Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information for

Copper-Catalyzed Synthesis of Primary Amides Through Reductive N–O Cleavage of Dioxazolones

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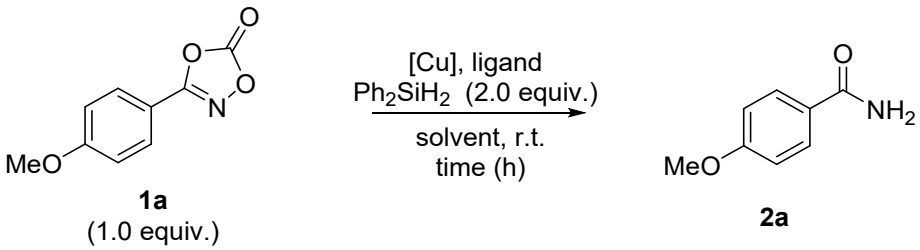
I. General Experimental Information

^{1}H-NMR, ^{13}C-NMR spectra were collected at ambient temperature using Agilent 400 MHz (400-MR DD2) spectrometer. The data are shown as follows: chemical shift in ppm from internal tetramethylsilane (TMS) on the δ scale, multiplicity (brs = broad singlet, br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Infrared (IR) spectra were recorded on a JASCO 4600 using either neat oil or solid products max in cm^{-1}. High resolution mass spectra were recorded on a high-resolution time of flight (TOF) analyzer by using electron spray ionization (ESI) source (Synapt G2, Waters) from KBSI (Ochang, Korea), and were gained by peak matching. Dioxazolones were prepared using reported procedures (Page S22). Unless otherwise noted, other chemicals including copper, ligands, silane and solvents were purchased from commercial sources, and were used without any further purification. Analytical thin layer chromatography (TLC) was carried out on Sigma Aldrich TLC silica gel 60 F_{254} 25 Aluminum sheets with UV 254 nm fluorescent indicator. Flash chromatography was also performed using 60Å (63 – 200 μm) mesh silica gel (SiO_2). Unless otherwise noted, all chemical reactions were carried out under argon atmosphere.

II. Expanded Optimization Table of the Synthesis of Primary Amides 2a from Dioxazolones 1a



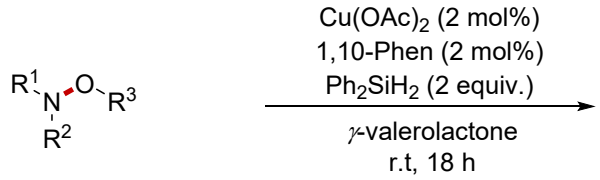
1a
(1.0 equiv.)

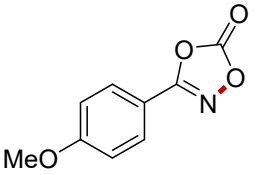
2a

Entry[a]	[Cu] source	Ligand	Solvent	Time (h)	Temp (°C)	Yield (%)
1	-	1,10-Phen	ɤ-valerolactone	18	r.t	N.R
2	CuCl	1,10-Phen	ɤ-valerolactone	18	r.t	41
3	$Cu(OAc)_1$	1,10-Phen	ɤ-valerolactone	18	r.t	92
4	$Cu(OAc)_2$	1,10-Phen	ɤ-valerolactone	18	r.t	98
5	$Cu(OAc)_2$	1,10-Phen	ɤ-valerolactone	18	r.t	41[b]
6	$Cu(OAc)_2$	-	ɤ-valerolactone	18	r.t	26
7	$Cu(OAc)_2$	DPPE	ɤ-valerolactone	18	r.t	< 5
8	$Cu(OAc)_2$	PPh_3	ɤ-valerolactone	18	r.t	< 5[c]
9	$Cu(OAc)_2$	PPh_3	ɤ-valerolactone	18	80	65[c]
10	$Cu(OAc)_2$	pyridine	ɤ-valerolactone	18	r.t	95[c]
11	$Cu(OAc)_2$	1,10-Phen	DMF	18	r.t	45
12	$Cu(OAc)_2$	1,10-Phen	DCE	18	r.t	95
	$Cu(OAc)_2$	1,10-Phen	DCE	18	r.t	< 5[d]
13	$Cu(OAc)_2$	1,10-Phen	MeOH	18	r.t	50
14	$Cu(OAc)_2$	1,10-Phen	acetophenone	18	r.t	95
15	$Cu(OAc)_2$	1,10-Phen	hexamethylacetone	18	r.t	27
16	$Cu(OAc)_2$	1,10-Phen	PhMe	18	r.t	80
17	$Cu(OAc)_2$	1,10-Phen	C_6H_6	18	r.t	57
18	$Cu(OAc)_2$	1,10-Phen	hexane	18	r.t	trace
19	$Cu(OAc)_2$	1,10-Phen	H_2O	18	r.t	7

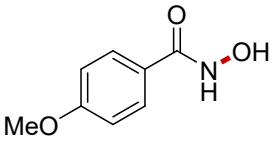
[a] Reaction conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (2 mol%), 1,10-phenanthroline (2 mol%), Ph_2SiH_2 (2.0 equiv) and solvent (1.0 mL) under argon at room temperature for 18 h. [b] Ph_2SiH_2 (1.0 equiv.) was used. [c] Ligand (4 mol%) was used. [d] Without Ph_2SiH_2

III. Scope of Reagents Containing *N–O* Bond Moiety





1a: 98%



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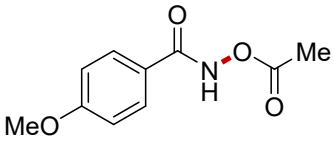
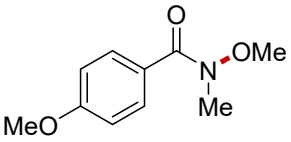


S2: 95%



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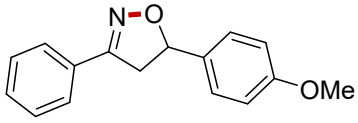

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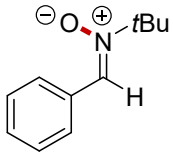

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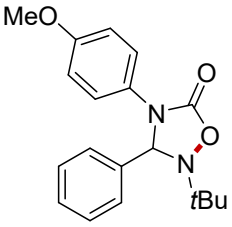
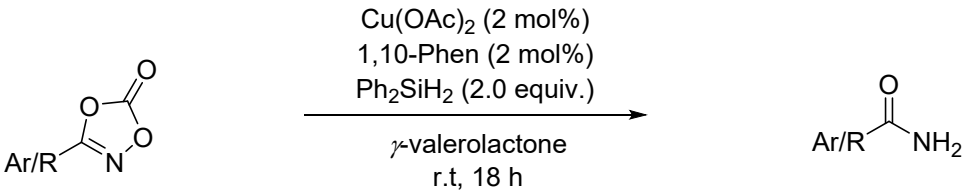


S6: no conversion

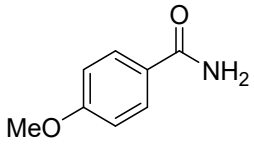
IV. Synthesis of Primary Amides 2a – 2y (Scheme 2)



1 **2**

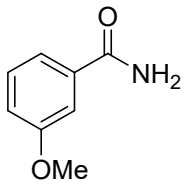
General procedure A. A conical vial (5.0 mL) was charged with $Cu(OAc)_2$ (0.0007 g, 0.004 mmol), 1,10-phenanthroline (0.0007 g, 0.004 mmol) and ɤ-valerolactone (1.0 mL)**.** The reaction mixture was flushed with argon balloon for 2 min to purge with argon atmosphere. The reaction vial was capped rapidly, and the mixture was stirred at room temperature for 15 min. Diphenylsilane (0.074 mL, 0.400 mmol) was then added via syringe and the resulting mixture was stirred at room temperature for additional 20 min until the solution turned from green to a dark orange color. Dioxazolone (0.200 mmol) **1** was added to the reaction mixture followed by flushing with argon balloon for 2 min to purge with argon atmosphere. The resulting mixture was stirred at room temperature for 18 h. After completion of the reaction, the mixture was purified by flash chromatography (1:2 - 2:1; EtOAc:hexane) to give primary amide **2**.

General procedure B. (Gram-scale reaction). A round bottom flask (250 mL) was charged with $Cu(OAc)_2$ (0.0727 g, 0.4 mmol), 1,10-phenanthroline (0.0721 g, 0.4 mmol) and 1,2-dichloroethane (100 mL)**.** The reaction mixture was flushed with argon balloon for 2 min to purge with argon atmosphere. The reaction flask was capped rapidly, and the mixture was stirred at room temperature for 15 min. Diphenylsilane (7.42 mL, 40.0 mmol) was then added via syringe, and the resulting mixture was stirred at room temperature for additional 20 min until the solution turned from green to a dark orange color. Dioxazolone (20.0 mmol) **1** was added to the reaction mixture followed by flushing with argon balloon for 2 min to purge with argon atmosphere. The resulting mixture was stirred at room temperature for 18 h. After completion of the reaction, the mixture was purified by flash chromatography (1:2 - 2:1; EtOAc:hexane) to give primary amide **2**.



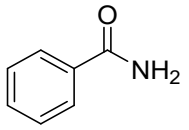
2a

4-Methoxybenzamide 2a[1]: Compound **2a** was prepared using general procedure **A** with the following reagents: Dioxazolone **1a** (0.0386 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2a** as a white solid (0.0296 g, 98%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.83 (d, J = 8.7 Hz, 3H), 7.18 (s, 1H), 6.95 (d, J = 8.7 Hz, 1H), 3.78 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.8, 162.0, 129.7, 126.9, 113.8, 55.7.



2b

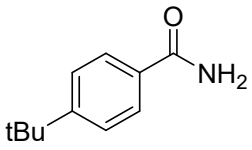
3-Methoxybenzamide 2b[2-3]: Compound **2b** was prepared using general procedure **A** with the following reagents: Dioxazolone **1b** (0.0386 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2b** as a white solid (0.0254 g, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.40 (d, J = 1.3 Hz, 1H), 7.33 (d, J = 4.9 Hz, 2H), 7.08 – 7.02 (m, 1H), 6.25 (s, 2H), 3.84 (s, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.0, 159.5, 136.1, 129.7, 120.1, 117.4, 113.0, 55.6.



2c

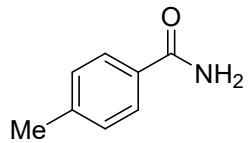
Benzamide 2c[1]: Compound **2c** was prepared using general procedure **A** with the following reagents: Dioxazolone **1c** (0.0386 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2c** as

a white solid (0.0208 g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.4 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 6.29 (s, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 169.7, 133.2, 132.0, 128.6, 127.3.



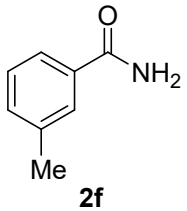
2d

4-(*tert*-Butyl)benzamide 2d[1]: Compound **2d** was prepared using general procedure **A** with the following reagents: Dioxazolone **1d** (0.0386 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2d** as a white solid (0.0344 g, 97%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.88 (s, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.26 (s, 1H), 1.27 (s, 9H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.2, 154.3, 131.9, 127.7, 125.3, 35.0, 31.3.



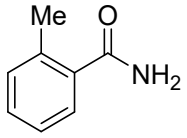
2e

4-Methylbenzamide 2e[1, 4]: Compound **2e** was prepared using general procedure **A** with the following reagents: Dioxazolone **1e** (0.0354 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2e** as a white solid (0.0268 g, 99%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 7.0 Hz, 2H), 6.00 (s, 1H), 5.62 (s, 1H), 2.41 (s, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.1, 141.4, 131.9, 129.1, 127.9, 21.3.



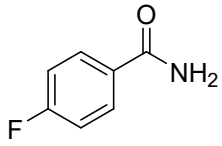
2f

3-Methylbenzamide 2f[5]: Compound **2f** was prepared using general procedure **A** with the following reagents: Dioxazolone **1f** (0.0354 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2f** as a white solid (0.0230 g, 85%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.60 – 7.56 (m, 1H), 7.35 – 7.31 (m, 2H), 2.40 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 170.0, 138.4, 133.2, 132.7, 128.4, 128.1, 124.3, 21.3.



2g

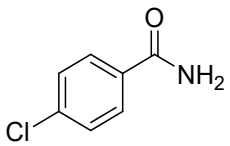
2-Methylbenzamide 2g[5]: Compound **2g** was prepared using general procedure **A** with the following reagents: Dioxazolone **1g** (0.0354 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2g** as a white solid (0.0219 g, 81%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.67 (s, 1H), 7.37 – 7.25 (m, 3H), 7.24 – 7.15 (m, 2H), 2.34 (s, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 171.4, 137.5, 135.5, 130.8, 129.5, 127.4, 125.8, 20.0.



2h

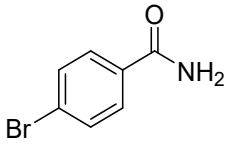
4-Fluorobenzamide 2h[1, 6]: Compound **2h** was prepared using general procedure **A** with the following reagents: Dioxazolone **1h** (0.0362 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction

mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2h** as a white solid (0.0195 g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.76 (m, 2H), 7.12 (t, *J* = 8.6 Hz, 2H), 6.03 (s, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.2, 164.3 (d, *J* = 248.3 Hz), 131.1 (d, *J* = 2.8 Hz), 130.5 (d, *J* = 9.0 Hz), 115.5 (d, *J* = 21.7 Hz).



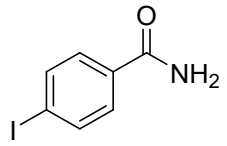
2i

4-Chlorobenzamide 2i[1]: Compound **2i** was prepared using general procedure **A** with the following reagents: Dioxazolone **1i** (0.0395 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2i** as a white solid (0.0296 g, 95%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.04 (s, 1H), 7.87 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.45 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.2, 136.5, 133.4, 129.8, 128.7.



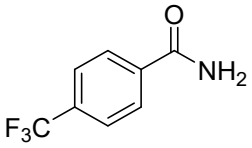
2j

4-Bromobenzamide 2j[1]: Compound **2j** was prepared using general procedure **A** with the following reagents: Dioxazolone **1j** (0.0484 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2j** as a white solid (0.0316 g, 79%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.60 (s, 1H), 7.37 (d, *J* = 7.0 Hz, 2H), 7.21 (d, *J* = 7.0 Hz, 2H), 7.01 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.3, 133.8, 131.6, 130.0, 125.4.



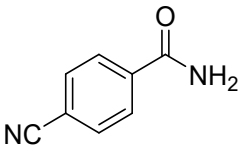
2k

4-Iodobenzamide 2k[1]: Compound **2k** was prepared using general procedure **A** with the following reagents: Dioxazolone **1k** (0.0578 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2k** as a white solid (0.0237 g, 48%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.00 (s, 1H), 7.82 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 8.4 Hz, 2H), 7.41 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.6, 137.5, 134.1, 129.9, 99.3.



2l

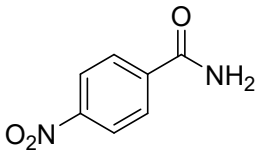
4-(Trifluoromethyl)benzamide 2l[1]: Compound **2l** was prepared using general procedure **A** with the following reagents: Dioxazolone **1l** (0.0462 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2l** as a white solid (0.0110 g, 29%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.18 (s, 1H), 8.05 (d, J = 8.1 Hz, 2H), 7.81 (d, J = 8.2 Hz, 2H), 7.61 (s, 1H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 167.1, 138.5, 131.5 (q, J = 31.8 Hz), 128.7, 125.6 (q, J = 4.0 Hz), 124.3 (q, J = 272.5 Hz)



2m

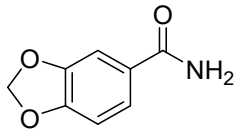
4-Cyanobenzamide 2m[7]: Compound **2m** was prepared using general procedure **A** with the following reagents: Dioxazolone **1m** (0.0376 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-

valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2m** as a white solid (0.0240 g, 82%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.20 (s, 1H), 8.00 (d, J = 8.6 Hz, 2H), 7.93 (d, J = 8.6 Hz, 2H), 7.66 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 166.9, 138.6, 132.8, 128.6, 118.8, 114.0.



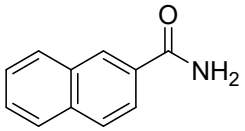
2n

4-Nitrobenzamide 2n[1]: Compound **2n** was prepared using general procedure **A** with the following reagents: Dioxazolone **1n** (0.0416 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2n** as a white solid (0.0326 g, 98%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.70 (s, 3H), 8.51 (s, 2H), 8.14 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6): δ 166.6, 149.4, 140.4, 129.3, 123.8.



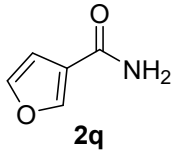
2o

Benzo[*d*][1,3]dioxole-5-carboxamide 2o[8-9]: Compound **2o** was prepared using general procedure **A** with the following reagents: Dioxazolone **1o** (0.0414 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2o** as a white solid (0.0314 g, 95%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 2H), 6.84 (d, J = 8.1 Hz, 2H), 6.03 (s, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.4, 150.1, 147.6, 128.7, 122.9, 108.1, 107.9, 102.0.



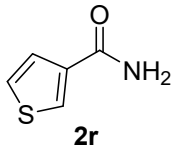
2p

2-Naphthamide 2p[1]: Compound **2p** was prepared using general procedure **A** with the following reagents: Dioxazolone **1p** (0.0426 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2p** as a white solid (0.0216 g, 63%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.47 (s, 1H), 8.13 (s, 1H), 8.02 – 7.90 (m, 4H), 7.63 – 7.53 (m, 2H), 7.46 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.4, 134.5, 132.5, 132.0, 129.3, 128.24, 128.20, 128.02, 128.00, 127.0, 124.8.



2q

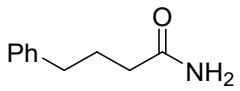
Furan-3-carboxamide 2q[10]: Compound **2q** was prepared using general procedure **A** with the following reagents: Dioxazolone **1q** (0.0306 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2q** as a white solid (0.0207 g, 93%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.13 (s, 1H), 7.68 (s, 1H), 7.62 (s, 1H), 7.17 (s, 1H), 6.78 (s, 1H).^{13}C-NMR (101 MHz, DMSO-d_6) δ 163.7, 145.7, 144.3, 123.3, 109.7.



2r

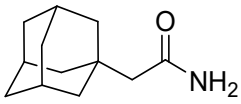
Thiophene-3-carboxamide 2r[1]: Compound **2r** was prepared using general procedure **A** with the following reagents: Dioxazolone **1r** (0.0338 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2r** as a white solid (0.0244 g, 96%). ^{1}H-NMR (DMSO-d_6) δ 8.11 (dd, J = 3.0, 1.3

Hz, 1H), 7.77 (s, 1H), 7.53 (dd, J = 5.0, 3.0 Hz, 1H), 7.46 (dd, J = 5.0, 1.3 Hz, 1H), 7.22 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 164.1, 138.4, 129.4, 127.5, 126.9.



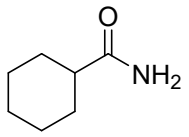
2s

4-Phenylbutanamide 2s[7]: Compound **2s** was prepared using general procedure **A** with the following reagents: Dioxazolone **1s** (0.0410 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2s** as a white solid (0.0274 g, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.28 (t, J = 7.3 Hz, 2H), 7.18 (t, J = 7.4 Hz, 3H), 6.11 (s, 1H), 5.61 (s, 1H), 2.66 (t, J = 7.5 Hz, 2H), 2.20 (t, J = 7.5 Hz, 2H), 2.02 – 1.90 (m, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 175.6, 141.4, 128.5, 128.4, 125.9, 35.1, 35.0, 26.8.



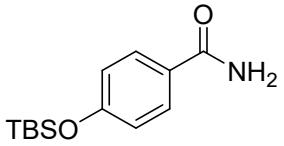
2t

2-((3*r*,5*r*,7*r*)-Adamantan-1-yl)acetamide 2t: Compound **2t** was prepared using general procedure **A** with the following reagents: Dioxazolone **1t** (0.0471 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **2t** as a white solid (0.0325 g, 84%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.11 (s, 1H), 6.62 (s, 1H), 1.89 (s, 3H), 1.77 (s, 2H), 1.72 – 1.45 (m, 12H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 172.6, 50.2, 42.5, 36.9, 32.3, 28.5. IR (neat): 3376, 3190, 2898, 1662, 1401, 1344, 1246, 1165, 990. HRMS(ESI) *m/z* calcd. for $C_{12}H_{20}NO$ $(M+H)^+$ 194.1539, observed 194.1546. m.p: 175 - 177 °C.



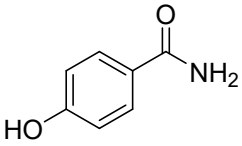
2u

Cyclohexanecarboxamide 2u[11]: Compound **2u** was prepared using general procedure **A** with the following reagents: Dioxazolone **1u** (0.0338 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2u** as a white solid (0.0201 g, 79%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.12 (s, 1H), 6.60 (s, 1H), 2.02 (ddd, J = 11.5, 7.3, 3.1 Hz, 1H), 1.80 – 1.51 (m, 5H), 1.43 – 0.87 (m, 5H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 177.8, 44.1, 29.6, 25.9, 25.7.



2v

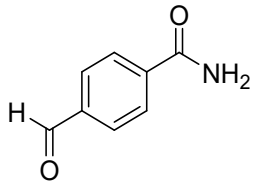
4-((*tert*-Butyldimethylsilyl)oxy)benzamide 2v[1]: Compound **2v** was prepared using general procedure **A** with the following reagents: Dioxazolone **1v** (0.0587 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2v** as a white solid (0.0352 g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.7 Hz, 2H), 6.24 (s, 1H), 0.97 (s, 9H), 0.21 (s, 6H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 169.3, 159.1, 129.2, 126.3, 119.9, 25.5, 18.2, -4.4.



2w

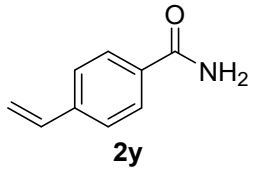
4-Hydroxybenzamide 2w[1]: Compound **2w** was prepared using general procedure **A** with the following reagents: Dioxazolone **1w** (0.0358 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-

valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2w** as a white solid (0.0244 g, 89%). ^{1}H-NMR(400 MHz, DMSO-d_6) δ 9.94 (s, 1H), 7.72 (d, J = 8.7 Hz, 3H), 7.06 (s, 1H), 6.93 – 6.67 (m, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.1, 160.5, 129.8, 125.3, 115.1.



2x

4-Formylbenzamide 2x: Compound **2x** was prepared using general procedure **A** with the following reagents: Dioxazolone **1x** (0.0382 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2x** as a white solid (0.0224 g, 75%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 10.06 (s, 1H), 8.17 (s, 1H), 8.04 (d, J = 8.3 Hz, 2H), 7.96 (d, J = 8.5 Hz, 2H), 7.60 (s, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 193.3, 167.5, 139.7, 138.2, 129.7, 128.5. IR (neat): 3375, 3166, 2811, 1654, 1506, 1401, 1208, 1121, 1012, 759. HRMS(ESI) *m/z* calcd. for $C_8H_8NO_3$ $(M+H_2O-H)^-$ 166.0510, observed 166.0582. m.p: 165 - 167 °C.

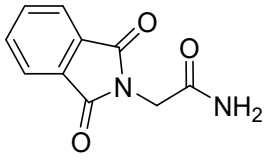


2y

4-Vinylbenzamide 2y[12]: Compound **2y** was prepared using general procedure **A** with the following reagents: Dioxazolone **1y** (0.0378 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2y** as a white solid (0.0177 g, 60%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.95 (s, 1H), 7.84 (d, J = 8.3 Hz, 2H), 7.52 (d, J = 8.3 Hz, 2H), 7.34 (s, 1H), 6.76 (dd, J = 17.7, 11.0 Hz, 1H), 5.93 (d, J = 17.7 Hz,

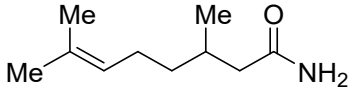
1H), 5.34 (d, J = 11.4 Hz, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 172.6, 144.9, 141.1, 138.6, 133.0, 131.0, 121.3.

V. Synthesis of Primary Amides Containing of Bioactive Motif 4a – 4f (Scheme 3)



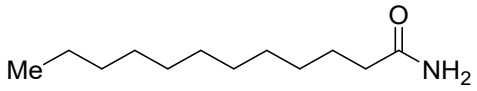
4a

2-(1,3-Dioxoisoindolin-2-yl)acetamide 4a[13]: Compound **4a** was prepared using general procedure **A** with the following reagents: Dioxazolone **3a** (0.0492 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **4a** as a white solid (0.0155 g, 38%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.03 – 7.79 (m, 4H), 7.67 (s, 1H), 7.24 (s, 1H), 4.13 (s, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.3, 168.0, 134.9, 132.1, 123.6.



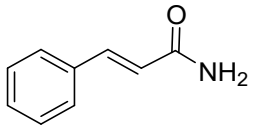
4b

3,7-Dimethyloct-6-enamide 4b[14]: Compound **4b** was prepared using general procedure **A** with the following reagents: Dioxazolone **3b** (0.0423 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 - 2:1; EtOAc:hexane) afforded **4b** as a white solid (0.0135 g, 40%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.21 (s, 1H), 6.67 (s, 1H), 5.05 (t, J = 7.1 Hz, 1H), 2.00 (d, J = 7.6 Hz, 1H), 1.97 – 1.86 (m, 2H), 1.84 – 1.74 (m, 2H), 1.62 (s, 3H), 1.54 (s, 3H), 1.37 – 0.98 (m, 2H), 0.83 (d, J = 6.3 Hz, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 174.1, 130.9, 124.9, 43.2, 36.9, 29.9, 25.9, 25.4, 19.8, 17.9.



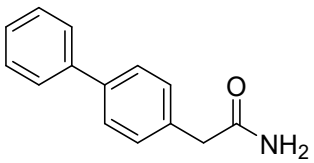
4c

Dodecanamide 4c[15]: Compound **4c** was prepared using general procedure **A** with the following reagents: Dioxazolone **3c** (0.0483 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **4c** as a white solid (0.0375 g, 94%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.19 (s, 1H), 6.64 (s, 1H), 1.99 (t, J = 7.5 Hz, 1H), 1.59 – 1.37 (m, 1H), 1.22 (s, 8H), 0.84 (t, J = 6.8 Hz, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 174.7, 35.5, 31.7, 29.5, 29.4, 29.3, 29.2, 29.1, 25.5, 22.5, 14.3.



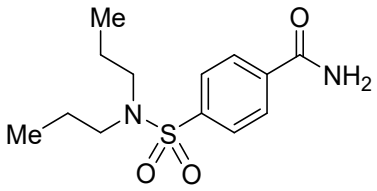
4d

Cinnamamide 4d[1]: Compound **4d** was prepared using general procedure **A** with the following reagents: Dioxazolone **3d** (0.0378 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **4d** as a white solid (0.0277 g, 94%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.58 – 7.49 (m, 3H), 7.45 – 7.30 (m, 4H), 7.10 (s, 1H), 6.60 (d, J = 15.9 Hz, 1H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.1, 139.6, 135.3, 129.8, 129.3, 127.9, 122.7.



4e

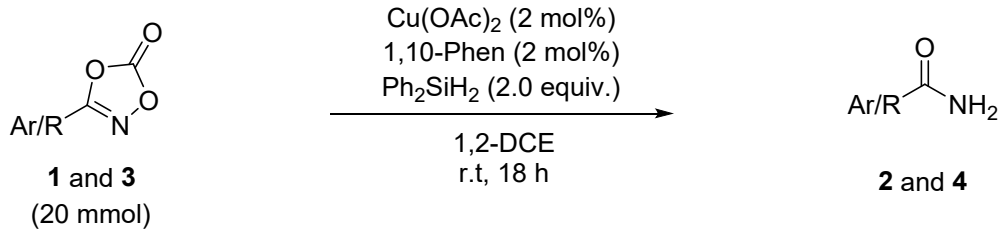
2-((1,1'-Biphenyl)-4-yl)acetamide 4e[16]: Compound **4e** was prepared using general procedure **A** with the following reagents: Dioxazolone **3e** (0.0507 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **4e** as a white solid (0.0127 g, 30%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.65 – 7.61 (m, 2H), 7.59 – 7.56 (m, 2H), 7.49 (s, 1H), 7.44 (dd, J = 10.4, 4.8 Hz, 2H), 7.35 – 7.34 (m, 1H), 7.33 – 7.30 (m, 2H), 6.89 (s, J = 16.3 Hz, 1H), 3.40 (s, 2H).



4f

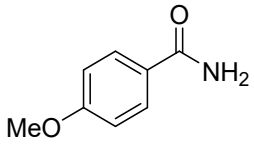
4-(*N*,*N*-Dipropylsulfamoyl)benzamide 4f[7]: Compound **4f** was prepared using general procedure **A** with the following reagents: Dioxazolone **3f** (0.0653 g, 0.200 mmol), diphenylsilane (0.074 mL, 0.400 mmol), ɤ-valerolactone (1.0 mL), $Cu(OAc)_2$ (0.0007 g, 0.004 mmol) and 1,10-phenanthroline (0.0007 g, 0.004 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **4f** as a white solid (0.0483 g, 85%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 8.17 (s, 1H), 8.02 (d, J = 8.2 Hz, 2H), 7.85 (d, J = 8.2 Hz, 2H), 7.60 (s, 1H), 3.10 – 2.86 (m, 4H), 1.44 (dd, J = 14.8, 7.4 Hz, 4H), 0.78 (t, J = 7.3 Hz, 6H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 167.1, 142.1, 138.2, 128.8, 127.1, 50.0, 22.0, 11.4.

VI. Gram-scale Synthesis of Primary Amides (Scheme 4)



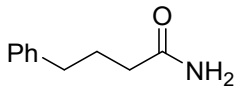
1 and **3**
(20 mmol)

2 and **4**



2a

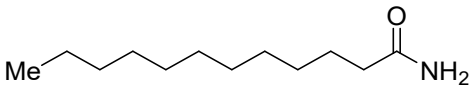
4-Methoxybenzamide 2a[1]: Compound **2a** was prepared using general procedure **B** with the following reagents: Dioxazolone **1a** (3.8632 g, 20.0 mmol), diphenylsilane (7.42 mL, 40.0 mmol), 1,2-dichloroethane (100 mL), $Cu(OAc)_2$ (0.0727 g, 0.40 mmol) and 1,10-phenanthroline (0.0721 g, 0.40 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2a** as a white solid (2.9231 g, 97%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.71 (d, J = 8.1 Hz, 2H), 7.25 (d, J = 7.0 Hz, 2H), 6.00 (s, 1H), 5.62 (s, 1H), 2.41 (s, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 168.1, 141.4, 131.9, 129.1, 127.9, 21.3.



2s

4-Phenylbutanamide 2s[7]: Compound **2s** was prepared using general procedure **B** with the following reagents: Dioxazolone **1s** (4.1042 g, 20.0 mmol), diphenylsilane (7.42 mL, 40.0 mmol), 1,2-dichloroethane (100 mL), $Cu(OAc)_2$ (0.0727 g, 0.40 mmol) and 1,10-phenanthroline (0.0721 g, 0.40 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2s** as a white solid (2.2941 g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.28 (t, J = 7.3 Hz, 2H), 7.18

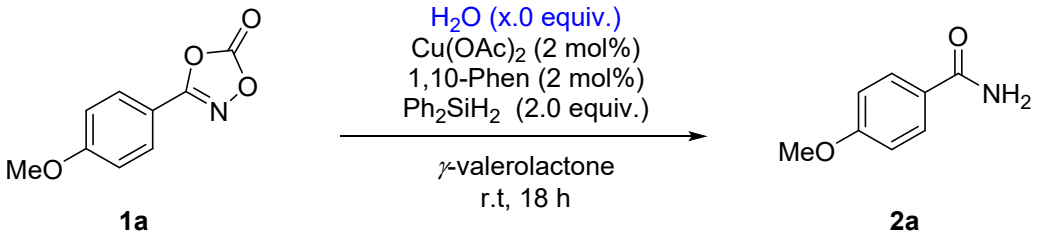
(t, J = 7.4 Hz, 3H), 6.11 (s, 1H), 5.61 (s, 1H), 2.66 (t, J = 7.5 Hz, 2H), 2.20 (t, J = 7.5 Hz, 2H), 2.02 – 1.90 (m, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 175.6, 141.4, 128.4, 128.3, 125.9, 35.1, 35.0, 26.8.



4c

Dodecanamide 4c[15]: Compound **2c** was prepared using general procedure **B** with the following reagents: Dioxazolone **1c** (4.8266 g, 20.0 mmol), diphenylsilane (7.42 mL, 40.0 mmol), 1,2-dichloroethane (100 mL), $Cu(OAc)_2$ (0.0727 g, 0.40 mmol) and 1,10-phenanthroline (0.0721 g, 0.40 mmol). The reaction mixture was stirred for 18 h at room temperature. Flash chromatography (1:2 – 2:1; EtOAc:hexane) afforded **2c** as a white solid (3.1942 g, 80%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 7.19 (s, 1H), 6.64 (s, 1H), 1.99 (t, J = 7.5 Hz, 1H), 1.59 – 1.37 (m, 1H), 1.22 (s, 8H), 0.84 (t, J = 6.8 Hz, 2H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 174.7, 35.5, 31.7, 29.5, 29.4, 29.3, 29.2, 29.1, 25.5, 22.5, 14.3.

VII. Catalytic Performance in the Presence of H_2O (Scheme 5)

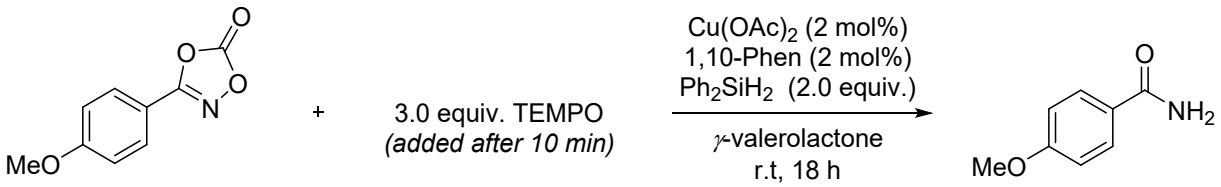


1a **2a**

A conical vial (5.0 mL) was charged with $Cu(OAc)_2$ (0.0007 g, 0.004 mmol), 1,10-phenanthroline (0.0007 g, 0.004 mmol), ɤ-valerolactone (1.0 mL) and H_2O (0.200 mmol, 3.6 µL)**.** The reaction mixture as flushed with argon balloon for 2 min to purge with argon atmosphere. The reaction vial was capped rapidly, and the mixture was stirred at room temperature for 15 min. Diphenylsilane (0.074 mL, 0.400 mmol) was then added via syringe, and the resulting mixture was stirred at room temperature for additional 20 min until the solution turned from green to a dark orange color. Dioxazolone **1a** (0.0386 g, 0.200 mmol) was added to the reaction mixture followed by flushing with argon balloon for 2 min to purge with argon atmosphere. The resulting mixture was stirred at room temperature for 18 h. After completion of the reaction, the mixture

was extracted with CH_2Cl_2 and dried with $MgSO_4$, which was purified by flash chromatography (1:2 – 2:1; EtOAc:hexane) to give primary amide **2a**.

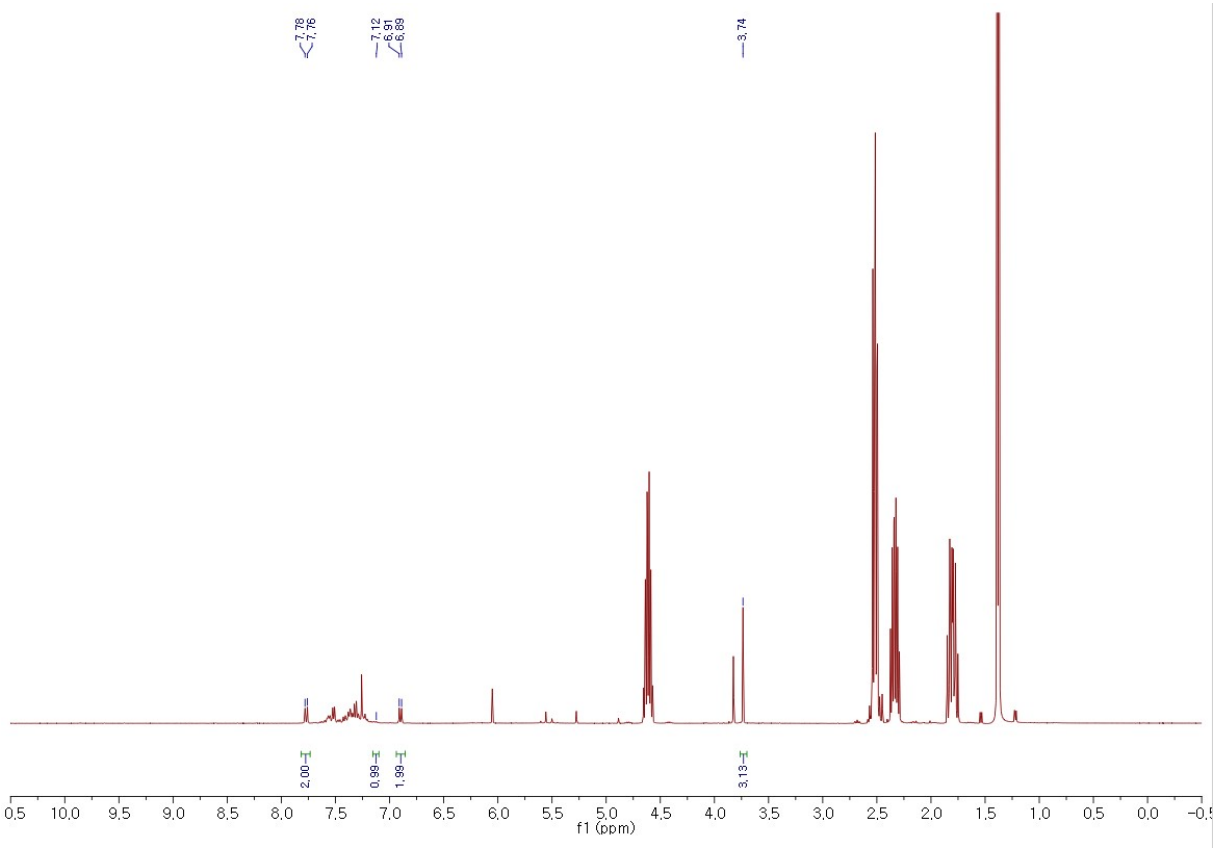
VIII. Radical Trapping Experiment



1a **2a**

A conical vial (5.0 mL) was charged with $Cu(OAc)_2$ (0.0007 g, 0.004 mmol), 1,10-phenanthroline (0.0007 g, 0.004 mmol) and ɤ-valerolactone (1.0 mL)**.** The reaction mixture was flushed with argon balloon for 2 min to purge with argon atmosphere. The reaction vial was capped rapidly, and the mixture was stirred at room temperature for 15 min. Diphenylsilane (0.074 mL, 0.400 mmol) was then added via syringe, and the resulting mixture was stirred at room temperature for additional 20 min until the solution turned from green to a dark orange color. Dioxazolone **1a** (0.0386 g, 0.200 mmol) was added to the reaction mixture followed by flushing with argon balloon for 2 min to purge with argon atmosphere and was stirred at room temperature for 10 min. 2,2,6,6-Tetramethylpiperidinooxy (TEMPO) (0.0938 g, 0.600 mmol) was added and stirred at room temperature for 18 h. Flash chromatography (1:2 – 2:1; EtOAc:hexane) to give primary amide **2a** (0.0160 g, 53%).

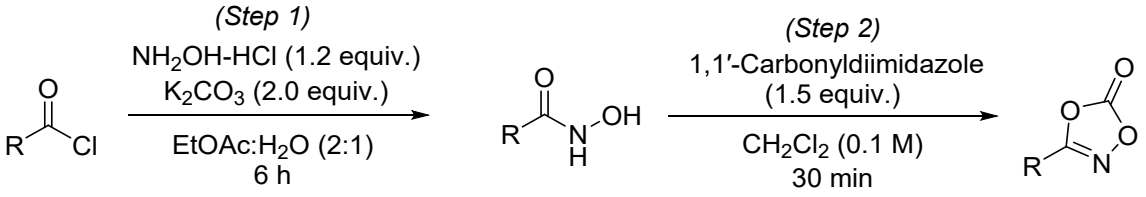
IX. Crude ^{1}H-NMR of Standard Reaction



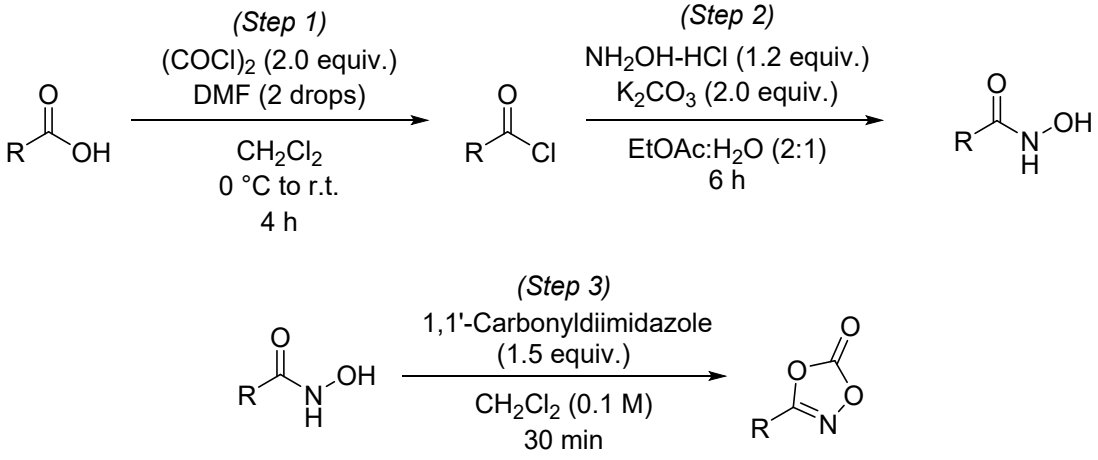
→ The formation of silyl formate was not observed, implying the catalytic reduction of CO_2 by copper hydride species was not favorable in this transformation.

IX. Preparation of Dioxazolones 1a – 1y and 3a – 3f

Three general procedures **C**, **D**, and **E** are shown below, used to synthesize the dioxazolones.

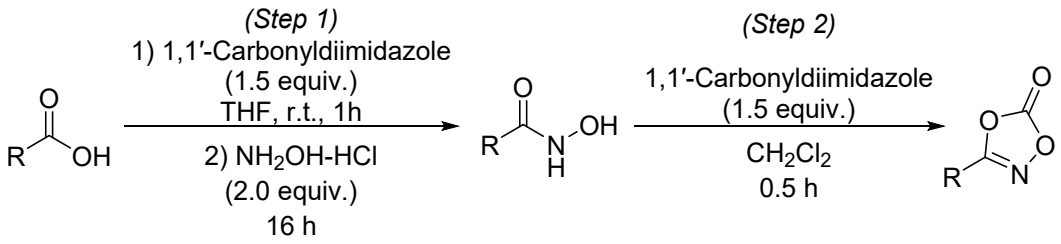


General procedure C. Step 1: A 250 mL round bottom flask was charged with K_2CO_3 (20.0 mmol), EtOAc (80 mL), and water (40 mL). At 0 °C, hydroxylamine hydrochloride (1.20 mmol) was added, then acid chloride (10.0 mmol) was dropwised slowly. The resulting solution was stirred at room temperature for 6 h. The organic phase was separated, and the aqueous phase was extracted with EtOAc (40 mL x 2). The combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude products were used in the next step without any further purification. **Step 2**: The hydroxamic acid (1.0 equiv.) was dissolved in 0.1 M CH_2Cl_2, followed by the addition of 1,1'-carbonyldiimidazole (CDI) (1.5 equiv.). After stirring at room temperature for 30 min, the solution was diluted with 1 N aqueous HCl solution and extracted with CH_2Cl_2 (2 times). The combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The crude mixture was purified by filtration over a short plug of silica with CH_2Cl_2 to give the product.

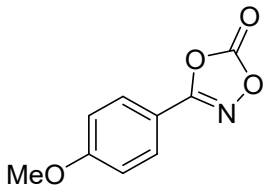


General procedure D. **Step 1**: At 0 °C, oxalyl chloride (4.0 mmol), and DMF (2 drops) were added to a solution of the carboxylic acid (2.0 mmol) in CH_2Cl_2 (30 mL). The mixture was stirred at room temperature for 4 h, then the reaction mixture was concentrated. The crude product was used directly in the next reaction.

Step 2: Hydroxylamine hydrochloride (1.2 equiv.) was added to a biphasic mixture of K_2CO_3 (2.0 equiv.) in a 2:1 mixture of EtOAc (16 mL) and H_2O (8 mL). The resulting solution was cooled to 0 °C followed by addition of the acid chloride (from step 1) dissolved in a minimum amount of EtOAc. The reaction was warmed to room temperature with additional stirring for 12 h. The phases were separated and the aqueous phase was extracted with EtOAc (2 times). The combined organic layers were dried over $MgSO_4$, filtered, and evaporated under reduced pressure. The crude products were used in the next step without any further purification. **Step 3**: The hydroxamic acid (1.0 equiv.) was dissolved in 0.1 M CH_2Cl_2, followed by the addition of carbonyldiimidazole (1.5 equiv.). After stirring at room temperatures for 30 min, the solution was diluted with 1 N aqueous HCl solution and extracted with CH_2Cl_2 (2 times). The combined organic layer was dried over Mg_2SO_4, and concentrated under reduced pressure. The crude mixture was purified by filtration over a short plug of silica with CH_2Cl_2 to give the product.

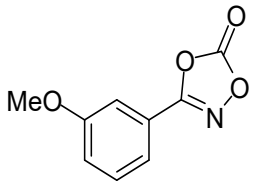


General procedure E. Step 1: 1,1'-Carbonyldiimidazole (CDI) (15.0 mmol, 1.5 equiv.) was added to a solution of carboxylic acid (10.0 mmol) in 30 mL of anhydrous tetrahydrofuran. The reaction mixture was stirred for 1 h. Hydroxylamine hydrochloride (1.39 g, 20.0 mmol) was added and the resulting mixture was stirred overnight. The mixture was diluted with 5% aq. $KHSO_4$ (30 mL) and extracted with EtOAc (2 x 30 mL). The combined organic phase was washed with brine (50 mL) and dried over $MgSO_4$. The extract was filtered and concentrated under reduced pressure. The crude product was used in the next step without any further purification. **Step 2**: Hydroxamic acid (from step 1) was dissolved in dichloromethane (1.0 M), followed by the addition of carbonyldiimidazole (1.5 equiv.). After stirring for 30 min at room temperature, the solution was diluted with 1 N aqueous HCl solution and extracted with dichloromethane (2 times). The combined organic layers were dried over magnesium sulfate, and concentrated under reduced pressure. The crude mixture was purified by filtration over a short plug of silica with dichloromethane to give the product.



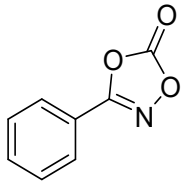
1a

3-(4-Methoxyphenyl)-1,4,2-dioxazol-5-one 1a[17] was prepared according to the general procedure **C**, resulting in white solid (1.55 g, 80%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 9.0 Hz, 2H), 7.02 (d, *J* = 9.0 Hz, 2H), 3.89 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.8, 163.3, 154.0, 128.5, 114.8, 112.0, 55.6.



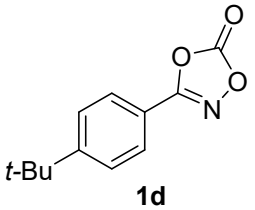
1b

3-(3-Methoxyphenyl)-1,4,2-dioxazol-5-one 1b[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.41 g, 73%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.43 (m, 2H), 7.33 (s, 1H), 7.21 – 7.13 (m, 1H), 3.87 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.4, 160.0, 153.7, 130.5, 121.1, 120.2, 119.0, 111.0, 55.5.



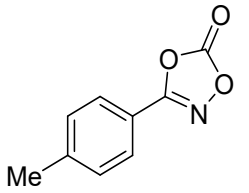
1c

3-Phenyl-1,4,2-dioxazol-5-one 1c[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.44 g, 88%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.3 Hz, 2H), 7.66 (t, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.6, 153.9, 133.9, 129.5, 126.7, 120.2.



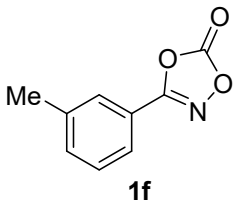
1d

3-(4-(*tert*-Butyl)phenyl)-1,4,2-dioxazol-5-one 1d[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.712 g, 65%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.6 Hz, 2H), 7.56 (d, *J* = 8.6 Hz, 2H), 1.36 (s, 9H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.5, 157.8, 153.9, 126.4, 126.4, 117.1, 35.3, 30.9



1e

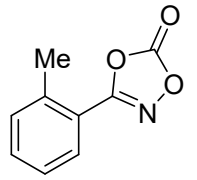
3-(*p*-Tolyl)-1,4,2-dioxazol-5-one 1e[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.36 g, 77%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 2.45 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.6, 153.9, 144.8, 130.0, 126.5, 117.2, 21.8.



1f

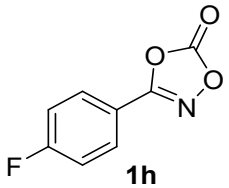
3-(*m*-Tolyl)-1,4,2-dioxazol-5-one 1f[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.06 g, 60%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 9.0 Hz, 2H), 7.49 – 7.39

(m, 2H), 2.44 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.6, 153.9, 139.4, 134.6, 129.2, 126.9, 123.8, 119.9, 21.3.



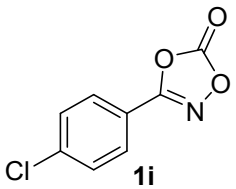
1g

3-(*o*-Tolyl)-1,4,2-dioxazol-5-one 1g[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.974 g, 55%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79 – 7.73 (m, 1H), 7.52 (td, *J* = 7.7 Hz, 1.2 Hz, 1H), 7.37 (t, *J* = 7.1 Hz, 2H), 2.60 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.9, 153.6, 139.1, 133.1, 132.0, 128.7, 126.4, 119.1, 22.1.



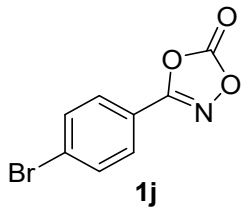
1h

3-(4-Fluorophenyl)-1,4,2-dioxazol-5-one 1h[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.652 g, 71%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 165.8 (d, *J* = 256.9 Hz), 162.7, 153.6, 129.1 (d, *J* = 9.4 Hz), 117.10, 116.6 (d, *J* = 53.5 Hz).



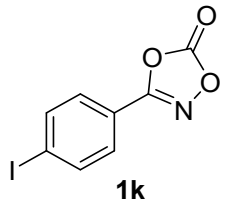
1i

3-(4-Chlorophenyl)-1,4,2-dioxazol-5-one 1i[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.28 g, 65%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 162.8, 153.5, 140.3, 129.8, 127.8, 118.5.



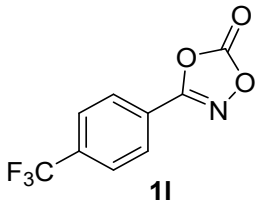
1j

3-(4-Bromophenyl)-1,4,2-dioxazol-5-one 1j[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (1.69 g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.57 (m, 4H). ^{13}C -NMR (101 MHz, $CDCl_3$) δ 162.9, 153.4, 132.8, 128.8, 127.9, 118.9.



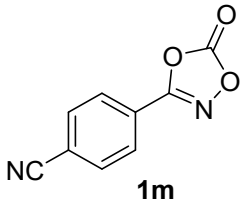
1k

3-(4-Iodophenyl)-1,4,2-dioxazol-5-one 1k[18] was prepared according to the reaction in general procedure **C**, resulting in white solid (1.30 g, 45%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.83 (d, *J* = 8.3 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.1, 153.4, 138.7, 127.7, 119.4, 101.3.



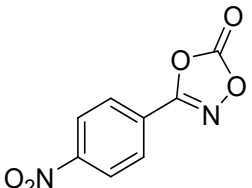
1l

3-(4-(Trifluoromethyl)phenyl)-1,4,2-dioxazol-5-one 1l[19] was prepared according to the reaction in general procedure **E**, resulting in white solid (0.693 g, 30%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.83 (d, *J* = 8.3 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 162.4, 153.2, 135.3 (q, *J* = 33.7 Hz), 127.1, 126.4 (q, *J* = 3.8 Hz), 123.4, 123.1 (q, *J* = 272.8 Hz).



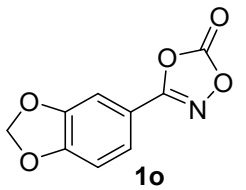
1m

4-(5-Oxo-1,4,2-dioxazol-3-yl)benzonitrile 1m[20] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.677 g, 36%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.7 Hz, 1H), 7.86 (d, *J* = 8.7 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 162.1, 153.0, 133.1, 127.1, 124.0, 117.3, 117.1.



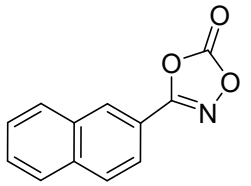
1n

3-(4-Nitrophenyl)-1,4,2-dioxazol-5-one 1n[17] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.779 g, 80%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 9.0 Hz, 2H), 8.09 (d, *J* = 9.0 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 161.9, 152.9, 150.7, 127.8, 125.6, 124.6.



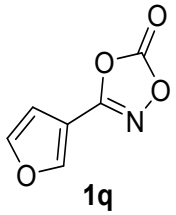
1o

3-(Benzo[*d*][1,3]dioxol-5-yl)-1,4,2-dioxazol-5-one 1o[17] was prepared according to the reaction in general procedure **E**, resulting in white solid (0.850 g, 82%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.1 Hz, 1H), 7.24 (s, 1H), 6.93 (d, *J* = 8.2 Hz, 1H), 6.10 (s, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.1, 153.8, 152.3, 148.6, 122.5, 113.3, 109.0, 106.0, 102.3.



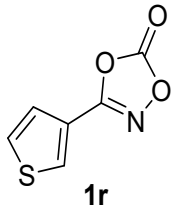
1p

3-(Naphthalen-2-yl)-1,4,2-dioxazol-5-one 1p[17] was prepared according to the reaction in general procedure **E**, resulting in white solid (1.49 g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 8.02 – 7.89 (m, 3H), 7.86 (dd, *J* = 8.6 Hz, 1.7 Hz, 1H), 7.64 (dd, *J* = 14.7, 7.0, 1.3 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.6, 153.8, 135.4, 132.3, 129.5, 129.14, 129.10, 128.3, 128.0, 127.6, 121.4, 117.1.



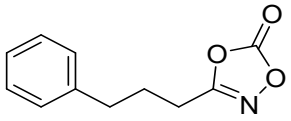
1q

3-(Furan-3-yl)-1,4,2-dioxazol-5-one 1q[17] was prepared according to the reaction in general procedure **E**, resulting in white solid (1.32 g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.59 (d, *J* = 1.7 Hz, 1H), 6.79 (d, *J* = 1.2 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 159.2, 153.4, 145.4, 145.2, 108.1, 107.1.



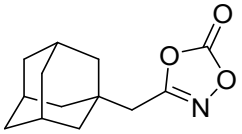
1r

3-(Thiophen-3-yl)-1,4,2-dioxazol-5-one 1r[18] was prepared according to the reaction in general procedure **E**, resulting in white solid (1.27 g, 75%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 2.8, 1.3 Hz, 1H), 7.51 (qd, *J* = 5.2, 2.1 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 160.3, 153.5, 130.4, 128.5, 124.6, 120.9.



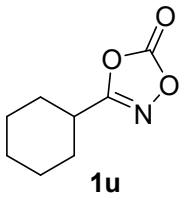
1s

3-(3-Phenylpropyl)-1,4,2-dioxazol-5-one 1s[17] was prepared according to the reaction in general procedure **D**, resulting in colorless liquid (1.21 g, 59%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.4 Hz, 2H), 7.25 (t, *J* = 7.4 Hz, 1H), 7.20 (d, *J* = 7.3 Hz, 2H), 2.76 (t, *J* = 7.4 Hz, 2H), 2.63 (t, *J* = 7.5 Hz, 2H), 2.07 (p, *J* = 7.5 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 166.4, 154.1, 139.8, 128.6, 128.4, 126.5, 34.5, 25.8, 24.0.



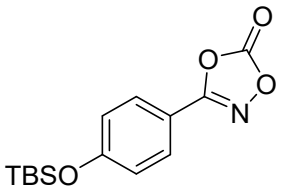
1t

3-(((3*r*,5*r*,7r)-Adamantan-1-yl)methyl)-1,4,2-dioxazol-5-one 1t[17] was prepared according to the reaction in general procedure **E**, resulting in white solid (2.00 g, 85%). ^{1}H-NMR (400 MHz, $CDCl_3$) 164.9, 154.3, 42.0, 38.8, 36.2, 33.1, 28.3. δ 2.37 (s, 2H), 2.02 (s, 3H), 1.68 (d, *J* = 26.2 Hz, 6H), 1.60 (d, *J* = 2.3 Hz, 6H).



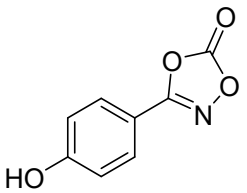
1u

3-Cyclohexyl-1,4,2-dioxazol-5-one 1u[19] was prepared according to the reaction in general procedure **E**, resulting in white solid (1.45 g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.67 (tt, *J* = 11.2, 3.6 Hz, 1H), 1.99 (ddd, *J* = 6.7, 5.8, 3.3 Hz, 2H), 1.92 – 1.80 (m, 2H), 1.78 – 1.66 (m, 1H), 1.60 – 1.45 (m, 2H), 1.33 (s, 2H), 1.32 – 1.20 (m, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 195.9, 180.9, 61.3, 54.8, 51.8, 51.5.



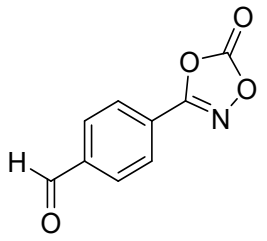
1v

3-(4-((*tert*-Butyldimethylsilyl)oxy)phenyl)-1,4,2-dioxazol-5-one 1v[17] was prepared according to the reaction in general procedure **E**, resulting in white solid (0.935 g, 32%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.6 Hz, 2H), 6.96 (d, *J* = 8.6 Hz, 2H), 0.99 (s, 9H), 0.25 (s, 6H).



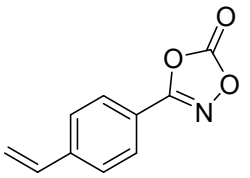
1w

3-(4-Hydroxyphenyl)-1,4,2-dioxazol-5-one 1w[21] was prepared according to the reaction in general procedure **E**, resulting in white solid (0.134 g, 15%). ^{1}H-NMR (400 MHz, DMSO-d_6) δ 10.62 (s, 1H), 7.66 (d, *J* = 8.8 Hz, 3H), 6.95 (d, *J* = 8.8 Hz, 3H). ^{13}C-NMR (101 MHz, DMSO-d_6) δ 163.5, 162.6, 154.5, 129.0, 116.8, 110.8.



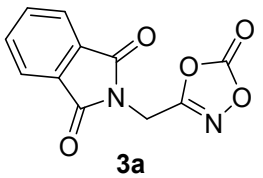
1x

4-(5-Oxo-1,4,2-dioxazol-3-yl)benzaldehyde 1x was prepared according to the reaction in general procedure **E**, resulting in white solid (0.612 g, 32%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.12 (s, 1H), 8.20 – 7.85 (m, 4H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 190.7, 162.6, 153.3, 139.6, 130.2, 127.3, 125.0. IR (neat): 2843, 1833, 1698, 1608, 1364, 1173, 1067, 969, 833, 748. HRMS(ESI) *m/z* calcd. for $C_8H_7NO_3$ $(M\text{-}CO_2)^+$ 165.0426, observed 165.0550. m.p: 115 - 117 °C.



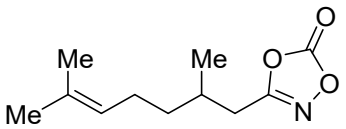
1y

3-(4-Vinylphenyl)-1,4,2-dioxazol-5-one 1y was prepared according to the reaction in general procedure **E**, resulting in white solid (0.935 g, 32%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.5 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 6.76 (dd, J = 17.6, 10.9 Hz, 1H), 5.92 (d, J = 17.6 Hz, 1H), 5.46 (d, J = 10.9 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.3, 153.8, 142.8, 135.4, 127.0, 126.8, 118.9, 117.7. IR (neat): 1828, 1606, 1509, 1352, 1164, 1067, 968, 848, 746, 676. HRMS(ESI) *m/z* calcd. for $C_9H_9NNaO_2$ $(M\text{-}CO_2\text{+}Na)^+$ 186.0531, observed 186.0525. m.p: 87 - 89 °C.



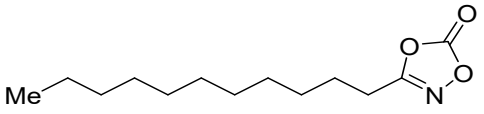
3a

2-((5-Oxo-1,4,2-dioxazol-3-yl)methyl)isoindoline-1,3-dione 3a[22] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.985 g, 40%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.90 (m, 2H), 7.84 – 7.77 (m, 2H), 4.89 (s, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 166.4, 161.3, 153.0, 134.8, 131.4, 124.1, 31.6.



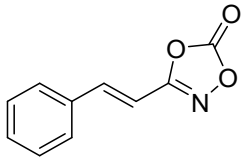
3b

3-(2,6-Dimethylhept-5-en-1-yl)-1,4,2-dioxazol-5-one 3b[23] was prepared according to the reaction in general procedure **D**, resulting in white solid resulting in white solid (1.450 g, 69%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 5.12 – 5.00 (m, 1H), 2.61 (dd, *J* = 15.2 Hz, 5.8 Hz, 1H), 2.44 (dd, *J* = 15.2 Hz, 8.0 Hz, 1H), 2.11 – 1.88 (m, 3H), 1.69 (d, *J* = 0.9 Hz, 3H), 1.61 (s, 3H), 1.48 – 1.26 (m, 2H), 1.02 (d, *J* = 6.7 Hz, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 166.0, 154.1, 132.3, 123.3, 36.2, 31.7, 29.7, 25.6, 25.1, 19.2, 17.6.



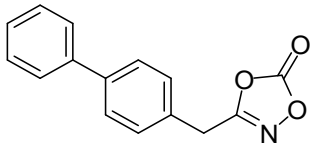
3c

3-Undecyl-1,4,2-dioxazol-5-one 3c[21] was prepared according to the reaction in general procedure **D**, resulting in white solid resulting in white solid (1.010 g, 42%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.65 – 2.57 (m, 2H), 1.71 (dt, *J* = 15.2, 7.5 Hz, 2H), 1.45 – 1.20 (m, 16H), 0.87 (t, *J* = 6.9 Hz, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 166.7, 154.2, 31.8, 29.5, 29.4, 29.2, 28.9, 28.6, 24.7, 24.4, 22.6, 14.0.



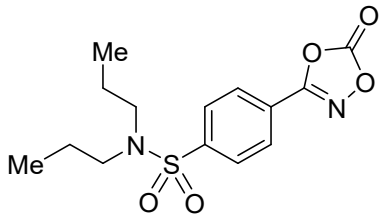
3d

(*E*)-3-Styryl-1,4,2-dioxazol-5-one 3d[18] was prepared according to the reaction in general procedure **D**, resulting in white solid resulting in white solid (0.387 g, 20%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 4.8, 1.3 Hz, 1H), 7.51 (d, *J* = 17.0 Hz, 1H), 7.45 (dd, *J* = 5.1, 1.9 Hz, 3H), 6.65 (d, *J* = 16.4 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 163.3, 153.4, 143.0, 133.4, 131.2, 129.2, 128.0, 105.8.



3e

3-([1,1'-Biphenyl]-4-ylmethyl)-1,4,2-dioxazol-5-one 3e was prepared according to the reaction in general procedure **D**, resulting in white solid resulting in white solid (0.253 g, 20%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.60 (m, 2H), 7.60 – 7.56 (m, 2H), 7.49 – 7.43 (m, 2H), 7.42 – 7.37 (m, 2H), 7.36 (d, *J* = 1.8 Hz, 1H), 3.98 (s, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 165.2, 153.9, 141.5, 140.1, 129.4, 129.2, 128.8, 127.9, 127.6, 127.1, 30.9. IR (neat): 2852, 1860, 1633, 1485, 1390, 1257, 1156, 991, 824, 733. HRMS(ESI) *m/z* calcd. for $C_{14}H_{12}NO$ $(M\text{-}CO_2\text{-}H)^-$ 210.0924, observed 210.0922. m.p: 75 - 77 °C.



3f

4-(5-Oxo-1,4,2-dioxazol-3-yl)-*N*,*N*-dipropylbenzenesulfonamide 3f[24] was prepared according to the reaction in general procedure **D**, resulting in white solid (0.895 g, 30%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.95 (m, 4H), 3.17 – 3.07 (m, 4H), 1.56 (dq, *J* = 14.9 Hz, 7.4 Hz, 4H), 0.87 (t, *J* = 7.4 Hz, 6H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 162.4, 153.2, 145.4, 127.8, 127.2, 123.3, 49.8, 21.9, 11.1.

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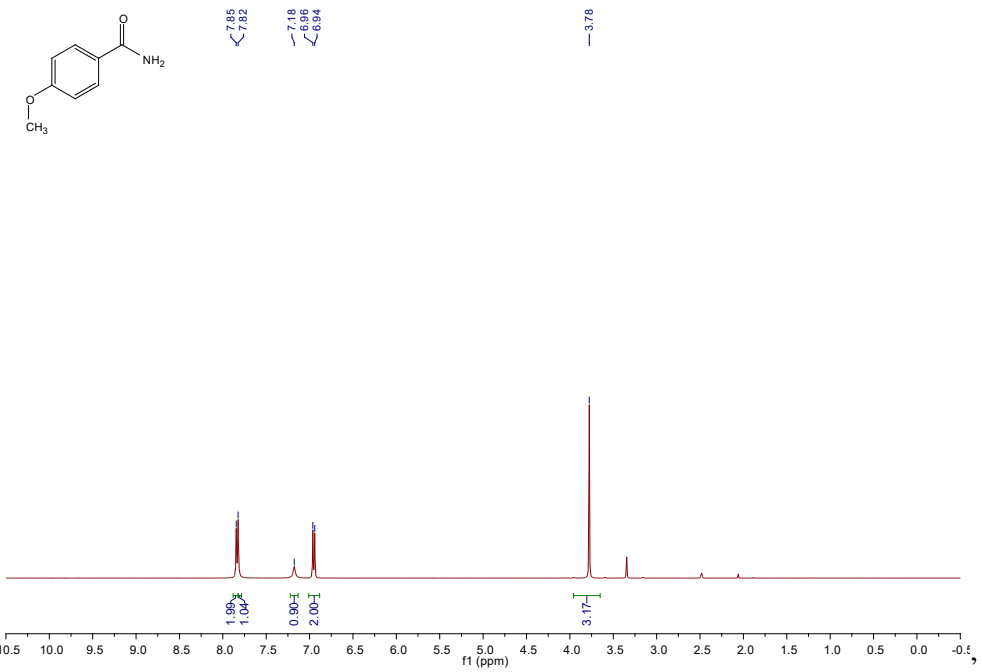
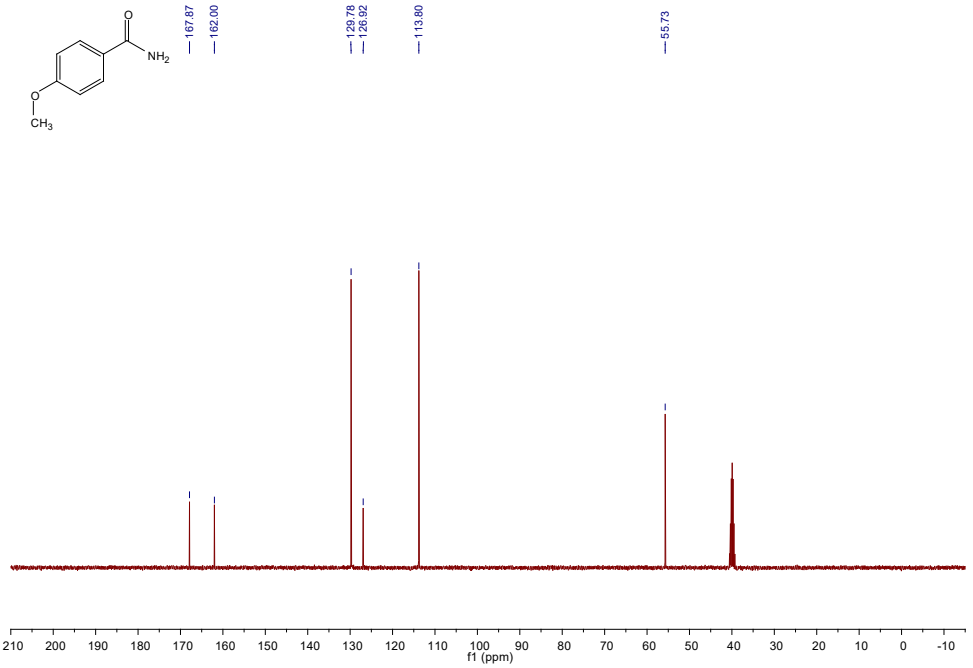
XI. ^{1}H- and ^{13}C-NMR Spectra

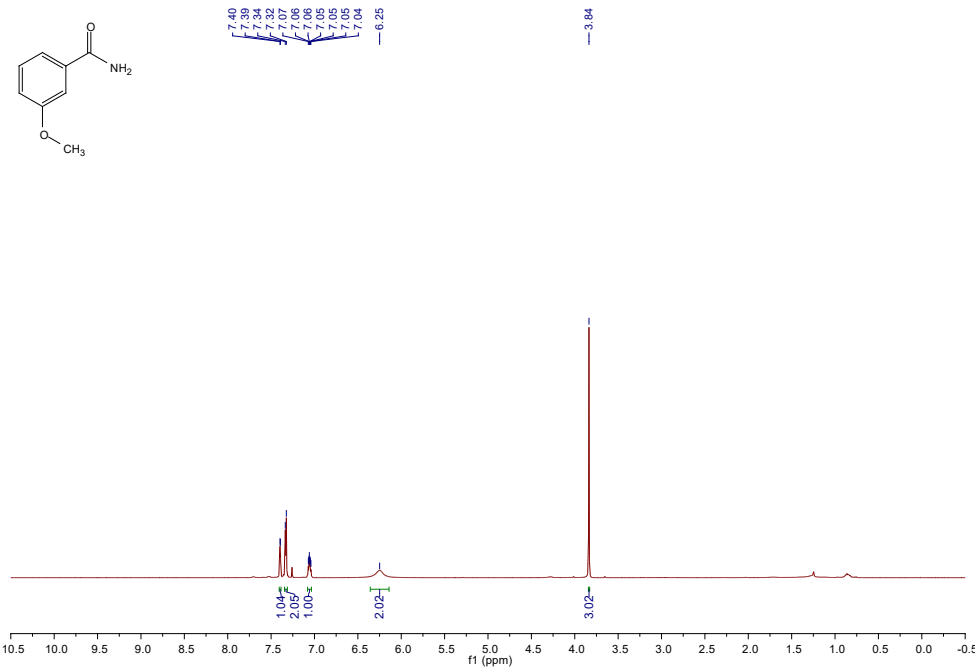
^{1}H-NMR (400 MHz, DMSO-d_6) of **2a**



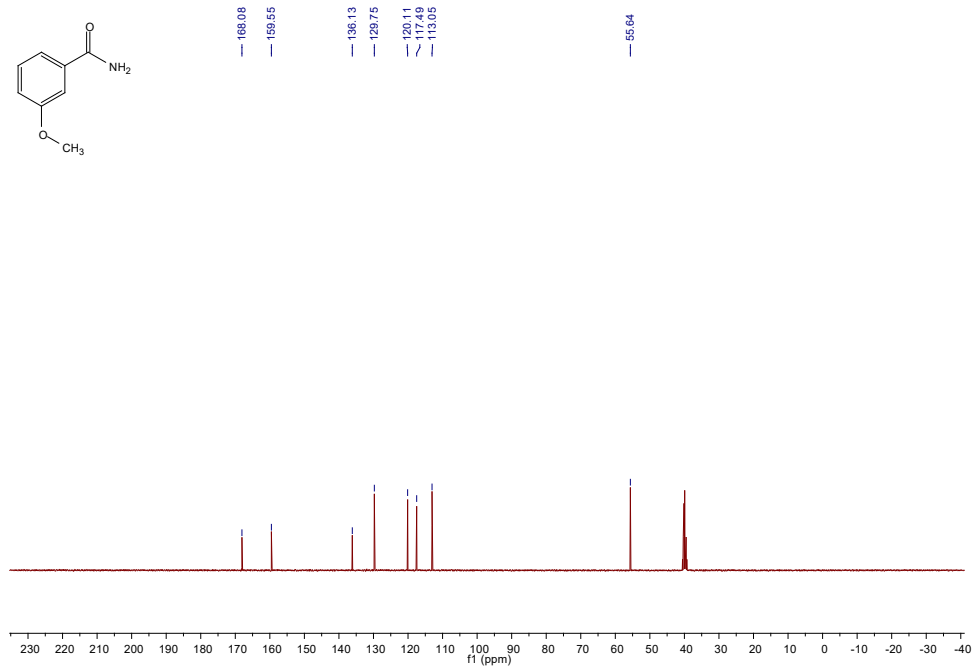
^{13}C-NMR (101 MHz, DMSO-d_6) of **2a**



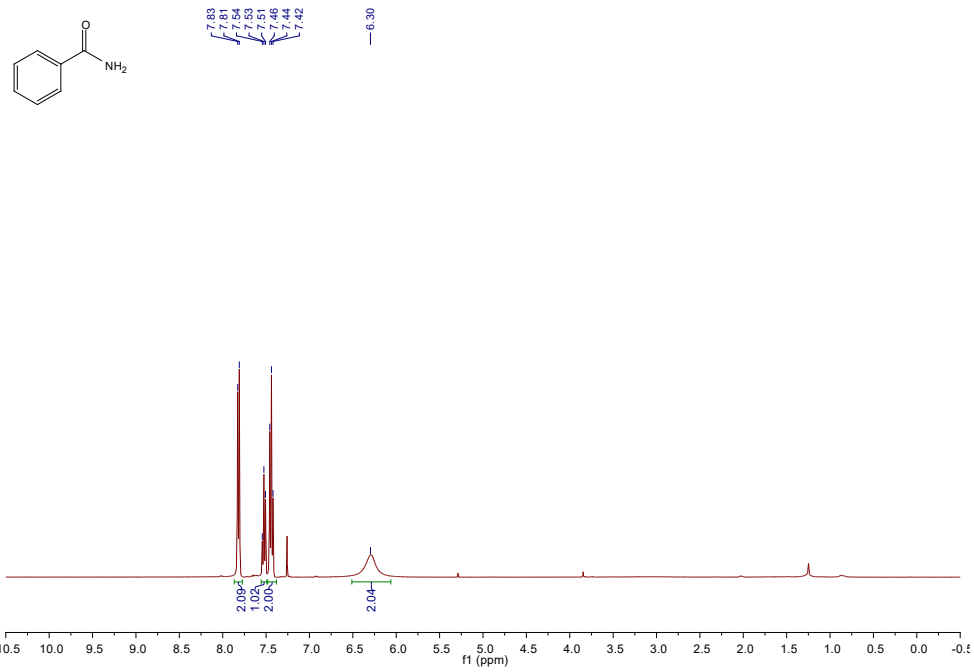
^{1}H-NMR (400 MHz, $CDCl_3$) of **2b**



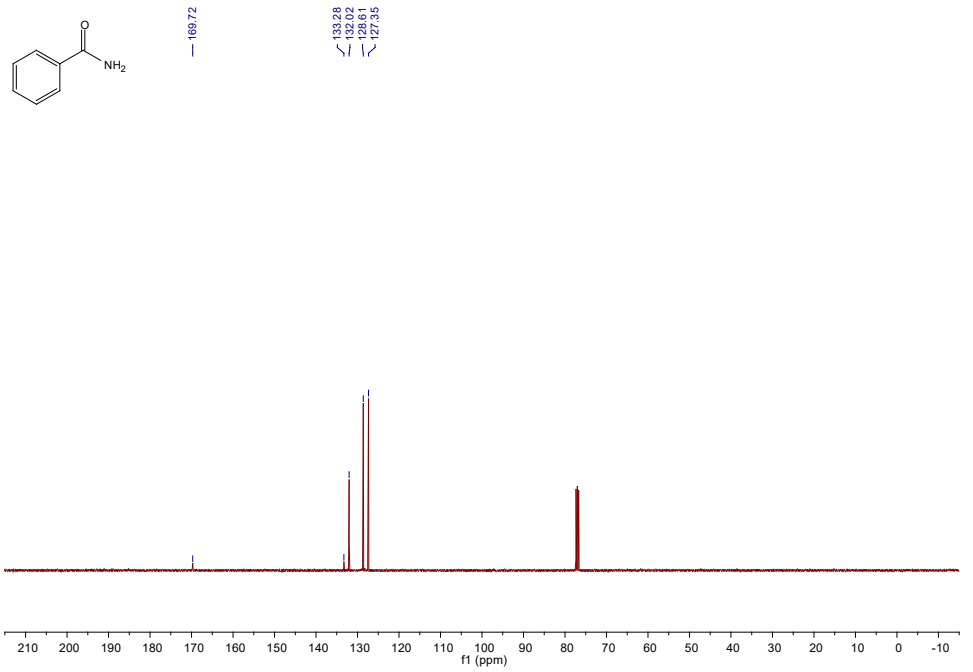
^{13}C-NMR (101 MHz, DMSO-d_6) of **2b**



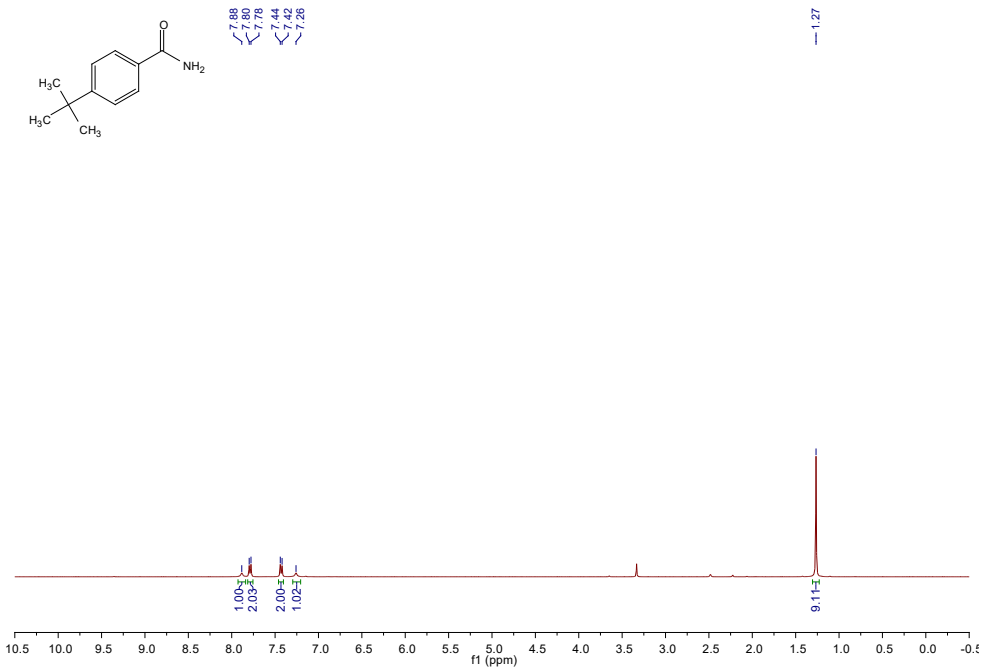
^{1}H-NMR (400 MHz, $CDCl_3$) of **2c**



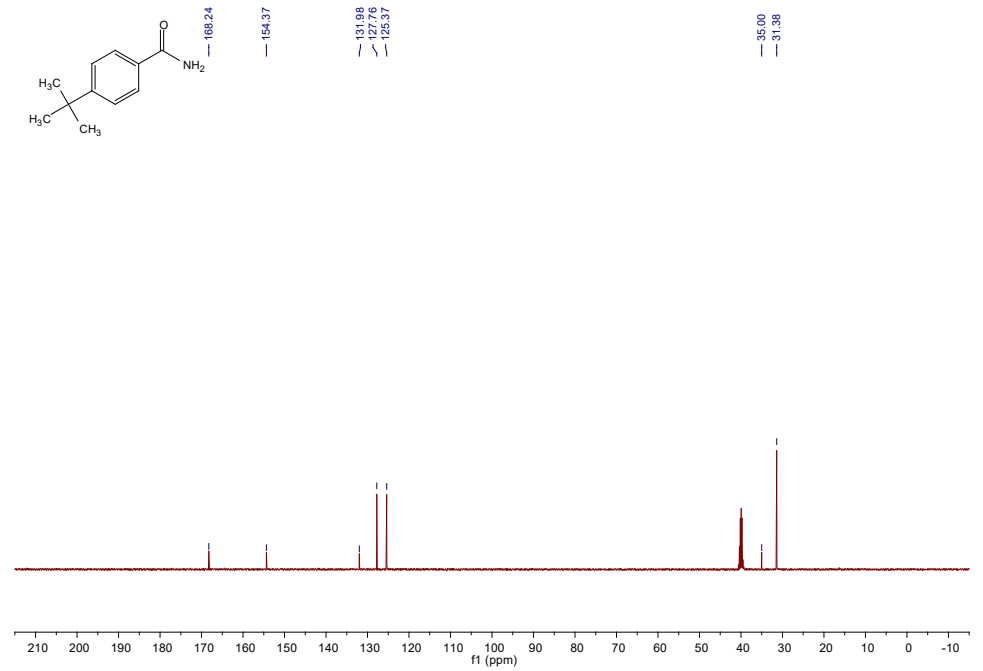
^{13}C-NMR (101 MHz, $CDCl_3$) of **2c**



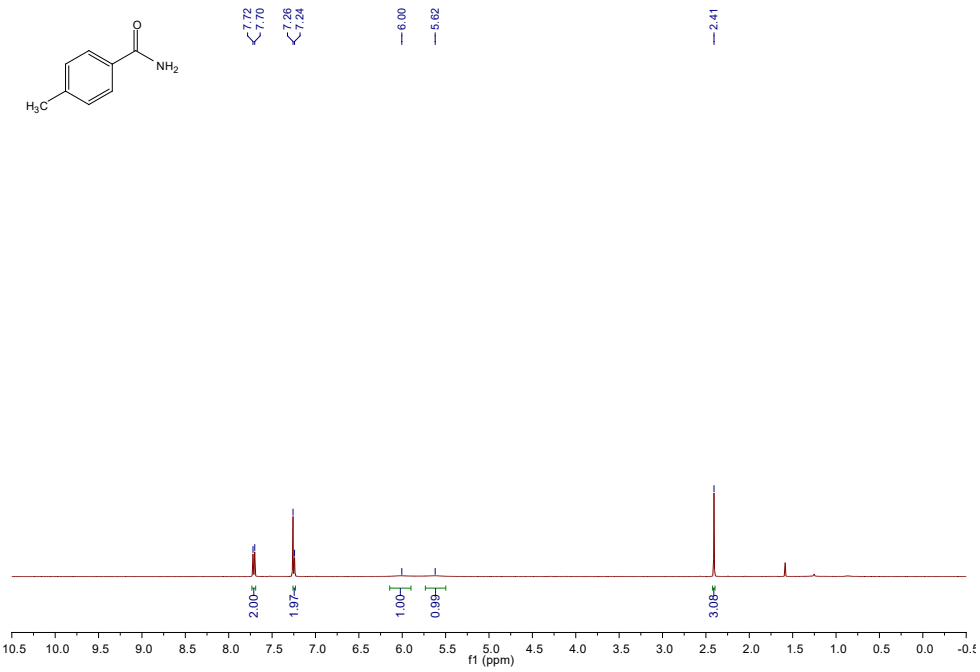
^{1}H-NMR (400 MHz, DMSO-d_6) of **2d**



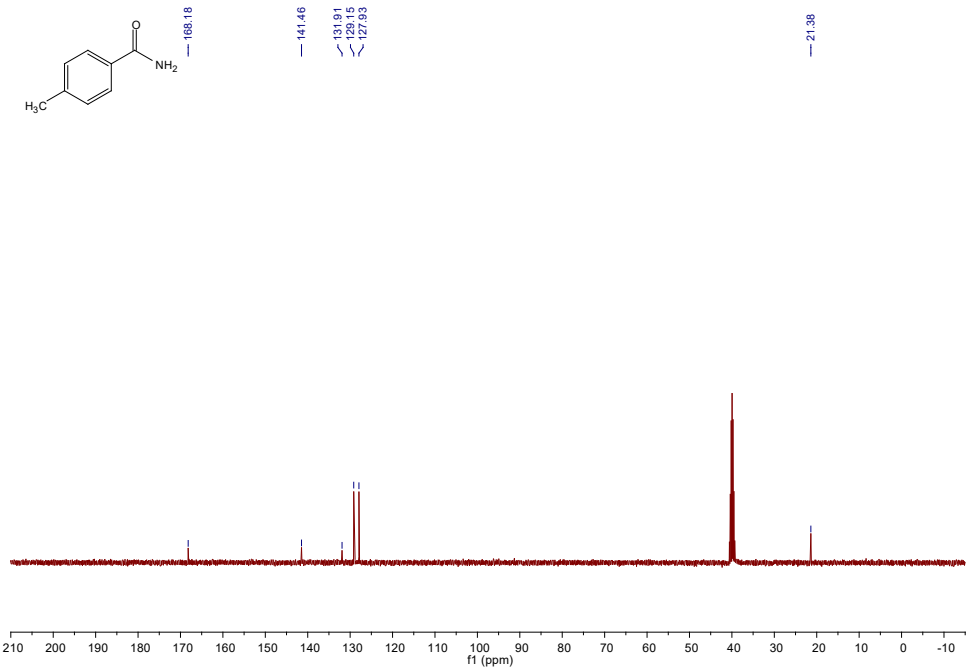
^{13}C-NMR (101 MHz, DMSO-d_6) of **2d**



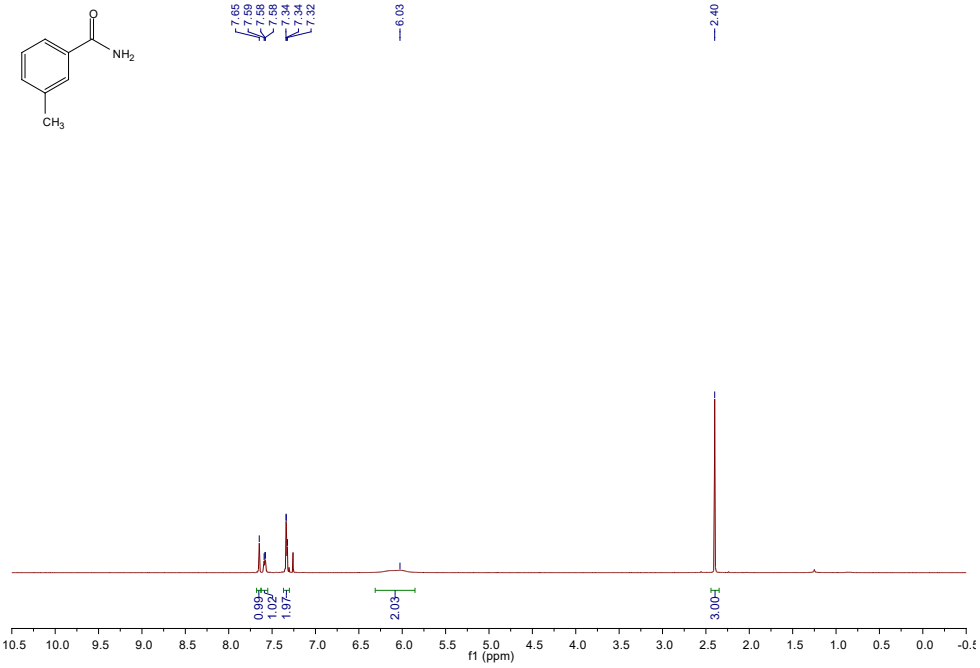
^{1}H-NMR (400 MHz, $CDCl_3$) of **2e**



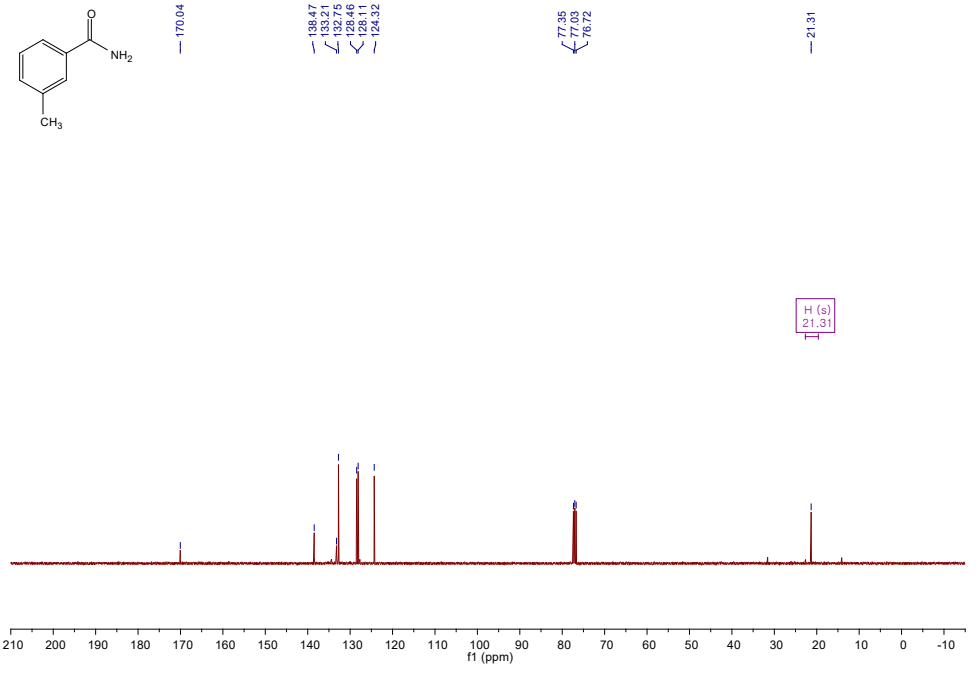
^{13}C-NMR (101 MHz, DMSO-d_6) of **2e**



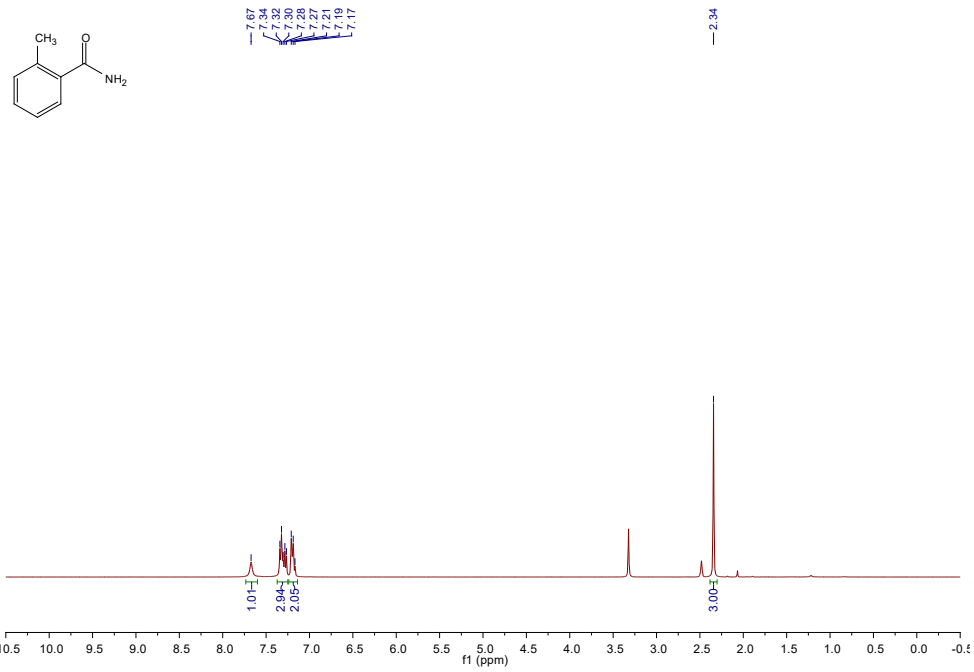
^{1}H-NMR (400 MHz, $CDCl_3$) of **2f**



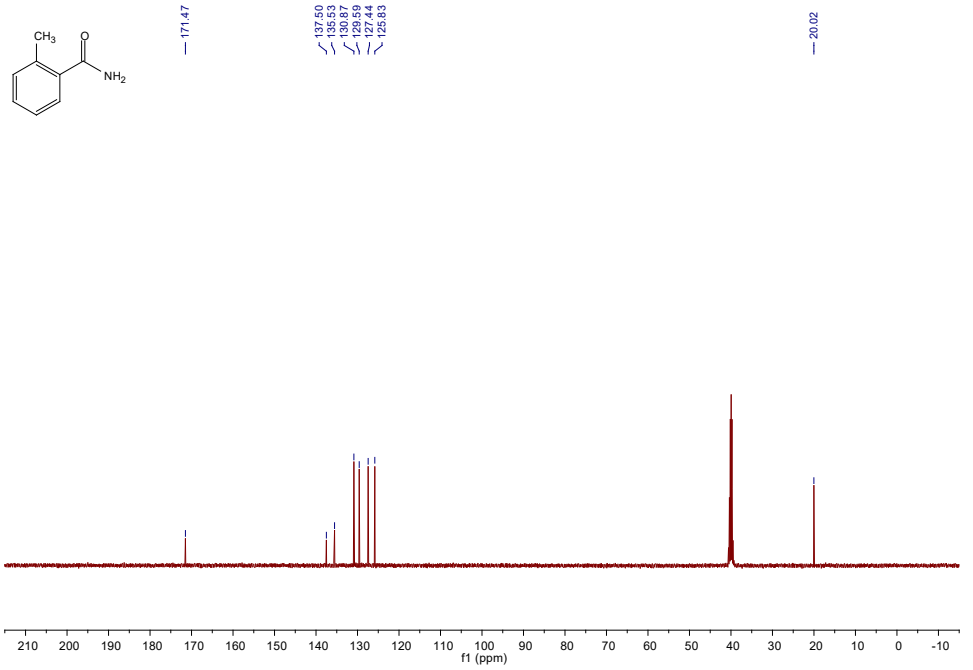
^{13}C-NMR (101 MHz, $CDCl_3$) of **2f**



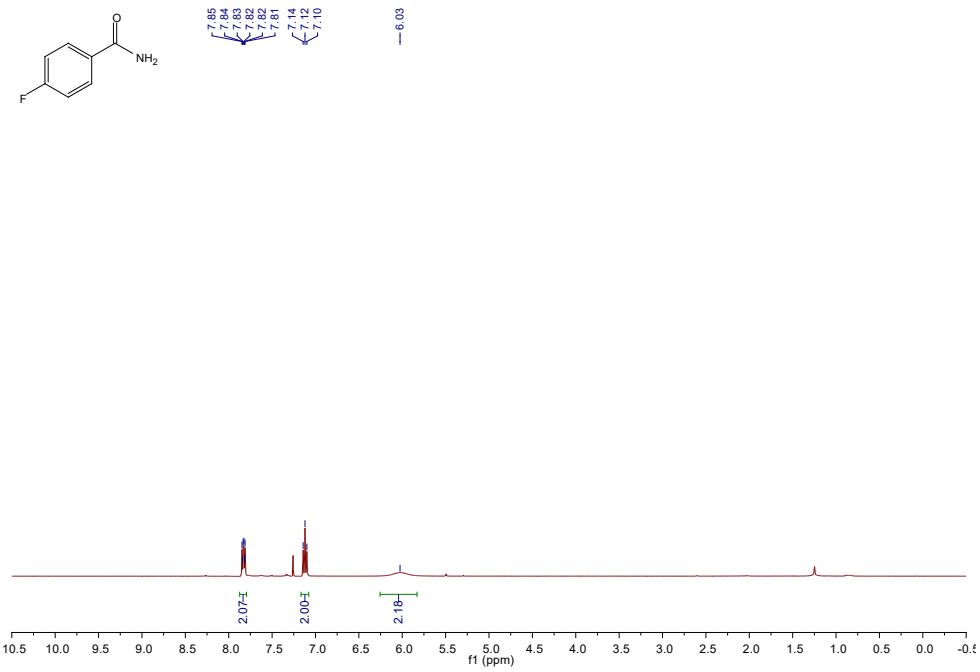
^{1}H-NMR (400 MHz, DMSO-d_6) of **2g**



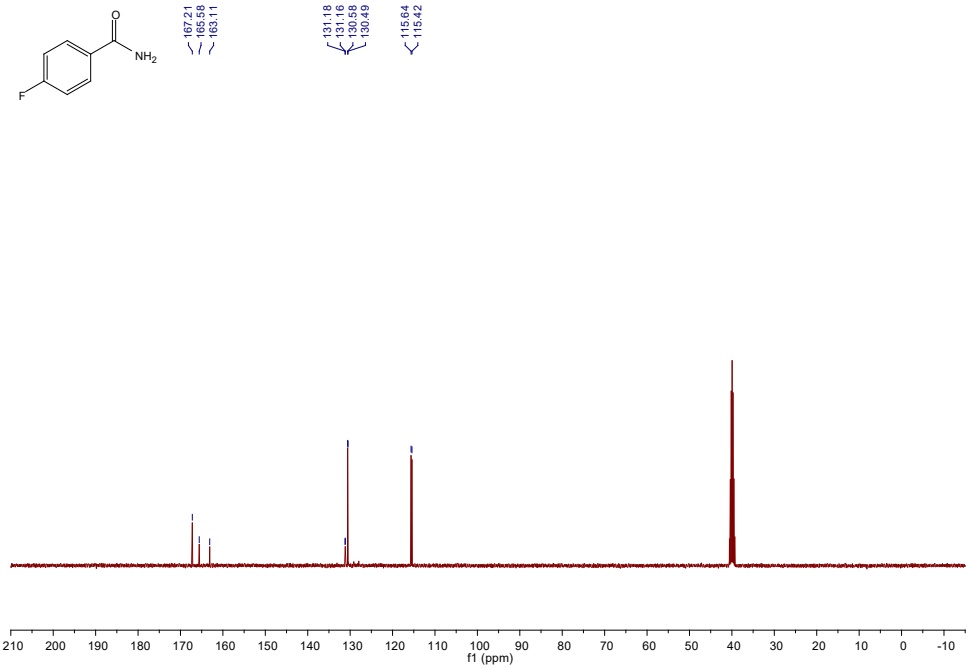
^{13}C-NMR (101 MHz, DMSO-d_6) of **2g**



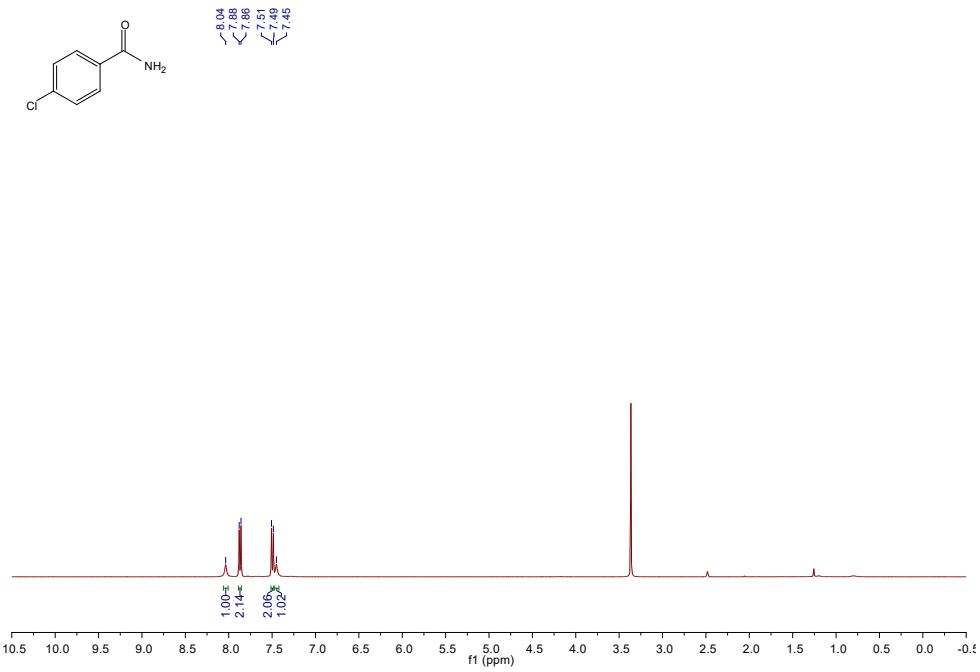
^{1}H-NMR (400 MHz, $CDCl_3$) of **2h**



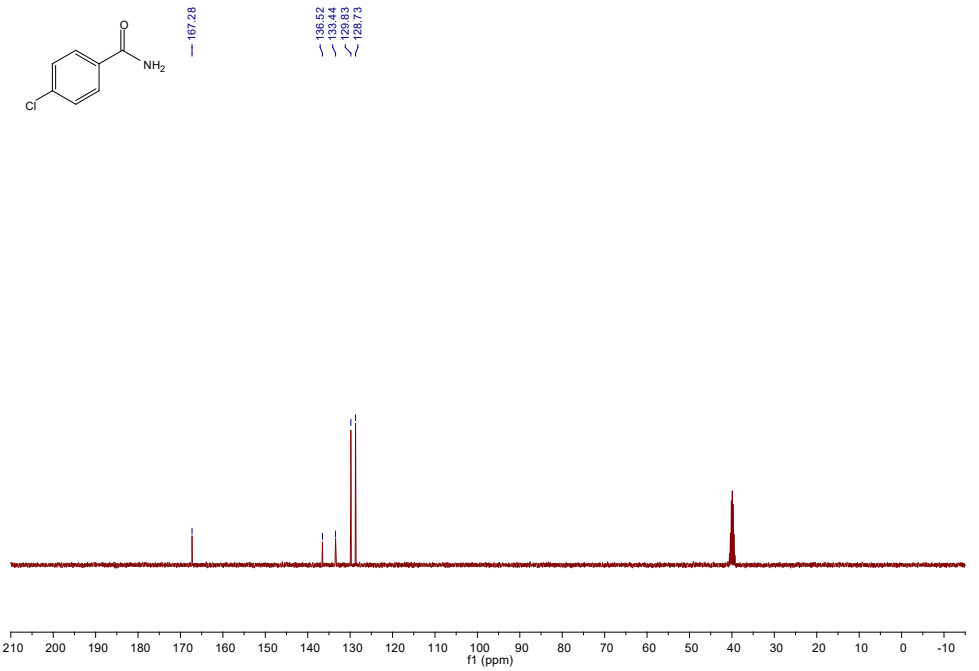
^{13}C-NMR (101 MHz, DMSO-d_6) of **2h**



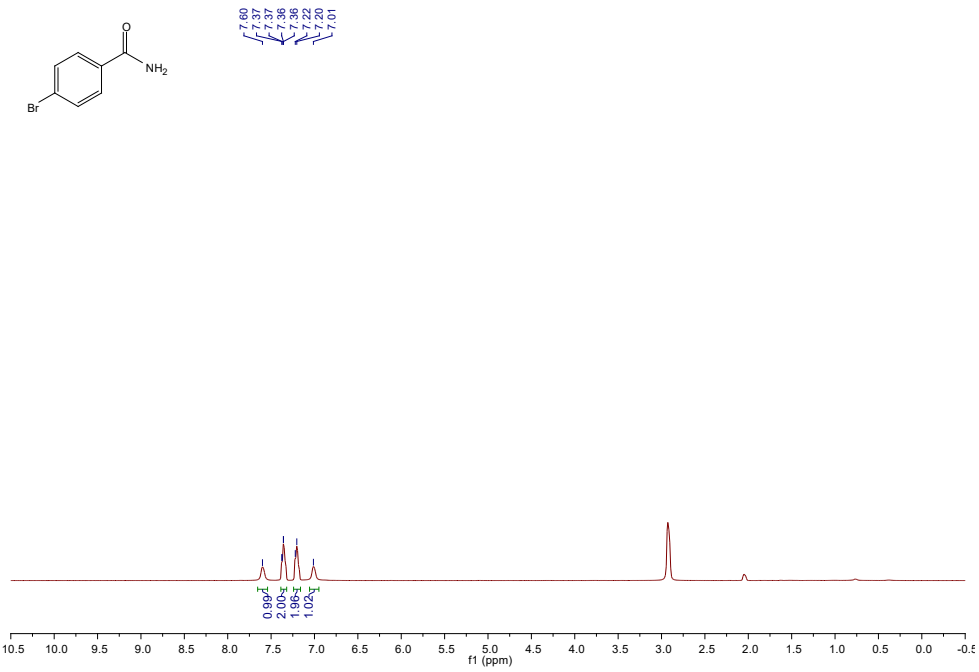
^{1}H-NMR (400 MHz, DMSO-d_6) of **2i**



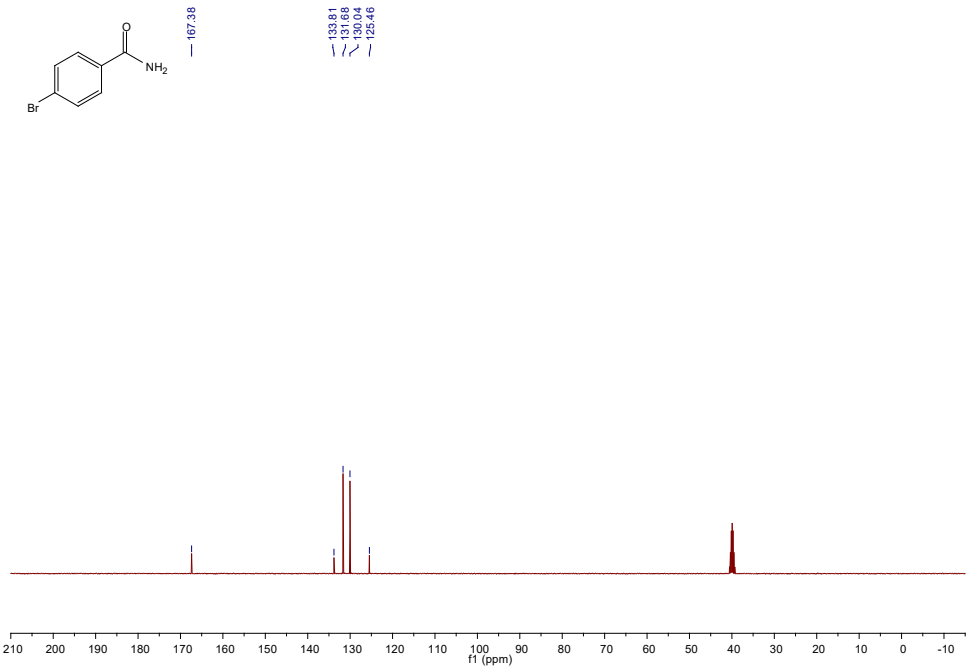
^{13}C-NMR (101 MHz, DMSO-d_6) of **2i**



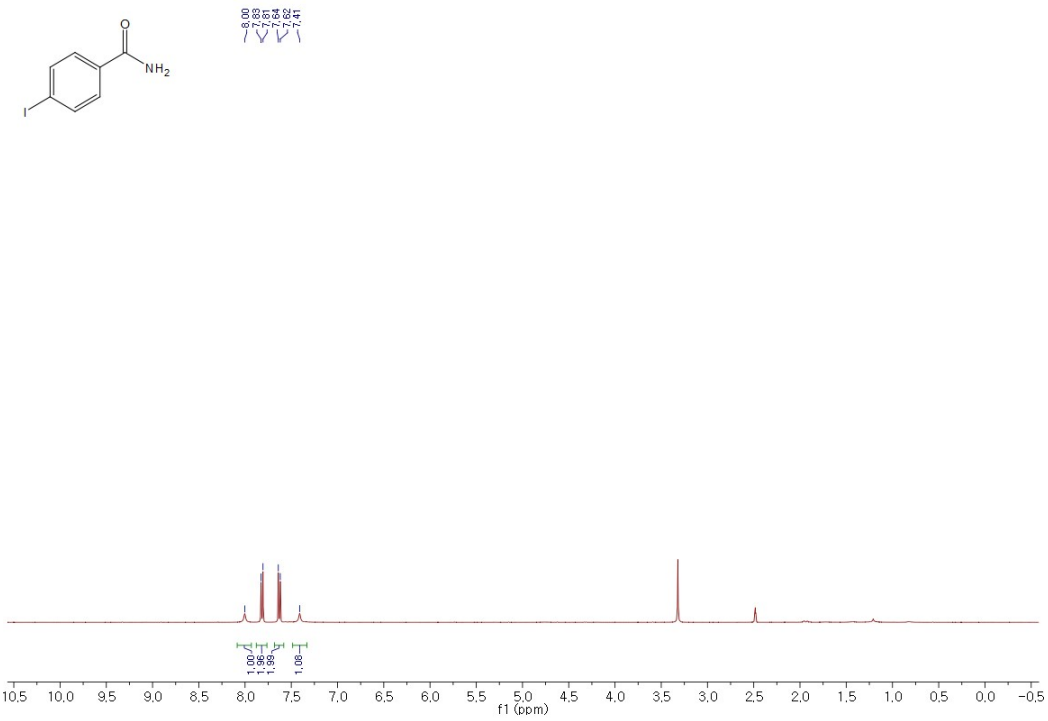
^{1}H-NMR (400 MHz, DMSO-d_6) of **2j**



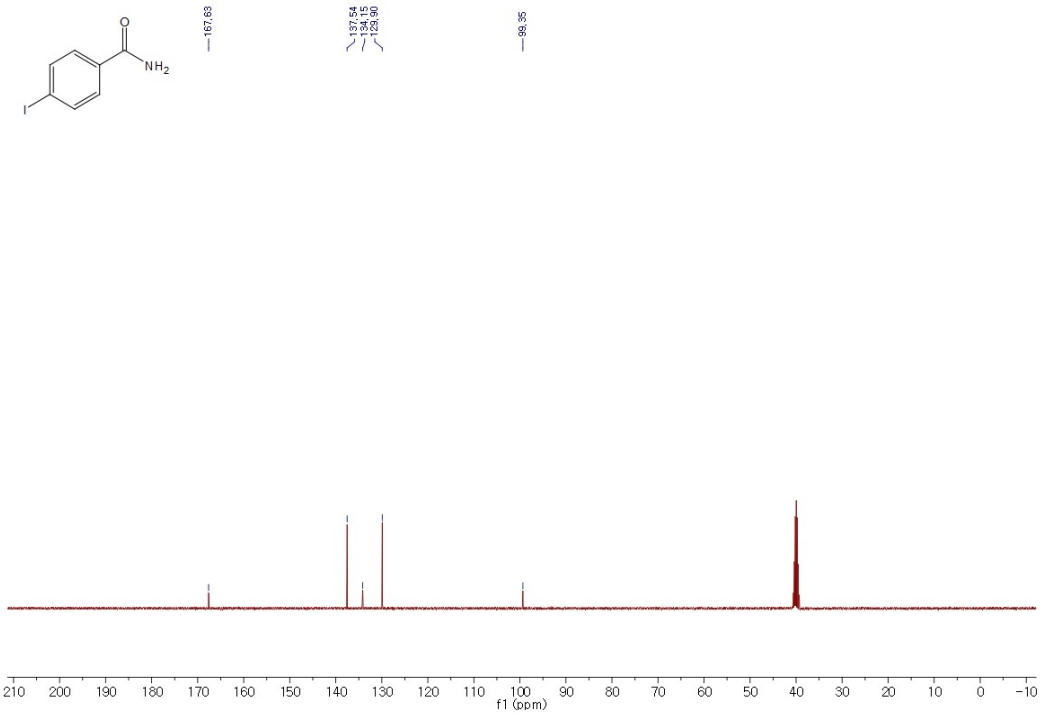
^{13}C-NMR (101 MHz, DMSO-d_6) of **2j**



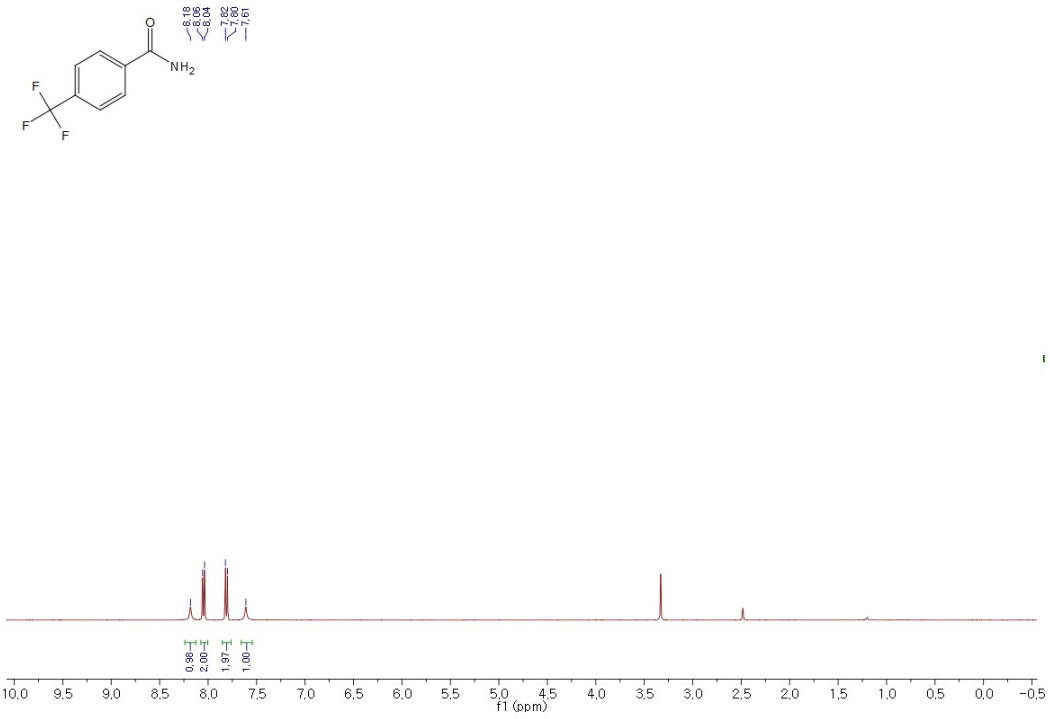
^{1}H-NMR (400 MHz, DMSO-d_6) of **2k**



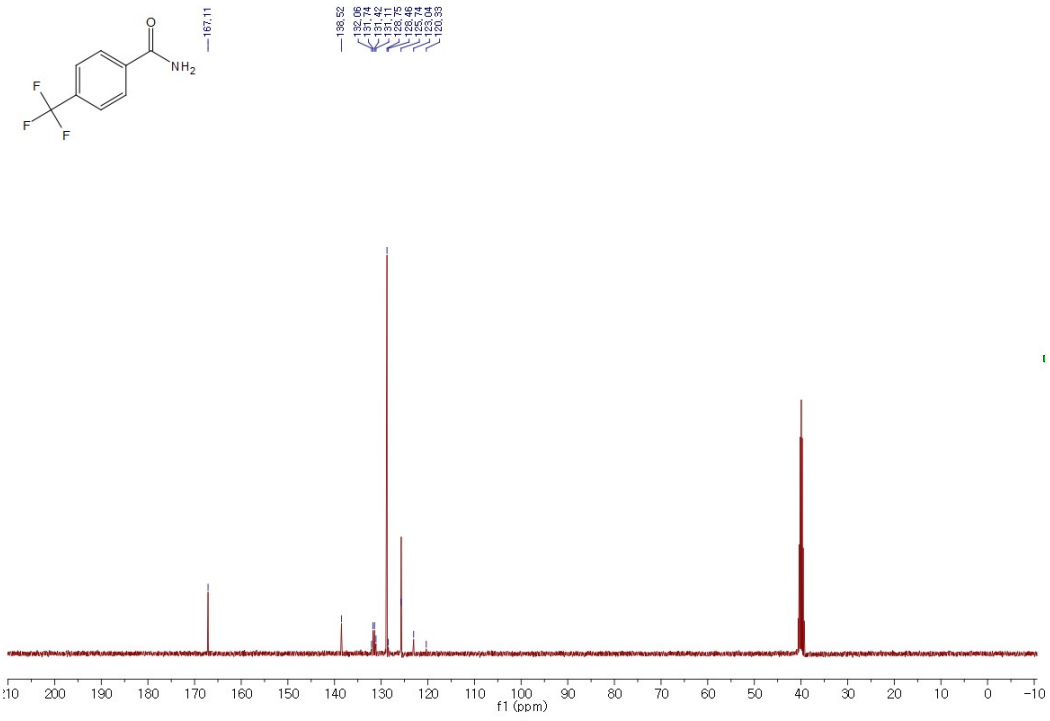
^{13}C-NMR (101 MHz, DMSO-d_6) of **2k**



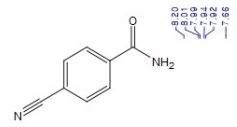
^{1}H-NMR (400 MHz, DMSO-d_6) of **2l**

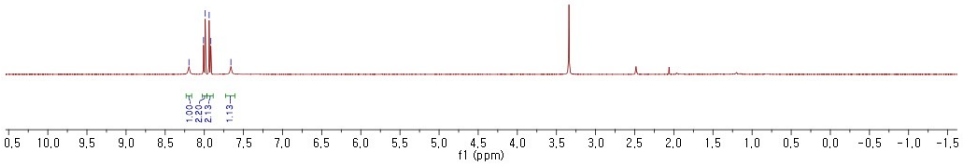


^{13}C-NMR (101 MHz, DMSO-d_6) of **2l**

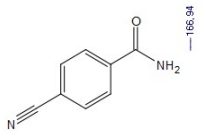


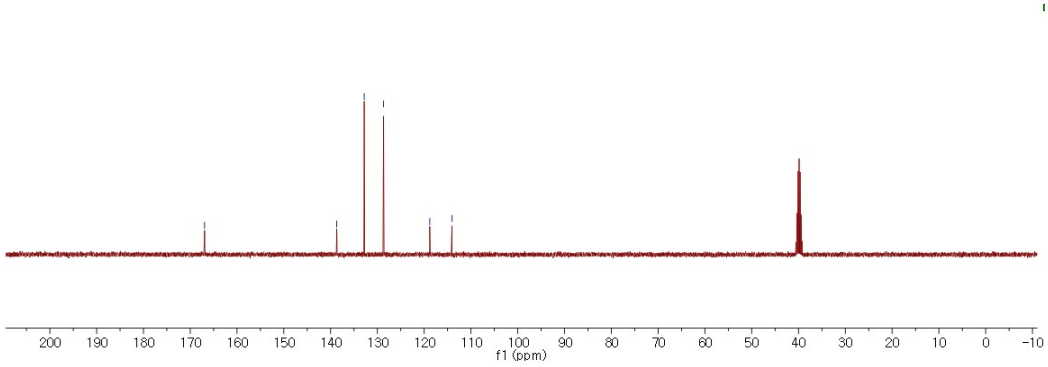
^{1}H-NMR (400 MHz, DMSO-d_6) of **2m**



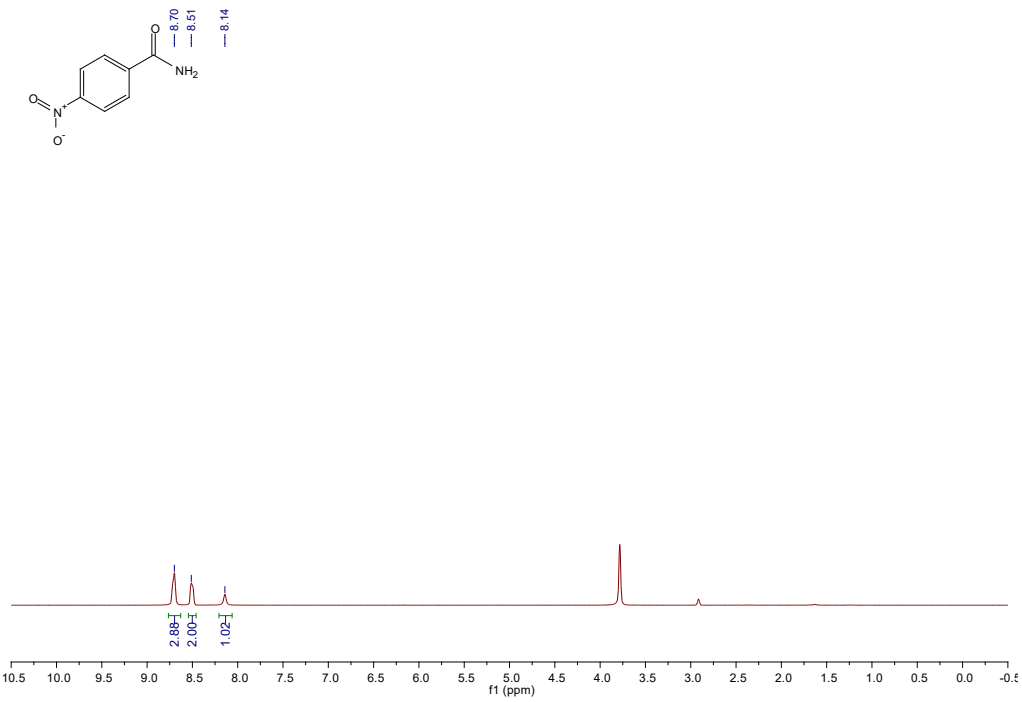


^{13}C-NMR (101 MHz, DMSO-d_6) of **2m**

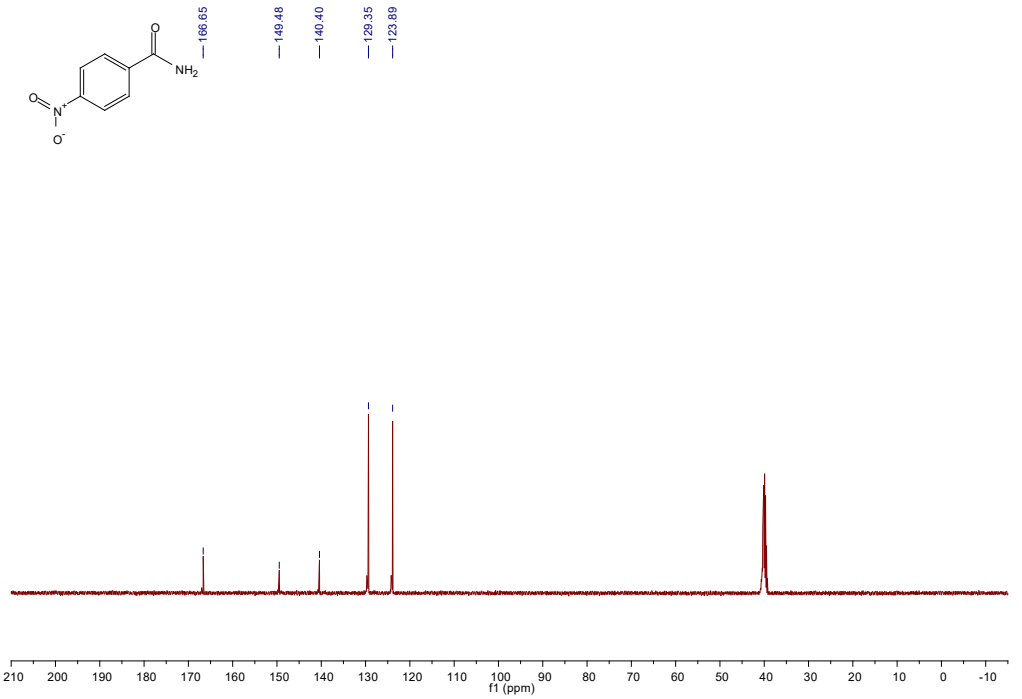




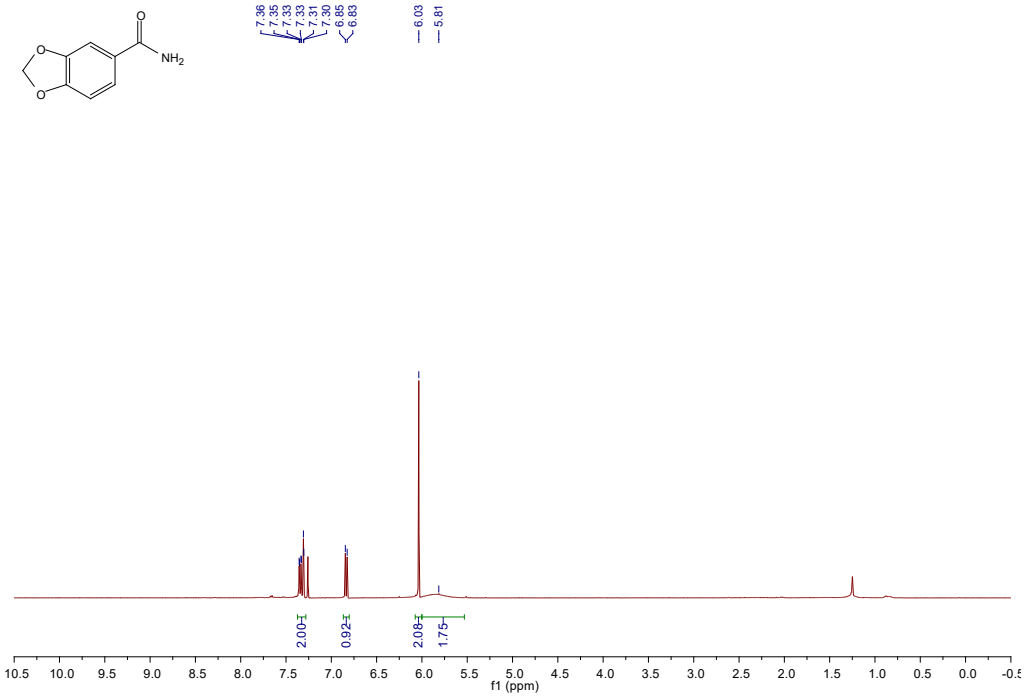
^{1}H-NMR (400 MHz, DMSO-d_6) of **2n**



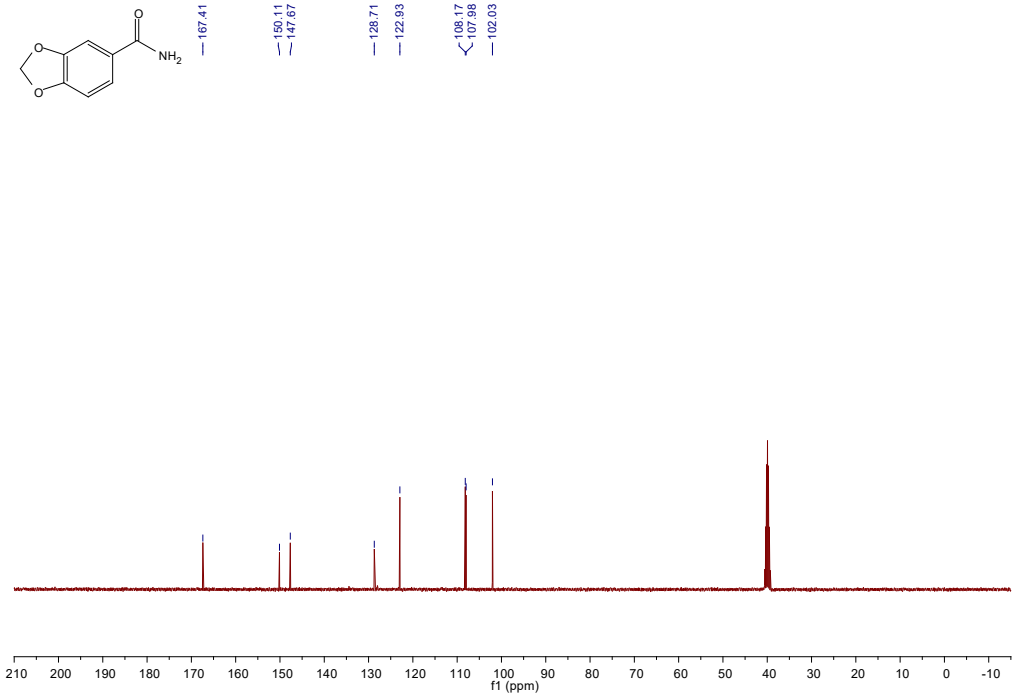
^{13}C-NMR (101 MHz, DMSO-d_6) of **2n**



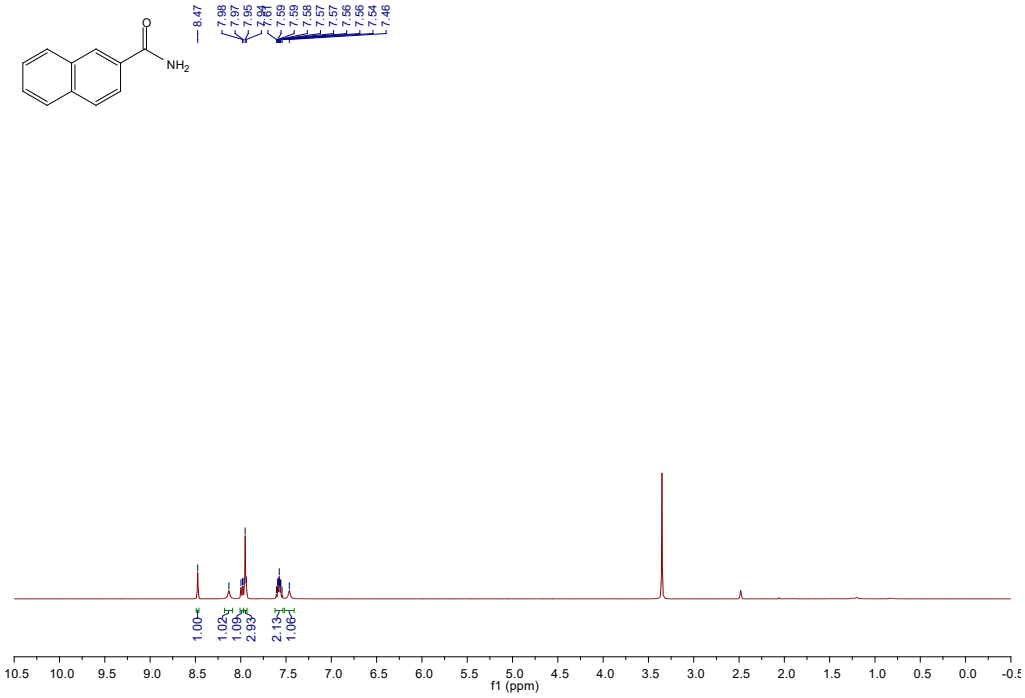
^{1}H-NMR (400 MHz, $CDCl_3$) of **2o**



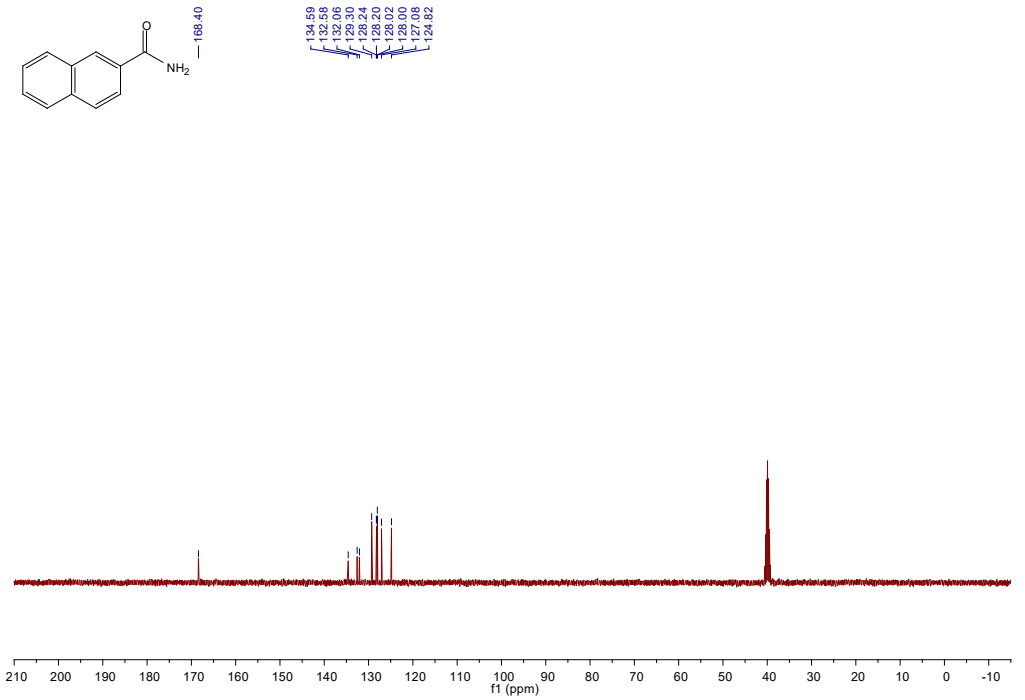
^{13}C-NMR (101 MHz, DMSO-d_6) of **2o**



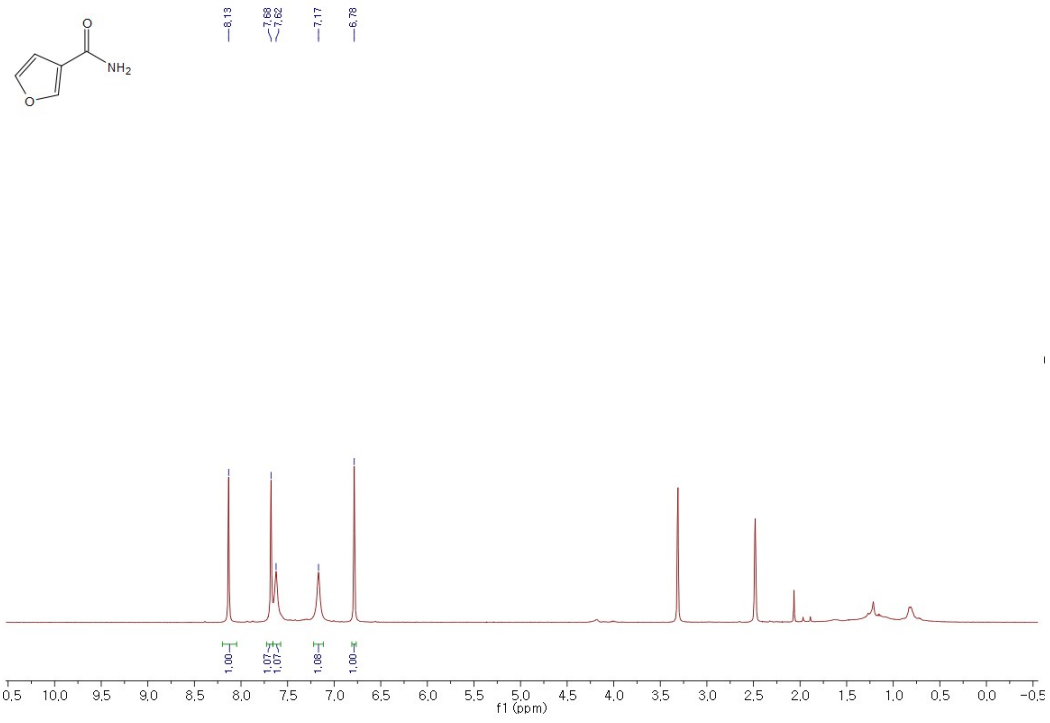
^{1}H-NMR (400 MHz, DMSO-d_6) of **2p**



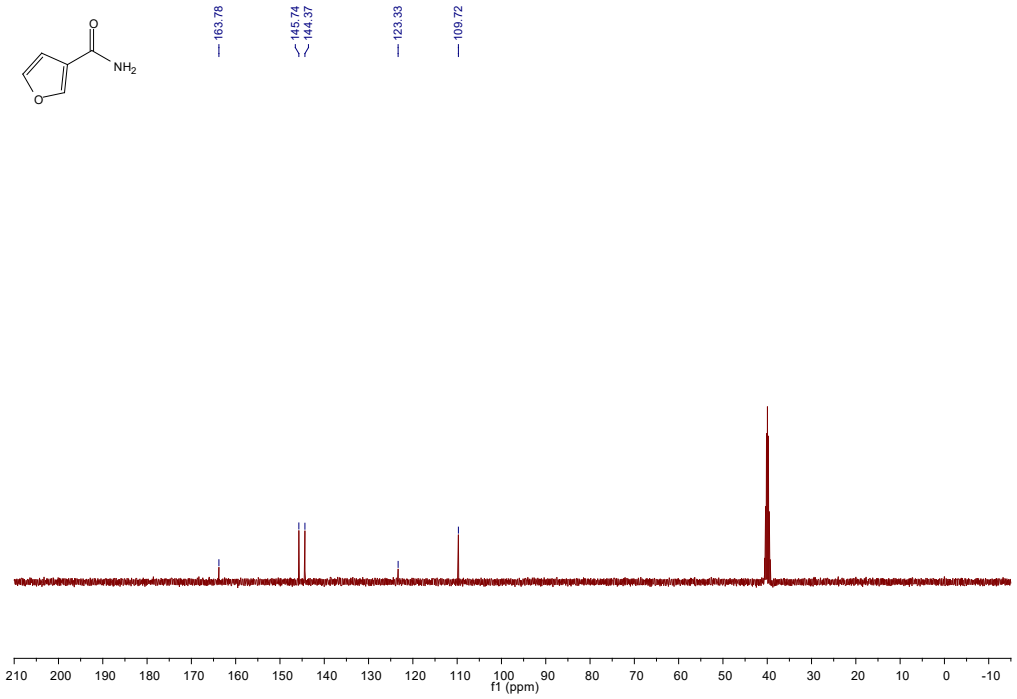
^{13}C-NMR (101 MHz, DMSO-d_6) of **2p**



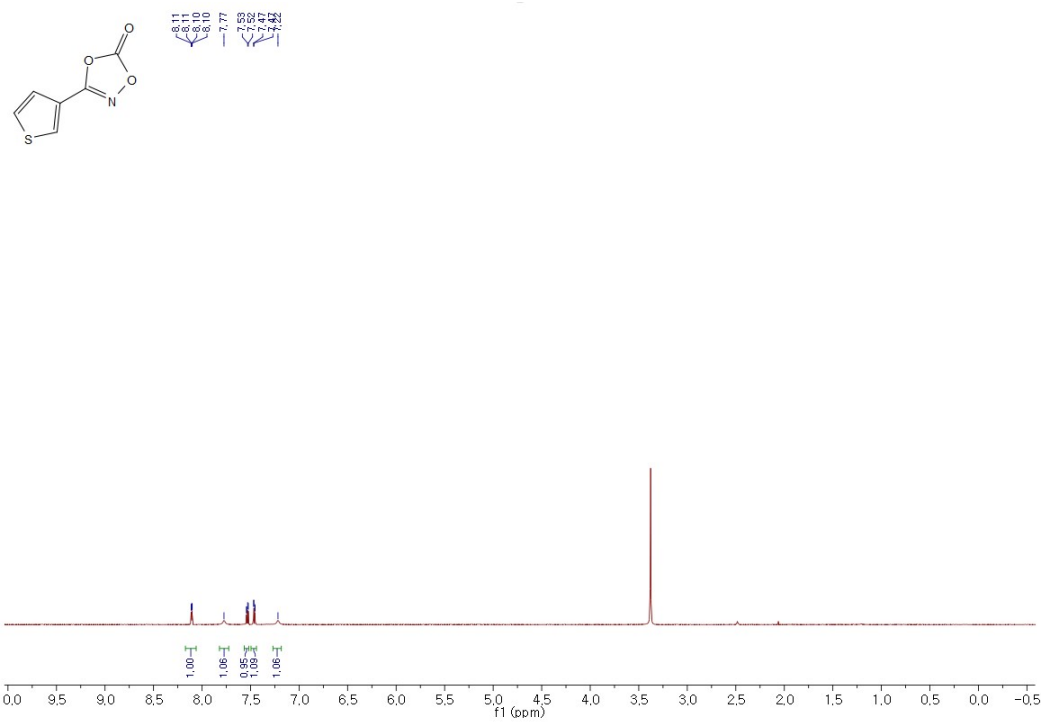
^{1}H-NMR (400 MHz, DMSO-d_6) of of **2q**



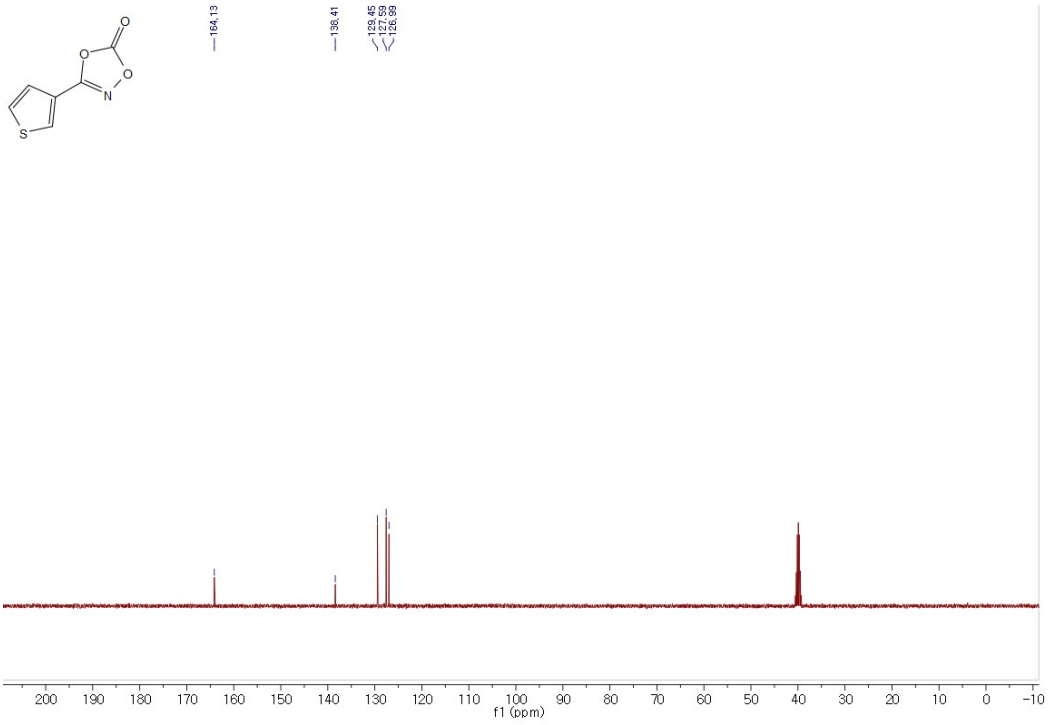
^{13}C-NMR (101 MHz, DMSO-d_6) of **2q**



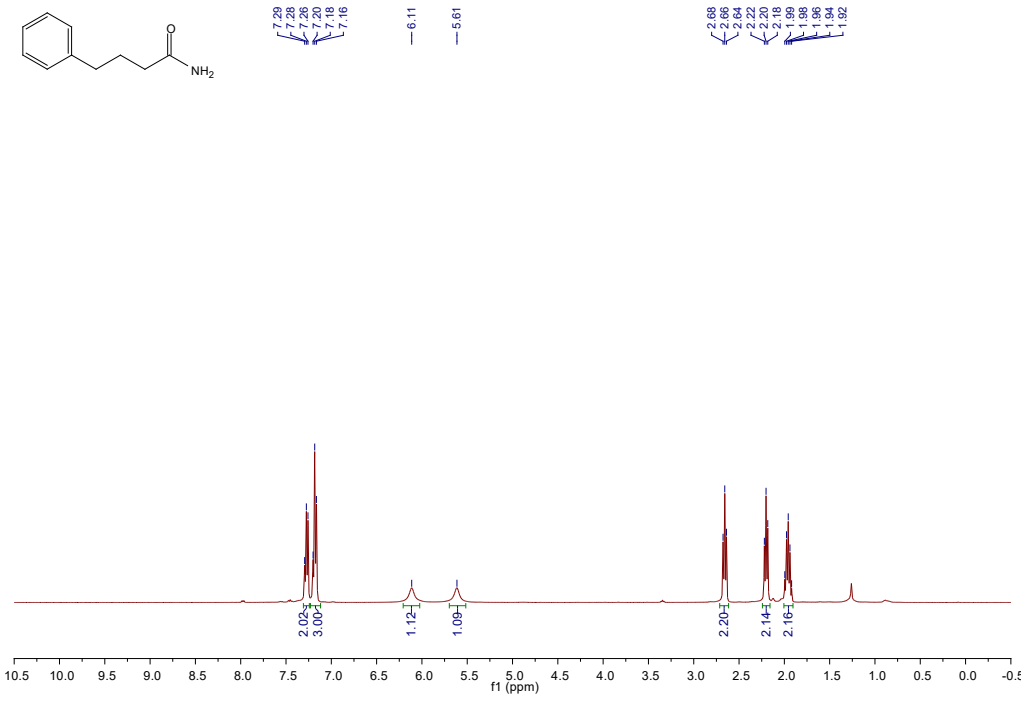
^{1}H-NMR (400 MHz, DMSO-d_6) of **2r**



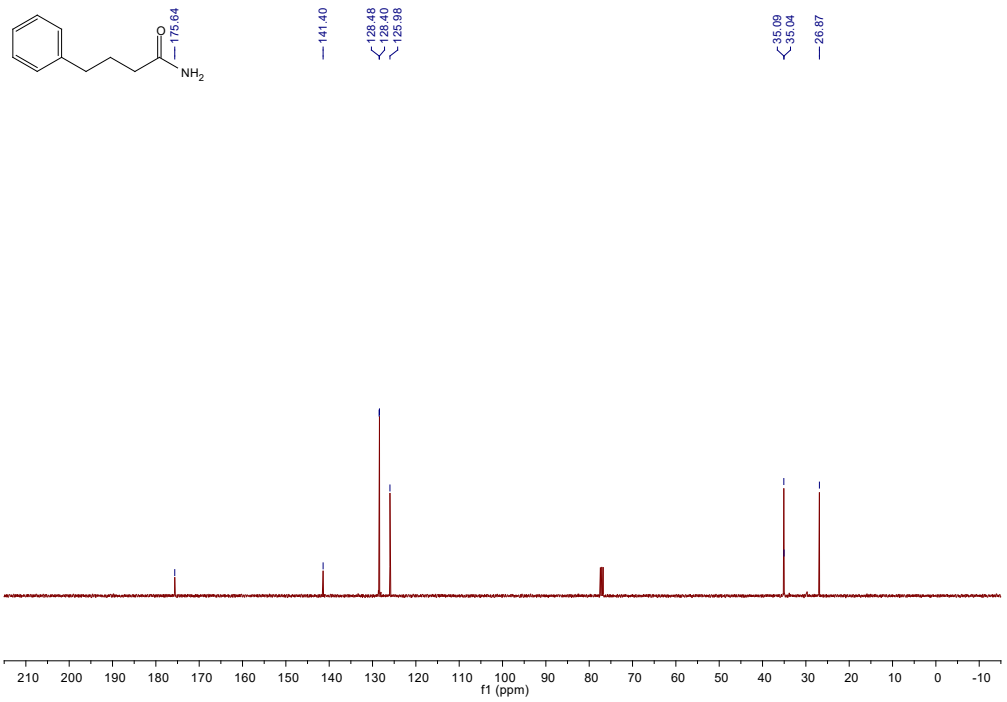
^{13}C-NMR (101 MHz, DMSO-d_6) of **2r**



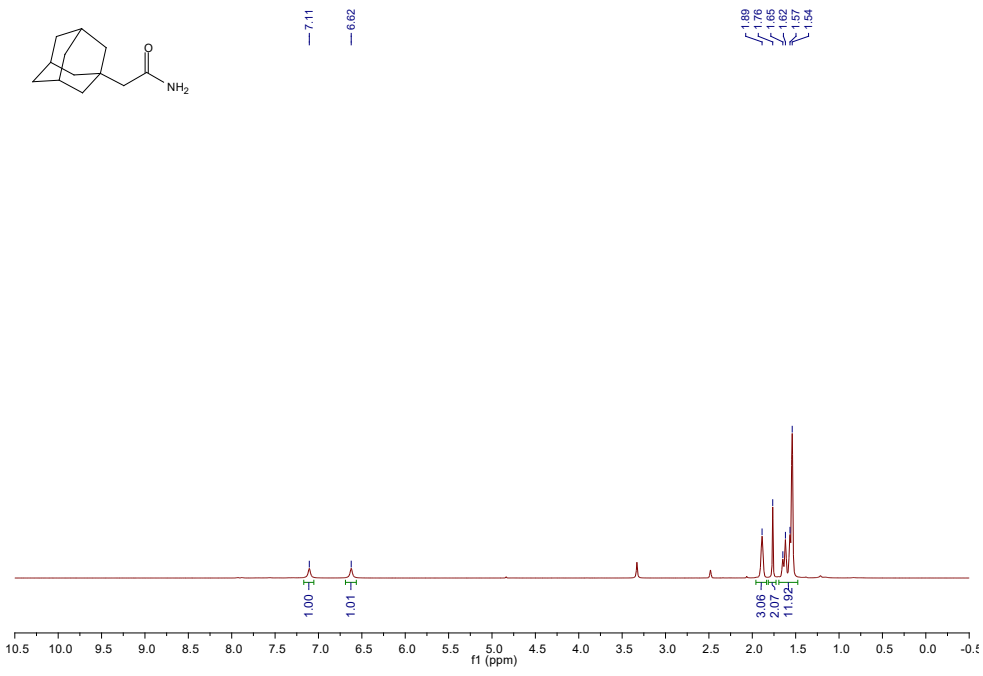
^{1}H-NMR (400 MHz, $CDCl_3$) of **2s**



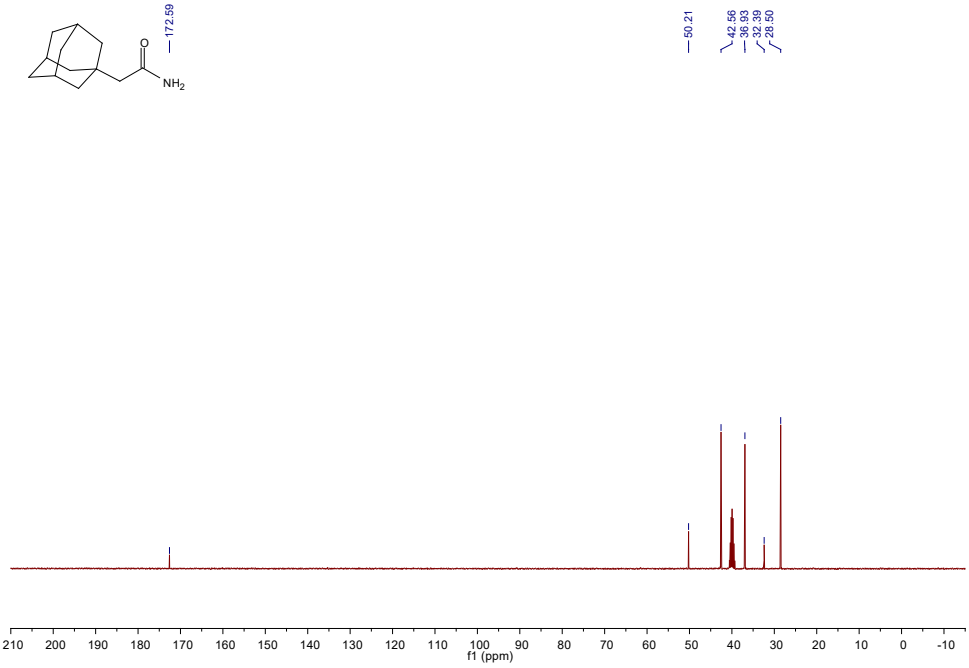
^{13}C-NMR (101 MHz, $CDCl_3$) of **2s**



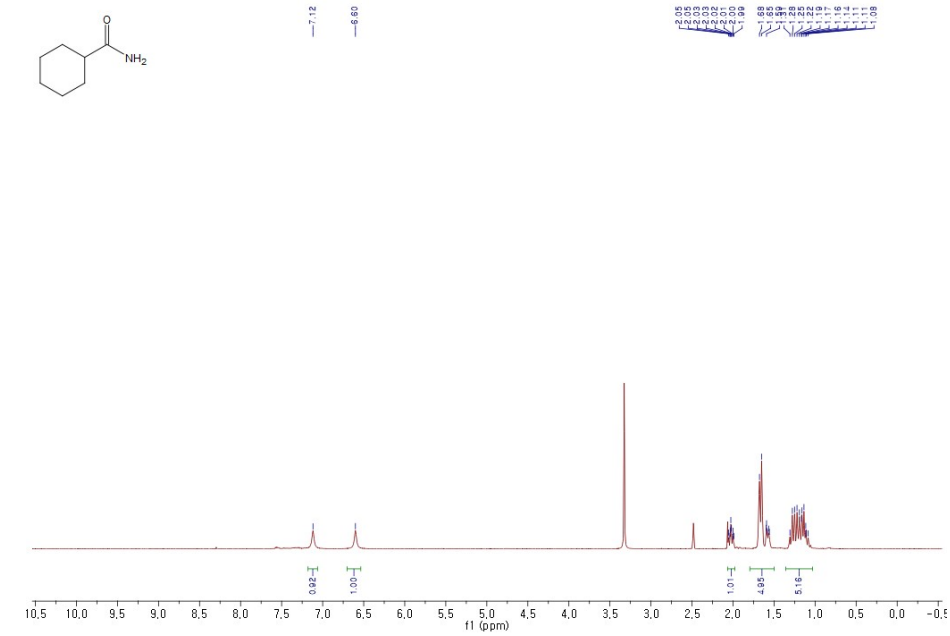
^{1}H-NMR (400 MHz, DMSO-d_6) of **2t**



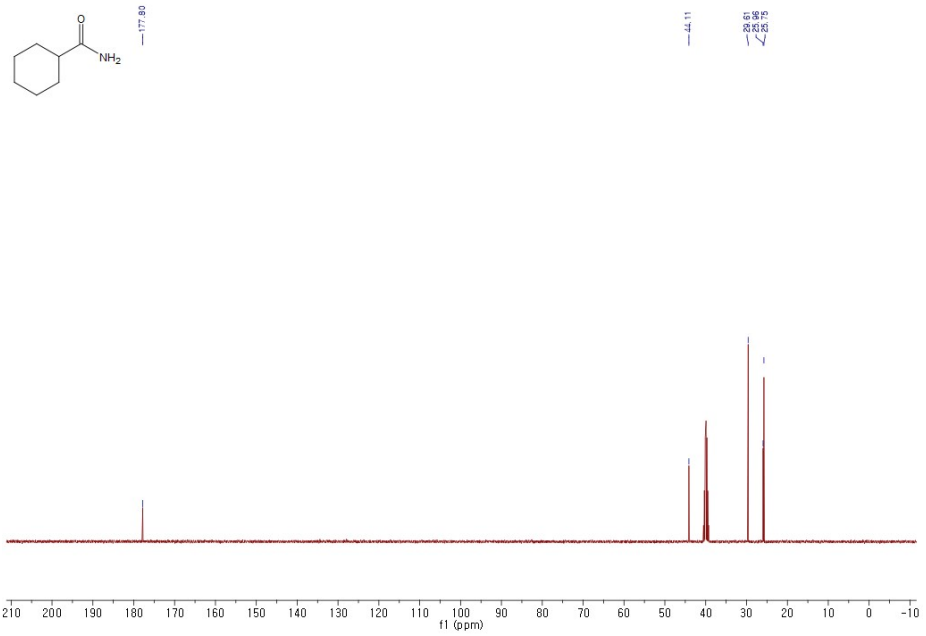
^{13}C-NMR (101 MHz, DMSO-d_6) of **2t**



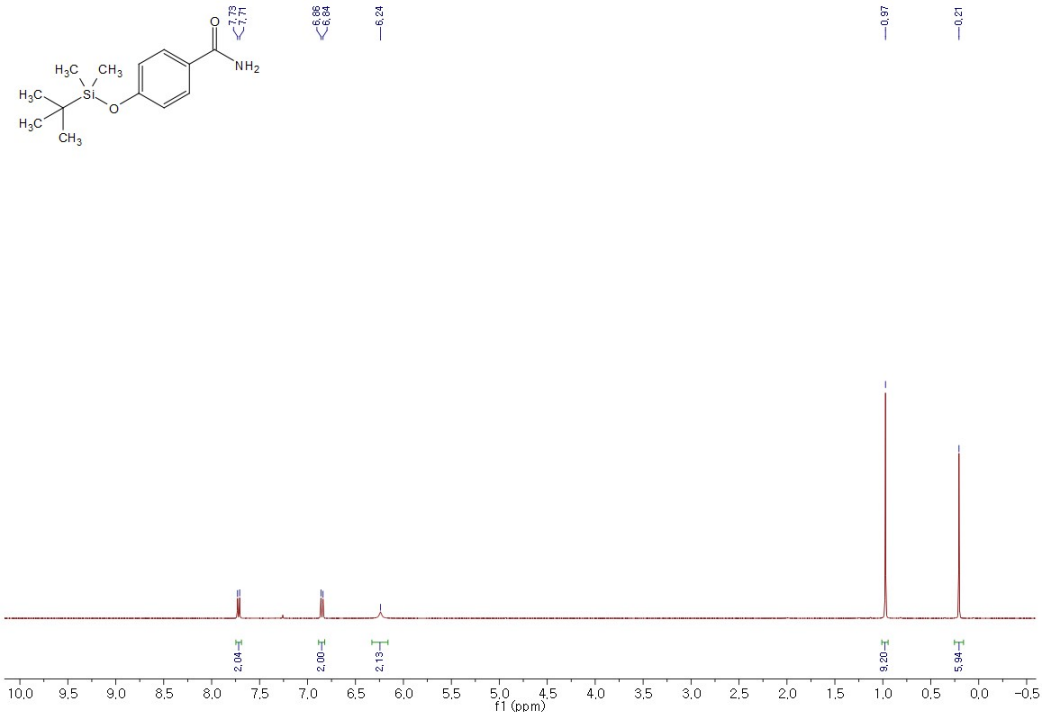
^{1}H-NMR (400 MHz, DMSO-d_6) of **2u**



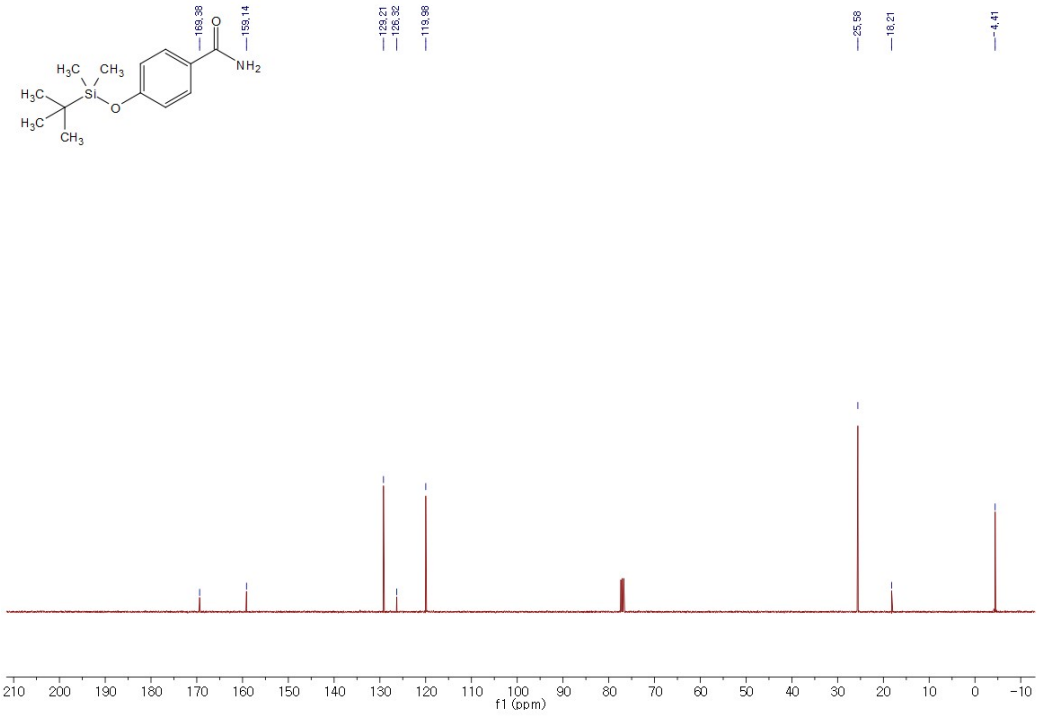
^{13}C-NMR (101 MHz, DMSO-d_6) of **2u**



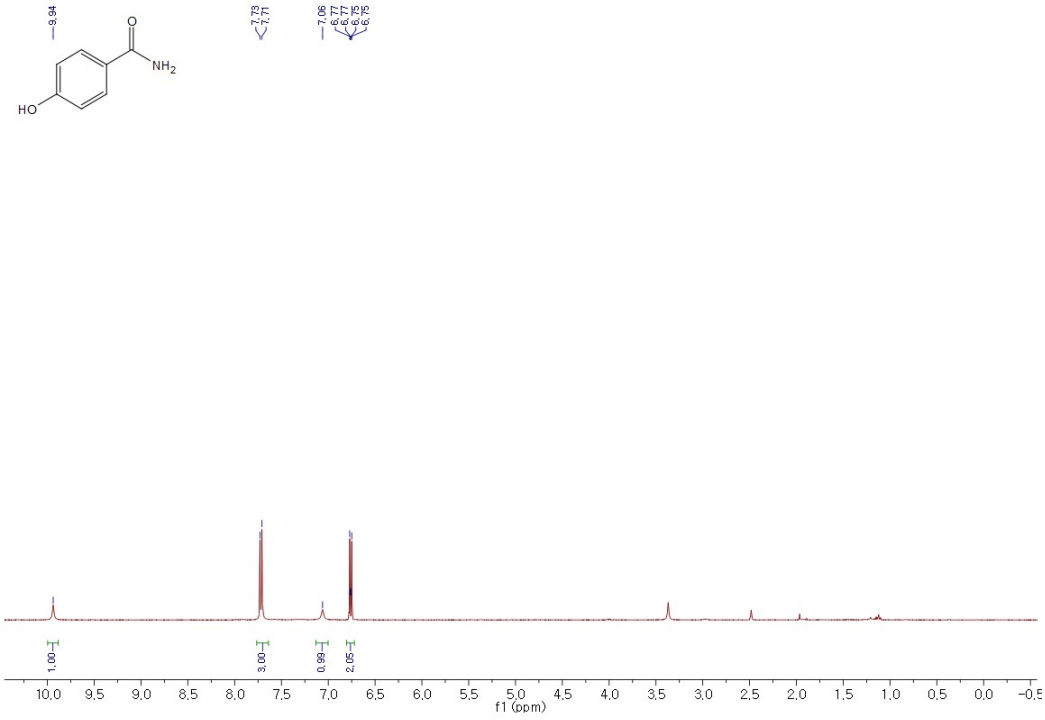
^{1}H-NMR (400 MHz, $CDCl_3$) of **2v**



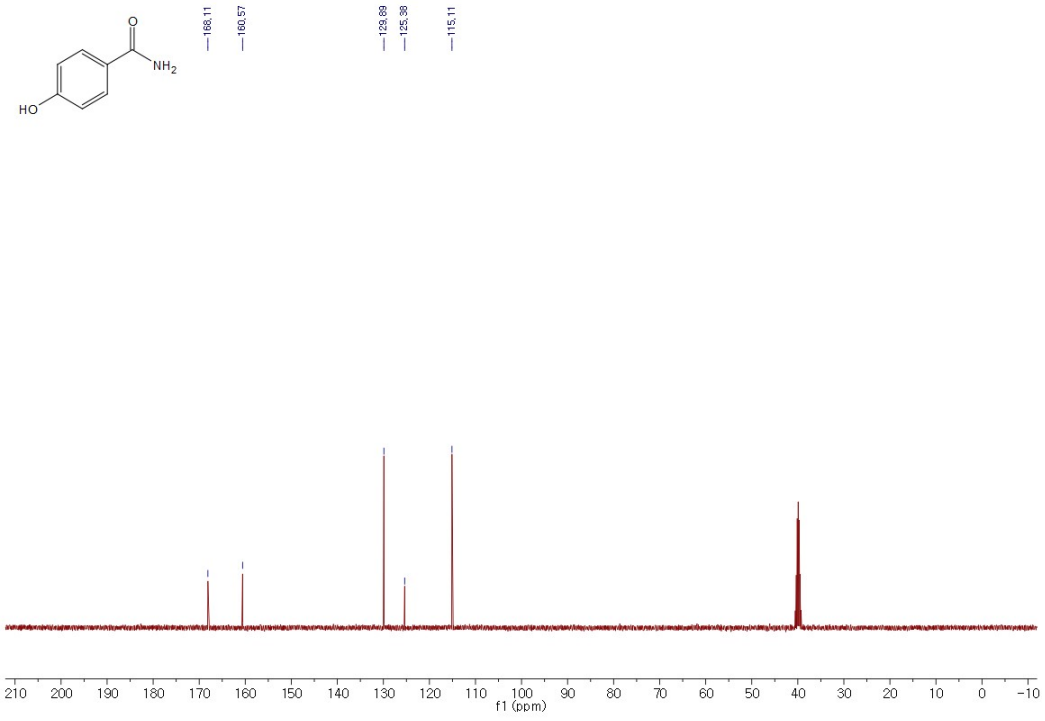
^{13}C-NMR (101 MHz, $CDCl_3$) of **2v**



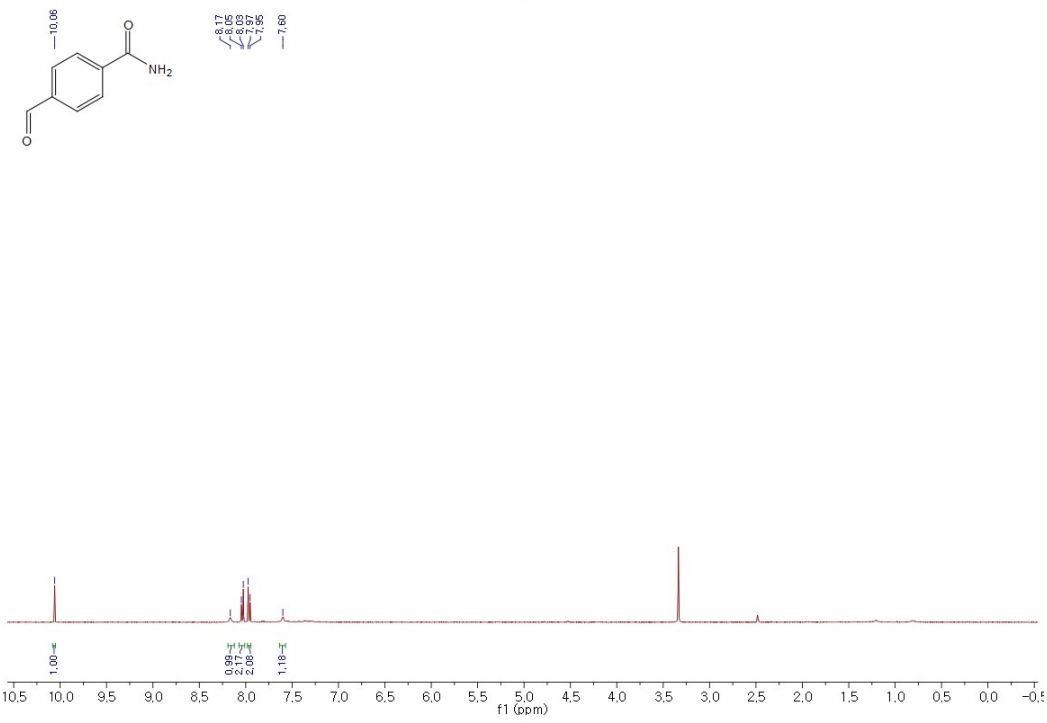
^{1}H-NMR (400 MHz, DMSO-d_6) of **2w**



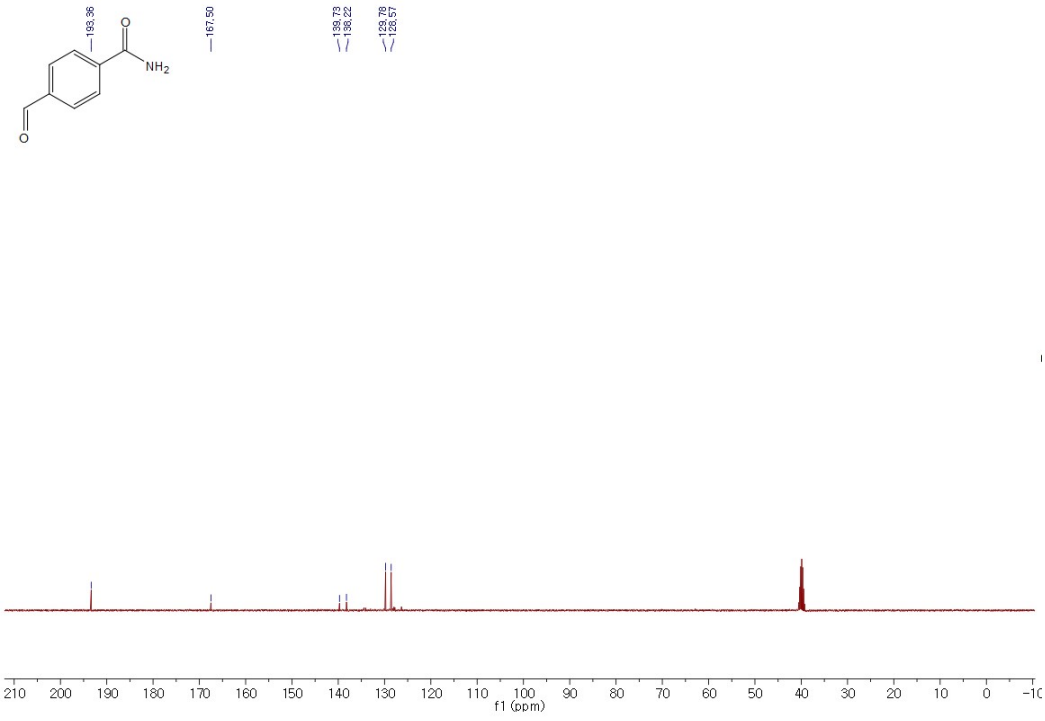
^{13}C-NMR (101 MHz, DMSO-d_6) of **2w**



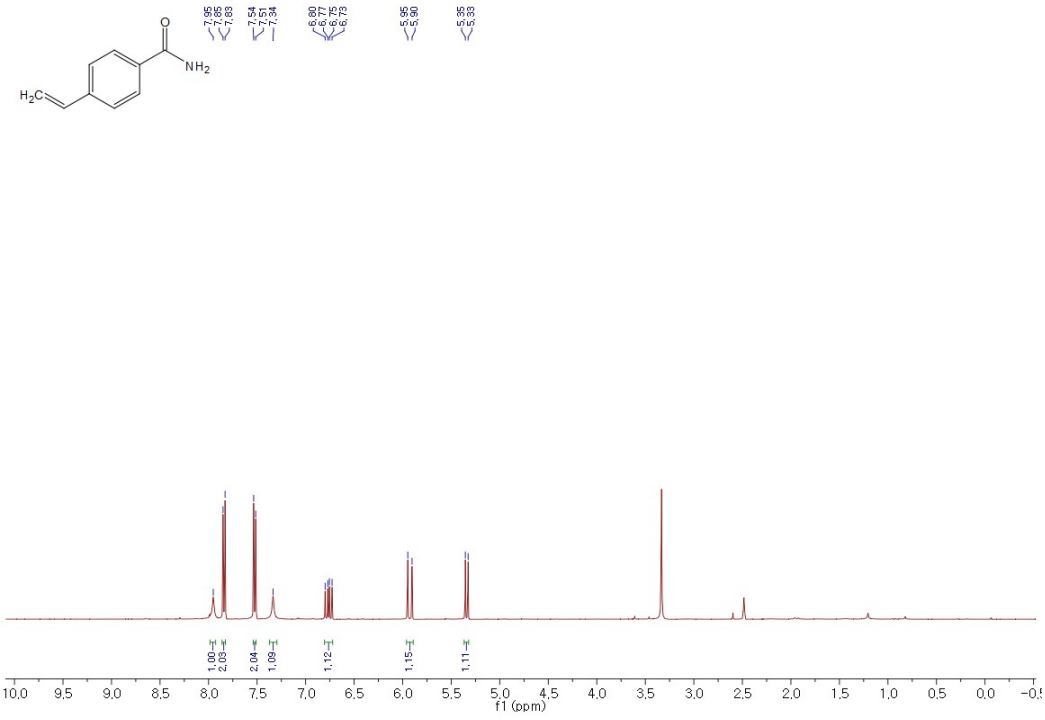
^{1}H-NMR (400 MHz, DMSO-d_6) of **2x**



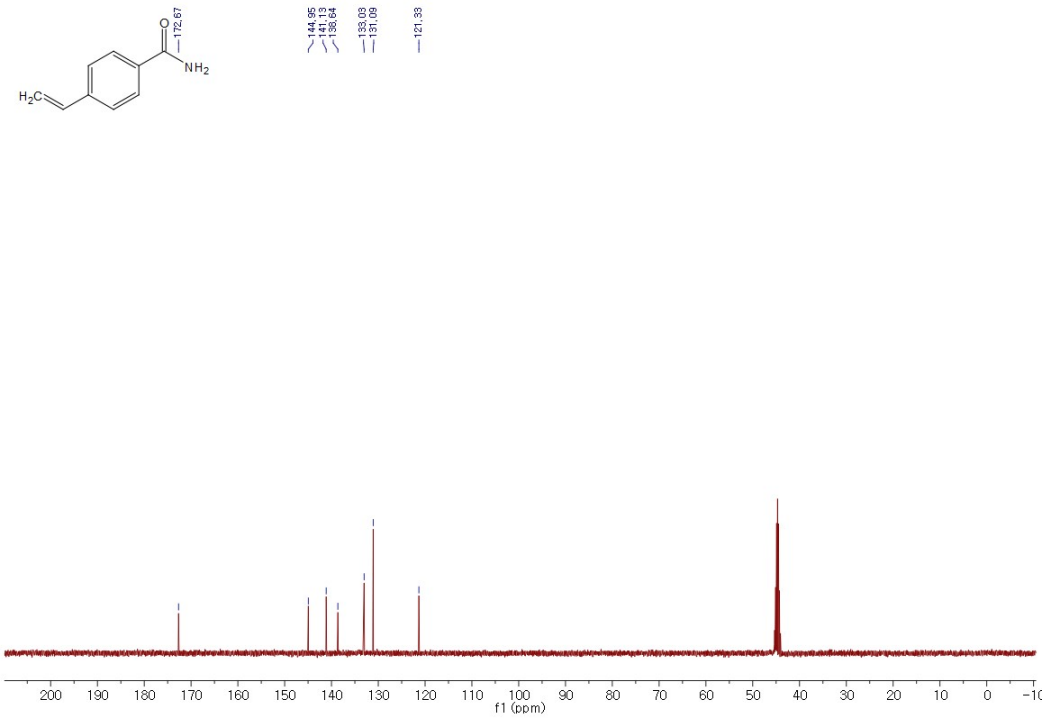
^{13}C-NMR (101 MHz, DMSO-d_6) of **2x**



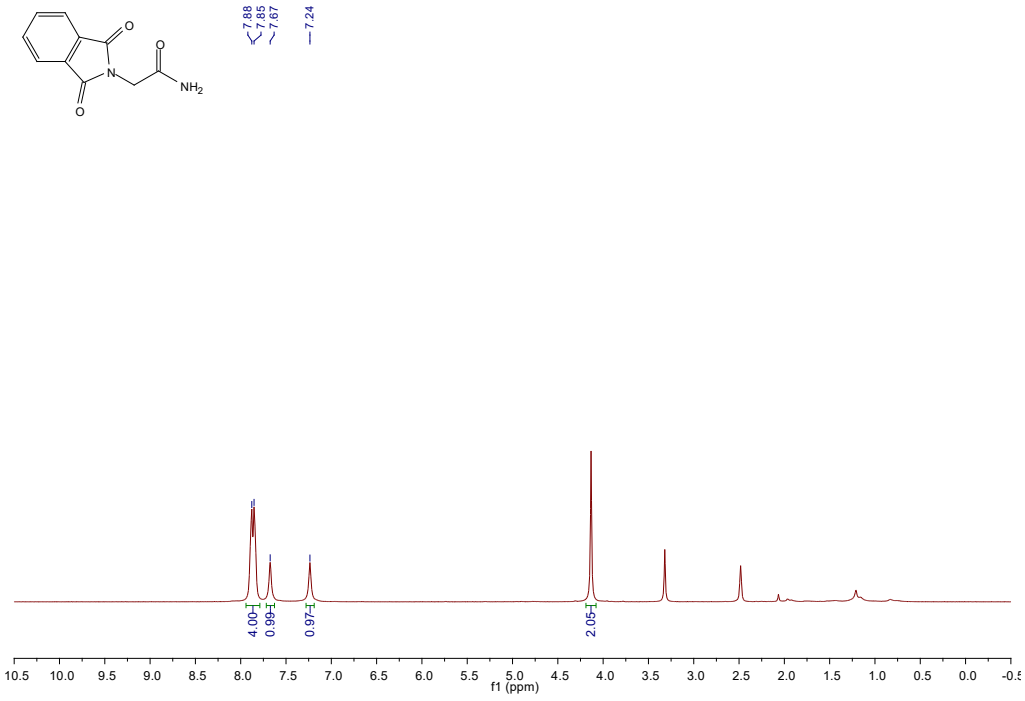
^{1}H-NMR (400 MHz, DMSO-d_6) of **2y**



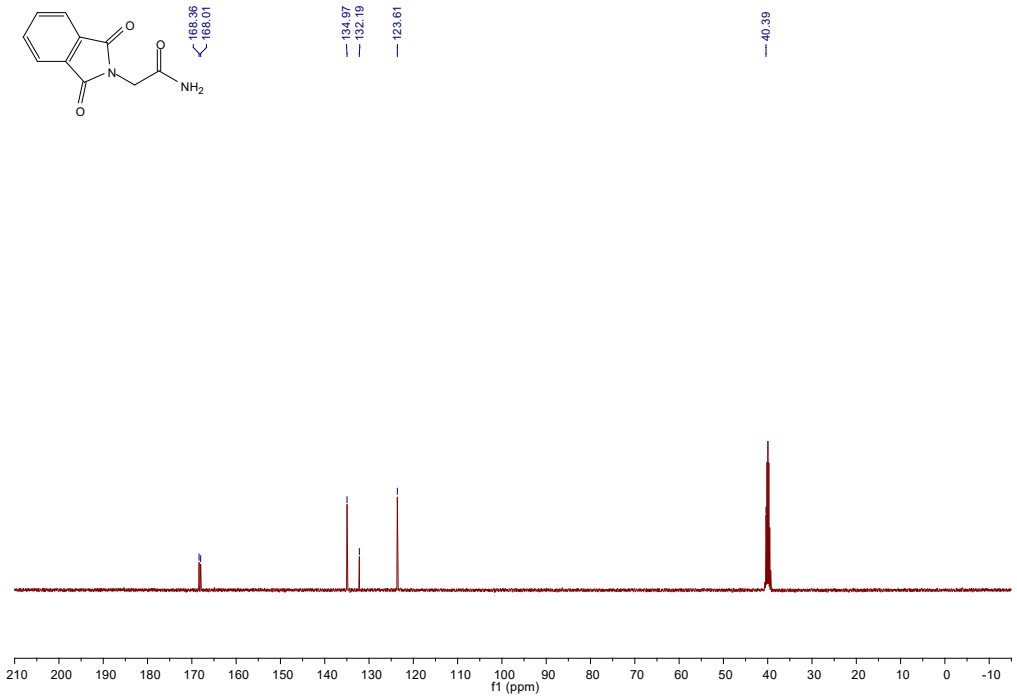
^{13}C-NMR (101 MHz, DMSO-d_6) of **2y**



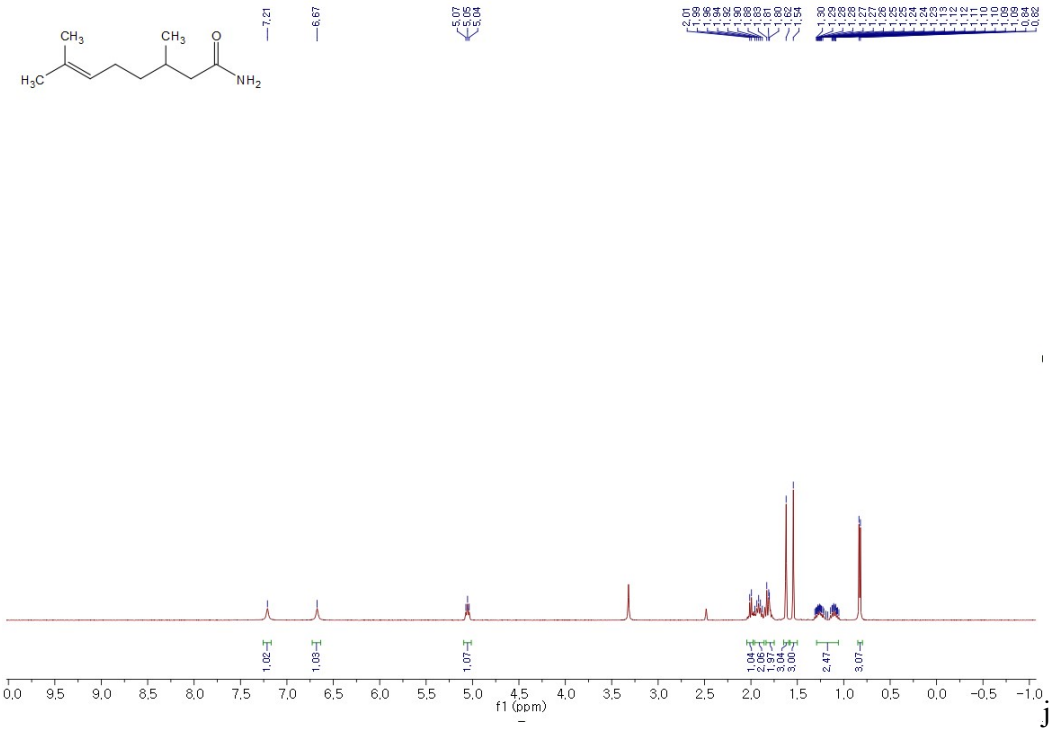
^{1}H-NMR (400 MHz, DMSO-d_6) of **4a**



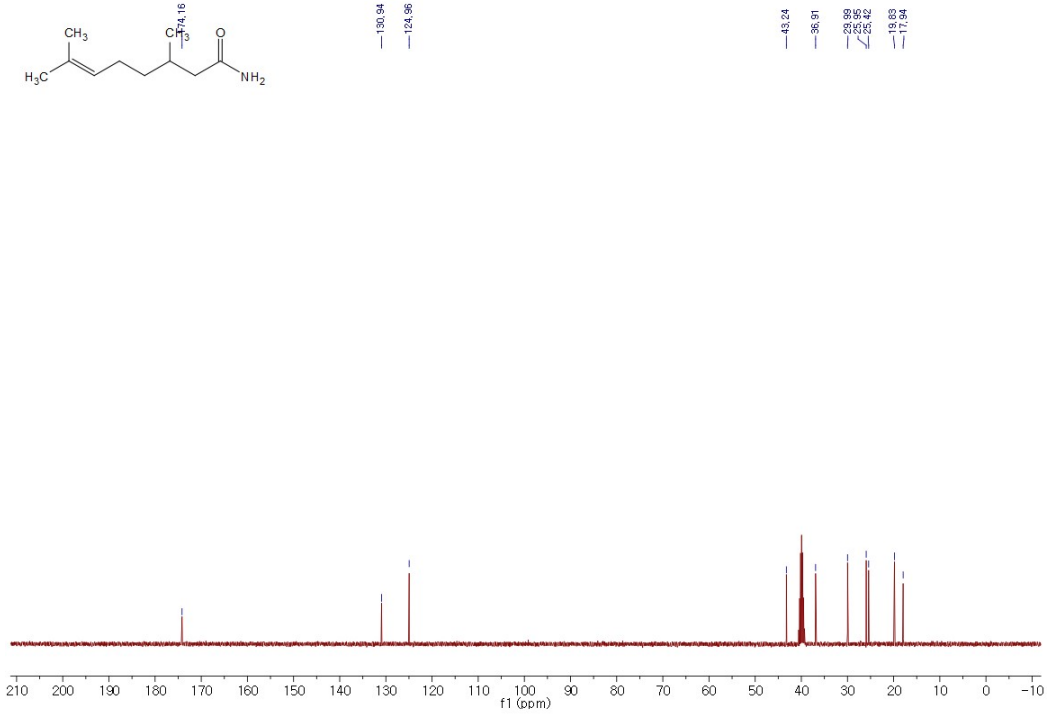
^{13}C-NMR (101 MHz, DMSO-d_6) of **4a**



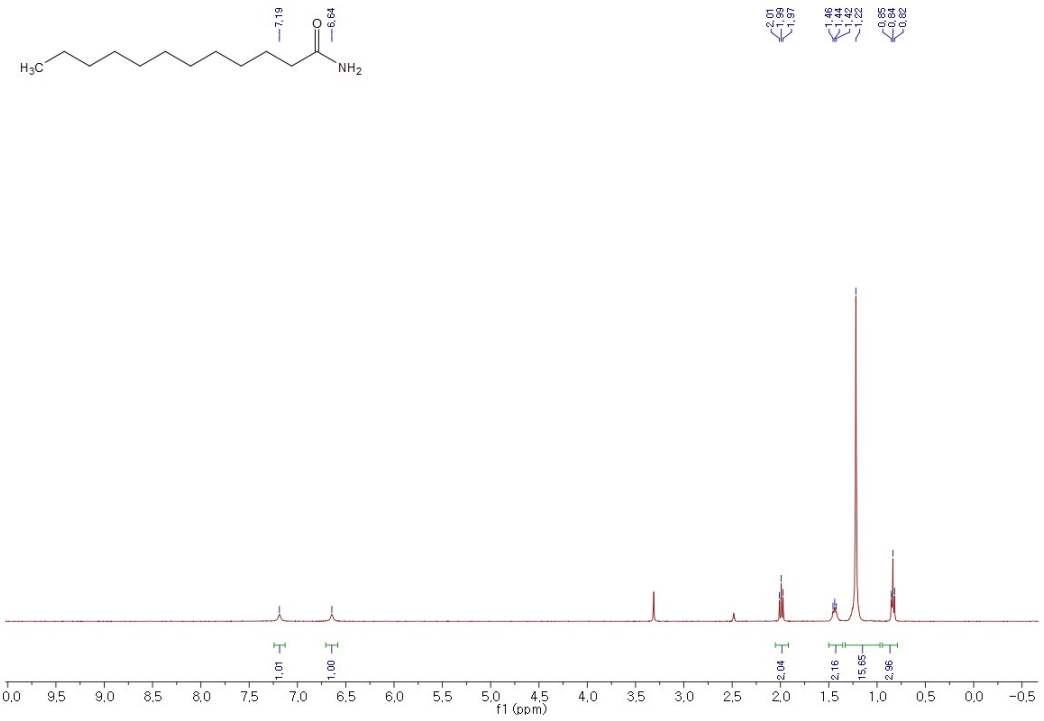
[1]H-NMR (400 MHz, DMSO-d_6) of **4b**



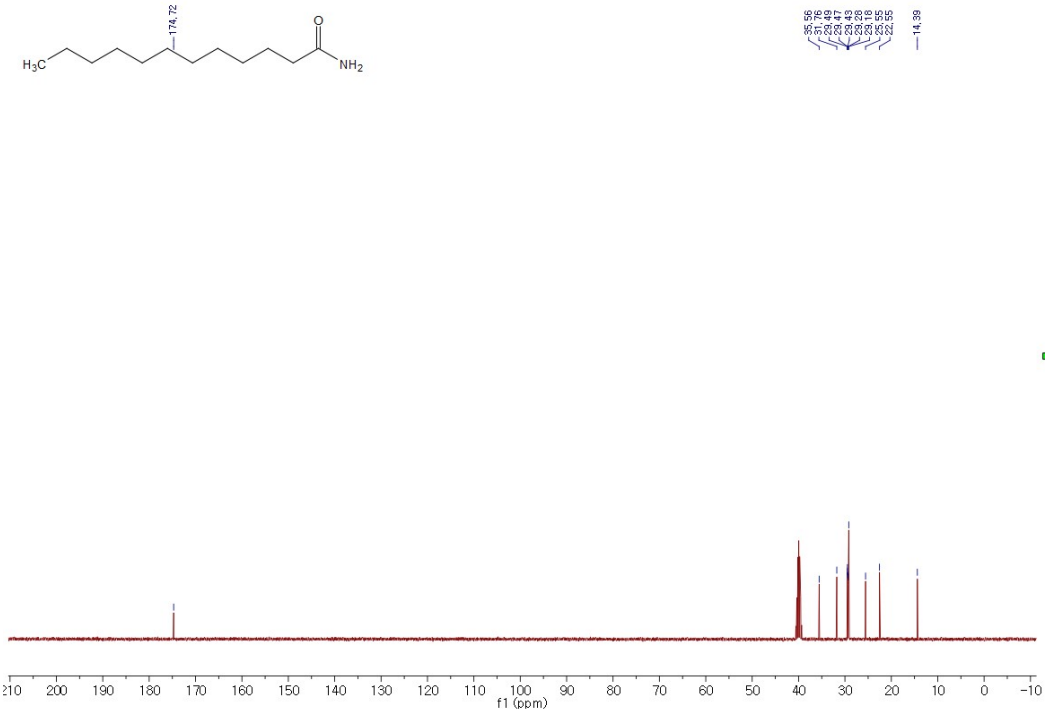
[13]C-NMR (101 MHz, DMSO-d_6) of **4b**



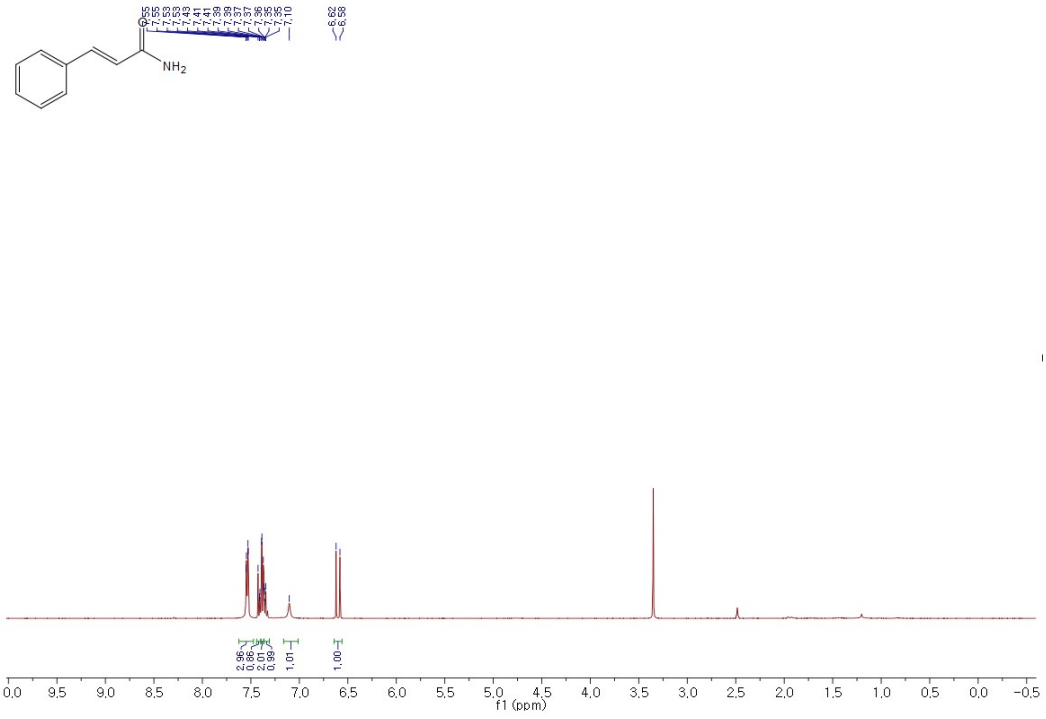
[1]H-NMR (400 MHz, DMSO-d_6) of **4c**



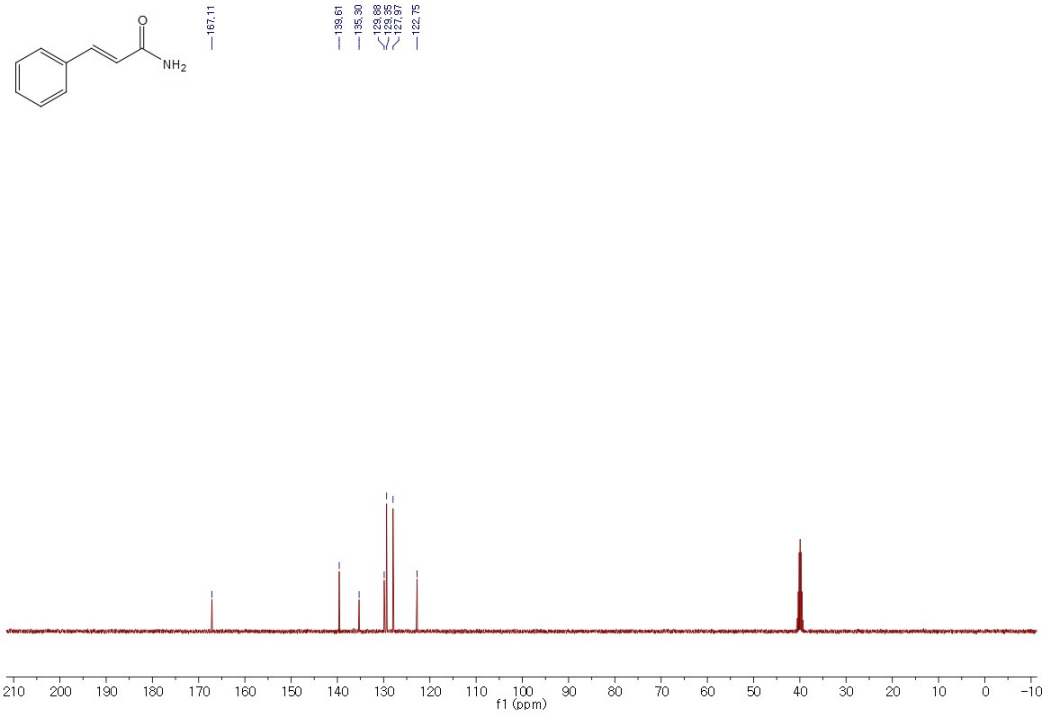
[13]C-NMR (101 MHz, DMSO-d_6) of **4c**



[1]H-NMR (400 MHz, DMSO-d_6) of **4d**



[13]C-NMR (101 MHz, DMSO-d_6) of **4d**



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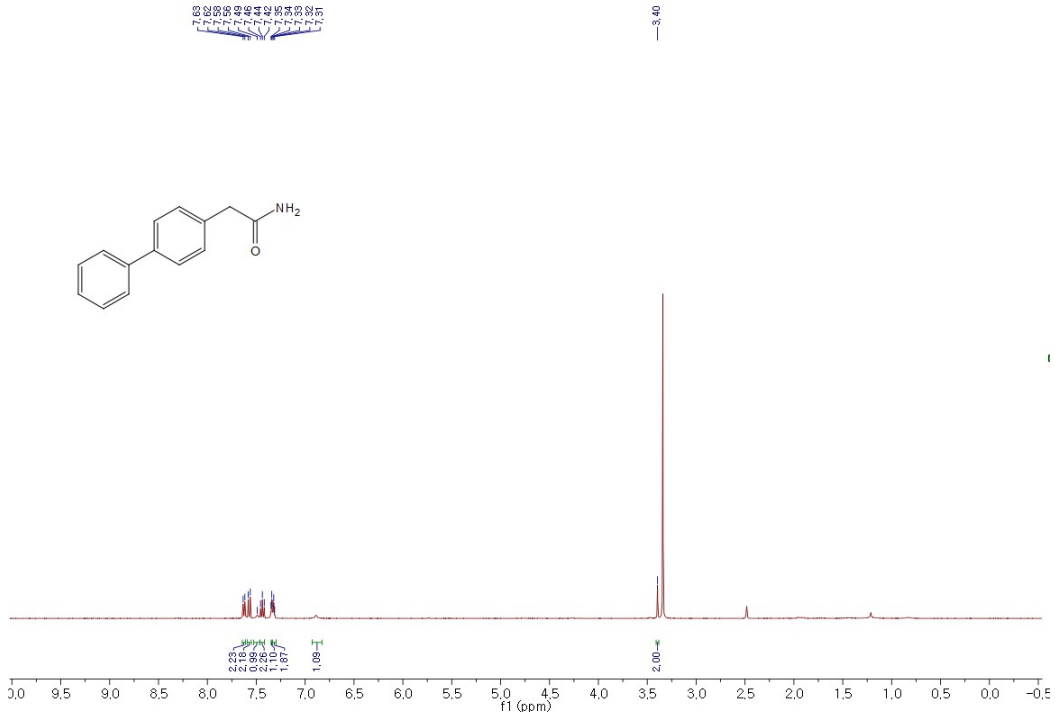
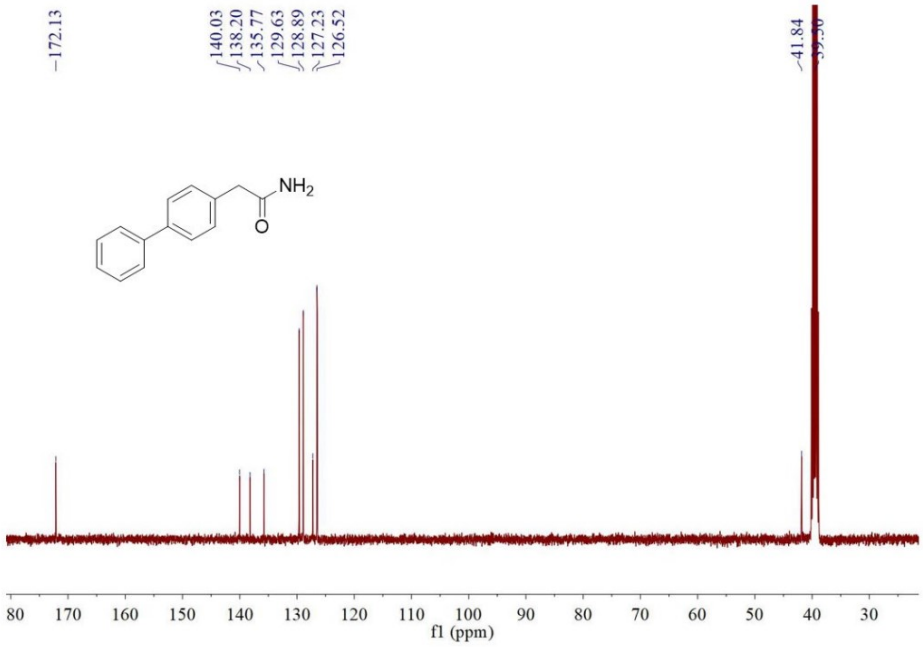
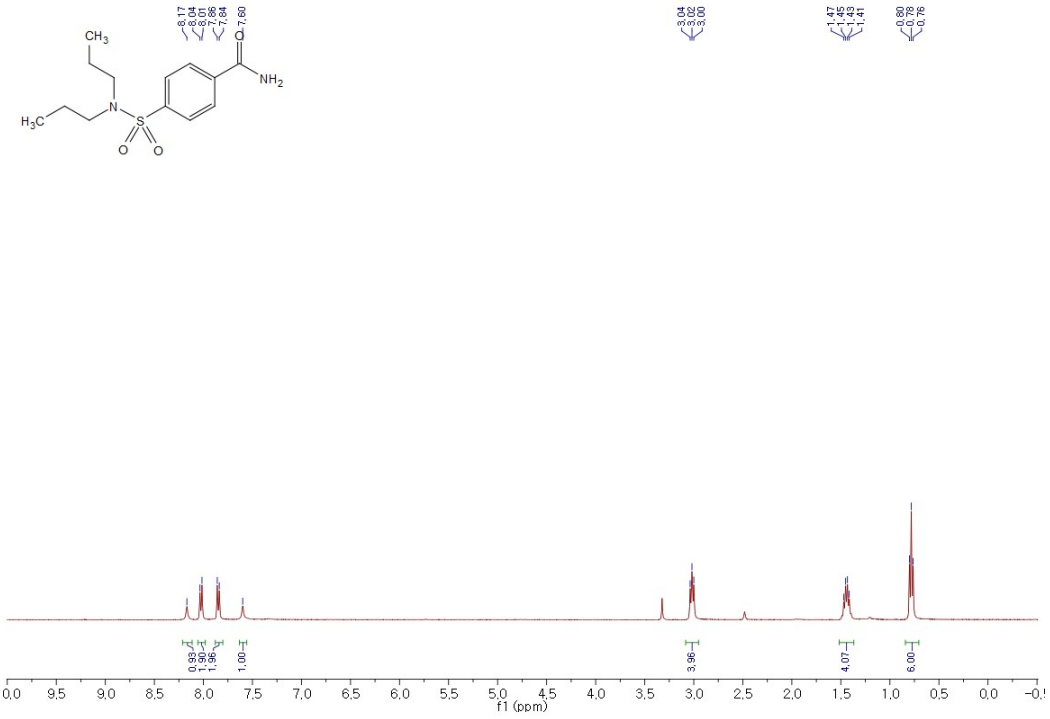


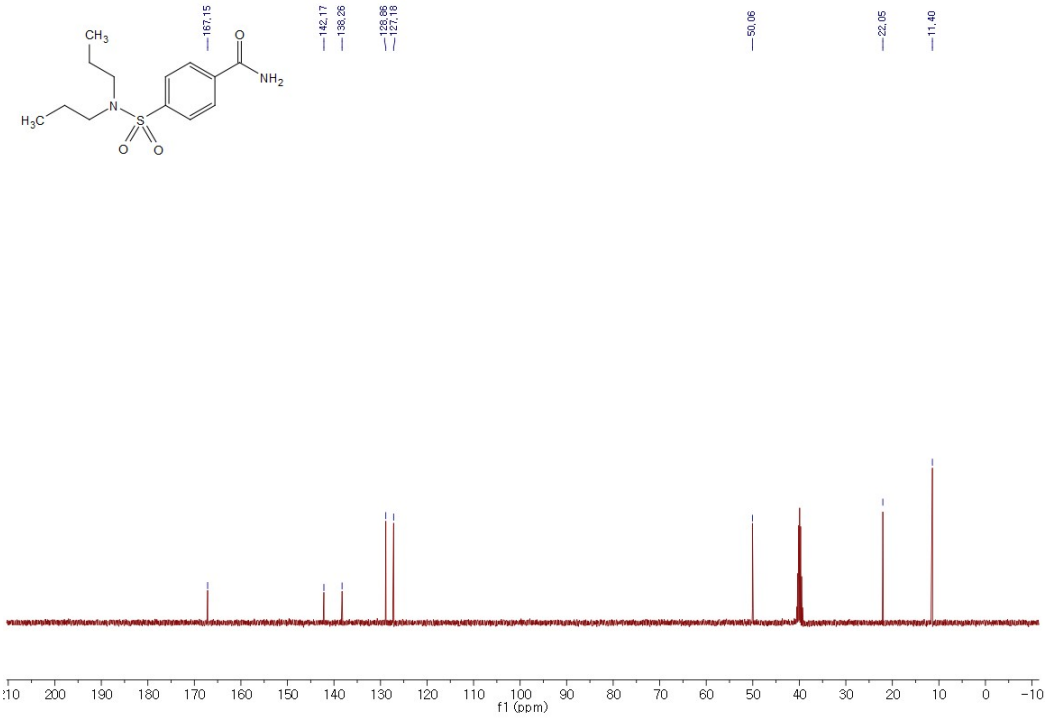
[13]C-NMR (101 MHz, DMSO-d_6) of **4e**



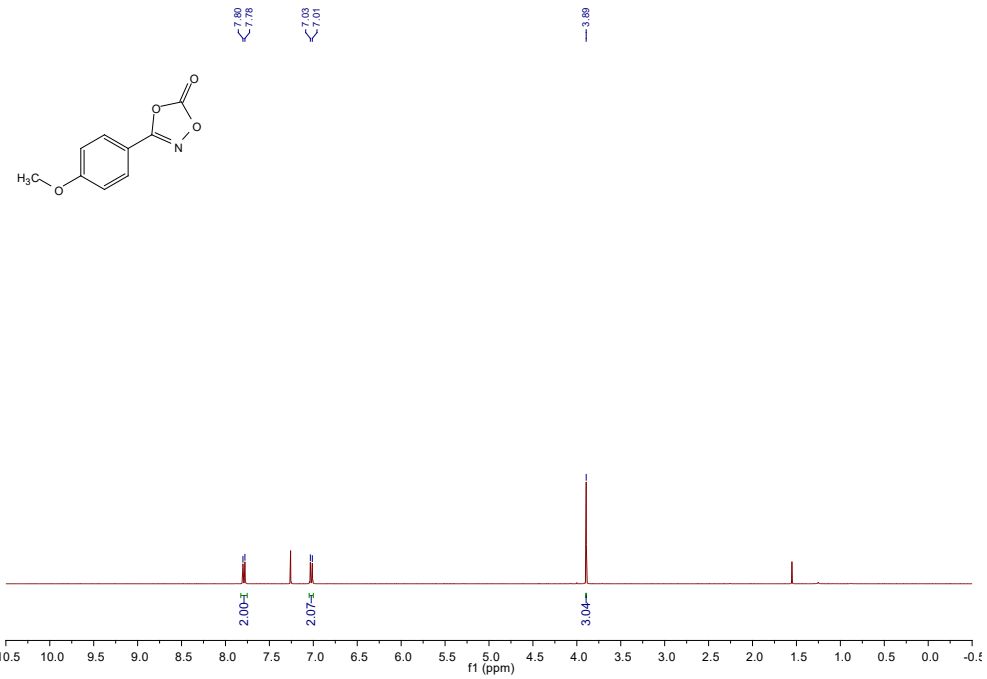
^{1}H-NMR (400 MHz, DMSO-d_6) of **4f**



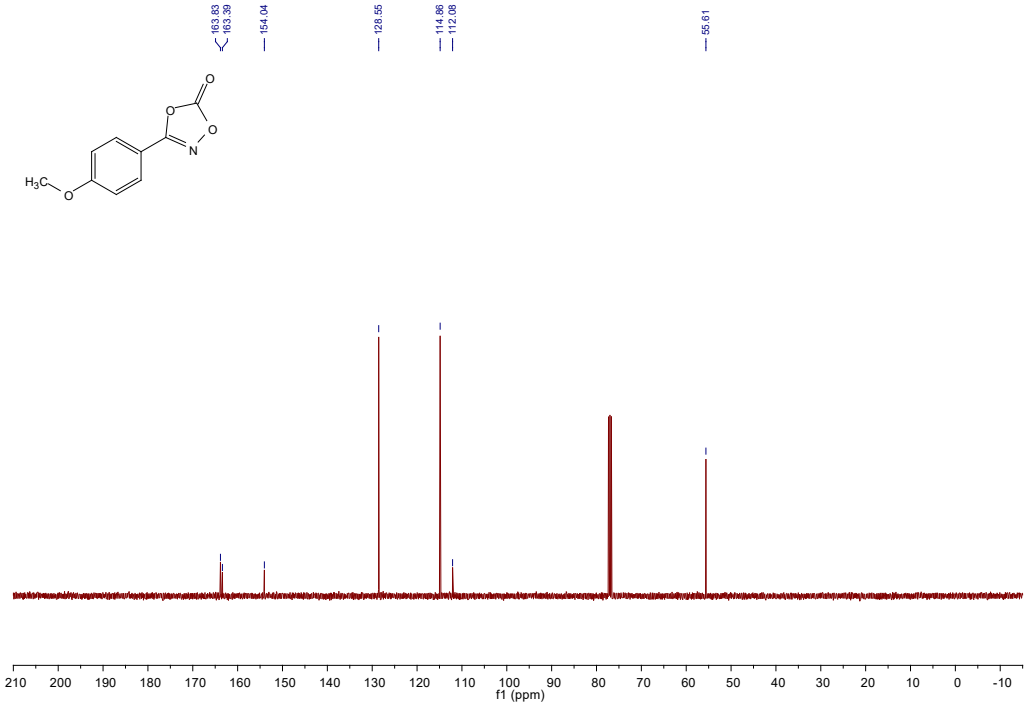
^{13}C-NMR (101 MHz, DMSO-d_6) of **4f**



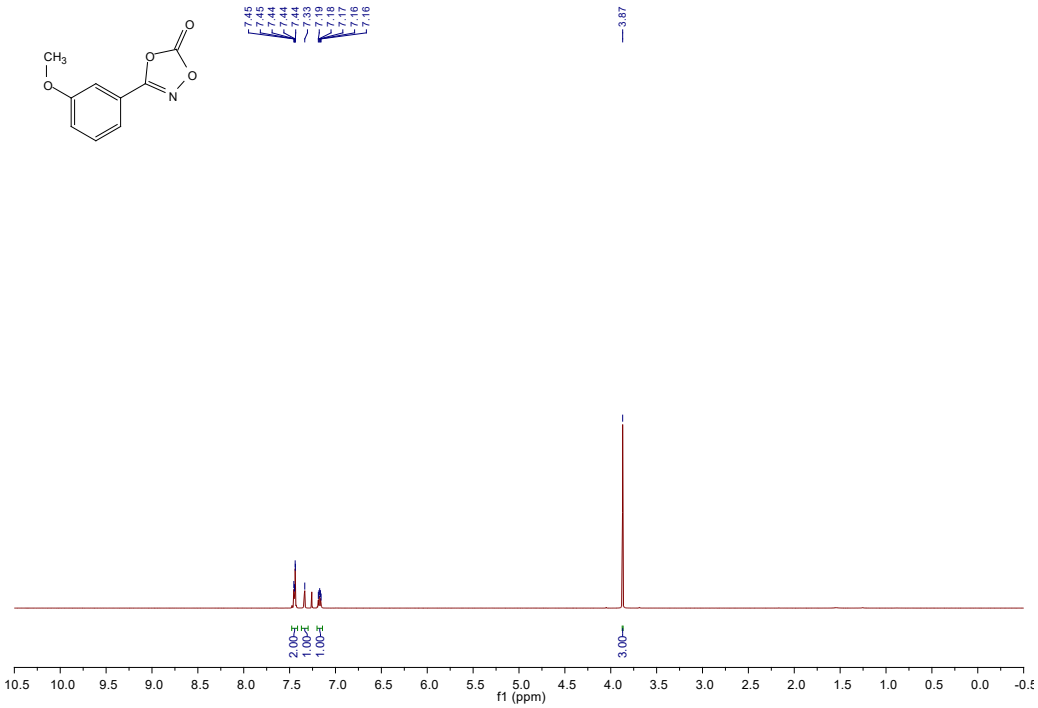
^{1}H-NMR (400 MHz, $CDCl_3$) of **1a**



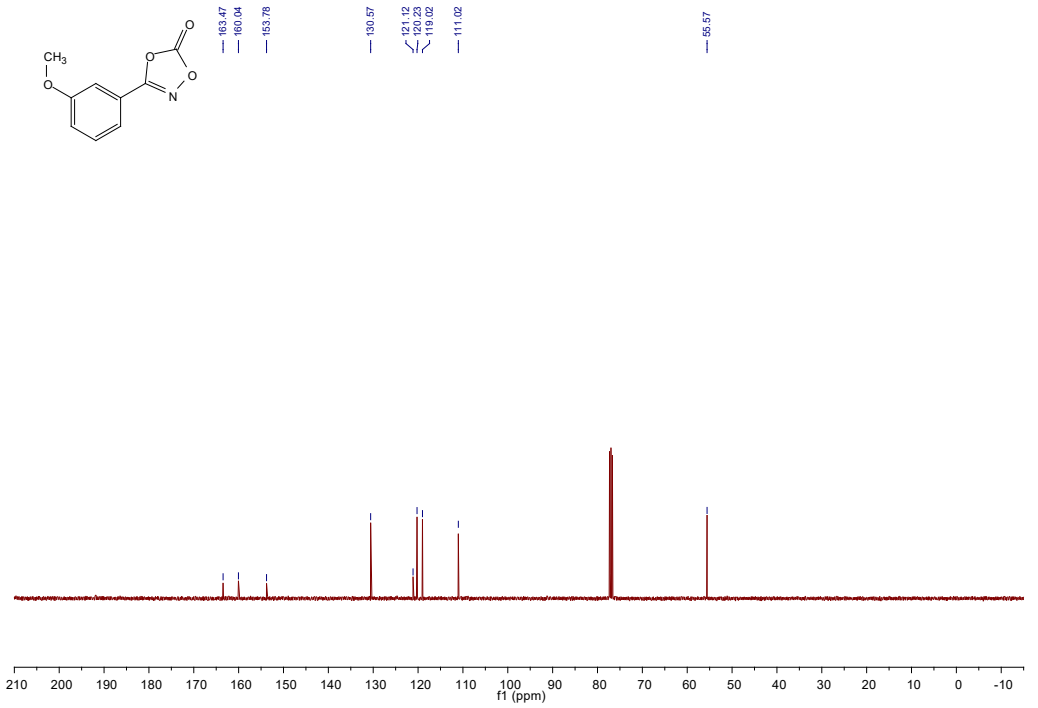
^{13}C-NMR (101 MHz, $CDCl_3$) of **1a**



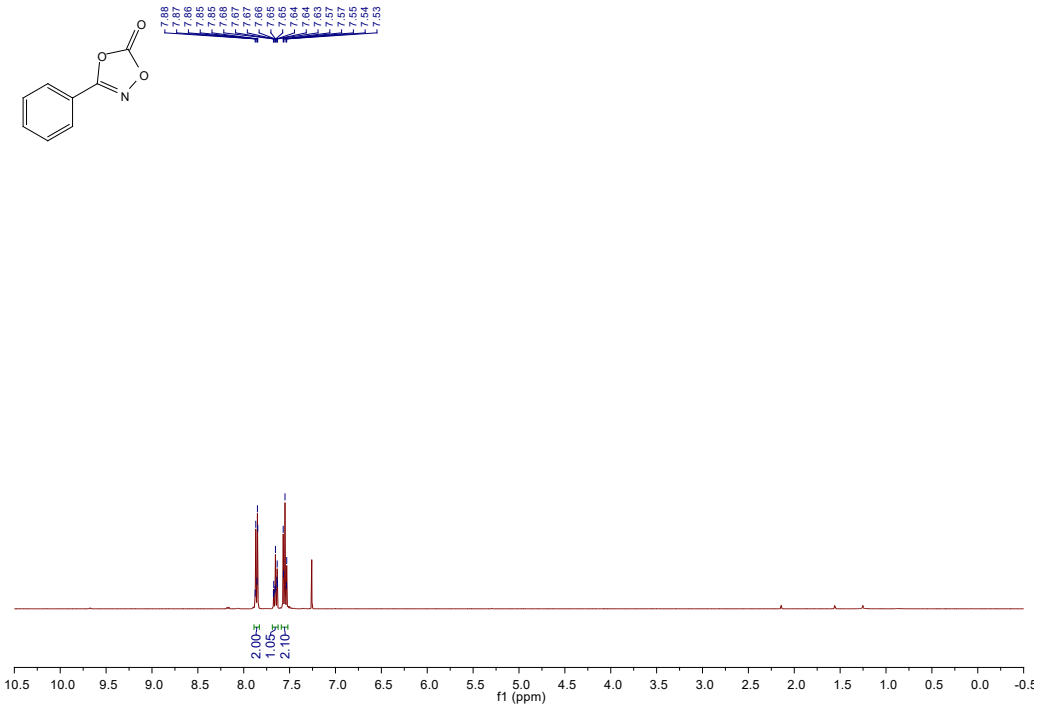
^{1}H-NMR (400 MHz, $CDCl_3$) of **1b**



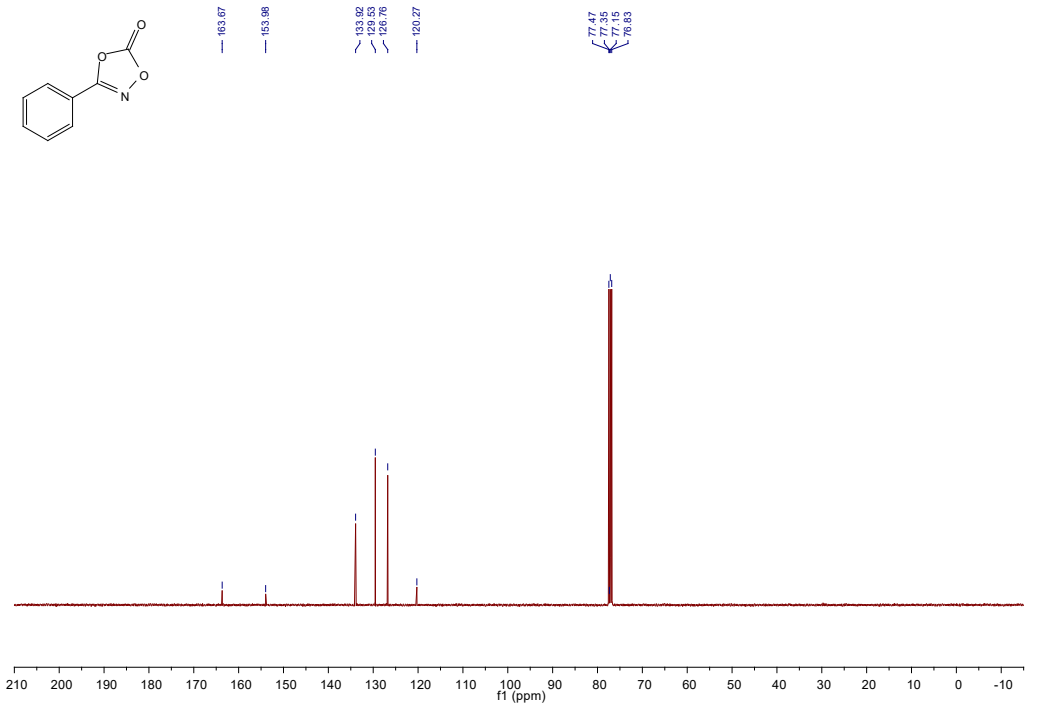
^{13}C-NMR (101 MHz, $CDCl_3$) of **1b**



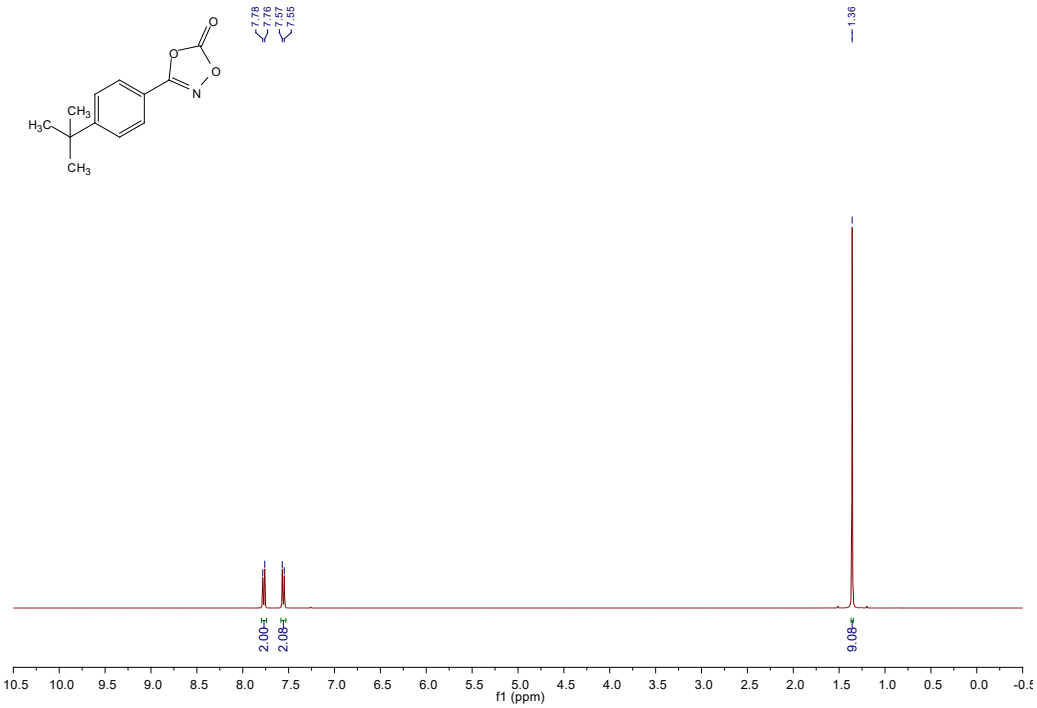
^{1}H-NMR (400 MHz, $CDCl_3$) of **1c**



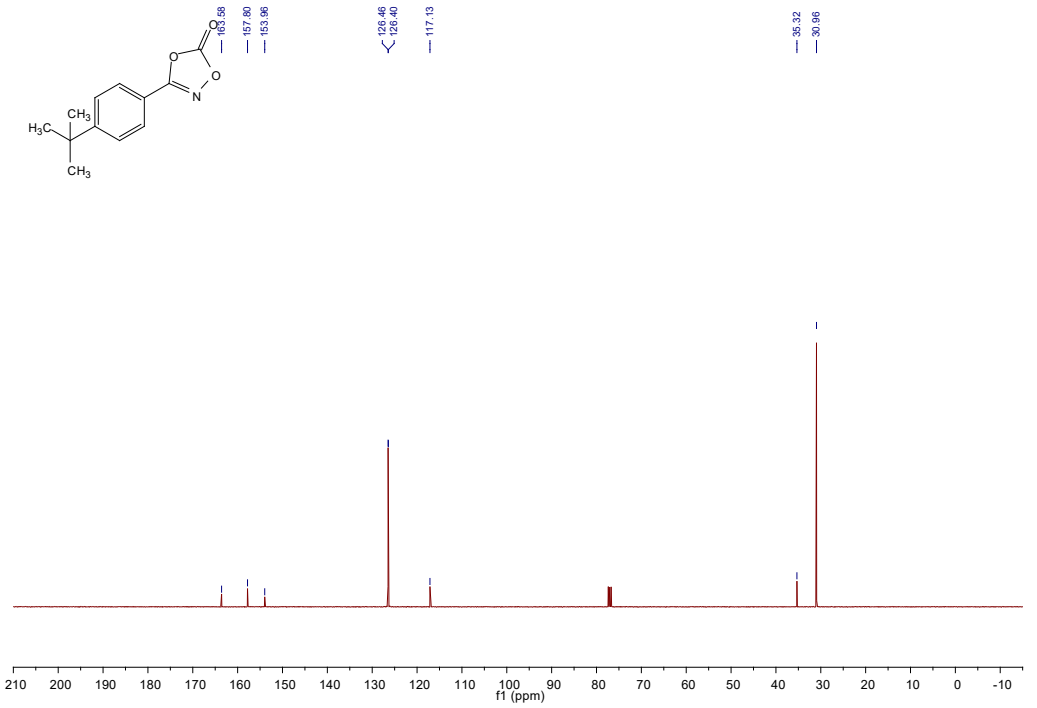
^{13}C-NMR (101 MHz, $CDCl_3$) of **1c**



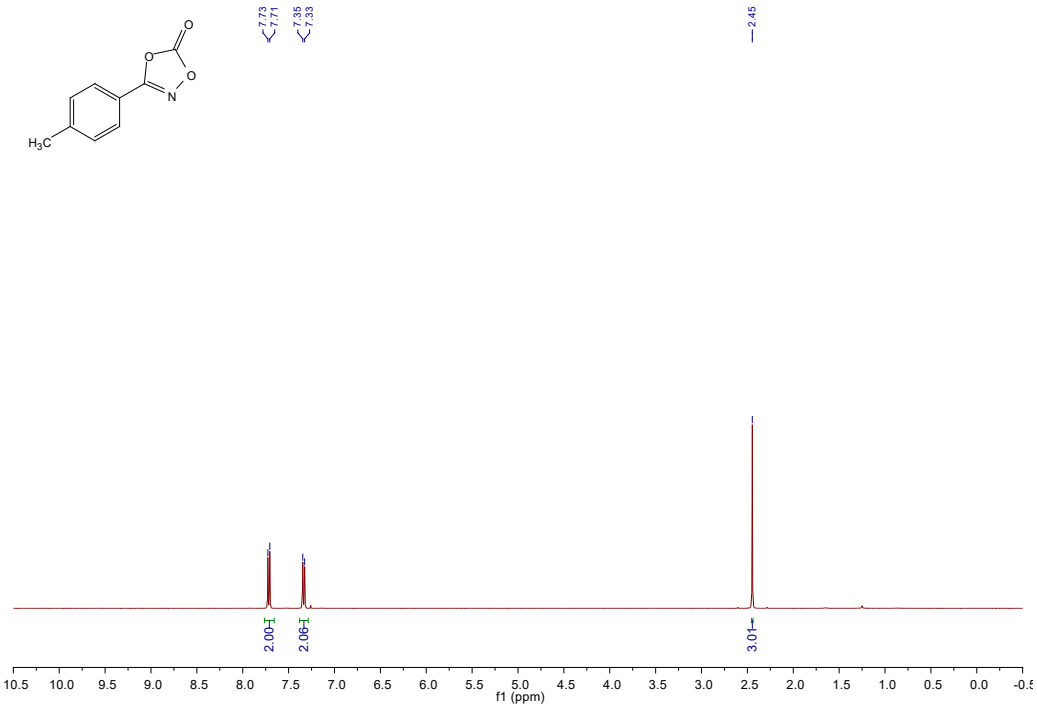
^{1}H-NMR (400 MHz, $CDCl_3$) of **1d**



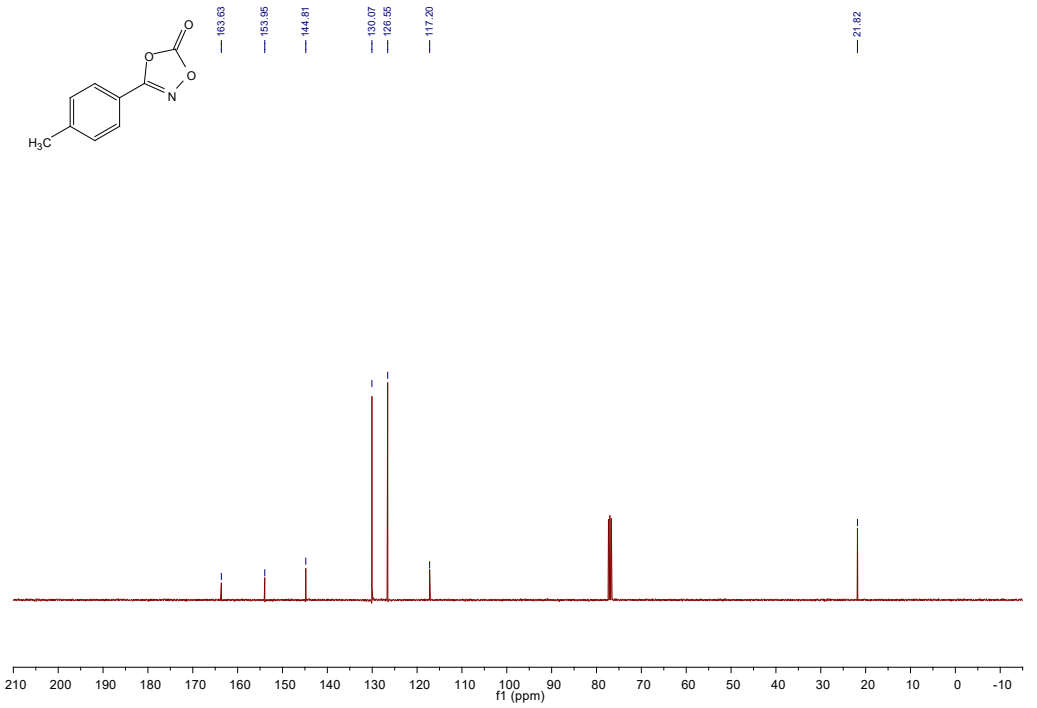
^{13}C-NMR (101 MHz, $CDCl_3$) of **1d**



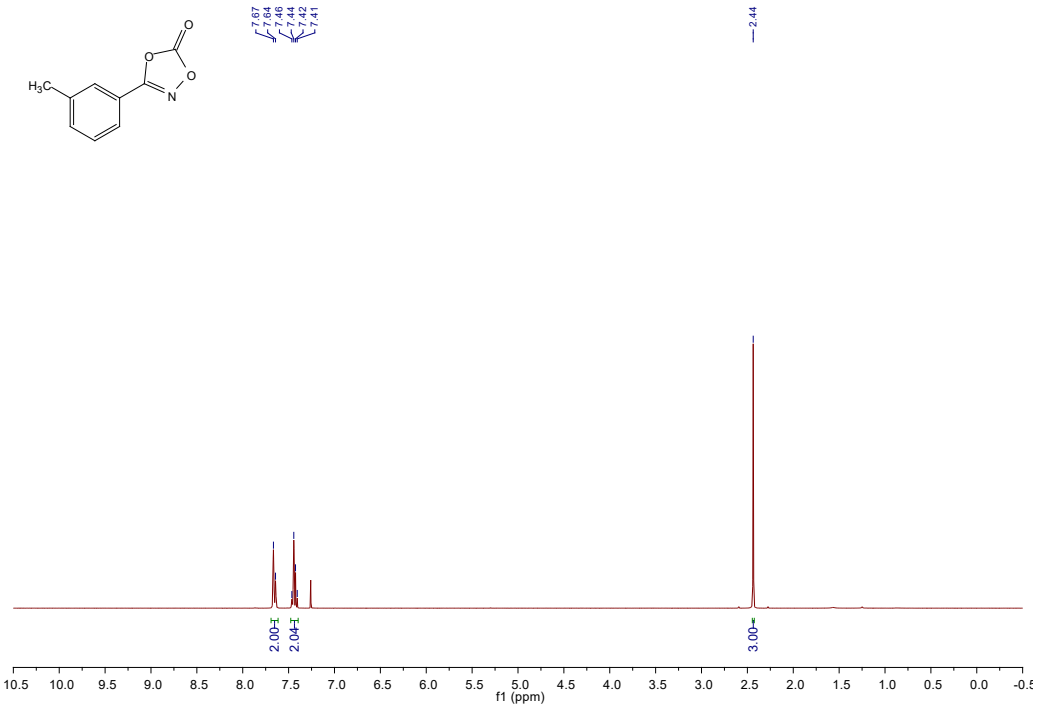
^{1}H-NMR (400 MHz, $CDCl_3$) of **1e**



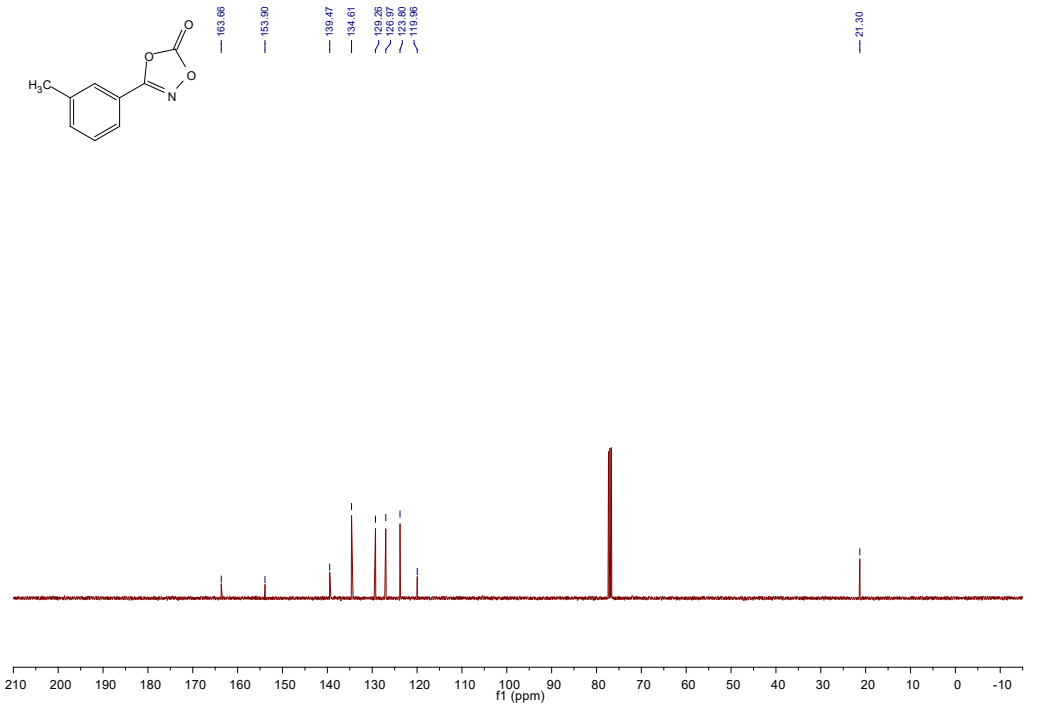
^{13}C-NMR (101 MHz, $CDCl_3$) of **1e**



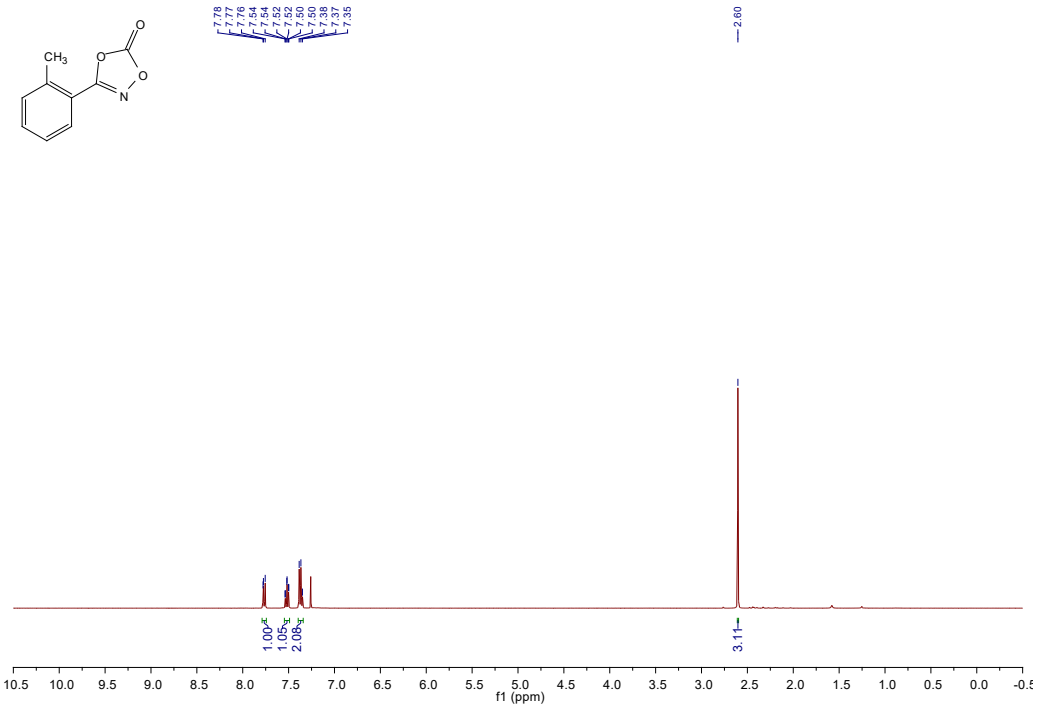
^{1}H-NMR (400 MHz, $CDCl_3$) of **1f**



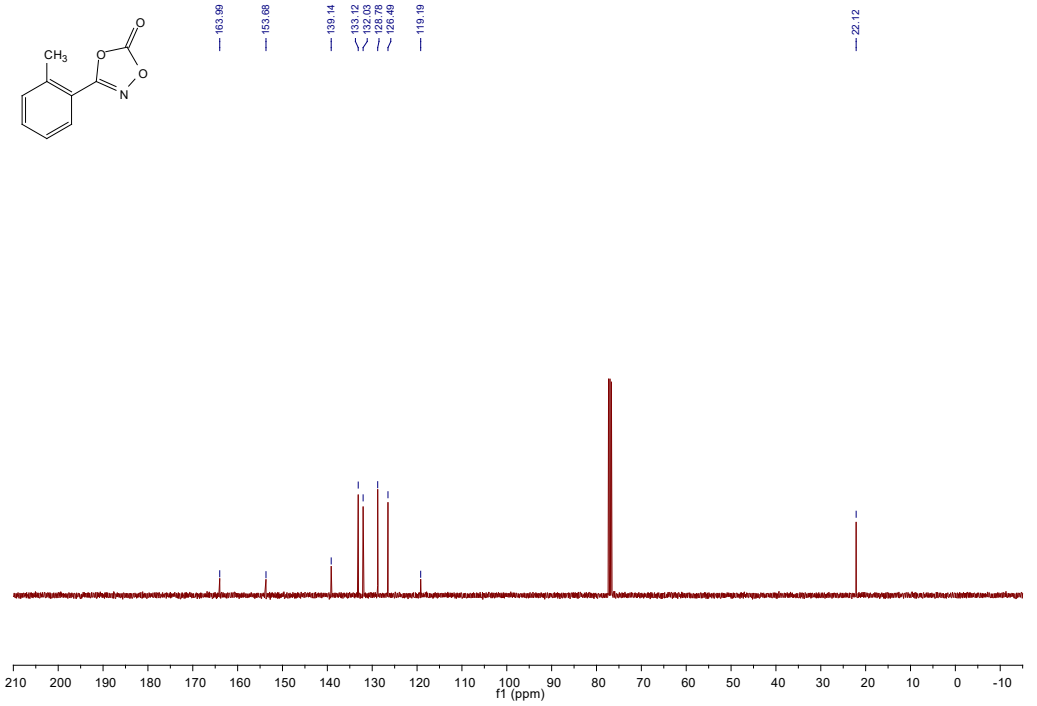
^{13}C-NMR (101 MHz, $CDCl_3$) of **1f**



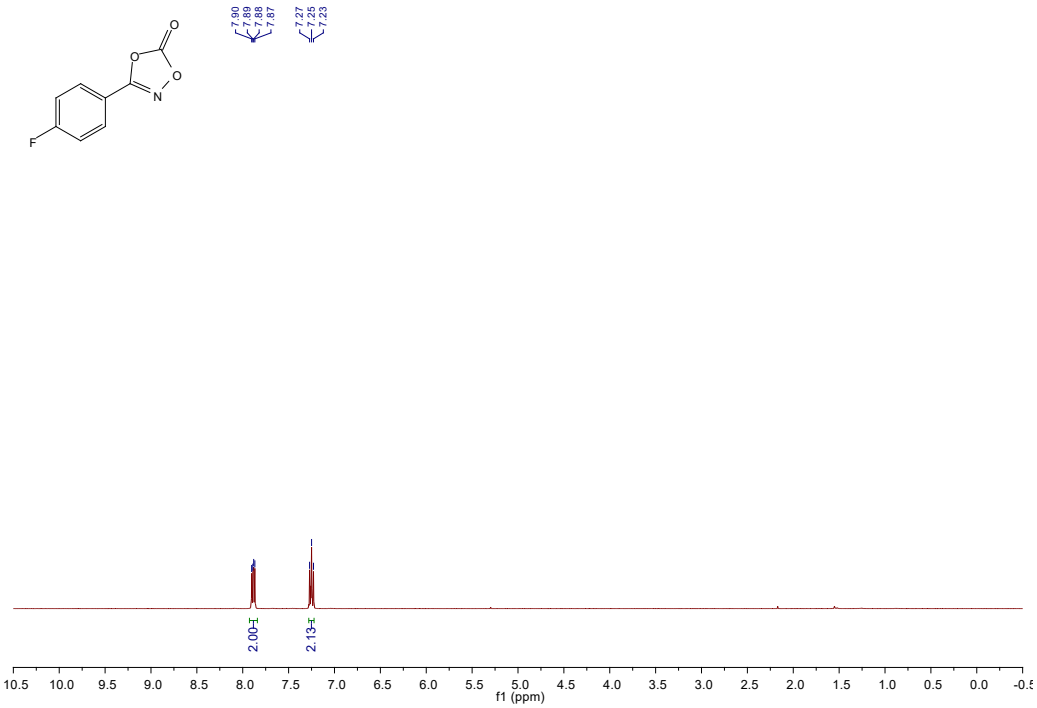
^{1}H-NMR (400 MHz, $CDCl_3$) of **1g**



^{13}C-NMR (101 MHz, $CDCl_3$) of **1g**



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



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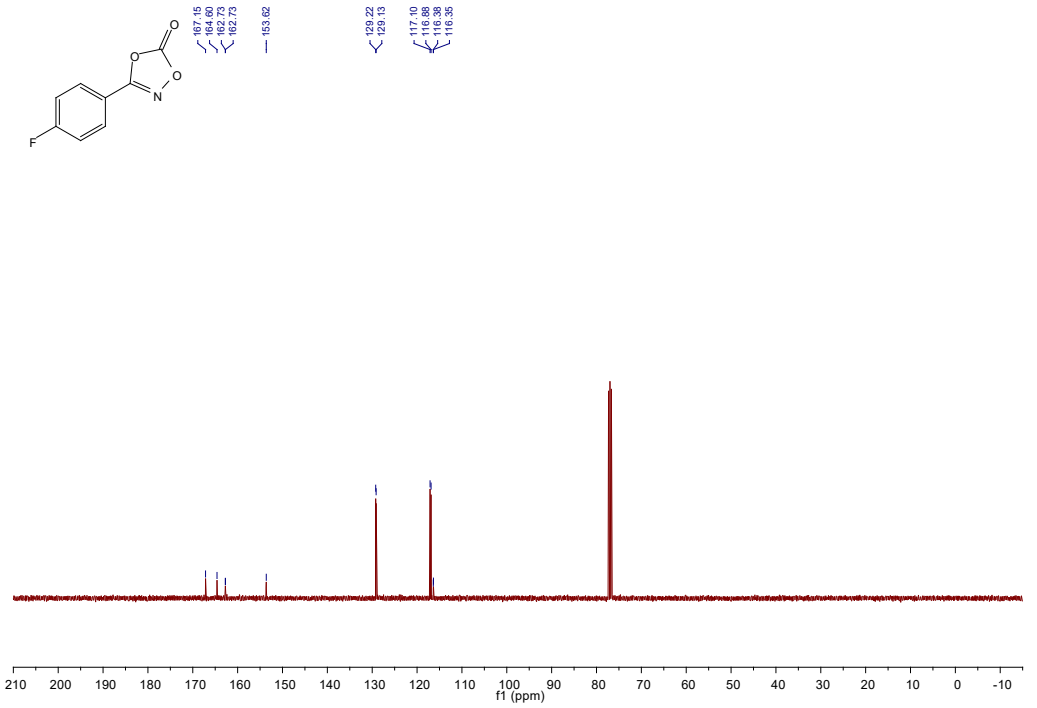
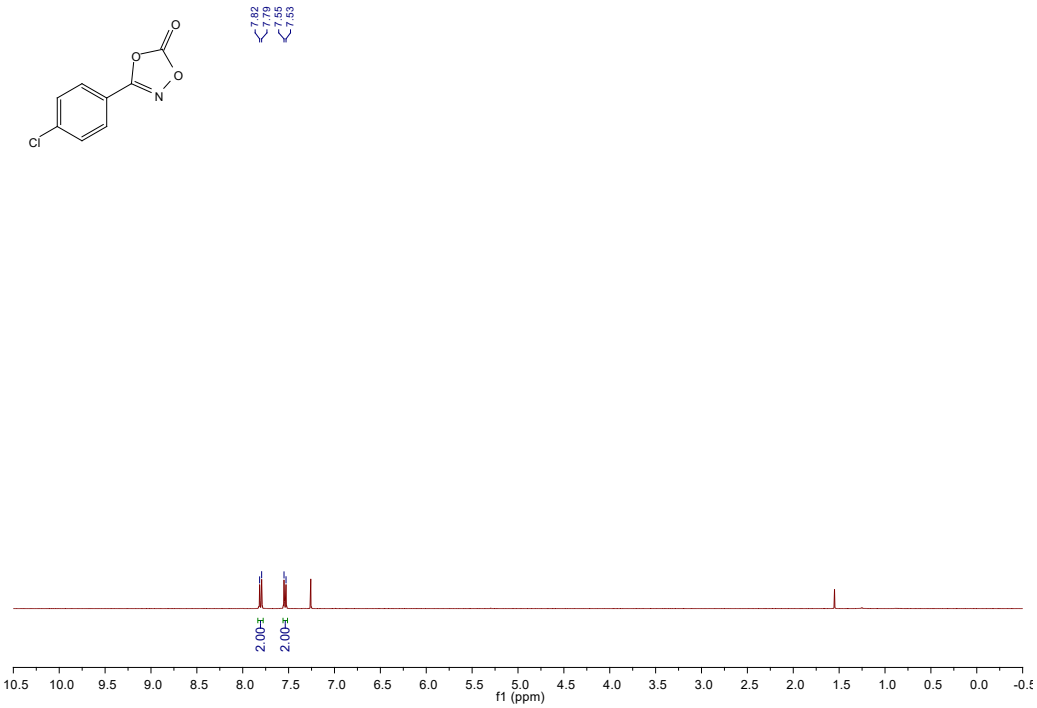
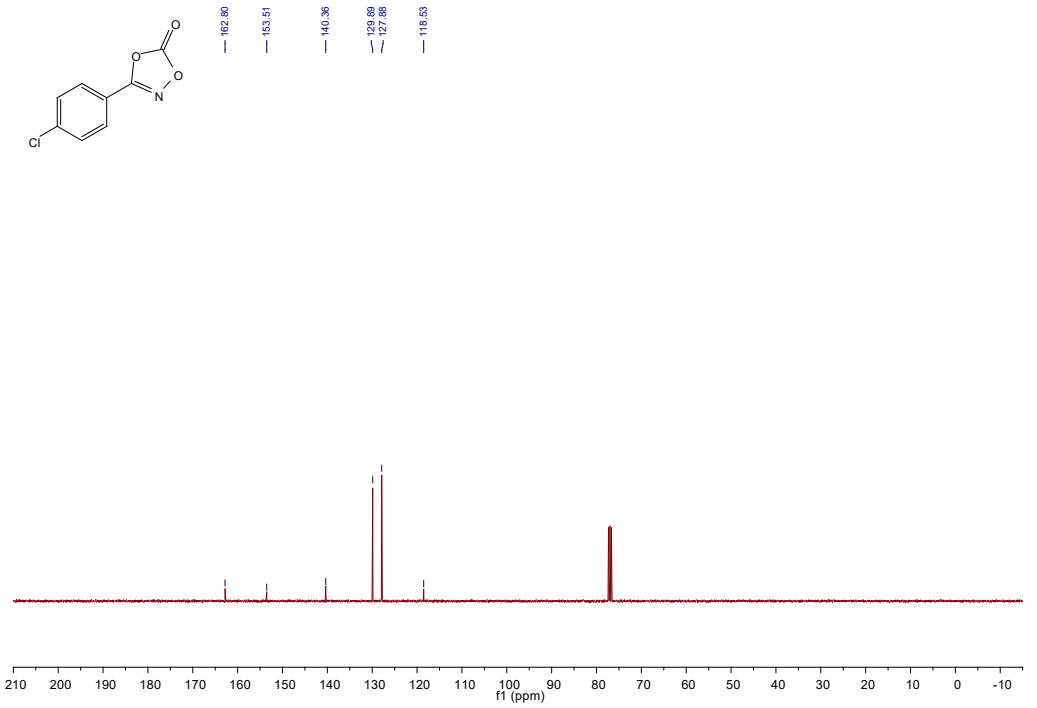


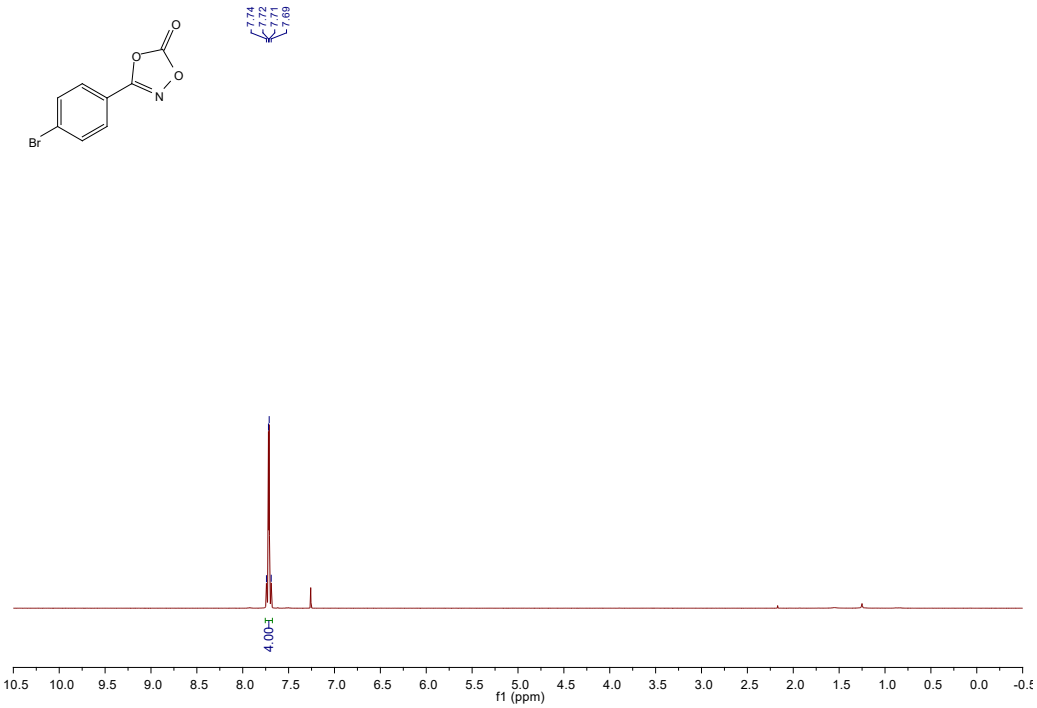
^{1}H-NMR (400 MHz, $CDCl_3$) of **1i**



^{13}C-NMR (101 MHz, $CDCl_3$) of **1i**



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



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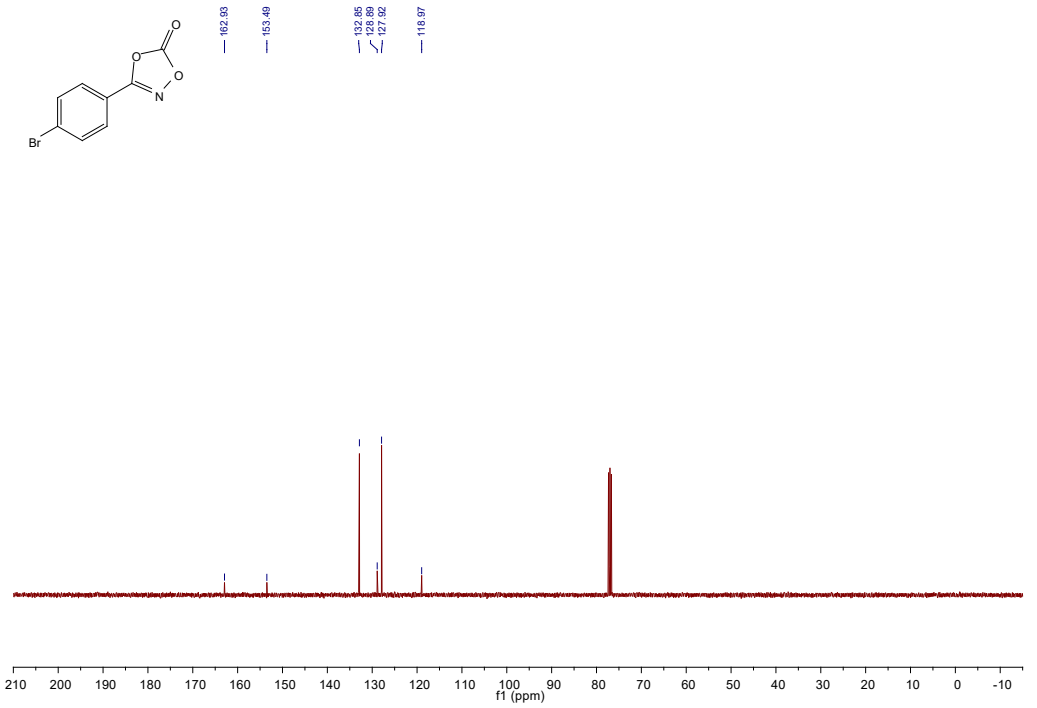
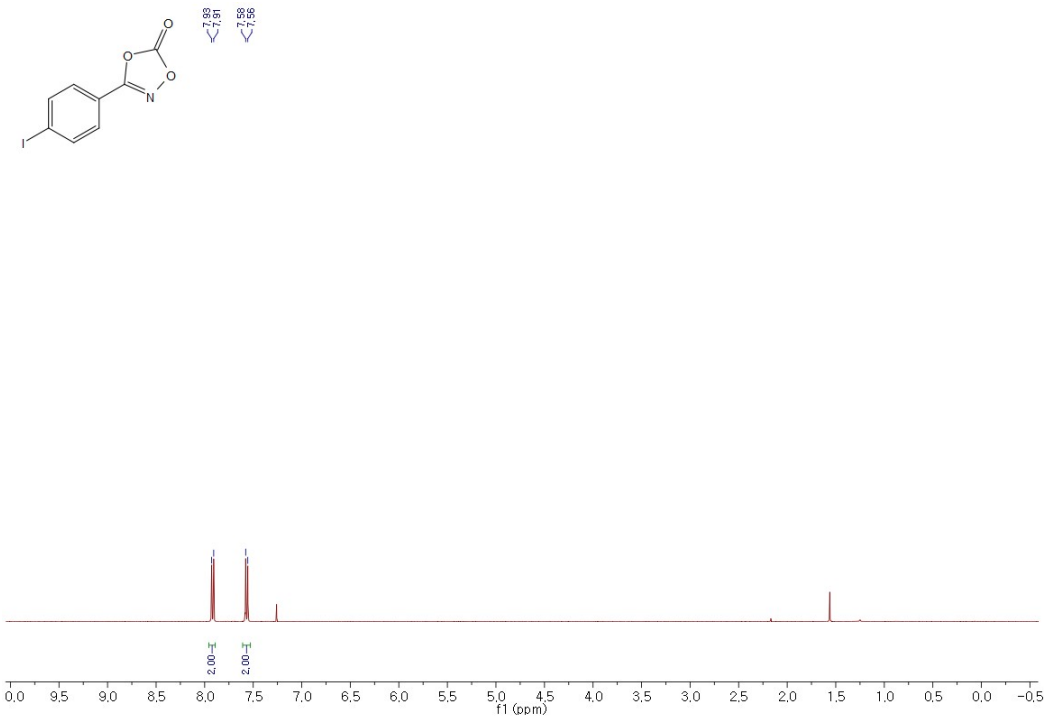
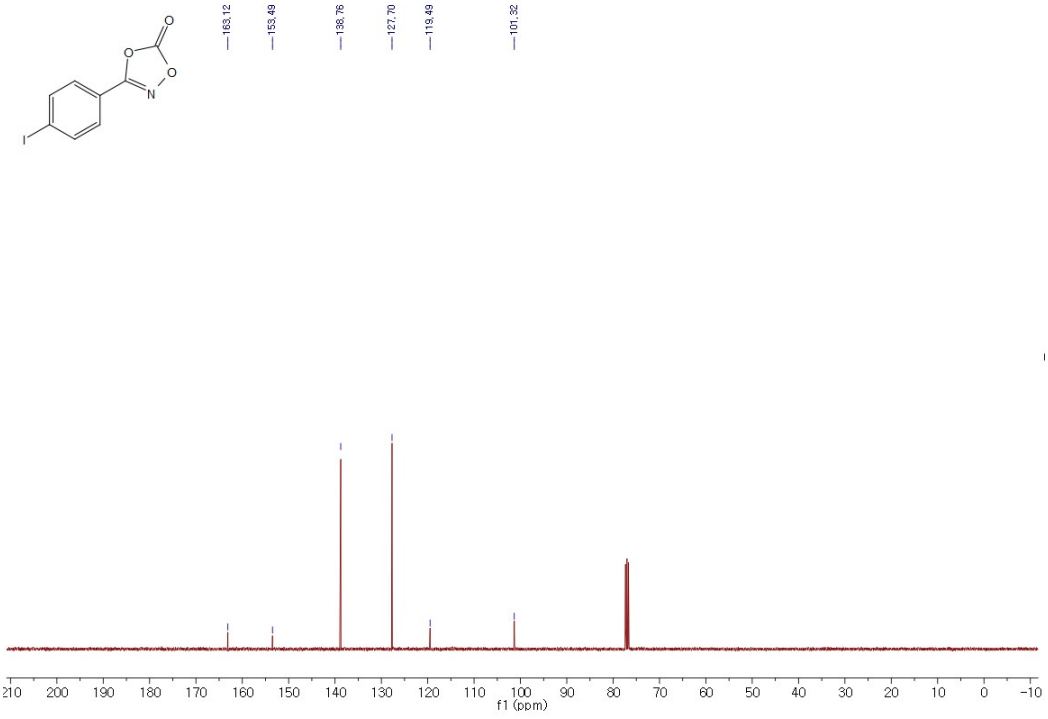


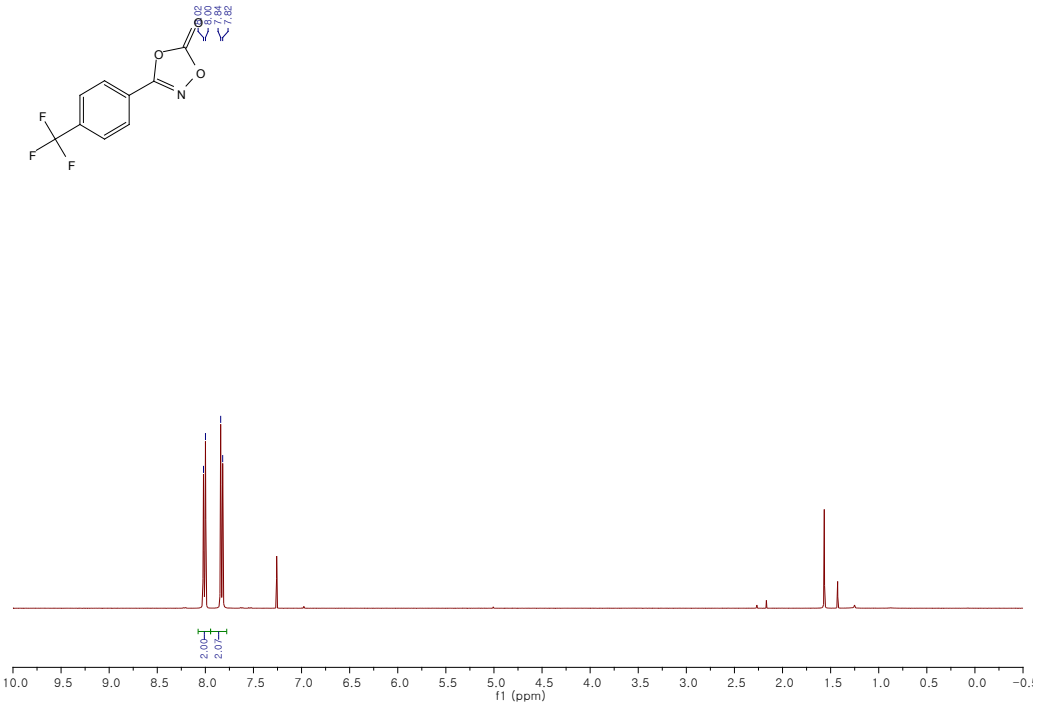
^{1}H-NMR (400 MHz, $CDCl_3$) of **1k**



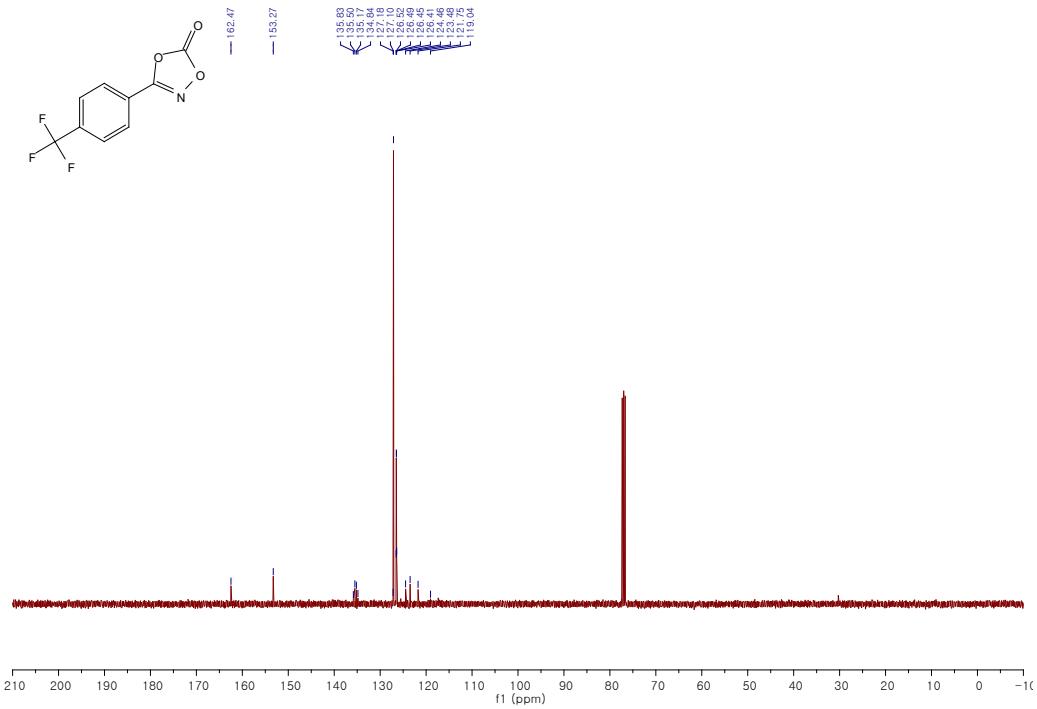
^{13}C-NMR (101 MHz, $CDCl_3$) of **1k**



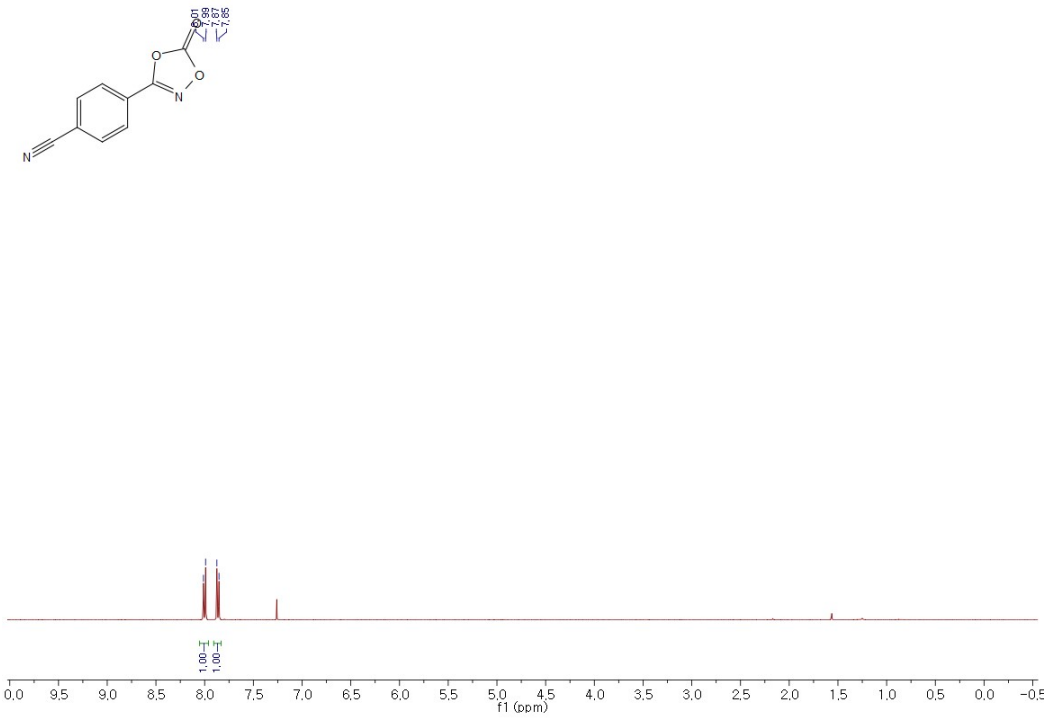
^{1}H-NMR (400 MHz, $CDCl_3$) of **1l**



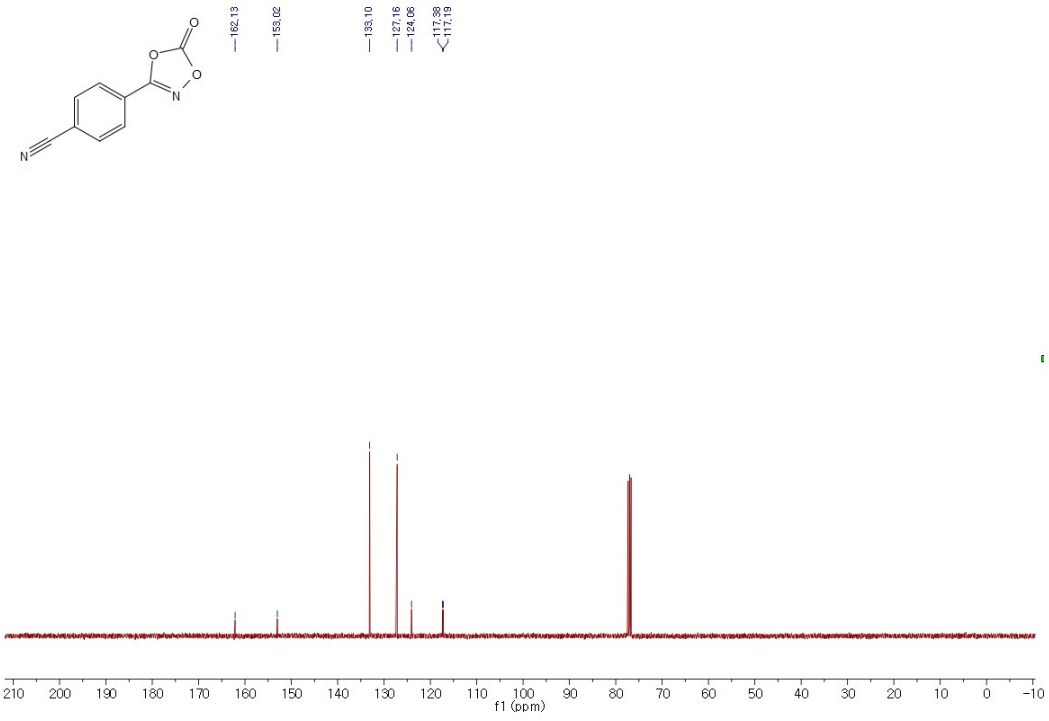
^{13}C-NMR (101 MHz, $CDCl_3$) of **1l**



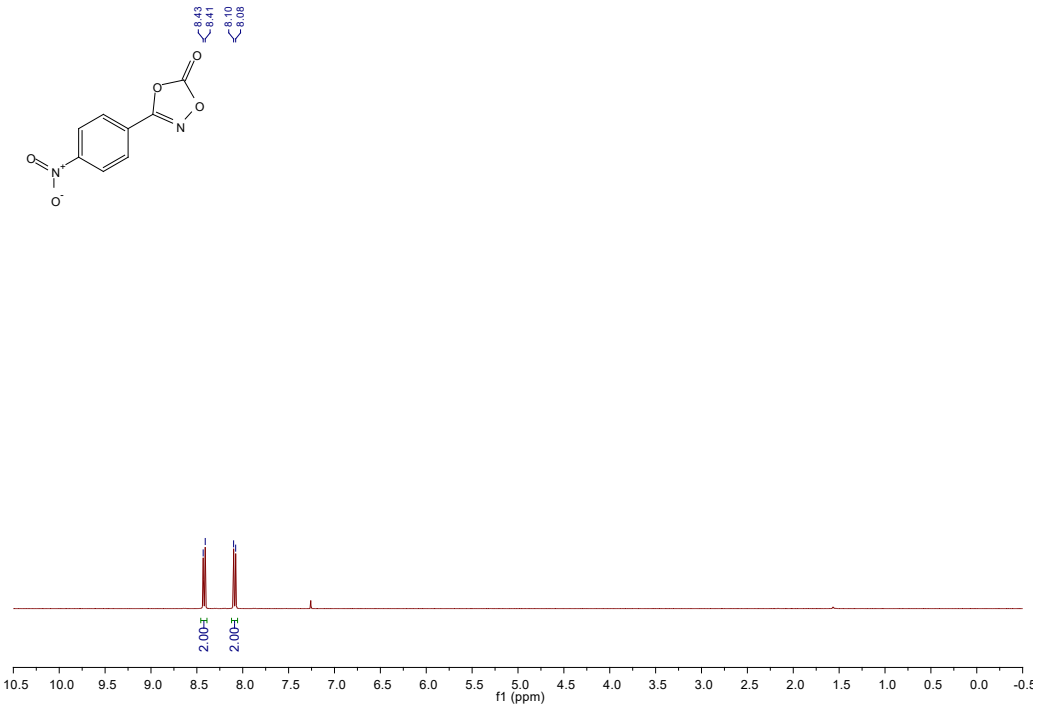
^{1}H-NMR (400 MHz, $CDCl_3$) of **1m**



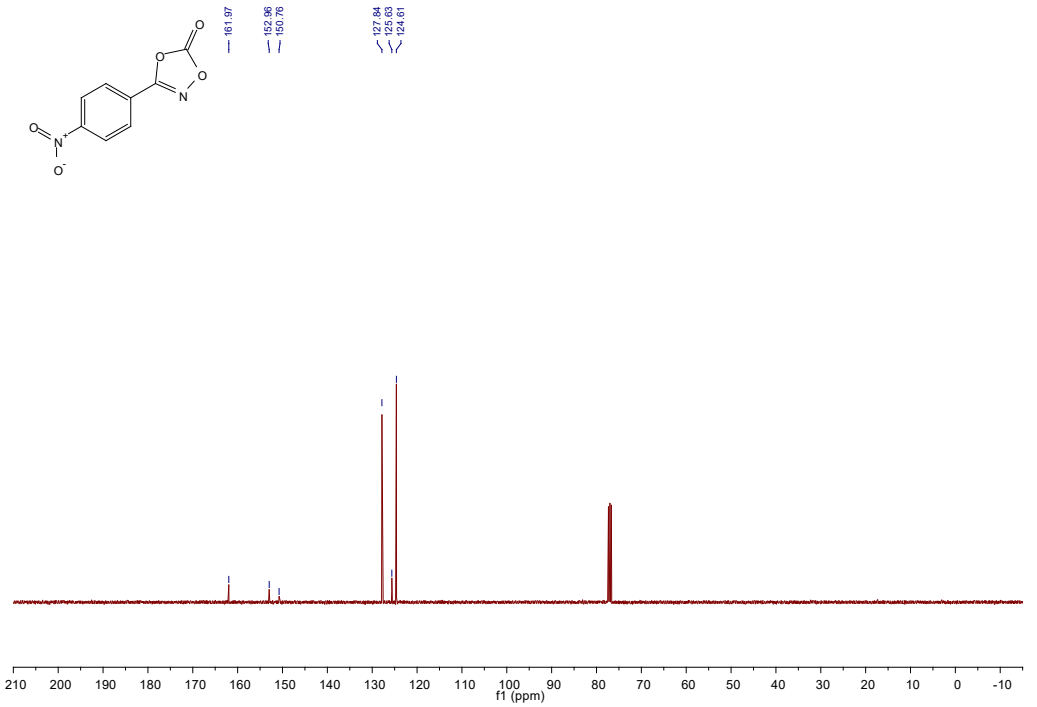
^{13}C-NMR (101 MHz, $CDCl_3$) of **1m**



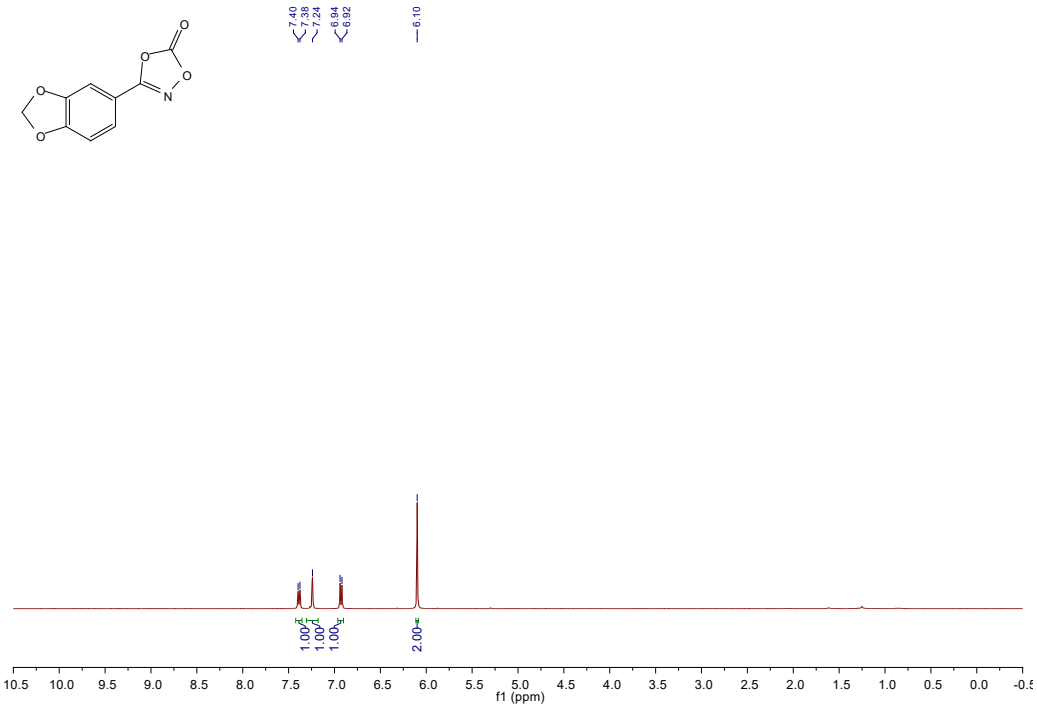
^{1}H-NMR (400 MHz, $CDCl_3$) of **1n**



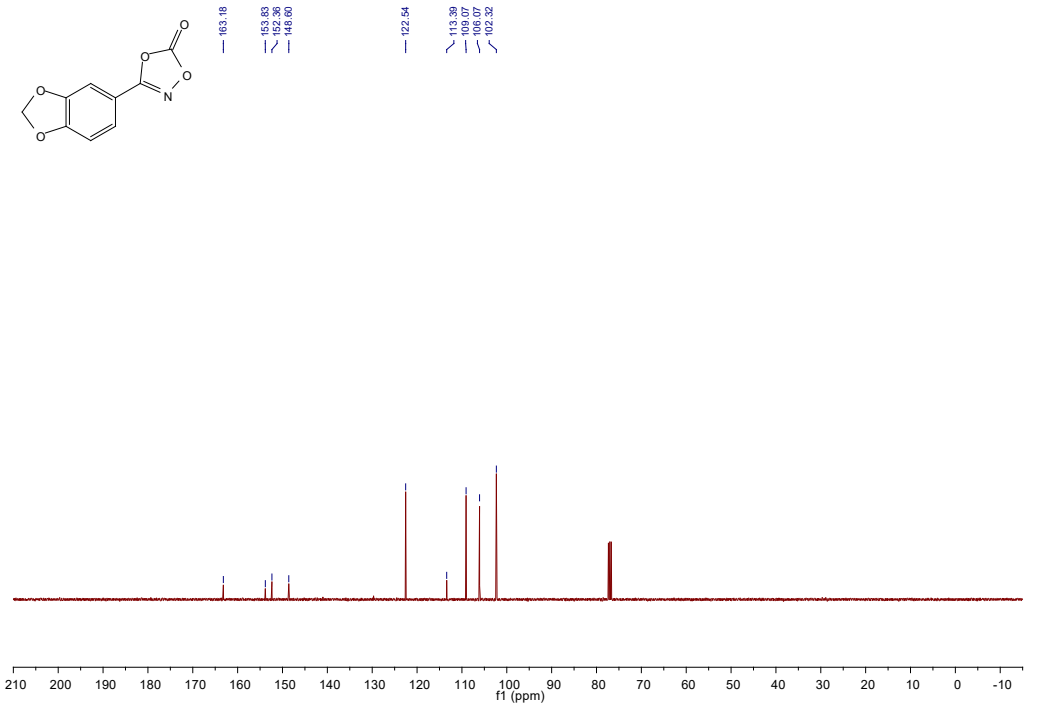
^{13}C-NMR (101 MHz, $CDCl_3$) of **1n**



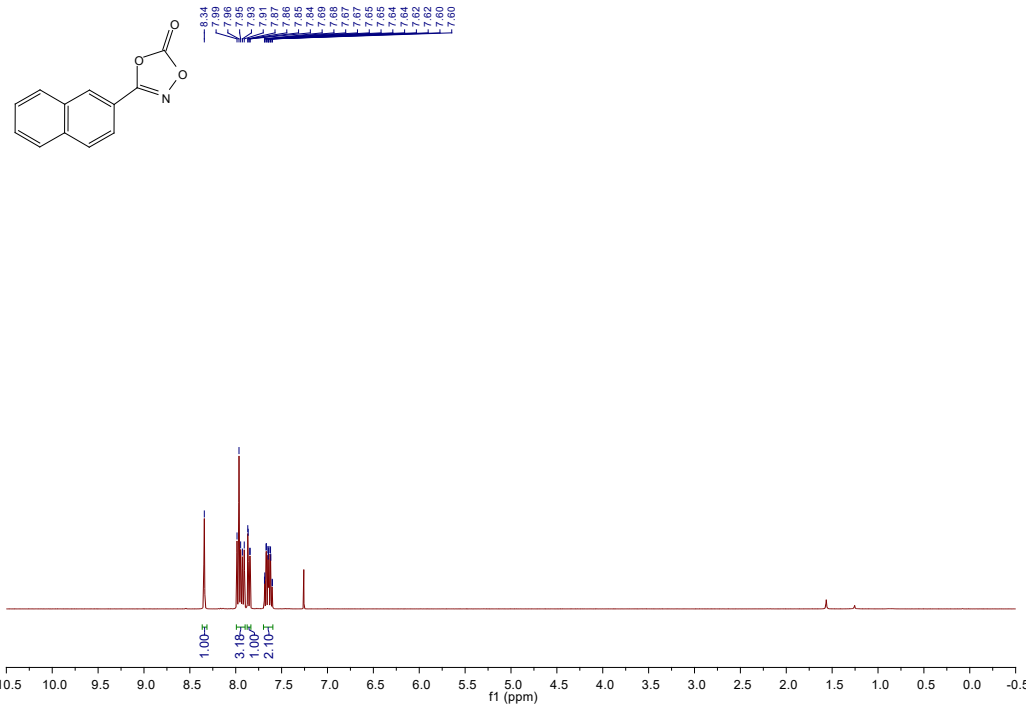
^{1}H-NMR (400 MHz, $CDCl_3$) of **1o**



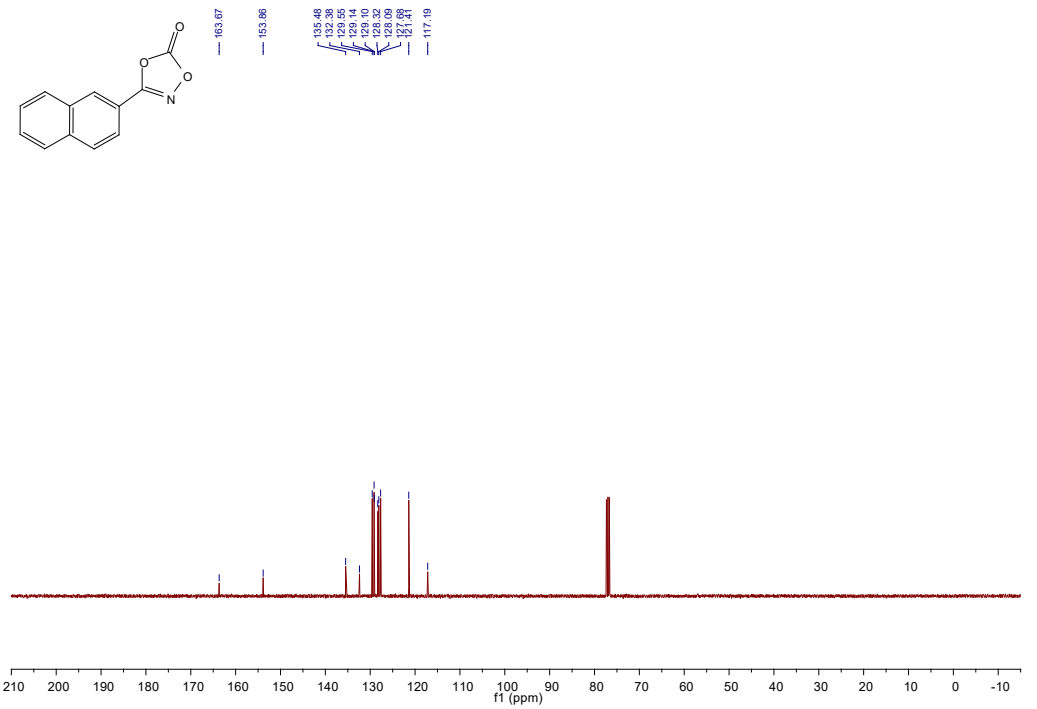
^{13}C-NMR (101 MHz, $CDCl_3$) of **1o**



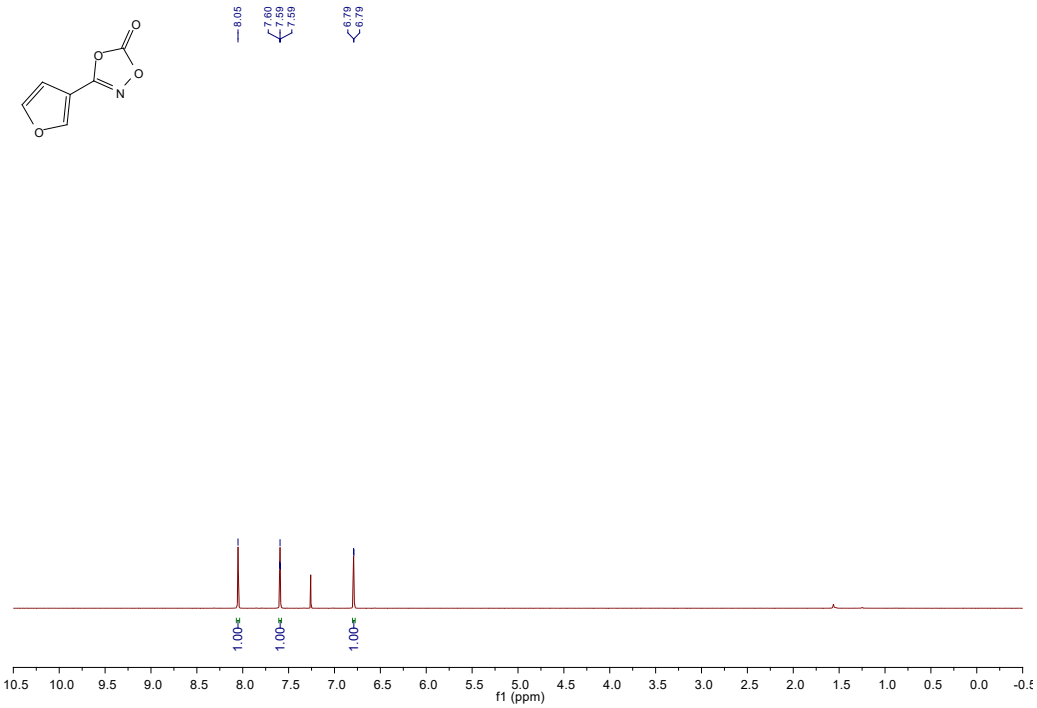
^{1}H-NMR (400 MHz, $CDCl_3$) of **1p**



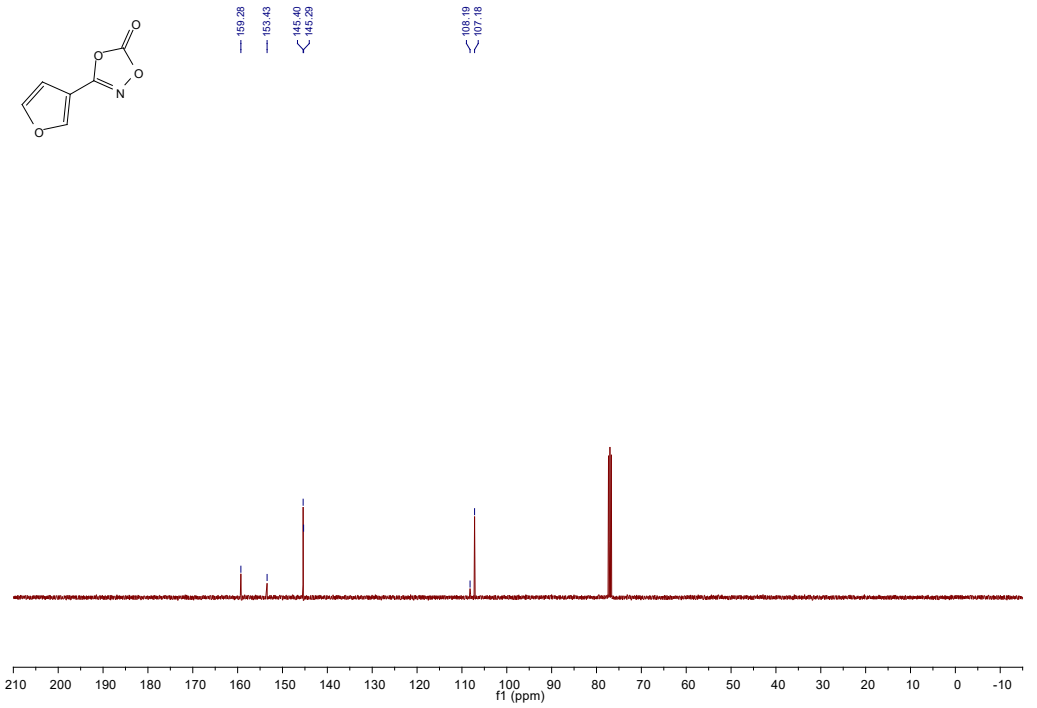
^{13}C-NMR (101 MHz, $CDCl_3$) of **1p**



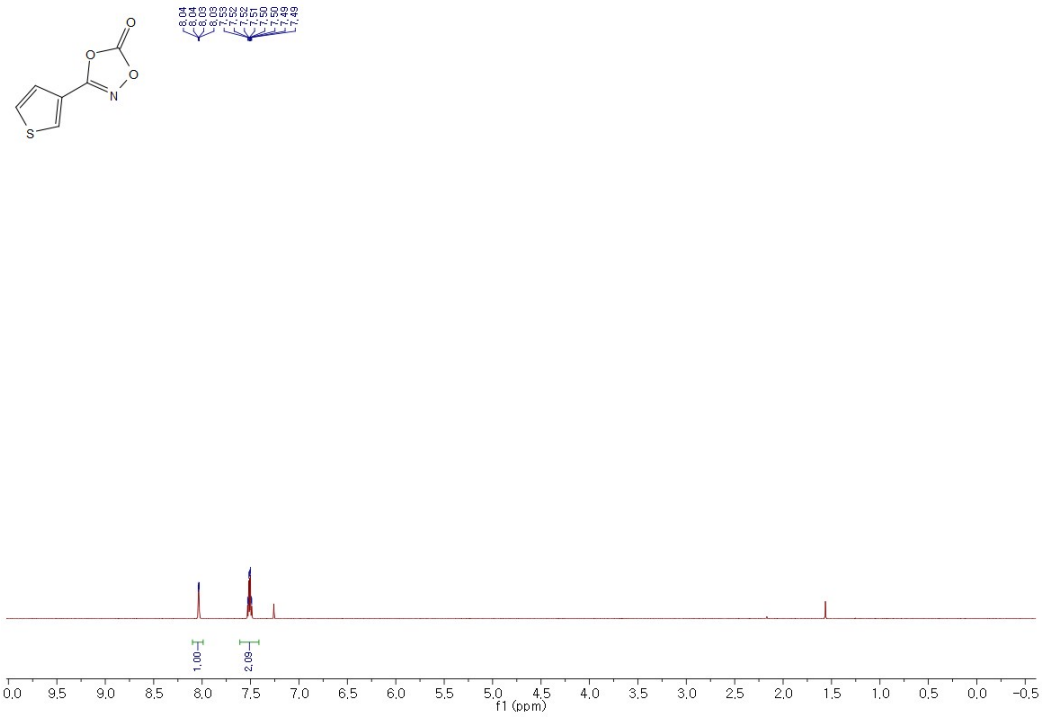
^{1}H-NMR (400 MHz, $CDCl_3$) of **1q**



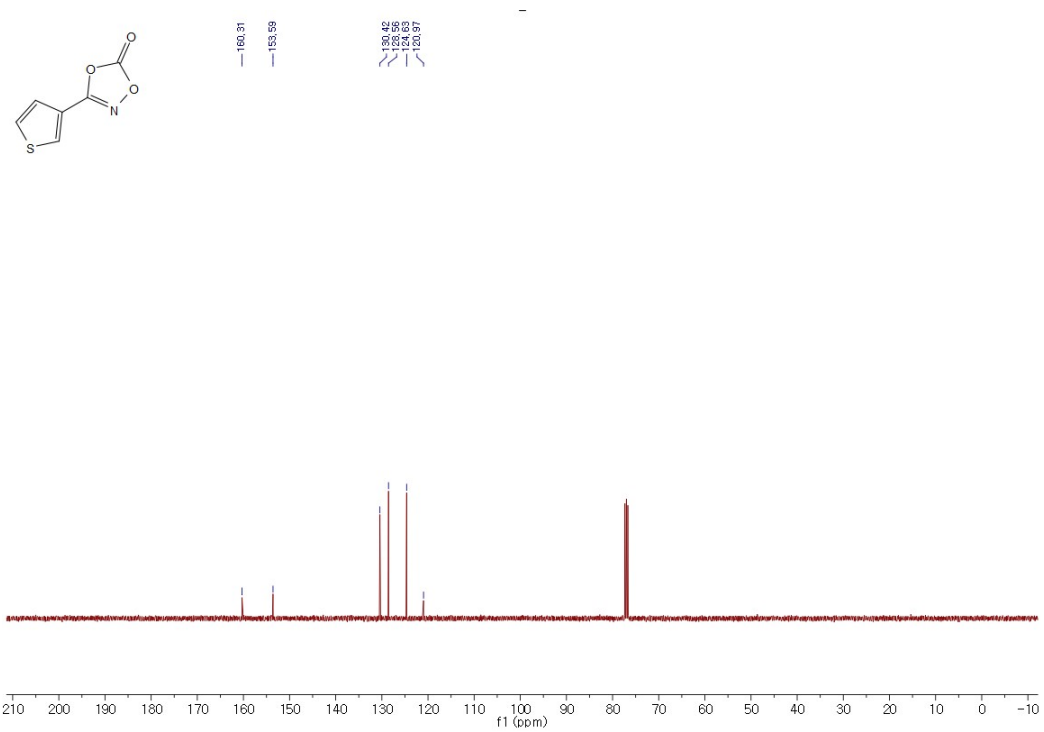
^{13}C-NMR (101 MHz, $CDCl_3$) of **1q**



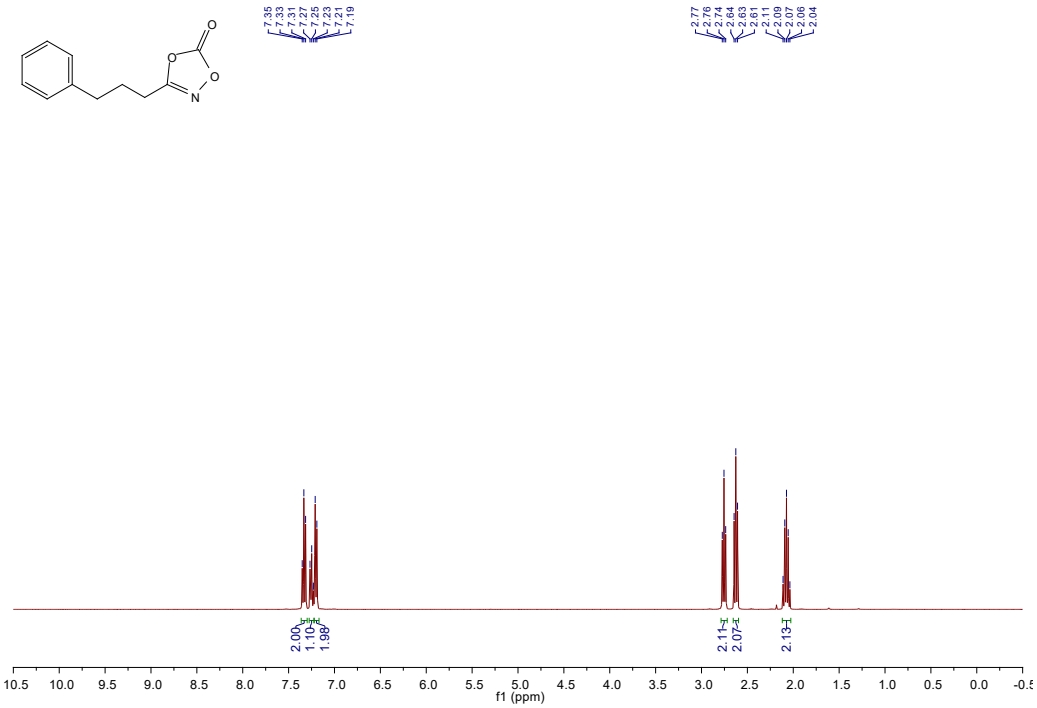
^{1}H-NMR (400 MHz, $CDCl_3$) of **1r**



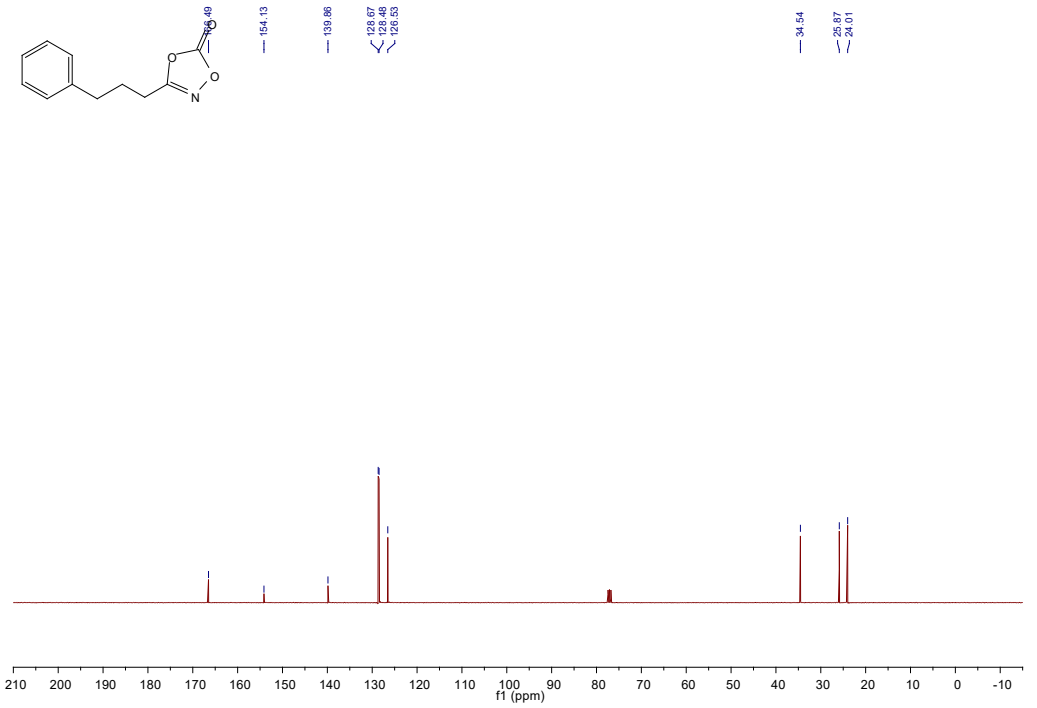
^{13}C-NMR (101 MHz, $CDCl_3$) of **1r**



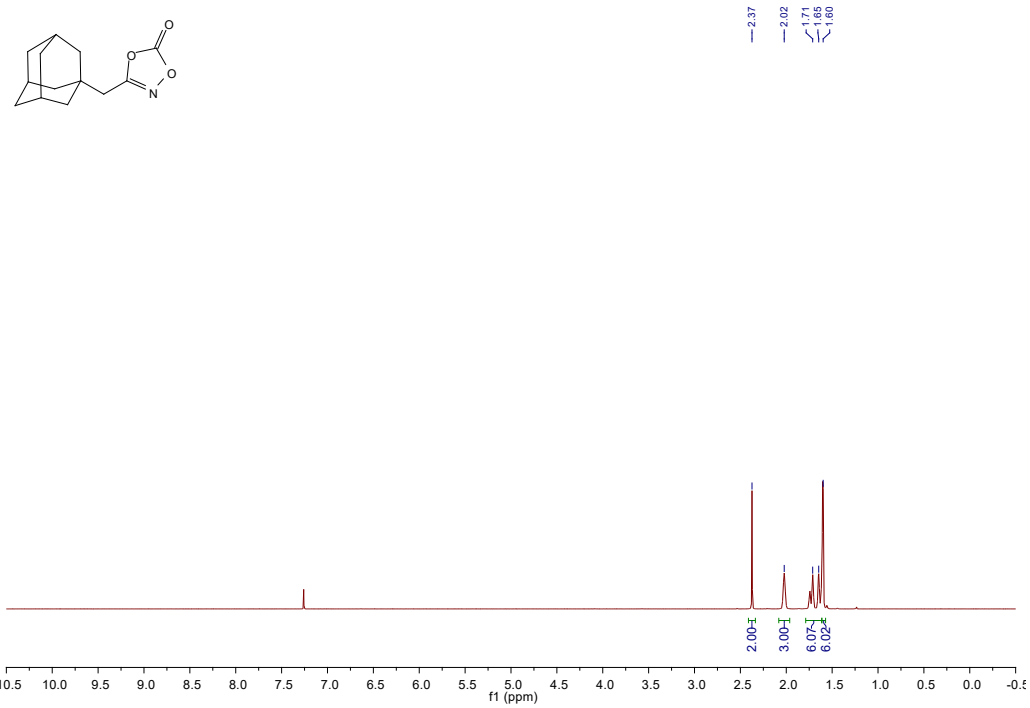
^{1}H-NMR (400 MHz, $CDCl_3$) of **1s**



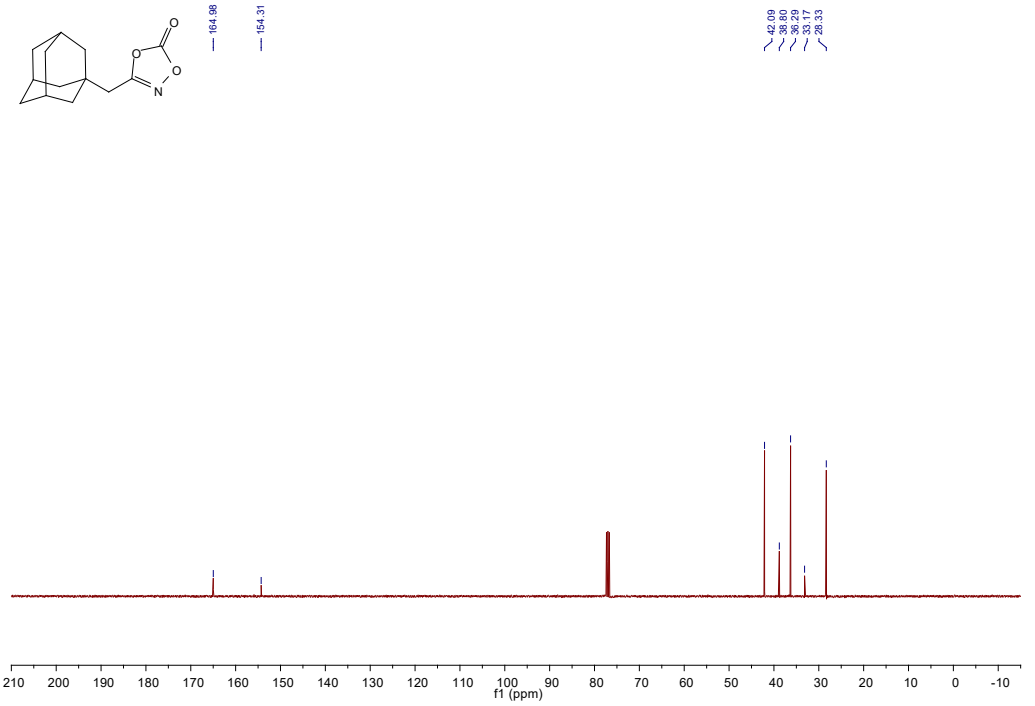
^{13}C-NMR (101 MHz, $CDCl_3$) of **1s**



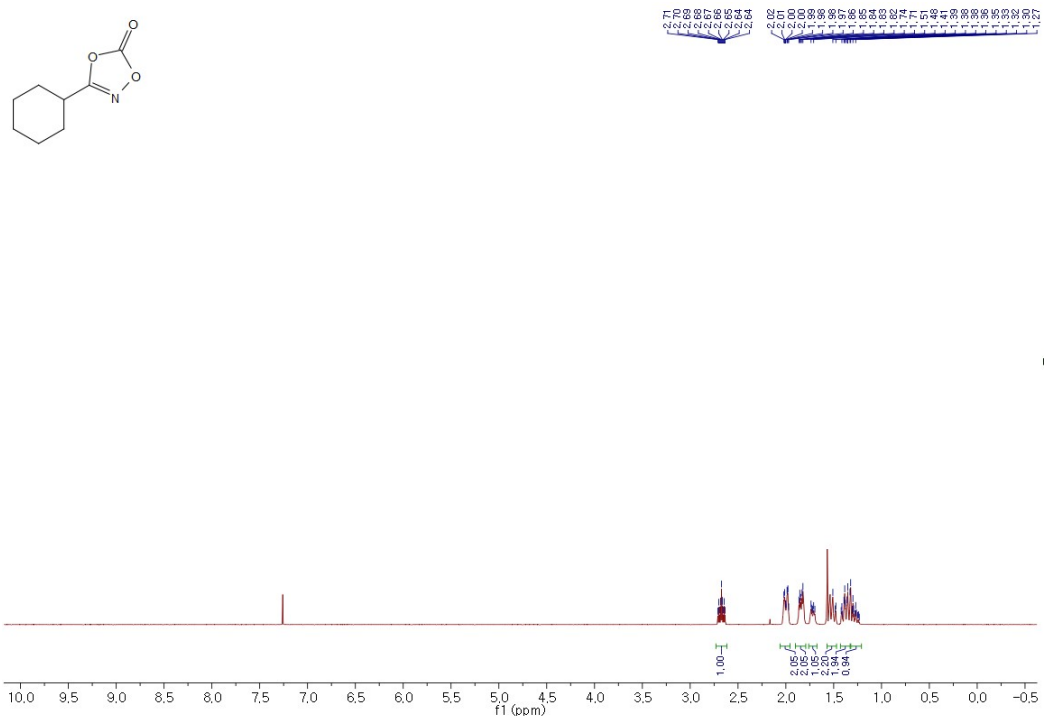
^{1}H-NMR (400 MHz, $CDCl_3$) of **1t**



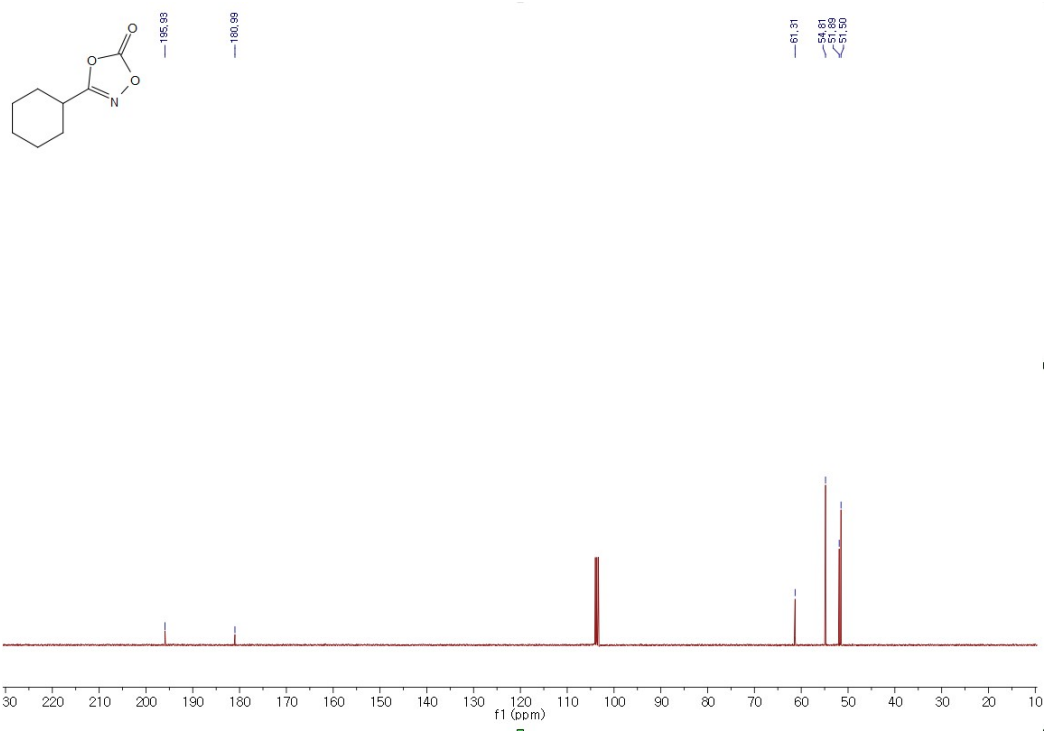
^{13}C-NMR (101 MHz, $CDCl_3$) of **1t**



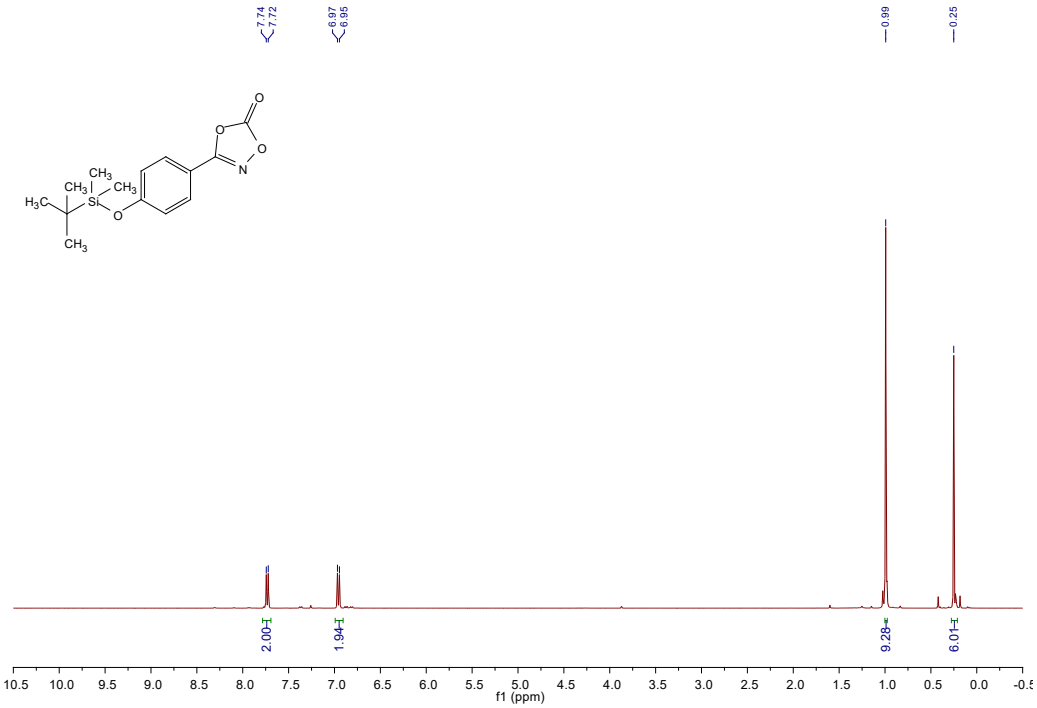
^{1}H-NMR (400 MHz, $CDCl_3$) of **1u**



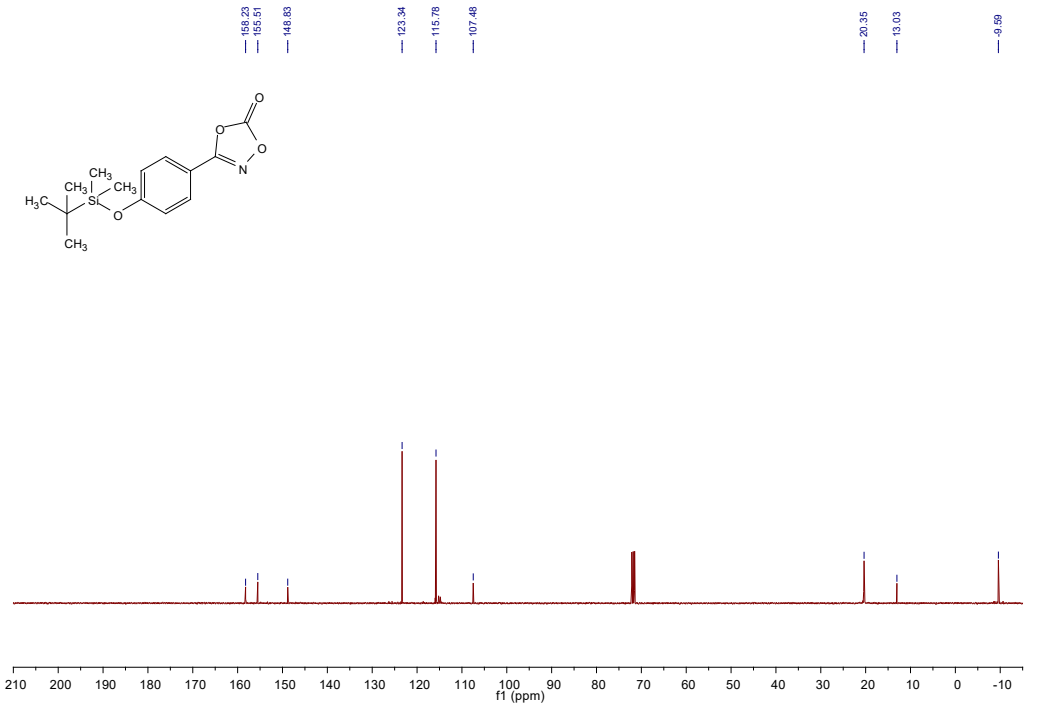
^{13}C-NMR (101 MHz, $CDCl_3$) of **1u**



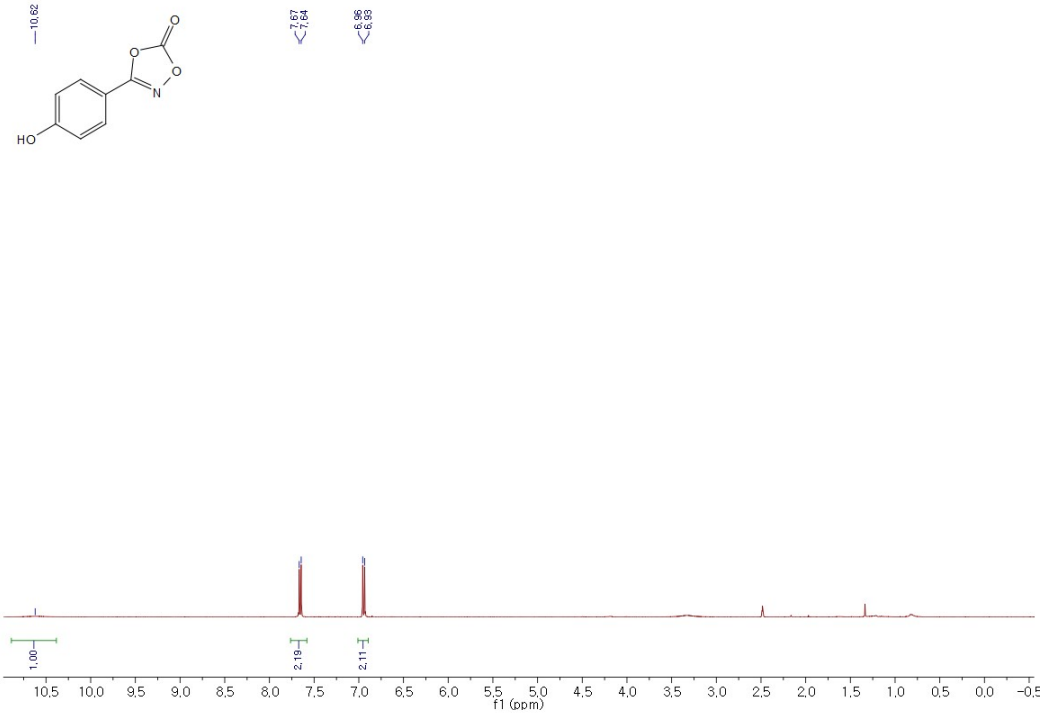
^{1}H-NMR (400 MHz, $CDCl_3$) of **1v**



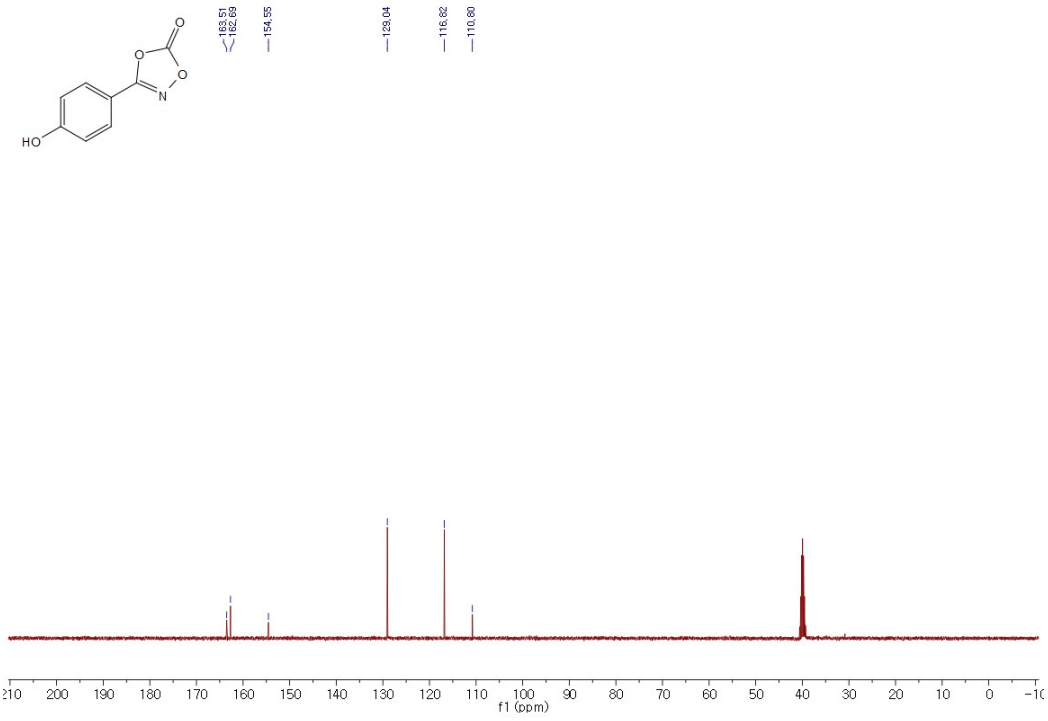
^{13}C-NMR (101 MHz, $CDCl_3$) of **1v**



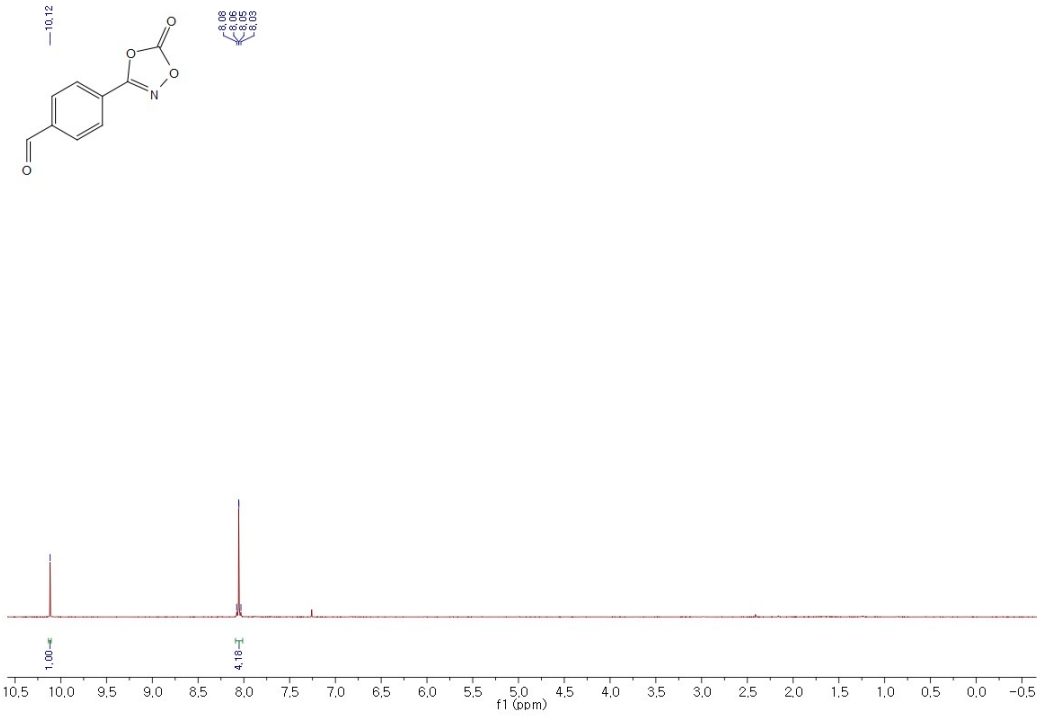
[1]H-NMR (400 MHz, DMSO-d_6) of **1w**



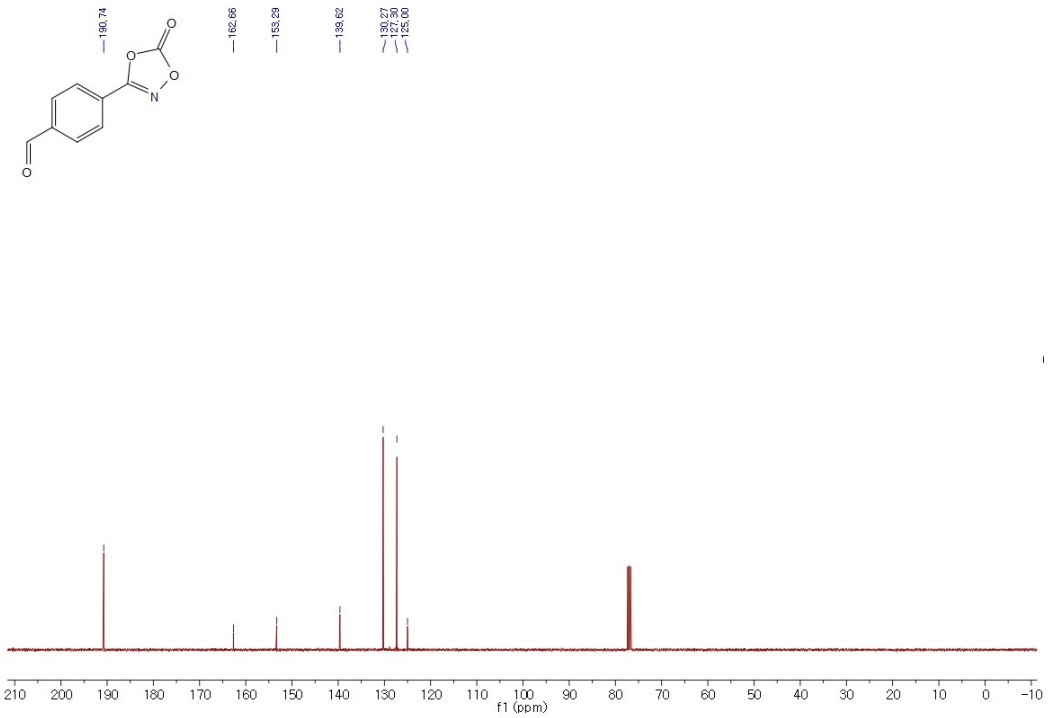
[13]C-NMR (101 MHz, DMSO-d_6) of **1w**



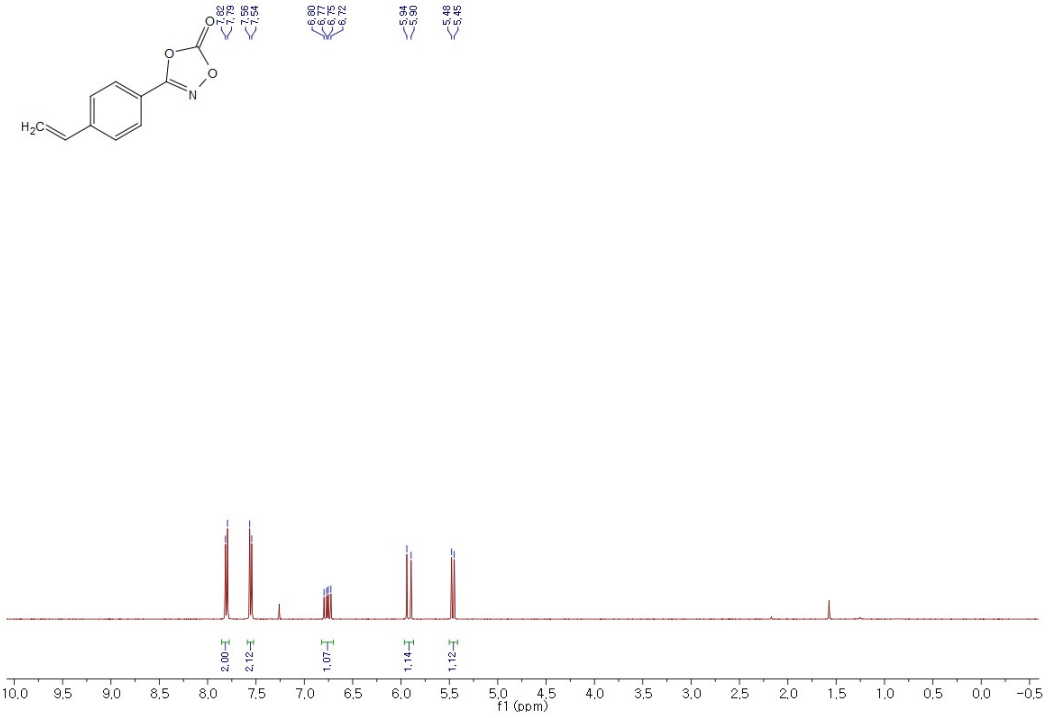
^{1}H-NMR (400 MHz, $CDCl_3$) of **1x**



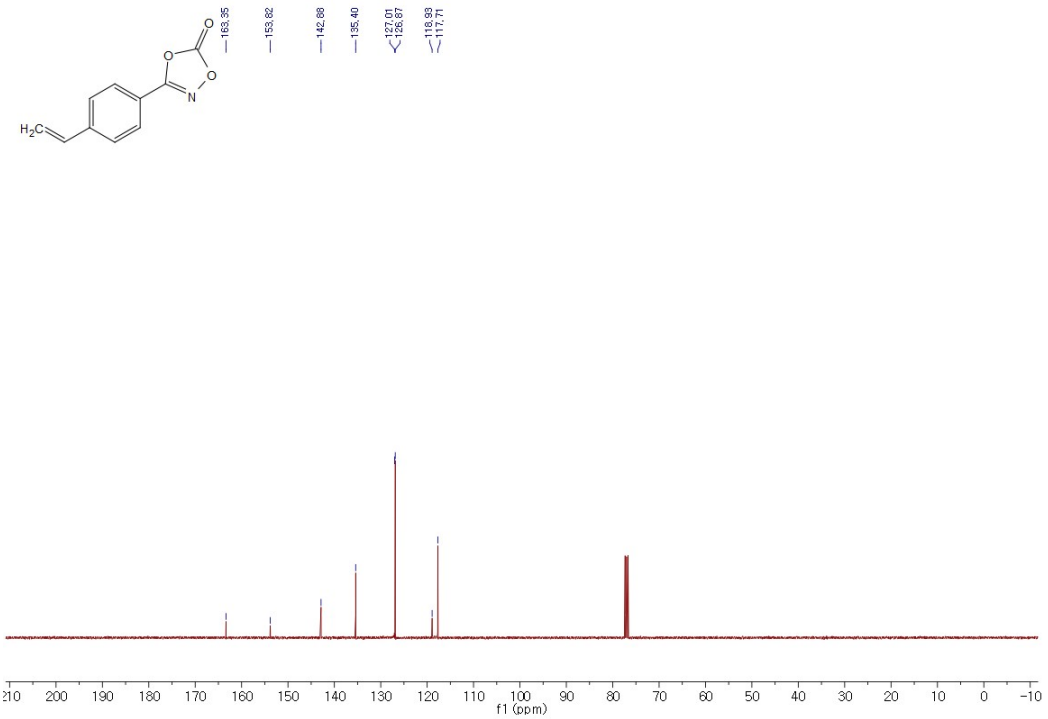
^{13}C-NMR (101 MHz, $CDCl_3$) of **1x**



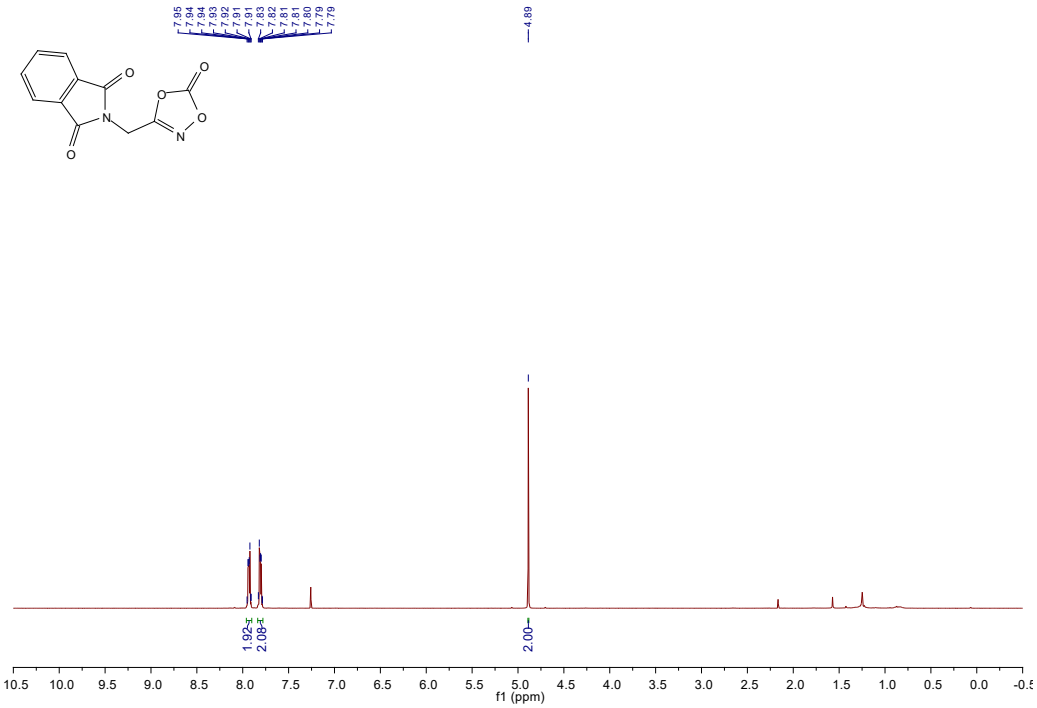
^{1}H-NMR (400 MHz, $CDCl_3$) of **1y**



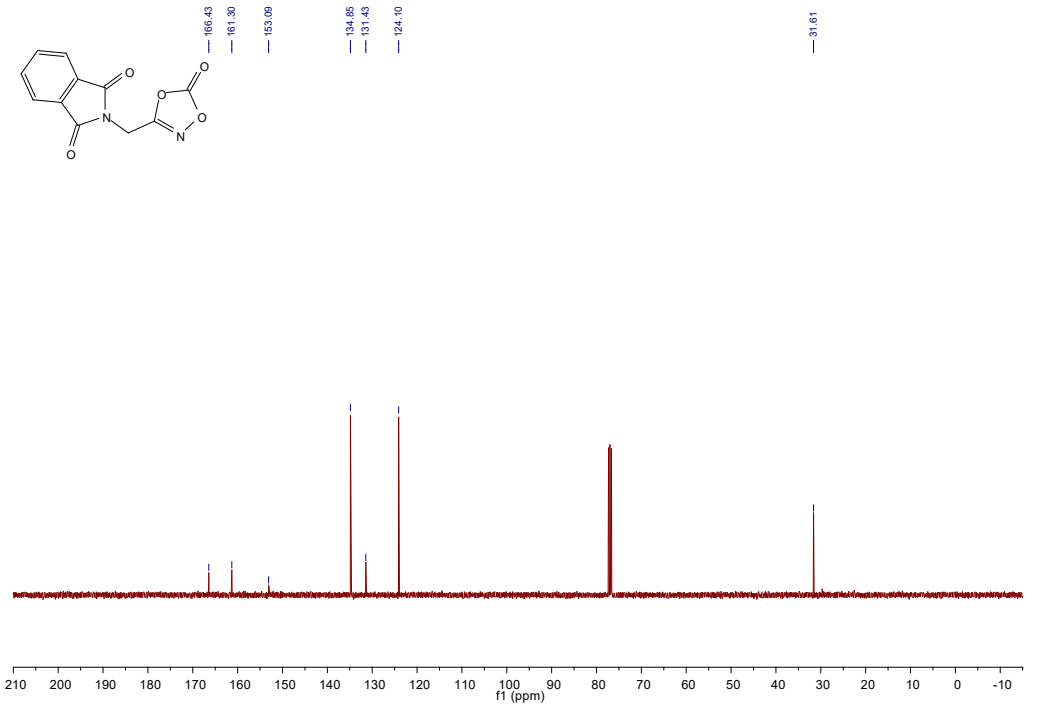
^{13}C-NMR (101 MHz, $CDCl_3$) of **1y**



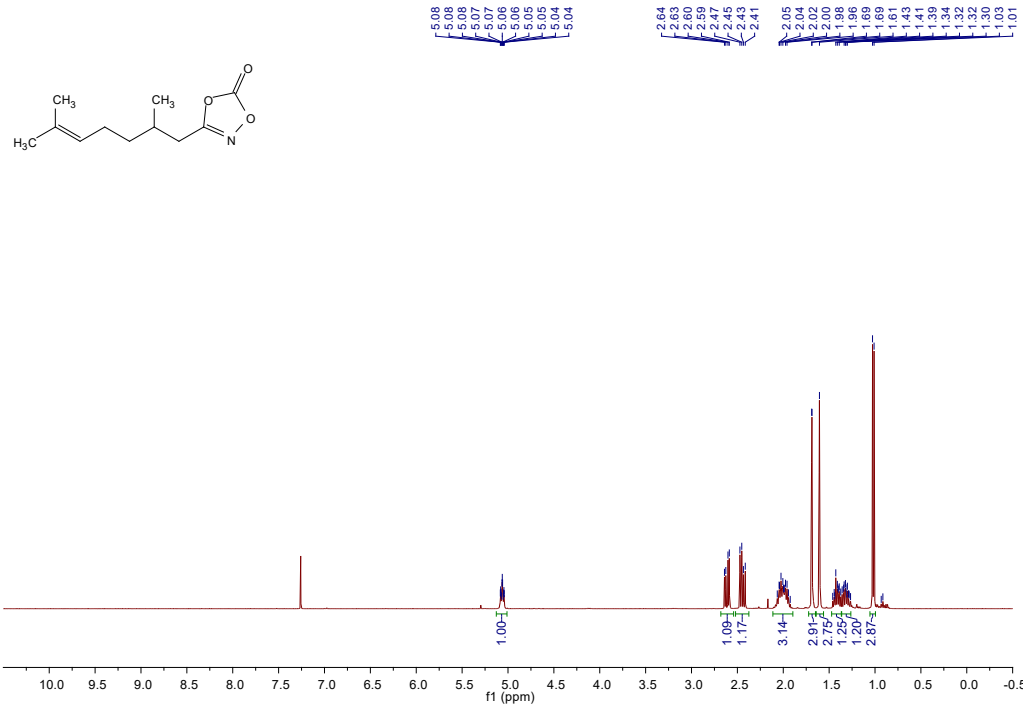
^{1}H-NMR (400 MHz, $CDCl_3$) of **3a**



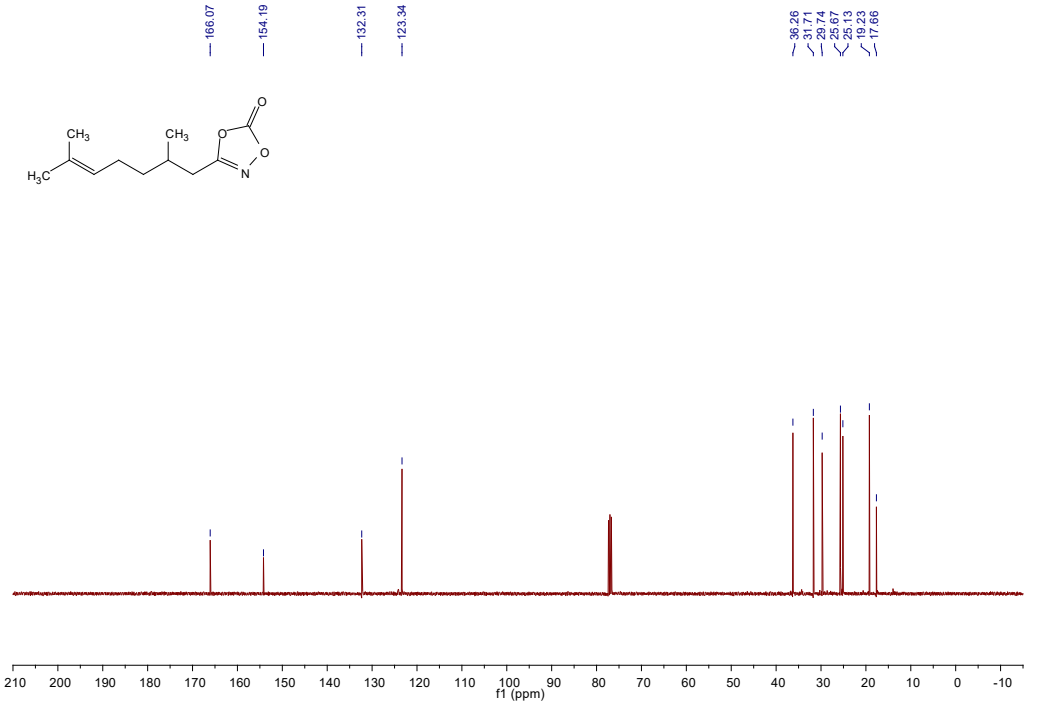
^{13}C-NMR (101 MHz, $CDCl_3$) of **3a**



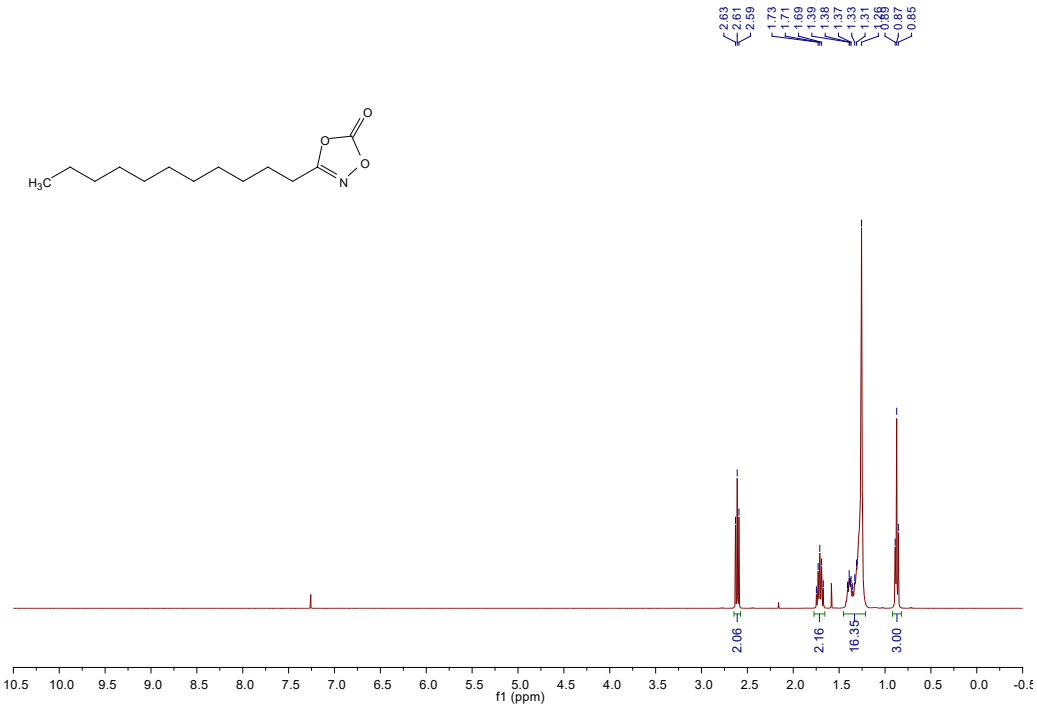
^{1}H-NMR (400 MHz, $CDCl_3$) of **3b**



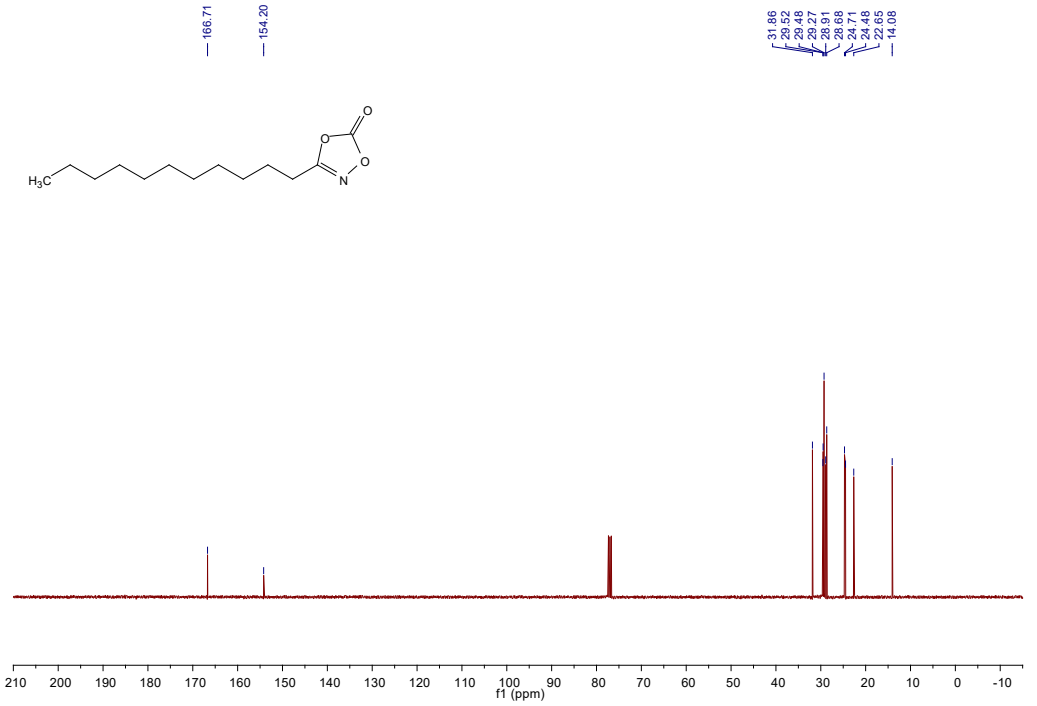
^{13}C-NMR (101 MHz, $CDCl_3$) of **3b**



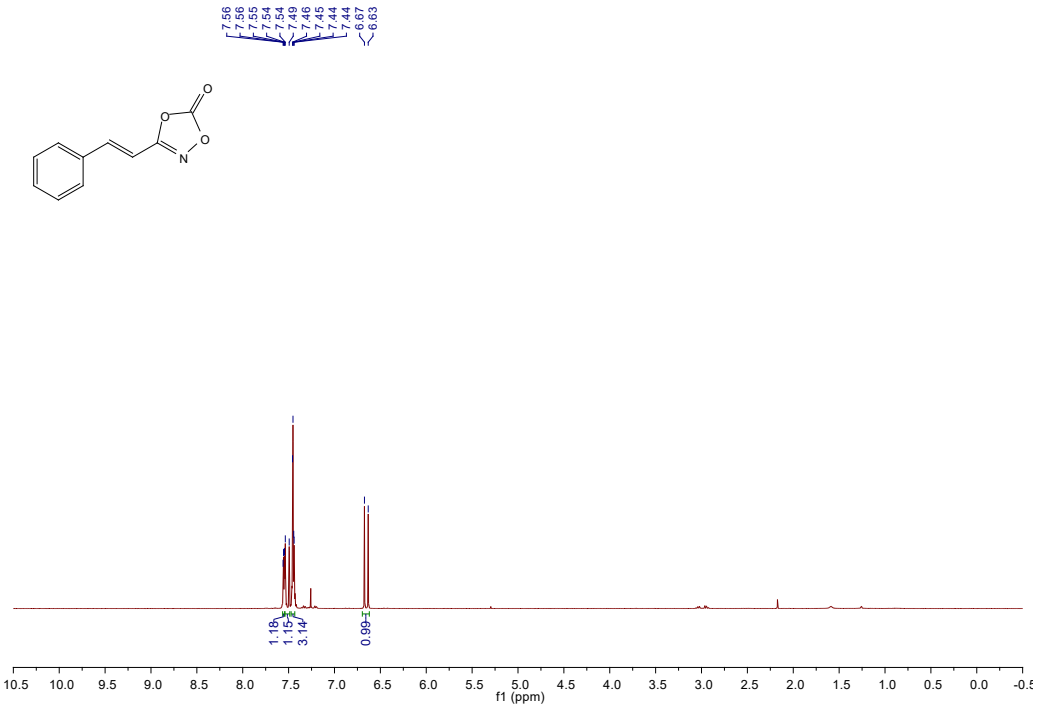
^{1}H-NMR (400 MHz, $CDCl_3$) of **3c**



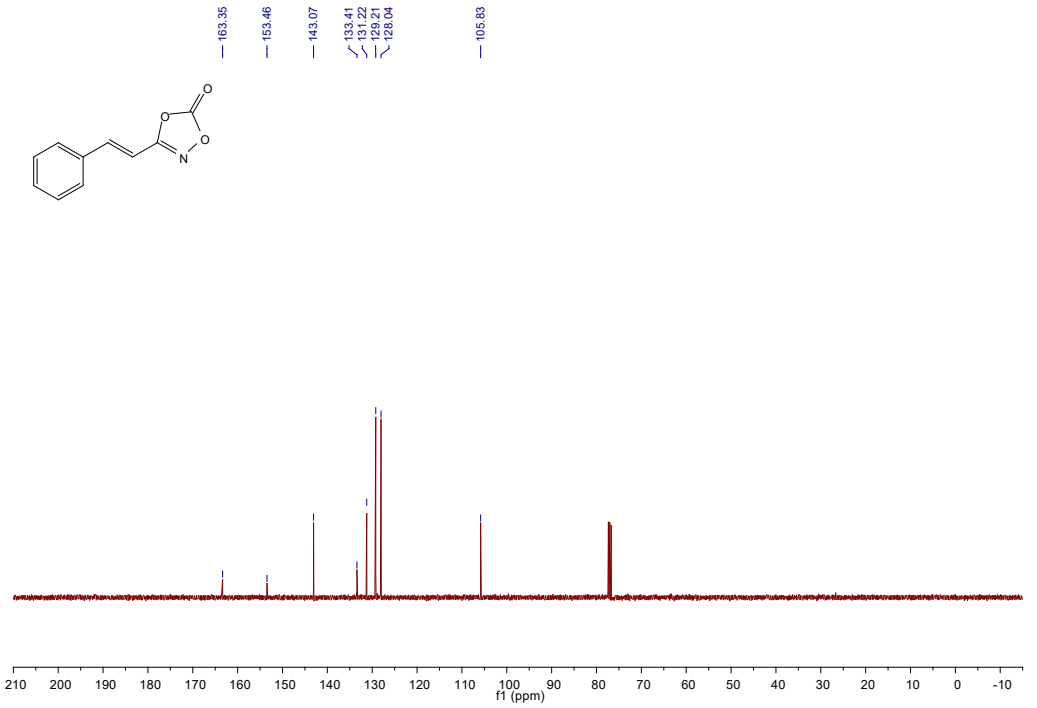
^{13}C-NMR (101 MHz, $CDCl_3$) of **3c**



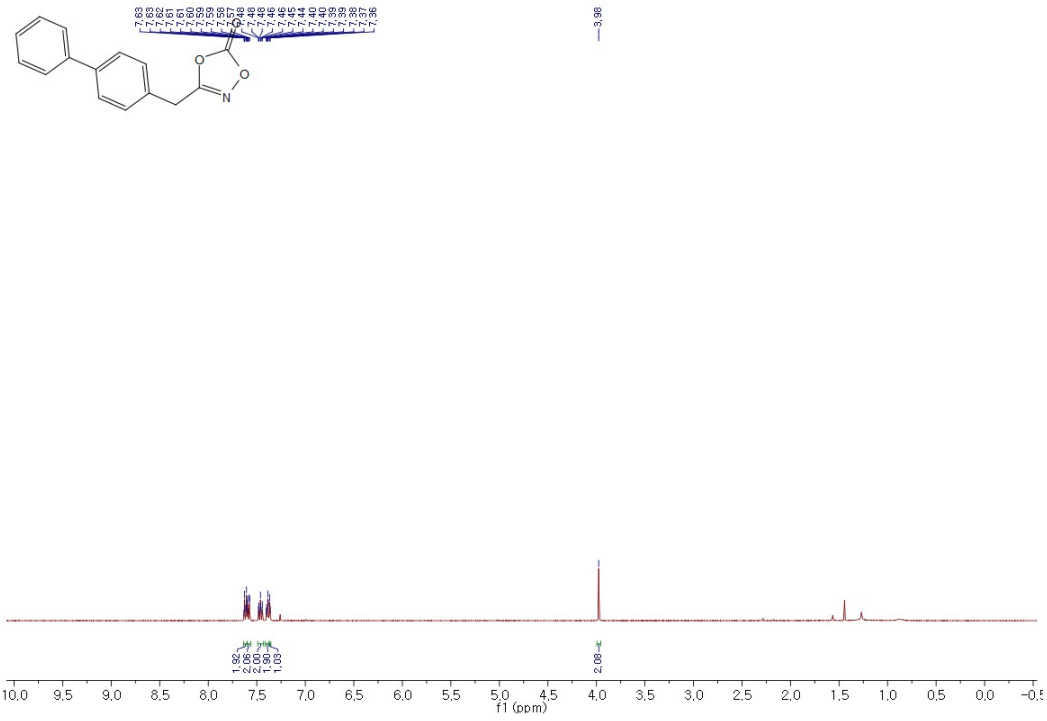
^{1}H-NMR (400 MHz, $CDCl_3$) of **3d**



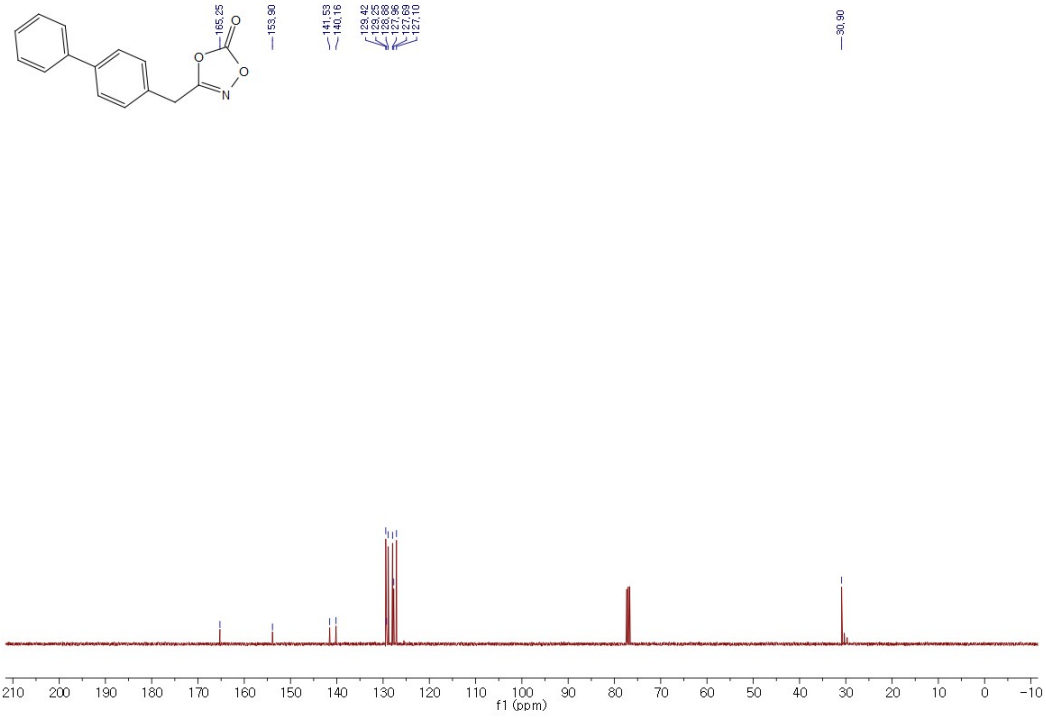
^{13}C-NMR (101 MHz, $CDCl_3$) of **3d**



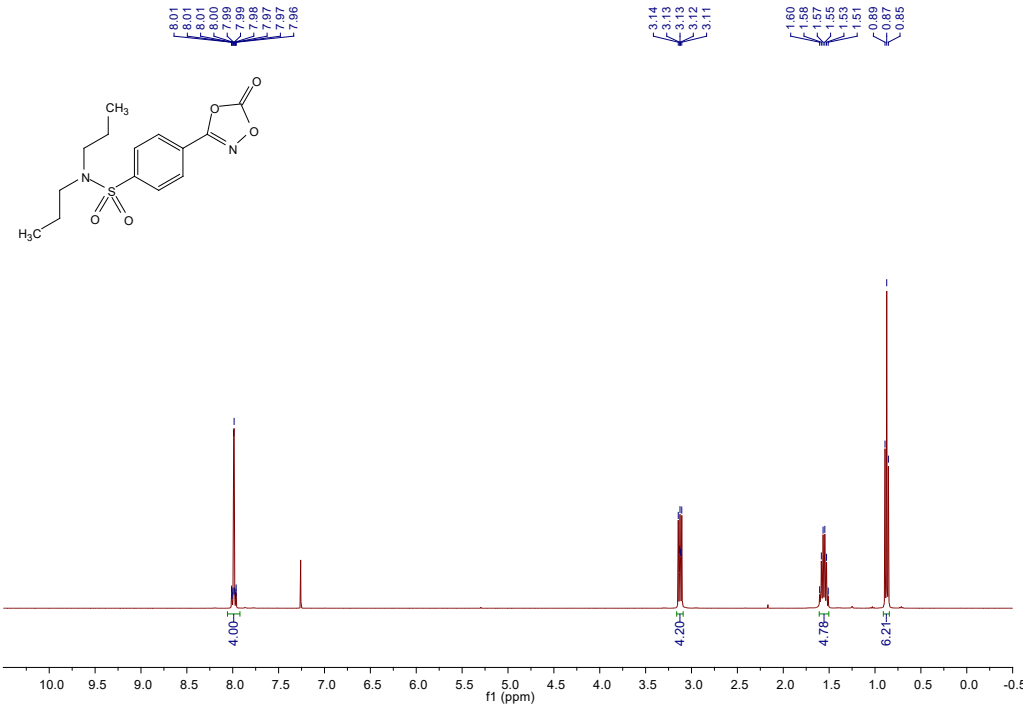
^{1}H-NMR (400 MHz, $CDCl_3$) of **3e**



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



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



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