Supporting Information

Photoinduced C(sp^3)–H Chalcogenation of Amides Derivatives and Ethers via Ligand-to-Metal Charge-Transfer.

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General Considerations

All reagents and solvents were purchased and used without further purification unless otherwise noted. All reactions were performed under an inert atmosphere unless otherwise stated. Room temperature refers to 26 °C, unless otherwise noted. Moisture-sensitive reactions were performed using flame-dried glassware under an atmosphere of dry argon (Ar). Air- and water sensitive reactions were setup in a Vacuum Atmosphere GENESIS glovebox held under an atmosphere of argon gas (working pressure 2−6 mbar).

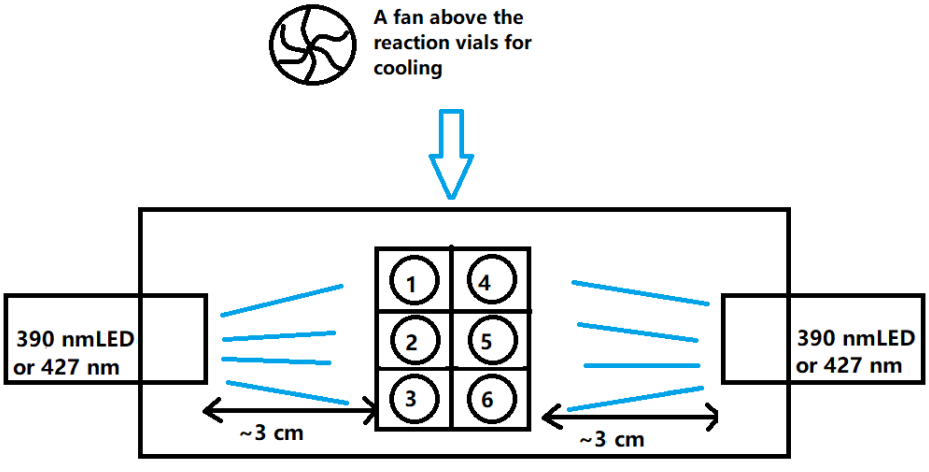
Flame-dried equipment was stored in a 130 °C oven before use and either allowed to cool in a cabinet desiccator or assembled hot and allowed to cool under an inert atmosphere. Chromatographic purification of products was accomplished using flash column chromatography Silicycle Silica flash F60 (particle size 40−63 μm, 230−400 mesh). Thin-layer chromatography was performed on EMD Millipore silica gel 60 F254 glass-backed plates (layer thickness 250 μm, particle size 10−12 μm, impregnated with a fluorescent indicator).

Visualization of the developed chromatogram was accomplished by fluorescence quenching under shortwave UV light and/or by staining with phosphomolybdic acid, p-anisaldehyde, or $KMnO_4$ stains.

LED Lamps. The following Kessil LED lamps were used in this work:

- 390 nm lamp: PR160L-390, 40W (purple visible light)
- 427 nm lamp: PR160L-427, 40W (blue visible light)
- 440 nm lamp: PR160L-440, 40W (blue visible light)
- 525 nm lamp: PR160L-525, 40W (green visible light)

Reaction Vials. We used ChemGlass microwave reaction vials with heavy walls made of borosilicate glass (product # CG-4920-01). The vial was placed approximately 3 cm away from the LED lamps, with the LEDs shining directly at the side of the vial as shown in following picture. Three reactions per lamp could be set up at the same time. And a fan above the reaction vials can keep the temperature around 35 °C. 10 mL microwave reaction vial secured by 20 mm aluminum seals with 0.125-inch thick, blue PTFE / white silicone septa was used for the reaction.



Bird's eye view

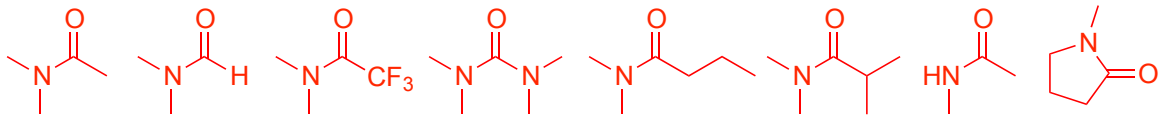
Instrumentation. For NMR spectrometry, NMR spectra were obtained on Bruker spectrometers operating at 400 or 500 MHz for ^{1}H NMR and 101 or 126 MHz for ^{13}C{1 H} NMR. The data were reported in the following order: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet), coupling constant, (Hz), relative integral made in reference to NMR solvent signals.

For mass spectrometry, gas chromatograph−mass spectrometry was obtained using a Hewlett-Packard GC System HP 6890 Series coupled with a HP 5973 Mass Selective Detector. High-resolution mass spectra were obtained using an Agilent Technologies 6520 Accurate-Mass Q-TOF LC/MS with electrospray ionization (ESI).

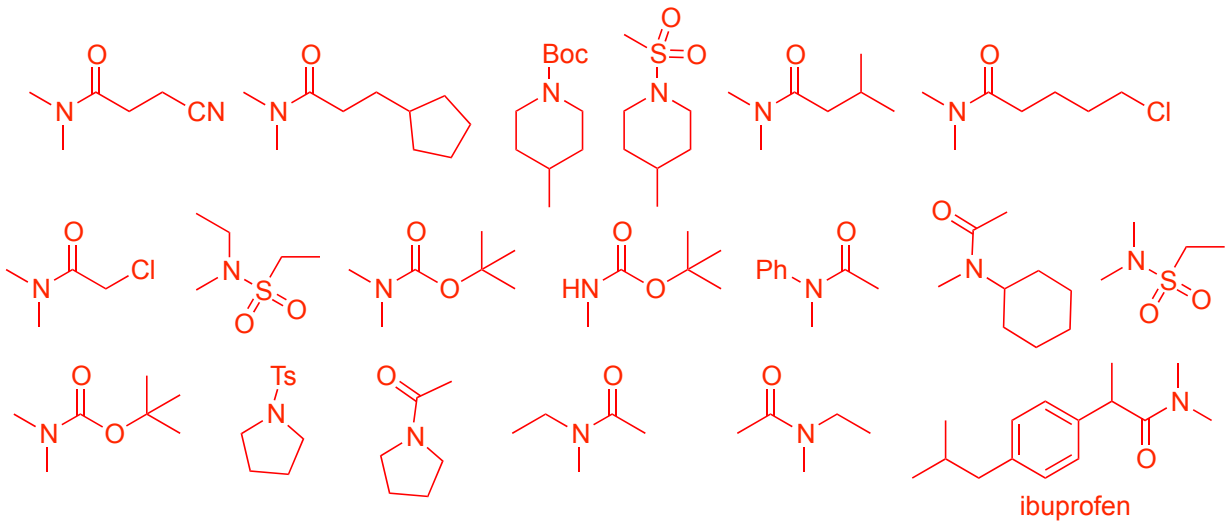
Materials General procedures for the synthesis of amide and disulfide derivatives followed reported procedures detailed below.

Procedure for Preparation of Starting Materials

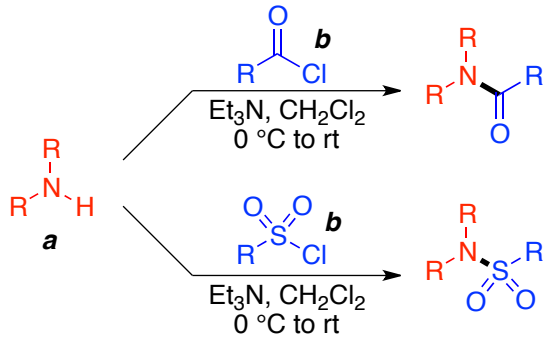
Following amides were purchased from Sigma-Aldrich, Oakwood Chemicals, and Fisher Chemicals:



Following amides and amide derivatives were synthesized following the procedure below:

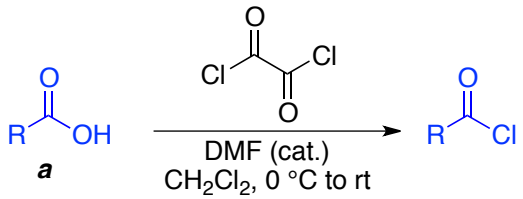


A: The General Procedure for the Preparation of Amides and Amide Derivatives



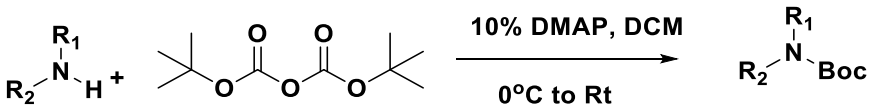
To a stirred solution of amine (**a**) (6 mmol) in DCM (20 mL) were added triethylamine（6.6 mmol）and the corresponding acyl chloride (1.0 mmol) dropwise at 0 °C. Then the mixture allowed to come to room temperature and stirred overnight (12h). Then the reaction mixture was poured in a separation funnel and washed with a brine solution (saturated NaCl) and extracted with 3 x 40 mL DCM. The combined organic phases were dried over anhydrous Na_2SO_4, concentrated under reduced pressure and purified by column chromatography to desired products.[1]

B: The General Procedure for the Preparation of Acid chloride



To a stirred solution of carboxylic acid (**a**) (6 mmol) and DMF (4 drops) in DCM (18 mL) at 0 °C was added $(COCl)_2$ (2 equiv) dropwise. After completion of addition, the solution was stirred for 5 minutes at 0 °C and then stirred at rt for 6 hrs. The solution was concentrated in vacuum to obtain the crude acyl chloride, which was used without further purification.[1,4]

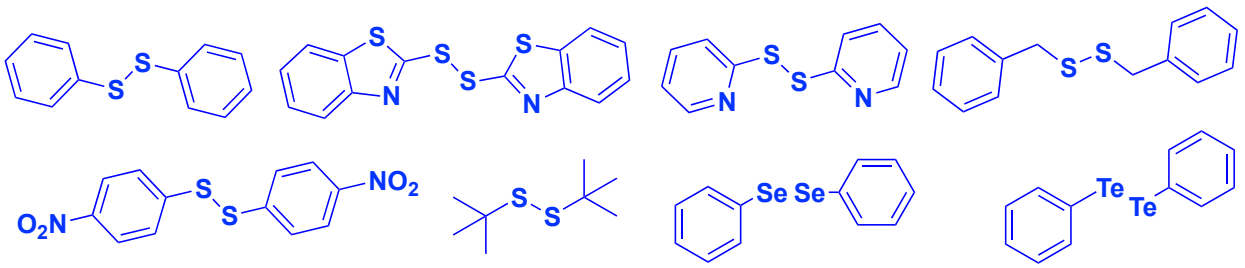
C: The General Procedure for the Preparation of N-Boc amines



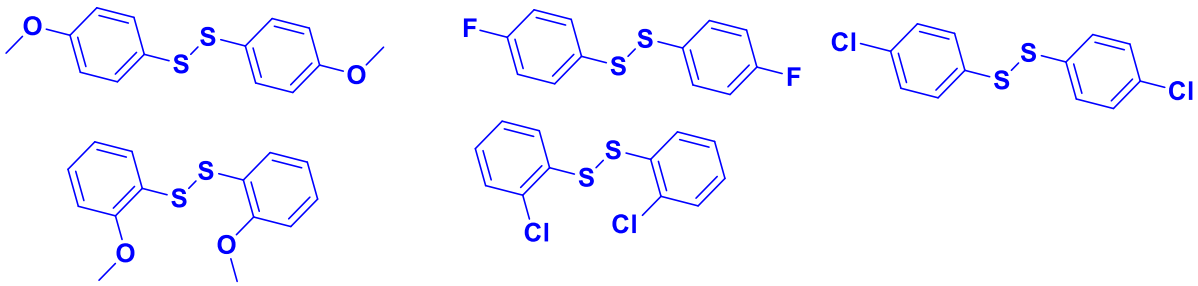
According to literature, the N-Boc amines can be synthesized by the condensation of corresponding amines with di-tert-butyl dicarbonate. The corresponding amines (1.0 equiv) and 4-dimethylaminopyridine (10 mol%) were mixed in a flask with a magnetic stirring bar. DCM was added as solvent. Then a solution of di-tert-butyl dicarbonate (1.1 equiv) in DCM was slowly added to the mixture under ice-water bath. And stirred at room temperature for 24hrs. The solution was washed by brine and DCM, then combined all the organic phase and dried using $MgSO_4$ and concentrated. This crude product was purified by flash column chromatography.[1-2]

Note: The reactions and the yields to synthesize amides were not optimized.

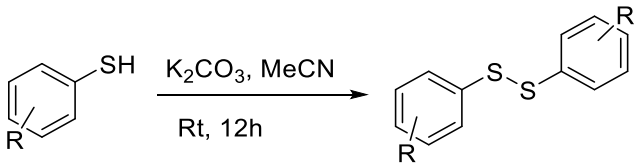
Following disulfide were purchased from Sigma-Aldrich, Oakwood Chemicals and Fisher Chemical.



Following disulfide derivatives were synthesized following the procedure below:



D. General Procedure for the Synthesis of disulfides.



A round bottom flask (50 mL) was charged with thiophenol (5 mmol), anhydrous potassium carbonate (0.69 g, 5 mmol), and MeCN (10 mL) sequentially. The resulting mixture was stirred at room temperature under air for 1 hour. And the desired disulfides were obtained quantitatively after filtration and concentration.[3]

Full Tables for Reaction Optimization

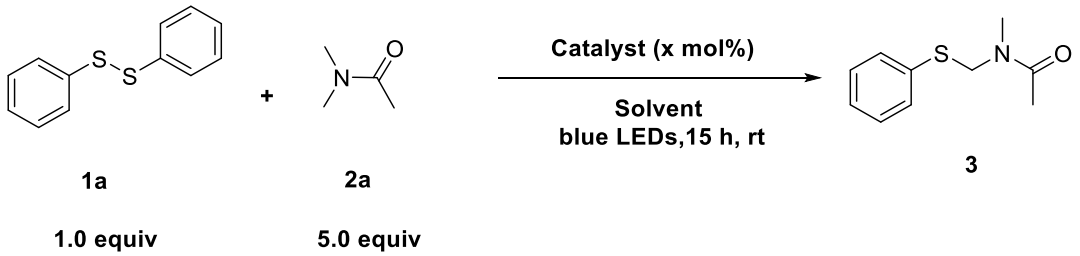


Table S1. Screening of solvents and concentrations.

Entry	Solvent	Volume (mL)	NMR Yield %
1	THF	2 mL	26%
2	DMSO	2 mL	trace
3	DCM	2 mL	trace
4	H_2O	2 mL	NR
5	DMA	2 mL	trace
6	**MeCN**	**2 mL**	**81%**
7	MeCN	1 mL	70%
8	MeOH	2 mL	trace
9	EtOH	2 mL	8%
10	Acetone	2 mL	58%
11	Acetone	2 mL	trace [a]

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and $FeCl_3$ (10 mol%) in the given solvent. Reaction under 390 nm Blue LED for 15 h. [a] Reaction performed under positive pressure of O_2 balloon.

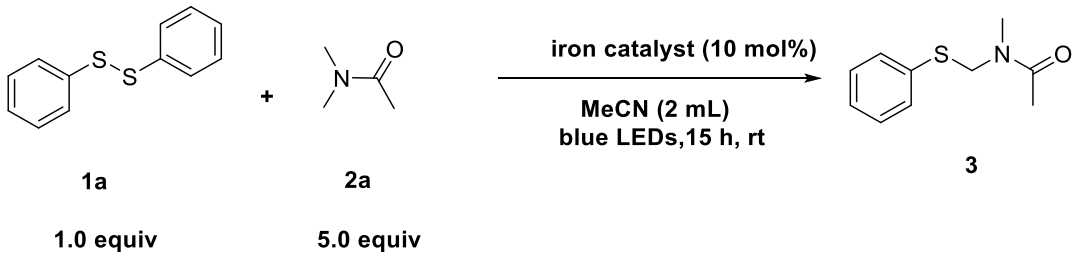


Table S2. Screening of iron catalysts.

Entry	Iron catalyst	NMR Yield/%
1	$FeCl_3$	81%
2	$FeBr_3$	NR
3	$Fe(NO_3)_3.9H_2O$	NR
4	$Fe(Cl)_3.6H_2O$	67%
5	$Fe(Cl)_2.4H_2O$	30%
6	$Fe(acac)_3$	NR

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and iron catalyst (10 mol%) in MeCN (2 mL). Reaction under 390 nm Blue LED for 15 h.

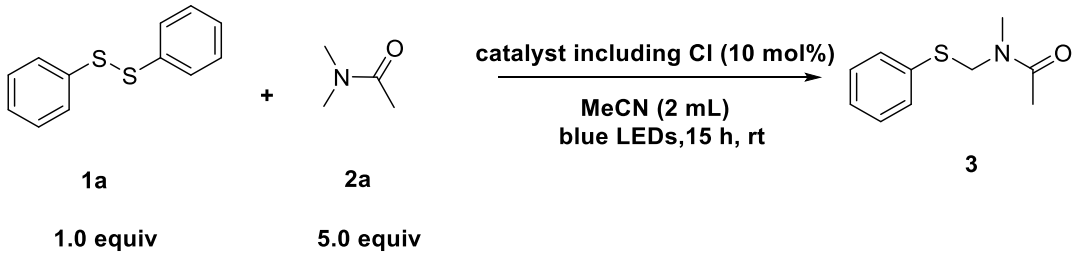


Table S3. Screening of other metal catalysts.

Entry	Cl catalyst	NMR Yield/%
1	$CuCl_2$	NR
2	CuCl	NR
3	$CeCl_3$	NR
4	$NiCl_2$	NR
5	TBACl	NR
6	$MgCl_2$	NR
7	LiCl	NR

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and metal chloride catalyst (10 mol%) in MeCN (2 mL). Reaction under 390 nm Blue LED for 15 h.

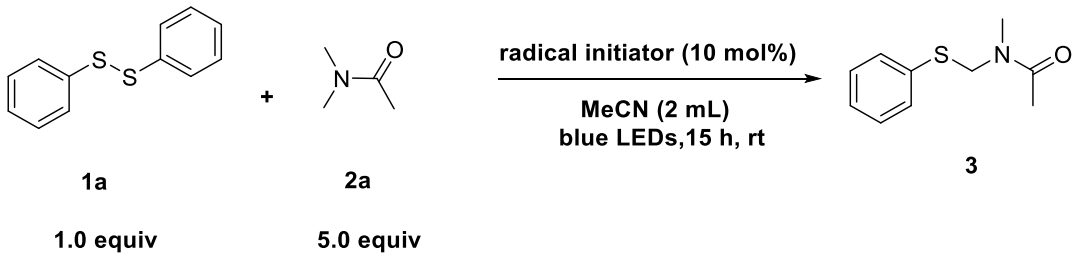


Table S4. Screening of other metal catalysts.

Entry	radical initiator	NMR Yield/%
1	I_2	NR
2	Tetrabutyl ammonium iodide (TBAI)	NR

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and radical initiator (10 mol%) in MeCN (2 mL). Reaction under 390 nm Blue LED for 15 h.

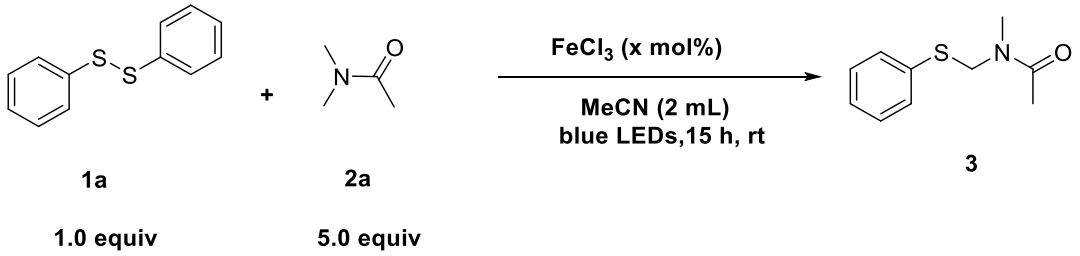


Table S5. Screening of $FeCl_3$ catalyst amounts.

Entry	Amount of $FeCl_3$ (mol%)	NMR Yield %
1	5	59%
2	20	75%
3	50	72%

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and $FeCl_3$ (xx mol%) in MeCN (2 mL). Reaction under 390 nm Blue LED for 15 h.

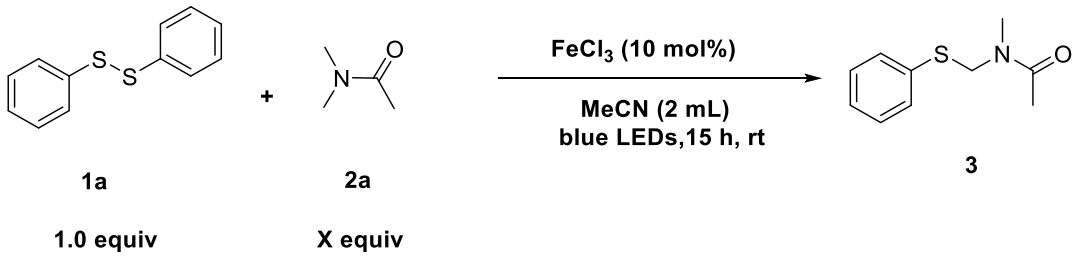


Table S6. Screening of amide equivalents.

Entry	DMA (equiv)	NMR Yield %
1	2	55%
2	10	39%

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**) , X equiv *N,N*-Dimethylacetamide (**2a**) and $FeCl_3$ (10 mol%) in MeCN (2 mL). Reaction under 390 nm Blue LED for 15 h.

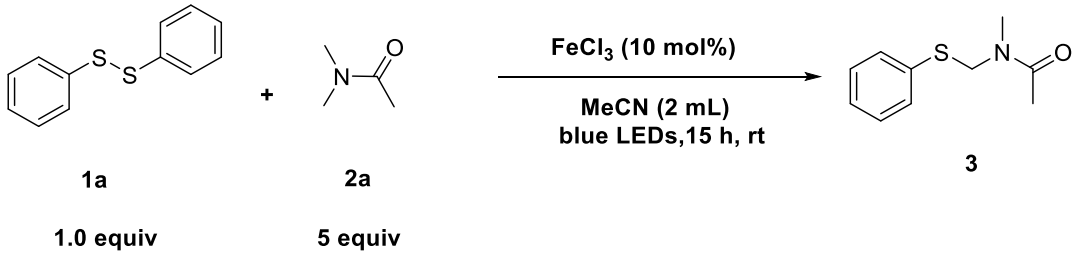


Table S7. Screening of light sources.

Entry	LED wavelength range	NMR Yield/%
1	427 nm	49%
2	440 nm	62%
3	CFL Light	NR
4	Dark	NR
5	Green LED	NR
6	UV lamp	NR

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and $FeCl_3$ (10 mol%) in MeCN (2 mL). Reaction under 390 nm LED for 15 h.

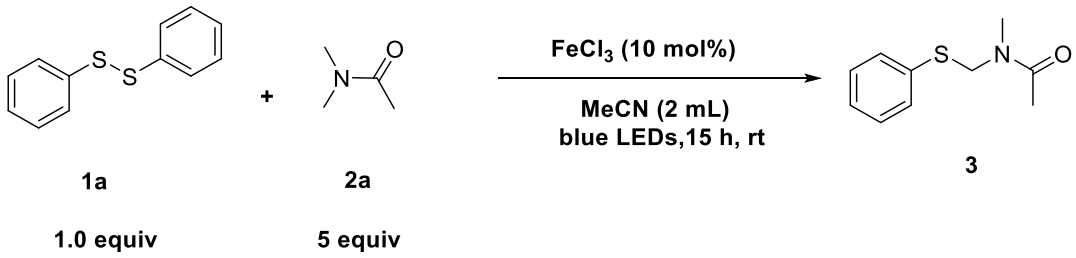


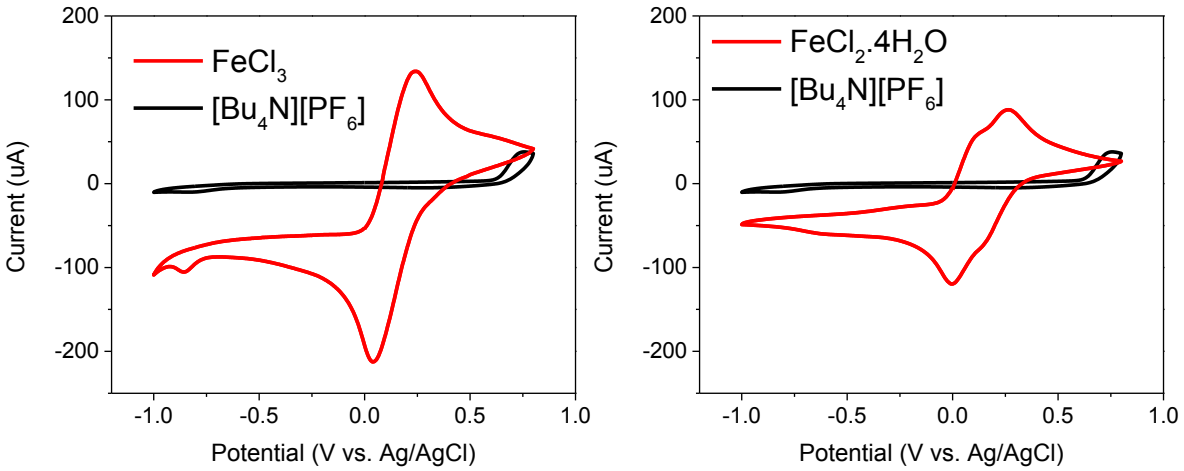
Table S8. Screening of reaction atmospheres.

Entry	Atmosphere	NMR Yield/%
1	air	81%
2	Argon	61%
3	Nitrogen	55%
4	O_2	NR

Reaction conditions: 0.2 mmol diphenyl disulfide (**1a**), 1.0 mmol *N,N*-Dimethylacetamide (**2a**) and $FeCl_3$ (10 mol%) in MeCN (2 mL). Reaction under 390 nm LED for 15 h.

$FeCl_3$ Cyclic Voltammetry study scans

The cyclic voltagramms were obtained with a Dropsens µstat 4000 potentiostat. The sample was prepared with 0.05 mmol of $FeCl_3$ (or $FeCl_2$) in 5mL of a solution of tetra-n-butylammonium hexafluorophosphate 0.1 M in acetonitrile. The solution was degassed with argon prior to the measurement and used a glassy carbon working electrode, platinum wire counter electrode and a 3.5 M NaCl Ag/AgCl reference electrode. The scan rate was 0.1 V/s.



UV-Vis Spectra

UV-Vis was obtained using Thermo Scientific Evolution 600 UV-Vis Spectrophotometer and Thorlabs 3500 µL micro quartz cuvettes (cat. No. CV10Q35A).

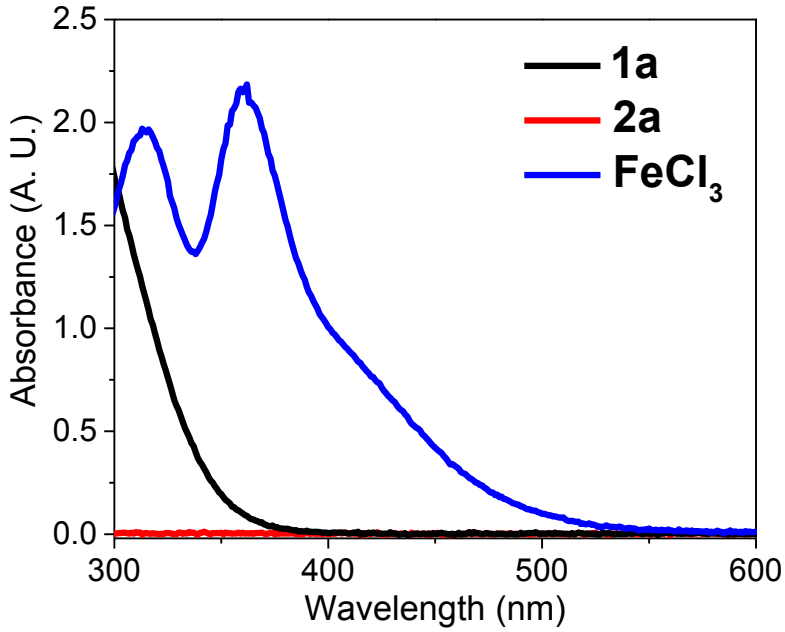


Figure S1. *UV-vis spectra of the different components of the reaction in MeCN.*

Stock solutions of **1a**, **2a** and $FeCl_3$ in MeCN were prepared with a concentration of 0.2, 1.0 and 0.02 mmol/mL, respectively. All spectra were taken with a concentration of $2.5x10^{-3}$ mmol/mL of **1a**, $1.2x10^{-2}$ mmol/mL of **2a** and $2.5x10^{-4}$ mmol/mL of $FeCl_3$.

1a= Diphenyldisulfide **2a**= *N,N*-Dimethylacetamide

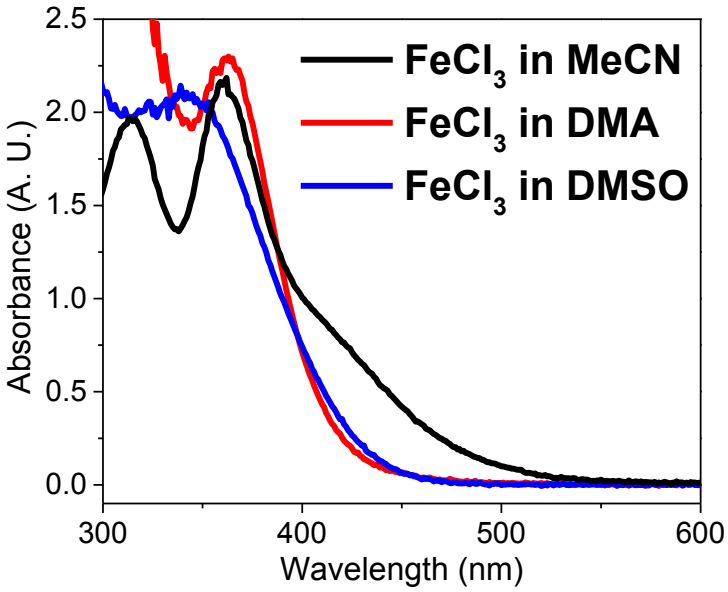


Figure S2. *Solvent effect.*

Stock solutions of $FeCl_3$ in MeCN, DMA and DMSO were prepared with a concentration of 0.02 mmol/mL. All spectra were taken with a concentration of $2.5x10^{-4}$ mmol/mL of $FeCl_3$.

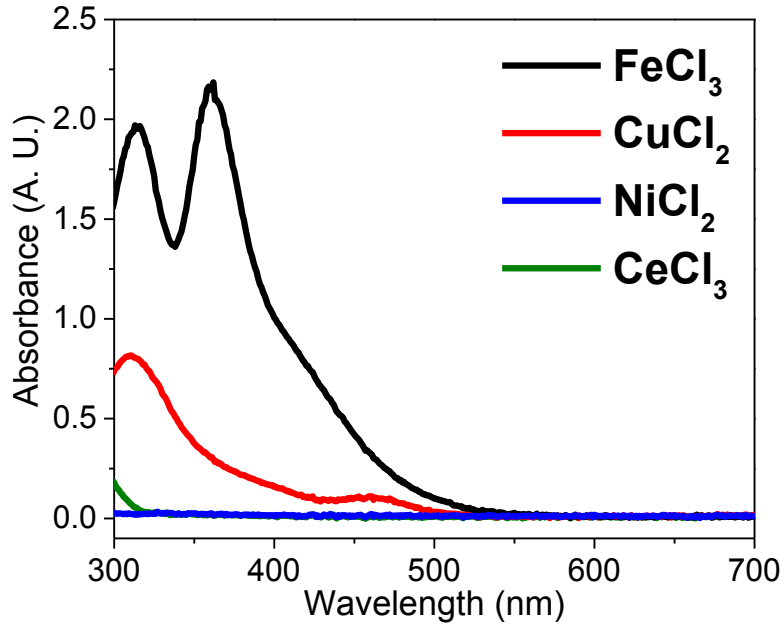


Figure S3. *Cation effect.*

Stock solutions of $FeCl_3$, $CuCl_2$, $NiCl_2$ and $CeCl_3$ in MeCN were prepared with a concentration of 0.02 mmol/mL. All spectra were taken with a concentration of $2.5x10^{-4}$ mmol/mL.

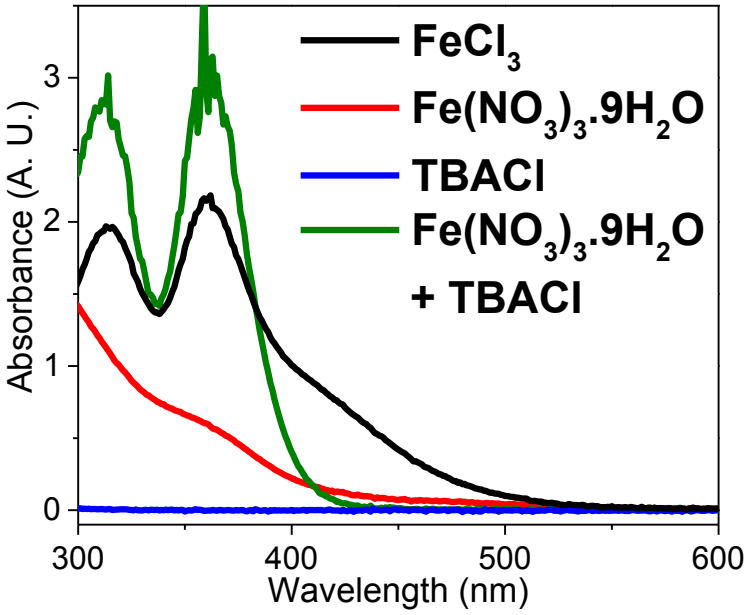
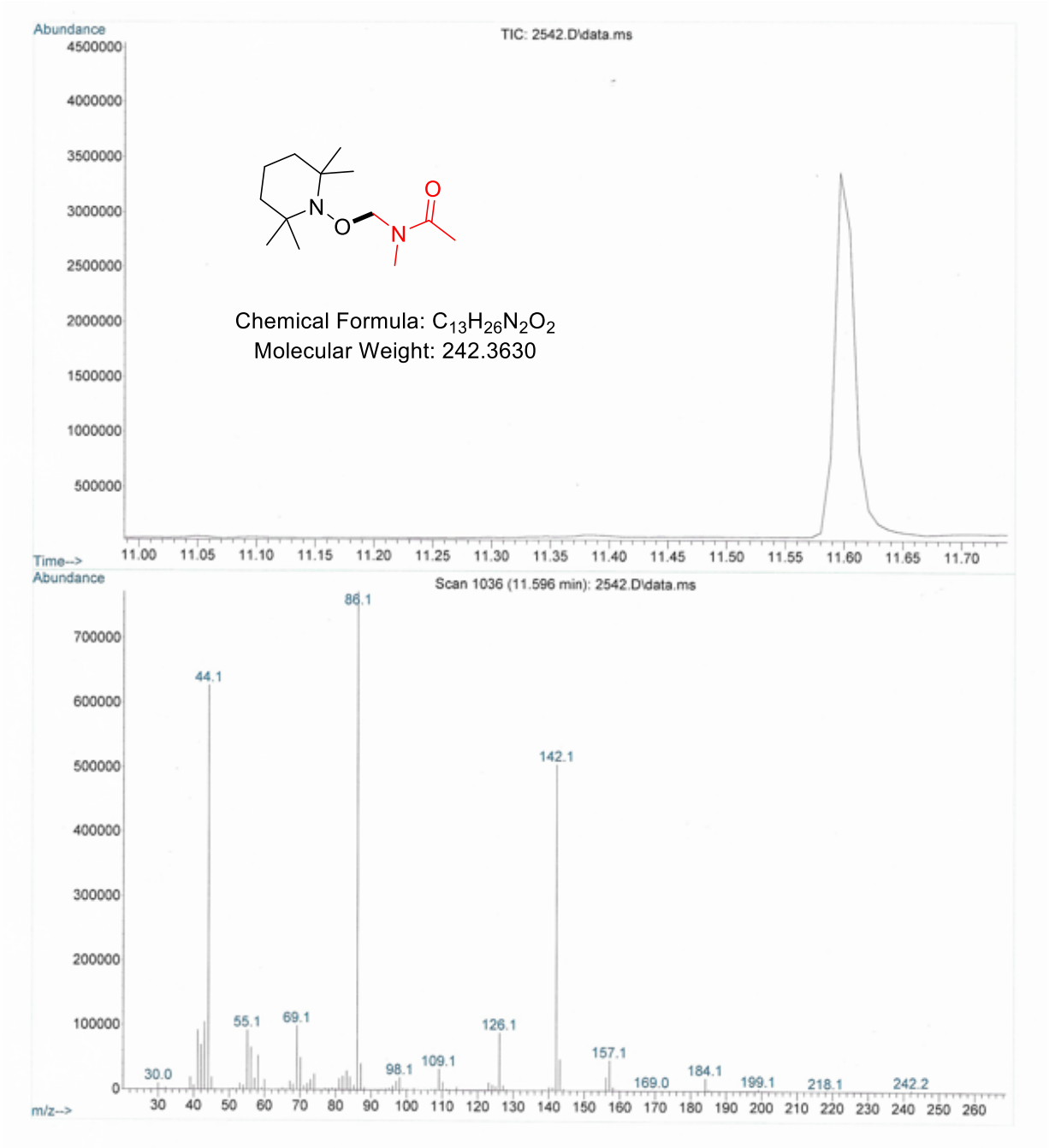
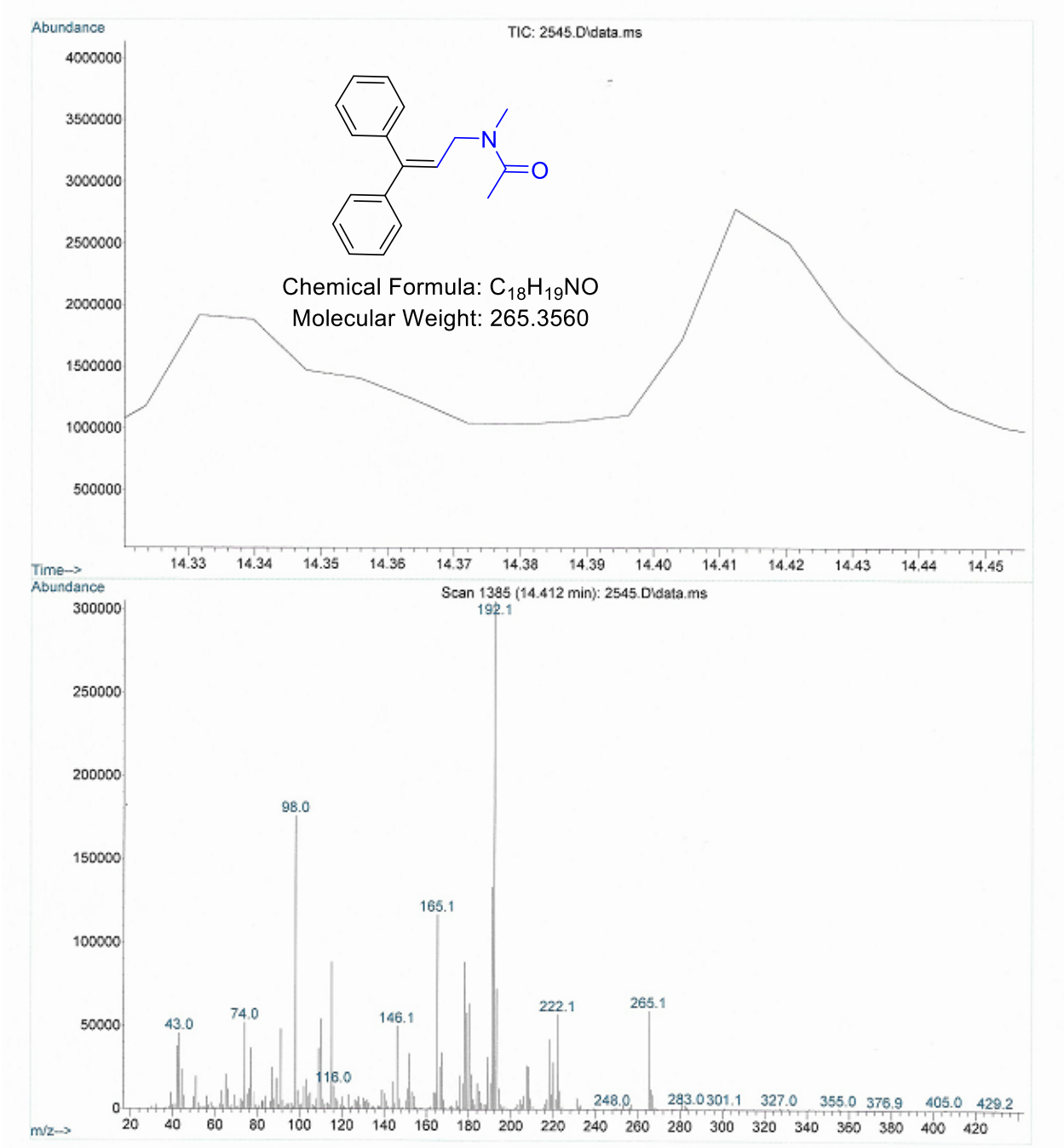


Figure S4. *Chlorine effect.*

Stock solutions of $FeCl_3$, $Fe(NO_3)_3.9H_2O$, tetrabutylammoniumchlride (TBACl) and $Fe(NO_3)_3.9H_2O$ + TBACl in MeCN were prepared with a concentration of 0.02 mmol/mL. All spectra were taken with a concentration of $2.5x10^{-4}$ mmol/mL.

GC-MS spectrum for the radical addition trapping reaction

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14.37
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14.39
14.40
14.41
14.42
14.43
14.44
14.45
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265.1
116.0
248.0
283.0
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327.0
355.0
376.9
405.0
429.2
0
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40
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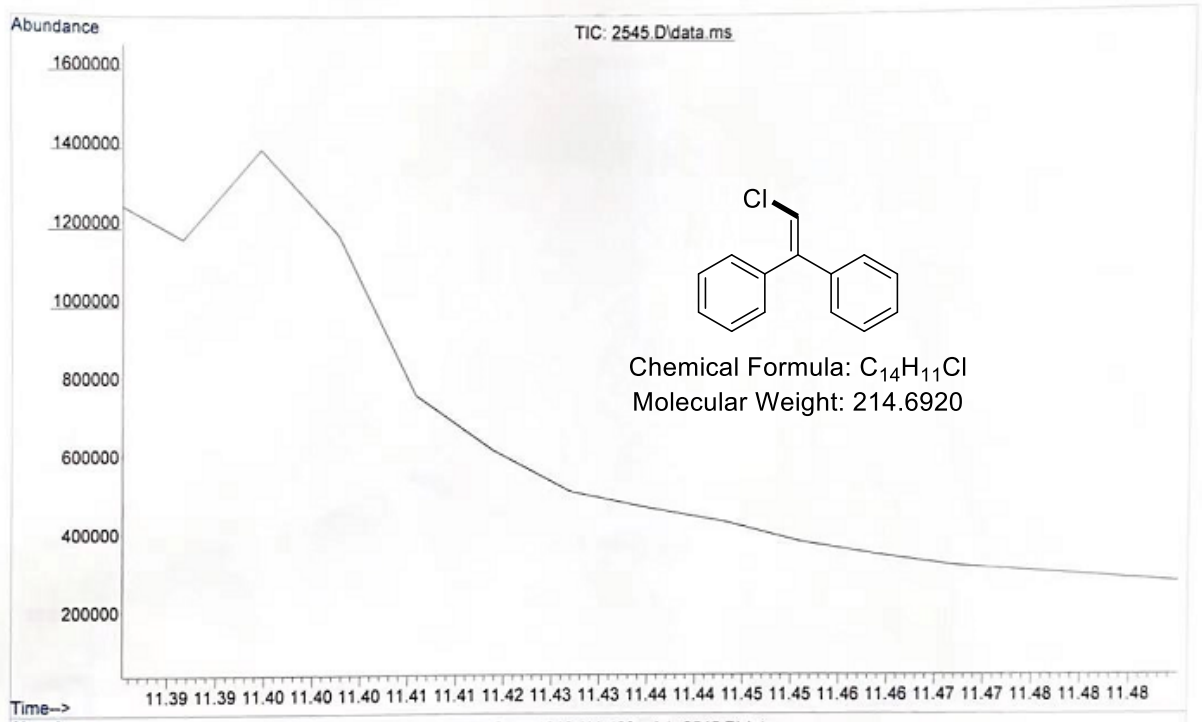
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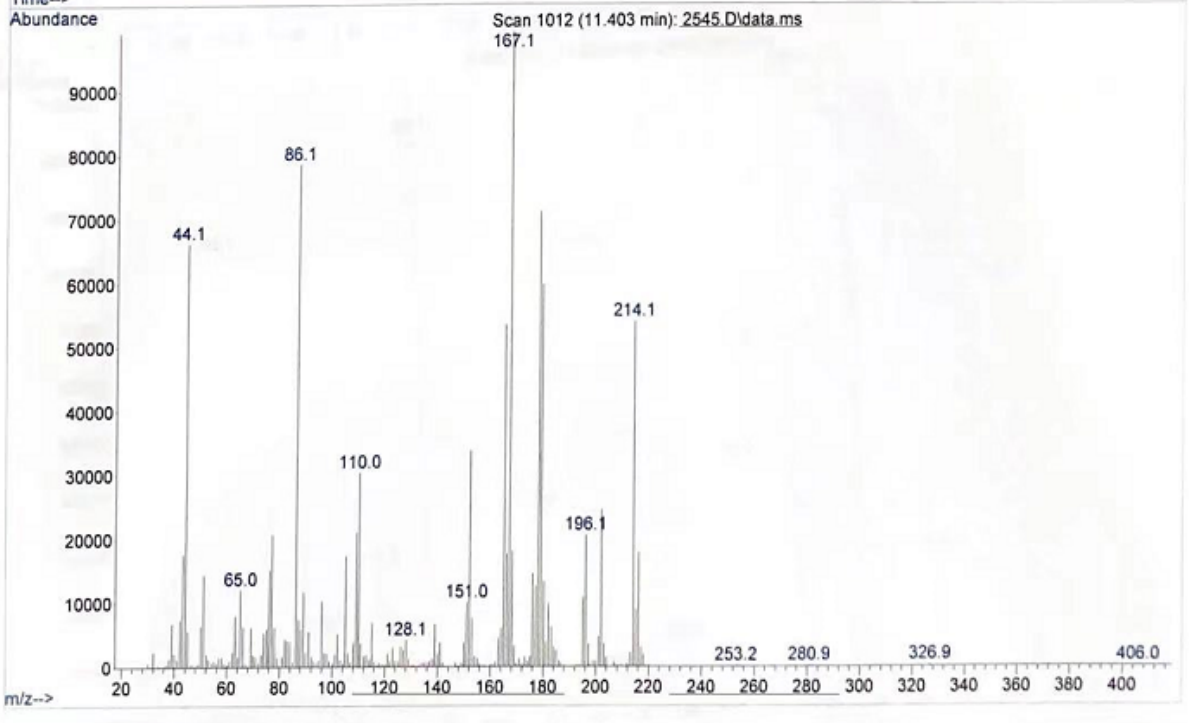
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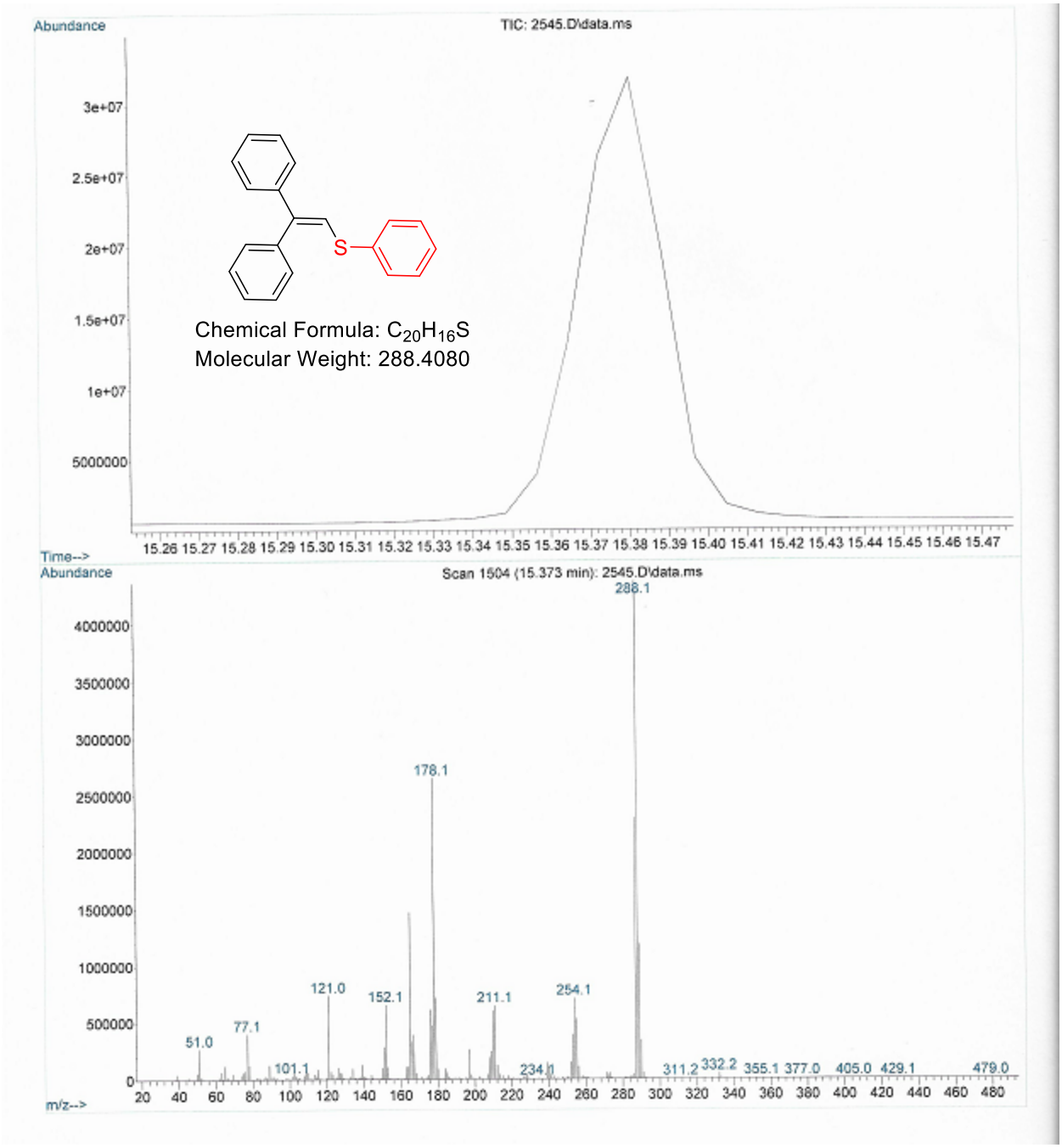
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Molecular Weight: 288.4080
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Time-->
Abundance
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311.2
332.2
355.1
377.0
405.0
429.1
479.0
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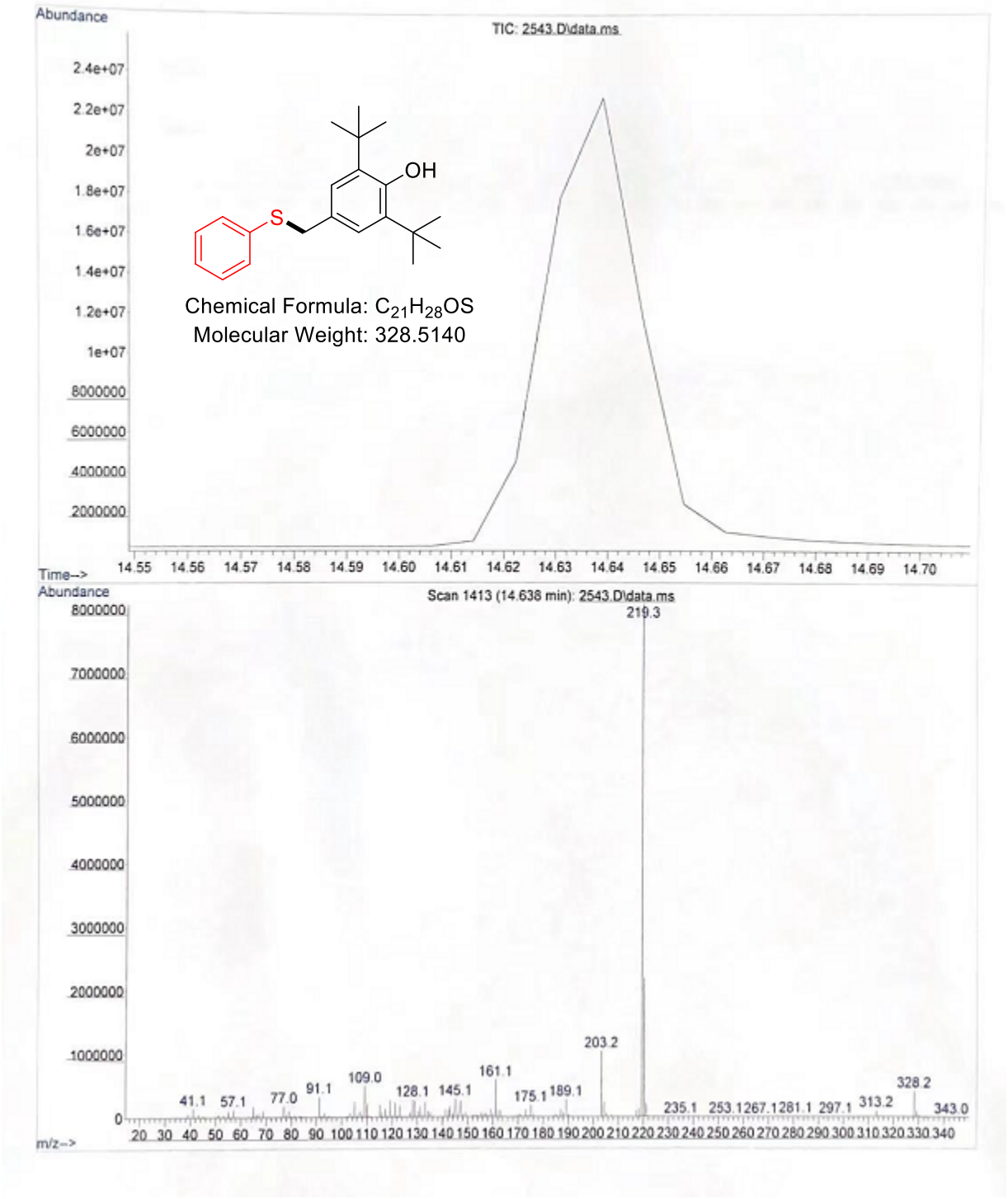
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2000000
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5000000
4000000
3000000
2000000
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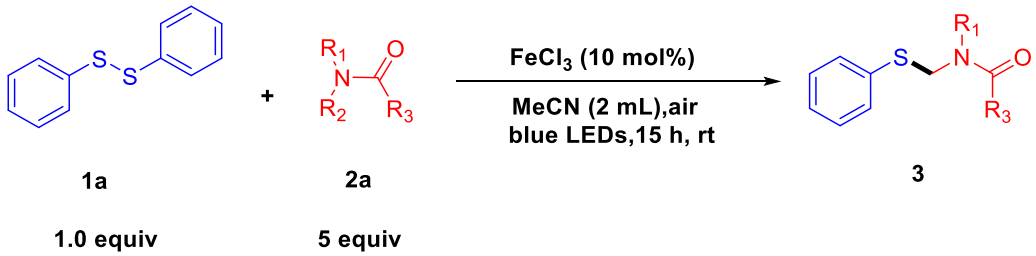
109.0
91.1
77.0
57.1
41.1
128.1
145.1
175.1
189.1
235.1
253.1
267.1
281.1
297.1
313.2
328.2
343.0
m/z-->
20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340

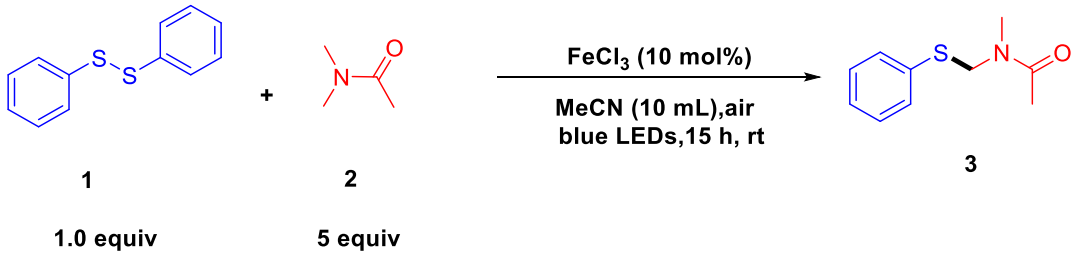
General Reaction Procedure and Characterizations Data.



General standard reaction procedure:

Anhydrous $FeCl_3$ (3.3 mg, 0.02 mol, 10 mol%) diphenyl disulfide (0.2 mmol, 1 equiv.), amides (1 mmol, 5 equiv.), and MeCN (2 mL, c = 1 M) were mixed in a 10 mL microwave vial equipped with a stir bar under air. The vial was sealed with a septum-cap and placed 2 cm away from a 390 nm blue LED (40W). The temperature was kept at approximately 35 °C through cooling with a fan (heating caused by LED lamp). After being stirred for 15 hours, the reaction mixture was poured into 20 mL of water, and the resulting mixture was extracted with EtOAc (3 × 20 mL). The combined organic phase was dried over $MgSO_4$, filtered through celite, and concentrated under vacuum. The residue was further purified by flash column chromatography on silica gel using EtOAc/Hexane.

Large-scale (1 mmol) synthesis and transformation of products:

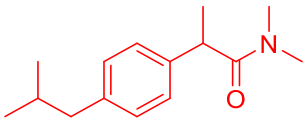


Anhydrous $FeCl_3$ (16.5 mg, 0.1 mol, 10 mol%) diphenyl disulfide (218mg, 1 mmol, 1 equiv), amides (0.465 mL, 5 mmol, 5 equiv.), and MeCN (10 mL, c = 1 M) were mixed in a 20 mL scintillation vial equipped with a stir bar under air. The vial was sealed with white Cap and placed 2 cm away from a 390 nm blue LED (40W). The temperature was kept at approximately 35 °C through cooling with a fan (heating caused by LED lamp). After being stirred for 24 hours, the reaction mixture was poured into 30 mL of water, and the resulting mixture was extracted with EtOAc (3 × 20 mL). The combined organic phase was dried over $MgSO_4$, filtered through celite, and concentrated under vacuum. The residue was further purified by flash column chromatography on silica gel using hexane/ethyl acetate (2:1) to get the desired product compound **3** as a light-yellow oil (148 mg, 73% isolated yield).

Analytical Data of Compounds

Starting materials:

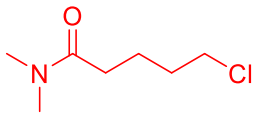
2-(4-isobutylphenyl)-*N*,*N*-dimethylpropanamide



Following the Procedure A for the synthesis of starting materials: to a stirred solution of dimethylamine (**a**) (60 mmol) in DCM (30 mL) were added triethylamine（66 mmol）and 2-(4-isobutylphenyl)propanoyl chloride (10.0 mmol, 1 equiv) dropwise at 0 °C. After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane to get solid. (1.98 g, 85% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15- 7.03 (d, 2H, *J* = 8.0 Hz), 6.99 (d, 2H, *J* = 8.0 Hz), 3.77 (q, 1H, *J* = 6.9 Hz), 2.85 (s, 3H), 2.79 (s, 3H), 2.35 (d, 2H, *J* = 7.2 Hz), 1.76 (dp, 1H, *J* = 13.5, 6.7 Hz), 1.33 (d, 3H, *J* = 6.9 Hz), 0.81 (d, 6H, *J* = 6.6 Hz).
^{13}C NMR (101 MHz, $CDCl_3$) δ 136.3, 131.3, 129.0, 127.1, 55.6, 38.4, 33.1, 28.8.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{15}H_{24}NO$ 234.1858, found 234.1852.

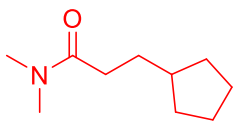
5-chloro-*N*,*N*-dimethylpentanamide



Following the Procedure A for the synthesis of starting materials: to a stirred solution of dimethylamine (**a**) (30 mmol) in DCM (30 mL) were added triethylamine（33 mmol）and 5-chloropentanoyl chloride (5.0 mmol, 1 equiv) dropwise at 0 °C. After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane to get dark yellow oil. (717 mg, 88% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.57 (t, 3H, *J* = 6.1 Hz), 3.02 (s, 6H), 2.47 (t, 2H, *J* = 6.9 Hz), 1.87−1.77 (m, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 177.0, 173.5, 64.0, 44.7, 44.5, 33.1, 32.1, 32.0, 31.8, 22.5, 22.1.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_7H_{15}ClNO$ 164.0842, found 164.0837.

3-cyclopentyl-*N*,*N*-dimethylpropanamide

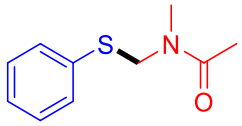


Following the Procedure A for the synthesis of starting materials: to a stirred solution of dimethylamine (**a**) (30 mmol) in DCM (30 mL) were added triethylamine（33 mmol）and 3-cyclopentylpropanoyl chloride (5.0 mmol, 1 equiv) dropwise at 0 °C. After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane to get yellow oil. (802 mg, 95% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.98 (d, 6H, *J* = 28.7 Hz), 2.42-2.24 (m, 2H), 1.87- 1.71 (m, 3H), 1.71- 1.43 (m, 6H), 1.22- 1.03 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 40.0, 37.3, 35.4, 32.7, 32.6, 31.5, 25.2.
HRMS (ESI-TOF) m/z: [M + Na]+ calcd. for $C_{10}H_{19}NONa$ 192.1364, found 192.1359.

Methodology products:

_N_-methyl-_N_-((phenylthio)methyl)acetamide, (3)

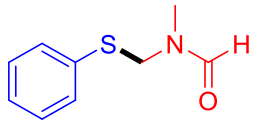


Known compound reported by reference 7.

Reaction in MeCN (2 mL), using *N N*-Dimethylacetamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (30.5 mg, 78% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.5-7.46 (m, 2H), 7.39-7.27 (m, 3H), 4.89 and 4.65 (2x s, 2H), 2.99 and 2.97 (2xs, 3H), 2.02 and 1.65 (2xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7, 135.0 and 134.0, 132.5 and 131.5, 129.4 and 129.0, 127.3, 57.9 and 51.7, 35.2 and 32.9, 21.9 and 20.7.

_N_-methyl-_N_-((phenylthio)methyl)formamide, (4)



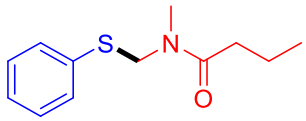
Known compound reported by reference 5.

Reaction in MeCN (2 mL), using *N,N-dimethylformamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/4 as a light-yellow oil. (23.5 mg, 65% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 and 7.52 (2×s, 1H), 7.24-7.46 (m, 5H), 4.81 and 4.54 (2xs, 2H), 2.95 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) 162.2 and 161.4, 134.8 and 134.4, 131.6 and 131.2, 129.6 and 129.3, 128.9 and 128.9, 127.3, 56.8 and 48.1, 33.2 and 28.7

_N_-methyl-_N_-((phenylthio)methyl)butyramide, (5)



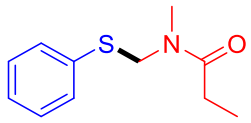
Reaction in MeCN (2 mL), using *N,N-dimethylbutyramide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (29.4 mg, 66% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50−7.36 (m, 2H), 7.29-7.11 (m, 3H), 4.83 and 4.60 (2×s, 2H), 2.91 and 2.90 (2xs, 3H), 2.13 (t, 1H, *J* = 7.4 Hz), 1.77 (t, 1H, *J* = 7.5 Hz), 1.48 (p, 1H, *J* = 7.4 Hz), 1.36 (h, 1H, *J* = 7.3 Hz), 0.82 and 0.69 (2xt, 3H, *J* = 12, 12 Hz).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.1 and 173.0, 134.8 and134.0, 132.7 and 131.6, 129.4 and 129.0, 128.8 and 127.2, 56.9, 51.8, 35.5, 34.4, 33.0, 18.3, 18.3, 13.8 and 13.8.

HRMS (ESI-TOF) m/z: [M + Na]+ calcd. for $C_{12}H_{17}NOSNa$ 246.0929, found 246.0923.

_N_-methyl-_N_-((phenylthio)methyl)propionamide, (6)



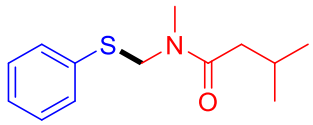
Known compound reported by reference 7.

Reaction in MeCN (2 mL), using N,N-dimethylpropionamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (26.8 mg, 53% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43-7.45 (m, 2H), 7.20-7.32 (m, 3H), 4.87 and 4.63 (2×s, 2H), 2.94 (s, 3H), 2.23 and 1.89 (2×t, 2H, *J* = 7.4 Hz), 1.02 and 0.89 (2×t, 3H, *J* = 7.4 Hz)

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.8 and 173.7, 134.6 and 134.3, 133.9, 132.5 and 132.2 , 132.0, 129.1 and 128.8, 128.7 and 128.6, 127.0 and 126.1, 56.5 and 51.8, 34.2 and 33.0, 26.6 and 25.5, 9.0 and 8.9

***N*,3-dimethyl-*N*-((phenylthio)methyl)butanamide, (7)**

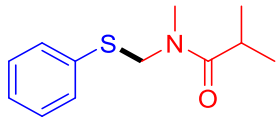


Reaction in MeCN (2 mL), using N,N,3-trimethylbutanamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (22.7 mg, 48% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 -7.34 (m, 2H), 7.30- 7.18 (m, 3H), 4.92 and 4.68 (2xs, 2H), 2.99 and 2.97 (2xs, 3H), 2.10 (d, 1H, J = 8.0 Hz), 2.01 (ddt, 1H, J = 29.9, 13.3, 6.5 Hz), 1.75 (d, 1H, J = 6.9 Hz), 0.87 and 0.79 (2xd, 3H, J = 6.6 Hz).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.6 and 172.5, 134.8 and 133.9, 132.7, 131.6, 129.4 and 129.0, 128.8, 127.2, 57.0, 51.8, 42.4, 41.3, 34.5, 33.0, 25.4 and 25.2, 22.6.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{13}H_{20}NOS$ 238.1266, found 238.1260.

***N*-methyl-*N*-((phenylthio)methyl)isobutyramide, (8)**



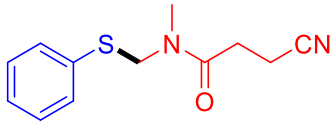
Known compound reported by reference 7

Reaction in MeCN (2 mL), using *N,N*-dimethylisobutyramide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (35.7 mg, 80% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (m, 2H), 7.41-7.19 (m, 3H), 4.93 and 4.72 (2xs, 2H), 3.04 and 3.00 (2xs, 3H), 2.75-2.68 and 2.37-2.30 (m, 1H), 1.01 and 0.89 (2xd, 3H, J = 6.7 Hz).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.4 and 177.2, 134.8 and 133.7, 132.4 and 131.9, 129.4 and 128.9, 128.8 and 128.7 127.2, 56.4, 51.8, 34.1, 33.2, 30.6, 30.0, 19.4 and 19.0.

3-cyano-*N*-methyl-*N*-((phenylthio)methyl)propanamide, (9)

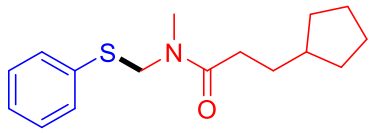


Reaction in MeCN (2 mL), using 3-cyano-*N,N*-dimethylpropanamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/2 as a light-yellow oil. (27.1 mg, 58% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49-7.47 (m, 2H), 7.37-7.35 (m, 3H), 4.95 and 4.77 (2xs, 2H), 3.71 and 3.62 (2xt, 2H, *J* = 6.6 Hz), 2.68 and 2.66 (2xt, 2H, *J* = 6.6 Hz), 1.74 and 1.63 (2xs, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.0, 134.9, 131.8, 131.6, 129.6, 129.3, 127.8, 118.2, 57.0, 42.2, 20.9, 16.4.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{12}H_{15}N_2OS$ 235.0905, found 235.0900.

3-cyclopentyl-*N*-methyl-*N*-((phenylthio)methyl)propanamide, (10)

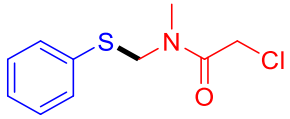


Reaction in MeCN (2 mL), using 3-cyclopentyl-*N,N*-dimethylpropanamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a light-yellow oil. (16.6 mg, 30% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50-7.48 (m, 2H), 7.38-7.24 (m, 3H), 4.93 and 4.70 (2xs, 2H), 3.01 and 3.00 (2xs, 3H), 2.26 and 1.90 (2xt, 2H, *J* = 1.6 Hz), 1.79-1.39 (m, 8H), 1.17-0.85 (m, 1H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4 and 173.4, 135.5 and 134.8, 134.7, 133.9, 132.7, 131.7, 129.4 and 128.9, 128.9, 127.2, 57.0, 51.8, 39.7 and 39.6, 34.4, 33.1, 32.9, 32.5 and 32.4, 31.8 and 31.6. 31.2 and 31.1, 25.1 and 25.1 22.7 and 14.1.

HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{16}H_{24}NOS$ 278.1579, found 278.1574.

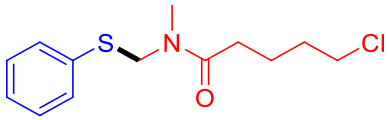
2-chloro-*N*-methyl-*N*-((phenylthio)methyl)acetamide, (11)



Reaction in MeCN (2 mL), using 2-chloro-*N,N*-dimethylacetamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil. (41.2 mg, 90% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41-7.39 (m, 2H), 7.32-7.18 (m, 3H), 4.81 and 4.62 (2xs, 2H), 3.92 and 3.40 (2xs, 2H), 3.00 and 2.95 (2xs, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.7 and 166.5, 135.1, 133.3, 132.1, 129.7 and 129.4, 129.1, 127.7, 57.2, and 52.6, 41.2 and 40.2, 34.6 and 33.6.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{10}H_{13}ClNOS$ 230.0406, found 230.0401.

5-chloro-*N*-methyl-*N*-((phenylthio)methyl)pentanamide, (12)

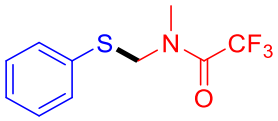


Reaction in MeCN (2 mL), using 5-chloro-*N,N*-dimethylpentanamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/30 to 1/5 as a light-yellow oil. (30.3 mg, 56% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52-7.47 (m, 2H), 7.39-7.24 (m, 3H),, 4.92 and 4.68 (2xs, 2H), 3.52 and 3.44 (2xt, 2H, *J* = 8.0 Hz), 3.01 and 3.00 (2xs, 3H), 2.28 (t, 2H, *J* = 1.6 Hz), 1.88 (t, 2H, *J* = 1.2 Hz), 1.60 (dddd, 2H, *J* = 13.2, 7.8, 5.4, 2.6 Hz).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.5 and 172.4, 135.0, 133.7, 132.9 and 132.5, 131.9 and 131.7, 130.8, 129.9 and 129.7, 129.4 and 129.3, 129.0 and 127.4, 56.8, 51.9, 44.7 and 44.6, 34.4, 33.1, 32.5, 32.0, 31.9 and 31.4, 22.1 and 22.1.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{13}H_{19}ClNOS$ 272.0876, found 272.0870.

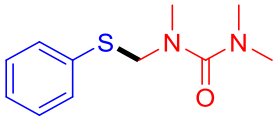
2,2,2-trifluoro-*N*-methyl-*N*-((phenylthio)methyl)acetamide, (13)



Reaction in MeCN (2 mL), using 2,2,2-trifluoro-*N,N*-dimethylacetamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/60 to 1/40 as a light-yellow oil. (30.8 mg, 62% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49-7.46 (m, 2H), 7.32-7.30 (m, 3H), 4.92, 4.86 and 4.77 (3xs, 2H), 3.17, 3.14 and 3.08 (3xs, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 134.8 and 134.2, 133.4, 132.9, 132.9, 129.8 and 129.6, 129.5 and 129.4, 129.3 and 129.1, 128.4 and 128.3, 126.6, 117.6, 114.7, 53.9 and 53.2, 34.2 and 33.9, 33.9, 29.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ -68.0, -69.8 and -69.9
HRMS (EI-QTOF) m/z: [M]+ calcd. for $C_{10}H_{10}F_3NOS$ 249.0435, found 249.0434.

1,1,3-trimethyl-3-((phenylthio)methyl)urea, (14)

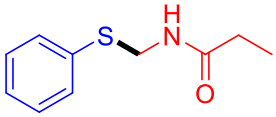


Reaction in MeCN (2 mL), using 1, 1, 3,3-tetramethylurea (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/3 as a light-yellow oil. (30.4 mg, 68% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38-7.36 (m, 2H), 7.29-7.10 (m, 3H), 4.71, 4.65 and 4.63 (3xs, 2H), 2.81 and 2.79 (2xs, 3H), 2.62 and 2.58 (2xs, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.2 and 164.1, 135.2 and 135.1, 134.0 and 133.6, 132.9 and 132.4, 131.9 and 131.5, 131.0, 130.3, 129.5 and 129.3, 129.0 and 123.0, 127.4 and 127.1, 68.3 and 57.2 and 57.2, 56.6, 38.5 and 38.4, 38.3, 36.3 and 36.2.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{11}H_{17}N_2OS$ 225.1062, found 225.1018.

***N*-((phenylthio)methyl)propionamide, (15)**

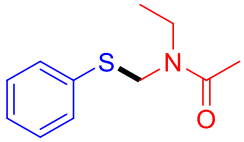


Reaction in MeCN (2 mL), using *N*-methylacetamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/3 as a light-yellow oil. (21.7 mg, 60% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37-7.33 (m, 2H), 7.26-7.22 (m, 3H), 5.85 (s, 1H), 4.62 and 4.61 (2xs, 2H), 2.08 (q, 2H, *J* = 7.6 Hz), 1.02 (t, 3H, *J* = 7.6 Hz).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.5, 133.8, 132.4 and 132.4, 131.7 and 131.6, 131.1 and 131.0, 129.5 and 129.4, 129.2, 128.3, 127.9 and 127.3, 127.3, 43.5, 29.7, 9.6.

HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{10}H_{14}NOS$ 196.0796, found 196.0791.

***N*-ethyl-*N*-((phenylthio)methyl)acetamide, (16)**

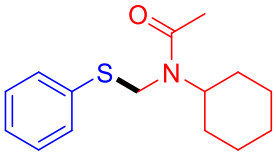


Reaction in MeCN (2 mL), using *N-ethyl-N-methylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/3 as a light-yellow oil. (29.2 mg, 70% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42-7.37 (m, 2H), 7.27 – 7.16 (m, 3H), 4.82 and 4.57 (2xs, 2H), 3.43 and 3.34 (2xq, 2H, *J* = 7.2 Hz), 1.99 and 1.63 (2xs, 3H), 1.06 (dt, 3H, *J* = 10.1, 7.2 Hz).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.2, 134.6 and 134.3, 132.7, 131.4 and 131.1, 129.4 and 129.3, 129.0 and 128.8, 127.2, 55.4, 49.0, 41.8, 39.7, 21.4 and 21.1, 13.6, 12.7.

HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{11}H_{16}NOS$ 210.0953, found 210.0947.

_N_-cyclohexyl-_N_-((phenylthio)methyl)acetamide, (17)

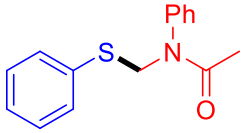


Reaction in MeCN (2 mL), using *N-cyclohexyl-N-methylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 as a light-yellow oil. (28.99 mg, 55% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53-7.45 (m, 2H), 7.33- 7.22(m, 3H), 4.81 and 4.67 (2xs, 2H), 4.27-4.19 (m, 1H), 3.59-3.41 (m, 1H), 2.15 and 2.02 (2xs, 3H), 1.87-1.71 (m, 4H), 1.65 (m, 1H), 1.58-1.21 (m, 4H), 1.10 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.9 and 170.3, 134.3, 132.9, 131.8, 129.7 and 129.3, 128.9, 128.0, 127.0, 58.0, 54.2, 51.9, 47.6, 32.1, 31.0, 26.0 25.6 and 25.2, 22.3 and 22.0.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{15}H_{22}NOS$ 264.1422, found 264.1419.

_N_-phenyl-_N_-((phenylthio)methyl)acetamide, (18)



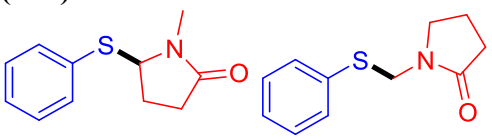
Reaction in MeCN (2 mL), using *N-methyl-N-phenylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 a light-yellow oil. (31.8 mg, 62% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39-7.28 (m, 4H), 7.24-7.11 (m, 6H), 5.21 (s, 2H), 1.82 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.4, 141.7, 134.6, 130.7, 129.6, 128.9, 128.5 and 128.4, 126.8, 53.1, 22.7.

HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{15}H_{16}NOS$ 258.0953, found 258.0947.

1-methyl-5-(phenylthio)pyrrolidin-2-one, (19a) and 1-((phenylthio)methyl)pyrrolidin-2-one (19b)



19a **19b**

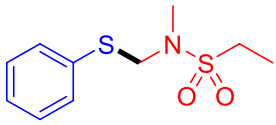
Known compound reported by reference 8.

Reaction in MeCN (2 mL), using *1-methylpyrrolidin-2-one* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a light-yellow oil, 19a (12.4 mg, 30% yield) and 19b (18.6 mg, 45% yield)

19a
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39-7.22 (m, 5H), 4.81 and 4.79 (d, 1H, *J* = 4.0 Hz), 2.98 (s, 3H), 2.53-2.41 (m, 1H), 2.22-2.05 (m, 2H), 1.74-1.64 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.5, 135.2, 129.3, 129.0, 69.6, 29.1, 28.1, 26.5.

19b
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51-7.40 (m, 2H), 7.37-7.25 (m, 3H), 4.77 (s, 2H), 3.44 (t, 2H, *J* = 7.1 Hz), 2.30 (t, 2H, *J* = 8.0 Hz), 2.04- 1.90 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.9, 133.7, 131.0, 129.1, 127.2, 46.8, 45.9, 30.9, 17.6.

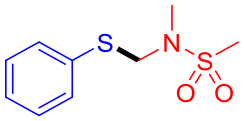
***N*-methyl-*N*-((phenylthio)methyl)ethanesulfonamide, (20)**



Reaction in MeCN (2 mL), using *N,N-dimethylethanesulfonamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a colorless oil, (32.3 mg, 66% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.38 (m, 2H), 7.35-7.29 (m, 3H), 4.88 and 4.83 (2xs, 2H), 3.00 and 2.98 (2xs, 3H), 2.86 (q, 2H *J* = 7.4 Hz), 1.41 and 1.30 (2xt, 3H, *J* = 7.4 Hz).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 133.9 and 133.2, 132.3, 129.3, 128.0, 56.7, 46.5, 34.2, 31.6, 7.9.
HRMS (ESI-TOF) m/z: [M + Na]+ calcd. for $C_{10}H_{15}NNaO_2S_2$ 268.0422, found 268.0436.

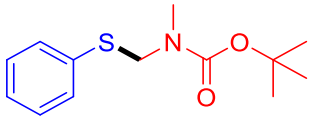
***N*-methyl-*N*-((phenylthio)methyl)methanesulfonamide, (21)**



Reaction in MeCN (2 mL), using *N,N-dimethylmethanesulfonamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/5 to 1/2 as a colorless oil, (34.6 mg, 75% yield)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.54 (m, 2H), 7.37- 7.29 (m, 3H), 4.91 and 4.86 (2xs, 2H), 2.97 and 2.93 (2xs, 3H), 2.79 and 2.72 (2xs, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 133.9, 132.2, 129.4, 128.0, 56.6, 38.2, 34.0.
HRMS (EI-QTOF) m/z: [M]+ calcd. for $C_9H_{13}NO_2S_2$ 231.0388, found 231.0386.

***tert*-butyl methyl((phenylthio)methyl)carbamate, (22)**



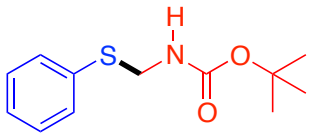
Known compound reported by reference 9.

Reaction in MeCN (2 mL), using *tert-butyl dimethylcarbamate* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/30 -1/10 as a colorless oil, (27.8 mg, 55% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42-7.40 (m, 2H), 7.23-7.19 (m, 3H), 4.71 and 4.62 (2xs, 2H), 2.84 and 2.79 (2xs, 3H), 1.29 and 1.15 (2xs, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 134.1, 133.8, 132.4, 129.0, 128.0, 127.3, 55.8, 54.2, 33.3 and 32.9, 28.2, and 28.0.

***tert*-butyl ((phenylthio)methyl)carbamate, (23)**



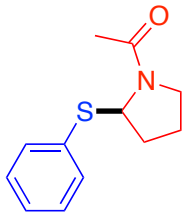
Known compound reported by reference 10.

Reaction in MeCN (2 mL), using *tert-butyl methylcarbamate* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/60 to 1/30 as a colorless oil, (21.5 mg, 45% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46-7.30 (m, 5H), 4.90 (s, 1H), 4.60 and 4.59 (2xs, 2H), 1.40 and 1.26 (2xs, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 154.9, 133.9, 131.7, 129.5 and 129.1, 127.4, 80.2, 45.8, 29.7, 28.3.

1-(2-(phenylthio)pyrrolidin-1-yl)ethan-1-one, (25)



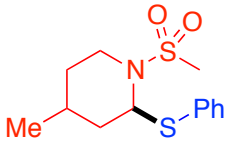
Known compound reported by reference 11.

Reaction in MeCN (2 mL), using *1-(pyrrolidin-1-yl)ethan-1-one* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/35 to 1/20 as a colorless oil, (31.8 mg, 72% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.46 (m, 3H), 7.33-7.26 (m, 2H), 5.19-5.13 (t, 1H, *J* = 4 Hz), 3.54- 3.48 (m, 2H), 2.19 (m, 2H), 2.07 (s, 3H), 2.05 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 134.5, 134.0, 133.0, 129.3, 128.9, 128.6, 127.8, 67.8 and 65.4, 47.4, 45.7, 34.3, 33.0, 23.4, 22.7 and 22.2, 21.5.

4-methyl-2-(phenylthio)-1-tosylpiperidine, (27)

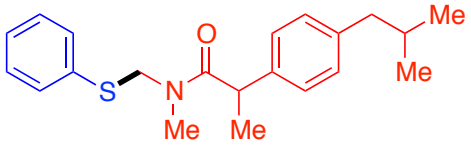


Reaction in MeCN (2 mL), using *4-methyl-1-tosylpiperidine* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/200 to 1/100 as a colorless solid, (46.9 mg, 65% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (dd, 2H, *J* = 12.0 Hz), 7.30 (dd, 3H, *J* = 10.5, 7.0 Hz), 5.75- 5.56 (t, 1H, *J* = 4.0 Hz), 3.78- 3.63 (m, 1H), 3.25 (td, 1H, *J* = 12.6, 2.7 Hz), 2.62 (s, 3H), 2.11- 1.94 (m, 2H), 1.76 (dt, 1H, *J* = 13.8, 3.1 Hz), 1.60 (td, 1H, *J* = 12.9, 4.6 Hz), 1.29 (ddt, 2H, *J* = 18.0, 13.3, 5.9 Hz), 0.96 (d, 3H, *J* = 6.5 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 133.6, 133.3, 129.3, 128.0, 65.5, 41.3, 40.3, 38.8, 33.6, 25.6, 21.6.
HRMS (ESI-TOF) m/z: [M + Na]+ calcd. for $C_{13}H_{19}NO_2S_2Na$ 308.0755, found 308.0749.

2-(4-isobutylphenyl)-N-methyl-N-((phenylthio)methyl)propanamide, (28)

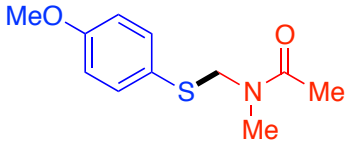


Reaction in MeCN (2 mL), using *2-(4-isobutylphenyl)-N,N-dimethylpropanamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/20 to 1/10 as a colourless solid, (49.1 mg, 72% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51- 7.20 (m, 5H), 7.04-6.97 (m, 4H), 5.00, 4.94 and 4.81 (d, 2H, *J* = 16.0 Hz), 4.16 (d, 1H, *J* = 12.0 Hz), 3.73 and 3.24 (q, 1H, *J* = 6.8 Hz), 2.97 and 2.83 (2xs, 3H), 2.42 (dd, 3H, *J* = 7.3, 4.4 Hz), 1.92-1.77 (m, 2H), 1.33 and 1.31 (2xs, 2H), 1.11 and 1.09 (2xs, 1H), 0.89 and 0.87 (2xs, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 174.1 and 174. 0, 140.3 and 140.1, 138.8 and 138.4, 134.8, 133.7, 132.8 and 132.1, 129.6 and 129.5, 129.4, 128.9 and 128.8, 127.3 and 127.0, 126.8, 56.5, 52.6, 45.0, 43.3, 42.5, 34.4, 33.5, 30.2 and 30.2, 22.4, 20.6 and 20.6.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{21}H_{28}NOS$ 342.1892, found 342.1886.

***N*-(((4-methoxyphenyl)thio)methyl)-*N*-methylacetamide, (29)**

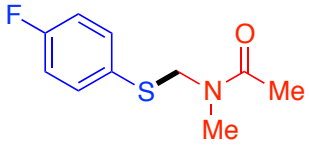


Known compound reported by reference 7.

Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (33.7 mg, 75% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60-7.34 (m, 2H), 7.08- 6.74 (m, 2H), 4.76 and 4.55 (2 xs, 2H), 3.79 and 2.97 (2 xs, 6H), 1.99 and 1.60 (2 xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.5 an d170.5, 160.6, 159.7, 137.1, 134.9, 124.2, 122.7, 114.9, 114.6, 58.2, 55.3 and 55.3, 53.2, 35.3, 32.7, 21.8, 20.6.

***N*-(((4-fluorophenyl)thio)methyl)-N-methylacetamide, (30)**

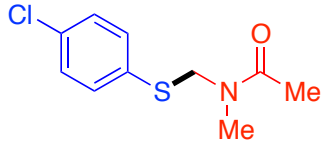


Known compound reported by reference 8.

Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (36.2 mg, 85% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64-7.39 (m, 2H), 7.02 (m, 2H), 4.87, 4.83 and 4.71 and 4.61 (2 xs, 2H), 3.08, 3.02 and 3.00, 2.97 (2 xs, 3H), 2.01 and 1.66 (2 xs, 3H).
^{19}F NMR (376 MHz, $CDCl_3$) δ -108.9, -110.0, -111.0, -111.2, -113.9.
^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.7 and 170.5, 164.7, 163.8, 161.3, 137.4 and 137.3, 134.5 and 134.5, 128.8 and 128.8, 127.6, 116.7 and 116.5, 116.2, 116.0, 58.1 and 58.1, 52.5, 35.2, 32.8, 21.8, 20.7.

***N*-(((4-chlorophenyl)thio)methyl)-*N*-methylacetamide, (31)**

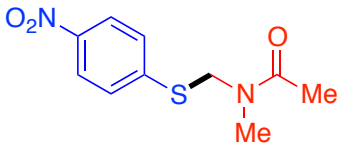


Known compound reported by reference 7.

Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (32.4 mg, 71% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40-7.32 (m, 2H), 7.30-7.24 (m, 2H), 4.87 and 4.64 (2 xs, 2H), 3.00 and 2.96 (2 xs, 3H), 2.02 and 1.71 (2 xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7 and 170.4, 136.1, 135.4, 133.4, 132.8 and 132.5, 129.6, and 129.1, 57.8, 51.6, 35.1, 32.9, 21.8, 20.8.

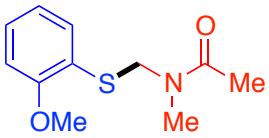
_N_-methyl-_N_-(((4-nitrophenyl)thio)methyl)acetamide, (32)



Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (21.6 mg, 45% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.12 (d, 2H, *J* = 8.0 Hz), δ 7.55 (d, 2H, *J* = 8.0 Hz), 5.05 and 4.85 (2xs, 2H), 3.06 and 2.98 (2xs, 3H), 2.08 and 1.96 (2xs, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.1, 146.0, 144.2, 132.2, 128.5, 124.2, 124.0, 56.6, 49.5, 34.9, 33.3, 31.6, 29.7, 22.7, 21.9, 21.1, 14.1.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{10}H_{13}N_2O_3S$ 241.0647, found 241.0641.

_N_-(((2-methoxyphenyl)thio)methyl)-_N_-methylacetamide, (33)

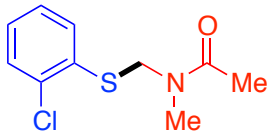


Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (39.6 mg, 88% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46-7.25 (m, 2H), 6.93-6.85 (m, 2H), 4.87 and 4.67 (2 xs, 2H), 3.91 (s, 3H), 2.99 and 2.95 (2 xs, 3H), 1.97 and 1.77 (2 xs, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7 and 170.6, 159.9, 158.7, 137.0, 133.6, 131.0, 129.2, 121.5, 121.3 and 121.0, 119.7, 111.1, 110.9, 55.9 and 55.8, 55.5, 50.2, 35.2, 33.0, 21.8, 20.5.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{11}H_{16}NO_2S$ 226.0902, found 226.0896.

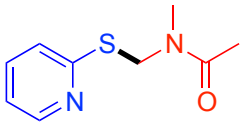
***N*-(((2-chlorophenyl)thio)methyl)-*N*-methylacetamide, (34)**



Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (32.9 mg, 72% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59–7.15 (m, 4H), 4.73 and 4.71 (2 xs, 2H), 3.05 and 3.03 (2 xs, 3H), 1.78 and 2.05 (2 xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8 and 170.5, 138.9, 137.1, 135.5, 133.2, 132.6, 131.1, 130.5 and 130.3, 129.8, 128.3, 127.6 and 127.3, 56.2, 50.7, 35.2, 33.1, 21.9, 20.5.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{10}H_{13}ClNOS$ 230.0406, found 230.0402.

***N*-methyl-*N*-((pyridin-2-ylthio)methyl)acetamide, (35)**

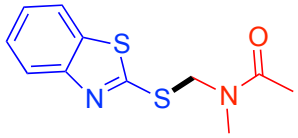


Known compound reported by reference 7.

Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (26.7 mg, 68% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.45-8.40 (m, 1H), 7.55-7.52 (m, 1H), 7.25-7.18 (m, 1H), 7.06-6.98 (m, 1H), 5.29 and 5.26 (2 xs, 2H), 3.09 and 2.97 (2 xs, 3H), 1.98 and 1.77 (2 xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 171.0, 157.9, 156.4, 149.4 and149.2, 136.5 and 136.3, 123.4, 122.7, 120.5 and 120.0, 51.5, 47.2, 35.9, 33.1, 21.9, 21.6.

***N*-((benzo[d]thiazol-2-ylthio)methyl)-*N*-methylacetamide, (36)**

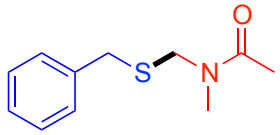


Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (30.2 mg, 60% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (d, 1H, J = 8.0 Hz), 7.47 (d, 1H, J = 8.0 Hz), 7.41-7.27 (m, 2H), 6.22 (s, 2H), 3.09 and 2.15 (2 xs, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 192.0, 172.0, 140.8, 127.6, 126.6, 125.3, 121.0, 113.9, 54.3, 34.6, 21.9.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{11}H_{13}N_2OS_2$ 253.0469, found 253.0464.

***N*-((benzylthio)methyl)-*N*-methylacetamide, (37)**

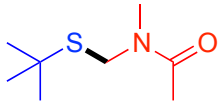


Known compound reported by reference 8.

Reaction in MeCN (2 mL), using *N,N*-dimethylacetamide (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (26.2 mg, 63% yield).
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38−7.22 (m, 5H), 4.57 and 4.34 (2 xs, 2H), 3.78 and 3.76 (2xs, 2H), 2.93 and 2.91 (2xs, 3H), 1.99 and 1.98 (2xs, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1 and 170.5, 138.8, 128.8 and 128.8, 128.7 and 128.4, 128.4, 127.6, 126.9, 52.4, 49.2, 35.8 and 35.1, 34.7, 21.9 and 21.2.

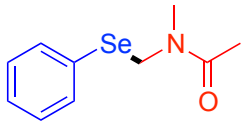
***N*-((*tert*-butylthio)methyl)-*N*-methylacetamide, (38)**



Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a colorless oil, (15.7 mg, 45% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.66 and 4.46 (2 xs, 2H), 3.04 and 2.98 (2 xs, 3H), 2.20 and 2.08 (2 xs, 3H), 1.37 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 50.2, 46.4, 43.3, 34.4, 31.6, 31.2, 22.7, 22.3, 14.1.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_8H_{18}NOS$ 176.1109, found 176.1103.

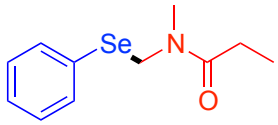
***N*-methyl-*N*-((phenylselanyl)methyl)acetamide, (39)**



Reaction in MeCN (2 mL), using *N,N-dimethylacetamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a light-yellow oil, (32 mg, 66% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65-7.61 (m, 2H), 7.38-7.27 (m, 3H), 4.94 and 4.78 (2 xs, 2H), 2.95 (s, 3H), 2.01 and 1.68 (2 xs, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 136.4, 134.4, 129.4, 129.1, 128.8, 127.7, 50.3, 46.3, 36.1, 33.3, 21.8, 20.7.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{10}H_{14}NOSe$ 244.0241, found 244.0235.

***N*-methyl-*N*-((phenylselanyl)methyl)propionamide, (40)**

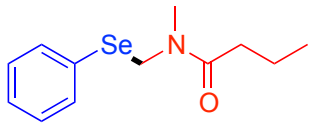


Reaction in MeCN (2 mL), using *N,N-dimethylpropionamide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a light-yellow oil, (25.6 mg, 50% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63-7.57 (m, 2H), 7.36-7.23 (m, 3H), 4.97 and 4.80 (2 xs, 2H), 2.95 and 2.94 (2xs, 3H), 2.26 and 1.94 (q, 2H, J = 16 Hz), 1.06 and 0.92 (t, 3H, J = 12 Hz),

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.6, 173.6, 136.3, 134.5, 129.3, 129.2, 129.1, 128.7, 127.6, 49.3, 46.7, 35.2, 33.6, 26.9, 25.8, 9.1, 9.0.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{11}H_{16}NOSe$ 258.0397, found 258.0392.

***N*-methyl-*N*-((phenylselanyl)methyl)butyramide, (41)**

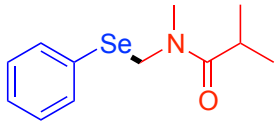


Reaction in MeCN (2 mL), using *N,N-dimethylbutyramide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a light-yellow oil, (27.5 mg, 51% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.49 (m, 2H), 7.29- 7.18 (m, 3H), 4.96 and 4.80 (2 xs, 2H), 2.95 (s, 3H), 2.04 and 1.81 (dt, 2H, J = 12.0, 12.0 Hz), 1.51and 1.37 (dq, 2H, J = 20.0, 20.0 Hz), 0.84 and 0.69 (dt, 3H, J = 12.0, 12.0Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.8, 136.3, 134.5, 129.3, 129.2, 129.1, 128.7, 127.7, 127.6, 49.5, 46.6, 35.5, 35.3, 34.5, 33.4, 18.2, 13.9, 13.8.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{12}H_{18}NOSe$ 272.0554, found 272.0550.

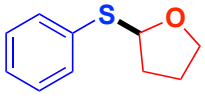
_N_-methyl-_N_-((phenylselanyl)methyl)isobutyramide, (42)



Reaction in MeCN (2 mL), using *N,N-dimethylisobutyramide* (1.0 mmol, 5 equiv). After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=1/10 to 1/3 as a light-yellow oil, (26.0 mg, 48% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.52 (m, 2H), 7.26-7.19 (m, 3H), 4.97 and 4.84 (2 xs, 2H), 3.00 and 2.96 (2 xs, 3H), 2.66-2.59 and 2.34-2.27 (m, 1H), 0.96 (dd, 6H, *J* = 8.0, 8.0 Hz).
^{13}C NMR (101 MHz, $CDCl_3$) δ 177.2, 176.9, 136.3, 134.6, 129.4, 129.1, 129.0, 128.7, 127.6, 127.5, 49.0, 46.7, 35.1, 33.6, 30.5, 30.1, 19.3, 19.0.
HRMS (ESI-TOF) m/z: [M + H]+ calcd. for $C_{12}H_{18}NOSe$ 272.0554, found 272.0550

2-(phenylthio)tetrahydrofuran, (44)

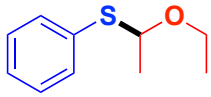


Known compound reported by reference 12.

Reaction in THF (2 mL), After workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=0/1 to 1/200 as a colorless oil, (25.2 mg, 70% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64-7.47 (m, 2H), 7.33-7.21 (m, 3H), 5.65 (dd, 1H, *J* = 7.2, 3.8 Hz), 4.12- 3.88 (m, 2H), 2.39-2.34 (m, 1H), 2.16- 1.83 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 135.7, 131.1, 128.8, 126.8, 87.2, 67.3, 32.7, 24.9.

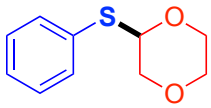
(1-ethoxyethyl)(phenyl)sulfane, (45)



Known compound reported by reference 13.
Reaction in Et_2O (2 mL), after workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=0/1 to 1/200 as a colorless oil, (24.0 mg, 66% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47-7.44 (m, 2H), 7.25-7.23 (m, 3H), 4.8 (q, 1H, *J* = 6.5 Hz), 3.50-3.42 (m, 2H), 1.48 (d, 3H, *J* = 6.5 Hz), 1.2 (t, 3H, *J* = 6.8 Hz)
^{13}C NMR (101 MHz, $CDCl_3$) δ 133.8, 128.7, 127.5, 84.5, 63.4, 22.7, 14.9.

2-(phenylthio)-1,4-dioxane, (46)

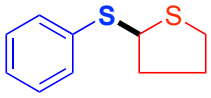


Known compound reported by reference 13.

Reaction in MeCN (2 mL), 1,4-dixoane (5 equiv, 1 mmol) after workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=0/1 to 1/200 as a colorless oil, (29.4 mg, 75% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52-7.48 (m, 2H), 7.34-7.27 (m, 3H), 5.12 (dd, 1H, *J* = 5.7, 3.0 Hz), 4.31-4.17 (m, 1H), 4.00 (dd, 1H, *J* = 11.7, 3.0 Hz), 3.75-3.66 (m, 4H)
^{13}C NMR (101 MHz, $CDCl_3$) δ 133.0, 130.6, 128.0, 126.4, 82.3, 69.0, 65.5, 62.8.

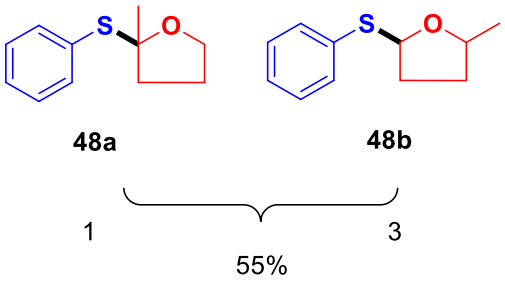
2-(phenylthio)tetrahydrothiophene, (47)



Known compound reported by reference 14.
Reaction in MeCN (2 mL), tetrahydrothiophene (5 equiv, 1 mmol) after workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=0/1 to 1/200 as a colorless oil, (31.4 mg, 80% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61- 7.23 (m, 5H), 4.86 (s, 1H), 2.96 (m, 2H), 2.16 (m, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 136.3, 131.3, 129.0, 127.1, 55.6, 38.4, 33.1, 28.8.

2-(phenylthio)tetrahydrothiophene, (48)



Reaction in 2-methyl tetrahydrofuran (2 mL), after workup, this product was isolated by flash column chromatography on silica gel, eluting with ethyl acetate/hexane=0/1 to 1/200 as a colorless oil, 49a:49b=0.3:1, 21.5 mg, 55% yield. Inseparable mixture of diastereomers and regioisomers

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52−7.49 (m, 2H), 7.39-7.30 (m, 4H), 7.29-7.16 (m, 4H), 5.70-5.67 (m, 1H), 4.32-4.30 (m, 1H), 4.24- 4.00 (m, 2H), 2.46-2.36 (m, 1H), 2.35-2.25 (m, 1H), 2.10-1.95 (m, 3H), 1.75-1.61 (m, 1H), 1.49-1.47 (m, 1H), 1.46-1.41 (m, 3H), 1.26 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 136.1, 135.5, 135.0, 134.7, 131.8, 131.5, 131.0, 130.8, 130.7, 129.5, 129.4, 129.3, 129.2, 128.8, 128.6, 128.5, 128.2, 128.1, 127.0, 126.9, 126.8, 126.6, 126.5, 87.1, 74.4, 33.6, 33.1, 32.5, 22.1, 20.1.

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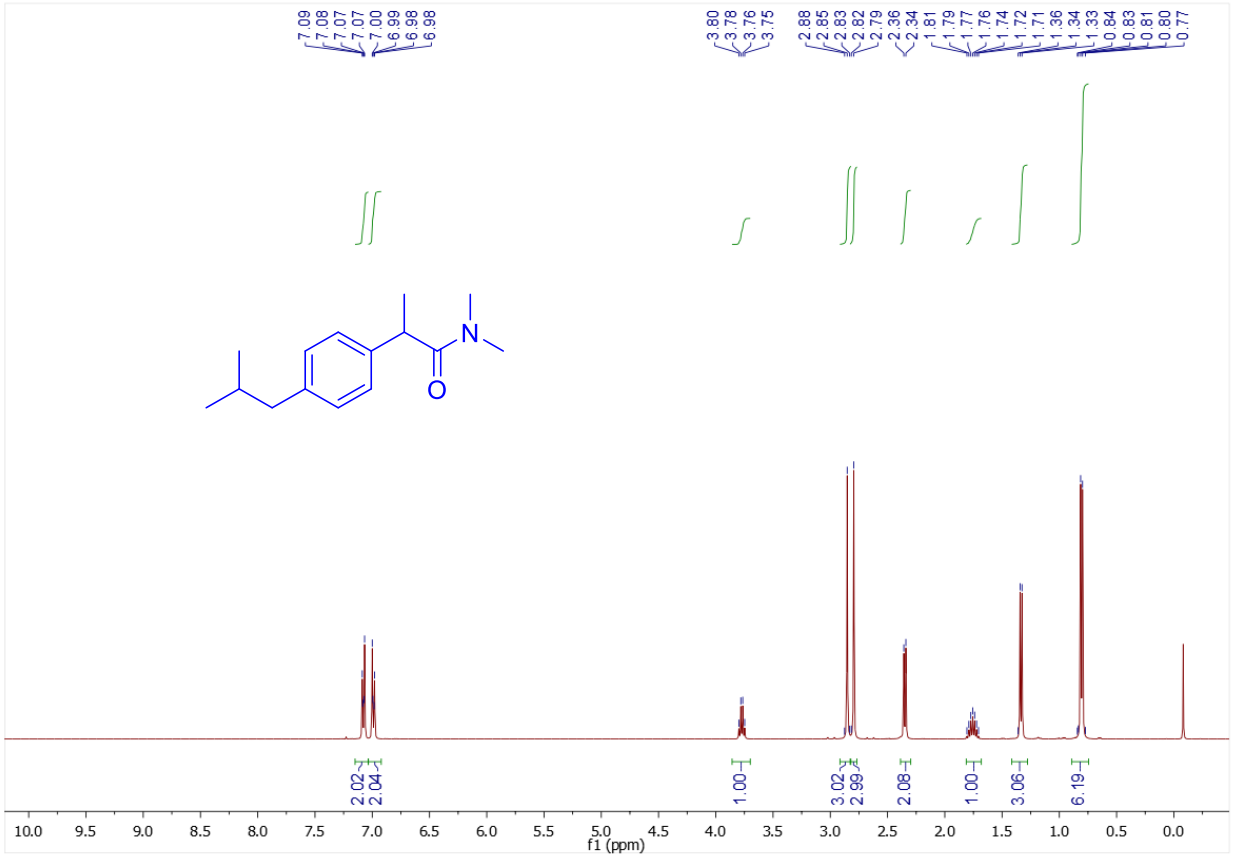
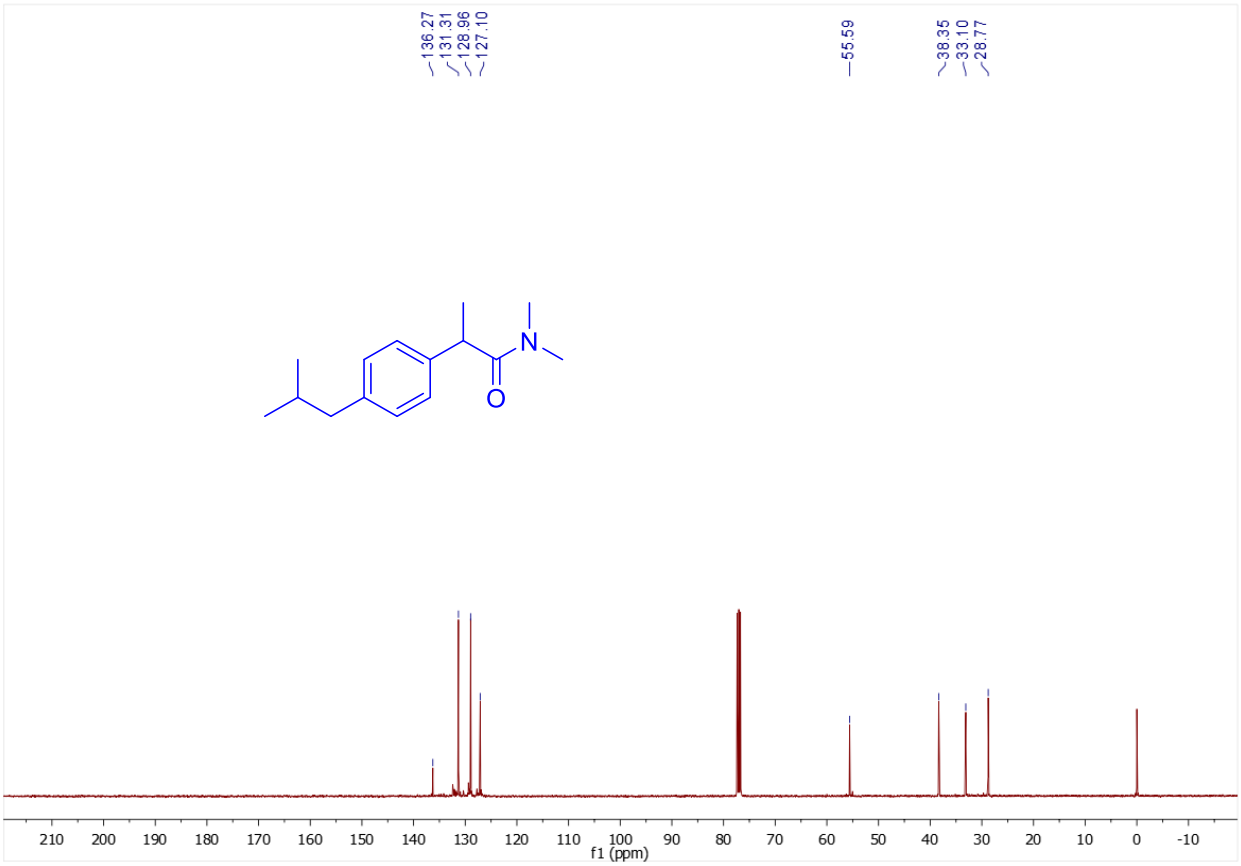
NMR Spectra

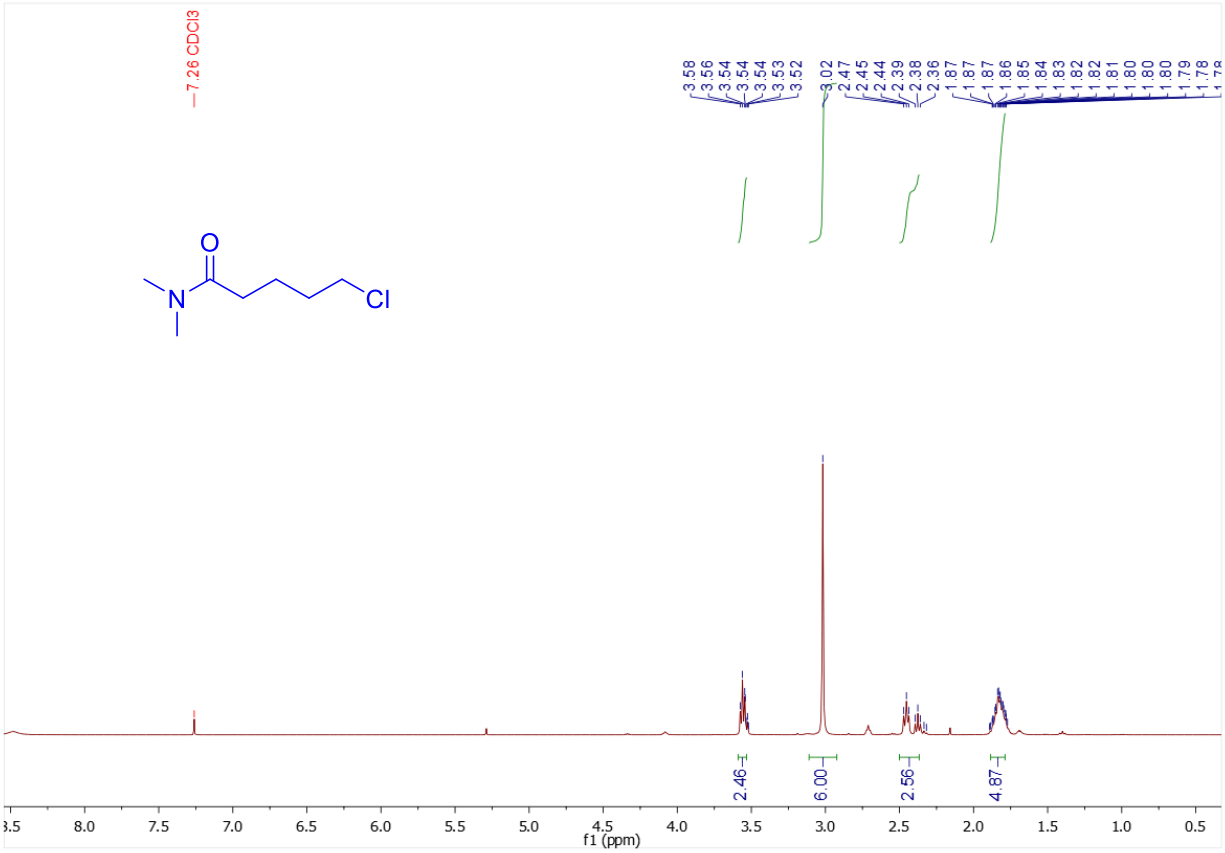
^{1}H-NMR (400 MHz, $CDCl_3$) of **2-(4-isobutylphenyl)-N,N-dimethylpropanamide**



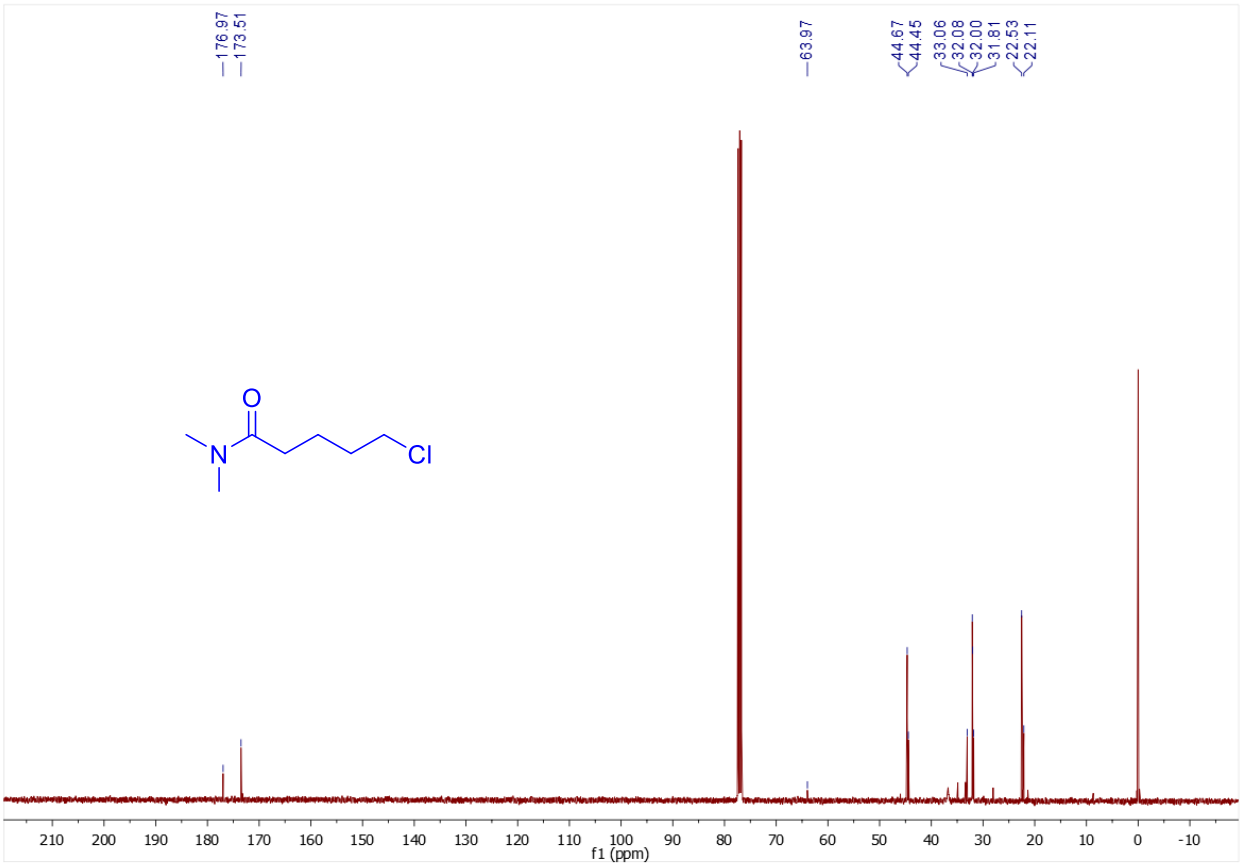
^{13}C-NMR (101 MHz, $CDCl_3$) of **2-(4-isobutylphenyl)-N,N-dimethylpropanamide**



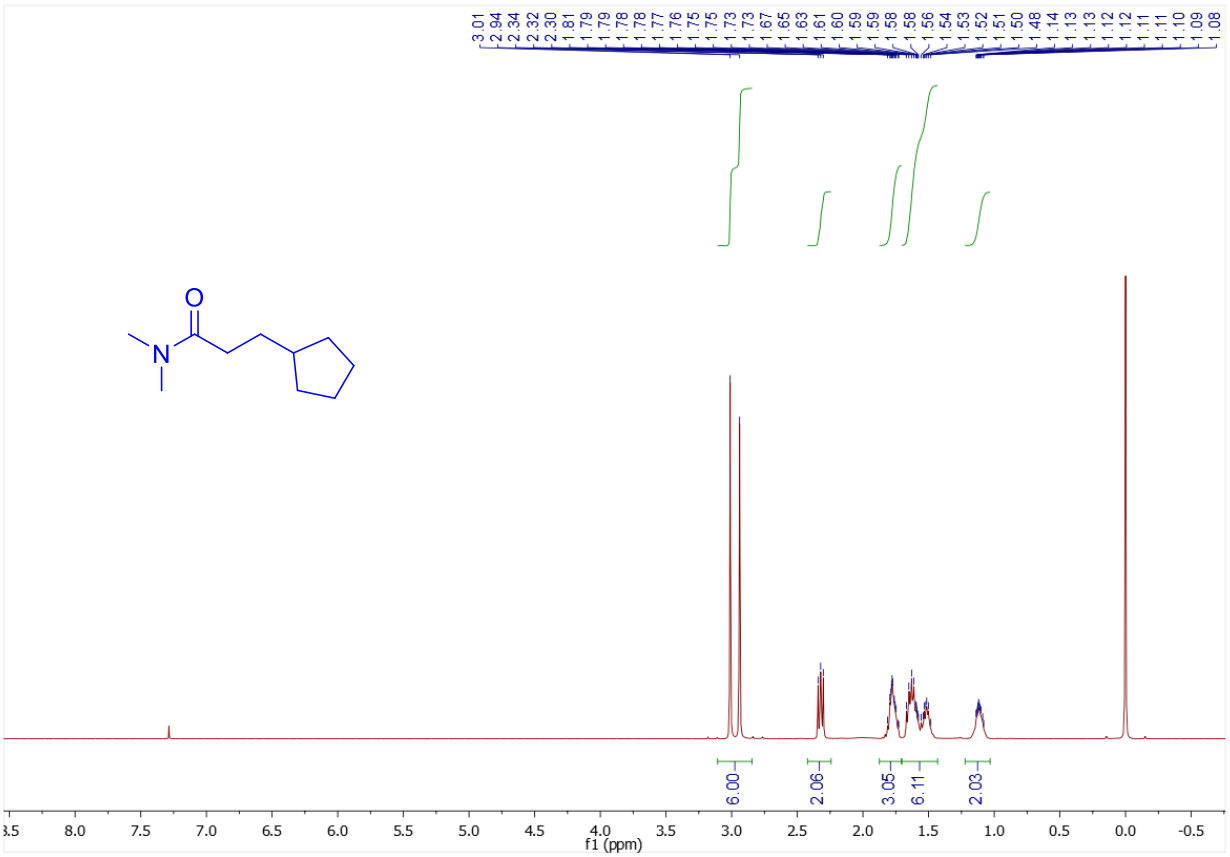
^{1}H-NMR (400 MHz, $CDCl_3$) of **5-chloro-N,N-dimethylpentanamide**



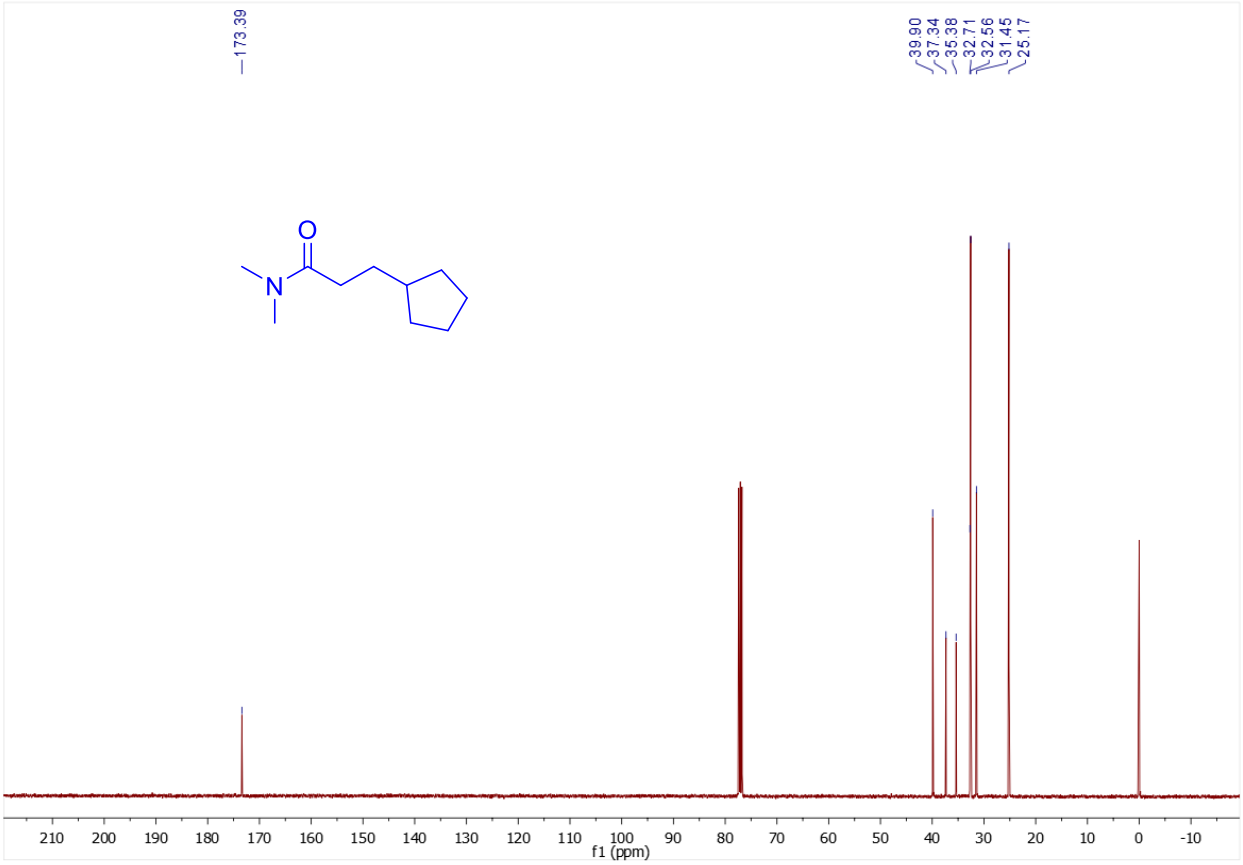
^{13}C-NMR (101 MHz, $CDCl_3$) of **5-chloro-N,N-dimethylpentanamide**



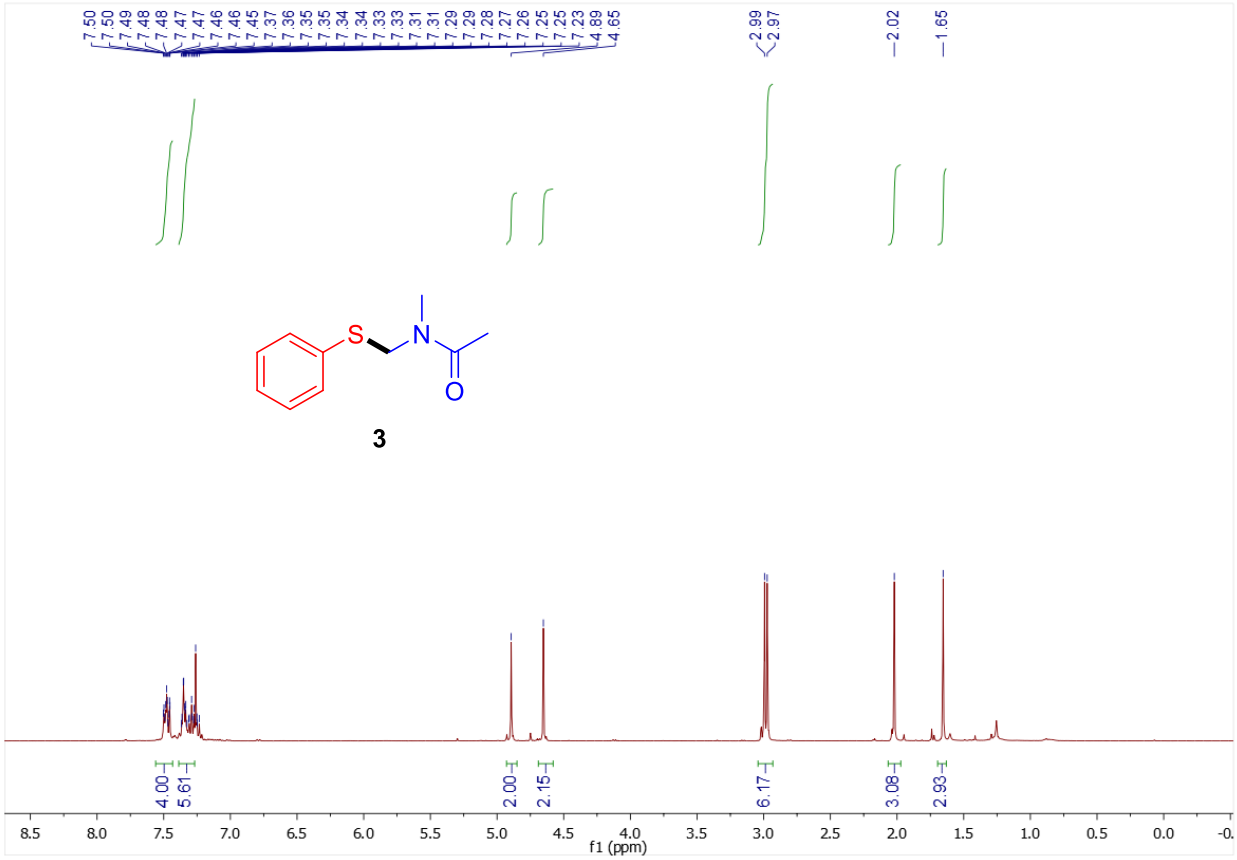
^{1}H-NMR (400 MHz, $CDCl_3$) of **3-cyclopentyl-N,N-dimethylpropanamide**



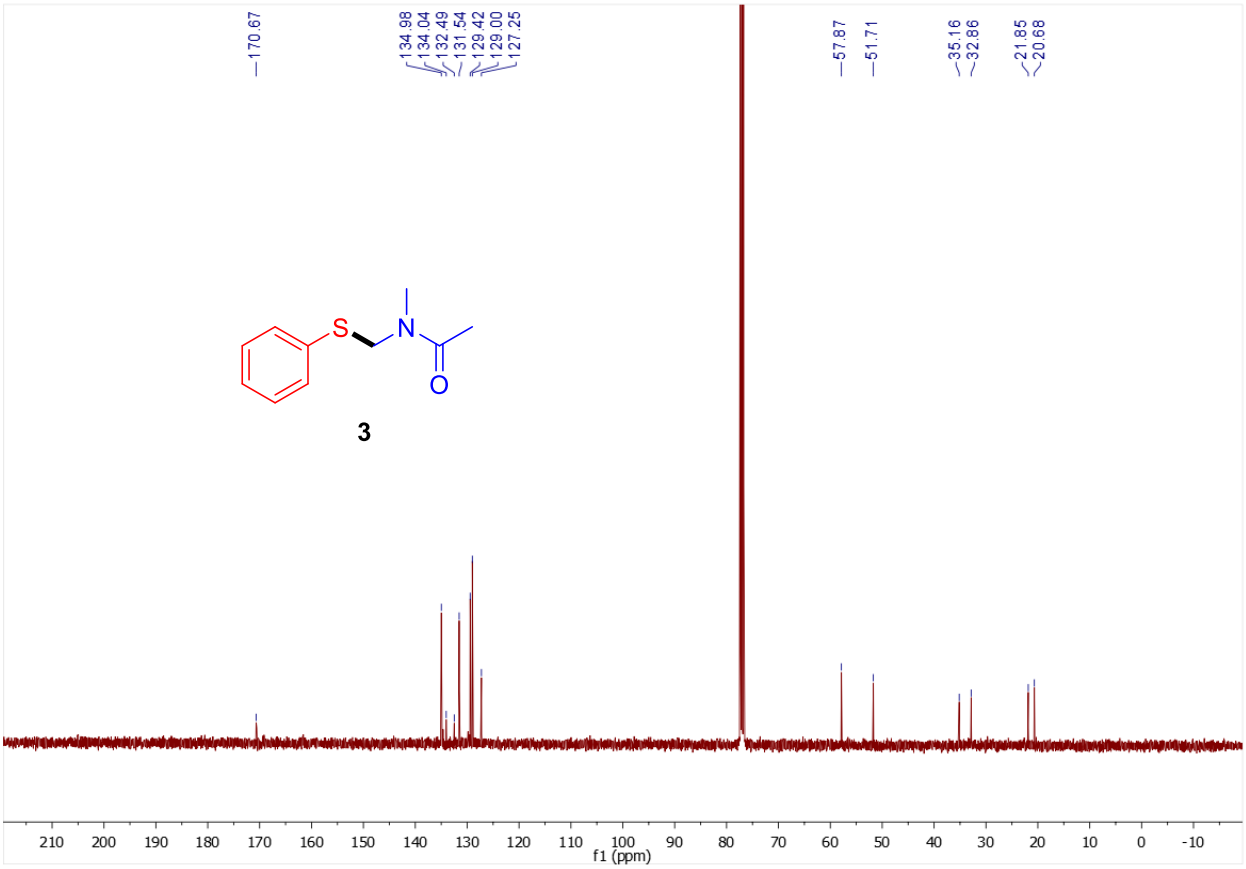
^{13}C-NMR (101 MHz, $CDCl_3$) of **3-cyclopentyl-N,N-dimethylpropanamide**



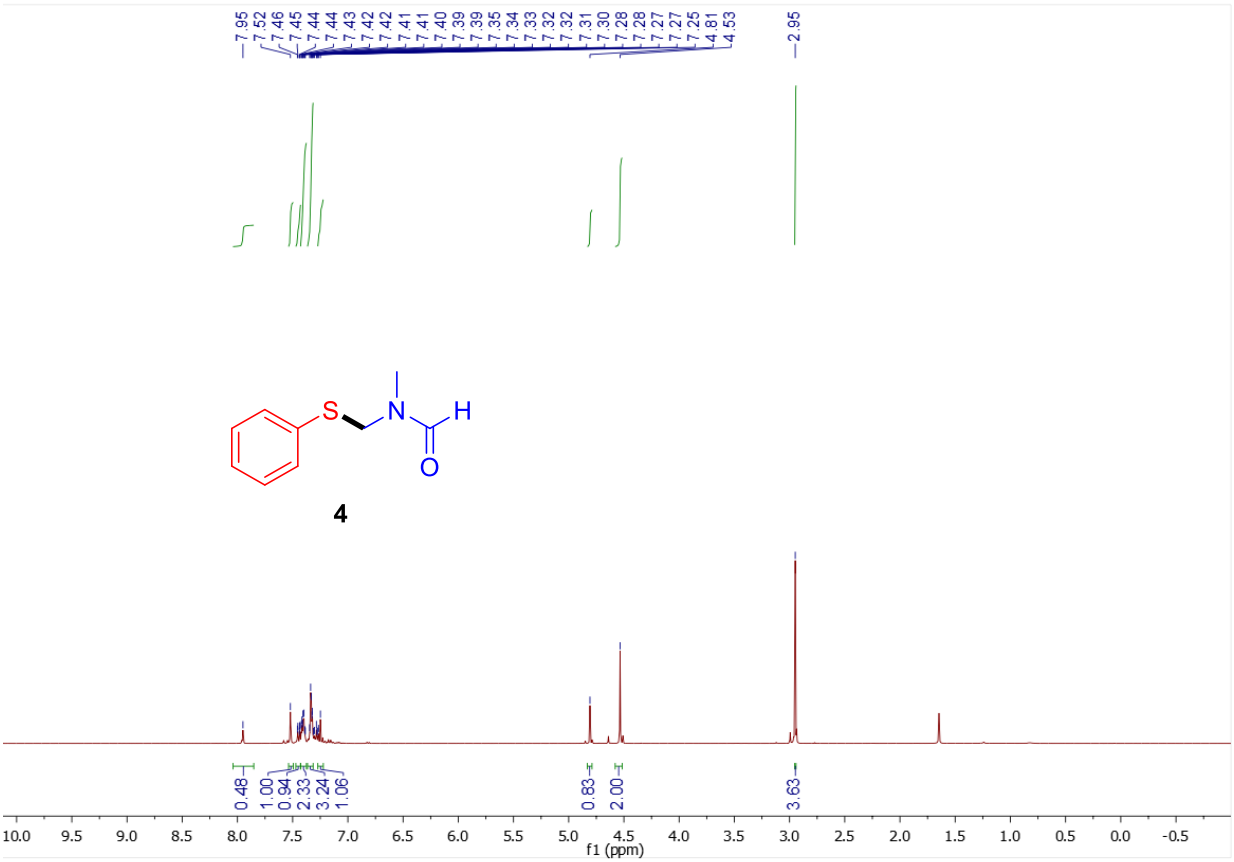
^{1}H-NMR (400 MHz, $CDCl_3$) of **3**



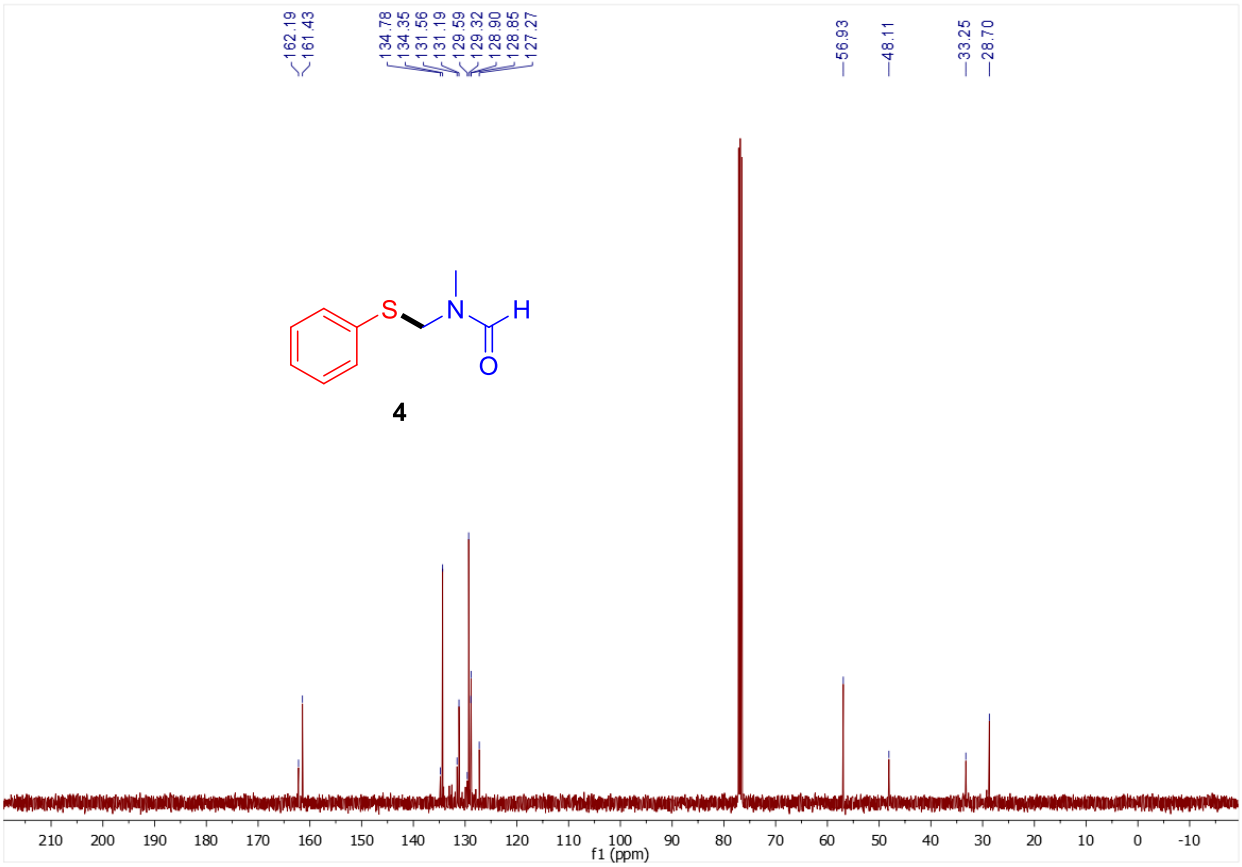
^{13}C-NMR (101 MHz, $CDCl_3$) of **3**



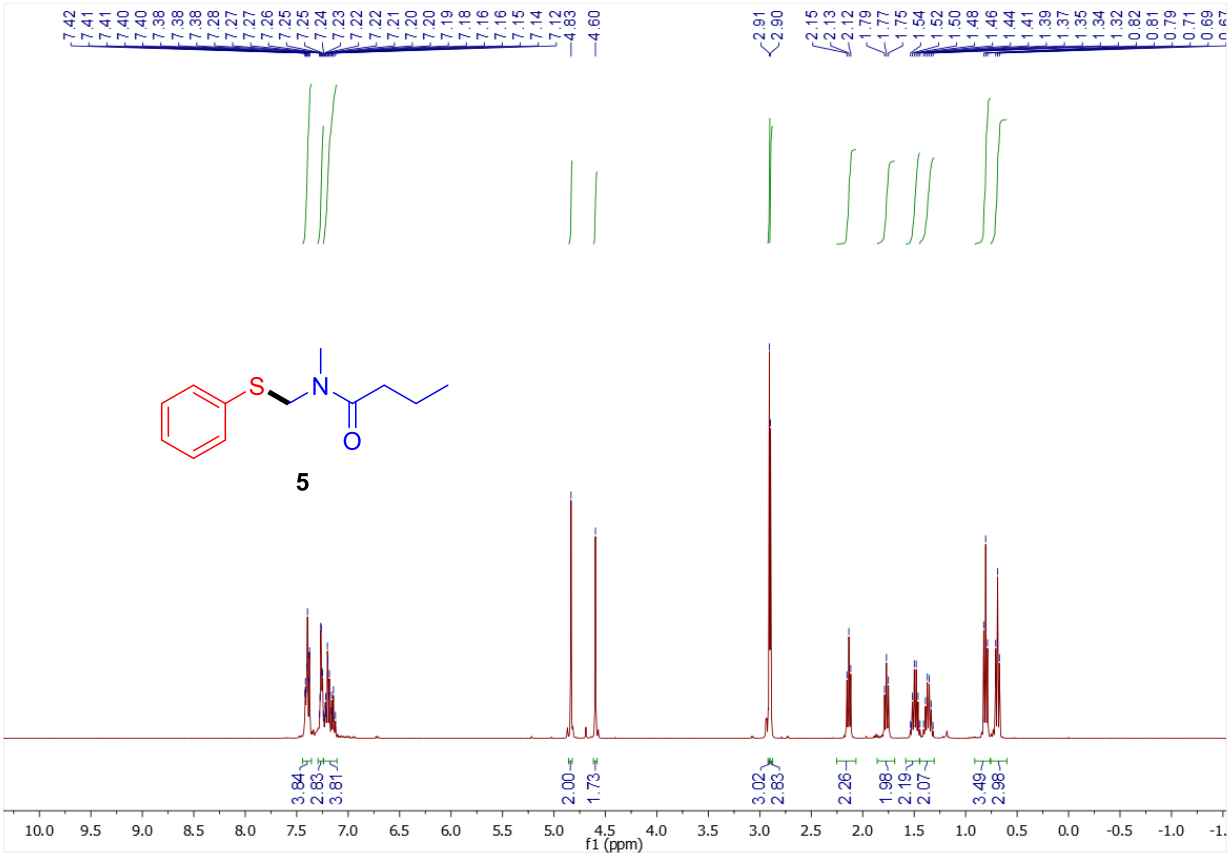
^{1}H-NMR (400 MHz, $CDCl_3$) of **4**



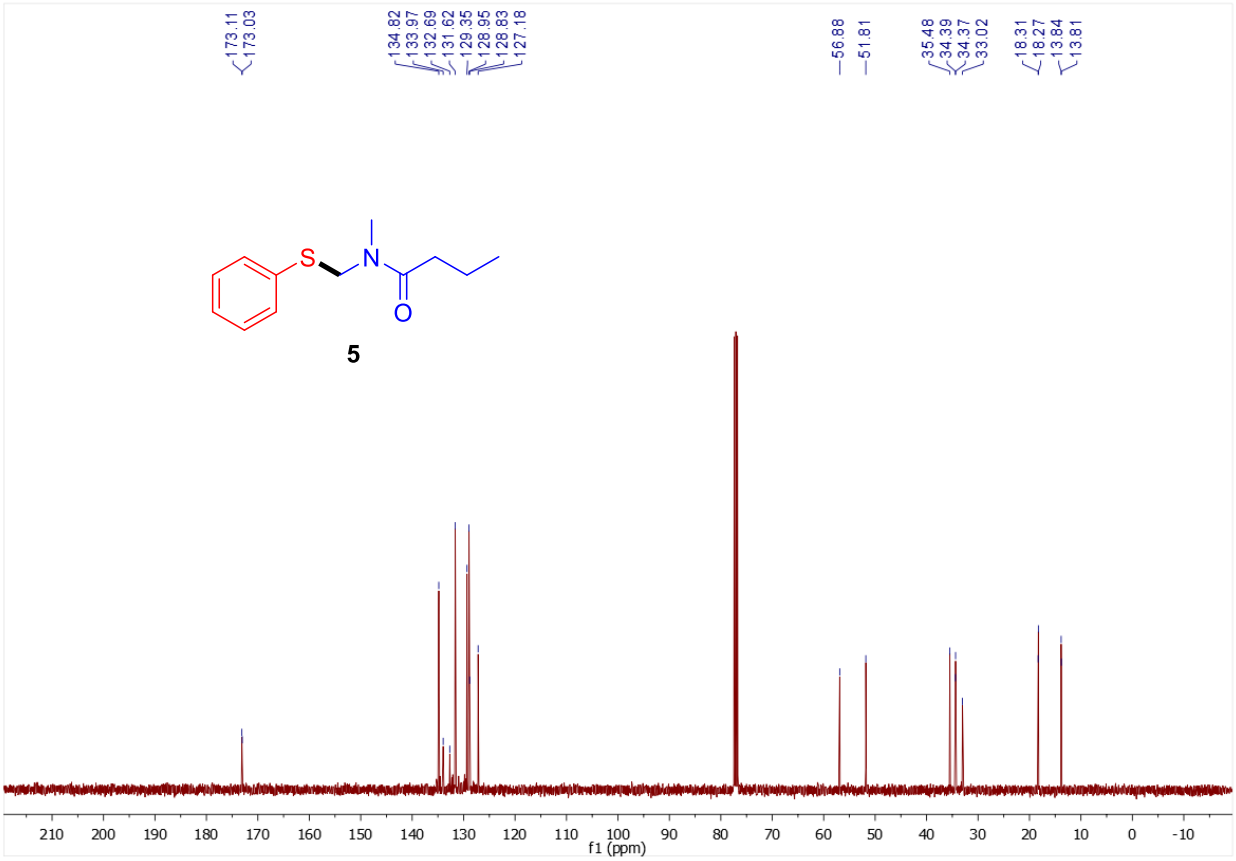
^{13}C-NMR (101 MHz, $CDCl_3$) of **4**



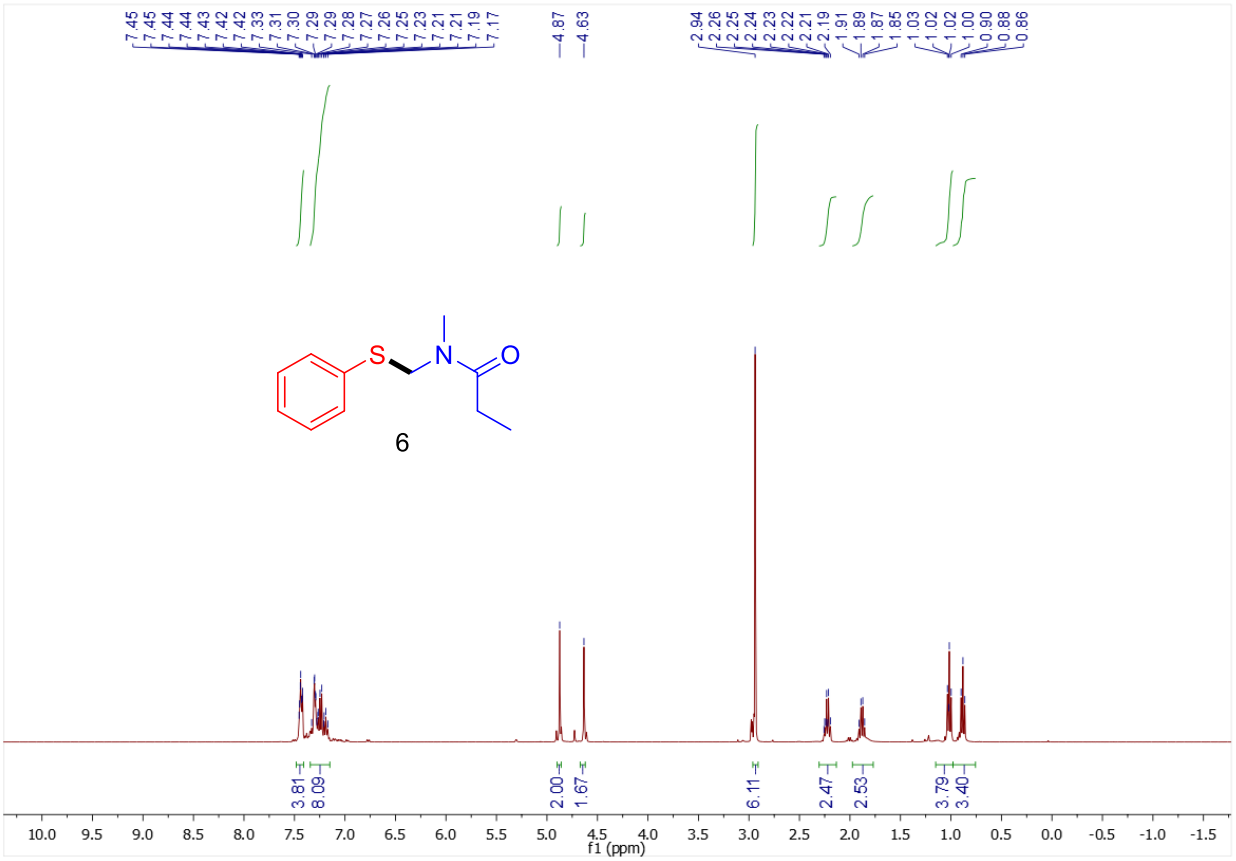
^{1}H-NMR (400 MHz, $CDCl_3$) of **5**



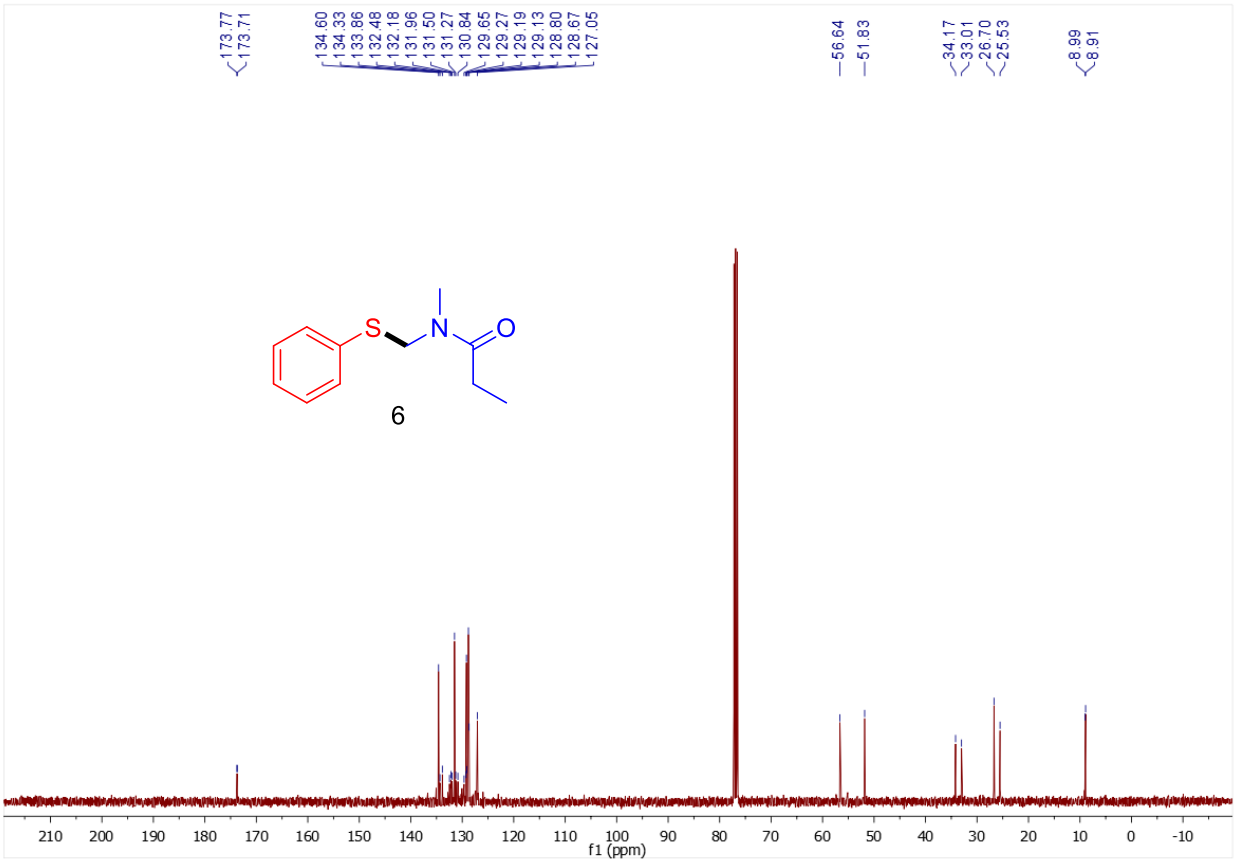
^{13}C-NMR (101 MHz, $CDCl_3$) of **5**



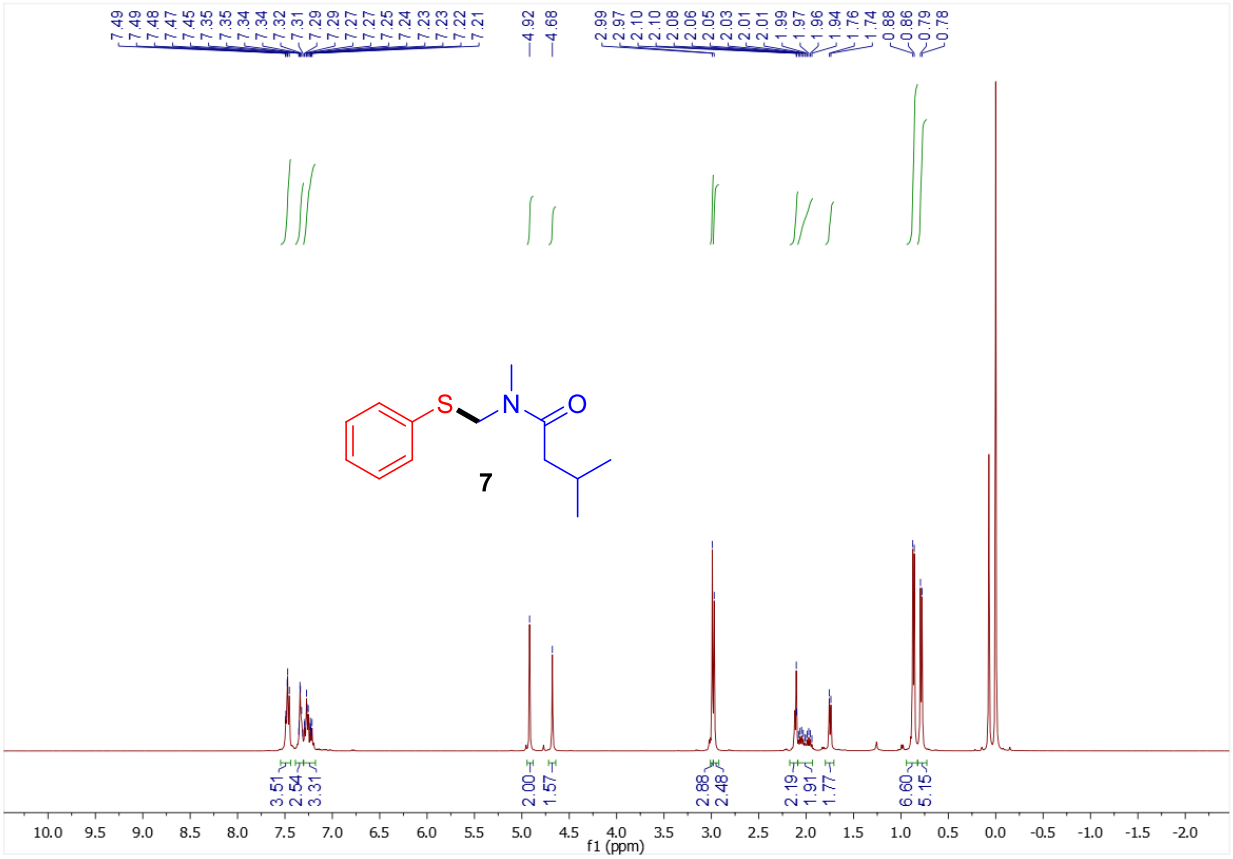
^{1}H-NMR (400 MHz, $CDCl_3$) of **6**



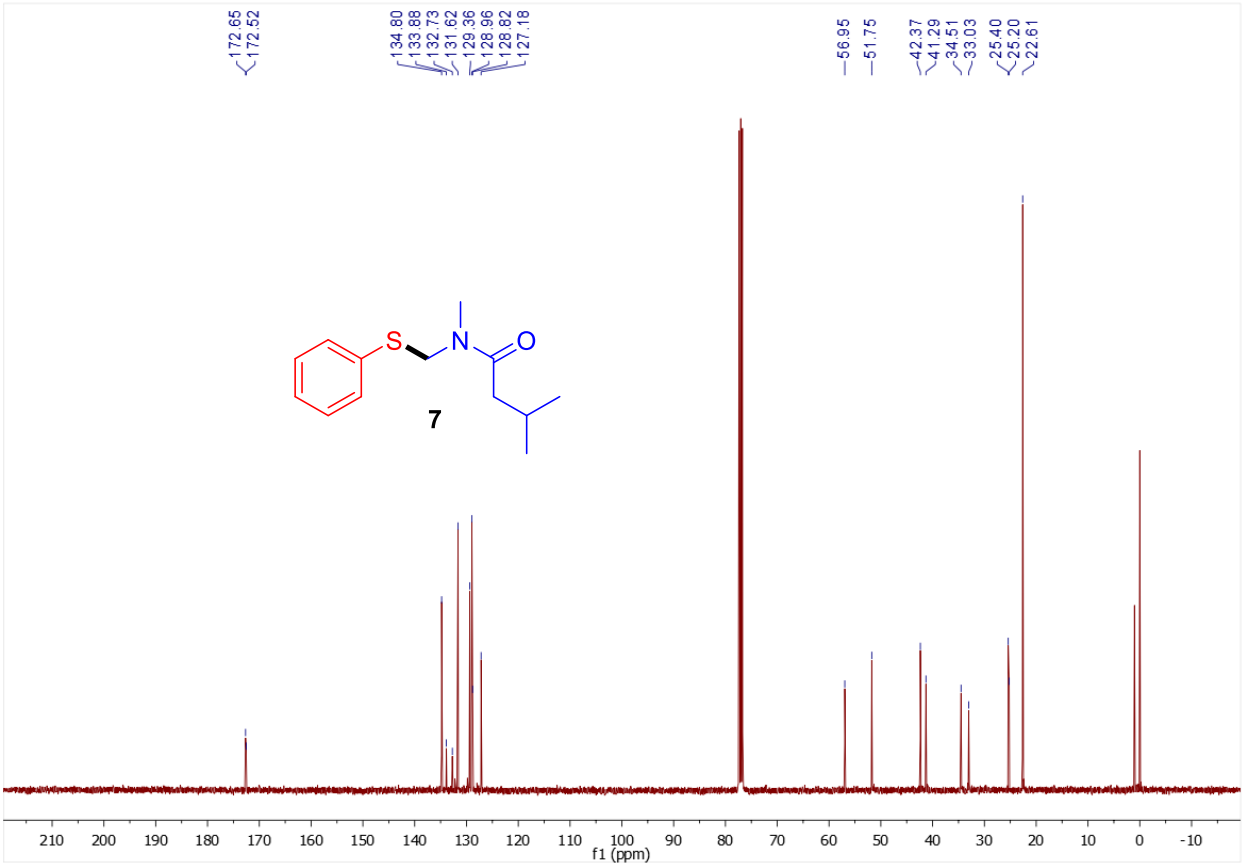
^{13}C-NMR (101 MHz, $CDCl_3$) of **6**



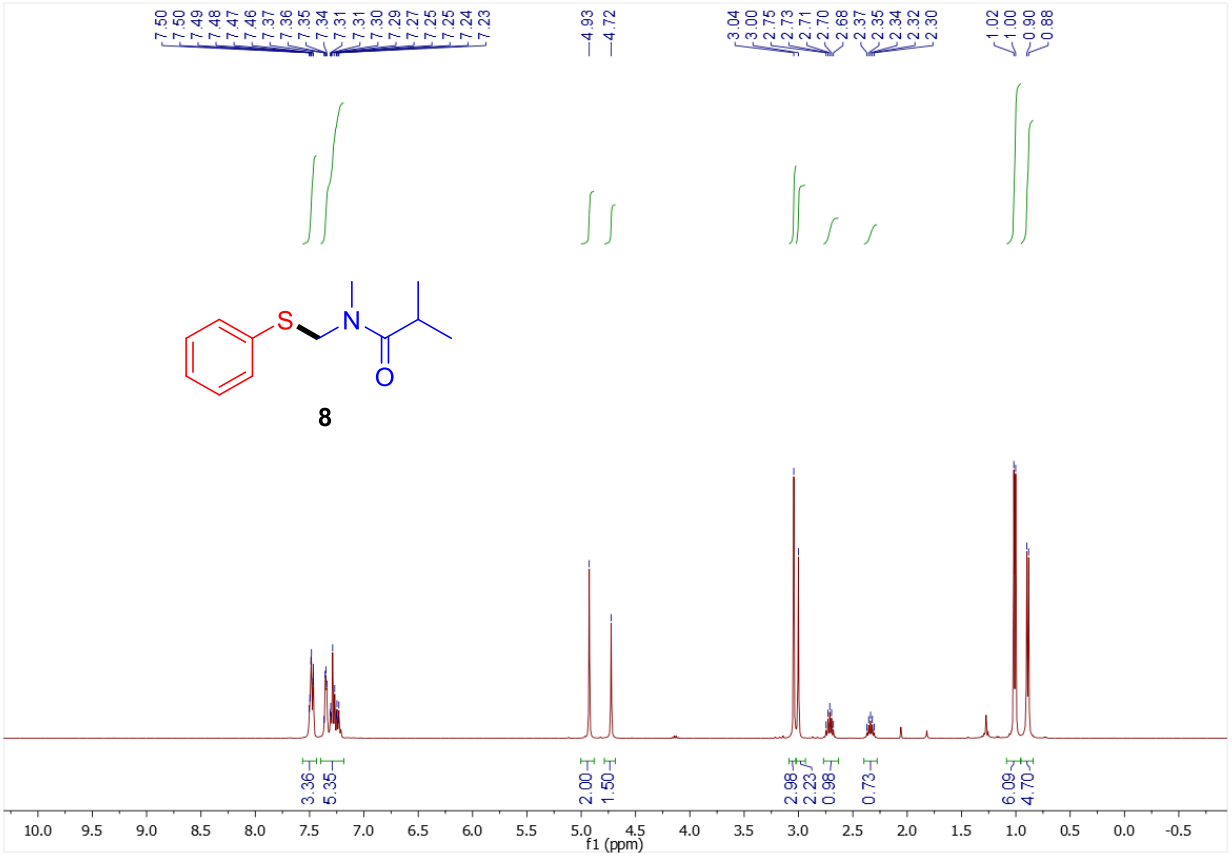
^{1}H-NMR (400 MHz, $CDCl_3$) of **7**



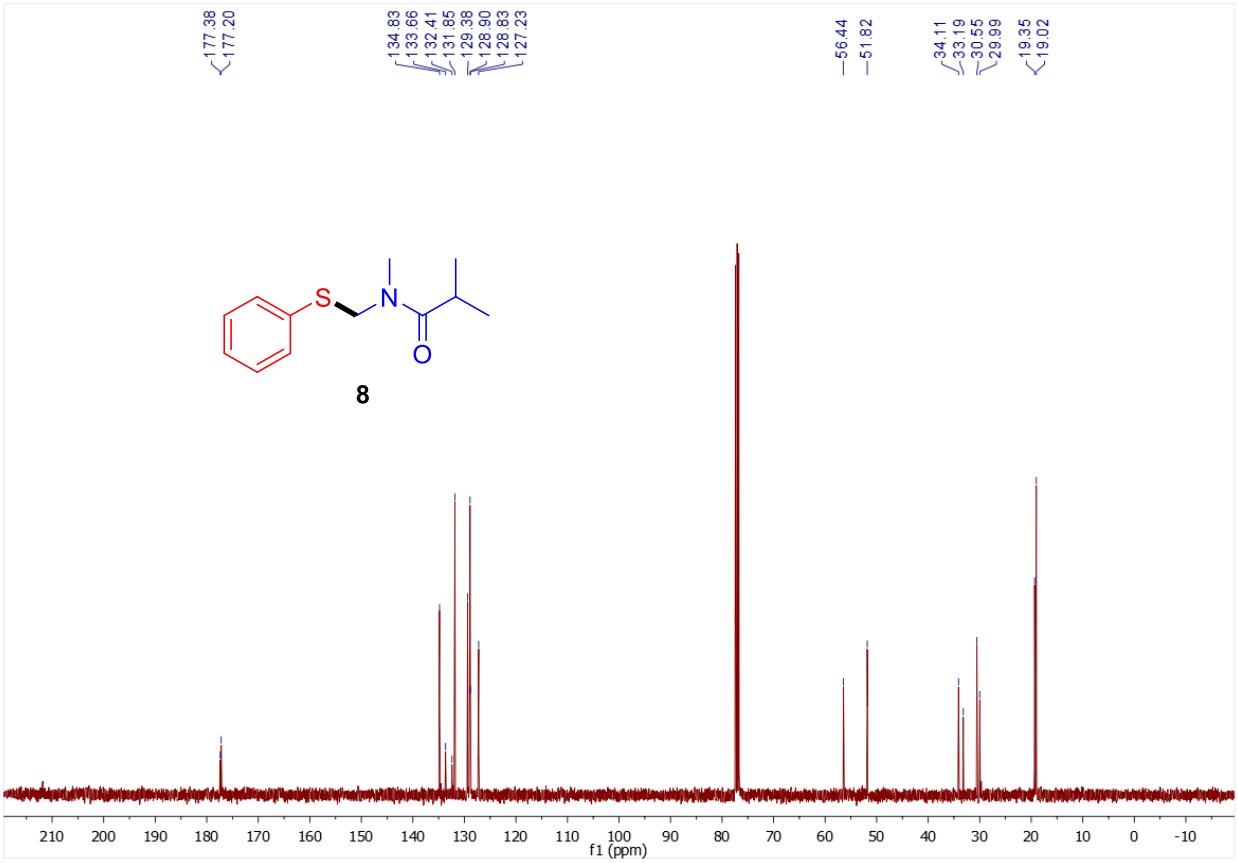
^{13}C-NMR (101 MHz, $CDCl_3$) of **7**



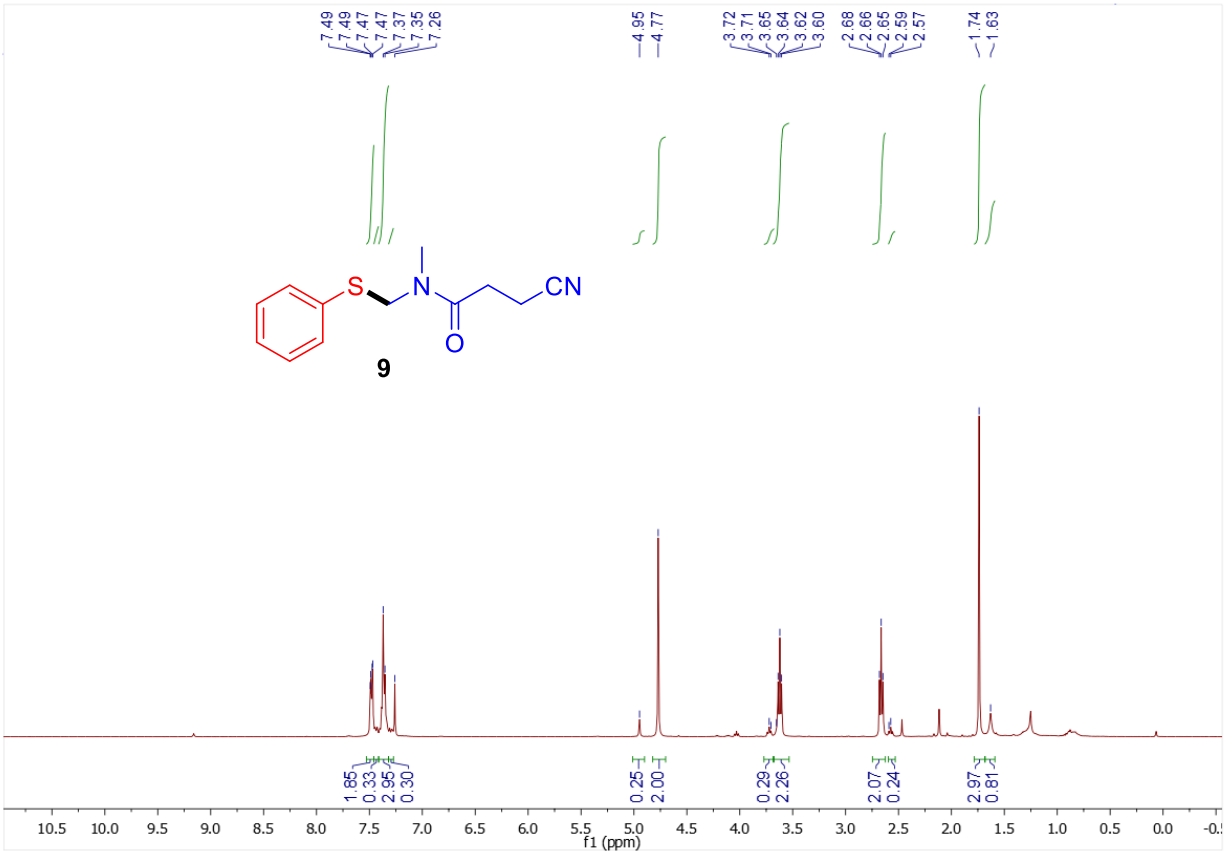
^{1}H-NMR (400 MHz, $CDCl_3$) of **8**



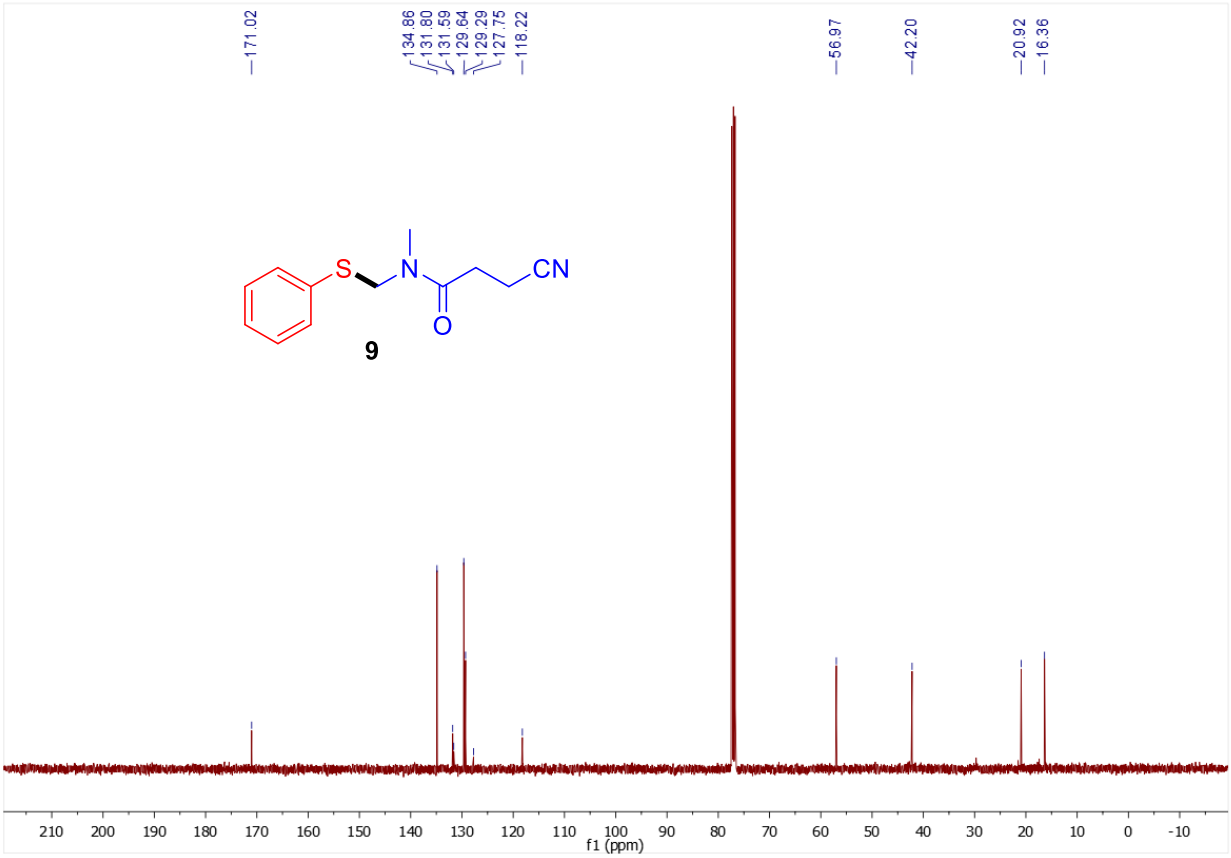
^{13}C-NMR (101 MHz, $CDCl_3$) of **8**



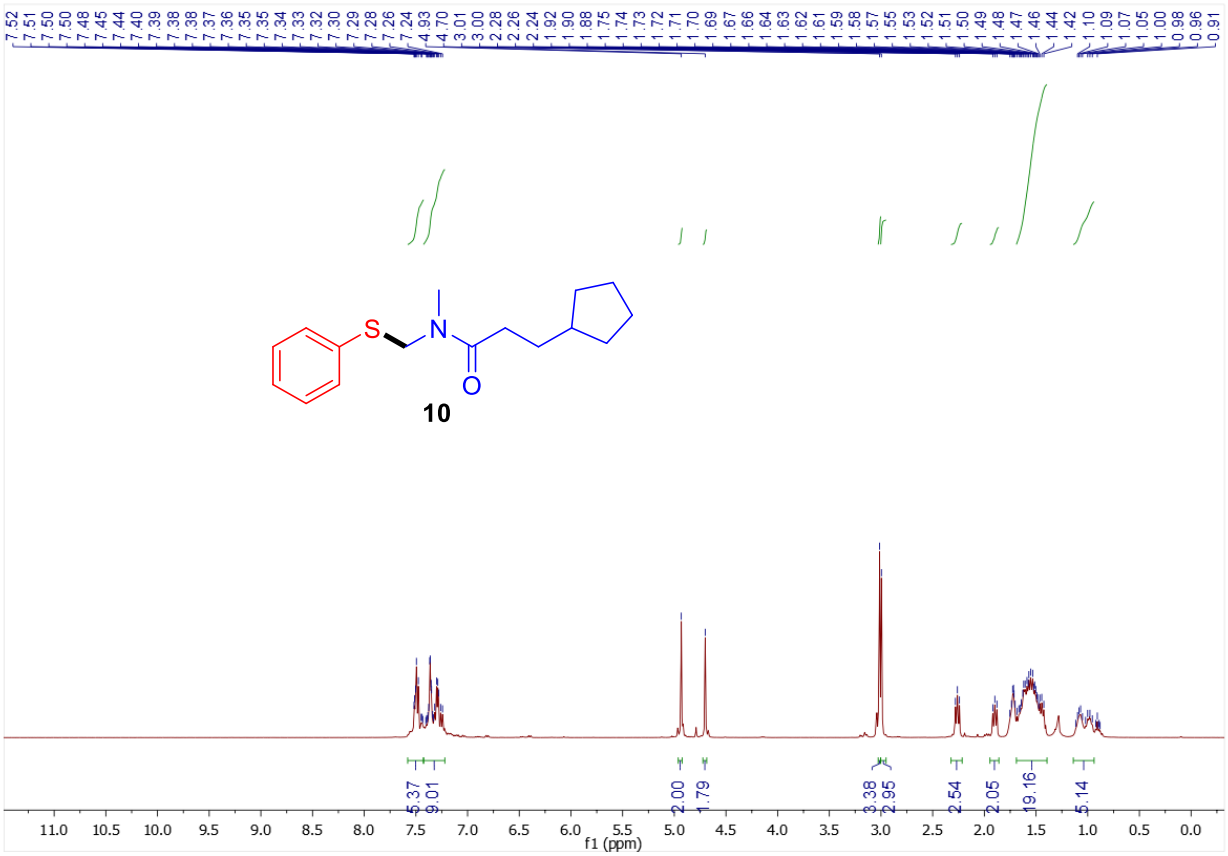
^{1}H-NMR (400 MHz, $CDCl_3$) of **9**



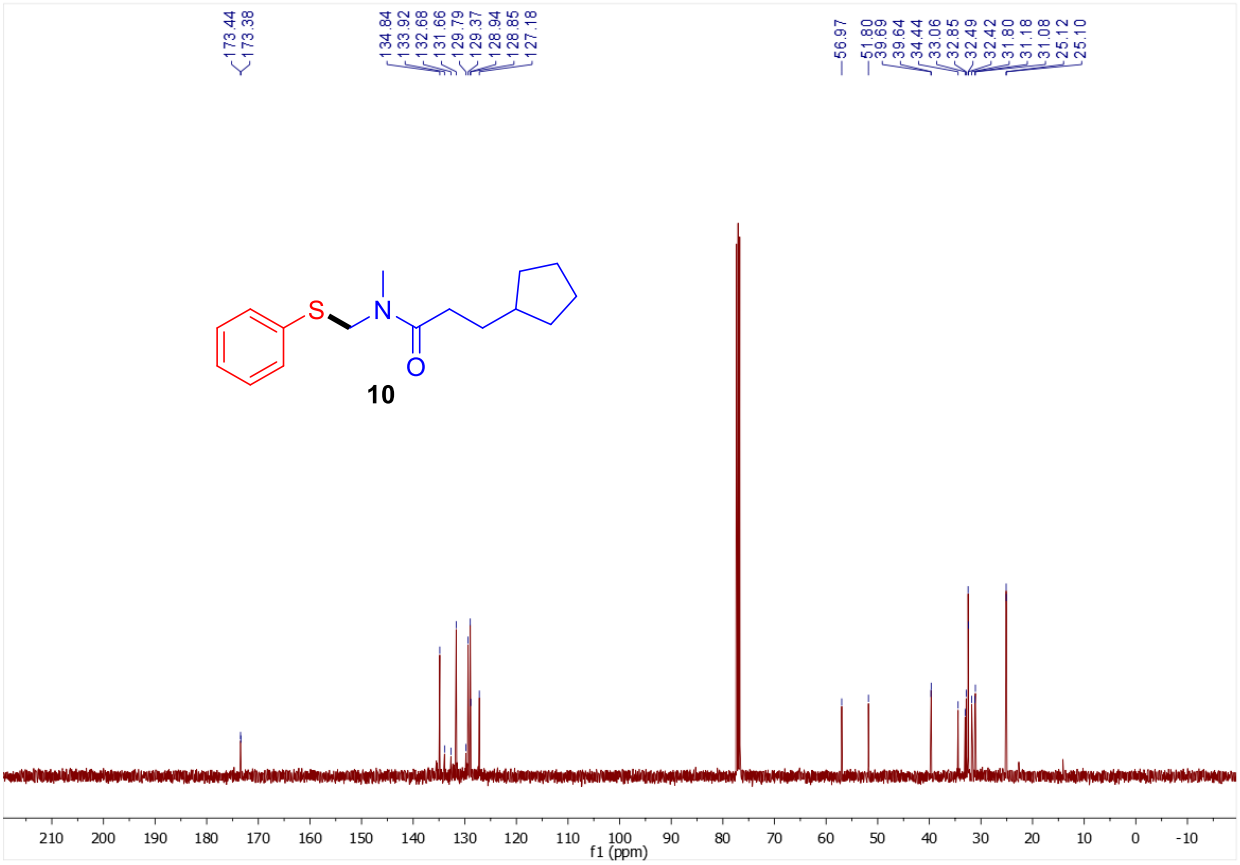
^{13}C-NMR (101 MHz, $CDCl_3$) of **9**



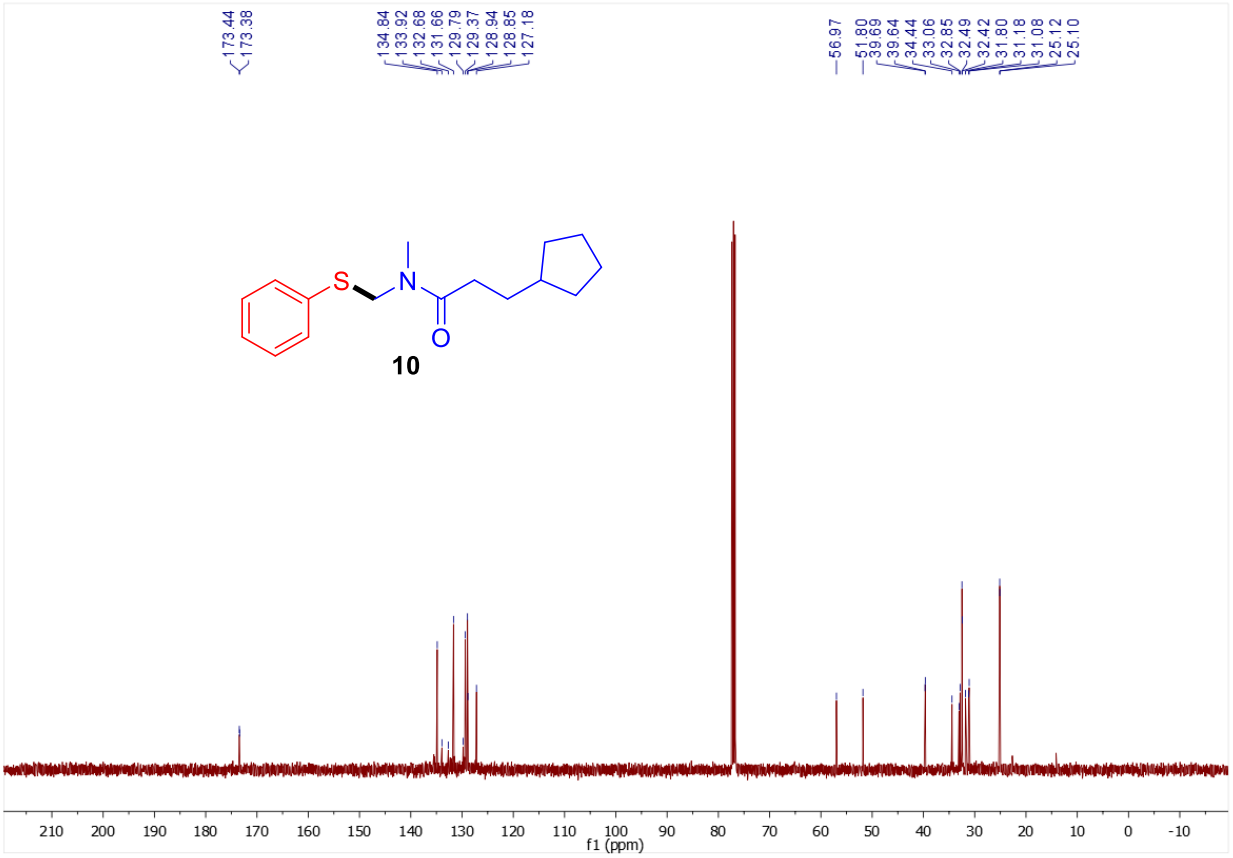
^{1}H-NMR (400 MHz, $CDCl_3$) of **10**



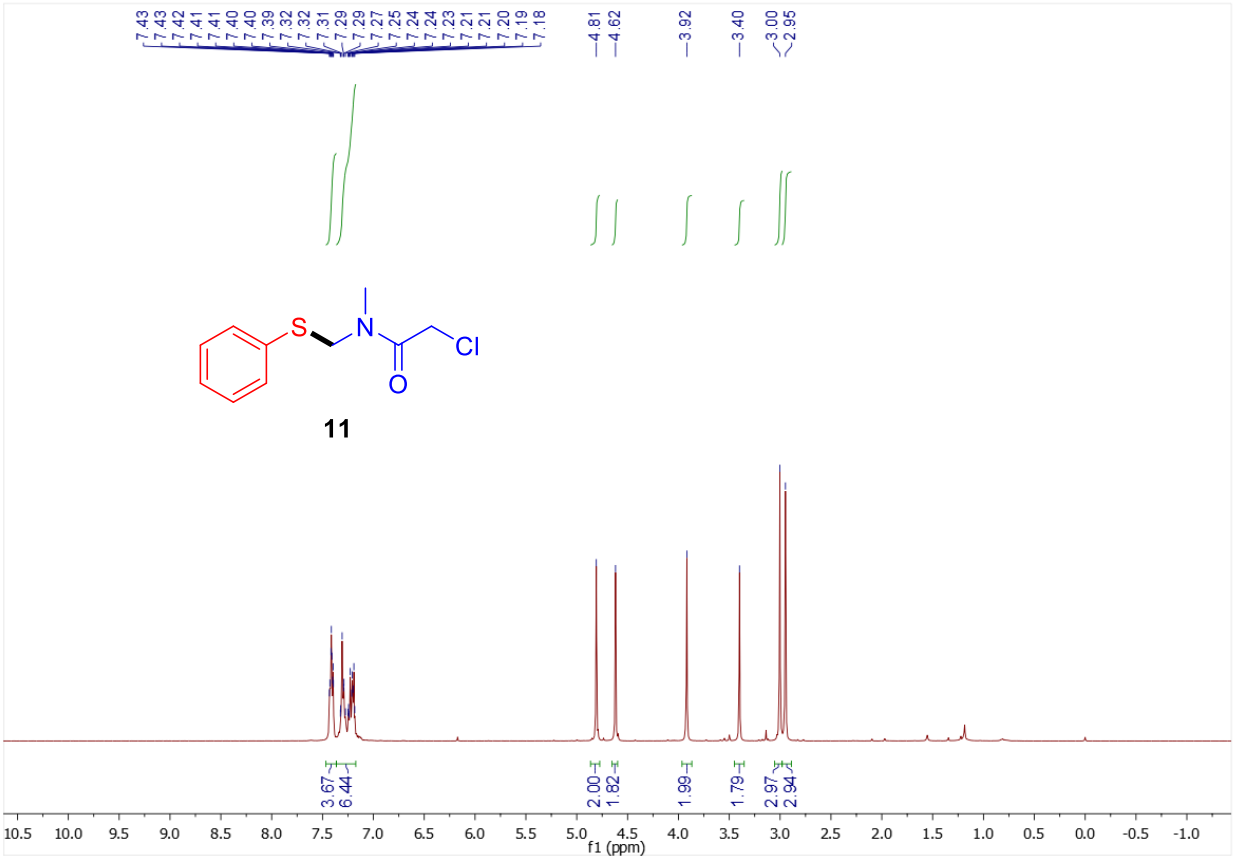
^{13}C-NMR (101 MHz, $CDCl_3$) of **10**



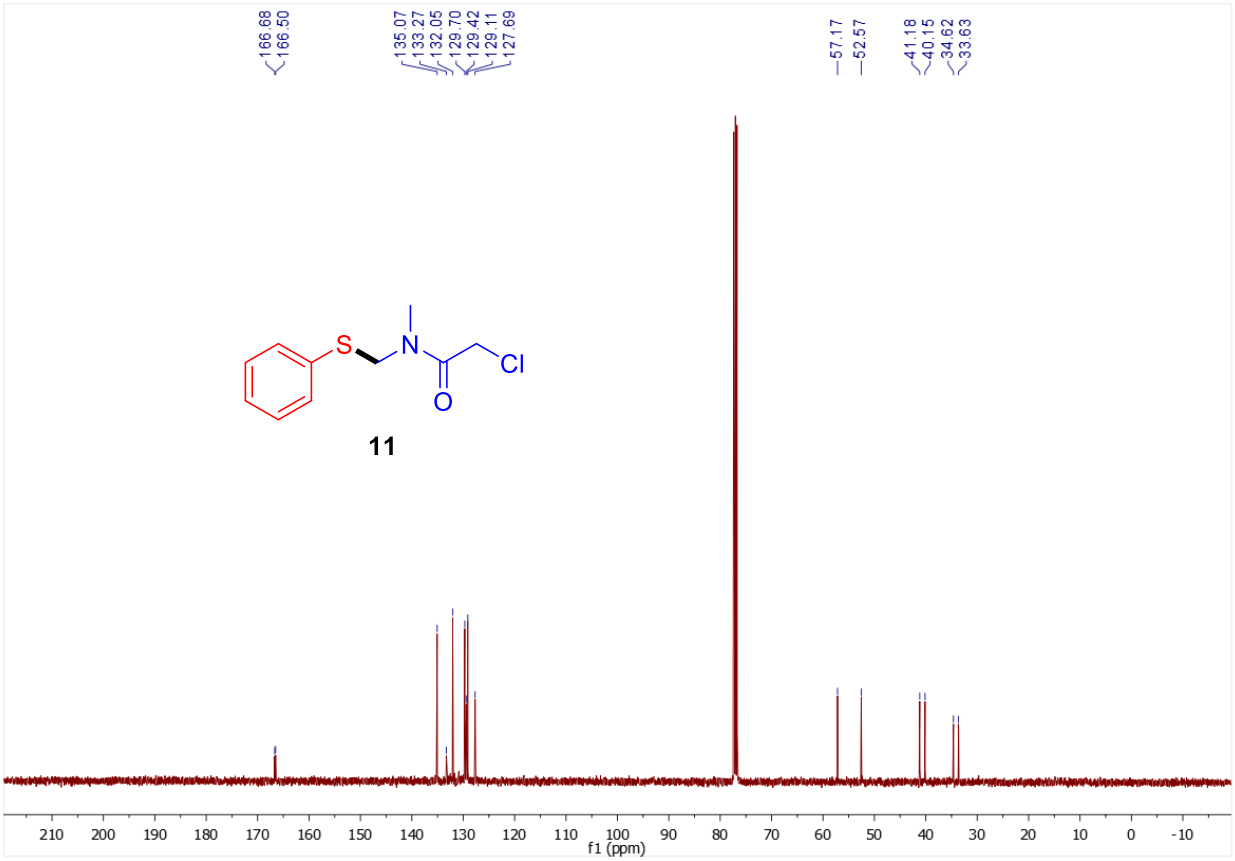
^{13}C-NMR (101 MHz, $CDCl_3$) of **10**



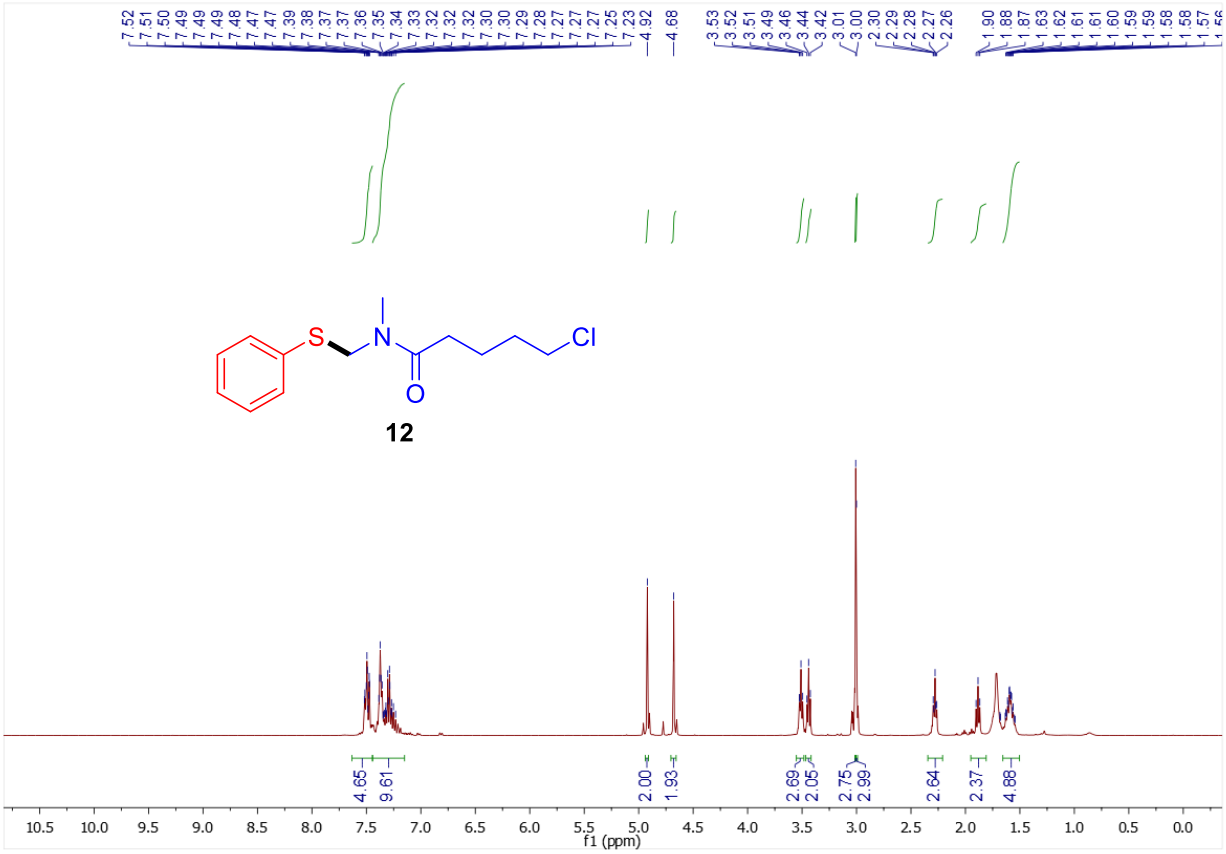
^{1}H-NMR (400 MHz, $CDCl_3$) of **11**



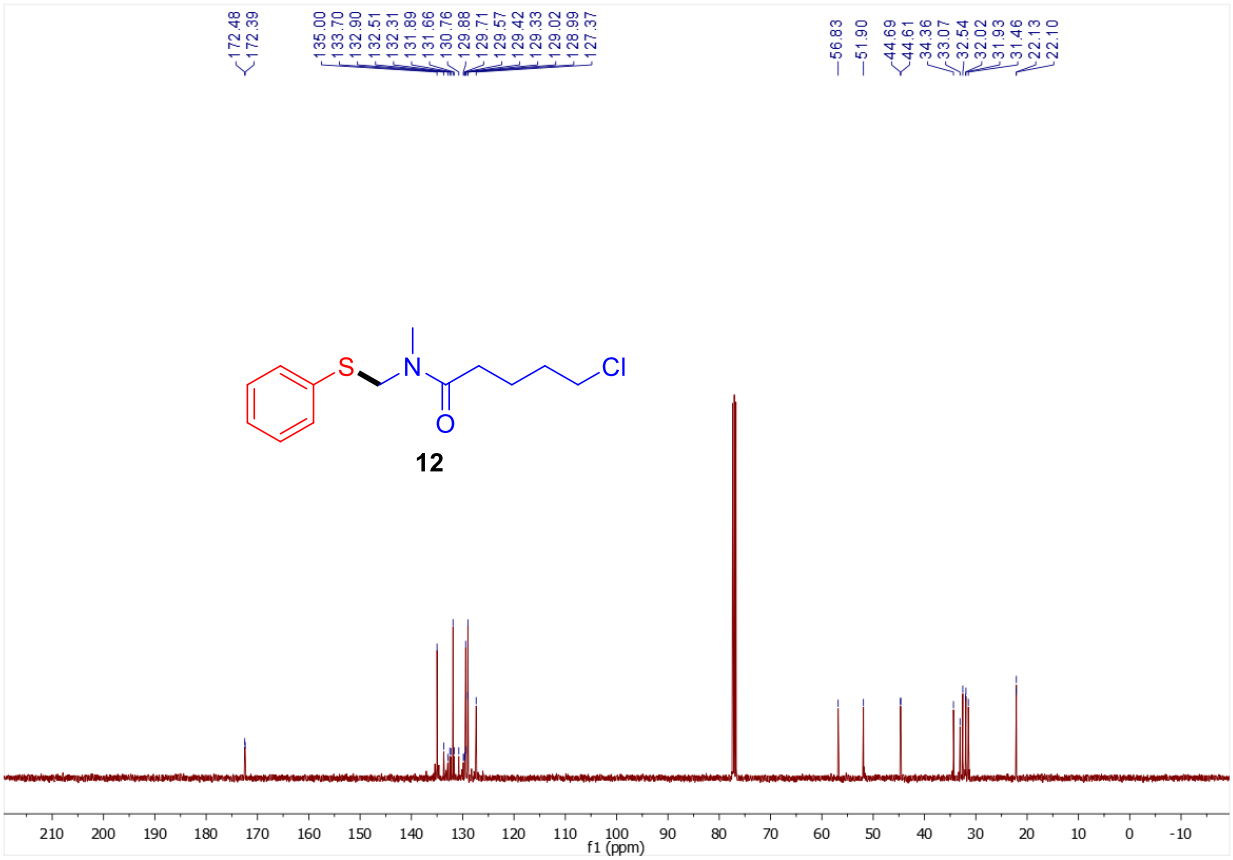
^{13}C-NMR (101 MHz, $CDCl_3$) of **11**



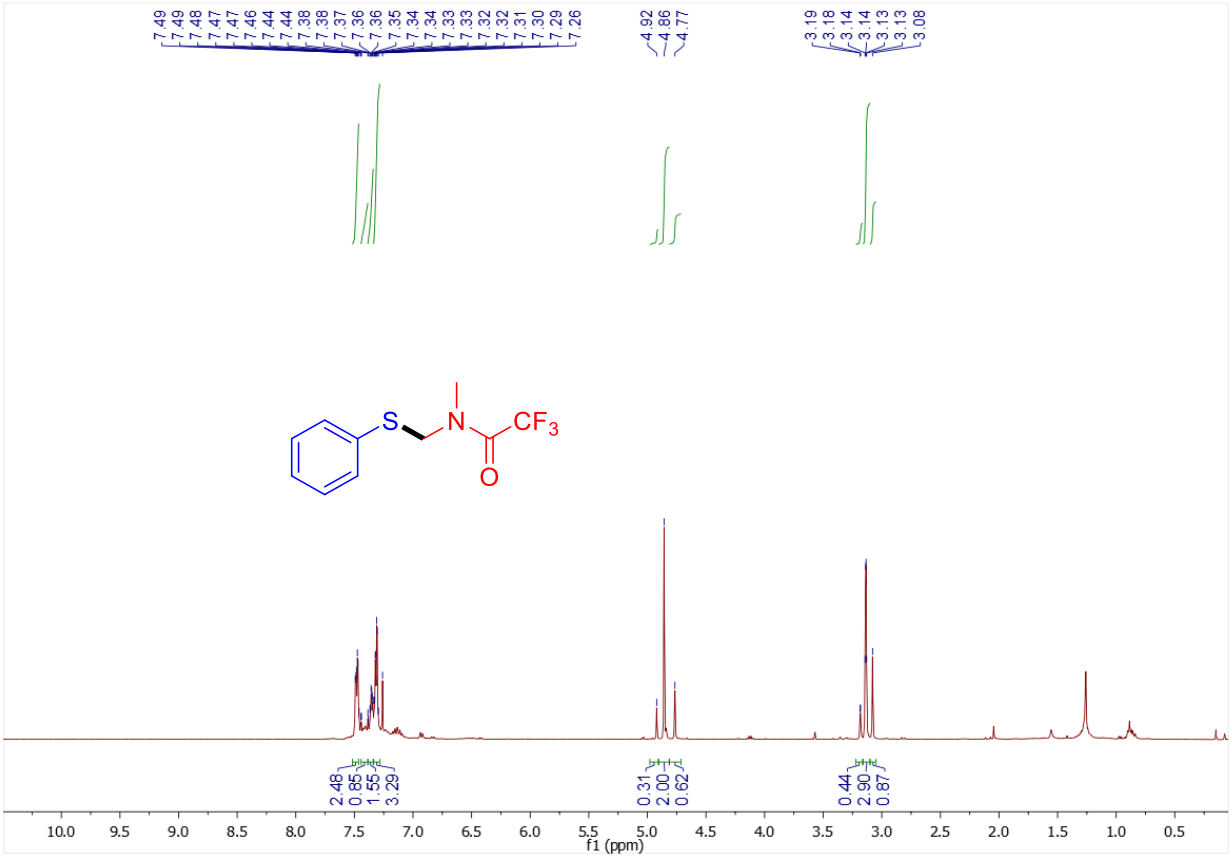
^{1}H-NMR (400 MHz, $CDCl_3$) of **12**



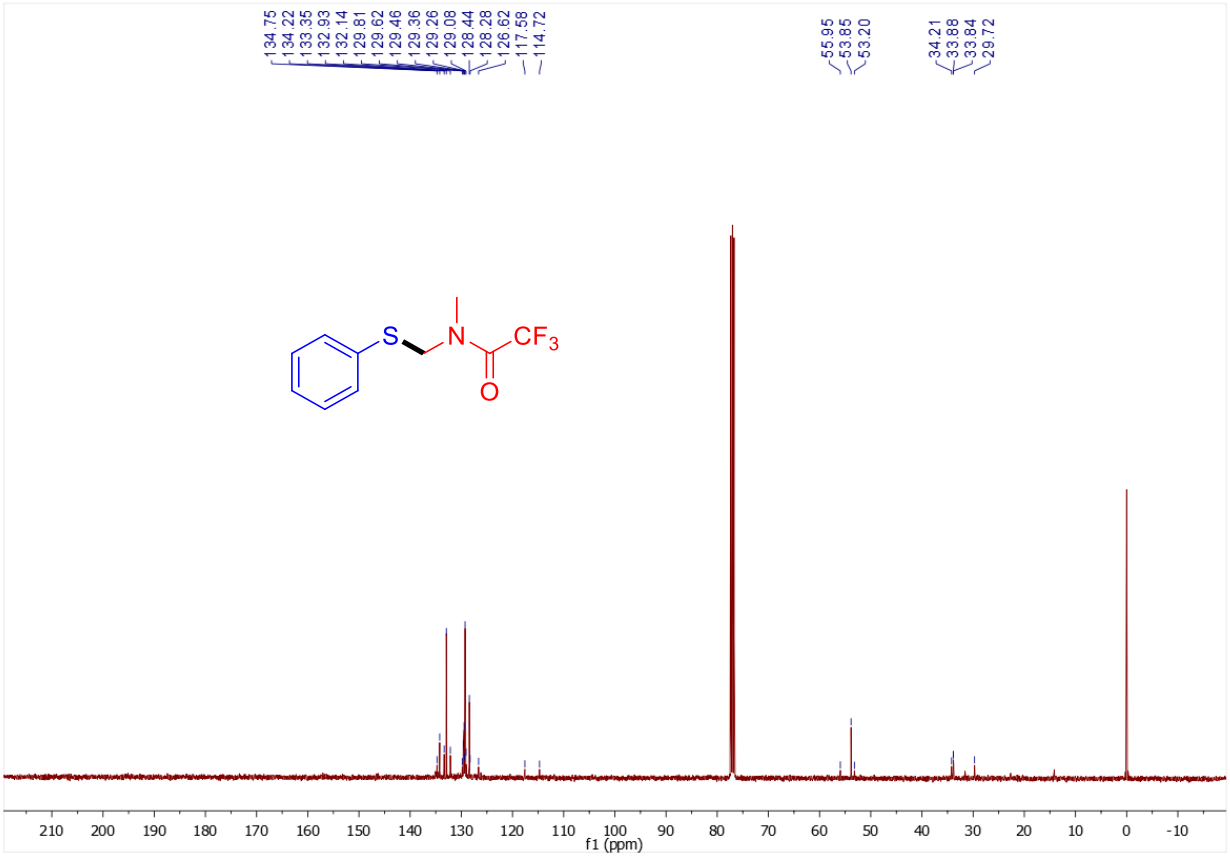
^{13}C-NMR (101 MHz, $CDCl_3$) of **12**



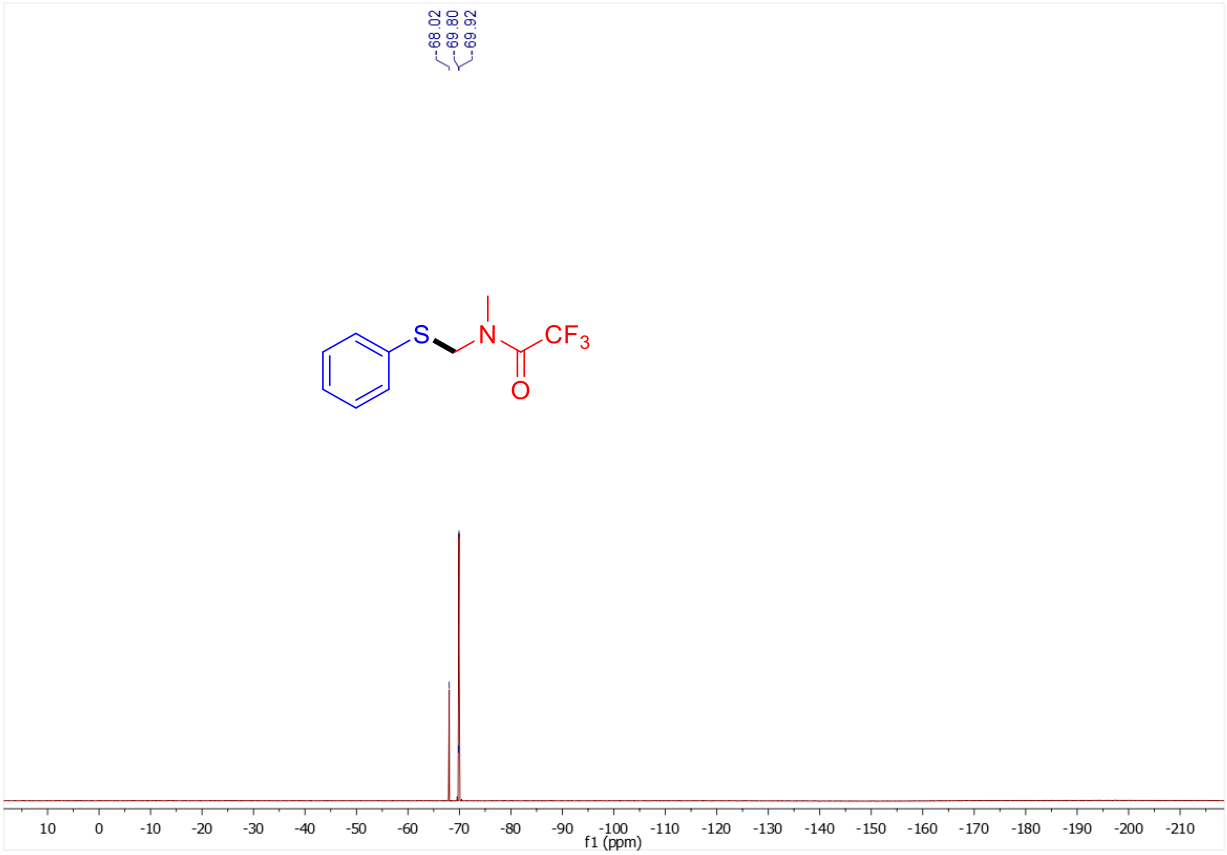
^{1}H-NMR (400 MHz, $CDCl_3$) of **13**



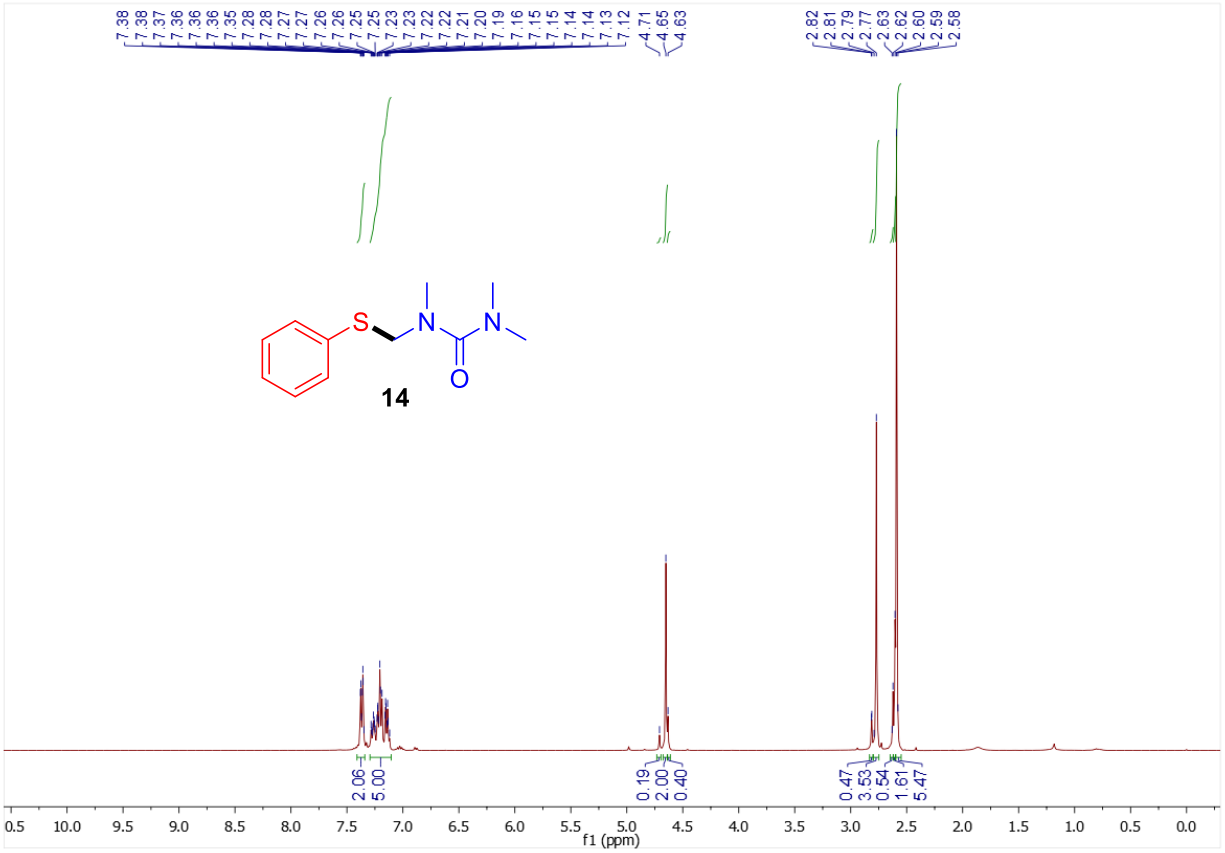
^{13}C-NMR (101 MHz, $CDCl_3$) of **13**



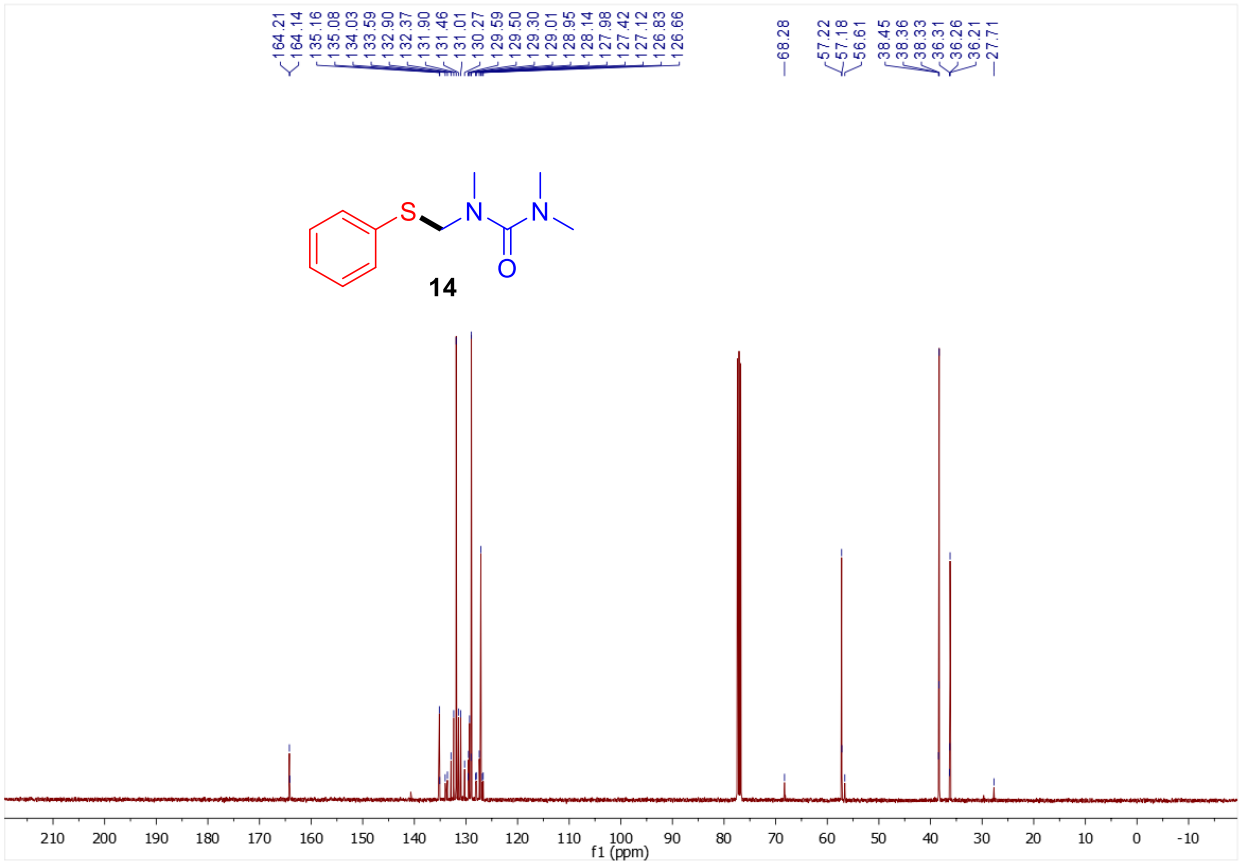
^{19}F NMR (376 MHz, $CDCl_3$) of 13



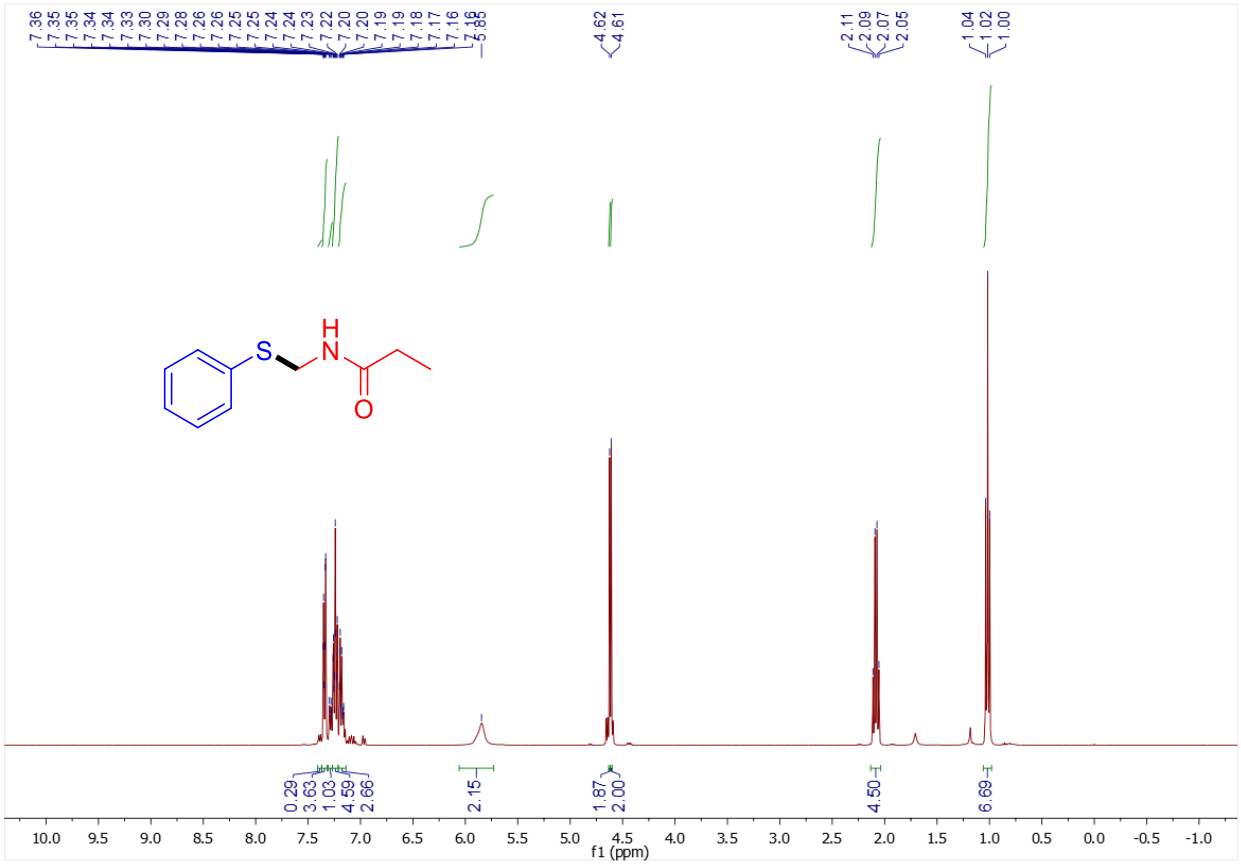
^{1}H-NMR (400 MHz, $CDCl_3$) of 14



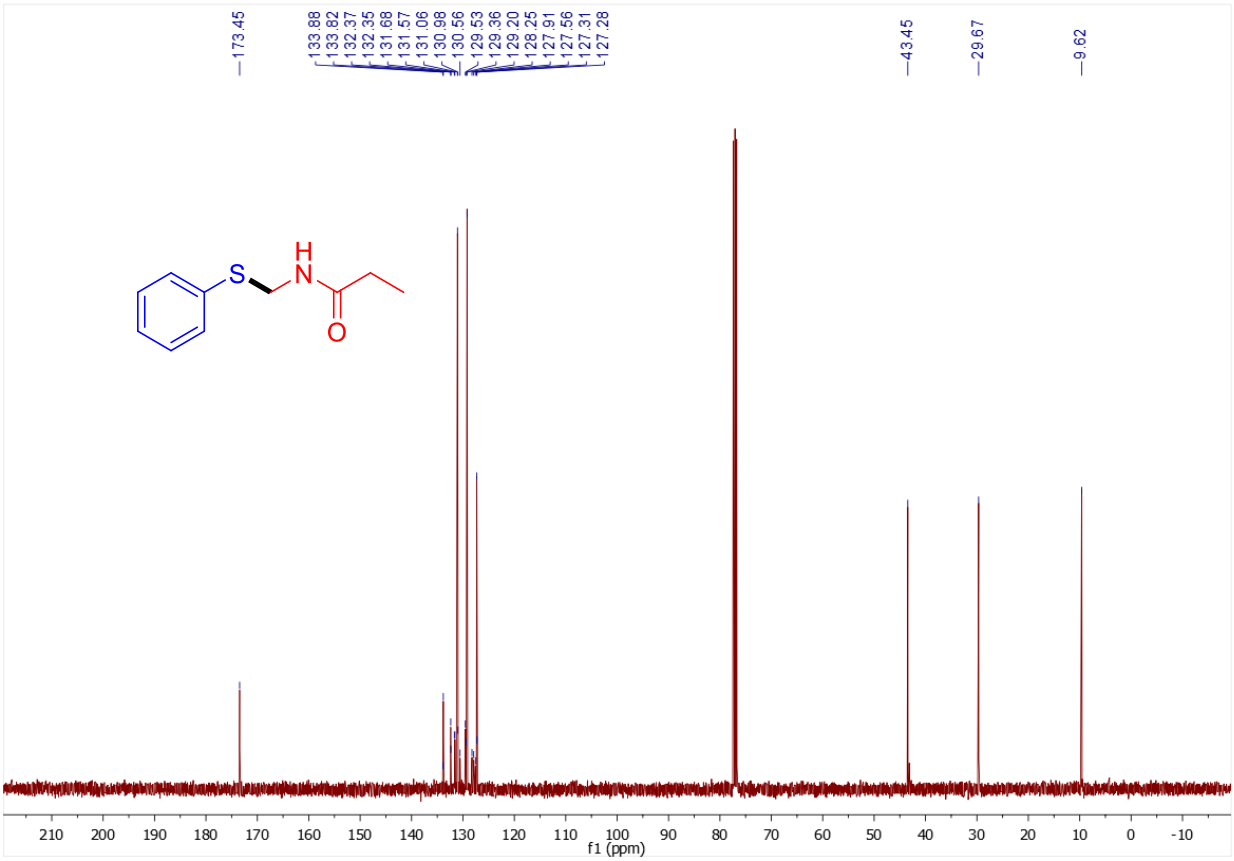
^{13}C-NMR (101 MHz, $CDCl_3$) of **14**



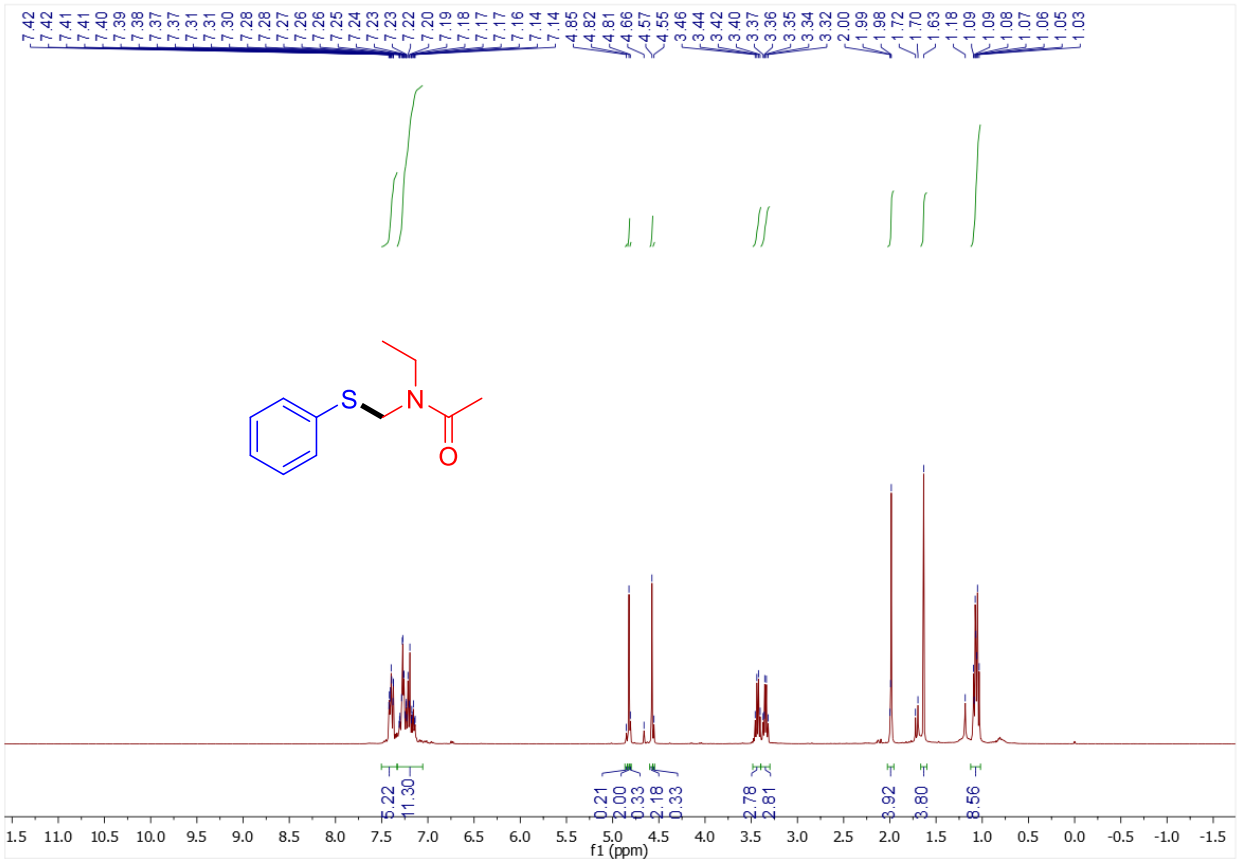
^{1}H-NMR (400 MHz, $CDCl_3$) of 15



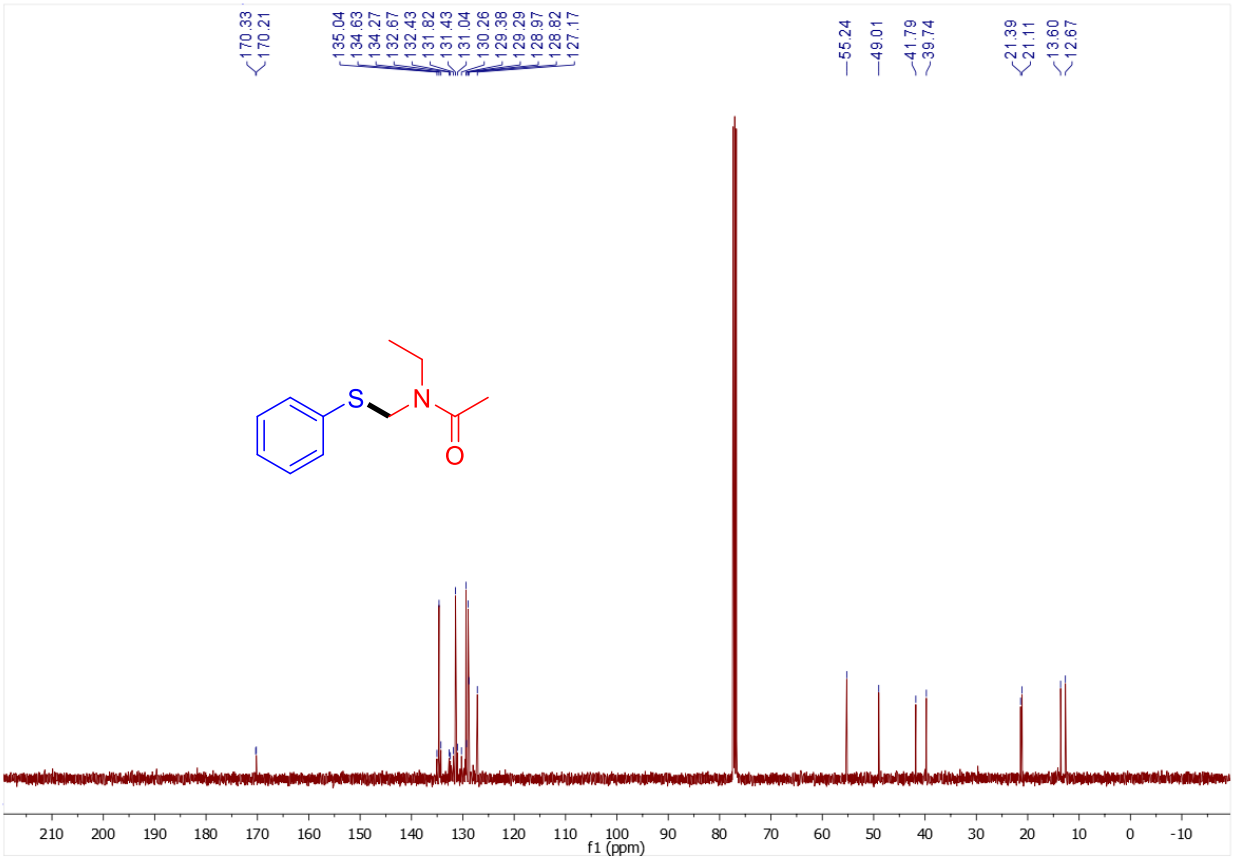
^{13}C-NMR (101 MHz, $CDCl_3$) of **15**



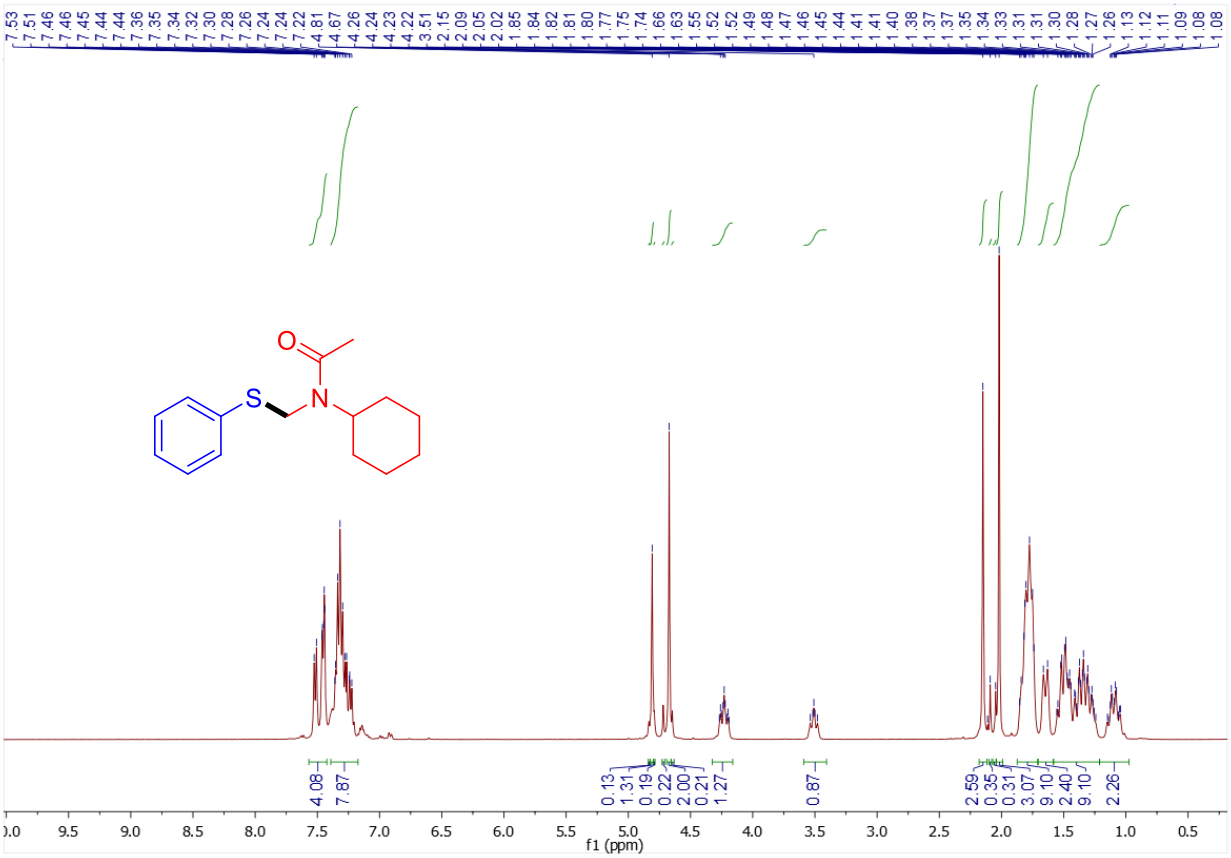
^{1}H-NMR (400 MHz, $CDCl_3$) of **16**



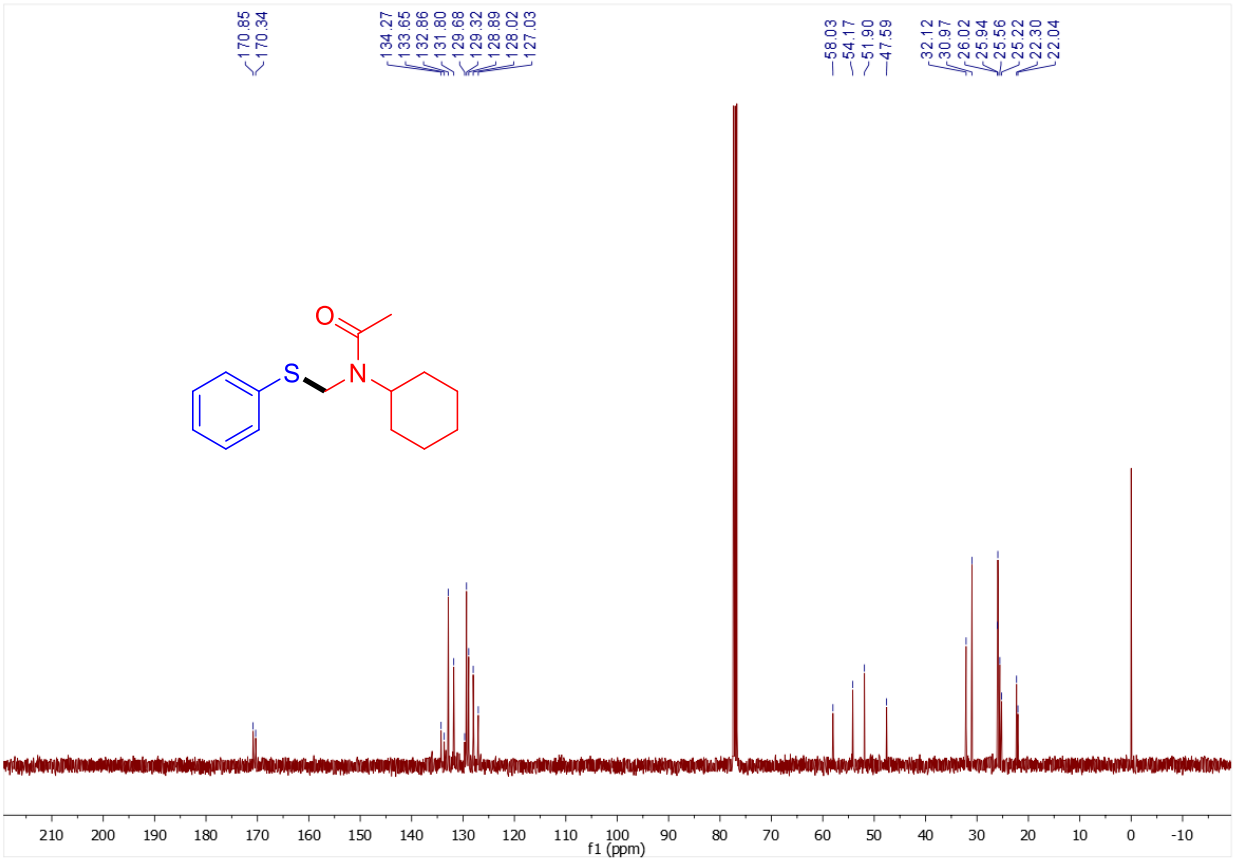
^{13}C-NMR (101 MHz, $CDCl_3$) of **16**



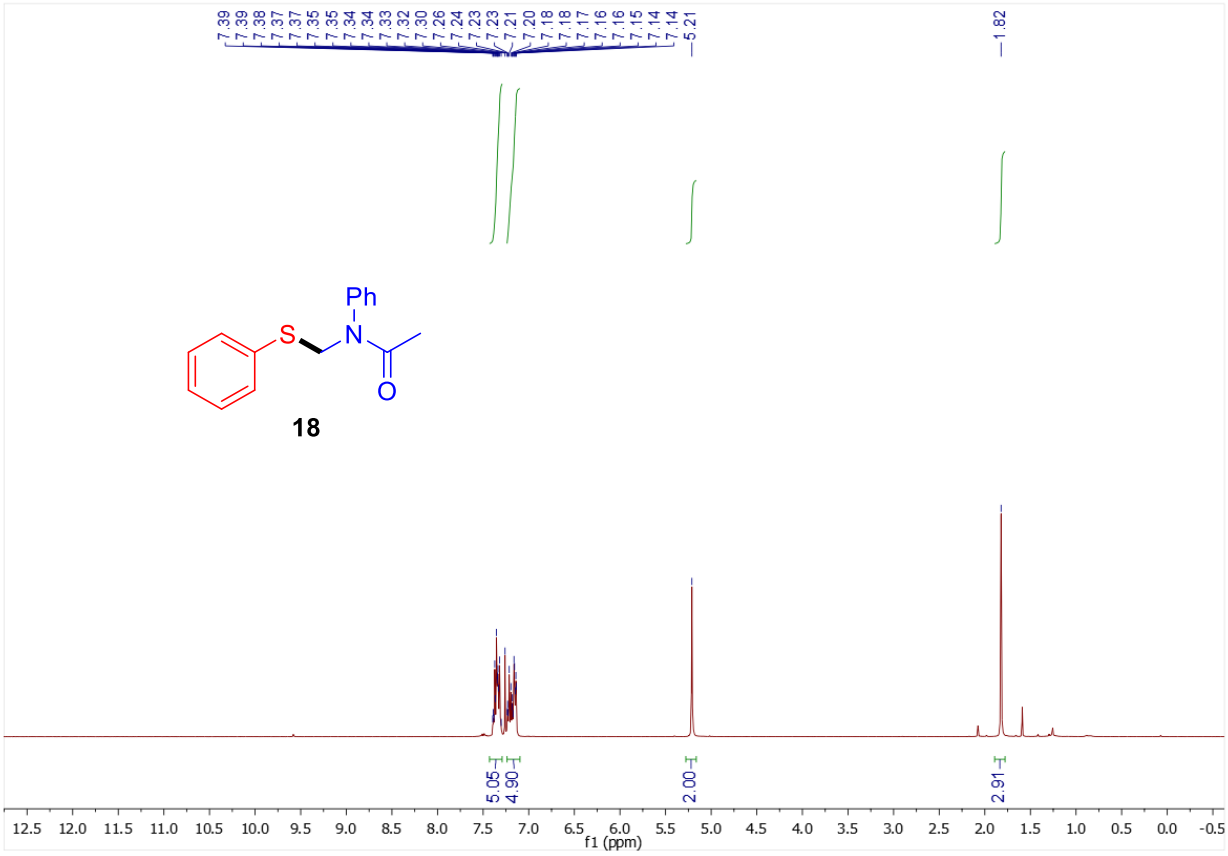
^{1}H-NMR (400 MHz, $CDCl_3$) of **17**



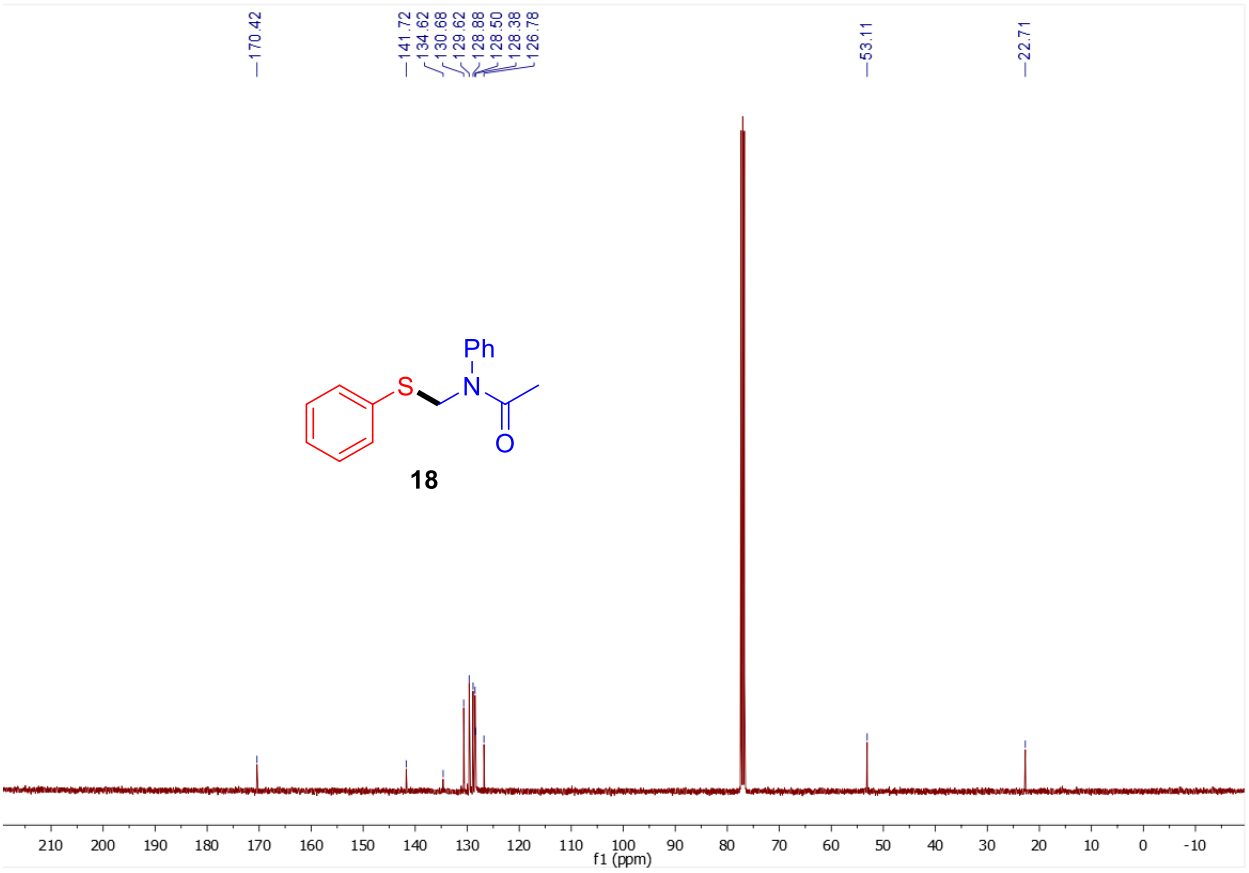
^{13}C-NMR (101 MHz, $CDCl_3$) of **17**



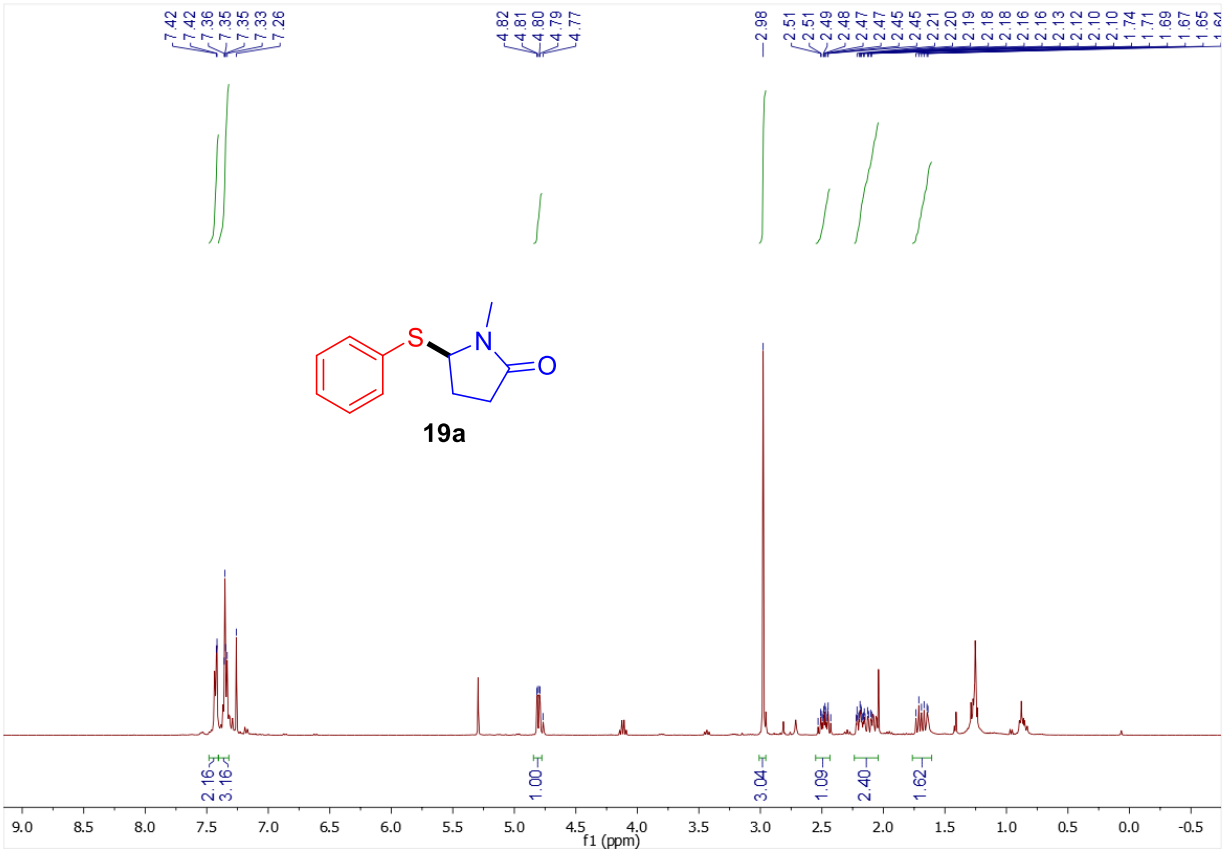
^{1}H-NMR (400 MHz, $CDCl_3$) of **18**



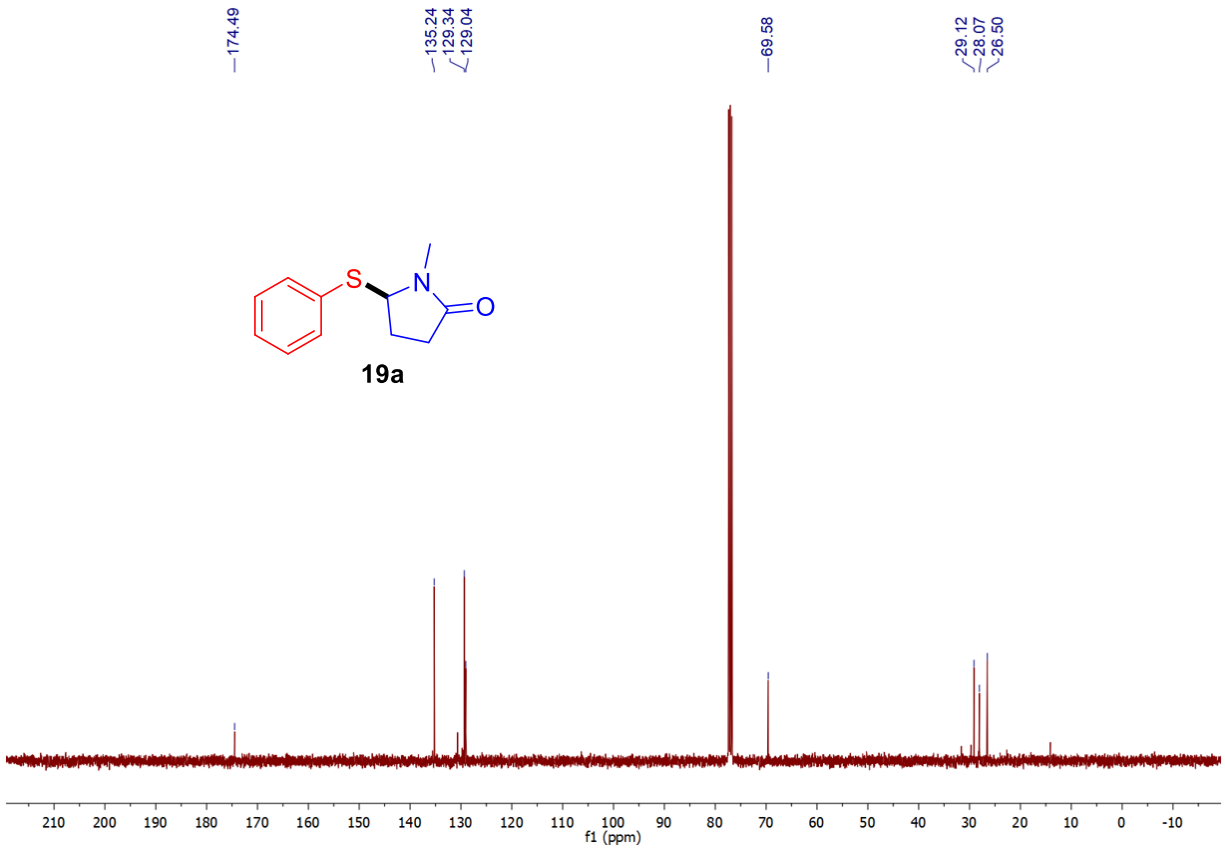
^{13}C-NMR (101 MHz, $CDCl_3$) of **18**



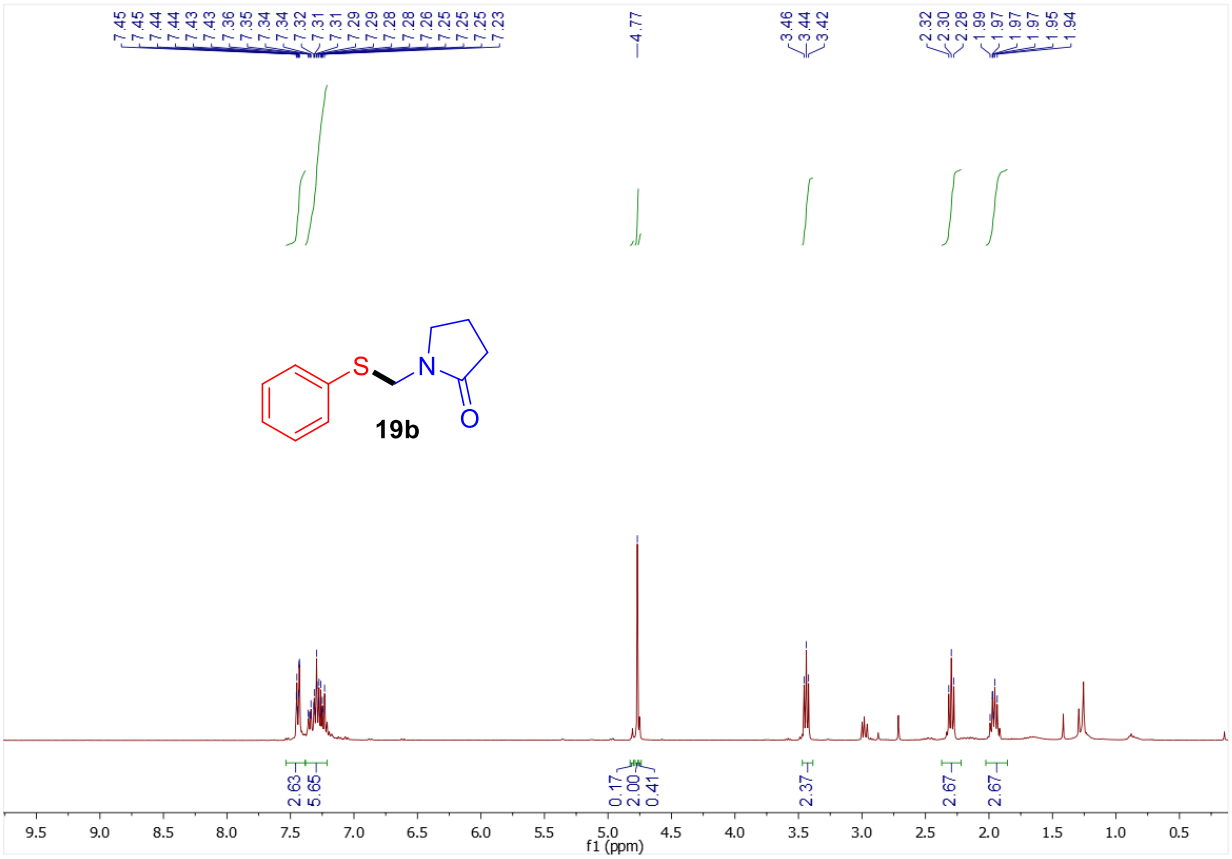
^{1}H-NMR (400 MHz, $CDCl_3$) of **19a**



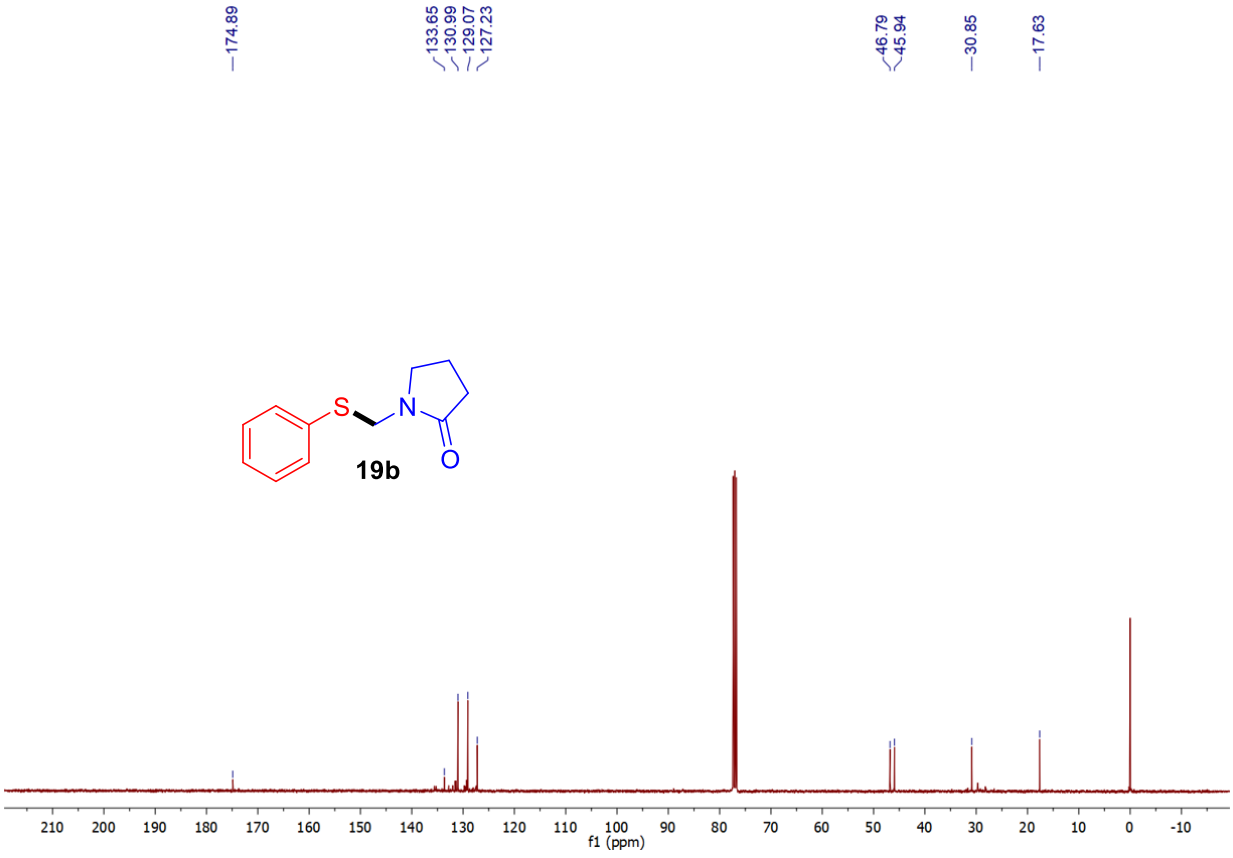
^{13}C-NMR (101 MHz, $CDCl_3$) of **19a**



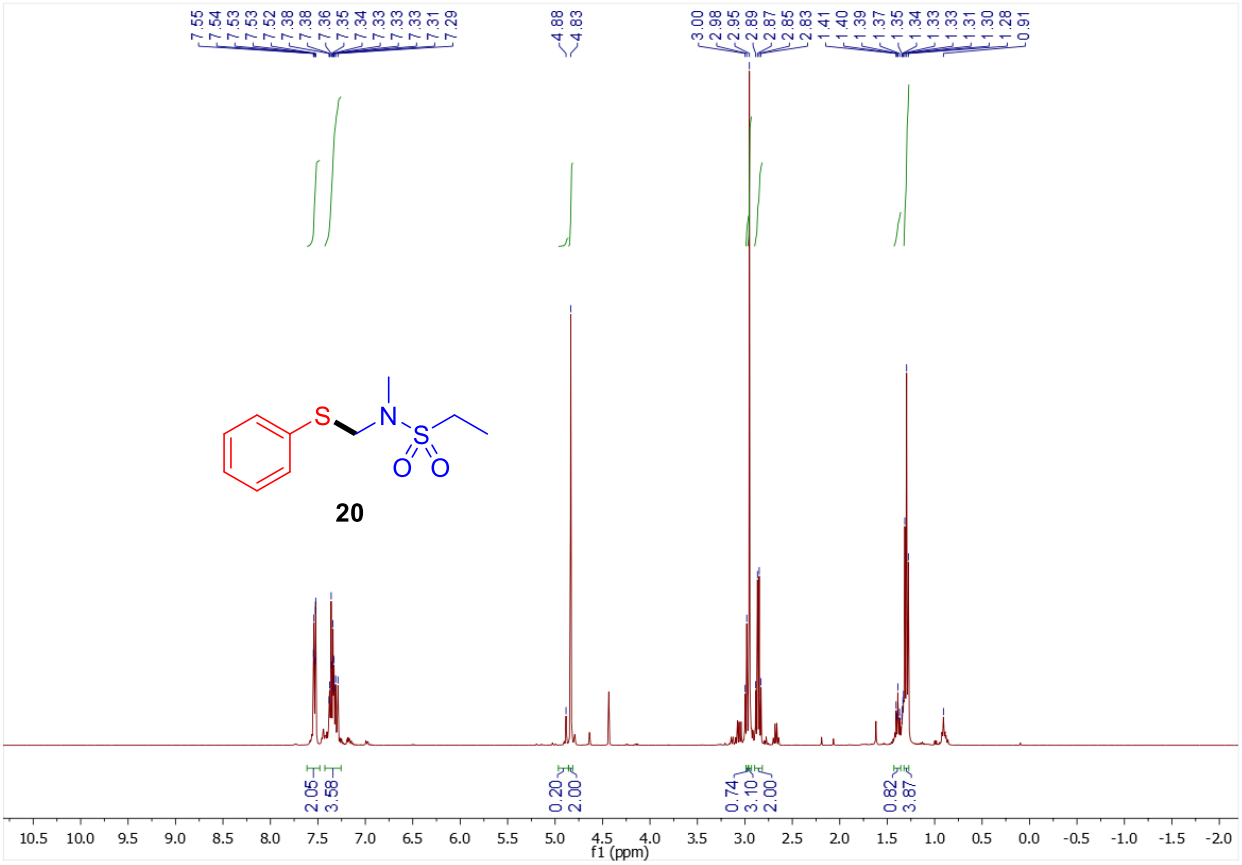
^{1}H-NMR (400 MHz, $CDCl_3$) of **19b**



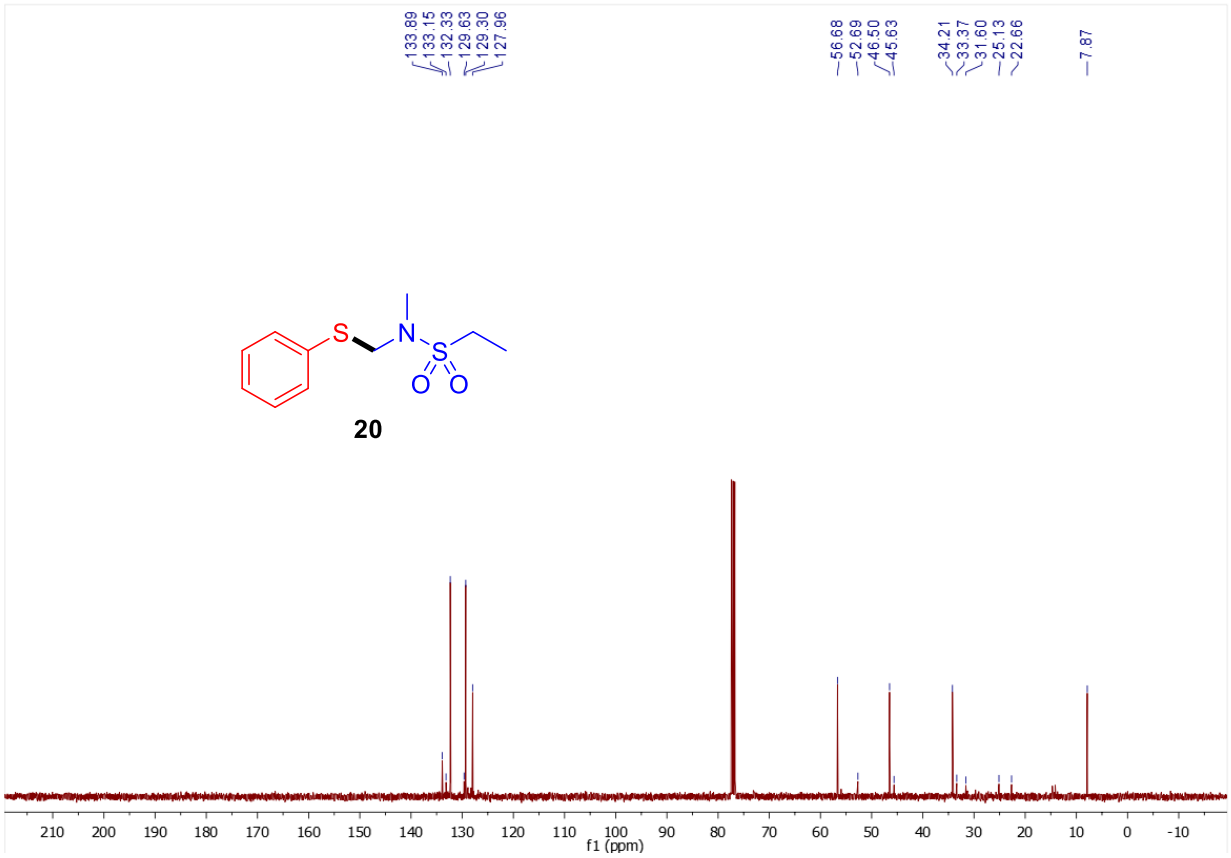
^{13}C-NMR (101 MHz, $CDCl_3$) of **19b**



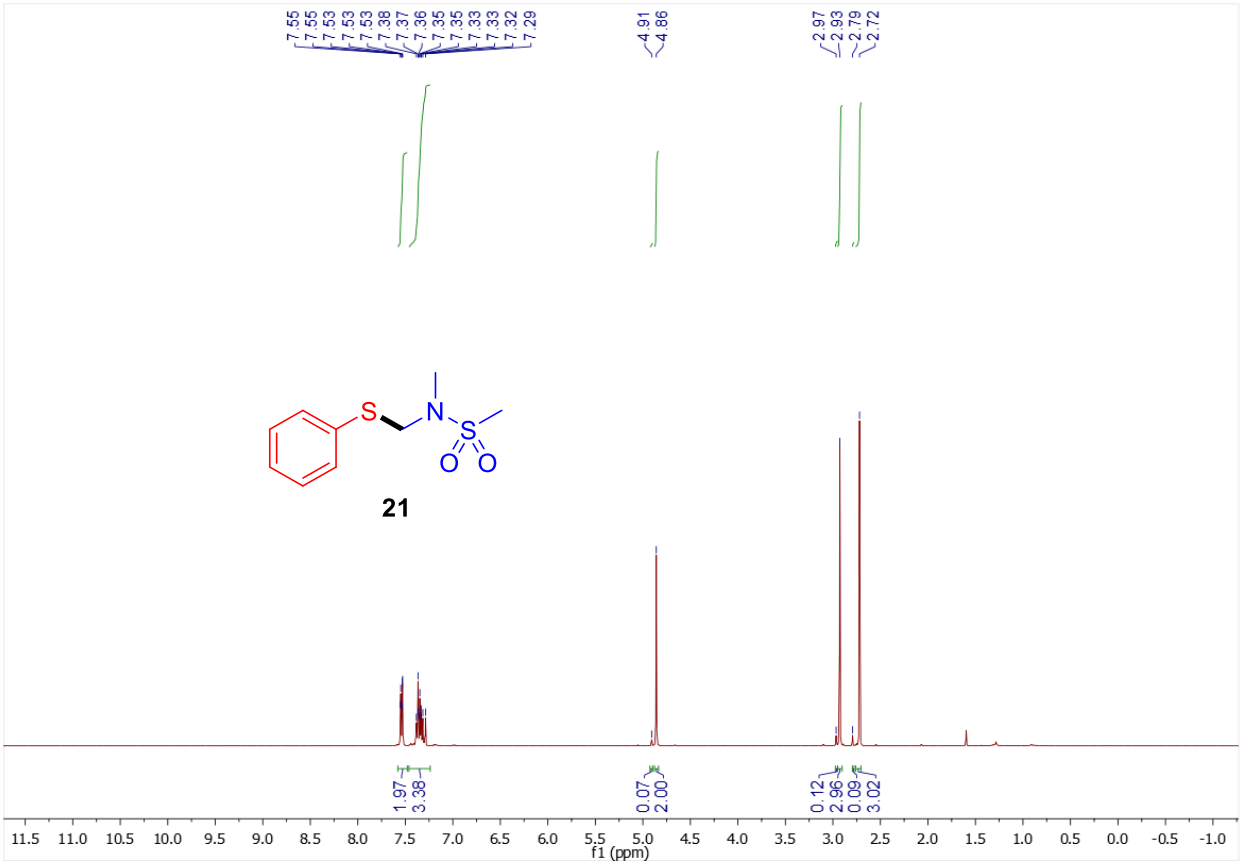
^{1}H-NMR (400 MHz, $CDCl_3$) of **20**



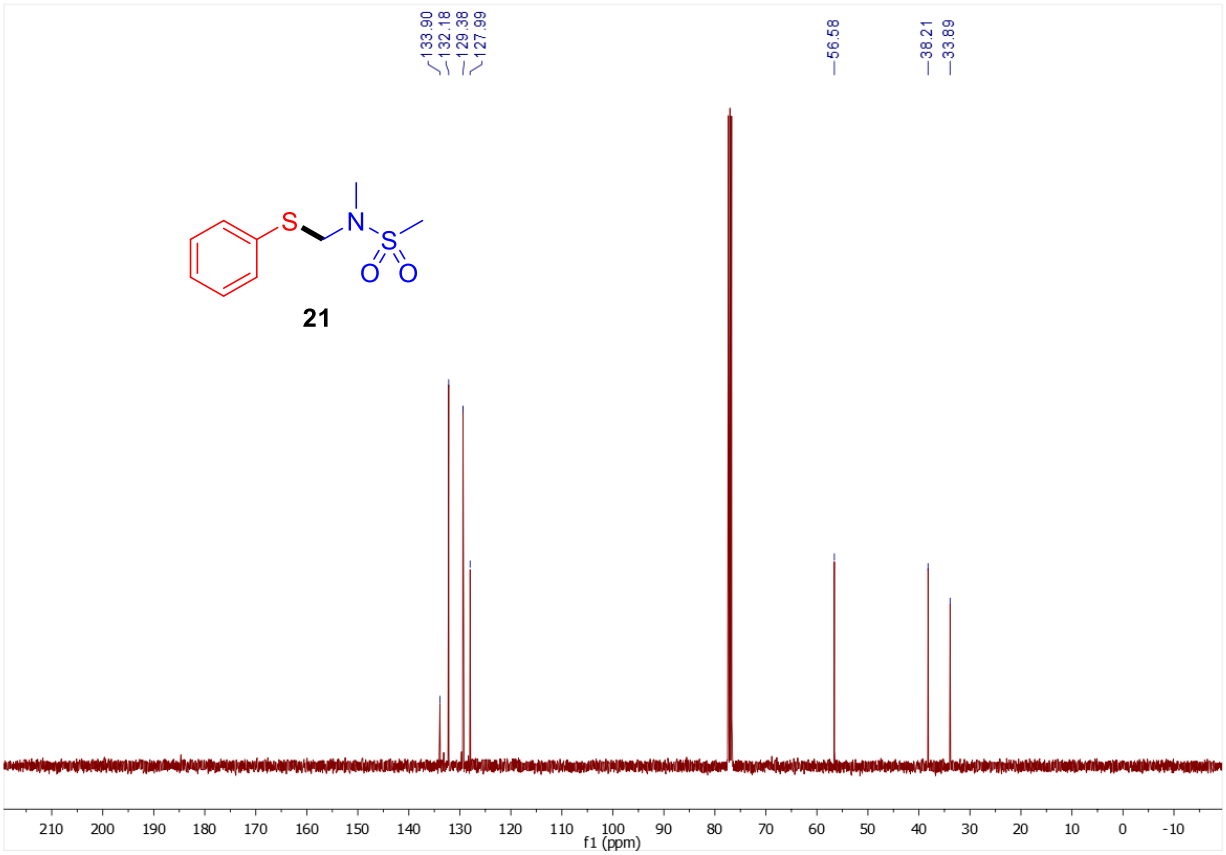
^{13}C-NMR (101 MHz, $CDCl_3$) of **20**



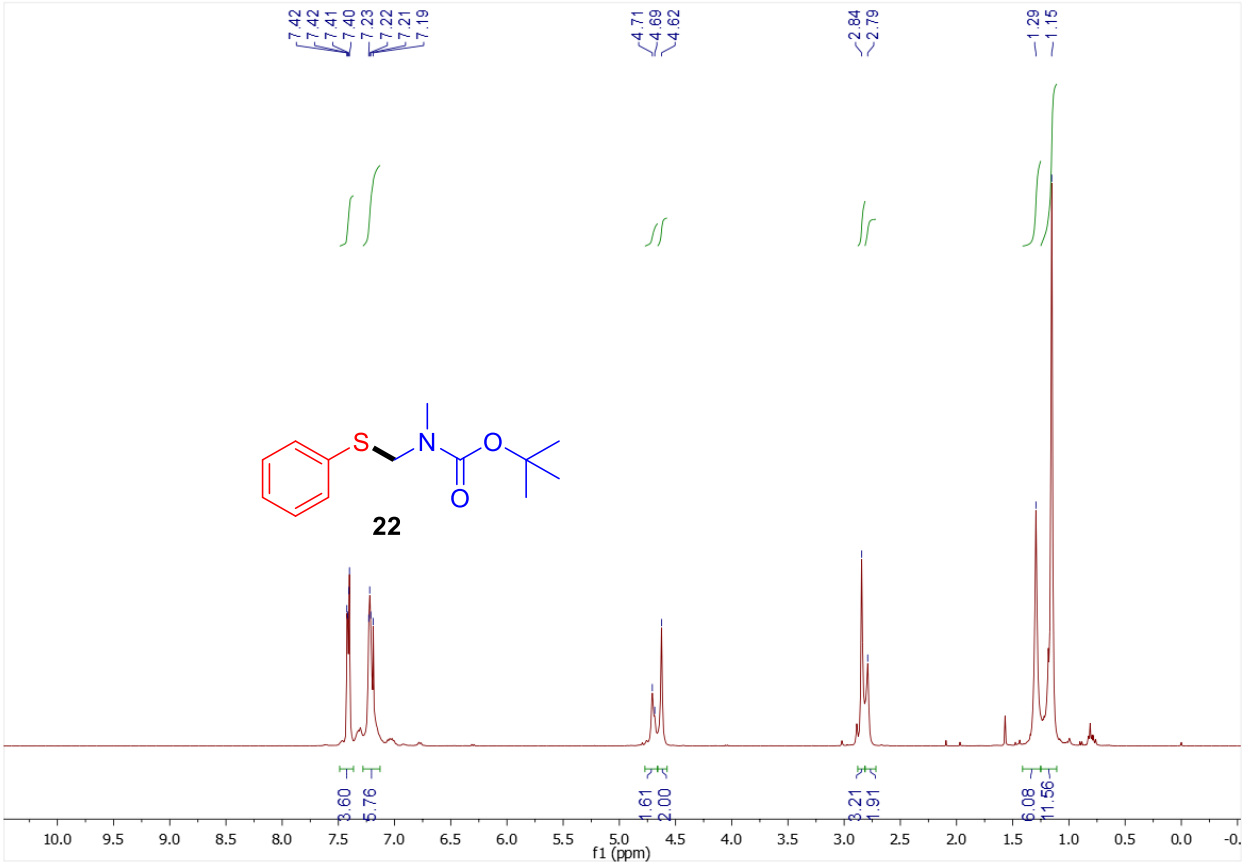
^{1}H-NMR (400 MHz, $CDCl_3$) of **21**



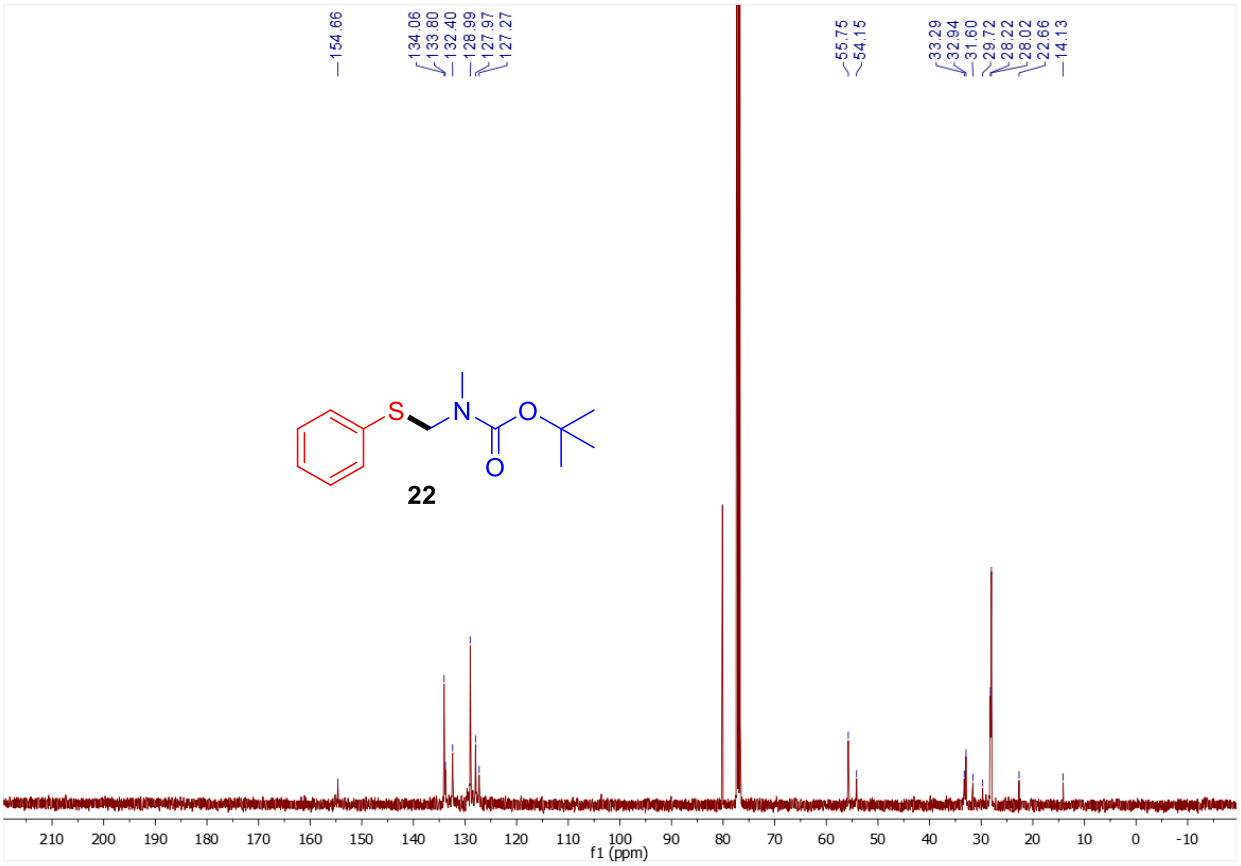
^{13}C-NMR (101 MHz, $CDCl_3$) of **21**



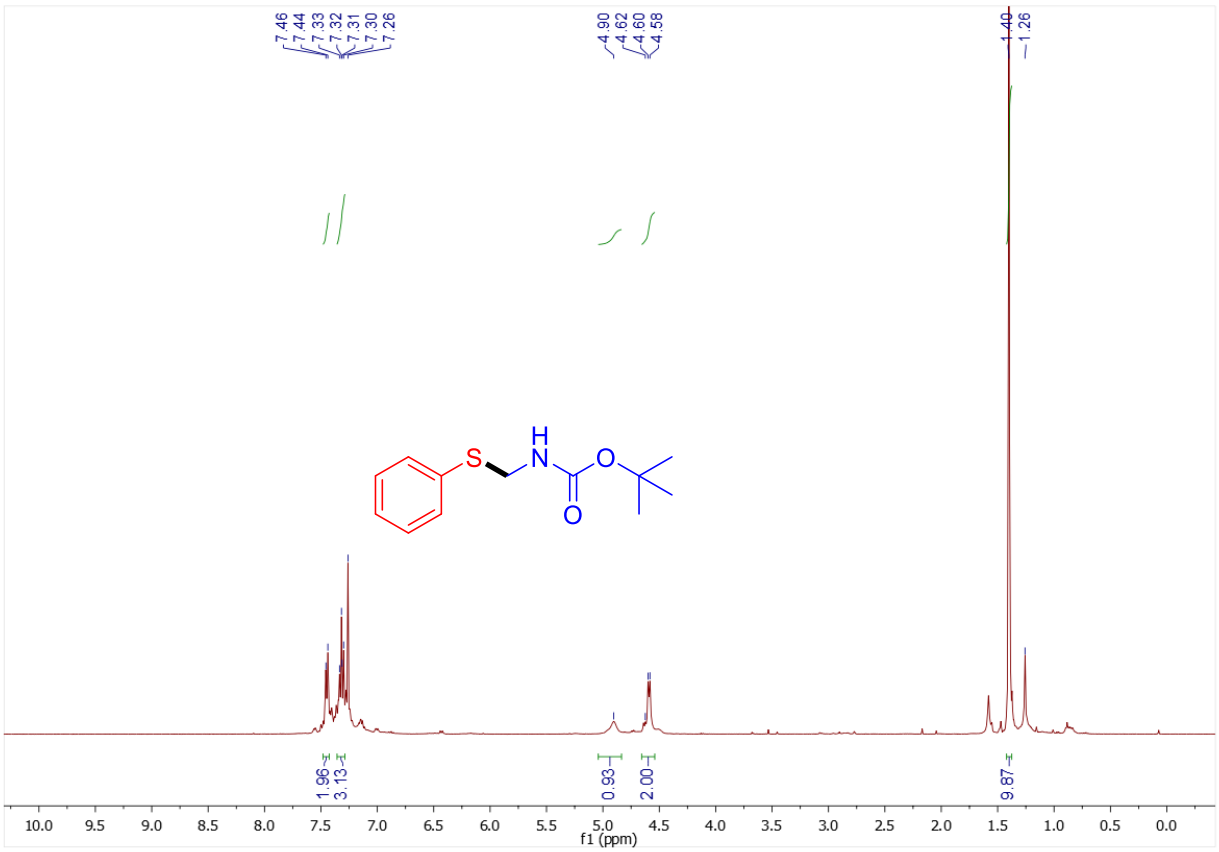
^{1}H-NMR (400 MHz, $CDCl_3$) of **22**



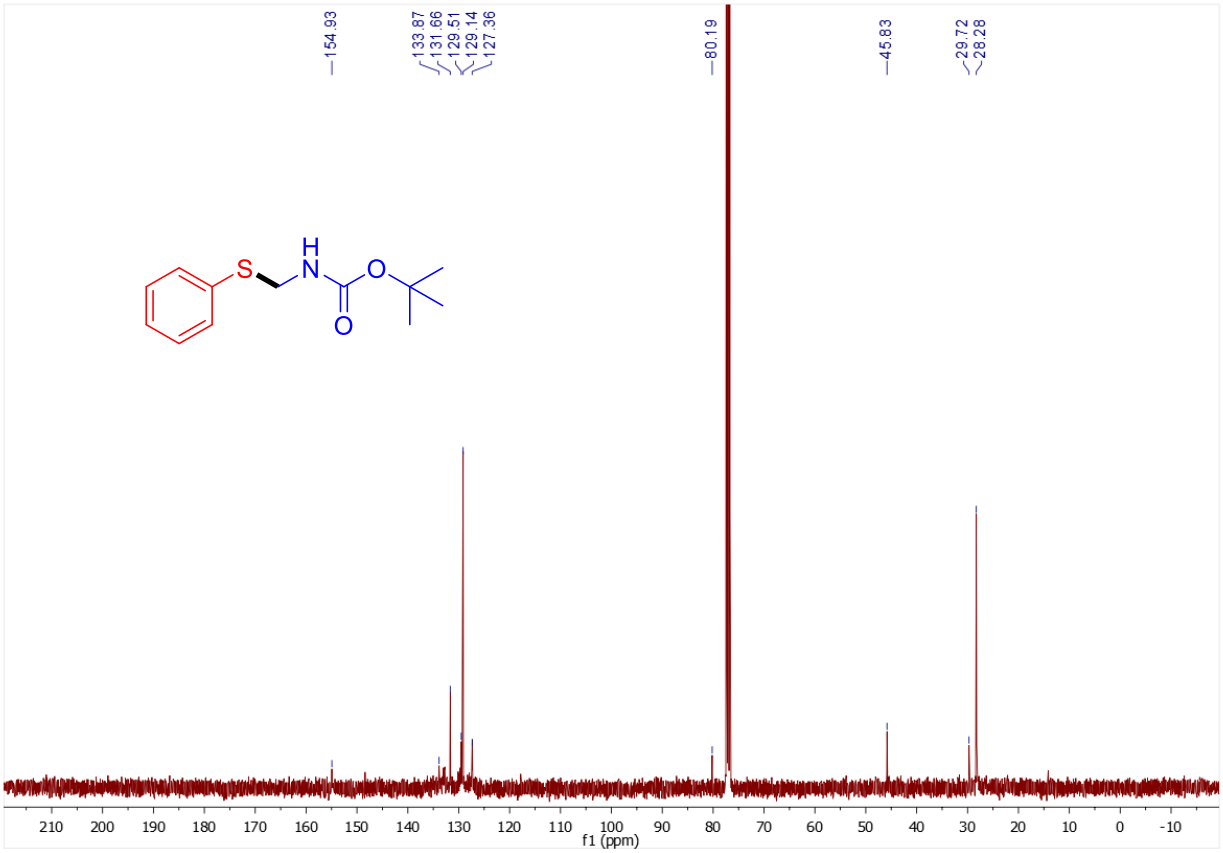
^{13}C-NMR (101 MHz, $CDCl_3$) of **22**



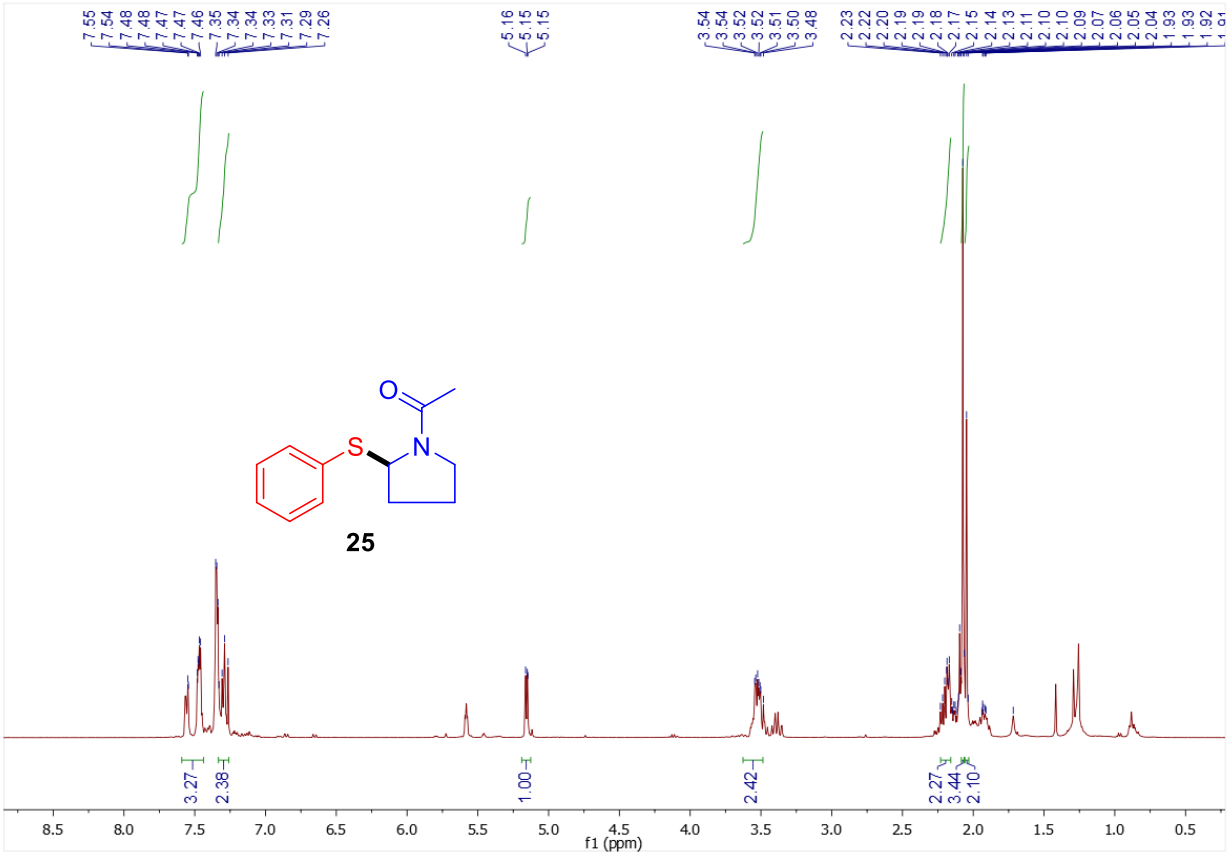
^{1}H-NMR (400 MHz, $CDCl_3$) of **23**



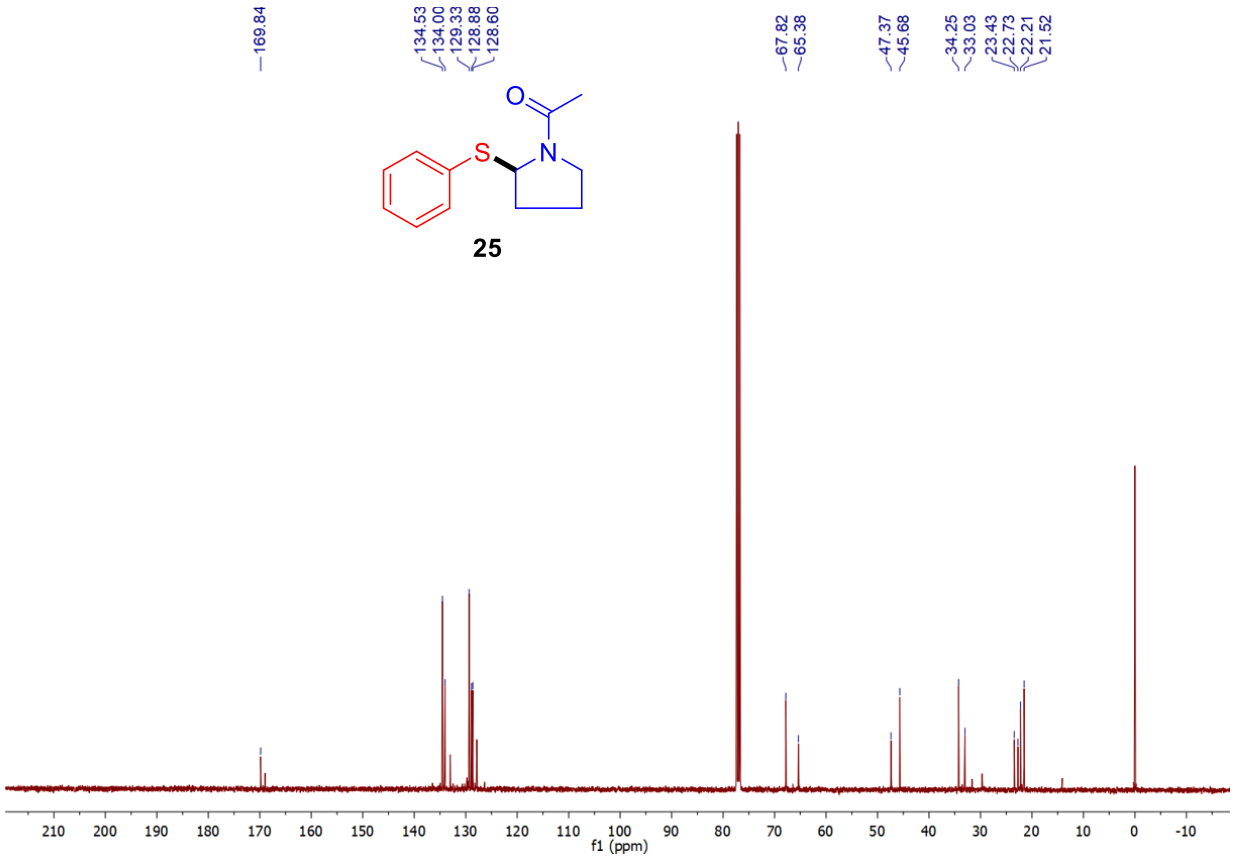
^{13}C-NMR (101 MHz, $CDCl_3$) of **23**



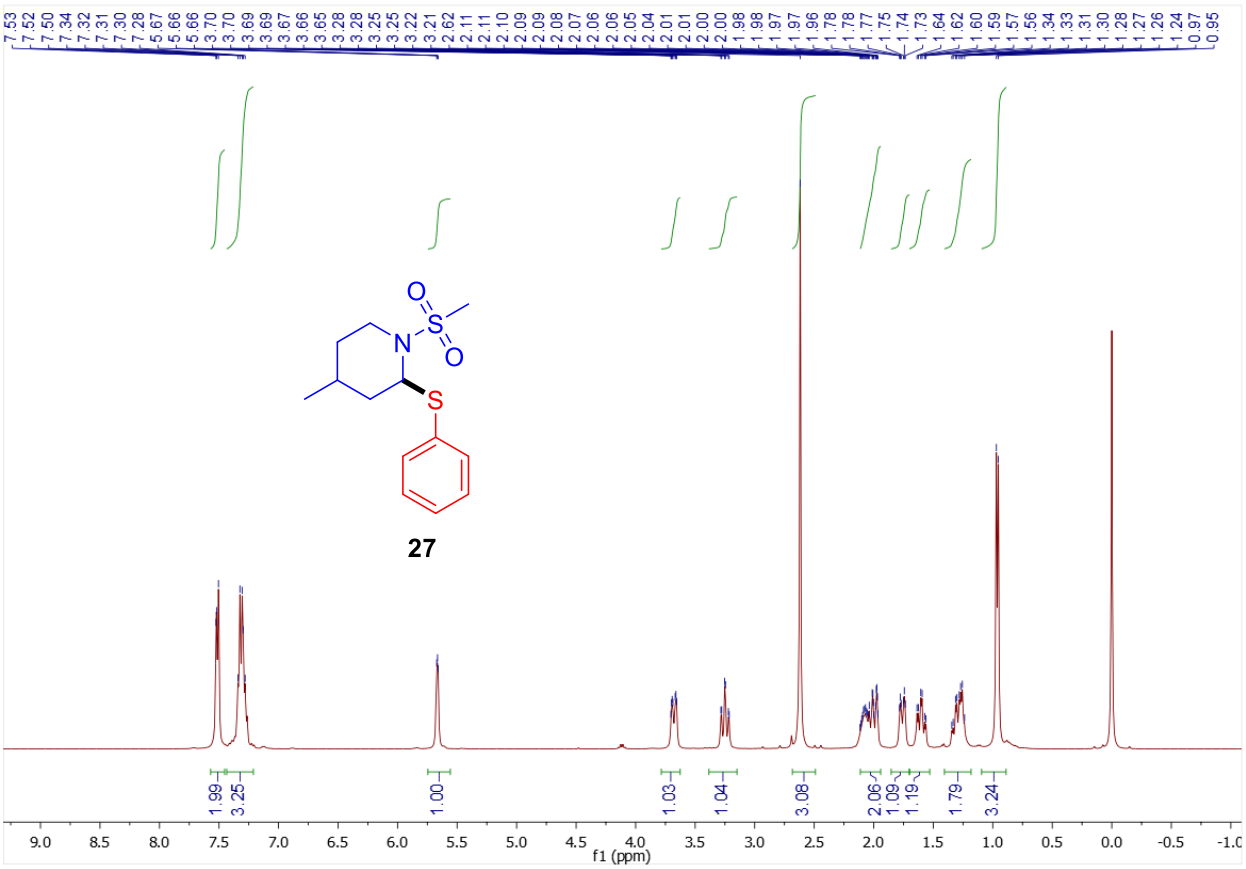
^{1}H-NMR (400 MHz, $CDCl_3$) of **25**



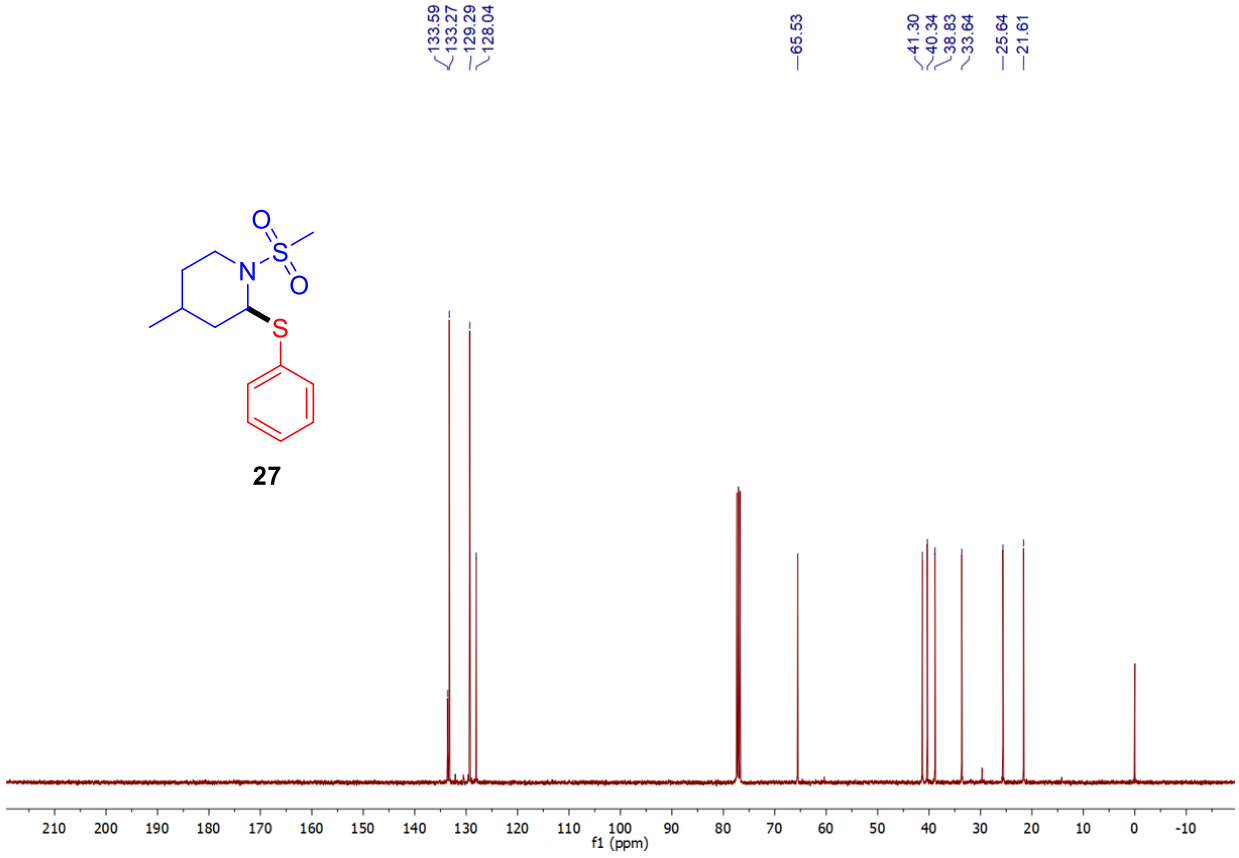
^{13}C-NMR (101 MHz, $CDCl_3$) of **25**



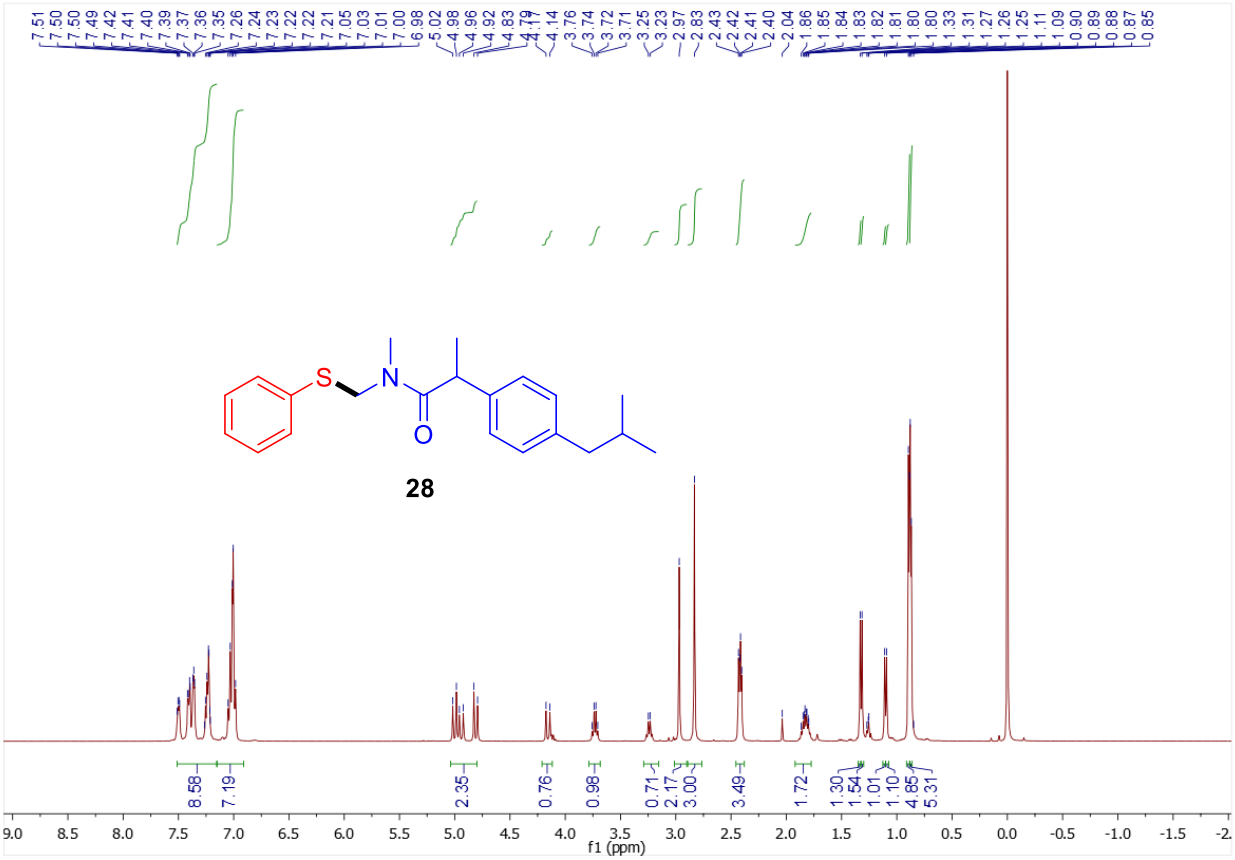
^{1}H-NMR (400 MHz, $CDCl_3$) of **27**



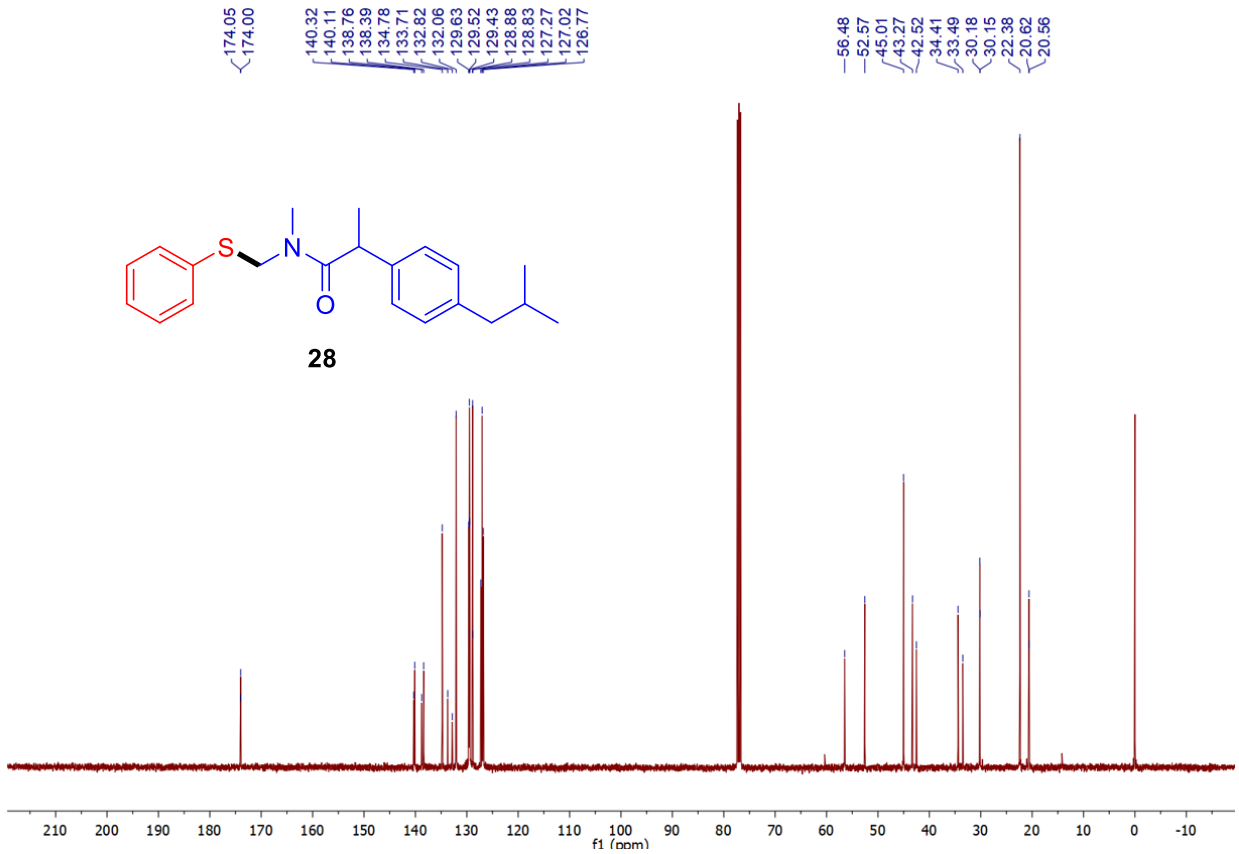
^{13}C-NMR (101 MHz, $CDCl_3$) of **27**



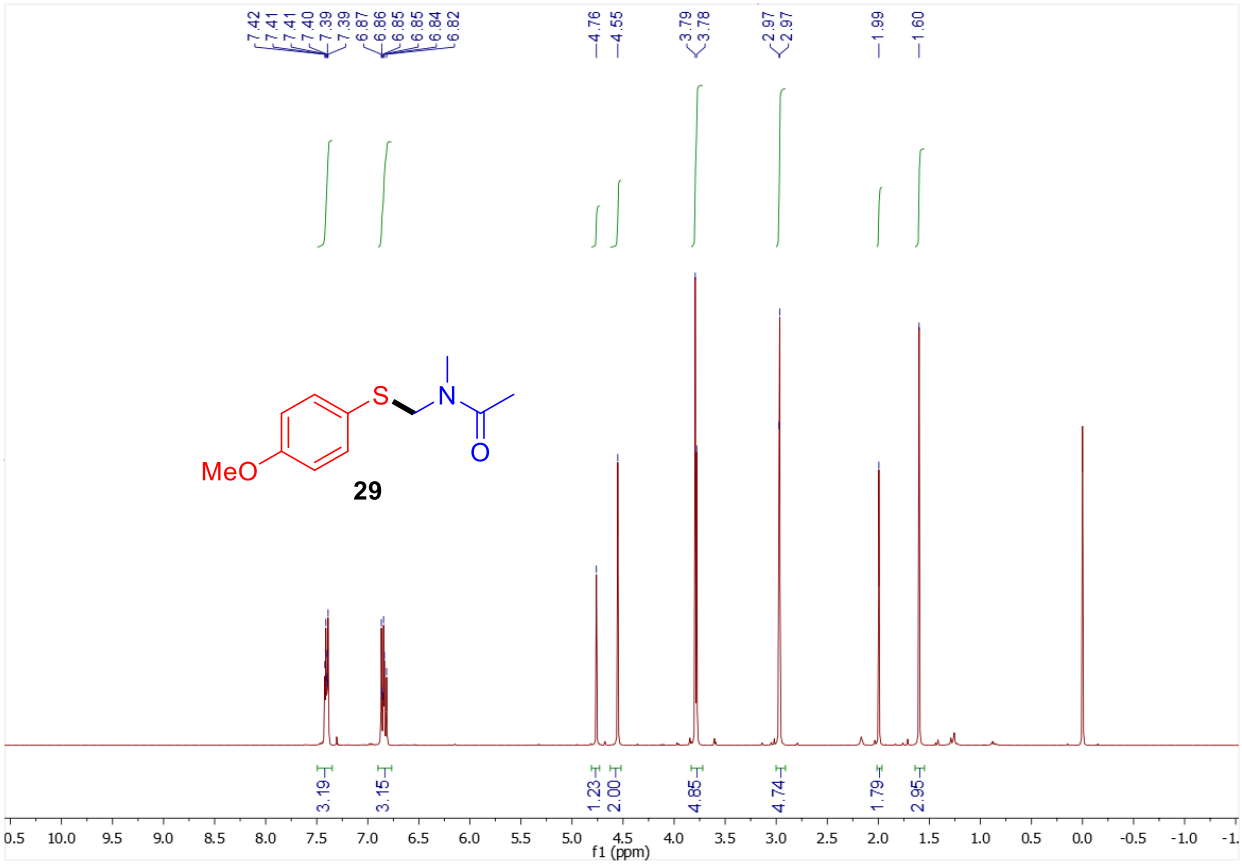
^{1}H-NMR (400 MHz, $CDCl_3$) of **28**



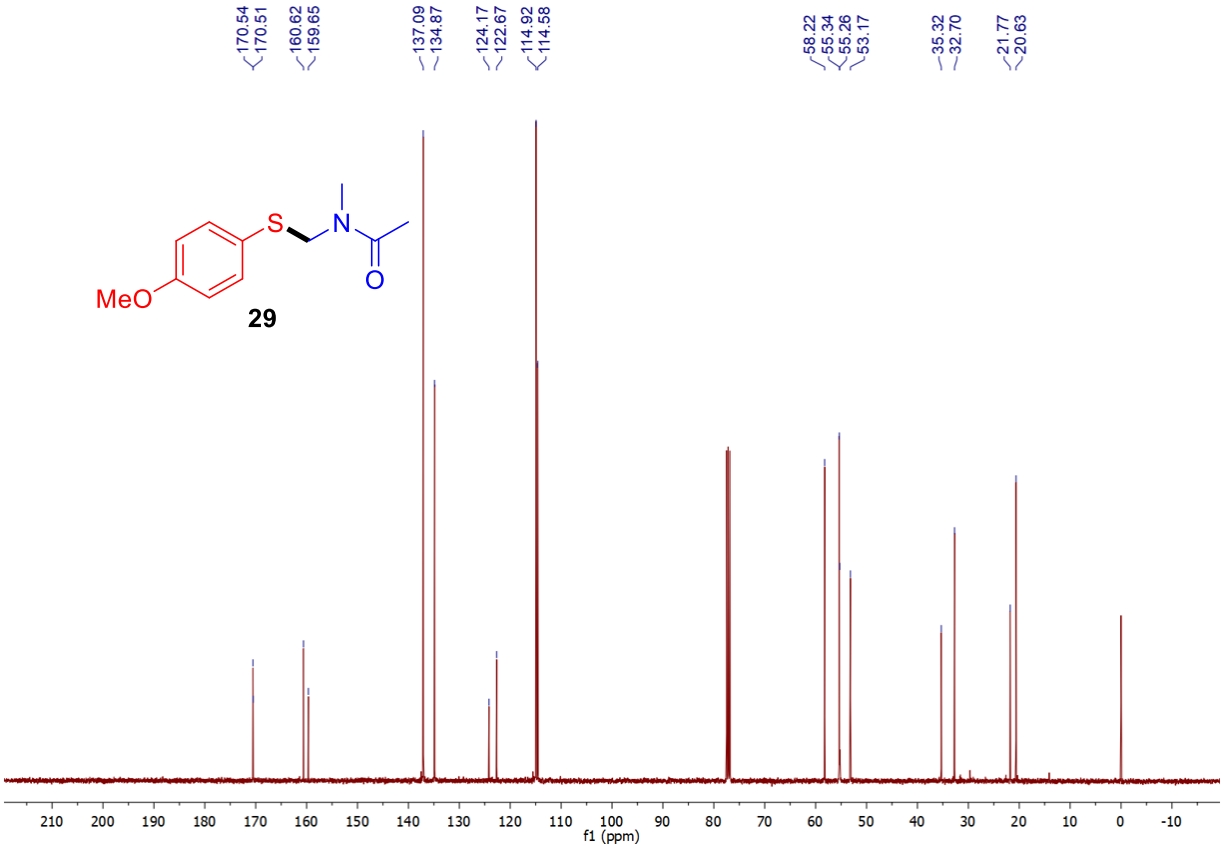
^{13}C-NMR (101 MHz, $CDCl_3$) of **28**



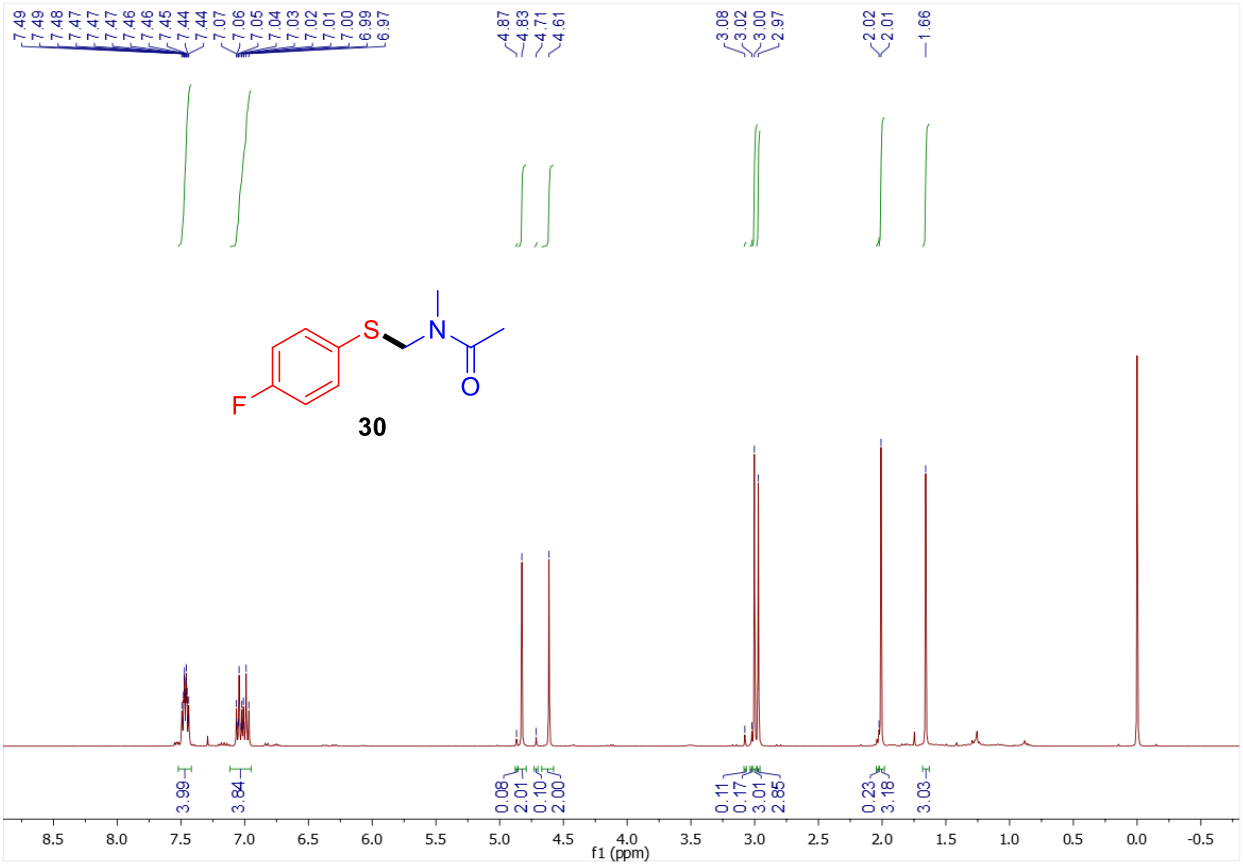
^{1}H-NMR (400 MHz, $CDCl_3$) of **29**



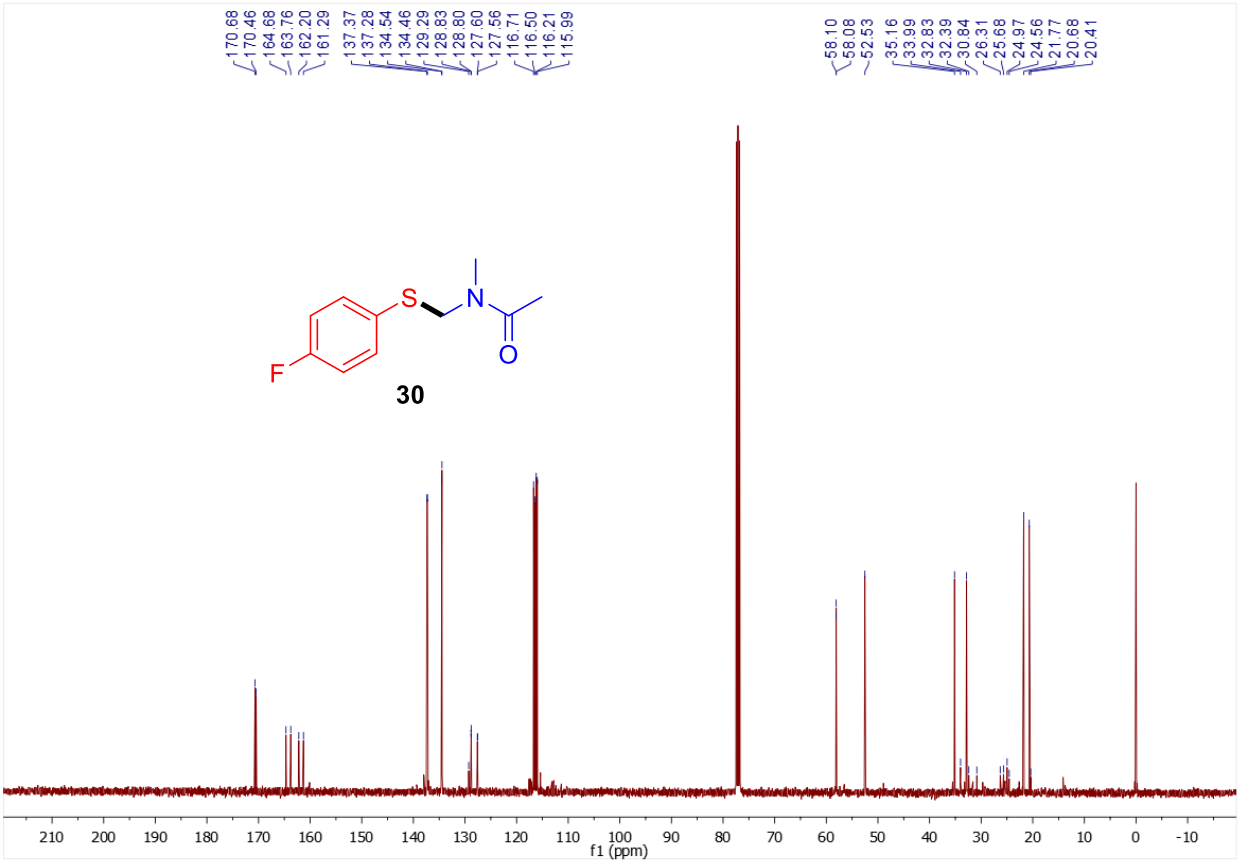
^{13}C-NMR (101 MHz, $CDCl_3$) of **29**



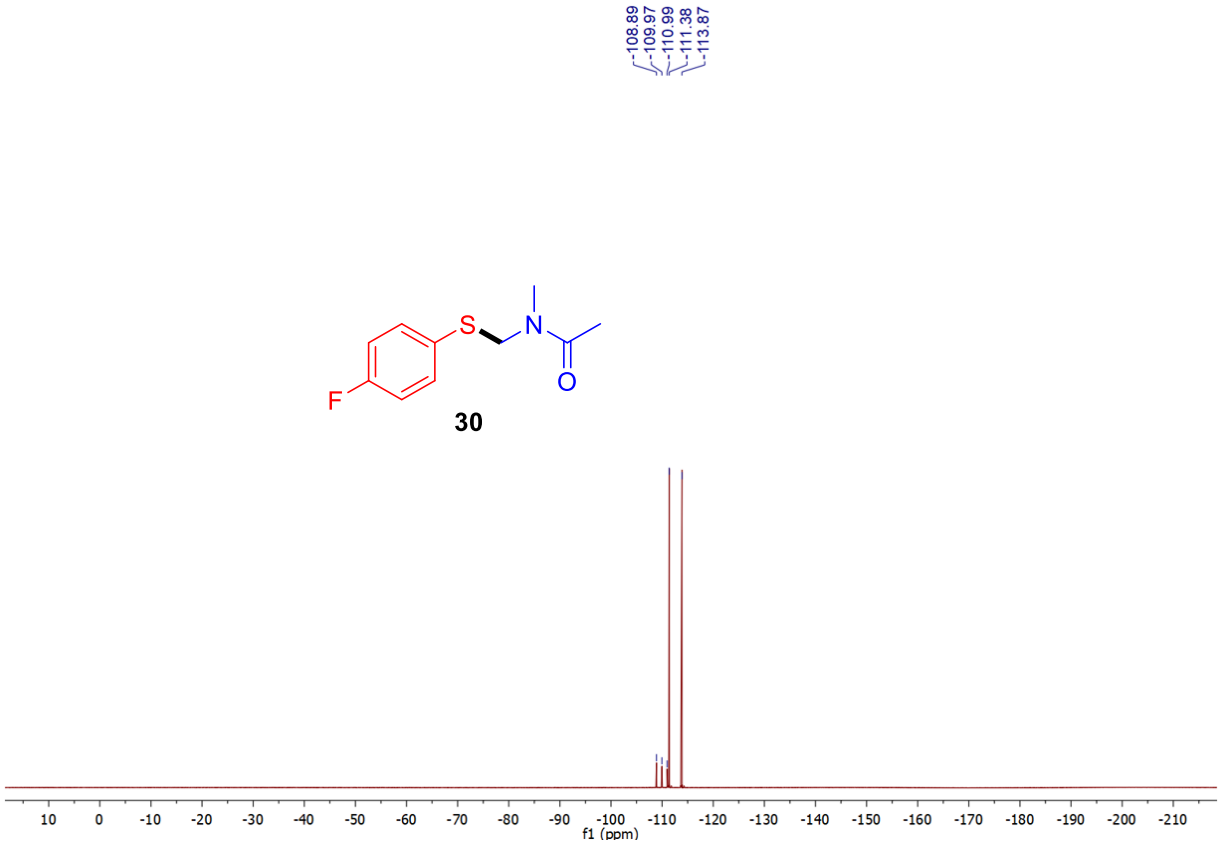
^{1}H-NMR (400 MHz, $CDCl_3$) of **30**



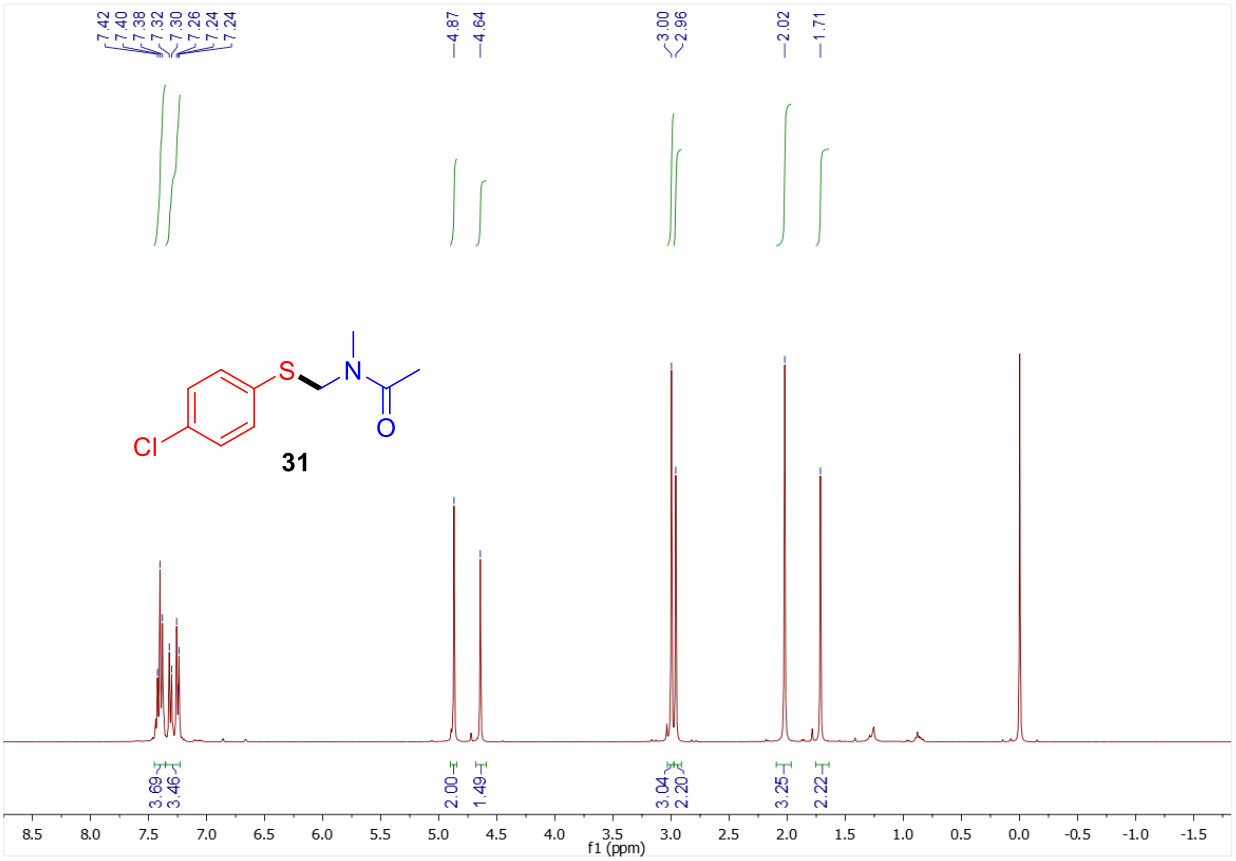
^{13}C-NMR (101 MHz, $CDCl_3$) of **30**



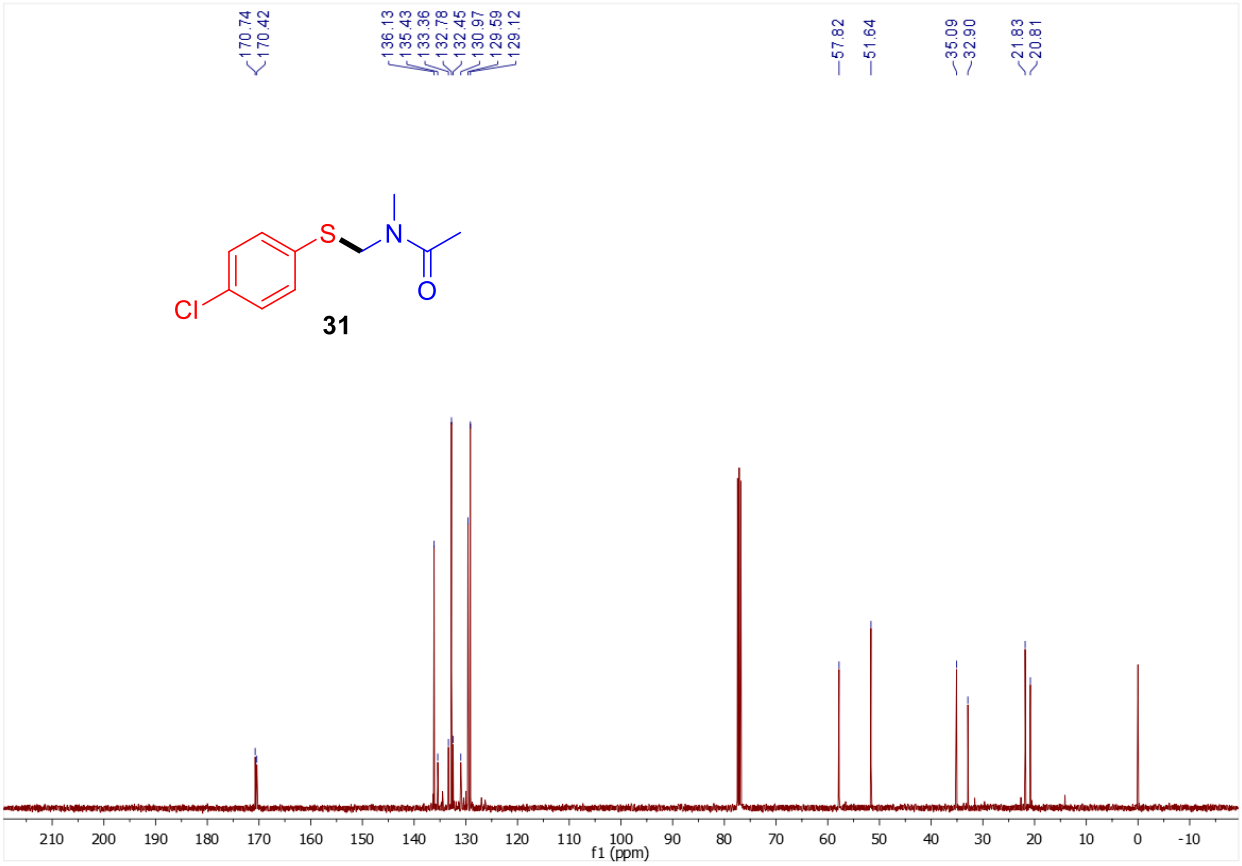
^{19}F NMR (376 MHz, $CDCl_3$) of **30**



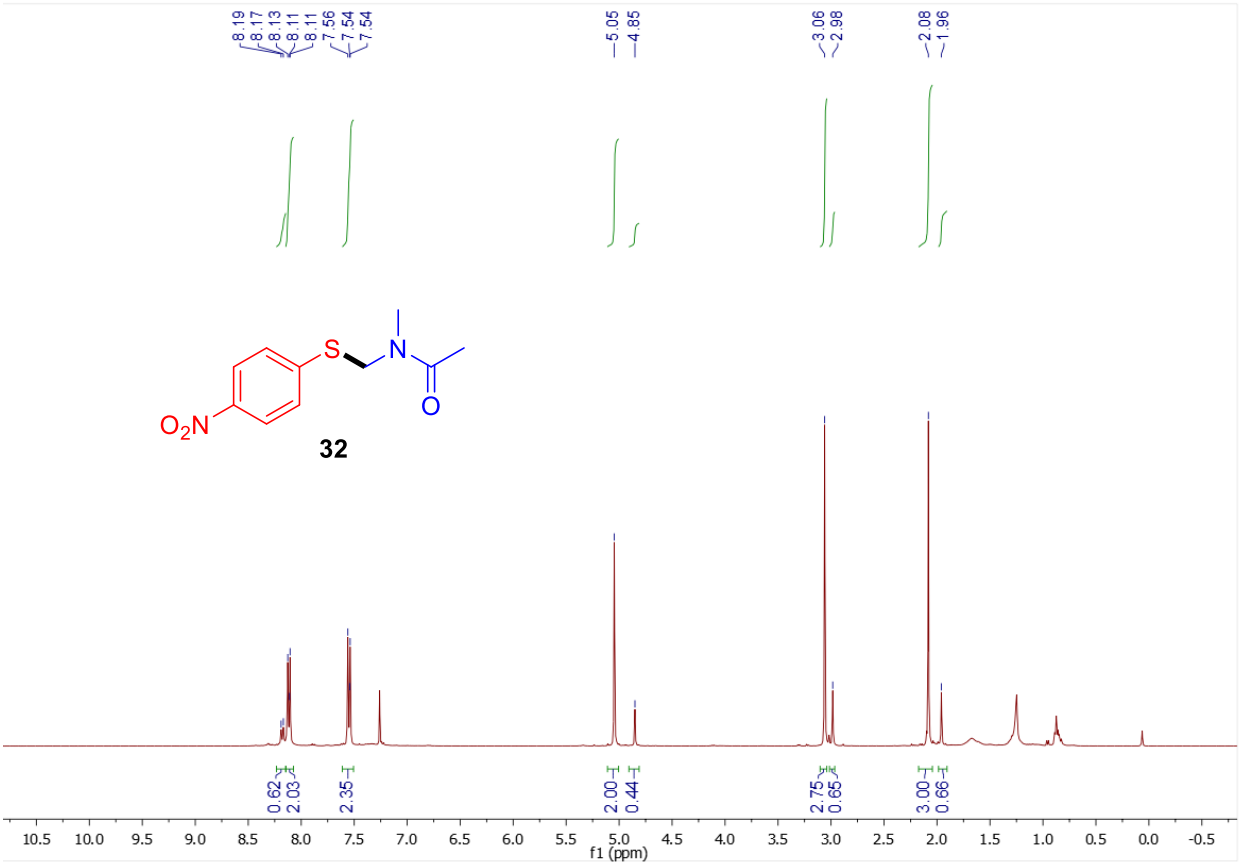
^{1}H-NMR (400 MHz, $CDCl_3$) of **31**



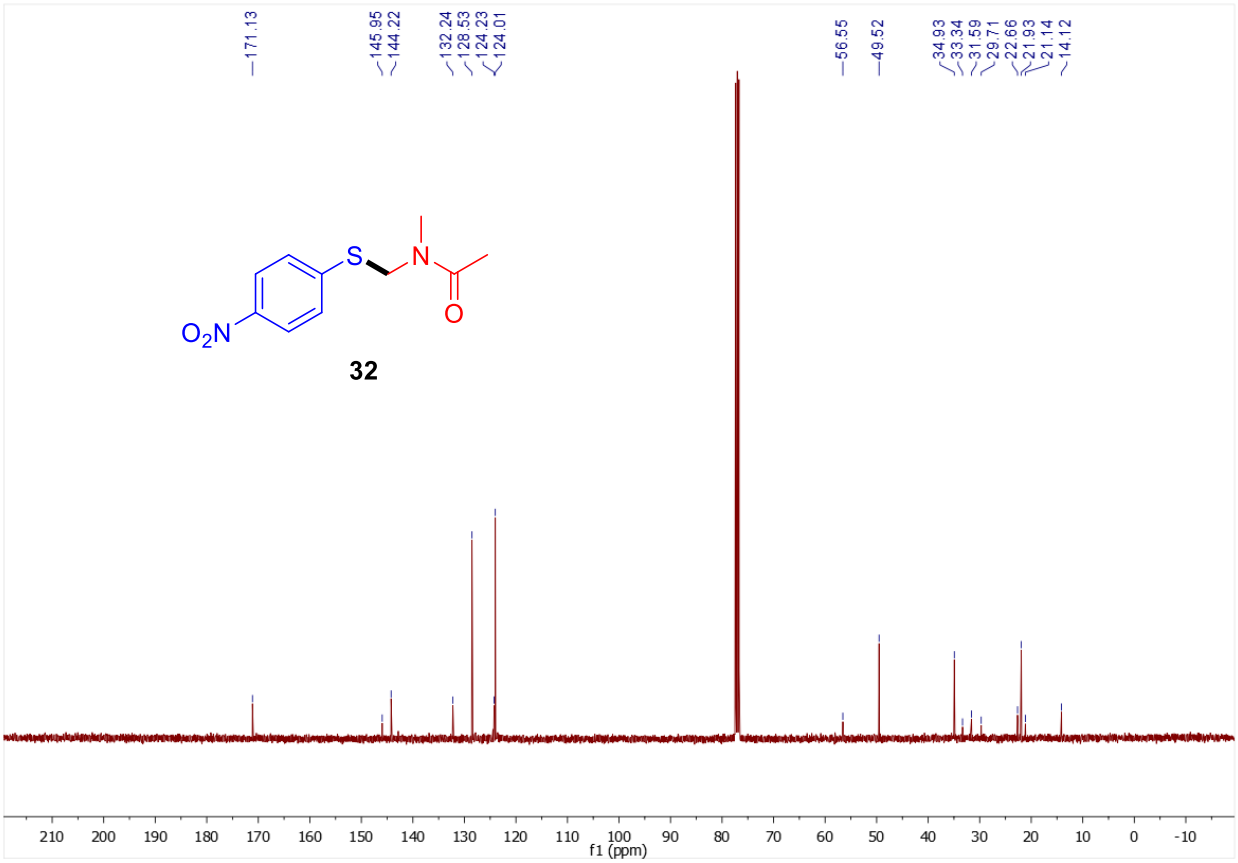
^{13}C-NMR (101 MHz, $CDCl_3$) of **31**



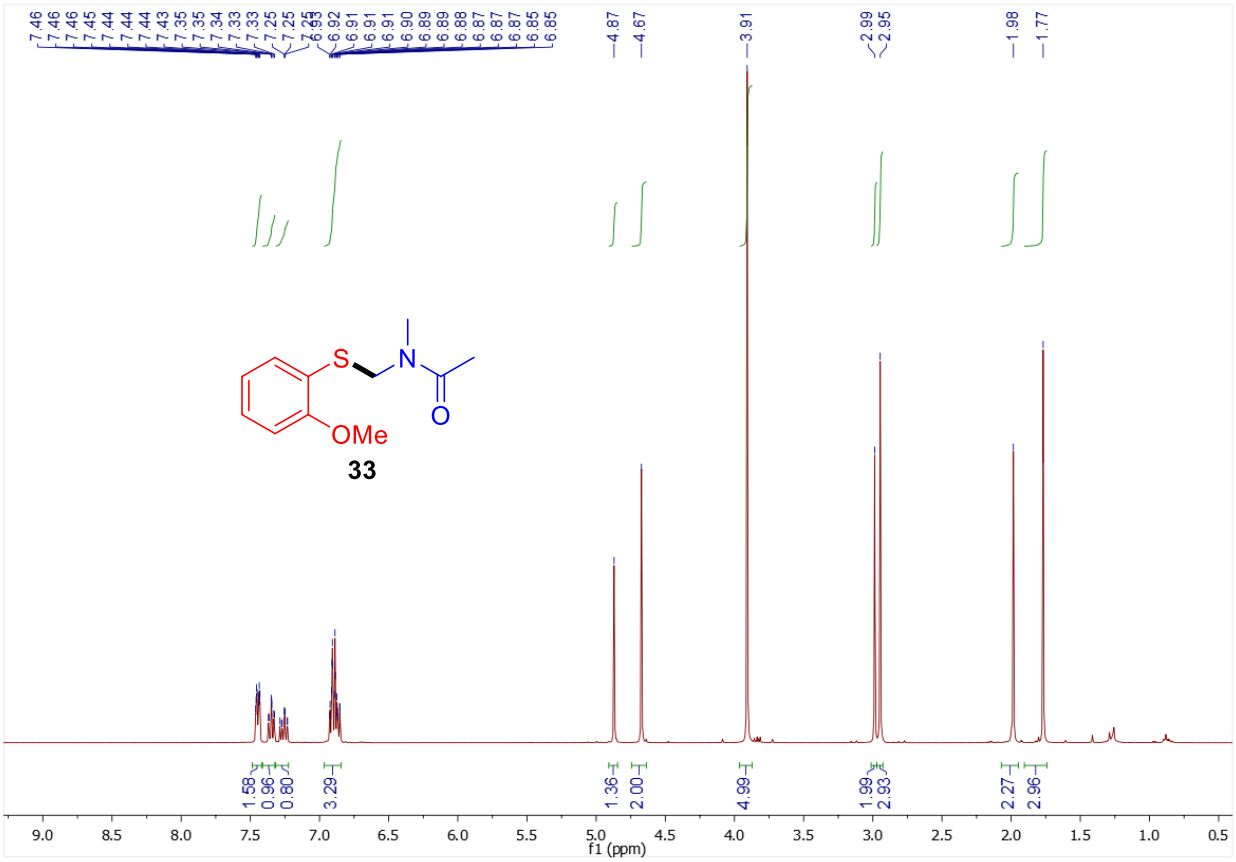
^{1}H-NMR (400 MHz, $CDCl_3$) of **32**



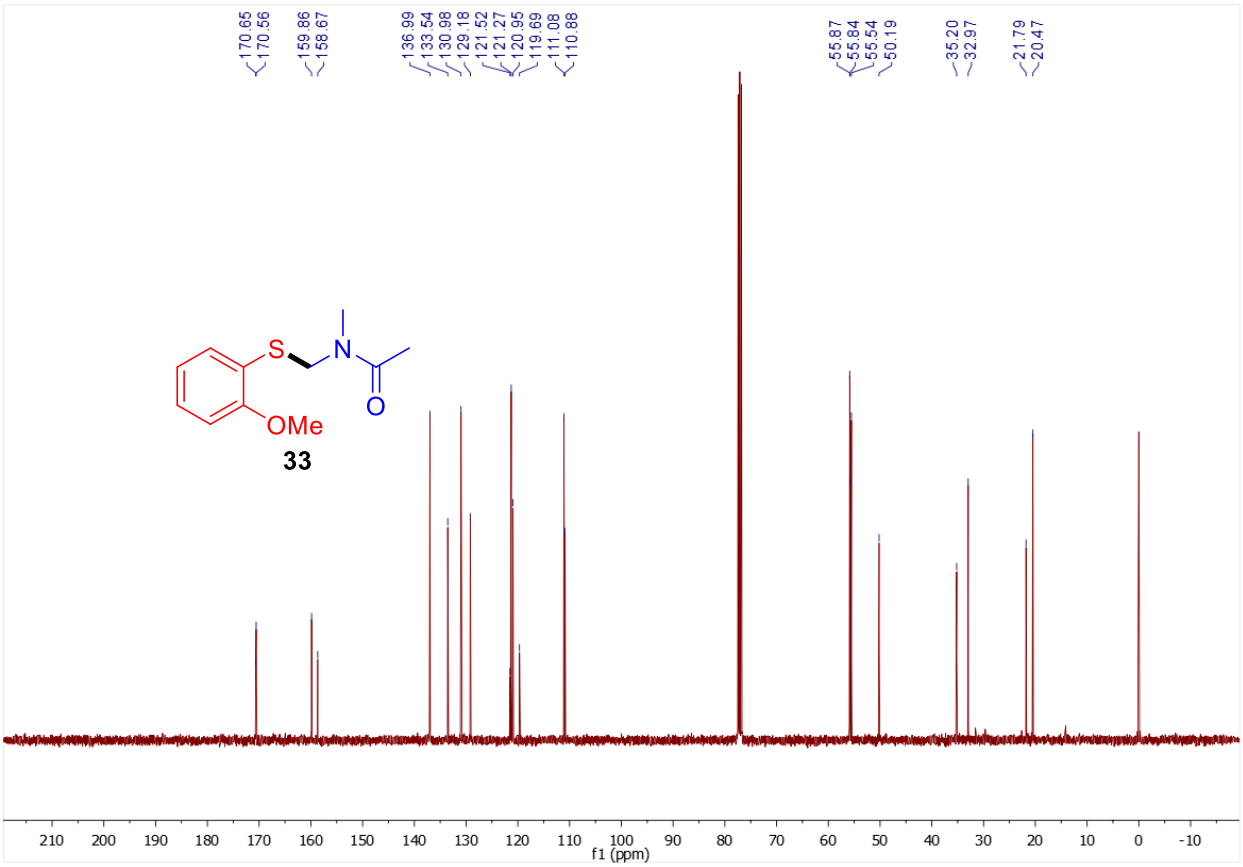
^{13}C-NMR (101 MHz, $CDCl_3$) of **32**



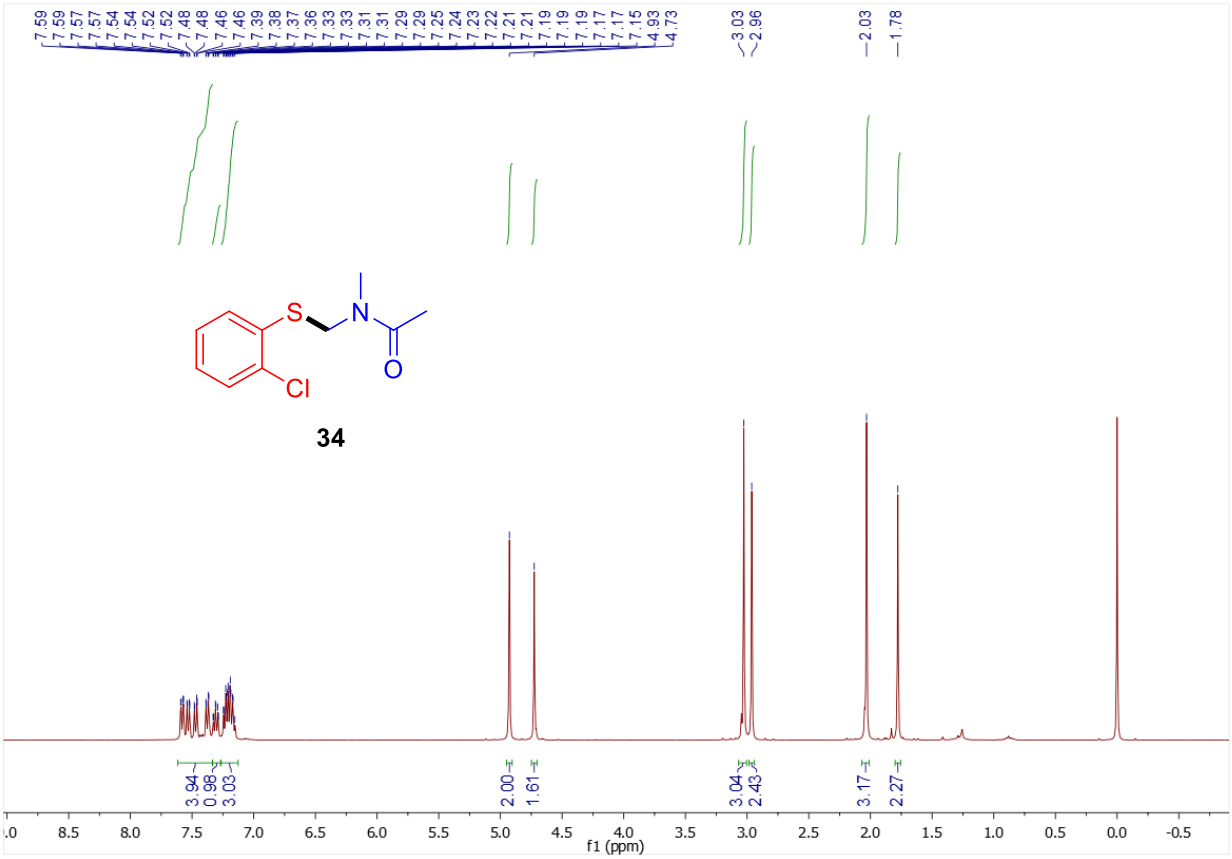
^{1}H-NMR (400 MHz, $CDCl_3$) of **33**



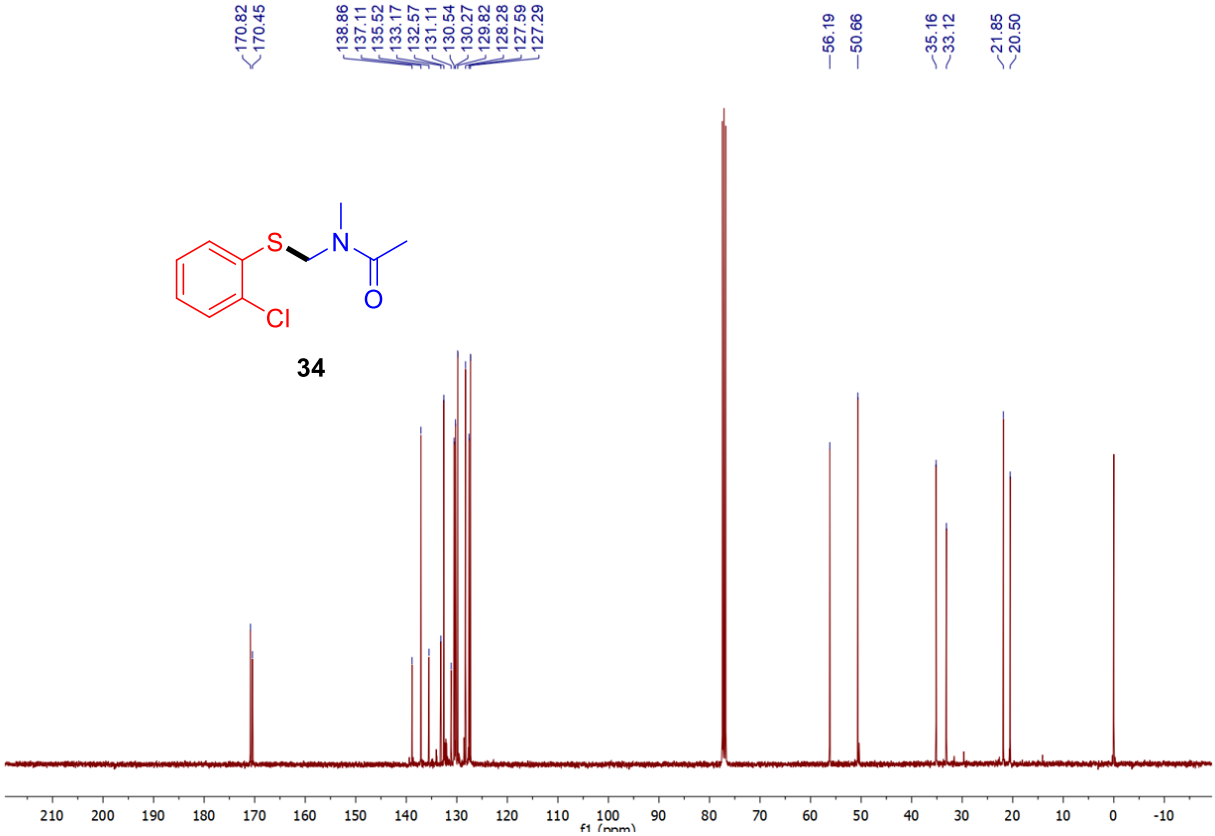
^{13}C-NMR (101 MHz, $CDCl_3$) of **33**



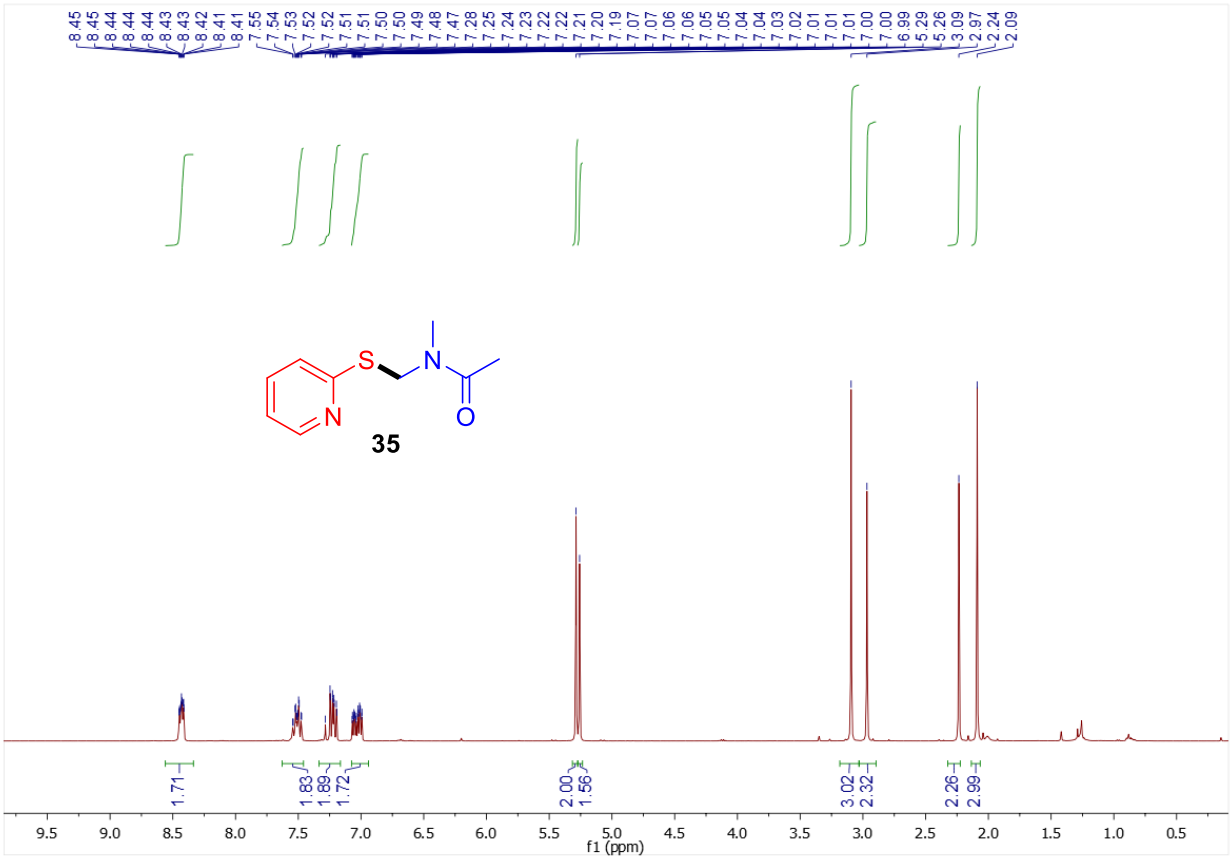
^{1}H-NMR (400 MHz, $CDCl_3$) of **34**



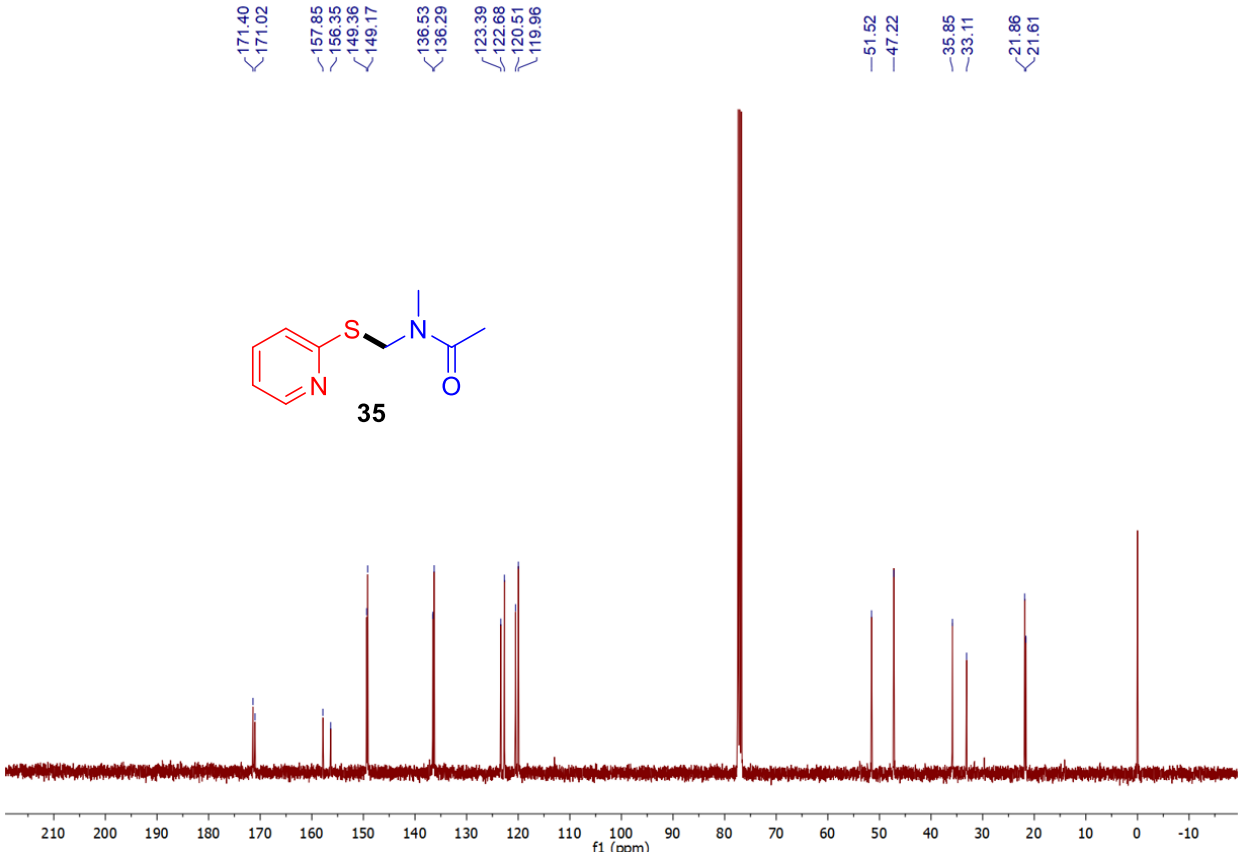
^{13}C-NMR (101 MHz, $CDCl_3$) of **34**



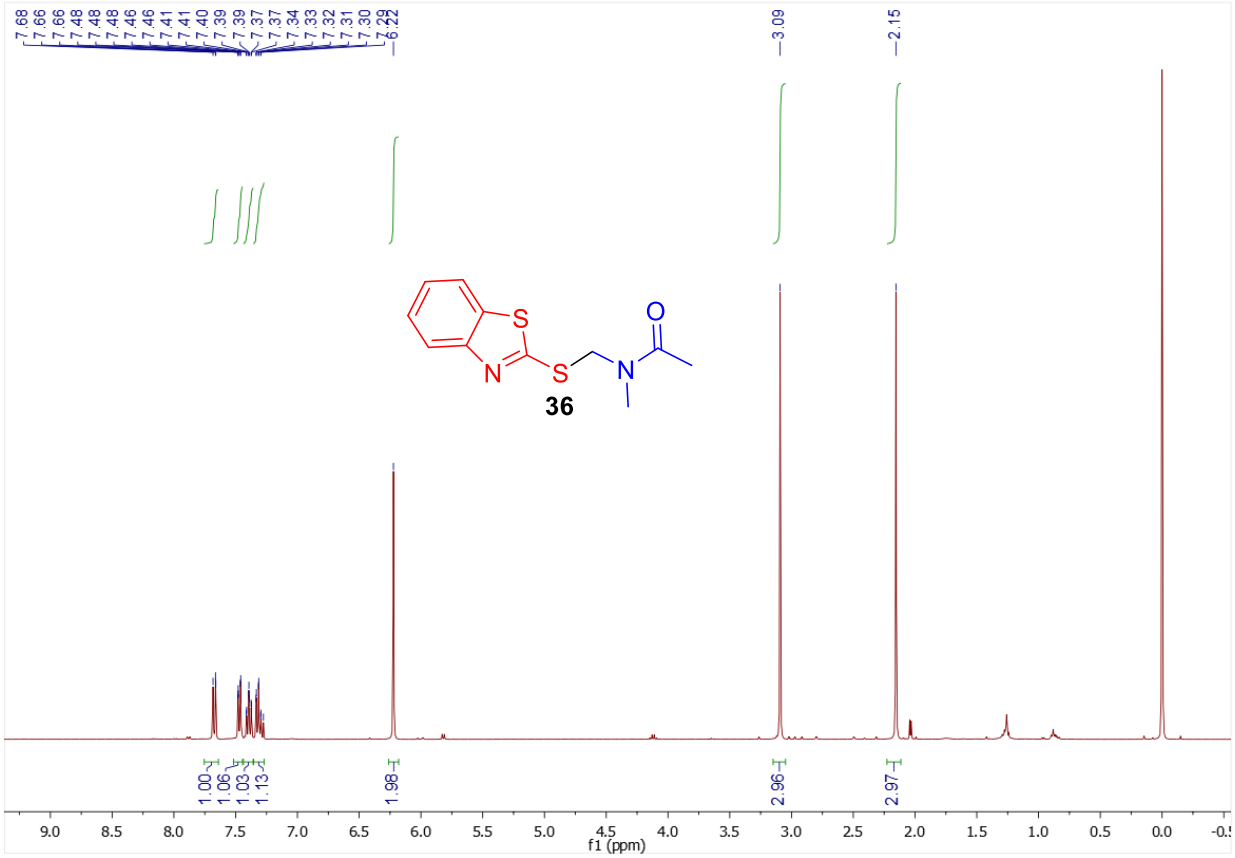
^{1}H-NMR (400 MHz, $CDCl_3$) of **35**



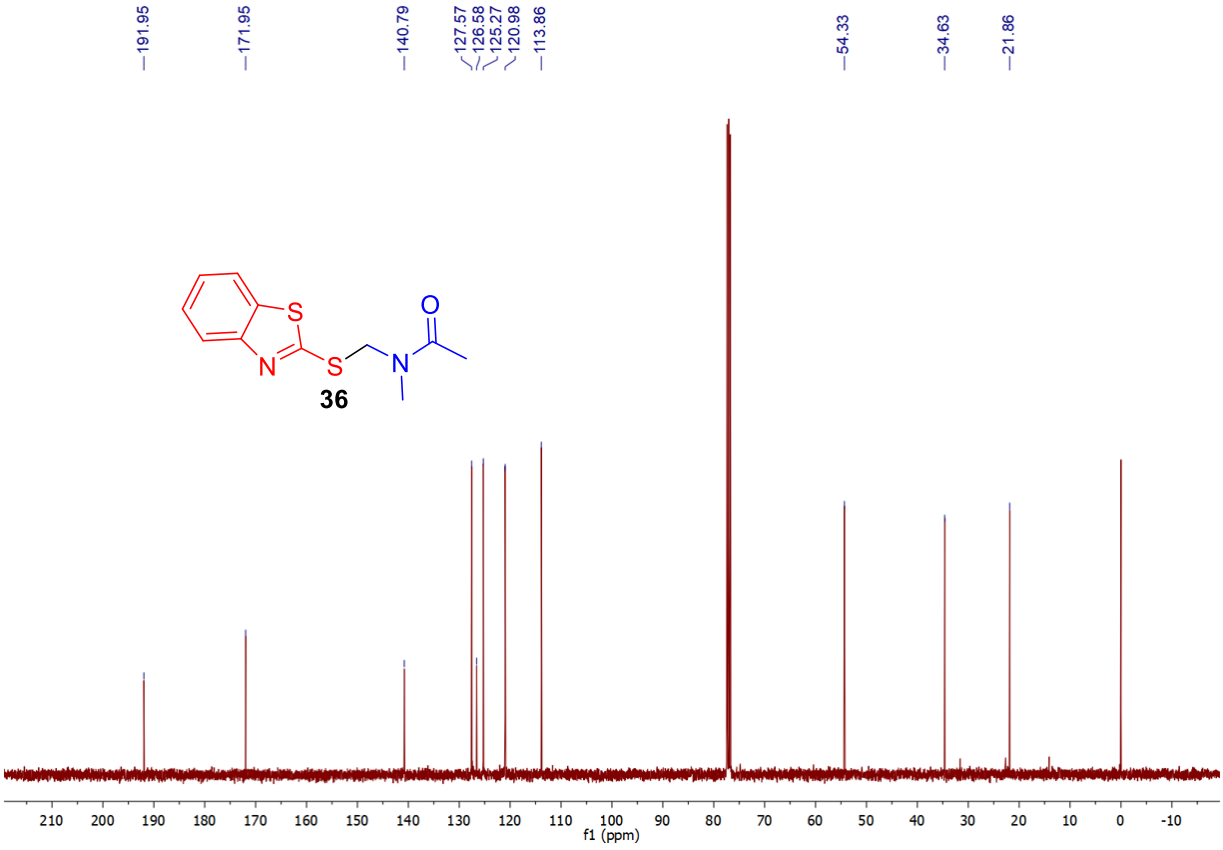
^{13}C-NMR (101 MHz, $CDCl_3$) of **35**



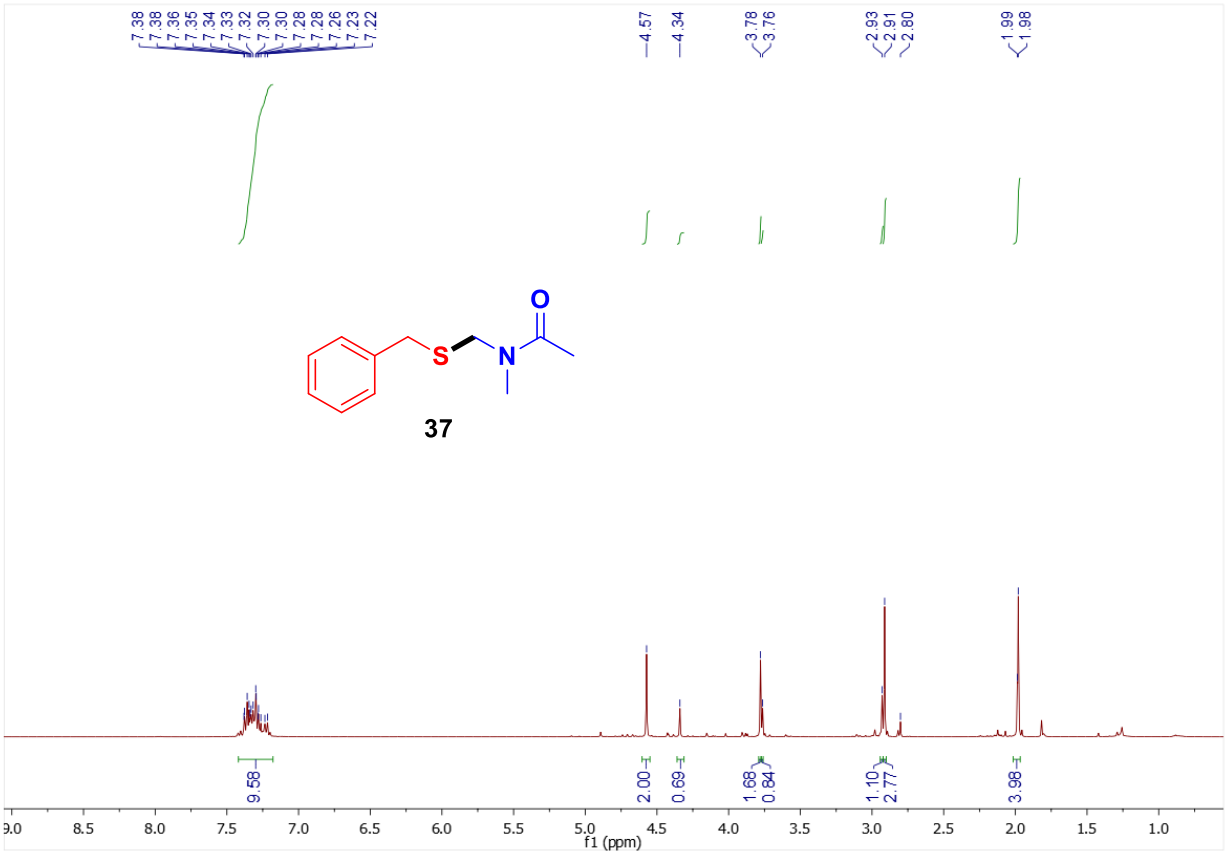
^{1}H-NMR (400 MHz, $CDCl_3$) of **36**



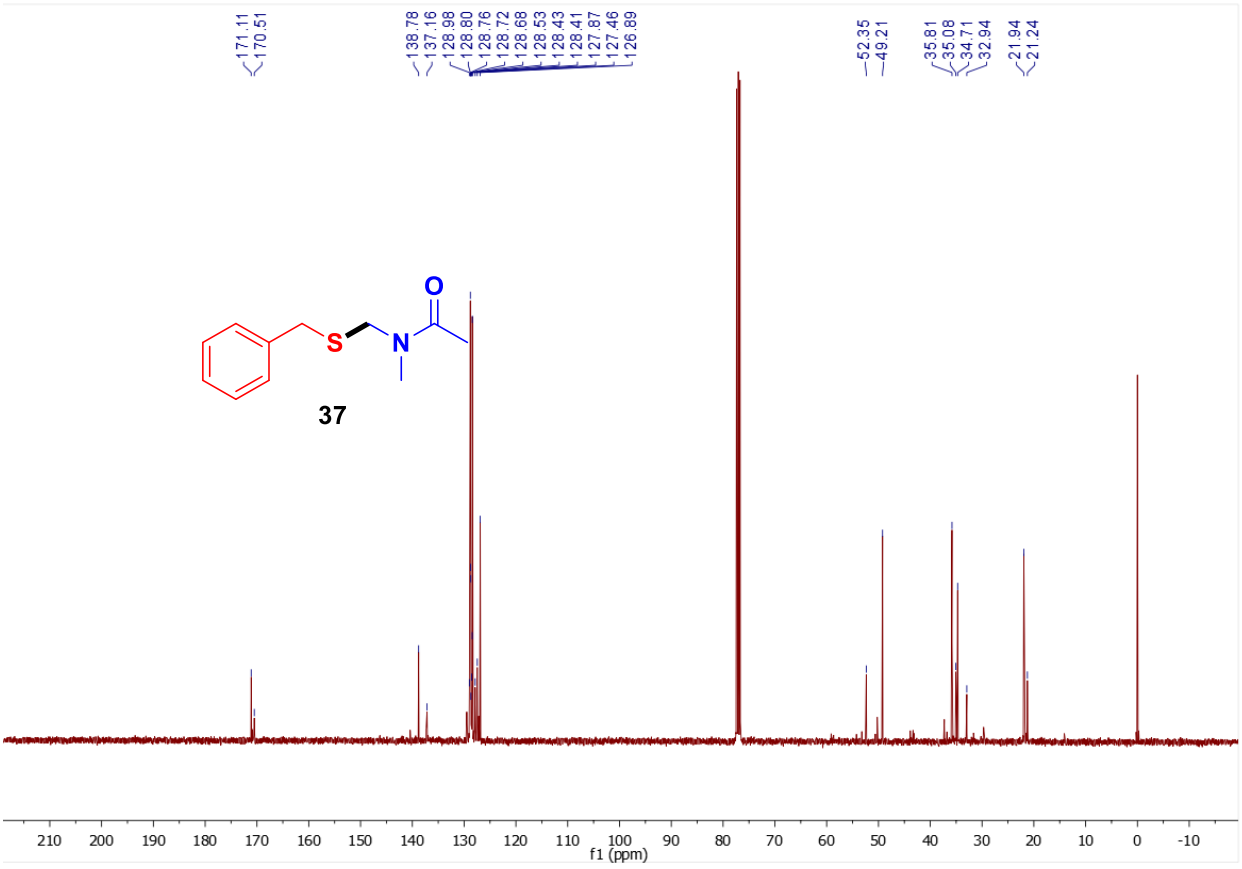
^{13}C-NMR (101 MHz, $CDCl_3$) of **36**



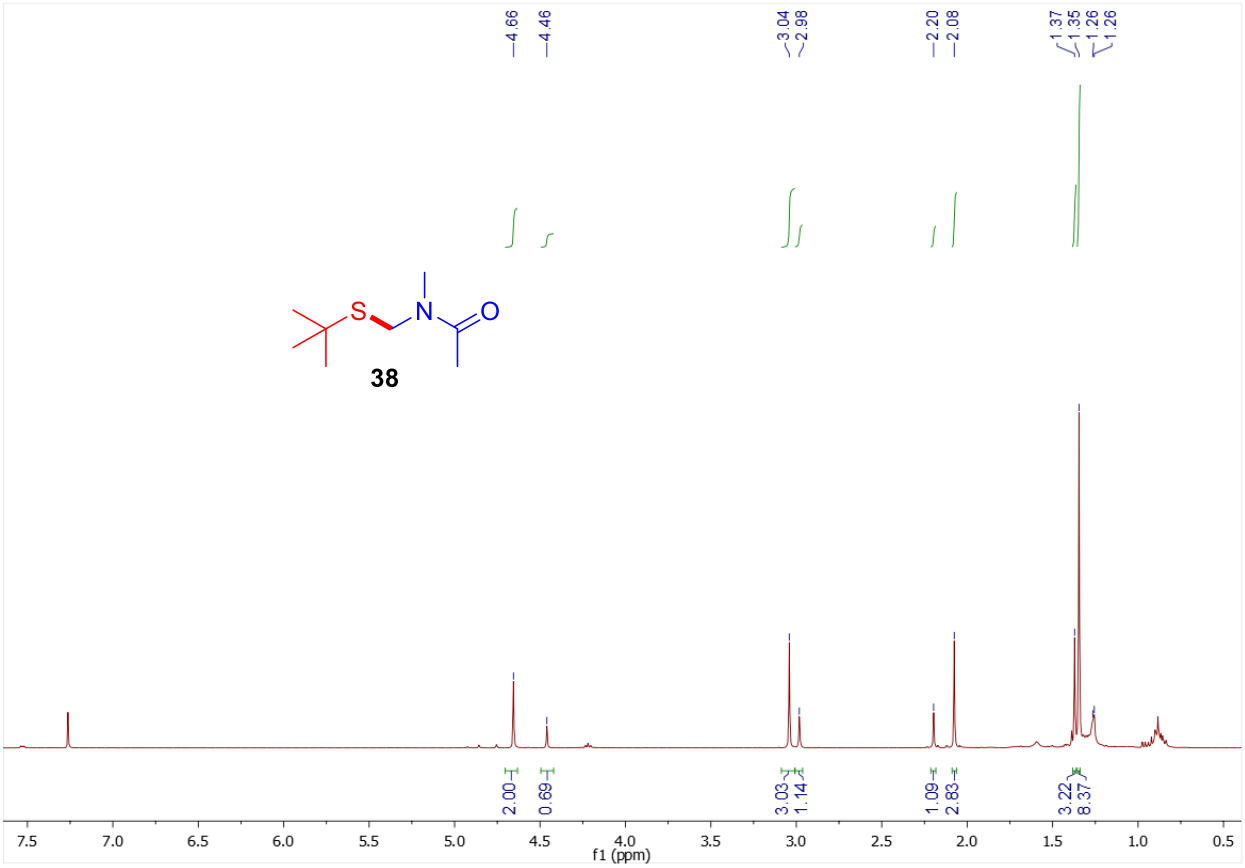
^{1}H-NMR (400 MHz, $CDCl_3$) of **37**



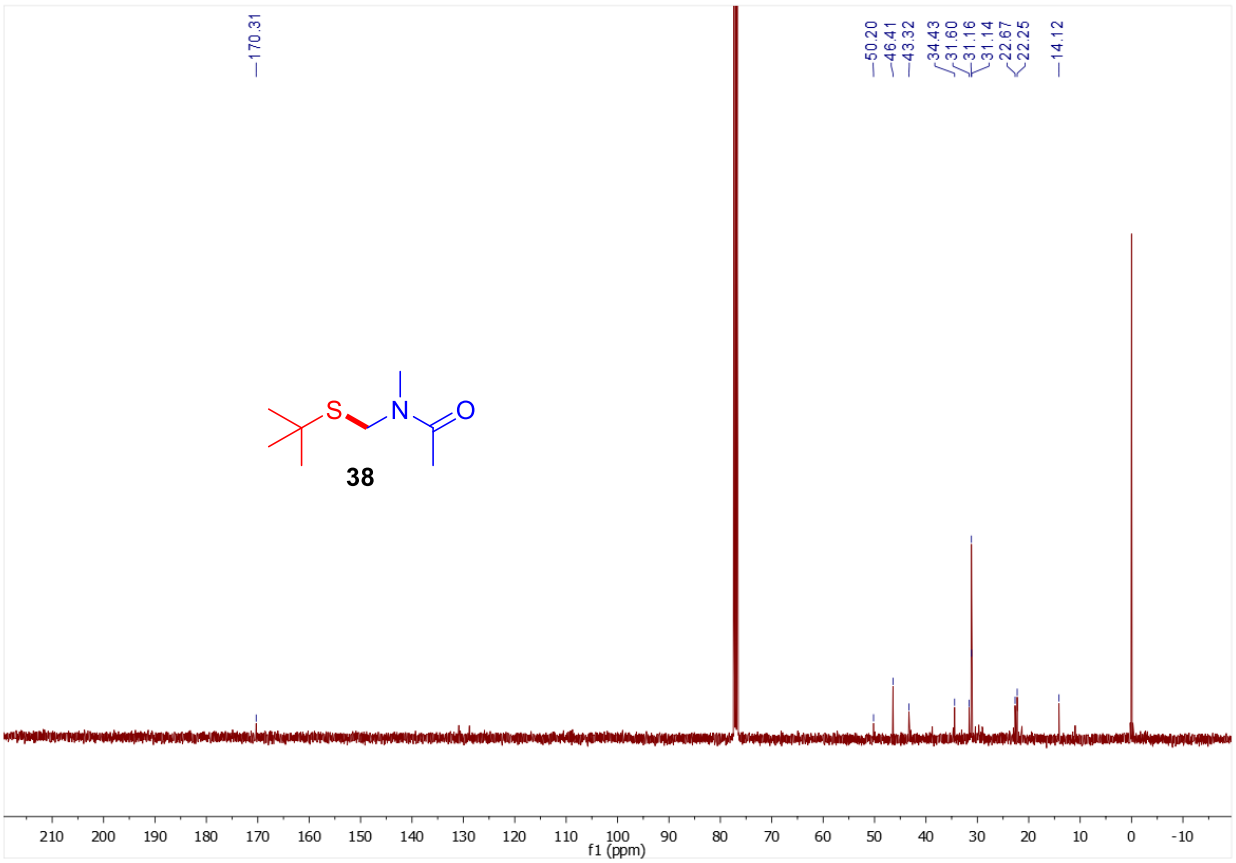
^{13}C-NMR (101 MHz, $CDCl_3$) of **37**



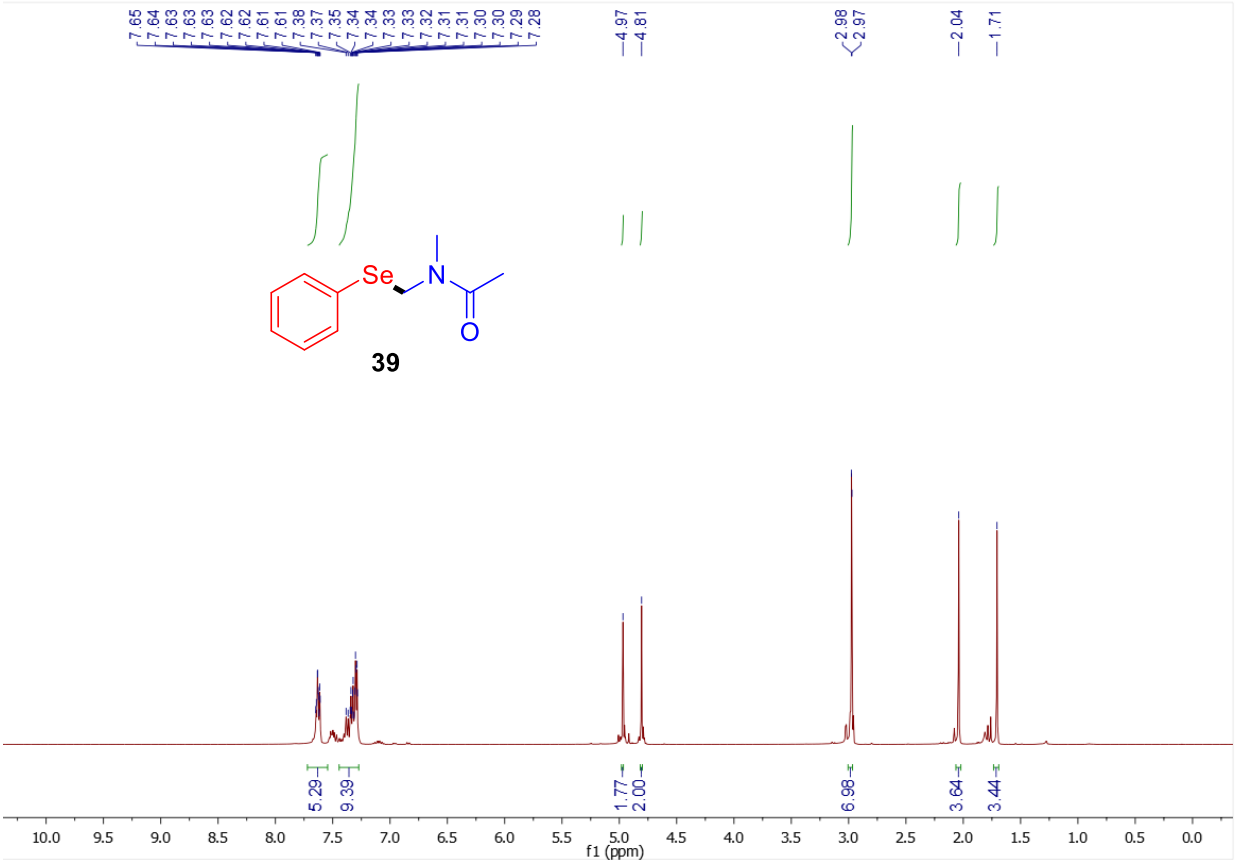
^{1}H-NMR (400 MHz, $CDCl_3$) of **38**



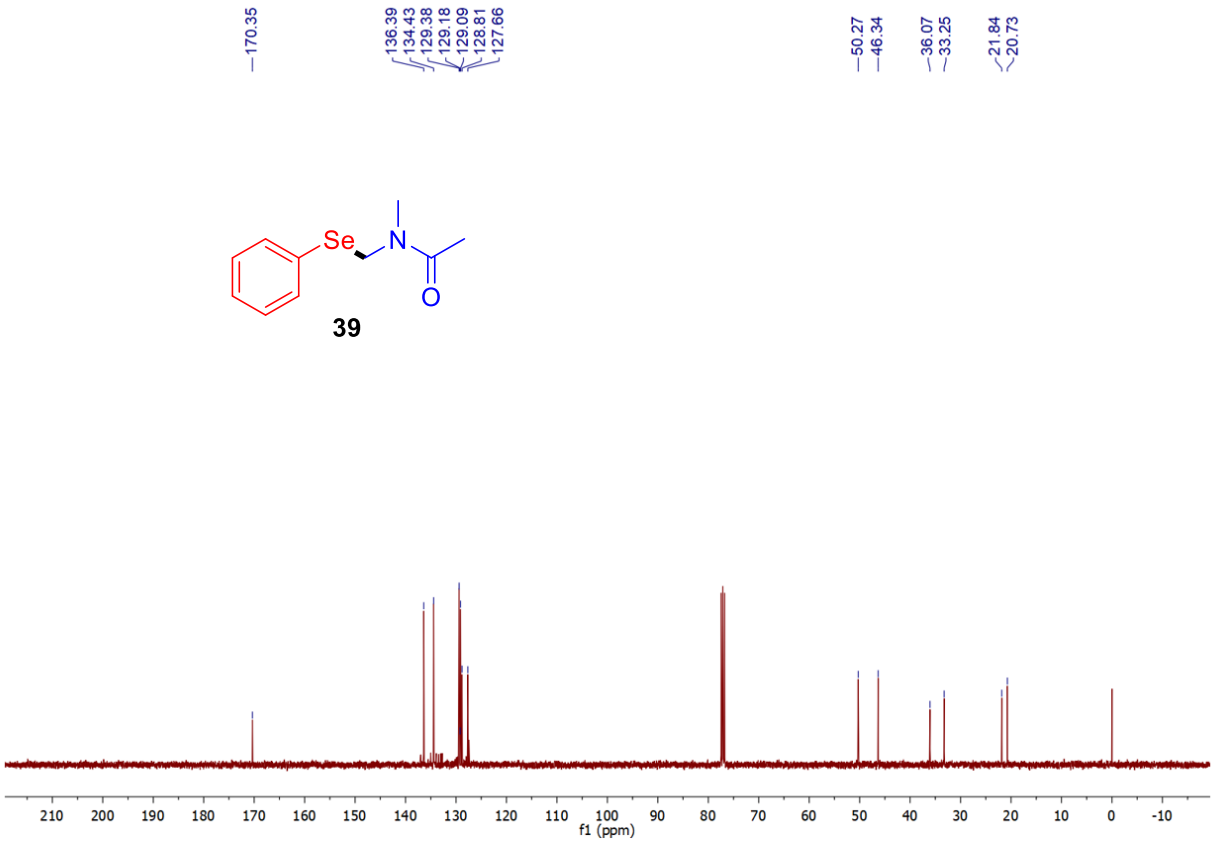
^{13}C-NMR (101 MHz, $CDCl_3$) of **38**



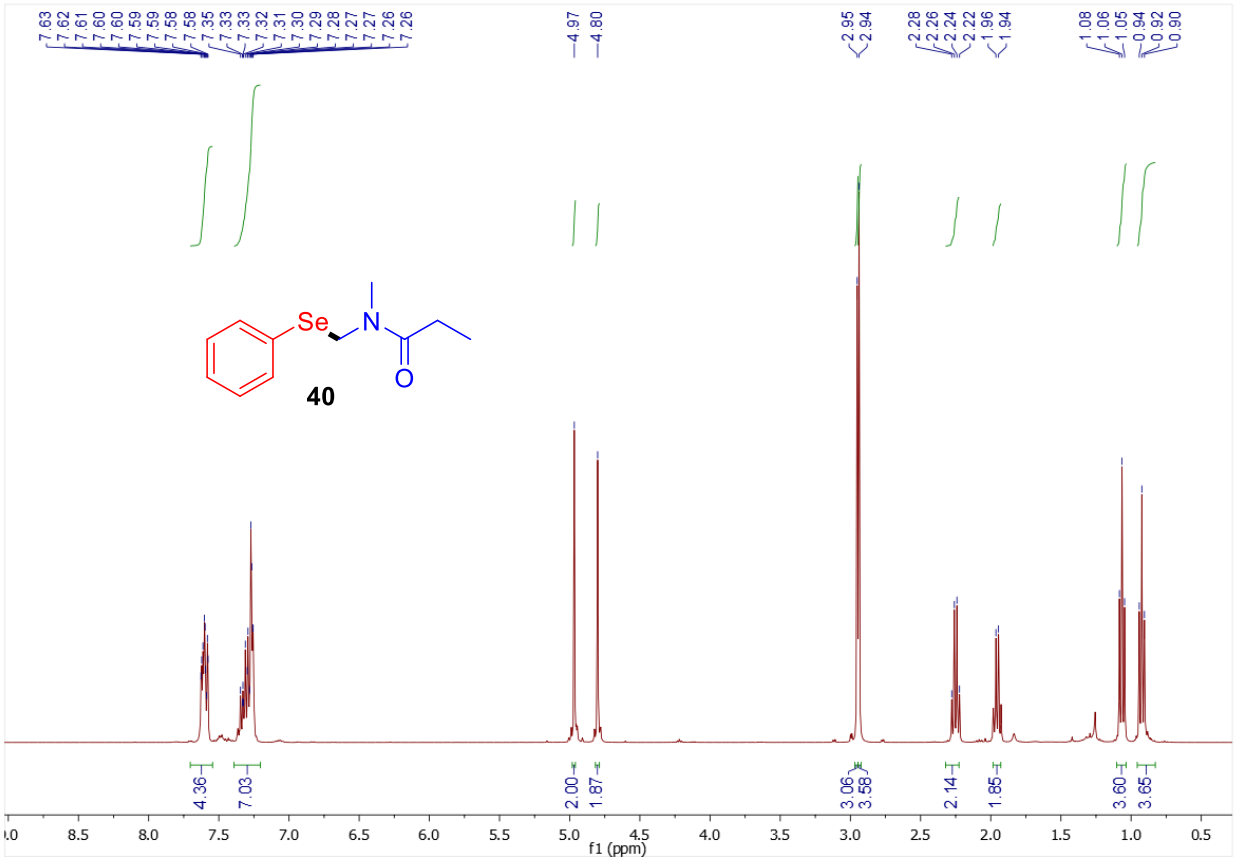
^{1}H-NMR (400 MHz, $CDCl_3$) of **39**



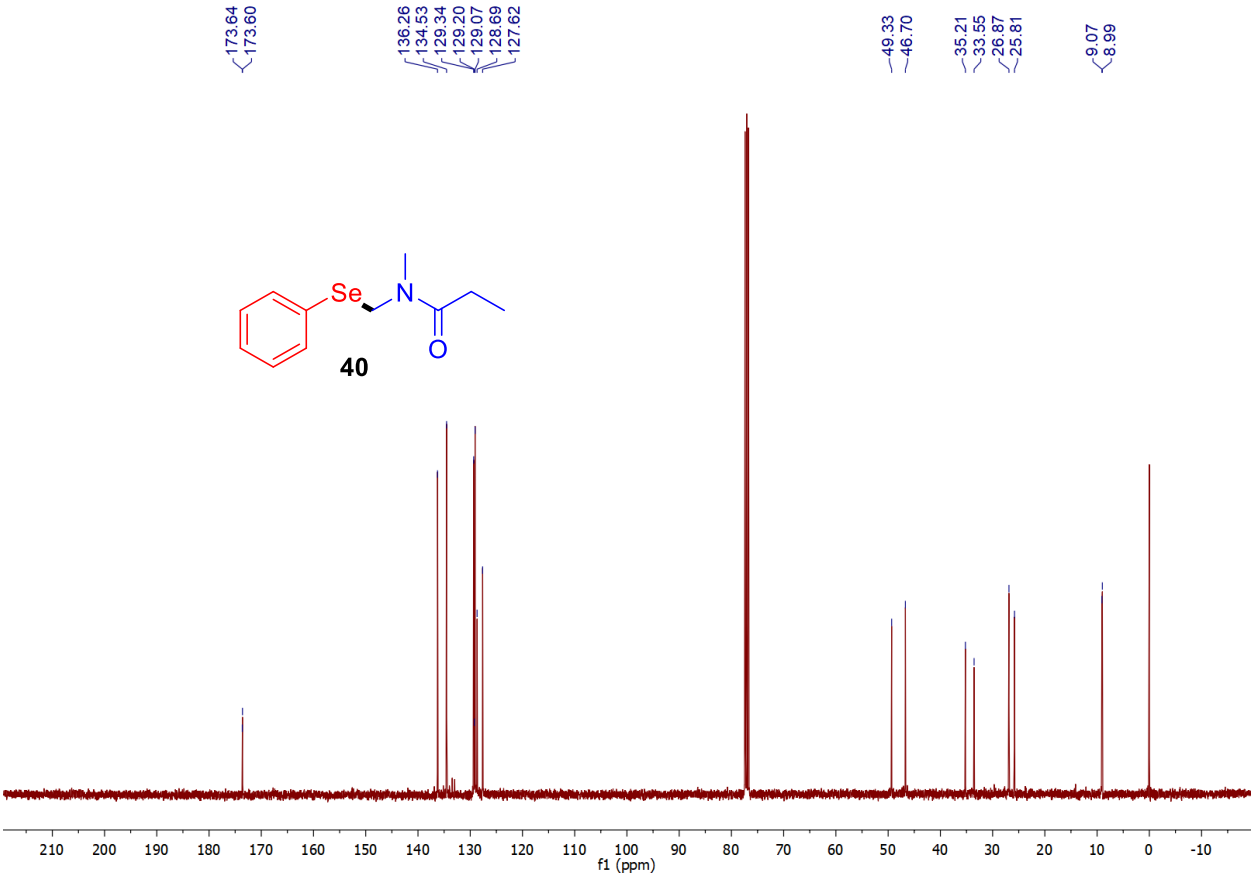
^{13}C-NMR (101 MHz, $CDCl_3$) of **39**



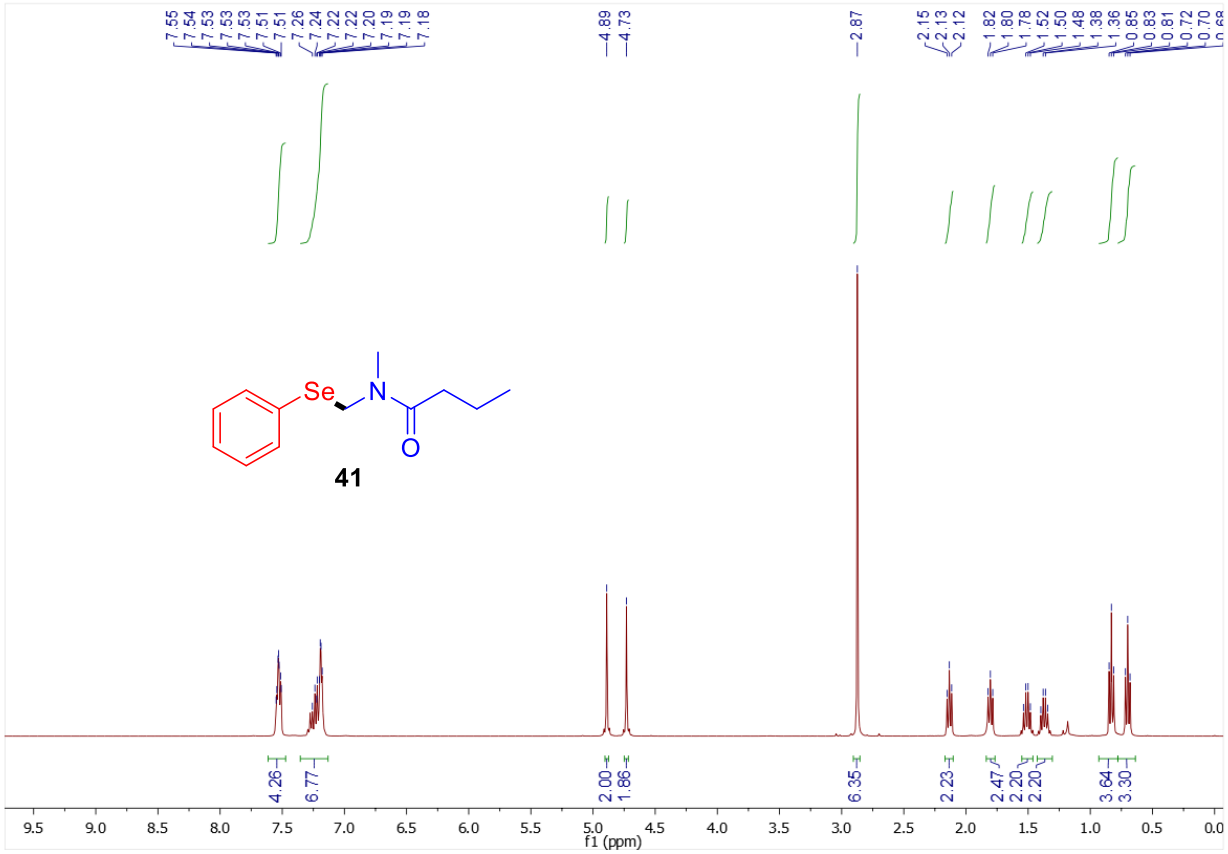
^{1}H-NMR (400 MHz, $CDCl_3$) of **40**



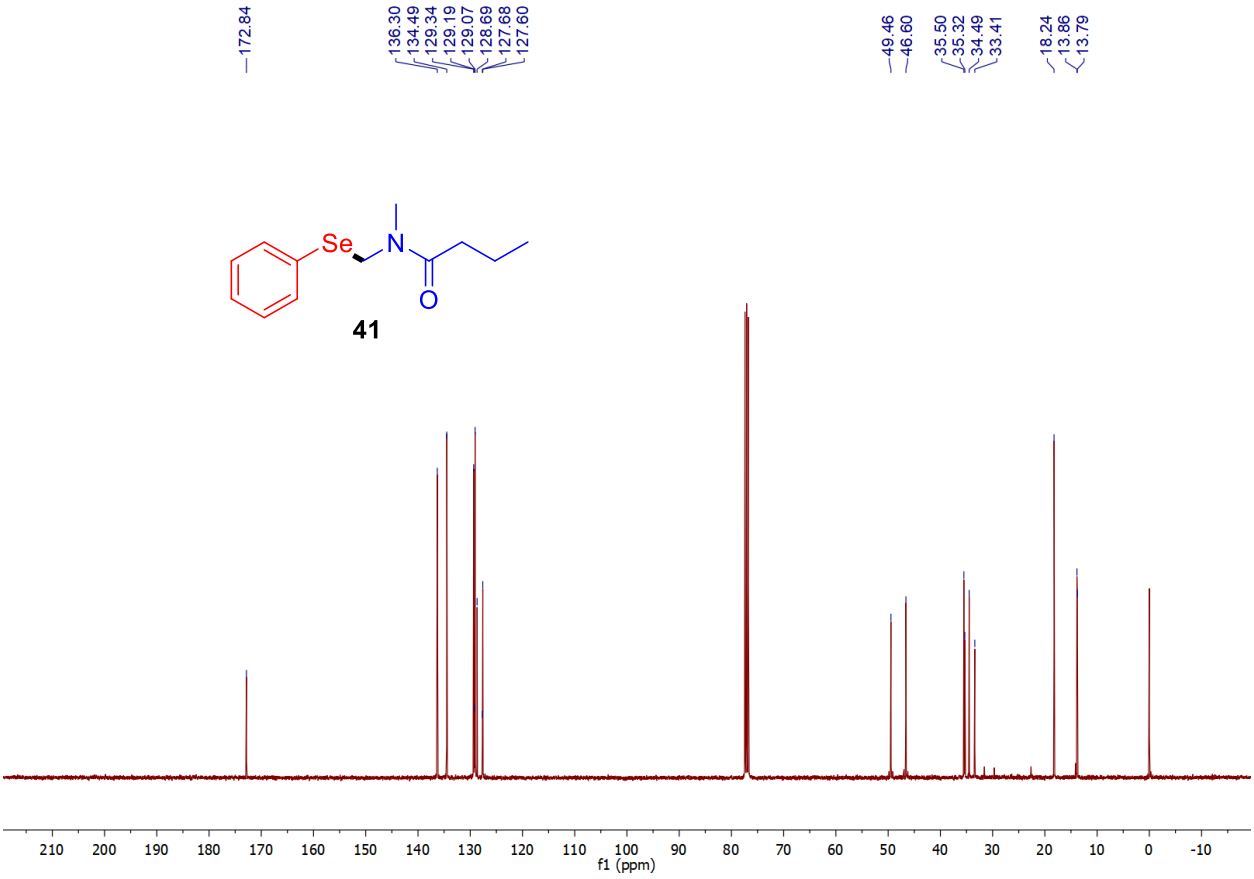
^{13}C-NMR (101 MHz, $CDCl_3$) of **40**



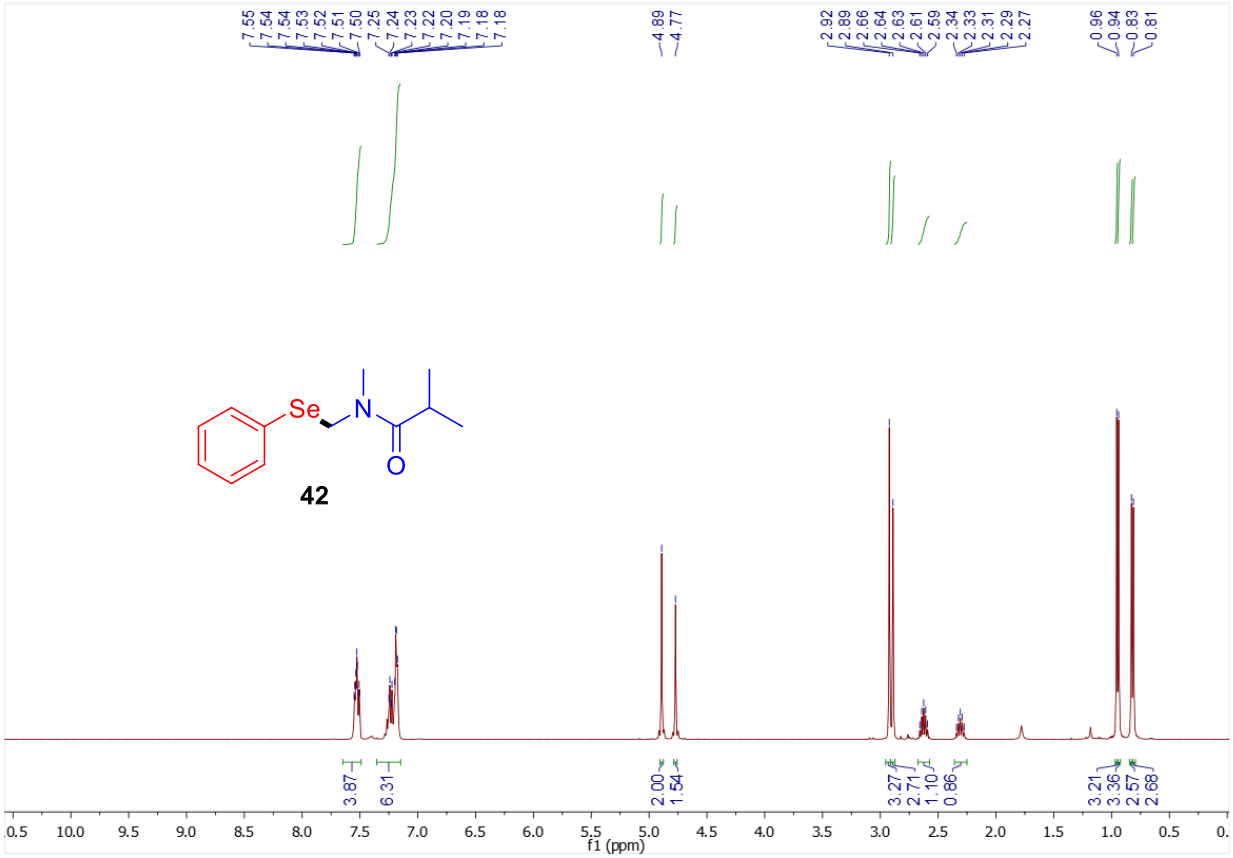
^{1}H-NMR (400 MHz, $CDCl_3$) of **41**



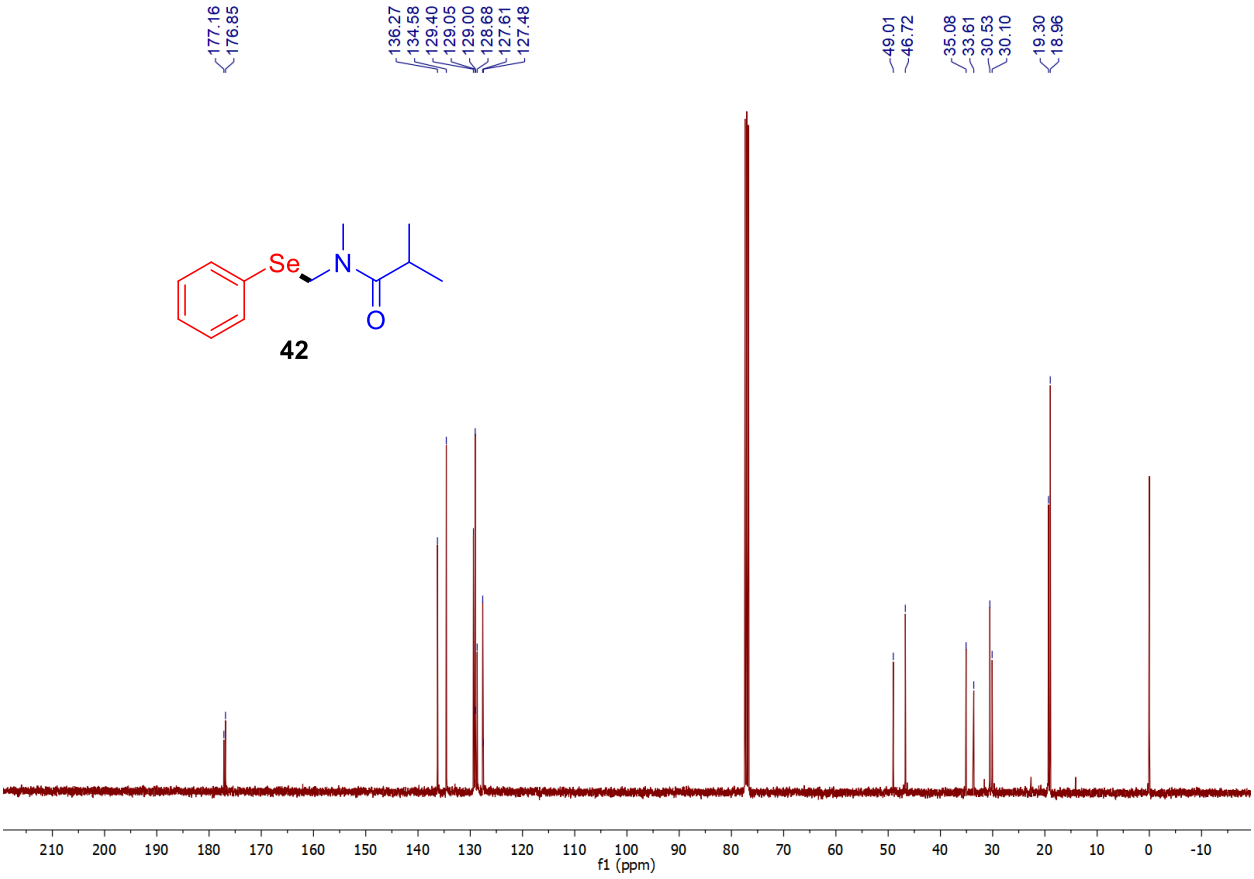
^{13}C-NMR (101 MHz, $CDCl_3$) of **41**



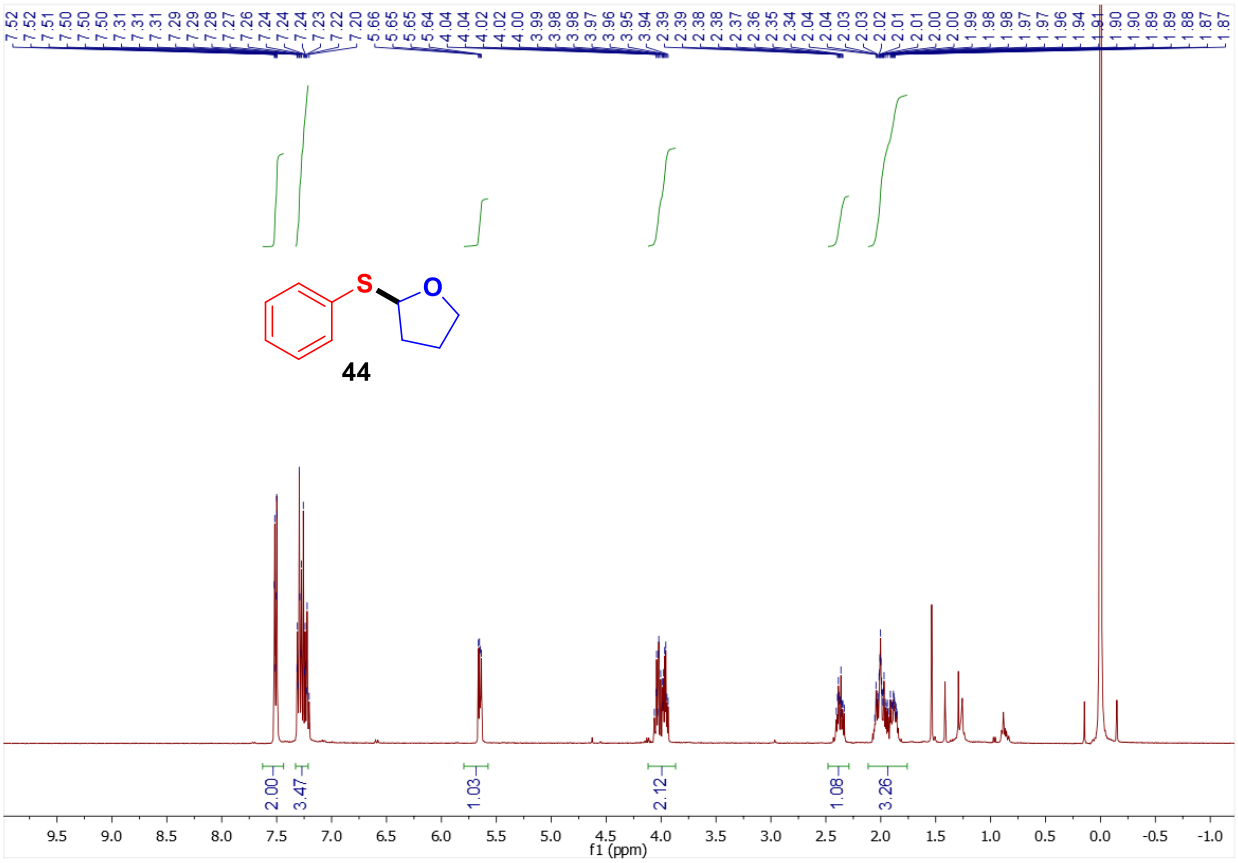
^{1}H-NMR (400 MHz, $CDCl_3$) of **42**



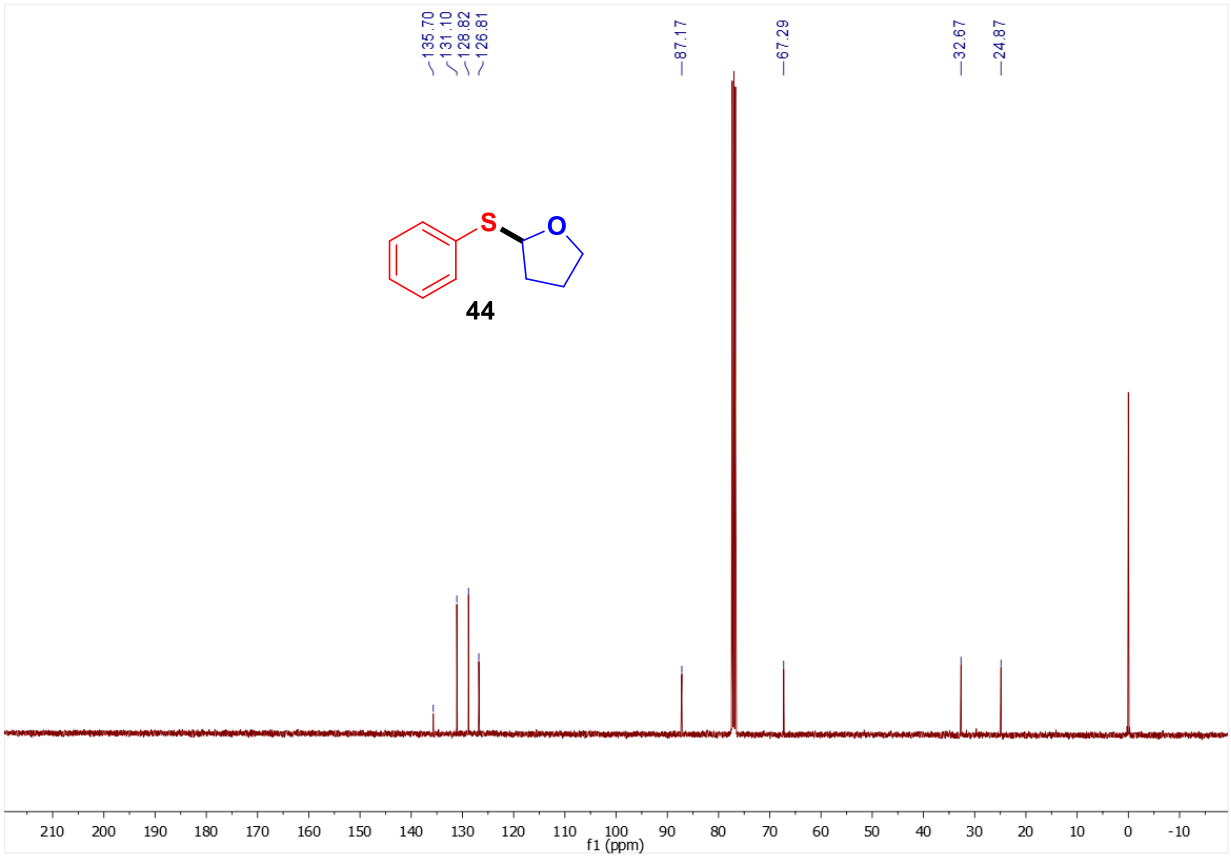
^{13}C-NMR (101 MHz, $CDCl_3$) of **42**



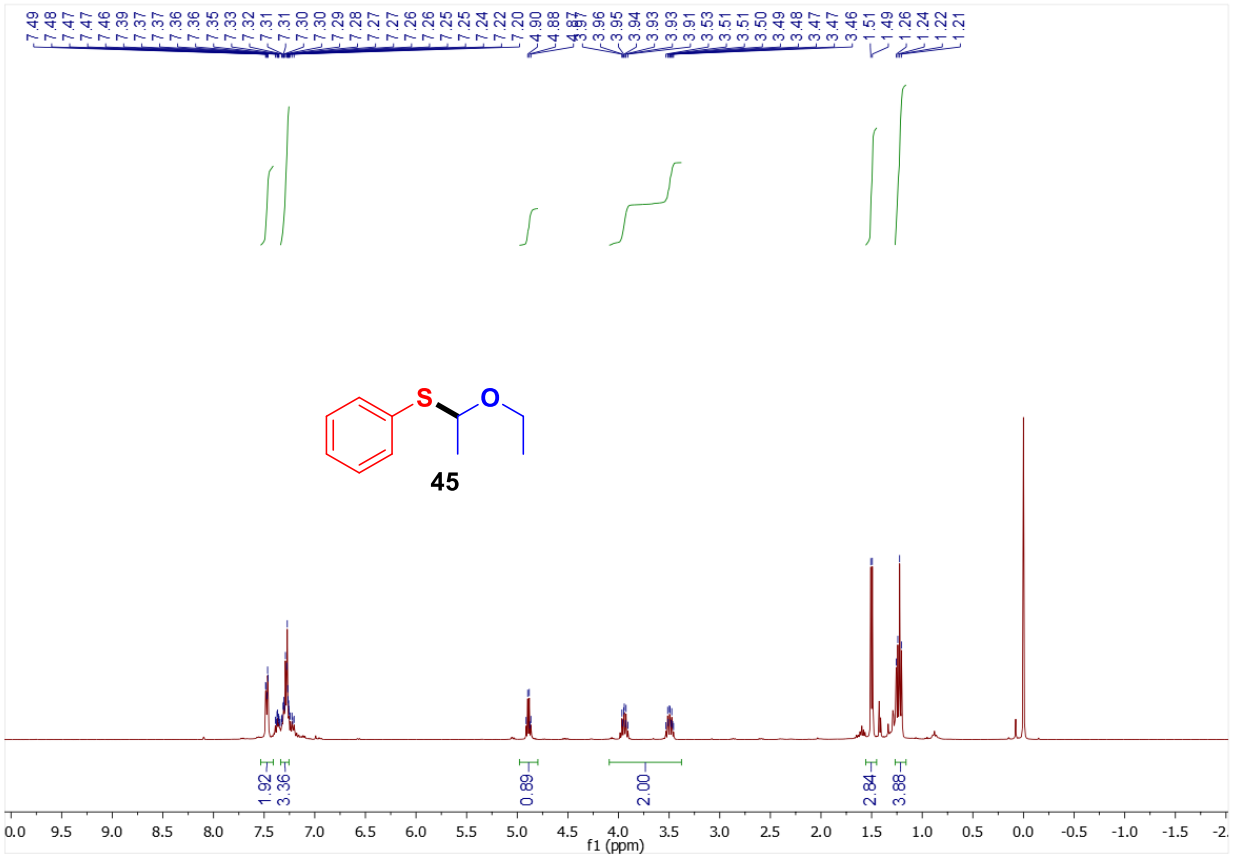
^{1}H-NMR (400 MHz, $CDCl_3$) of **44**



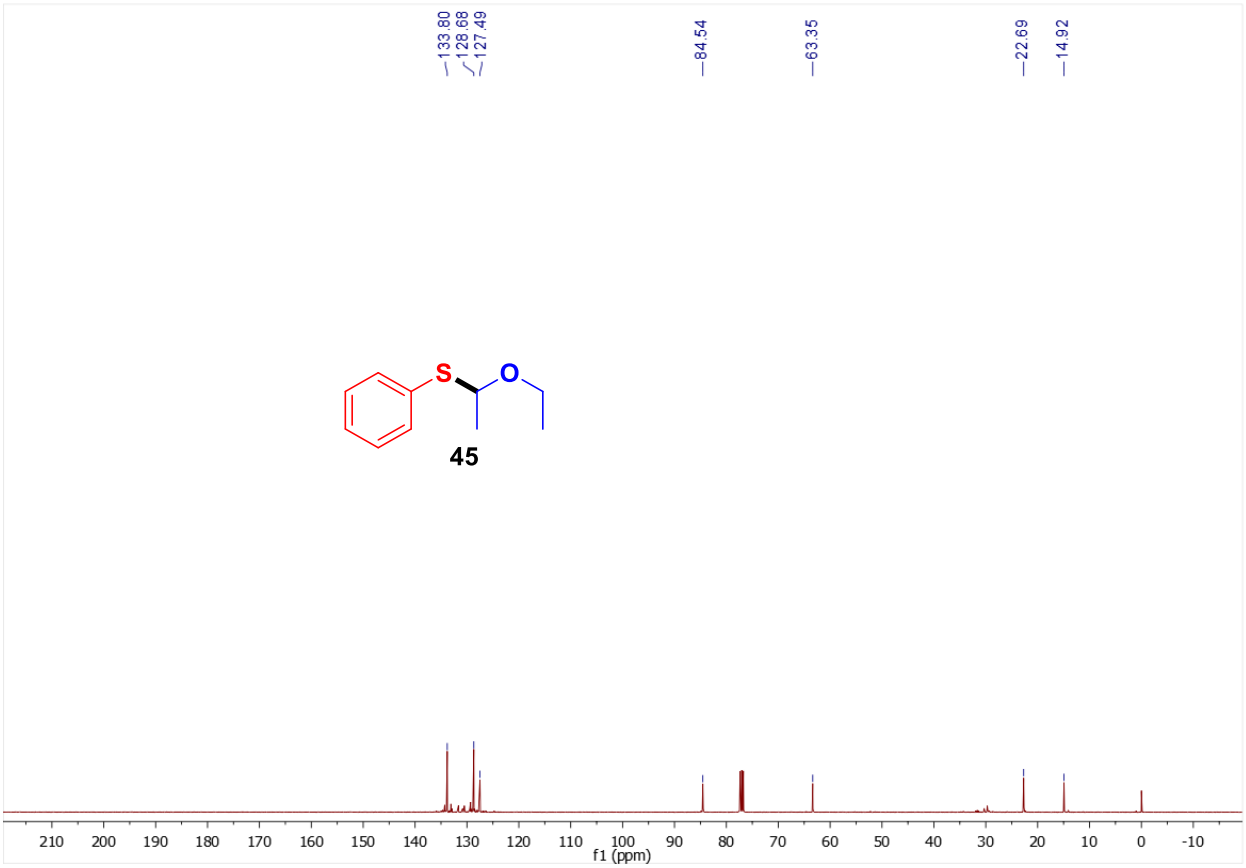
^{13}C-NMR (101 MHz, $CDCl_3$) of **44**



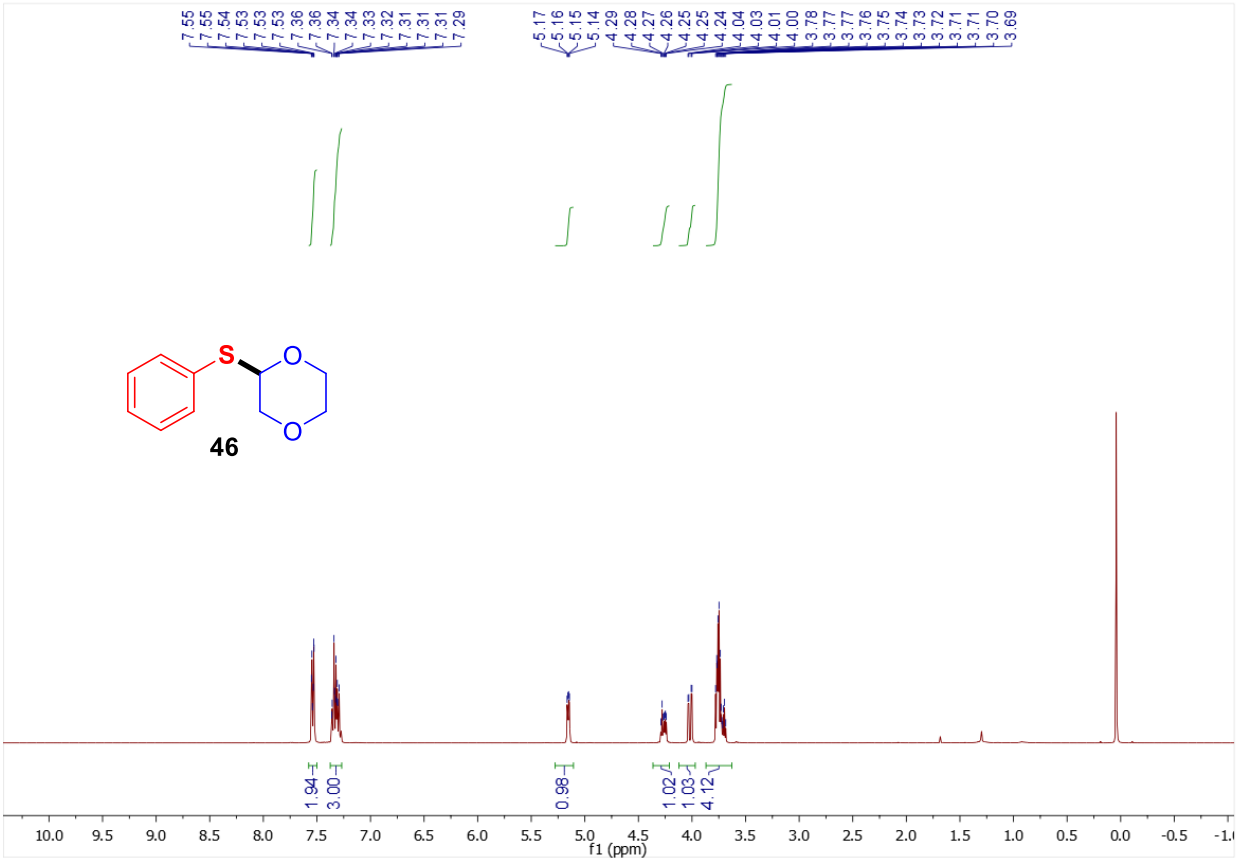
^{1}H-NMR (400 MHz, $CDCl_3$) of **45**



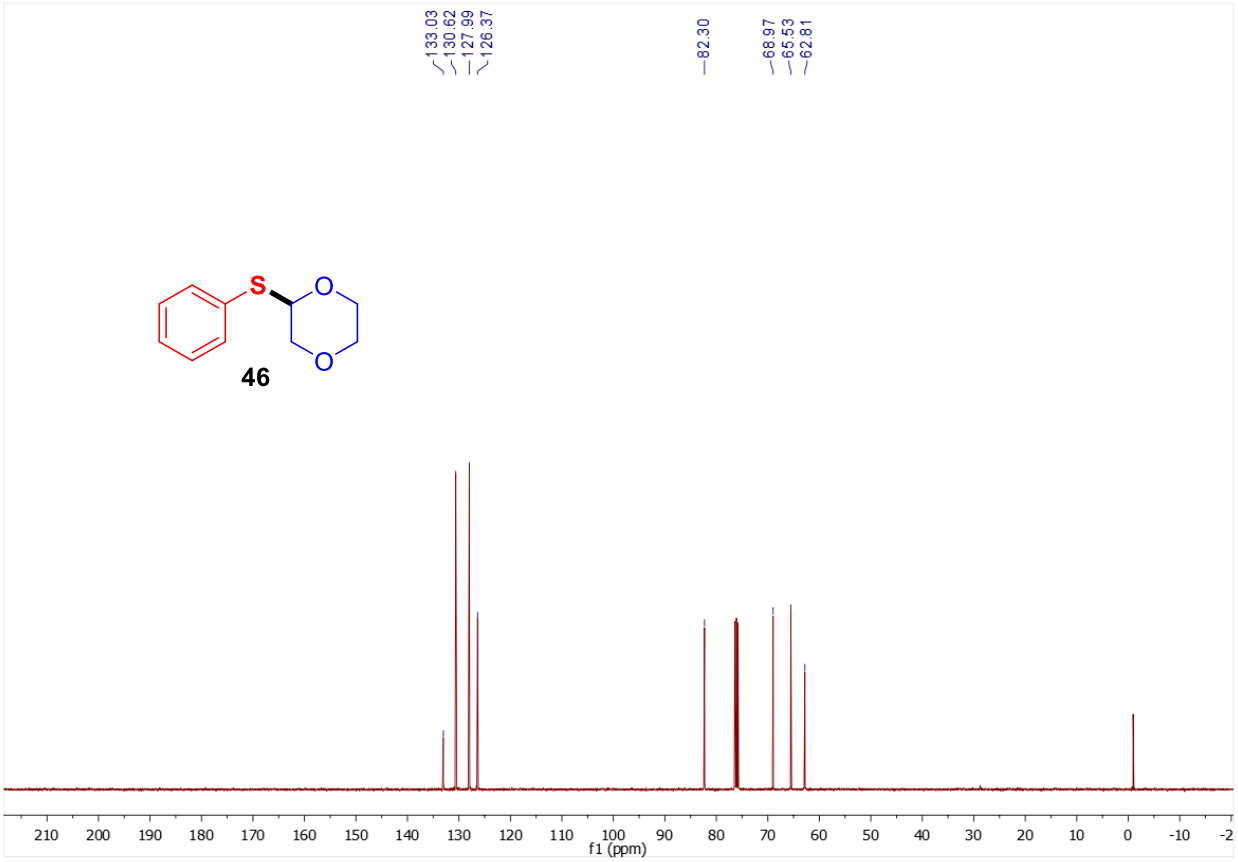
^{13}C-NMR (101 MHz, $CDCl_3$) of **45**



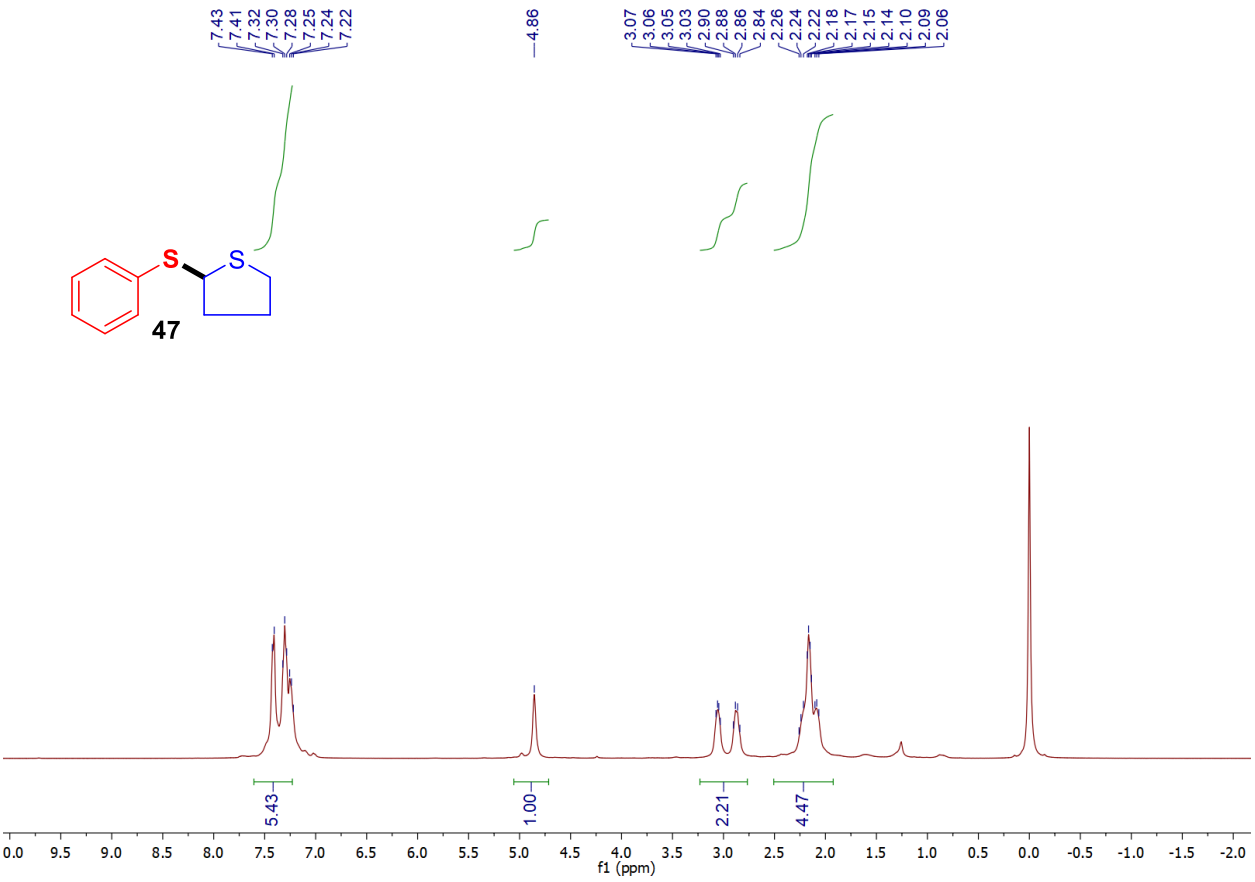
^{1}H-NMR (400 MHz, $CDCl_3$) of **46**



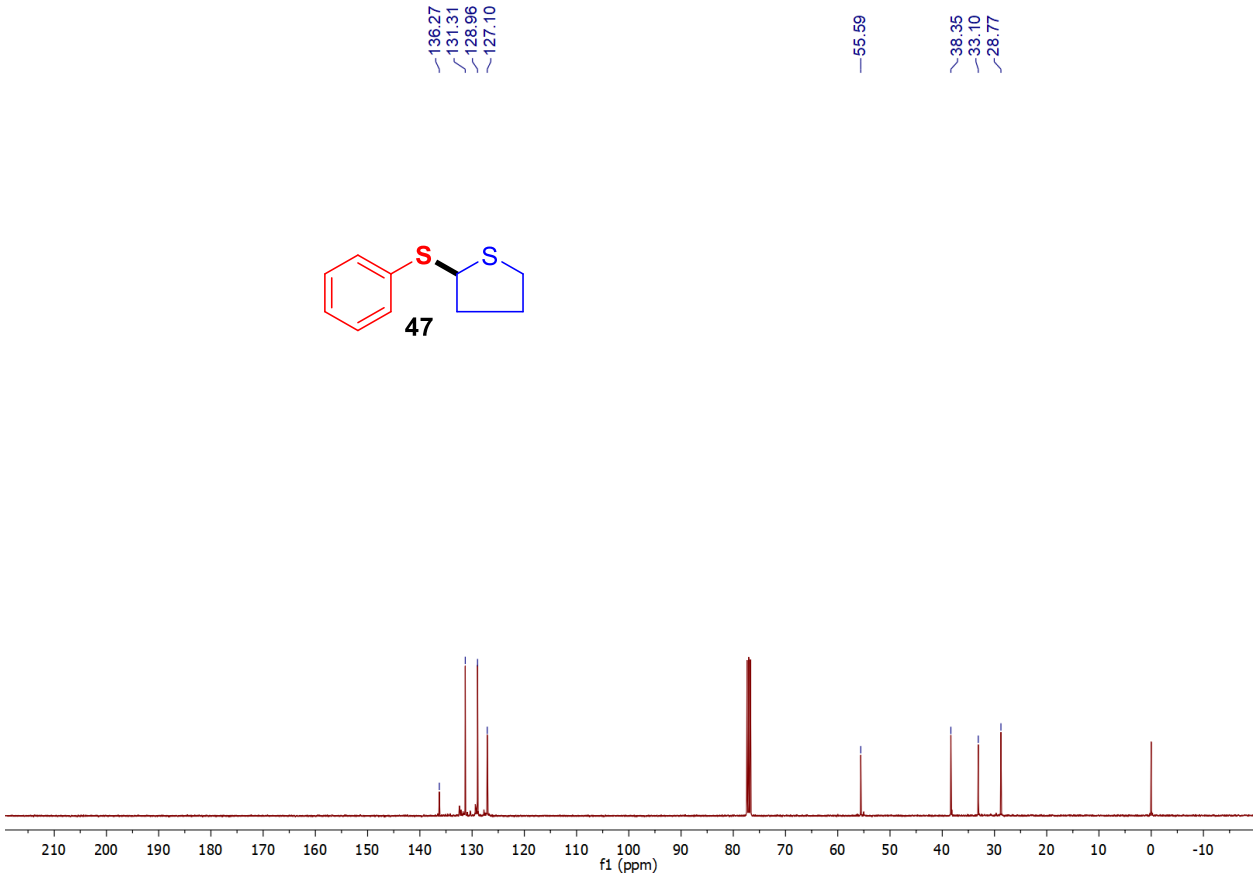
^{13}C-NMR (101 MHz, $CDCl_3$) of **46**



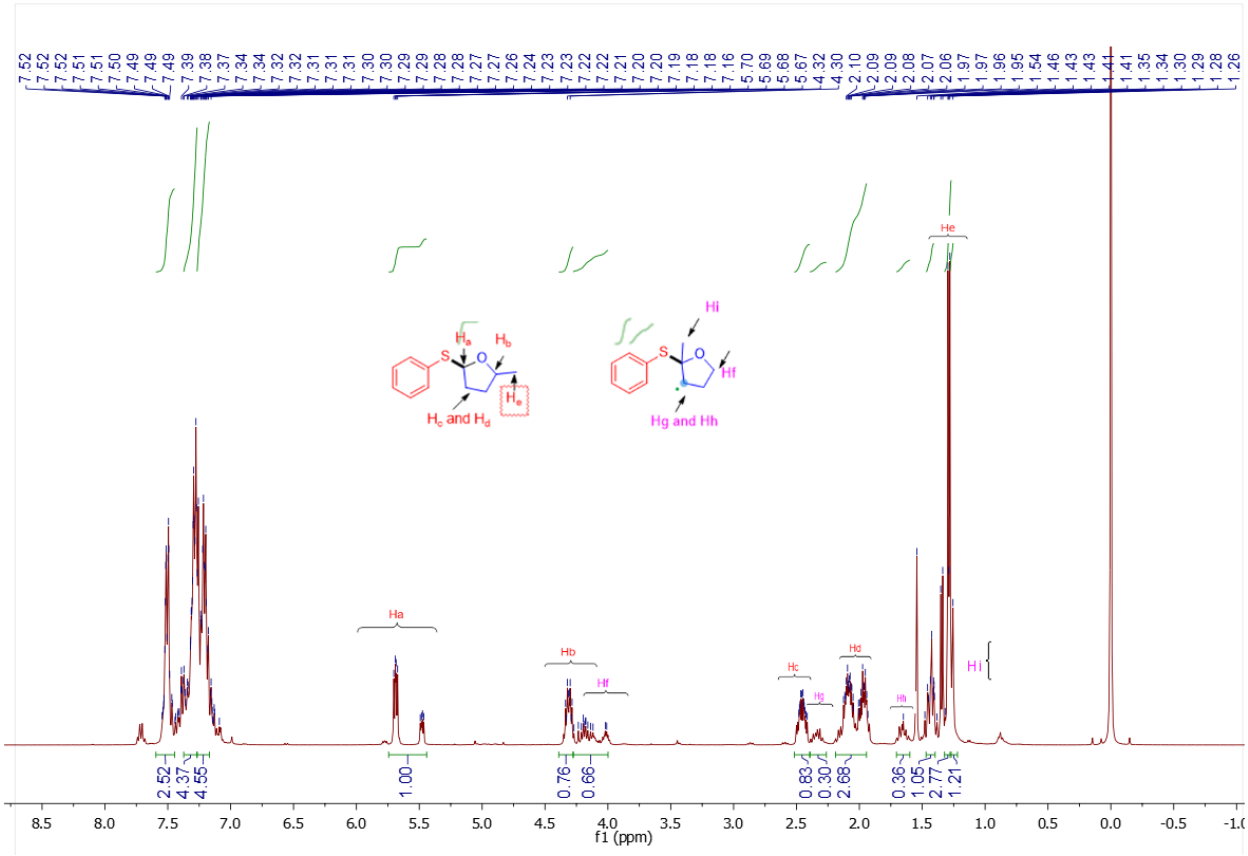
^{1}H-NMR (400 MHz, $CDCl_3$) of **47**



^{13}C-NMR (101 MHz, $CDCl_3$) of **47**



^{1}H-NMR (400 MHz, $CDCl_3$) of **48a and 48b**



^{13}C-NMR (101 MHz, $CDCl_3$) of **48a and 48b**