Supplementary Information (SI) for Green Chemistry.
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Supporting Information

Radical Click Reaction to Construct C–S Bond *via* Reductive Coupling of Phthalimide Derivatives

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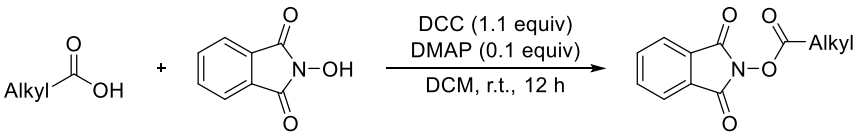
1. General remarks

^{1}H NMR, ^{13}C NMR data were obtained on AVANCE III Bruker 400 or 500 MHz nuclear resonance spectrometers unless otherwise noted. Chemical shifts (in ppm) were referenced to tetramethylsilane (TMS) (δ = 0.00 ppm) in $CDCl_3$ or dimethyl sulfoxide (δ = 2.50 ppm) in DMSO-d_6 as an internal standard. The data of ^{1}H NMR was reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, p = pentet and br = broad), coupling constant (*J* values) in Hz and integration. ^{13}C NMR spectra were obtained by the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm) or DMSO-d_6 (δ = 39.50 ppm). Flash chromatography was performed using 300-400 mesh silica gel with the indicated eluent according to standard techniques. Analytical thin-layer chromatography (TLC) was performed on pre-coated, glassbacked silica gel plates. Analysis of crude reaction mixture was done on an Agilent 7890 GC System with an Agilent 5975 Mass Selective Detector. Visualization of the developed chromatogram was performed by UV absorbance (254 nm) unless otherwise noted. High-resolution mass spectral (HRMS) data were recorded on Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer using electrospray ionization (ESI) mode. Unless specified, all the materials and ligands are commercially available and used as received.

2. General procedures

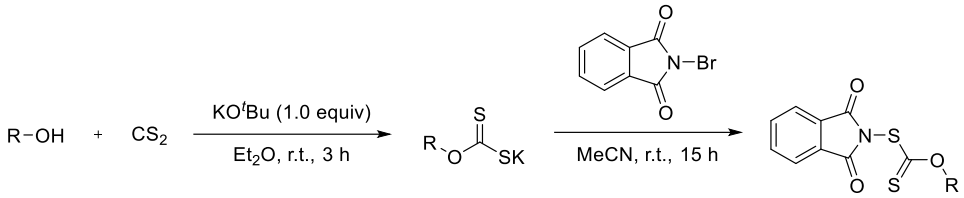
2.1 General procedure for synthesis of *N*-hydroxyphthalimide Esters (NHPI esters)[1]:



NHPI esters were prepared according to the previously reported procedures. A 100 mL round-bottom flask, equipped with a stir bar, was charged with carboxylic acid (1.0 equiv), *N*-hydroxyphthalimide(1.1 equiv), and DMAP (0.1 equiv).

Dichloromethane was added (0.2 M), and the mixture was stirred. DCC (1.1 equiv) was then added and the mixture allowed to stir until the acid was consumed (determined by TLC). The reaction mixture was filtered and rinsed with additional dichloromethane. The solvent was removed from the filtrate under reduced pressure, and the resulting residue was purified by flash chromatography to afford the desired NHPI ester.

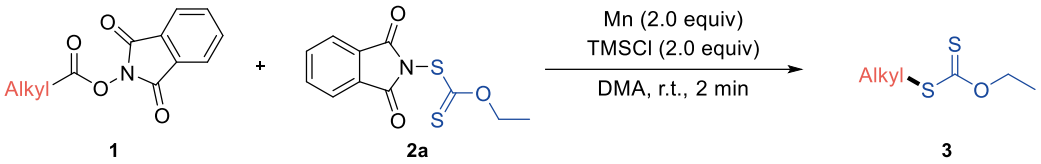
2.2 General procedure for synthesis of xanthate-derived reagents[2]:



Xanthate-derived reagents were prepared according to literature methods. An oven-dried 50 mL round-bottom flask equipped with a magnetic stir bar was charged sequentially with alcohol (2.0 mmol), KO^{t}Bu (1.0 equiv), and dry Et_2O (20 mL). The reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of CS_2 (1.5 equiv) and continued to be stirred for 3 hours at room temperature. The precipitate formed was collected by filtration, washed with Et_2O (2 × 10 mL), and dried in vacuo to afford the desired carbonodithioate salt product. No further purification is required and can be used directly for the next step.

In a 500 mL round-bottom flask, carbonodithioate salt (30.0 mmol) was suspended in MeCN (150 mL). To this suspension was added a solution of *N*-bromophthalimide (1.0 equiv) in MeCN (150 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 desired xanthate-derived reagent.

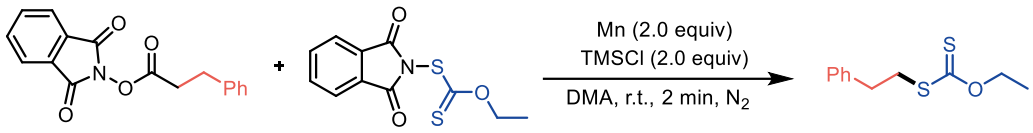
2.3 General procedure for decarboxylative reductive cross-coupling of products 3:



N-hydroxyphthalimide esters **1** (0.20 mmol), *N*-ethylxanthylphthalimides **2** (0.40 mmol, 2.0 equiv), Mn power (22.0 mg, 0.40 mmol) were placed into an oven-dried 10 mL Schlenk tube that was equipped with a stirring bar. The vessel was evacuated and filled with N_2 (three times), The chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) was added, followed by the addition of DMF (0.50 mL) *via* syringe. The reaction mixture was stirred at room temperature for 2 min. After this time, the crude reaction mixture was diluted with ethyl acetate (5.0 mL) and washed with water (3.0 mL). The aqueous layer was then extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The solvent was removed under vacuum and the residue was purified by flask column chromatography to afford the pure products **3**.

3. Optimization of the reaction conditions

3.1 Table S1. Optimization of the reductant



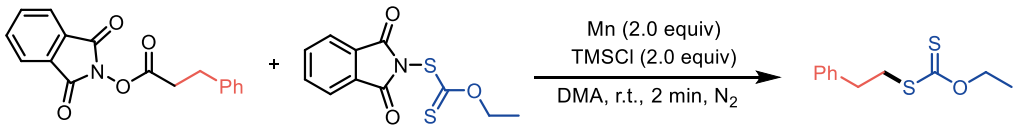
1a **2a** **3aa**

Entry	variation from standard conditions	yield /%
1	none	85%
2	Fe instead of Mn	n.d.
3	Zn instead of Mn	17%
4	B_2Pin_2 instead of Mn	n.d.
5[a]	B_2Pin_2 instead of Mn	n.d.
6	TDAE instead of Mn	n.d.
7	$(Et_2O)_2MeSiH$ instead of Mn	n.d.
8	Et_3N instead of Mn	2%
9	Cu instead of Mn	n.d.
10	Mg instead of Mn	n.d.

[a]add K_3PO_4 (2.0 equiv)

Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), reductant (2.0 equiv), TMSCl (2.0 equiv) and DMA (0.5 mL), at RT for 2 min under N_2. Isolated yields.

3.2 Table S2. Optimization of the additive

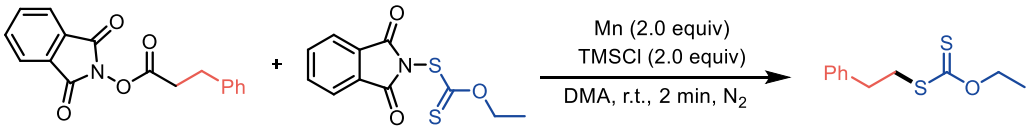


1a **2a** **3aa**

Entry	variation from standard conditions	yield /%
1	none	85%
2	TMSBr instead of TMSCl	72%
3	TESCl instead of TMSCl	55%
4	DMPSCl instead of TMSCl	57%
5	TBSCl instead of TMSCl	32%
6	TBDPSCl instead of TBDPCl	25%
7	Me_2SiCl_2 instead of TMSCl	56%
8	*i*-Pr_2SiCl_2 instead of TMSCl	14%
9	$MeSiCl_3$ instead of TMSCl	49%
10	$PhSiCl_3$ instead of TMSCl	53%

Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), Mn (2.0 equiv), additive (2.0 equiv) and DMA (0.5 mL), at RT for 2 min under N_2. Isolated yields.

3.3 Table S3. Optimization of the solvent and concentration

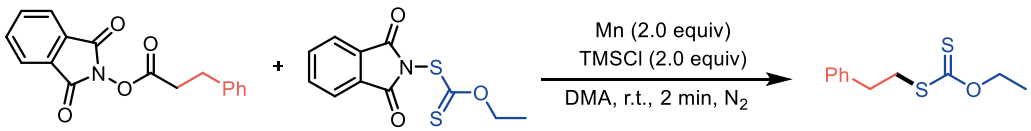


1a **2a** **3aa**

Entry	variation from standard conditions	yield /%
1	none	85%
2	DMSO instead of DMA	46%
3	NMP instead of DMA	53%
4	MeCN instead of DMA	28%
5	DMF instead of DMA	51%
6	THF instead of DMA	14%
7	DCE instead of DMA	n.d.
8	Dioxane instead of DMA	n.d.
9	DMA (1.0 mL)	73%
10	DMA (2.0 mL)	71%

Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), Mn (2.0 equiv), TMSCl (2.0 equiv) and solvent, at RT for 2 min under N_2. Isolated yields.

3.4 Table S4. Optimization of the ratio

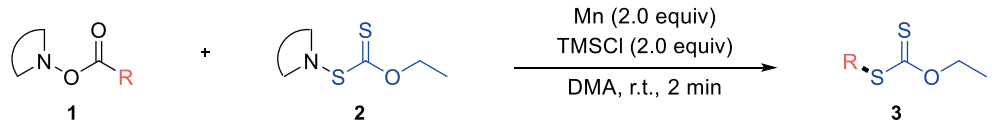


1a **2a** **3aa**

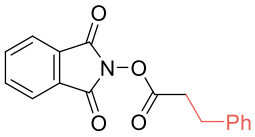
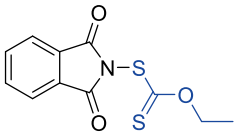
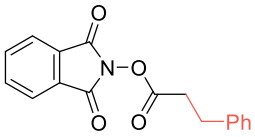
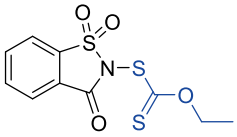
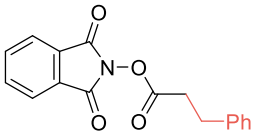
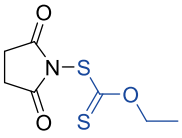
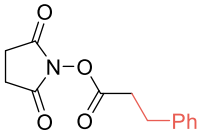
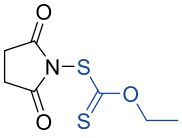
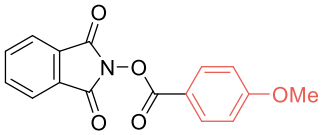
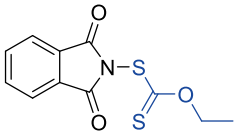
Entry	variation from standard conditions	yield /%
1	none	85%
2	**2a** (1.5 equiv) instead of **2a** (2.0 equiv)	58%
3	**2a** (1.0 equiv) instead of **2a** (2.0 equiv)	54%

Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), Mn (2.0 equiv), TMSCl (2.0 equiv) and DMA (0.5 mL), at RT for 2 min under N_2. Isolated yields.

3.5 Table S5. Optimization of the substrates

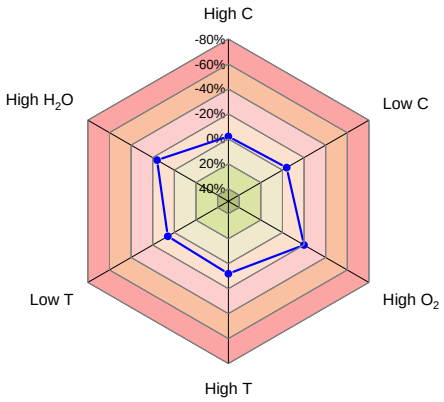


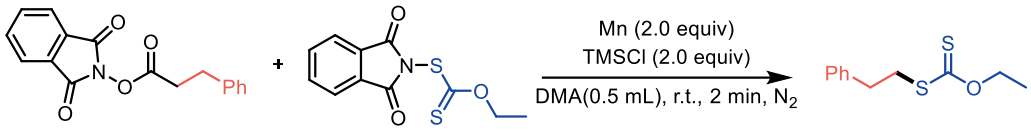
1 + **2** → **3**

Entry	**1**	**2**	yield of **3**
1			85% (standard)
2			4%
3			65%
4			trace
5			n.d.

Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), Mn (2.0 equiv), TMSCl (2.0 equiv) and DMA (0.5 mL), at RT for 2 min under N_2. Isolated yields.

3.6 Table S6. Sensitivity assessment



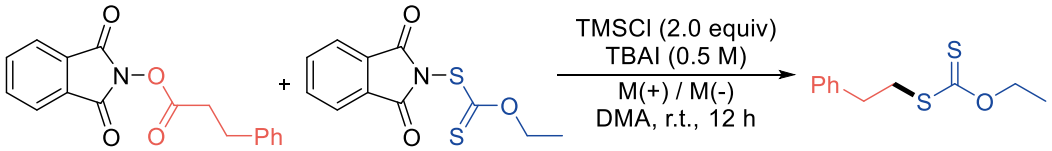


1a 2a 3aa

Entry	experiment	variation from standard conditions	yield /%	deviation /%
1	standard	none	85%	-
2	Low concentration (-10%)	DMA (0.75 mL)	82%	-4%
3	High concentration (+10%)	DMA (0.25 mL)	83%	-2%
4	High H_2O	+H_2O (10 μL)	71%	-16%
5	High O_2	air environment	68%	-20%
6	Low temperature	0 °C	80%	-6%
7	High temperature	50 °C	78%	-8%

Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), Mn (2.0 equiv), TMSCl (2.0 equiv) and DMA, at T for 2 min under N_2. Isolated yields.

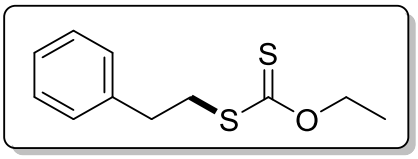
3.7 Table S7. Reaction attempts with electrochemical strategy



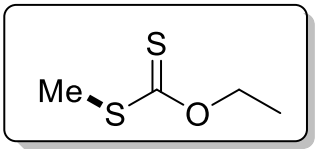
Entry	M(+) / M(-)	yield
1	CF(+) / CF(-)	n.d.
2	Zn(+) / Ni(-)	n.d.
3	Al(+) / Ni(-)	n.d.
4	CF(+) / Ni(-)	n.d.
5	CF(+) / Pt(-)	trace
6	Zn(+) / CF(-)	trace

Reaction conditions: undivided cell, constant current = 6 mA, **1a** (0.2 mmol), **2a** (0.4 mmol), TMSCl (2.0 equiv), TBAI (1.5 mmol) in DMA (3.0 mL), at RT for 12 h under N_2. GC yields.

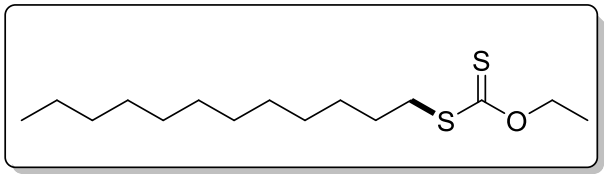
4. Characterization data



***O*-Ethyl *S*-phenethyl carbonodithioate (3aa)[3]:** The representative procedure was followed using phenethyl 1,3-dioxoisoindoline-2-carboxylate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3aa** (38.3 mg, 85%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 7.36 – 7.16 (m, 5H), 4.65 (q, *J* = 7.0 Hz, 2H), 3.35 (t, *J* = 8.0 Hz, 2H), 2.99 (t, *J* = 8.0 Hz, 2H), 1.43 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 214.7, 139.8, 128.6, 128.5, 126.6, 69.9, 37.0, 34.8, 13.8; **MS (EI)** *m/z* (relative intensity): 226 (M, 20), 104 (100), 91 (20).

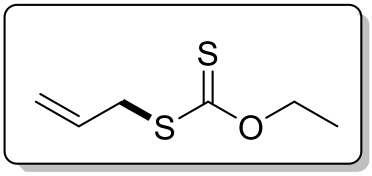


***O*-Ethyl *S*-methyl carbonodithioate (3ba)[4]:** The representative procedure was followed using methyl 1,3-dioxoisoindoline-2-carboxylate (**1b**) (41.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ba** (19.7 mg, 72%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 4.58 (q, *J* = 7.5 Hz, 2H), 2.48 (s, 3H), 1.35 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.7, 69.8, 18.8, 13.7; **HRMS (ESI)**: m/z for $C_4H_8OS_2$ $[M+H]^+$ calcd 137.0089, found 137.0091.

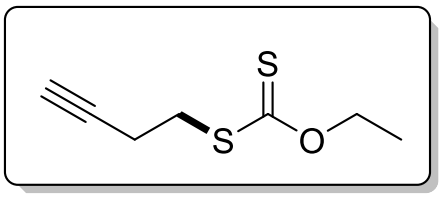


***S*-Dodecyl *O*-ethyl carbonodithioate (3ca)[5]:** The representative procedure was followed using dodecyl 1,3-dioxoisoindoline-2-carboxylate (**1c**) (71.8 mg, 0.20 mmol)

and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ca** (38.0 mg, 66%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.58 (q, *J* = 7.2 Hz, 2H), 3.04 (t, *J* = 7.6 Hz, 2H), 1.61 (p, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.31 – 1.18 (m, 18H), 0.81 (t, *J* = 6.4 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.2, 69.7, 35.9, 31.9, 29.6, 29.6, 29.5, 29.4, 29.3, 29.1, 28.9, 28.3, 22.7, 14.1, 13.8; **HRMS (ESI)**: m/z for $C_{15}H_{30}OS_2$ $[M+Na]^+$ calcd 313.1630, found 313.1634.

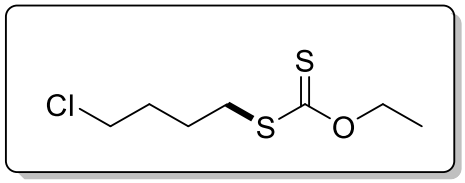


***S*-Allyl *O*-ethyl carbonodithioate (3da)[4]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl but-3-enoate (**1d**) (46.2 mg, 0.20 mmol) and S-(1,3-dioxoisoindolin-2-yl) O-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3da** (28.1 mg, 87%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 5.86 – 5.77 (m, 1H), 5.23 (d, *J* = 17.0 Hz, 1H), 5.10 (d, *J* = 10.0 Hz, 1H), 4.60 – 4.56 (m, 2H), 3.71 (d, *J* = 7.0 Hz, 2H), 1.36 (d, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 213.9, 131.7, 118.8, 70.0, 38.7, 13.8; **HRMS (ESI)**: m/z for $C_6H_{10}OS_2$ $[M+H]^+$ calcd 163.0246, found 163.0245.

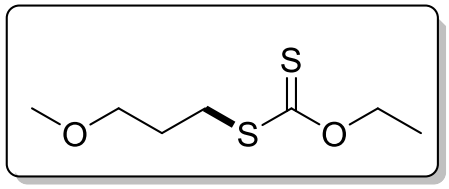


***S*-(But-3-yn-1-yl) *O*-ethyl carbonodithioate (3ea):** The representative procedure was followed using but-3-yn-1-yl 1,3-dioxoisoindoline-2-carboxylate (**1e**) (48.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ea** (30.3 mg, 87%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 4.59 (q, *J* = 7.0 Hz, 2H), 3.22 (t, *J* = 7.0 Hz, 2H), 2.58 – 2.52 (m, 2H), 1.99 – 1.96 (m, 1H), 1.36 (t, *J* = 7.0 Hz, 3H);

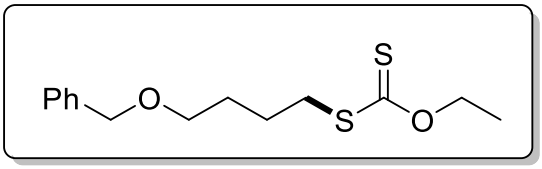
^{13}C NMR (125 MHz, $CDCl_3$) δ 214.0, 81.8, 70.1, 69.8, 34.4, 18.5, 13.7; **HRMS (ESI)**: m/z for $C_7H_{10}OS_2$ $[M+Na]^+$ calcd 197.0065, found 197.0061.



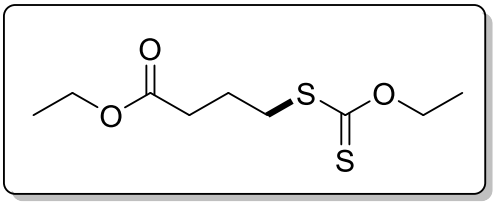
***S*-(4-Chlorobutyl) *O*-ethyl carbonodithioate (3fa):** The representative procedure was followed using 4-chlorobutyl 1,3-dioxoisoindoline-2-carboxylate (**1f**) (56.2 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3fa** (31.2 mg, 73%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.58 (q, J = 7.2 Hz, 2H), 3.50 (t, J = 6.0 Hz, 2H), 3.09 (t, J = 6.8 Hz, 2H), 1.89 – 1.72 (m, 4H), 1.36 (t, J = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.7, 69.9, 44.3, 35.0, 31.5, 25.8, 13.8; **HRMS (ESI)**: m/z for $C_7H_{13}ClOS_2$ $[M+Na]^+$ calcd 234.9989, found 234.9992.



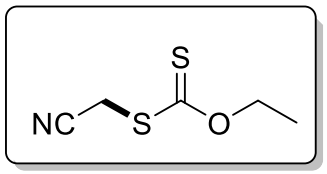
***O*-Ethyl *S*-(3-methoxypropyl) carbonodithioate (3ga):** The representative procedure was followed using 3-methoxypropyl 1,3-dioxoisoindoline-2-carboxylate (**1g**) (52.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 100:1) yielded **3ga** (31.2 mg, 71%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.58 (q, J = 7.2 Hz, 2H), 3.39 (t, J = 6.4 Hz, 2H), 3.27 (s, 3H), 3.13 (t, J = 7.2 Hz, 2H), 1.90 (p, J = 6.4 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.8, 70.9, 69.8, 58.6, 32.6, 28.4, 13.7; **HRMS (ESI)**: m/z for $C_7H_{14}O_2S_2$ $[M+Na]^+$ calcd 217.0327, found 217.0323.



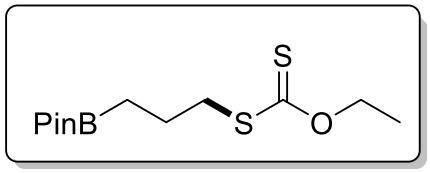
***S*-[4-(Benzyloxy)butyl] *O*-ethyl carbonodithioate (3ha):** The representative procedure was followed using 4-(benzyloxy)butyl 1,3-dioxoisoindoline-2-carboxylate (**1h**) (70.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 100:1) yielded **3ha** (50.7 mg, 89%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.31 – 7.14 (m, 5H), 4.55 (q, *J* = 7.2 Hz, 2H), 4.41 (s, 2H), 3.41 (t, *J* = 6.4 Hz, 2H), 3.06 (t, *J* = 7.2 Hz, 2H), 1.80 – 1.58 (m, 4H), 1.32 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 138.4, 128.3, 127.5, 127.5, 72.8, 69.7, 69.5, 35.6, 28.8, 25.3, 13.7; **HRMS (ESI)**: m/z for $C_{14}H_{20}O_2S_2$ $[M+Na]^+$ calcd 307.0797, found 307.0794.



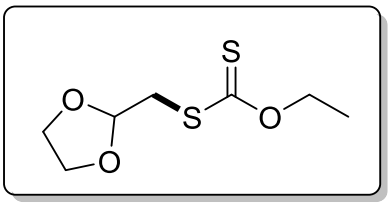
Ethyl 4-[(ethoxycarbonothioyl)thio] butanoate (3ia): The representative procedure was followed using 4-ethoxy-4-oxobutyl 1,3-dioxoisoindoline-2-carboxylate (**1i**) (52.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **3ia** (39.0 mg, 82%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 4.64 (q, *J* = 7.0 Hz, 2H), 4.13 (q, *J* = 7.0 Hz, 2H), 3.17 (t, *J* = 7.0 Hz, 2H), 2.42 (t, *J* = 7.5 Hz, 2H), 2.02 (p, *J* = 7.5 Hz, 2H), 1.42 (t, *J* = 7.0 Hz, 3H), 1.26 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 214.5, 172.7, 70.0, 60.5, 34.9, 33.0, 23.9, 14.2, 13.8; **HRMS (ESI)**: m/z for $C_9H_{16}O_3S_2$ $[M+Na]^+$ calcd 259.0433, found 259.0437.



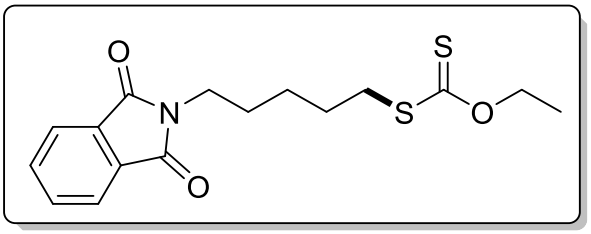
***S*-(2-Cyanomethyl) *O*-ethyl carbonodithioate (3ja):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-cyanoacetate (**1j**) (46.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 5:1) yielded **3ja** (20.3 mg, 63%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 4.65 (q, *J* = 7.0 Hz, 2H), 3.83 (s, 2H), 1.40 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 208.8, 115.2, 71.4, 21.2, 13.5; **HRMS (ESI)**: m/z for $C_5H_7NOS_2$ $[M+Na]^+$ calcd 183.9861, found 183.9864.



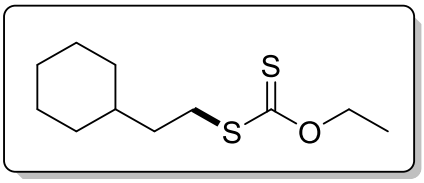
***O*-Ethyl *S*-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl) carbonodithioate (3ka):** The representative procedure was followed using 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl 1,3-dioxoisoindoline-2-carboxylate (**1k**) (71.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **3ka** (45.9 mg, 79%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.57 (q, *J* = 7.2 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H), 1.73 (p, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.18 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.2, 83.1, 69.7, 38.0, 24.8, 23.1, 13.8; **^{11}B NMR (160 MHz, $CDCl_3$) δ** 33.84; **HRMS (ESI)**: m/z for $C_{12}H_{23}BO_3S_2$ $[M+Na]^+$ calcd 313.1074, found 313.1071.



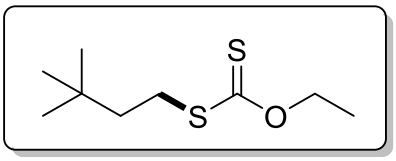
S*-[(1,3-Dioxolan-2-yl)methyl] *O*-ethyl carbonodithioate (3la):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-(1,3-dioxolan-2-yl)acetate (**1l**) (55.5 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 50:1) yielded **3la** (24.4 mg, 59%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** ***δ 5.10 (t, *J* = 4.4 Hz, 1H), 4.59 (q, *J* = 7.2 Hz, 2H), 3.98 – 3.93 (m, 2H), 3.86 – 3.82 (m, 2H), 3.38 (d, *J* = 4.4 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** ***δ*** 214.1, 101.8, 70.4, 65.4, 39.0, 13.8; **HRMS (ESI)**: m/z for $C_7H_{12}O_3S_2$ $[M+Na]^+$ calcd 231.0120, found 231.0124.



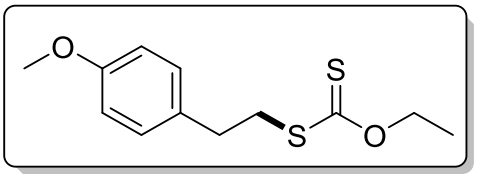
S*-[5-(1,3-Dioxoisoindolin-2-yl)pentyl] *O*-ethyl carbonodithioate (3ma):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 6-(1,3-dioxoisoindolin-2-yl)hexanoate (**1m**) (81.3 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 10:1) yielded **3ma** (57.1 mg, 85%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** ***δ 7.79 – 7.74 (m, 2H), 7.66 – 7.62 (m, 2H), 4.56 (q, *J* = 7.2 Hz, 2H), 3.61 (t, *J* = 7.2 Hz, 2H), 3.03 (t, *J* = 7.2 Hz, 2H), 1.72 – 1.58 (m, 4H), 1.42 – 1.35 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** ***δ*** 214.8, 168.3, 133.8, 132.0, 123.1, 69.7, 37.6, 35.5, 28.0, 27.9, 26.0, 13.7; **HRMS (ESI)**: m/z for $C_{16}H_{19}NO_3S_2$ $[M+Na]^+$ calcd 360.0699, found 360.0702.



***S*-(2-Cyclohexylethyl) *O*-ethyl carbonodithioate (3na):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-cyclohexylpropanoate (**1n**) (60.3 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3na** (36.7 mg, 79%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.57 (q, *J* = 7.2 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H), 1.69 – 1.57 (m, 5H), 1.54 – 1.45 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.31 – 1.03 (m, 4H), 0.92 – 0.76 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.2, 69.7, 37.0, 35.7, 33.6, 32.9, 26.4, 26.1, 13.8; **HRMS (ESI)**: m/z for $C_{11}H_{20}OS_2$ $[M+Na]^+$ calcd 255.0848, found 255.0845.

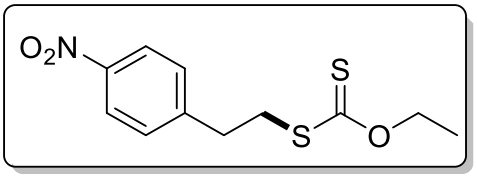


***S*-(3,3-Dimethylbutyl) *O*-ethyl carbonodithioate (3oa):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 4,4-dimethylpentanoate (**1o**) (55.1 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3oa** (34.5 mg, 84%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.58 (q, *J* = 7.2 Hz, 2H), 3.06 – 2.95 (m, 2H), 1.54 – 1.44 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 0.89 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.1, 69.7, 42.4, 31.8, 30.9, 29.1, 13.8; **HRMS (ESI)**: m/z for $C_9H_{18}OS_2$ $[M+Na]^+$ calcd 229.0691, found 229.0694.

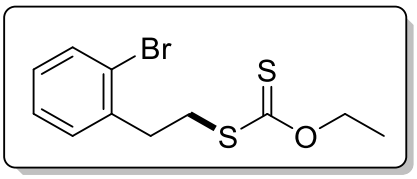


***O*-Ethyl *S*-(4-methoxyphenethyl) carbonodithioate (3pa)[3]:** The representative

procedure was followed using 1,3-dioxoisoindolin-2-yl 3-(4-methoxyphenyl) propanoate (**1p**) (65.1 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3pa** (40.6 mg, 79%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.09 (d, *J* = 8.4 Hz, 2H), 6.78 (d, *J* = 8.4 Hz, 2H), 4.58 (q, *J* = 7.2 Hz, 2H), 3.72 (s, 3H), 3.25 (t, *J* = 7.6 Hz, 2H), 2.85 (t, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.8, 158.3, 131.9, 129.5, 113.9, 69.9, 55.2, 37.3, 33.9, 13.8; **HRMS (ESI)**: m/z for $C_{12}H_{16}O_2S_2$ $[M+Na]^+$ calcd 279.0484, found 279.0480.

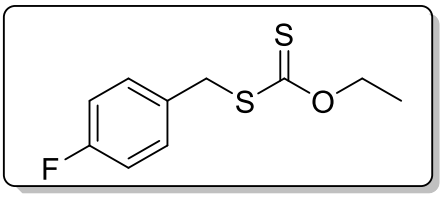


***O*-Ethyl *S*-(4-nitrophenethyl) carbonodithioate (3qa)[8]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-(4-nitrophenyl) propanoate (**1q**) (68.1 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 5:1) yielded **3qa** (41.7 mg, 77%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 8.09 (d, *J* = 8.8 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 2H), 4.58 (q, *J* = 7.2 Hz, 2H), 3.31 (t, *J* = 7.2 Hz, 2H), 3.04 (t, *J* = 7.2 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.1, 147.3, 146.7, 129.5, 123.7, 70.2, 36.2, 34.6, 13.7; **HRMS (ESI)**: m/z for $C_{11}H_{13}NO_3S_2$ $[M+Na]^+$ calcd 294.0229, found 294.0231.

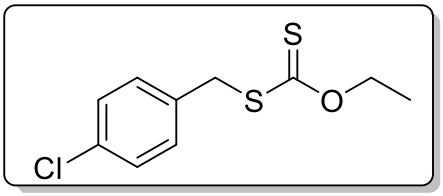


***S*-(2-Bromophenethyl) *O*-ethyl carbonodithioate (3ra):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-(2-bromophenyl) propanoate (**1r**) (74.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl

carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ra** (44.0 mg, 72%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** ***δ*** 7.49 – 7.41 (m, 1H), 7.25 – 7.12 (m, 2H), 7.03 – 6.99 (m, 1H), 4.56 (q, *J* = 7.2 Hz, 2H), 3.33 – 3.24 (m, 2H), 3.06 – 3.03 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** ***δ*** 214.3, 138.9, 132.8, 130.9, 128.3, 127.5, 124.3, 69.9, 35.1, 35.1, 13.8; **HRMS (ESI)**: m/z for $C_{11}H_{13}{}^{79}BrOS_2$ $[M+Na]^+$ calcd 326.9483, found 326.9480.

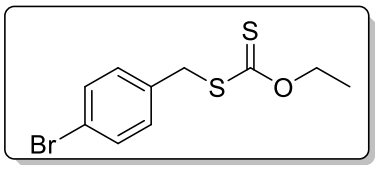


O*-Ethyl *S*-(4-fluorobenzyl) carbonodithioate (3sa)**[4]**:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-(4-fluorophenyl) acetate (**1s**) (59.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3sa** (36.4 mg, 79%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** ***δ 7.26 – 7.19 (m, 2H), 6.95 – 6.86 (m, 2H), 4.57 (q, *J* = 7.2 Hz, 2H), 4.25 (s, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** ***δ*** 213.7, 162.1 (d, ^1J = 244.9 Hz), 131.5 (d, ^4J = 3.3 Hz), 130.6 (d, ^3J = 8.0 Hz), 115.4 (d, ^2J = 21.2 Hz), 70.1, 39.6, 13.7; **HRMS (ESI)**: m/z for $C_{10}H_{11}FOS_2$ $[M+Na]^+$ calcd 253.0128, found 253.0125.

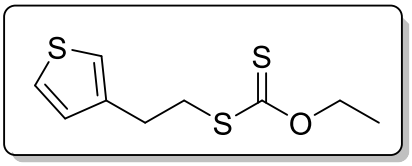


S*-(4-Chlorobenzyl) *O*-ethyl carbonodithioate (3ta)**[3]**:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-(4-chlorophenyl) acetate (**1t**) (63.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ta** (41.7 mg, 85%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** ***δ 7.22 – 7.18 (m, 4H), 4.56 (q,

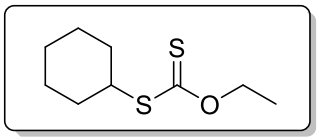
J = 7.2 Hz, 2H), 4.24 (s, 2H), 1.33 (t, *J* =7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.5, 134.4, 133.3, 130.3, 128.7, 70.2, 39.6, 13.7; **HRMS (ESI)**: m/z for $C_{10}H_{11}ClOS_2$ $[M+Na]^+$ calcd 268.9832, found 268.9830.



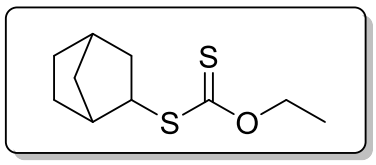
***S*-(4-Bromobenzyl) *O*-ethyl carbonodithioate (3ua)**[4]**:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-(4-bromophenyl) acetate (**1u**) (71.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ua** (44.0 mg, 76%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 4.57 (q, *J* = 7.2 Hz, 2H), 4.23 (s, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.5, 134.9, 131.7, 130.7, 121.4, 70.2, 39.6, 13.8; **HRMS (ESI)**: m/z for $C_{10}H_{11}{}^{79}BrOS_2$ $[M+Na]^+$ calcd 312.9327, found 312.9324.



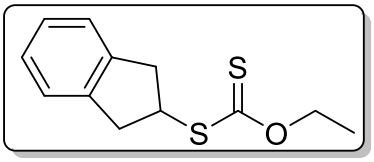
***O*-Ethyl *S*-(2-(thiophen-3-yl)ethyl) carbonodithioate (3va)**[3]**:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-(thiophen-3-yl)propanoate (**1v**) (60.3 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3va** (26.8 mg, 58%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.21 – 7.19 (m, 1H), 6.97 (d, *J* = 2.8 Hz, 1H), 6.93 – 6.92 (m, 1H), 4.58 (q, *J* = 7.2 Hz, 2H), 3.29 (t, *J* = 7.6 Hz, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.7, 140.0, 128.0, 125.8, 121.4, 69.9, 36.4, 29.3, 13.8; **HRMS (ESI)**: m/z for $C_9H_{12}OS_3$ $[M+Na]^+$ calcd 254.9942, found 254.9934.



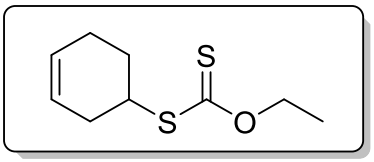
***S*-Cyclohexyl *O*-ethyl carbonodithioate (3wa)[3]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (**1w**) (54.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3wa** (27.7 mg, 68%) as yellow oil;**^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.57 (q, *J* = 7.2 Hz, 2H), 3.62 – 3.56 (m, 1H), 2.04 – 1.95 (m, 2H), 1.69 – 1.65 (m, 2H), 1.58 – 1.37 (m, 4H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.28 – 1.16 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.5, 69.4, 48.7, 32.3, 25.9, 25.5, 13.8; **HRMS (ESI)**: m/z for $C_9H_{16}OS_2$ $[M+Na]^+$ calcd 227.0535, found 227.0532.



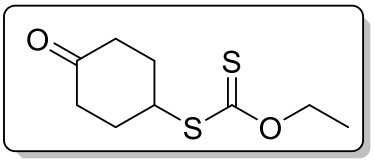
***S*-(Bicyclo[2.2.1]heptan-2-yl) *O*-ethyl carbonodithioate (3xa)[4]:** The representative procedure was followed using 11,3-dioxoisoindolin-2-yl bicyclo[2.2.1]heptane-2-carboxylate (**1x**) (57.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3xa** (29.8 mg, 69%) as yellow oil;**^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.56 (q, *J* = 7.2 Hz, 2H), 3.48 – 3.44 (m, 1H), 2.34 – 2.28 (m, 2H), 1.79 – 1.73 (m, 1H), 1.64 – 1.47 (m, 2H), 1.44 – 1.37 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.23 – 1.09 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.0, 69.3, 50.8, 42.8, 37.1, 36.4, 36.2, 28.9, 28.4, 13.8; **HRMS (ESI)**: m/z for $C_{10}H_{16}OS_2$ $[M+Na]^+$ calcd 239.0540, found 239.1271.



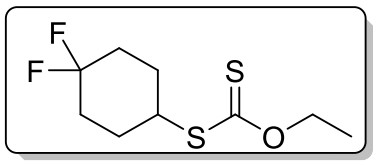
***S*-(2,3-Dihydro-1H-inden-2-yl) *O*-ethyl carbonodithioate (3ya)[6]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2,3-dihydro-1H-indene-2-carboxylate (**1y**) (61.4 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ya** (29.6 mg, 62%) as yellow oil;**^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.16 – 7.07 (m, 4H), 4.59 (q, *J* = 7.2 Hz, 2H), 4.39 – 4.32 (m, 1H), 3.48 – 3.42 (m, 2H), 2.99 – 2.94 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.6, 141.1, 126.8, 124.4, 69.7, 47.5, 39.2, 13.8; **HRMS (ESI)**: m/z for $C_{12}H_{14}OS_2$ $[M+Na]^+$ calcd 261.0378, found 261.0374.



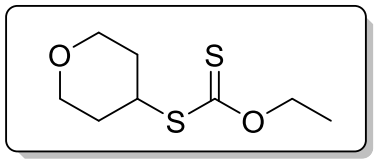
***S*-(Cyclohex-3-en-1-yl) *O*-ethyl carbonodithioate (3za):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl cyclohex-3-ene-1-carboxylate (**1z**) (54.2 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3za** (24.2 mg, 60%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 5.69 – 5.54 (m, 2H), 4.58 (q, *J* = 7.2 Hz, 2H), 3.91 – 3.79 (m, 1H), 2.50 – 2.43 (m, 1H), 2.15 – 2.02 (m, 4H), 1.76 – 1.67 (m, 1H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.5, 127.0, 125.0, 69.5, 45.0, 30.64, 27.9, 24.5, 13.8; **HRMS (ESI)**: m/z for $C_9H_{14}OS_2$ $[M+Na]^+$ calcd 225.0378, found 225.0375.



***O*-Ethyl *S*-(4-oxocyclohexyl) carbonodithioate (3a'a):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 4-oxocyclohexane-1-carboxylate (**1a'**) (57.4 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **3a'a** (28.6 mg, 66%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.61 (q, *J* = 7.2 Hz, 2H), 4.02 – 3.96 (m, 1H), 2.49 – 2.38 (m, 4H), 2.37 – 2.29 (m, 2H), 2.01 – 1.91 (m, 2H), 1.37 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 213.2, 209.2, 70.0, 46.1, 40.1, 31.4, 13.8; **HRMS (ESI)**: m/z for $C_9H_{14}O_2S_2$ $[M+Na]^+$ calcd 241.0327, found 241.0330.

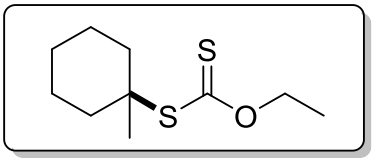


***S*-(4,4-Difluorocyclohexyl) *O*-ethyl carbonodithioate (3b'a):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 4,4-difluorocyclohexane-1-carboxylate (**1b'**) (61.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3b'a** (28.7 mg, 60%) as yellow oil;**^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.57 (q, *J* = 7.2 Hz, 2H), 3.70 – 3.63 (m, 1H), 2.12 – 2.00 (m, 4H), 1.92 – 1.72 (m, 4H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 213.5, 69.9, 45.9, 32.9 (t, ^2J = 24.6 Hz), 28.0 (t, ^3J = 4.9 Hz), 13.8; **^{19}F NMR (377 MHz, $CDCl_3$) δ** -95.98, -98.86; **HRMS (ESI)**: m/z for $C_9H_{14}F_2OS_2$ $[M+Na]^+$ calcd 263.0346, found 263.0344.

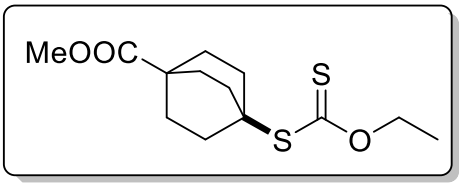


***O*-Ethyl *S*-(tetrahydro-2H-pyran-4-yl) carbonodithioate (3c'a)[7]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl tetrahydro-2H-

pyran-4-carboxylate (**1c'**) (55.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 50:1) yielded **3c'a** (23.7 mg, 58%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.58 (q, *J* = 7.2 Hz, 2H), 3.90 – 3.85 (m, 2H), 3.83 – 3.75 (m, 1H), 3.53 – 3.45 (m, 2H), 2.04 – 1.96 (m, 2H), 1.74 – 1.64 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.5, 69.7, 67.3, 45.3, 32.0, 13.8; **MS (EI)** *m/z* (relative intensity): 206 (M, 20), 123 (25), 84 (80), 55 (100).

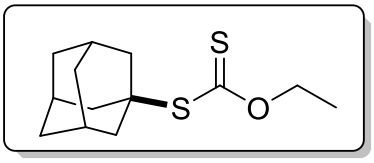


***O*-Ethyl *S*-(1-methylcyclohexyl) carbonodithioate (3d'a)[7]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 1-methylcyclohexane-1-carboxylate (**1d'**) (57.4 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3d'a** (29.0 mg, 67%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.60 (q, *J* = 7.2 Hz, 2H), 2.02 – 1.95 (m, 2H), 1.58 – 1.55 (m, 2H), 1.52 (s, 3H), 1.52 – 1.47 (m, 2H), 1.47 – 1.45 (s, 2H), 1.45 – 1.41 (m, 2H), 1.39 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.4, 69.2, 56.6, 37.6, 25.6, 22.4, 13.7; **MS (EI)** *m/z* (relative intensity): 218 (M, 20), 123 (30), 97 (100), 55 (90).

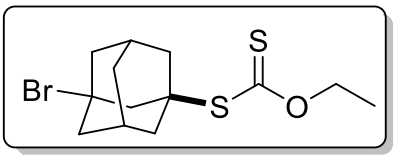


Methyl 4-[(ethoxycarbonothioyl)thio] bicyclo[2.2.2]octane-1-carboxylate (3e'a)[7]: The representative procedure was followed using 1-(1,3-dioxoisoindolin-2-yl) 4-methyl bicyclo[2.2.2]octane-1,4-dicarboxylate (**1e'**) (71.4 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **3e'a** (34.5 mg,

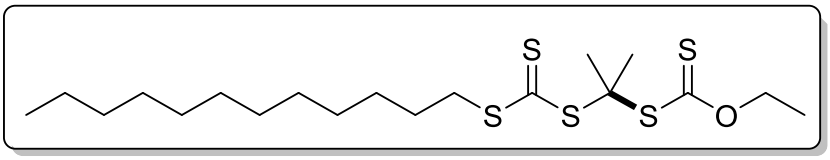
60%) as white solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.59 (q, *J* = 7.2 Hz, 2H), 3.58 (s, 3H), 2.00 – 1.96 (m, 6H), 1.85 – 1.81 (m, 6H), 1.39 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 213.3, 177.2, 69.3, 51.8, 51.0, 37.8, 30.1, 28.9, 13.7; **HRMS (ESI)**: m/z for $C_{13}H_{21}O_3S_2$ $[M+H]^+$ calcd 289.0932, found 289.0931.



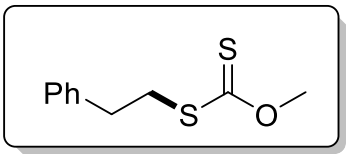
***S*-(Adamantan-1-yl) *O*-ethyl carbonodithioate (3f'a)[6]:** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl adamantane-1-carboxylate (**1f'**) (65.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3f'a** (29.5 mg, 58%) as white solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.59 (q, *J* = 7.2 Hz, 2H), 2.07 (d, *J* = 2.4 Hz, 6H), 2.01 (s, 3H), 1.65 (s, 6H), 1.42 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.3, 69.2, 54.5, 41.8, 36.2, 29.8, 13.7; **HRMS (ESI)**: m/z for $C_{13}H_{21}OS_2$ $[M+H]^+$ calcd 257.1028, found 257.1036.



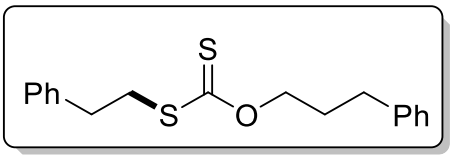
***S*-(-3-Bromoadamantan-1-yl) *O*-ethyl carbonodithioate (3g'a):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-bromoadamantane-1-carboxylate (**1g'**) (80.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3g'a** (45.3 mg, 68%) as white solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.61 (q, *J* = 7.2 Hz, 2H), 2.69 (s, 2H), 2.24 (s, 4H), 2.21 – 2.18 (m, 2H), 2.10 – 2.06 (m, 2H), 2.00 – 1.97 (m, 2H), 1.64 (s, 2H), 1.43 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 212.5, 69.4, 63.0, 55.2, 52.4, 47.7, 39.9, 34.2, 33.0, 13.7; **HRMS (ESI)**: m/z for $C_{13}H_{19}{}^{79}BrOS_2$ $[M+Na]^+$ calcd 356.9953, found 356.9957.



***S*-(2-(((Dodecylthio) carbonothioyl) thio) propan-2-yl) *O*-ethyl carbonodithioate (3h'a):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 2-(((dodecylthio)carbonothioyl)thio)-2-methylpropanoate (**1h'**) (101.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3h'a** (40.0 mg, 45%) as yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 4.72 (q, *J* = 7.2 Hz, 2H), 2.87 – 2.78 (m, 2H), 1.69 – 1.64 (m, 2H), 1.57 (s, 6H), 1.48 (t, *J* = 7.2 Hz, 3H), 1.45 – 1.35 (m, 6H), 1.33 – 1.26 (m, 12H), 0.88 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 220.1, 213.4, 71.3, 39.3, 31.9, 29.7, 29.6, 29.6, 29.6, 29.6, 29.5, 29.3, 29.2, 29.1, 28.4, 22.7, 14.1, 13.7; **HRMS (ESI)**: m/z for $C_{19}H_{36}OS_5$ $[M+Na]^+$ calcd 463.1262, found 463.1266.

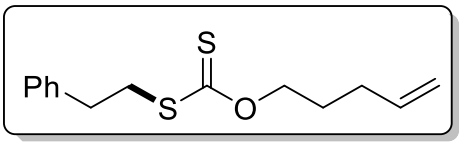


***O*-Methyl *S*-phenethyl carbonodithioate (3ab):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-methyl carbonodithioate (**2b**) (101.2 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ab** (36.8 mg, 87%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.26 – 7.20 (m, 2H), 7.18 – 7.13 (m, 3H), 4.10 (s, 3H), 3.30 (t, *J* = 7.6 Hz, 2H), 2.92 (t, *J* = 7.6 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 215.6, 140.0, 139.7, 128.6, 128.5, 126.6, 60.1, 37.3, 34.7; **HRMS (ESI)**: m/z for $C_{10}H_{12}OS_2$ $[M+Na]^+$ calcd 235.0222, found 235.0220.

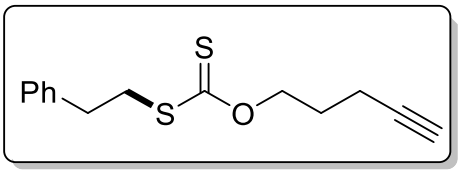


***S*-Phenethyl *O*-(3-phenylpropyl) carbonodithioate (3ac):** The representative

procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-(3-phenylpropyl) carbonodithioate (**2c**) (142.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ac** (48.6 mg, 77%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.27 – 7.10 (m, 10H), 4.53 (t, *J* = 6.4 Hz, 2H), 3.28 (t, *J* = 7.6 Hz, 2H), 2.92 (t, *J* = 6.4 Hz, 2H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.05 (p, *J* = 6.6 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.7, 140.8, 139.7, 128.6, 128.5, 128.4, 126.6, 126.1, 73.1, 37.1, 34.8, 32.1, 29.8; **HRMS (ESI)**: m/z for $C_{18}H_{20}OS_2$ $[M+Na]^+$ calcd 339.0848, found 339.0852.

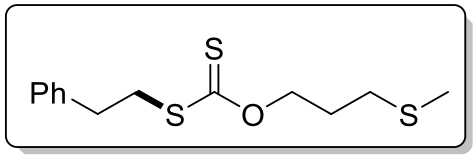


***O*-(Pent-4-en-1-yl) *S*-phenethyl carbonodithioate (3ad):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-(pent-4-en-1-yl) carbonodithioate (**2d**) (122.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ad** (37.3 mg, 70%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.26 – 7.22 (m, 2H), 7.18 – 7.16 (m, 3H), 5.80 – 5.70 (m, 1H), 5.03 – 4.92 (m, 2H), 4.53 (t, *J* = 6.6 Hz, 2H), 3.31 – 3.26 (m, 2H), 2.94 – 2.90 (m, 2H), 2.15 – 2.09 (m, 2H), 1.84 (p, *J* = 6.7 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.8, 139.7, 137.1, 128.6, 128.6, 126.6, 115.6, 73.3, 37.1, 34.9, 30.0, 27.4; **HRMS (ESI)**: m/z for $C_{14}H_{18}OS_2$ $[M+Na]^+$ calcd 289.0691, found 289.0690.

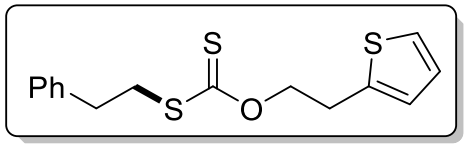


***O*-(Pent-4-yn-1-yl) *S*-phenethyl carbonodithioate (3ae):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-(pent-4-yn-1-yl) carbonodithioate

(**2e**) (122.0 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ae** (35.3 mg, 67%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.25 – 7.22 (m, 2H), 7.18 – 7.13 (m, 3H), 4.61 (t, *J* = 6.4 Hz, 2H), 3.31 – 3.26 (m, 2H), 2.94 – 2.88 (m, 2H), 2.29 – 2.24 (m, 2H), 1.99 – 1.89 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.7, 139.6, 128.5, 128.5, 126.6, 82.6, 72.1, 69.3, 37.2, 34.7, 27.2, 15.3; **HRMS (ESI)**: m/z for $C_{14}H_{16}OS_2$ $[M+Na]^+$ calcd 287.0535, found 287.0530.

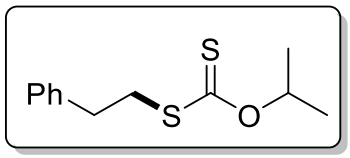


***O*-[3-(Methylthio)propyl] *S*-phenethyl carbonodithioate (3af):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-(3-(methylthio)propyl) carbonodithioate (**2f**) (130.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 200:1) yielded **3af** (47.2 mg, 82%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.26 – 7.21 (m, 2H), 7.16 (d, *J* = 7.2 Hz, 3H), 4.62 (t, *J* = 6.3 Hz, 2H), 3.31 – 3.26 (m, 2H), 2.94 – 2.89 (m, 2H), 2.53 (t, *J* = 7.2 Hz, 2H), 2.05 – 1.98 (m, 5H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 214.6, 139.6, 128.5, 128.5, 126.6, 72.2, 37.1, 34.7, 30.50, 27.8, 15.5; **HRMS (ESI)**: m/z for $C_{13}H_{18}OS_3$ $[M+Na]^+$ calcd 309.0412, found 309.0414.

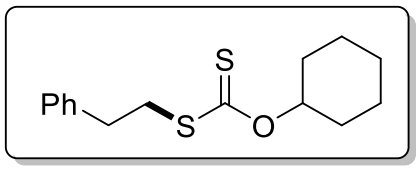


***S*-Phenethyl *O*-[2-(thiophen-2-yl)ethyl] carbonodithioate (3ag):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-(2-(thiophen-2-yl)ethyl) carbonodithioate (**2g**) (139.6 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ag** (43.8 mg, 71%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.24 (t, *J* = 7.6 Hz, 2H), 7.16 (t, *J* = 6.8 Hz, 3H), 7.10 (d, *J* = 4.8 Hz, 1H),

6.89 – 6.86 (m, 1H), 6.82 (d, *J* = 3.2 Hz, 1H), 4.73 (t, *J* = 6.8 Hz, 2H), 3.30 – 3.23 (m, 4H), 2.93 – 2.86 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.5, 139.7, 139.2, 128.6, 128.6, 127.0, 126.6, 125.8, 124.1, 73.4, 37.1, 34.7, 28.8; **HRMS (ESI)**: m/z for $C_{15}H_{16}OS_3$ $[M+Na]^+$ calcd 331.0255, found 331.0258.

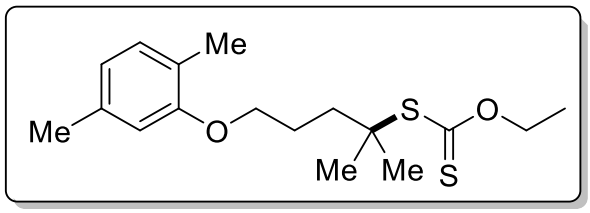


***O*-Isopropyl *S*-phenethyl carbonodithioate (3ah):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-isopropyl carbonodithioate (**2h**) (112.4 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ah** (39.6 mg, 82%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.25 – 7.21 (m, 2H), 7.16 (d, *J* = 7.7 Hz, 3H), 5.71 (hept, *J* = 6.4 Hz, 1H), 3.27 – 3.22 (m, 2H), 2.92 – 2.88 (m, 2H), 1.32 (d, *J* = 6.4 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 213.8, 139.8, 128.5, 128.5, 126.5, 77.7, 36.8, 34.9, 21.3; **HRMS (ESI)**: m/z for $C_{12}H_{16}OS_2$ $[M+Na]^+$ calcd 263.0535, found 263.0534.

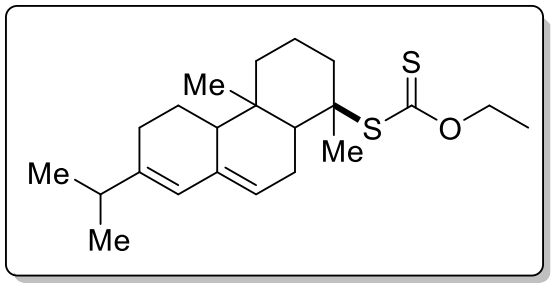


***O*-(Pent-4-yn-1-yl) *S*-phenethyl carbonodithioate (3ai):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 3-phenylpropanoate (**1a**) (59.0 mg, 0.20 mmol) and *O*-cyclohexyl *S*-(1,3-dioxoisoindolin-2-yl) carbonodithioate (**2i**) (128.4 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **3ai** (36.0 mg, 64%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.27 – 7.21 (m, 2H), 7.17 (d, *J* = 7.2 Hz, 3H), 5.54 – 5.48 (m, 1H), 3.28 – 3.23 (m, 2H), 2.94 – 2.89 (m, 2H), 1.97 – 1.89 (m, 2H), 1.71 – 1.67 (m, 2H), 1.58 – 1.47 (m, 3H), 1.41 – 1.23 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 213.7, 139.8, 128.6, 128.53, 126.6, 82.4, 36.8, 35.0, 30.9, 25.2, 23.6; **HRMS (ESI)**: m/z for $C_{15}H_{20}OS_2$ $[M+Na]^+$ calcd

303.0848, found 303.0845.

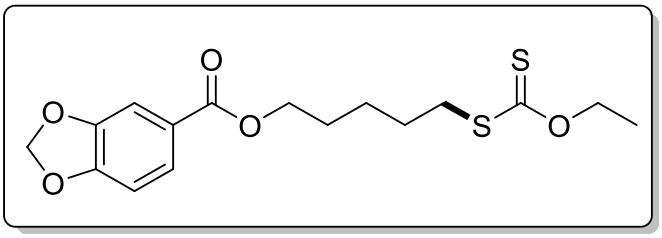


***S*-[5-(2,5-Dimethylphenoxy)-2-methylpentan-2-yl] *O*-ethyl carbonodithioate (7aa):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (**7a**) (79.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 50:1) yielded **7aa** (35.4 mg, 54%) as yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.97 – 6.88 (m, 1H), 6.63 – 6.49 (m, 2H), 4.68 – 4.49 (m, 2H), 3.92 – 3.82 (m, 2H), 2.28 – 2.20 (m, 3H), 2.13 – 2.06 (m, 3H), 1.94 – 1.78 (m, 4H), 1.46 – 1.35 (m, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.9, 156.8, 136.5, 130.3, 123.4, 120.7, 111.9, 69.3, 67.7, 55.0, 38.1, 27.9, 25.2, 21.4, 15.7, 13.7; **HRMS (ESI)**: m/z for $C_{17}H_{26}O_2S_2$ $[M+Na]^+$ calcd 349.1266, found 349.1264.

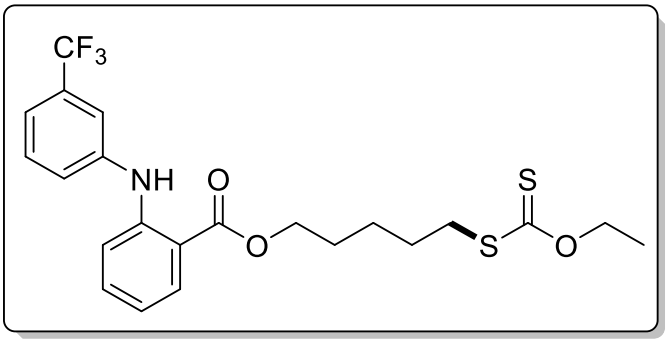


***O*-Ethyl *S*-(7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,4b,5,6,10,10a-decahydrophenanthren -1-yl) carbonodithioate (7ba):** The representative procedure was followed using 1,3-dioxoisoindolin-2-yl 7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,4b,5,6,10,10a-decahydrophenanthrene-1-carboxylate (**7b**) (89.4 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether) yielded **7ba** (34.2 mg, 45%) as yellow

solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 5.68 (s, 1H), 5.33 – 5.29 (m, 1H), 4.57 – 4.54 (m, 2H), 2.19 – 2.06 (m, 4H), 1.99 – 1.98 (m, 4H), 1.84 – 1.68 (m, 4H), 1.50 – 1.48 (m, 3H), 1.38 – 1.35 (m, 6H), 0.92 (d, *J* = 3.2 Hz, 3H), 0.91 (d, *J* = 3.2 Hz, 3H), 0.73 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.2, 145.3, 135.2, 122.3, 120.7, 69.3, 61.9, 51.0, 46.6, 39.3, 38.1, 36.9, 34.8, 27.4, 25.0, 22.6, 21.4, 21.0, 20.8, 19.4, 14.2, 13.8; **HRMS (ESI)**: m/z for $C_{22}H_{34}OS_2$ $[M+Na]^+$ calcd 401.1943, found 401.1940.

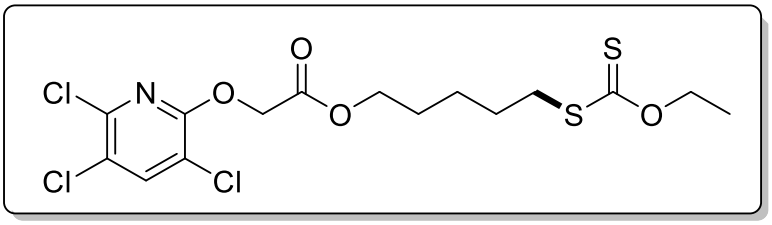


5-[(Ethoxycarbonothioyl)thio]pentyl benzo[d][1,3]dioxole-5-carboxylate (7ca): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl benzo[d][1,3]dioxole-5-carboxylate (**7c**) (85.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ca** (33.0 mg, 46%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.57 (d, *J* = 8.4 Hz, 1H), 7.38 (s, 1H), 6.76 (d, *J* = 8.4 Hz, 1H), 5.96 (s, 2H), 4.57 (q, *J* = 7.2 Hz, 2H), 4.21 (t, *J* = 6.4 Hz, 2H), 3.07 (t, *J* = 7.2 Hz, 2H), 1.70 (q, *J* = 8.0 Hz, 4H), 1.51 – 1.46 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 165.9, 151.5, 147.6, 125.2, 124.3, 109.4, 107.9, 101.7, 69.8, 64.5, 35.6, 28.2, 28.0, 25.3, 13.7; **HRMS (ESI)**: m/z for $C_{16}H_{20}O_5S_2$ $[M+Na]^+$ calcd 379.0644, found 379.0640.

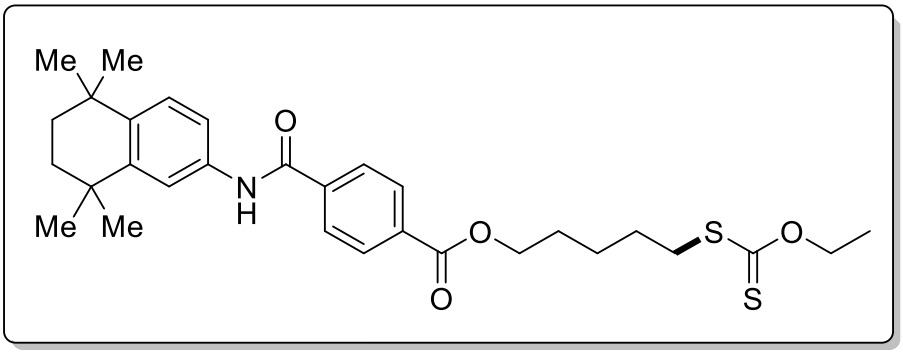


5-[(Ethoxycarbonothioyl)thio]pentyl 2-((3-(trifluoromethyl)phenyl) amino) benzo

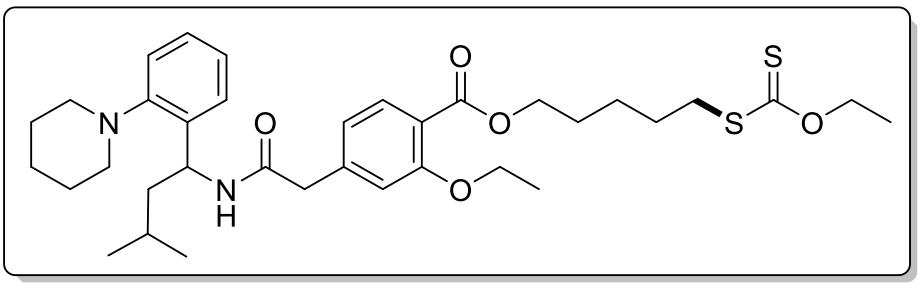
ate (7da): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 2-((3-(trifluoromethyl)phenyl)amino)benzoate (**7d**) (108.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7da** (44.7 mg, 47%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 9.55 (s, 1H), 7.92 – 7.90 (m, 1H), 7.41 (s, 1H), 7.35 – 7.27 (m, 3H), 7.21 – 7.20 (m, 2H), 6.80 – 6.73 (m, 1H), 4.56 (q, *J* = 7.0 Hz, 2H), 4.24 (t, *J* = 6.5 Hz, 2H), 3.10 – 3.06 (m, 2H), 1.78 – 1.68 (m, 4H), 1.55 – 1.49 (m, 2H), 1.33 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 168.3, 146.7, 141.6, 134.2, 131.8 (q, ^2J = 32.0 Hz), 131.7, 129.8, 124.4, 121.2 (q, ^1J = 270.6 Hz), 119.4 (q, ^4J = 3.9 Hz), 118.3, 117.9 (q, ^4J = 3.9 Hz), 114.3, 113.1, 69.8, 64.5, 35.6, 28.2, 28.1, 25.4, 13.8; **^{19}F NMR (377 MHz, $CDCl_3$)** δ -62.79; **HRMS (ESI)**: m/z for $C_{22}H_{24}F_3NO_3S_2$ $[M+Na]^+$ calcd 494.1042, found 494.1040.



5-[(Ethoxycarbonothioyl)thio]pentyl 2-((3,5,6-trichloropyridin-2-yl)oxy)acetate (7ea): The representative procedure was followed using 11,3-dioxoisoindolin-2-yl 6-(2-((3,5,6-trichloropyridin-2-yl)oxy)acetoxy)hexanoate (**7e**) (102.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ea** (44.3 mg, 50%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.70 (s, 1H), 4.86 (s, 2H), 4.58 (q, *J* = 7.2 Hz, 2H), 4.13 (t, *J* = 6.4 Hz, 2H), 3.04 (t, *J* = 7.2 Hz, 2H), 1.66 – 1.59 (m, 4H), 1.37 – 1.33 (m, 5H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 167.7, 155.6, 143.1, 140.5, 122.8, 117.1, 69.8, 65.1, 63.8, 35.6, 28.1, 27.9, 25.1, 13.8; **HRMS (ESI)**: m/z for $C_{15}H_{18}Cl_3NO_4S_2$ $[M+Na]^+$ calcd 467.9635, found 467.9631.

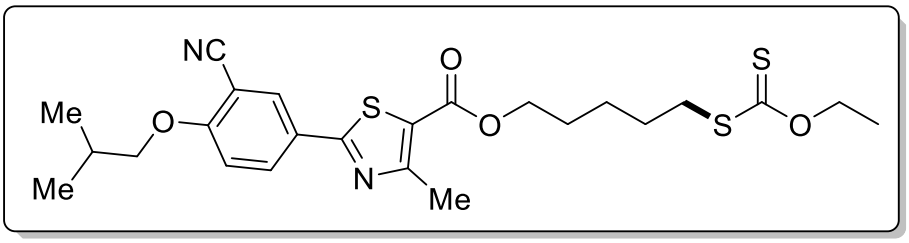


5-[(Ethoxycarbonothioyl)thio]pentyl 4-((5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)carbamoyl)benzoate (7fa): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 4-((5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)carbamoyl)benzoate (**7f**) (122.1 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7fa** (46.4 mg, 43%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 8.02 (d, *J* = 8.4 Hz, 2H), 7.90 (s, 1H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.47 (s, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.22 (d, *J* = 8.4 Hz, 1H), 4.57 (q, *J* = 7.2 Hz, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 3.08 (t, *J* = 7.2 Hz, 2H), 1.79 – 1.67 (m, 4H), 1.61 (s, 4H), 1.53 – 1.46 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H), 1.21 (d, *J* = 4.8 Hz, 12H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 165.7, 145.8, 141.7, 139.0, 135.0, 129.9, 127.2, 127.1, 118.2, 69.9, 65.1, 35.6, 35.0, 35.0, 34.4, 34.0, 31.8, 31.8, 28.2, 28.0, 25.3, 13.8; **HRMS (ESI)**: m/z for $C_{30}H_{39}NO_4S_2$ $[M+Na]^+$ calcd 564.2213, found 564.2218.

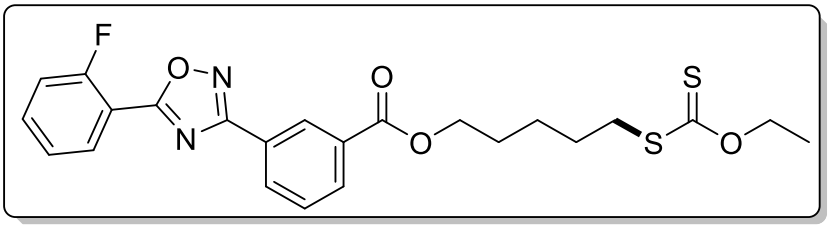


5-[(Ethoxycarbonothioyl)thio]pentyl 2-ethoxy-4-(2-((3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)amino)-2-oxoethyl)benzoate (7ga): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 2-ethoxy-4-(2-((3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)amino)-2-oxoethyl)benzoate (**7g**) (142.3 mg,

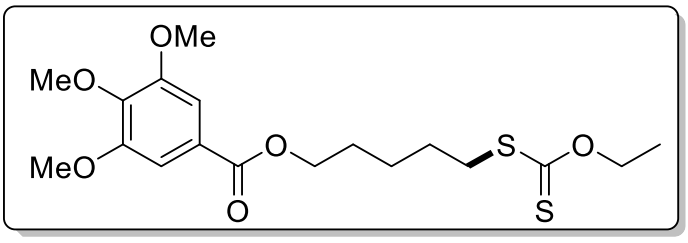
0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ga** (65.3 mg, 51%) as yellow solid; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 7.66 (d, *J* = 7.5 Hz, 1H), 7.14 – 7.11 (m, 2H), 7.00 – 6.97 (m, 2H), 6.78 – 6.75 (m, 2H), 6.65 (d, *J* = 7.5 Hz, 1H), 5.33 – 5.28 (m, 1H), 4.57 (q, *J* = 7.0 Hz, 2H), 4.21 (t, *J* = 6.5 Hz, 2H), 3.97 – 3.90 (m, 2H), 3.46 (s, 2H), 3.10 – 3.03 (m, 2H), 2.86 (s, 2H), 2.54 (s, 2H), 1.73 – 1.67 (m, 4H), 1.65 – 1.63 (m, 2H), 1.53 (s, 2H), 1.52 – 1.50 (m, 2H), 1.50 – 1.48 (m, 2H), 1.46 – 1.42 (m, 2H), 1.33 (d, *J* = 7.0 Hz, 6H), 1.04 – 0.94 (m, 1H), 0.84 (d, *J* = 6.5 Hz, 6H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 214.9, 168.7, 166.3, 158.8, 152.5, 141.1, 138.6, 132.1, 127.9, 127.6, 125.0, 122.7, 120.7, 119.2, 113.7, 69.8, 64.4, 60.7, 49.7, 46.6, 44.2, 35.6, 33.9, 29.6, 28.2, 28.1, 26.7, 25.4, 25.3, 24.1, 22.7, 22.5, 14.7, 13.7; **HRMS (ESI)**: m/z for $C_{35}H_{50}N_2O_5S_2$ $[M+Na]^+$ calcd 665.3053, found 665.3057.



5-[(Ethoxycarbonothioyl)thio]pentyl 2-(3-cyano-4-isobutoxyphenyl)-4-methylthia zole-5-carboxylate (7ha): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 2-(3-cyano-4-isobutoxyphenyl)-4-methylthia zole-5-carboxylate (**7h**) (115.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ha** (50.3 mg, 50%) as yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ** 8.10 (d, *J* = 1.5 Hz, 1H), 8.02 – 8.00 (m, 1H), 6.95 (s, 1H), 4.57 (q, *J* = 7.0 Hz, 2H), 4.27 (t, *J* = 7.0 Hz, 2H), 3.83 (s, 2H), 3.13 – 3.06 (m, 2H), 2.69 (s, 3H), 2.14 (d, *J* = 7.0 Hz, 1H), 1.75 – 1.68 (m, 4H), 1.53 – 1.48 (m, 2H), 1.32 (d, *J* = 4.0 Hz, 3H), 1.02 (d, *J* = 6.4 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$) δ** 214.9, 167.1, 162.4, 162.0, 161.0, 132.5, 132.5, 132.0, 126.0, 121.9, 115.3, 112.6, 102.9, 75.7, 69.8, 65.0, 61.3, 35.6, 28.1, 25.3, 19.0, 17.4, 14.3, 13.8; **HRMS (ESI)**: m/z for $C_{24}H_{30}N_2O_4S_3$ $[M+Na]^+$ calcd 529.1260, found 529.1267.

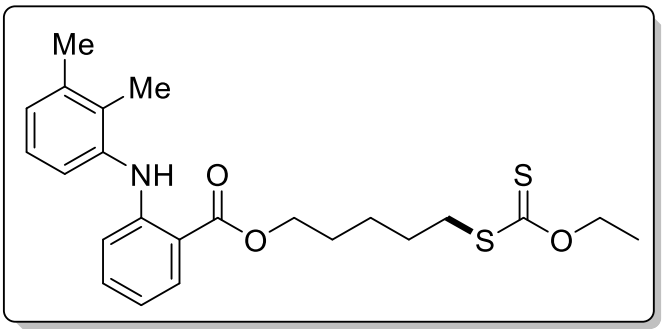


5-[(Ethoxycarbonothioyl)thio]pentyl 3-[5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl]benzoate (7ia): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 3-(5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl)benzoate (**7i**) (108.6 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ia** (51.4 mg, 54%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 8.76 (s, 1H), 8.30 (d, *J* = 8.0 Hz, 1H), 8.19 – 8.12 (m, 2H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.30 – 7.22 (m, 2H), 4.57 (q, *J* = 7.0 Hz, 2H), 4.31 (t, *J* = 6.5 Hz, 2H), 3.09 (t, *J* = 7.5 Hz, 2H), 1.82 – 1.70 (m, 4H), 1.55 – 1.51 (m, 2H), 1.34 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 215.0, 168.1, 165.9, 160.8(d, ^1J = 260.0 Hz), 134.7 (d, ^3J = 8.8 Hz), 132.2, 131.7, 131.2, 131.0, 129.0, 128.7, 127.2, 124.7(d, ^4J = 3.8 Hz), 117.2(d, ^2J = 20.0 Hz), 112.7(d, ^2J = 11.3 Hz), 100.0, 69.8, 65.0, 35.6, 28.3, 28.1, 25.3, 13.8; **^{19}F NMR (471 MHz, $CDCl_3$)** δ -108.17; **HRMS (ESI)**: m/z for $C_{23}H_{23}FN_2O_4S_2$ $[M+Na]^+$ calcd 497.0975, found 497.0982.

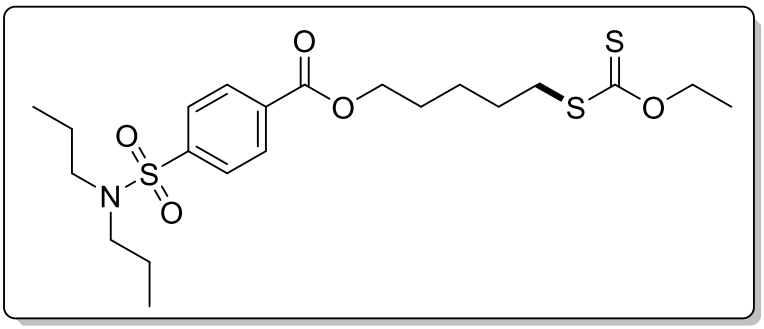


5-[(Ethoxycarbonothioyl)thio]pentyl 3,4,5-trimethoxybenzoate (7ja): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 3,4,5-trimethoxybenzoate (**7j**) (94.2 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ja** (48.1 mg, 60%) as yellow oill; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 7.29 (s, 2H), 4.63 (q, *J* = 7.0 Hz,

2H), 4.32 (t, *J* = 6.5 Hz, 2H), 3.91 (s, 9H), 3.15 (t, *J* = 7.0 Hz, 2H), 1.84 – 1.75 (m, 4H), 1.57 (q, *J* = 8.0 Hz, 2H), 1.40 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 214.8, 166.1, 152.8, 152.8, 142.1, 125.4, 125.3, 106.7, 69.7, 64.7, 61.0, 60.8, 56.1, 56.1, 35.6, 28.2, 27.9, 25.2, 14.3, 13.7; **HRMS (ESI)**: m/z for $C_{18}H_{26}O_6S_2$ $[M+Na]^+$ calcd 425.1063, found 425.1061.



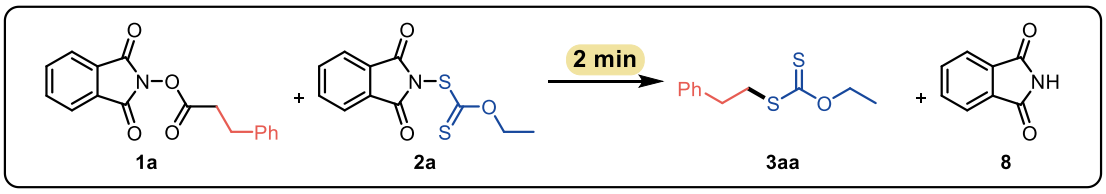
5-[(Ethoxycarbonothioyl)thio]pentyl 2-((2,3-dimethylphenyl)amino)benzoate (7ka): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 2-((2,3-dimethylphenyl)amino)benzoate (**7k**) (100.0 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7ka** (55.6 mg, 64%) as yellow oill; **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 9.18 (s, 1H), 7.90 – 7.86 (m, 1H), 7.18 – 7.13 (m, 1H), 7.08 (d, *J* = 7.5 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 6.61 – 6.55 (m, 1H), 4.57 (q, *J* = 7.0 Hz, 2H), 4.23 (t, *J* = 6.5 Hz, 2H), 3.08 (t, *J* = 7.5 Hz, 2H), 2.25 (s, 3H), 2.10 (s, 3H), 1.77 – 1.69 (m, 4H), 1.54 – 1.50 (m, 2H), 1.34 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 215.0, 168.6, 149.4, 138.7, 138.1, 134.1, 132.3, 131.4, 126.7, 125.9, 122.9, 116.0, 113.6, 110.9, 69.8, 64.2, 35.6, 28.3, 28.1, 25.4, 20.6, 14.0, 13.8; **HRMS (ESI)**: m/z for $C_{23}H_{29}NO_3S_2$ $[M+Na]^+$ calcd 454.1481, found 454.1478.



5-[(Ethoxycarbonothioyl)thio]pentyl 4-(*N*,*N*-dipropylsulfamoyl)benzoate (7la): The representative procedure was followed using 6-((1,3-dioxoisoindolin-2-yl)oxy)-6-oxohexyl 4-(N,N-dipropylsulfamoyl)benzoate (**7l**) (108.8 mg, 0.20 mmol) and *S*-(1,3-dioxoisoindolin-2-yl) *O*-ethyl carbonodithioate (**2a**) (106.8 mg, 0.40 mmol). Isolation by column chromatography (petroleum ether : EtOAc = 20:1) yielded **7la** (45.1 mg, 47%) as yellow oill; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.08 (d, J = 8.0 Hz, 2H), 7.80 (d, J = 8.0 Hz, 2H), 4.56 (q, J = 7.2 Hz, 2H), 4.28 (t, J = 6.4 Hz, 2H), 3.10 – 3.00 (m, 6H), 1.81 – 1.65 (m, 4H), 1.54 – 1.42 (m, 6H), 1.33 (t, J = 7.2 Hz, 3H), 0.79 (t, J = 7.2 Hz, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 214.7, 165.0, 144.0, 133.5, 130.0, 126.8, 69.7, 65.1, 49.8, 35.4, 28.0, 27.9, 25.1, 21.8, 13.6, 11.0; **HRMS (ESI)**: m/z for $C_{21}H_{33}NO_5S_3$ $[M+Na]^+$ calcd 498.1413, found 498.1410.

5. Applications

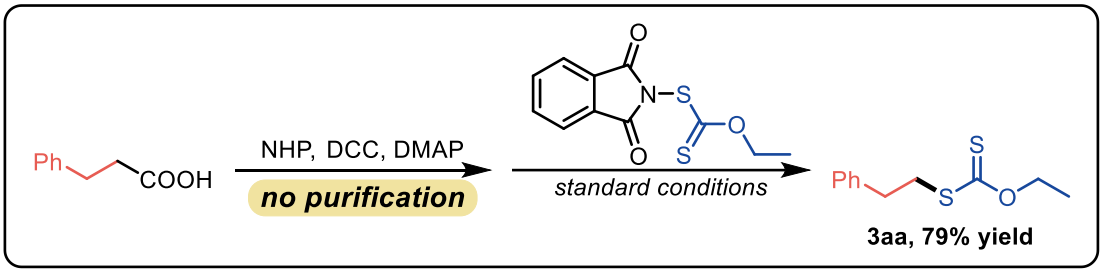
Gram Scale Synthesis:



N-Hydroxyphthalimide ester **1a** (2.95 g, 10.0 mmol), *N*-ethylxanthylphthalimide **2a** (5.34 g, 20.0 mmol), Mn power (11.0 g, 20.0 mmol) were placed into an oven-dried flask vial that was equipped with a stirring bar. The vessel was evacuated and filled with N_2 (three times). The chlorotrimethylsilane (2.17 g, 2.5 mL, 20.0 mmol) was added, followed by the addition of DMF (25.0 mL) *via* syringe. The reaction mixture

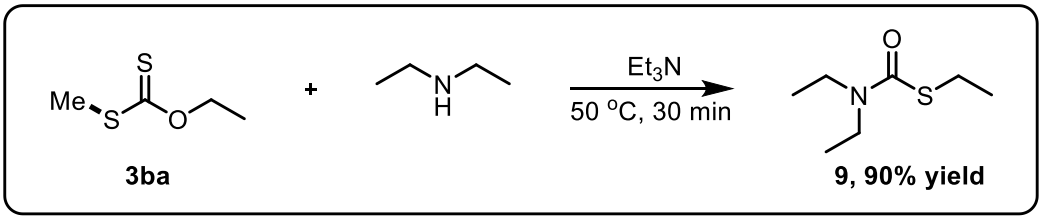
was stirred at r.t. for 2 min. After this time, the crude reaction mixture was diluted with ethyl acetate and washed with water. The aqueous layer was then extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The solvent was removed under vacuum and the residue was purified by flask column chromatography to afford the pure product **3aa** as yellow oil (1.81 g, 8.0 mmol, 80% yield) and isoindoline-1,3-dione **8** as white solid (3.26 g, 22.2 mmol, 74% yield).

One-pot synthesis:

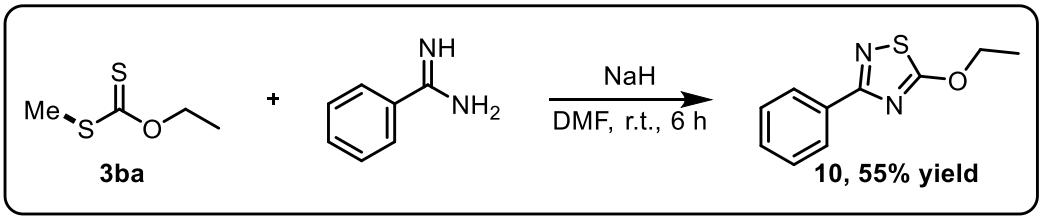


An oven-dried 10 mL Schlenk tube with a magnetic stir bar charged with benzenepropionic acid (0.5 mmol), *N*-hydroxyphthalimide(1.1 equiv), and DMAP (0.1 equiv). Dichloromethane was added (0.2 M), and the mixture was stirred. DCC (1.1 equiv) was then added and the mixture allowed to stir until the acid was consumed (determined by TLC). Then, the reaction mixture was filtered add added water, and organics were extracted in DCM. After drying with Na_2SO_4, the organics were evaporated, and a solid product was obtained. Then, the solid product, *N*-ethylxanthylphthalimide **2a** (1.0 mmol, 2.0 equiv), Mn power (1.0 mmol) were placed into an oven-dried 10 mL Schlenk tube that was equipped with a stirring bar. The vessel was evacuated and filled with N_2 (three times). The chlorotrimethylsilane (1.0 mmol) was added, followed by the addition of DMF (1.50 mL) *via* syringe. The reaction mixture was stirred at r.t. for 2 min. After this time, the crude reaction mixture was diluted with ethyl acetate (5.0 mL) and washed with water (3.0 mL). The aqueous layer was then extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The

solvent was removed under vacuum and the residue was purified by flask column chromatography to afford the product **3aa** as yellow oil (89.3 mg, 0.40 mmol, 79% yield).

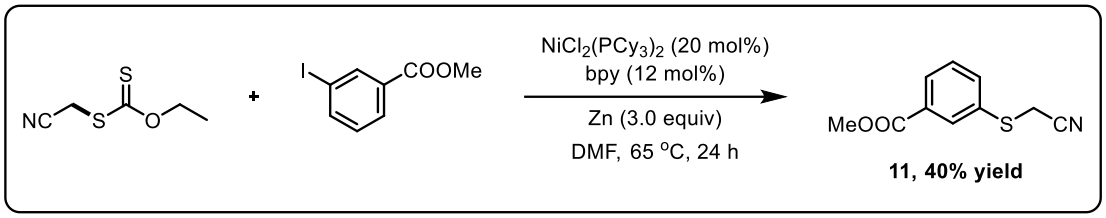


Adapted from the literature procedure.[9] MeSC(S)OEt (**3ba**; 136.0 mg, 1.0 mmol) was heated to 50 °C in an oil bath and a mixture of neat Et_2NH (80.4 mg, 1.1 mmol) and Et_3N (30.3 mg, 0.3 mmol) was added dropwise over 8–10 min with stirring. The reaction was complete after 30 min when **3ba** had disappeared. After this time, the crude reaction mixture was diluted with DCM and water. The aqueous layer was then extracted with DCM, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The solvent was removed under vacuum and the residue was purified by flask column chromatography (petroleum ether : EtOAc = 30:1) yielded **9** (145.0 mg, 90%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$)** ***δ*** 4.43 (q, *J* = 7.0 Hz, 2H), 3.75 (q, *J* = 7.0 Hz, 2H), 3.40 (q, *J* = 7.0 Hz, 2H), 1.26 (t, *J* = 7.0 Hz, 3H), 1.16 (t, *J* = 7.0 Hz, 3H), 1.09 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** ***δ*** 186.9, 66.5, 47.1, 42.8, 14.0, 12.8, 11.6.

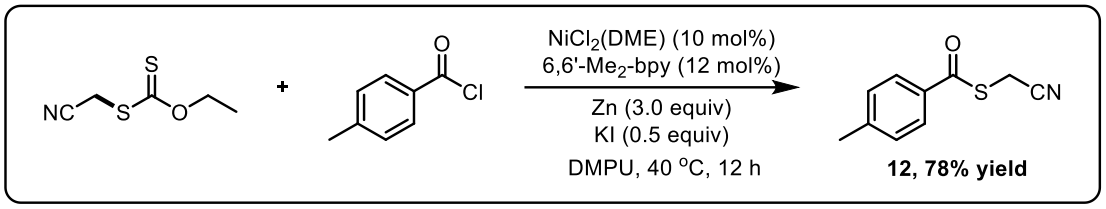


Adapted from the literature procedure.[10] To a stirred suspension of NaH (60% suspension in mineral oil) (67 mg, 2 mmol, 2.0 equiv) in dry DMF (3 mL, 0.33M), benzimidamide (1 mmol, 1.0 equiv) was added at room temperature under N_2 atmosphere. After stirring for 5-10 min, a solution of **3ba** (1 mmol, 1.0 equiv) in DMF (3 mL, 0.33M) was added dropwise and stirring was continued at room temperature

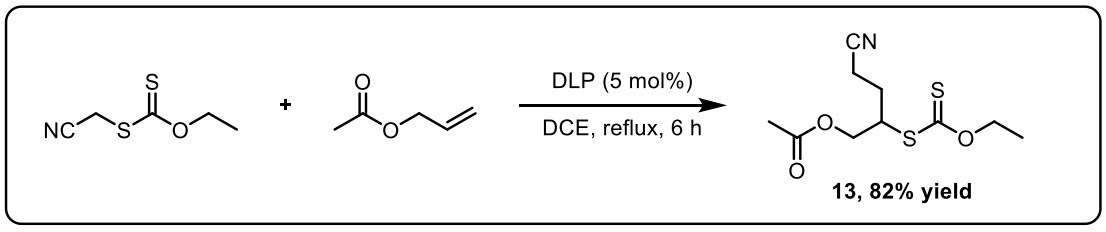
for 4-6 h (monitored by TLC). The reaction mixture was quenched with saturated aqueous NH_4Cl solution, extracted with EtOAc, the combined organic layer was washed with water, and brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The solvent was removed under vacuum and the residue was purified by flask column chromatography (petroleum ether : EtOAc = 5:1) yielded **10** (113.3 mg, 55%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 7.77 (d, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 4.42 (q, *J* = 7.0 Hz, 2H), 1.32 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 199.3, 166.6, 134.6, 132.4, 128.7, 127.2, 65.9, 14.1.



Adapted from the literature procedure.[11] An oven-dried 100 x 16 mm screw-capped vial with a magnetic stir bar was transferred to an N_2-filled glovebox. First, bpy (6.6 mg, 0.036 mmol, 12 mol%), $NiCl_2(PCy_3)_2$ (41.4 mg, 0.060 mmol, 20 mol%), Zn (117.9 mg, 0.90 mmol, 3.0 equiv), and DMF (1.50 mL, 0.20 M) were added to the vial sequentially. Next, methyl 3-iodobenzoate (0.48 mmol, 1.6 equiv) and *S*-(cyanomethyl) *O*-ethyl carbonodithioate (0.30 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined screw cap and removed from the glovebox. The reaction was stirred at 65 ℃ for 24 h, then quenched upon the addition of H_2O. The aqueous layer was extracted with EtOAc, and the combined organic layers were extracted with H_2O. The combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by flask column chromatography (petroleum ether : EtOAc = 5:1) yielded **11** (24.8 mg, 40%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$) δ** 8.12 (s, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 8.0 Hz, 1H), 3.87 (s, 3H), 3.56 (s, 2H); **^{13}C NMR (125 MHz, $CDCl_3$) δ** 166.0, 136.4, 133.0, 132.7, 131.6, 129.9, 129.6, 116.0, 52.4, 21.1.



Adapted from the literature procedure.[11] An oven-dried 100 x 16 mm screw-capped vial with a magnetic stir bar was transferred to an N_2-filled glovebox. First, 6,6'-Dimethyl-2,2'-dipyridyl (6.6 mg, 0.036 mmol, 12 mol%), $NiCl_2$(DME) (9.3 mg, 0.030 mmol, 10 mol%), KI (24.9 mg, 0.15 mmol, 50 mol%), Zn (58.9 mg, 0.90 mmol, 3.0 equiv) and DMPU (1.5 mL, 0.20 M) were added to the vial sequentially. Next, 4-methylbenzoyl chloride (47.6 μL, 0.36 mmol, 1.2 equiv) and *S*-(cyanomethyl) *O*-ethyl carbonodithioate (0.30 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined screw cap and removed from the glovebox. The reaction was stirred at 40 ℃ for 12 h, then quenched upon the addition of H_2O. The aqueous layer was extracted with EtOAc, and the combined organic layers were extracted with H_2O. The combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by flask column chromatography (petroleum ether : EtOAc = 5:1) yielded **12** (44.7 mg, 78%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.75 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 3.77 (s, 2H), 2.35 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 187.3, 145.6, 132.6, 129.5, 127.5, 115.9, 21.7, 14.2.



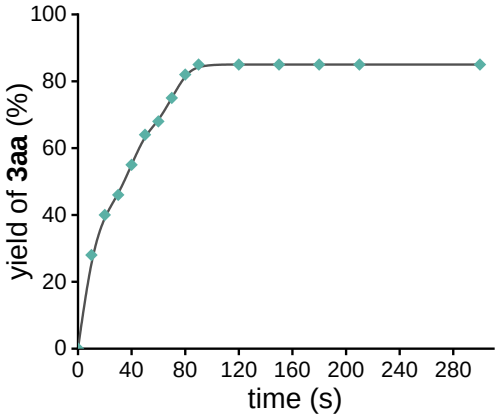
Adapted from the literature procedure.[12] To a 1-dram vial with a magnetic stir bar was added *S*-(cyanomethyl) *O*-ethyl carbonodithioate (1.0 mmol, 1 equiv) and dilauroyl peroxide (0.05 equiv). The vial was brought into the glovebox, and allyl acetate (2.0 mmol, 2 equiv) was added, followed by 1,2- dichloroethane (1 M). The vial was placed under a balloon of argon and heated at 85 ℃ for 2 h. If tertiary xanthate

remained by TLC (generally a spot with a higher Rf than the desired addition product), the vial was cooled to rt and brought back into the glovebox. Additional DLP (0.1 equiv) was added, and the reaction was stirred at 84 ℃ for 4 h, then quenched upon the addition of H_2O. The aqueous layer was extracted with EtOAc, and the combined organic layers were extracted with H_2O. The combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by flask column chromatography (petroleum ether : EtOAc = 5:1) yielded **13** (214.1 mg, 82%) as yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 4.66 (q, J = 7.0 Hz, 2H), 4.38 – 4.30 (m, 1H), 4.27 – 4.19 (m, 1H), 4.09 – 4.03 (m, 1H), 2.63 – 2.48 (m, 2H), 2.23 – 2.16 (m, 1H), 2.08 (s, 3H), 2.03 – 1.95 (m, 1H), 1.43 (t, J = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 211.4, 170.3, 118.5, 70.6, 64.8, 48.0, 27.0, 20.6, 14.9, 13.6.

6. Kinetic studies

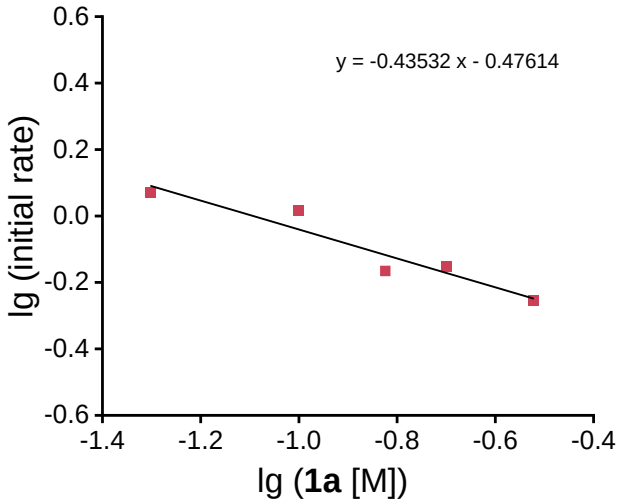
6.1 Time-yield curve

The reaction was performed with **1a** (0.30 mmol), **2a** (0.60 mmol, 2.0 equiv), Mn power (33.0 mg, 0.60 mmol) and chlorotrimethylsilane (0.60 mmol) in DMA. The reaction tube was rapidly placed on a stir plate (stirring at 450 rpm) which signified time = 0. Aliquots (~20 μL) were removed from the reaction at the indicated times, directly injected into 1.0 mL ethyl acetate: H_2O (1:1) in a vial and subjected to analysis. Time-yield curve indicated that the reaction started up very fleetly and was completely finished within two minutes.



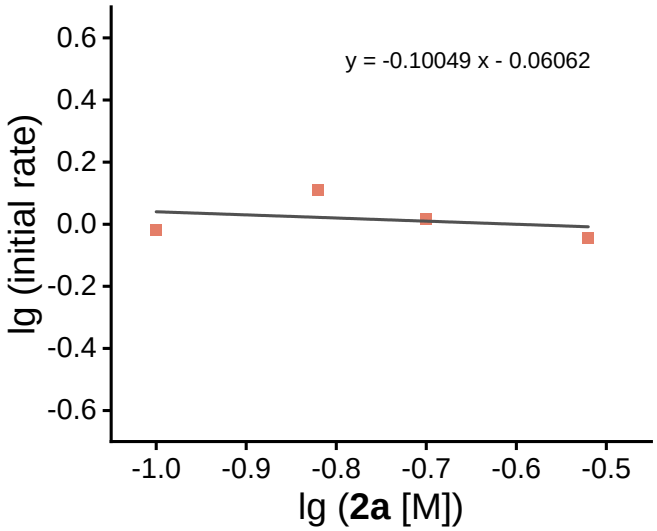
6.2 Order in the *N*-Hydroxyphthalimide ester 1a

The reaction was performed with **2a** (0.60 mmol, 2.0 equiv), Mn power (33.0 mg, 0.60 mmol) and chlorotrimethylsilane (0.60 mmol) in DMA in the presence of 0.05 mmol, 0.10 mmol, 0.15 mmol, 0.20 mmol, 0.30 mmol *N*-Hydroxyphthalimide ester **1a** with the reaction time of 2 min. The initial rates at various **1a** were different, showing a negative order (ca. -0.5-order) dependency on the initial concentration of *N*-Hydroxyphthalimide ester **1a**.



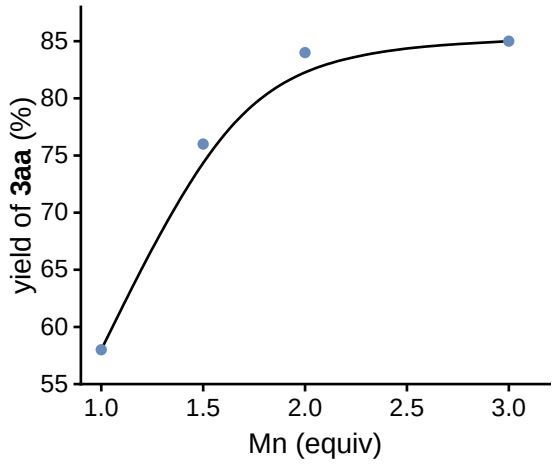
6.3 Order in the *N*-ethylxanthyl phthalimide 2a

The reaction was performed with **1a** (0.30 mmol), Mn power (33.0 mg, 0.60 mmol) and chlorotrimethylsilane (0.60 mmol) in DMA in the presence of 0.05 mmol, 0.10 mmol, 0.15 mmol, 0.20 mmol, 0.30 mmol *N*-ethylxanthyl phthalimide **2a** with the reaction time of 2 min. The almost unchanged initial rates for different **2a** suggested that the reaction is zero-order in the component.



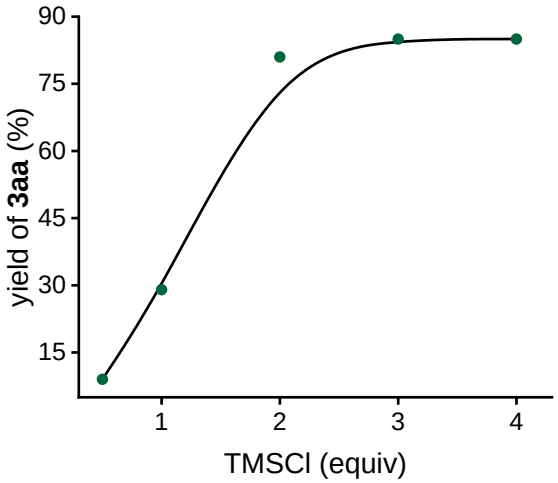
6.4 Effect of reductant input

The reaction was performed with **1a** (0.30 mmol), **2a** (0.60 mmol, 2.0 equiv) and chlorotrimethylsilane (0.60 mmol) in DMA in the presence of 0.30 mmol, 0.45 mmol, 0.60 mmol, 0.90 mmol Mn power with the reaction time of 2 min. The crude reaction mixture was diluted with ethyl acetate and washed with water. The yield was confirmed by GC-MS.



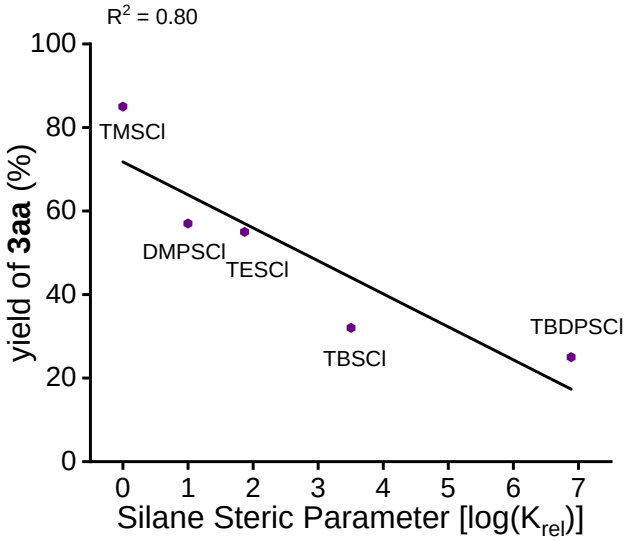
6.5 Effect of additive input

The reaction was performed with **1a** (0.30 mmol), **2a** (0.60 mmol, 2.0 equiv) and Mn power (33.0 mg, 0.60 mmol) in DMA in the presence of 0.15 mmol, 0.30 mmol, 0.60 mmol, 0.90 mmol, 1.20 mmol chlorotrimethylsilane with the reaction time of 2 min. The crude reaction mixture was diluted with ethyl acetate and washed with water. The yield was confirmed by GC-MS.



6.6 Effect of halosilane additives size

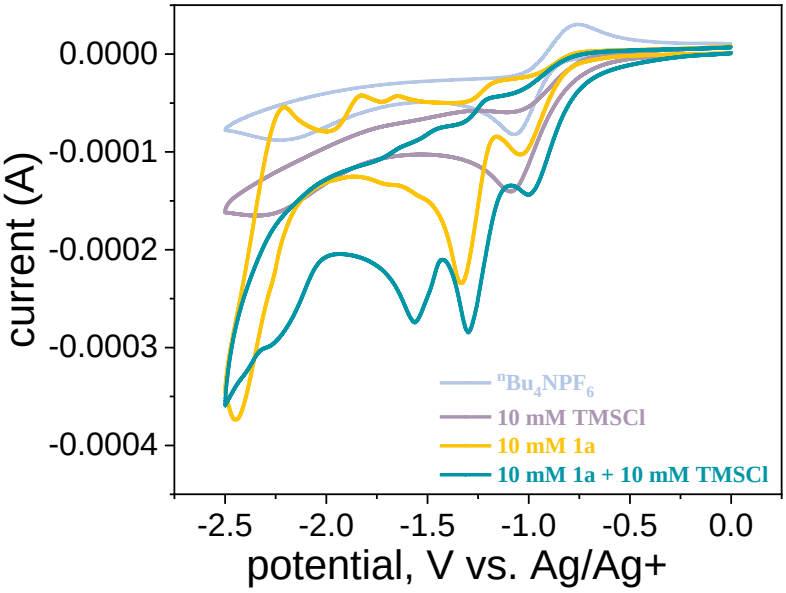
The reaction was performed with **1a** (0.30 mmol), **2a** (0.60 mmol, 2.0 equiv) and Mn power (33.0 mg, 0.60 mmol) in DMA in the presence of 0.60 mmol different halosilane additives with the reaction time of 2 min. The crude reaction mixture was diluted with ethyl acetate and washed with water. The yield was confirmed by GC-MS.

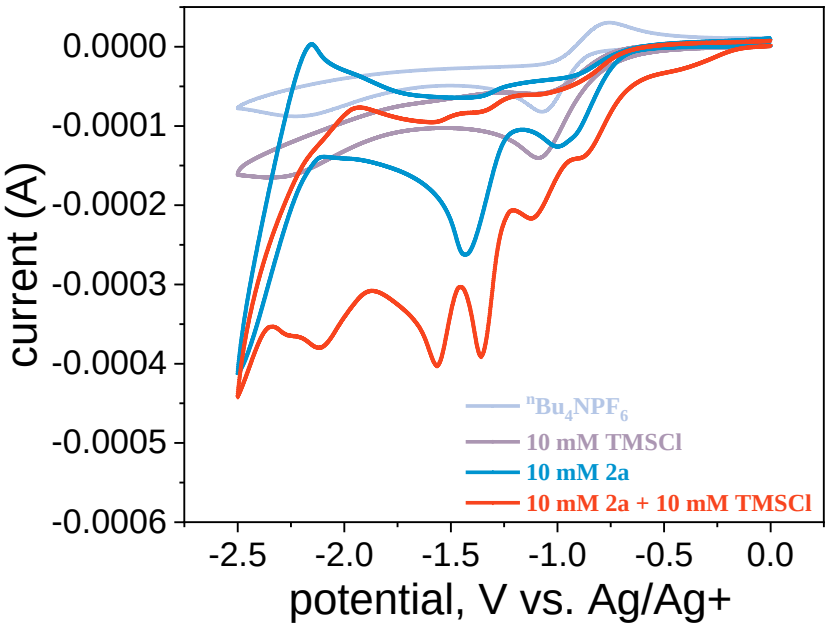


6.7 Cyclic voltammetry experiments

General Details: Cyclic voltammograms were obtained in a N2-filled glovebox using a standard three electrode cell: A glassy-carbon electrode (3mm-diameter, disc-electrode) was used as the working electrode, a Pt plate was used as the auxiliary

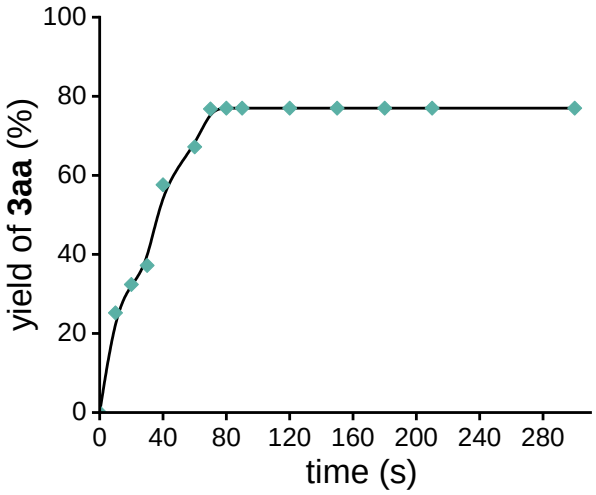
electrode and an Ag/Ag + electrode was used as a reference electrode. The measurements were carried out at a scan rate of 100 mV/s $^{-1}$ in MeCN/ $^{n}Bu_4NPF_4$ (0.1 M).





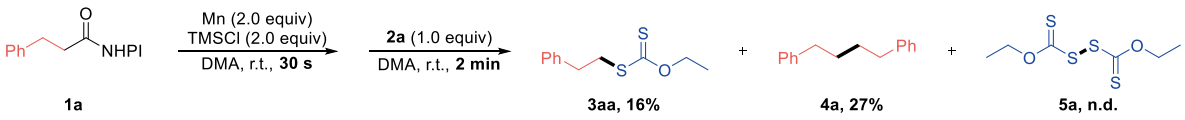
6.8 Time-yield curve at 50 °C

The reaction was performed with **1a** (0.30 mmol), **2a** (0.60 mmol, 2.0 equiv), Mn power (33.0 mg, 0.60 mmol) and chlorotrimethylsilane (0.60 mmol) in DMA at 50 °C. The reaction tube was rapidly placed on a stir plate (stirring at 450 rpm) which signified time = 0. Aliquots (~20 μL) were removed from the reaction at the indicated times, directly injected into 1.0 mL ethyl acetate: H_2O (1:1) in a vial and subjected to analysis. Time-yield curve indicated that the reaction started up very fleetly and was finished completely within 70 seconds accompanied by a slightly reduced yield (77%).



7. Mechanistic studies

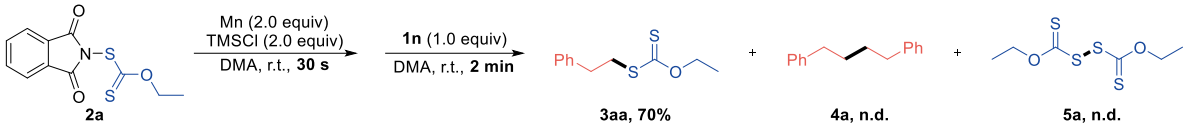
7.1 Sequential experiment



1a **3aa, 16%** **4a, 27%** **5a, n.d.**

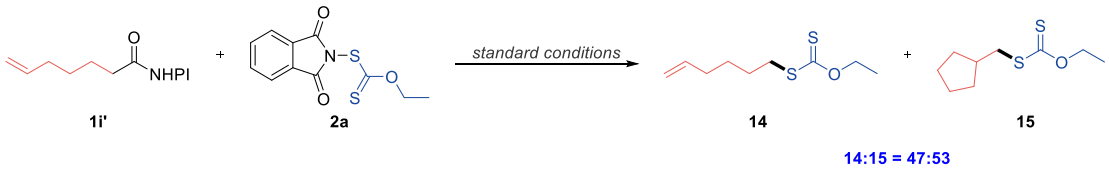
To a 10 ml Schlenk tube was added sequentially Mn (22.0 mg, 0.40 mmol, 2.0 equiv), **1a** (59.0 mg, 0.20 mmol, 1.0 equiv), the chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) was added, followed by the addition of DMA *via* syringe. The resulting solution was stirred for 30 s at r.t. under N_2. Then **2a** (53.4 mg, 0.20 mmol, 1.0 equiv) was added reverse nitrogen flow. The resulting solution was stirred for another 2 min

at room temperature. The crude reaction mixture was diluted with ethyl acetate and washed with water. The yield was confirmed by GC-MS.

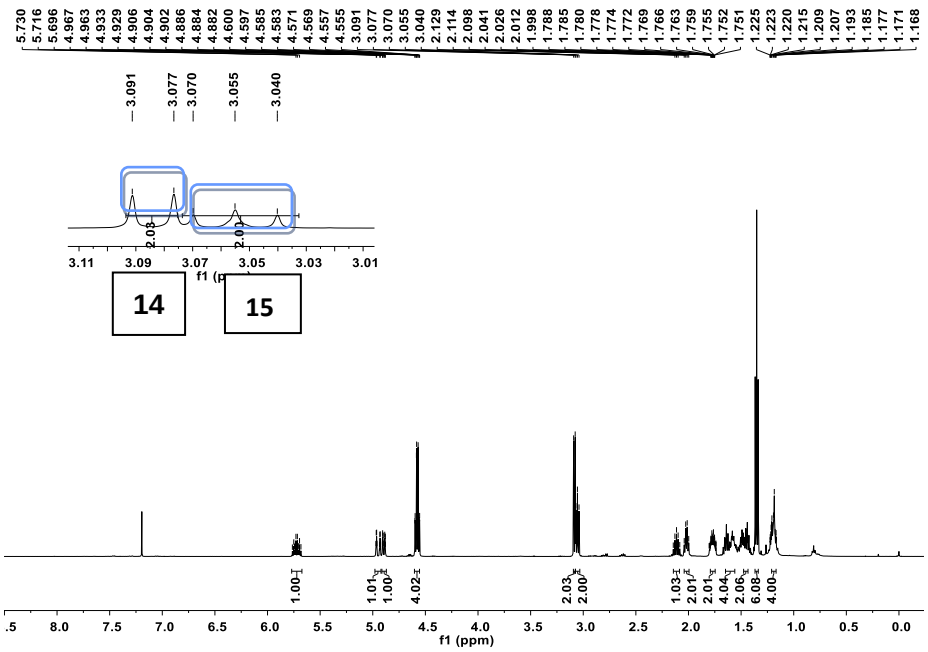


To a 10 ml Schlenk tube was added sequentially Mn (22.0 mg, 0.40 mmol, 2.0 equiv), **2a** (53.4 mg, 0.20 mmol, 1.0 equiv), the chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) was added, followed by the addition of DMA *via* syringe. The resulting solution was stirred for 30 s at r.t. under N_2. Then **1a** (59.0 mg, 0.20 mmol, 1.0 equiv) was added reverse nitrogen flow. The resulting solution was stirred for another 2 min at room temperature. The crude reaction mixture was diluted with ethyl acetate and washed with water. The yield was confirmed by GC-MS.

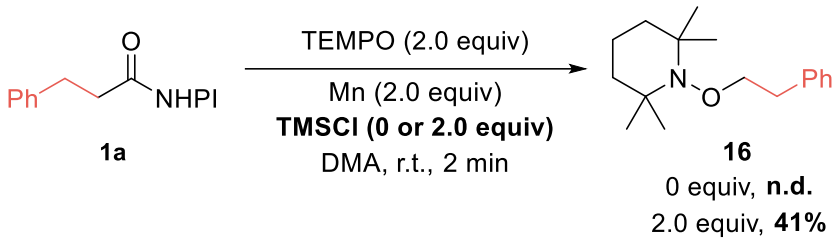
7.2 Radical clock experiment



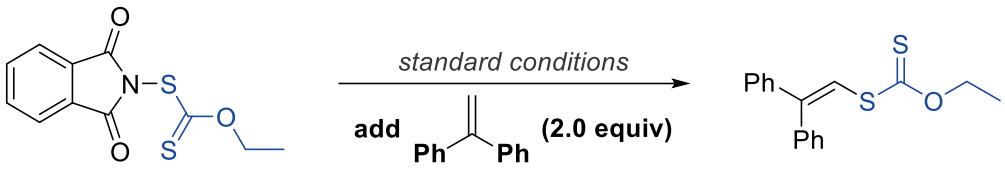
To a 10 ml Schlenk tube was added sequentially **1i'** (54.6 mg, 0.20 mmol, 1.0 equiv), **2a** (106.8 mg, 0.40 mmol, 2.0 equiv), Mn power (22.0 mg, 0.40 mmol, 2.0 equiv). Then chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) was added, followed by the addition of DMA *via* syringe. The reaction mixture was stirred at r.t. for 2 min. After this time, the crude reaction mixture was diluted with ethyl acetate (5.0 mL) and washed with water (3.0 mL). The aqueous layer was then extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The mixture of **14** and **15** was separated by column chromatography (petroleum ether) in the overall yield of 81% as yellow oil.



7.3 Free radical capture experiments



To a 10 ml Schlenk tube was added sequentially tetramethylpiperidinooxy (62.4 mg, 0.40 mmol, 2.0 equiv), **1a** (59.0 mg, 0.20 mmol, 1.0 equiv), Mn power (33.0 mg, 0.60 mmol, 3.0 equiv). The chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) was added, followed by the addition of DMA *via* syringe. The resulting solution was stirred for 2 min at r.t. under N_2. After this time, the crude reaction mixture was diluted with ethyl acetate (5.0 mL) and washed with water (3.0 mL). The aqueous layer was then extracted with ethyl acetate, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated. The solvent was removed under vacuum and the residue was purified by flask column chromatography to afford the pure product **16**. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.23 – 7.09 (m, 5H), 3.87 (t, J = 7.2 Hz, 2H), 2.75 (t, J = 7.2 Hz, 2H), 1.34 – 1.33 (m, 4H), 1.31 – 1.11 (m, 2H), 0.99 (s, 12H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 139.6, 129.1, 128.1, 125.9, 77.5, 59.7, 39.6, 35.4, 32.9, 20.1, 17.1, 1.0.



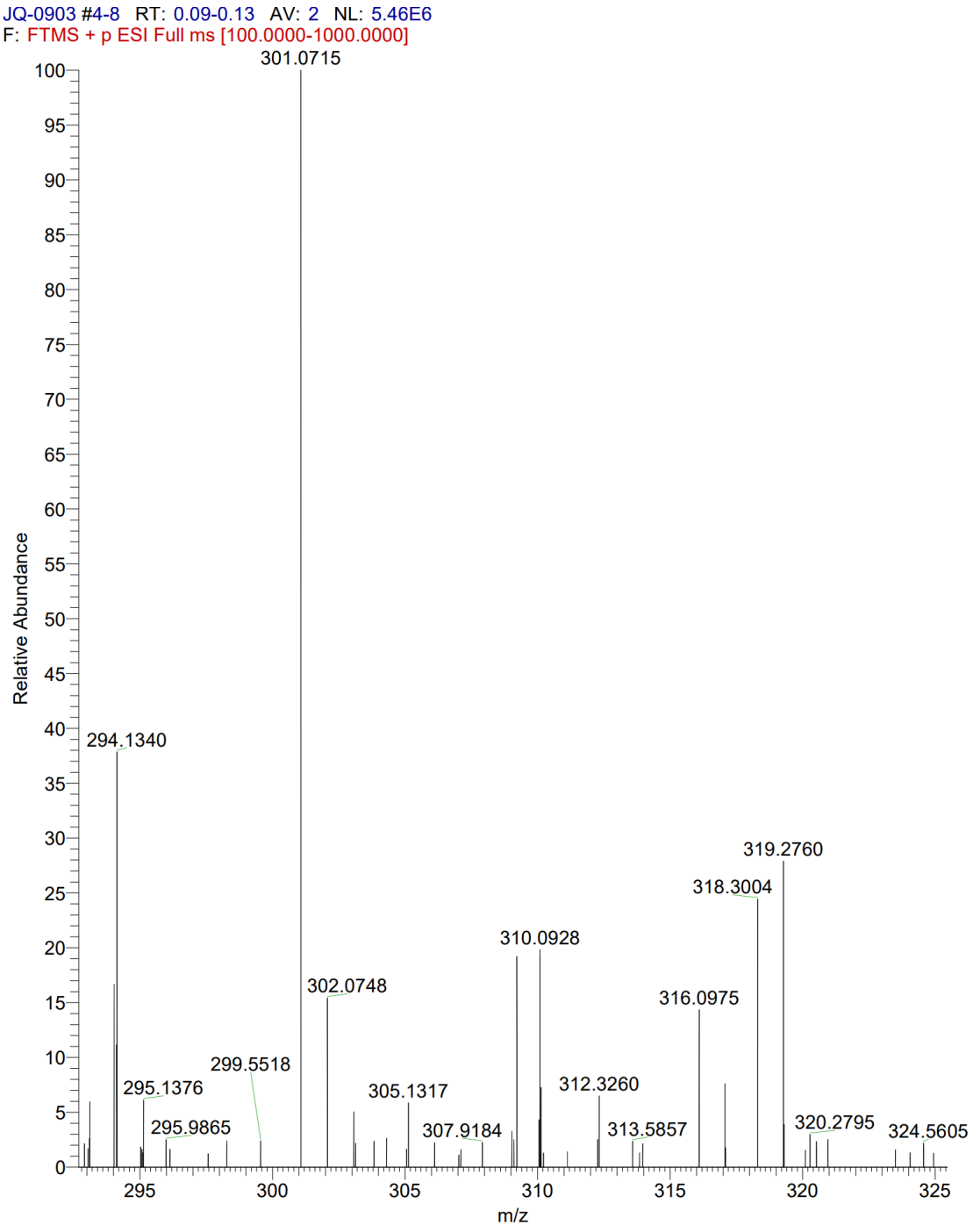
2a **17**

detected by HRMS

$[M+H]^+$ calcd: 301.0715

HRMS found: 301.0715

To a 10 ml Schlenk tube was added sequentially **2a** (53.4 mg, 0.20 mmol, 1.0 equiv), Mn power (33.0 mg, 0.60 mmol, 3.0 equiv). The chlorotrimethylsilane (43.5 mg, 50.7 μL, 0.40 mmol) and ethene-1,1-diyldibenzene (72.0 mg, 0.40 mmol, 2.0 equiv) were added, followed by the addition of DMA *via* syringe. The resulting solution was stirred for 2 min at r.t. under N_2. After this time, the crude reaction mixture was diluted with ethyl acetate (5.0 mL) and washed with water (3.0 mL). The existence of **17** was confirmed by HRMS.


JQ-0903 #4-8 RT: 0.09-0.13 AV: 2 NL: 5.46E6
F: FTMS + p ESI Full ms [100.0000-1000.0000]
301.0715
294.1340
319.2760
318.3004
310.0928
302.0748
316.0975
299.5518
295.1376
305.1317
312.3260
295.9865
307.9184
313.5857
320.2795
324.5605
Relative Abundance
m/z

8. References

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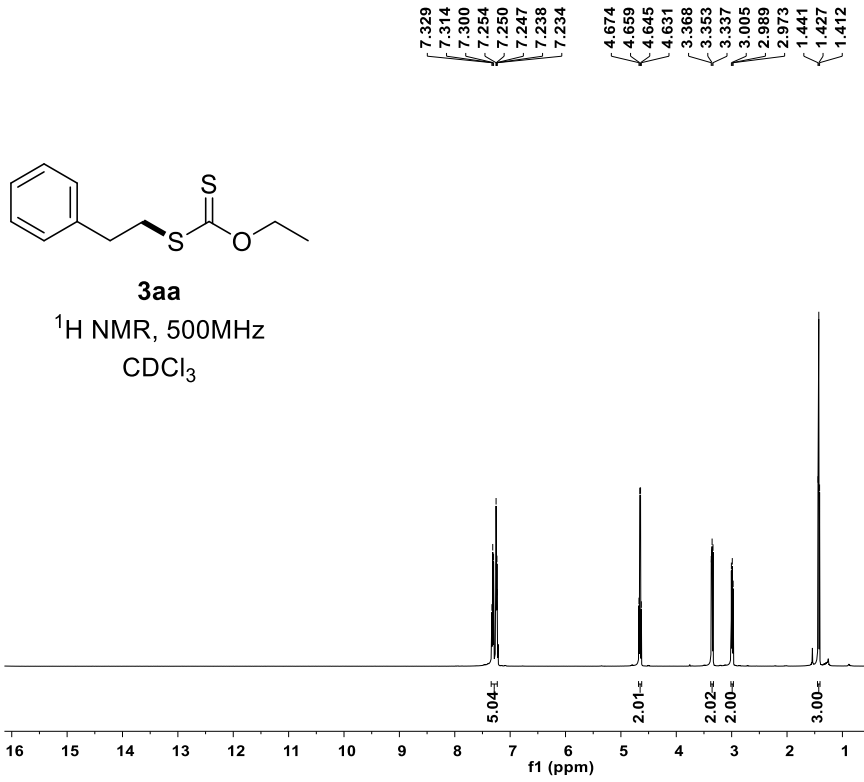
11) L. Tai, L. Chen, Y. Shi and L.-A. Chen, *Org. Chem. Front.*, 2023, **10**, 2505–2516.

12) E. N. Jenkins, W. L. Czaplyski and E. J. Alexanian, *Org. Lett.*, 2017, **19**, 2350−2353.

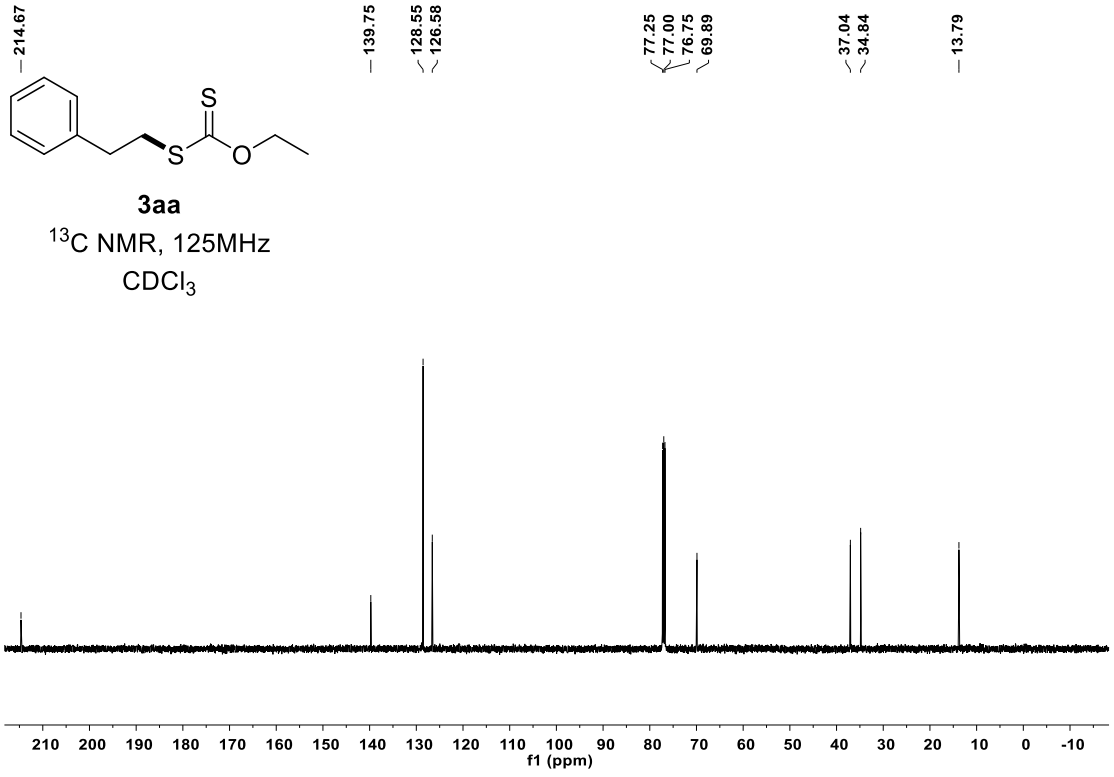
9. NMR Spectrum



3aa

^{1}H NMR, 500MHz

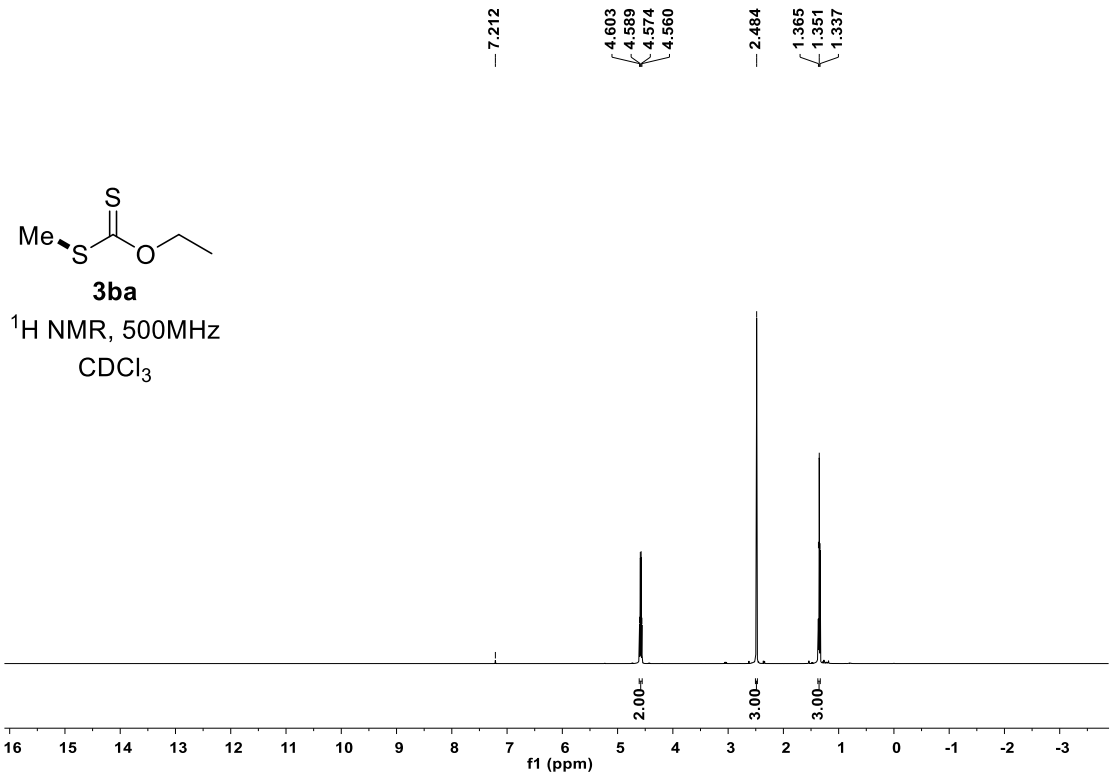
$CDCl_3$

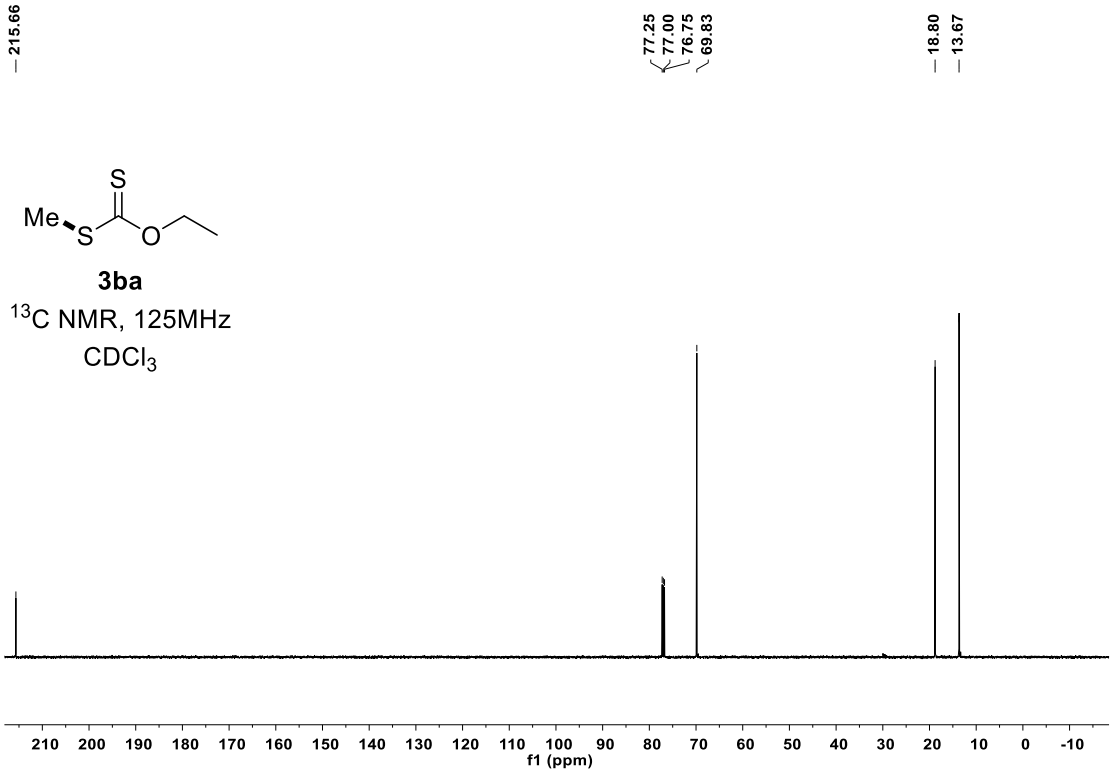


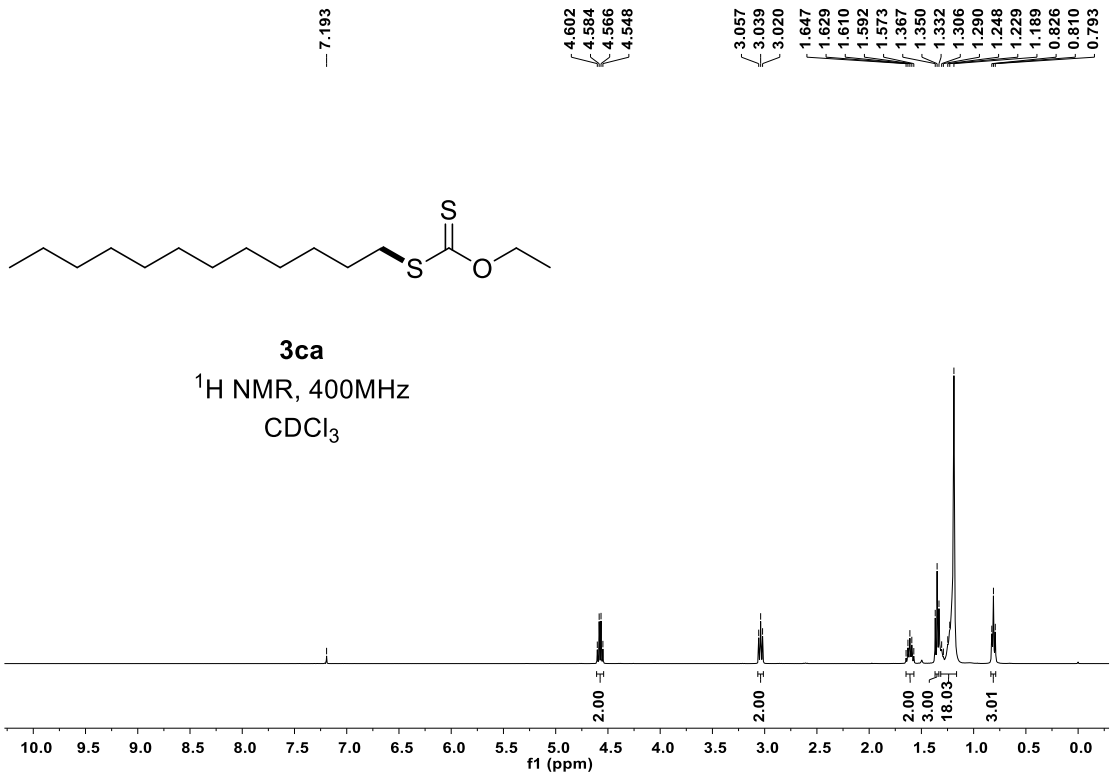
3aa

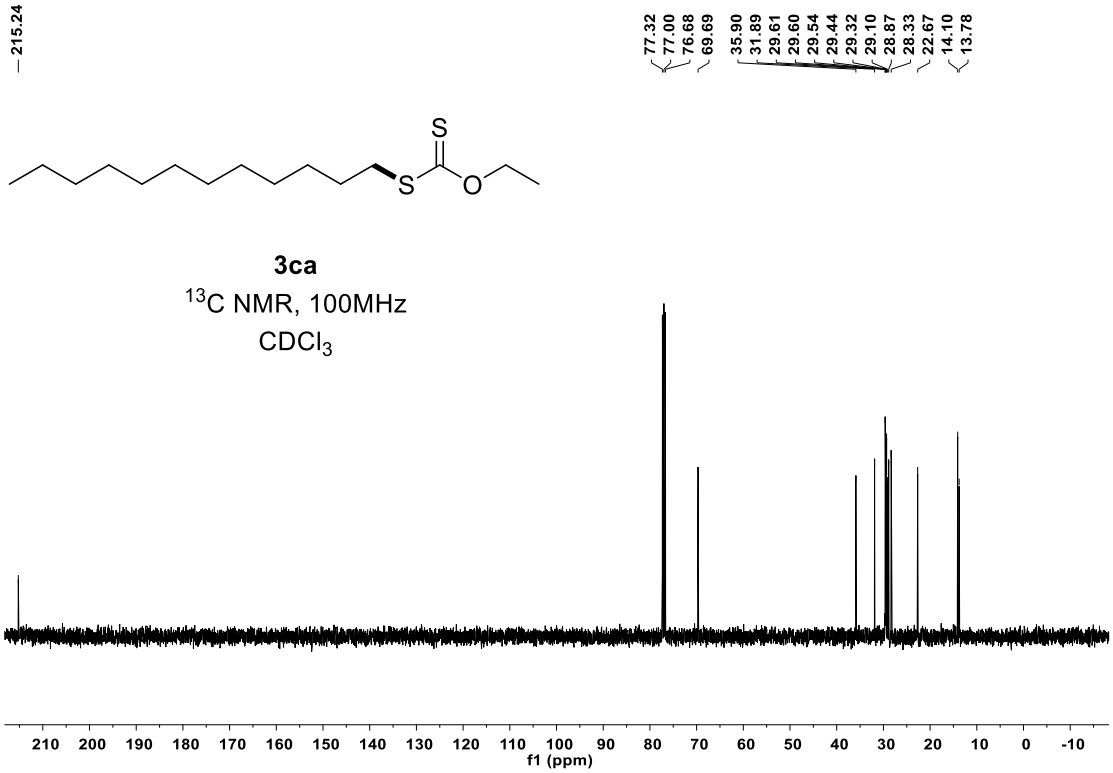
^{13}C NMR, 125MHz

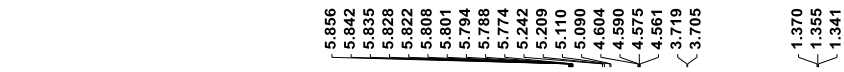
$CDCl_3$

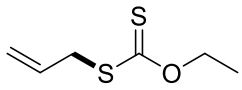

7.212
4.603
4.589
4.574
4.560
2.484
1.365
1.351
1.337
Me
S
S
O
3ba
1H NMR, 500MHz
CDCl3
2.00
3.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


215.66
77.25
77.00
76.75
69.83
18.80
13.67
Me
S
S
O
3ba
13C NMR, 125MHz
CDCl3
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)


7.193
4.602
4.584
4.566
4.548
3.057
3.039
3.020
1.647
1.629
1.610
1.592
1.573
1.367
1.350
1.332
1.306
1.290
1.248
1.229
1.189
0.826
0.810
0.793
S
S
O
3ca
1H NMR, 400MHz
CDCl3
2.00
2.00
2.00
3.00
18.03
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


215.24
77.32
77.00
76.68
69.69
35.90
31.89
29.61
29.60
29.54
29.44
29.32
29.10
28.87
28.33
22.67
14.10
13.78
S
S
O
3ca
13C NMR, 100MHz
CDCl3
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)

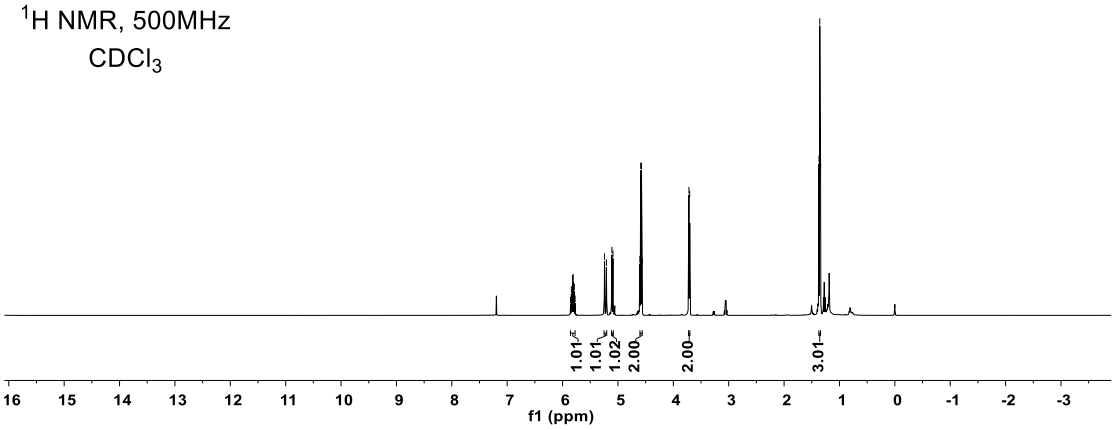


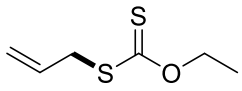


3da

^{1}H NMR, 500MHz

$CDCl_3$

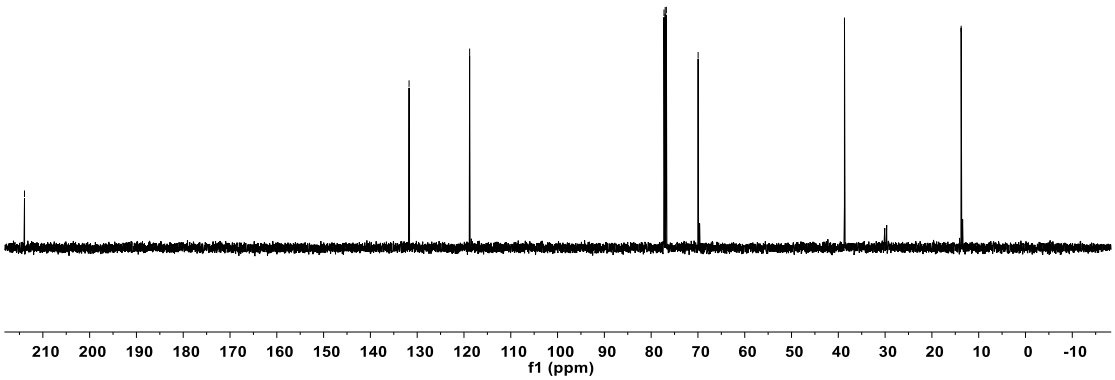


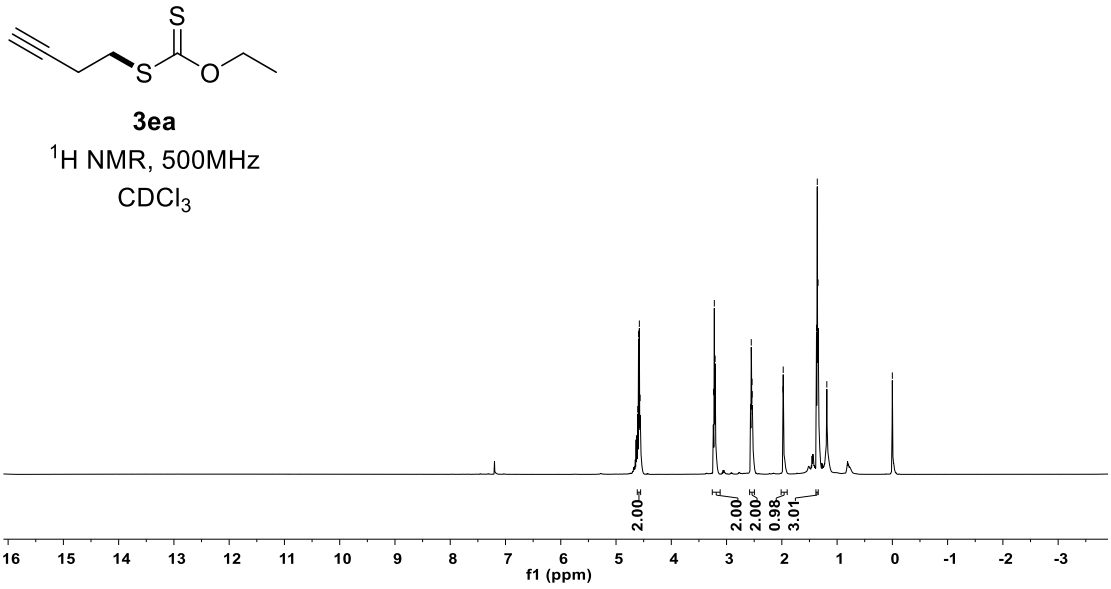


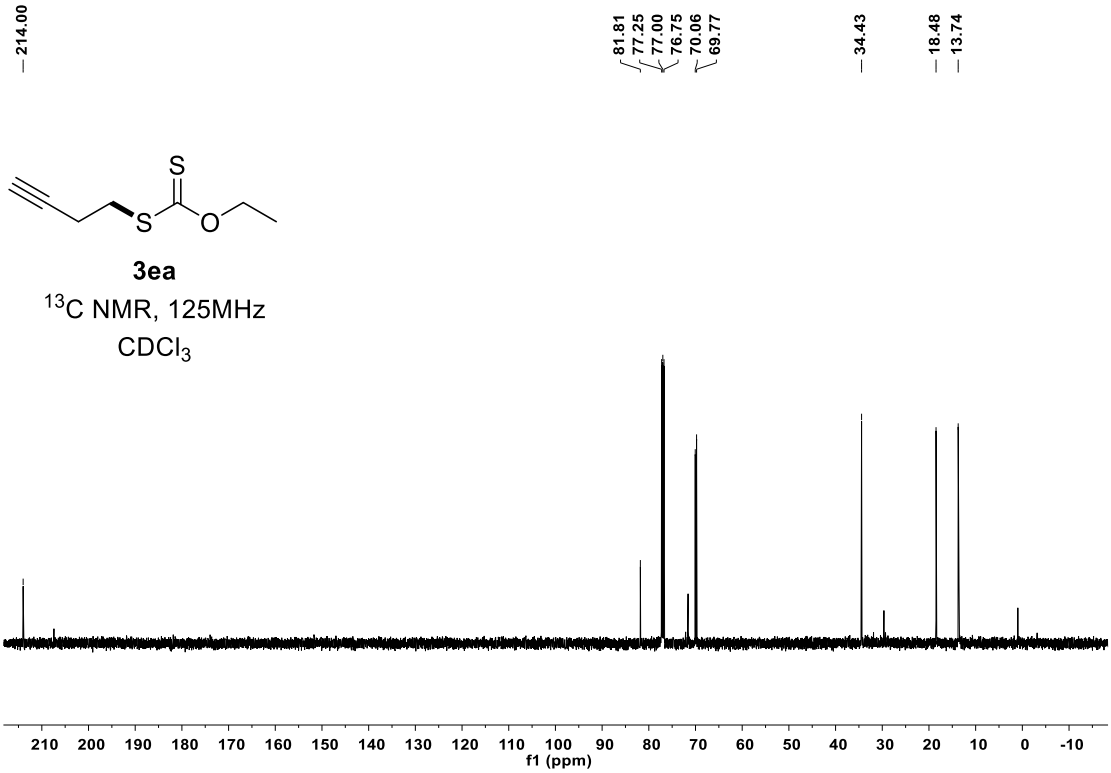
3da

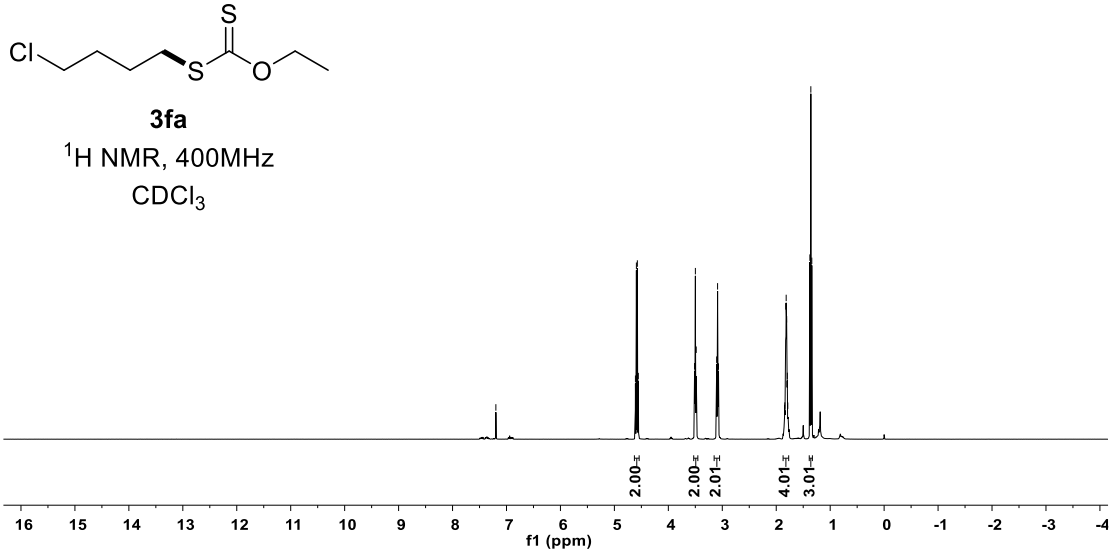
^{13}C NMR, 125MHz

$CDCl_3$




4.607
4.592
4.578
4.564
3.236
3.221
3.207
2.567
2.563
2.553
2.549
2.543
2.539
2.535
1.979
1.975
1.373
1.358
1.344
1.186
0.000
S
S
O
3ea
1H NMR, 500MHz
CDCl3
2.00
2.00
2.00
0.98
3.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

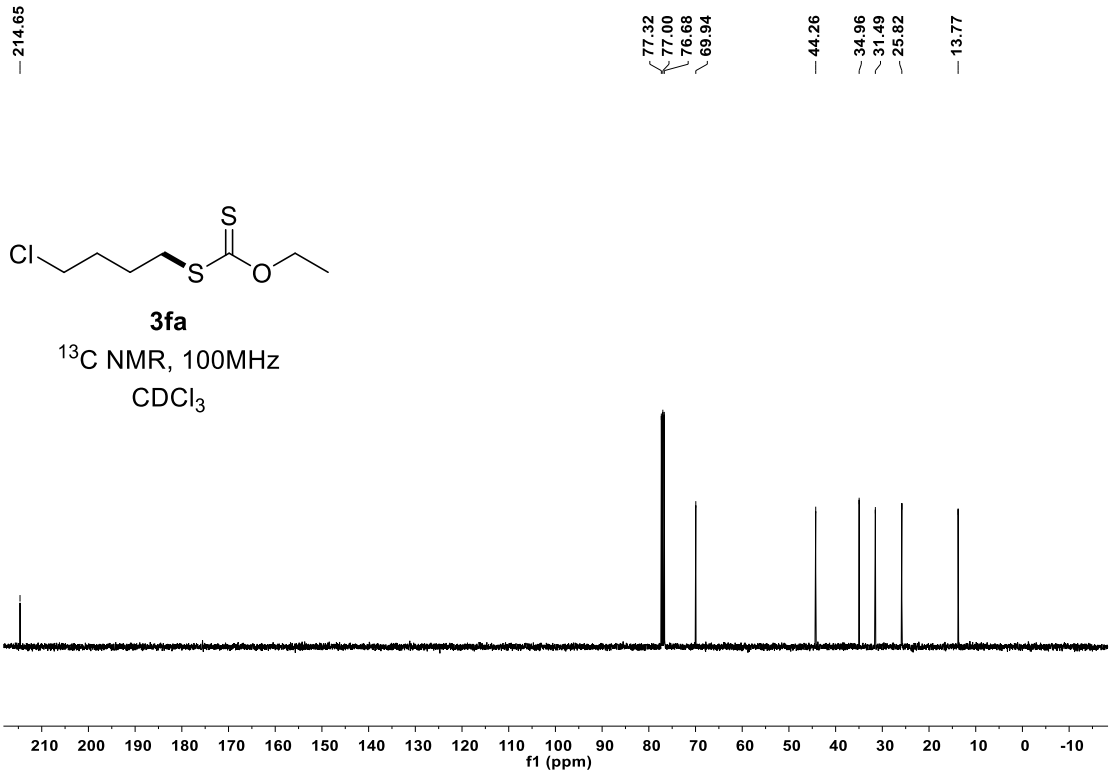

214.00
81.81
77.25
77.00
76.75
70.06
69.77
34.43
18.48
13.74
S
S
O
3ea
13C NMR, 125MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3fa

^{1}H NMR, 400MHz

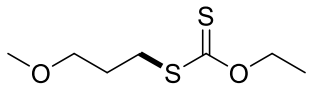
$CDCl_3$



3fa

^{13}C NMR, 100MHz

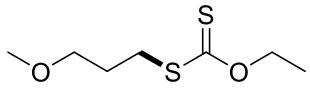
$CDCl_3$



3ga

^{1}H NMR, 400MHz

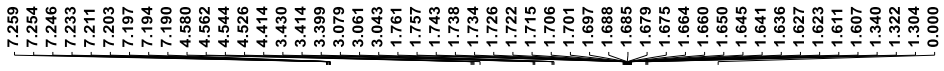
$CDCl_3$

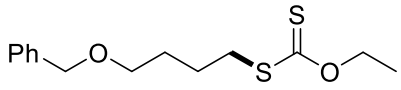


3ga

^{13}C NMR, 100MHz

$CDCl_3$

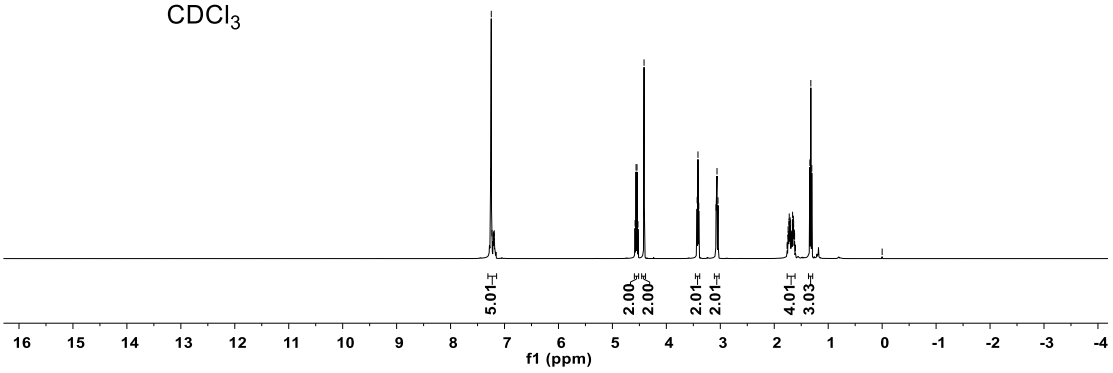


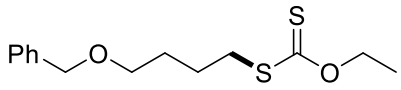


3ha

^{1}H NMR, 400MHz

$CDCl_3$

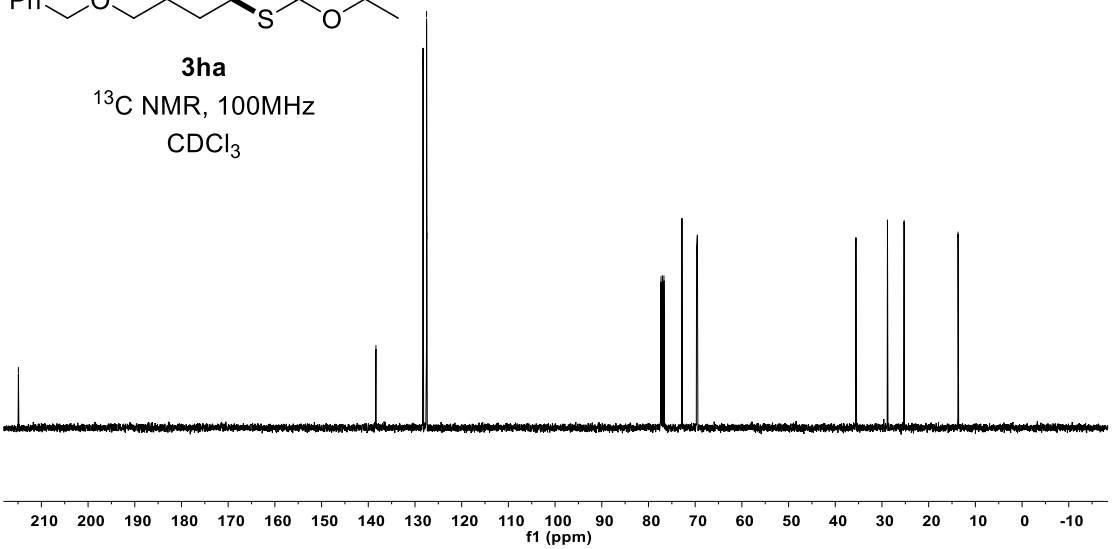


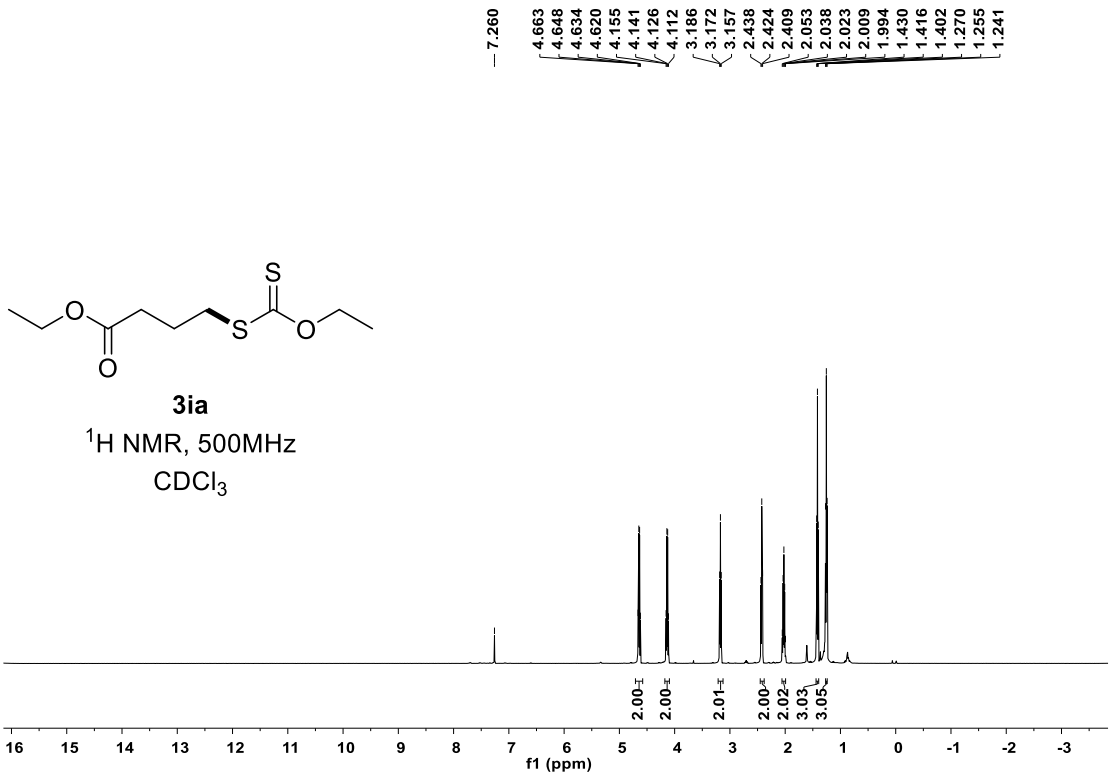


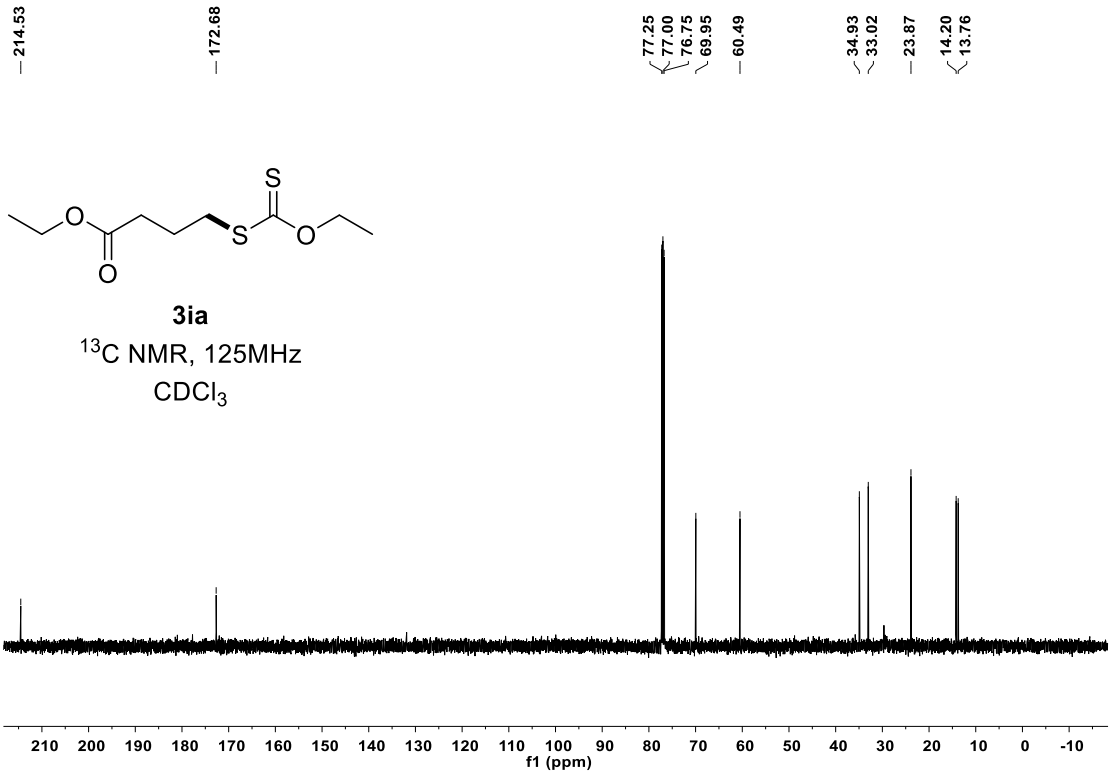
3ha

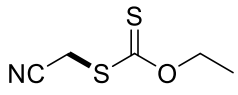
^{13}C NMR, 100MHz

$CDCl_3$




7.260
4.663
4.648
4.634
4.620
4.155
4.141
4.126
4.112
3.186
3.172
3.157
2.438
2.424
2.409
2.053
2.038
2.023
2.009
1.994
1.430
1.416
1.402
1.270
1.255
1.241
S
O
S
O
O
3ia
1H NMR, 500MHz
CDCl3
2.00
2.00
2.01
2.00
2.02
3.03
3.05
f1 (ppm)

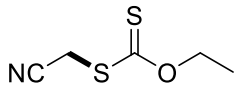

214.53
172.68
77.25
77.00
76.75
69.95
60.49
34.93
33.02
23.87
14.20
13.76
S
O
S
O
O
3ia
13C NMR, 125MHz
CDCl3
f1 (ppm)



3ja

^{1}H NMR, 500MHz

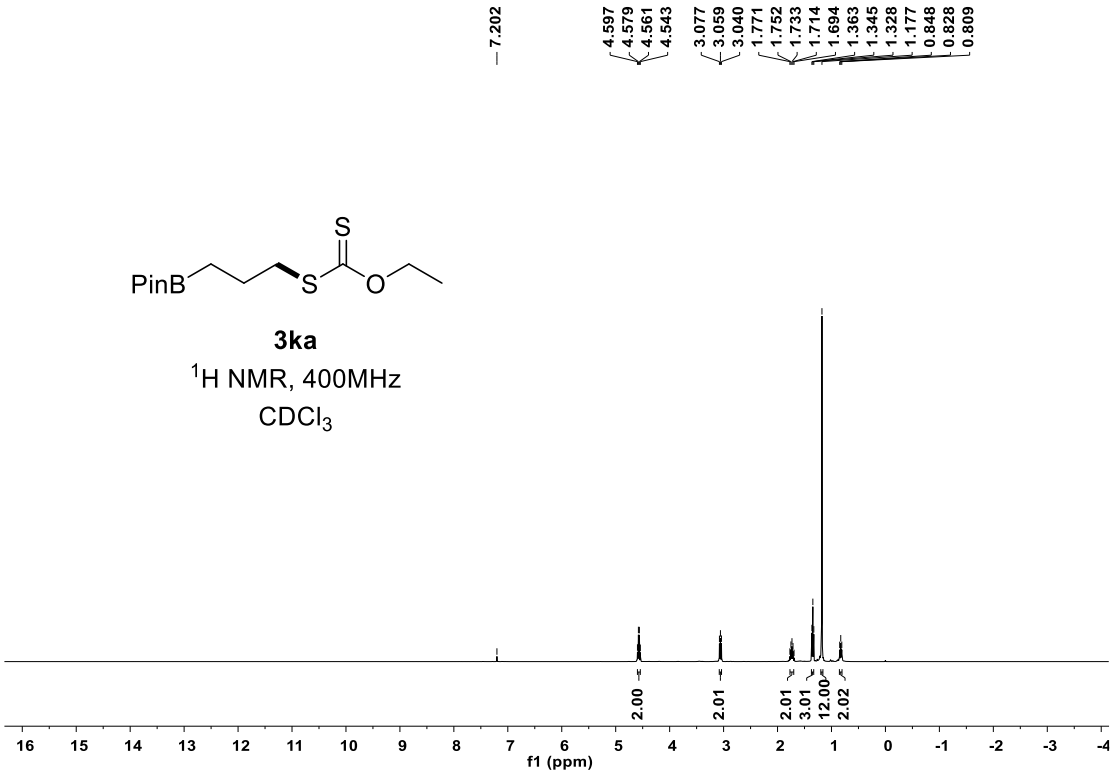
$CDCl_3$



3ja

^{13}C NMR, 125MHz

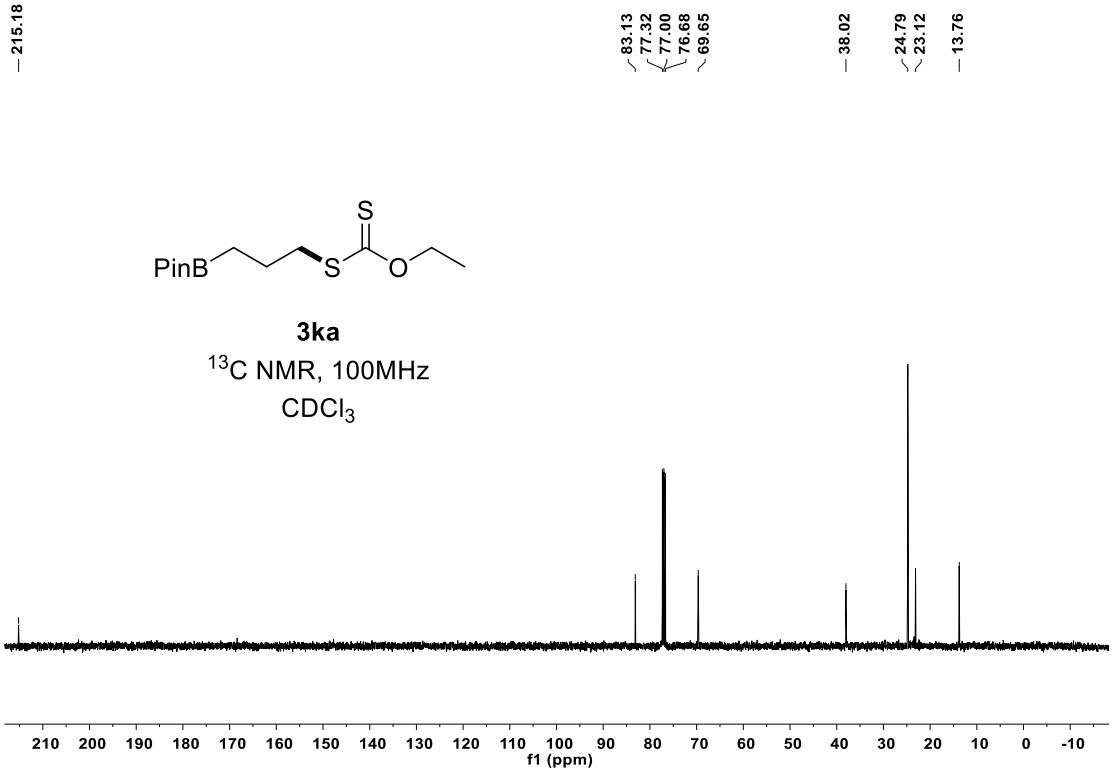
$CDCl_3$



3ka

^{1}H NMR, 400MHz

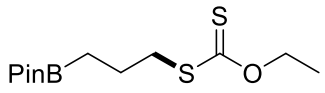
$CDCl_3$



3ka

^{13}C NMR, 100MHz

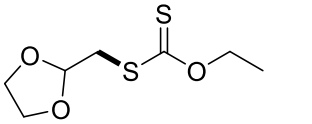
$CDCl_3$



3ka

^{11}B NMR, 160MHz

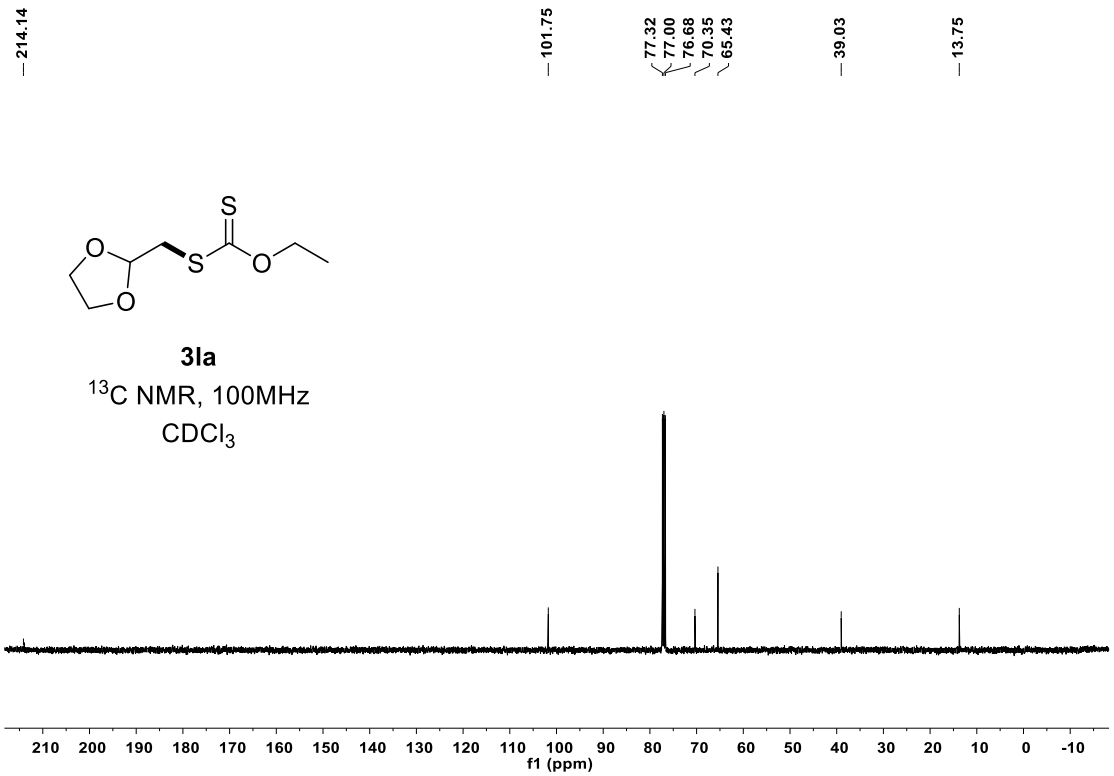
$CDCl_3$



3la

^{1}H NMR, 400MHz

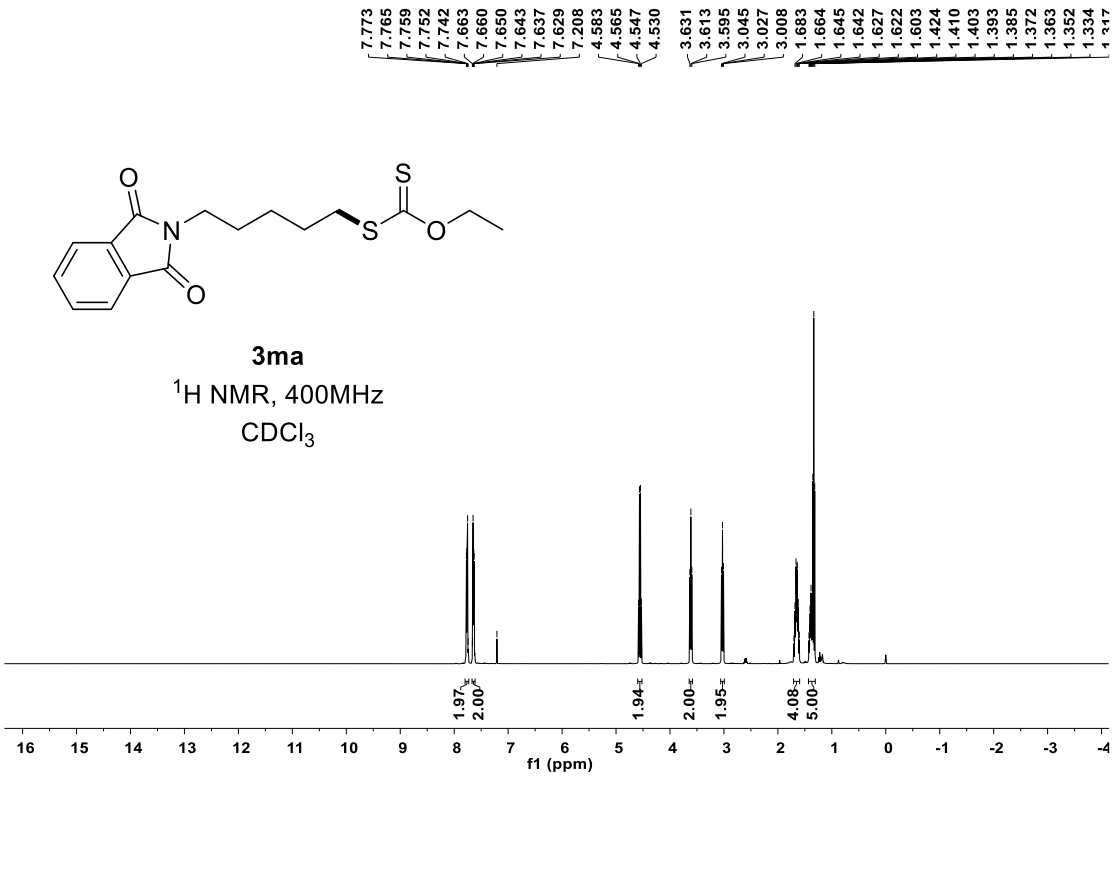
$CDCl_3$



3la

^{13}C NMR, 100MHz

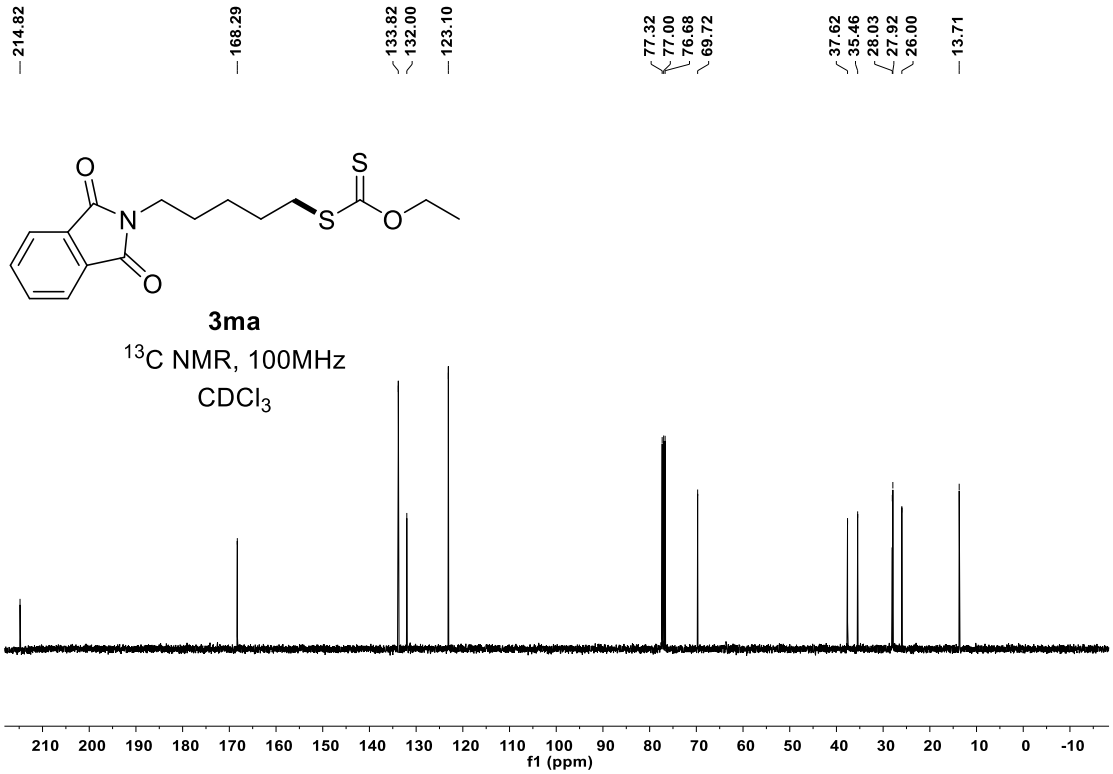
$CDCl_3$

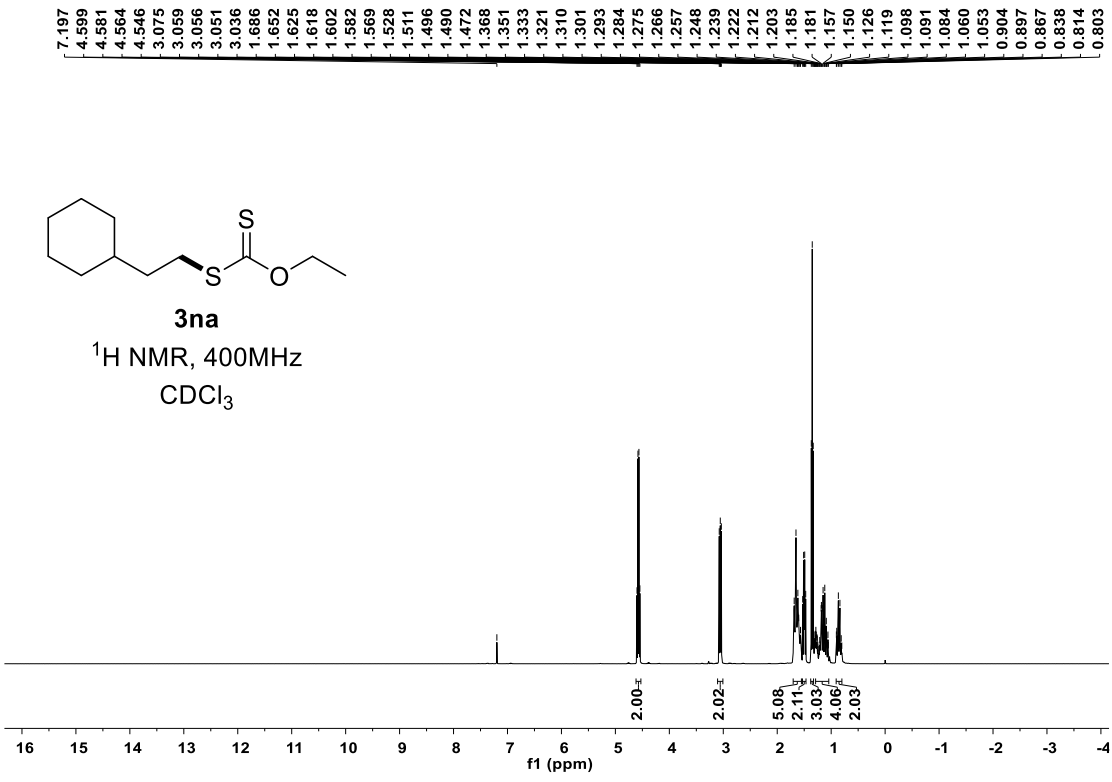


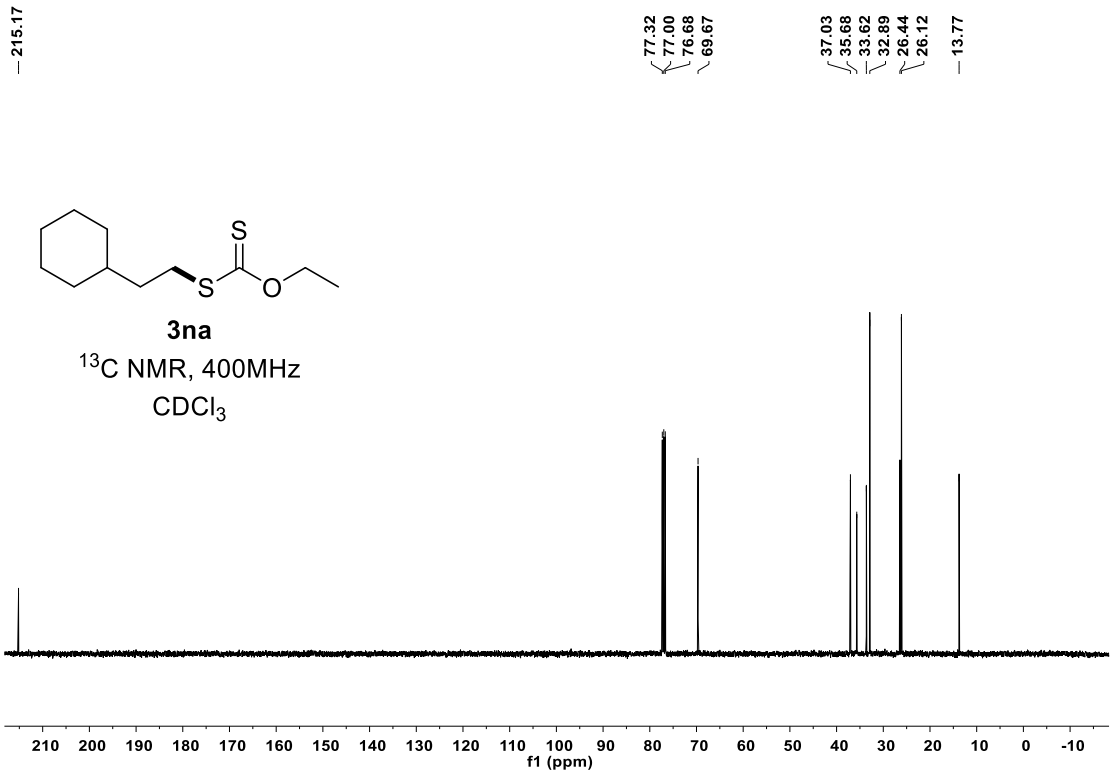
3ma

^{1}H NMR, 400MHz

$CDCl_3$


214.82
168.29
133.82
132.00
123.10
77.32
77.00
76.68
69.72
37.62
35.46
28.03
27.92
26.00
13.71
O
N
O
S
S
O
3ma
13C NMR, 100MHz
CDCl3
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)

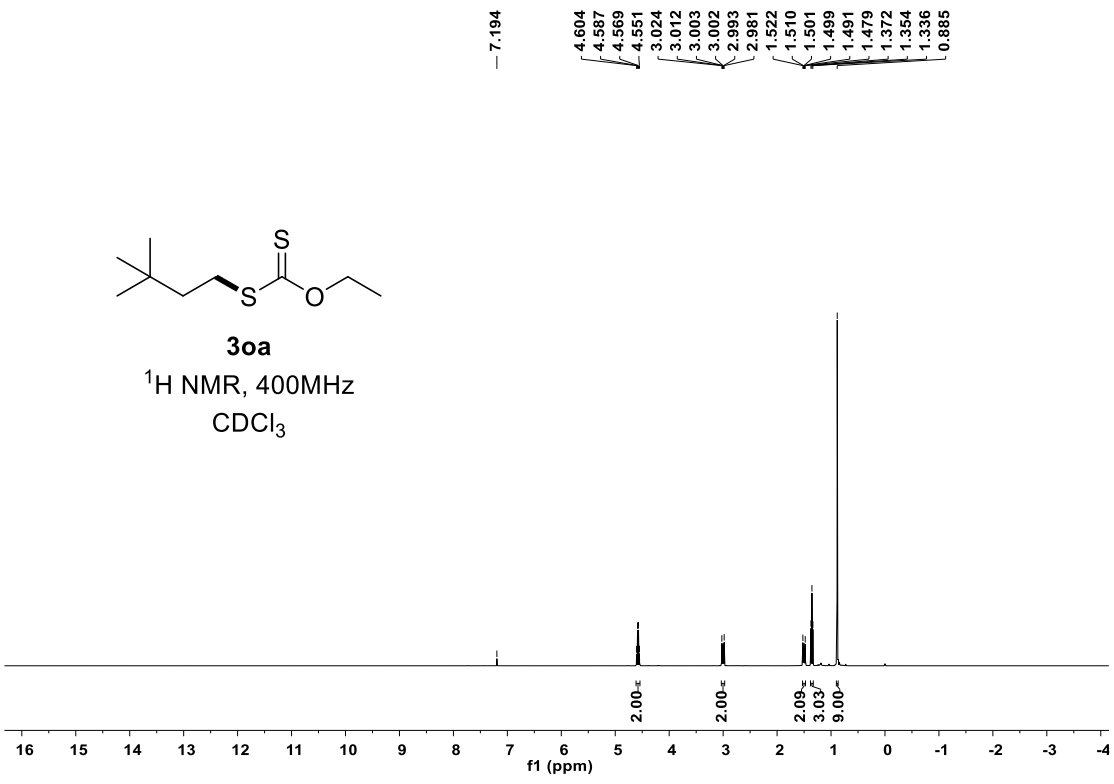

7.197
4.599
4.581
4.564
4.546
3.075
3.059
3.056
3.051
3.036
1.686
1.652
1.625
1.618
1.602
1.582
1.569
1.528
1.511
1.496
1.490
1.472
1.368
1.351
1.333
1.321
1.310
1.301
1.293
1.284
1.275
1.266
1.257
1.248
1.239
1.222
1.212
1.203
1.185
1.181
1.157
1.150
1.126
1.119
1.098
1.091
1.084
1.060
1.053
0.904
0.897
0.867
0.838
0.814
0.803
S
S
O
3na
1H NMR, 400MHz
CDCl3
2.00
2.02
5.08
2.11
3.03
4.06
2.03
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)



3na

^{13}C NMR, 400MHz

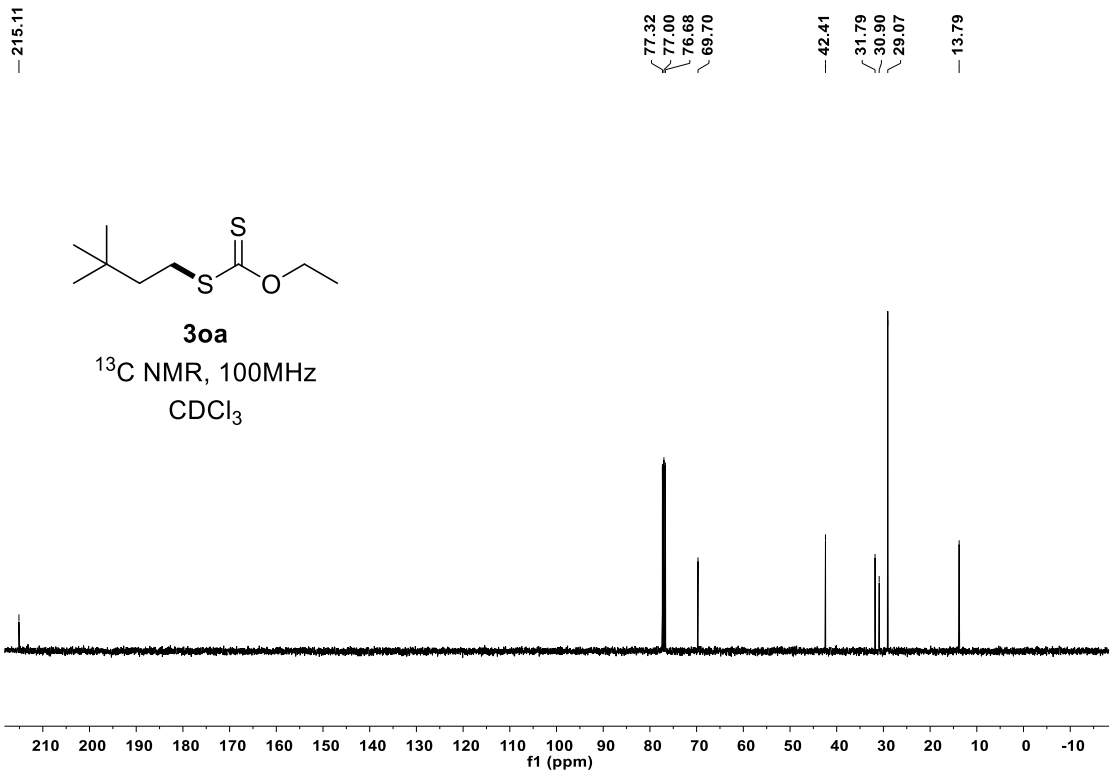
$CDCl_3$

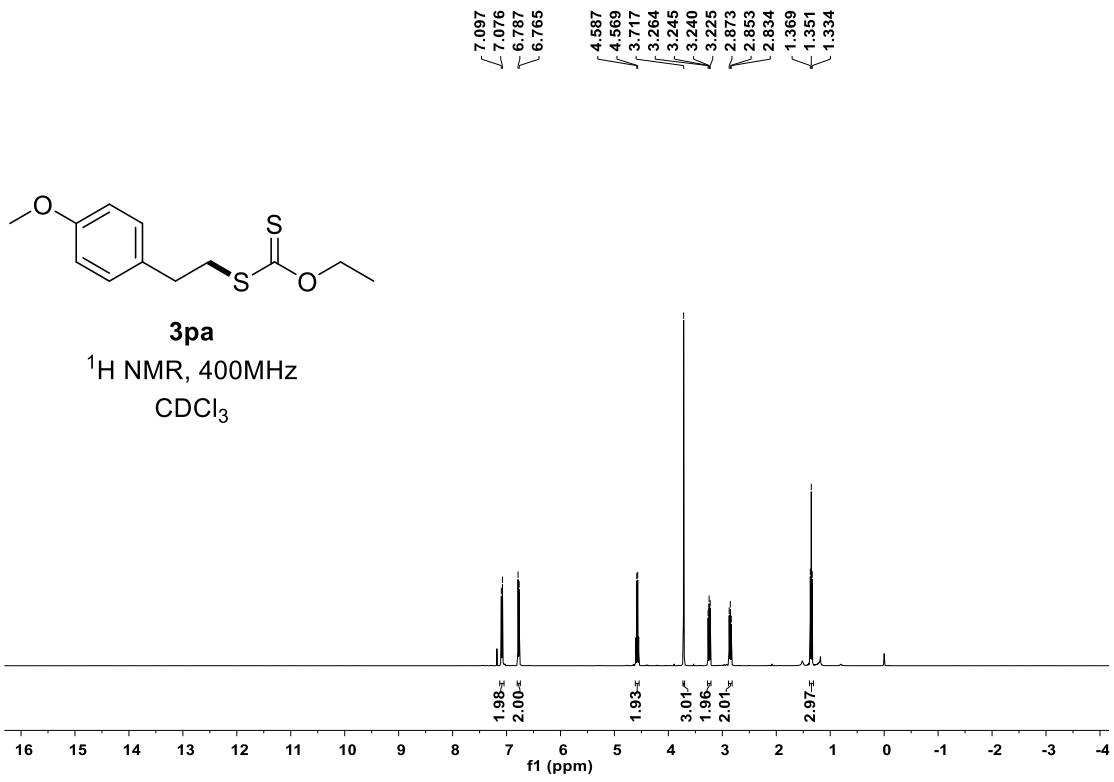


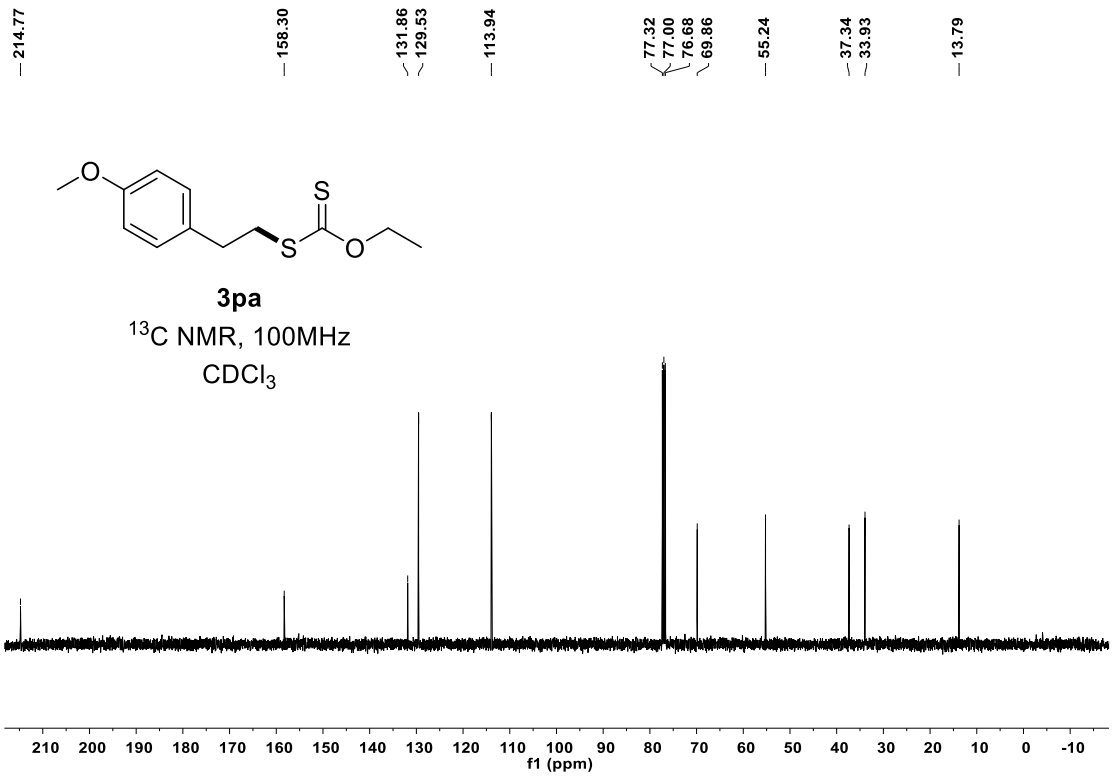
3oa

^{1}H NMR, 400MHz

$CDCl_3$


215.11
77.32
77.00
76.68
69.70
42.41
31.79
30.90
29.07
13.79
S
S
O
3oa
13C NMR, 100MHz
CDCl3
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)

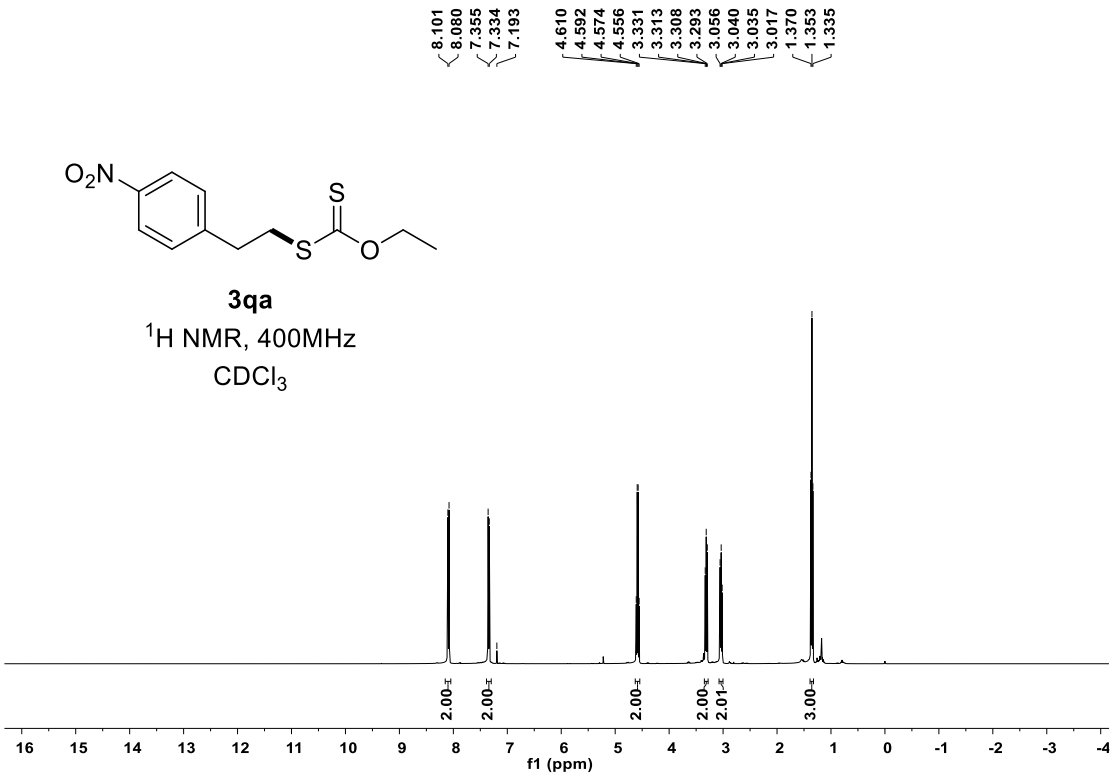

7.097
7.076
6.787
6.765
4.587
4.569
3.717
3.264
3.245
3.240
3.225
2.873
2.853
2.834
1.369
1.351
1.334
O
S
S
O
3pa
1H NMR, 400MHz
CDCl3
1.98
2.00
1.93
3.01
1.96
2.01
2.97
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)



3pa

^{13}C NMR, 100MHz

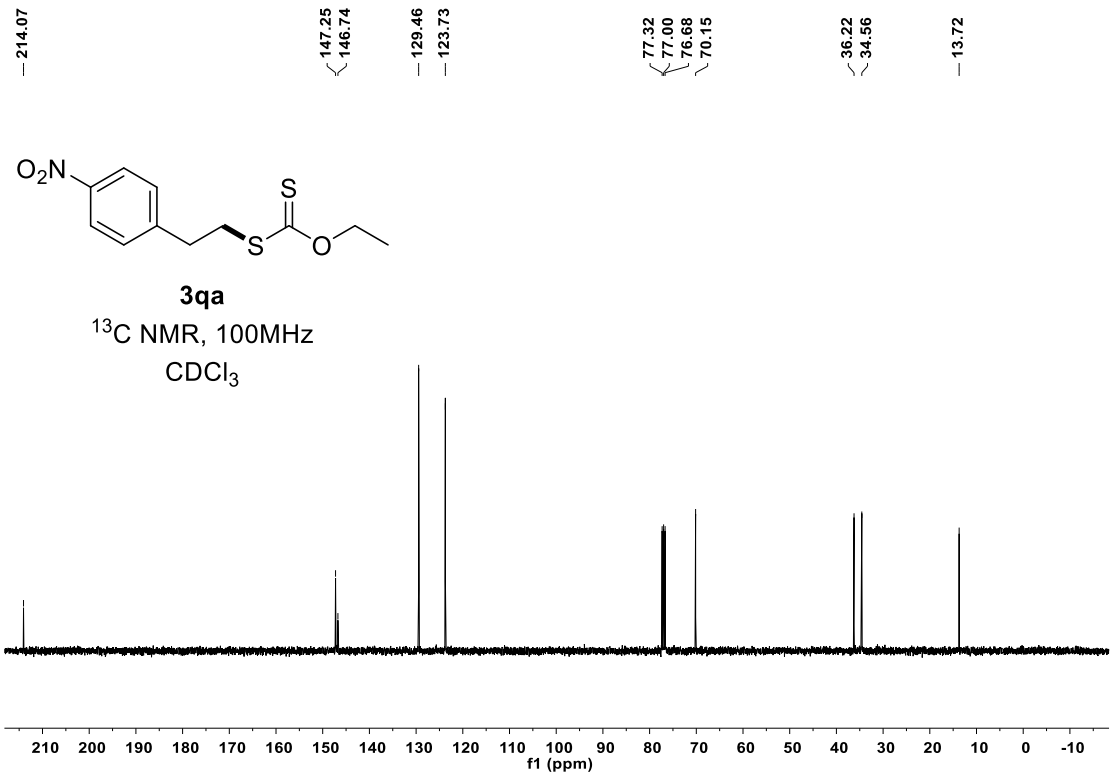
$CDCl_3$

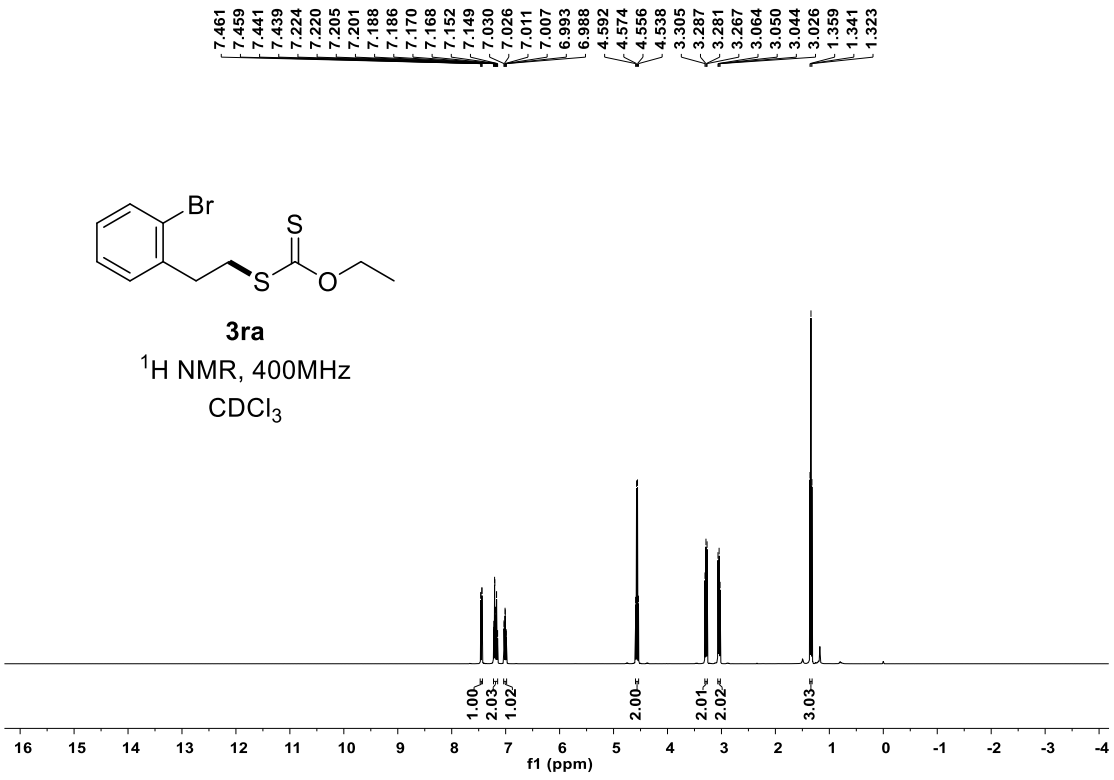


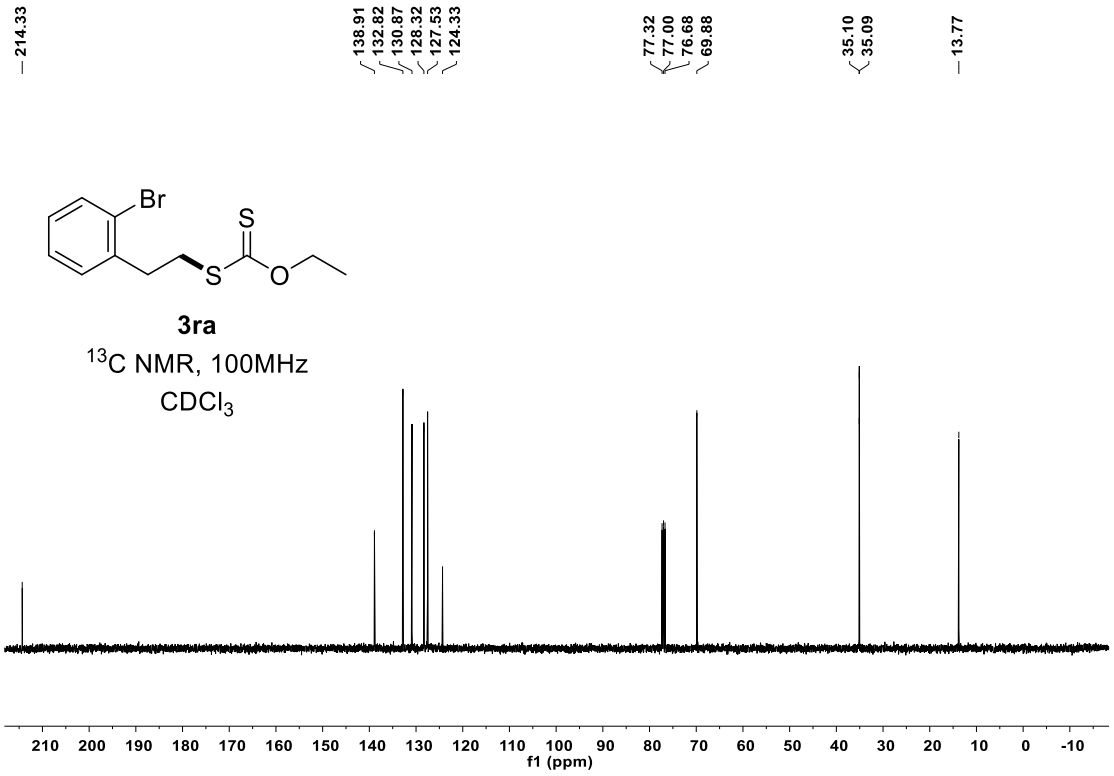
3qa

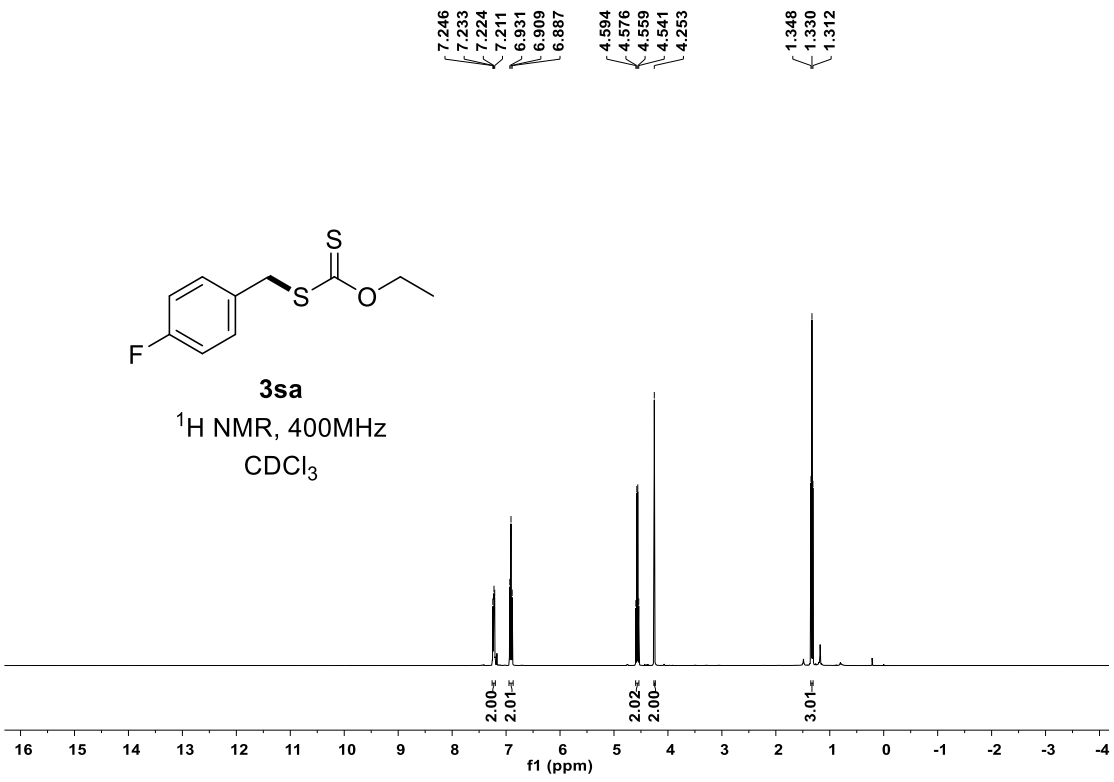
^{1}H NMR, 400MHz

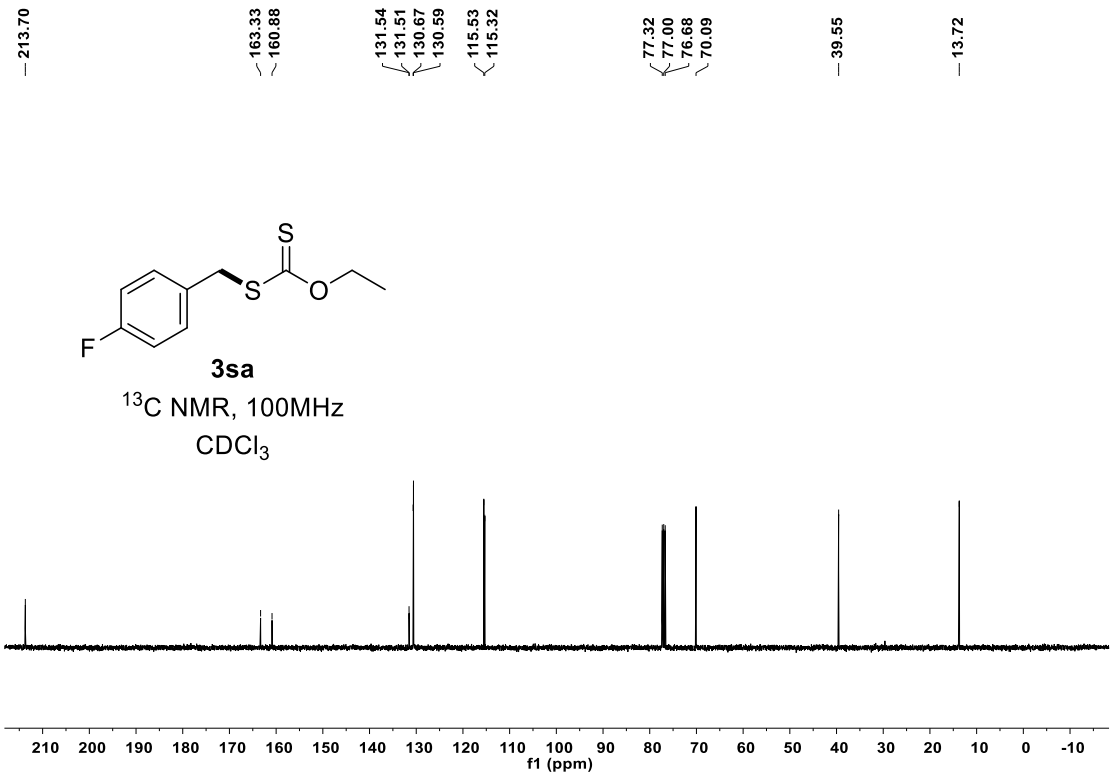
$CDCl_3$


214.07
147.25
146.74
129.46
123.73
77.32
77.00
76.68
70.15
36.22
34.56
13.72
O2N
S
S
O
3qa
13C NMR, 100MHz
CDCl3
f1 (ppm)

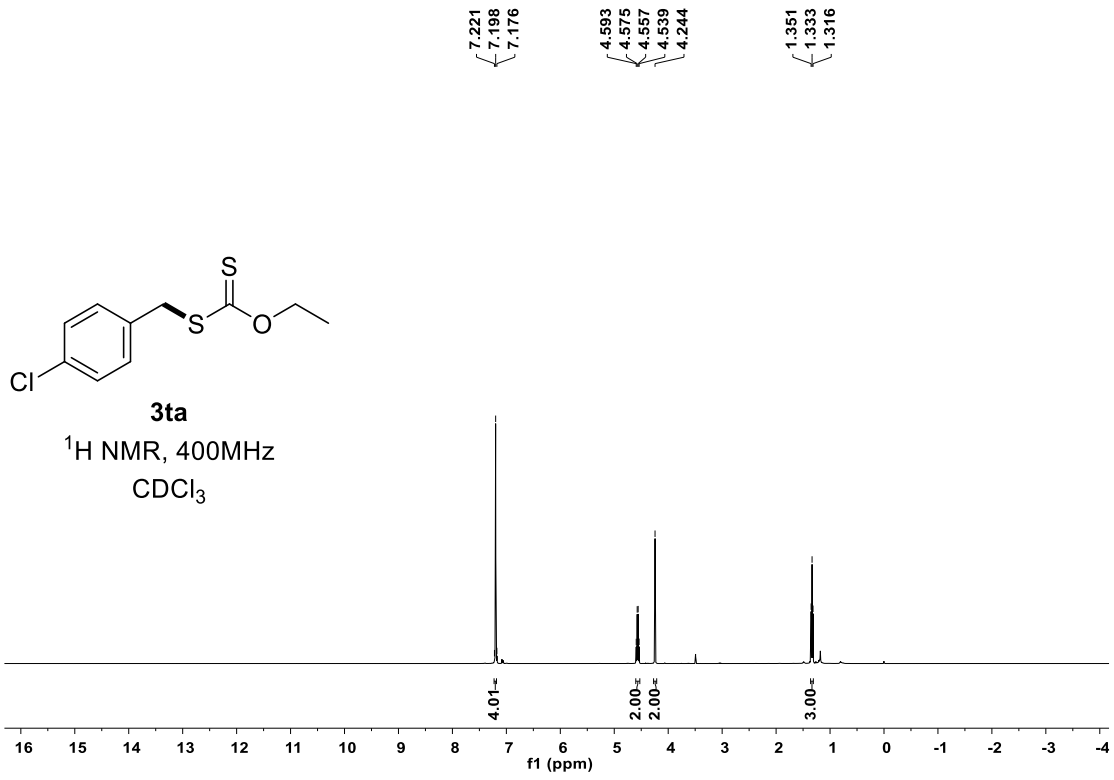

7.461
7.459
7.441
7.439
7.224
7.220
7.205
7.201
7.188
7.186
7.170
7.168
7.152
7.149
7.030
7.026
7.011
7.007
6.993
6.988
4.592
4.574
4.556
4.538
3.305
3.287
3.281
3.267
3.064
3.050
3.044
3.026
1.359
1.341
1.323
Br
S
S
O
3ra
1H NMR, 400MHz
CDCl3
1.00
2.03
1.02
2.00
2.01
2.02
3.03
f1 (ppm)

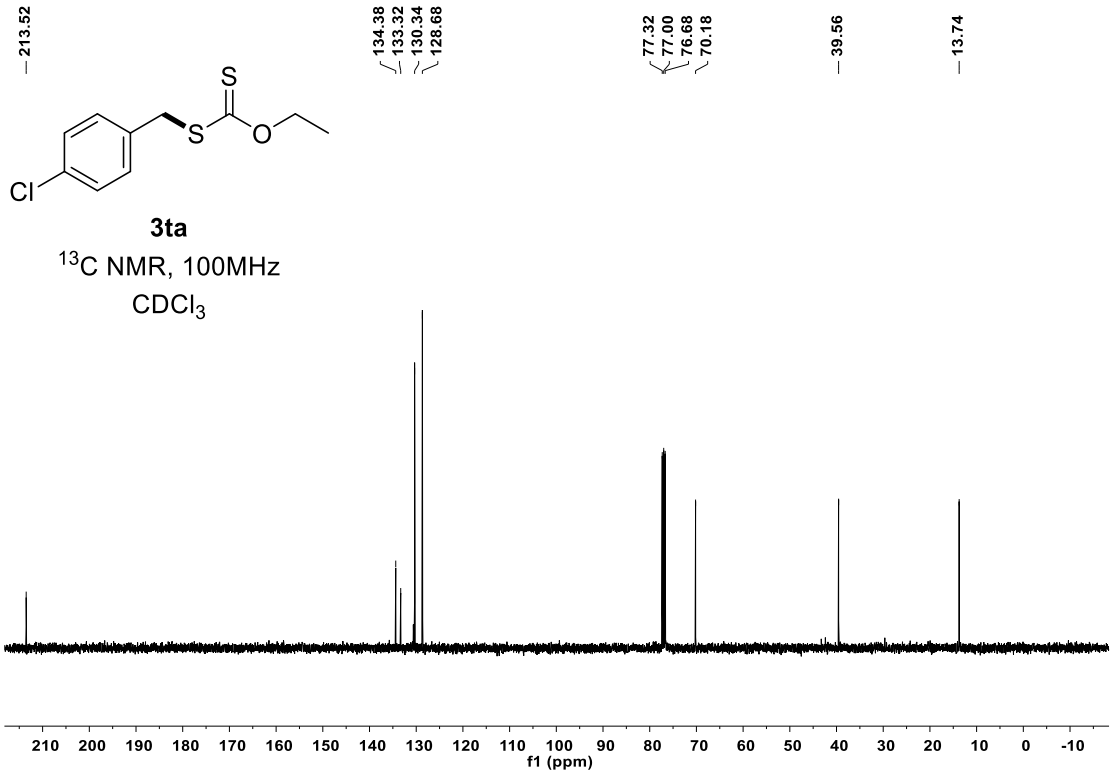

214.33
138.91
132.82
130.87
128.32
127.53
124.33
77.32
77.00
76.68
69.88
35.10
35.09
13.77
Br
S
S
O
3ra
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

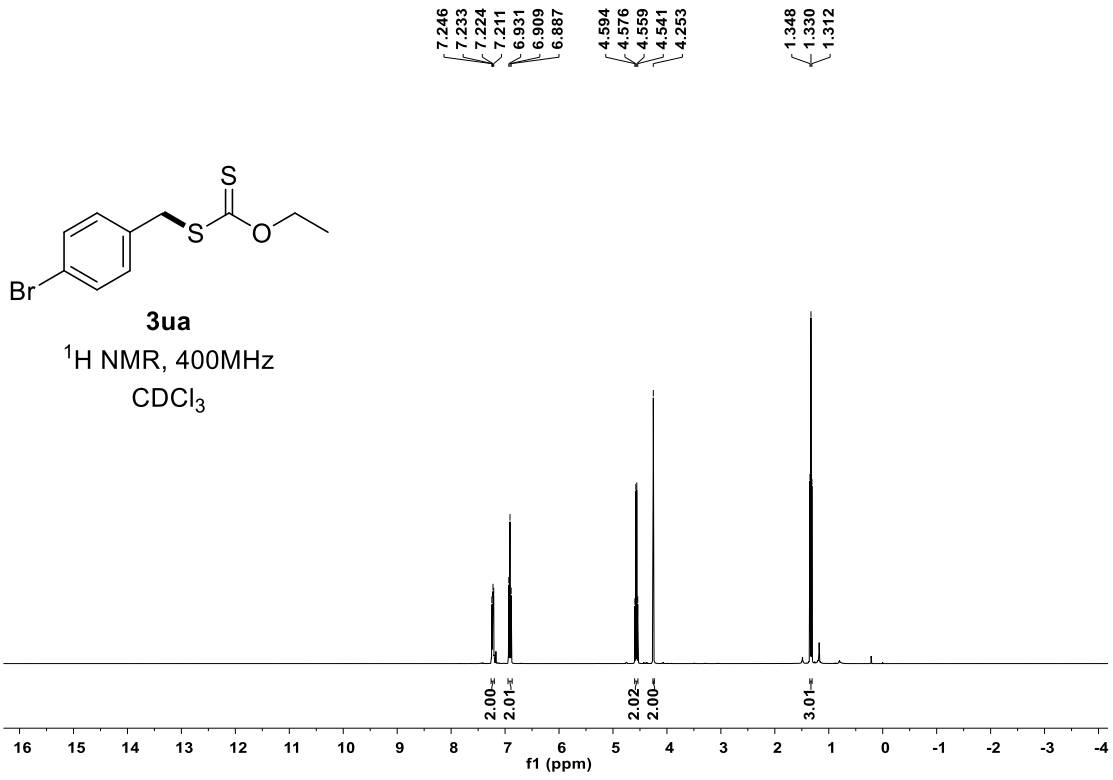

7.246
7.233
7.224
7.211
6.931
6.909
6.887
4.594
4.576
4.559
4.541
4.253
1.348
1.330
1.312
S
S
O
F
3sa
1H NMR, 400MHz
CDCl3
2.00
2.01
2.02
2.00
3.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


213.70
163.33
160.88
131.54
131.51
130.67
130.59
115.53
115.32
77.32
77.00
76.68
70.09
39.55
13.72
S
S
O
F
3sa
13C NMR, 100MHz
CDCl3
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)


-114.63
S
S
O
F
3sa
19F NMR, 471MHz
CDCl3
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


7.221
7.198
7.176
4.593
4.575
4.557
4.539
4.244
1.351
1.333
1.316
S
S
O
Cl
3ta
1H NMR, 400MHz
CDCl3
4.01
2.00
2.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

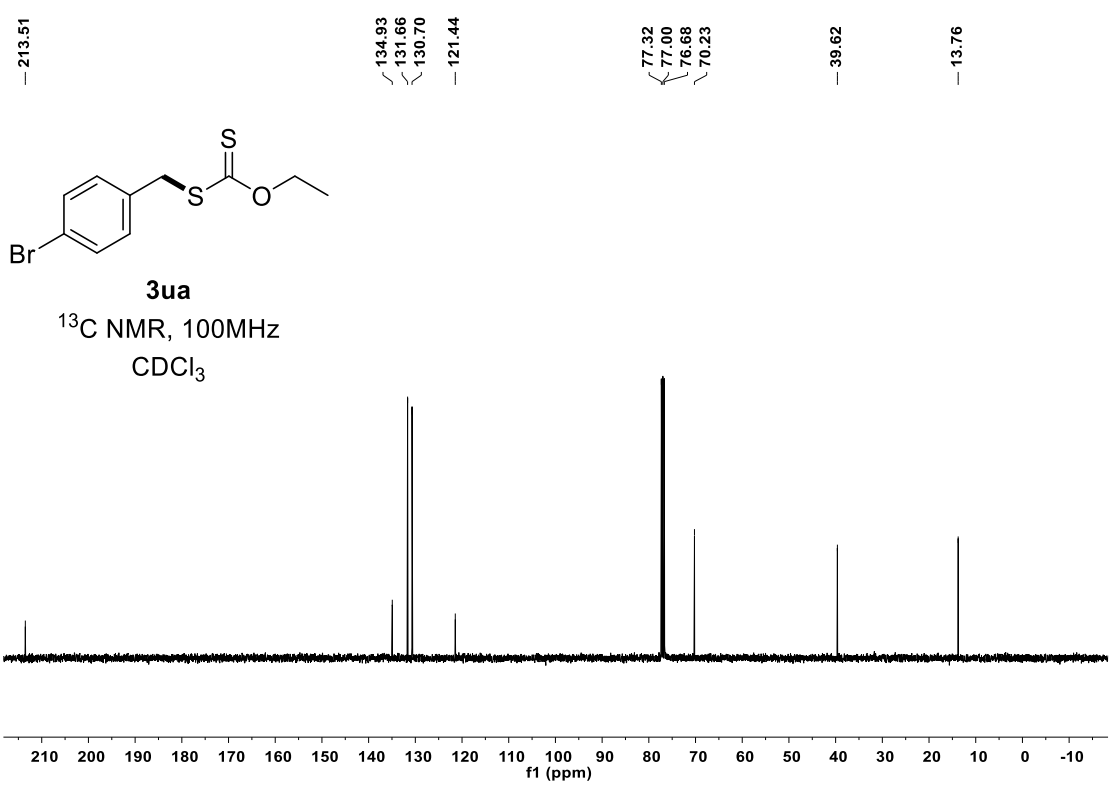

213.52
134.38
133.32
130.34
128.68
77.32
77.00
76.68
70.18
39.56
13.74
S
S
O
Cl
3ta
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3ua

^{1}H NMR, 400MHz

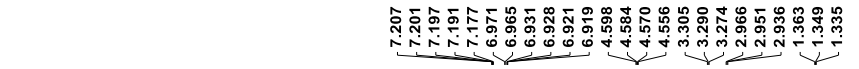
$CDCl_3$

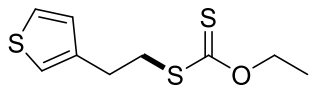


3ua

^{13}C NMR, 100MHz

$CDCl_3$

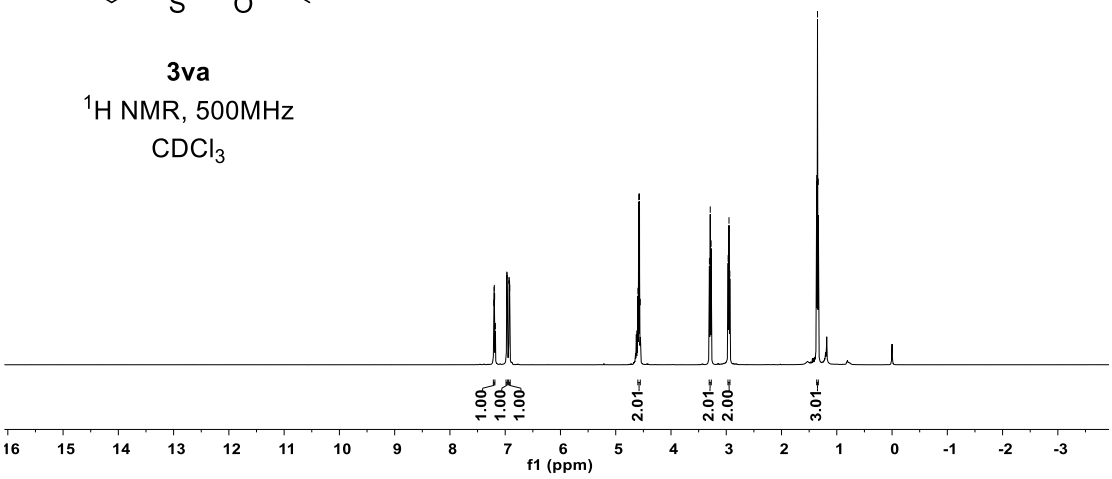


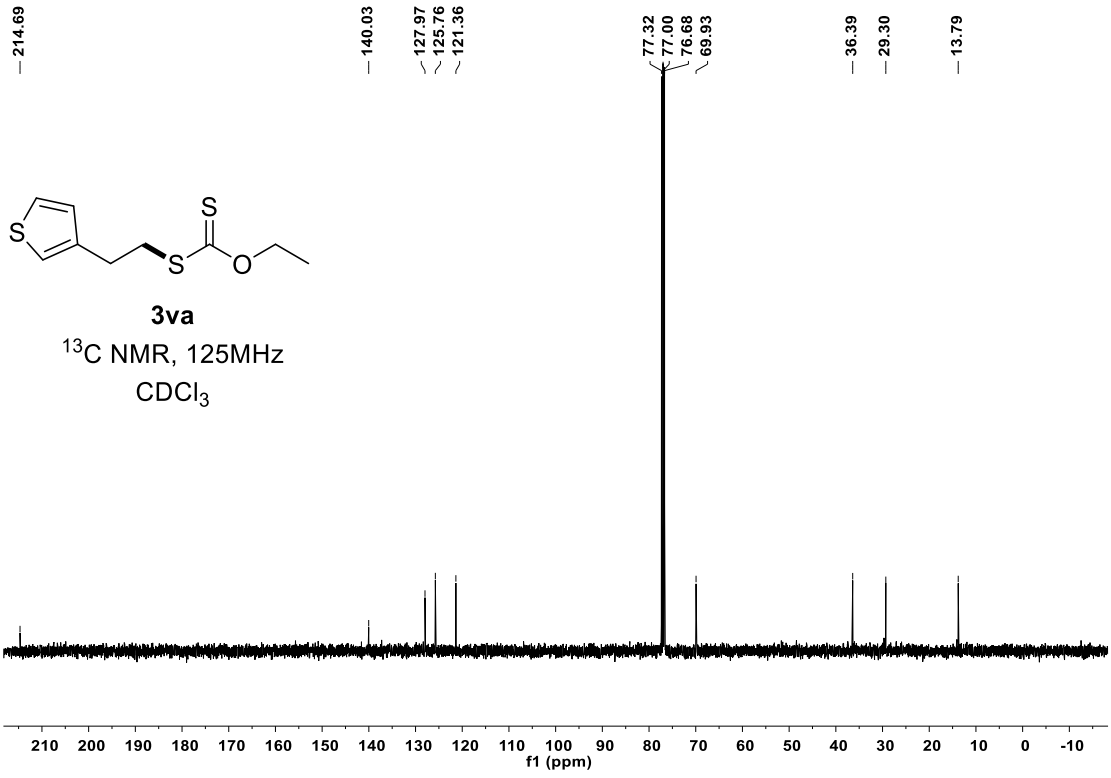


3va

^{1}H NMR, 500MHz

$CDCl_3$

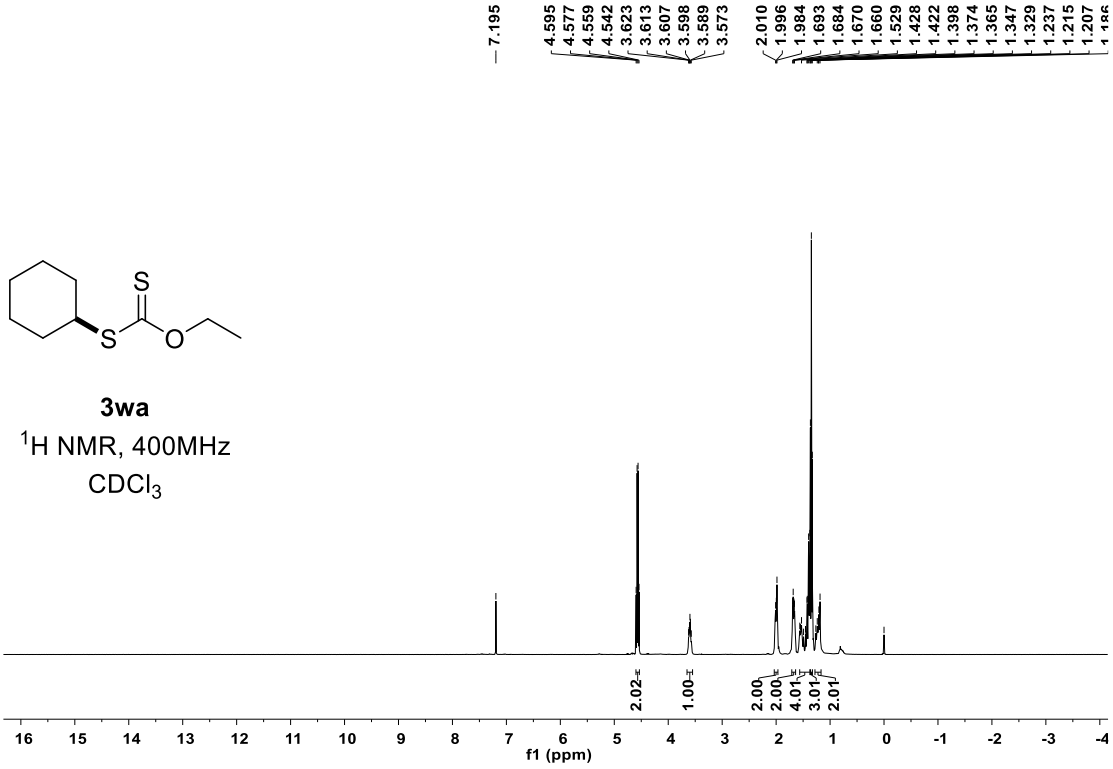


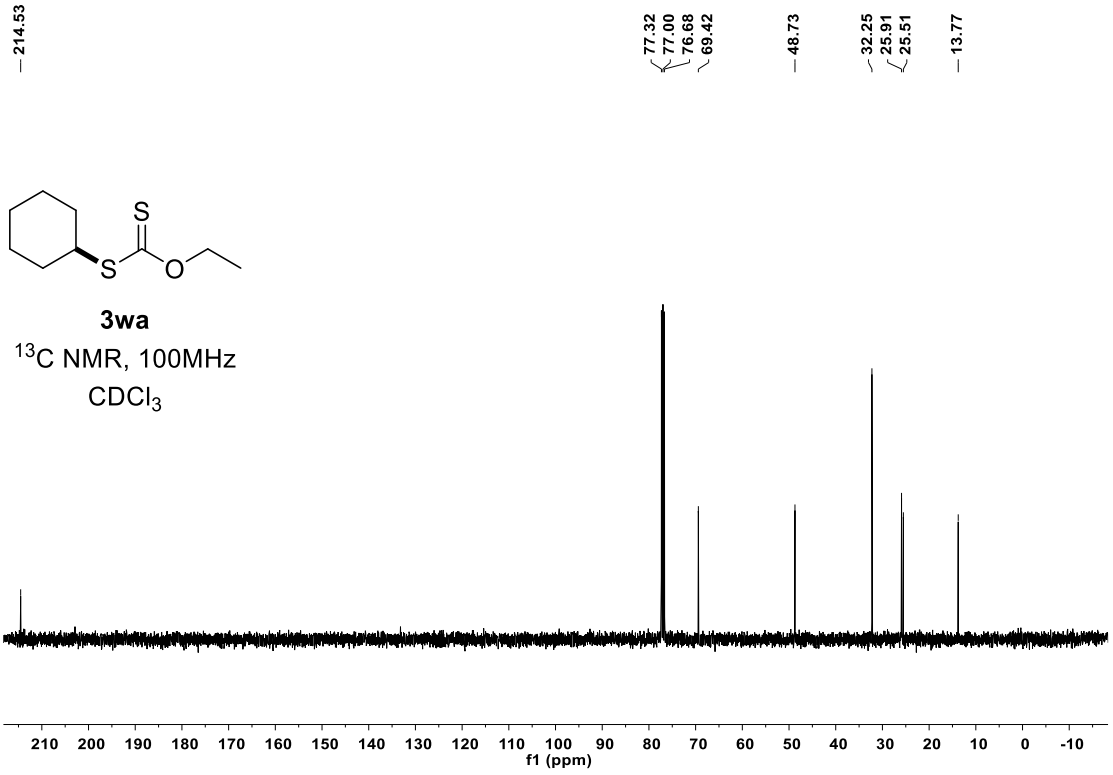


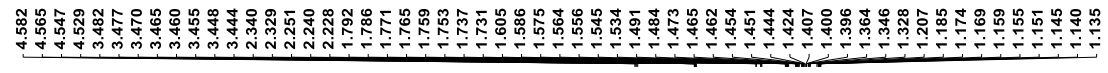
3va

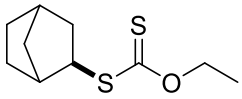
^{13}C NMR, 125MHz

$CDCl_3$


7.195
4.595
4.577
4.559
4.542
3.623
3.613
3.607
3.598
3.589
3.573
2.010
1.996
1.984
1.693
1.684
1.670
1.660
1.529
1.428
1.422
1.398
1.374
1.365
1.347
1.329
1.237
1.215
1.207
S
S
O
3wa
1H NMR, 400MHz
CDCl3
2.02
1.00
2.00
2.00
4.01
3.01
2.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


214.53
77.32
77.00
76.68
69.42
48.73
32.25
25.91
25.51
13.77
S
S
O
3wa
13C NMR, 100MHz
CDCl3
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)

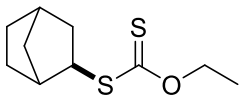




3xa

^{1}H NMR, 400MHz

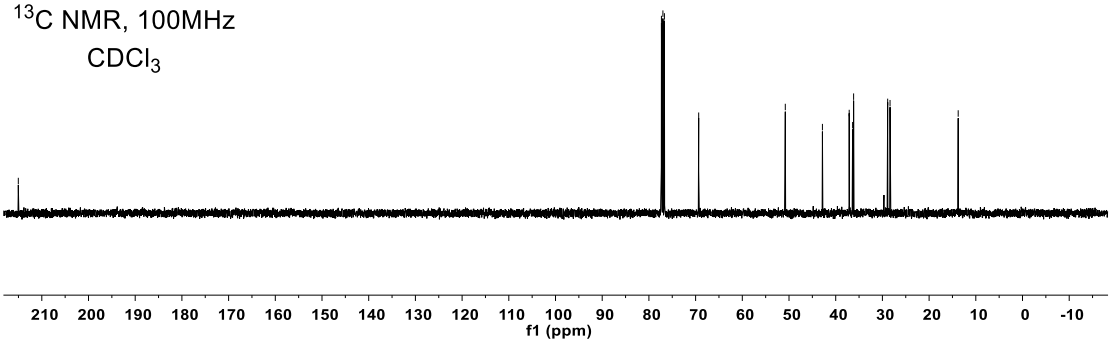
$CDCl_3$

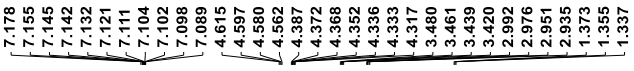


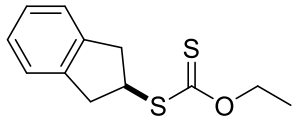
3xa

^{13}C NMR, 100MHz

$CDCl_3$



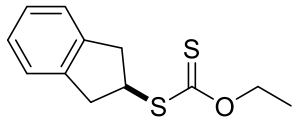




3ya

^{1}H NMR, 400MHz

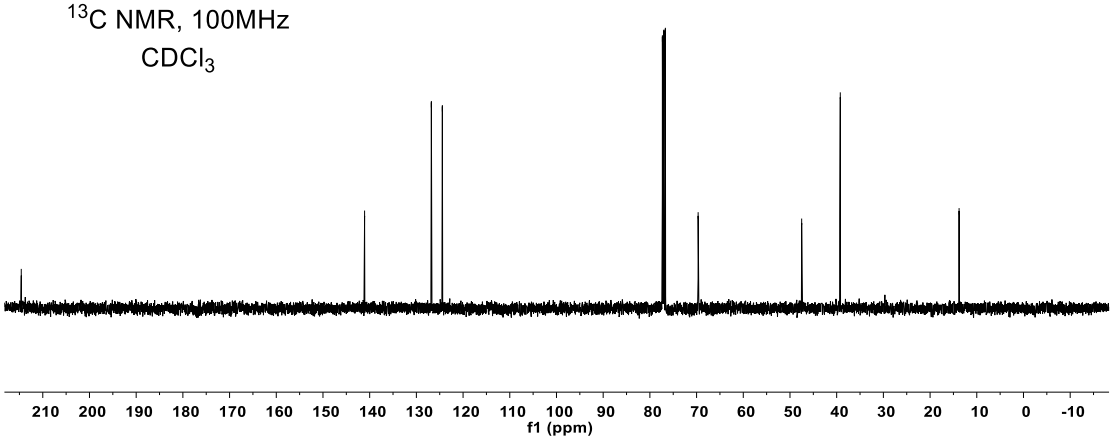
$CDCl_3$

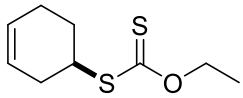


3ya

^{13}C NMR, 100MHz

$CDCl_3$

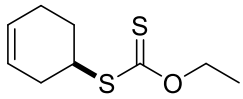




3za

^{1}H NMR, 400MHz

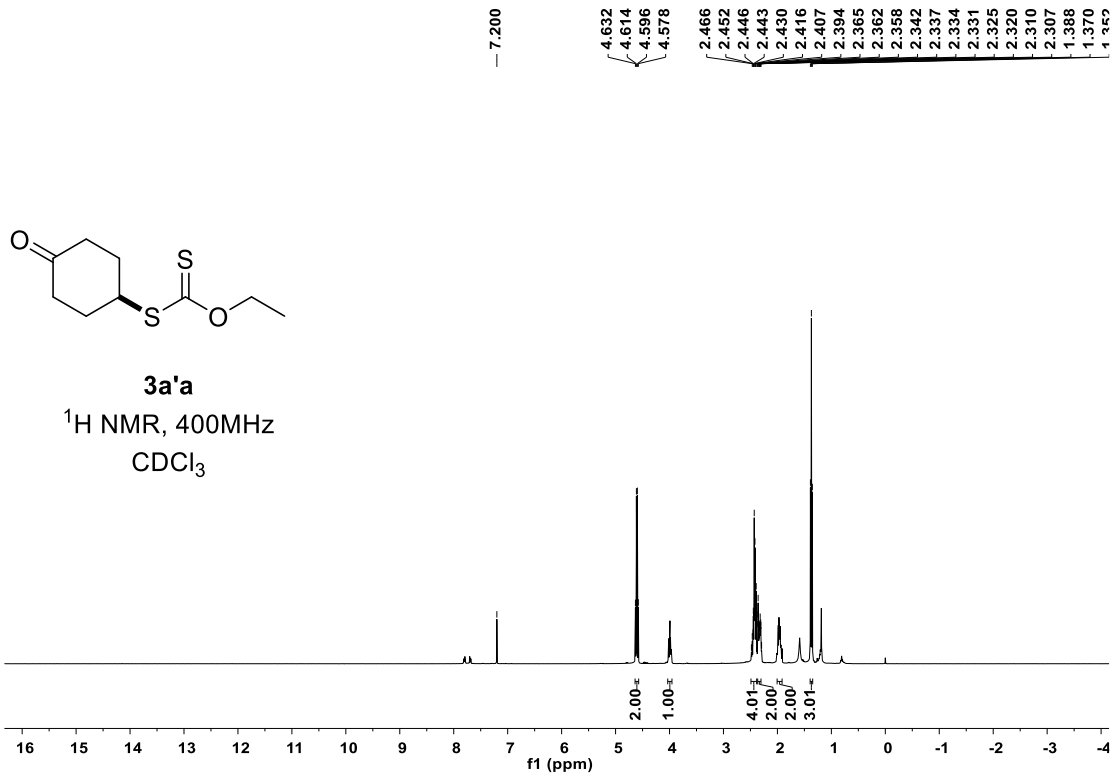
$CDCl_3$



3za

^{13}C NMR, 100MHz

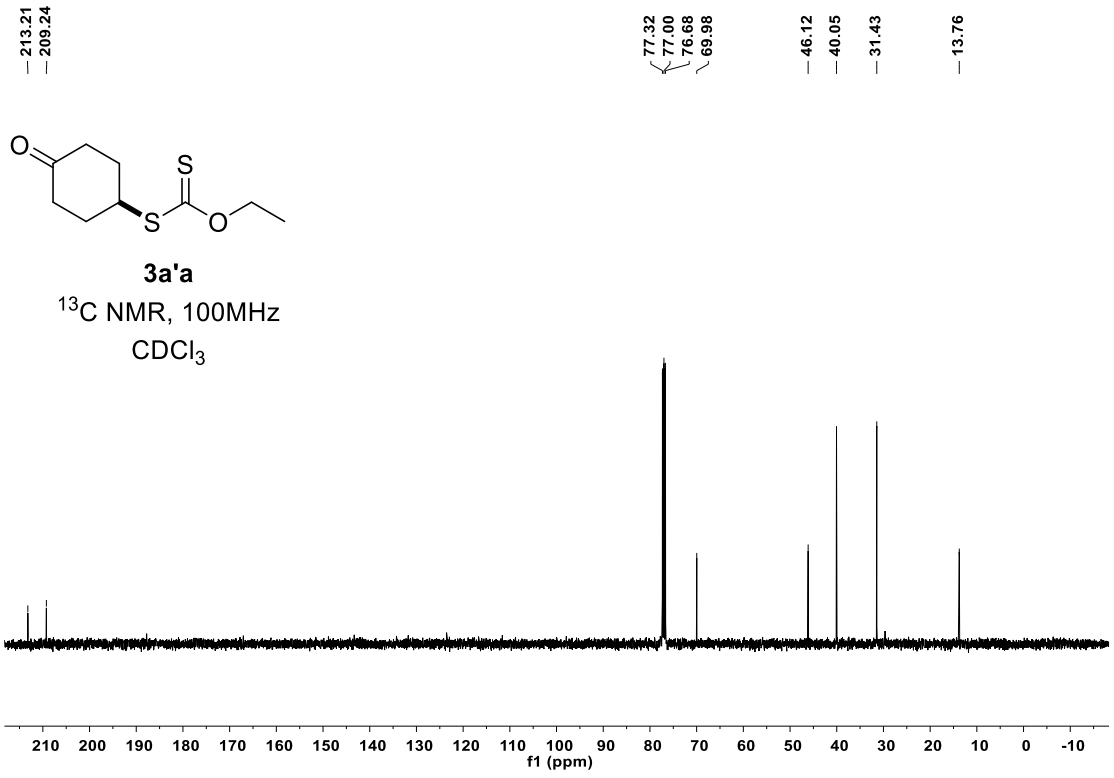
$CDCl_3$



3a'a

^{1}H NMR, 400MHz

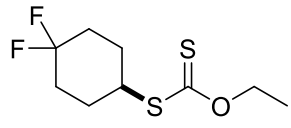
$CDCl_3$



3a'a

^{13}C NMR, 100MHz

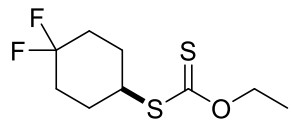
$CDCl_3$



3b'a

^{1}H NMR, 400MHz

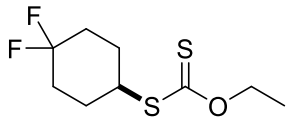
$CDCl_3$



3b'a

^{13}C NMR, 100MHz

$CDCl_3$



3b'a

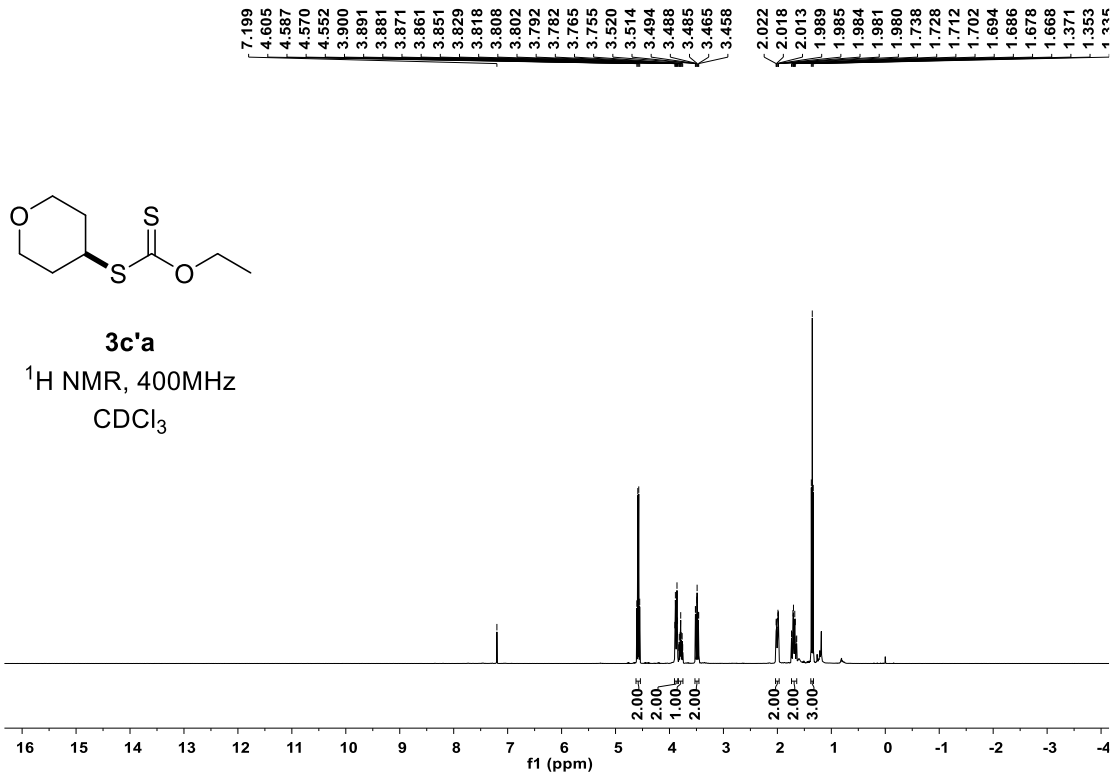
^{19}F NMR, 377MHz

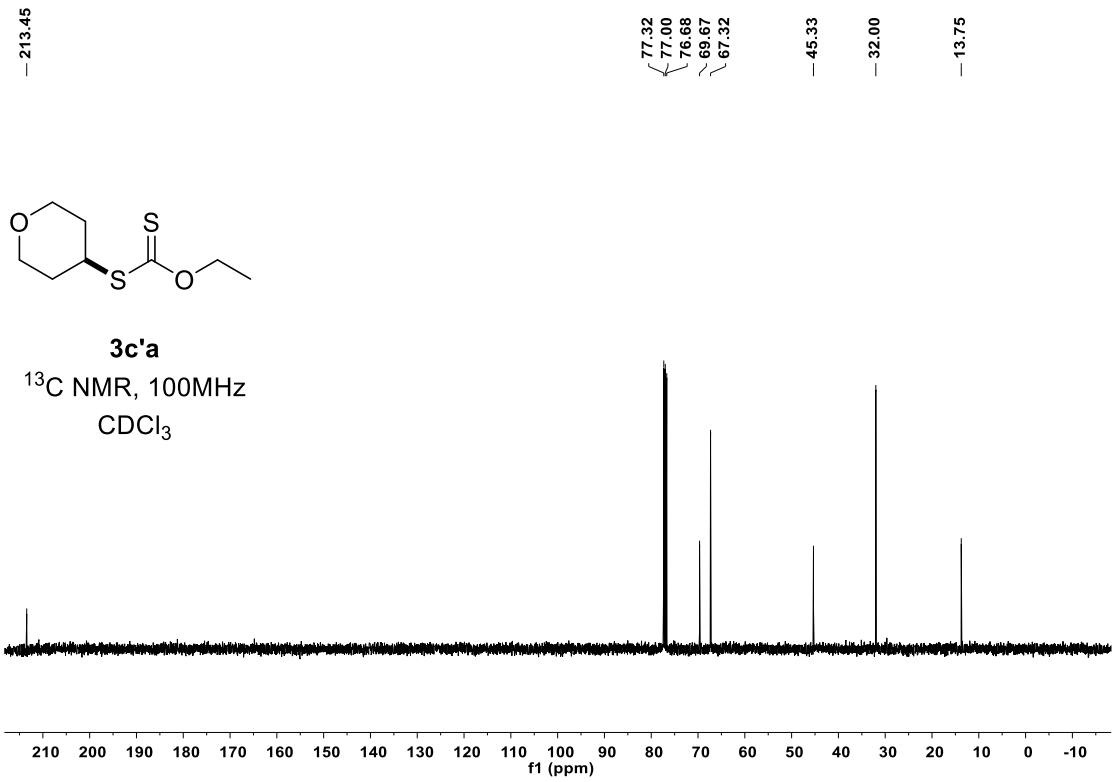
$CDCl_3$

3c'a

^{1}H NMR, 400MHz

$CDCl_3$

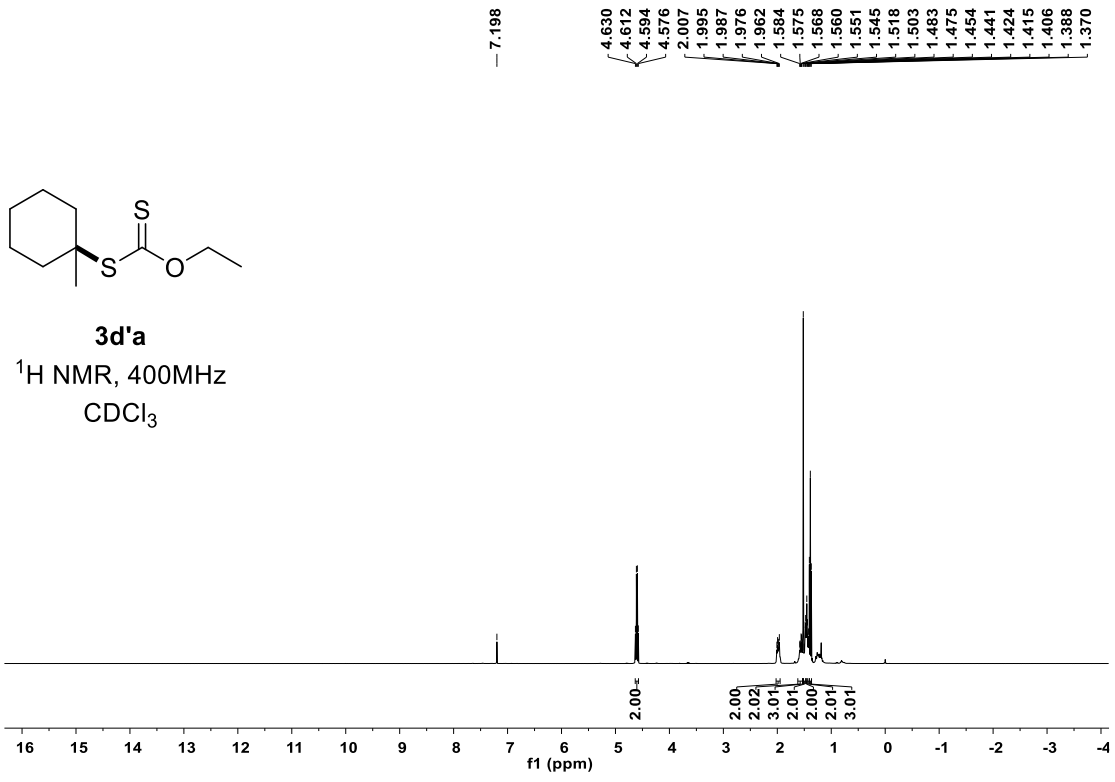




3c'a

^{13}C NMR, 100MHz

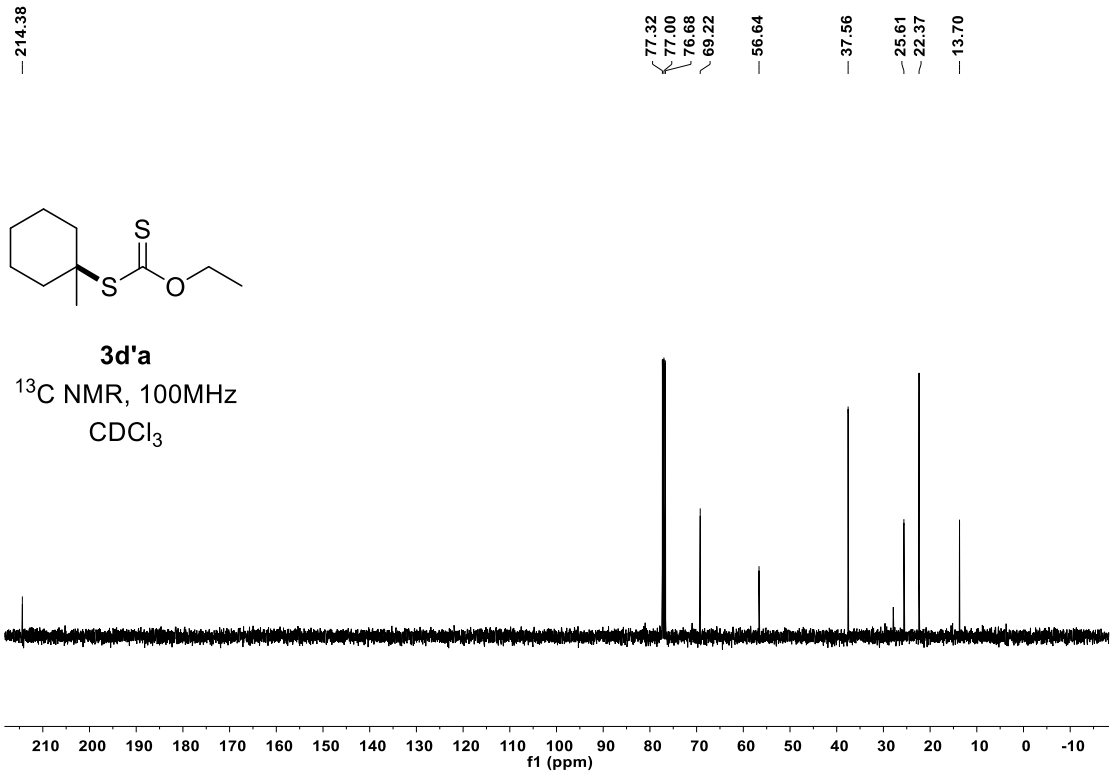
$CDCl_3$

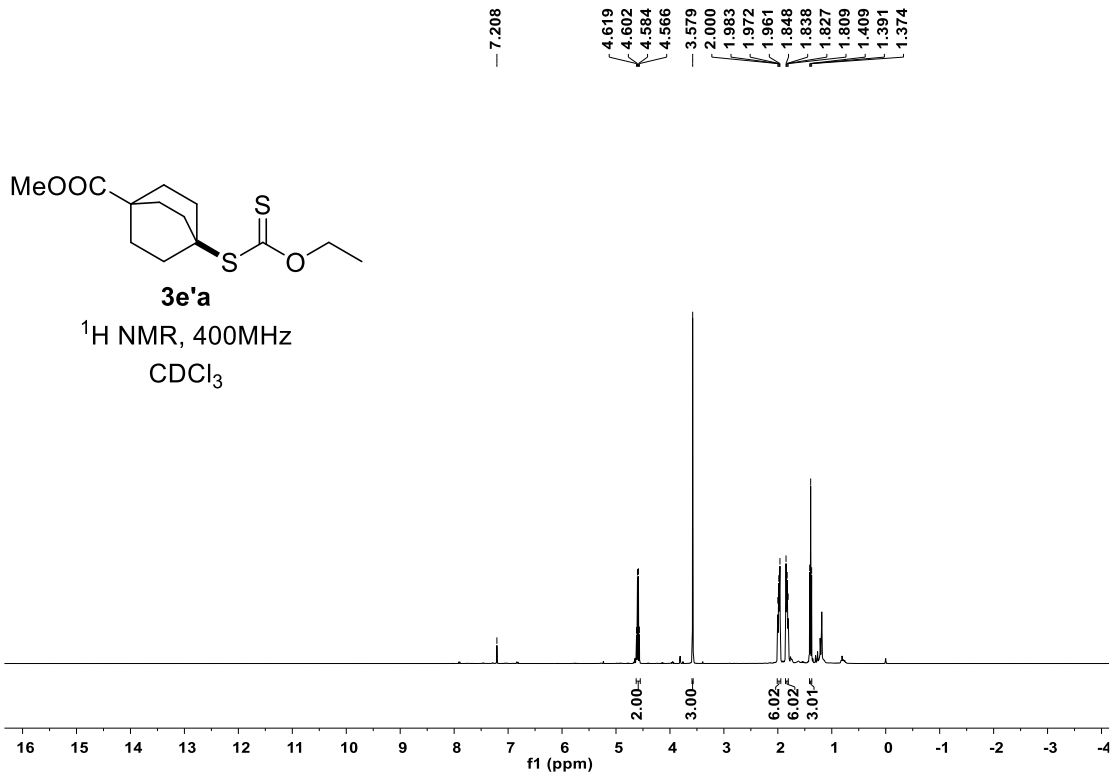


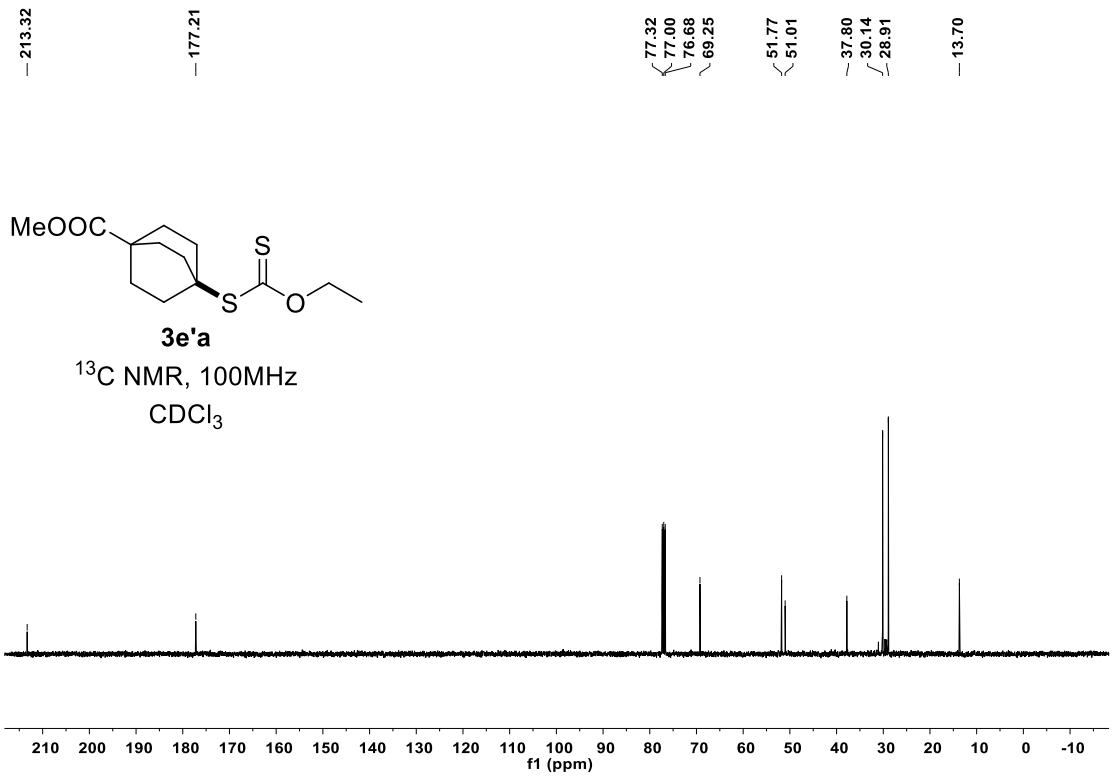
3d'a

^{1}H NMR, 400MHz

$CDCl_3$


214.38
77.32
77.00
76.68
69.22
56.64
37.56
25.61
22.37
13.70
S
S
O
3d'a
13C NMR, 100MHz
CDCl3
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)

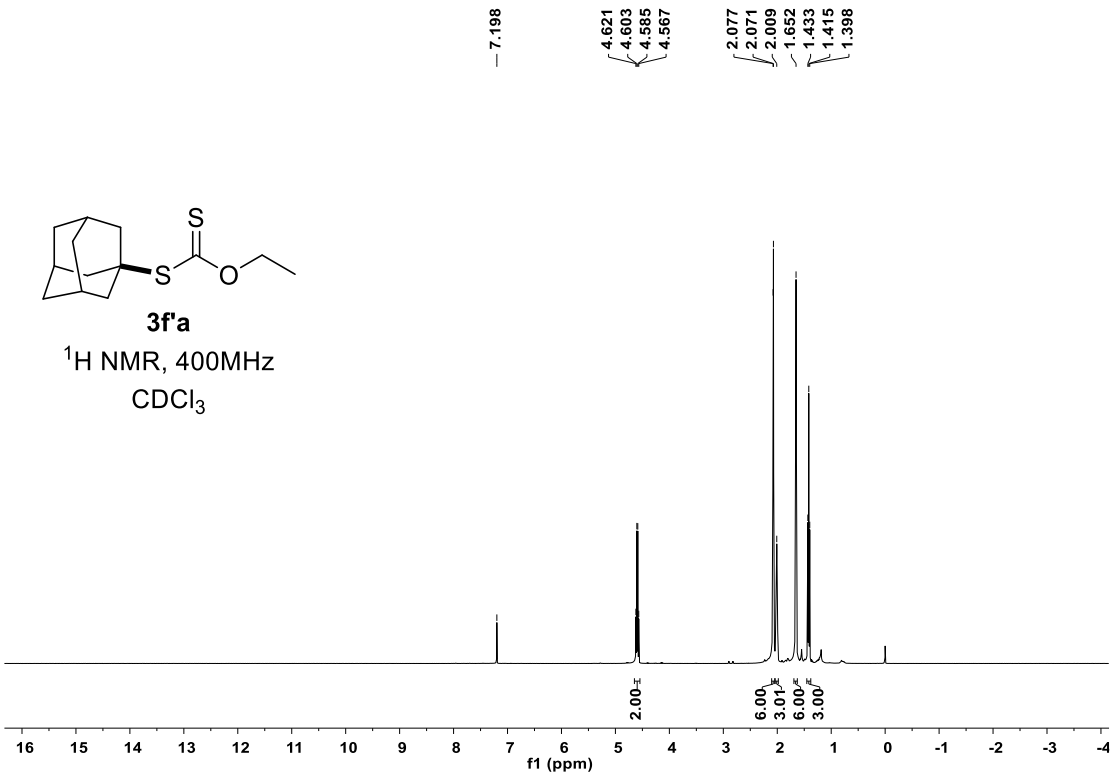

7.208
4.619
4.602
4.584
4.566
3.579
2.000
1.983
1.972
1.961
1.848
1.838
1.827
1.809
1.409
1.391
1.374
MeOOC
S
S
O
3e'a
1H NMR, 400MHz
CDCl3
2.00
3.00
6.02
6.02
3.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)



3e'a

^{13}C NMR, 100MHz

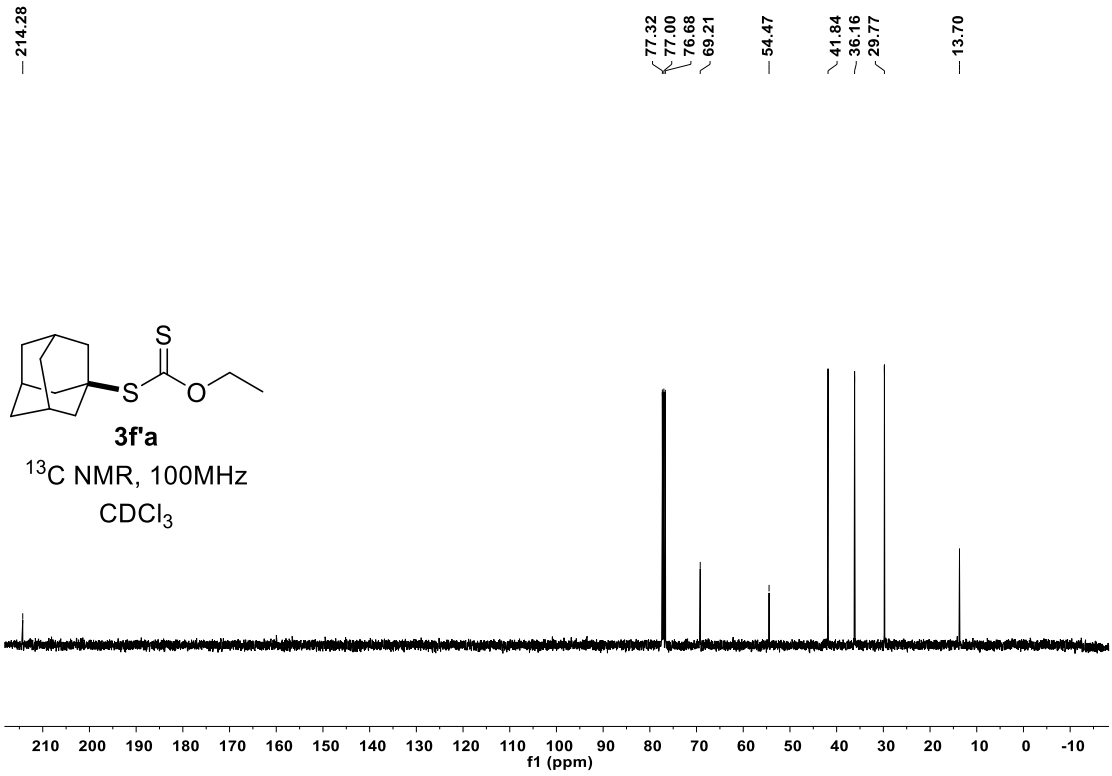
$CDCl_3$

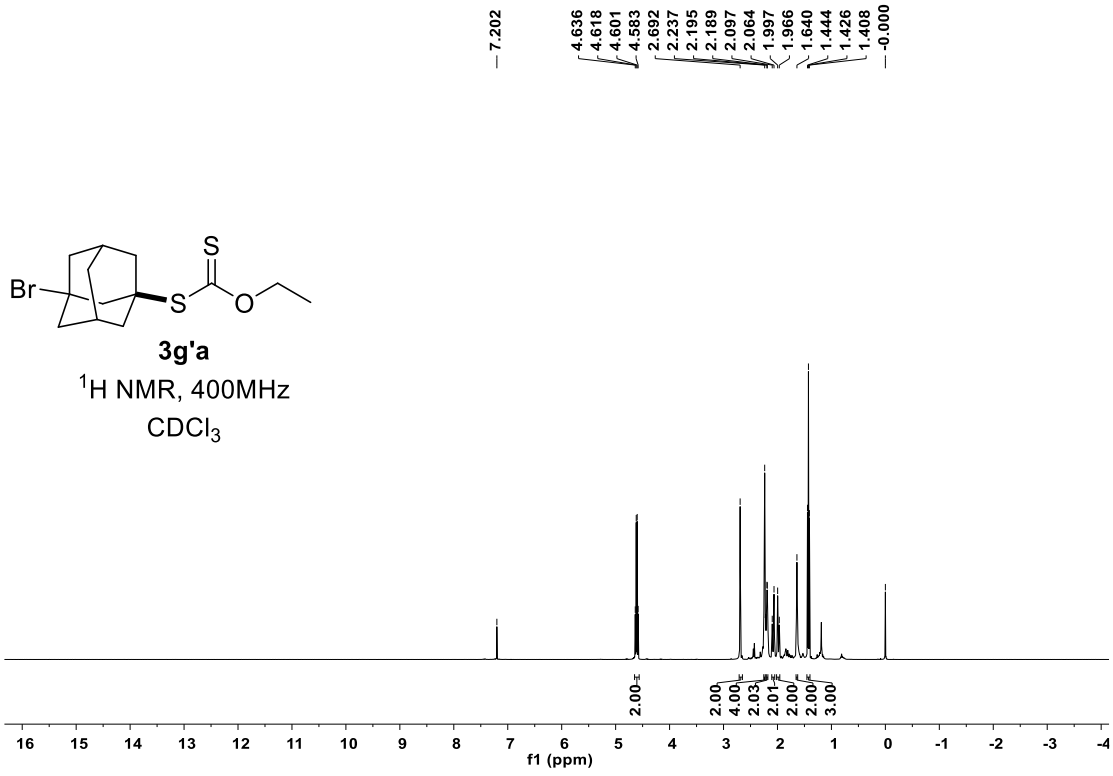


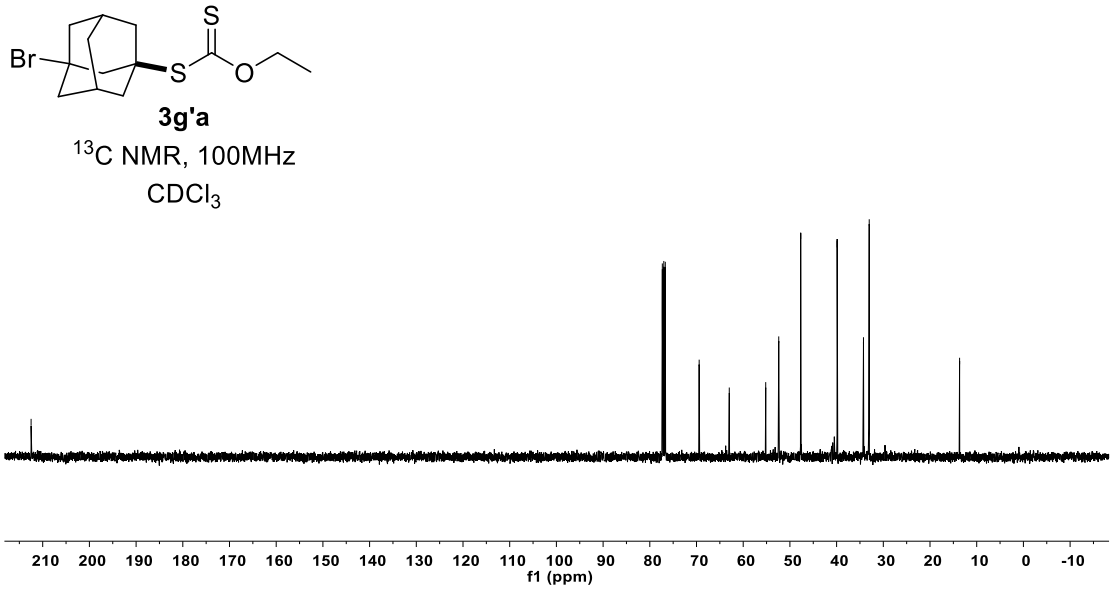
3f'a

^{1}H NMR, 400MHz

$CDCl_3$


214.28
77.32
77.00
76.68
69.21
54.47
41.84
36.16
29.77
13.70
S
S
O
3f'a
13C NMR, 100MHz
CDCl3
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)

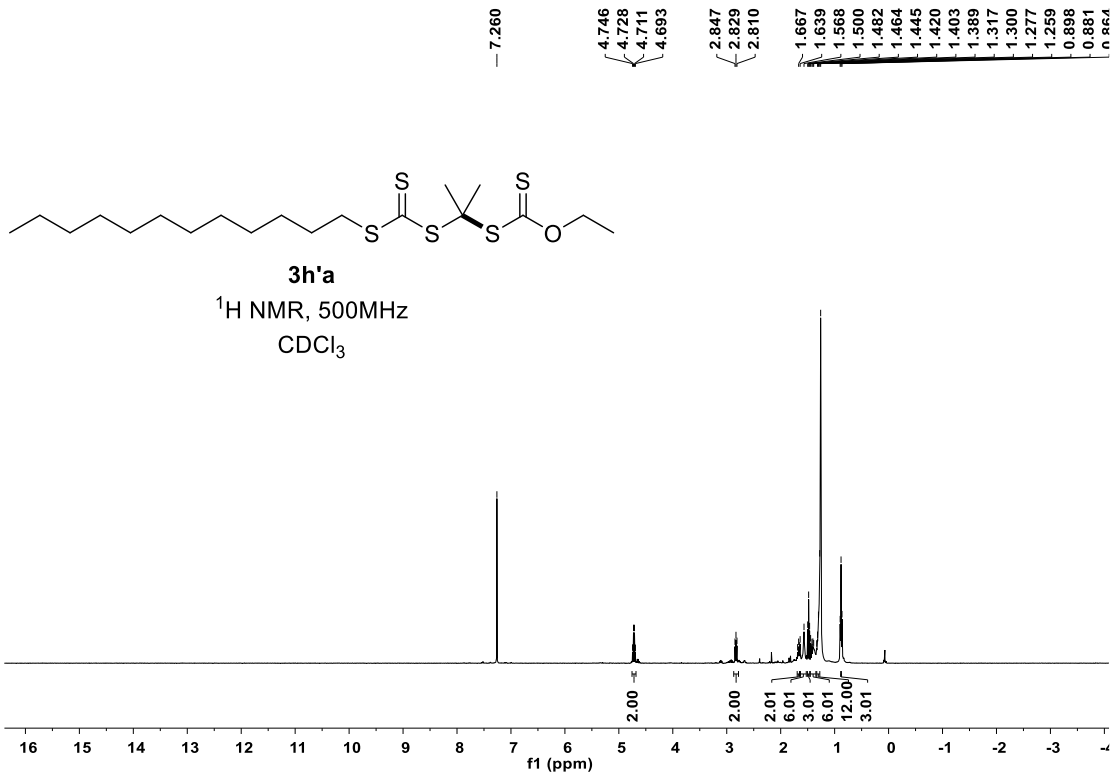

7.202
4.636
4.618
4.601
4.583
2.692
2.237
2.195
2.189
2.097
2.064
1.997
1.966
1.640
1.444
1.426
1.408
-0.000
Br
S
S
O
3g'a
1H NMR, 400MHz
CDCl3
2.00
2.00
4.00
2.03
2.01
2.00
2.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)



3g'a

^{13}C NMR, 100MHz

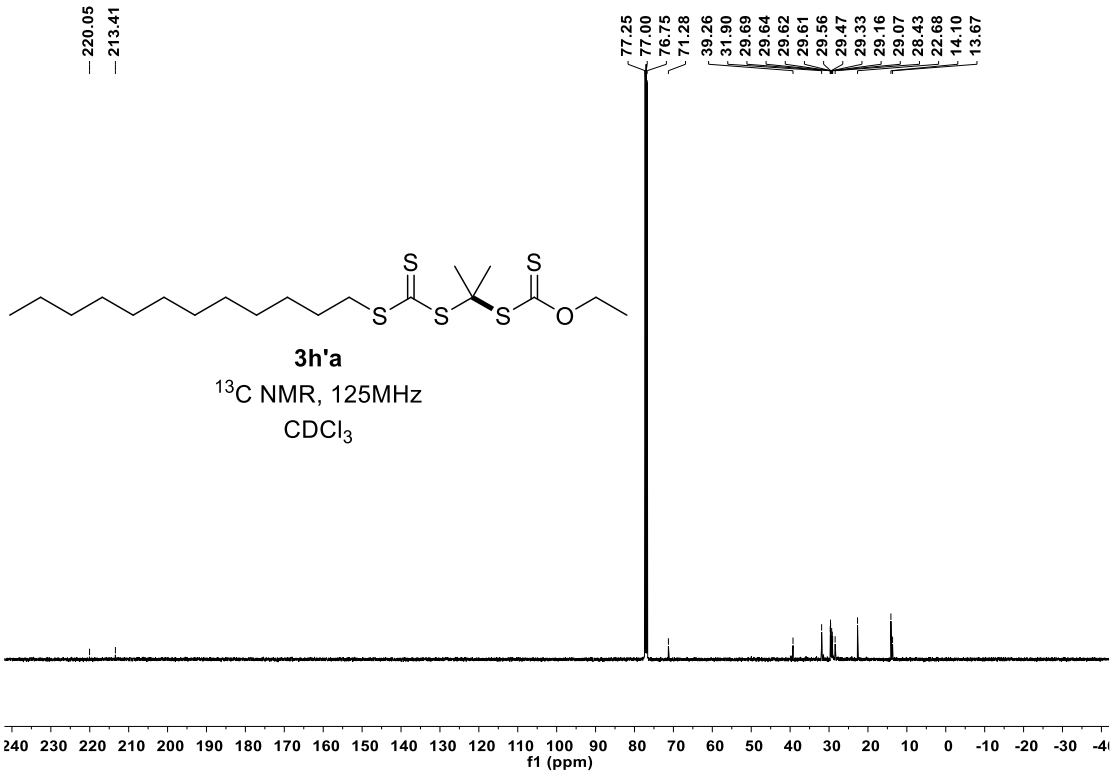
$CDCl_3$

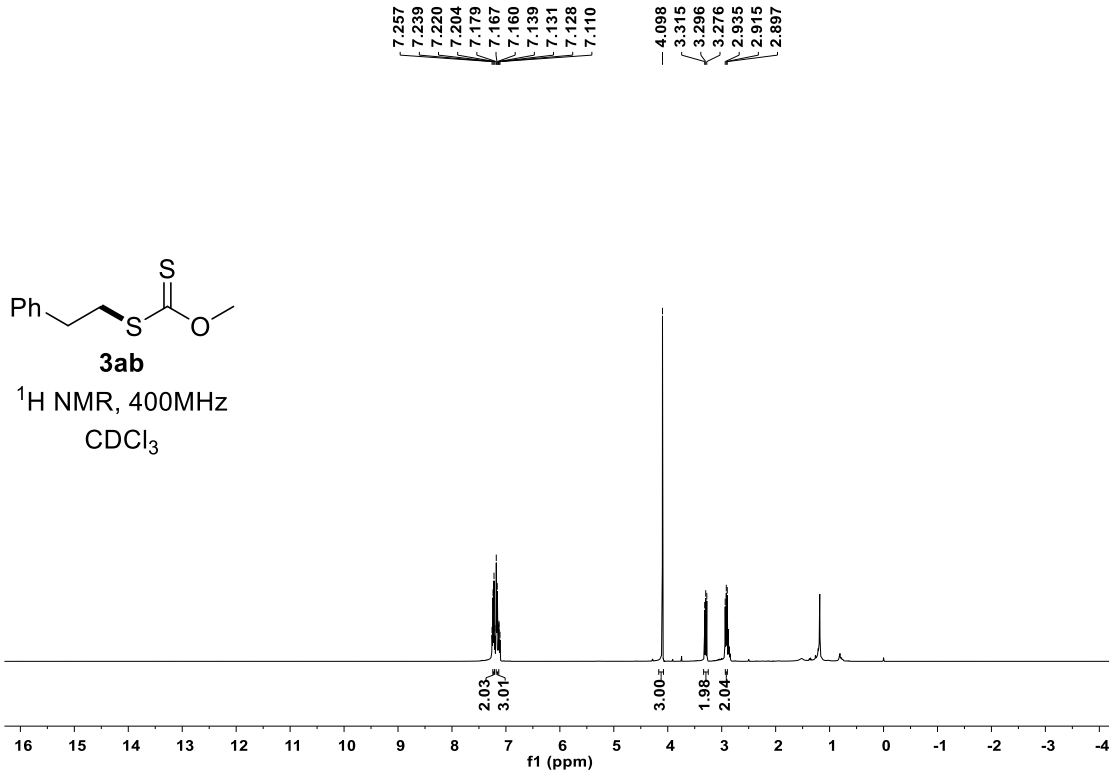


3h'a

^{1}H NMR, 500MHz

$CDCl_3$


220.05
213.41
77.25
77.00
76.75
71.28
39.26
31.90
29.69
29.64
29.62
29.61
29.56
29.47
29.33
29.16
29.07
28.43
22.68
14.10
13.67
S
S
S
S
S
O
3h'a
13C NMR, 125MHz
CDCl3
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)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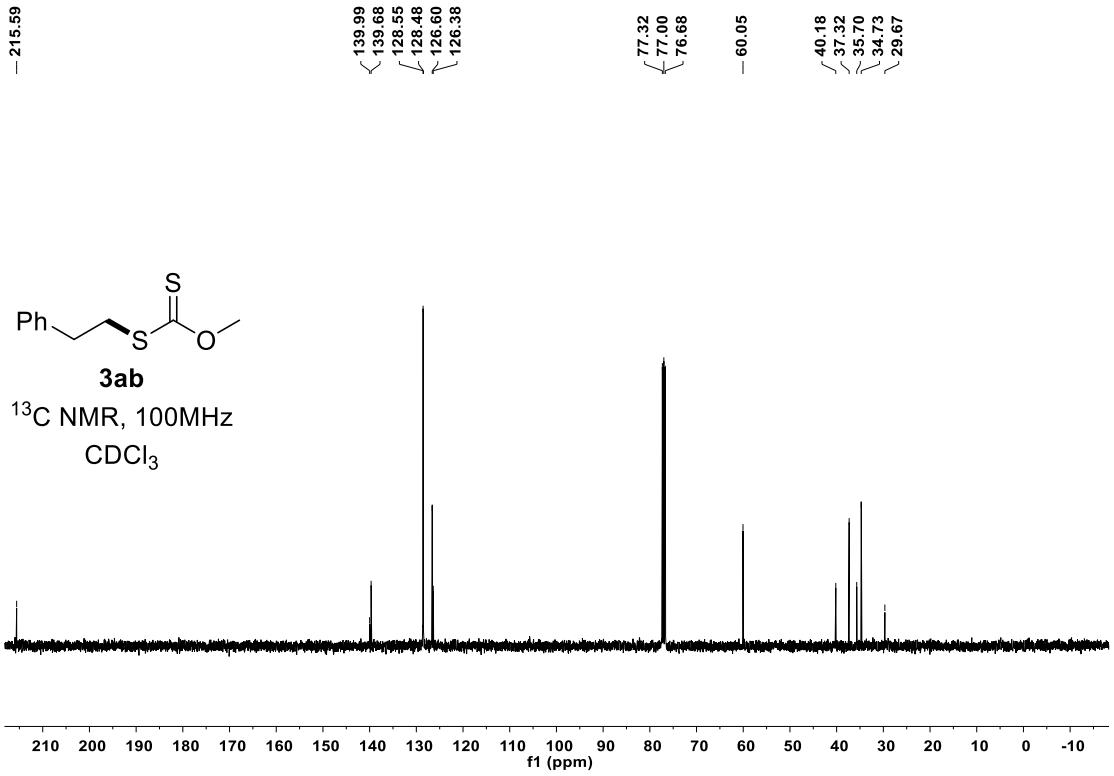
7.220
7.204
7.179
7.167
7.160
7.139
7.131
7.128
7.110
4.098
3.315
3.296
3.276
2.935
2.915
2.897
Ph
S
S
O
3ab
1H NMR, 400MHz
CDCl3
2.03
3.01
3.00
1.98
2.04
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)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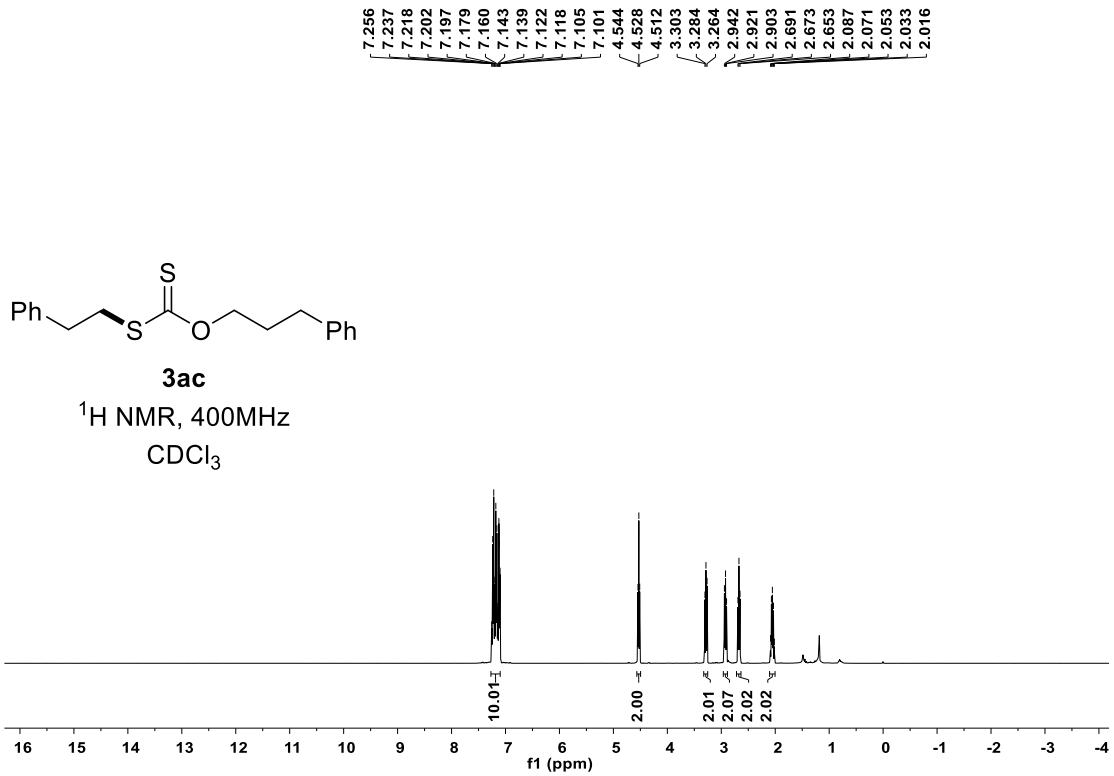
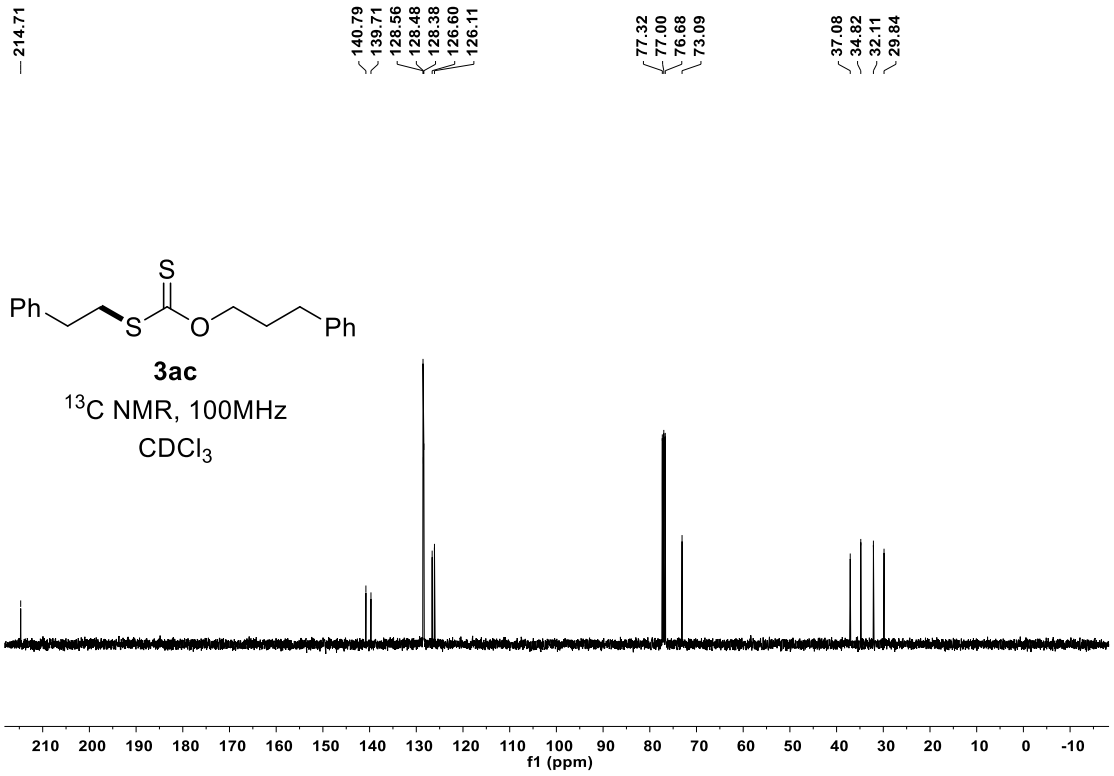
139.68
128.55
128.48
126.60
126.38
77.32
77.00
76.68
60.05
40.18
37.32
35.70
34.73
29.67
Ph
S
S
O
3ab
13C NMR, 100MHz
CDCl3
f1 (ppm)


7.256
7.237
7.218
7.202
7.197
7.179
7.160
7.143
7.139
7.122
7.118
7.105
7.101
4.544
4.528
4.512
3.303
3.284
3.264
2.942
2.921
2.903
2.691
2.673
2.653
2.087
2.071
2.053
2.033
2.016
Ph
S
S
O
Ph
3ac
1H NMR, 400MHz
CDCl3
10.01
2.00
2.01
2.07
2.02
2.02
f1 (ppm)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140.79
139.71
128.56
128.48
128.38
126.60
126.11
77.32
77.00
76.68
73.09
37.08
34.82
32.11
29.84
Ph
S
S
O
Ph
3ac
13C NMR, 100MHz
CDCl3
f1 (ppm)


7.260
7.240
7.230
7.224
7.180
7.161
7.142
5.796
5.780
5.771
5.763
5.754
5.737
5.728
5.721
5.712
5.695
5.015
5.011
4.972
4.968
4.964
4.961
4.958
4.938
4.935
4.932
4.551
4.534
4.518
3.305
3.286
3.266
2.940
2.920
2.901
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2.127
2.110
2.107
2.091
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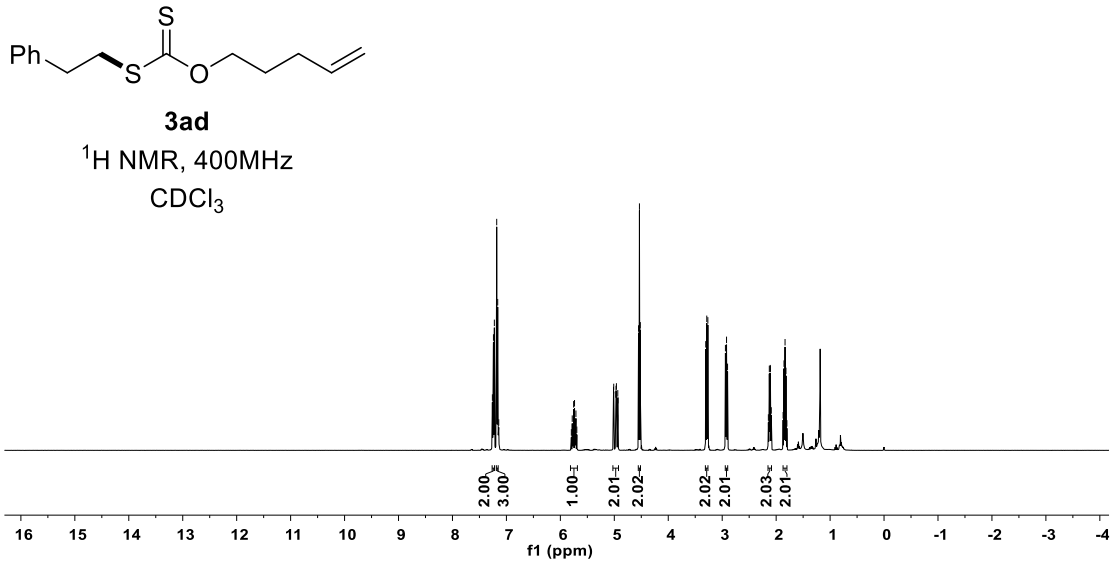
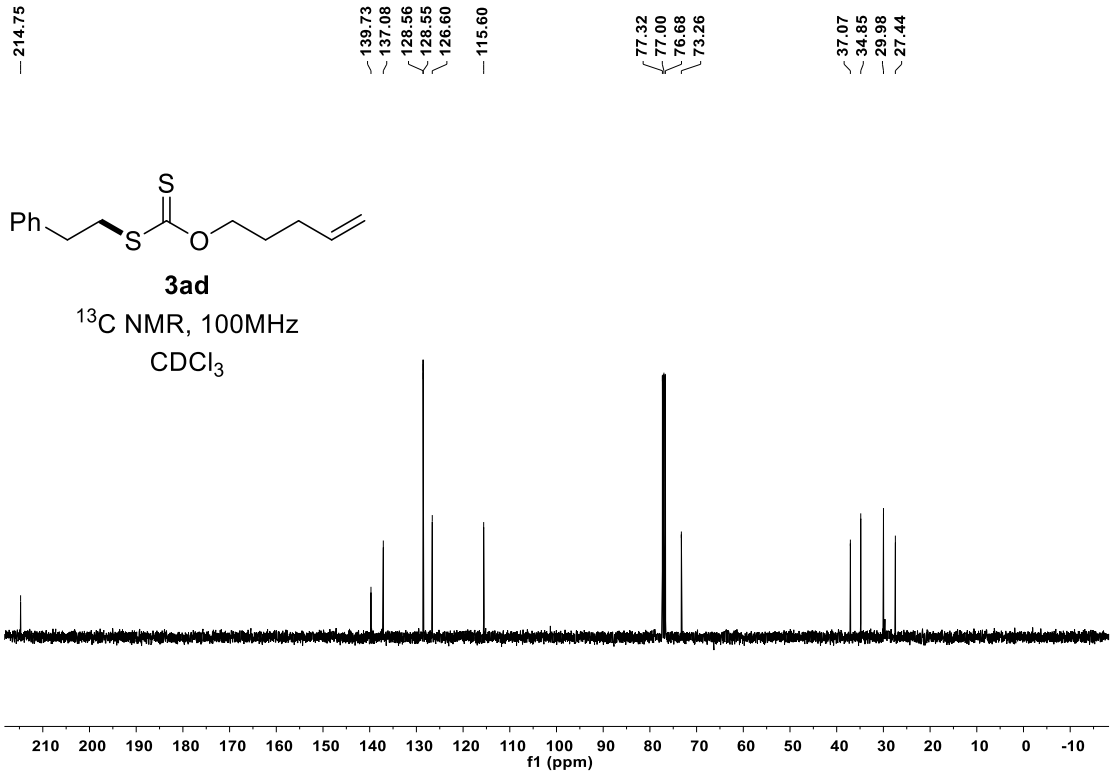
1.854
1.835
1.818
1.801


Ph
S
S
O
3ad
1H NMR, 400MHz
CDCl3
2.00
3.00
1.00
2.01
2.02
2.02
2.01
2.03
2.01
f1 (ppm)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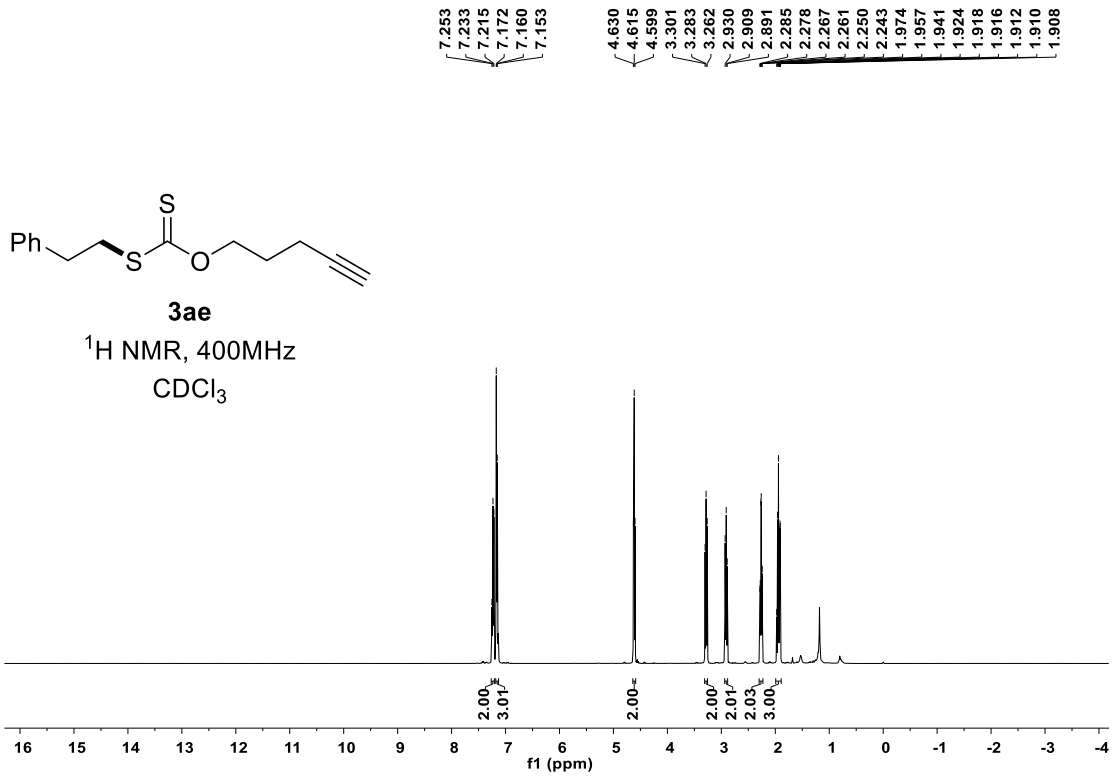
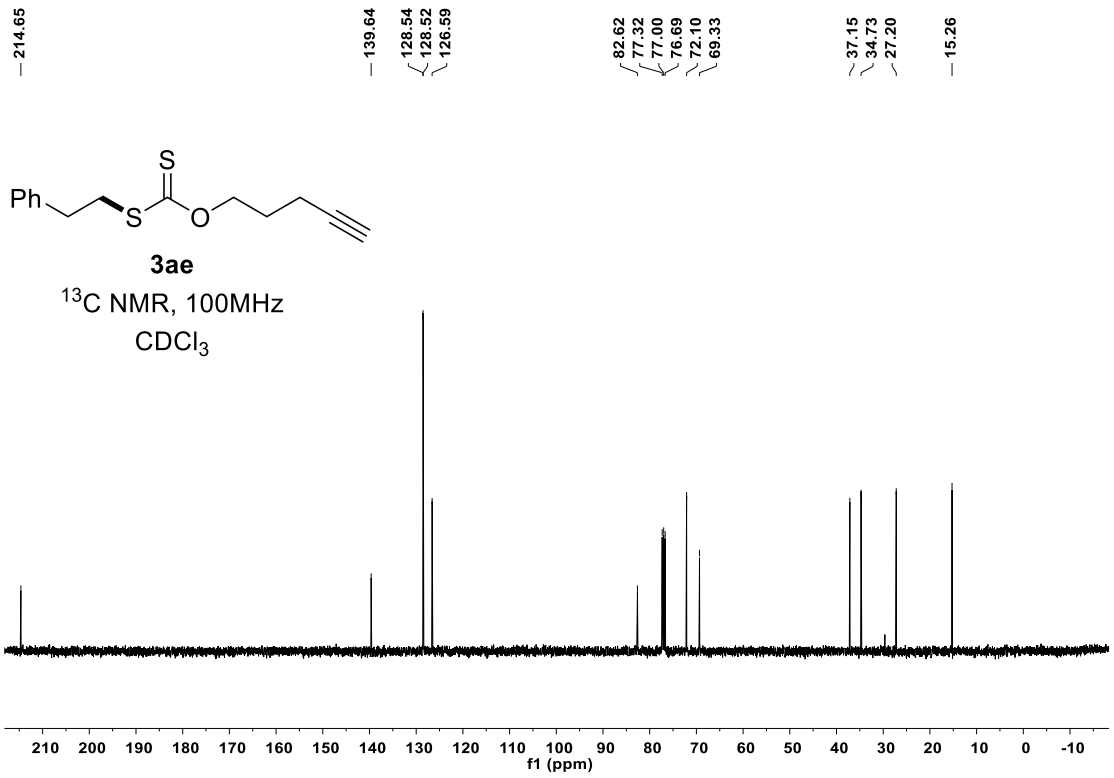
128.55
126.60
115.60
77.32
77.00
76.68
73.26
37.07
34.85
29.98
27.44
Ph
S
S
O
3ad
13C NMR, 100MHz
CDCl3
f1 (ppm)


7.253
7.233
7.215
7.172
7.160
7.153
4.630
4.615
4.599
3.301
3.283
3.262
2.930
2.909
2.891
2.285
2.278
2.267
2.261
2.250
2.243
1.974
1.957
1.941
1.924
1.918
1.916
1.912
1.910
1.908
Ph
S
S
O
3ae
1H NMR, 400MHz
CDCl3
2.00
3.01
2.00
2.00
2.01
2.03
3.00
f1 (ppm)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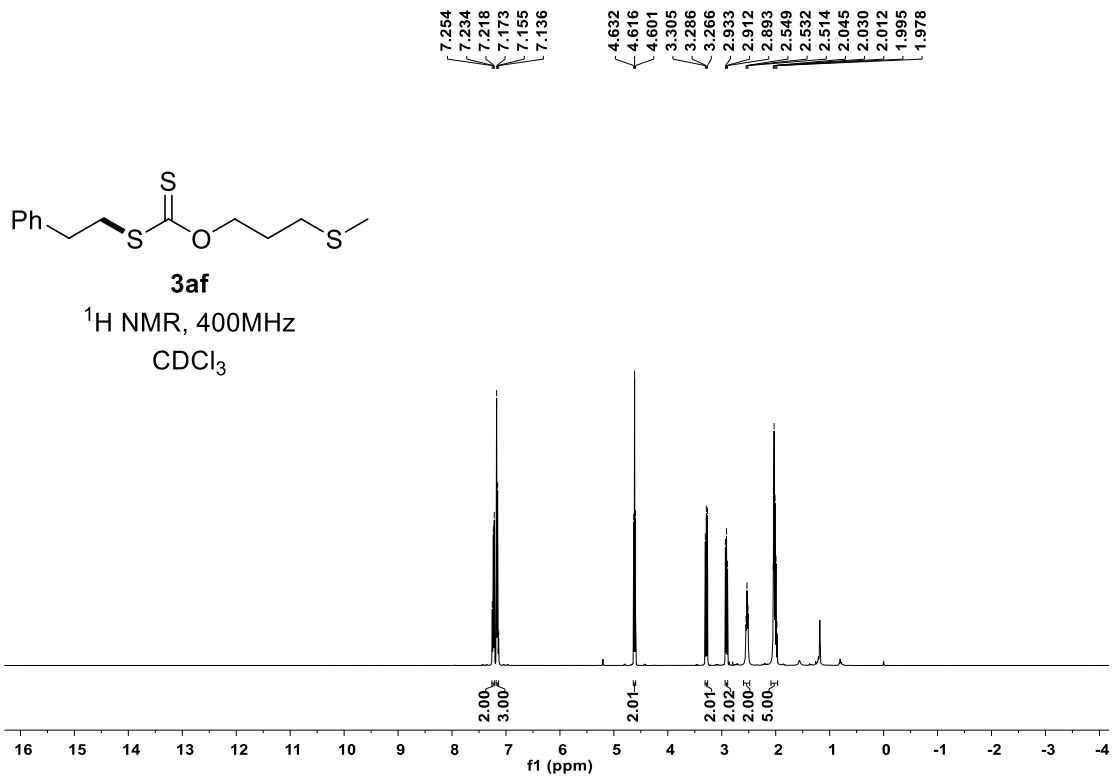
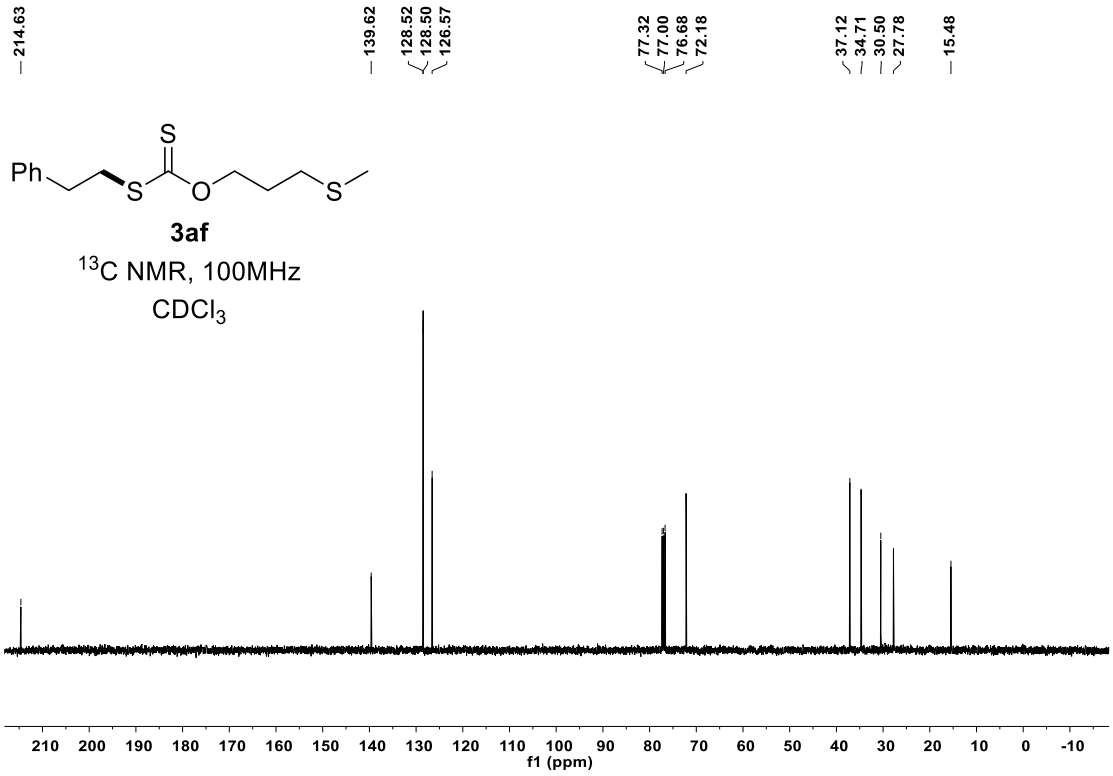
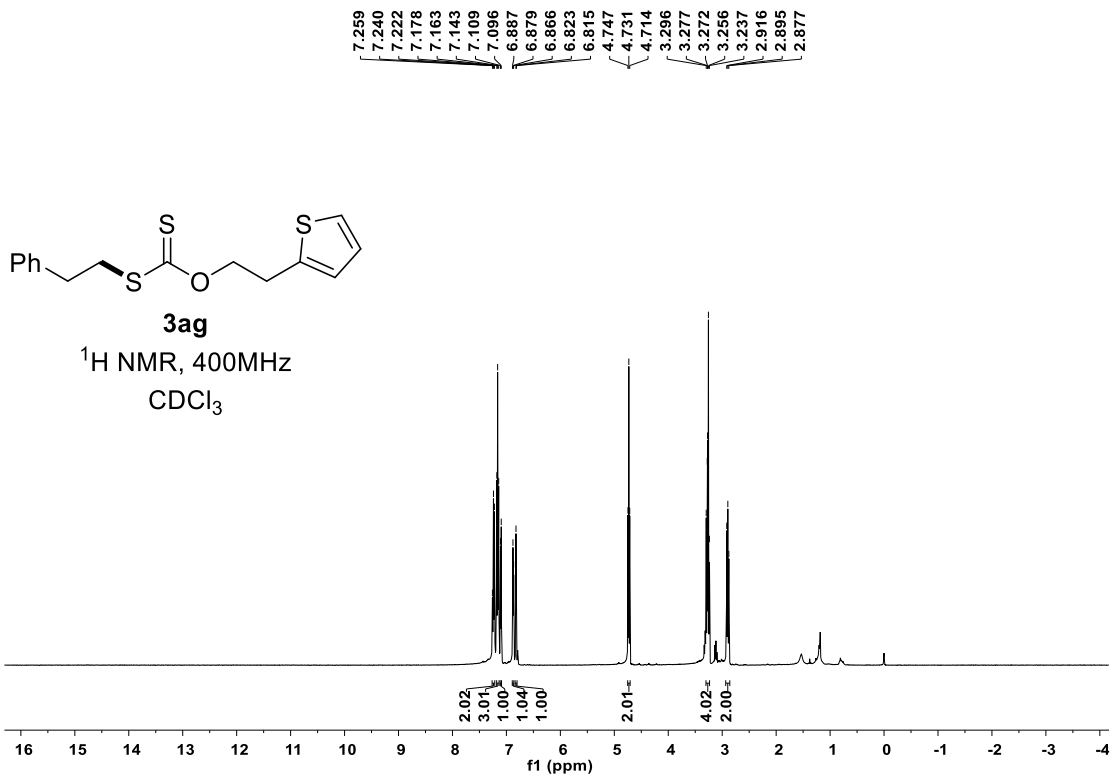
139.64
128.54
128.52
126.59
82.62
77.32
77.00
76.69
72.10
69.33
37.15
34.73
27.20
15.26
Ph
S
S
O
3ae
13C NMR, 100MHz
CDCl3
f1 (ppm)


7.254
7.234
7.218
7.173
7.155
7.136
4.632
4.616
4.601
3.305
3.286
3.266
2.933
2.912
2.893
2.549
2.532
2.514
2.045
2.030
2.012
1.995
1.978
Ph
S
S
O
S
3af
1H NMR, 400MHz
CDCl3
2.00
3.00
2.01
2.01
2.02
2.00
5.00
f1 (ppm)


214.63
139.62
128.52
128.50
126.57
77.32
77.00
76.68
72.18
37.12
34.71
30.50
27.78
15.48
Ph
S
S
O
S
3af
13C NMR, 100MHz
CDCl3
f1 (ppm)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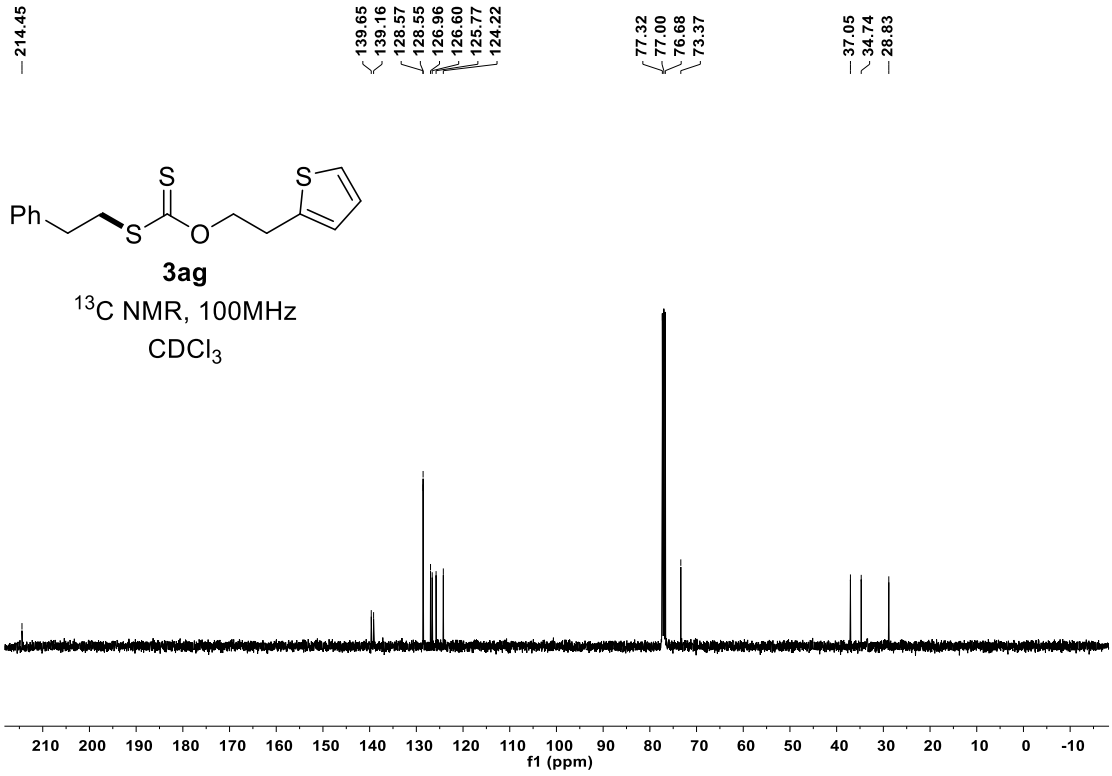
7.240
7.222
7.178
7.163
7.143
7.109
7.096
6.887
6.879
6.866
6.823
6.815
4.747
4.731
4.714
3.296
3.277
3.272
3.256
3.237
2.916
2.895
2.877
Ph
S
S
O
S
3ag
1H NMR, 400MHz
CDCl3
2.02
3.01
1.00
1.04
1.00
2.01
4.02
2.00
f1 (ppm)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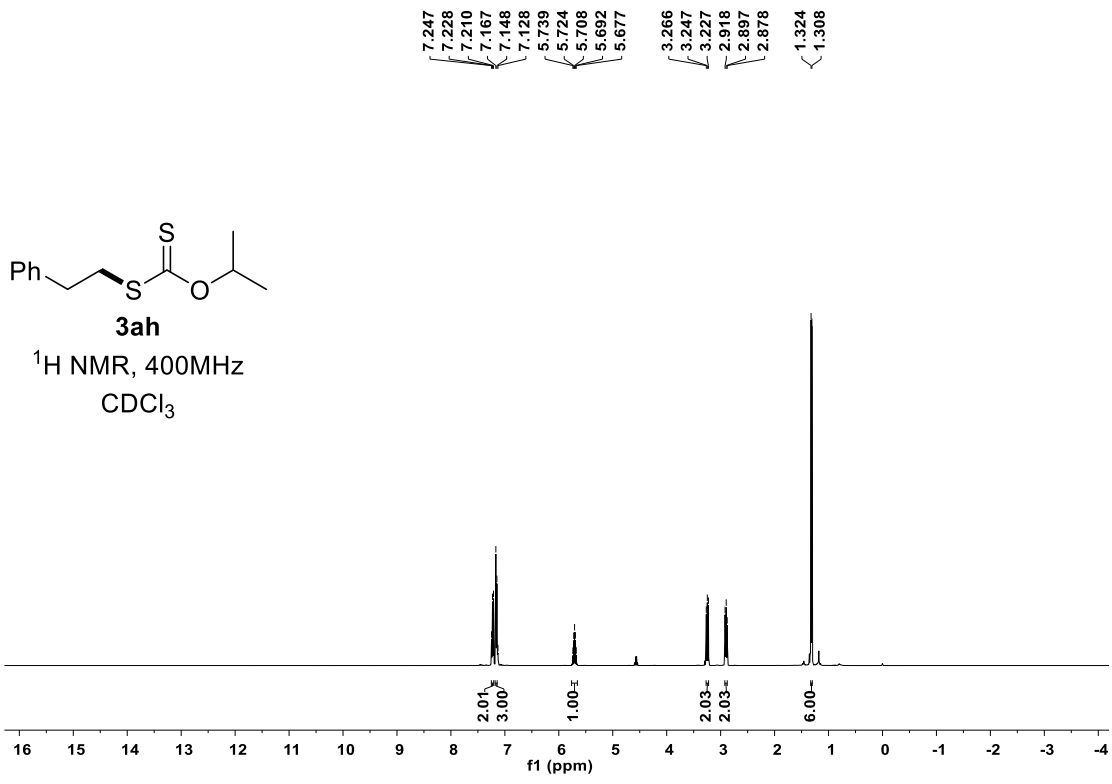
139.65
139.16
128.57
128.55
126.96
126.60
125.77
124.22
77.32
77.00
76.68
73.37
37.05
34.74
28.83
Ph
S
S
O
S
3ag
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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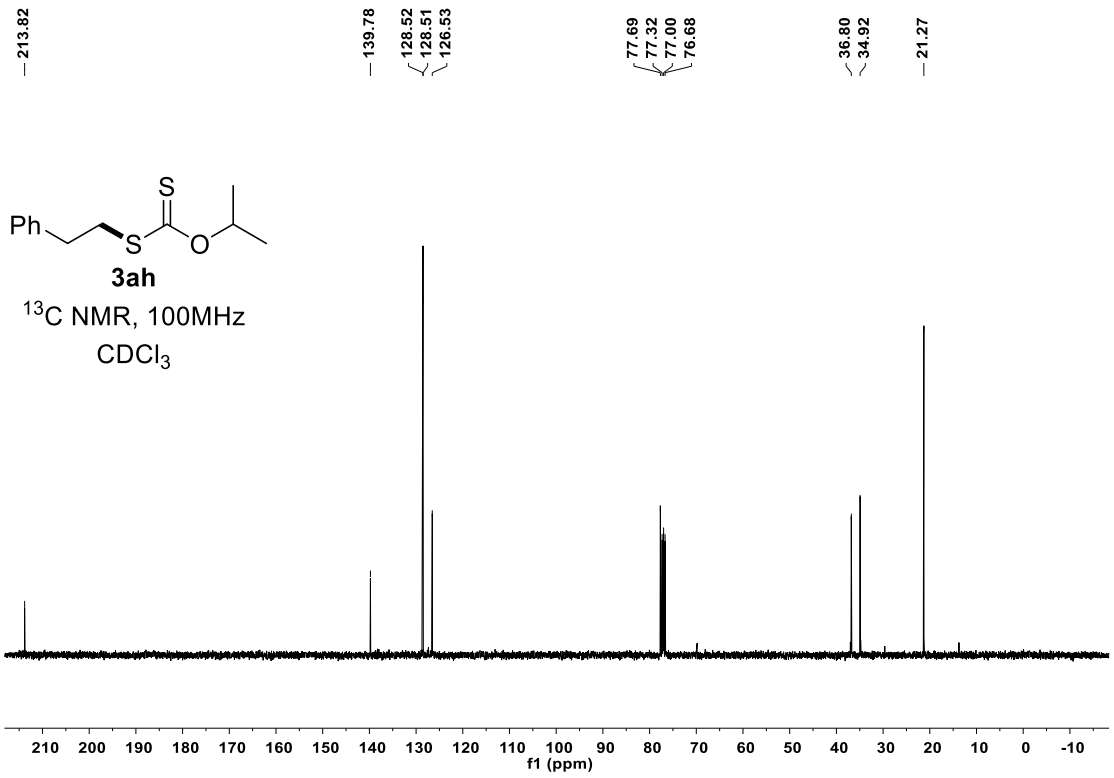
7.228
7.210
7.167
7.148
7.128
5.739
5.724
5.708
5.692
5.677
3.266
3.247
3.227
2.918
2.897
2.878
1.324
1.308
Ph
S
S
O
3ah
1H NMR, 400MHz
CDCl3
2.01
3.00
1.00
2.03
2.03
6.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)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128.51
126.53
77.69
77.32
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76.68
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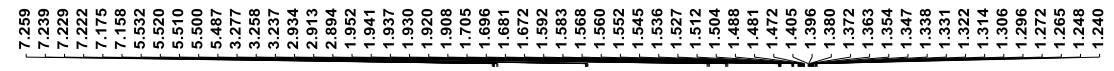
34.92
21.27
Ph
S
S
O
3ah
13C NMR, 100MHz
CDCl3
f1 (ppm)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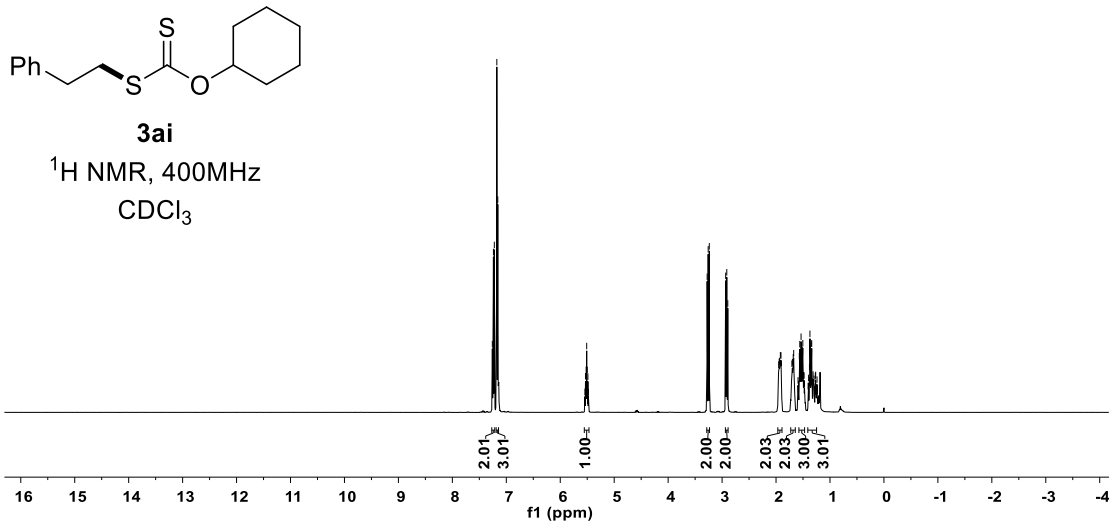
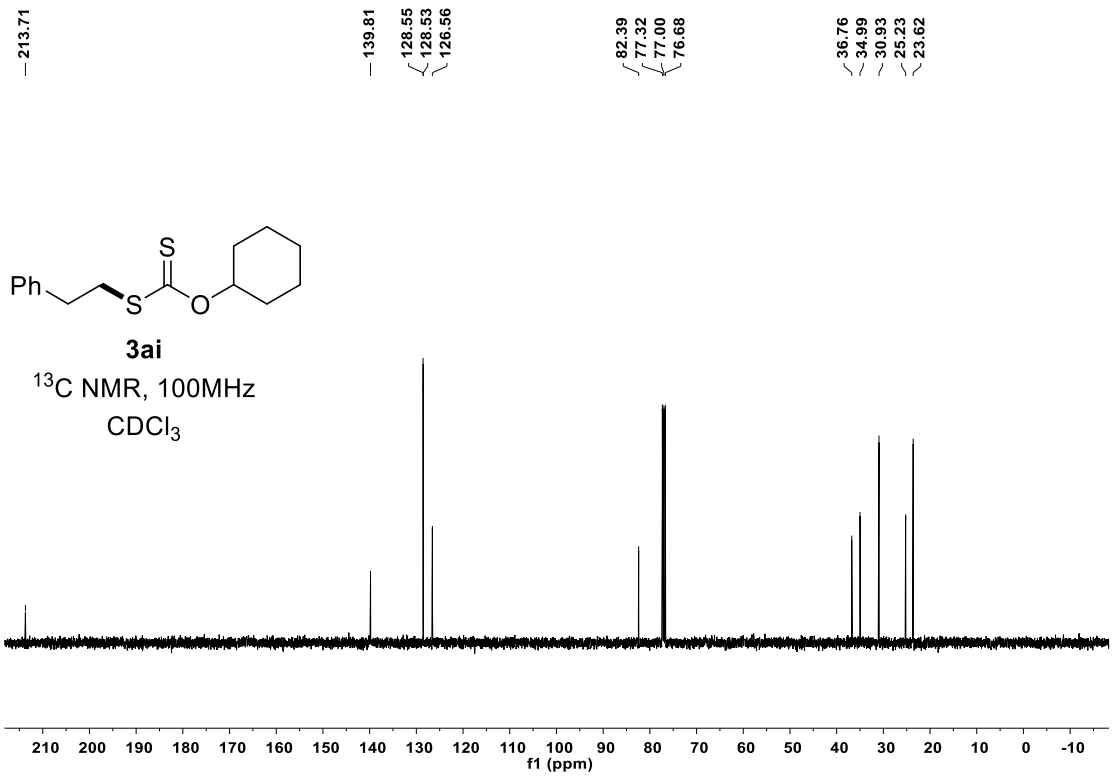
7.239
7.229
7.222
7.175
7.158
5.532
5.520
5.510
5.500
5.487
3.277
3.258
3.237
2.934
2.913
2.894
1.952
1.941
1.937
1.930
1.920
1.908
1.705
1.696
1.681
1.672
1.592
1.583
1.568
1.560
1.552
1.545
1.536
1.527
1.512
1.504
1.488
1.481
1.472
1.405
1.396
1.380
1.372
1.363
1.354
1.347
1.338
1.331
1.322
1.314
1.306
1.296
1.272
1.265
1.248
1.240


Ph
S
S
O
3ai
1H NMR, 400MHz
CDCl3
2.01
3.01
1.00
2.00
2.00
2.03
2.03
3.00
3.01
f1 (ppm)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139.81
128.55
128.53
126.56
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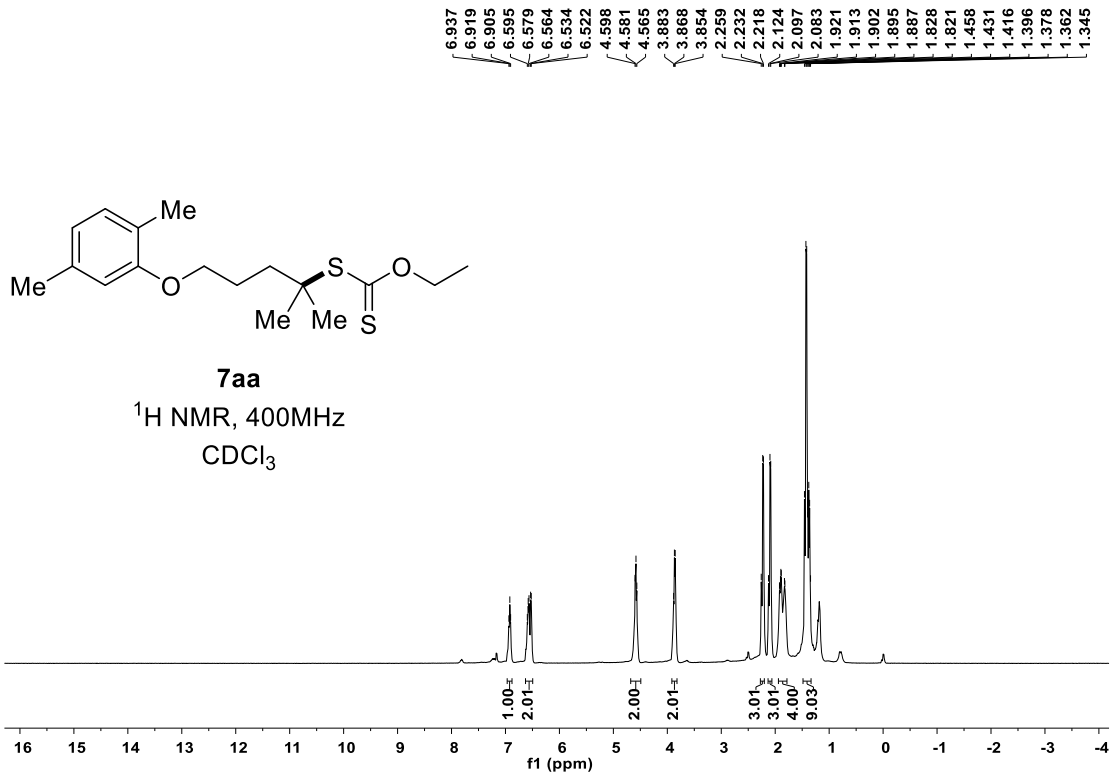
77.32
77.00
76.68
36.76
34.99
30.93
25.23
23.62
Ph
S
S
O
3ai
13C NMR, 100MHz
CDCl3
f1 (ppm)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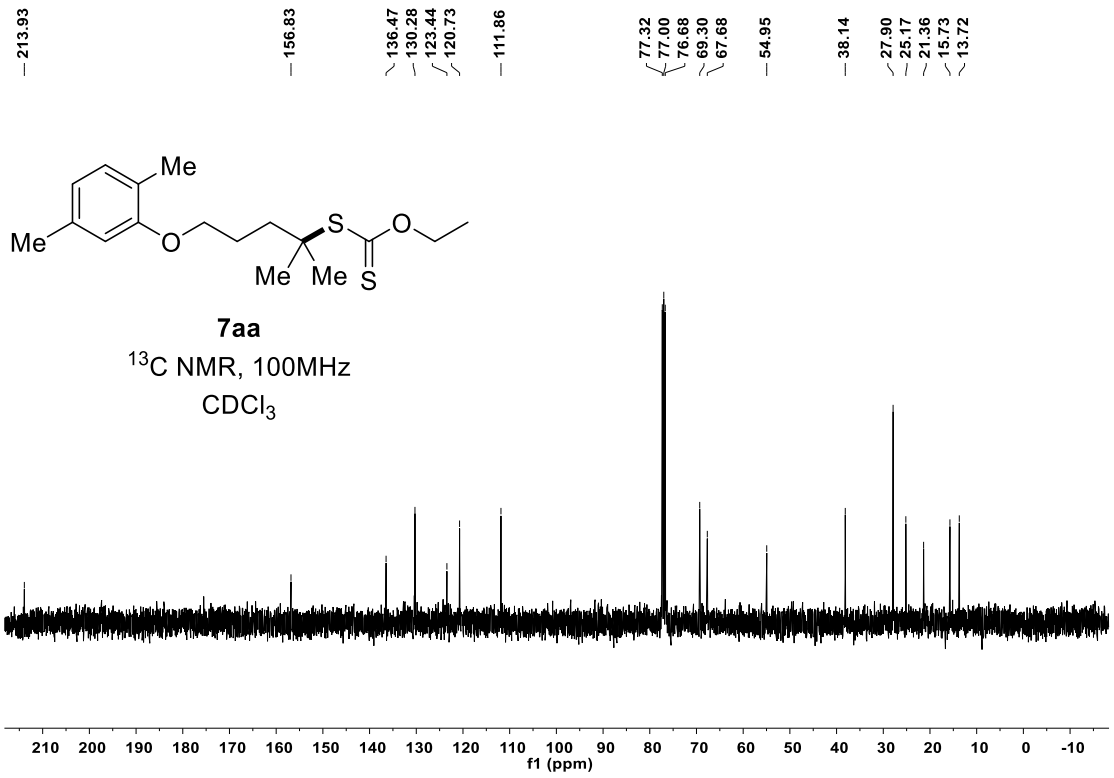
6.919
6.905
6.595
6.579
6.564
6.534
6.522
4.598
4.581
4.565
3.883
3.868
3.854
2.259
2.232
2.218
2.124
2.097
2.083
1.921
1.913
1.902
1.895
1.887
1.828
1.821
1.458
1.431
1.416
1.396
1.378
1.362
1.345
Me
Me
O
S
O
Me
Me
S
7aa
1H NMR, 400MHz
CDCl3
1.00
2.01
2.00
2.01
3.01
3.01
4.00
9.03
f1 (ppm)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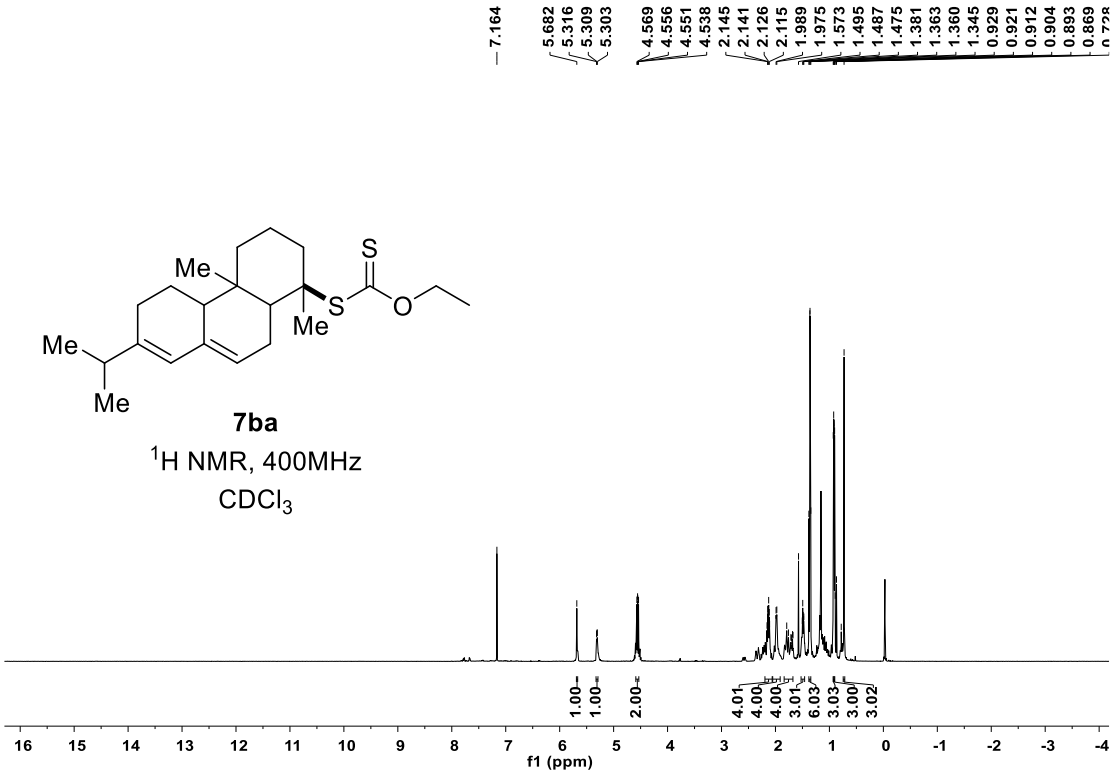
156.83
136.47
130.28
123.44
120.73
111.86
77.32
77.00
76.68
69.30
67.68
54.95
38.14
27.90
25.17
21.36
15.73
13.72
Me
Me
O
S
O
Me
Me
S
7aa
13C NMR, 100MHz
CDCl3
f1 (ppm)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5.682
5.316
5.309
5.303
4.569
4.556
4.551
4.538
2.145
2.141
2.126
2.115
1.989
1.975
1.573
1.495
1.487
1.475
1.381
1.363
1.360
1.345
0.929
0.921
0.912
0.904
0.893
0.869
Me
S
O
Me
Me
Me
7ba
1H NMR, 400MHz
CDCl3
1.00
1.00
2.00
4.01
4.00
4.00
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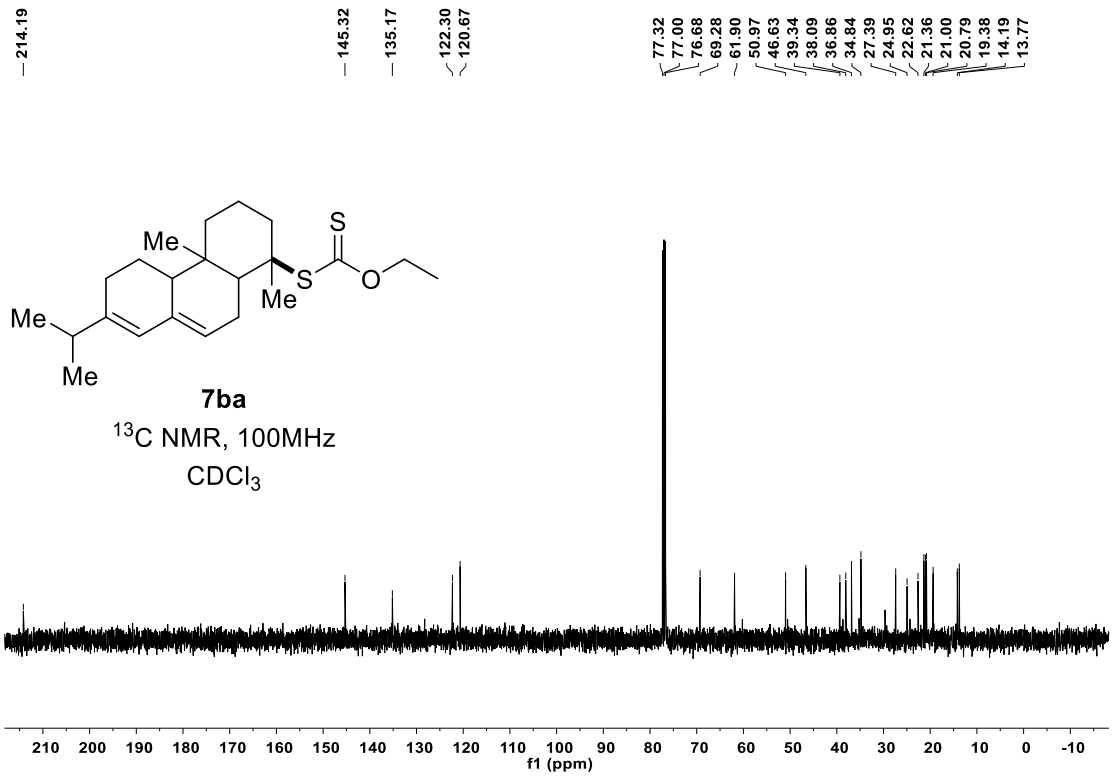
6.03
3.03
3.00
3.02
f1 (ppm)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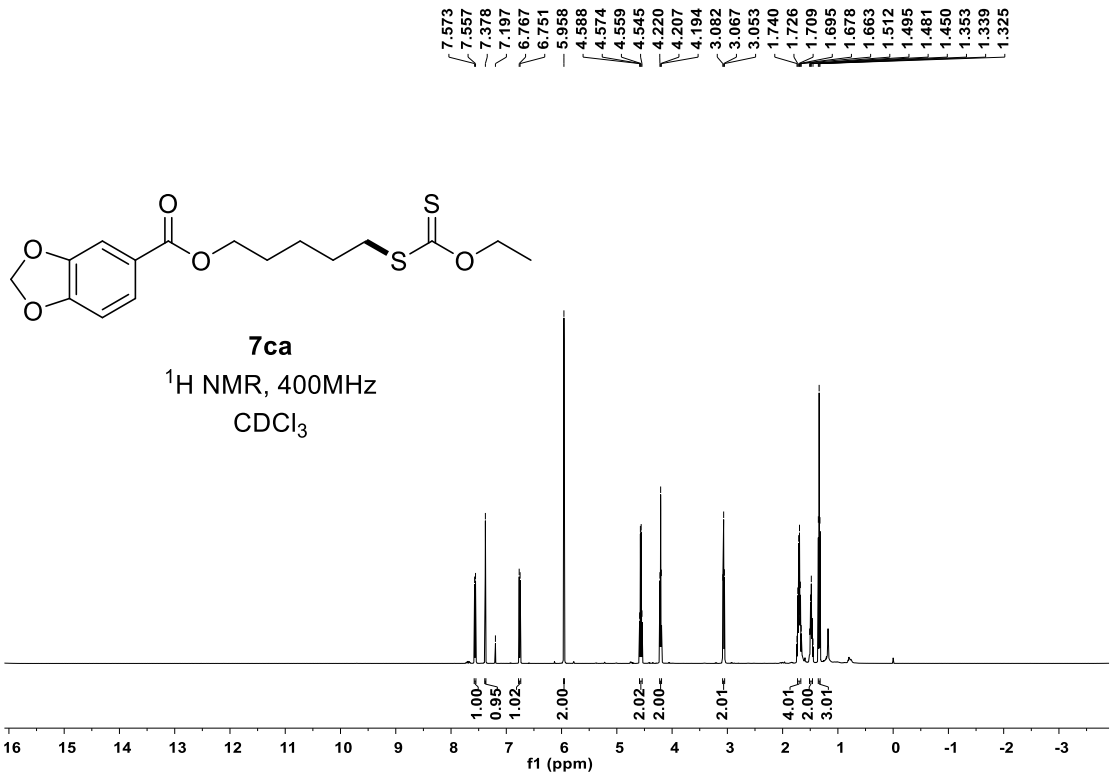
145.32
135.17
122.30
120.67
77.32
77.00
76.68
69.28
61.90
50.97
46.63
39.34
38.09
36.86
34.84
27.39
24.95
22.62
21.36
21.00
20.79
19.38
14.19
13.77
Me
Me
Me
S
S
O
7ba
13C NMR, 100MHz
CDCl3
f1 (ppm)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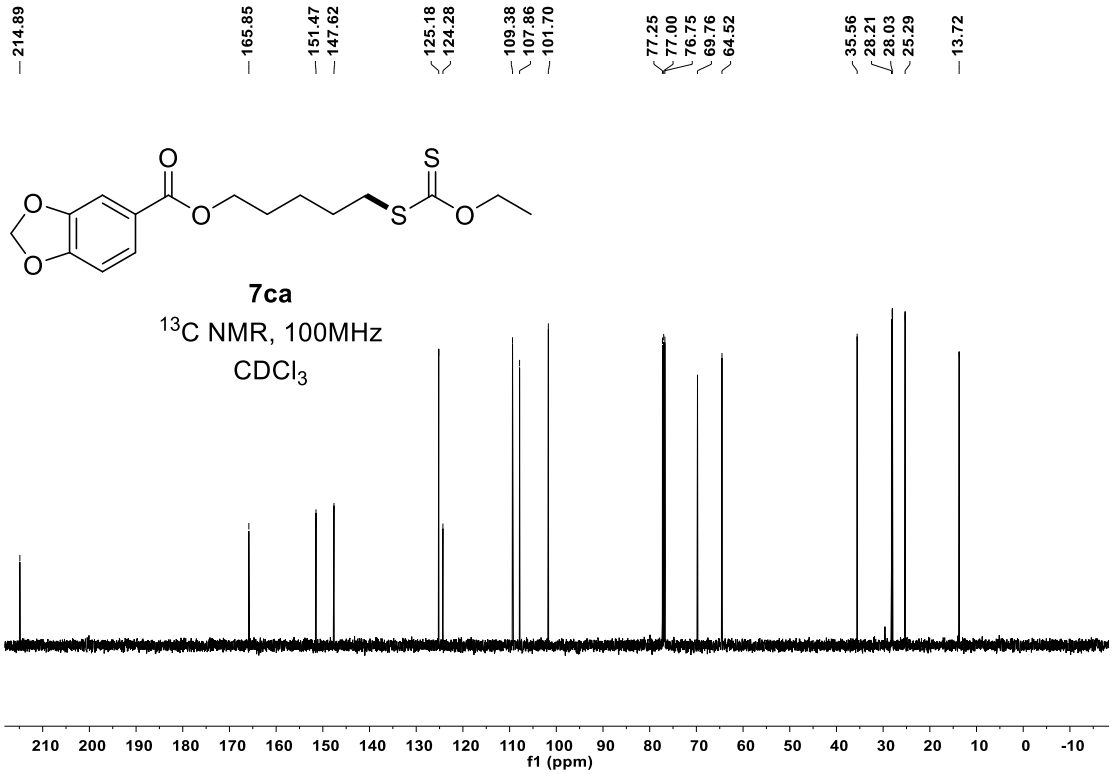
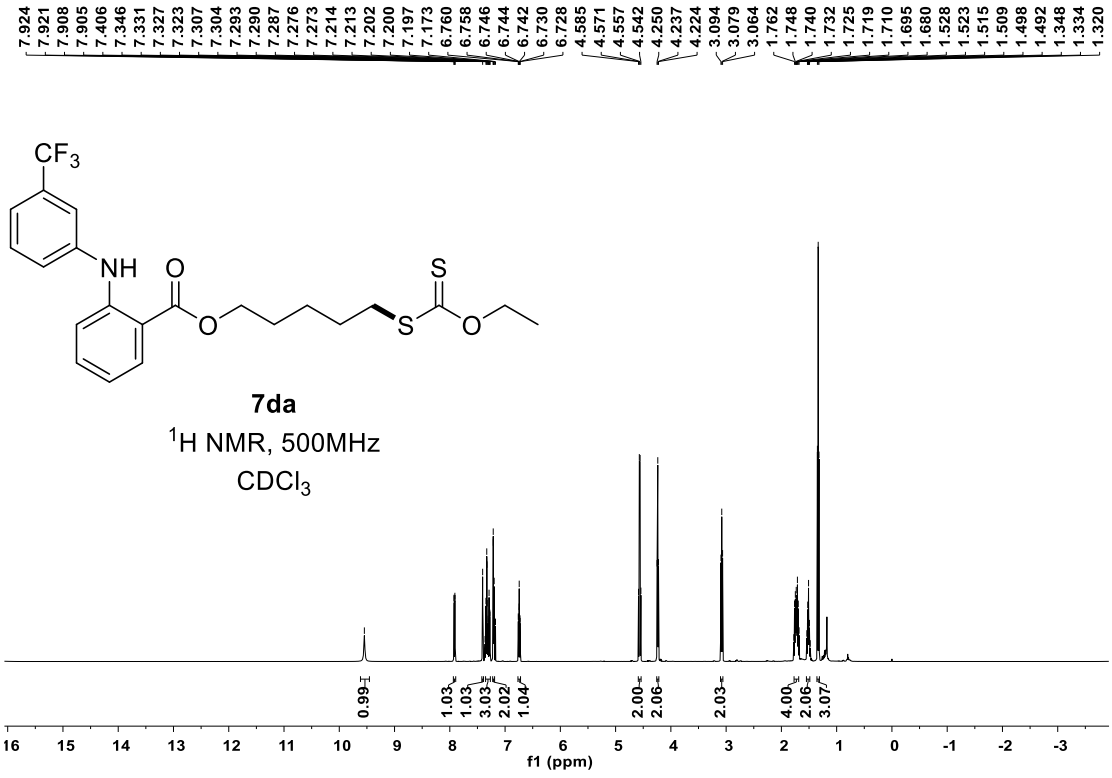
7.557
7.378
7.197
6.767
6.751
5.958
4.588
4.574
4.559
4.545
4.220
4.207
4.194
3.082
3.067
3.053
1.740
1.726
1.709
1.695
1.678
1.663
1.512
1.495
1.481
1.450
1.353
1.339
1.325
O
O
O
O
S
S
O
7ca
1H NMR, 400MHz
CDCl3
1.00
0.95
1.02
2.00
2.02
2.00
2.01
4.01
2.00
3.01
f1 (ppm)


214.89
165.85
151.47
147.62
125.18
124.28
109.38
107.86
101.70
77.25
77.00
76.75
69.76
64.52
35.56
28.21
28.03
25.29
13.72
O
S
O
O
O
S
O
7ca
13C NMR, 100MHz
CDCl3
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)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7.327
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7.307
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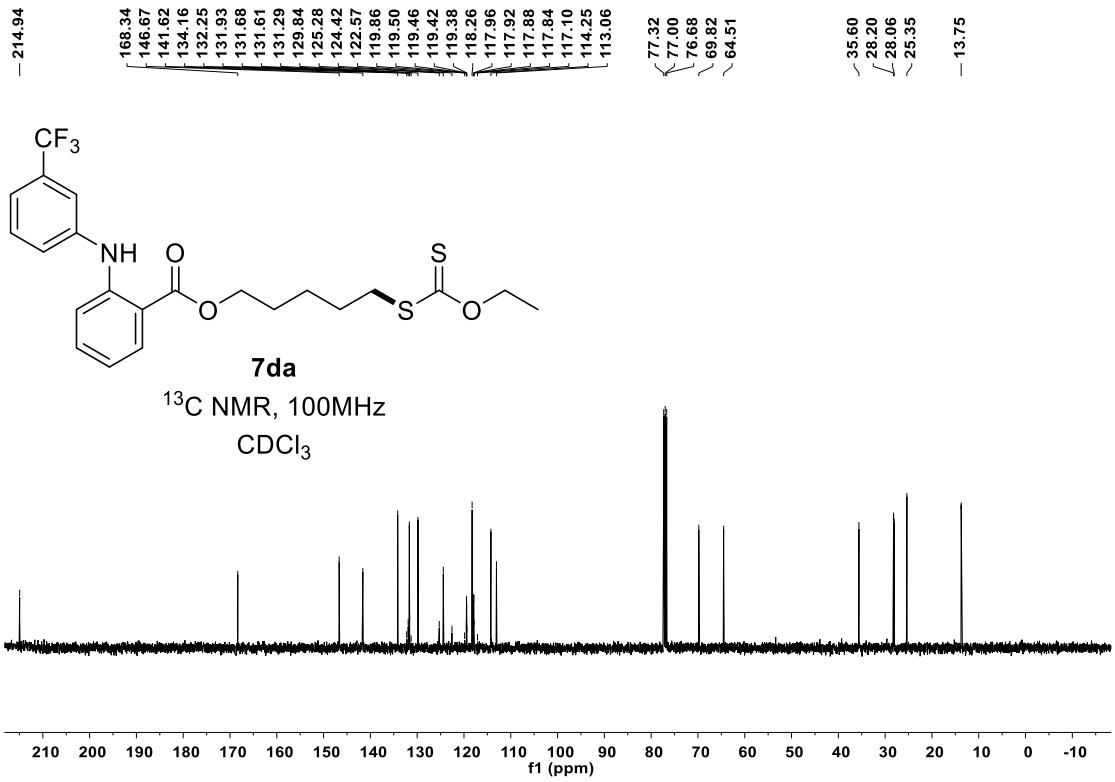
7.293
7.290
7.287
7.276
7.273
7.214
7.213
7.202
7.200
7.197
7.173
6.760
6.758
6.746
6.744
6.742
6.730
6.728
4.585
4.571
4.557
4.542
4.250
4.237
4.224
3.094
3.079
3.064
1.762
1.748
1.740
1.732
1.725
1.719
1.710
1.695
1.680
1.528
1.523
1.515
1.509
1.498
1.492
1.348
1.334
1.320
CF3
NH
O
O
S
S
O
7da
1H NMR, 500MHz
CDCl3
0.99
1.03
1.03
3.03
2.02
1.04
2.00
2.06
2.03
4.00
2.06
3.07
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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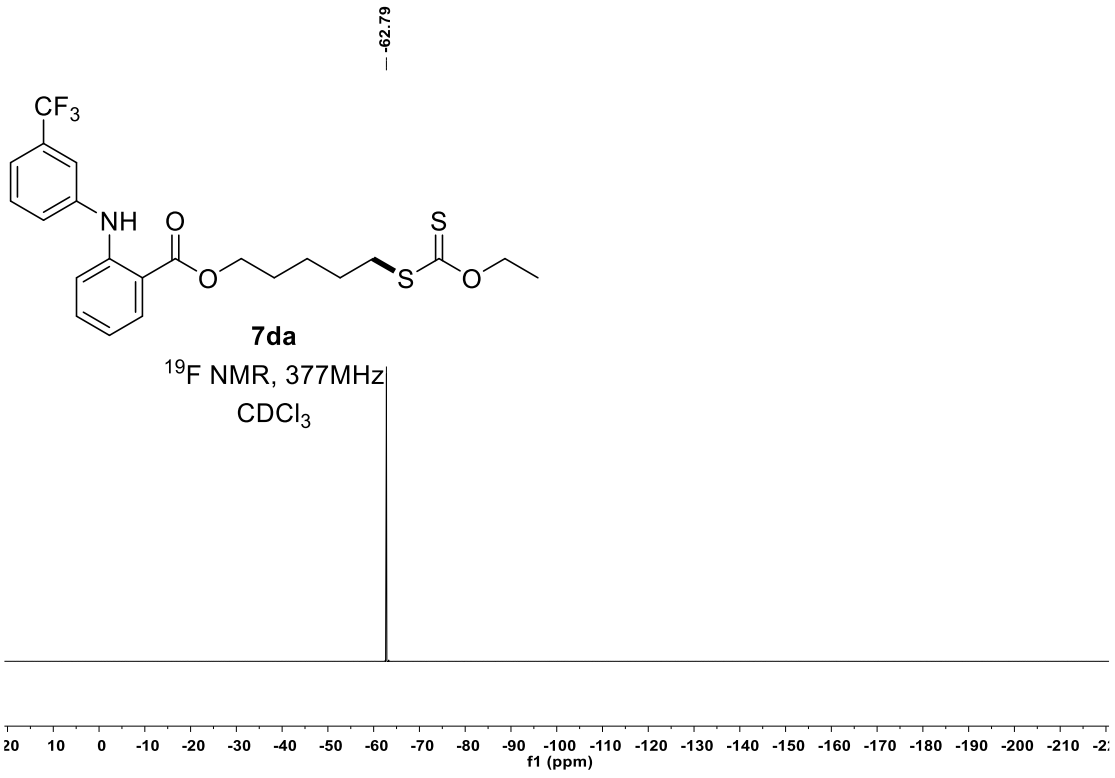
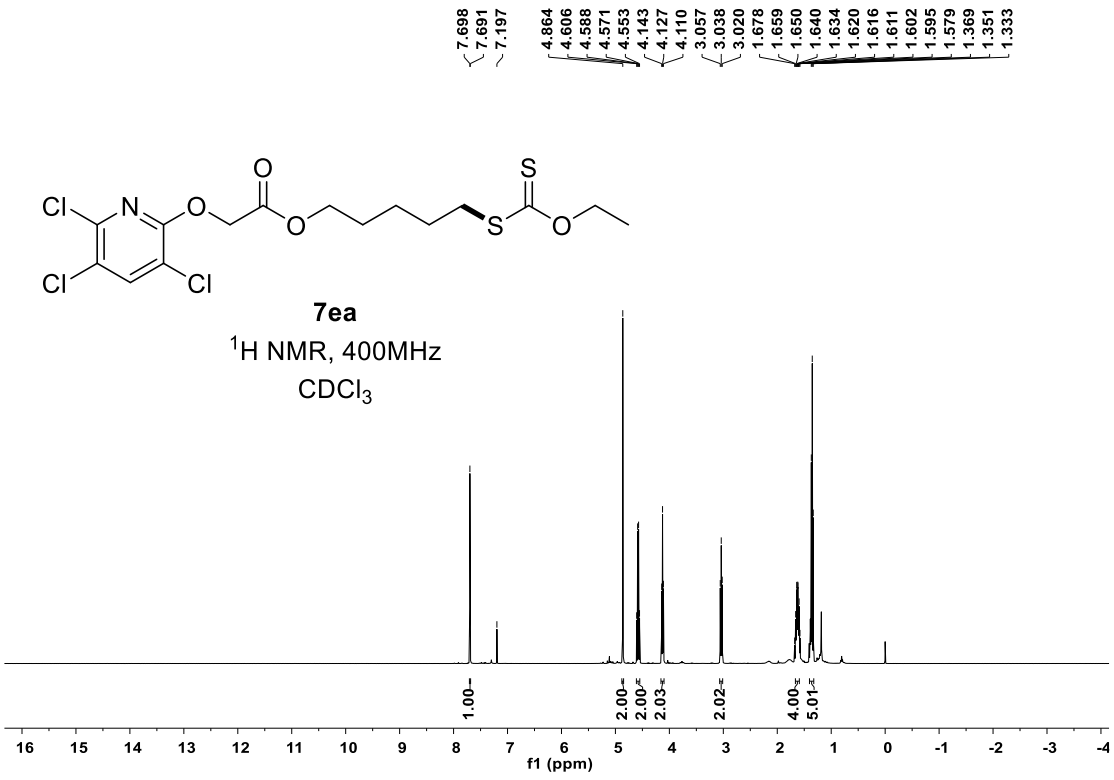
168.34
146.67
141.62
134.16
132.25
131.93
131.68
131.61
131.29
129.84
125.28
124.42
122.57
119.86
119.50
119.46
119.42
119.38
118.26
117.96
117.92
117.88
117.84
117.10
114.25
113.06
77.32
77.00
76.68
69.82
64.51
35.60
28.20
28.06
25.35
13.75
CF3
NH
O
O
S
S
O
7da
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-62.79
CF3
NH
O
O
S
S
O
7da
19F NMR, 377MHz
CDCl3
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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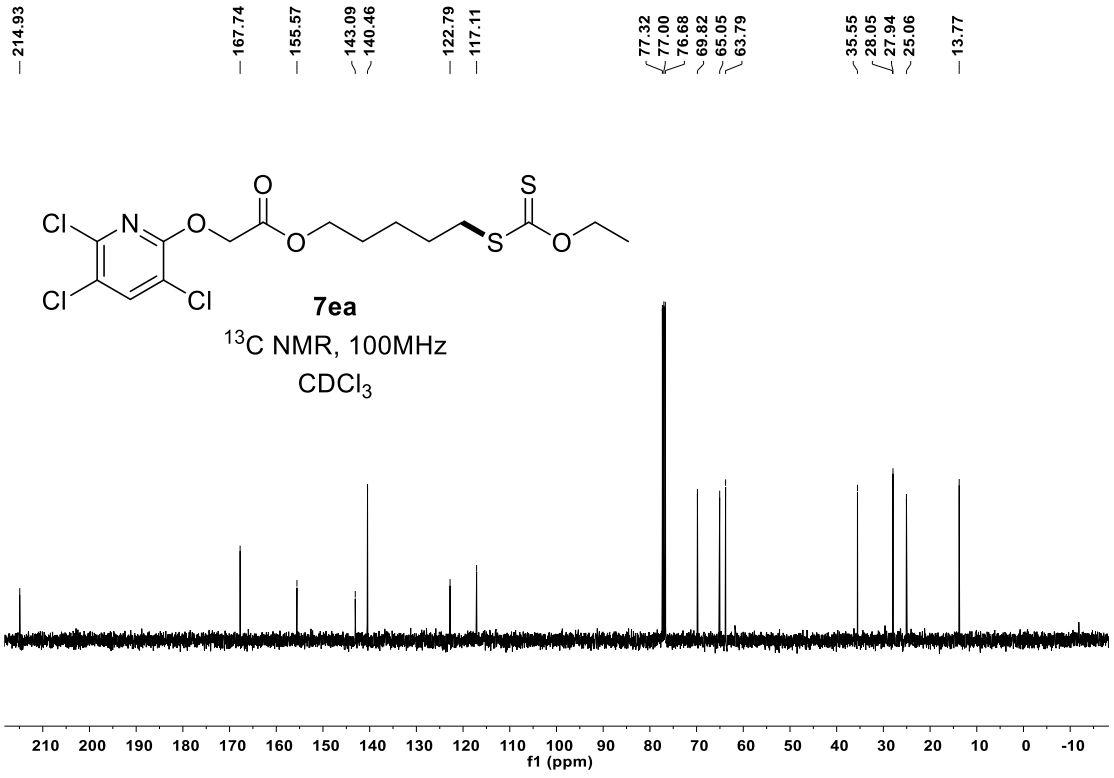
7.691
7.197
4.864
4.606
4.588
4.571
4.553
4.143
4.127
4.110
3.057
3.038
3.020
1.678
1.659
1.650
1.640
1.634
1.620
1.616
1.611
1.602
1.595
1.579
1.369
1.351
1.333
Cl
N
O
O
O
S
S
O
Cl
Cl
Cl
7ea
1H NMR, 400MHz
CDCl3
1.00
2.00
2.00
2.03
2.02
4.00
5.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)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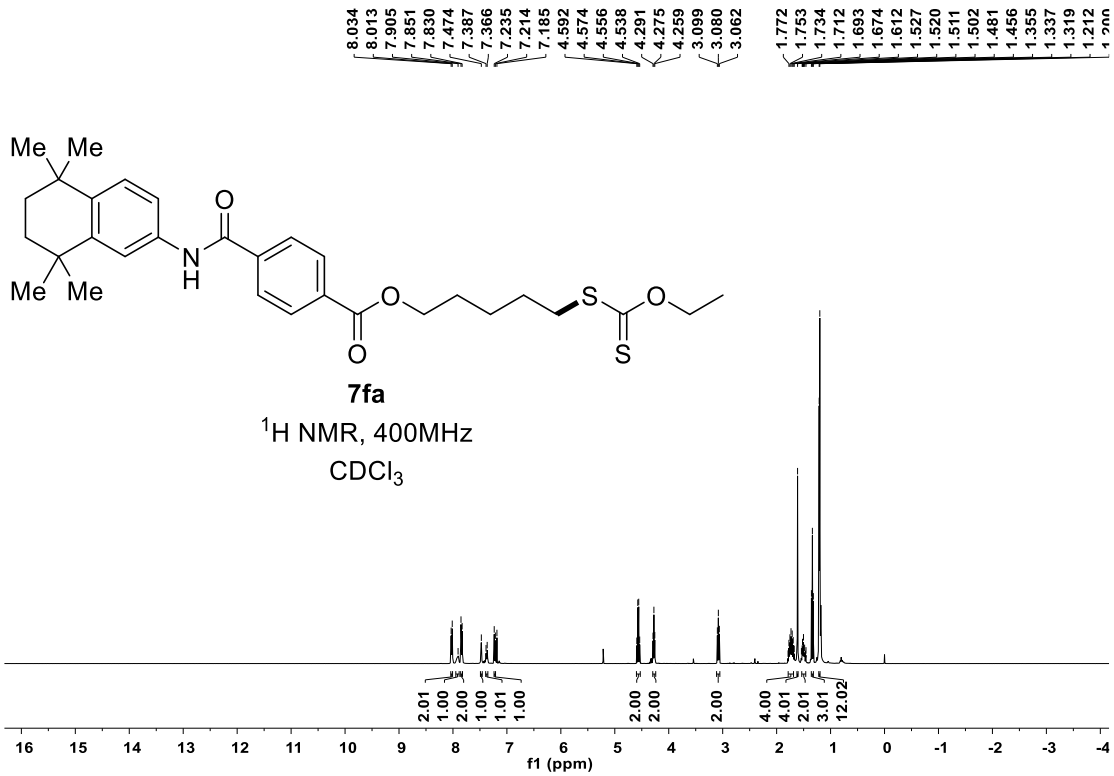
155.57
143.09
140.46
122.79
117.11
77.32
77.00
76.68
69.82
65.05
63.79
35.55
28.05
27.94
25.06
13.77
Cl
N
O
O
O
S
S
O
Cl
Cl
Cl
7ea
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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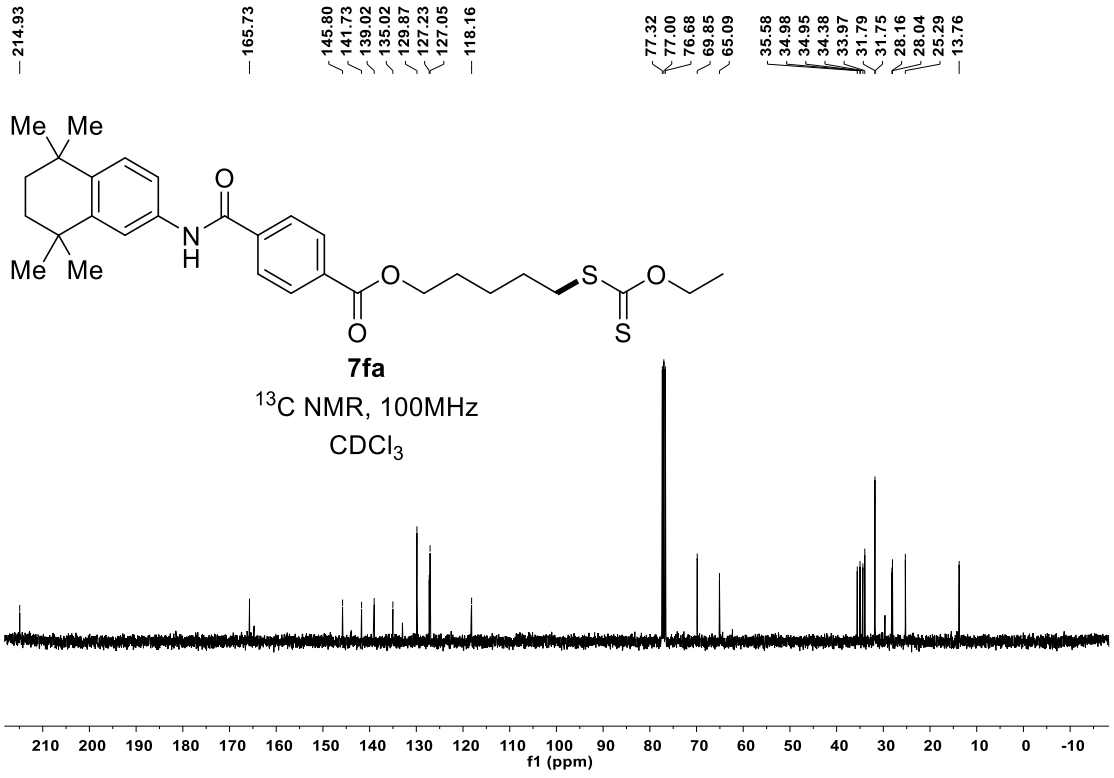
8.013
7.905
7.851
7.830
7.474
7.387
7.366
7.235
7.214
7.185
4.592
4.574
4.556
4.538
4.291
4.275
4.259
3.099
3.080
3.062
1.772
1.753
1.734
1.712
1.693
1.674
1.612
1.527
1.520
1.511
1.502
1.481
1.456
1.355
1.337
1.319
1.212
Me Me
Me Me
O
N
H
O
O
S
O
S
7fa
1H NMR, 400MHz
CDCl3
2.01
1.00
2.00
1.00
1.01
1.00
2.00
2.00
2.00
4.00
4.01
2.01
3.01
12.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)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141.73
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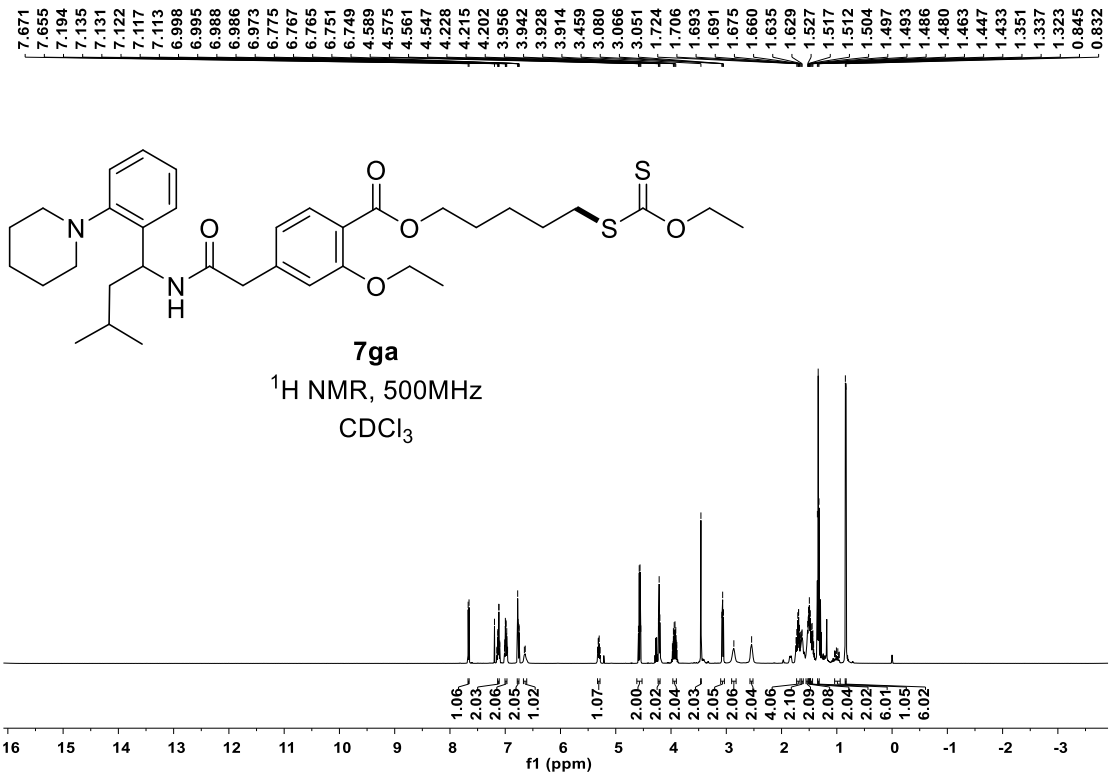
135.02
129.87
127.23
127.05
118.16
77.32
77.00
76.68
69.85
65.09
35.58
34.98
34.95
34.38
33.97
31.79
31.75
28.16
28.04
25.29
13.76
Me Me
Me Me
O
N
H
O
O
S
O
S
7fa
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.194
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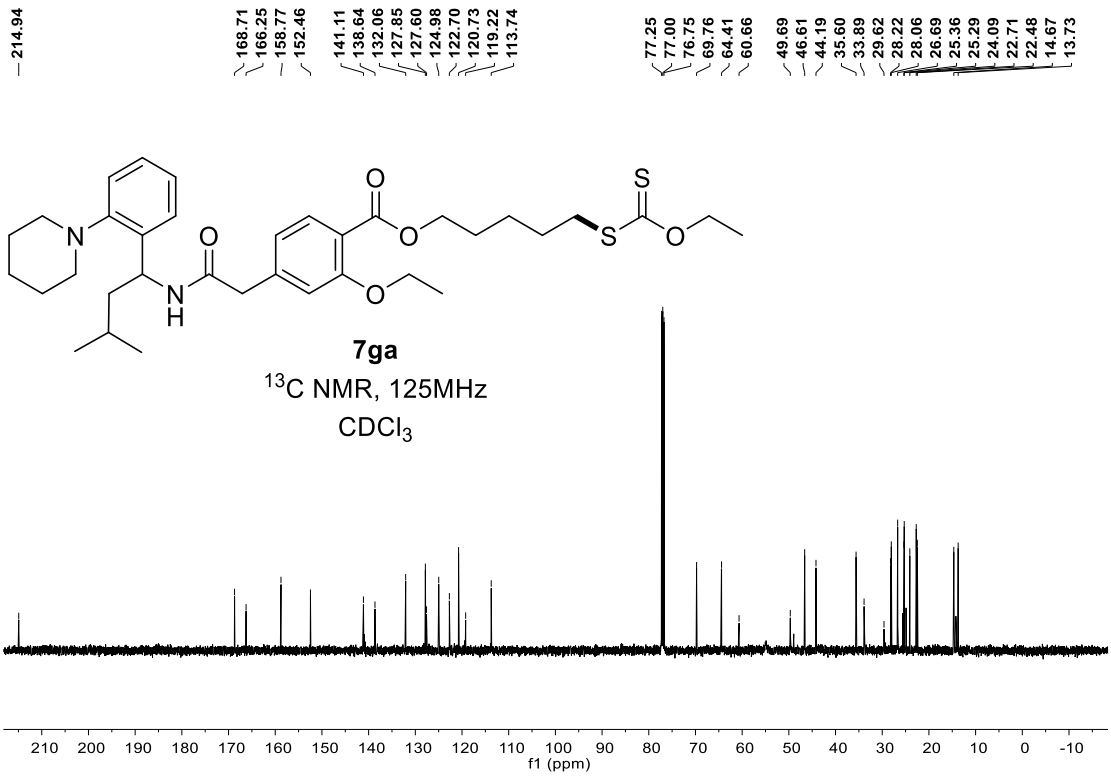
7.131
7.122
7.117
7.113
6.998
6.995
6.988
6.986
6.973
6.775
6.767
6.765
6.751
6.749
4.589
4.575
4.561
4.547
4.228
4.215
4.202
3.956
3.942
3.928
3.914
3.459
3.080
3.066
3.051
1.724
1.706
1.693
1.691
1.675
1.660
1.635
1.629
1.527
1.517
1.512
1.504
1.497
1.493
1.486
1.480
1.463
1.447
1.433
1.351
1.337
1.323
0.845
0.832
7ga
1H NMR, 500MHz
CDCl3
1.06
2.03
2.06
2.05
1.02
1.07
2.00
2.02
2.04
2.03
2.05
2.06
2.04
4.06
2.10
2.09
2.08
2.04
2.02
6.01
1.05
6.02
f1 (ppm)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168.71
166.25
158.77
152.46
141.11
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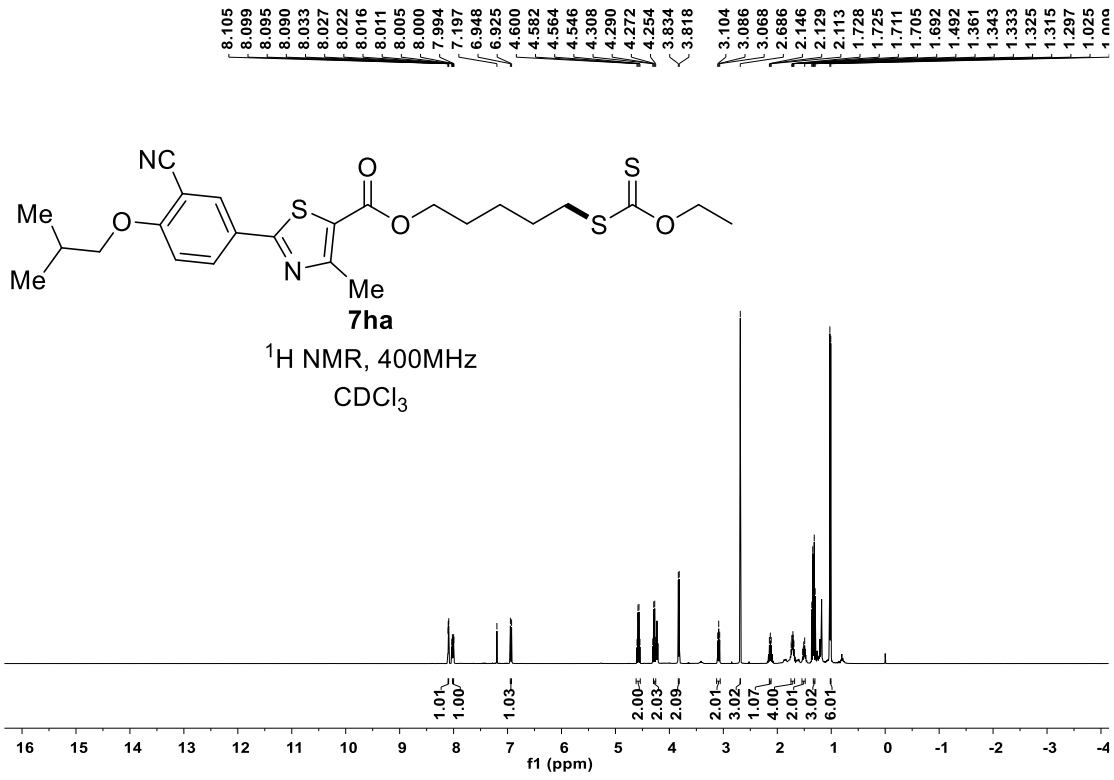
132.06
127.85
127.60
124.98
122.70
120.73
119.22
113.74
77.25
77.00
76.75
69.76
64.41
60.66
49.69
46.61
44.19
35.60
33.89
29.62
28.22
28.06
26.69
25.36
25.29
24.09
22.71
22.48
14.67
13.73
7ga
13C NMR, 125MHz
CDCl3
f1 (ppm)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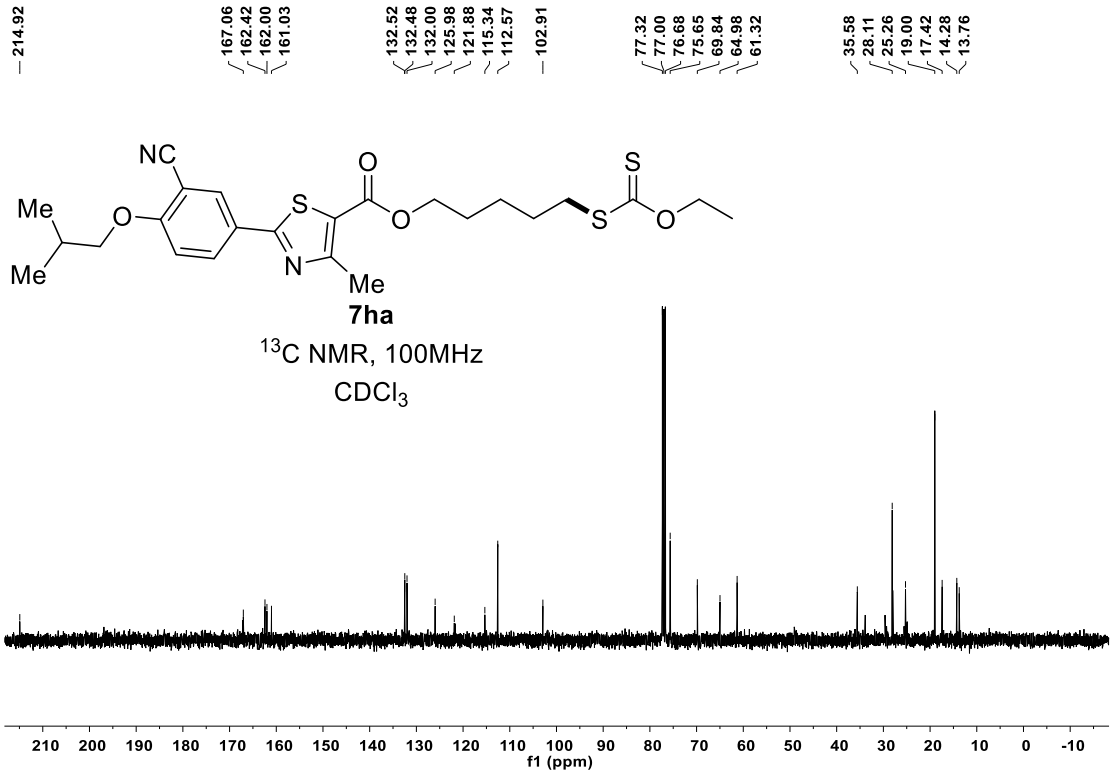
8.095
8.090
8.033
8.027
8.022
8.016
8.011
8.005
8.000
7.994
7.197
6.948
6.925
4.600
4.582
4.564
4.546
4.308
4.290
4.272
4.254
3.834
3.818
3.104
3.086
3.068
2.686
2.146
2.129
2.113
1.728
1.725
1.711
1.705
1.692
1.492
1.361
1.343
1.333
1.325
1.315
1.297
1.025
NC
O
S
Me
O
S
O
S
N
Me
Me
7ha
1H NMR, 400MHz
CDCl3
1.01
1.00
1.03
2.00
2.03
2.09
2.01
3.02
1.07
4.00
2.01
3.02
6.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)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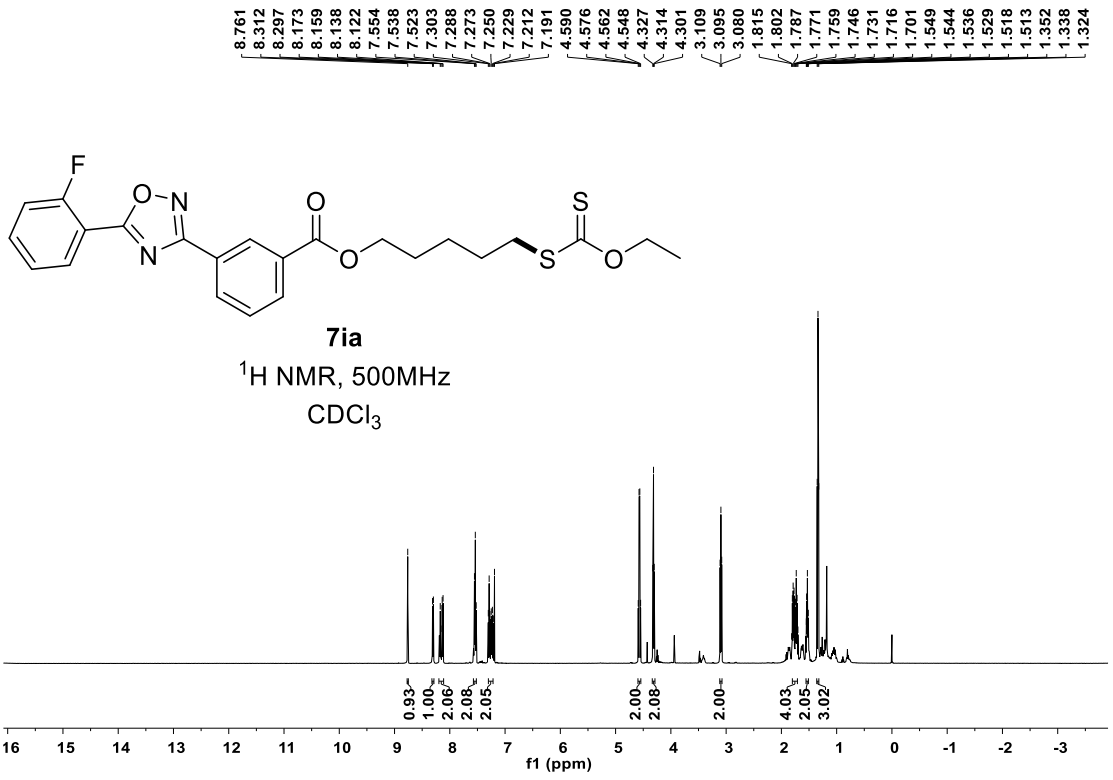
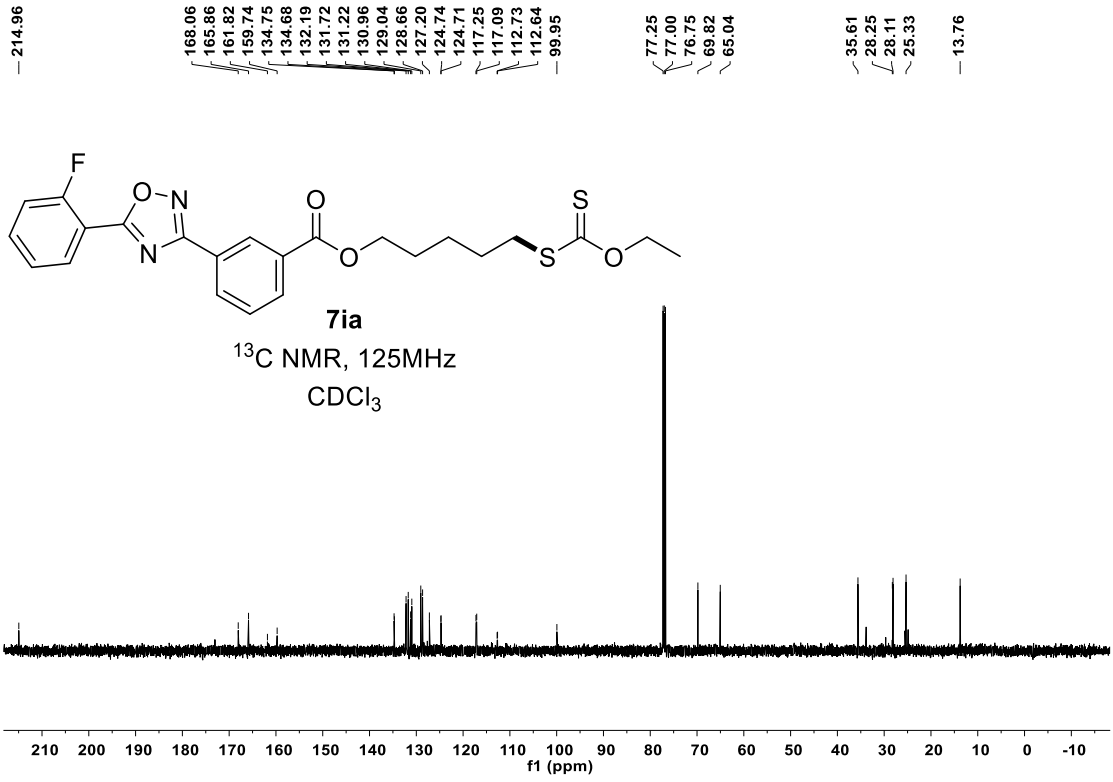
167.06
162.42
162.00
161.03
132.52
132.48
132.00
125.98
121.88
115.34
112.57
102.91
77.32
77.00
76.68
75.65
69.84
64.98
61.32
35.58
28.11
25.26
19.00
17.42
14.28
13.76
NC
O
S
Me
O
S
O
S
N
Me
Me
7ha
13C NMR, 100MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.761
8.312
8.297
8.173
8.159
8.138
8.122
7.554
7.538
7.523
7.303
7.288
7.273
7.250
7.229
7.212
7.191
4.590
4.576
4.562
4.548
4.327
4.314
4.301
3.109
3.095
3.080
1.815
1.802
1.787
1.771
1.759
1.746
1.731
1.716
1.701
1.549
1.544
1.536
1.529
1.518
1.513
1.352
1.338
1.324
F
O
N
N
O
O
S
S
O
7ia
1H NMR, 500MHz
CDCl3
0.93
1.00
2.06
2.08
2.05
2.00
2.08
2.00
4.03
2.05
3.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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165.86
161.82
159.74
134.75
134.68
132.19
131.72
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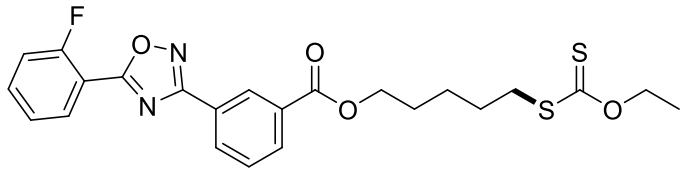
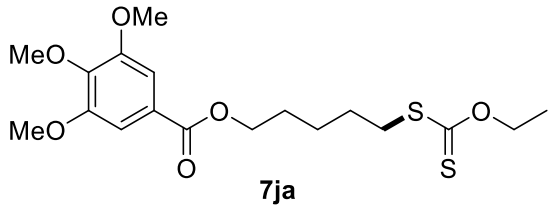
130.96
129.04
128.66
127.20
124.74
124.71
117.25
117.09
112.73
112.64
99.95
77.25
77.00
76.75
69.82
65.04
35.61
28.25
28.11
25.33
13.76
F
O
N
N
O
O
S
S
O
7ia
13C NMR, 125MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



7ia

^{19}F NMR, 471MHz

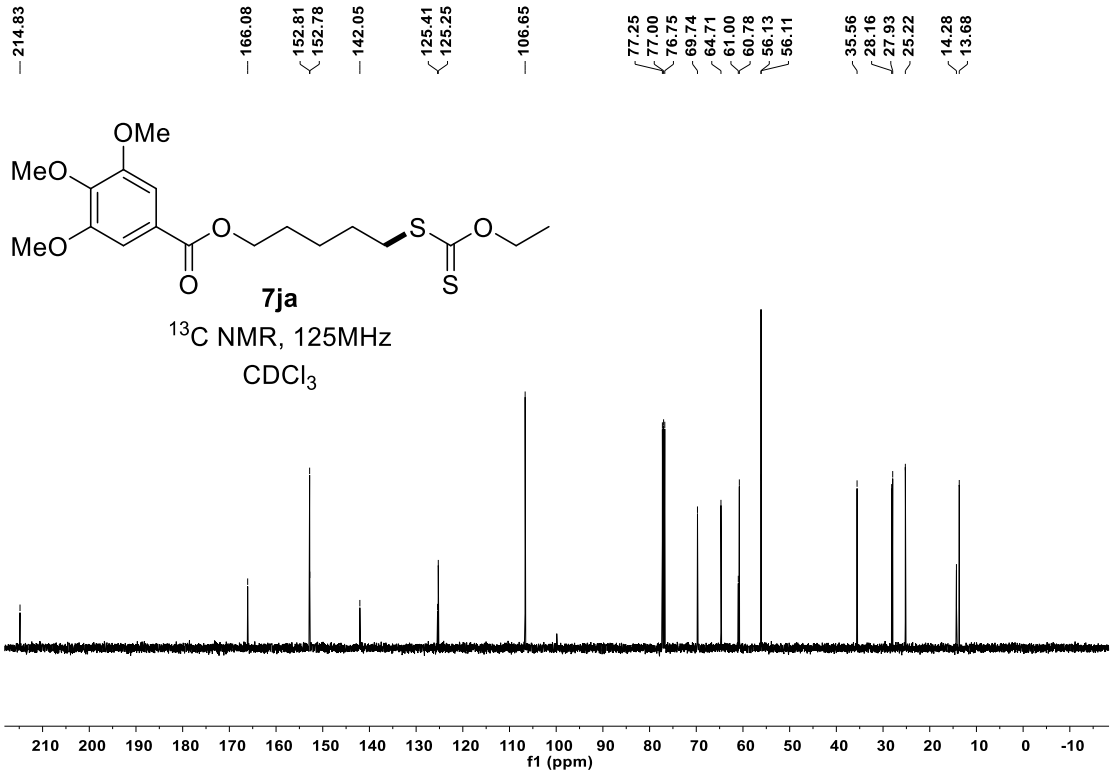
$CDCl_3$



7ja

^{1}H NMR, 500MHz

$CDCl_3$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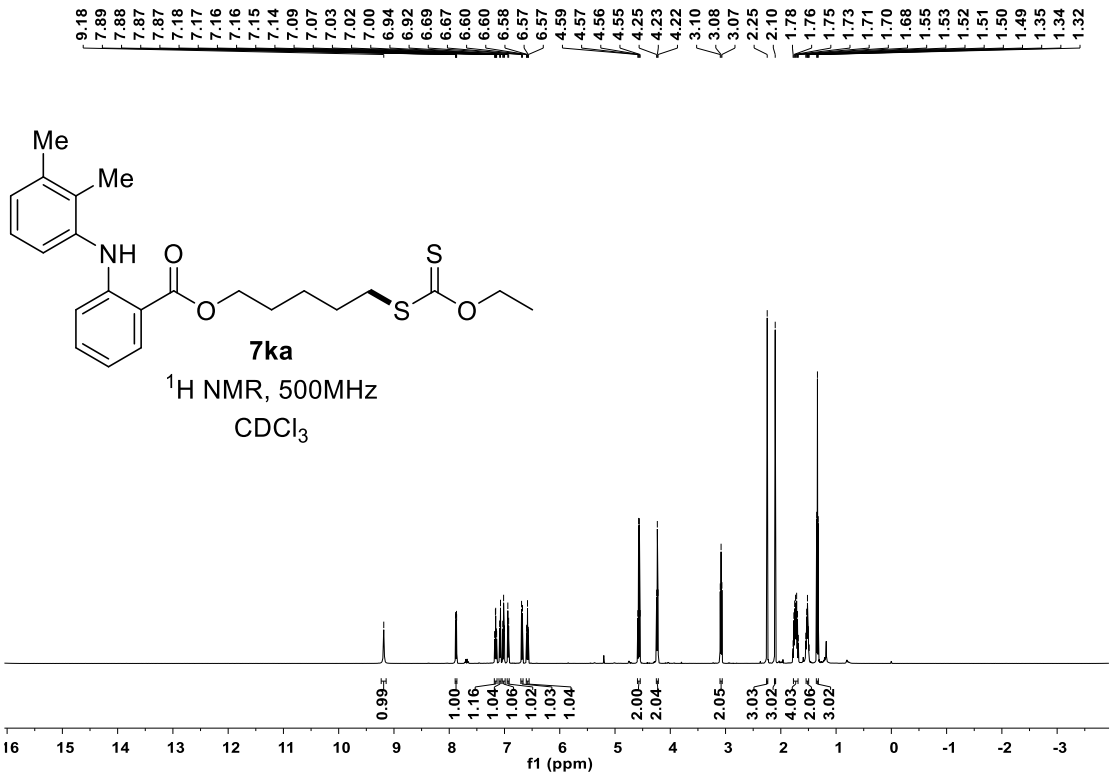
166.08
152.81
152.78
142.05
125.41
125.25
106.65
77.25
77.00
76.75
69.74
64.71
61.00
60.78
56.13
56.11
35.56
28.16
27.93
25.22
14.28
13.68
OMe
MeO
MeO
O
O
S
O
O
S
7ja
13C NMR, 125MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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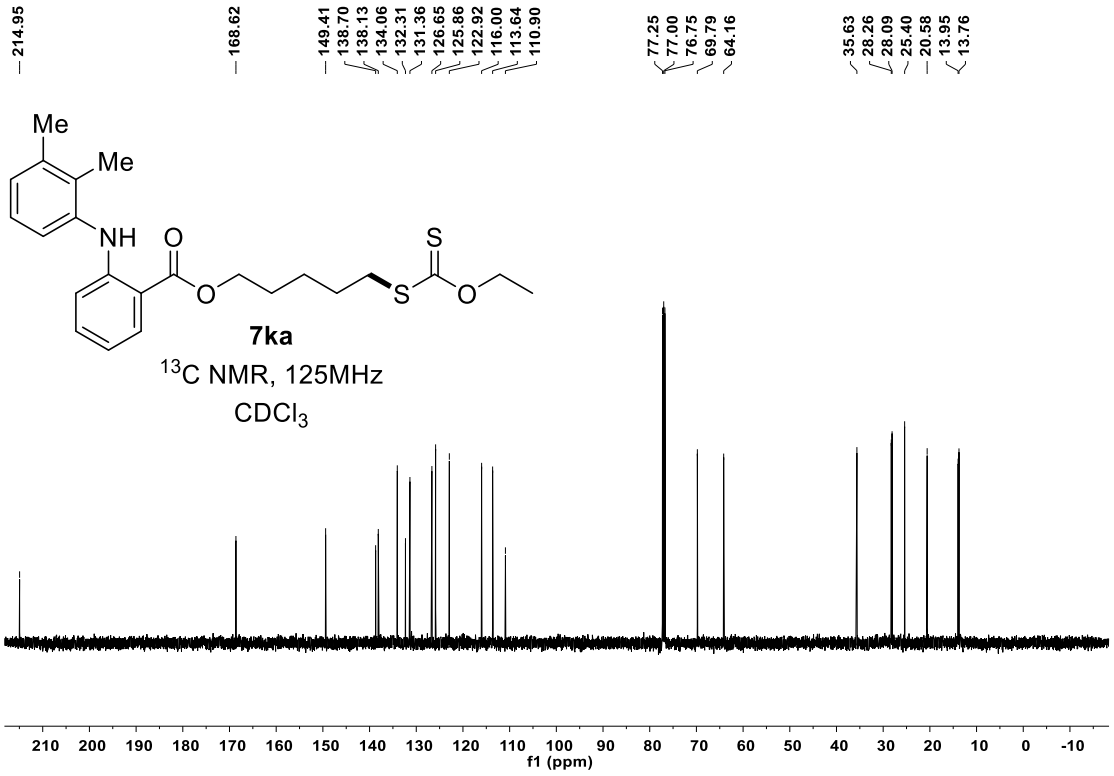
7.89
7.88
7.87
7.87
7.18
7.17
7.16
7.16
7.15
7.14
7.09
7.07
7.03
7.02
7.00
6.94
6.92
6.69
6.67
6.60
6.60
6.58
6.57
6.57
4.59
4.57
4.56
4.55
4.25
4.23
4.22
3.10
3.08
3.07
2.25
2.10
1.78
1.76
1.75
1.73
1.71
1.70
1.68
1.55
1.53
1.52
1.51
1.50
1.49
1.35
1.34
1.32
Me
Me
NH
O
O
S
S
O
7ka
1H NMR, 500MHz
CDCl3
0.99
1.00
1.16
1.04
1.06
1.02
1.03
1.04
2.00
2.04
2.05
3.03
3.02
4.03
2.06
3.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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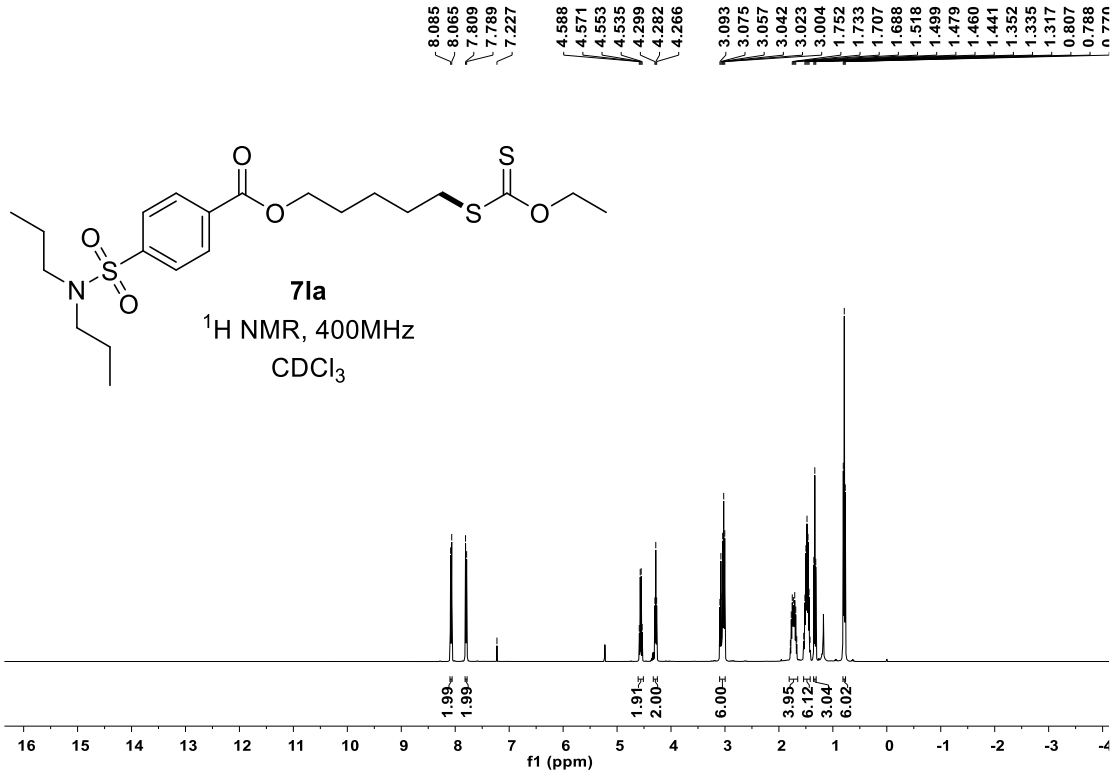
149.41
138.70
138.13
134.06
132.31
131.36
126.65
125.86
122.92
116.00
113.64
110.90
77.25
77.00
76.75
69.79
64.16
35.63
28.26
28.09
25.40
20.58
13.95
13.76
Me
Me
NH
O
O
S
S
O
7ka
13C NMR, 125MHz
CDCl3
f1 (ppm)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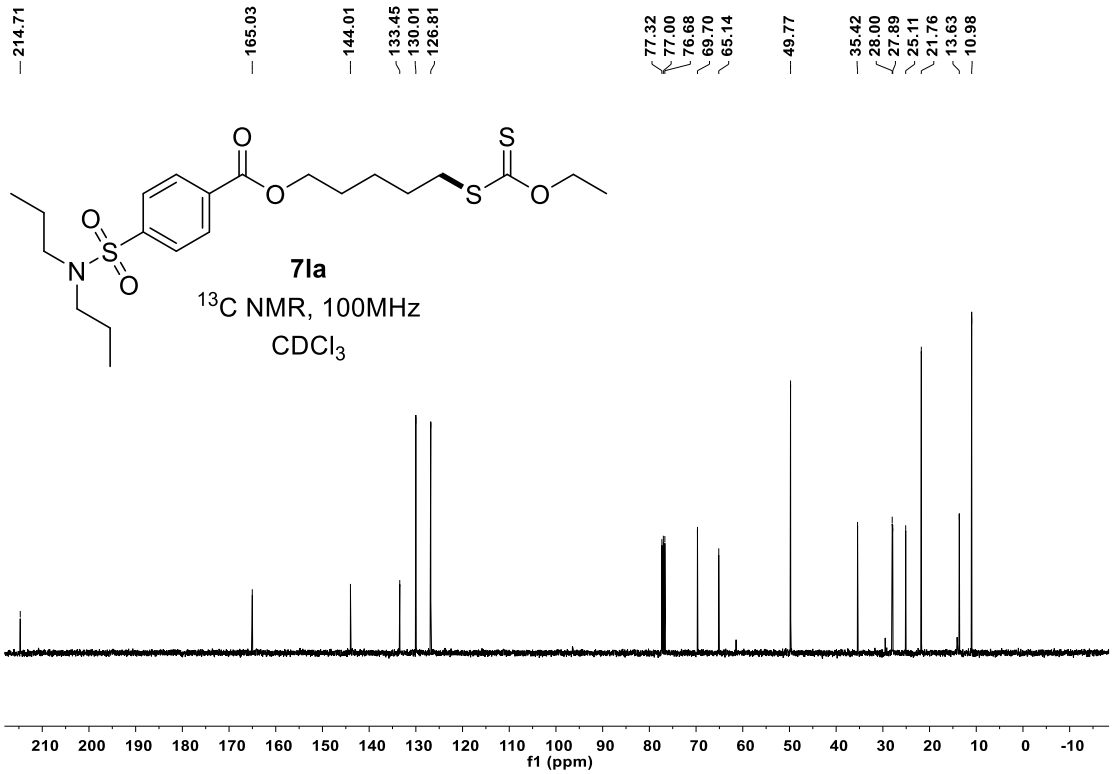
8.065
7.809
7.789
7.227
4.588
4.571
4.553
4.535
4.299
4.282
4.266
3.093
3.075
3.057
3.042
3.023
3.004
1.752
1.733
1.707
1.688
1.518
1.499
1.479
1.460
1.441
1.352
1.335
1.317
0.807
0.788
O
O
S
O
S
O
O
N
S
O
7la
1H NMR, 400MHz
CDCl3
1.99
1.99
1.91
2.00
6.00
3.95
6.12
3.04
6.02
f1 (ppm)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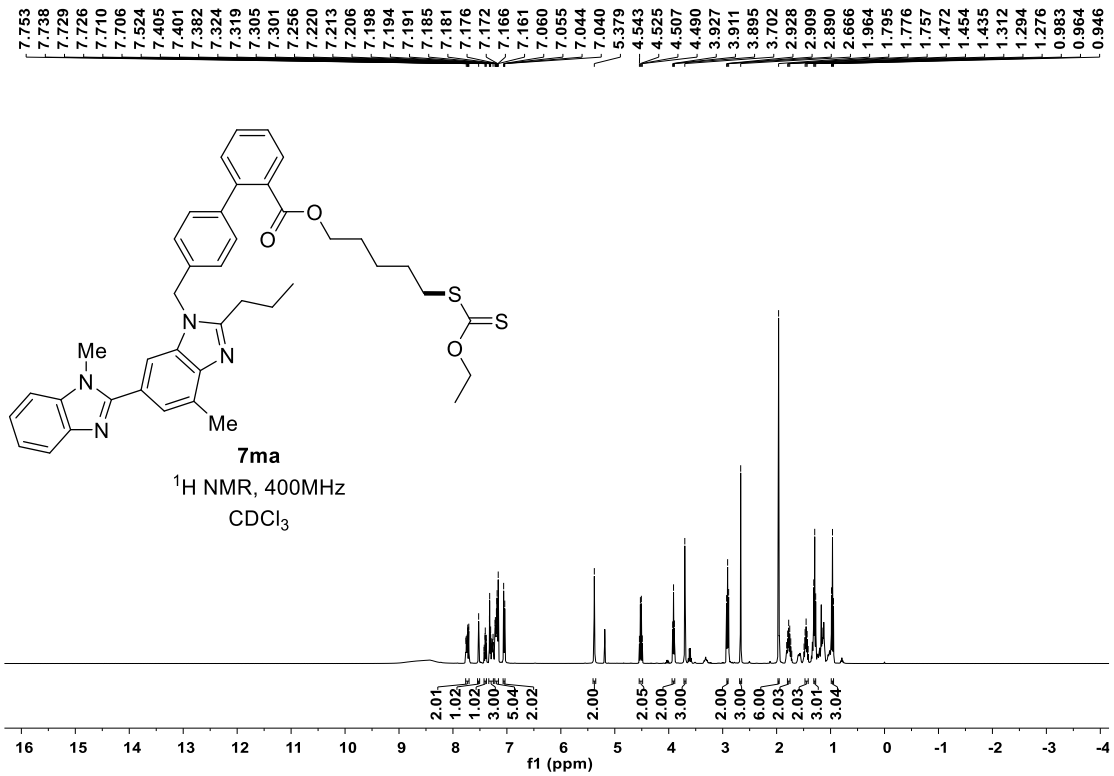
144.01
133.45
130.01
126.81
77.32
77.00
76.68
69.70
65.14
49.77
35.42
28.00
27.89
25.11
21.76
13.63
10.98
7Ia
13C NMR, 100MHz
CDCl3
f1 (ppm)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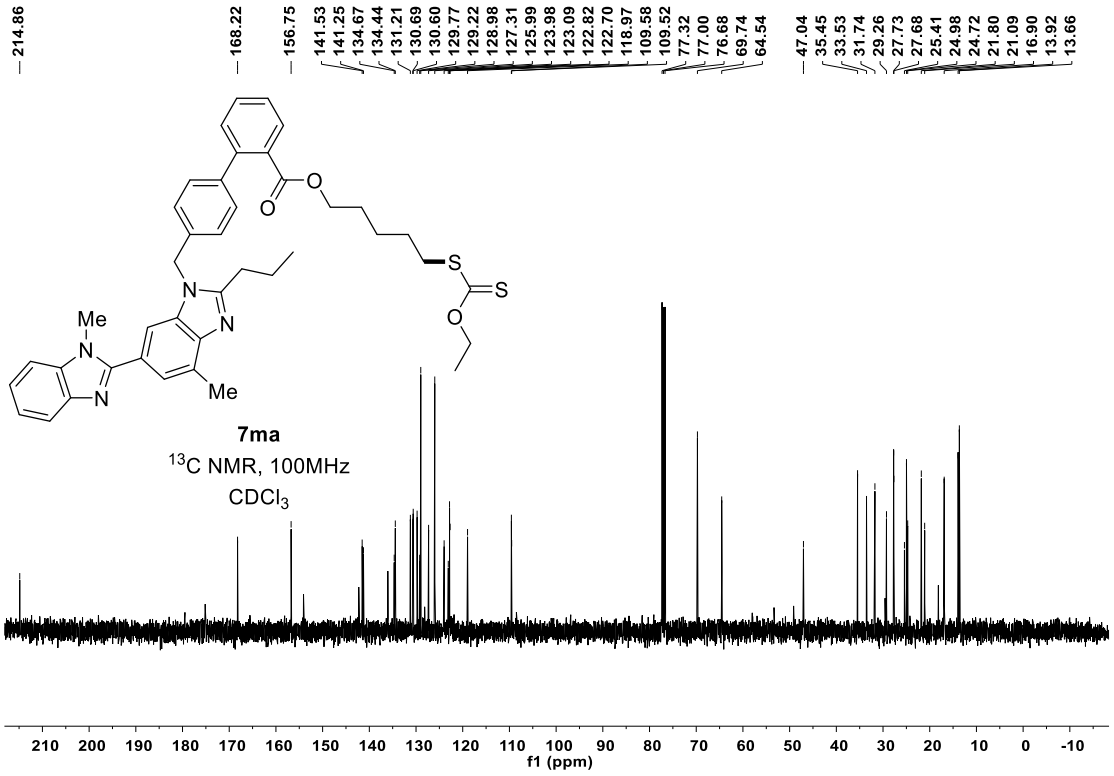
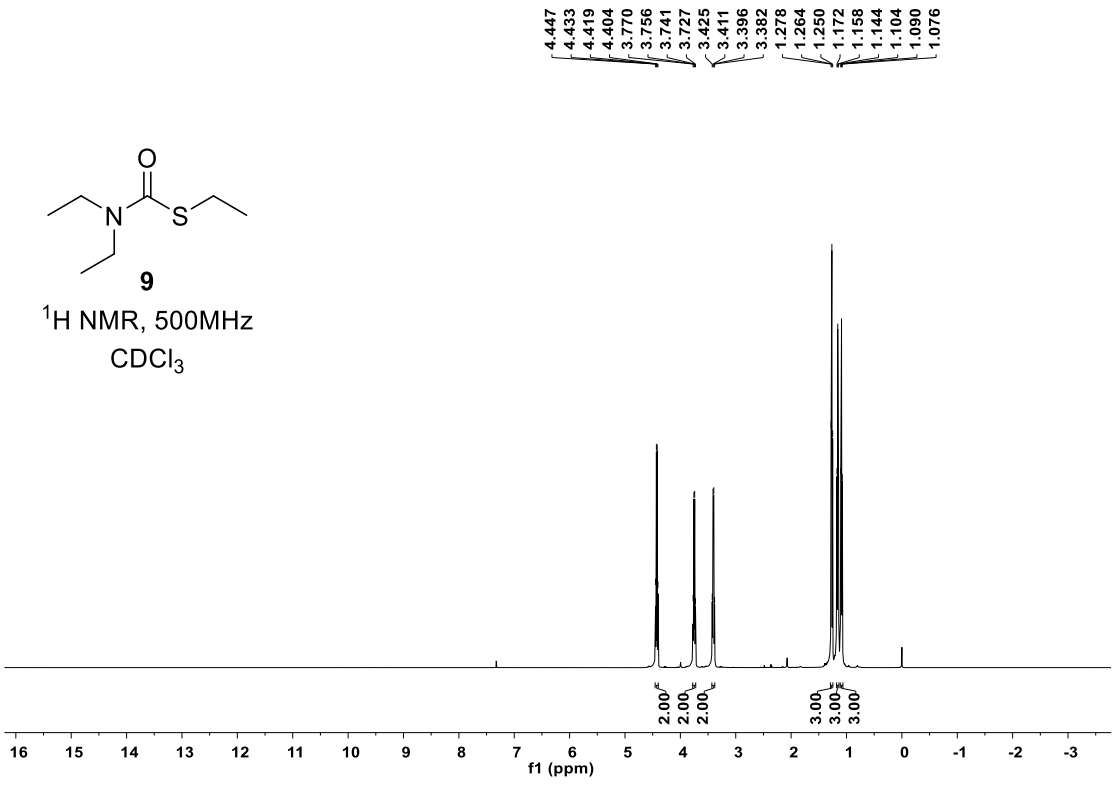
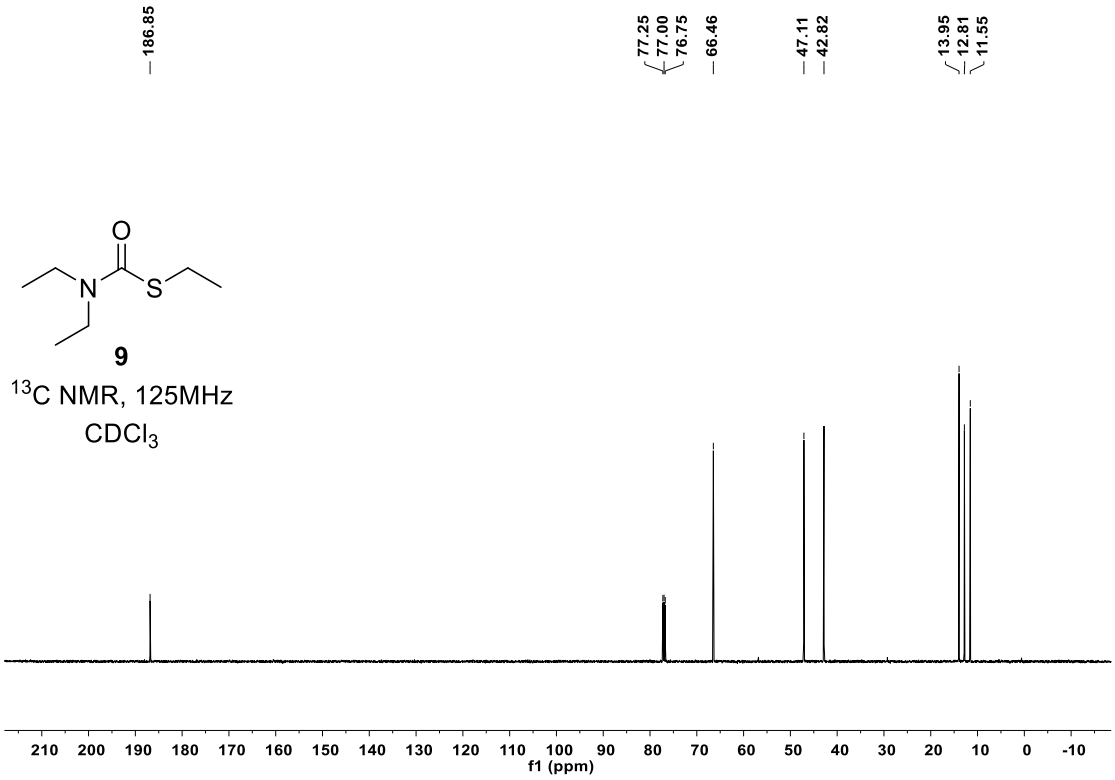
7.729
7.726
7.710
7.706
7.524
7.405
7.401
7.382
7.324
7.319
7.305
7.301
7.256
7.220
7.213
7.206
7.198
7.194
7.191
7.185
7.181
7.176
7.172
7.166
7.161
7.060
7.055
7.044
7.040
5.379
4.543
4.525
4.507
4.490
3.927
3.911
3.895
3.702
2.928
2.909
2.890
2.666
1.964
1.795
1.776
1.757
1.472
1.454
1.435
1.312
1.294
1.276
0.983
0.964
0.946
7ma
1H NMR, 400MHz
CDCl3
f1 (ppm)


214.86
168.22
156.75
141.53
141.25
134.67
134.44
131.21
130.69
130.60
129.77
129.22
128.98
127.31
125.99
123.98
123.09
122.82
122.70
118.97
109.58
109.52
77.32
77.00
76.68
69.74
64.54
47.04
35.45
33.53
31.74
29.26
27.73
27.68
25.41
24.98
24.72
21.80
21.09
16.90
13.92
13.66
Me
Me
7ma
13C NMR, 100MHz
CDCl3
f1 (ppm)

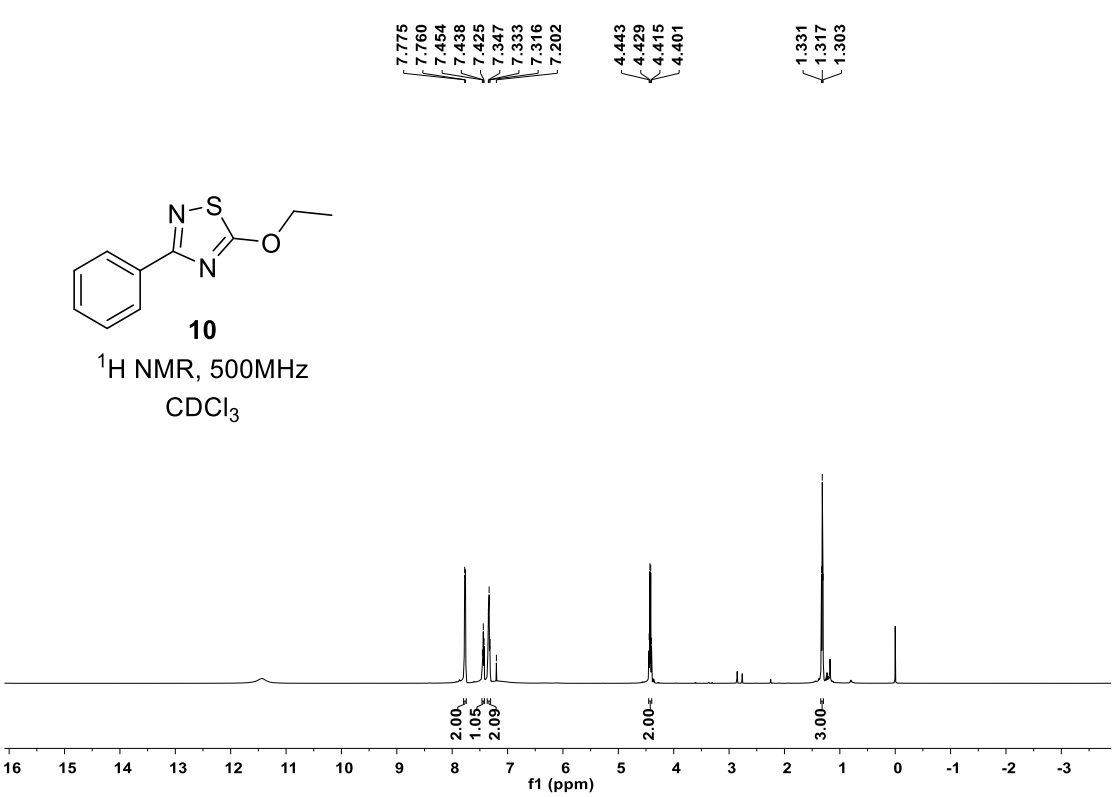

4.447
4.433
4.419
4.404
3.770
3.756
3.741
3.727
3.425
3.411
3.396
3.382
1.278
1.264
1.250
1.172
1.158
1.144
1.104
1.090
1.076
9
1H NMR, 500MHz
CDCl3
2.00
2.00
2.00
3.00
3.00
3.00
f1 (ppm)



9

^{13}C NMR, 125MHz

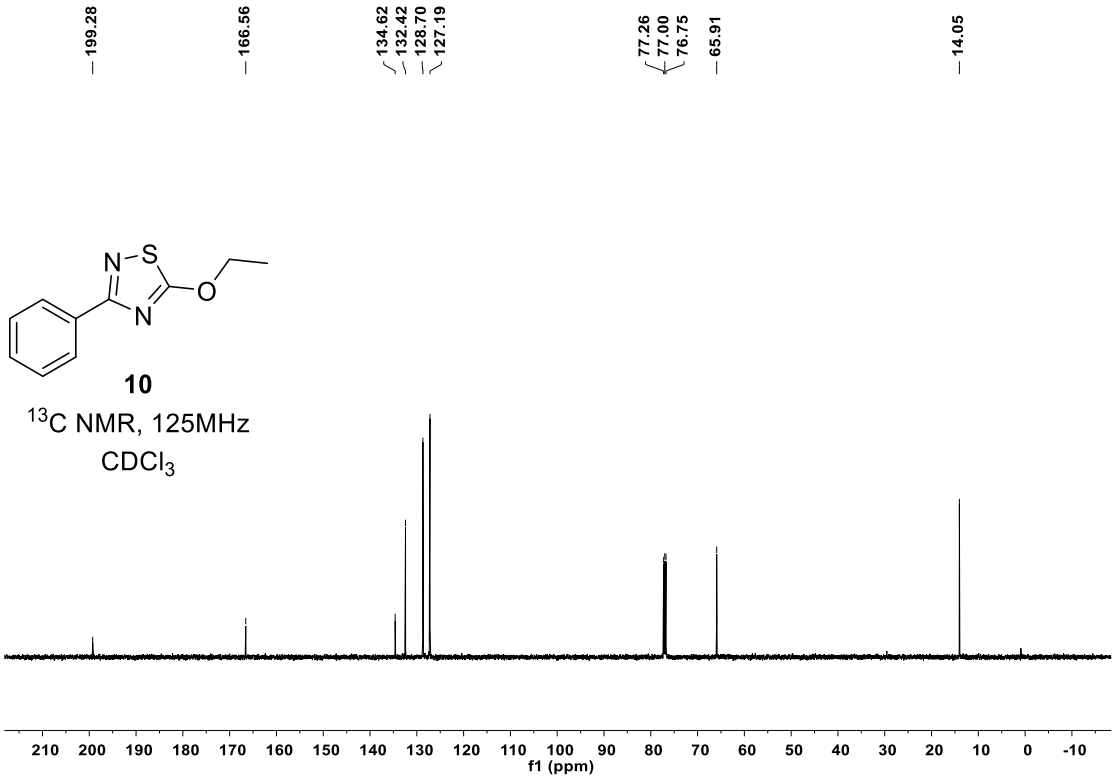
$CDCl_3$



10

^{1}H NMR, 500MHz

$CDCl_3$


199.28
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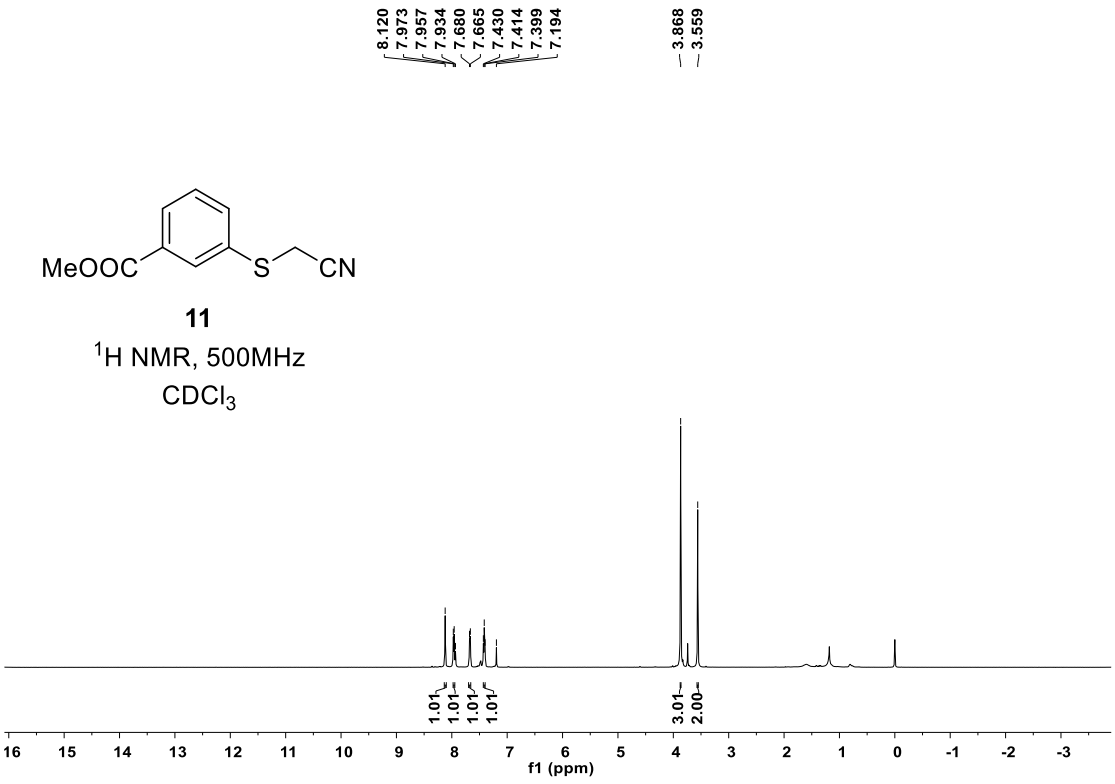
134.62
132.42
128.70
127.19
77.26
77.00
76.75
65.91
14.05
N
S
O
N
10
13C NMR, 125MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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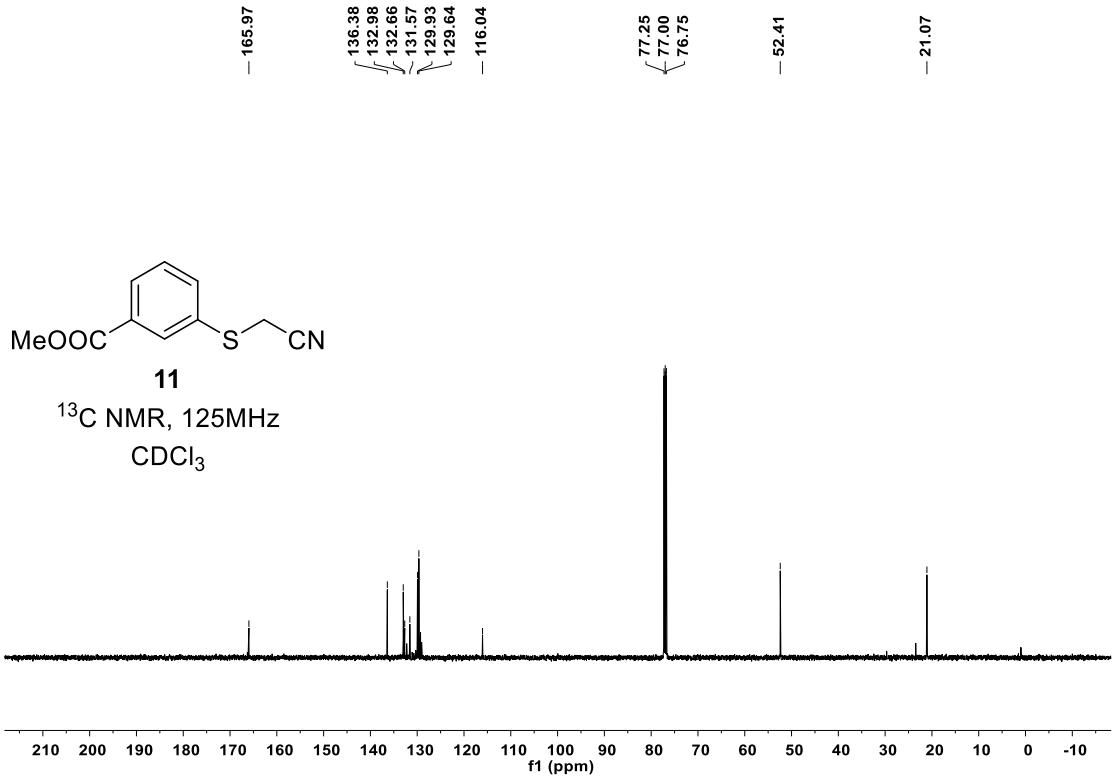
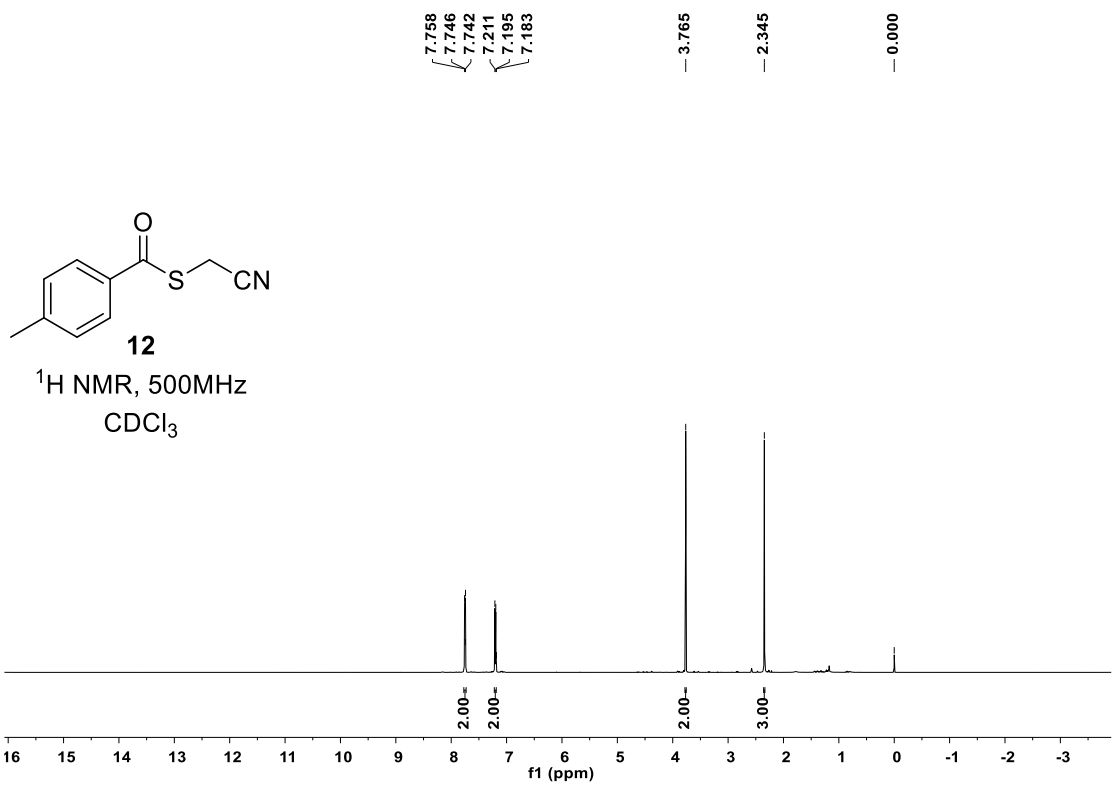
7.957
7.934
7.680
7.665
7.430
7.414
7.399
7.194
3.868
3.559
MeOOC
S
CN
11
1H NMR, 500MHz
CDCl3
1.01
1.01
1.01
1.01
3.01
2.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


165.97
136.38
132.98
132.66
131.57
129.93
129.64
116.04
77.25
77.00
76.75
52.41
21.07
MeOOC
S
CN
11
13C NMR, 125MHz
CDCl3
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)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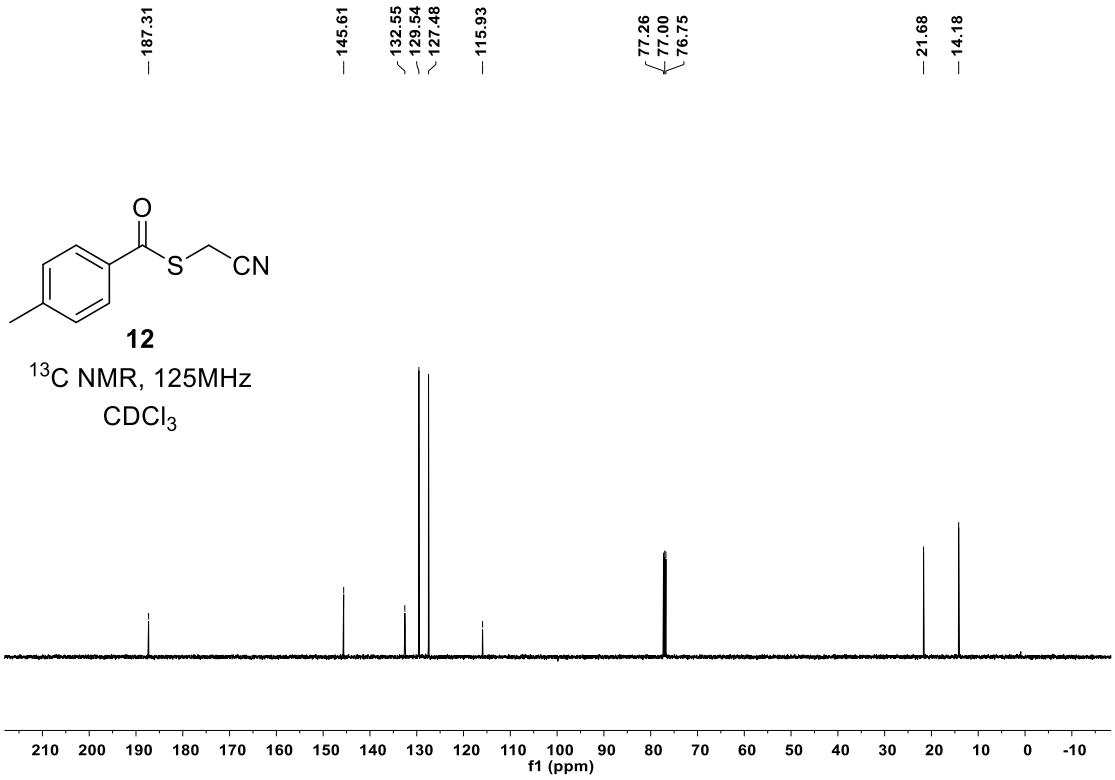
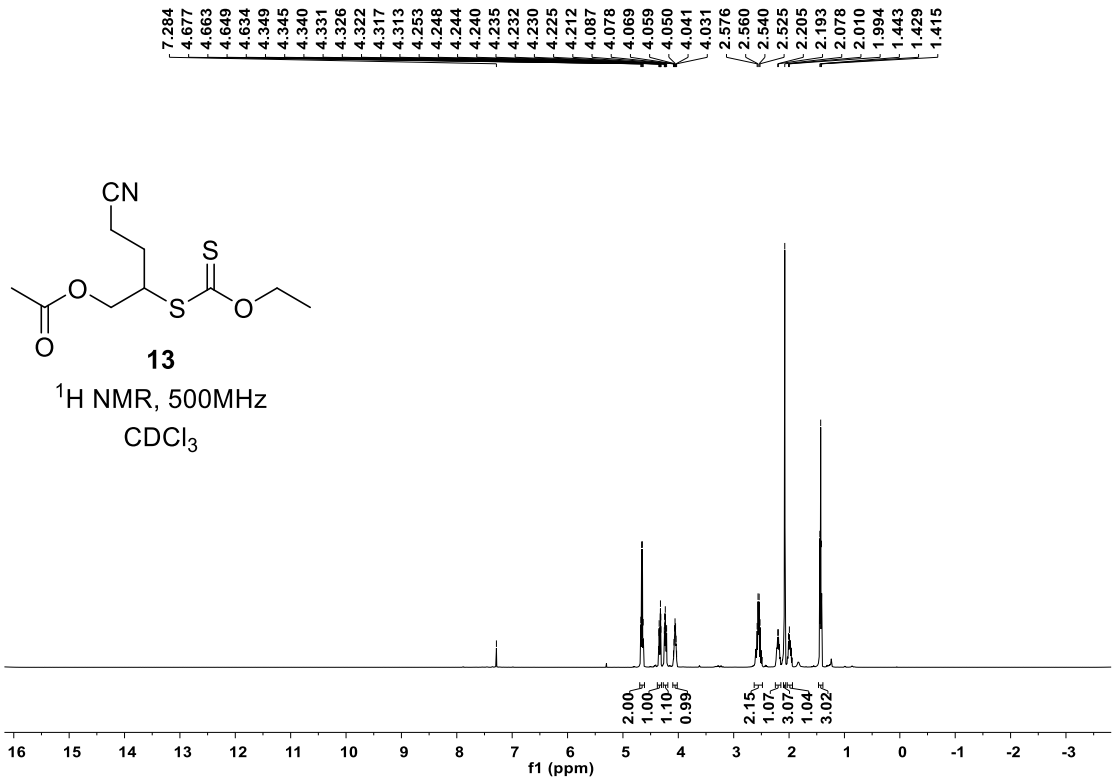
7.746
7.742
7.211
7.195
7.183
3.765
2.345
0.000
O
S
CN
12
1H NMR, 500MHz
CDCl3
2.00
2.00
2.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


187.31
145.61
132.55
129.54
127.48
115.93
77.26
77.00
76.75
21.68
14.18
O
S
CN
12
13C NMR, 125MHz
CDCl3
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)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4.649
4.634
4.349
4.345
4.340
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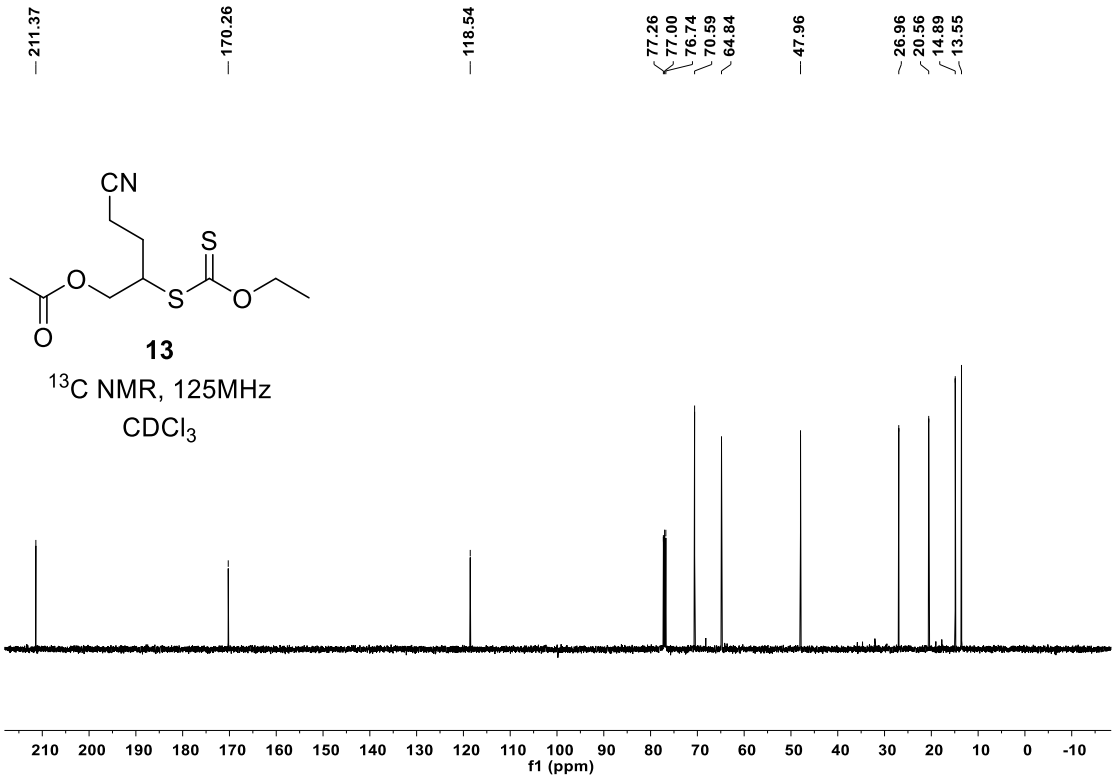
4.326
4.322
4.317
4.313
4.253
4.248
4.244
4.240
4.235
4.232
4.230
4.225
4.212
4.087
4.078
4.069
4.059
4.050
4.041
4.031
2.576
2.560
2.540
2.525
2.205
2.193
2.078
2.010
1.994
1.443
1.429
1.415
CN
S
O
O
S
O
O
13
1H NMR, 500MHz
CDCl3
2.00
1.00
1.10
0.99
2.15
1.07
3.07
1.04
3.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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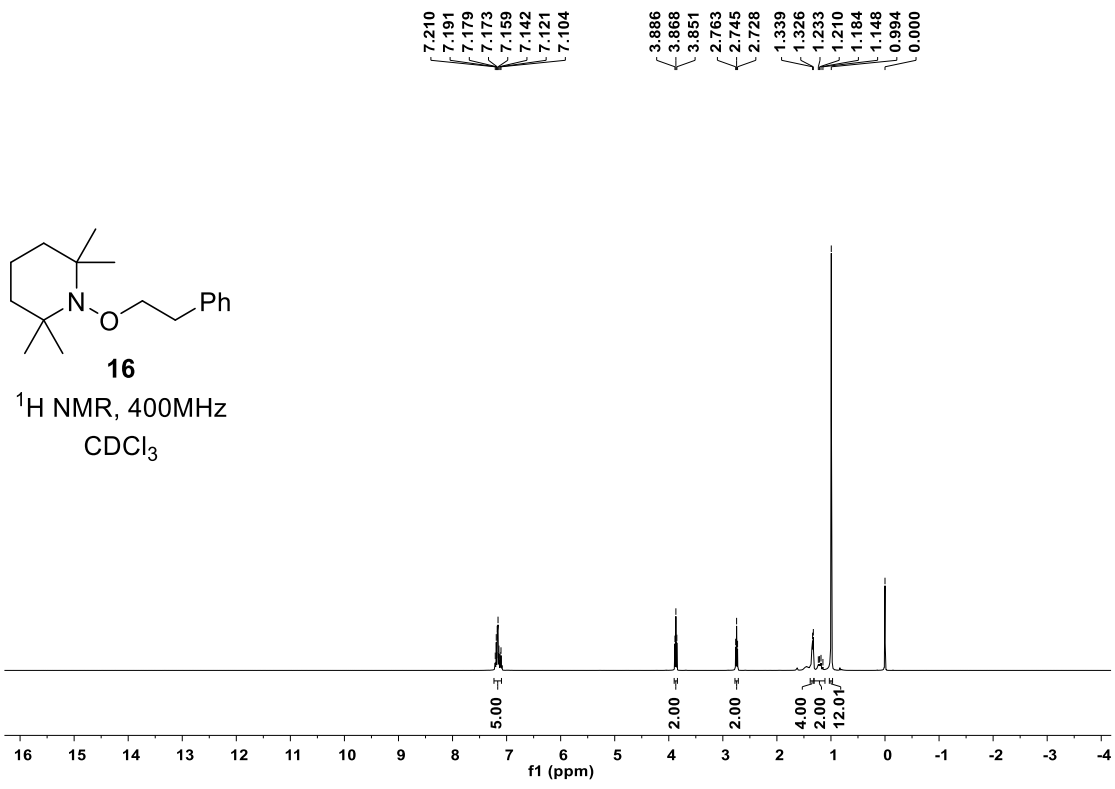
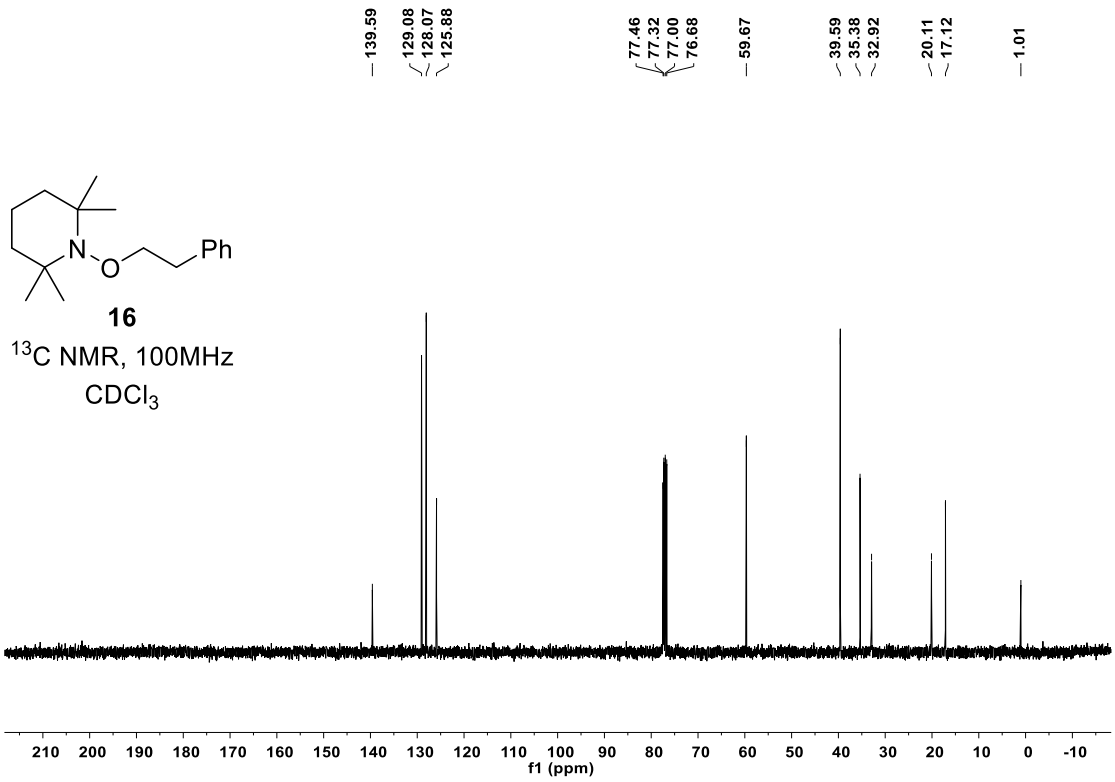
170.26
118.54
77.26
77.00
76.74
70.59
64.84
47.96
26.96
20.56
14.89
13.55
CN
S
O
S
O
O
13
13C NMR, 125MHz
CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.210
7.191
7.179
7.173
7.159
7.142
7.121
7.104
3.886
3.868
3.851
2.763
2.745
2.728
1.339
1.326
1.233
1.210
1.184
1.148
0.994
0.000
N
O
Ph
16
1H NMR, 400MHz
CDCl3
5.00
2.00
2.00
4.00
2.00
12.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


139.59
129.08
128.07
125.88
77.46
77.32
77.00
76.68
59.67
39.59
35.38
32.92
20.11
17.12
1.01
N
O
Ph
16
13C NMR, 100MHz
CDCl3
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)