Supporting Information

Manganese-promoted reductive cross-coupling of disulfides with dialkyl carbonates

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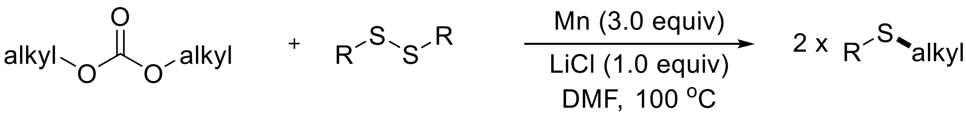
1. General remarks

^1H NMR, ^{13}C NMR data were obtained on AVANCE III Bruker 500 MHz nuclear resonance spectrometers unless otherwise noted. Chemical shifts (in ppm) were referenced to tetramethylsilane (TMS) (δ = 0.00 ppm) in $CDCl_3$ or dimethyl sulfoxide (δ = 2.50 ppm) in DMSO-d_6 as an internal standard. The data of ^1H NMR was reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet and br = broad), coupling constant (J values) in Hz and integration. ^{13}C NMR spectra were obtained by the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.0 ppm) or DMSO-d_6 (δ = 39.50 ppm). Flash chromatography was performed using 300-400 mesh silica gel with the indicated eluent according to standard techniques. Carbonate ester was purchased from Energy Chemical or prepared following our previously published procedures.[1] Analytical thin-layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel plates. Analysis of crude reaction mixture was done on an Agilent 7890 GC System with an Agilent 5975 Mass Selective Detector. Visualization of the developed chromatogram was performed by UV absorbance (254 nm) unless otherwise noted. High-resolution mass spectral (HRMS) data were recorded on Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer using electrospray ionization (ESI) mode.

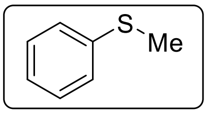
2. General procedure



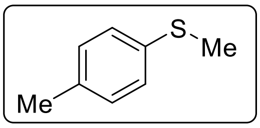
1 **2** **3**

To a 10 mL Schlenk tube was added sequentially disulfide **2** (0.2 mmol), Mn power (32.9 mg, 0.6 mmol) and LiCl (8.8 mg, 0.2 mmol). After the Schlenk tube was filled with nitrogen, carbonate ester **1** (0.4 mmol) and DMF (0.5 mL) were added via syringe. The resulting mixture was stirred at 100 °C for 12 h under N_2. After the reaction was completed, H_2O (5 mL) was added into the reaction mixture and extracted with ethyl acetate (5 mL x 3). The organic layer was dried, filtered, and concentrated. The residue was purified by column chromatography to afford product **3**.

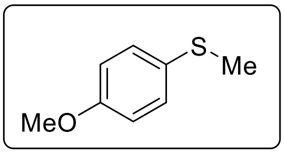
3. Characterization data



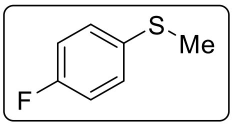
Methyl(phenyl)sulfane (3aa).[2] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3aa** (44.1 mg, 91%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.27 – 7.23 (m, 4H), 7.12 – 7.09 (m, 1H), 2.44 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 138.8, 128.7, 126.6, 124.9, 15.7.



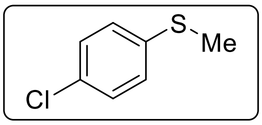
Methyl(*p*-tolyl)sulfane (3ba).[2] The representative procedure was followed using 1,2-di-*p*-tolyldisulfane **(1b)** (49.3 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ba** (49.7 mg, 90%) as a yellow liquid. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.17 (d, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 2.44 (s, 3H), 2.30 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 135.0, 134.7, 129.5, 127.3, 20.9, 16.8.



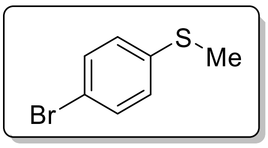
(4-Methoxyphenyl)(methyl)sulfane (3ca).[3] The representative procedure was followed using 1,2-bis(4-methoxyphenyl)disulfane **(1c)** (55.7 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 20 : 1) yielded **3ca** (48.1 mg, 78%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.20 (d, *J* = 10.0 Hz, 2H), 6.78 (d, *J* = 10.0 Hz, 2H), 3.72 (s, 3H), 2.37 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 158.2, 130.2, 128.7, 114.6, 55.3, 18.2.



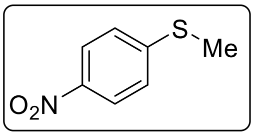
(4-Fluorophenyl)(methyl)sulfane (3da).[2] The representative procedure was followed using 1,2-bis(4-fluorophenyl)disulfane **(1d)** (50.9 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3da** (38.6 mg, 68%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.17 – 7.15 (m, 2H), 6.92 – 6.89 (m, 2H), 2.37 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 162.1 (d, *J* = 243.7 Hz), 133.2 (d, *J* = 3.8 Hz), 129.2 (d, *J* = 7.5 Hz), 115.8 (d, *J* = 22.5 Hz), 17.1; **^{19}F NMR (471 MHz, $CDCl_3$)** ***δ*** **-**117.3.



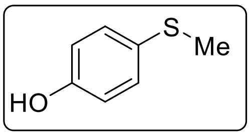
(4-Chlorophenyl)(methyl)sulfane (3ea).[2] The representative procedure was followed using 1,2-bis(4-chlorophenyl)disulfane **(1e)** (57.4 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ea** (42.3 mg, 67%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.25 (d, *J* = 10.0 Hz, 2H), 7.18 (d, *J* = 10.0 Hz, 2H), 2.47 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 132.7, 130.9, 128.9, 127.9, 16.1.



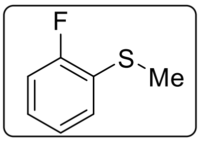
(4-Bromophenyl)(methyl)sulfane (3fa).[4] The representative procedure was followed using 1,2-bis(4-bromophenyl)disulfane **(1f)** (74.8 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3fa** (57.0 mg, 71%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.38 (d, *J* = 4.2 Hz, 2H), 7.10 (d, *J* = 4.2 Hz, 2H), 2.44 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 137.7, 131.7, 128.1, 118.6, 15.9.



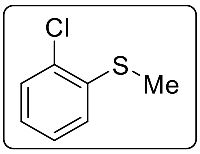
Methyl(4-nitrophenyl)sulfane (3ga).[2] The representative procedure was followed using 1,2-bis(4-nitrophenyl)disulfane **(1g)** (61. 7 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 50 : 1) yielded **3ga** (50.1 mg, 74%) as a yellow soild. **^{1}H NMR (500 MHz, CDCl$_3$) *δ*** 8.06 (d, *J* = 10.0 Hz, 2H), 7.22 (d, *J* = 10.0 Hz, 2H), 2.48 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$) *δ*** 148.8, 144.8, 125.0, 123.9, 14.9.



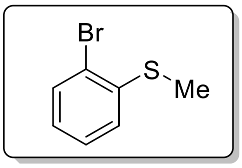
4-(Methylthio)phenol (3ha).[5] The representative procedure was followed using 4,4'-disulfanediyldiphenol **(1h)** (50.1 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 1 : 1) yielded **3ha** (40.3 mg, 71%) as a white solid. **^{1}H NMR (500 MHz, CDCl$_3$) *δ*** 7.22 (d, *J* = 10.0 Hz, 2H), 6.78 (d, *J* = 10.0 Hz, 2H), 2.44 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$) *δ*** 154.1, 130.4, 128.8, 116.0, 18.0. Melting point: 85 °C.



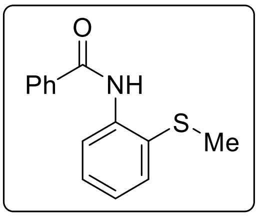
(2-Fluorophenyl)(methyl)sulfane (3ia).[2] The representative procedure was followed using 1,2-bis(2-fluorophenyl)disulfane **(1i)** (50.9 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ia** (40.3 mg, 71%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$) *δ*** 7.22 – 6.18 (m, 1H), 7.10 – 7.06 (m, 1H), 7.06 – 7.01 (m, 1H), 7.04 – 7.02 (m, 1H), 2.39 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$) *δ*** 161.4 (d, *J* = 242.5 Hz), 128.8 (d, *J* = 2.5 Hz), 127.3 (d, *J* = 7.5 Hz), 125.4 (d, *J* = 16.2 Hz), 124.4 (d, *J* = 3.8 Hz), 115.3 (d, *J* = 21.2 Hz), 15.6 (d, *J* = 2.5 Hz); **^{19}F NMR (471 MHz, CDCl$_3$) *δ*** -111.31.



(2-Chlorophenyl)(methyl)sulfane (3ja).[2] The representative procedure was followed using 1,2-bis(2-chlorophenyl) disulfane **(1j)** (57.4 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ja** (55.0 mg, 87%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 7.28 – 7.26 (m, 1H), 7.19 – 7.15 (m, 1H), 7.10 – 7.08 (m, 1H), 7.02 – 6.99 (m, 1H), 2.40 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 142.1, 137.7, 131.8, 129.4, 127.2, 125.5, 15.2.

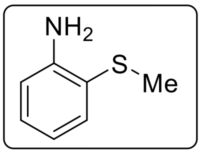


(2-Bromophenyl)(methyl)sulfane (3ka).[4] The representative procedure was followed using 1,2-bis(2-bromophenyl)disulfane **(1k)** (74.8 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ka** (49.3 mg, 61%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 7.42 (d, *J* = 8.0 Hz, 1H), 7.19 (t, *J* =7.5 Hz, 1H), 7.03 (d, *J* =8.0 Hz, 1H), 6.89 (t, *J* =7.5 Hz, 1H), 2.56 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 139.7, 132.7, 127.8, 125.7, 125.5, 121.8, 15.8.

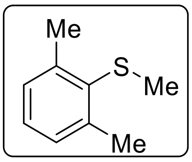


N*-[2-(Methylthio)phenyl]benzamide (3la)**.[6] The representative procedure was followed using *N*,*N'*-[disulfanediylbis(2,1-phenylene)]dibenzamide **(1l)** (91.3 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 20 : 1) yielded **3la** (45.7 mg, 47%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ 9.11 (br, 1H), 8.40 (d, *J* = 10.0 Hz, 2H), 7.84 (d, *J* =

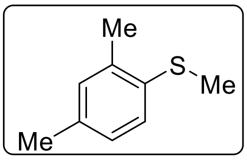
5.0 Hz, 2H), 7.44 – 7.36 (m, 4 H), 7.24 – 7.20 (m, 1 H), 6.99 – 6.96 (m, 1 H), 2.26 (s, 3H); **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 162.9, 136.4, 132.7, 131.0, 129.8, 126.9, 126.7, 125.9, 123.5, 122.3, 118.4, 18.9. HRMS (ESI) m/z ([M+ H]+) Calcd. for $C_{14}H_{13}NOS$ 244.0791, found: 244.0790.



2-(Methylthio)aniline (3ma).[7] The representative procedure was followed using 2 2,2'-disulfanediyldianiline **(1m)** (49.7 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 10 : 1) yielded **3ma** (29.5 mg, 53%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl3)** ***δ*** 7.27 – 7.25 (m, 1H), 7.01 – 6.98 (m, 1H), 6.64 – 6.61 (m, 2H), 4.17 (br, 2H), 2.26 (s, 3H); **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 147.1, 133.4, 128.9,120.3, 118.8, 114.9, 17.7. HRMS (ESI) m/z ([M+ H]+) Calcd. for C_7H_9NS 140.0529, found: 140.0528.

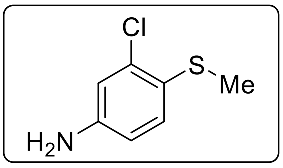


(2,6-Dimethylphenyl)(methyl)sulfane (3na).[2] The representative procedure was followed using 1,2-bis(2,6-dimethylphenyl)disulfane **(1n)** (54.9 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3na** (38.3 mg, 63%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl3)** ***δ*** 7.09 (s, 2H), 2.55 (s, 6H), 2.22 (s, 3H); **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 142.7, 135.2, 128.1, 128.0.

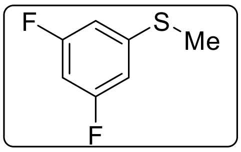


(2,4-Dimethylphenyl)(methyl)sulfane (3oa).[2] The representative procedure was followed using 1,2-bis(2,4-dimethylphenyl)disulfane **(1o)** (54.9 mg, 0.2 mmol) and

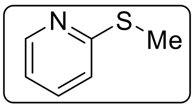
DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3oa** (45.0 mg, 74%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.10 (d, *J* =10.0 Hz, 1H), 6.91 (d, *J* =10.0 Hz, 2H), 2.35 (s, 3H), 2.44 (s, 3H) 2.31 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 136.2, 134.7, 133.9, 130.8, 127.2, 125.8, 20.8, 20.0, 15.9.



3-Chloro-4-(methylthio)aniline (3pa).[8] The representative procedure was followed using 4,4'-disulfanediylbis(3-chloroaniline) **(1p)** (63.5 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 10 : 1) yielded **3pa** (49.1 mg, 71%) as a brown solid. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.16 (d, *J* = 8.0 Hz, 1H), 6.59 – 6.56 (m, 2H), 4.25 (br, 2H), 2.21 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 148.1, 134.6, 134.5, 118.5, 118.4, 114.3, 17.8. HRMS (ESI) m/z ([M+ H]+) Calcd. for C_7H_8ClNS 174.0139, found: 174.0138. Melting point: 74 ºC

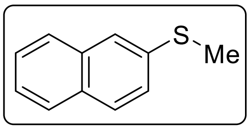


(3,5-Difluorophenyl)(methyl)sulfane (3qa).[2] The representative procedure was followed using 1,2-bis(3,5-difluorophenyl)disulfane **(1q)** (58.1 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3qa** (44.2 mg, 69%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 6.75 – 6.72 (m, 2H), 6.58 – 6.54 (m, 1H), 2.47 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 163.1 (dd, *J* = 261.3, 13.8 Hz), 143.0, 108.6 (dd, *J* = 21.3, 7.5 Hz), 100.3 (dd, *J* = 25.5, 7.0 Hz), 15.3; **^{19}F NMR (471 MHz, CDCl$_3$)** ***δ*** -109.72.

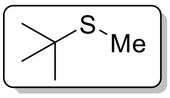


2-(Methylthio)pyridine (3ra).[3] The representative procedure was followed using 1,2-

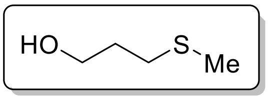
di(pyridin-2-yl)disulfane **(1r)** (44.1 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 20 : 1) yielded **3ra** (20.0 mg, 40%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 8.36 – 8.35 (m, 1H), 7.41 – 7.37 (m, 1H), 7.10 – 7.08 (m, 1H), 6.89 – 6.87 (m, 1H), 2.48 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 159.9, 149.4, 135.7, 121.4, 119.0, 13.2.



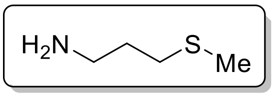
Methyl(naphthalen-2-yl)sulfane (3sa).[2] The representative procedure was followed using 1,2-di(naphthalen-2-yl)disulfane **(1s)** (63.7 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3sa** (59.2 mg, 85%) as a white solid. **^{1}H NMR (500 MHz, CDCl$_3$)** δ 7.76 – 7.75 (m, 1H), 7.72 – 7.70 (m, 2H), 7.55 (s, 1H), 7.46 – 7.43 (m, 1H), 7.40 – 7.35 (m, 2H), 2.56 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 136.1, 133.9, 131.3, 128.2, 127.8, 126.8, 126.6, 125.7, 125.3, 123.4, 15.8. Melting point: 62 ºC.



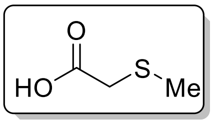
Tert*-Butyl(methyl)sulfane (3ta)**.[9] The representative procedure was followed using 1,2-di-*tert*-butyldisulfane **(1t)** (35.6 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ta** (32.0 mg, 77%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ 2.05 (s, 3H), 1.31 (s, 9H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 40.6, 30.1, 11.3.



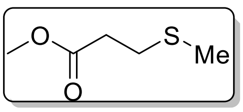
3-(Methylthio)propan-1-ol (3ua).[10] The representative procedure was followed using 3,3'-disulfanediylbis(propan-1-ol) **(1u)** (36.4 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ua** (23.4 mg, 55%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 3.75 – 3.72 (m, 2H), 2.63 – 2.60 (m, 2H), 3.52 – 2.50 (br, 1H), 2.12 (s, 3H), 1.88 – 1.83 (m, 2H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 61.4, 31.3, 30.8, 15.3.



3-(Methylthio)propan-1-amine (3va).[11] The representative procedure was followed using 3,3'-disulfanediylbis(propan-1-amine) **(1v)** (36.2 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (petroleum ether : ethyl acetate = 10 : 1) yielded **3va** (26.4 mg, 66%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 2.74 – 2.71 (m, 2H), 2.50 – 2.46 (m, 2H), 2.03 (s, 3H), 1.70 – 1.65 (m, 2H), 1.19 (br, 2H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 40.90, 32.61, 31.43, 15.3. HRMS (ESI) m/z ([M+ H]+) Calcd. for $C_4H_{11}NS$ 106.0685, found: 106.0684.

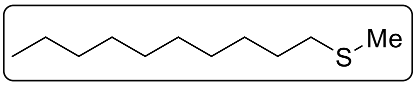


2-(Methylthio)acetic acid (3wa).[12] The representative procedure was followed using 2,2'-disulfanediyldiacetic acid **(1w)** (36.4 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (DCM : MeOH = 5 : 1) yielded **3wa** (18.2 mg, 43%) as a colorless oil; **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 10.75 (br, 1H), 3.20 (s, 2H), 2.20 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 176.5, 35.3, 16.1.

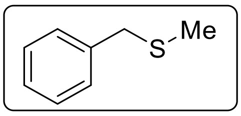


Methyl 3-(methylthio)propanoate (3xa).[13] The representative procedure was followed using dimethyl 3,3'-disulfanediyldipropionate **(1x)** (47.6 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane: ethyl acetate = 20 : 1) yielded **3xa** (38.1 mg, 43%) as a colorless oil. **^{1}H NMR (500 MHz,**

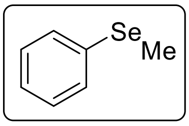
$CDCl_3$) δ 3.70 (s, 3H), 2.79 – 2.76 (m, 2H), 2.65 – 2.62 (m, 2H), 2.13 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 172.2, 51.6, 34.1, 28.9, 15.3.



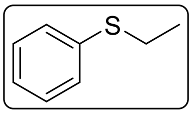
Decyl(methyl)sulfane (3ya).[14] The representative procedure was followed using 1,2-didecyldisulfane **(1y)** (69.3 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ya** (55.0 mg, 79%) as a colorless oil. **^1H NMR (500 MHz, $CDCl_3$)** δ 2.49 (t, J = 7.5 Hz, 2H), 2.10 (s, 3H),1.62 – 1.56 (m, 2H), 1.31 – 1.26 (m, 14H), 0.89 – 0.87 (m, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 34.3, 31.2, 29.6, 29.5, 29.3, 29.2, 29.1, 28.8, 22.7, 15.5, 14.1.



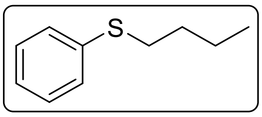
Benzyl(methyl)sulfane (3za).[15] The representative procedure was followed using 1,2-dibenzyldisulfane **(1z)** (49.3 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3za** (44.7 mg, 81%) as a colorless oil. **^1H NMR (500 MHz, $CDCl_3$)** δ 7.25 – 7.22 (m, 4H), 7.28 – 7.16 (m, 1H), 3.06 (s, 2H), 1.92 (s, 3H); **^{13}CNMR (125 MHz, $CDCl_3$)** δ 138.2, 128.8, 128.4, 126.9, 38.3, 14.9.



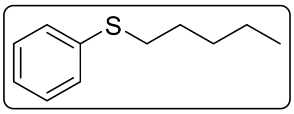
Methyl(phenyl)selane (3a'a).[16] The representative procedure was followed using 1,2-diphenyldiselane **(1a')** (30.8 mg, 0.2 mmol) and DMC **(2a)** (36.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3a'a** (62.4 mg, 51%) as a colorless oil. **^1H NMR (500 MHz, $CDCl_3$)** δ 8.38 – 8.36 (m, 1H), 7.42 – 7.39 (m, 1H), 7.12 – 7.10 (m, 1H), 6.91 – 6.89 (m, 1H), 2.50 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 149.4, 135.7, 121.5, 119.1, 13.2.



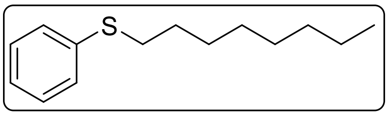
Ethyl(phenyl)sulfane (3ab).[17] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and diethyl carbonate **(2b)** (47.3 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ab** (37.5 mg, 68%) as a yellow oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.32 – 7.30 (m, 2H), 7.27 – 7.24 (m, 2H), 7.16 – 7.13 (m, 1H), 2.92 (q, *J* =7.5 Hz, 2H), 1.29 (t, *J* =7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 136.6, 128.9, 128.7, 125.6, 27.5, 14.3.



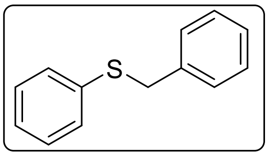
Butyl(phenyl)sulfane (3ac).[18] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and dibutyl carbonate **(2c)** (69.7 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ac** (39.8 mg, 60%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.26 – 7.24(m, 2H), 7.21 – 7.17(m, 2H), 7.10 – 7.07(m, 1H), 2.47(t, *J* = 7.5 Hz, 2H), 1.58 – 1.53 (m, 2H), 1.40 – 1.35 (m, 2H), 0.85 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 137.0, 128.8, 125.6, 33.2, 31.2, 21.9, 13.6.



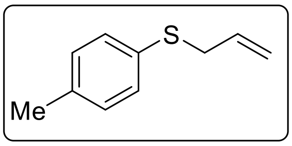
Benzyl(methyl)sulfane (3ad).[19] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and dipentyl carbonate **(2d)** (80.9 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ad** (51.1 mg, 71%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.25 – 7.17 (m, 4H), 7.09-7.06 (m, 1H), 2.84 (t, *J* = 7.5 Hz, 2H), 1.61 – 1.55 (m, 2H), 1.36 – 1.23 (m, 4H), 0.82 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 137.0, 128.8, 125.6, 33.5, 31.0, 28.8, 22.2, 13.9.



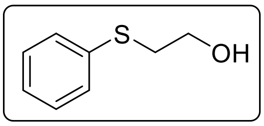
Benzyl(methyl)sulfane (3ae).[14] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and dimethyl carbonate **(2e)** (114.6 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ae** (53.3 mg, 60%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.25 – 7.23 (m, 2H), 7.21-7.17 (m, 2H), 7.09 – 7.06 (m, 1H), 2.83 (t, *J* = 7.5 Hz, 2H), 1.60 – 1.54 (m, 2H), 1.35 – 1.32 (m, 2H), 1.20 – 1.18 (m, 8H), 0.80 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 137.0, 128.8, 125.6, 33.5, 31.8, 29.2, 29.1, 28.8, 22.6, 14.1.



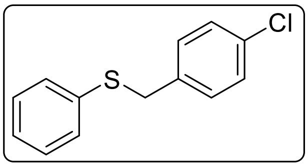
Benzyl(methyl)sulfane (3af).[20] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and dibenzyl carbonate **(2f)** (96.9 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3af** (57.6 mg, 72%) as a white solid. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.28 – 7.18 (m, 9H), 7.13 – 7.11 (m, 1H), 4.0 (s, 2H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 137.3, 136.3, 129.6, 128.7, 128.71, 128.70, 128.4, 127.0, 126.2, 38.9. Melting point: 42 ºC.



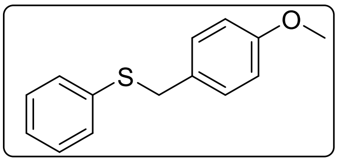
Benzyl(methyl)sulfane (3ag).[21] The representative procedure was followed using 1,2-di-p-tolyldisulfane **(1a)** (49.3 mg, 0.2 mmol) and dimethyl carbonate **(2g)** (56.9 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ag** (35.4 mg, 54%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.17 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 5.82 – 5.73 (m, 1H), 5.01 – 4.94 (m, 2H), 3.40 (d, *J* = 7.0 Hz, 2H), 2.22 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 136.3, 133.8, 132.0, 130.1, 129.7, 129.5, 128.5, 117.3, 37.8, 20.9.



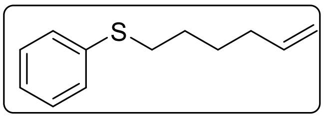
2-(Phenylthio)ethan-1-ol (3ah).[22] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and bis(2-hydroxyethyl) carbonate **(2h)** (60.2 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane: ethyl acetate = 4 : 1) yielded **3ah** (37.59 mg, 61%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 7.26 (d, *J* = 11.5 Hz, 2H), 7.17 (t, *J* = 7.5 Hz, 2H), 7.09 (t, *J* = 7.5 Hz, 2H), 3.61 (t, *J* = 6.5 Hz, 2H), 2.97 (t, *J* = 6.5 Hz, 2H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 134.9, 129.7, 128.9, 126.3, 60.2, 36.7.



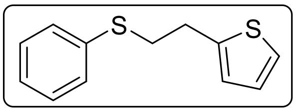
(4-Chlorobenzyl)(phenyl)sulfane (3ai).[23] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and bis(4-chlorobenzyl) carbonate **(2i)** (124.0 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ai** (62.79 mg, 61%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 7.17 – 7.19 – 7.12 (m, 6H), 7.09 – 7.07 (m, 3H), 3.94 (s, 2H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 136.1, 135.6, 132.8, 130.1, 130.0, 128.8, 128.5, 126.6, 38.3.



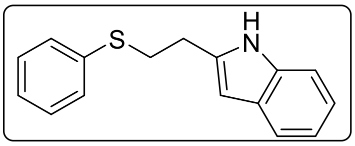
(4-Methoxybenzyl)(phenyl)sulfane (3aj).[24] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and bis(4-methoxybenzyl) carbonate **(2j)** (120.8 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane: ethyl acetate = 20 : 1) yielded **3aj** (58.88 mg, 64%) as a colorless oil. **^{1}H NMR (500 MHz, $CDCl_3$) *δ*** 7.21 (d, *J* = 7.5 Hz, 2H), 7.17 – 7.06 (m, 5H), 3.98 (s, 2H), 3.67 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) *δ*** 157, 136.5, 129.9, 129.6, 129.3, 128.7, 126.2, 113.8, 55.2, 38.3.



Hex-5-en-1-yl(phenyl)sulfane (3ak).[24] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and di(hex-5-en-1-yl) carbonate **(2k)** (90.5 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3ak** (27.67 mg, 36%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.26 – 7.18 (m, 4H), 7.11 – 7.07 (m, 1H), 5.77 – 5.67 (m, 1H), 4.96 – 4.86 (m, 2H), 2.85 (t, *J* = 9.0 Hz, 2H), 2.00 (q, *J* = 9.0 Hz, 2H), 1.64 – 1.56 (m, 2H), 1.49 – 1.44 (m, 2H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 138.4, 136.9, 128.9, 128.8, 125.7, 114.7, 33.5, 33.2, 28.6, 28.0.

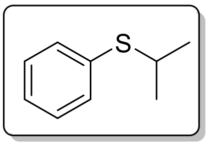


2-[2-(Phenylthio)ethyl]thiophene (3al). The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and bis(2-(thiophen-2-yl)ethyl) carbonate **(2l)** (112.8 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane) yielded **3al** (29.03 mg, 33%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.30 (d, *J* = 7.5 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.14 (d, *J* = 7.5 Hz, 1H),7.08 (d, *J* = 5.0 Hz, 1H), 6.87 – 6.85 (m, 1H), 6.77(d, *J* = 5.0 Hz, 1H), 3.13 (t, *J* = 7.0 Hz, 2H), 3.06 (t, *J* = 7.0 Hz, 2H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 142.6, 129.6, 129.0, 126.8, 126.2, 125.0, 123.7, 35.4, 29.9.

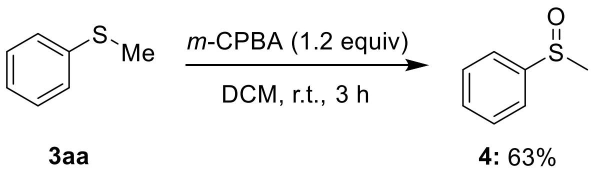


2-[2-(Phenylthio)ethyl]-1H-indole (3am). The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and bis(2-(1H-indol-3-yl)ethyl) carbonate **(2m)** (139.3 mg, 0.4 mmol). Isolation by column chromatography (*n*-hexane: ethyl acetate = 4 : 1) yielded **3am** (43.53 mg, 43%) as a colorless oil. **^{1}H NMR (500**

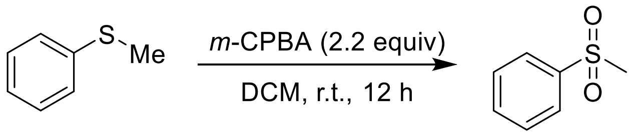
MHz, CDCl3) ***δ*** 7.83 (s, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H),7.24 – 7.19 (m, 2H), 7.14 – 7.08 (m, 2H), 7.05 – 7.02 (m, 1H), 6.92(s, 1H), 3.17 (t, *J* = 8.5 Hz, 2H), 3.01 (t, *J* = 8.5 Hz, 2H); **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 136.6, 136.2, 129.0, 128.9, 127.1, 125.8, 122.1, 121.7, 119.4, 118.6, 114.6, 111.2, 34.2, 25.3. HRMS (ESI) m/z ([M+ H]+) Calcd. for $C_{16}H_{15}NS$ 254.0998, found: 254.0999.



Isopropyl(phenyl)sulfane (3an).[25] The representative procedure was followed using 1,2-diphenyldisulfane **(1a)** (43.7 mg, 0.2 mmol) and di-isopropyl carbonate **(2h)** (58.4 mg, 0.4 mmol) at 180 ºC. Isolation by column chromatography (*n*-hexane) yielded **3an** (38.3 mg, 63%) as a colorless oil. **^{1}H NMR (500 MHz, CDCl3)** ***δ*** 7.34 – 7.32 (m, 2H), 7.24 – 7.21 (m, 2H), 7.17 – 7.14 (m, 1H), 3.33 – 3.28 (m, H), 1.22 (d, *J* = 7.0 Hz, 6H); **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 135.5, 131.9, 128.8, 126.7, 38.2, 23.1.

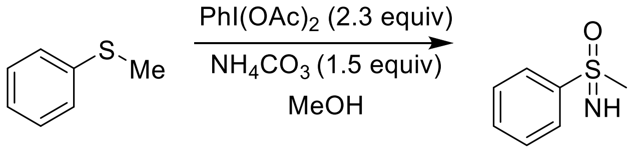


To a 10 mL Schlenk tube was added sequentially **3aa** (0.2 mmol) and DCM (0.4 mL) via syringe. Then *m*-CPBA (41.4 mg, 0.24 mmol, 1.2 equiv) was added. The resulting mixture was stirred at room temperature for 3 h. After the reaction was completed, the reaction was quenched with NaOH (1.0 M, 2.0 mL) and the crude mixture was extracted with DCM. The organic layer was dried, filtered, and concentrated. The residue was purified the residue was purified by column chromatography to afford product **4** as a colorless oil.[25] **^{1}H NMR (500 MHz, CDCl3)** ***δ*** 7.58 – 7.56 (m, 2H), 7.47 – 7.42 (m, 3H), 2.64 (s, 3H). **^{13}C NMR (125 MHz, CDCl3)** ***δ*** 145.5, 130.9, 129.2, 123.4, 43.8.[27]



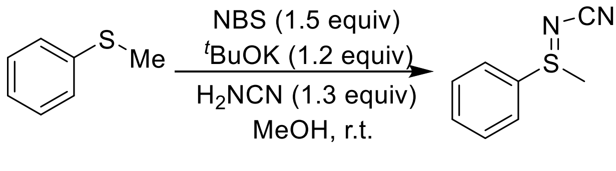
3aa **5:** 77%

To a 10 mL Schlenk tube was added sequentially **3aa** (0.2 mmol) and DCM (0.4 mL) via syringe. Then *m*-CPBA (76 mg, 0.44 mmol, 2.2 equiv.) was added. The resulting mixture was stirred at room temperature for 3 h. After the reaction was completed, the reaction was quenched with NaOH (1.0 M, 2.0 mL) and crude mixture was extracted with DCM. The organic layer was dried, filtered, and concentrated. The residue was purified by column chromatography to afford product **5** as a colorless oil.[26] **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.87 – 7.85 (m, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.50 – 7.47 (m, 2H), 2.97 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 140.4, 133.6, 129.2, 127.1, 44.3.

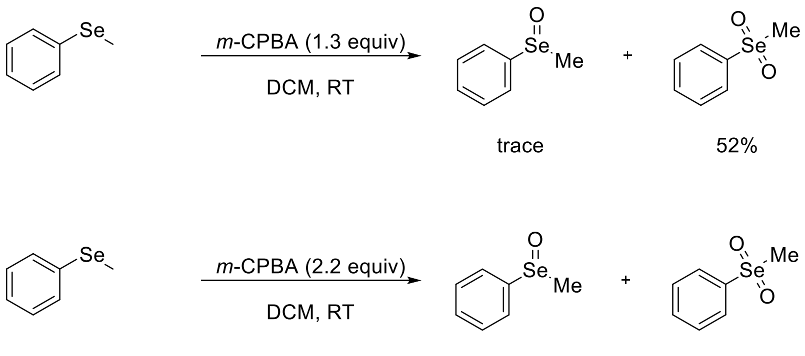


3aa **6:** 80%

To a 10 mL Schlenk tube was added **3aa** (0.2 mmol) and MeOH (0.4 mL) via syringe. Then $(NH_4)_2CO_3$ (28.8mg, 0.3 mmol, 1.5 equiv) was added, followed by the $PhI(OAc)_2$ (148.1 mg, 0.46 mmol, 2.3 equiv) in one portion. The resulting mixture was stirred at room temperature for 0.5 h. After the reaction was completed, the solvent was removed under reduced pressure. Then, H_2O (5 mL) was added into the reaction mixture and extracted with ethyl acetate (5 mL x 3). The organic layer was dried, filtered, and concentrated. The residue was purified by column chromatography to afford product **6** as a colorless oil.[25] **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 7.93 – 7.89 (m, 2H), 7.53 – 7.43 (m, 3H), 3.01 – 2.99 (s, 3H), 2.78 (br, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 143.1, 132.7, 128.9, 127.3, 45.9.



To a 10 mL Schlenk tube was added sequentially **3aa** (0.2 mmol) and MeOH (0.4 mL) via syringe. Then H_2NCN (11.0 mg, 0.26 mmol, 1.3 equiv) was added, followed by the ^{t}BuOK (27 mg, 0.24 mmol, 1.2 equiv) in one portion. Subsequently NBS (53.4 mg, 1.5 equiv) was added. After the reaction was completed, the solvent was removed under reduced pressure. Then saturated aqueous $Na_2S_2O_3$ was added. The crude mixture was extracted with DCM. The organic layer was dried, filtered, and concentrated. The residue was purified by column chromatography to afford product **7** as a colorless oil.[28] **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.73 – 7.71 (m, 2H), 7.56 – 7.52 (m, 3H), 2.95 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 135.8, 132.8, 130.0, 125.6, 120.3, 36.3.



To a 10 mL Schlenk tube was added sequentially **3a'a** (0.2 mmol) and DCM (0.4 mL) via syringe. Then *m*-CPBA (76 mg, 0.44 mmol, 2.2 equiv.) was added. The resulting mixture was stirred at room temperature for 3 h. After the reaction was completed, the reaction was quenched with NaOH (1.0 M, 2.0 mL) and crude mixture was extracted with DCM. The organic layer was dried, filtered, and concentrated. The residue was purified by column chromatography to afford product (methylselenonyl)benzene as a colorless oil.[29] **^{1}H NMR (500 MHz, CDCl$_3$)** ***δ*** 7.96 – 7.94 (m, 2H), 7.67 – 7.58 (m, 3H), 3.23 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** ***δ*** 142.5, 134.4, 130.3, 126.5, 44.3.

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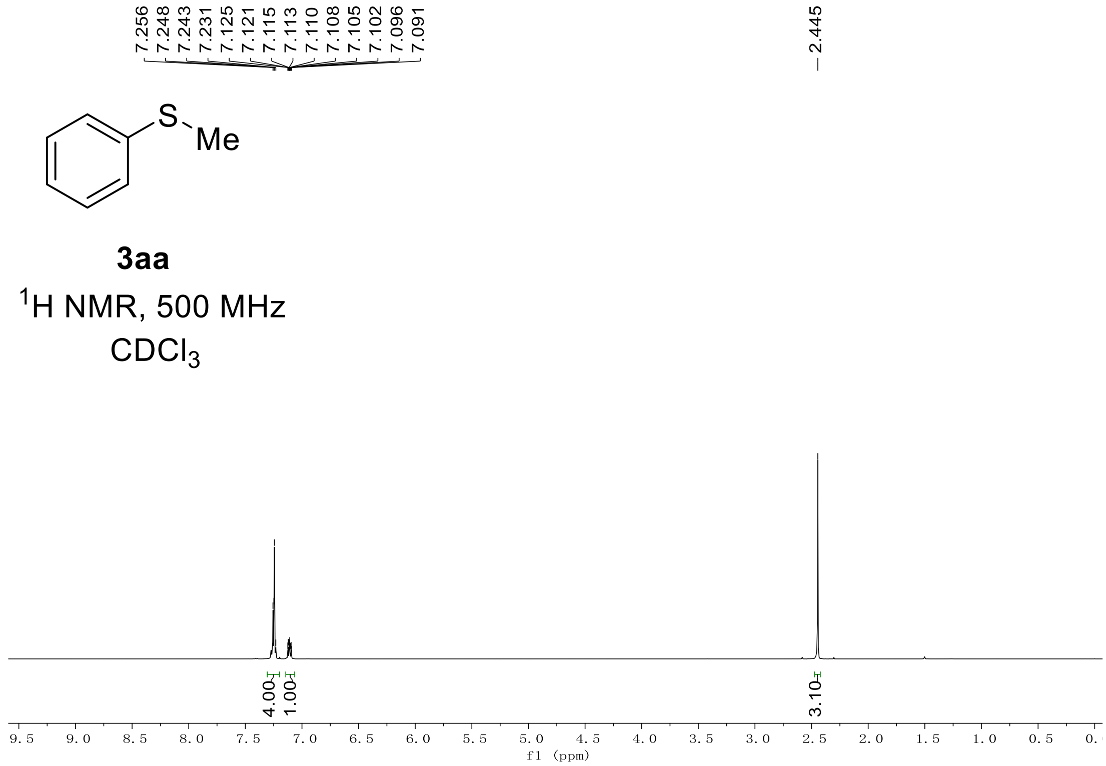
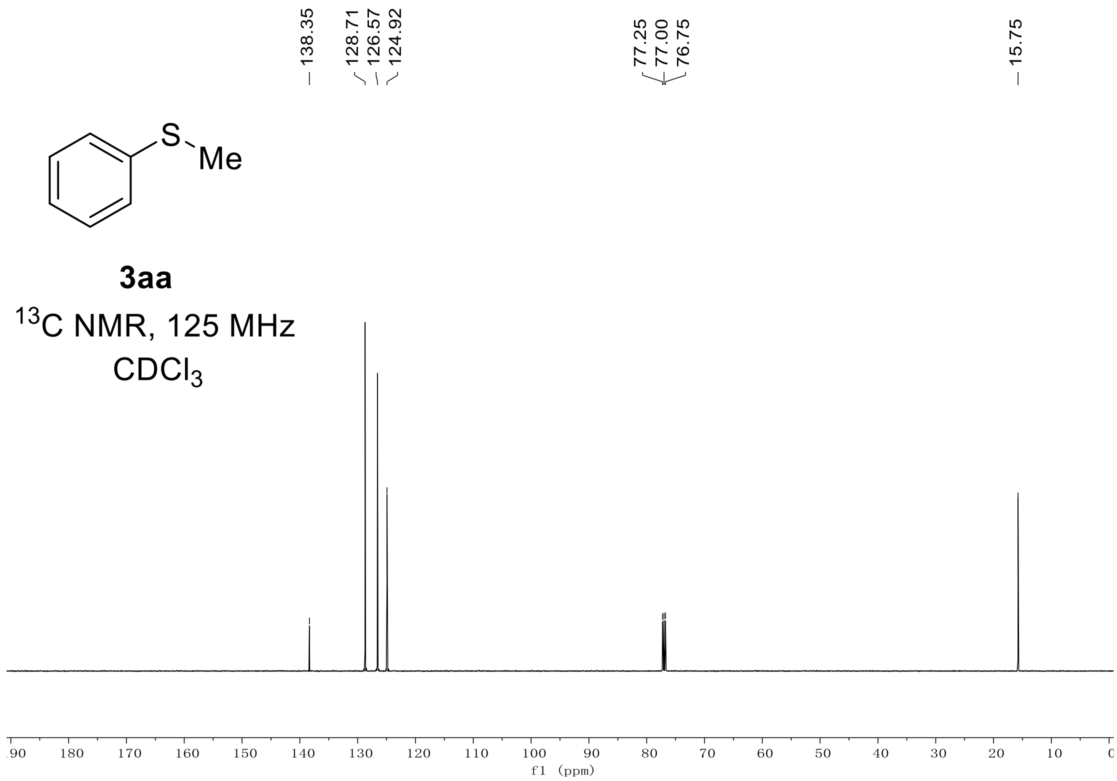
5. NMR Spectra



3aa

^{1}H NMR, 500 MHz

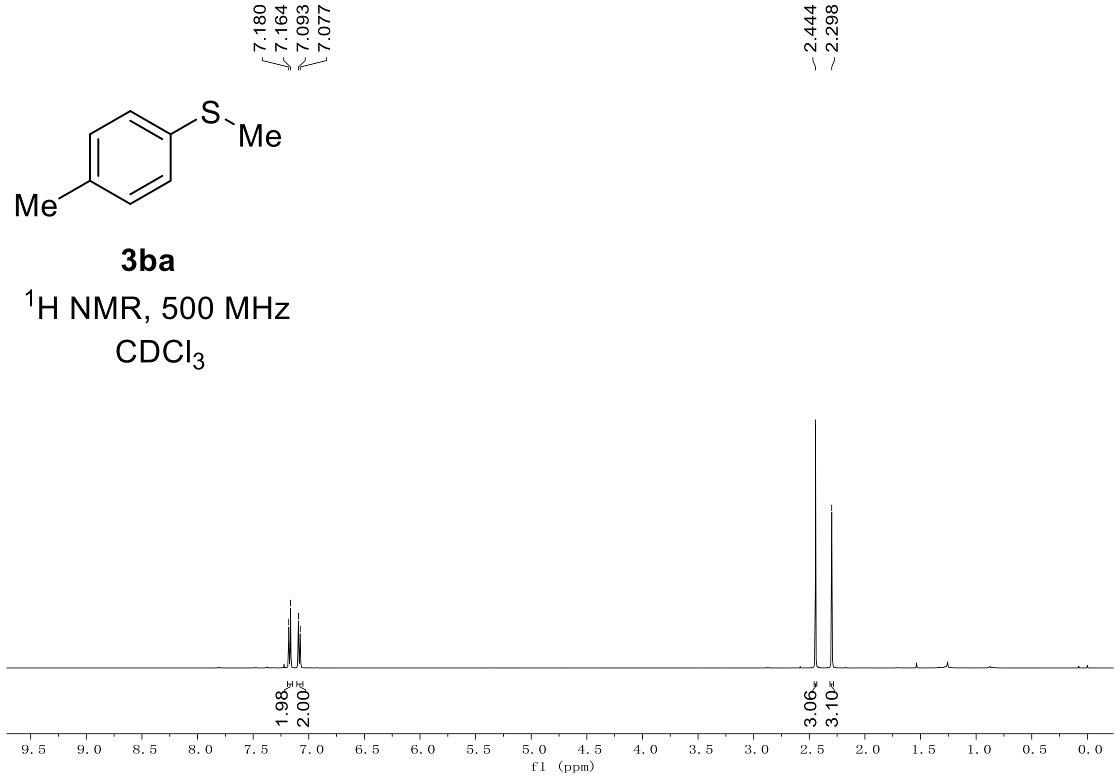
$CDCl_3$



3aa

^{13}C NMR, 125 MHz

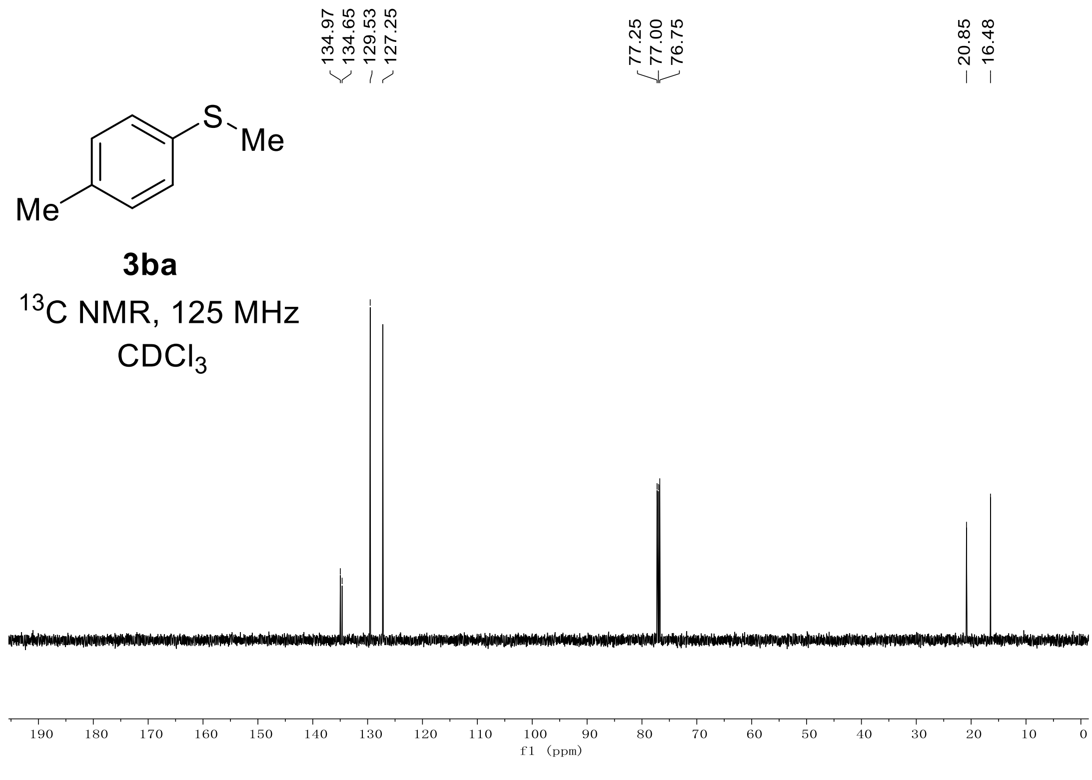
$CDCl_3$



3ba

^{1}H NMR, 500 MHz

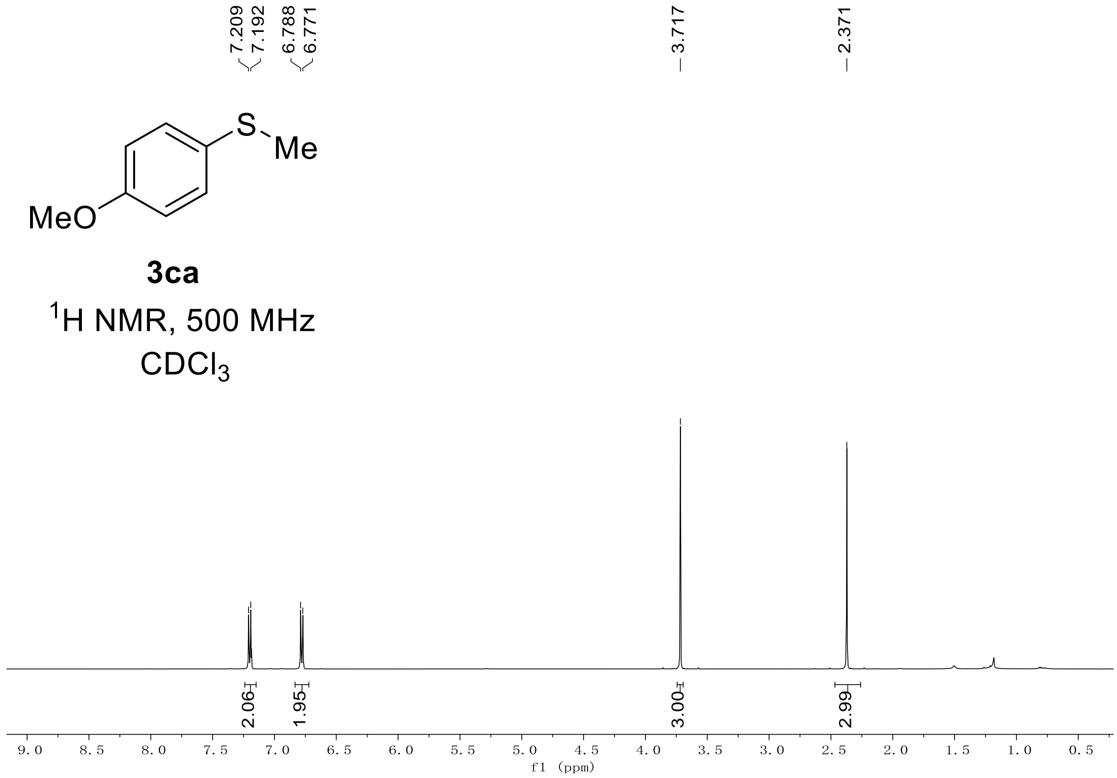
$CDCl_3$

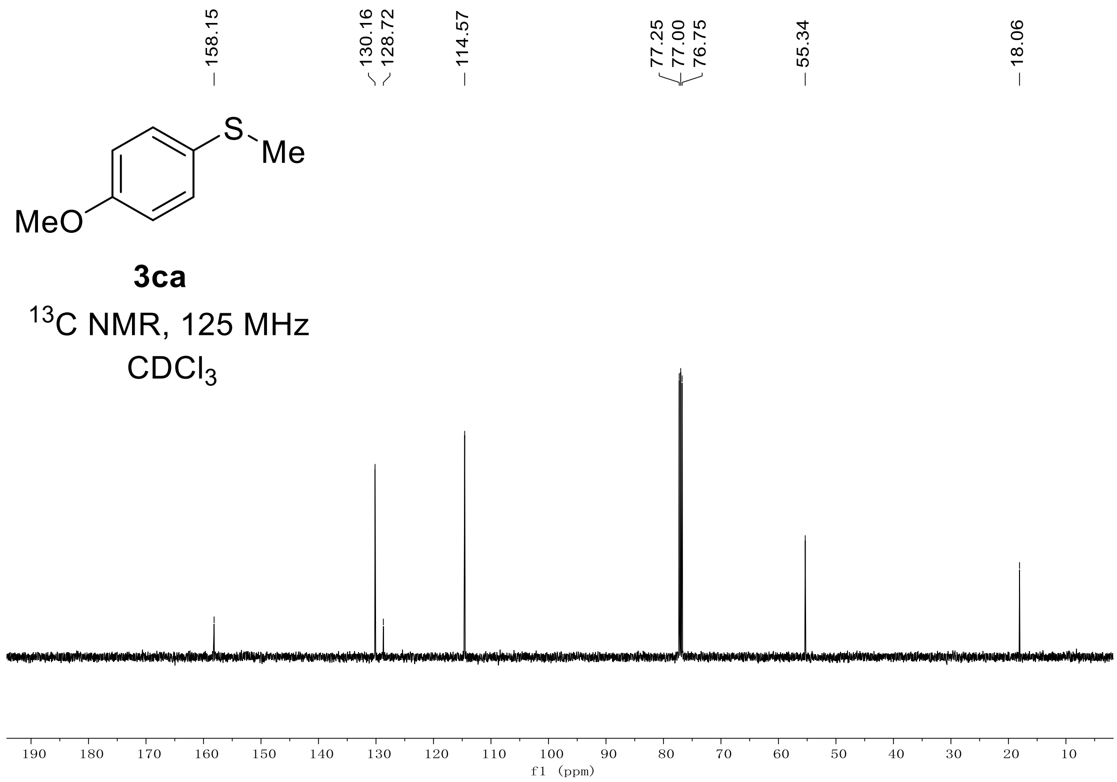


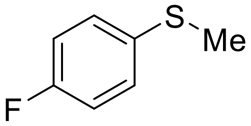
3ba

^{13}C NMR, 125 MHz

$CDCl_3$


7.209
7.192
6.788
6.771
3.717
2.371
S
Me
MeO
3ca
^{1}H NMR, 500 MHz
$CDCl_3$
2.06
1.95
3.00
2.99
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)

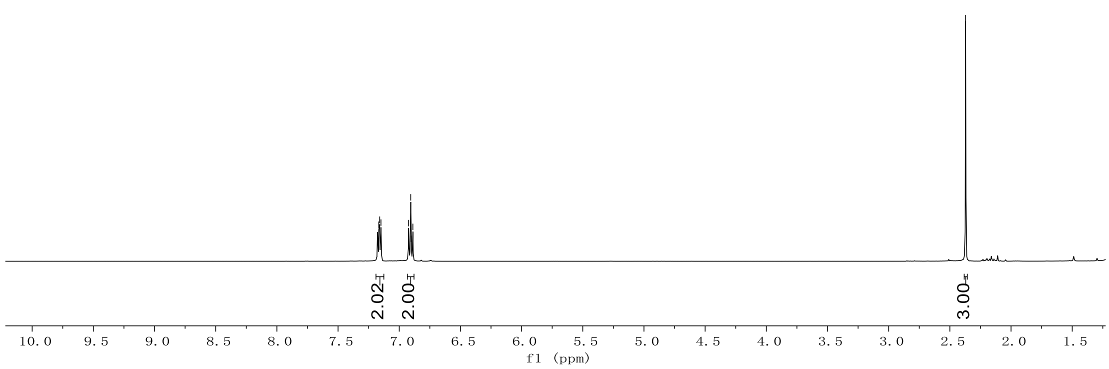

158.15
130.16
128.72
114.57
77.25
77.00
76.75
55.34
18.06
S
Me
MeO
3ca
^{13}C NMR, 125 MHz
$CDCl_3$
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

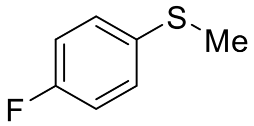


3da

^{1}H NMR, 500 MHz

$CDCl_3$

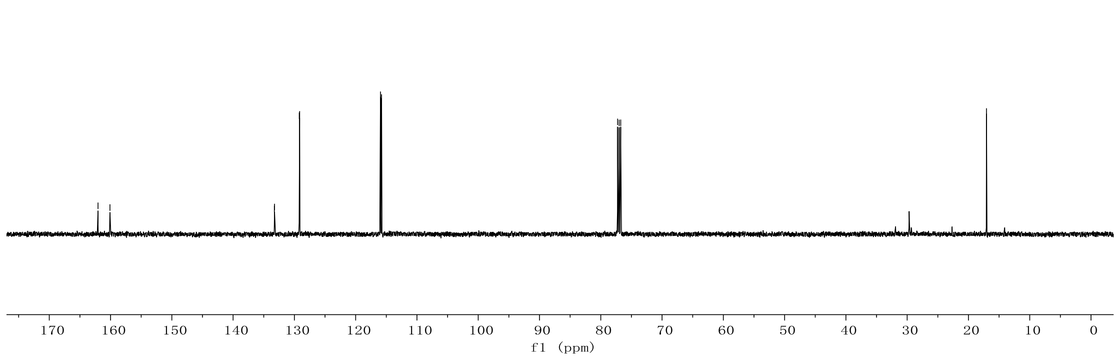


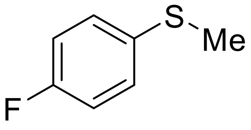


3da

^{13}C NMR, 125 MHz

$CDCl_3$

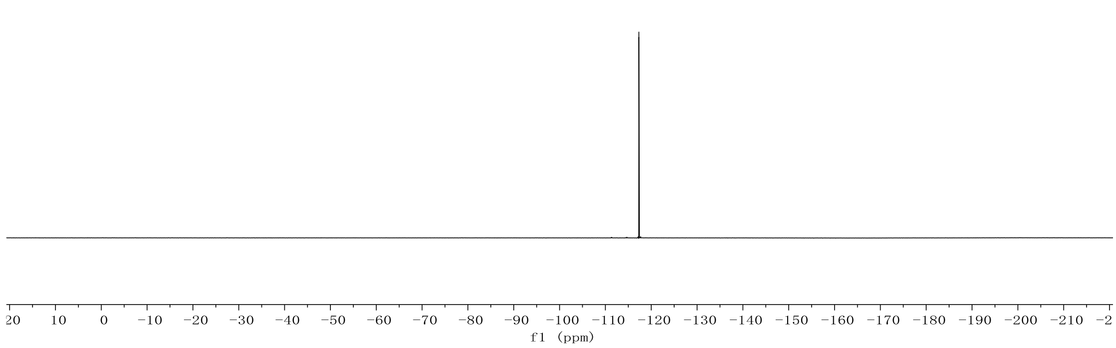


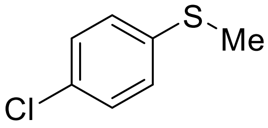


3da

^{19}F NMR, 471 MHz

$CDCl_3$

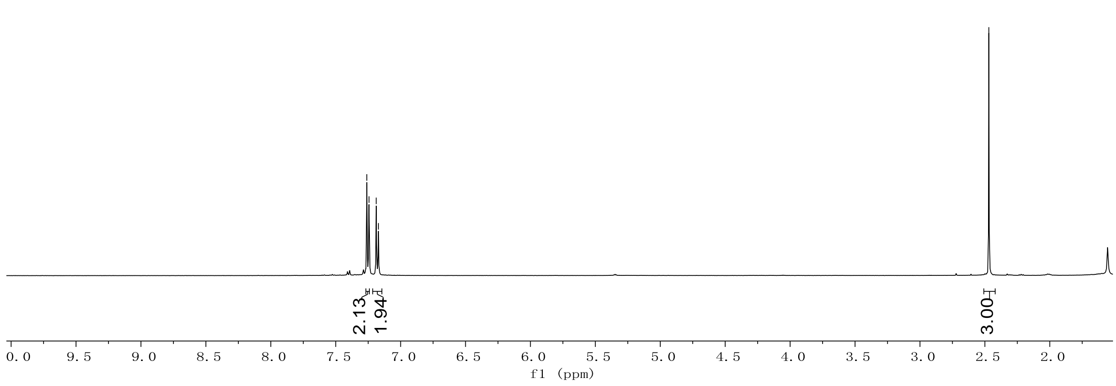


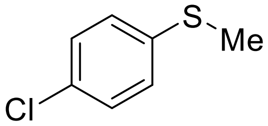


3ea

^{1}H NMR, 500 MHz

$CDCl_3$

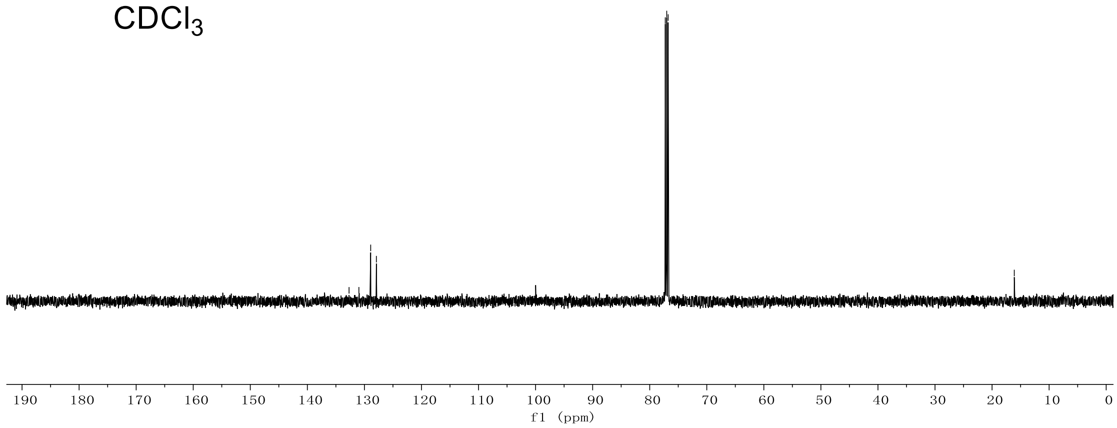


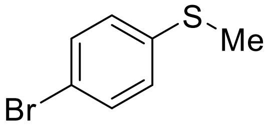


3ea

^{13}C NMR, 125 MHz

$CDCl_3$

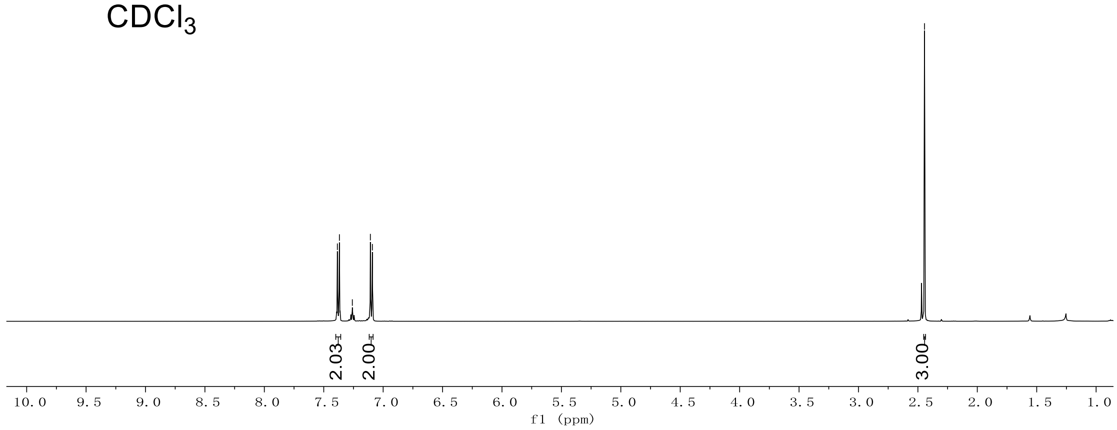


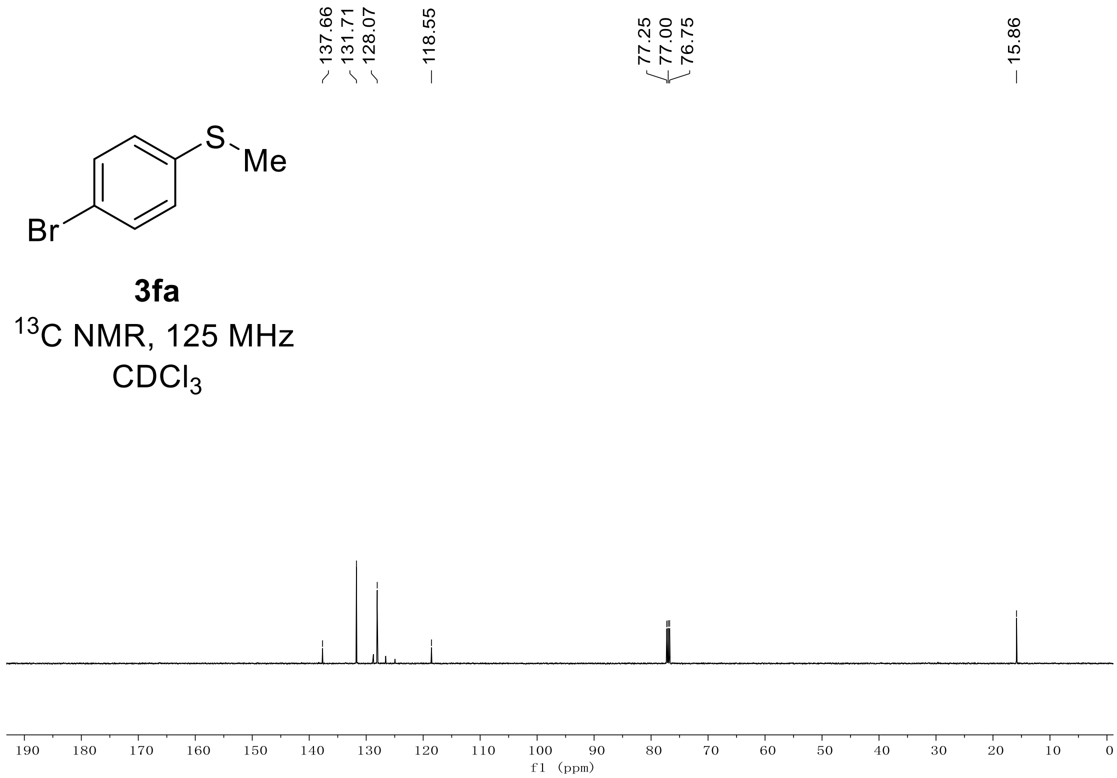


3fa

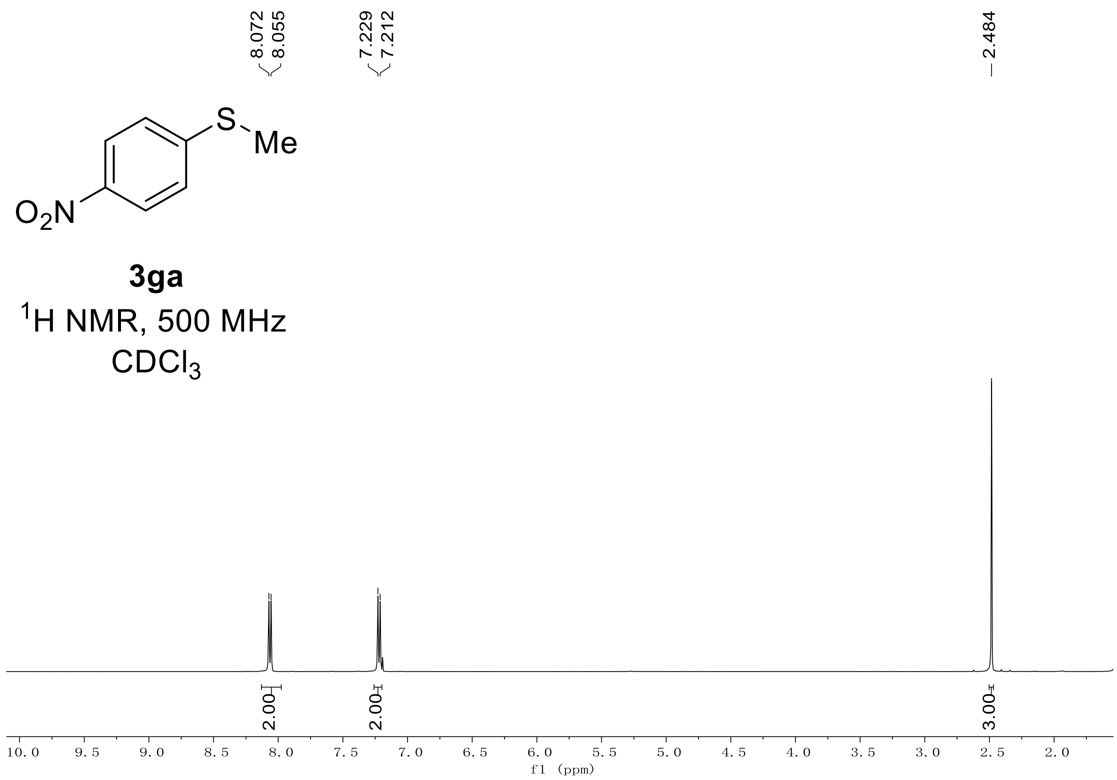
^{1}H NMR, 500 MHz

$CDCl_3$

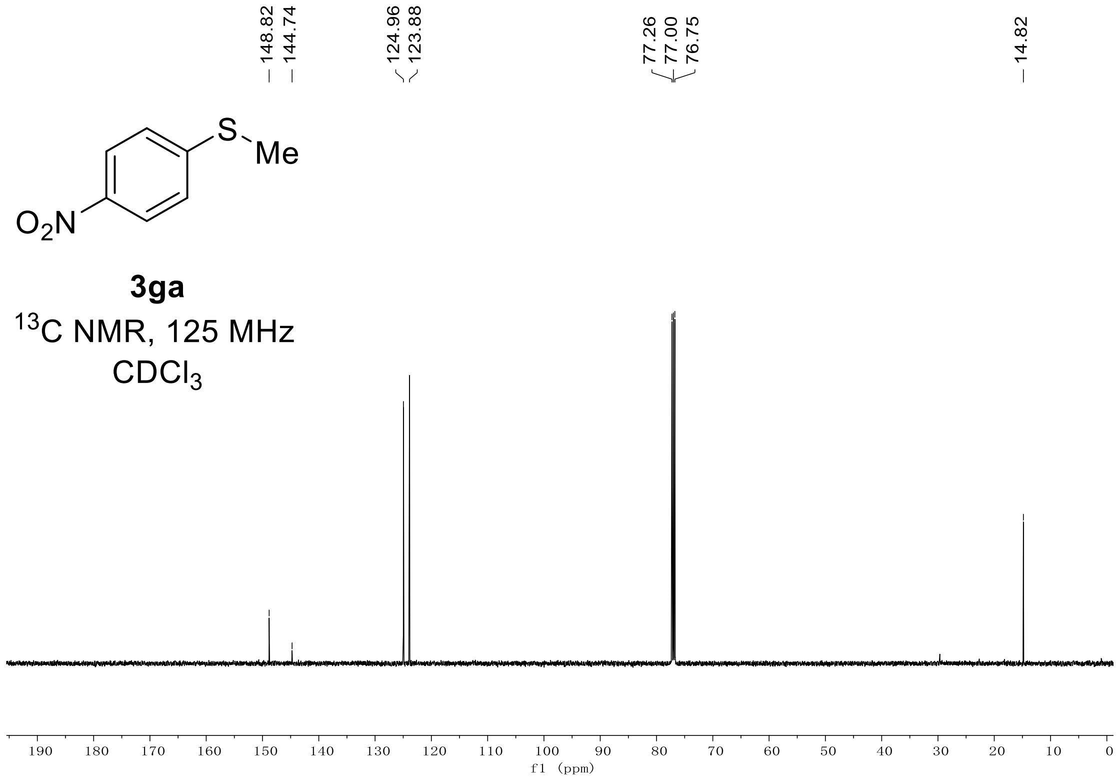


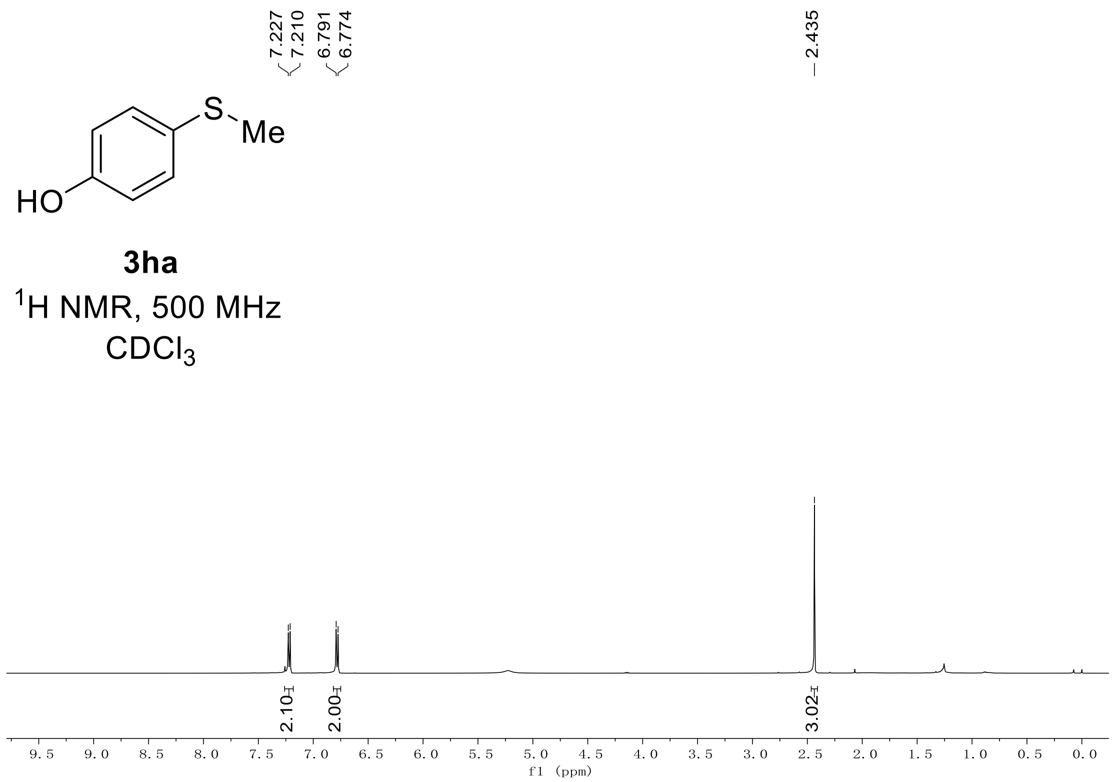


137.66
131.71
128.07
118.55
77.25
77.00
76.75
15.86
S
Me
Br
3fa
^{13}C NMR, 125 MHz
$CDCl_3$
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



8.072
8.055
7.229
7.212
2.484
S
Me
O_2N
3ga
^{1}H NMR, 500 MHz
$CDCl_3$
2.00
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
f1 (ppm)


148.82
144.74
124.96
123.88
77.26
77.00
76.75
14.82
S
Me
O_2N
3ga
^{13}C NMR, 125 MHz
$CDCl_3$
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.210
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6.774
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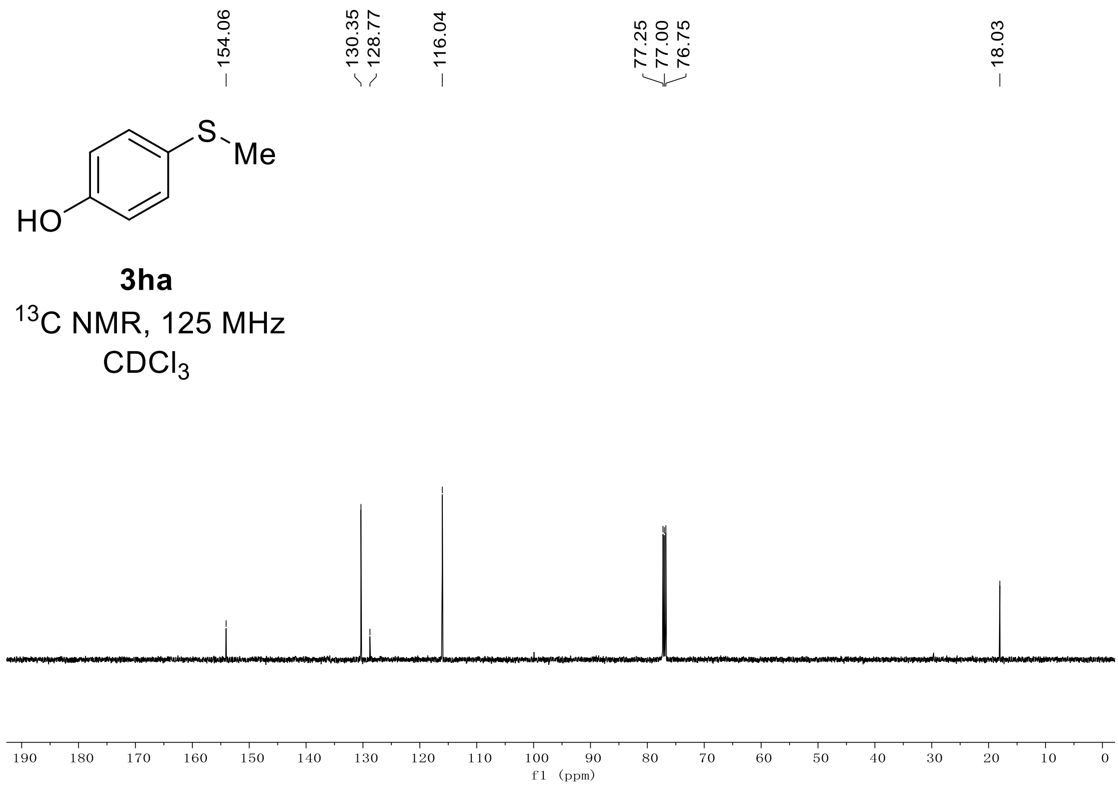
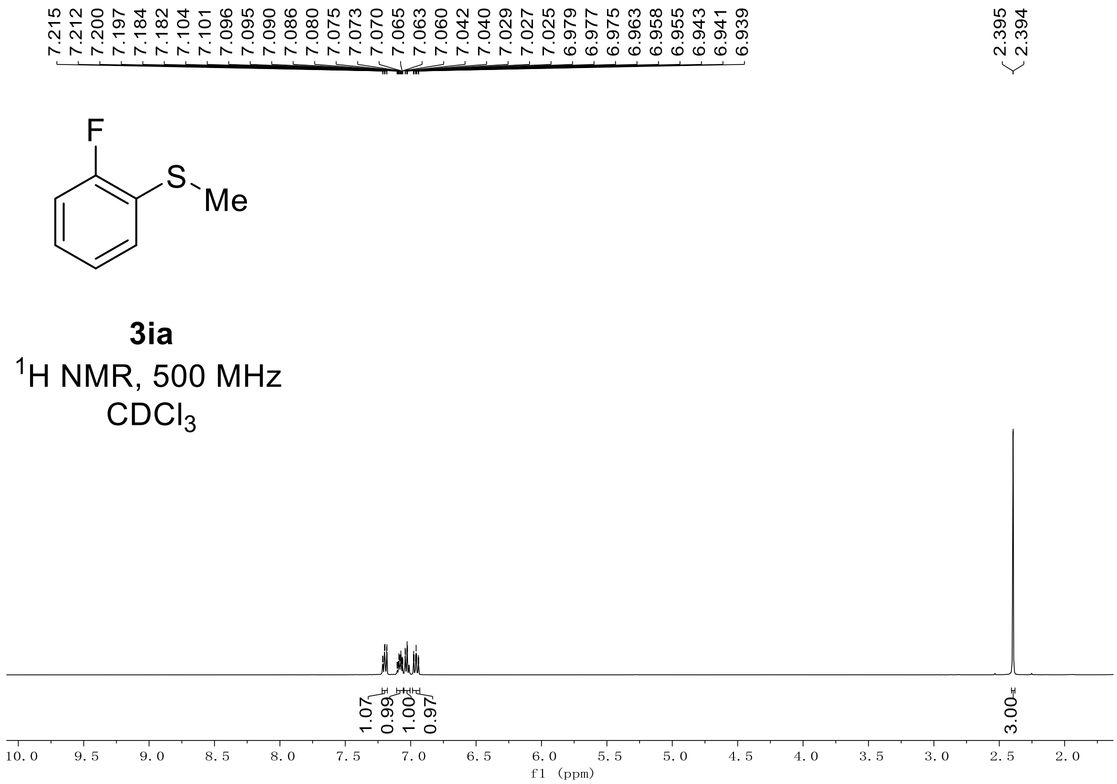
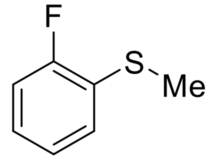
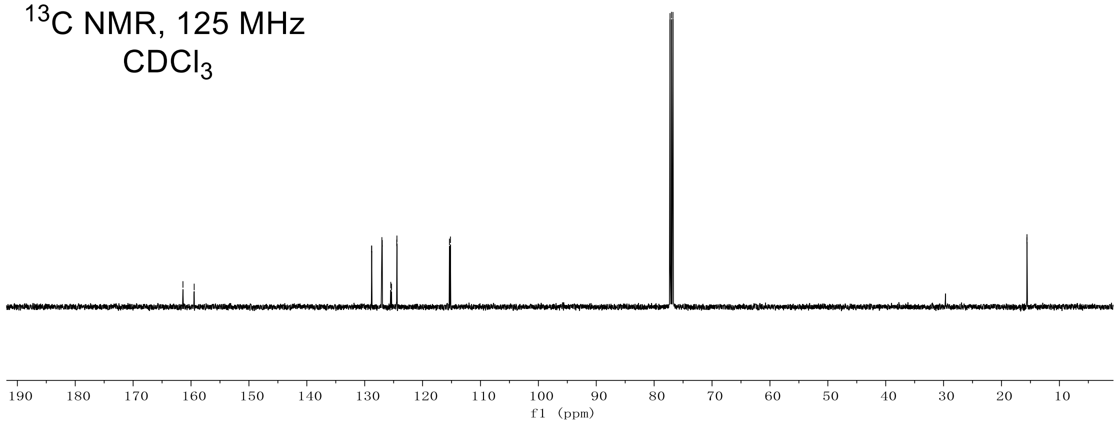
S
Me
HO
3ha
^{1}H NMR, 500 MHz
$CDCl_3$
2.10
2.00
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

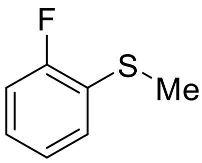

154.06
130.35
128.77
116.04
77.25
77.00
76.75
18.03
S
Me
HO
3ha
^{13}C NMR, 125 MHz
$CDCl_3$
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.215
7.212
7.200
7.197
7.184
7.182
7.104
7.101
7.096
7.095
7.090
7.086
7.080
7.075
7.073
7.070
7.065
7.063
7.060
7.042
7.040
7.029
7.027
7.025
6.979
6.977
6.975
6.963
6.958
6.955
6.943
6.941
6.939
2.395
2.394
F
S
Me
3ia
^{1}H NMR, 500 MHz
$CDCl_3$
1.07
0.99
1.00
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
f1 (ppm)

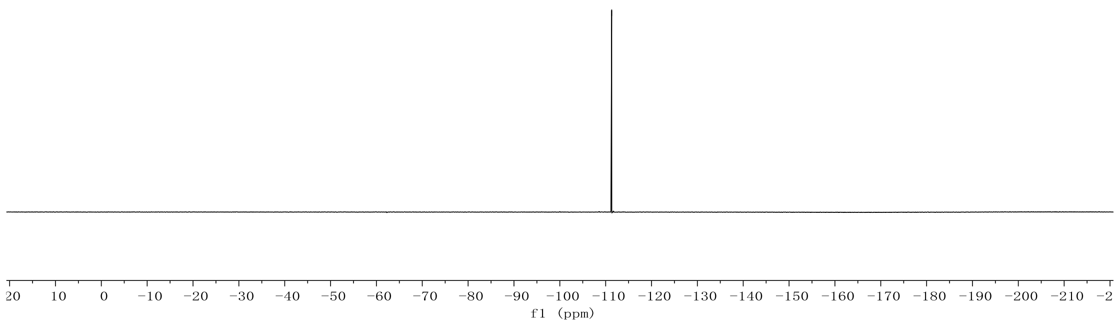


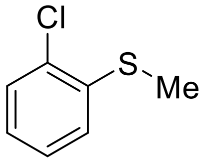
3ia
^{13}C NMR, 125 MHz
$CDCl_3$





3ia
^{19}F NMR, 471 MHz
$CDCl_3$

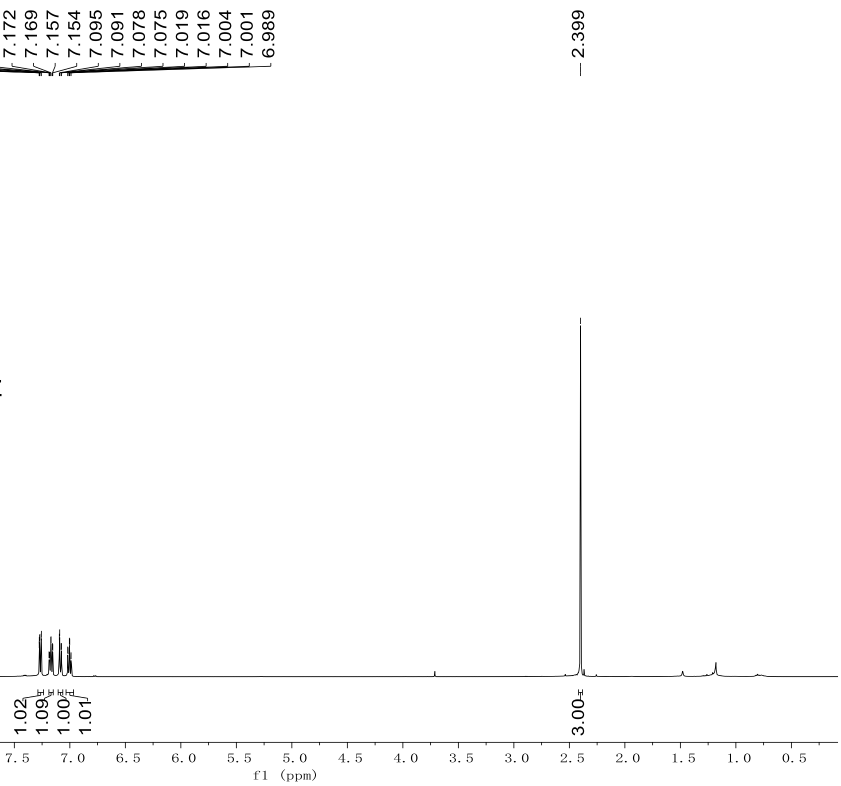


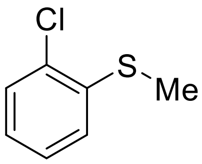


3ja

^{1}H NMR, 500 MHz

$CDCl_3$

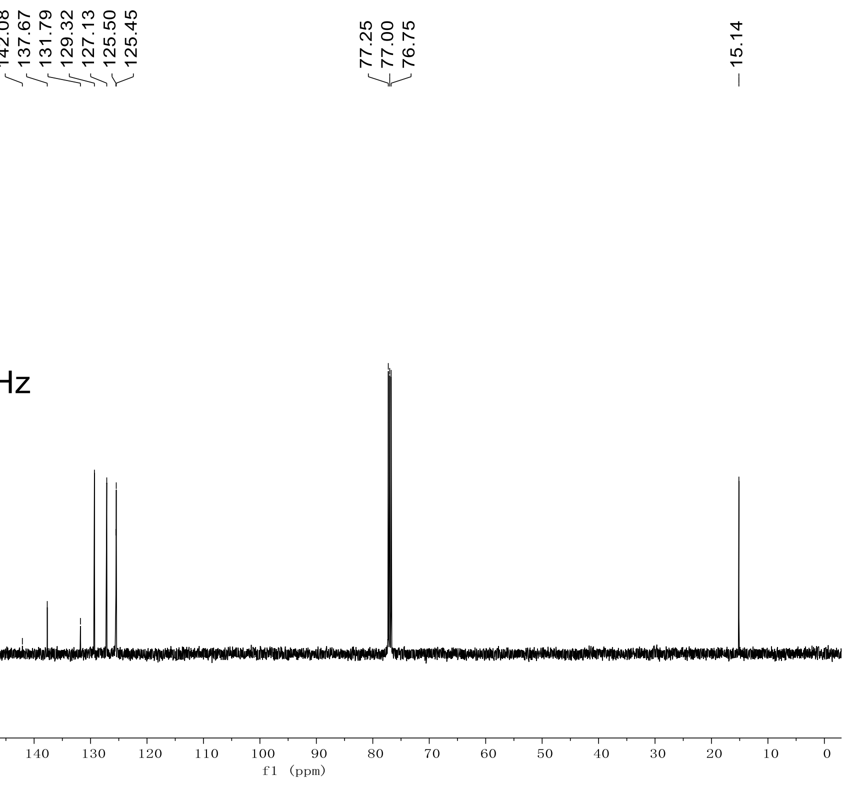


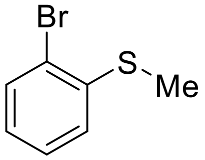


3ja

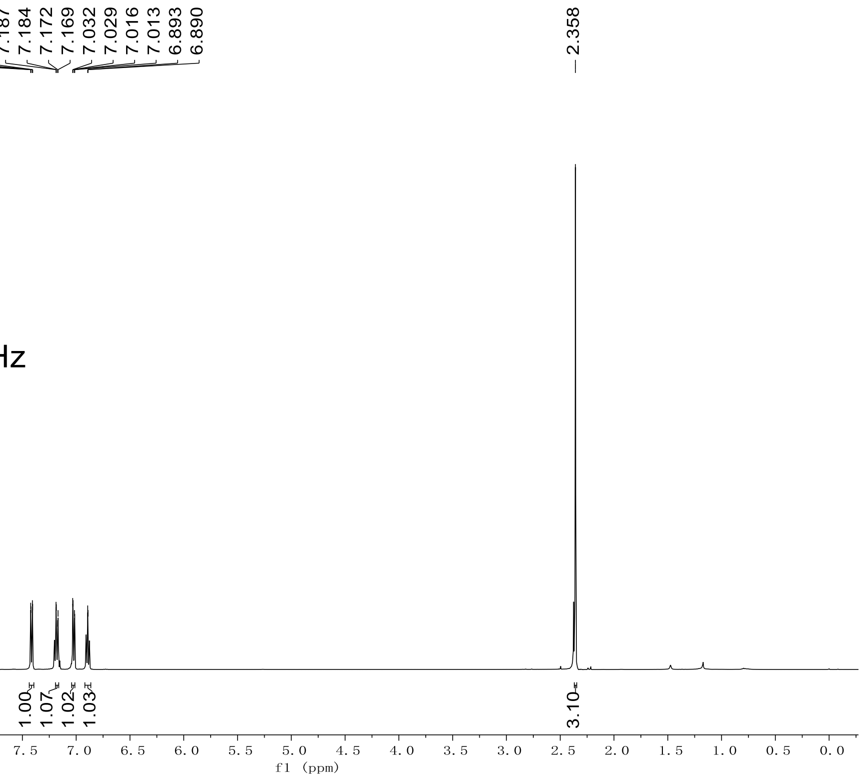
^{13}C NMR, 125 MHz

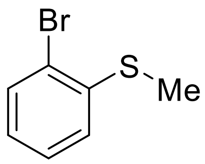
$CDCl_3$



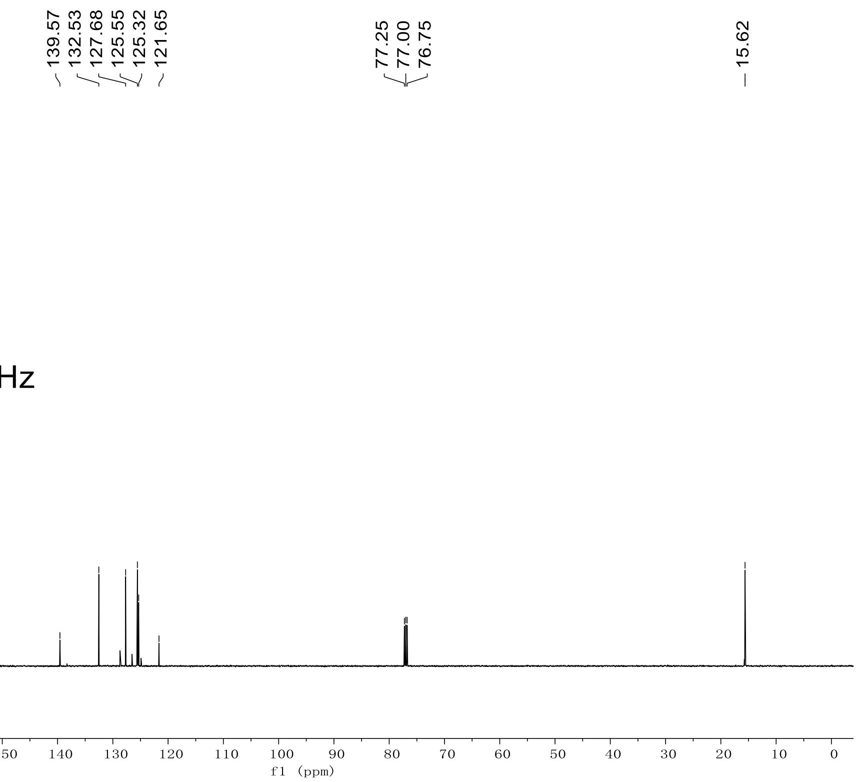


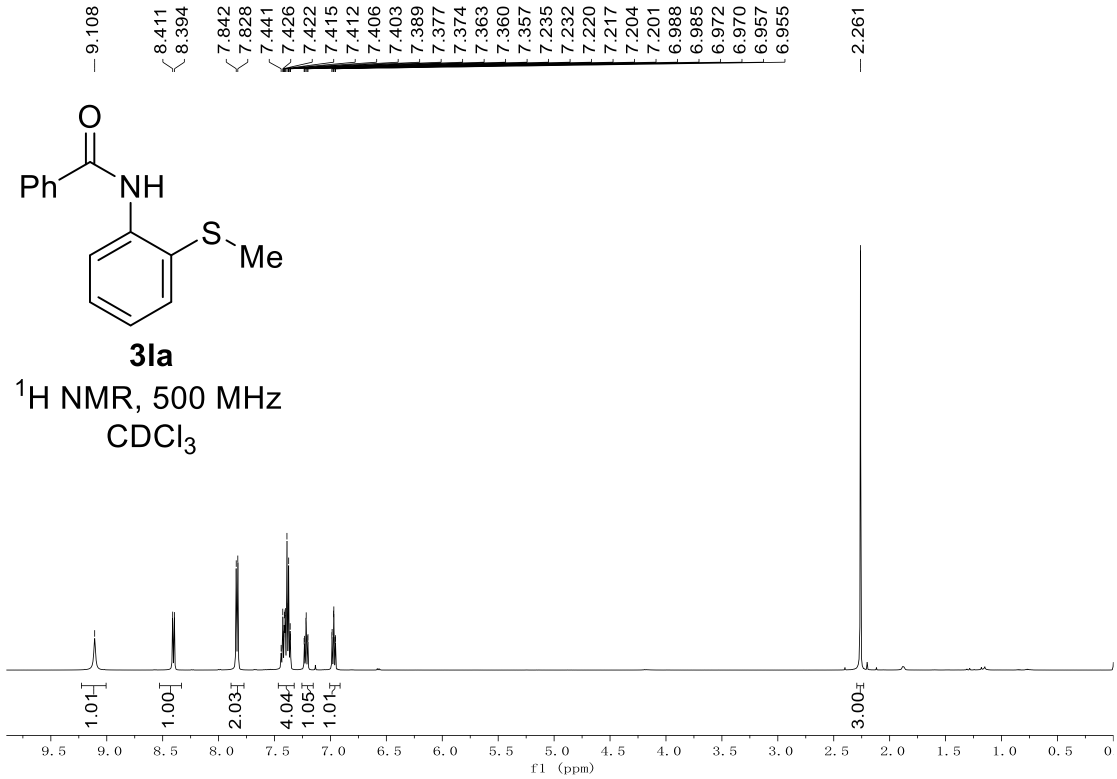
3ka
^{1}H NMR, 500 MHz
$CDCl_3$





3ka
^{13}C NMR, 125 MHz
$CDCl_3$

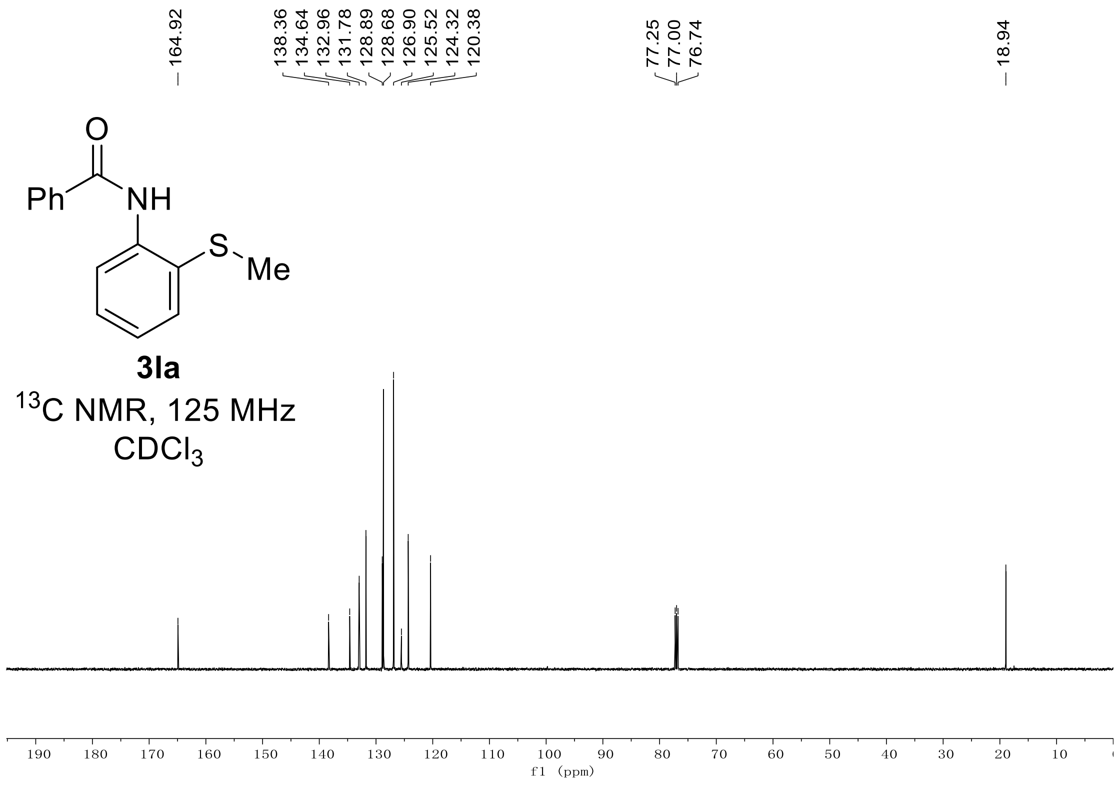




3la

^{1}H NMR, 500 MHz

$CDCl_3$



3la

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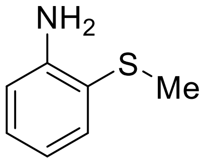

3ma

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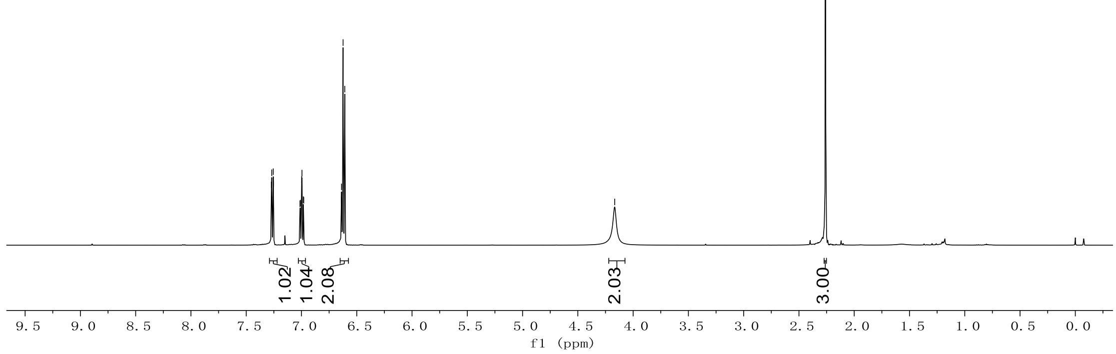
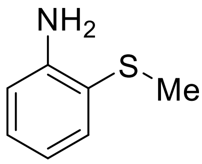
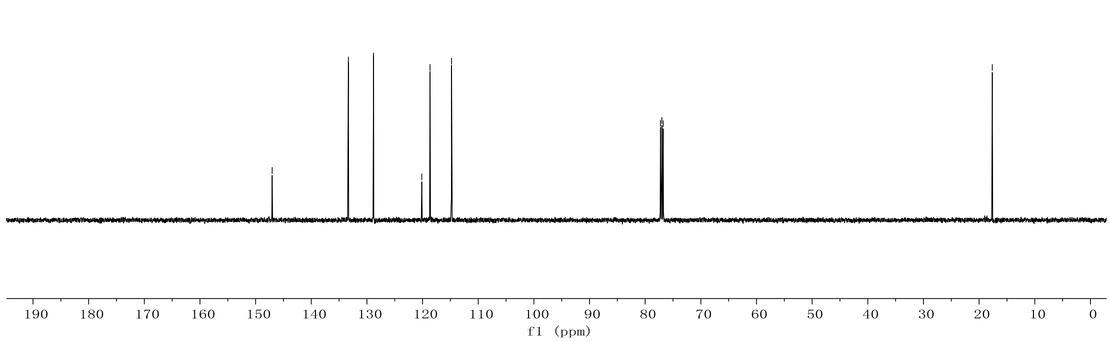


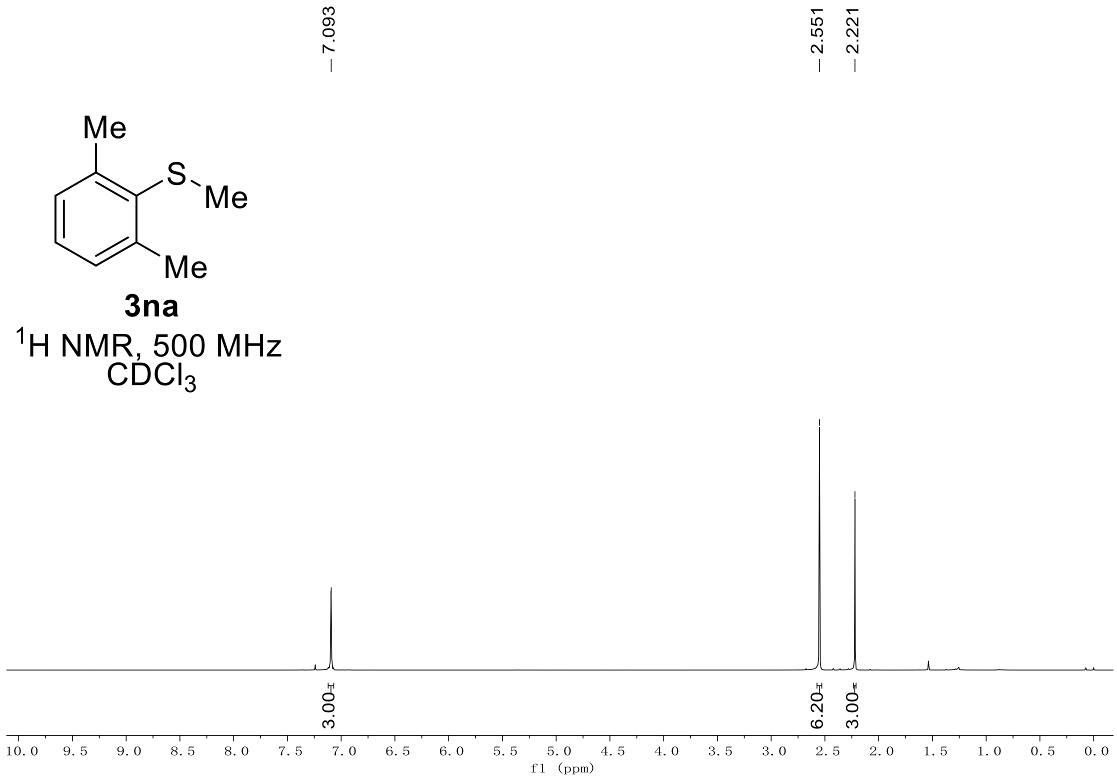


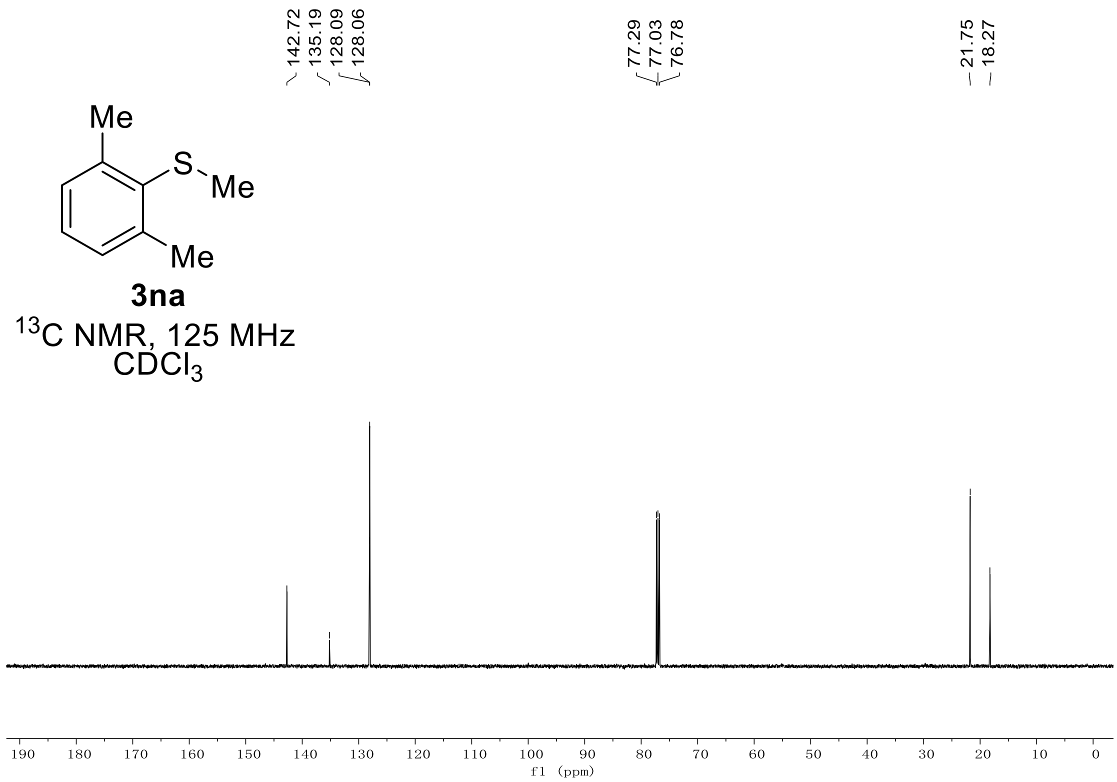
3ma

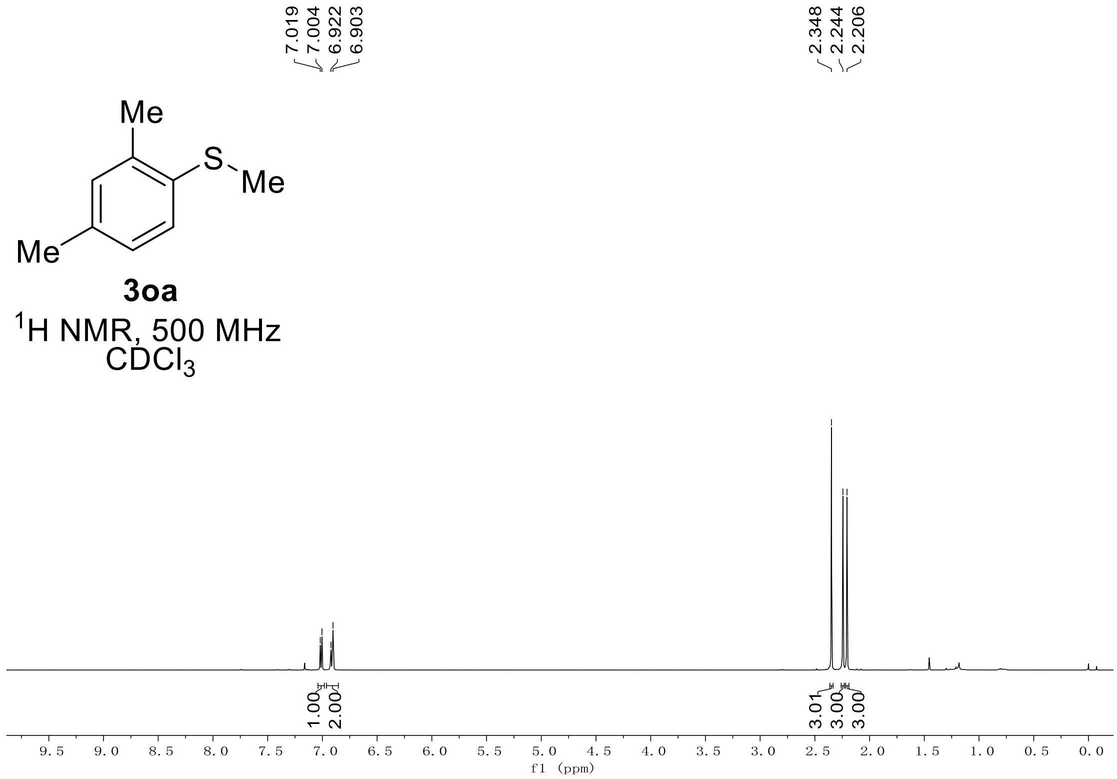
^{13}C NMR, 125 MHz

$CDCl_3$




7.093
2.551
2.221
Me
S
Me
Me
3na
1H NMR, 500 MHz
CDCl3
3.00
6.20
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)

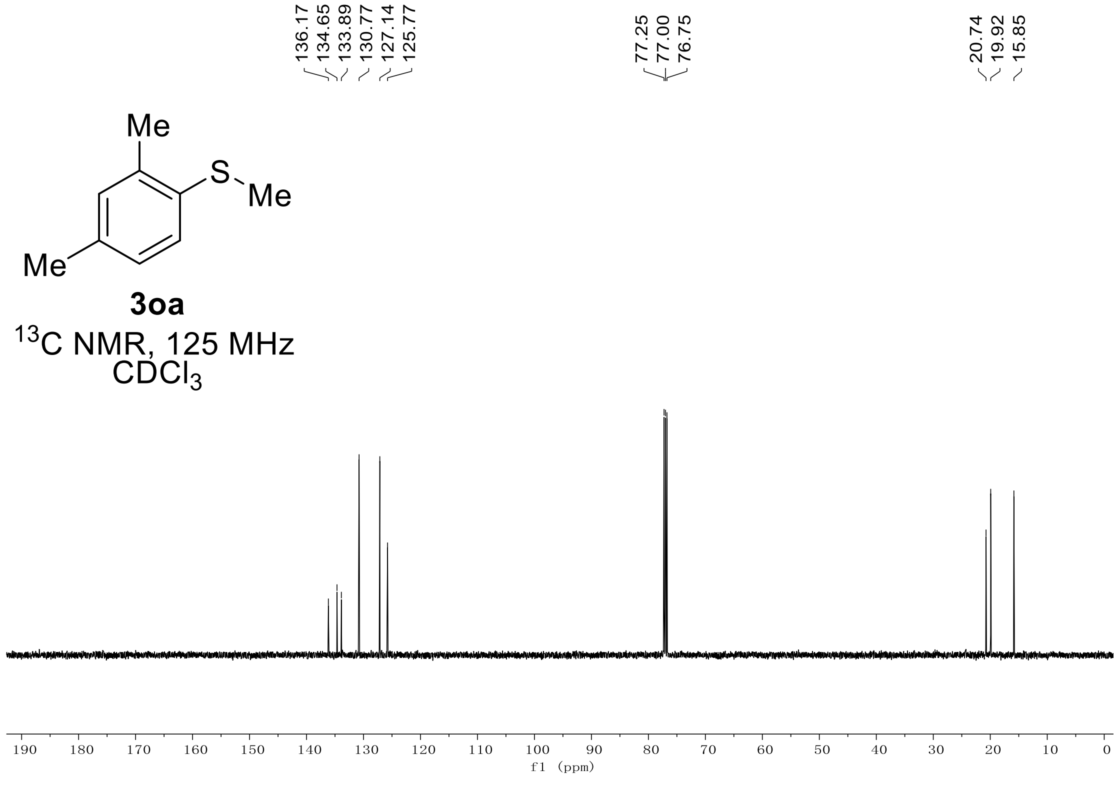

142.72
135.19
128.09
128.06
77.29
77.03
76.78
21.75
18.27
Me
S
Me
Me
3na
13C NMR, 125 MHz
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



3oa

^{1}H NMR, 500 MHz

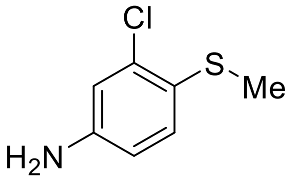
$CDCl_3$



3oa

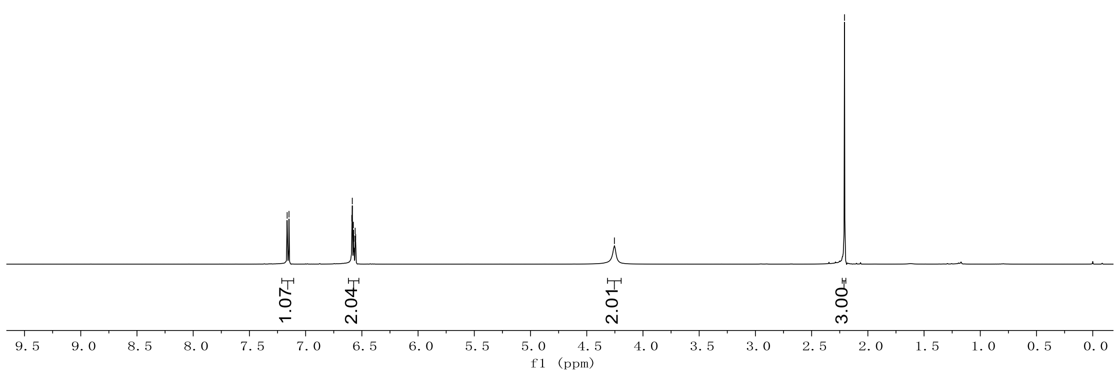
^{13}C NMR, 125 MHz

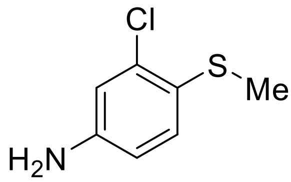
$CDCl_3$



3pa

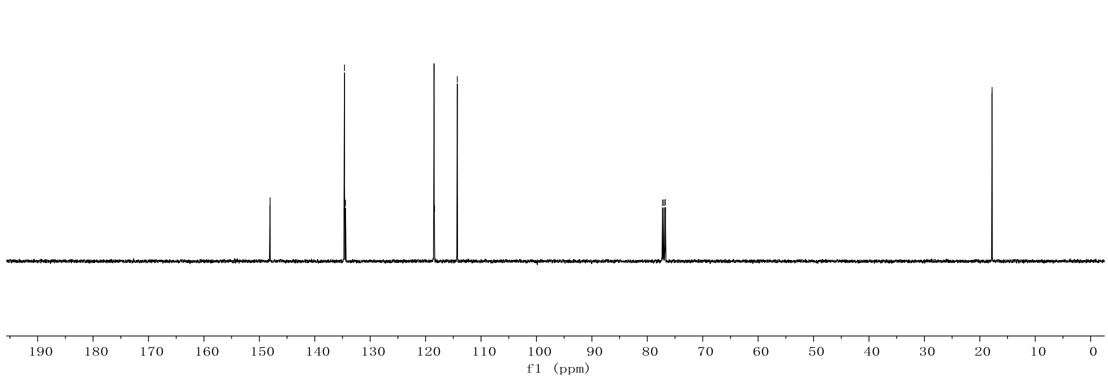
^{1}H NMR, 500 MHz
$CDCl_3$

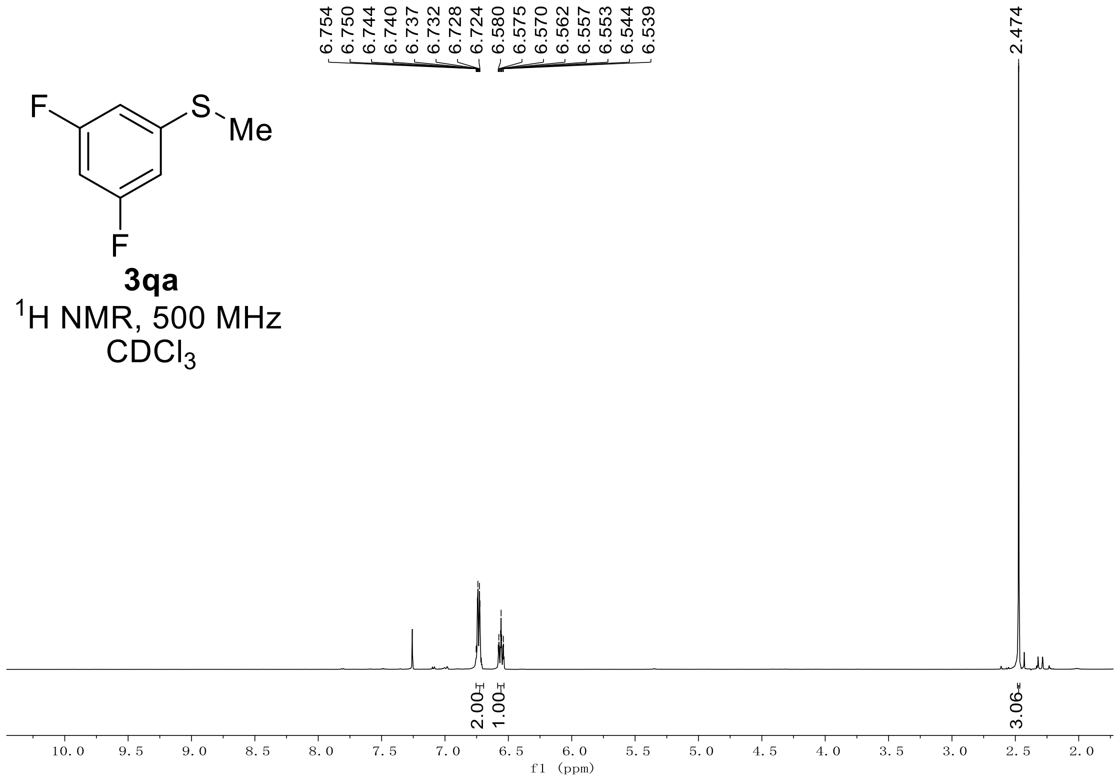




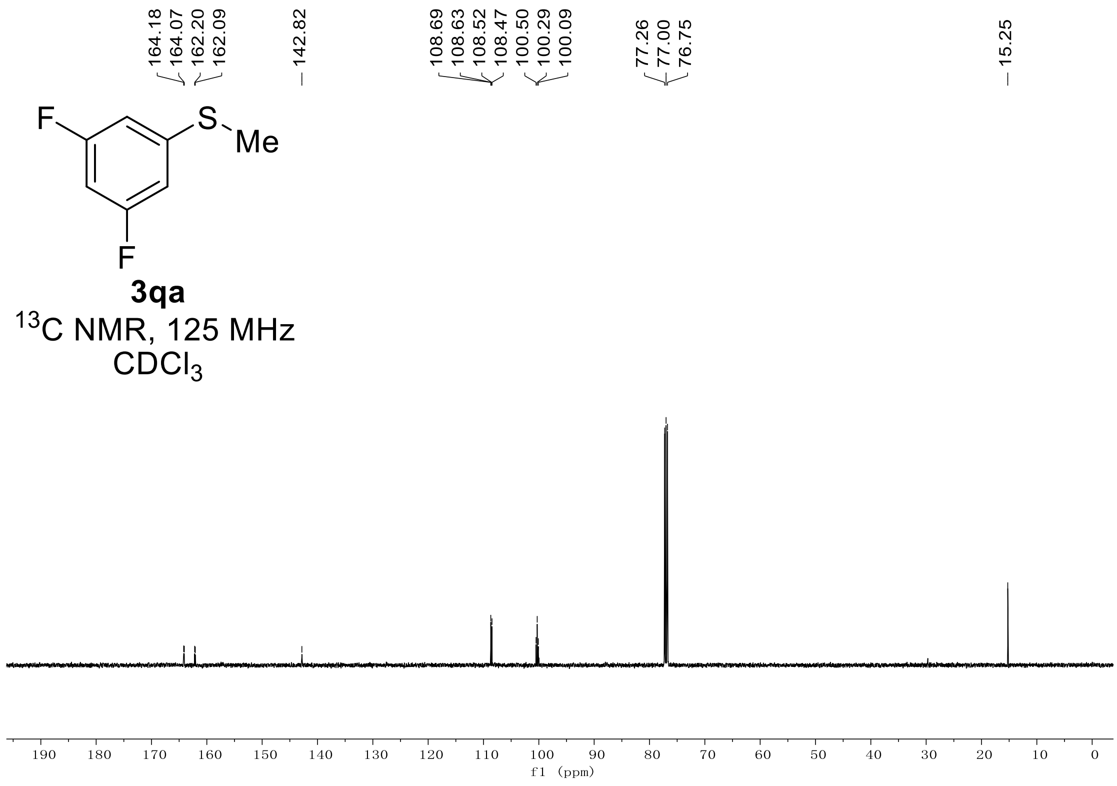
3pa

^{13}C NMR, 125 MHz
$CDCl_3$

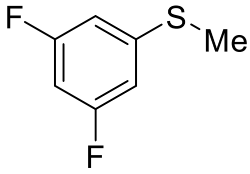




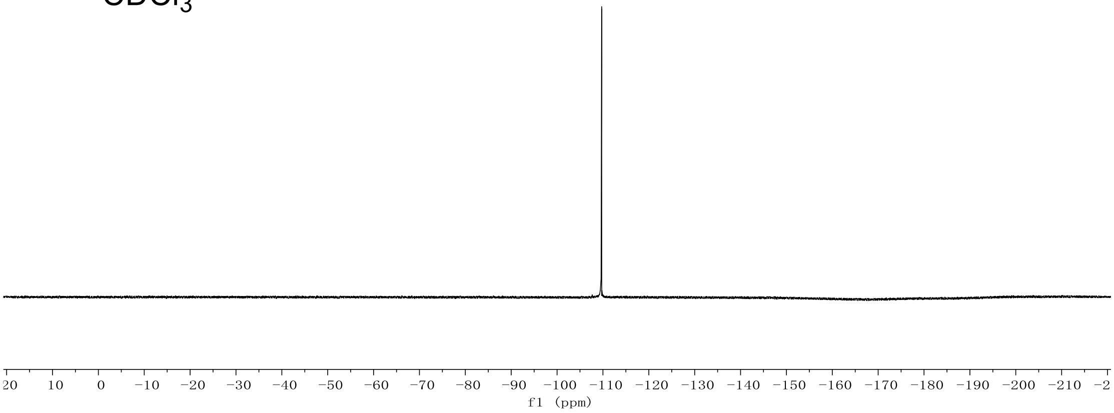
F
S
Me
F
3qa
1H NMR, 500 MHz
CDCl3
6.754
6.750
6.744
6.740
6.737
6.732
6.728
6.724
6.580
6.575
6.570
6.562
6.557
6.553
6.544
6.539
2.474
2.00
1.00
3.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)

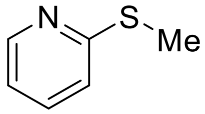


F
S
Me
F
3qa
13C NMR, 125 MHz
CDCl3
164.18
164.07
162.20
162.09
142.82
108.69
108.63
108.52
108.47
100.50
100.29
100.09
77.26
77.00
76.75
15.25
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

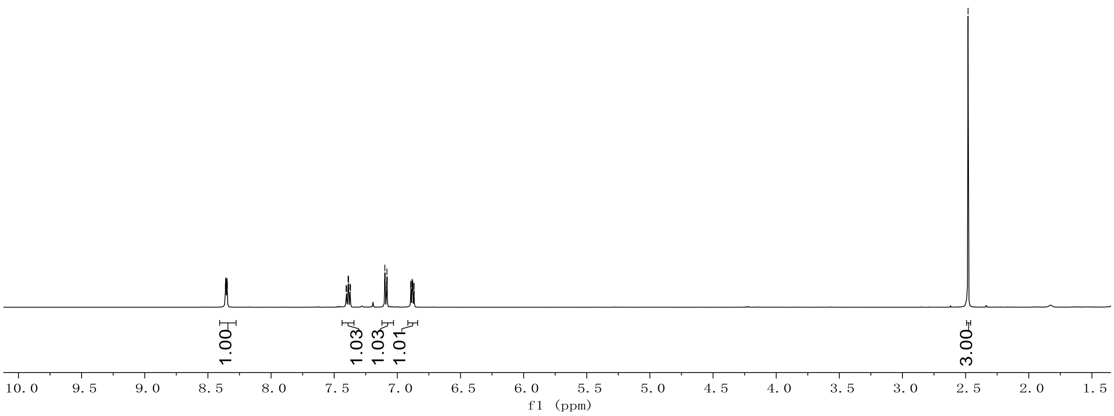


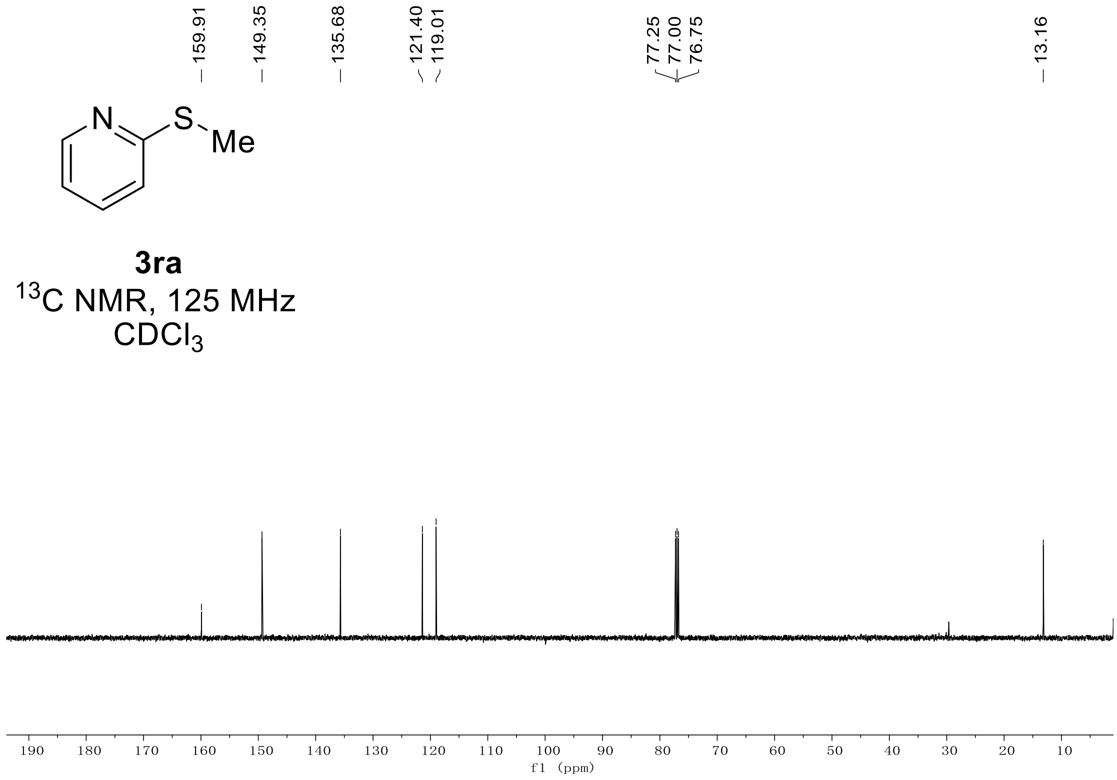
3qa
^{19}F NMR, 471 MHz
$CDCl_3$

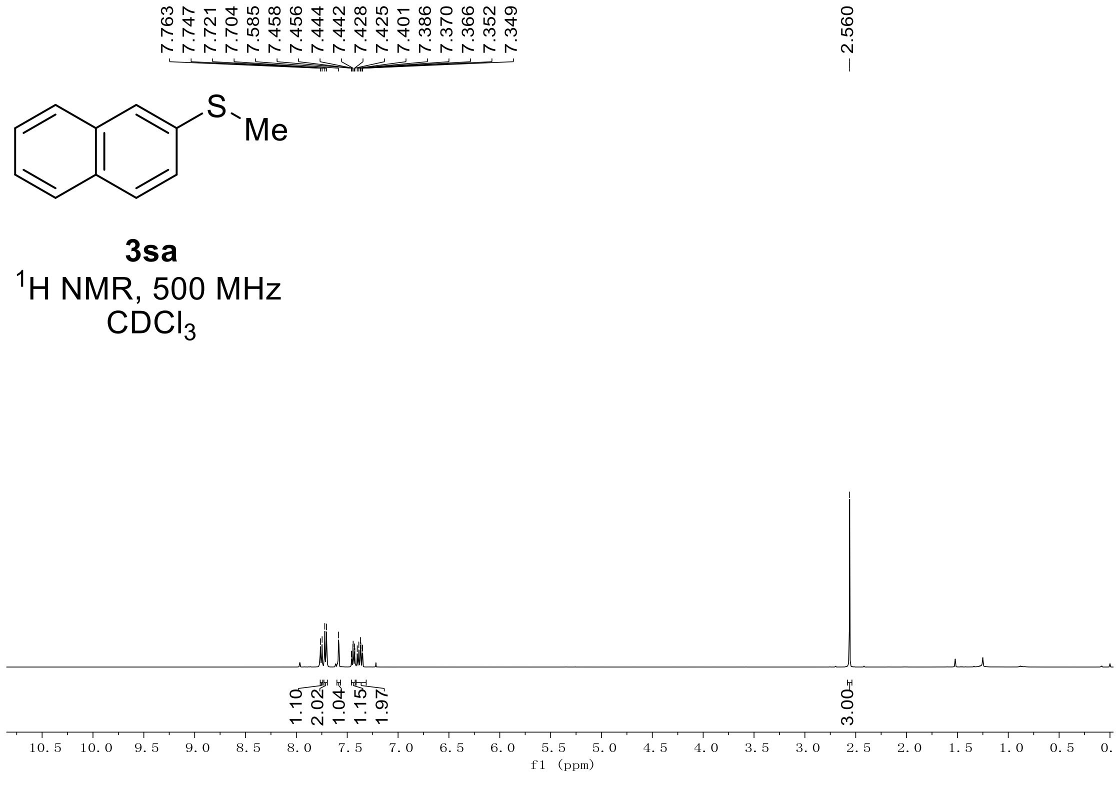


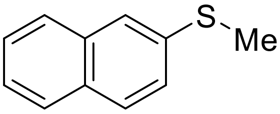


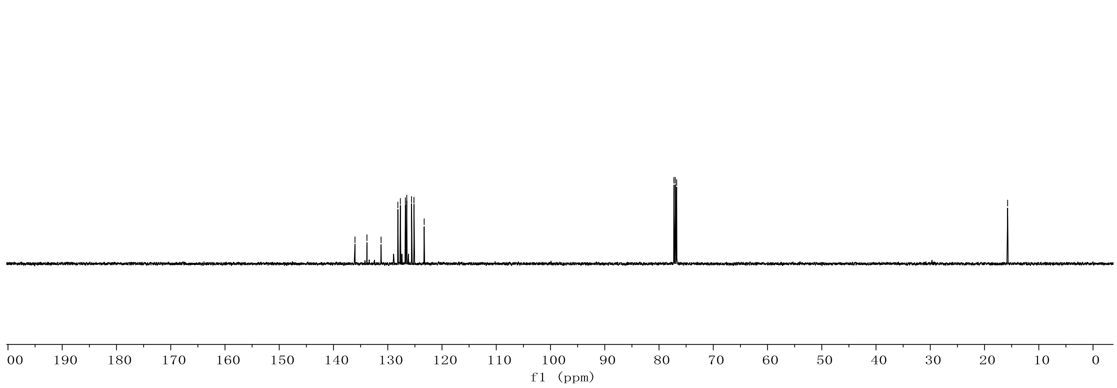
3ra
^{1}H NMR, 500 MHz
$CDCl_3$



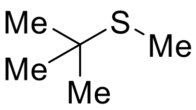

159.91
149.35
135.68
121.40
119.01
77.25
77.00
76.75
13.16
N
S
Me
3ra
13C NMR, 125 MHz
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

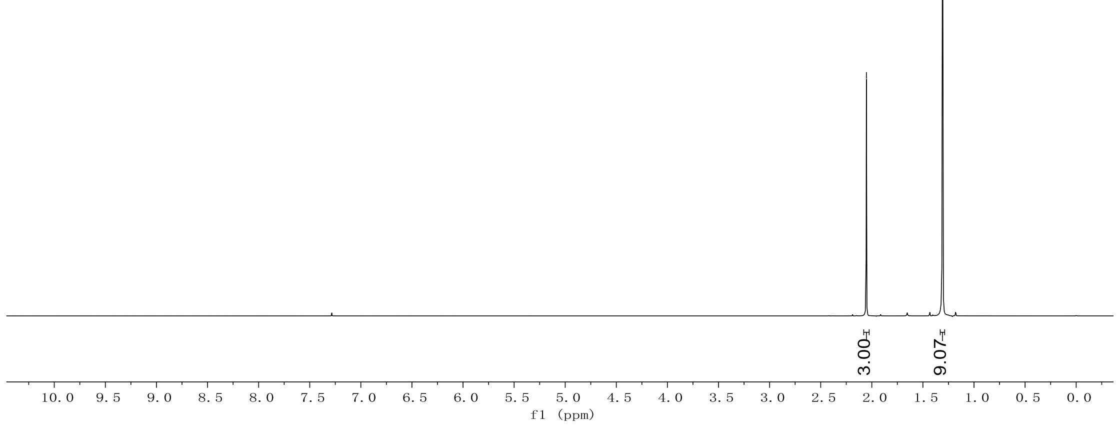

7.763
7.747
7.721
7.704
7.585
7.458
7.456
7.444
7.442
7.428
7.425
7.401
7.386
7.370
7.366
7.352
7.349
2.560
S
Me
3sa
1H NMR, 500 MHz
CDCl3
1.10
2.02
1.04
1.15
1.97
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)



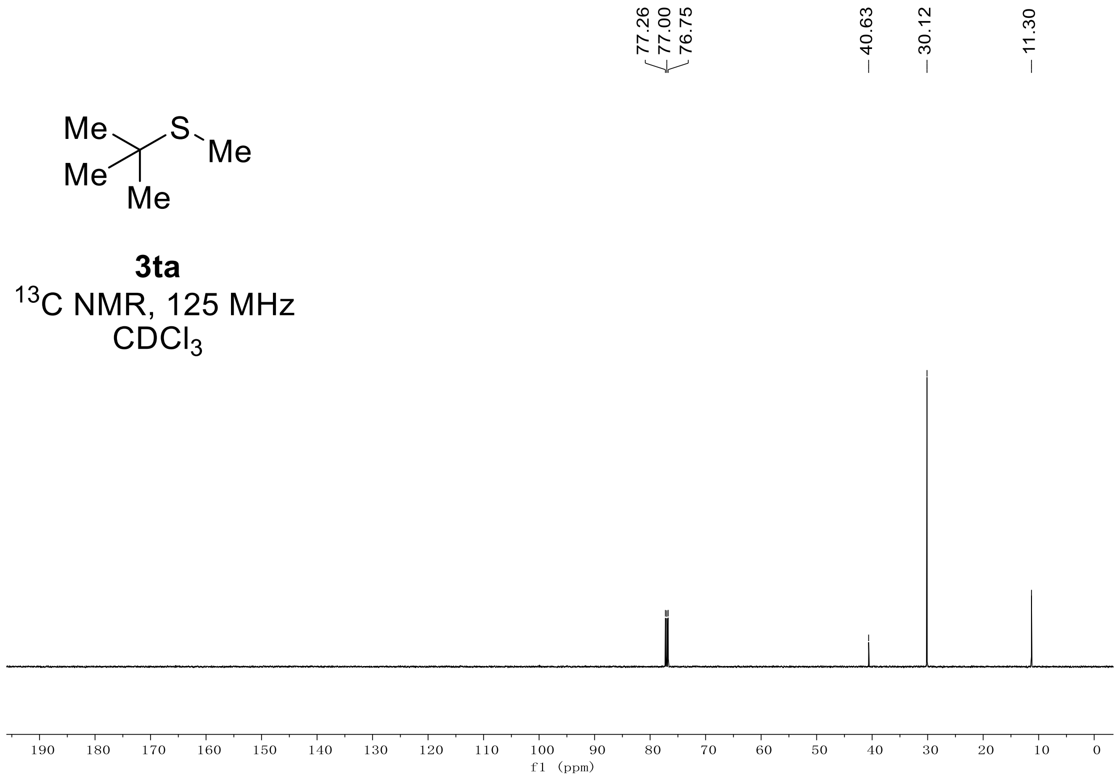


3sa
^{13}C NMR, 125 MHz
$CDCl_3$

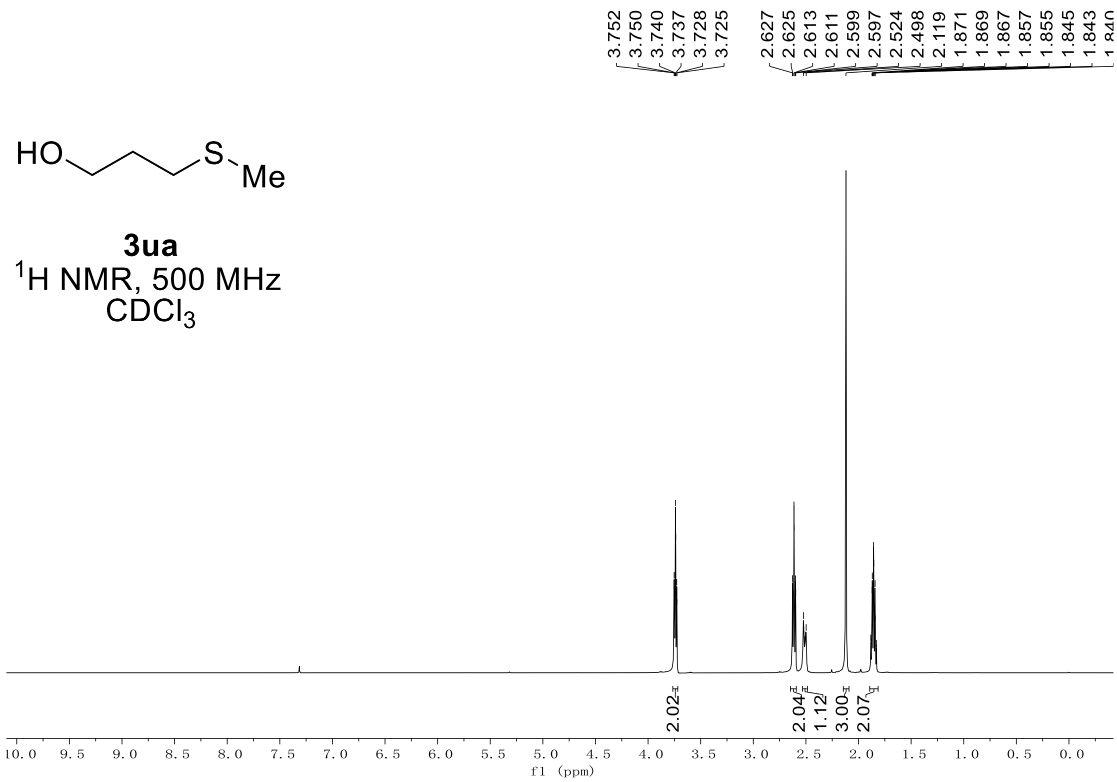




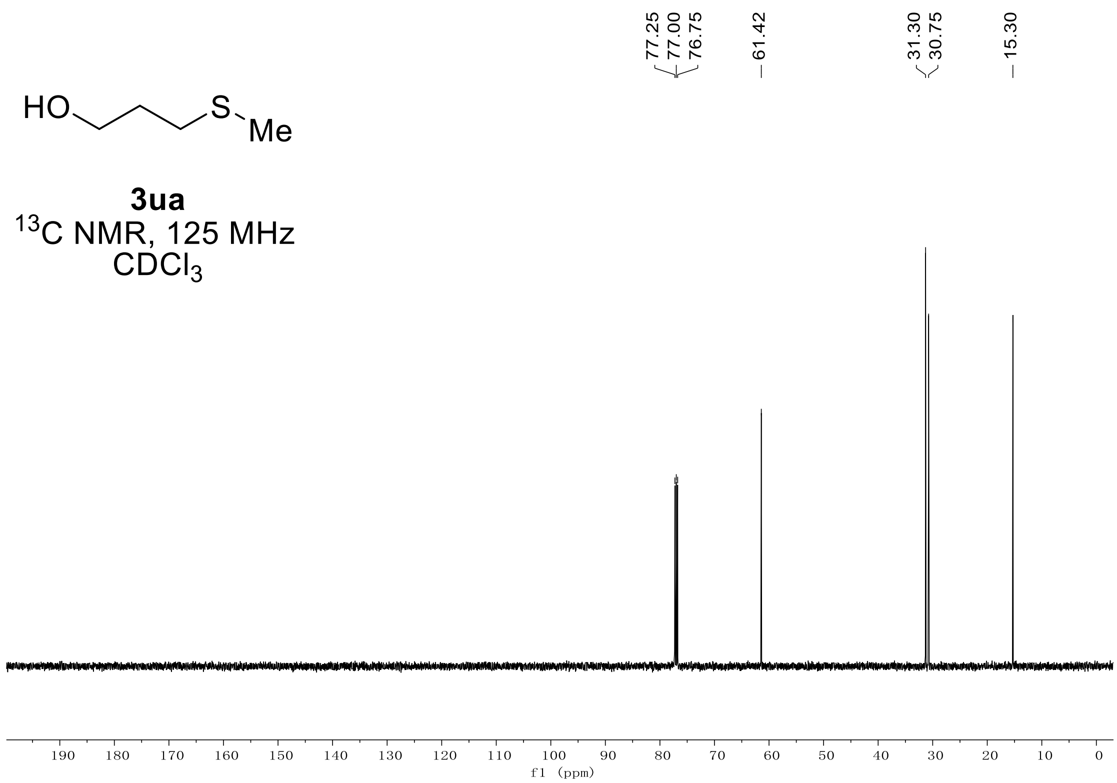
3ta
^{1}H NMR, 500 MHz
$CDCl_3$



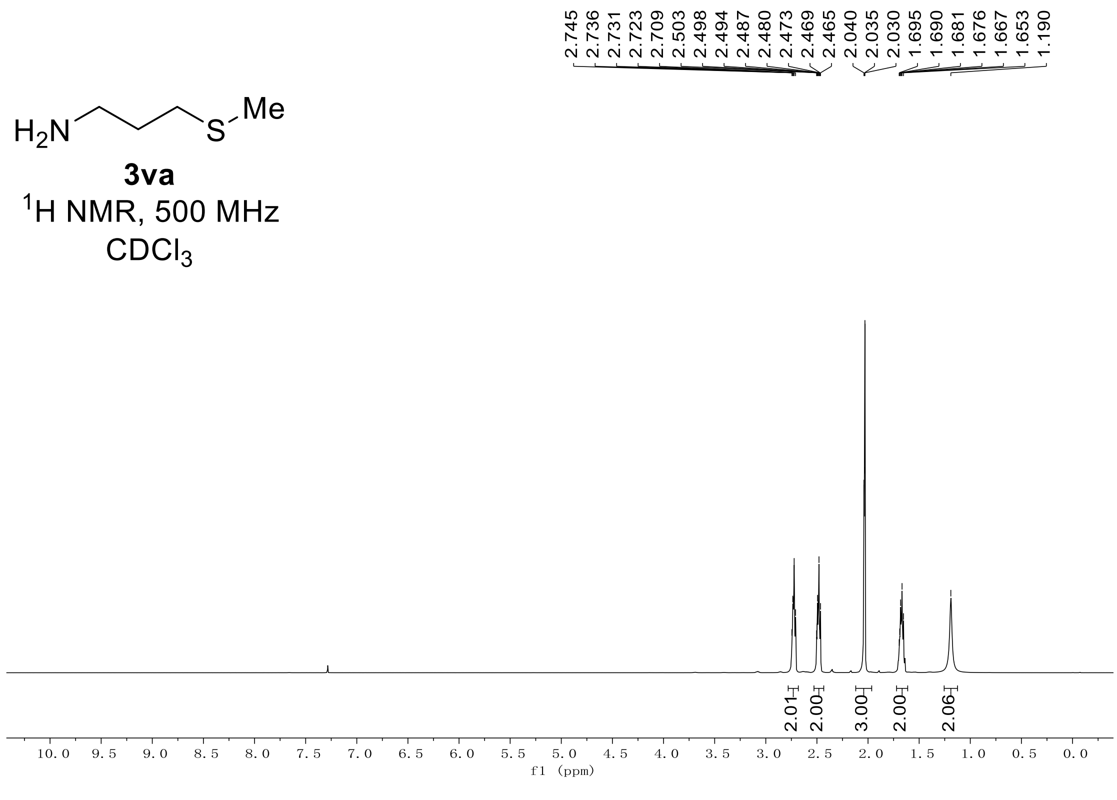
3ta
^{13}C NMR, 125 MHz
$CDCl_3$



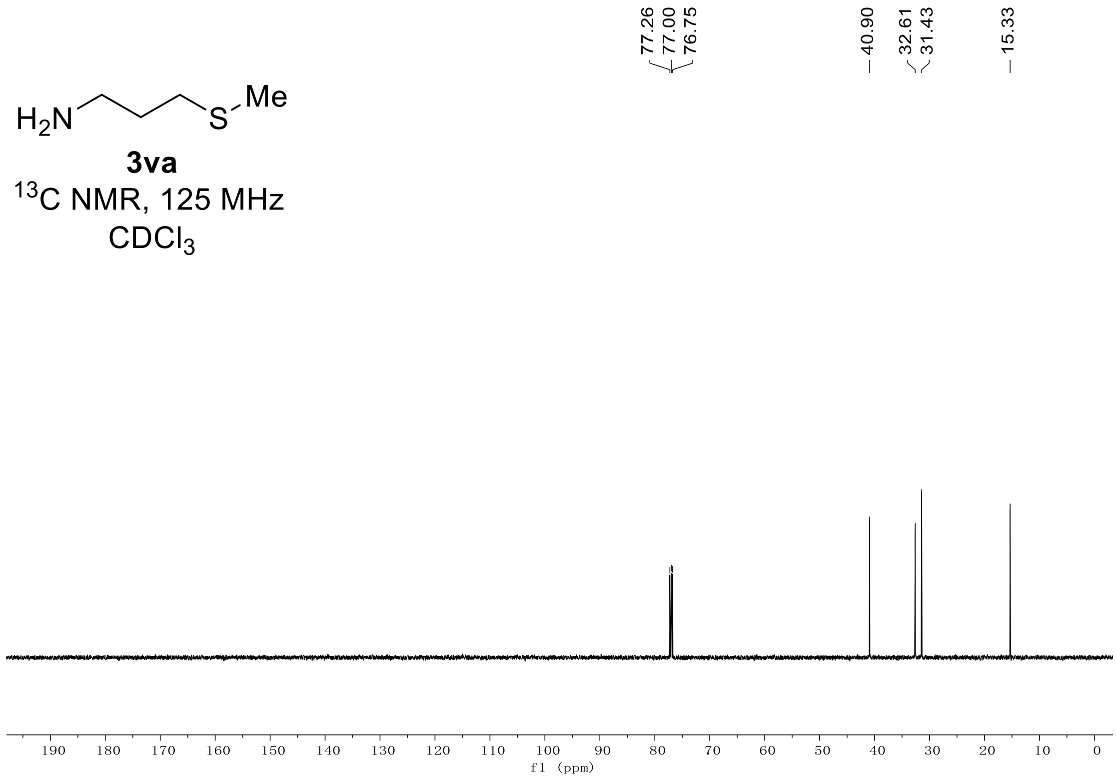
3ua
^{1}H NMR, 500 MHz
$CDCl_3$

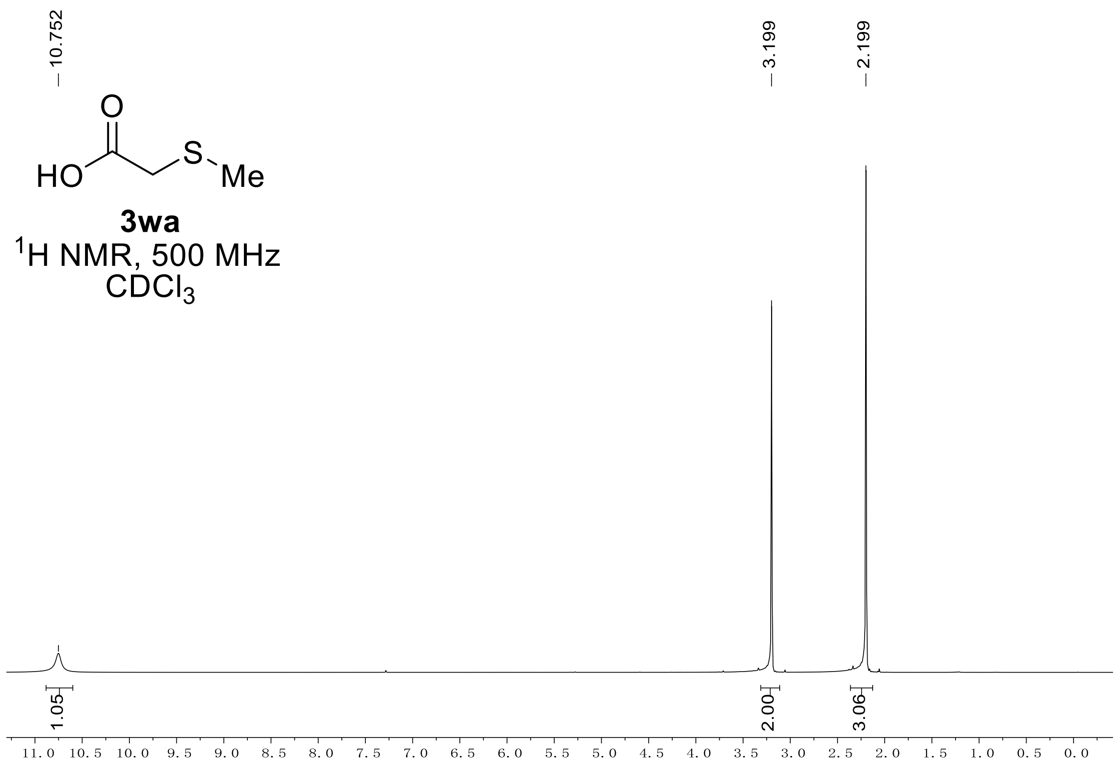


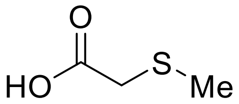
3ua
^{13}C NMR, 125 MHz
$CDCl_3$



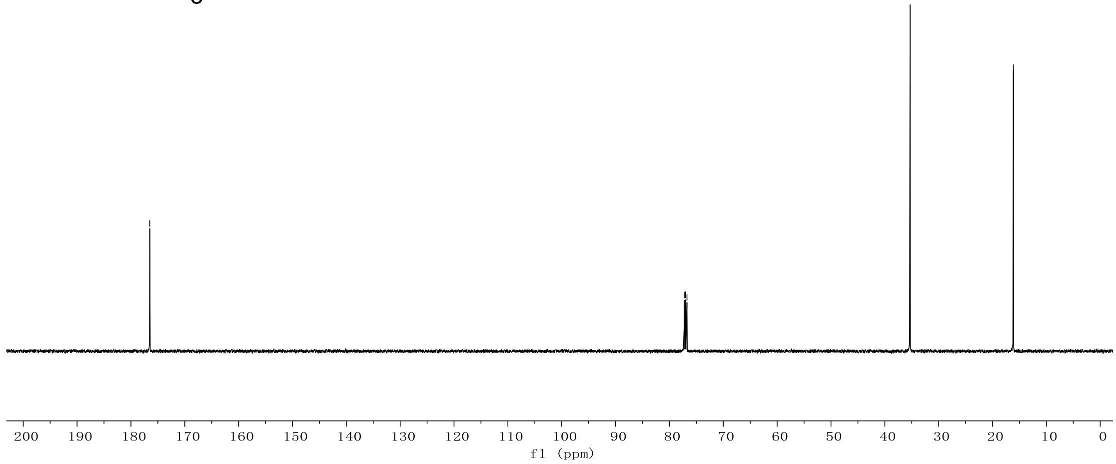
3va
^{1}H NMR, 500 MHz
$CDCl_3$

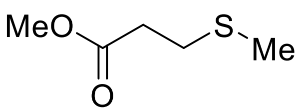

77.26
77.00
76.75
40.90
32.61
31.43
15.33
H2N
S
Me
3va
13C NMR, 125 MHz
CDCl3
f1 (ppm)


10.752
3.199
2.199
O
HO
S
Me
3wa
1H NMR, 500 MHz
CDCl3
1.05
2.00
3.06
f1 (ppm)

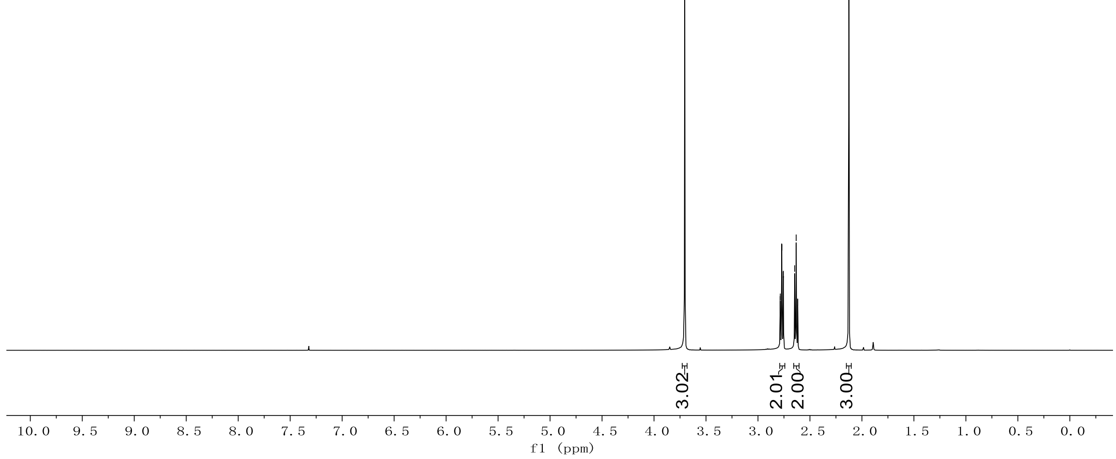


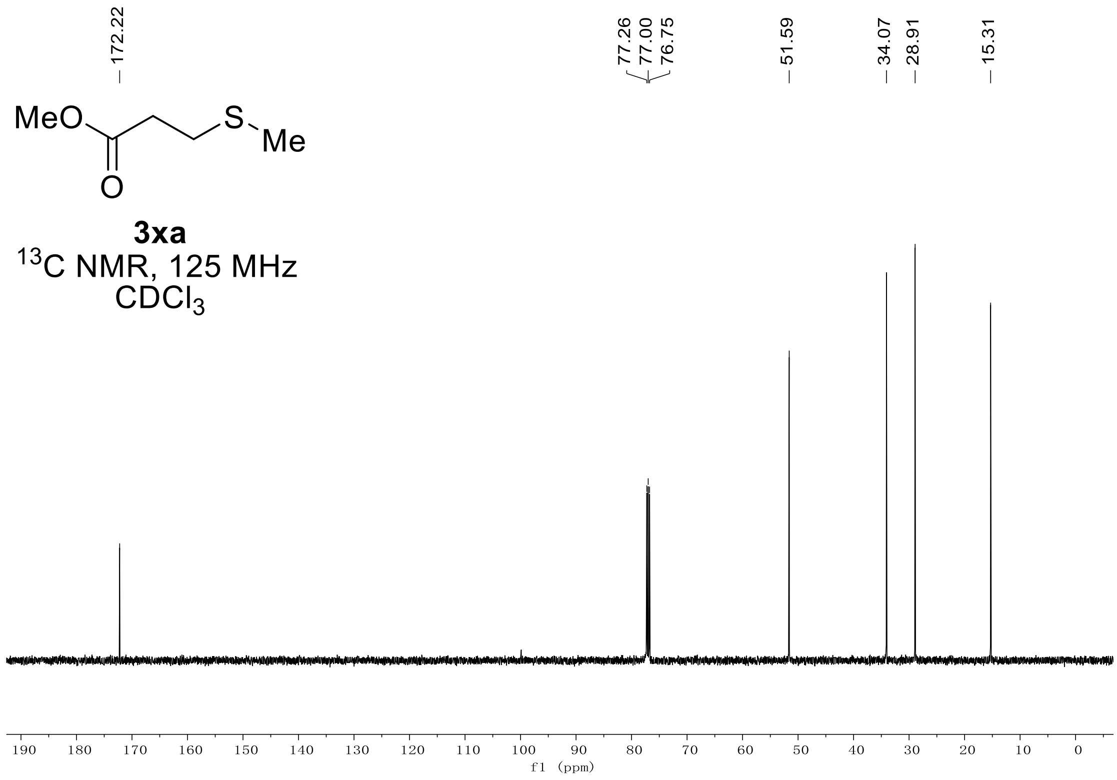
3wa
^{13}C NMR, 125 MHz
$CDCl_3$



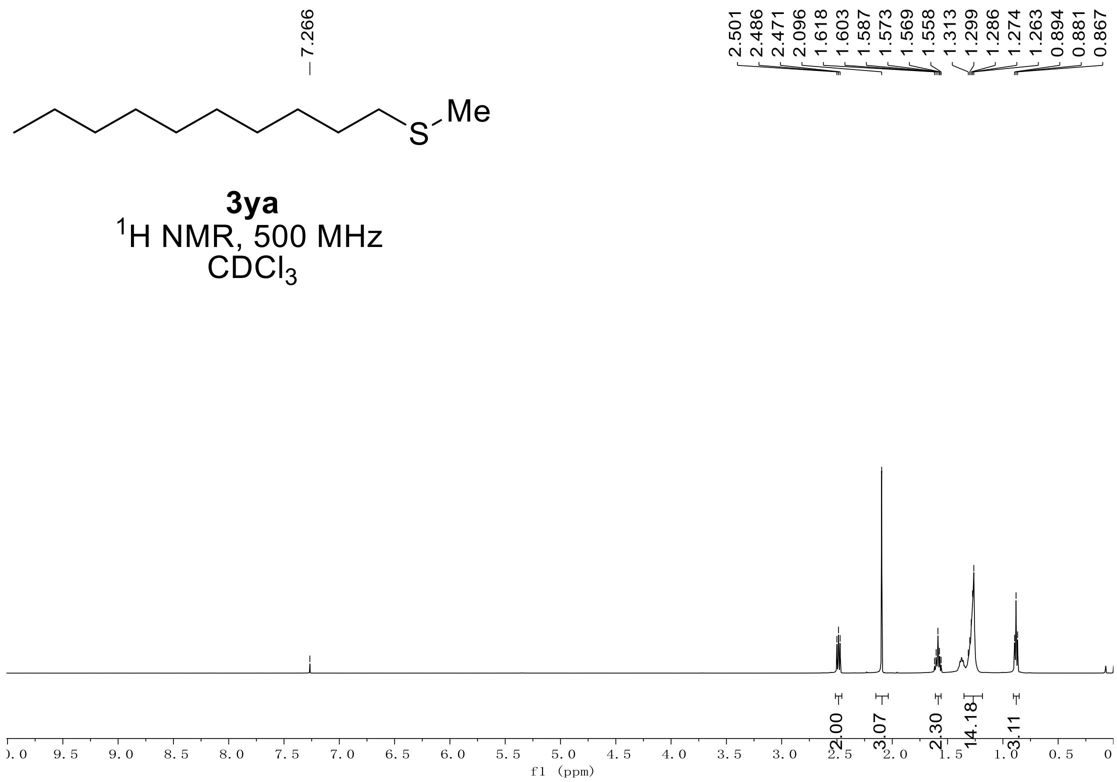


3xa
^{1}H NMR, 500 MHz
$CDCl_3$

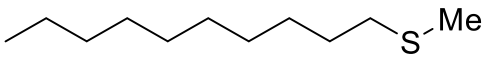




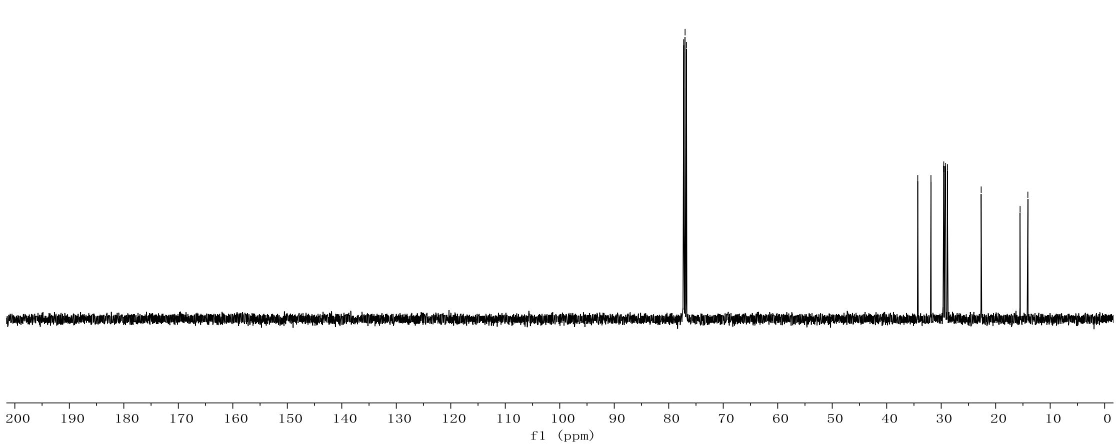
3xa
^{13}C NMR, 125 MHz
$CDCl_3$

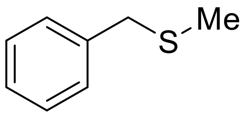


3ya
^{1}H NMR, 500 MHz
$CDCl_3$

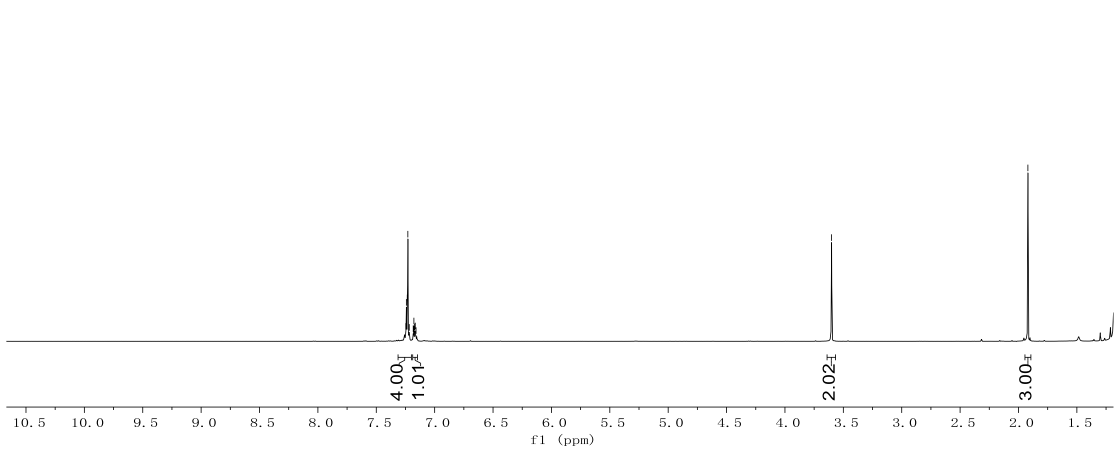


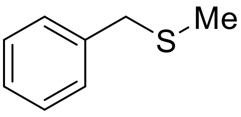
3ya
^{13}C NMR, 125 MHz
$CDCl_3$



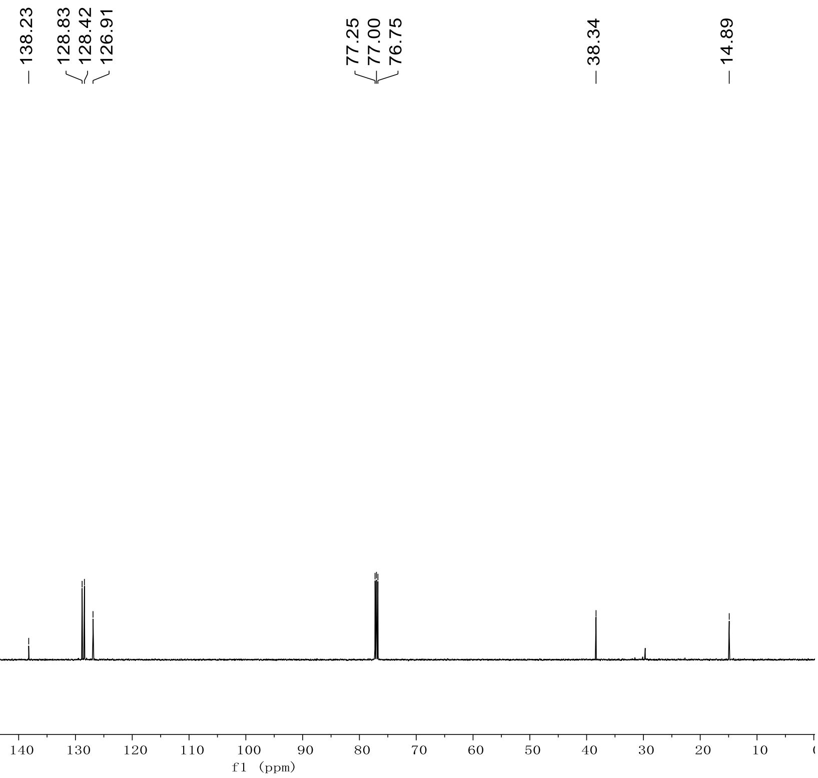


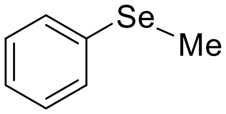
3za
^{1}H NMR, 500 MHz
$CDCl_3$



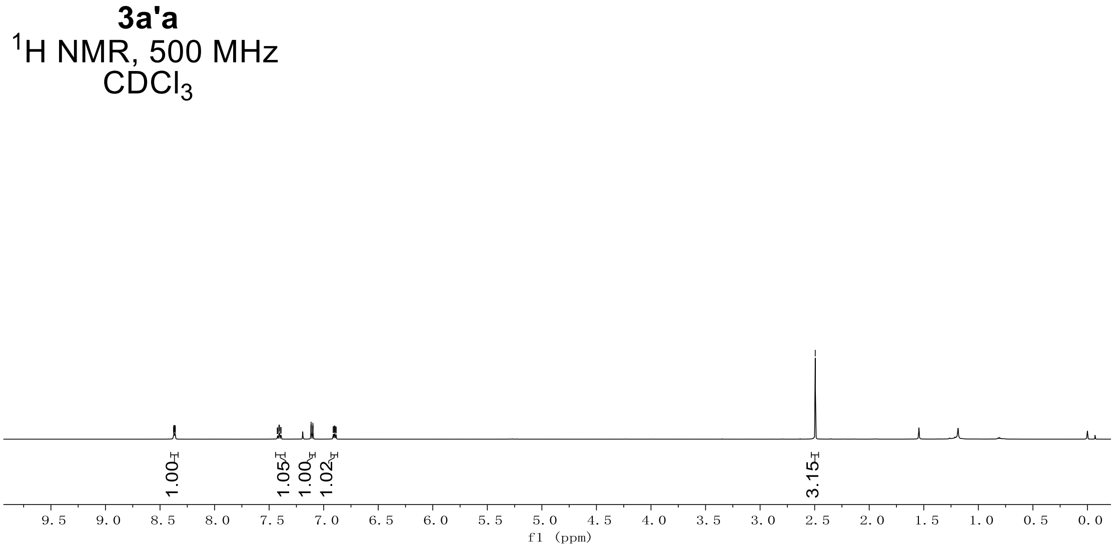


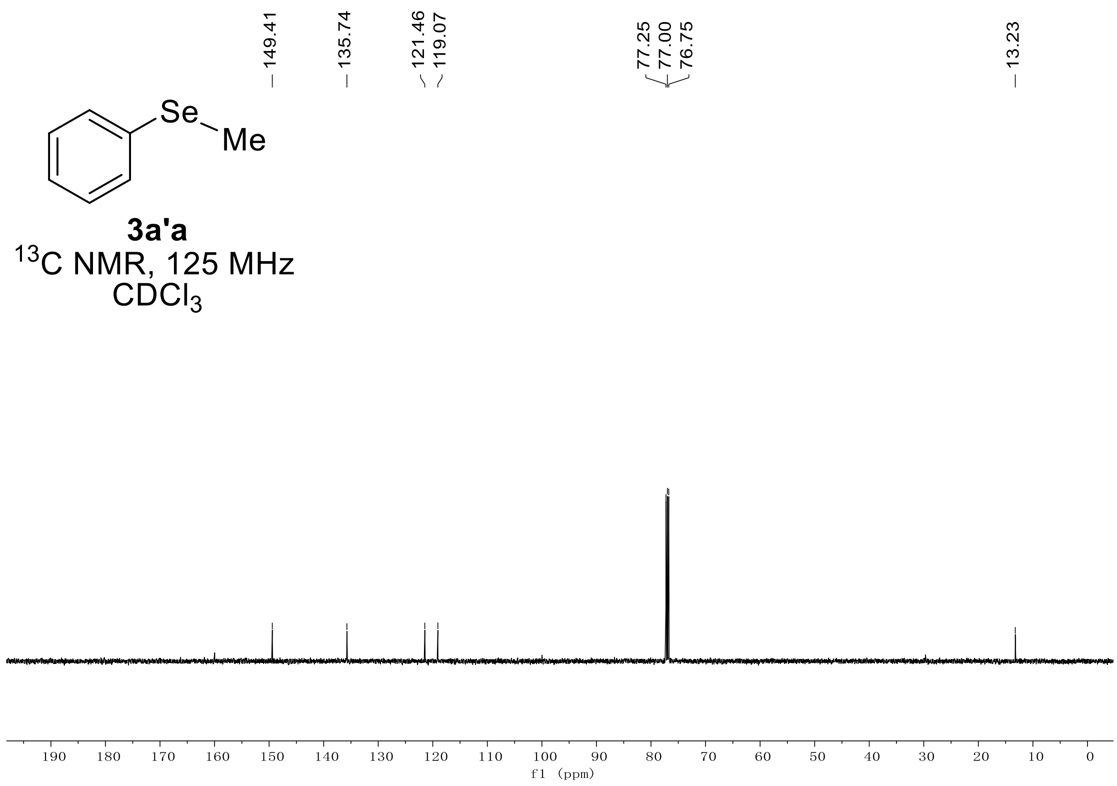
3za
^{13}C NMR, 125 MHz
$CDCl_3$

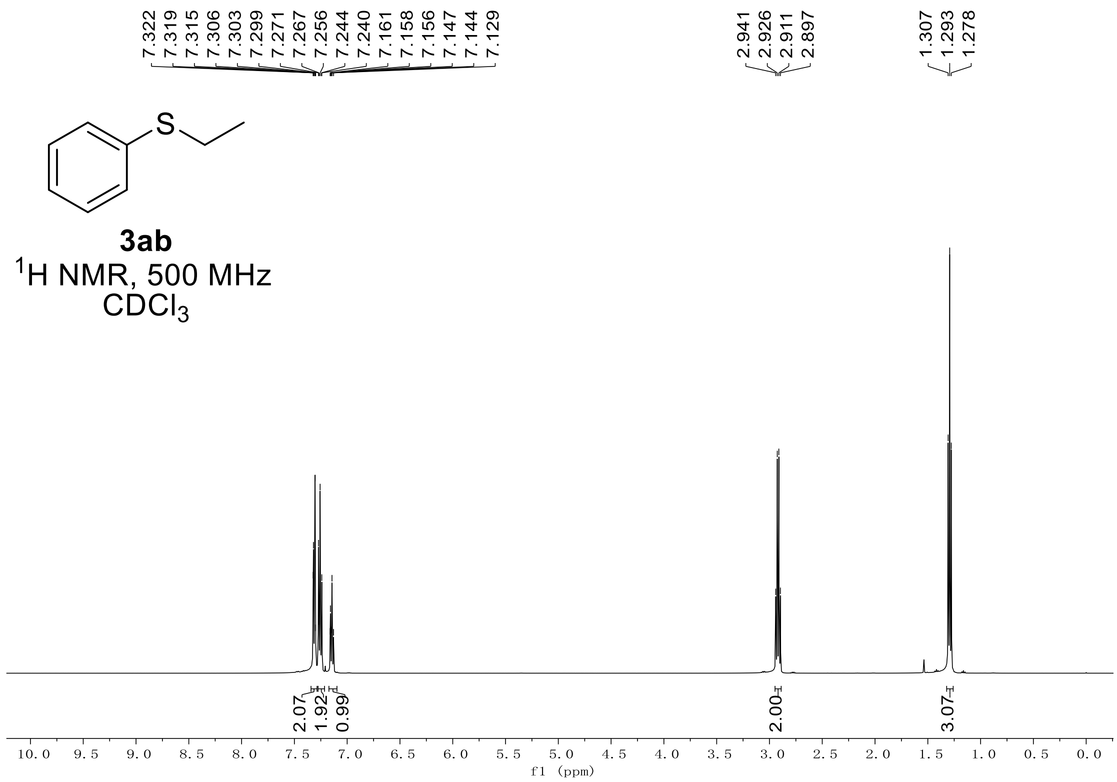




3a'a
^{1}H NMR, 500 MHz
$CDCl_3$



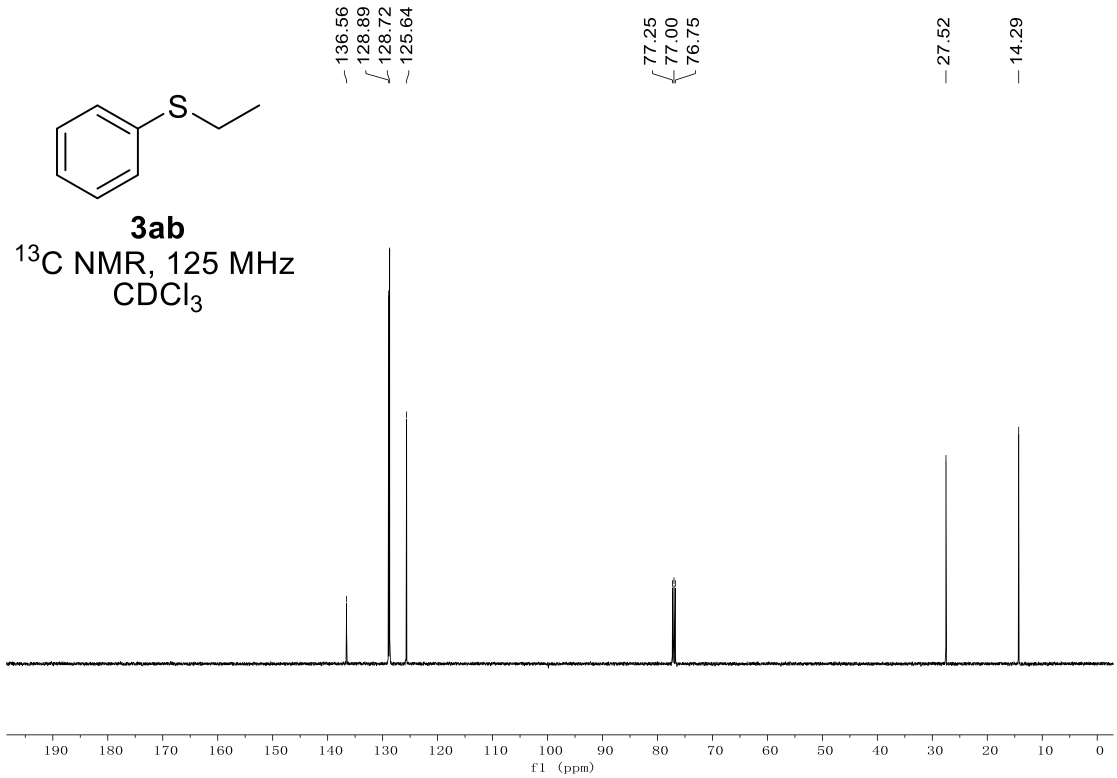

149.41
135.74
121.46
119.07
77.25
77.00
76.75
13.23
Se
Me
3a'a
13C NMR, 125 MHz
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.322
7.319
7.315
7.306
7.303
7.299
7.271
7.267
7.256
7.244
7.240
7.161
7.158
7.156
7.147
7.144
7.129
2.941
2.926
2.911
2.897
1.307
1.293
1.278
S
3ab
1H NMR, 500 MHz
CDCl3
2.07
1.92
0.99
2.00
3.07
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)

3ab

^{13}C NMR, 125 MHz

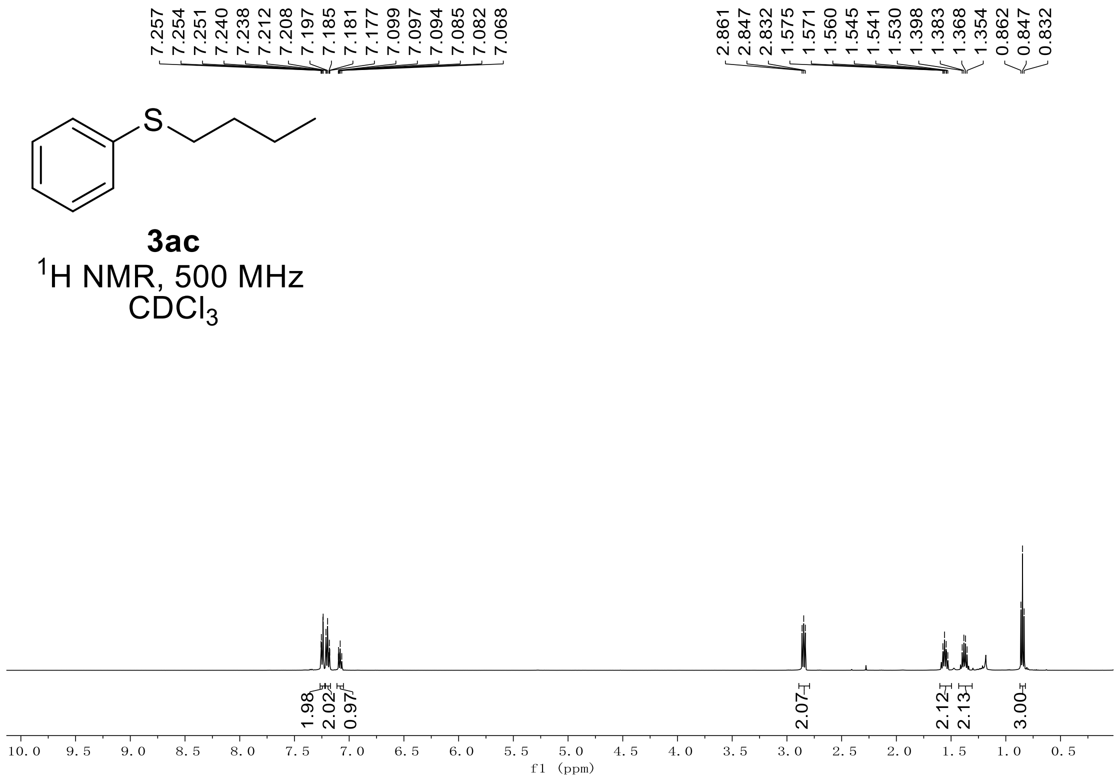
$CDCl_3$

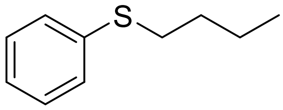


3ac

^{1}H NMR, 500 MHz

$CDCl_3$

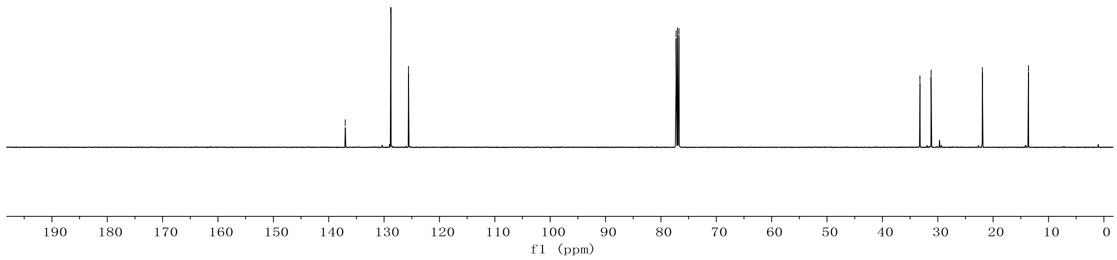


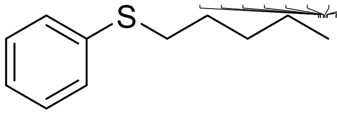


3ac

^{13}C NMR, 500 MHz

$CDCl_3$

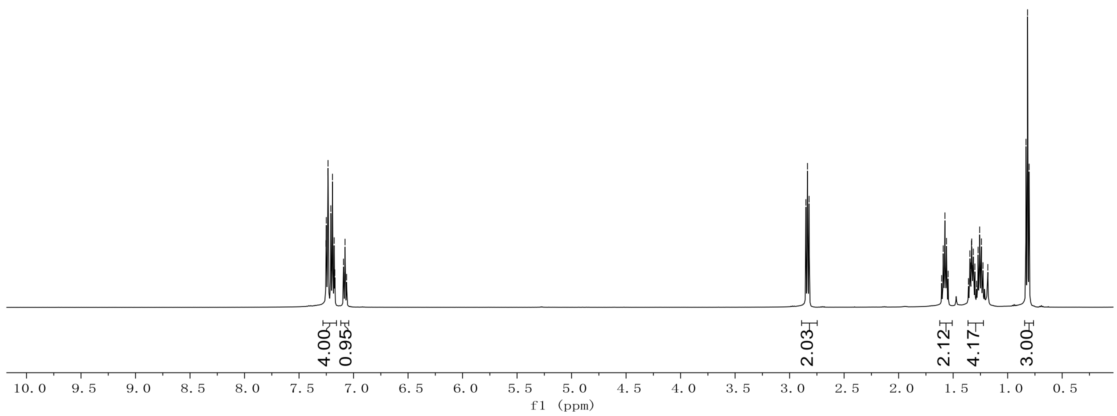


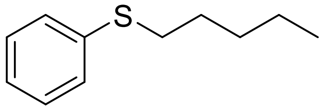


3ad

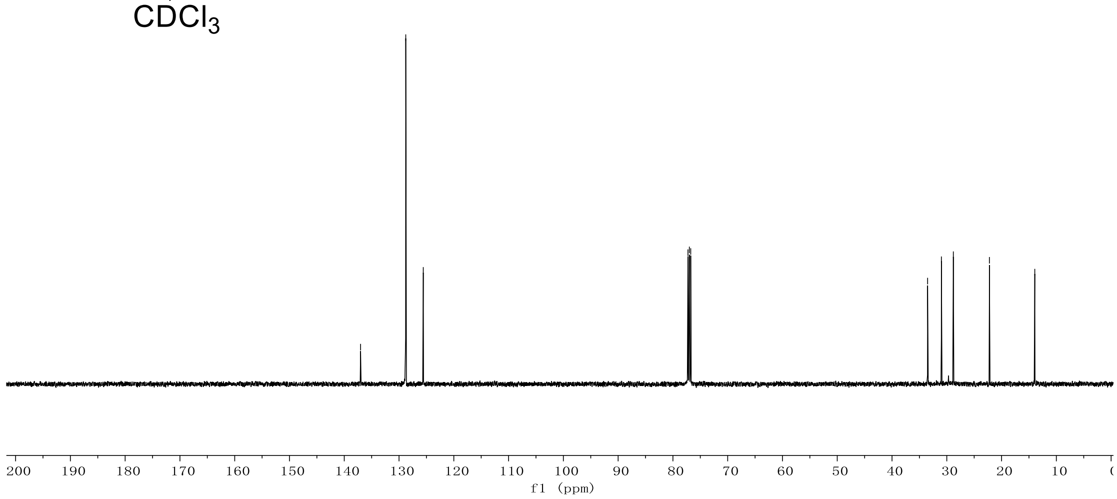
^{1}H NMR, 500 MHz

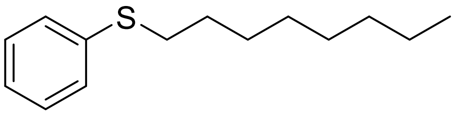
$CDCl_3$



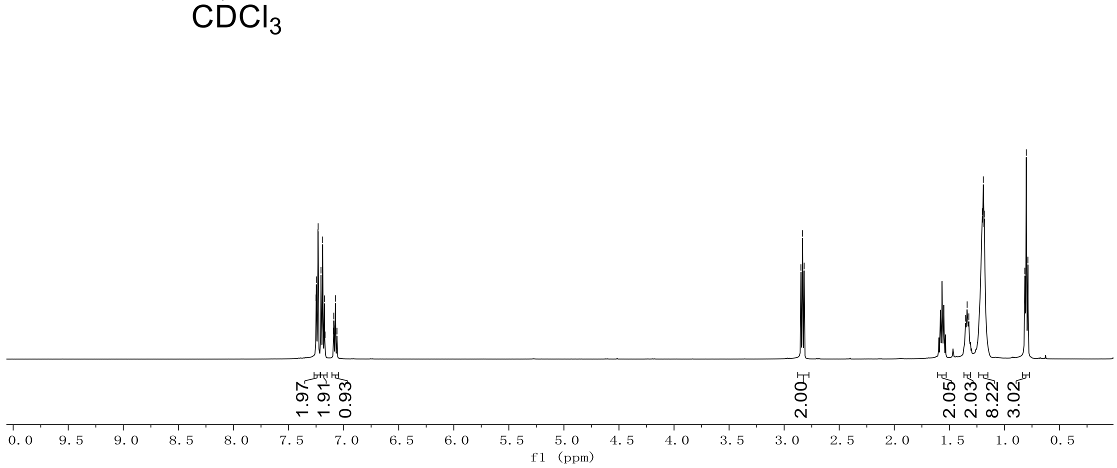


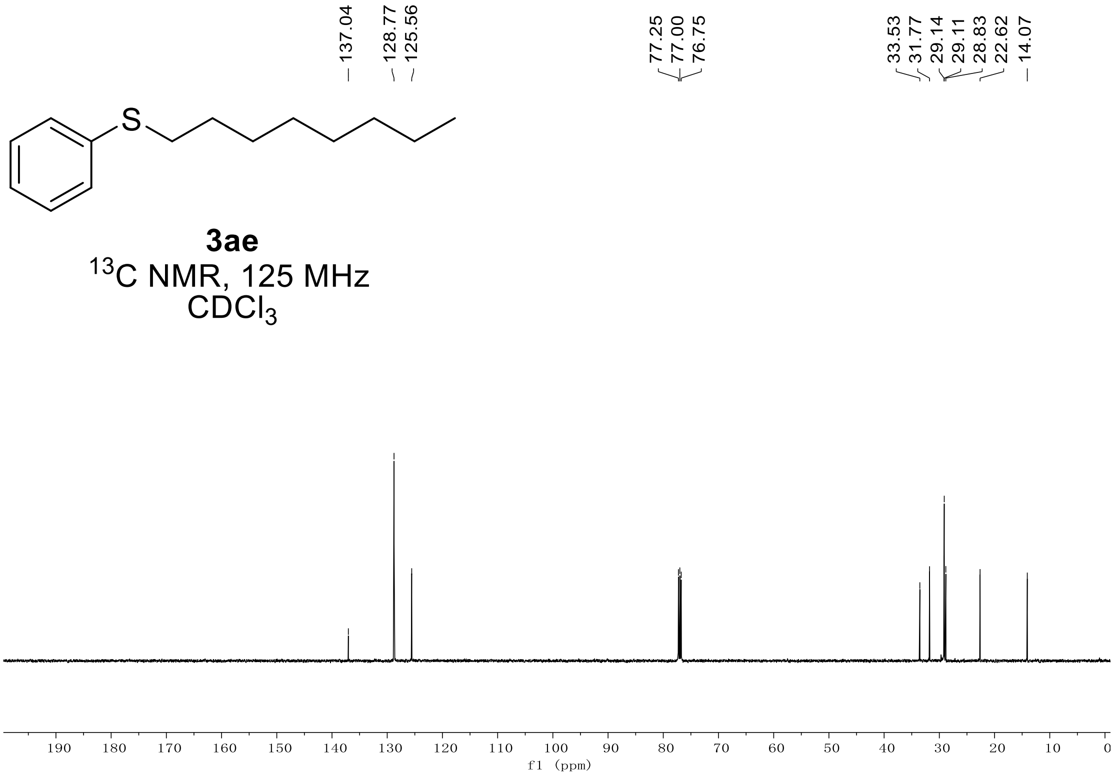
3ad
^{13}C NMR, 125 MHz
$CDCl_3$



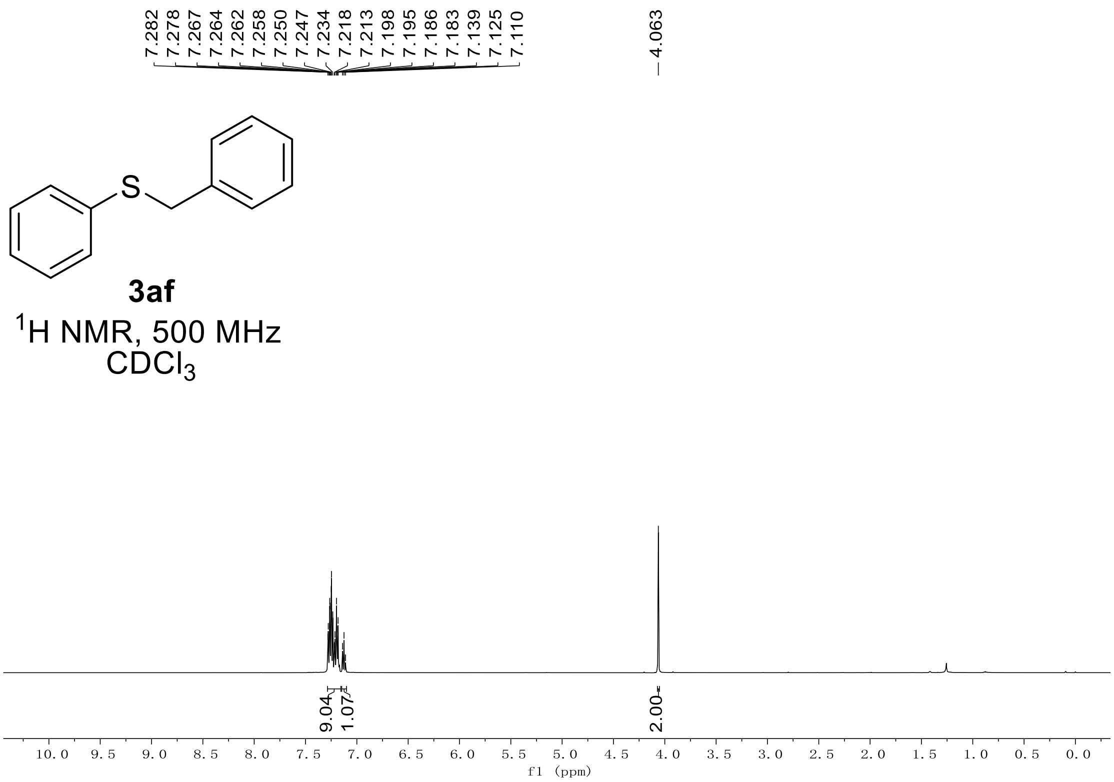


3ae
^{1}H NMR, 500 MHz
$CDCl_3$

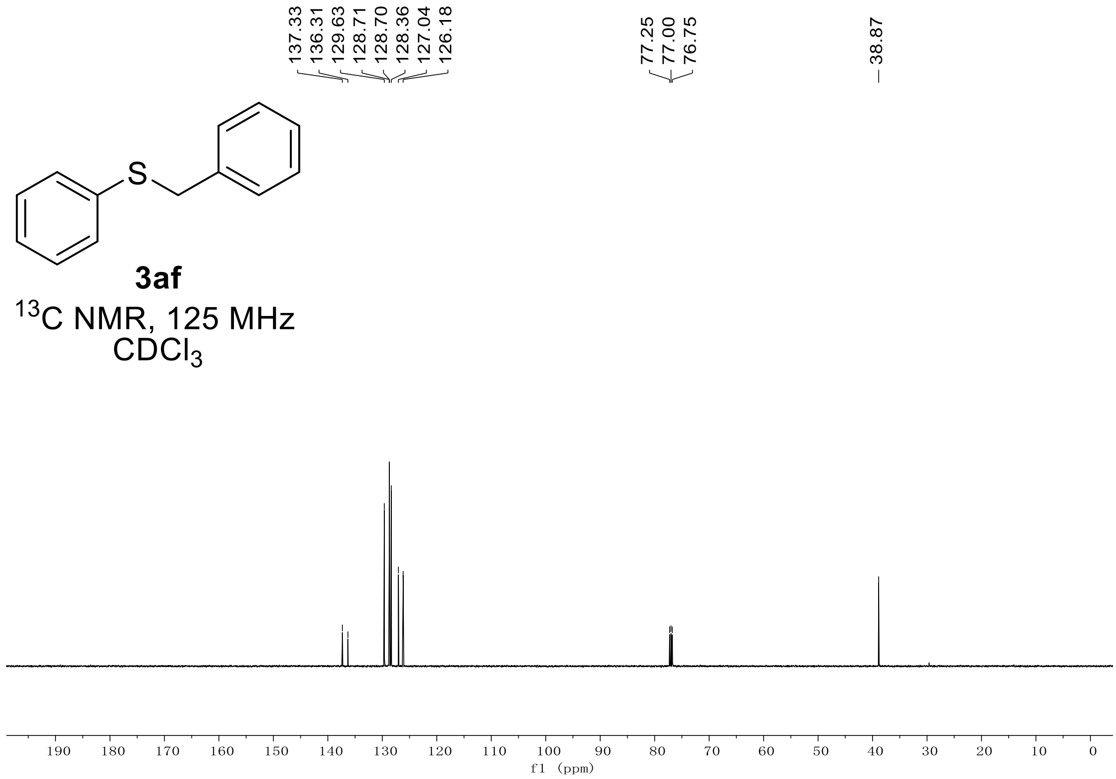


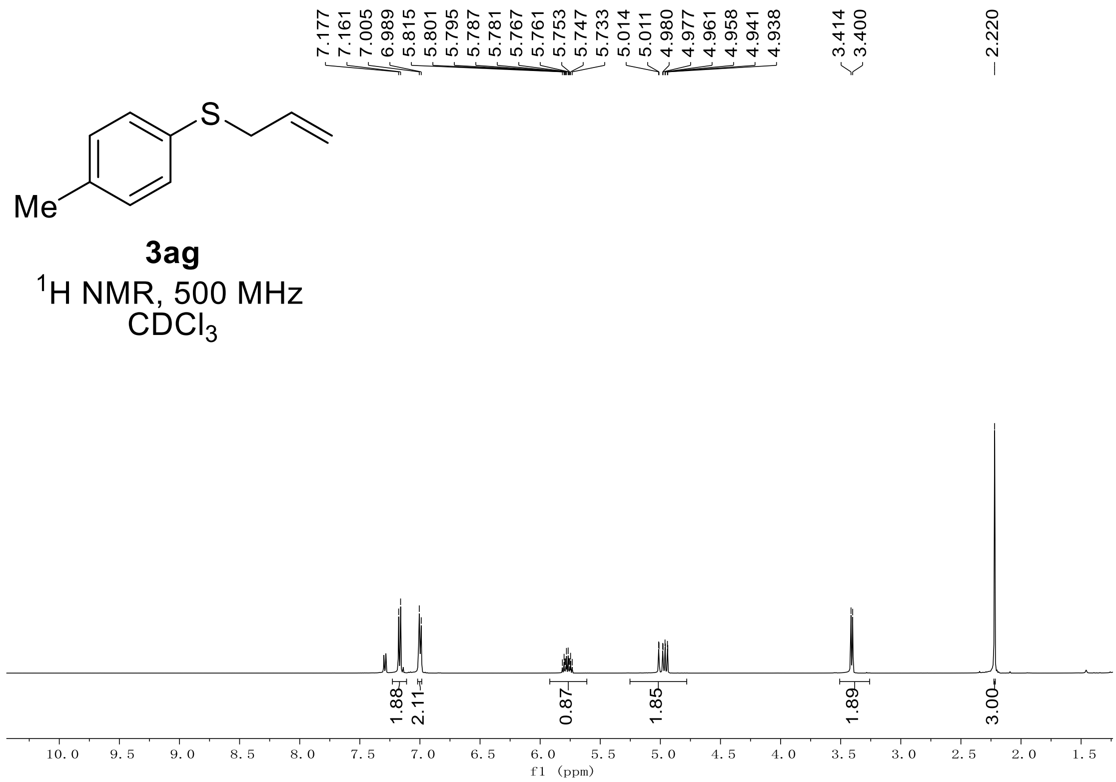


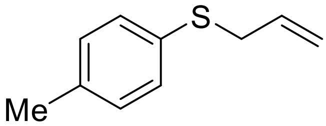
3ae
^{13}C NMR, 125 MHz
$CDCl_3$



3af
^{1}H NMR, 500 MHz
$CDCl_3$


137.33
136.31
129.63
128.71
128.70
128.36
127.04
126.18
77.25
77.00
76.75
38.87
S
3af
13C NMR, 125 MHz
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

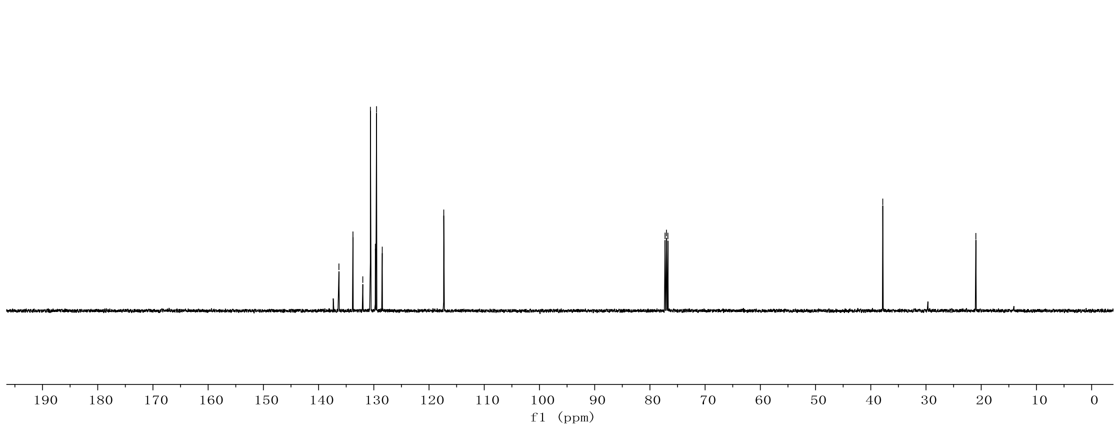

7.177
7.161
7.005
6.989
5.815
5.801
5.795
5.787
5.781
5.767
5.761
5.753
5.747
5.733
5.014
5.011
4.980
4.977
4.961
4.958
4.941
4.938
3.414
3.400
2.220
S
Me
3ag
1H NMR, 500 MHz
CDCl3
1.88
2.11
0.87
1.85
1.89
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

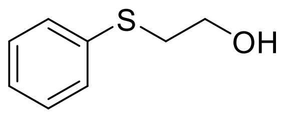


3ag

^{13}C NMR, 125 MHz

$CDCl_3$

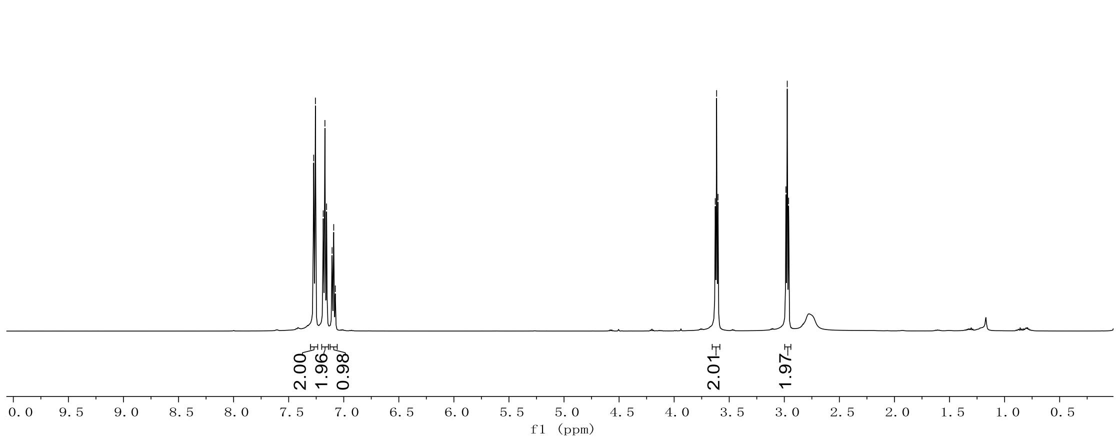


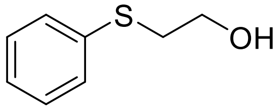


3ah

^{1}H NMR, 500 MHz

$CDCl_3$

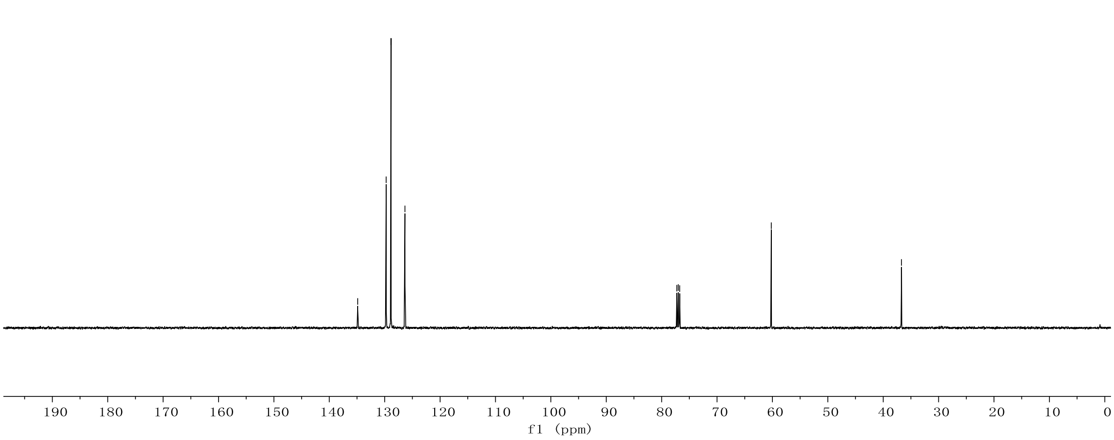


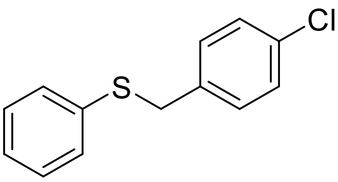


3ah

^{13}C NMR, 125 MHz

$CDCl_3$

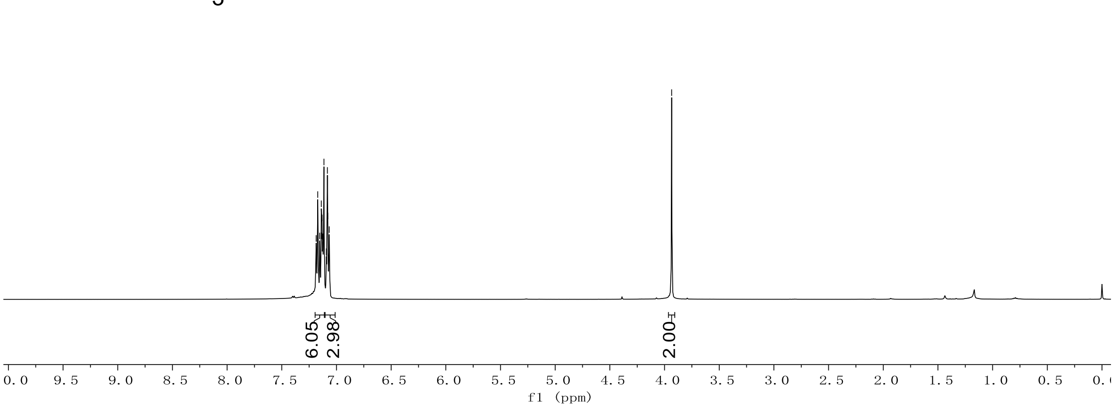


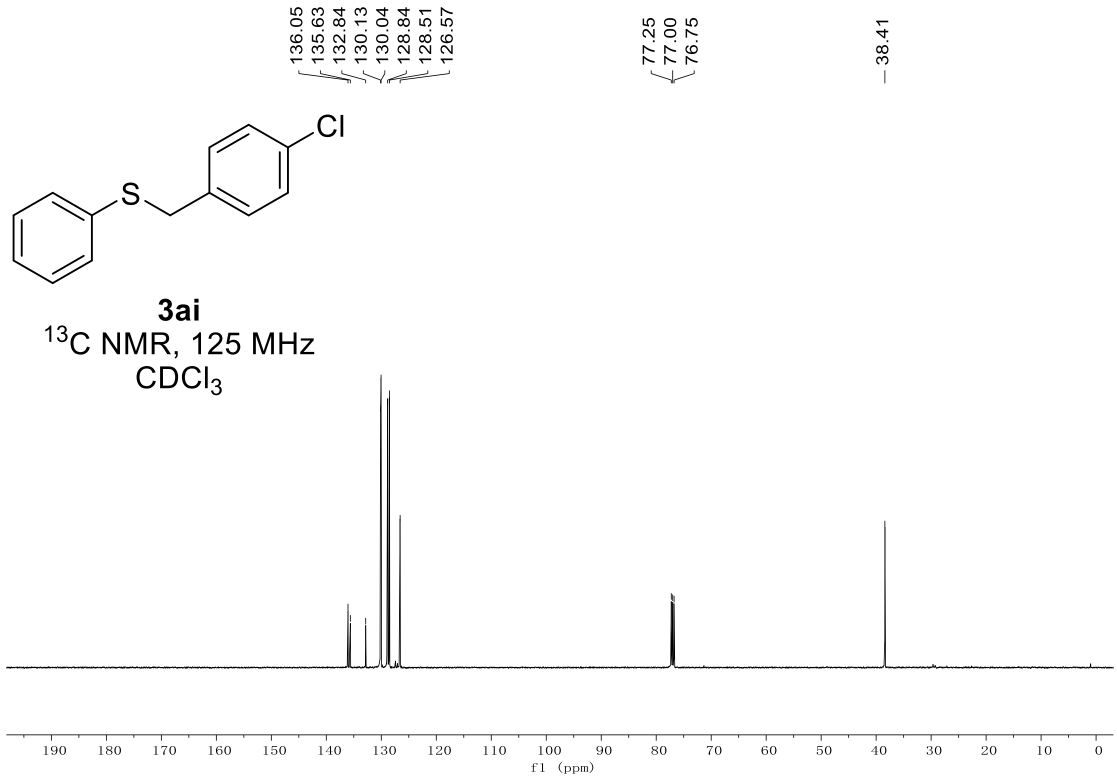


3ai

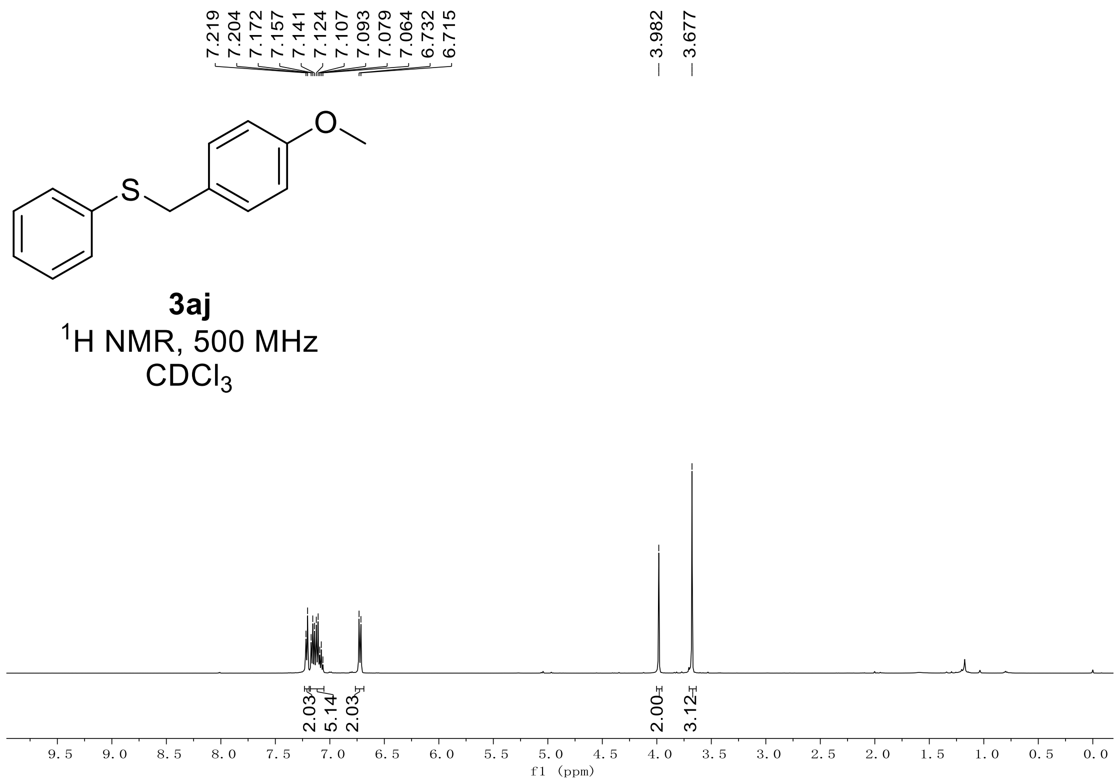
^{1}H NMR, 500 MHz

$CDCl_3$

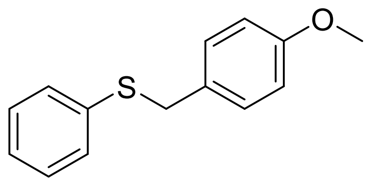




3ai
^{13}C NMR, 125 MHz
$CDCl_3$



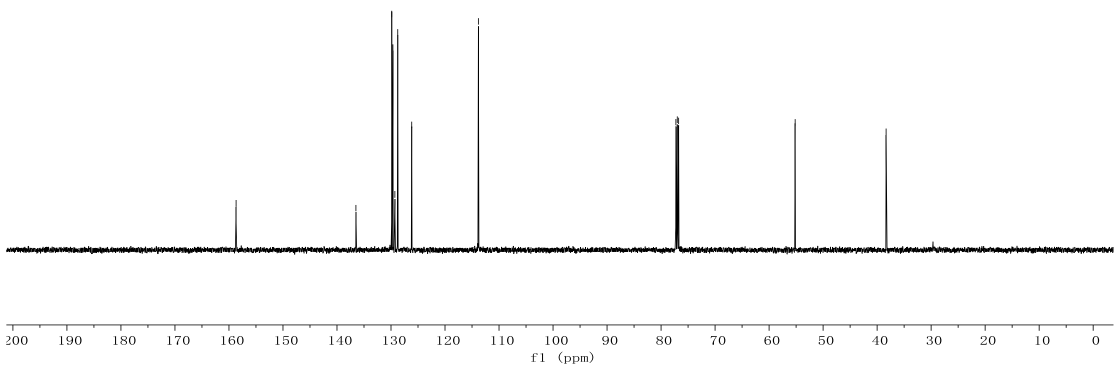
3aj
^{1}H NMR, 500 MHz
$CDCl_3$

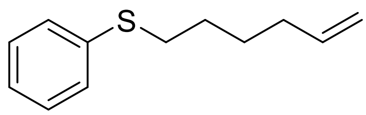


3aj

^{13}C NMR, 125 MHz

$CDCl_3$

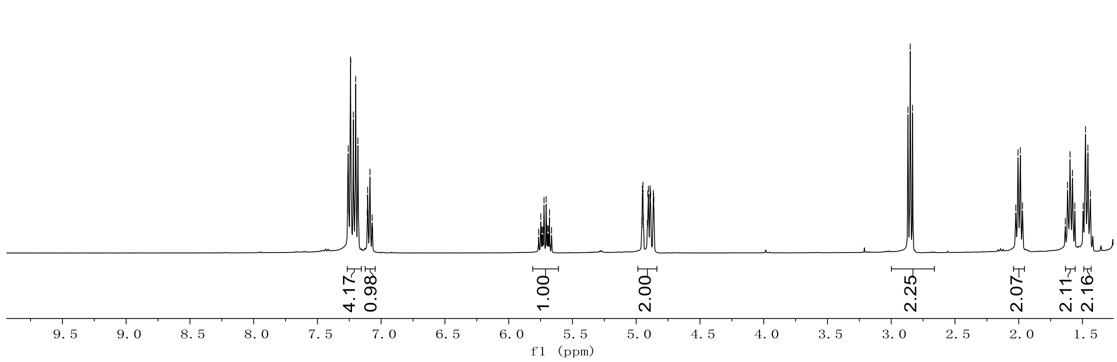


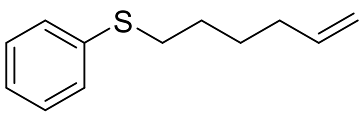


3ak

^{1}H NMR, 500 MHz

$CDCl_3$

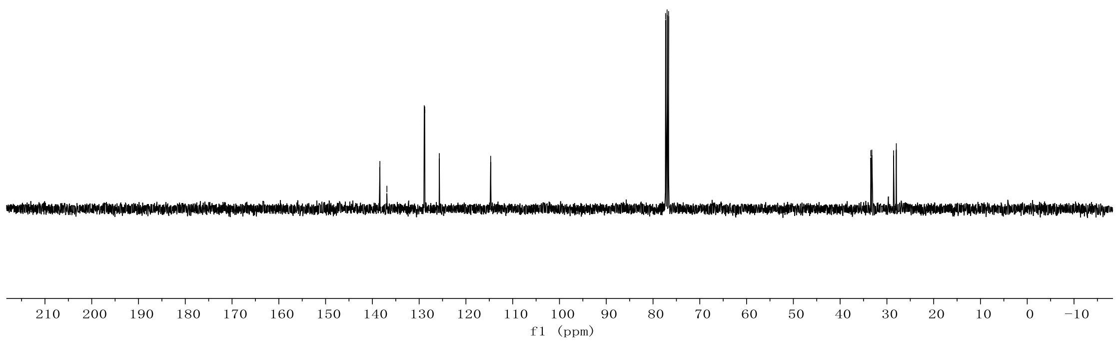


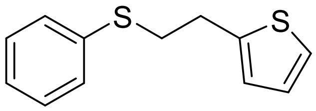


3ak

^{13}C NMR, 125 MHz

$CDCl_3$

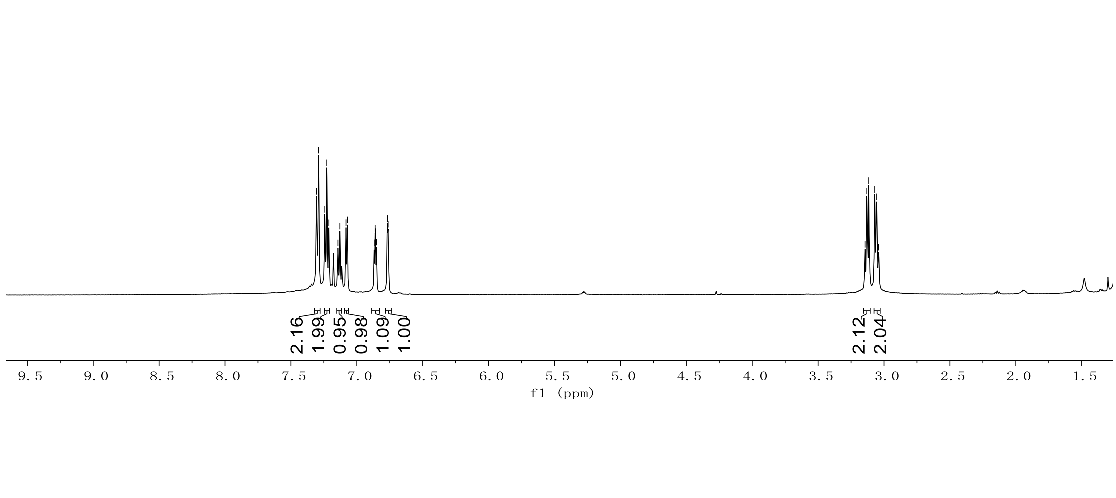


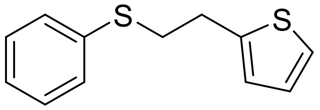


3al

^{1}H NMR, 500 MHz

$CDCl_3$

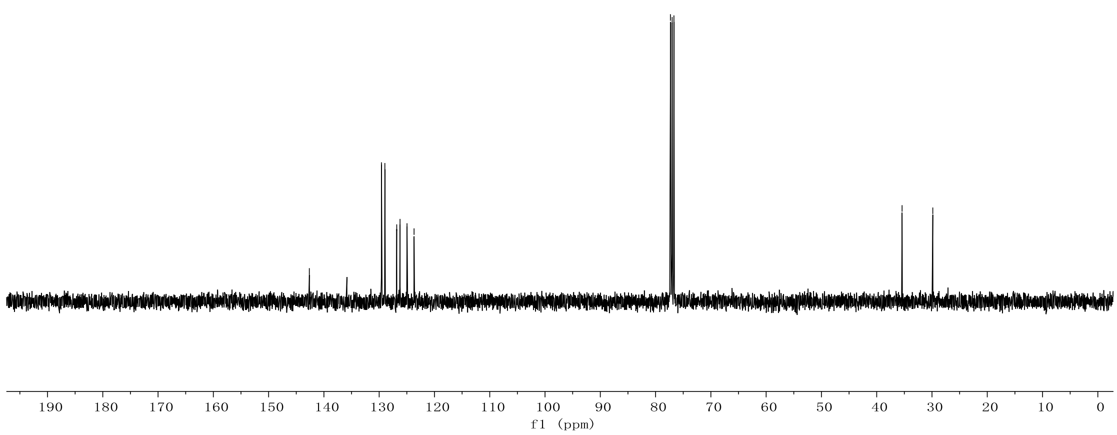


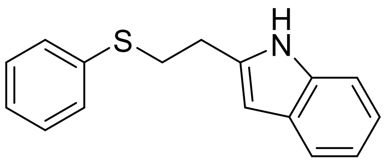


3al

^{13}C NMR, 125 MHz

$CDCl_3$

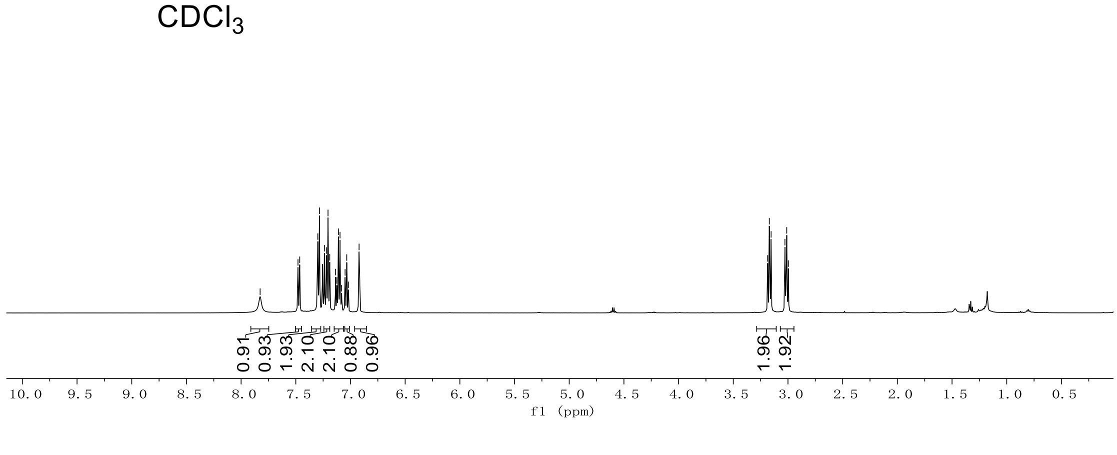


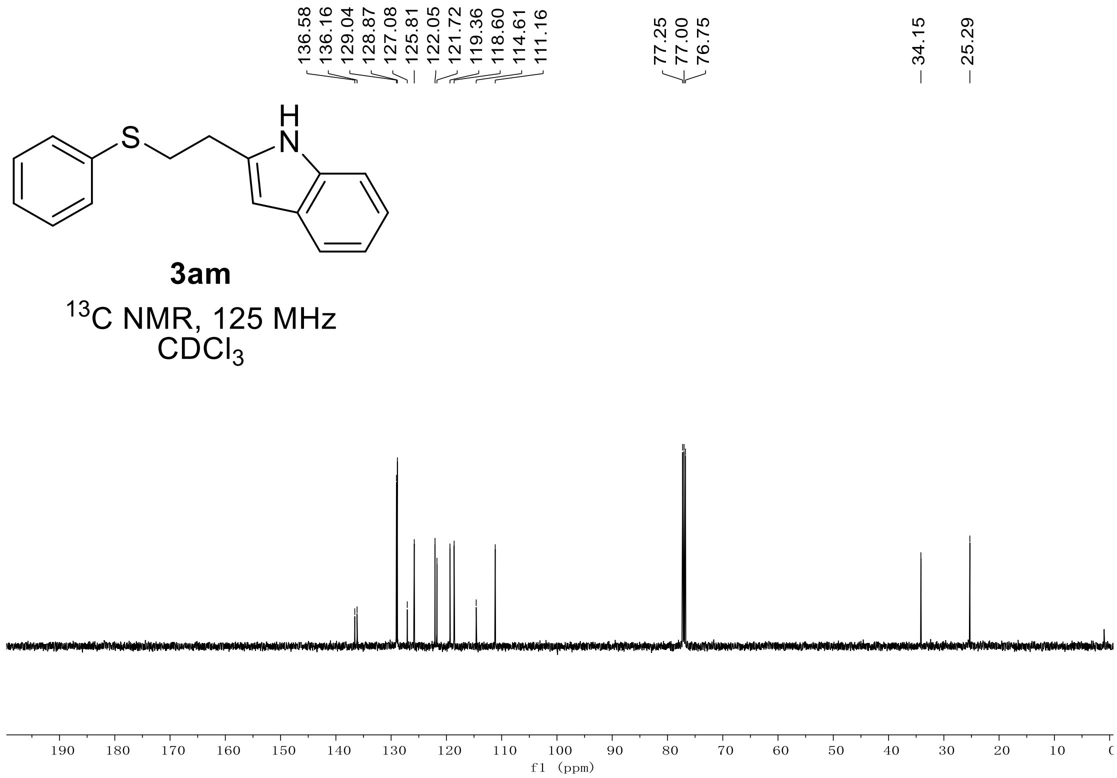


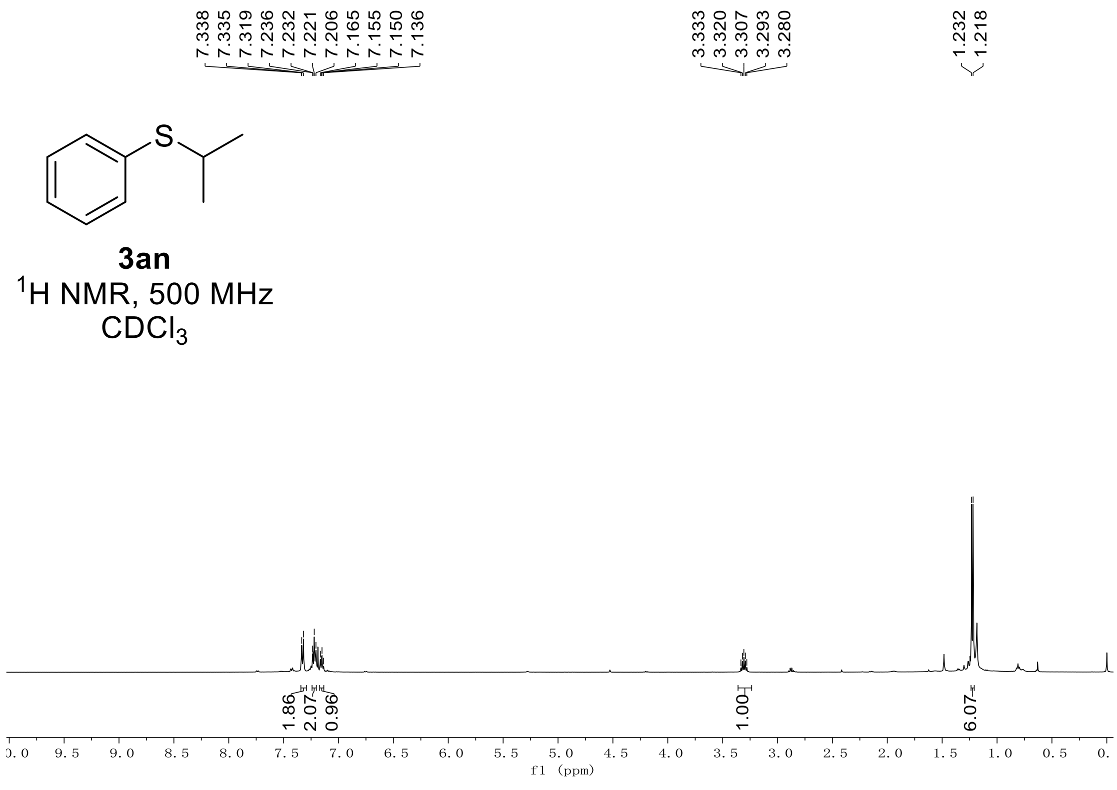
3am

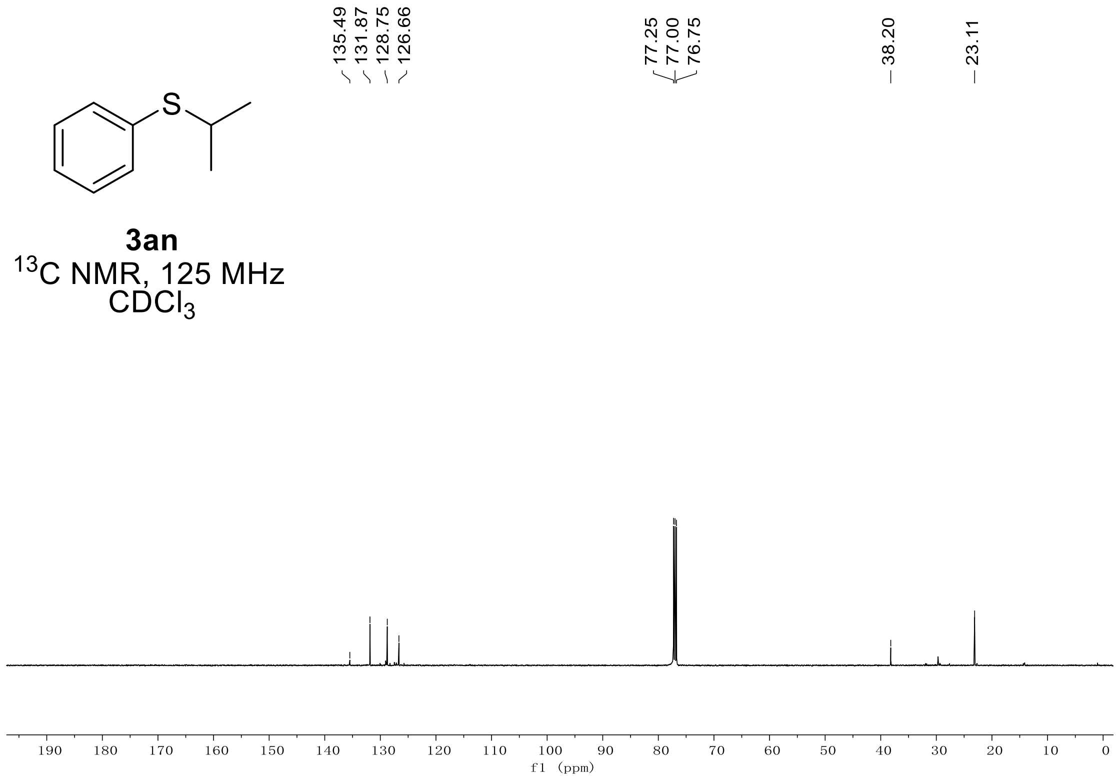
^{1}H NMR, 500 MHz

$CDCl_3$

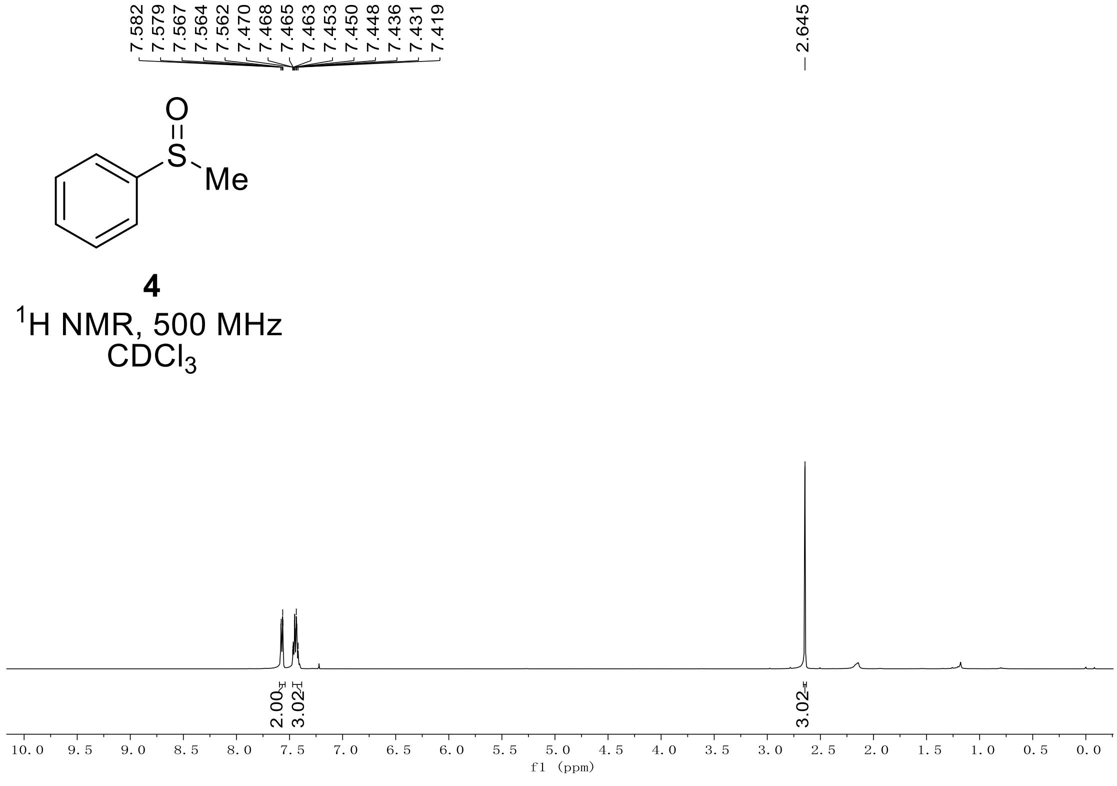



136.58
136.16
129.04
128.87
127.08
125.81
122.05
121.72
119.36
118.60
114.61
111.16
77.25
77.00
76.75
34.15
25.29
H
N
S
3am
^{13}C NMR, 125 MHz
$CDCl_3$
f1 (ppm)

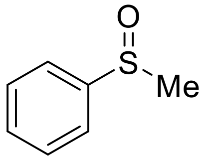

7.338
7.335
7.319
7.236
7.232
7.221
7.206
7.165
7.155
7.150
7.136
3.333
3.320
3.307
3.293
3.280
1.232
1.218
S
3an
^{1}H NMR, 500 MHz
$CDCl_3$
1.86
2.07
0.96
1.00
6.07
f1 (ppm)



3an
^{13}C NMR, 125 MHz
$CDCl_3$

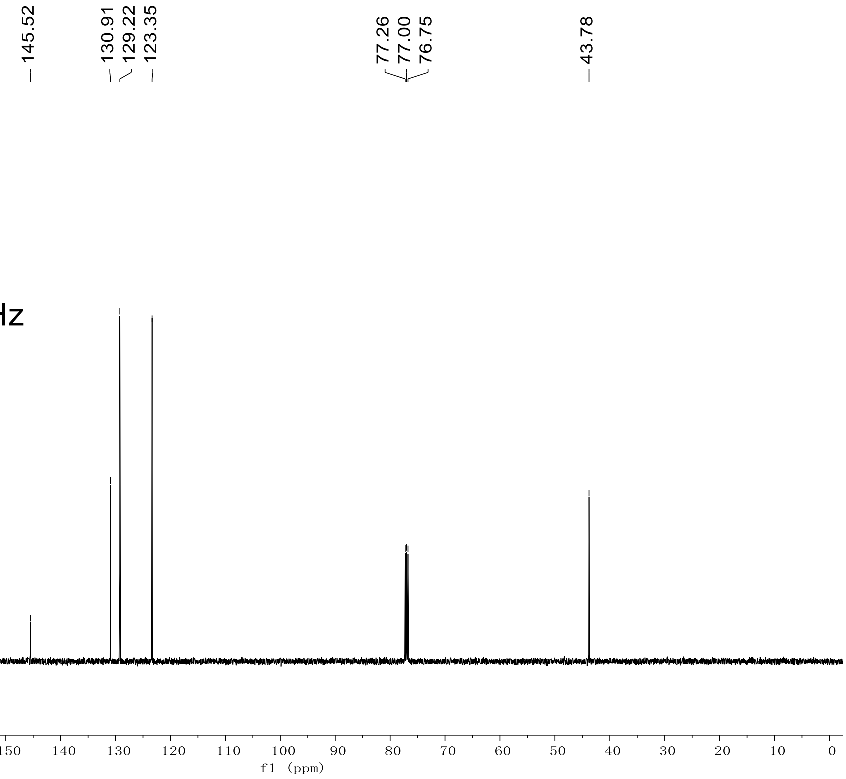


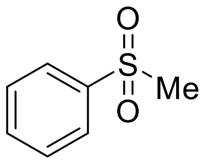
4
^{1}H NMR, 500 MHz
$CDCl_3$



4

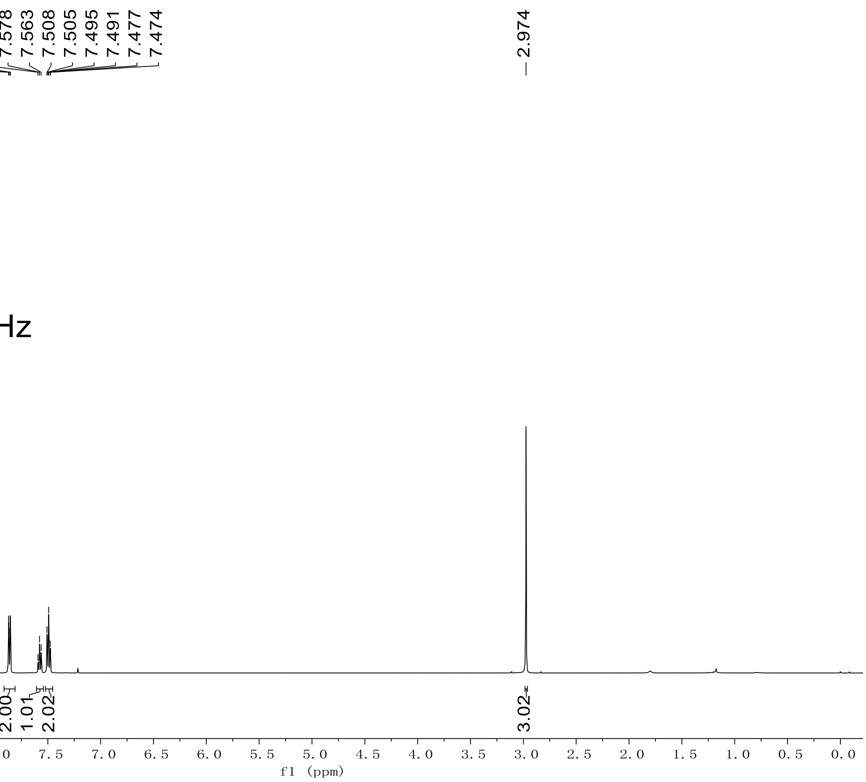
^{13}C NMR, 125 MHz
$CDCl_3$

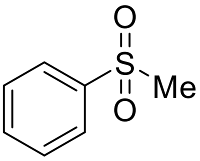




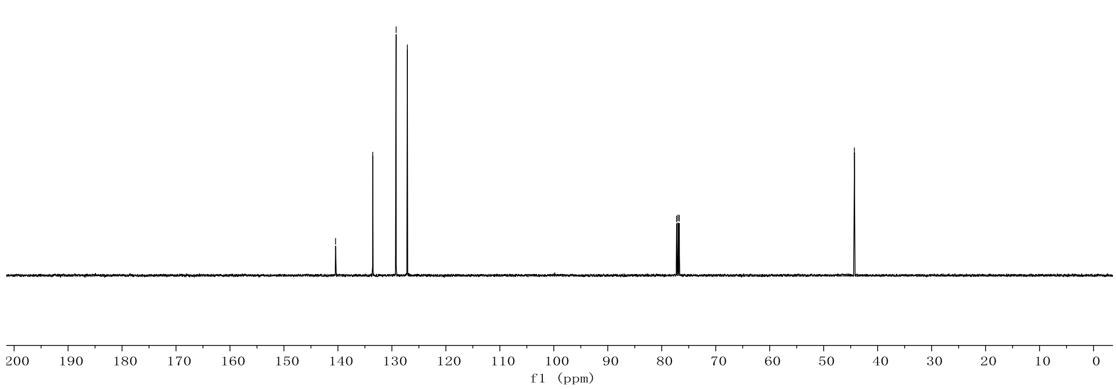
5

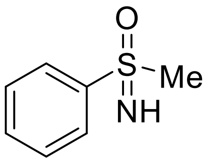
^{1}H NMR, 500 MHz
$CDCl_3$



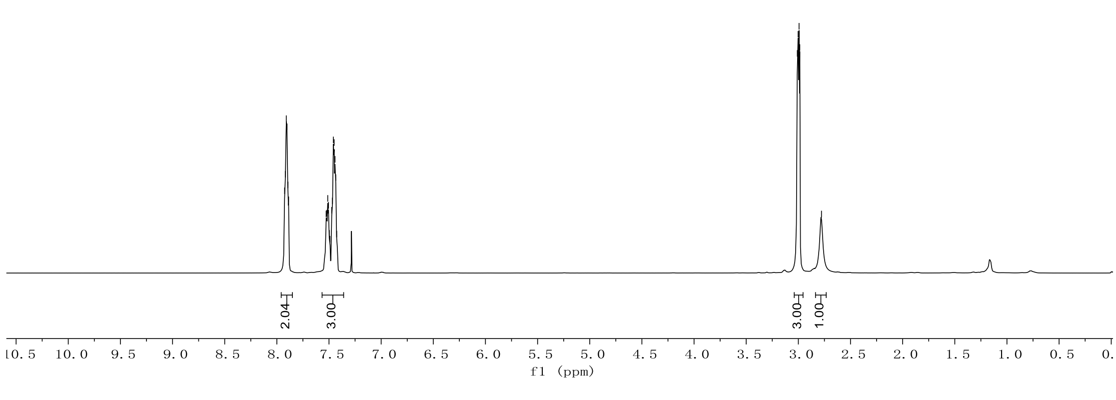


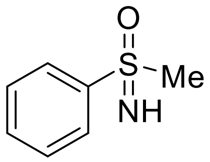
5
^{13}C NMR, 125 MHz
$CDCl_3$



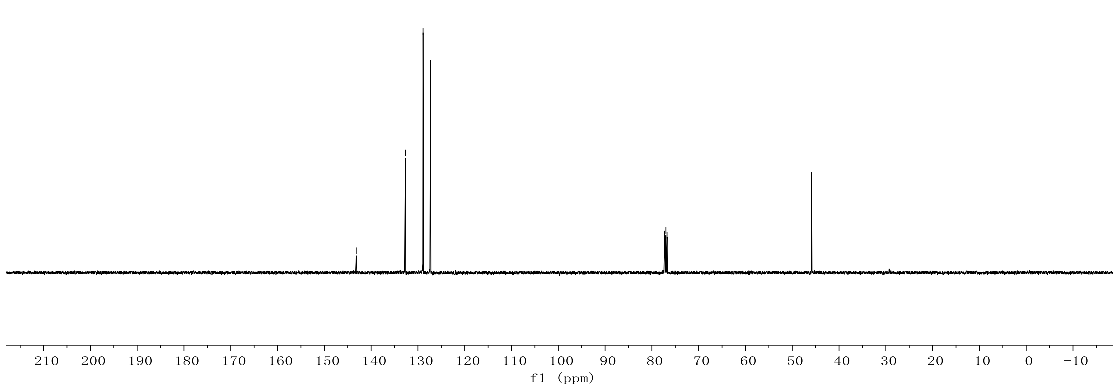


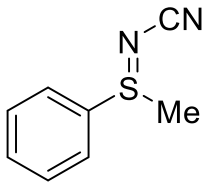
6
^{1}H NMR, 500 MHz
$CDCl_3$



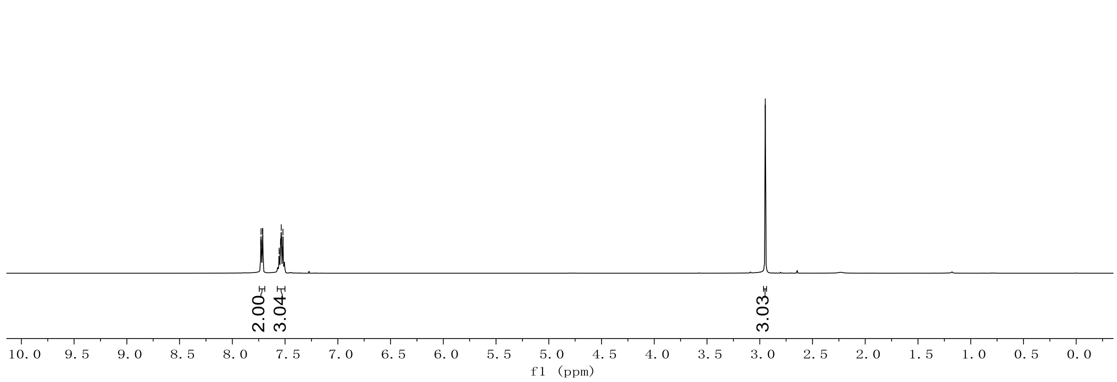


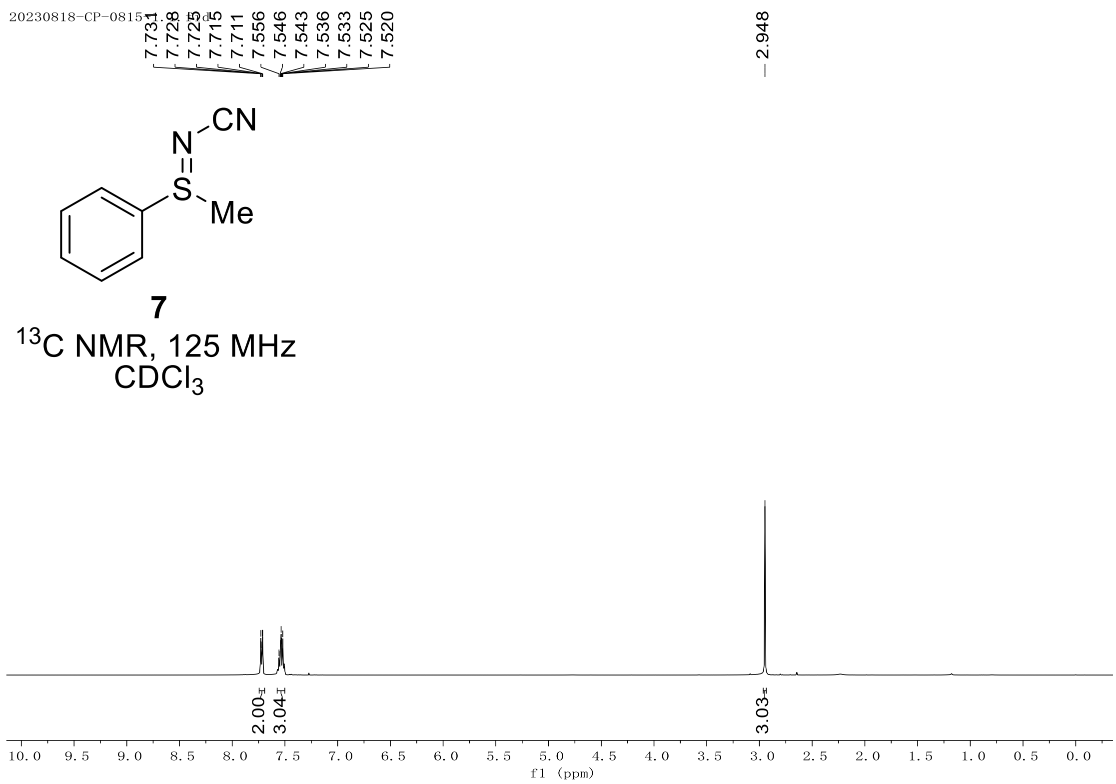
6
^{13}C NMR, 125 MHz
$CDCl_3$

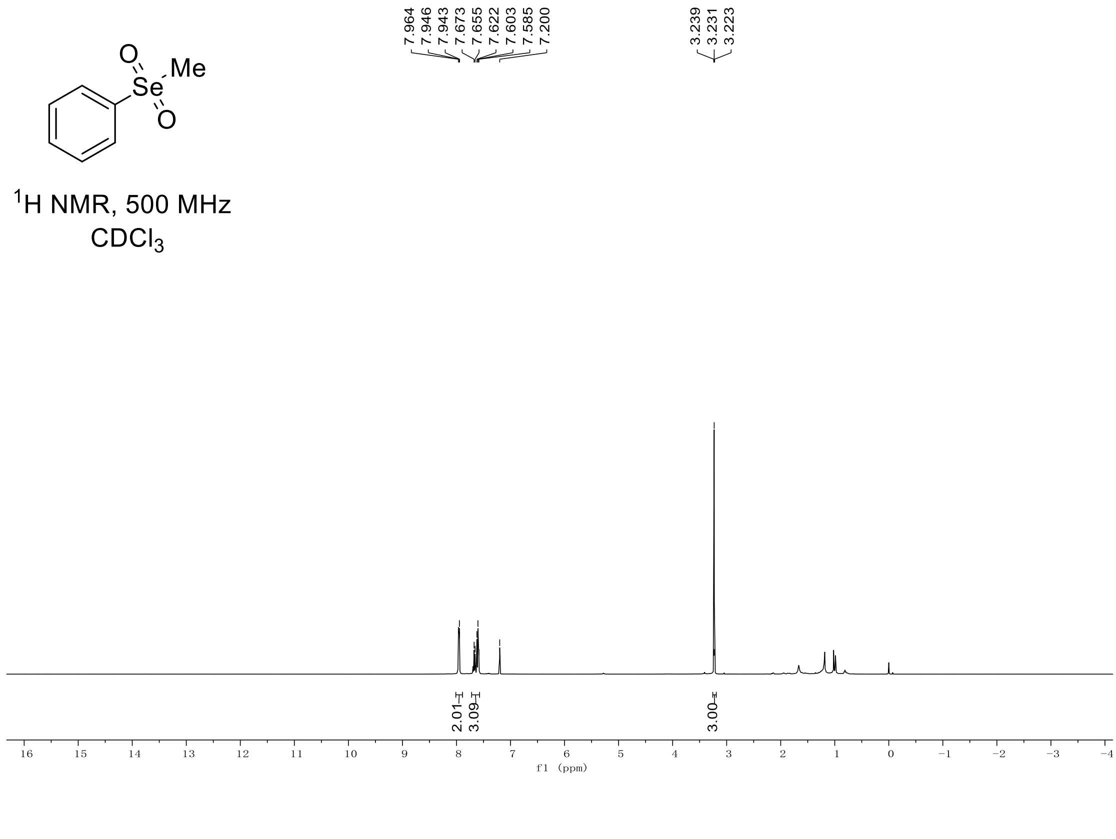


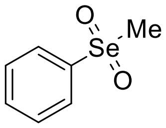


7
^{1}H NMR, 500 MHz
$CDCl_3$

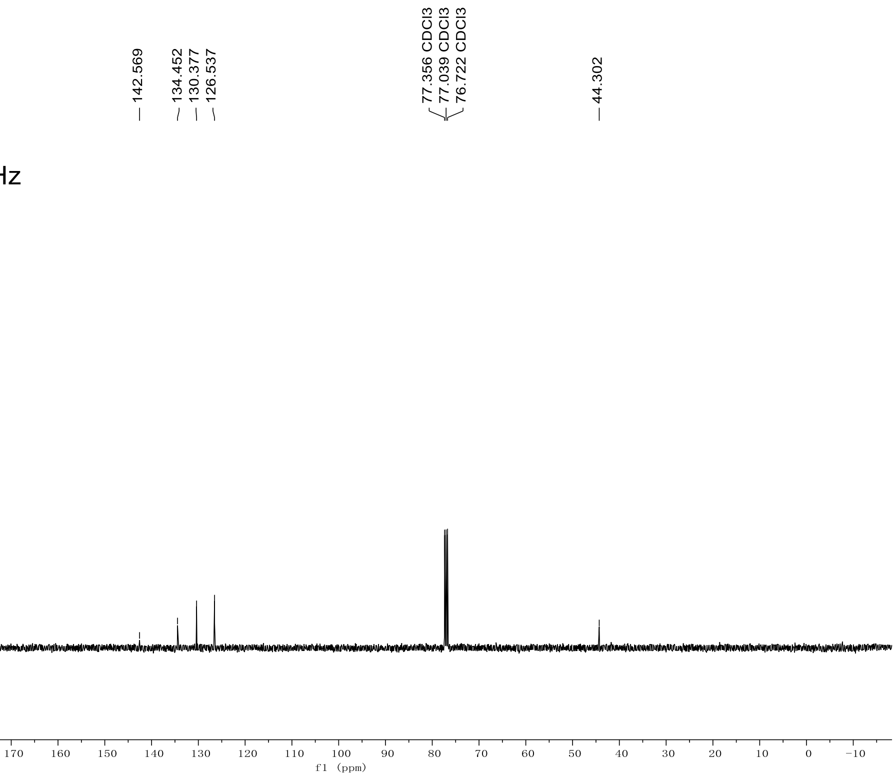



20230818-CP-0815
7.731
7.728
7.725
7.715
7.711
7.556
7.546
7.543
7.536
7.533
7.525
7.520
2.948
N
CN
S
Me
7
^{13}C NMR, 125 MHz
$CDCl_3$
2.00
3.04
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

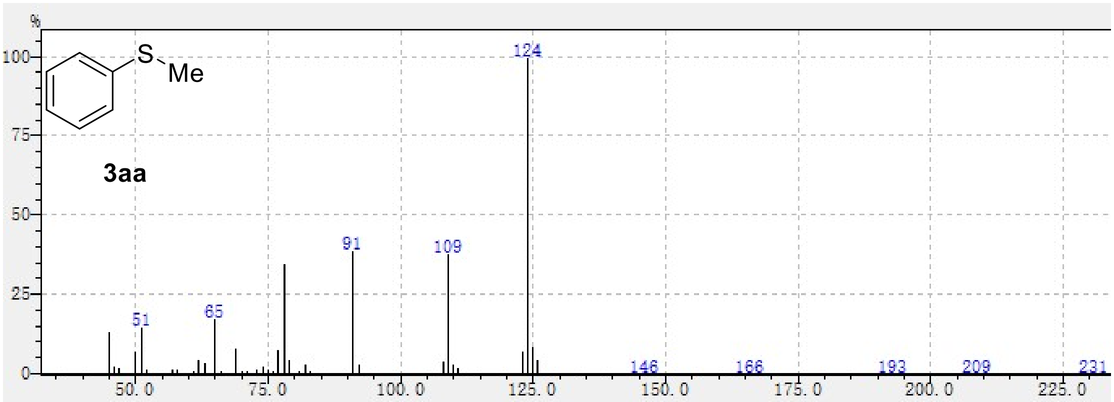

7.964
7.946
7.943
7.673
7.655
7.622
7.603
7.585
7.200
3.239
3.231
3.223
O
Se
Me
O
^{1}H NMR, 500 MHz
$CDCl_3$
2.01
3.09
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

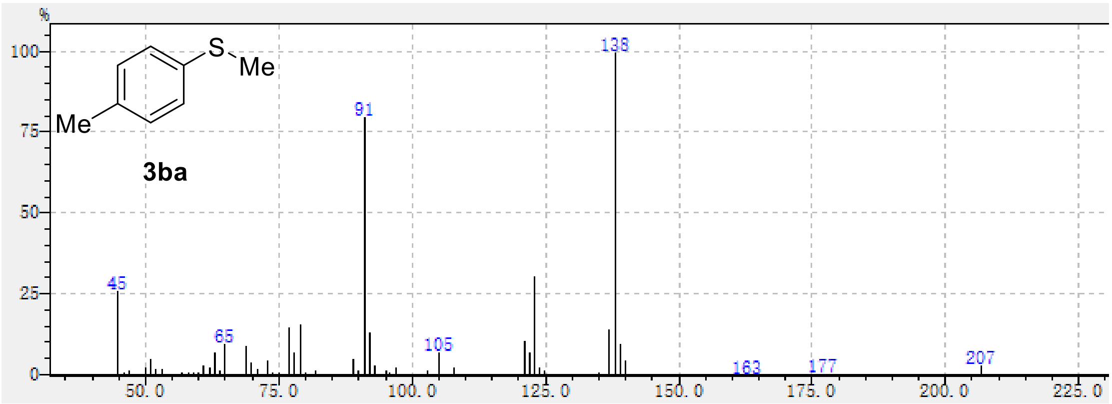


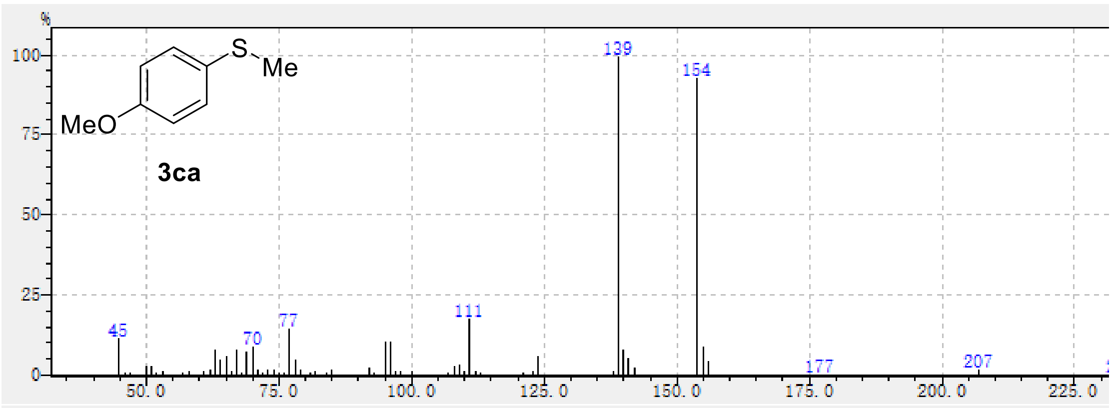
^{13}C NMR, 125 MHz
$CDCl_3$

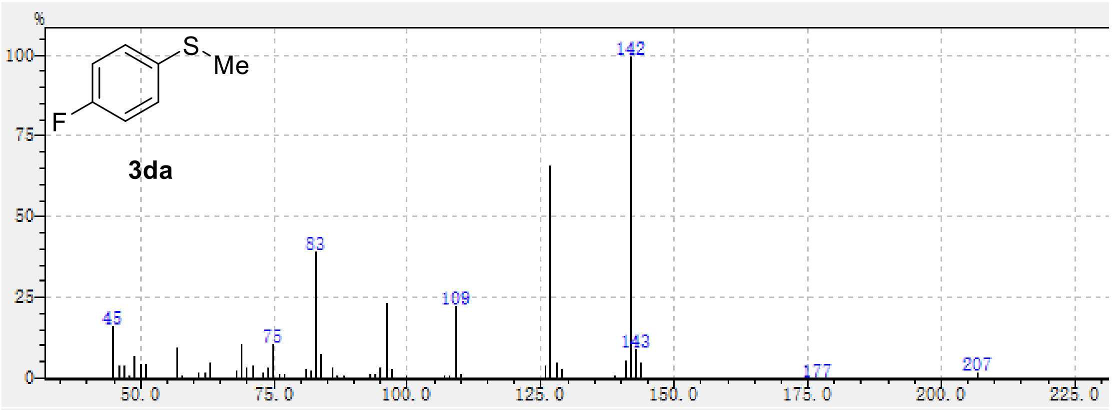


6. MS Spectra

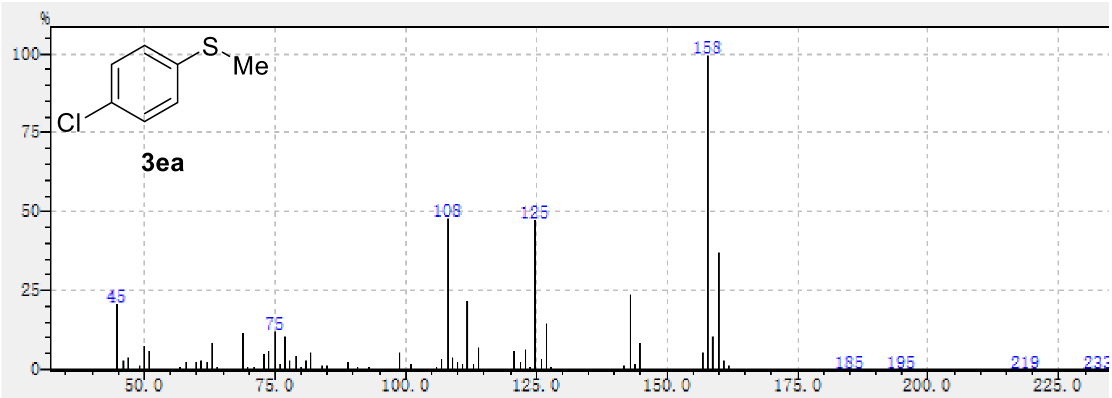

S
Me
3aa
%
100
75
50
25
0
124
91
109
51
65
146
166
193
209
231
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0


S
Me
Me
3ba
%
100
75
50
25
0
138
91
45
65
105
163
177
207
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0

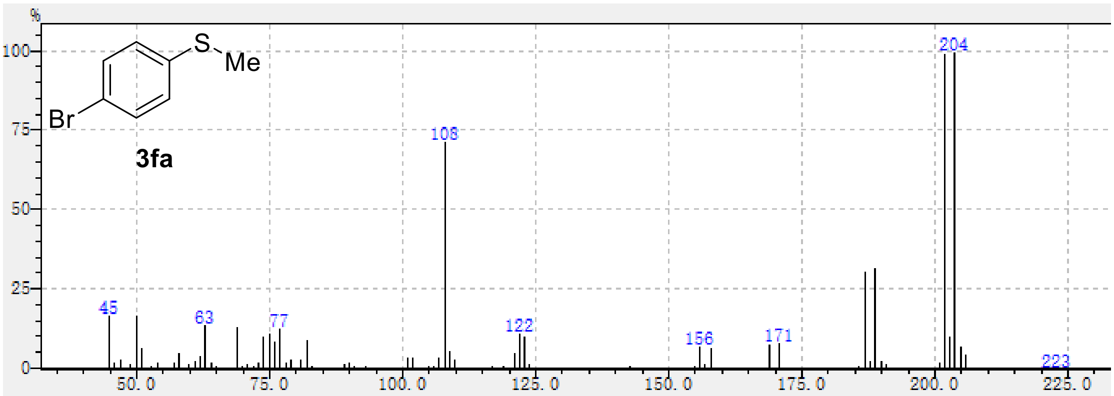

S
Me
MeO
3ca
%
100
75
50
25
0
139
154
45
70
77
111
177
207
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



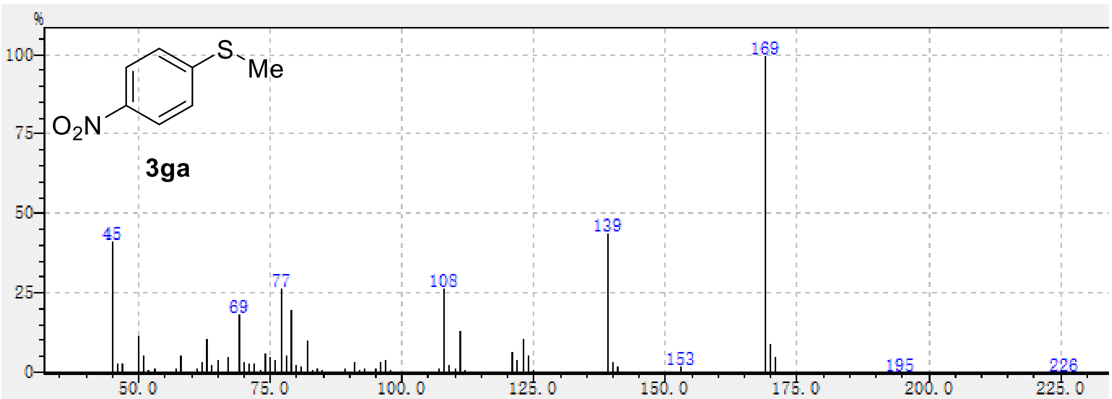
S
Me
F
3da
%
100
75
50
25
0
45
75
83
109
142
143
177
207
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



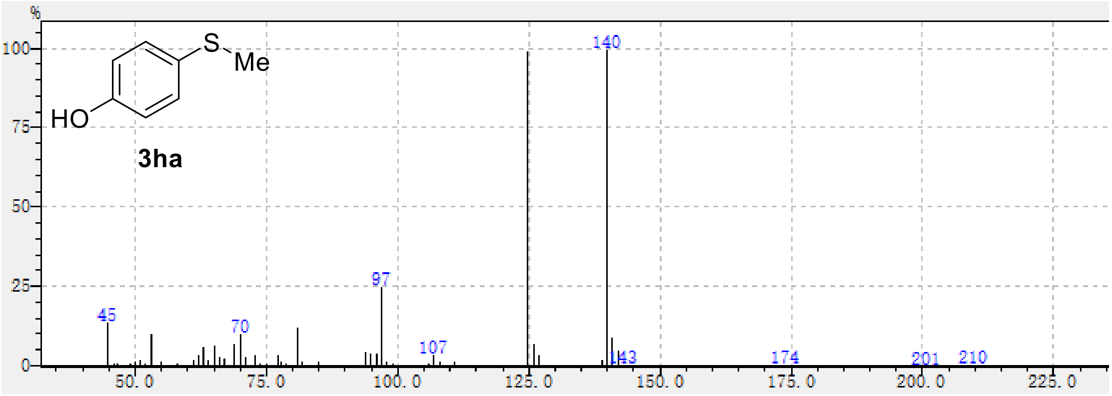
S
Me
Cl
3ea
%
100
75
50
25
0
45
75
108
125
158
185
195
219
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



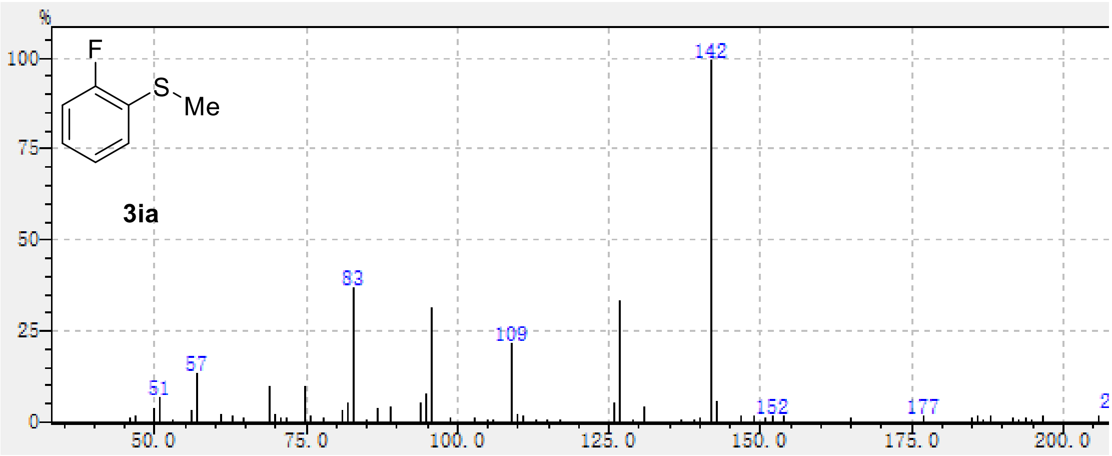
S
Me
Br
3fa
%
100
75
50
25
0
45
63
77
108
122
156
171
204
223
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



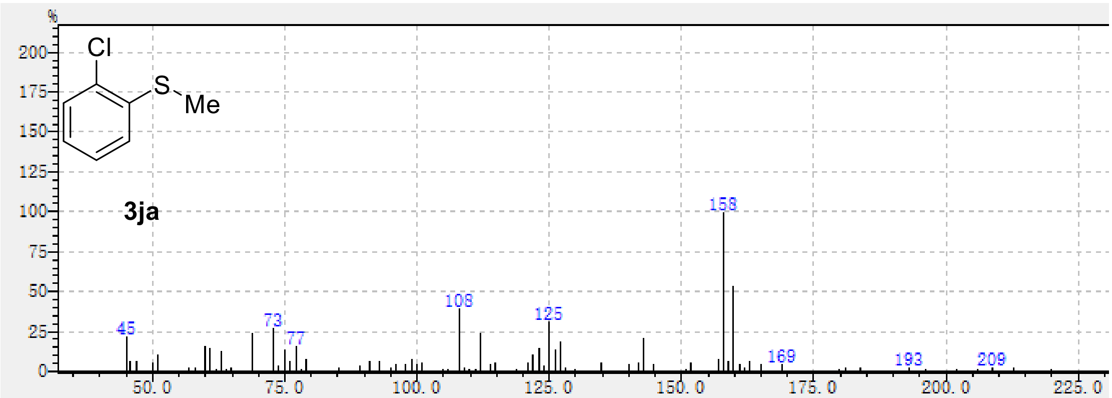
S
Me
O2N
3ga
%
100
75
50
25
0
45
69
77
108
139
153
169
195
226
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



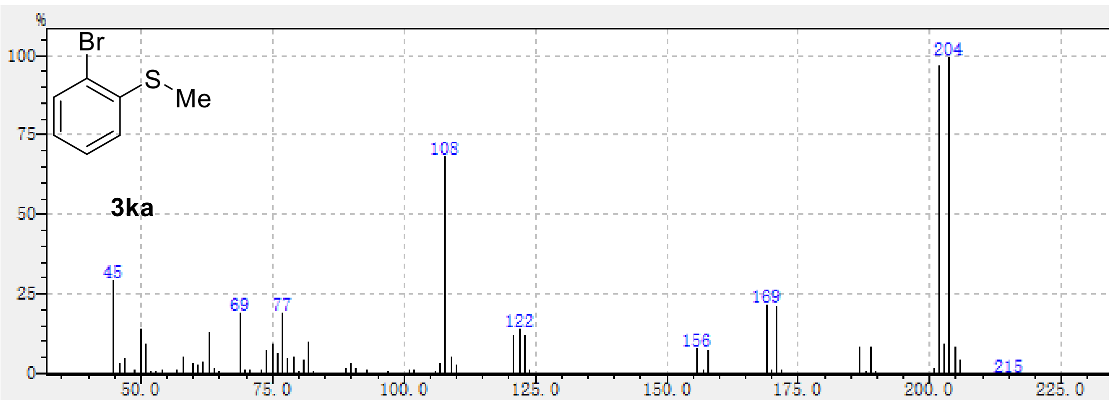
S
Me
HO
3ha
140
97
45
70
107
143
174
201
210
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0



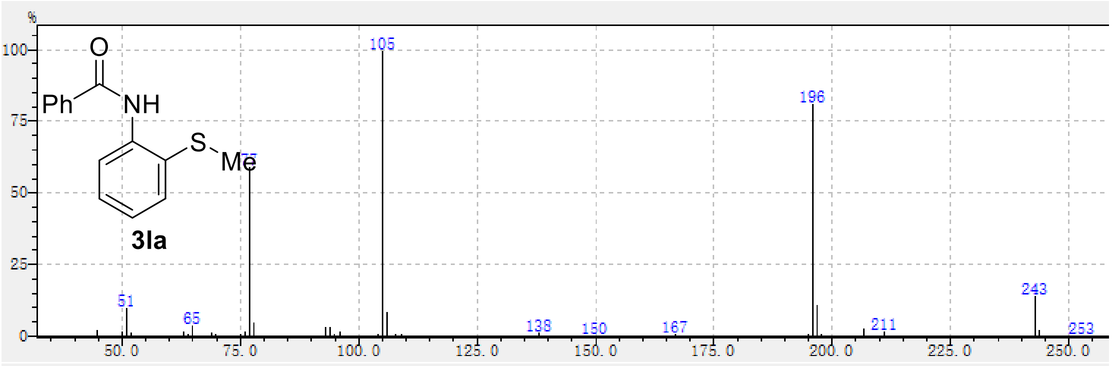
F
S
Me
3ia
142
83
109
57
51
152
177
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0

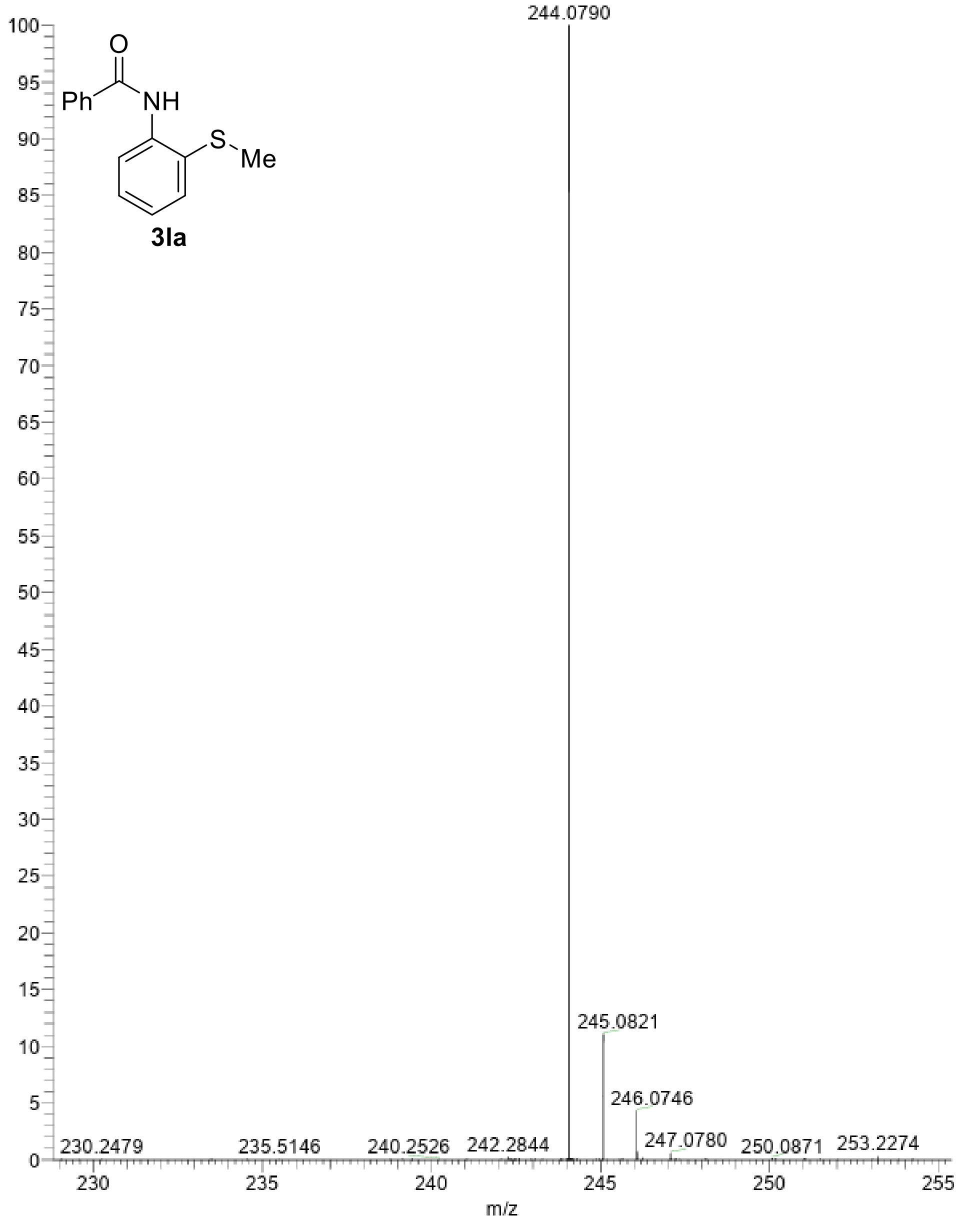


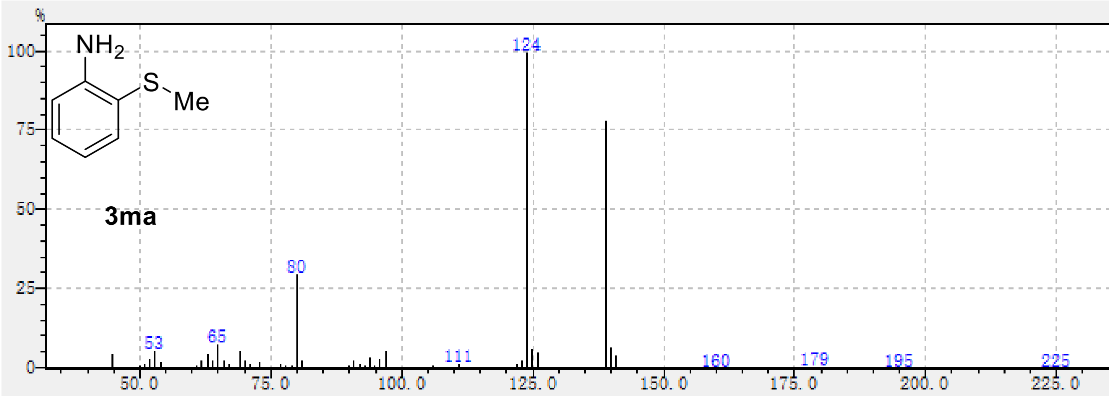
Cl
S
Me
3ja
158
108
125
73
77
45
169
193
209
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0

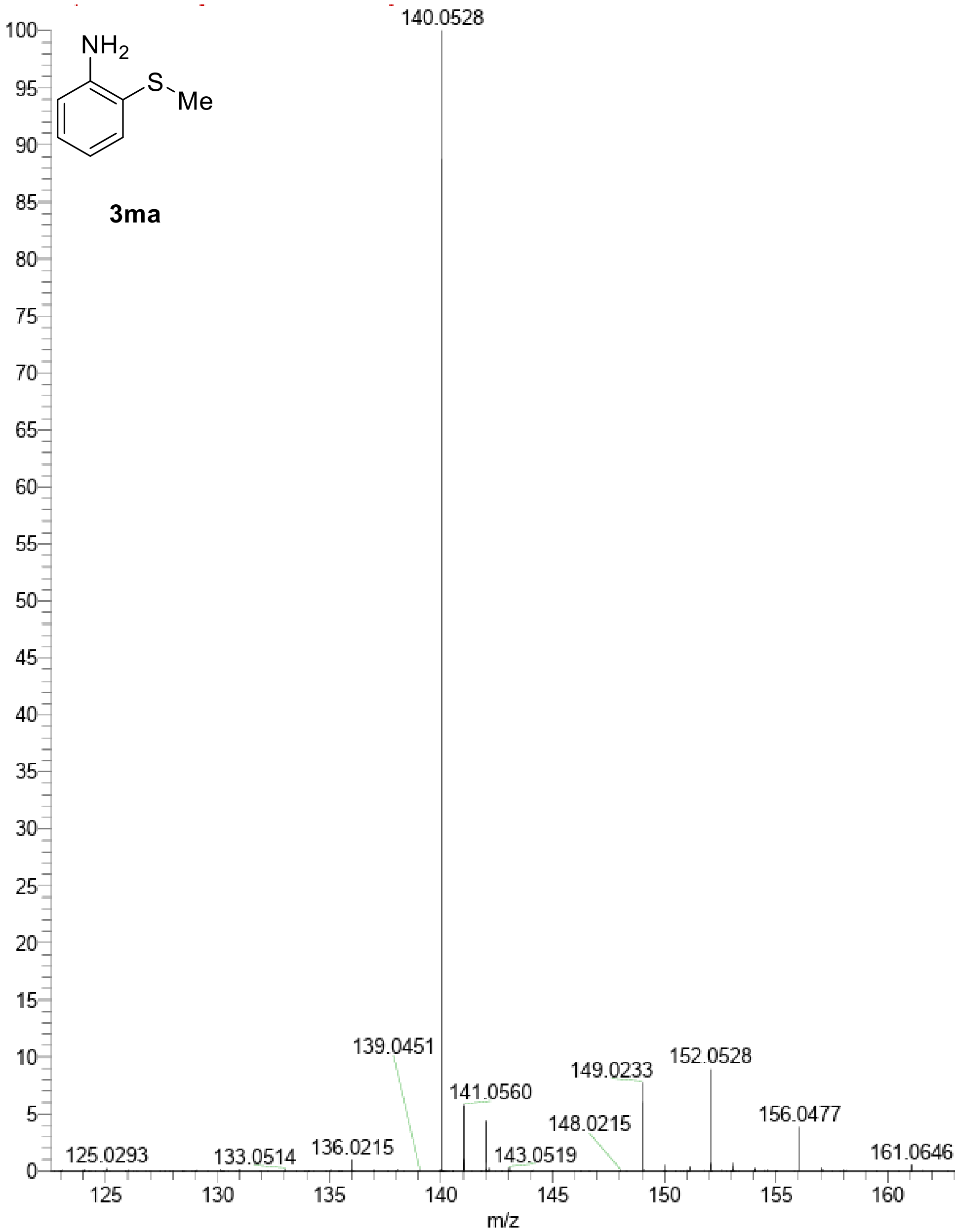


Br
S
Me
3ka
204
108
45
69
77
122
169
156
215
50. 0
75. 0
100. 0
125. 0
150. 0
175. 0
200. 0
225. 0


Ph
NH
O
S
Me
3la
105
196
77
51
65
138
150
167
211
243
253


Ph
NH
O
S
Me
3la
244.0790
245.0821
246.0746
247.0780
230.2479
235.5146
240.2526
242.2844
250.0871
253.2274
m/z


NH2
S
Me
3ma
124
80
53
65
111
160
179
195
225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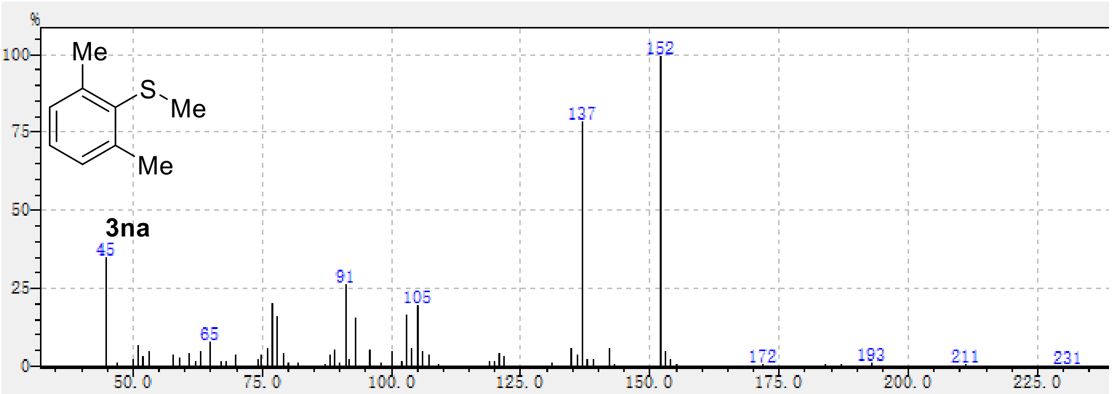
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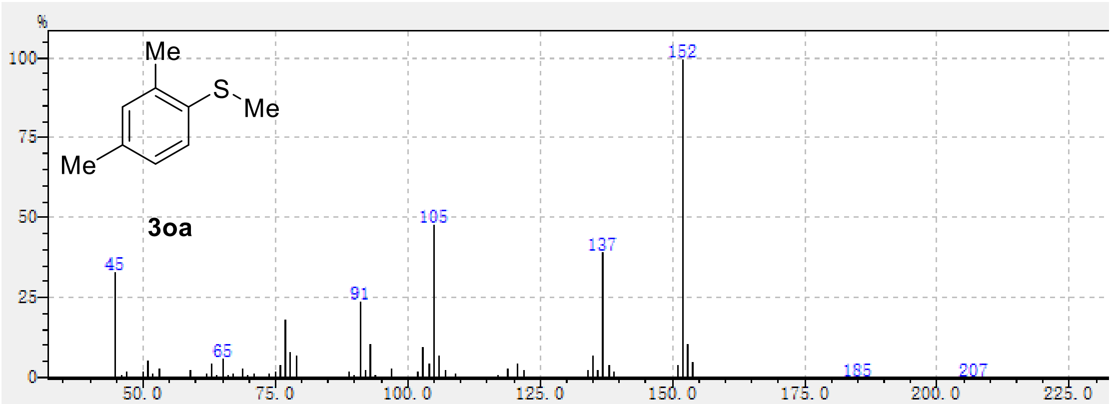
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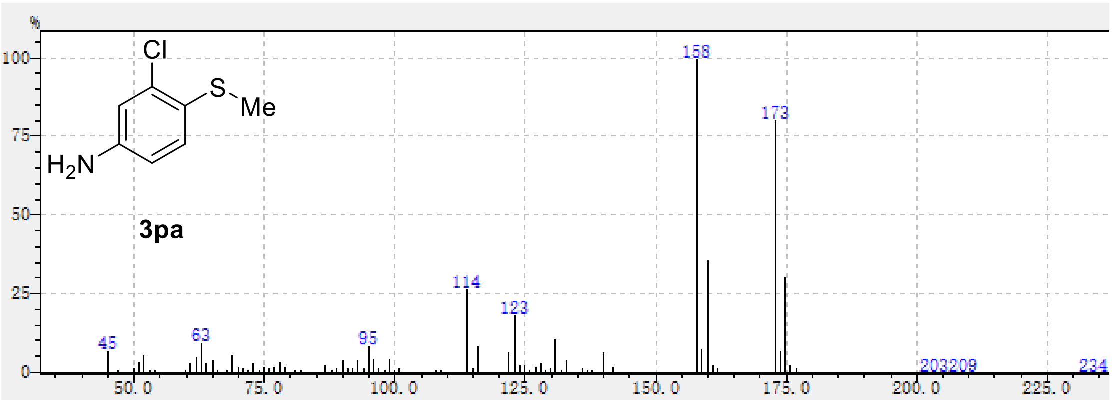
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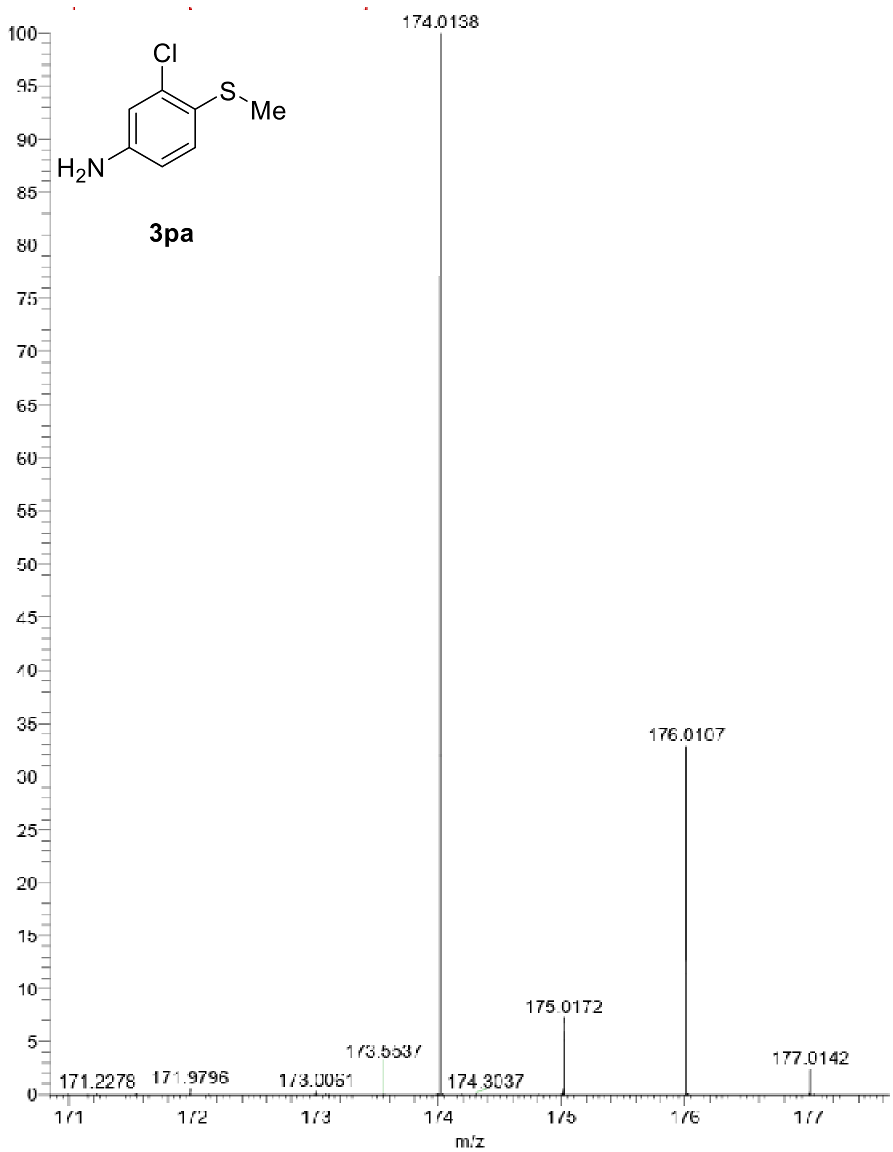
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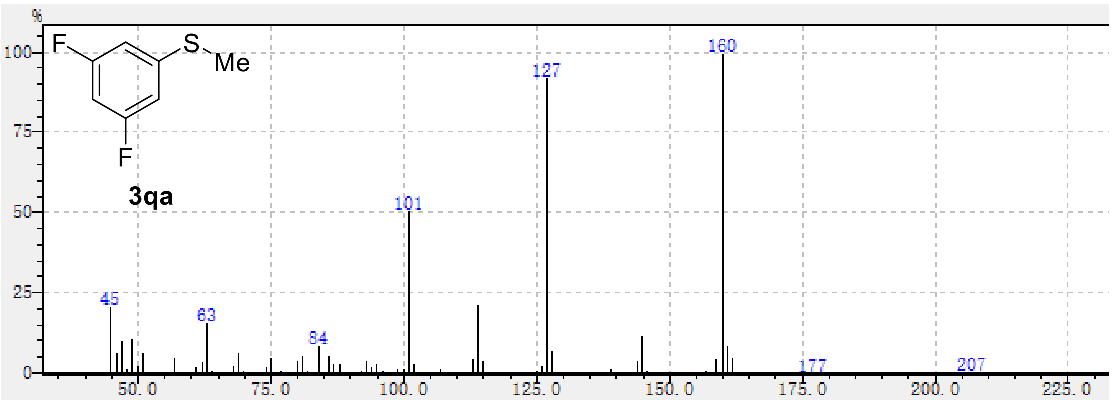
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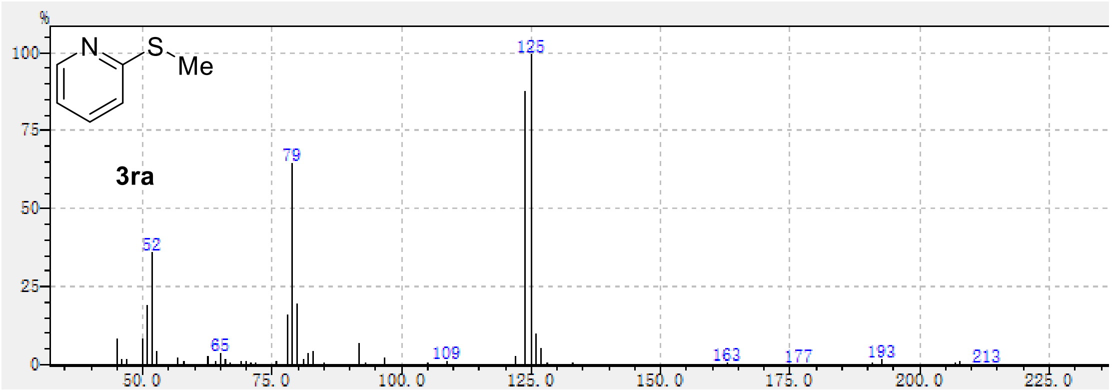

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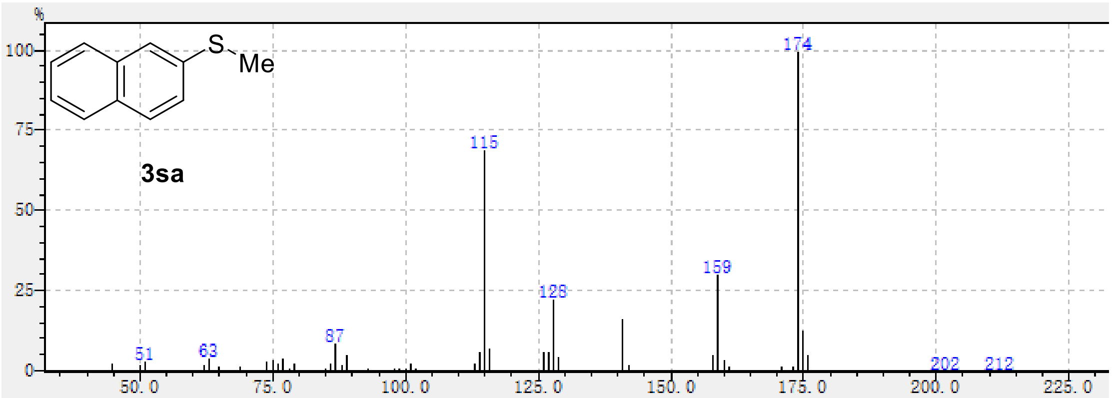

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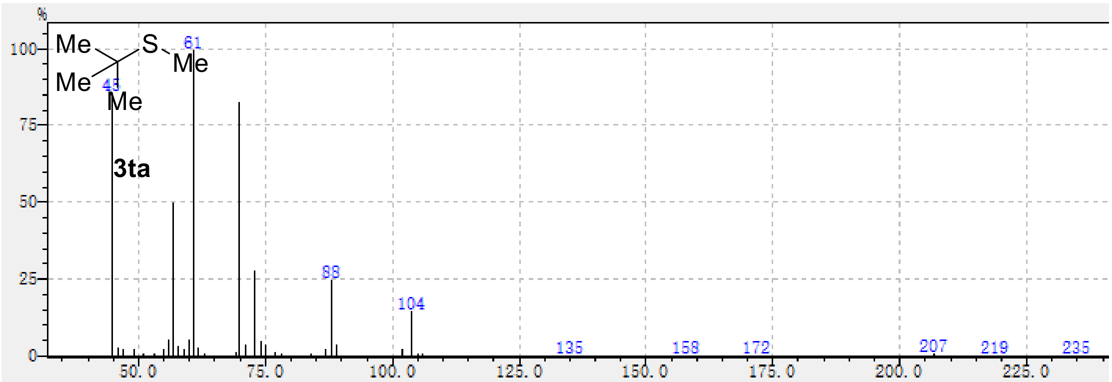

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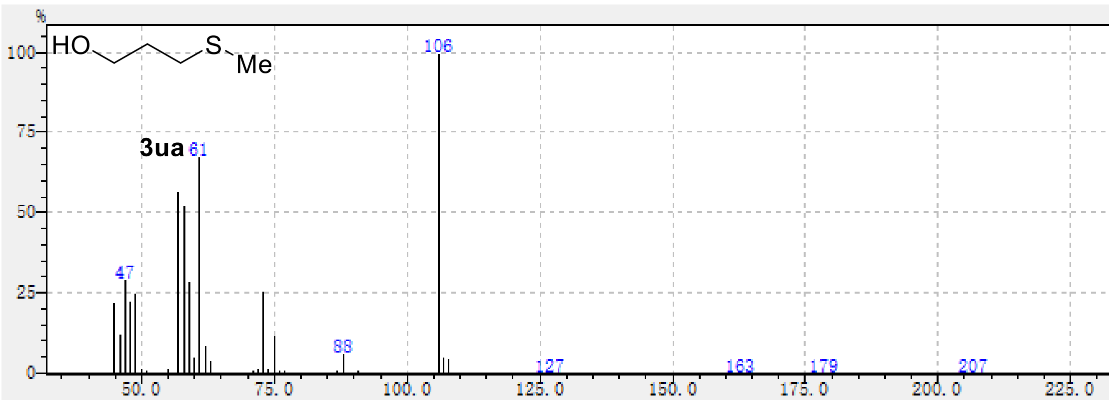

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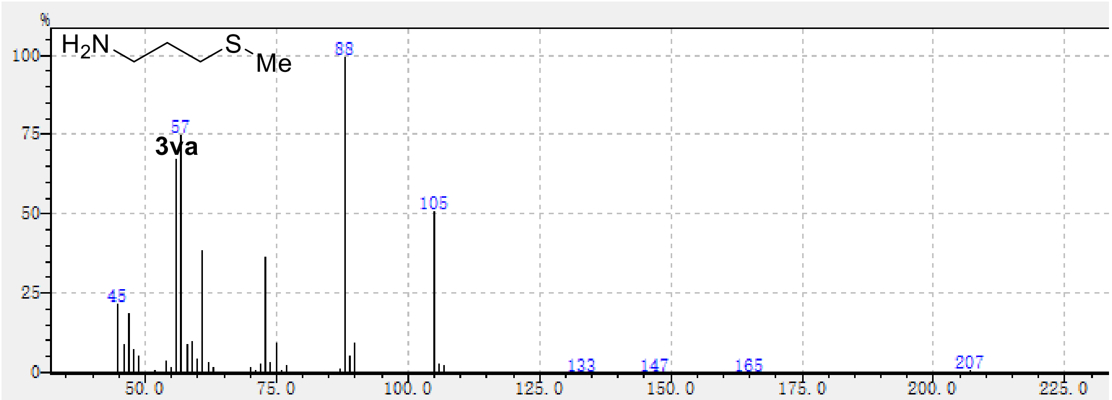

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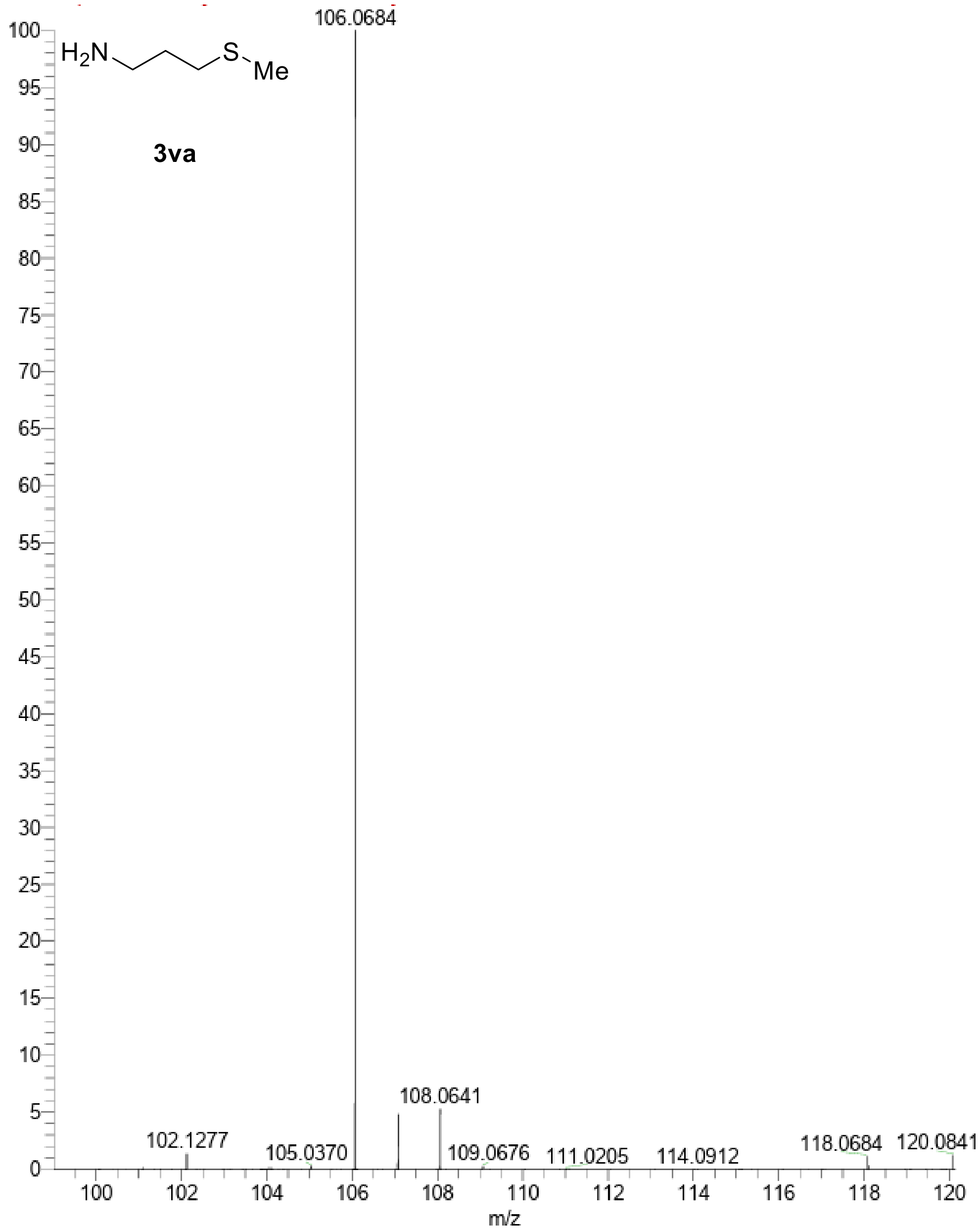

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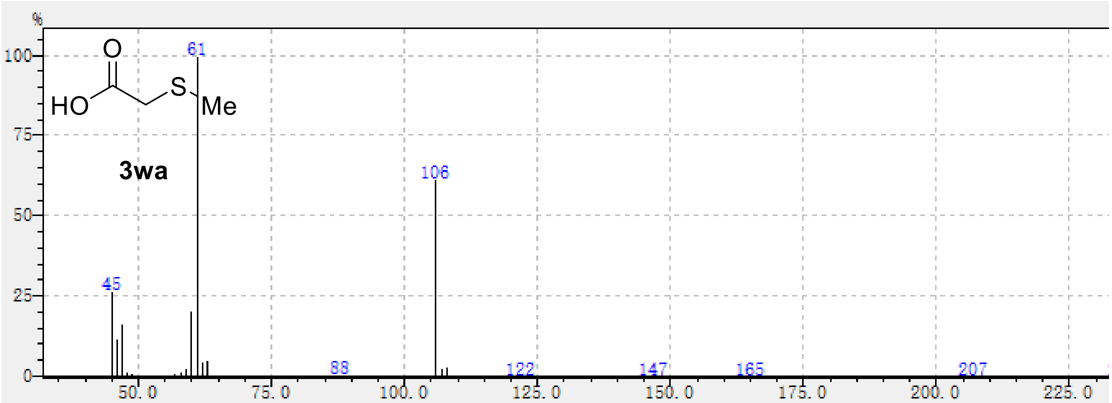
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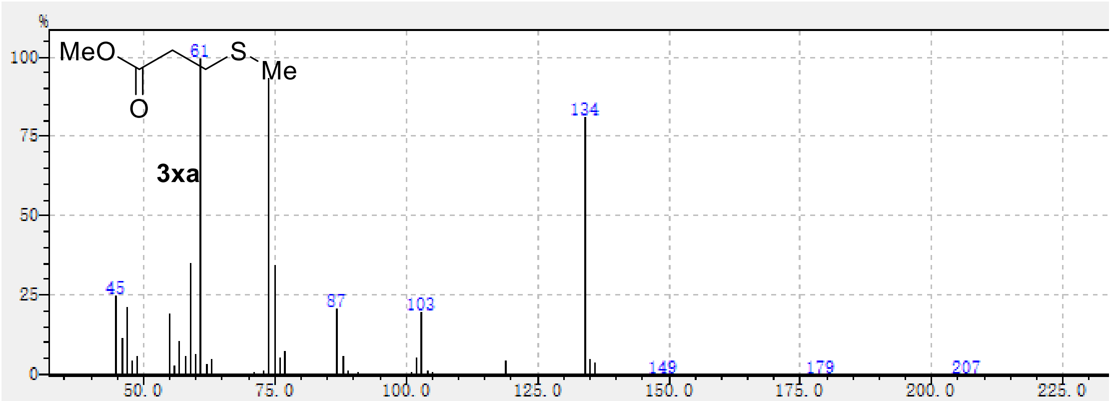
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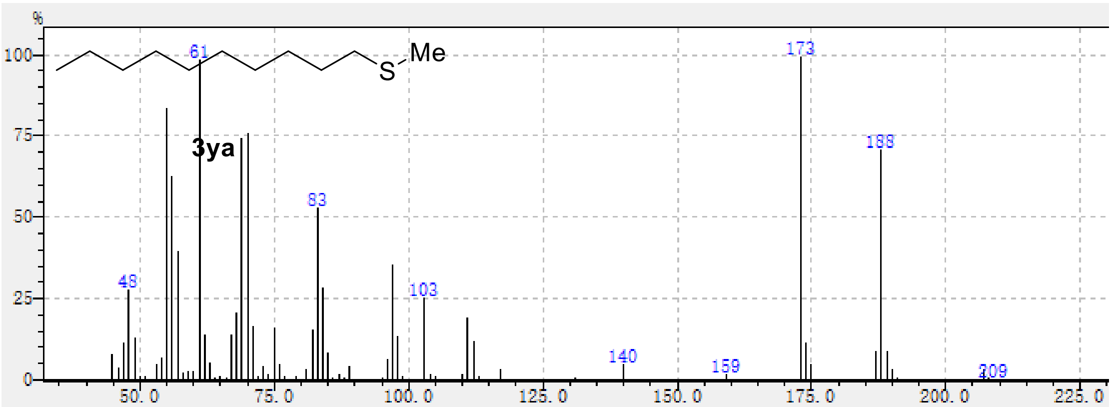
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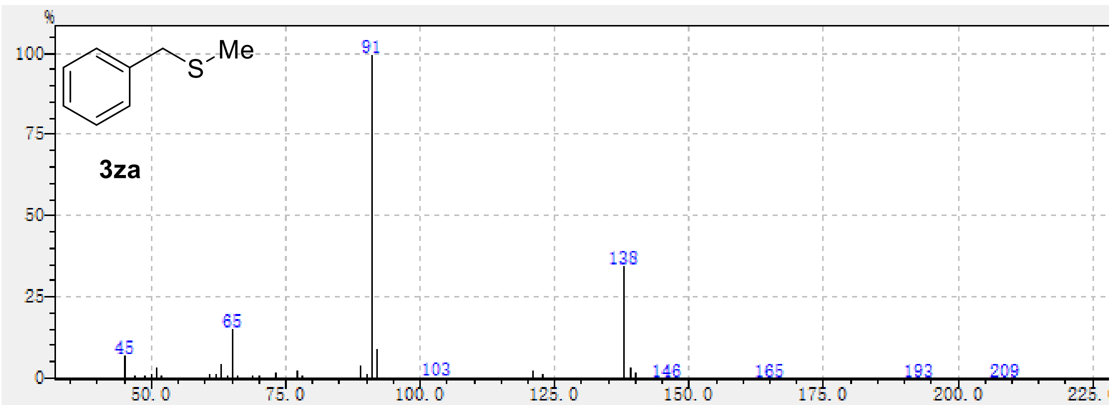
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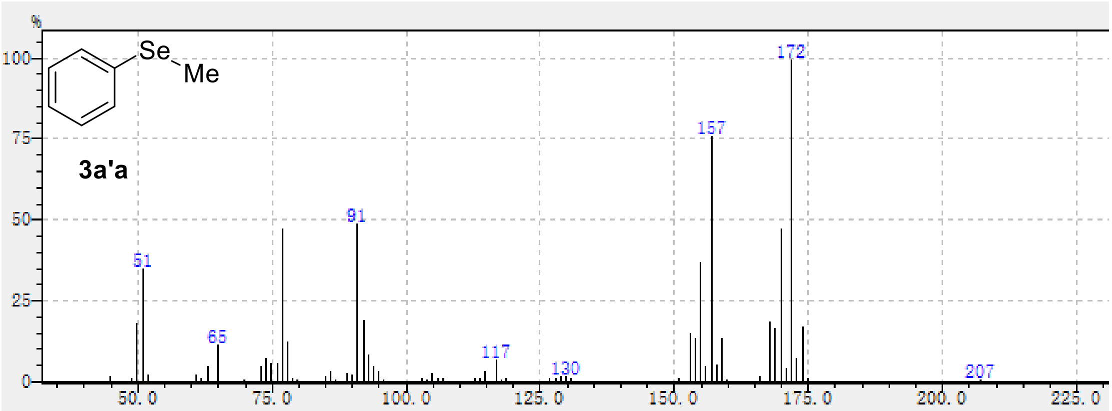
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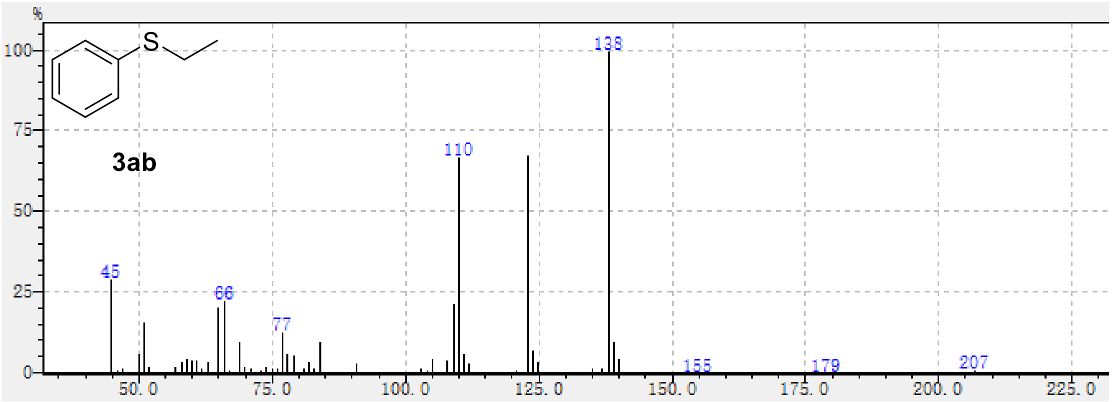
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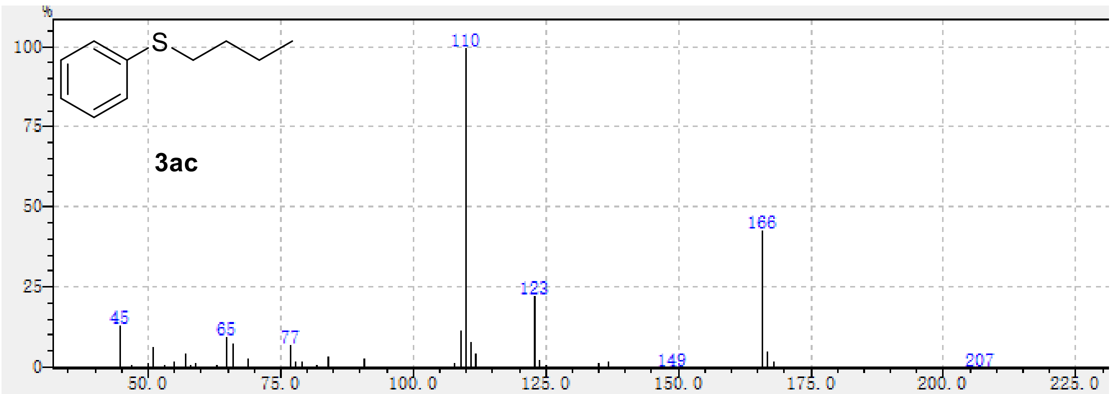
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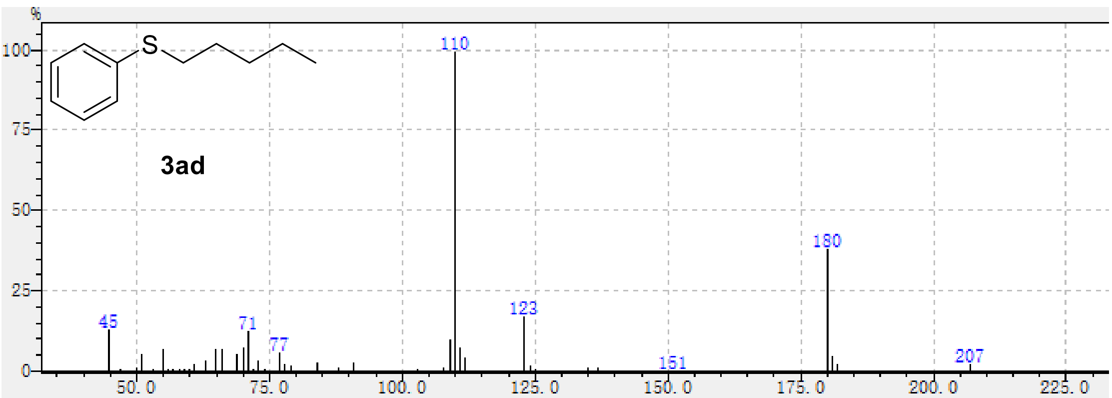
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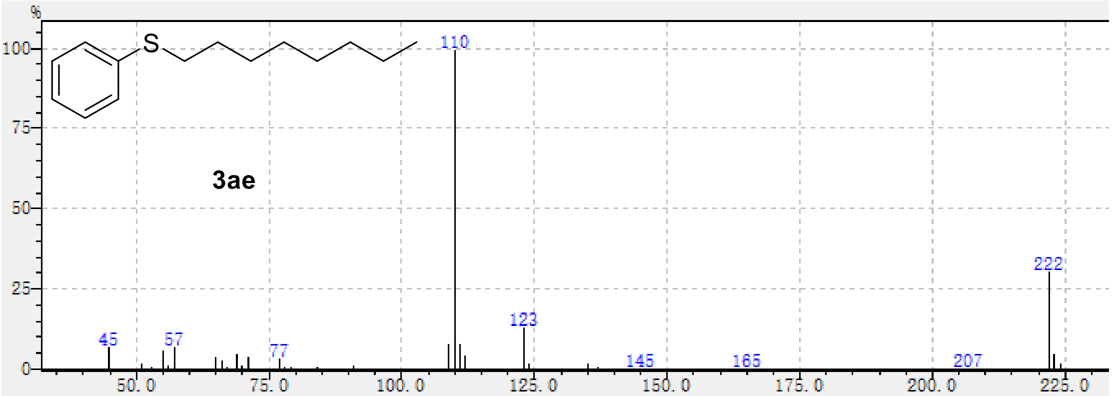
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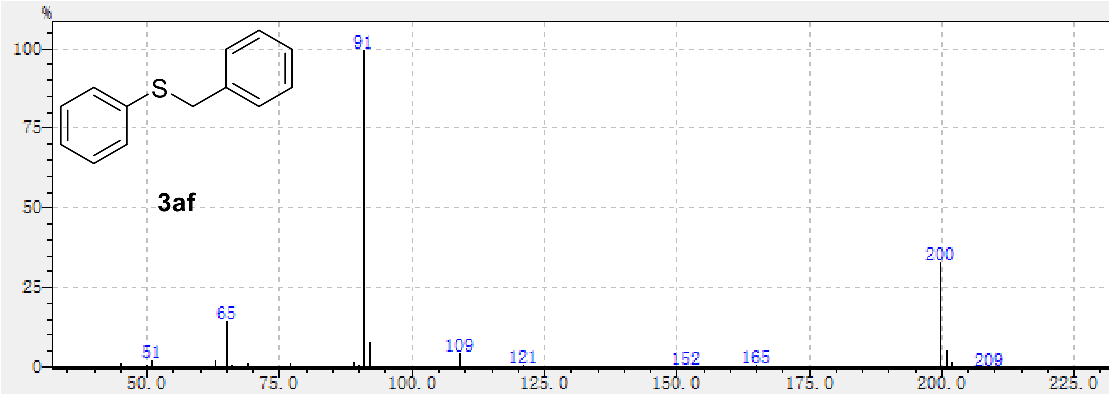
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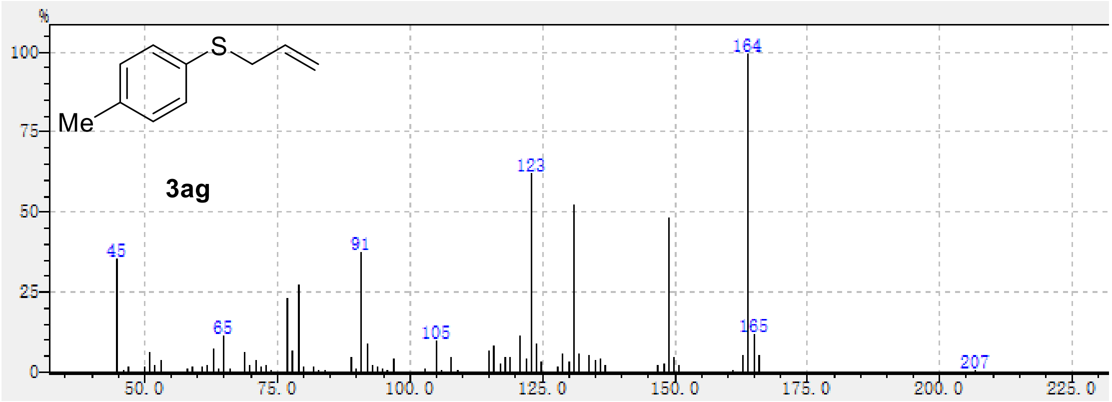
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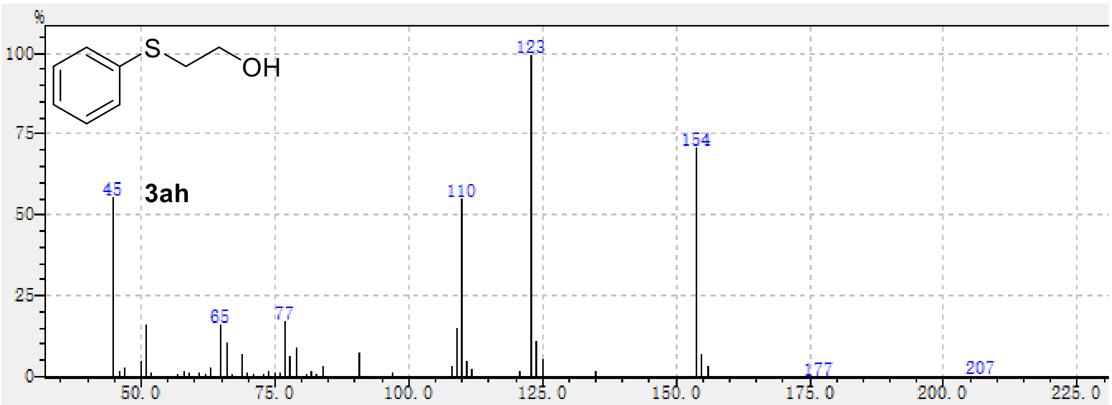
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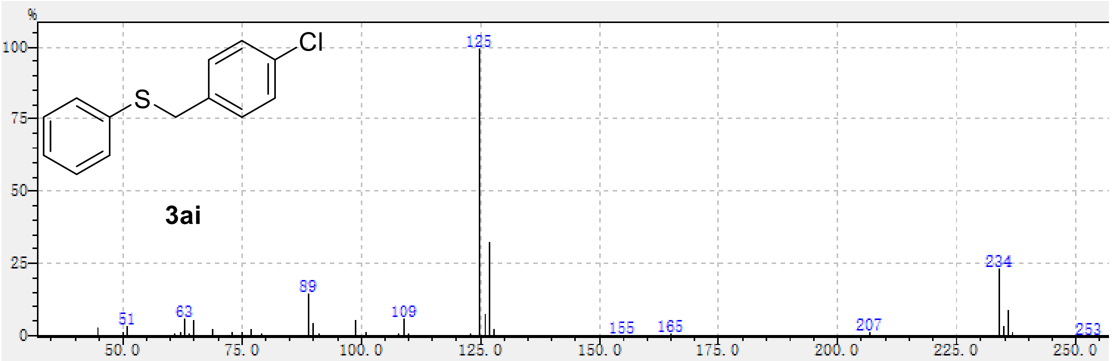

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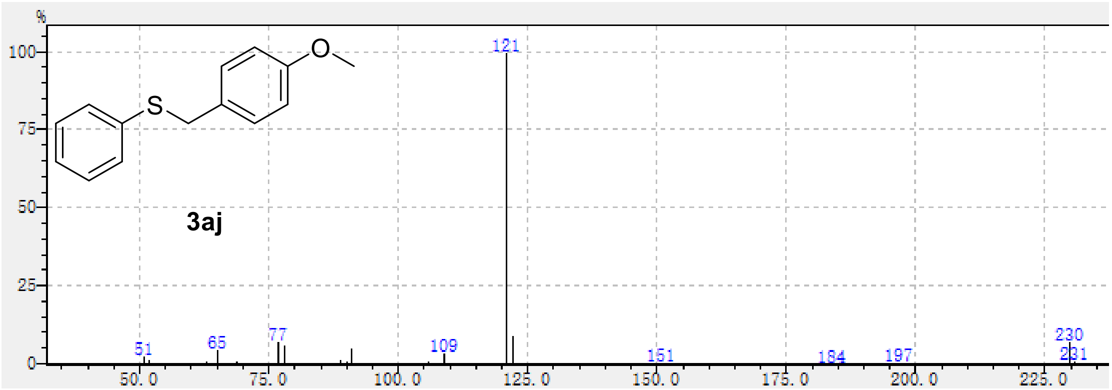

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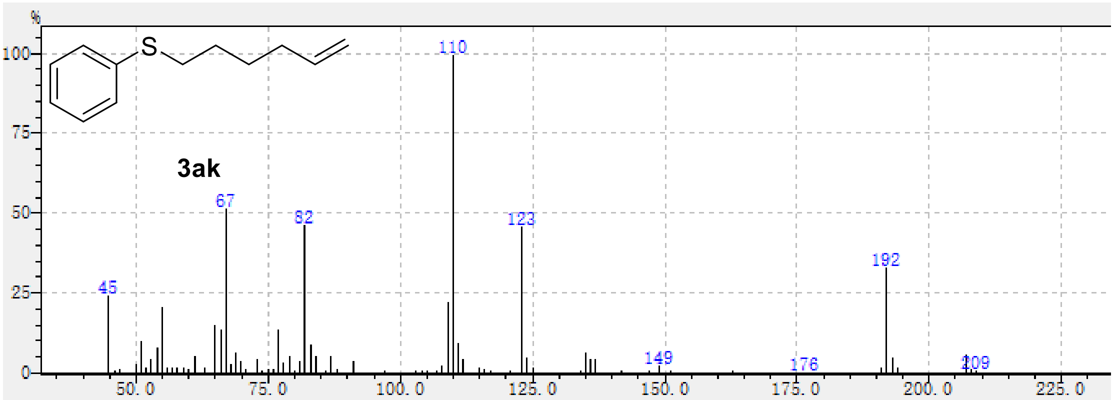

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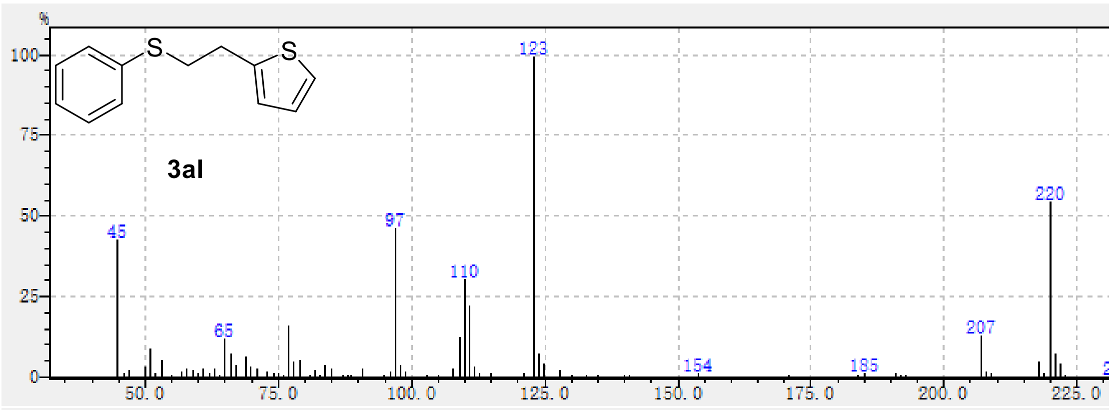

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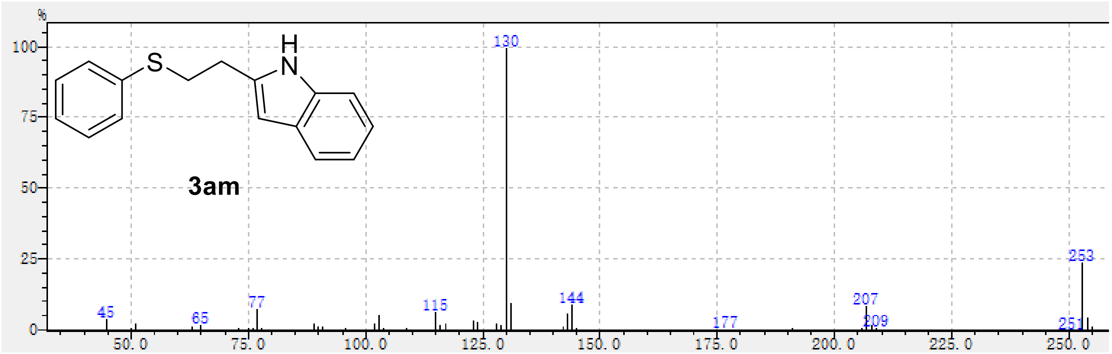
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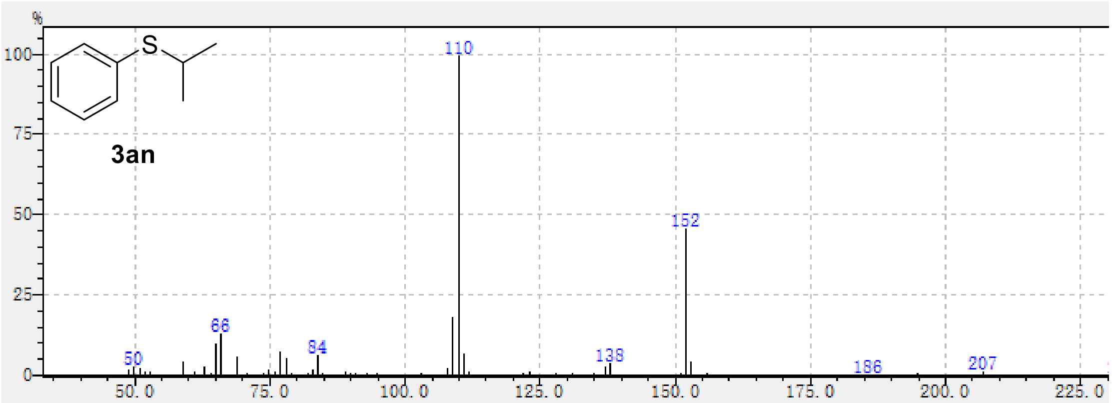
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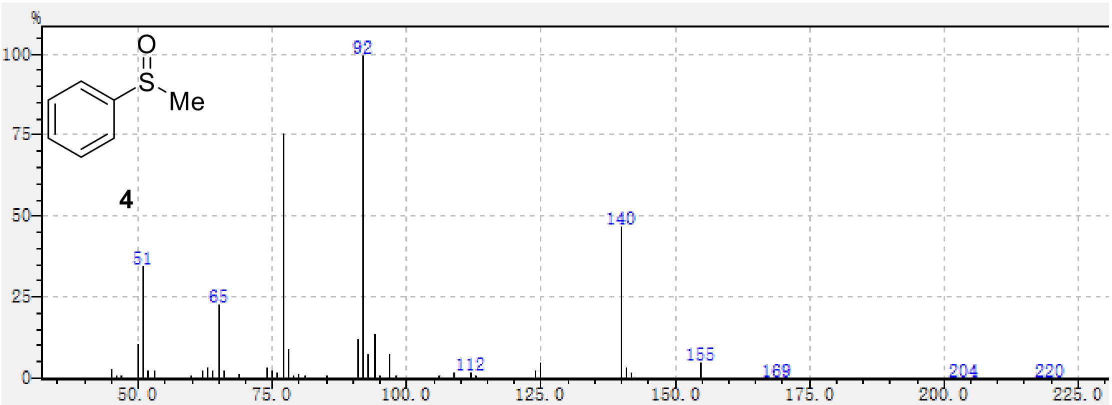
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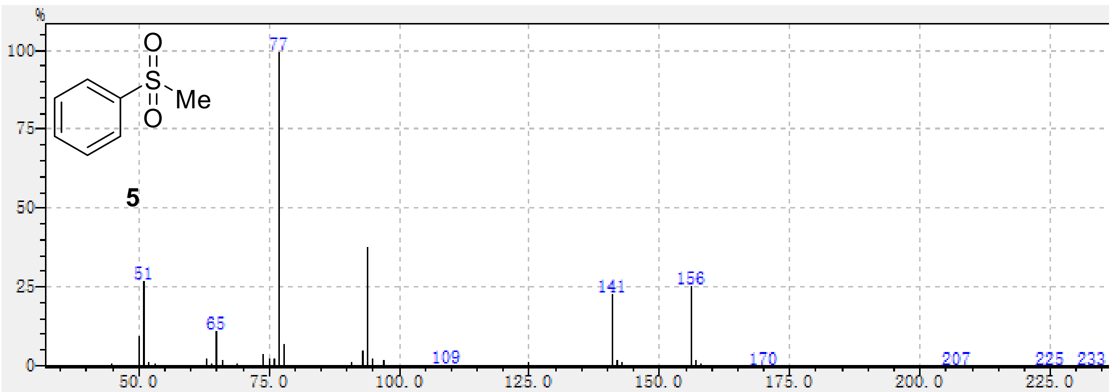
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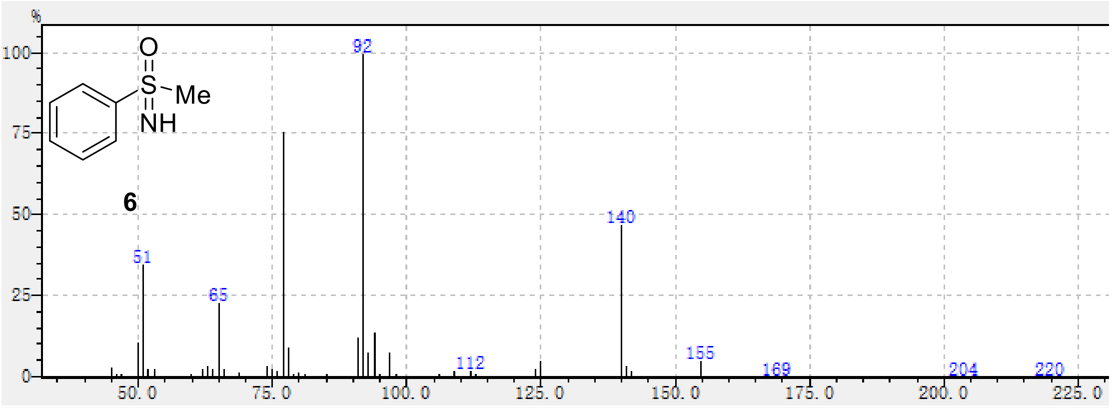
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