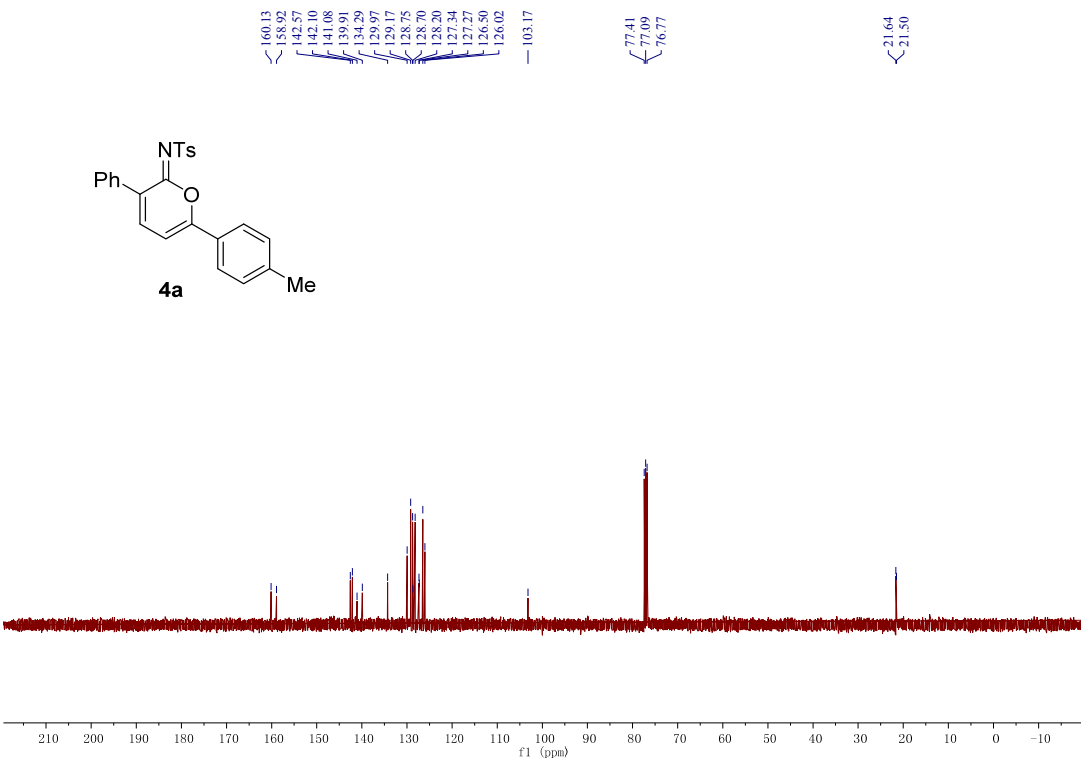
G. References

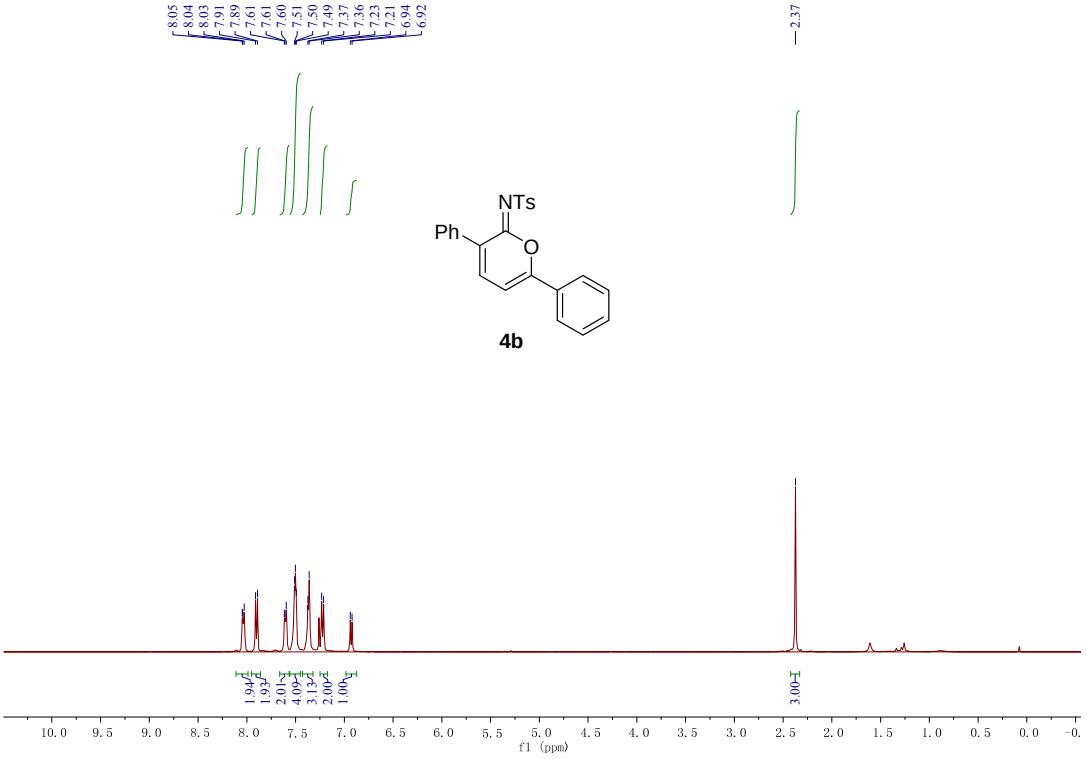
[1**]** Jiang, Y.; Zhong, V. Y. K.; Emmanuvel, L.; Park, C.-M. *Chem. Commun*, **2012**, *48*, 3133.

[2] Yadav, M. R.; Rit, K. R.; Sahoo, A. K. *Org. Lett*. **2013**, *15*, 1638.

H. NMR spectra of the products



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7.21
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6.92
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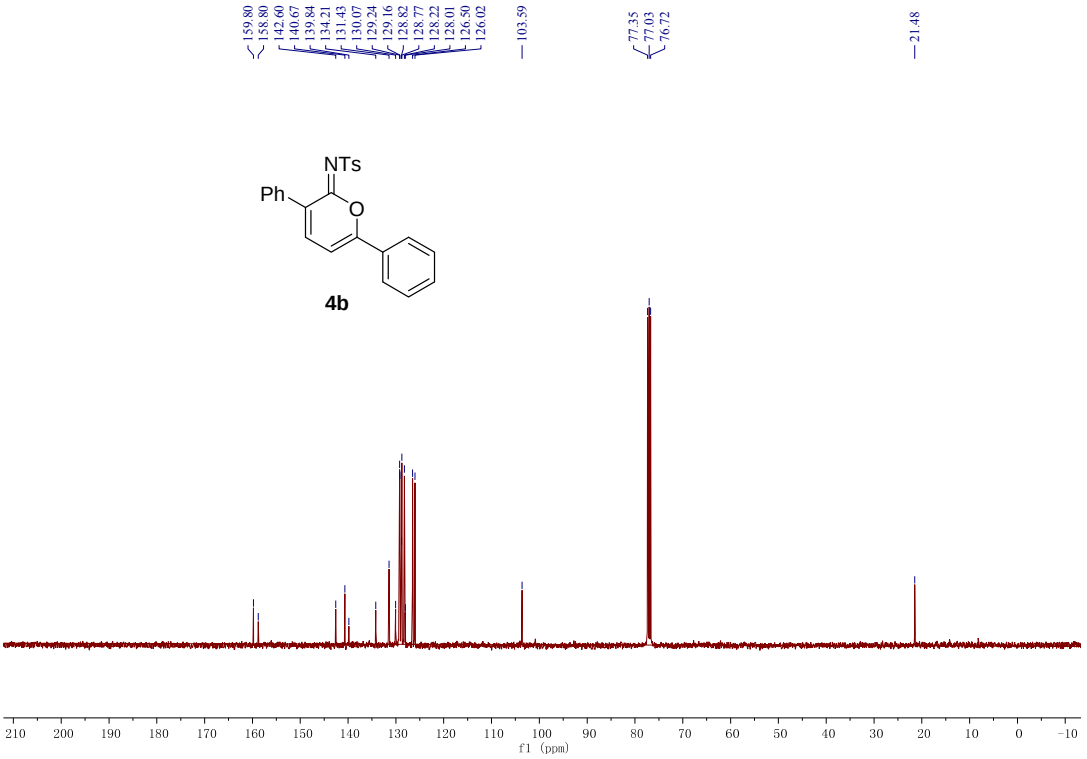
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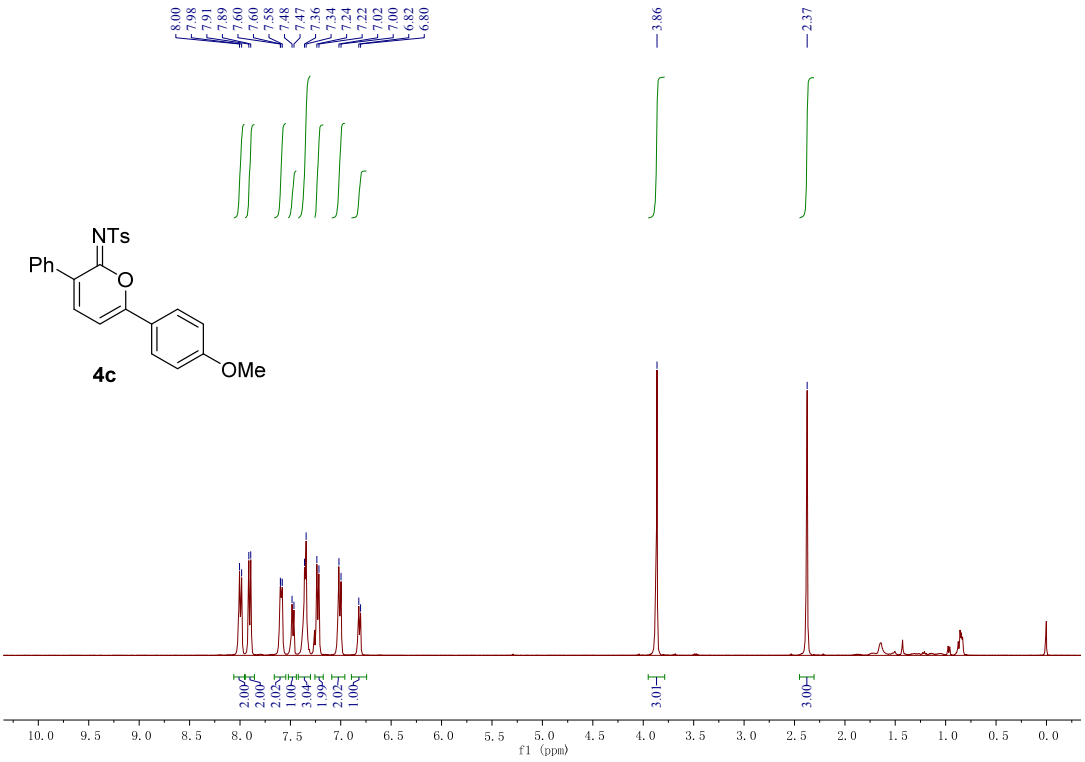
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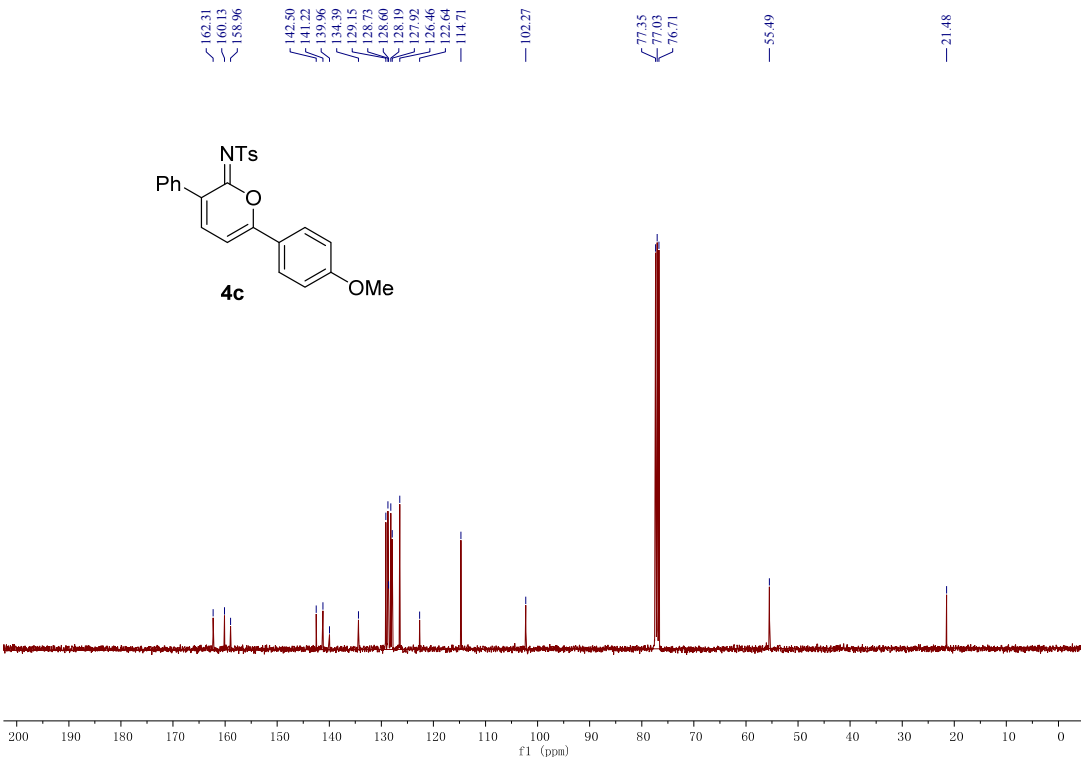
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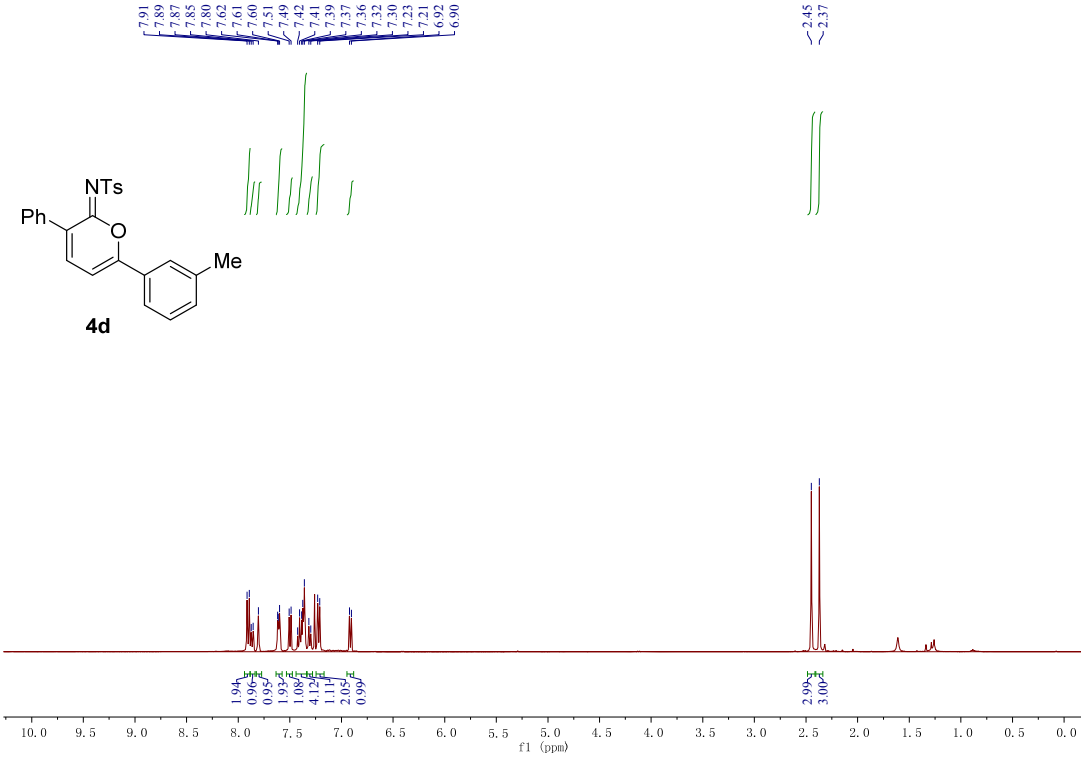
f1 (ppm)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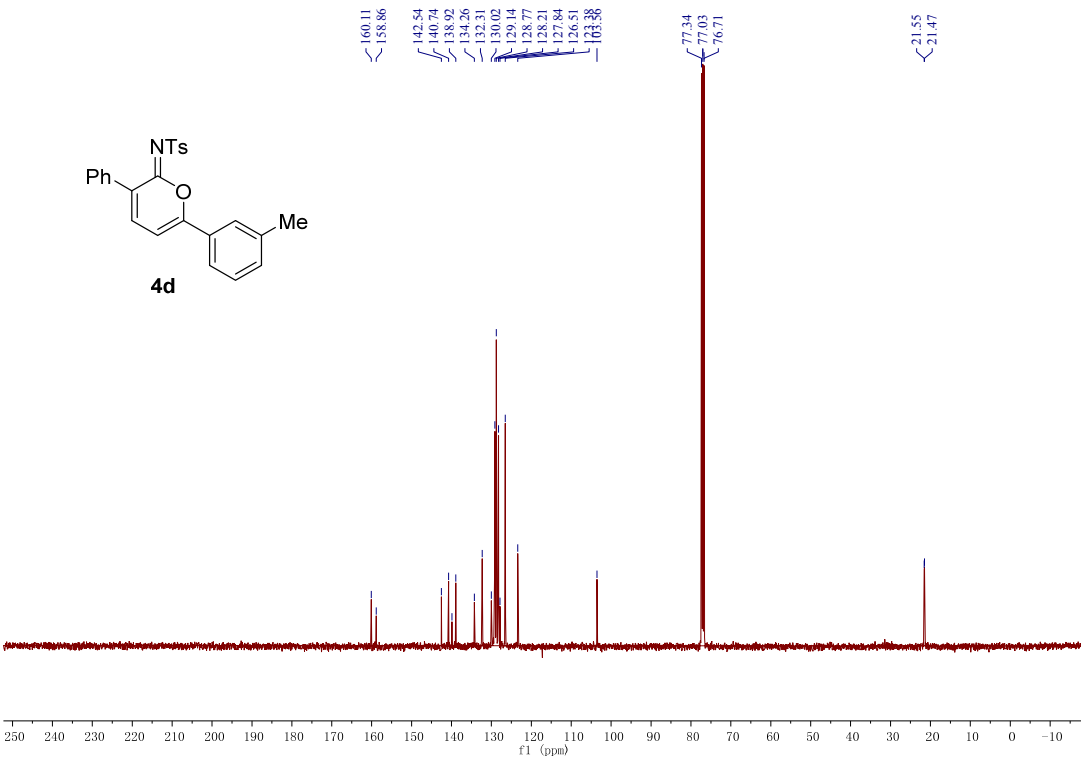
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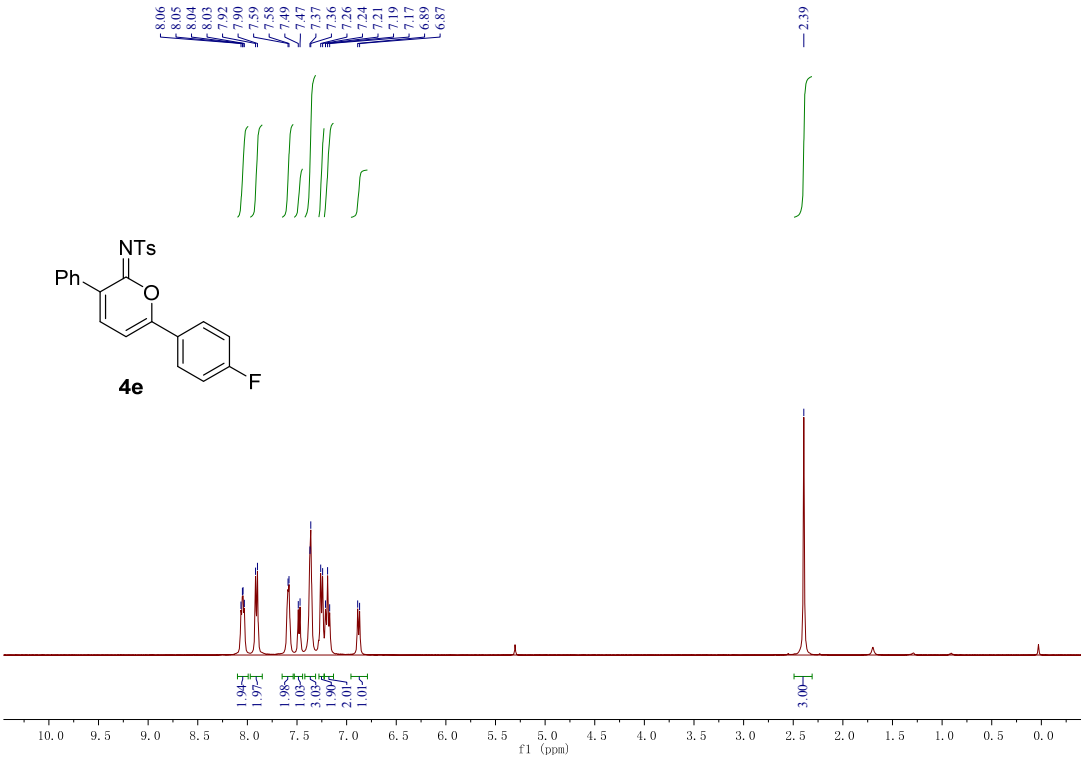
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1.01
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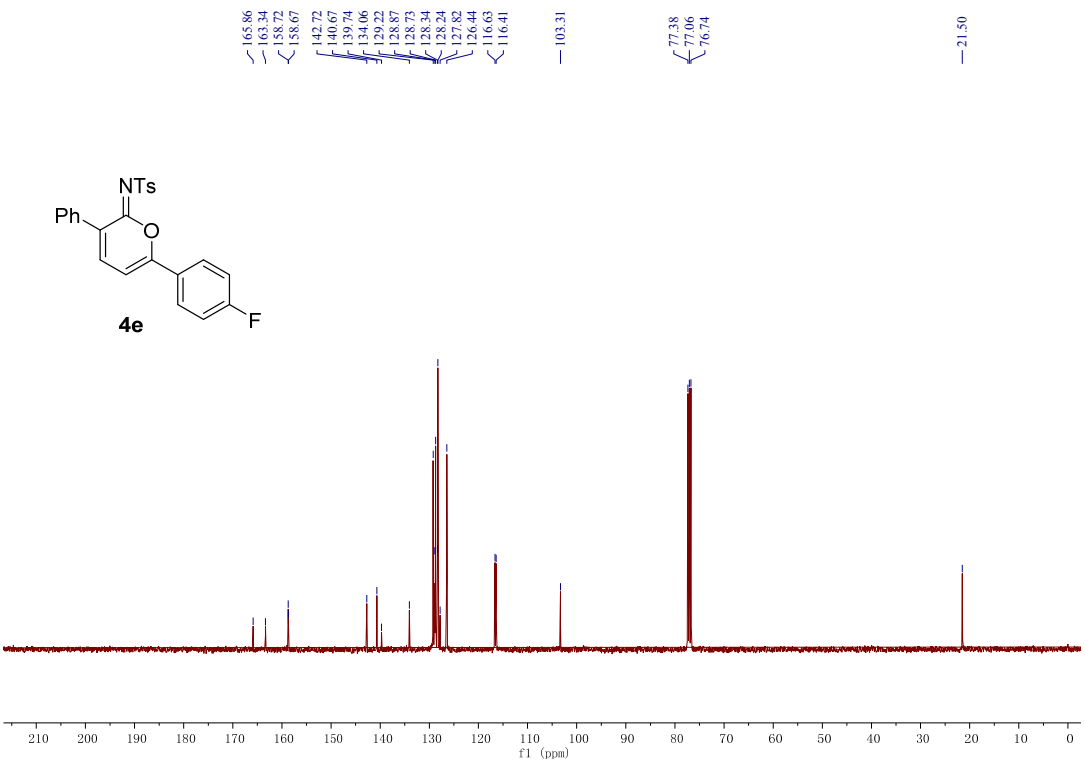
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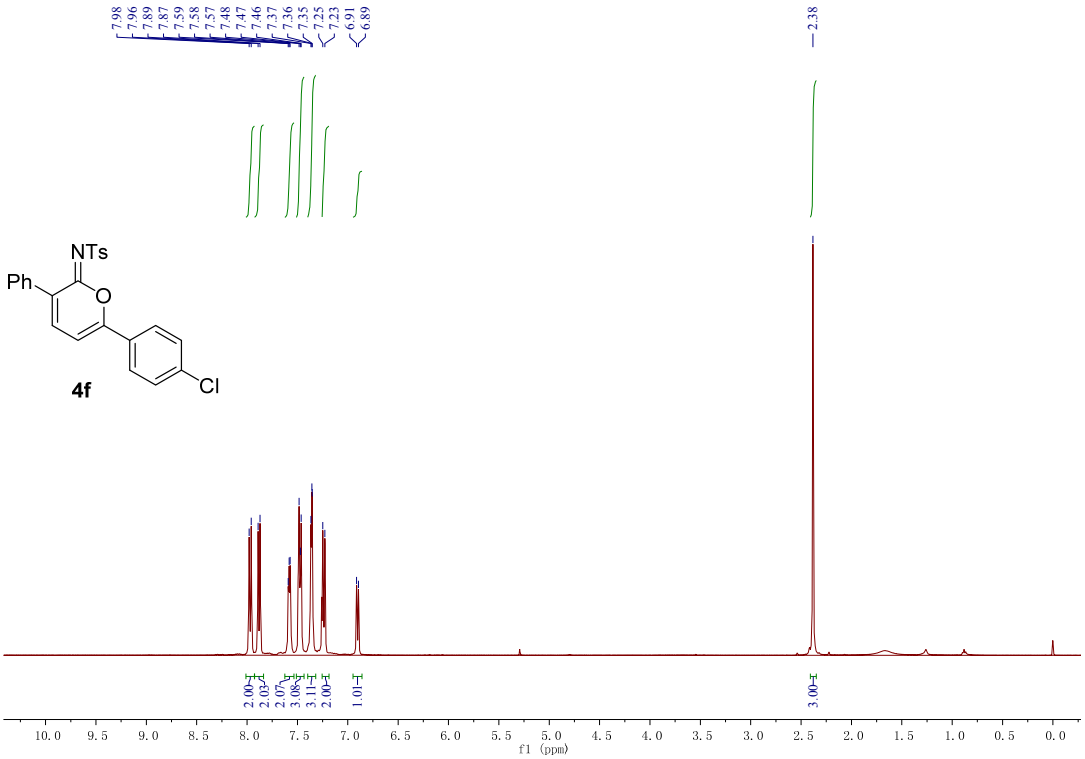
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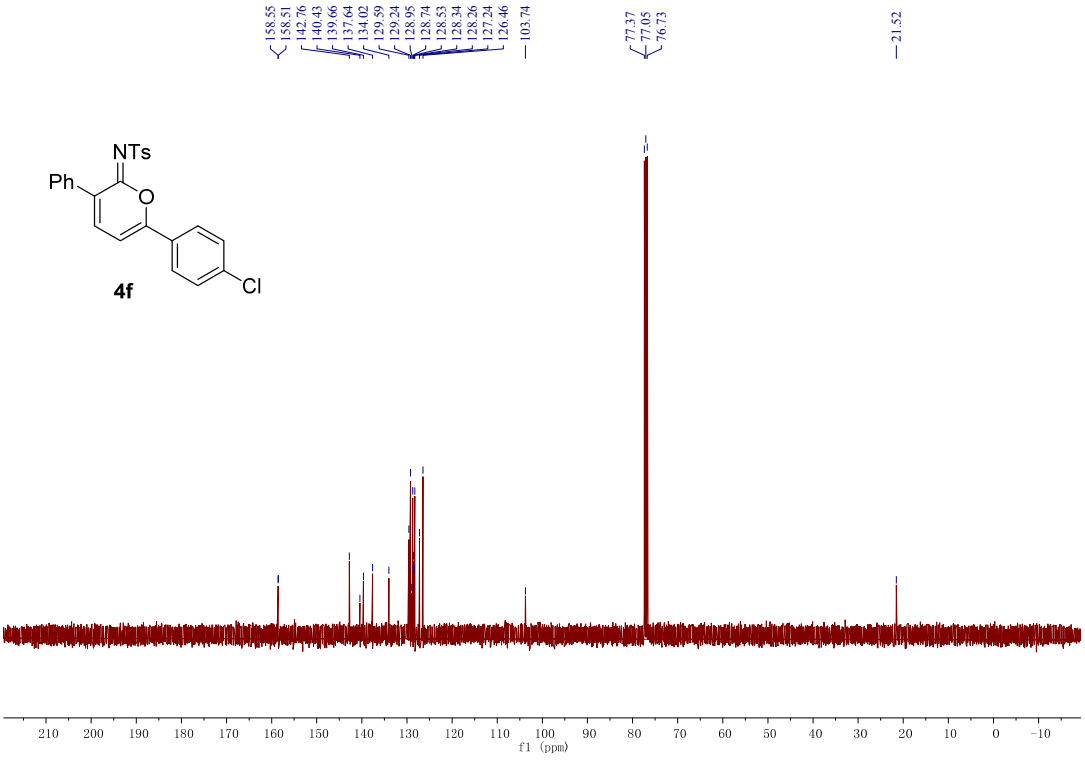
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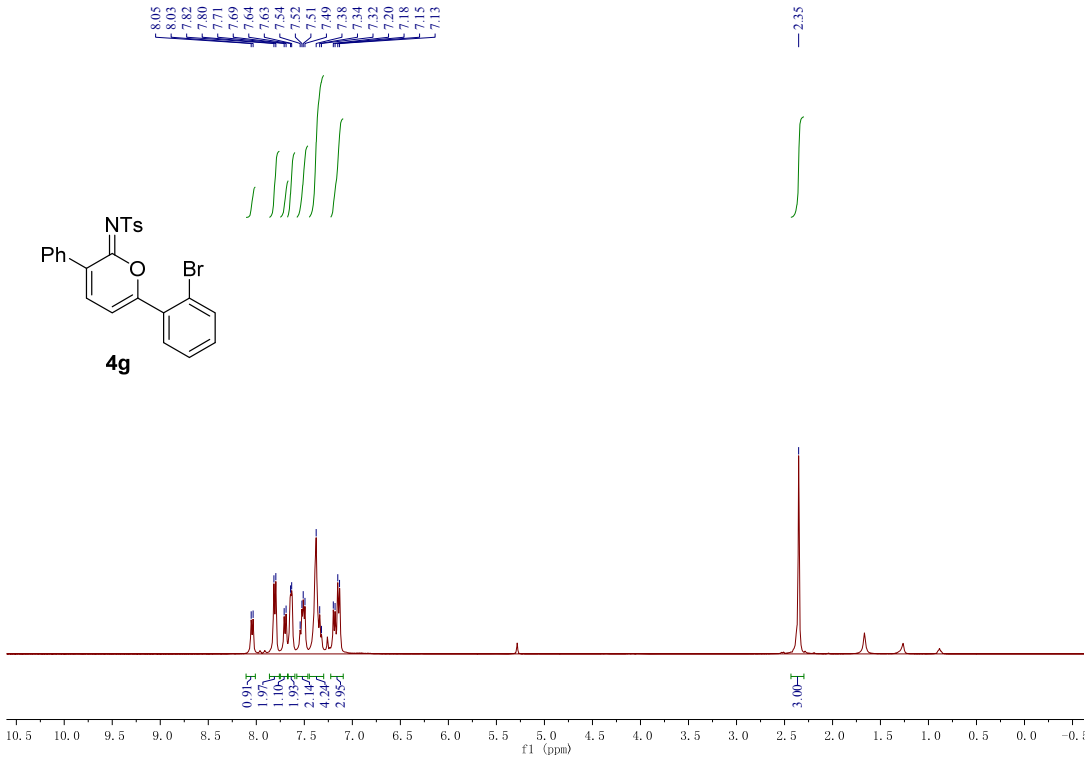
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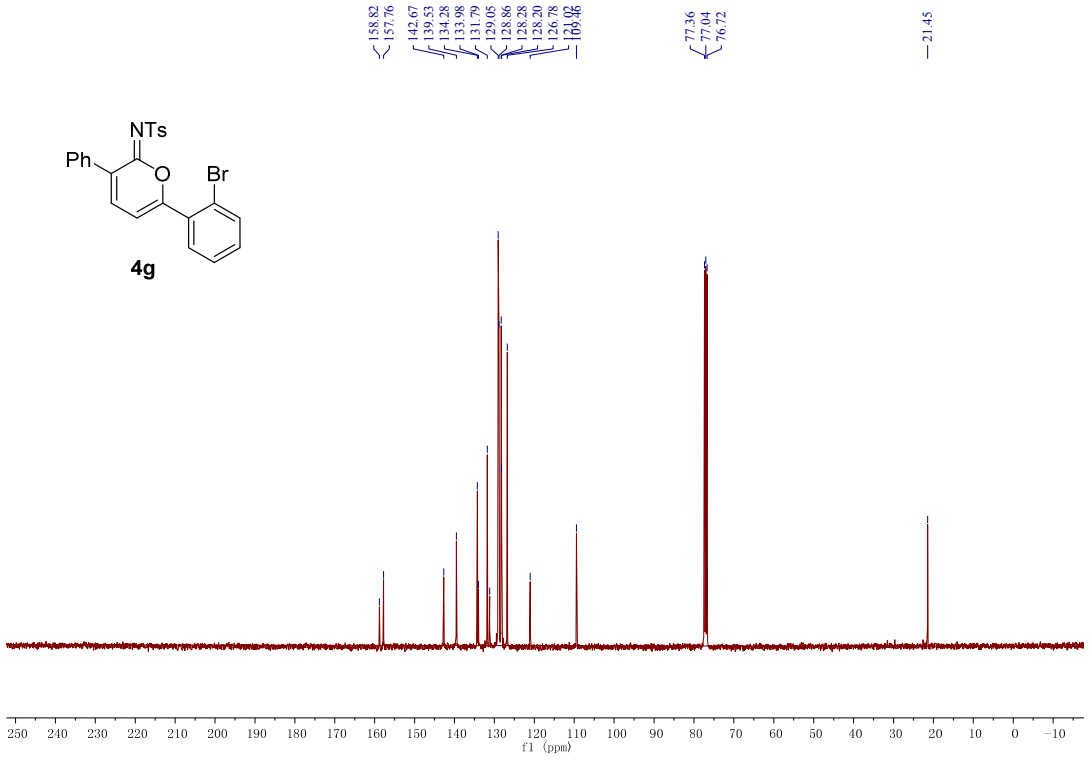
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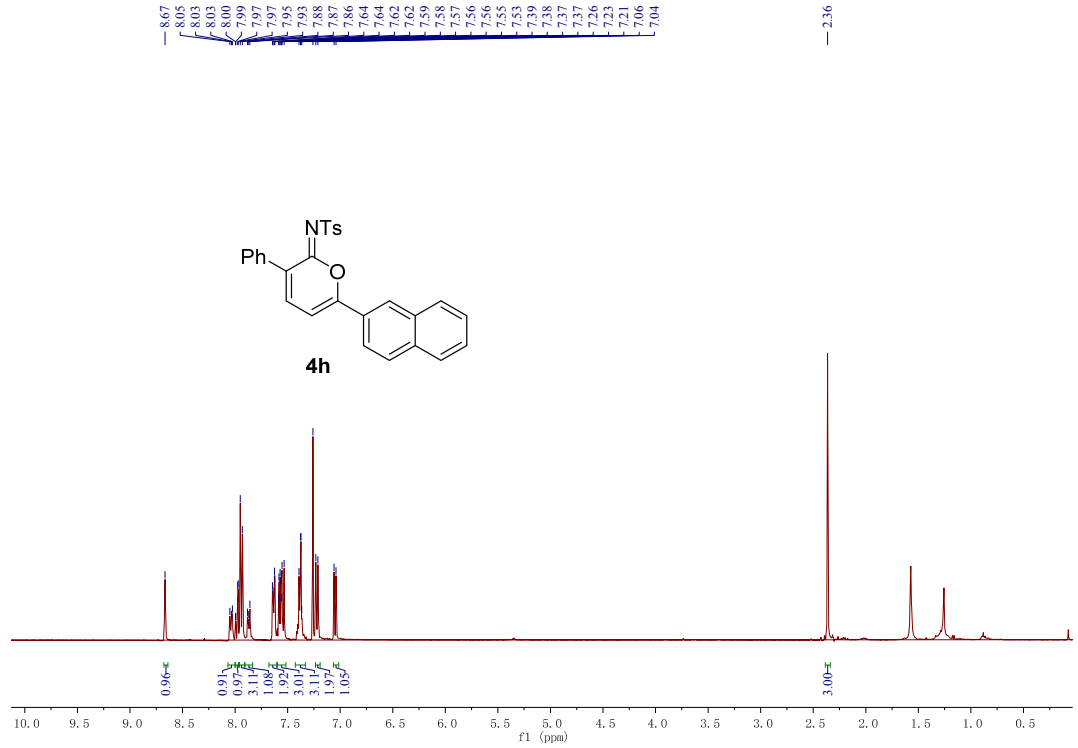
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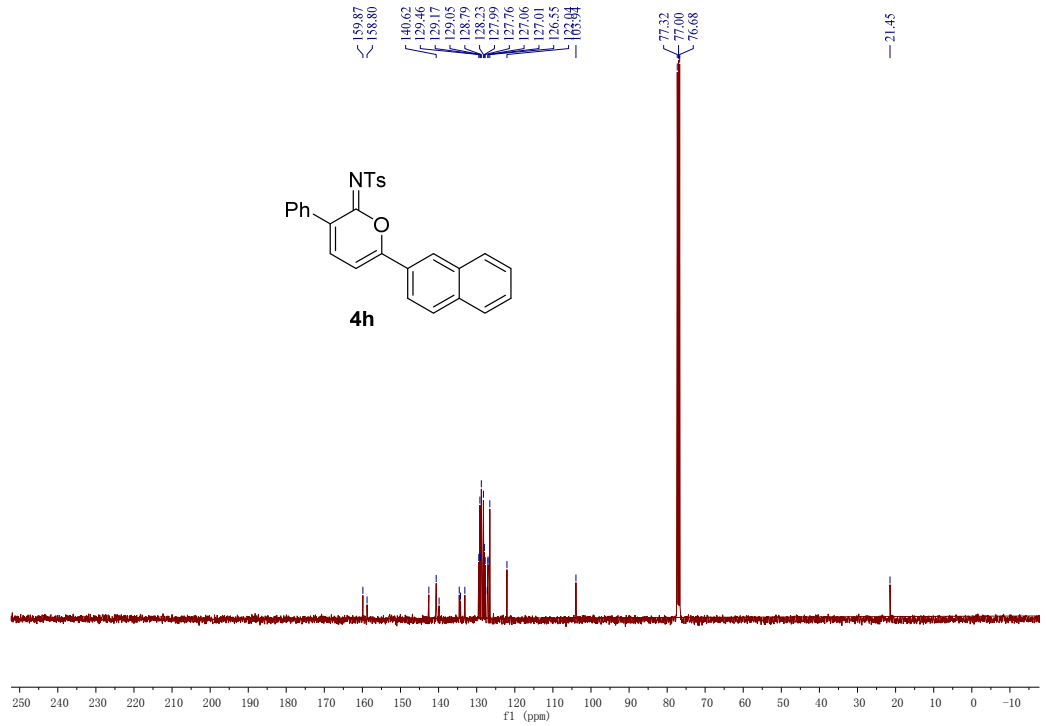
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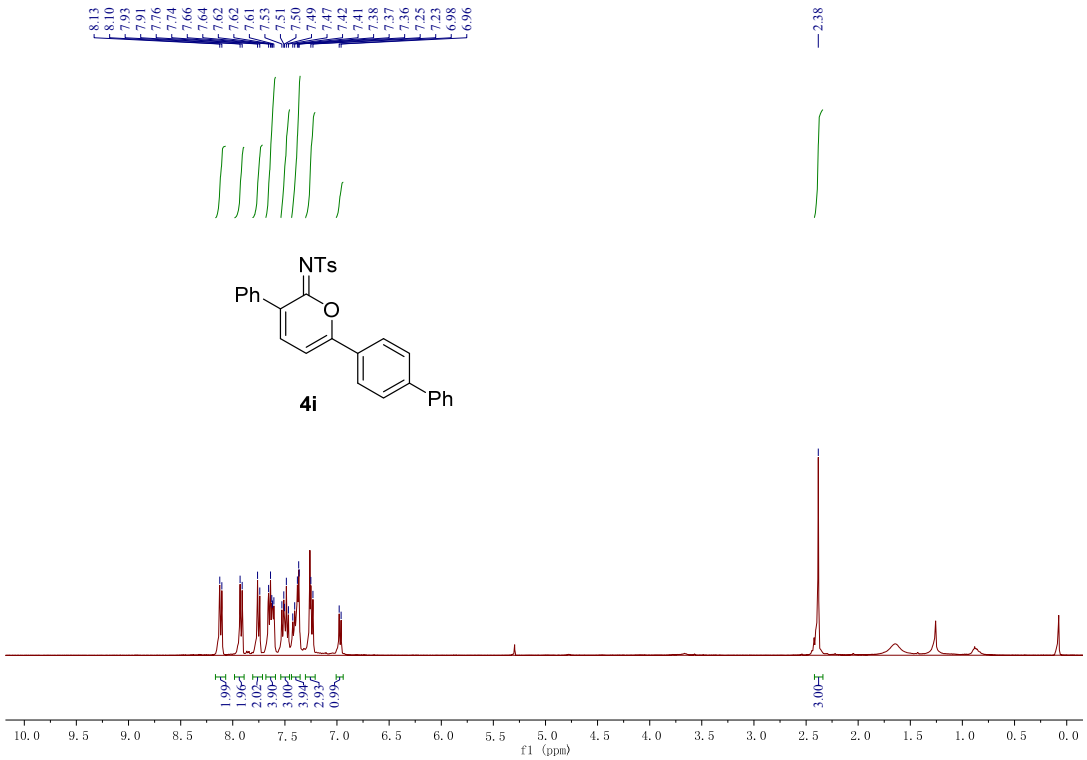
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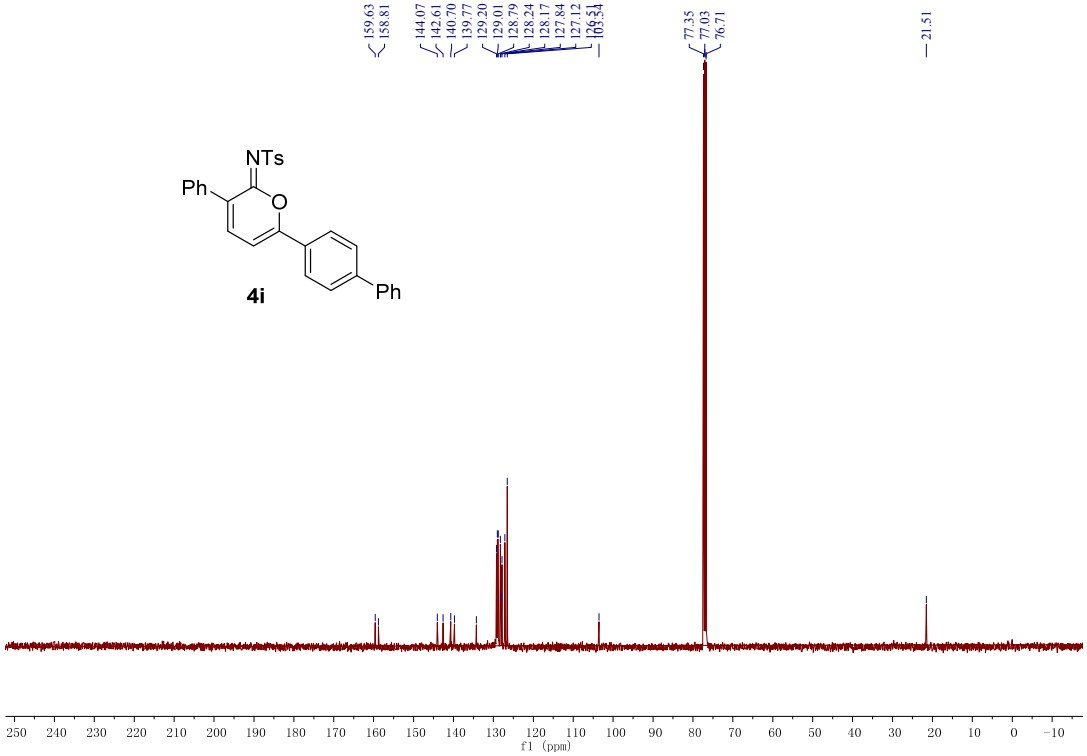
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Ph
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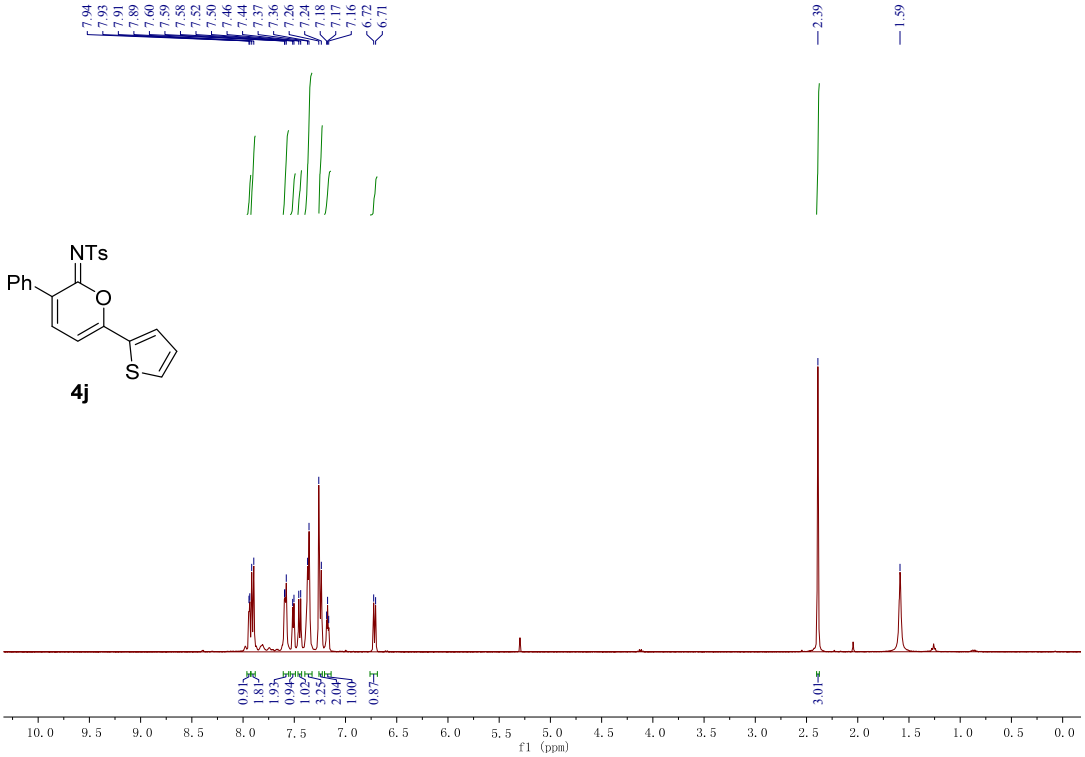
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Ph
O
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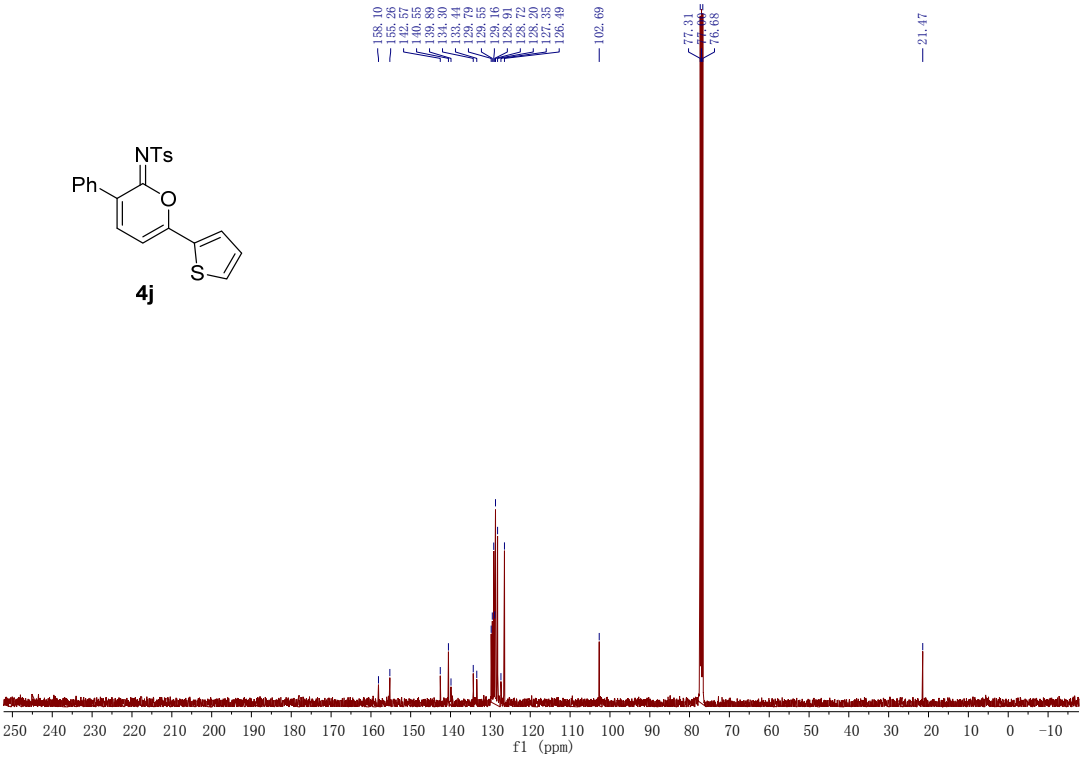
3.01
f1 (ppm)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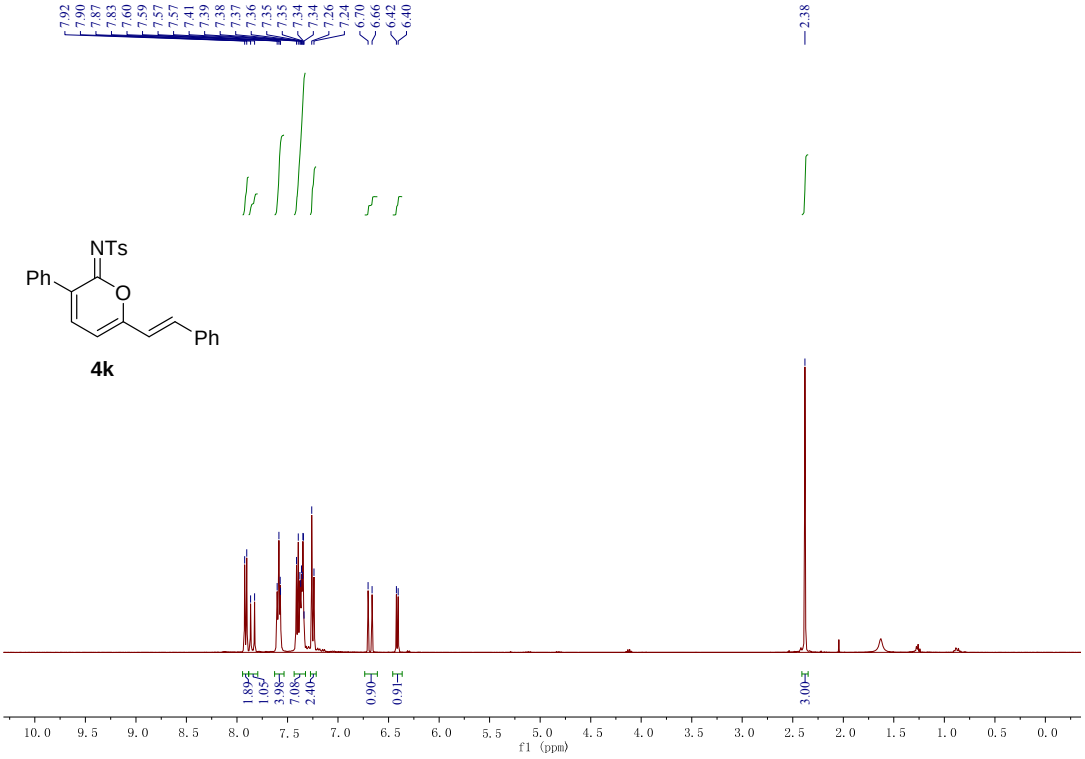
155.26
142.57
140.55
139.89
134.30
133.44
129.79
129.55
129.16
128.91
128.72
128.20
127.35
126.49
102.69
77.31
76.68
21.47
NTs
Ph
O
S
4j
f1 (ppm)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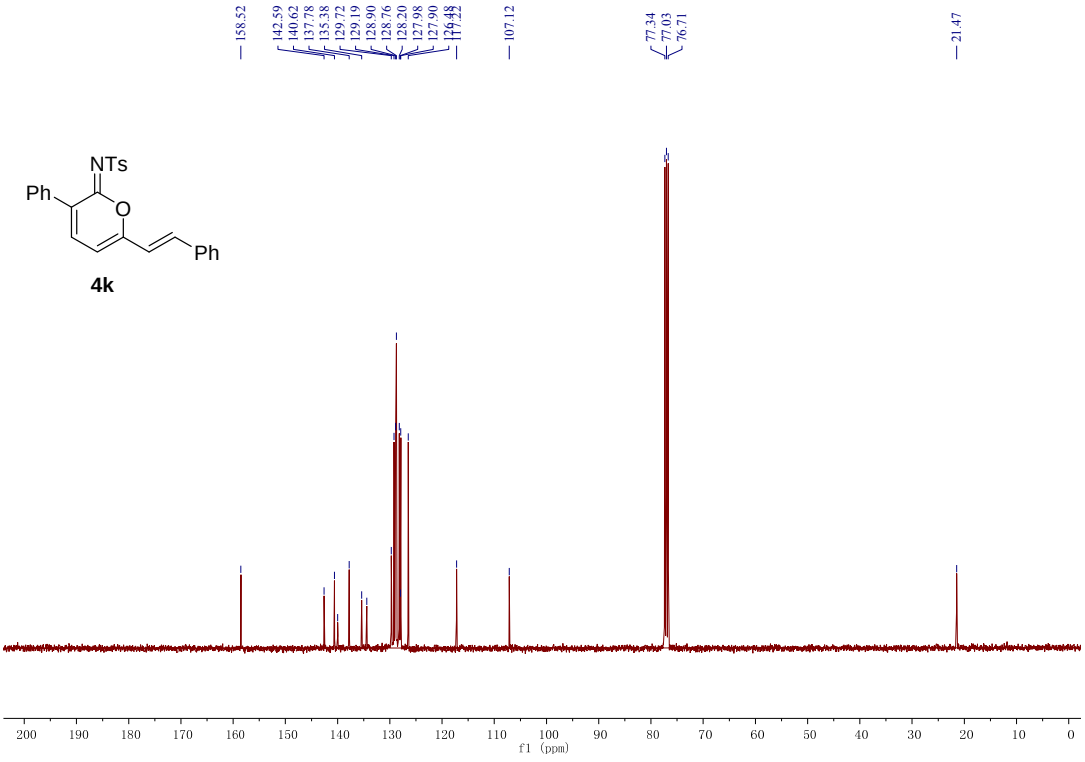
7.90
7.87
7.83
7.60
7.59
7.57
7.57
7.41
7.39
7.38
7.37
7.36
7.35
7.35
7.34
7.34
7.26
7.24
6.70
6.66
6.42
6.40
2.38
NTs
Ph
O
Ph
4k
1.89
1.05
3.98
7.08
2.40
0.90
0.91
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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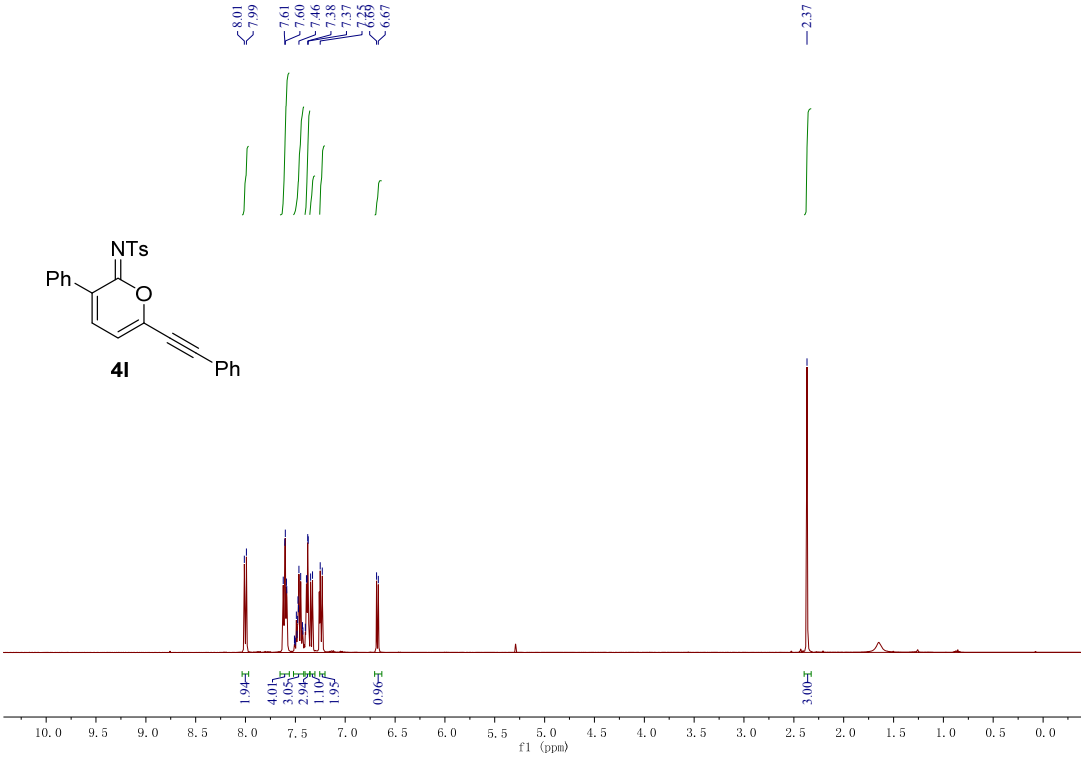
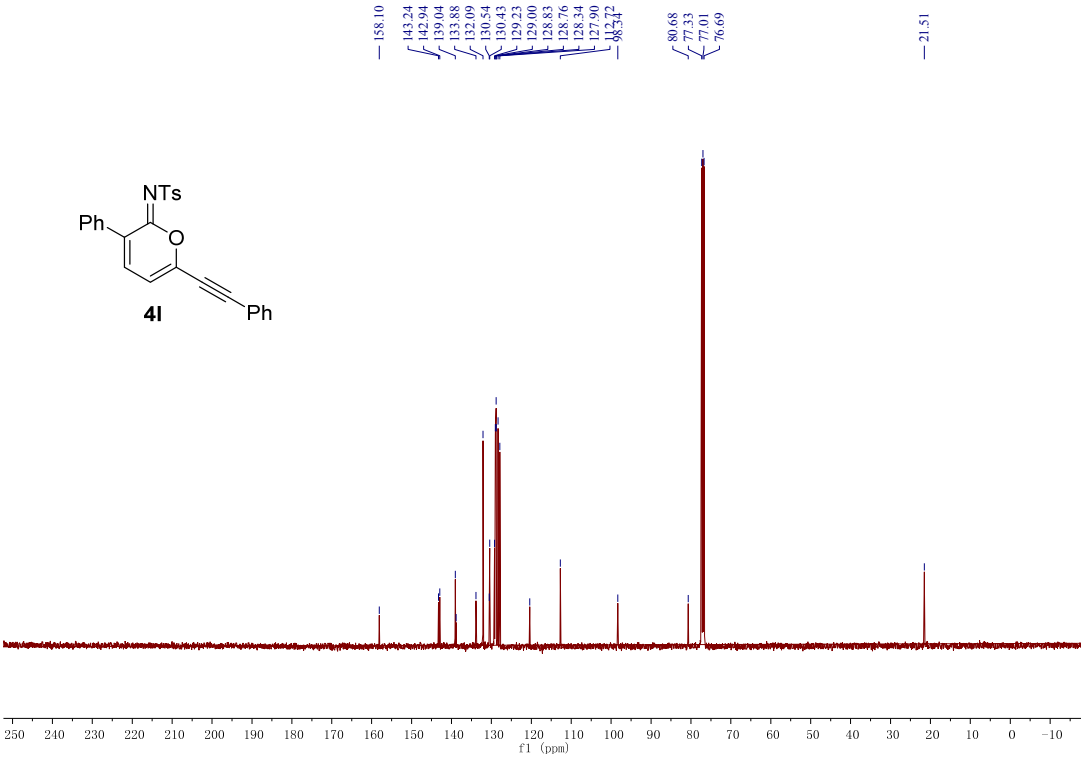
142.59
140.62
137.78
135.38
129.72
129.19
128.90
128.76
128.20
127.98
127.90
107.12
77.34
77.03
76.71
21.47
NTs
Ph
O
Ph
4k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.01
7.99
7.61
7.60
7.46
7.38
7.37
6.68
6.67
2.37
NTs
Ph
O
Ph
4l
1.94
4.01
3.05
2.94
1.10
1.95
0.96
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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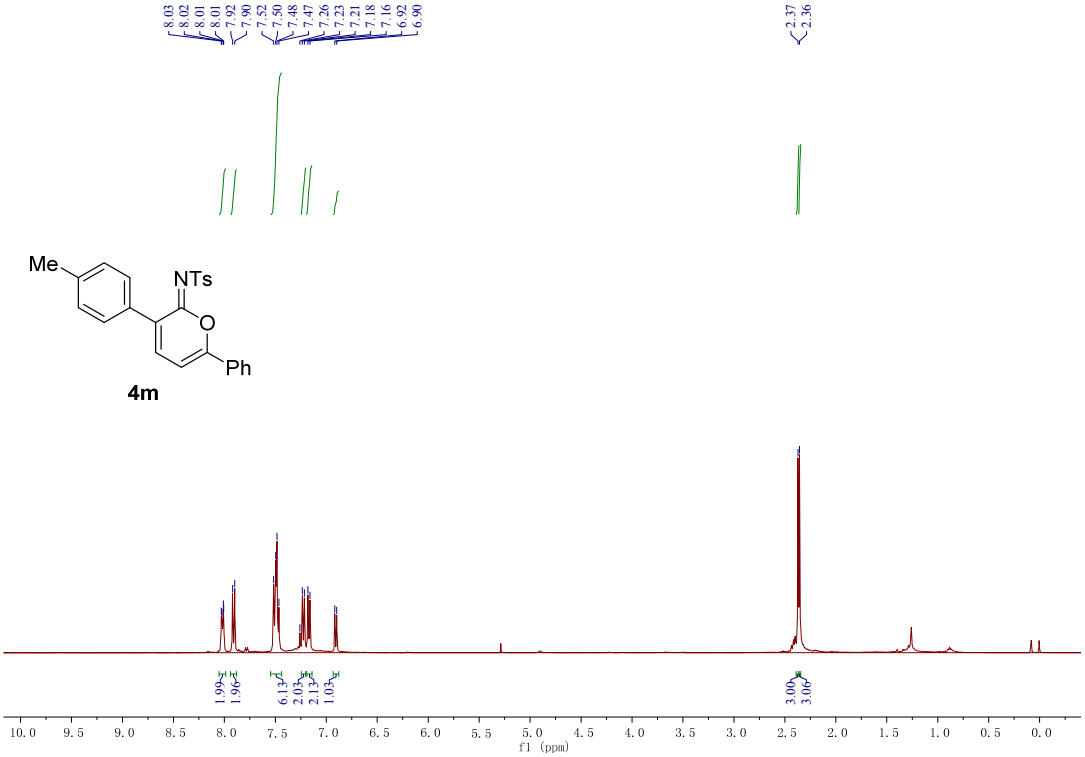
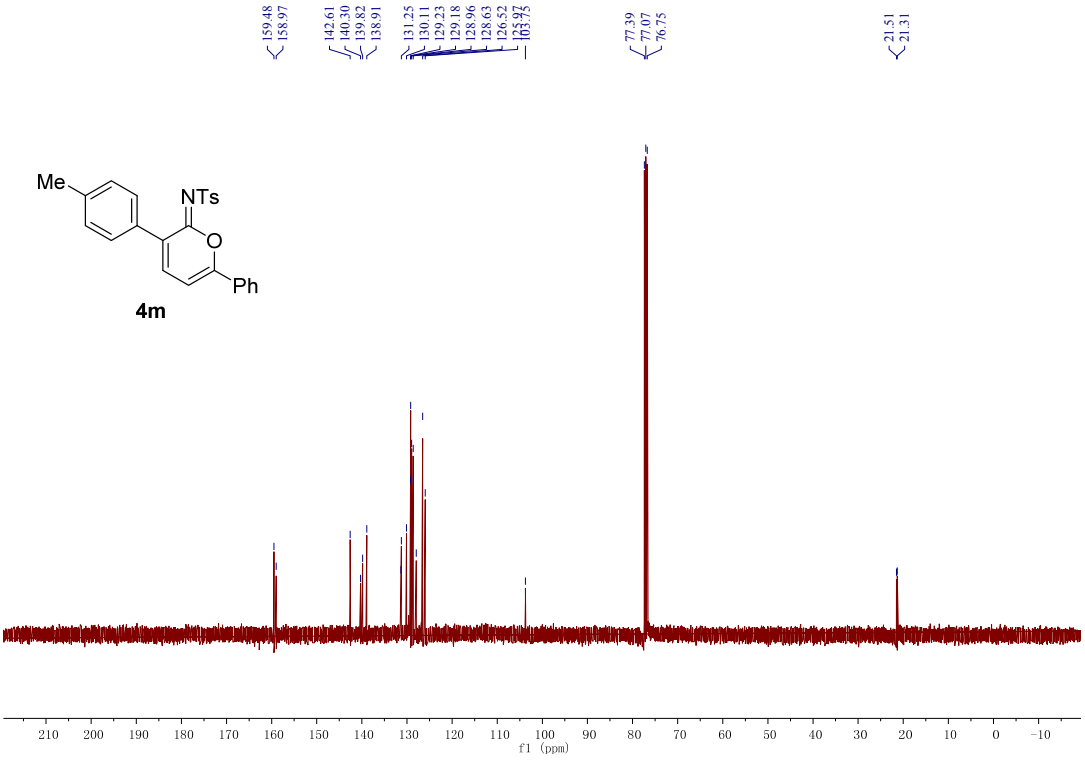
143.24
142.94
139.04
133.88
132.09
130.54
130.43
129.23
129.00
128.83
128.76
128.34
127.90
112.72
98.34
80.68
77.33
77.01
76.69
21.51
NTs
Ph
O
Ph
4l
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.03
8.02
8.01
8.01
7.92
7.90
7.52
7.50
7.48
7.47
7.26
7.23
7.21
7.18
7.16
6.92
6.90
2.37
2.36
Me
NTs
O
Ph
4m
1.99
1.96
6.13
2.03
2.13
1.03
3.00
3.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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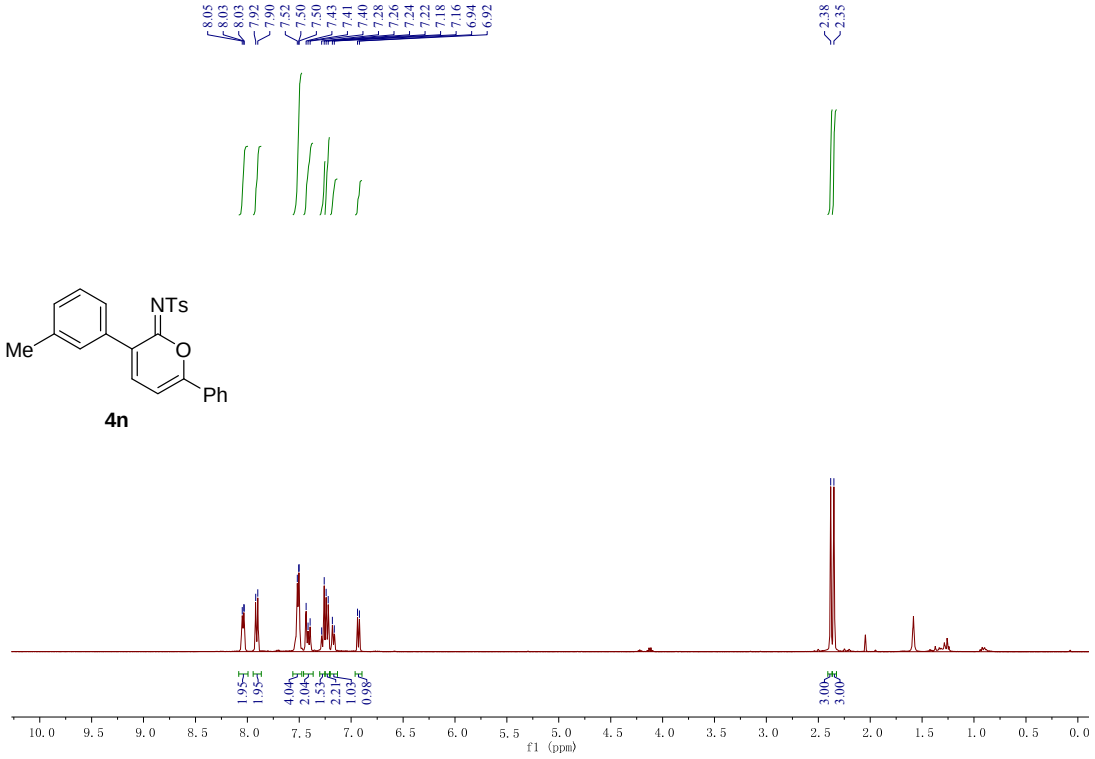
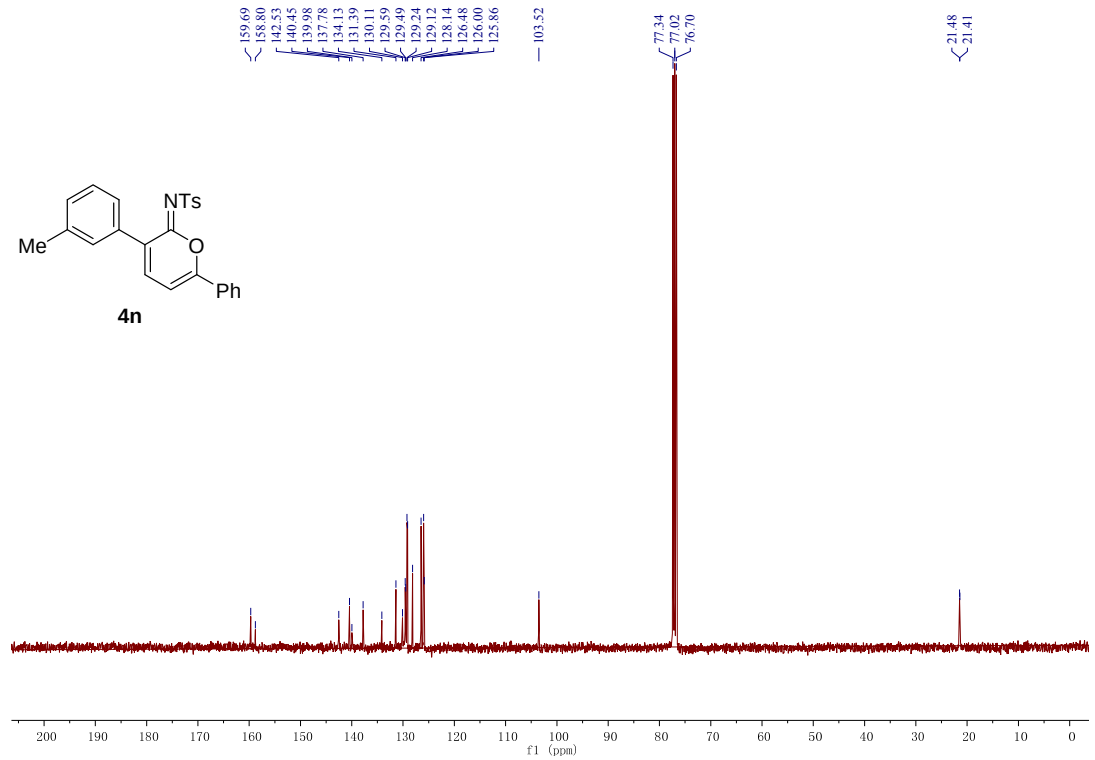
158.97
142.61
140.30
139.82
138.91
131.25
130.11
129.23
129.18
128.96
128.63
126.52
105.75
77.39
77.07
76.75
21.51
21.31
Me
NTs
O
Ph
4m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.05
8.03
8.03
7.92
7.90
7.52
7.50
7.50
7.43
7.41
7.40
7.28
7.26
7.24
7.22
7.18
7.16
6.94
6.92
2.38
2.35
NTs
Me
O
Ph
4n
1.95
1.95
4.04
2.04
1.53
2.21
1.03
0.98
3.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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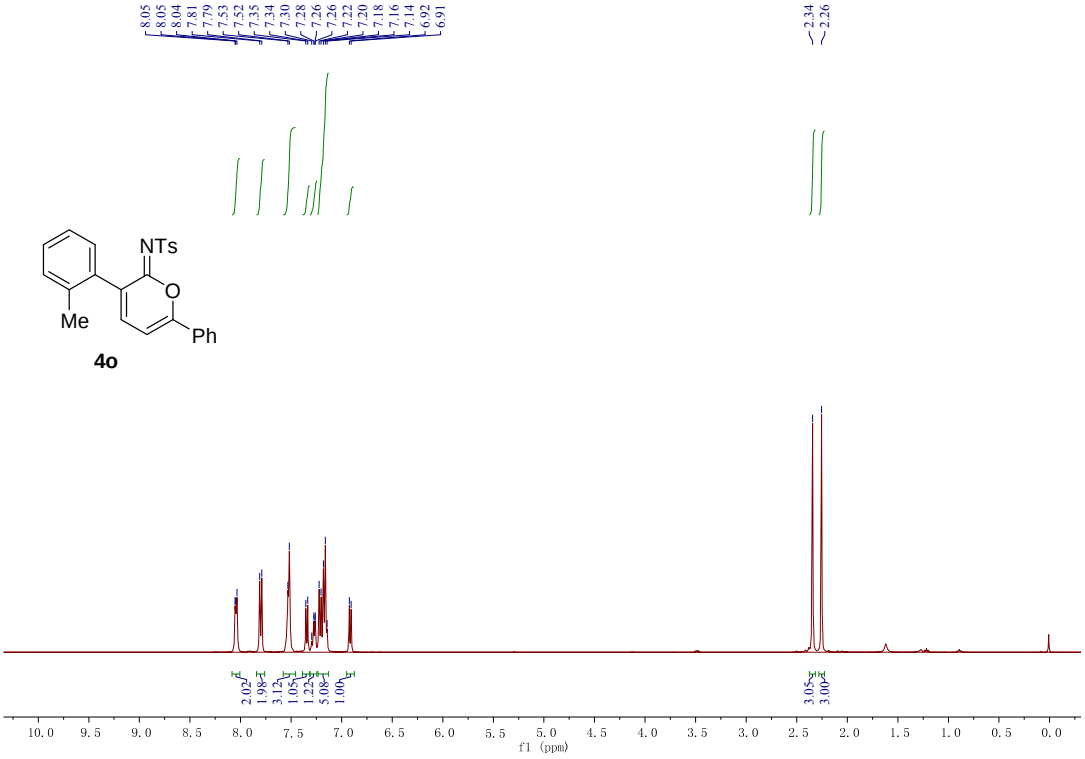
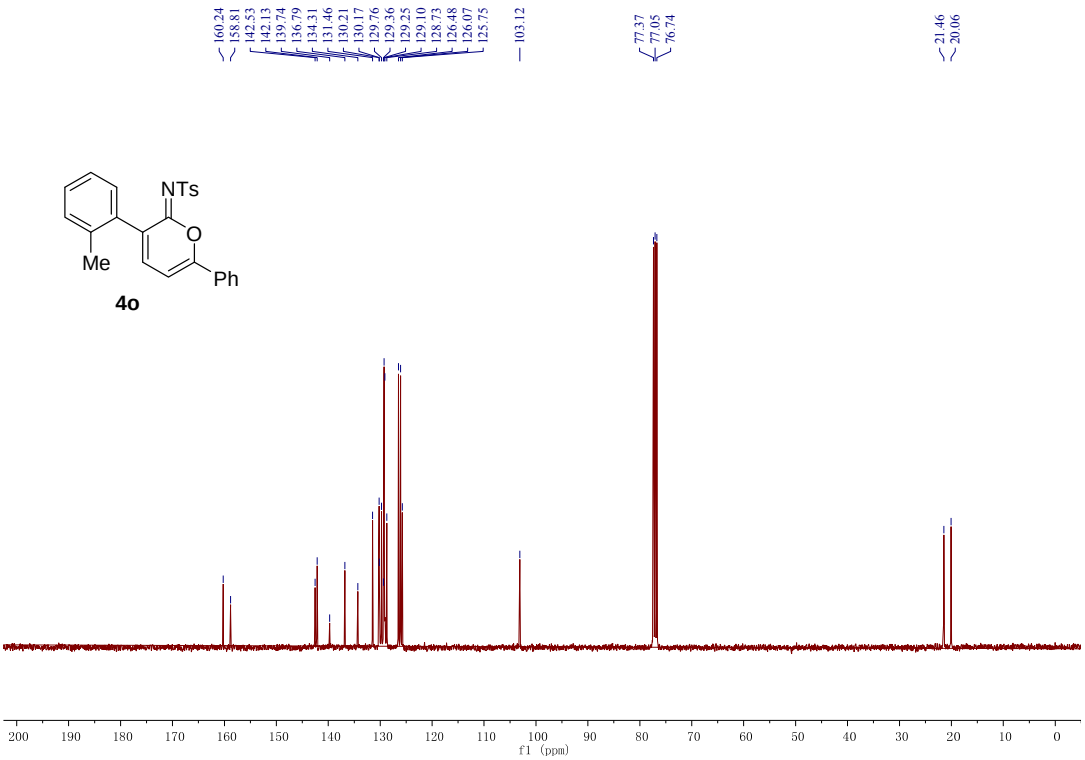
158.80
142.53
140.45
139.98
137.78
134.13
131.39
130.11
129.59
129.49
129.24
129.12
128.14
126.48
126.00
125.86
103.52
77.34
77.02
76.70
21.48
21.41
NTs
Me
O
Ph
4n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.05
8.05
8.04
7.81
7.79
7.53
7.52
7.35
7.34
7.30
7.28
7.26
7.26
7.22
7.20
7.18
7.16
7.14
6.92
6.91
2.34
2.26
NTs
O
Me
Ph
4o
2.02
1.98
3.12
1.05
1.22
5.08
1.00
3.05
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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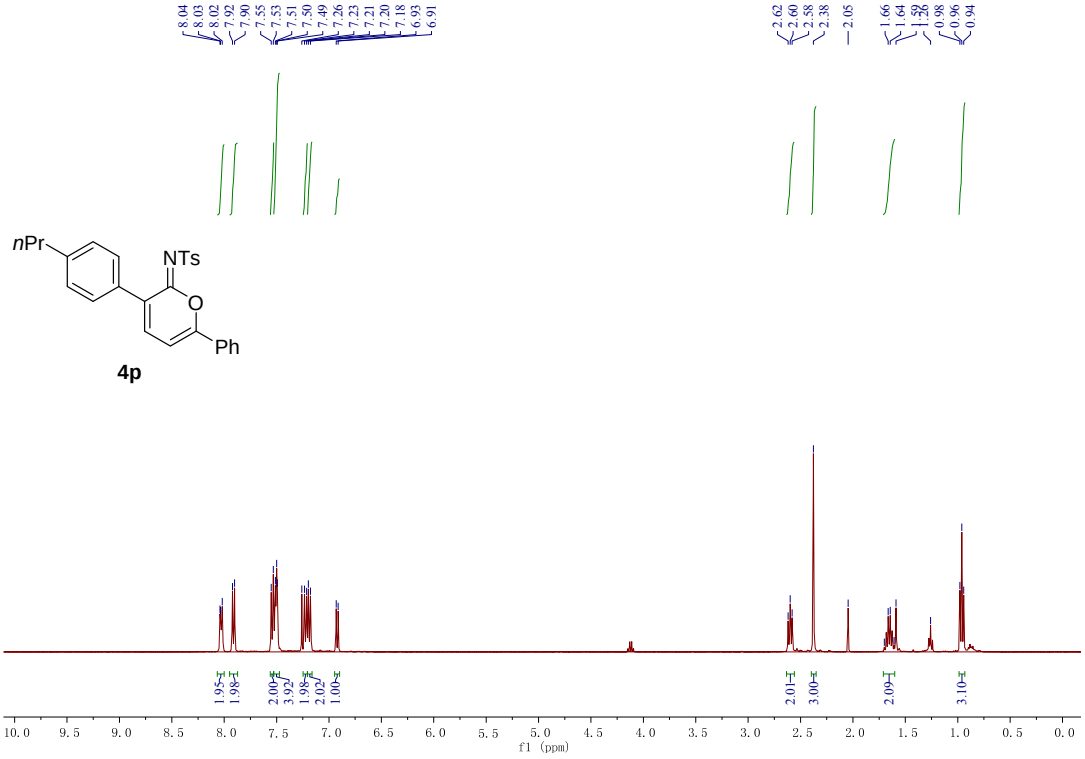
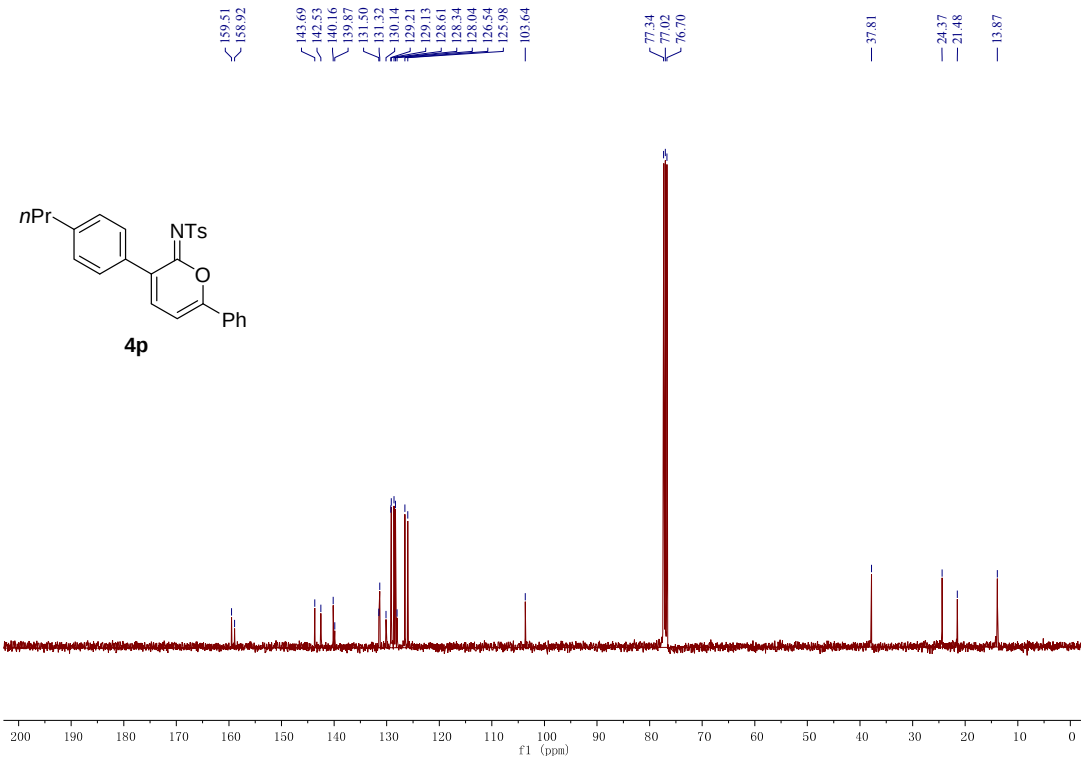
158.81
142.53
142.13
139.74
136.79
134.31
131.46
130.21
130.17
129.76
129.36
129.25
129.10
128.73
126.48
126.07
125.75
103.12
77.37
77.05
76.74
21.46
20.06
NTs
O
Me
Ph
4o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.04
8.03
8.02
7.92
7.90
7.55
7.53
7.51
7.50
7.49
7.26
7.23
7.21
7.20
7.18
6.93
6.91
2.62
2.60
2.58
2.38
2.05
1.66
1.64
1.59
1.28
0.98
0.96
0.94
nPr
NTs
O
Ph
4p
1.95
1.98
2.00
3.92
1.98
2.02
1.00
2.01
3.00
2.09
3.10
f1 (ppm)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158.92
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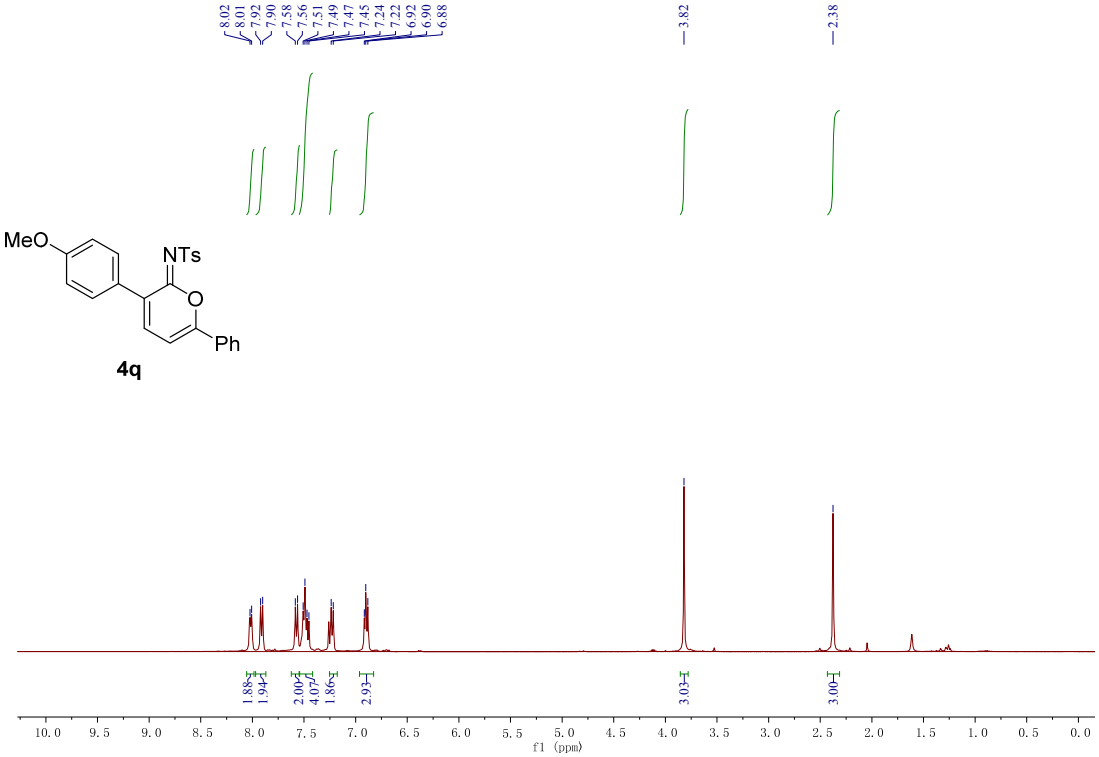
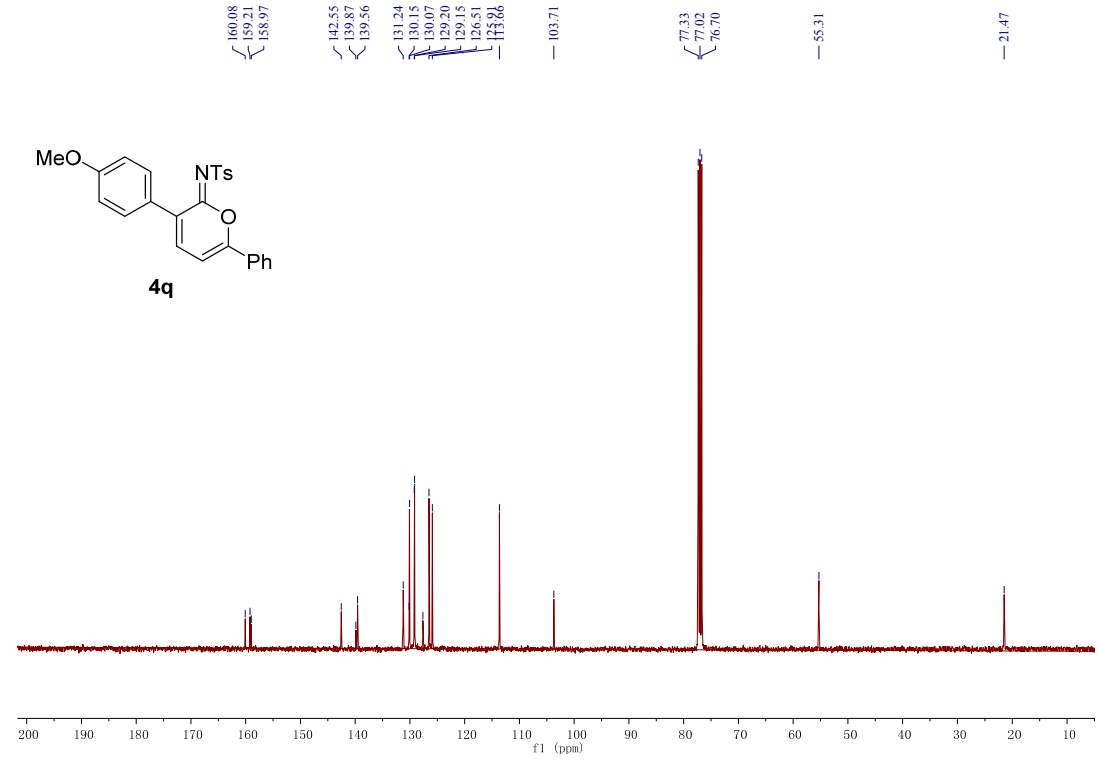
140.16
139.87
131.50
131.32
130.14
129.21
129.13
128.61
128.34
128.04
126.54
125.98
103.64
77.34
77.02
76.70
37.81
24.37
21.48
13.87
nPr
NTs
O
Ph
4p
f1 (ppm)


8.02
8.01
7.92
7.90
7.58
7.56
7.51
7.49
7.47
7.45
7.24
7.22
6.92
6.90
6.88
3.82
2.38
MeO
NTs
O
Ph
4q
1.88
1.94
2.00
4.07
1.86
2.93
3.03
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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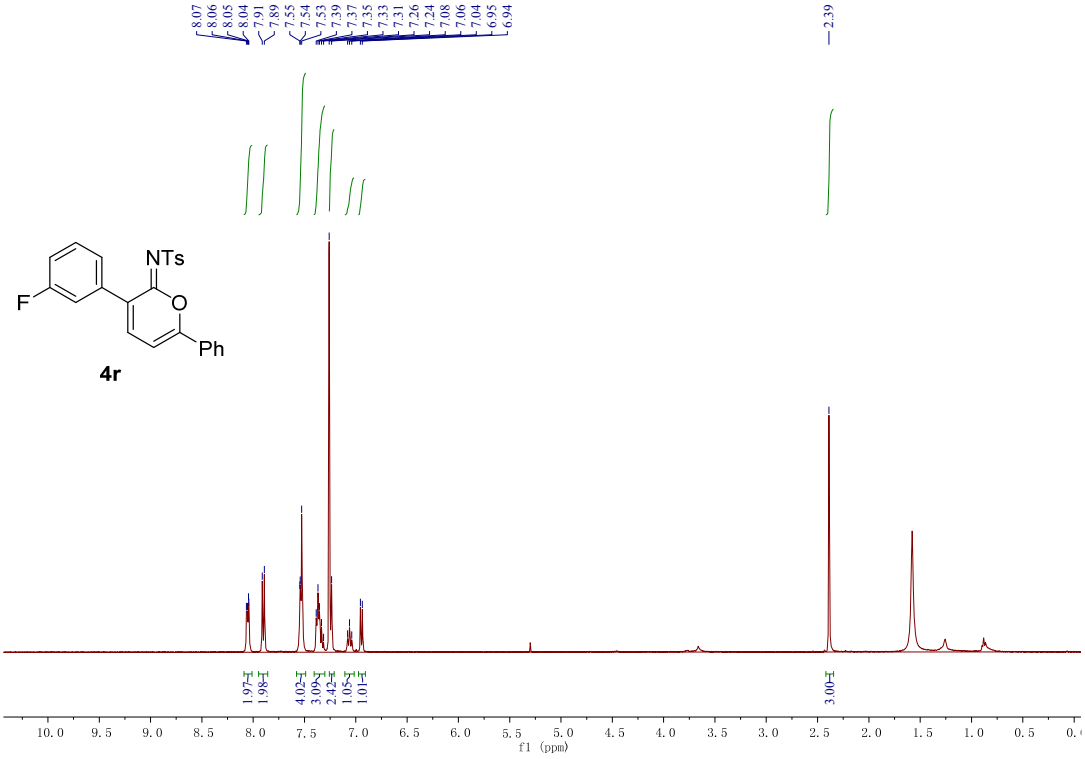
159.21
158.97
142.55
139.87
139.56
131.24
130.15
130.07
129.20
129.15
126.51
113.86
103.71
77.33
77.02
76.70
55.31
21.47
MeO
NTs
O
Ph
4q
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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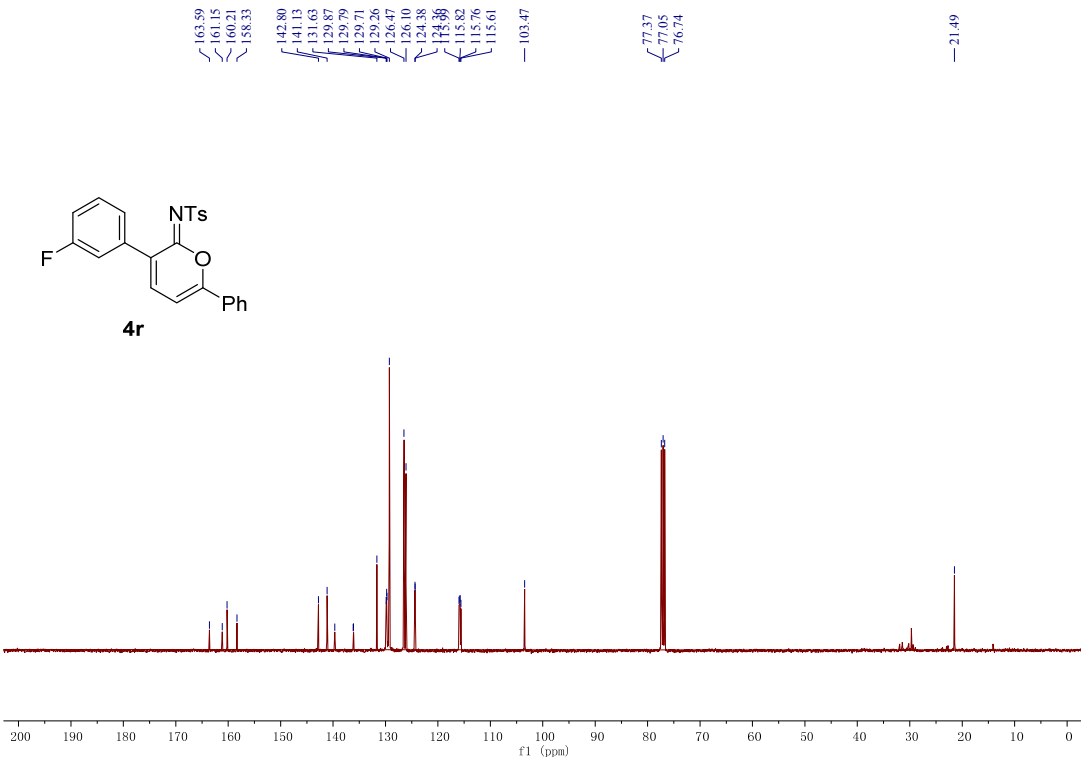
8.06
8.05
8.04
7.91
7.89
7.55
7.54
7.53
7.39
7.37
7.35
7.33
7.31
7.26
7.24
7.08
7.06
7.04
6.95
6.94
2.39
NTs
F
O
Ph
4r
1.97
1.98
4.02
3.09
2.42
1.05
1.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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158.33
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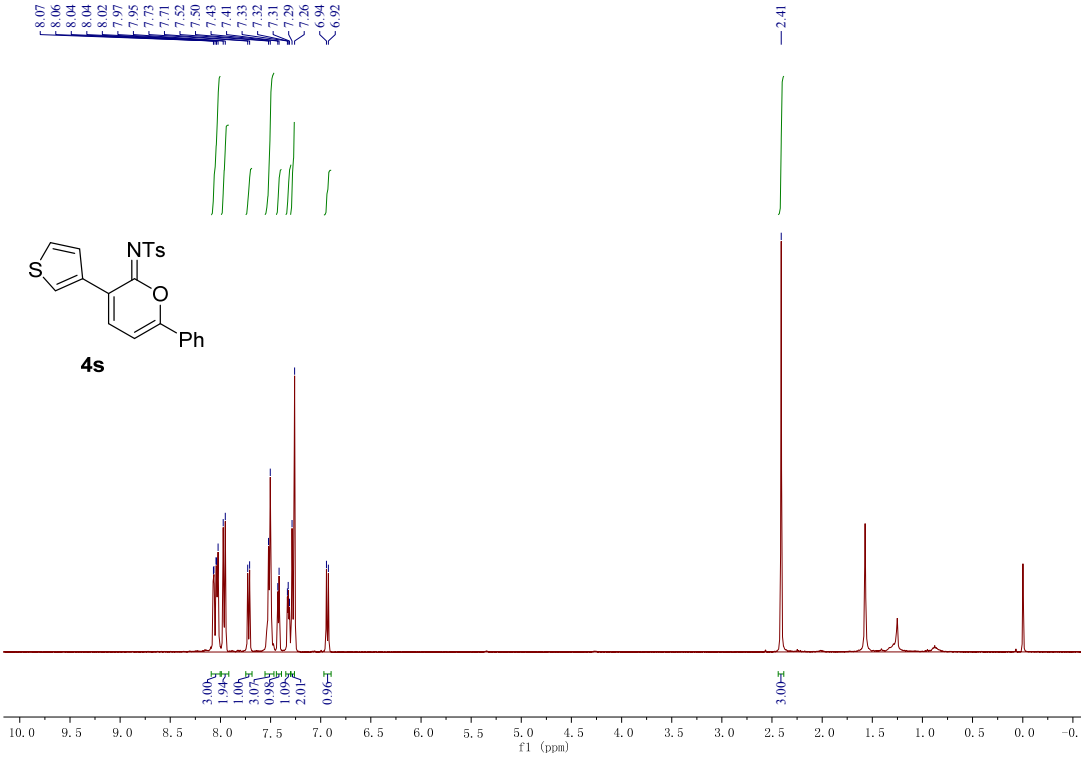
141.13
131.63
129.87
129.79
129.71
129.26
126.47
126.10
124.38
115.82
115.76
115.61
103.47
77.37
77.05
76.74
21.49
NTs
F
O
Ph
4r
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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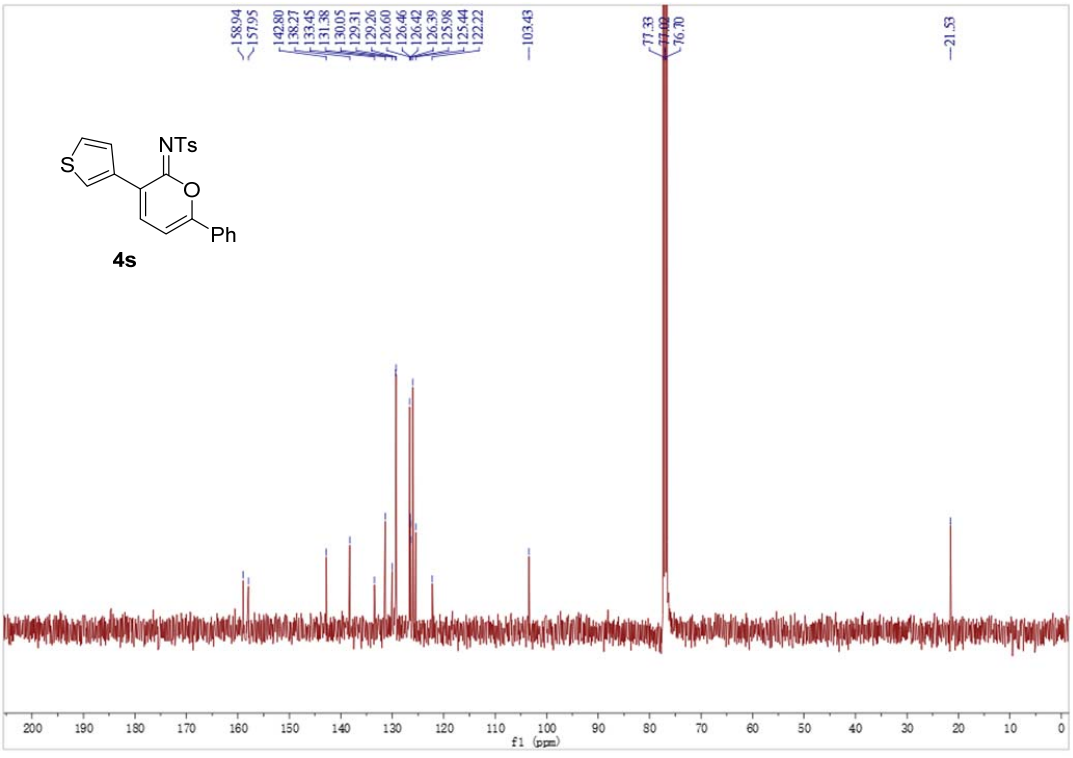
8.06
8.04
8.04
8.02
7.97
7.95
7.73
7.71
7.52
7.50
7.43
7.41
7.33
7.32
7.31
7.29
7.26
6.94
6.92
2.41
NTs
S
O
Ph
4s
3.00
1.94
1.00
3.07
0.98
1.09
2.01
0.96
3.00
f1 (ppm)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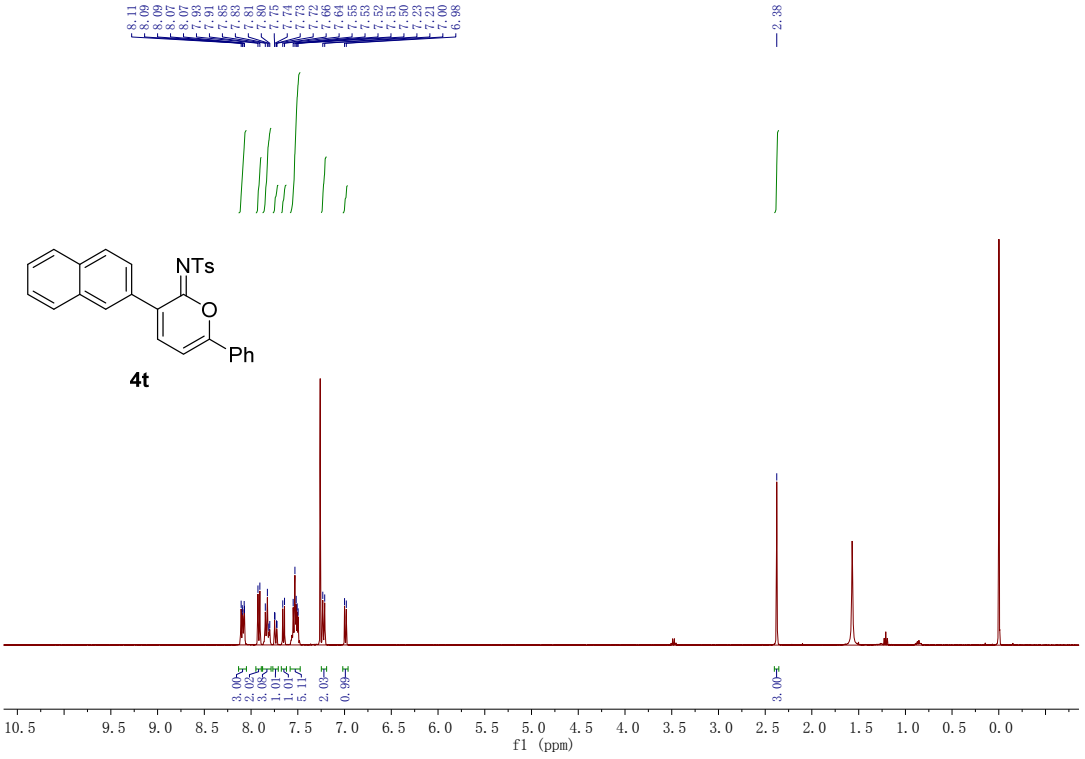
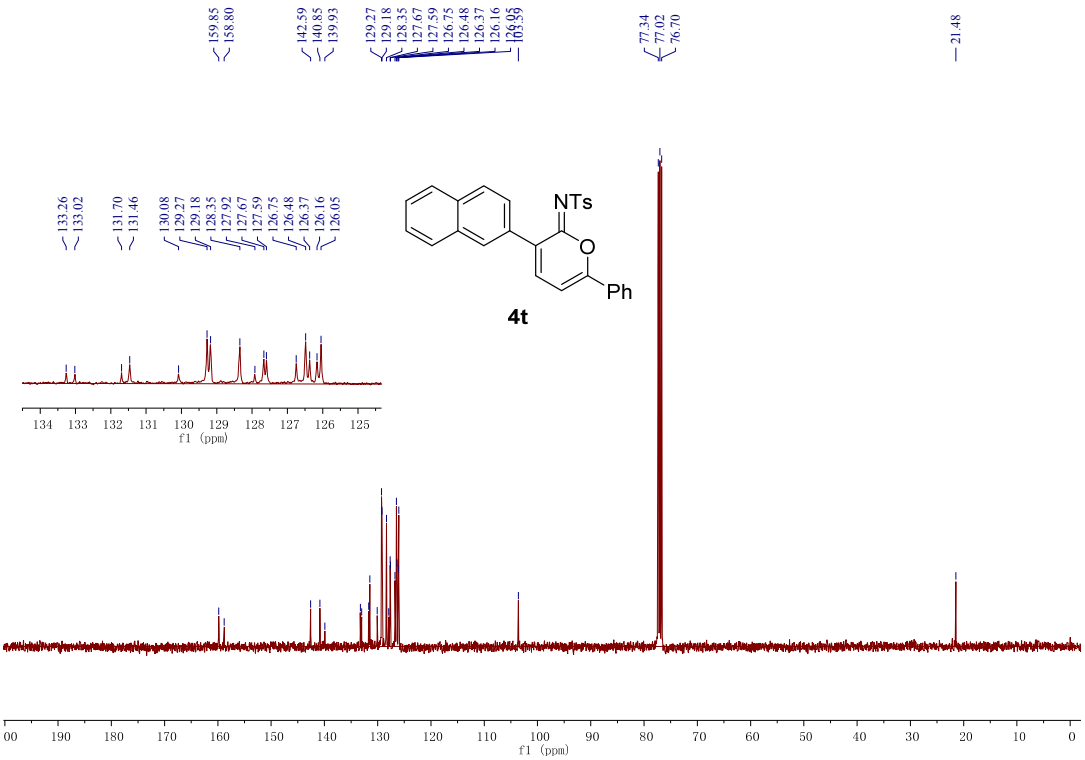
157.95
142.80
138.27
133.45
131.38
130.05
129.31
129.26
126.60
126.46
126.42
126.39
125.98
125.44
122.22
103.43
77.33
77.00
76.70
21.53
NTs
S
O
Ph
4s
f1 (ppm)


8.11
8.09
8.09
8.07
8.07
7.93
7.91
7.85
7.83
7.81
7.80
7.75
7.74
7.73
7.72
7.66
7.64
7.55
7.53
7.52
7.51
7.50
7.23
7.21
7.00
6.98
2.38
NTs
O
Ph
4t
3.00
2.02
3.08
1.00
1.01
5.11
2.03
0.99
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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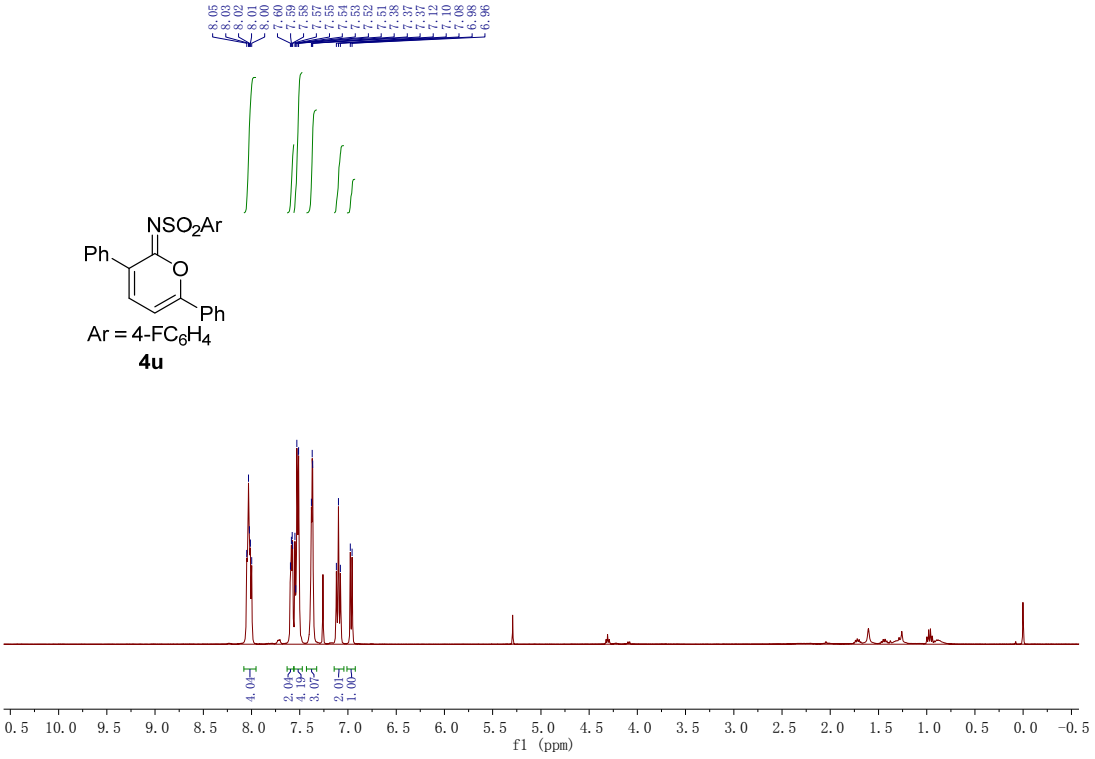
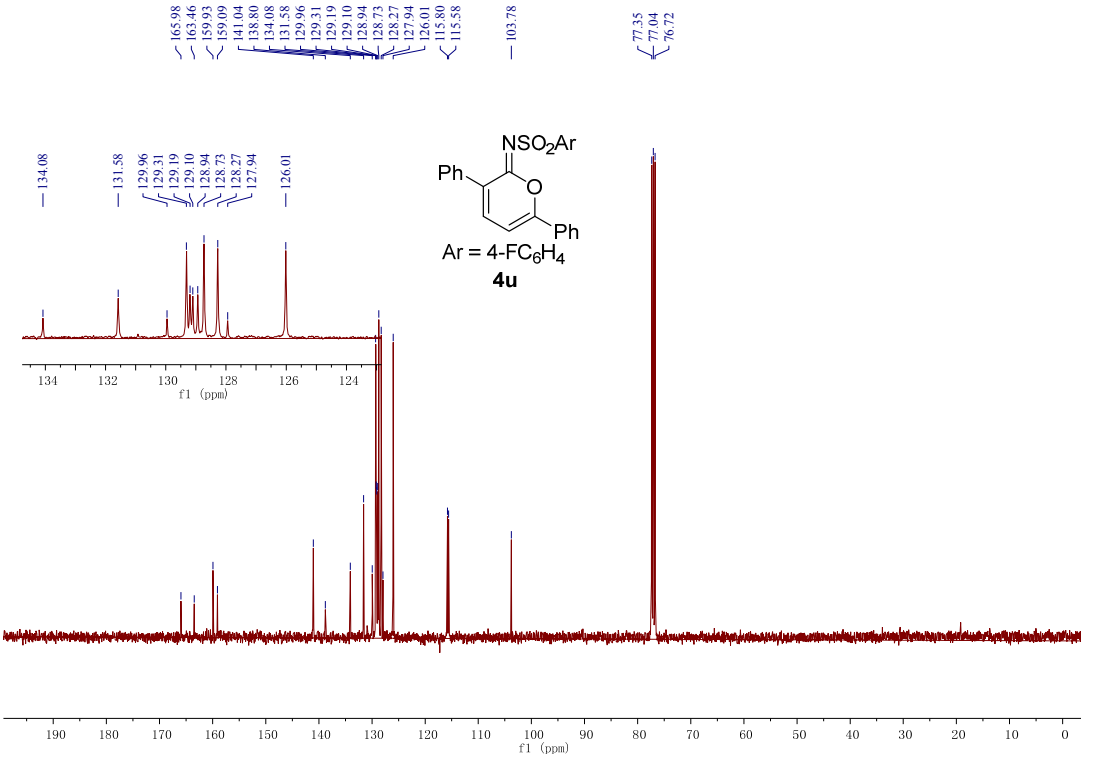
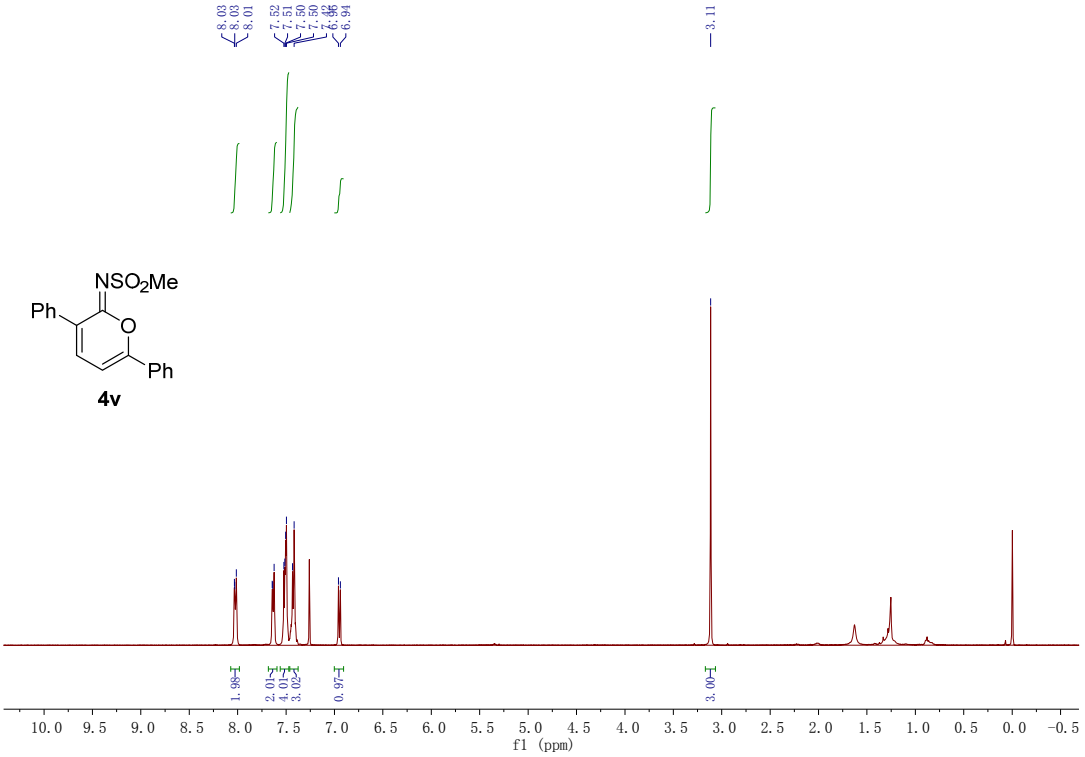
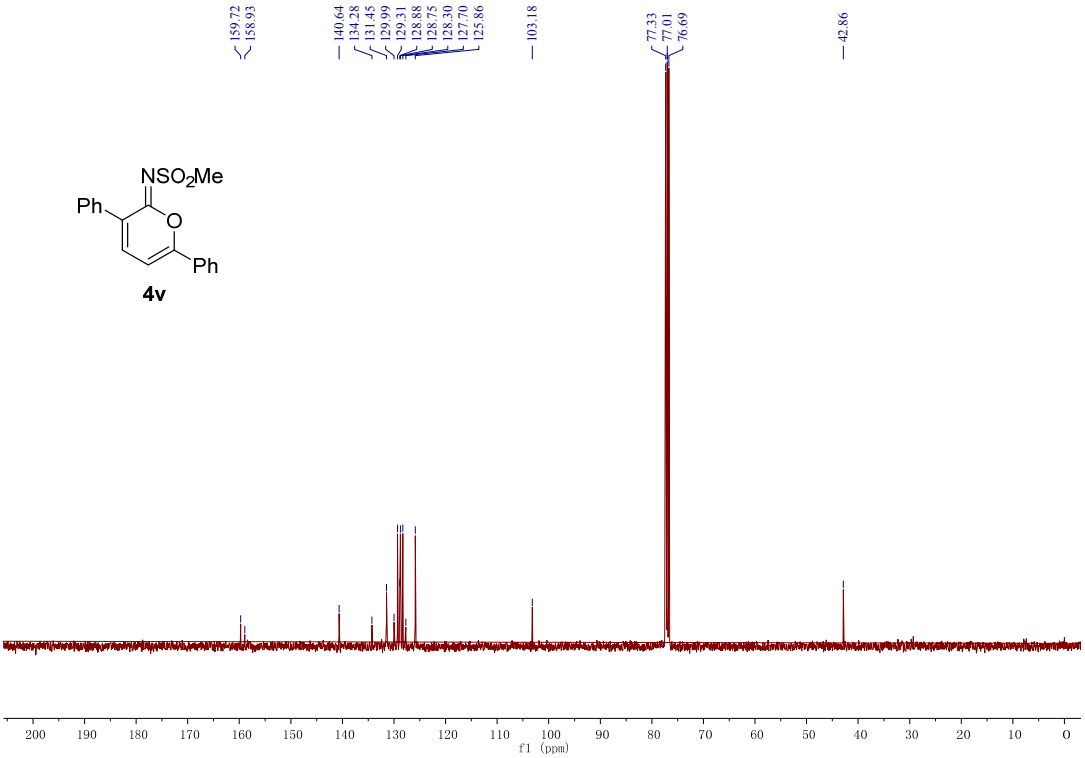
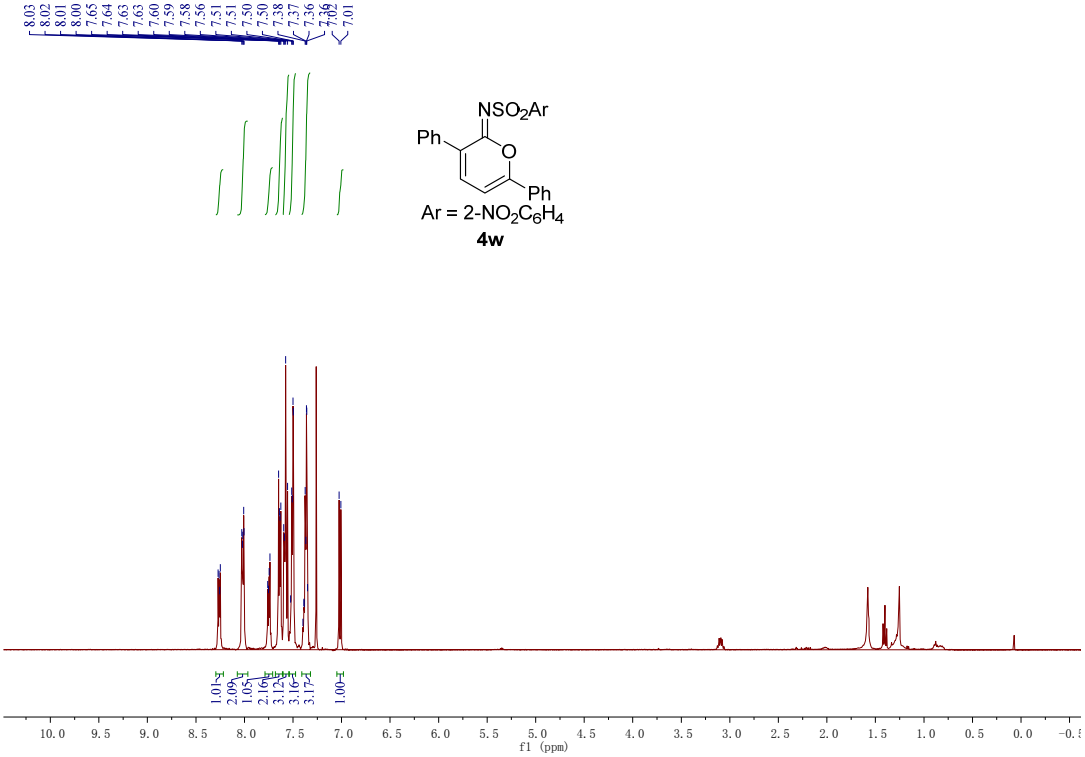
129.18
128.35
127.67
127.59
126.75
126.48
126.37
126.16
126.05
103.59
77.34
77.02
76.70
21.48
133.26
133.02
131.70
131.46
130.08
129.27
129.18
128.35
127.92
127.67
127.59
126.75
126.48
126.37
126.16
126.05
NTs
O
Ph
4t
134 133 132 131 130 129 128 127 126 125
f1 (ppm)
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

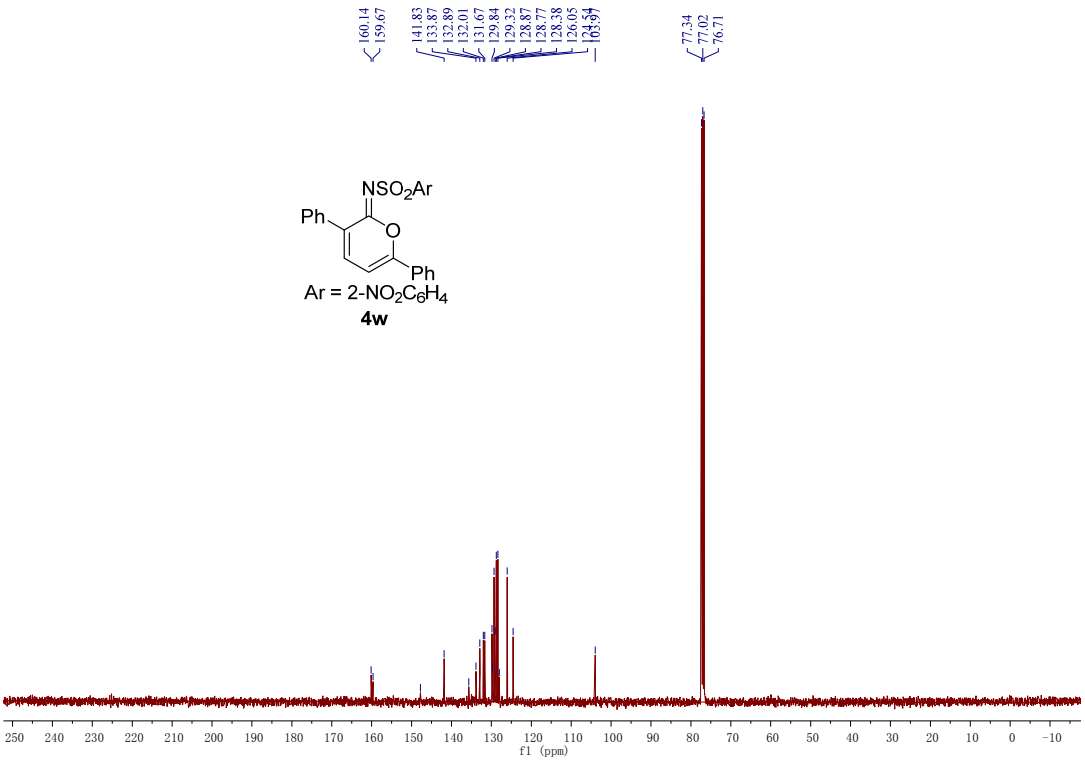

8.05
8.03
8.02
8.01
8.00
7.60
7.59
7.58
7.57
7.55
7.54
7.53
7.52
7.51
7.38
7.37
7.37
7.12
7.10
7.08
6.98
6.96
NSO2Ar
Ph
O
Ph
Ar = 4-FC6H4
4u
4.04
2.04
4.19
3.07
2.01
1.00
f1 (ppm)

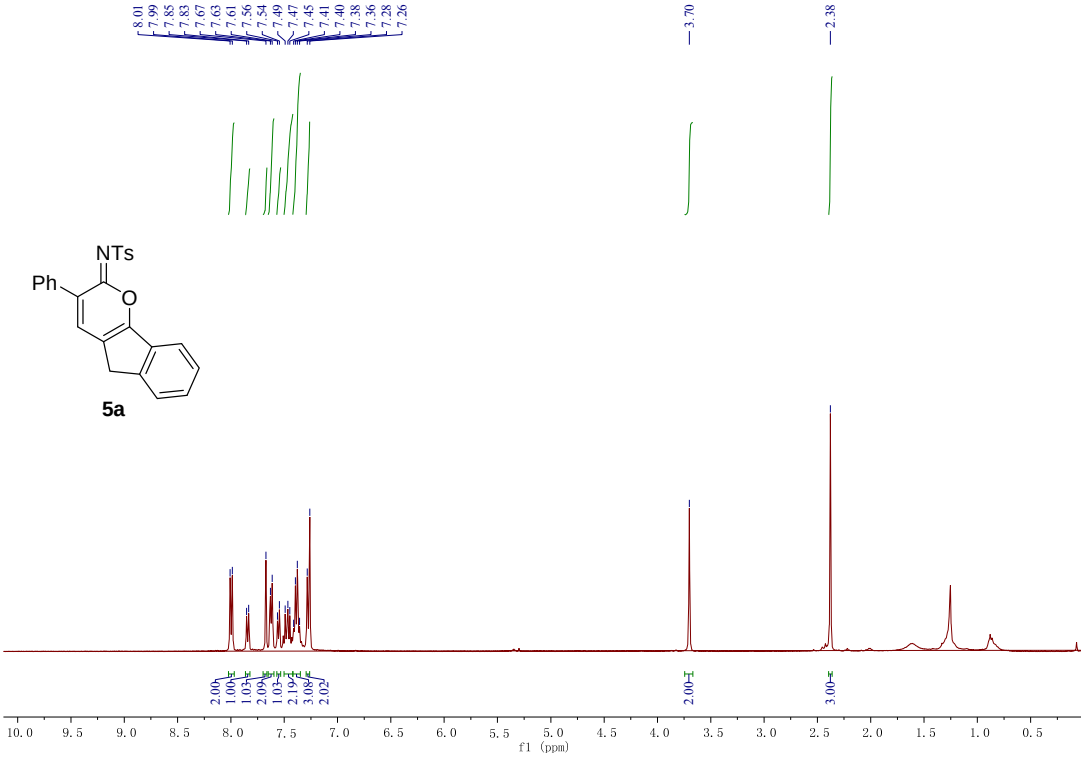

165.98
163.46
159.93
159.09
141.04
138.80
134.08
131.58
129.96
129.31
129.19
129.10
128.94
128.73
128.27
127.94
126.01
115.80
115.58
103.78
77.35
77.04
76.72
134.08
131.58
129.96
129.31
129.19
129.10
128.94
128.73
128.27
127.94
126.01
NSO2Ar
Ph
O
Ph
Ar = 4-FC6H4
4u
f1 (ppm)
f1 (ppm)

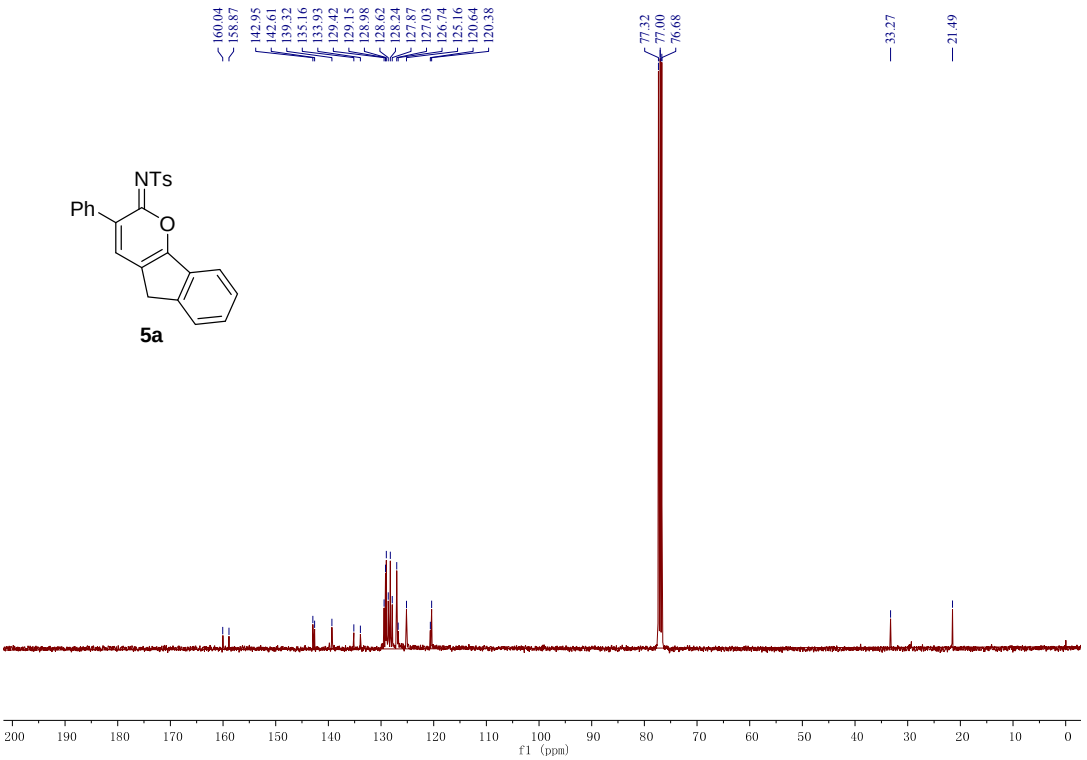

8.03
8.03
8.01
7.52
7.51
7.50
7.50
6.94
3.11
NSO2Me
Ph
O
Ph
4v
1.98
2.01
4.01
3.02
0.97
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

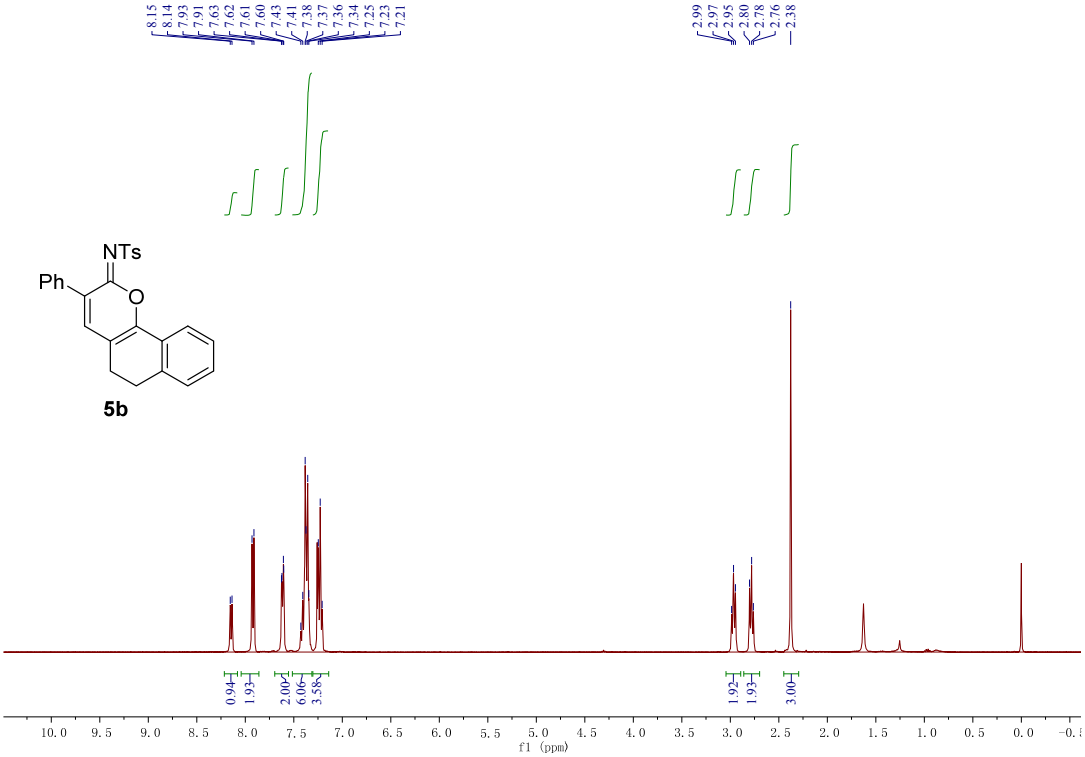

159.72
158.93
140.64
134.28
131.45
129.99
129.31
128.88
128.75
128.30
127.70
125.86
103.18
77.33
77.01
76.69
42.86
NSO2Me
Ph
O
Ph
4v
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

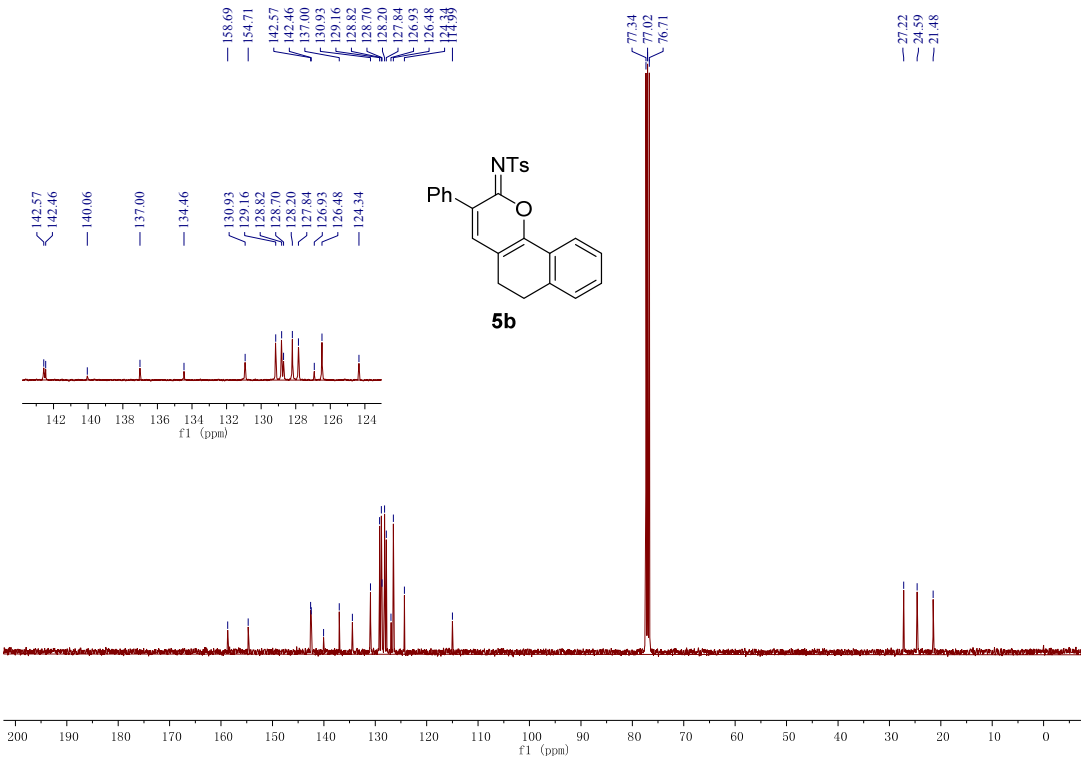

8.03
8.02
8.01
8.00
7.65
7.64
7.63
7.60
7.59
7.58
7.56
7.51
7.51
7.50
7.50
7.38
7.37
7.36
7.02
7.01
NSO2Ar
Ph
O
Ph
Ar = 2-NO2C6H4
4w
1.01
2.09
1.05
2.16
3.12
3.16
3.17
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

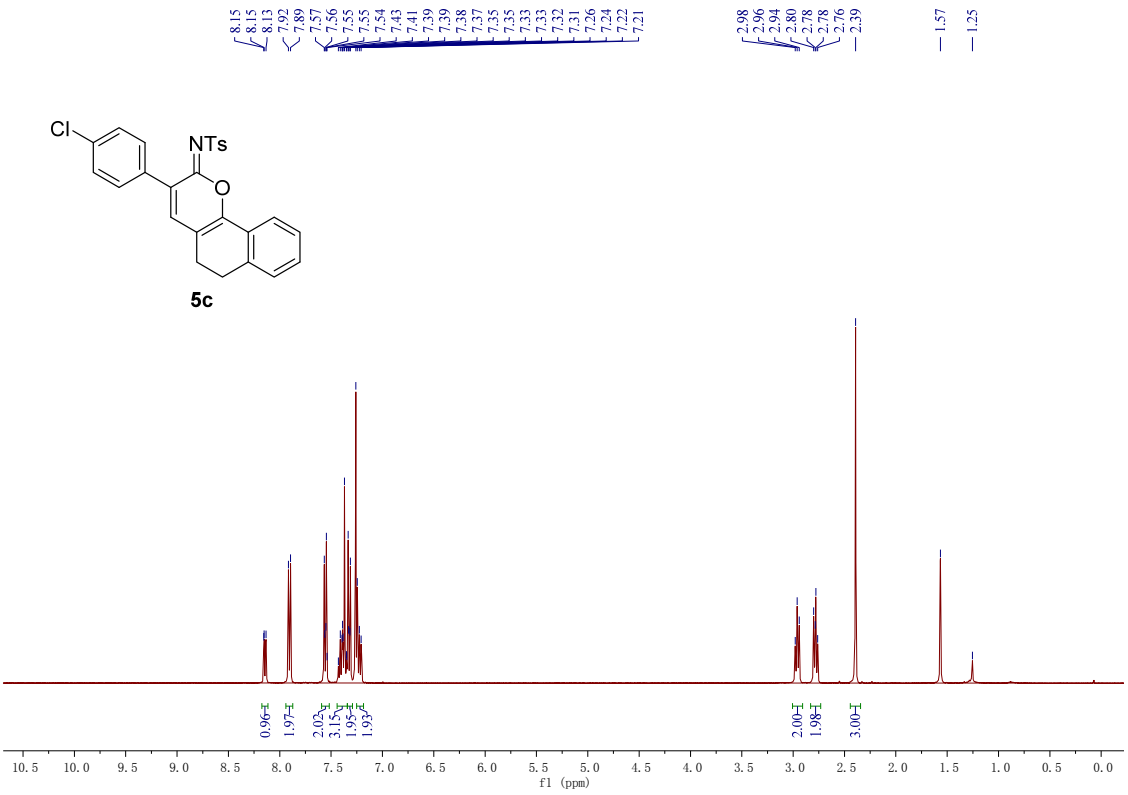

160.14
159.67
141.83
133.87
132.89
132.01
131.67
129.84
129.32
128.87
128.77
128.38
126.05
77.34
77.02
76.71
NSO2Ar
Ph
O
Ph
Ar = 2-NO2C6H4
4w
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

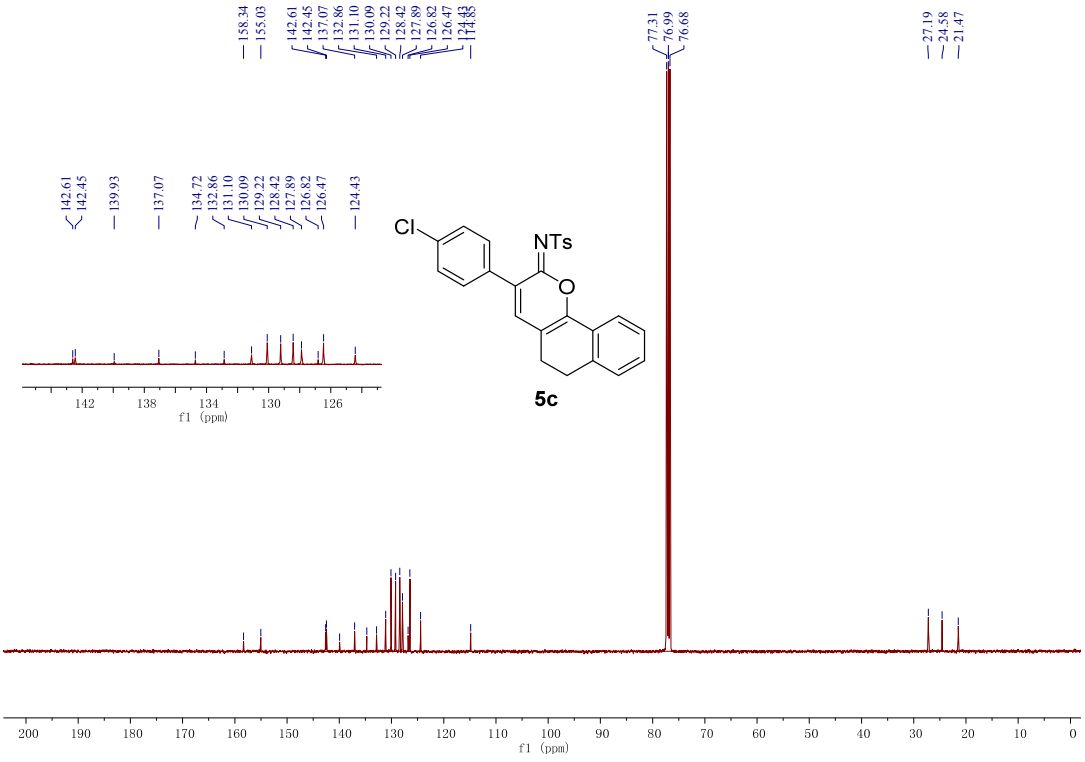

8.01
7.99
7.85
7.83
7.67
7.63
7.61
7.56
7.54
7.49
7.47
7.45
7.41
7.40
7.38
7.36
7.28
7.26
3.70
2.38
NTs
Ph
O
5a
2.00
1.00
1.03
2.09
1.03
2.19
3.08
2.02
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

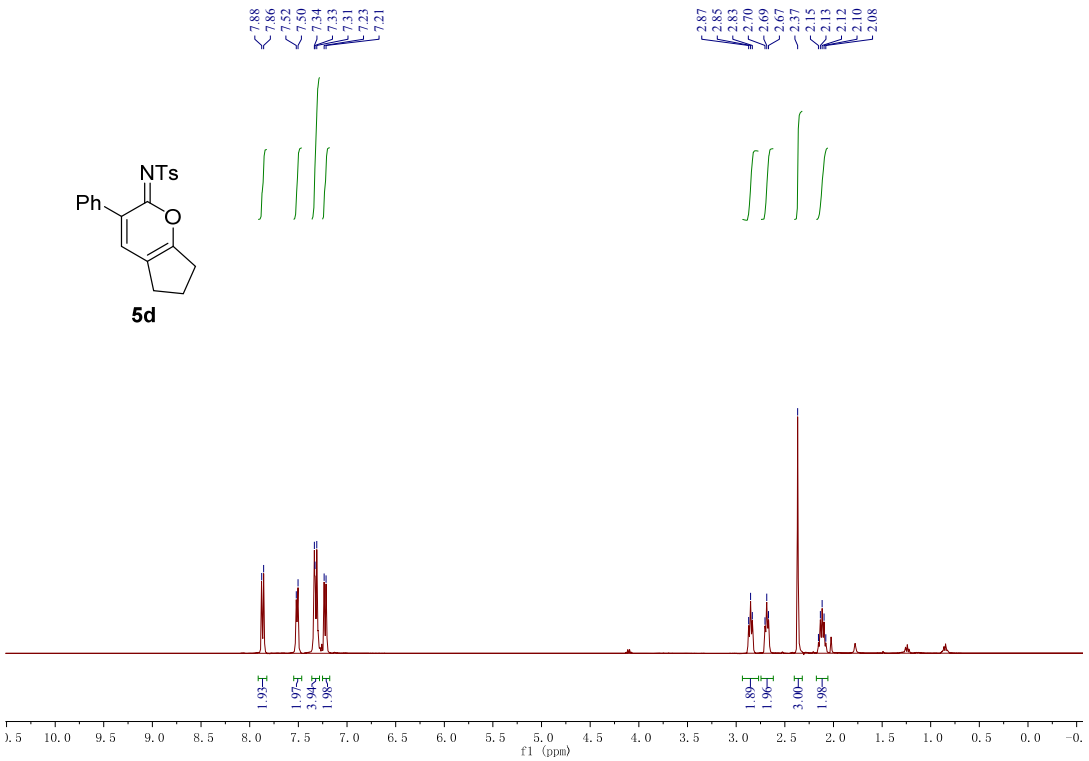

160.04
158.87
142.95
142.61
139.32
135.16
133.93
129.42
129.15
128.98
128.62
128.24
127.87
127.03
126.74
125.16
120.64
120.38
77.32
77.00
76.68
33.27
21.49
NTs
Ph
O
5a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

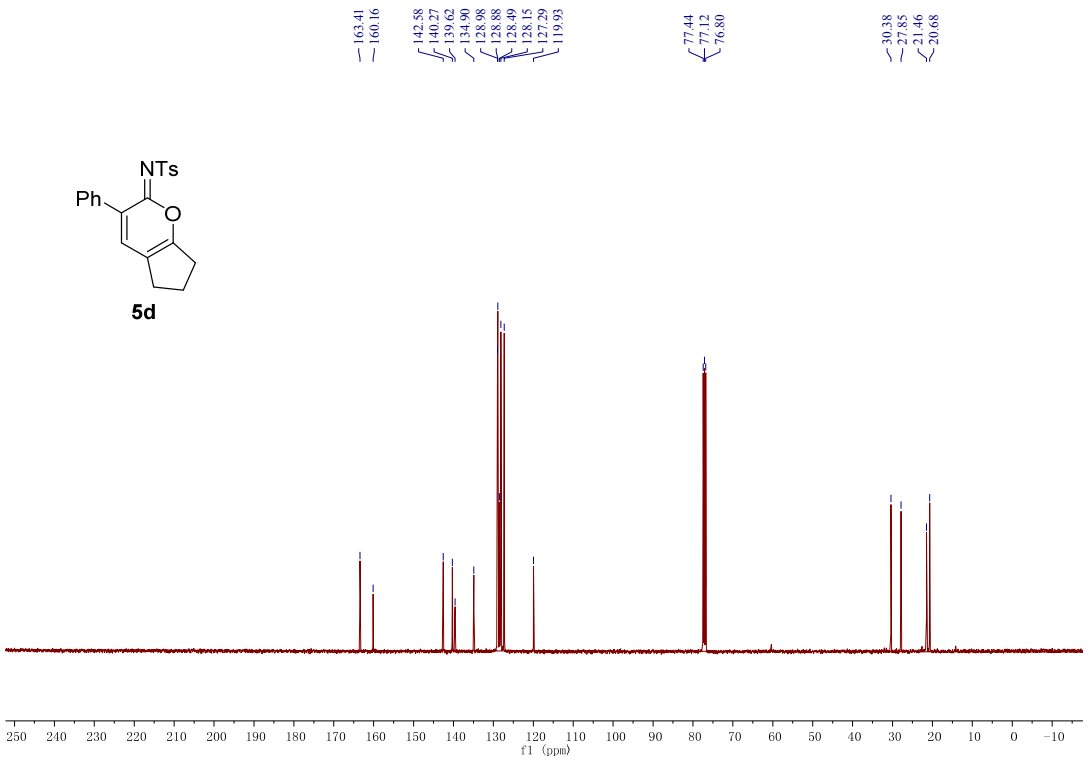

8.15
8.14
7.93
7.91
7.63
7.62
7.61
7.60
7.43
7.41
7.38
7.37
7.36
7.34
7.25
7.23
7.21
2.99
2.97
2.95
2.80
2.78
2.76
2.38
NTs
Ph
O
5b
0.94
1.93
2.00
6.06
3.58
1.92
1.93
3.00
f1 (ppm)

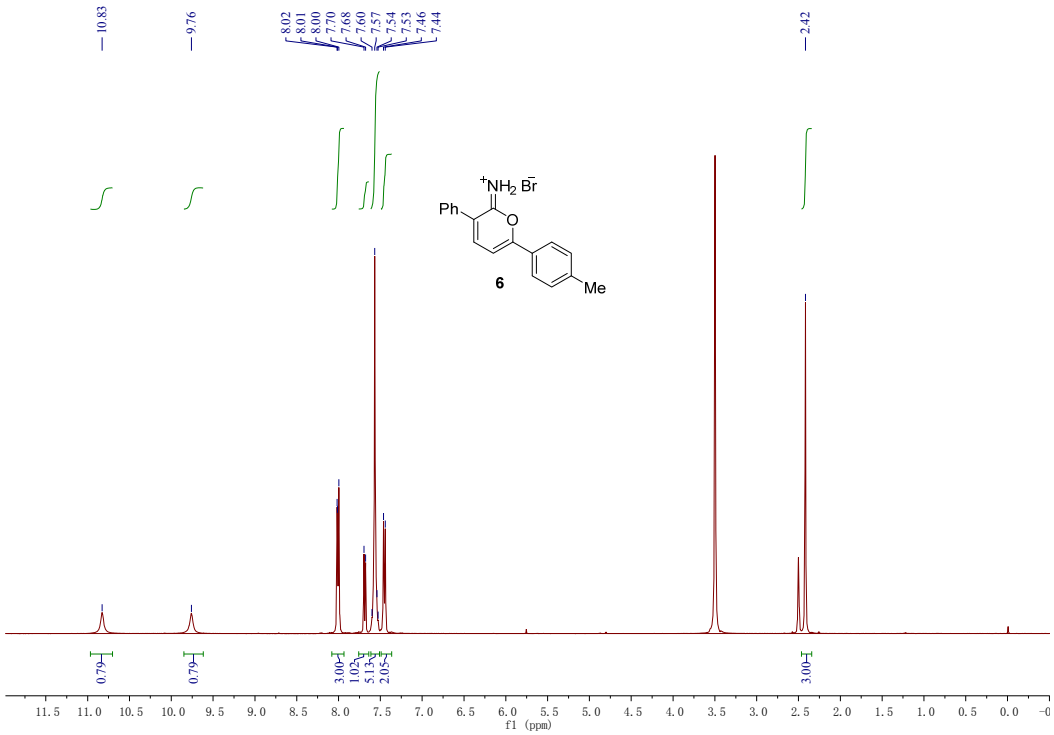

158.69
154.71
142.57
142.46
137.00
130.93
129.16
128.82
128.70
128.20
127.84
126.93
126.48
124.34
77.34
77.02
76.71
27.22
24.59
21.48
NTs
Ph
O
5b
f1 (ppm)

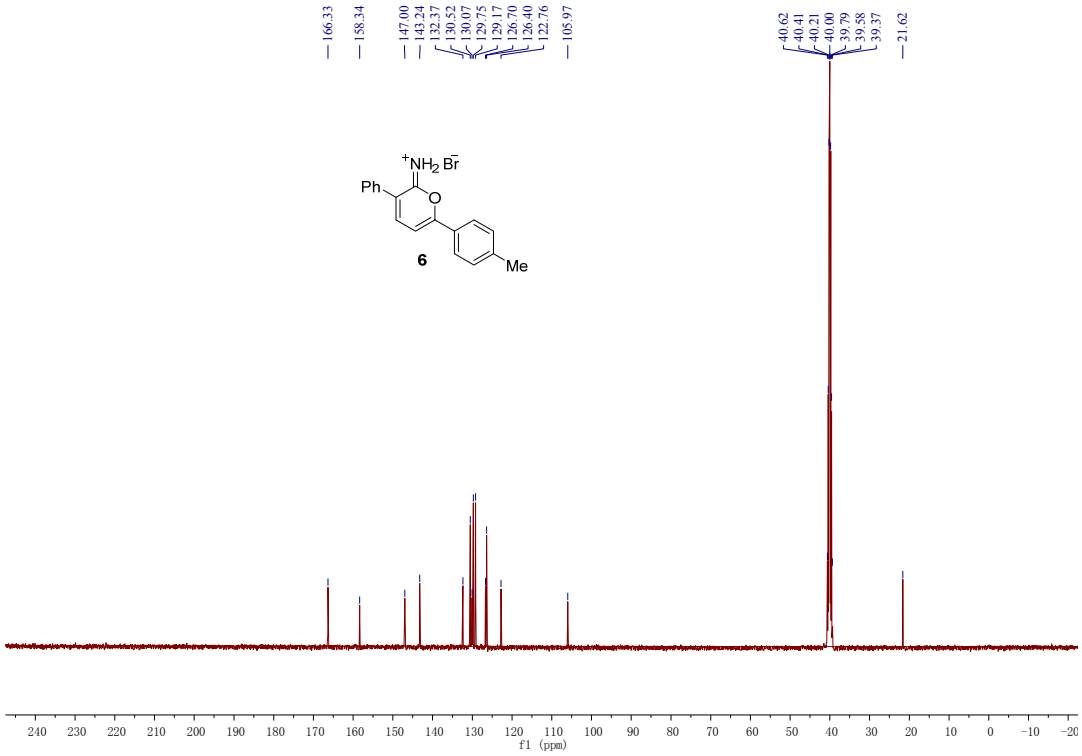

8.15
8.15
8.13
7.92
7.89
7.57
7.56
7.55
7.55
7.54
7.43
7.41
7.39
7.39
7.38
7.37
7.35
7.35
7.33
7.33
7.32
7.31
7.26
7.24
7.22
7.21
2.98
2.96
2.94
2.80
2.78
2.78
2.76
2.39
1.57
1.25
Cl
NTs
O
5c
0.96
1.97
2.03
3.15
1.95
1.93
2.00
1.98
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

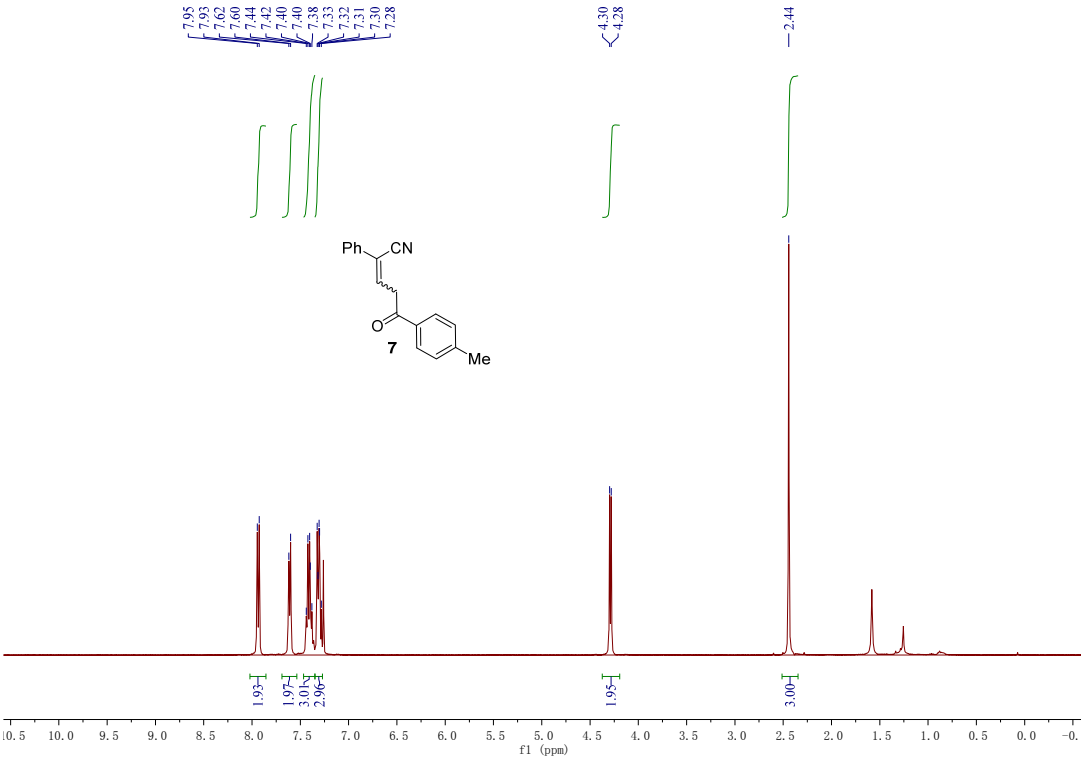

158.34
155.03
142.61
142.45
137.07
132.86
131.10
130.09
129.22
128.42
127.89
126.82
126.47
124.43
114.85
77.31
76.99
76.68
27.19
24.58
21.47
142.61
142.45
139.93
137.07
134.72
132.86
131.10
130.09
129.22
128.42
127.89
126.82
126.47
124.43
142 138 134 130 126
f1 (ppm)
Cl
NTs
O
5c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

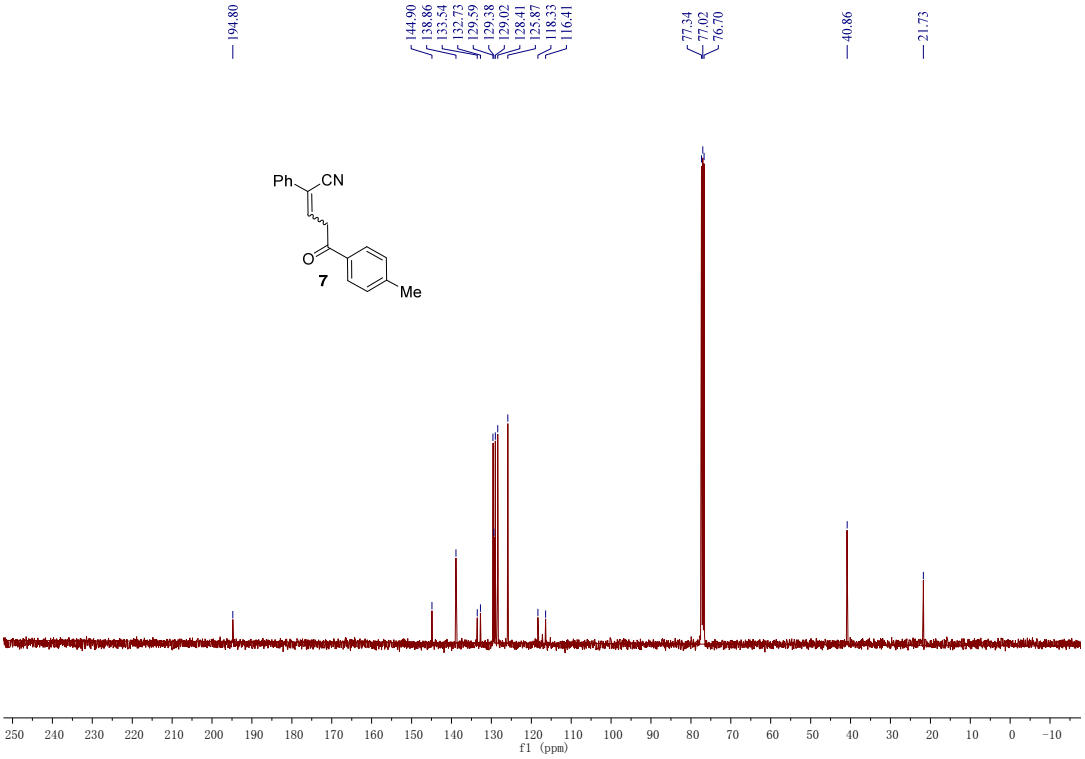

7.88
7.86
7.52
7.50
7.34
7.33
7.31
7.23
7.21
2.87
2.85
2.83
2.70
2.69
2.67
2.37
2.15
2.13
2.12
2.10
2.08
NTs
Ph
O
5d
1.93
1.97
3.94
1.98
1.89
1.96
3.00
1.98
f1 (ppm)

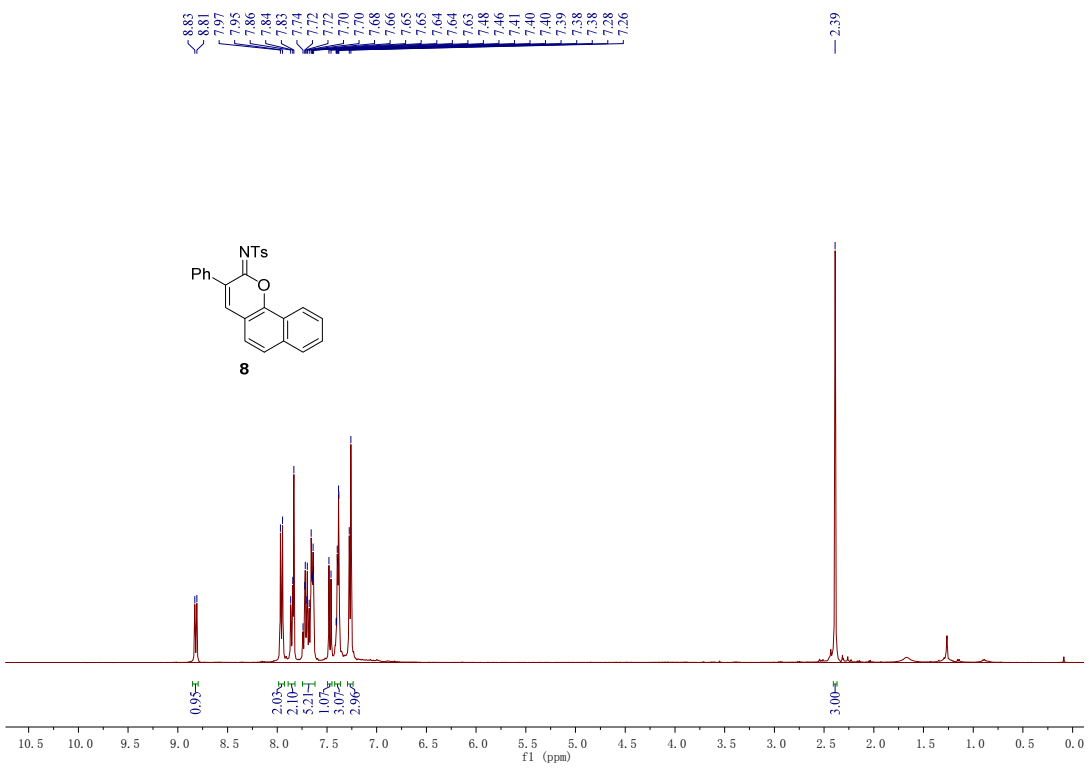

163.41
160.16
142.58
140.27
139.62
134.90
128.98
128.88
128.49
128.15
127.29
119.93
77.44
77.12
76.80
30.38
27.85
21.46
20.68
NTs
Ph
O
5d
f1 (ppm)

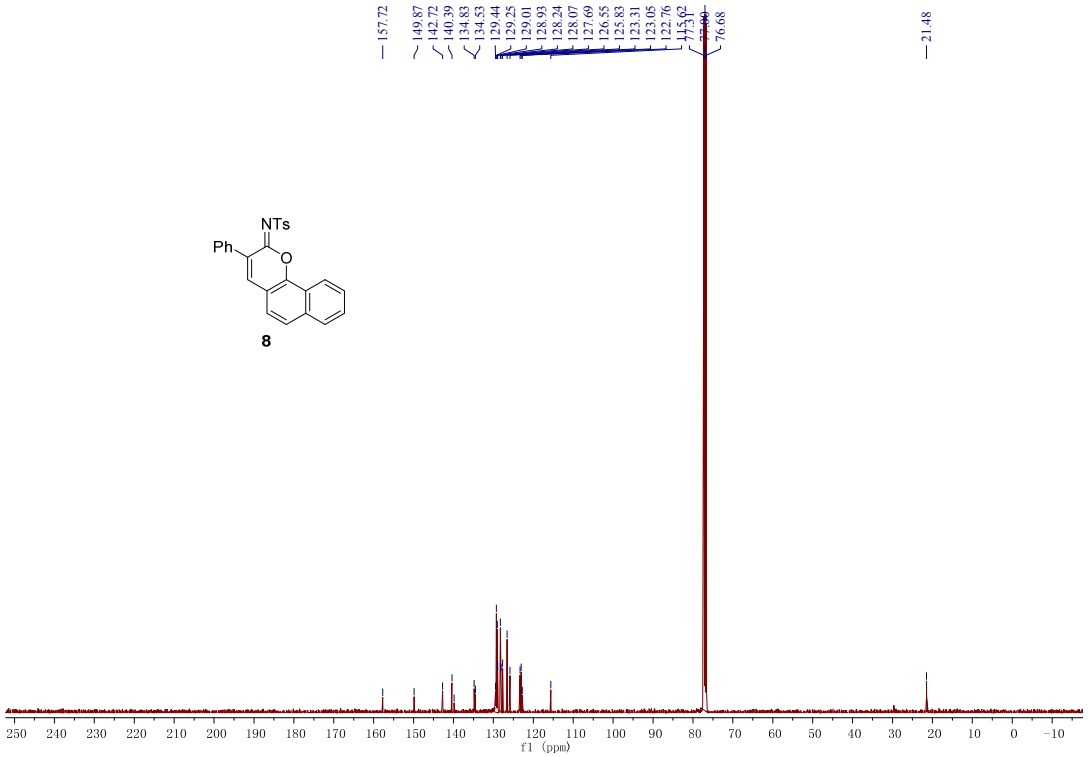

10.83
9.76
8.02
8.01
8.00
7.70
7.68
7.60
7.57
7.54
7.53
7.46
7.44
2.42
0.79
0.79
3.00
1.02
5.13
2.05
3.00
f1 (ppm)
Ph
NH2 Br
O
Me
6

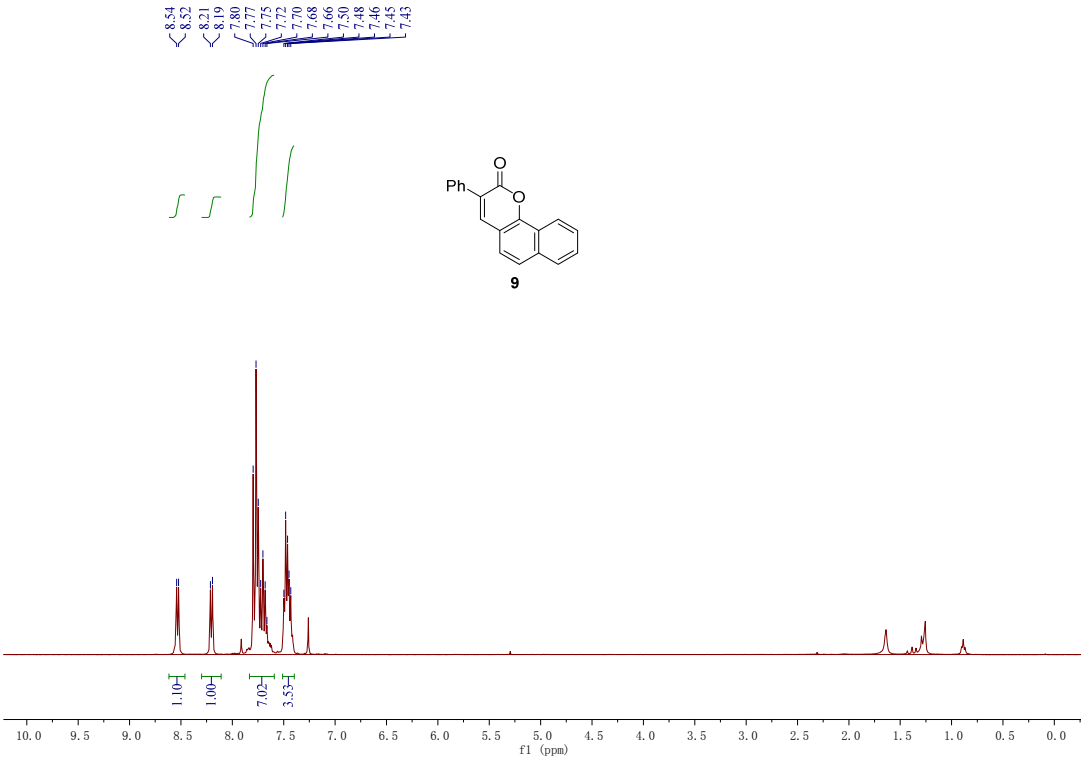

166.33
158.34
147.00
143.24
132.37
130.52
130.07
129.75
129.17
126.70
126.40
122.76
105.97
40.62
40.41
40.21
40.00
39.79
39.58
39.37
21.62
f1 (ppm)
Ph
NH2 Br
O
Me
6


7.95
7.93
7.62
7.60
7.44
7.42
7.40
7.40
7.38
7.33
7.32
7.31
7.30
7.28
4.30
4.28
2.44
Ph
CN
O
7
Me
1.93
1.97
3.01
2.96
1.95
3.00
f1 (ppm)

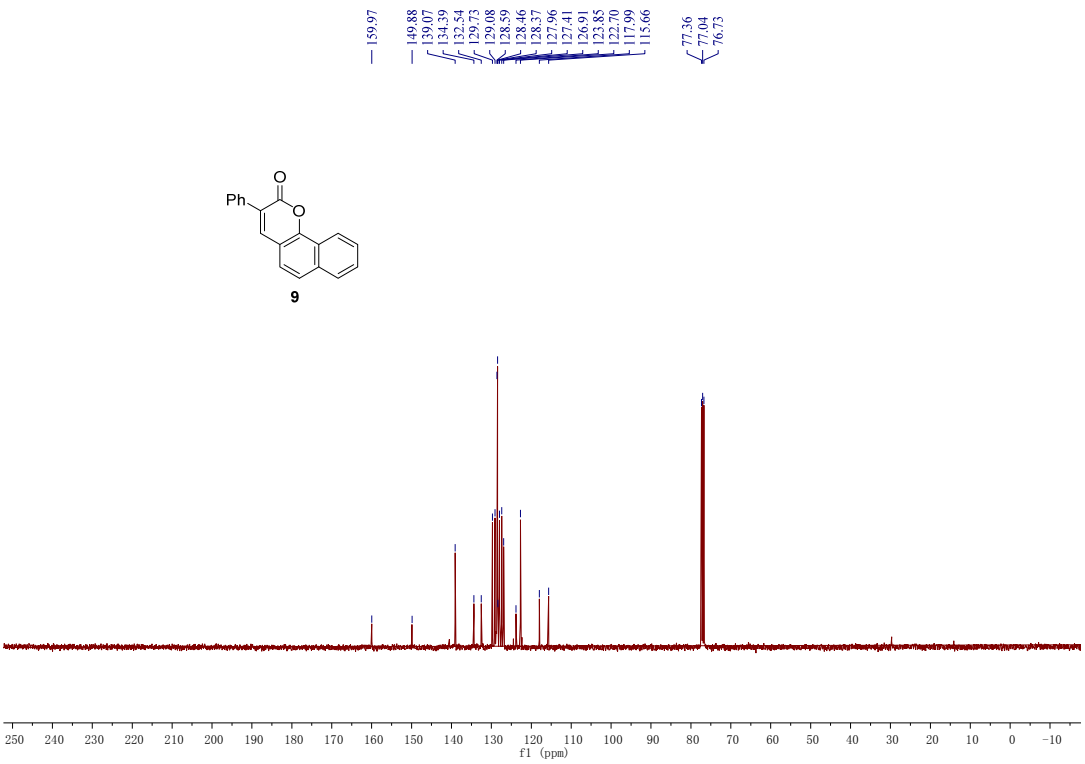

194.80
144.90
138.86
133.54
132.73
129.59
129.38
129.02
128.41
125.87
118.33
116.41
77.34
77.02
76.70
40.86
21.73
Ph
CN
O
7
Me
f1 (ppm)


8.83
8.81
7.97
7.95
7.86
7.84
7.83
7.74
7.72
7.72
7.70
7.70
7.68
7.66
7.65
7.65
7.64
7.64
7.63
7.48
7.46
7.41
7.40
7.40
7.39
7.38
7.28
7.26
2.39
NTs
Ph
O
8
0.95
2.03
2.10
5.21
1.07
3.07
2.96
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


157.72
149.87
142.72
140.39
134.83
134.53
129.44
129.25
129.01
128.93
128.24
128.07
127.69
126.55
125.83
123.31
123.05
122.76
115.62
77.31
76.68
21.48
NTs
Ph
O
8
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.54
8.52
8.21
8.19
7.80
7.77
7.75
7.72
7.70
7.68
7.66
7.50
7.48
7.46
7.45
7.43
Ph
O
O
1.10
1.00
7.02
3.53
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

9


159.97
149.88
139.07
134.39
132.54
129.73
129.08
128.59
128.46
128.37
127.96
127.41
126.91
123.85
122.70
117.99
115.66
77.36
77.04
76.73
Ph
O
O
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

9