Supplementary Information (SI) for Green Chemistry.

Supporting Information for

Overcoming Protic-Solvent Limitations in Dithiane Chemistry: Photocatalyzed Giese Addition and One-Pot Ketone Synthesis

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I. General Experimental Information

All reactions were performed under an atmosphere of argon under conditions unless otherwise indicated. Methanol, dichloromethane, and acetone were purchased from Sigma-Aldrich, and buffer solution (pH 9, Cat. No: 042421.K2) was purchased from Thermo Scientific; all solvents were degassed by bubbling of nitrogen gas for 30 minutes prior to use. Pressure tubes (13 x 100 mm, PYREXPLUS, and 50 mL flask, purchased from Chem Glass) were oven-dried overnight and cooled under a stream of nitrogen before use. All commercial reagents were used as received without further purification.

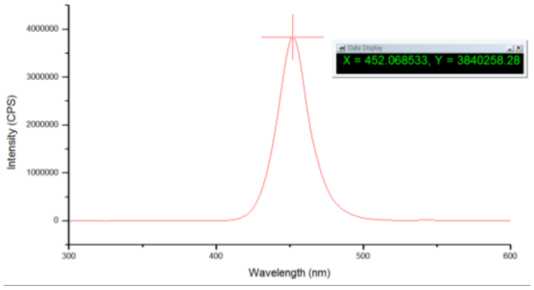
The progress of the reaction was checked on TLC plates (Merck 5554 Kiesel gel 60 F_{254}), and the spots were visualized under 254 nm UV light and/or charring after dipping the TLC plate into a *p*-anisaldehyde solution (5.6 mL of *p*-anisaldehyde, 2.3 mL acetic acid and 3.0 mL of concentrated sulfuric acid in 200 mL of ethanol). Column chromatography was performed on silica gel (Merck 9385 Kiesel gel 60) using Hexanes: EtOAc (v/v).

High-resolution mass spectra (HRMS) were obtained on a Jeol JMS HRMS (Electron Ionization, EI) at the Korea Basic Science Center (KBSI), Daegu, Korea and accurate masses are reported for the molecular ion $[M^+]$, $[M+H]^+$ or $[M+Na]^+$. Low-resolution mass spectra were acquired on an Agilent 7890A/5975C GC/MS (Electron Ionization, EI) at Pusan National University. Nuclear magnetic resonance spectra (^{1}H NMR, and ^{13}C NMR) were recorded with a Bruker 300 or 400 MHz spectrometer. Chemical shift values were recorded as parts per million relatives to tetramethylsilane as an internal standard unless otherwise indicated, and coupling constants in Hertz. Absorption spectra of the photocatalysts and emission spectra of the visible light sources were measured on a Varian Carry 100, Horiba Fluoromax-4P spectrophotometer, and Ocean Optics QE65000. Cyclic voltammograms was recorded on a Bio-Logic (SP-300 model).

II. Reaction Setup

Irradiation of photochemical reactions was carried out using two MR16 5W blue LED spotlight lamps milligram scale reaction. The pictures of two utilized spotlight lamps and their description are given below:

MR16 5W blue LEDs spotlight



Emission spectrum of 5W blue LEDs

	Specification
Power	5 W
Voltage	12 V
Wavelength	452 nm

Figure S1. Description for 3 W blue LED spotlight lamp

For milligram scale reactions, two MR16 10W blue LEDs spotlight lamps were mentioned 3 cm away from the reaction vial using a customized reactor that was made of acrylic plate.

Figure S2. Miligram scale reaction set up

For gram scale reaction, one 40W Kessil PR160L-440 nm blue LED lamps (100% intensity) is positioned 8 cm away from the reaction flask as shown in **Figure S3**.

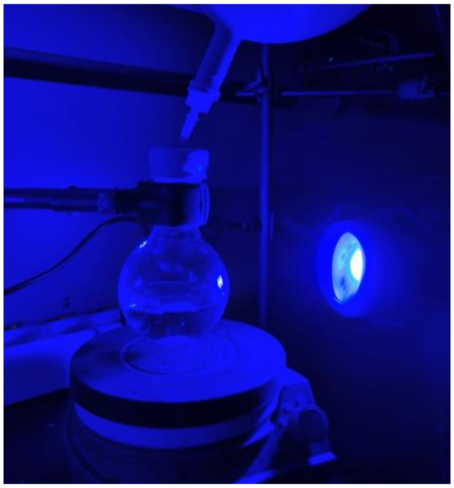
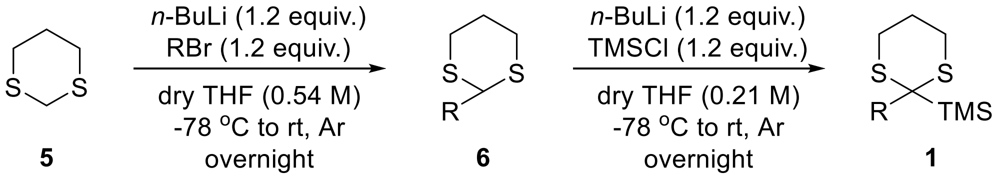


Figure S3. Gram scale reaction set up

In the optimized reaction conditions, the reaction is not significantly affected by temperature. The fan was used or not used according to the external temperature to maintain 20 ~ 30 ^{o}C in the reactor.

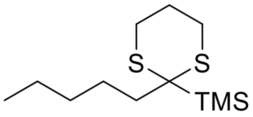
III. Preparation and characterization data of starting materials

Preparation of trimethylsilyl-1,3-dithianes derivatives



Trimetrylsilyl-1,3-dithianes **1a** were used directly from commercial sources reagents (Across). All the following trimethylsilyl-1,3-dithianes derivatives **1b-1f** were prepared by following reported protocol[1]. All Michael acceptors **2** were commercially available. Among these, synthesized trimethylsilyl-1,3-dithianes derivatives **1b-1d** compound data were matched to previously reported paper[2,3].

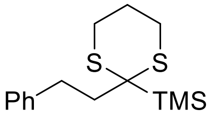
***Trimethyl(2-pentyl-1,3-dithian-2-yl)silane* (1b)[1]**



1b

^{1}H NMR (300 MHz, Chloroform-*d*) δ 3.10 – 2.97 (m, 2H), 2.50 – 2.39 (m, 2H), 2.23 – 2.14 (m, 2H), 2.12 – 1.79 (m, 2H), 1.54 – 1.41 (m, 2H), 1.42 – 1.24 (m, 4H), 0.96 – 0.86 (m, 3H), 0.18 (s, 9H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 38.95, 37.37, 32.56, 27.54, 25.31, 23.49, 22.74, 14.27, -2.36.

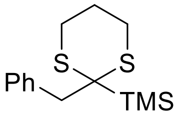
***Trimethyl(2-phenethyl-1,3-dithian-2-yl)silane* (1c)[1]**



1c

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.37 – 7.28 (m, 2H), 7.26 – 7.17 (m, 3H), 3.13 – 2.98 (m, 2H), 2.88 – 2.73 (m, 2H), 2.55 – 2.41 (m, 4H), 2.14 – 1.98 (m, 1H), 1.92 (dtt, *J* = 13.7, 12.4, 3.3 Hz, 1H), 0.27 (s, 9H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 142.50, 128.64, 128.52, 126.06, 39.75, 38.77, 34.48, 25.20, 23.46, -2.31.

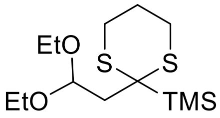
(2-benzyl-1,3-dithian-2-yl)trimethylsilane **(1d)**[2]



1d

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.58 – 7.49 (m, 2H), 7.37 – 7.29 (m, 3H), 3.46 (s, 2H), 2.80 – 2.67 (m, 2H), 2.48 – 2.35 (m, 2H), 1.96 (dtt, *J* = 13.6, 4.4, 2.9 Hz, 1H), 1.89 – 1.75 (m, 1H), 0.11 (s, 9H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 138.99, 131.26, 127.99, 126.86, 44.81, 38.00, 24.40, 24.22, -3.10.

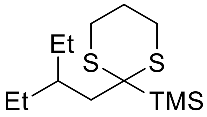
(2-(2,2-diethoxyethyl)-1,3-dithian-2-yl)trimethylsilane **(1e)**



1e

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.69 (t, *J* = 4.3 Hz, 1H), 3.72 – 3.62 (m, 2H), 3.55 – 3.46 (m, 2H), 3.13 – 3.03 (m, 2H), 2.55 (d, *J* = 4.3 Hz, 2H), 2.43 (dt, *J* = 14.2, 3.8 Hz, 2H), 2.09 – 2.00 (m, 1H), 1.88 (qt, *J* = 13.5, 3.2 Hz, 1H), 1.21 (t, *J* = 7.1 Hz, 6H), 0.20 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 102.05, 61.09, 41.48, 36.28, 25.05, 23.52, 15.43, -2.67; HRMS (ESI$^+$) (m/z) calcd. for $C_{13}H_{28}O_2S_2Si$ (M+Na)$^+$ 331.1192, found 331.1186.

(2-(2-ethylbutyl)-1,3-dithian-2-yl)trimethylsilane **(1f)**



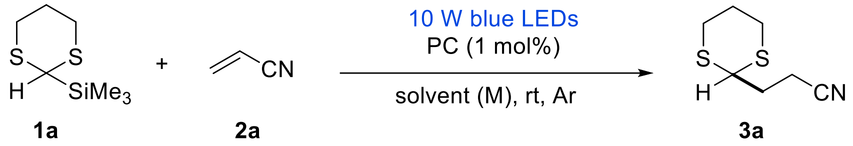
1f

^{1}H NMR (300 MHz, Chloroform-*d*) δ 3.07 – 2.92 (m, 2H), 2.56 – 2.44 (m, 2H), 2.09 (d, *J* = 4.5 Hz, 2H), 2.05 – 1.97 (m, 1H), 1.97 – 1.82 (m, 1H), 1.62 – 1.47 (m, 3H), 1.39 (dt, *J* = 13.8, 6.7 Hz, 2H), 0.86 (t, *J* = 7.3 Hz, 6H), 0.22 (s, 9H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 41.04, 39.53, 38.16, 26.06, 25.15, 24.22, 10.43, -2.02; HRMS (EI) (m/z) calcd. For $C_{13}H_{28}S_2Si$ (M$^+$) 276.1402, found 276.1402.

IV. Reaction optimizations

Reaction optimization was conducted according to the following procedures: To a re-sealable pressure tube (13 x 100 mm) equipped with a small magnetic stir bar were added silane **1a** (0.2 mmol, 1.0 equiv), alkene **2a** (0.4 mmol, 2.0 equiv), and photocatalyst (0.002 mmol, 1 mol%) under argon atmosphere. The reaction mixture was dissolved in degassed solvents. The mixture was irradiated with 2 x 5W blue LEDs using a customized milligram scale reaction set up (as shown in **Figure S2**) under constant stirring condition at room temperature. After completion of the reaction, the solvent was removed under reduced pressure. Yields were assessed by quantitative ^{1}H NMR spectroscopy using mesitylene as an internal standard or purified by silica gel column chromatography using ethyl acetate/hexanes as the eluent to afford the corresponding product **3a**.

Table S1. Optimization table

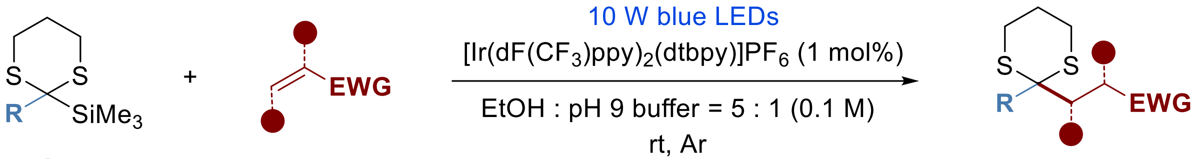


entry	Photocatalyst (1 mol%)	solvent (M)	time (h)	yield (%)[a]
1	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeCN (1.0)	14	16
2	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	DMF (1.0)	14	30
3	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeOH (1.0)	14	60
4	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeOH (0.1)	14	70
5	4CzIPN	MeOH (0.1)	12	26[f]
6	Cl-4CzIPN	MeOH (0.1)	12	39[f]
7	Ru(bpz)$_3$PF$_6$	MeOH (0.1)	12	<5
8	Cl-4CzIPN	MeCN (0.1)	12	22[f]
9	Cl-4CzIPN	DMF (0.1)	12	<5
10	Cl-4CzIPN	EtOH (0.1)	12	26[f]
11	Cl-4CzIPN	EtOAc (0.1)	12	9[f]
12	Cl-4CzIPN	DCM (0.1)	12	10[f]
13	Cl-4CzIPN	Acetone (0.1)	12	7[f]
14	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeCN:MeOH = 3:1 (0.1)	14	76
15	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeCN:MeOH = 3:1 (0.1)	16	83[f]
16	Cl-4CzIPN	MeCN:MeOH = 3:1 (0.1)	12	23[f]
17	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	MeOH (0.1)[e]	14	78
18	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH (0.1)[e]	14	76
19	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	14	99
20	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	6	79
21	**[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$**	**EtOH:pH 9 Buffer = 5:1 (0.1)**	**12**	**96**
22	4CzIPN	EtOH:pH 9 Buffer = 5:1 (0.1)	12	<5
23	Cl-4CzIPN	EtOH:pH 9 Buffer = 5:1 (0.1)	12	<5
24	Ru(bpz)$_3$PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	12	<5
25[b]	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	12	nd
26[c]	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	12	nd
27[d]	[Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$	EtOH:pH 9 Buffer = 5:1 (0.1)	12	49[f]

[a]Isolated yield. [b]In the absence of a photocatalyst. [c]In the absence of light source. [d]In air atmosphere. [e]NaHCO$_3$ (1 equiv). [f]^{1}H NMR yield (IS = mesitylene). nd = not detected.

V. General procedure and characterization data of dithiane 3

A. General procedure for dithianes 3

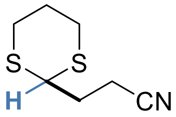


1 **2** **3**

To a re-sealable pressure tube (13 x 100 mm) equipped with a small magnetic stir bar were added silane **1** (0.2 mmol, 1.0 equiv), alkene **2** (0.4 mmol, 2.0 equiv), and $[Ir(dF(CF_3)ppy)_2(dtbpy)]PF_6$ (0.002 mmol, 1 mol%) under argon atmosphere. The reaction mixture was dissolved in degassed solvents (2 mL, 0.1 M) of EtOH/pH 9 buffer in a ratio of 5:1 or MeCN/MeOH in a ratio of 3:1. The mixture was irradiated with 2 x 5W blue LEDs using a customized milligram scale reaction set up (as shown in **Figure S2**) under constant stirring condition at room temperature. After completion of the reaction, the solvent was removed under reduced pressure, and residue was purified by silica gel column chromatography using ethyl acetate/hexanes as the eluent to afford the corresponding product **3**.

B. Characterization data of dithianes 3

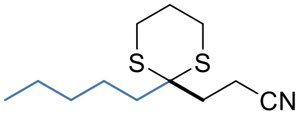
3-(1,3-dithian-2-yl)propanenitrile **(3a)**[3]



3a

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3a** was obtained as a colorless liquid (33.3 mg, 96% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.10 (t, $J = 7.2$ Hz, 1H), 2.90 – 2.83 (m, 4H), 2.59 (t, $J = 7.3$ Hz, 2H), 2.18 – 2.06 (m, 3H), 1.99 – 1.79 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 118.7, 45.0, 30.9, 29.7, 25.6, 14.8.

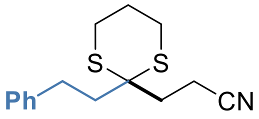
3-(2-pentyl-1,3-dithian-2-yl)propanenitrile **(3b)**



3b

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3b** was obtained as a white solid (43.8 mg, 90% yield); R_f = 0.20 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 2.85 (ddd, *J* = 14.6, 9.7, 3.3 Hz, 2H), 2.73 (ddd, *J* = 14.5, 6.4, 3.5 Hz, 2H), 2.57 – 2.42 (m, 2H), 2.35 – 2.27 (m, 2H), 2.08 – 1.80 (m, 2H), 1.80 – 1.67 (m, 2H), 1.52 – 1.38 (m, 2H), 1.36 – 1.23 (m, 4H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 119.7, 52.0, 38.9, 33.7, 31.9, 26.1, 25.0, 23.4, 22.5, 14.1, 13.3; HRMS (ESI$^+$) (m/z) calcd. For $C_{12}H_{21}NS_2$ (M+H)$^+$ 244.1188, found 244.1181.

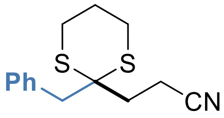
***3-(2-phenethyl-1,3-dithian-2-yl)propanenitrile* (3c)**



3c

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h) using 7% ethyl acetate in hexanes as eluent, **3c** was obtained as a white solid (38.3 mg, 69% yield); R_f = 0.20 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.35 – 7.27 (m, 2H), 7.21 (td, *J* = 6.3, 1.6 Hz, 3H), 2.95 – 2.75 (m, 6H), 2.62 – 2.53 (m, 2H), 2.41 – 2.33 (m, 2H), 2.13 – 2.05 (m, 2H), 2.05 – 1.87 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 141.2, 128.7, 128.5, 126.4, 119.7, 51.9, 41.1, 34.1, 30.6, 26.1, 24.8, 13.3; HRMS (EI) (m/z) calcd. for $C_{15}H_{19}NS_2$ (M$^+$) 277.0959, found 277.0961.

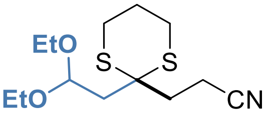
***3-(2-benzyl-1,3-dithian-2-yl)propanenitrile* (3d)**



3d

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h) using 7% ethyl acetate in hexanes as eluent, **3d** was obtained as a light yellow solid (18.3 mg, 35% yield); R_f = 0.20 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.37 – 7.25 (m, 5H), 7.28 – 7.23 (m, 2H), 3.16 (s, 2H), 2.92 – 2.76 (m, 4H), 2.64 – 2.54 (m, 2H), 2.24 – 2.13 (m, 2H), 2.04 – 1.87 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 134.8, 130.9, 128.3, 127.6, 119.8, 52.4, 45.7, 33.6, 26.4, 24.5, 13.6; HRMS (EI) (m/z) calcd. for $C_{14}H_{17}NS_2$ (M$^+$) 263.0802, found 263.0800.

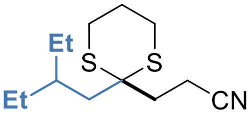
3-(2-(2,2-diethoxyethyl)-1,3-dithian-2-yl)propanenitrile (3e)



3e

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3e** was obtained as a light yellow liquid (40.1 mg, 69% yield); $R_f = 0.40$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.74 (t, *J* = 4.8 Hz, 1H), 3.71 – 3.59 (m, 2H), 3.57 – 3.44 (m, 2H), 2.90 – 2.81 (m, 2H), 2.77 (ddd, *J* = 14.5, 6.9, 3.7 Hz, 2H), 2.63 – 2.55 (m, 2H), 2.39 – 2.30 (m, 2H), 2.11 (d, *J* = 4.9 Hz, 2H), 2.05 – 1.86 (m, 2H), 1.21 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 119.88, 99.97, 62.16, 49.67, 42.37, 34.18, 26.05, 24.89, 15.36, 13.16; HRMS (ESI$^+$) (m/z) calcd. for $C_{13}H_{23}NO_2S_2$ (M+Na)$^+$ 312.1062, found 312.1056.

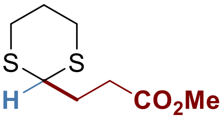
3-(2-(2-ethylbutyl)-1,3-dithian-2-yl)propanenitrile (3f)



3f

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3f** was obtained as a colorless liquid (44.3 mg, 86% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 2.87 (ddd, *J* = 13.5, 10.3, 3.0 Hz, 2H), 2.70 (ddd, *J* = 14.5, 6.1, 3.4 Hz, 2H), 2.57 – 2.48 (m, 2H), 2.38 – 2.28 (m, 2H), 2.05 – 1.93 (m, 1H), 1.92 – 1.77 (m, 1H), 1.66 – 1.53 (m, 3H), 1.47 – 1.27 (m, 4H), 0.84 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 77.58, 52.93, 43.05, 36.18, 34.52, 27.12, 26.32, 24.72, 13.64, 10.68; HRMS (EI) (m/z) calcd. for $C_{13}H_{23}NS_2$ (M$^+$) 257.1272, found 257.1273.

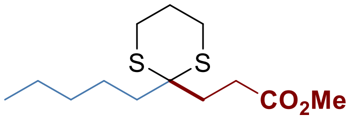
Methyl 3-(1,3-dithian-2-yl)propanoate (3g)[3]



3g

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3g** was obtained as a colorless liquid (40.8 mg, 99% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.04 (t, *J* = 7.1 Hz, 1H), 3.67 (s, 3H), 2.87 – 2.79 (m, 4H), 2.54 (t, *J* = 7.5 Hz, 2H), 2.15 – 2.03 (m, 3H), 1.97 – 1.76 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 173.2, 51.9, 46.3, 31.1, 30.4, 30.0, 25.9.

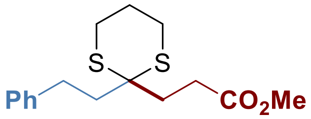
Methyl 3-(2-pentyl-1,3-dithian-2-yl)propanoate (3h)



3h

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3h** was obtained as a colorless liquid (46.4 mg, 84% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 3.68 (s, 3H), 2.92 (ddd, *J* = 14.6, 9.9, 3.2 Hz, 2H), 2.71 (ddd, *J* = 14.5, 6.5, 3.3 Hz, 2H), 2.52 – 2.41 (m, 2H), 2.35 – 2.23 (m, 2H), 2.09 – 1.80 (m, 2H), 1.80 – 1.69 (m, 2H), 1.53 – 1.38 (m, 2H), 1.36 – 1.24 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 173.98, 52.63, 51.90, 39.04, 32.71, 32.12, 29.91, 26.14, 25.39, 23.47, 22.62, 14.18; HRMS (EI) (m/z) calcd. for $C_{13}H_{24}O_2S_2$ (M^+) 276.1218, found 276.1221.

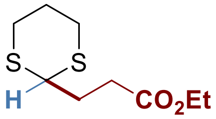
Methyl 3-(2-phenethyl-1,3-dithian-2-yl)propanoate (3i)



3i

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3i** was obtained as a colorless liquid (40.6 mg, 65% yield); $R_f = 0.50$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.22 (dd, *J* = 6.6, 5.4 Hz, 2H), 7.17 – 7.10 (m, 3H), 3.63 (s, 3H), 2.85 (ddd, *J* = 12.4, 9.0, 3.2 Hz, 2H), 2.77 – 2.67 (m, 4H), 2.48 (dd, *J* = 9.3, 6.5 Hz, 2H), 2.30 (dd, *J* = 9.6, 6.4 Hz, 2H), 2.52 – 2.43 (m, 2H), 2.34 – 2.25 (m, 2H), 2.07 – 1.79 (m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.83, 141.66, 128.62, 128.55, 126.14, 52.48, 51.92, 41.10, 33.13, 30.59, 29.76, 26.11, 25.21; HRMS (ESI$^+$) (m/z) calcd. for $C_{16}H_{22}O_2S_2$ $(M+H)^+$ 311.1134, found 311.1126.

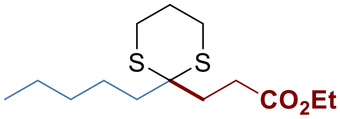
Ethyl 3-(1,3-dithian-2-yl)propanoate (3j)[4]



3j

Following the general procedure (24 h) using 5% ethyl acetate in hexanes as eluent, **3j** was obtained as a colorless liquid (31.5 mg, 72% yield); $R_f = 0.50$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.13 (q, *J* = 7.1 Hz, 2H), 4.05 (t, *J* = 7.1 Hz, 1H), 2.87 – 2.81 (m, 4H), 2.53 (t, *J* = 7.5 Hz, 2H), 2.15 – 2.05 (m, 3H), 1.92 – 1.80 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.78, 60.67, 46.41, 31.38, 30.47, 30.02, 25.94, 14.33.

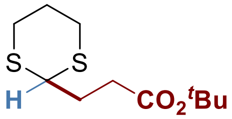
Ethyl 3-(2-pentyl-1,3-dithian-2-yl)propanoate **(3k)**



3k

Following the general procedure (24 h) using 6% ethyl acetate in hexanes as eluent, **3k** was obtained as a colorless liquid (39.7 mg, 68% yield); R_f = 0.60 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.11 (q, *J* = 7.1 Hz, 2H), 2.90 (ddd, *J* = 13.2, 10.0, 2.9 Hz, 2H), 2.69 (ddd, *J* = 14.5, 6.5, 3.2 Hz, 2H), 2.47 – 2.38 (m, 2H), 2.32 – 2.23 (m, 2H), 2.03 – 1.94 (m, 1H), 1.92 – 1.80 (m, 1H), 1.77 – 1.69 (m, 2H), 1.49 – 1.37 (m, 2H), 1.36 – 1.20 (m, 7H), 0.87 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.46, 60.61, 52.63, 38.99, 32.65, 32.07, 30.07, 26.09, 25.36, 23.42, 22.56, 14.30, 14.12; HRMS (ESI$^+$) (m/z) calcd. for $C_{14}H_{26}O_2S_2$ (M+Na)$^+$ 313.1266, found 313.1261.

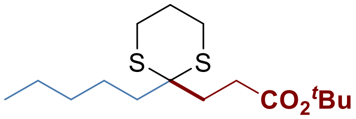
Tert-butyl 3-(1,3-dithian-2-yl)propanoate **(3l)**[5]



3l

Following the general procedure (24 h) using 5% ethyl acetate in hexanes as eluent, **3l** was obtained as a colorless liquid (48.6 mg, 98% yield); R_f = 0.60 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.04 (t, *J* = 7.0 Hz, 1H), 2.89 – 2.78 (m, 4H), 2.44 (t, *J* = 7.5 Hz, 2H), 2.14 – 2.03 (m, 3H), 1.93 – 1.81 (m, 1H), 1.44 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.09, 80.65, 46.56, 32.64, 30.66, 30.04, 28.22, 25.99.

Tert-butyl 3-(2-pentyl-1,3-dithian-2-yl)propanoate **(3m)**

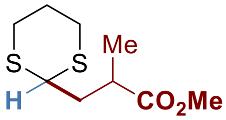


3m

Following the general procedure (24 h) using 6% ethyl acetate in hexanes as eluent, **3m** was obtained as a colorless liquid (20 mg, 31% yield); R_f = 0.60 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.93 (ddd, *J* = 13.4, 10.1, 2.9 Hz, 2H), 2.70 (ddd, *J* = 14.5, 6.4, 3.2 Hz, 2H), 2.40 – 2.30 (m, 2H), 2.30 – 2.21 (m, 2H), 2.06 – 1.95 (m, 1H), 1.93 – 1.81 (m, 1H), 1.78

– 1.69 (m, 2H), 1.45 (s, 9H), 1.34 – 1.23 (m, 4H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.86, 80.53, 52.80, 39.09, 32.73, 32.14, 31.23, 28.23, 26.15, 25.44, 23.48, 22.62, 14.17; HRMS (ESI$^+$) (m/z) calcd. for $C_{16}H_{30}O_2S_2$ (M+Na)$^+$ 341.1579, found 341.1580.

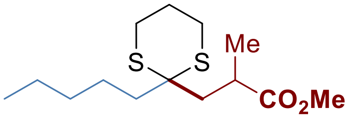
***Methyl 3-(1,3-dithian-2-yl)-2-methylpropanoate* (3n)**[6]



3n

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3n** was obtained as a colorless liquid (43.6 mg, 99% yield); R_f = 0.20 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.08 – 3.97 (t, 1H), 3.68 (s, 3H), 2.88 – 2.72 (m, 5H), 2.29 – 2.02 (m, 2H), 1.94 – 1.71 (m, 2H), 1.19 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 176.3, 52.0, 45.0, 38.8, 36.7, 30.1, 26.0, 17.5.

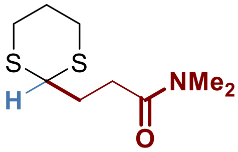
***Methyl 2-methyl-3-(2-pentyl-1,3-dithian-2-yl)propanoate* (3o)**



3o

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3o** was obtained as a colorless liquid (45.4 mg, 78% yield); R_f = 0.50 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.64 (s, 3H), 2.92 (dddd, *J* = 14.0, 10.7, 9.2, 2.8 Hz, 2H), 2.81 (dd, *J* = 14.8, 9.2 Hz, 1H), 2.70 (ddd, *J* = 13.9, 5.8, 2.9 Hz, 1H), 2.64 (ddd, *J* = 9.2, 7.1, 2.1 Hz, 1H), 2.61 – 2.55 (m, 1H), 2.03 – 1.94 (m, 1H), 1.88 – 1.74 (m, 4H), 1.57 – 1.43 (m, 1H), 1.40 – 1.22 (m, 6H), 1.19 (d, *J* = 7.1 Hz, 3H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.46, 52.61, 51.90, 41.66, 39.30, 36.29, 32.08, 26.59, 26.11, 25.15, 23.46, 22.56, 19.89, 14.12; HRMS (ESI$^+$) (m/z) calcd. for $C_{14}H_{26}O_2S_2$ (M+H)$^+$ 291.1447, found 291.1436.

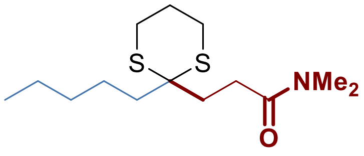
***3-(1,3-dithian-2-yl)-N,N-dimethylpropanamide* (3p)**



3p

Following the general procedure (24 h) using 10% ethyl acetate in hexanes as eluent, **3p** was obtained as a white solid (33.8 mg, 77% yield); R_f = 0.10 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.11 (t, *J* = 6.9 Hz, 1H), 2.99 (s, 3H), 2.93 (s, 3H), 2.87 – 2.80 (m, 4H), 2.51 (t, *J* = 7.4 Hz, 2H), 2.16 – 2.03 (m, 3H), 1.93 – 1.76 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 171.77, 46.78, 37.24, 35.54, 30.64, 30.09, 29.89, 26.00; HRMS (ESI$^+$) (m/z) calcd. for $C_9H_{17}NOS_2$ (M+H)$^+$ 220.0824, found 220.0817.

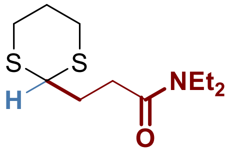
N,N-dimethyl-3-(2-pentyl-1,3-dithian-2-yl)propenamide **(3q)**



3q

Following the general procedure (24 h) using 6% ethyl acetate in hexanes as eluent, **3q** was obtained as a colorless liquid (27.7 mg, 48% yield); R_f = 0.60 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.02 (s, 3H), 2.96 – 2.85 (m, 5H), 2.72 (ddd, *J* = 14.3, 6.8, 3.1 Hz, 2H), 2.49 – 2.40 (m, 2H), 2.33 – 2.24 (m, 2H), 2.02 – 1.84 (m, 2H), 1.82 – 1.73 (m, 2H), 1.51 – 1.39 (m, 2H), 1.38 – 1.19 (m, 4H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.49, 53.07, 39.25, 37.35, 33.03, 32.12, 26.06, 25.38, 23.58, 22.57, 14.13; HRMS (ESI$^+$) (m/z) calcd. for $C_{14}H_{27}NOS_2$ (M+Na)$^+$ 312.1426, found 312.1413.

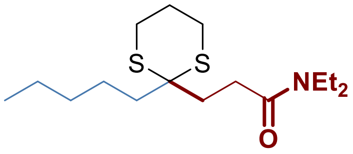
3-(1,3-dithian-2-yl)-N,N-diethylpropanamide **(3r)**



3r

Following the general procedure (24 h) using 10% ethyl acetate in hexanes as eluent, **3r** was obtained as a light yellow liquid (14.8 mg, 30% yield); R_f = 0.10 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.12 (t, *J* = 6.9 Hz, 1H), 3.36 (q, *J* = 7.2 Hz, 2H), 3.30 (q, *J* = 7.2 Hz, 2H), 2.86 – 2.80 (m, 4H), 2.52 (t, *J* = 7.4 Hz, 2H), 2.19 – 2.04 (m, 3H), 1.92 – 1.79 (m, 1H), 1.17 (t, *J* = 7.1 Hz, 3H), 1.09 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.06, 46.84, 42.11, 40.44, 30.94, 30.09, 29.67, 26.04, 14.38, 13.20; HRMS (ESI$^+$) (m/z) calcd. for $C_{11}H_{21}NOS_2$ (M+H)$^+$ 248.1137, found 248.1135.

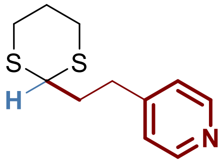
N,N-diethyl-3-(2-pentyl-1,3-dithian-2-yl)propenamide (3s)



3s

Following the general procedure (24 h) using 8% ethyl acetate in hexanes as eluent, **3s** was obtained as a white solid (36.9 mg, 58%); $R_f = 0.40$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.33 (dt, *J* = 16.7, 7.1 Hz, 4H), 2.91 (ddd, *J* = 12.6, 9.4, 3.1 Hz, 2H), 2.73 (ddd, *J* = 14.5, 6.9, 3.3 Hz, 2H), 2.48 – 2.39 (m, 2H), 2.35 – 2.26 (m, 2H), 2.03 – 1.83 (m, 2H), 1.82 – 1.74 (m, 2H), 1.51 – 1.41 (m, 2H), 1.36 – 1.22 (m, 5H), 1.18 (t, *J* = 7.1 Hz, 3H), 1.10 (t, *J* = 7.1 Hz, 3H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.62, 53.17, 42.13, 40.40, 39.27, 33.30, 32.14, 28.42, 26.08, 25.40, 23.60, 22.58, 14.50, 14.14, 13.19; HRMS (ESI$^+$) (m/z) calcd. for $C_{16}H_{31}NOS_2$ (M+Na)$^+$ 340.1739, found 340.1725.

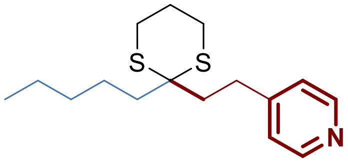
4-(2-(1,3-dithian-2-yl)ethyl)pyridine (3t)



3t

Following the general procedure (12 h) using 20% ethyl acetate in hexanes as eluent, **3t** was obtained as a yellow liquid (39.6 mg, 88% yield); $R_f = 0.10$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.58 – 8.39 (m, 2H), 7.17 – 7.07 (m, 2H), 3.94 (t, *J* = 7.0 Hz, 1H), 2.87 – 2.74 (m, 6H), 2.18 – 1.98 (m, 3H), 1.95 – 1.75 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 149.9, 149.9, 124.0, 46.4, 35.7, 31.9, 30.3, 26.0; HRMS (EI) (m/z) calcd. for $C_{11}H_{15}NS_2$ (M$^+$) 225.0646, found 225.0646.

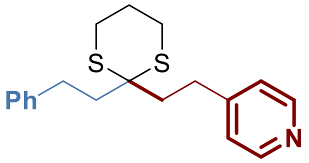
4-(2-(2-pentyl-1,3-dithian-2-yl)ethyl)pyridine (3u)



3u

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h) using 7% ethyl acetate in hexanes as eluent, **3u** was obtained as a colorless liquid (55 mg, 93% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.56 – 8.43 (m, 2H), 7.21 – 7.08 (m, 2H), 2.91 – 2.79 (m, 4H), 2.79 – 2.71 (m, 2H), 2.23 – 2.12 (m, 2H), 2.07 – 1.82 (m, 4H), 1.55 – 1.41 (m, 2H), 1.41 – 1.25 (m, 4H), 0.96 – 0.84 (t, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 151.4, 149.6, 124.1, 53.0, 39.0, 38.7, 32.1, 30.5, 26.1, 25.5, 23.8, 22.7, 14.2; HRMS (EI) (m/z) calcd. for $C_{16}H_{25}NS_2$ (M^+) 295.1428, found 295.1429.

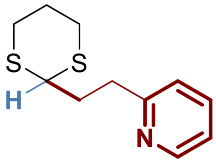
***4-(2-(2-phenethyl-1,3-dithian-2-yl)ethyl)pyridine* (3v)**



3v

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3v** was obtained as a yellow liquid (49.4 mg, 75% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.50 (d, *J* = 5.3 Hz, 2H), 7.35 – 7.27 (m, 2H), 7.24 – 7.17 (m, 3H), 7.16 – 7.10 (m, 2H), 2.90 – 2.75 (m, 8H), 2.30 – 2.16 (m, 4H), 2.05 – 1.92 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 151.0, 149.7, 141.6, 128.6, 128.5, 126.2, 124.0, 52.9, 40.6, 39.4, 30.9, 30.4, 26.1, 25.2; HRMS (EI) (m/z) calcd. for $C_{19}H_{23}NS_2$ (M^+) 329.1272, found 329.1273.

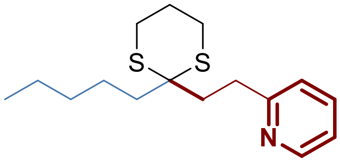
***2-(2-(1,3-dithian-2-yl)ethyl)pyridine* (3w)**



3w

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h) using 10% ethyl acetate in hexanes as eluent, **3w** was obtained as a light yellow liquid (24.8 mg, 55% yield); $R_f = 0.10$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.53 (ddd, *J* = 5.1, 1.7, 0.9 Hz, 1H), 7.60 (td, *J* = 7.7, 1.8 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.13 (ddd, *J* = 7.4, 4.9, 1.0 Hz, 1H), 4.03 (t, *J* = 7.1 Hz, 1H), 3.01 (dd, *J* = 8.6, 6.9 Hz, 2H), 2.88 – 2.81 (m, 4H), 2.28 – 2.17 (m, 2H), 2.16 – 2.04 (m, 1H), 1.95 – 1.78 (m, 1H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 160.69, 149.39, 136.56, 123.19, 121.39, 46.76, 35.13, 35.00, 30.25, 26.05; HRMS (EI) (m/z) calcd. for $C_{11}H_{15}NS_2$ (M^+) 225.0646, found 225.0644.

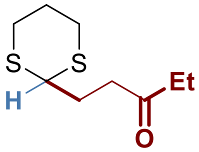
***2-(2-(2-pentyl-1,3-dithian-2-yl)ethyl)pyridine* (3x)**



3x

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3x** was obtained as a yellow liquid (41.4 mg, 70% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.53 (ddd, *J* = 4.9, 1.9, 1.1 Hz, 1H), 7.60 (td, *J* = 7.6, 1.9 Hz, 1H), 7.18 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.11 (ddd, *J* = 7.6, 4.9, 1.2 Hz, 1H), 3.01 – 2.85 (m, 4H), 2.77 (ddd, *J* = 14.5, 6.7, 3.6 Hz, 2H), 2.42 – 2.30 (m, 2H), 2.07 – 1.83 (m, 5H), 1.58 – 1.43 (m, 2H), 1.40 – 1.21 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 161.6, 149.0, 137.0, 123.3, 121.4, 53.3, 38.9, 38.0, 33.5, 32.2, 26.2, 25.6, 23.7, 22.7, 14.2; HRMS (EI) (m/z) calcd. for $C_{16}H_{25}NS_2$ (M$^+$) 295.1428, found 295.1426.

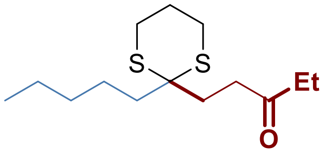
***1-(1,3-dithian-2-yl)pentan-3-one* (3y)**



3y

Following the general procedure (12 h) using 5% ethyl acetate in hexanes as eluent, **3y** was obtained as a yellow liquid (36.4 mg, 89% yield); $R_f = 0.30$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.05 (t, *J* = 7.0 Hz, 1H), 2.89 – 2.76 (m, 4H), 2.65 (t, *J* = 7.3 Hz, 2H), 2.45 (q, *J* = 7.3 Hz, 2H), 2.14 – 2.01 (m, 3H), 1.95 – 1.78 (m, 1H), 1.06 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 210.3, 46.6, 38.9, 36.2, 30.1, 29.2, 26.0, 7.9; HRMS (EI) (m/z) calcd. for $C_9H_{16}OS_2$ (M$^+$) 204.0643, found 204.0640.

***1-(2-pentyl-1,3-dithian-2-yl)pentan-3-one* (3z)**

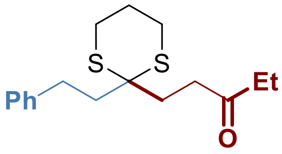


3z

Following the general procedure (12 h) using 6% ethyl acetate in hexanes as eluent, **3z** was obtained as a colorless liquid (44.3 mg, 81% yield); $R_f = 0.60$ (ethyl acetate:hexanes, 1:4); ^{1}H

NMR (400 MHz, Chloroform-*d*) δ 2.88 (ddd, *J* = 12.8, 9.5, 3.1 Hz, 2H), 2.71 (ddd, *J* = 14.5, 6.9, 3.3 Hz, 2H), 2.60 – 2.51 (m, 2H), 2.47 (q, *J* = 7.3 Hz, 2H), 2.28 – 2.20 (m, 2H), 2.04 – 1.81 (m, 2H), 1.80 – 1.72 (m, 2H), 1.49 – 1.38 (m, 2H), 1.36 – 1.23 (m, 4H), 1.06 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.84, 52.96, 39.28, 37.75, 36.43, 32.14, 31.49, 26.15, 25.38, 23.60, 22.63, 14.17, 8.03; HRMS (ESI$^+$) (m/z) calcd. for $C_{14}H_{26}OS_2$ (M+Na)$^+$ 297.1317, found 297.1305.

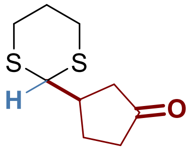
1-(2-phenethyl-1,3-dithian-2-yl)pentan-3-one **(3aa)**



3aa

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3aa** was obtained as a light yellow liquid (45.8 mg, 74% yield); R$_f$ = 0.50 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.16 (dd, *J* = 6.6, 5.5 Hz, 2H), 7.11 – 7.05 (m, 3H), 2.76 (ddd, *J* = 11.9, 8.1, 3.7 Hz, 2H), 2.71 – 2.62 (m, 4H), 2.55 – 2.46 (m, 2H), 2.36 (q, *J* = 7.3 Hz, 2H), 2.22 – 2.14 (m, 2H), 2.03 – 1.94 (m, 2H), 1.92 – 1.74 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.61, 141.77, 128.59, 128.53, 126.10, 52.82, 41.33, 37.58, 36.39, 31.86, 30.70, 26.11, 25.19, 8.01; HRMS (ESI$^+$) (m/z) calcd. for $C_{17}H_{23}OS_2$ (M+H)$^+$ 309.1341, found 309.1327.

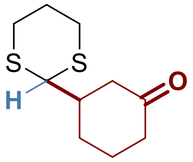
3-(1,3-dithian-2-yl)cyclopentan-1-one **(3ab)**[7]



3ab

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3ab** was obtained as a colorless liquid (21.8 mg, 54% yield); R$_f$ = 0.20 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.10 (d, *J* = 7.5 Hz, 1H), 2.91 – 2.80 (m, 4H), 2.59 – 2.41 (m, 2H), 2.40 – 2.06 (m, 5H), 1.95 – 1.77 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 217.5, 52.4, 42.9, 41.7, 38.8, 30.5, 29.8, 27.2, 26.0.

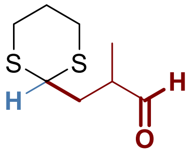
3-(1,3-dithian-2-yl)cyclohexan-1-one **(3ad)**[5]



3ad

Following the general procedure (24 h) using 7% ethyl acetate in hexanes as eluent, **3ad** was obtained as a light yellow liquid (19.9 mg, 46% yield); $R_f = 0.30$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.07 (d, *J* = 4.9 Hz, 1H), 2.93 – 2.82 (m, 4H), 2.59 – 2.50 (m, 1H), 2.49 – 2.23 (m, 3H), 2.22 – 2.03 (m, 4H), 1.93 – 1.78 (m, 1H), 1.74 – 1.56 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 210.66, 53.62, 45.33, 43.65, 41.28, 30.85, 30.83, 28.54, 26.21, 24.90.

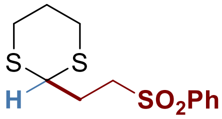
3-(1,3-dithian-2-yl)-2-methylpropanal **(3af)**[17]



3af

Following the general procedure (12 h) using 6% ethyl acetate in hexanes as eluent, **3af** was obtained as a colorless liquid (17.1 mg, 45% yield); $R_f = 0.60$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.69 – 9.64 (m, 1H), 4.06 (t, *J* = 7.5 Hz, 1H), 2.90 – 2.80 (m, 4H), 2.75 (h, *J* = 7.0 Hz, 1H), 2.25 (dt, *J* = 14.6, 7.4 Hz, 1H), 2.17 – 2.07 (m, 1H), 1.88 (tp, *J* = 14.5, 7.4 Hz, 1H), 1.79 – 1.71 (m, 1H), 1.16 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.61, 44.78, 43.56, 36.06, 30.12, 30.06, 25.92, 13.83.

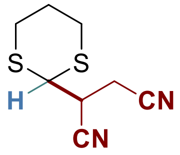
3-(1,3-dithian-2-yl)-2-methylpropanal **(3ag)**[5]



3ag

Following the general procedure (12 h) using 7% ethyl acetate in hexanes as eluent, **3ag** was obtained as a white solid (32.6 mg, 57% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.90 (m, 2H), 7.70 – 7.65 (m, 1H), 7.61 – 7.56 (m, 2H), 4.02 (t, *J* = 7.0 Hz, 1H), 3.34 – 3.30 (m, 2H), 2.87 – 2.75 (m, 4H), 2.24 – 2.18 (m, 2H), 2.12 – 2.04 (m, 1H), 1.89 – 1.78 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.92, 134.02, 129.54, 128.21, 53.42, 45.01, 29.65, 28.52, 25.62.

2-(1,3-dithian-2-yl)succinonitrile (3ah)[3]

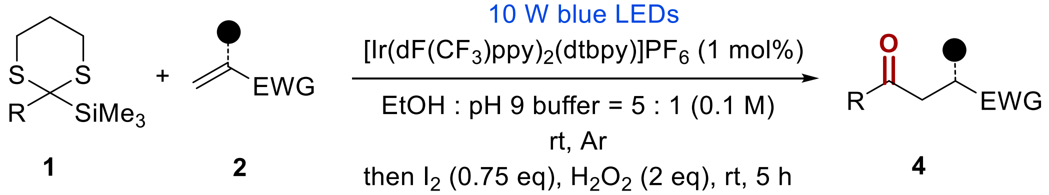


3ah

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h) using 7% ethyl acetate in hexanes as eluent, **3ah** was obtained as a colorless liquid (24 mg, 60% yield); $R_f = 0.20$ (ethyl acetate:hexanes, 1:4); ^1H NMR (300 MHz, Chloroform-*d*) δ 4.13 (d, *J* = 7.1 Hz, 1H), 3.43 (td, *J* = 7.1, 6.3 Hz, 1H), 3.13 – 2.91 (m, 4H), 2.93 – 2.78 (m, 2H), 2.21 – 1.91 (m, 2H); ^{13}C NMR (75 MHz, Chloroform-*d*) δ 116.8, 115.3, 44.1, 34.9, 28.4, 28.3, 24.6, 20.0.

VI. General procedure and characterization data of ketone 4

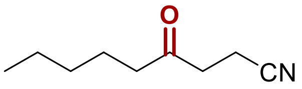
A. General procedure for ketone 4



To a re-sealable pressure tube (13 x 100 mm) equipped with a small magnetic stir bar were added silane **1** (0.2 mmol, 1.0 equiv), alkene **2** (0.4 mmol, 2.0 equiv), and [Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$ (0.002 mmol, 1 mol%) under argon atmosphere. The reaction mixture was dissolved in degassed solvents (2 mL, 0.1 M) of EtOH/pH 9 buffer in a ratio of 5:1 or MeCN/MeOH in a ratio of 3:1. The mixture was irradiated with 2 x 5W blue LEDs using a customized milligram scale reaction set up (as shown in **Figure S2**) under constant stirring condition at room temperature. After completion of reaction, addition of I$_2$ (0.15 mmol, 0.75 equiv) and H$_2$O$_2$ (30%) (0.4 mmol, 2 equiv) were conducted and the resulting mixture was stirred at room temperature for 5 h when its TLC examination showed complete disappearance of the starting material. The reaction was quenched by the addition of Na$_2$S$_2$O$_3$ solution, and the resulting mixture was extracted with ethyl acetate. The combined organic extracts were washed with brine and dried. The solvent then removed under reduced pressure and residue was purified by flash column chromatography on silica gel to afford the corresponding ketone product **4**.

B. Characterization data of ketone 4

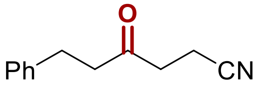
4-oxononanenitrile **(4b)**[8]



4b

Following the general procedure (12 h for photoreaction) using 10% ethyl acetate in hexanes as eluent, **4b** was obtained as a colorless liquid (20.8 mg, 68% yield); R$_f$ = 0.40 (ethyl acetate:hexanes, 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.79 (t, *J* = 7.2 Hz, 2H), 2.58 (t, *J* = 7.2 Hz, 2H), 2.44 (t, *J* = 7.5 Hz, 2H), 1.64-1.56 (m, 2H), 1.37 – 1.22 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.47, 119.20, 42.61, 37.80, 31.39, 23.50, 22.50, 13.99, 11.50.

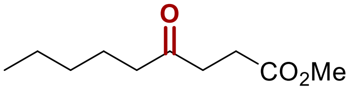
4-oxo-6-phenylhexanenitrile (4c)[9]



4c

Following the general procedure (using MeCN:MeOH (3:1) (0.1 M) in 16 h for photoreaction then I_2 (6 equiv) using EtOH/pH 9 buffer in a ratio of 5:1 at 0 °C for 5 h) using 32% ethyl acetate in hexanes as eluent, **4c** was obtained as a light yellow liquid (26.3 mg, 70% yield); $R_f = 0.40$ (ethyl acetate:hexanes, 1:3); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 2H), 7.24 – 7.14 (m, 3H), 2.93 (t, *J* = 7.5 Hz, 2H), 2.76 (td, *J* = 7.5, 7.4 Hz, 4H), 2.56 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 205.39, 140.43, 128.73, 128.37, 126.48, 119.09, 44.03, 38.09, 29.72, 11.42.

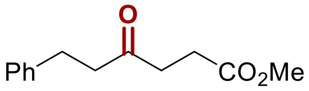
Methyl 4-oxononanoate (4h)[10]



4h

Following the general procedure (12 h for photoreaction) using 10% ethyl acetate in hexanes as eluent, **4h** was obtained as a colorless liquid (22.4 mg, 60% yield); $R_f = 0.40$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.67 (s, 3H), 2.71 (t, *J* = 6.5 Hz, 2H), 2.58 (t, *J* = 6.5 Hz, 2H), 2.43 (t, *J* = 7.5 Hz, 2H), 1.63 – 1.53 (m, 2H), 1.33 – 1.21 (m, 4H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.33, 173.48, 51.91, 42.90, 37.13, 31.49, 27.84, 23.62, 22.56, 14.04.

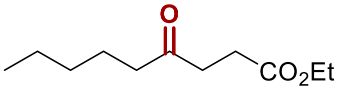
Methyl 4-oxo-6-phenylhexanoate (4i)[11]



4i

Following the general procedure (24 h for photoreaction then I_2 (6 equiv) at 0 °C for 5 h) using 10% ethyl acetate in hexanes as eluent, **4i** was obtained as a colorless liquid (28.4 mg, 64% yield); $R_f = 0.40$ (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.17 (m, 2H), 7.15 – 7.05 (m, 3H), 3.60 (s, 3H), 2.87 – 2.81 (m, 2H), 2.75 – 2.68 (m, 2H), 2.64 (t, *J* = 6.5 Hz, 2H), 2.51 (t, *J* = 6.5 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.06, 173.35, 141.03, 128.61, 128.41, 126.24, 51.92, 44.36, 37.33, 29.77, 27.79.

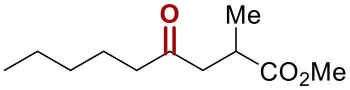
***Ethyl 4-oxononanoate* (4k)**[12]



4k

Following the general procedure (24 h for photoreaction) using 10% ethyl acetate in hexanes as eluent, **4k** was obtained as a colorless liquid (25.3 mg, 63% yield); R_f = 0.40 (ethyl acetate:hexanes, 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.12 (q, *J* = 7.1 Hz, 2H), 2.71 (t, *J* = 6.6 Hz, 2H), 2.57 (t, *J* = 6.6 Hz, 2H), 2.44 (t, *J* = 7.5 Hz, 2H), 1.63 – 1.55 (m, 2H), 1.34 – 1.22 (m, 7H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.37, 173.01, 60.73, 42.93, 37.16, 31.51, 28.14, 23.63, 22.58, 14.31, 14.05.

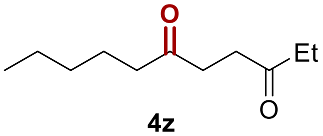
***Methyl 2-methyl-4-oxononanoate* (4o)**



4o

Following the general procedure (12 h for photoreaction) using 10% ethyl acetate in hexanes as eluent, **4o** was obtained as a colorless liquid (25.8 mg, 63% yield); R_f = 0.60 (ethyl acetate:hexanes, 1:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.66 (s, 3H), 2.99 – 2.81 (m, 2H), 2.49 – 2.32 (m, 3H), 1.61 – 1.51 (m, 2H), 1.33 – 1.21 (m, 4H), 1.16 (d, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.26, 176.48, 51.98, 45.88, 43.06, 34.70, 31.48, 23.54, 22.57, 17.25, 14.04; HRMS (ESI$^+$) (m/z) calcd. for $C_{11}H_{20}O_3$ (M+Na)$^+$ 223.1305, found 223.1308.

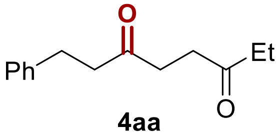
***Undecane-3,6-dione* (4z)**[13]



4z

Following the general procedure (12 h for photoreaction) using 10% ethyl acetate in hexanes as eluent, **4z** was obtained as a white solid (23.2 mg, 63% yield); R_f = 0.40 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.67 (s, 4H), 2.52 – 2.39 (m, 4H), 1.62 – 1.52 (m, 2H), 1.35 – 1.20 (m, 4H), 1.05 (t, *J* = 7.3 Hz, 3H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.30, 210.03, 42.96, 36.19, 36.08, 35.73, 31.51, 23.65, 22.57, 14.05, 7.93.

***1-phenyloctane-3,6-dione* (4aa)**[14]



4aa

Following the general procedure (24 h for photoreaction then I_2 (6 equiv) at 0 ºC for 5 h) using 10% ethyl acetate in hexanes as eluent, **4aa** was obtained as a light yellow liquid (33.8 mg, 77% yield); R_f = 0.40 (ethyl acetate:hexanes, 1:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.17 (m, 2H), 7.15 – 7.06 (m, 3H), 2.86 – 2.79 (m, 2H), 2.76 – 2.69 (m, 2H 2.65 – 2.56 (m, 4H), 2.41 (q, *J* = 7.3 Hz, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.17, 208.74, 141.13, 128.59, 128.40, 126.19, 44.42, 36.34, 36.02, 35.72, 29.81, 7.91.

VII. Electrochemical measurements

Cyclic voltammograms

Electrochemical study was performed using a Bio-Logic (SP-300 model). The redox potentials of dithiane **5**, silane **1a** (vs Ag/AgCl) were determined through cyclic voltammetry using a 5.0 mM solution of the material in 0.1 M solution of Bu_4NPF_6 (purged MeCN with N_2). Measurements employed a glassy carbon electrode, platinum wire counter electrode, 3.0 M NaCl Ag/AgCl reference electrode, a scan rate 50 mV/s. The obtained value was referenced to Ag/AgCl and converted to SCE by subtracting 0.032 V.

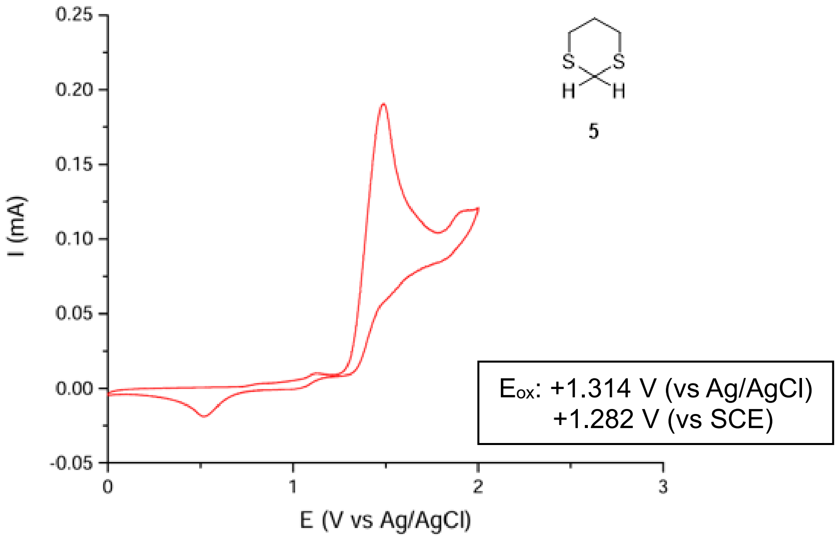
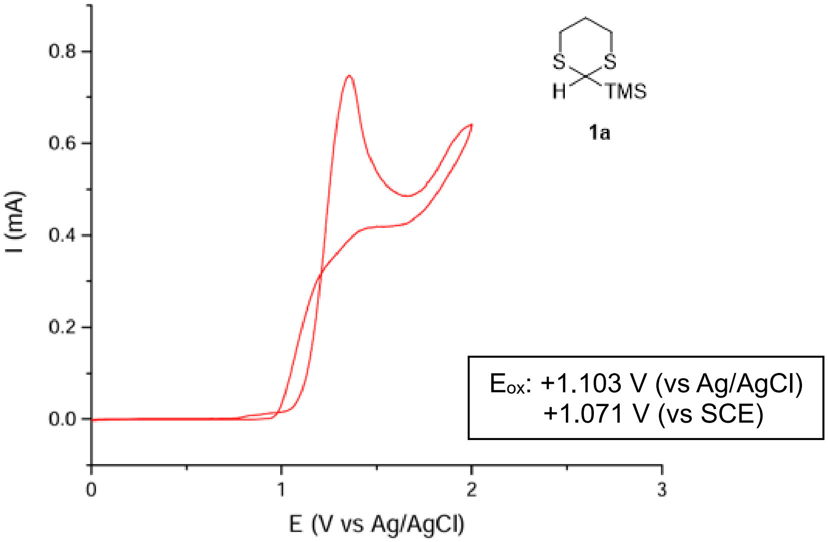


Figure S4. Cyclic voltammogram study for **5**



E_{ox}: +1.103 V (vs Ag/AgCl)
+1.071 V (vs SCE)

Figure S5. Cyclic voltammogram study for **1a**

VIII. Mechanistic studies

A. Luminescence quenching experiment

Catalyst [Ir(dF(CF$_3$)ppy)$_2$(dttbpy)]]PF$_6$ was excited at 435 nm in MeCN solution, and the emission intensity was observed. MeCN solution was degassed with a stream of argon gas for 30 min. In a typical experiment, the emission spectrum of a 1.0 x 10^{-4} M solution of [Ir(dF(CF$_3$)ppy)$_2$(dttbpy)]]PF$_6$ in MeCN solution was collected. Then, **1a** or **2a** was added to the measured solution in a quartz cuvette and the emission spectrum of the sample was measured. I$_0$ and I signify the intensities of the emission in the absence and presence of the quencher at ~471 nm. The steady decrease of the emission intensity of the catalyst solution with the gradual increase of the amount **1a** as presented in **Figure S6** supports that reaction mechanism occurred through single electron transfer (SET). Stern-Volmer plot also provided to show the reaction between photocatalysts and the quencher **1a**.

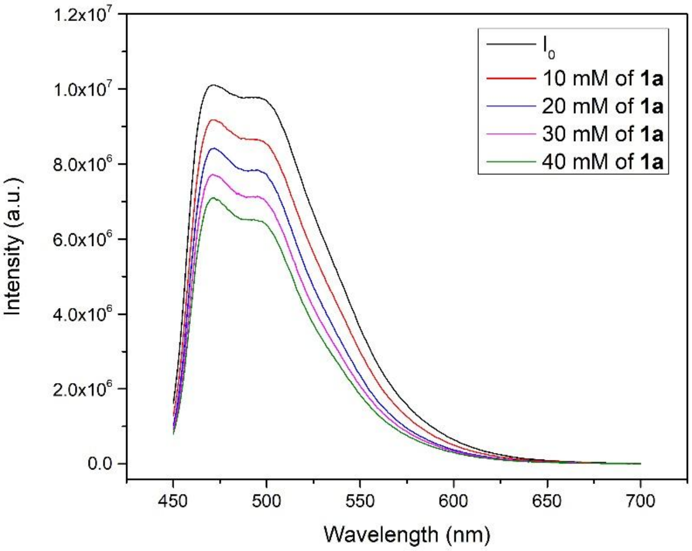


Figure S6. Luminescence quenching by **1a**

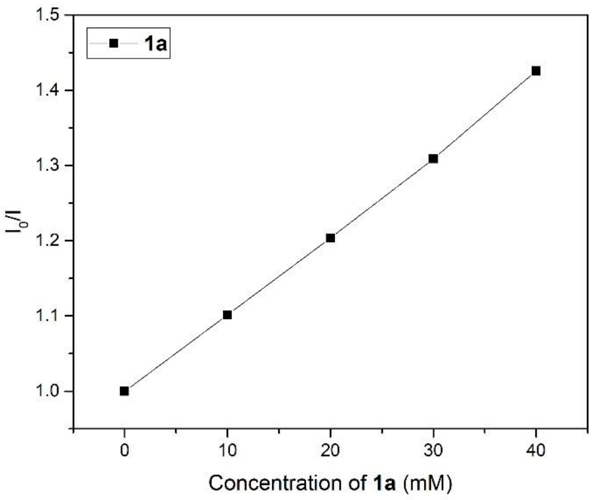
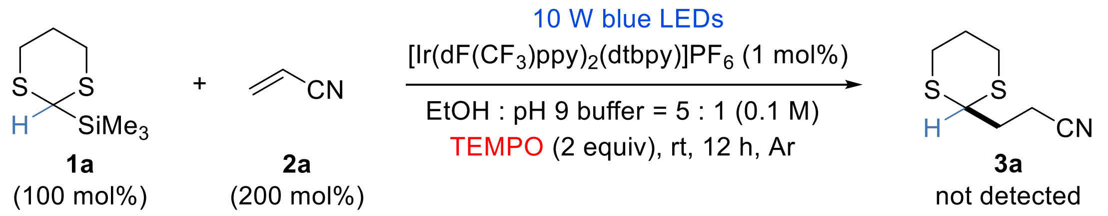


Figure S7. Stern-Volmer equation for **1a**

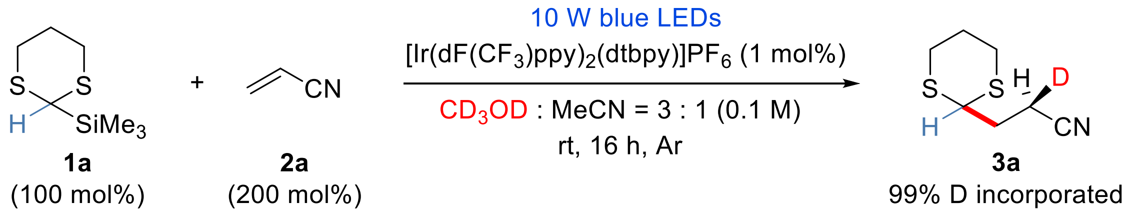
B. Radical trapping experiment

To investigate the nature of the reactions, controlled reactions were carried out with the radical scavenger 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) and Giese adduct **3a** was not detected, complete inhibition of the reaction, implying that the reaction follows the radical pathway.



C. Deuterium labelling experiment

To investigate the proton source for the formation of product, control experiments were performed with MeOH-*d* instead of MeOH as a solvent, keeping other parameters unchanged. ^{1}H NMR confirmed the presence of deuterium atom in the final crude product (^{1}H NMR spectrum of crude product is attached in spectra section).



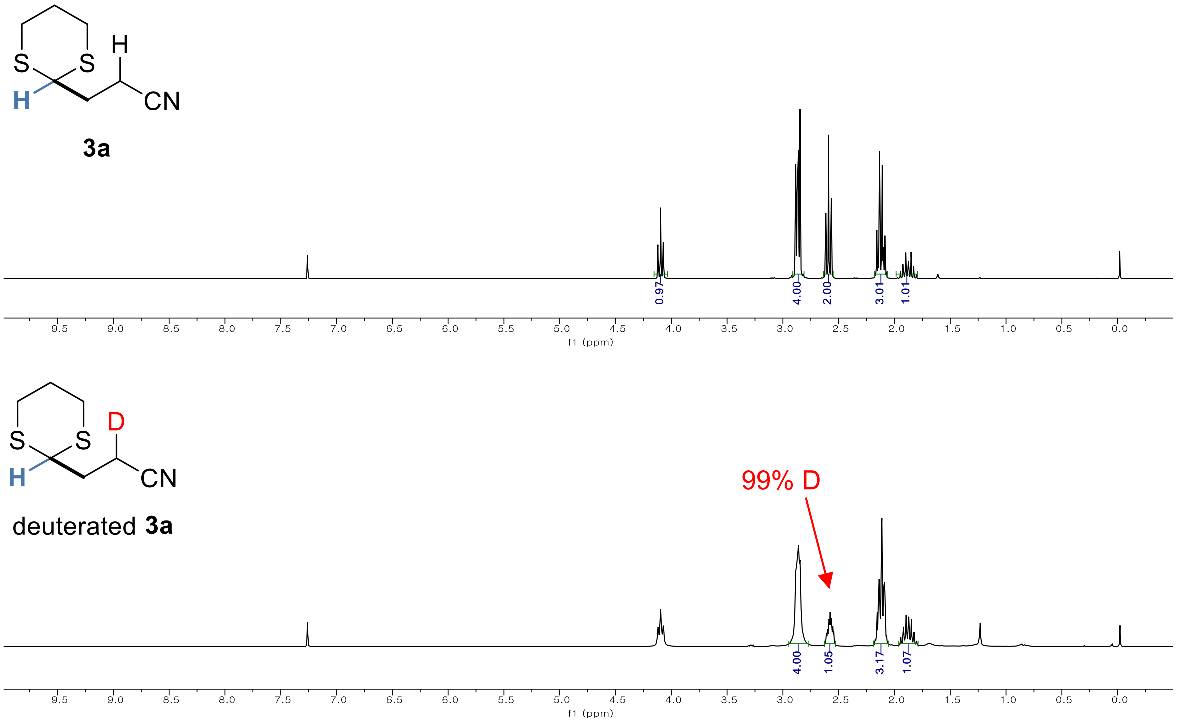
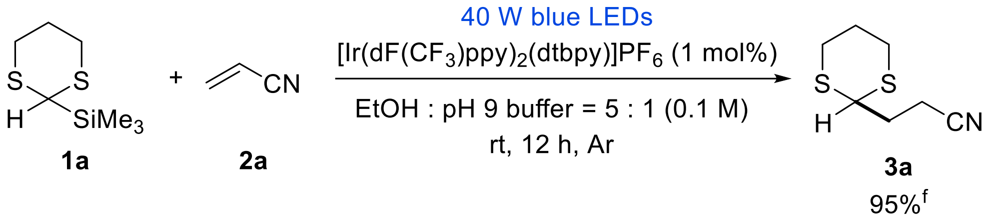


Figure S8. Comparison between **3a** and deuterated **3a**

D. Byproducts analysis experiment

To gain insight into the mechanism of $SiMe_3$ cation generation, by product analysis was performed on the reaction mixture. GC-MS analysis identified ethoxytrimethylsilane and hexamethyldisiloxane (Me_3Si-O-$SiMe_3$) as byproducts. The formation of ethoxytrimethylsilane is consistent with the reaction of $SiMe_3$ cation with ethanol present in the protic solvent system, while hexamethyldisiloxane likely arises from condensation of trimethylsilanol intermediate, although the latter was not detected under the reaction conditions. Both byproducts were confirmed by comparison with known mass spectral data[18].



[f] ^{1}H NMR yield (IS = mesitylene)

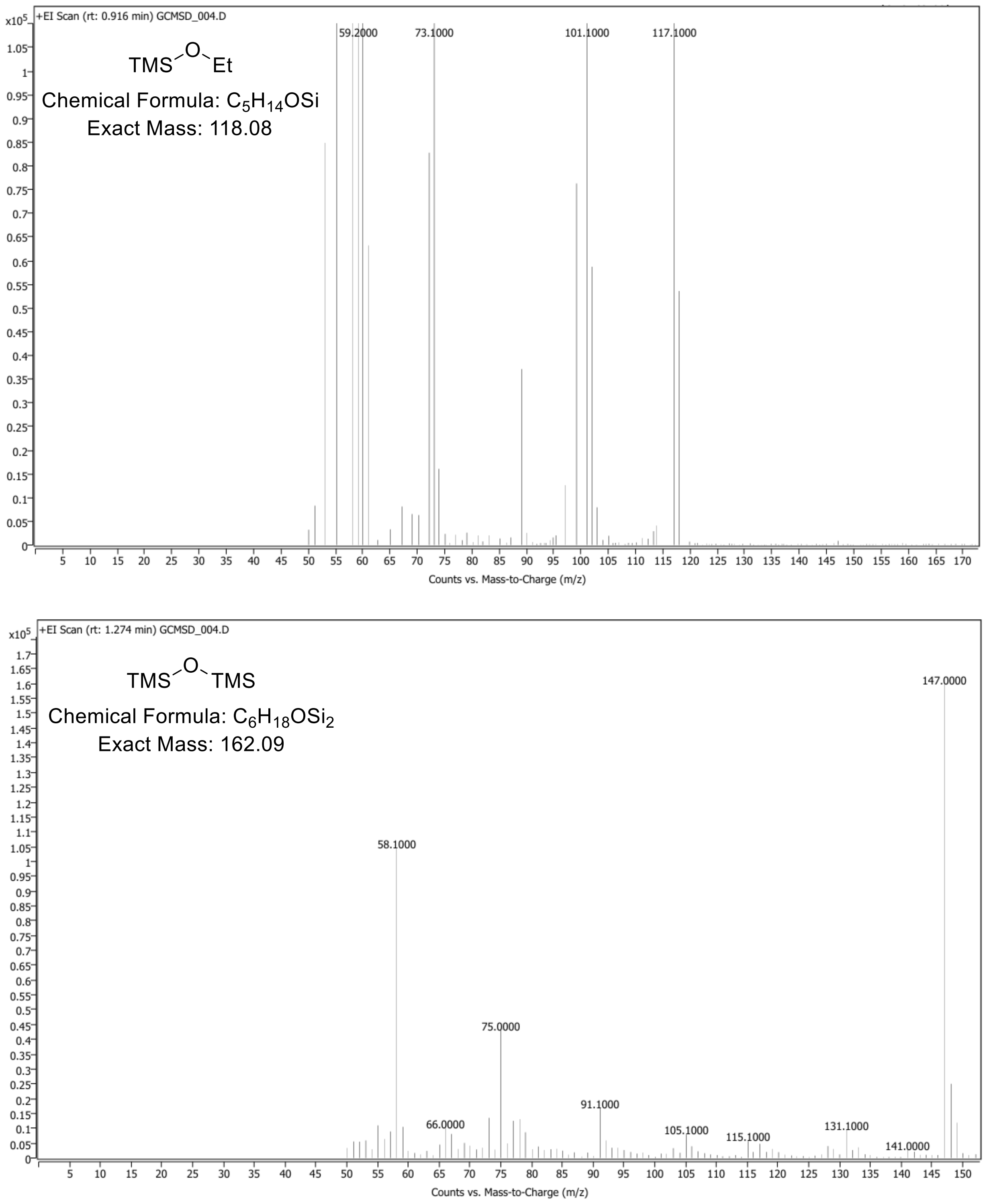
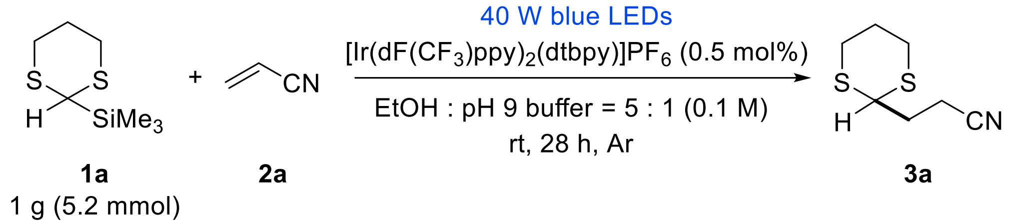


Figure S9. GC-MS spectra of byproducts identified in the reaction mixture: ethoxymethylsilane ($C_5H_{14}OSi$, exact mass: 118.08, top) and hexamethyldisiloxane ($C_6H_{18}OSi_2$, exact mass: 162.09, bottom).

IX. Gram scale experiment



An oven-dried 100 mL round-bottomed flask containing a magnetic stir bar was charged with silane **1a** (1.0 g, 5.2 mmol, 1.0 equiv), alkene **2a** (10.4 mmol, 2.0 equiv), and [Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$ (0.026 mmol, 0.5 mol%) under argon atmosphere. The reaction mixture was dissolved in degassed solvents (52 mL, 0.1 M) of EtOH/pH 9 buffer in a ratio of 5:1. The mixture was irradiated with 40W Kessil blue LEDs (100% intensity) under constant stirring condition at room temperature for 28 h. After completion of the reaction confirmed by the TLC analysis, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography using ethyl acetate/hexanes as the eluent to afford the corresponding product **3a** (713.6 mg, 79%).

X. Calculation of green chemistry metrics

To evaluate the green chemistry aspects of the developed reaction, we calculated the green chemistry metrics, including Atom Economy (AE), Atom Efficiency (AEf), Reaction Mass Efficiency (RME), E-Factor, and Process Mass Intensity (PMI)[15]. Additionally, we compared these metrics with two other efficient synthetic methods for ketones. The chemistry metrics were calculated using the formulas provided below.

$$\text{Atom Economy (AE)(\%)} = \frac{\text{Molecular Weight of Desired Product}}{\text{Sum of Molecular Weight of All Reactants}} \times 100$$

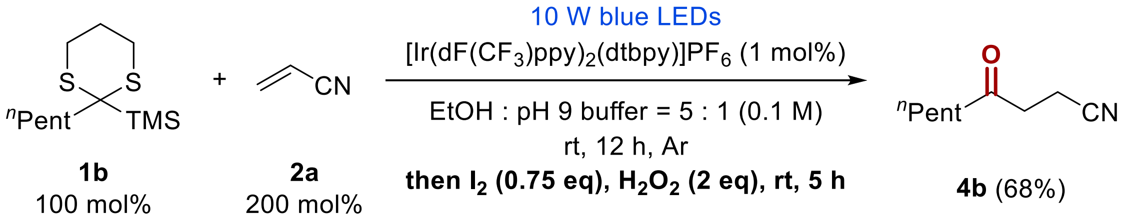
$$\text{Atom Efficiency (AEf)(\%)} = \text{Atom Economy (AE)(\%)} \times \text{yield}$$

$$\text{Reaction Mass Efficiency (RME)(\%)} = \frac{\text{Mass of Desired Product}}{\text{Mass of Reactants used}} \times 100$$

$$\text{E} - \text{Factor} = \frac{\text{Mass of Waste (kg)}}{\text{Mass of Product (kg)}}$$

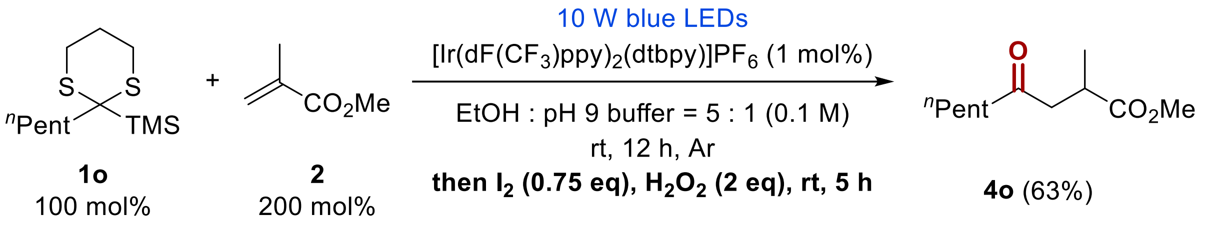
$$\text{Process Mass Intensity (PMI)} = \frac{\text{Total Mass of Inputs (Reactants, Solvents, etc.)}}{\text{Mass of Desired Product}}$$

A. Our work (4b)



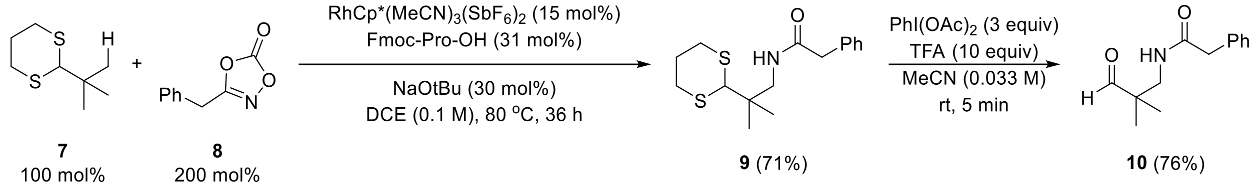
	mmol	mg	MW	Green Chemistry Metrics	
1b	0.2	52.51	262.55	AE (%)	35.09%
2a	0.4	21.22	53.06	AEf (%)	23.86%
4b	0.136	20.84	153.23	RME (%)	23.86%
I_2	0.15	38.07	253.81	E-Factor	68.23
H_2O_2	0.4	13.6	34.01	PMI	69.23
catalyst	0.001	2.2	1121.91		
solvent		1315			

B. Our work (4o)



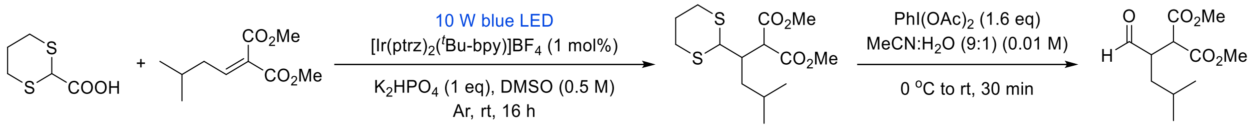
	mmol	mg	MW	Green Chemistry Metrics	
1b	0.2	52.51	262.55	AE (%)	37.73%
2a	0.4	40.05	100.121	AEf (%)	23.77%
4b	0.126	25.84	200.28	RME (%)	23.77%
I_2	0.15	38.07	253.81	E-Factor	56.91
H_2O_2	0.4	13.6	34.01	PMI	57.91
catalyst	0.001	2.2	1121.91		
solvent		1315			

C. Dixon group work (2019 Chemical Science)[16]



	mmol	mg	MW	Green Chemistry Metrics	
7	0.1	17.63	176.336	AE (%)	8.32%
8	0.2	35.43	177.159	AEf (%)	4.49%
10	0.054	11.84	219.284	RME (%)	5.84%
$RhCp^*(MeCN)_3(SbF_6)_2$	0.02	3.81	253.81	E-Factor	265.68
Fmoc-Pro-OH	0.031	10.46	337.37	PMI	266.68
NaOtBu	0.03	2.88	96.1		
$PhI(OAc)_2$	0.21	68.61	322.1		
TFA	0.71	80.95	114.02		
solvent (DCE)		1253			
Solvent (MeCN)		1685.02			

D. Cozzi group work (2017 Chemical Science)[5]



11 100 mol% **12** 200 mol% **13** (87%) **14** (76%)

	mmol	mg	MW	**Green Chemistry Metrics**	
11	0.2	32.85	164.237	AE (%)	21.32%
12	0.4	80.09	200.234	AEf (%)	14.07%
14	0.13	30.39	230.26	RME (%)	15.00%
K_2HPO_4	0.2	34.84	174.176	E-Factor	61.84
$PhI(OAc)_2$	0.28	89.67	322.1	PMI	62.84
catalyst	0.002	1.7	835.74		
solvent (DMSO)		440			
solvent (MeCN)		1230.88			

E. Comparison of Green Chemistry Metrics

	AE (%)	AEf (%)	RME (%)	E-Factor	PMI
Our work (4b)	35.09%	23.86%	23.86%	68.23	69.23
Our work (4o)	37.73%	23.77%	23.77%	56.91	57.91
Dixon group work (2019 Chemical Science)	8.32%	4.49%	5.84%	265.68	266.68
Cozzi group work (2019 Chemical Science)	21.32%	14.07%	15.00%	61.84	62.84

XI. References

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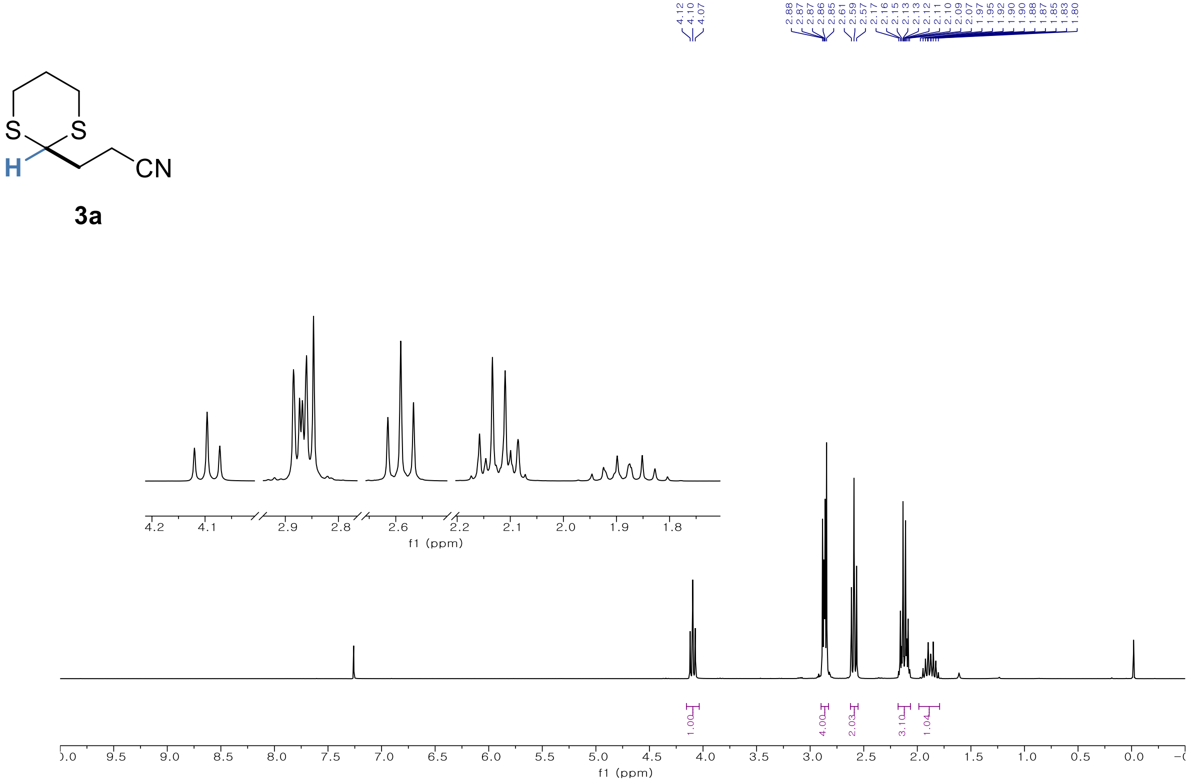
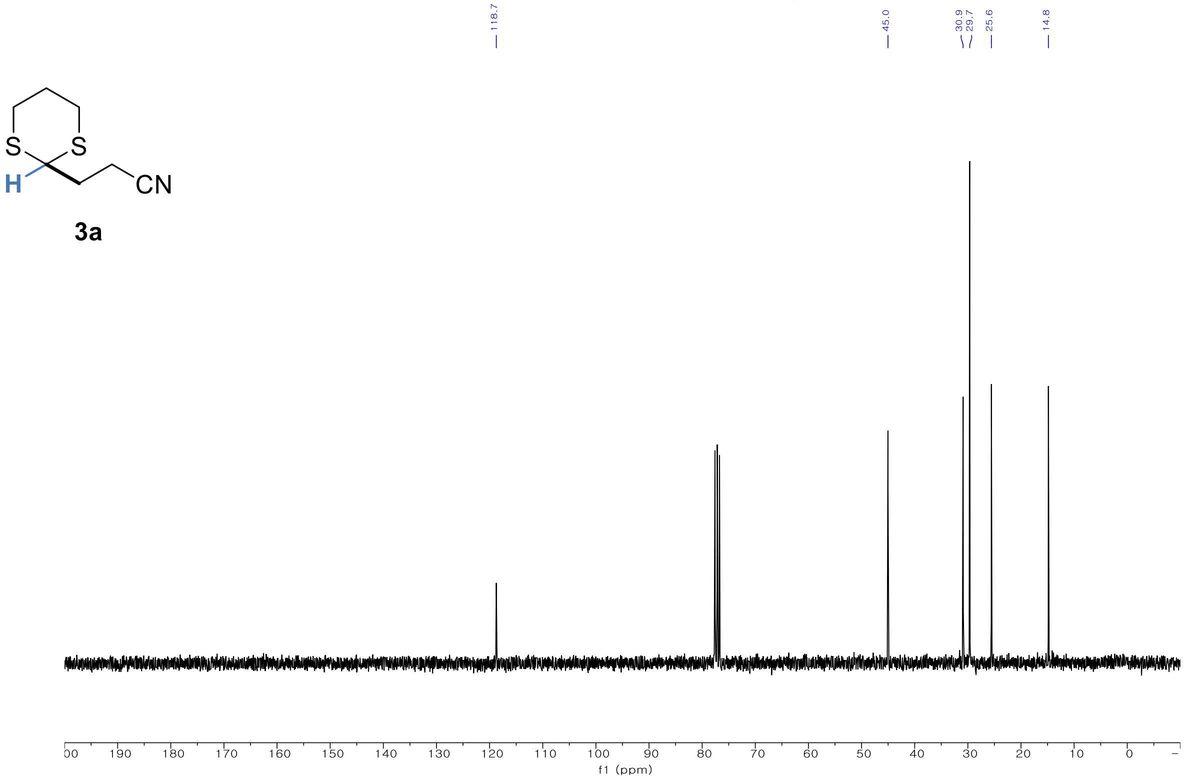
18. Source "Integrated Spectral Data Base System of Organic Compounds" data were obtained from the National Institute of Advanced Industrial Science and Technology (Japan).

XII. ^{1}H and ^{13}C spectra



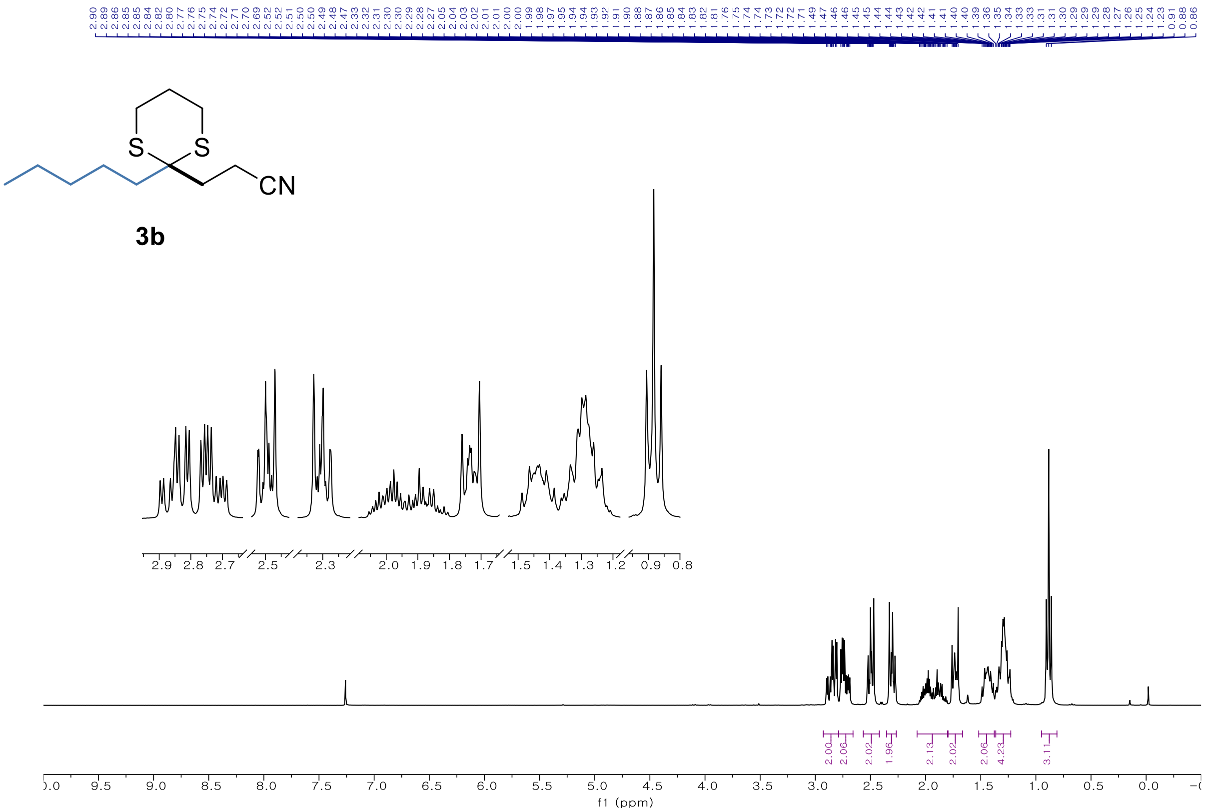
3a

^{1}H-NMR (300 MHz, Chloroform-*d*) of **3a**

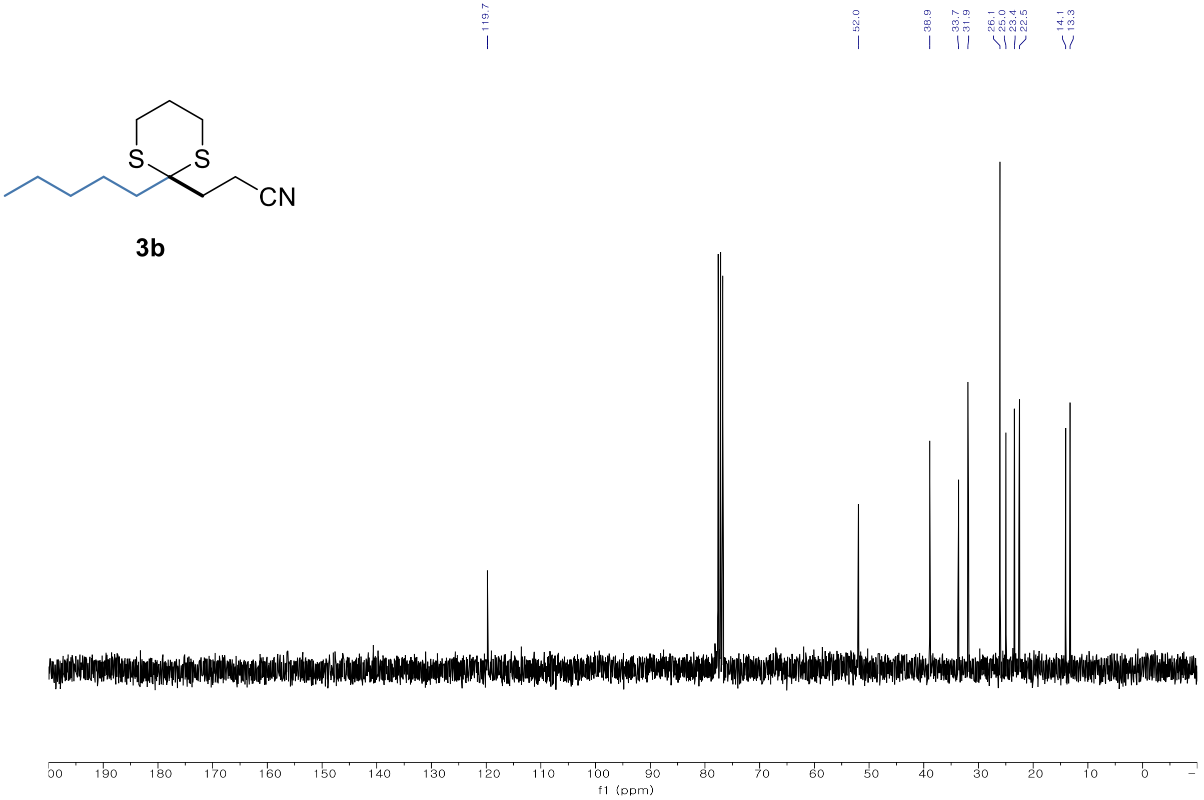


3a

^{13}C-NMR (75 Hz, Chloroform-*d*) of **3a**



^{1}H-NMR (300 MHz, Chloroform-*d*) of **3b**



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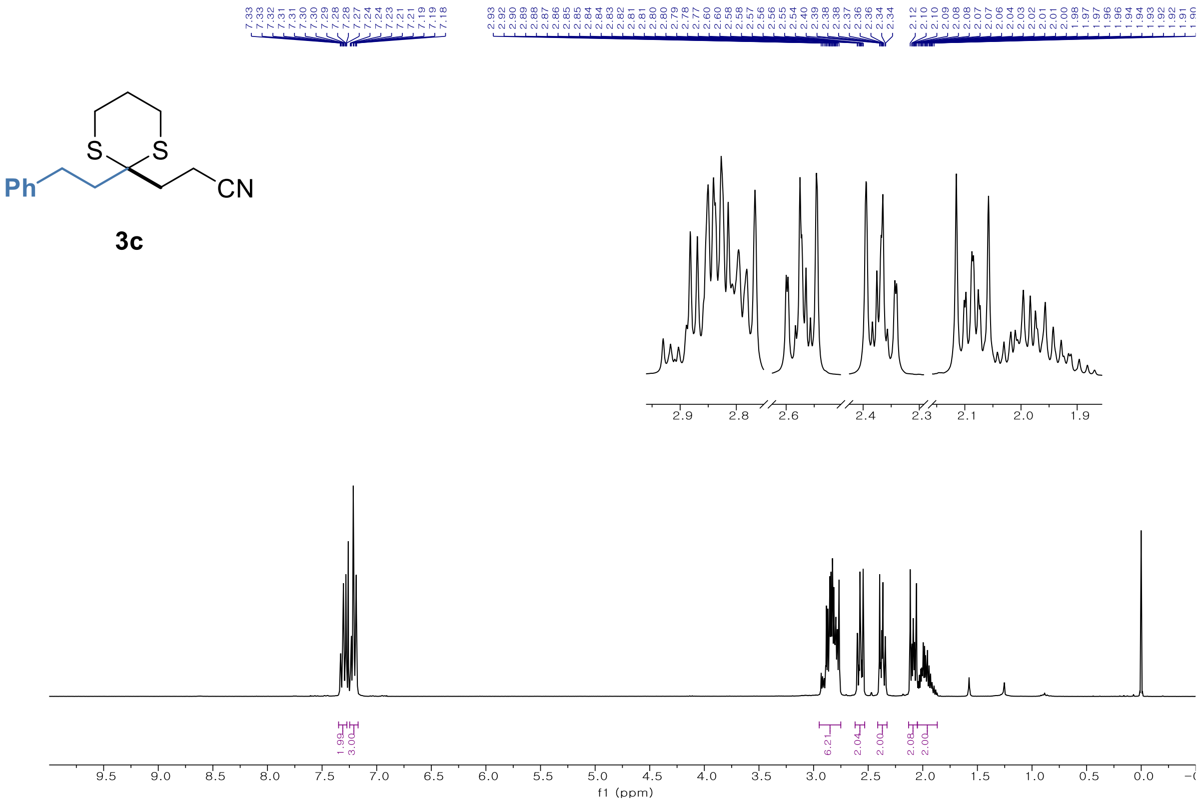
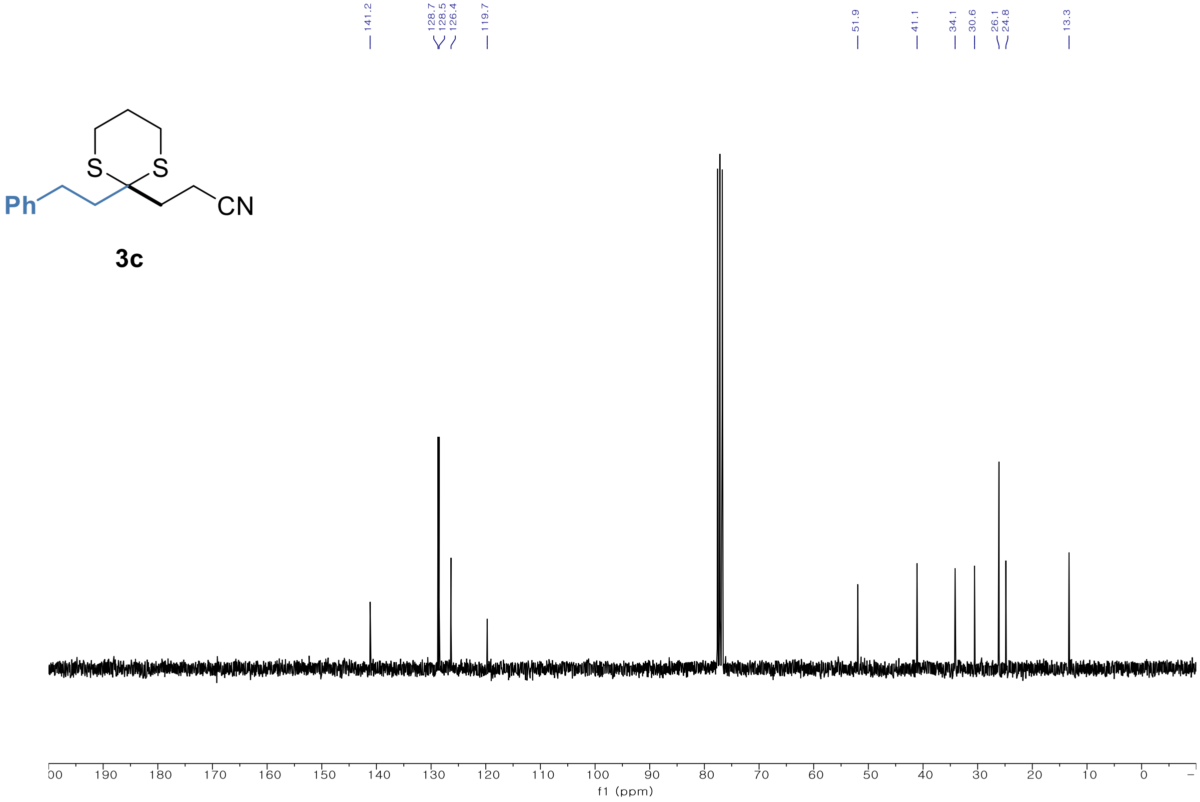


^{1}H-NMR (300 MHz, Chloroform-*d*) of **3c**



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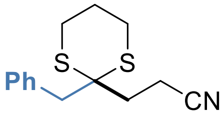
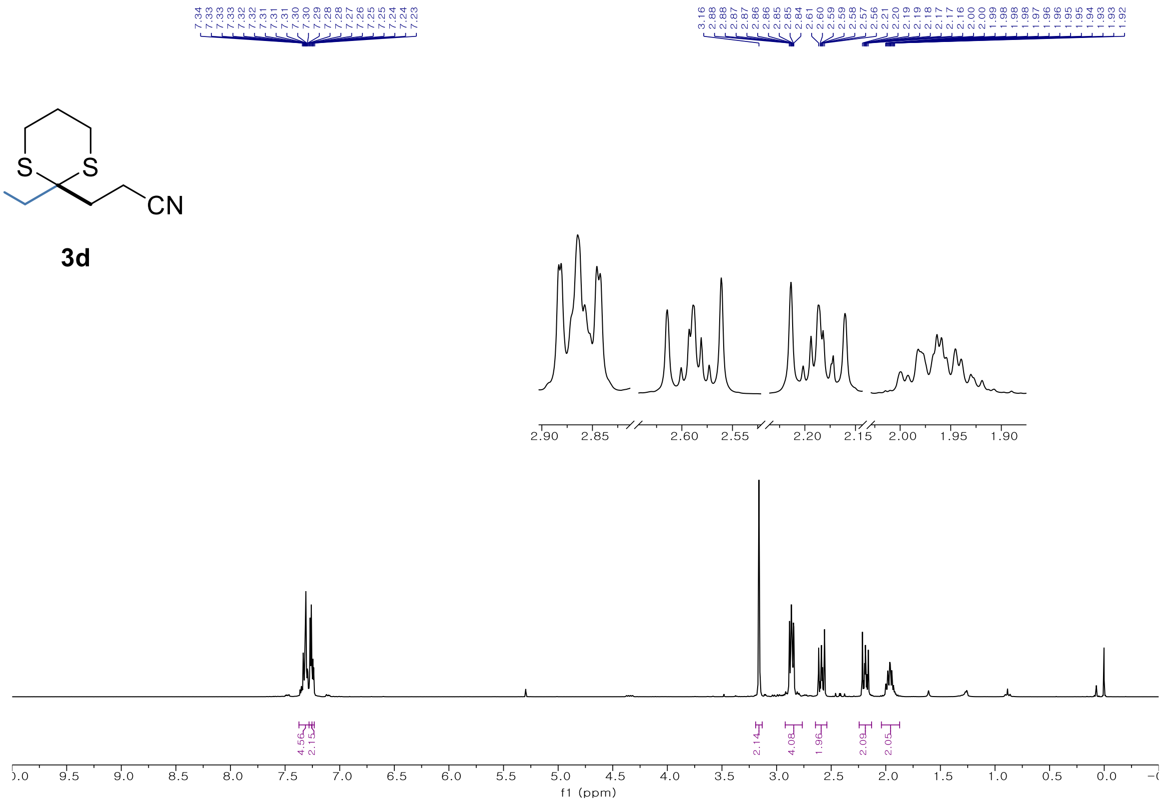


3d



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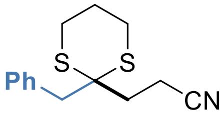

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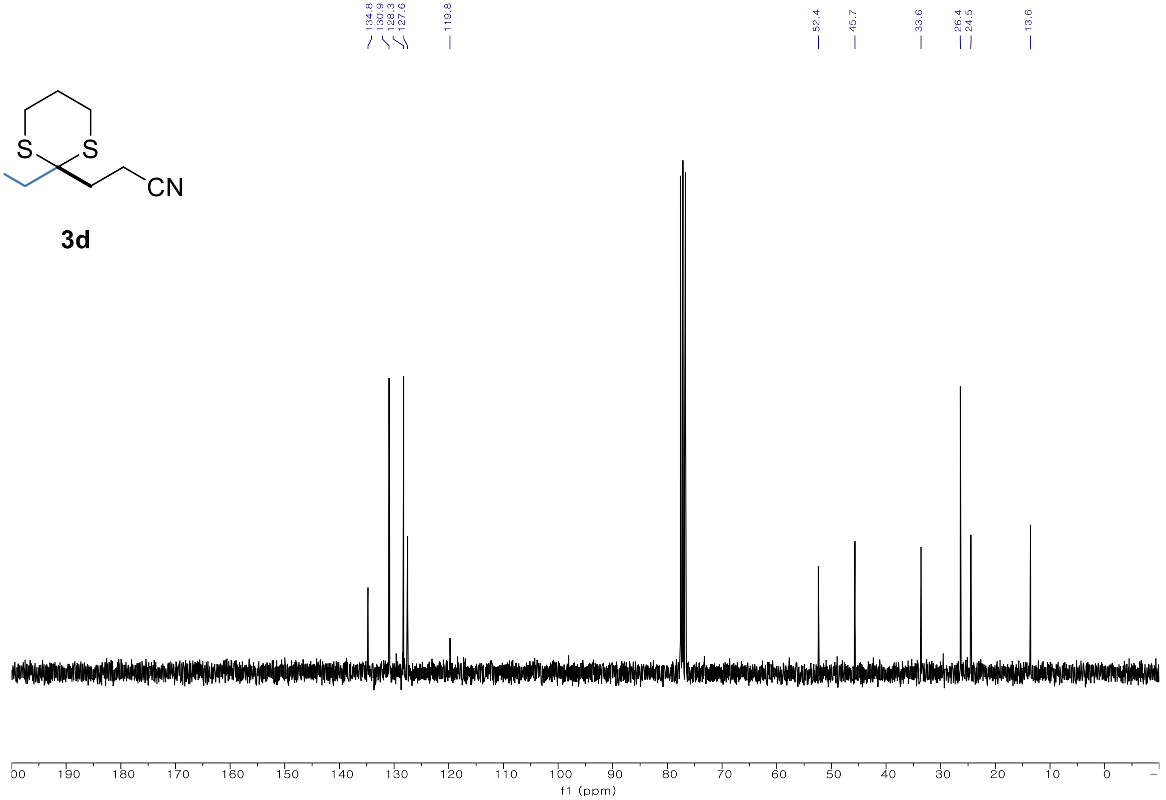

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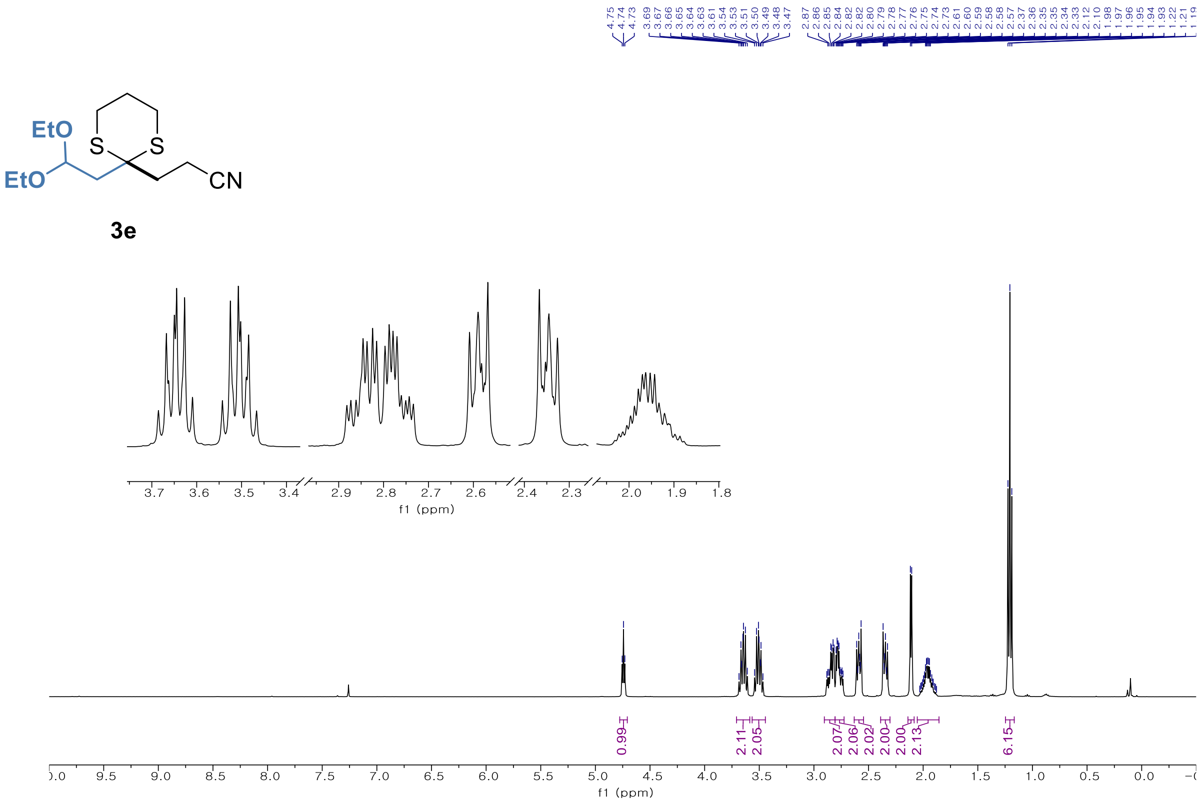
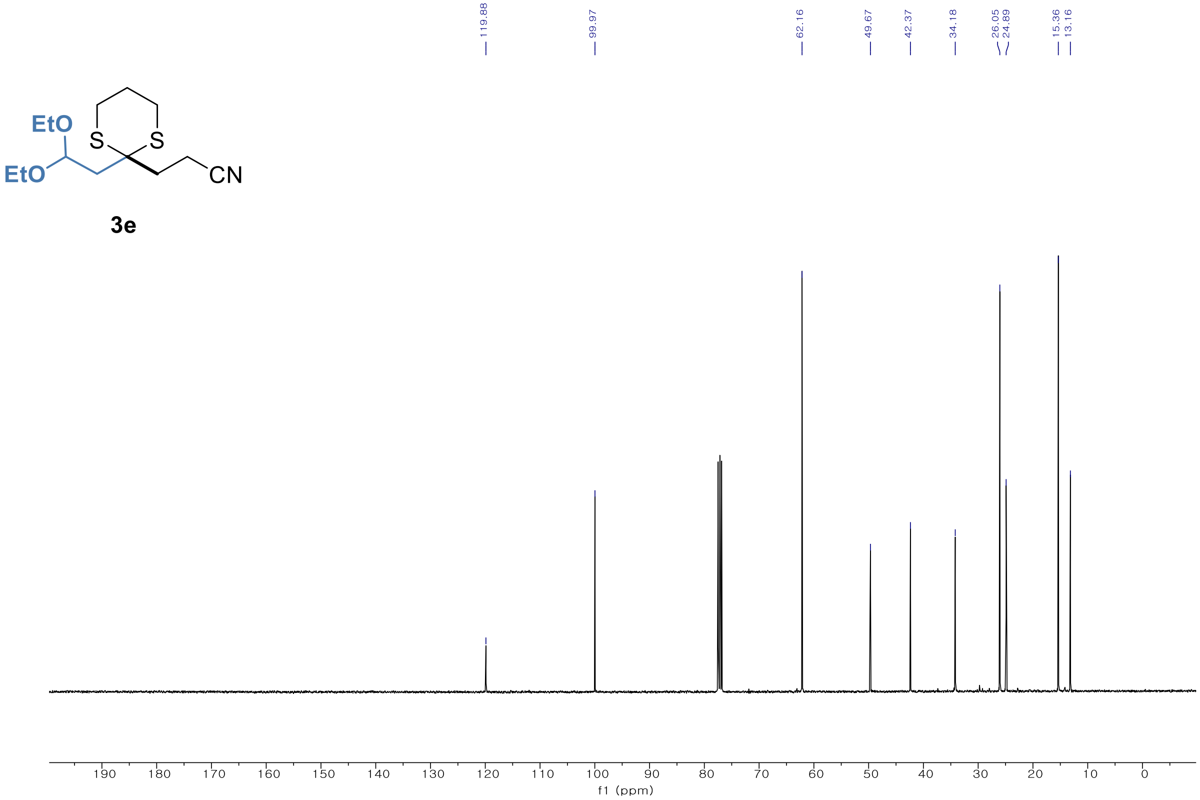


^{1}H-NMR (400 MHz, Chloroform-*d*) of **3e**



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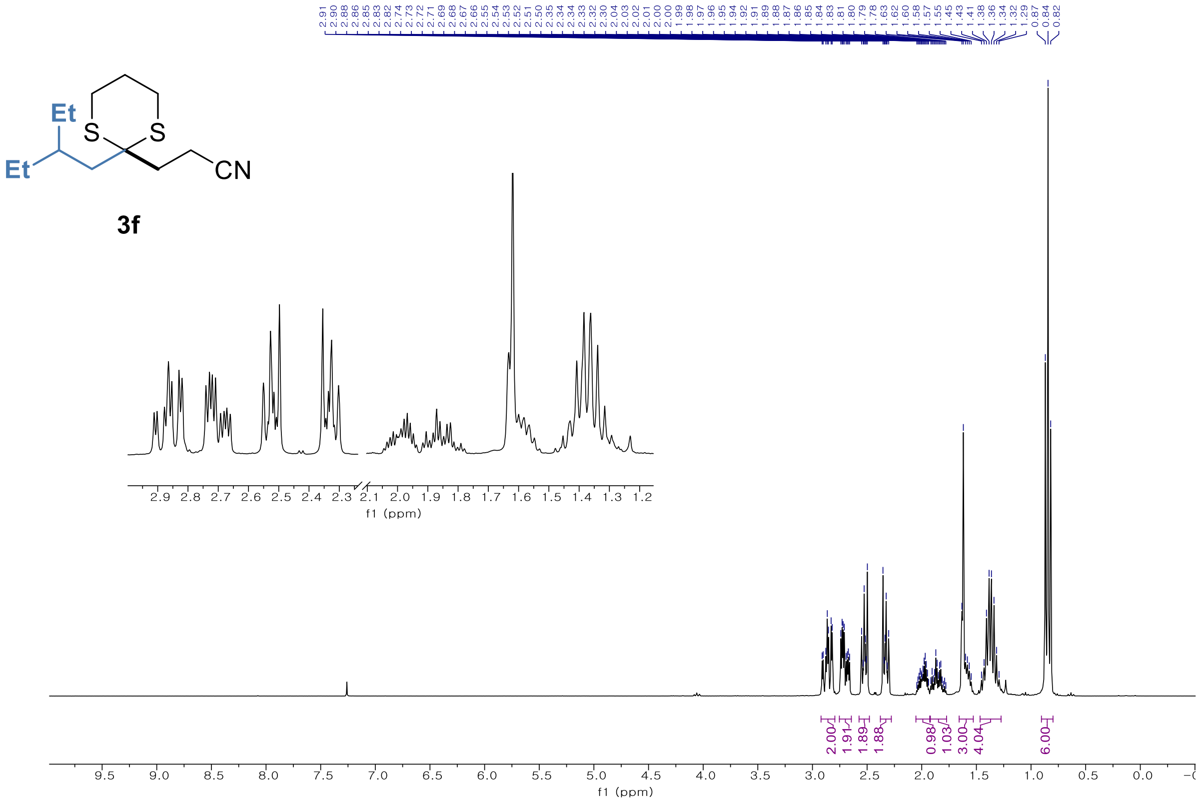
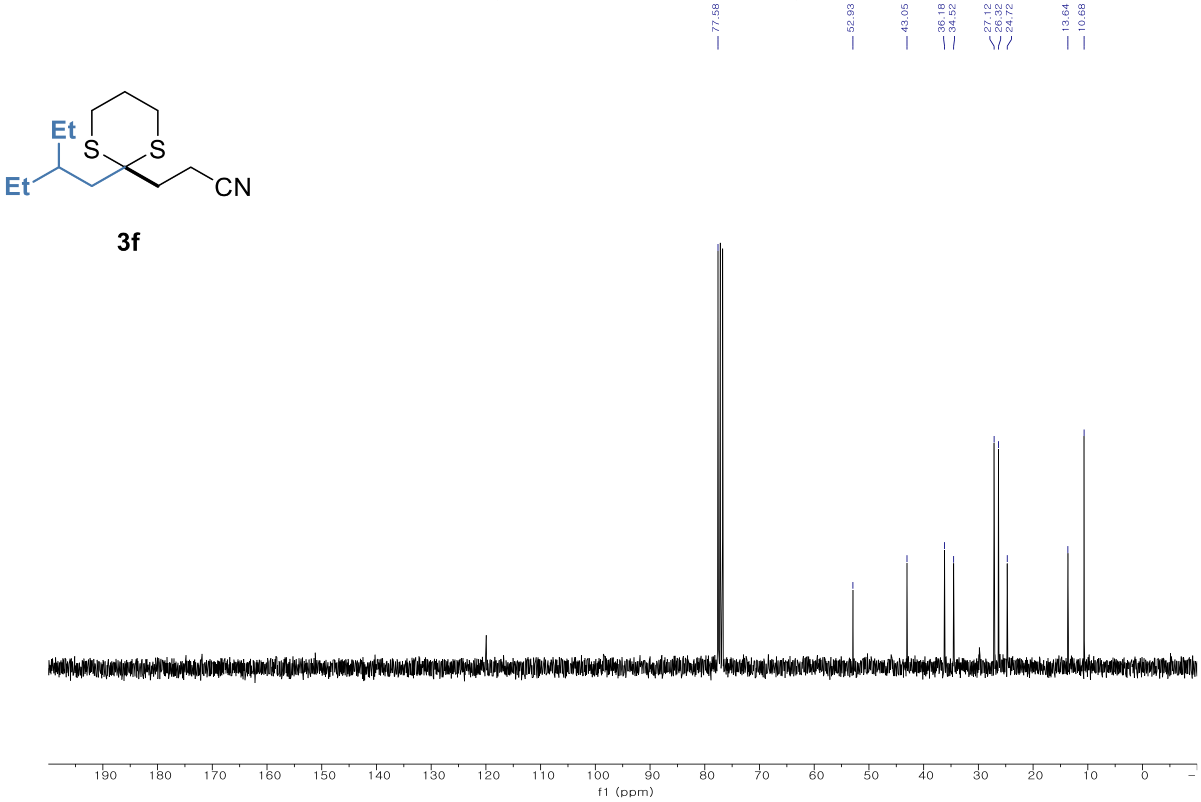


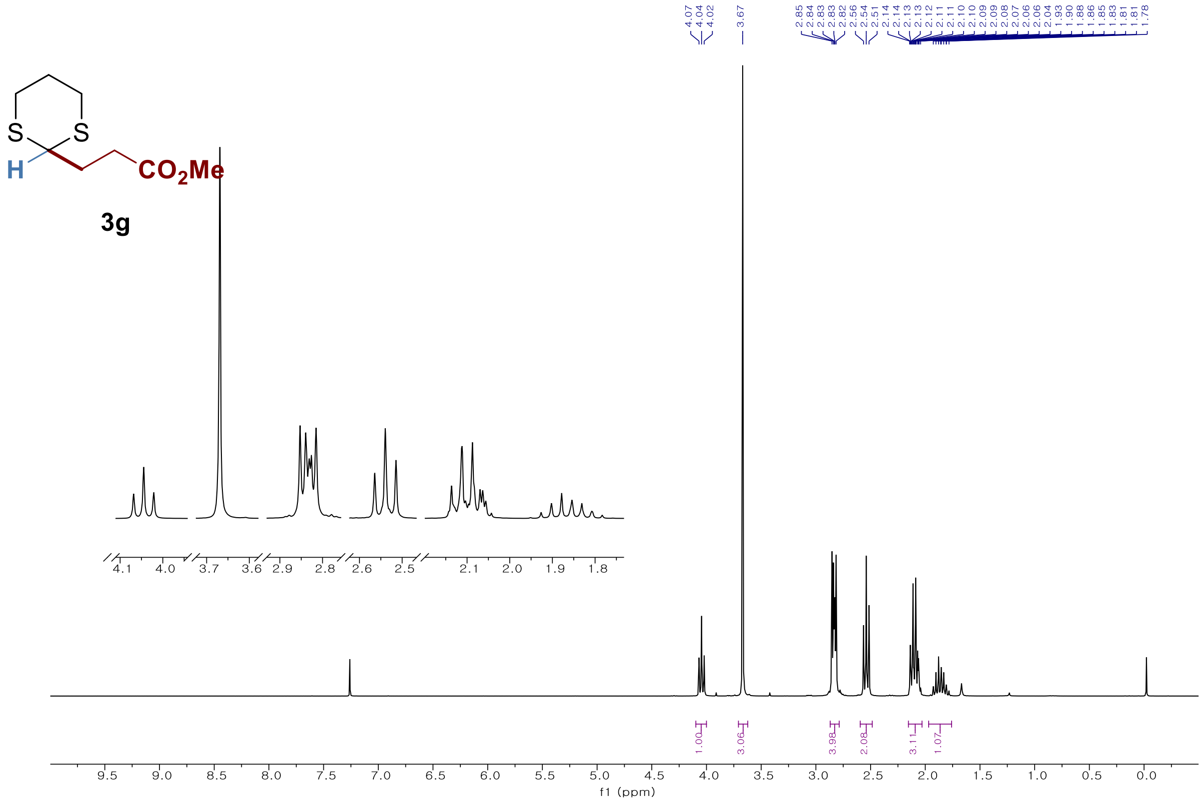
3f

^{1}H-NMR (300 MHz, Chloroform-*d*) of **3f**



3f

^{13}C-NMR (75 Hz, Chloroform-*d*) of **3f**



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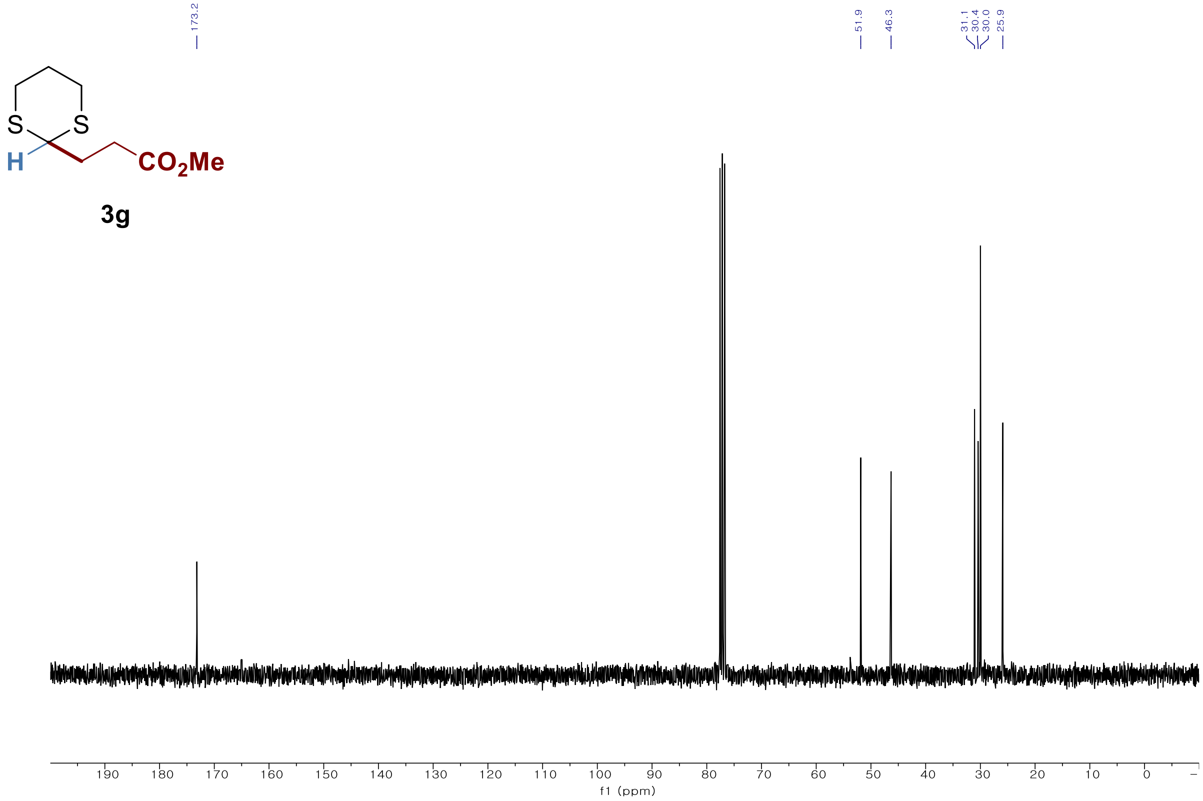

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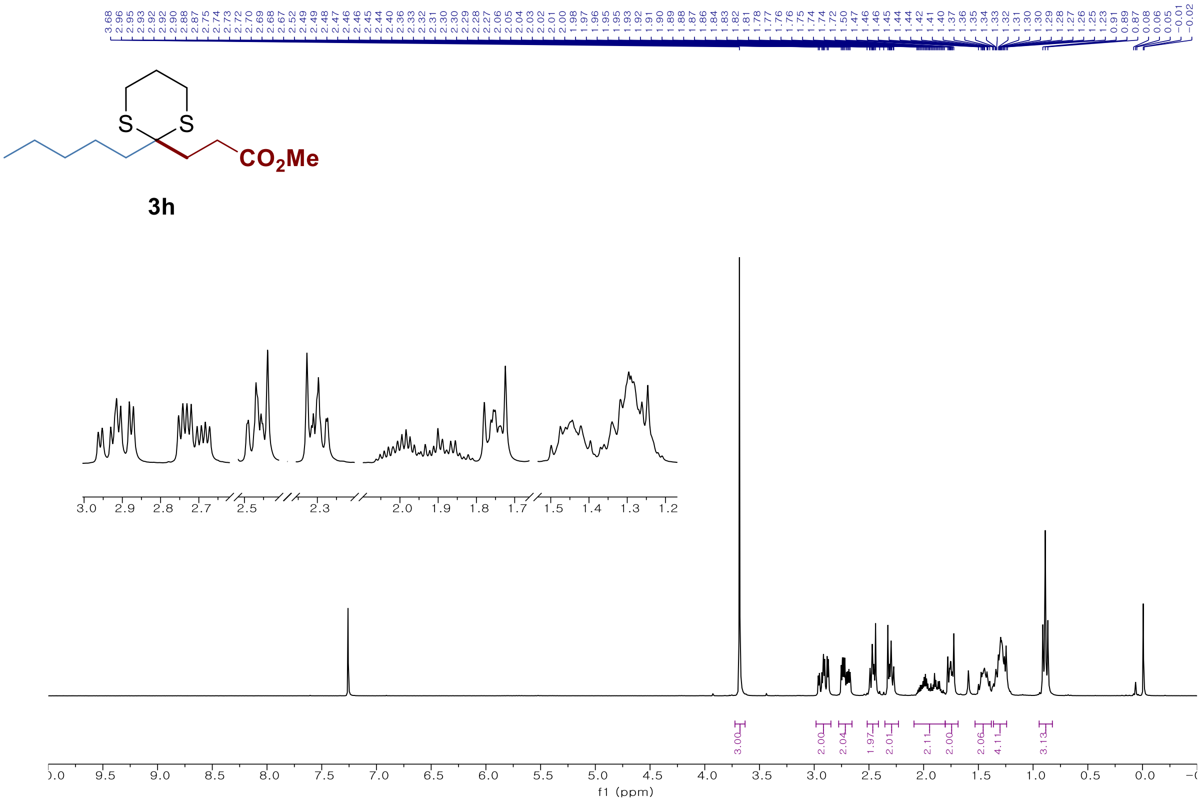

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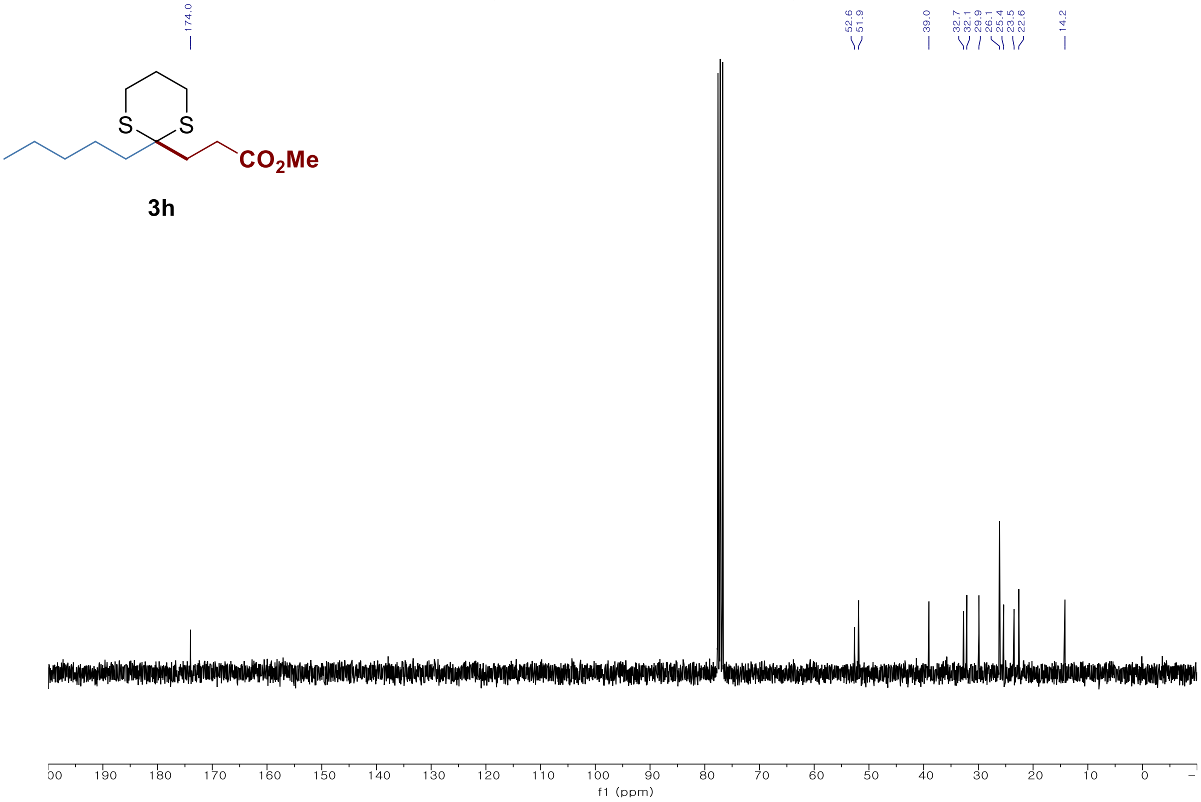
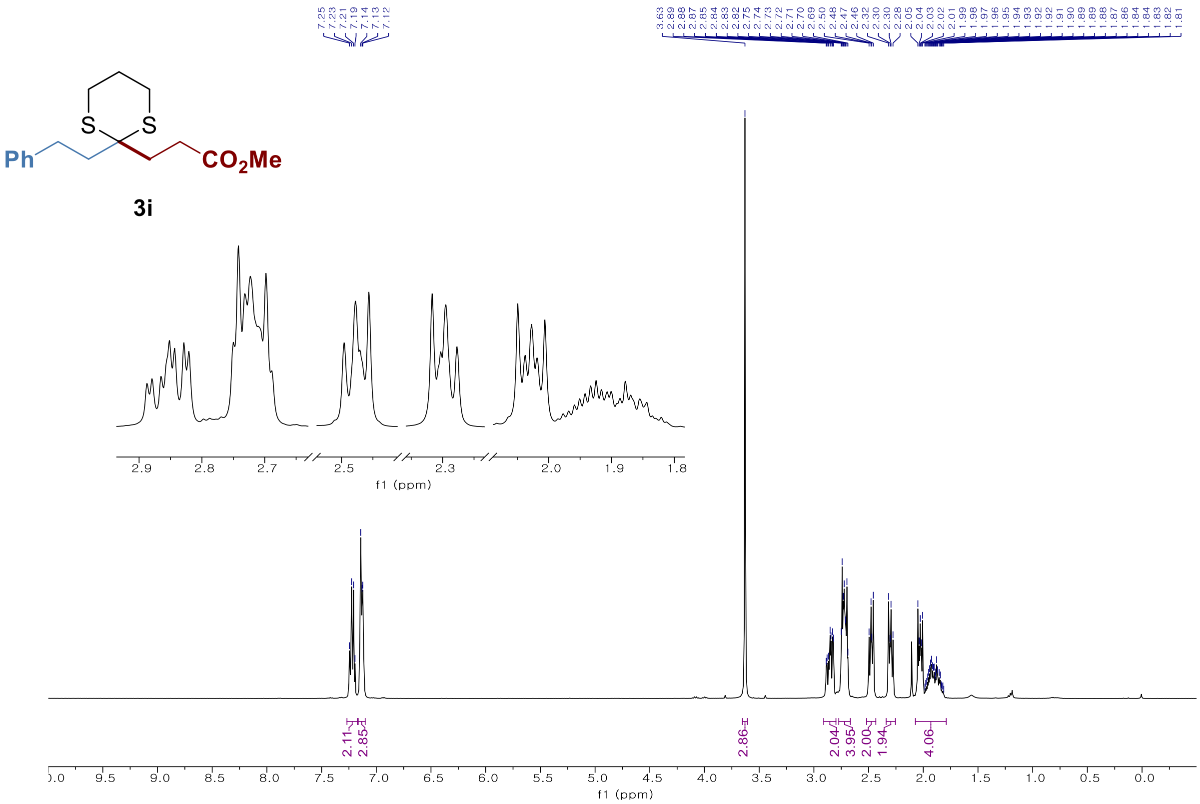
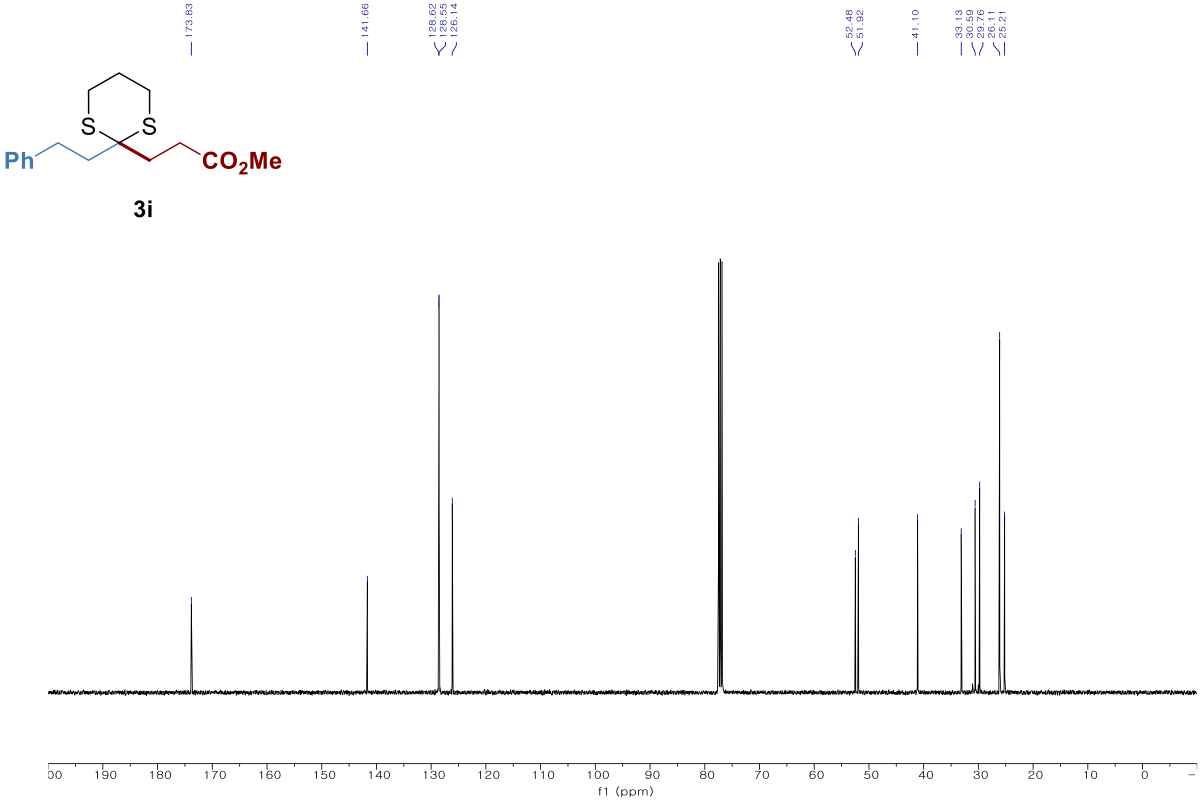


^{13}C-NMR (75 Hz, Chloroform-*d*) of **3h**



3i

^{1}H-NMR (400 MHz, Chloroform-*d*) of **3i**



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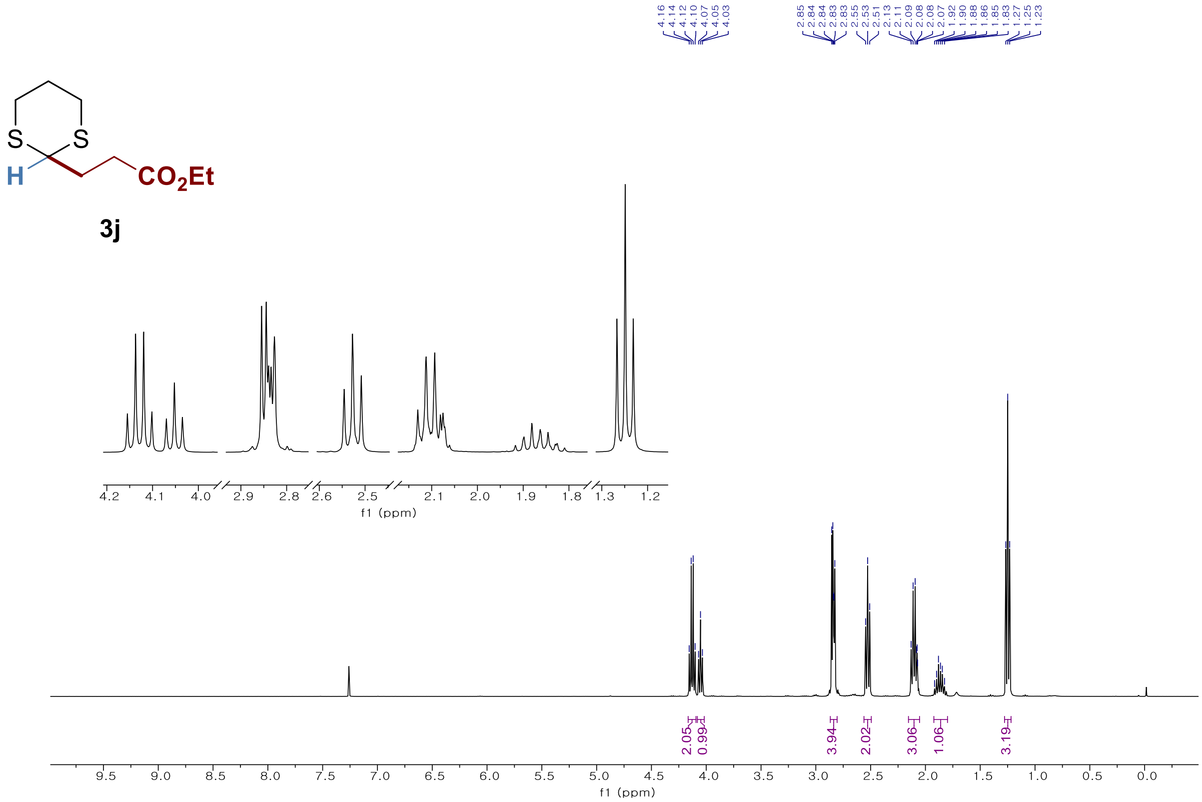
^{13}C-NMR (101 Hz, Chloroform-*d*) of **3i**



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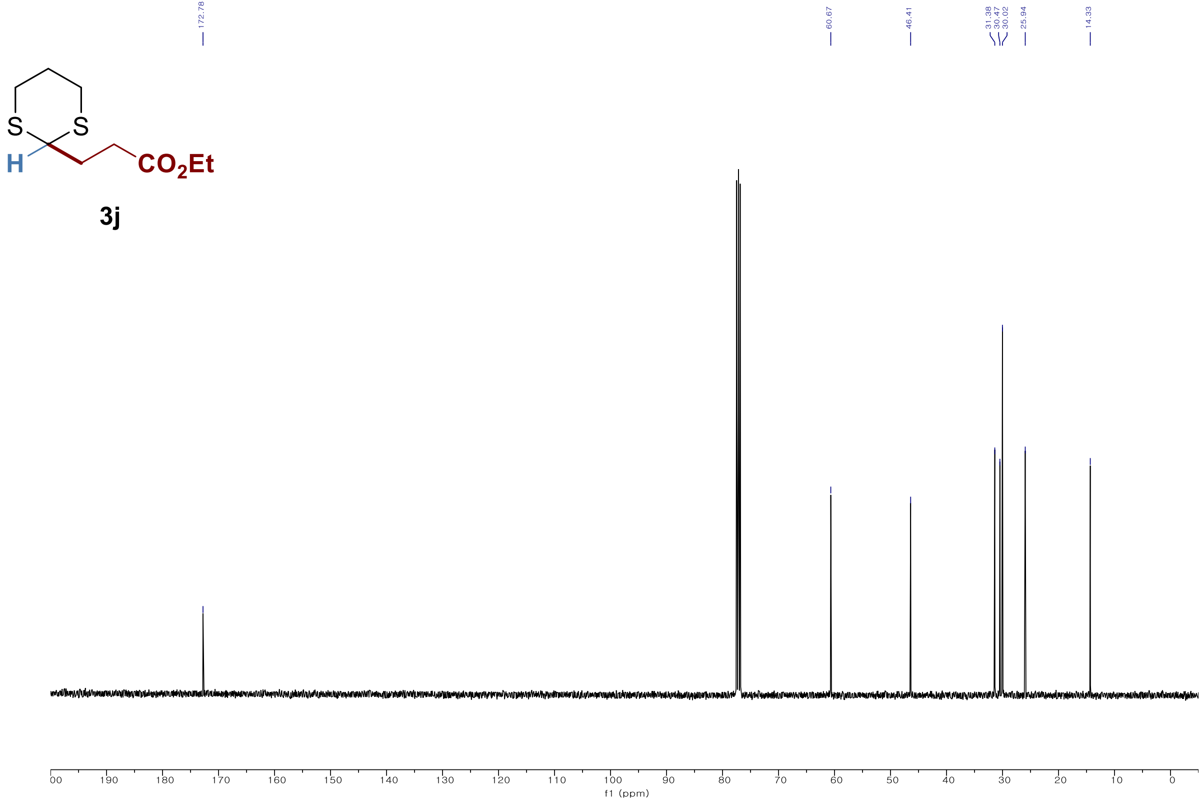

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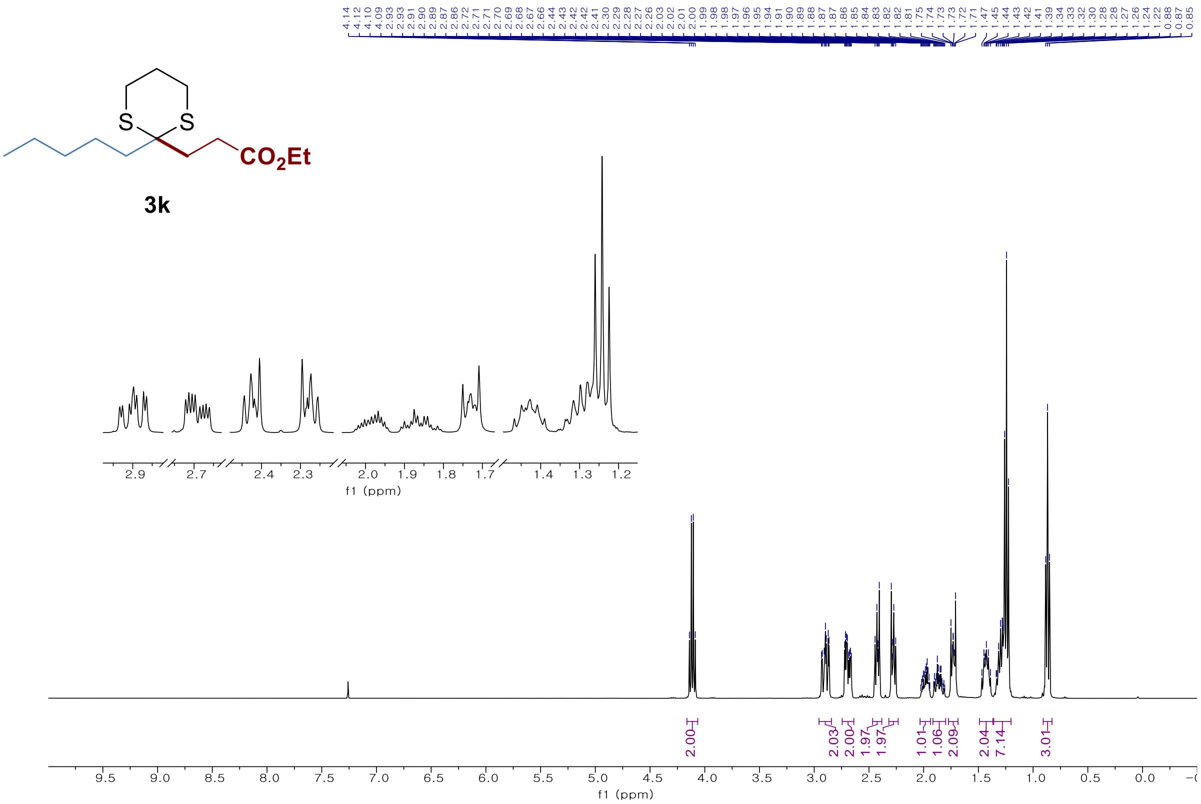

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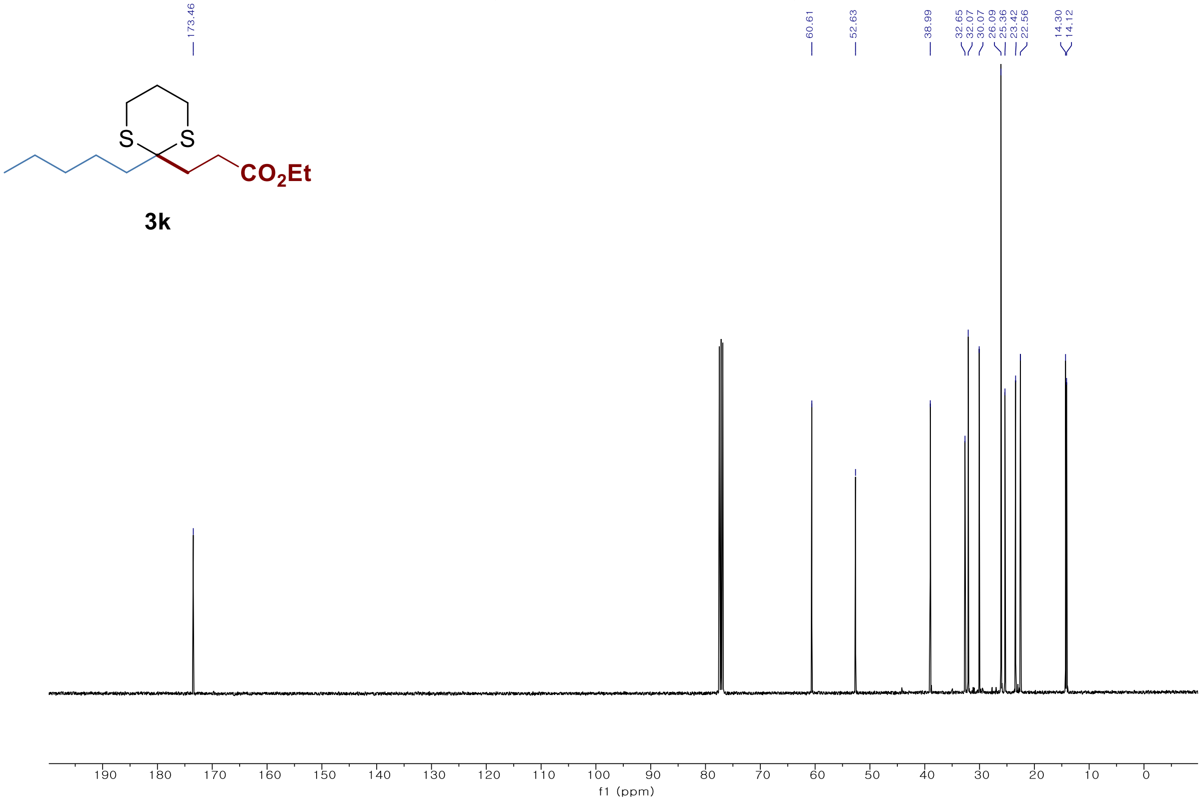

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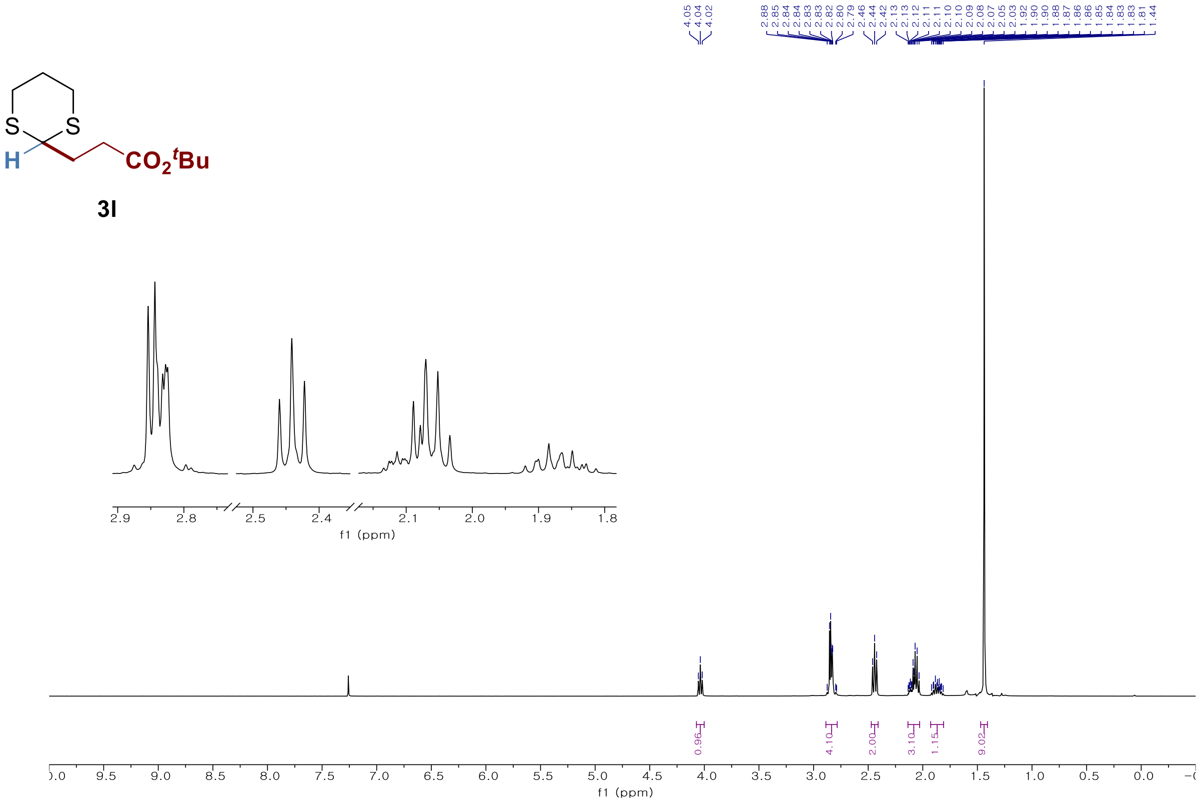
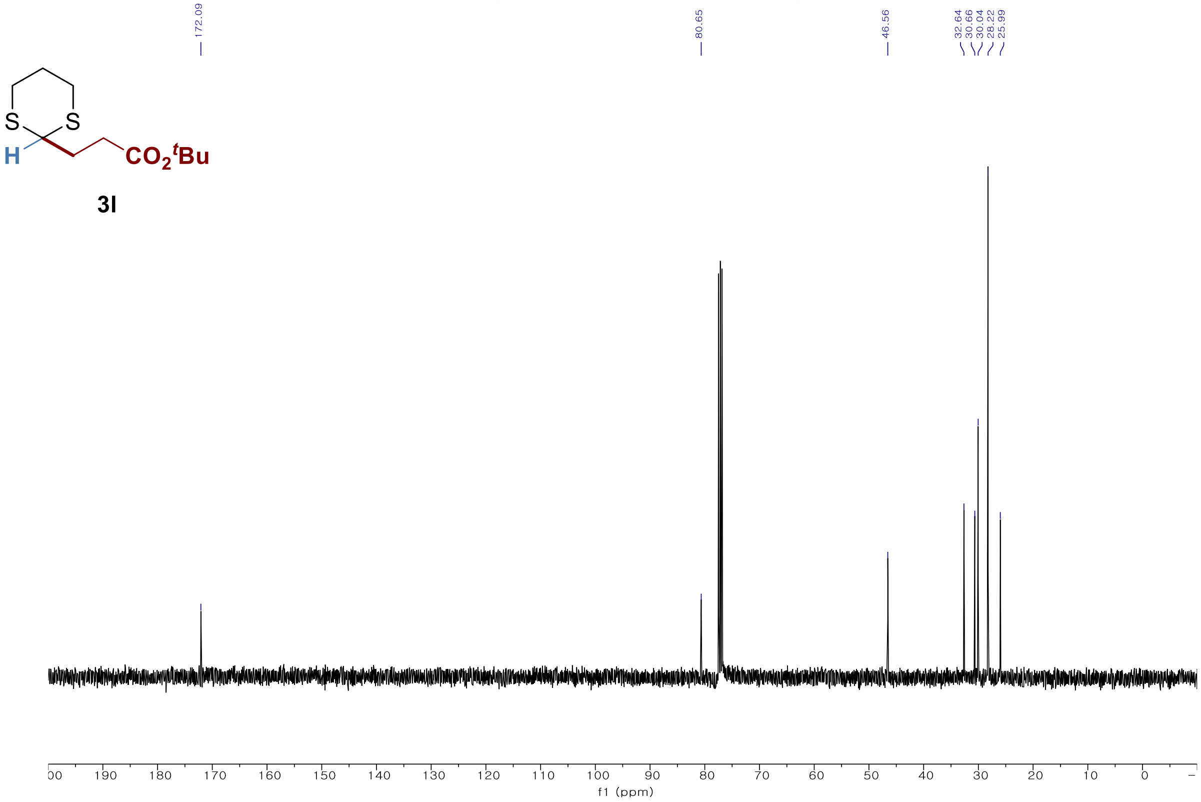


^{1}H-NMR (400 MHz, Chloroform-*d*) of **3l**



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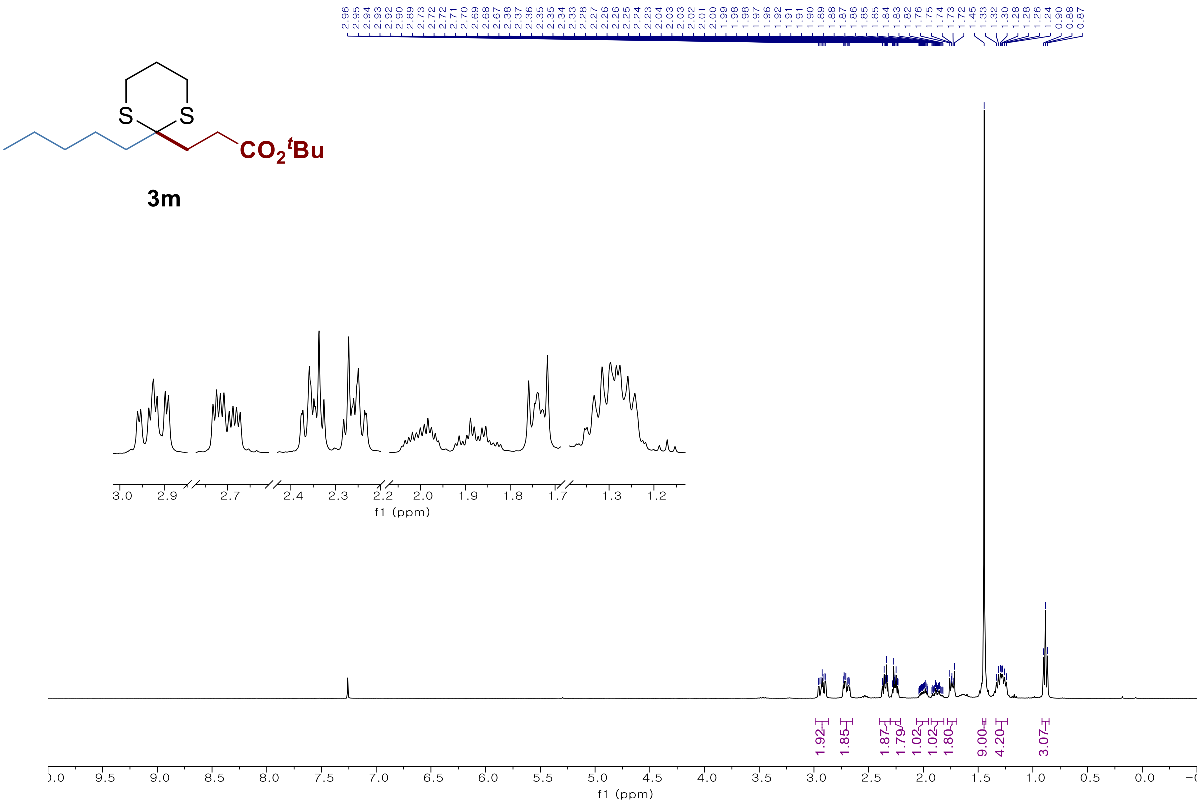
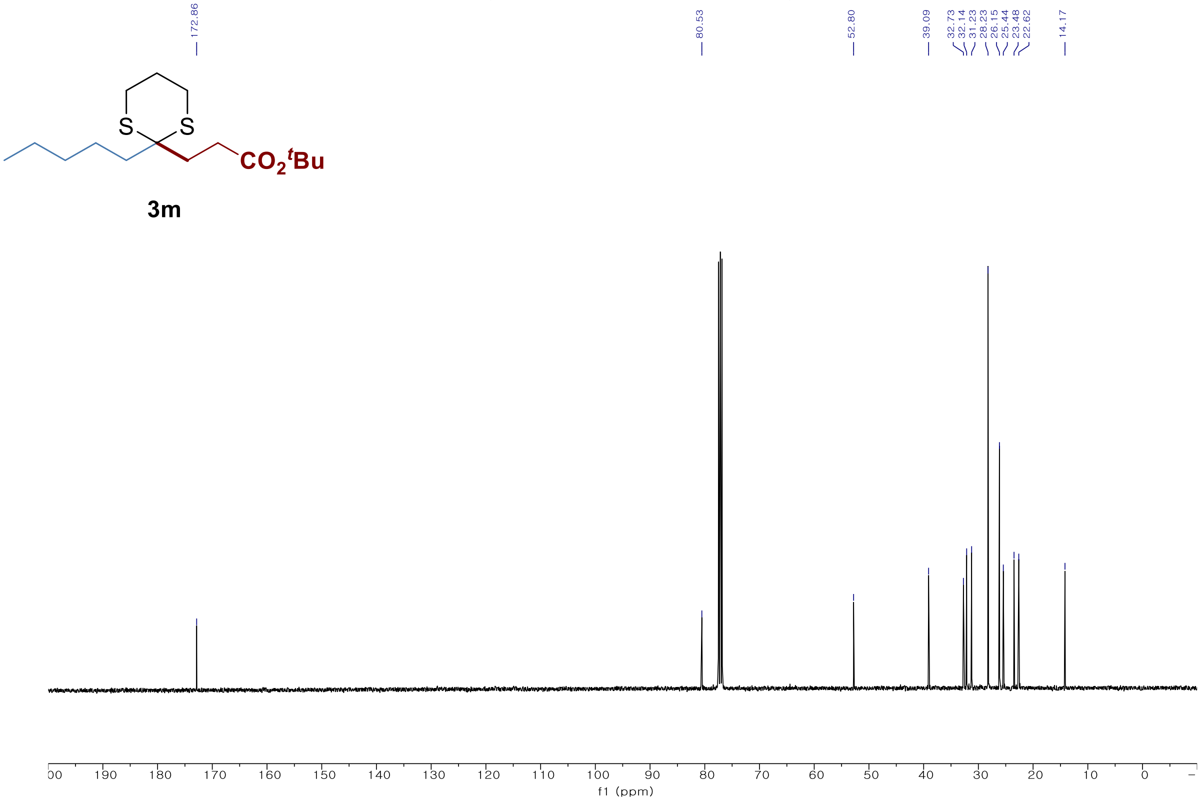


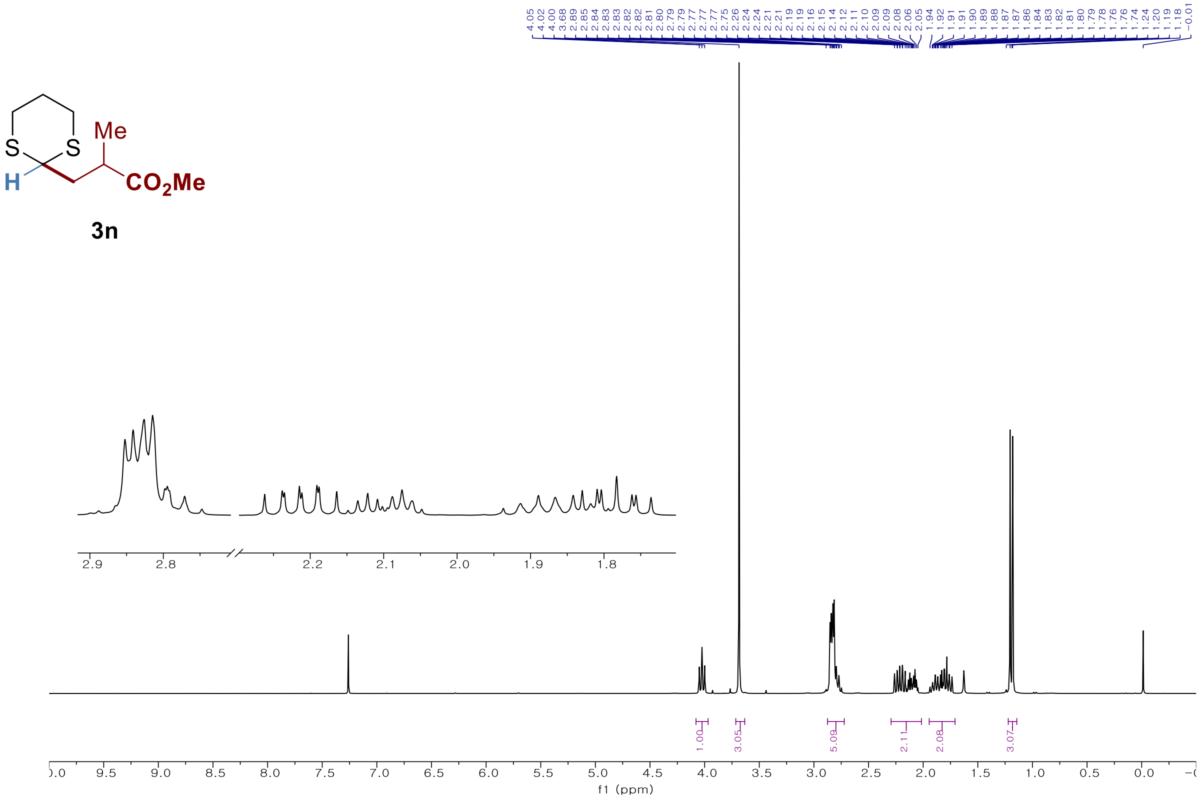
3m

^{1}H-NMR (400 MHz, Chloroform-*d*) of **3m**



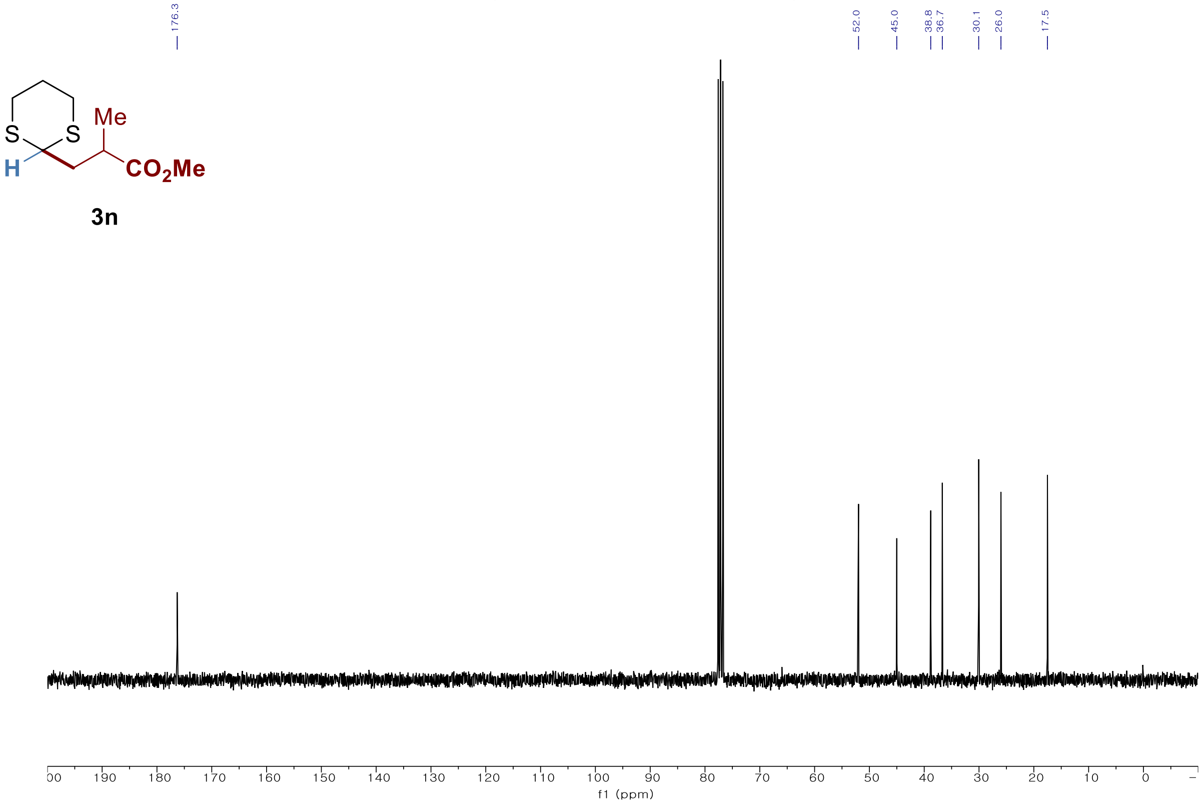
3m

^{13}C-NMR (101 Hz, Chloroform-*d*) of **3m**



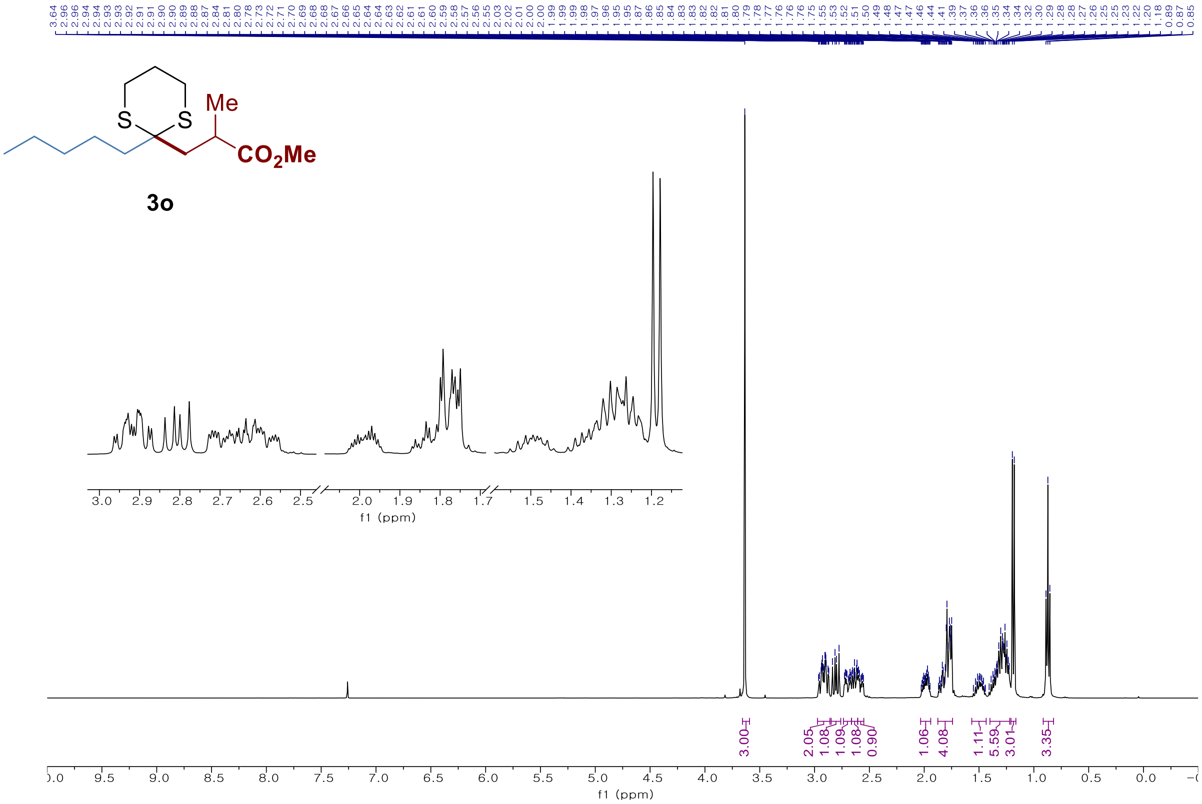
3n

^{1}H-NMR (300 MHz, Chloroform-*d*) of **3n**



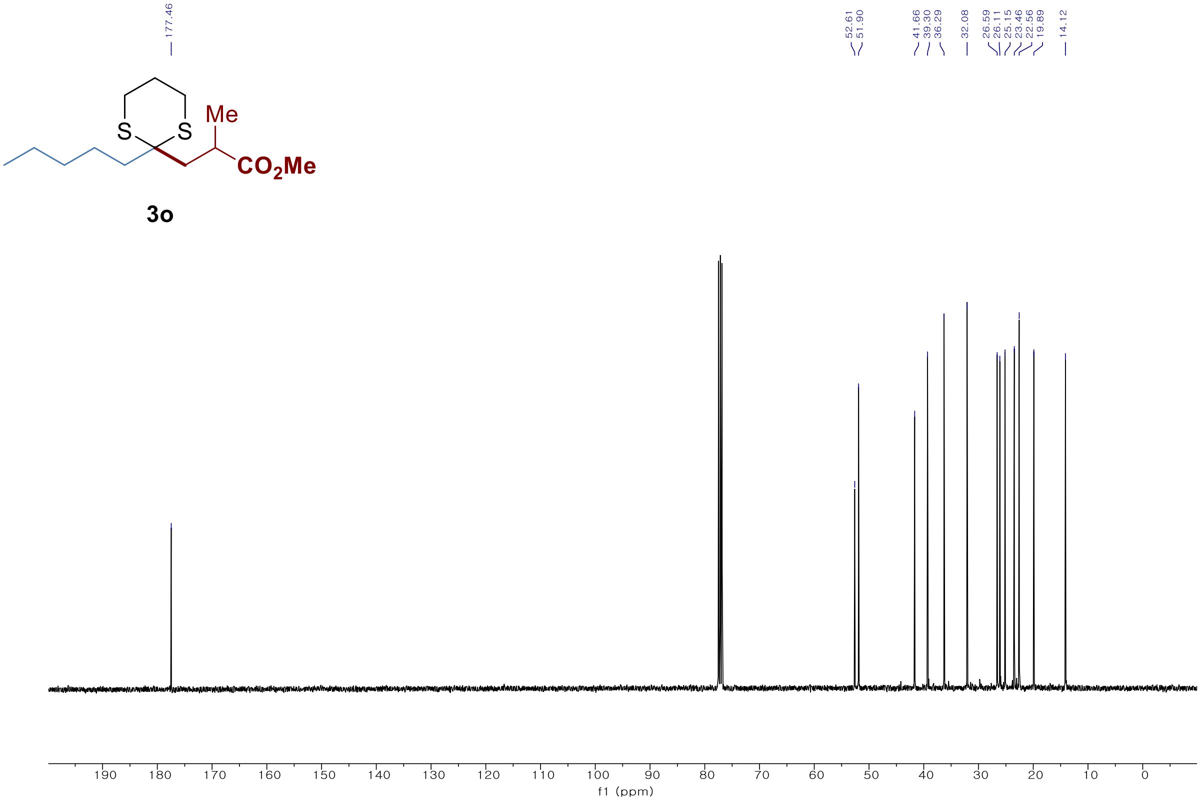
3n

^{13}C-NMR (75 Hz, Chloroform-*d*) of **3n**



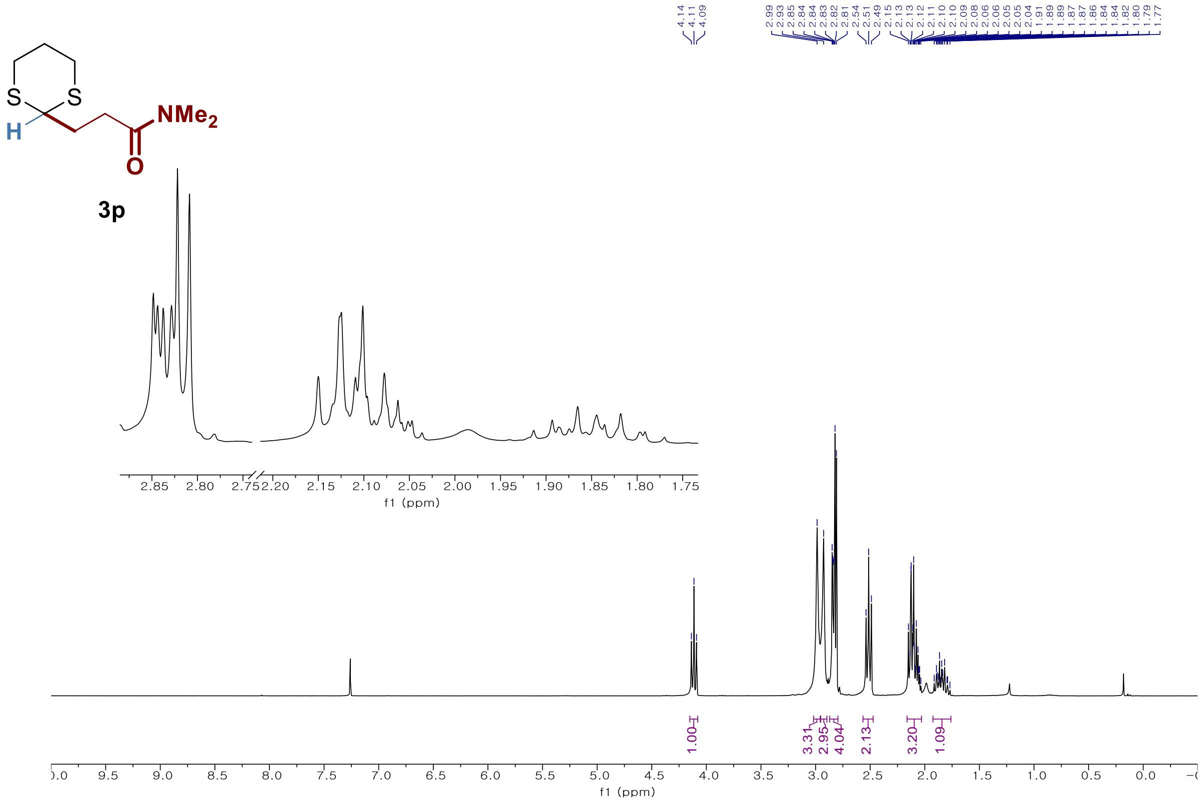
3o

^{1}H-NMR (400 MHz, Chloroform-*d*) of **3o**

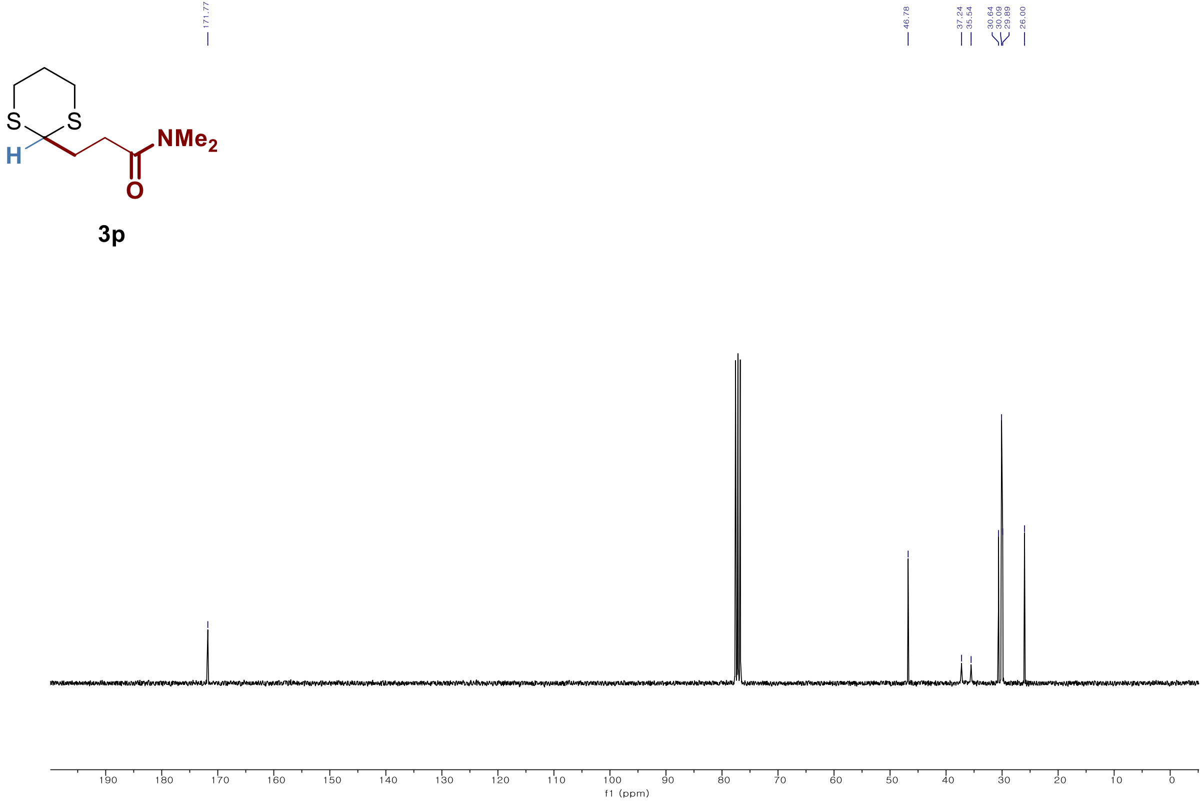


3o

^{13}C-NMR (101 Hz, Chloroform-*d*) of **3o**



^{1}H-NMR (300 MHz, Chloroform-*d*) of **3p**



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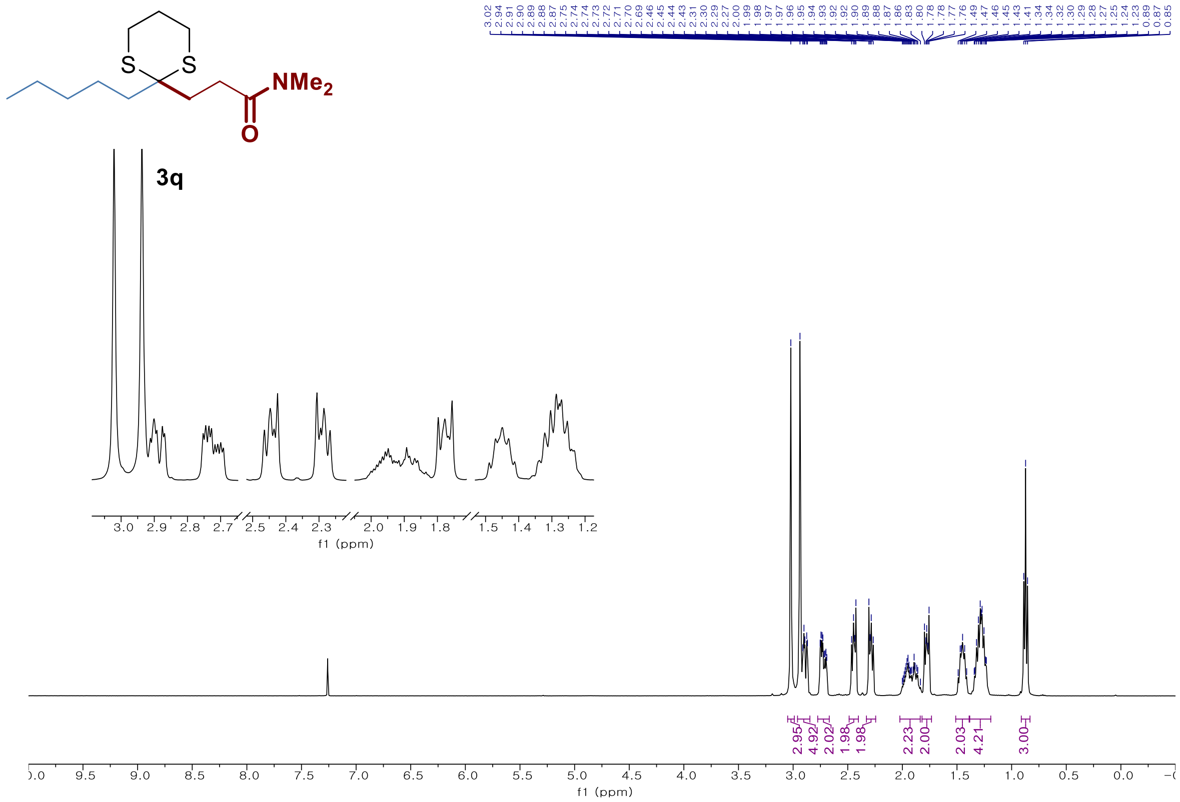
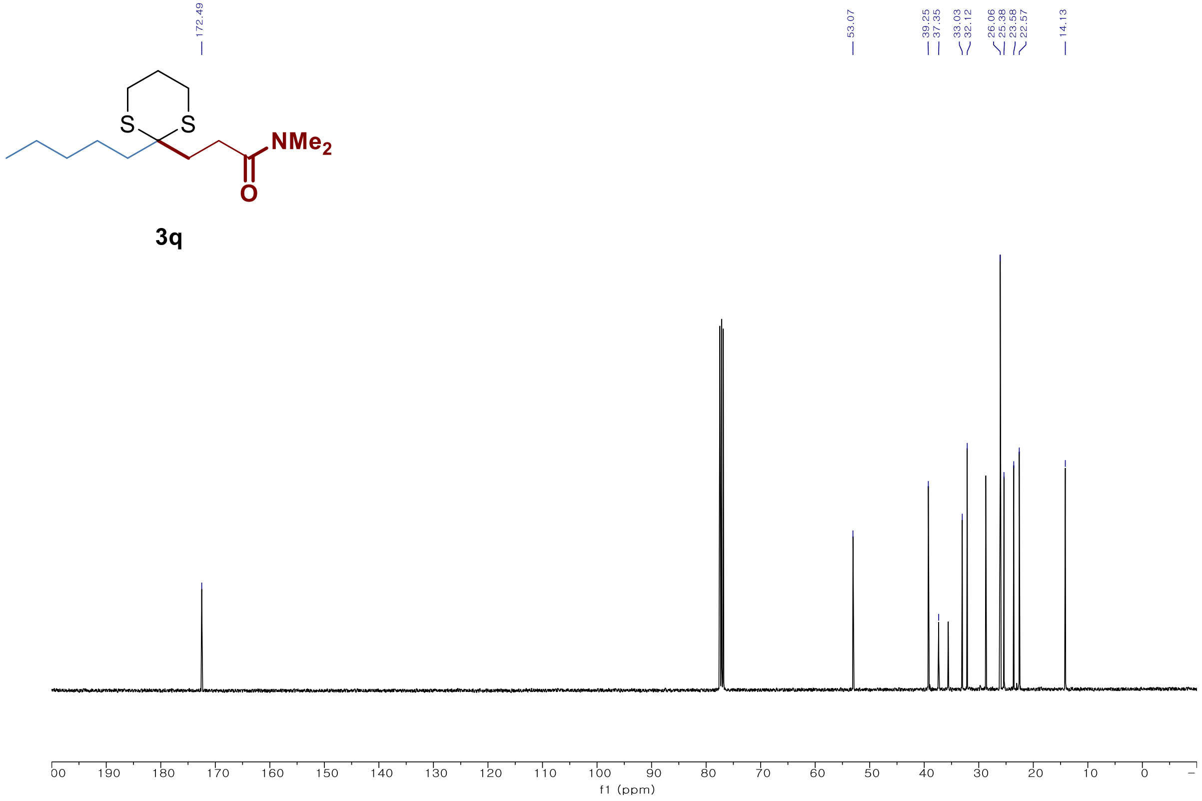
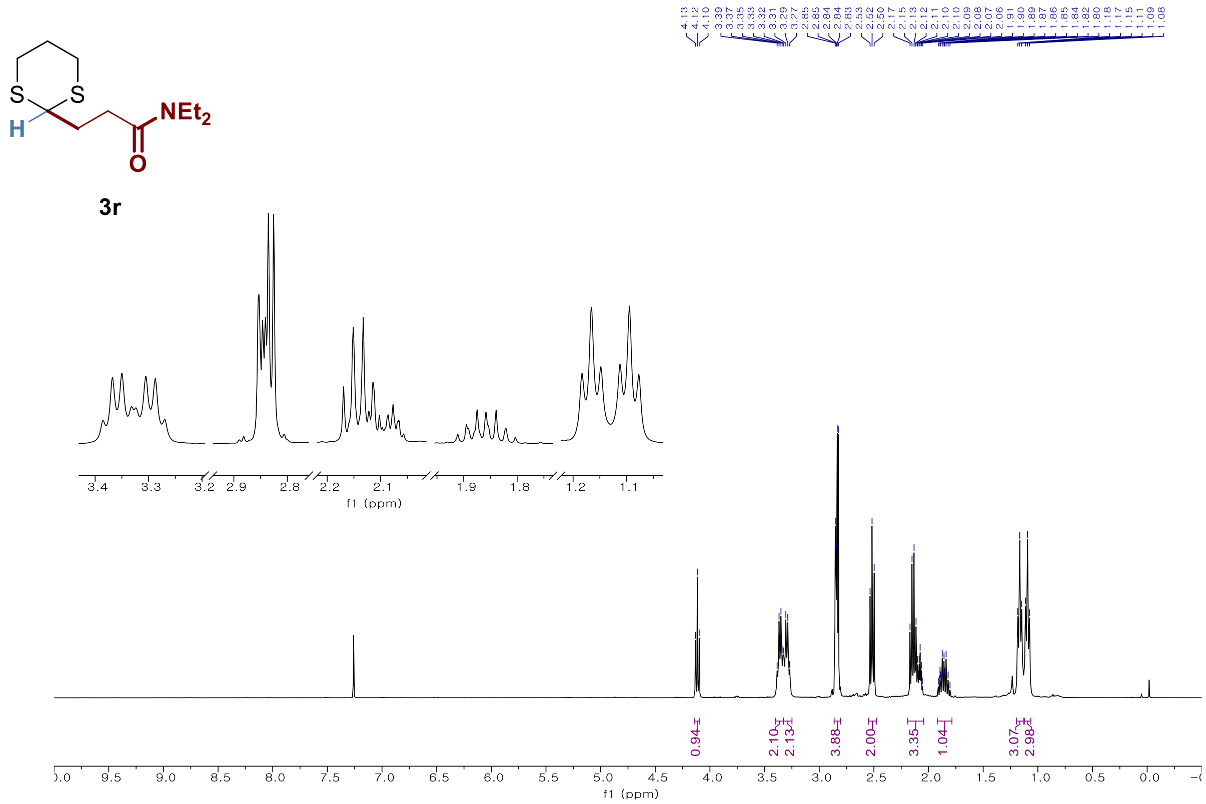


^{1}H-NMR (400 MHz, Chloroform-*d*) of **3q**

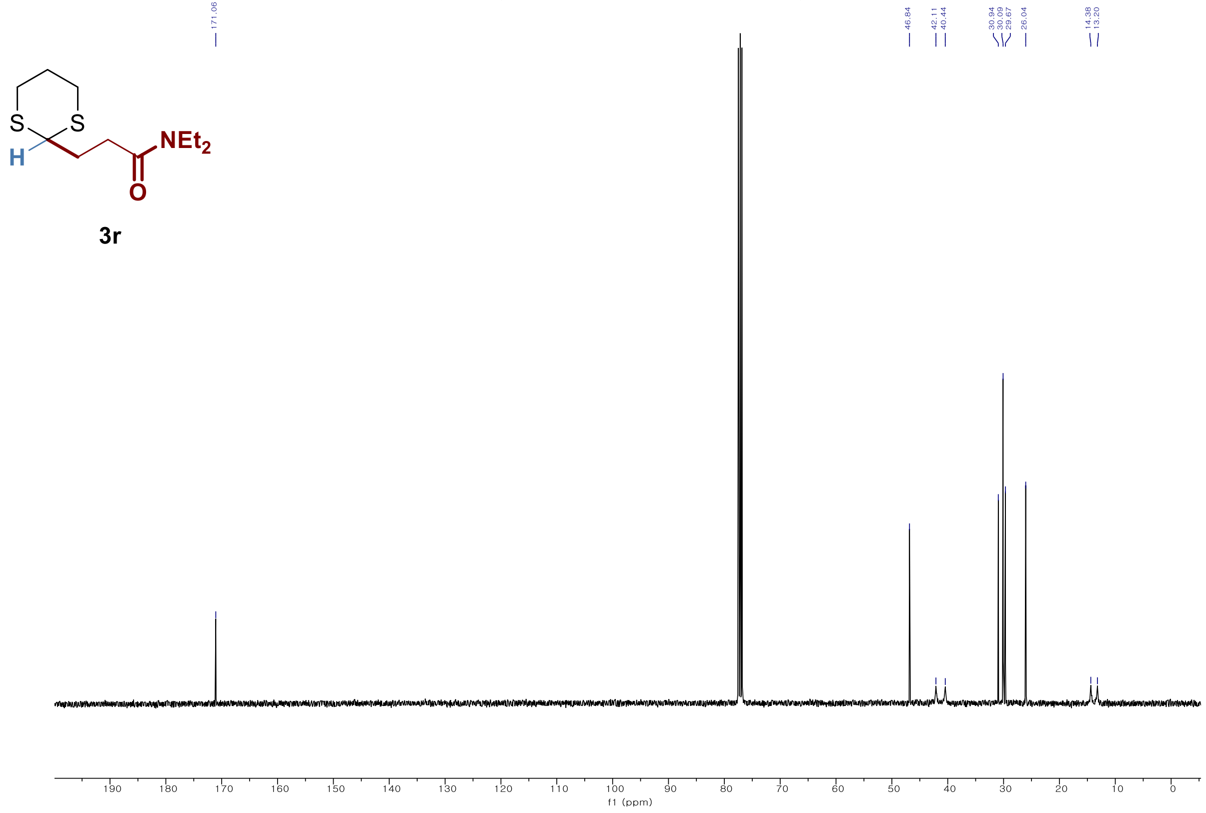


^{13}C-NMR (101 Hz, Chloroform-*d*) of **3q**



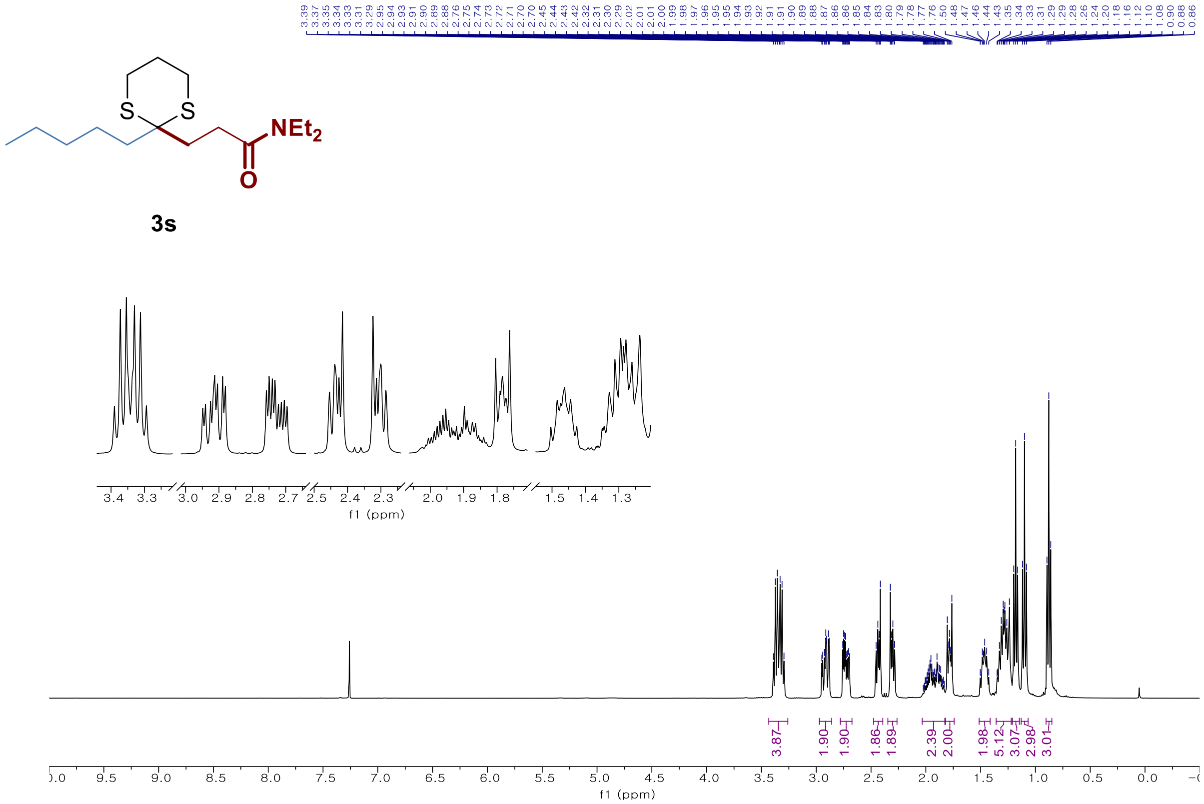
3r

^{1}H-NMR (400 MHz, Chloroform-*d*) of **3r**

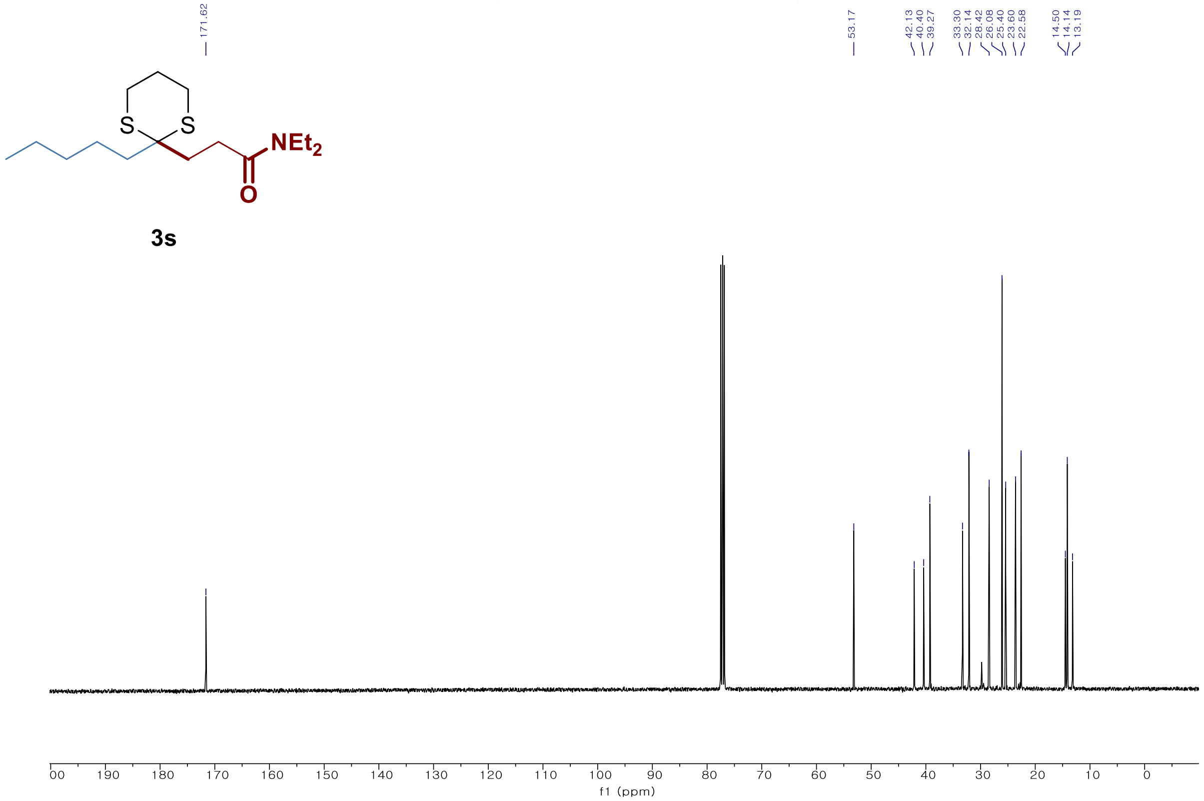


3r

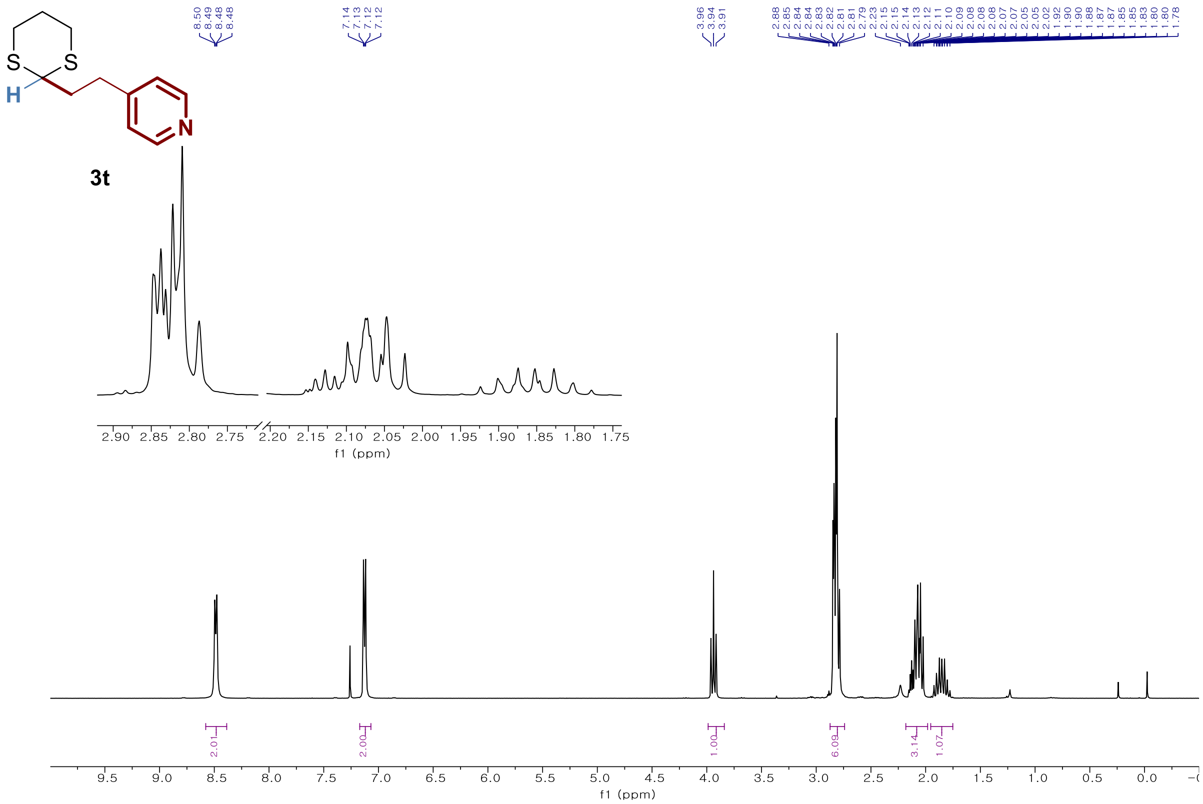
^{13}C-NMR (101 Hz, Chloroform-*d*) of **3r**



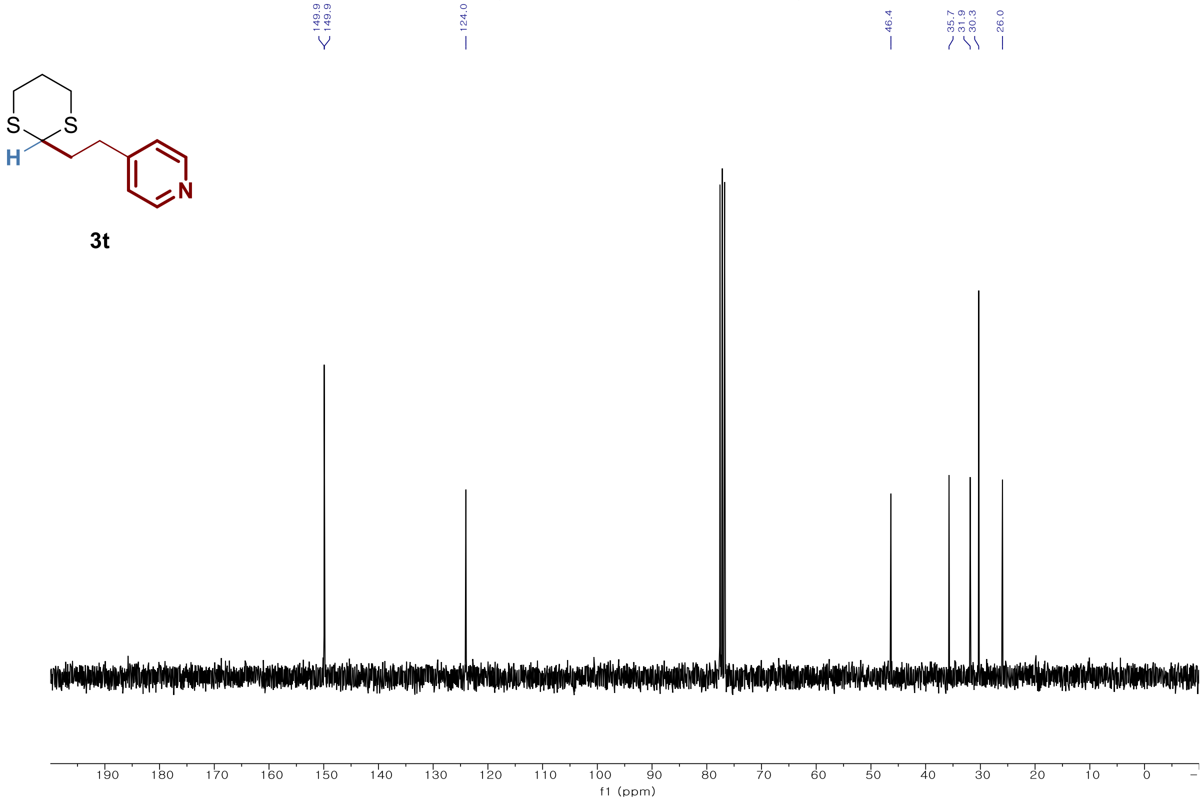
^{1}H-NMR (400 MHz, Chloroform-*d*) of **3s**



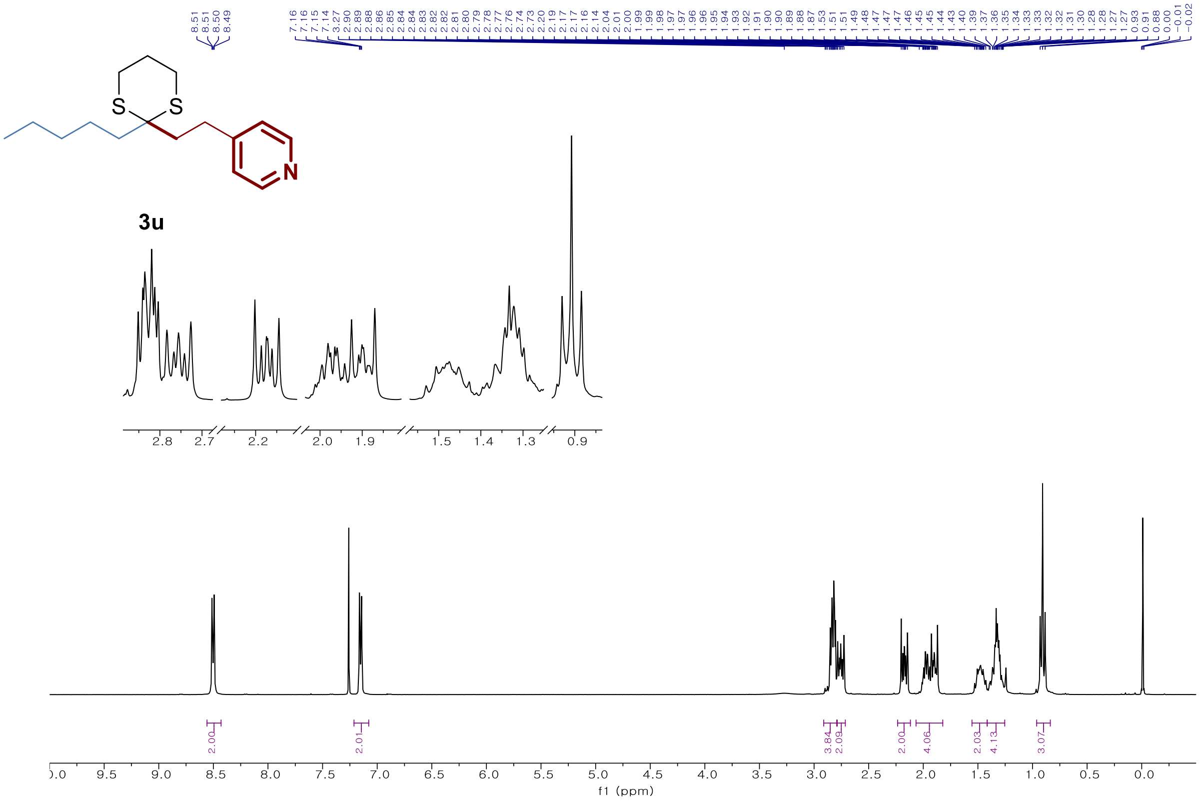
^{13}C-NMR (101 Hz, Chloroform-*d*) of **3s**



^{1}H-NMR (300 MHz, Chloroform-*d*) of **3t**

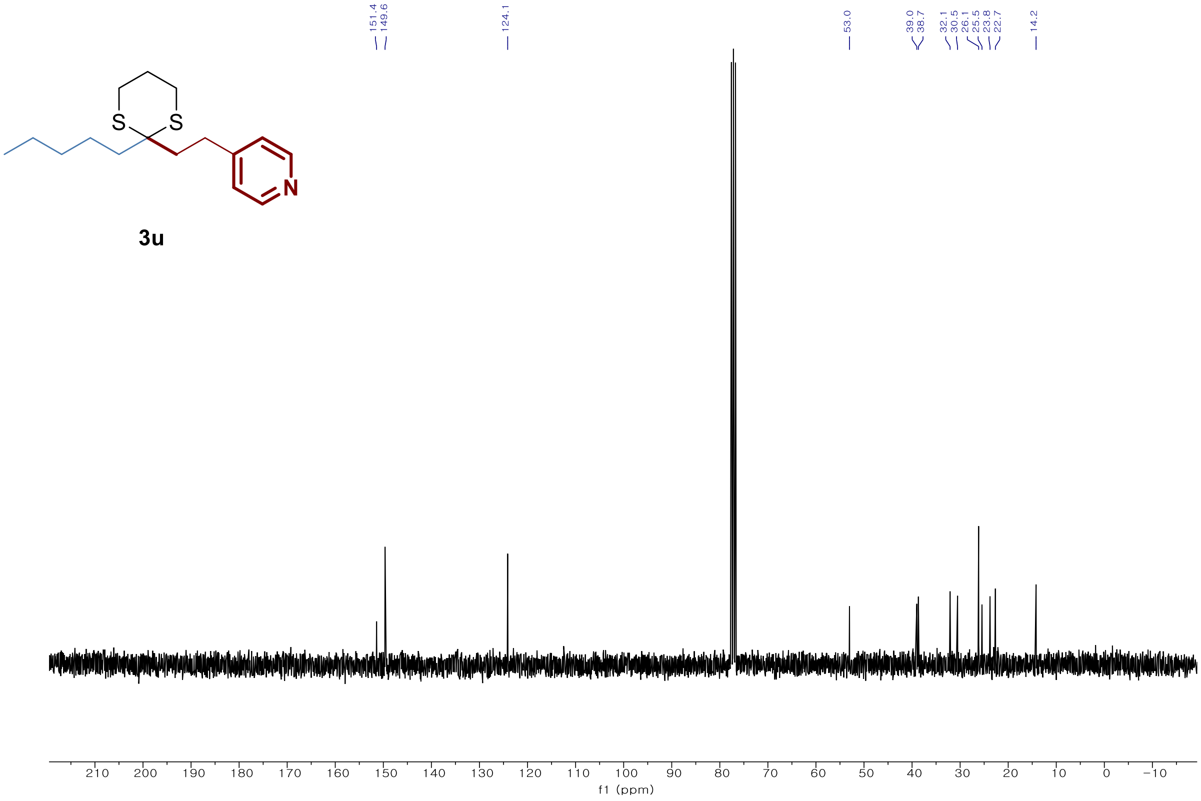


^{13}C-NMR (75 Hz, Chloroform-*d*) of **3t**



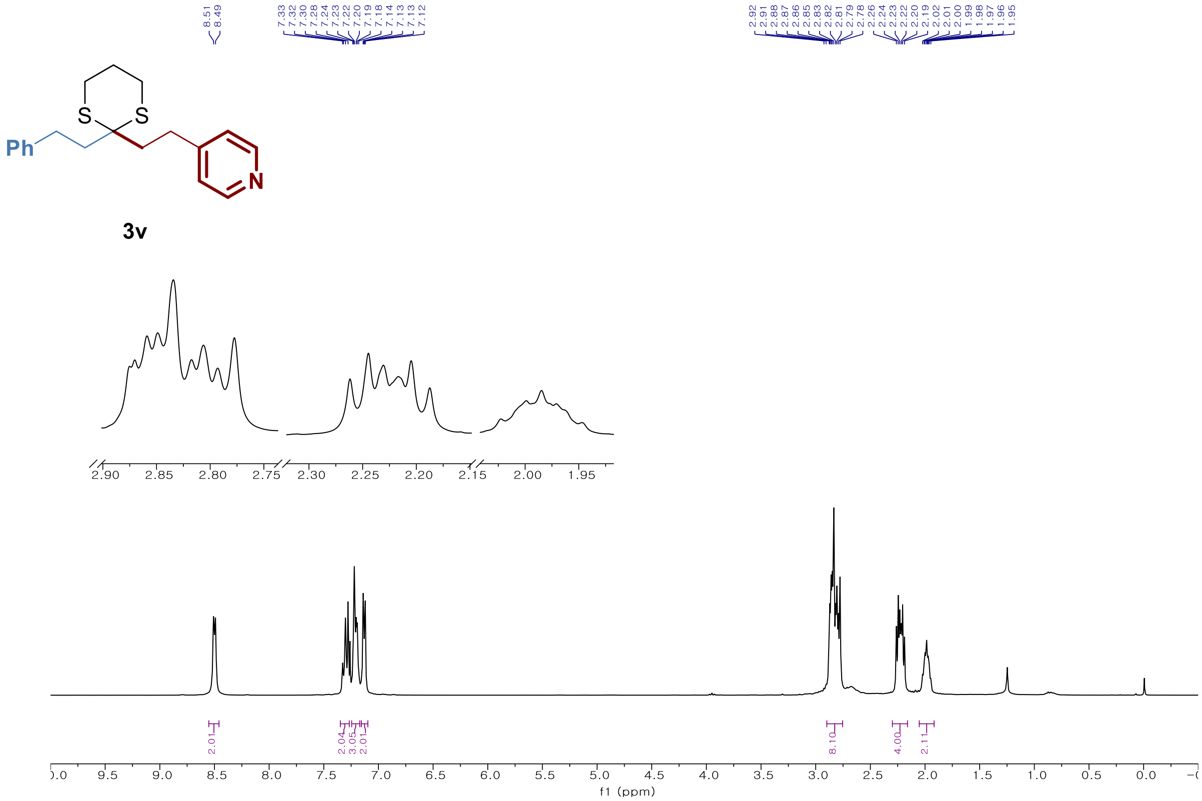
3u

^{1}H-NMR (300 MHz, Chloroform-*d*) of **3u**

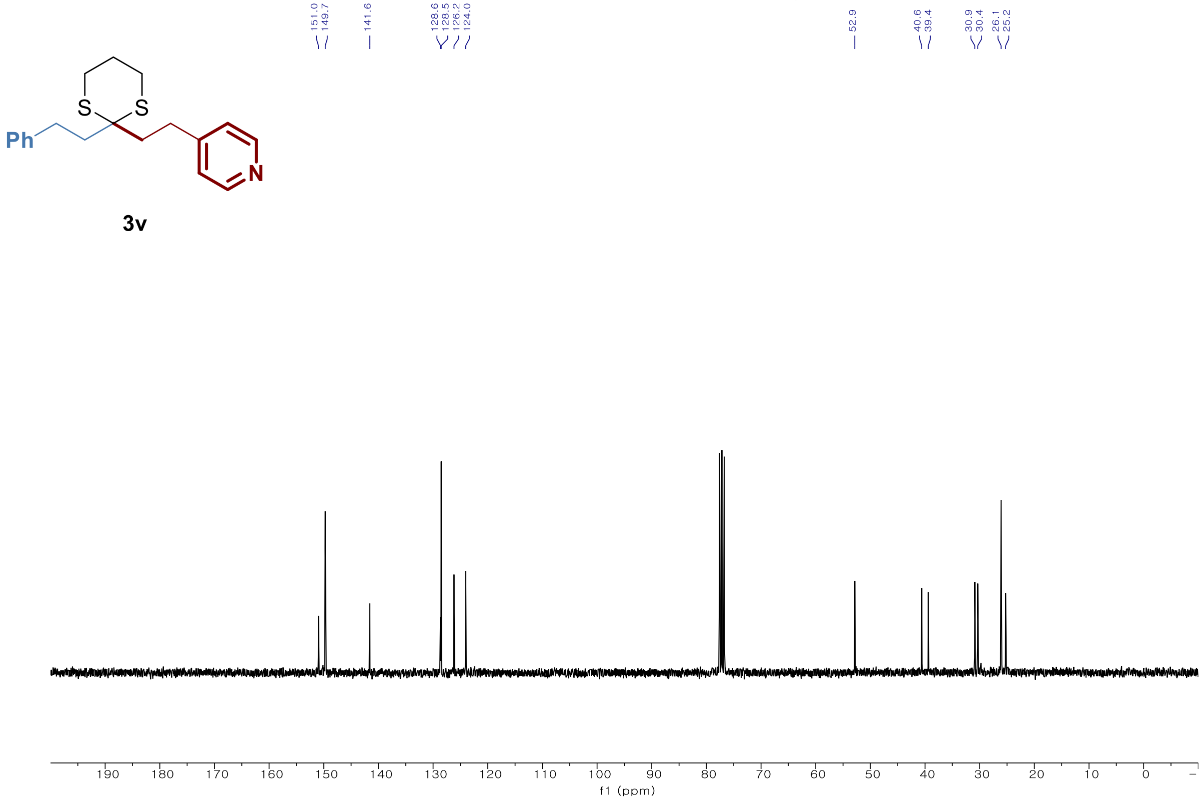


3u

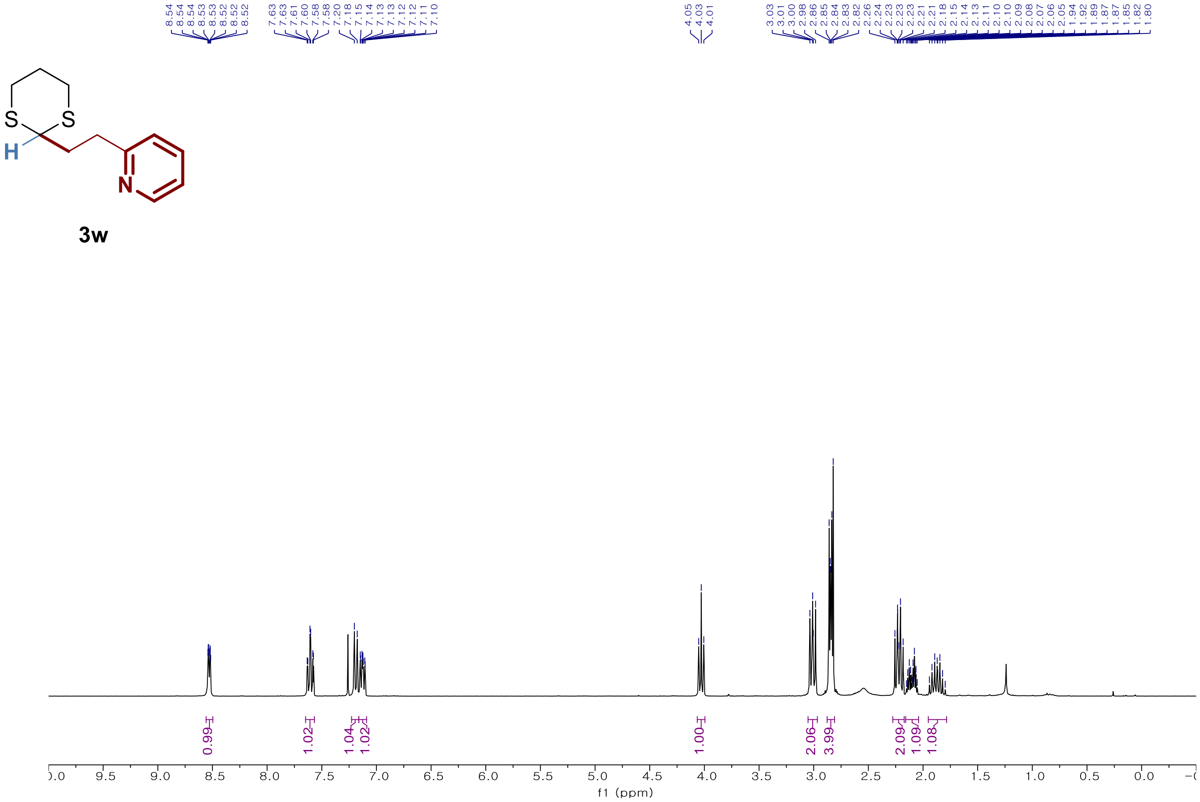
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3u**



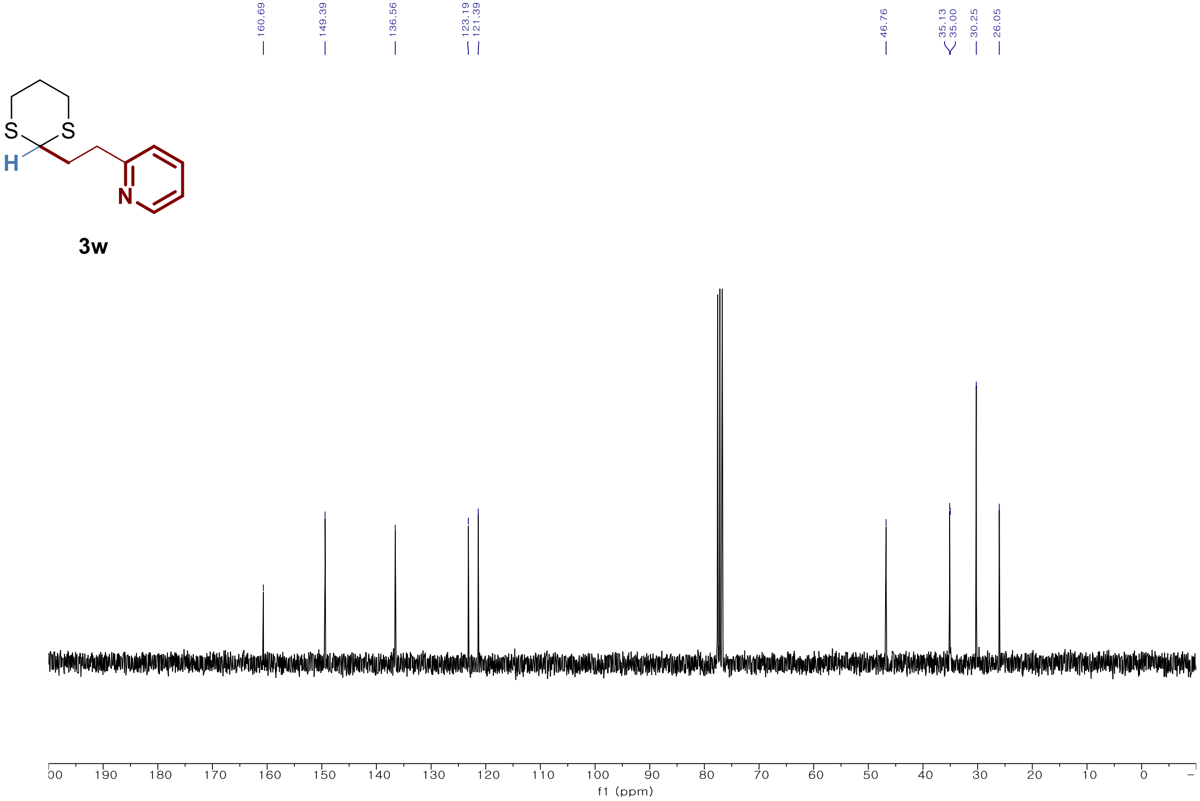
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3v**



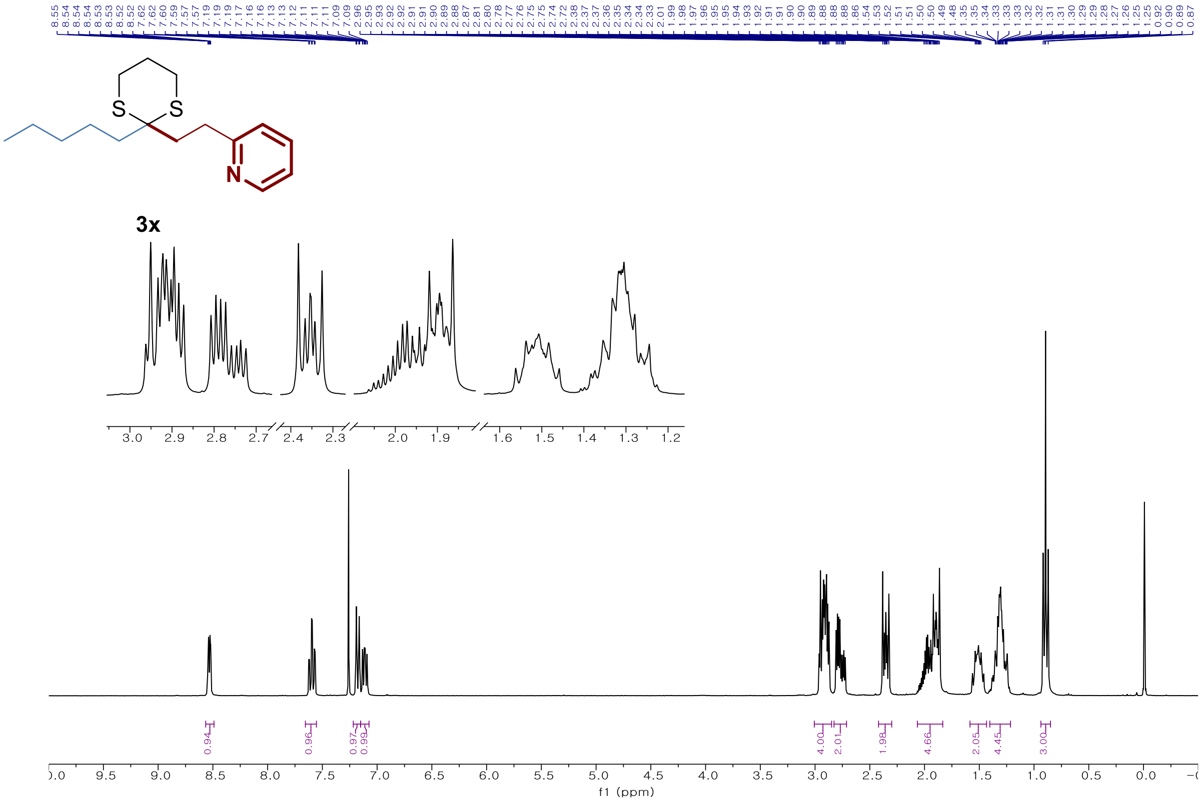
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3v**



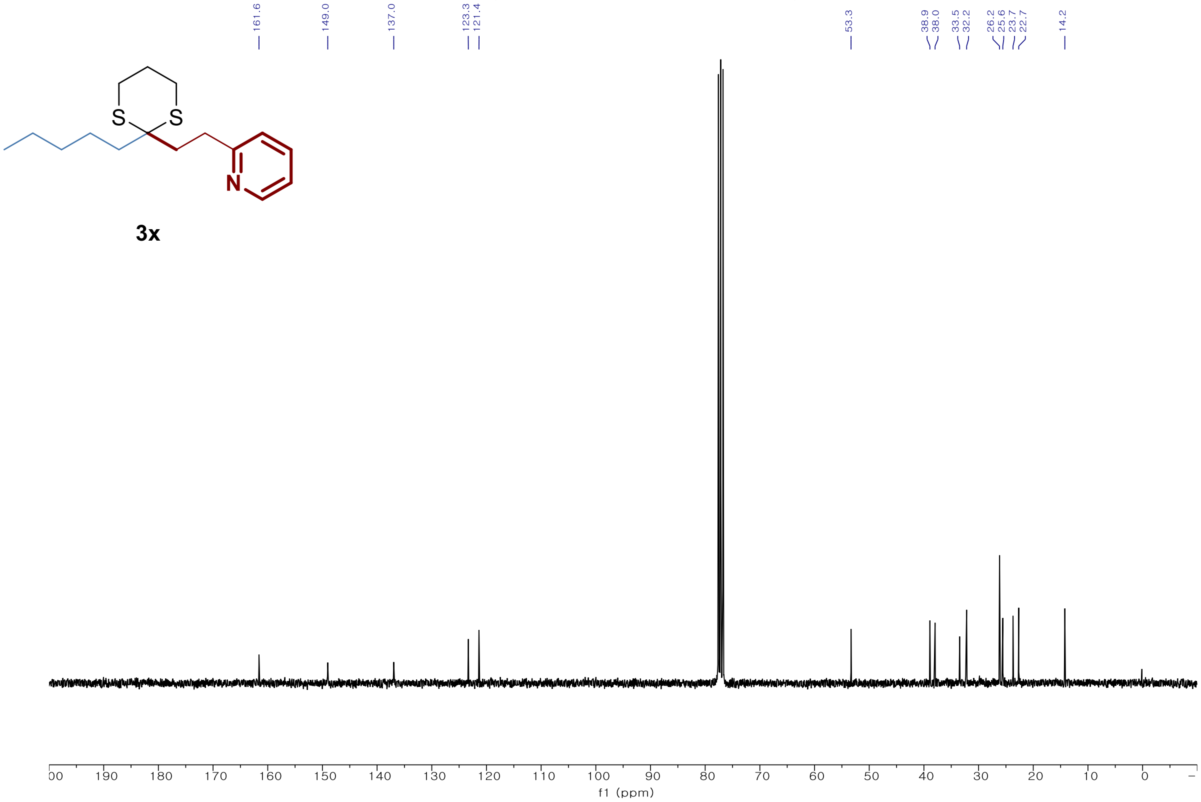
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3w**



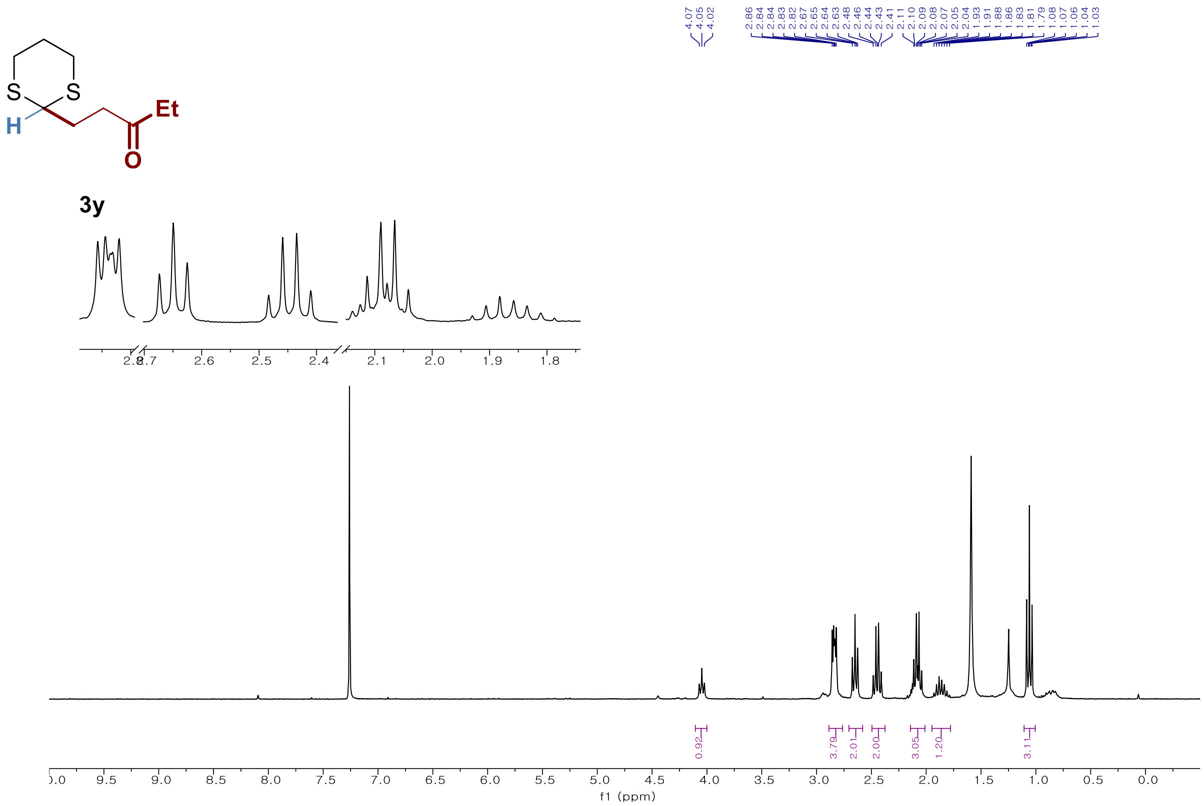
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3w**



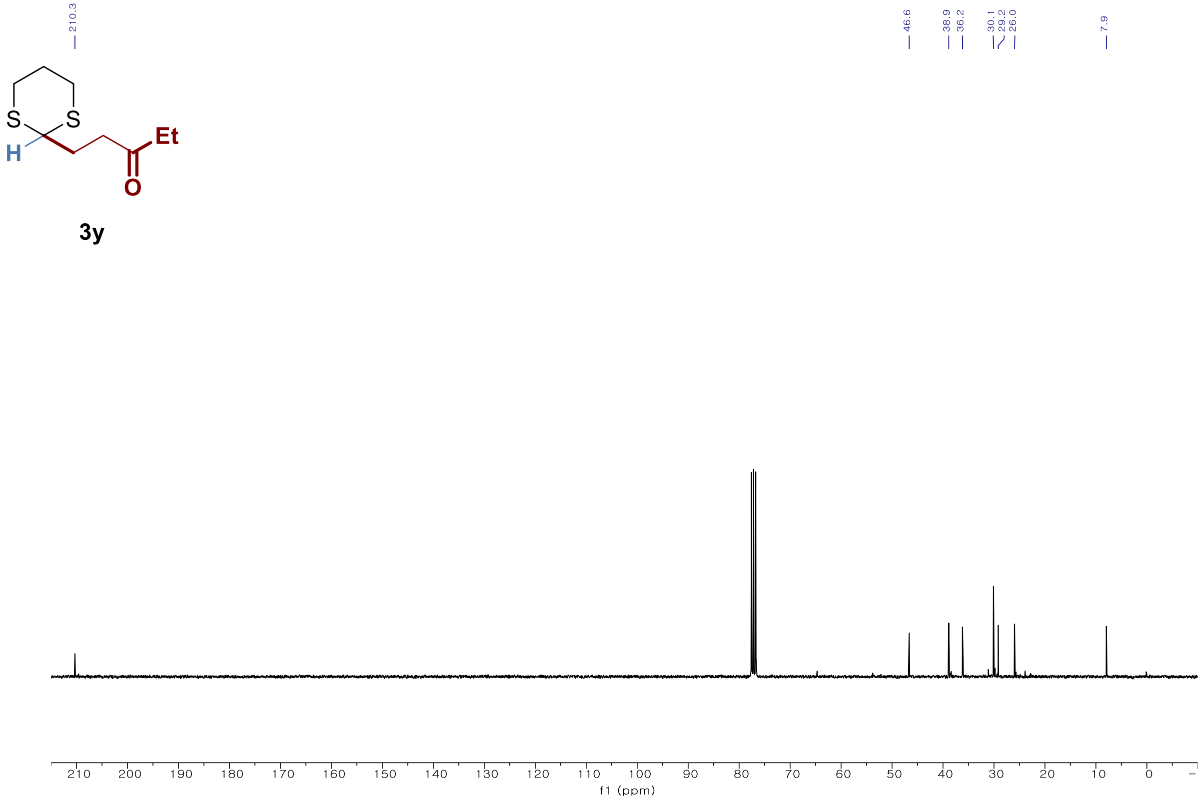
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3x**



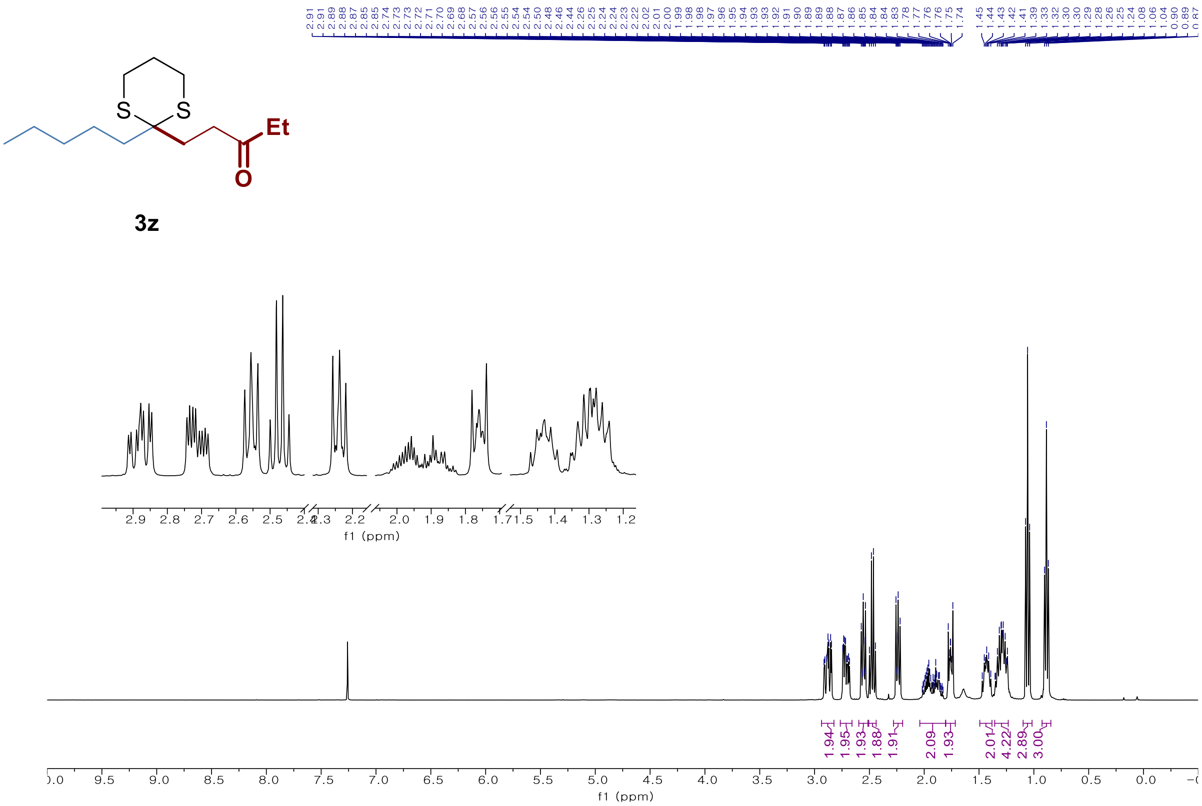
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3x**



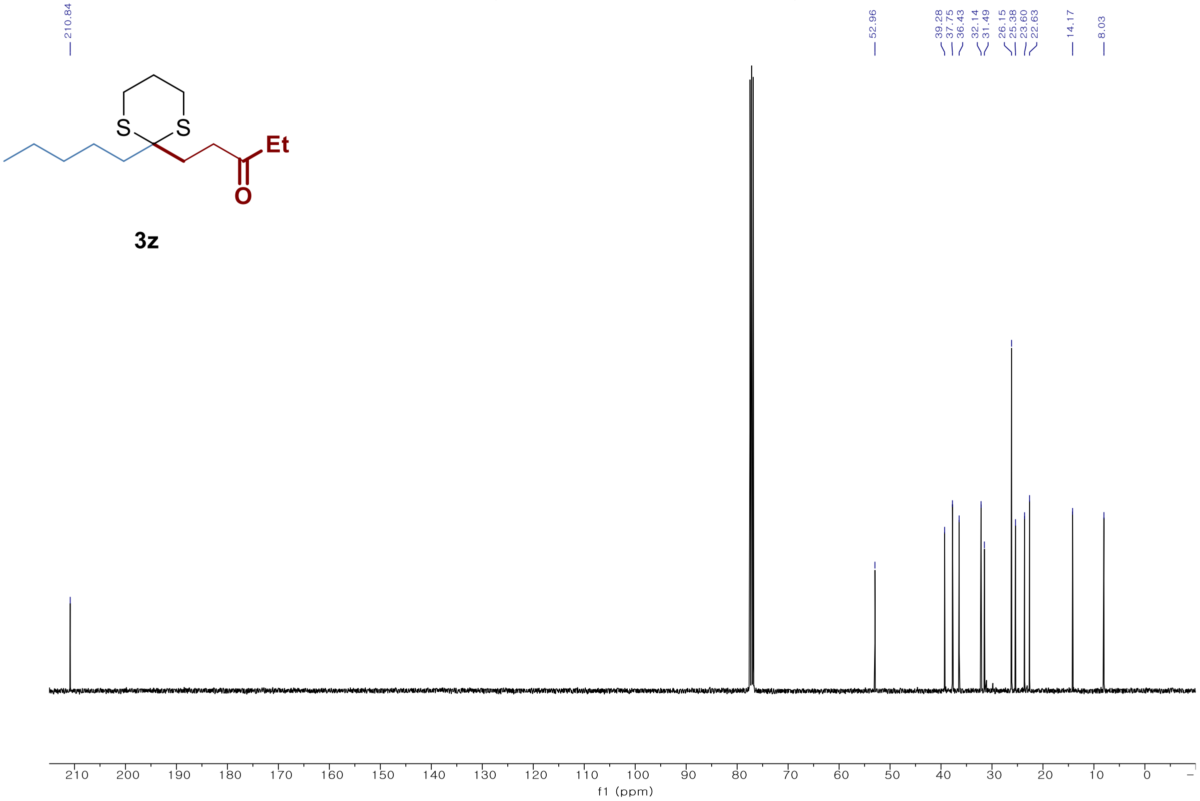
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3y**



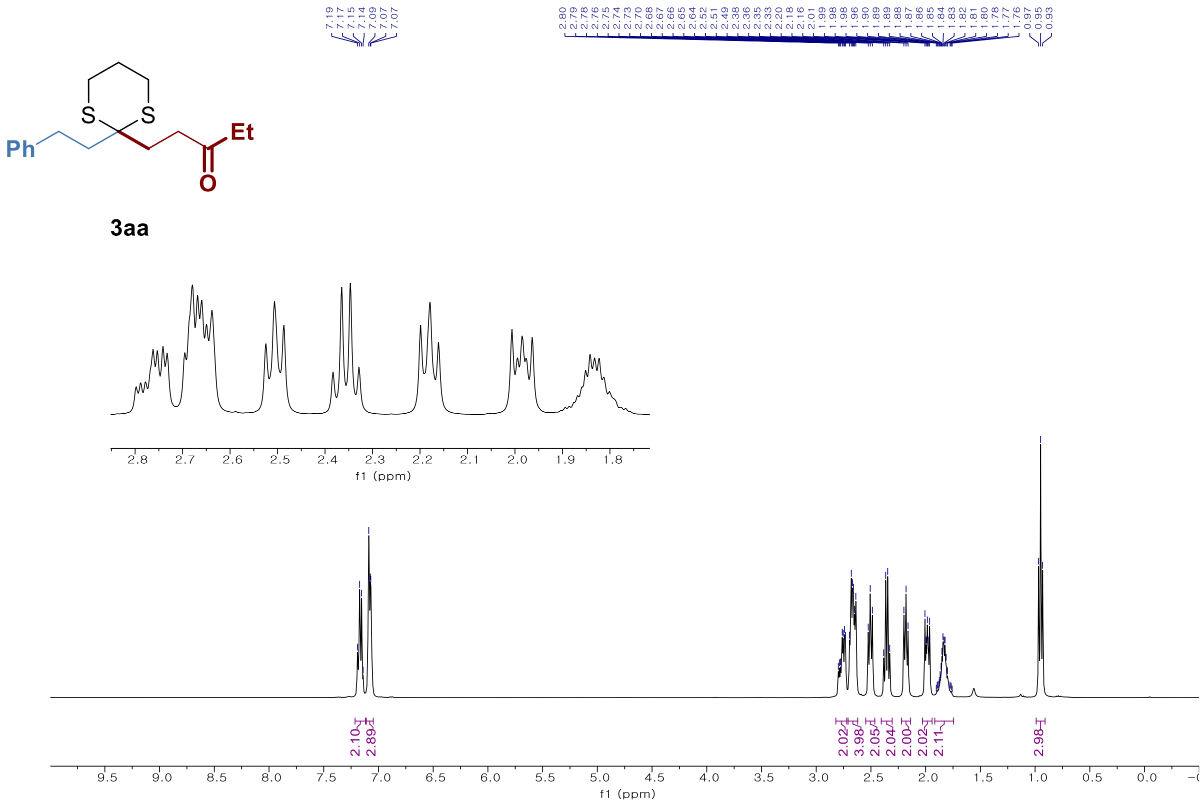
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3y**



^{1}H-NMR (400 MHz, Chloroform-*d*) of **3z**

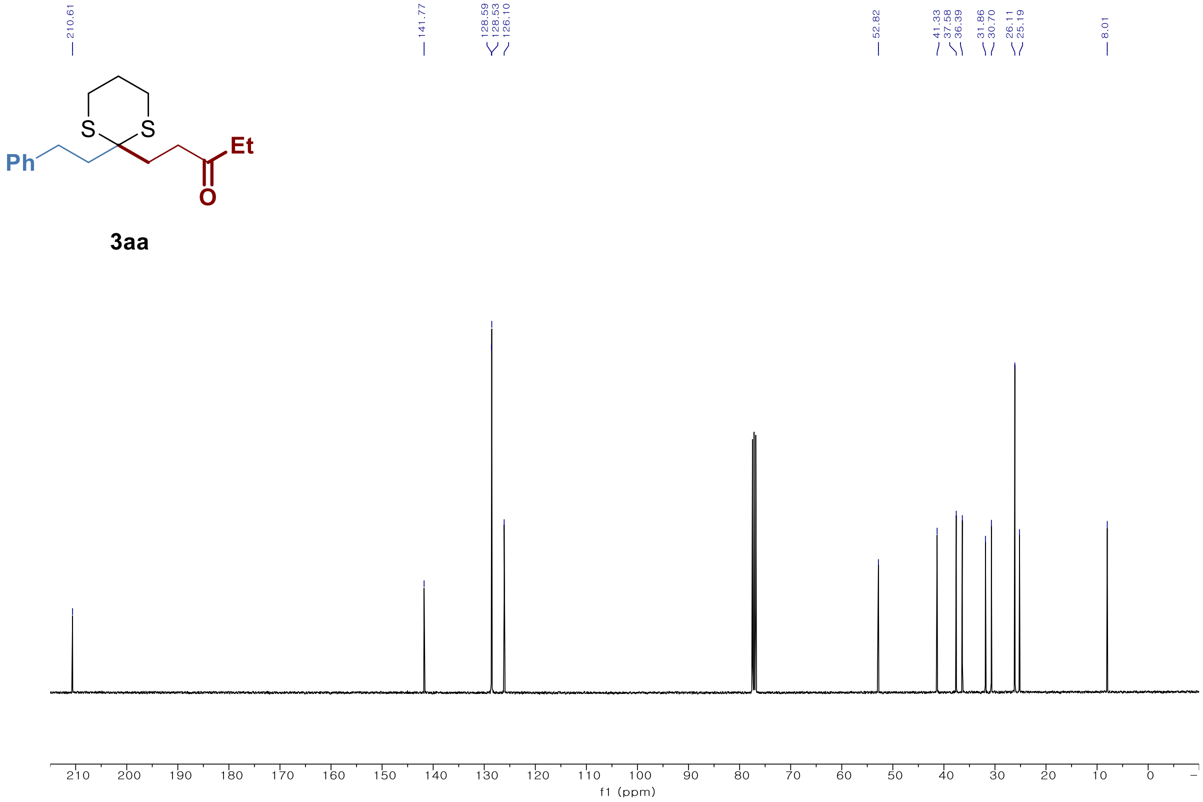


^{13}C-NMR (101 Hz, Chloroform-*d*) of **3z**



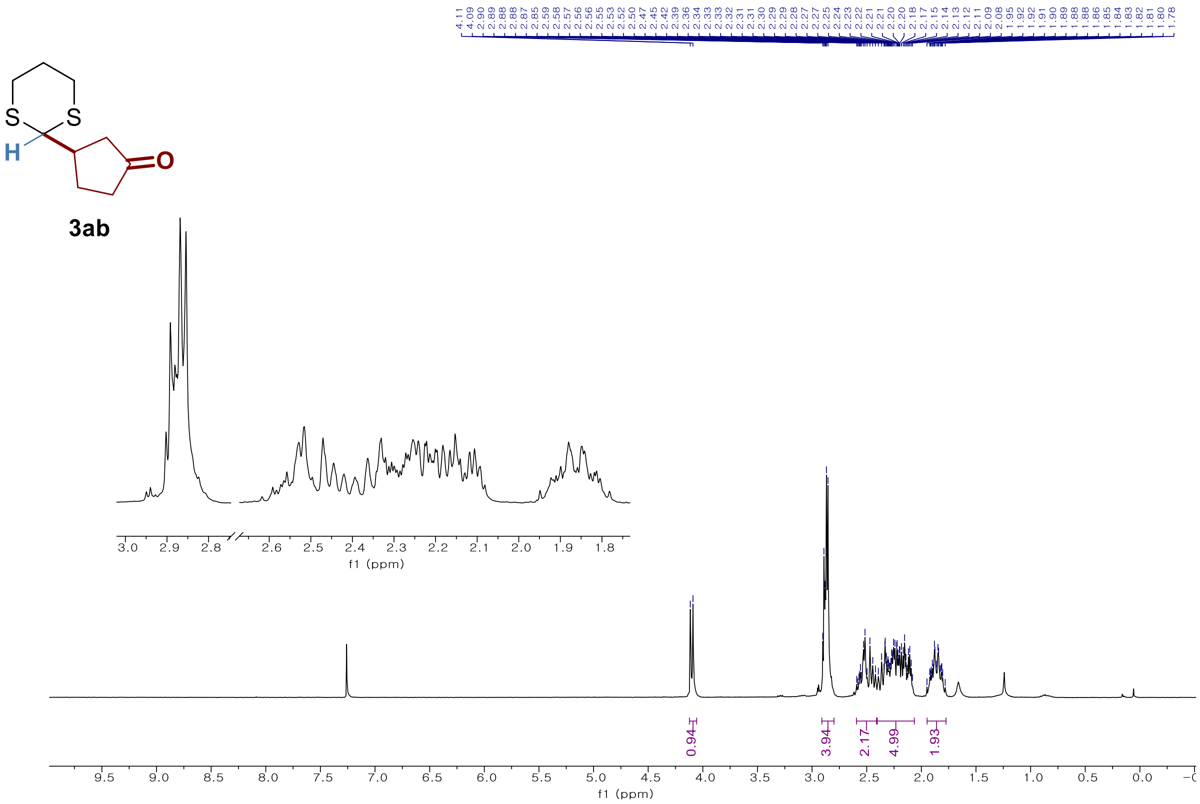
3aa

^{1}H-NMR (400 MHz, Chloroform-*d*) of **3aa**

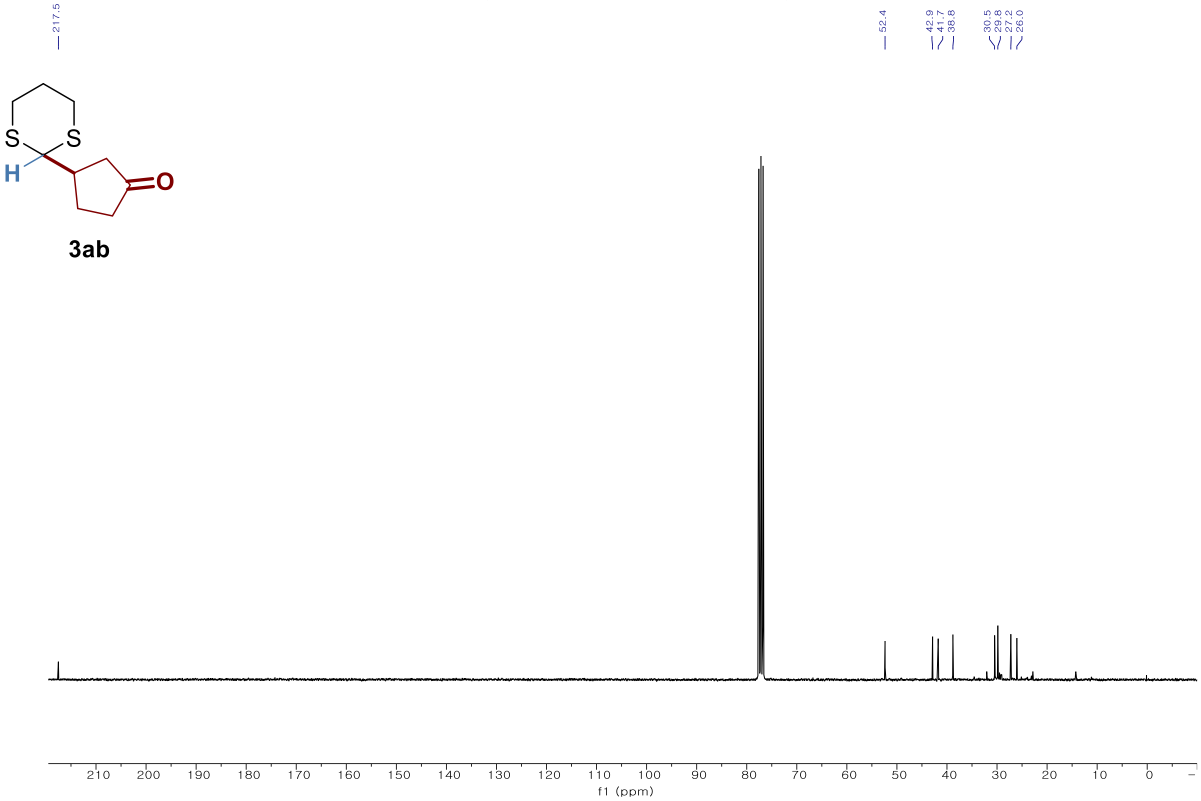


3aa

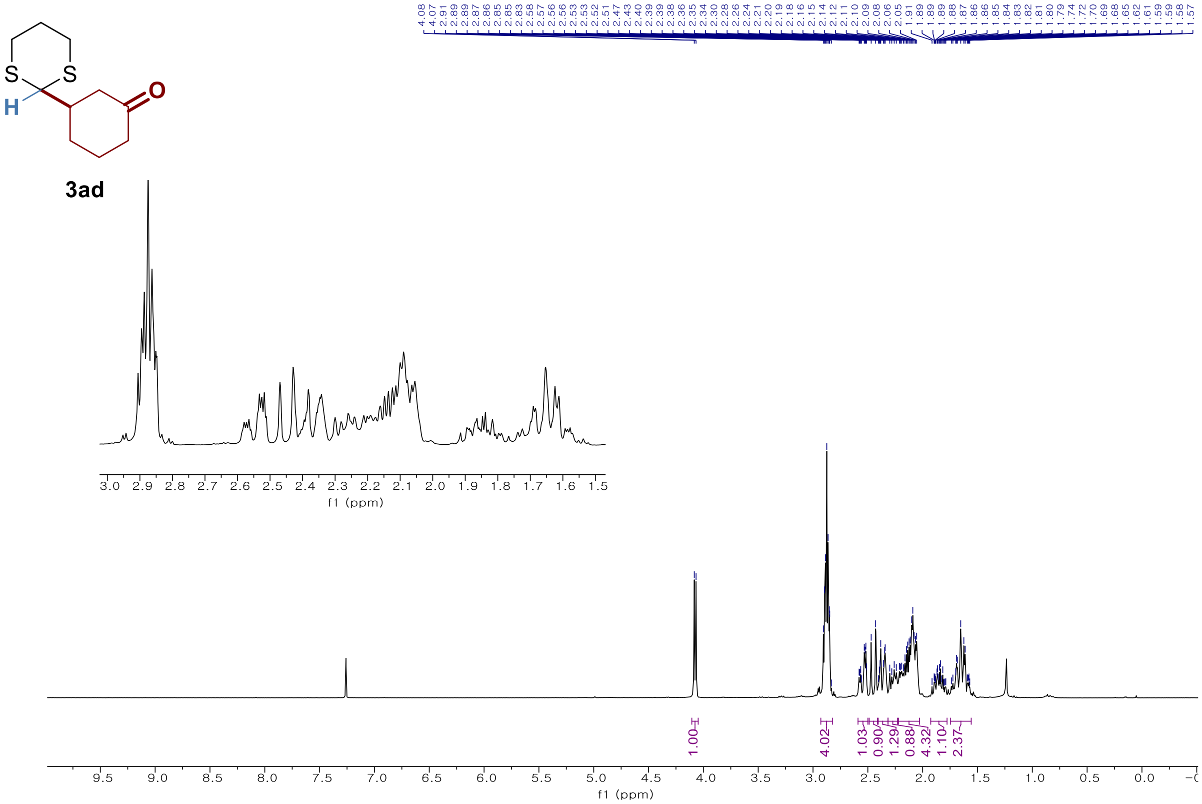
^{13}C-NMR (101 Hz, Chloroform-*d*) of **3aa**



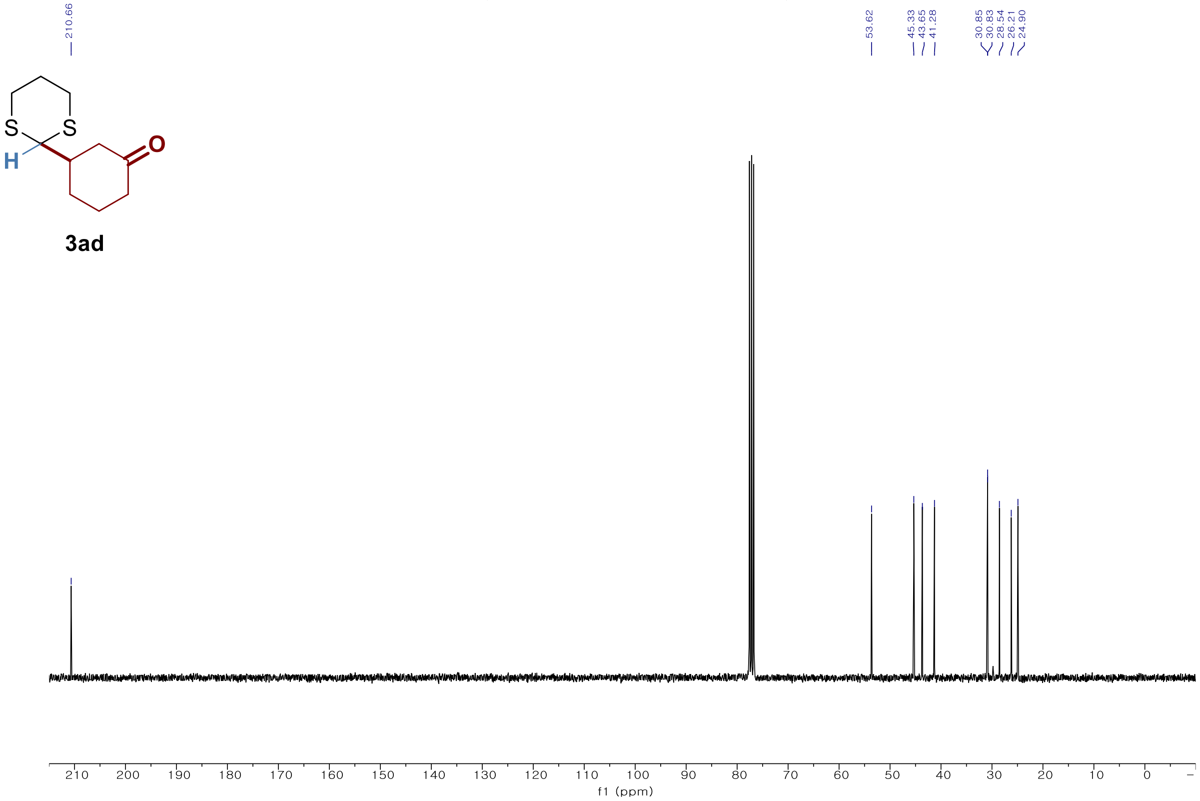
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3ab**



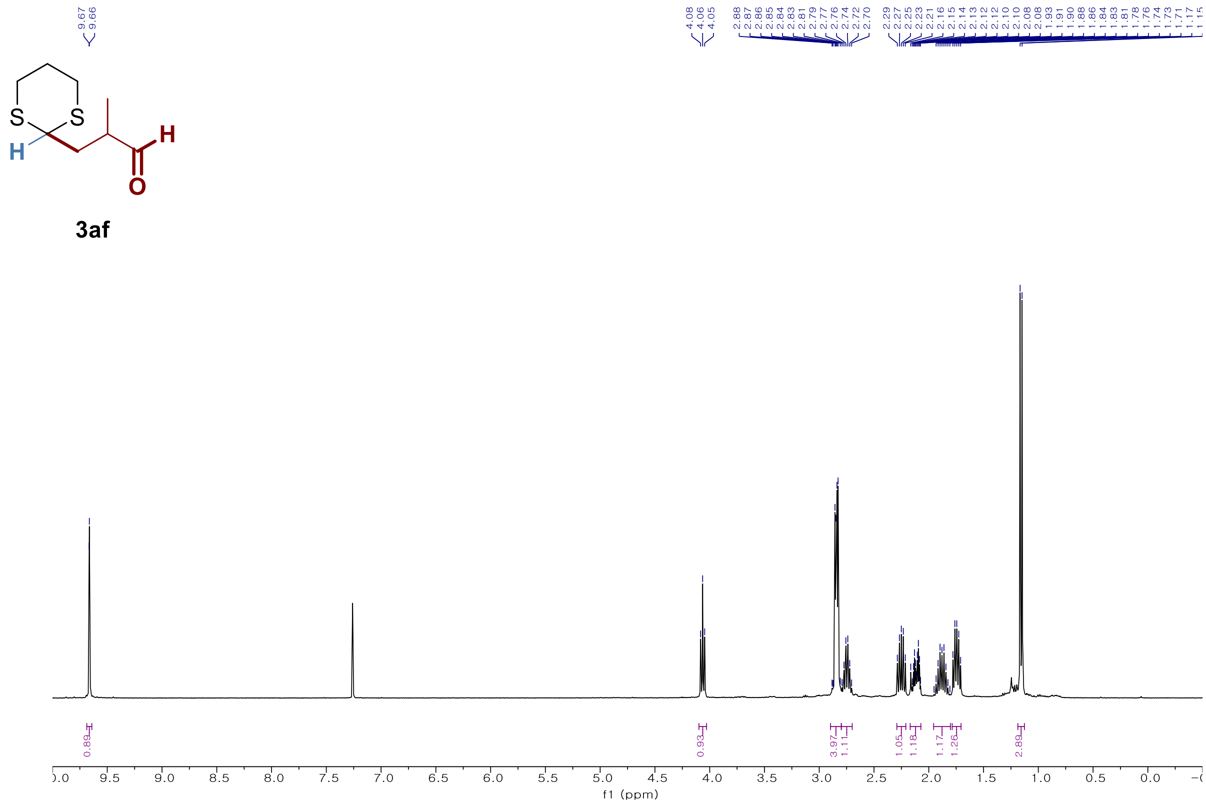
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3ab**



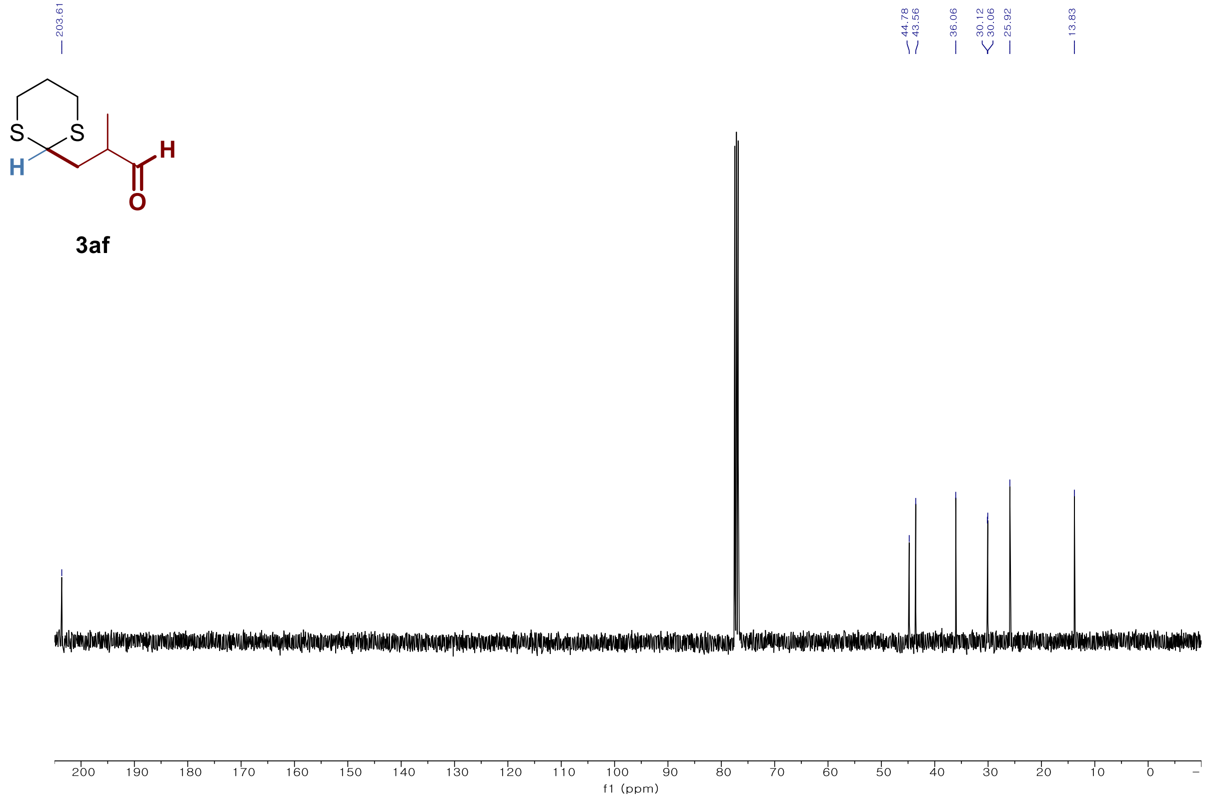
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3ad**



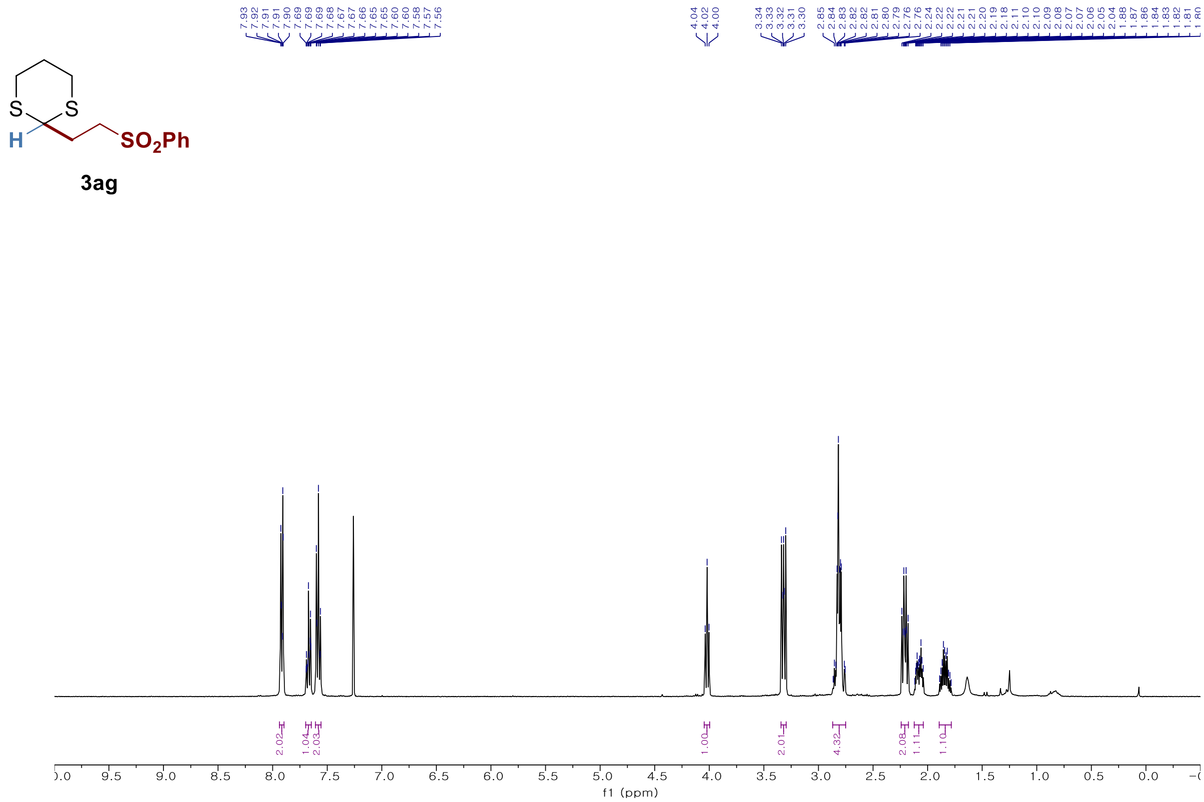
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3ad**



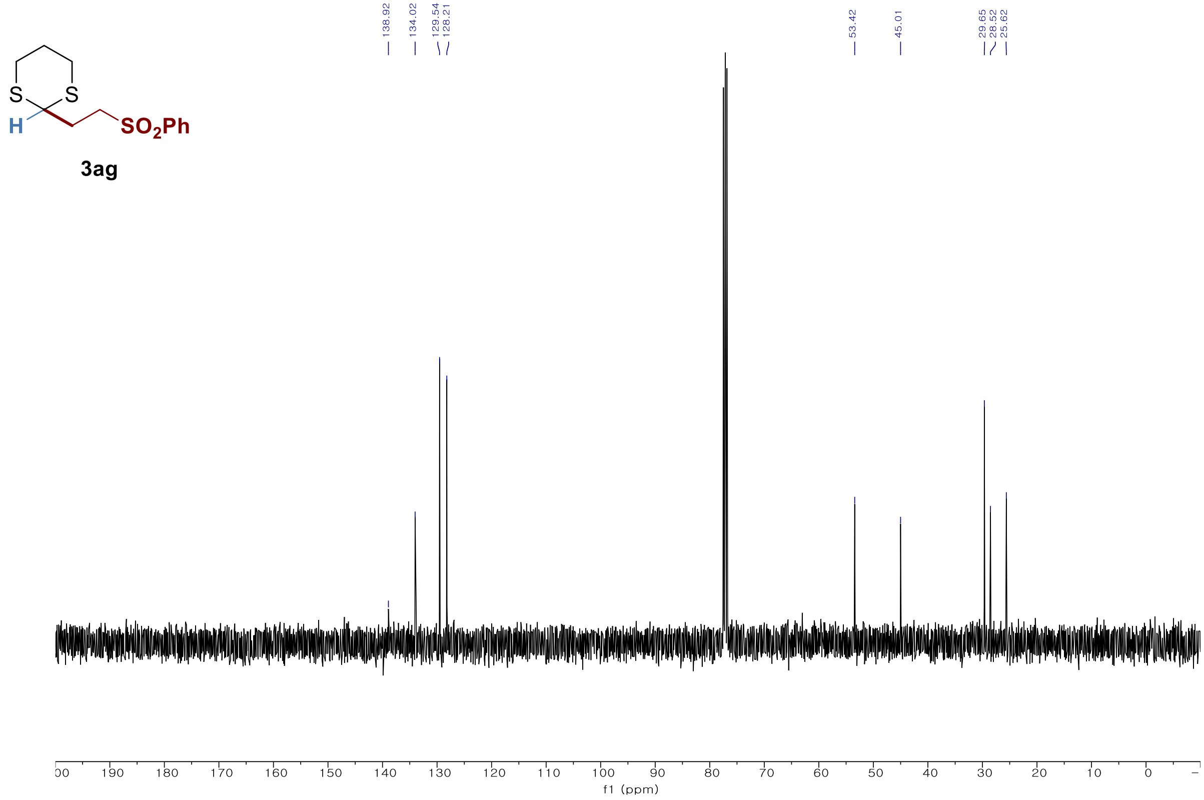
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3af**



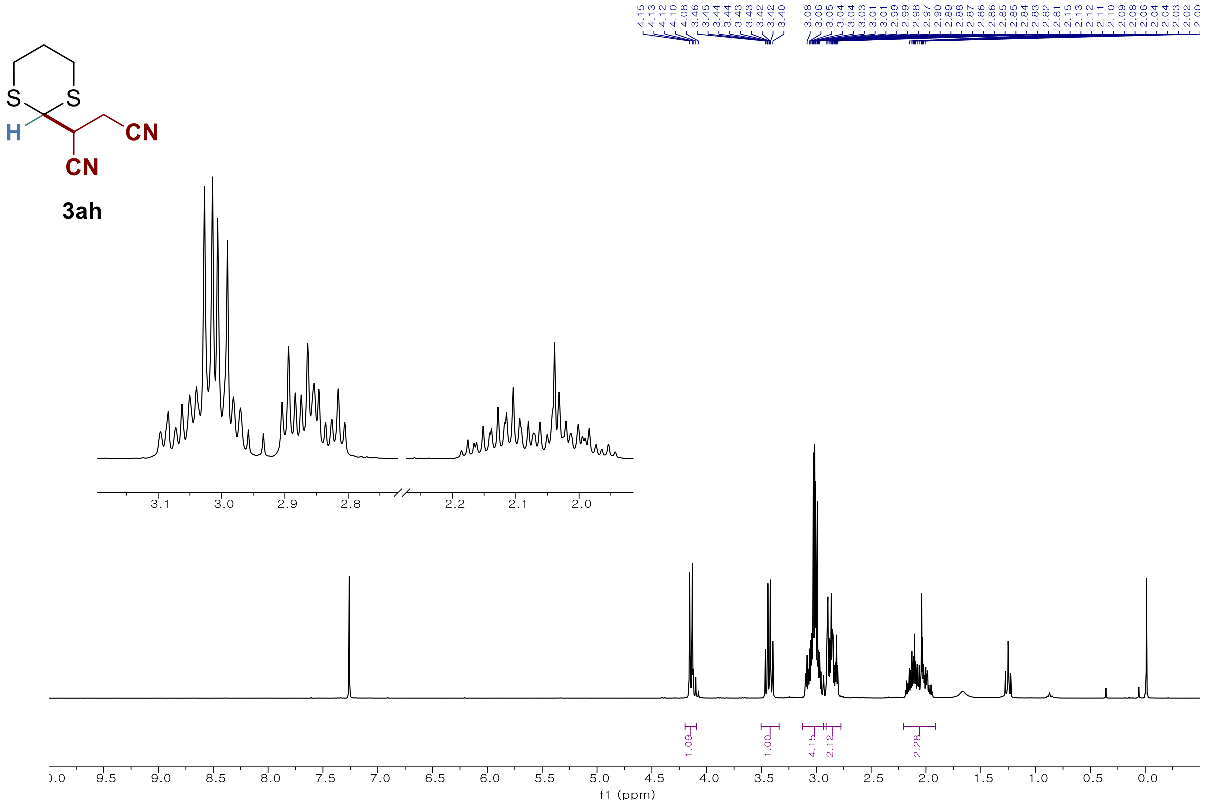
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3af**



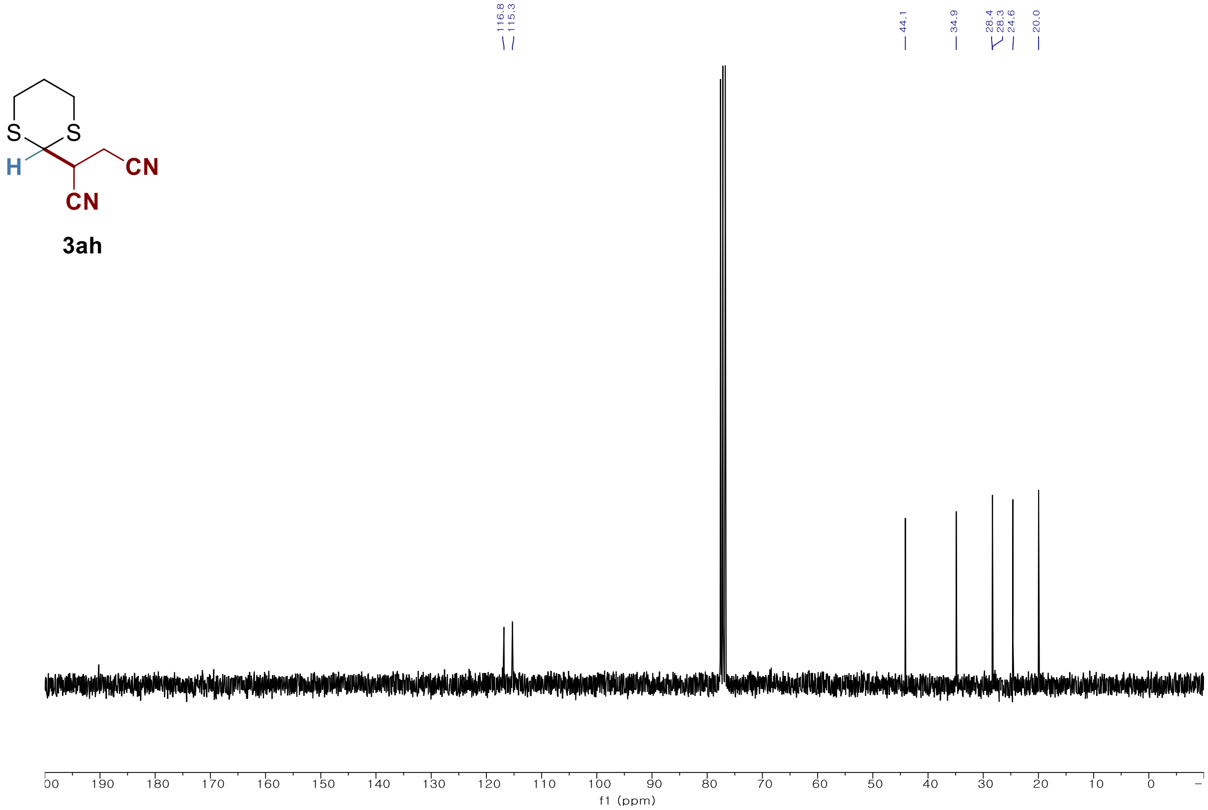
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3ag**



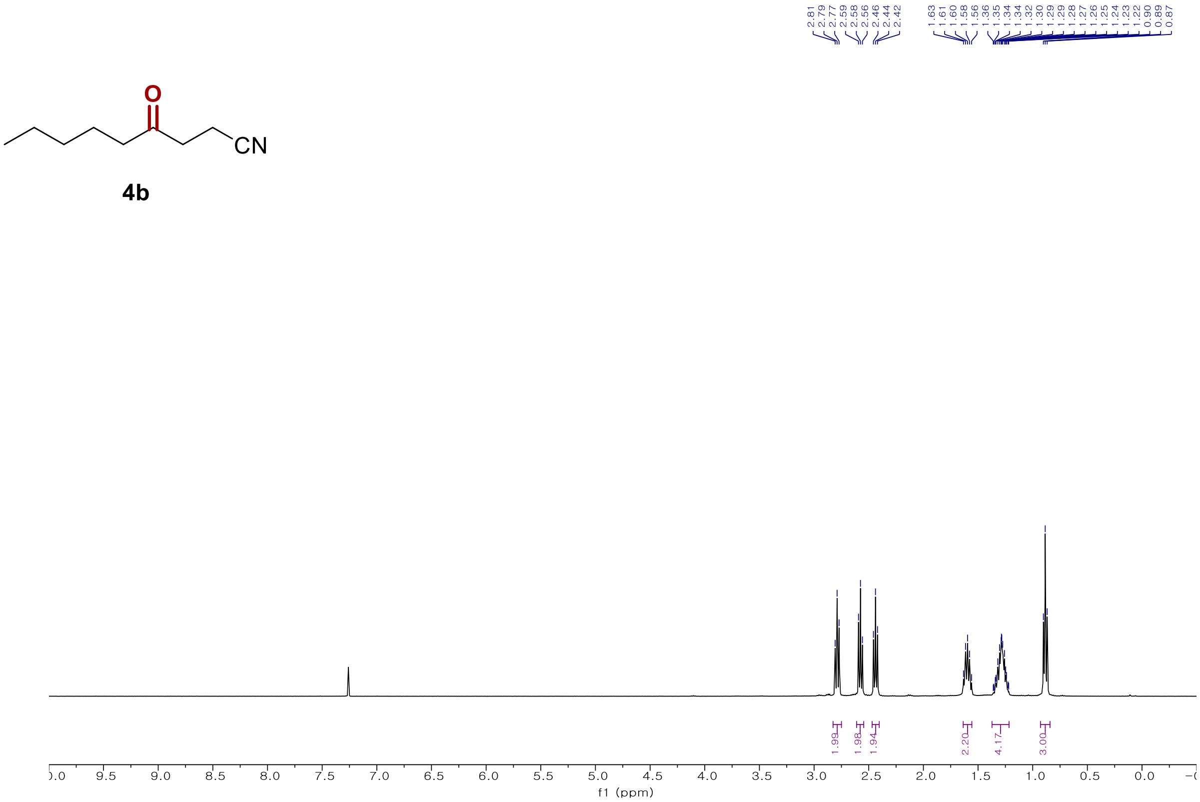
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3ag**



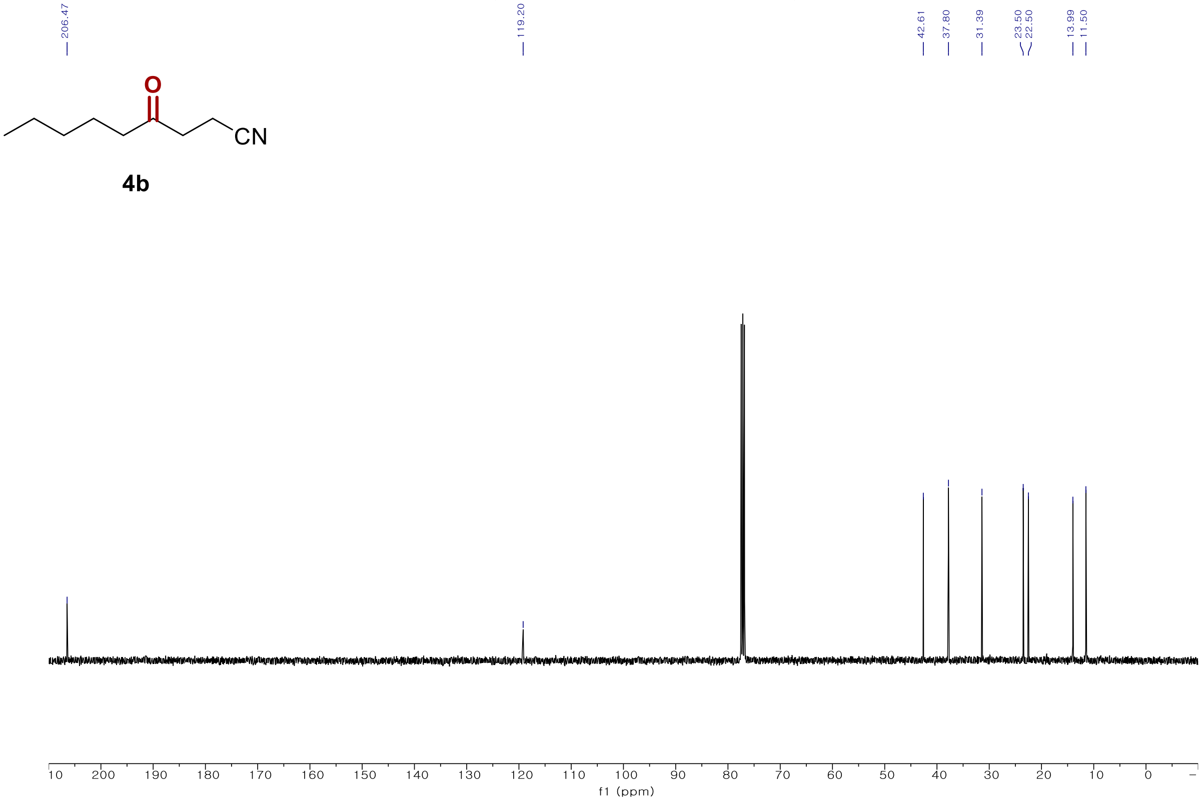
^{1}H-NMR (300 MHz, Chloroform-*d*) of **3ah**



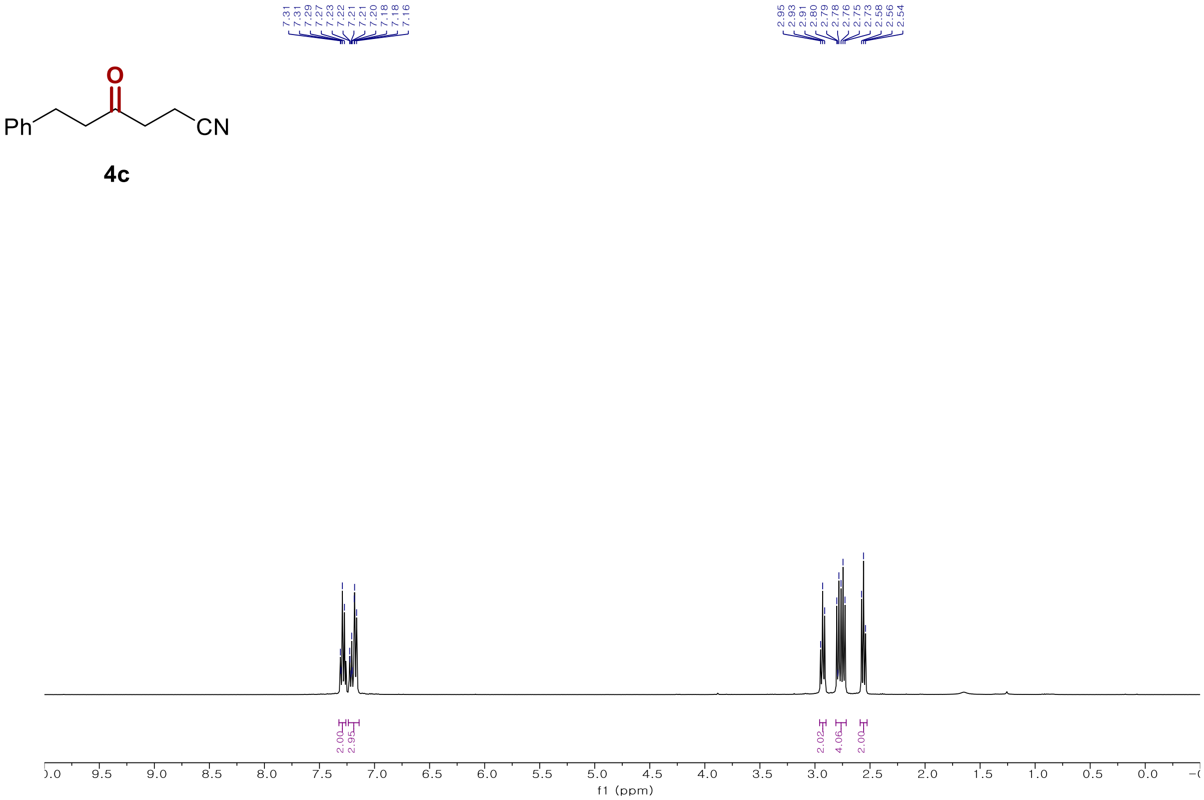
^{13}C-NMR (75 Hz, Chloroform-*d*) of **3ah**



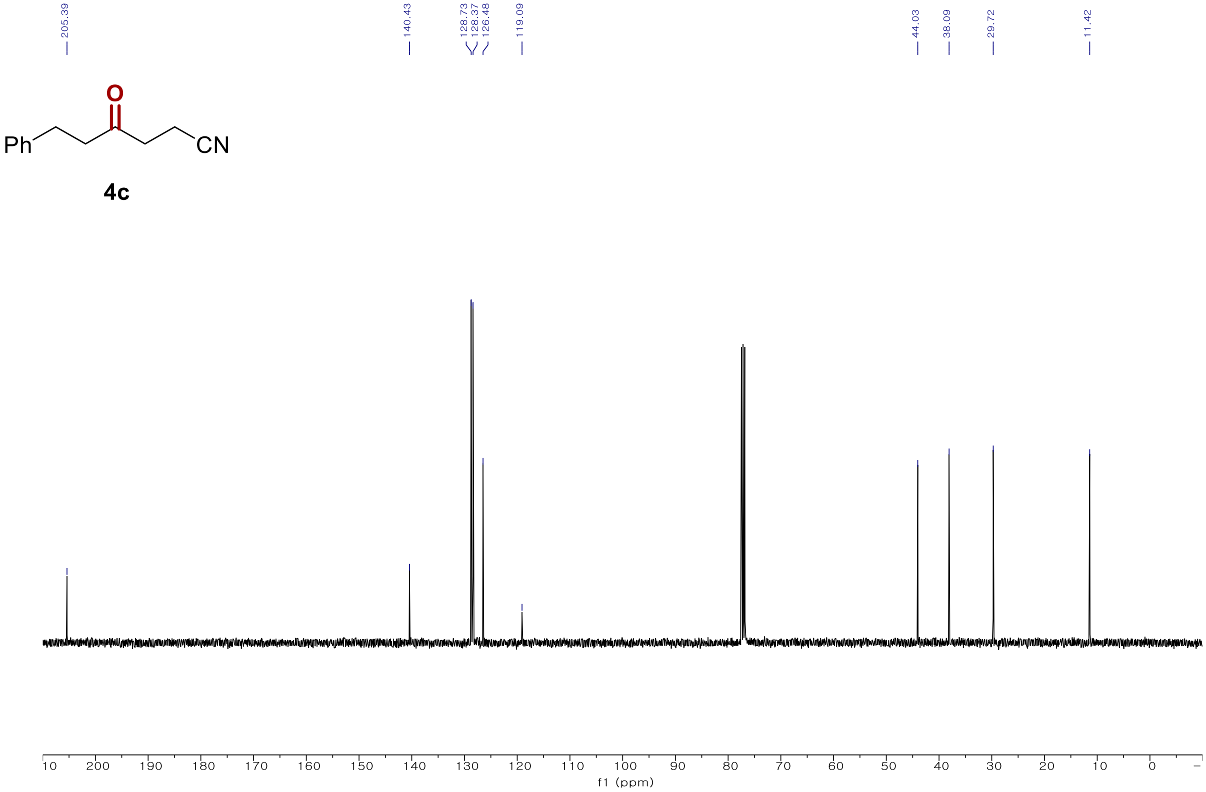
^{1}H-NMR (400 MHz, Chloroform-*d*) of **4b**



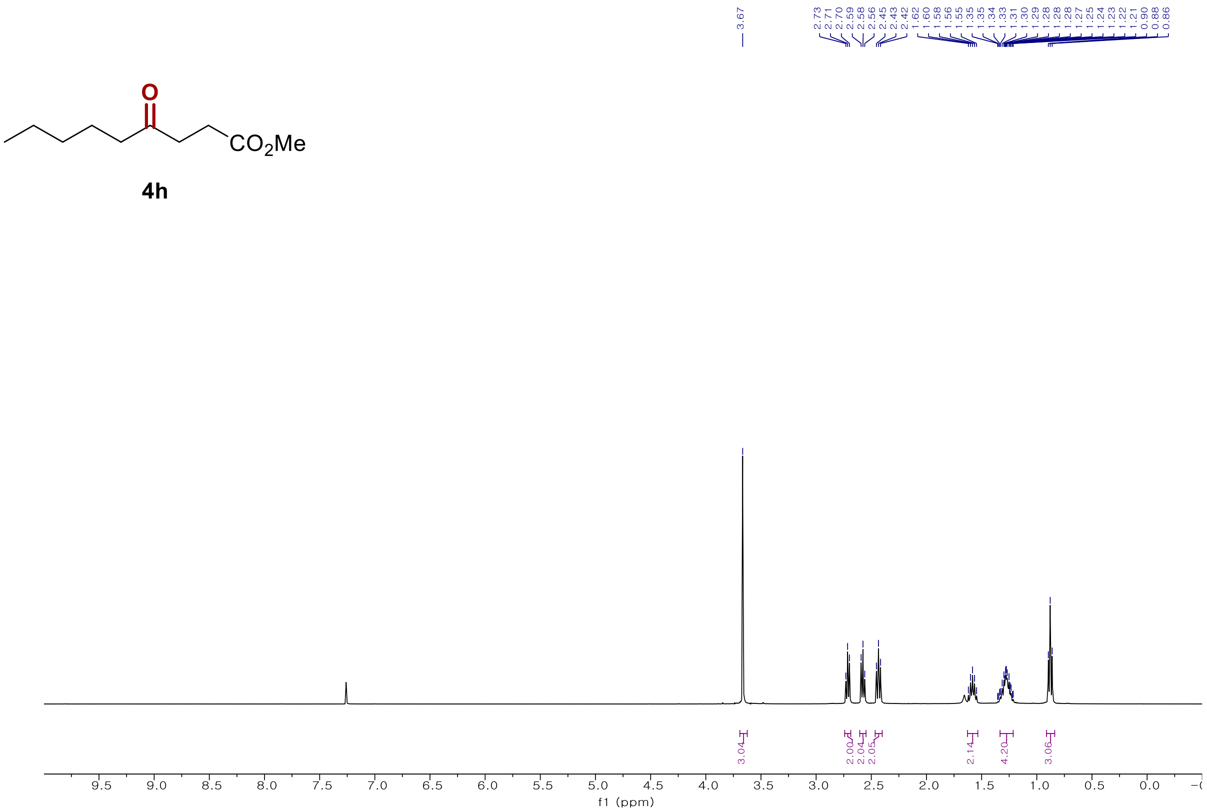
^{13}C-NMR (101 Hz, Chloroform-*d*) of **4b**



^{1}H-NMR (400 MHz, Chloroform-*d*) of **4c**

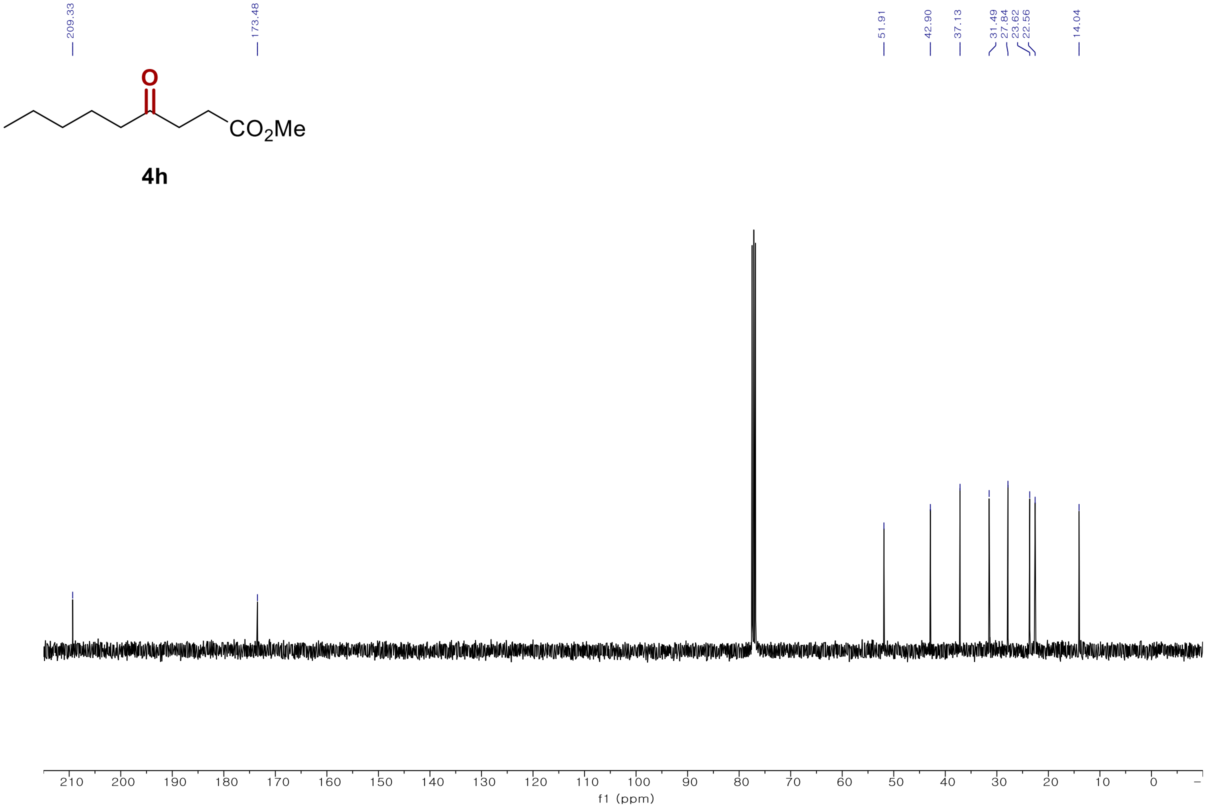


^{13}C-NMR (101 Hz, Chloroform-*d*) of **4c**



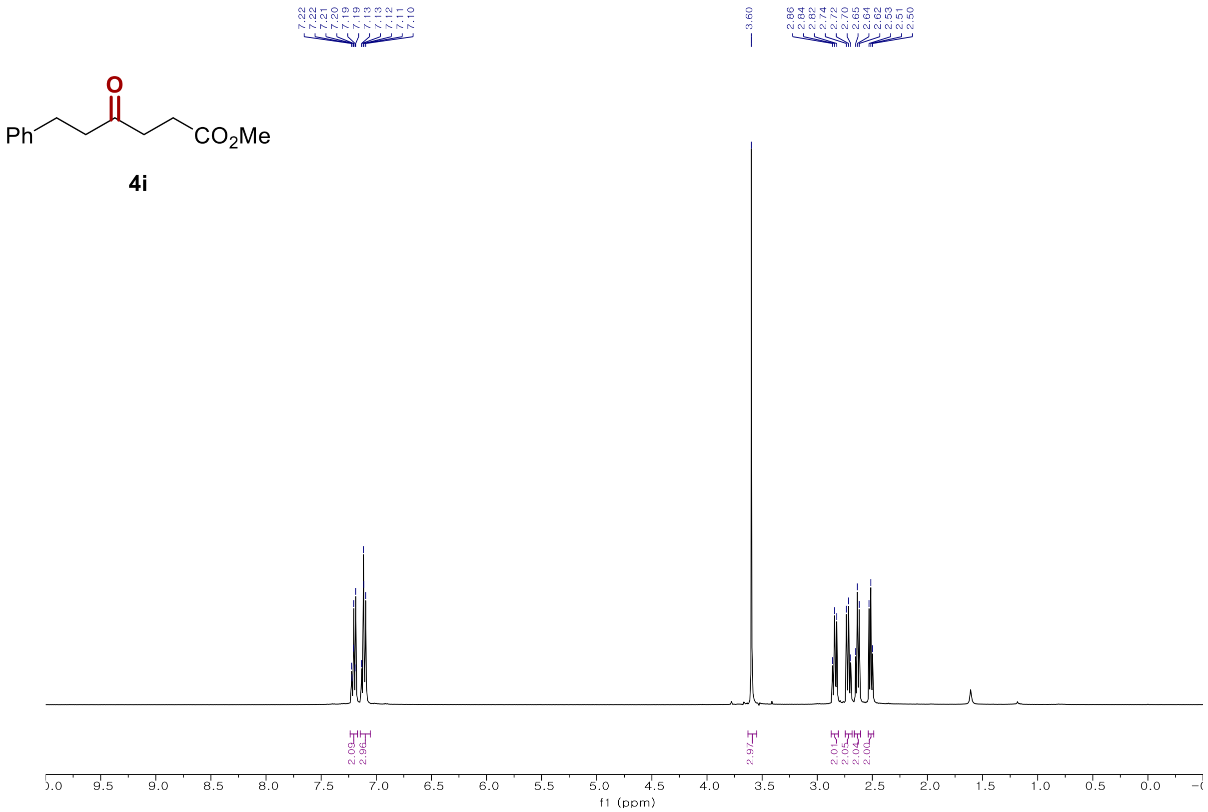
4h

^{1}H-NMR (400 MHz, Chloroform-*d*) of **4h**

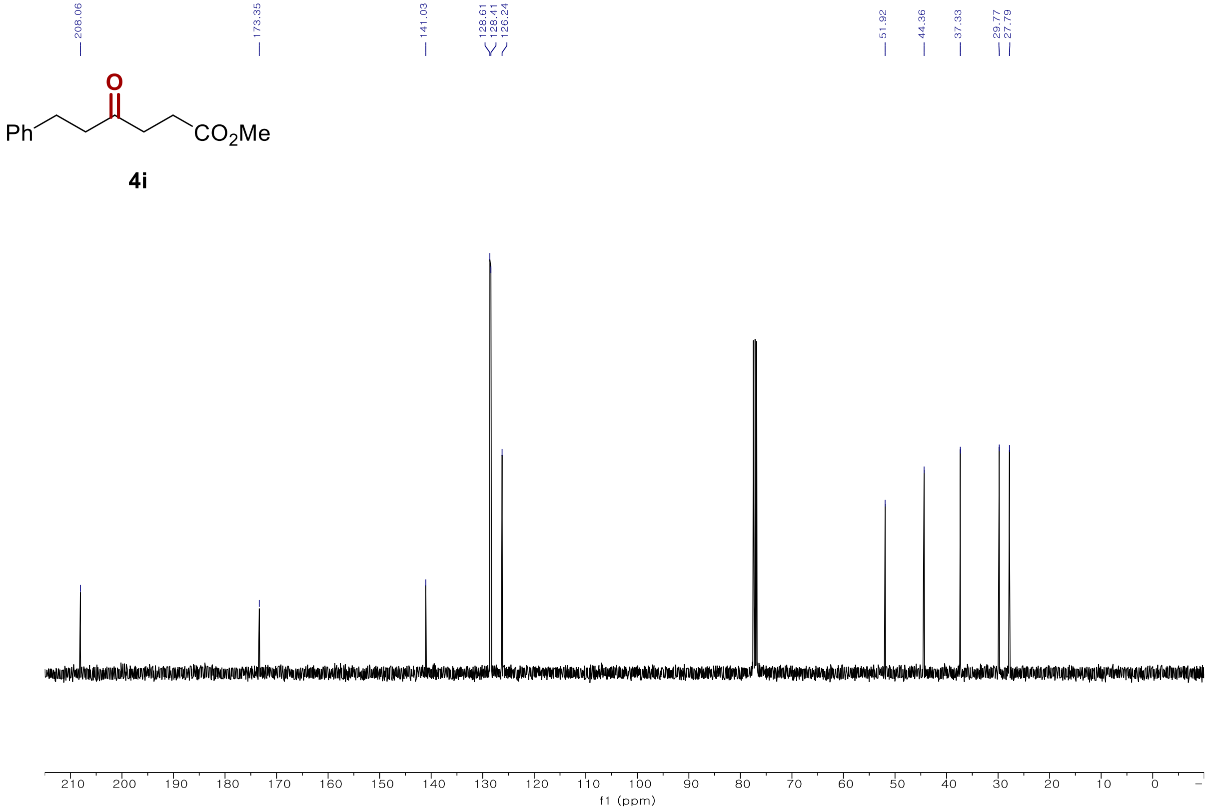


4h

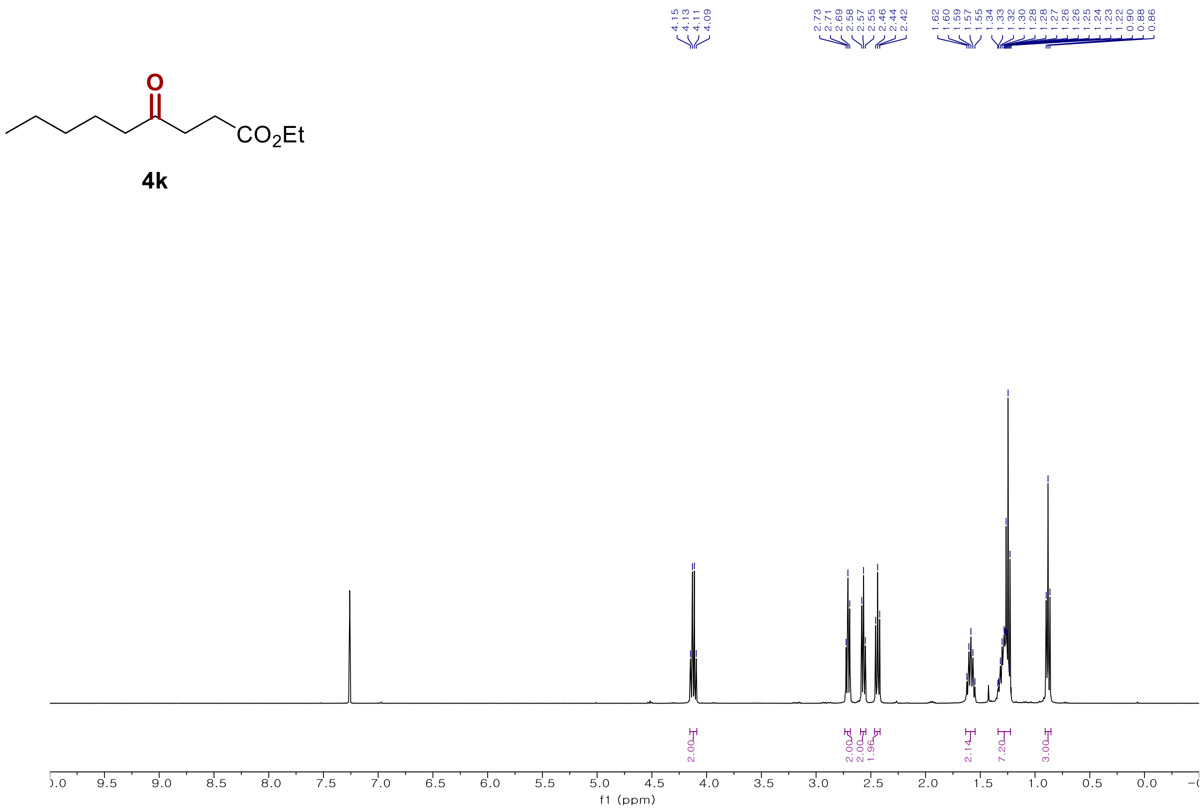
^{13}C-NMR (101 Hz, Chloroform-*d*) of **4h**



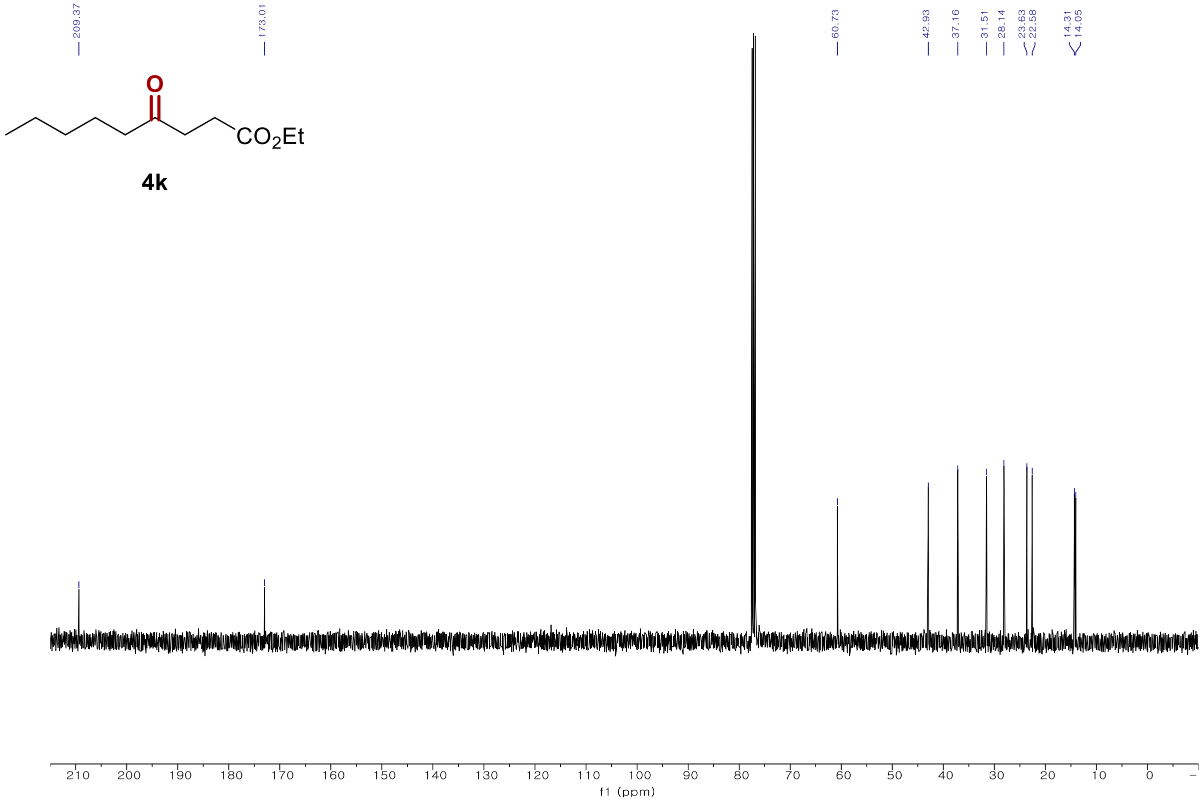
^{1}H-NMR (400 MHz, Chloroform-*d*) of **4i**



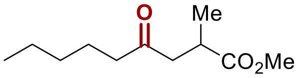
^{13}C-NMR (101 Hz, Chloroform-*d*) of **4i**



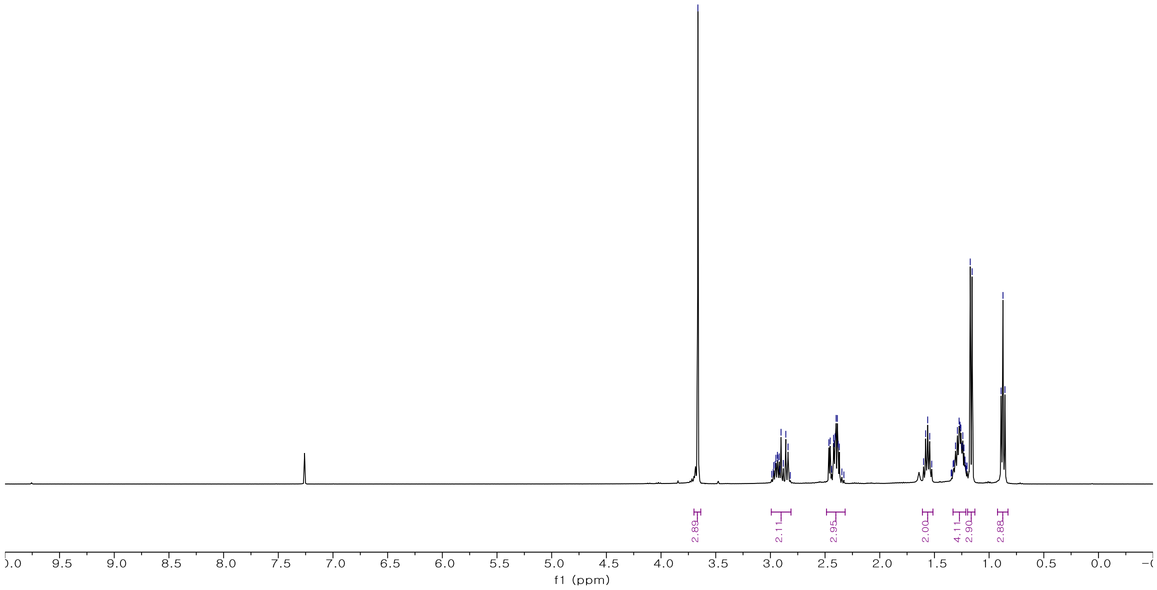
^{1}H-NMR (400 MHz, Chloroform-*d*) of **4k**



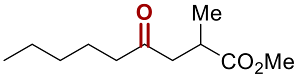
^{13}C-NMR (101 Hz, Chloroform-*d*) of **4k**



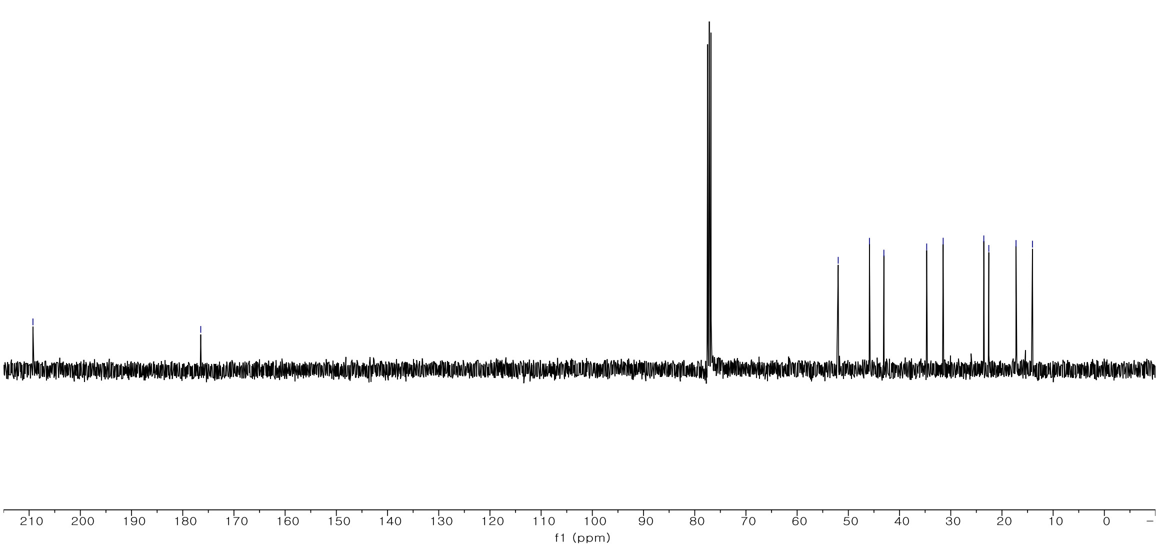
4o



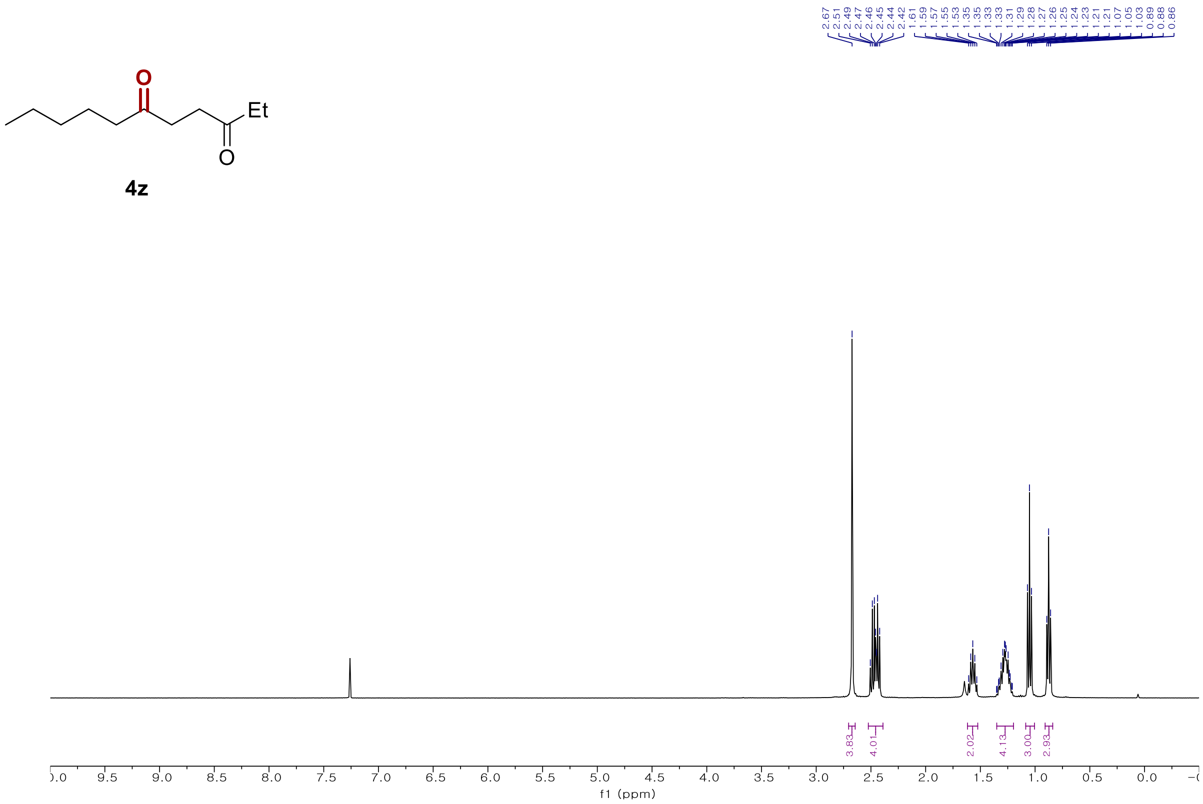
^{1}H-NMR (400 MHz, Chloroform-*d*) of **4o**



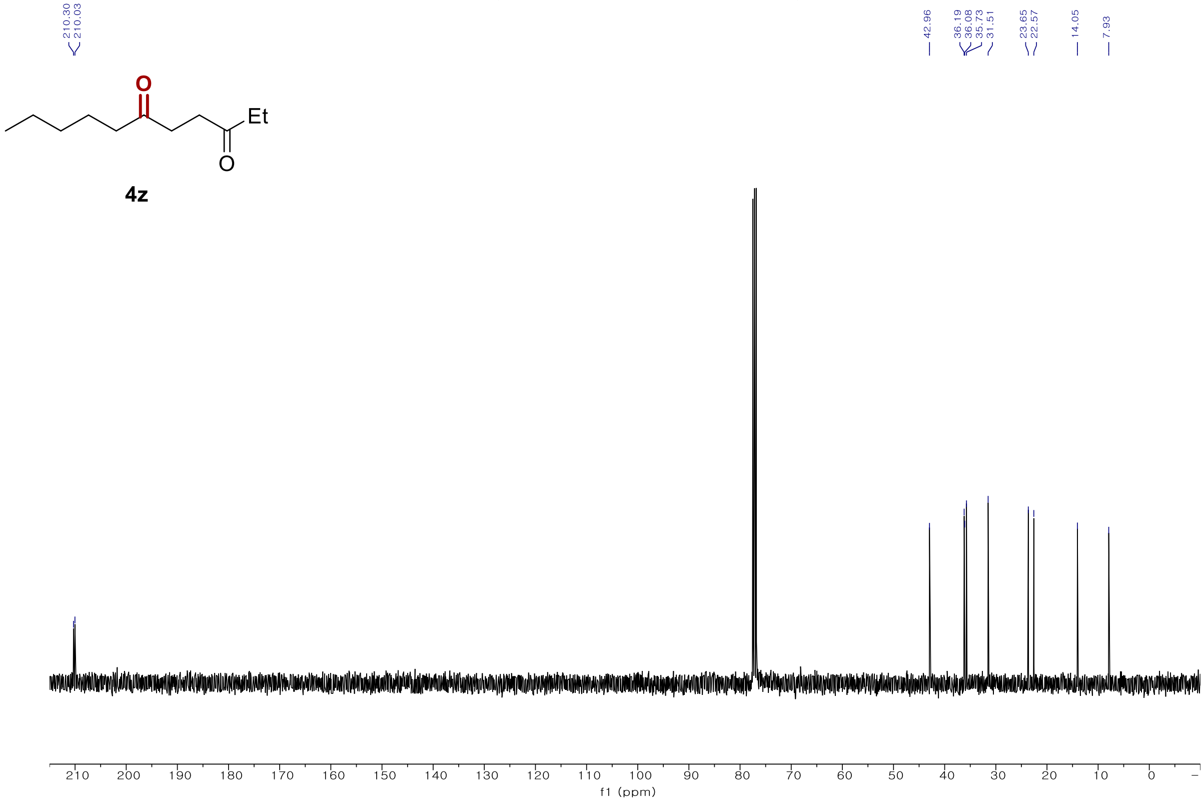
4o



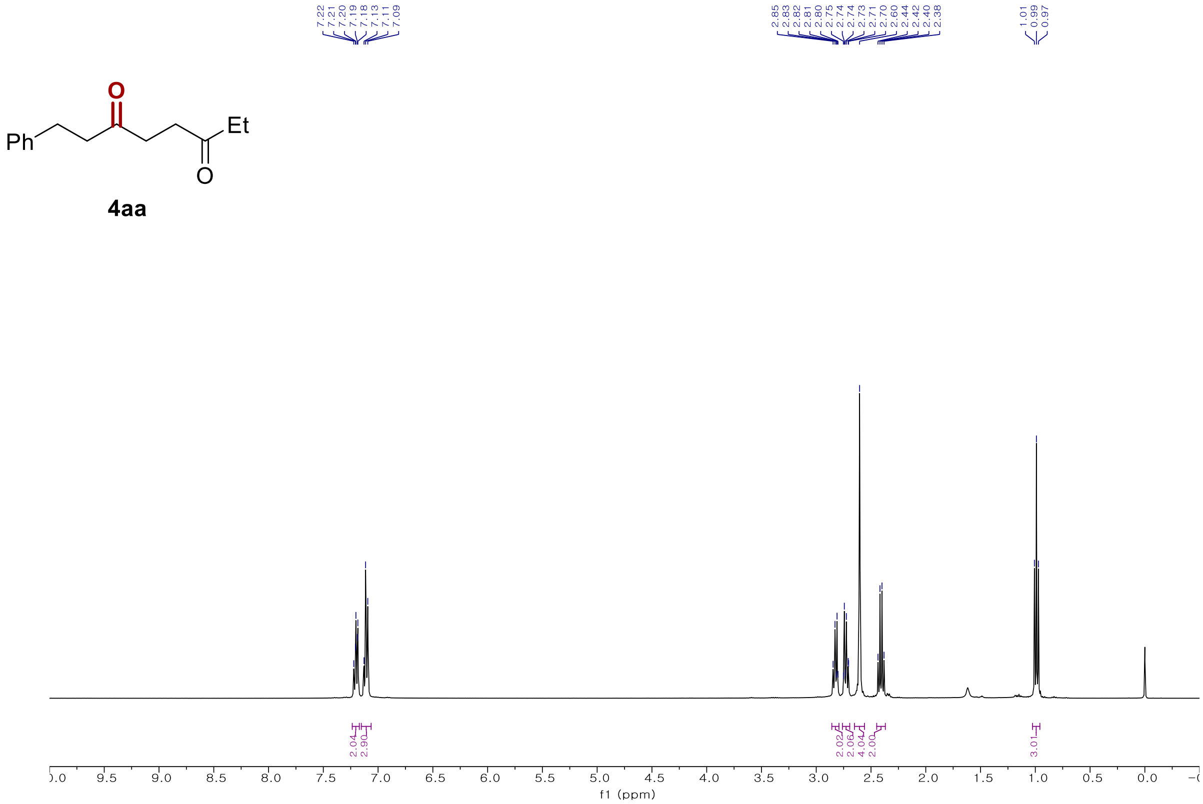
^{13}C-NMR (101 Hz, Chloroform-*d*) of **4o**



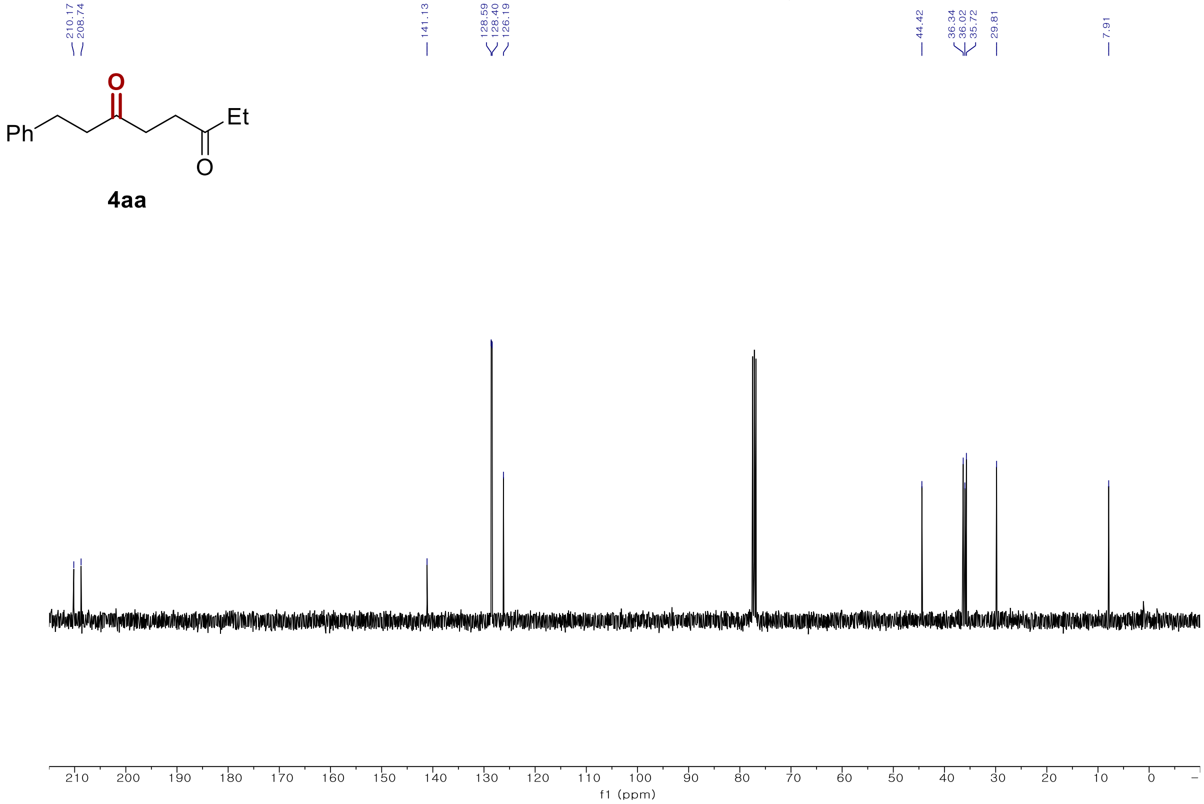
^{1}H-NMR (400 MHz, Chloroform-*d*) of **4z**



^{13}C-NMR (101 Hz, Chloroform-*d*) of **4z**



^{1}H-NMR (400 MHz, Chloroform-*d*) of **4aa**



^{13}C-NMR (101 Hz, Chloroform-*d*) of **4aa**