Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.


# Direct Dehydroxy(sulfhydryl)xanthylation of Alcohols and Thiols

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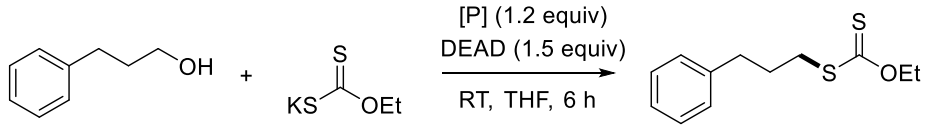
**I. General information**.

All reactions were carried out under an argon atmosphere using standard Schlenk-Lines. $^{1}H$, $^{13}C$ and $^{19}F$ NMR spectra were acquired on 400 MHz, 101 MHz, 376 MHz on JOEL-ZETA 400 MHz or Bruker-AVANCE III-400 MHz spectrometer (400 MHz for $^{1}H$; 101 MHz for $^{13}C$; 376 MHz for $^{19}F$). $^{1}H$ NMR and $^{13}C$ NMR chemical shifts were determined relative to internal standard TMS at δ 0.0 ppm and $^{19}F$ NMR chemical shifts were determined relative to $CFCl_3$ as inter standard. Chemical shifts (δ) are reported in ppm, and coupling constants (*J*) are in hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. All reactions were monitored by TLC with 0.25 mm coated commercial silica gel plates (TLC Silica Gel 60 $F_{254}$). Flash column chromatograph was carried out using 300-400 mesh silica gel at medium pressure. Infrared (IR) data were recorded as films on potassium bromide plates on a Bruker Tensor 27 FT-IR spectrometer. Absorbance frequencies are reported in reciprocal cetimeters ($cm^{-1}$). Mass spectra were acquired on a Bruker Daltonics MicroTof-Q II mass spectrometer or Agilent 7890B-5977A mass spectrometer.

**Materials.** All reagents were received from commercial sources unless otherwise noted. Solvents were freshly dried and degassed according to the purification handbook Purification of Laboratory Chemicals before using.

## II. Optimization of reaction conditions

**Table S1. Initial attempt on Mitsunobu reaction of alkyl alcohol and potassium ethylxanthate.** [a, b]

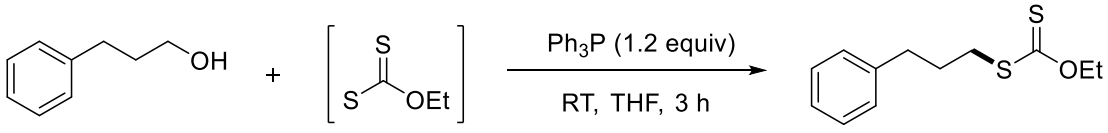



**1as** **4as**

| Entry | **1as** (equiv) | potassium ethylxanthate (equiv) | [P] | Yield (**4as**, %)[b] |
|---|---|---|---|---|
| 1 | 1 | 1.5 | $PCy_3$ | ND |
| 2 | 1 | 1.5 | $P(^nBu)_3$ | ND |
| 3 | 1 | 1.5 | $PPh_3$ | ND |
| 4 | 1 | 1.5 | tris(4-fluorophenyl)phosphine | ND |
| 5 | 1 | 1.5 | tris(4-methoxyphenyl)phosphine | ND |

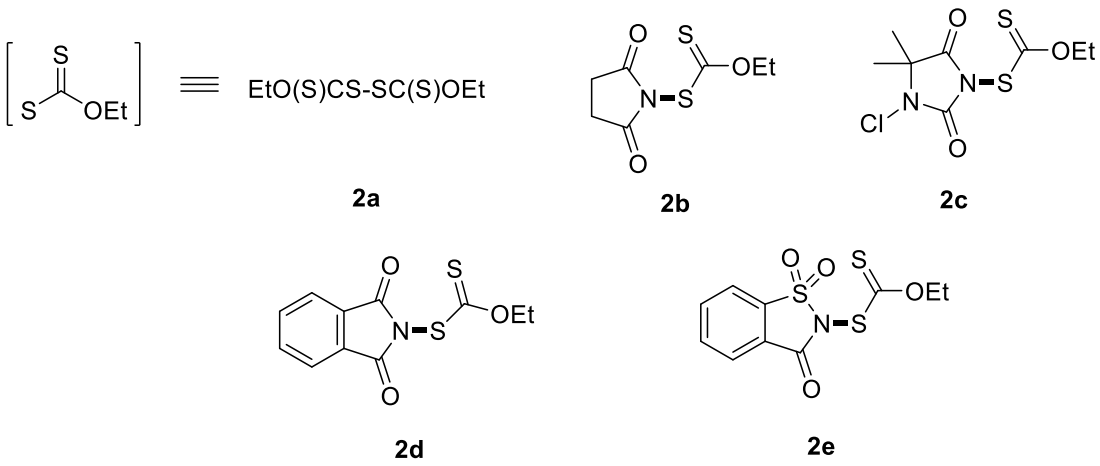
[a] Standard conditions: 3-phenyl-1-propanol (**1as**, 27.2 mg, 0.20 mmol), potassium ethylxanthate (48.1 mg, 0.30 mmol, 1.5 equiv), DEAD (52.2 mg, 0.30 mmol, 1.5 equiv) and $PPh_3$ (62.9 mg, 0.24 mmol, 1.2 equiv) were added in 2.0 mL THF and reacted at RT for 6 h. [b] Yields were determined by $^1H$ NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard.

**Table S2. Optimization of electrophilic xanthyl reagents.** [a, b]




**1as** **2** **4as**

| Entry | **1as** (1 equiv) | **2** (1.2 equiv) | Yield (**4as**, %)[b] |
|---|---|---|---|
| 1 | 1 | **2a** | ND |
| 2 | 1 | **2b** | Trace |
| 3 | 1 | **2c** | ND |
| 4 | 1 | **2d** | 55 |
| 5 | 1 | **2e** | 20 |

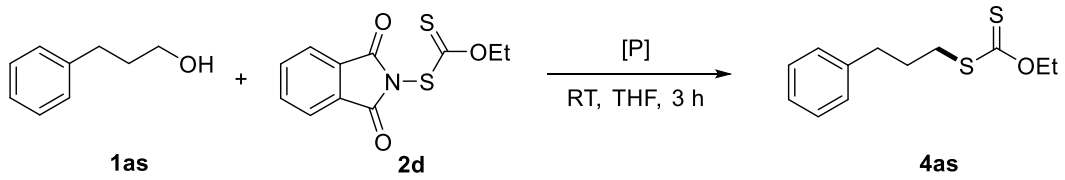



**2a** **2b** **2c**

**2d** **2e**

[a] Standard conditions: 3-phenyl-1-propanol (**1as**, 27.2 mg, 0.20 mmol), [SC(S)OEt] (**2**, 0.24 mmol, 1.2 equiv) and $PPh_3$ (62.9 mg, 0.24 mmol, 1.2 equiv) were added in 2.0 mL THF and reacted at RT for 3 h. [b] Yields were determined by $^1H$ NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard.

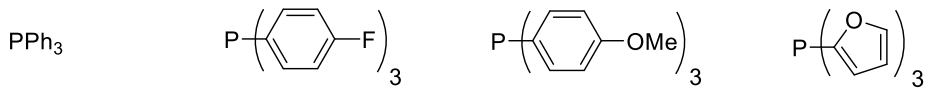
**Table S3. Optimization on phosphine activator.** [a, b]




**1as** **2d** **4as**

| Entry | **1as** (equiv) | **2d** (equiv) | [P] (1.2 equiv) | Yield (**4as**, %)[b] |
|---|---|---|---|---|
| 1 | 1 | 1.2 | **3a** | ND |
| 2 | 1 | 1.2 | **3b** | 16 |
| 3 | 1 | 1.2 | **3c** | 46 |
| 4 | 1 | 1.2 | **3d** | ND |
| 5 | 1 | 1.2 | **3e** | 55 |
| 6 | 1 | 1.2 | **3f** | 48 |
| 7 | 1 | 1.2 | **3g** | 17 |
| 8 | 1 | 1.2 | **3h** | 18 |

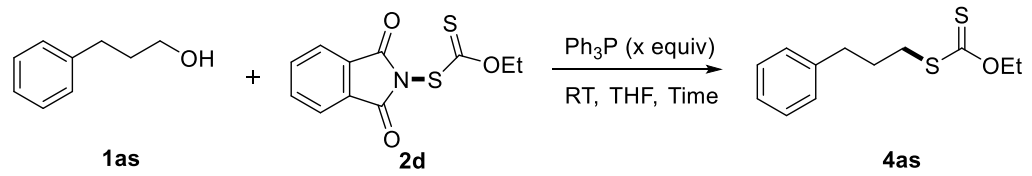
| $PCy_3$ | $P(NMe_2)_3$ | $P(^nBu)_3$ | $P(OMe)_3$ |
|---|---|---|---|
| **3a** | **3b** | **3c** | **3d** |




**3e** **3f** **3g** **3h**

[a] Standard conditions: 3-phenyl-1-propanol (**1as**, 27.2 mg, 0.20 mmol), **2d** (64.1 mg, 0.24 mmol, 1.2 equiv) and [P] (0.24 mmol, 1.2 equiv) were added in 2.0 mL THF and reacted at RT for 3 h; [b]Yields were determined by $^1$H NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard.

**Table S4. Optimization of reaction conditions for direct dehydroxyxanthylation of alcohols.** [a, b]



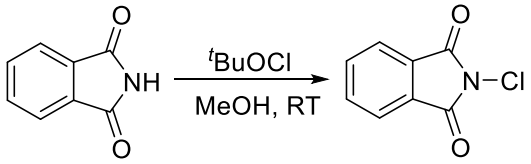

**1as** **2d** **4as**

| Entry | **1as:2d** | $Ph_3P$ (x equiv) | Solvent | Time (h) | Atmosphere | Yield (**4as**, %)[b] |
|---|---|---|---|---|---|---|
| 1 | 1:1.2 | 1.2 | THF | 3 | air | 55 |
| 2 | 1:1.3 | 1.2 | THF | 3 | air | 64 |
| 3 | 1:1.5 | 1.2 | THF | 3 | air | 68 |
| 4 | 1:1.5 | 1.2 | DCE | 3 | air | 46 |
| 5 | 1:1.5 | 1.2 | $CH_3CN$ | 3 | air | Trace |
| 6 | 1:1.5 | 1.2 | THF | 3 | $N_2$ | 82 |
| 7 | 1:1.5 | 1.2 | THF | 0.5 | $N_2$ | 87 (84)[c] |
| 8 | 1:1.5 | 1.2 | THF | 1 | $N_2$ | 67 |
| 9 | 1:1.5 | 1.2 | THF | 6 | $N_2$ | 72 |
| 10 | 1:1.5 | 1.2 | THF | 12 | $N_2$ | 83 |
| 11 | 1:1.2 | 1.2 | THF | 0.5 | $N_2$ | 74 |
| 12 | 1:1 | 1.2 | THF | 0.5 | $N_2$ | 47 |
| 13 | 1.2:1 | 1.2 | THF | 0.5 | $N_2$ | 52 |
| 14 | 1.5:1 | 1.2 | THF | 0.5 | $N_2$ | 55 |
| 15 | 1:1.5 | 1.0 | THF | 0.5 | $N_2$ | 70 |
| 16 | 1:1.5 | 0.7 | THF | 0.5 | $N_2$ | 42 |
| 17 | 1:1.5 | 0.3 | THF | 0.5 | $N_2$ | 27 |

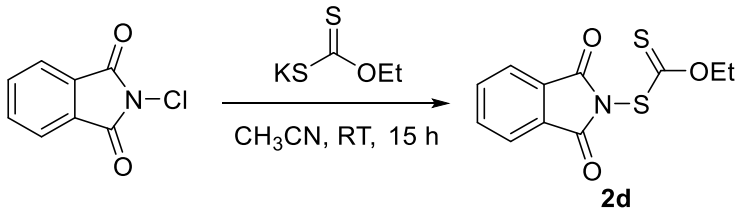
[a] Standard conditions: 3-phenyl-1-propanol (**1as**), **2d** and $PPh_3$ (x equiv) were added in 2.0 mL solvent and reacted at RT; [b]Yields were determined by $^1H$ NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard. [c]Isolated yield.

## III. General procedure

### 1. General procedure for the preparation of *N*-ethylxanthyl phthalimide 2d and its analogues.

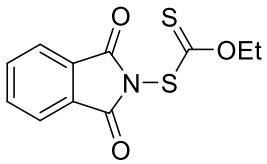



**Preparation of N-chlorophthalimide.**[1] To a suspension of phthalimide (7.4 g, 50.0 mmol) in MeOH (500 mL) was added $^{t}$BuOCl (7.3 g, 7.6 mL, 67.4 mmol, 1.35 equiv) quickly. The mixture was stirred for 5 min and standing for 5 min. The precipitate was filtered and dried under high vacuum to obtain a white powder (8.9 g, 98% yield). No further purification is required and can be used directly for the next step.



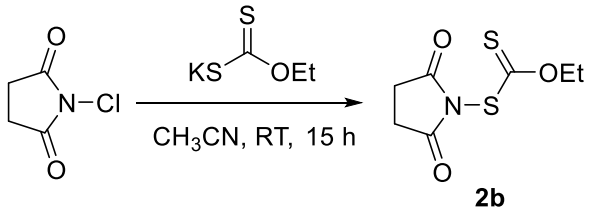

**Synthesis of *N*-ethylxanthyl phthalimide 2d.**[2] In a 1 L round-bottom flask, potassium ethyl xanthate (7.9 g, 49.0 mmol, 1.0 equiv) was suspended in MeCN (300 mL). To this suspension was added a solution of *N*-chlorophthalimide (8.9 g, 49.0 mmol, 1.0 equiv) in MeCN (300 mL) *via* constant pressure funnel over 20 min. The reaction mixture was stirred for 15 h and the suspension was concentrated in vacuo. The resultant solid was purified by flash column chromatography to afford the title compound.

**S-(1,3-Dioxoisoindolin-2-yl) O-ethyl carbonodithioate 2d.**




White solid (9.4 g, 72%). Mp: 115-116 °C. Eluent: ethyl acetate/petroleum ether (1:5, $R_f$ = 0.3). $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.82 (dd, *J* = 5.6, 3.1 Hz, 2 H), 4.60 (q, *J* = 7.1 Hz, 2 H), 1.30 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101

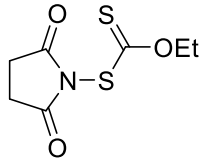
MHz, $CDCl_3$) δ 208.8, 166.2, 135.1, 132.1, 124.3, 71.4, 13.6 ppm. Spectra were consistent with literature data.[3]



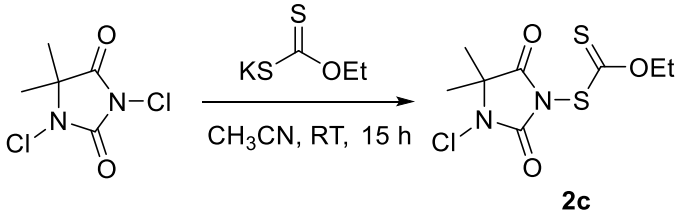

**2b**

**Synthesis of reagent 2b.** According to the synthesis method of *N*-ethylxanthyl phthalimide **2d**, potassium ethyl xanthate (8.0 g, 50.0 mmol, 1.0 equiv) was suspended in MeCN (300 mL). To this suspension was added a solution of *N*-chlorosuccinimide (6.7 g, 50.0 mmol, 1.0 equiv) in MeCN (200 mL) *via* constant pressure funnel over 20 min. The reaction mixture was stirred for 15 h and the suspension was concentrated in vacuo. The resultant solid was purified by flash column chromatography to afford the title compound.

***S*-(2,5-Dioxopyrrolidin-1-yl) *O*-ethyl carbonodithioate 2b.**




Yellow oil (1.1 g, 10%). Eluent: ethyl acetate/petroleum ether (1:5, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.60 (q, *J* = 7.1 Hz, 2 H), 2.81 (s, 4 H), 1.43 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 184.4, 172.6, 70.7, 28.5, 13.4 ppm. Spectra were consistent with literature data.[3]

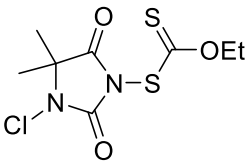



**2c**

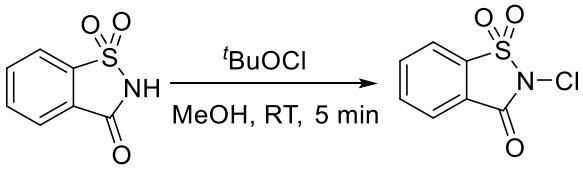
**Synthesis of reagent 2c.** According to the synthesis method of *N*-ethylxanthyl phthalimide **2d**, potassium ethyl xanthate (4.0 g, 25.0 mmol, 2.5 equiv) was suspended in MeCN (300 mL). To this suspension was added a solution of 1,3-dichloro-5,5-dimethylhydantoin (2.0 g, 10.0 mmol, 1.0 equiv) in MeCN (200 mL) *via* constant

pressure funnel over 20 min. The reaction mixture was stirred for 15 h and the suspension was concentrated in vacuo. The resultant solid was purified by flash column chromatography to afford the title compound.

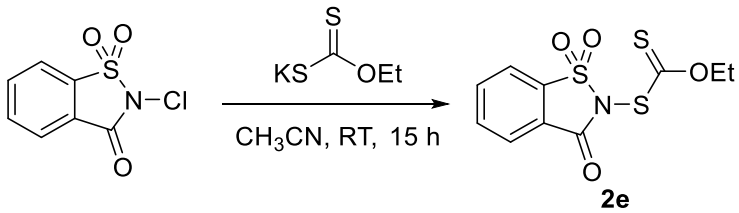
***S*-(3-Chloro-4,4-dimethyl-2,5-dioxoimidazolidin-1-yl) *O*-ethylcarbonodithioate 2c.**



Yellow solid (933.1 mg, 33%). Mp: 92-93 °C. Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.62 (q, *J* = 7.1 Hz, 2 H), 1.48 (s, 6 H), 1.45 (d, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 183.2, 172.9, 152.0, 70.1, 58.5, 25.2, 13.5 ppm. Spectra were consistent with literature data.[3]



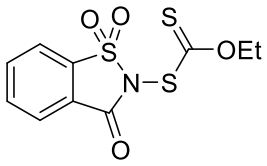

**Preparation of N-chlorosaccharin.**[1] To a suspension of saccharin (12.0 g, 65.6 mmol) in MeOH (120 mL) was added $^t$BuOCl (9.6 g, 10.0 mL, 88.4 mmol, 2.7 equiv) quickly. The suspension turned to clear solution and quickly a large amount of white precipitate was formed. The mixture was stirred for 5 min and standing for 5 min. The precipitate was filtered and dried under high vacuum to obtain a white powder (12.0 g, 84% yield). No further purification is required and can be used directly for the next step.




**Synthesis of reagent 2e.** According to the synthesis method of *N*-ethylxanthyl phthalimide **2d**, potassium ethyl xanthate (5.2 g, 32.2 mmol, 1.0 equiv) was suspended in MeCN (300 mL). To this suspension was added a solution of *N*-chlorosaccharin (7.0 g, 32.2 mmol, 1.0 equiv) in MeCN (200 mL) *via* constant pressure funnel over 20 min. The reaction mixture was stirred for 15 h and the suspension was concentrated in vacuo.

The resultant solid was purified by flash column chromatography to afford the title compound.

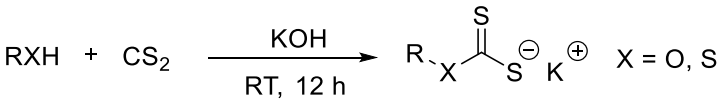
**_S_-(1,1-Dioxido-3-oxobenzo[_d_]isothiazol-2(3_H_)-yl) _O_-ethyl carbonodithioate 2e.**



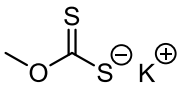
White solid (2.0 g, 20%). Mp: 100-101 °C. Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.19–8.14 (m, 1 H), 7.99–7.92 (m, 2 H), 7.92–7.86 (m, 1 H), 4.73 (q, *J* = 7.1 Hz, 2 H), 1.51 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 180.4, 156.2, 136.4, 135.0, 126.5, 125.8, 121.4, 121.3, 69.0, 13.5 ppm. Spectra were consistent with literature data.[3]

**2. General procedure for the synthesis of potassium xanthate** [4]

Primary alcohols (1.0 equiv) were treated with KOH (1.0 equiv) and carbon disulfide (1.2 equiv) in diethyl ether. The reaction mixture was stirred at room temperature for 3-12 h. The precipitates were collected by filtration, washed with diethyl ether, and dried in vacuo to afford the title products.

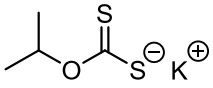


**Synthesis of potassium _O_-methyl carbonodithioate salt.**



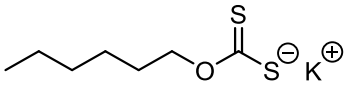
Methanol (640 mg, 0.8 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a white solid (1.81 g, 62% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium _O_-isopropyl carbonodithioate salt.**



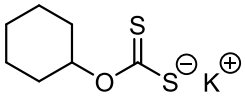
Isopropyl alcohol (1.2 g, 1.5 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a pale-yellow solid (2.78 g, 80% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-hexyl carbonodithioate salt.**



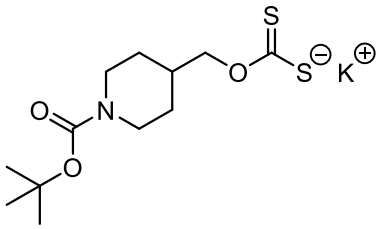
Hexan-1-ol (2.04 g, 2.5 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a white solid (3.37 g, 78% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-cyclohexyl carbonodithioate salt.**



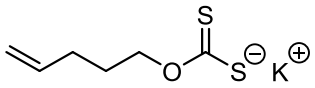
Cyclohexanol (2.0 g, 2.1 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a pale-yellow solid (3.30 g, 77% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-((1-(*tert*-butoxycarbonyl)piperidin-4-yl)methyl) carbonodithioate salt.**



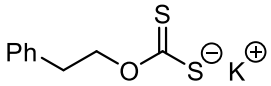
*N*-Boc-4-piperidinemethanol (4.3 g, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a pale-yellow solid (4.80 g, 73% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-(pent-4-en-1-yl) carbonodithioate salt.**



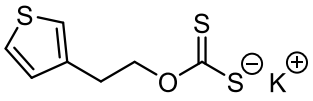
Pent-4-en-1-ol (1.7 g, 2.1 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a white solid (3.16 g, 79% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-phenethyl carbonodithioate salt.**



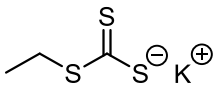
2-Phenylethanol (2.44 g, 2.4 mL, 20 mmol) was treated with crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.83 g, 24 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a pale-yellow solid (4.29 g, 91% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium *O*-(2-(thiophen-3-yl)ethyl) carbonodithioate salt.**



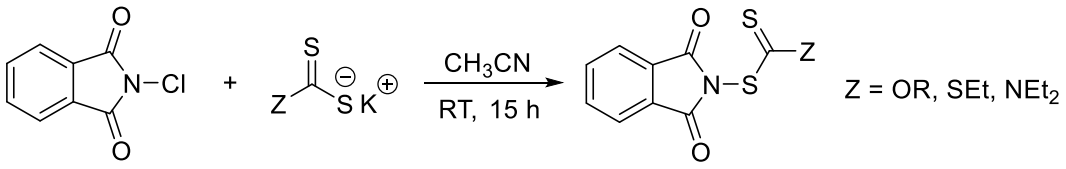
2-(Thiophen-3-yl)ethan-1-ol (2.4 g, 2.1 mL, 18.7 mmol) was treated with crushed potassium hydroxide (1.05 g, 18.7 mmol, 1.0 equiv) followed by addition of carbon disulfide (1.70 g, 22.4 mmol, 1.2 equiv). The solution was stirred at room temperature for 12 h and then concentrated in vacuo to give a residue that was washed with diethyl ether and dried in vacuo to afford the title product as a pale-yellow solid (1.36 g, 30% yield). No further purification is required and can be used directly for the next step.

**Synthesis of potassium ethyl carbonotrithioate salt.**



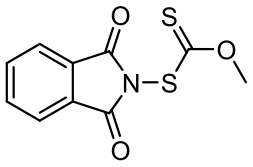
Ethanethiol (1.24 g, 1.5 mL, 20 mmol) was treated with freshly crushed potassium hydroxide (1.12 g, 20 mmol, 1.0 equiv) and carbon disulfide (1.68 g, 22 mmol, 1.1 equiv) in diethyl ether (15 mL). The suspension was stirred at room temperature for 3 h, and the white precipitate was collected by filtration, washed with diethyl ether and dried in vacuo to afford the desired product as a yellow solid (3.27 g, 93% yield). No further purification is required and can be used directly for the next step.

## 3. General procedure for the synthesis of xanthate-derived reagents.



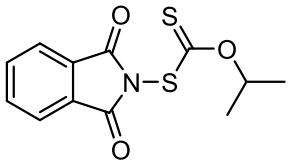

**According to the synthesis method of *N*-ethylxanthyl phthalimide 2d.** In a 1 L round-bottom flask, potassium thiolate (1.0 equiv) was suspended in MeCN. To this suspension was added a solution of *N*-chlorophthalimide (1.0 equiv) in MeCN via constant pressure funnel over 20 min. The reaction mixture was stirred for 15 h and the suspension was concentrated in vacuo. The resultant solid was purified by flash column chromatography to afford the title compounds.

***S*-(1,3-Dioxoisoindolin-2-yl) *O*-methyl carbonodithioate 2aa**



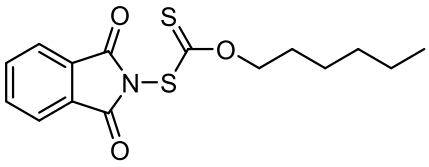
Potassium O-methyl carbonodithioate salt (730 mg, 4.99 mmol) reacts with N-chlorophthalimide (907 mg, 4.99 mmol) to afford **2aa** (810 mg, 64% yield) as a white solid (mp: 154-155 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.98 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.84 (dd, *J* = 5.5, 3.1 Hz, 2 H), 4.16 (s, 3 H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 209.8, 166.2, 135.2, 132.2, 124.5, 61.2 ppm. HRMS (ESI): m/z for $C_{10}H_7NO_3S_2$ $[M+Na]^+$ calcd 275.9760, found 275.9756.

***S*-(1,3-Dioxoisoindolin-2-yl) *O*-isopropyl carbonodithioate 2ab**



Potassium O-isopropyl carbonodithioate (870 mg, 4.99 mmol) reacts with N-chlorophthalimide (907 mg, 4.99 mmol) to afford **2ab** (960 mg, 68% yield) as a white solid (mp: 114-115 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.83 (dd, *J* = 5.5, 3.1 Hz, 2 H), 5.62 (m, 1 H), 1.28 (d, *J* = 6.3 Hz, 6 H).$^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.1, 166.4, 135.2, 132.1, 124.4, 80.4, 21.3.ppm. HRMS (ESI): m/z for $C_{12}H_{11}NO_3S_2$ $[M+Na]^+$ calcd 304.0073, found 304.0068.

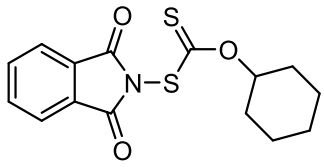
***S*-(1,3-Dioxoisoindolin-2-yl) *O*-hexyl carbonodithioate 2ac**



Potassium O-hexyl carbonodithioate salt (1.1 g, 4.99 mmol) reacts with N-chlorophthalimide (907 mg, 4.99 mmol) to afford **2ac** (680 mg, 42% yield) as a white solid (mp: 63-64 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.82 (dd, *J* =

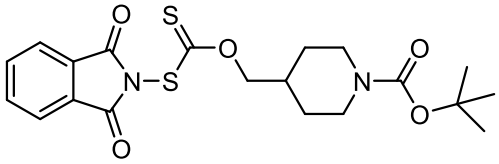
5.6, 3.1 Hz, 2 H), 4.49 (t, *J* = 6.4 Hz, 2 H), 1.66 – 1.53 (m, 2 H), 1.19 – 1.06 (m, 6 H), 0.75 (t, *J* = 6.8 Hz, 3 H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.7, 166.2, 135.2, 132.0, 124.4, 75.4, 31.2, 28.0, 25.4, 22.5, 14.0 ppm. HRMS (ESI): m/z for $C_{15}H_{17}NO_3S_2$ $[M+Na]^+$ calcd 346.0542, found 346.0545.

***O*-Cyclohexyl *S*-(1,3-dioxoisoindolin-2-yl) carbonodithioate 2ad**



Potassium O-cyclohexyl carbonodithioate salt (1.1 g, 4.99 mmol) reacts with N-chlorophthalimide (907 mg, 4.99 mmol) to afford **2ad** (1.2 g, 76% yield) as a white solid (mp: 117-118 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.99 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.84 (dd, *J* = 5.5, 3.1 Hz, 2 H), 5.48 (tt, *J* = 7.9, 3.7 Hz, 1 H), 1.85 (d, *J* = 13.2 Hz, 2 H), 1.60 – 1.37 (m, 5 H), 1.37 – 1.26 (m, 2 H), 1.24 – 1.15 (m, 1 H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.2, 163.8, 136.6, 131.1, 126.4, 83.4, 31.9, 26.8, 22.5 ppm. HRMS (ESI): m/z for $C_{15}H_{15}NO_3S_2$ $[M+Na]^+$ calcd 344.0386, found 344.0387.

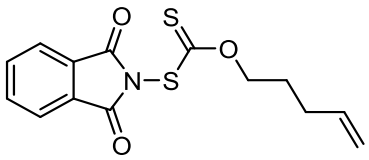
***Tert*-butyl 4-(((((1,3-dioxoisoindolin-2-yl)thio)carbonothioyl)oxy)methyl)piperidine-1-carboxylate 2ae**



Potassium O-((1-(*tert*-butoxycarbonyl)piperidin-4-yl)methyl) carbonodithioate salt (1.6 g, 4.99 mmol) reacts with N-chlorophthalimide (907 mg, 4.99 mmol) to afford **2ae** (1.4 g, 62% yield) as a white solid (mp: 121-122 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.83 (dd, *J* = 5.5, 3.1 Hz, 2 H), 4.38 (d, *J* = 6.5 Hz, 2 H), 3.99 (d, *J* = 11.4 Hz, 2 H), 2.55 (s, 2 H), 1.82 (s, 1 H), 1.52 (d, *J* = 14.5 Hz, 2 H), 1.42 (s, 9 H), 1.06 (m, 2 H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.7, 166.3, 154.7, 135.3, 132.1, 124.5, 79.6, 78.6, 35.4, 28.5, 28.4 ppm. HRMS (ESI): m/z for $C_{20}H_{24}N_2O_5S_2$ [M+Na]+ calcd

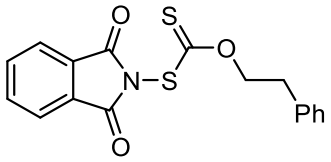
459.1019, found 459.1022.

***S*-(1,3-Dioxoisoindolin-2-yl) *O*-(pent-4-en-1-yl) carbonodithioate 2af**



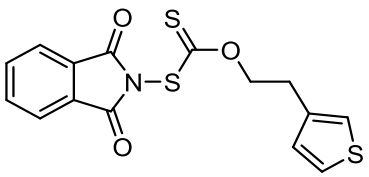
Potassium O-(pent-4-en-1-yl) carbonodithioate salt (3.0 g, 14.97 mmol) reacts with N-chlorophthalimide (2.7 g, 14.97 mmol) to afford **2af** (2.3 g, 50% yield) as a white solid (mp: 44-45 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 5.4, 3.3 Hz, 2 H), 7.83 (dd, *J* = 5.5, 3.1 Hz, 2 H), 5.73–5.50 (m, 1 H), 4.93–4.82 (m, 2 H), 4.53 (t, *J* = 6.4 Hz, 2 H), 1.96 (q, *J* = 7.3 Hz, 2 H), 1.78–1.69 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.7, 166.3, 136.7, 135.2, 132.1, 124.5, 115.9, 74.5, 29.7, 27.3 ppm. Spectra were consistent with literature data.[3]

***S*-(1,3-Dioxoisoindolin-2-yl) *O*-phenethyl carbonodithioate 2ag**



Potassium O-phenethyl carbonodithioate salt (3.9 g, 16.37 mmol) reacts with N-chlorophthalimide (3.0 g, 16.37 mmol) to afford **2ag** (3.9 g, 70% yield) as a white solid (mp: 103-104 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.7). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.84 (dd, *J* = 5.6, 3.1 Hz, 2 H), 7.13 (d, *J* = 3.0 Hz, 3 H), 7.03 (s, 2 H), 4.76 (t, *J* = 6.9 Hz, 2 H), 2.98 (t, *J* = 6.9 Hz, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.7, 166.2, 136.5, 135.1, 132.1, 128.7, 128.6, 126.9, 124.5, 75.0, 34.5 ppm. Spectra were consistent with literature data.[3]

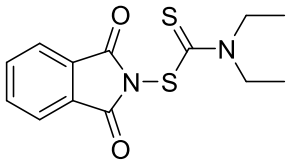
***S*-(1,3-Dioxoisoindolin-2-yl) *O*-(2-(thiophen-3-yl)ethyl) carbonodithioate 2ah**



Potassium O-(2-(thiophen-3-yl)ethyl) carbonodithioate salt (922 mg, 3.81 mmol) reacts with N-chlorophthalimide (691 mg, 3.81 mmol) to afford **2ah** (1.2 g, 91% yield) as a

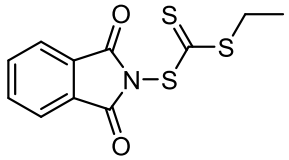
white solid (mp: 90-91 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.83 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.13 (dd, *J* = 5.0, 3.0 Hz, 1 H), 6.82 (s, 1 H), 6.79 (d, *J* = 5.1 Hz, 1 H), 4.74 (t, *J* = 6.7 Hz, 2 H), 2.99 (t, *J* = 6.7 Hz, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 208.7, 166.2, 136.6, 135.2, 132.0, 127.9, 126.1, 124.5, 121.9, 74.4, 29.0 ppm. Spectra were consistent with literature data.[3]

**1,3-Dioxoisoindolin-2-yl diethylcarbamodithioate 2ai**




Sodium diethyldithiocarbamate (1.7 g, 10.0 mmol) reacts with N-chlorophthalimide (1.8 g, 10.0 mmol) to afford **2ai** (941 mg, 32% yield) as a yellow solid (mp: 109-110 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 5.5, 3.0 Hz, 2 H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2 H), 4.05 (q, *J* = 7.1 Hz, 2 H), 3.51 (q, *J* = 7.3 Hz, 2 H), 1.41 (t, *J* = 7.2 Hz, 3 H), 1.23 (t, *J* = 7.3 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 174.4, 165.1, 134.8, 132.2, 124.3, 48.3, 47.6, 13.9, 11.1 ppm. Spectra were consistent with literature data.[3]

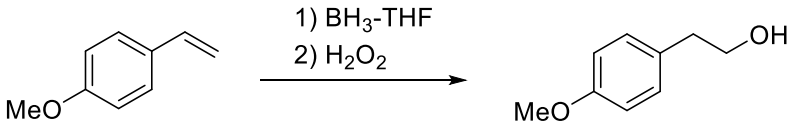
**1,3-Dioxoisoindolin-2-yl ethyl carbonotrithioate 2aj**




Potassium ethyl carbonotrithioate salt (1.8 g, 10.0 mmol) reacts with N-chlorophthalimide (1.8 g, 10.0 mmol) to afford **2aj** (2.2 g, 79% yield) as a yellow solid (mp: 130-131 °C) after chromatography (eluent: ethyl acetate/petroleum ether 1:5, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 5.5, 3.1 Hz, 2 H), 7.86 (dd, *J* = 5.5, 3.1 Hz, 2 H), 3.31 (d, *J* = 14.9 Hz, 2 H), 1.29 (t, *J* = 7.5 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 222.0, 166.1, 135.3, 131.9, 124.7, 31.1, 12.7 ppm. Spectra were consistent with literature data.[3]

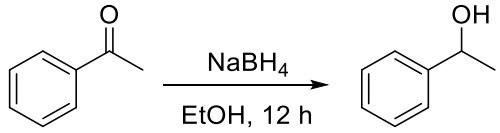
**4. General procedure for the synthesis of hydroxylated substrates.**

**General procedure for the synthesis of 2-(4-methoxyphenyl)ethan-1-ol 7aa**



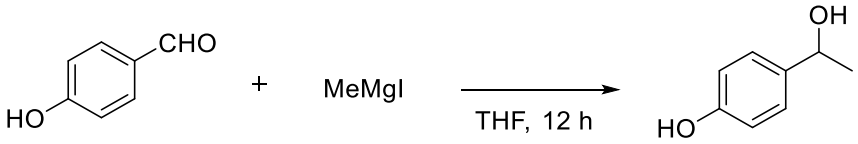

To 1-methoxy-4-vinylbenzene (1.3 g, 10 mmol) in THF solution was slowly added $BH_3$-THF (1.0 M, 1.1 equiv). The reaction mixture is stirred at room temperature for 3 h. Then 10 mL of 30% hydrogen peroxide solution was added to the reaction mixture at 0 °C and the resulting mixture reacted for another 30 min. The reaction mixture was extracted with 3×20 mL of ether and washed with 50 mL of saturated sodium chloride solution. The combined organic layers were dried with magnesium sulfate and the solvent was evaporated under reduced pressure. The crude product was purified by flash silica gel column chromatography to a white soild (1.3 g, 83% yield). Spectra were consistent with literature data. [5]

**General procedure for the synthesis of 1-phenylethan-1-ol 7ab**




To a 100 mL round-bottom flask were added acetophenone (1.2 g, 10.0 mmol, 1.0 equiv), $NaBH_4$ (800 mg, 20.0 mmol, 2.0 equiv) and anhydrous EtOH (30 mL). The reaction mixture was stirred at room temperature for 12 h. The filtrate was concentrated, and the residue was purified with silica gel chromatography to obtain a colorless oil (1.1 g, 86% yield). Spectra were consistent with literature data. [6]

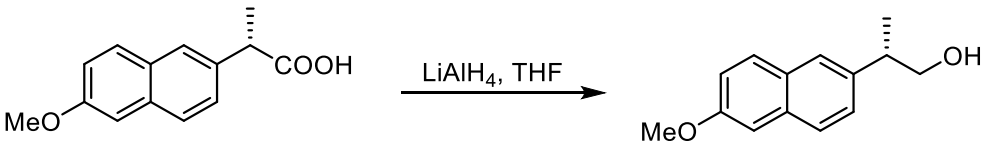
**General procedure for the synthesis of 4-(1-hydroxyethyl)phenol 7ac**




To a solution of 4-hydroxybenzaldehyde (1.2 g, 10.0 mmol, 1.0 equiv) in THF at 0 ℃ was slowly added methyl methylmagnesium iodide (3M in diethylether, 3.0 equiv) under Ar atmosphere. The reaction was stirred overnight at room temperature. Then the reaction mixture was quenched with saturated $NH_4Cl$ solution, and extracted with ethyl

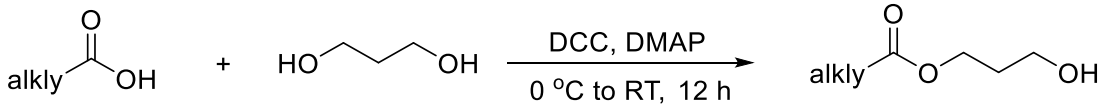
acetate. The combined organic layers were washed with saturated sodium chloride solution, and dried over anhydrous $Na_2SO_4$. The solvent is evaporated under reduced pressure and crude residue was purified by silica gel chromatography to obtain a white soild (553 mg, 40%). Spectra were consistent with literature data. [7]

**General procedure for the synthesis of (*S*)-2-(6-methoxynaphthalen-2-yl)propan-1-ol 9aa**



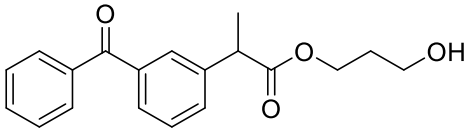

To the stirred suspension of $LiAlH_4$ (379 mg, 10.0 mmol, 2.0 equiv) in THF (48.0 mL) was slowly added the solution of (*S*)-2-(6-methoxynaphthalen-2-yl)propanoic acid (1.2 g, 5.00 mmol, 1.0 equiv) in THF (5.00 mL) at 0 ℃ under $N_2$ atmosphere. The resulting mixture was allowed to warm to room temperature and stirred for 30 min. The reaction mixture was cooled to 0 °C and quenched by addition of 10% NaOH aq. (0.2 mL). Then AcOEt and celite were added to the mixture. The resulting mixture was stirred vigorously for 10 min and filtered. The filtrate was evaporated in vacuo and the residue was purified by chromatography on silica gel ethyl acetate/petroleum ether (1:2), to give (*S*)-2-(6-methoxynaphthalen-2-yl)propan-1-ol (820 mg, 76%) as a white soild. Spectra were consistent with literature data.[8]

**General procedure for the synthesis of 3-hydroxypropyl 2-(3-benzoylphenyl)propanoate**



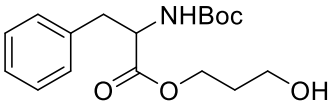

To a 250 mL round-bottom flask were added acid (20.0 mmol, 1.0 equiv), DMAP (2.0 mmol, 10 mol%), anhydrous DCM (50 mL), and 3-bromopropan-1-ol (3.1 g, 22.0 mmol, 1.1 equiv). A solution of DCC (22.0 mmol, 1.1 equiv) in DCM (10.0 mL) was added dropwise at 0 °C. After stirring for 12 h at room temperature, the reaction mixture was filtered with a pad of celite and washed with ethyl acetate. The filtrate was concentrated, and the residue was purified with silica gel chromatography.

**3-Hydroxypropyl 2-(3-benzoylphenyl)propanoate 9ab**



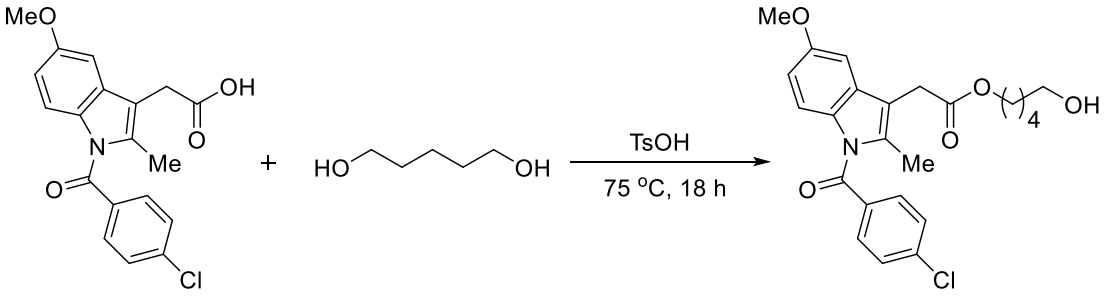
Colorless oil (3.5 g, 56%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.77 – 7.69 (m , 3 H), 7.60 (dt, *J* = 7.6, 1.5 Hz, 1 H), 7.56 – 7.47 (m, 2 H), 7.46 – 7.34 (m, 3 H), 4.17 (td, *J* = 6.4, 1.6 Hz, 2 H), 3.76 (q, *J* = 7.2 Hz, 1 H), 3.52 (q, *J* = 5.3 Hz, 2 H), 2.84 (s, 1 H), 1.77 (m, 2 H), 1.48 (d, *J* = 7.2 Hz, 3 H) ; $^{13}$C NMR (101 MHz, $CDCl_3$) δ 196.9, 174.5, 141.0, 137.9, 137.4, 132.8, 131.7, 130.2, 129.2, 128.7, 128.4, 62.0, 58.8, 45.5, 31.6, 18.5 ppm. HRMS (ESI): m/z for $C_{19}H_{20}O_4$ $[M+Na]^+$ calcd 335.1254, found 335.1251.

**3-Hydroxypropyl (*tert*-butoxycarbonyl)phenylalaninate 9ac**



Colorless oil (4.7 g, 72%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.15 (m, 3H), 7.13 – 7.07 (m, 2H), 5.23 (d, *J* = 8.2 Hz, 1H), 4.53 – 4.45 (m, 1H), 4.27 – 4.09 (m, 2H), 3.53 (m, 2H), 3.04 (m, 3H), 1.81 – 1.71 (m, 2H), 1.36 (s, 9H) ; $^{13}$C NMR (101 MHz, $CDCl_3$) δ 172.4, 155.4, 136.2, 129.4, 128.6, 127.1, 80.0, 77.6, 62.5, 58.6, 54.7, 38.3, 31.4, 28.4 ppm. HRMS (ESI): m/z for $C_{17}H_{25}NO_5$ $[M+Na]^+$ calcd 346.1625, found 346.1616.

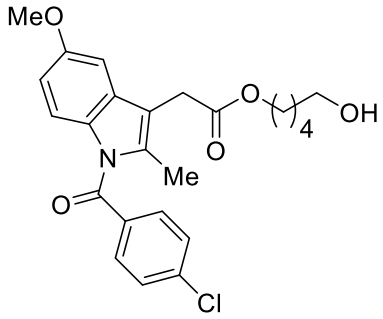
**General procedure for the synthesis of 5-hydroxypentyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate**




An oven-dried 50-mL round-bottom flask, equipped with a stir bar, was charged with butanediol (9.0 g, 10.6 mL, 100 mmol, 20 equiv). Then indomethacin (1.8 g, 5 mmol, 1 equiv) and *p*-toluenesulfonic acid (43 mg, 0.25 mmol, 0.05 equiv) was added to the

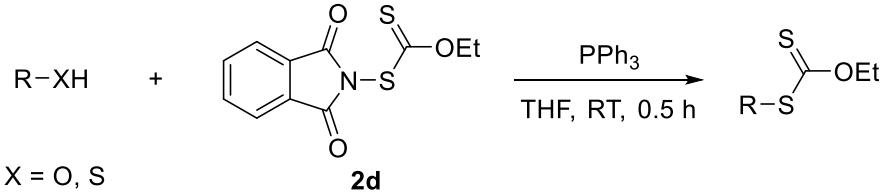
solution. The mixture was stirred at 75 °C for 18 h. Finally, the reaction mixture was quench with 10 % $Na_2CO_3$ aqueous solution and washed with ethyl acetate. The solvent was removed under reduced pressure and the residue was purified with silica gel chromatography.

**5-Hydroxypentyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate 9ad**




Yellow solid (730 mg, 33%). Mp: 68-69 °C. Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.1).$^1$H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.6 Hz, 2 H), 7.45 (d, *J* = 8.5 Hz, 2 H), 6.96 (d, *J* = 2.5 Hz, 1 H), 6.86 (d, *J* = 9.0 Hz, 1 H), 6.65 (dd, *J* = 9.0, 2.6 Hz, 1 H), 4.09 (t, *J* = 6.5 Hz, 2 H), 3.82 (s, 3 H), 3.65 (s, 2 H), 3.55 (t, *J* = 6.5 Hz, 2 H), 2.37 (s, 3 H), 1.84 – 1.72 (m, 1 H), 1.68 – 1.57 (m, 2 H), 1.57 – 1.45 (m, 2 H), 1.39 – 1.30 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 171.1, 168.5, 156.1, 139.4, 136.0, 134.0, 131.3, 130.9, 130.8, 129.2, 115.0, 112.8, 111.6, 101.6, 65.1, 62.6, 55.8, 32.3, 30.5, 28.5, 22.3, 13.5 ppm. HRMS (ESI): m/z for $C_{24}H_{26}ClNO_5$ $[M+Na]^+$ calcd 466.1392, found 466.1385.

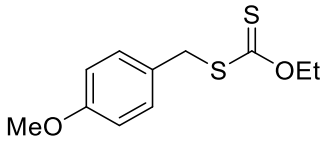
**5. General procedure for direct dehydroxy(sulfhydryl)xanthylation of alcohols and thiols with *N*-ethylxanthyl phthalimide 2d**




Alkyl alcohol or thiol (0.5 mmol, 1.0 equiv), N-ethylxanthyl phthalimide **2d** (200.2 mg, 0.75 mmol, 1.5 equiv), $PPh_3$ (157.4 mg, 0.6 mmol, 1.2 equiv) were placed into an oven-dried 25 mL Schlenk tube that was equipped with a stirring bar. Then Freshly distilled

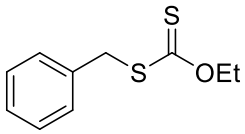
THF (3.0 mL) was added to the Schlenk tube under nitrogen atmosphere. The reaction was stirred at room temperature for 0.5 h. The solvent was removed under vacuum and the residue was purified by flask column chromatography to obtain alkyl xanthate products.

***O*-Ethyl *S*-(4-methoxybenzyl) carbonodithioate 4aa**



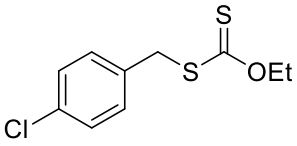

Colorless oil (104.1 mg, 86%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.8). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.23 (m, 2 H), 6.84 (d, *J* = 8.7 Hz, 2 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 4.31 (s, 2 H), 3.78 (s, 3 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.4, 159.1, 130.4, 127.5, 114.1, 70.1, 55.4, 40.1, 13.9 ppm. Spectra were consistent with literature data [9].

***S*-Benzyl *O*-ethyl carbonodithioate 4ab**



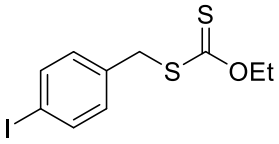

Colorless oil (93.3 mg, 88%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.36 (m, 3 H), 7.35 – 7.26 (m, 2 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 4.41 (s, 2 H), 1.45 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.1, 135.8, 129.2, 128.8, 127.7, 70.2, 40.6, 13.9 ppm. Spectra were consistent with literature data.[10]

***S*-(4-Chlorobenzyl) *O*-ethyl carbonodithioate 4ac**



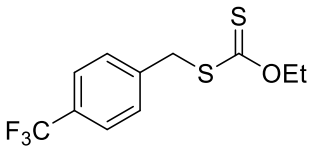

Yellow oil (103.3 mg, 84%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.27 (s, 4 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 4.32 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.6, 134.6, 133.5, 130.6, 128.9, 70.4, 39.7, 14.0 ppm. Spectra were consistent with literature data.[11]

***O*-Ethyl *S*-(4-iodobenzyl) carbonodithioate 4ad**



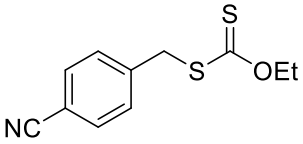
Colorless oil (155.5 mg, 92%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.4 Hz, 2 H), 7.10 (d, *J* = 8.4 Hz, 2 H), 4.65 (q, *J* = 7.1 Hz, 2 H), 4.30 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.6, 137.8, 135.8, 131.1, 93.1, 70.4, 39.9, 13.9 ppm. HRMS (ESI): m/z for $C_{10}H_{11}IOS_2$ $[M+Na]^+$ calcd 360.9188, found 360.9179.

***O*-Ethyl *S*-(4-(trifluoromethyl)benzyl) carbonodithioate 4ae**



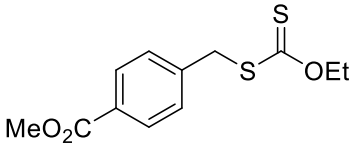
Colorless oil (93.8 mg, 67%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.1 Hz, 2 H), 7.47 (d, *J* = 8.1 Hz, 2 H), 4.65 (q, *J* = 7.1 Hz, 2 H), 4.40 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.4 140.4, 129.5, 125.6, 125.6, 124.2 (q, *J* = 271.9 Hz), 70.5, 39.8 13.8; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -62.4 (s, 3 F) ppm. HRMS (ESI): m/z for $C_{11}H_{11}F_3OS_2$ $[M+Na]^+$ calcd 303.0096, found 303.0094.

***S*-(4-Cyanobenzyl) *O*-ethyl carbonodithioate 4af**



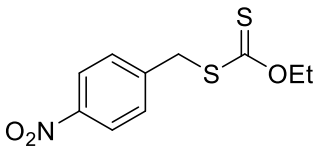
White solid (51.0 mg, 43%). Mp: 68-69 °C. Eluent: petroleum ether ($R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.3 Hz, 2 H), 7.45 (d, *J* = 8.6 Hz, 2 H), 4.61 (q, *J* = 7.1 Hz, 2 H), 4.37 (s, 2 H), 1.38 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.0, 142.0, 132.4, 129.9, 118.7, 111.4, 70.7, 39.8, 13.9 ppm. HRMS (ESI): m/z for $C_{11}H_{11}NOS_2$ $[M+Na]^+$ calcd 260.0174 found 260.0176.

**Methyl 4-(((ethoxycarbonothioyl)thio)methyl)benzoate 4ag**



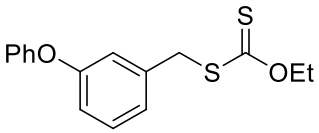
White solid (112.1 mg, 83%). Mp: 60-61 °C. Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 2 H), 7.41 (d, *J* = 8.3 Hz, 2 H), 4.63 (q, *J* = 7.1 Hz, 2 H), 4.38 (s, 2 H), 3.90 (s, 3 H), 1.40 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.4, 166.8, 141.4, 130.0, 129.4, 129.2, 70.4, 52.3, 40.0, 13.9 ppm. HRMS (ESI): m/z for $C_{12}H_{14}O_3S_2$ $[M+Na]^+$ calcd 293.0277 found 293.0269.

**_O_-Ethyl _S_-(4-nitrobenzyl) carbonodithioate 4ah**



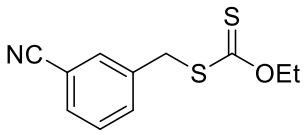

White solid (74.5 mg, 58%). Mp: 63-64 °C. Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.8 Hz, 2 H), 7.53 (d, *J* = 8.9 Hz, 2 H), 4.65 (q, *J* = 7.1 Hz, 2 H), 4.44 (s, 2 H), 1.42 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 212.9, 147.3, 144.1, 130.0, 123.9, 70.8, 39.5, 13.9 ppm. Spectra were consistent with literature data. [11]

**_O_-Ethyl _S_-(3-phenoxybenzyl) carbonodithioate 4ai**




Colorless oil (107.9 mg, 71%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.7). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 8.6, 7.4 Hz, 2 H), 7.26 (t, *J* = 7.9 Hz, 1 H), 7.14 – 7.09 (m, 1 H), 7.07 (m, 1 H), 7.03 – 6.95 (m, 3 H), 6.90 (m, *J* = 8.2, 2.5, 1.0 Hz, 1 H), 4.63 (q, *J* = 7.1 Hz, 2 H), 4.31 (s, 2 H), 1.40 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.8, 157.6, 156.9, 137.8, 130.0, 129.9, 123.9, 123.6, 119.4, 119.2, 117.9, 70.3, 40.2, 13.9 ppm. HRMS (ESI): m/z for $C_{16}H_{16}O_2S_2$ $[M+Na]^+$ calcd 327.0484, found 327.0481.

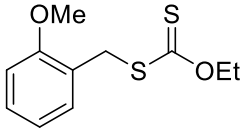
**_S_-(3-Cyanobenzyl) _O_-ethyl carbonodithioate 4aj**




White solid (53.3 mg, 45%). Mp: 63-64 °C. Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.48 (m, 3 H), 7.42 (dt, *J* = 7.8, 3.8 Hz,

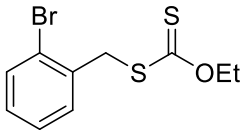
1 H), 4.63 (dd, *J* = 6.9, 3.4 Hz, 2 H), 4.36 (s, 2 H), 1.40 (t, *J* = 8.6 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.0, 138.1, 133.6, 132.6, 131.3, 129.5, 118.7, 112.7, 70.7, 39.3, 13.9 ppm. HRMS (ESI): m/z for $C_{11}H_{11}NOS_2$ $[M+Na]^+$ calcd 260.0174, found 260.0176.

**_O_-Ethyl _S_-(2-methoxybenzyl) carbonodithioate 4ak**



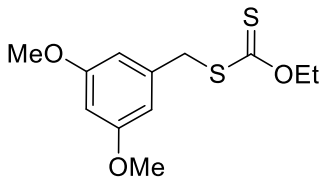

Colorless oil (62.9 mg, 52%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.7). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 7.4, 1.7 Hz, 1 H), 7.26 (dd, *J* = 15.7, 1.8 Hz, 1 H), 6.94 – 6.83 (m, 2 H), 4.66 (q, *J* = 7.1 Hz, 2 H), 4.38 (s, 2 H), 3.85 (s, 3 H), 1.43 (t, *J* = 7.1 Hz, 3H); $^{13}$C NMR (101 MHz,$CDCl_3$) δ 215.1, 157.6, 130.8, 129.2, 124.2, 120.5, 110.6, 70.0, 55.6, 35.4, 13.9 ppm.HRMS (ESI): m/z for $C_{11}H_{14}O_2S_2$ $[M+Na]^+$ calcd 265.0327, found 265.0319.

**_S_-(2-Bromobenzyl) _O_-ethyl carbonodithioate 4al**




Colorless oil (71.1 mg, 49%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.0 Hz, 1 H), 7.48 (d, *J* = 7.7 Hz, 1 H), 7.29 – 7.21 (m, 1 H), 7.13 (t, *J* = 7.7 Hz, 1 H), 4.66 (q, *J* = 7.0 Hz, 2 H), 4.49 (s, 2 H), 1.42 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.8, 135.6, 133.1, 131.3, 129.4, 127.7, 124.9, 70.3, 40.8, 13.9 ppm. Spectra were consistent with literature data.[12]

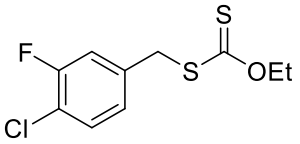
**_S_-(3,5-Dimethoxybenzyl) _O_-ethyl carbonodithioate 4am**




Colorless oil (125.2 mg, 92%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.7). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (d, *J* = 2.3 Hz, 2 H), 6.37 (t, *J* = 2.3 Hz, 1 H), 4.66 (q, *J* = 7.1 Hz, 2 H), 4.31 (s, 2 H), 3.77 (s, 7 H), 1.43 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR

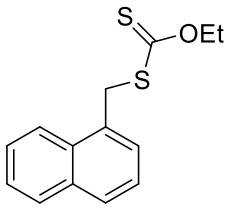
(101 MHz, $CDCl_3$) δ 214.1, 161.0, 137.9, 107.1, 99.7, 70.2, 55.4, 40.7, 13.9 ppm. HRMS (ESI): m/z for $C_{12}H_{16}O_3S_2$ $[M+Na]^+$ calcd 295.0433, found 295.0428.

**_S_-(4-Chloro-3-fluorobenzyl) _O_-ethyl carbonodithioate 4an**



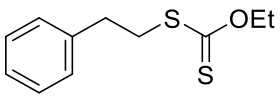

Colorless oil (67.3 mg, 51%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.32 (t, *J* = 7.9 Hz, 1 H), 7.16 (dd, *J* = 9.8, 2.1 Hz, 1 H), 7.08 (m, 1 H), 4.65 (q, *J* = 7.1 Hz, 2 H), 4.32 (s, 2 H), 1.42 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, Chloroform-*d*) δ 213.2, 158.0 (d, *J* = 249.2 Hz), 137.2 (d, *J* = 6.7 Hz), 130.7, 125.6 (d, *J* = 3.6 Hz), 120.1 (d, *J* = 17.6 Hz), 117.3 (d, *J* = 21.7 Hz), 70.6, 39.3, 13.9; $^{19}$F NMR (376 MHz, $CDCl_3$) δ -114.7 (m, 1 F) ppm. HRMS (ESI): m/z for $C_{10}H_{10}ClFOS_2$ $[M+Na]^+$ calcd 286.9738, found 286.9735.

**_O_-Ethyl _S_-(naphthalen-1-ylmethyl) carbonodithioate 4ao**




White solid (79.9 mg, 61%). Mp: 79-80 °C. Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.02 (m, 1 H), 7.92 – 7.87 (m, 1 H), 7.83 (dd, *J* = 8.2, 1.1 Hz, 1 H), 7.61 – 7.51 (m, 3 H), 7.43 (dd, *J* = 8.3, 7.0 Hz, 1 H), 4.87 (s, 2 H), 4.72 (q, *J* = 7.1 Hz, 2 H), 1.46 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.3, 134.0, 131.7, 130.9, 129.0, 128.9, 128.2, 126.7, 126.2, 125.5, 123.8, 70.2, 38.8, 14.0 ppm. HRMS (ESI): m/z for $C_{14}H_{14}OS_2$ $[M+Na]^+$ calcd 285.0378, found 285.0374.

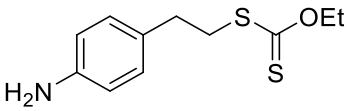
**_O_-Ethyl _S_-phenethyl carbonodithioate 4ap**




Colorless oil (102.9 mg, 91%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.32 (m, 2 H), 7.31 – 7.24 (m, 3 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 3.43 – 3.33 (m, 2 H), 3.07 – 2.97 (m, 2 H), 1.46 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz,

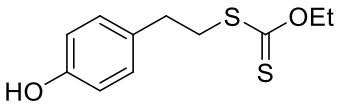
$CDCl_3$) δ 214.8, 140.0, 128.8, 128.7, 126.8, 70.1, 37.3, 35.0, 14.0 ppm. Spectra were consistent with literature data.[13]

**_S_-(4-Aminophenethyl) _O_-ethyl carbonodithioate 4aq**



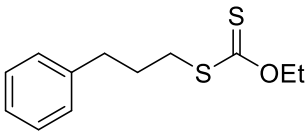

Yellow oil (66.3 mg, 55%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, *J* = 8.3 Hz, 2 H), 6.65 (d, *J* = 8.4 Hz, 2 H), 4.67 (q, *J* = 7.1 Hz, 2 H), 3.62 (s, 2 H), 3.35 – 3.25 (m, 2 H), 2.94 – 2.84 (m, 2 H), 1.44 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.0, 145.2, 129.8, 129.6, 115.4, 70.1, 37.6, 34.2, 14.0 ppm. HRMS (ESI): m/z for $C_{11}H_{15}NOS_2$ $[M+Na]^+$ calcd 264.0487, found 264.0481.

**_O_-Ethyl _S_-(4-hydroxyphenethyl) carbonodithioate 4ar**



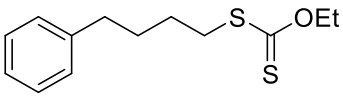

Colorless oil (120.0 mg, 99%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.6 Hz, 2 H), 6.83 (d, *J* = 8.5 Hz, 2 H), 6.23 (s, 1 H), 4.67 (q, *J* = 7.2 Hz, 2 H), 3.41 – 3.24 (m, 2 H), 2.99 – 2.86 (m, 2 H), 1.44 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.1, 154.2, 132.2, 130.0, 115.7, 70.2, 37.4, 34.1, 14.0 ppm. HRMS (ESI): m/z for $C_{11}H_{14}O_2S_2$ $[M+Na]^+$ calcd 265.0327, found 265.0319.

**_O_-Ethyl _S_-(3-phenylpropyl) carbonodithioate 4as**



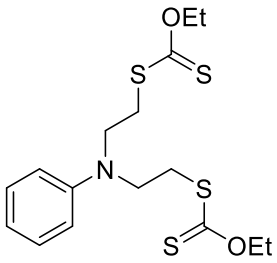

Yellow oil (100.8 mg, 84%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.3 Hz, 2 H), 7.24 (t, *J* = 7.8 Hz, 3 H), 4.68 (q, *J* = 7.1 Hz, 2 H), 3.22 – 3.12 (m, 2 H), 2.83 – 2.74 (m, 2 H), 2.08 (m, 2 H), 1.45 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.9, 141.2, 128.7, 128.6, 126.3, 70.0, 35.3, 35.1, 30.3, 14.0 ppm. HRMS (ESI): m/z for $C_{12}H_{16}OS_2$ $[M+Na]^+$ calcd 263.0535, found 263.0525.

**_O_-Ethyl _S_-(4-phenylbutyl) carbonodithioate 4at**



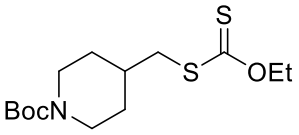

Colorless oil (104.2 mg, 82%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.6 Hz, 2 H), 7.25 (t, *J* = 7.2 Hz, 3 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 3.20 (t, *J* = 5.3 Hz, 2 H), 2.78 – 2.64 (m, 2 H), 1.80 (m, 4 H), 1.45 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.2, 142.1, 128.6, 128.5, 126.0, 70.0, 35.9, 35.6, 30.8, 28.2, 14.0 ppm. HRMS (ESI): m/z for $C_{13}H_{18}OS_2$ $[M+Na]^+$ calcd 277.0691, found 277.0689.

***O,O'*-Diethyl *S,S'*-((phenylazanediyl)bis(ethane-2,1-diyl)) bis(carbonodithioate) 4au**




Yellow oil (99.2 mg, 51%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 8.0 Hz, 2 H), 6.82 (d, *J* = 7.9 Hz, 2 H), 6.74 (t, *J* = 7.2 Hz, 1 H), 4.67 (q, *J* = 7.1 Hz, 4 H), 3.71 – 3.62 (m, 4 H), 3.35 – 3.24 (m, 4 H), 1.44 (t, *J* = 7.2 Hz, 6 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.5, 146.6, 129.7, 117.2, 112.2, 70.5, 49.9, 32.6, 14.0 ppm. HRMS (ESI): m/z for $C_{16}H_{23}NO_2S_4$ $[M+Na]^+$ calcd 412.0504, found 412.0500.

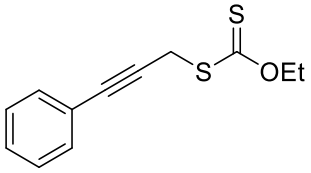
***Tert*-butyl 4-(((ethoxycarbonothioyl)thio)methyl)piperidine-1-carboxylate 4av**




Colorless oil (110.1 mg, 69%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.63 (q, *J* = 7.1 Hz, 2 H), 4.12 (d, *J* = 22.0 Hz, 2 H), 3.06 (d, *J* = 6.4 Hz, 2 H), 2.77 – 2.55 (m, 3 H), 1.87 – 1.72 (m, 3 H), 1.43 (s, 9 H), 1.40 (d, *J* = 7.1 Hz, 3 H), 1.17 (dd, *J* = 12.5, 4.3 Hz, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.0, 154.8, 79.5, 70.2, 42.0, 35.9, 31.6, 28.5, 13.9 ppm. HRMS (ESI): m/z for

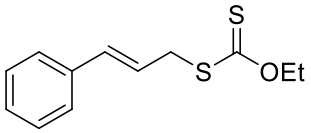
$C_{14}H_{25}NO_3S_2$ $[M+Na]^+$ calcd 342.1168, found 342.1165.

**_O_-Ethyl _S_-(3-phenylprop-2-yn-1-yl) carbonodithioate 4aw**



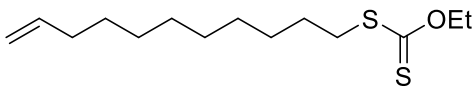

Colorless oil (69.6 mg, 59%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.40 (m, 2 H), 7.31 (dd, *J* = 5.2, 2.0 Hz, 3 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 4.12 (s, 2 H), 1.45 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 212.6, 131.9, 128.6, 128.4, 122.8, 83.7, 83.1, 70.5, 25.7, 13.9 ppm. HRMS (ESI): m/z for $C_{12}H_{12}OS_2$ $[M+Na]^+$ calcd 259.0222, found 259.0217.

**_S_-Cinnamyl _O_-ethyl carbonodithioate 4ax**



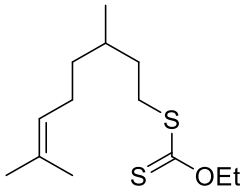

Colorless oil (59.5 mg, 50 %). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.2 Hz, 2 H), 7.32 (t, *J* = 7.4 Hz, 2 H), 7.28 – 7.23 (m, 1 H), 6.63 (d, *J* = 15.7 Hz, 1 H), 6.31 – 6.20 (m, 1 H), 4.67 (q, *J* = 7.1 Hz, 2 H), 3.97 (dd, *J* = 7.3, 1.3 Hz, 2 H), 1.44 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.1, 136.6, 134.1, 128.7, 128.0, 126.6, 123.2, 70.2, 38.7, 14.0 ppm. HRMS (ESI): m/z for $C_{12}H_{14}OS_2$ $[M+Na]^+$ calcd 261.0378, found 261.0373.

**_O_-Ethyl _S_-(undec-10-en-1-yl) carbonodithioate 4ay**



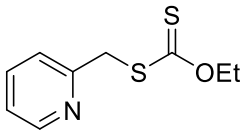

Colorless oil (98.7 mg, 72%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.80 (m, 1 H), 5.03 – 4.87 (m, 2 H), 4.63 (q, *J* = 7.1 Hz, 2 H), 3.14 – 3.06 (m, 2 H), 2.12 – 1.98 (m, 2 H), 1.67 (p, *J* = 7.3 Hz, 2 H), 1.46 – 1.34 (m, 7 H), 1.28 (d, *J* = 6.5 Hz, 8 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.3, 139.3, 114.2, 69.8, 36.0, 33.9, 29.5, 29.2, 29.0, 29.0, 28.5, 13.9 ppm. Spectra were consistent with literature data.[14]

**_S_-(2,6-Dimethylhept-5-en-1-yl) _O_-ethyl carbonodithioate 4az**



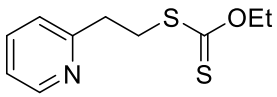
Colorless oil (115.8 mg, 89%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.09 (m, 1H), 4.64 (qd, *J* = 7.2, 1.0 Hz, 2H), 3.22 – 3.01 (m, 2H), 2.09 – 1.89 (m, 2H), 1.72 – 1.65 (m, 4H), 1.60 (s, 3H), 1.55 – 1.46 (m, 2H), 1.42 (td, *J* = 7.1, 1.0 Hz, 3H), 1.37 – 1.32 (m, 1H), 1.25 (s, 1H), 0.98 – 0.90 (m, 3H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.3, 131.5, 124.6, 69.9, 36.8, 35.4, 33.9, 32.2, 25.8, 25.5, 19.3, 17.8, 13.9 ppm. HRMS (ESI): m/z for $C_{13}H_{24}OS_2$ $[M+Na]^+$ calcd 283.1161, found 283.1160.

**_O_-Ethyl _S_-(pyridin-2-ylmethyl) carbonodithioate 4ba**



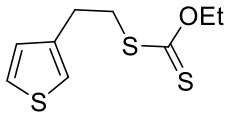
Yellow oil (59.7 mg, 56%). Eluent: ethyl acetate/petroleum ether (1:3, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.61 (s, 1 H), 8.54 – 8.47 (m, 1 H), 7.72 – 7.64 (m, 1 H), 7.29 – 7.19 (m, 1 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 4.35 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.2, 150.3, 148.9, 136.6, 134.4, 123.7, 70.5, 37.4, 13.9 ppm. HRMS (ESI): m/z for $C_9H_{11}NOS_2$ $[M+Na]^+$ calcd 236.0174, found 236.0165.

**_O_-Ethyl _S_-(2-(pyridin-2-yl)ethyl) carbonodithioate 4bb**



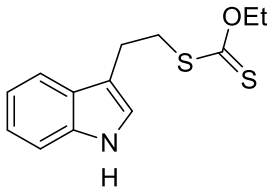
Yellow oil (64.7 mg, 57%). Eluent: ethyl acetate/petroleum ether (1:3, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 4.1 Hz, 1 H), 7.62 (td, *J* = 7.7, 1.8 Hz, 1 H), 7.23 – 7.11 (m, 2 H), 4.64 (q, *J* = 7.2 Hz, 2 H), 3.53 (dd, *J* = 8.2, 6.6 Hz, 2 H), 3.18 (t, *J* = 7.4 Hz, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.9, 159.3, 149.5, 136.7, 123.5, 121.9, 70.1, 36.8, 35.2, 13.9 ppm. HRMS (ESI): m/z for $C_{10}H_{13}NOS_2$ $[M+Na]^+$ calcd 250.0331, found 250.0326.

**_O_-Ethyl _S_-(2-(thiophen-3-yl)ethyl) carbonodithioate 4bc**



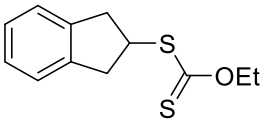

Colorless oil (51.0 mg, 44%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 4.9, 2.9 Hz, 1 H), 7.08 – 7.04 (m, 1 H), 7.02 (dd, *J* = 4.9, 1.4 Hz, 1 H), 4.66 (q, *J* = 7.1 Hz, 2 H), 3.42 – 3.34 (m, 2 H), 3.10 – 3.00 (m, 2 H), 1.44 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.8, 140.2, 128.1, 125.9, 121.5, 70.1, 36.5, 29.4, 14.0 ppm. HRMS (ESI): m/z for $C_9H_{12}OS_3$ $[M+Na]^+$ calcd 254.9942, found 254.9934.

**_S_-(2-(1_H_-Indol-3-yl)ethyl) _O_-ethyl carbonodithioate 4bd**



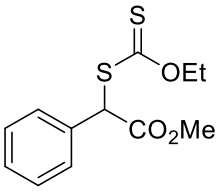

Colorless oil (87.5 mg, 66%). Eluent: ethyl acetate/petroleum ether (1:3, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.04 – 7.92 (m, 1 H), 7.71 (dd, *J* = 7.8, 1.1 Hz, 1 H), 7.38 (dt, *J* = 8.0, 1.0 Hz, 1 H), 7.24 (m, *J* = 8.1, 7.1, 1.4 Hz, 1 H), 7.17 (m, *J* = 8.0, 7.0, 1.1 Hz, 1 H), 7.09 – 7.01 (m, 1 H), 4.67 (q, *J* = 7.1 Hz, 2 H), 3.54 – 3.40 (m, 2 H), 3.18 (t, *J* = 7.8 Hz, 2 H), 1.43 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.2, 136.3, 127.2, 122.3, 122.2, 119.6, 118.9, 114.4, 111.4, 70.1, 36.5, 24.8, 14.0 ppm. HRMS (ESI): m/z for $C_{13}H_{15}NOS_2$ $[M+Na]^+$ calcd 288.0487, found 288.0486.

**_S_-(2,3-Dihydro-1_H_-inden-2-yl) _O_-ethyl carbonodithioate 4be**



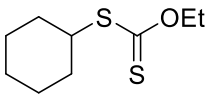

Yellow oil (116.6 mg, 98%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 4.2 Hz, 2 H), 7.22 (t, *J* = 4.2 Hz, 2 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 4.46 (m, 1 H), 3.54 (d, *J* = 16.2 Hz, 2 H), 3.07 (d, *J* = 11.0 Hz, 2 H), 1.46 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.7, 141.3, 127.0, 124.6, 69.9, 47.7, 39.4, 14.0 ppm. HRMS (ESI): m/z for $C_{12}H_{14}OS_2$ $[M+Na]^+$ calcd 261.0378, found 261.0374.

**Methyl 2-((ethoxycarbonothioyl)thio)-2-phenylacetate 4bf**



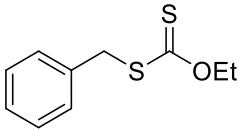
Yellow oil (62.1 mg, 46%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.39 (m, 2 H), 7.37 – 7.32 (m, 3 H), 5.47 (s, 1 H), 4.67 – 4.54 (m, 2 H), 3.75 (s, 3 H), 1.40 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 211.7, 170.0, 133.3, 129.2, 129.0, 128.7, 70.5, 57.0, 53.2, 13.7 ppm. Spectra were consistent with literature data.[15]

***S*-Cyclohexyl *O*-ethyl carbonodithioate 4bi**



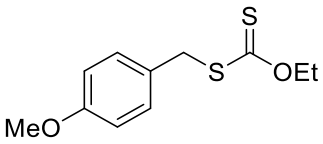
Colorless oil (46.9 mg, 46%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.63 (q, *J* = 7.1 Hz, 2 H), 3.73 – 3.59 (m, 1 H), 2.16 – 1.98 (m, 2 H), 1.74 (dt, *J* = 7.8, 3.6 Hz, 2 H), 1.68 – 1.53 (m, 2 H), 1.50 – 1.44 (m, 2 H), 1.41 (d, *J* = 7.1 Hz, 3 H), 1.29 – 1.22 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 212.5, 69.6, 48.8, 32.4, 26.0, 25.2, 13.9 ppm. Spectra were consistent with literature data.[16]

***S*-Benzyl *O*-ethyl carbonodithioate 4bj**



Colorless oil (46.6 mg, 44%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.36 (m, 3 H), 7.35 – 7.26 (m, 2 H), 4.69 (q, *J* = 7.1 Hz, 2 H), 4.41 (s, 2 H), 1.45 (t, *J* = 7.2 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.1, 135.8, 129.2, 128.8, 127.7, 70.2, 40.6, 13.9 ppm. Spectra were consistent with literature data.[11]

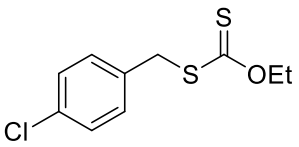
***O*-Ethyl *S*-(4-methoxybenzyl) carbonodithioate 4bk**



Colorless oil (63.0 mg, 52%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.8). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.23 (m, 2 H), 6.84 (d, *J* = 8.7 Hz, 2 H), 4.64 (q, *J* =

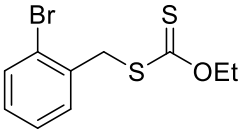
7.1 Hz, 2 H), 4.31 (s, 2 H), 3.78 (s, 3 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.4, 159.1, 130.4, 127.5, 114.1, 70.1, 55.4, 40.1, 13.9 ppm. Spectra were consistent with literature data. [9]

***S*-(4-Chlorobenzyl) *O*-ethyl carbonodithioate 4bl**



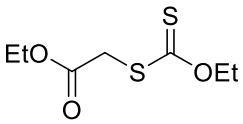

Yellow oil (50.5 mg, 41%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.27 (s, 4 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 4.32 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.6, 134.6, 133.5, 130.6, 128.9, 70.4, 39.7, 14.0 ppm. Spectra were consistent with literature data.[11]

***S*-(2-Bromobenzyl) *O*-ethyl carbonodithioate 4bm**



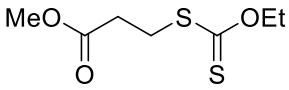

Colorless oil (72.5 mg, 50%). Eluent: petroleum ether ($R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.0 Hz, 1 H), 7.48 (d, *J* = 7.7 Hz, 1 H), 7.29 – 7.21 (m, 1 H), 7.13 (t, *J* = 7.7 Hz, 1 H), 4.66 (q, *J* = 7.0 Hz, 2 H), 4.49 (s, 2 H), 1.42 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.8, 135.6, 133.1, 131.3, 129.4, 127.7, 124.9, 70.3, 40.8, 13.9 ppm. Spectra were consistent with literature data.[12]

**Ethyl 2-((ethoxycarbonothioyl)thio)acetate 4bn**



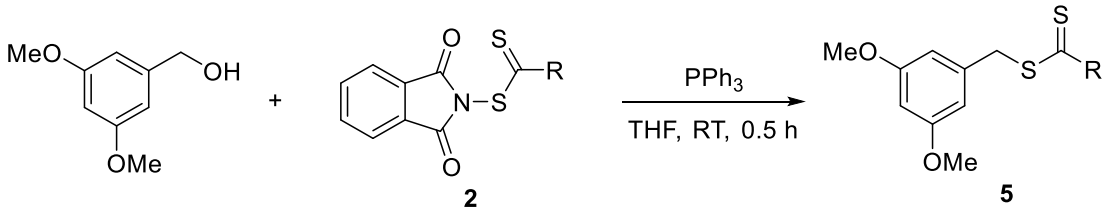

Yellow oil (64.5 mg, 62%). Eluent: petroleum ether ($R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.63 (q, *J* = 7.1 Hz, 2 H), 4.21 (q, *J* = 7.1 Hz, 2 H), 3.91 (s, 2 H), 1.41 (t, *J* = 7.1 Hz, 3 H), 1.28 (d, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 212.7, 168.0, 70.8, 62.1, 38.0, 14.3, 13.8 ppm. Spectra were consistent with literature data. [17]

**Methyl 3-((ethoxycarbonothioyl)thio)propanoate 4bo**



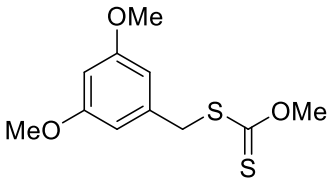
Yellow oil (70.7 mg, 68%). Eluent: petroleum ether ($R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.60 (q, *J* = 7.1 Hz, 2 H), 3.66 (s, 3 H), 3.33 (t, *J* = 7.1 Hz, 2 H), 2.73 (t, *J* = 7.1 Hz, 2 H), 1.37 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.3, 172.1, 70.2, 52.0, 33.3, 30.6, 13.9 ppm. Spectra were consistent with literature data.[18]

## 6. General procedure for direct dehydroxyxanthylation/xanthamidation/thioxanthylation of alcohols with *N*-xanthyl/xanthamide/thioxanthate phthalimides



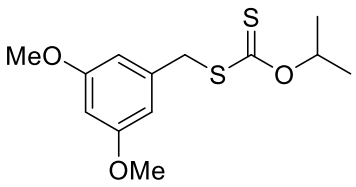

3,5-Dimethoxybenzyl alcohol (336.4 mg, 0.5 mmol, 1.0 equiv), *N*-xanthyl/xanthamide/thioxanthate phthalimides **2** (0.75 mmol, 1.5 equiv), $PPh_3$ (157.4 mg, 0.6 mmol, 1.2 equiv) were placed into an oven-dried 25 mL Schlenk tube that was equipped with a stirring bar. Then Freshly distilled THF (3.0 mL) was added to the Schlenk tube under nitrogen atmosphere. The reaction was stirred at room temperature for 0.5 h. The solvent was removed under vacuum and the residue was purified by flask column chromatography to obtain alkyl xanthate/xanthamide/thioxanthate products.

***S*-(3,5-Dimethoxybenzyl) *O*-methyl carbonodithioate 5aa**



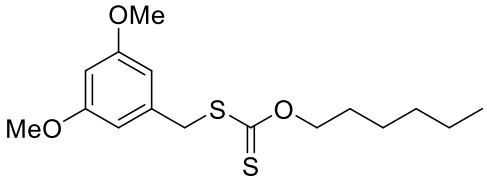

Colorless oil (63.2 mg, 49%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (d, *J* = 2.3 Hz, 2 H), 6.37 (t, *J* = 2.3 Hz, 1 H), 4.32 (s, 2 H), 4.19 (s, 3 H), 3.78 (s, 6 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.0, 161.0, 137.7, 107.1, 99.8, 60.3, 55.5, 41.1 ppm. HRMS (ESI): m/z for $C_{11}H_{14}O_3S_2$ $[M+Na]^+$ calcd 281.0277, found 281.0278.

***S*-(3,5-Dimethoxybenzyl) *O*-isopropyl carbonodithioate 5ab**



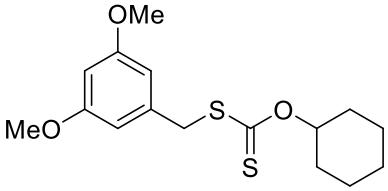
Colorless oil (74.4 mg, 52%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (s, 2 H), 6.37 (s, 1 H), 5.86 – 5.68 (m, 1 H), 4.28 (s, 2 H), 3.77 (s, 6 H), 1.39 (d, *J* = 6.2 Hz, 7 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.3, 161.0, 138.1, 107.1, 99.7, 78.2, 55.4, 40.5, 21.4 ppm. HRMS (ESI): m/z for $C_{12}H_{16}O_3S_2$ $[M+Na]^+$ calcd 309.0590, found 309.0585.

***S*-(3,5-Dimethoxybenzyl) *O*-hexyl carbonodithioate 5ac**



Colorless oil (82.1 mg, 50%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.49 (d, *J* = 2.3 Hz, 2 H), 6.36 (t, *J* = 2.3 Hz, 1 H), 4.58 (t, *J* = 6.7 Hz, 2 H), 4.30 (s, 2 H), 3.76 (s, 6 H), 1.78 (dq, *J* = 8.2, 6.7 Hz, 2 H), 1.38 (dd, *J* = 4.0, 2.2 Hz, 2 H), 1.34 – 1.28 (m, 4 H), 0.96 – 0.82 (m, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.2, 161.0, 137.9, 107.1, 99.7, 74.5, 55.4, 40.7, 31.5, 28.3, 25.7, 22.6, 14.1 ppm. HRMS (ESI): m/z for $C_{16}H_{24}O_3S_2$ $[M+Na]^+$ calcd 351.1059, found 351.1056.

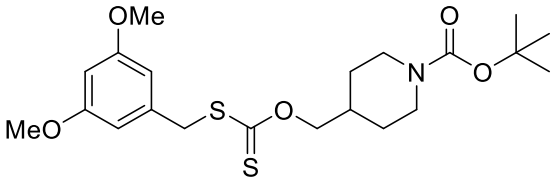
***O*-Cyclohexyl *S*-(3,5-dimethoxybenzyl) carbonodithioate 5ad**



Colorless oil (86.4 mg, 53%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (d, *J* = 2.3 Hz, 2 H), 6.36 (t, *J* = 2.3 Hz, 1 H), 5.57 (tt, *J* = 8.9, 3.9 Hz, 1 H), 4.29 (s, 2 H), 3.77 (s, 6 H), 2.03 – 1.91 (m, 2 H), 1.74 (td, *J* = 8.3, 3.3 Hz, 2 H), 1.63 – 1.54 (m, 2 H), 1.48 – 1.38 (m, 2 H), 1.40 – 1.18 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.1, 161.0, 138.0, 107.1, 99.7, 82.8, 55.4, 40.5, 31.0, 25.4, 23.7 ppm. HRMS (ESI): m/z for $C_{16}H_{22}O_3S_2$ $[M+Na]^+$ calcd 349.0903, found 349.0902.

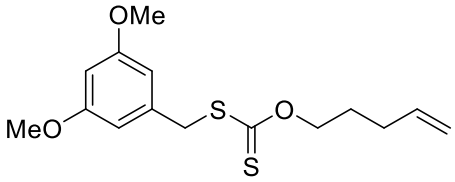
***Tert*-butyl 4-(((((3,5-dimethoxybenzyl)thio)carbonothioyl)oxy)methyl)piperidine-**

**1-carboxylate 5ae**



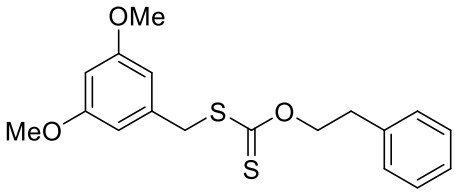

Colorless oil (145.6 mg, 66%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.1). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.48 (d, *J* = 2.3 Hz, 2 H), 6.35 (t, *J* = 2.3 Hz, 1 H), 4.44 (d, *J* = 6.5 Hz, 2 H), 4.29 (s, 2 H), 4.21 – 4.00 (m, 2 H), 3.76 (s, 7 H), 2.70 (s, 2 H), 2.07 – 1.90 (m, 1 H), 1.73 – 1.61 (m, 2 H), 1.45 (s, 9 H), 1.26 – 1.18 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.2, 161.0, 154.9, 137.7, 107.1, 99.7, 79.6, 77.8, 55.4, 40.8, 35.5, 28.7, 28.5 ppm. HRMS (ESI): m/z for $C_{21}H_{31}NO_5S_2$ $[M+Na]^+$ calcd 464.1536, found 464.1527.

***S*-(3,5-Dimethoxybenzyl) *O*-(pent-4-en-1-yl) carbonodithioate 5af**




Colorless oil (71.8 mg, 46%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.3). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (d, *J* = 2.3 Hz, 2 H), 6.37 (t, *J* = 2.3 Hz, 1 H), 5.89 – 5.73 (m, 1 H), 5.12 – 4.95 (m, 2 H), 4.61 (t, *J* = 6.6 Hz, 2 H), 4.31 (s, 2 H), 3.77 (d, *J* = 1.2 Hz, 6 H), 2.17 (qd, *J* = 7.3, 6.3, 1.5 Hz, 2 H), 1.96 – 1.84 (m, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.2, 161.0, 137.9, 137.2, 115.7, 107.1, 99.7, 73.6, 55.4, 40.8, 30.1, 27.5 ppm. HRMS (ESI): m/z for $C_{15}H_{20}O_3S_2$ $[M+Na]^+$ calcd 335.0746, found 335.0749.

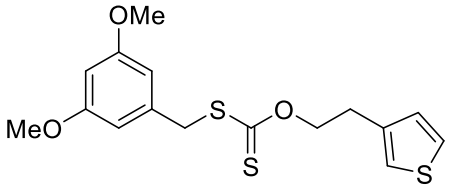
***S*-(3,5-Dimethoxybenzyl) *O*-phenethyl carbonodithioate 5ag**




Colorless oil (83.5 mg, 48%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2 H), 7.26 – 7.22 (m, 3 H), 6.46 (d, *J* = 2.3 Hz, 2 H), 6.36 (t, *J* = 2.3 Hz, 1 H), 4.80 (t, *J* = 7.1 Hz, 2 H), 4.27 (s, 2 H), 3.76 (s, 6 H),

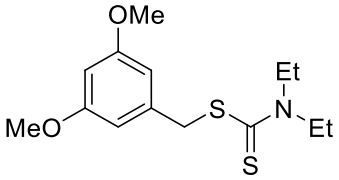
3.10 (t, *J* = 7.1 Hz, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 212.9, 161.0, 137.7, 137.2, 129.1, 128.7, 126.9, 107.1, 99.8, 74.1, 55.5, 40.7, 34.7 ppm. HRMS (ESI): m/z for $C_{18}H_{20}O_3S_2$ $[M+Na]^+$ calcd 371.0746 found 371.0741.

***S*-(3,5-Dimethoxybenzyl) *O*-(2-(thiophen-2-yl)ethyl) carbonodithioate 5ah**



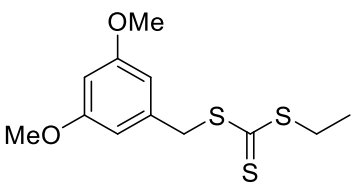

Colorless oil (102.7 mg, 58%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.4). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 4.9, 3.0 Hz, 1 H), 7.08 – 7.04 (m, 1 H), 7.00 (dd, *J* = 4.9, 1.3 Hz, 1 H), 6.50 (d, *J* = 2.3 Hz, 2 H), 6.39 (t, *J* = 2.3 Hz, 1 H), 4.81 (t, *J* = 6.9 Hz, 2 H), 4.30 (s, 2 H), 3.78 (s, 6 H), 3.15 (t, *J* = 6.5 Hz, 2 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.0, 161.0, 137.7, 137.4, 128.4, 126.0, 122.1, 107.2, 99.8, 73.6, 55.5, 40.8, 29.2 ppm. HRMS (ESI): m/z for $C_{16}H_{18}O_3S_3$ $[M+Na]^+$ calcd 377.0310, found 377.0305.

**3,5-Dimethoxybenzyl diethylcarbamodithioate 5ai**




Colorless oil (143.6 mg, 96%). Eluent: ethyl acetate/petroleum ether (1:5, $R_f$ = 0.5). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.54 (d, *J* = 2.3 Hz, 2 H), 6.35 (t, *J* = 2.3 Hz, 1 H), 4.47 (s, 2 H), 4.05 – 4.00 (m, 2 H), 3.76 (s, 6 H), 3.71 (t, *J* = 7.2 Hz, 2 H), 1.26 (d, *J* = 5.3 Hz, 6 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 195.2, 160.9, 138.3, 107.4, 99.7, 55.4, 49.6, 46.8, 42.5, 12.6, 11.7 ppm. HRMS (ESI): m/z for $C_{14}H_{21}NO_2S_2$ $[M+Na]^+$ calcd 322.0906, found 322.0898.

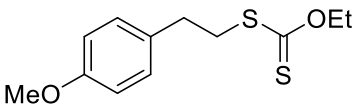
**3,5-Dimethoxybenzyl ethyl carbonotrithioate 5aj**




Yellow oil (64.8 mg, 45%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1$H

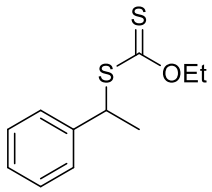
NMR (400 MHz, $CDCl_3$) δ 6.49 (d, *J* = 2.3 Hz, 2 H), 6.37 (t, *J* = 2.3 Hz, 1 H), 4.55 (s, 2 H), 3.77 (s, 6 H), 3.38 (q, *J* = 7.4 Hz, 2 H), 1.36 (t, *J* = 7.5 Hz, 3 H); $^{13}C$ NMR (101 MHz, $CDCl_3$) δ 223.6, 161.0, 137.3, 107.3, 99.9, 55.5, 41.6, 31.4, 13.2 ppm. HRMS (ESI): m/z for $C_{12}H_{16}O_2S_3$ $[M+Na]^+$ calcd 311.0205, found 311.0201.

***O*-Ethyl *S*-(4-methoxyphenethyl) carbonodithioate 8aa**



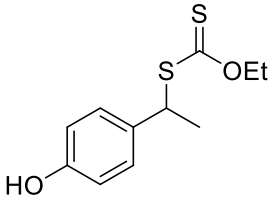

Colorless oil (70.5 mg, 55%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.5). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.6 Hz, 2 H), 6.85 (d, *J* = 8.6 Hz, 2 H), 4.65 (q, *J* = 7.1 Hz, 2 H), 3.80 (s, 3 H), 3.36 – 3.27 (m, 2 H), 2.97 – 2.87 (m, 2 H), 1.43 (t, *J* = 7.1 Hz, 3 H); $^{13}C$ NMR (101 MHz, $CDCl_3$) δ 214.9, 158.4, 132.0, 129.7, 114.1, 70.0, 55.4, 37.5, 34.1, 13.9 ppm. HRMS (ESI): m/z for $C_{12}H_{16}O_2S_2$ $[M+Na]^+$ calcd 279.0484, found 279.0480.

***O*-Ethyl *S*-(1-phenylethyl) carbonodithioate 8ab**




Colorless oil (52.0 mg, 46%). Eluent: petroleum ether ($R_f$ = 0.6). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.34 (m, 2 H), 7.32 (m, 2 H), 7.26 – 7.24 (m, 1 H), 4.88 (q, *J* = 7.2 Hz, 1 H), 4.60 (q, *J* = 7.1 Hz, 2 H), 1.70 (d, *J* = 7.2 Hz, 3 H), 1.37 (t, *J* = 7.1 Hz, 3 H); $^{13}C$ NMR (101 MHz, $CDCl_3$) δ 213.5, 141.9, 128.7, 127.6, 127.6, 69.8, 49.3, 21.8, 13.8 ppm. Spectra were consistent with literature data.[16]

***O*-Ethyl *S*-(1-(4-hydroxyphenyl)ethyl) carbonodithioate 8ac**

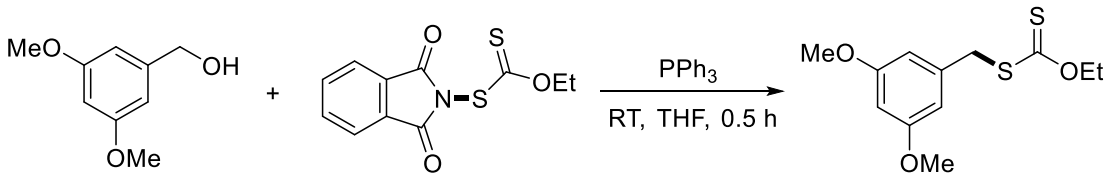



Yellow oil (81.1 mg, 67%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.3). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.5 Hz, 2 H), 6.78 (d, *J* = 8.6 Hz, 2 H), 5.20 (s, 1 H), 4.84 (q, *J* = 7.1 Hz, 1 H), 4.61 (q, *J* = 7.1 Hz, 2 H), 1.68 (d, *J* = 7.2 Hz, 3 H), 1.38

(t, *J* = 7.1 Hz, 3 H).; $^{13}$C NMR (101 MHz, $CDCl_3$) δ 213.8, 155.0, 133.9, 128.9, 115.5, 69.8, 48.8, 21.8, 13.9 ppm. HRMS (ESI): m/z for $C_{11}H_{14}O_2S_2$ [M+Na]+ calcd 265.0327, found 265.0319.

## IV. Synthetic applications

### Large-scale reactions

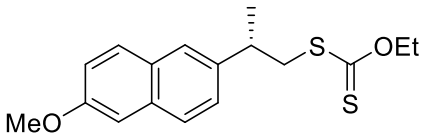



10 mmol, **1am** 15 mmol, **2d** 2.23 g, 82%, **4am**

3,5-Dimethoxybenzyl alcohol (**1am**, 1.68 g, 10 mmol, 1.0 equiv), *N*-ethylxanthyl phthalimide **2d** (4.0 g, 15 mmol, 1.5 equiv), $PPh_3$ (3.15 g, 12 mmol, 1.2 equiv) were placed into an oven-dried 100 mL Schlenk tube that was equipped with a stirring bar. Then Freshly distilled THF (50 mL) was added to the Schlenk tube under nitrogen atmosphere. The reaction was stirred at room temperature for 0.5 h. The solvent was removed under vacuum and the residue was purified by flask column chromatography to obtain alkyl xanthate product **4am** (2.23 g, 82%).

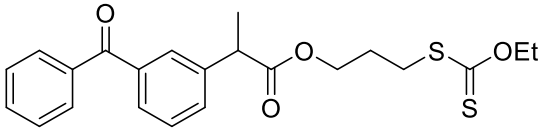
### Late-stage dehydroxyxanthylation of bioactive molecules or druglike scaffolds

#### *(S)*-*O*-Ethyl *S*-(2-(6-methoxynaphthalen-2-yl)propyl) carbonodithioate 10aa



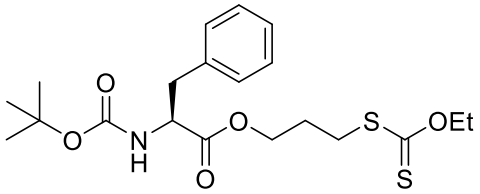

Colorless oil (91.2 mg, 57%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.7). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 8.7, 2.8 Hz, 2 H), 7.61 (d, *J* = 1.8 Hz, 1 H), 7.38 (dd, *J* = 8.5, 1.9 Hz, 1 H), 7.19 – 7.11 (m, 2 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 3.92 (s, 3 H), 3.52 (dd, *J* = 13.3, 6.9 Hz, 1 H), 3.38 (dd, *J* = 13.3, 7.6 Hz, 1 H), 3.28 (m 1 H), 1.48 (d, *J* = 6.9 Hz, 3 H), 1.41 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.1, 157.6, 140.1, 133.7, 129.3, 129.1, 127.2, 126.1, 125.3, 119.0, 105.7, 70.1, 55.4, 44.0, 38.9, 21.1, 13.9 ppm. HRMS (ESI): m/z for $C_{17}H_{20}O_2S_2$ $[M+Na]^+$ calcd 343.0797, found 343.0800.

**3-((Ethoxycarbonothioyl)thio)propyl 2-(3-benzoylphenyl)propanoate 10ab**



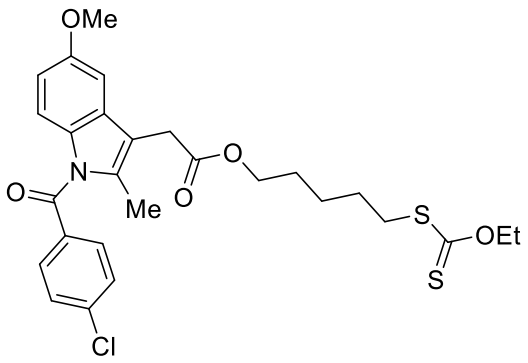
Colorless oil (122.8 mg, 59%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.74 (m, 3 H), 7.67 (dt, *J* = 7.6, 1.4 Hz, 1 H), 7.64 – 7.57 (m, 1 H), 7.55 (dt, *J* = 7.8, 1.6 Hz, 1 H), 7.51 – 7.41 (m, 3 H), 4.61 (q, *J* = 7.1 Hz, 2 H), 4.16 (t, *J* = 6.2 Hz, 2 H), 3.81 (q, *J* = 7.1 Hz, 1 H), 3.11 – 3.04 (m, 2 H), 2.05 – 1.95 (m, 2 H), 1.55 (d, *J* = 7.2 Hz, 3 H), 1.40 (t, *J* = 7.1 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.5, 196.6, 174.1, 140.9, 138.0, 137.6, 132.7, 131.6, 130.2, 129.3, 129.2, 128.7, 128.4, 70.2, 63.5, 45.5, 32.3, 27.8, 18.5, 13.9 ppm. HRMS (ESI): m/z for $C_{22}H_{24}O_4S_2$ $[M+Na]^+$ calcd 439.1008, found 439.0999.

**3-((Ethoxycarbonothioyl)thio)propyl (*tert*-butoxycarbonyl)-*L*-phenylalaninate 10ac**



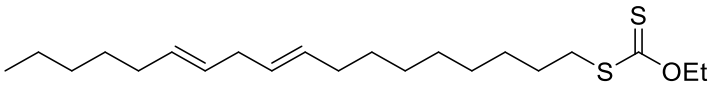
Colorless oil (136.7 mg, 64%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2 H), 7.24 – 7.18 (m, 1 H), 7.15 – 7.09 (m, 2 H), 5.02 (d, *J* = 8.4 Hz, 1 H), 4.61 (q, *J* = 7.1 Hz, 2 H), 4.13 (t, *J* = 6.1 Hz, 2 H), 3.05 (d, *J* = 6.4 Hz, 2 H), 2.99 (td, *J* = 7.1, 5.5 Hz, 2 H), 2.02 (s, 1 H), 1.94 (qd, *J* = 8.8, 8.4, 6.2 Hz, 2 H), 1.42 – 1.36 (m, 13 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.5, 172.0, 155.2, 136.1, 129.4, 128.7, 127.2, 80.1, 70.2, 63.8, 54.7, 38.6, 32.2, 28.4, 27.7, 13.9 ppm. HRMS (ESI): m/z for $C_{20}H_{29}NO_5S_2$ $[M+Na]^+$ calcd 450.1379, found 450.1361.

**5-((Ethoxycarbonothioyl)thio)pentyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate 10ad**



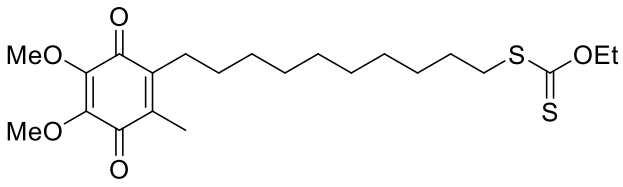

Yellow oil (227.1 mg, 83%). Eluent: ethyl acetate/petroleum ether (1:1, $R_f$ = 0.2). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.5 Hz, 2 H), 7.47 (d, *J* = 8.5 Hz, 2 H), 6.96 (d, *J* = 2.5 Hz, 1 H), 6.86 (d, *J* = 9.0 Hz, 1 H), 6.66 (dd, *J* = 9.0, 2.5 Hz, 1 H), 4.63 (q, *J* = 7.1 Hz, 2 H), 4.10 (t, *J* = 6.5 Hz, 2 H), 3.83 (s, 3 H), 3.66 (s, 2 H), 3.05 (t, *J* = 7.4 Hz, 2 H), 2.39 (s, 3 H), 1.73 – 1.56 (m, 5 H), 1.49 – 1.34 (m, 5 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.1, 171.0, 168.4, 156.1, 139.4, 136.0, 134.0, 131.3, 130.9, 130.7, 129.2, 115.1, 112.7, 111.7, 101.4, 70.0, 64.9, 55.8, 35.7, 30.5, 28.2, 28.1, 25.3, 13.9, 13.5 ppm. HRMS (ESI): m/z for $C_{27}H_{30}ClNO_5S_2$ $[M+Na]^+$ calcd 570.1146, found 570.1131.

**_O_-Ethyl _S_-((9Z,12Z)-octadeca-9,12-dien-1-yl) carbonodithioate 10ae**



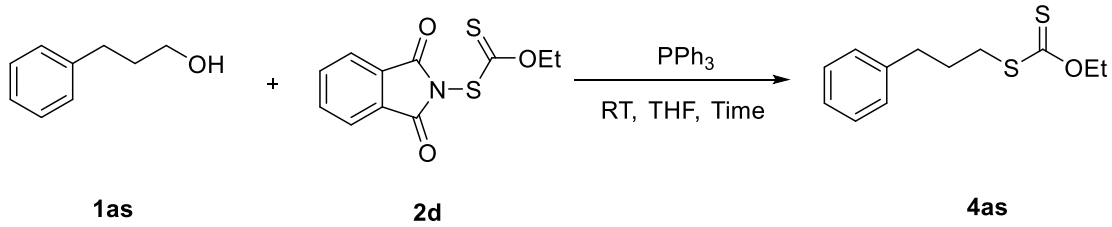

Yellow oil (181.4 mg, 98%). Eluent: ethyl acetate/petroleum ether (1:10, $R_f$ = 0.8). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.45 – 5.26 (m, 4 H), 4.64 (q, *J* = 7.1 Hz, 2 H), 3.18 – 3.05 (m, 2 H), 2.77 (t, *J* = 6.6 Hz, 2 H), 2.05 (q, *J* = 6.9 Hz, 4 H), 1.74 – 1.62 (m, 2 H), 1.42 (d, *J* = 7.1 Hz, 3 H), 1.37 – 1.23 (m, 16 H), 0.89 (t, *J* = 6.8 Hz, 3 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 215.3, 130.3, 130.2, 128.1, 128.0, 69.8, 36.0, 31.6, 29.7, 29.5, 29.3, 29.2, 29.0, 28.5, 27.3, 25.7, 22.7, 14.2, 13.9 ppm. HRMS (ESI): m/z for $C_{21}H_{38}OS_2$ $[M+Na]^+$ calcd 393.2256, found 393.2260.

**_S_-(10-(4,5-Dimethoxy-2-methyl-3,6-dioxocyclohexa-1,4-dien-1-yl)decyl) _O_-ethyl carbonodithioate 10af**

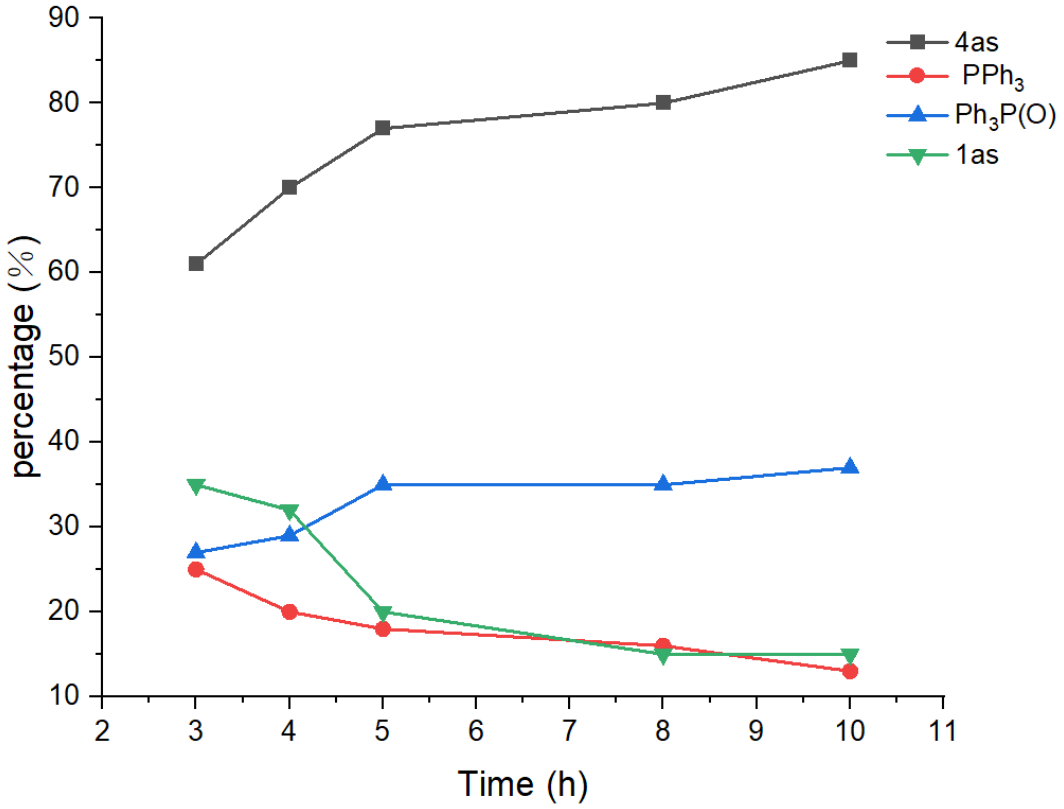


Yellow oil (126.1 mg, 57%). Eluent: ethyl acetate/petroleum ether (1:2, $R_f$ = 0.6). $^1$H NMR (400 MHz, $CDCl_3$) δ 3.94 (q, *J* = 7.1 Hz, 2 H), 3.28 (d, *J* = 1.4 Hz, 6 H), 2.40 (t, *J* = 7.4 Hz, 2 H), 1.74 (t, *J* = 7.4 Hz, 2 H), 1.31 (s, 3 H), 1.02 – 0.92 (m, 2 H), 0.72 (d, *J* = 7.1 Hz, 3 H), 0.58 (d, *J* = 8.5 Hz, 15 H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 214.7, 184.1, 183.6, 143.7, 142.5, 138.1, 69.2, 60.6, 35.3, 29.2, 28.8, 28.7, 28.5, 28.3, 28.1, 27.8, 25.8, 13.2, 11.3 ppm. HRMS (ESI): m/z for $C_{22}H_{34}O_5S_2$ $[M+Na]^+$ calcd 465.1740, found 465.1732.

## V. Investigation of the reaction process




**1as** **2d** **4as**




An oven-dried 25 mL Schlenk tube equipped with a stirring bar, was charged with **1as** (68.0 mg, 0.5 mmol, 1.0 equiv), N-ethylxanthyl phthalimide (**2d**, 200.3 mg, 0.75 mmol, 1.5 equiv), $PPh_3$ (157.4 mg, 0.6 mmol, 1.2 equiv) and THF (3.0 mL) in argon

atmosphere. The reaction mixture was stirred at room temperature. After simple filtration and removal of solvent, the crude products were subjected to $^{1}H$ NMR spectroscopy or GC analysis. The results show that the reaction has different proportions for the products of **4as**, **1as**, $PPh_3$ and $Ph_3P(O)$ at different time periods.

| Time (min) | Yield of **1as** | Yield of **4as** | Yield of $PPh_3$ | Yield of $Ph_3P(O)$ |
|---|---|---|---|---|
| 3 | 35 % | 61 % | 25 % | 27 % |
| 4 | 32 % | 70 % | 20 % | 29 % |
| 5 | 20 % | 77 % | 18 % | 35 % |
| 8 | 18 % | 80 % | 16 % | 35 % |
| 10 | 15 % | 82 % | 13 % | 37 % |

## VI. References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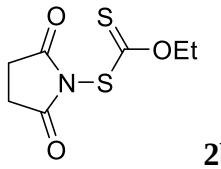
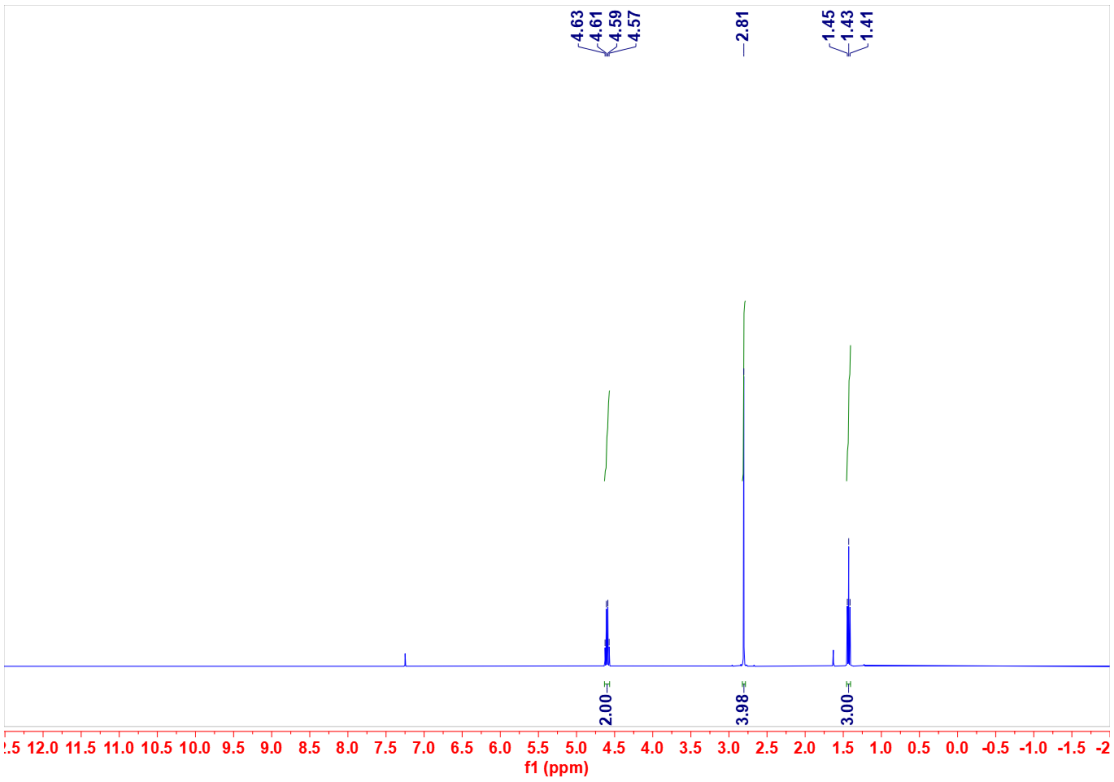
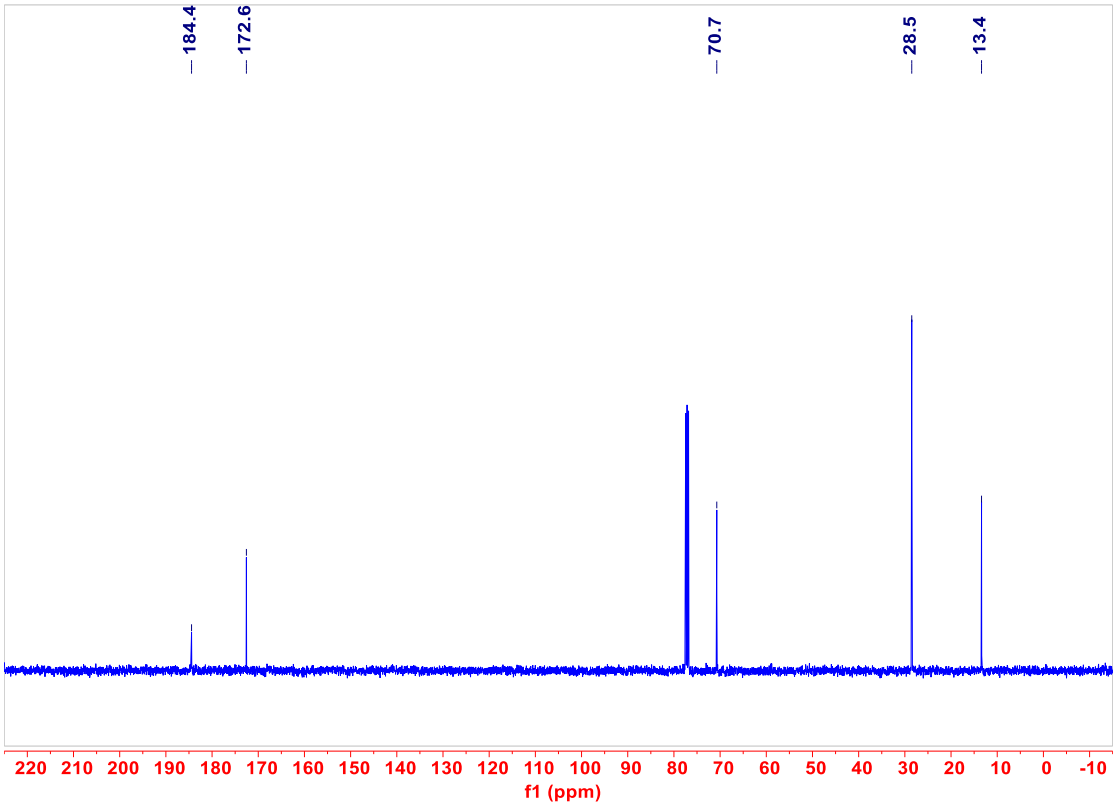
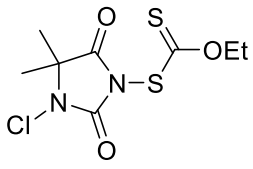
## VII. $^{1}H$, $^{19}F$, $^{13}C$ NMR spectra of corresponding compunds



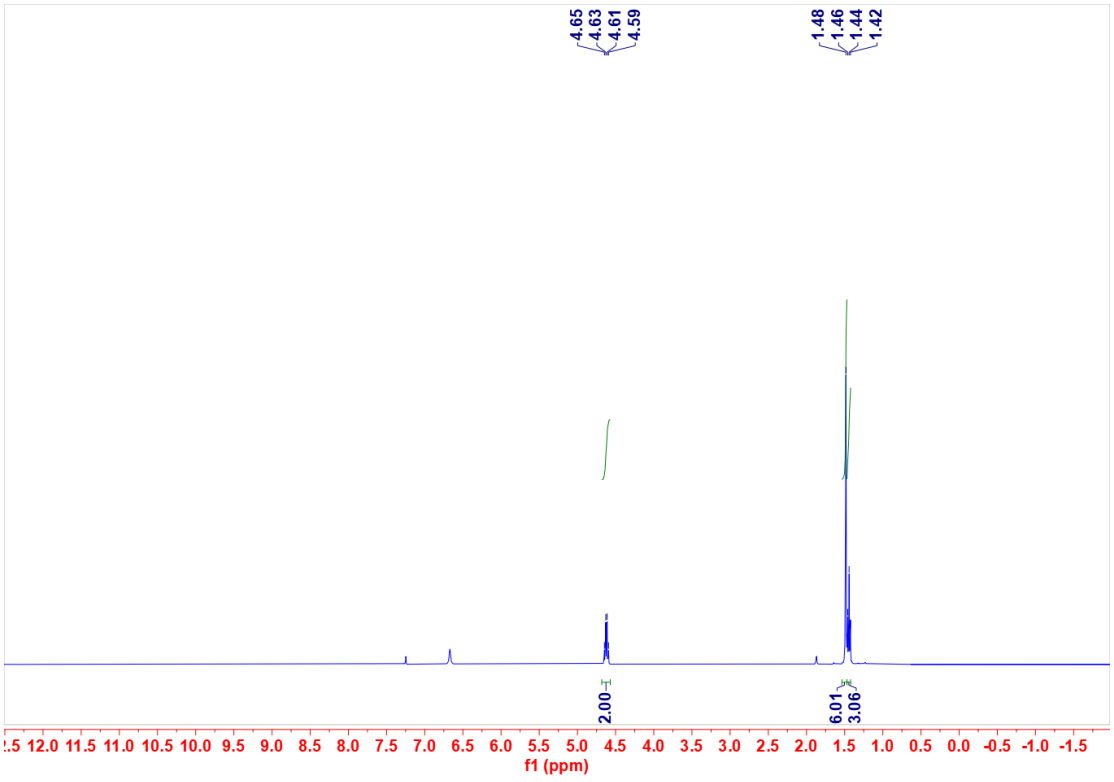

**2b**

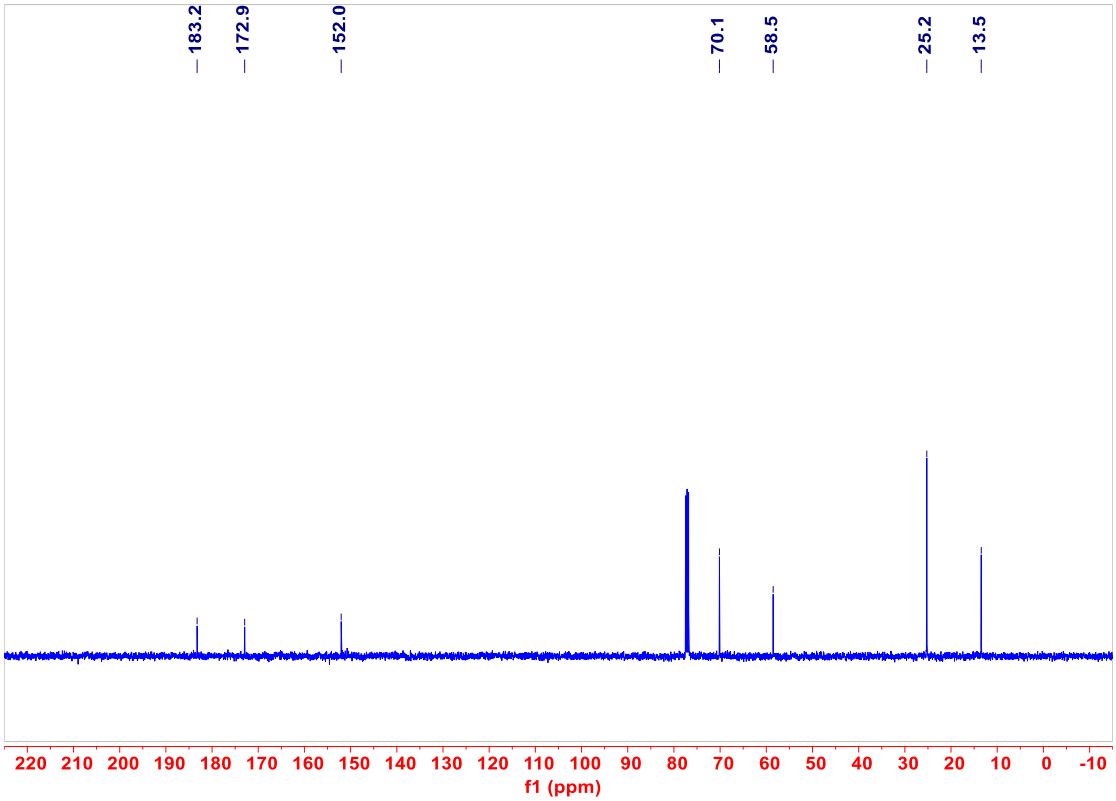


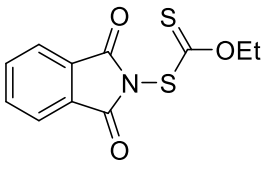




O
S
OEt
N
S
Cl
N
O

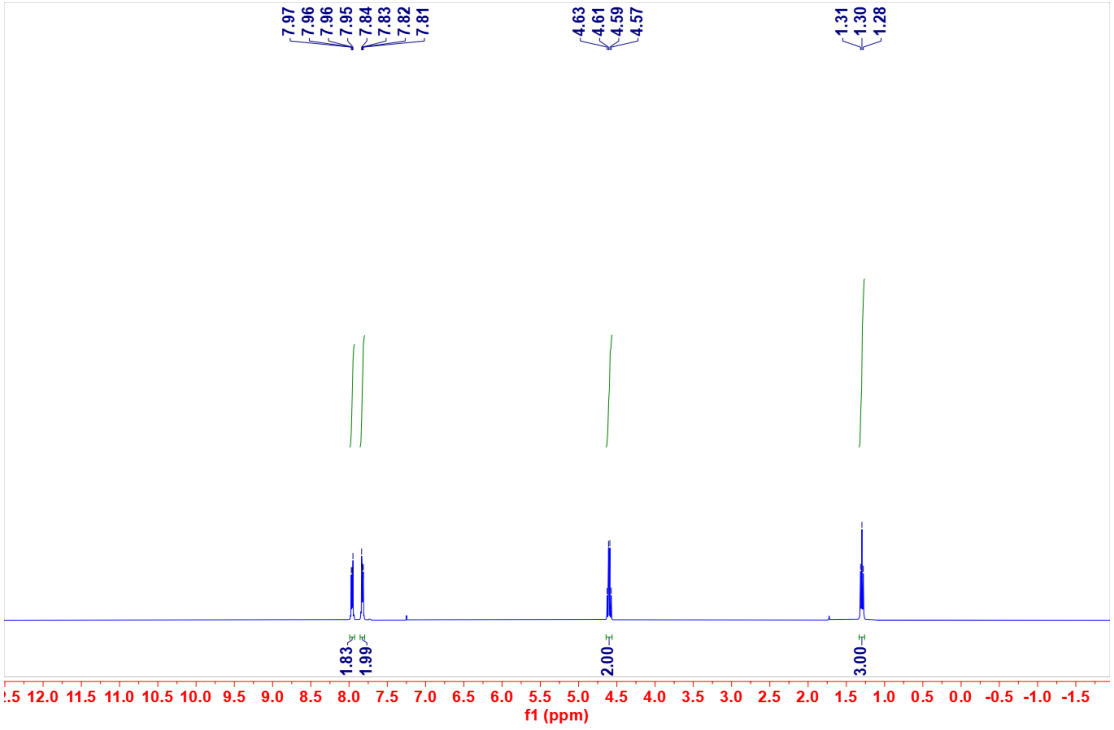
**2c**

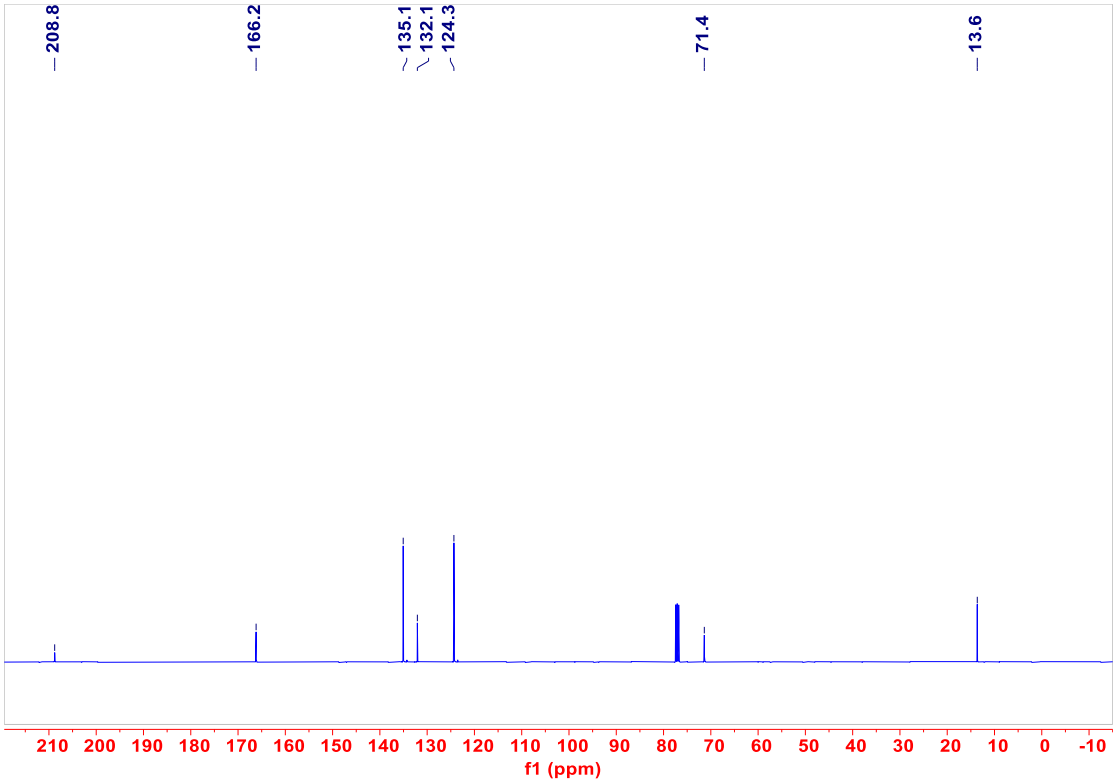

4.65
4.63
4.61
4.59
1.48
1.46
1.44
1.42
2.00
6.01
3.06
f1 (ppm)

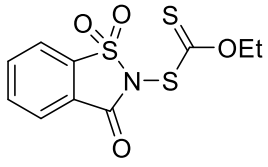

183.2
172.9
152.0
70.1
58.5
25.2
13.5
f1 (ppm)


O
S
OEt
N
S
O

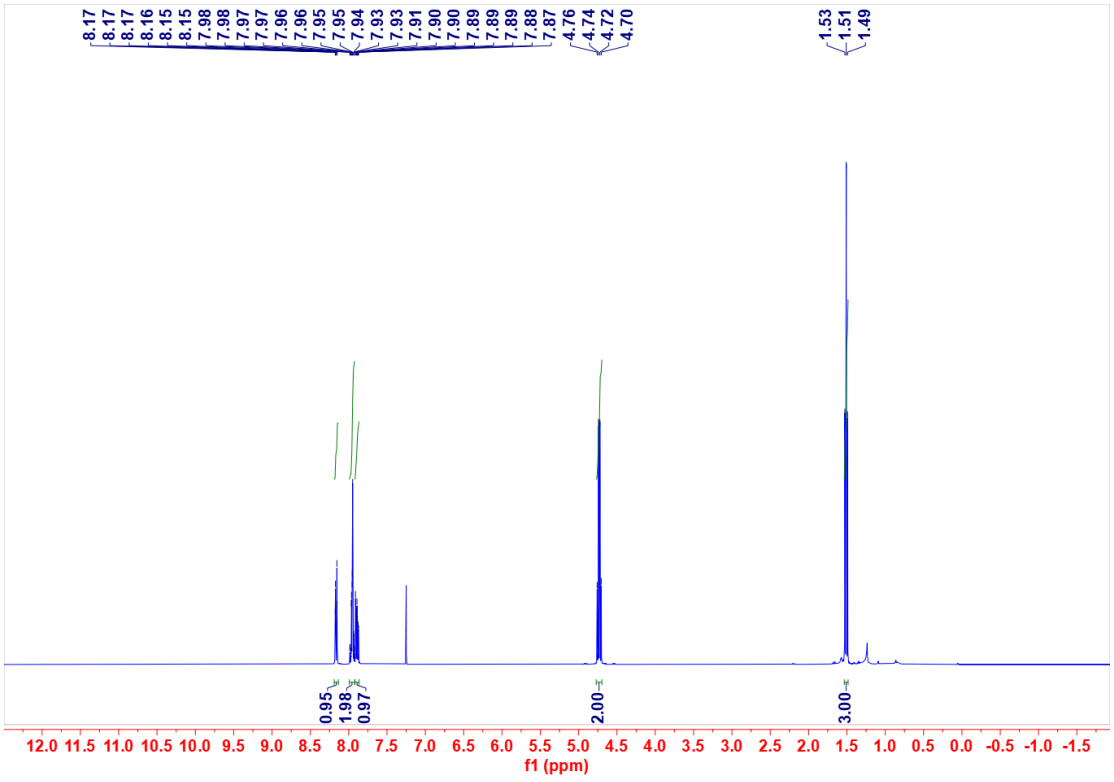
**2d**

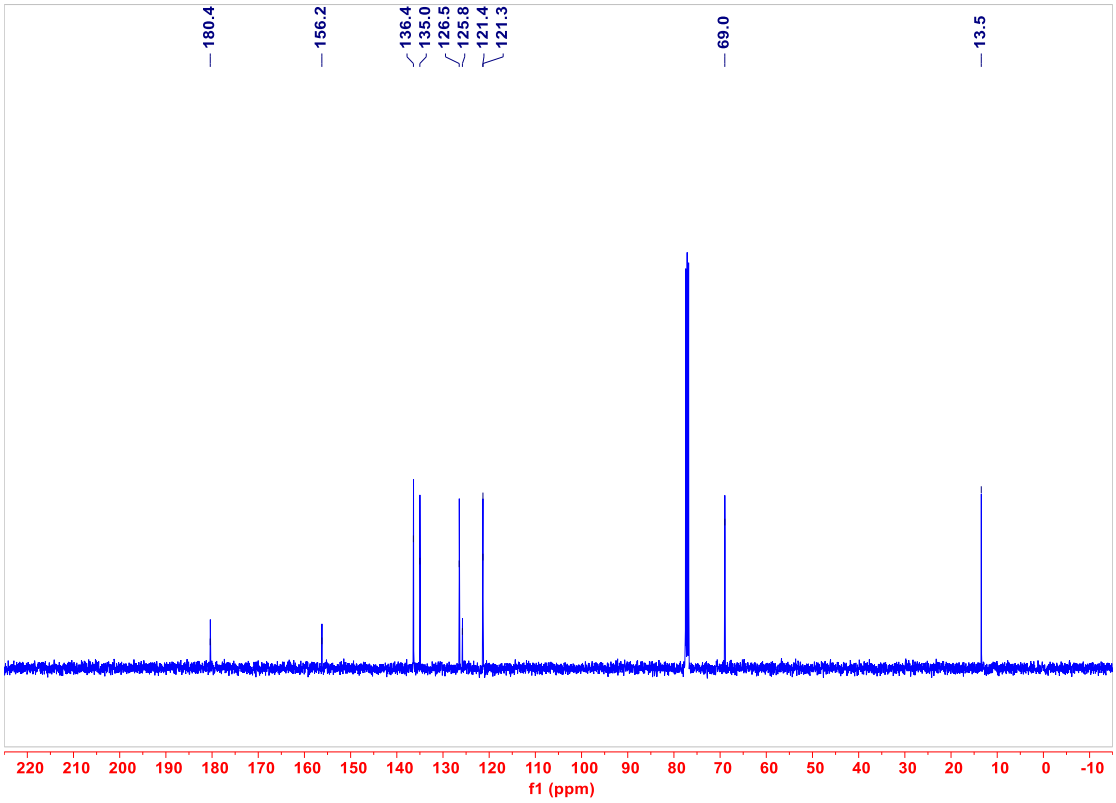

7.97
7.96
7.96
7.95
7.84
7.83
7.82
7.81
4.63
4.61
4.59
4.57
1.31
1.30
1.28
1.83
1.99
2.00
3.00
f1 (ppm)

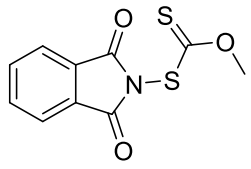

208.8
166.2
135.1
132.1
124.3
71.4
13.6
f1 (ppm)


O
O
S
S
OEt
N
S
O

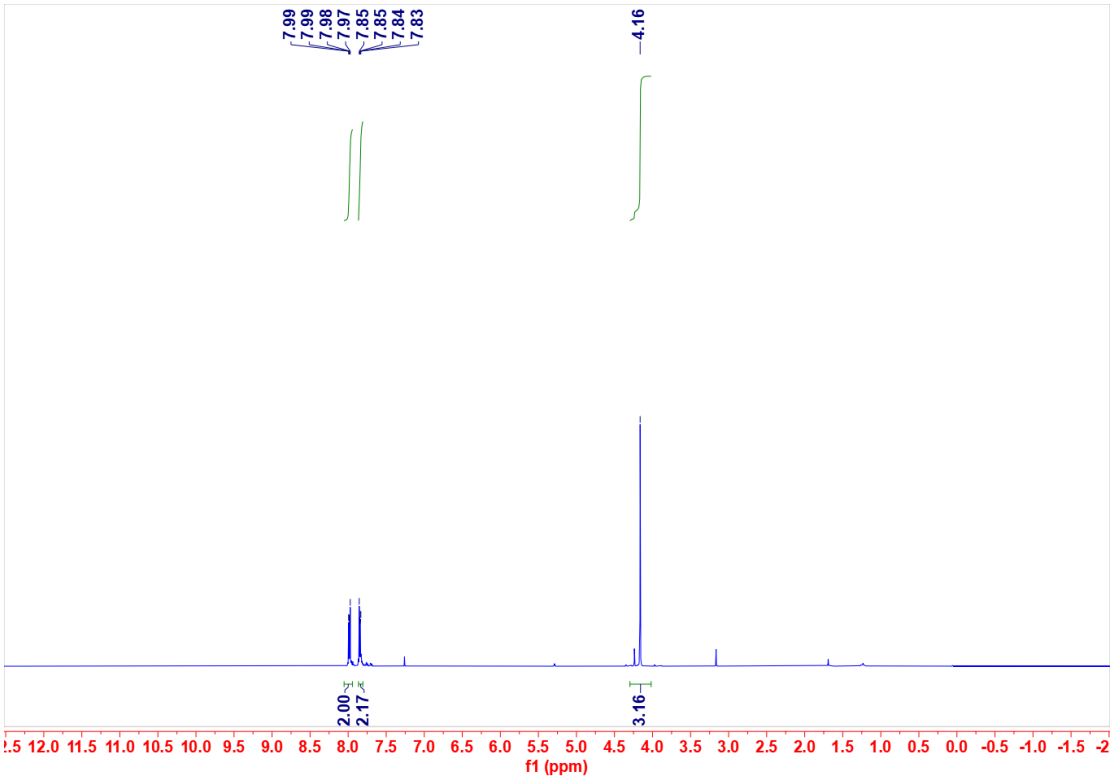
**2e**


8.17
8.17
8.17
8.16
8.15
8.15
7.98
7.98
7.97
7.97
7.96
7.96
7.95
7.95
7.94
7.93
7.93
7.91
7.90
7.90
7.89
7.89
7.89
7.88
7.87
4.76
4.74
4.72
4.70
1.53
1.51
1.49
0.95
1.98
0.97
2.00
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


180.4
156.2
136.4
135.0
126.5
125.8
121.4
121.3
69.0
13.5
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



**2aa**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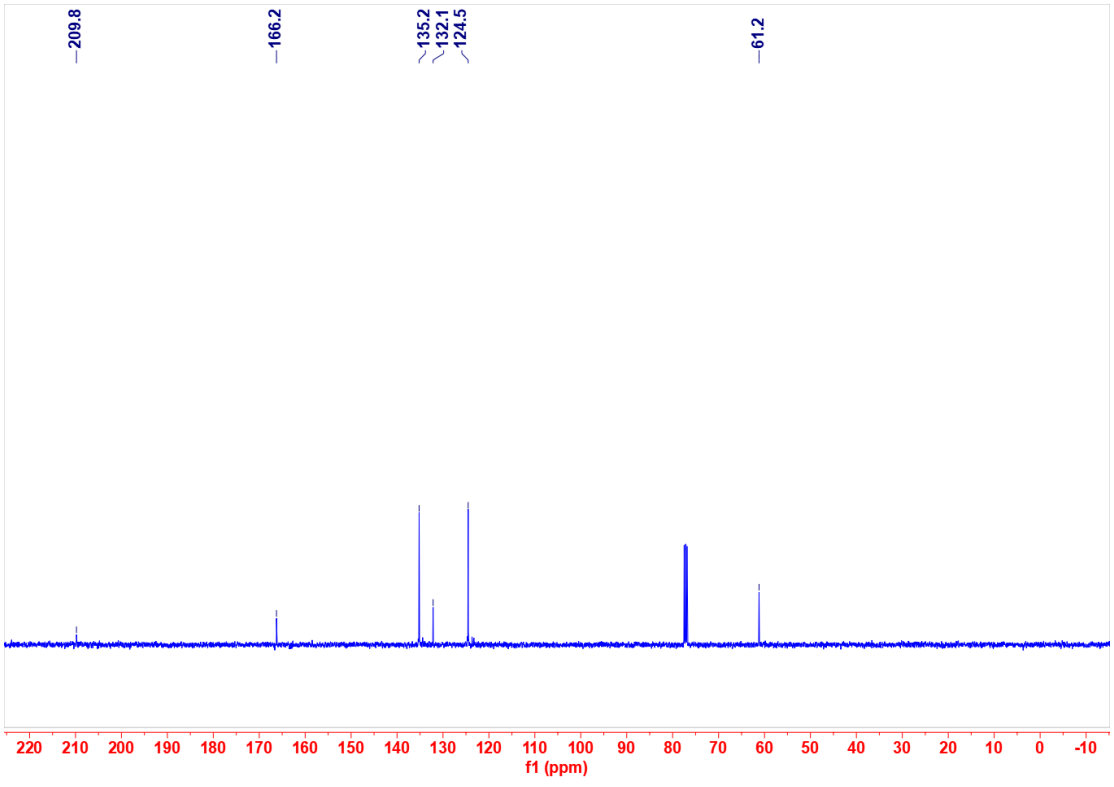
7.99
7.98
7.97
7.85
7.85
7.84
7.83
4.16
2.00
2.17
3.16
f1 (ppm)


209.8
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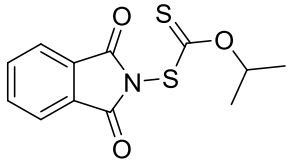
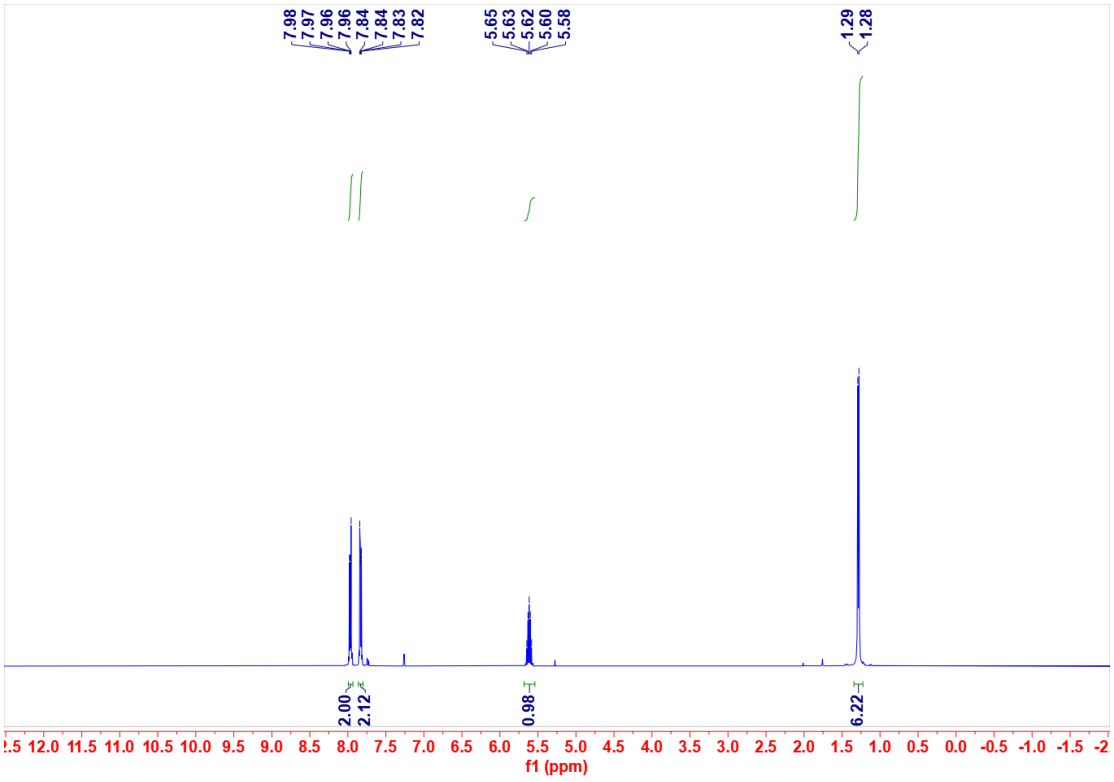
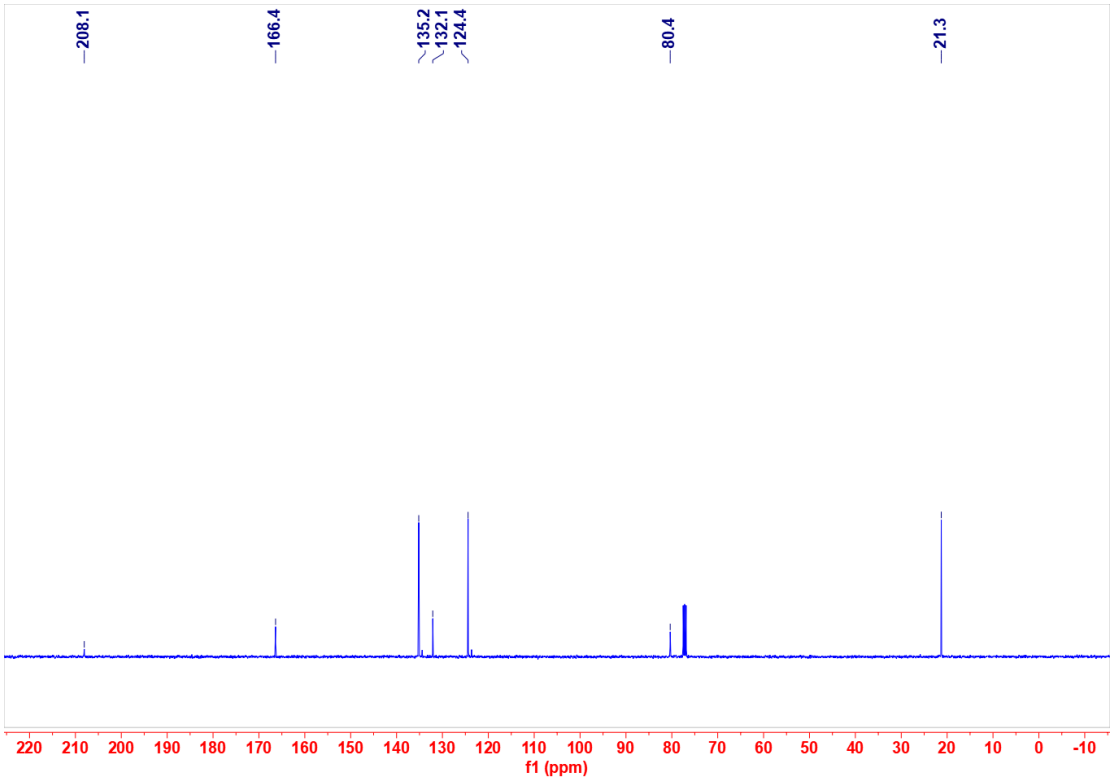
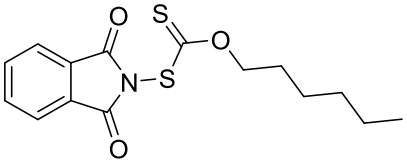
135.2
132.1
124.5
61.2
f1 (ppm)



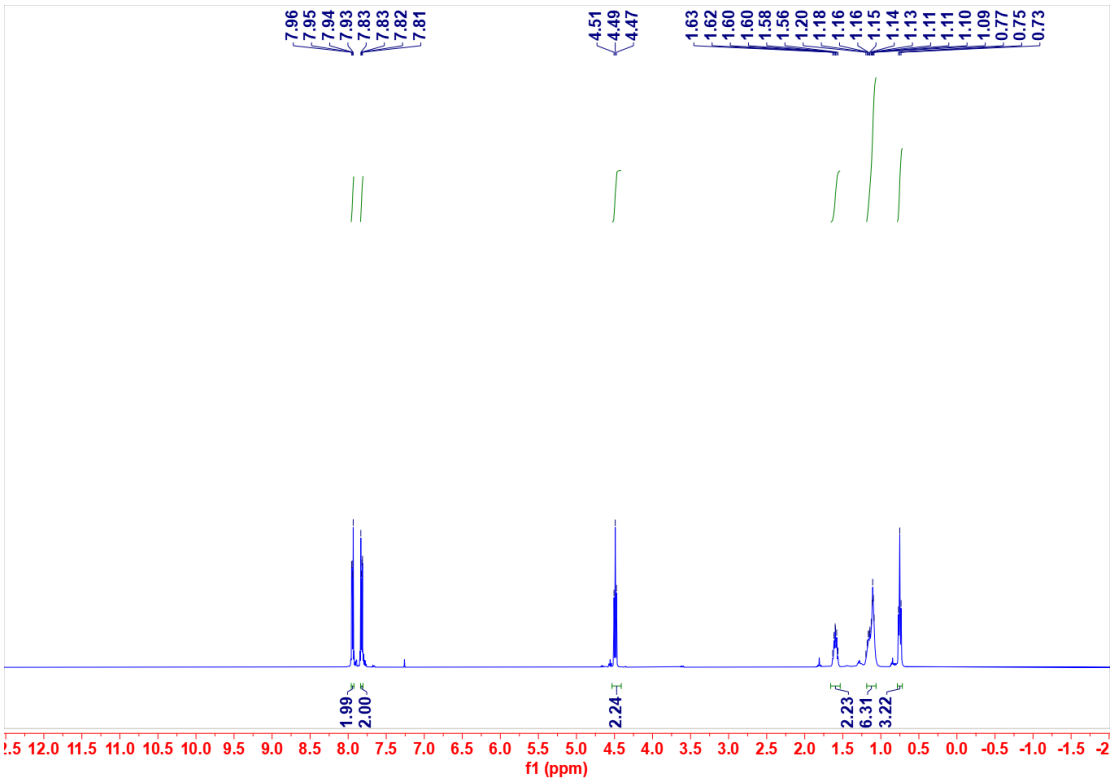
**2ab**

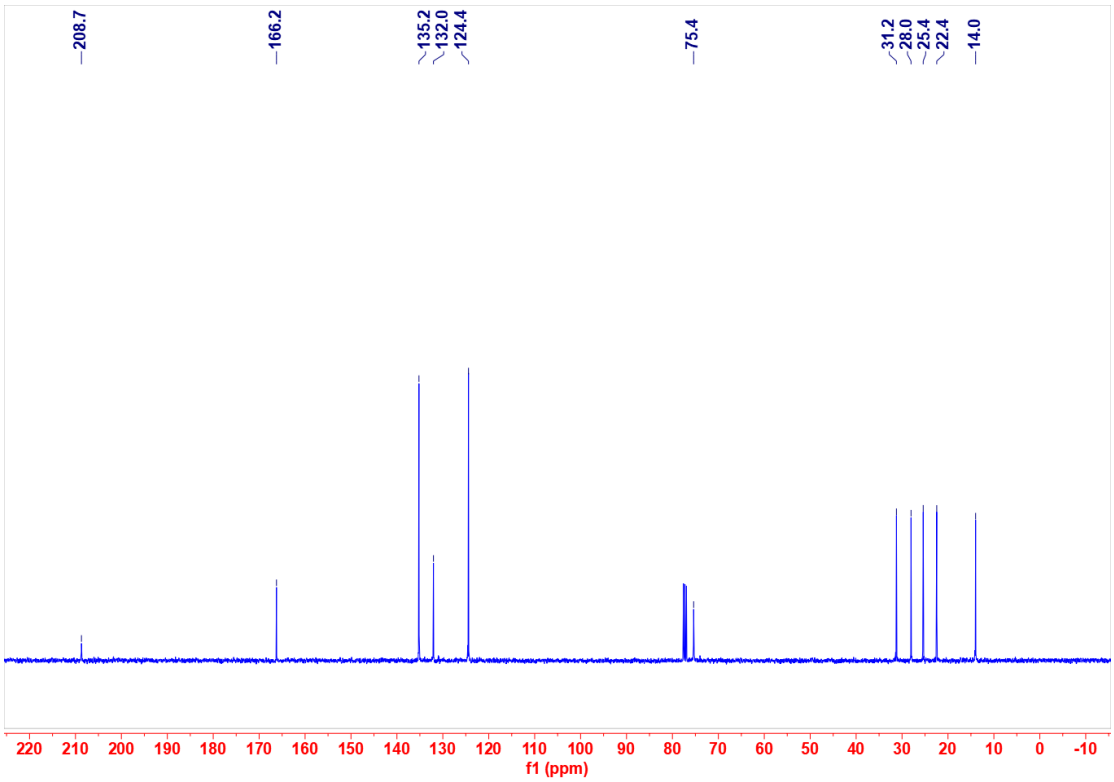

7.98
7.97
7.96
7.96
7.84
7.84
7.83
7.82
5.65
5.63
5.62
5.60
5.58
1.29
1.28
2.00
2.12
0.98
6.22
f1 (ppm)

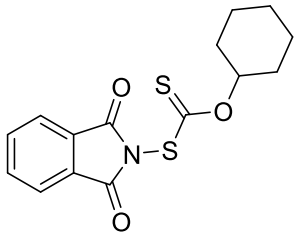

208.1
166.4
135.2
132.1
124.4
80.4
21.3
f1 (ppm)


O
S
O
N
S
O

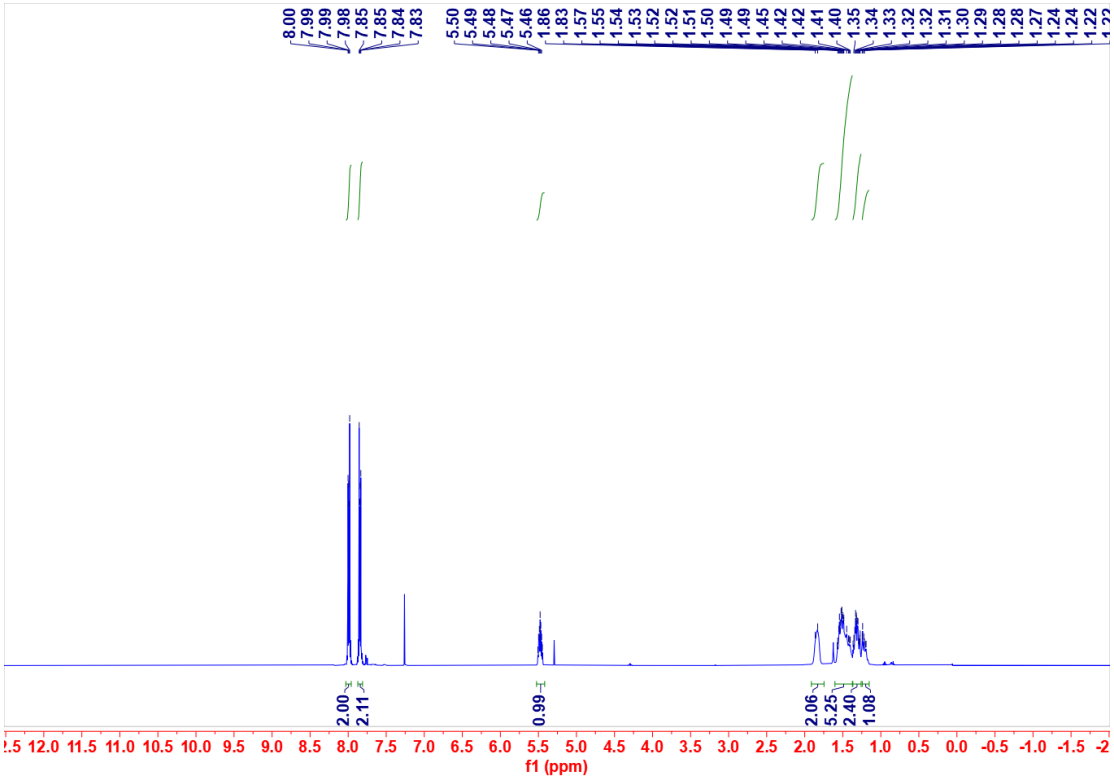
**2ac**

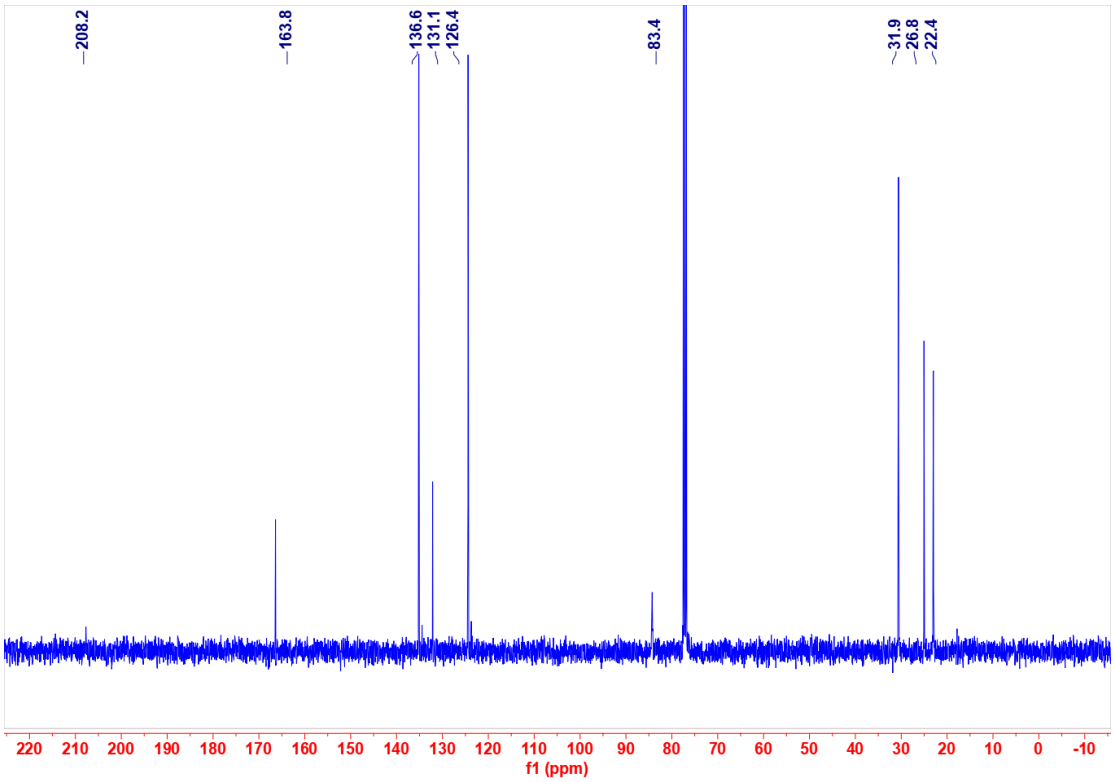

7.96
7.95
7.94
7.93
7.83
7.83
7.82
7.81
4.51
4.49
4.47
1.63
1.62
1.60
1.60
1.58
1.56
1.20
1.18
1.16
1.16
1.15
1.14
1.13
1.11
1.11
1.10
1.09
0.77
0.75
0.73
1.99
2.00
2.24
2.23
6.31
3.22
f1 (ppm)

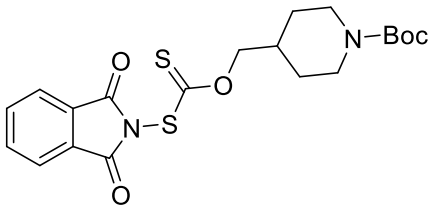

208.7
166.2
135.2
132.0
124.4
75.4
31.2
28.0
25.4
22.4
14.0
f1 (ppm)


O
S
O
N
S
O

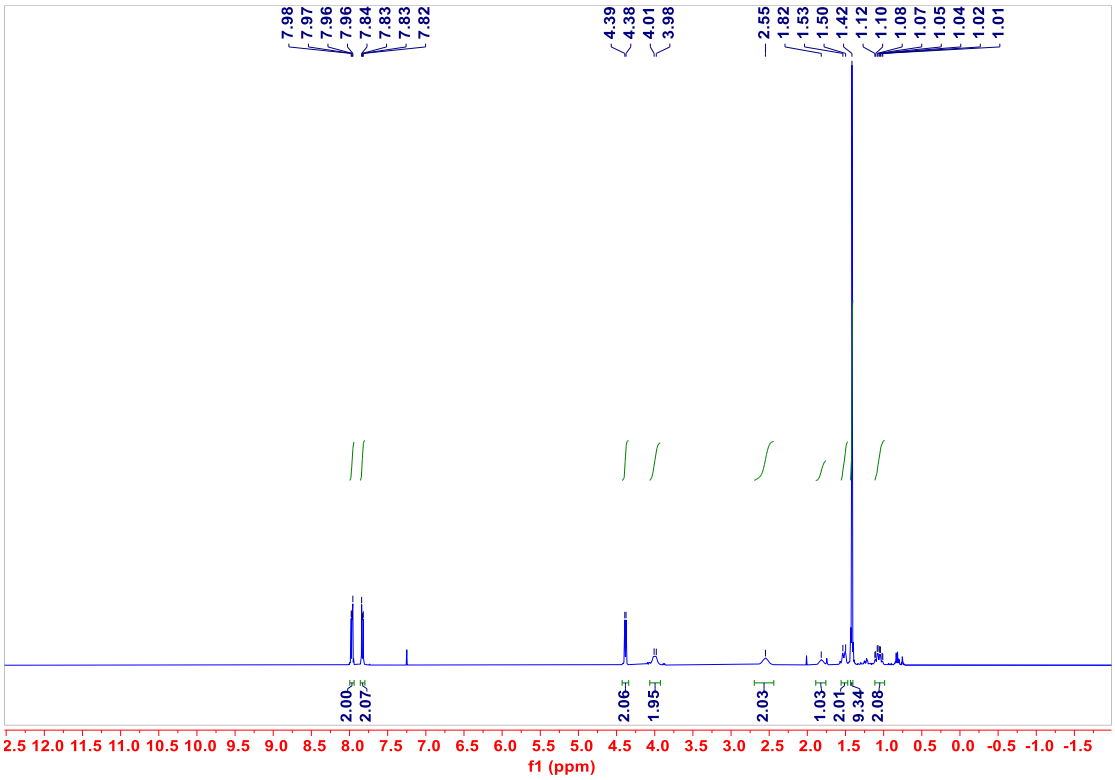
**2ad**

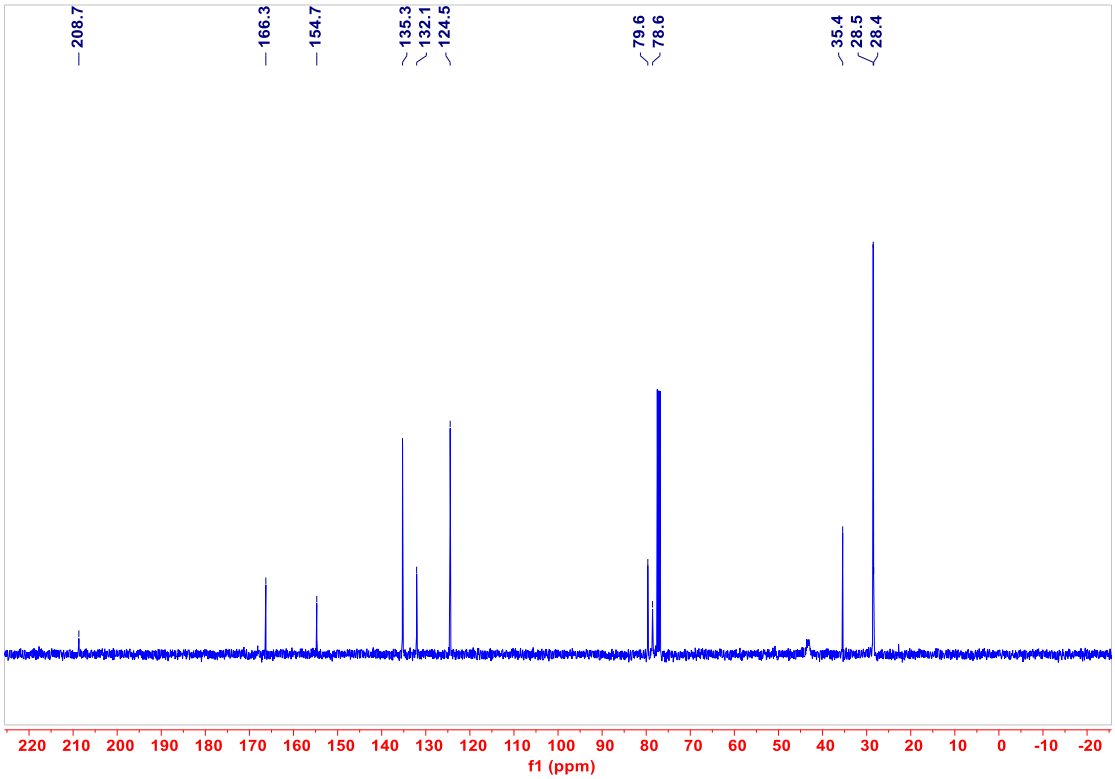

8.00
7.99
7.99
7.98
7.85
7.85
7.84
7.83
5.50
5.49
5.48
5.47
5.46
1.86
1.83
1.57
1.55
1.54
1.53
1.52
1.52
1.51
1.50
1.49
1.49
1.45
1.42
1.42
1.41
1.40
1.35
1.34
1.33
1.32
1.32
1.31
1.30
1.29
1.28
1.28
1.27
1.24
1.24
1.22
2.00
2.11
0.99
2.06
5.25
2.40
1.08
f1 (ppm)

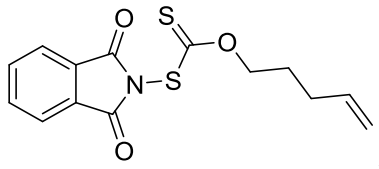

208.2
163.8
136.6
131.1
126.4
83.4
31.9
26.8
22.4
f1 (ppm)


O
S
O
N–Boc
N–S
O

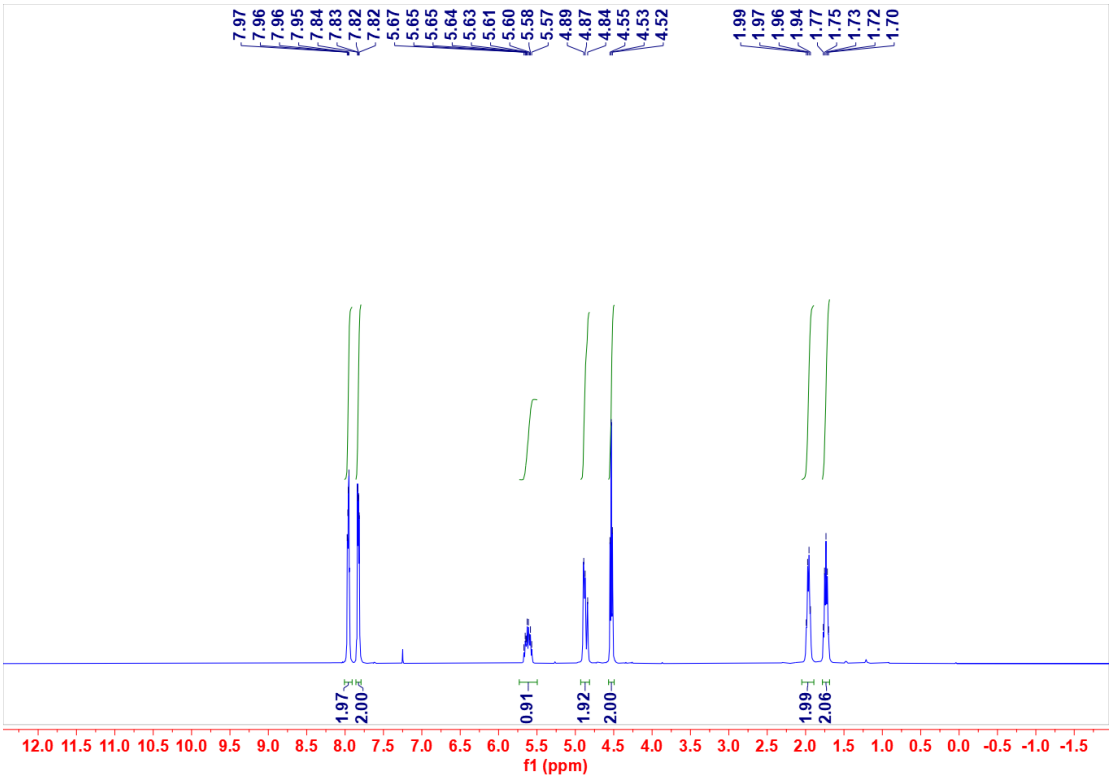
**2ae**

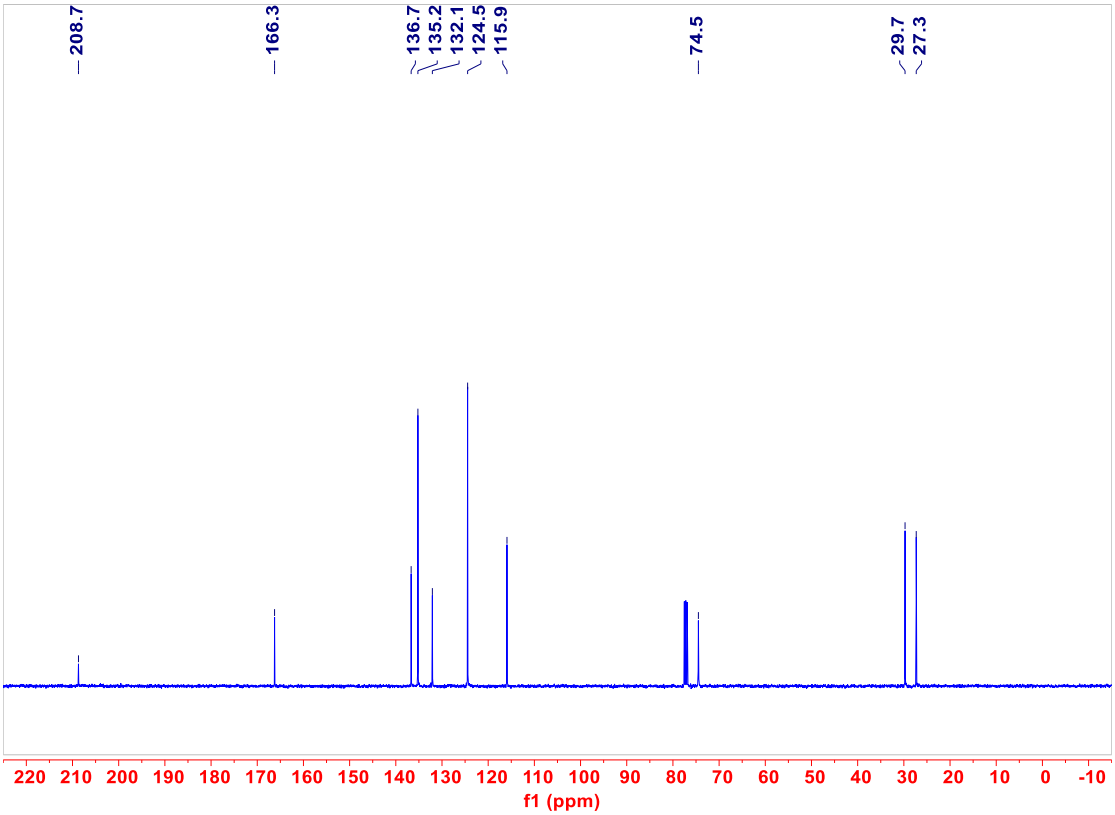

7.98
7.97
7.96
7.96
7.84
7.83
7.83
7.82
4.39
4.38
4.01
3.98
2.55
1.82
1.53
1.50
1.42
1.12
1.10
1.08
1.07
1.05
1.04
1.02
1.01
2.00
2.07
2.06
1.95
2.03
1.03
2.01
9.34
2.08
f1 (ppm)

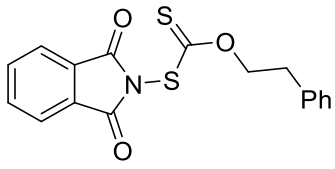

208.7
166.3
154.7
135.3
132.1
124.5
79.6
78.6
35.4
28.5
28.4
f1 (ppm)


O
S
O
N
S
O

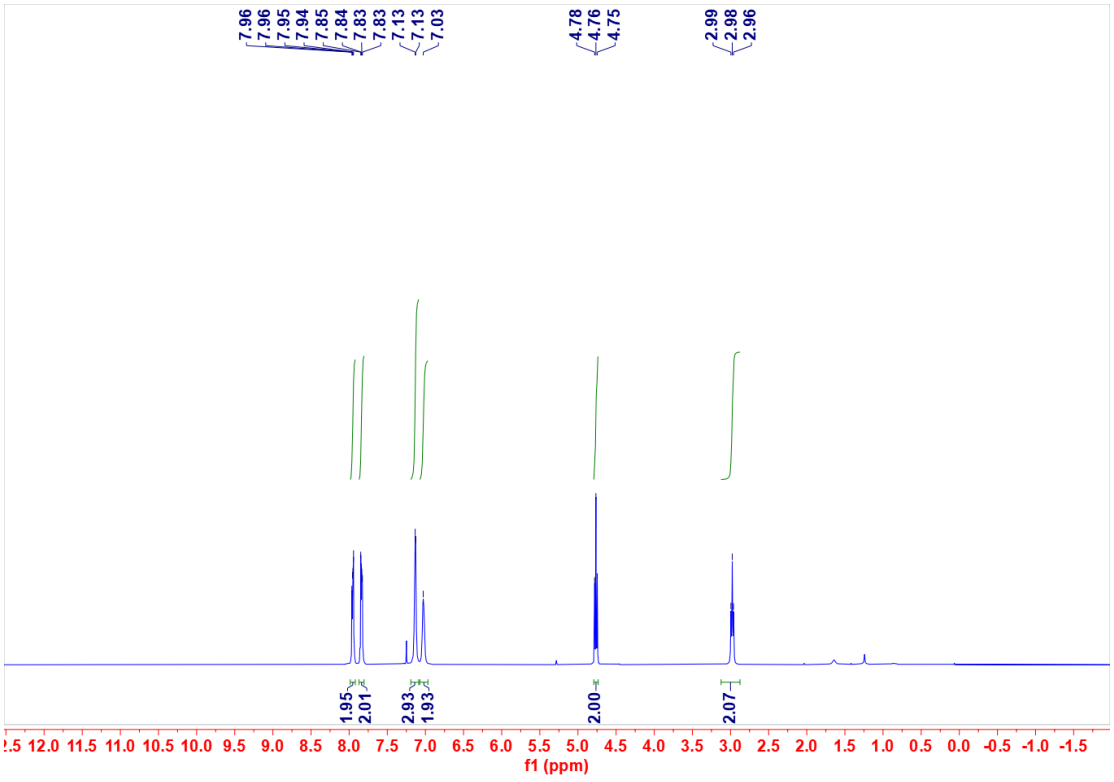
**2af**


7.97
7.96
7.96
7.95
7.84
7.83
7.82
7.82
5.67
5.65
5.65
5.64
5.63
5.61
5.60
5.58
5.57
4.89
4.87
4.84
4.55
4.53
4.52
1.99
1.97
1.96
1.94
1.77
1.75
1.73
1.72
1.70
1.97
2.00
0.91
1.92
2.00
1.99
2.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


208.7
166.3
136.7
135.2
132.1
124.5
115.9
74.5
29.7
27.3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
S
O
N
S
Ph
O

**2ag**


7.96
7.96
7.95
7.94
7.85
7.84
7.83
7.83
7.13
7.13
7.03
4.78
4.76
4.75
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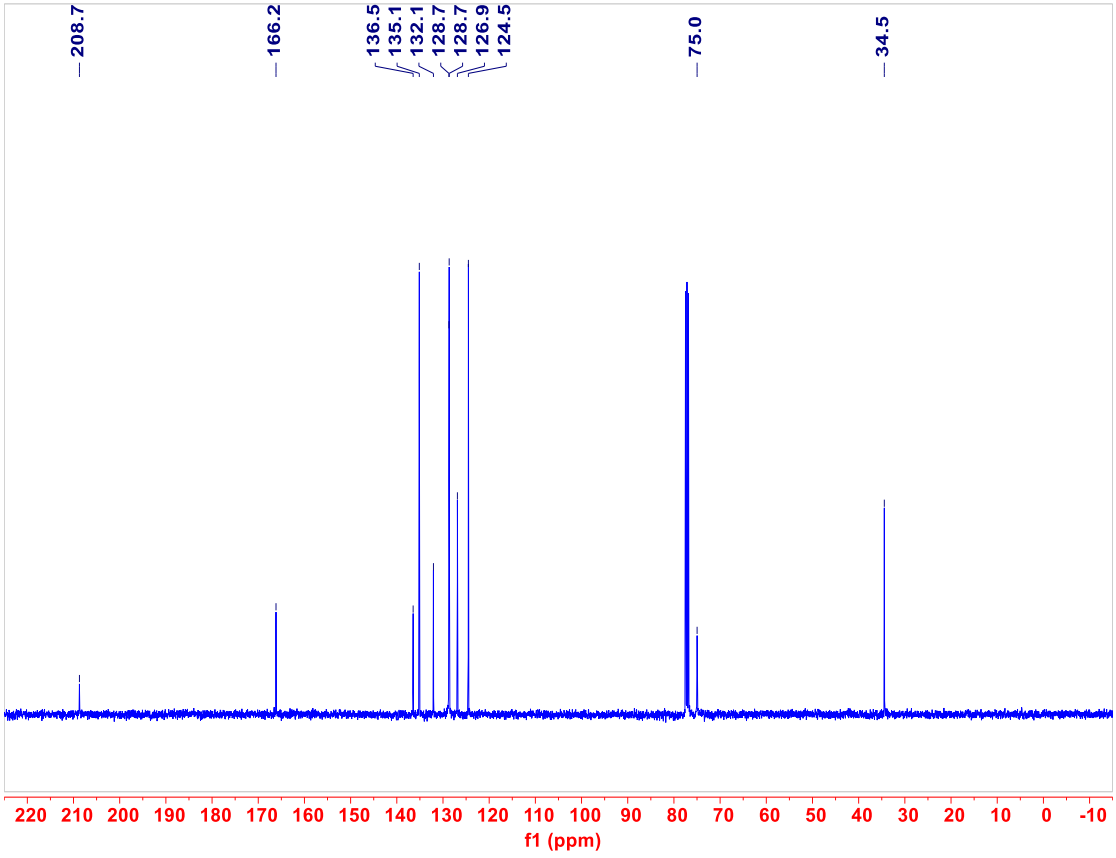
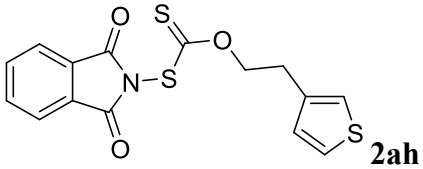
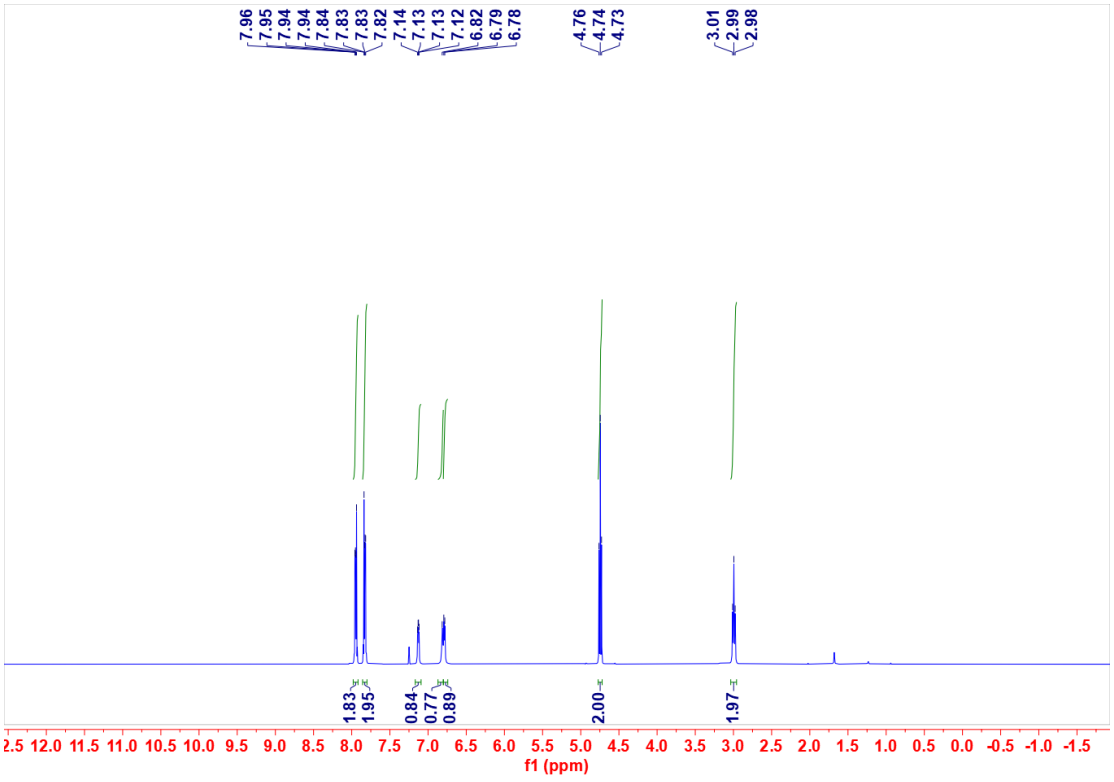
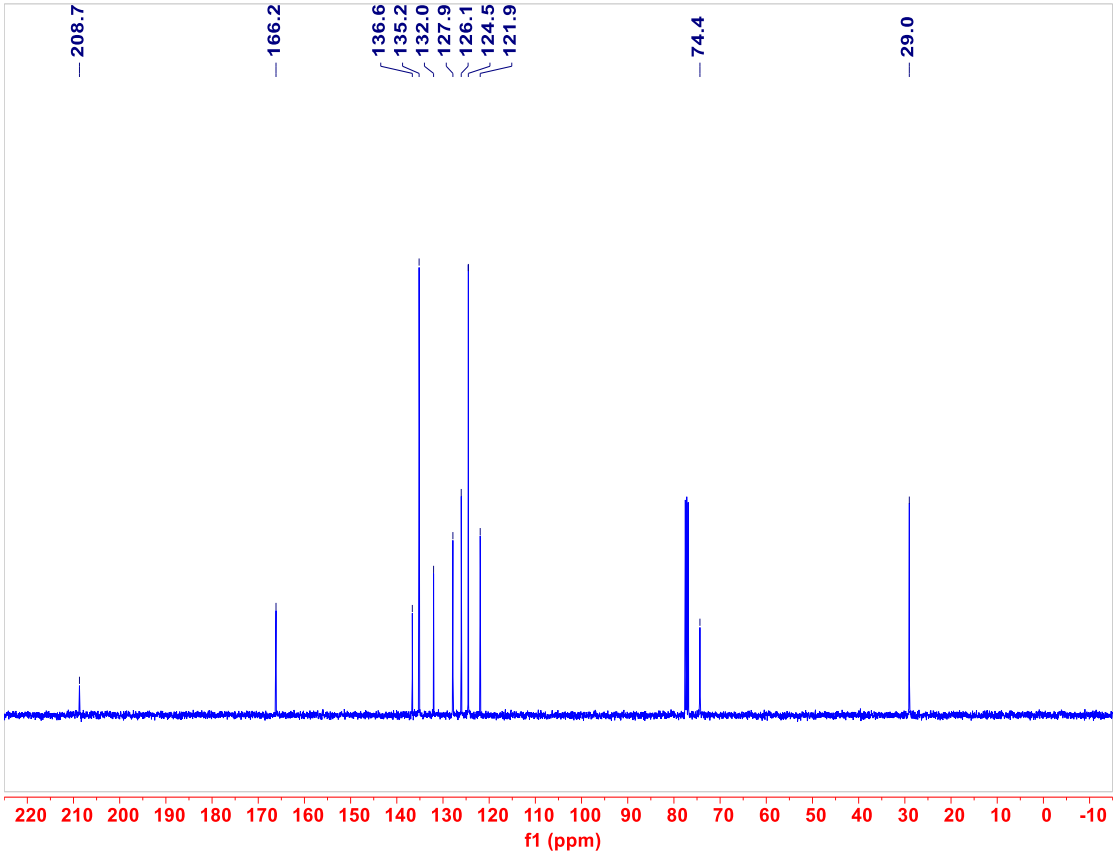
1.95
2.01
2.93
1.93
2.00
2.07
f1 (ppm)

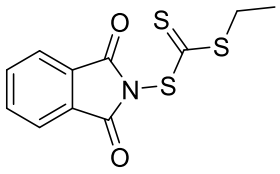

208.7
166.2
136.5
135.1
132.1
128.7
128.7
126.9
124.5
75.0
34.5
f1 (ppm)


O
S
O
N
S
O
S

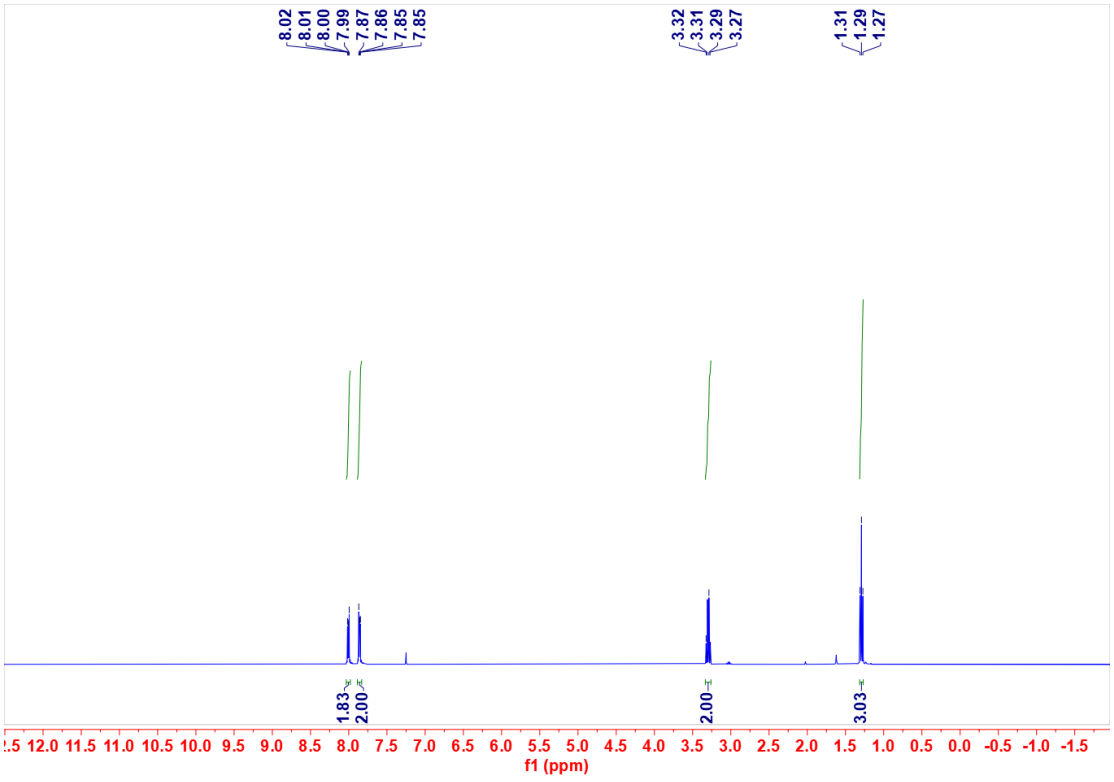
**2ah**

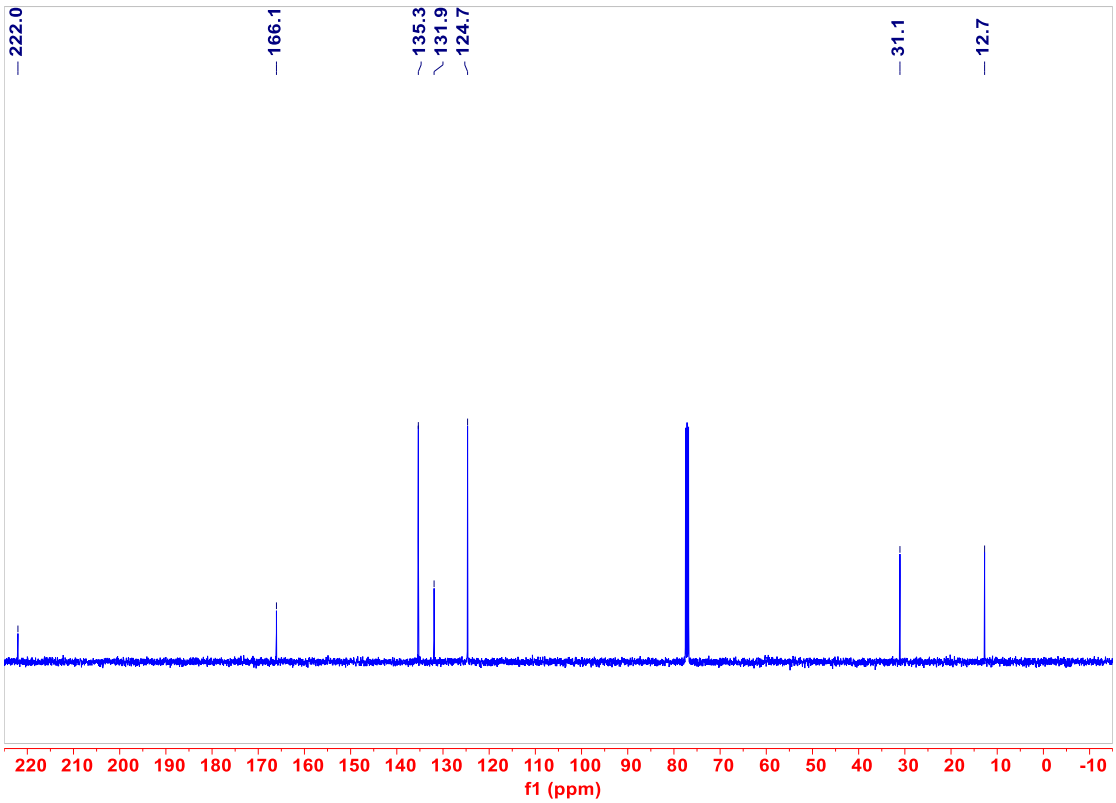

7.96
7.95
7.94
7.94
7.84
7.83
7.83
7.82
7.14
7.13
7.13
7.12
6.82
6.79
6.78
4.76
4.74
4.73
3.01
2.99
2.98
1.83
1.95
0.84
0.77
0.89
2.00
1.97
f1 (ppm)

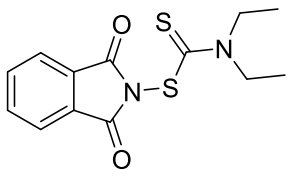

208.7
166.2
136.6
135.2
132.0
127.9
126.1
124.5
121.9
74.4
29.0
f1 (ppm)



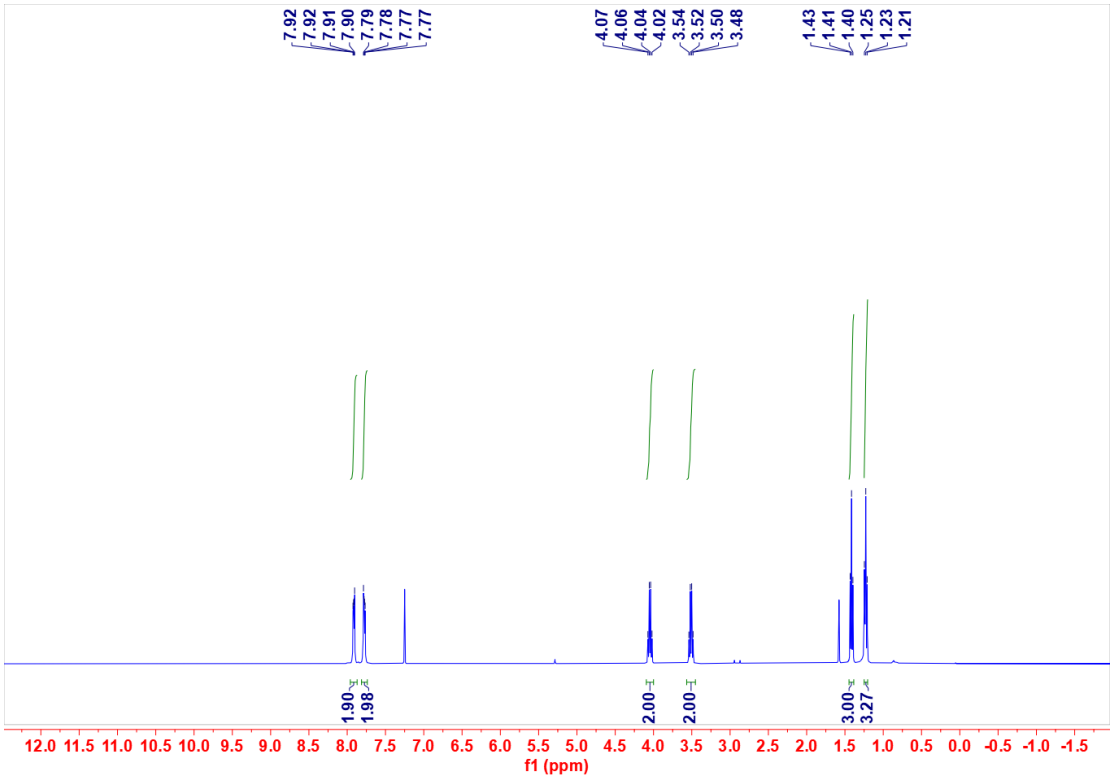
**2ai**

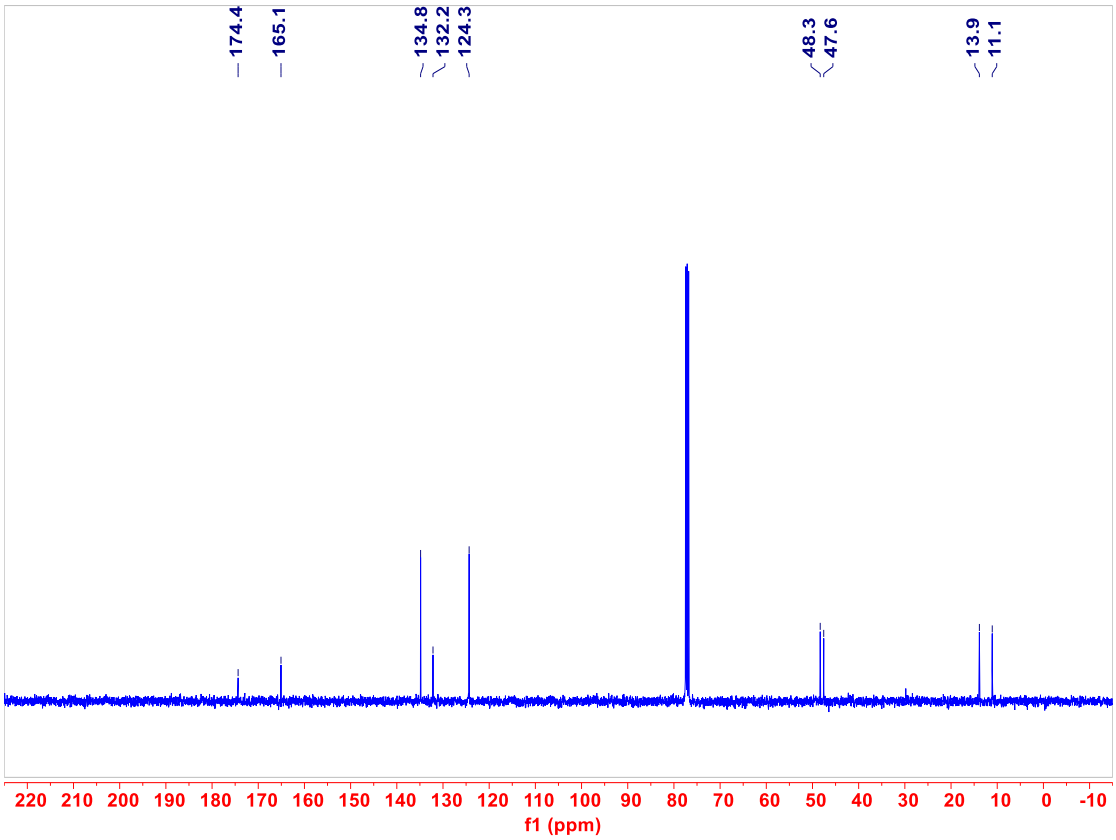

8.02
8.01
8.00
7.99
7.87
7.86
7.85
7.85
3.32
3.31
3.29
3.27
1.31
1.29
1.27
1.83
2.00
2.00
3.03
f1 (ppm)

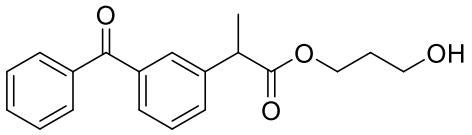

222.0
166.1
135.3
131.9
124.7
31.1
12.7
f1 (ppm)



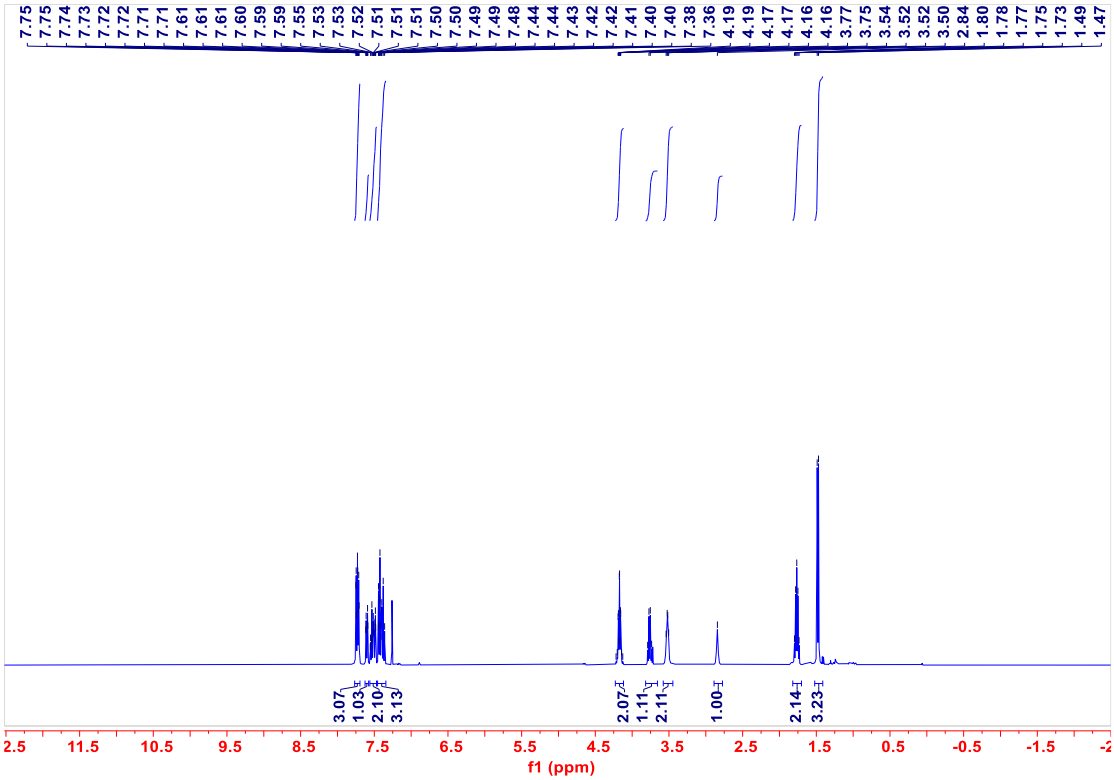
**2aj**

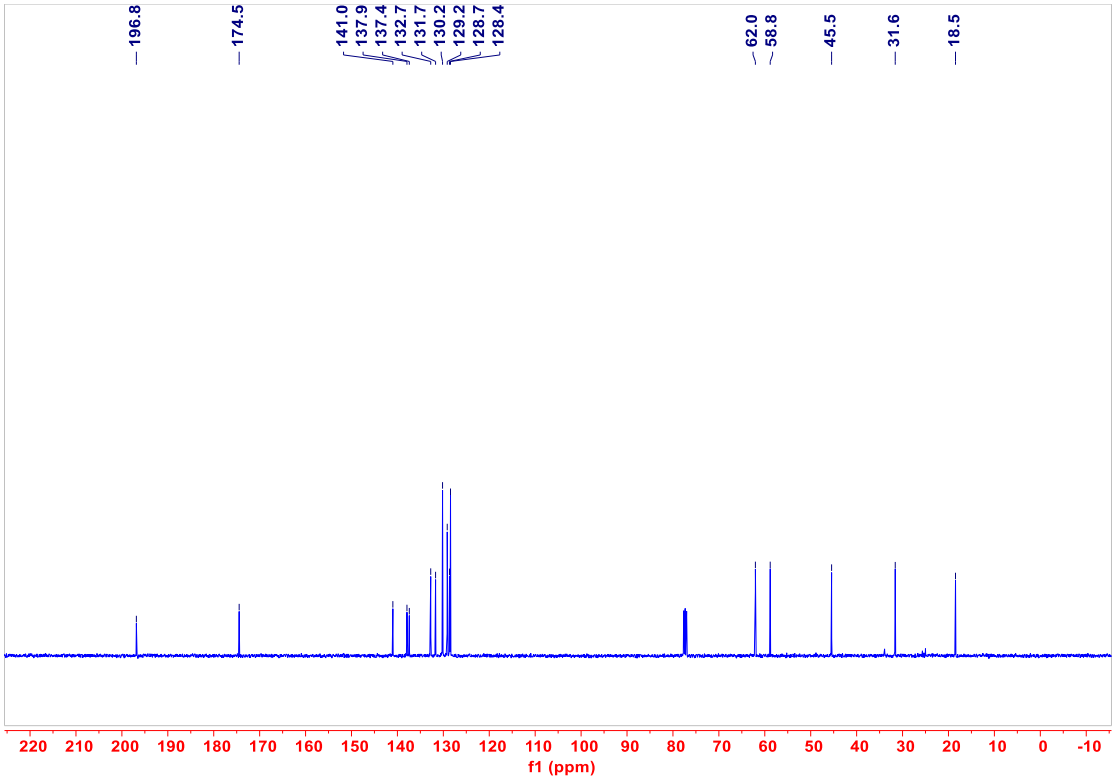

7.92
7.92
7.91
7.90
7.79
7.78
7.77
7.77
4.07
4.06
4.04
4.02
3.54
3.52
3.50
3.48
1.43
1.41
1.40
1.25
1.23
1.21
1.90
1.98
2.00
2.00
3.00
3.27
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

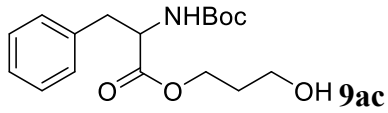

174.4
165.1
134.8
132.2
124.3
48.3
47.6
13.9
11.1
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
O
O
OH

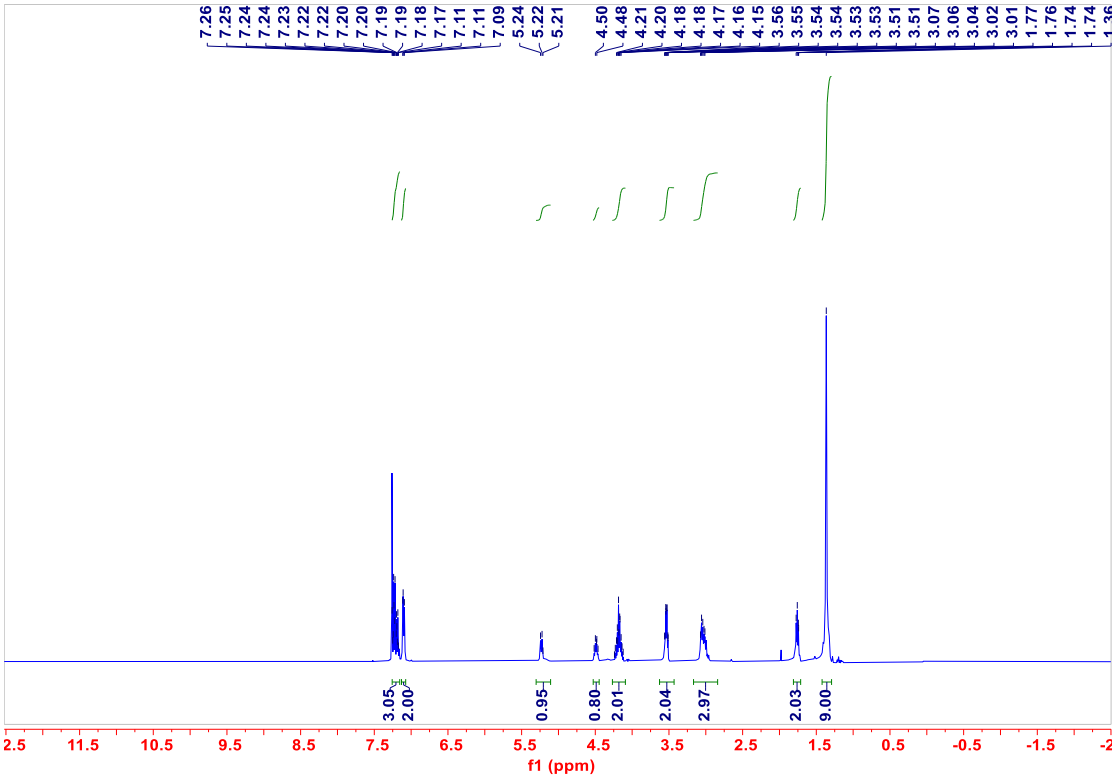
**9ab**

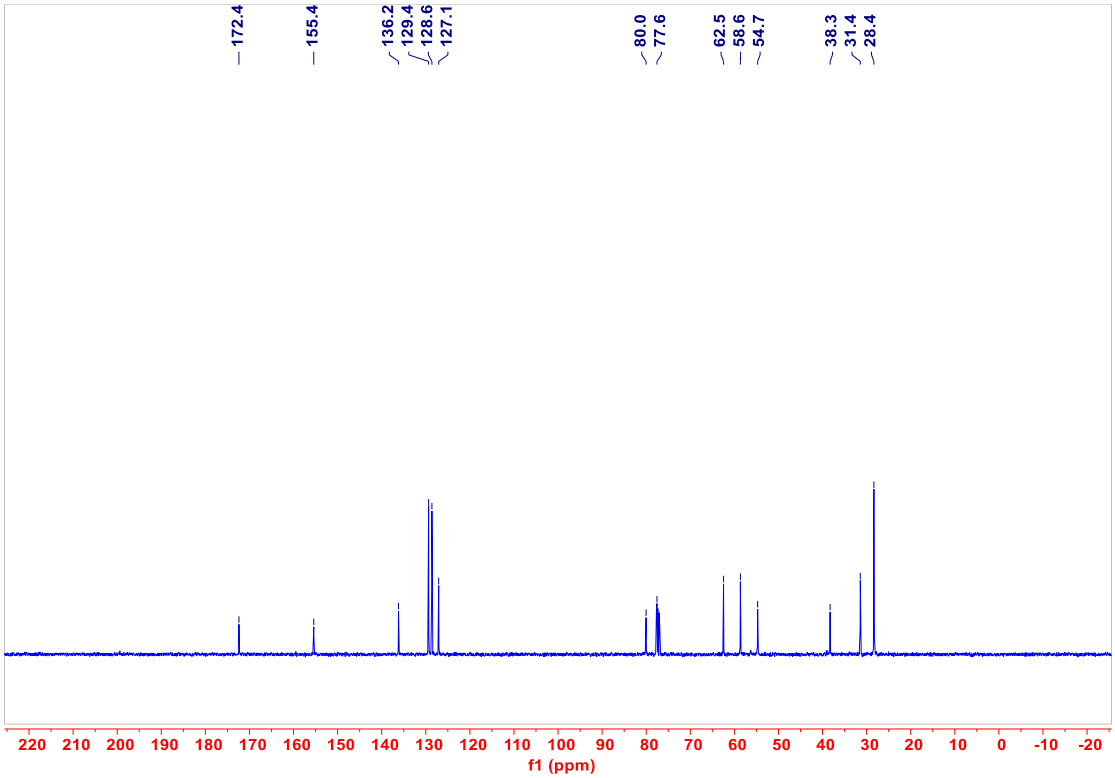

7.75 7.75 7.74 7.73 7.72 7.72 7.71 7.71 7.61 7.61 7.61 7.60 7.59 7.59 7.55 7.53 7.53 7.52 7.51 7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.44 7.44 7.43 7.42 7.42 7.41 7.40 7.40 7.38 7.36 4.19 4.19 4.17 4.17 4.16 4.16 3.77 3.75 3.54 3.52 3.52 3.50 2.84 1.80 1.78 1.77 1.75 1.73 1.49 1.47
3.07 1.03 2.10 3.13 2.07 1.11 2.11 1.00 2.14 3.23
2.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5
f1 (ppm)

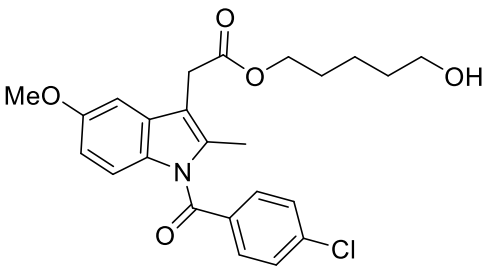

196.8 174.5 141.0 137.9 137.4 132.7 131.7 130.2 129.2 128.7 128.4 62.0 58.8 45.5 31.6 18.5
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


NHBoc
O
O
OH

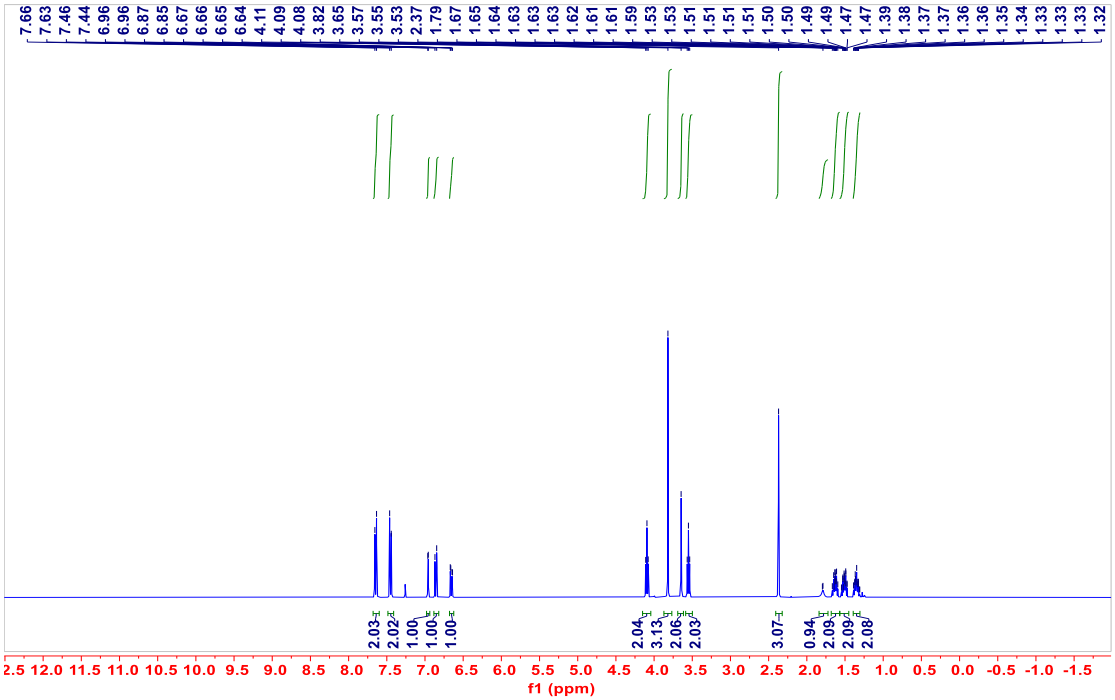
**9ac**

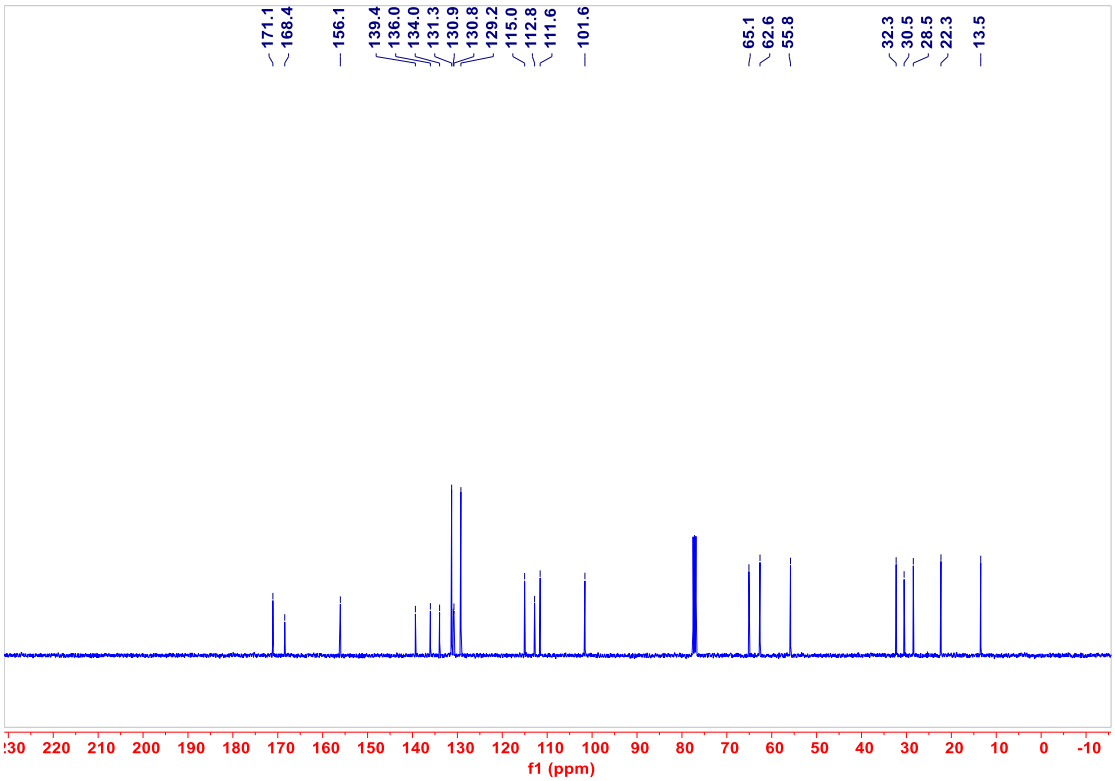

7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.20 7.20 7.19 7.19 7.18 7.17 7.11 7.11 7.09 5.24 5.22 5.21 4.50 4.48 4.21 4.20 4.18 4.18 4.17 4.16 4.15 3.56 3.55 3.54 3.54 3.53 3.53 3.51 3.51 3.07 3.06 3.04 3.02 3.01 1.77 1.76 1.74 1.74
3.05 2.00 0.95 0.80 2.01 2.04 2.97 2.03 9.00
2.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5 -2
f1 (ppm)

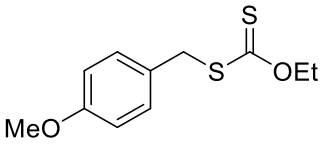

172.4 155.4 136.2 129.4 128.6 127.1 80.0 77.6 62.5 58.6 54.7 38.3 31.4 28.4
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


O
O
OH
MeO
N
O
Cl

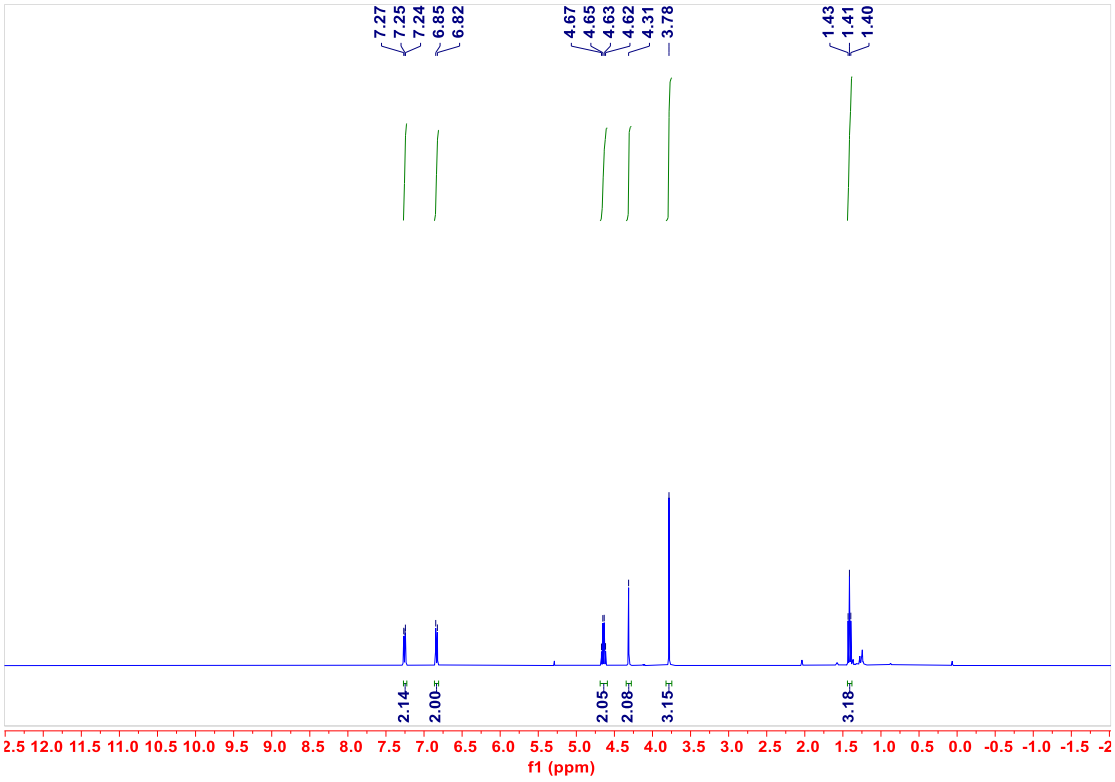
**9ad**

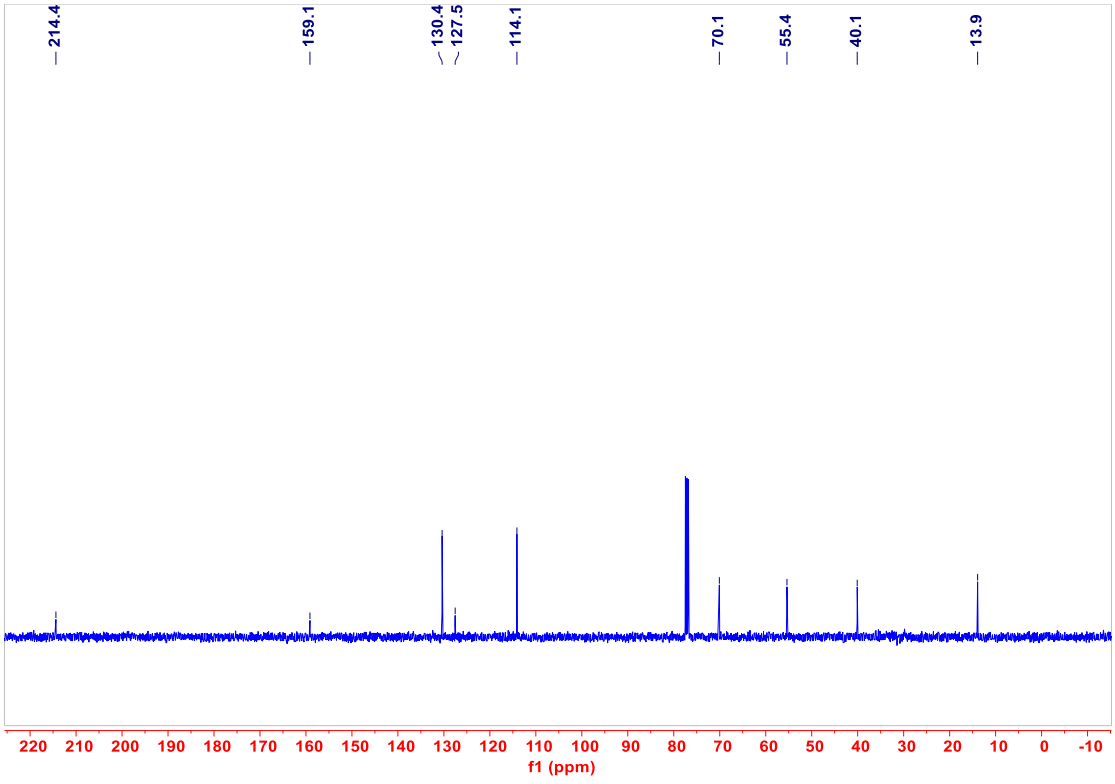

7.66 7.63 7.46 7.44 6.96 6.96 6.87 6.85 6.67 6.66 6.65 6.64 4.11 4.09 4.08 3.82 3.65 3.57 3.55 3.53 2.37 1.79 1.67 1.65 1.64 1.63 1.63 1.63 1.62 1.61 1.61 1.59 1.53 1.53 1.51 1.51 1.51 1.51 1.51 1.50 1.50 1.49 1.49 1.47 1.47 1.39 1.38 1.37 1.37 1.36 1.36 1.35 1.34 1.33 1.33 1.33 1.32
2.03 2.02 1.00 1.00 1.00 2.04 3.13 2.06 2.03 3.07 0.94 2.09 2.09 2.08
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

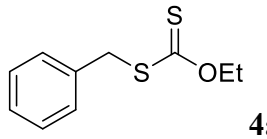

171.1 168.4 156.1 139.4 136.0 134.0 131.3 130.9 130.8 129.2 115.0 112.8 111.6 101.6 65.1 62.6 55.8 32.3 30.5 28.5 22.3 13.5
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt
MeO

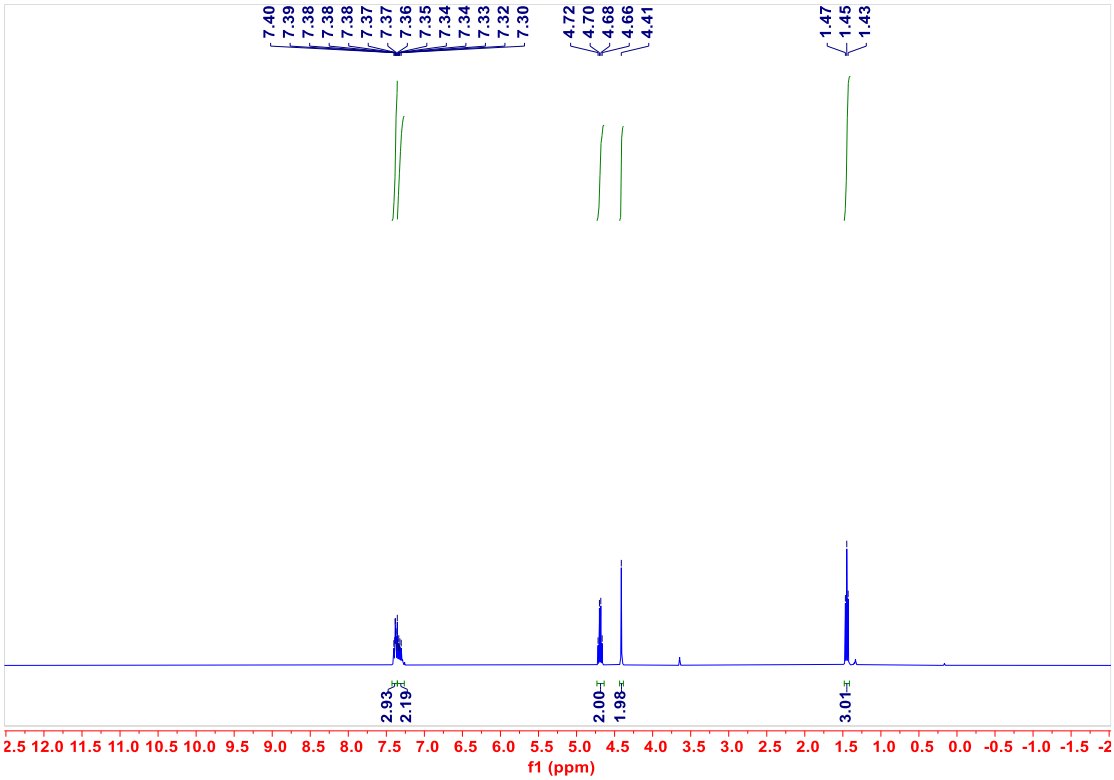
**4aa**

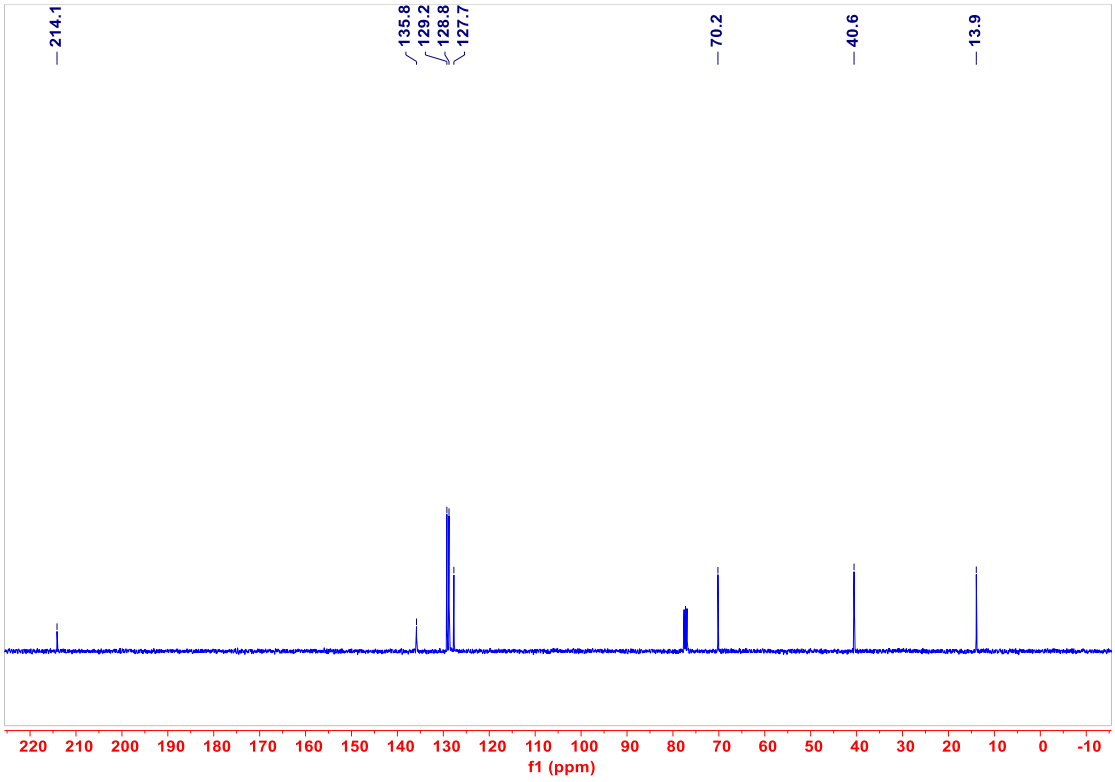

7.27
7.25
7.24
6.85
6.82
4.67
4.65
4.63
4.62
4.31
3.78
1.43
1.41
1.40
2.14
2.00
2.05
2.08
3.15
3.18
f1 (ppm)

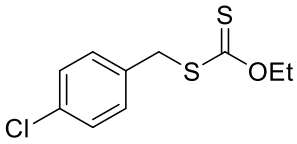

214.4
159.1
130.4
127.5
114.1
70.1
55.4
40.1
13.9
f1 (ppm)


S
S
OEt

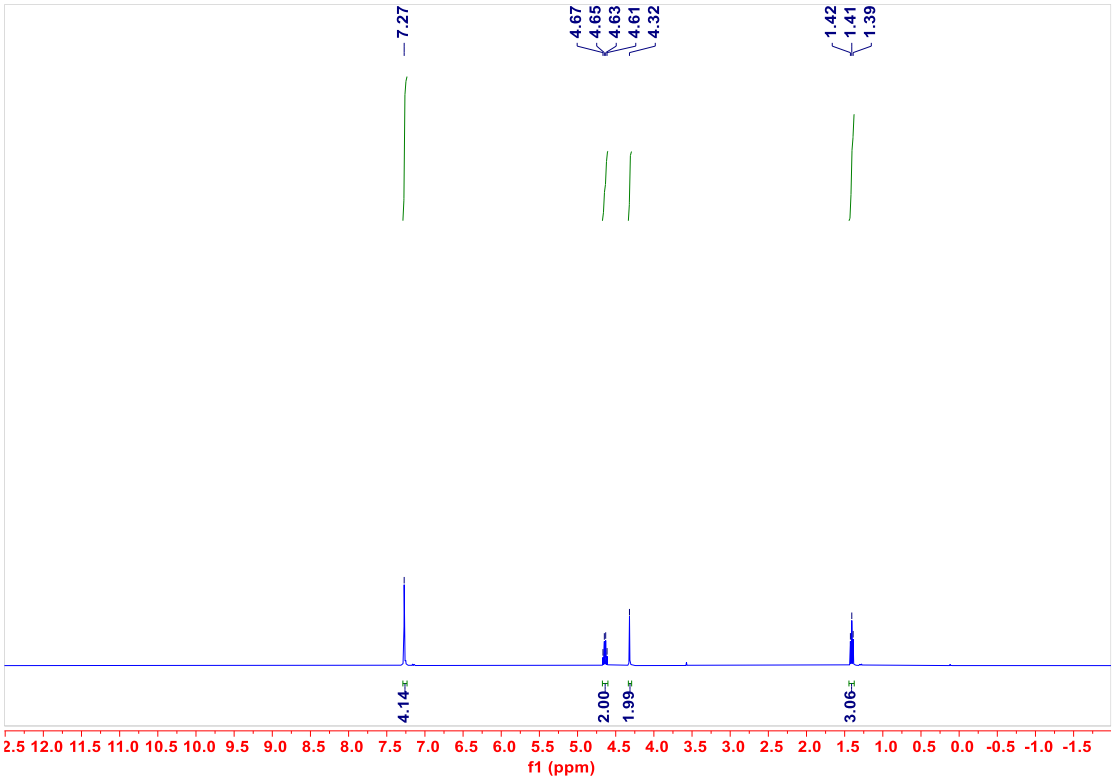
**4ab**

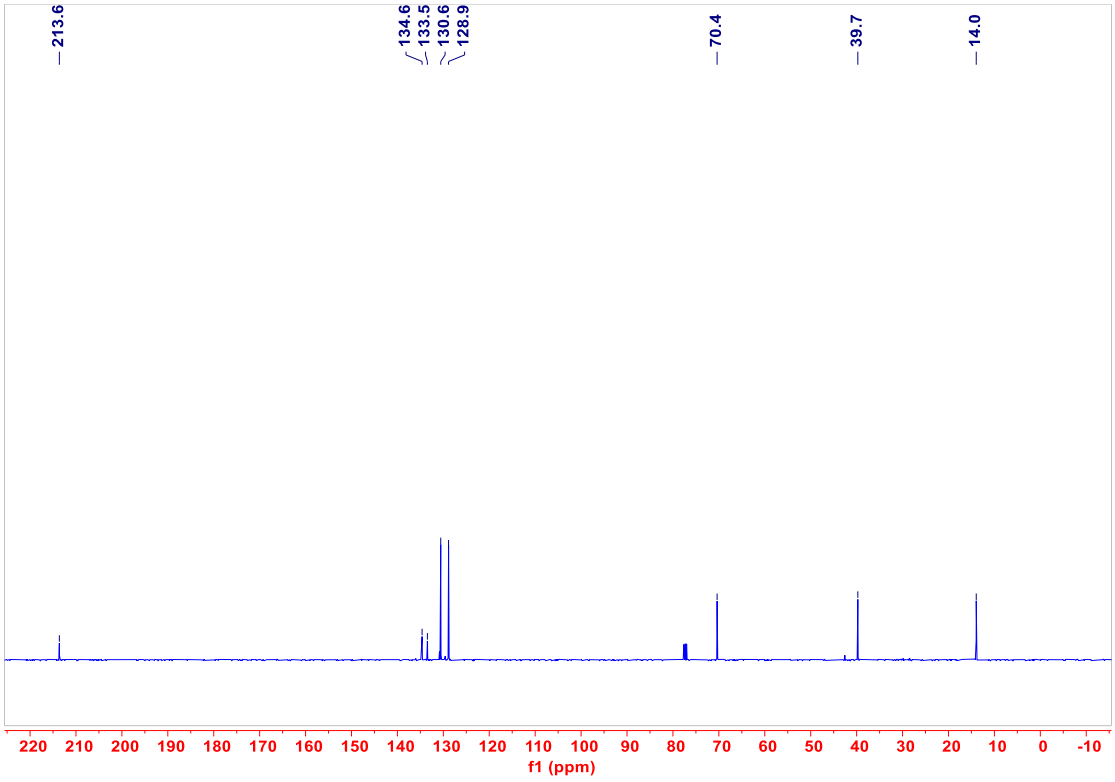

7.40
7.39
7.38
7.38
7.38
7.37
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.30
4.72
4.70
4.68
4.66
4.41
1.47
1.45
1.43
2.93
2.19
2.00
1.98
3.01
f1 (ppm)

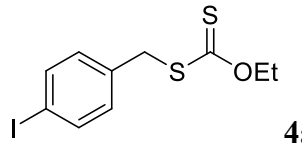

214.1
135.8
129.2
128.8
127.7
70.2
40.6
13.9
f1 (ppm)


S
S
OEt
Cl

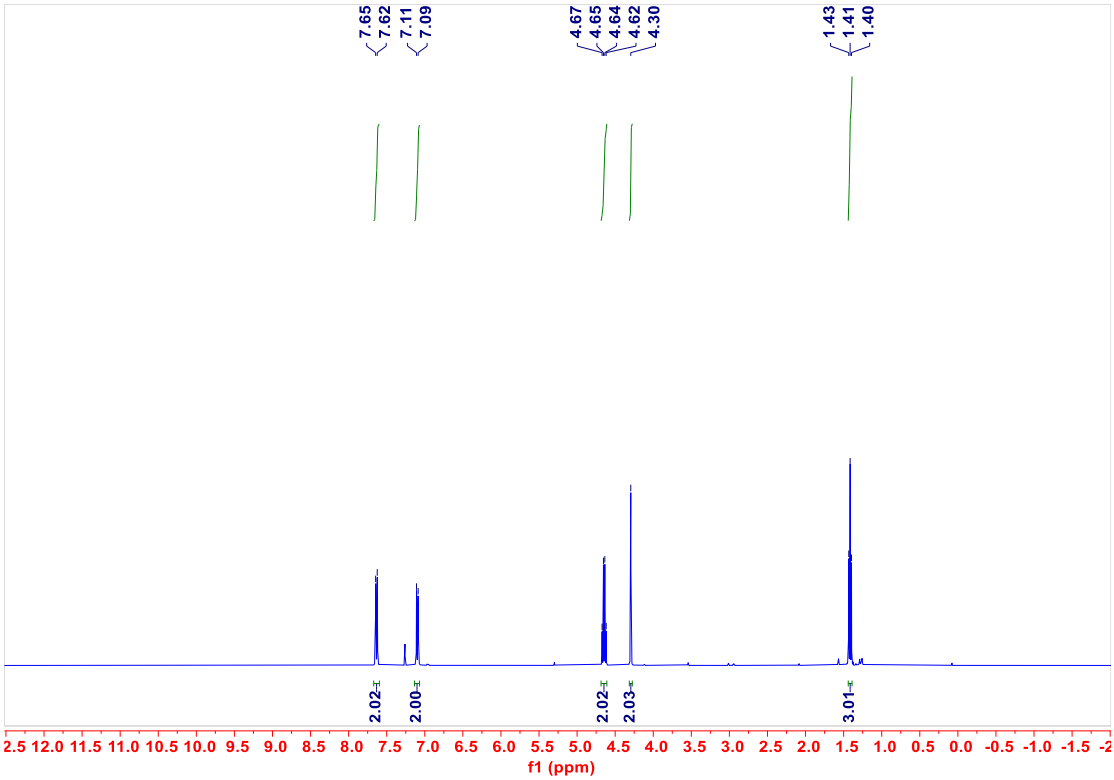
**4ac**

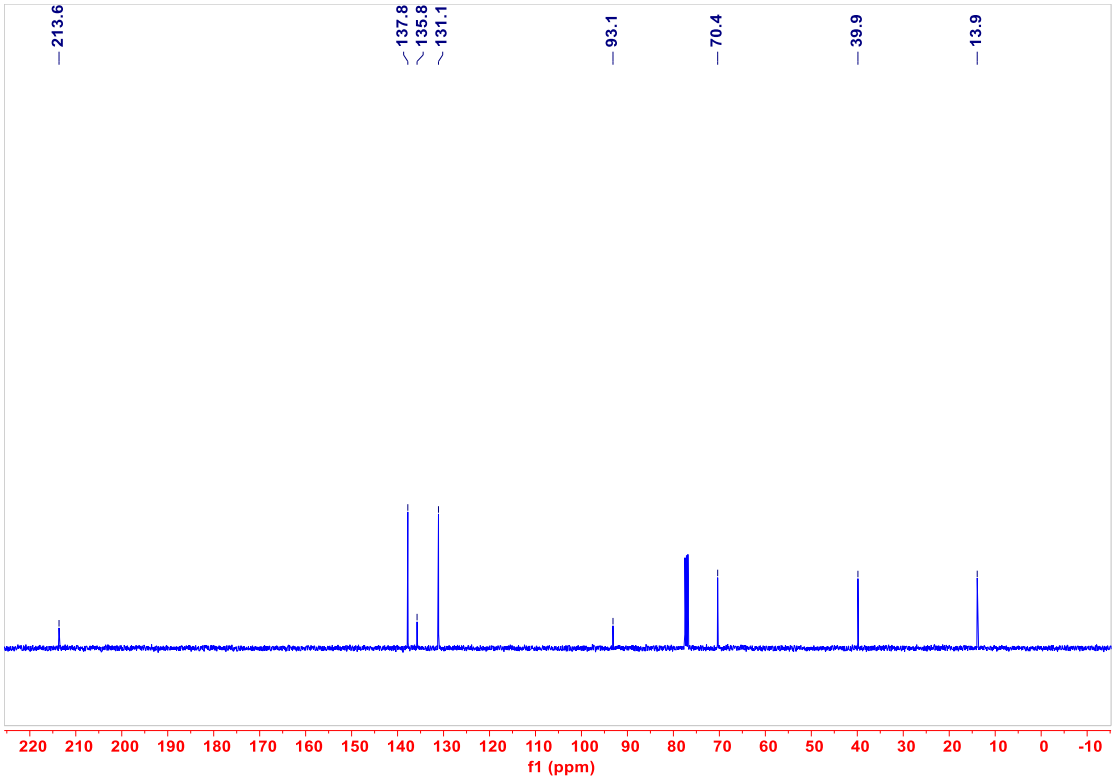

7.27
4.67
4.65
4.63
4.61
4.32
1.42
1.41
1.39
4.14
2.00
1.99
3.06
f1 (ppm)

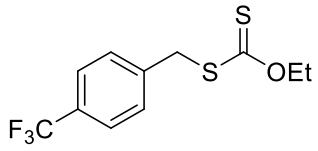

213.6
134.6
133.5
130.6
128.9
70.4
39.7
14.0
f1 (ppm)


S
S
OEt
I

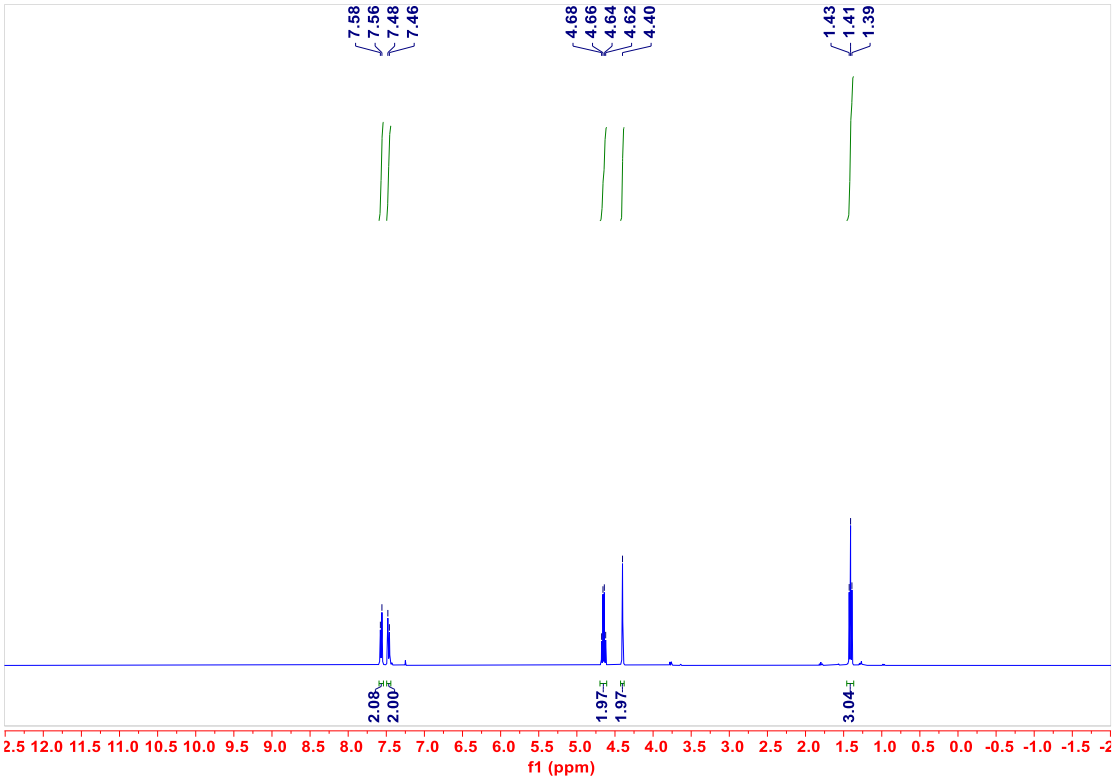
**4ad**

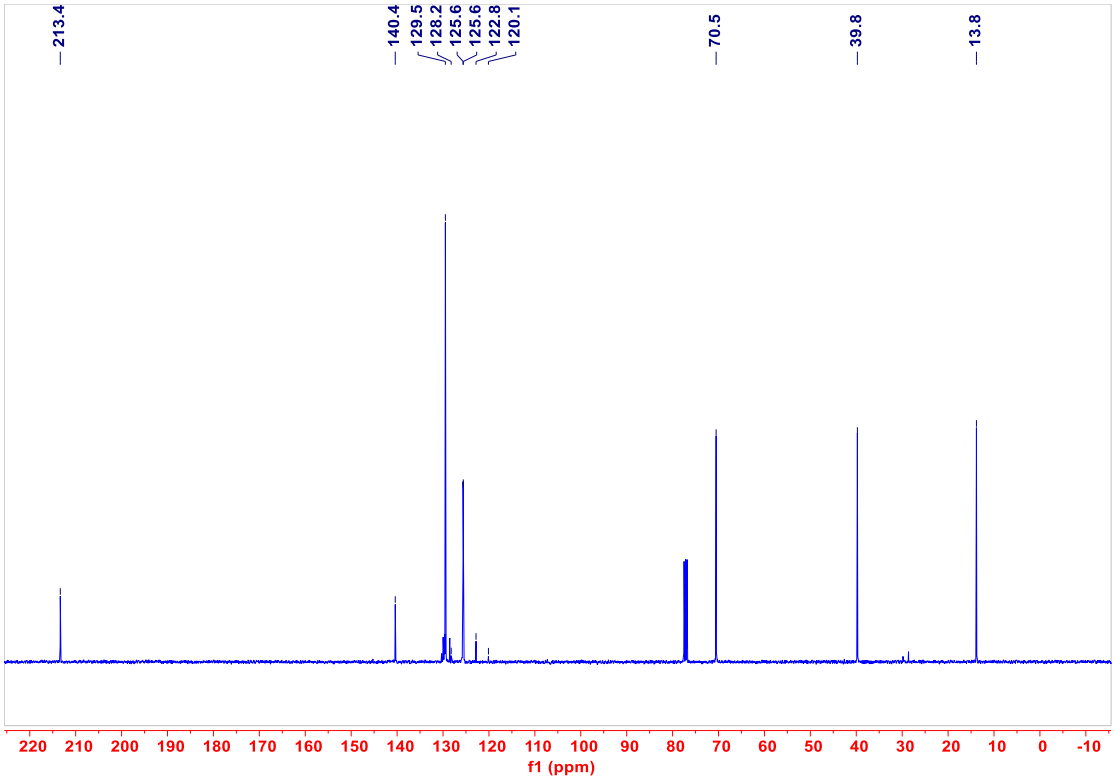

7.65
7.62
7.11
7.09
4.67
4.65
4.64
4.62
4.30
1.43
1.41
1.40
2.02
2.00
2.02
2.03
3.01
f1 (ppm)

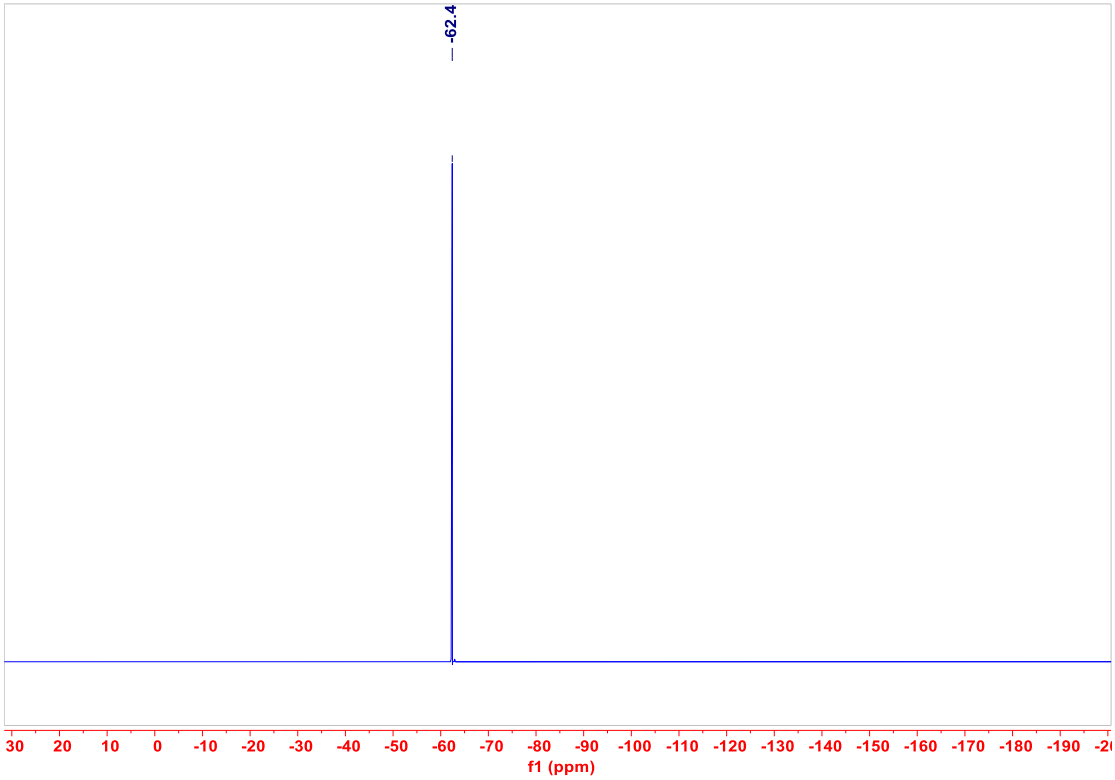

213.6
137.8
135.8
131.1
93.1
70.4
39.9
13.9
f1 (ppm)

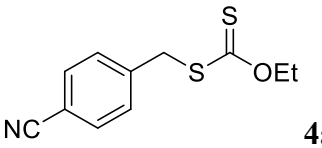

S
S
OEt
F3C

**4ae**

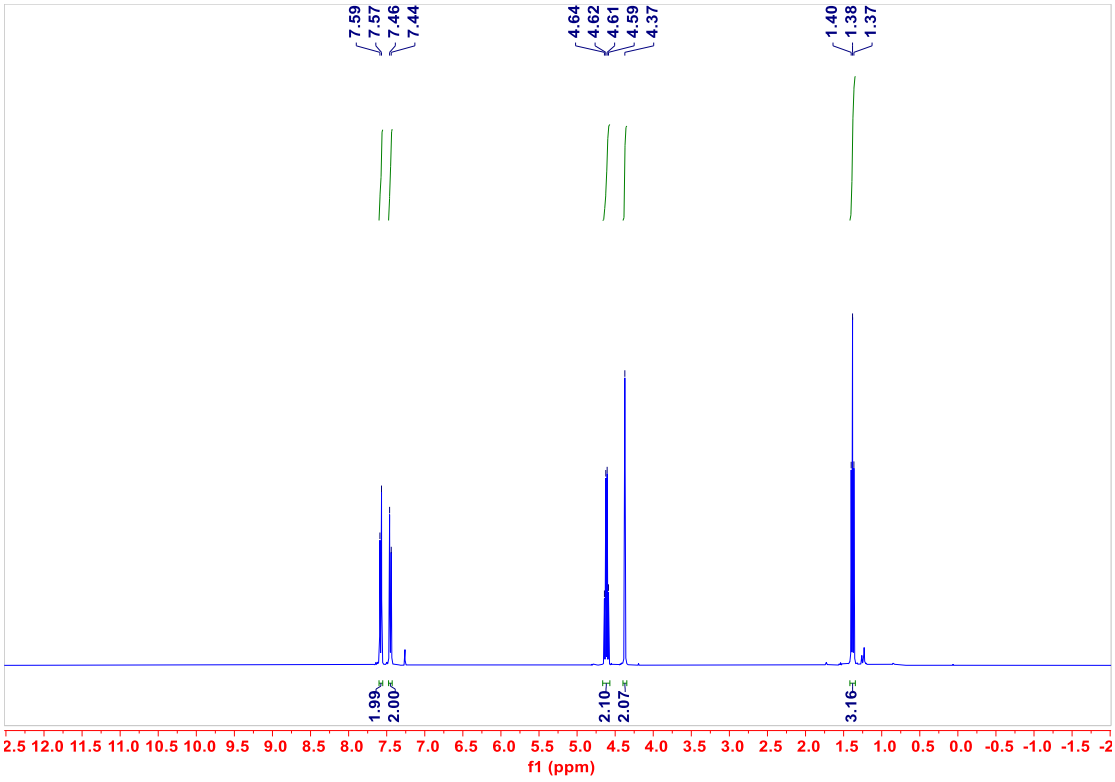

7.58
7.56
7.48
7.46
4.68
4.66
4.64
4.62
4.40
1.43
1.41
1.39
2.08
2.00
1.97
1.97
3.04
f1 (ppm)

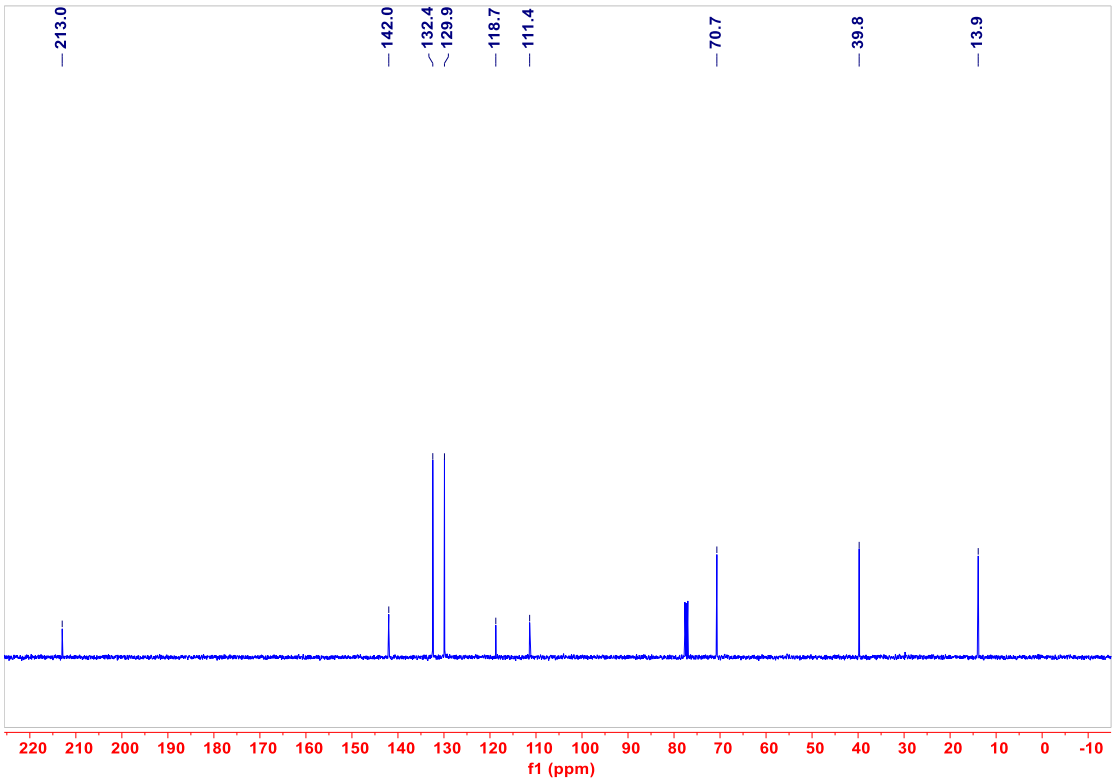

213.4
140.4
129.5
128.2
125.6
125.6
122.8
120.1
70.5
39.8
13.8
f1 (ppm)

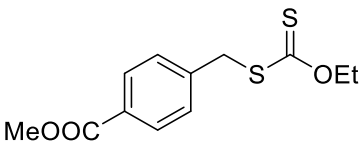

-62.4
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
f1 (ppm)


S
S
OEt
NC

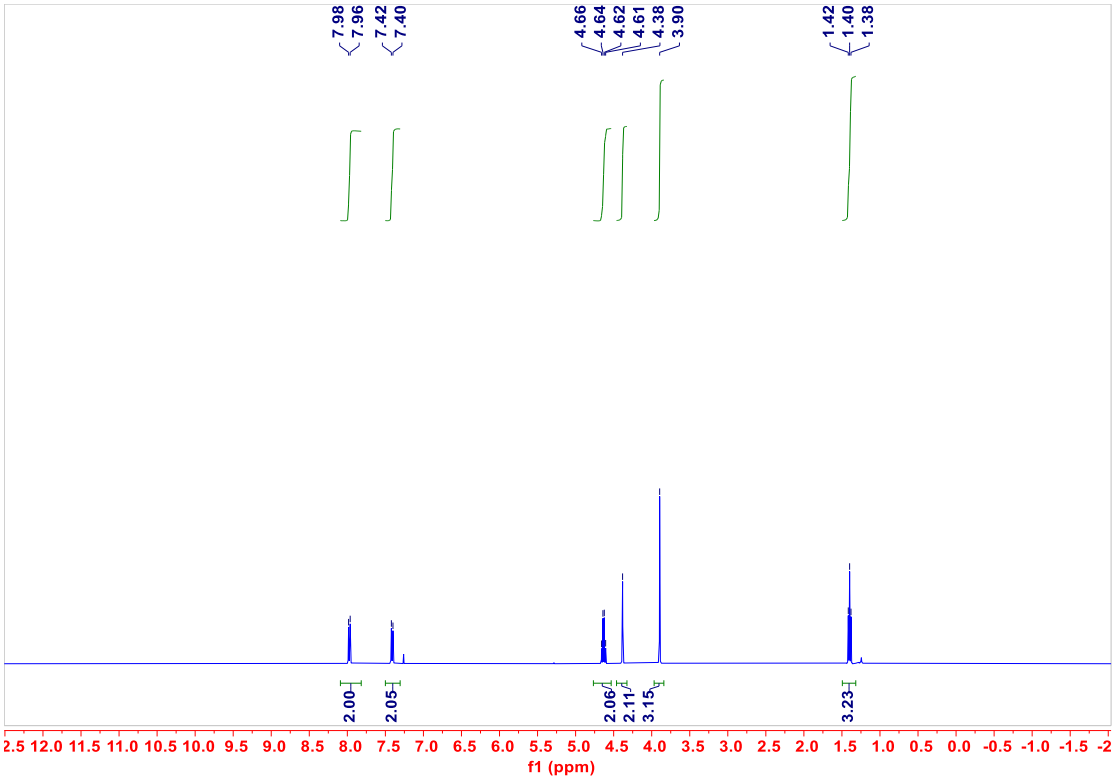
**4af**

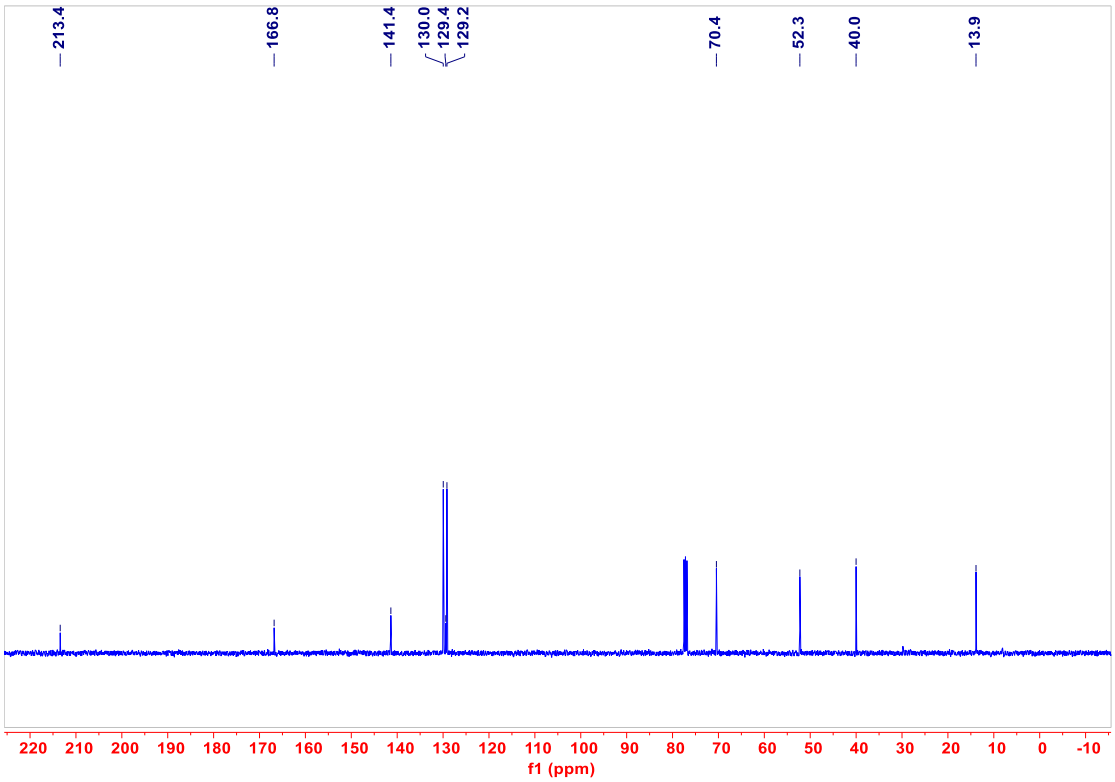

7.59
7.57
7.46
7.44
4.64
4.62
4.61
4.59
4.37
1.40
1.38
1.37
1.99
2.00
2.10
2.07
3.16
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

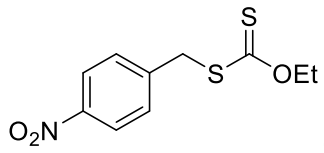

213.0
142.0
132.4
129.9
118.7
111.4
70.7
39.8
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt
MeOOC

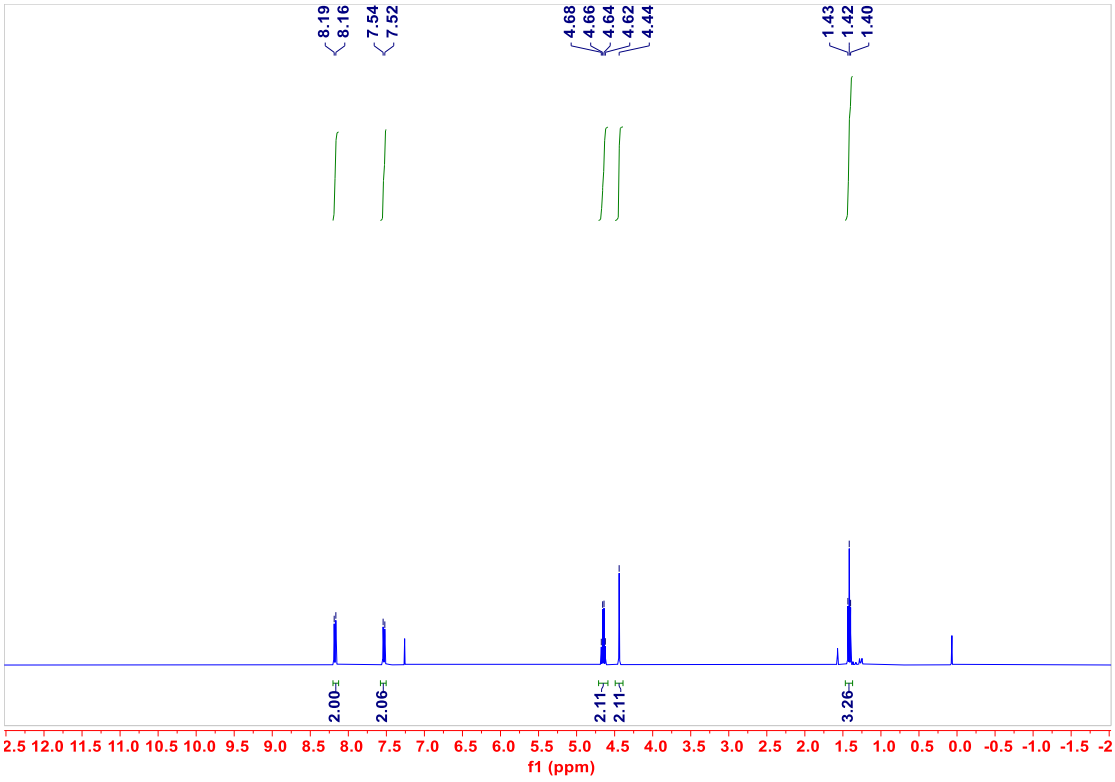
**4ag**

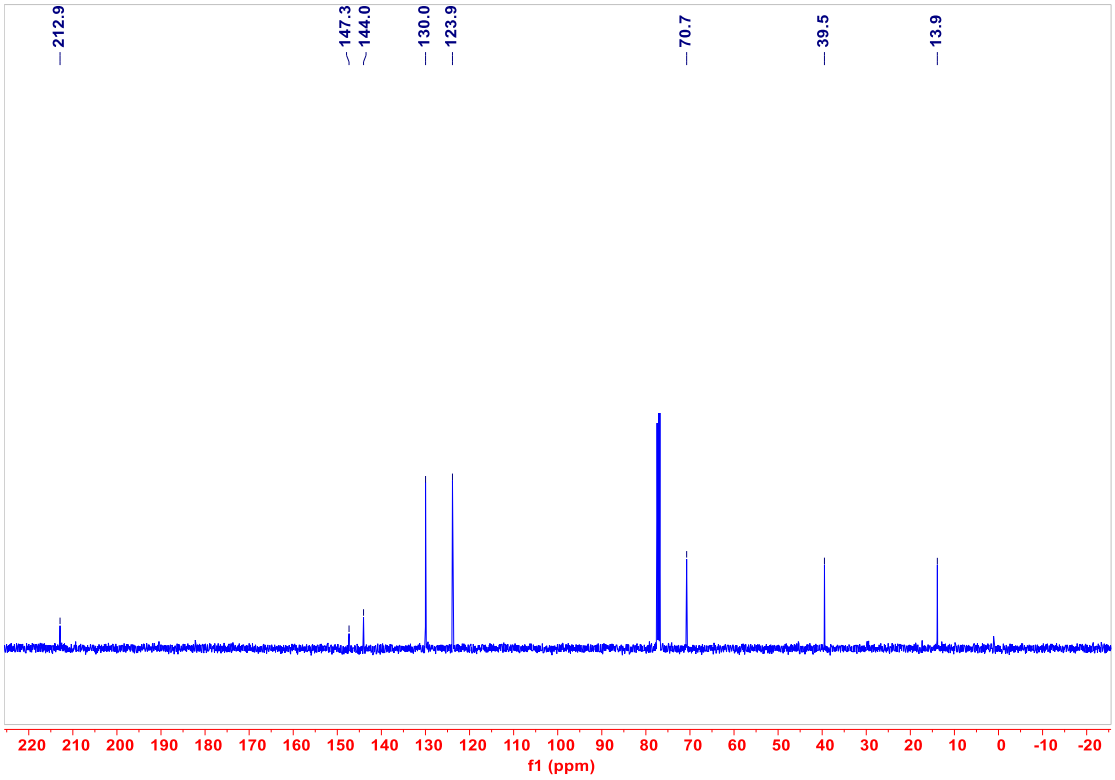

7.98
7.96
7.42
7.40
4.66
4.64
4.62
4.61
4.38
3.90
1.42
1.40
1.38
2.00
2.05
2.06
2.11
3.15
3.23
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

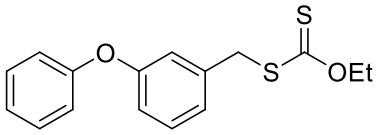

213.4
166.8
141.4
130.0
129.4
129.2
70.4
52.3
40.0
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt
O2N

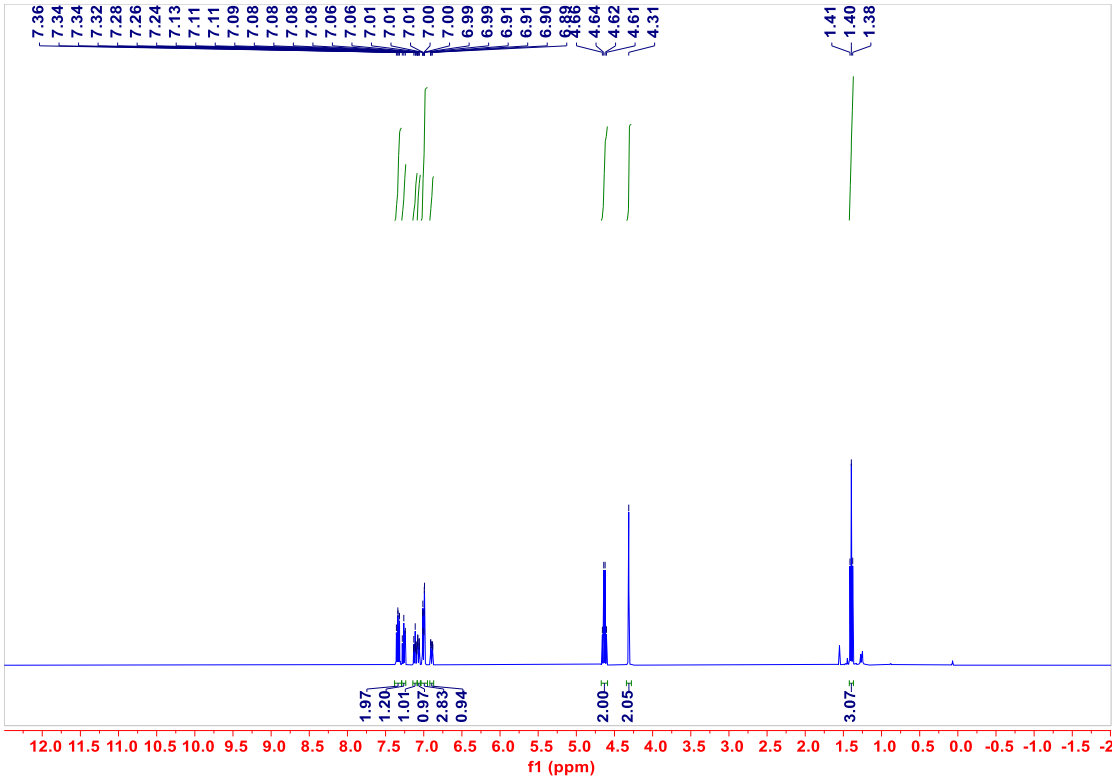
**4ah**

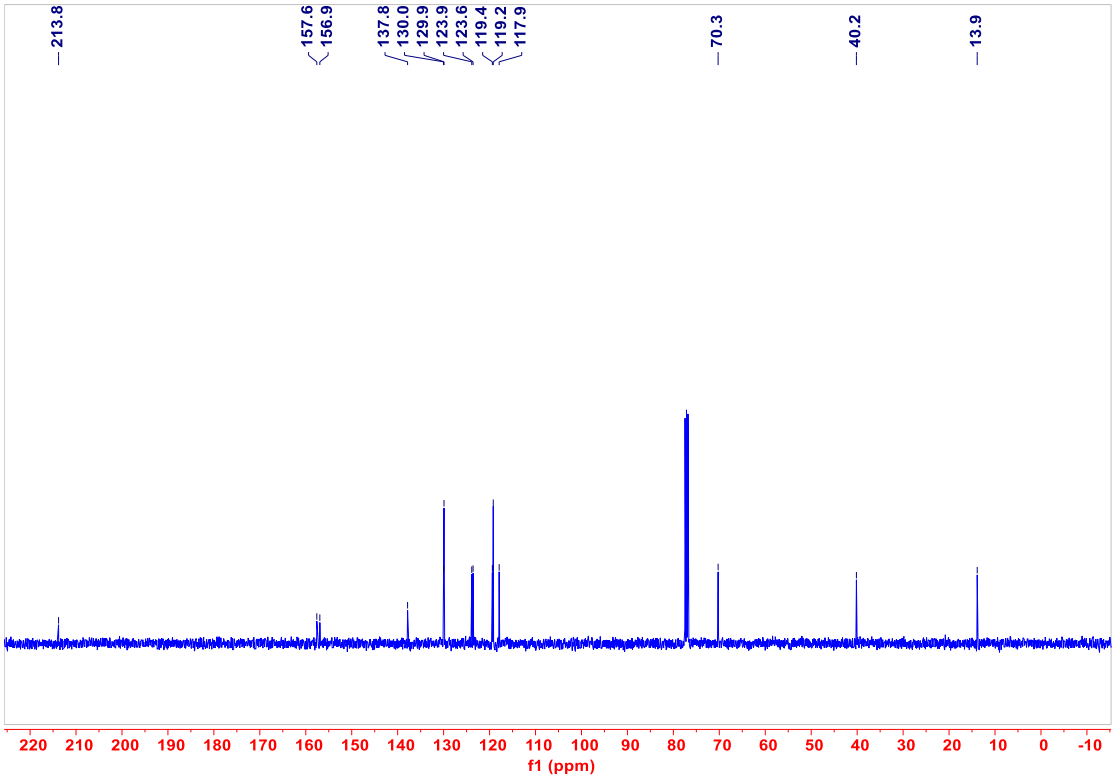

8.19
8.16
7.54
7.52
4.68
4.66
4.64
4.62
4.44
1.43
1.42
1.40
2.00
2.06
2.11
2.11
3.26
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

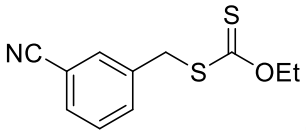

212.9
147.3
144.0
130.0
123.9
70.7
39.5
13.9
f1 (ppm)


S
O
S
OEt

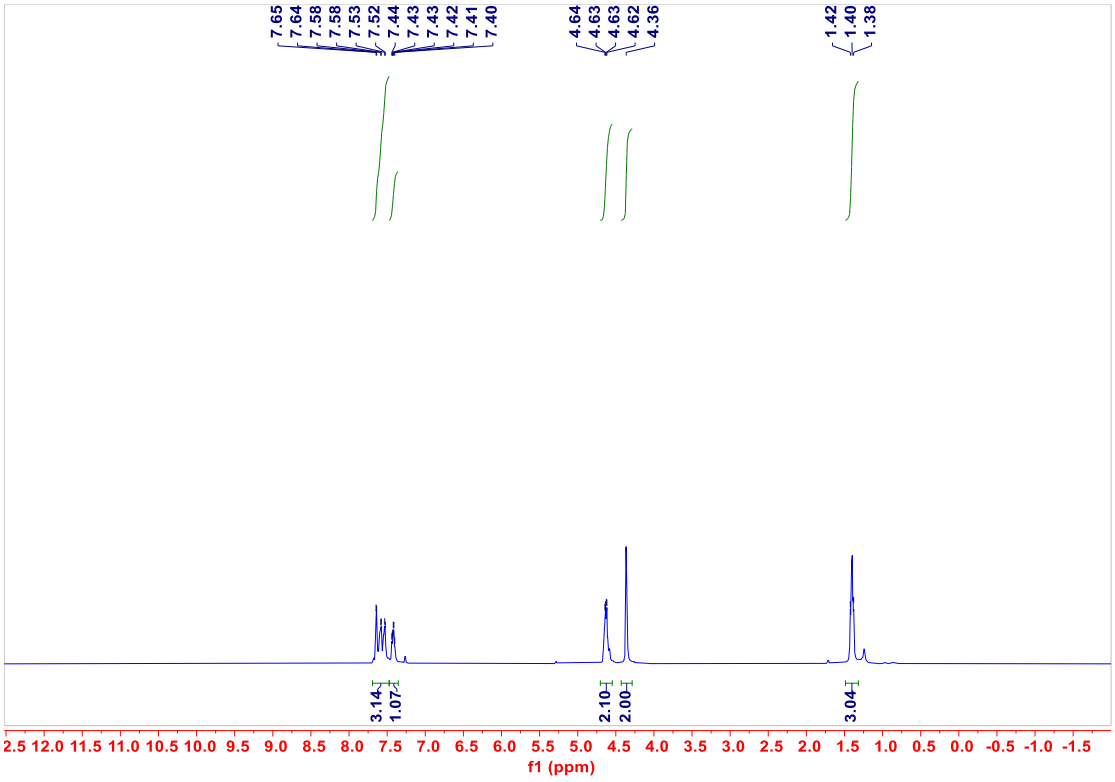
**4ai**

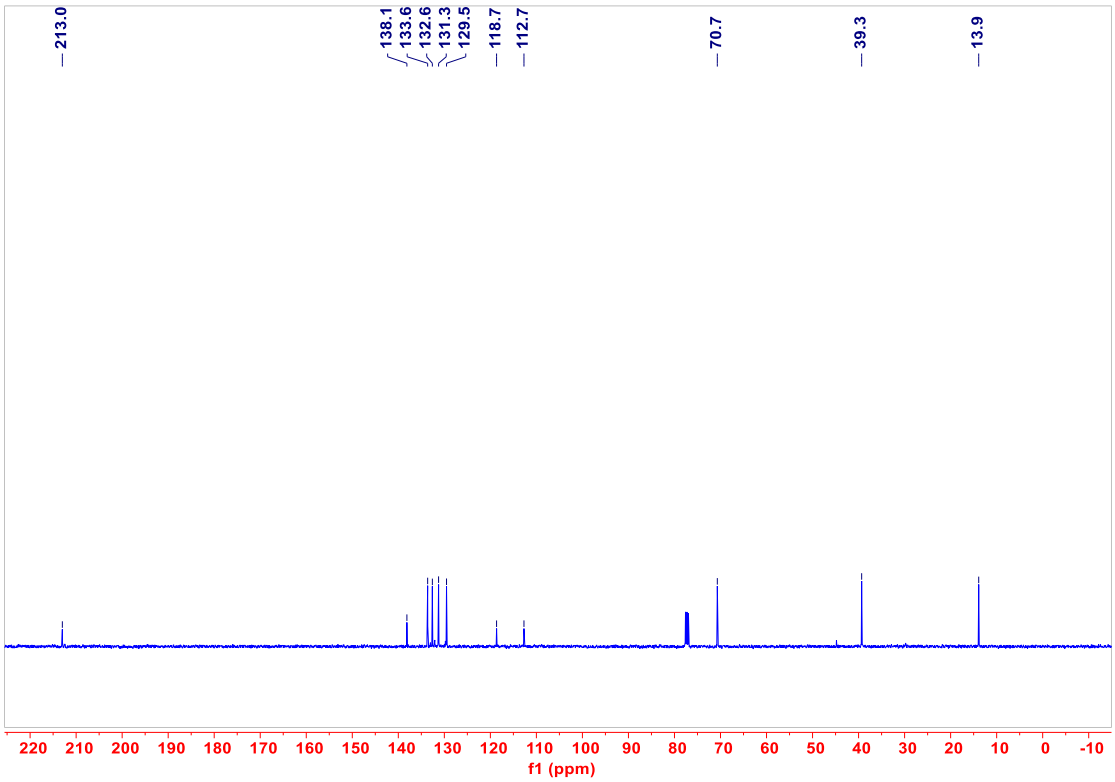

7.36
7.34
7.34
7.32
7.28
7.26
7.24
7.13
7.11
7.11
7.09
7.08
7.08
7.08
7.08
7.06
7.06
7.01
7.01
7.01
7.00
7.00
6.99
6.99
6.91
6.90
4.64
4.62
4.61
4.31
1.41
1.40
1.38
1.97
1.20
1.01
0.97
2.83
0.94
2.00
2.05
3.07
f1 (ppm)

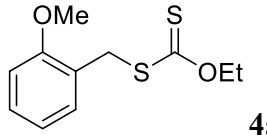

213.8
157.6
156.9
137.8
130.0
129.9
123.9
123.6
119.4
119.2
117.9
70.3
40.2
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


NC
S
S
OEt

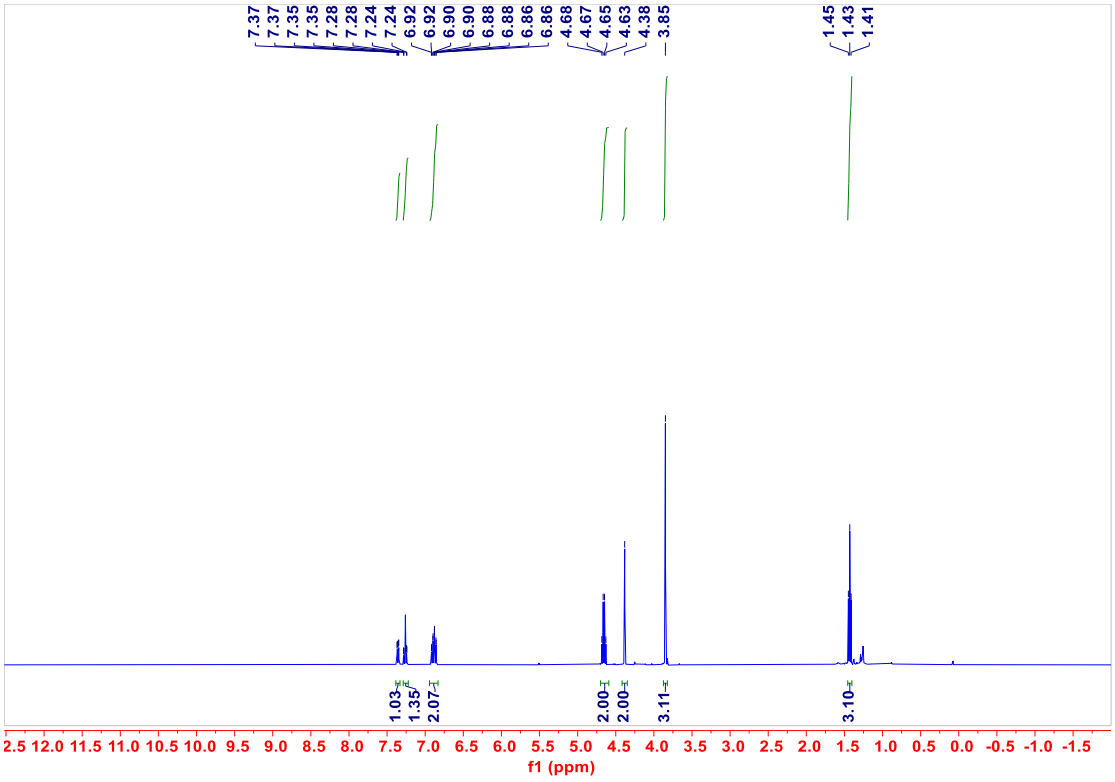
**4aj**

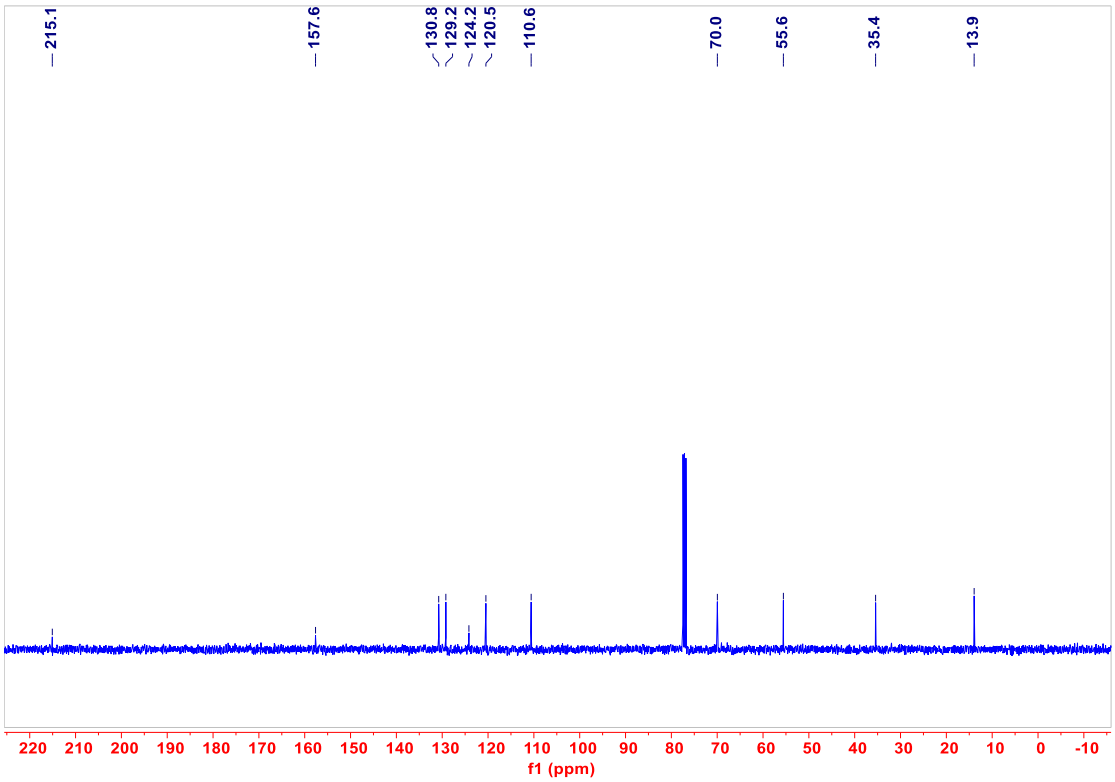

7.65
7.64
7.58
7.58
7.53
7.52
7.44
7.43
7.43
7.42
7.41
7.40
4.64
4.63
4.63
4.62
4.36
1.42
1.40
1.38
3.14
1.07
2.10
2.00
3.04
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

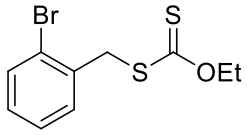

213.0
138.1
133.6
132.6
131.3
129.5
118.7
112.7
70.7
39.3
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
S
S
OEt

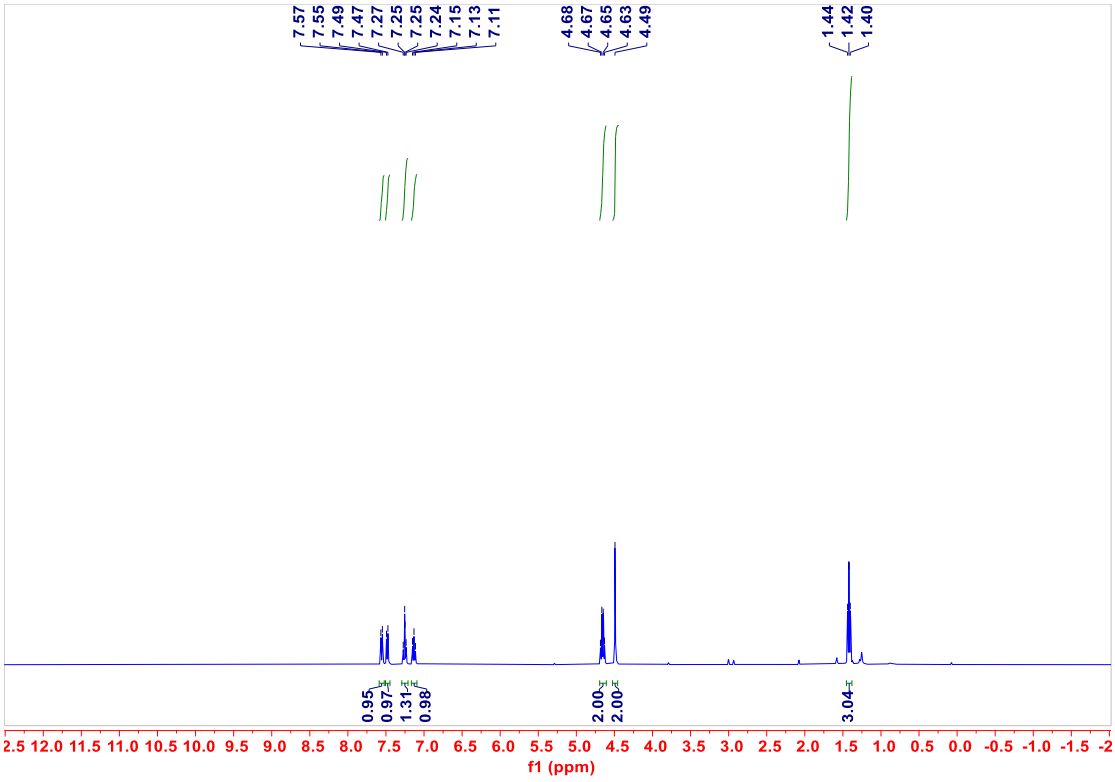
**4ak**


7.37
7.37
7.35
7.35
7.28
7.28
7.24
7.24
6.92
6.92
6.90
6.90
6.88
6.88
6.86
6.86
4.68
4.67
4.65
4.63
4.38
3.85
1.45
1.43
1.41
1.03
1.35
2.07
2.00
2.00
3.11
3.10
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


215.1
157.6
130.8
129.2
124.2
120.5
110.6
70.0
55.6
35.4
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Br
S
S
OEt

**4al**


7.57
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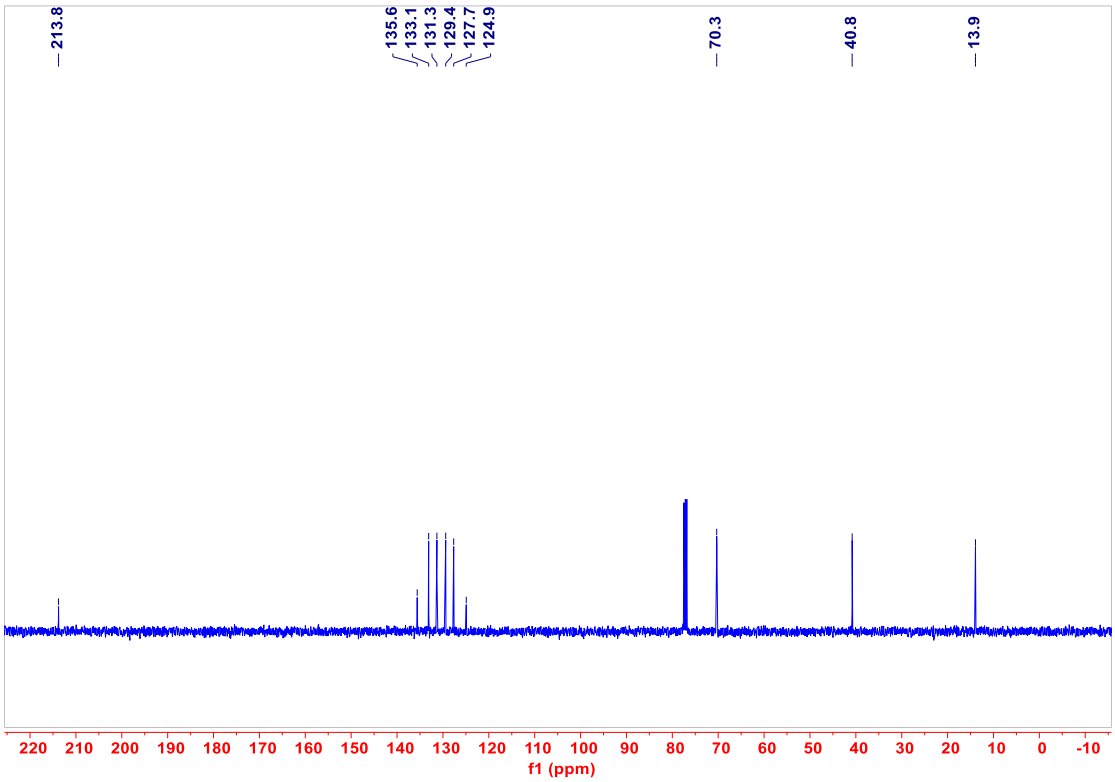
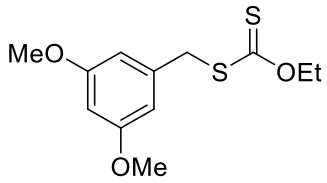
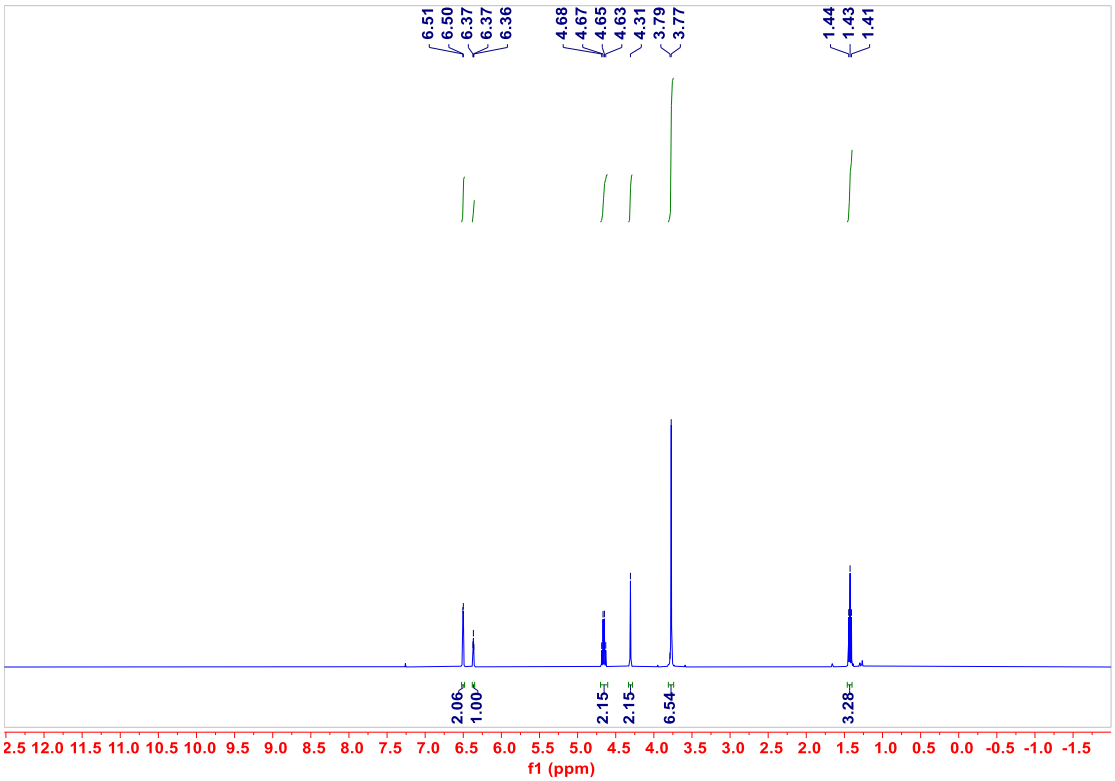
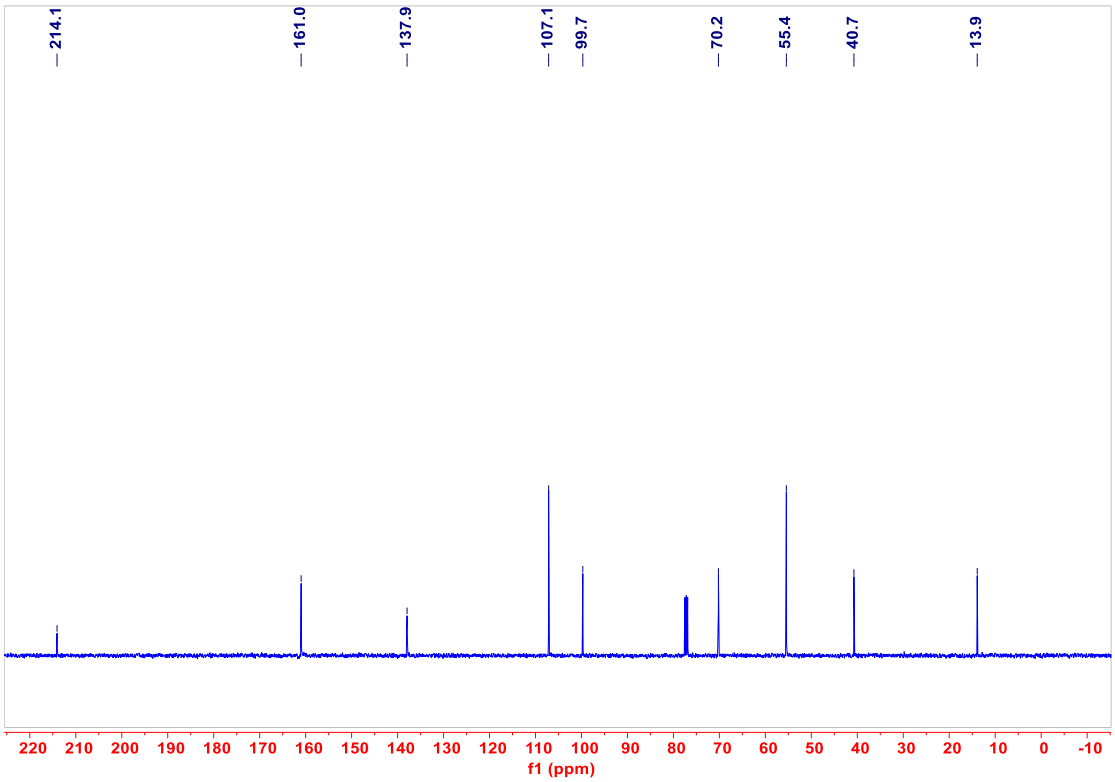
7.49
7.47
7.27
7.25
7.25
7.24
7.15
7.13
7.11
4.68
4.67
4.65
4.63
4.49
1.44
1.42
1.40
0.95
0.97
1.31
0.98
2.00
2.00
3.04
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

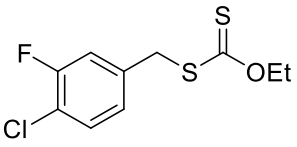

213.8
135.6
133.1
131.3
129.4
127.7
124.9
70.3
40.8
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


MeO
S
S
OEt
OMe

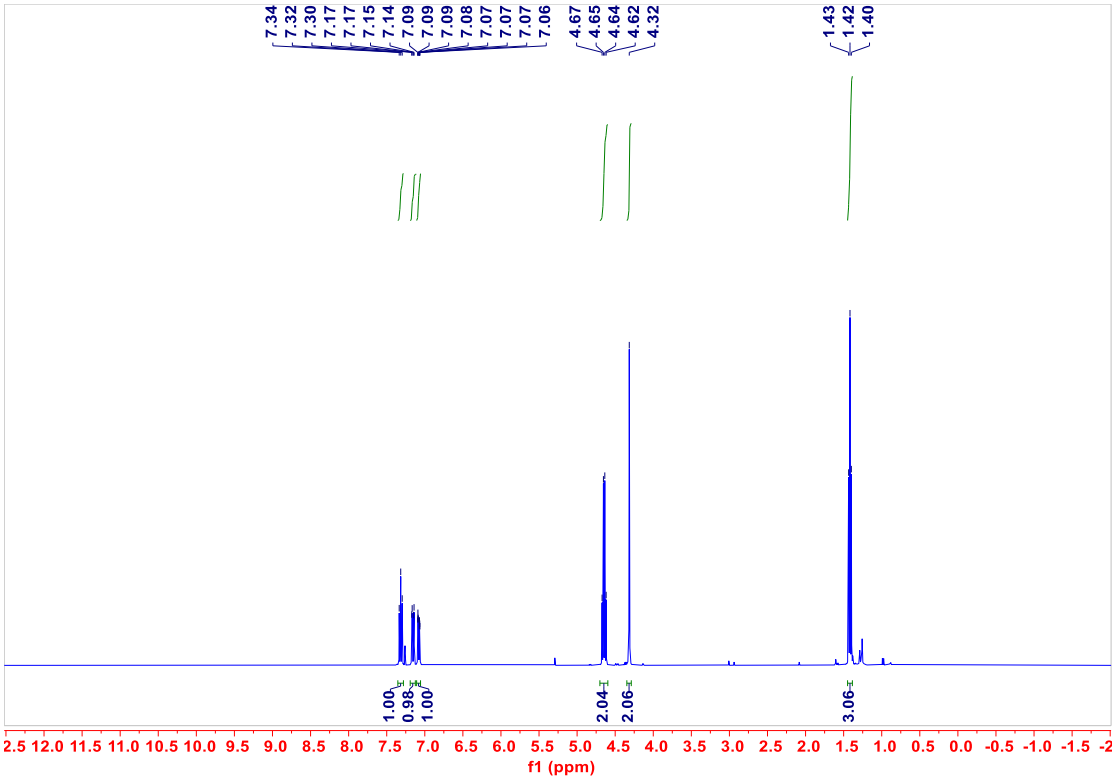
**4am**

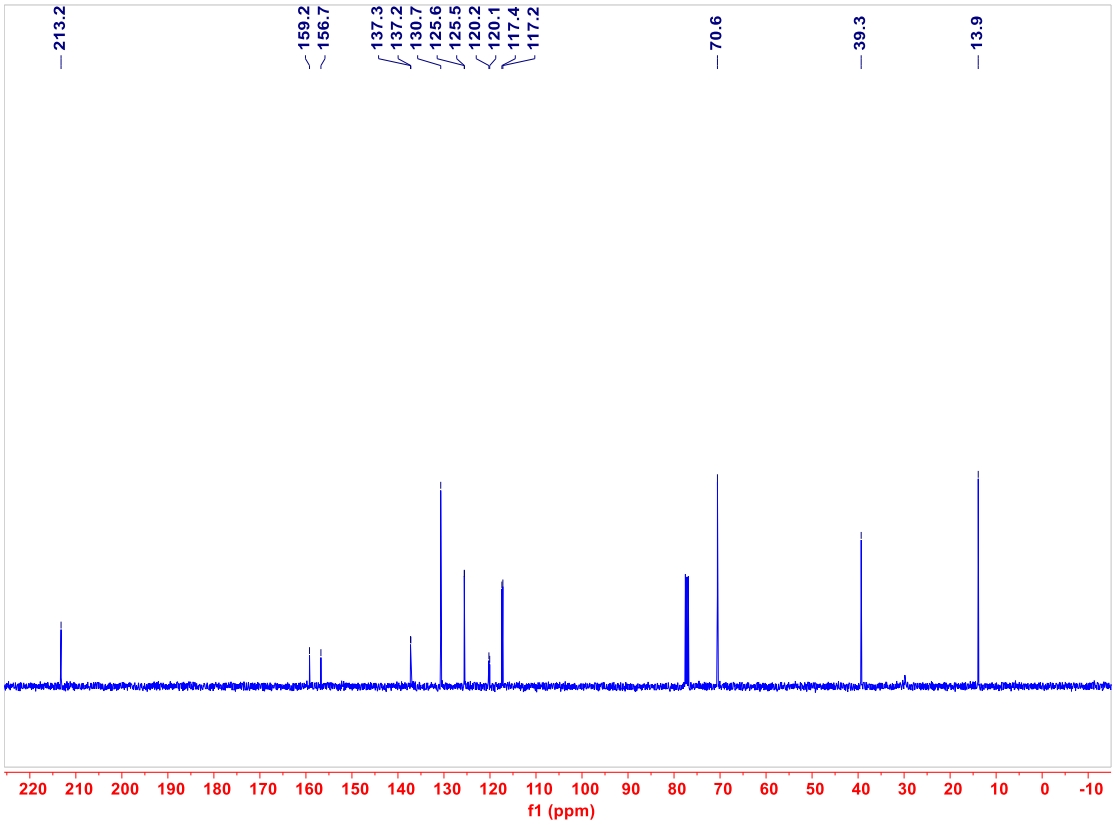

6.51
6.50
6.37
6.37
6.36
4.68
4.67
4.65
4.63
4.31
3.79
3.77
1.44
1.43
1.41
2.06
1.00
2.15
2.15
6.54
3.28
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

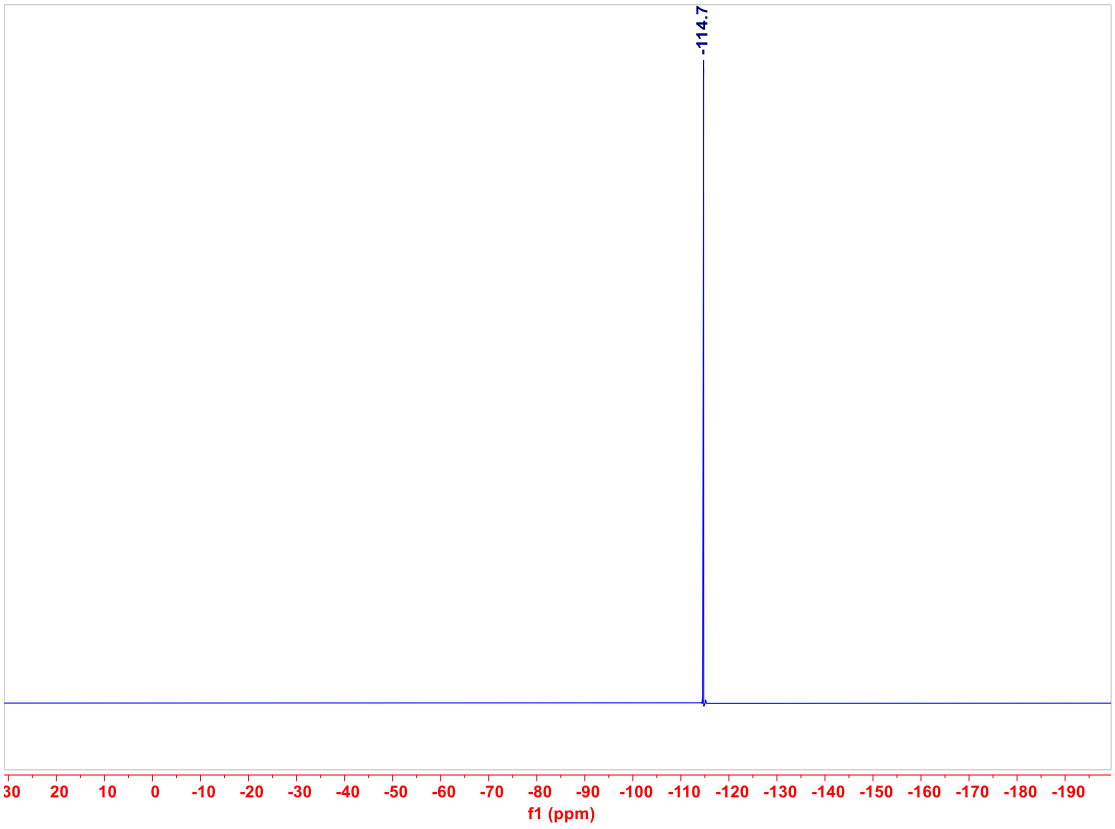

214.1
161.0
137.9
107.1
99.7
70.2
55.4
40.7
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

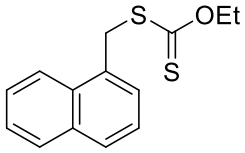

S
F
S
OEt
Cl

**4an**

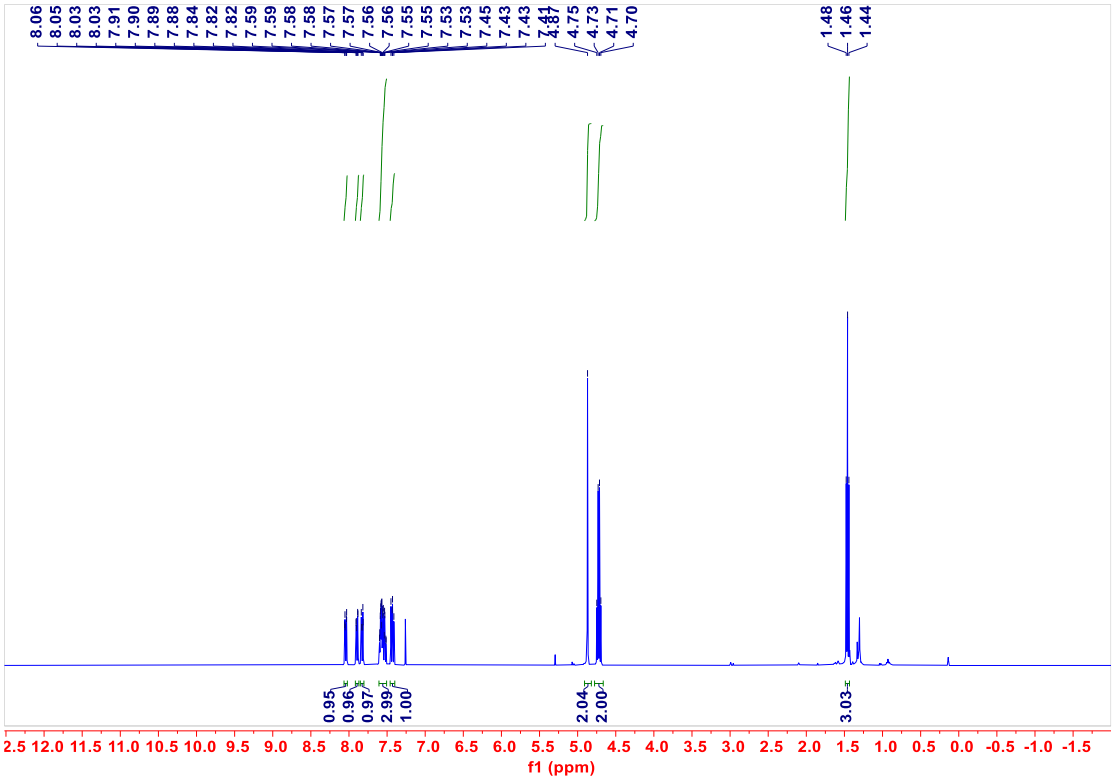

7.34
7.32
7.30
7.17
7.17
7.15
7.14
7.09
7.09
7.09
7.08
7.07
7.07
7.07
7.06
4.67
4.65
4.64
4.62
4.32
1.43
1.42
1.40
1.00
0.98
1.00
2.04
2.06
3.06
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

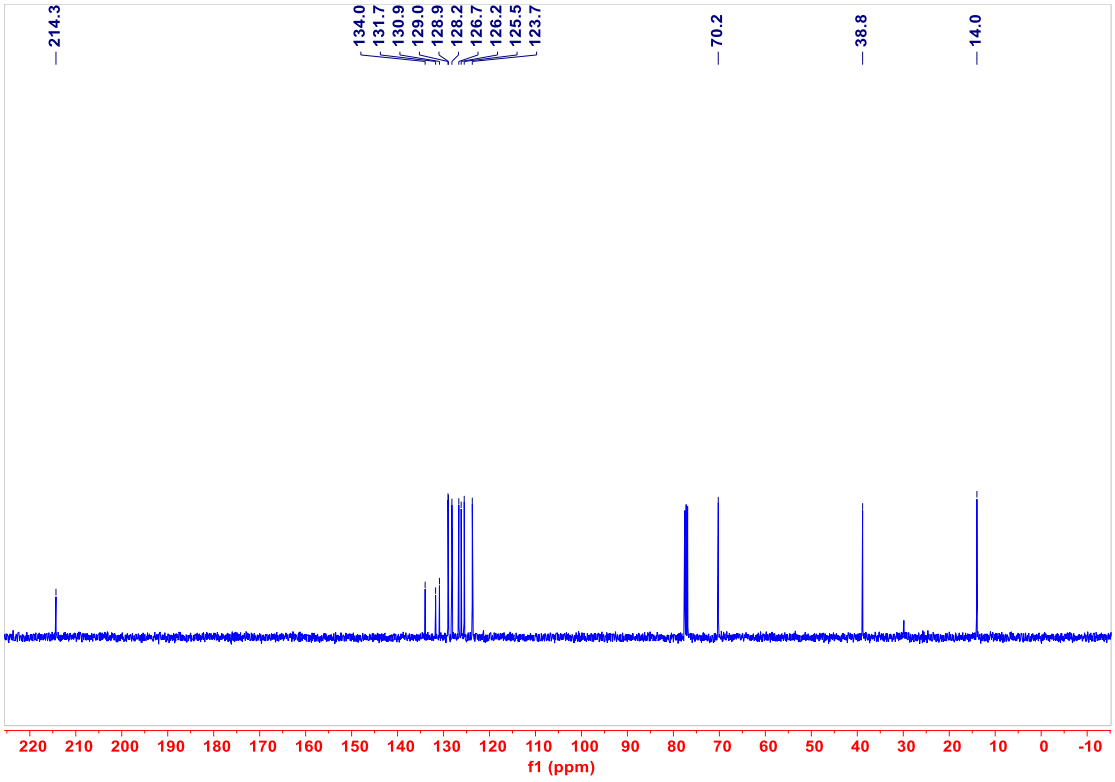

213.2
159.2
156.7
137.3
137.2
130.7
125.6
125.5
120.2
120.1
117.4
117.2
70.6
39.3
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

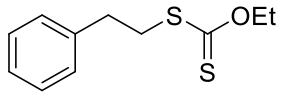

-114.7
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
f1 (ppm)


S
OEt
S

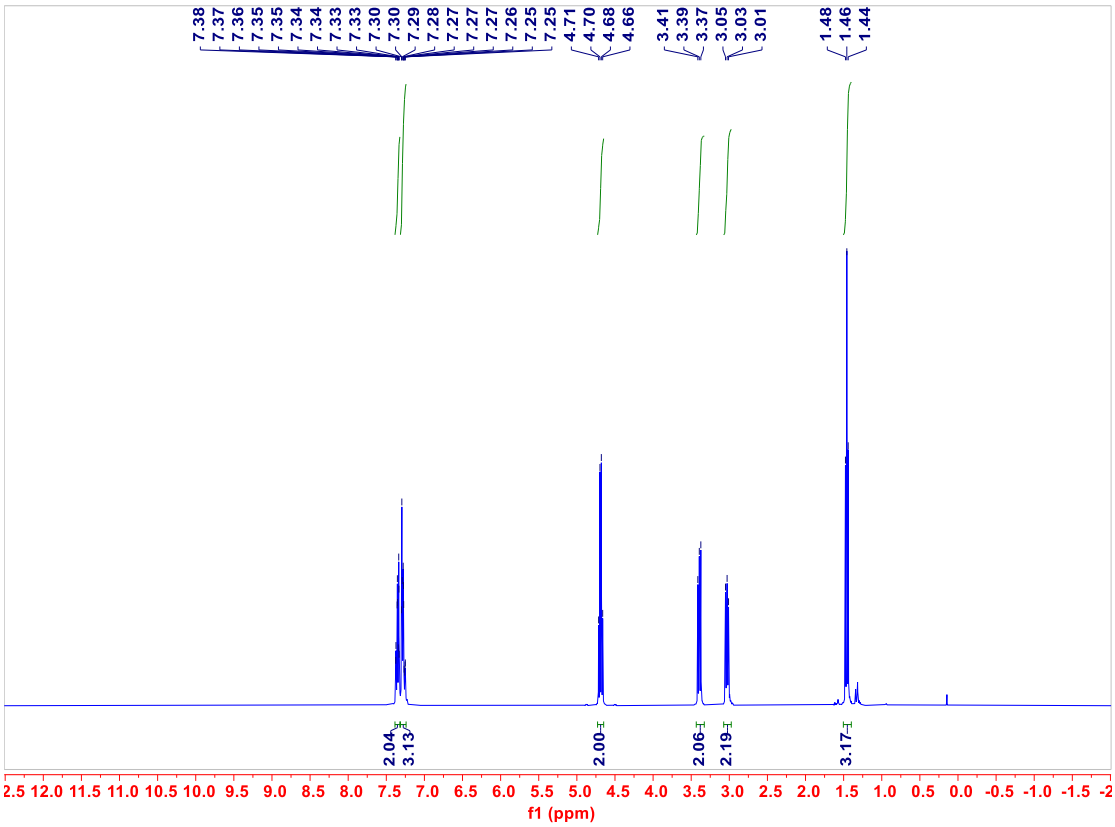
**4ao**

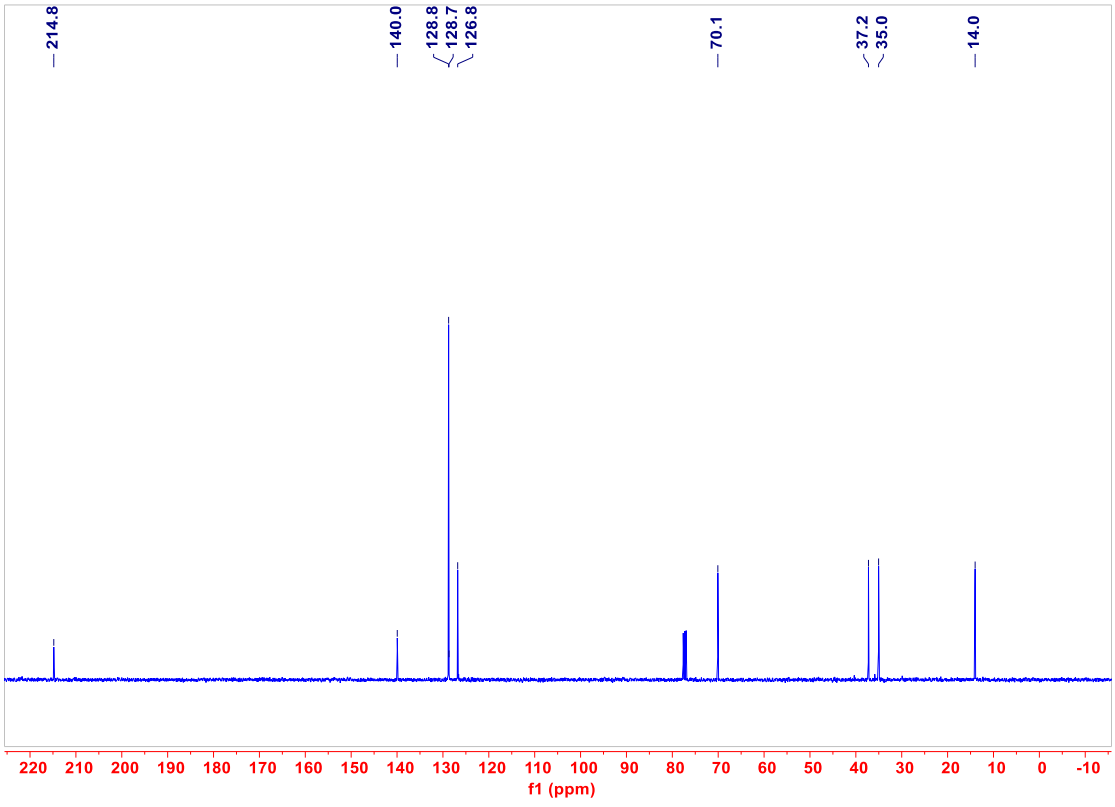

8.06 8.05 8.03 8.03 7.91 7.90 7.89 7.88 7.84 7.82 7.82 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.56 7.55 7.55 7.53 7.53 7.45 7.43 7.43 4.75 4.73 4.71 4.70
1.48 1.46 1.44
0.95 0.96 0.97 2.99 1.00 2.04 2.00 3.03
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

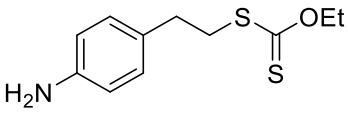

214.3
134.0 131.7 130.9 129.0 128.9 128.2 126.7 126.2 125.5 123.7
70.2
38.8
14.0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
OEt
S

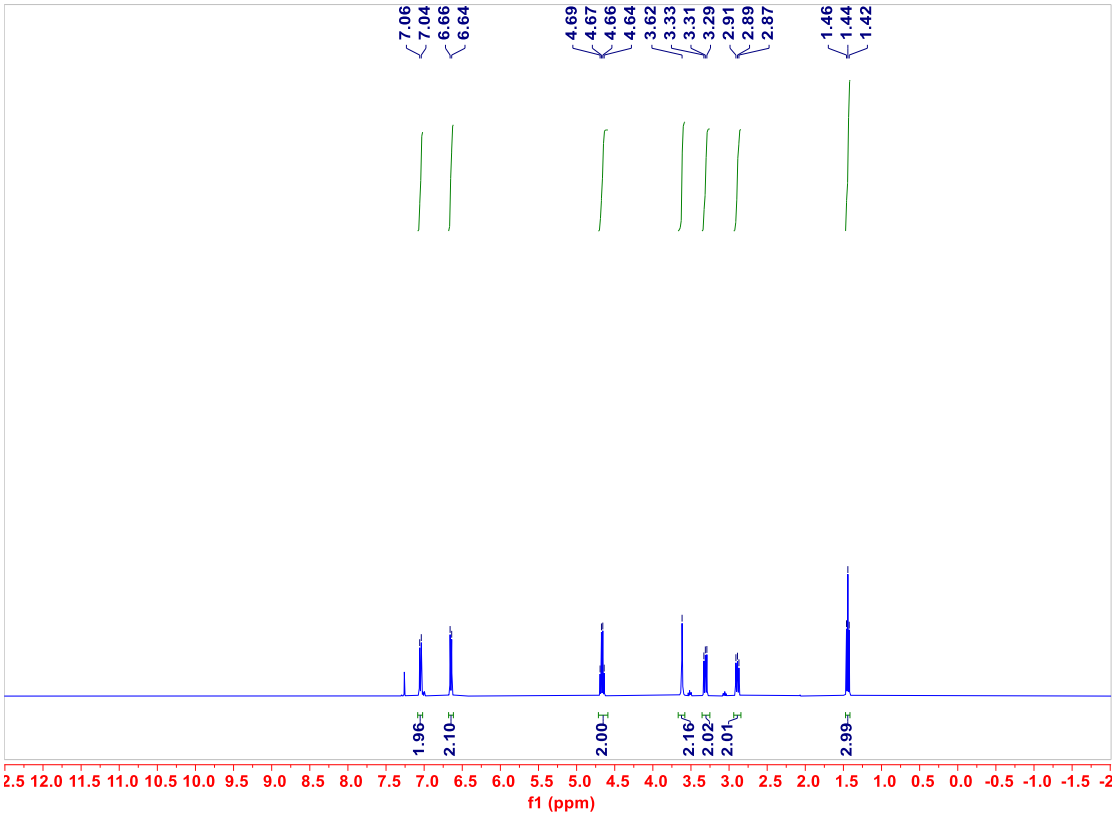
**4ap**


7.38 7.37 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.30 7.30 7.29 7.28 7.27 7.27 7.27 7.26 7.25 7.25
4.71 4.70 4.68 4.66
3.41 3.39 3.37 3.05 3.03 3.01
1.48 1.46 1.44
2.04 3.13 2.00 2.06 2.19 3.17
f1 (ppm)


214.8
140.0
128.8 128.7 126.8
70.1
37.2 35.0
14.0
f1 (ppm)


S
OEt
S
$H_2N$

**4aq**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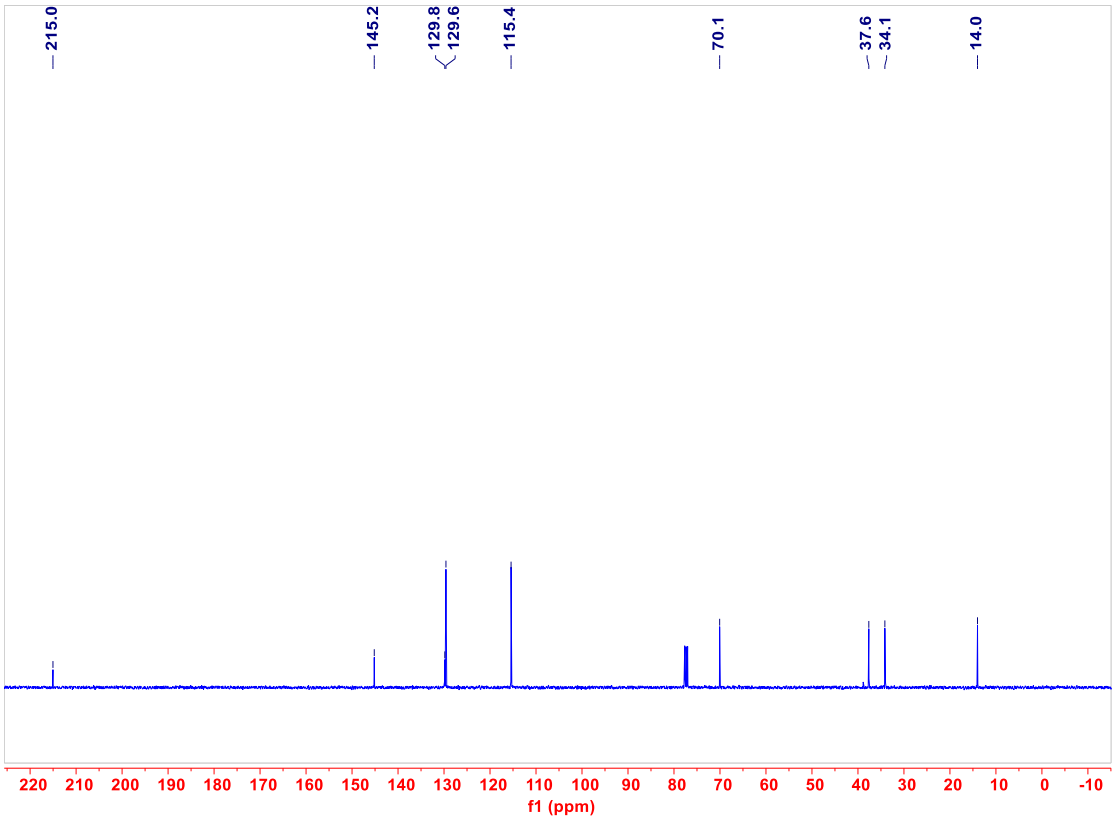
7.04
6.66
6.64
4.69
4.67
4.66
4.64
3.62
3.33
3.31
3.29
2.91
2.89
2.87
1.46
1.44
1.42
1.96
2.10
2.00
2.16
2.02
2.01
2.99
f1 (ppm)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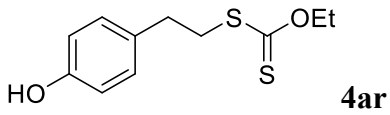
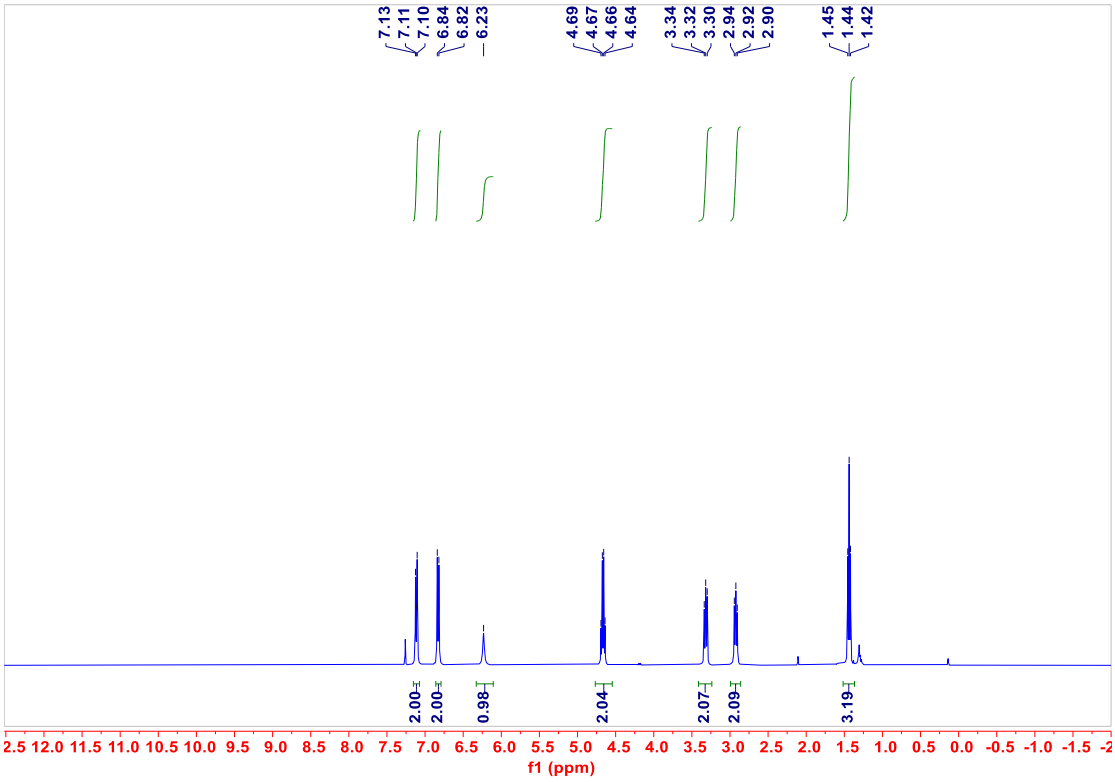
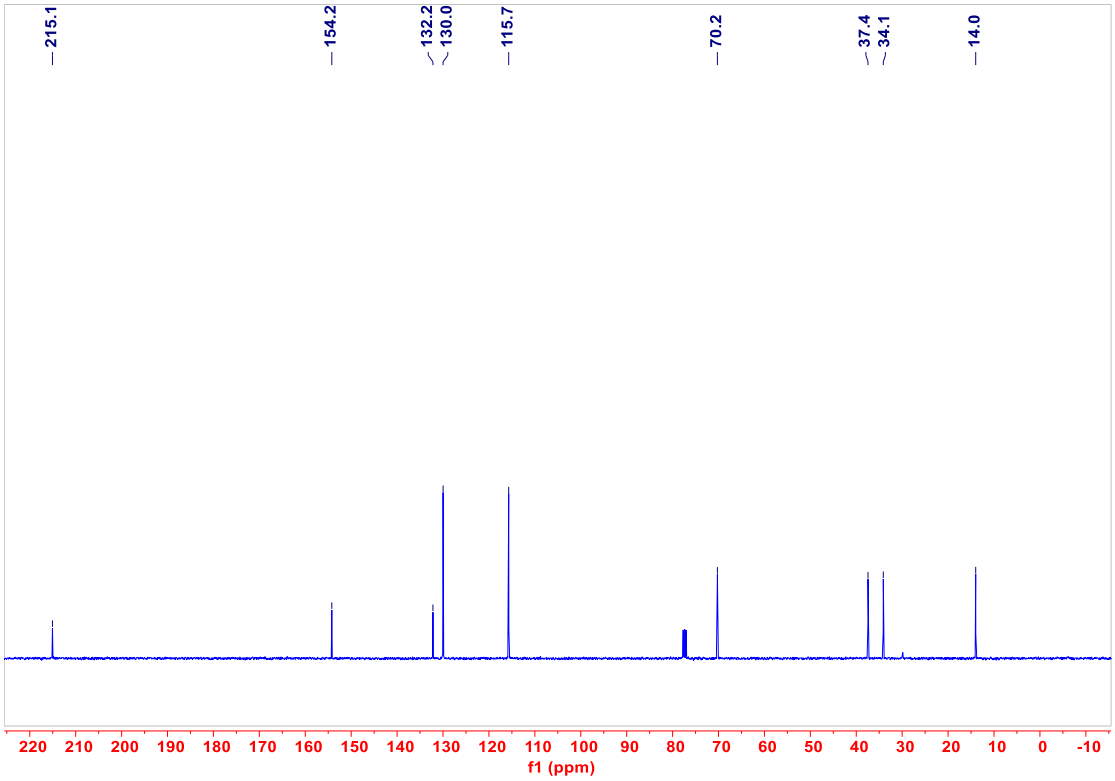
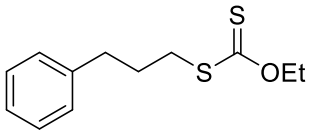
145.2
129.8
129.6
115.4
70.1
37.6
34.1
14.0
f1 (ppm)


S
OEt
S
HO

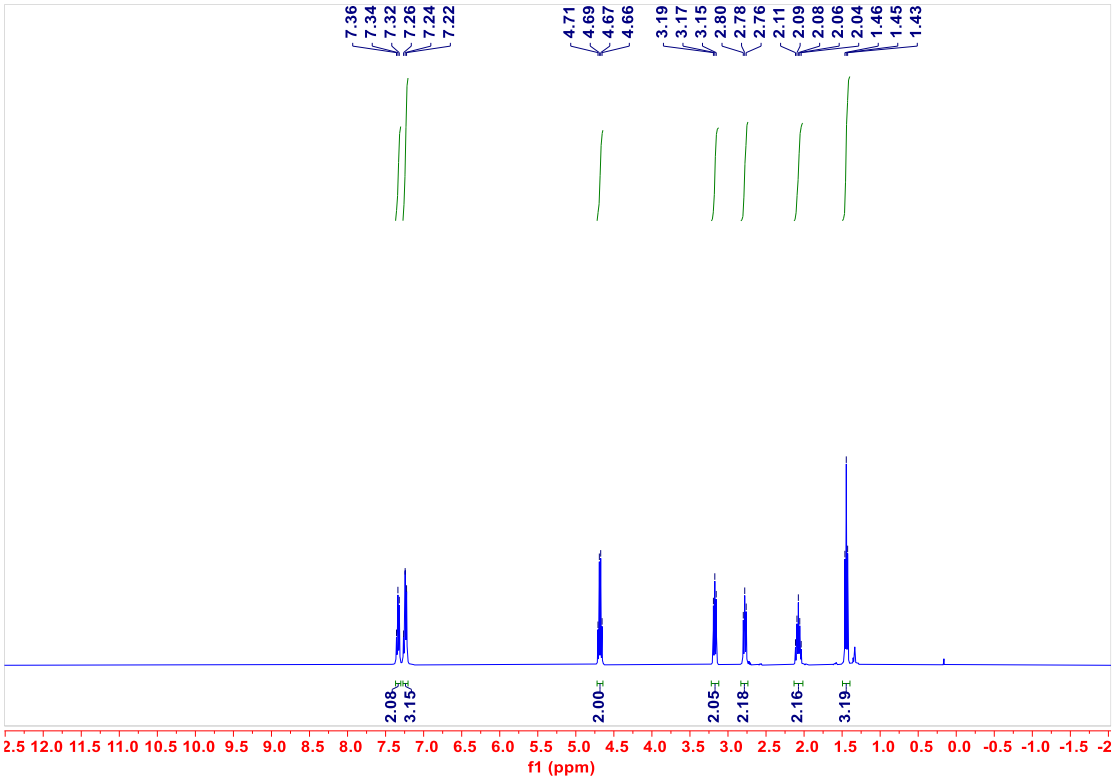
**4ar**


7.13
7.11
7.10
6.84
6.82
6.23
4.69
4.67
4.66
4.64
3.34
3.32
3.30
2.94
2.92
2.90
1.45
1.44
1.42
2.00
2.00
0.98
2.04
2.07
2.09
3.19
f1 (ppm)


215.1
154.2
132.2
130.0
115.7
70.2
37.4
34.1
14.0
f1 (ppm)


S
S
OEt

**4as**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7.34
7.32
7.26
7.24
7.22
4.71
4.69
4.67
4.66
3.19
3.17
3.15
2.80
2.78
2.76
2.11
2.09
2.08
2.06
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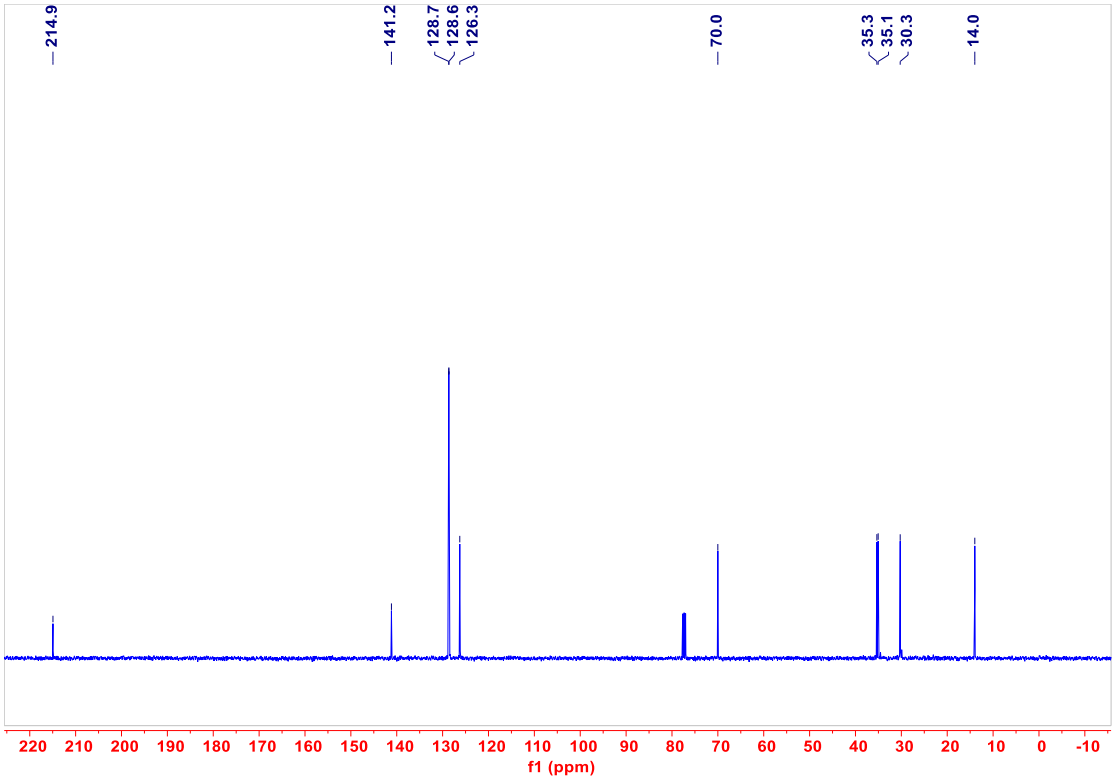
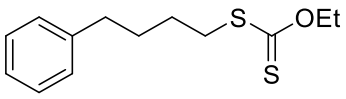
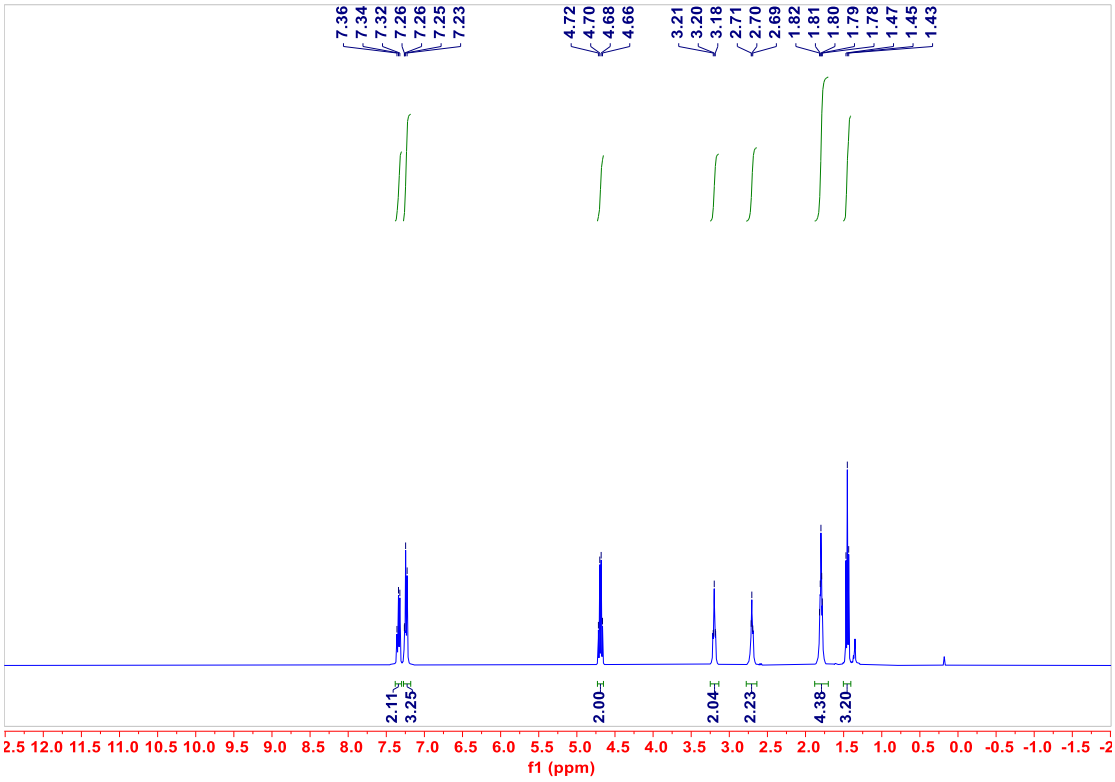
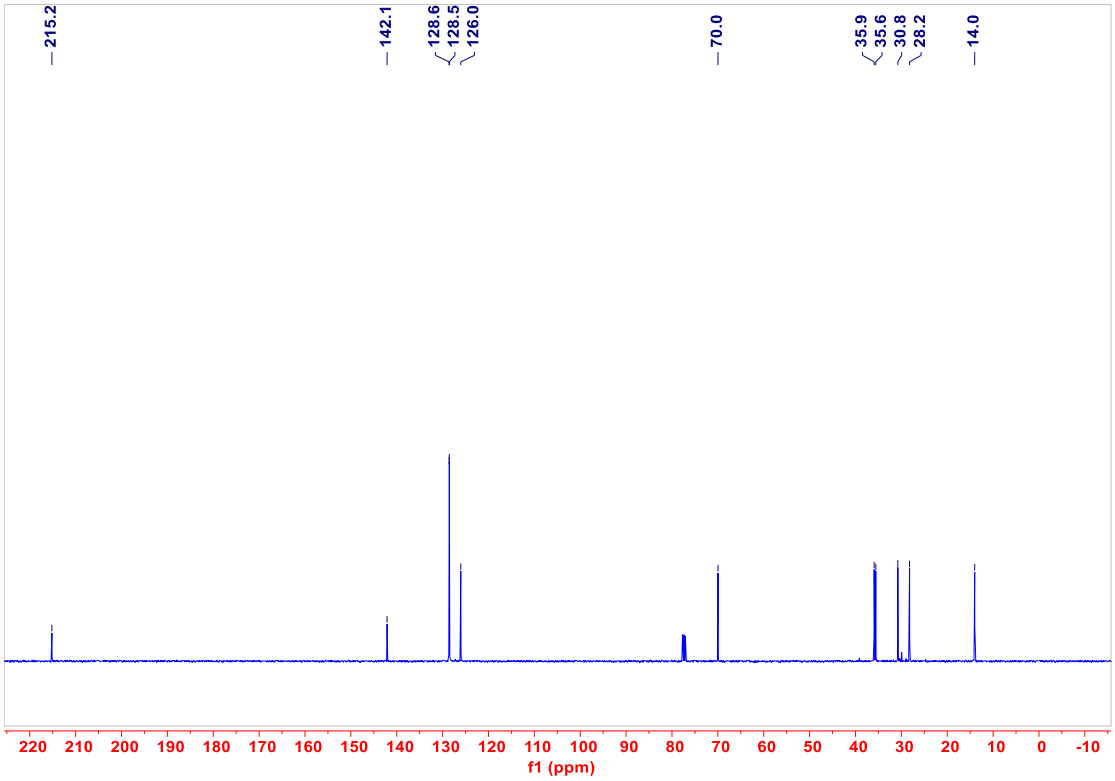
2.05
2.18
2.16
3.19
f1 (ppm)

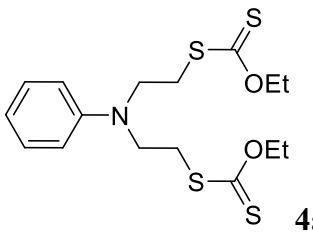

214.9
141.2
128.7
128.6
126.3
70.0
35.3
35.1
30.3
14.0
f1 (ppm)


S
OEt
S

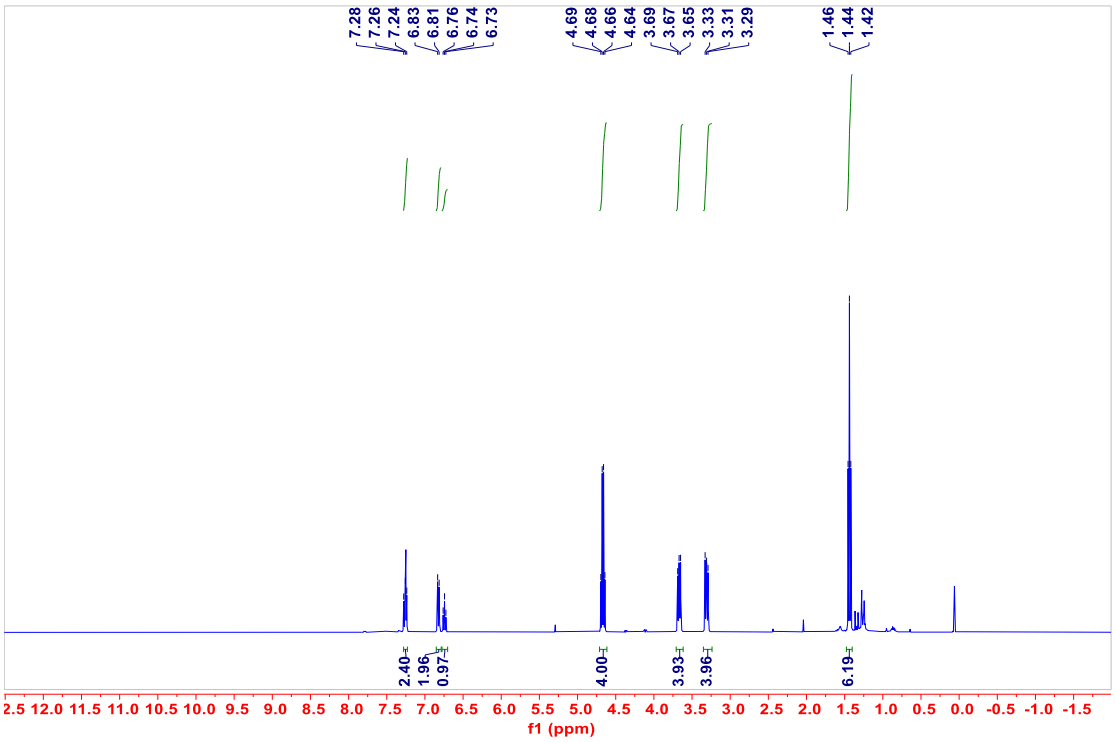
**4at**


7.36
7.34
7.32
7.26
7.26
7.25
7.23
4.72
4.70
4.68
4.66
3.21
3.20
3.18
2.71
2.70
2.69
1.82
1.81
1.80
1.79
1.78
1.47
1.45
1.43
2.11
3.25
2.00
2.04
2.23
4.38
3.20
f1 (ppm)


215.2
142.1
128.6
128.5
126.0
70.0
35.9
35.6
30.8
28.2
14.0
f1 (ppm)


S
S
OEt
N
OEt
S
S

**4au**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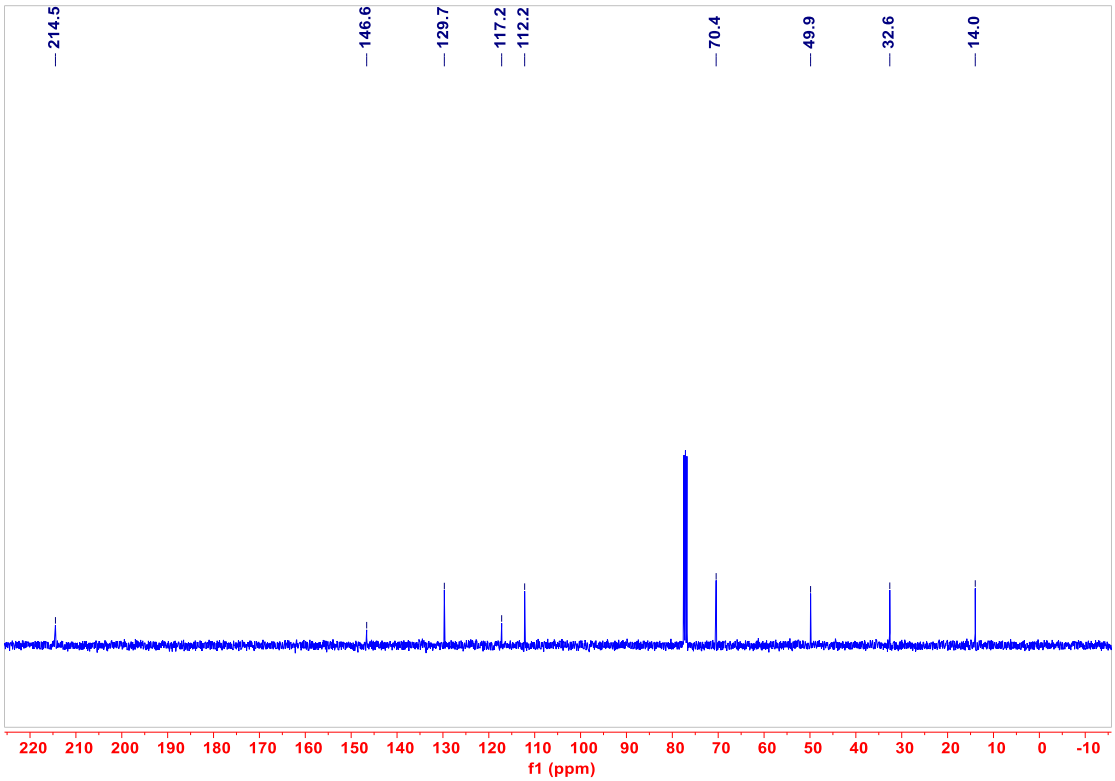
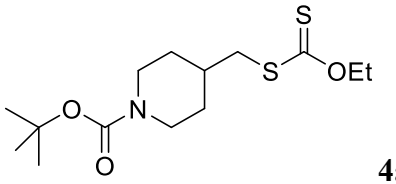
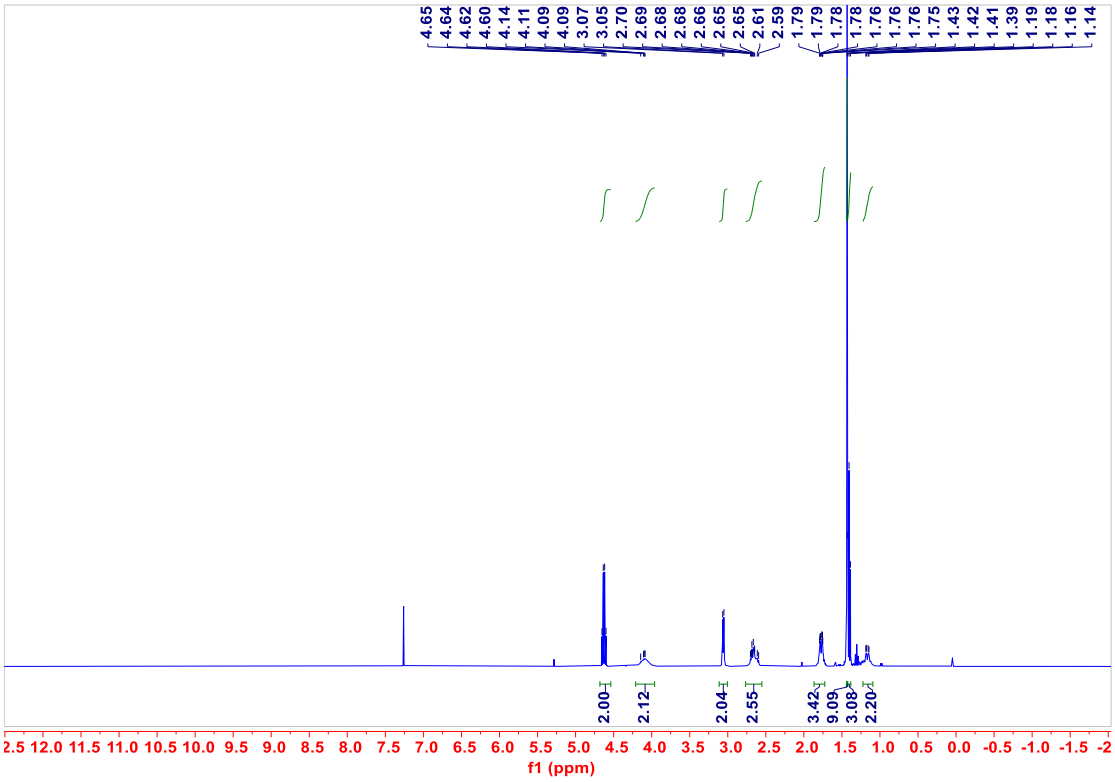
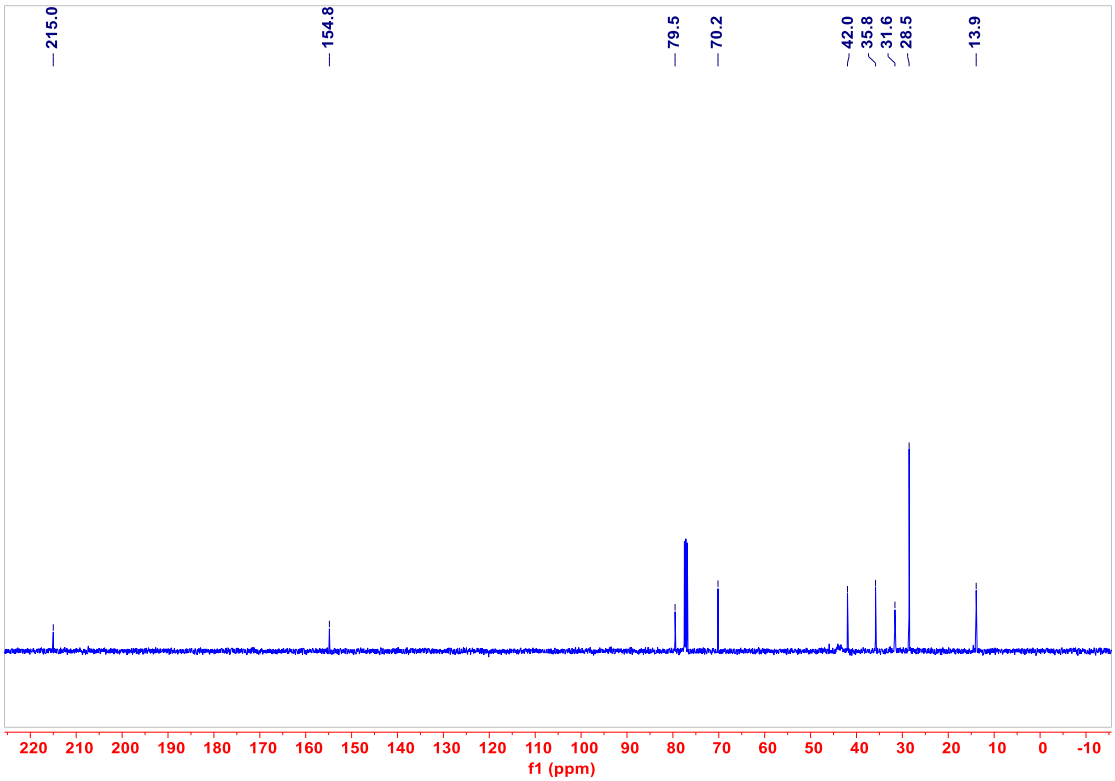
7.26
7.24
6.83
6.81
6.76
6.74
6.73
4.69
4.68
4.66
4.64
3.69
3.67
3.65
3.33
3.31
3.29
1.46
1.44
1.42
2.40
1.96
0.97
4.00
3.93
3.96
6.19
f1 (ppm)

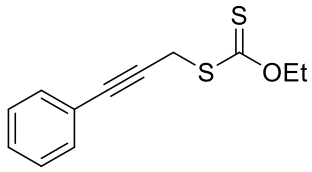

214.5
146.6
129.7
117.2
112.2
70.4
49.9
32.6
14.0
f1 (ppm)


S
S
OEt
O
N
O

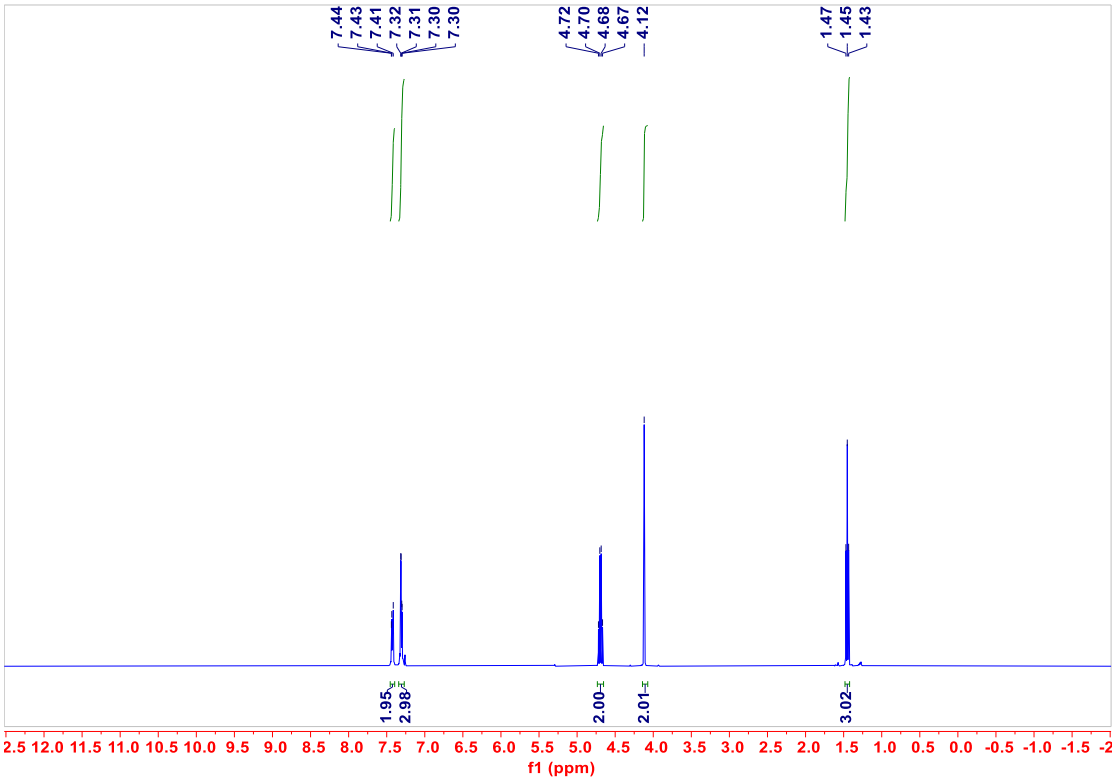
**4av**


4.65 4.64 4.62 4.60 4.14 4.11 4.09 4.09 3.07 3.05 2.70 2.69 2.68 2.68 2.66 2.65 2.65 2.61 2.59 1.79 1.79 1.78 1.78 1.76 1.76 1.76 1.75 1.43 1.42 1.41 1.39 1.19 1.18 1.16 1.14
2.00 2.12 2.04 2.55 3.42 9.09 3.08 2.20
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)


215.0
154.8
79.5
70.2
42.0
35.8
31.6
28.5
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt

**4aw**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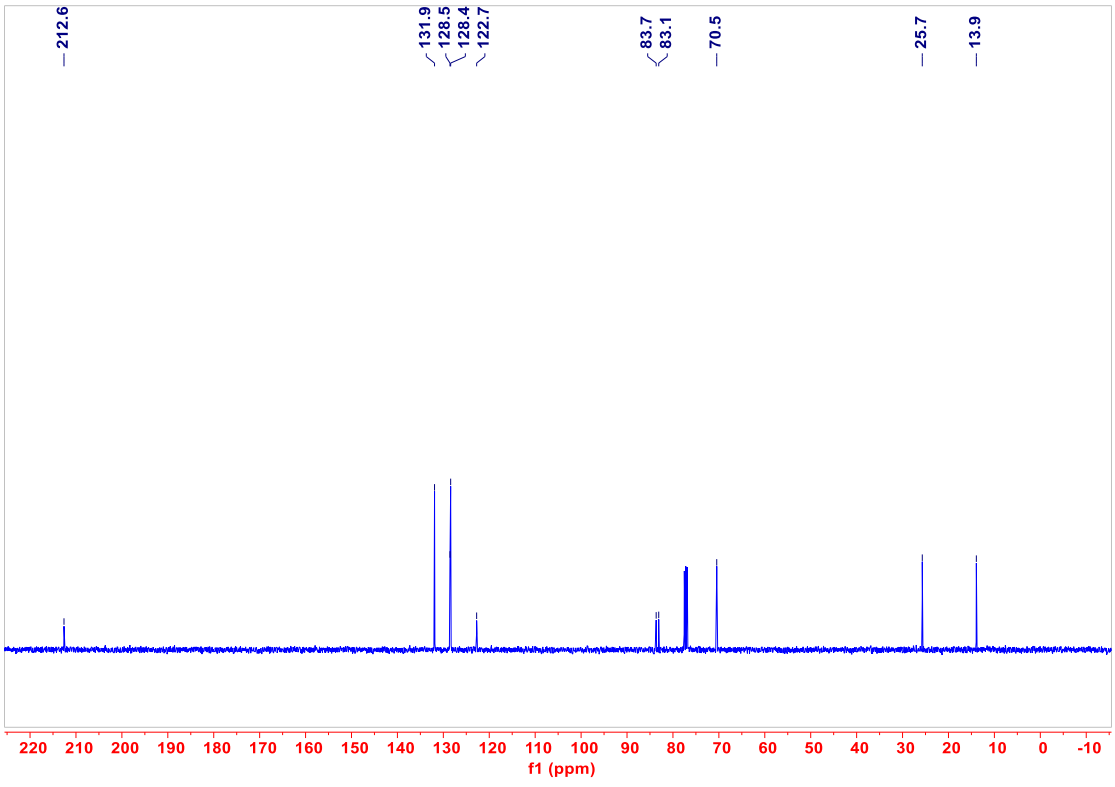
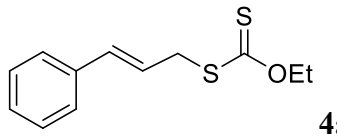
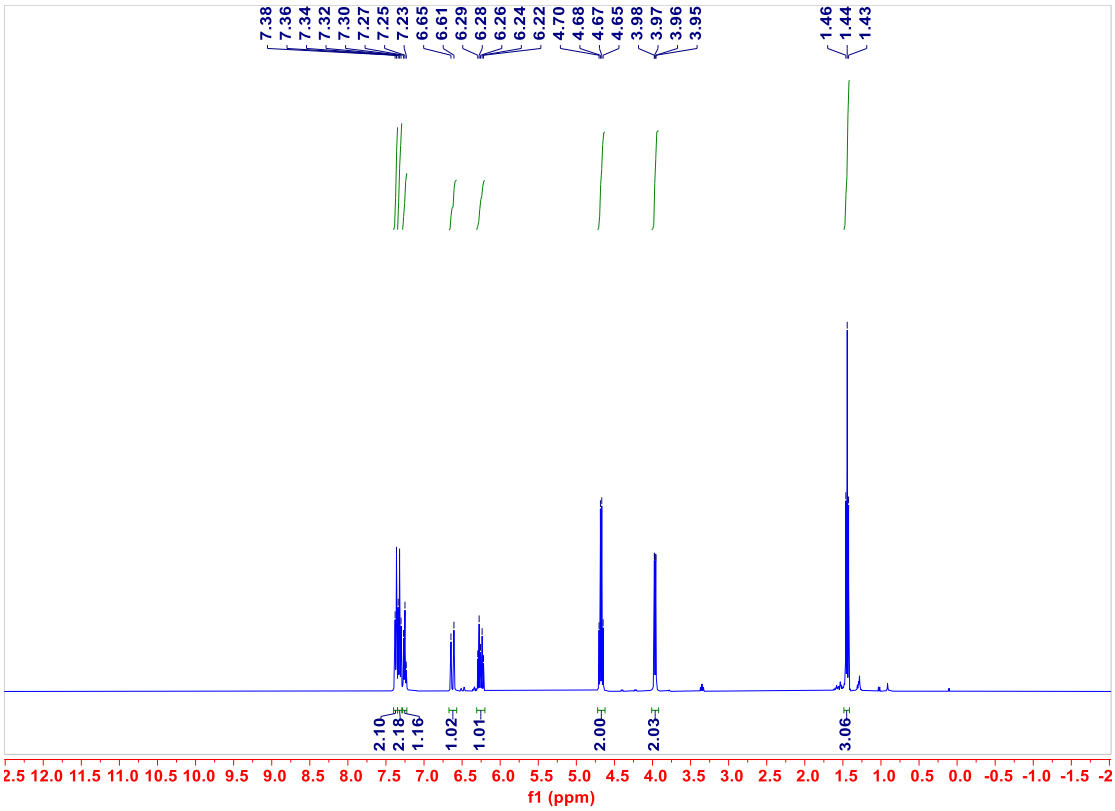
7.43
7.41
7.32
7.31
7.30
7.30
4.72
4.70
4.68
4.67
4.12
1.47
1.45
1.43
1.95
2.98
2.00
2.01
3.02
f1 (ppm)


212.6
131.9
128.5
128.4
122.7
83.7
83.1
70.5
25.7
13.9
f1 (ppm)


S
S
OEt

**4ax**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7.36
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7.32
7.30
7.27
7.25
7.23
6.65
6.61
6.29
6.28
6.26
6.24
6.22
4.70
4.68
4.67
4.65
3.98
3.97
3.96
3.95
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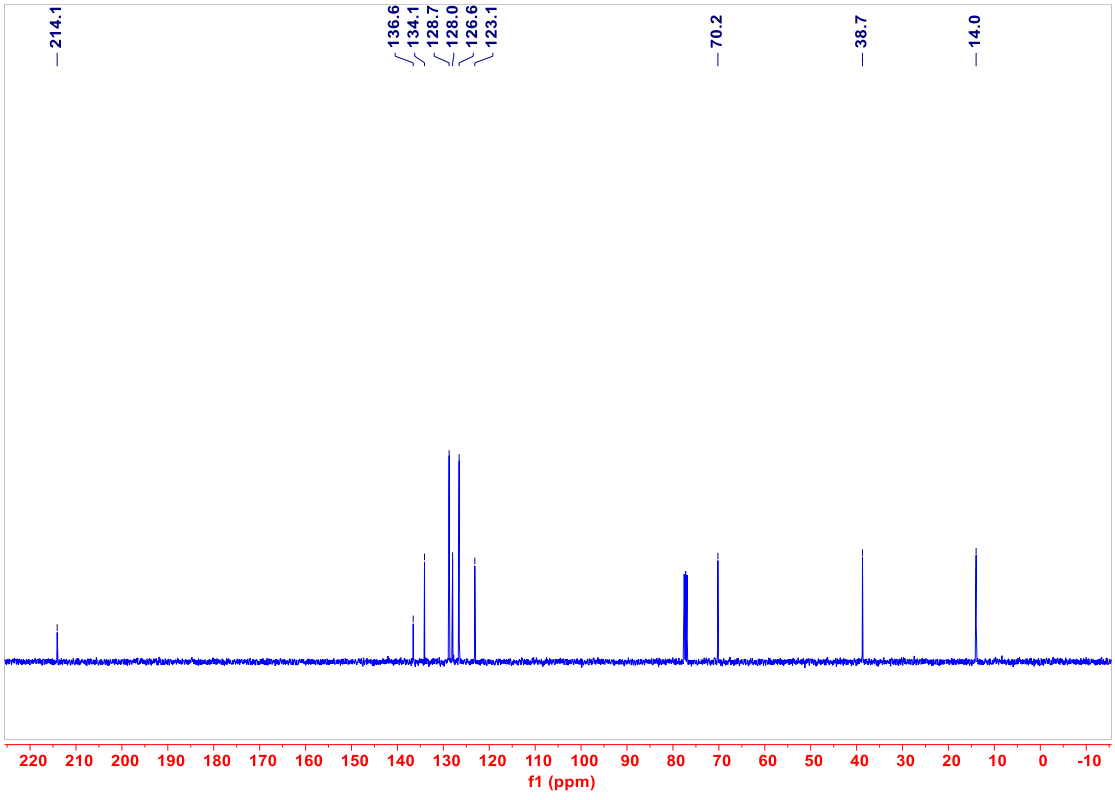
1.02
1.01
2.00
2.03
3.06
f1 (ppm)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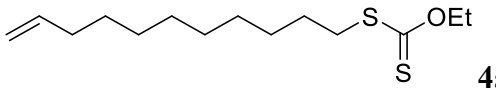
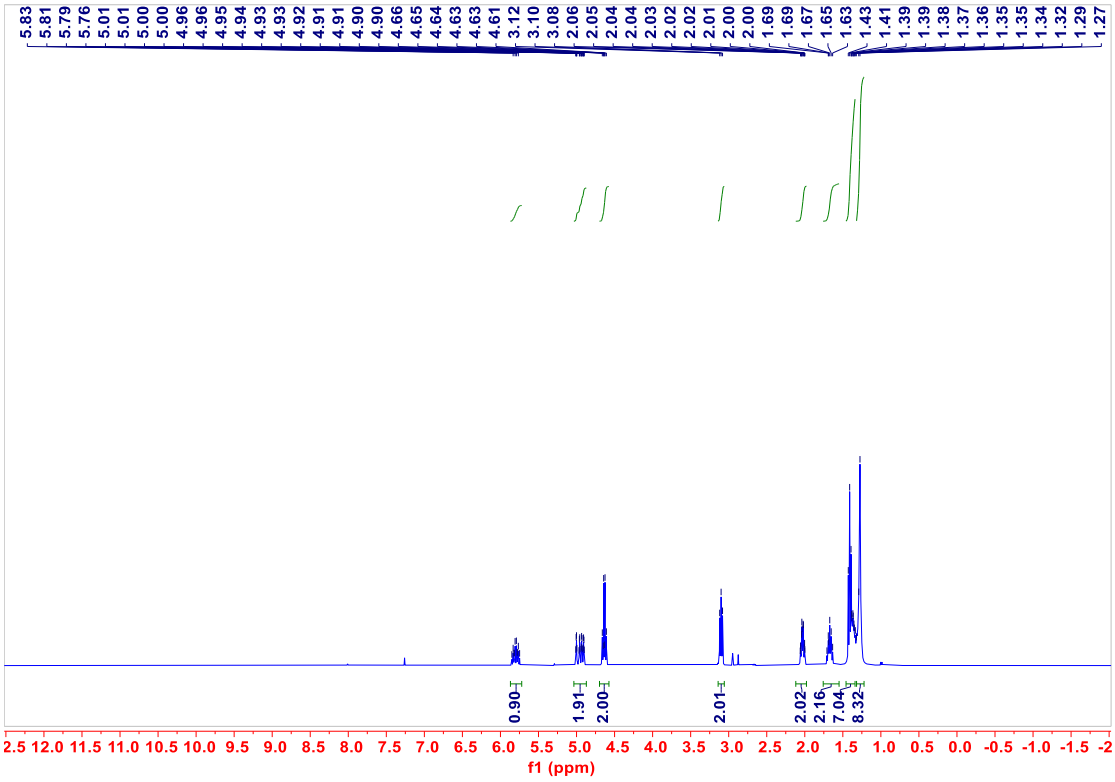
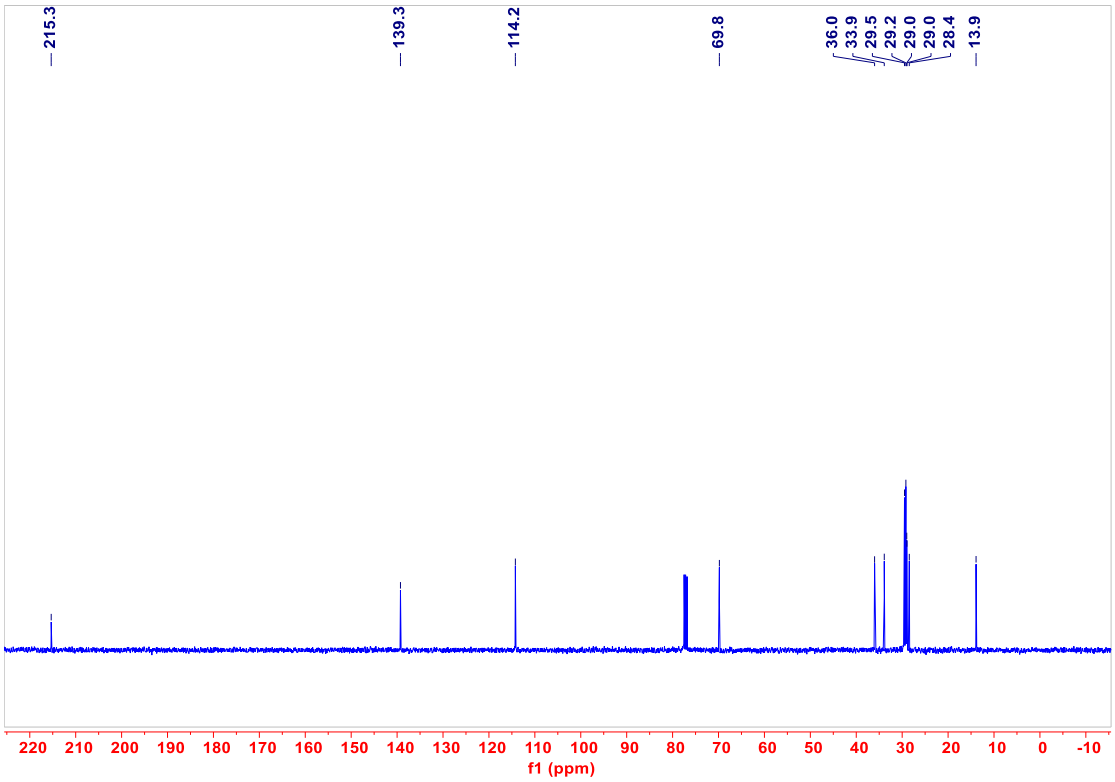
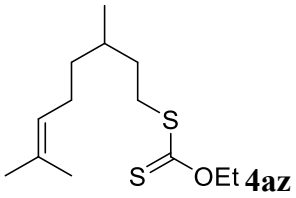
136.6
134.1
128.7
128.0
126.6
123.1
70.2
38.7
14.0
f1 (ppm)


S
OEt
S

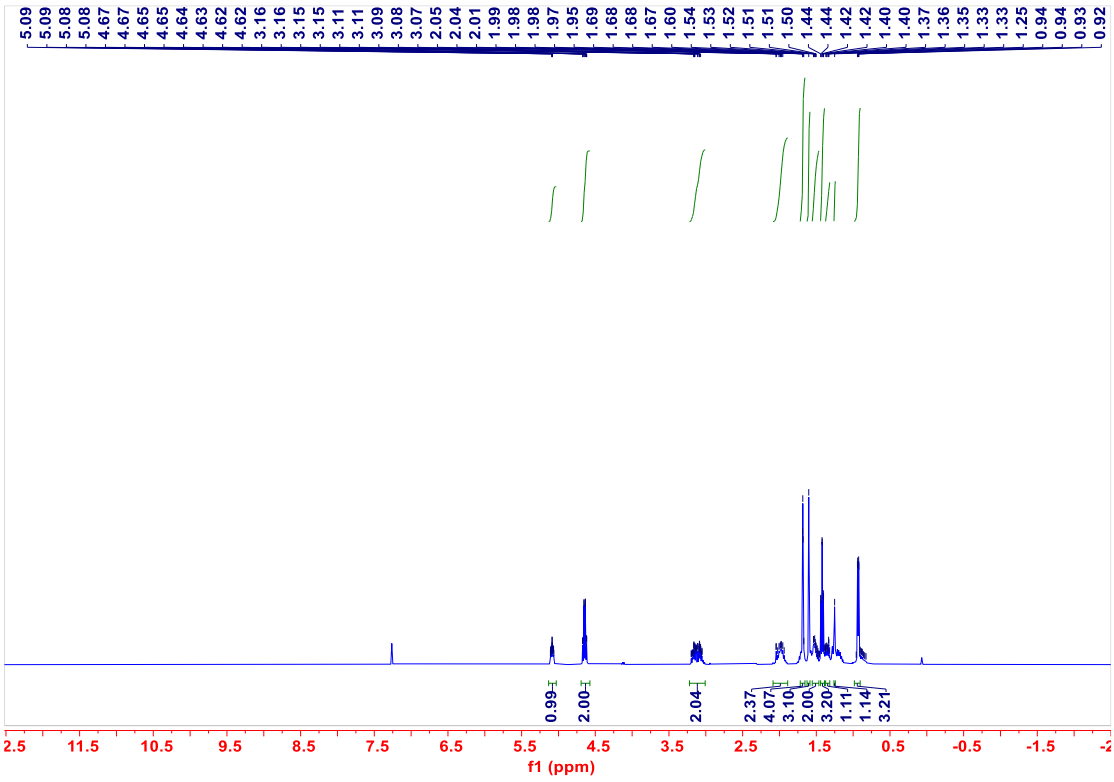
**4ay**

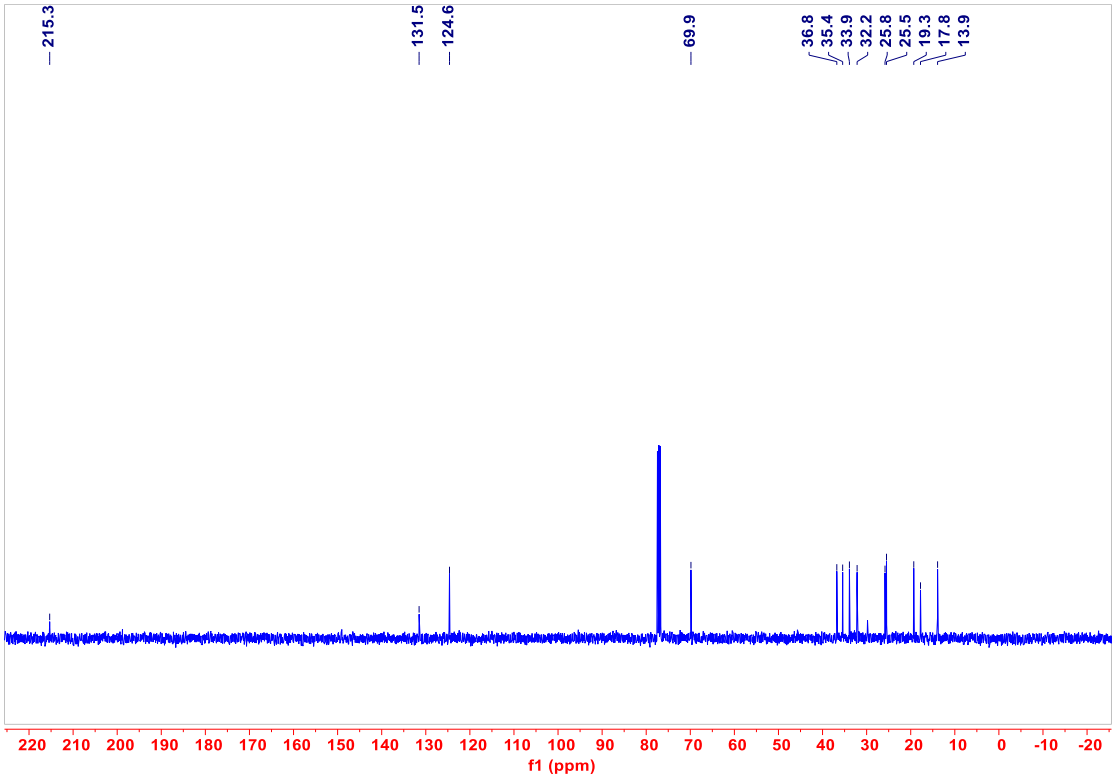

5.83 5.81 5.79 5.76 5.01 5.01 5.00 5.00 4.96 4.96 4.95 4.94 4.93 4.93 4.92 4.91 4.91 4.90 4.90 4.66 4.65 4.64 4.63 4.63 4.61 3.12 3.10 3.08 2.06 2.05 2.04 2.04 2.03 2.02 2.02 2.01 2.00 2.00 1.69 1.69 1.67 1.65 1.63 1.43 1.41 1.39 1.39 1.38 1.37 1.36 1.35 1.35 1.34 1.32 1.29 1.27
0.90 1.91 2.00 2.01 2.02 2.16 7.04 8.32
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)


215.3
139.3
114.2
69.8
36.0
33.9
29.5
29.2
29.0
29.0
28.4
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt

**4az**


5.09 5.09 5.08 5.08 4.67 4.67 4.65 4.64 4.63 4.62 4.62 3.16 3.16 3.15 3.15 3.11 3.11 3.09 3.08 3.07 2.05 2.04 2.01 1.99 1.98 1.98 1.97 1.95 1.69 1.68 1.68 1.67 1.60 1.54 1.53 1.52 1.51 1.51 1.50 1.44 1.44 1.42 1.42 1.40 1.40 1.37 1.36 1.35 1.33 1.33 1.25 0.94 0.94 0.93 0.92
0.99 2.00 2.04 2.37 4.07 3.10 2.00 3.20 1.11 1.14 3.21
2.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5
f1 (ppm)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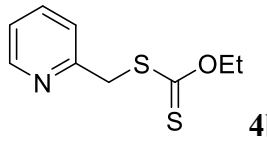
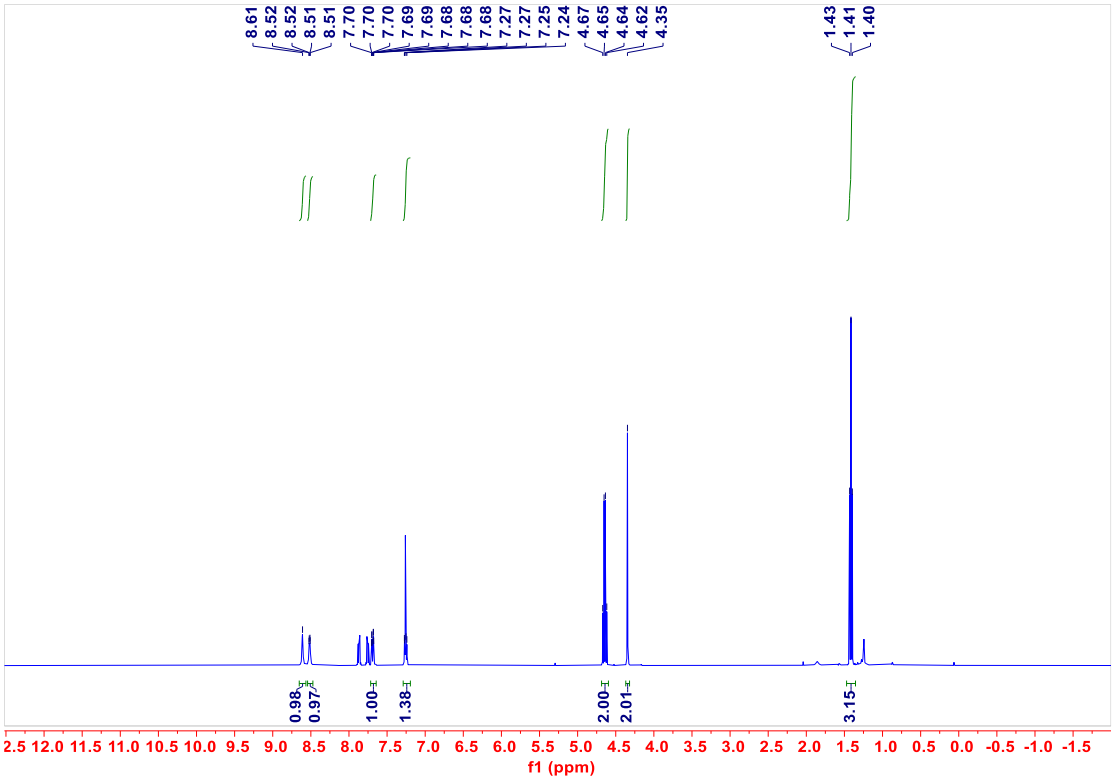
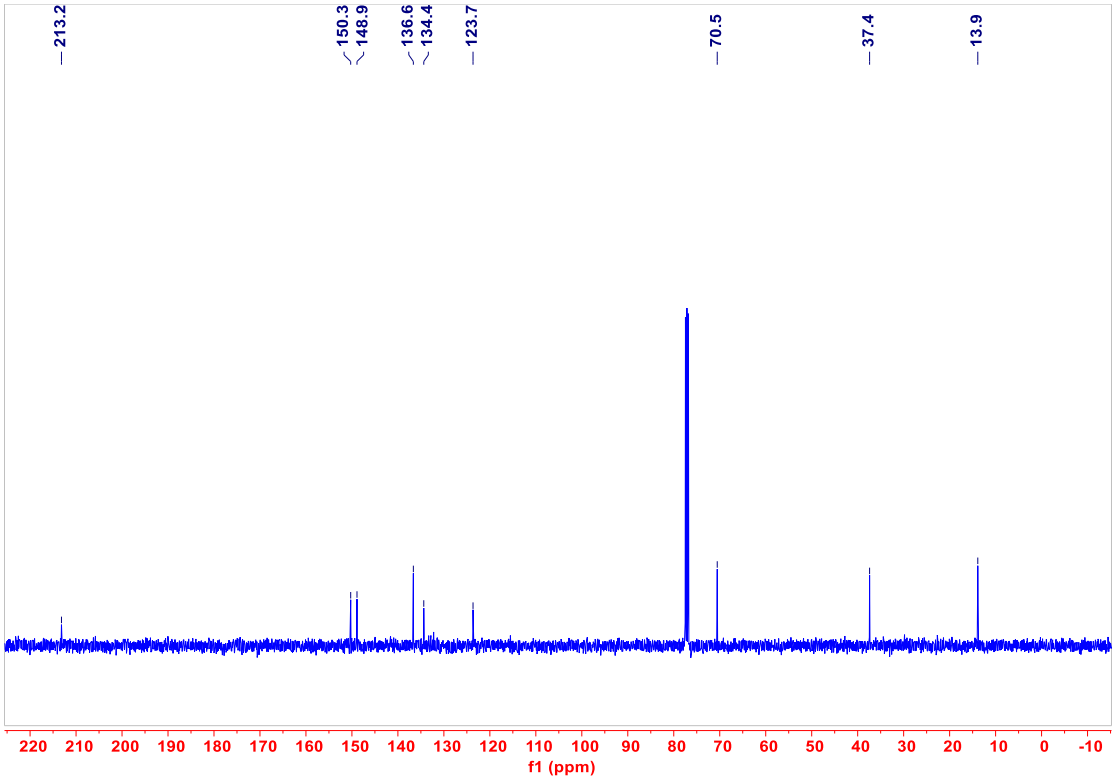
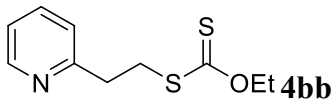
131.5
124.6
69.9
36.8
35.4
33.9
32.2
25.8
25.5
19.3
17.8
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


N
S
OEt
S

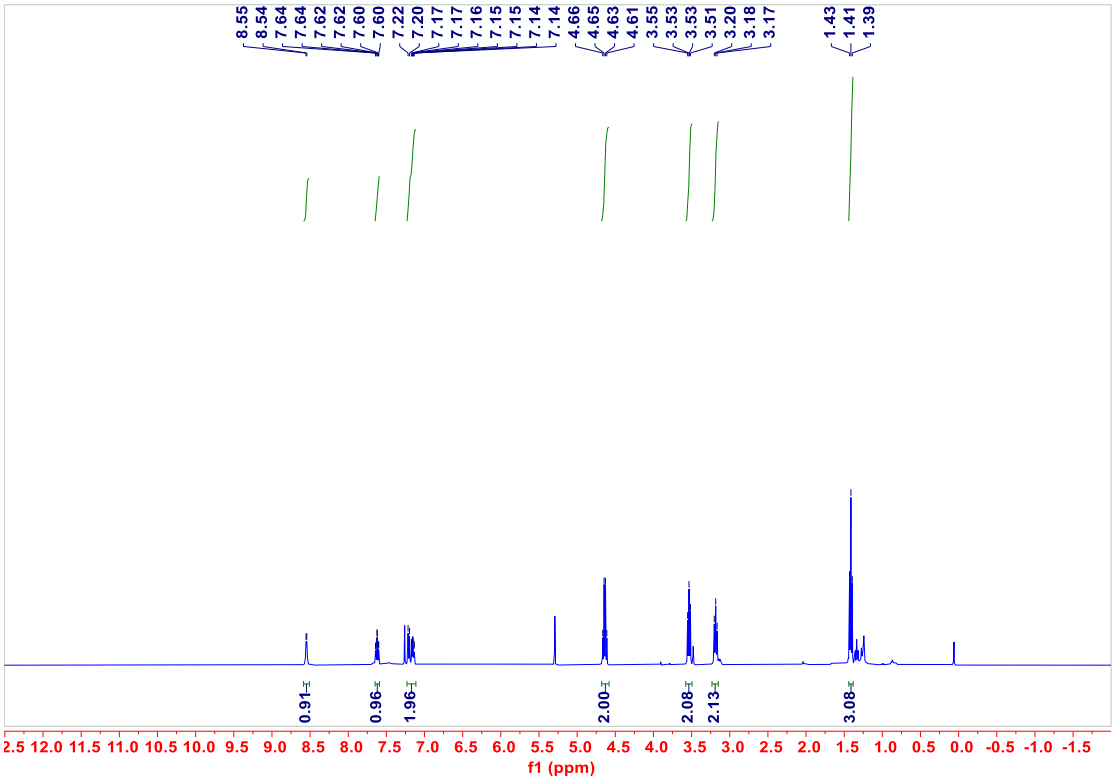
**4ba**

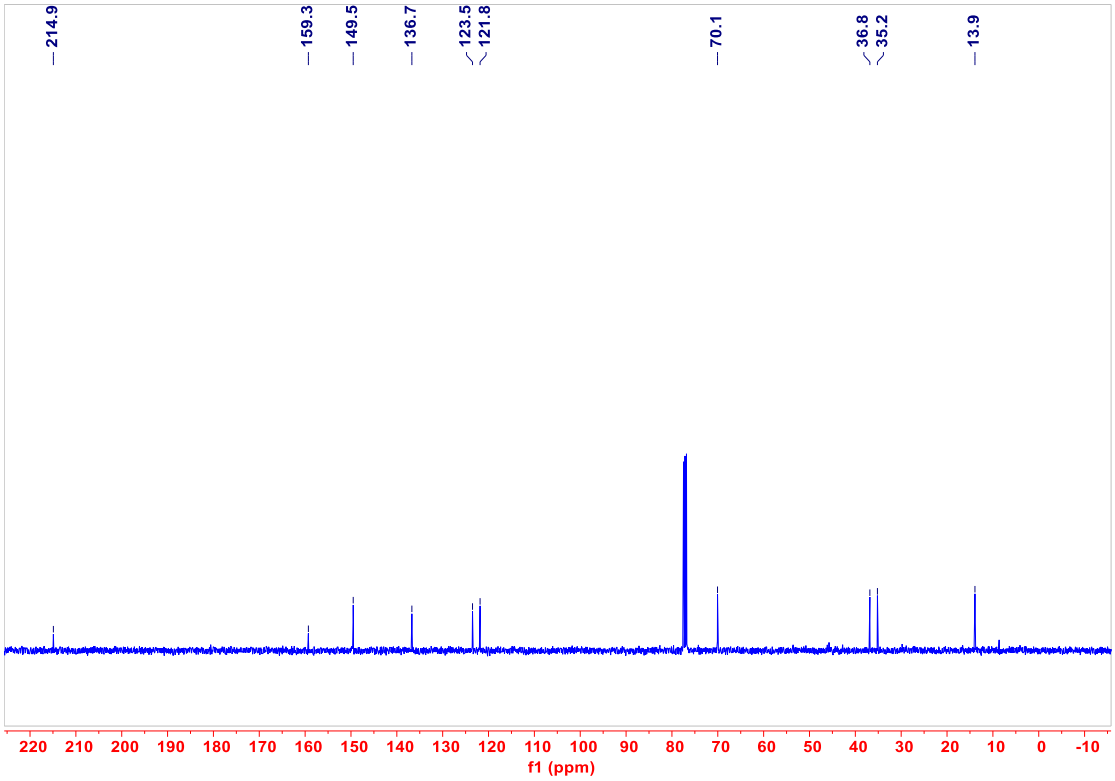

8.61
8.52
8.52
8.51
8.51
7.70
7.70
7.70
7.69
7.69
7.68
7.68
7.68
7.27
7.27
7.25
7.24
4.67
4.65
4.64
4.62
4.35
1.43
1.41
1.40
0.98
0.97
1.00
1.38
2.00
2.01
3.15
f1 (ppm)

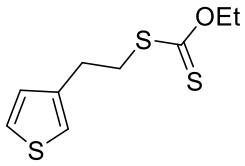

213.2
150.3
148.9
136.6
134.4
123.7
70.5
37.4
13.9
f1 (ppm)


S
N
S
OEt

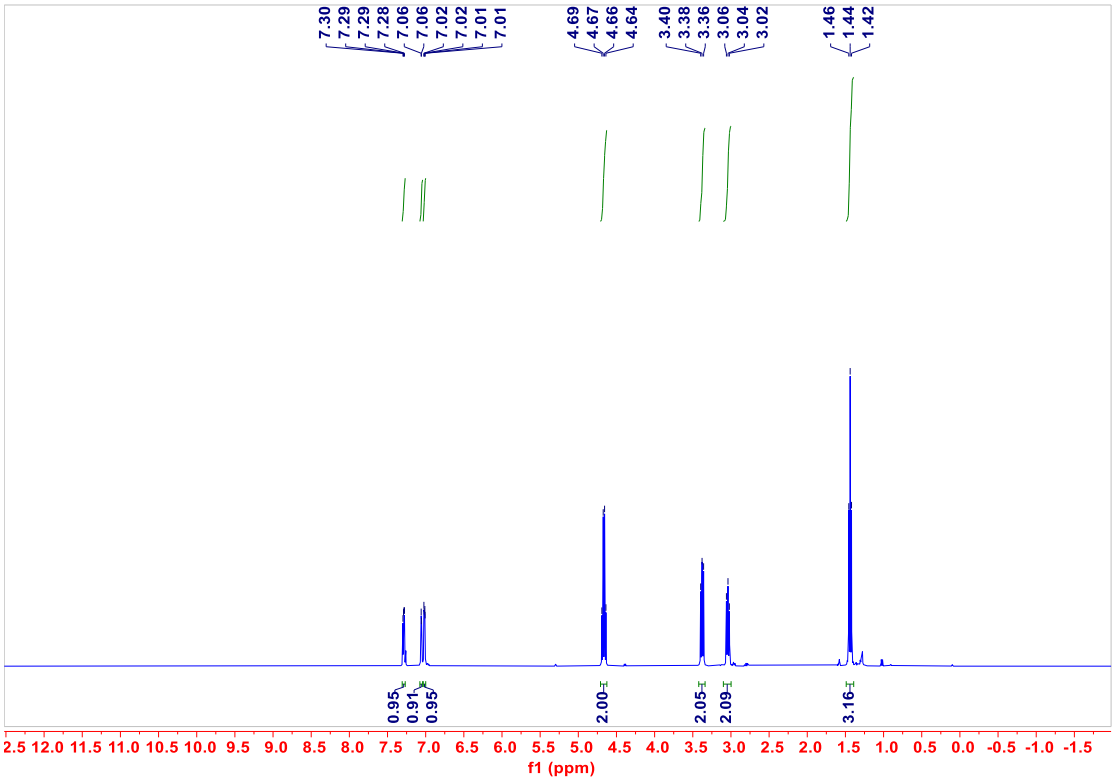
**4bb**

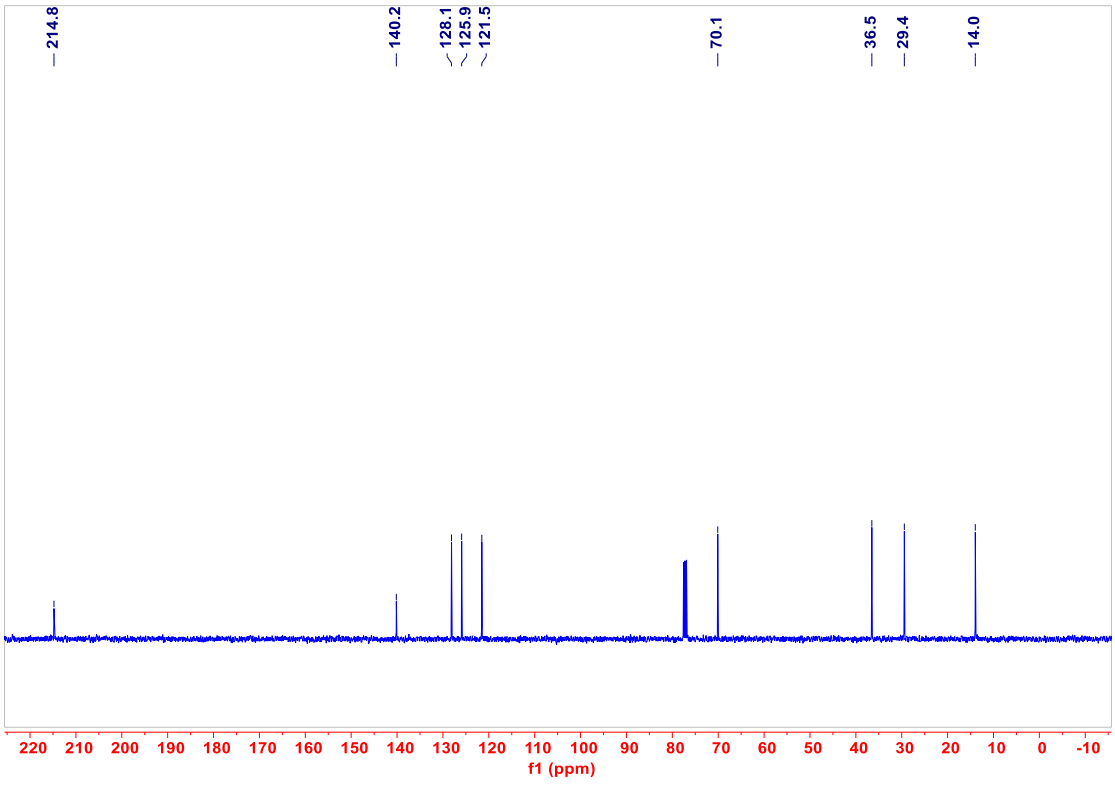

8.55
8.54
7.64
7.62
7.62
7.60
7.60
7.22
7.20
7.17
7.17
7.16
7.15
7.15
7.14
7.14
4.66
4.65
4.63
4.61
3.55
3.53
3.53
3.51
3.20
3.18
3.17
1.43
1.41
1.39
0.91
0.96
1.96
2.00
2.08
2.13
3.08
f1 (ppm)

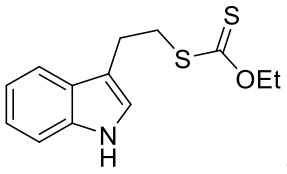

214.9
159.3
149.5
136.7
123.5
121.8
70.1
36.8
35.2
13.9
f1 (ppm)


OEt
S
S
S

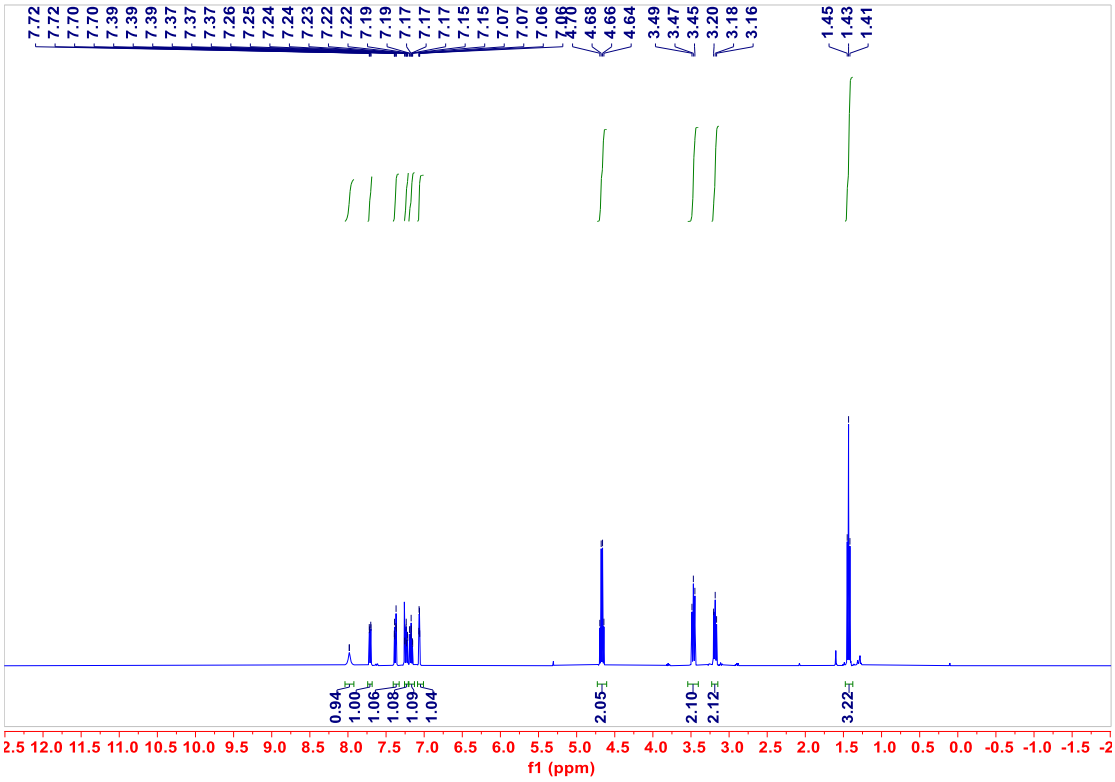
**4bc**

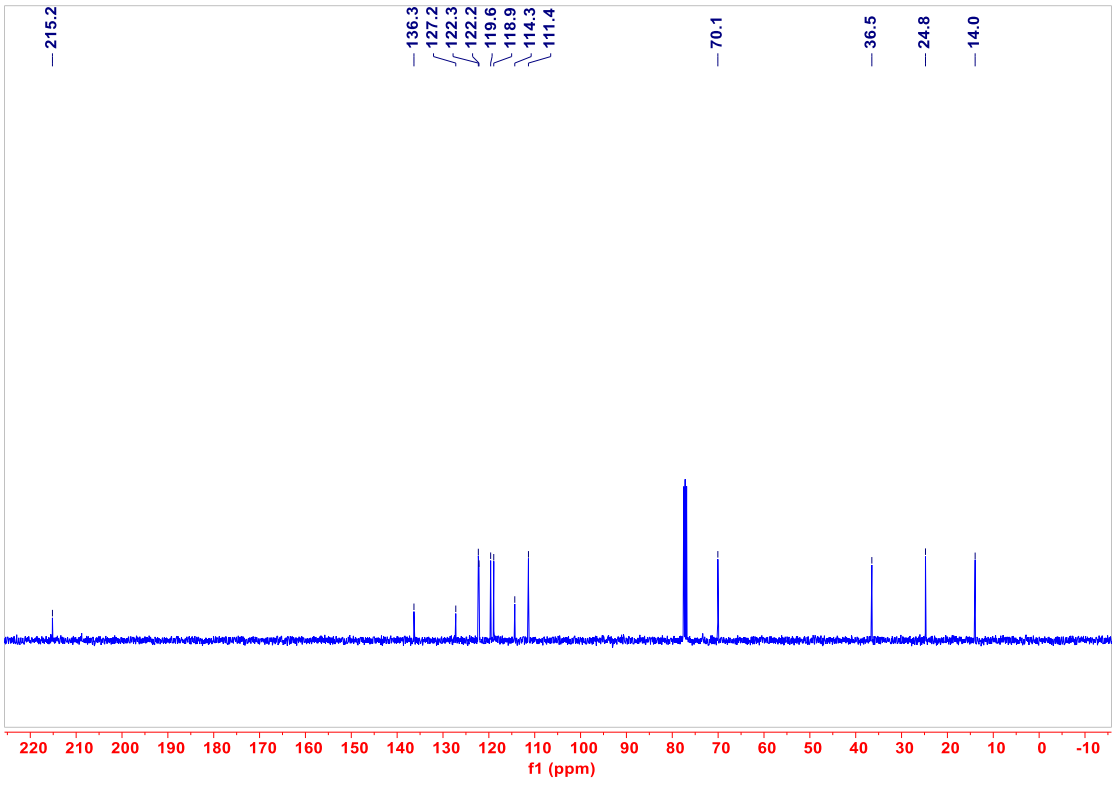

7.30
7.29
7.29
7.28
7.06
7.06
7.02
7.02
7.01
7.01
4.69
4.67
4.66
4.64
3.40
3.38
3.36
3.06
3.04
3.02
1.46
1.44
1.42
0.95
0.91
0.95
2.00
2.05
2.09
3.16
f1 (ppm)

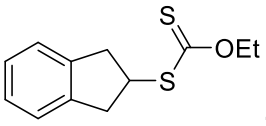

214.8
140.2
128.1
125.9
121.5
70.1
36.5
29.4
14.0
f1 (ppm)


S
S
OEt
N
H

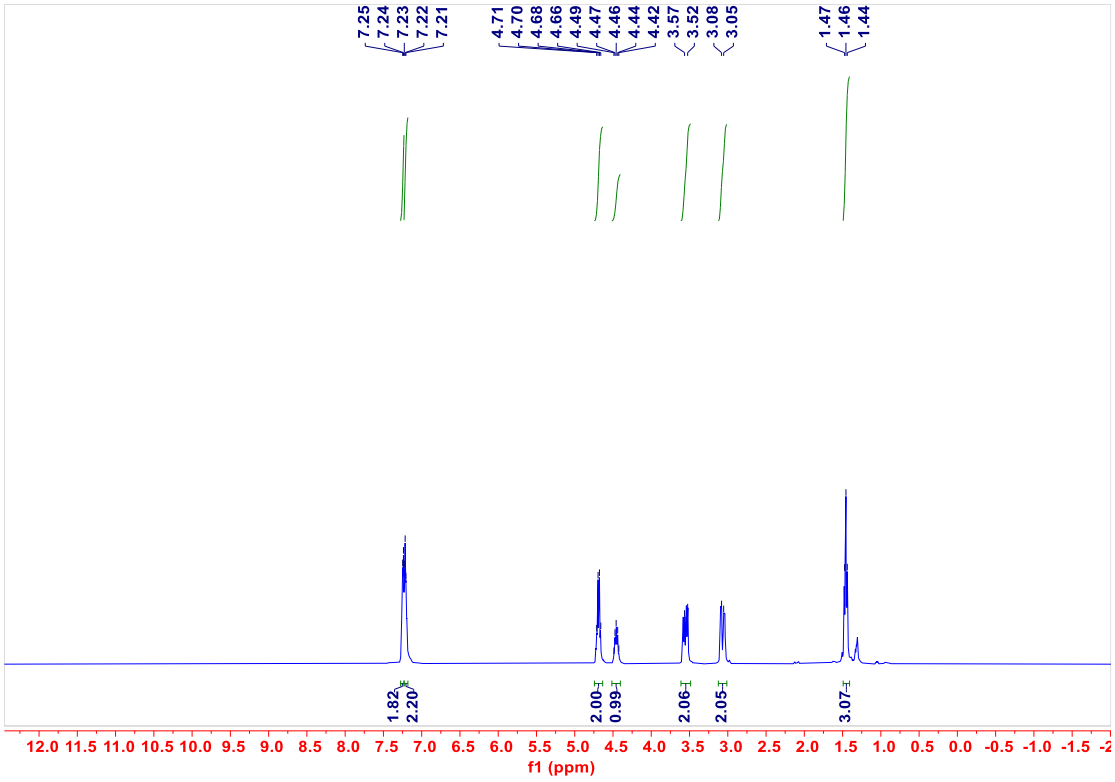
**4bd**


7.72 7.72 7.70 7.70 7.39 7.39 7.37 7.37 7.26 7.25 7.24 7.24 7.23 7.22 7.22 7.19 7.17 7.17 7.15 7.15 7.07 7.07 7.06 4.68 4.66 4.64 3.49 3.47 3.45 3.20 3.18 3.16 1.45 1.43 1.41
0.94 1.00 1.06 1.08 1.09 1.04 2.05 2.10 2.12 3.22
f1 (ppm)


215.2 136.3 127.2 122.3 122.2 119.6 118.9 114.3 111.4 70.1 36.5 24.8 14.0
f1 (ppm)


S
OEt
S

**4be**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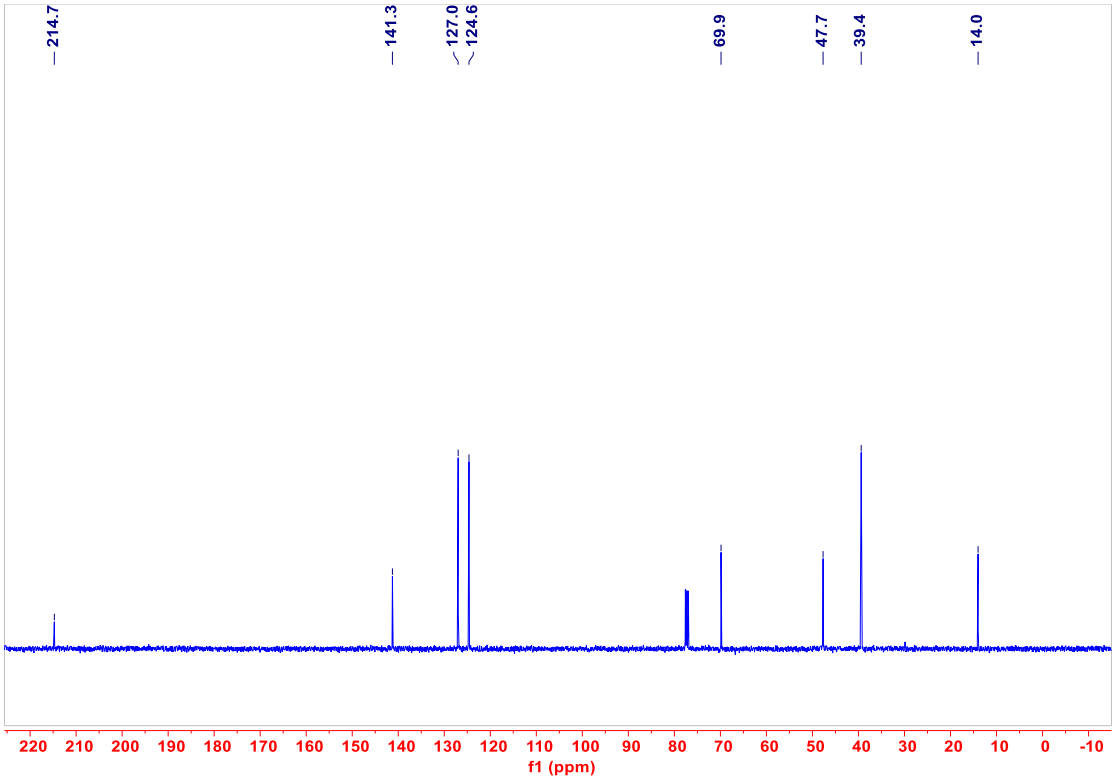
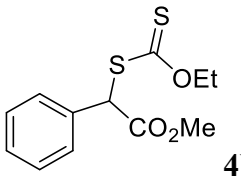
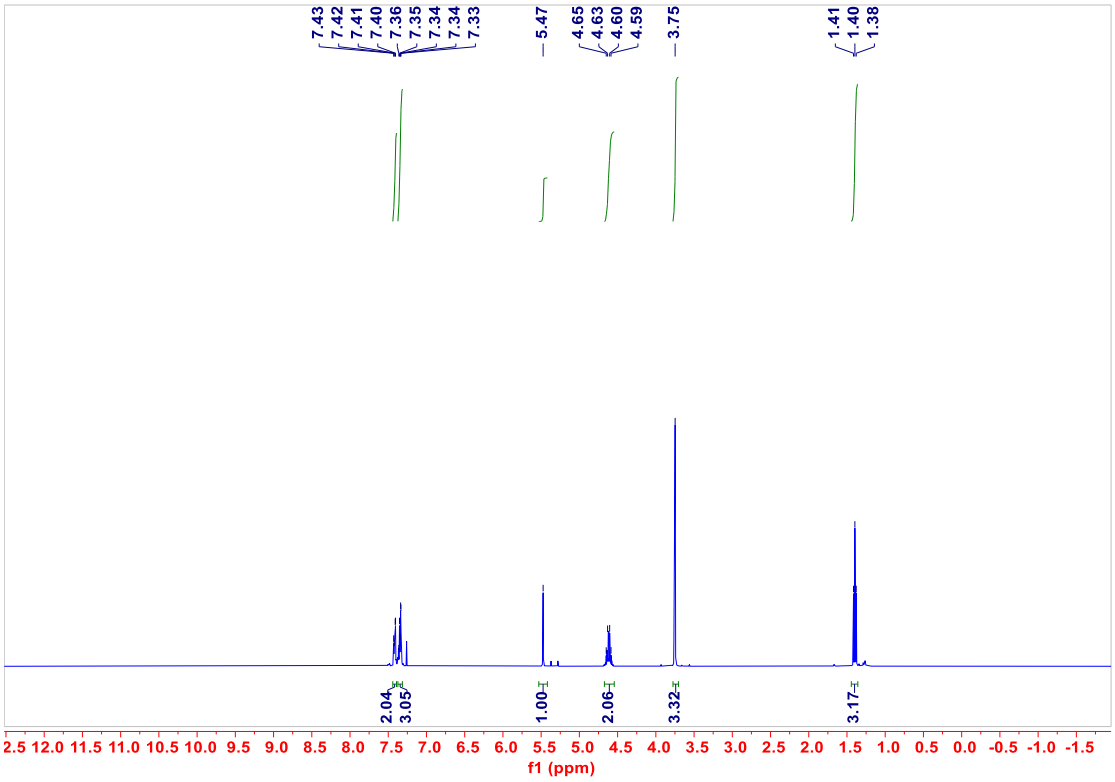
7.24
7.23
7.22
7.21
4.71
4.70
4.68
4.66
4.49
4.47
4.46
4.44
4.42
3.57
3.52
3.08
3.05
1.47
1.46
1.44
1.82
2.20
2.00
0.99
2.06
2.05
3.07
f1 (ppm)


214.7
141.3
127.0
124.6
69.9
47.7
39.4
14.0
f1 (ppm)


S
S
OEt
CO2Me

**4bf**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7.42
7.41
7.40
7.36
7.35
7.34
7.34
7.33
5.47
4.65
4.63
4.60
4.59
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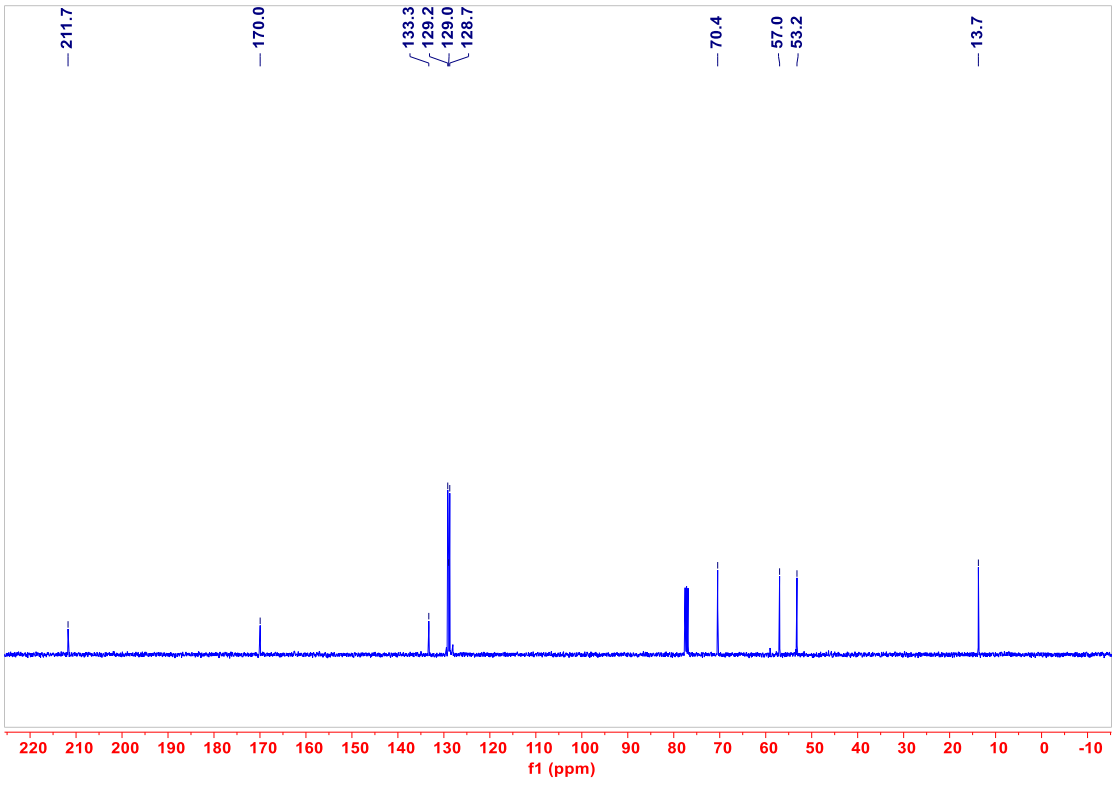
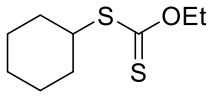
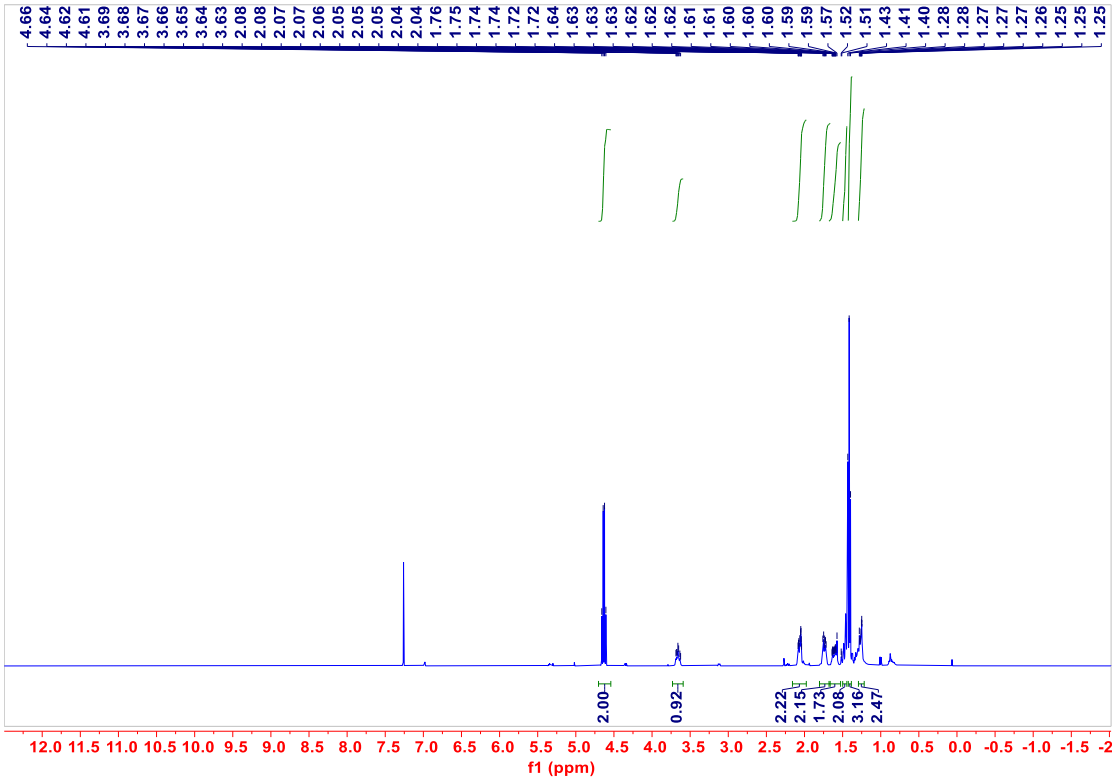
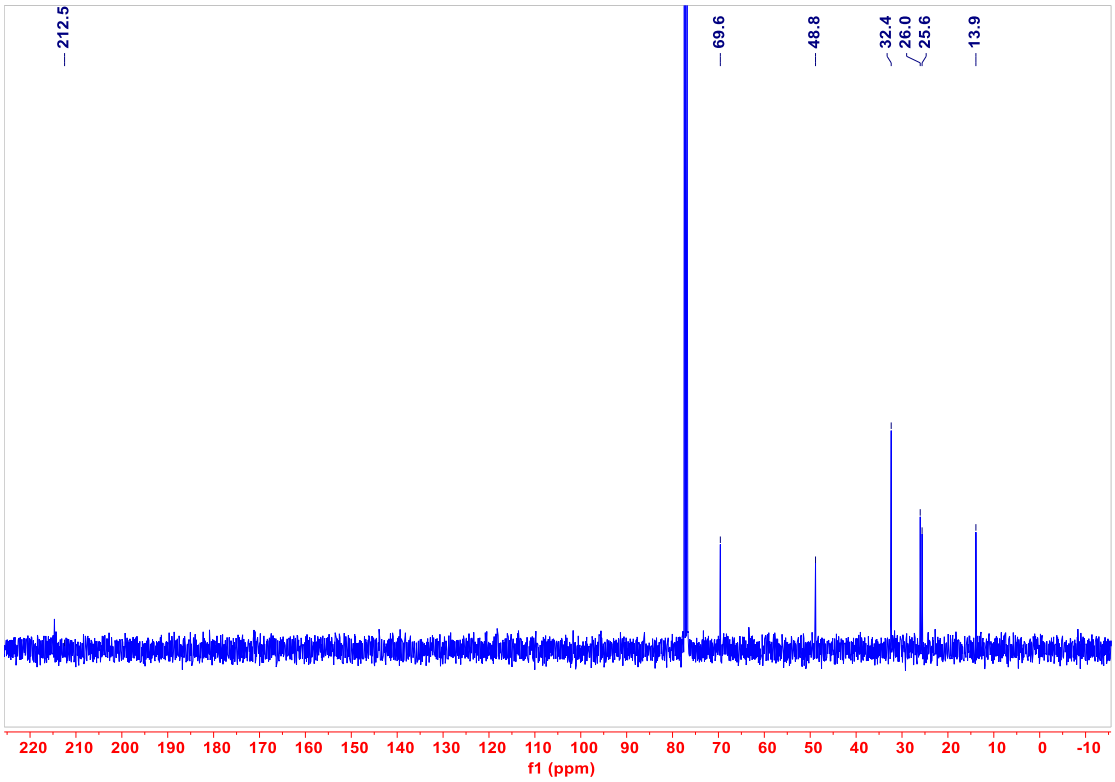
1.40
1.38
2.04
3.05
1.00
2.06
3.32
3.17
f1 (ppm)

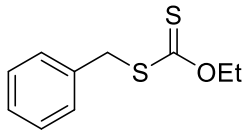

211.7
170.0
133.3
129.2
129.0
128.7
70.4
57.0
53.2
13.7
f1 (ppm)


S
OEt
S

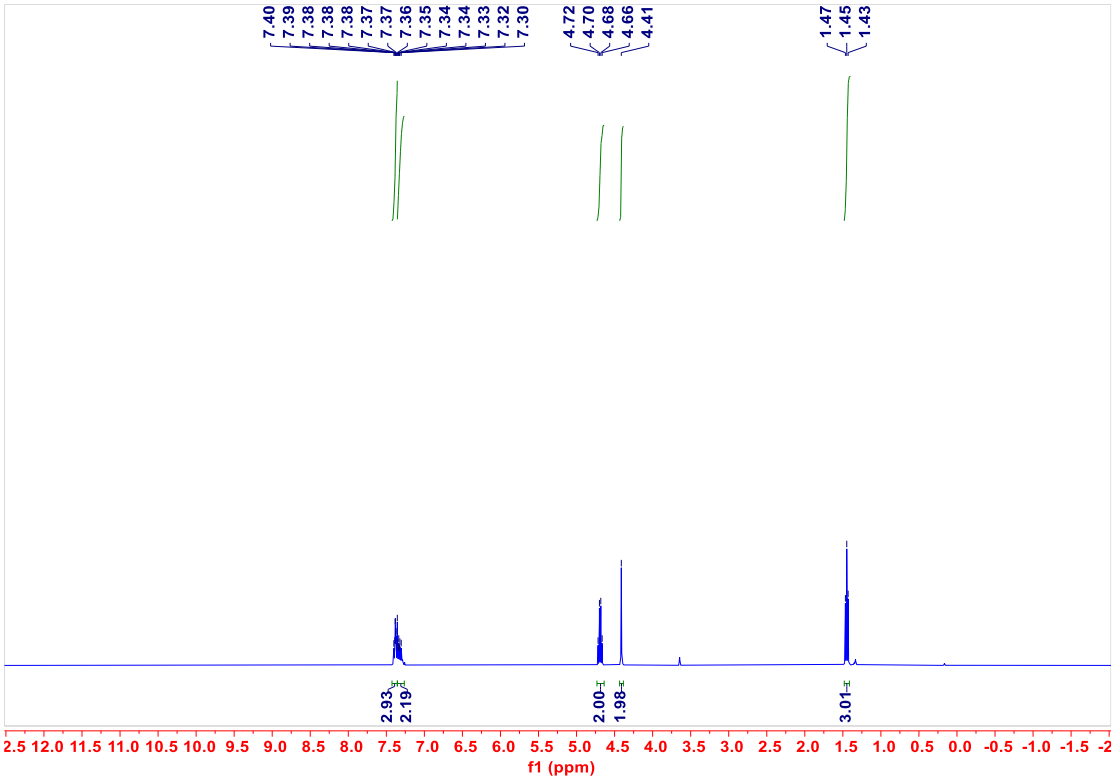
**4bi**


4.66 4.64 4.62 4.61 3.69 3.68 3.67 3.66 3.65 3.64 3.63 2.08 2.08 2.07 2.07 2.06 2.05 2.05 2.05 2.04 2.04 1.76 1.75 1.74 1.74 1.72 1.72 1.64 1.63 1.63 1.62 1.62 1.62 1.61 1.61 1.60 1.60 1.60 1.59 1.59 1.57 1.52 1.51 1.43 1.41 1.40 1.28 1.28 1.27 1.27 1.27 1.26 1.25 1.25 1.25
2.00
0.92
2.22
2.15
1.73
2.08
3.16
2.47
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)


212.5
69.6
48.8
32.4
26.0
25.6
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt

**4bj**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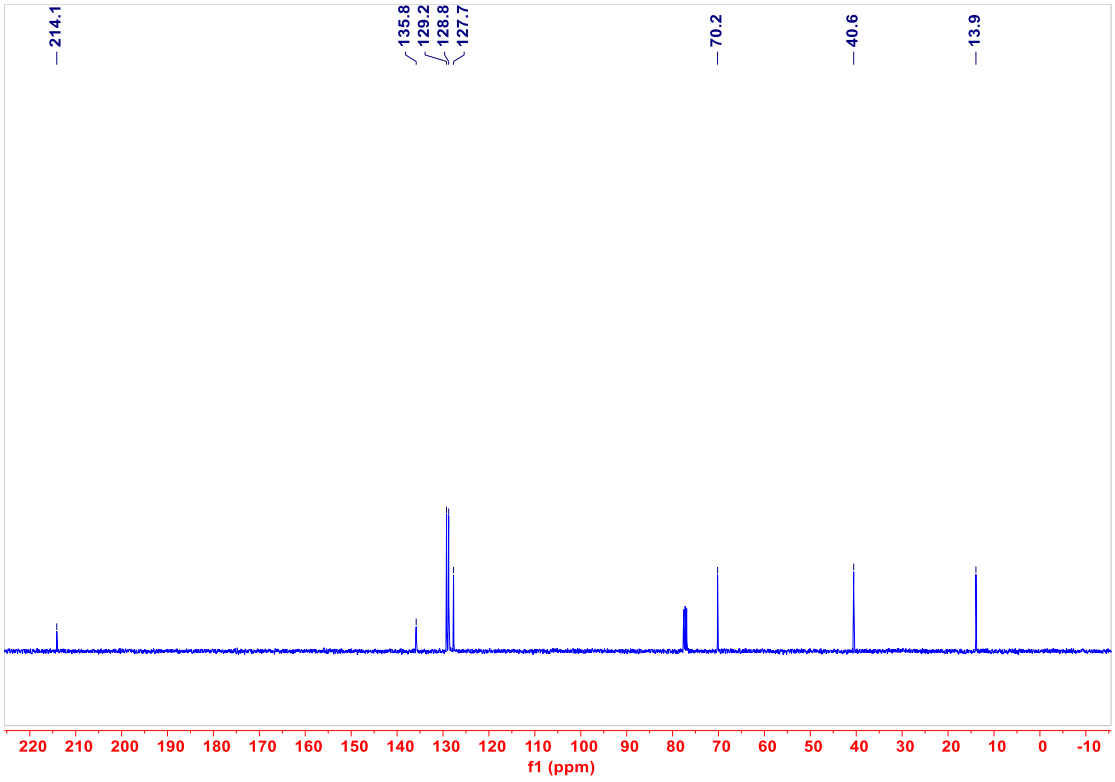
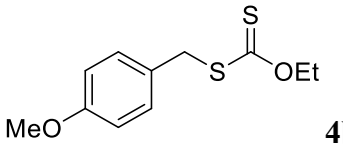
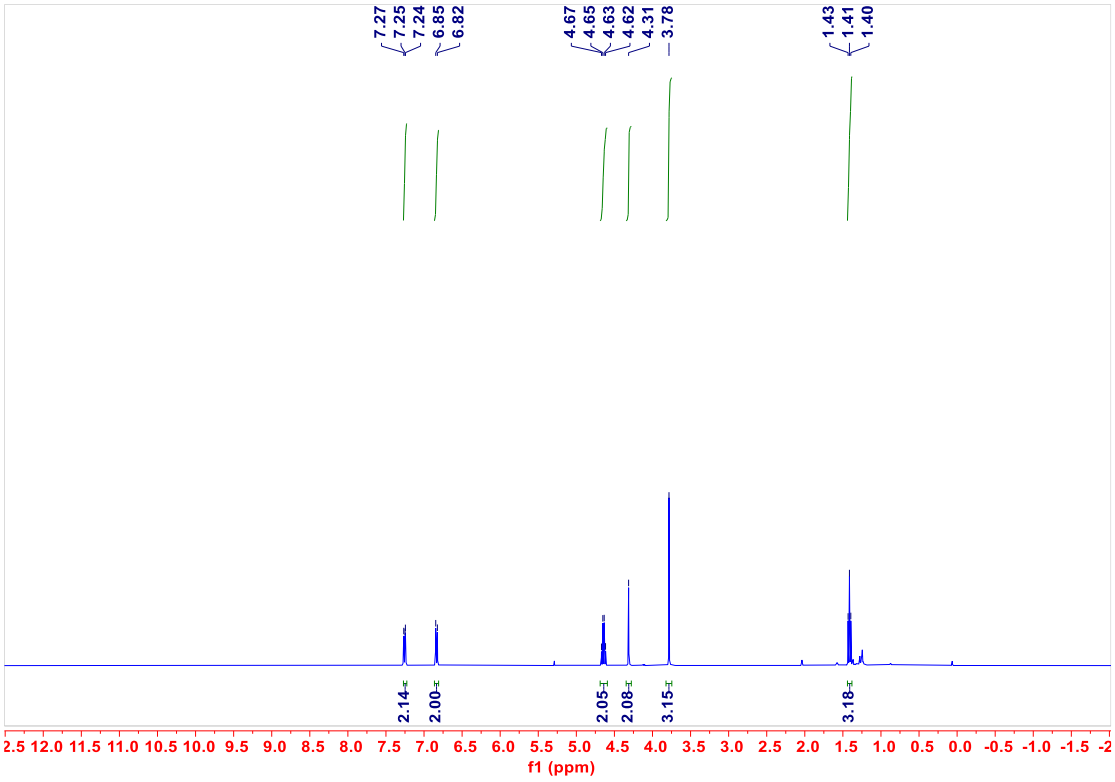
7.38
7.38
7.38
7.37
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.30
4.72
4.70
4.68
4.66
4.41
1.47
1.45
1.43
2.93
2.19
2.00
1.98
3.01
f1 (ppm)


214.1
135.8
129.2
128.8
127.7
70.2
40.6
13.9
f1 (ppm)


S
S
OEt
MeO

**4bk**


7.27
7.25
7.24
6.85
6.82
4.67
4.65
4.63
4.62
4.31
3.78
1.43
1.41
1.40
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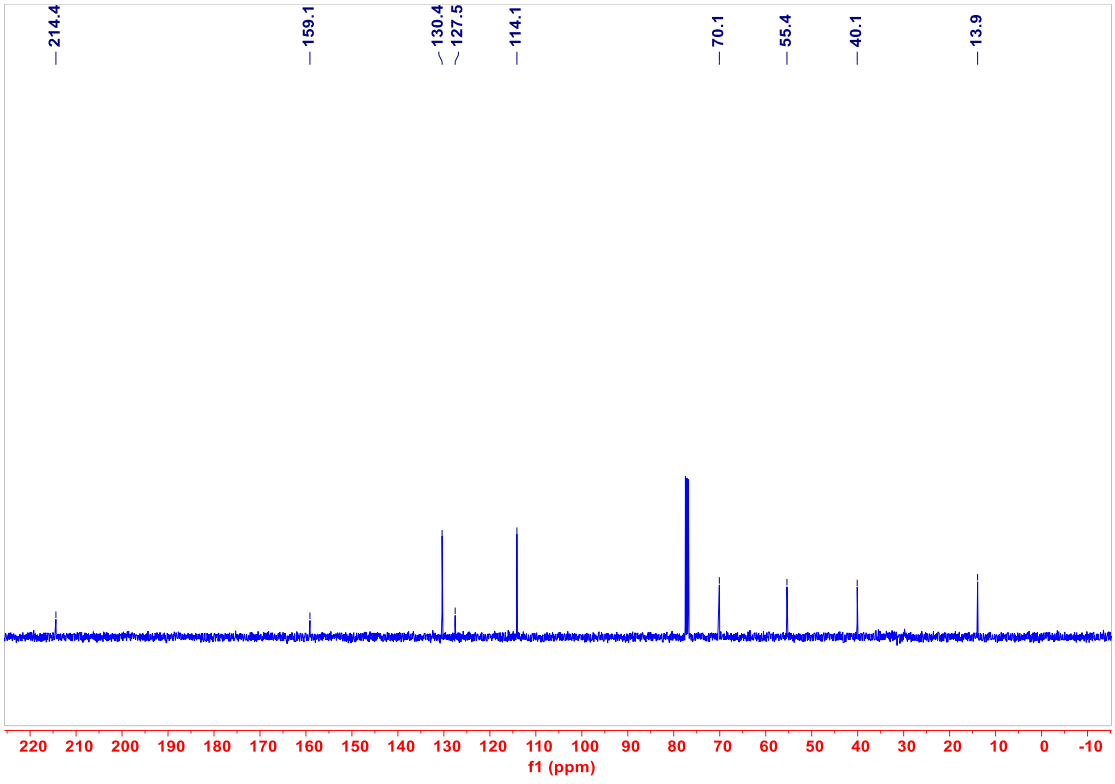
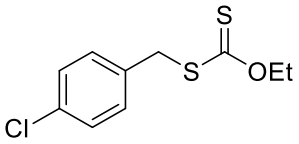
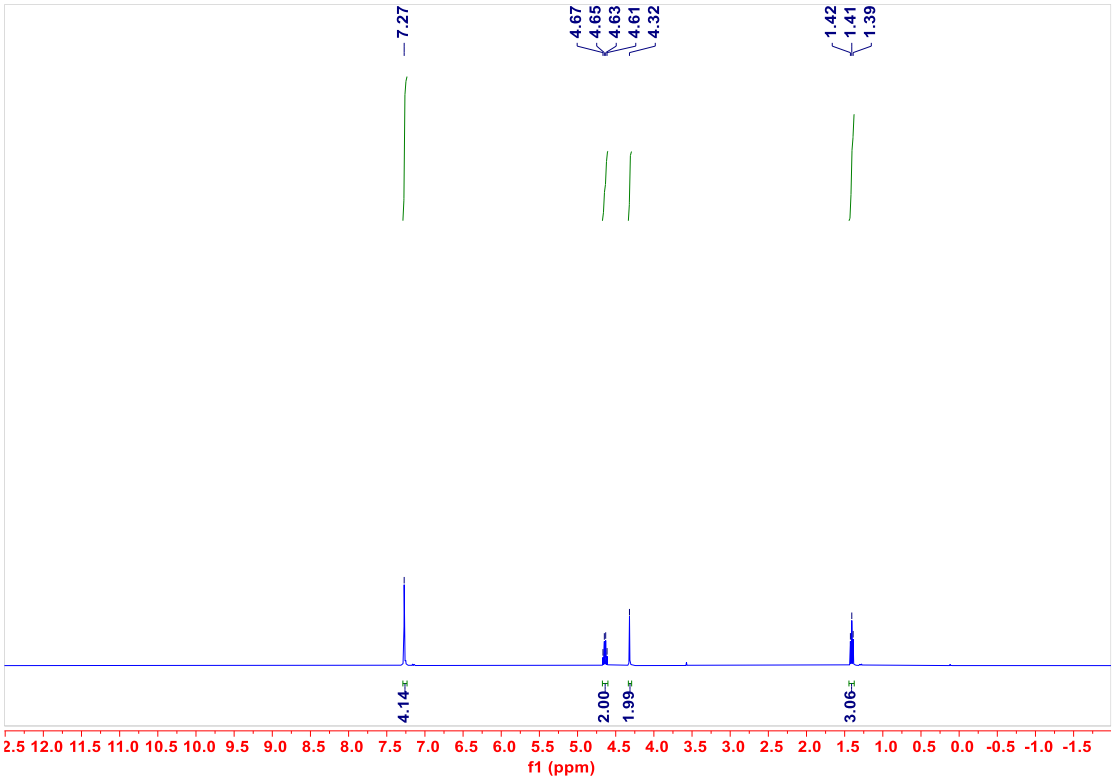
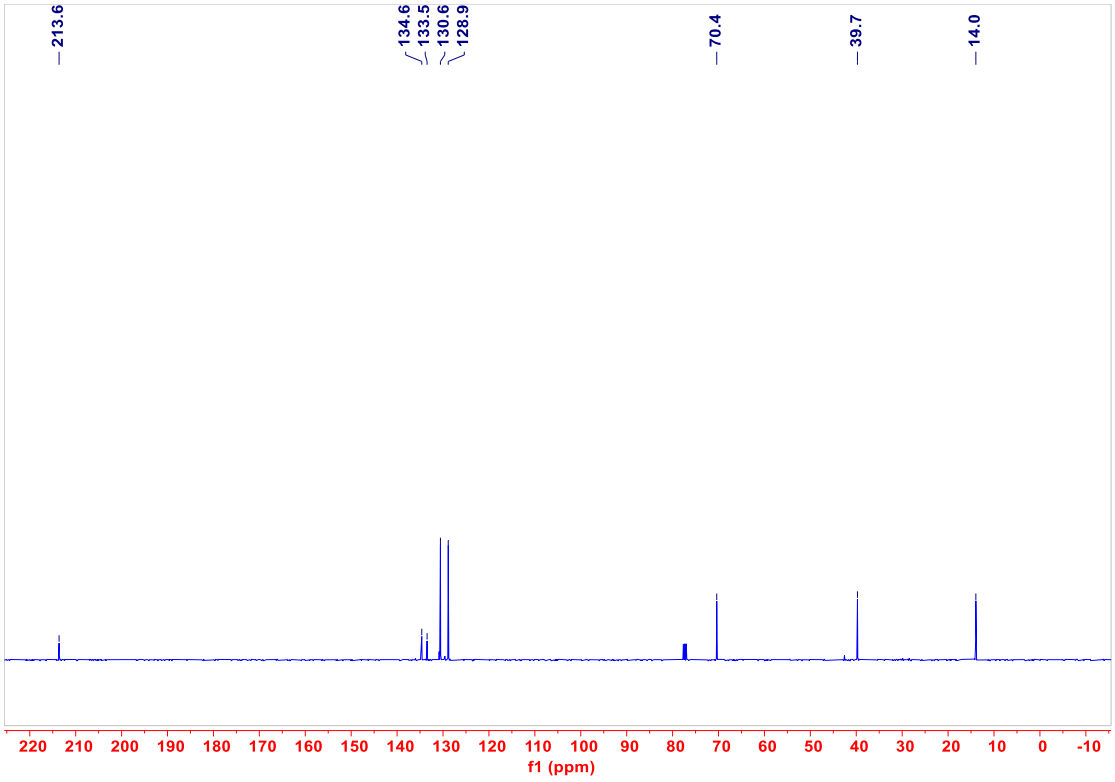
2.05
2.08
3.15
3.18
f1 (ppm)

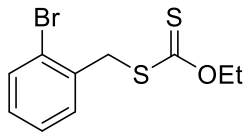

214.4
159.1
130.4
127.5
114.1
70.1
55.4
40.1
13.9
f1 (ppm)


S
S
OEt
Cl

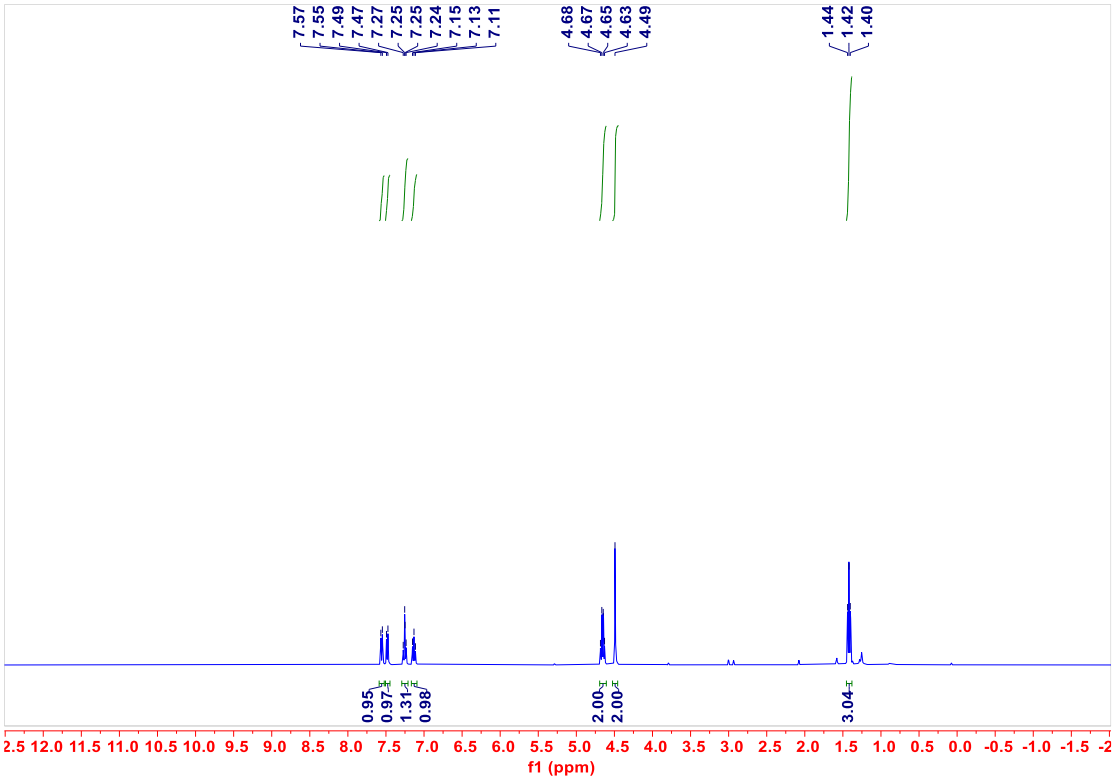
**4bl**

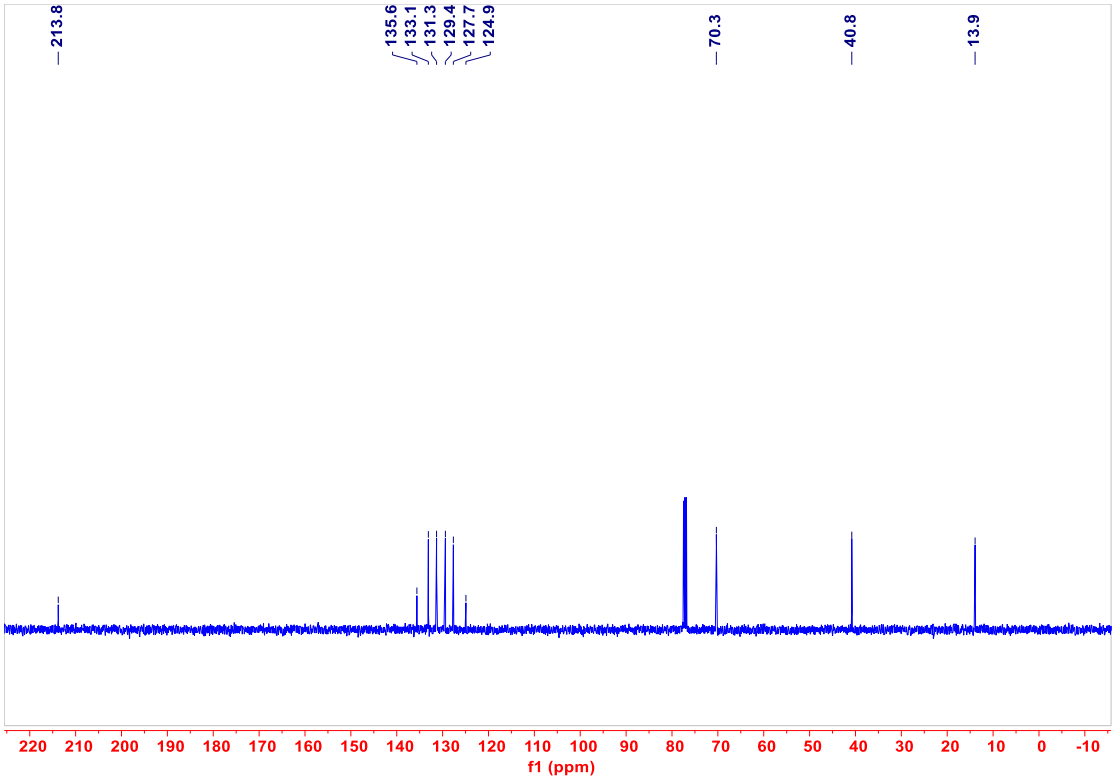

7.27
4.67
4.65
4.63
4.61
4.32
1.42
1.41
1.39
4.14
2.00
1.99
3.06
f1 (ppm)

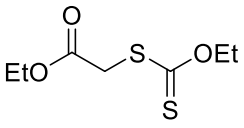

213.6
134.6
133.5
130.6
128.9
70.4
39.7
14.0
f1 (ppm)


Br
S
S
OEt

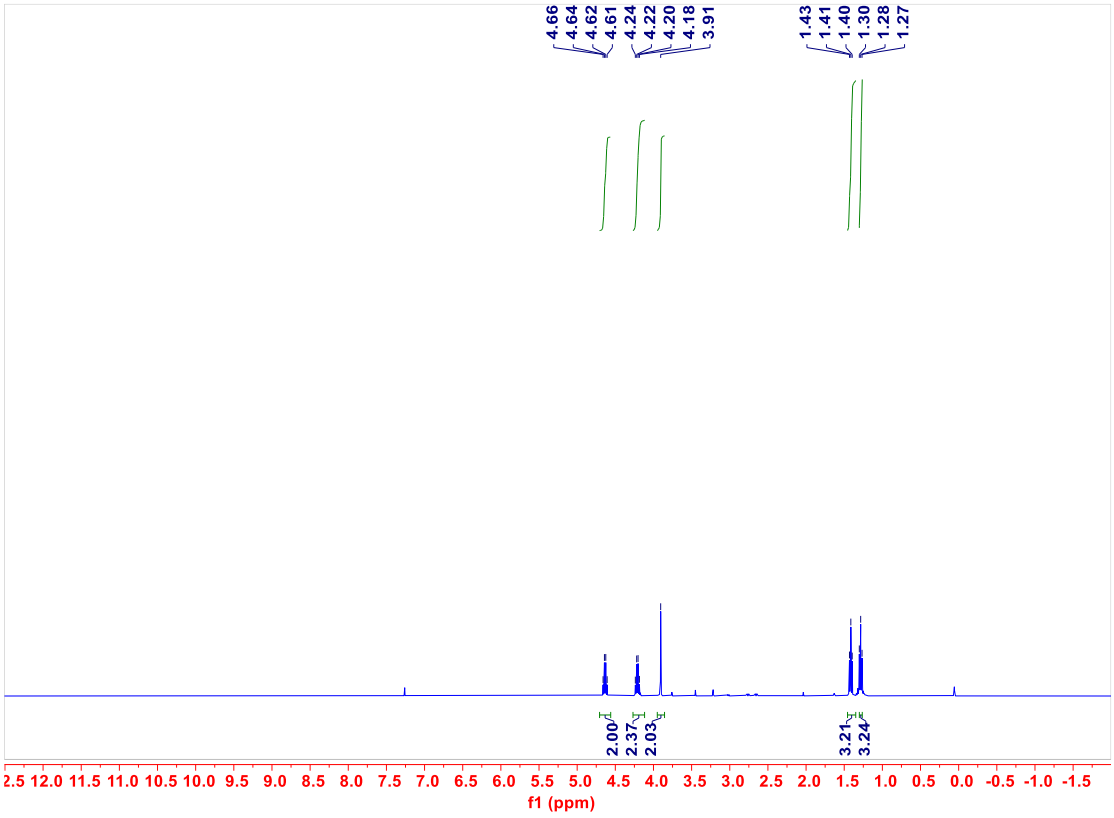
**4bm**

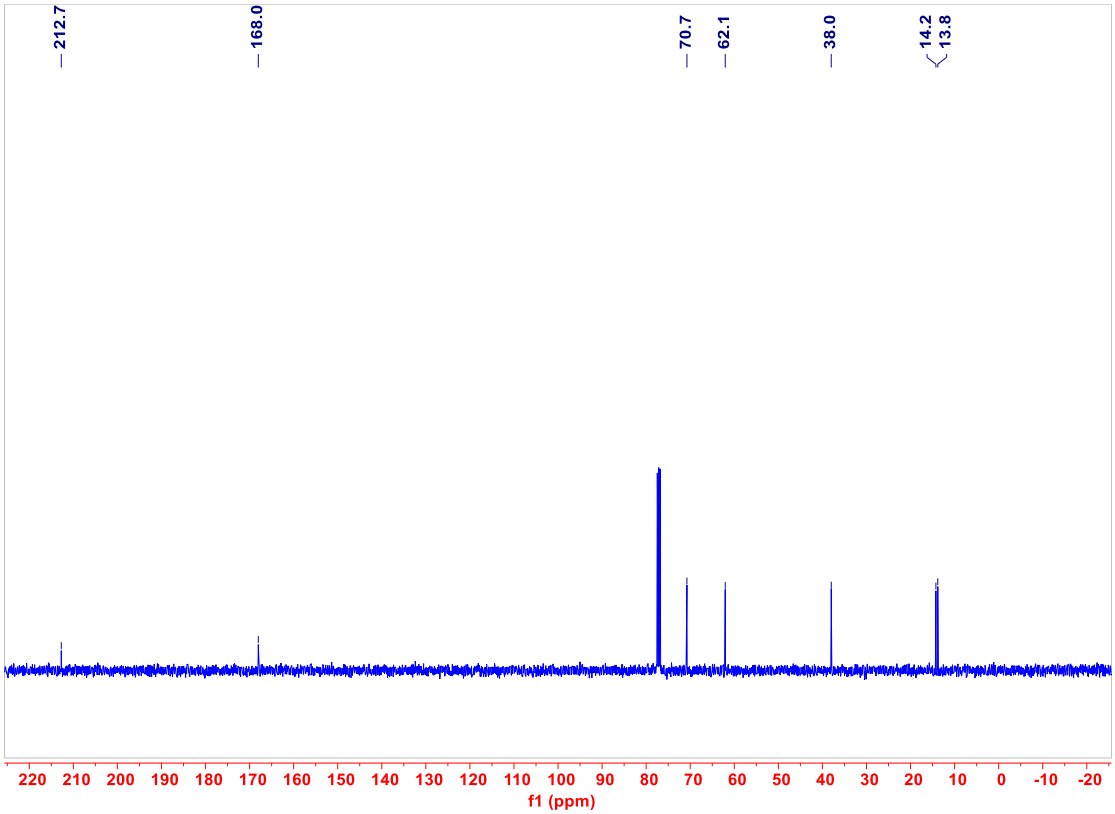

7.57
7.55
7.49
7.47
7.27
7.25
7.25
7.24
7.15
7.13
7.11
4.68
4.67
4.65
4.63
4.49
1.44
1.42
1.40
0.95
0.97
1.31
0.98
2.00
2.00
3.04
f1 (ppm)

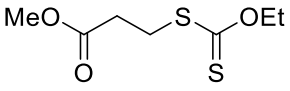

213.8
135.6
133.1
131.3
129.4
127.7
124.9
70.3
40.8
13.9
f1 (ppm)


O
EtO
S
OEt
S

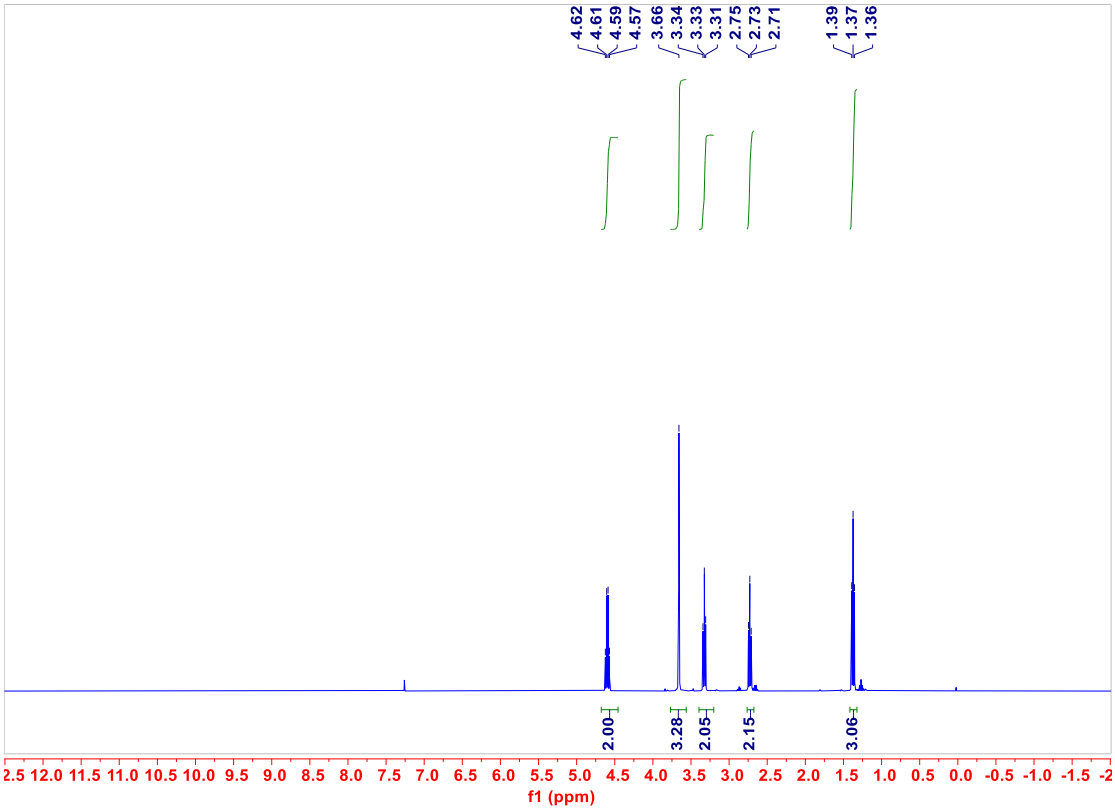
**4bn**


4.66
4.64
4.62
4.61
4.24
4.22
4.20
4.18
3.91
1.43
1.41
1.40
1.30
1.28
1.27
2.00
2.37
2.03
3.21
3.24
f1 (ppm)


212.7
168.0
70.7
62.1
38.0
14.2
13.8
f1 (ppm)


MeO
O
S
S
OEt

**4bo**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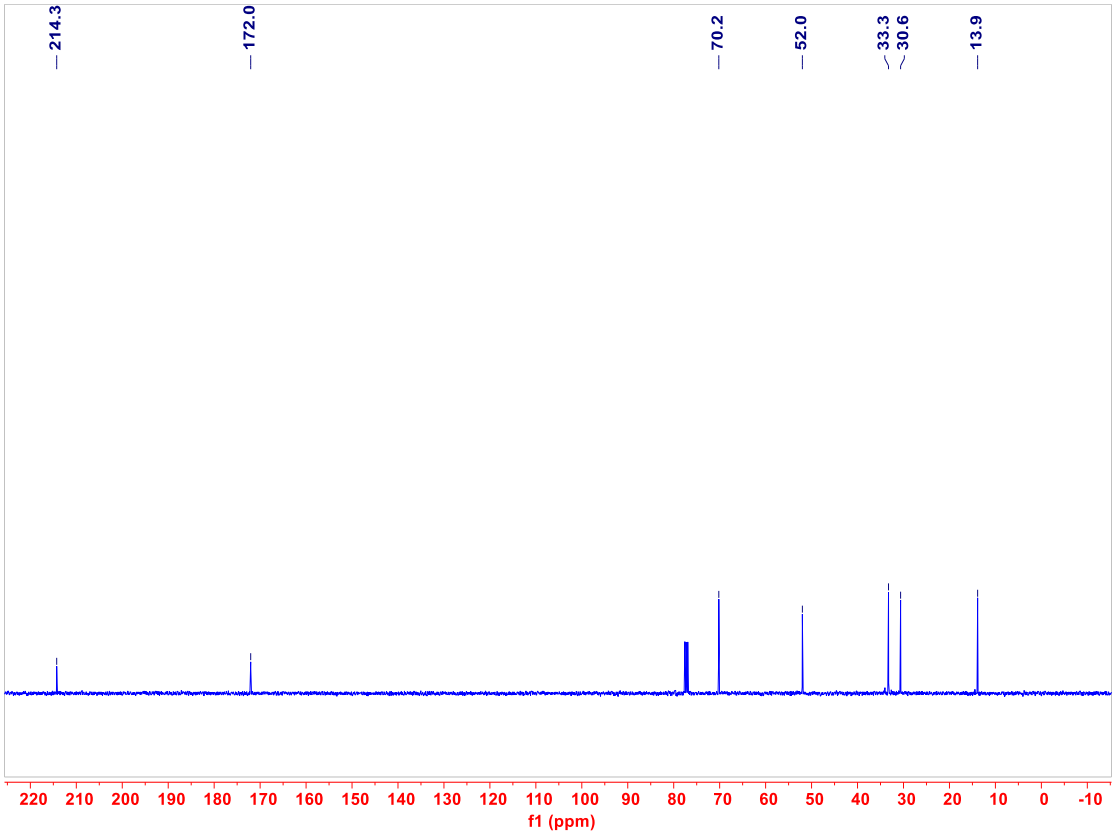
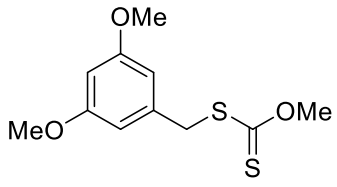
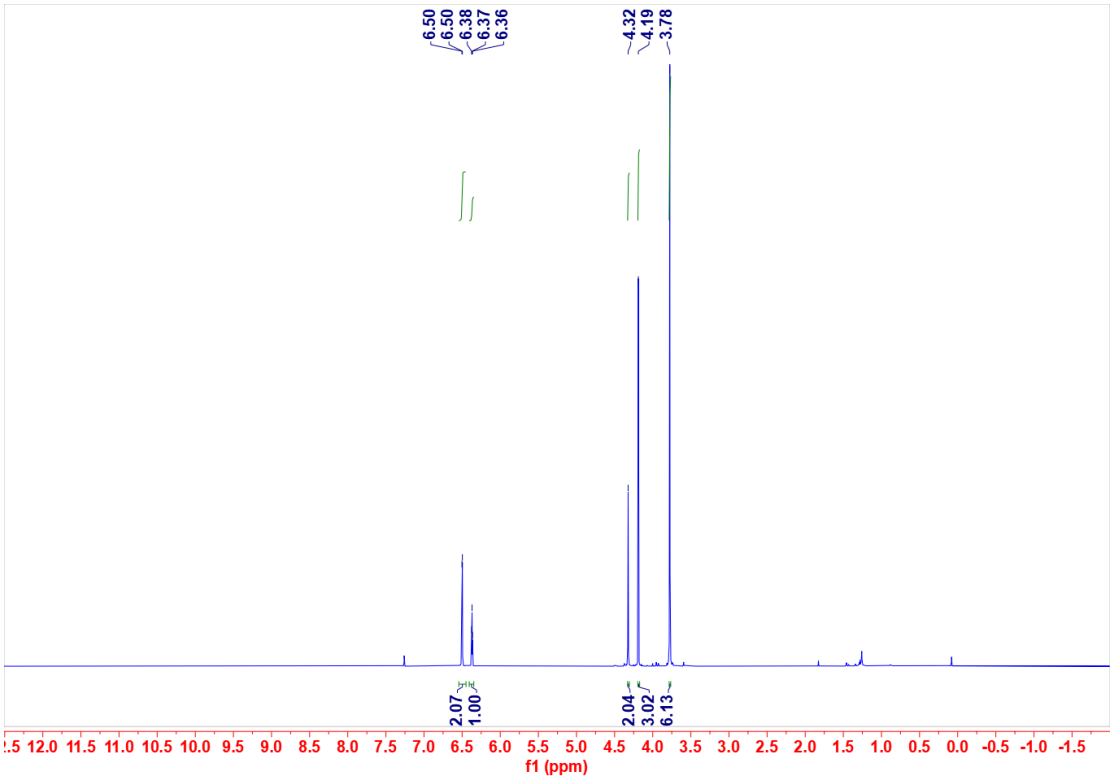
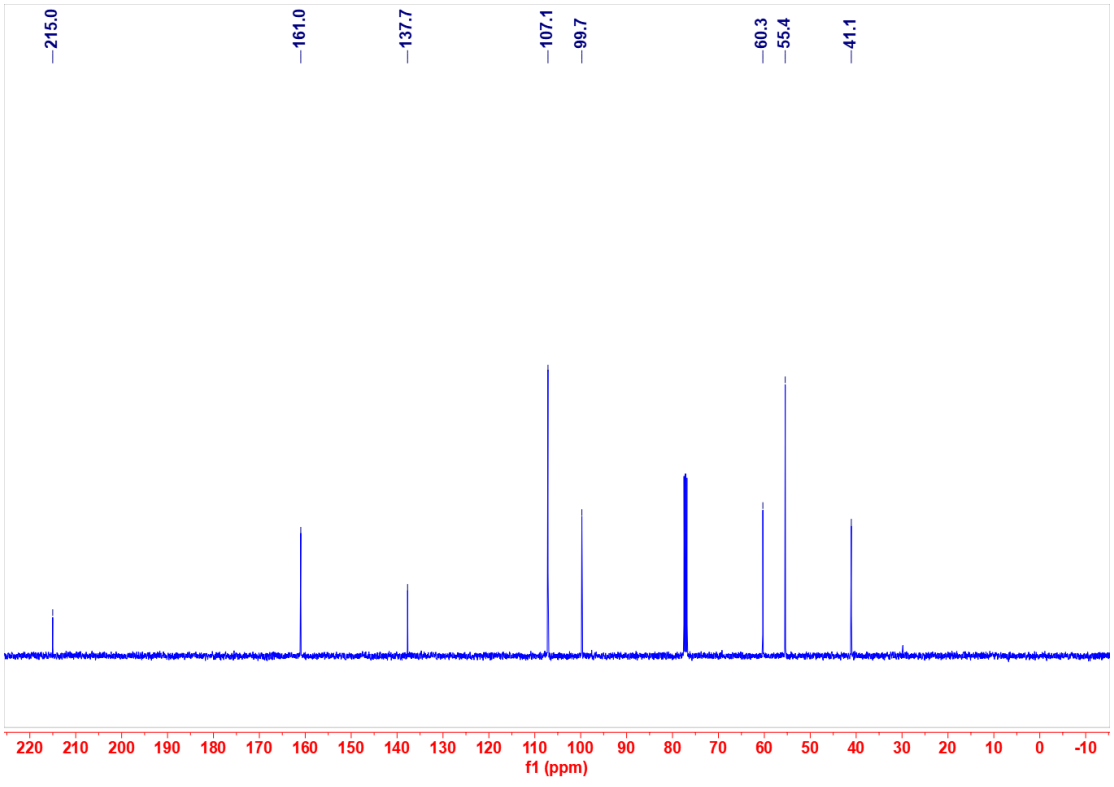
4.61
4.59
4.57
3.66
3.34
3.33
3.31
2.75
2.73
2.71
1.39
1.37
1.36
2.00
3.28
2.05
2.15
3.06
f1 (ppm)

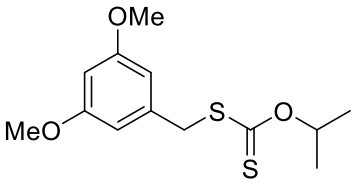

214.3
172.0
70.2
52.0
33.3
30.6
13.9
f1 (ppm)


OMe
MeO
S
OMe
S

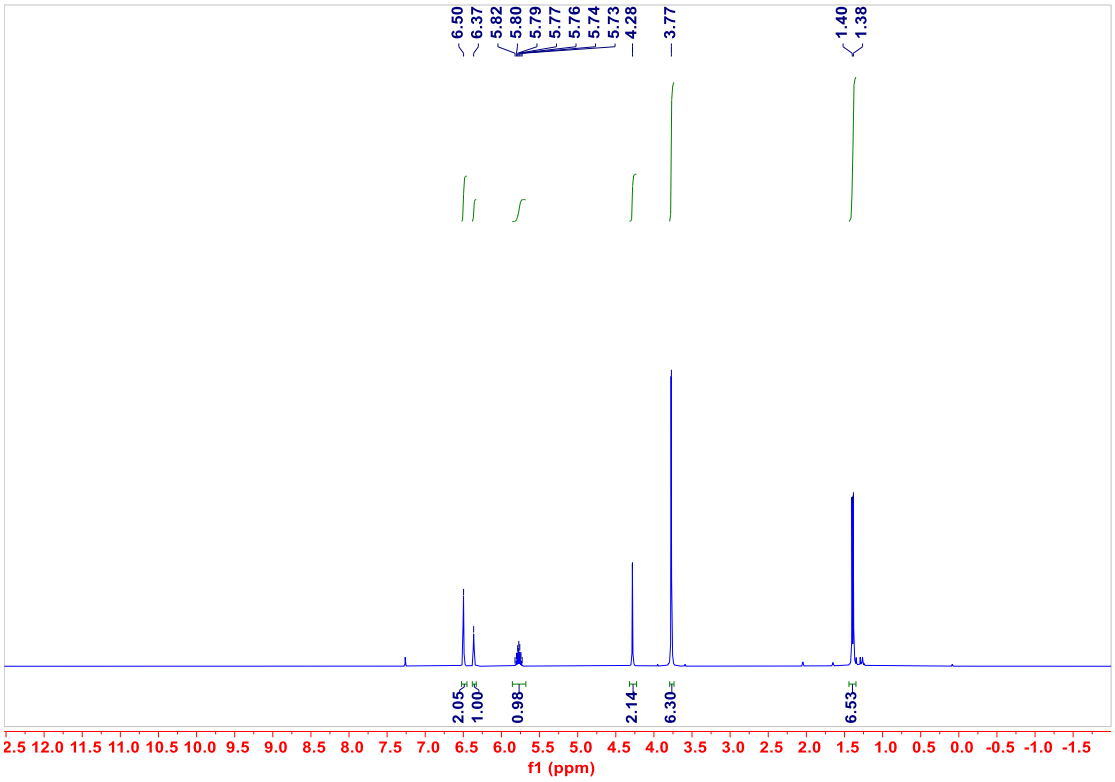
**5aa**


6.50
6.50
6.38
6.37
6.36
4.32
4.19
3.78
2.07
1.00
2.04
3.02
6.13
f1 (ppm)


215.0
161.0
137.7
107.1
99.7
60.3
55.4
41.1
f1 (ppm)


OMe
MeO
S
O
S

**5ab**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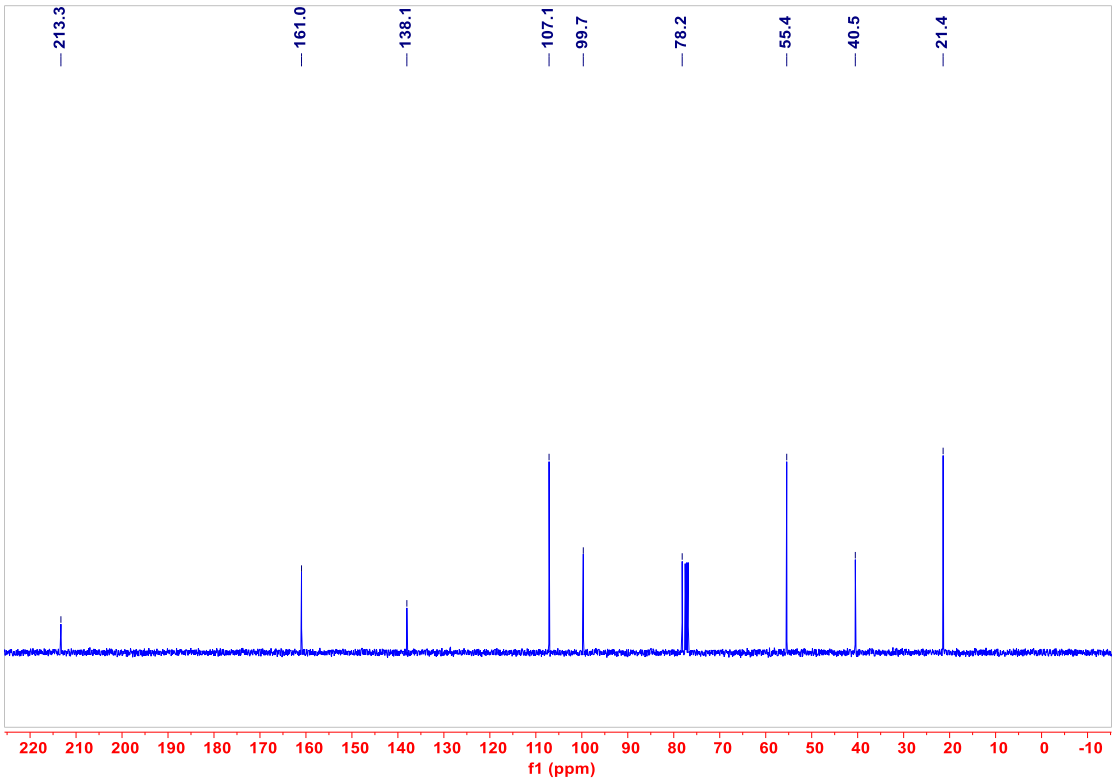
6.37
5.82
5.80
5.79
5.77
5.76
5.74
5.73
4.28
3.77
1.40
1.38
2.05
1.00
0.98
2.14
6.30
6.53
f1 (ppm)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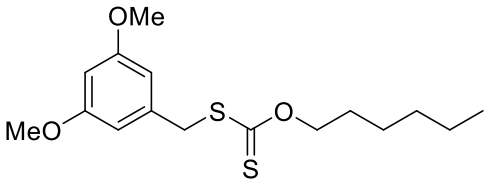
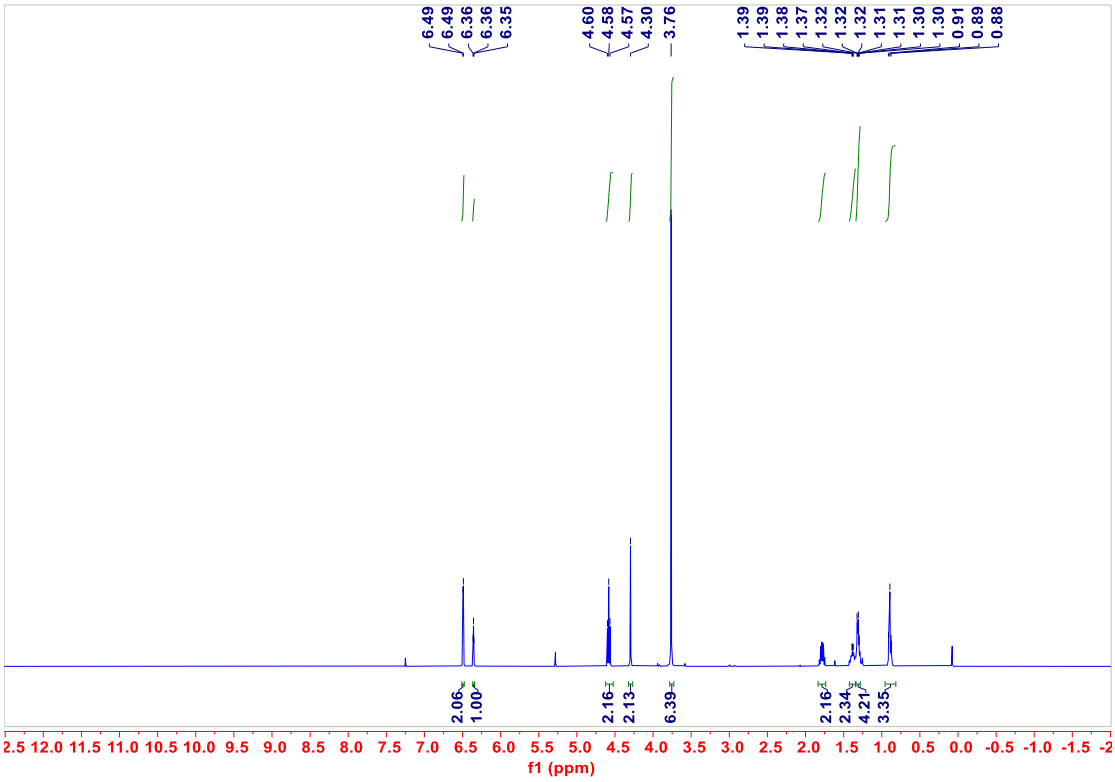
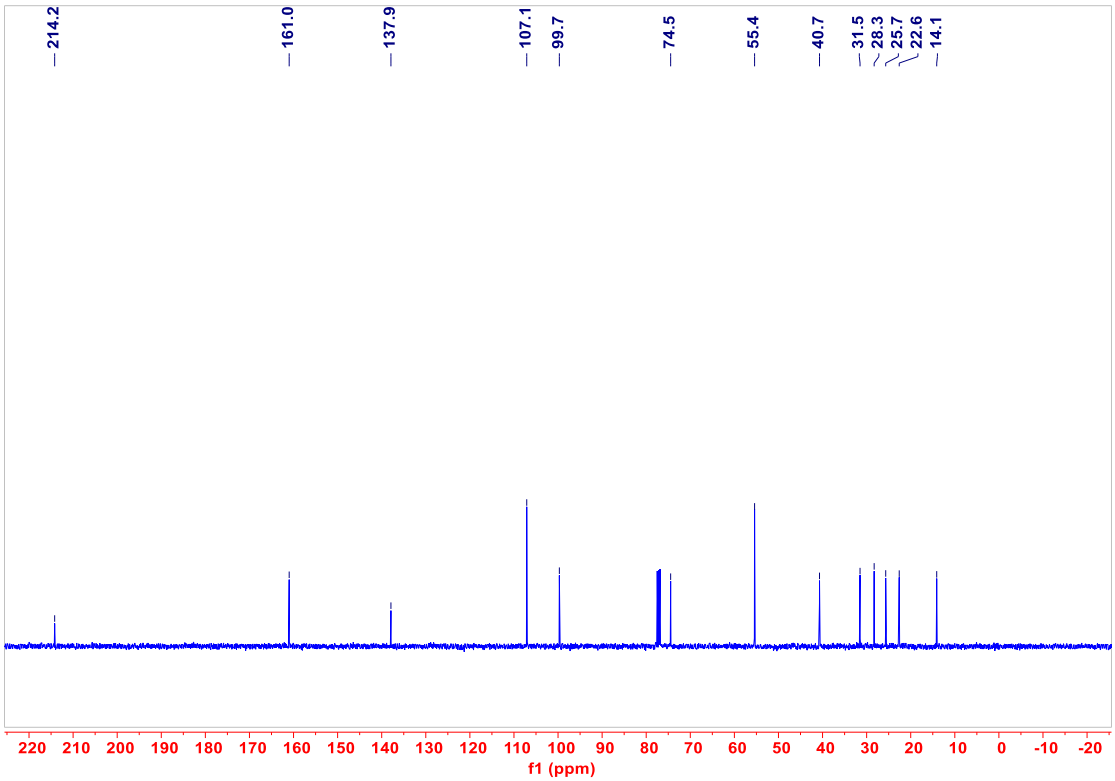
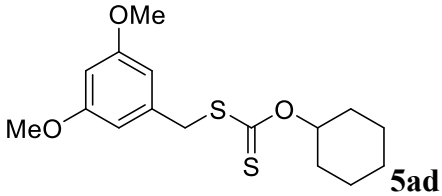
161.0
138.1
107.1
99.7
78.2
55.4
40.5
21.4
f1 (ppm)


OMe
MeO
S
O
S

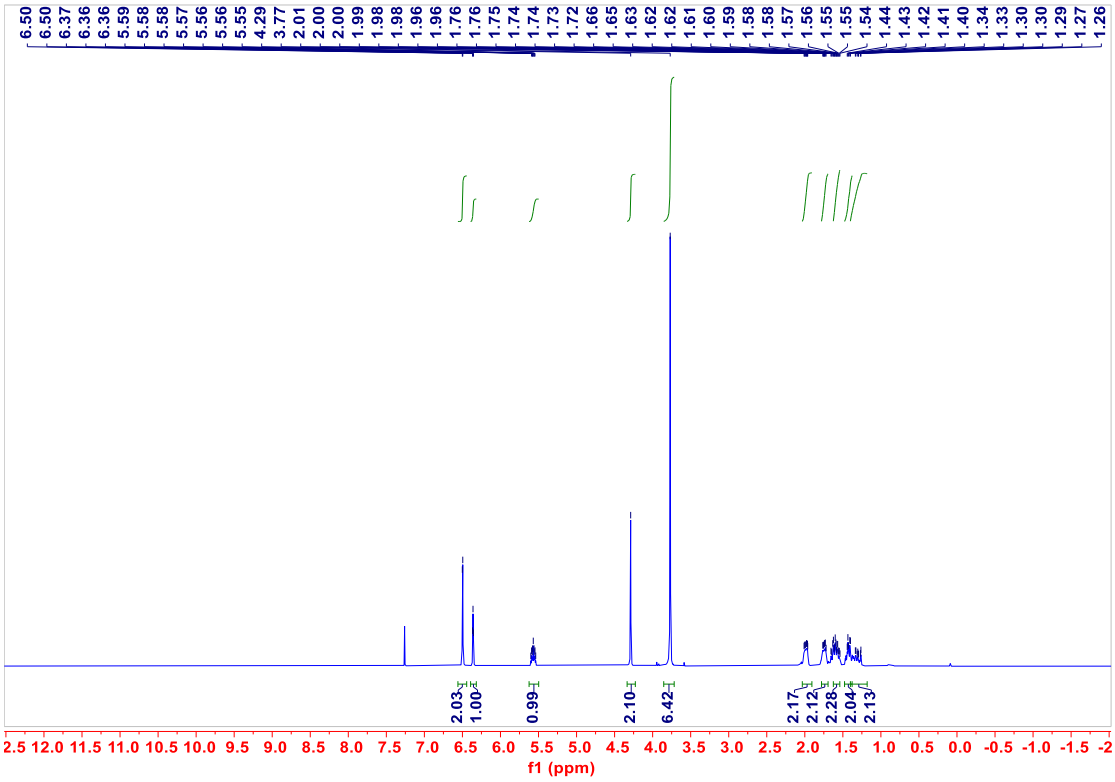
**5ac**

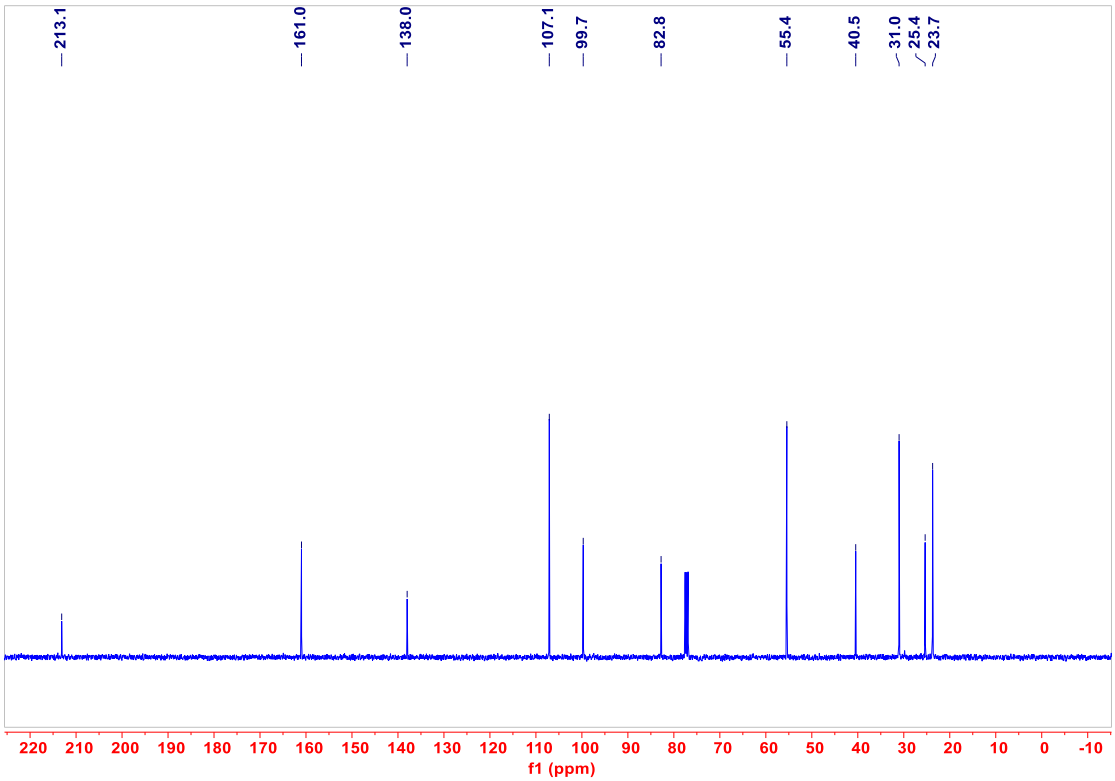

6.49
6.49
6.36
6.36
6.35
4.60
4.58
4.57
4.30
3.76
1.39
1.39
1.38
1.37
1.32
1.32
1.32
1.31
1.31
1.30
1.30
0.91
0.89
0.88
2.06
1.00
2.16
2.13
6.39
2.16
2.34
4.21
3.35
f1 (ppm)

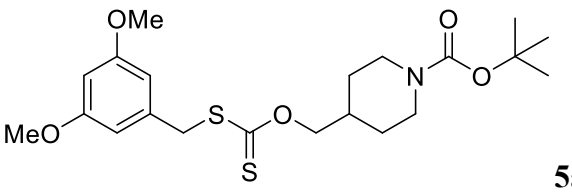

214.2
161.0
137.9
107.1
99.7
74.5
55.4
40.7
31.5
28.3
25.7
22.6
14.1
f1 (ppm)


OMe
MeO
S
O
S

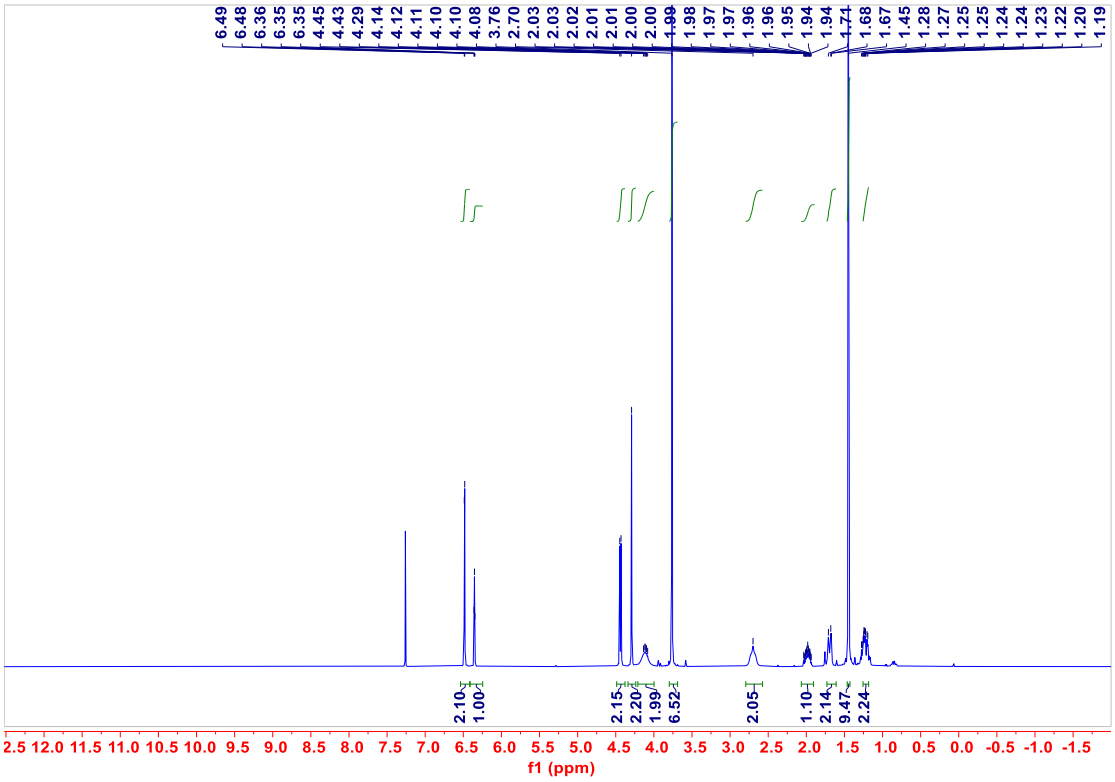
**5ad**

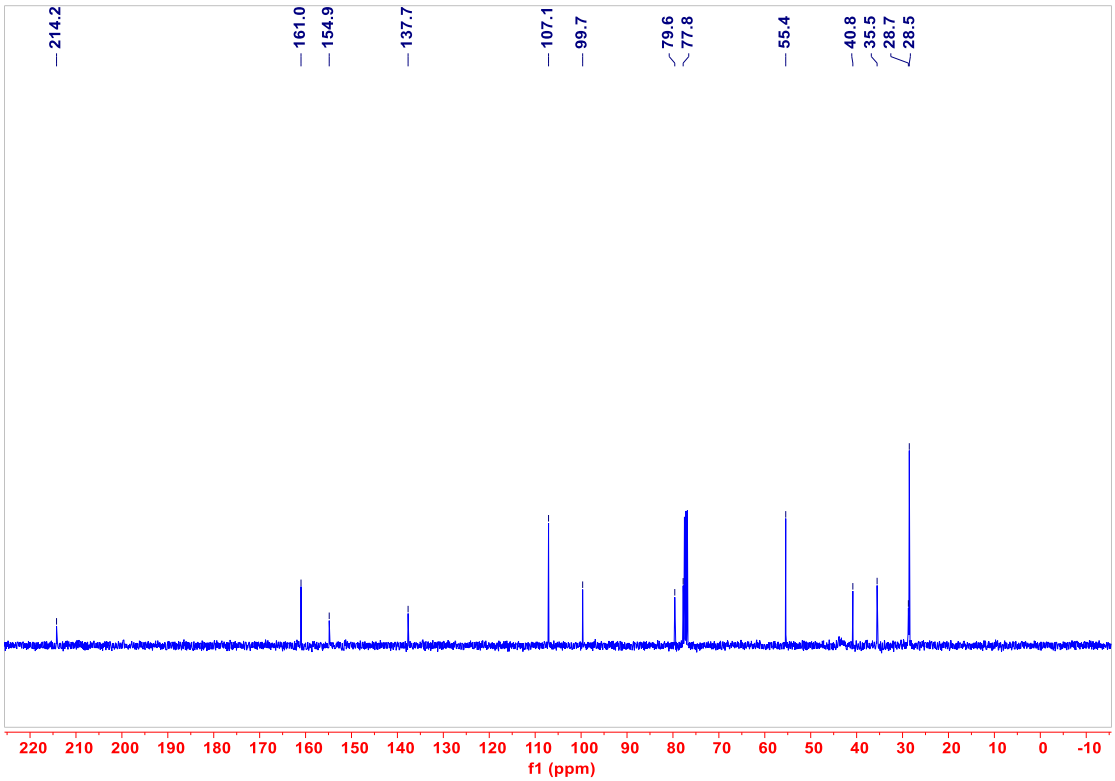

6.50 6.50 6.37 6.36 6.36 5.59 5.58 5.58 5.57 5.56 5.56 5.55 4.29 3.77 2.01 2.00 2.00 1.99 1.98 1.98 1.96 1.96 1.76 1.76 1.75 1.74 1.74 1.73 1.72 1.66 1.65 1.63 1.62 1.62 1.61 1.60 1.59 1.58 1.58 1.57 1.56 1.55 1.55 1.54 1.44 1.43 1.42 1.41 1.40 1.34 1.33 1.30 1.30 1.29 1.27 1.26
2.03 1.00 0.99 2.10 6.42 2.17 2.12 2.28 2.04 2.13
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

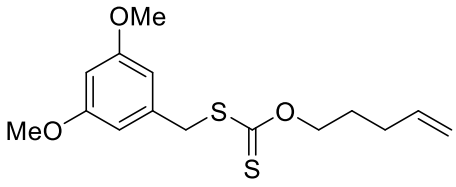

213.1
161.0
138.0
107.1
99.7
82.8
55.4
40.5
31.0
25.4
23.7
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
MeO
S
O
S
N
O
O

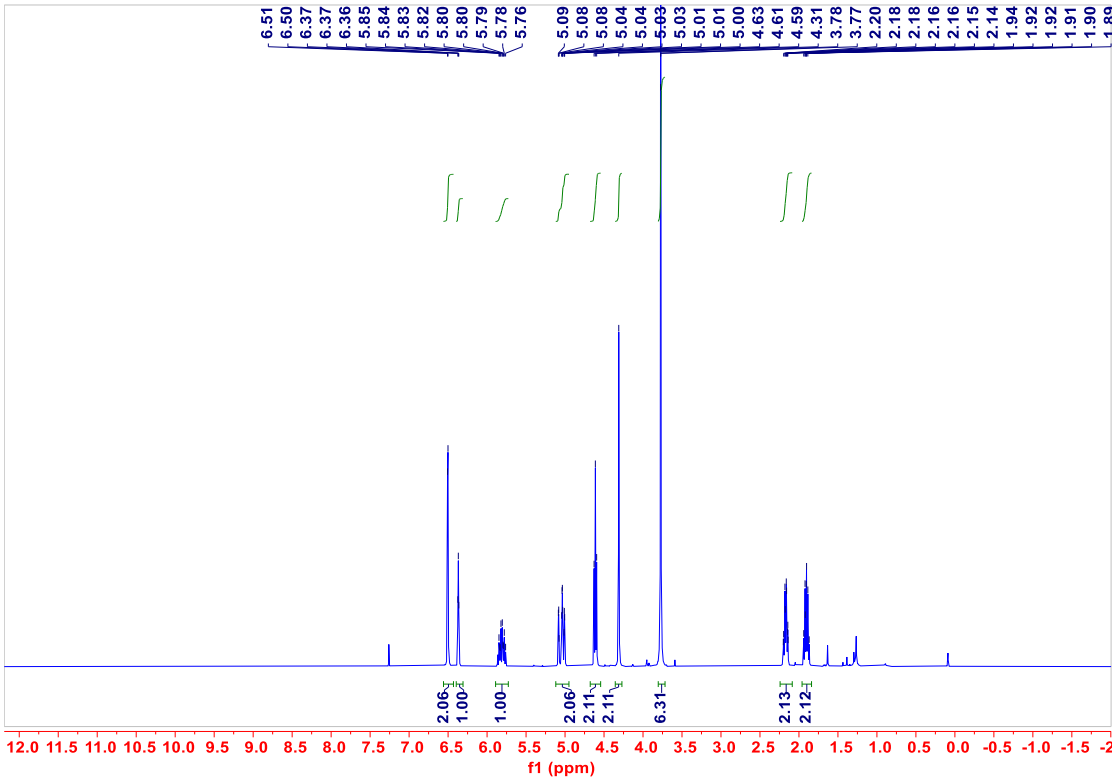
**5ae**

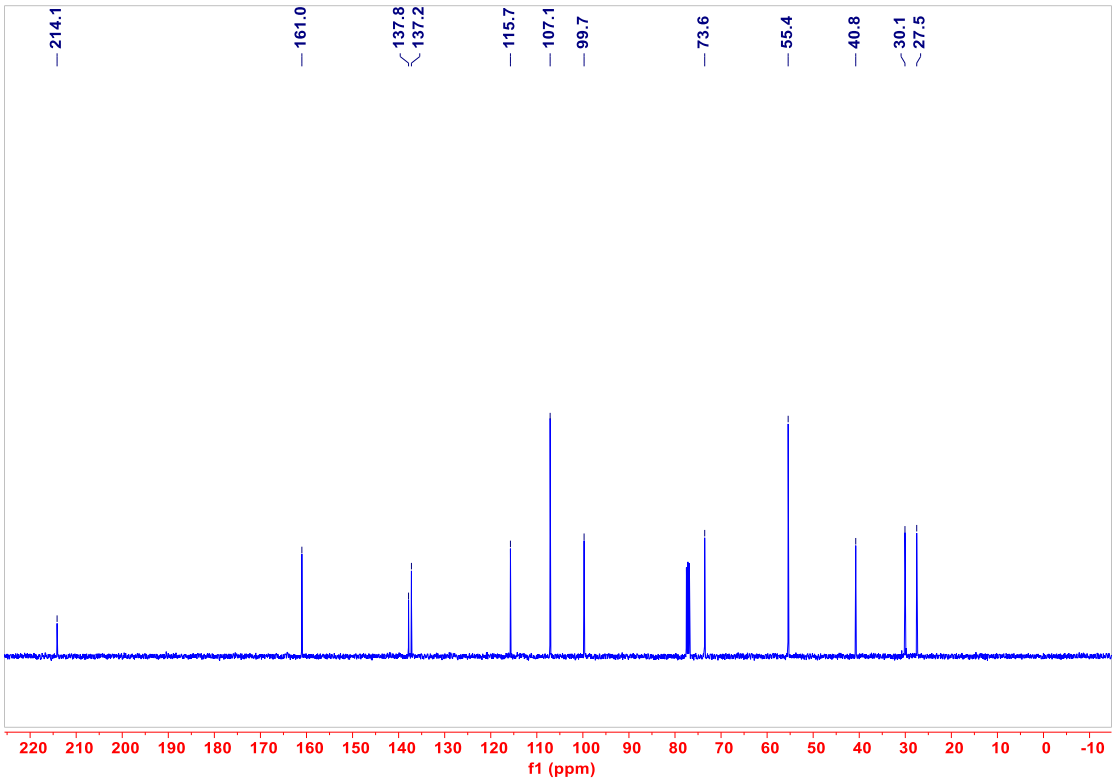

6.49 6.48 6.36 6.35 6.35 4.45 4.43 4.29 4.14 4.12 4.11 4.10 4.10 4.08 3.76 2.70 2.03 2.03 2.02 2.01 2.01 2.00 2.00 1.99 1.98 1.97 1.97 1.96 1.96 1.95 1.94 1.94 1.71 1.68 1.67 1.45 1.28 1.27 1.25 1.25 1.24 1.24 1.23 1.22 1.20 1.19
2.10 1.00 2.15 2.20 1.99 6.52 2.05 1.10 2.14 9.47 2.24
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


214.2
161.0 154.9 137.7 107.1 99.7 79.6 77.8 55.4 40.8 35.5 28.7 28.5
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
MeO
S
O
S

**5af**


6.51 6.50 6.37 6.37 6.36 5.85 5.84 5.83 5.82 5.80 5.80 5.79 5.78 5.76
5.09 5.08 5.08 5.04 5.04 5.03 5.03 5.01 5.01 5.00 4.63 4.61 4.59 4.31 3.78 3.77 2.20 2.18 2.18 2.16 2.16 2.15 2.14 1.94 1.92 1.92 1.91 1.90
2.06 1.00 1.00 2.06 2.11 2.11 6.31 2.13 2.12
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)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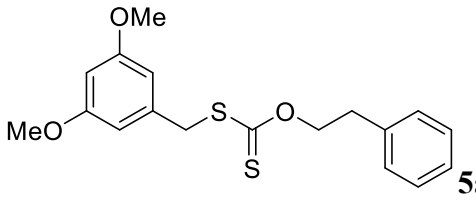
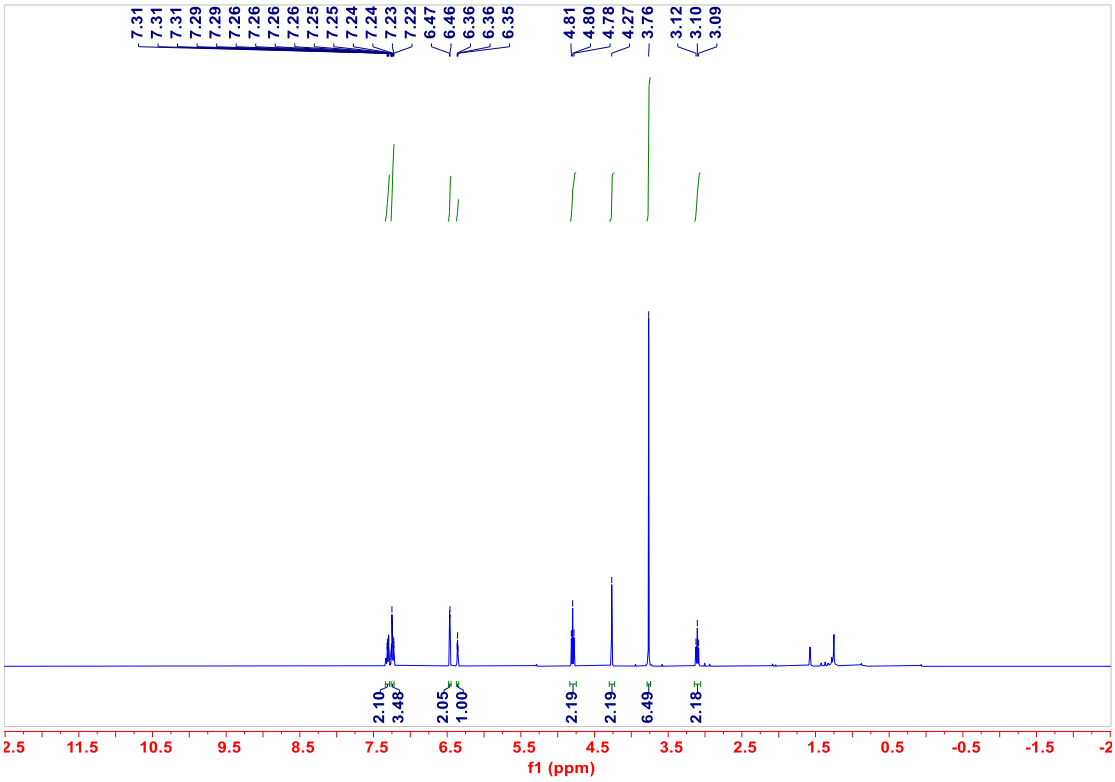
137.8
137.2
115.7
107.1
99.7
73.6
55.4
40.8
30.1
27.5
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
MeO
S
O
S

**5ag**


7.31
7.31
7.31
7.29
7.29
7.26
7.26
7.26
7.26
7.25
7.25
7.24
7.24
7.23
7.22
6.47
6.46
6.36
6.36
6.35
4.81
4.80
4.78
4.27
3.76
3.12
3.10
3.09
2.10
3.48
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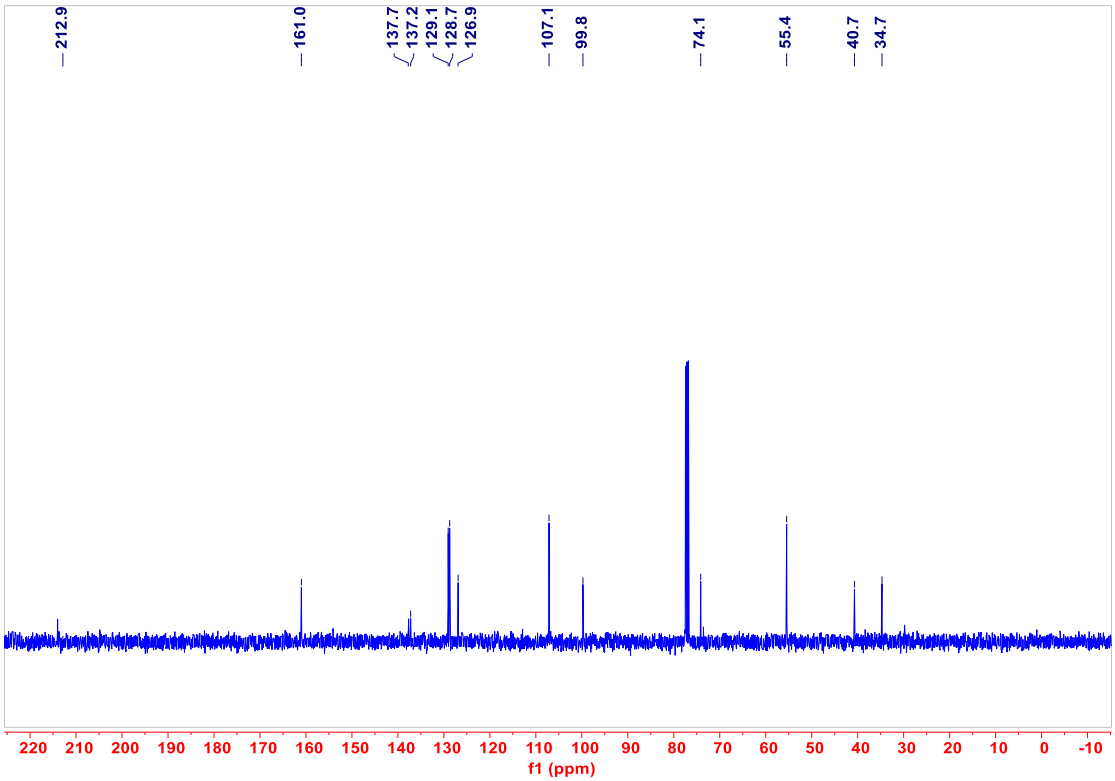
2.19
2.19
6.49
2.18
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
-2
f1 (ppm)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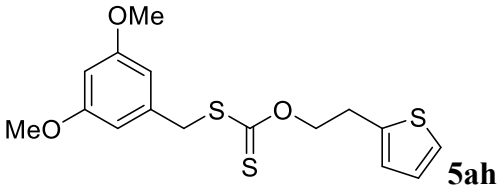
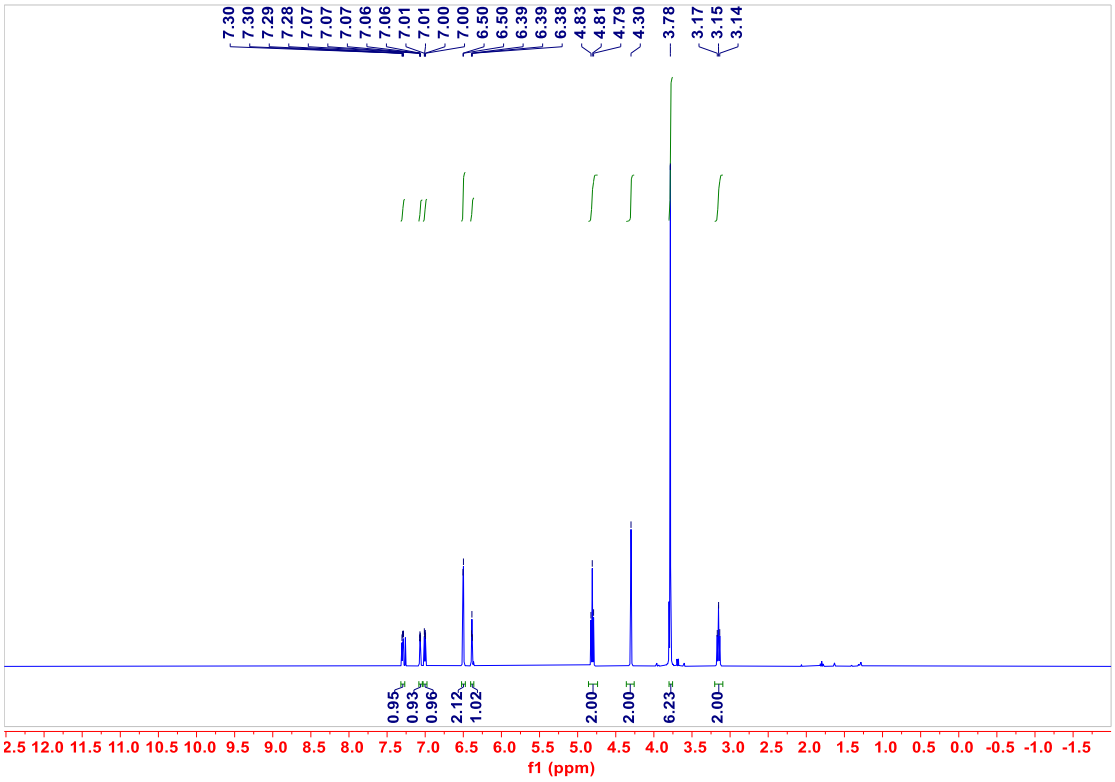
137.2
129.1
128.7
126.9
107.1
99.8
74.1
55.4
40.7
34.7
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


OMe
MeO
S
O
S
S

**5ah**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7.30
7.29
7.28
7.07
7.07
7.07
7.06
7.06
7.01
7.01
7.00
7.00
6.50
6.50
6.39
6.39
6.38
4.83
4.81
4.79
4.30
3.78
3.17
3.15
3.14
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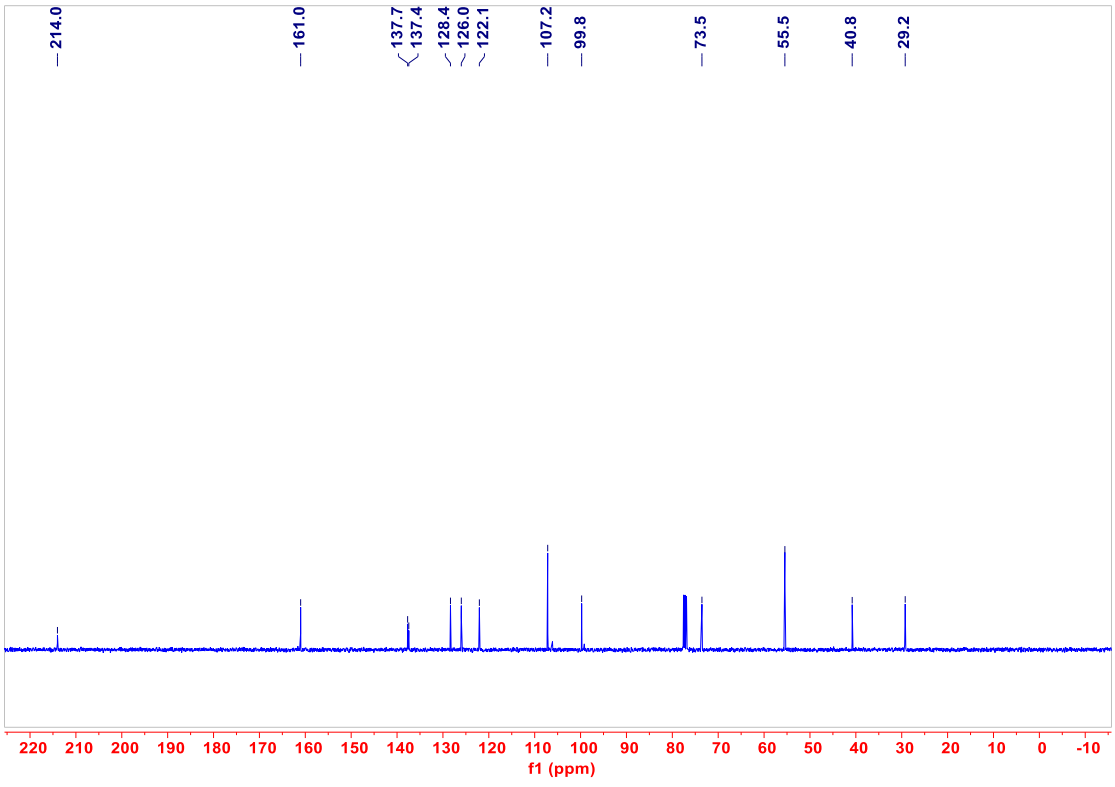
0.93
0.96
2.12
1.02
2.00
2.00
6.23
2.00
f1 (ppm)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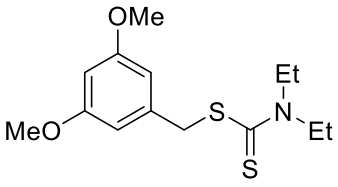
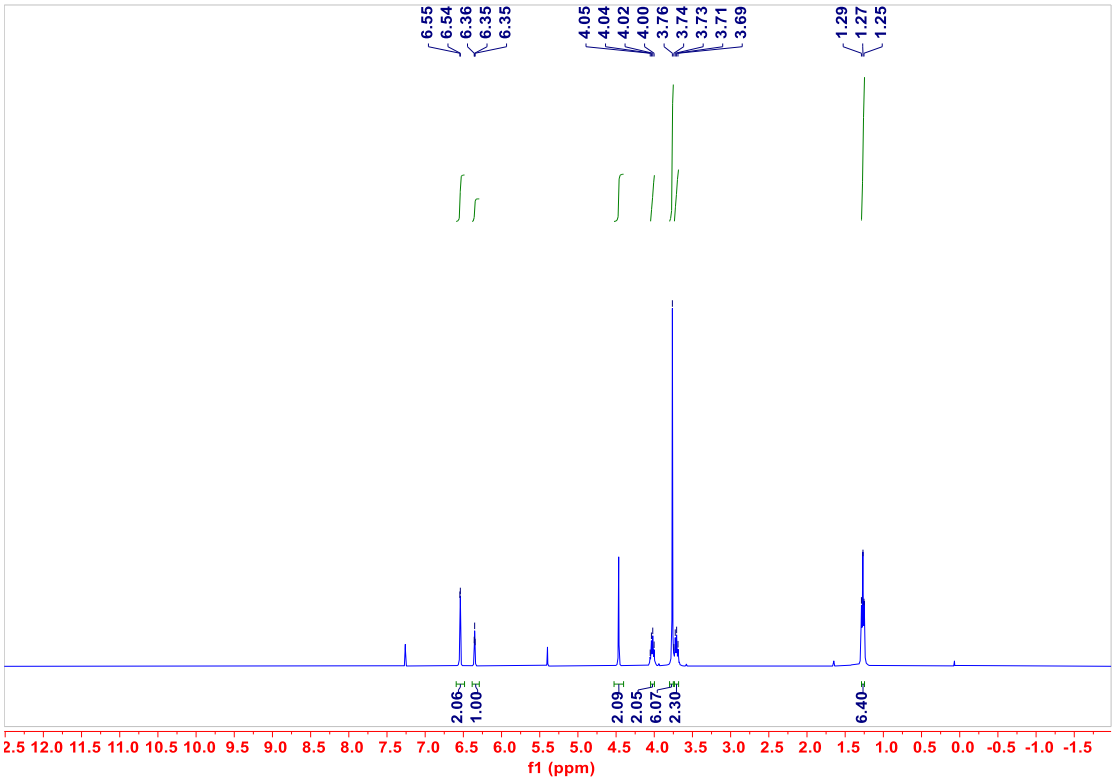
137.7
137.4
128.4
126.0
122.1
107.2
99.8
73.5
55.5
40.8
29.2
f1 (ppm)


OMe
MeO
S
N
Et
Et
S

**5ai**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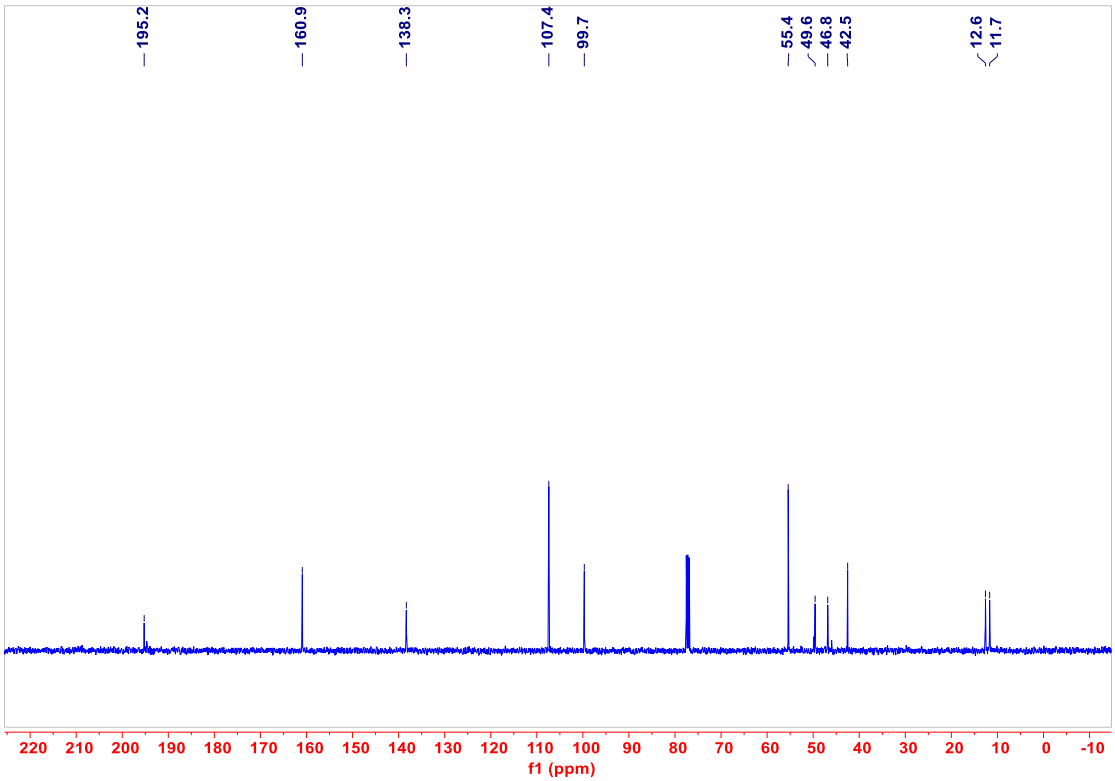
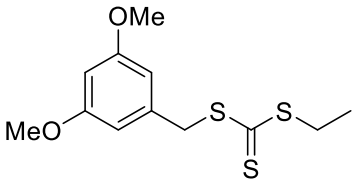
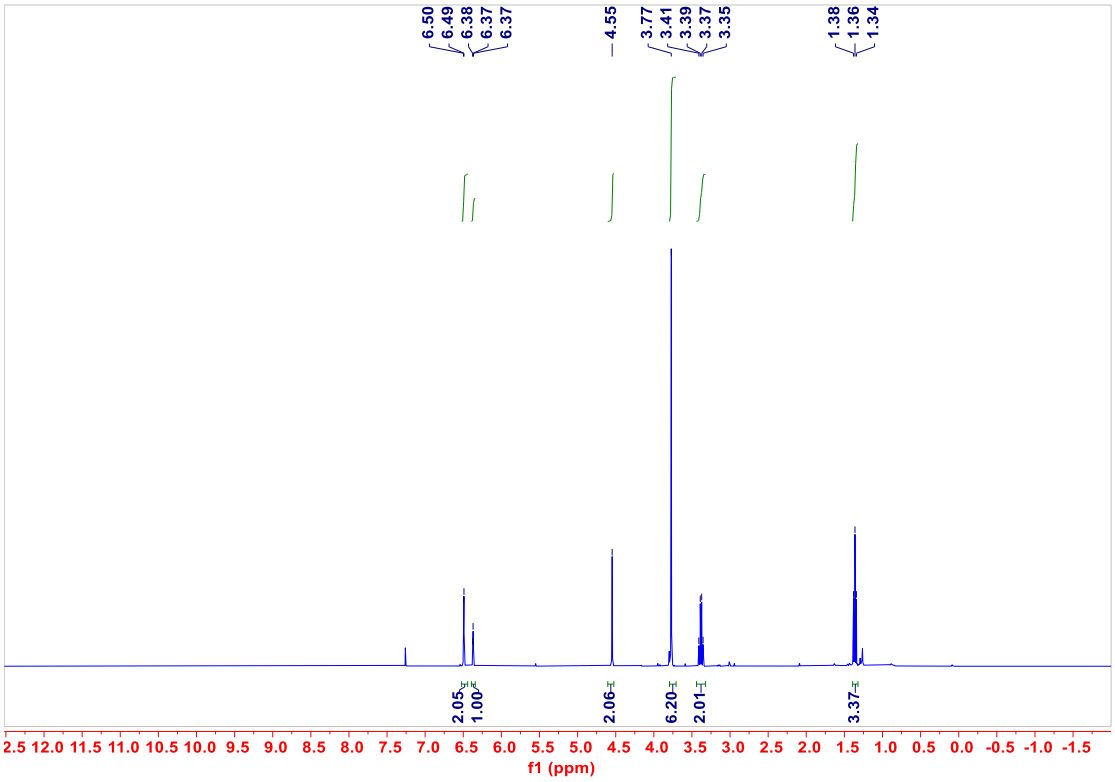
6.35
4.05
4.04
4.02
4.00
3.76
3.74
3.73
3.71
3.69
1.29
1.27
1.25
2.06
1.00
2.09
2.05
6.07
2.30
6.40
f1 (ppm)


195.2
160.9
138.3
107.4
99.7
55.4
49.6
46.8
42.5
12.6
11.7
f1 (ppm)


OMe
MeO
S
S
S

**5aj**


6.50
6.49
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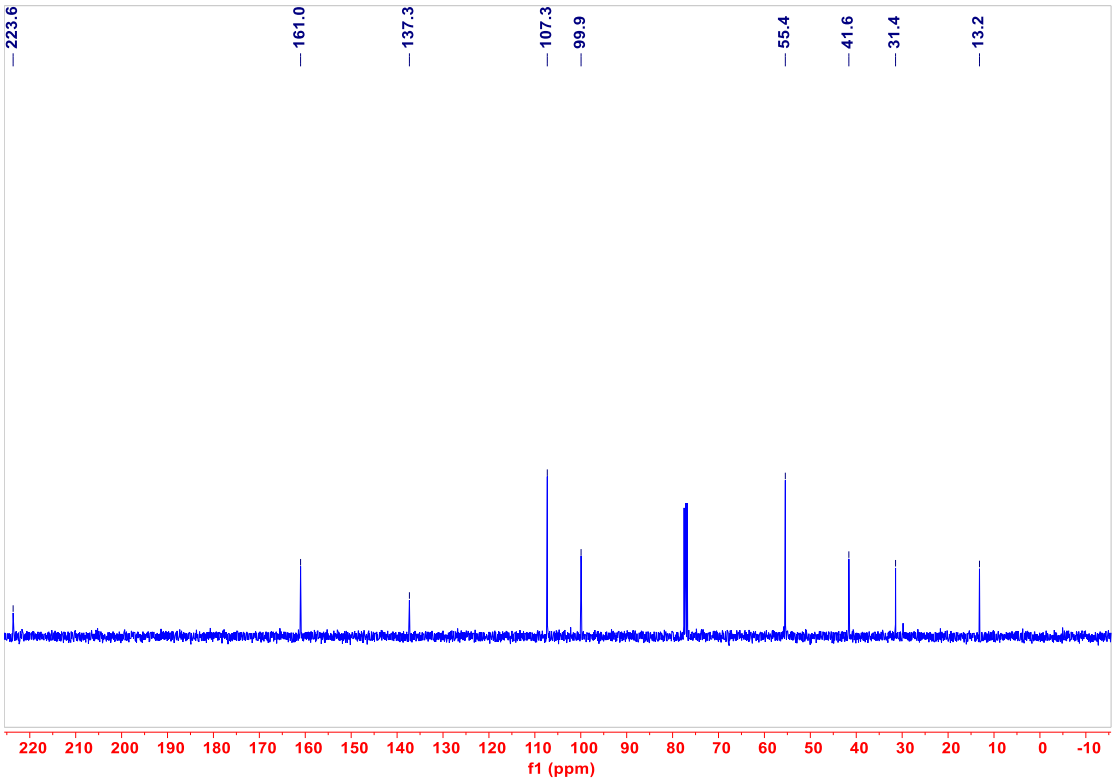
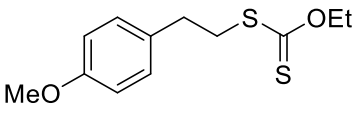
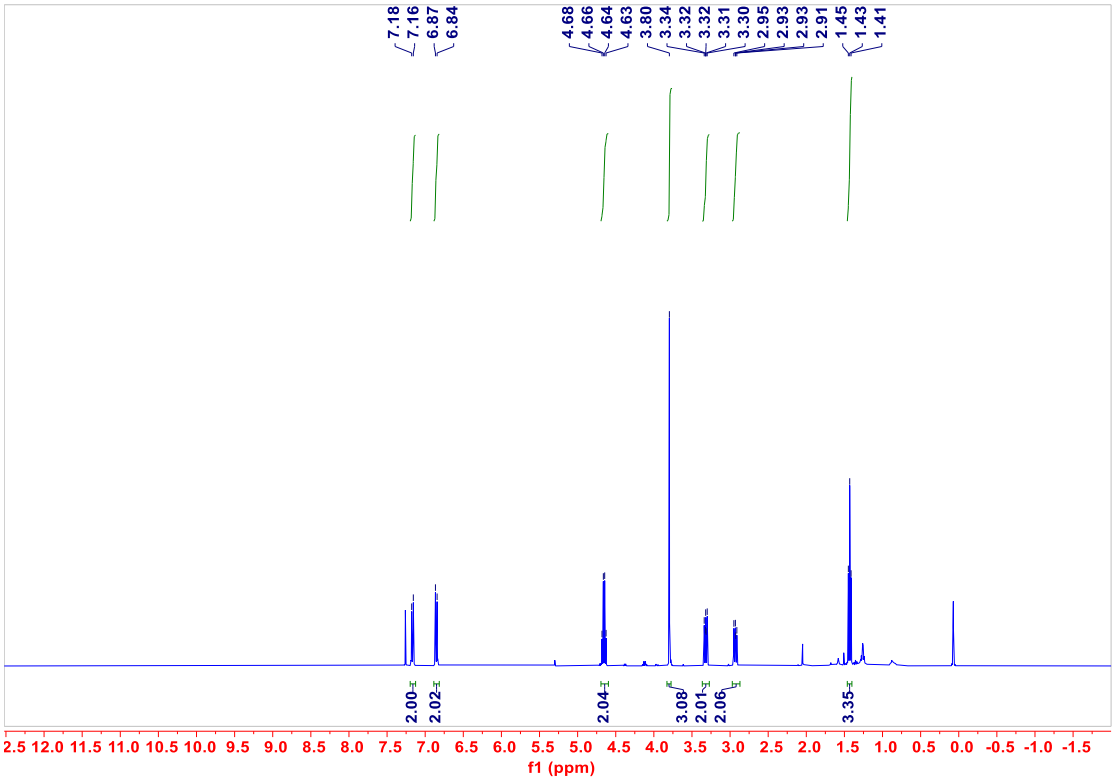
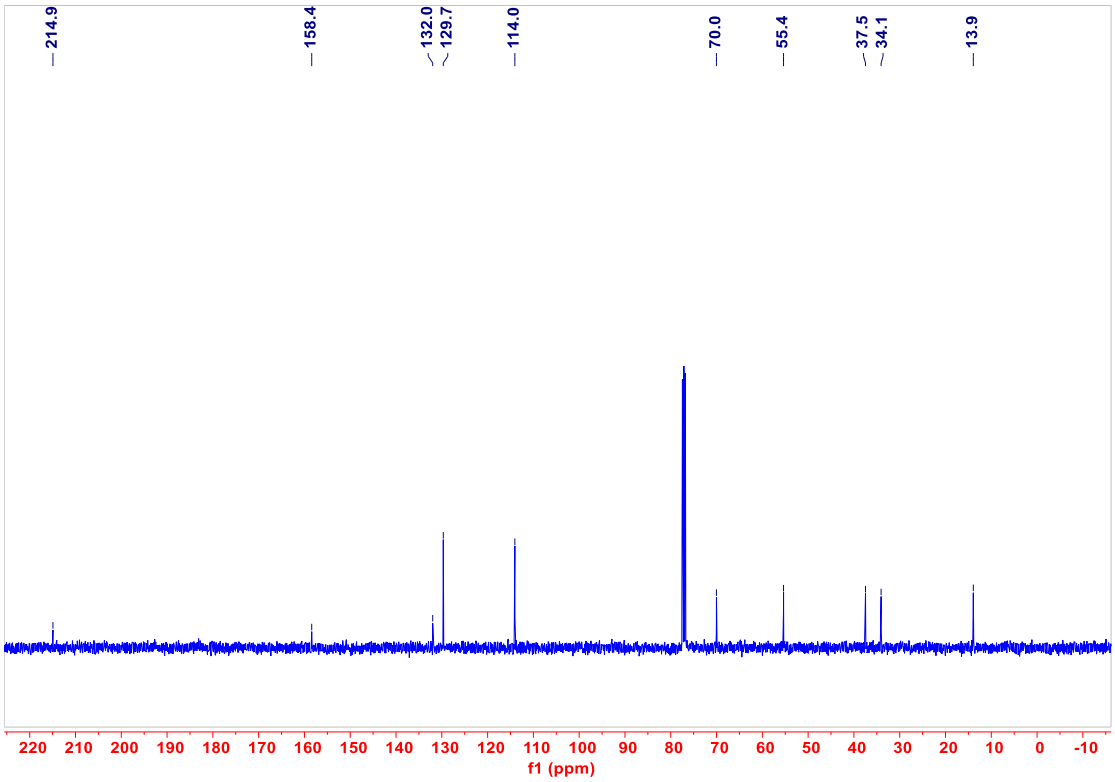
6.37
6.37
4.55
3.77
3.41
3.39
3.37
3.35
1.38
1.36
1.34
2.05
1.00
2.06
6.20
2.01
3.37
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

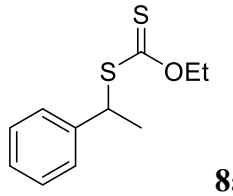

223.6
161.0
137.3
107.3
99.9
55.4
41.6
31.4
13.2
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
OEt
S
MeO

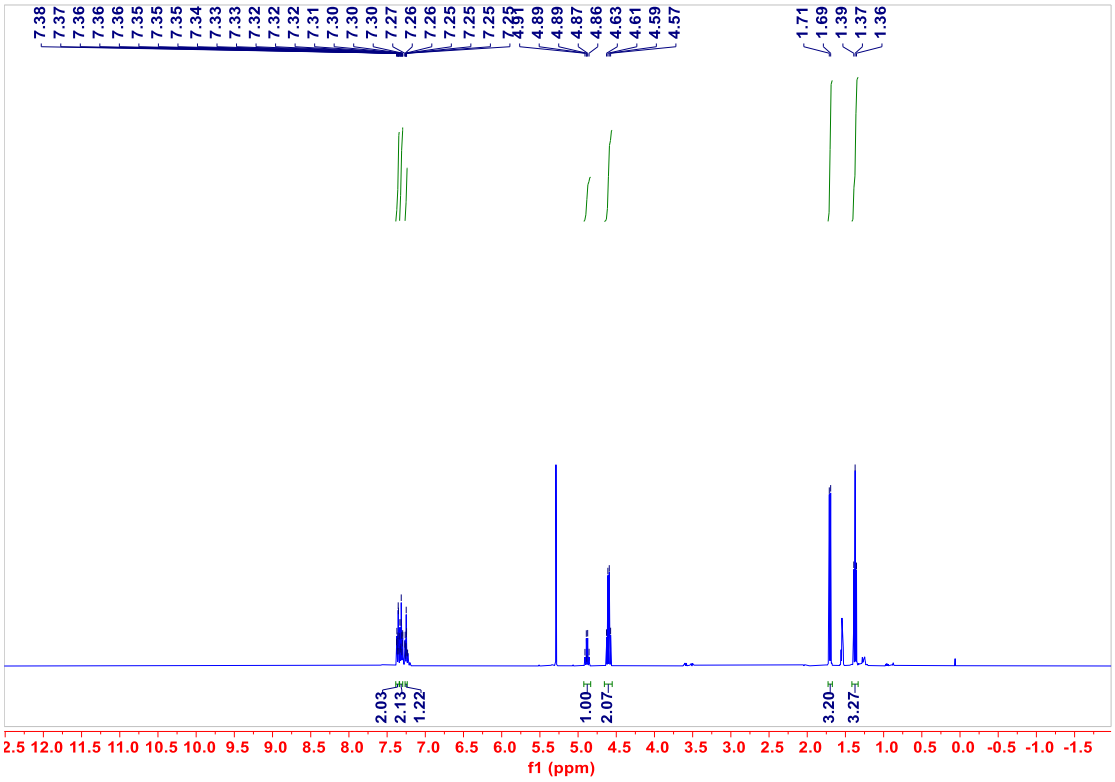
**8aa**

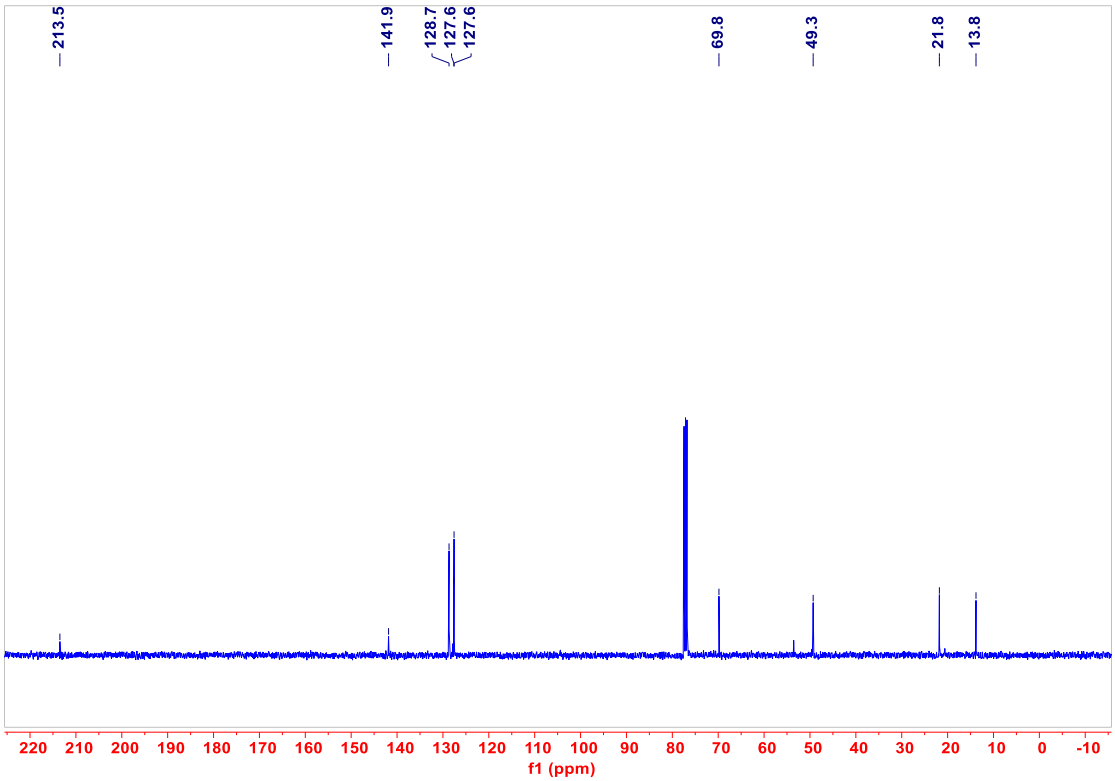

7.18
7.16
6.87
6.84
4.68
4.66
4.64
4.63
3.80
3.34
3.32
3.32
3.31
3.30
2.95
2.93
2.93
2.91
1.45
1.43
1.41
2.00
2.02
2.04
3.08
2.01
2.06
3.35
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

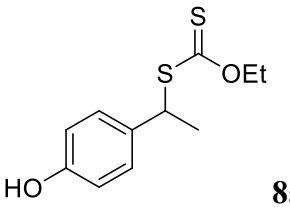

214.9
158.4
132.0
129.7
114.0
70.0
55.4
37.5
34.1
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


S
S
OEt

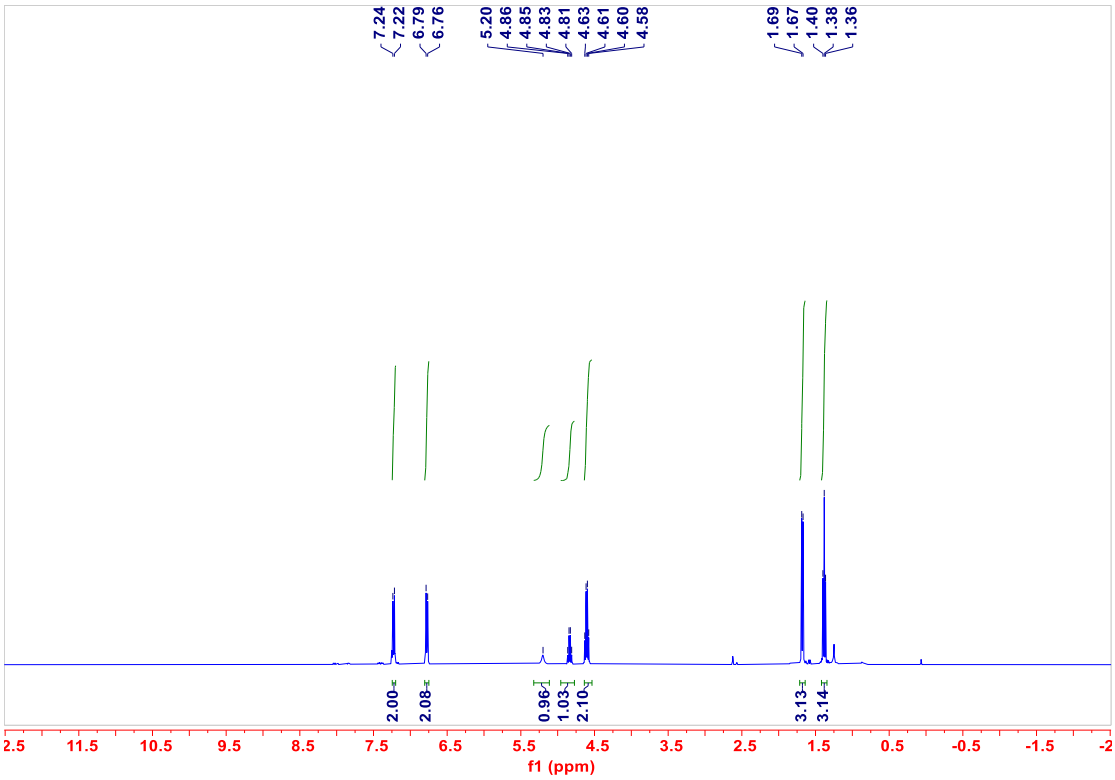
**8ab**

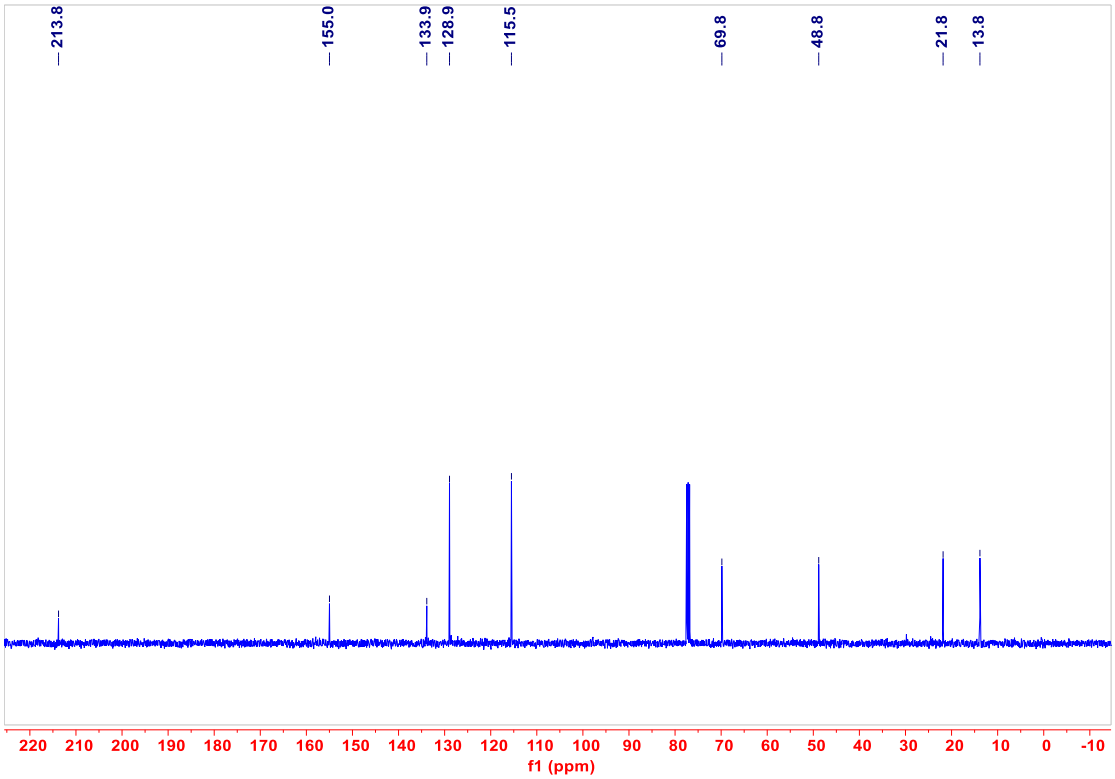

7.38 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.33 7.33 7.32 7.32 7.32 7.31 7.30 7.30 7.30 7.27 7.26 7.26 7.25 7.25 7.25 4.91 4.89 4.89 4.87 4.86 4.63 4.61 4.59 4.57
1.71 1.69 1.39 1.37 1.36
2.03 2.13 1.22 1.00 2.07 3.20 3.27
f1 (ppm)

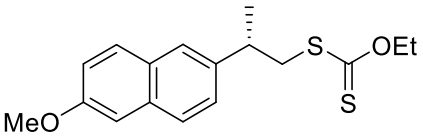

213.5 141.9 128.7 127.6 127.6 69.8 49.3 21.8 13.8
f1 (ppm)


S
S
OEt
HO

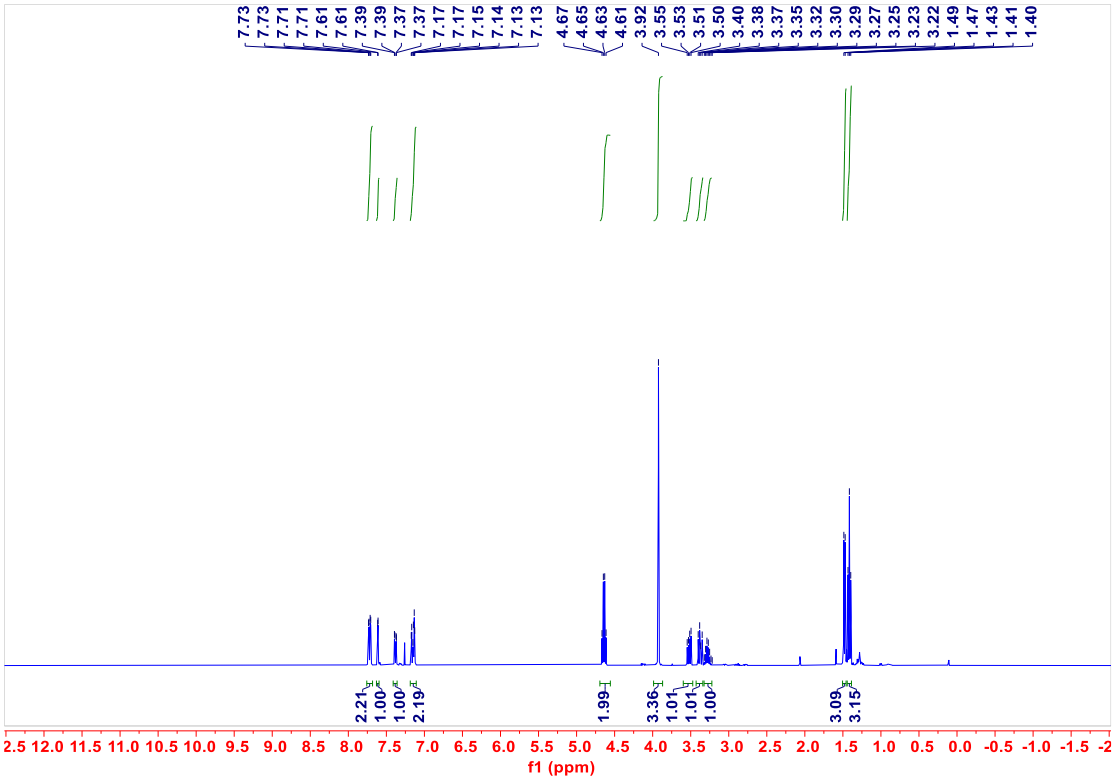
**8ac**

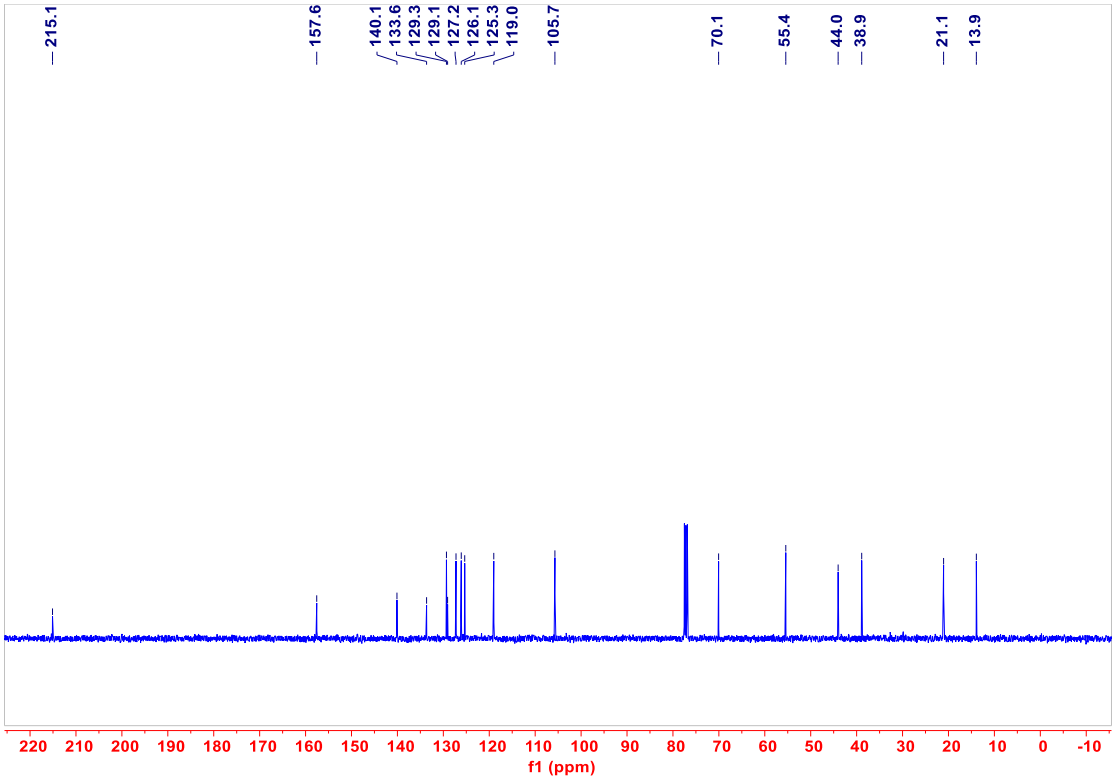

7.24
7.22
6.79
6.76
5.20
4.86
4.85
4.83
4.81
4.63
4.61
4.60
4.58
1.69
1.67
1.40
1.38
1.36
2.00
2.08
0.96
1.03
2.10
3.13
3.14
f1 (ppm)

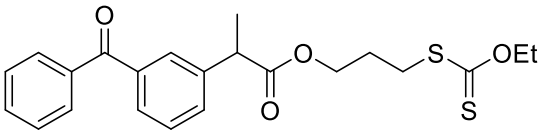

213.8
155.0
133.9
128.9
115.5
69.8
48.8
21.8
13.8
f1 (ppm)


MeO
S
OEt
S

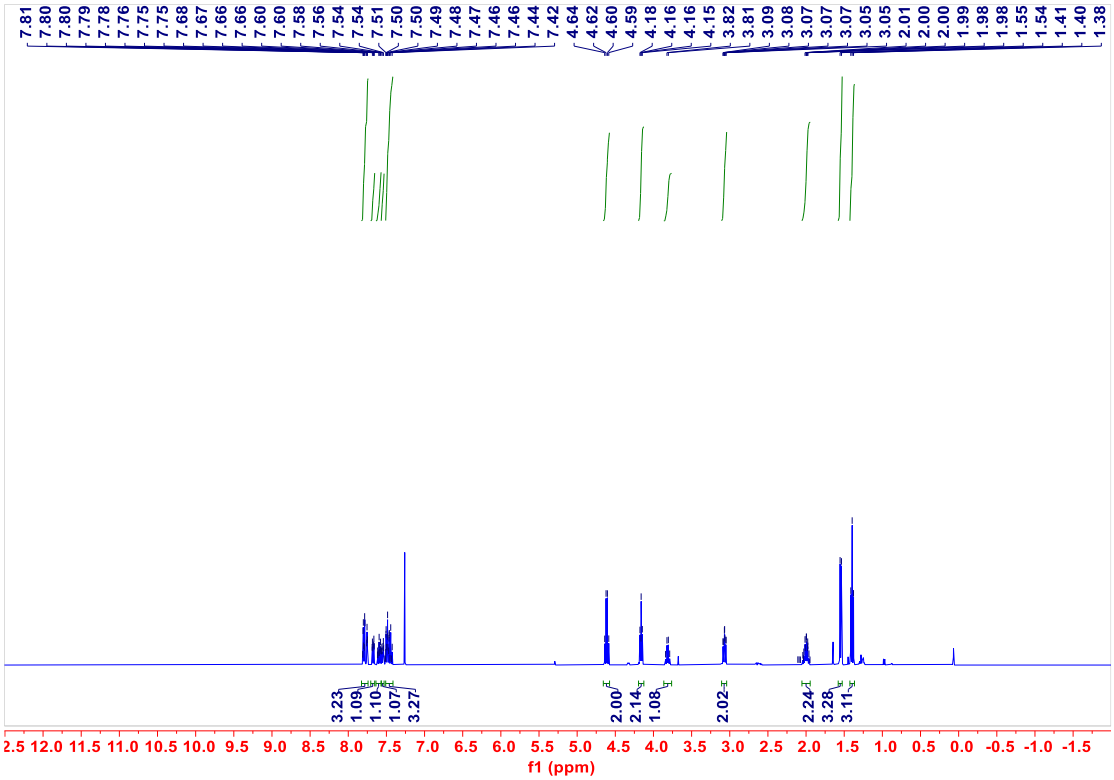
**10aa**

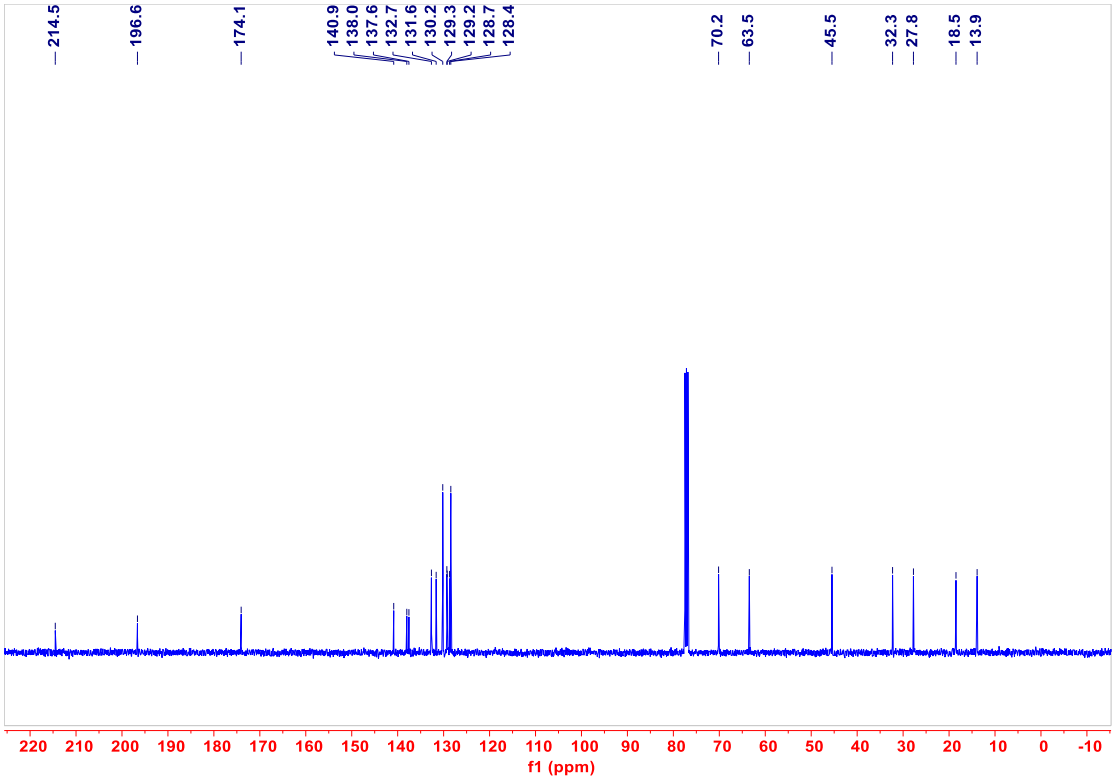

7.73 7.73 7.71 7.71 7.61 7.61 7.39 7.39 7.37 7.37 7.17 7.17 7.15 7.14 7.13 7.13
4.67 4.65 4.63 4.61 3.92 3.55 3.53 3.51 3.50 3.40 3.38 3.37 3.35 3.32 3.30 3.29 3.27 3.25 3.23 3.22 1.49 1.47 1.43 1.41 1.40
2.21 1.00 1.00 2.19 1.99 3.36 1.01 1.01 1.00 3.09 3.15
f1 (ppm)

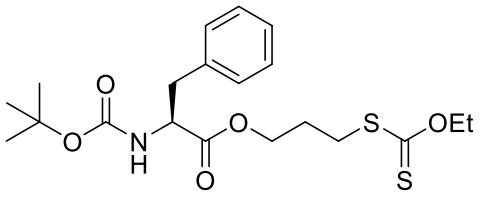

215.1 157.6 140.1 133.6 129.3 129.1 127.2 126.1 125.3 119.0 105.7 70.1 55.4 44.0 38.9 21.1 13.9
f1 (ppm)


O
O
O
S
OEt
S

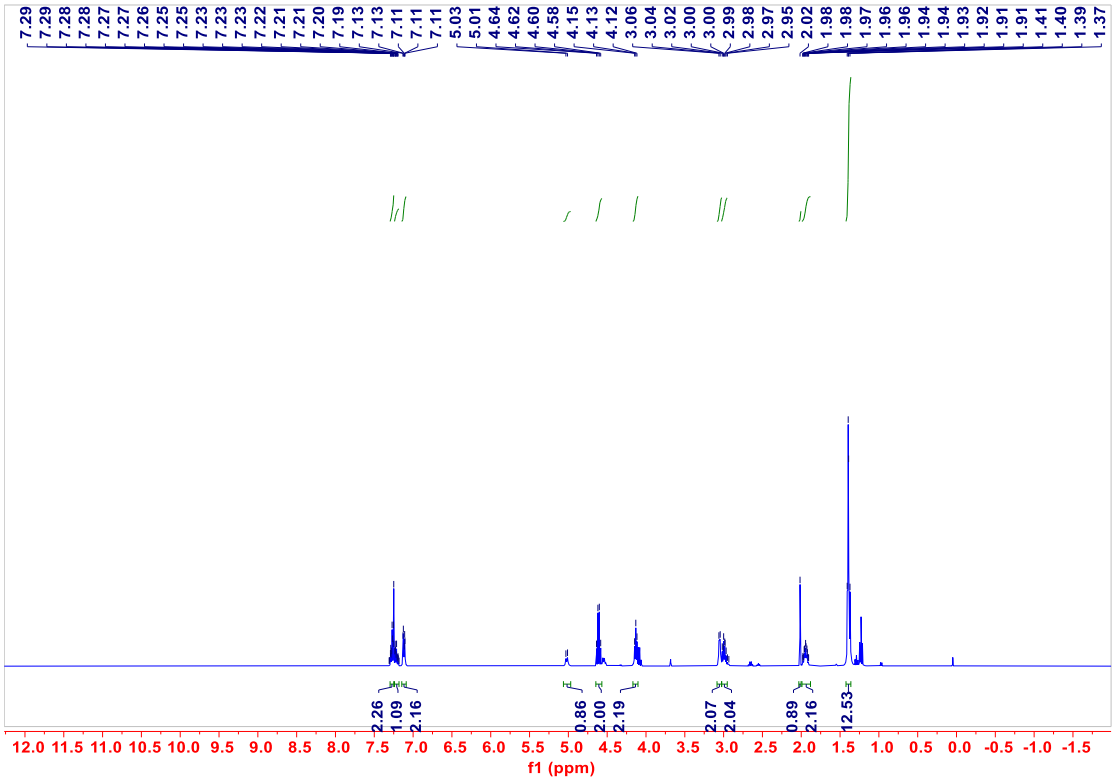
**10ab**

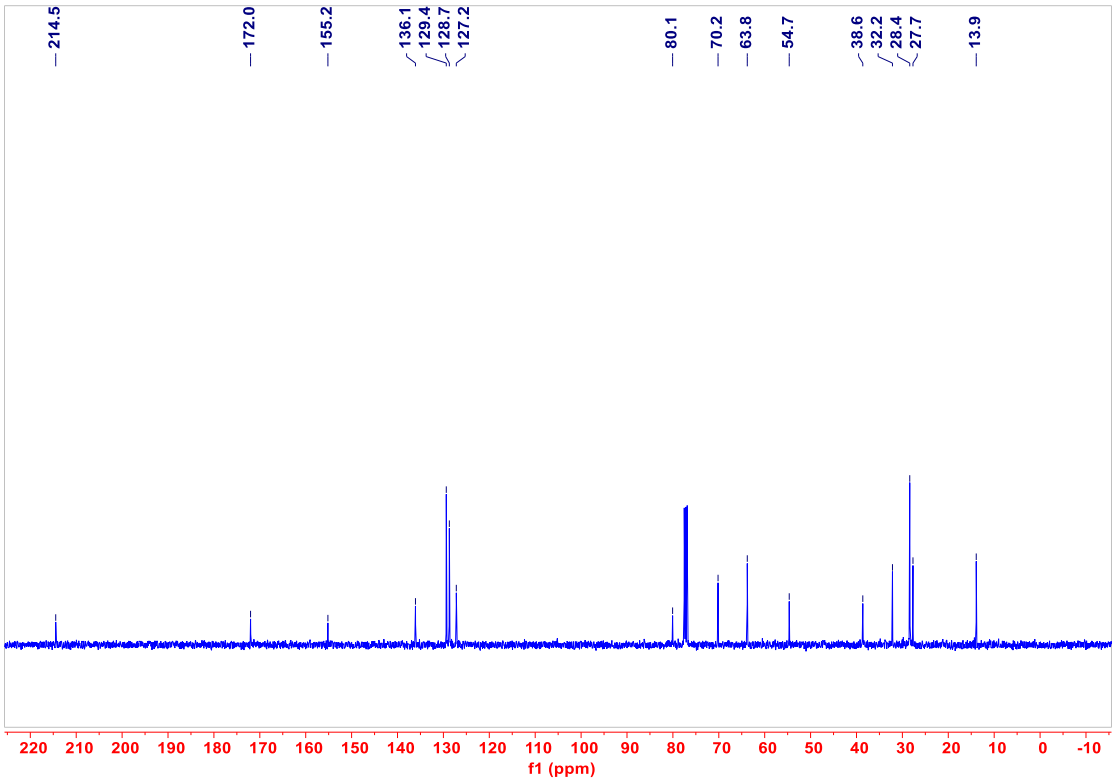

7.81 7.80 7.80 7.79 7.78 7.76 7.75 7.75 7.68 7.67 7.66 7.66 7.60 7.60 7.58 7.56 7.54 7.54 7.51 7.50 7.50 7.49 7.48 7.47 7.46 7.46 7.44 7.42
4.64 4.62 4.60 4.59 4.18 4.16 4.16 4.15 3.82 3.81 3.09 3.08 3.07 3.07 3.07 3.05 3.05 2.01 2.00 2.00 1.99 1.98 1.98 1.55 1.54 1.41 1.40 1.38
3.23 1.09 1.10 1.07 3.27 2.00 2.14 1.08 2.02 2.24 3.28 3.11
f1 (ppm)

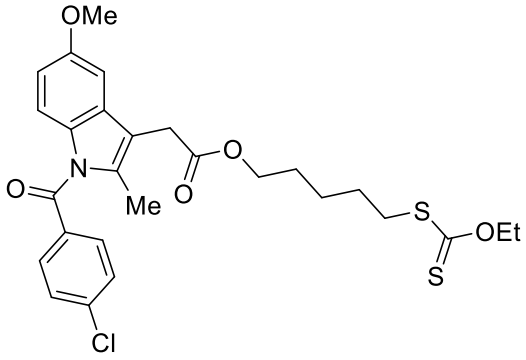

214.5 196.6 174.1 140.9 138.0 137.6 132.7 131.6 130.2 129.3 129.2 128.7 128.4 70.2 63.5 45.5 32.3 27.8 18.5 13.9
f1 (ppm)


O
O
N
H
O
O
S
OEt
S

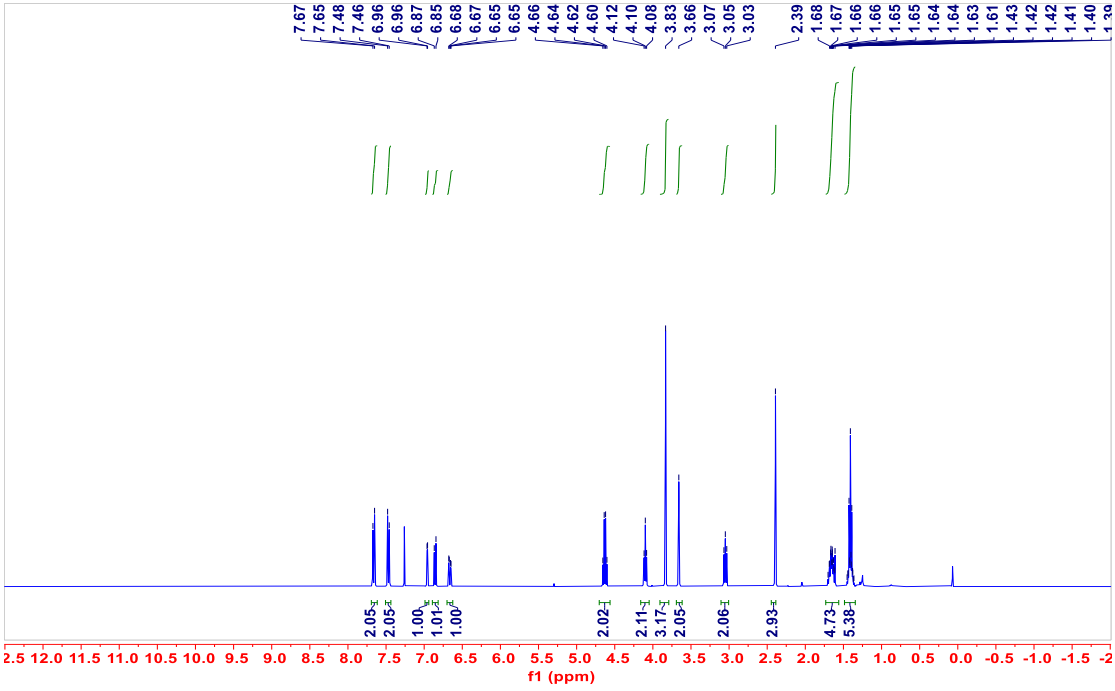
**10ac**

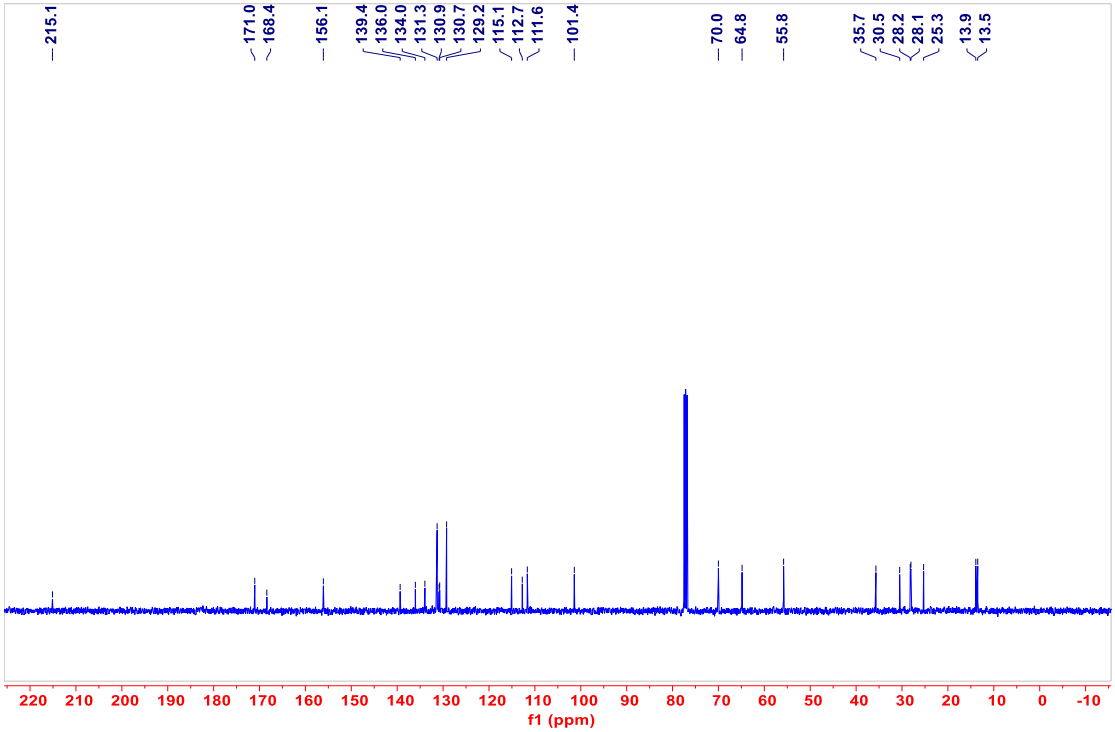

7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.25 7.23 7.23 7.23 7.22 7.21 7.21 7.20 7.19 7.13 7.13 7.11 7.11 7.11
5.03 5.01 4.64 4.62 4.60 4.58 4.15 4.13 4.12 3.06 3.04 3.02 3.00 3.00 2.99 2.98 2.97 2.95
2.02 1.98 1.98 1.97 1.96 1.96 1.94 1.94 1.93 1.92 1.91 1.91 1.41 1.40 1.39 1.37
2.26 1.09 2.16 0.86 2.00 2.19 2.07 2.04 0.89 2.16 12.53
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

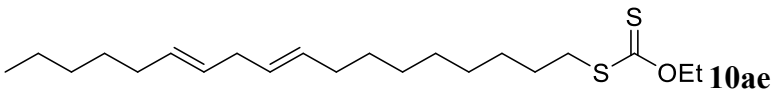

214.5
172.0
155.2
136.1
129.4
128.7
127.2
80.1
70.2
63.8
54.7
38.6
32.2
28.4
27.7
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
N
Me
O
O
O
S
OEt
S
Cl

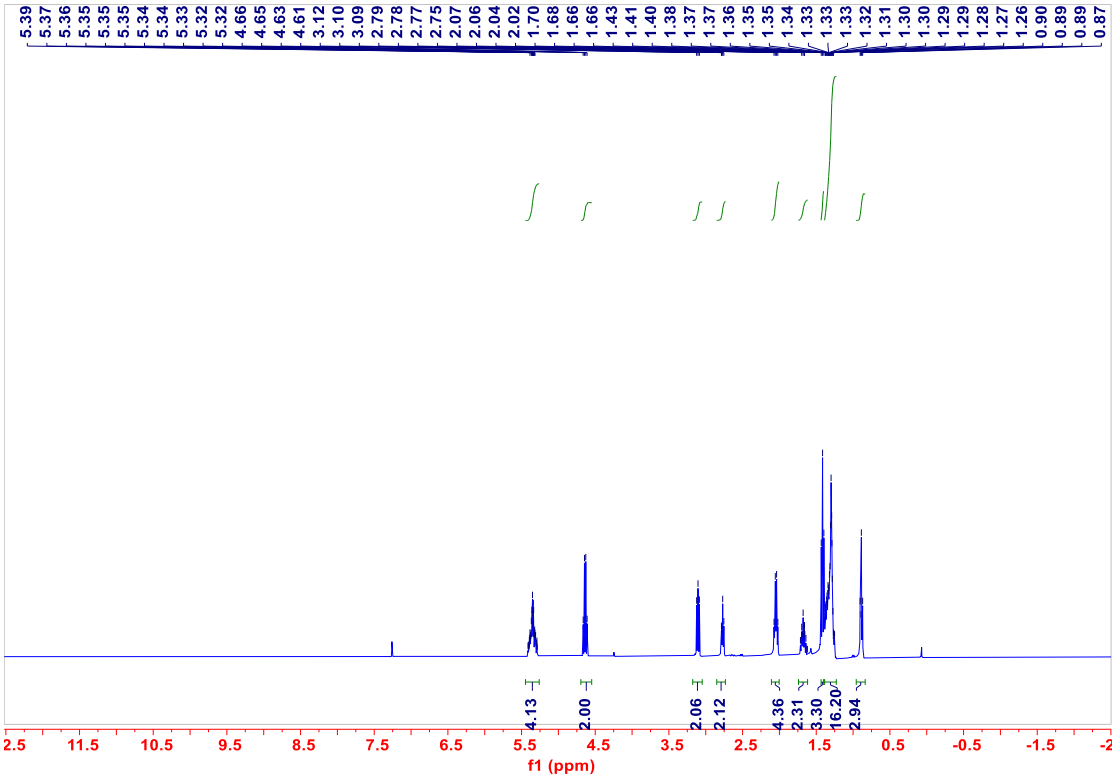
**10ad**

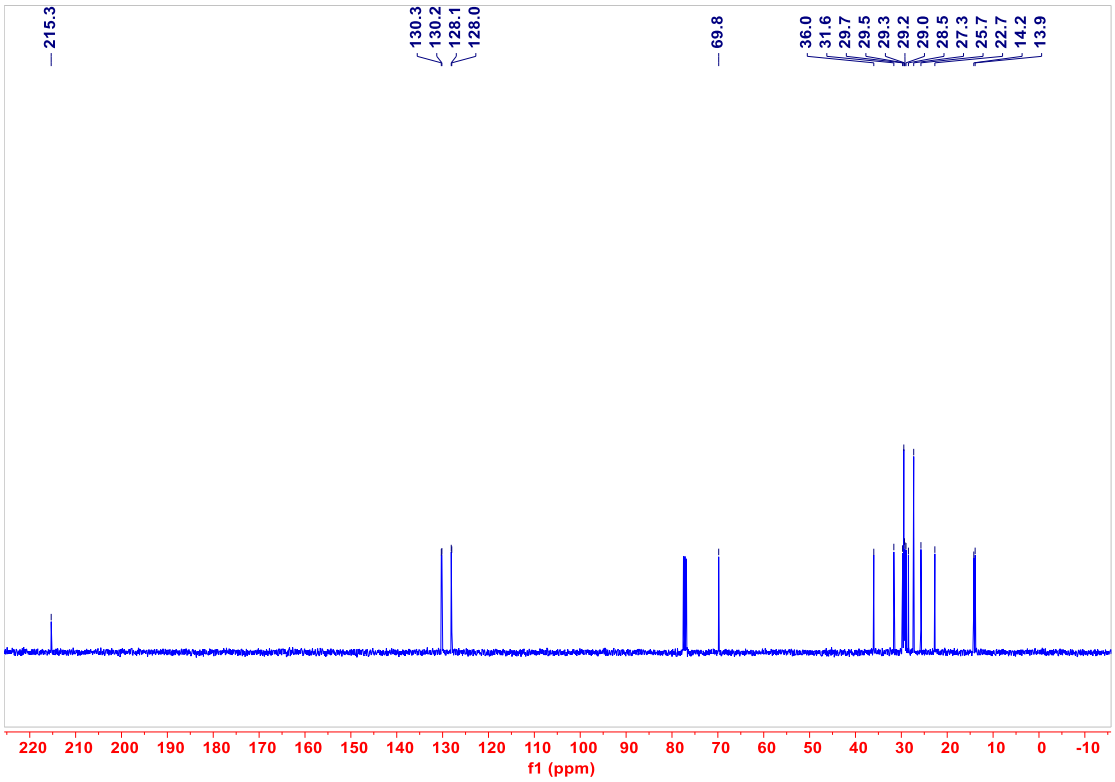

7.67 7.65 7.48 7.46 6.96 6.96 6.87 6.85 6.68 6.67 6.65 6.65 4.66 4.64 4.62 4.60 4.12 4.10 4.08 3.83 3.66 3.07 3.05 3.03 2.39 1.68 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.61 1.43 1.42 1.42 1.41 1.40
2.05 2.05 1.00 1.01 1.00 2.02 2.11 3.17 2.05 2.06 2.93 4.73 5.38
f1 (ppm)

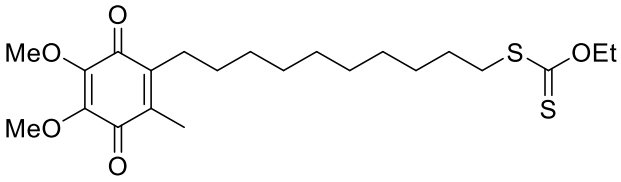

215.1
171.0 168.4 156.1 139.4 136.0 134.0 131.3 130.9 130.7 129.2 115.1 112.7 111.6 101.4
70.0 64.8 55.8 35.7 30.5 28.2 28.1 25.3 13.9 13.5
f1 (ppm)


S
S
OEt

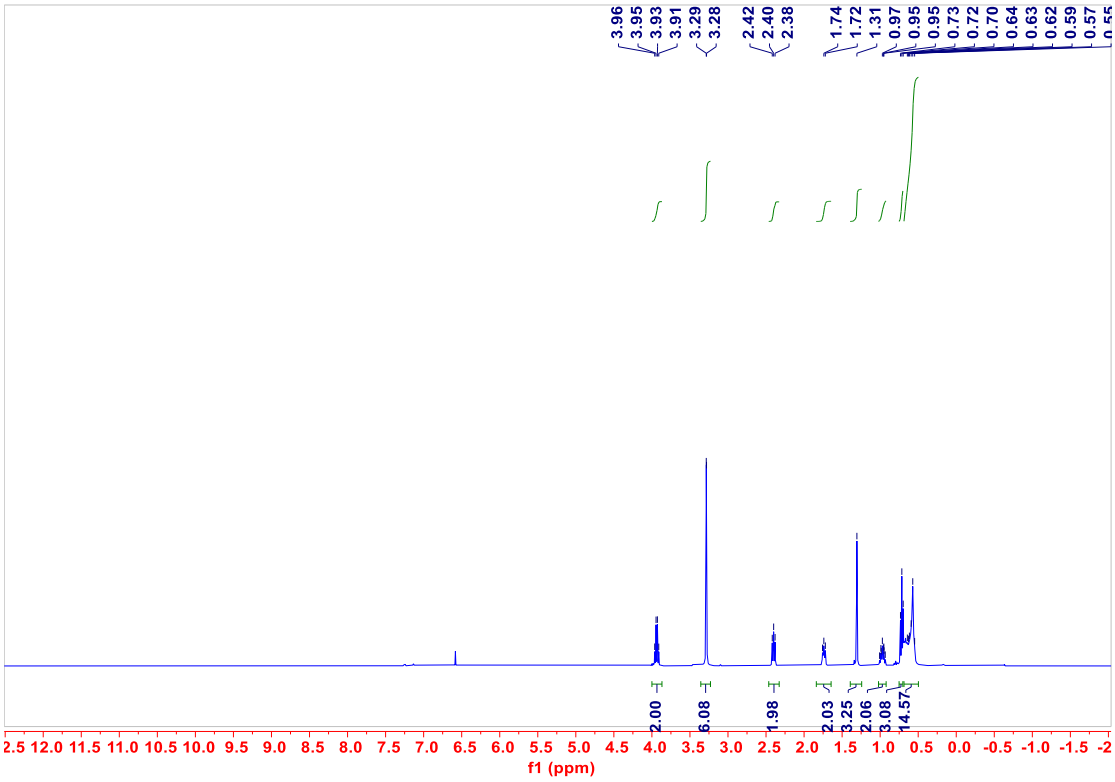
**10ae**

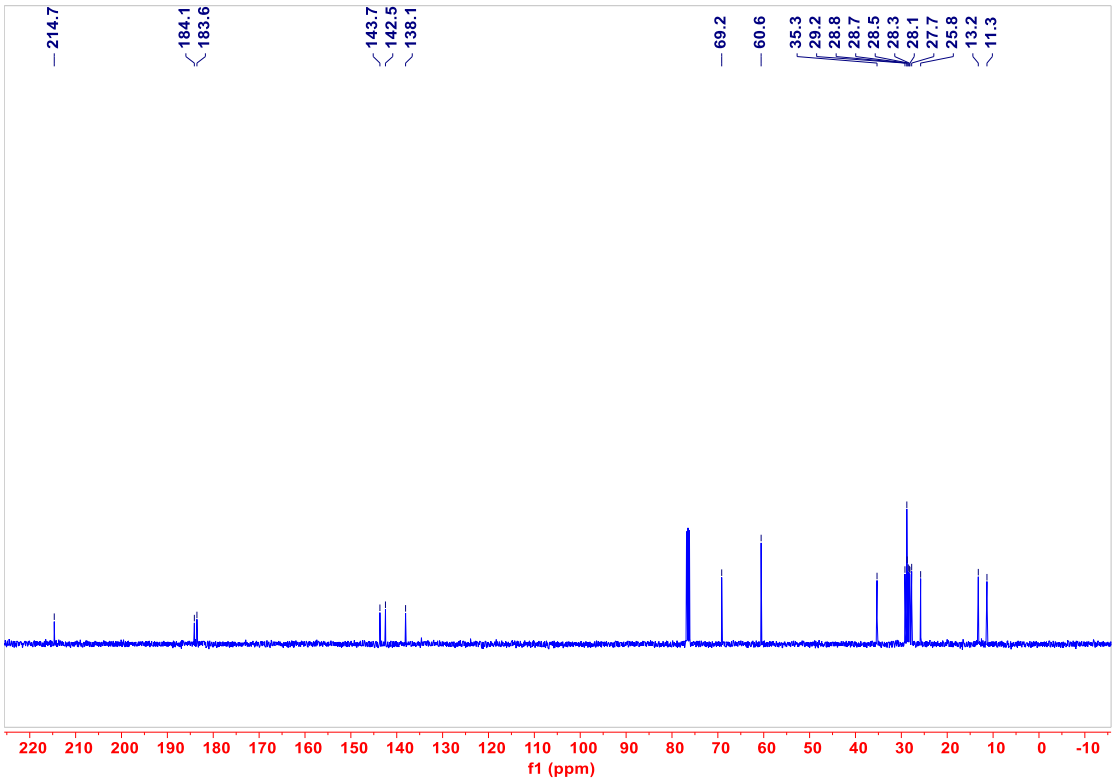

5.39 5.37 5.36 5.35 5.35 5.35 5.34 5.33 5.32 5.32 4.66 4.65 4.63 4.61 3.12 3.10 3.09 2.79 2.78 2.77 2.75 2.07 2.06 2.04 2.02 1.70 1.68 1.66 1.66 1.43 1.41 1.40 1.38 1.37 1.37 1.36 1.35 1.35 1.34 1.33 1.33 1.33 1.32 1.31 1.30 1.30 1.29 1.29 1.28 1.27 1.26 0.90 0.89 0.89 0.87
4.13
2.00
2.06
2.12
4.36
2.31
3.30
16.20
2.94
2.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5 -2
f1 (ppm)


215.3
130.3
130.2
128.1
128.0
69.8
36.0
31.6
29.7
29.5
29.3
29.2
29.0
28.5
27.3
25.7
22.7
14.2
13.9
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


MeO
MeO
O
O
S
OEt
S

**10af**


3.96
3.95
3.93
3.91
3.29
3.28
2.42
2.40
2.38
1.74
1.72
1.31
0.97
0.95
0.95
0.73
0.72
0.70
0.64
0.63
0.62
0.59
0.57
2.00
6.08
1.98
2.03
3.25
2.06
3.08
14.57
f1 (ppm)


214.7
184.1
183.6
143.7
142.5
138.1
69.2
60.6
35.3
29.2
28.8
28.7
28.5
28.3
28.1
27.7
25.8
13.2
11.3
f1 (ppm)