Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Metal-Free Photoinduced Denitrogenative Alkylation of Vinyl Azides with Alkyl Radicals toward Ketones

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1. Supplementary Methods

All new compounds were fully characterized. NMR spectra were recorded on Bruker Avance II 400, Vaian DLG400, Bruker Avance NEO 600M NMR Spectroscopy and calibrated using residual undeuterated solvent ($CDCl_3$ = 7.26 ppm ^{1}H NMR, 77.00 ppm ^{13}C NMR; DMSO-d6 = 2.50 ppm ^{1}H NMR, 39.52 ppm ^{13}C NMR). Mass spectra were conducted at Thermo LTQ Orbitrap XL (ESI). Anhydrous solvents, such as Dichloromethane (DCM), *N*, *N*-Dimethylacetamide (DMA), *N*, *N*-Dimethylformamide (DMF), Dimethyl sulfoxide (DMSO), Acetonitrile (MeCN), Tetrahydrofuran (THF), Ethyl acetate (EA), were purchased from Adamas. Flash column chromatography was carried out using silica gel (General-Reagent, AR, 200-300 mesh, for column chromatography). All reactions were carried out in dried 8 mL vial under Nitrogen. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification.

Photoreaction setup: All photoredox reactions were subjected to irradiation from double 10W Kessil PR160L blue LED bulbs (456 nm), with the reaction tube placed approximately ~ 4.5 cm from the bulbs and using a fan to keep at room temperature (Figure S1).

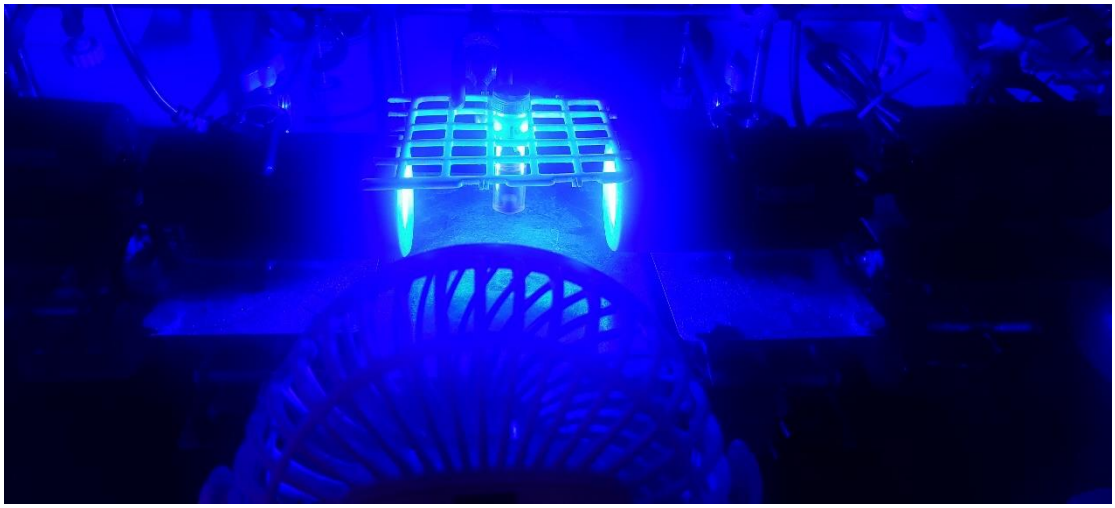
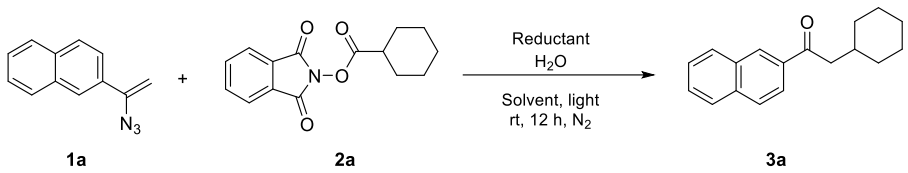


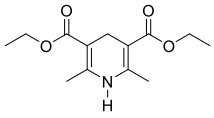
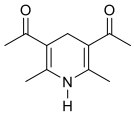
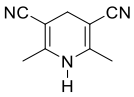
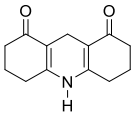
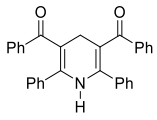
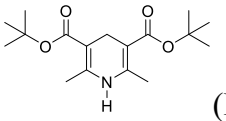
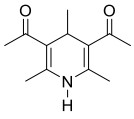
Figure S1. Photoreaction setup

2. Optimization of reaction conditions



1a + **2a** → **3a**

Table S1. Exploration of Reductant[a]

Entry	Reductant	Yield of **3a** (%)
1	 (**ED 1**)	56
2	 (**ED 2**)	79
3	 (**ED 3**)	33
4	 (**ED 4**)	32
5	 (**ED 5**)	71
6	 (**ED 6**)	52
7	 (**ED 7**)	Trace

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), reductant (0.15 mmol, 1.5 eq), H_2O (0.2 mmol, 2 eq), DMSO (1 mL), Kessil PR160L (456 nm, 100% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S2. Exploration of solvent (1 mL)[a]

Entry	Solvent	Yield of **3a** (%)
1	DMSO	79
2	DMA	77
3	DMF	72
4	Acetone	55
5	CH_3CN	40
6	EA	18
7	DCM	9

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O (0.2 mmol, 2 eq), solvent (1 mL), Kessil PR160L (456 nm, 100 % light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S3. Optimization of the ratio between **2a** and **ED 2** [a]

Entry	**2a**	**ED 2**	Yield of **3a** (%)
1	1.5	1.5	79
2	1.2	1.2	70
3	1.5	1.2	56
4	1.5	2	78
5	2	2	78

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a**, 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**), H_2O (0.2 mmol, 2 eq), DMSO (1 mL), Kessil PR160L (456 nm, 100% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S4. Exploration of wavelength λ (nm)[a]

Entry	Light (nm)	Yield of **3a** (%)
1	456 nm	79
2	440 nm	69
3	427 nm	67

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O (0.2 mmol, 2 eq), DMSO (1 mL), Kessil PR160L (100% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S5. Exploration of light intensity[a]

Entry	Intensity (%)	Yield of **3a** (%)
1	25	84
2	50	83
3	75	80
4	100	79

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O (0.2 mmol, 2 eq), DMSO (1 mL), Kessil PR160L (456 nm), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S6. Exploration of H_2O equivalent[a]

Entry	H_2O equivalent (eq)	Yield of **3a** (%)
1	1.2	80
2	1.5	85
3	2.0	84
4	2.5	83
5	3	80

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O, DMSO (1 mL), Kessil PR160L (456 nm, 25% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S7. Optimization of reaction concentration[a]

Entry	DMSO (mL)	Yield of **3a** (%)
1	0.5	62
2	0.75	83
3	1	85
4	1.5	78
5	2	71

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O (0.15 mmol, 1.5 eq), DMSO, Kessil PR160L (456 nm, 25% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

Table S8. Optimization of reaction time[a]

Entry	Time (h)	Yeild of **3a** (%)
1	2	72
2	4	74
3	6	76
4	8	80
5	12	85
6	18	76
7	24	75

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **2a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.15 mmol, 1.5 eq), H_2O (0.15 mmol, 1.5 eq), DMSO (1 mL), Kessil PR160L (456 nm, 25% light intensity), rt, time, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

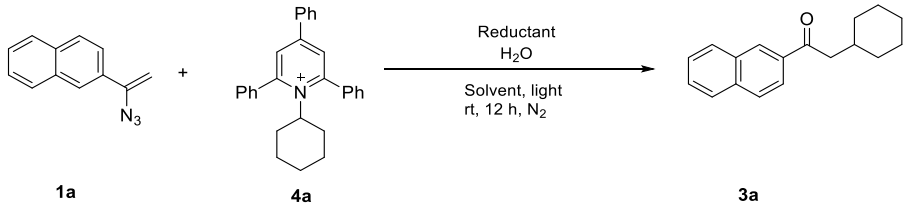
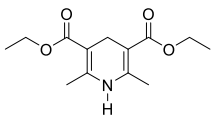
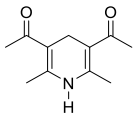
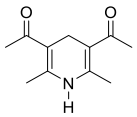
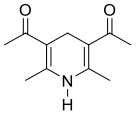
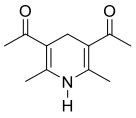


Table S9. Exploration of Reductant[a]

Entry	Reductant	Yield of **3a** (%)
1	Et_3N	6
2	DIPEA	5
3	 (**ED 1**)	11
4	 (**ED 2**)	39
5[b]	 (**ED 2**)	36

Entry	Reductant	Yield of **3a** (%)
6[c]	 (**ED 2**)	39
7[d]	 (**ED 2**)	21

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **4a** (0.15 mmol, 1.5 eq), reductant (0.2 mmol, 2 eq), H_2O (0.15 mmol, 1.5 eq), DMSO (1 mL), Kessil PR160L (456 nm,100% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard. [b] 25% light intensity was used. [c]**ED 2** (0.15 mmol) was used. [d]**ED 2** (0.25 mmol) was used.

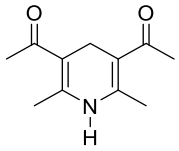
Table S10. Exploration of solvent (1 mL)[a]

Entry	Solvent	Yield of **3a** (%)
1	DMA	54
2	DMF	37
3	MeOH	40
4	DMSO	39
5	THF	38
6	Acetone	32
7	CH_3CN	26
8	EA	20
9	DCM	20

[a]Reaction conditions: **1a** (0.1 mmol, 1 eq), **4a** (0.15 mmol, 1.5 eq), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.2 mmol, 1.5 eq), solvent (1 mL), Kessil PR160L (456 nm, 100% light intensity), rt, 12 h, N_2 atmosphere. Yields were determined by ^1H NMR with CH_2Br_2 (7 μL, 0.1 mmol) as an internal standard.

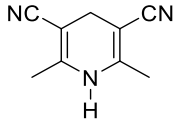
3. Preparation of Reductant

3,5-Diacetyl-1,5-dihydro-2,6-dimethylpyridine (ED 2) [1]



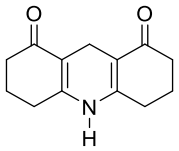
In 25 mL Schlenk flask, a mixture of paraformaldehyde (0.30 g, 1.0 equiv), acetylacetone (2.0 mL, 2.0 equiv) and NH_4OAc (1.16 g, 1.50 equiv) in ethanol (8 mL). The reaction mixture was stirred at 80 °C for 2 h under argon atmosphere. The precipitate was filtered and further recrystallized from methanol to afford **ED 2** as yellow solid (1.54 g, 80% yield): **^{1}H NMR (400 MHz, DMSO-d6)** δ 8.27 (s, 1H), 3.26 (s, 2H), 2.13 (s, 6H), 2.10 (s, 6H). All data are in accordance with the literature.[1]

2,6-dimethyl-1,4-dihydropyridine-3,5-dicarbonitrile (ED 3) [2]



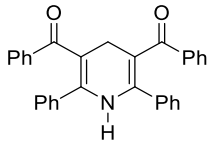
In 25 mL Schlenk flask, β-aminocrotononitrile (1.0 g, 1.7 equiv) and hexamethylenetetramine (1.0 g, 1.0 equiv) were dissolved in glacial acetic acid (7.5 mL). The temperature of the solution rose spontaneously to 75 °C. After the solution had cooled to room temperature, scratching produced a yellow precipitate. After standing overnight, the precipitate was filtered off and washed with ether. The solid was recrystallized from methanol to afford **ED 3** as yellow solid (0.4 g, 35% yield): **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 9.08 (s, 1H), 3.13 (s, 2H), 1.92 (s, 6H). **^{13}C NMR (101 MHz, DMSO-*d*$_6$)** δ 148.40, 119.63, 76.96, 24.65, 17.64. **HRMS m/z (ESI)** calculated for $C_9H_9N_3$ $(M+H)^+$ 160.0869, found .160.0871.

3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H) dione (ED 4) [3]



In a round bottom flask, 1,3-cyclohexanedione (2.24 g, 2 equiv), aldehyde (0.30 g, 1 equiv) and NH_4OAc (1.16 g, 1.5 equiv) were dissolved in water (50 mL). The mixture was stirred for 6 h in reflux condition. The precipitate was filtered and further recrystallized from methanol to afford **ED 4** as yellow solid (1.26 g, 58% yield): **^{1}H NMR (600 MHz, DMSO-d6)** δ 8.96 (s, 1H), 2.81 (s, 2H), 2.35 (t, J = 6.2 Hz, 4H), 2.23 (t, J = 6.5 Hz, 4H), 1.87 (p, J = 6.2 Hz, 4H). **^{13}C NMR (101 MHz, DMSO-d6)** δ 195.19, 152.03, 108.42, 36.35, 26.12, 20.89, 18.65. **HRMS m/z (ESI)** calculated for $C_{13}H_{15}NO_2$ $(M+H)^+$ 218.1176, found 218.1172.

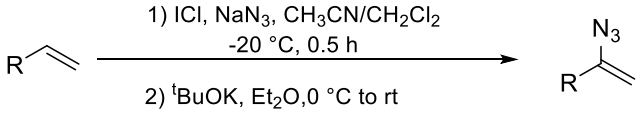
(2,6-diphenyl-1,4-dihydropyridine-3,5-diyl) bis(phenylmethanone) (ED 5) [4]



In 25 mL Schlenk flask, a mixture of 1,3-diphenylpropanedione (0.89 g, 2.0 equiv), methenamine (0.08 g, 0.15 equiv) and NH_4OAc (0.15 g, 1.0 equiv) in ethanol (2 mL). The reaction mixture was stirred at 80 °C for 2-5 h under argon atmosphere. The precipitate was filtered and further recrystallized from methanol to afford **ED 5** as yellow solid (0.31 g, 35% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 – 7.54 (m, 4H), 7.29 – 7.25 (m, 4H), 7.23 – 7.18 (m, 2H), 7.17 – 7.08 (m, 10H), 6.03 (s, 1H), 3.82 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 196.59, 145.78, 139.35, 135.37, 131.17, 129.68, 129.04, 128.84, 128.49, 127.70, 108.77, 29.29. **HRMS m/z (ESI)** calculated for $C_{31}H_{23}NO_2$ $(M+H)^+$ 442.1802, found 442.1800.

4. Preparation of vinyl azides

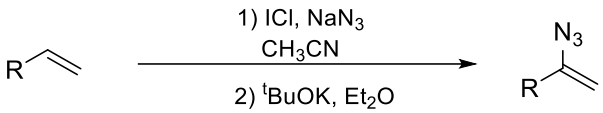
Method A:[5]



In 100mL pressure bottle, NaN_3 (12.5 mmol, 2.5 equiv) in CH_3CN (4 mL) was added, and then dropwise a solution of iodine monochloride (7.5 mmol, 1.5 equiv) in CH_2Cl_2 (7 mL) to the suspension at –20 ºC, and the mixture was stirred at the same temperature. After 0.5 h, aryl or alkyl substituted alkene (5 mmol, 1.0 equiv) in CH_2Cl_2 (7 mL) was slowly added dropwise to the above reaction solution, and the mixture was stirred for 1 h at 28 ºC. The reaction was quenched with saturated aqueous $Na_2S_2O_3$, and extracted the aqueous layer with Et_2O. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of solvents, the resulting crude material was used immediately for the next step without any further purification.

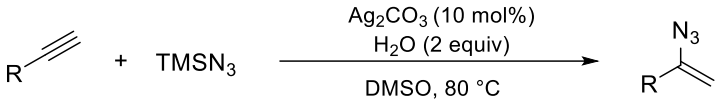
To a solution of the above obtained compound in Et_2O (26 mL) was added ^{t}BuOK (6 mmol, 1.2 equiv) portionwise at 0 ºC, and the mixture was stirred for 1 h at 28 ºC. The reaction was quenched by adding 7% aqueous $NaHCO_3$, and the aqueous layer was extracted with Et_2O. The combined Et_2O solution was washed with brine and dried over Na_2SO_4. The solvent was evaporated and resulting crude materials were purified by flash column chromatography to give the pure vinyl azides.

Method B:[6]



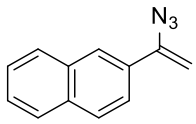
In an oven dried Schlenk flask, ICl (2.2 mmol, 1.1 equiv.) was taken in CH_3CN (2 mL) under nitrogen. To this, a solution of sodium azide (4 mmol, 2.0 equiv.) in CH_3CN (5.0 mL) was added and the resulting solution was stirred at 25 °C for 1.5 h. The reaction mixture was then cooled to 0 °C and a solution of styrene or its derivatives (2 mmol, 1.0 equiv.) in CH_3CN (1.0 mL) was added under nitrogen at 0 °C. The resulting mixture was allowed to attain ambient temperature (25 °C) and stirred at this temperature for 2 h. The reaction mixture was then quenched with saturated aqueous $Na_2S_2O_3$ solution and diluted with 2.0 mL EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc (3* 4.0 mL). Combined organic layer was washed with brine (10.0 mL), dried over Na_2SO_4 and concentrated under reduced pressure to obtain yellow oil. This residue was dissolved in Et_2O (5 mL) and ^tBuOK (4.0 mmol, 2.0 equiv.) was added to this solution under nitrogen atmosphere. The reaction mixture was stirred for 2 h at 25 °C. The reaction mixture was then diluted with 2.0 mL of H_2O. The organic layer was separated and the aqueous layer was extracted with Et_2O (3 * 4.0 mL). Combined organic layer was washed with brine (5.0 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by silica-gel flash column chromatography to obtain vinyl azides.

Method C:[7]



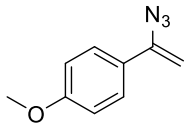
To a solution of aryl or alkyl substituted phenylacetylene (5 mmol, 1 equiv), $TMSN_3$ (10 mmol, 2.0 equiv) and H_2O (10 mmol, 2.0 equiv) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (0.5 mmol, 0.1 equiv) was added. The mixture was then stirred for 0.5-2.0 h until alkyne consumed as indicated by TLC. The resulting mixture was concentrated and extracted by dichloromethane (3 * 15 mL). The organic layer was washed with brine (3 *40 mL), dried over Na_2SO_4 and concentrated. Purification of the crude product with flash column chromatography (silica gel) to afford the pure vinyl azides.

2-(1-Azidovinyl) naphthalene (1a)



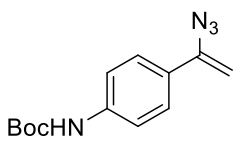
According to Method A, first step, the reaction was carried out with the 2-vinylnaphthalene (1.52 g, 10.0 mmol), iodine monochloride (2.43 g, 15 mmol), NaN_3 (1.30 g, 25 mmol), CH_3CN (8 mL), CH_2Cl_2 (26 mL). After 1.5 h, the reaction was quenched with saturated aqueous $Na_2S_2O_3$, and extracted the aqueous layer with Et_2O. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of solvent, the resulting crude material was used immediately for the next step without any further purification. Second step, Et_2O (26 mL), ^tBuOK (1.35 g, 12 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford **1a** as yellow solid (1.36 g, 70% yield): **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.05 (d, *J* = 2.4 Hz, 1H), 7.88 – 7.84 (m, 1H), 7.85 – 7.81 (m, 2H), 7.67 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.53 – 7.47 (m, 2H), 5.59 (d, *J* = 2.6 Hz, 1H), 5.06 (d, *J* = 2.6 Hz, 1H). All data are in accordance with the literature.[8]

1-(1-Azidovinyl)-4-methoxybenzene (1b)



According to Method A, first step, the reaction was carried out with the 1-(1-azidovinyl)-4-methoxybenzene (0.66 mL, 5.0 mmol), iodine monochloride (1.20 g, 7.5 mmol), NaN_3 (0.65 g, 12.5 mmol), CH_3CN (4 mL), CH_2Cl_2 (13 mL). After 1.5 h, the reaction was quenched with saturated aqueous $Na_2S_2O_3$, and extracted the aqueous layer with Et_2O. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of solvent, the resulting crude material was used immediately for the next step without any further purification. Second step, Et_2O (13 mL), ^tBuOK (0.67 g, 6 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford **1b** as yellow solid (0.49 g, 56% yield): **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.54 – 7.45 (m, 2H), 6.89 – 6.86 (m, 2H), 5.31 (d, *J* = 2.4 Hz, 1H), 4.86 (d, *J* = 2.4 Hz, 1H), 3.82 (s, 3H). All data are in accordance with the literature.[8]

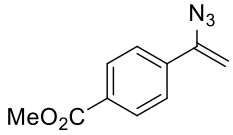
Tert-butyl (4-(1-azidovinyl) phenyl) carbamate (1c)



According to Method C, first step, the reaction was carried out with the 1-(1-azidovinyl)-4-methoxybenzene (0.65 g, 3.0 mmol), iodine monochloride (0.54 g, 3.3 mmol), NaN_3 (0.39 g, 6.0 mmol), CH_3CN (12.5 mL). After 1.5 h, the reaction was quenched with saturated aqueous $Na_2S_2O_3$, and extracted the aqueous layer with Et_2O. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of

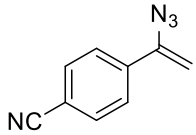
solvents, the resulting crude material was used immediately for the next step without any further purification. Second step, Et_2O (7.5 mL), ^tBuOK (0.67 g, 6 mmol), The crude product was purified by flash column chromatography on silica gel (PE: EA=100:1) to afford **1c** as white solid (0.40 g, 52% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 8.7 Hz, 2H), 6.52 (s, 1H), 5.35 (d, *J* = 2.4 Hz, 1H), 4.88 (d, *J* = 2.5 Hz, 1H), 1.52 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ152.48, 144.49, 139.17, 128.85, 126.25, 118.01, 96.63, 80.78, 28.27. **HRMS m/z (ESI)** calculated for $C_{13}H_{16}N_4O_2$ $(M + Na)^+$ 283.1165, found 283.1160.

Methyl 4-(1-azidovinyl) benzoate (1d)



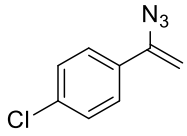
According to Method B, methyl 4-ethynylbenzoate (0.48 g, 3.0 mmol), $TMSN_3$ (0.8 mL, 6.0 mmol) and H_2O (0.11mL, 6.0 mmol) in DMSO (15 mL) at 80 ℃, Ag_2CO_3 (80 mg, 0.3 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=50:1) to afford **1d** as white solid (0.37 g, 60% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 2H), 5.52 (d, *J* = 2.8 Hz, 1H), 5.02 (d, *J* = 2.8 Hz, 1H), 3.89 (s, 3H). All data are in accordance with the literature.[9]

4-(1-Azidovinyl) benzonitrile (1e)



According to Method B, 4-ethynylbenzonitrile (0.38 g, 3.0 mmol), $TMSN_3$ (0.79 mL, 6.0 mmol) and H_2O (0.11 mL, 6.0 mmol) in DMSO (15 mL) at 80 ℃, Ag_2CO_3 (80 mg, 0.3 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=5:1) to afford **1e** as yellow solid (0.30 g, 58% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 – 7.59 (m, 4H), 5.59 (d, *J* = 3.0 Hz, 1H), 5.11 (d, *J* = 3.0 Hz, 1H). All data are in accordance with the literature.[10]

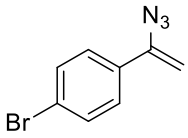
1-(1-Azidovinyl)-4-chlorobenzene (1f)



According to Method B, 1-chloro-4-ethynylbenzene (0.68 g, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE) to afford **1f** as yellow liquid (0.45 g, 50% yield). **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.55 – 7.47 (m, 2H), 7.36 – 7.29 (m, 2H), 5.43 (d, *J* = 2.7 Hz, 1H), 4.97 (d, *J* = 2.7 Hz, 1H).

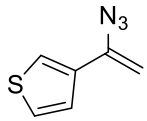
All data are in accordance with the literature.[8]

1-(1-Azidovinyl)-4-bromobenzene (1g)



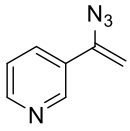
According to Method B, 1-bromo-4-ethynylbenzene (0.90 g, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=50:1) to afford **1g** as yellow solid (0.71 g, 63% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48 (d, *J* = 8.7 Hz, 2H), 7.43 (d, *J* = 8.7 Hz, 2H), 5.44 (d, *J* = 2.7 Hz, 1H), 4.97 (d, *J* = 2.7 Hz, 1H). All data are in accordance with the literature.[8]

3-(1-Azidovinyl) thiophene (1h)



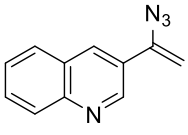
According to Method B, 3-ethynylthiophene (0.50 mL, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford **1h** as yellow solid (0.37 g, 49% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46 (dd, *J* = 3.1, 1.4 Hz, 1H), 7.35 – 7.27 (m, 1H), 7.28 – 7.20 (m, 1H), 5.36 (d, *J* = 2.5 Hz, 1H), 4.92 (d, *J* = 2.5 Hz, 1H). All data are in accordance with the literature.[11]

3-(1-Azidovinyl) pyridine (1i)



According to Method B, 3-ethynylpyridine (0.50 mL, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford **1i** as yellow solid (0.38 g, 52% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.81 (d, J = 2.3 Hz, 1H), 8.56 (dd, *J* = 4.8, 1.7 Hz, 1H), 7.82 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.35 – 7.24 (m, 1H), 5.51 (d, *J* = 2.8 Hz, 1H), 5.04 (d, *J* = 2.8 Hz, 1H). All data are in accordance with the literature.[12]

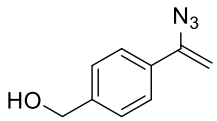
3-(1-Azidovinyl) quinolone (1j)



According to Method B, 3-ethynylquinoline (0.76 g, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The

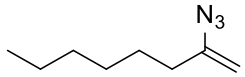
crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford **1j** as white solid (0.29 g, 30% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.13 (d, *J* = 2.3 Hz, 1H), 8.30 (d, *J* = 2.5 Hz, 1H), 8.10 (dd, *J* = 8.5, 1.0 Hz, 1H), 7.85 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.73 (ddd, *J* = 8.4, 6.9, 1.4 Hz, 1H), 7.57 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 5.70 (d, *J* = 2.9 Hz, 1H), 5.16 (d, *J* = 3.0 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 147.93, 147.67, 142.76, 132.49, 130.14, 129.16, 128.33, 127.33, 127.27, 99.22. **HRMS m/z (ESI)** calculated for $C_{11}H_8N_4$ $(M+H)^+$ 197.0822, found 197.0818.

(4-(1-Azidovinyl) phenyl) methanol (1k)



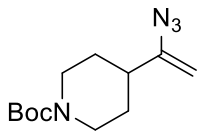
According to Method C, 3-ethynylquinoline (0.66 g, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford **1k** as yellow solid (0.45 g, 52% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.8 Hz, 2H), 5.43 (d, *J* = 2.4 Hz, 1H), 4.96 (d, *J* = 2.4 Hz, 1H), 4.71 (d, *J* = 4.1 Hz, 2H), 1.66 (s, 1H). All data are in accordance with the literature.[9]

2-Azidooct-1-ene (1l)



According to Method B, oct-1-yne (0.69 mL, 5.0 mmol), $TMSN_3$ (1.32 mL, 10.0 mmol) and H_2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE) to afford **1l** as yellow liquid (0.48 g, 63% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.62 (q, *J* = 1.3 Hz, 2H), 2.18 – 1.99 (m, 2H), 1.54 – 1.41 (m, 2H), 1.35 – 1.24 (m, 6H), 0.96 – 0.83 (m, 3H). All data are in accordance with the literature.[12]

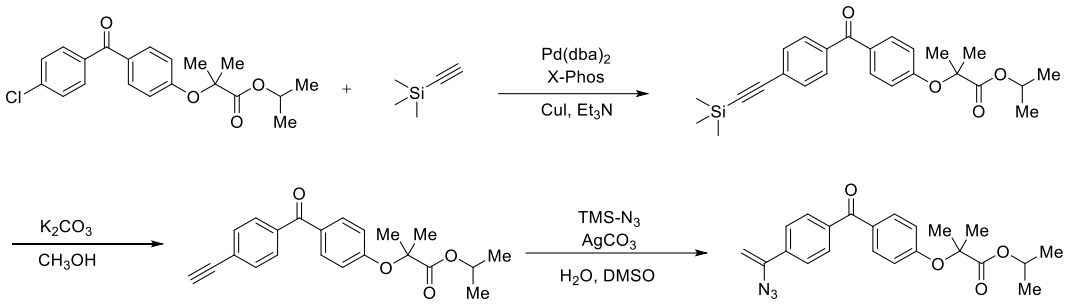
Tert-butyl 4-(1-azidovinyl) piperidine-1-carboxylate (1m)

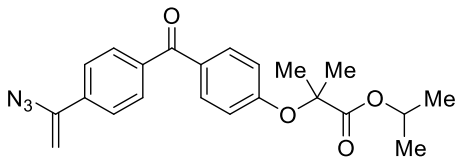


According to Method B, tert-butyl 4-ethynylpiperidine-1-carboxylate (1.04 g, 5.0 mmol), TMSN3 (1.32 mL, 10.0 mmol) and H2O (0.18mL, 10.0 mmol) in DMSO (20 mL) at 80 ℃, Ag_2CO_3 (138 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford **1m** as yellow solid (0.76 g, 60% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.73 (d, *J* = 1.1 Hz, 1H), 4.63 (d, *J* = 2.1 Hz,

1H), 4.14 (s, 2H), 2.84 – 2.55 (m, 2H), 2.01 (tt, *J* = 11.8, 3.5 Hz, 1H), 1.78 – 1.69 (m, 2H), 1.44 (s, 9H), 1.40 – 1.27 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 154.65, 150.58, 96.17, 79.43, 40.47, 30.08, 28.39. **HRMS m/z (ESI)** calculated for $C_{12}H_{20}N_4O_2$ $(M + H)^+$ 253.1659, found 253.1654.

Isopropyl 2-(4-(4-(1-azidovinyl)benzoyl) phenoxy)-2-methylpropanoate (1n)



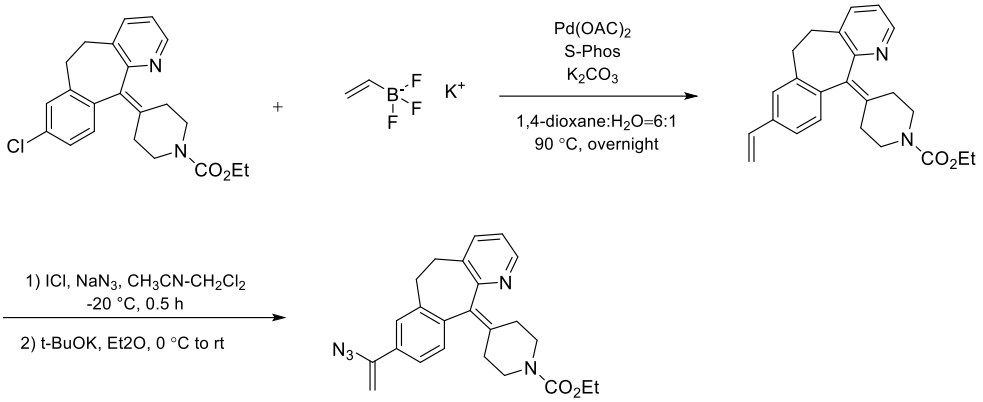


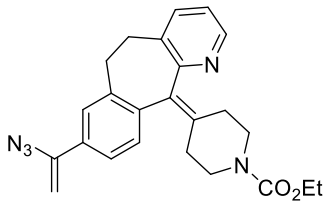
Fenofibtate (1.80 g, 5 mmol), $Pd(dba)_2$ (288 mg, 0.4 mmol), X-Phos (477 mg, 0.8 mmol), CuI (95 mg, 0.8 mmol) into an oven dried Schlenk flask with a stir bar. Evacuate and backfill the reaction flask with argon (repeate this process for a total of 3 times). And then ethynyltrimethylsilane (0.8 mL, 6 mmol), Et_3N (30 mL) were added. Stir the reaction at 100 ℃ for 36 h. Allow to cool the reaction mixture to room temperature and pass through a pad of Celite and rinsed with EA. Partition the filtrate between EA and H_2O. Extract the aqueous layer with EA. Wash the combined organic layers with brine, dry over Na_2SO_4, filter and concentrate. Purify the residue by flash column chromatography (PE: EA=50:1) to obtain the isopropyl 2-methyl-2-(4-(4-((trimethylsilyl)ethynyl) benzoyl) phenoxy) propanoate as a yellow solid (1.4 g, 60% yield). In 100 mL round-bottom flask, K_2CO_3 (1.2 eq), MeOH was added. The reaction was stirred at room temperature for 3 h, and the solvent was removed under vacuum. The solid was redissolved in EA and was washed with aqueous $NaHCO_3$ three times. The organic layer was dried over Na_2SO_4 and evaporated under vacuum. The crude product was used in the next step without further purification.

According to Method B, isopropyl 2-(4-(4-ethynylbenzoyl) phenoxy)-2-methylpropanoate (1.05 g 3.0 mmol), $TMSN_3$ (0.79 mL, 6.0 mmol) and H_2O (0.11 mL, 6.0 mmol) in DMSO (15 mL) at 80 ℃, Ag_2CO_3 (80 mg, 0.5 mmol) was added. The crude product was purified by flash column chromatography on silica gel (PE: EA=50:1)

to afford **1n** as yellow liquid (0.53 g, 45% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 – 7.70 (m, 4H), 7.66 (d, *J* = 8.5 Hz, 2H), 6.93 – 6.83 (m, 2H), 5.57 (d, *J* = 2.7 Hz, 1H), 5.15 – 5.01 (m, 2H), 1.66 (s, 6H), 1.20 (d, *J* = 6.2 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.73, 173.09, 159.66, 144.28, 138.46, 137.45, 131.96, 130.39, 129.89, 125.28, 117.19, 99.48, 79.37, 69.29, 25.34, 21.48. **HRMS m/z (ESI)** calculated for $C_{22}H_{23}N_3O_4$ $(M+H)^+$ 394.1761, found 394.1757.

Ethyl 4-(8-(1-azidovinyl)-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate (1o)





Charge loratadine (1.53 g, 4 mmol), $Pd(OAc)_2$ (45 mg, 0.2 mmol, 5 mol%), S-Phos (164 mg, 0.4 mmol,), potassium vinyl trifluoroborate (0.8 mg, 6.0 mmol), K_2CO_3 (1.60 g, 12 mmol) into an oven dried Schlenk flask with a stir bar. Evacuate and backfill the reaction flask with argon (repeat this process for a total of 3 times. Add dioxane/H_2O (6:1, 50 mL) via syringe. Stir the reaction for 2 h and add an additional amount of potassium vinyl trifluoroborate (0.60 g, 4.5 mmol), K_2CO_3 (1.0 g, 7.2 mmol) to the reaction mixture. Stir the reaction mixture at 90 °C overnight. Allow to cool the reaction mixture to room temperature and pass through a pad of celiteand rinsed with EA. Partition the filtrate between EA and H_2O. Extract the aqueous layer with EA. Wash the combined organic layers with brine, dry over Na_2SO_4, filter and concentrate. Purify the residue by flash column chromatography (PE: EA=2:1) to obtain the ethyl 4-(8-vinyl-5,6-dihydro-11H-benzo [5,6] cyclohepta[1,2-b] pyridin-11-ylidene) piperidine-1-carboxylate as a yellow solid (1.35 g, 90% yield).

According to Method A, first step, the reaction was carried out with the ethyl 4-

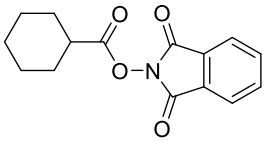
(8-vinyl-5,6-dihydro-11H-benzo [5,6] cyclohepta[1,2-b] pyridin-11-ylidene) piperidine-1-carboperoxoate (1.17 g, 3.0 mmol), iodine monochloride (0.73 g, 4.5 mmol), NaN_3 (0.39 g, 7.5 mmol), CH_3CN (3 mL), DCM (8 mL). After 1.5 h, the reaction was quenched with saturated aqueous $Na_2S_2O_3$, and extracted the aqueous layer with Et_2O. The combined organic layers were washed with brine and dried over Na_2SO_4. After evaporation of solvent, the resulting crude material was used immediately for the next step without any further purification. Second step, Et_2O (8 mL), ^tBuOK (0.4 g, 3.6 mmol), The crude product was purified by flash column chromatography on silica gel (PE:EA=2:1) to afford **1o** as yellow solid (0.75 g, 60% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.42 – 8.34 (m, 1H), 7.51 – 7.32 (m, 3H), 7.17 (d, *J* = 8.4 Hz, 1H), 7.07 (dd, *J* = 7.7, 4.8 Hz, 1H), 5.39 (d, *J* = 2.5 Hz, 1H), 4.90 (d, *J* = 2.5 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.80 (t, *J* = 13.7 Hz, 2H), 3.48 – 3.30 (m, 2H), 3.18 – 3.07 (m, 2H), 2.84 (q, *J* = 7.0, 5.2 Hz, 2H), 2.54 – 2.42 (m, 1H), 2.40 – 2.23 (m, 3H), 1.23 (t, J = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 157.12, 155.42, 146.57, 144.64, 140.08, 137.77, 137.37, 137.17, 134.70, 133.47, 133.28, 129.39, 126.14, 123.19, 122.13, 97.60, 61.22, 44.78, 44.74, 31.86, 31.57, 30.70, 30.47, 14.61. **HRMS m/z (ESI)** calculated for $C_{24}H_{25}N_5O_2$ $(M+H)^+$ 416.2081, found 416.2105.

5. Preparation of Alkyl *N*-Hydroxyphthalimide Esters (NHPI Esters)

Method D:[13]

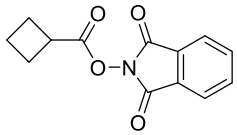
The alkyl redox active esters were synthesized according to the following method: the corresponding carboxylic acids or *N*-protected amino acids (6.0 mmol, 1.2 equiv), *N*-hydroxyphthalimide (5.0 mmol, 1.0 equiv), and 4-dimethylaminopyridine (0.25 mmol, 0.05 equiv) were mixed in a flask with a magnetic stirring bar, 30 mL CH_2Cl_2 was added. Then a solution of *N*, *N′*-dicyclohexylcarbodiimide (6.0 mmol, 1.2 equiv) in CH_2Cl_2 (10 mL) was added slowly at room temperature. The reaction mixture was maintained at room temperature with stirring for 1-3 h. When the *N*-hydroxyphthalimide was completely converted, the white precipitate was filtered off and the solution was concentrated on a rotary evaporator. The residue was purified by flash column chromatography to give corresponding redox active esters.

1,3-Dioxoisoindolin-2-yl cyclohexanecarboxylate (2a)



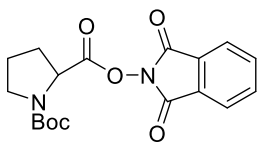
According to Method D, the reaction was carried out with the cyclohexanecarboxylic acid (768 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′* -dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2a** as white solid (1.09 g, 80% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 2.81 – 2.66 (m, 1H), 2.16 – 2.00 (m, 2H), 1.88 – 1.77 (m, 2H), 1.69 – 1.64 (m, 2H), 1.54 – 1.14 (m, 4H). All data are in accordance with the literature.[14]

1,3-Dioxoisoindolin-2-yl cyclobutanecarboxylate (2b)



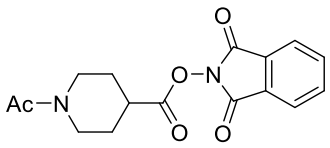
According to Method D, the reaction was carried out with the Cyclobutanecarboxylic acid (600 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2b** as white solid (0.83 g, 68% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.45-3.54 (m, 1H), 2.34 – 2.52 (m, 4H), 2.00-2.12 (m, 2H). All data are in accordance with the literature.[14]

1-(Tert-butyl) 2-(1,3-dioxoisoindolin-2-yl) pyrrolidine-1,2-dicarboxylate (2c)



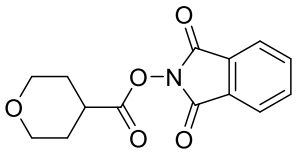
According to Method D, the reaction was carried out with the tert-butyl (*2S*)-2-acetylpyrrolidine-1-carboxylate (1.29 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2c** as white solid (1.17 g, 65% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 4.63-4.59 (m, 1H), 3.66-3.59 (m, 1H), 3.52-3.44 (m, 1H), 2.49 – 2.32 (m, 2H), 2.13 – 1.93 (m, 2H), 1.51 (s, 9H). All data are in accordance with the literature.[15]

1,3-Dioxoisoindolin-2-yl 1-acetylpiperidine-4-carboxylate (2d)



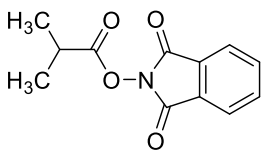
According to Method D, the reaction was carried out with the 1-acetyl-piperidine-4-carboxylic acid (1.03 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2d** as white solid (0.74 g, 47% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.90 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 4.36 (dt, *J* = 13.5, 4.4 Hz, 1H), 3.84 (dt, *J* = 14.5, 4.8 Hz, 1H), 3.28 (m, 1H), 3.13 – 2.95 (m, 2H), 2.16-2.08 (m, 5H), 2.02 – 1.79 (m, 2H). All data are in accordance with the literature.[16]

1,3-Dioxoisoindolin-2-yl tetrahydro-2H-pyran-4-carboxylate (2e)



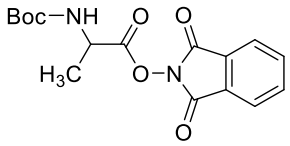
According to Method D, the reaction was carried out with the Tetrahydropyran-4-carboxylic acid (780 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 2: 1) to afford **2e** as white solid (0.74 g, 54% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (dd *J* = 5.1, 2.7 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 4.03 (dt, *J* = 11.8, 3.9 Hz, 2H), 3.57-3.50 (m, 2H), 3.04-2.97 (m, 1H), 2.07-1.94 (m, 4H). All data are in accordance with the literature.[15]

1,3-Dioxoisoindolin-2-yl isobutyrate (2f)



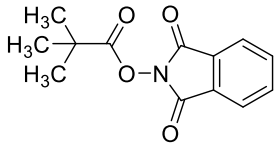
According to Method D, the reaction was carried out with the isobutyric acid (529 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1). afford **2f** as white solid (0.82 g, 70% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.88 (dd, *J* =5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 2.96 (m, 1H), 1.38 (d, *J* = 7.0 Hz, 6H). All data are in accordance with the literature.[17]

1,3-Dioxoisoindolin-2-yl (tert-butoxycarbonyl)-L-alaninate (2g)



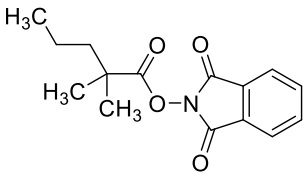
According to Method D, the reaction was carried out with the *N*-(tert-Butoxycarbonyl)-L-alanine (1.13 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1) to afford **2g** as white solid (0.75 g, 45% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 5.04-4.52 (m, 2H), 1.63 (d, *J* = 7.2 Hz, 3H), 1.47 (s, 9H). All data are in accordance with the literature.[14]

1,3-Dioxoisoindolin-2-yl pivalate (2h)



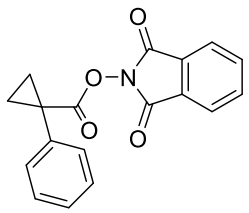
According to Method D, the reaction was carried out with the pivalic acid (612 mg, 6mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2h** as a white solid (0.84 g, 68% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 1.43 (s, 9H). All data are in accordance with the literature.[14]

1,3-Dioxoisoindolin-2-yl 2,2-dimethylpentanoate (2i)



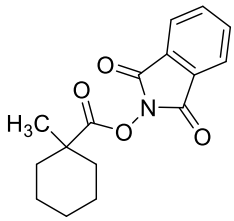
According to Method D, the reaction was carried out with the 2,2-dimethylpentanoic acid (0.43ml, 3.0 mmol), *N*-hydroxyphthalimide (408 mg, 2.5 mmol), 4-dimethylaminopyridine (15.8 mg, 0.13 mmol), *N, N′*-dicyclohexylcarbodiimide (515 mg, 3.0 mmol), DCM (10 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2i** as white solid (0.45 g, 65% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 1.72 – 1.66 (m, 2H), 1.49 – 1.40 (m, 2H), 1.37 (s, 6H), 0.96 (t, *J* = 7.3 Hz, 3H). All data are in accordance with the literature.[18]

1,3-Dioxoisoindolin-2-yl 1-phenylcyclopropane-1-carboxylate (2j)



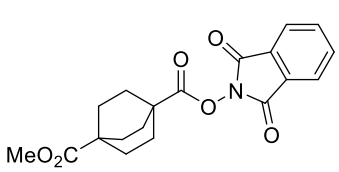
According to Method D, the reaction was carried out with the 1-Phenylcyclopropanecarboxylic acid (970 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2j** as white solid (0.80 g, 52% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.56 – 7.48 (m, 2H), 7.43 – 7.28 (m, 3H), 1.92-1.89 (m, 2H), 1.51-1.48 (m, 2H). All data are in accordance with the literature.[15]

1,3-Dioxoisoindolin-2-yl 1-methylcyclohexane-1-carboxylate (2k)



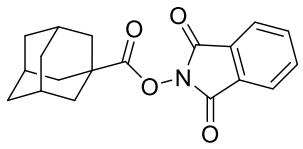
According to Method D, the reaction was carried out with the 1-Methylcyclohexanecarboxylic acid (427 mg, 3.0 mmol), *N*-hydroxyphthalimide (408 mg, 2.5 mmol), 4-dimethylaminopyridine (15.8 mg, 0.13 mmol), *N, N′*-dicyclohexylcarbodiimide (515 mg, 3.0 mmol), DCM (10 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2k** as white solid (0.38 g, 52% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 2.38 – 2.18 (m, 2H), 1.71 – 1.57 (m, 5H), 1.45 (s, 3H), 1.44 – 1.33 (m, 3H). All data are in accordance with the literature.[14]

1-(1,3-Dioxoisoindolin-2-yl) 4-methyl bicyclo [2.2.2] octane-1,4-dicarboxylate (2l)



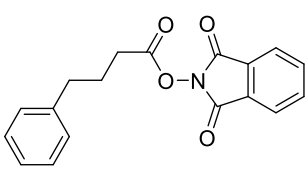
According to Method D, the reaction was carried out with the bicycle [2.2.2] octane-1,4-dicarboxylic acid monomethyl ester (1.27 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 2: 1) to afford **2l** as white solid (1.36 g, 76% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.67 (s, 3H), 2.14 – 1.99 (m, 6H), 1.97 – 1.83 (m, 6H). All data are in accordance with the literature.[19]

1,3-Dioxoisoindolin-2-yl (3r,5r,7r)-adamantane-1-carboxylate (2m)



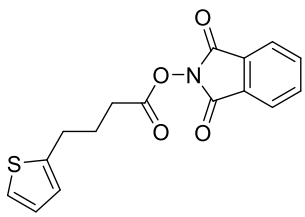
According to Method D, the reaction was carried out with the 1-Adamantane Carboxylic Acid (1.08 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylamino-pyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodii-mide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2m** as white solid (1.30 g, 80 % yield).**^{1}H NMR (600 MHz, CDCl$_3$)** δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 2.09-2.16 (m, 9H), 1.75-1.81 (m, 6H). All data are in accordance with the literature.[15]

1,3-Dioxoisoindolin-2-yl 4-phenylbutanoate (2n)



According to Method D, the reaction was carried out with the 4-phenylbutanoic acid (984 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 g, 5.0 mmol), 4-dimethylamino-pyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodii-mide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2n** as white solid (1.24 g, 80 % yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.82 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.29 – 7.20 (m, 2H), 7.19-7. 12(m, 3H), 2.71 (t, *J* = 7.5 Hz, 2H), 2.60 (t, *J* = 7.3 Hz, 2H), 2.05 (p, *J* = 7.5 Hz, 2H). All data are in accordance with the literature.[17]

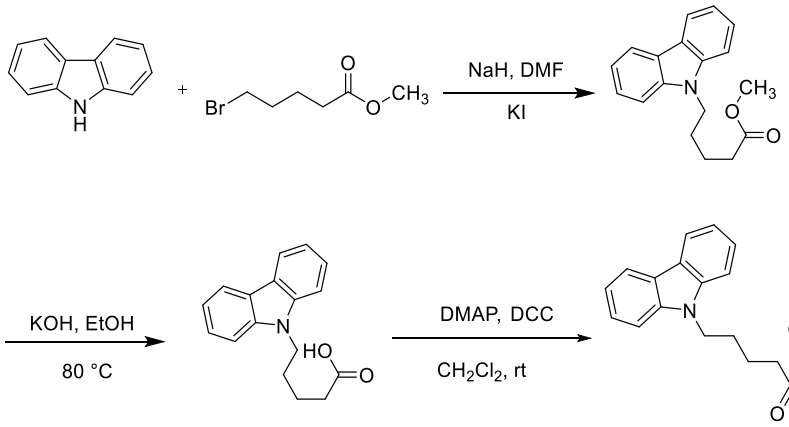
1,3-Dioxoisoindolin-2-yl 4-(thiophen-2-yl) butanoate (2o)

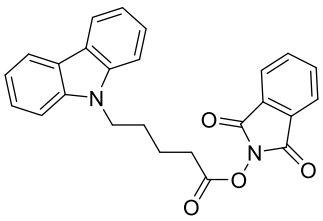


According to Method D, the reaction was carried out with the 2-thiophenebutyric acid (1.02 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2o** as white solid (1.04 g, 66% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.15 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.94 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.90 – 6.83 (m, 1H), 3.01 (t, *J* = 7.3 Hz, 2H), 2.72 (t, *J* = 7.3 Hz, 2H), 2.16 (p, *J* = 7.3 Hz, 2H). All data are in accordance with the

literature.[20]

1,3-Dioxoisoindolin-2-yl 5-(9H-carbazol-9-yl) pentanoate (2p)



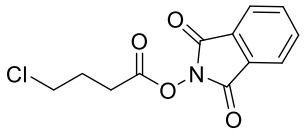


Carbazole (2.01 g, 12.0 mmol) and sodium hydride (60 wt. % in mineral oil, 0.29 g, 12.0 mmol) were placed under argon and dissolved in DMF (10 mL). The mixture was stirred at room temperature for 30 min, followed by addition of 5-bromovaleric acid methyl ester (2.33 g, 12.0 mmol) and potassium iodide (10 mg, 0.06 mmol). The reaction was heated to 80 °C for 2 h upon which a precipitate formed and the reaction turned from dark brown to dark orange. The reaction was then quenched with water (30 mL) and ethyl acetate (30 mL). The organic layer was isolated and the aqueous layer extracted with ethyl acetate (2*10 mL). The combined organic layers were washed with water (2*20 mL), brine (15 mL), dried by Na_2SO_4 and concentrated in vacuo. Purification by column chromatography (PE: EA = 5: 1-2: 1) afforded the title compound (2.34 g, 69 %) as a light yellow solid. In a flask with a solution of KOH (3.02 g, 51.35 mmol) in ethanol (30 mL) and water (8 mL) was added methyl 5-(9H-carbazol-9-yl) pentanoate (2.1 g, 7.5 mmol). The reaction mixture was stirred at 80 ºC for 6 hours. The reaction crude was cooled, diluted with water and acidified with HCl (6 M) in an ice bath. A white solid was isolated by filtration and washed with water. The solid obtained was purified by recrystallization using PE/EA to give a white solid.

According to Method D, the reaction was carried out with the gemfibrozil (1.41 g, 4.8mmol), *N*-hydroxyphthalimide (652 mg, 4.0 mmol), 4-dimethylaminopyridine (24.4 mg, 0.2 mmol), *N, N*′-dicyclohexylcarbodiimide (820 mg, 4.8 mmol), DCM (16 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1-2: 1) to afford **2p** as yellow solid (0.81 g, 49% yield). **^{1}H NMR (400 MHz,**

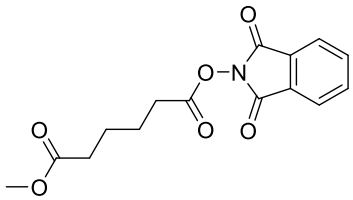
$CDCl_3$) δ 8.11 (d, *J* = 7.7 Hz, 2H), 7.92 – 7.82 (m, 2H), 7.82 – 7.71 (m, 2H), 7.55 – 7.38 (m, 4H), 7.27-7.23 (m, 2H), 4.38 (t, *J* = 6.9 Hz, 2H), 2.67 (t, *J* = 7.1 Hz, 2H), 2.15 – 1.98 (m, 2H), 1.98 – 1.77 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.15, 161.85, 140.26, 134.71, 128.82, 125.70, 123.91, 122.84, 120.35, 118.89, 108.54, 42.42, 30.63, 28.07, 22.40. **HRMS m/z (ESI)** calculated for $C_{25}H_{20}N_2O_4$ $(M+H)^+$ 413.1496, found 413.1518.

1,3-Dioxoisoindolin-2-yl 4-chlorobutanoate (2q)



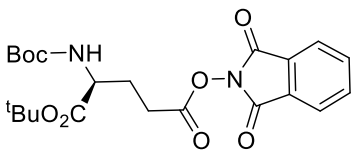
According to Method D, the reaction was carried out with the 1-Adamantane Carboxylic Acid (735 mg, 0.61mL, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1) to afford **2q** as white solid (0.84 g, 63% yield).**^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.67 (t, *J* = 6.3 Hz, 2H), 2.87 (t, *J* = 7.2 Hz, 2H), 2.34 – 2.19 (m, 2H). All data are in accordance with the li6terature.[20]

1,3-Dioxoisoindolin-2-yl methyl adipate (2r)



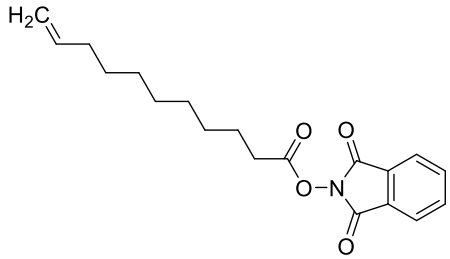
According to Method D, the reaction was carried out with the adipic acid monomethyl ester (961 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2r** as white solid (0.93 g, 61% yield).**^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83 (dd, J = 5.5, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.63 (s, 3H), 2.65 (t, *J* = 7.1 Hz, 2H), 2.34 (t, *J* = 6.9 Hz, 2H), 1.83 – 1.70 (m, 4H). All data are in accordance with the literature.[17]

1-(Tert-butyl) 5-(1,3-dioxoisoindolin-2-yl) (tert-butoxycarbonyl)-D-glutamate (2s)



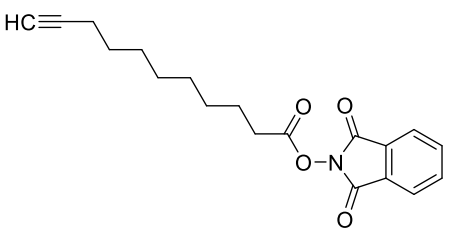
According to Method D, the reaction was carried out with the boc-D-glutamic acid α-tert-butyl ester (1.82 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1) to afford **2s** as white solid (0.90 g, 40% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 5.19 (d, *J* = 8.2 Hz, 1H), 4.34 – 4.13 (m, 1H), 3.01 – 2.60 (m, 2H), 2.33-2.29 (m, 1H), 2.21 – 1.92 (m, 1H), 1.49 (s, 9H), 1.45 (s, 9H). All data are in accordance with the literature.[21]

1,3-Dioxoisoindolin-2-yl undec-10-enoate (2t)



According to Method D, the reaction was carried out with the undec-10-enoic acid (1.11 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10: 1) to afford **2t** as white solid (1.17 g, 71% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 5.92 – 5.71 (m, 1H), 5.05 – 4.86 (m, 2H), 2.65 (t, *J* = 7.5 Hz, 2H), 2.09 – 1.98 (m, 2H), 1.78 (p, *J* = 7.5 Hz, 2H), 1.48 – 1.28 (m, 10H). All data are in accordance with the literature.[22]

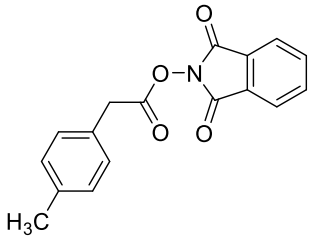
1,3-Dioxoisoindolin-2-yl undec-10-ynoate (2u)



According to Method D, the reaction was carried out with the undec-10-ynoic acid (1.09 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2u** as white solid (1.15 g, 70% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5,

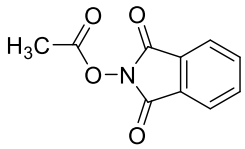
3.1 Hz, 2H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.19 (td, *J* = 7.0, 2.7 Hz, 2H), 1.94 (t, *J* = 2.6 Hz, 1H), 1.82-1.75 (m, 2H), 1.57-1.50 (m, 2H), 1.46 – 1.31 (m, 8H). All data are in accordance with the literature.[17]

1,3-Dioxoisoindolin-2-yl 2-(p-tolyl) acetate (2v)



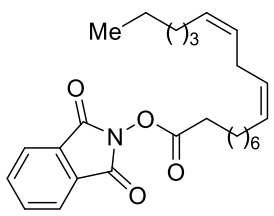
According to Method D, the reaction was carried out with the 4-tolylacetic acid (900 mg, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2v** as white solid (1.05 g, 71% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 3.93 (s, 2H), 2.33 (s, 3H). All data are in accordance with the literature.[23]

1,3-Dioxoisoindolin-2-yl acetate (2w)



According to Method D, the reaction was carried out with the Acetic Acid (360 mg, 0.33ml, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2w** as white solid (0.74 g, 72% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 2.40 (s, 3H). All data are in accordance with the literature.[21]

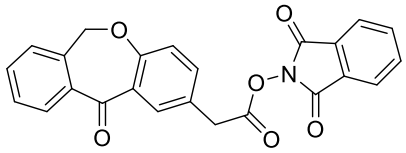
1,3-Dioxoisoindolin-2-yl (9Z,12Z)-octadeca-9,12-dienoate (2x)



According to Method D, the reaction was carried out with the linoleic acid (1.68 g, 1.87ml, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2x** as colorless oil (1.36 g, 64% yield): **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5,

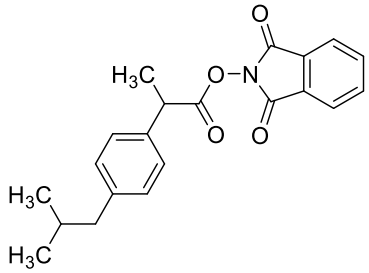
3.1 Hz, 2H), 5.49 – 5.20 (m, 4H), 2.78 (t, *J* = 6.5 Hz, 2H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.12 – 1.99 (m, 4H), 1.79 (p, *J* = 7.5 Hz, 2H), 1.49 – 1.41 (m, 2H), 1.40 – 1.26 (m, 12H), 0.89 (t, *J* = 6.8 Hz, 3H). All data are in accordance with the literature.[24]

1,3-Dioxoisoindolin-2-yl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl)acetate (2y)



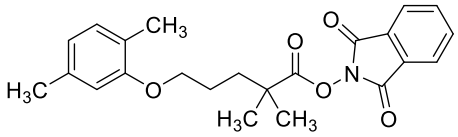
According to Method D, the reaction was carried out with the isoxepac (1.60 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1) to afford **2y** as white solid (1.05 g, 51% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.22 (d, *J* = 2.4 Hz, 1H), 7.93 – 7.85 (m, 3H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.63 – 7.51 (m, 2H), 7.48 (td, *J* = 7.6, 1.3 Hz, 1H), 7.37 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 1H), 5.20 (s, 2H), 4.02 (s, 2H). All data are in accordance with the literature.[15]

1,3-Dioxoisoindolin-2-yl 2-(4-isobutylphenyl) propanoate (2z)



According to Method D, the reaction was carried out with the ibuprofen (1.24 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 4: 1) to afford **2z** as white solid (0.84 g, 48% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 4.10 (q, *J* = 7.2 Hz, 1H), 2.48 (d, *J* = 7.2 Hz, 2H), 1.87 (dt, *J* = 13.5, 6.8 Hz, 1H), 1.67 (d, *J* = 7.2 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H). All data are in accordance with the literature.[25]

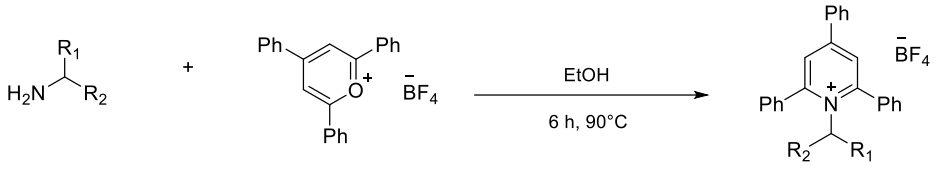
1,3-Dioxoisoindolin-2-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (2aa)



According to Method D, the reaction was carried out with the gemfibrozil (1.50 g, 6.0 mmol), *N*-hydroxyphthalimide (815 mg, 5.0 mmol), 4-dimethylaminopyridine (30.5 mg, 0.25 mmol), *N, N′*-dicyclohexylcarbodiimide (1.03 g, 6.0 mmol), DCM (20 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5: 1) to afford **2aa** as white solid (1.19 g, 60% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.01 (d, *J* = 7.6 Hz, 1H), 6.66 (d, *J* = 6.6 Hz, 2H), 4.06 – 3.97 (m, 2H), 2.32 (s, 3H), 2.19 (s, 3H), 1.96 (d, *J* = 2.8 Hz, 4H), 1.46 (s, 6H). All data are in accordance with the literature.[15]

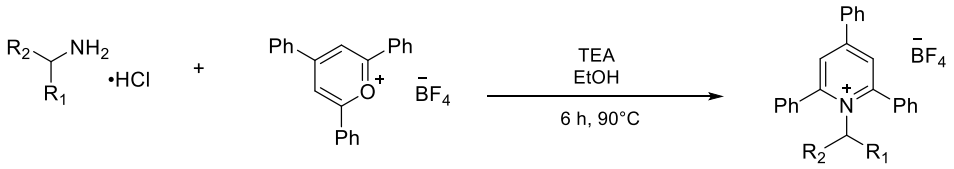
6.Preparation of Katritzky Salts

Method E:[26]



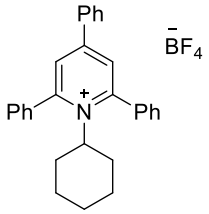
An oven dried equipped with a stir bar was charged with the amine (6 mmol, 1.2 equiv), 2,4,6- triphenylpyryliumtetrafluoroborate (5mmol, 1.0 equiv) and EtOH (2.5 mL) was added. The mixture was stirred and heated at reflux in an oil bath at 90 ºC for 6 hours. The mixture was then allowed to cool to room temperature. If product precipitation occurred during reflux, the solid was filtered, washed with EtOH (3*5 mL) and then Et_2O (3*20 mL), and dried under high vacuum. If product precipitation did not occur during reflux, the solution was diluted with Et_2O (3*volume of EtOH was used) and vigorously stirred for 1 h to induce trituration. The resulting solid pyridinium salt was filtered and washed with Et_2O (3*10 mL). If the salt still did not precipitate, it was subjected to silica gel chromatography with acetone/DCM.

Method F: [26]



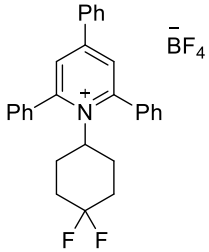
An oven dried equipped with a stir bar was charged with the amine hydrochloride (6mmol, 1.2 equiv.), 2,4,6-triphenylpyryliumtetrafluoroborate (5mmol, 1.0 equiv.), TEA (6mmol, 1.2 equiv.) and EtOH (5 mL) was added. The mixture was stirred and heated at reflux in an oil bath at 90 ºC for 6 hours. The mixture was then allowed to cool to room temperature. If product precipitation occurred during reflux, the solid was filtered, washed with EtOH (3*5 mL) and then Et_2O (3*20 mL), and dried under high vacuum. If product precipitation did not occur during reflux, the solution was diluted with Et_2O (3*volume of EtOH was used) and vigorously stirred for 1 hour to induce trituration. The resulting solid pyridinium salt was filtered and washed with Et_2O (3*10 mL). If the salt still did not precipitate, it was subjected to silica gel chromatography with acetone/DCM.

1-Cyclohexyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (4a)



According to Method E, the reaction was carried out with the corresponding amine (1.38 mL, 12.0 mmol), triphenylpyryliumtetrafluoroborate (3.96 g, 10.0 mmol), EtOH (5 mL). Et_2O was used to wash thrice to obtain the precipitate **4a** as white solid (3.14 g, 66% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ: 7.86-7.74 (m, 8H), 7.61-7.50 (m, 9H), 4.61-4.67 (m, 1H), 2.11 (d, *J* = 12.0 Hz, 2H), 1.61-1.34 (m, 5H), 0.80-0.60 (m, 3H). All data are in accordance with the literature. All data are in accordance with the literature.[26]

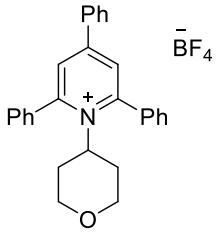
1-(4,4-Difluorocyclohexyl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (4b)



According to Method F, the reaction was carried out with the corresponding amine hydrochloride (0.82 g, 4.8 mmol), triphenylpyryliumtetrafluoroborate (1.58 g, 4.0 mmol), TEA (0.64 mL, 4.8 mmol), EtOH (4 mL). Et_2O was used to wash thrice to obtain the precipitate **4b** as white solid (1.31 g, 64% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ: 7.87-7.76 (m, 8H), 7.62-7.48 (m, 9H), 4.74-4.70 (m,

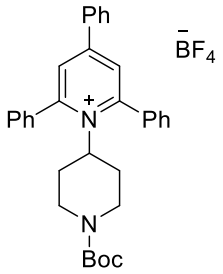
1H), 2.29-2.26 (m, 2H), 1.96-1.79 (m, 4H), 1.30-1.15 (m, 2H). All data are in accordance with the literature. All data are in accordance with the literature.[26]

2,4,6-Triphenyl-1-(tetrahydro-2H-pyran-4-yl) pyridin-1-ium tetrafluoroborate (4c)



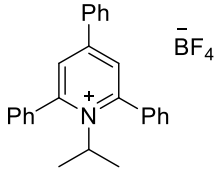
According to Method E, the reaction was carried out with the corresponding amine (0.63 mL, 6.0 mmol), triphenylpyryliumtetrafluoroborate (1.98 g, 5.0 mmol), EtOH (2.5 mL). Et_2O was used to wash thrice to obtain the precipitate **4c** as white solid (1.96 g, 82% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 – 7.71 (m, 6H), 7.69 – 7.46 (m, 9H), 7.46 – 7.35 (m, 2H), 4.94 – 4.81 (m, 1H), 3.70 (dd, *J* = 11.6, 4.1 Hz, 2H), 2.85 – 2.68 (m, 2H), 2.15 – 2.00 (m, 2H), 1.98 – 1.80 (m, 2H). All data are in accordance with the literature.[26]

1-(1-(Tert- butoxycarbonyl) piperidin-4-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (4d)



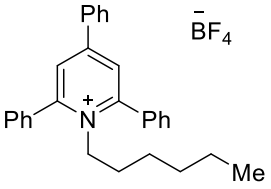
According to Method E, the reaction was carried out with the corresponding amine (1.20 g, 6.0 mmol), triphenylpyryliumtetrafluoroborate (1.98 g, 5.0 mmol), EtOH (2.5 mL). The crude product was purified by flash column chromatography on silica gel (DCM: acetone=5:1) to afford **4d** as white solid (1.84 g, 64% yield): **^{1}H NMR (400 MHz, CDCl3)** δ: 7.86 (s, 2H), 7.78-7.74 (m, 6H), 7.61-7.48 (m, 9H), 4.79 (t, *J* = 12.1 Hz, 1H), 4.08-3.81 (m, 2H), 2.23-1.94 (m, 4H), 1.78 – 1.53 (m, 2H), 1.31 (s, 9H). All data are in accordance with the literature.[26]

1-Isopropyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (4e)



According to Method E, the reaction was carried out with the corresponding amine (0.41 mL, 4.8 mmol), triphenylpyryliumtetrafluoroborate (1.58 g, 4.0 mmol), EtOH (2 mL). Et_2O was used to wash thrice to obtain the precipitate **4e** as white solid (1.04 g, 60% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ: 7.83-7.75 (m, 8H), 7.59-7.49 (m, 9H), 5.17-5.10 (m, 1H), 1.37 (d, *J* = 7.0 Hz, 6H). All data are in accordance with the literature.[26]

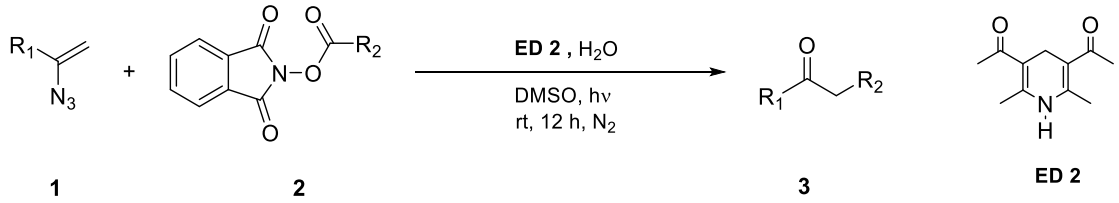
1-Hexyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (4f)



According to Method E, the reaction was carried out with the corresponding amine (0.63 mL, 4.8 mmol), triphenylpyrylium-tetrafluoroborate (1.58 g, 4.0 mmol), EtOH (2 mL). Et_2O was used to wash thrice to obtain the precipitate **4f** as white solid (1.72 g, 90% yield): **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.85 – 7.70 (m, 8H), 7.63 – 7.46 (m, 9H), 4.48 – 4.34 (m, 2H), 1.48 – 1.38 (m, 2H), 1.00 – 0.88 (m, 2H), 0.80 – 0.70 (m, 4H), 0.67-0.64 (m, 3H). All data are in accordance with the literature.[26]

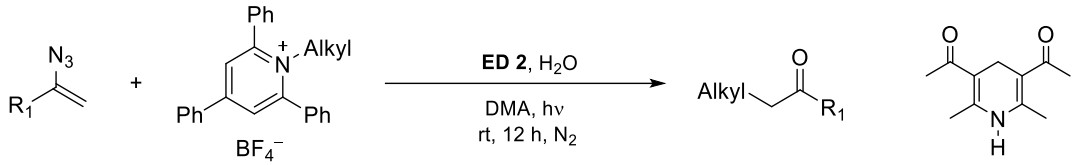
7. Experimental Procedures and Characterization of Products

Procedure A:



In a nitrogen-filled glovebox, an oven-dried 8.0 mL vial with a stirring bar was added with vinyl azides (**1**) (0.3 mmol, 1.0 equiv.), Alkyl *N*-hydroxyphthalimide Esters (NHPI Esters) (**2**) (0.45 mmol, 1.5 equiv), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.45 mmol, 1.5 equiv). A mixture of Dimethyl sulfoxide (DMSO) (3 mL) and H_2O (0.45 mmol, 1.5 eq) were added. The resulting mixture was stirred at room temperature under blue LEDS (456 nm, 25% light intensity) irradiation for 12 h. After this time, the reaction mixture was diluted with EA and washed with a saturated brine for 5-6 times. The organic layer was separated, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography.

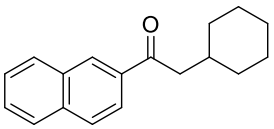
Procedure B:



1 **4** **3** **ED 2**

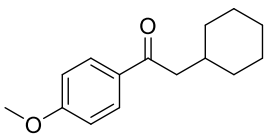
In a nitrogen-filled glovebox, an oven-dried 8.0 mL vial with a stirring bar was added with vinyl azides (**1**) (0.3 mmol, 1.0 equiv.), Katritzky Salts (**4**) (0.45 mmol, 1.5 equiv), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (0.45 mmol, 1.5 equiv). A mixture of *N, N*-Dimethylacetamide (DMA) (3 mL) and H_2O (0.45 mmol, 1.5 eq) were added. The resulting mixture was stirred at room temperature under blue LEDS (456 nm, 100% light intensity) irradiation for 12 h. After this time, the reaction mixture was diluted with EA and washed with a saturated brine for 5-6 times. The organic layer was separated, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography.

2-Cyclohexyl-1-(naphthalen-2-yl) ethan-1-one (3a)



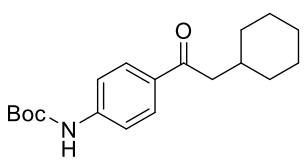
According to Procedure A, the reaction was carried out with **1a** (58.5 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1) to afford 61 mg (80% yield) of **3a** as yellow solid: **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.03 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.93 – 7.86 (m, 2H), 7.64 – 7.52 (m, 2H), 2.96 (d, *J* = 6.8 Hz, 2H), 2.09 – 2.01 (m, 1H), 1.84 – 1.78 (m, 2H), 1.74 – 1.64 (m, 3H), 1.35 – 1.26 (m, 2H), 1.22 – 1.14 (m, 1H), 1.07 (qd, *J* = 12.3, 3.5 Hz, 2H).**^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.26, 135.48, 134.84, 132.54, 129.69, 129.54, 128.35, 128.30, 127.73, 126.67, 124.03, 46.27, 34.75, 33.48, 26.26, 26.16. All data are in accordance with the literature.[27]

2-Cyclohexyl-1-(4-methoxyphenyl) ethan-1-one (3b)



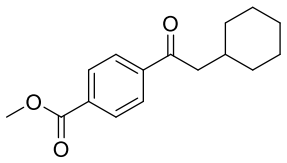
According to Procedure A, the reaction was carried out with **1b** (52 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexane-carboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1) to afford 47 mg (68% yield) of **3b** as colourless oil:**^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.27 (d, *J* = 8.9 Hz, 2H), 6.26 (d, *J* = 8.9 Hz, 2H), 3.20 (s, 3H), 2.10 (d, *J* = 6.9 Hz, 2H), 1.38 – 1.22 (m, 1H), 1.14 – 0.93 (m, 5H), 0.72 – 0.45 (m, 3H), 0.34 (qd, *J* = 12.3, 2.9 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.90, 163.27, 130.58, 130.39, 113.61, 55.40, 45.88, 34.78, 33.44, 26.24, 26.13. All data are in accordance with the literature.[27]

Tert-butyl (4-(2-cyclohexylacetyl) phenyl) carbamate (3c)



According to Procedure A, the reaction was carried out with **1c** (78 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1) to afford 53 mg (56% yield) of **3c** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 6.94 (s, 1H), 2.76 (d, *J* = 6.8 Hz, 2H), 1.99-1.88 (m, 1H), 1.78 – 1.61 (m, 5H), 1.51 (s, 9H), 1.35 – 1.14 (m, 3H), 0.99 (qd, *J* = 12.2, 2.9 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.12, 152.23, 142.77, 132.04, 129.62, 117.42, 81.12, 45.90, 34.76, 33.40, 28.21, 26.21, 26.11. **HRMS m/z (ESI)** calcd for $C_{19}H_{27}NO_3$ $(M + H)^+$ 318.2064, found 318.2060.

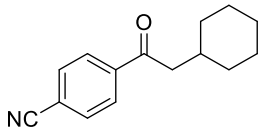
Methyl 4-(2-cyclohexylacetyl) benzoate (3d)



According to Procedure A, the reaction was carried out with **1d** (61 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2)** (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1) to afford 52 mg (66% yield) of

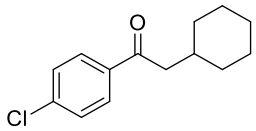
3d as yellow solid: **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.11 (d, *J* = 8.6 Hz, 2H), 7.98 (d, *J* = 8.5 Hz, 2H), 3.94 (s, 3H), 2.84 (d, *J* = 6.8 Hz, 2H), 2.02 – 1.92 (m, 1H), 1.79 – 1.65 (m, 5H), 1.31 – 1.24 (m, 2H), 1.20 – 1.12 (m, 1H), 1.05 – 0.96 (m, 2H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 203.56, 166.28, 140.67, 133.64, 129.79, 128.00, 52.42, 46.54, 34.42, 33.38, 26.19, 26.10. **HRMS m/z (ESI)** calcd for $C_{16}H_{20}O_3$ $(M + H)^+$ 261.1485, found 261.1496.

4-(2-Cyclohexylacetyl) benzonitrile (3e)



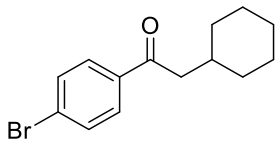
According to Procedure A, the reaction was carried out with **1e** (51 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexane-carboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1) to afford 27 mg (39% yield) of **3e** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.01 (d, *J* = 8.1 Hz, 2H), 7.75 (d, *J* = 8.1 Hz, 2H), 2.82 (d, *J* = 6.7 Hz, 2H), 2.01-1.90 (m, 1H), 1.82 – 1.64 (m, 5H), 1.38 – 1.21 (m, 2H), 1.20 – 1.10 (m, 1H), 1.08 – 0.93 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.73, 140.32, 132.44, 128.48, 117.95, 116.10, 46.39, 34.30, 33.31, 26.10, 26.02. **HRMS m/z (ESI)** calcd for $C_{15}H_{17}NO$ $(M - H)^-$ 227.1310, found 226.1244.

1-(4-Chlorophenyl)-2-cyclohexylethan-1-one (3f)



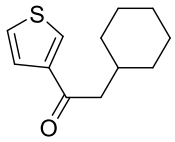
According to Procedure A, the reaction was carried out with **1f** (54 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexane-carboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1) to afford 55 mg (78% yield) of **3f** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93 – 7.84 (m, 2H), 7.47 – 7.38 (m, 2H), 2.78 (d, J = 6.7 Hz, 2H), 2.03 – 1.90 (m, 1H), 1.81 – 1.62 (m, 5H), 1.33 – 1.24 (m, 2H), 1.21 – 1.09 (m, 1H), 1.06 – 0.93 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.98, 139.26, 135.75, 129.55, 128.83, 46.16, 34.53, 33.40, 26.20, 26.11. All data are in accordance with the literature.[27]

1-(4-Bromophenyl)-2-cyclohexylethan-1-one (3g)



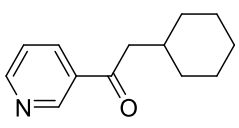
According to Procedure A, the reaction was carried out with **1g** (67 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexane-carboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1) to afford 74 mg (88% yield) of **3g** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.80 (d, *J* = 8.6 Hz, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 2.78 (d, *J* = 6.8 Hz, 2H), 1.99-1.89 (m, 1H), 1.77 – 1.62 (m, 5H), 1.32-1.15 (m, 3H), 1.00 (qd, *J* = 13.7, 13.1, 3.6 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.20, 136.15, 131.83, 129.68, 127.98, 46.15, 34.53, 33.39, 26.20, 26.10. All data are in accordance with the literature.[27]

2-Cyclohexyl-1-(thiophen-3-yl) ethan-1-one (3h)



According to Procedure A, the reaction was carried out with **1h** (45 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 100:1) to afford 41 mg (66% yield) of **3h** as yellow oil: **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.02 (d, *J* = 2.9 Hz, 1H), 7.54 (d, *J* = 5.1 Hz, 1H), 7.33 – 7.29 (m, 1H), 2.72 (d, *J* = 6.8 Hz, 2H), 2.00 – 1.93 (m, 1H), 1.79 – 1.74 (m, 2H), 1.73 – 1.64 (m, 3H), 1.33 – 1.26 (m, 2H), 1.20 – 1.13 (m, 1H), 1.05 – 0.98 (m, 2H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 194.55, 142.97, 131.76, 127.03, 126.16, 47.60, 34.66, 33.42, 26.21, 26.10. **HRMS m/z (ESI)** calcd for $C_{12}H_{16}OS$ $(M + H)^+$ 209.0995, found 209.1005.

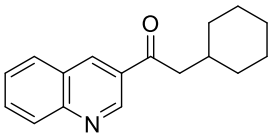
2-Cyclohexyl-1-(pyridin-3-yl) ethan-1-one (3i)



According to Procedure A, the reaction was carried out with **1i** (44 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarb-oxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10:1+2%Et_3N) to afford 39 mg (64% yield) of **3i** as colorless oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.13 (d, *J* = 2.3 Hz, 1H), 8.75 (dd, *J* = 4.8, 1.7 Hz, 1H), 8.20 (dt, *J* =

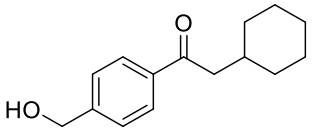
8.0, 2.0 Hz, 1H), 7.39 (dd, *J* = 8.0, 4.8 Hz, 1H), 2.82 (d, J = 6.8 Hz, 2H), 2.02-1.91(m, 1H), 1.85 – 1.62 (m, 5H), 1.35 – 1.11 (m, 3H), 1.01(qd, J = 12.0, 2.8 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.94, 153.27, 149.69, 135.35, 132.53, 123.57, 46.43, 34.32, 33.31, 26.11, 26.03.

2-Cyclohexyl-1-(quinolin-3-yl) ethan-1-one (3j)



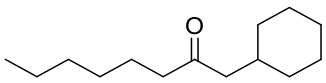
According to Procedure A, the reaction was carried out with **1j** (59 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexane-carboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 10:1) to afford 29 mg (38% yield) of **3j** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.42 (d, *J* = 2.2 Hz, 1H), 8.70 (d, *J* = 2.2 Hz, 1H), 8.16 (d, *J* = 8.5 Hz, 1H), 7.95 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.83 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.73 – 7.52 (m, 1H), 2.95 (d, *J* = 6.8 Hz, 2H), 2.09-1.98 (m, 1H), 1.84 – 1.66 (m, 5H), 1.36 – 1.18 (m, 3H), 1.07 (qd, *J* = 12.3, 3.2 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.99, 149.68, 149.24, 137.02, 131.87, 129.57, 129.39, 129.34, 127.49, 126.90, 46.54, 34.53, 33.41, 26.16, 26.09. All data are in accordance with the literature.[28]

2-Cyclohexyl-1-(4-(hydroxymethyl) phenyl) ethan-1-one (3k)



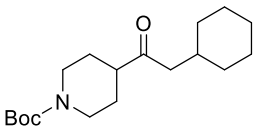
According to Procedure A, the reaction was carried out with **1k** (53 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 5:1+5%Et_3N) to afford 32 mg (46% yield) of **3k** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 4.76 (s, 2H), 2.80 (d, *J* = 6.8 Hz, 2H), 2.03-1.90 (m, 2H), 1.85 – 1.61 (m, 5H), 1.30 – 1.14 (m, 3H), 1.01 (qd, *J* = 12.2, 2.8 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.09, 145.91, 136.67, 128.44, 126.61, 64.61, 46.25, 34.63, 33.40, 26.21, 26.12. **HRMS m/z (ESI)** calcd for $C_{15}H_{20}O_2$ $(M + H)^+$ 233.1536, found 233.1546.

1-Cyclohexyloctan-2-one (3l)



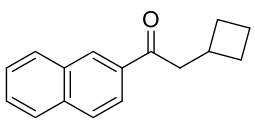
According to Procedure A, the reaction was carried out with **1l** (46 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1) to afford 28 mg (44% yield) of **3l** as colourless oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 2.35 (t, *J* = 7.5 Hz, 2H), 2.25 (d, *J* = 6.9 Hz, 2H), 1.85-1.76 (m, 1H), 1.69-1.61 (m, 5H), 1.57-1.50 (m, 2H), 1.31-1.21 (m, 8H), 1.00 – 0.79 (m, 5H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 211.33, 50.51, 43.51, 33.93, 33.24, 31.59, 28.90, 26.20, 26.08, 23.75, 22.47, 13.99. **HRMS m/z (ESI)** calcd for $C_{14}H_{26}O$ $(M + H)^+$ 211.2056, found 211.2064.

Tert-butyl 4-(2-cyclohexylacetyl) piperidine-1-carboxylate (3m)



According to Procedure A, the reaction was carried out with **1m** (76 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1+5%Et_3N) to afford 39 mg (42% yield) of **3m** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.08 (s, 2H), 2.75 (t, *J* = 12.7 Hz, 2H), 2.45-2.37 (m, 1H), 2.30 (d, *J* = 6.9 Hz, 2H), 1.92 – 1.70 (m, 3H), 1.71 – 1.58 (m, 5H), 1.56 – 1.44 (m, 2H), 1.44 (s, 9H), 1.34 – 1.21 (m, 3H), 0.89 (qd, *J* = 12.9, 12.1, 3.8 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 211.99, 154.64, 79.53, 48.98, 48.32, 43.79, 33.63, 33.27, 28.40, 27.36, 26.18, 26.05, 9.56. **HRMS m/z (ESI)** calcd for $C_{18}H_{31}NO_3$ $(M + Na)^+$ 332.2196, found 332.2188.

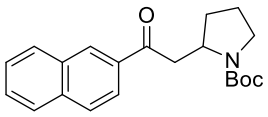
2-Cyclobutyl-1-(naphthalen-2-yl) ethan-1-one (3n)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2b** (110 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 46 mg (68% yield) of **3n** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.05

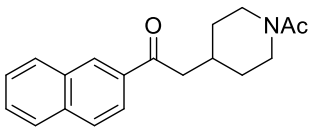
– 7.94 (m, 3H), 7.88 (dd, *J* = 8.4, 5.2 Hz, 3H), 7.64 – 7.51 (m, 2H), 3.22 (d, *J* = 7.3 Hz, 3H), 2.91 (p, *J* = 7.8 Hz, 1H), 2.28 – 2.15 (m, 2H), 1.98 – 1.86 (m, 2H), 1.85 – 1.72 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.62, 135.49, 134.50, 132.54, 129.64, 129.49, 128.33, 128.28, 127.72, 126.66, 123.90, 45.66, 32.15, 28.66, 18.89. **HRMS m/z (ESI)** calcd for $C_{16}H_{16}O$ $(M + H)^+$ 225.1274, found 225.1279.

Tert-butyl 2-(2-(naphthalen-2-yl)-2-oxoethyl) pyrrolidine-1-carboxylate (3o)



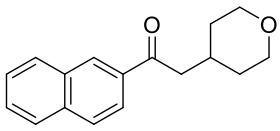
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2c** (162 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =2:1) to afford 24 mg (24% yield) of **3o** as white solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.54 (s, 1H), 8.04 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.96 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.91 – 7.79 (m, 2H), 7.63 – 7.44 (m, 2H), 4.48 – 4.31 (m, 1H), 3.76 (s, 1H), 3.39 (t, *J* = 6.5 Hz, 2H), 2.98 (dd, *J* = 15.5, 9.8 Hz, 1H), 2.17 – 2.01 (m, 1H), 1.97 – 1.73 (m, 3H), 1.47 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.72, 154.30, 135.51, 134.19, 132.48, 129.95, 129.54, 128.34, 128.32, 127.63, 126.61, 123.79, 79.31, 54.32, 46.40, 43.41, 30.87, 28.48, 23.19. All data are in accordance with the literature.[29]

2-(1-Acetylpiperidin-4-yl)-1-(naphthalen-2-yl) ethan-1-one (3p)



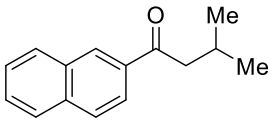
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2d** (142 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =2:1) to afford 50 mg (56% yield) of **3p** as white solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.43 (s, 1H), 8.04 – 7.91 (m, 2H), 7.87 (t, *J* = 8.0 Hz, 2H), 7.68 – 7.42 (m, 2H), 4.71 – 4.54 (m, 1H), 3.88 – 3.49 (m, 1H), 3.16 – 2.93 (m, 3H), 2.65 – 2.52 (m, 1H), 2.42 – 2.19 (m, 1H), 2.07 (s, 3H), 1.97 – 1.72 (m, 2H), 1.31 – 1.19 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.80, 168.69, 135.53, 134.36, 132.42, 129.62, 129.47, 128.47, 127.70, 126.78, 123.67, 46.49, 44.73, 41.64, 32.65, 32.40, 31.92, 21.40. **HRMS m/z (ESI)** calcd for $C_{19}H_{21}NO_2$ $(M + Na)^+$ 318.1465, found 318.1472.

1-(Naphthalen-2-yl)-2-(tetrahydro-2H-pyran-4-yl) ethan-1-one (3q)



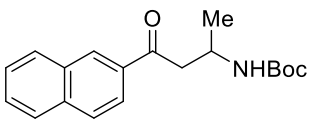
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2e** (124 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 55 mg (72% yield) of **3q** as white solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.05 – 7.94 (m, 2H), 7.89 (t, *J* = 7.9 Hz, 2H), 7.70 – 7.50 (m, 2H), 3.97 (dd, *J* = 10.7, 3.6 Hz, 2H), 3.46 (td, *J* = 11.8, 2.1 Hz, 2H), 3.03 (d, *J* = 6.7 Hz, 2H), 2.45 – 2.23 (m, 1H), 1.76 – 1.70 (m, 2H), 1.53 – 1.35 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.14, 135.56, 134.57, 132.50, 129.67, 129.53, 128.47, 128.45, 127.75, 126.78, 123.80, 67.87, 45.40, 33.10, 31.54. All data are in accordance with the literature.[30]

3-Methyl-1-(naphthalen-2-yl) butan-1-one (3r)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2f** (105 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 47 mg (74% yield) of **3r** as yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (d, *J* = 1.8 Hz, 1H), 8.07 – 7.93 (m, 2H), 7.88 (t, *J* = 7.7 Hz, 2H), 7.64 – 7.50 (m, 2H), 2.97 (d, *J* = 6.9 Hz, 2H), 2.52 – 2.30 (m, 1H), 1.05 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.14, 135.45, 134.73, 132.52, 129.61, 129.49, 128.33, 128.27, 127.70, 126.65, 123.95, 47.51, 25.29, 22.78. All data are in accordance with the literature.[31]

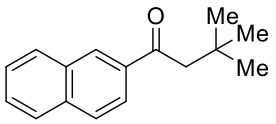
Tert-butyl (4-(naphthalen-2-yl)-4-oxobutan-2-yl) carbamate (3s)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2g** (150.4 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 38 mg (40% yield) of **3s** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.49 (s, 1H), 8.02 (d, *J* = 8.6 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.88 (t, *J* =

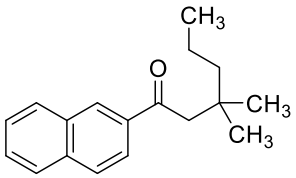
8.2 Hz, 2H), 7.66 – 7.47 (m, 2H), 5.08 (s, 1H), 4.24 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.47 (dd, *J* = 16.1, 4.6 Hz, 1H), 3.17 (dd, *J* = 16.2, 6.7 Hz, 1H), 1.43 (s, 9H), 1.31 (d, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.81, 155.20, 135.63, 134.36, 132.50, 129.98, 129.63, 128.52, 128.48, 127.73, 126.77, 123.71, 79.25, 44.53, 44.03, 28.38, 20.54. **HRMS m/z (ESI)** calcd for $C_{19}H_{23}NO_3$ $(M + Na)^+$ 336.1570, found 336.1577.

3,3-Dimethyl-1-(naphthalen-2-yl) butan-1-one (3t)



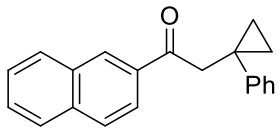
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2h** (111 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 37 mg (54% yield) of **3t** as white solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.45 (s, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, J = 8.0 Hz, 1H), 7.92 – 7.85 (m, 2H), 7.63 – 7.50 (m, 2H), 3.00 (s, 2H), 1.12 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.36, 135.92, 135.37, 132.50, 129.79, 129.54, 128.30, 128.25, 127.68, 126.64, 124.09, 50.11, 31.52, 30.13. **HRMS m/z (ESI)** calcd for $C_{16}H_{18}O$ $(M + H)^+$ 227.1430, found 227.1435.

3,3-Dimethyl-1-(naphthalen-2-yl) hexan-1-one (3u)



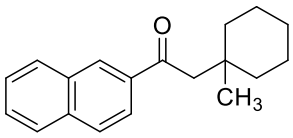
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2i** (124 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydroyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mpmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 40 mg (52% yield) of **3u** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.44 (s, 1H), 8.02 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 7.9 Hz, 1H), 7.88 (dd, *J* = 8.4, 5.5 Hz, 2H), 7.63 – 7.50 (m, 2H), 2.99 (s, 2H), 1.46 – 1.33 (m, 4H), 1.06 (s, 6H), 0.91 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.56, 136.08, 135.35, 132.50, 129.67, 129.54, 128.29, 128.24, 127.69, 126.63, 124.07, 48.19, 45.20, 34.18, 27.73, 17.42, 14.83. **HRMS m/z (ESI)** calcd for $C_{18}H_{22}O$ $(M + H)^+$ 255.1743, found 255.1756.

1-(Naphthalen-2-yl)-2-(1-phenylcyclopropyl) ethan-1-one (3v)



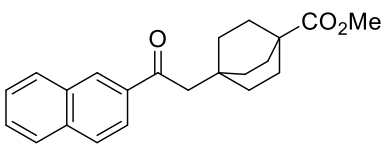
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2j** (138 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =100:1-PE) to afford 56 mg (65% yield) of **3v** as yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.27 (d, *J* = 1.7 Hz, 1H), 7.94 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.92 – 7.79 (m, 3H), 7.67 – 7.48 (m, 2H), 7.39 (d, *J* = 7.4 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.13 (t, *J* = 7.3 Hz, 1H), 3.45 (s, 2H), 1.09 – 1.01 (m, 2H), 1.03 – 0.94 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.52, 144.84, 135.39, 134.62, 132.39, 129.84, 129.50, 128.70, 128.30, 128.23, 128.14, 127.67, 126.61, 126.11, 123.86, 48.23, 22.33, 13.45. **HRMS m/z (ESI)** calcd for $C_{21}H_{18}O$ $(M + Na)^+$ 309.1250, found 309.1264.

2-(1-Methylcyclohexyl)-1-(naphthalen-2-yl) ethan-1-one (3w)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2k** (129 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =100:1-PE) to afford 34 mg (43% yield) of **3w** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.45 (d, *J* = 1.8 Hz, 1H), 8.03 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.97 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.92 – 7.84 (m, 2H), 7.65 – 7.50 (m, 2H), 3.02 (s, 2H), 1.59 – 1.36 (m, 10H), 1.07 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.77, 136.34, 135.34, 132.50, 129.74, 129.55, 128.27, 128.22, 127.68, 126.62, 124.11, 48.69, 38.34, 34.39, 26.19, 25.30, 22.04. **HRMS m/z (ESI)** calcd for $C_{19}H_{22}O$ $(M + H)^+$ 267.1743, found 267.1749.

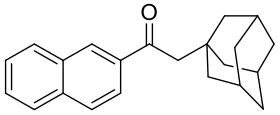
Methyl 4-(2-(naphthalen-2-yl)-2-oxoethyl) bicycle [2.2.2] octane-1-carboxylate (3x)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2l** (161 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 54 mg (54 % yield)

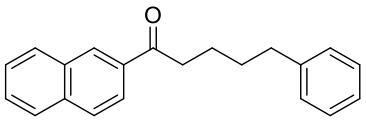
of **3x** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.41 (s, 1H), 7.99 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.96 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.90 – 7.82 (m, 2H), 7.63 – 7.50 (m, 2H), 3.62 (s, 3H), 2.90 (s, 2H), 1.89 – 1.72 (m, 6H), 1.72 – 1.58 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.82, 178.18, 135.80, 135.40, 132.44, 129.86, 129.53, 128.35, 127.67, 126.69, 123.97, 51.54, 47.91, 38.54, 31.66, 30.82, 28.40. **HRMS m/z (ESI)** calcd for $C_{22}H_{24}O_3$ $(M + H)^+$ 337.1798, found 337.1816.

2-((1S,3R,5S)-Adamantan-1-yl)-1-(naphthalen-2-yl) ethan-1-one (3y)



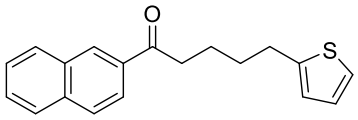
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2m** (146 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 53 mg (58% yield) of **3y** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.04 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 7.92 – 7.83 (m, 2H), 7.66 – 7.50 (m, 2H), 2.85 (s, 2H), 2.03 – 1.90 (m, 3H), 1.75 – 1.63 (m, 12H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 201.17, 137.18, 136.35, 133.44, 131.03, 130.57, 129.24, 128.65, 127.59, 125.16, 52.27, 44.01, 37.69, 35.02, 29.82, 29.67. **HRMS m/z (ESI)** calcd for $C_{22}H_{24}O$ $(M + H)^+$ 305.1900, found 305.1908.

1-(Naphthalen-2-yl)-5-phenylpentan-1-one (3z)



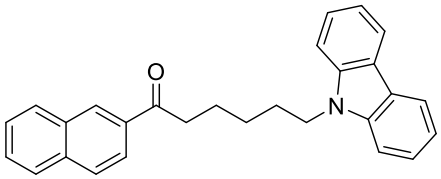
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2n** (139 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 51 mg (59% yield) of **3z** as yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.08 – 8.00 (m, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.93 – 7.85 (m, 2H), 7.65 – 7.52 (m, 2H), 7.32-7.28 (m, 2H), 7.23-7.18 (m, 3H), 3.13 (t, *J* = 7.2 Hz, 2H), 2.71 (t, *J* = 7.5 Hz, 2H), 1.91-1.84 (m, 2H), 1.82-1.74 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.21, 142.23, 135.50, 134.34, 132.52, 129.58, 129.51, 128.39, 128.33, 128.29, 127.74, 126.69, 125.73, 123.90, 38.46, 35.79, 31.10, 24.12. All data are in accordance with the literature.[32]

1-(Naphthalen-2-yl)-5-(thiophen-2-yl) pentan-1-one (3aa)



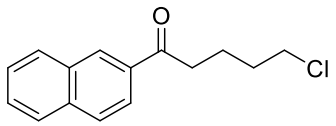
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2o** (142 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 25 mg (28% yield) of **3aa** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (d, J = 1.7 Hz, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 8.00 – 7.94 (m, 1H), 7.94 – 7.84 (m, 2H), 7.66 – 7.52 (m, 2H), 7.12 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.92 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.86 – 6.79 (m, 1H), 3.14 (t, *J* = 7.0 Hz, 2H), 2.92 (t, *J* = 6.9 Hz, 2H), 1.96 – 1.78 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.02, 145.07, 135.52, 134.31, 132.53, 129.59, 129.52, 128.40, 128.35, 127.74, 126.71, 126.69, 124.15, 123.88, 122.91, 38.29, 31.40, 29.78, 23.86. **HRMS m/z (ESI)** calculated for $C_{19}H_{18}OS$ $(M+H)^+$ 295.1151, found 295.1159.

6-(9H-Carbazol-9-yl)-1-(naphthalen-2-yl) hexan-1-one (3ab)



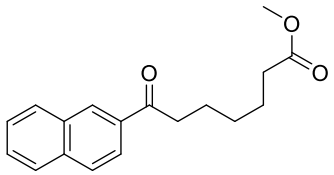
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2p** (186 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 54 mg (46% yield) of **3ab** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.42 (s, 1H), 8.13 (d, *J* = 7.8 Hz, 2H), 8.01 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 – 7.92 (m, 1H), 7.91 – 7.84 (m, 2H), 7.65 – 7.51 (m, 2H), 7.53 – 7.38 (m, 4H), 7.30 – 7.20 (m, 2H), 4.34 (t, *J* = 7.1 Hz, 2H), 3.04 (t, *J* = 7.3 Hz, 2H), 1.98 (p, *J* = 7.3 Hz, 2H), 1.85 (p, *J* = 7.4 Hz, 2H), 1.57 – 1.45 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.99, 140.36, 135.50, 134.21, 132.48, 129.56, 129.51, 128.38, 128.35, 127.71, 126.69, 125.59, 123.80, 122.79, 120.31, 118.73, 108.61, 42.81, 38.29, 28.92, 27.01, 24.10. **HRMS m/z (ESI)** calculated for $C_{28}H_{25}NO$ $(M+H)^+$ 392.2009, found 392.2028.

5-Chloro-1-(naphthalen-2-yl) pentan-1-one (3ac)



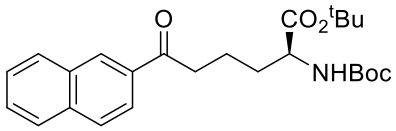
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2q** (120 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =20:1) to afford 24 mg (32% yield) of **3ac** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.47 (d, J = 1.8 Hz, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.89 (t, *J* = 8.0 Hz, 2H), 7.66 – 7.52 (m, 2H), 3.61 (t, *J* = 6.1 Hz, 2H), 3.15 (t, *J* = 6.7 Hz, 2H), 2.06 – 1.86 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.55, 135.57, 134.17, 132.52, 129.61, 129.52, 128.47, 128.43, 127.76, 126.77, 123.80, 44.70, 37.60, 32.07, 21.65. All data are in accordance with the literature.[33]

Methyl 7-(naphthalen-2-yl)-7-oxoheptanoate (3ad)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2r** (137 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =20:1) to afford 35 mg (41% yield) of **3ad** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.02 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.93 – 7.82 (m, 2H), 7.65 – 7.50 (m, 2H), 3.67 (s, 3H), 3.11 (t, *J* = 7.3 Hz, 2H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.82 (p, *J* = 7.4 Hz, 2H), 1.71 (p, *J* = 7.5 Hz, 2H), 1.56 – 1.37 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.09, 174.08, 135.52, 134.34, 132.54, 129.57, 129.51, 128.40, 128.34, 127.74, 126.71, 123.88, 51.45, 38.33, 33.88, 28.82, 24.76, 23.99. **HRMS m/z (ESI)** calculated for $C_{18}H_{20}O_3$ $(M+Na)^+$ 307.1305, found 307.1312.

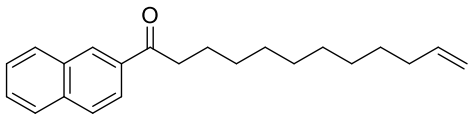
Tert-butyl(S)-2-((tert-butoxycarbonyl) amino)-6-(naphthalen-2-yl)-6 oxohexanoate (3ae)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2s** (202 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol)

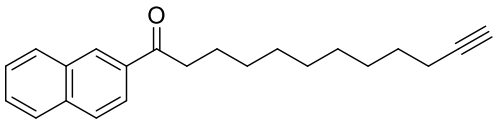
in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 58 mg (45% yield) of **3ae** as colorless oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.45 (s, 1H), 8.01 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.89 – 7.83 (m, 2H), 7.60 – 7.51 (m, 2H), 5.14 (d, *J* = 8.3 Hz, 1H), 4.34 – 4.18 (m, 1H), 3.18 – 3.08 (m, 2H), 1.99 – 1.81 (m, 4H), 1.45 (s, 9H), 1.44 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.47, 171.73, 155.39, 135.50, 134.17, 132.48, 129.57, 129.48, 128.37, 128.34, 127.68, 126.68, 123.76, 81.83, 79.55, 53.70, 37.80, 32.34, 28.27, 27.93, 19.88. **HRMS m/z (ESI)** calculated for $C_{25}H_{33}NO_5$ $(M+Na)^+$ 450.2251, found 450.2266.

1-(Naphthalen-2-yl) dodec-11-en-1-one (3af)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2t** (148 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1-10:1) to afford 50 mg (54% yield) of **3af** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.47 (s, 1H), 8.08 – 8.01 (m, 1H), 7.97 (d, J = 8.0 Hz, 1H), 7.94 – 7.85 (m, 2H), 7.65 – 7.51 (m, 2H), 5.92 – 5.73 (m, 1H), 5.07 – 4.88 (m, 2H), 3.10 (t, J = 7.4 Hz, 2H), 2.04 (q, J = 7.1 Hz, 2H), 1.80 (p, J = 7.5 Hz, 2H), 1.46 – 1.25 (m, 12H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.55, 139.20, 135.48, 134.41, 132.54, 129.57, 129.50, 128.35, 128.28, 127.73, 126.67, 123.95, 114.09, 38.68, 33.77, 29.46, 29.42, 29.42, 29.39, 29.09, 28.90, 24.52. **HRMS m/z (ESI)** calculated for $C_{22}H_{28}O$ $(M+H)^+$ 309.2213, found 309.2220.

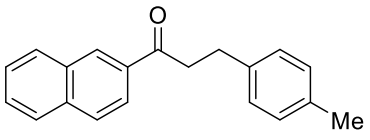
1-(Naphthalen-2-yl) dodec-11-yn-1-one (3ag)



According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2u** (147 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1-10:1) to afford 42 mg (46% yield) of **3ag** as yellow solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.52 – 8.44 (m, 1H), 8.04 (dd, *J* = 8.6, 1.7 Hz, 1H), 8.01 – 7.95 (m, 1H), 7.93 – 7.84 (m, 2H), 7.64 – 7.52 (m, 2H), 3.10 (t, *J* =

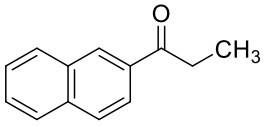
7.4 Hz, 2H), 2.22 – 2.12 (m, 2H), 1.94 (t, *J* = 2.6 Hz, 1H), 1.80 (p, *J* = 7.4 Hz, 2H), 1.56 – 1.47 (m, 2H), 1.46 – 1.28 (m, 10H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.45, 135.47, 134.39, 132.52, 129.54, 129.48, 128.33, 128.27, 127.71, 126.65, 123.92, 84.72, 68.05, 38.63, 29.39, 29.33, 29.30, 29.00, 28.67, 28.42, 24.48, 18.34. **HRMS m/z (ESI)** calculated for $C_{22}H_{26}O$ $(M+H)^+$ 307.2056, found 307.2064.

1-(Naphthalen-2-yl)-3-(p-tolyl) propan-1-one (3ah)



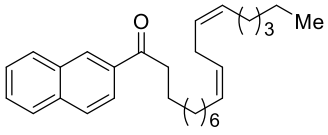
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2v** (133 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =100:1) to afford 43 mg (52% yield) of **3ah** as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.46 (s, 1H), 8.04 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.92 – 7.83 (m, 2H), 7.65 – 7.45 (m, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 3.52 – 3.37 (m, 2H), 3.09 (t, *J* = 7.7 Hz, 2H), 2.33 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.24, 138.23, 135.63, 135.55, 134.21, 132.52, 129.66, 129.51, 129.21, 128.41, 128.38, 128.31, 127.74, 126.72, 123.84, 40.70, 29.86, 20.97. All data are in accordance with the literature.[34]

1-(2-Naphthyl)-1-propanone (3ai)



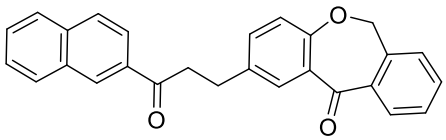
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2w** (92 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: Toluene=10:1-20:1) to afford 23 mg (41% yield) of **3ai** as yellow solid. **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.48 (d, *J* = 1.8 Hz, 1H), 8.05 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.94 – 7.85 (m, 2H), 7.63 – 7.52 (m, 2H), 3.15 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.79, 135.51, 134.25, 132.56, 129.51, 129.49, 128.37, 128.29, 127.75, 126.68, 123.91, 31.85, 8.39. All data are in accordance with the literature.[35]

1-(Naphthalen-2-yl) octadeca-9,12-dien-1-one (3aj)



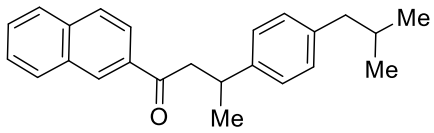
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2x** (182 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 100:1) to afford 61 mg (50% yield) of **3aj** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.47 (s, 1H), 8.04 (d, *J* = 8.6 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.94 – 7.84 (m, 2H), 7.66 – 7.49 (m, 2H), 5.44 – 5.29 (m, 4H), 3.10 (t, *J* = 7.4 Hz, 2H), 2.79 (t, *J* = 6.5 Hz, 2H), 2.12 – 2.01 (m, 4H), 1.80 (p, *J* = 7.4 Hz, 2H), 1.44 – 1.27 (m, 16H), 0.89 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.47, 135.48, 134.41, 132.54, 130.17, 130.10, 129.55, 129.50, 128.35, 128.27, 127.96, 127.91, 127.73, 126.66, 123.94, 38.66, 31.50, 29.64, 29.46, 29.40, 29.32, 29.26, 27.20, 27.18, 25.61, 24.51, 22.55, 14.05. **HRMS m/z (ESI)** calculated for $C_{29}H_{40}O$ $(M + H)^+$ 405.3152, found 405.3171.

2-(3-(Naphthalen-2-yl)-3-oxopropyl) dibenzo[b,e]oxepin-11(6H)-one (3ak)



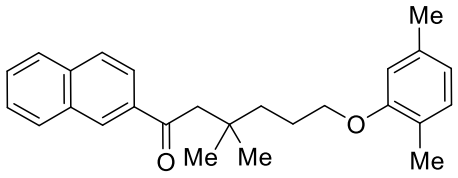
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2y** (186 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 55 mg (47% yield) of **3ak** as yellow oil: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.47 (s, 1H), 8.16 (d, *J* = 2.3 Hz, 1H), 8.04 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.98 – 7.82 (m, 4H), 7.63 – 7.50 (m, 3H), 7.51 – 7.39 (m, 2H), 7.35 (d, *J* = 7.4 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 5.16 (s, 2H), 3.46 (t, *J* = 7.6 Hz, 2H), 3.15 (t, *J* = 7.6 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.88, 191.03, 159.81, 140.44, 136.01, 135.67, 135.55, 134.98, 134.08, 132.66, 132.48, 130.96, 129.70, 129.52, 129.44, 129.15, 128.43, 128.41, 127.71, 126.73, 125.20, 123.78, 120.81, 73.58, 40.18, 29.19. **HRMS m/z (ESI)** calculated for $C_{27}H_{20}O_3$ $(M+H)^+$ 393.1485, found 393.1504.

3-(4-Isobutylphenyl)-1-(naphthalen-2-yl) butan-1-one (3al)



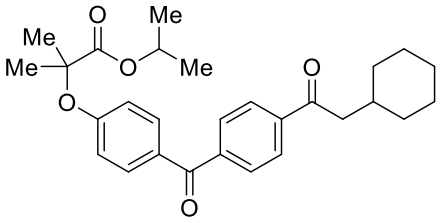
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2z** (158 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 50:1) to afford 62 mg (63% yield) of **3al** as yellow solid: **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.44 (s, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 – 7.92 (m, 1H), 7.87 (dd, *J* = 8.3, 3.3 Hz, 2H), 7.64 – 7.48 (m, 2H), 7.30 – 7.20 (m, 2H), 7.20 – 7.08 (m, 2H), 3.67 – 3.48 (m, 1H), 3.44 (dd, *J* = 16.2, 5.6 Hz, 1H), 3.31 (dd, *J* = 16.2, 8.4 Hz, 1H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.96 – 1.71 (m, 1H), 1.40 (d, *J* = 6.9 Hz, 3H), 0.91 (dd, *J* = 6.6, 0.9 Hz, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 199.16, 143.77, 139.56, 135.46, 134.58, 132.48, 129.67, 129.49, 129.21, 128.32, 127.69, 126.65, 126.53, 123.89, 47.27, 44.99, 35.42, 30.14, 22.35, 21.82. **HRMS m/z (ESI)** calculated for $C_{24}H_{26}O$ $(M+H)^+$ 331.2056, found 331.2071.

6-(2,5-Dimethylphenoxy)-3,3-dimethyl-1-(naphthalen-2-yl) hexan-1-one (3am)



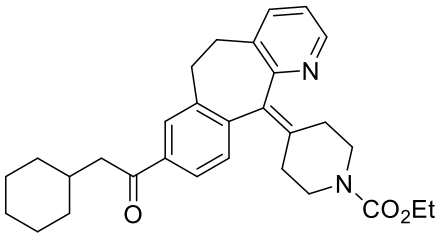
According to Procedure A, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **2aa** (178 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA = 20:1) to afford 63 mg (56% yield) of **3am** as yellow oil: **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.49 (s, 1H), 8.08 (d, *J* = 8.6 Hz, 1H), 8.00 (d, *J* = 7.9 Hz, 1H), 7.91 (t, *J* = 7.9 Hz, 2H), 7.74 – 7.55 (m, 2H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.77 – 6.64 (m, 2H), 3.97 (t, *J* = 6.5 Hz, 2H), 3.08 (s, 2H), 2.35 (s, 3H), 2.24 (s, 3H), 2.01 – 1.82 (m, 2H), 1.77 – 1.62 (m, 2H), 1.18 (s, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 200.13, 156.99, 136.36, 135.95, 135.36, 132.48, 130.22, 129.63, 129.51, 128.32, 128.25, 127.66, 126.64, 123.99, 123.52, 120.60, 112.02, 68.35, 48.02, 38.64, 33.85, 27.66, 24.47, 21.34, 15.73. **HRMS m/z (ESI)** calculated for $C_{26}H_{30}O_2$ $(M+H)^+$ 375.2319, found 375.2337.

Isopropyl 2-(4-(4-(2-cyclohexylacetyl) benzoyl) phenoxy)-2-methylpropanoate (3an)



According to Procedure A, the reaction was carried out with **1n** (118 mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =10:1) to afford 54 mg (40% yield) of **3an** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.11 – 7.96 (m, 2H), 7.86 – 7.70 (m, 4H), 6.93 – 6.83 (m, 2H), 5.09 (p, *J* = 6.2 Hz, 1H), 2.87 (d, *J* = 6.7 Hz, 2H), 2.12 – 1.89 (m, 1H), 1.83 – 1.68 (m, 5H), 1.67 (s, 6H), 1.37 – 1.25 (m, 2H), 1.21 (d, *J* = 6.3 Hz, 6H), 1.19 – 0.98 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.70, 194.63, 172.96, 159.93, 141.72, 139.58, 132.04, 129.89, 129.61, 127.87, 117.18, 79.39, 69.29, 46.46, 34.44, 33.34, 26.15, 26.06, 25.30, 21.45. **HRMS m/z (ESI)** calculated for $C_{28}H_{34}O_5$ $(M+H)^+$ 451.2479, found 451.2502.

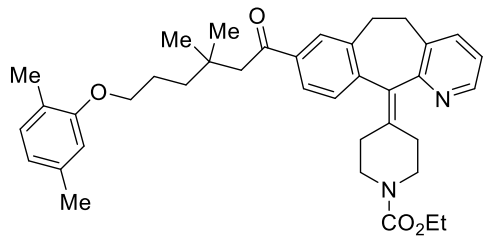
Ethyl 4-(8-(2-cyclohexylacetyl)-5,6-dihydro-11H-benzo [5,6] cyclohepta [1,2-b] pyridin-11-ylidene) piperidine-1-carboperoxoate (3ao)



According to Procedure A, the reaction was carried out with **1o** (129mg, 0.3 mmol), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (123 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =2:1) to afford 60 mg (42% yield) of **3ao** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.48 – 8.30 (m, 1H), 7.80 – 7.65 (m, 2H), 7.53 – 7.37 (m, 1H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.09 (dd, *J* = 7.7, 4.8 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.81 (s, 2H), 3.57 – 3.30 (m, 2H), 3.21 – 3.06 (m, 2H), 3.04 – 2.81 (m, 2H), 2.76 (d, *J* = 6.8 Hz, 2H), 2.62 – 2.45 (m, 1H), 2.45 – 2.25 (m, 3H), 2.00 – 1.86 (m, 1H), 1.77 – 1.66 (m, 3H), 1.29 – 1.20 (m, 6H), 1.21 – 1.05 (m, 2H), 1.04 – 0.91 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.83, 156.52, 155.44, 146.73, 144.16, 138.19, 137.77, 137.60, 136.54, 134.64, 133.46, 129.34, 128.69, 126.11, 122.32, 61.28, 46.11, 44.77, 44.74, 34.50, 33.38, 31.74, 31.61, 30.80, 30.50, 26.19, 26.08, 14.63. **HRMS m/z (ESI)** calculated for $C_{30}H_{36}N_2O_3$

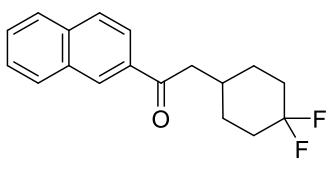
$(M+H)^+$ 473.2799, found 473.2786.

Ethyl 4-(8-(6-(2,5-dimethylphenoxy)-3,3-dimethylhexanoyl)-5,6-dihydro-11H-benzo [5,6] cyclohepta[1,2-b] pyridin-11-ylidene) piperidine-1-carboperoxoate (3ap)



According to Procedure A, the reaction was carried out with **1o** (129mg, 0.3 mmol), **2aa** (178 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMSO (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =2:1) to afford 89 mg (50% yield) of **3ap** as white solid:**^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.38 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.81 – 7.65 (m, 2H), 7.40 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.33 – 7.22 (m, 1H), 7.07 (dd, *J* = 7.7, 4.8 Hz, 1H), 6.95 (d, *J* = 7.4 Hz, 1H), 6.69 – 6.53 (m, 2H), 4.11 (q, *J* = 7.3 Hz, 2H), 3.92 – 3.70 (m, 4H), 3.53 – 3.26 (m, 2H), 3.24 – 3.05 (m, 2H), 2.94 – 2.84 (m, 2H), 2.84 – 2.79 (m, 2H), 2.54 – 2.42 (m, 1H), 2.36 – 2.27 (m, 3H), 2.26 (s, 3H), 2.12 (s, 3H), 1.76 (dq, *J* = 10.4, 6.6 Hz, 2H), 1.60 – 1.50 (m, 2H), 1.25 – 1.21 (m, 3H), 1.04 (s, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 199.78, 156.93, 156.51, 155.42, 146.71, 144.02, 138.16, 137.78, 137.61, 137.59, 136.37, 134.61, 133.46, 130.19, 129.30, 128.66, 126.11, 123.47, 122.32, 120.55, 111.94, 68.29, 61.27, 47.83, 44.74, 38.48, 33.69, 31.76, 31.60, 30.78, 30.49, 27.61, 24.40, 21.33, 15.71, 14.63. **HRMS m/z (ESI)** calculated for $C_{38}H_{46}N_2O_4$ $(M+H)^+$ 595.3530, found 595.3516.

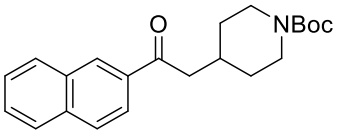
2-(4,4-Difluorocyclohexyl)-1-(naphthalen-2-yl) ethan-1-one (3aq)



According to Procedure B, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **4b** (230 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMA (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =50:1) to afford 52 mg (60% yield) of **3aq** as white solid: **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.46 (d, *J* = 1.7 Hz, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.95 – 7.86 (m, 2H), 7.64 – 7.53 (m, 2H), 3.04 (d, *J* = 6.7 Hz, 2H), 2.25 – 2.07 (m, 3H), 1.96 – 1.73 (m, 4H), 1.50 – 1.35 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.09, 135.60, 134.46, 132.50, 129.69, 129.55, 128.53, 127.78, 126.84, 123.77,123.36, (d, *J*=243.5 Hz), 44.29, 44.27, 33.45(dd, *J*=25.6 Hz), 32.16, 29.09(d, *J*=9.6 Hz). **^{19}F NMR (376 MHz, $CDCl_3$)** δ -91.65 (d, *J*=235.0 Hz), -102.12 (d, *J*= 234.6 Hz). **HRMS m/z (ESI)** calculated for $C_{18}H_{18}F_2O$ $(M+H)^+$ 289.1398, found 289.1392.

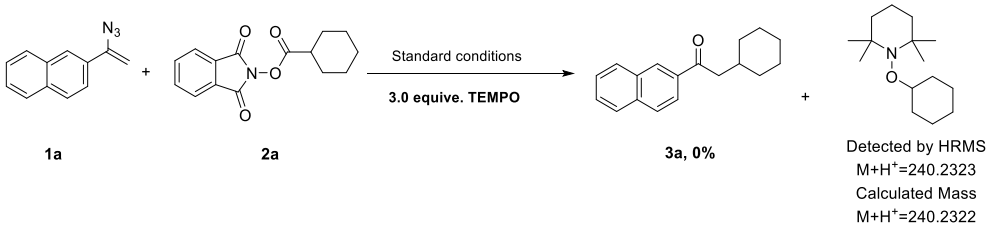
Tert-butyl 4-(2-(naphthalen-2-yl)-2-oxoethyl) piperidine-1-carboxylate (3ar)



According to Procedure B, the reaction was carried out with 2-(1-azidovinyl) naphthalene (58.5 mg, 0.3 mmol), **4d** (260 mg, 0.45 mmol), 2,6-dimethyl-3,5-diacetyl-1,4-dihydropyridine (**ED 2**) (86.8 mg, 0.45 mmol), H_2O (8.1 μL, 0.45 mmol) in DMA (3 mL). The crude product was purified by flash column chromatography on silica gel (PE: EA =5:1) to afford 49 mg (46% yield) of **3ar** as white solid: **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.44 (s, 1H), 8.01 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.88 (t, *J* = 7.8 Hz, 2H), 7.66 – 7.49 (m, 2H), 4.24 – 3.96 (m, 2H), 3.01 (d, *J* = 6.7 Hz, 2H), 2.76 (t, *J* = 12.9 Hz, 2H), 2.28 – 2.13 (m, 1H), 1.85 – 1.69 (m, 2H), 1.46 (s, 9H), 1.31 – 1.22 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.14, 154.81, 135.55, 134.52, 132.48, 129.66, 129.52, 128.46, 127.74, 126.78, 123.78, 79.26, 45.00, 43.85, 32.53, 32.18, 28.42. [36]

8. Mechanistic Studies

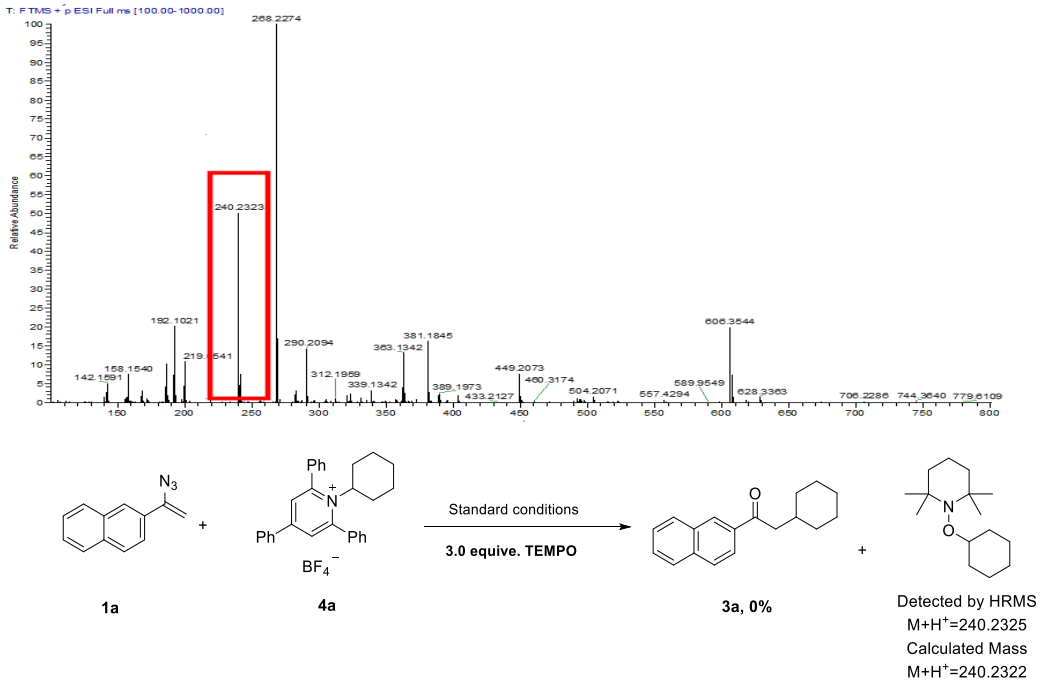
Radical trapping experiment with TEMPO



In a nitrogen-filled glovebox, an over-dried 8.0 mL vial with a stirring bar was added with **1a** (19.5mg, 0.1 mmol), **2a** (41 mg, 0.15 mmol), **ED 2** (28.9 mg, 0.15 mmol) and **TEMPO** (46.8 mg, 0.3 mmol), Dimethyl sulfoxide (**DMSO**) (1.0 mL) and H_2O (2.7 μL, 0.15 mmol) were then added. The resulting mixture was stirred at room temperature under blue LEDS (456 nm, 25% light intensity) irradiation for 12 h. After this time, the reaction mixture was diluted with EA and washed with a saturated brine

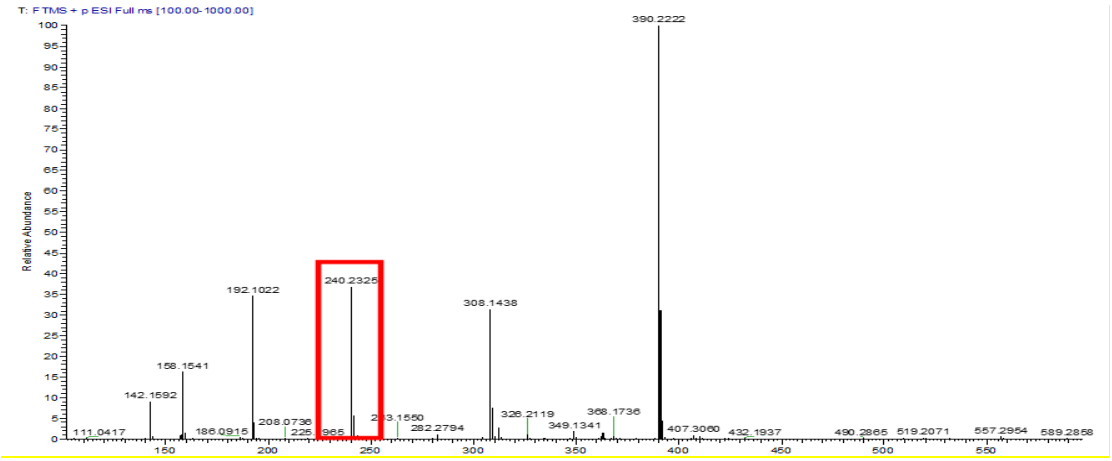
for 5-6 times. The organic layer was separated, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The yield of 3a was 0% as determined by ^1H NMR using CH_2Br_2 as an internal standard. The TEMPO-trapped product was detected by HRMS.

The HRMS spectrum of crude reaction mixture with the TEMPO additive:



In a nitrogen-filled glovebox, an over-dried 8.0 mL vial with a stirring bar was added with **1a** (19.5mg, 0.1 mmol), **4a** (71.5 mg, 0.15 mmol), **ED 2** (28.9 mg, 0.15 mmol) and **TEMPO** (46.8 mg, 0.3 mmol), N, N-Dimethylacetamide (**DMA**) (1.0 mL) and H_2O (2.7 μL, 0.15 mmol) were then added. The resulting mixture was stirred at room temperature under blue LEDS (456 nm, 100% light intensity) irradiation for 12 h. After this time, the reaction mixture was diluted with EA and washed with a saturated brine for 5-6 times. The organic layer was separated, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The yield of 3a was 0% as determined by ^1H NMR using CH_2Br_2 as an internal standard. The TEMPO-trapped product was detected by HRMS.

The HRMS spectrum of crude reaction mixture with the TEMPO additive:



UV/Vis Absorption Spectra

The UV/Vis absorption spectra of Dimethyl sulfoxide (**DMSO**) (path length = 1 cm) solutions of **2a** (0.15 M), **ED 2** (0.15 M) and a mixture of **2a** (0.15 M) and **ED 2** (0.15 M) are shown in **Figure S2**. A bathochromic shift of the mixture observed on the **Figure S2** indicated that an EDA complex is formed between **2a** and **ED 2**.

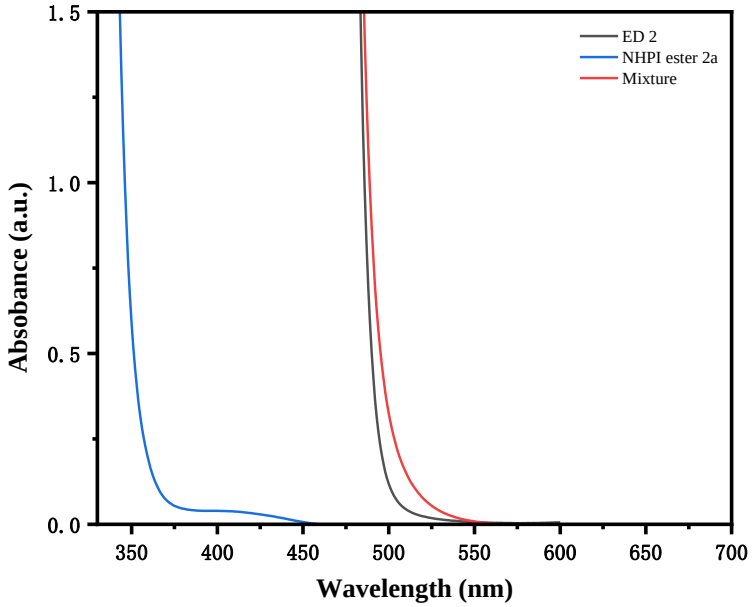


Figure S2. UV/Vis absorption spectra

The UV/Vis absorption spectra of N, N-Dimethylacetamide (**DMA**) (path length = 1 cm) solutions of **4a** (0.15 M), **ED 2** (0.15 M) and a mixture of **4a** (0.15 M) and **ED 2** (0.15 M) are shown in **Figure S3**. A bathochromic shift of the mixture observed on the. **Figure S3** indicated that an EDA complex is formed between **4a** and **ED 2**.

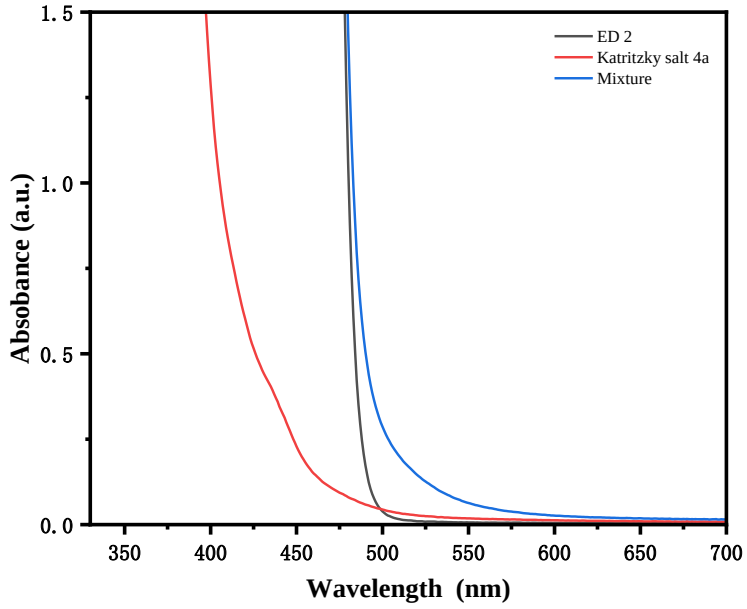


Figure S3 UV/Vis absorption spectra

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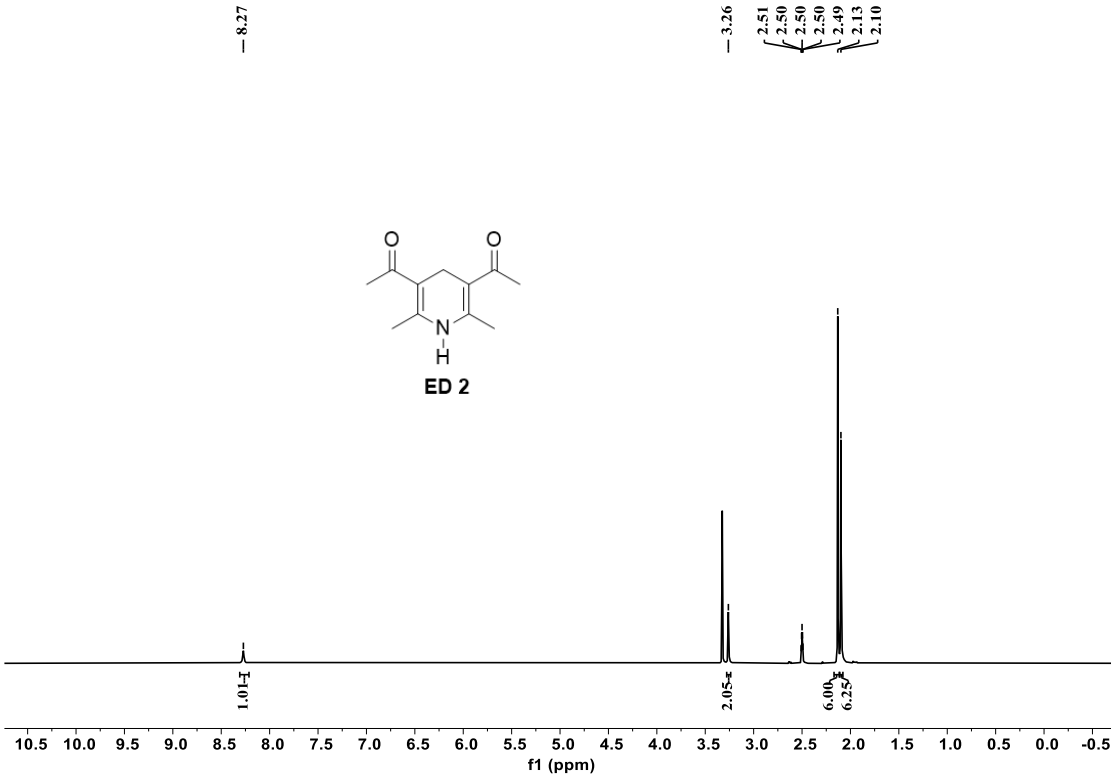
^{1}H NMR (400 MHz, DMSO-d6)



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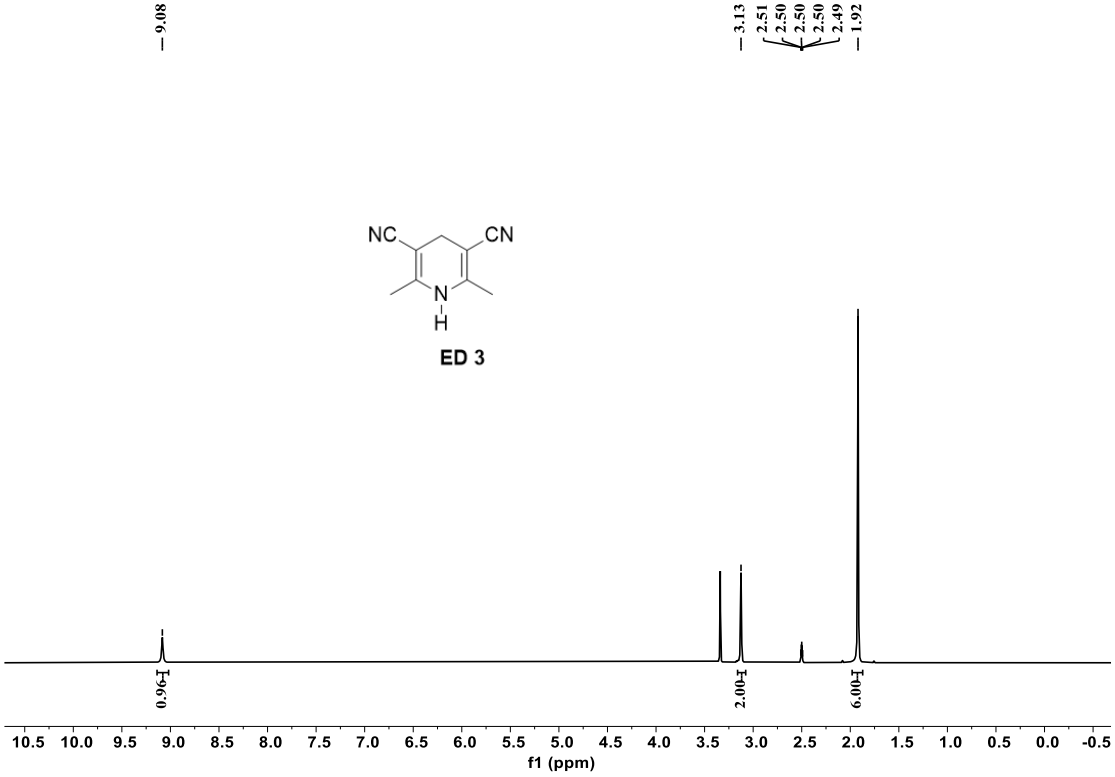
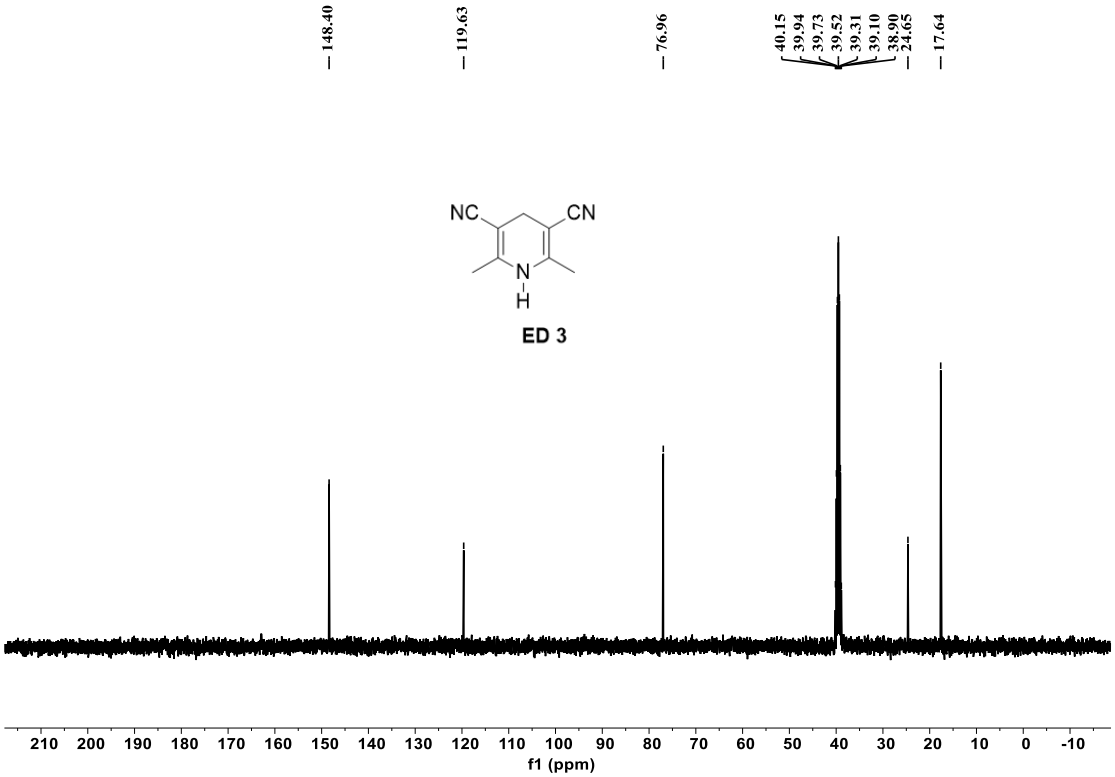


^{13}C NMR (101 MHz, DMSO-d6)



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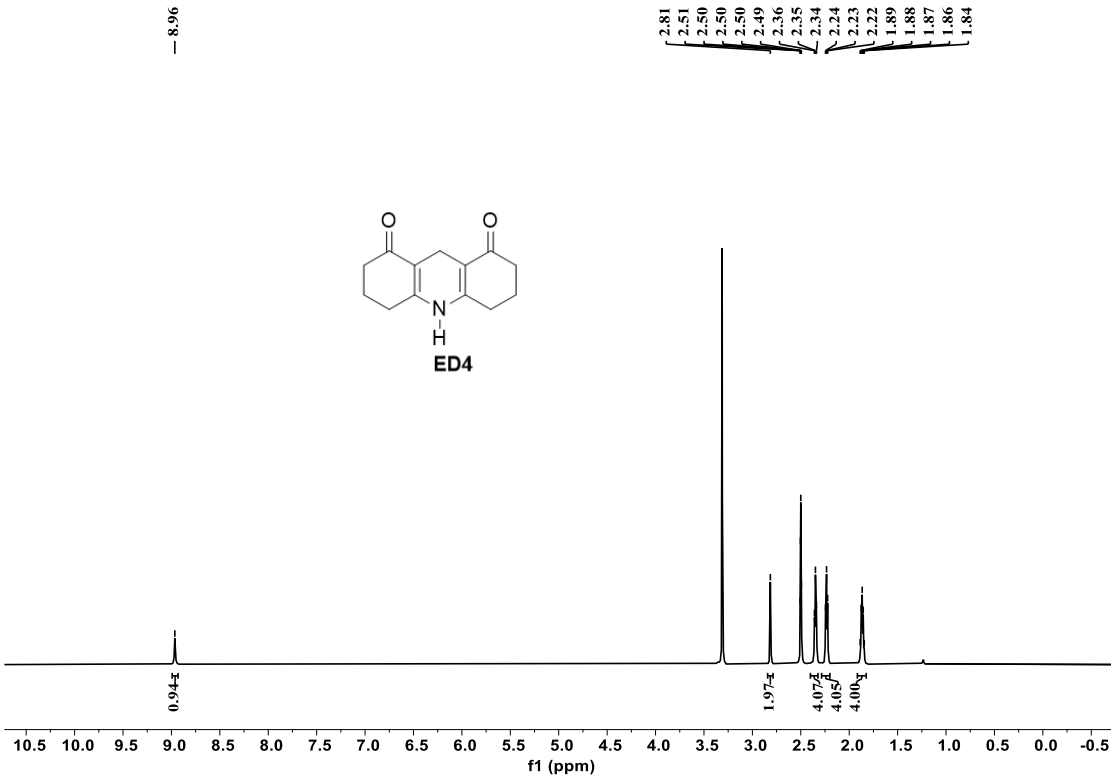

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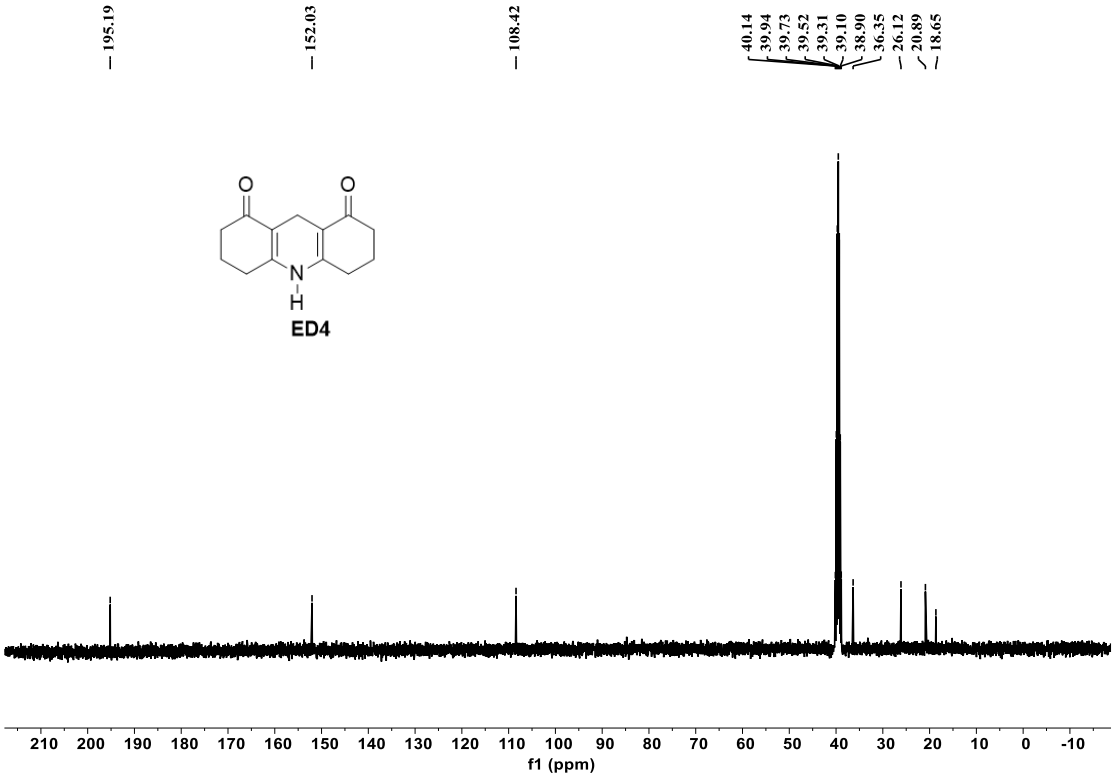

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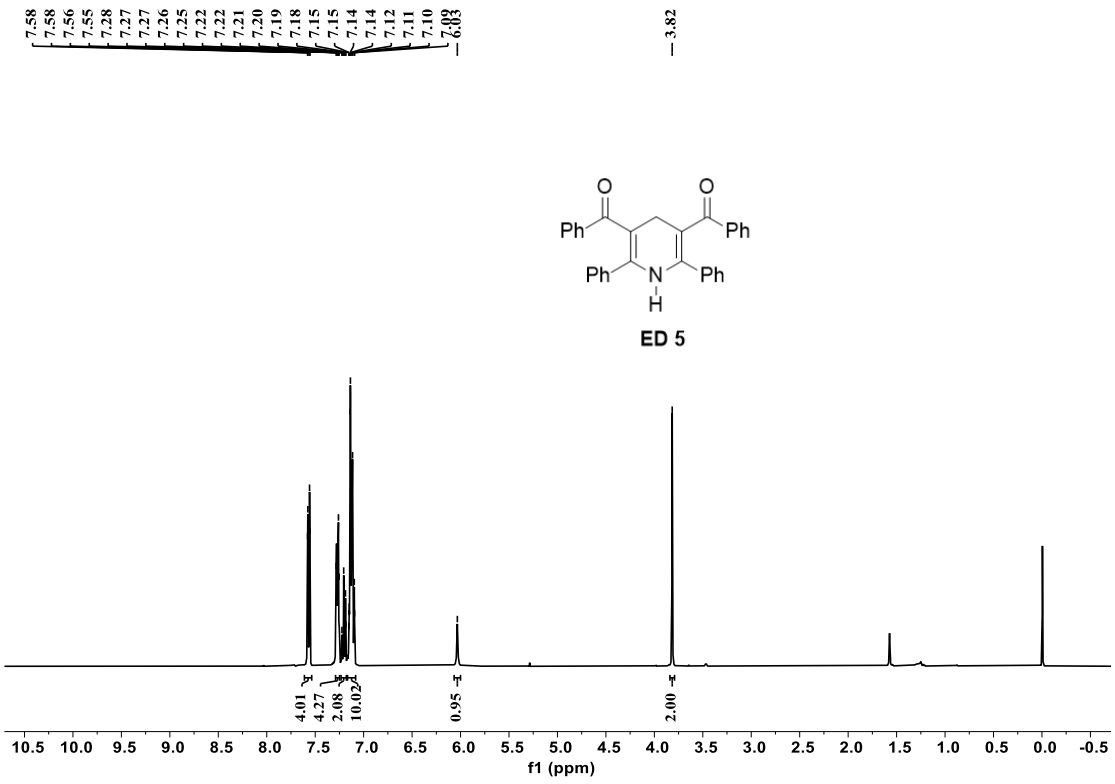

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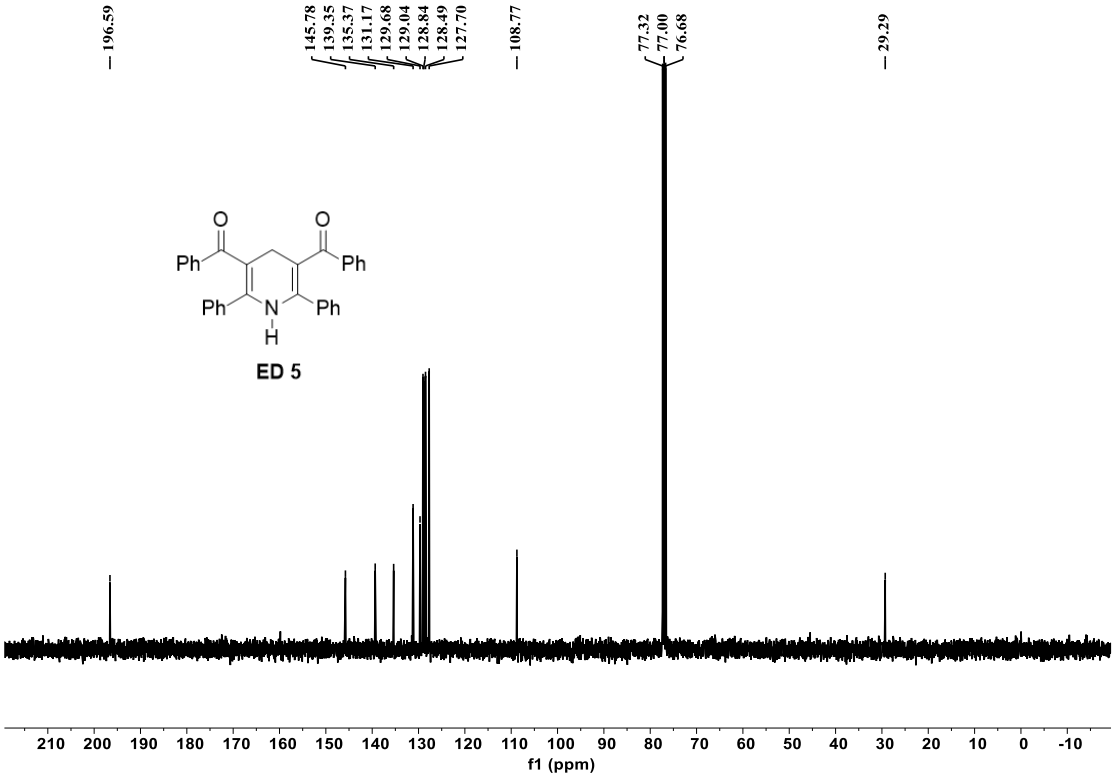

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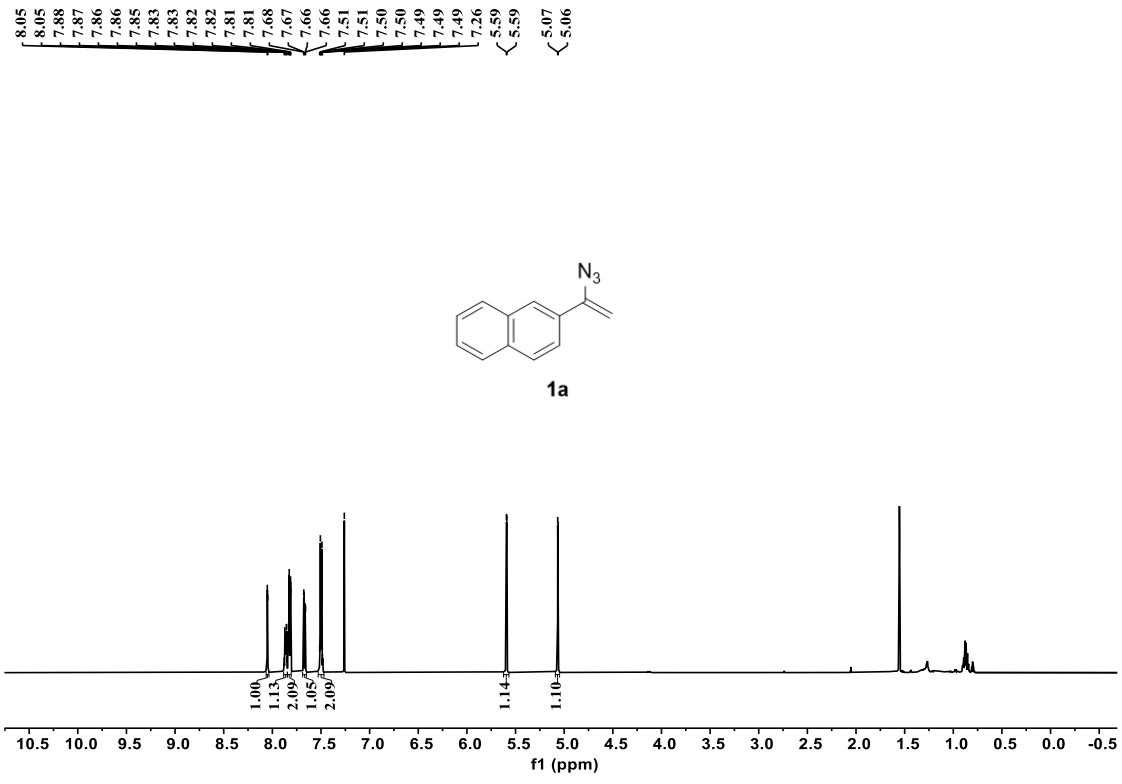

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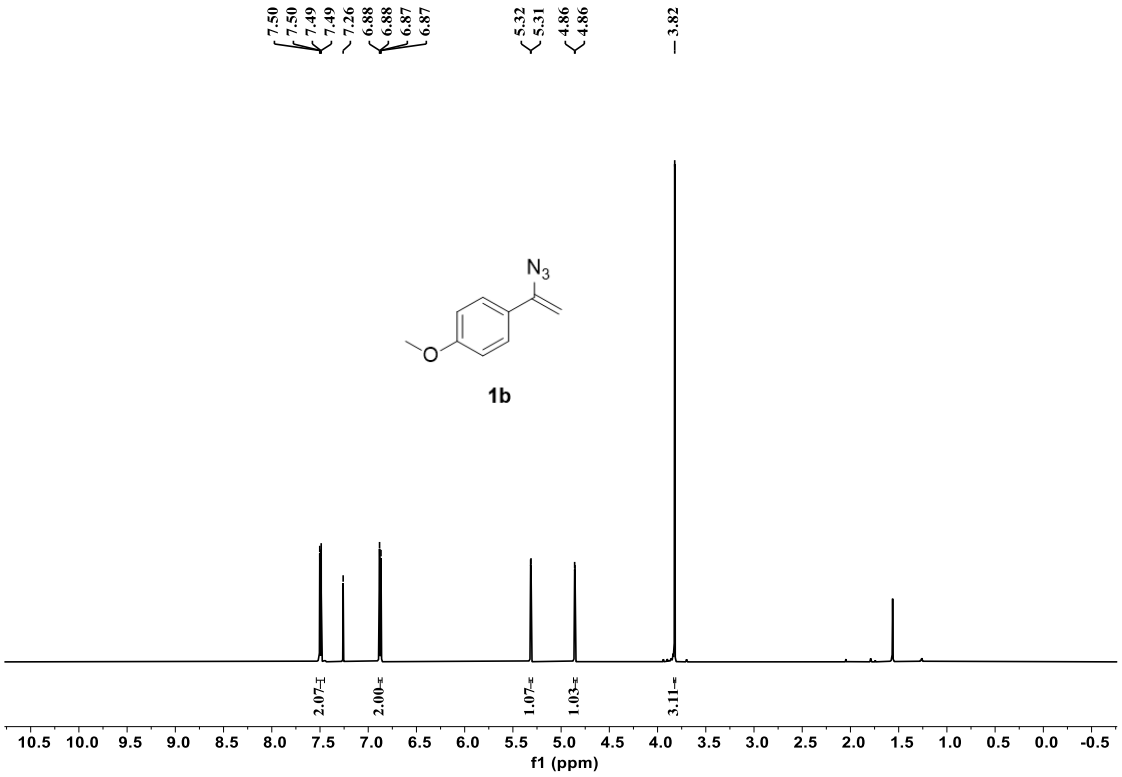

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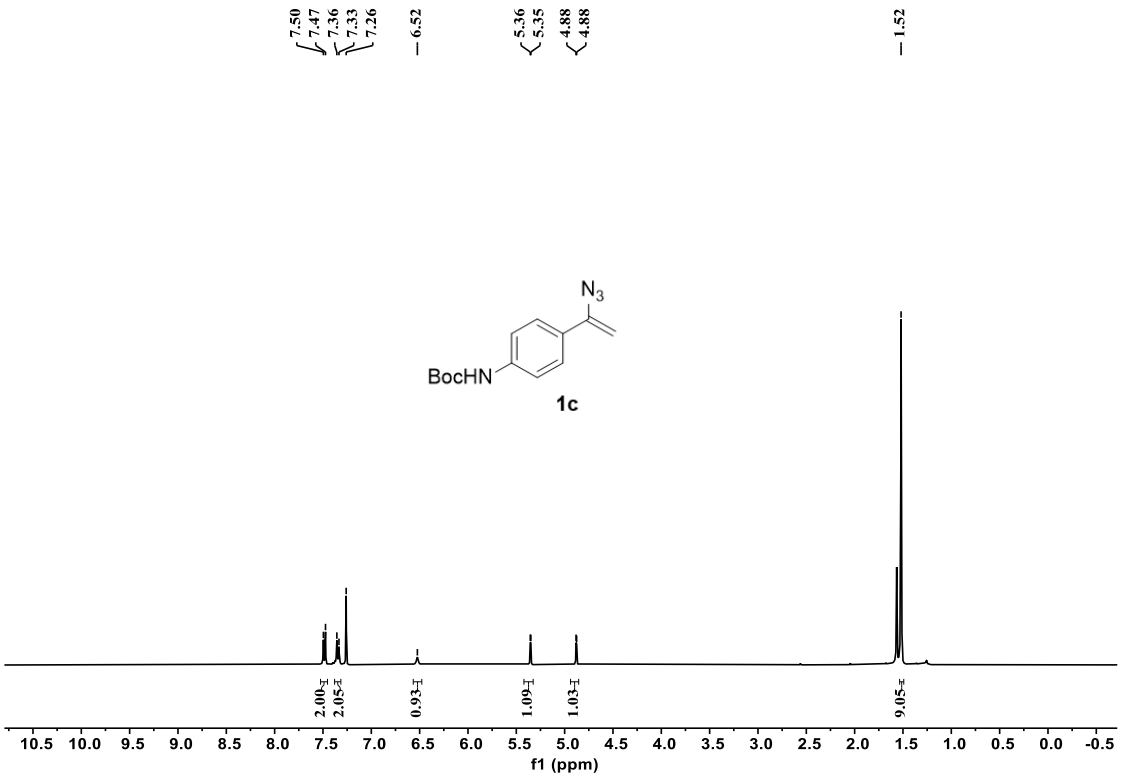

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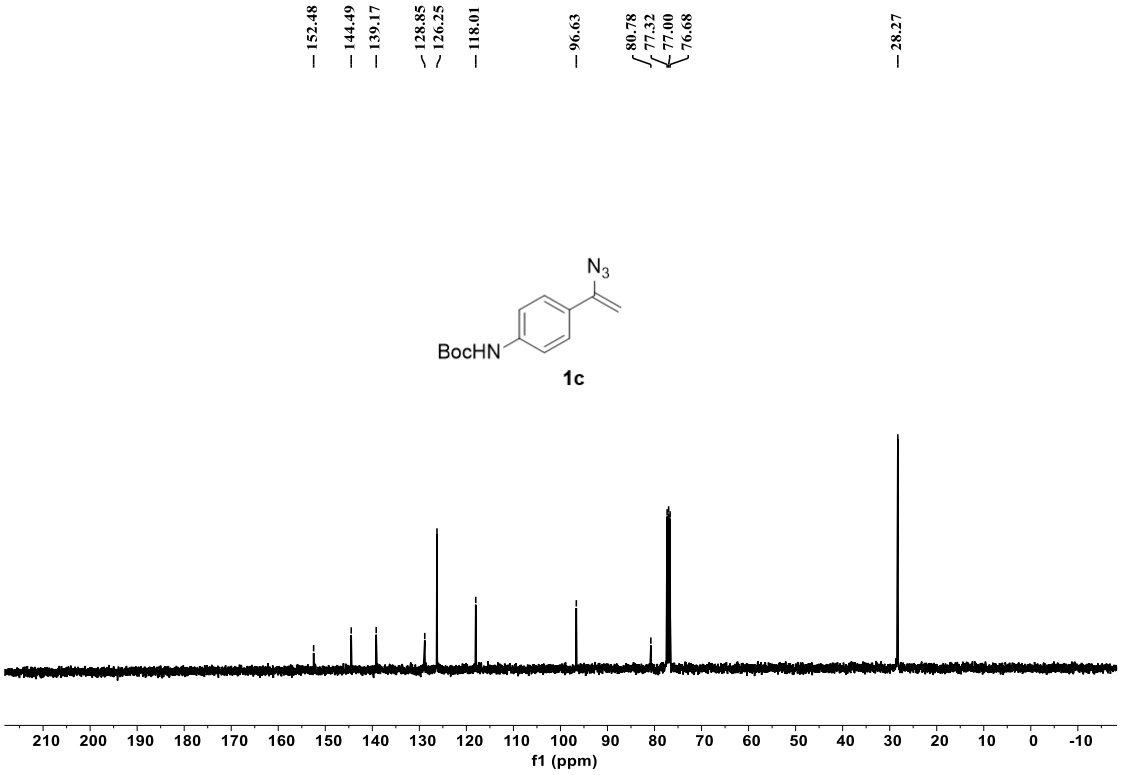

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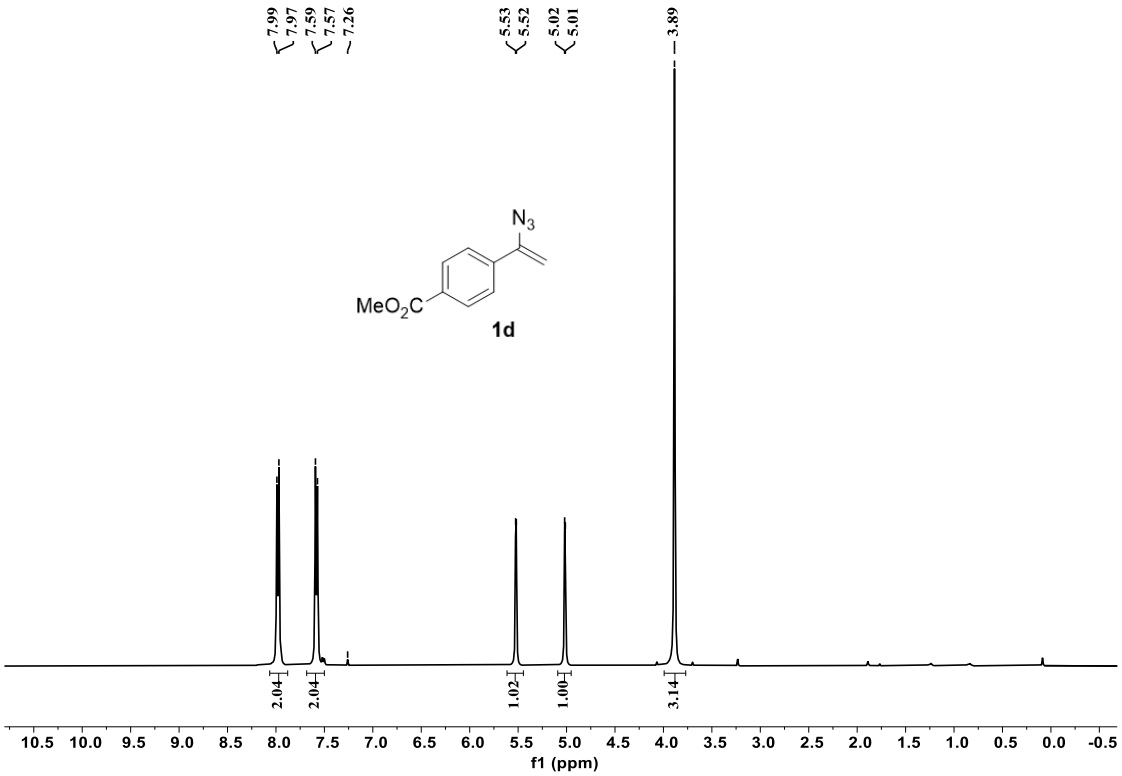

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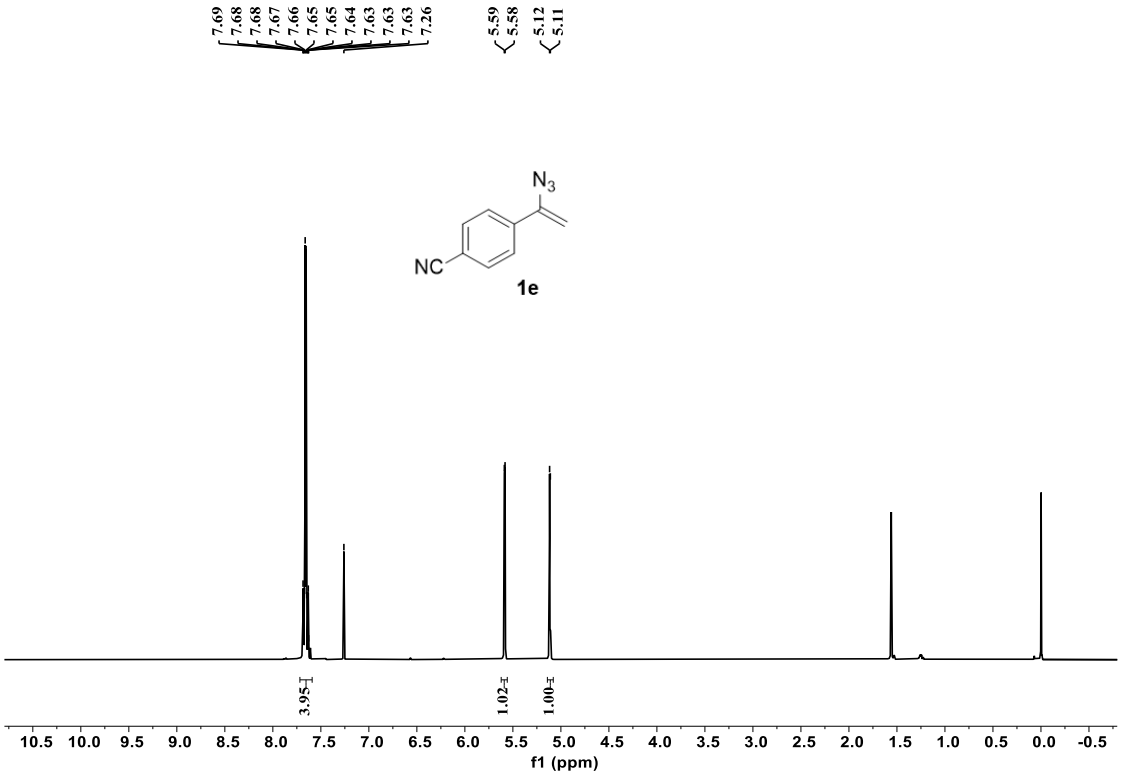

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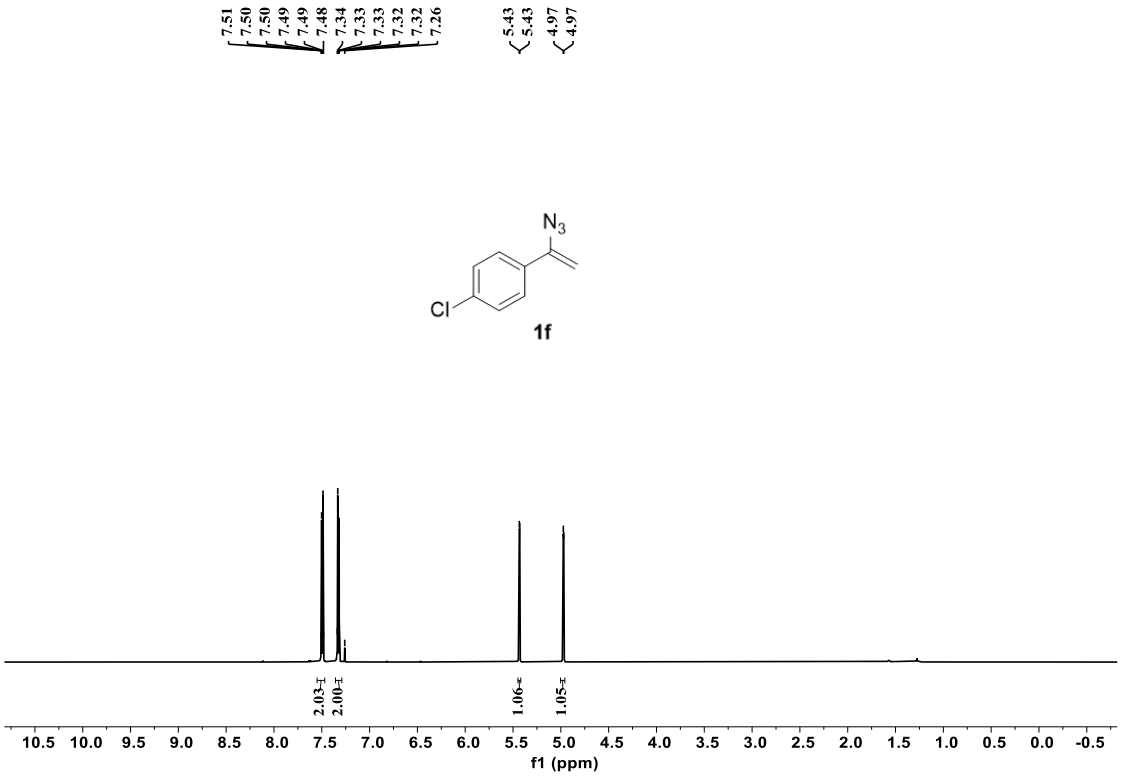

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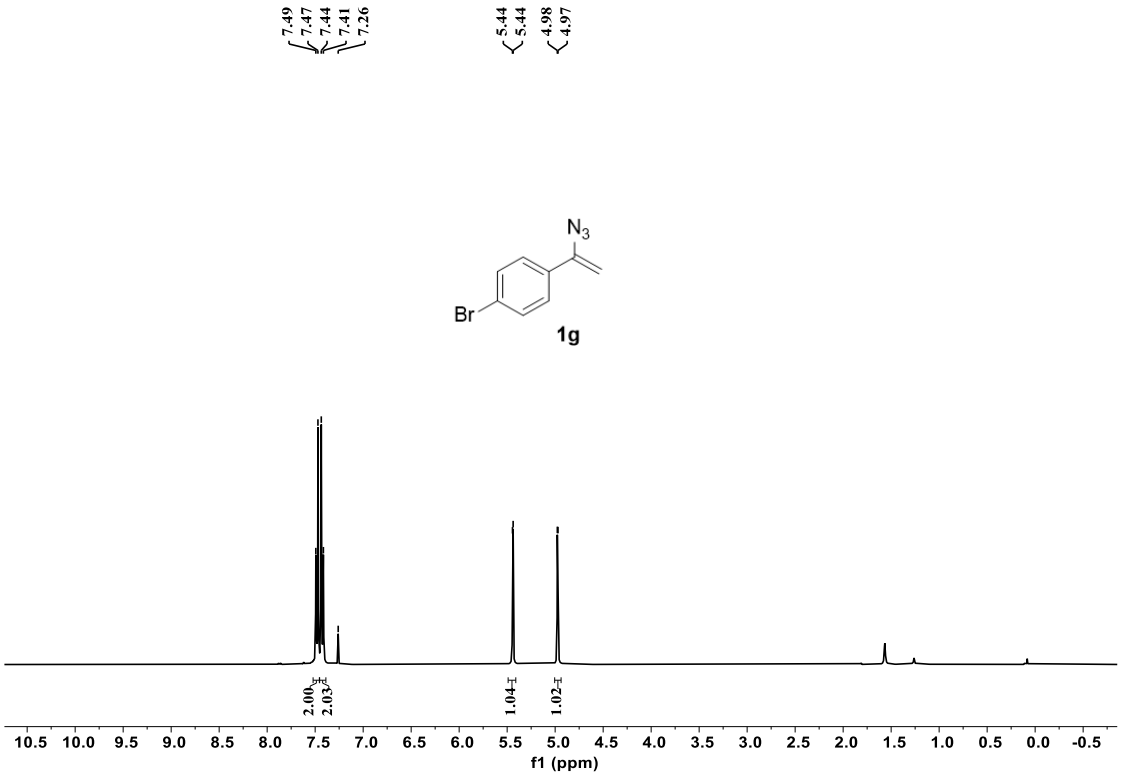

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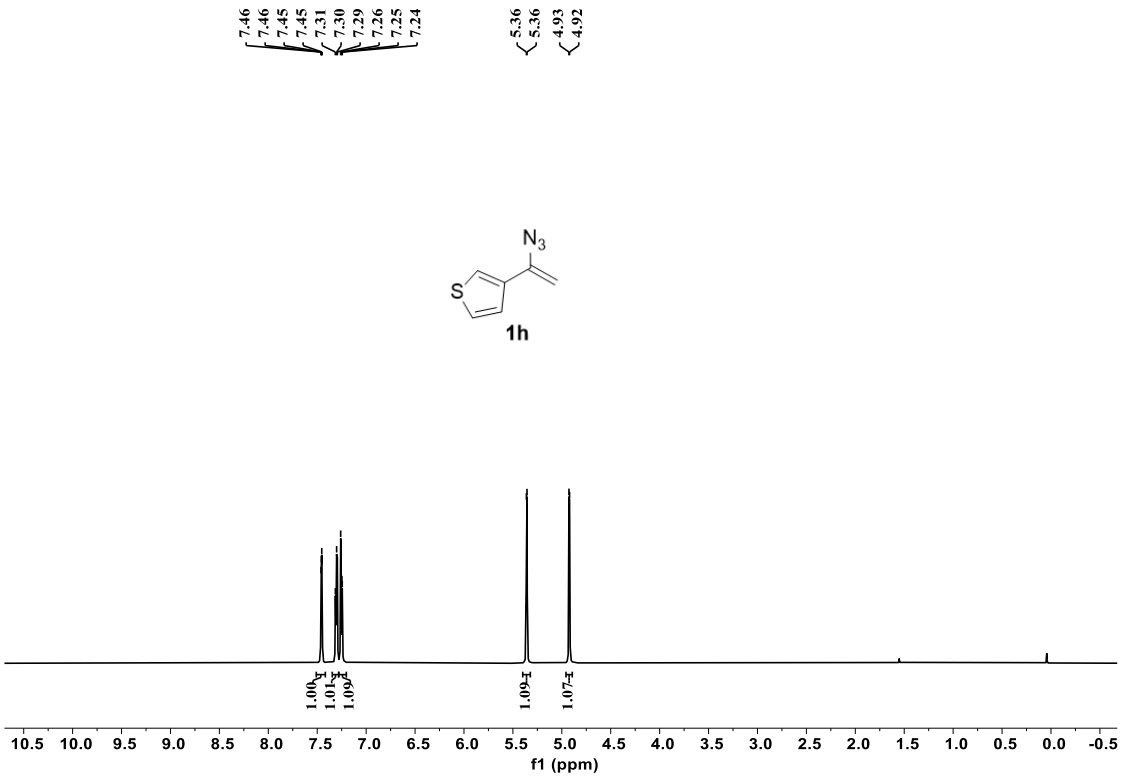

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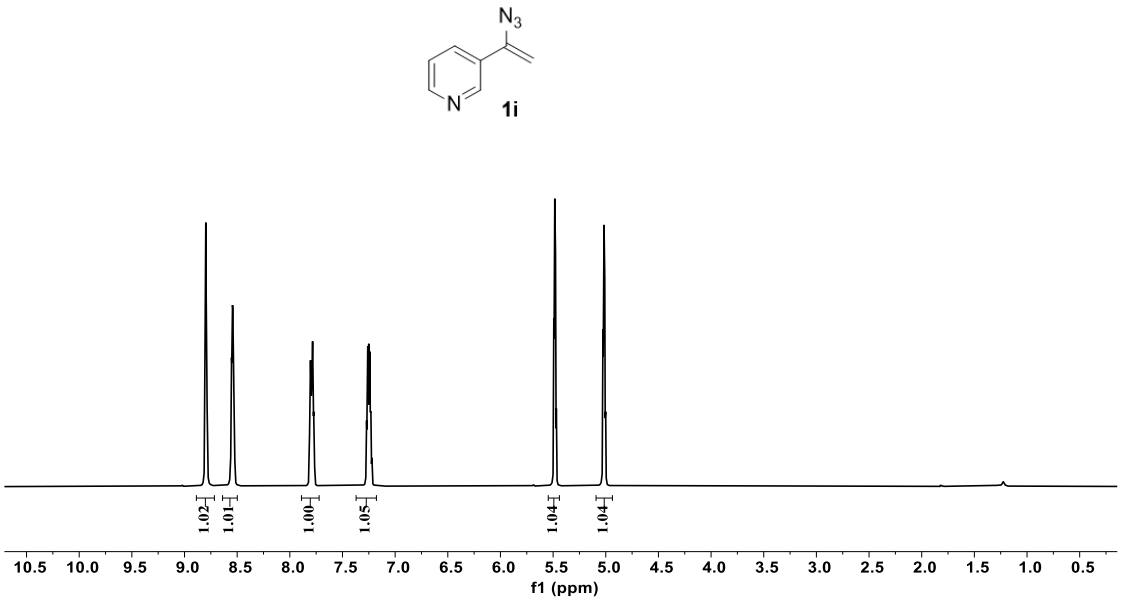

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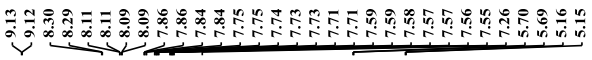

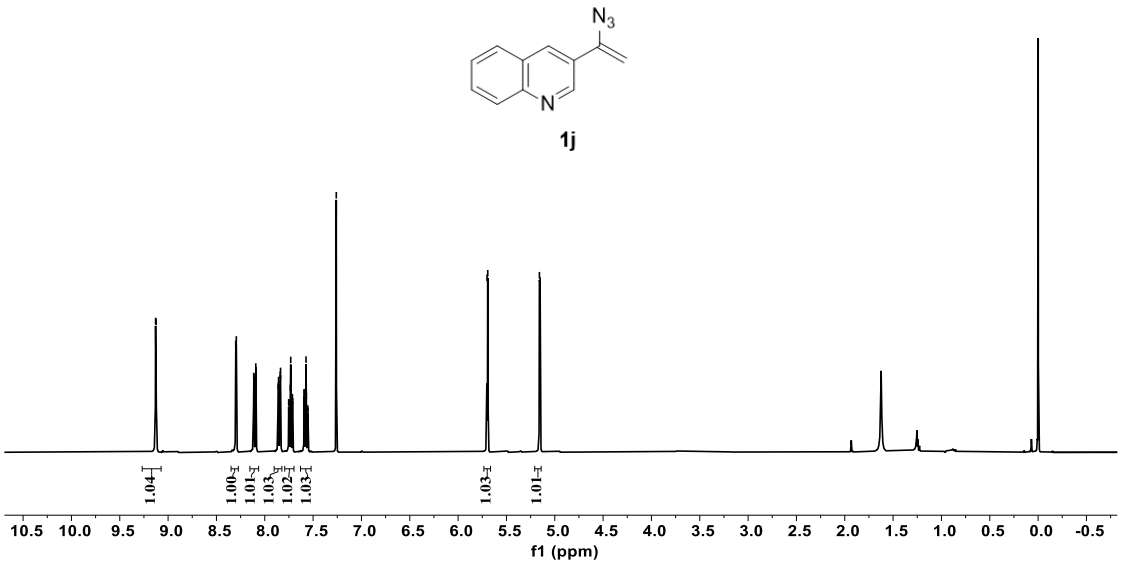

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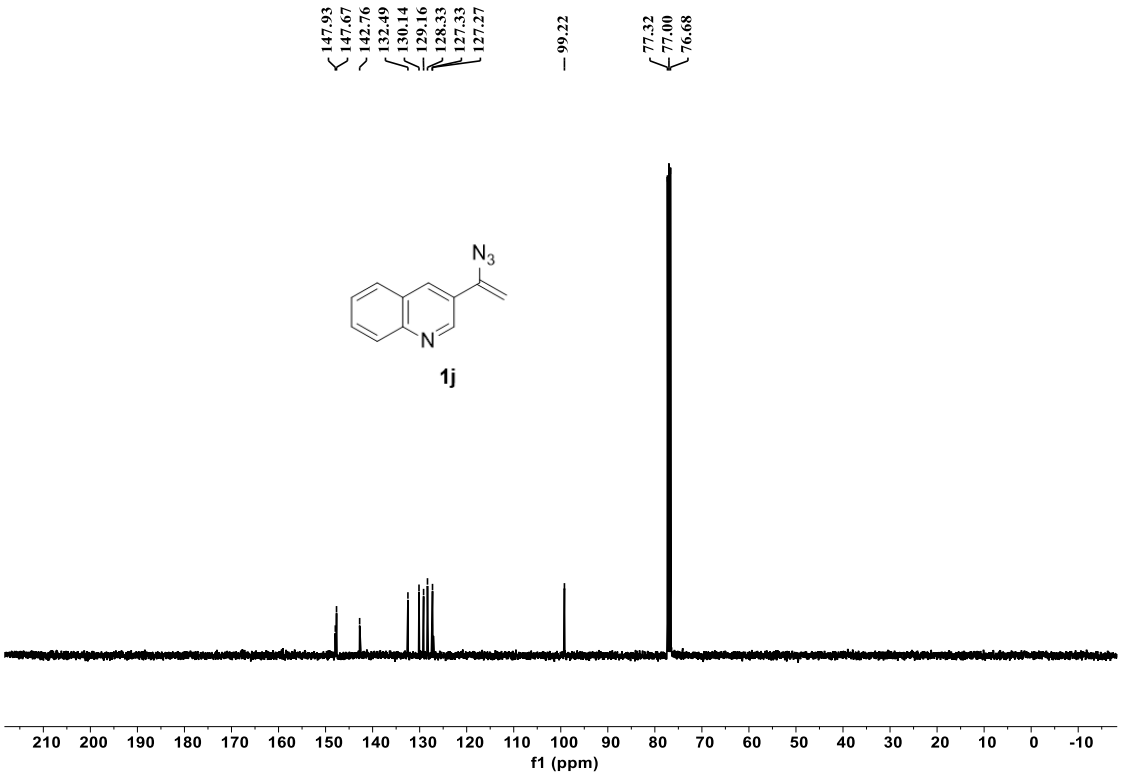

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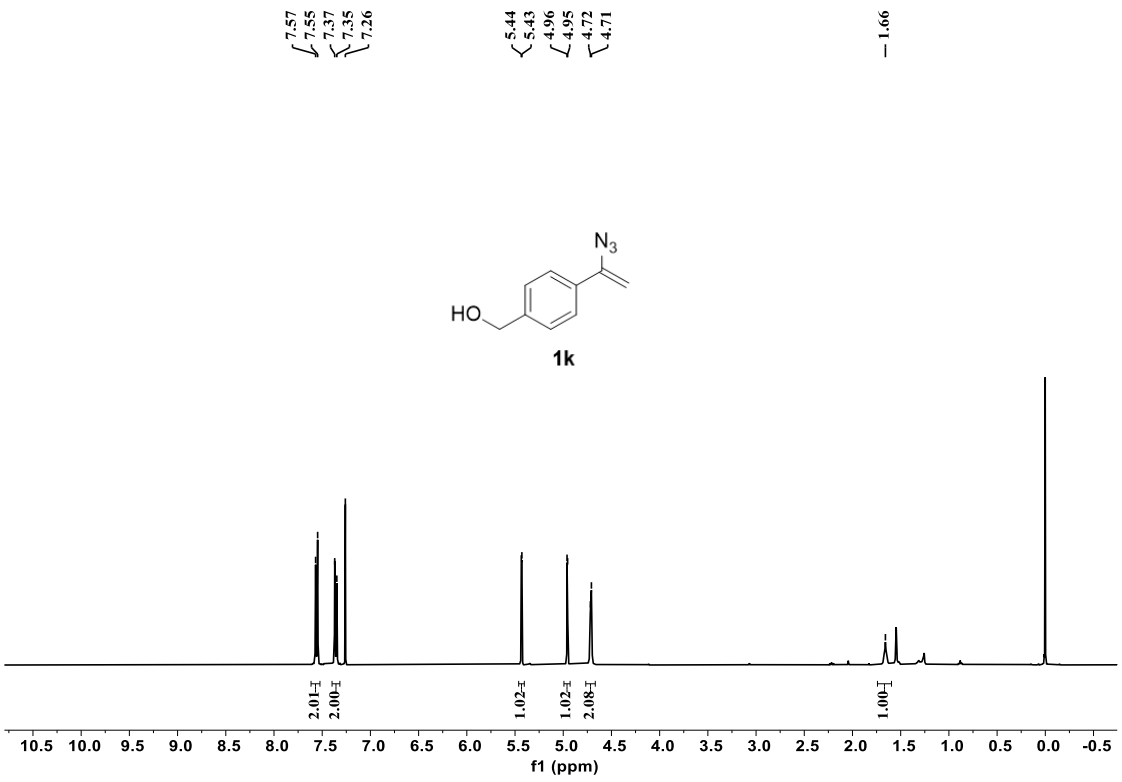

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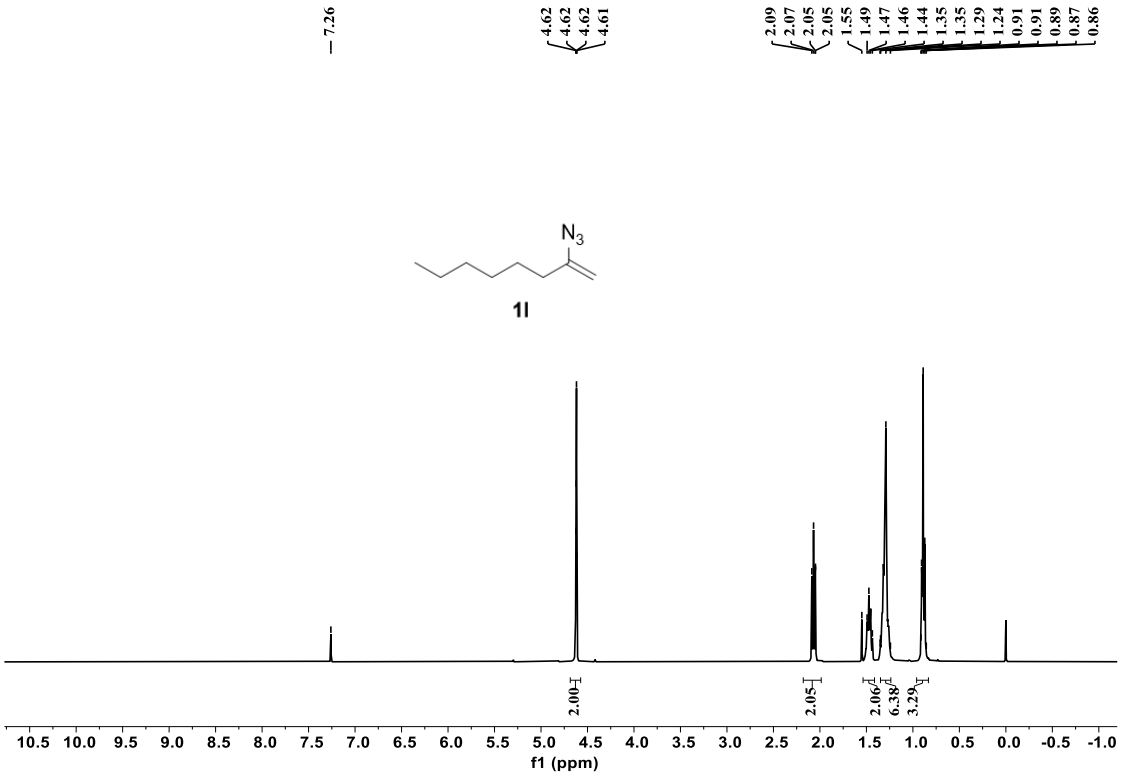

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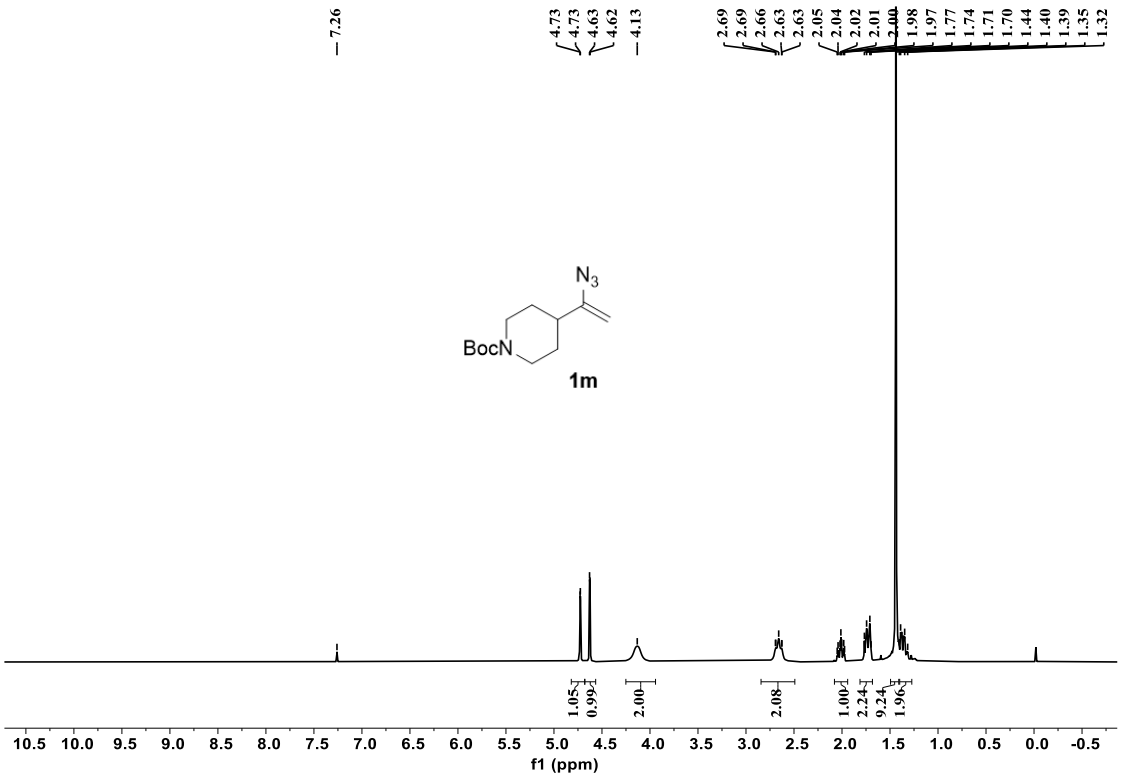

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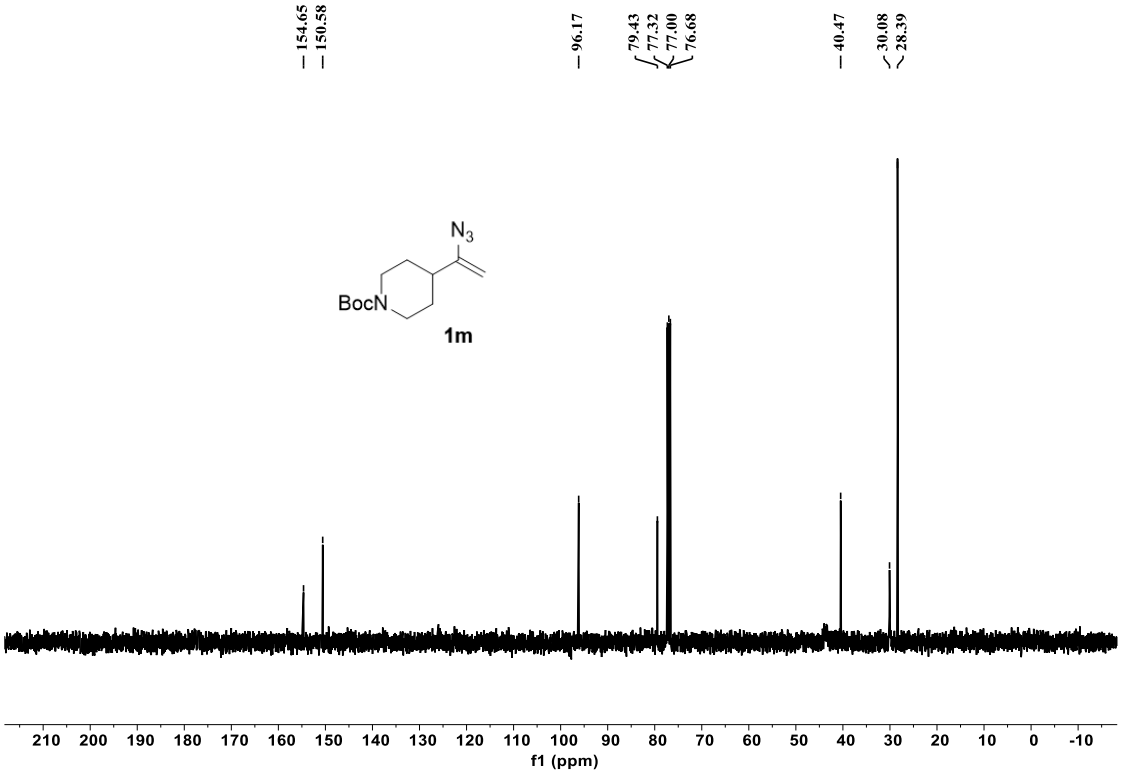

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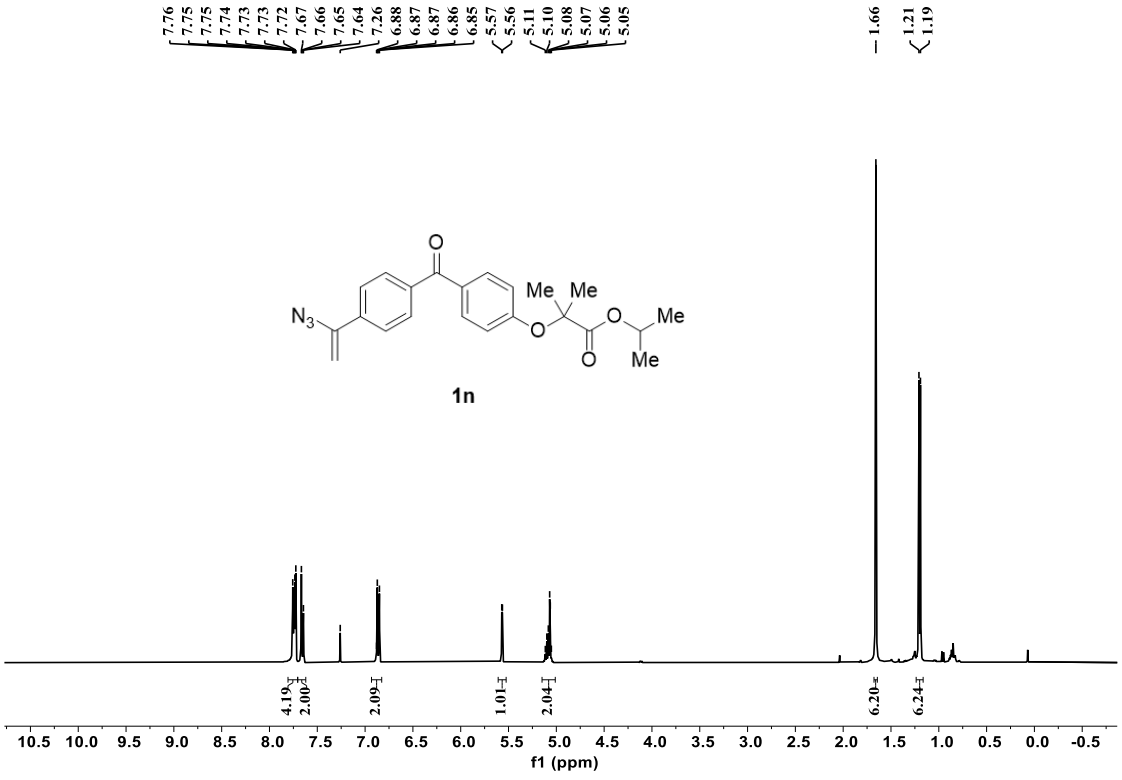

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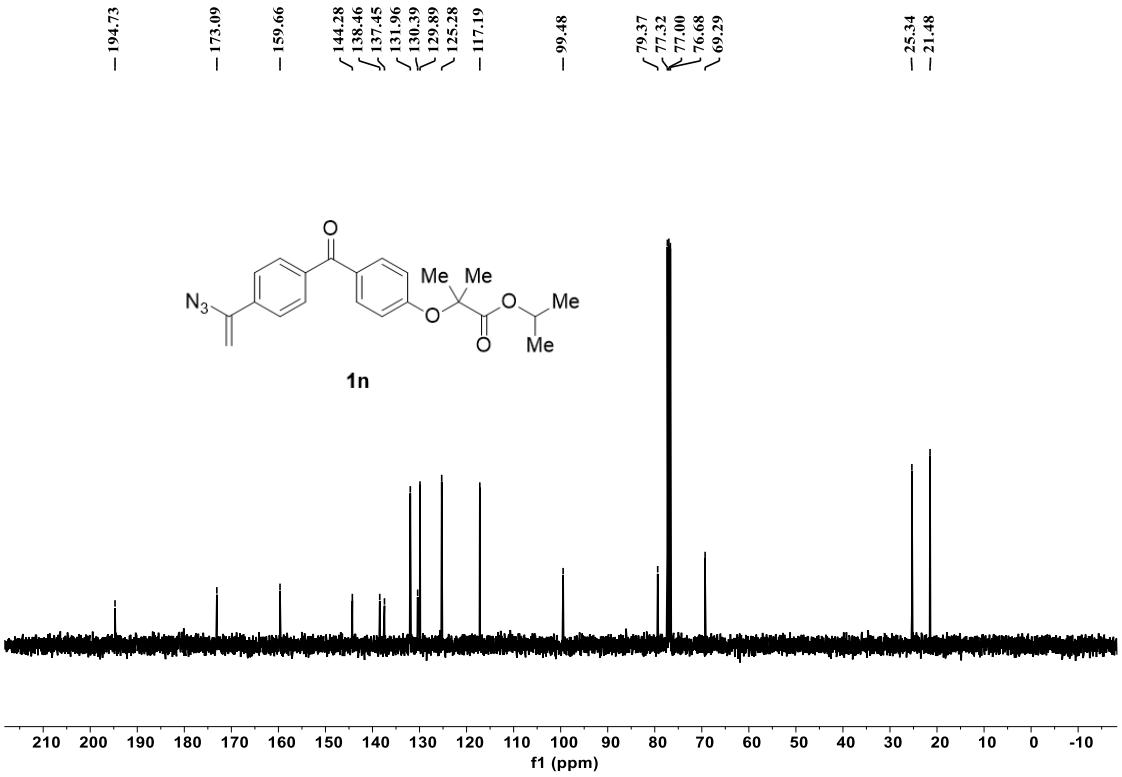

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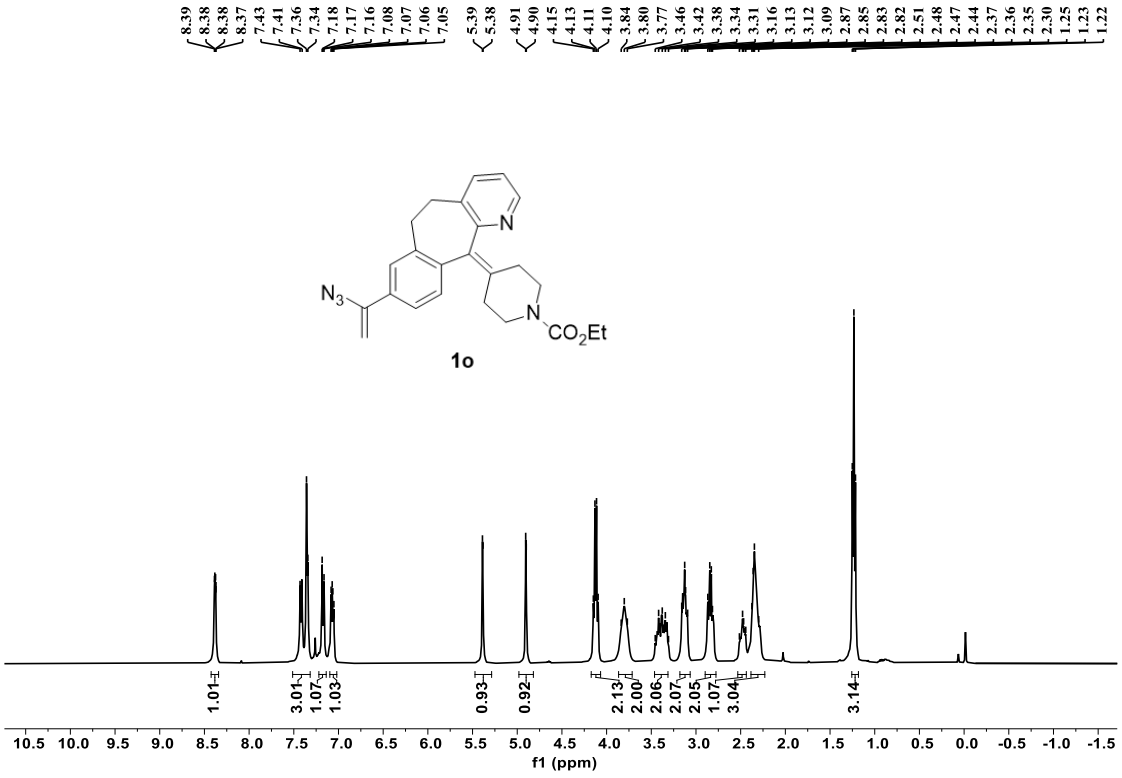

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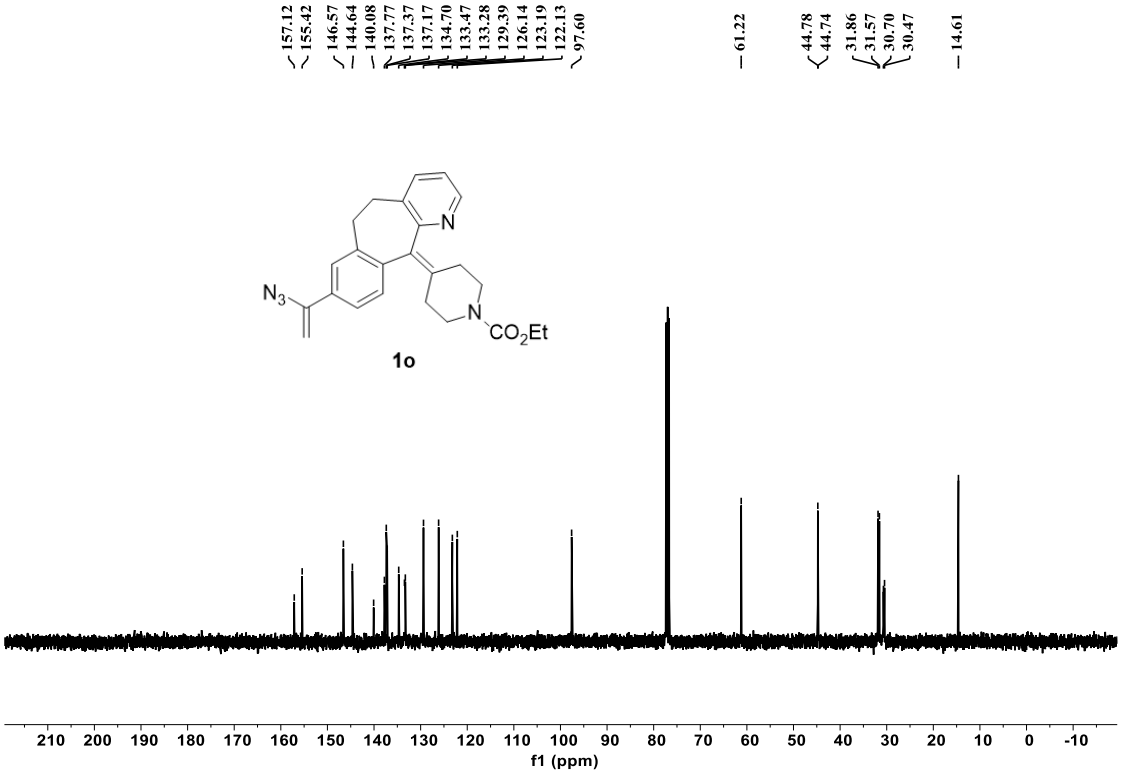

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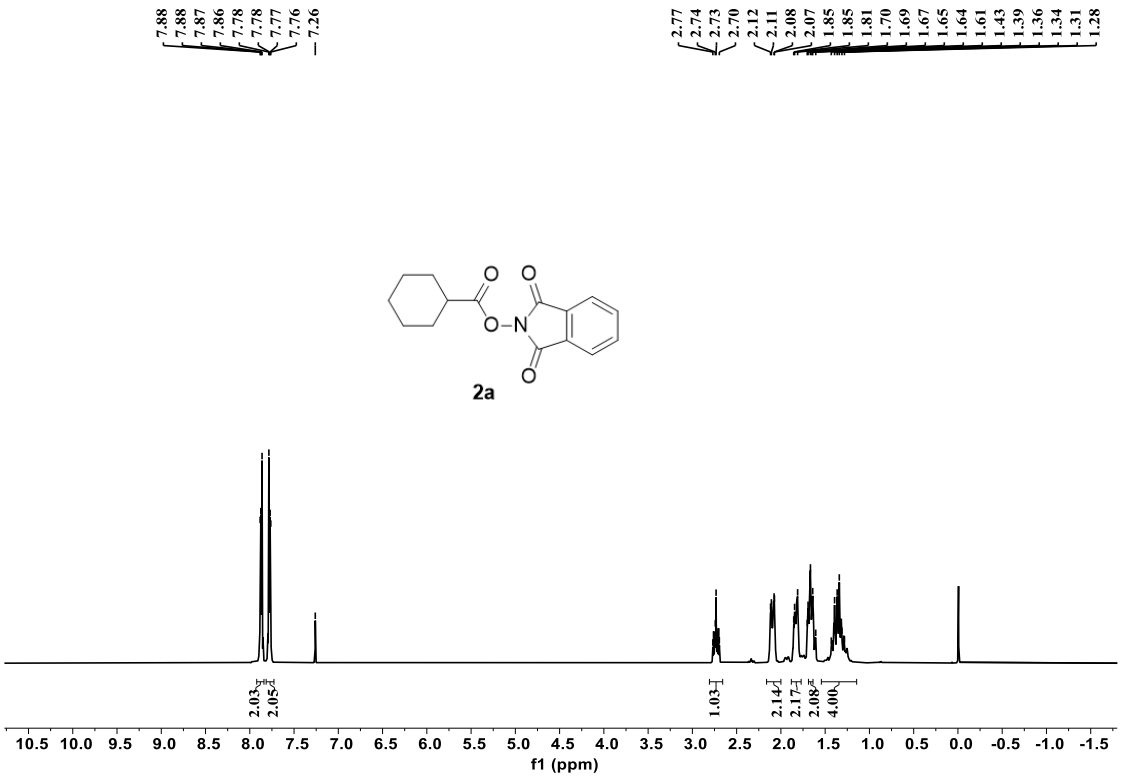

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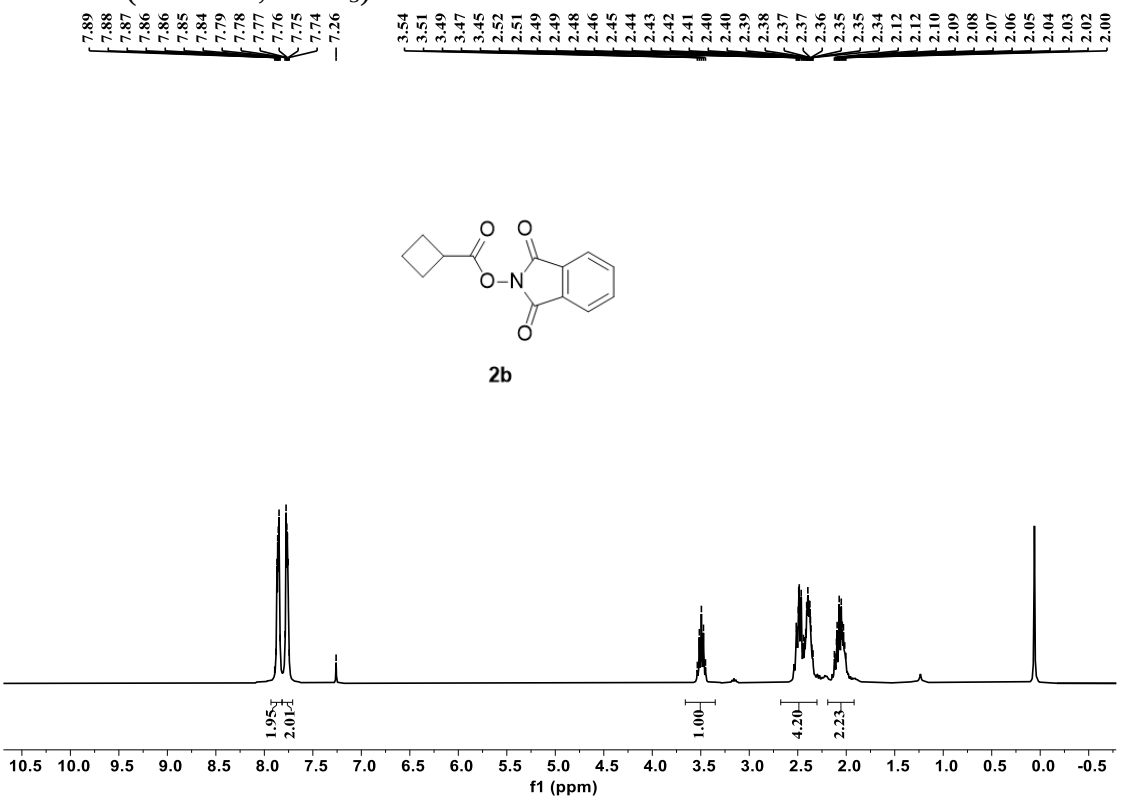

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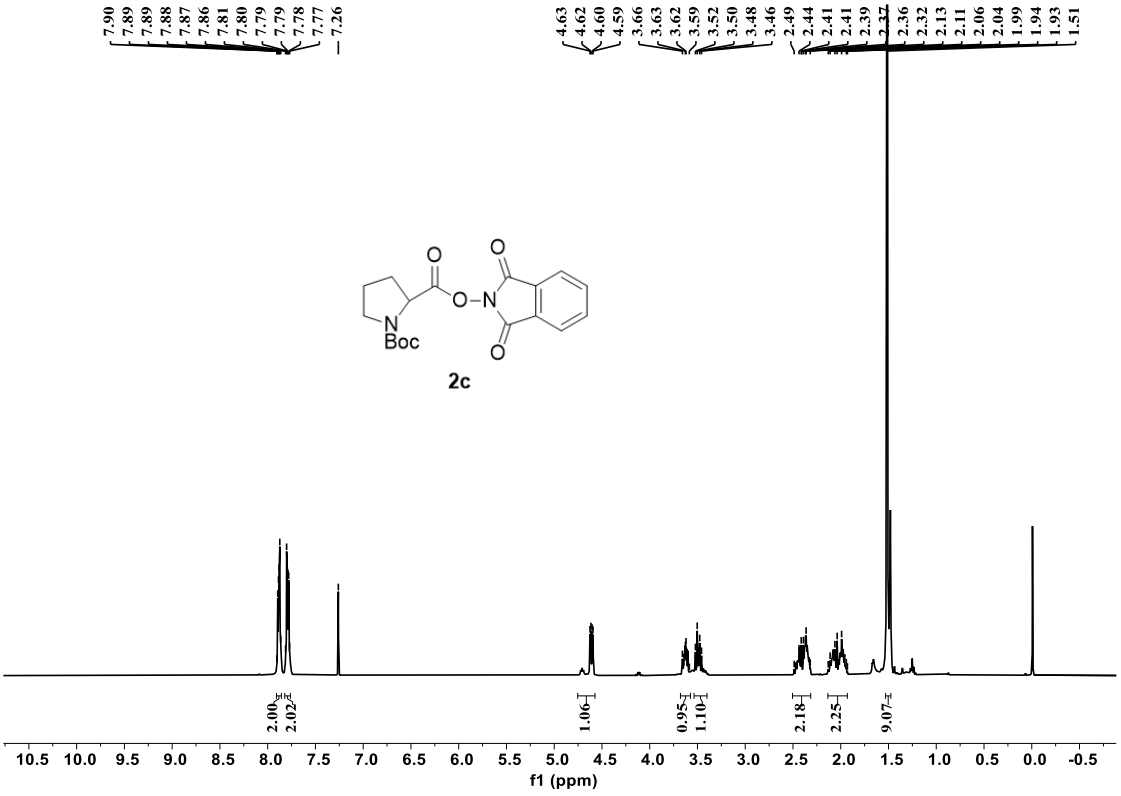

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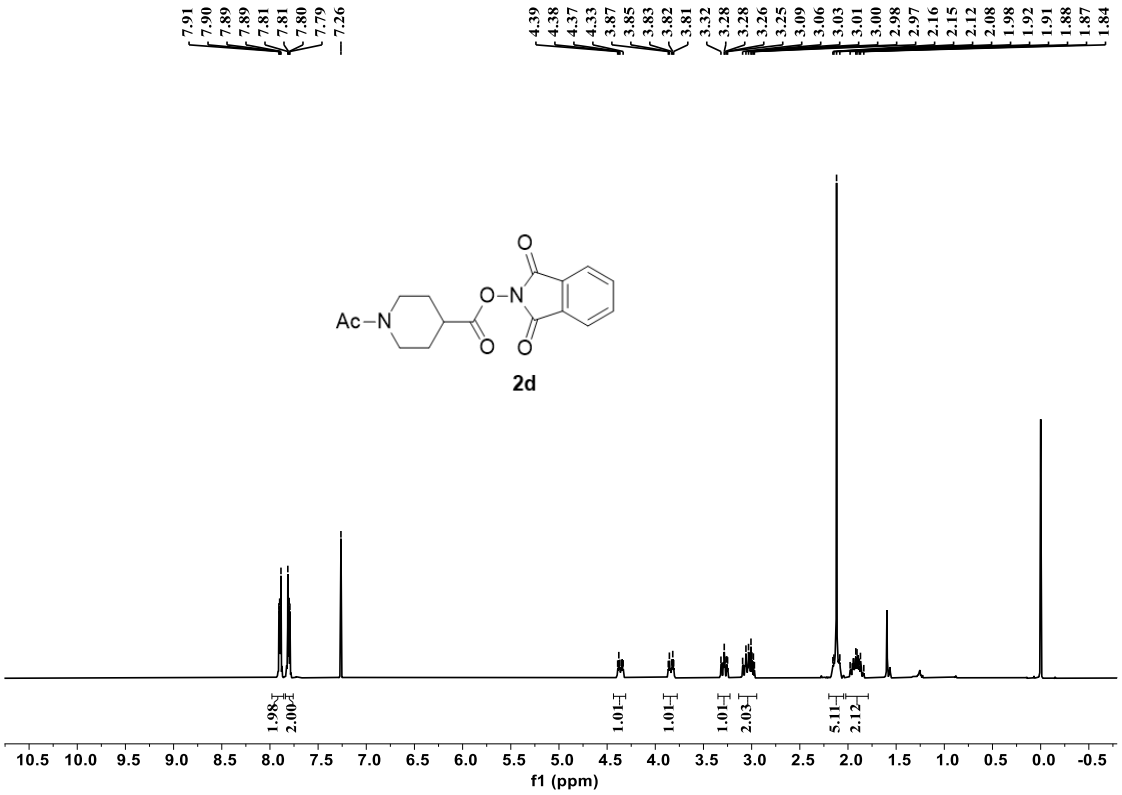

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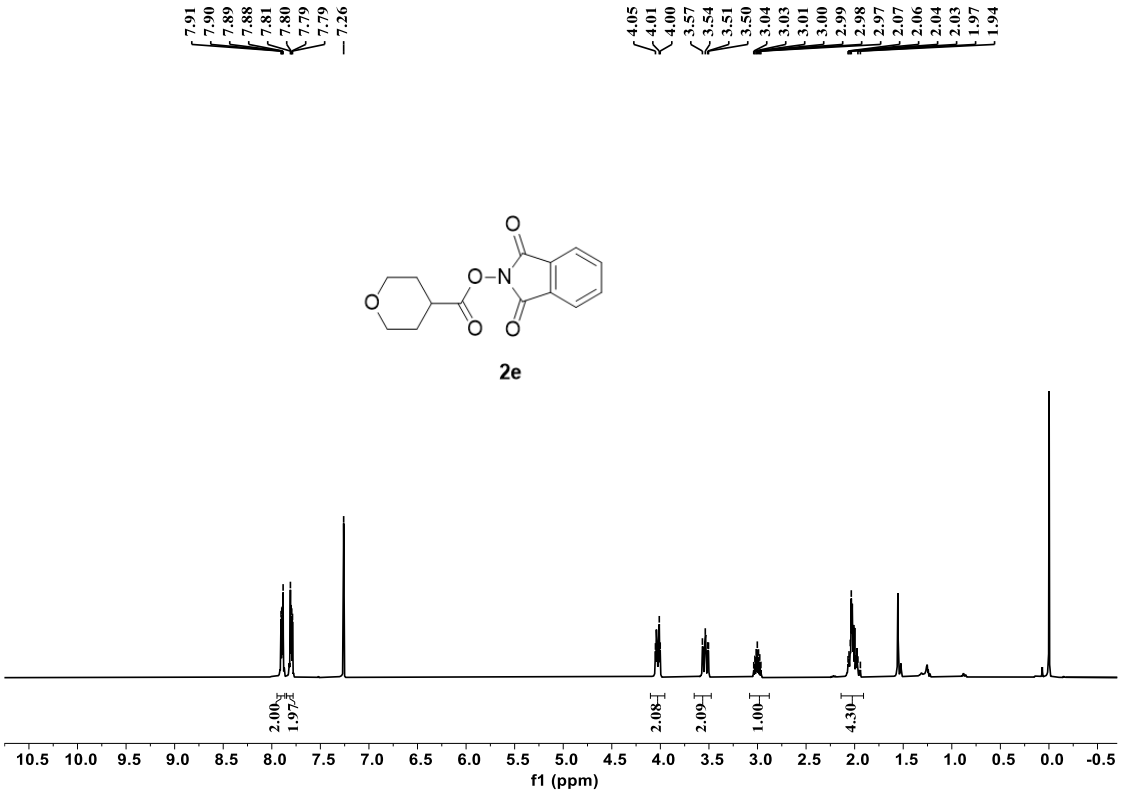

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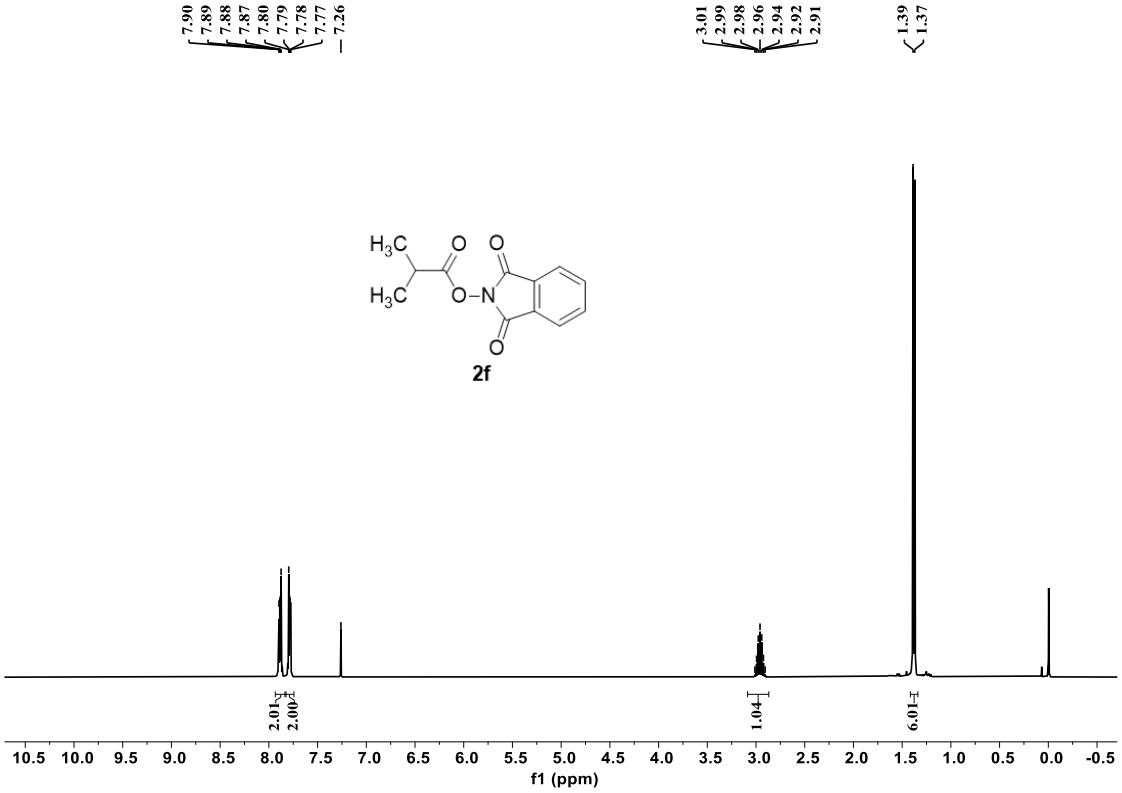

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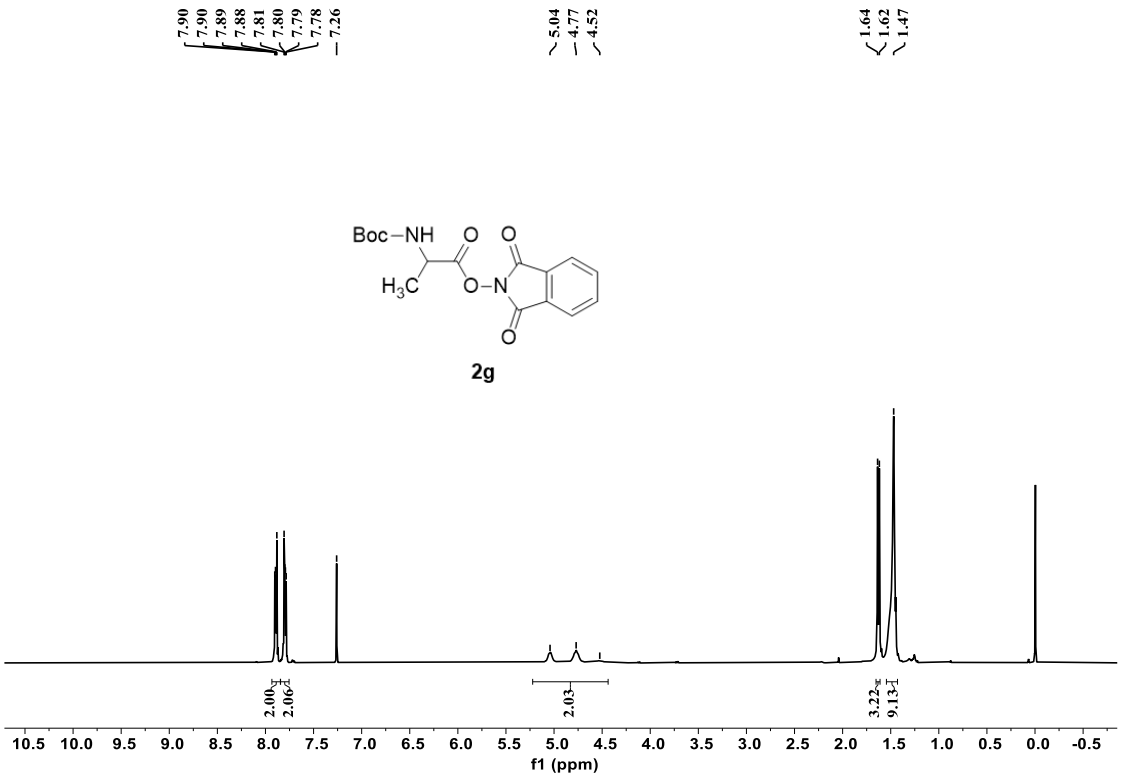

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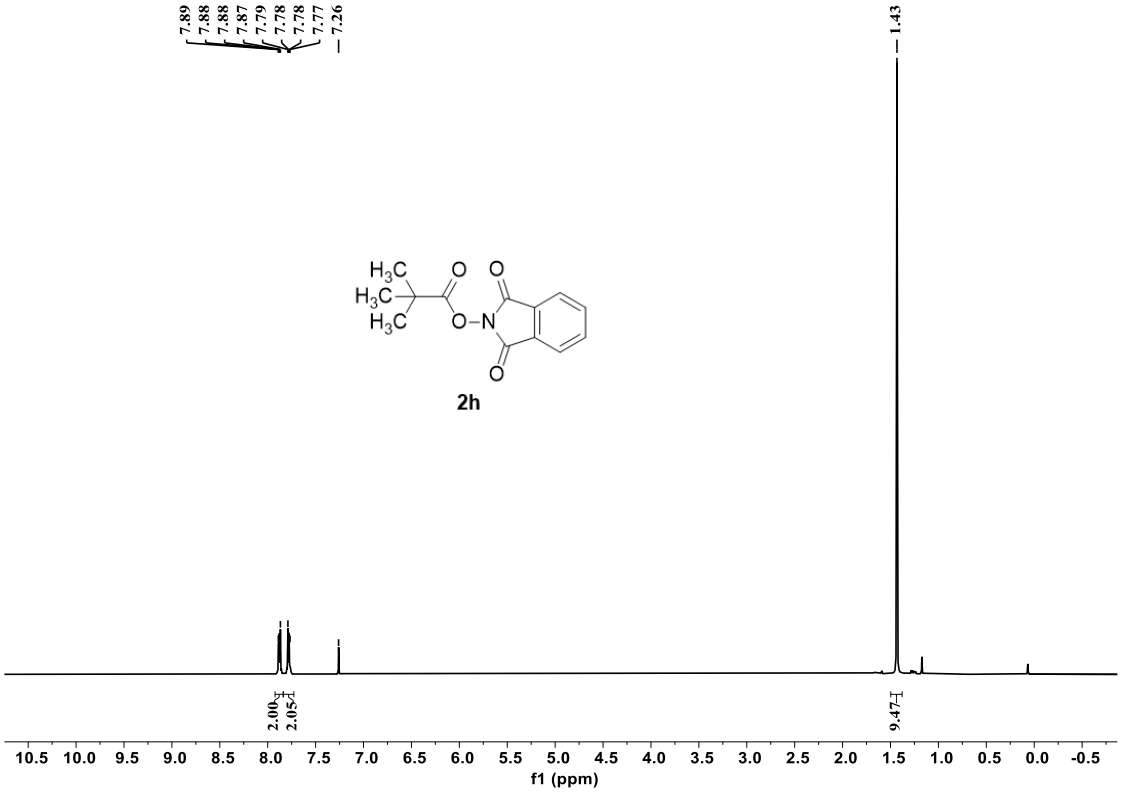

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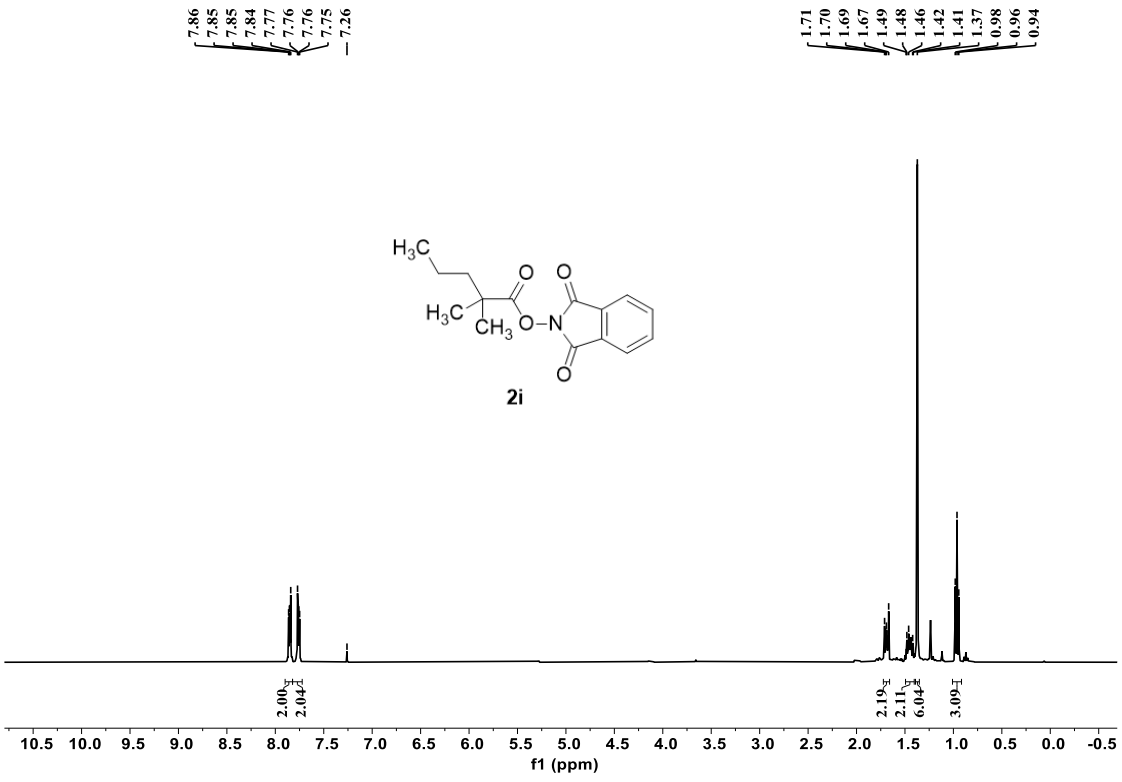

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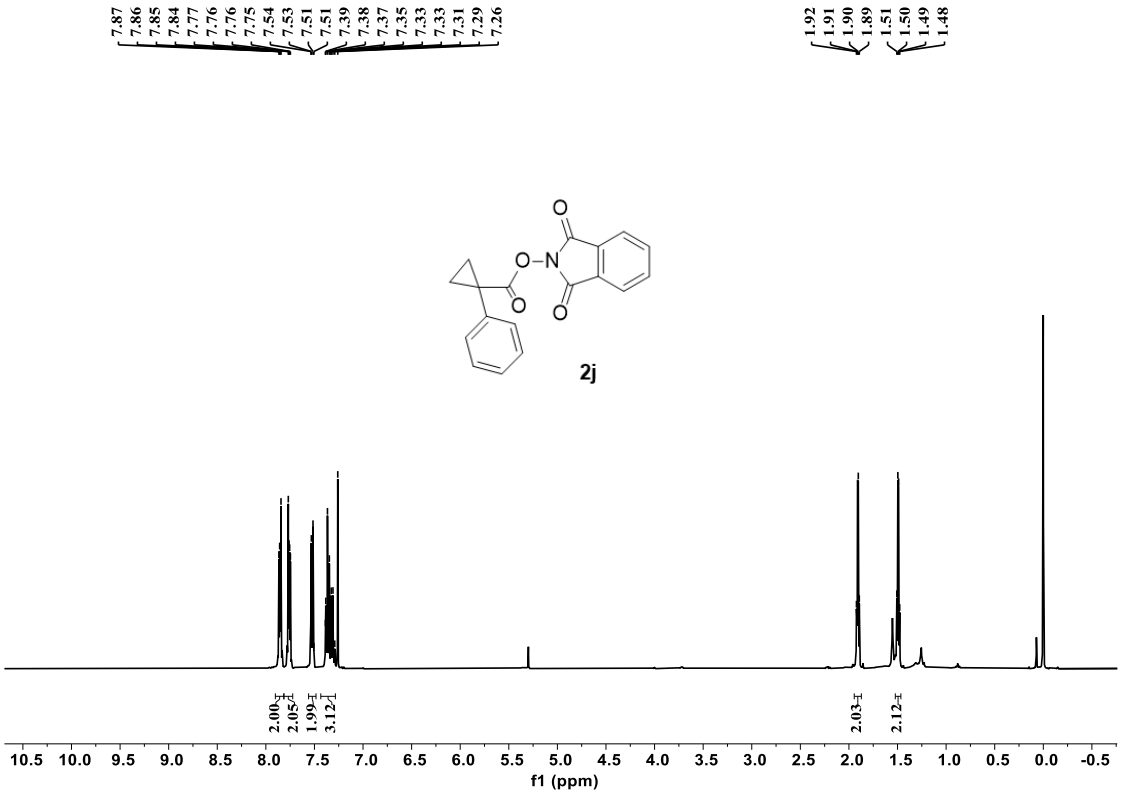

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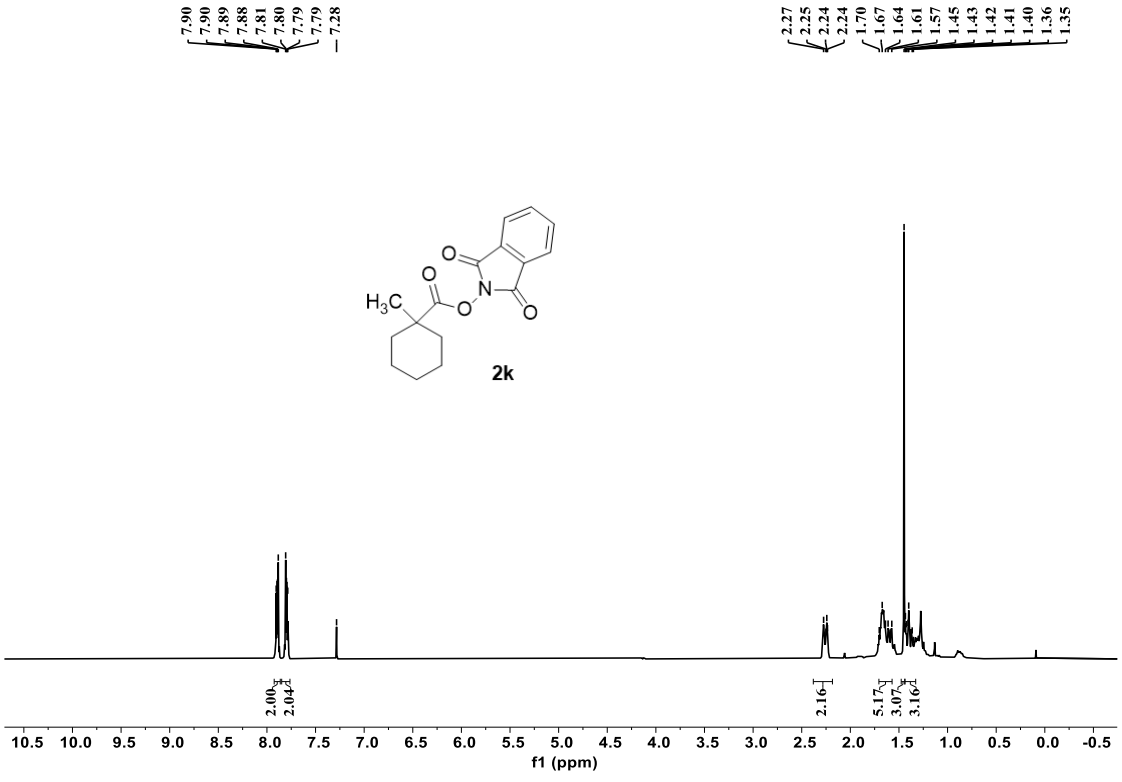

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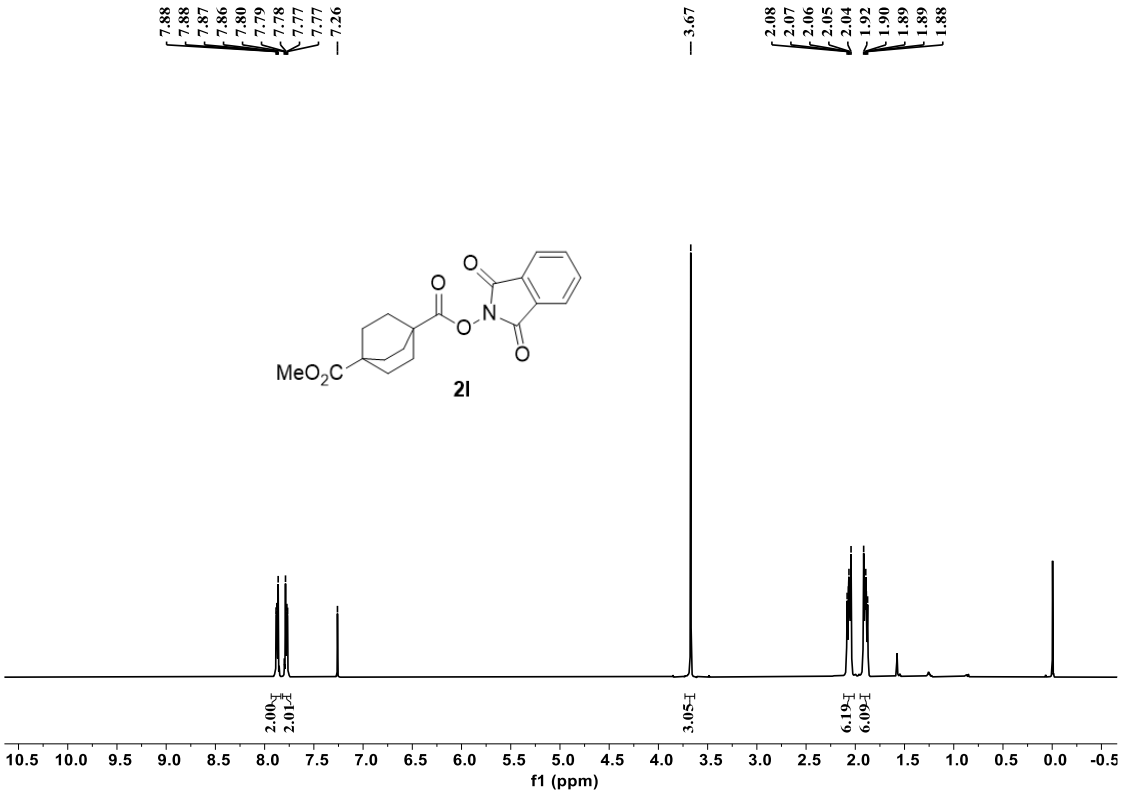

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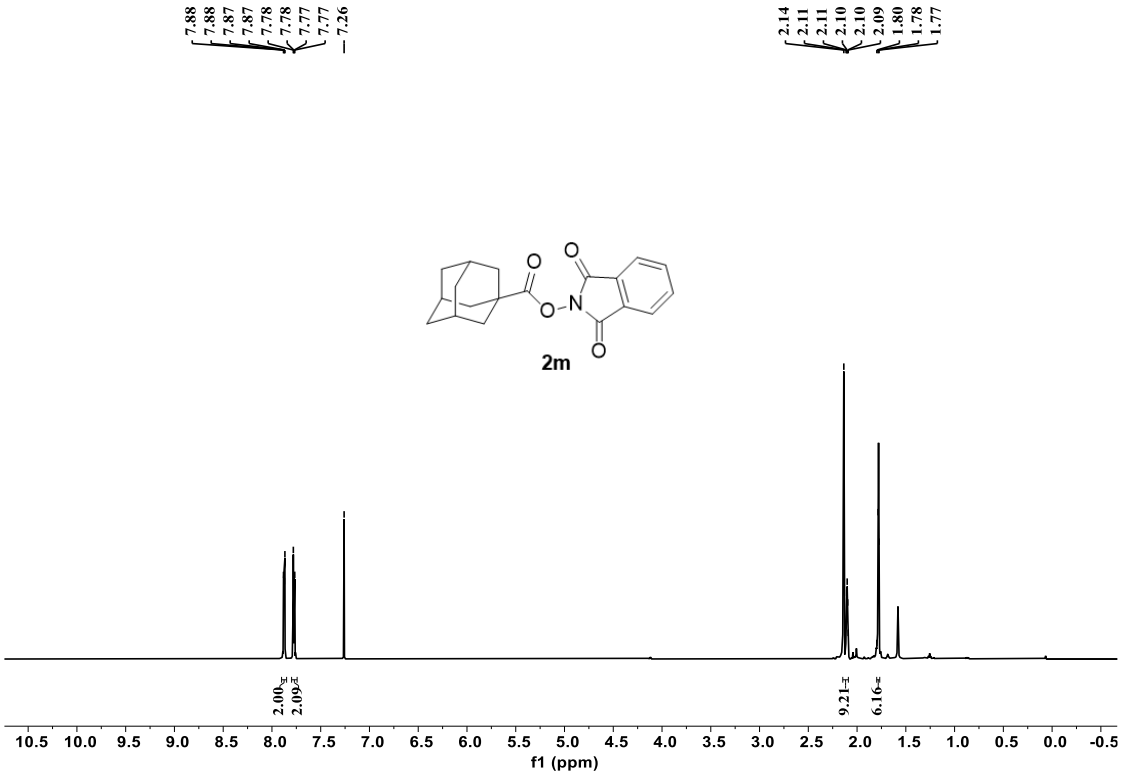

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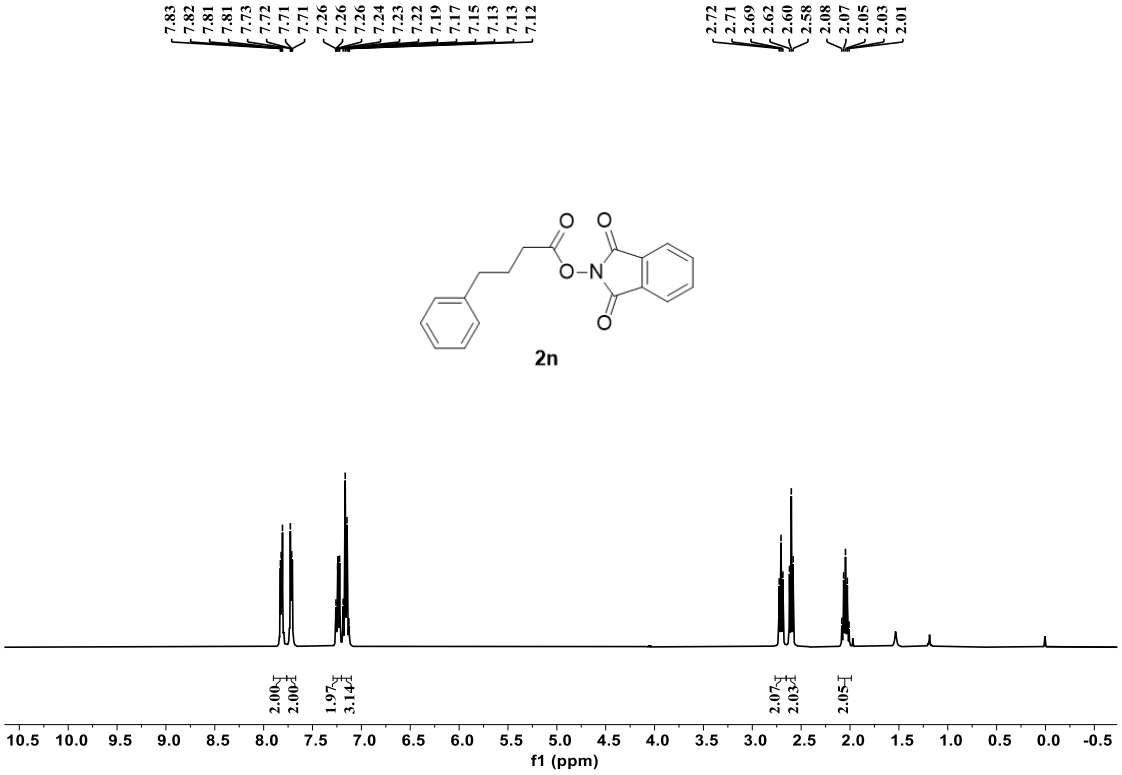

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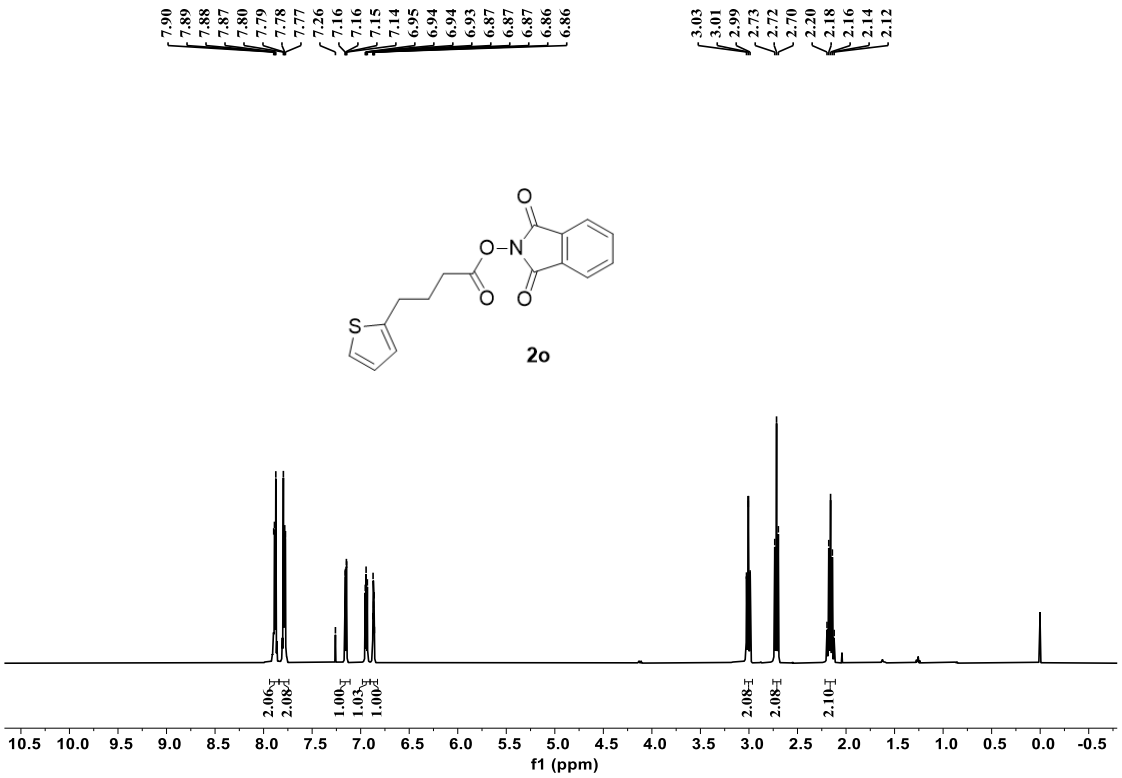

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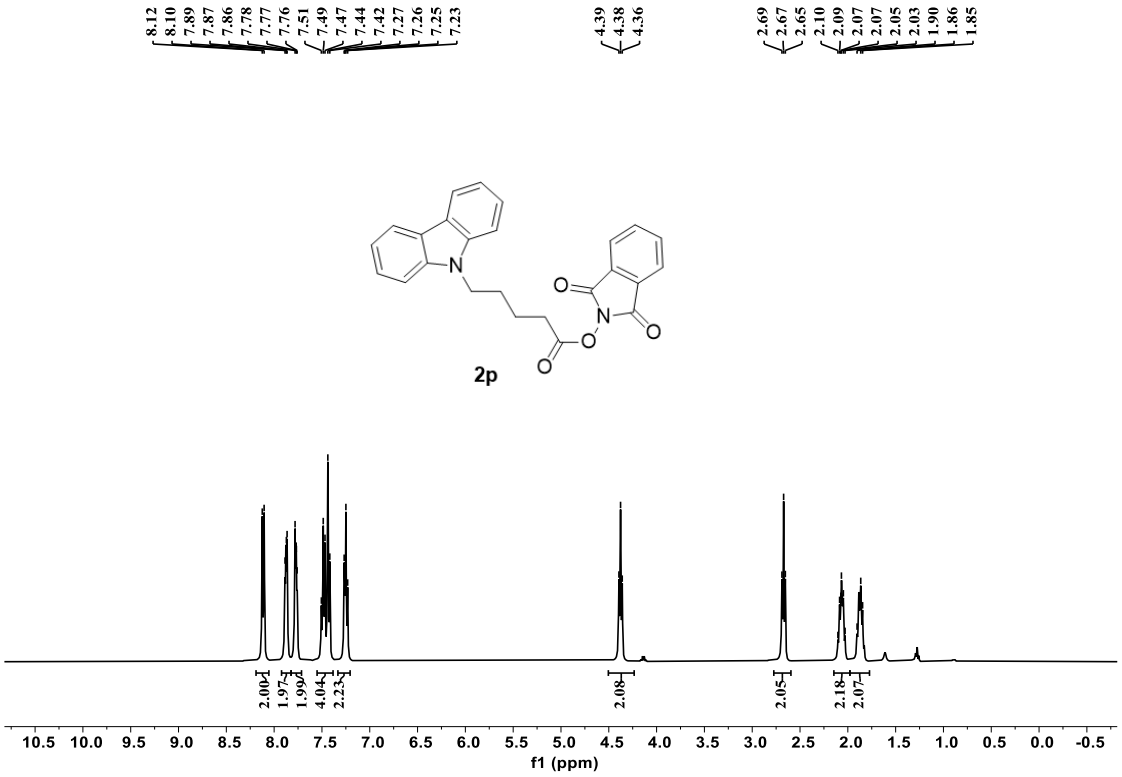

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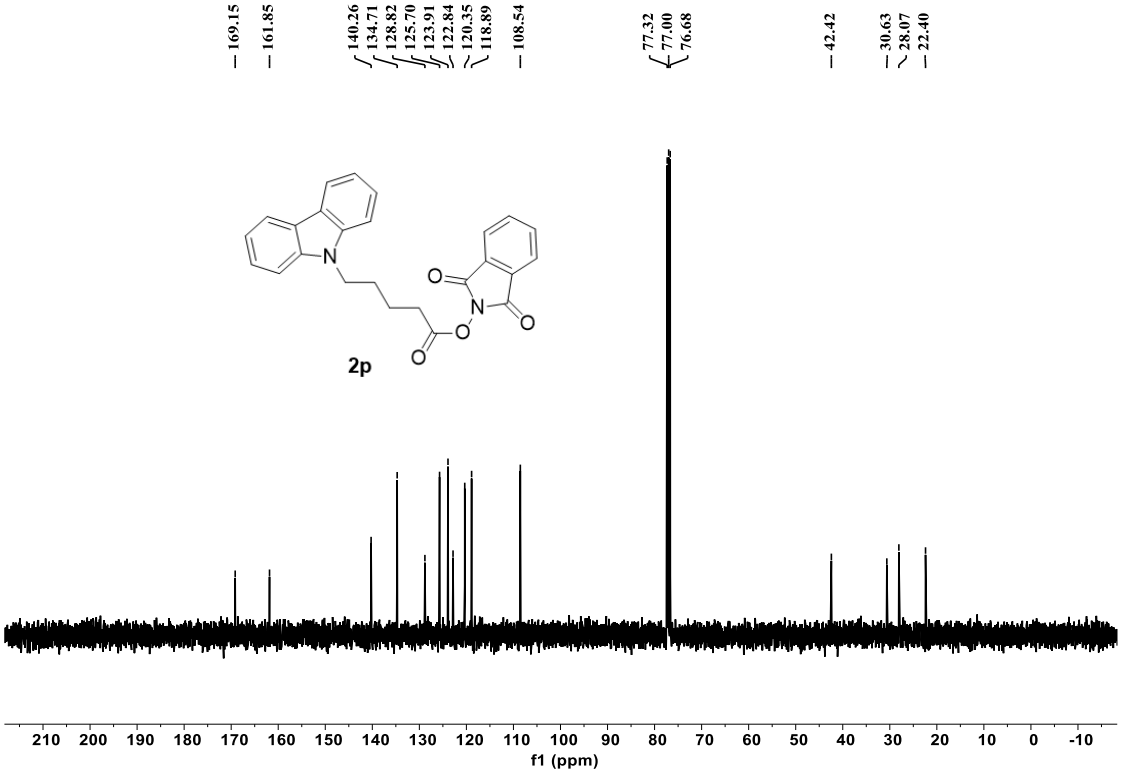

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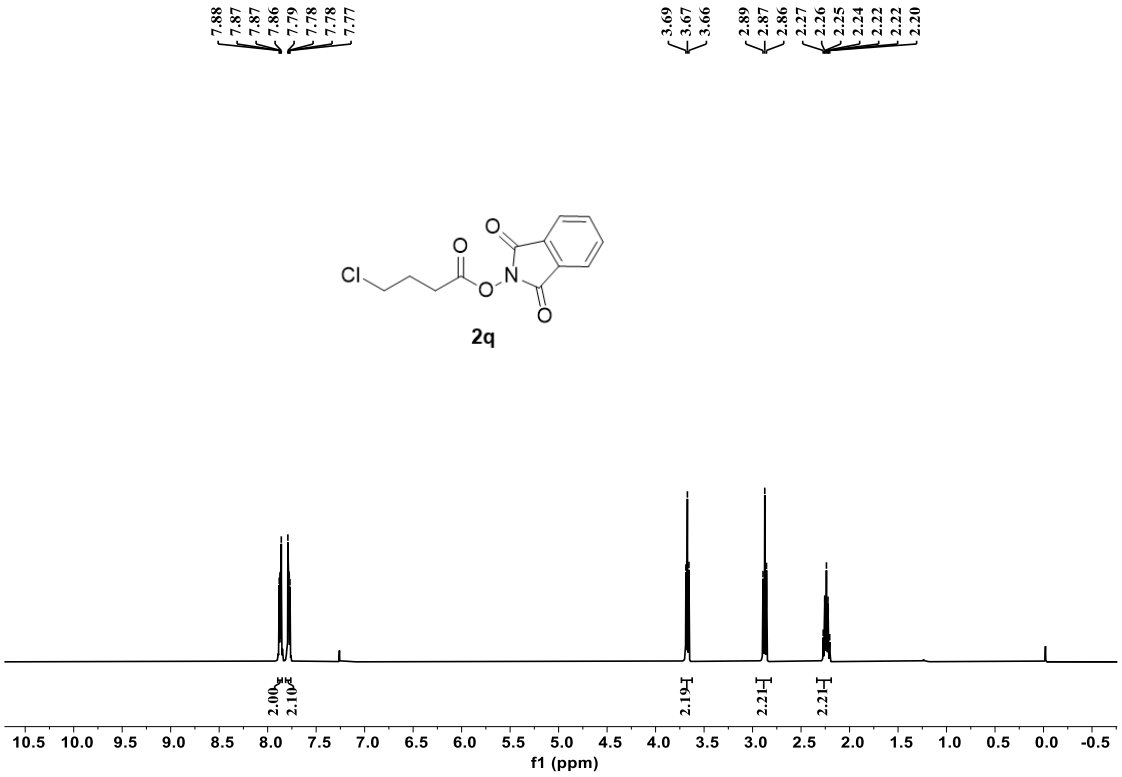

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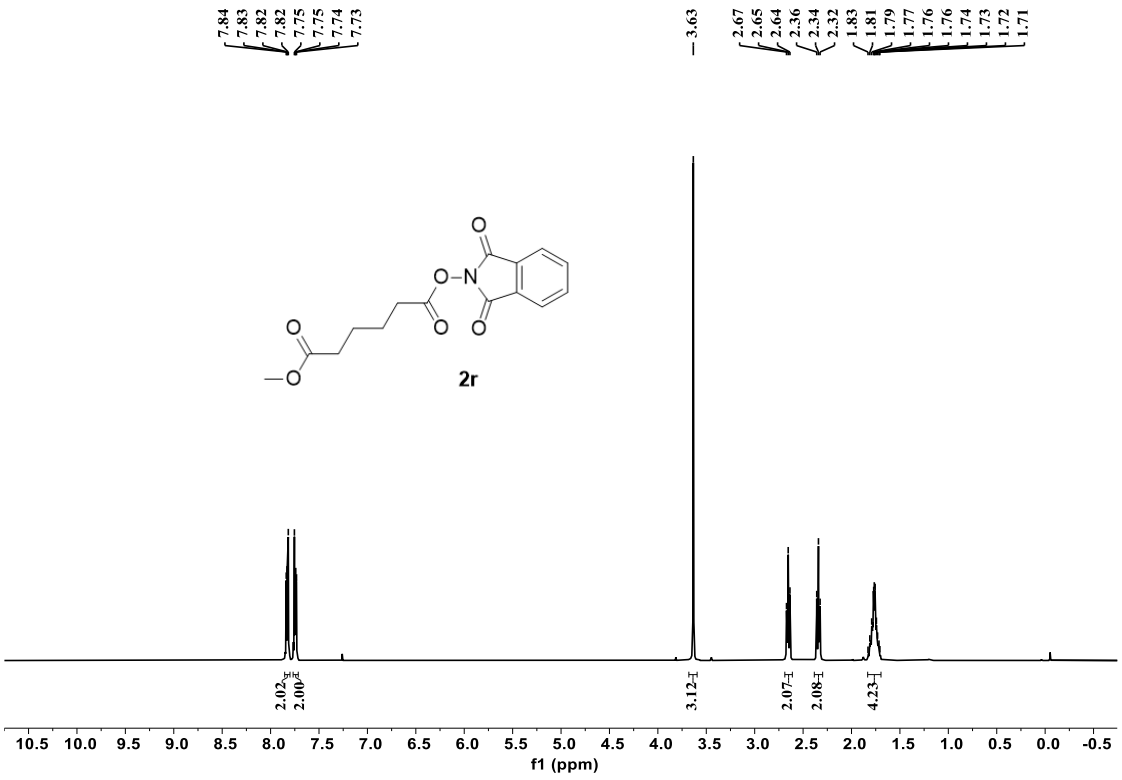

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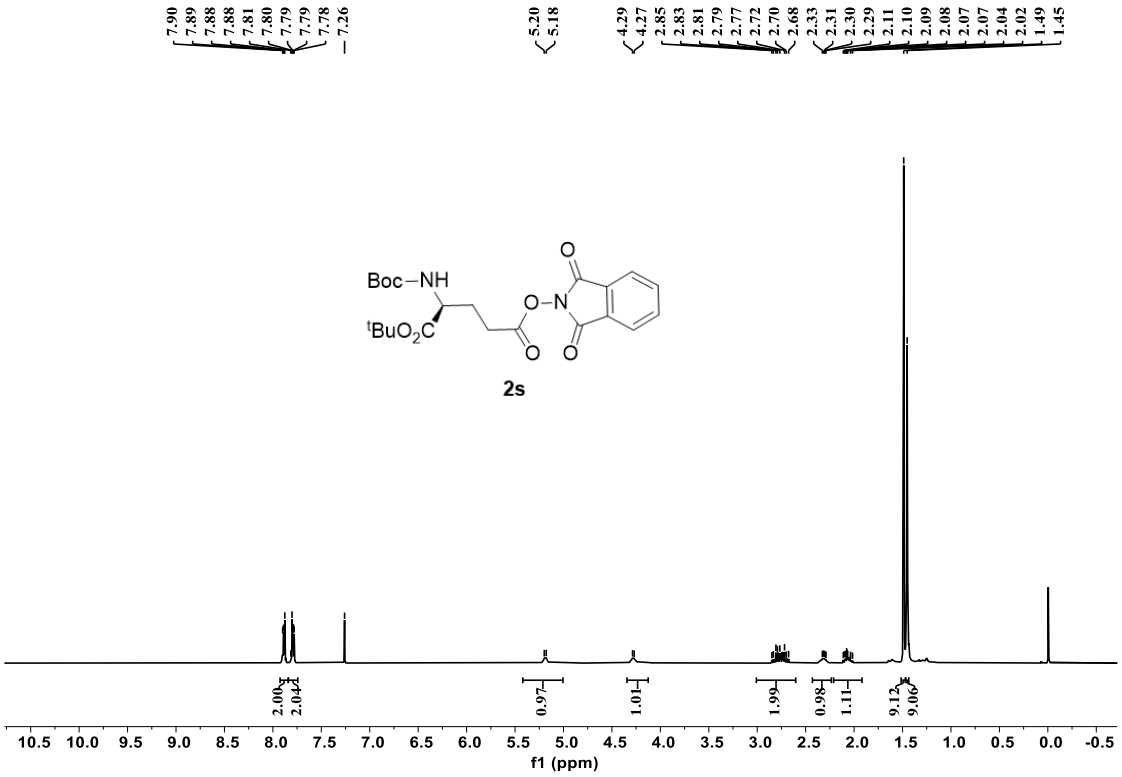

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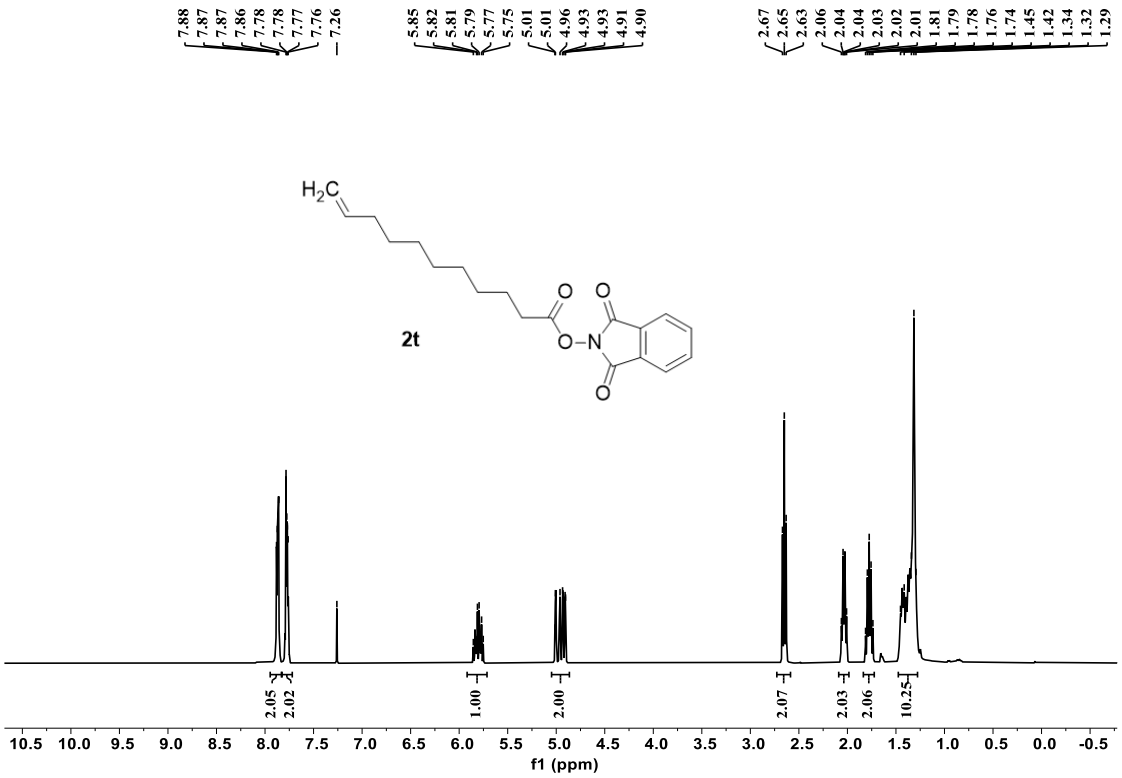

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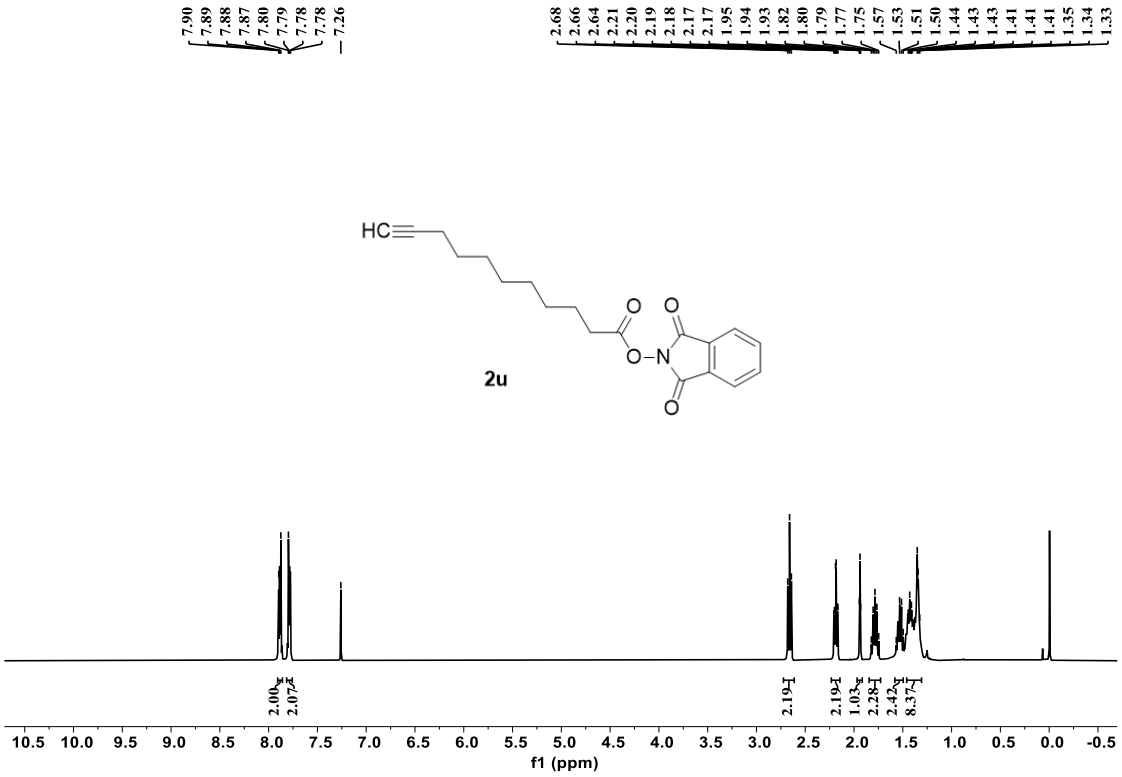

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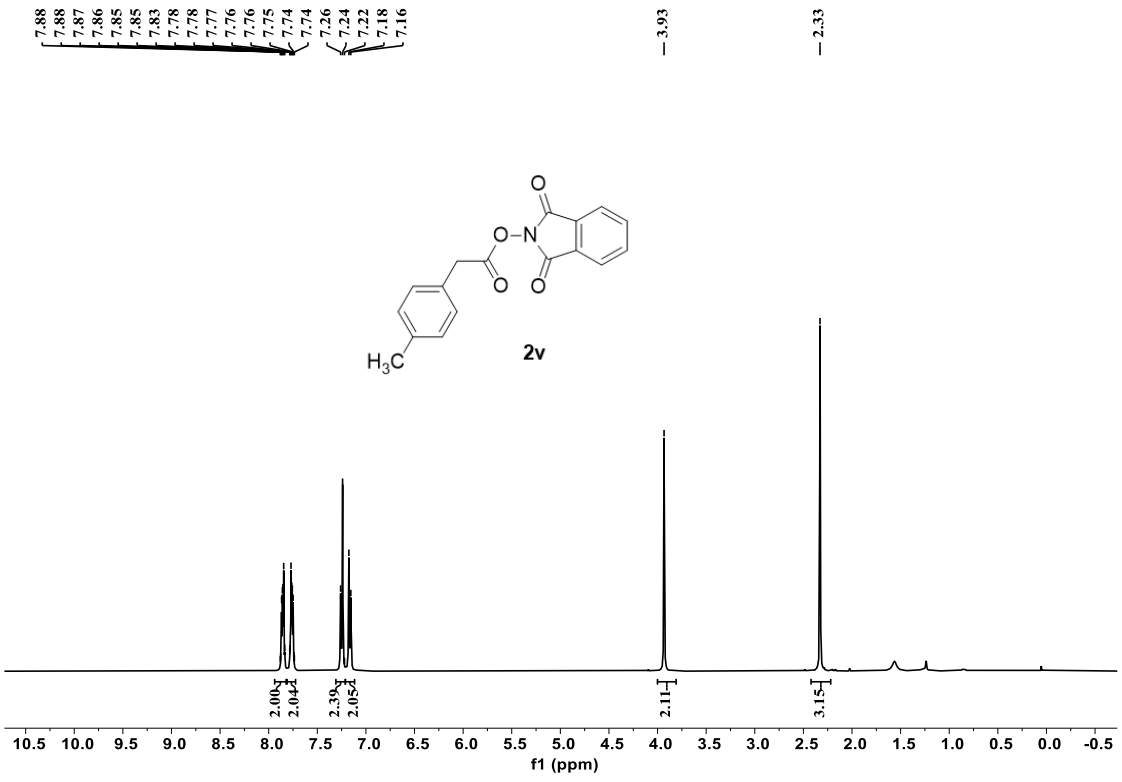

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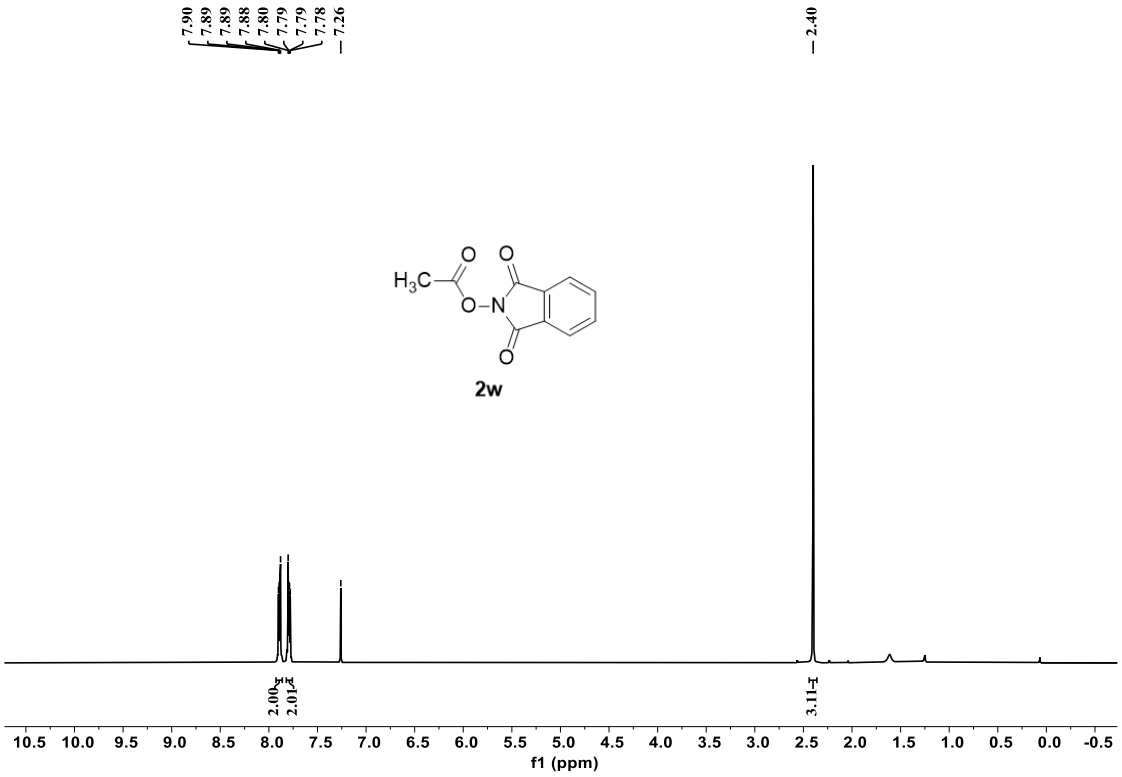

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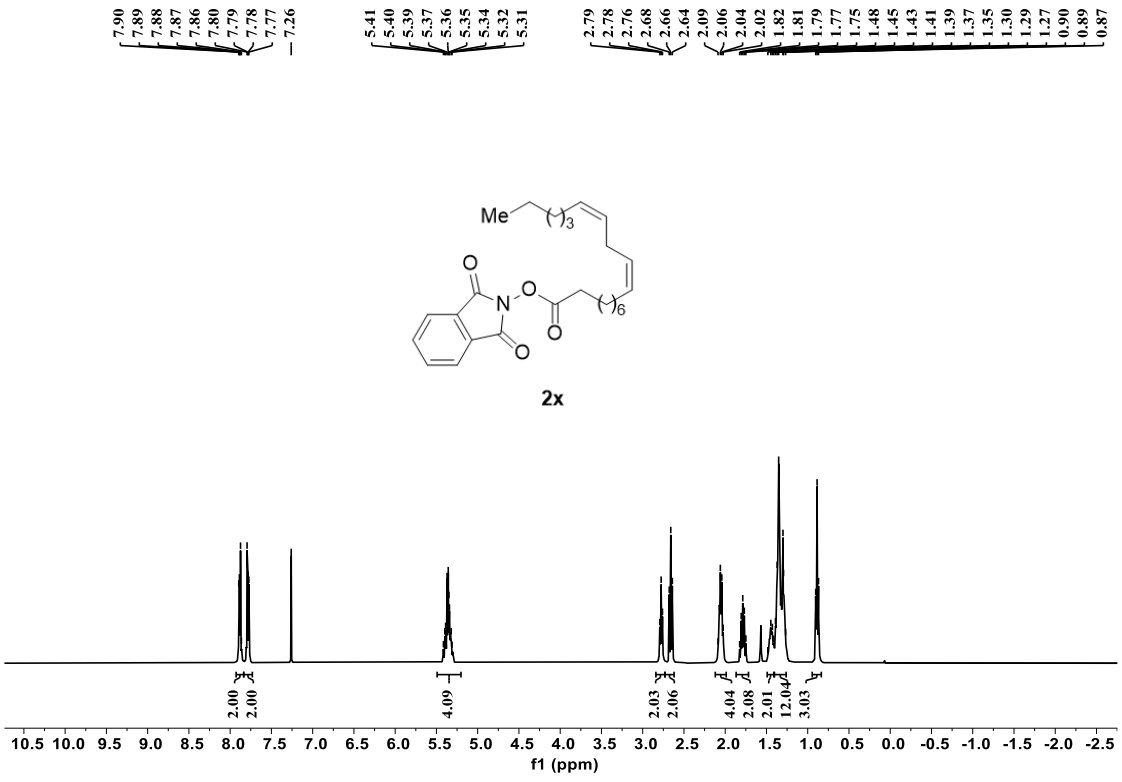

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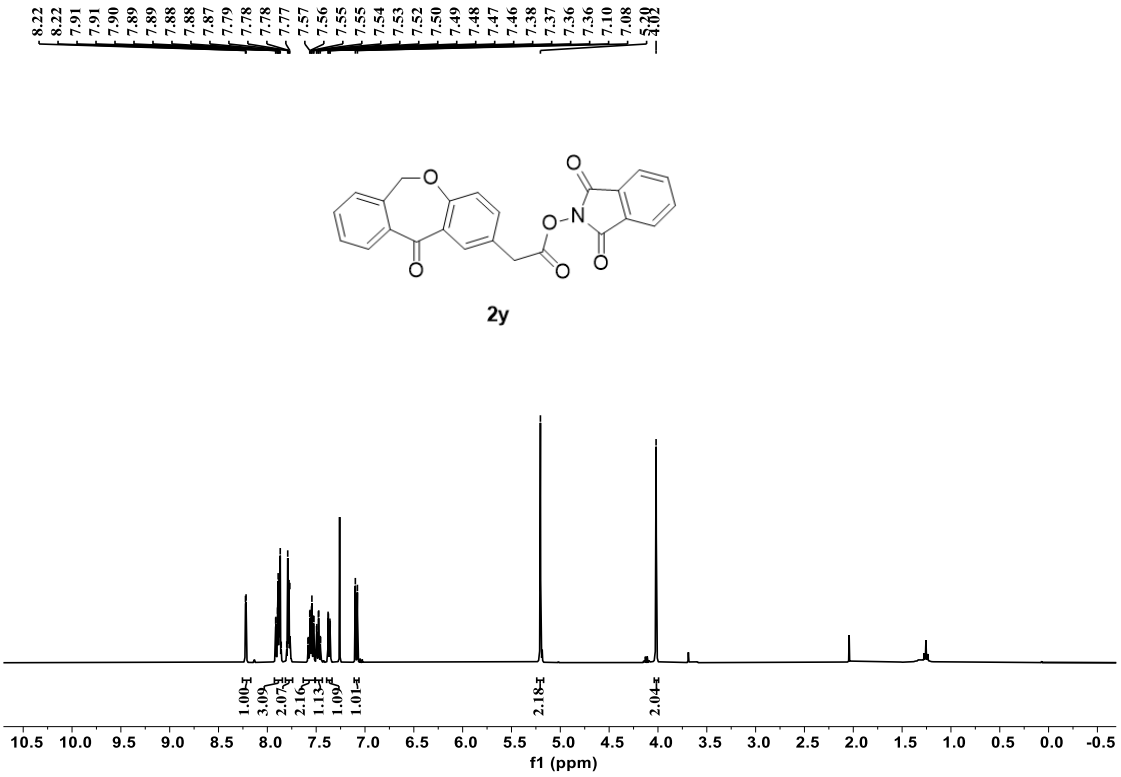

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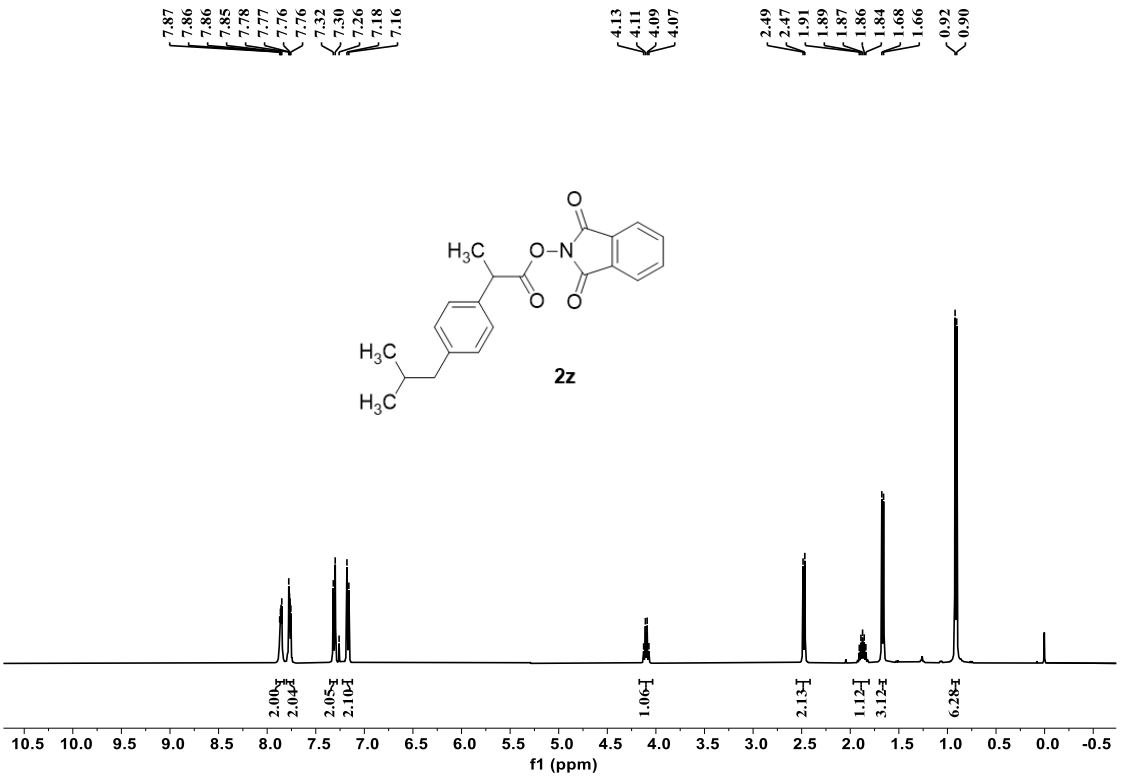

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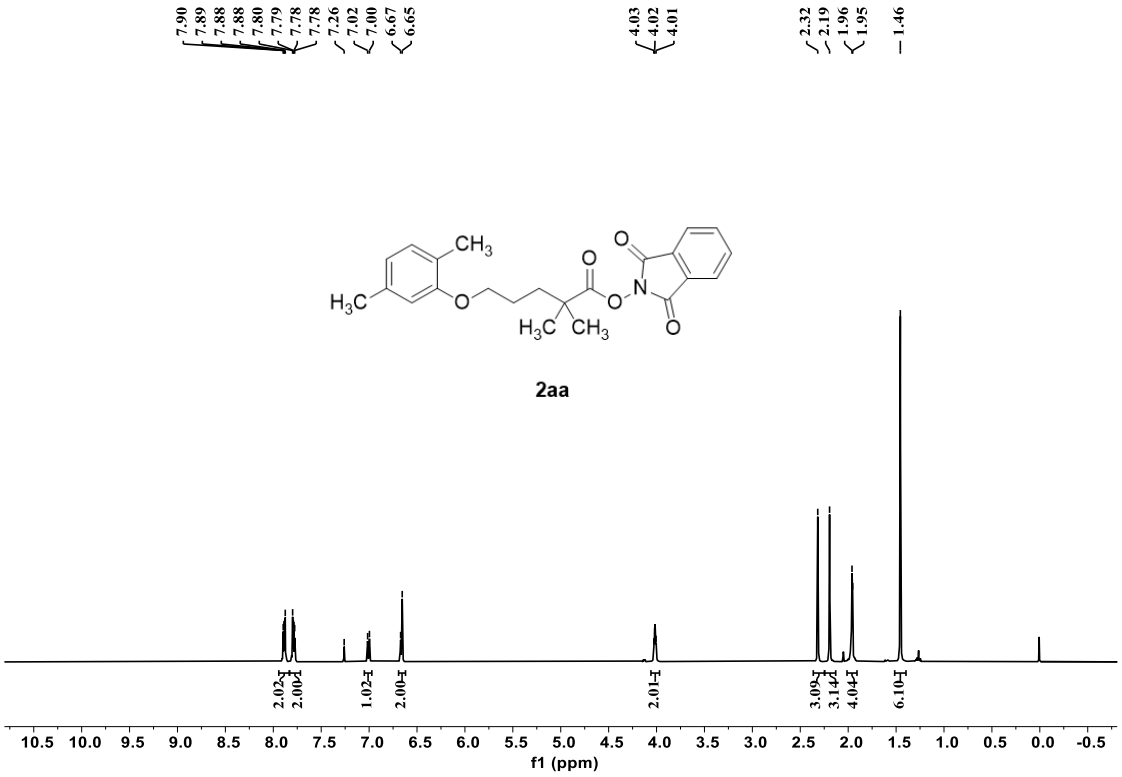

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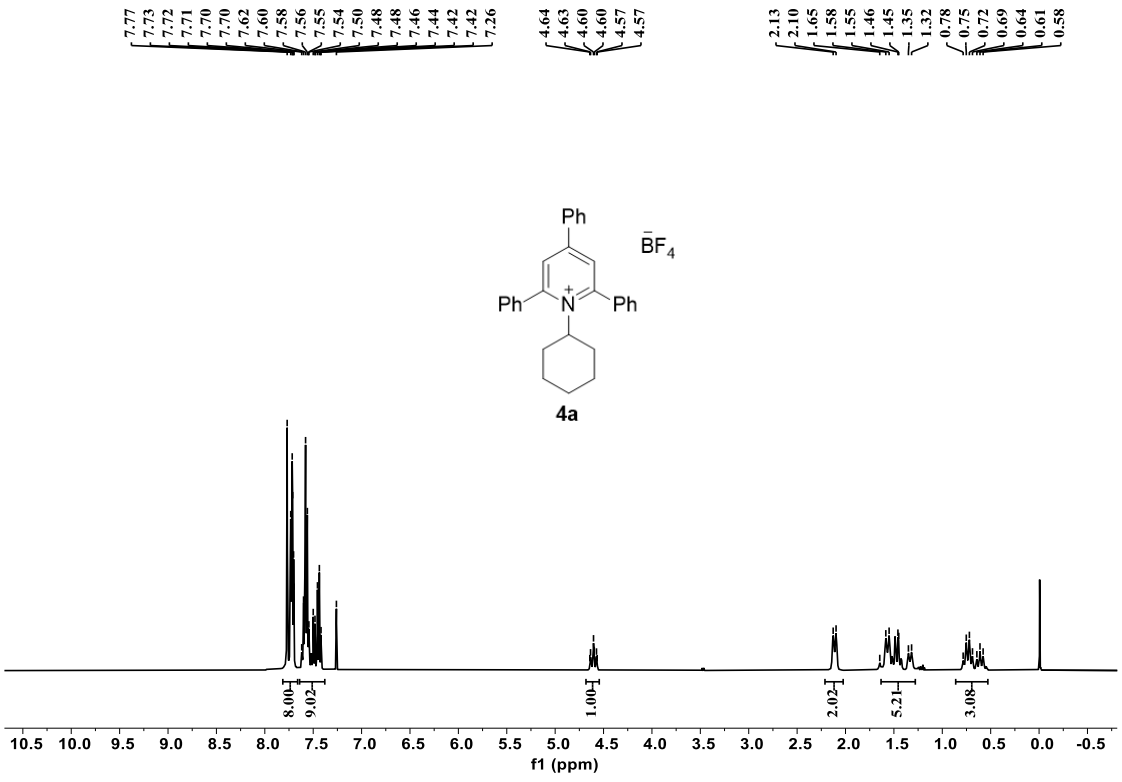

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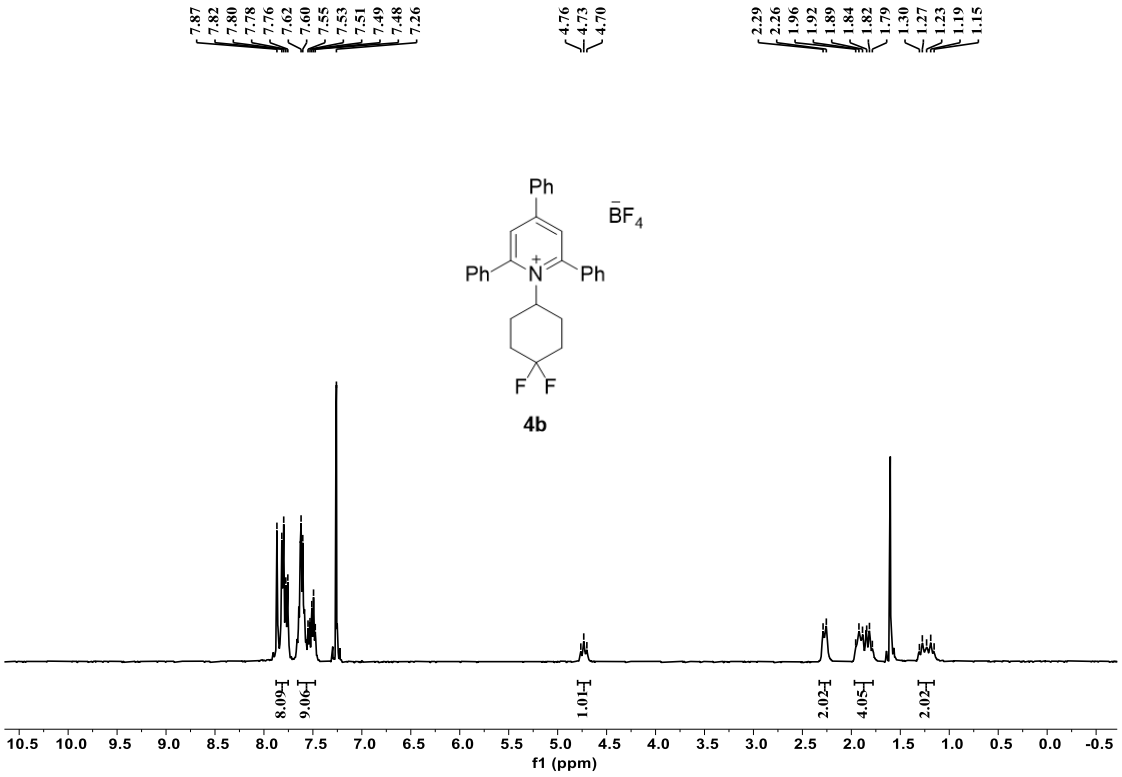

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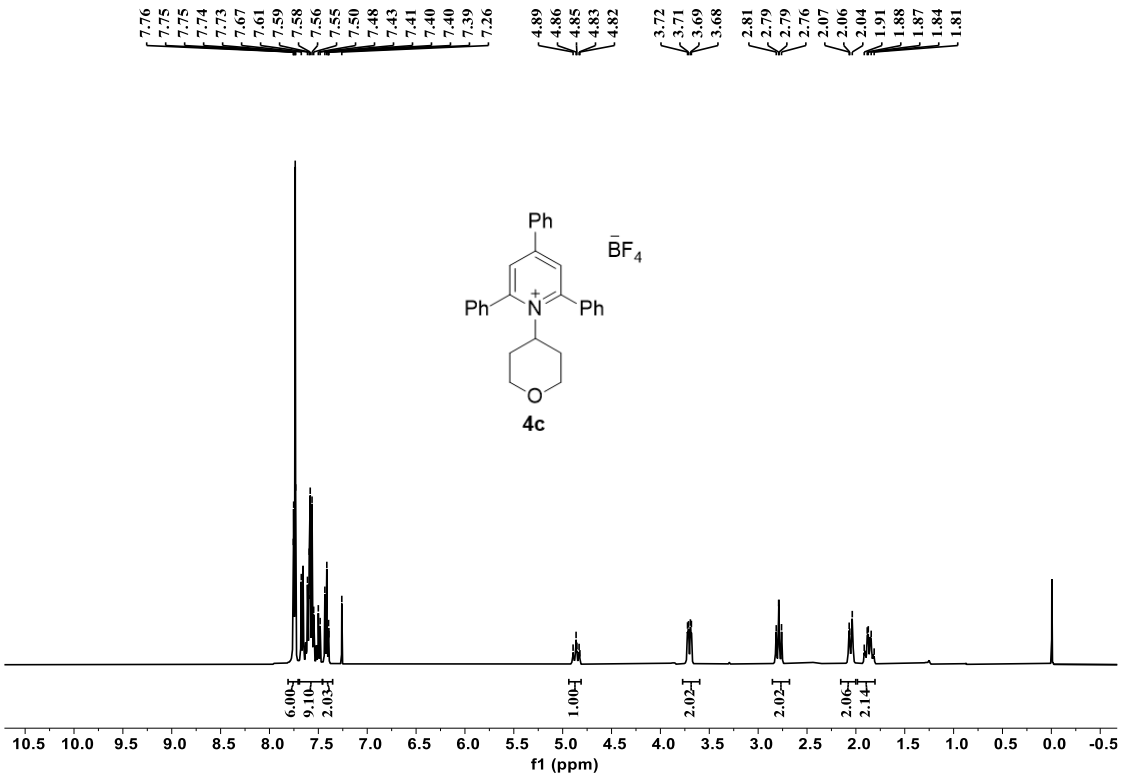
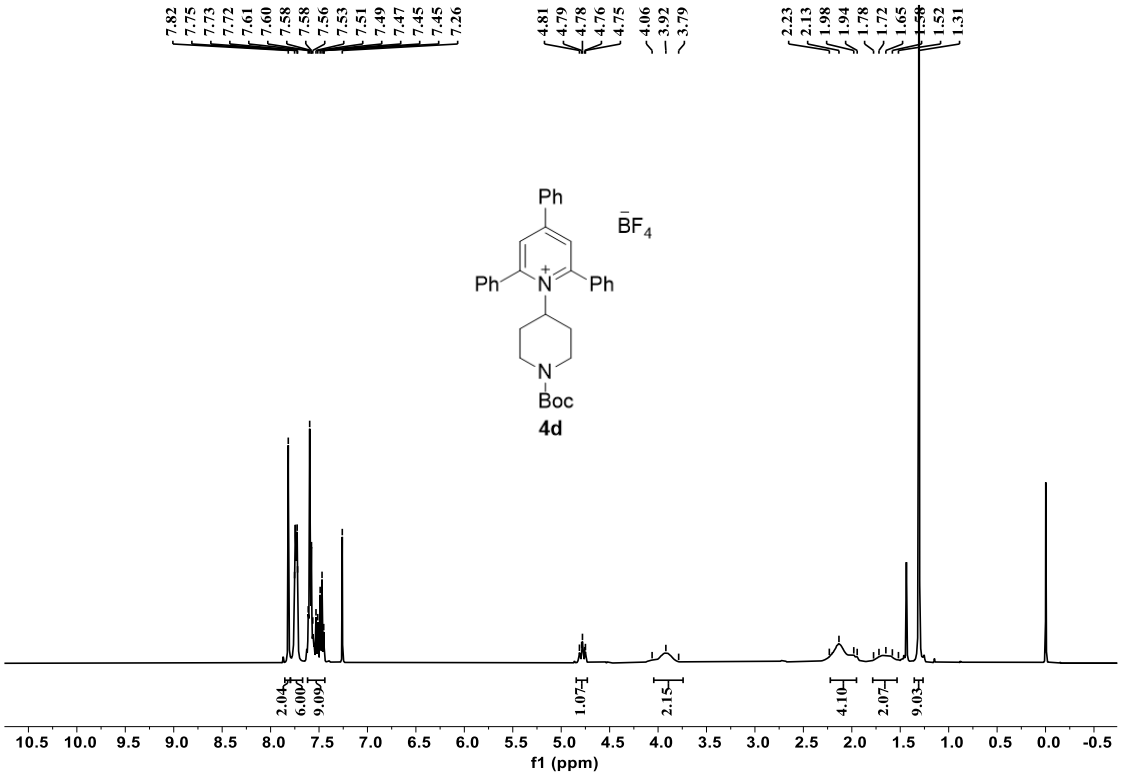


^{1}H NMR (400 MHz, $CDCl_3$)



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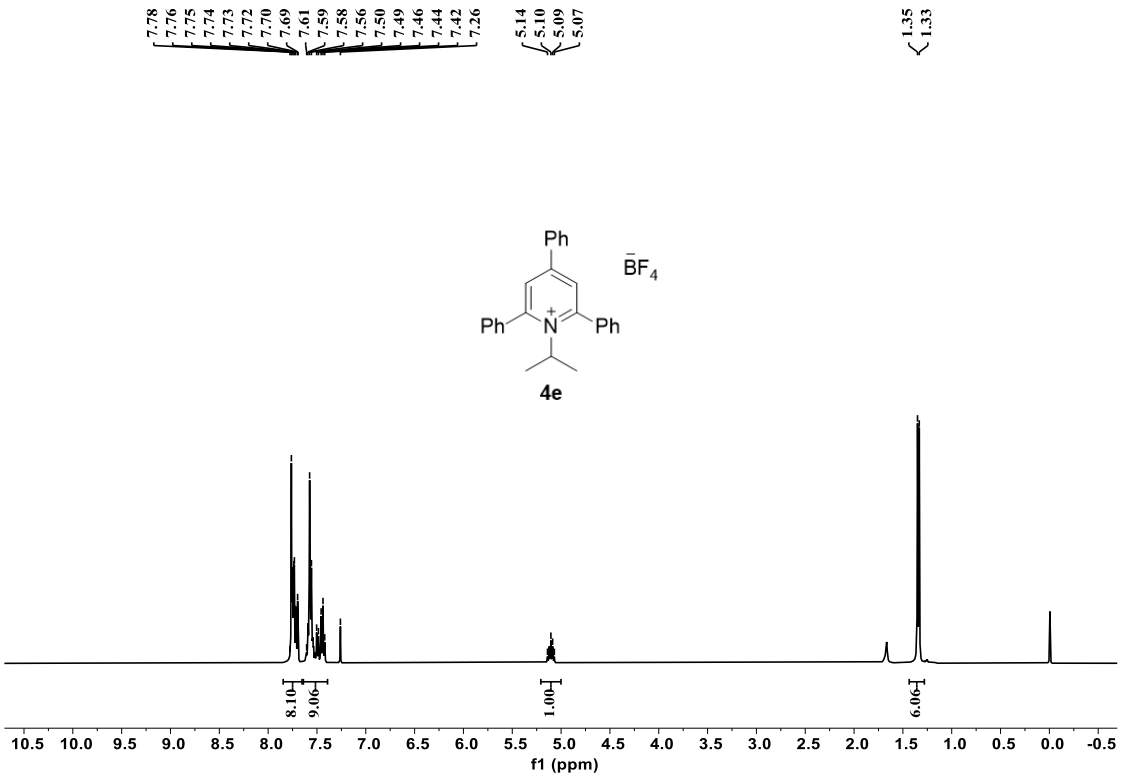
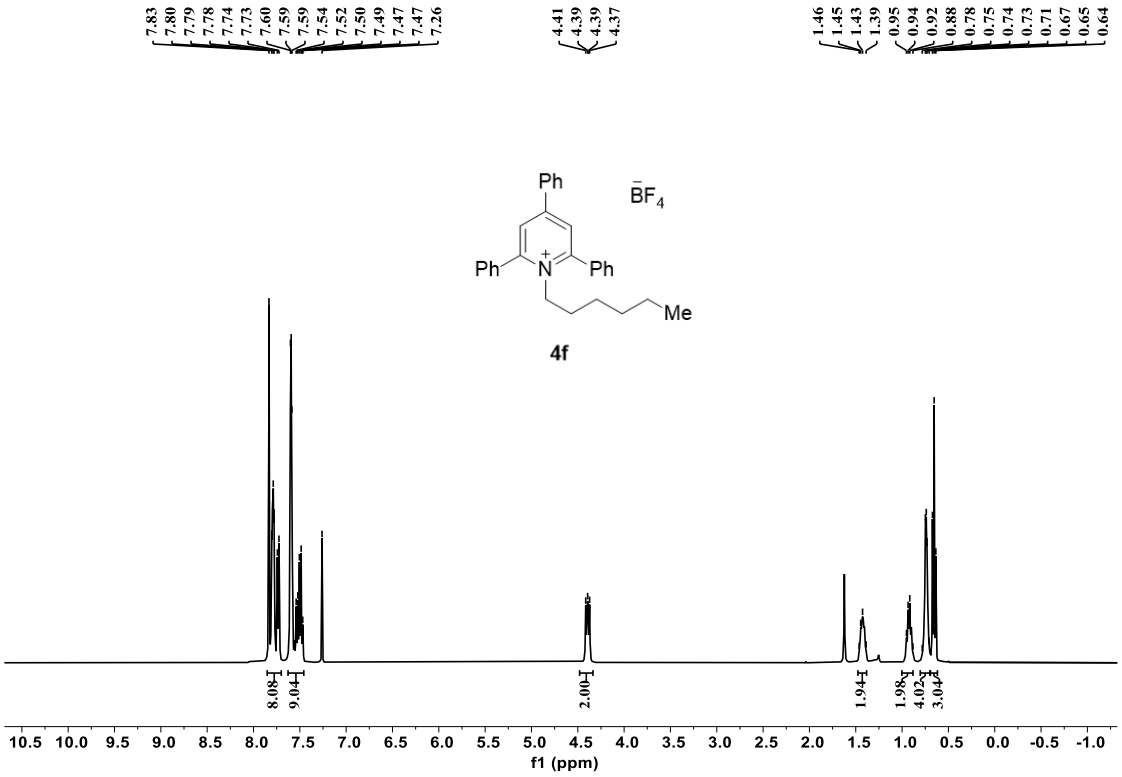
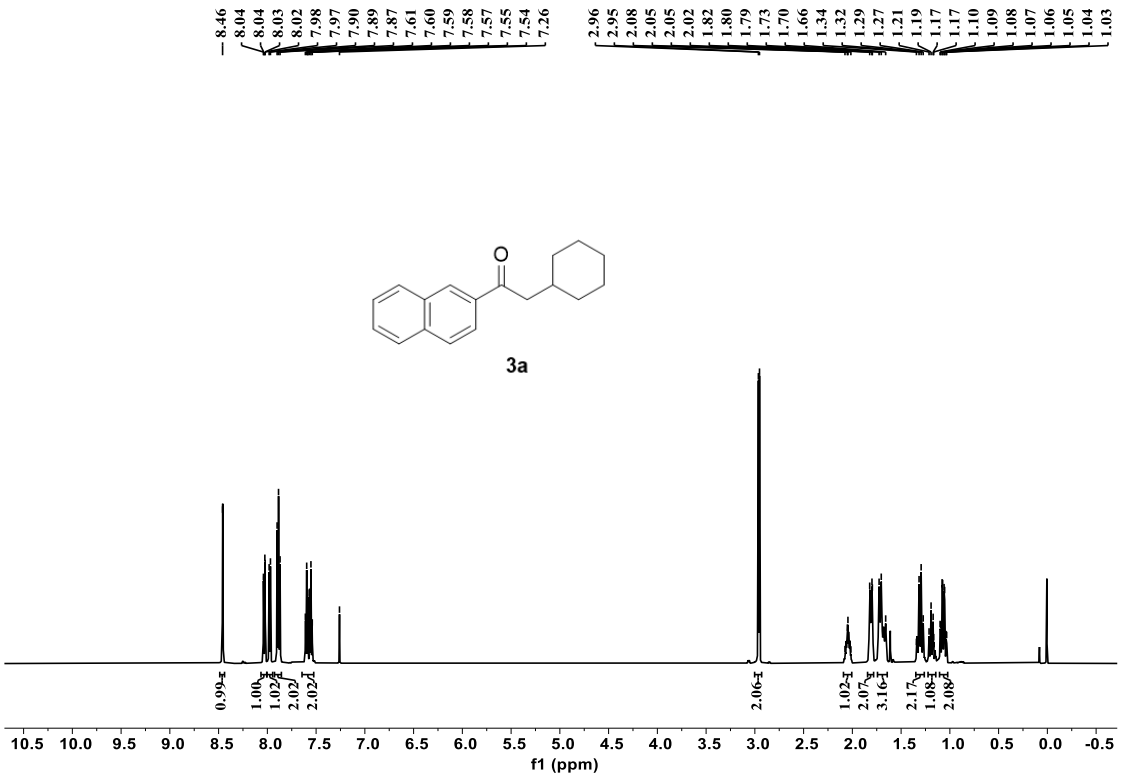


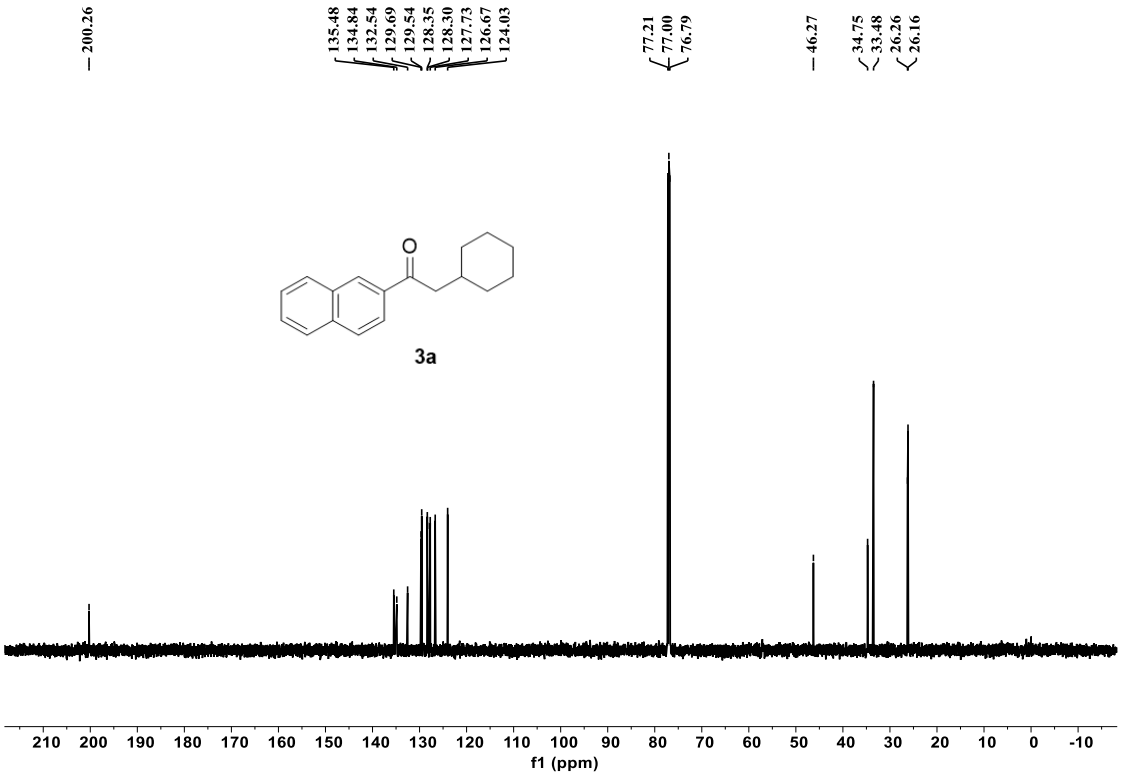
^{1}H NMR (400 MHz, $CDCl_3$)



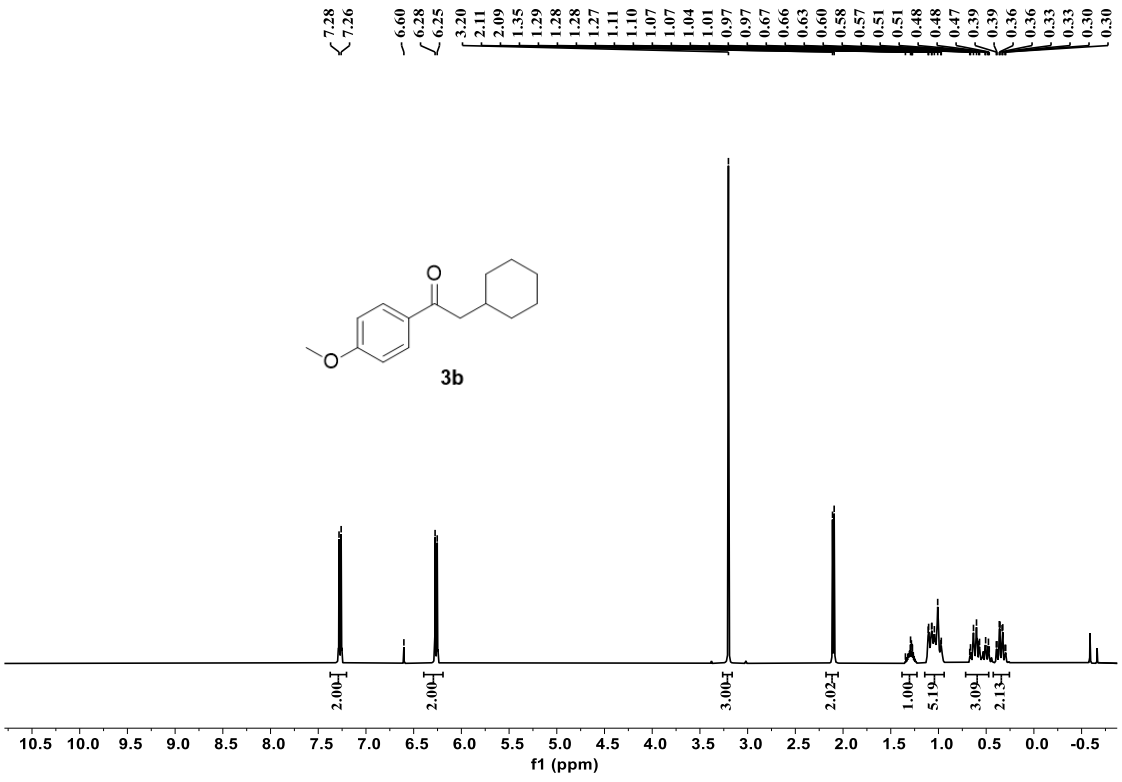
^{1}H NMR (600 MHz, $CDCl_3$)



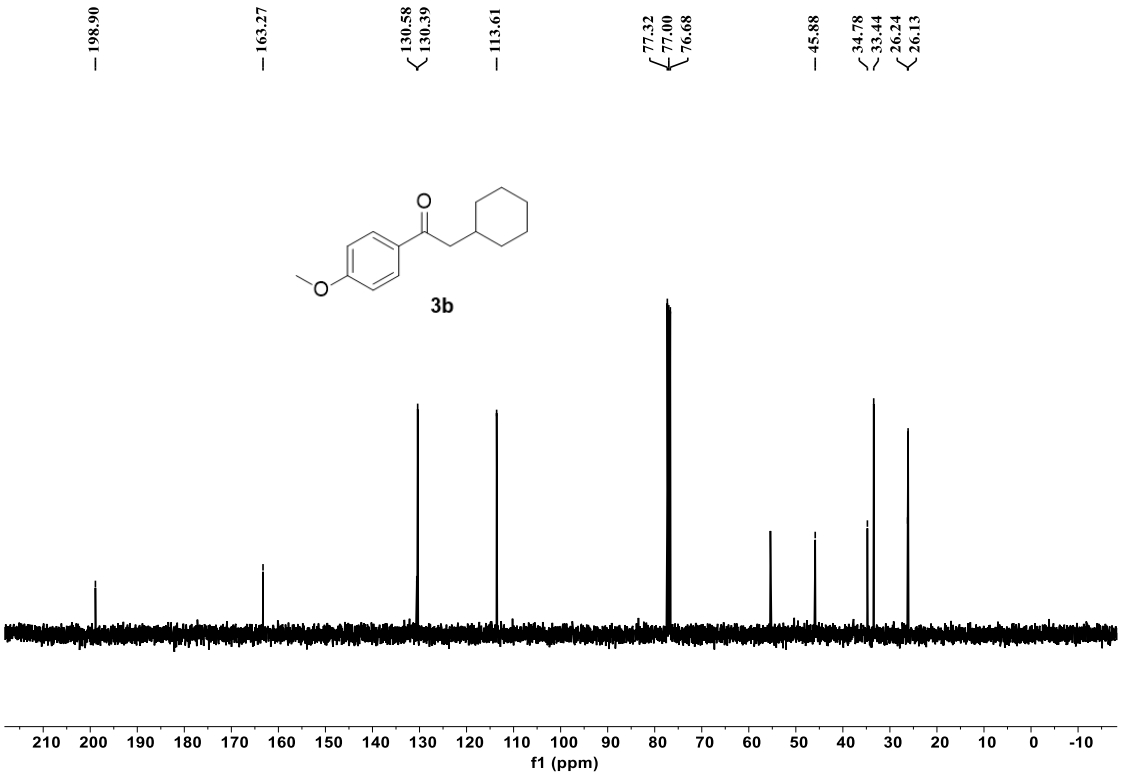
^{13}C NMR (151 MHz, $CDCl_3$)



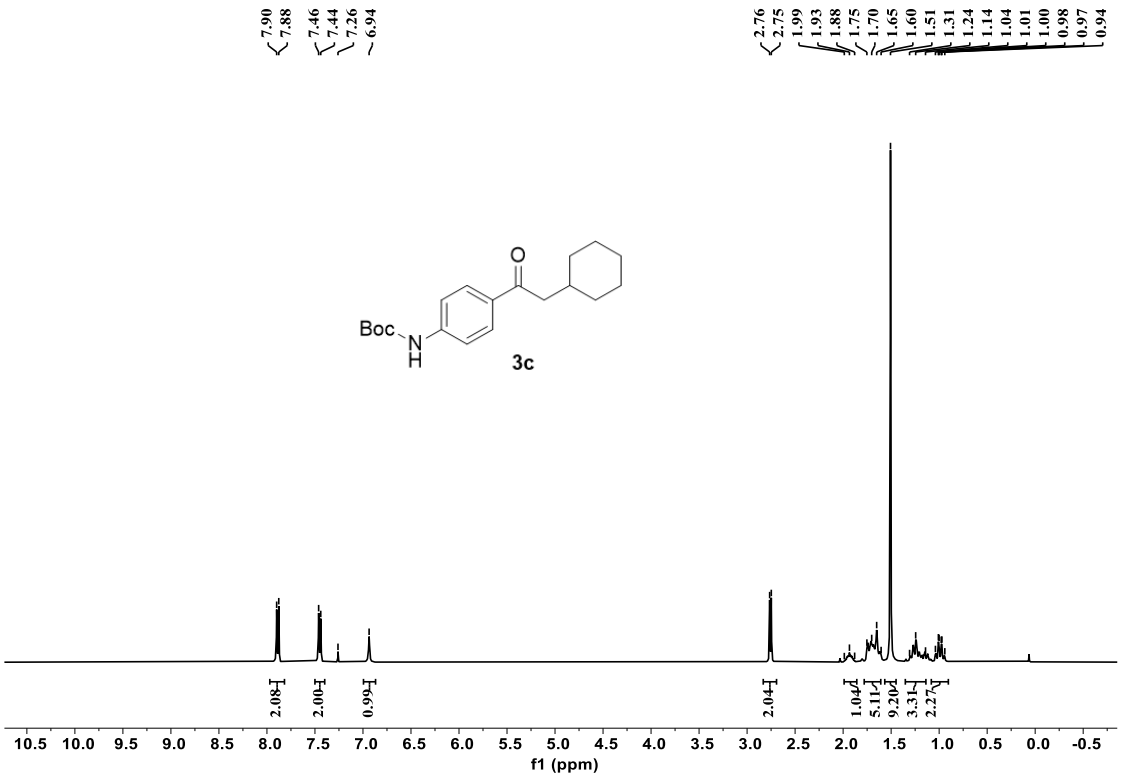
^{1}H NMR (400 MHz, $CDCl_3$)



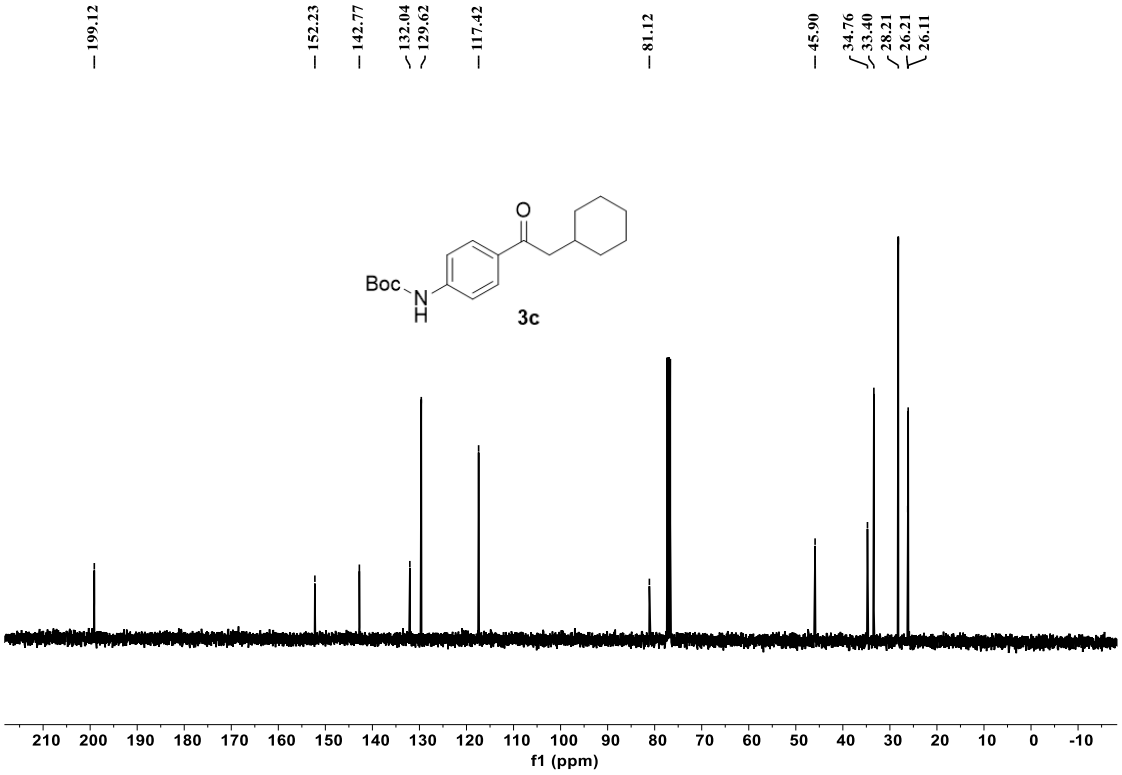
^{13}C NMR (101 MHz, $CDCl_3$)



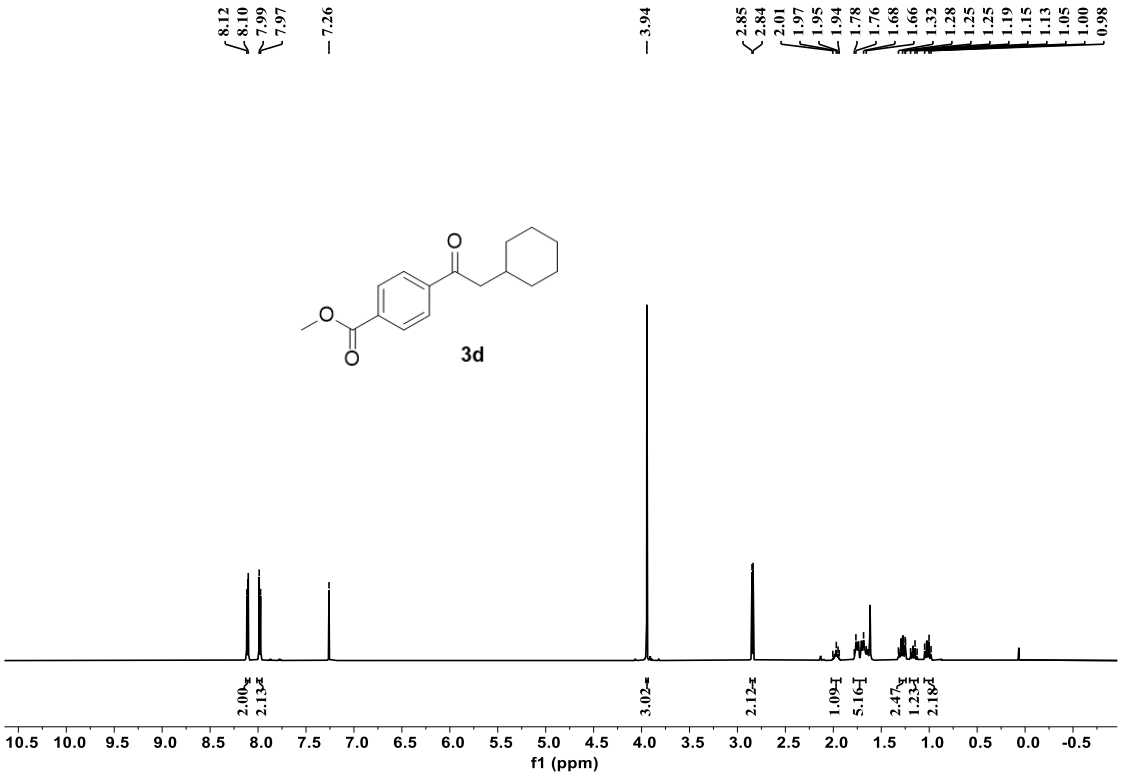
^{1}H NMR (400 MHz, $CDCl_3$)



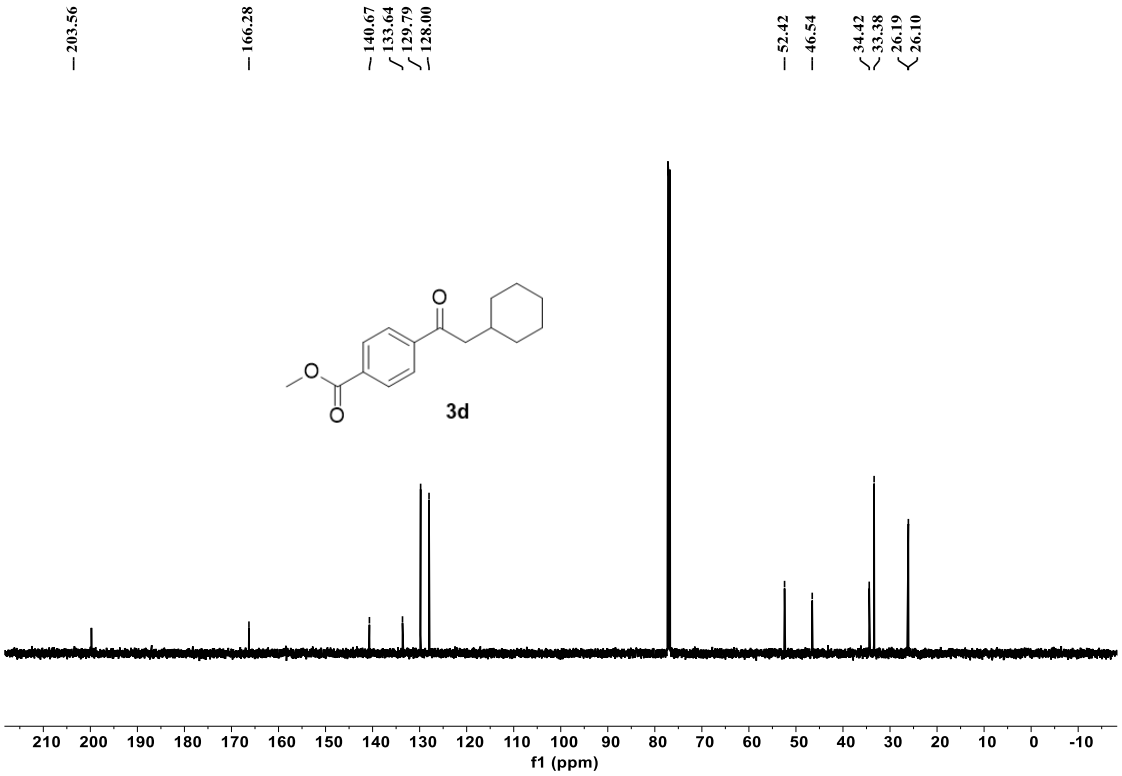
^{13}C NMR (101 MHz, $CDCl_3$)



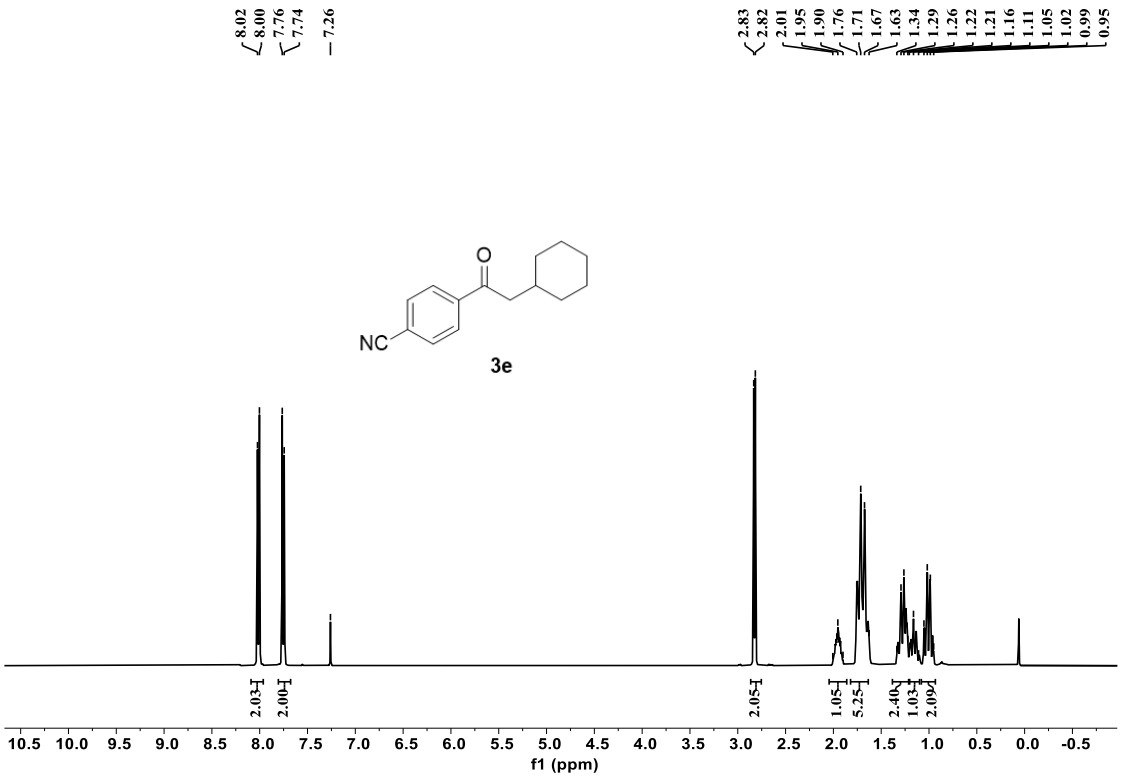
^{1}H NMR (600 MHz, $CDCl_3$)



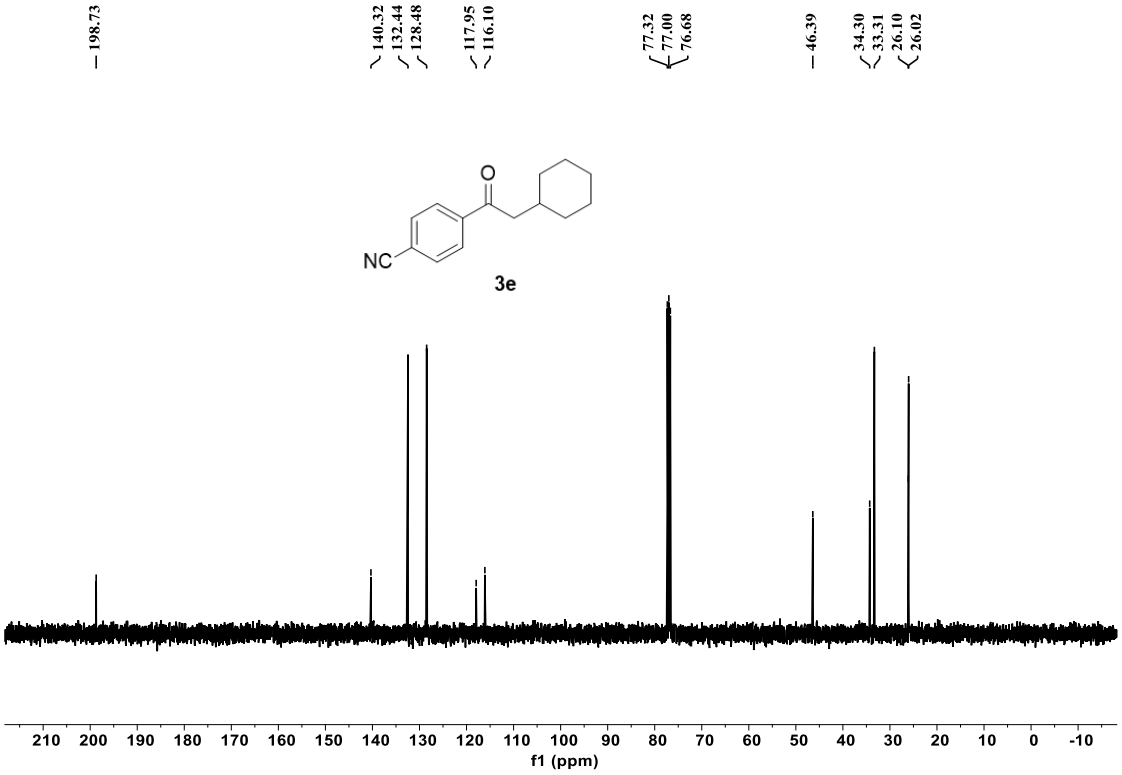
^{13}C NMR (151 MHz, $CDCl_3$)



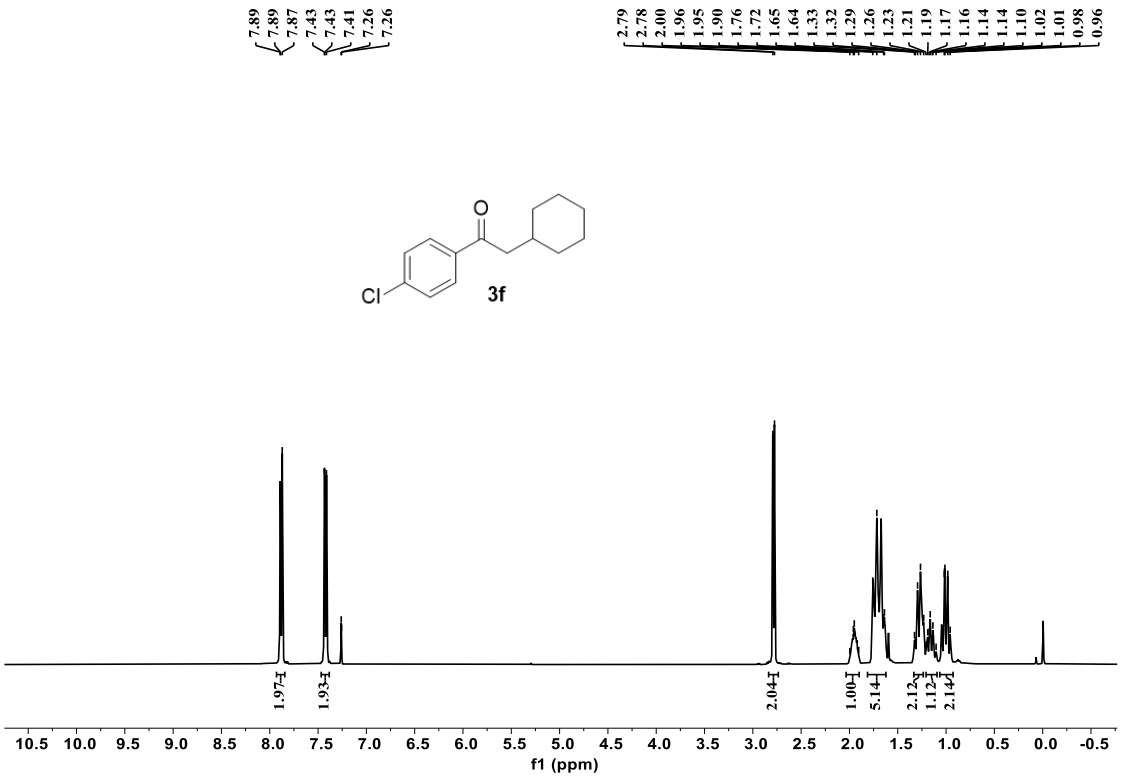
^{1}H NMR (400 MHz, $CDCl_3$)



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



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



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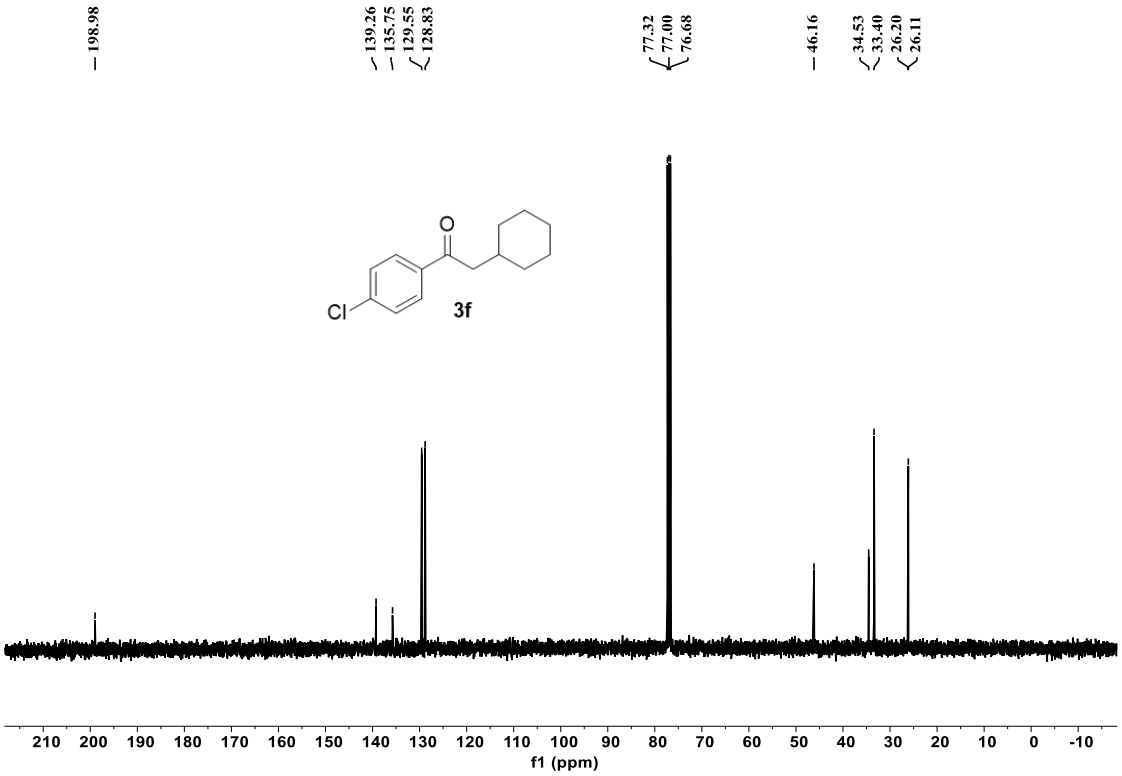

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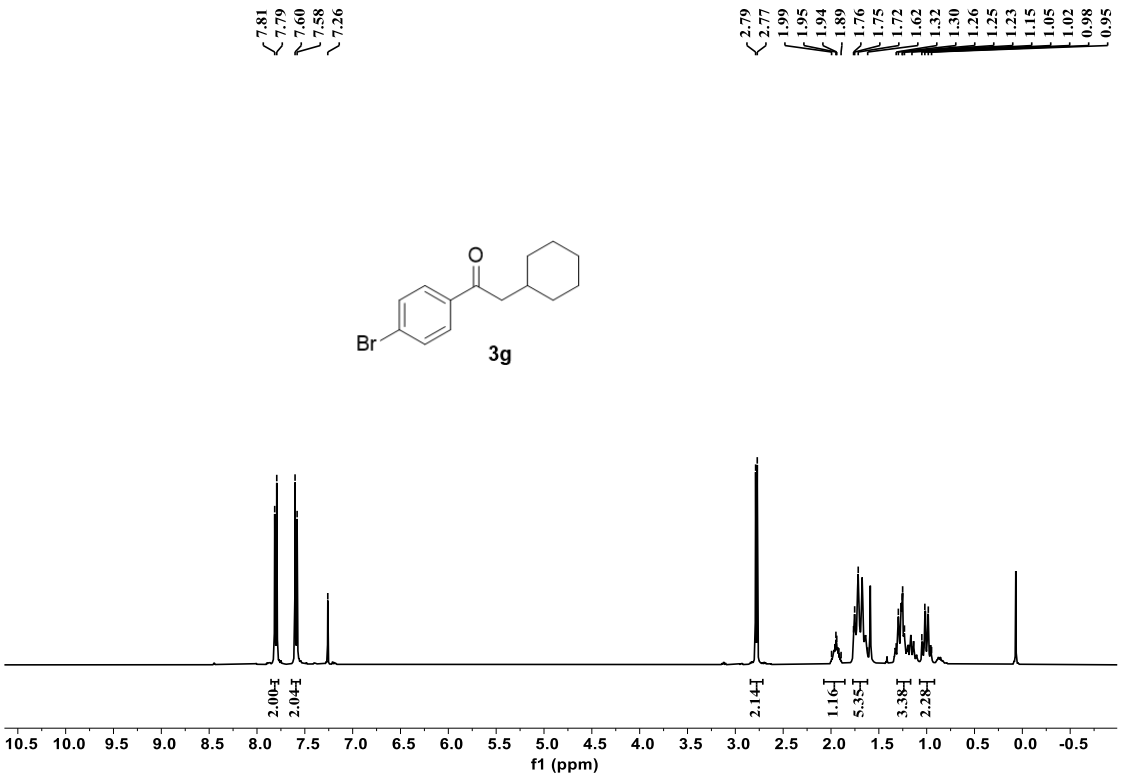

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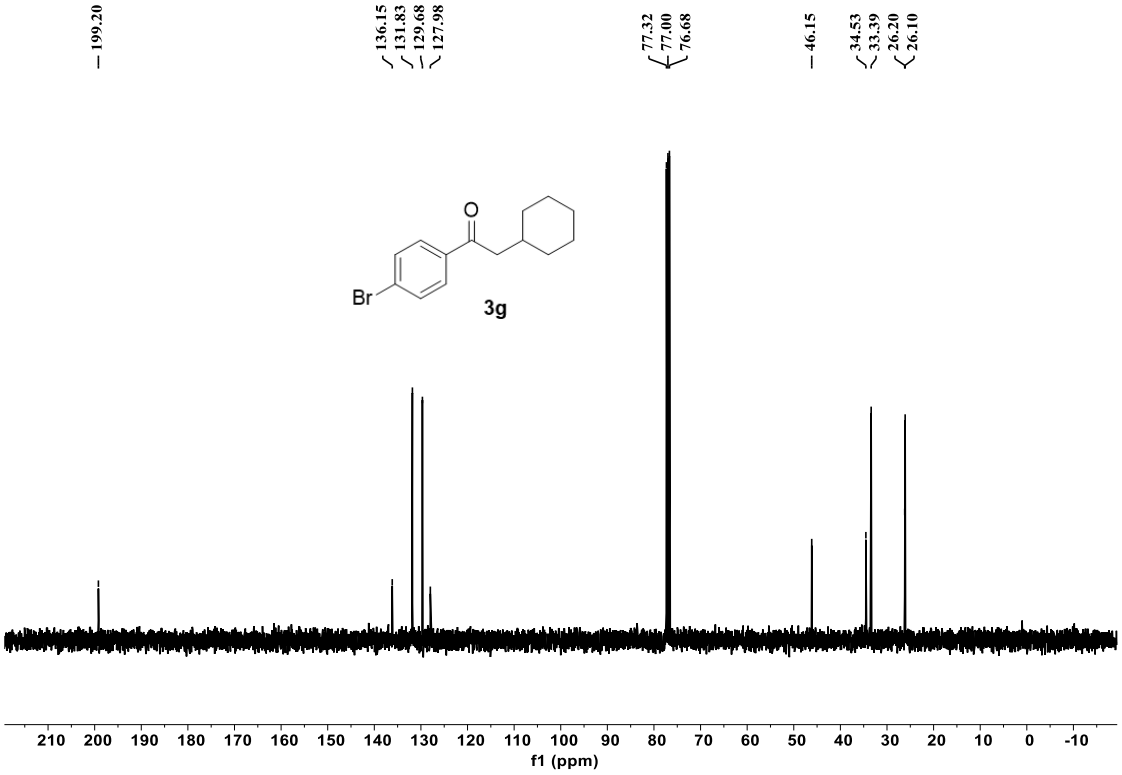
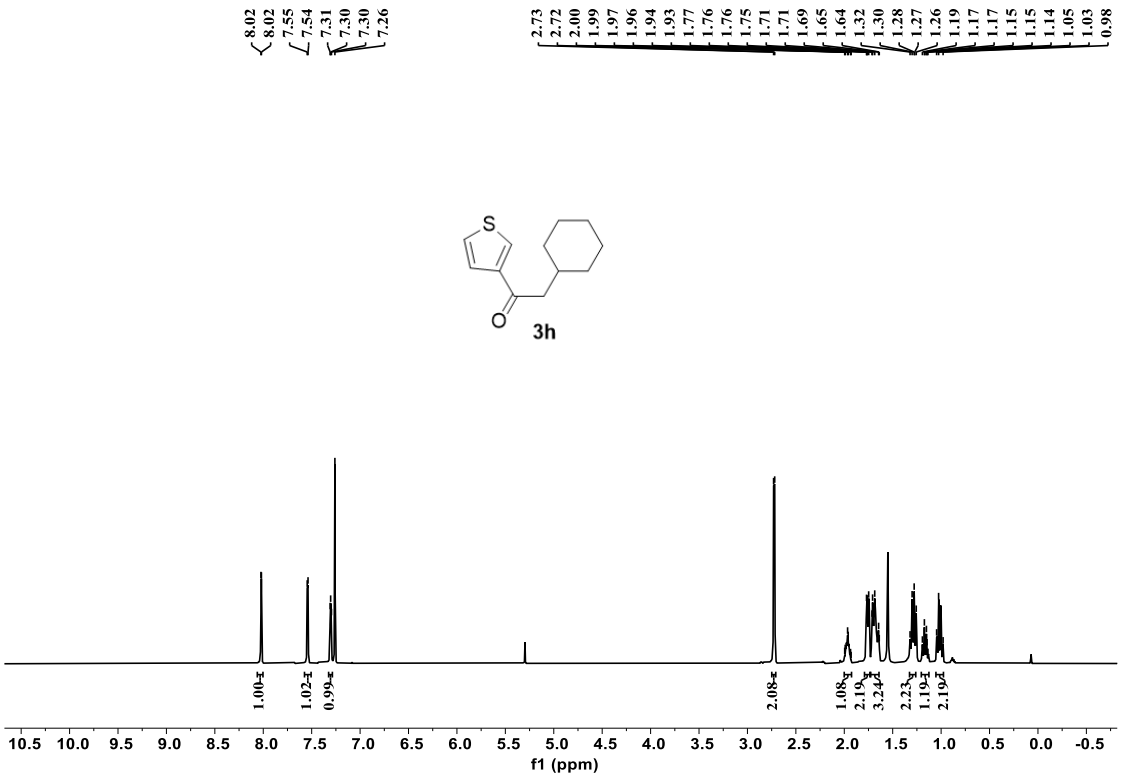
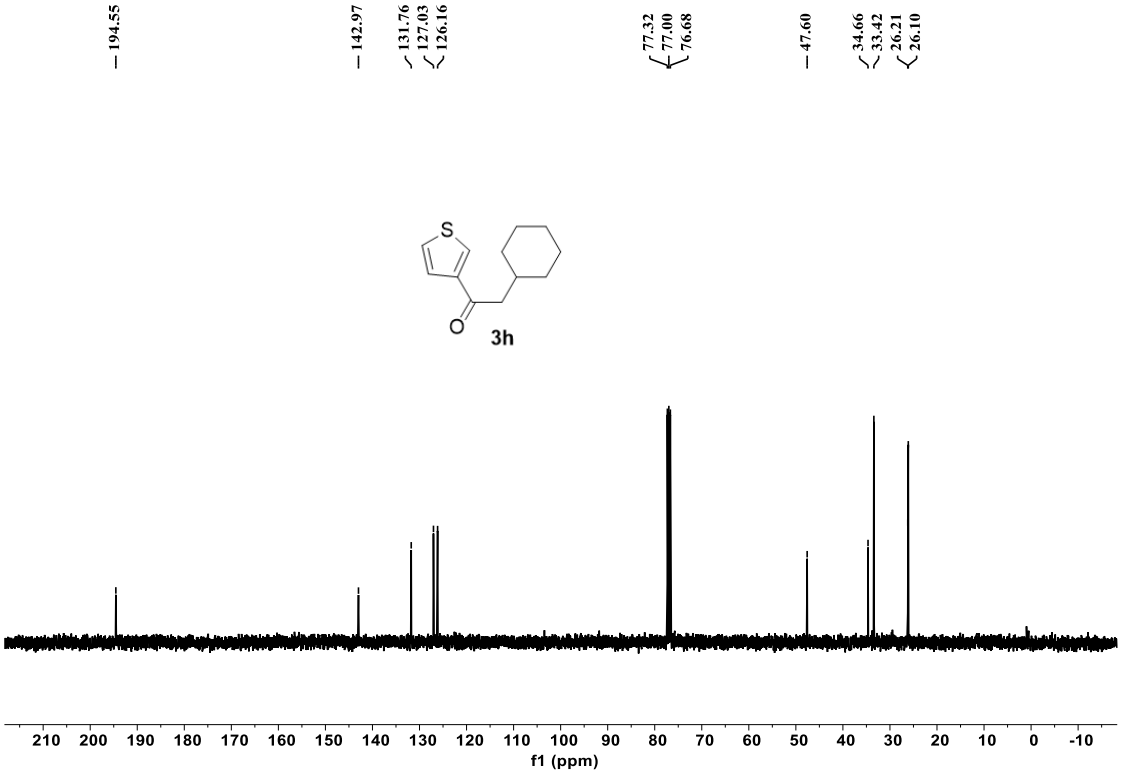


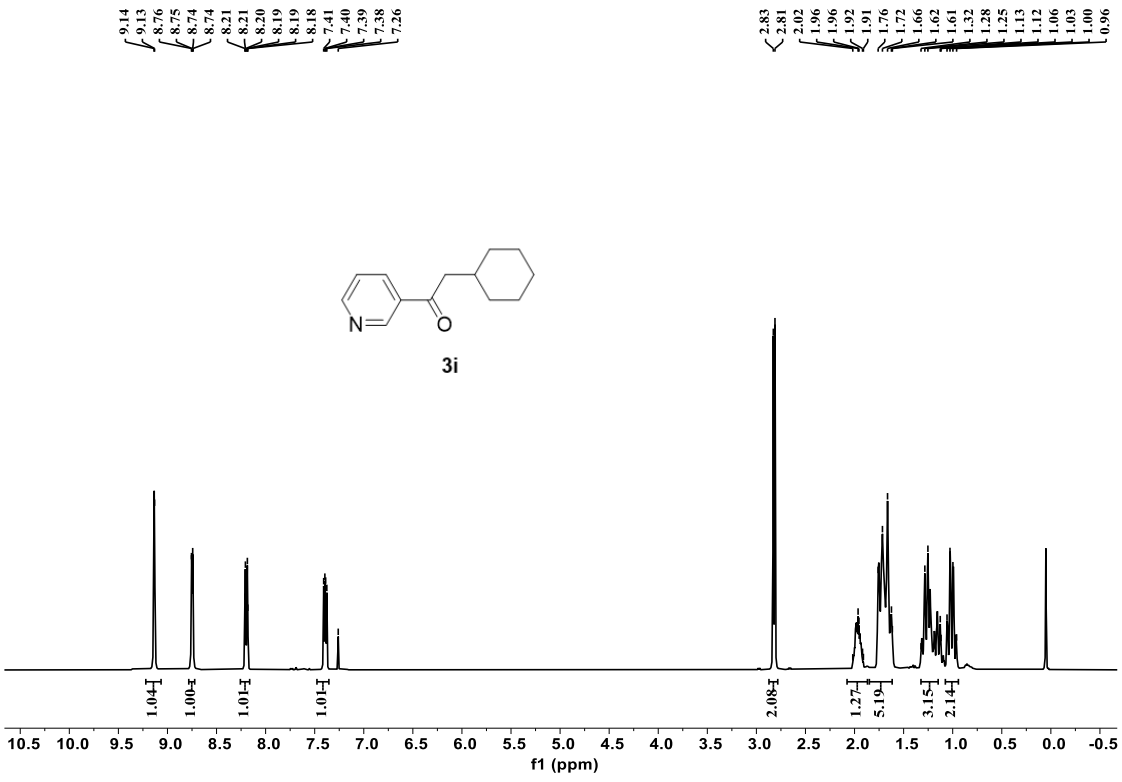
^{1}H NMR (600 MHz, $CDCl_3$)



^{13}C NMR (151 MHz, $CDCl_3$)



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



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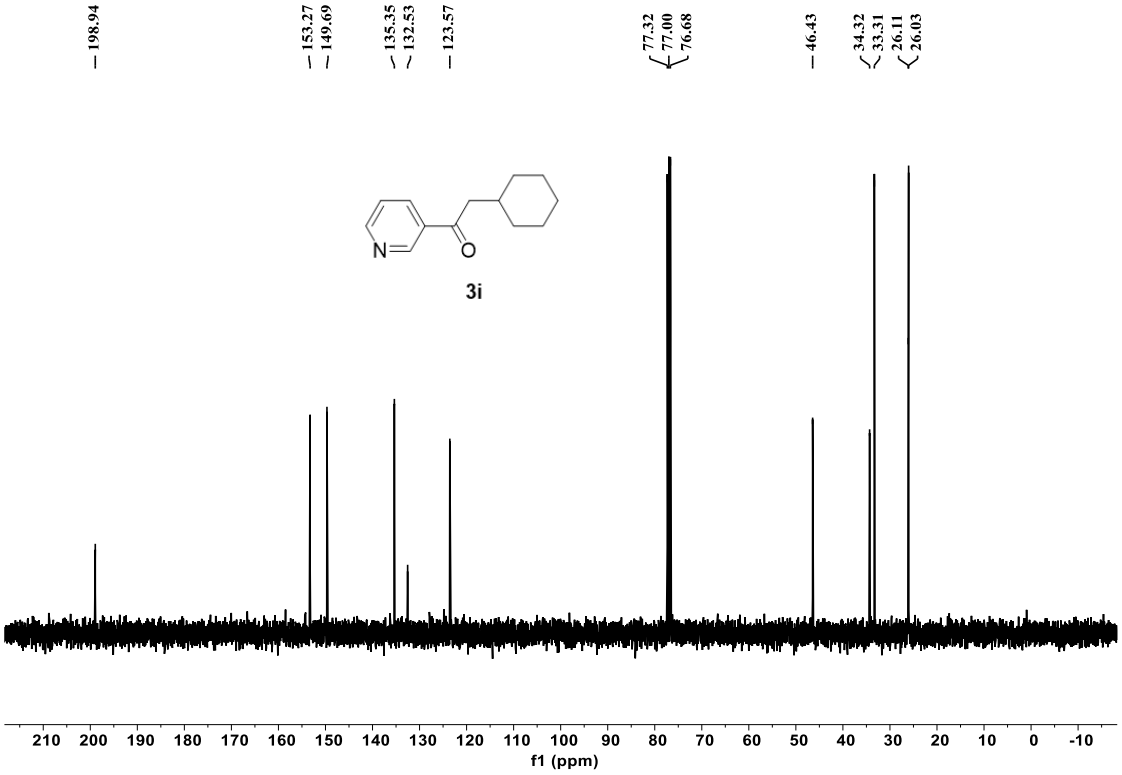

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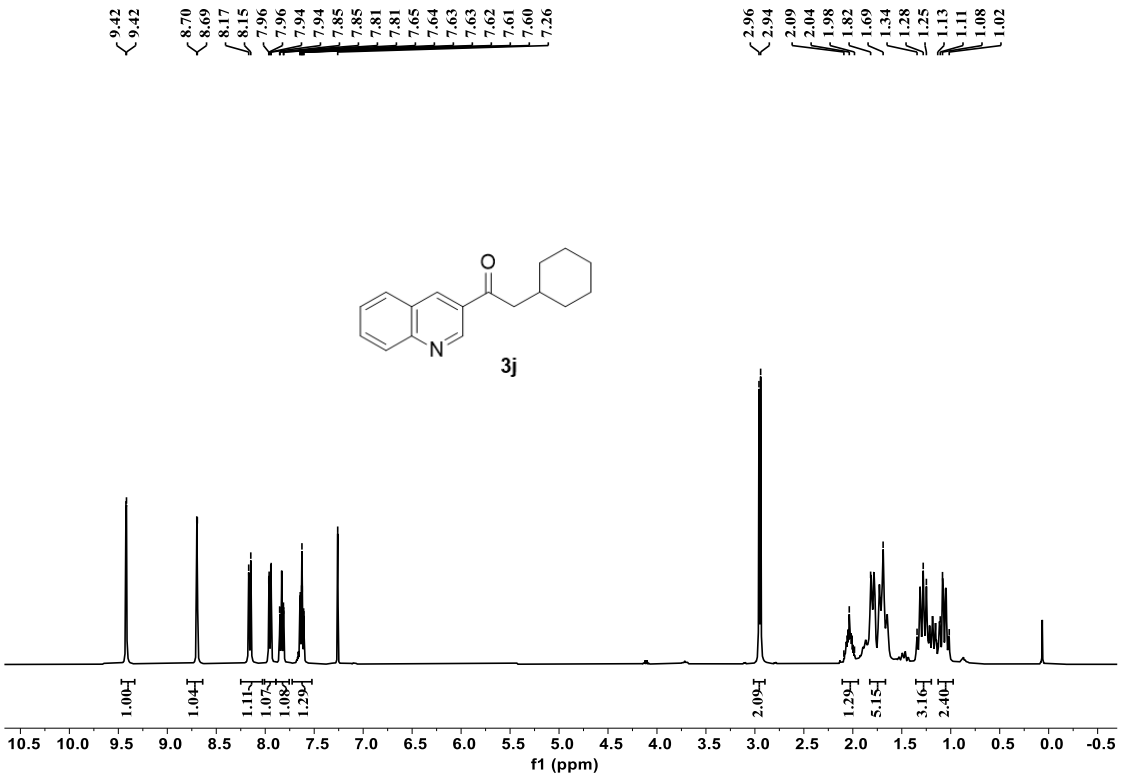

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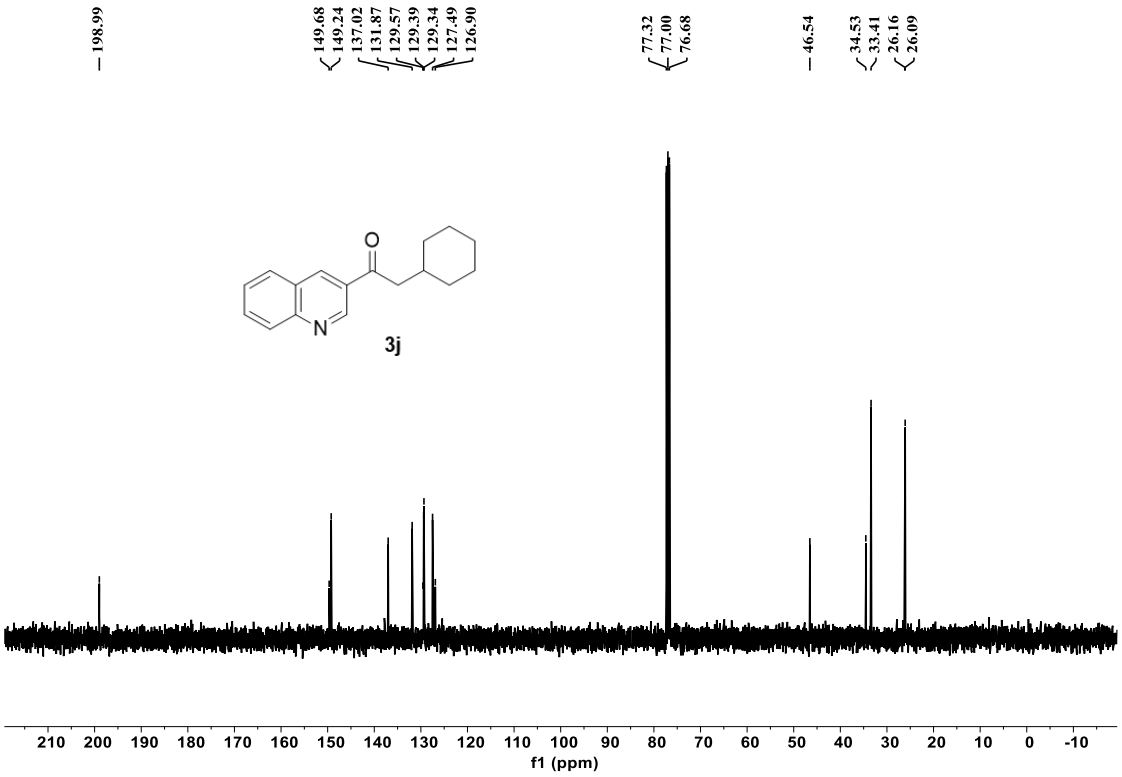

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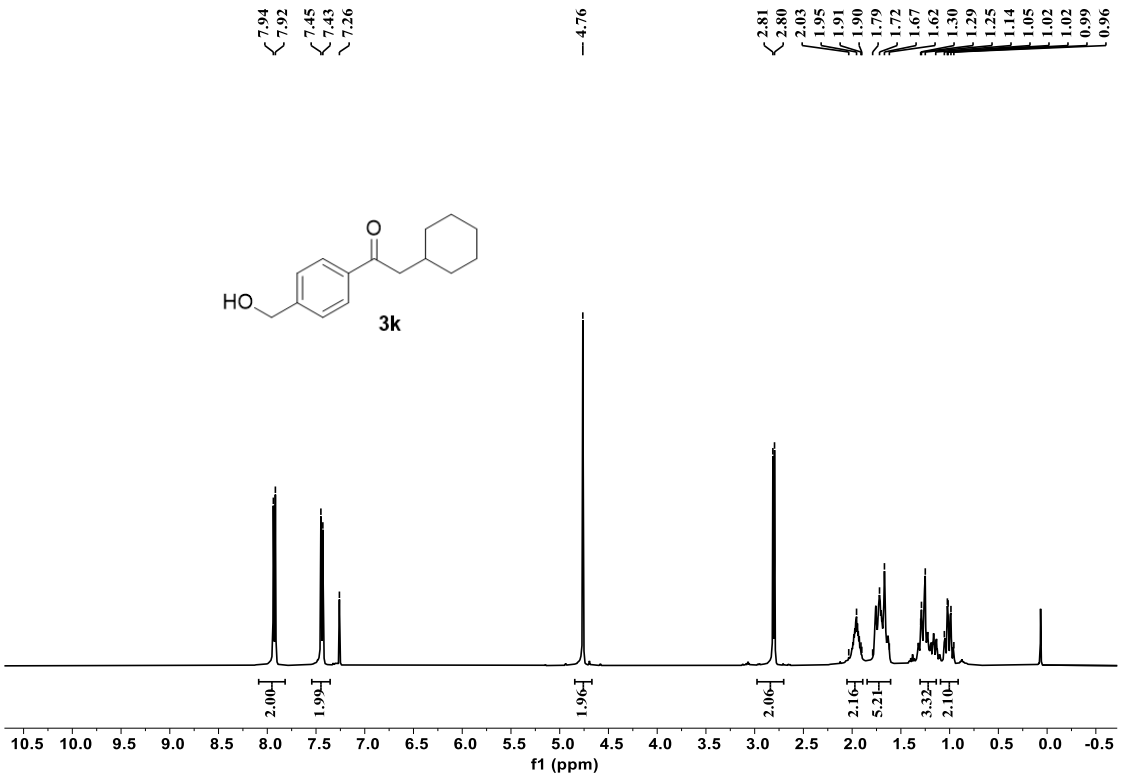

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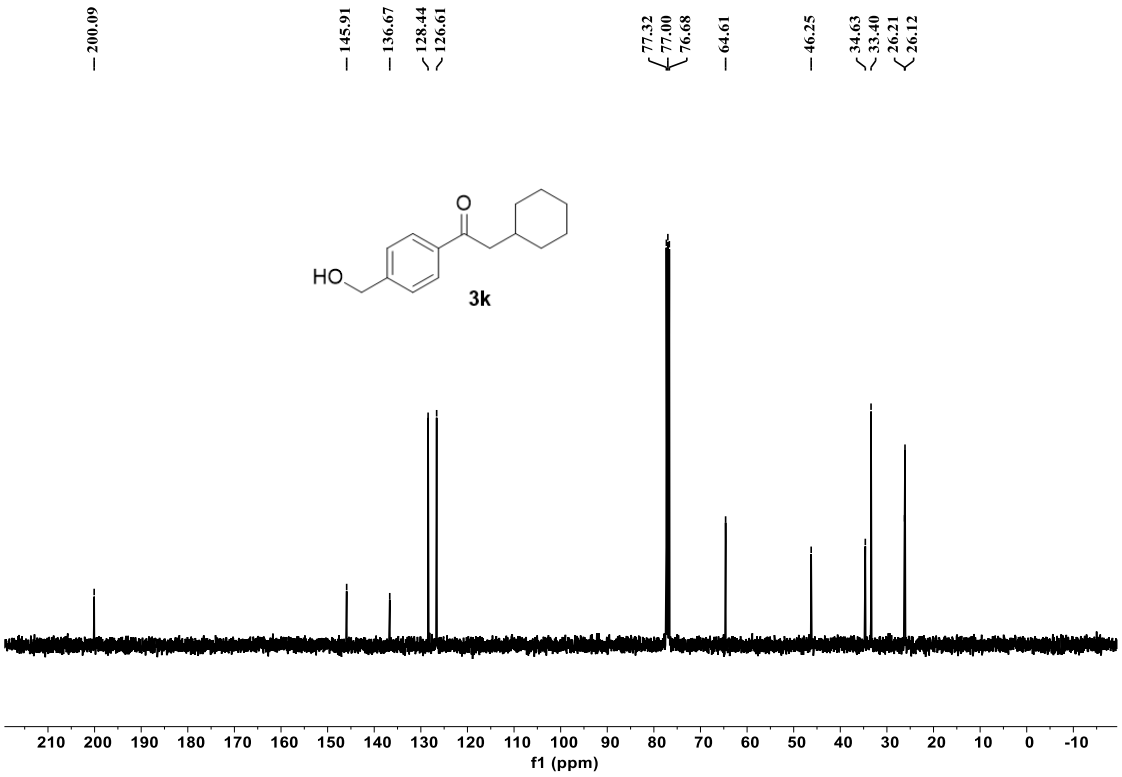

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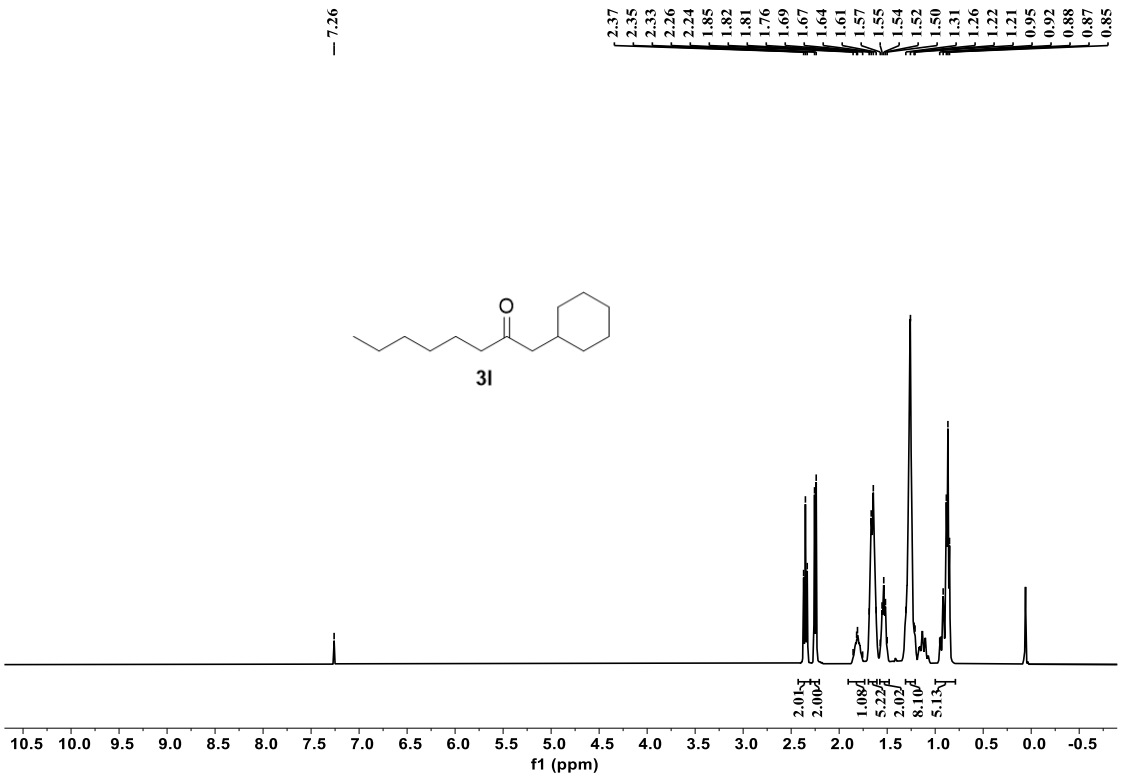

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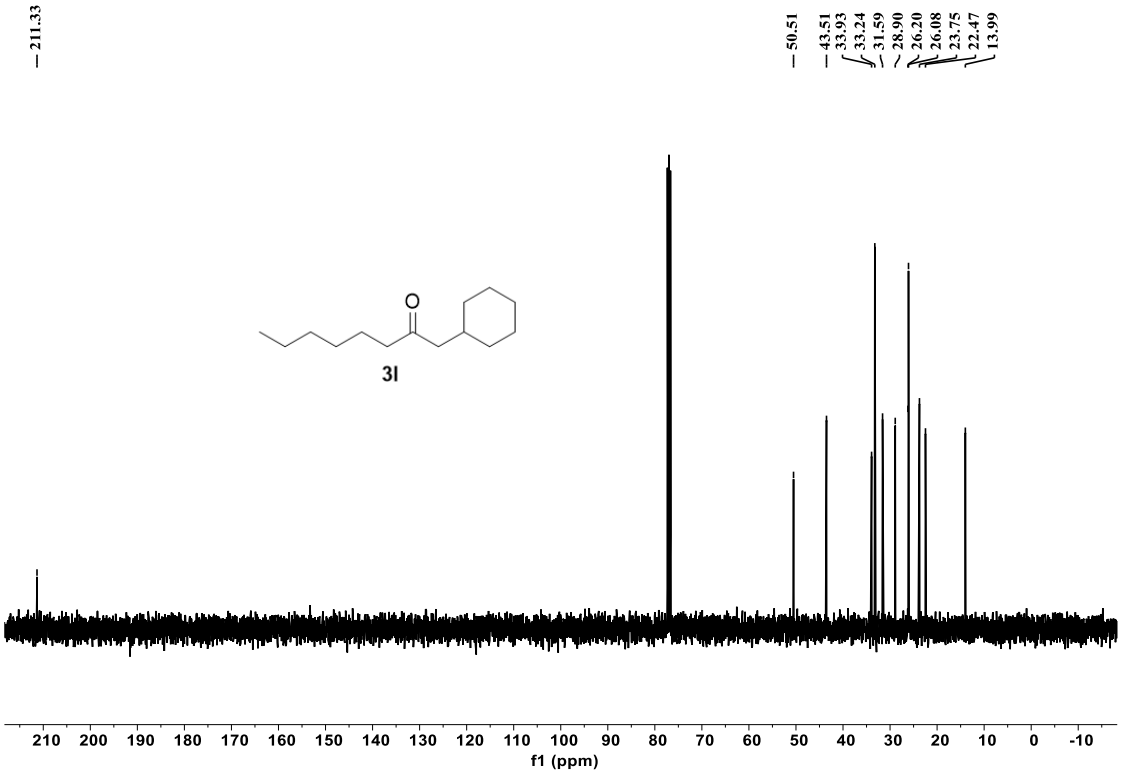

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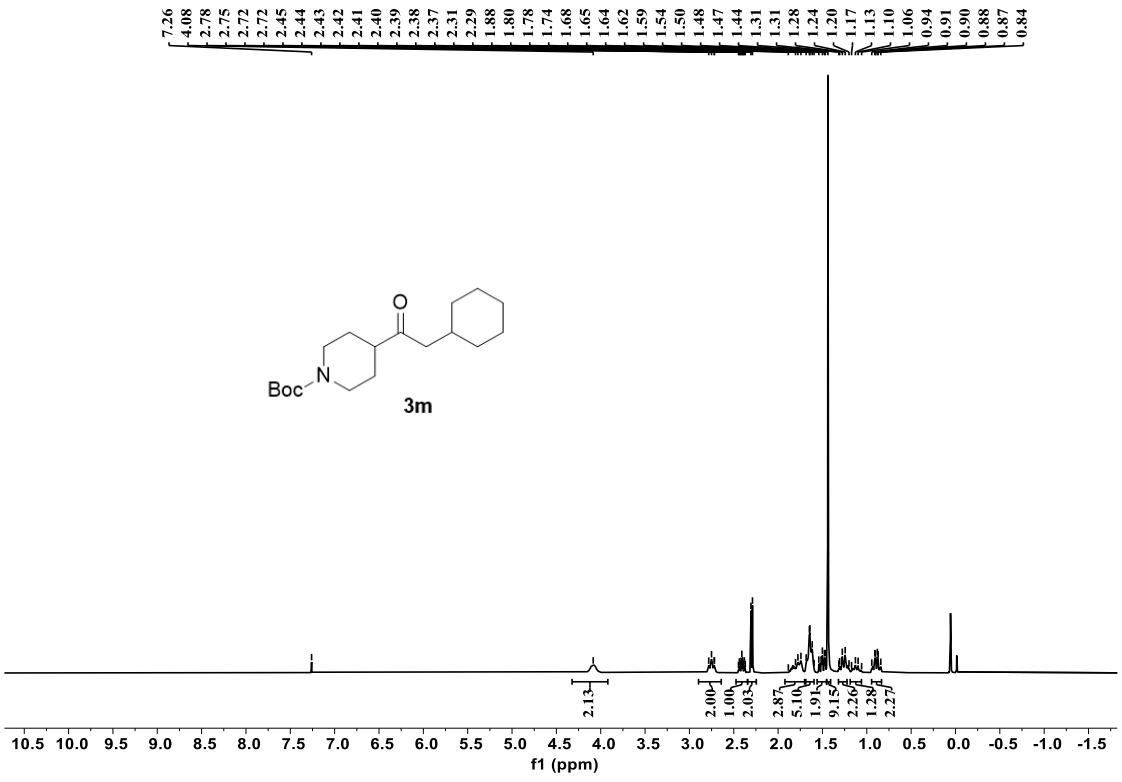

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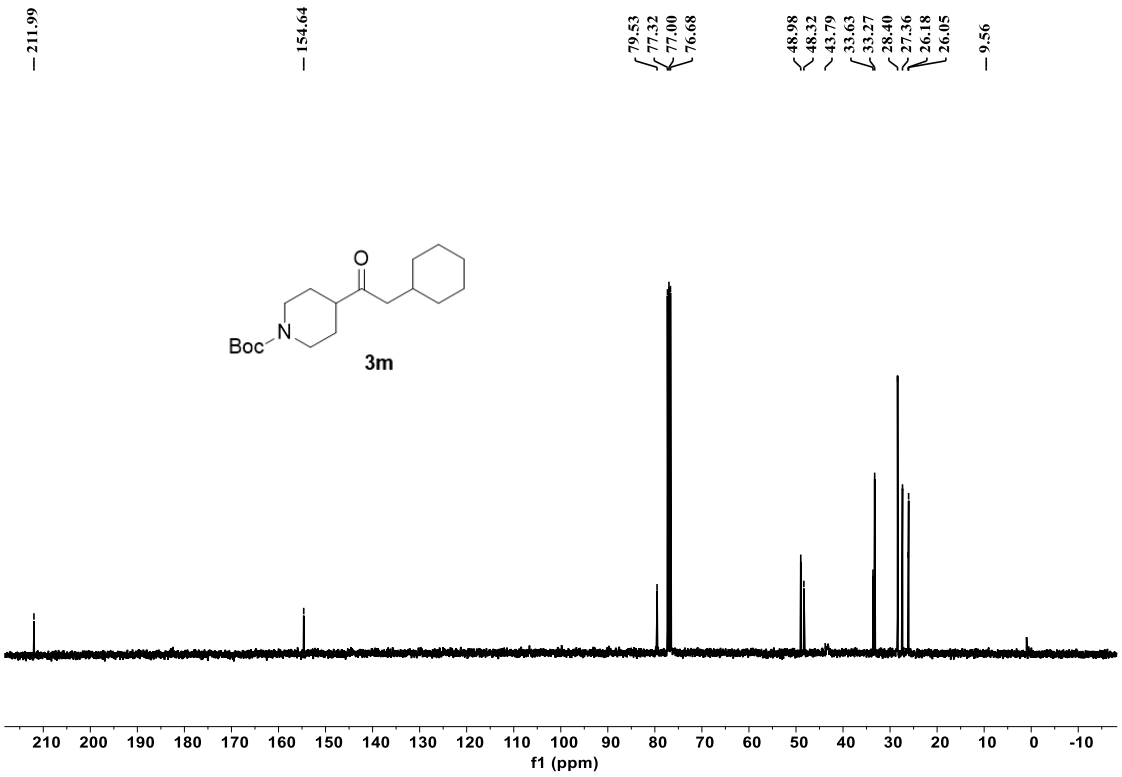

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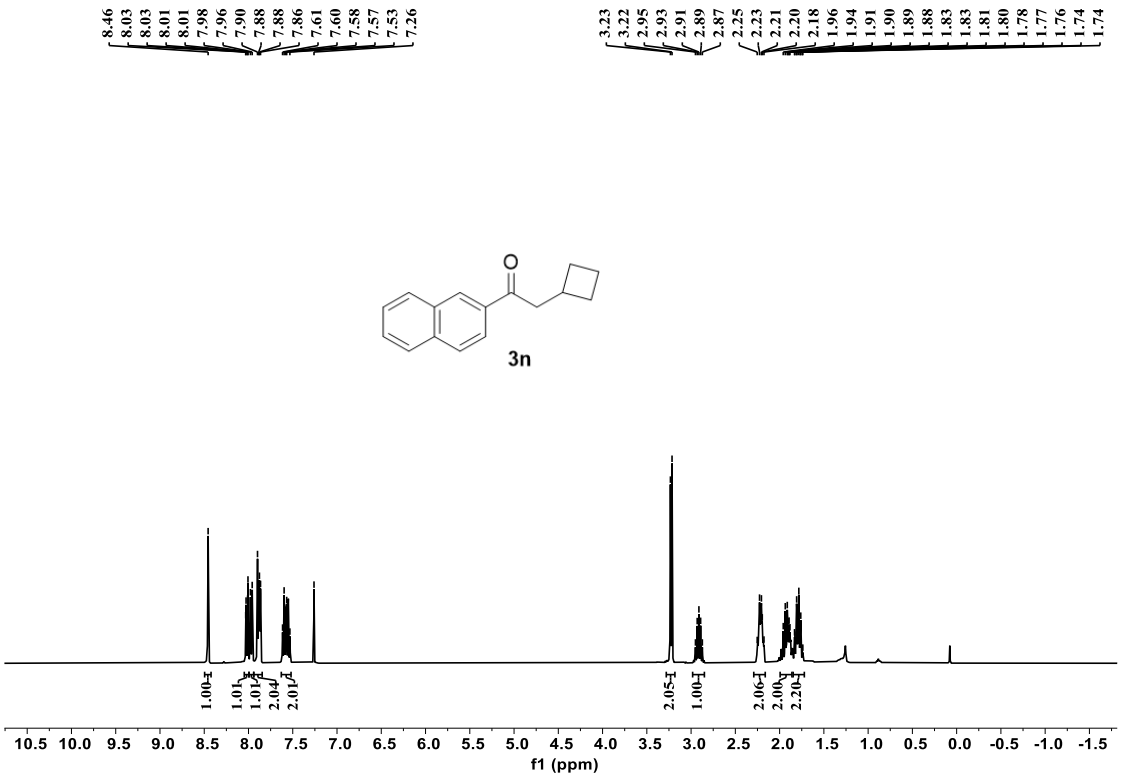

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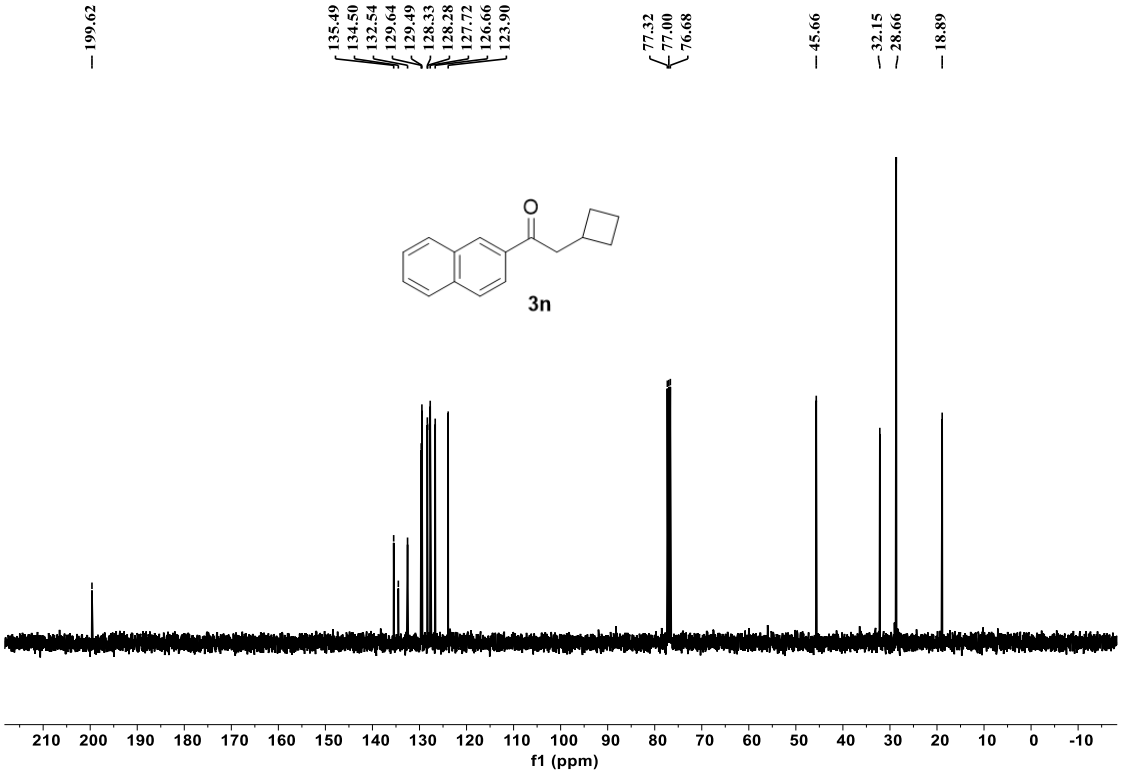

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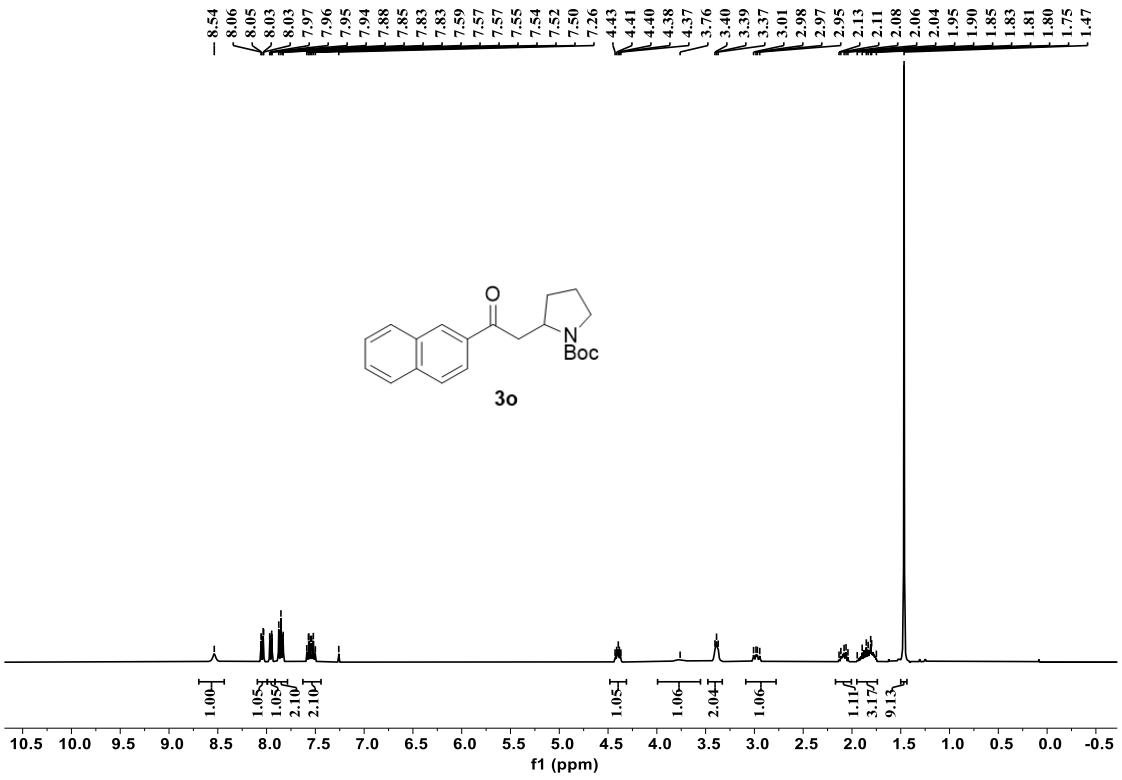

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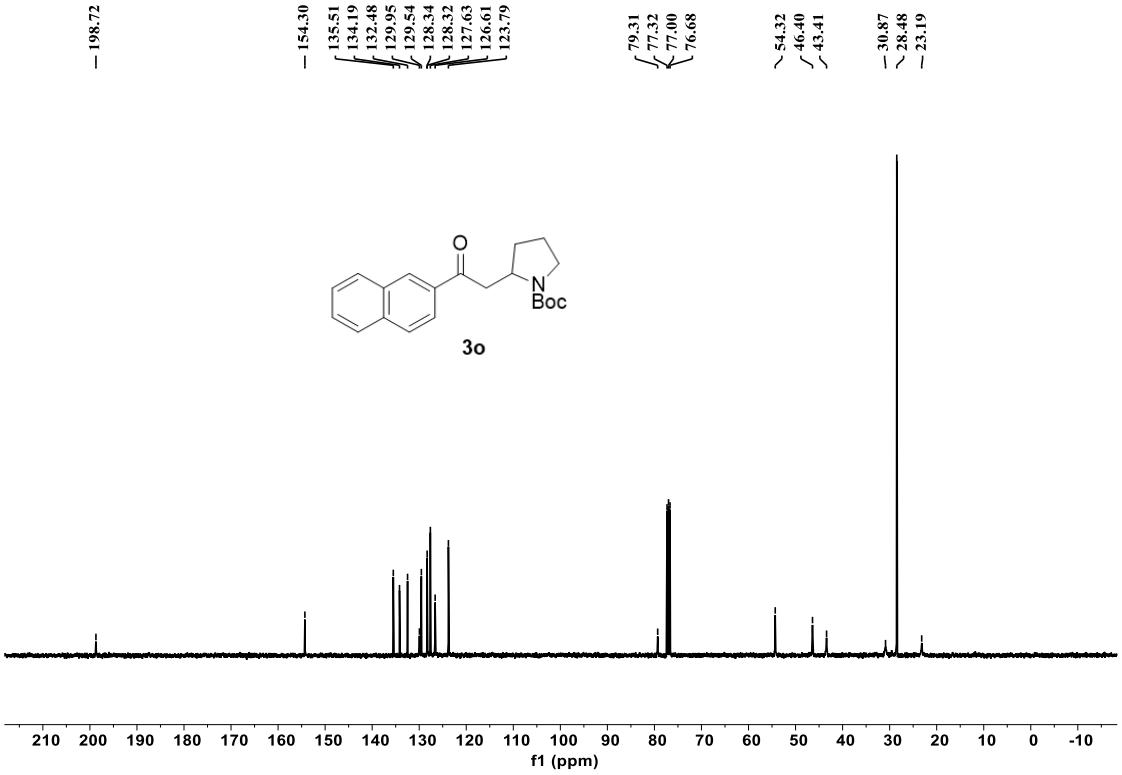

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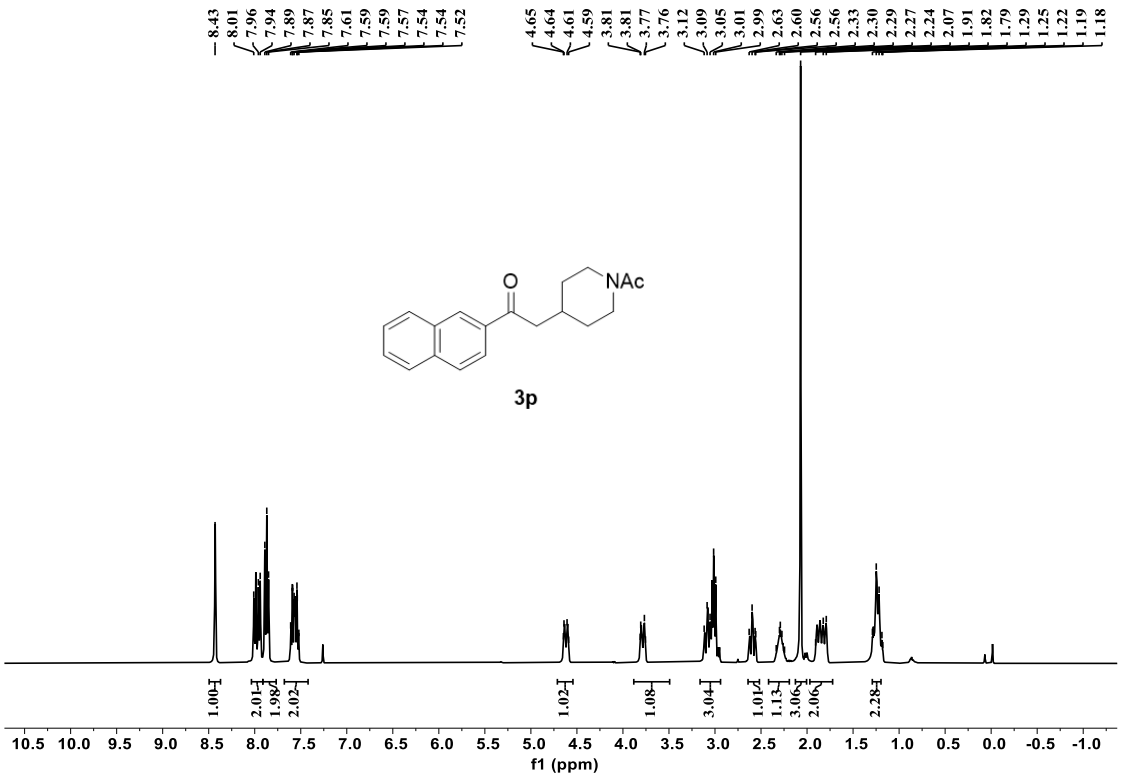

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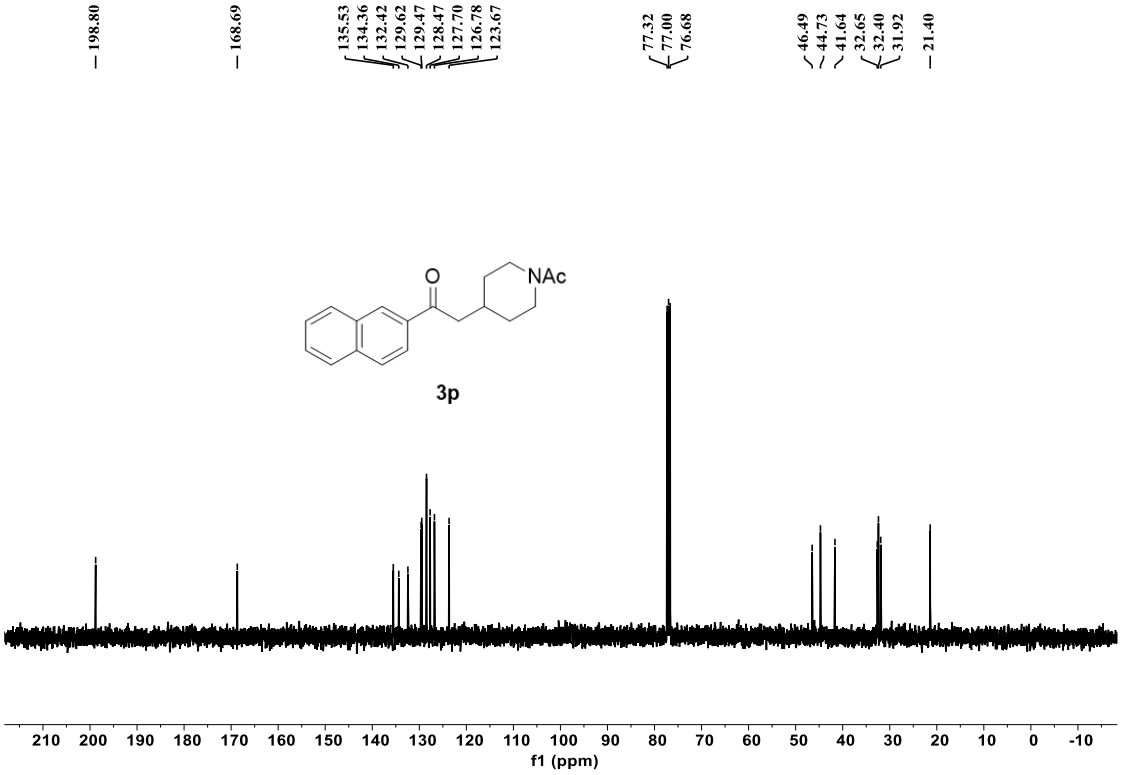

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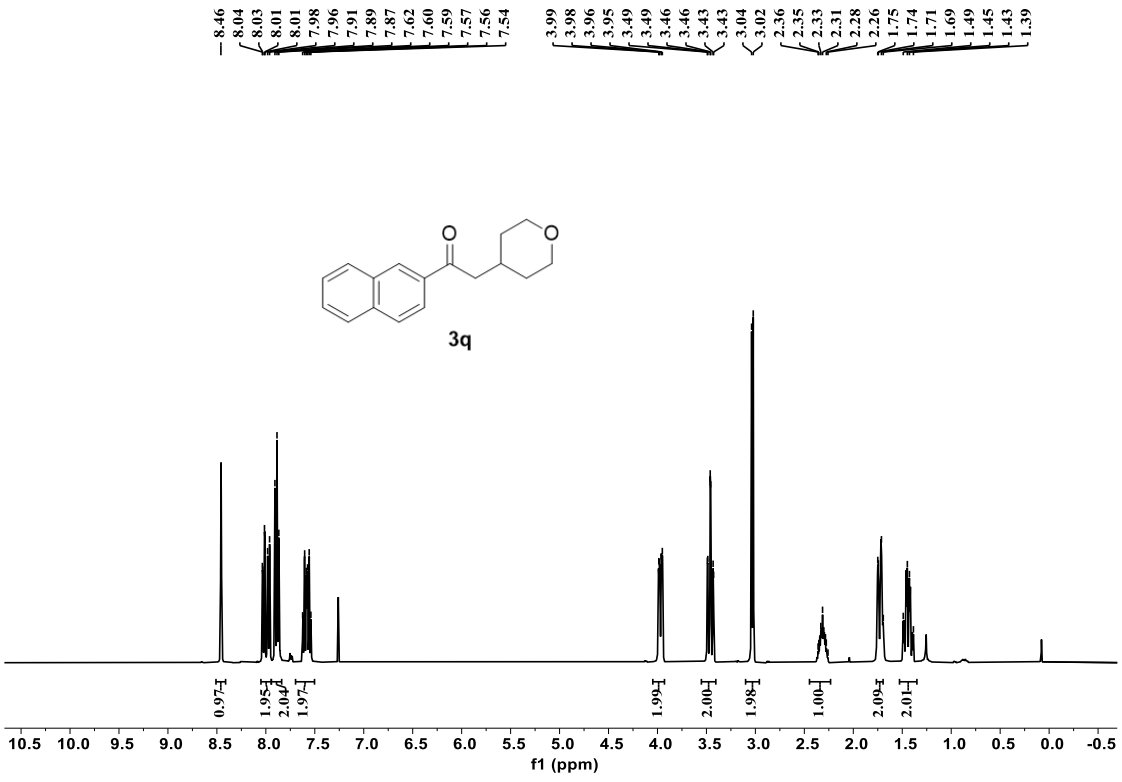

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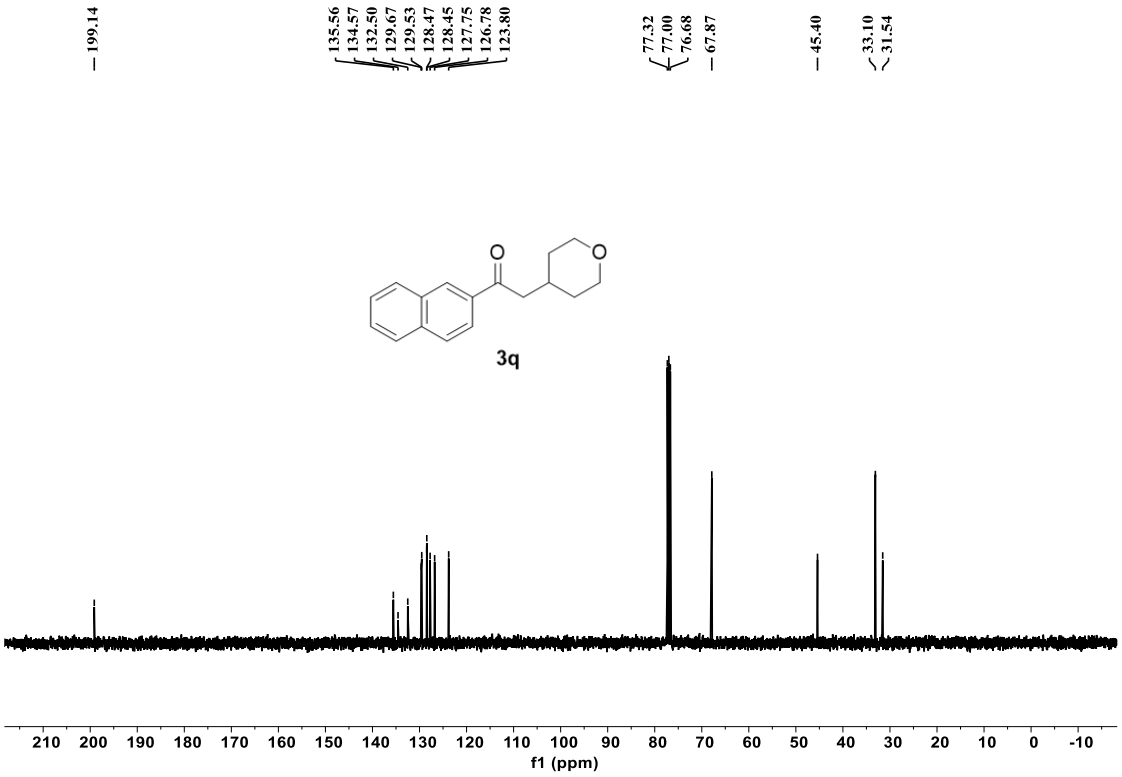

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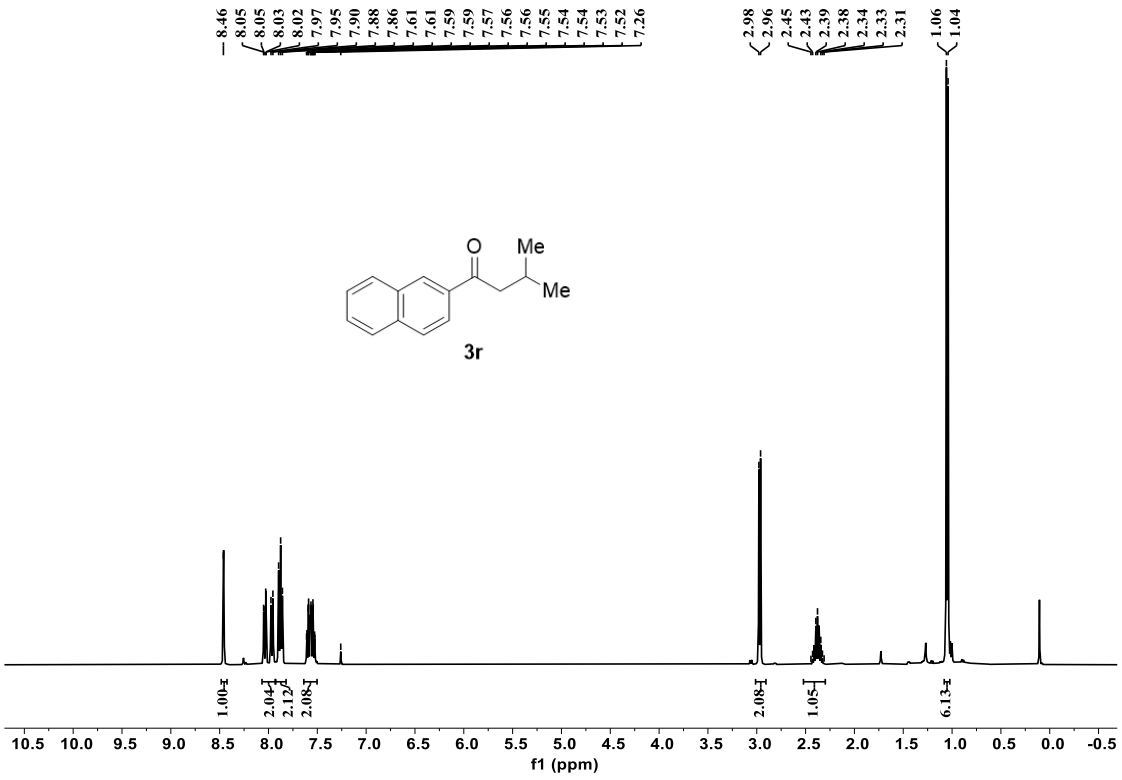

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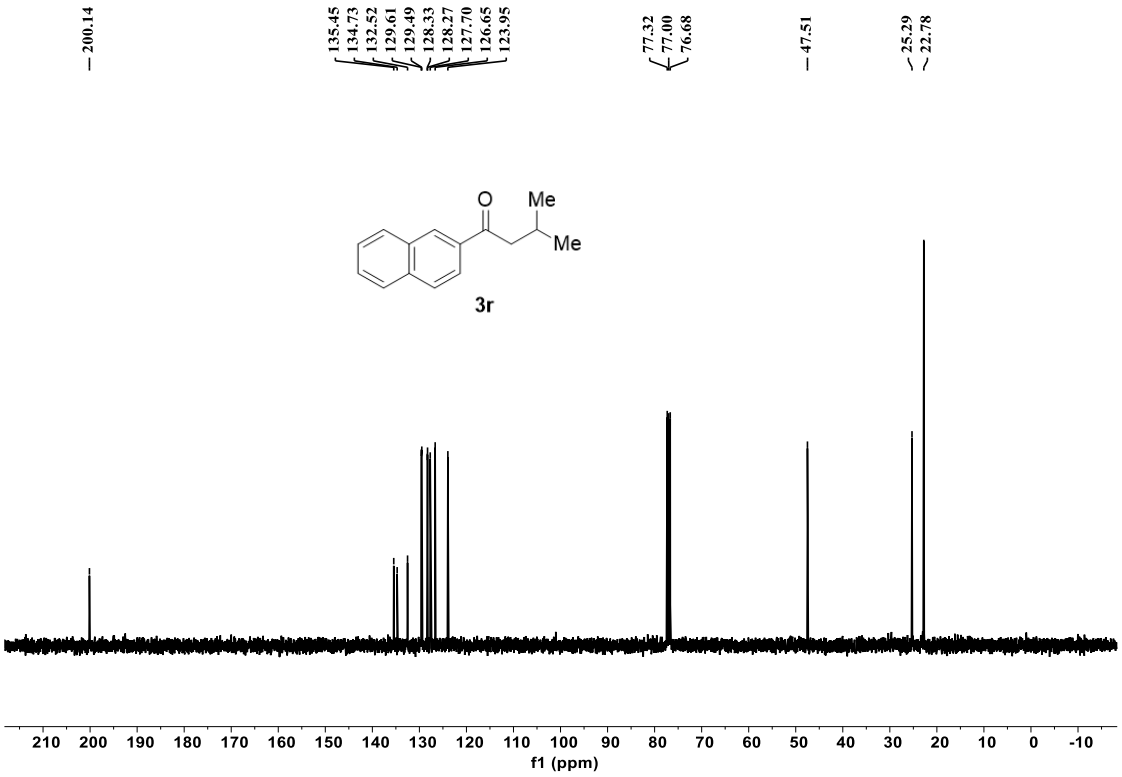

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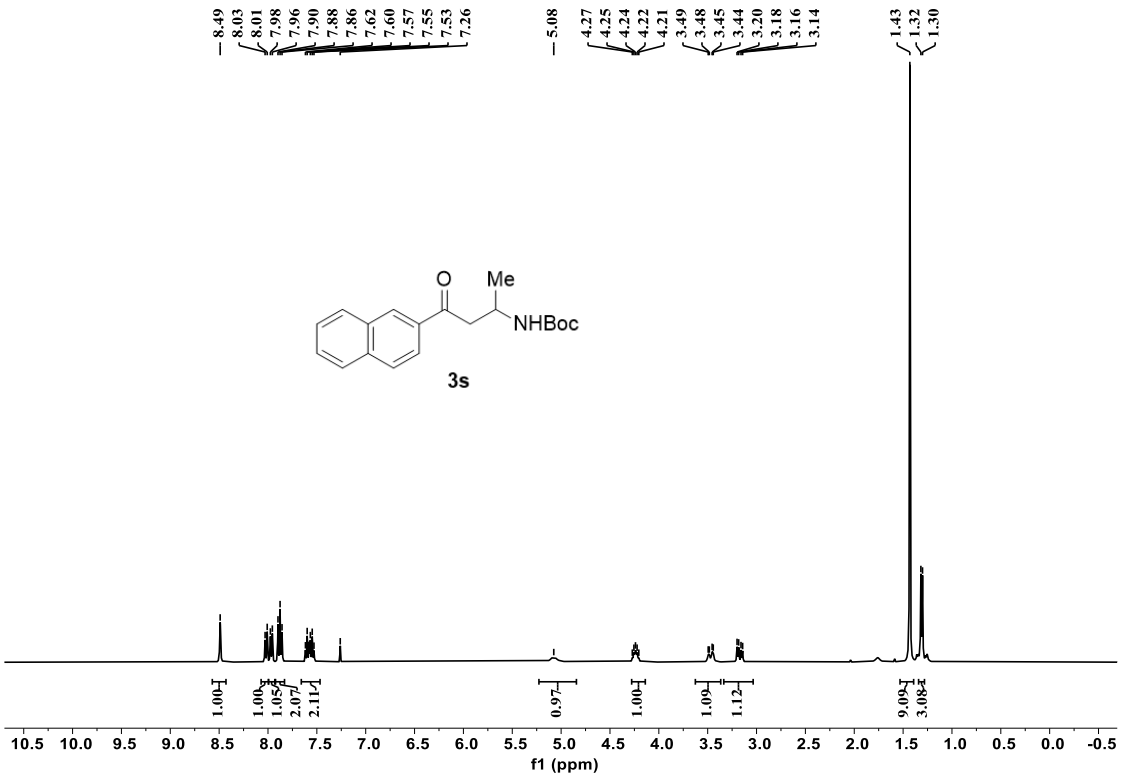
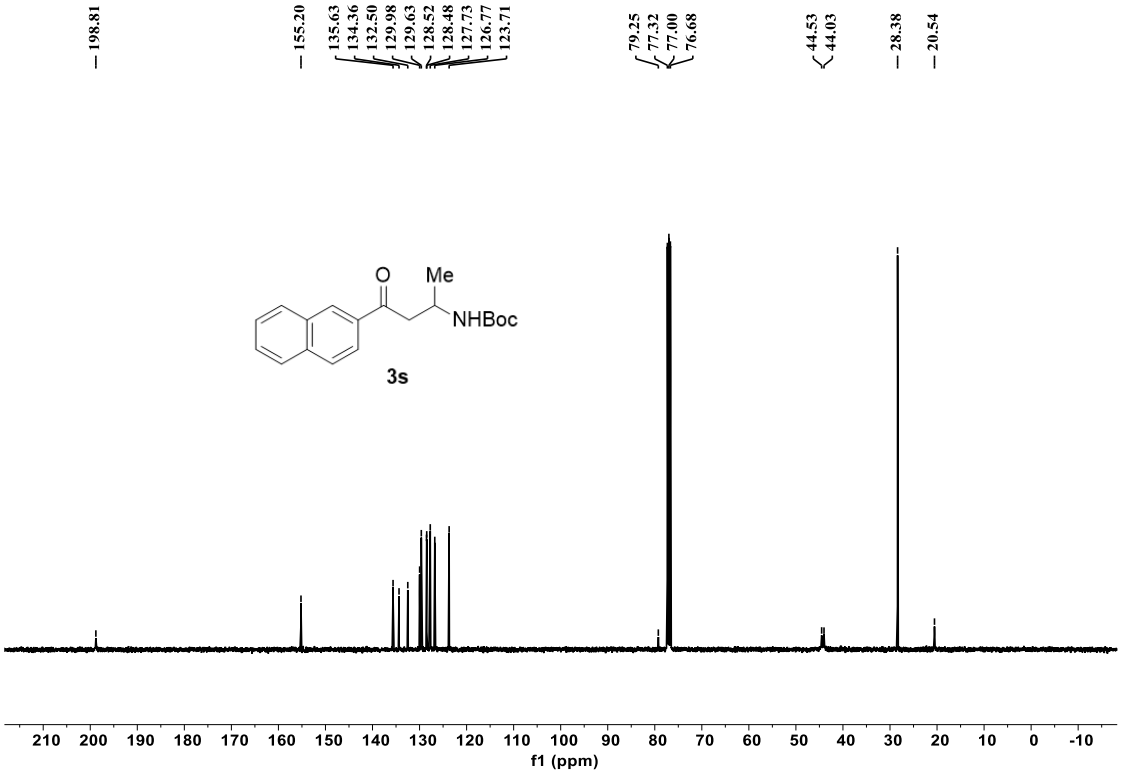


^{13}C NMR (101 MHz, $CDCl_3$)



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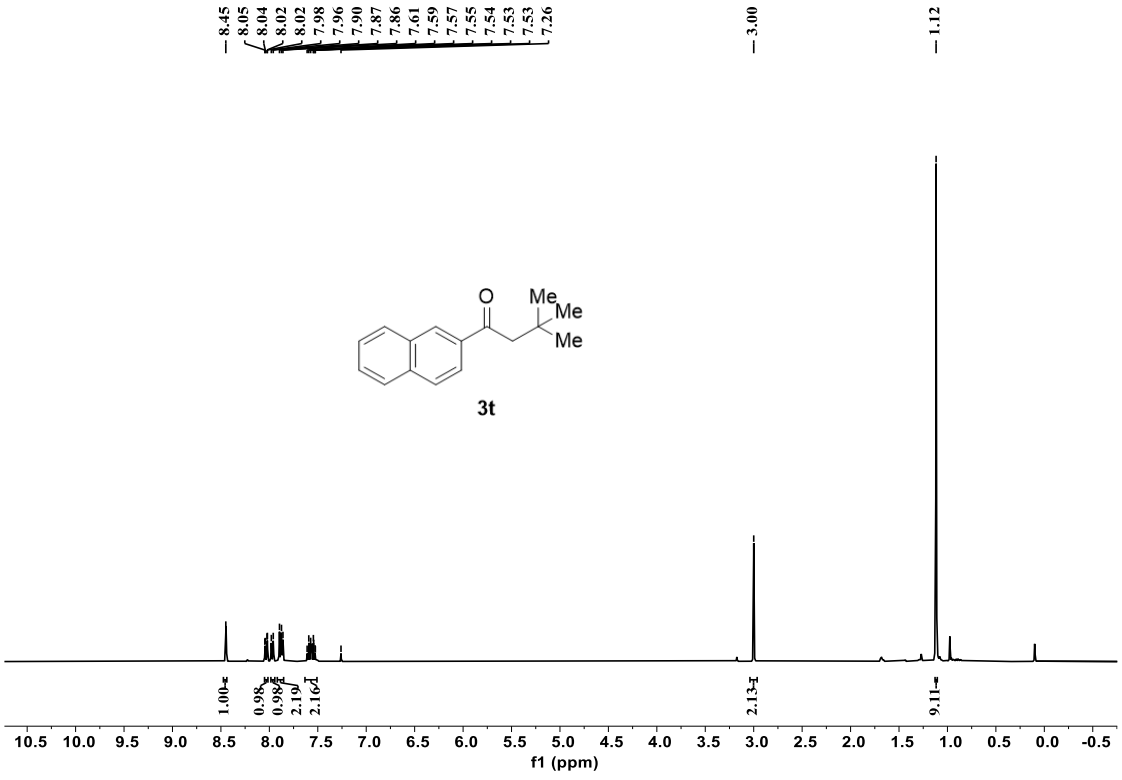
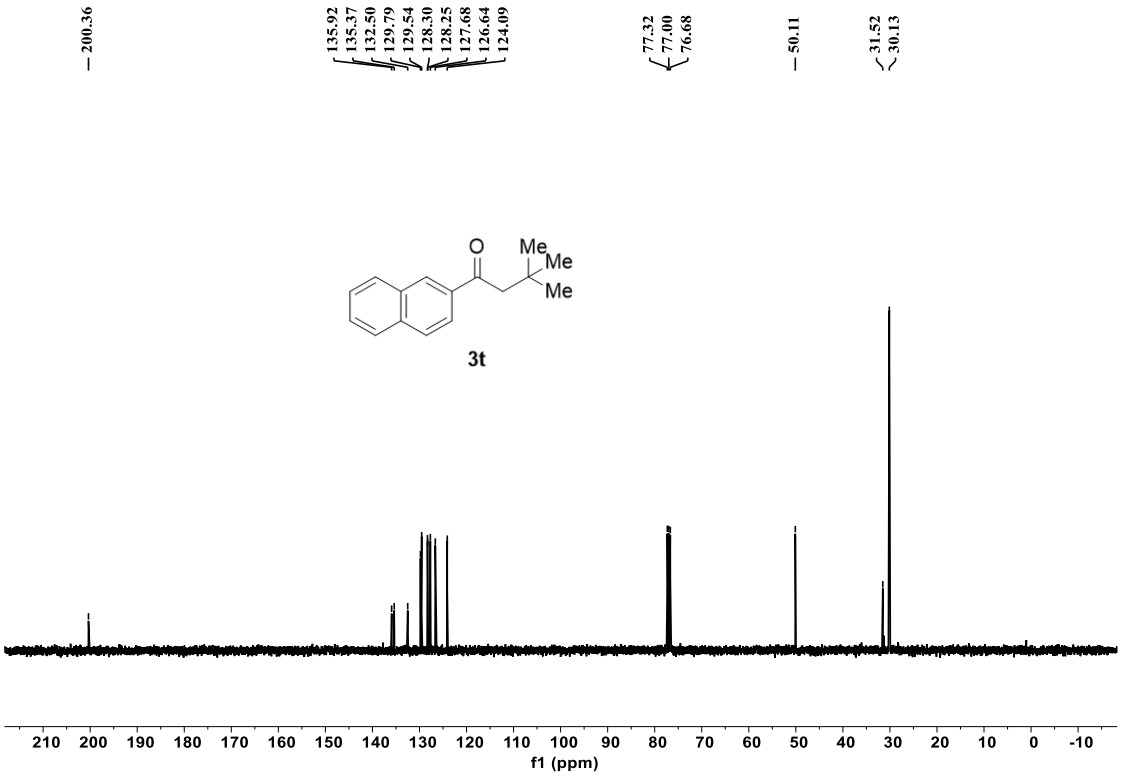


^{13}C NMR (101 MHz, $CDCl_3$)



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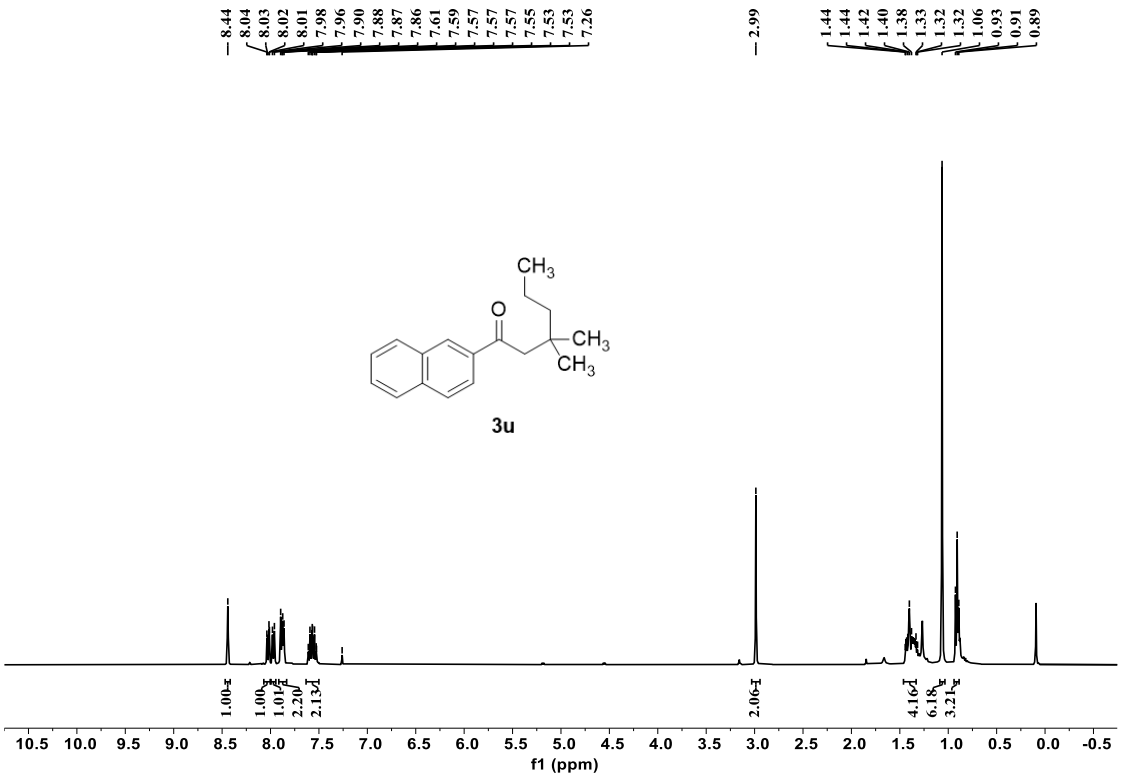

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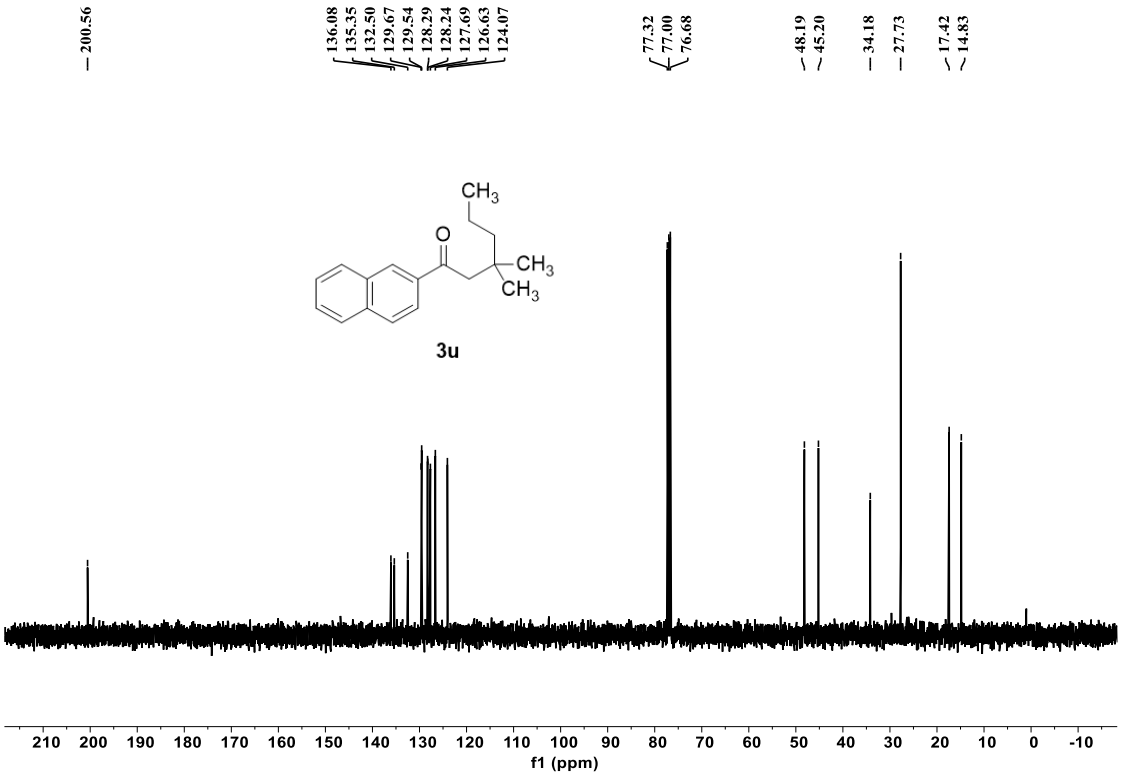

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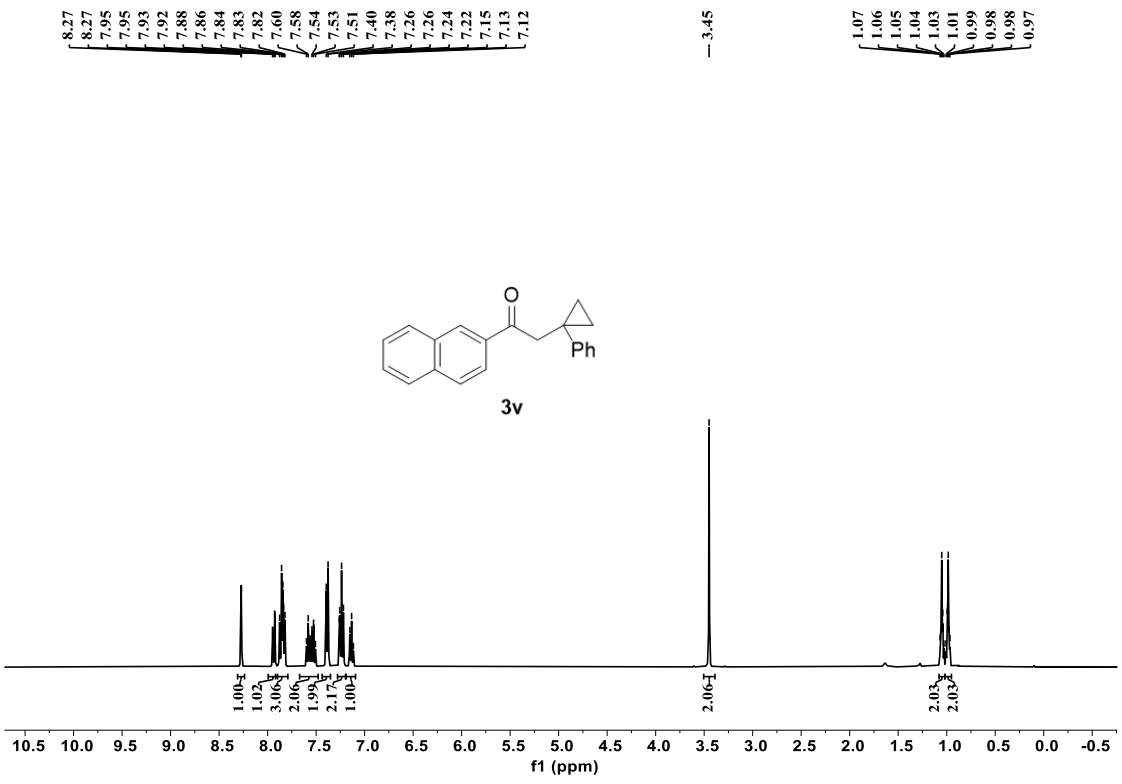

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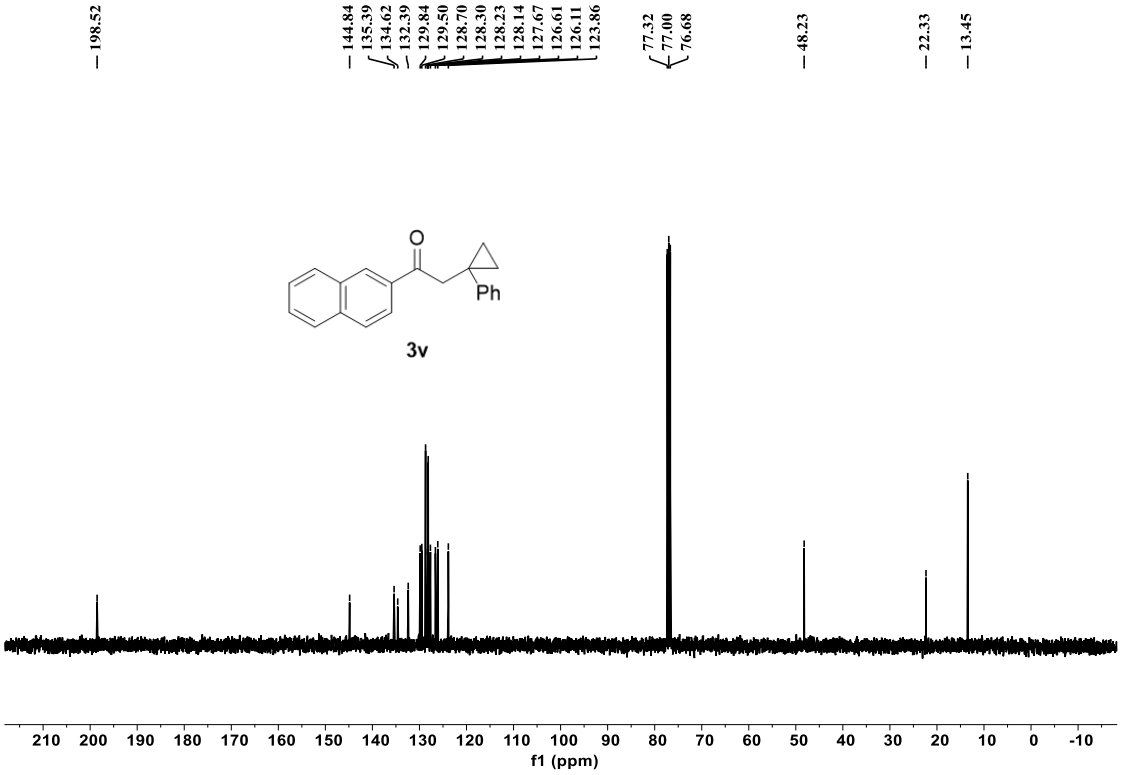
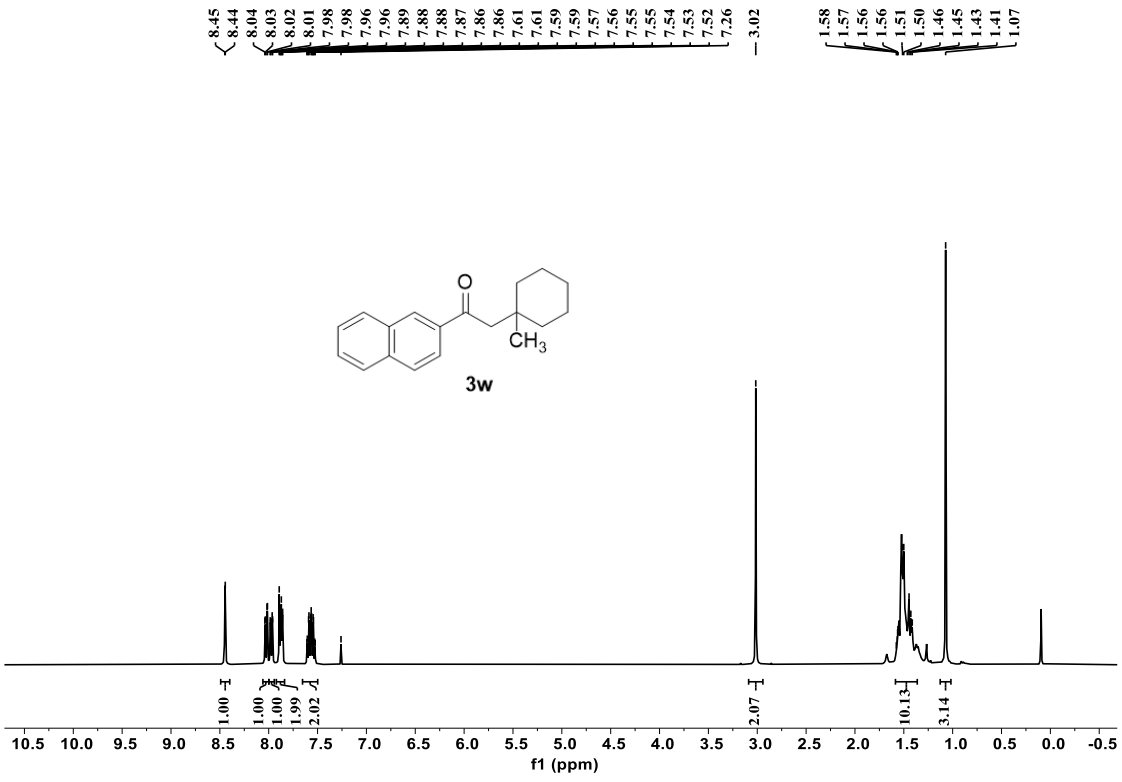
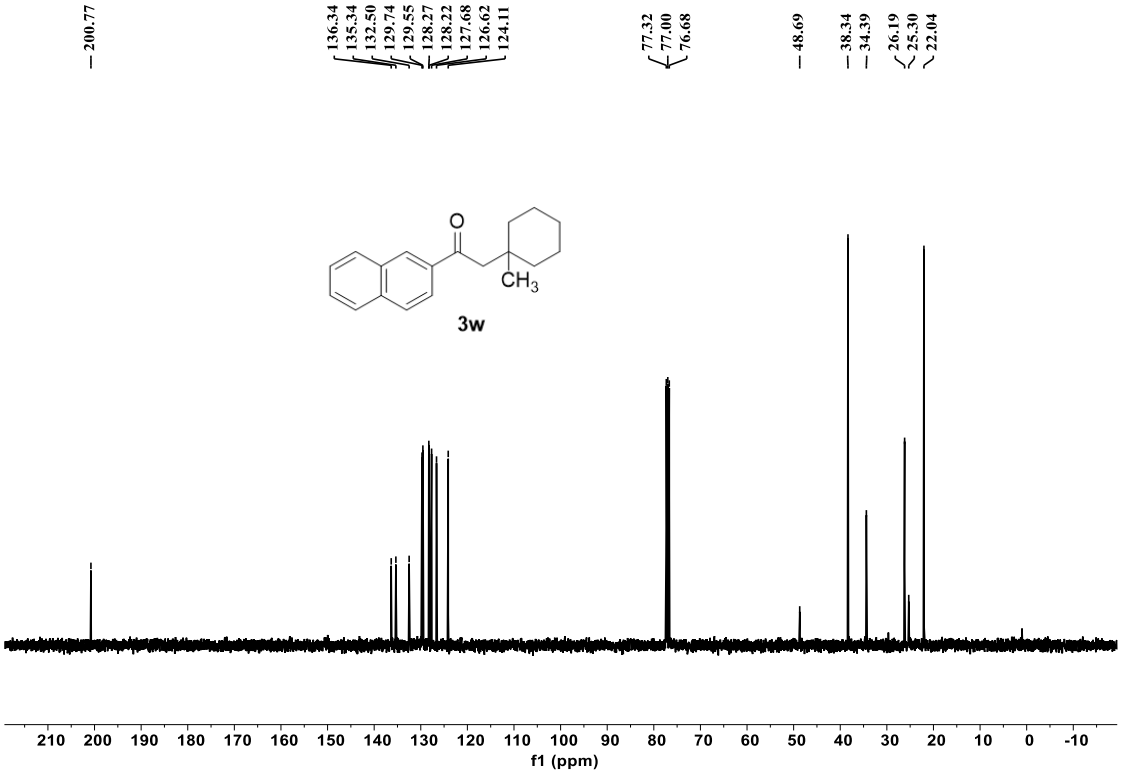


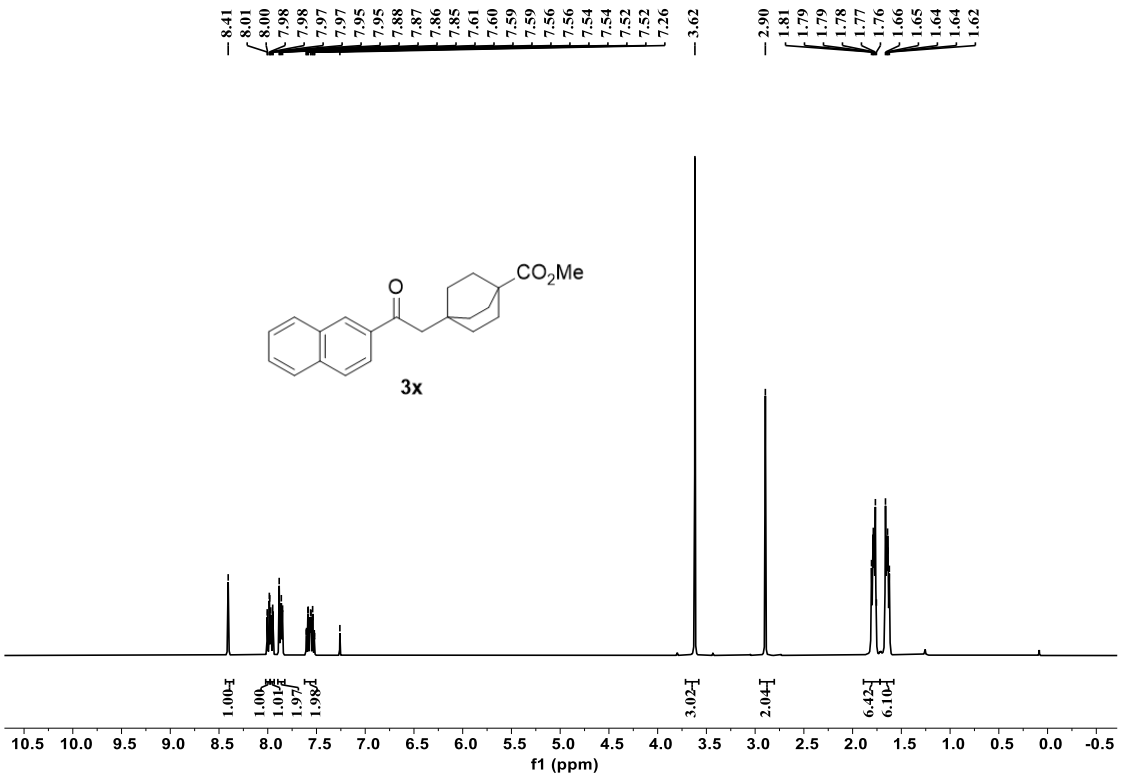
^{1}H NMR (400 MHz, $CDCl_3$)



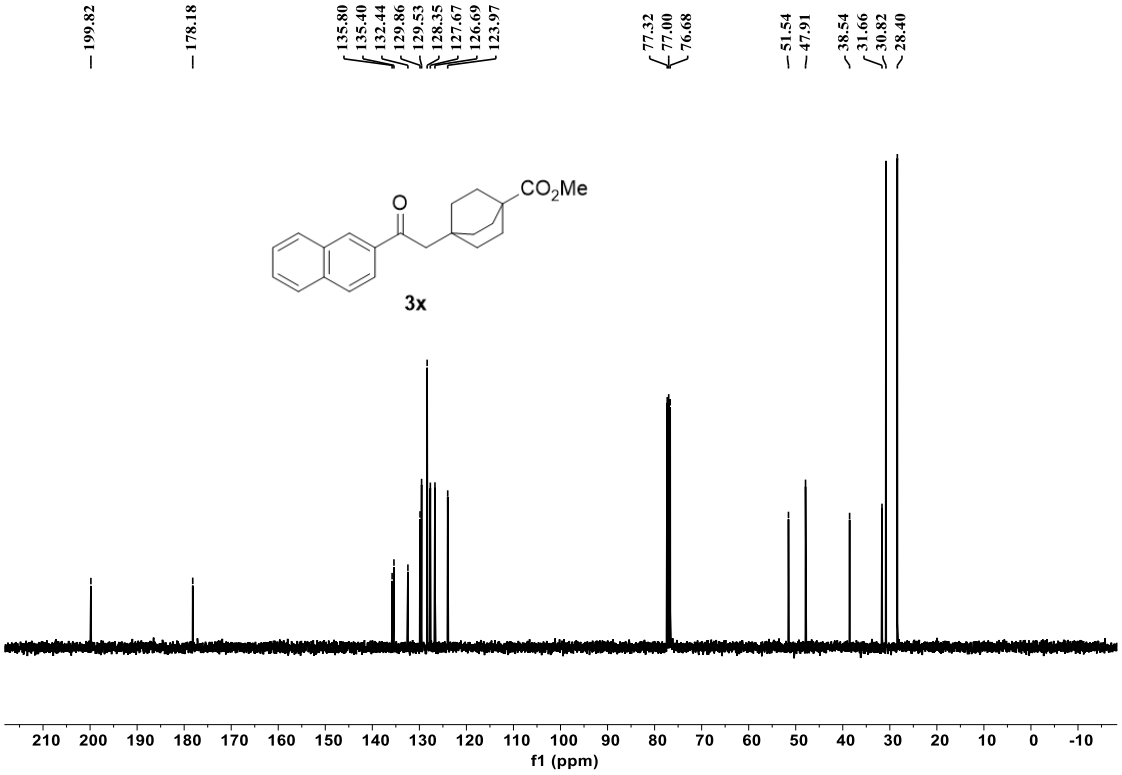
^{13}C NMR (101 MHz, $CDCl_3$)



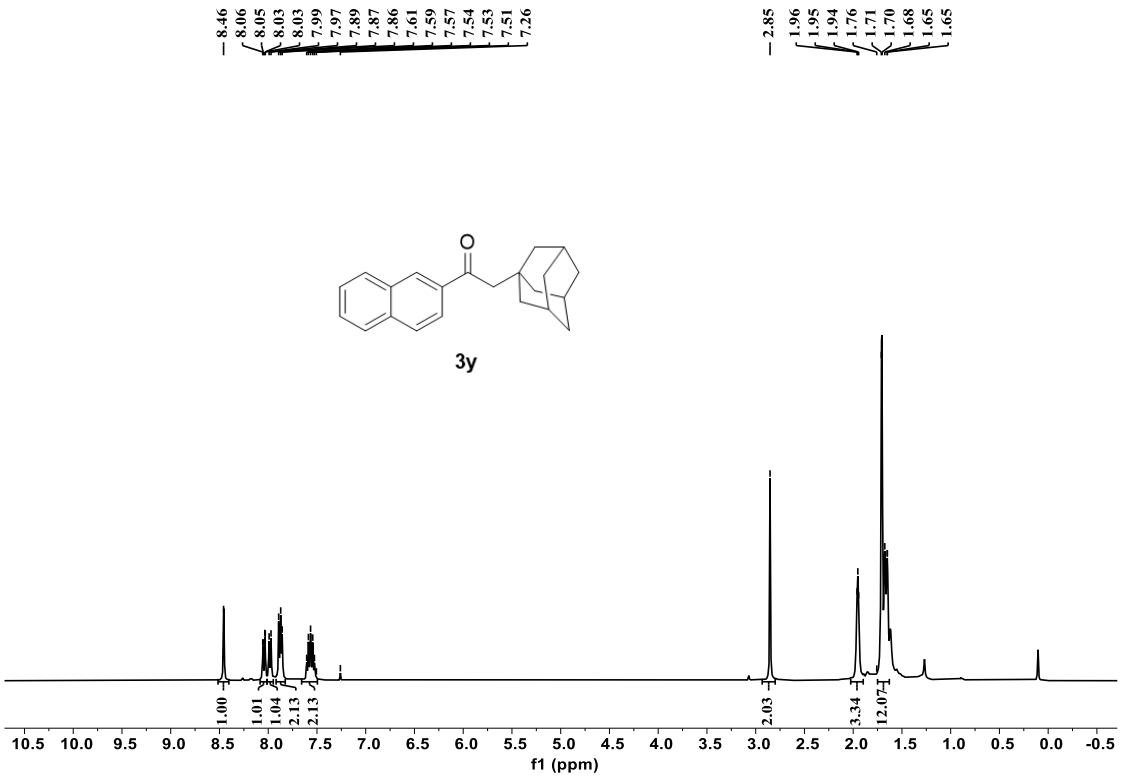
^{1}H NMR (400 MHz, $CDCl_3$)



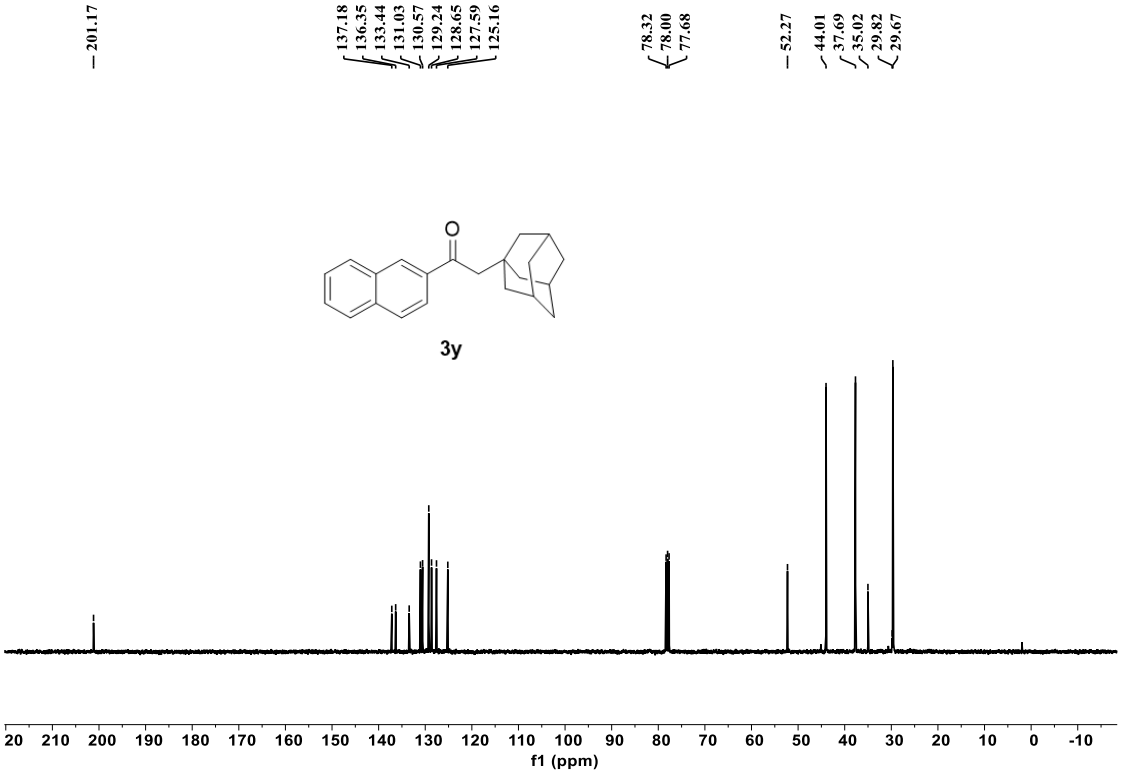
^{13}C NMR (101 MHz, $CDCl_3$)



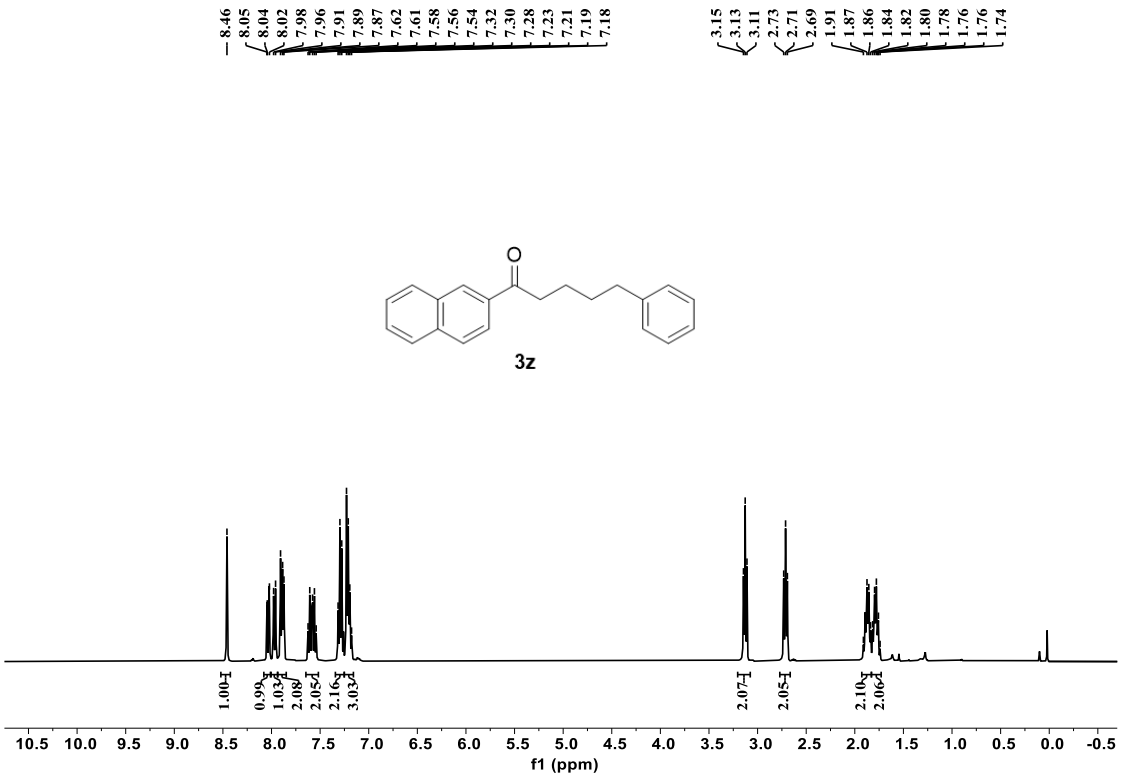
^{1}H NMR (400 MHz, $CDCl_3$)



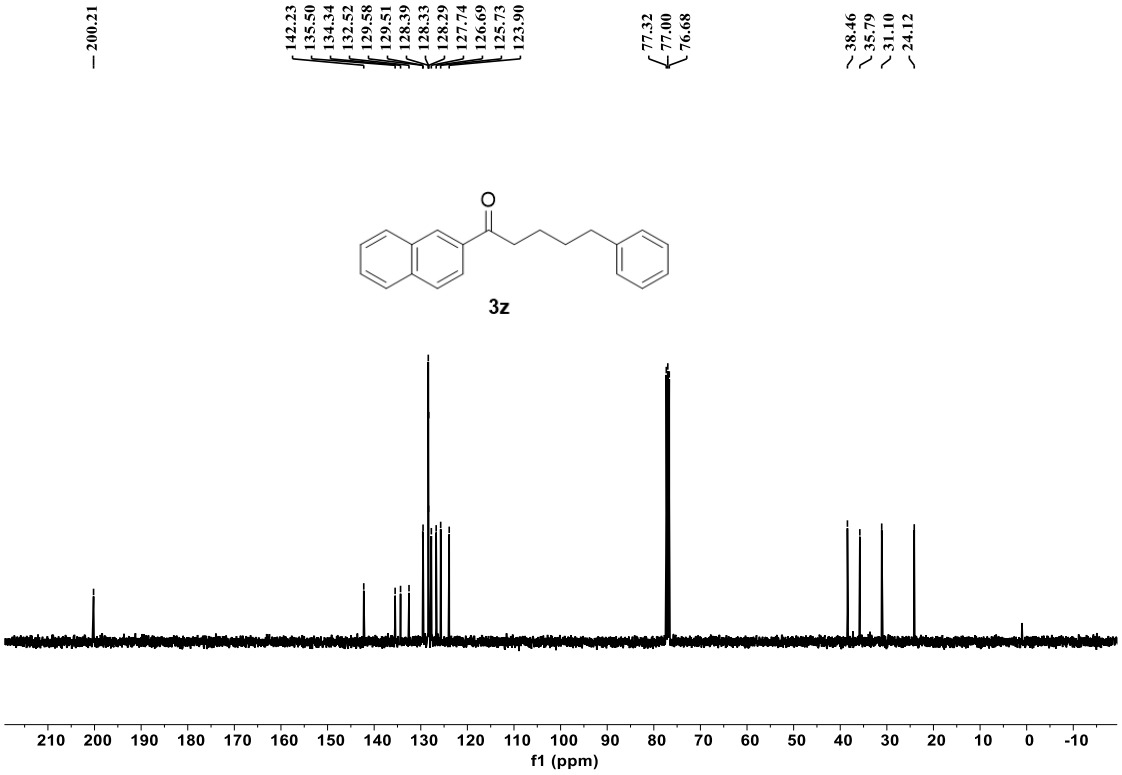
^{13}C NMR (101 MHz, $CDCl_3$)



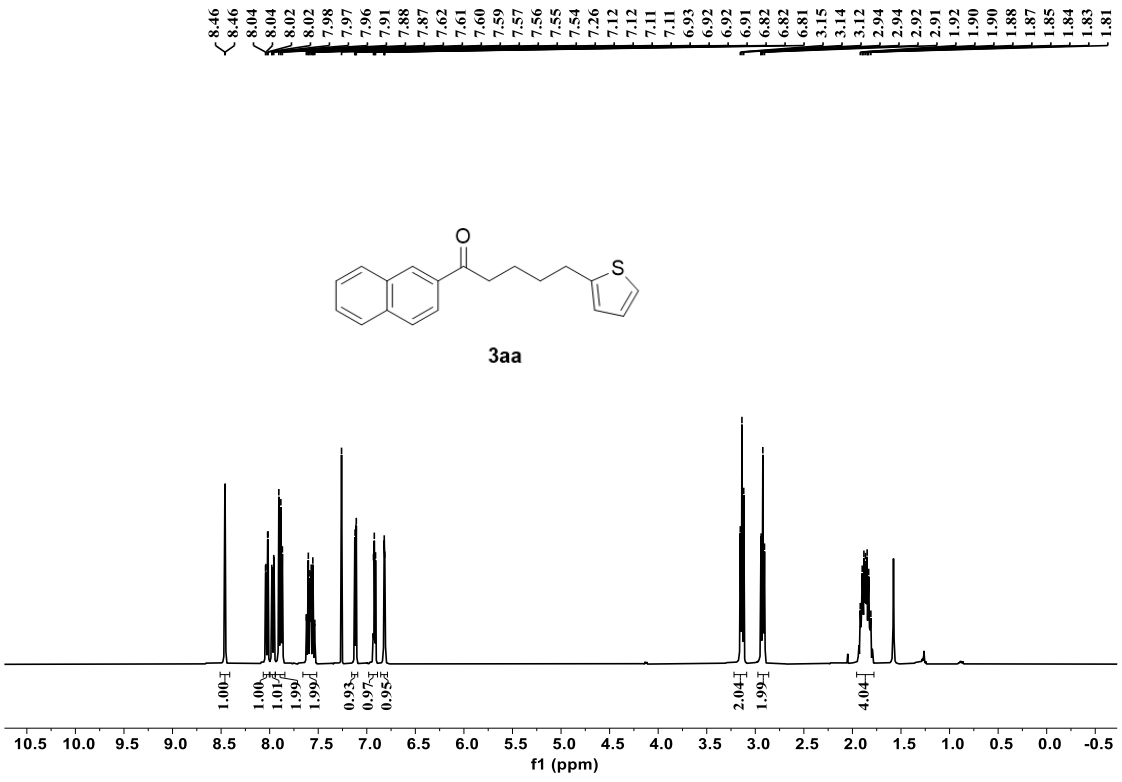
^{1}H NMR (400 MHz, $CDCl_3$)



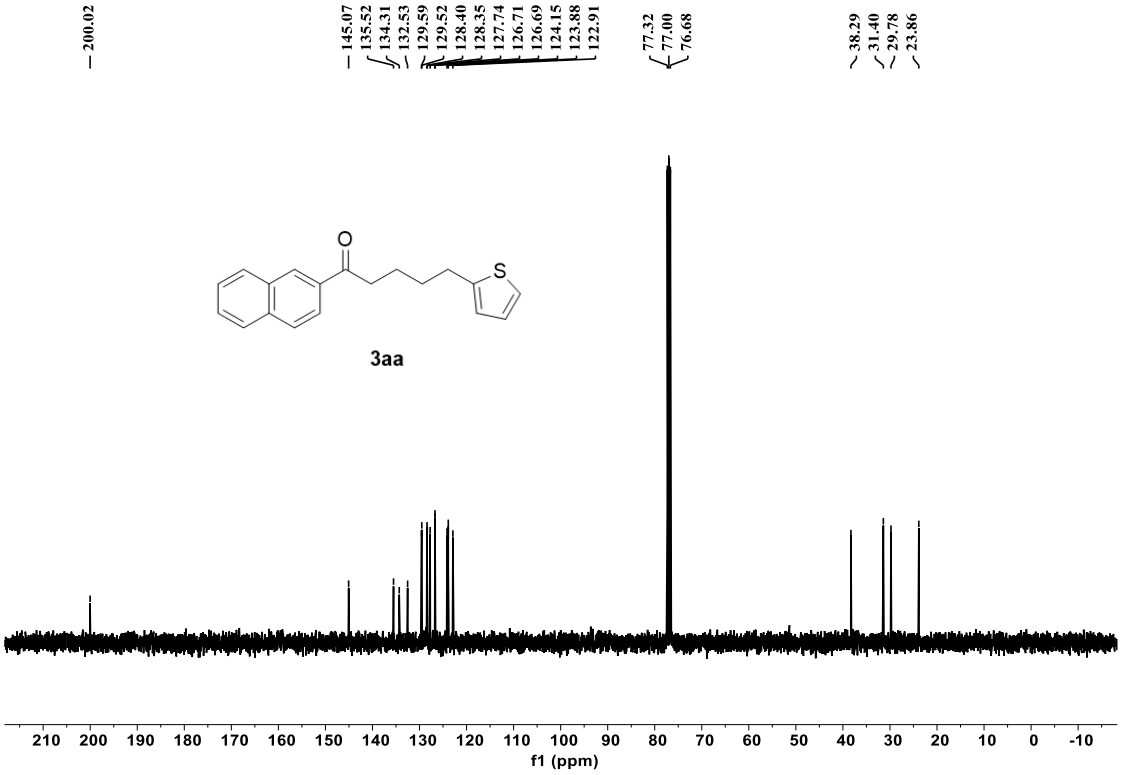
^{13}C NMR (101 MHz, $CDCl_3$)



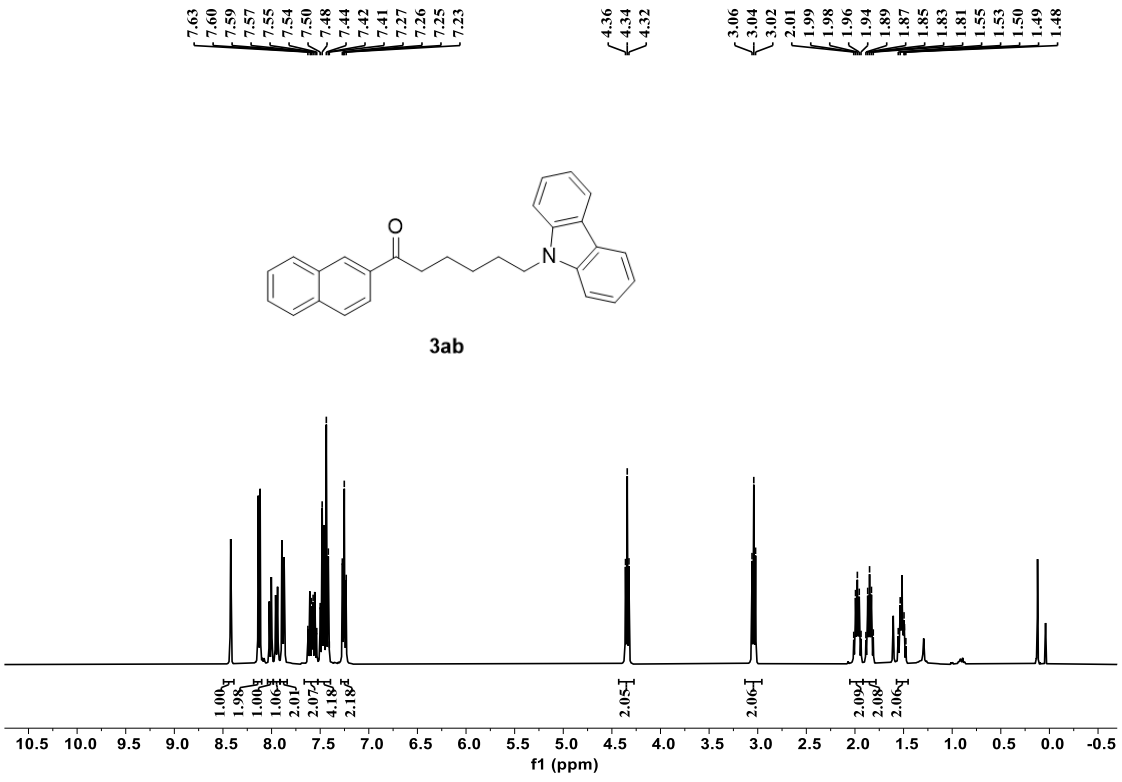
^{1}H NMR (400 MHz, $CDCl_3$)



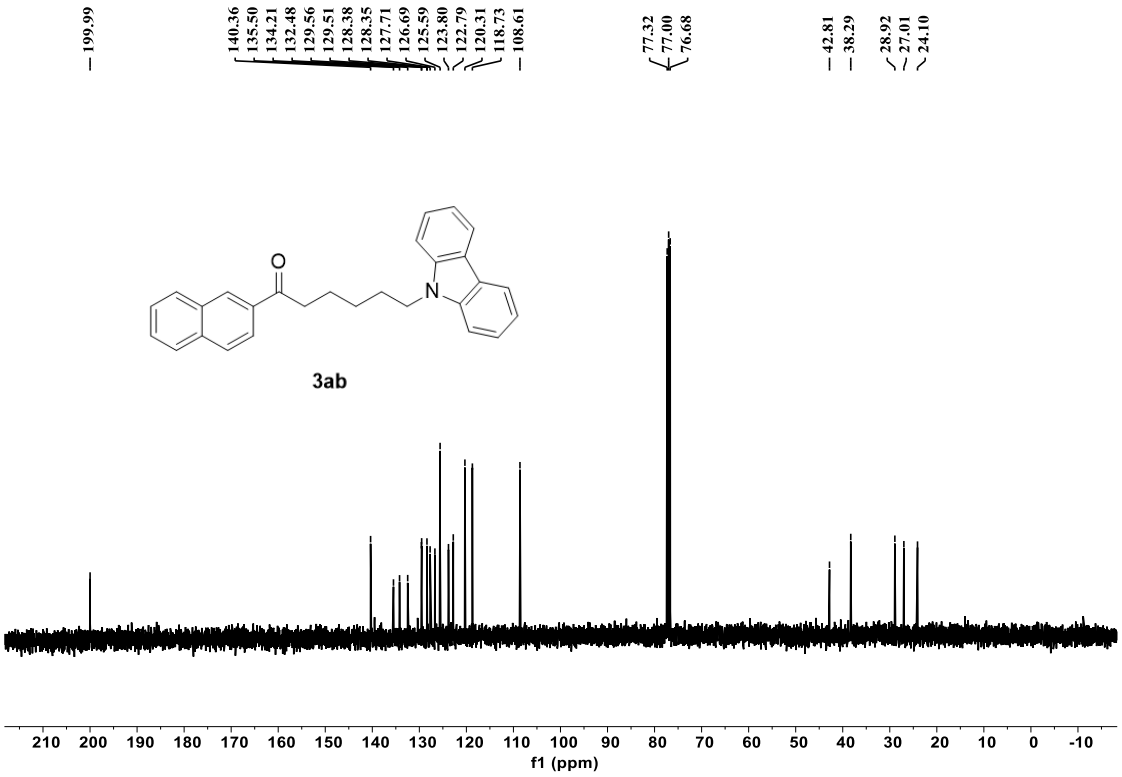
^{13}C NMR (101 MHz, $CDCl_3$)



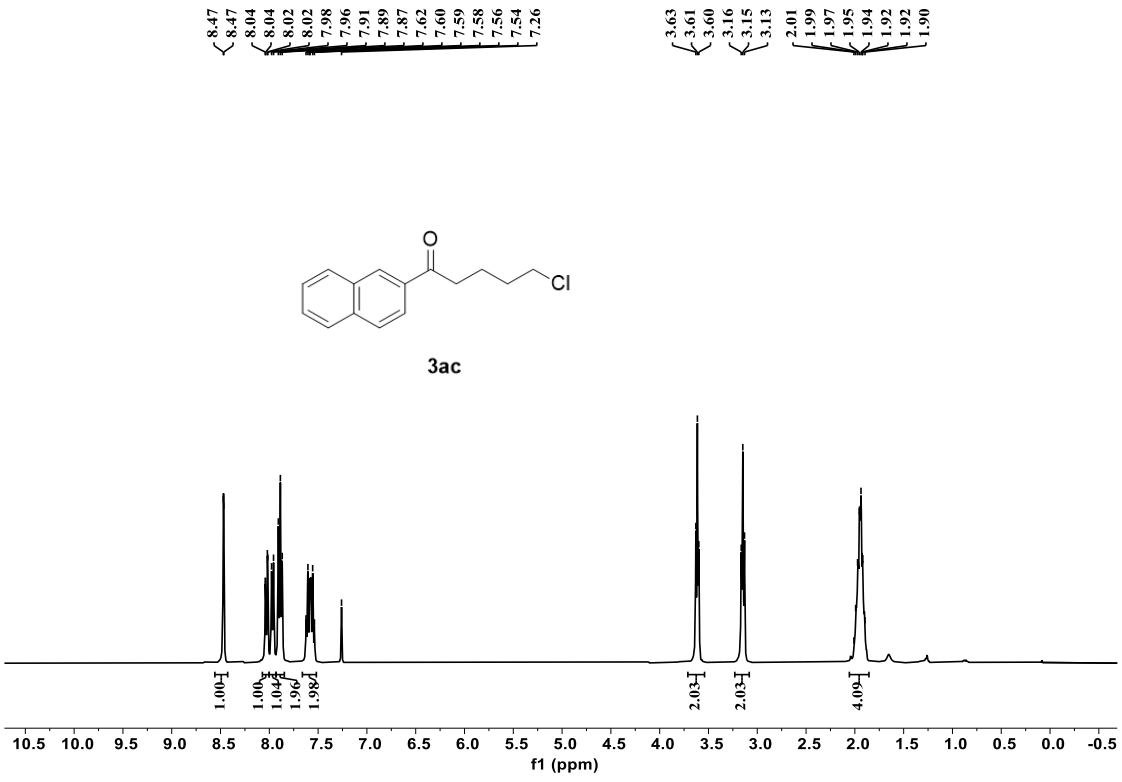
^{1}H NMR (400 MHz, $CDCl_3$)



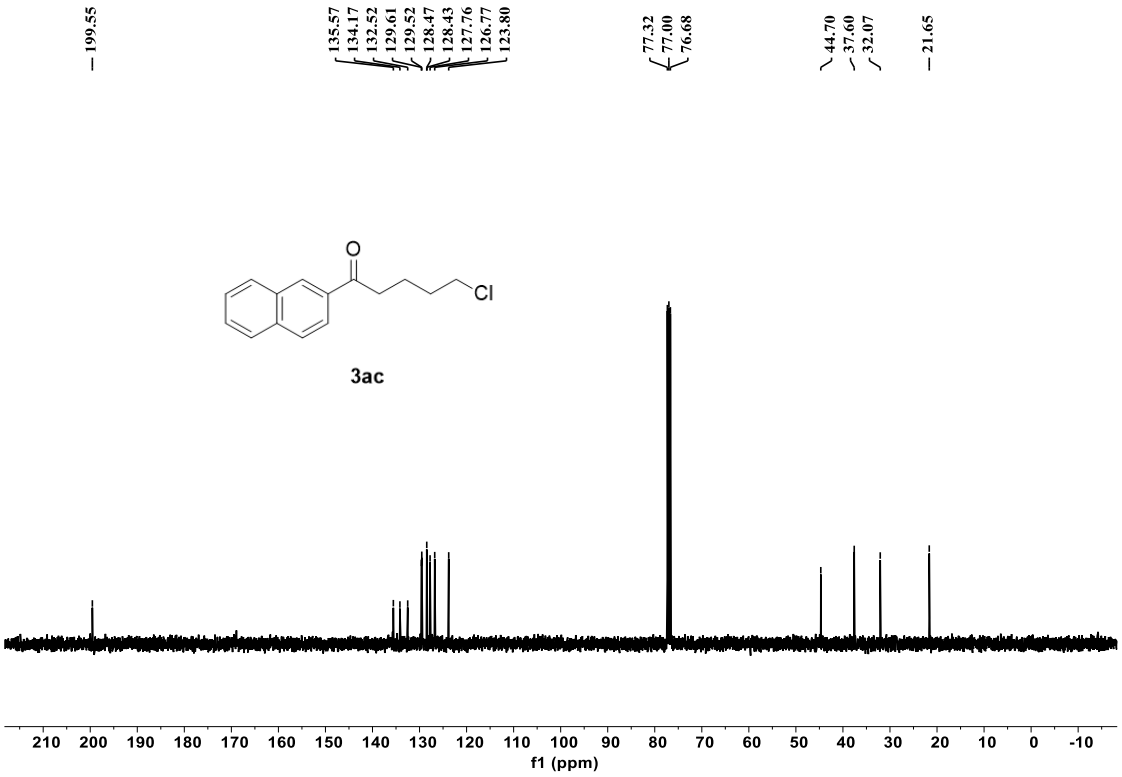
^{13}C NMR (101 MHz, $CDCl_3$)



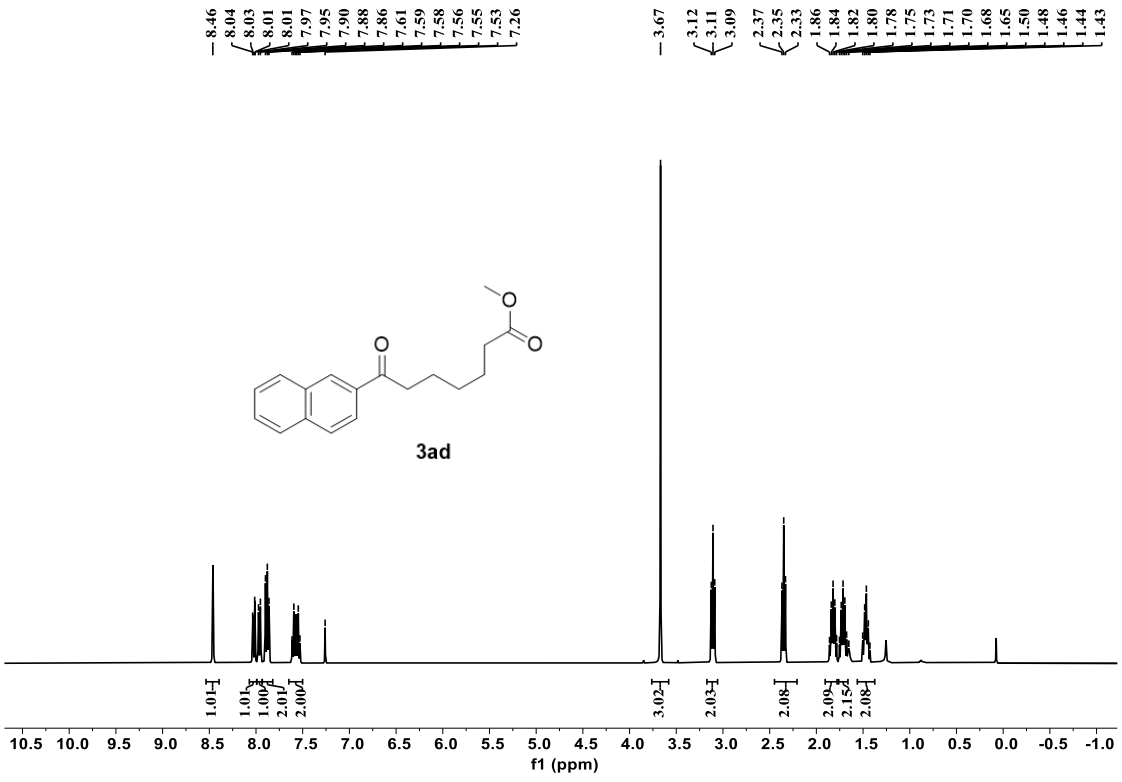
^{1}H NMR (400 MHz, $CDCl_3$)



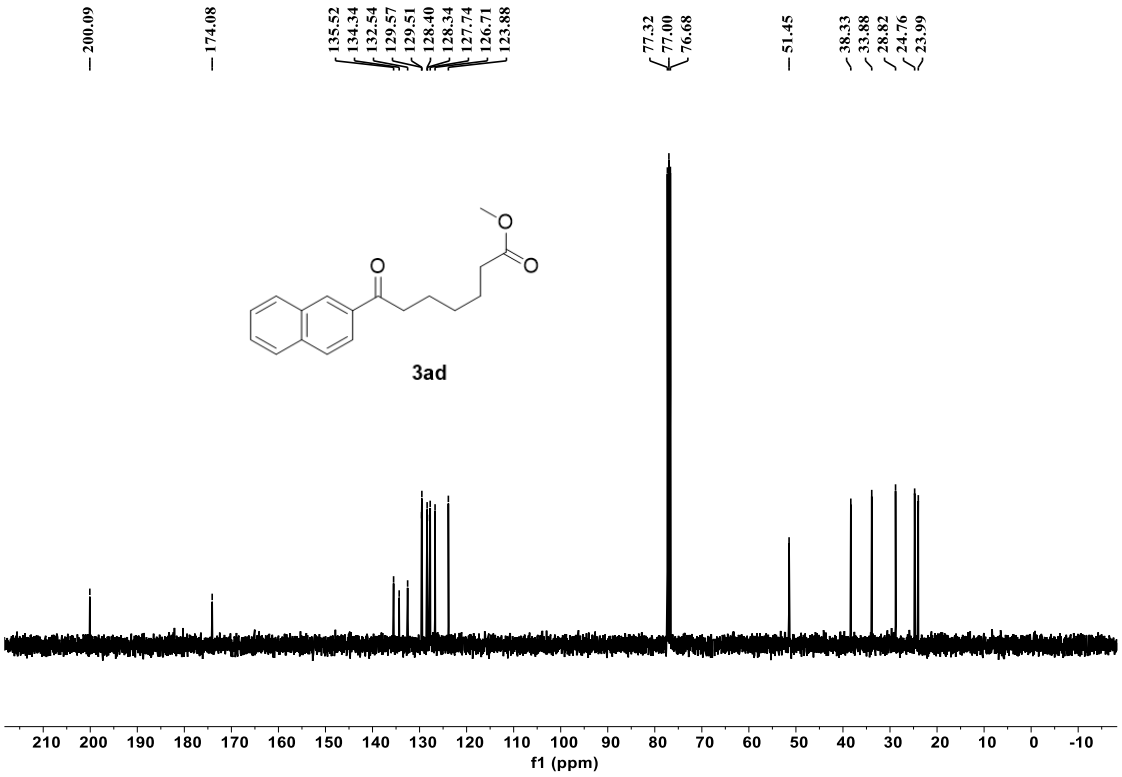
^{13}C NMR (101 MHz, $CDCl_3$)



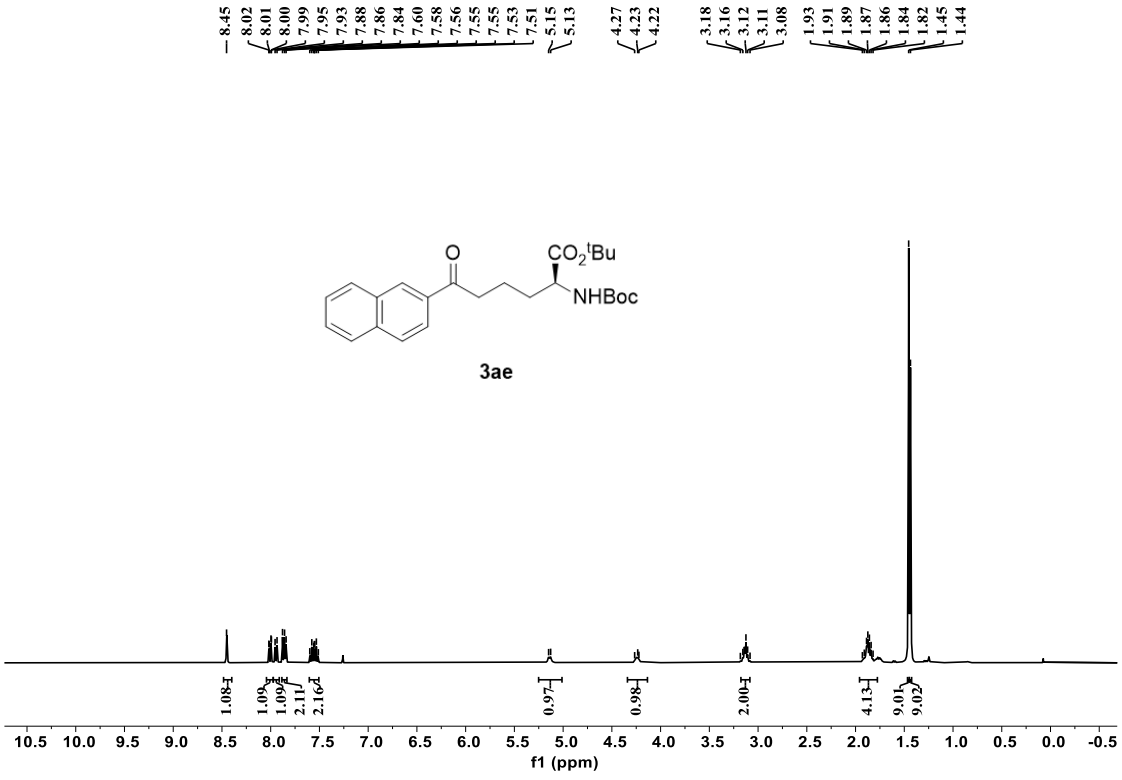
^{1}H NMR (400 MHz, $CDCl_3$)



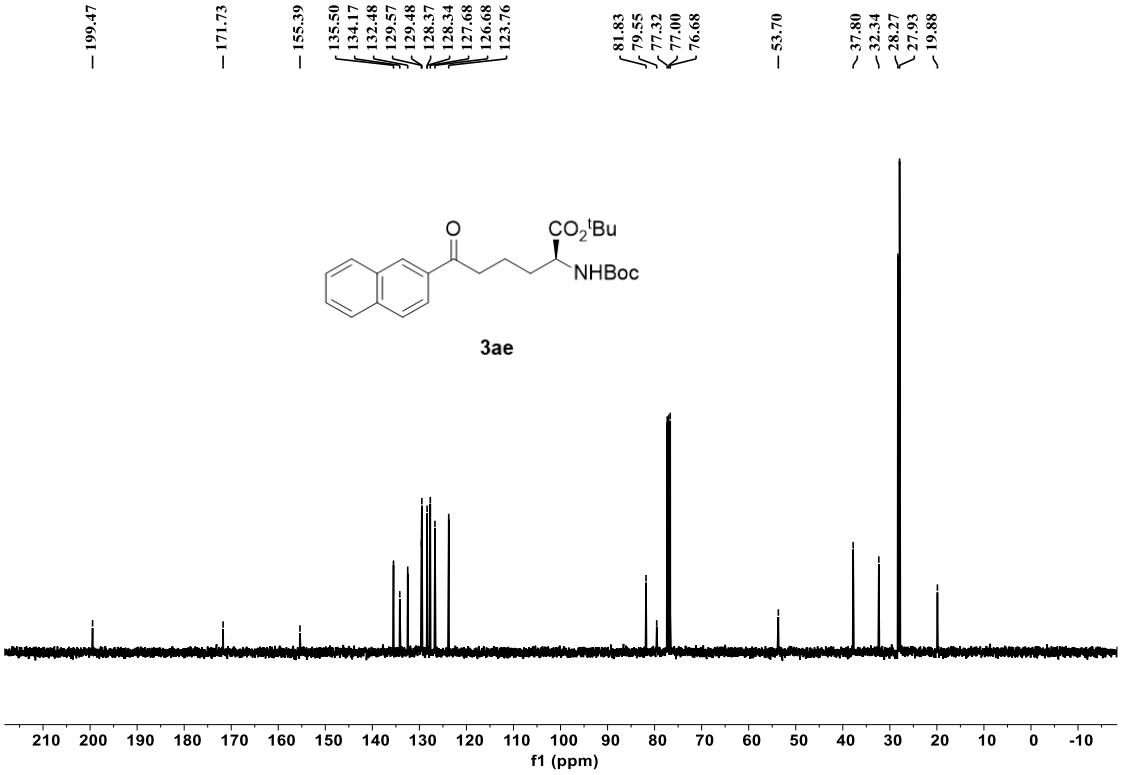
^{13}C NMR (101 MHz, $CDCl_3$)



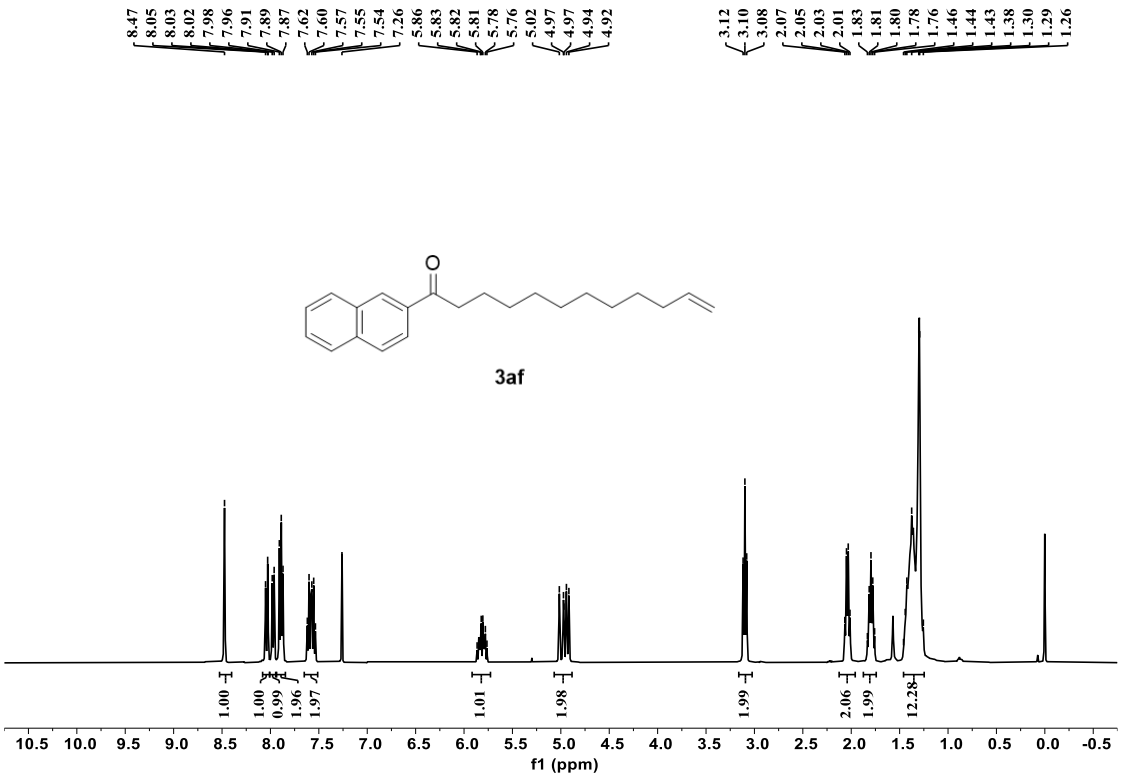
^{1}H NMR (400 MHz, $CDCl_3$)



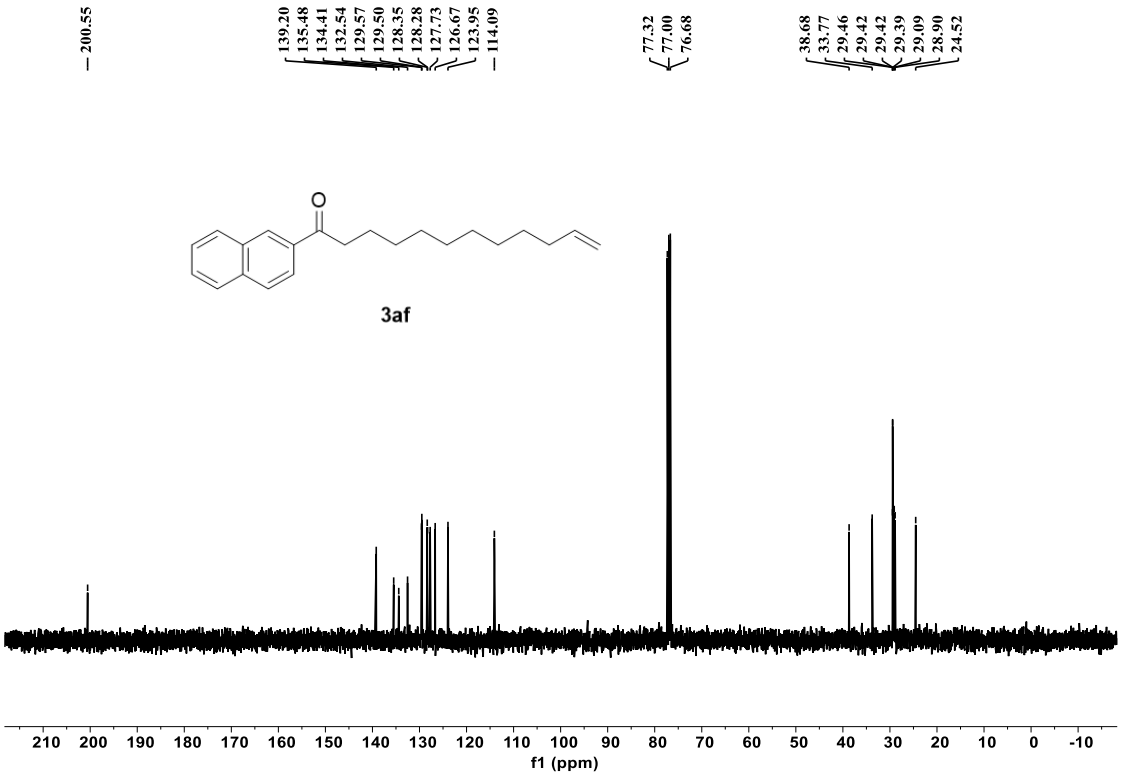
^{13}C NMR (101 MHz, $CDCl_3$)



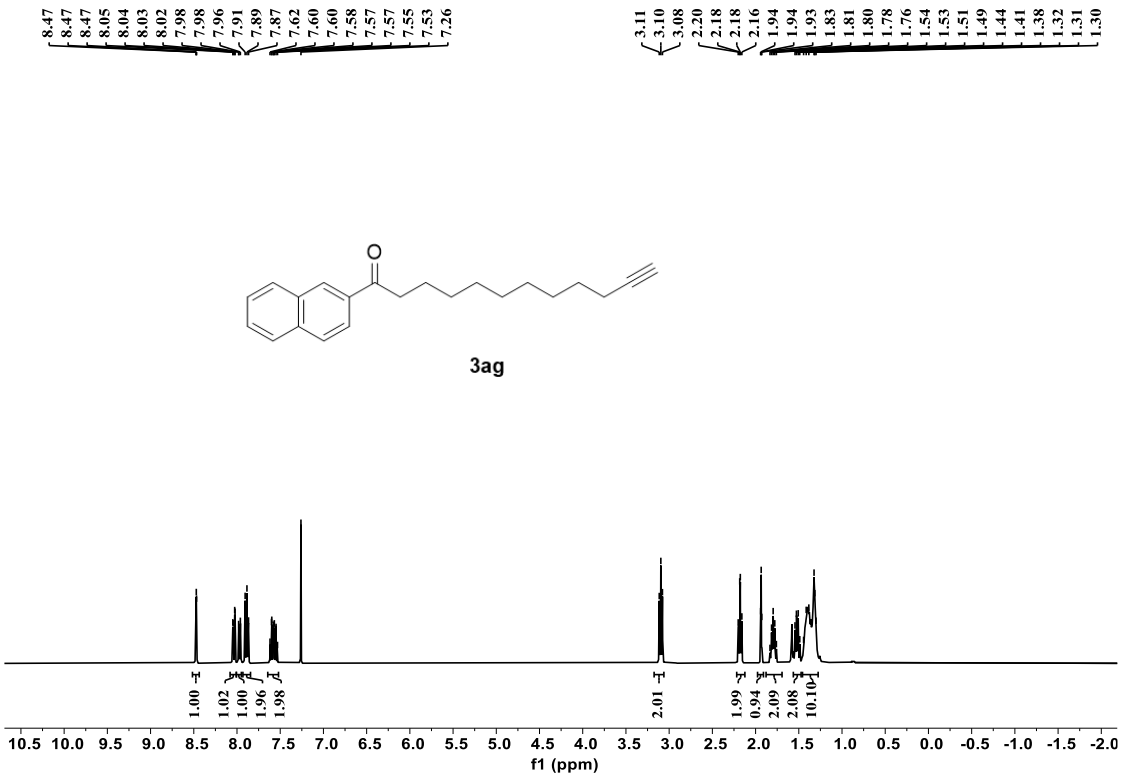
^{1}H NMR (400 MHz, $CDCl_3$)



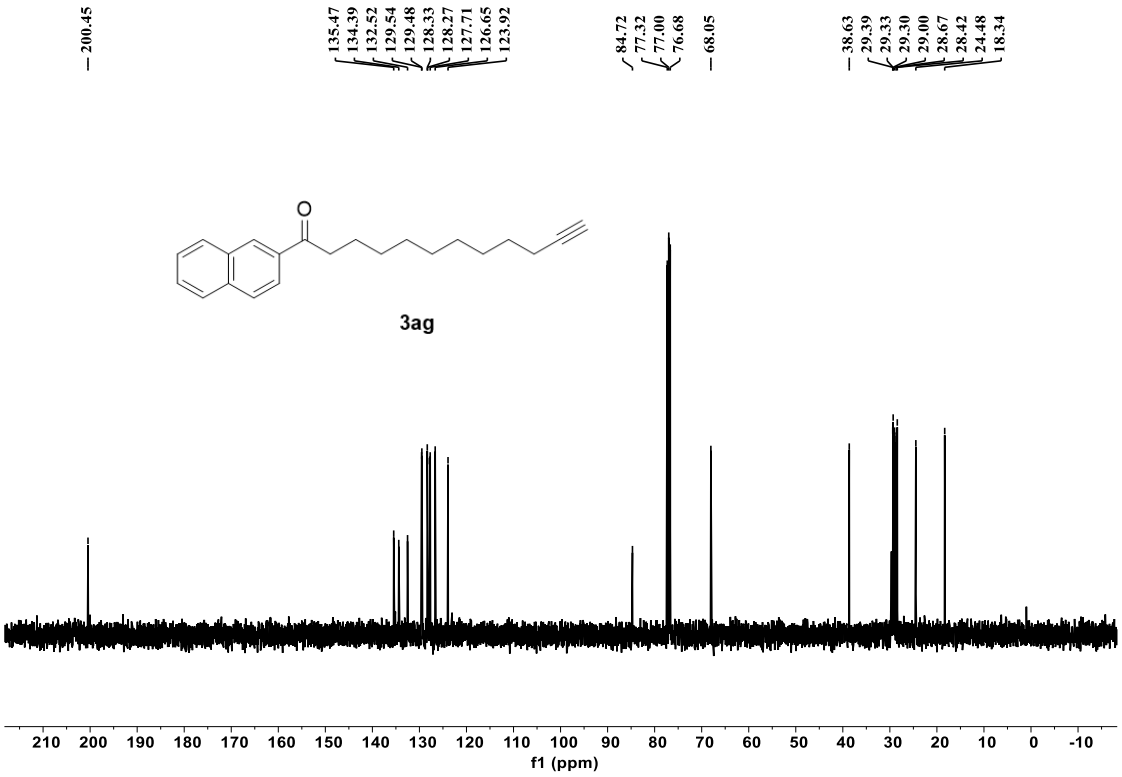
^{13}C NMR (101 MHz, $CDCl_3$)



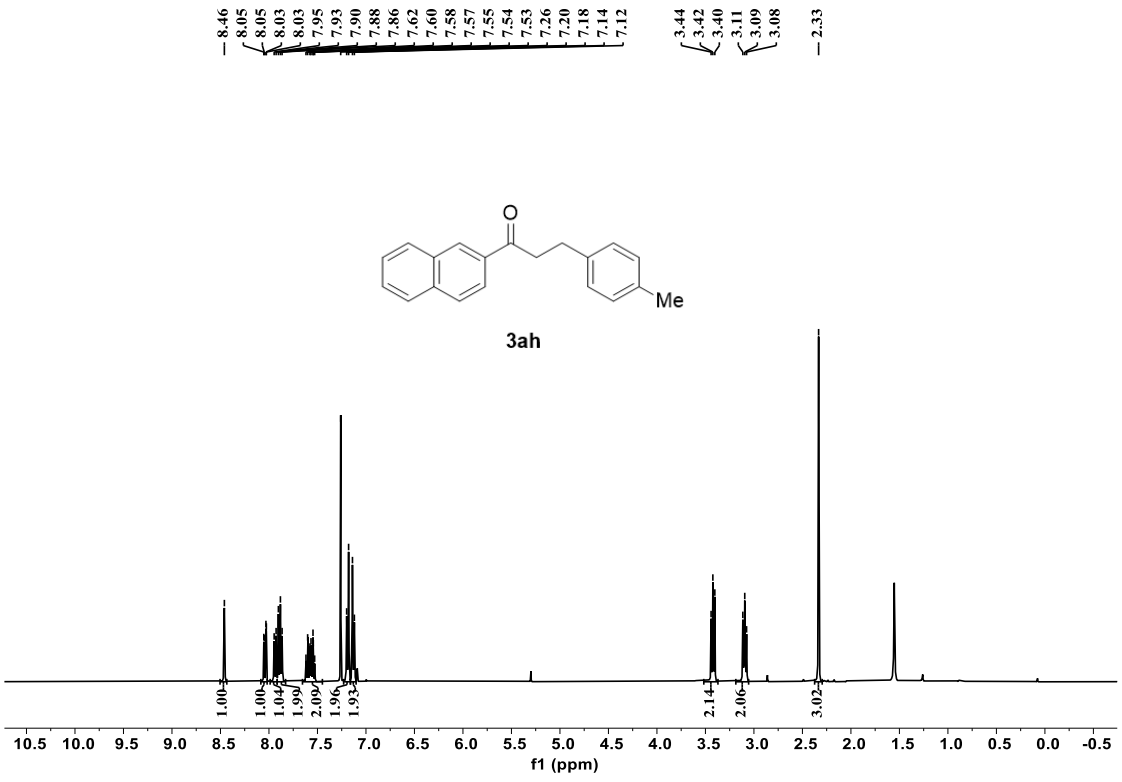
^{1}H NMR (400 MHz, $CDCl_3$)



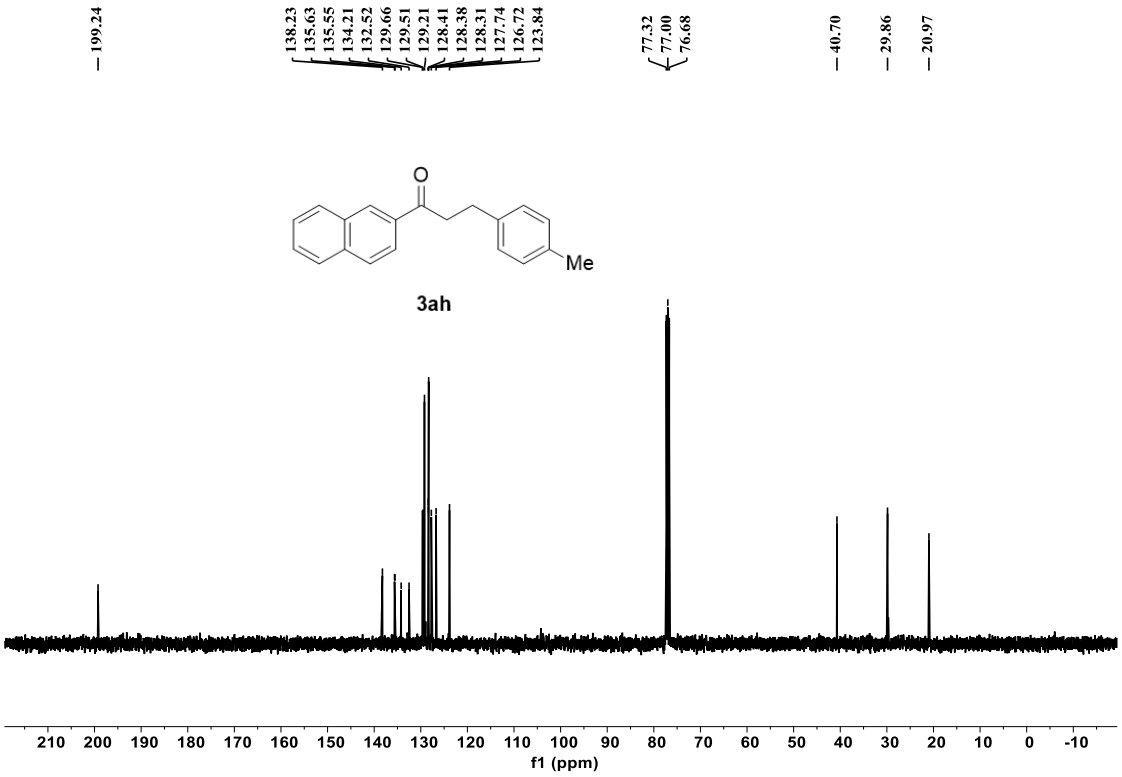
^{13}C NMR (101 MHz, $CDCl_3$)



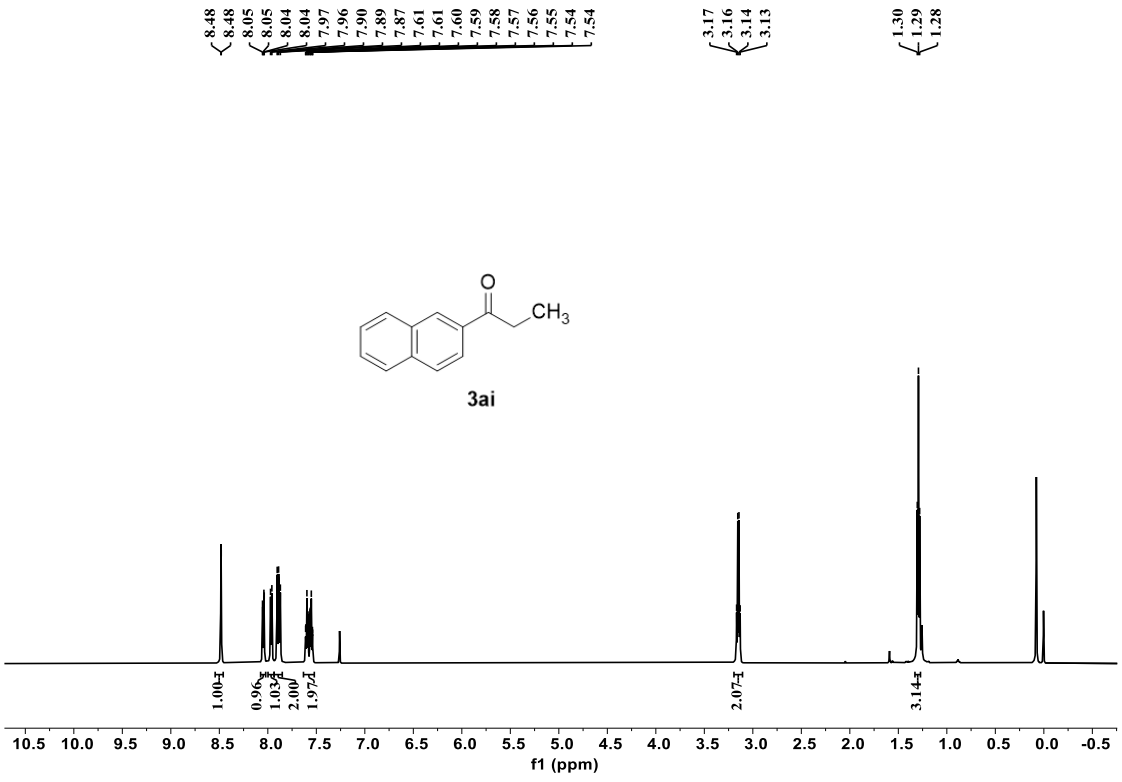
^{1}H NMR (400 MHz, $CDCl_3$)



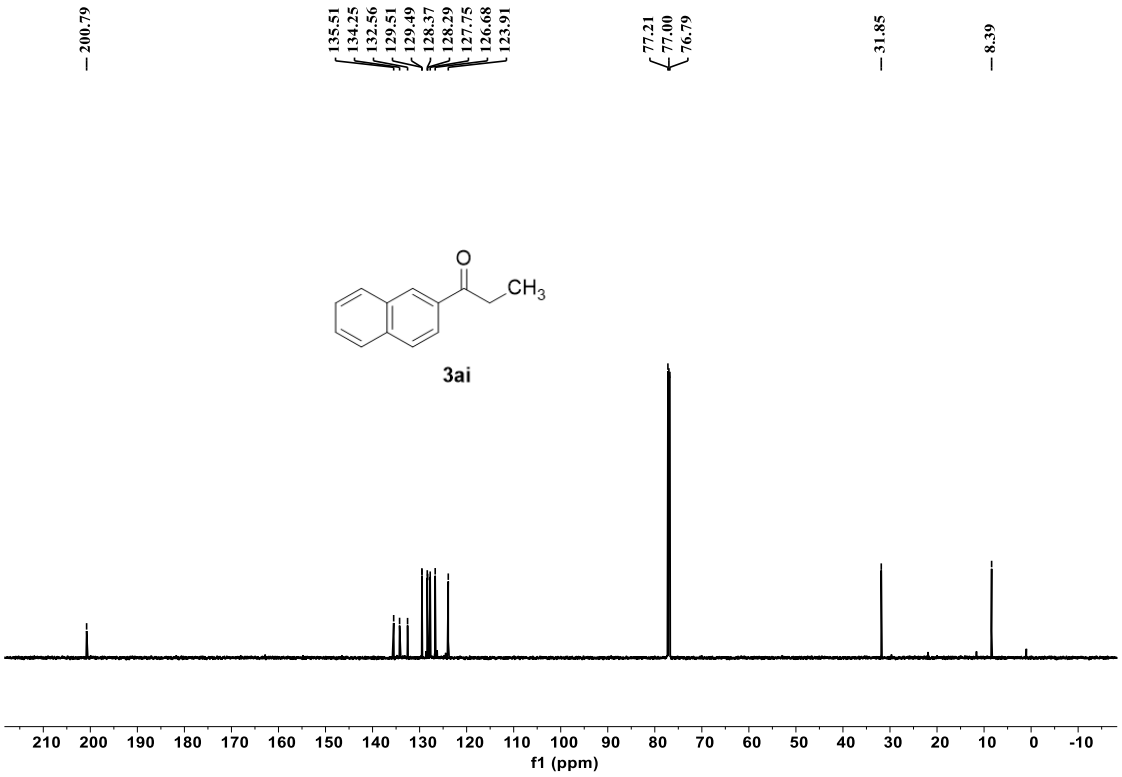
^{13}C NMR (101 MHz, $CDCl_3$)



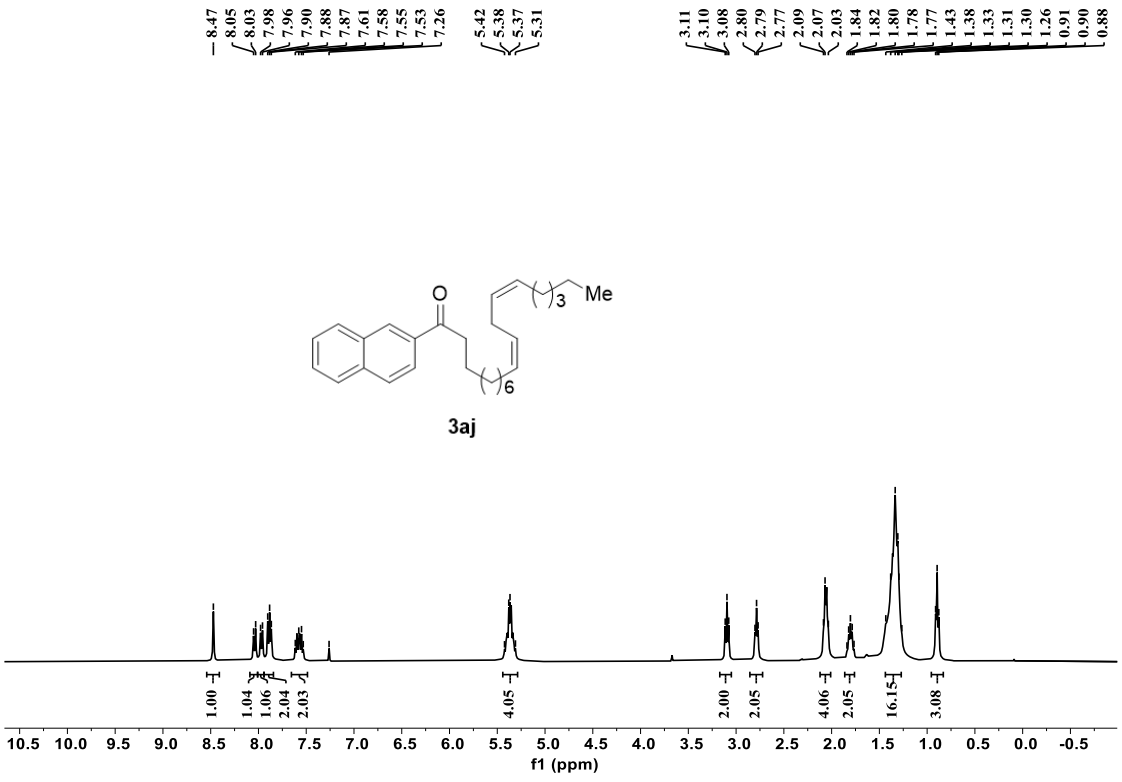
^{1}H NMR (600 MHz, $CDCl_3$)



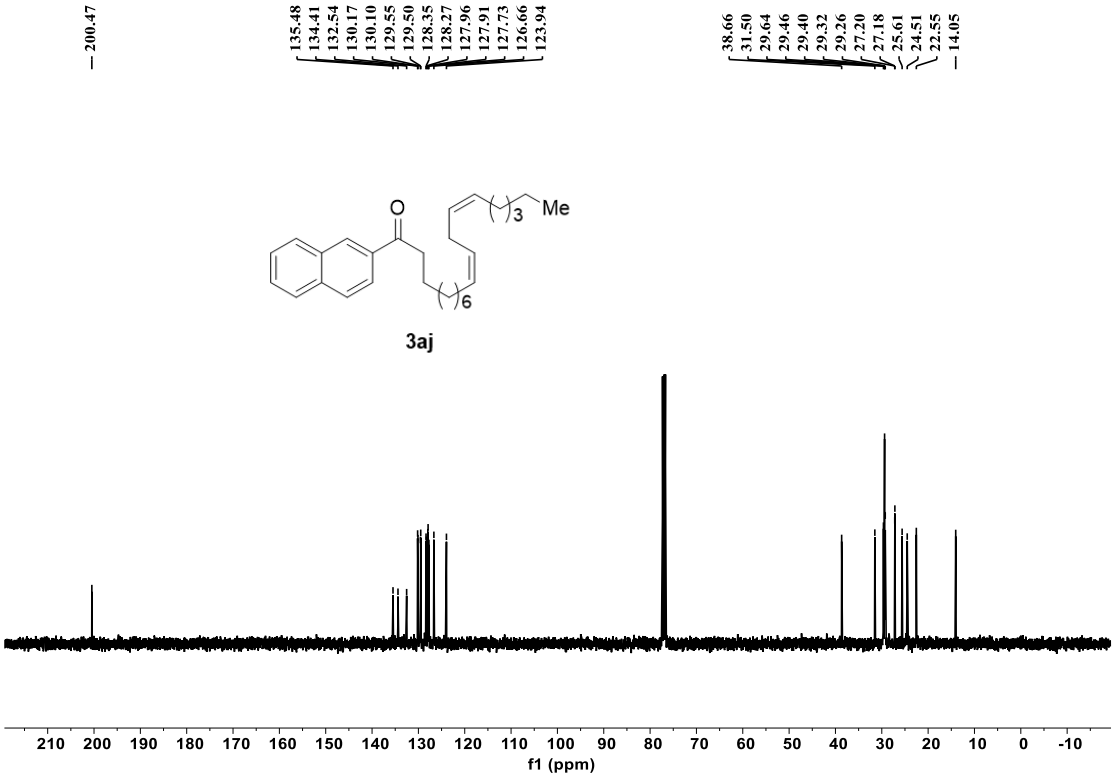
^{13}C NMR (151 MHz, $CDCl_3$)



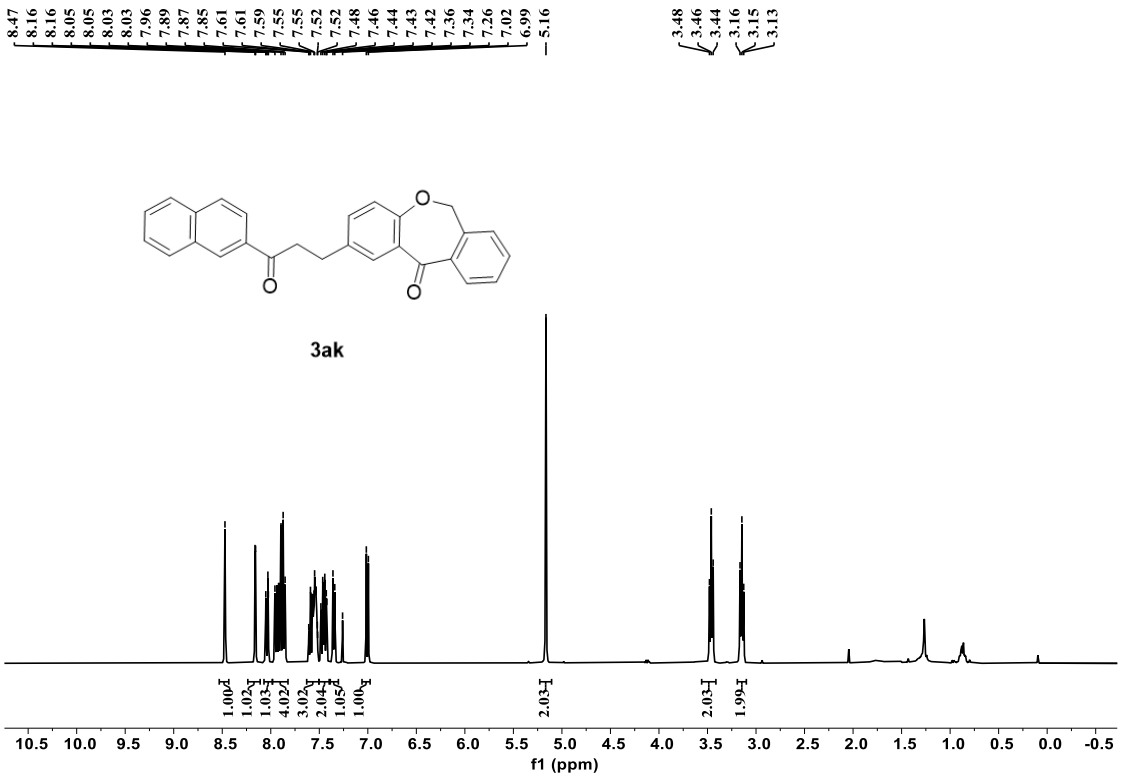
^{1}H NMR (400 MHz, $CDCl_3$)



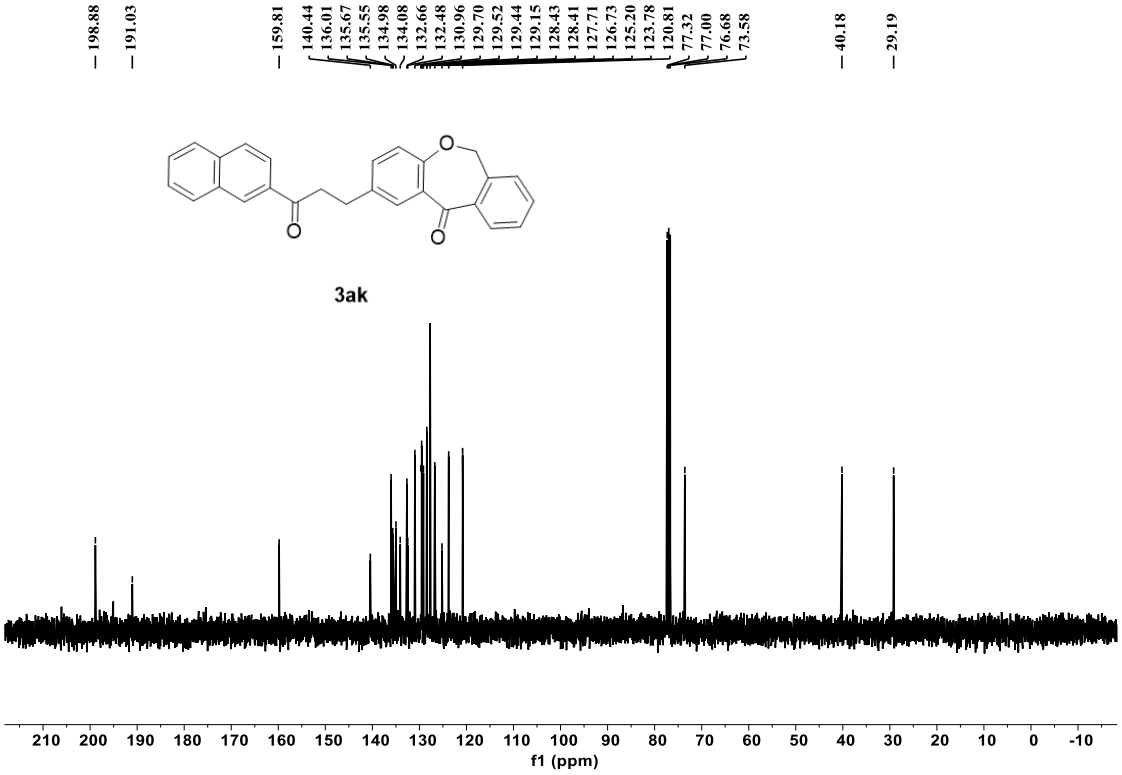
^{13}C NMR (101 MHz, $CDCl_3$)



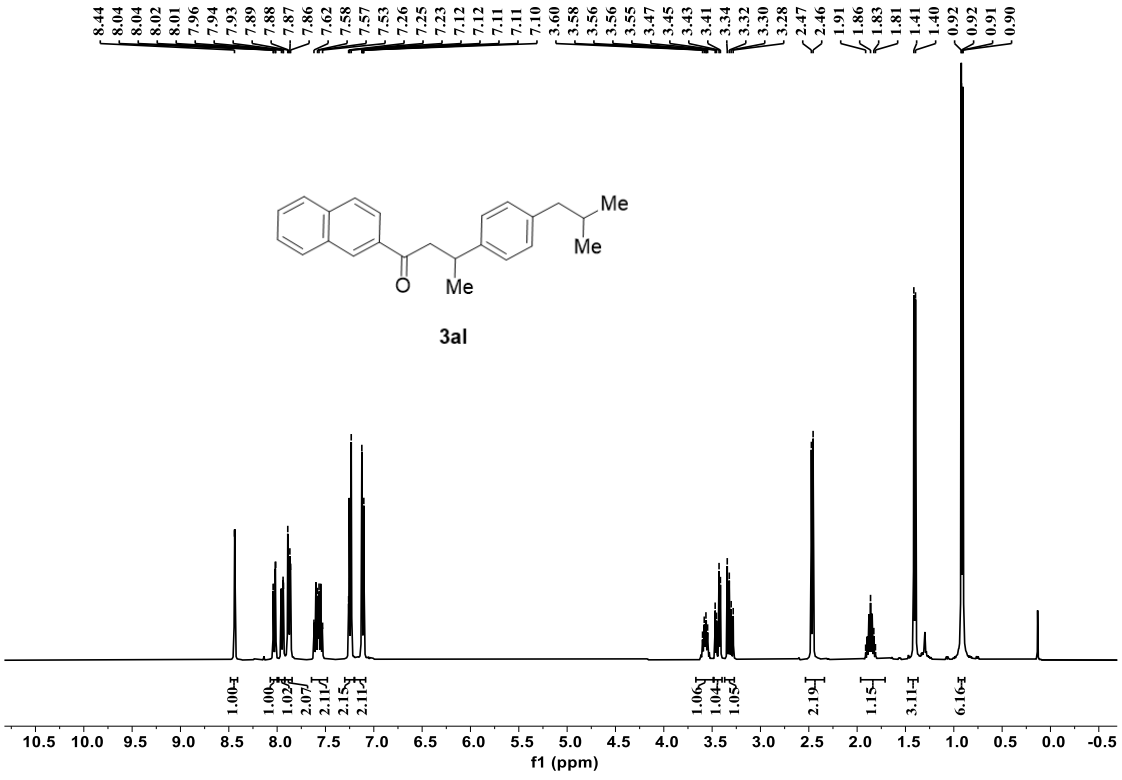
^{1}H NMR (400 MHz, $CDCl_3$)



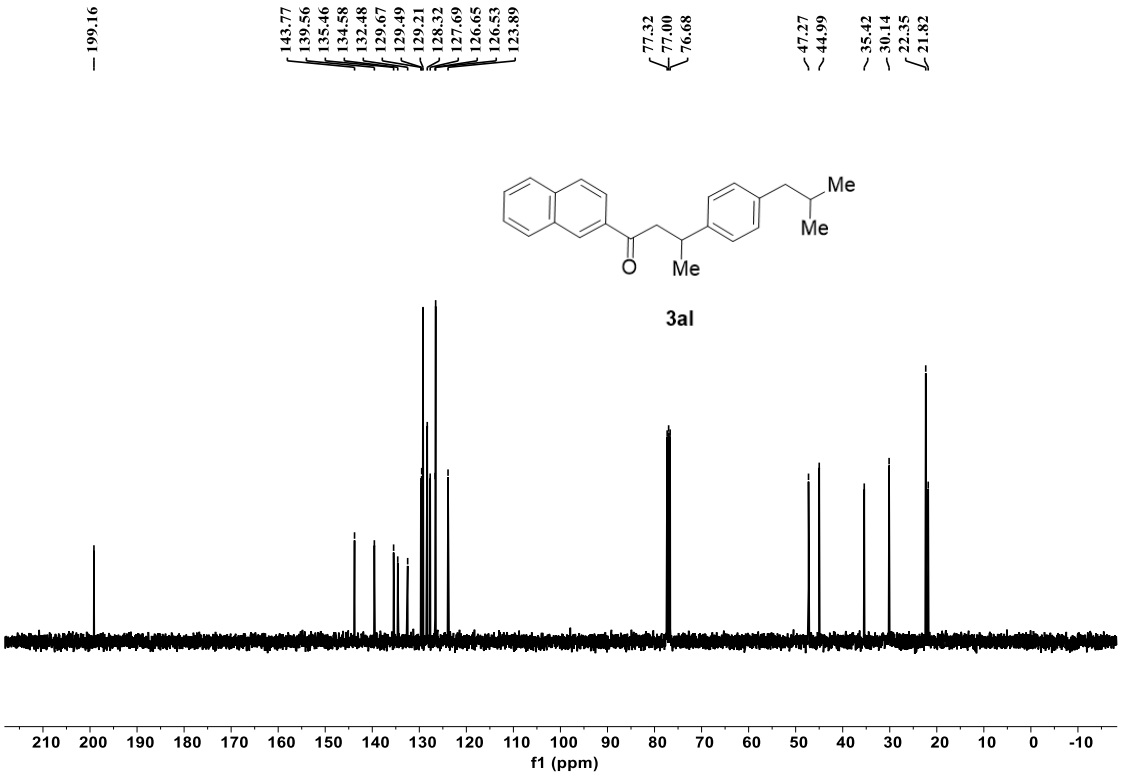
^{13}C NMR (101 MHz, $CDCl_3$)



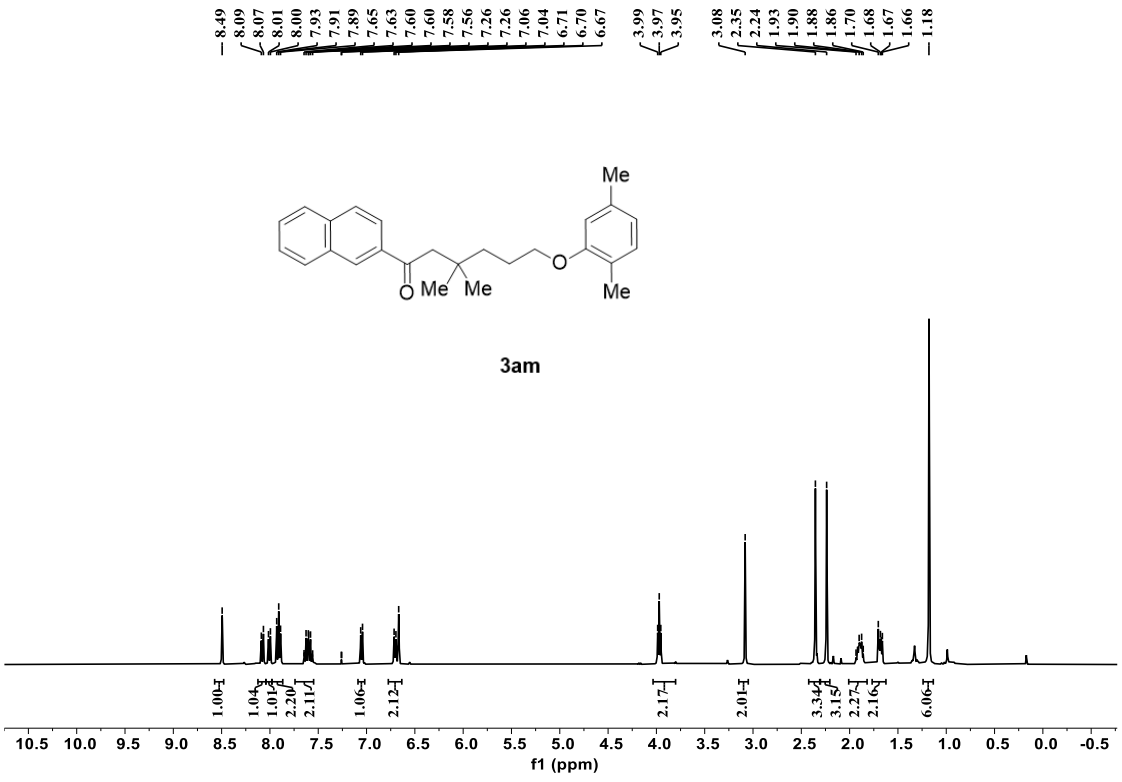
^{1}H NMR (400 MHz, $CDCl_3$)



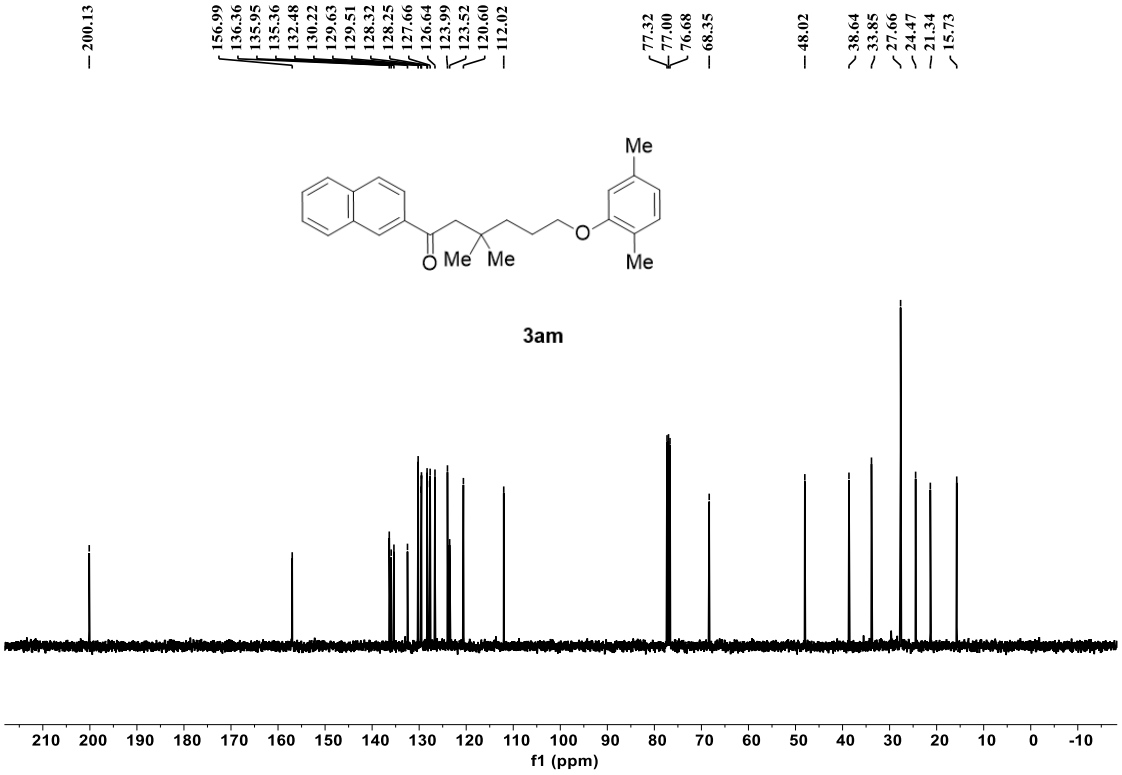
^{13}C NMR (101 MHz, $CDCl_3$)



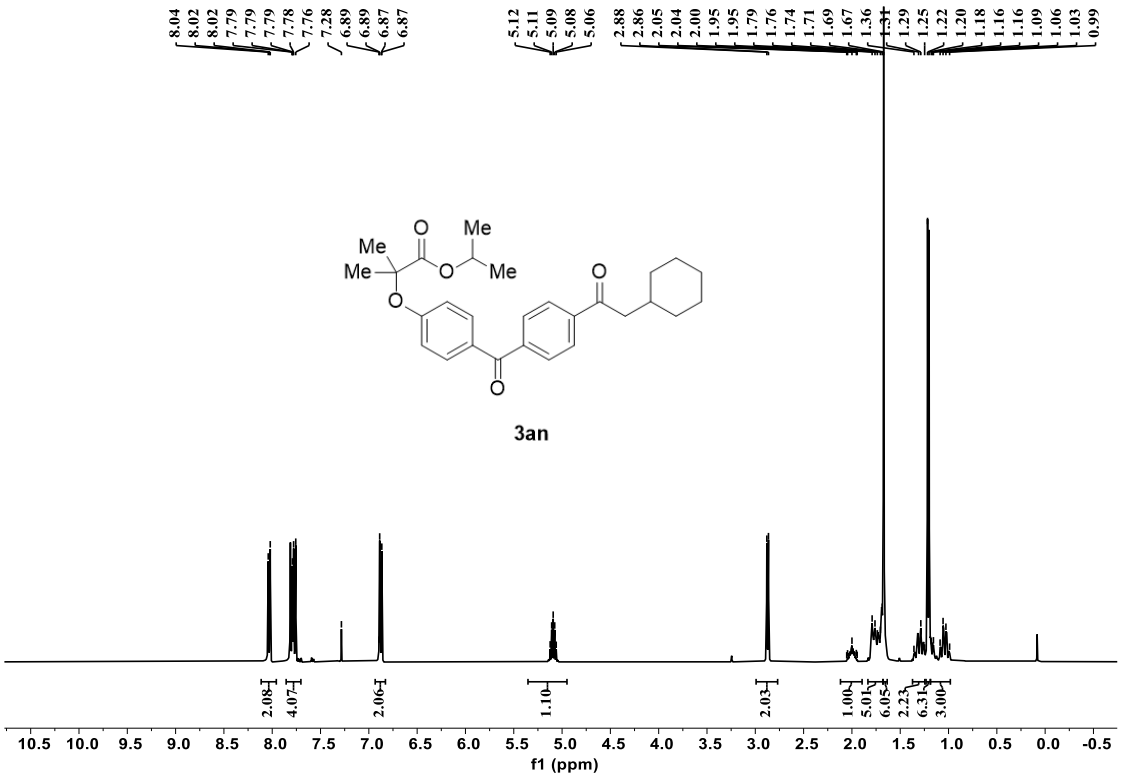
^{1}H NMR (400 MHz, $CDCl_3$)



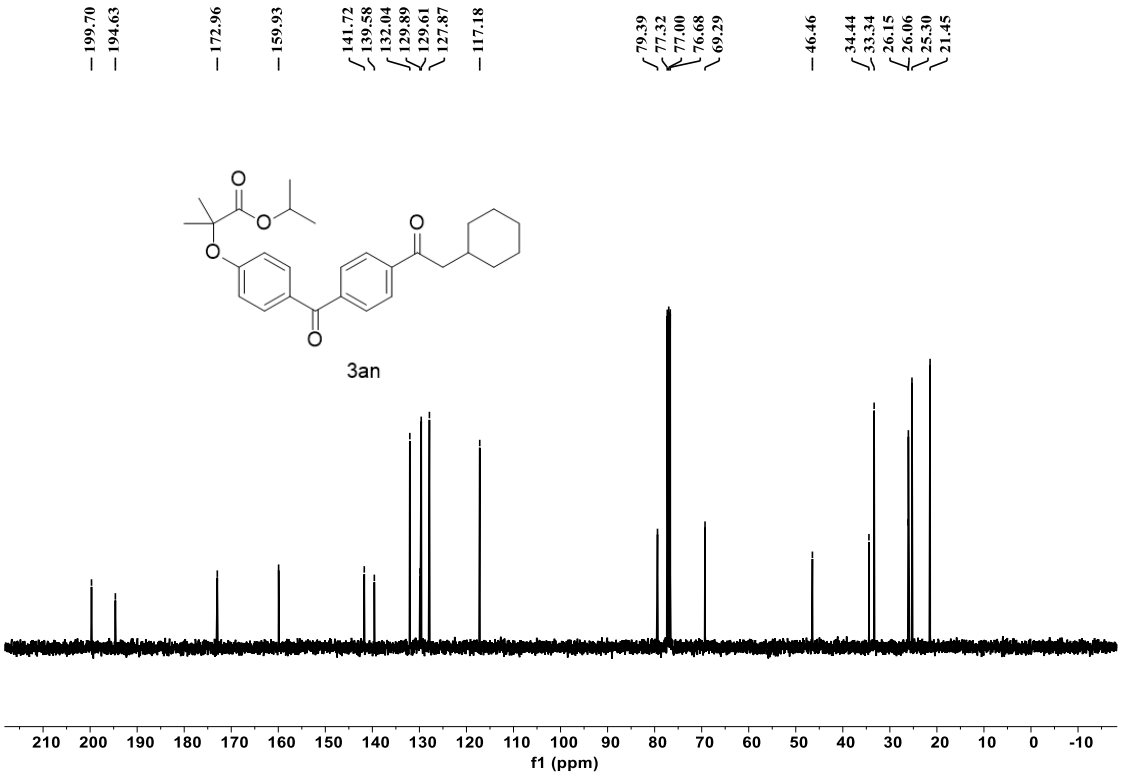
^{13}C NMR (101 MHz, $CDCl_3$)



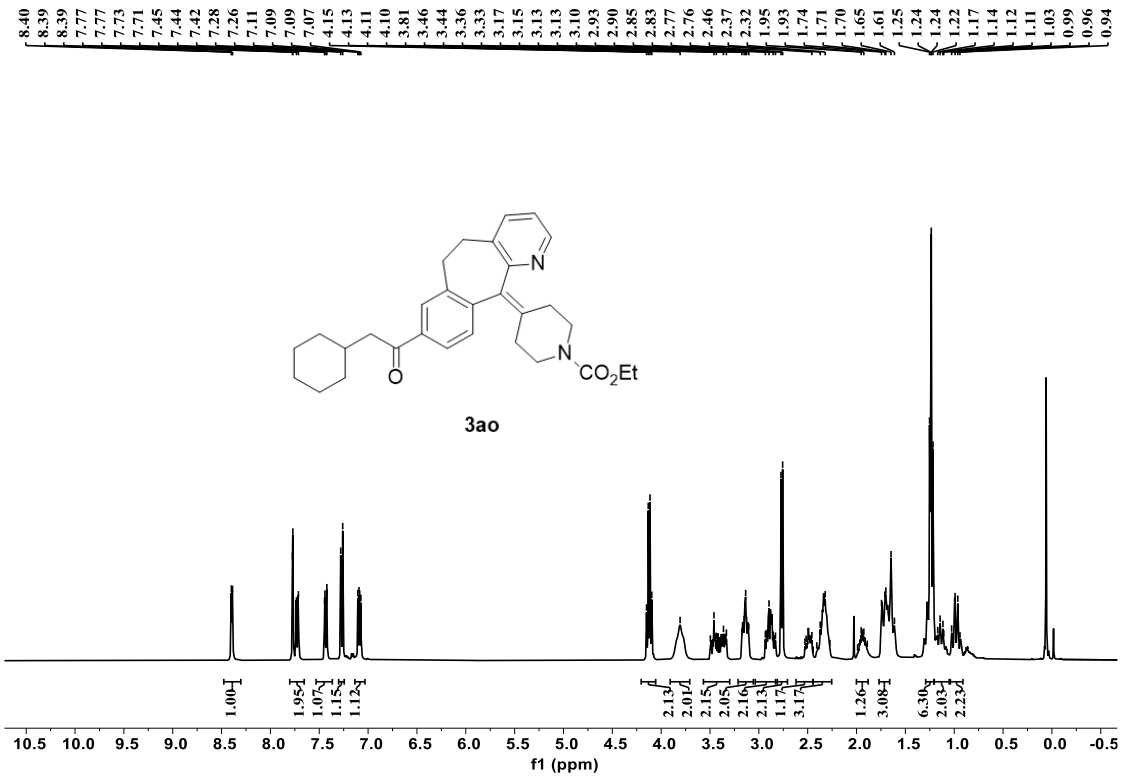
^{1}H NMR (400 MHz, $CDCl_3$)



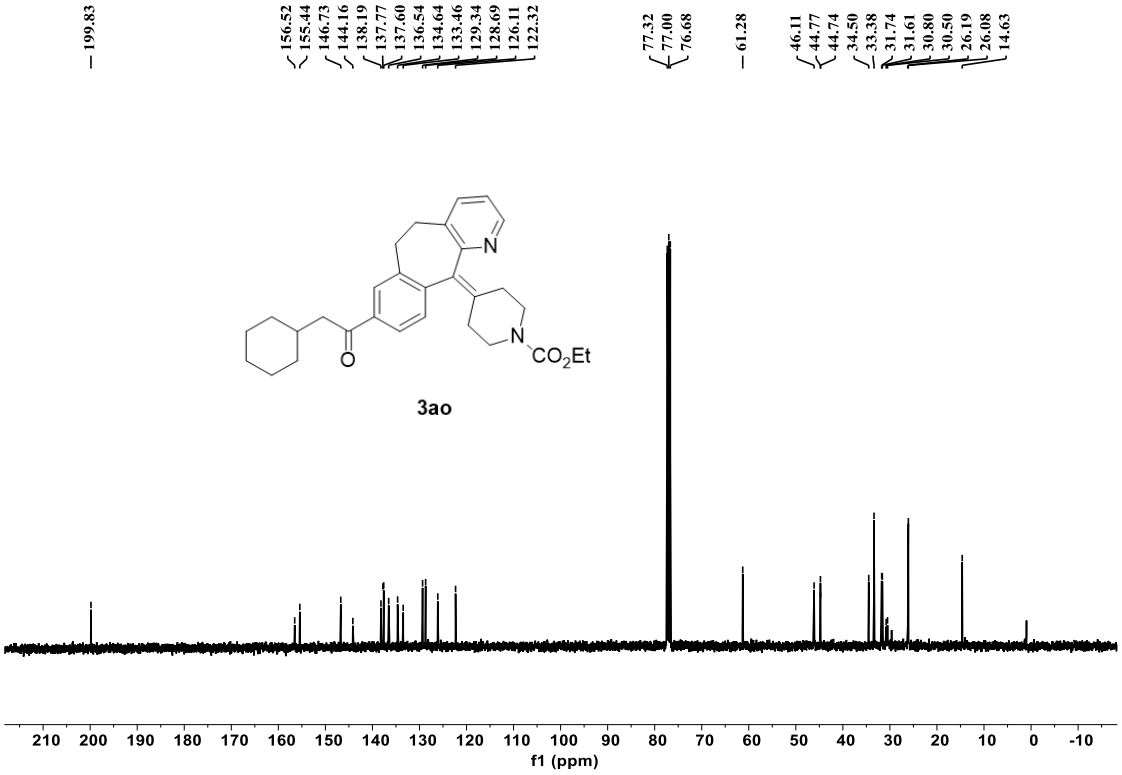
^{13}C NMR (101 MHz, $CDCl_3$)



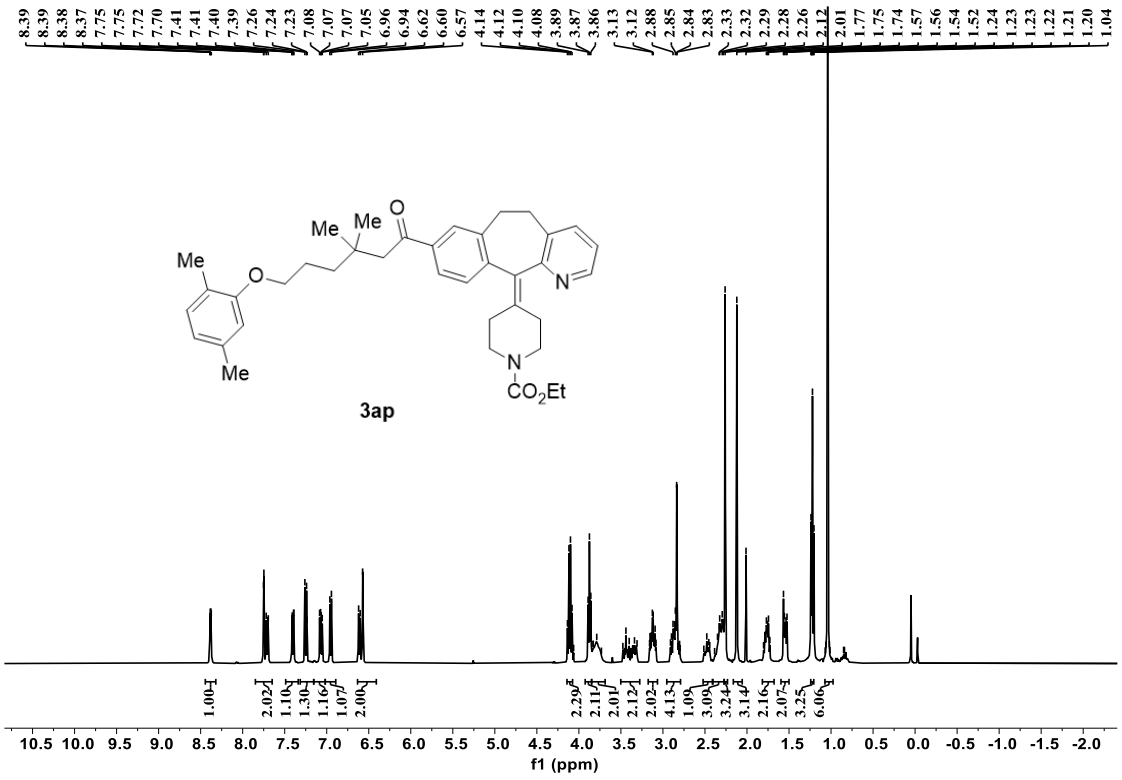
^{1}H NMR (400 MHz, $CDCl_3$)



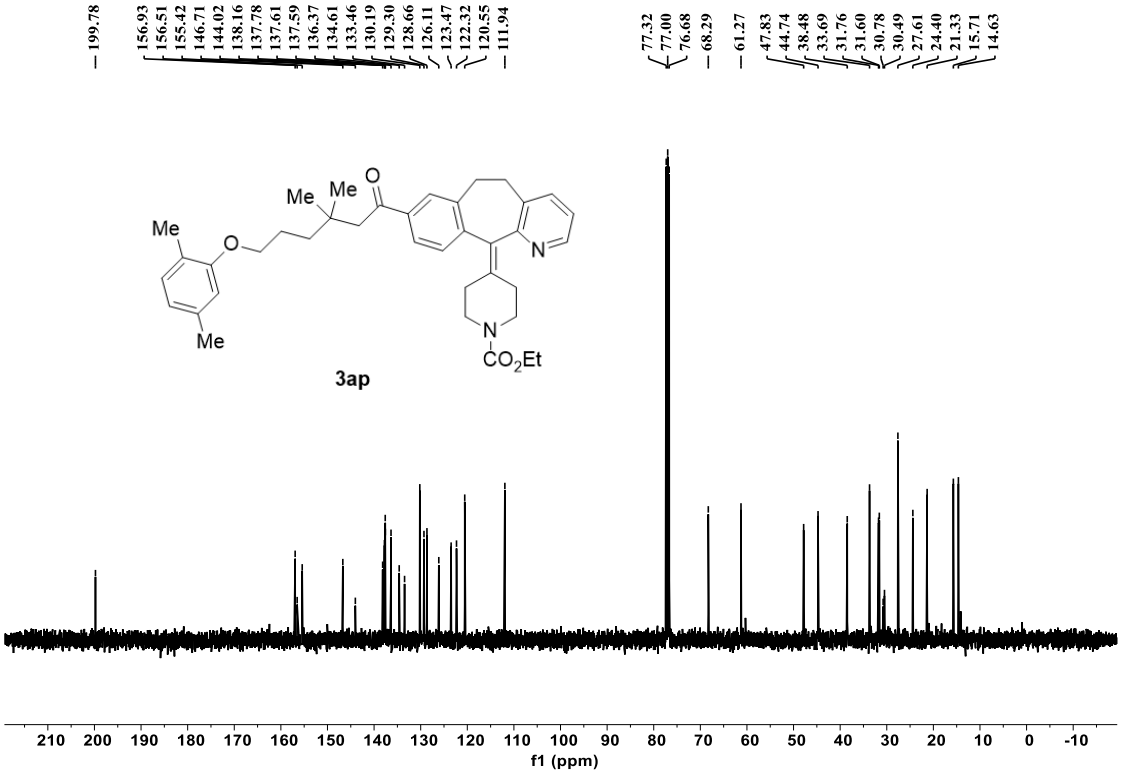
^{13}C NMR (101 MHz, $CDCl_3$)



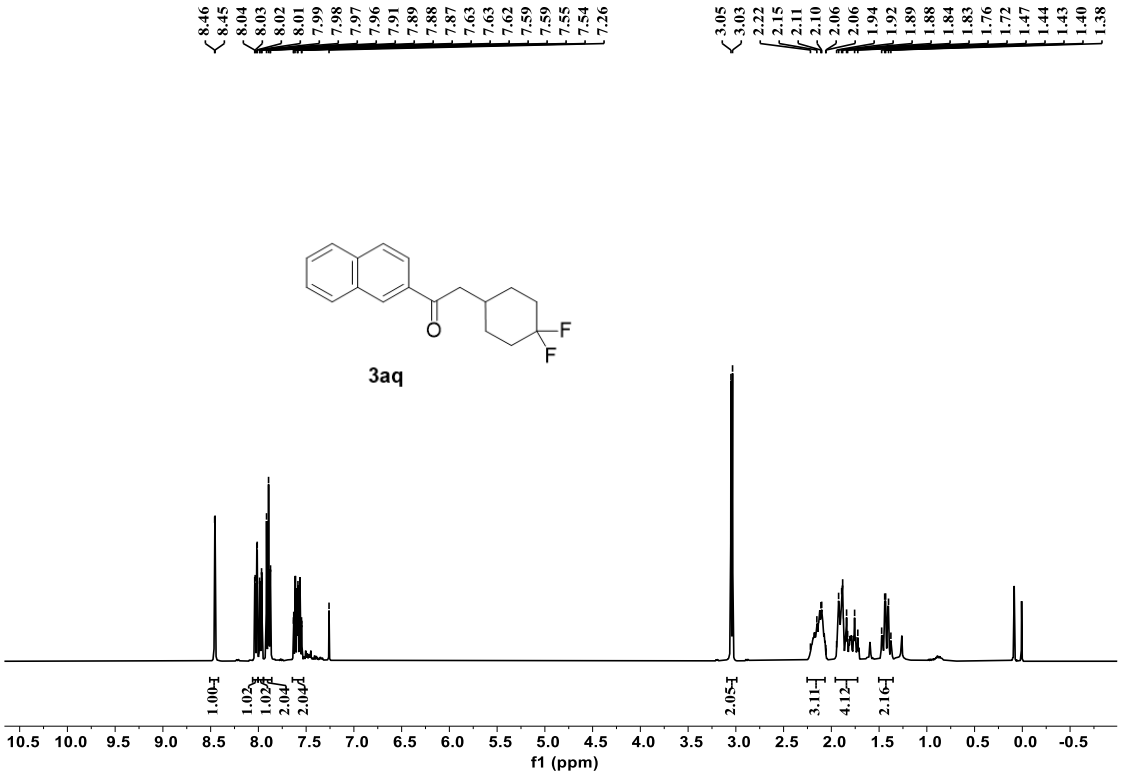
^{1}H NMR (400 MHz, $CDCl_3$)



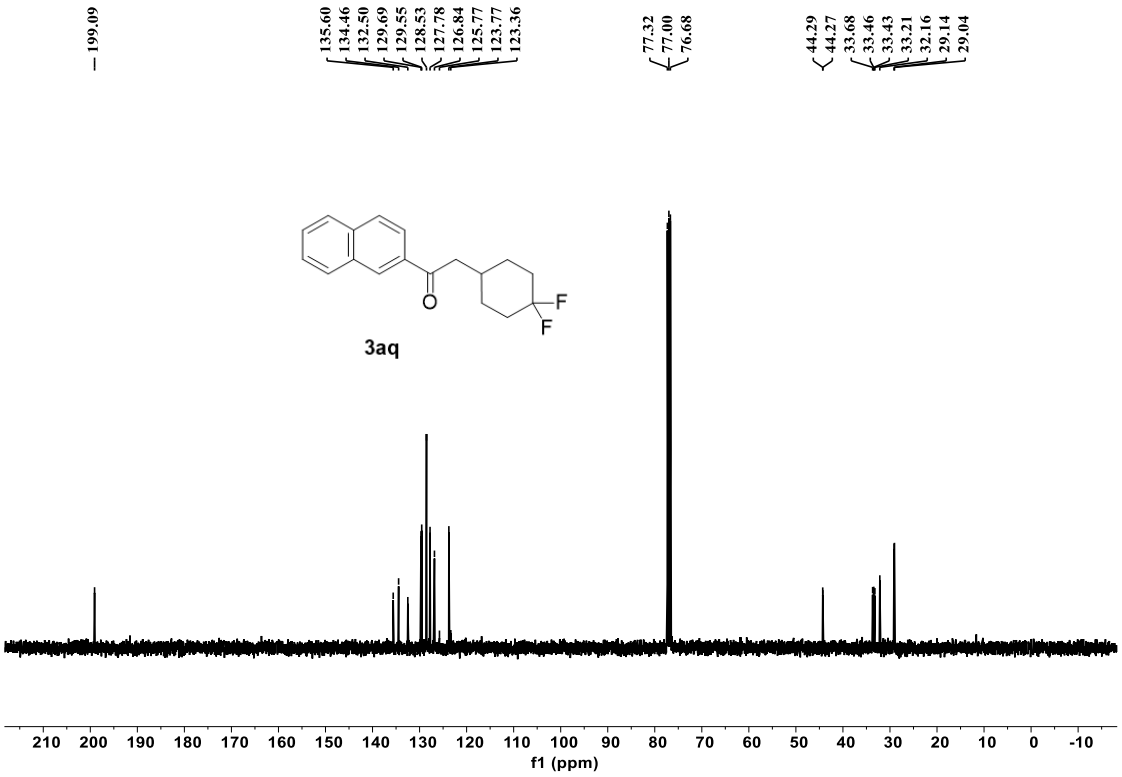
^{13}C NMR (101 MHz, $CDCl_3$)



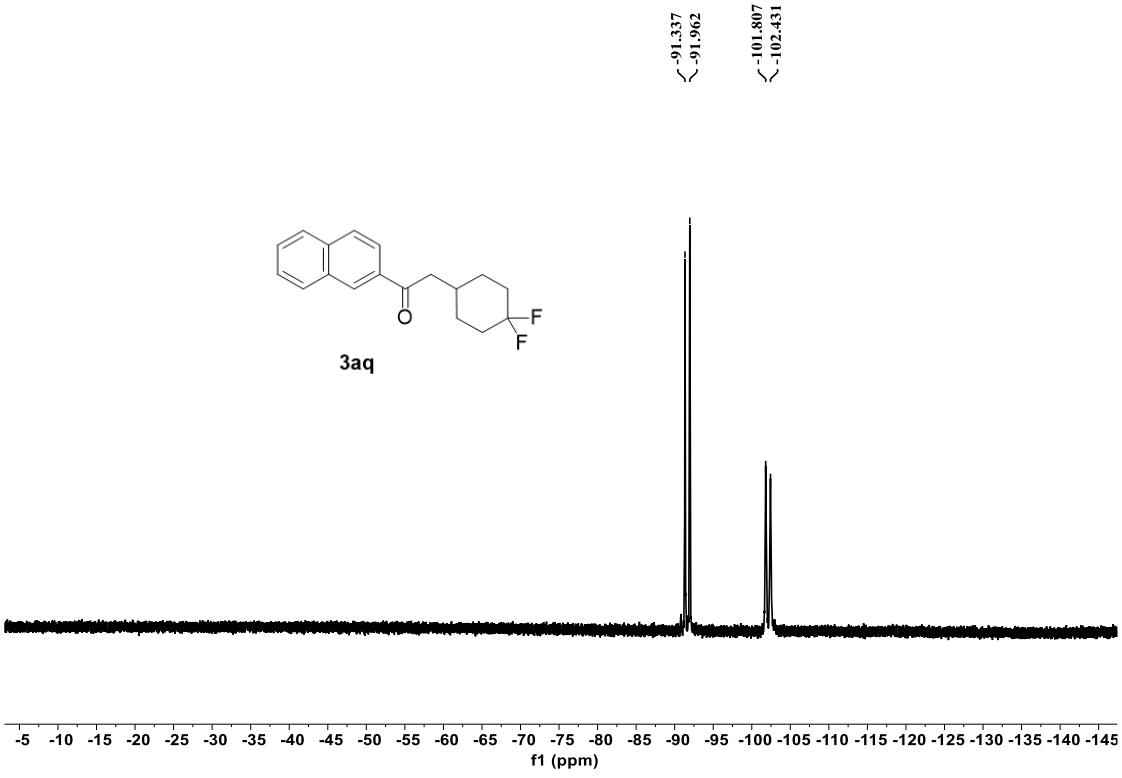
^{1}H NMR (400 MHz, $CDCl_3$)



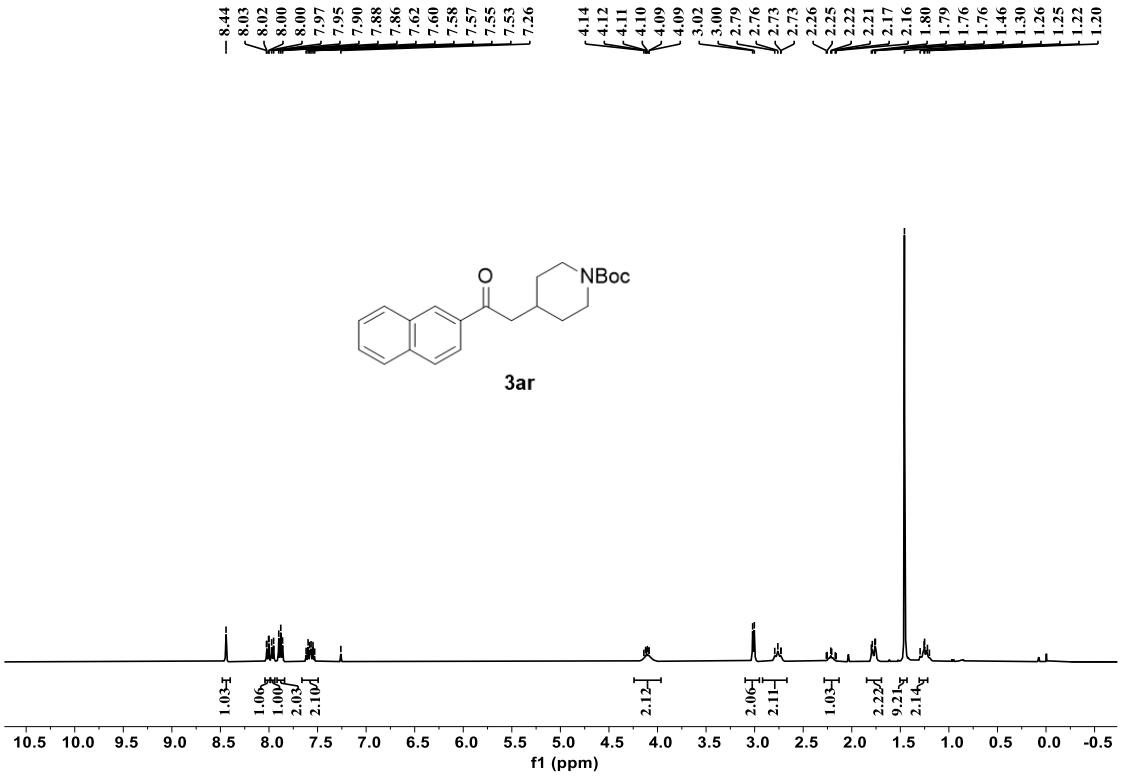
^{13}C NMR (101 MHz, $CDCl_3$)



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



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



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