Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

For

Construction of Highly Functionalized Naphthalenes Using *in situ* Ene−Allene Strategy

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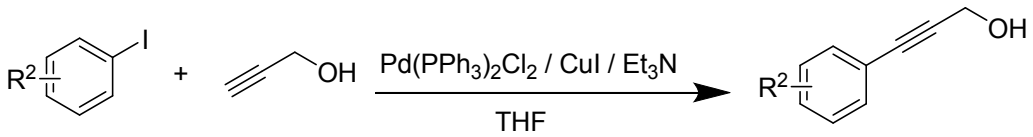
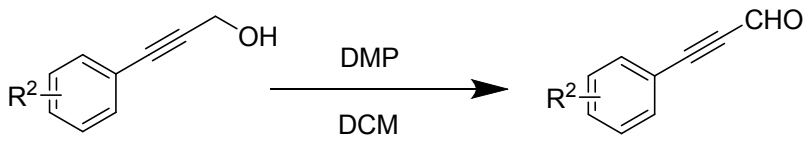
1. General information

All reactions were carried out in oven-dried glassware sealed with rubber septa under nitrogen condition. All solvents were distilled under nitrogen atmosphere prior to use. THF was dried over sodium hydride, and toluene was dried over sodium. Purification of products was conducted by flash chromatography on silica gel (200-300 mesh). NMR spectra were measured on a Varian 400 (^{1}H at 400 MHz, ^{13}C at 100 MHz) magnetic resonance spectrometer. Chemical shifts are reported in ppm using tetramethylsilane as internal standard (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet). Infrared spectra were recorded on a Nicolet Avatar 330 Fourier transform spectrometer (FT-IR) and are reported in wave numbers (cm^{-1}). MS data were obtained on an Agilent 5975C inert 350 EI mass spectrometer (GC-MS). HRMS data were obtained on a VG ZAB-HS mass spectrometer, Brucker Apex IV FTMS spectrometer. Compounds described in the literature were characterized by comparison of their ^{1}H, and/or ^{13}C NMR spectra to the previously reported data.

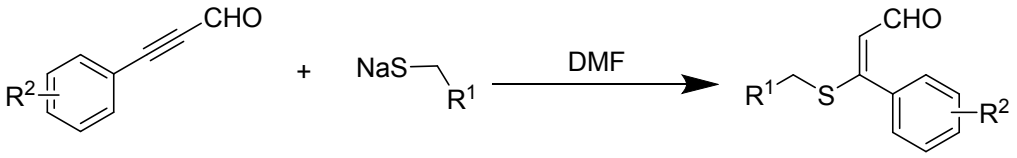
2. General procedure for substrates



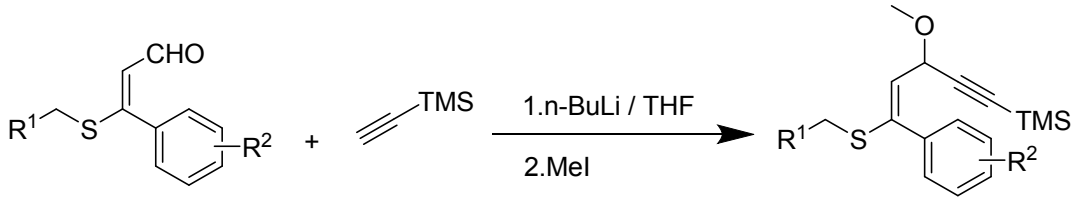
Under nitrogen condition, $Pd(PPh_3)_2Cl_2$ (1mmol), CuI (2mmol) and iodine substrates (50mmol) were successively added to a 250mL vial equipped with a stir bar. THF (100mL) was added using a syringe, then propargyl alcohol (60mmol) was added to the mixture. Et_3N (150mmol) was added at last. The reaction was stirred at room temperature for 8h. Solvent was removed in vacuoto leaving a crude mixture, which is purified by silica gel column chromatography to afford pure product (PE:EA=5:1).



To a solution of the substrates (25mmol) in DCM (50mL) were added DMP (25mmol) at 0 °C. The resulting solution was stirred for 1 h at room temperature. The reaction was quenched with saturated sodium hyposulfite solution (50mL), extracted with DCM (3×25mL) and dried over anhydrous Na_2SO_4. After removal of solvent, the residue left was the crude product which can be directly used in the next step.

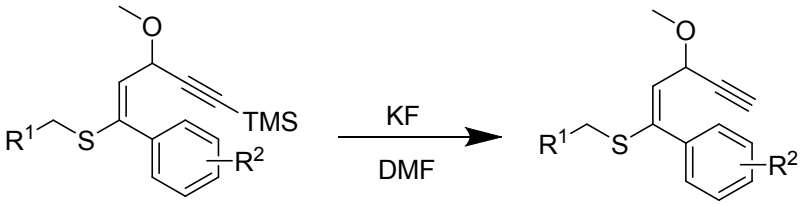


Sodium sulfur compounds (10mmol, 15% in water) were added to a solution of substrates (10mmol) in DMF (20 mL) at 0 °C. The resulting mixture was stirred for 1min. The reaction was then quenched by adding 50mL water and extracted with ether (3×15mL). The combined organics were dried over anhydrous Na_2SO_4. After removal of solvent the residue, left was purified by flash column chromatography with silica gel using mixture of petroleum ether and ethyl acetate (10:1) to give the title substrates.

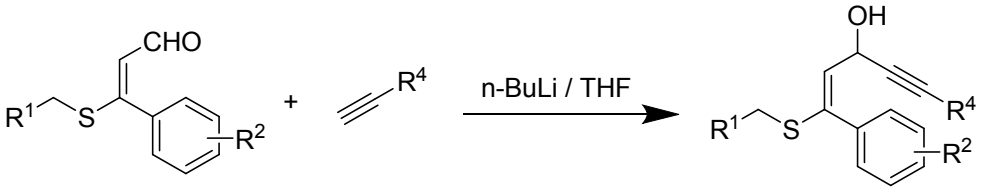


To a solution of the ethynyltrimethylsilane (5mmol) in dry THF (5mL) was added n-BuLi (2mL, 2.5mmol/mL) at -78 °C under argon. The resulting solution was stirred for 0.5 h at room temperature. Then the substrate (5mmol) in dry THF (5mL) was added slowly at -78 °C and the solution was stirred for 2 h at room temperature.

Methyl iodide (12mmol) was added at last. The reaction was stirred at 50 °C for 8h. The reaction was quenched with water (20mL), extracted with ethyl acetate (3×15mL) and dried over anhydrous Na_2SO_4. After removal of solvent, the residue left was the crude product which can be directly used in the next step.

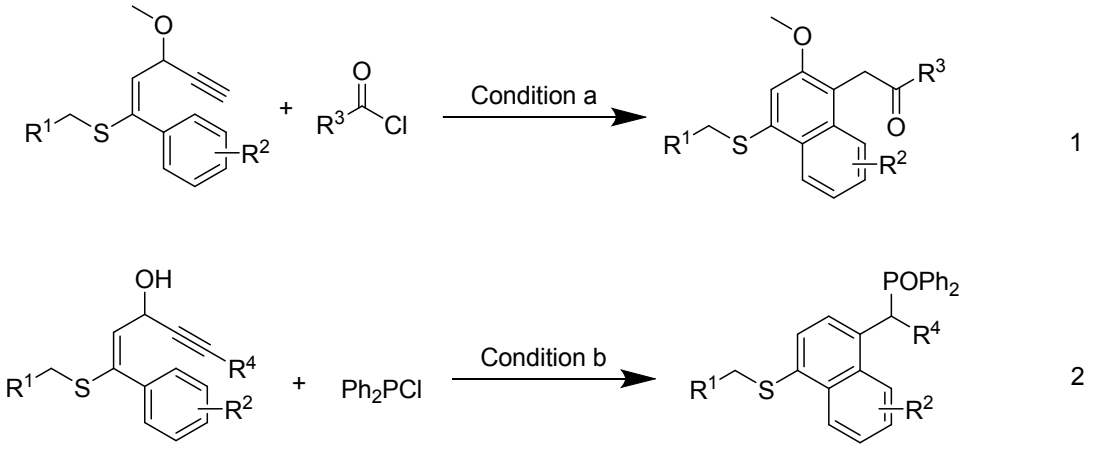


Potassium fluoride (5mmol) was added to a solution of substrates (2mmol) in DMF (4mL) at room temperature. The resulting mixture was stirred for 2 h. The reaction was then quenched by adding 20mL water and extracted with ether (3×15mL). The combined organics were dried over anhydrous Na_2SO_4. After removal of solvent, the residue left was purified by flash column chromatography with silica gel using mixture of petroleum ether and ethyl acetate (20:1) to give the title substrates.



To a solution of the alkynyl compounds (2.5mmol) in dry THF (5mL) was added n-BuLi (1mL, 2.5mmol/mL) at -78 °C under argon. The resulting solution was stirred for 0.5 h at room temperature. Then substrates (5mmol) in dry THF (5mL) were added slowly at -78 °C and the solution was stirred for 2 h at room temperature. The reaction was quenched with water (20mL), extracted with ethyl acetate (3×15mL) and dried over anhydrous Na_2SO_4. Solvent was removed in vacuoto leave a crude mixture, which is purified by silica gel column chromatography to afford pure product (PE:EA=5:1).

3. General procedure for the reaction



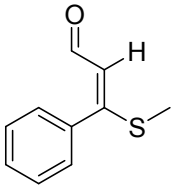
R^1=H or Aryl
R2,R3,R4=Alkyl or Aryl

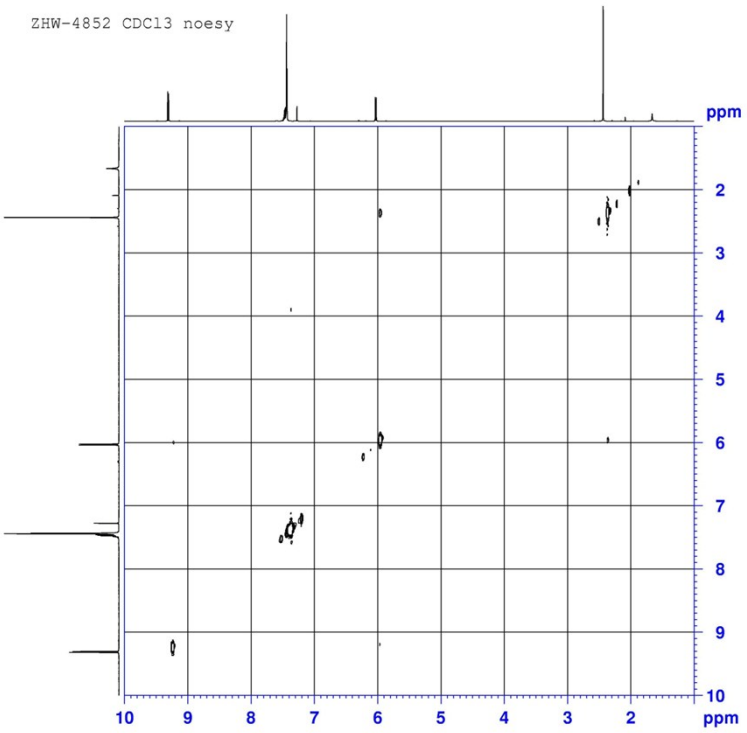
Condition a: $Pd(PPh_3)_2Cl_2$ / CuI / Et_3N / dry THF / 60 °C
Condition b: $Ga(OTf)_3$ / Et_3N / dry THF / 0 °C~60 °C

For the reaction 1: Under nitrogen condition, $Pd(PPh_3)_2Cl_2$ (18mg, 0.025mmol, 0.05eq), CuI (5mg, 0.025mmol, 0.05eq) and substrates (0.5mmol, 1eq) were successively added to a 25mL vial equipped with a stir bar. Dry THF (5.0mL) was added using a syringe, then acyl chloride (0.6mmol, 1.2eq) was added to the mixture. Et_3N (150mg, 1.5mmol, 3eq) was added at last. The reaction was stirred at 60 °C for 12h. Solvent was removed in vacuoto leave a crude mixture, which is purified by silica gel column chromatography to afford pure product (PE:EA=20:1).

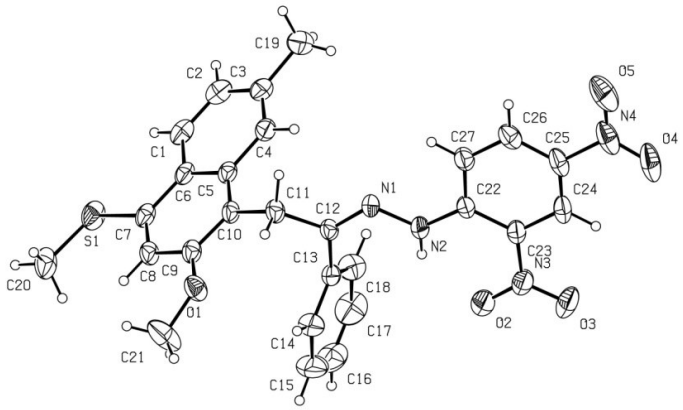
For the reaction 2: Under nitrogen condition, substrates (0.5mmol, 1eq), Et_3N (150mg, 1.5mmol, 3eq) and dry THF (5.0ml) were successively added to a 25mL vial equipped with a stir bar. Chlorodiphenyl phosphine (120mg, 0.6mmol, 1.1eq) was added using a syringe at 0 °C. The reaction was stirred at room temperature for 2h. Then $Ga(OTf)_3$ (13mg, 0.025mmol, 0.05eq) was added to the mixture, and the reaction was stirred at 60 °C for 8h. Solvent was removed in vacuoto leave a crude mixture, which is purified by silica gel column chromatography to afford pure product (DCM:EA=5:1).

The noesy of (E)-3-(methylthio)-3-phenylacrylaldehyde.





ORTEP Representation (2,4-dinitrophenyl)hydrazone of 3d



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Bond precision:  C-C = 0.0036 A          Wavelength=0.71073

Cell:           a=11.3897(16)    b=8.3907(12)         c=26.863(3)
                alpha=90         beta=100.495(12)     gamma=90
Temperature:    293 K

                Calculated                Reported
Volume          2524.3(6)                 2524.3(6)
Space group     P 21/n                    P 1 21/n 1
Hall group      -P 2yn                    -P 2yn
Moiety formula  C27 H24 N4 O5 S           C27 H24 N4 O5 S
Sum formula     C27 H24 N4 O5 S           C27 H24 N4 O5 S
Mr              516.56                    516.56
Dx,g cm-3       1.359                     1.359
Z               4                         4
Mu (mm-1)       0.174                     0.174
F000            1080.0                    1080.0
F000'           1080.97
h,k,lmax        13,10,32                  13,10,32
Nref            4618                      4578
Tmin,Tmax       0.967,0.979               0.955,1.000
Tmin'           0.966

Correction method= # Reported T Limits: Tmin=0.955 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.991          Theta(max)= 25.350

R(reflections)= 0.0467( 2882)     wR2(reflections)= 0.1208( 4578)

S = 1.012                 Npar= 337
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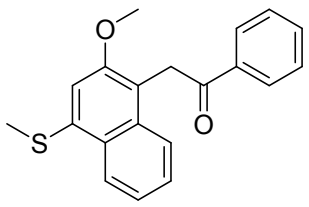
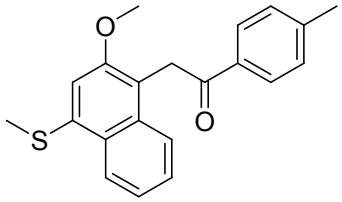
4. Characterization data



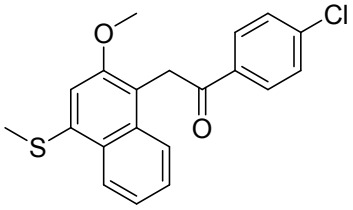
3a

2-(2-methoxy-4-(methylthio)naphthalen-1-yl)-1-phenylethanone(3a): Pale amorphous solid, 140mg, 87% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.23 (m, 1H), 8.11 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.60 – 7.54 (m, 1H), 7.50 – 7.43 (m, 3H), 7.38 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 7.26 (s, 1H), 4.74 (s, 2H), 3.92 (s, 3H), 2.58 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.8, 154.4, 137.0, 136.7, 133.8, 133.0, 128.5, 128.3, 127.8, 127.1, 125.0, 123.7, 123.7, 115.0, 112.4, 56.7, 35.7, 16.8; IR (neat) 3415, 1620, 1384cm^{-1}; HRMS (EI-TOF) calcd for $C_{20}H_{18}O_2S$ 322.1028, found 322.1031.



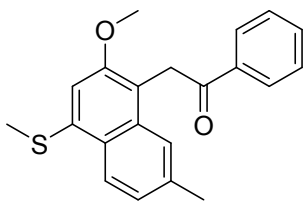
3b

2-(2-methoxy-4-(methylthio)naphthalen-1-yl)-1-p-tolylethanone(3b): Pale amorphous solid, 138mg, 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 – 8.21 (m, 1H), 8.01 (d, *J* = 8.2 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.37 (ddd, *J* = 8.1, 6.8, 1.3 Hz, 2H), 7.28 (s, 1H), 7.24 (s, 1H), 4.71 (s, 2H), 3.92 (s, 3H), 2.57 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.4, 154.2, 143.8, 129.2, 128.4, 127.0, 124.9, 123.8, 123.7, 56.8, 35.6, 21.7, 16.8; IR (neat) 3416, 1621, 1189cm^{-1}; HRMS (EI-TOF) calcd for $C_{21}H_{20}O_2S$ 336.1184, found 336.1185.



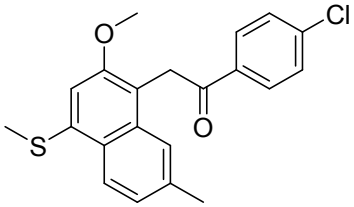
3c

1-(4-chlorophenyl)-2-(2-methoxy-4-(methylthio)naphthalen-1-yl)ethanone(3c): Pale amorphous solid, 153mg, 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.4 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.41 (dt, *J* = 15.2, 7.9 Hz, 4H), 7.24 (d, *J* = 0.9 Hz, 1H), 4.68 (s, 2H), 3.92 (s, 3H), 2.57 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 154.3, 139.4, 137.0, 135.2, 133.6, 129.8, 128.8, 127.2, 124.9, 123.8, 123.6, 114.5, 112.1, 56.7, 35.8, 16.7; IR (neat) 3415, 1621, 1191cm^{-1}; HRMS (EI-TOF) calcd for $C_{20}H_{17}ClO_2S$ 356.0638, found 356.0634.



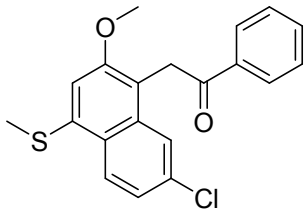
3d

1-(4-chlorophenyl)-2-(2-methoxy-4-(methylthio)naphthalen-1-yl)ethanone(3d): Pale amorphous solid, 136mg, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.09 (m, 3H), 7.60 – 7.55 (m, 1H), 7.48 (dd, *J* = 10.3, 4.8 Hz, 3H), 7.22 – 7.17 (m, 2H), 4.71 (s, 2H), 3.90 (s, 3H), 2.56 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 154.5, 137.1, 136.8, 136.4, 134.0, 133.0, 128.5, 128.3, 126.0, 124.8, 122.8, 114.3, 111.5, 56.6, 35.7, 22.0, 16.8; IR (neat) 3415, 1620, 1191cm^{-1}; HRMS (EI-TOF) calcd for $C_{21}H_{20}O_2S$ 336.1184, found 336.1187.



3e

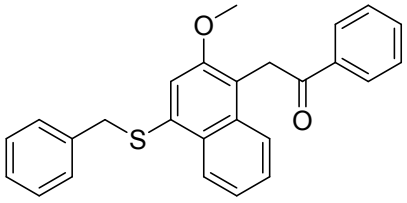
1-(4-chlorophenyl)-2-(2-methoxy-7-methyl-4-(methylthio)naphthalen-1-yl)ethanone(3e): Pale amorphous solid, 148mg, 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.6 Hz, 1H), 8.06 – 8.01 (m, 2H), 7.48 (s, 1H), 7.45 – 7.40 (m, 2H), 7.21 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.16 (s, 1H), 4.65 (s, 2H), 3.90 (s, 3H), 2.56 (s, 3H), 2.44 (s, 3H); ^{13}C NMR(100MHz, $CDCl_3$) δ 197.0, 154.4, 139.3, 136.9, 136.7, 135.3, 133.9, 129.8, 128.8, 126.0, 124.8, 122.7, 113.9, 111.2, 56.6, 35.8, 22.0, 16.7; IR (neat) 3416, 1621, 1204cm^{-1}; HRMS (EI-TOF) calcd for $C_{21}H_{19}ClO_2S$ 370.0794, found 370.0797.



3f

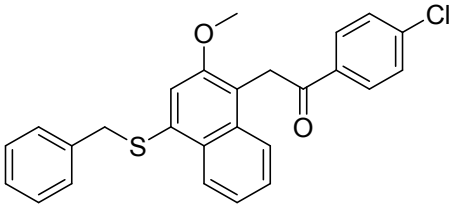
2-(7-chloro-2-methoxy-4-(methylthio)naphthalen-1-yl)-1-phenylethanone(3f): Pale amorphous solid, 153mg, 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 9.0 Hz, 1H), 8.15 – 8.09 (m, 2H), 7.67 (d, *J* = 1.9 Hz, 1H), 7.59 (d, *J* = 7.4 Hz, 1H),

7.50 (t, *J* = 7.6 Hz, 2H), 7.30 (dd, *J* = 9.0, 2.0 Hz, 1H), 7.22 (s, 1H), 4.68 (s, 2H), 3.91 (s, 3H), 2.57 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.2, 155.3, 137.1, 136.8, 134.8, 133.3, 133.2, 130.3, 128.6, 128.3, 126.7, 126.0, 124.4, 122.6, 114.2, 112.2, 56.5, 35.5, 16.8; IR (neat) 3416, 1620, 1191cm^{-1}; HRMS (EI-TOF) calcd for $C_{20}H_{17}ClO_2S$ 356.0638, found 356.0638.



3g

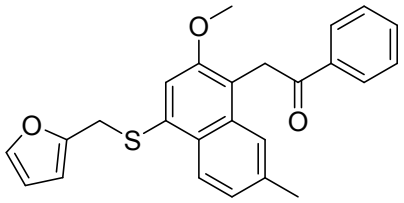
2-(4-(benzylthio)-2-methoxynaphthalen-1-yl)-1-phenylethanone(3g): Pale amorphous solid, 148mg, 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (dd, *J* = 8.3, 0.9 Hz, 1H), 8.13 – 8.07 (m, 2H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.46 (ddd, *J* = 8.3, 7.2, 4.0 Hz, 3H), 7.40 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 7.23 (m, 6H), 4.73 (s, 2H), 4.12 (s, 2H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 153.9, 137.5, 137.0, 134.0, 133.7, 133.0, 129.0, 128.9, 128.5, 128.4, 128.3, 127.2, 127.0, 125.8, 124.0, 123.8, 117.7, 116.7, 56.5, 39.7, 35.9; IR (neat) 3415, 1620, 1383cm^{-1}; HRMS (EI-TOF) calcd for $C_{26}H_{22}O_2S$ 398.1341, found 398.1341.



3h

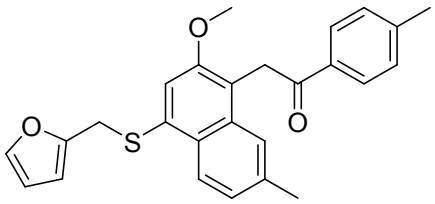
2-(4-(benzylthio)-2-methoxynaphthalen-1-yl)-1-(4-chlorophenyl)ethanone(3h): Pale amorphous solid, 155mg, 72% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (dd, *J* = 8.3, 0.8 Hz, 1H), 8.05 – 7.97 (m, 2H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.43 (m, 5H), 7.23 – 7.20 (m, 3H), 7.19 (s, 2H), 4.67 (s, 2H), 4.12 (s, 2H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.8, 153.8, 139.4, 137.5, 135.2, 134.0, 133.8, 129.7, 129.0, 128.9,

128.8, 128.5, 127.2, 127.1, 125.8, 124.0, 123.6, 117.5, 116.2, 56.4, 39.6, 36.0; IR (neat) 3416, 1621, 1191cm^{-1}; HRMS (EI-TOF) calcd for $C_{26}H_{21}ClO_2S$ 432.0951, found 432.0955.



3i

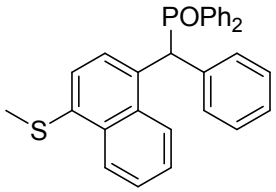
2-(4-(furan-2-ylmethylthio)-2-methoxy-7-methylnaphthalen-1-yl)-1-phenylethanone(3i): Pale amorphous solid, 152mg, 76% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.6 Hz, 1H), 8.13 – 8.06 (m, 2H), 7.57 (d, *J* = 7.4 Hz, 1H), 7.50 – 7.46 (m, 3H), 7.34 (d, *J* = 1.0 Hz, 1H), 7.23 (d, *J* = 9.4 Hz, 2H), 6.24 – 6.17 (m, 1H), 5.93 (d, *J* = 2.9 Hz, 1H), 4.72 (s, 2H), 4.08 (s, 2H), 3.80 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 154.0, 150.9, 142.1, 137.0, 136.8, 134.2, 133.0, 132.6, 128.5, 128.3, 127.7, 126.4, 125.8, 122.7, 118.0, 116.7, 110.5, 108.2, 56.5, 35.9, 32.4, 22.0; IR (neat) 3416, 1623, 1385cm^{-1}; HRMS (EI-TOF) calcd for $C_{25}H_{22}O_3S$ 402.1290, found 402.1285.



3j

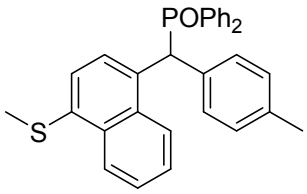
2-(4-(furan-2-ylmethylthio)-2-methoxy-7-methylnaphthalen-1-yl)-1-*p*-tolylethanone(3j): Pale amorphous solid, 146mg, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 8.6 Hz, 1H), 8.01 (d, *J* = 8.2 Hz, 2H), 7.50 (s, 1H), 7.36 – 7.32 (m, 1H), 7.29 (s, 1H), 7.27 – 7.20 (m, 3H), 6.22 (dd, *J* = 3.1, 1.9 Hz, 1H), 5.93 (d, *J* = 2.8 Hz, 1H), 4.70 (s, 2H), 4.08 (s, 2H), 3.79 (s, 3H), 2.43 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 154.0, 150.9, 143.8, 142.1, 136.7, 134.5, 134.2,

132.5, 129.2, 128.4, 127.7, 126.4, 125.8, 122.8, 118.1, 117.0, 110.5, 108.2, 105.0, 56.5, 35.7, 32.4, 22.0, 21.7; IR (neat) 3415, 1619, 1384cm^{-1}; HRMS (EI-TOF) calcd for $C_{24}H_{24}O_3S$ 416.1446, found 416.1443.



5a

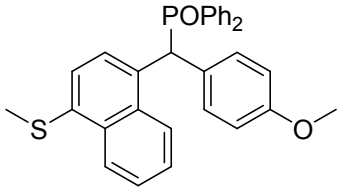
((4-(methylthio)naphthalen-1-yl)(phenyl)methyl)diphenylphosphine oxide(5a): Pale amorphous solid, 187mg, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 7.8 Hz, 1H), 8.26 – 8.19 (m, 1H), 8.02 (dd, *J* = 6.6, 3.1 Hz, 1H), 7.68 – 7.60 (m, 2H), 7.44 (m, 5H), 7.33 – 7.21 (m, 8H), 7.14 – 7.04 (m, 3H), 5.47 (d, *J* = 10.6 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.7, 135.4 (d, *J* = 6.0 Hz), 132.7, 132.4, 131.8 (d, *J* = 5.0 Hz), 131.7, 131.4 (d, *J* = 2.0 Hz), 131.3 (d, *J* = 8.0 Hz), 131.2, 131.1 (d, *J* = 8.0 Hz), 130.8, 128.4, 128.2 (d, *J* = 6.0 Hz), 128.1, 126.9 (d, *J* = 2.0 Hz), 126.7, 125.5, 125.1, 123.1, 122.5, 47.7 (d, *J* = 67.0 Hz), 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.82; IR (neat) 3416, 1621, 1179cm^{-1}; HRMS (EI-TOF) calcd for $C_{30}H_{25}OPS$ 464.1364, found 464.1365.



5b

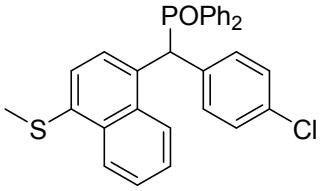
((4-(methylthio)naphthalen-1-yl)(p-tolyl)methyl)diphenylphosphine oxide(5b): Pale amorphous solid, 186mg, 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 7.7 Hz, 1H), 8.27 – 8.17 (m, 1H), 8.08 – 7.98 (m, 1H), 7.69 – 7.60 (m, 2H), 7.52 (dd, *J* = 10.5, 7.9 Hz, 2H), 7.46 – 7.38 (m, 3H), 7.25 (m, 8H), 6.90 (d, *J* = 7.9 Hz, 2H), 5.47 (d, *J* = 10.5 Hz, 1H), 2.47 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

136.5 (d, J = 2.0 Hz), 135.5, 133.6, 133.0, 132.6, 132.4 (d, J = 6.0 Hz), 132.0, 131.9, 131.7 (d, J = 9.0 Hz), 131.3 (d, J = 9.0 Hz), 131.1 (d, J = 2.0 Hz), 129.9 (d, J = 6.0 Hz), 128.8 (d, J = 2.0 Hz), 128.4, 128.3 (d, J = 7.0 Hz), 128.1 (d, J = 3.0 Hz), 128.0, 126.7, 125.5, 125.1, 123.2, 122.6, 47.2 (d, J = 67.0 Hz), 21.0, 15.7; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.7; IR (neat) 3416, 1621, 1180cm^{-1}; HRMS (EI-TOF) calcd for $C_{31}H_{27}OPS$ 478.1520, found 478.1523.



5c

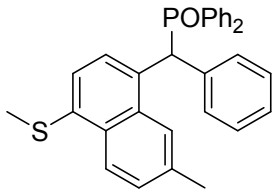
((4-methoxyphenyl)(4-(methylthio)naphthalen-1-yl)methyl)diphenylphosphine oxide(5c): Pale amorphous solid, 185mg, 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, J = 7.8 Hz, 1H), 8.26 – 8.17 (m, 1H), 8.00 (dd, J = 6.5, 3.0 Hz, 1H), 7.66 – 7.60 (m, 2H), 7.51 – 7.39 (m, 5H), 7.31 (m, 3H), 7.25 – 7.20 (m, 3H), 7.18 – 7.12 (m, 2H), 6.63 (d, J = 8.6 Hz, 2H), 5.42 (d, J = 10.4 Hz, 1H), 3.66 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 135.5, 133.6, 132.9, 132.5, 131.8 (d, J = 4.0 Hz), 131.6 (d, J = 9.0 Hz), 131.4 (d, J = 2.0 Hz), 131.3, 131.2, 131.1, 128.4 (d, J = 11.0 Hz), 128.2 (d, J = 4.0 Hz), 128.1, 127.3 (d, J = 6.0 Hz), 126.7, 125.5, 125.1, 123.2, 122.5, 113.6 (d, J = 2.0 Hz), 105.0, 55.1, 46.8 (d, J = 67.0 Hz), 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.9; IR (neat) 3417, 1621, 1183cm^{-1}; HRMS (EI-TOF) calcd for $C_{31}H_{27}O_2PS$ 494.1469, found 494.1465.



5d

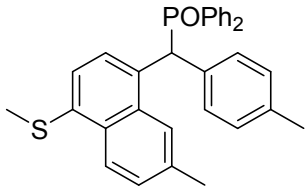
((4-chlorophenyl)(4-(methylthio)naphthalen-1-yl)methyl)diphenylphosphine

oxide(5d): Pale amorphous solid, 209mg, 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 7.7 Hz, 1H), 8.22 (dd, *J* = 6.5, 3.2 Hz, 1H), 8.00 – 7.88 (m, 1H), 7.68 – 7.58 (m, 2H), 7.47 (m, 5H), 7.37 – 7.28 (m, 3H), 7.24 – 7.15 (m, 5H), 7.06 (d, *J* = 8.4 Hz, 2H), 5.44 (d, *J* = 10.5 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.1, 134.1 (d, *J* = 5.0 Hz), 133.1, 132.9 (d, *J* = 3.0 Hz), 132.5, 132.1, 131.8, 131.6 (d, *J* = 2.0 Hz), 131.5 (d, *J* = 2.0 Hz), 131.3 (d, *J* = 5.0 Hz), 131.1 (d, *J* = 9.0 Hz), 131.0 (d, *J* = 9.0 Hz), 130.3 128.6, 128.5, 128.3 (d, *J* = 2.0 Hz), 128.2 (d, *J* = 7.0 Hz), 126.8, 125.7, 125.2, 122.9, 122.3, 47.1 (d, *J* = 66.0 Hz), 15.5; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.5; IR (neat) 3417, 1620, 1189cm^{-1}; HRMS (EI-TOF) calcd for $C_{30}H_{24}ClOPS$ 498.0974, found 498.0973.



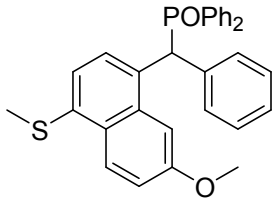
5e

((7-methyl-4-(methylthio)naphthalen-1-yl)(phenyl)methyl)diphenylphosphine oxide(5e): Pale amorphous solid, 186mg, 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 7.8 Hz, 1H), 8.10 (d, *J* = 8.5 Hz, 1H), 7.71 (s, 1H), 7.62 (dd, *J* = 10.7, 7.7 Hz, 2H), 7.50 – 7.38 (m, 3H), 7.34 – 7.25 (m, 5H), 7.24 – 7.15 (m, 4H), 7.10 (d, *J* = 5.4 Hz, 3H), 5.42 (d, *J* = 10.5 Hz, 1H), 2.46 (d, *J* = 14.5 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.3, 135.5 (d, *J* = 6.0 Hz), 135.4, 133.5, 132.8, 132.5, 131.9 (d, *J* = 8.0 Hz), 131.8, 131.4 (d, *J* = 3.0 Hz), 131.1 (d, *J* = 9.0 Hz), 130.1 (d, *J* = 5.0 Hz), 130.0, 128.4 (d, *J* = 8.0 Hz), 128.1, 127.6, 126.8, 124.9, 122.3, 121.7, 47.7 (d, *J* = 67.0 Hz), 22.1, 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.8; IR (neat) 3416, 1621, 1188cm^{-1}; HRMS (EI-TOF) calcd for $C_{31}H_{27}OPS$ 478.1520, found 478.1524.



5f

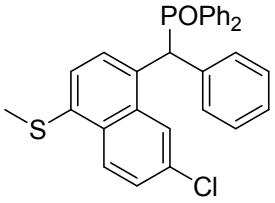
((7-methyl-4-(methylthio)naphthalen-1-yl)(p-tolyl)methyl)diphenylphosphine oxide(5f): Pale amorphous solid, 187mg, 76% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 7.8 Hz, 1H), 8.10 (d, *J* = 8.6 Hz, 1H), 7.71 (s, 1H), 7.66 – 7.57 (m, 2H), 7.55 – 7.46 (m, 2H), 7.41 (t, *J* = 6.9 Hz, 1H), 7.25 (m, 9H), 6.91 (d, *J* = 7.9 Hz, 2H), 5.42 (d, *J* = 10.4 Hz, 1H), 2.45 (d, *J* = 8.5 Hz, 6H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.5 (d, *J* = 2.0 Hz), 136.2, 135.2, 133.6, 133.0, 132.5 (d, *J* = 10.0 Hz), 132.4, 132.1, 131.9 (d, *J* = 9.0 Hz), 131.4 (d, *J* = 3.0 Hz), 131.3 (d, *J* = 3.0 Hz), 131.1 (d, *J* = 8.0 Hz), 130.3, 130.0 (d, *J* = 5.0 Hz), 128.8 (d, *J* = 2.0 Hz), 128.4 (d, *J* = 10.0 Hz), 128.2, 128.0, 127.6, 124.9, 122.3, 121.7, 47.2 (d, *J* = 67.0 Hz), 22.1, 21.0, 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.7; IR (neat) 3419, 1623, 1187cm^{-1}; HRMS (EI-TOF) calcd for $C_{32}H_{29}OPS$ 492.1677, found 492.1678.



5g

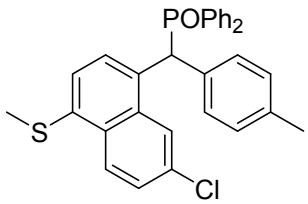
((7-methoxy-4-(methylthio)naphthalen-1-yl)(p-tolyl)methyl)diphenylphosphine oxide(5g): Pale amorphous solid, 197mg, 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 7.7 Hz, 1H), 8.14 (d, *J* = 9.2 Hz, 1H), 7.71 – 7.60 (m, 2H), 7.48 – 7.34 (m, 4H), 7.31 – 7.20 (m, 7H), 7.15 – 7.05 (m, 5H), 5.29 (d, *J* = 10.7 Hz, 1H), 3.80 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 135.4 (d, *J* = 5.0 Hz), 133.5, 133.2 (d, *J* = 9.0 Hz), 132.8, 131.8, 131.5 (d, *J* = 2.0 Hz), 131.3 (d, *J* = 5.0 Hz), 131.2 (d, *J* = 8.0 Hz), 131.1, 130.2 (d, *J* = 5.0 Hz), 129.7 (d, *J* = 3.0 Hz), 128.9 (d, *J* = 7.0

Hz), 128.5 (d, *J* = 11.0 Hz), 128.1, 128.0 (d, *J* = 8.0 Hz), 127.2, 126.9, 120.4, 116.7, 114.1, 103.4, 55.3, 48.5 (d, *J* = 66.0 Hz), 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.4; IR (neat) 3420, 1621, 1182cm^{-1}; HRMS (EI-TOF) calcd for $C_{31}H_{27}O_2PS$ 494.1469, found 494.1474.



5h

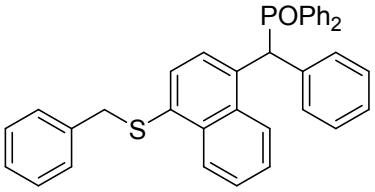
((7-chloro-4-(methylthio)naphthalen-1-yl)(phenyl)methyl)diphenylphosphine oxide(5h): Pale amorphous solid, 187mg, 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 7.9 Hz, 1H), 8.15 (d, *J* = 9.0 Hz, 1H), 7.90 (s, 1H), 7.66 – 7.57 (m, 2H), 7.51 – 7.42 (m, 3H), 7.40 – 7.31 (m, 4H), 7.24 (d, *J* = 5.4 Hz, 5H), 7.15 – 7.08 (m, 3H), 5.31 (d, *J* = 10.5 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.9, 135.9, 135.2 (d, *J* = 5.0 Hz), 132.8, 131.9, 131.6 (d, *J* = 3.0 Hz), 131.5 (d, *J* = 3.0 Hz), 131.3 (d, *J* = 9.0 Hz), 131.0 (d, *J* = 8.0 Hz), 130.0, 129.4 (d, *J* = 7.0 Hz), 128.6, 128.4, 128.2, 128.1, 127.1 (d, *J* = 2.0 Hz), 126.8, 126.3, 122.7, 122.3, 47.7 (d, *J* = 62.0 Hz), 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.8; IR (neat) 3421, 1621, 1190cm^{-1}; HRMS (EI-TOF) calcd for $C_{30}H_{24}ClOPS$ 498.0974, found 498.0976.



5i

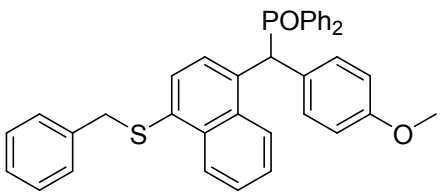
((7-chloro-4-(methylthio)naphthalen-1-yl)(p-tolyl)methyl)diphenylphosphine oxide(5i): Pale amorphous solid, 209mg, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 7.8 Hz, 1H), 8.14 (d, *J* = 9.0 Hz, 1H), 7.89 (d, *J* = 1.9 Hz, 1H), 7.63 – 7.58 (m, 2H), 7.51 (dd, *J* = 11.1, 7.2 Hz, 2H), 7.42 (d, *J* = 7.4 Hz, 1H), 7.38 – 7.30

(m, 4H), 7.26 – 7.22 (m, 3H), 7.14 (d, *J* = 6.6 Hz, 2H), 6.93 (d, *J* = 7.9 Hz, 2H), 5.29 (d, *J* = 10.4 Hz, 1H), 2.49 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 135.8, 133.1, 132.7 (d, *J* = 12.0 Hz), 132.5, 132.1 (d, *J* = 5.0 Hz), 131.5, 131.3 (d, *J* = 8.0 Hz), 131.0 (d, *J* = 8.0 Hz), 130.2, 130.1, 129.8 (d, *J* = 5.0 Hz), 129.4 (d, *J* = 7.0 Hz), 129.1, 128.5, 128.4, 128.2 (d, *J* = 12.0 Hz), 126.8, 126.2, 122.8, 122.3, 105.0, 47.2 (d, *J* = 66.0 Hz), 21.0, 15.6; ^{31}P NMR (160 MHz, $CDCl_3$) δ 32.4; IR (neat) 3416, 1622, 1188cm^{-1}; HRMS (EI-TOF) calcd for $C_{31}H_{26}ClOPS$ 512.1131, found 512.1135.



5j

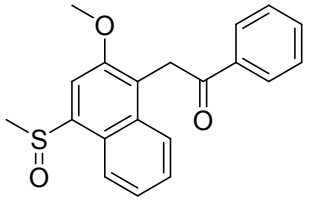
diphenyl(phenyl(4-(phenylthio)naphthalen-1-yl)methyl)phosphine oxide(5j): Pale amorphous solid, 191mg, 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.46 (m, 5H), 7.43 (t, *J* = 7.1 Hz, 3H), 7.36 (d, *J* = 6.8 Hz, 1H), 7.32 – 7.28 (m, 7H), 7.27 (s, 3H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.2 Hz, 3H), 7.03 (d, *J* = 6.7 Hz, 2H), 6.90 (s, 1H), 5.02 (d, *J* = 10.3 Hz, 1H), 3.63 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.3, 138.9 (d, *J* = 9.0 Hz), 135.8 (d, *J* = 4.0 Hz), 134.1, 131.7 (d, *J* = 4.0 Hz), 131.5, 131.3 (d, *J* = 8.0 Hz), 131.2, 129.0, 128.7, 128.6, 128.5, 128.4 (d, *J* = 11.0 Hz), 128.2, 128.1, 127.3, 127.0, 126.3, 125.4, 124.6, 46.9 (d, *J* = 67.0 Hz), 34.4; ^{31}P NMR (160 MHz, $CDCl_3$) δ 31.3; IR (neat) 3415, 1621, 1190cm^{-1}; HRMS (EI-TOF) calcd for $C_{36}H_{29}OPS$ 540.1677, found 540.1677.



5k

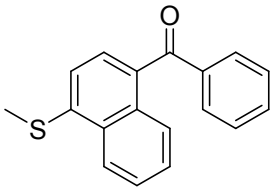
((4-methoxyphenyl)(4-(phenylthio)naphthalen-1-yl)methyl)diphenylphosphine

oxide(5k): Pale amorphous solid, 194mg, 68% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.5 Hz, 3H), 7.47 (d, *J* = 8.0 Hz, 3H), 7.45 – 7.35 (m, 3H), 7.30 (d, *J* = 7.3 Hz, 6H), 7.27 (s, 2H), 7.20 (d, *J* = 7.5 Hz, 3H), 7.02 (d, *J* = 6.8 Hz, 2H), 6.89 (s, 1H), 6.67 (d, *J* = 8.5 Hz, 2H), 4.98 (d, *J* = 10.3 Hz, 1H), 3.70 (s, 3H), 3.60 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 143.2, 139.9, 138.7, 134.1, 131.6 (d, *J* = 8.0 Hz), 131.5 (d, *J* = 10.0 Hz), 131.3 (d, *J* = 4.0 Hz), 131.2, 130.9, 128.6 (d, *J* = 9.0 Hz), 128.4, 128.3 (d, *J* = 9.0 Hz), 128.1, 127.8, 127.5, 127.2, 126.3, 125.4, 124.6, 113.8, 55.2, 45.9 (d, *J* = 67.0 Hz), 34.4; ^{31}P NMR (160 MHz, $CDCl_3$) δ 31.3; IR (neat) 3416, 1621, 1186cm^{-1}; HRMS (EI-TOF) calcd for $C_{37}H_{31}O_2PS$ 570.1782, found 570.1785.



6

2-(2-methoxy-4-(methylsulfinyl)naphthalen-1-yl)-1-phenylethanone(6): Colorless oil, 152mg, 90% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (dd, *J* = 6.8, 2.9 Hz, 1H), 8.15 (s, 1H), 8.06 (d, *J* = 8.6 Hz, 2H), 7.81 (s, 1H), 7.58 – 7.46 (m, 5H), 4.82 (s, 2H), 3.98 (s, 3H), 3.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.48, 144.76, 139.95, 134.89, 134.55, 129.74, 129.06, 127.70, 126.11, 124.46, 124.31, 124.28, 123.80, 116.10, 56.75, 44.40, 35.96; IR (neat) 3417, 1622, 1191cm^{-1}; HRMS (EI-TOF) calcd for $C_{20}H_{18}O_3S$ 338.0977, found 338.0974.

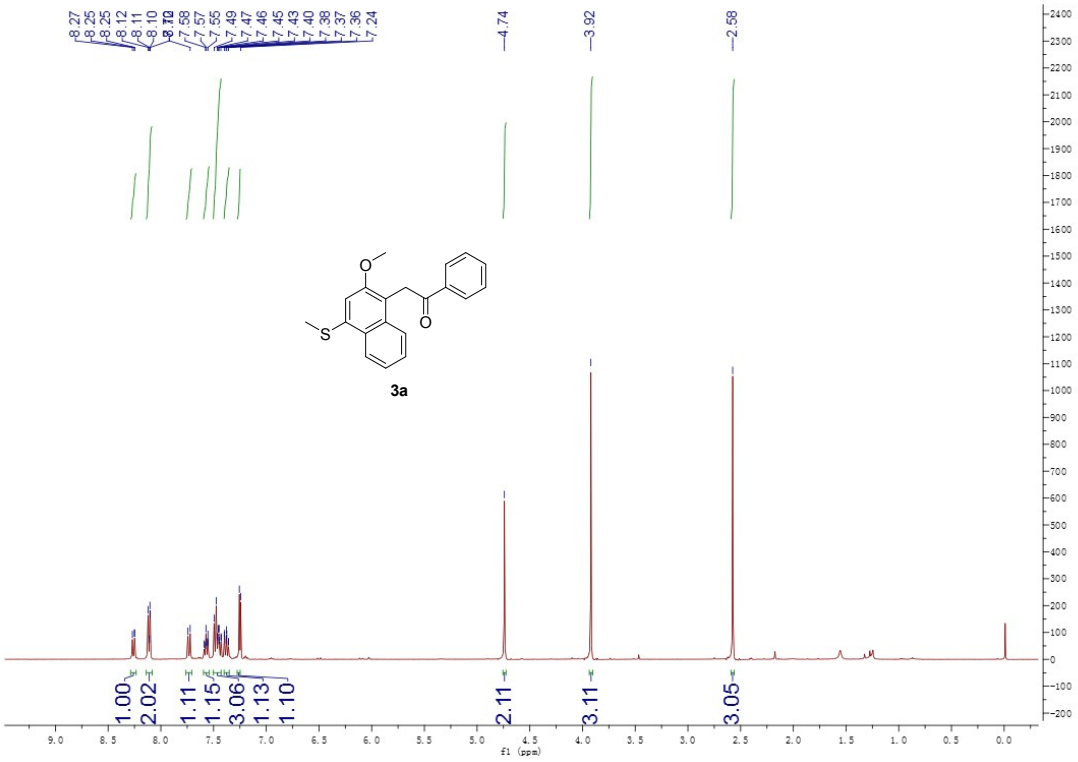


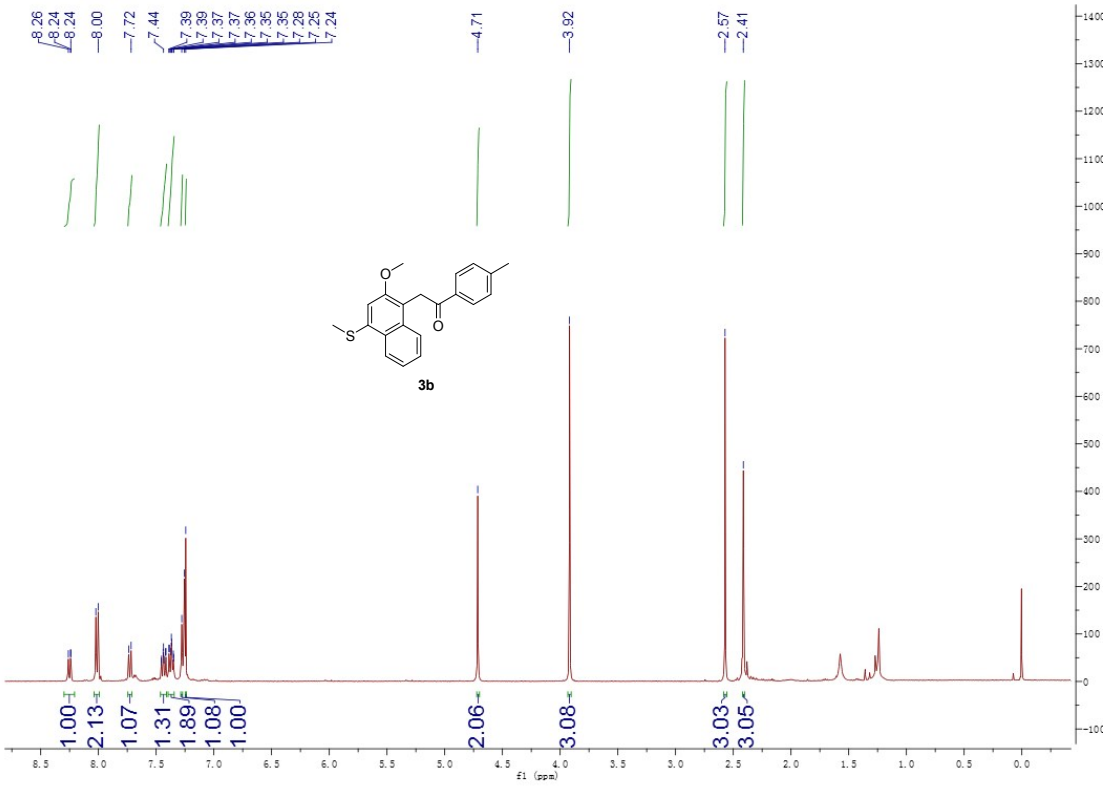
7

(4-(methylthio)naphthalen-1-yl)(phenyl)methanone(7): Colorless oil, 90mg, 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.27 (m, 1H), 8.23 – 8.18 (m, 1H),

7.87 – 7.80 (m, 2H), 7.60 – 7.51 (m, 4H), 7.45 (t, J = 7.7 Hz, 2H), 7.29 (d, J = 7.7 Hz, 1H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.59, 141.23, 138.64, 133.00, 132.82, 131.15, 130.98, 130.35, 128.37, 127.64, 126.52, 126.41, 124.11, 119.58, 15.28; IR (neat) 3417, 1620, 1192cm^{-1}; HRMS (EI-TOF) calcd for $C_{18}H_{14}OS$ 278.0765, found 278.0763.

5. The ^{1}H and ^{13}C NMR spectra of compounds

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7.25
7.24
4.71
3.92
2.57
2.41
O
S
O
3b
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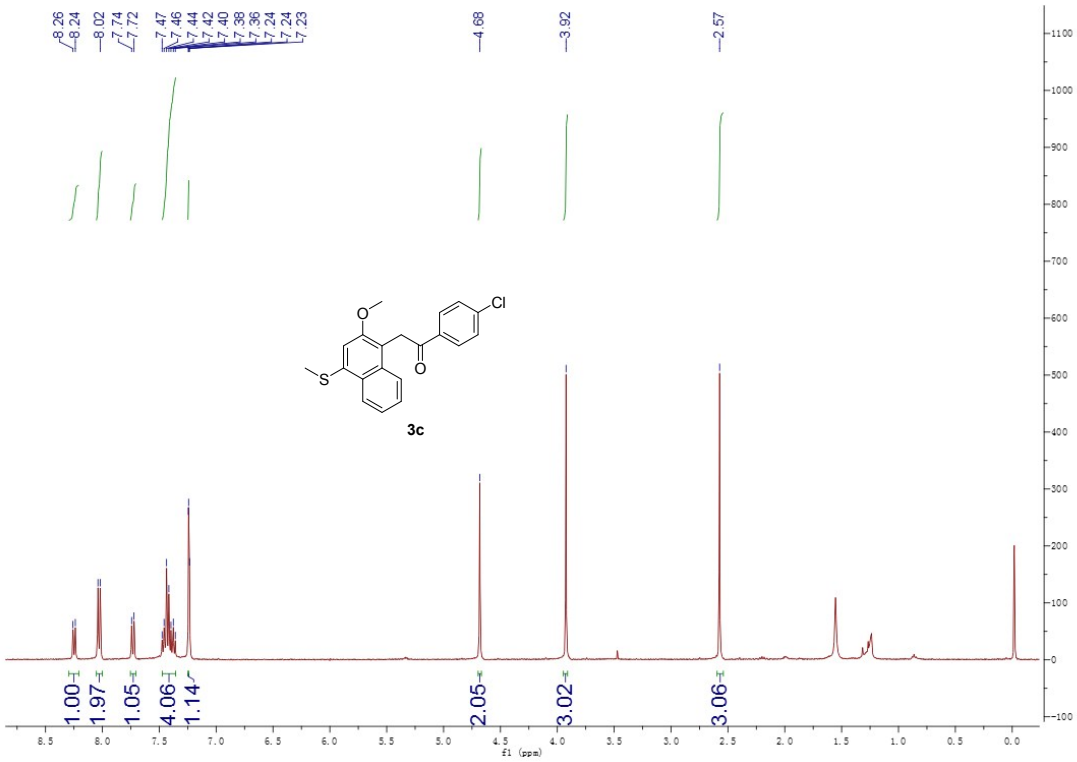
3.05
f1 (ppm)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7.24
7.23
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3.92
2.57
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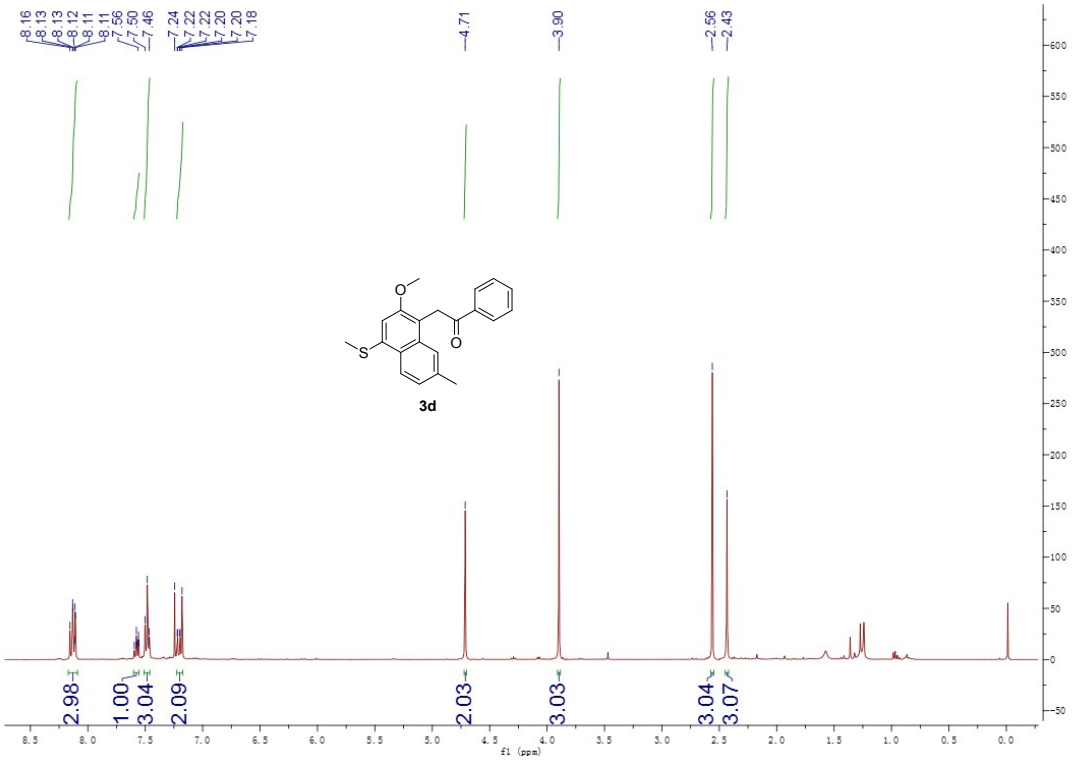
f1 (ppm)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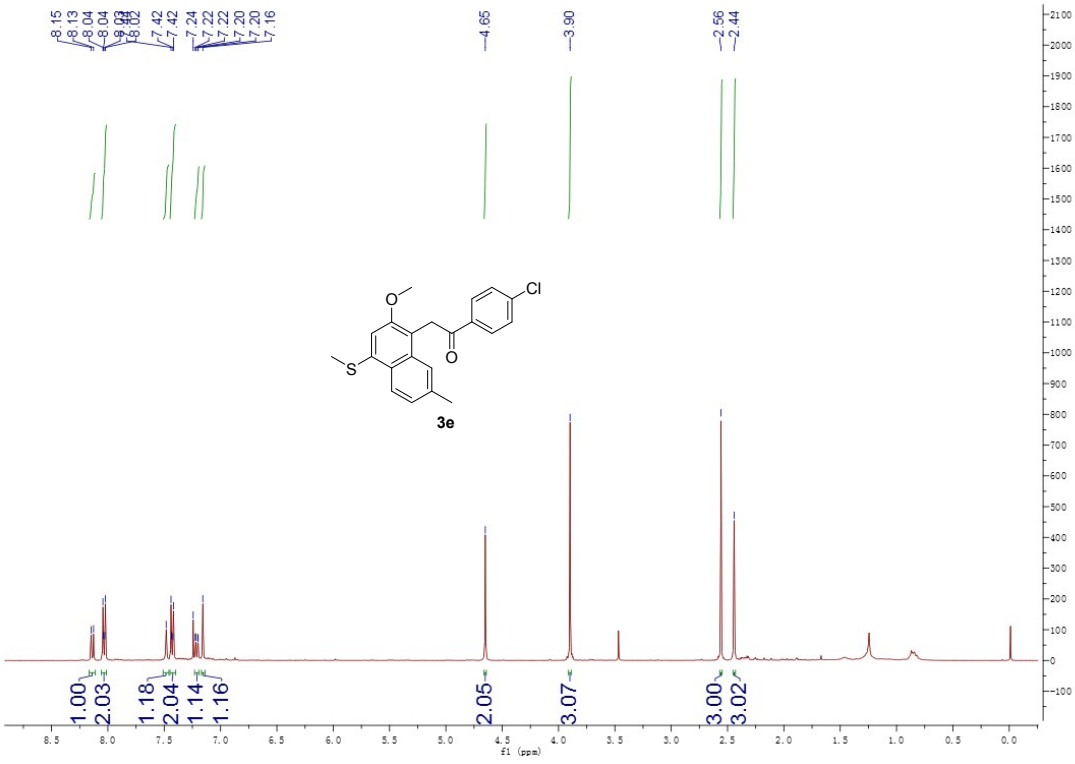
f1 (ppm)

3d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Cl
O
S
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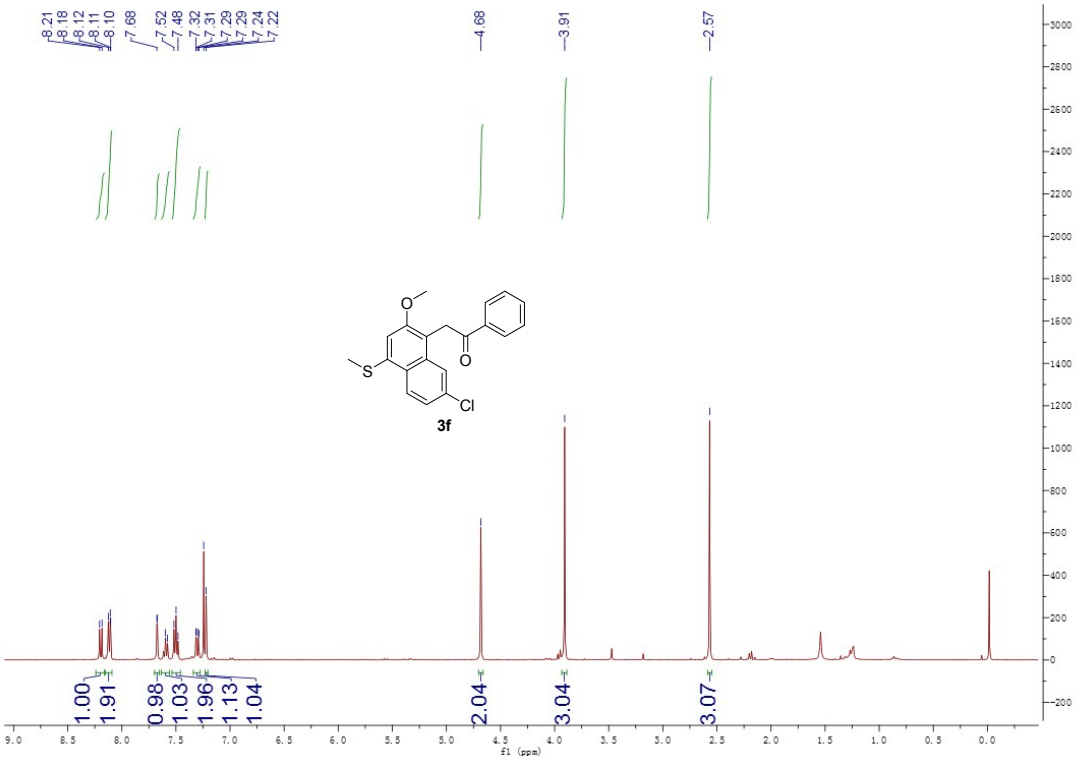
f1 (ppm)

3e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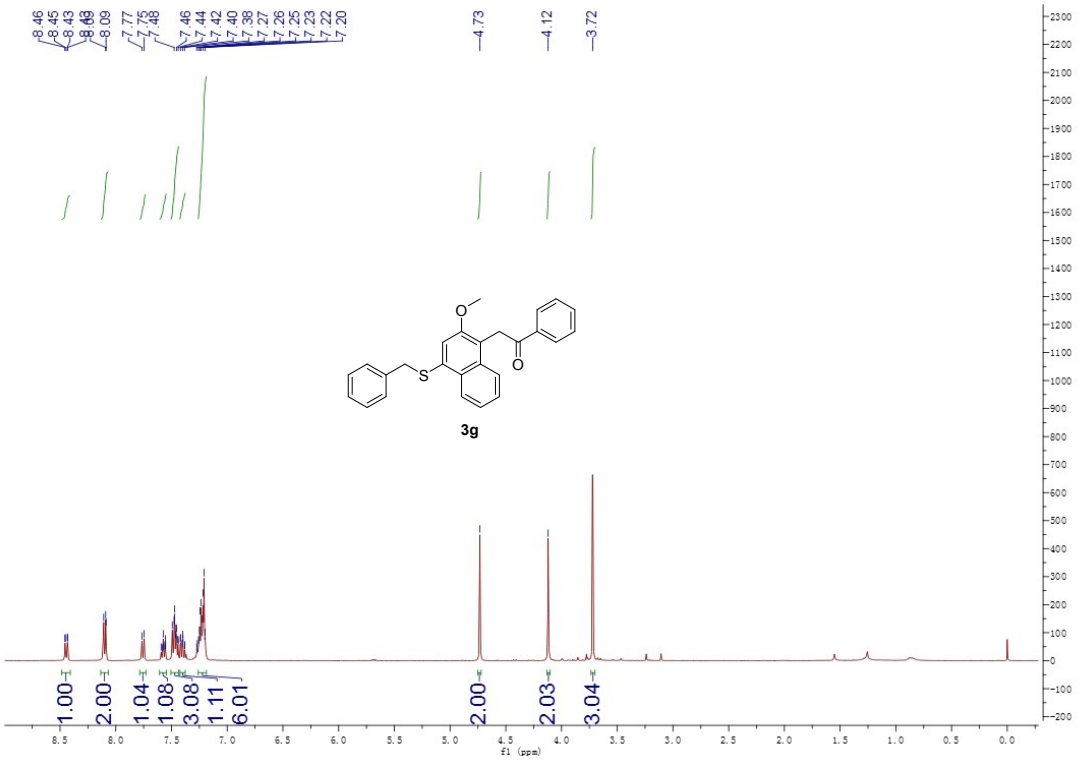
f1 (ppm)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7.23
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7.20
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4.12
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S
O
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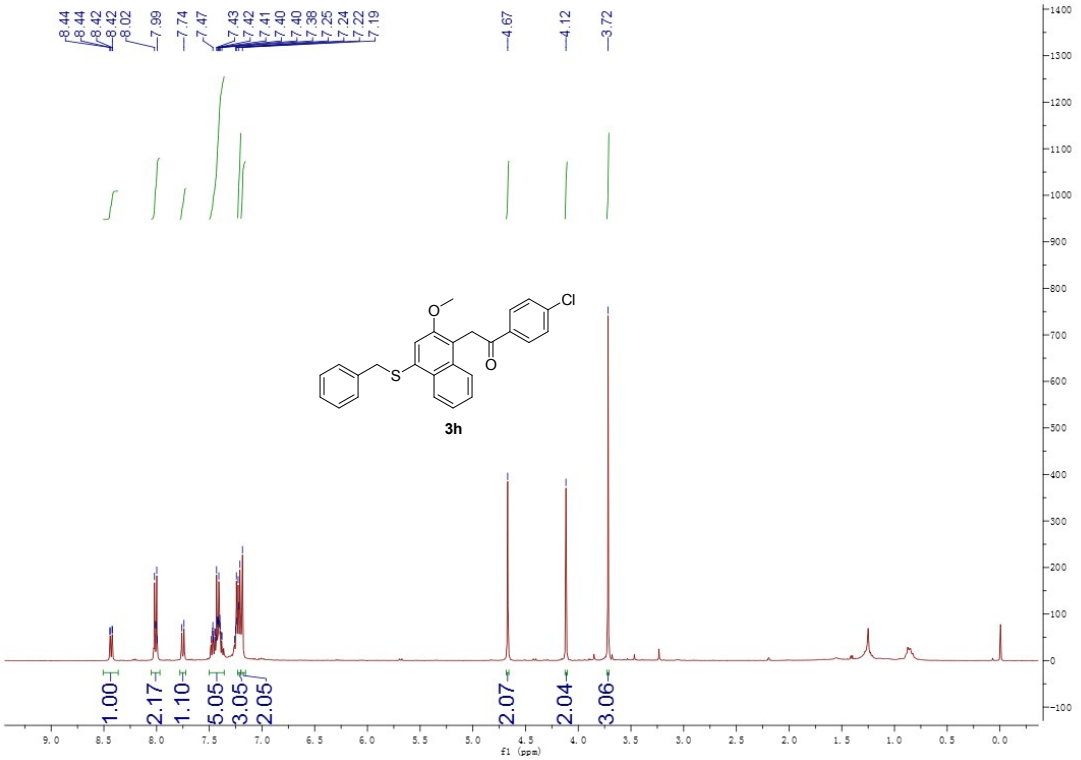
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Cl
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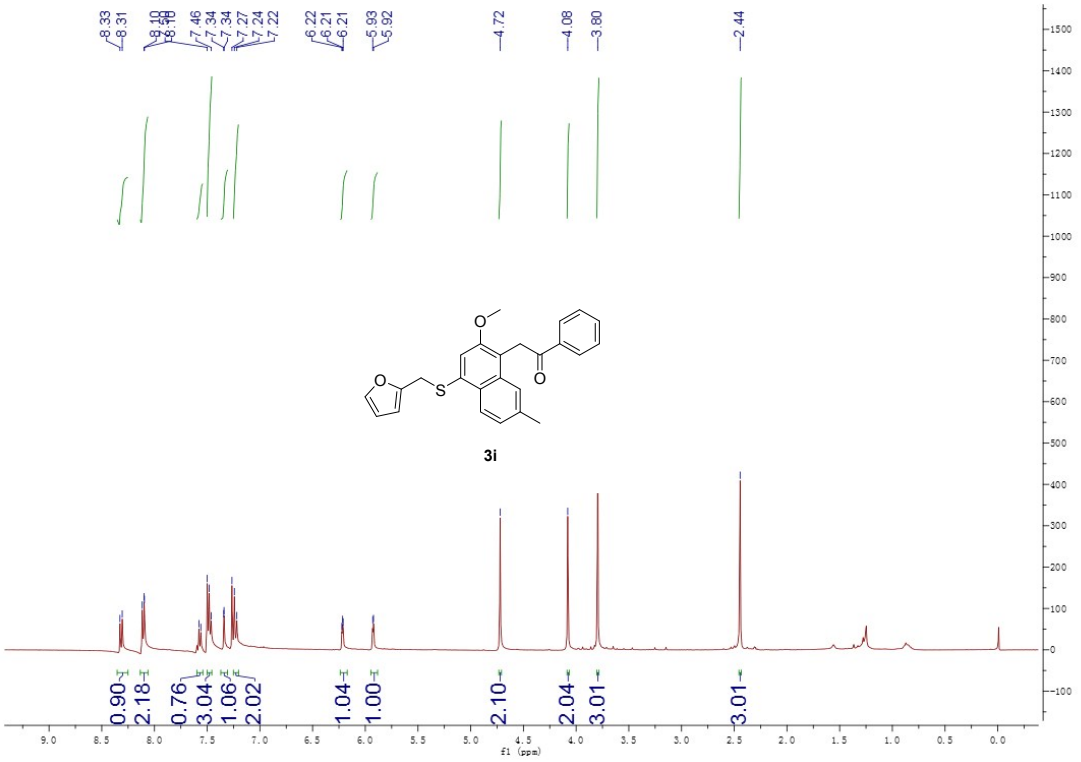
f1 (ppm)

3h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5.92
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4.08
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O
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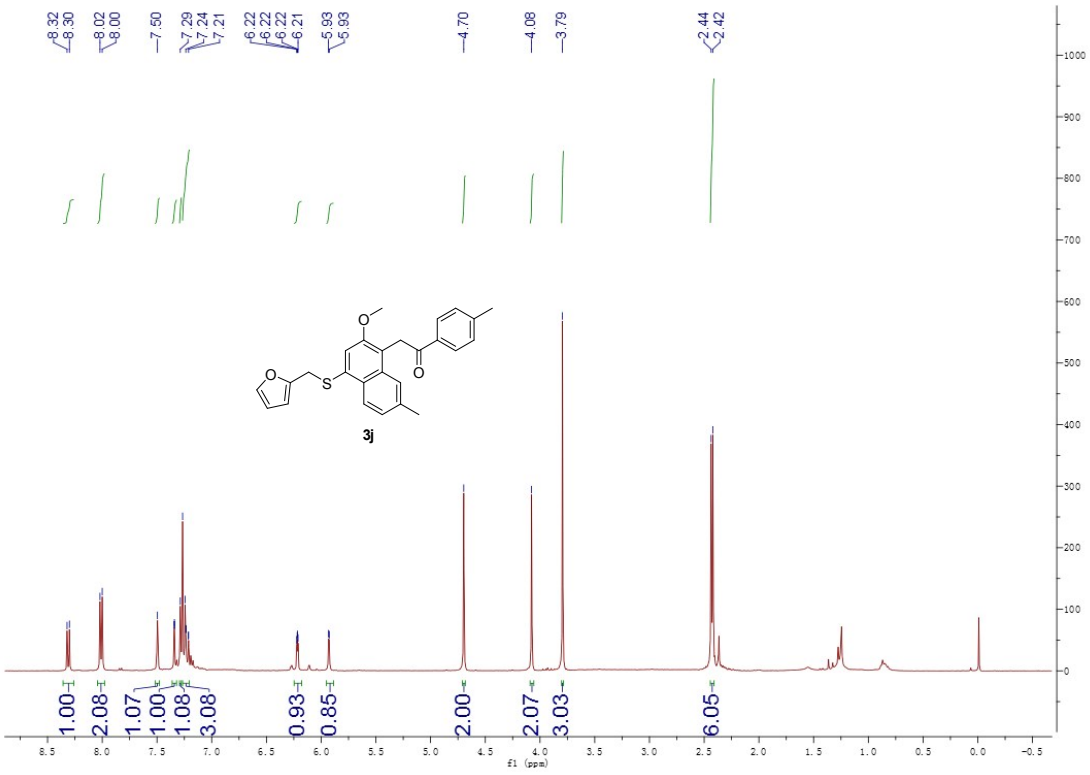
f1 (ppm)

3i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O
O
S
O
3j
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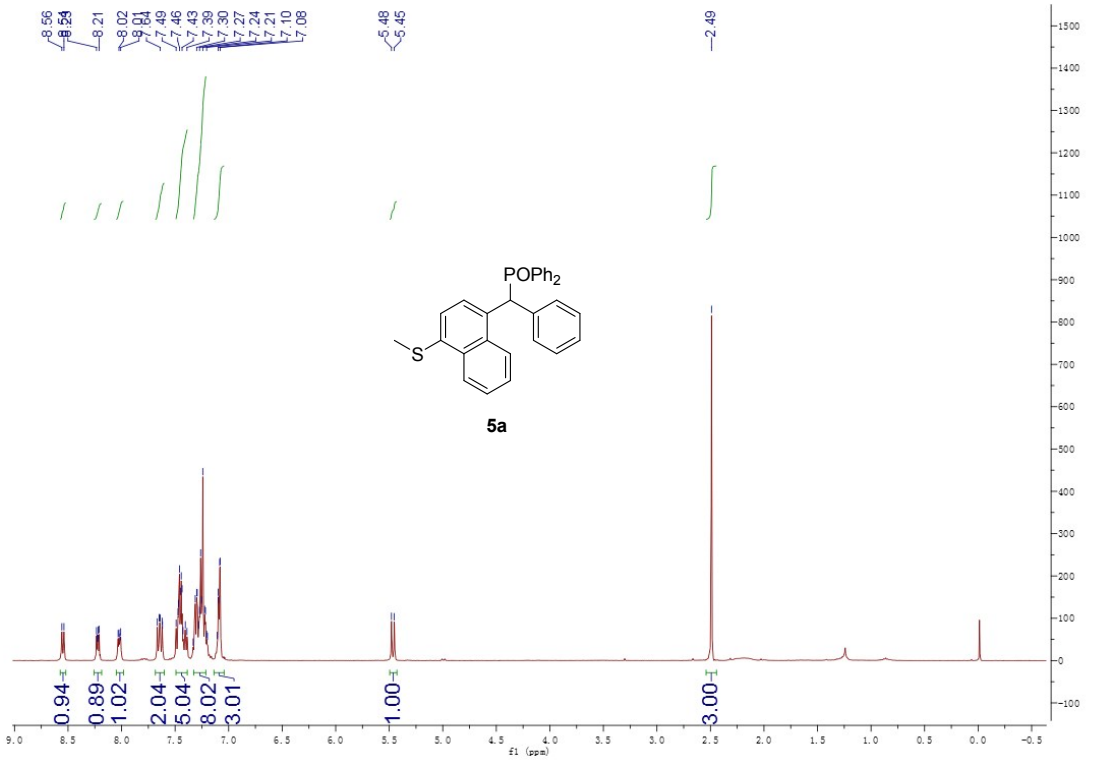
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5.45
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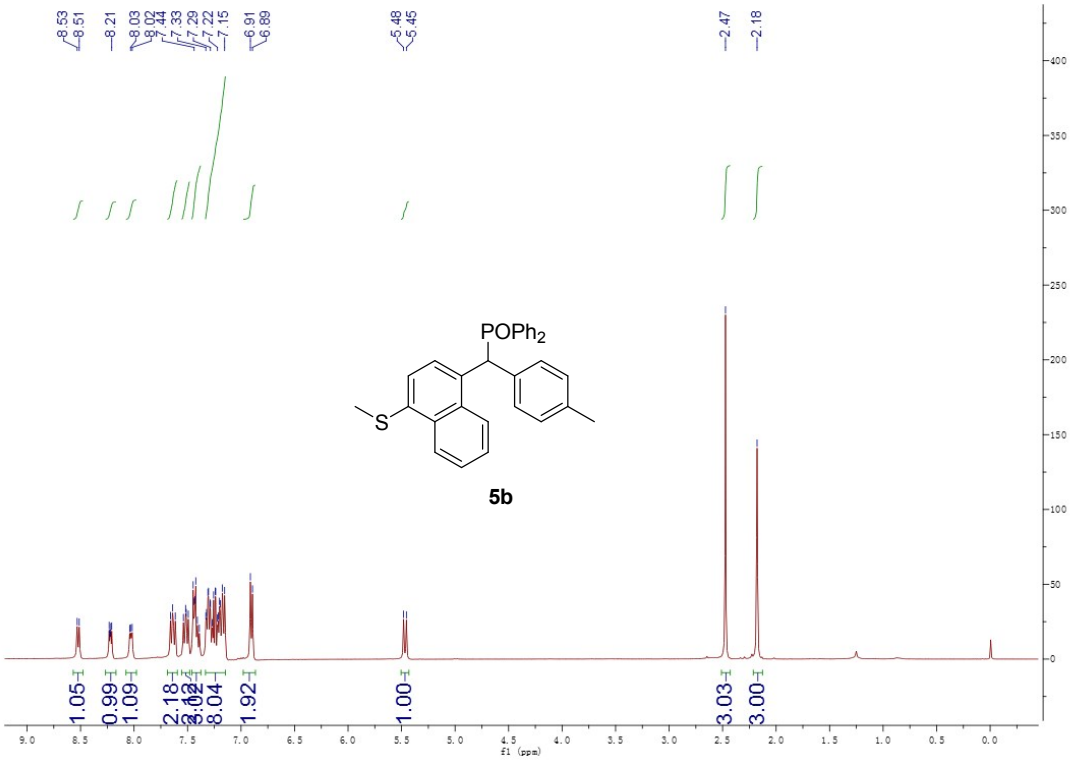
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POPh2
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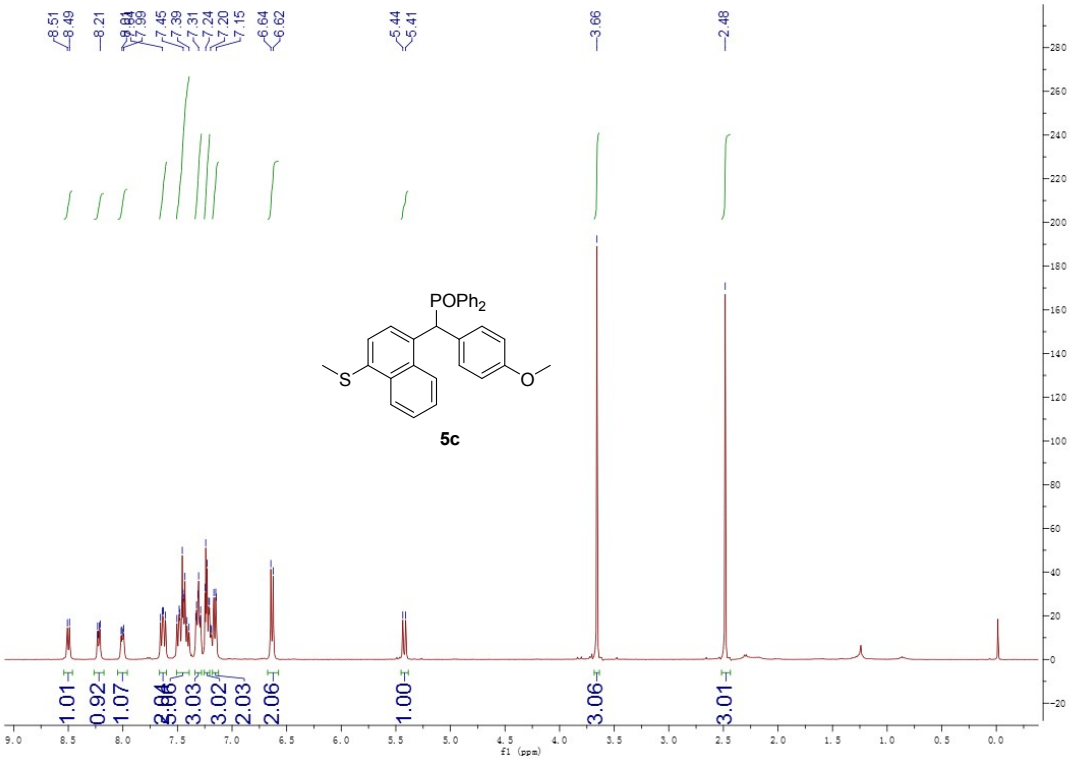
5b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O
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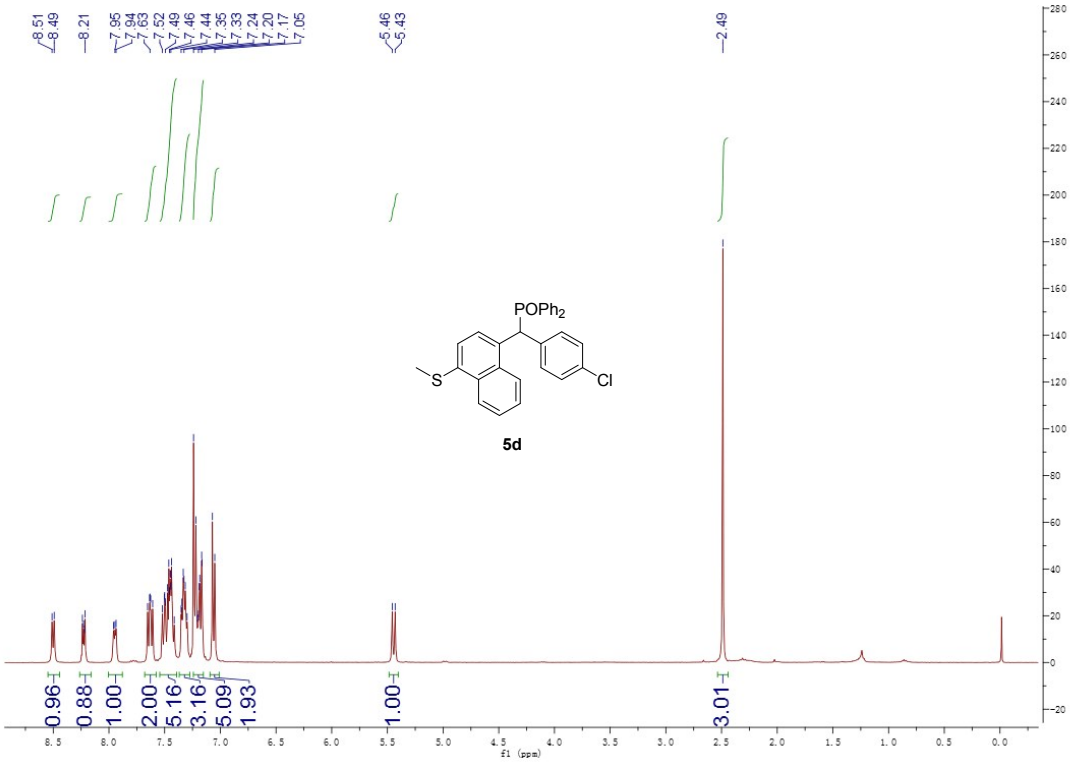
f1 (ppm)

5c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POPh2
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Cl
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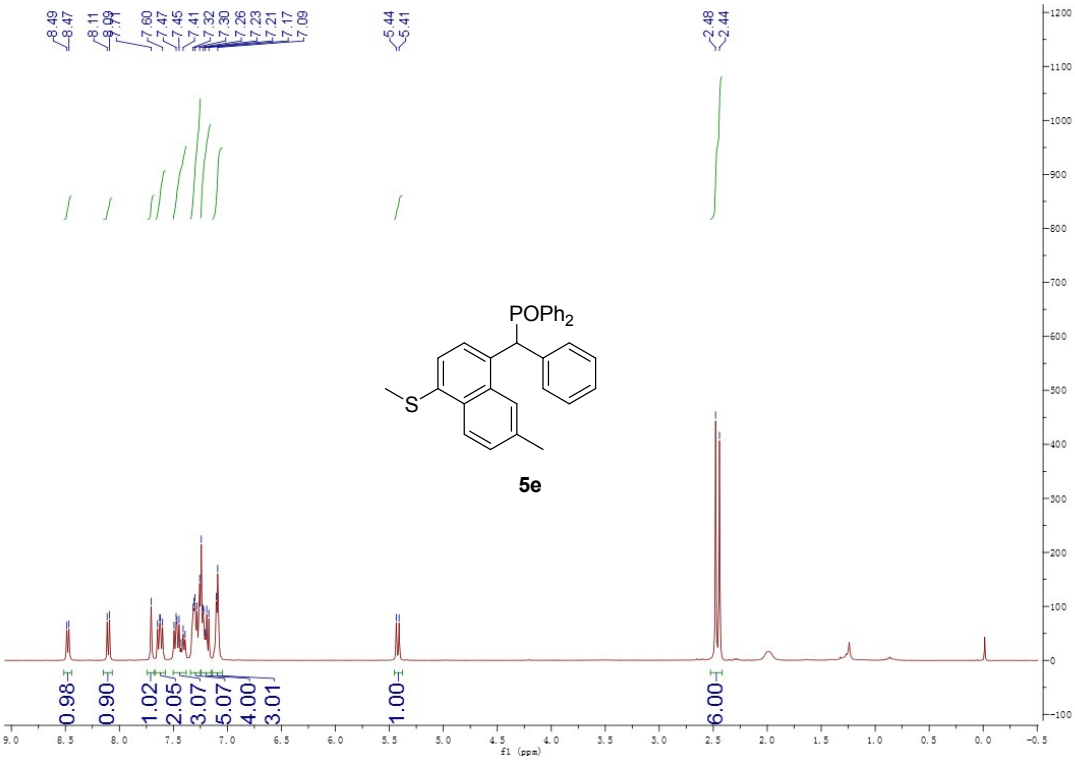
5d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5.44
5.41
2.48
2.44
POPh2
S
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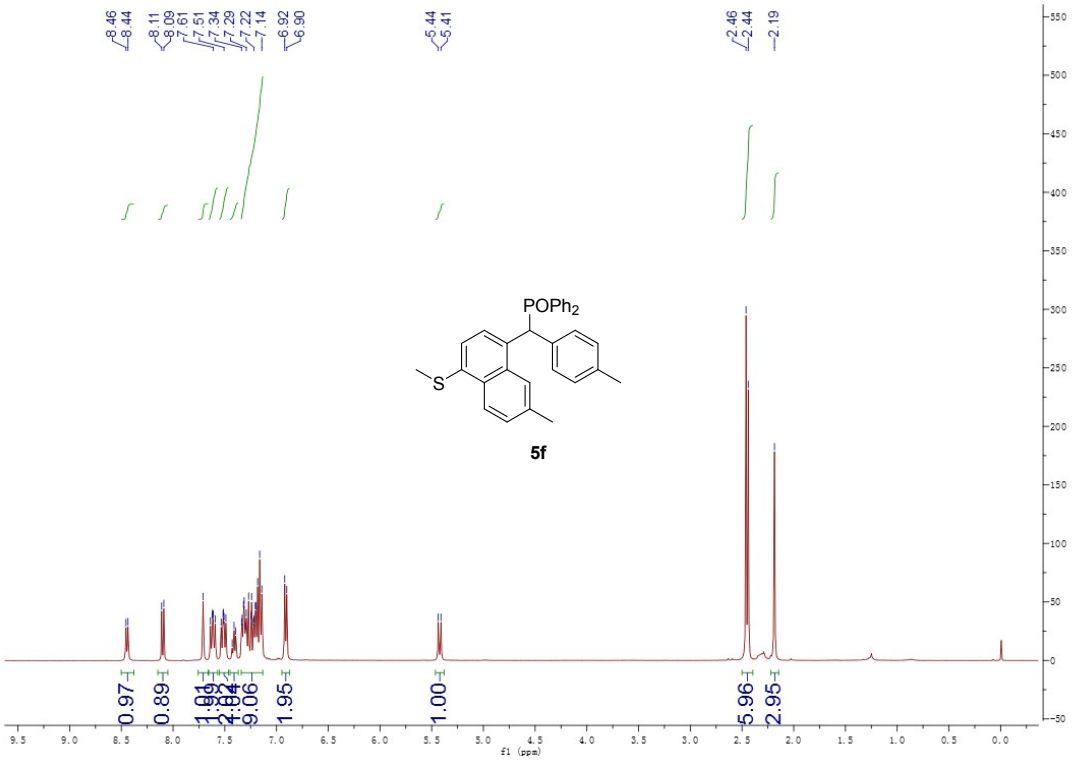
f1 (ppm)

5e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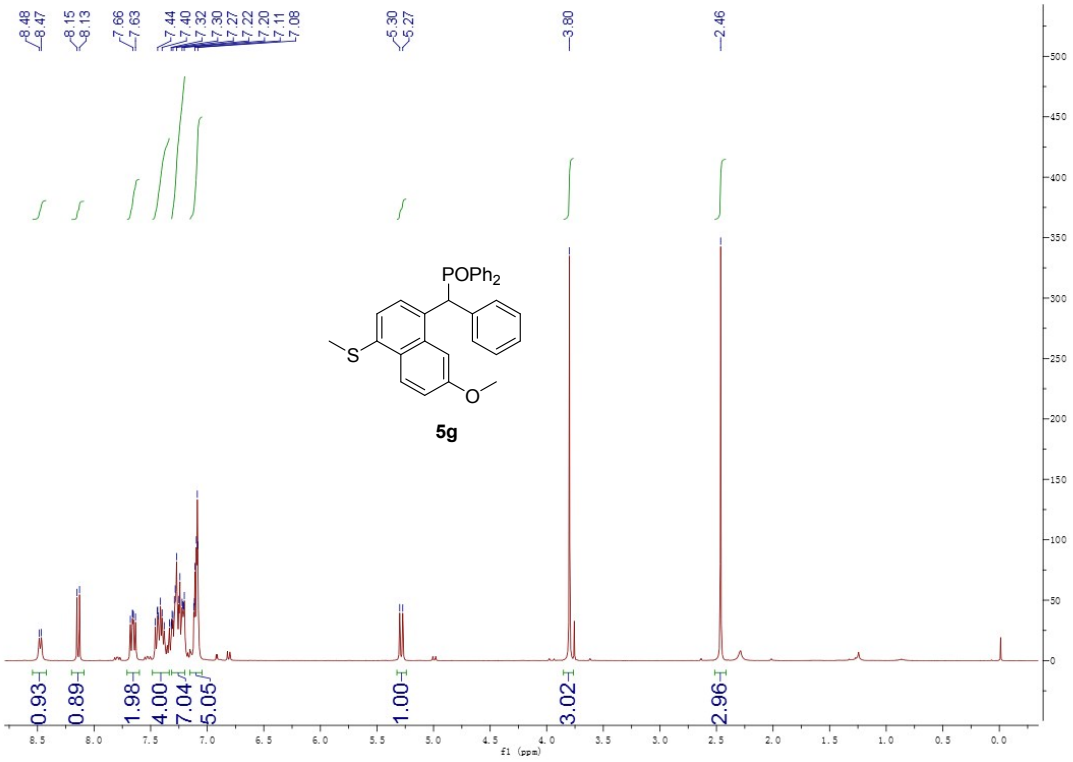
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7.08
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3.80
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POPh2
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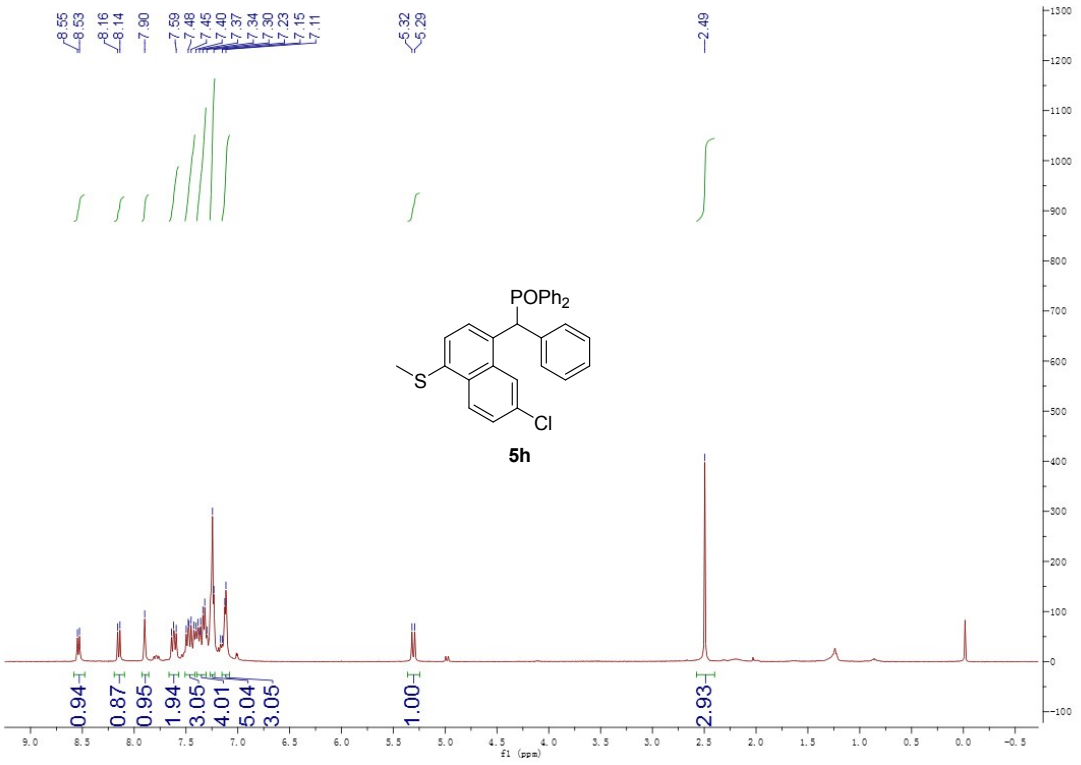
5g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Cl
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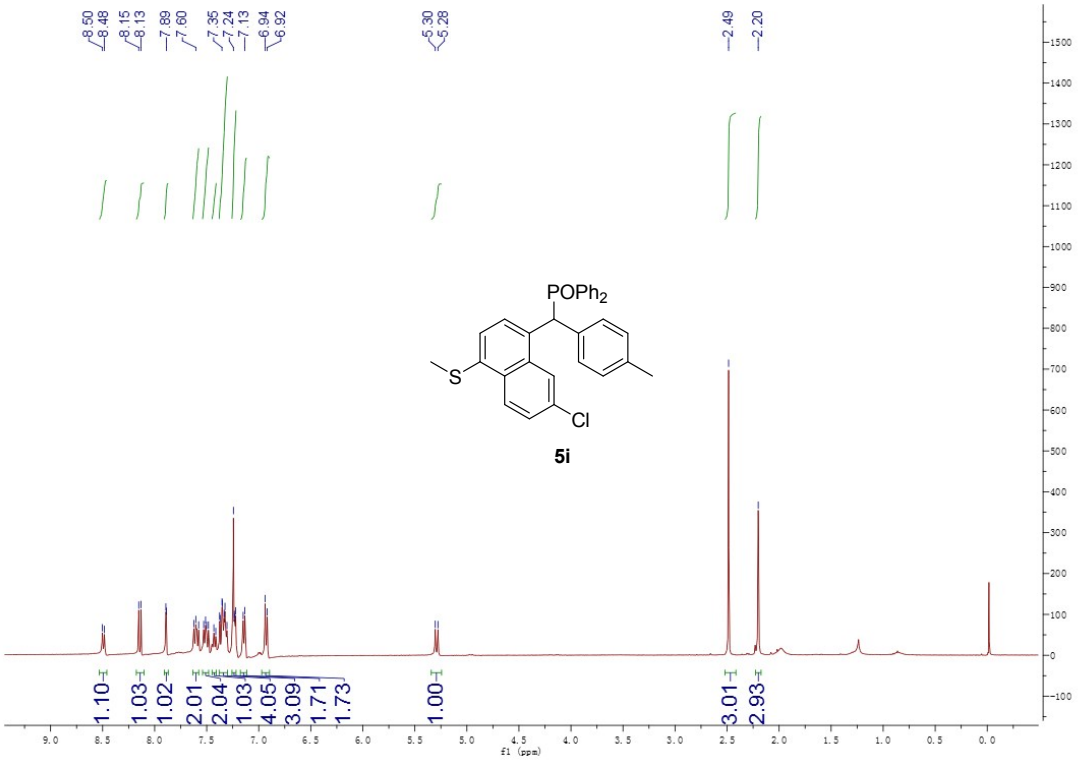
5h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POPh2
S
Cl
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1.03
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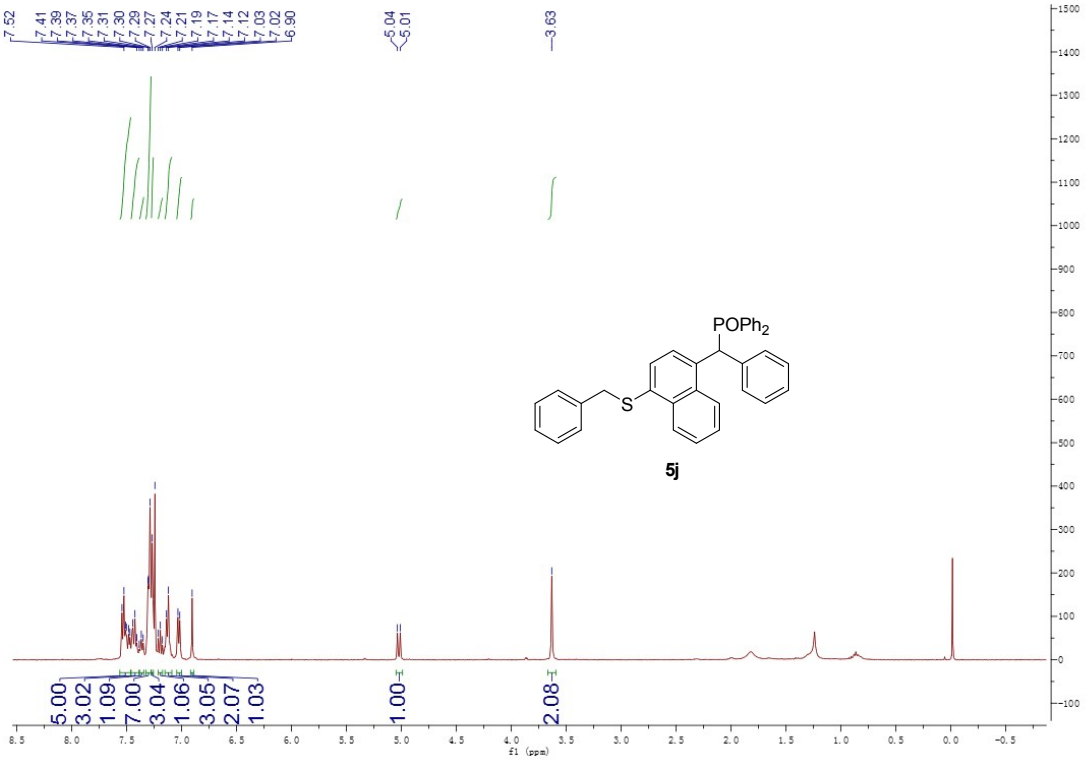
5i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7.02
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5.04
5.01
3.63
POPh2
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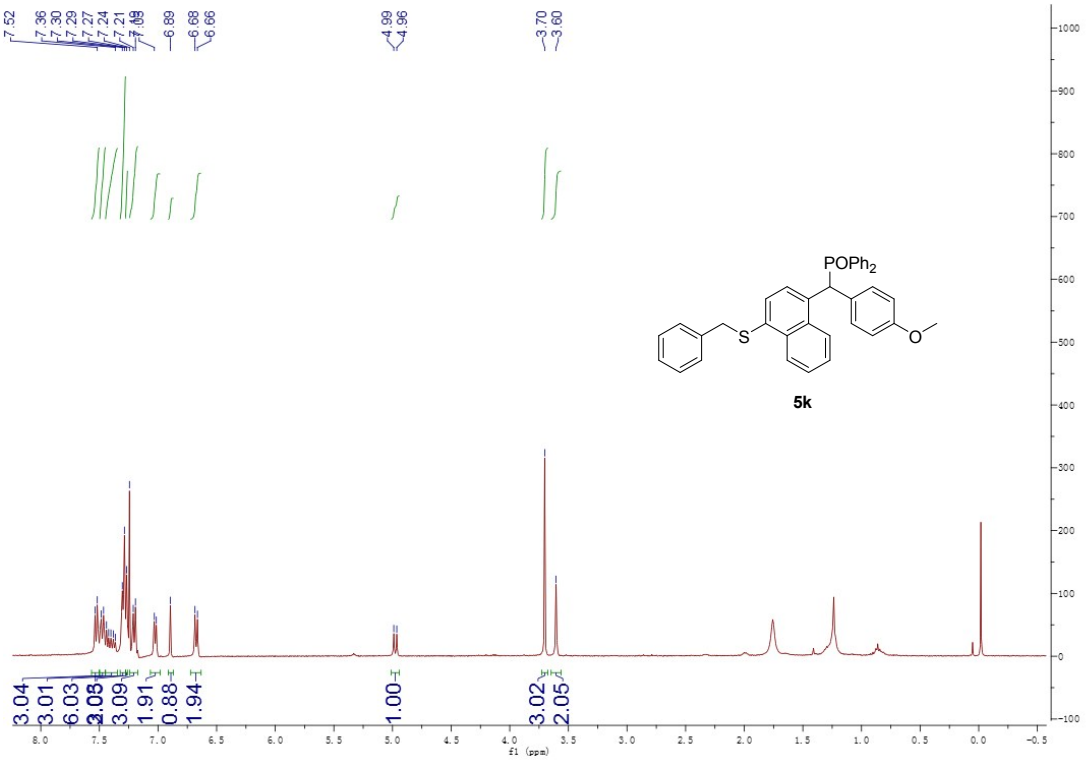
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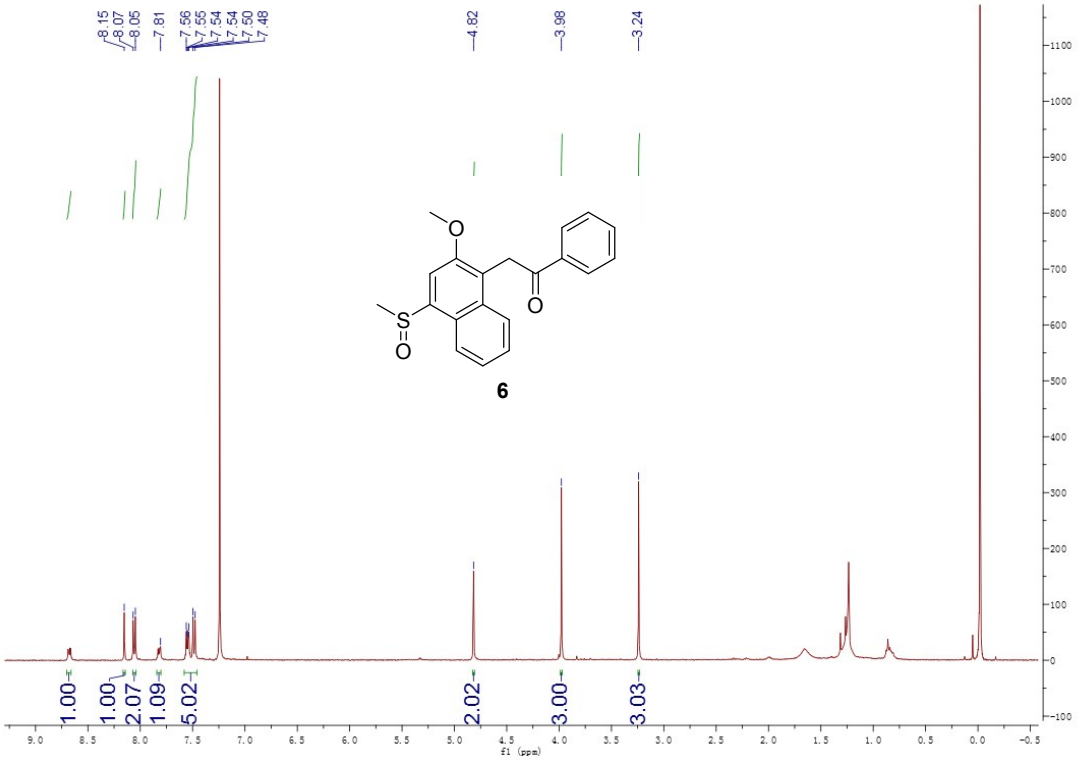
f1 (ppm)

5k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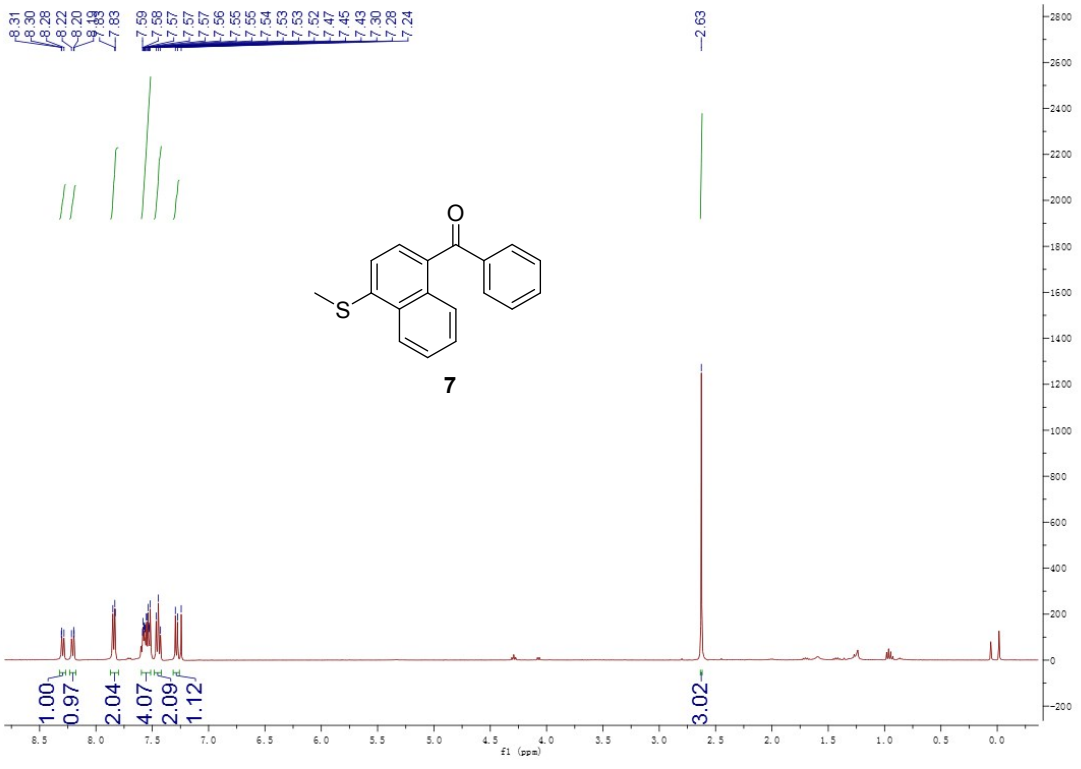
6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7.43
7.30
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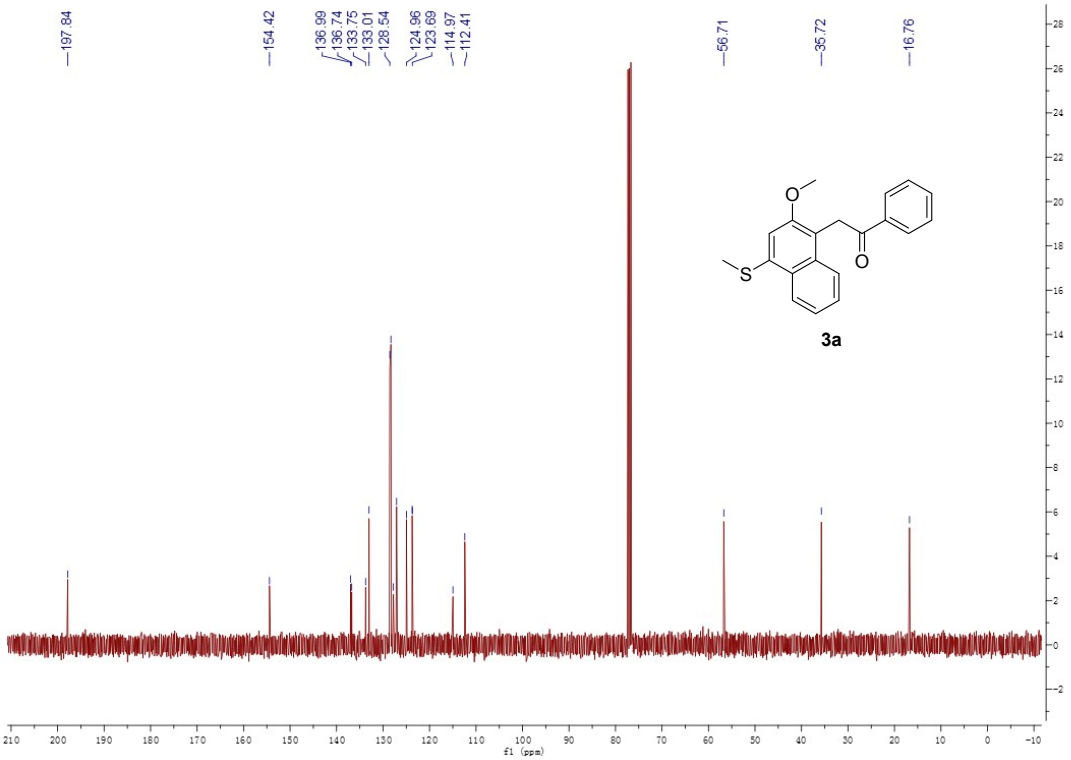
f1 (ppm)

7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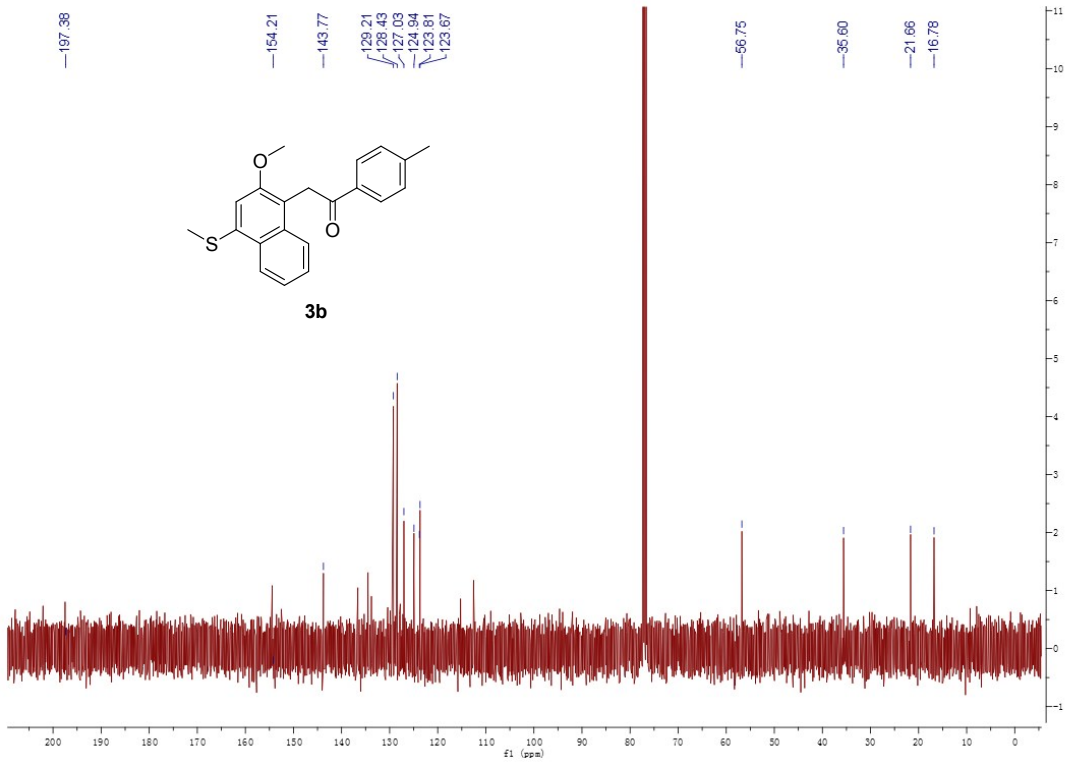
S
O
f1 (ppm)

3a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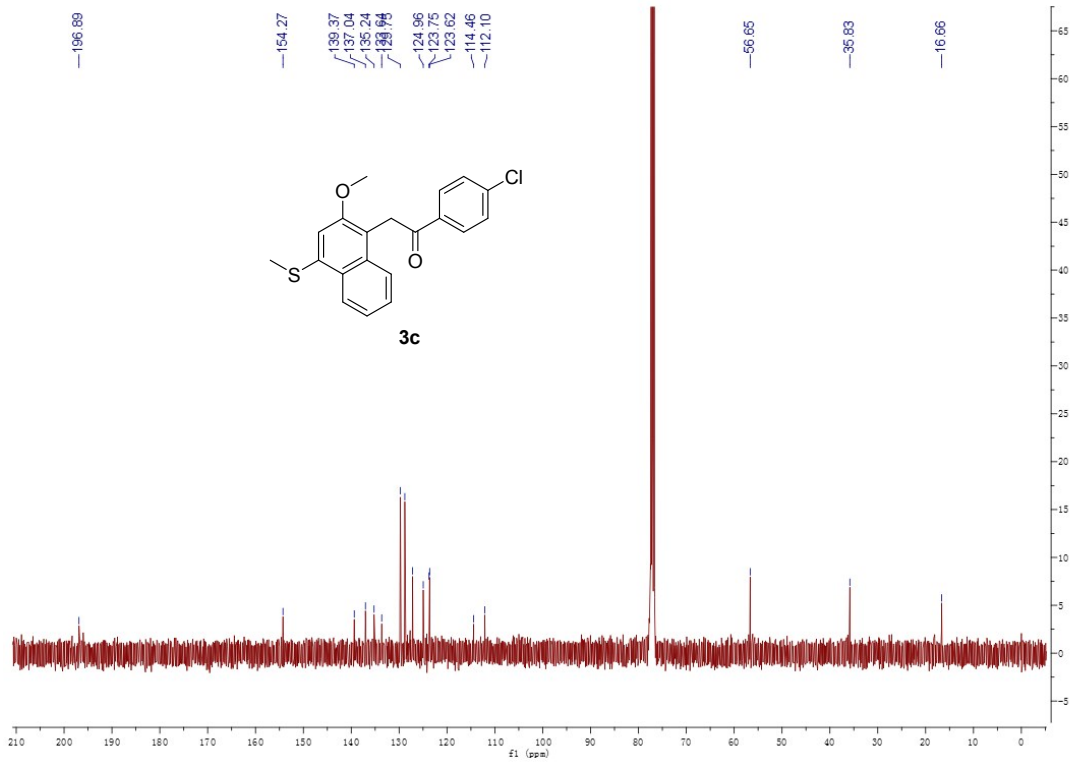
f1 (ppm)

3b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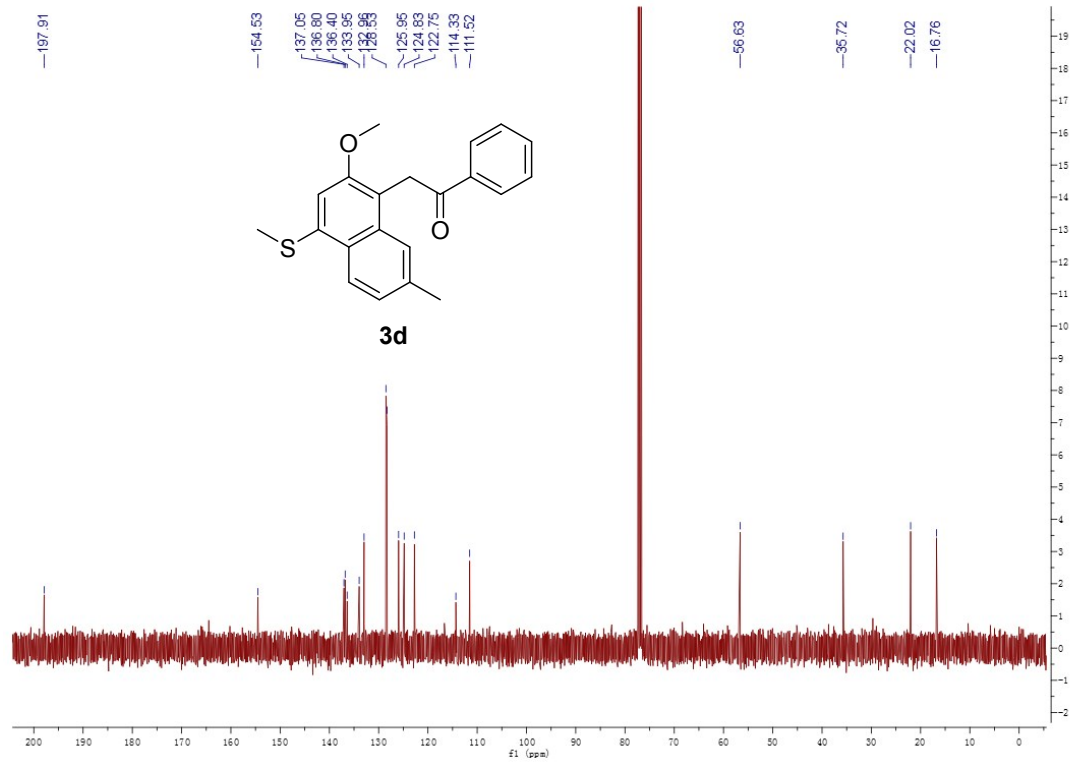
16.66
O
Cl
S
O
f1 (ppm)

3c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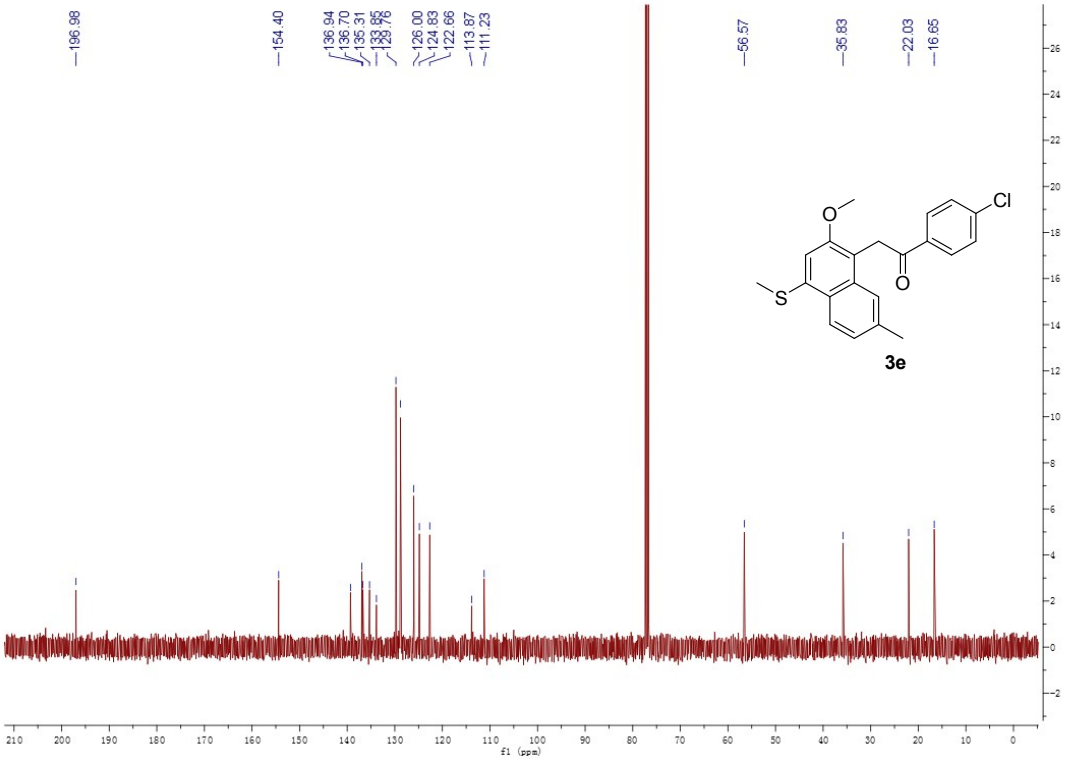
22.02
16.76
O
S
O
f1 (ppm)

3d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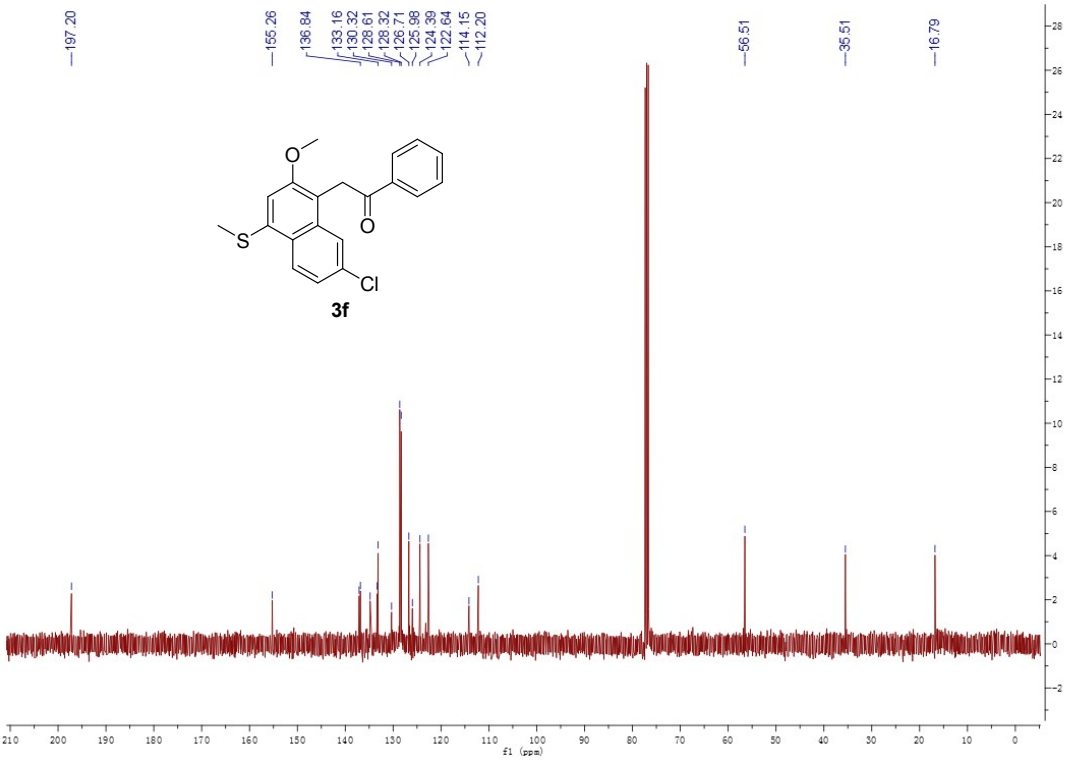
S
f1 (ppm)

3e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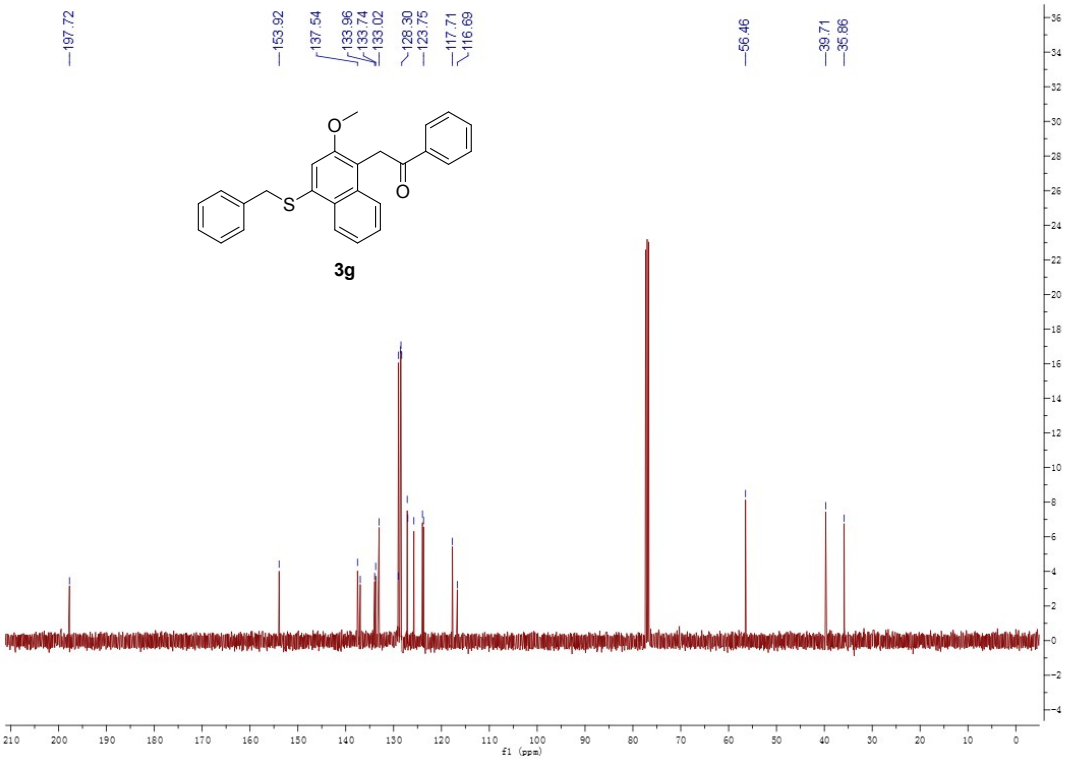
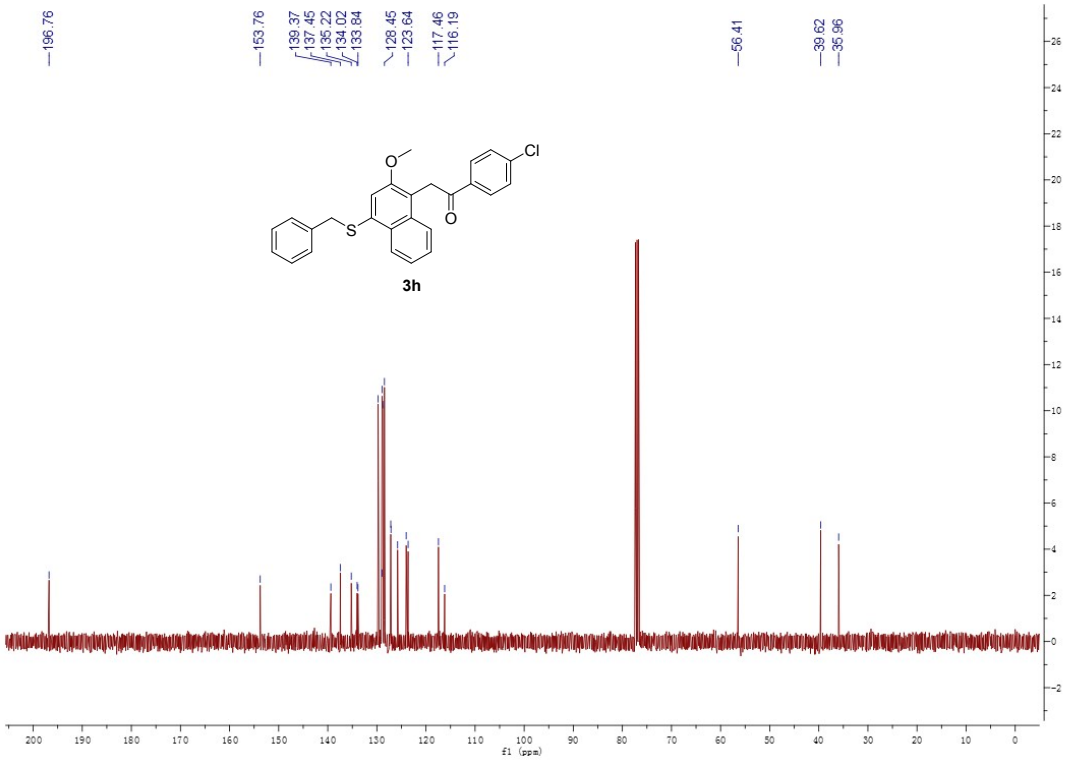
35.51
16.79
O
O
S
Cl
f1 (ppm)

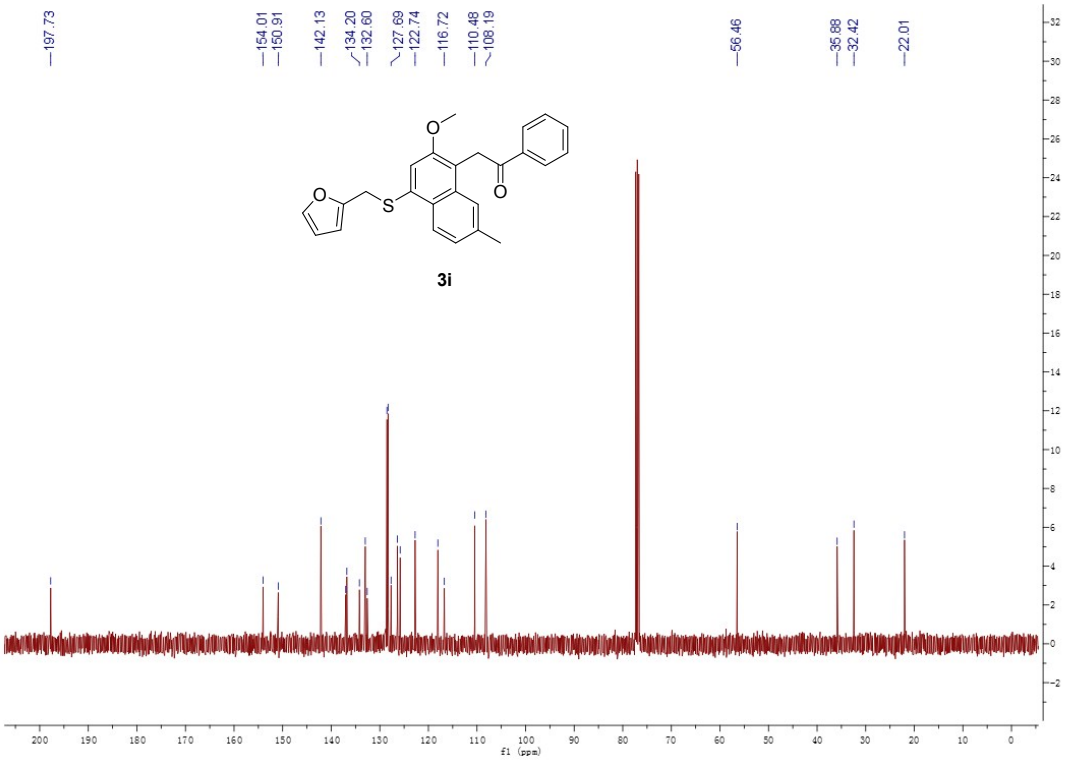
3f


197.72
153.92
137.54
133.96
133.74
133.02
128.30
123.75
117.71
116.69
56.46
39.71
35.86
O
S
O
f1 (ppm)

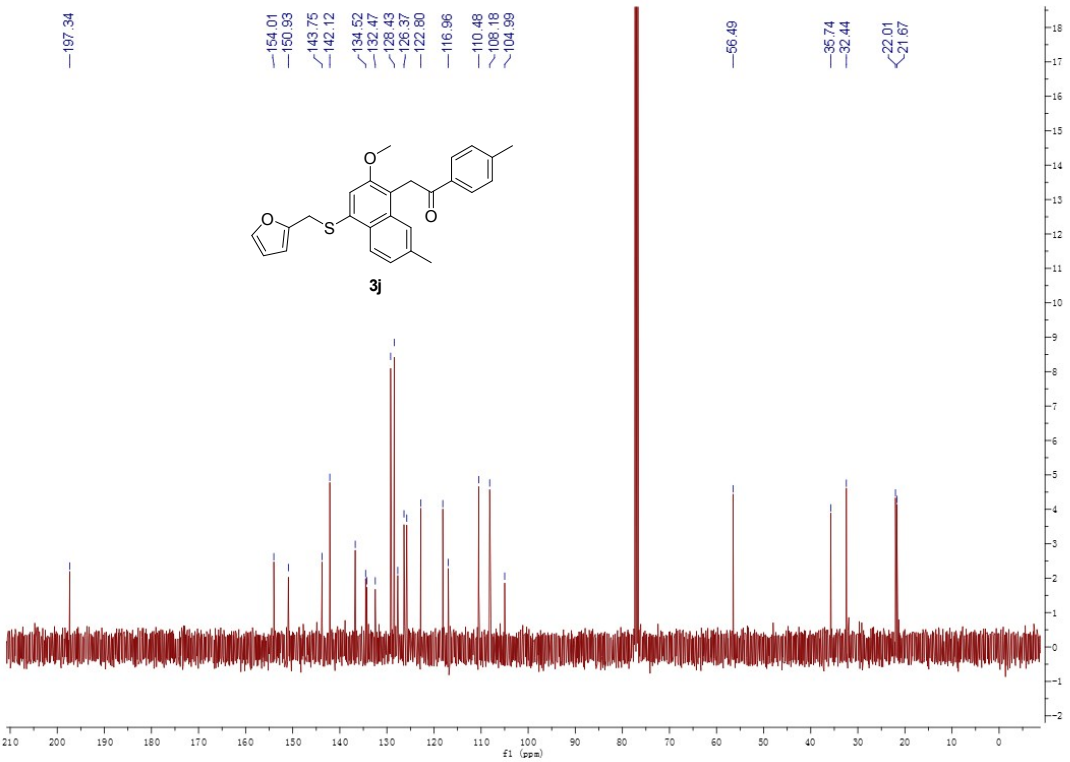
3g


196.76
153.76
139.37
137.45
135.22
134.02
133.84
128.45
123.64
117.46
116.19
56.41
39.62
35.96
O
Cl
S
O
f1 (ppm)

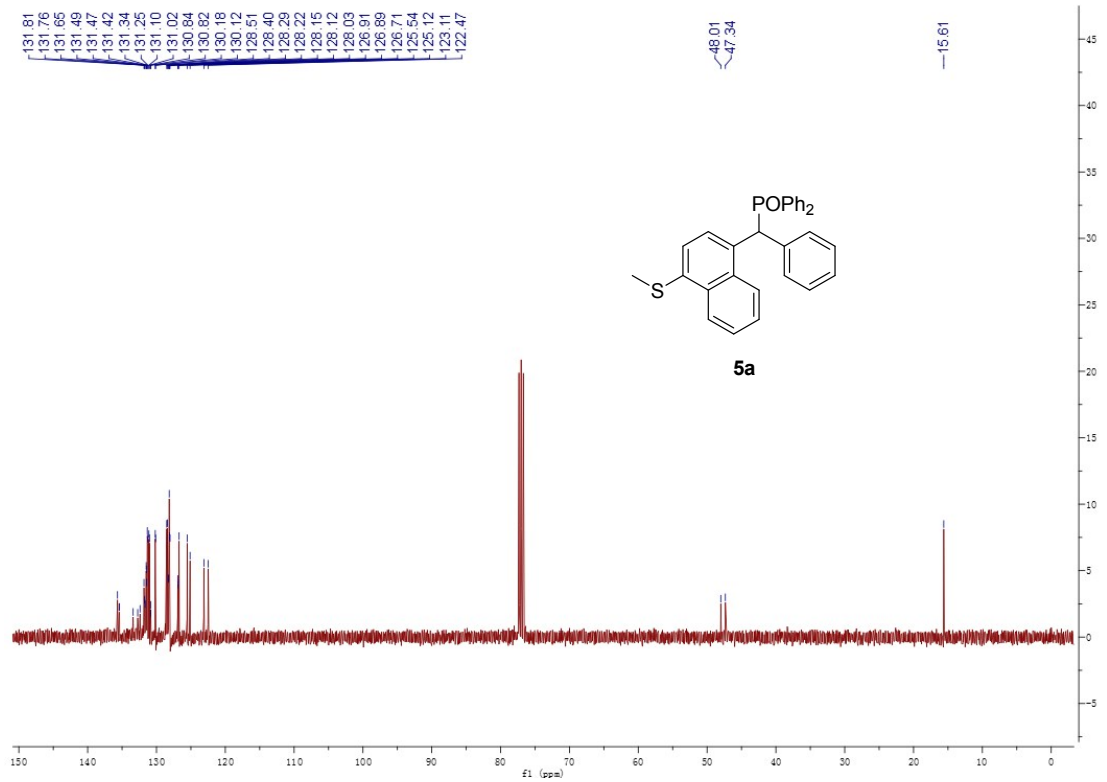
3h


197.73
154.01
150.91
142.13
134.20
132.60
127.69
122.74
116.72
110.48
108.19
56.46
35.88
32.42
22.01
O
O
S
O
f1 (ppm)

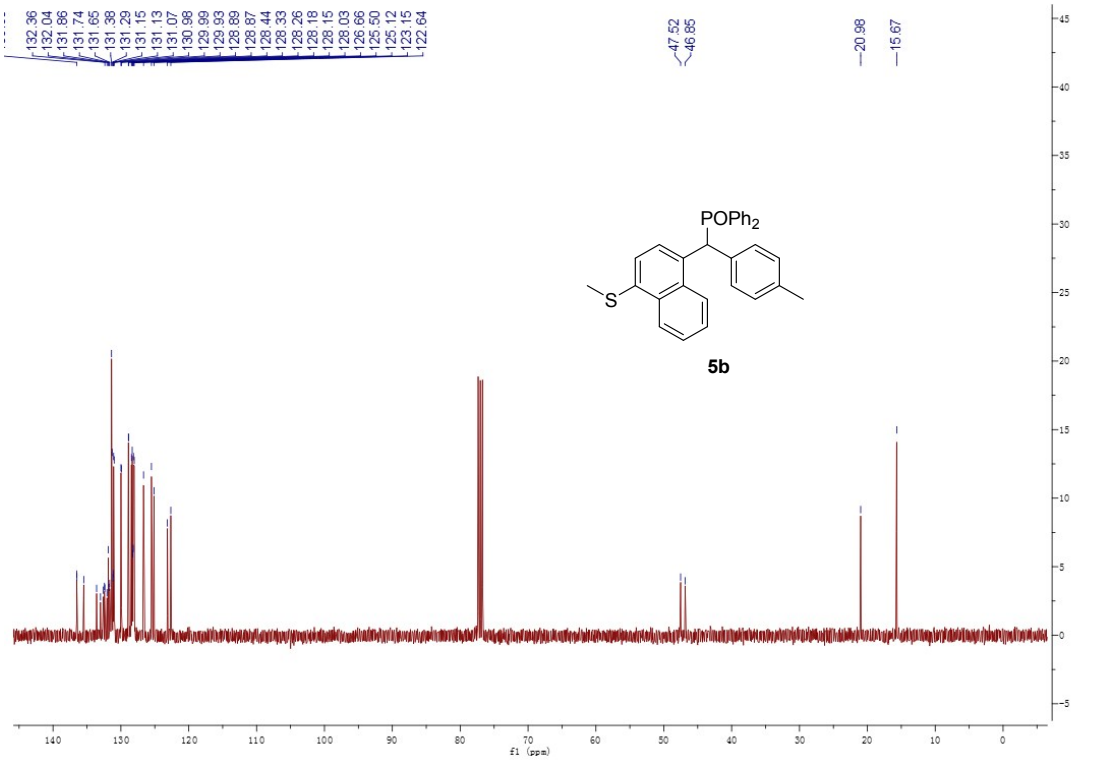
3i


197.34
154.01
150.93
143.75
142.12
134.52
132.47
128.43
126.37
122.80
116.96
110.48
108.18
104.99
56.49
35.74
32.44
22.01
21.67
O
O
S
O
f1 (ppm)

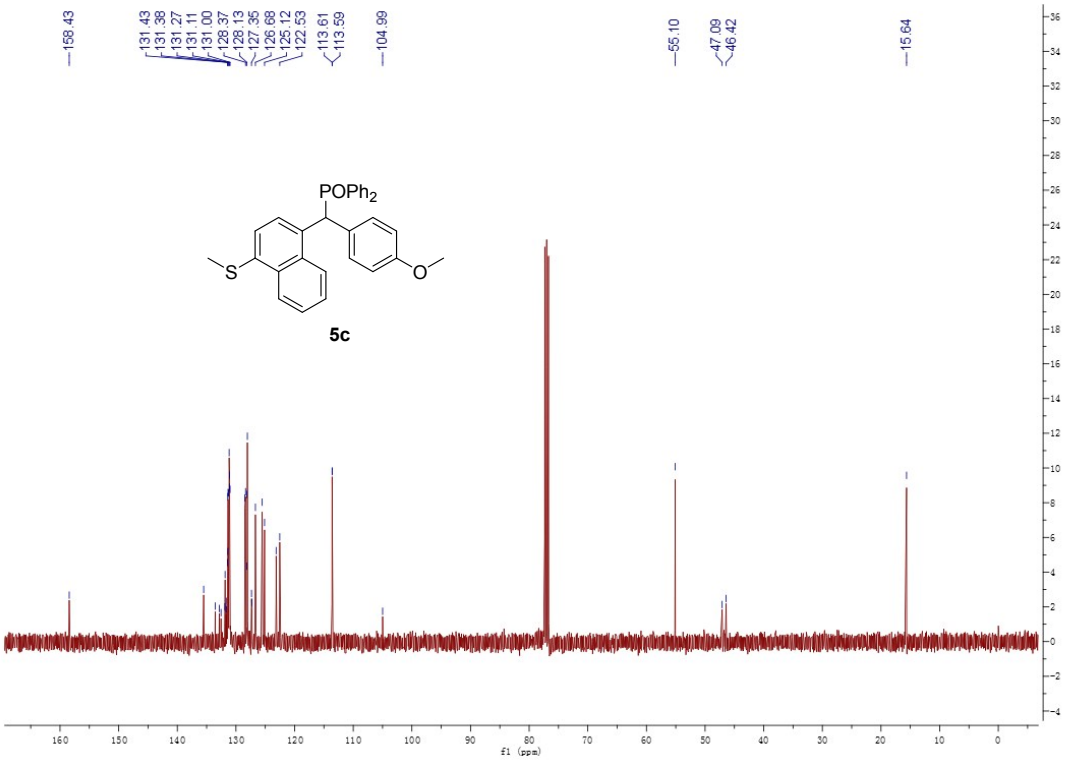
3j


131.81
131.76
131.65
131.49
131.47
131.42
131.34
131.25
131.10
131.02
130.84
130.82
130.18
130.12
128.51
128.40
128.29
128.22
128.15
128.12
128.03
126.91
126.89
126.71
125.54
125.12
123.11
122.47
48.01
47.34
15.61
POPh2
S
f1 (ppm)

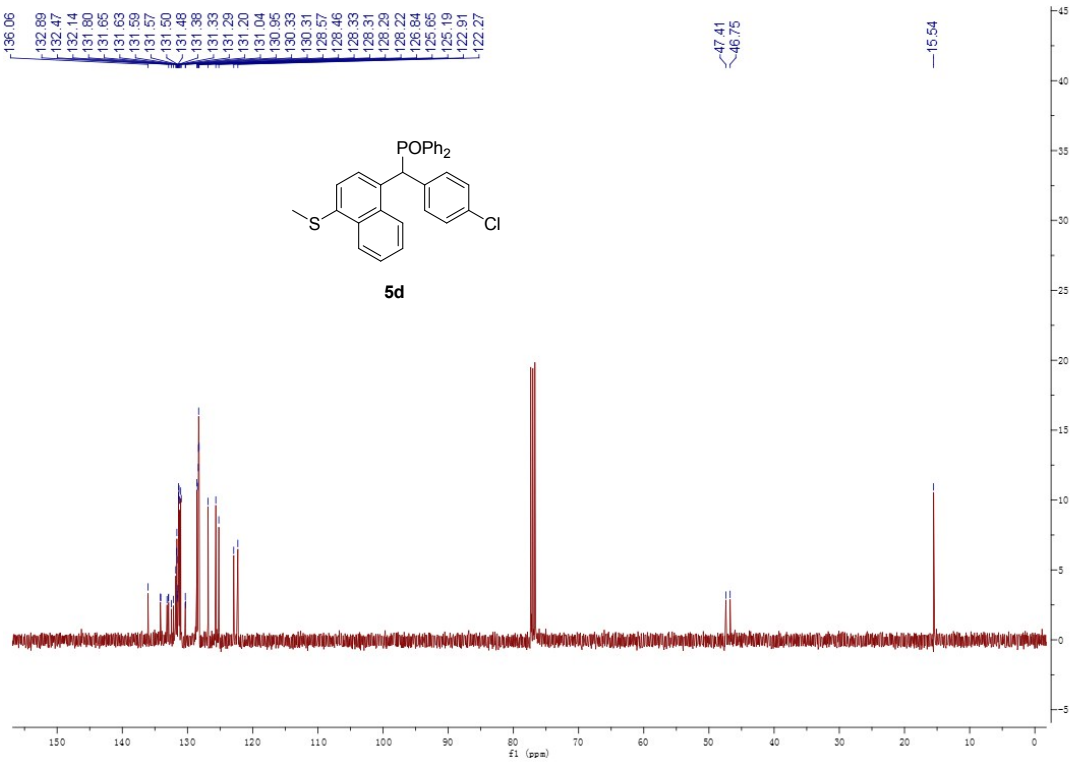
5a


132.36
132.04
131.86
131.74
131.65
131.38
131.29
131.15
131.13
131.07
130.98
129.99
129.93
128.89
128.87
128.44
128.33
128.26
128.18
128.15
128.03
126.66
125.50
125.12
123.15
122.64
47.52
46.85
20.98
15.67
POPh2
S
f1 (ppm)

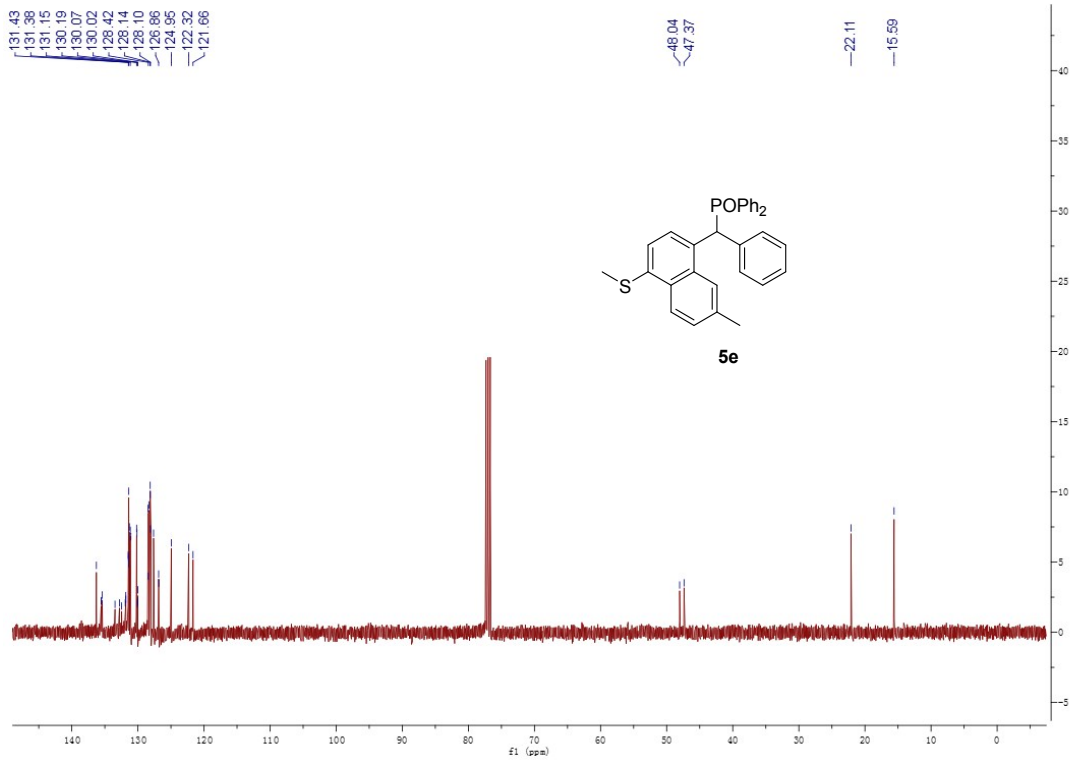
5b


158.43
131.43
131.38
131.27
131.11
131.00
128.37
128.13
127.35
126.68
125.12
122.53
113.61
113.59
104.99
55.10
47.09
46.42
15.64
POPh2
S
O
f1 (ppm)

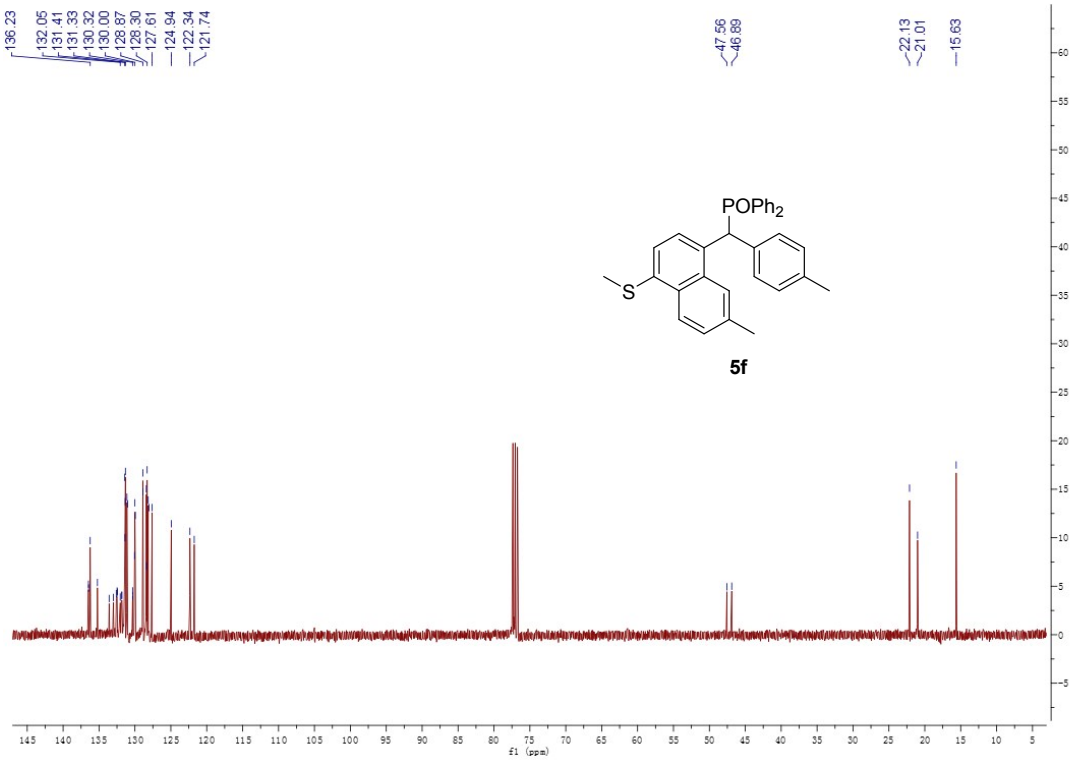
5c


136.06
132.89
132.47
132.14
131.80
131.65
131.63
131.59
131.57
131.50
131.48
131.38
131.33
131.29
131.20
131.04
130.95
130.33
130.31
128.57
128.46
128.33
128.31
128.29
128.22
126.84
125.65
125.19
122.91
122.27
47.41
46.75
15.54
POPh2
S
Cl
f1 (ppm)

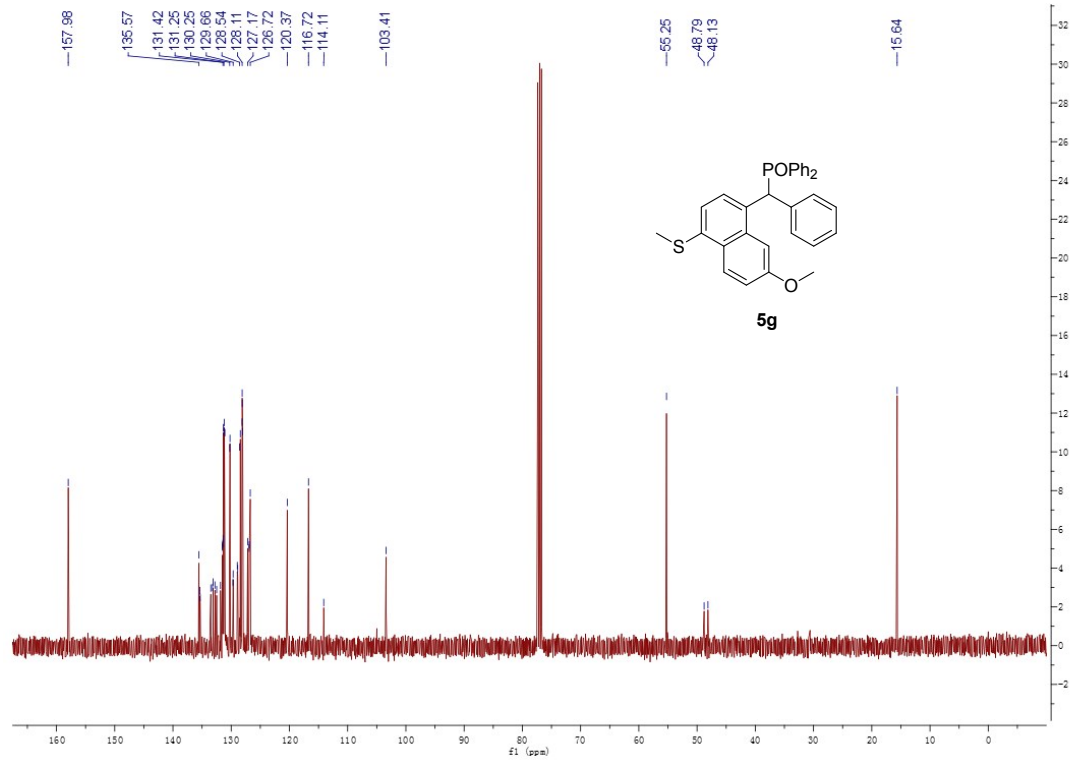
5d


131.43
131.38
131.15
130.19
130.07
130.02
128.42
128.14
128.10
126.86
124.95
122.32
121.66
48.04
47.37
22.11
15.59
POPh2
S
f1 (ppm)

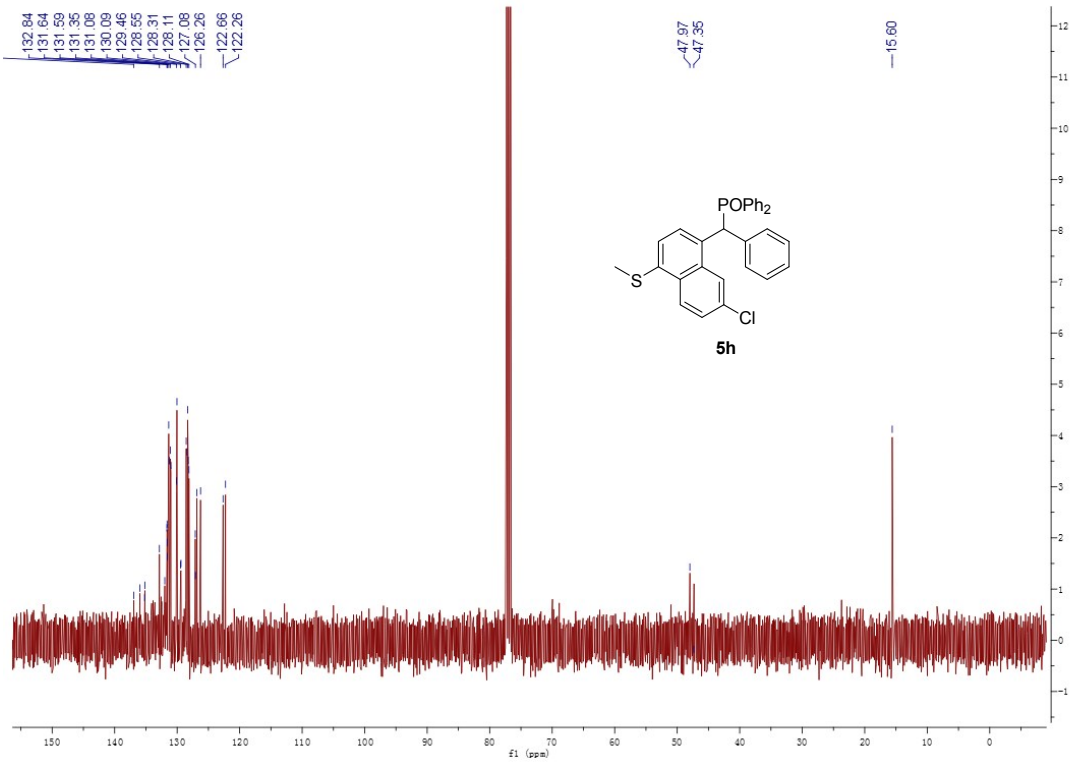
5e


136.23
132.05
131.41
131.33
130.32
130.00
128.87
128.30
127.61
124.94
122.34
121.74
47.56
46.89
22.13
21.01
15.63
POPh2
S
f1 (ppm)

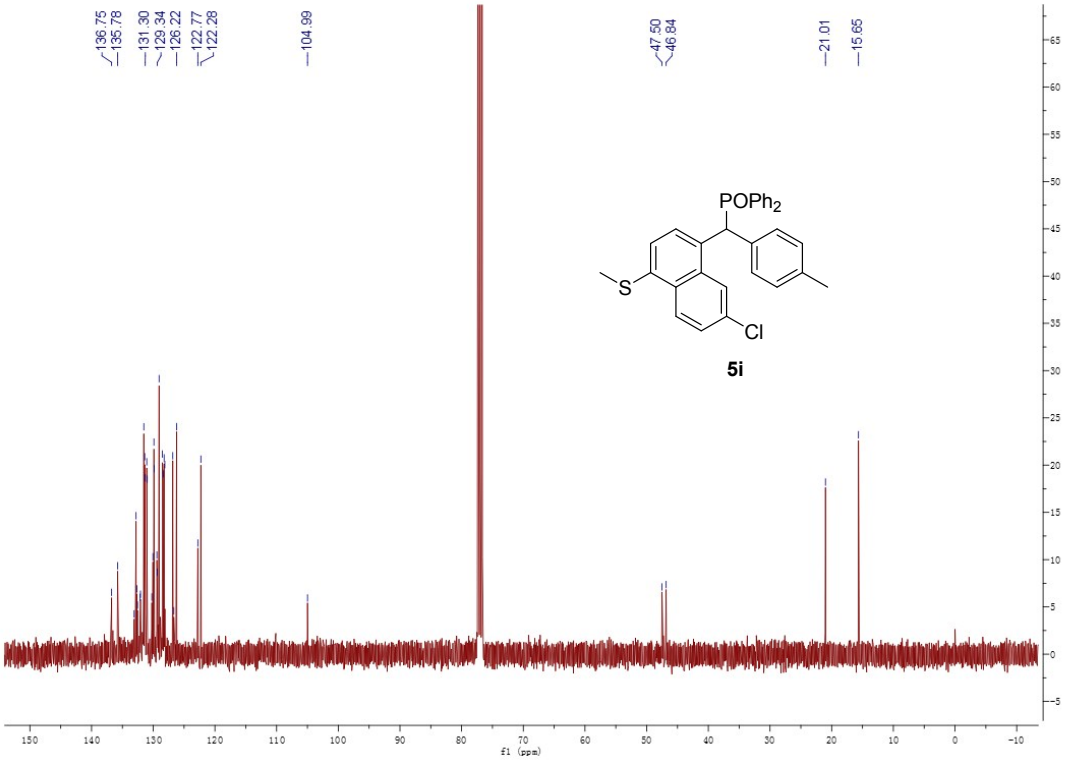
5f


157.98
135.57
131.42
131.25
130.25
129.66
128.54
128.11
127.17
126.72
120.37
116.72
114.11
103.41
55.25
48.79
48.13
15.64
POPh2
S
O
f1 (ppm)

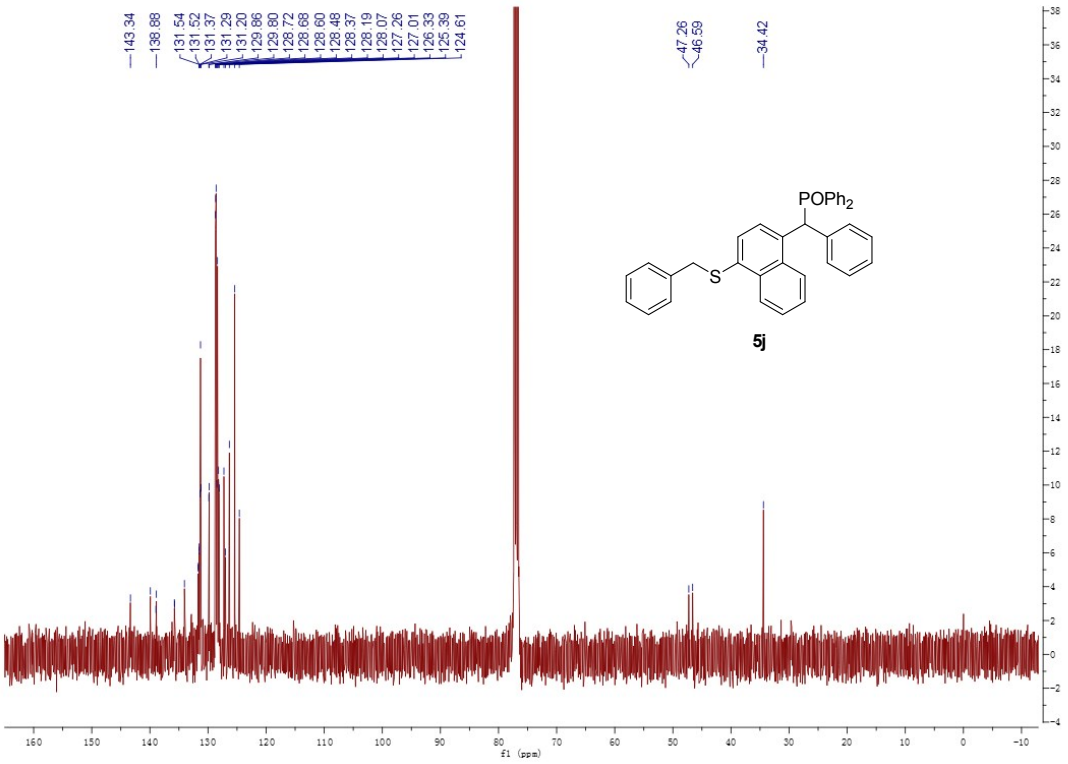
5g


132.84
131.64
131.59
131.35
131.08
130.09
129.46
128.55
128.31
128.11
127.08
126.26
122.66
122.26
47.97
47.35
15.60
POPh2
S
Cl
f1 (ppm)

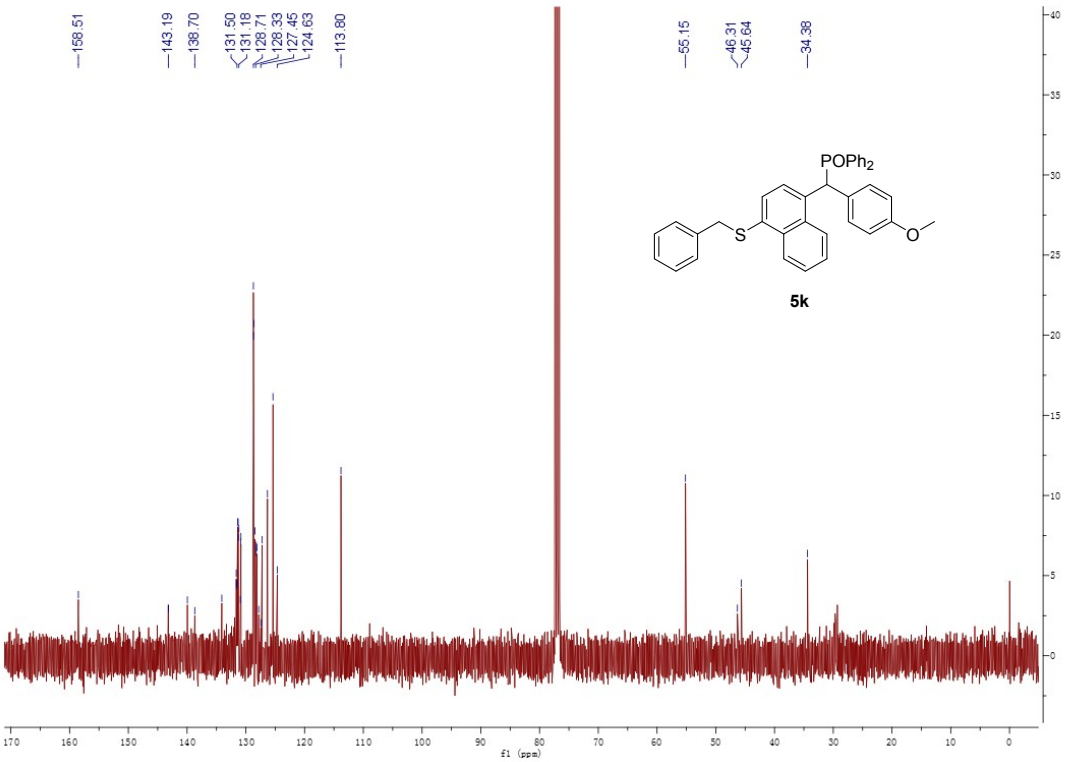
5h


136.75
135.78
131.30
129.34
126.22
122.77
122.28
104.99
47.50
46.84
21.01
15.65
POPh2
S
Cl
f1 (ppm)

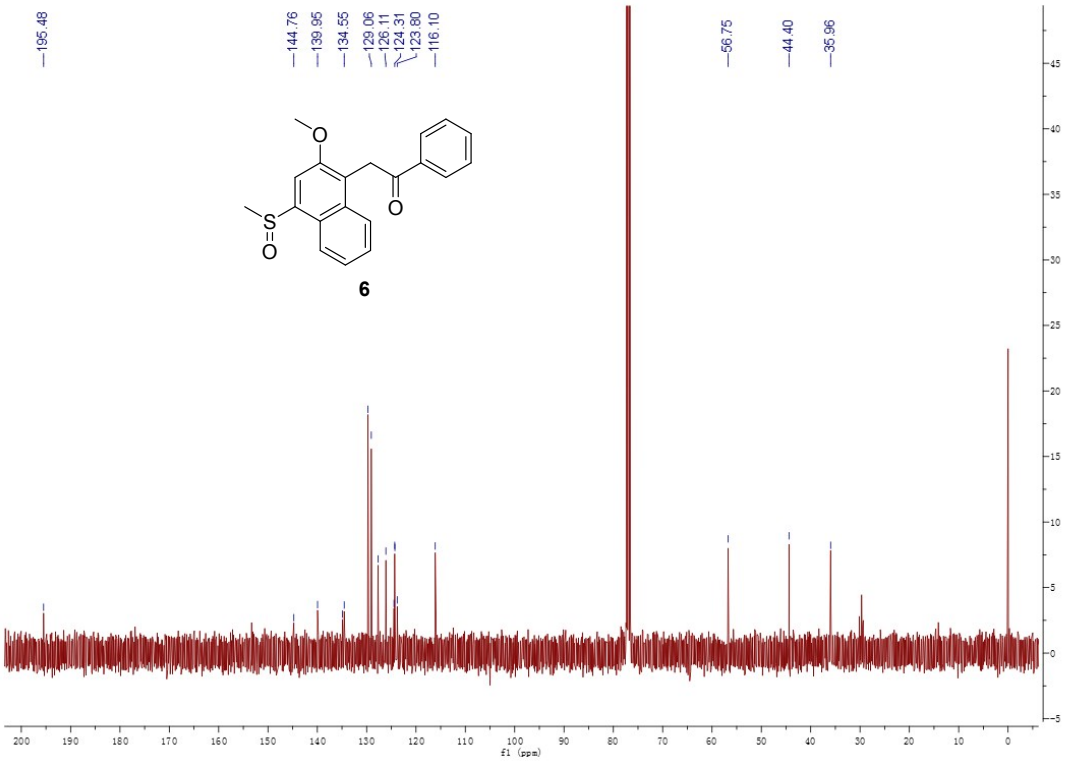
5i


143.34
138.88
131.54
131.52
131.37
131.29
131.20
129.86
129.80
128.72
128.68
128.60
128.48
128.37
128.19
128.07
127.26
127.01
126.33
125.39
124.61
47.26
46.59
34.42
POPh2
S
f1 (ppm)

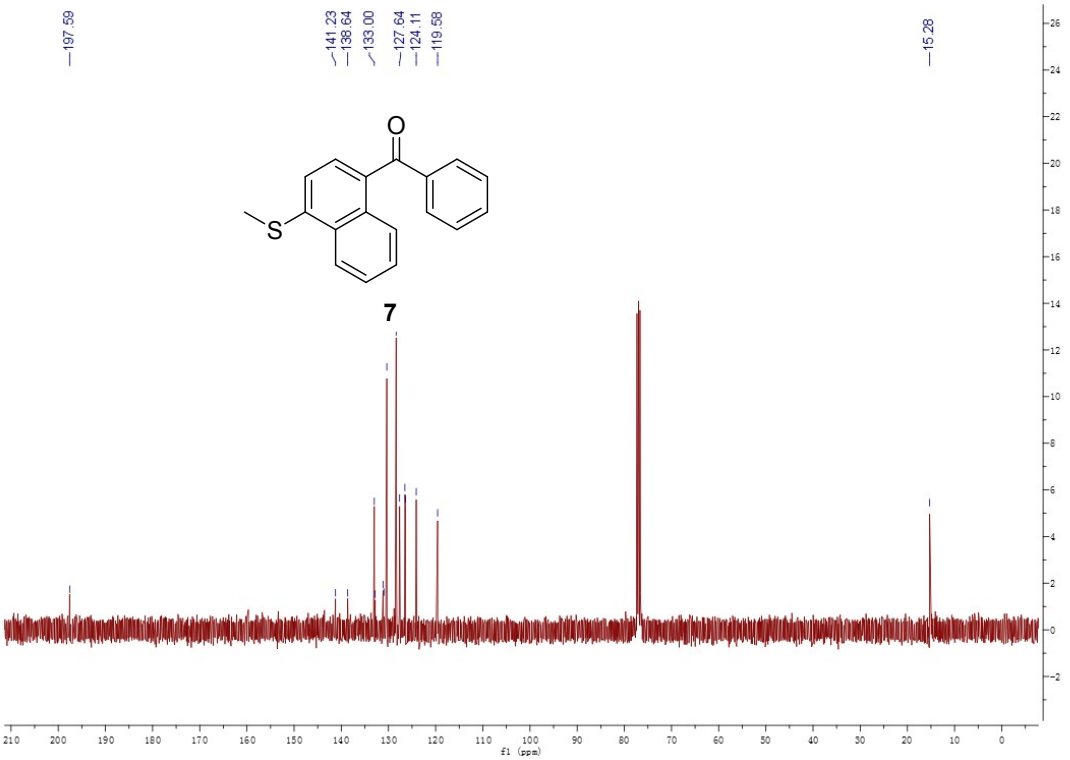
5j


158.51
143.19
138.70
131.50
131.18
128.71
128.33
127.45
124.63
113.80
55.15
46.31
45.64
34.38
POPh2
S
O
f1 (ppm)

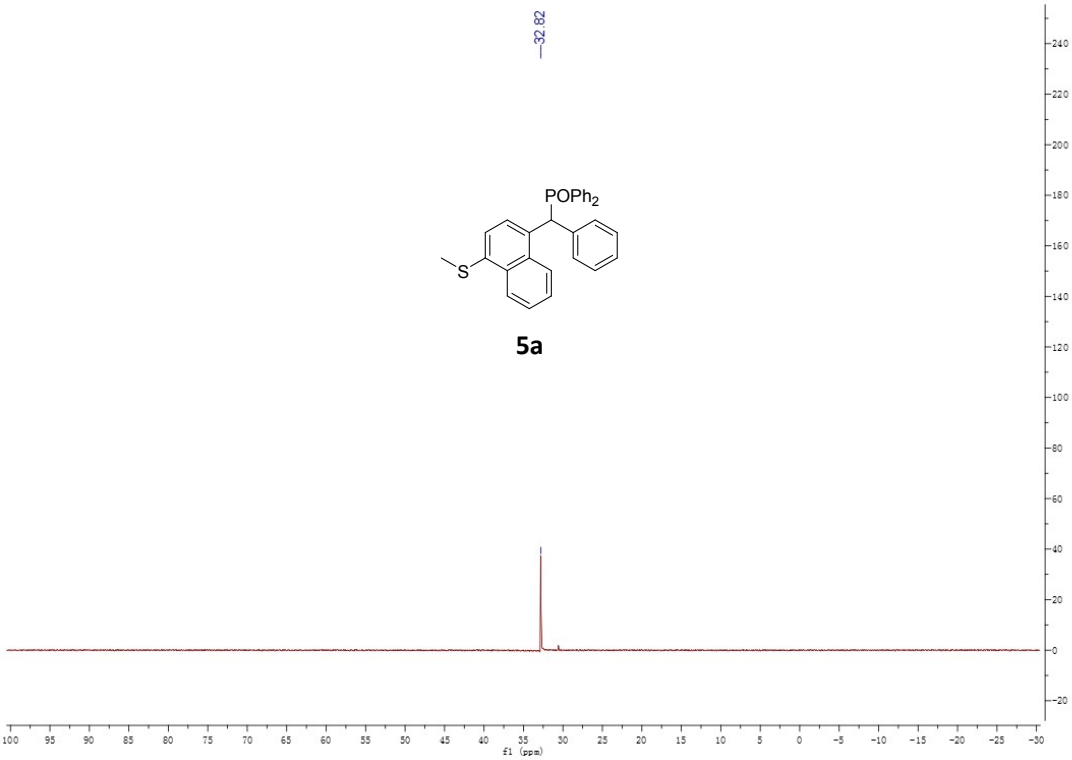
5k


195.48
144.76
139.95
134.55
129.06
126.11
124.31
123.80
116.10
56.75
44.40
35.96
O
O
S
O=
f1 (ppm)

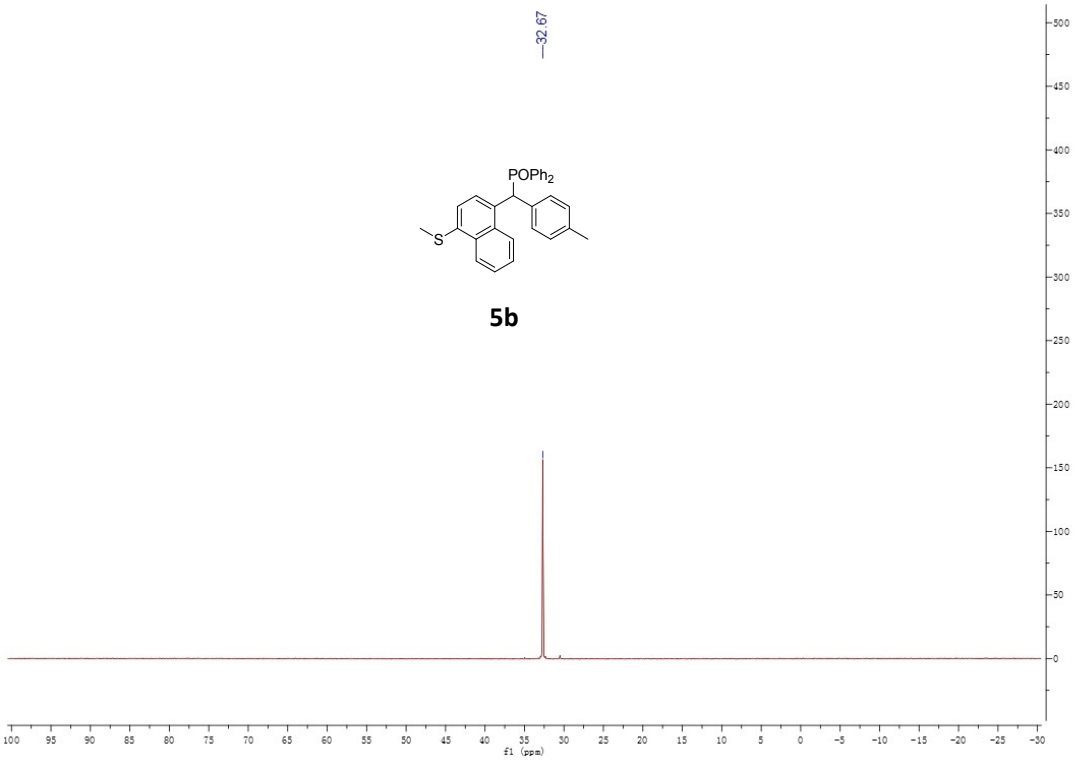
6


197.59
141.23
138.64
133.00
127.64
124.11
119.58
15.28
O
S
f1 (ppm)

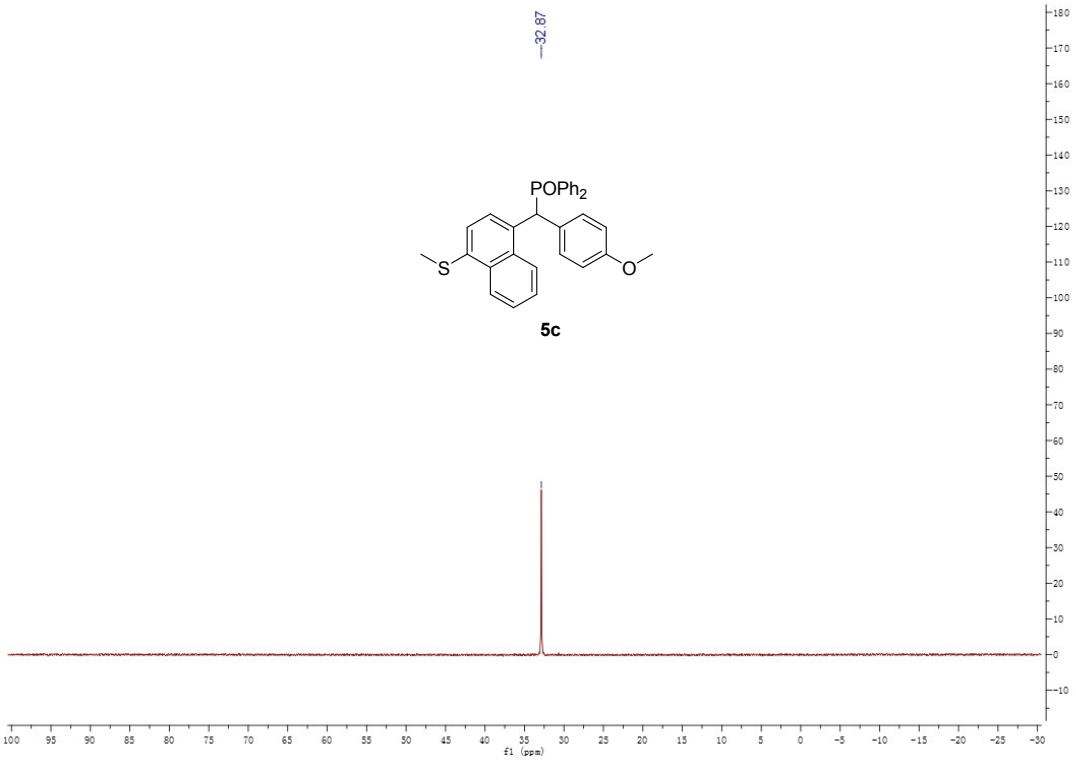
7


32.82
POPh2
S
f1 (ppm)

5a

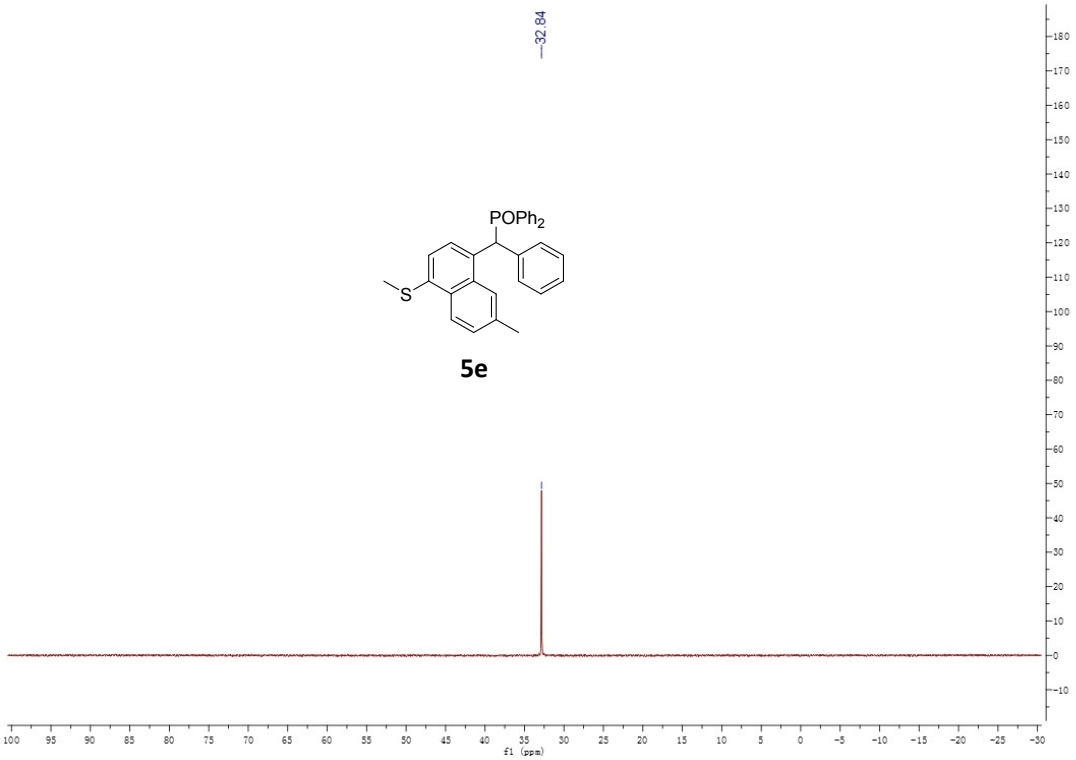


5b

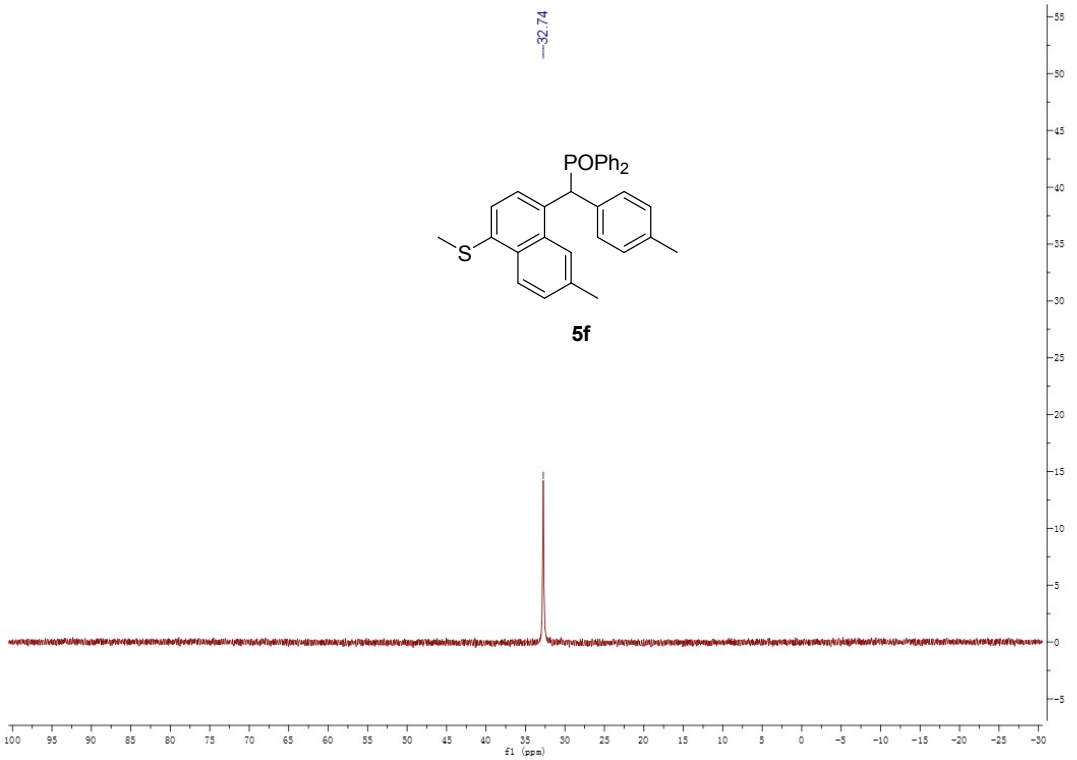


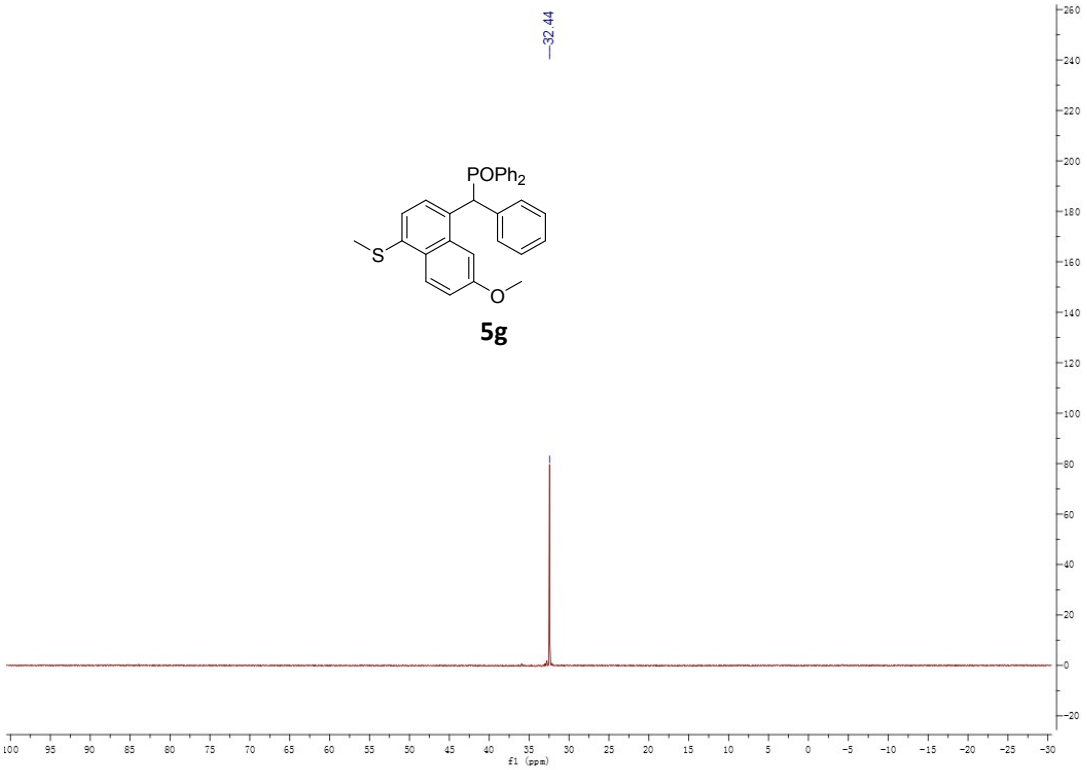
5c

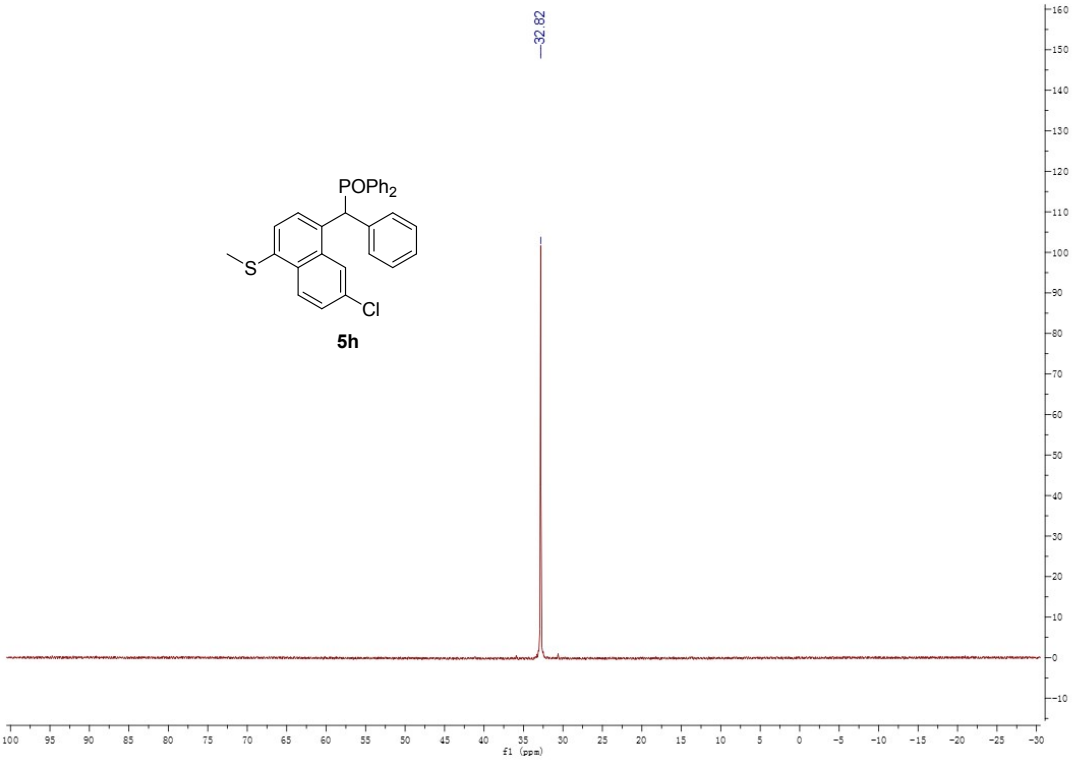
5d



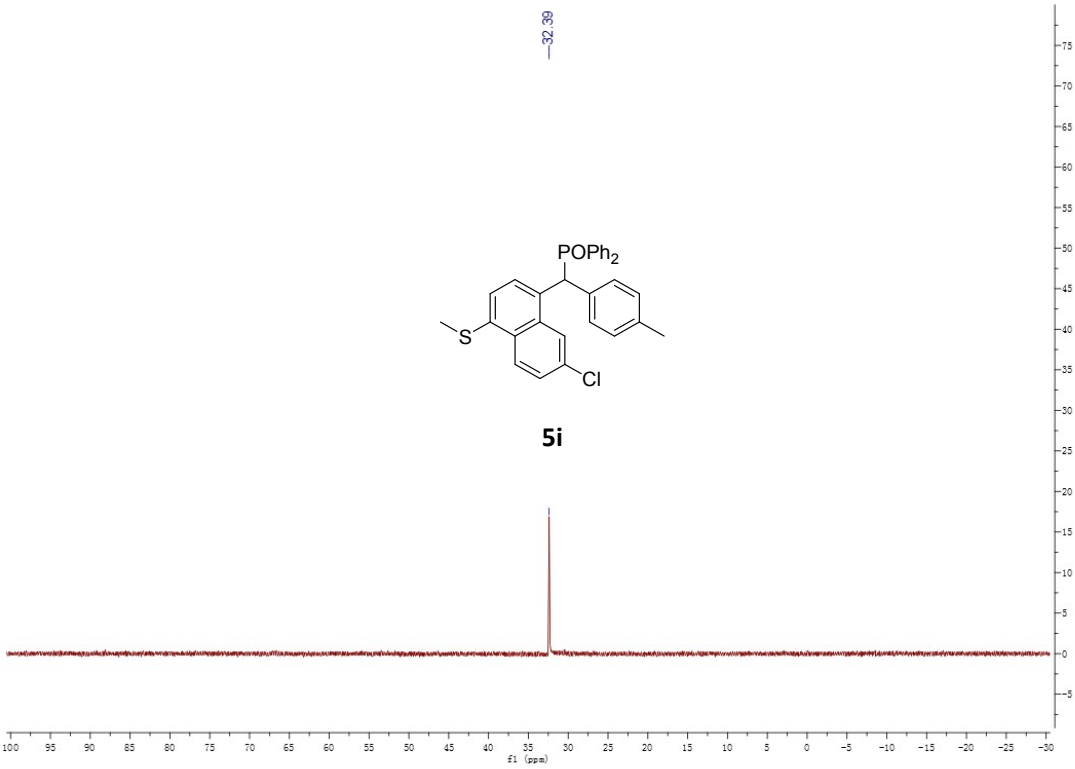
5e


32.74
POPh2
S
5f
f1 (ppm)

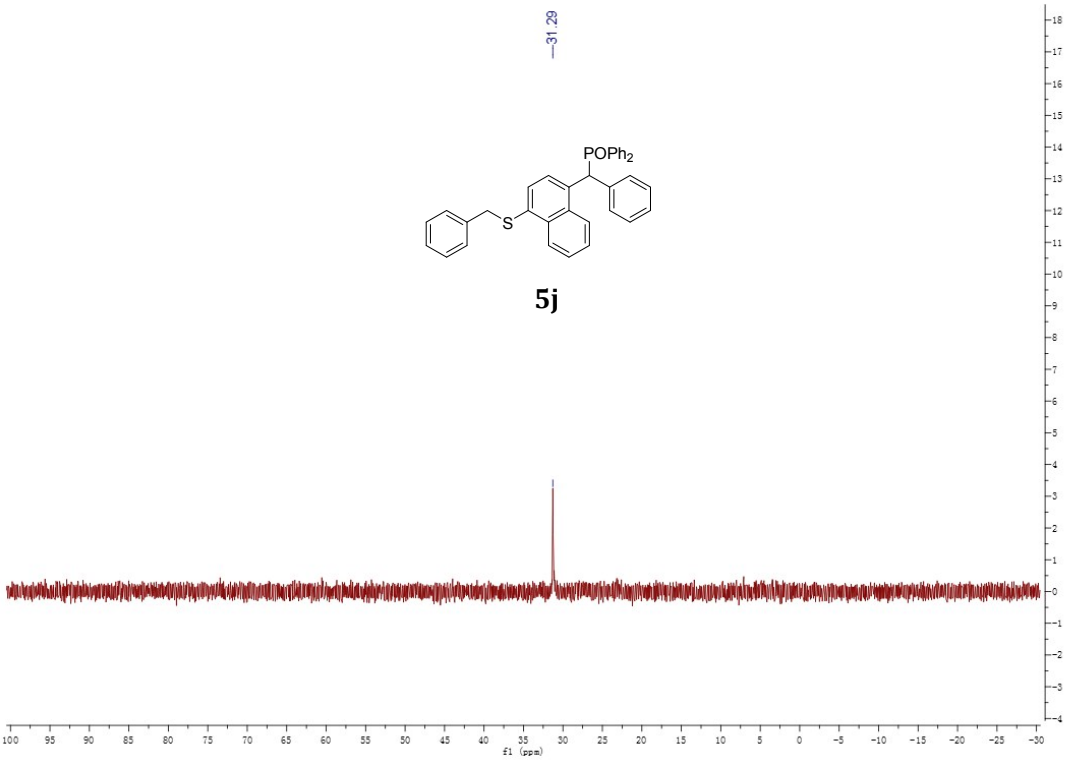

32.44
POPh2
S
O
5g
f1 (ppm)



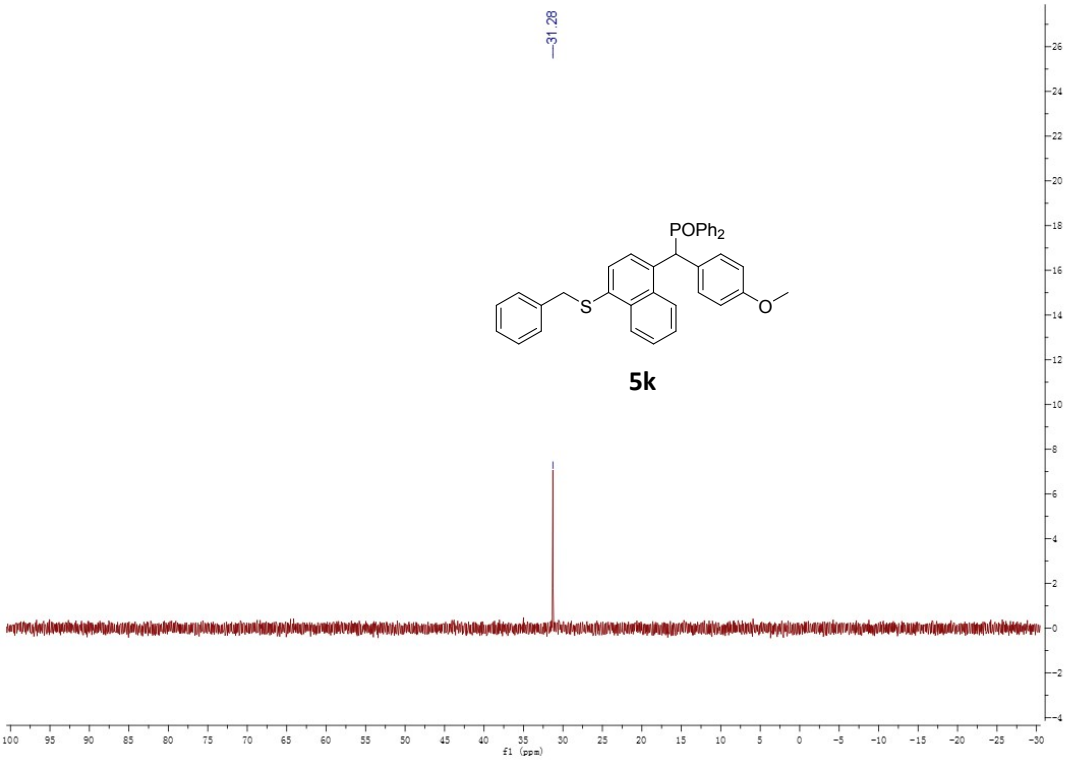
5h



5i


31.29
POPh2
S
100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30
f1 (ppm)

5j


31.28
POPh2
S
O
100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30
f1 (ppm)

5k