Supporting Information

Copper-Catalyzed Alkylarylation of Vinylarenes with Cycloalkylsilyl Peroxides and Boronic Acids

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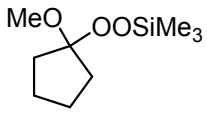
General Information

All catalytic reactions were conducted in oven-dried Schlenk-tube under an atmosphere of nitrogen. Reactions were monitored by thin layer chromatography (aluminum backed plates pre-coated (0.25 mm) with Merck Silica Gel 60F-254.) and visualized using UV light. Column chromatography purifications were carried out using 200-300 mesh silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Advance III-400 and Bruker Advance III-600 in solvents as indicated. Chemical shift are reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: ^{1}H NMR: $\delta = 7.26$; ^{13}C NMR: $\delta = 77.0$). Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). FT-IR spectra were recorded on a Bruker V 70 spectrometer and only major peaks are reported in cm^{-1}. HRMS were obtained on WATERS I-Class VION IMS QTof. Unless otherwise stated, all reagents were purchased from commercial sources and used without further purification.

General Procedure for the Synthesis of Peroxides

All of cycloalkylsilyl peroxides were prepared according to the literature.[1] All of the NMR spectra of known compounds were in full accordance with the data in the literatures.

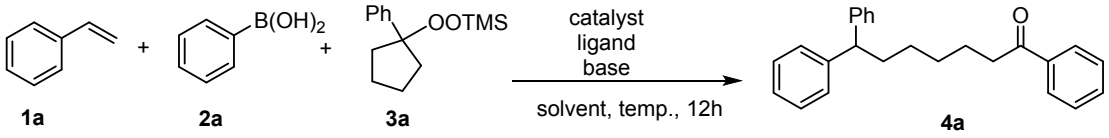
Unknown Compound:



((1-methoxycyclopentyl)peroxy)trimethylsilane (3g): colorless oil; R_f 0.60 (EtOAc/petroleum ether = 1:30); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.30 (s, 3H), 1.99 – 1.92 (m, 2H), 1.74 – 1.63 (m, 6H), 0.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 117.00, 50.37, 33.67, 23.92, -1.26. IR (neat): υ_{max} 2959, 2906, 2876, 2831, 1468, 1452, 1439, 1408, 1331, 1310, 1250, 1190, 1161, 1103, 1049, 1032, 997, 972, 893, 841, 743, 692, 617 cm^{-1}; HRMS (ESI) calcd for $C_9H_{21}O_3Si$ $[M+H]^+$ 205.1254, found 205.1255.

Optimization of Reaction Conditions

General Procedure for the Reaction of Styrene 1a, Phenylboronic Acid 2a and Cyclopentylsilyl Peroxide 3a

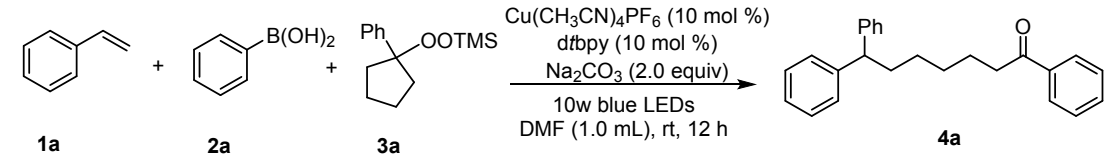


1a 2a 3a 4a

In an N_2-filled glovebox, to an oven dried 10 mL Schlenk-tube containing a magnetic stir bar was added catalyst, ligand, phenylboronic acid **2a** and Base. Subsequently, a solution of styrene **1a** and cyclopentyl silyl peroxide **3a** in solvent was injected into the tube by syringe. Then, the reaction mixture was moved outside the glovebox and stirred at the specified temperature for the indicated reaction time. After that, the mixture was diluted with EtOAc (10 mL) and saturated saline (10 mL). The organic phase was washed with saturated brine (3×8 mL) and the water phase was extracted with EtOAc (3×6 mL). The combined organic phase was dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel (petroleum ether/ethylacetate 120:1) directly to give the desired product **4a** as a white solid. The results are summarized as following

Table S1. Optimization of the Reaction of Styrene 1a, Boronic Acid 2a and Cycloalkylsilyl Peroxide 3a

Control Experiment[a]

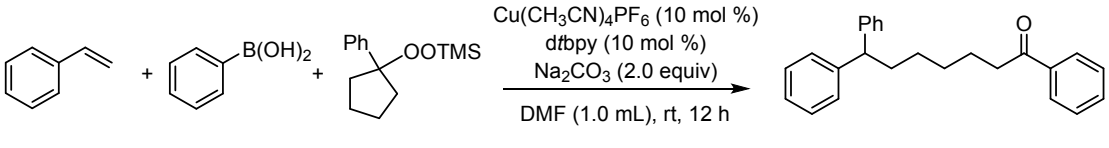


1a 0.4 mmol + **2a** 0.4 mmol + **3a** 0.2 mmol → **4a**

Entry	[Cu]	Ligand	*hv*	Base	Yield(%)[b]
1	+	+	+	+	40
2[c]	-	+	+	+	0
3[d]	+	-	+	+	15
4[e]	+	+	-	+	42
5[f]	+	+	+	-	9

[a]Reaction conditions: **1a** (41.7 mg, 0.4 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (50.1 mg, 0.2 mmol), $Cu(CH_3CN)_4PF_6$ (7.5 mg, 10 mol %), d*t*bpy (5.4 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard. [c]Without Cu. [d]Without ligand. [e]No hv. [f]Without base.

Substrate ratio[a]

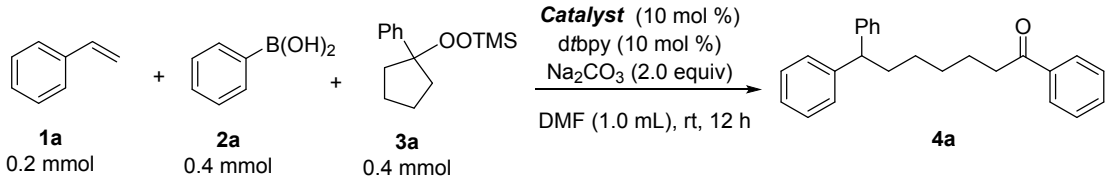


1a + **2a** + **3a** → **4a**

Entry	Ratio (**1a** : **2a** : **3a**)	Yield (%)[b]
1	2 : 2 : 1	42
2	1 : 1.5 : 1.5	36
3	**1 : 2 : 2**	**55**
4	1 : 2.5 : 2	54
5	1.5 : 1 : 2	30

[a]Reaction conditions: **1a** (x mmol), **2a** (y mmol), **3a** (z mmol), $Cu(CH_3CN)_4PF_6$ (7.5 mg, 10 mol %), d*t*bpy (5.4 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

***Catalyst*[a]**

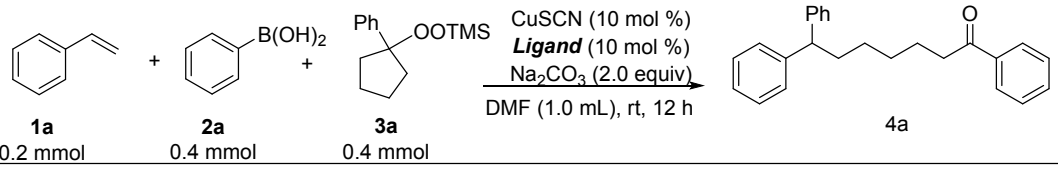


1a 0.2 mmol + **2a** 0.4 mmol + **3a** 0.4 mmol → **4a**

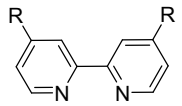
Entry	Catalyst	Yield (%)[b]
1	$Cu(CH_3CN)_4PF_6$	55
2	CuI	52
3	CuOTf	34
4	CuOAc	43
5	CuCl	25
6	CuBr	48
7	**CuSCN**	**57**
8	$Cu(CH_3CN)_4BF_4$	35
9	CuTc	28
10	$FeCl_2$	0
11	$NiBr_2$·diglyme	0
12	$NiCl_2(py)_4$	0
13	$PdCl_2$	0
14	$Pd(OAc)_2$	0

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), Catalyst (10 mol %), d*t*bpy (5.4 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

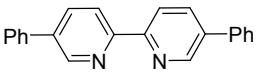
Ligand[a]



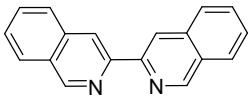
1a 0.2 mmol | **2a** 0.4 mmol | **3a** 0.4 mmol | 4a



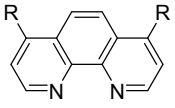
R = ^{t}Bu, L1
R = H, L2
R = Me, L3
R = Ph, L4
R = OMe, L5
R = Br, L6
R = CO_2Me, L7



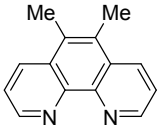
L8



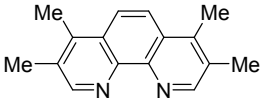
L9



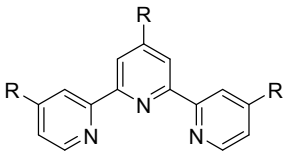
R = H, L10
R = Ph, L11
R = Me, L12
R = OMe, L13



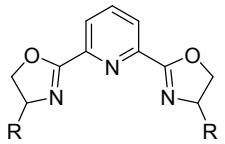
L14



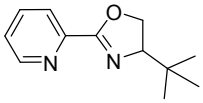
L15



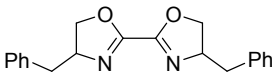
R = H, L16
R = ^{t}Bu, L17



R = H, L18
R = Me, L19
R = ^{i}Pr, L20



L21

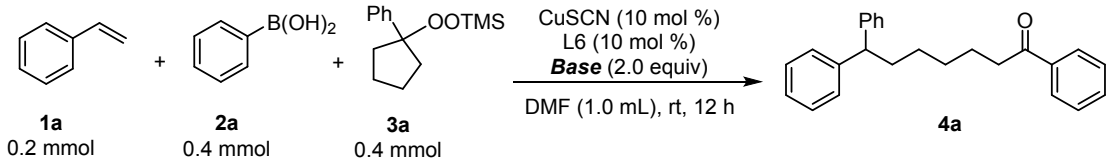


L22

Entry	Ligand	Yield (%)[b]
1	-	29
2	L1	57
3	L2	44
4	L3	49
5	L4	39
6	L5	61
7	**L6**	**70**
8	L7	65
9	L8	40
10	L9	37
11	L10	43
12	L11	58
13	L12	55
14	L13	55
15	L14	54
16	L15	55
17	L16	30
18	L17	trace
19	L18	42
20	L19	37
21	L20	51
22	L21	30
23	L22	31

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (2.4 mg, 10 mol %), Ligand (10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Base[a]

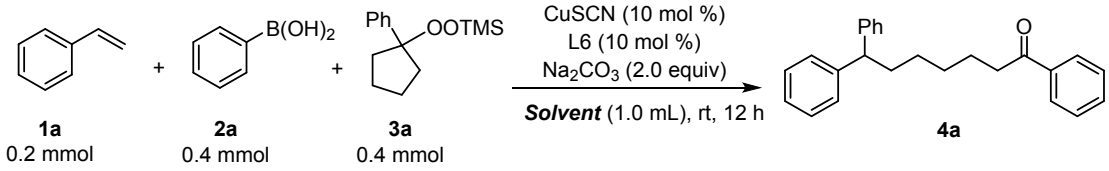


1a 0.2 mmol | **2a** 0.4 mmol | **3a** 0.4 mmol | **4a**

Entry	Base	Yield (%)[b]
1	**Na_2CO_3**	**70**
2	Li_2CO_3	58
3	Cs_2CO_3	50
4	K_2CO_3	53
5	$KHCO_3$	56
6	$NaHCO_3$	68
7	NaOAc	trace
8	NaOH	45
9	Na_3PO_4	59
10	Na_2HPO_4	54
11	NaF	58
12	Et_3N	43
13	DBU	trace

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (2.4 mg, 10 mol %), L6 (6.27 mg, 10 mol %), Base (0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Solvent[a]

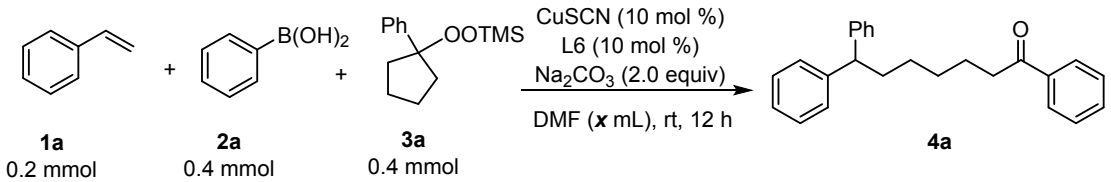


1a 0.2 mmol + **2a** 0.4 mmol + **3a** 0.4 mmol → **4a**

Entry	Solvent	Yield (%)[b]
1	**DMF**	**70**
2	DMAc	48
3	DMSO	58
4	MeOH	10
5	MeCN	33
6	1,4-dioxane	53
7	THF	54
8	MTBE	trace
9	Toluene	0
10	$PhCF_3$	0
11	Cyclohexane	0
12	DMF : DCM = 4 : 1	51

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (2.4 mg, 10 mol %), L6 (6.27 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), Solvent (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Concentration[a]

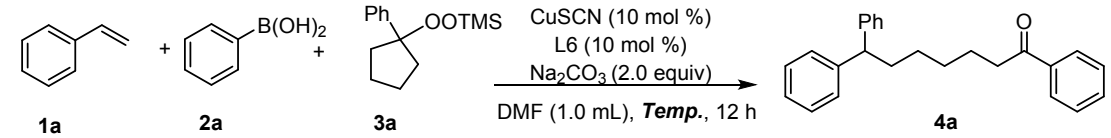


1a 0.2 mmol | **2a** 0.4 mmol | **3a** 0.4 mmol | **4a**

Entry	Concentration (*x* mL)	Yield (%) [b]
1	0.5	63
2	**1**	**70**
3	2	62
4	3	58

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (2.4 mg, 10 mol %), L6 (6.27 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (*x* mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Temperature[a]

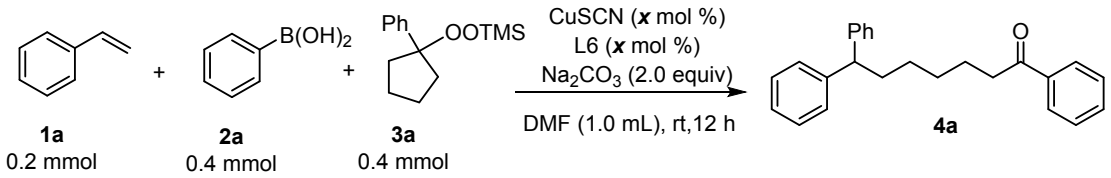


1a 0.2 mmol | **2a** 0.4 mmol | **3a** 0.4 mmol | **4a**

Entry	Temp. (*x* °C)	Yield (%) [b]
1	**rt**	**70**
2	40	60
3	50	58

[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8 mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (2.4 mg, 10 mol %), L6 (6.27 mg, 10 mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at *x* °C for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Loading of catalyst[a]

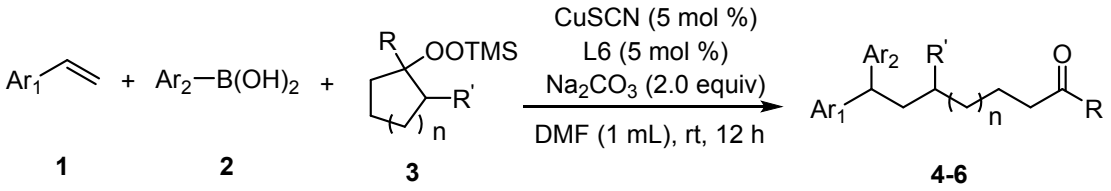


1a 0.2 mmol + **2a** 0.4 mmol + **3a** 0.4 mmol → **4a**

Entry	Loading (*x* mol %)	Yield (%) [b]
1	10	70
2	**5**	**80**
3	2.5	51

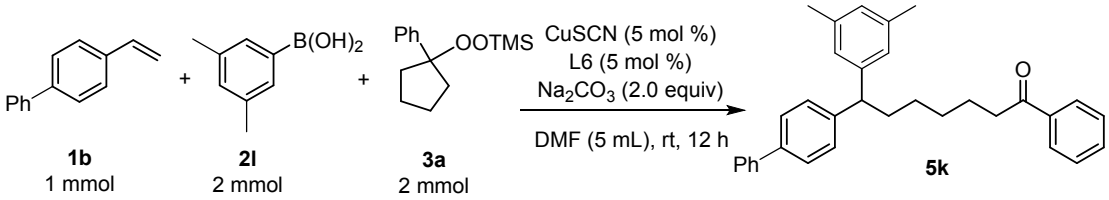
[a]Reaction conditions: **1a** (20.8 mg, 0.2 mmol), **2a** (48.8mg, 0.4 mmol), **3a** (100.2 mg, 0.4 mmol), CuSCN (x mol %), L6 (x mol %), Na_2CO_3 (42.4 mg, 0.4 mmol), DMF (1 mL), the reaction mixture was stirred at room temperature for 12 h. [b]Yields were determined by NMR using CH_2Br_2 as internal standard.

Representative Procedure for the Reaction of Vinylarenes 1, Boronic Acids 2 and Cycloalkylsilyl Peroxides 3



In an N_2-filled glovebox, to an oven dried 10 mL Schlenk-tube containing a magnetic stir bar was added CuSCN (1.2 mg, 5 mol %), 4,4'-dibromo-2,2'-bipyridine (3.1 mg, 5 mol %), boronic acids **2** (0.4 mmol) and Na_2CO_3 (42.4 mg, 0.4 mmol). Subsequently, a solution of vinylarenes **1** (0.2 mmol) and cycloalkylsilyl peroxides **3** (0.4 mmol) in DMF (1 mL) was injected into the tube by syringe. Then, the reaction mixture was moved outside the glovebox and stirred at room temperature for 12 h. After that, the mixture was diluted with EtOAc (10 mL) and saturated saline (10 mL). The organic phase was washed with saturated brine (3×8 mL) and the water phase was extracted with EtOAc (3×6 mL). The combined organic phase was dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel directly to give the desired product **4-6** in isolated yields list in Table 2 and Table 3.

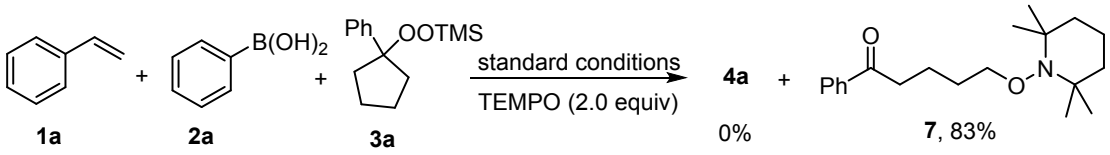
1 mmol Scale Reaction



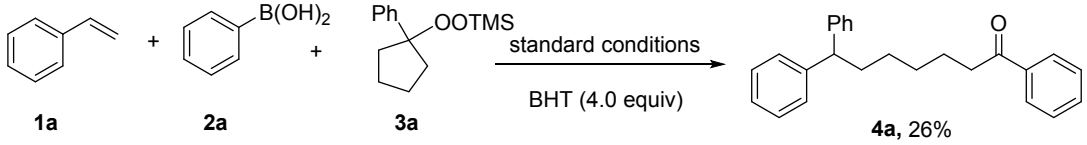
In an N_2-filled glovebox, to an oven dried 50 mL round bottom flask containing a magnetic stir bar was added CuSCN (6.1 mg, 5 mol %), 4,4'-dibromo-2,2'-bipyridine (15.7 mg, 5 mol %), 4-vinylbiphenyl **1b** (180.3 mg, 1 mmol)**,** 3,5-dimethylphenylboronic acid **2l** (300.0 mg, 2 mmol) and Na_2CO_3 (212.0 mg, 2 mmol). Subsequently, a solution of and cyclopentyl silyl peroxide **3a** (501.0 mg, 2 mmol) in DMF (5 mL) was injected into the tube by syringe. Then, the reaction mixture was moved outside the glovebox and stirred at room temperature for 12 h. After that, the mixture was diluted with EtOAc (10 mL) and saturated saline (10 mL). The organic phase was washed with saturated brine (6×8 mL) and the water phase was extracted with EtOAc (3×6 mL). The combined organic phase was dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel (petroleum ether/ethylacetate 80:1) directly to give the desired product **5k** in 68% isolated yield as a white solid.

Investigation of the Reaction Mechanism

Radical Trapping Experiments

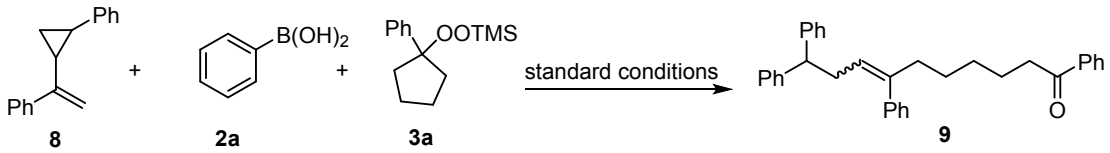


When 2.0 equiv of TEMPO was added to the reaction under the standard conditions, no product **4a** was detected. Meanwhile, the TEMPO-adduct **7** was isolated in 80% yield. This result indicates that radical intermediate was probably involved in this transformation.



When 4.0 equiv of BHT was added to the reaction under the standard conditions, the yield of **4a** was reduced to 26% yield. This result indicates that the reaction might proceed via a radical pathway.

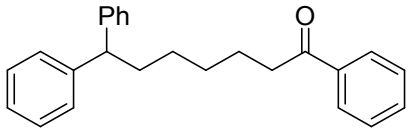
Radical Clock Experiment



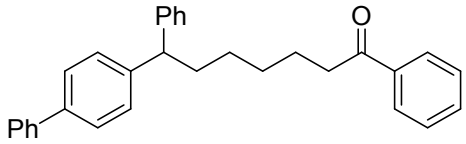
In an N_2-filled glovebox, to an oven dried 10 mL Schlenk-tube containing a magnetic stir bar was added CuSCN (1.2 mg, 5 mol %), 4,4'-dibromo-2,2'-bipyridine (3.1 mg, 5 mol %), phenylboronic acid **2a** (48.8 mg, 0.4 mmol) and Na_2CO_3 (42.4 mg, 0.4 mmol). Subsequently, a solution of **8** (44.1 mg, 0.2 mmol) and cycloalkylsilyl peroxide **3a** (100.2 mg, 0.4 mmol) in DMF (1 mL) was injected into the tube by syringe. Then, the reaction mixture was moved outside the glovebox and stirred at room temperature for 12 h. After that, the mixture was diluted with EtOAc (10 mL) and saturated saline (10 mL). The organic phase was washed with saturated brine (3×8 mL) and the water phase was extracted with EtOAc (3×6 mL). The combined

organic phase was dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel (petroleum ether/ethylacetate 120:1) directly to give the desired product **9** in 58% isolated yield as colorless oil with 6.7 : 1 *Z*/*E* ratio. This result indicates that radical intermediate was probably involved in this transformation.

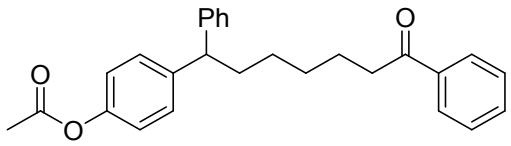
Characterization of Products 4, 5 , 6, 7 and 9



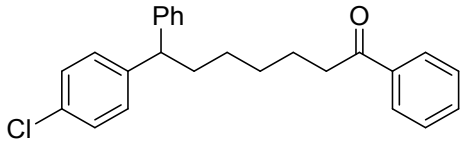
1,7,7-triphenylheptan-1-one (4a): white solid, mp: 93–94 °C (72%, 49.3 mg); R_f 0.32 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.49 (m, 1H), 7.46 – 7.42 (m, 2H), 7.29 – 7.19 (m, 8H), 7.20 – 7.11 (m, 2H), 3.88 (t, *J* = 7.8 Hz, 1H), 2.90 (t, *J* = 7.4 Hz, 2H), 2.10 – 1.99 (m, 2H), 1.74 – 1.66 (m, 2H), 1.45 – 1.37 (m, 2H), 1.35 – 1.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.42, 145.16, 137.04, 132.84, 128.51, 128.36, 128.01, 127.82, 126.01, 51.28, 38.44, 35.48, 29.23, 27.81, 24.12 ppm; IR (neat): υ_{max} 3022, 2929, 2858, 1723, 1682, 1590, 1494, 1449, 1407, 1361, 1259, 1218, 1070, 1021, 969, 800, 740, 695 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{26}NaO$ $[M+Na]^+$ 365.1876, found 365.1868.



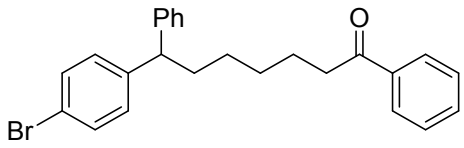
7-([1,1'-biphenyl]-4-yl)-1,7-diphenylheptan-1-one (4b): white solid, mp: 95–96 °C (71%, 60.0 mg); R_f 0.32 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.60 – 7.51 (m, 5H), 7.47 – 7.42 (m, 4H), 7.36 – 7.28 (m, 7H), 7.22 – 7.19 (m, 1H), 3.96 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.14 – 2.10 (m, 2H), 1.77 – 1.72 (m, 2H), 1.50 – 1.43 (m, 2H), 1.41 – 1.34 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.41, 145.05, 144.30, 140.92, 138.89, 137.01, 132.85, 128.65, 128.51, 128.43, 128.19, 128.00, 127.83, 127.10, 126.99, 126.95, 126.09, 50.95, 38.41, 35.47, 29.21, 27.81, 24.07 ppm; IR (neat): υ_{max} 3028, 2930, 2859, 1683, 1594, 1488, 1452, 1406, 1364, 1217, 1075, 1006, 836, 758, 697 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{30}NaO$ $[M+Na]^+$ 441.2189, found 441.2194.



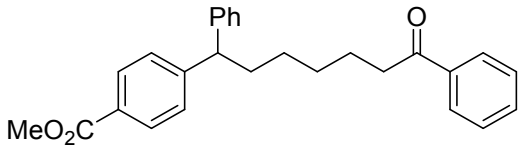
4-(7-oxo-1,7-diphenylheptyl)phenyl acetate (4c)： colorless oil (43%, 34.4 mg); R_f 0.10 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.93 (d, *J* = 6.6 Hz, 2H), 7.56 – 7.52 (m, 1H), 7.46 – 7.43 (m, 2H), 7.28 – 7.25 (m, 2H), 7.23 – 7.19 (m, 4H), 7.18 – 7.15 (m, 1H), 6.99 – 6.97 (m, 2H), 3.88 (t, *J* = 7.8 Hz, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.26 (s, 3H), 2.08 - 1.99 (m, 2H), 1.73 – 1.68 (m, 2H), 1.44 – 1.37 (m, 2H), 1.33 – 1.28 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.42, 169.52, 148.78, 144.76, 142.71, 137.01, 132.85, 128.68, 128.51, 128.41, 128.01, 127.82, 126.13, 121.28, 50.67, 38.41, 35.52, 29.18, 27.76, 24.07, 21.11 ppm; IR (neat): υ_{max} 2919, 1756, 1679, 1501, 1451, 1366, 1259, 1203, 1079, 1015, 908, 801, 749, 695 cm^{-1} ; HRMS (ESI) calcd for $C_{27}H_{28}NaO_3$ $[M+Na]^+$ 423.1931, found 423.1923.



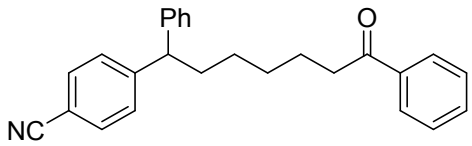
7-(4-chlorophenyl)-1,7-diphenylheptan-1-one (4d): colorless oil (60%, 45.2 mg); R_f 0.45 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 6.6 Hz, 2H), 7.55 – 7.50 (m, 1H), 7.44 – 7.41 (m, 2H), 7.27 – 7.24 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.20 – 7.13 (m, 5H), 3.85 (t, *J* = 7.8 Hz, 1H), 2.90 (t, *J* = 7.5 Hz, 2H), 2.06 – 1.97 (m, 2H), 1.72 – 1.67 (m, 2H), 1.44 – 1.37 (m, 2H), 1.31 – 1.26 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.30, 144.58, 143.64, 136.96, 132.85, 131.66, 129.13, 128.49, 128.44, 127.96, 127.68, 126.20, 50.56, 38.32, 35.30, 29.11, 27.68, 24.00 ppm; IR (neat): υ_{max} 3029, 2931, 2859, 1683, 1592, 1490, 1451, 1407, 1362, 1260, 1217, 1092, 1016, 811, 750, 695 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{25}ClNaO$ $[M+Na]^+$ 399.1486, found 399.1483.



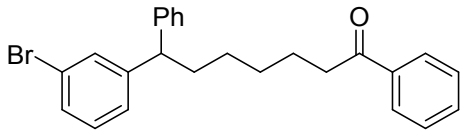
7-(4-bromophenyl)-1,7-diphenylheptan-1-one (4e): colorless oil (53%, 44.5 mg); R_f 0.44 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 6.6 Hz, 2H), 7.56 – 7.52 (m, 1H), 7.45 – 7.43 (m, 2H), 7.38 – 7.36 (m, 2H), 7.28 – 7.23 (m, 2H), 7.20 – 7.14 (m, 3H), 7.10 – 7.08 (m, 2H), 3.84 (t, *J* = 7.8 Hz, 1H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.06 – 1.97 (m, 2H), 1.72 – 1.67 (m, 2H), 1.45 – 1.37 (m, 2H), 1.32 – 1.26 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.35, 144.50, 144.18, 136.98, 132.88, 131.41, 129.57, 128.52, 128.47, 127.99, 127.69, 126.24, 119.78, 50.64, 38.35, 35.26, 29.13, 27.69, 24.02 ppm; IR (neat): υ_{max} 3028, 2931, 2858, 1682, 1591, 1486, 1451, 1404, 1361, 1216, 1072, 1008, 967, 816, 748, 697 cm^{-1} ; HRMS (ESI) calcd for $C_{25}H_{25}BrNaO$ $[M+Na]^+$ 443.0981, found 443.0980.



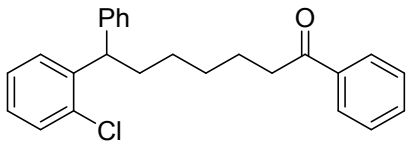
methyl 4-(7-oxo-1,7-diphenylheptyl)benzoate (4f): white solid, mp: 54–55 °C (65%, 52.0 mg); R_f 0.20 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.94 – 7.91 (m, 4H), 7.55 – 7.52 (m, 1H), 7.45 – 7.43 (m, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.30 – 7.25 (m, 2H), 7.22 – 7.20 (d, *J* = 7.2 Hz, 2H), 7.18 – 7.16 (m, 1H), 3.94 (t, *J* = 7.8 Hz, 1H), 3.87 (s, 3H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.09 – 2.04 (m, 2H), 1.73 – 1.68 (m, 2H), 1.44 – 1.39 (m, 2H), 1.33 – 1.25 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.33, 166.99, 150.55, 144.19, 136.97, 132.87, 129.75, 128.50, 127.98, 127.85, 127.78, 126.31, 51.93, 51.24, 38.34, 35.16, 29.12, 27.69, 24.00 ppm; IR (neat): υ_{max} 2937, 1717, 1682, 1602, 1498, 1443, 1363, 1275, 1183, 1103, 1016, 801, 746, 697 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{28}NaO_3$ $[M+Na]^+$ 423.1931, found 423.1923.



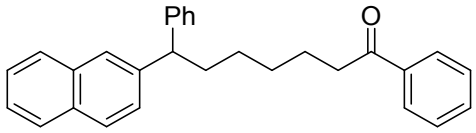
4-(7-oxo-1,7-diphenylheptyl)benzonitrile (4g): white solid, mp: 74–75 ^{o}C (45%, 33.1 mg); R_f 0.13 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.93 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.53 (m, 3H), 7.47 – 7.44 (m, 2H), 7.34 – 7.32 (m, 2H), 7.31 – 7.28 (m, 2H), 7.22 – 7.19 (m, 3H), 3.95 (t, *J* = 7.8 Hz, 1H), 2.93 (t, *J* = 7.5 Hz, 2H), 2.12 – 2.00 (m, 2H), 1.76 – 1.69 (m, 2H), 1.47 – 1.39 (m, 2H), 1.36 – 1.27 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.23, 150.75, 143.41, 136.92, 132.90, 132.21, 128.64, 128.57, 128.51, 127.94, 127.72, 126.59, 118.94, 109.84, 51.27, 38.25, 34.95, 29.03, 27.61, 23.91 ppm; IR (neat): υ_{max} 3059, 2932, 2860, 2226, 1682, 1597, 1497, 1452, 1409, 1363, 1261, 1217, 1081, 1018, 806, 741, 697 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{25}NNaO$ $[M+Na]^+$ 390.1828, found 390.1823.



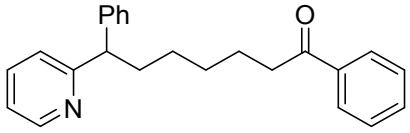
7-(3-bromophenyl)-1,7-diphenylheptan-1-one (4h): colorless oil (52%, 43.7 mg); R_f 0.38 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.94 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.53 (m, 1H), 7.47 – 7.45 (m, 2H), 7.39 (s, 1H), 7.32 – 7.27 (m, 3H), 7.24 – 7.12 (m, 5H), 3.86 (t, *J* = 7.8 Hz, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 2.09 – 2.00 (m, 2H), 1.75 – 1.70 (m, 2H), 1.47 – 1.39 (m, 2H), 1.34 – 1.29 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.36, 147.60, 144.22, 136.99, 132.87, 130.82, 129.95, 129.15, 128.52, 128.51, 127.99, 127.75, 126.48, 126.32, 122.49, 50.98, 38.37, 35.25, 29.13, 27.69, 24.02 ppm; IR (neat): υ_{max} 3059, 2930, 2858, 1682, 1583, 1457, 1361, 1214, 1075, 1001, 878, 753, 696 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{25}BrNaO$ $[M+Na]^+$ 443.0981, found 443.0980.



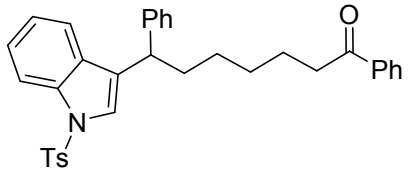
7-(2-chlorophenyl)-1,7-diphenylheptan-1-one (4i): colorless oil (58%, 43.6 mg); R_f 0.44 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 6.6 Hz, 2H), 7.56 – 7.51 (m, 1H), 7.45 – 7.43 (m, 2H), 7.32 – 7.28 (m, 2H), 7.27 – 7.23 (m, 4H), 7.22 – 7.14 (m, 2H), 7.09 (td, *J* = 7.5, 1.8 Hz), 4.46 (t, *J* = 7.8 Hz, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.08 – 1.97 (m, 2H), 1.73 – 1.68 (m, 2H), 1.46 – 1.38 (m, 2H), 1.37 – 1.30 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.43, 143.66, 142.44, 137.01, 134.20, 132.86, 129.60, 128.52, 128.44, 128.31, 128.14, 128.01, 127.19, 126.88, 126.17, 46.54, 38.43, 35.19, 29.19, 27.55, 24.09 ppm; IR (neat): υ_{max} 3061, 2924, 2857, 1682, 1590, 1449, 1363, 1259, 1211, 1083, 1026, 801, 744, 691 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{25}ClNaO$ $[M+Na]^+$ 399.1486, found 399.1476.



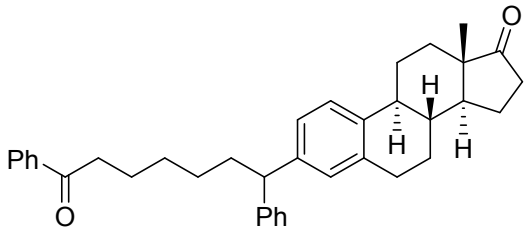
7-(naphthalen-2-yl)-1,7-diphenylheptan-1-one (4j): white solid, mp: 67–68 °C (70%, 54.7 mg); R_f 0.36 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.94 (d, *J* = 6.6 Hz, 2H), 7.83 – 7.79 (m, 2H), 7.77 – 7.73 (m, 2H), 7.57 – 7.53 (m, 1H), 7.48 – 7.42 (m, 4H), 7.36 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.33 – 7.27 (m, 4H), 7.21 – 7.18 (m, 1H), 4.08 (t, *J* = 7.8 Hz, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 2.25 – 2.13 (m, 2H), 1.77 – 1.72 (m, 2H), 1.52 – 1.43 (m, 2H), 1.43 – 1.35 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.42, 145.01, 142.57, 136.99, 133.49, 132.83, 132.08, 128.50, 128.38, 127.99, 127.93, 127.67, 127.52, 126.73, 126.08, 125.87, 125.82, 125.29, 51.26, 38.40, 35.20, 29.21, 27.80, 24.07 ppm; IR (neat): υ_{max} 3051, 2928, 2857, 1680, 1591, 1500, 1449, 1363, 1212, 1075, 963, 853, 812, 744, 694 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{28}NaO$ $[M+Na]^+$ 415.2032, found 415.2026.



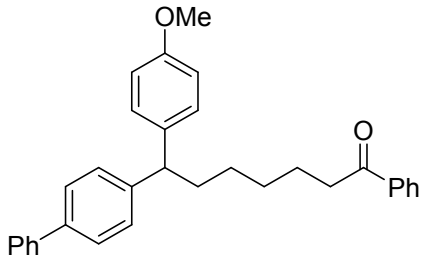
1,7-diphenyl-7-(pyridin-2-yl)heptan-1-one (4k): colorless oil (57%, 39.3 mg); R_f 0.21 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.56 – 8.54 (m, 1H), 7.92 (d, *J* = 7.8 Hz, 2H), 7.55 – 7.52(m, 2H), 7.45 – 7.42 (m, 2H), 7.34 – 7.32 (m, 2H), 7.30 – 7.24 (m, 2H), 7.20 – 7.14 (m, 1H), 7.14 (d, *J* = 7.9 Hz, 1H), 7.09 – 7.04 (m, 1H), 4.04 (t, *J* = 7.8 Hz, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.29 – 2.23 (m, 1H), 2.11 – 2.05 (m, 1H), 1.72 – 1.67 (m, 2H), 1.45 – 1.40 (m, 2H), 1.35 – 1.28(m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.47, 163.96, 149.18, 143.82, 137.01, 136.33, 132.82, 128.49, 128.41, 127.99, 127.97, 126.30, 122.61, 121.17, 53.69, 38.42, 34.81, 29.18, 27.70, 24.11 ppm; IR (neat): υ_{max} 3059, 2930, 2857, 1682, 1585, 1442, 1363, 1260, 1216, 1019, 799, 749, 695 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{26}NO$ $[M+H]^+$ 344.2009, found 344.2009.



1,7-diphenyl-7-(1-tosyl-1H-indol-3-yl)heptan-1-one (4l): colorless oil (63%, 67.4 mg); R_f 0.32 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 – 7.90 (m, 3H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.58 – 7.52 (m, 1H), 7.47 – 7.43 (m, 3H), 7.25 – 7.15 (m, 9H), 7.11 – 7.05 (m, 1H), 4.00 (t, *J* = 7.4 Hz, 1H), 2.92 (t, *J* = 7.4 Hz, 2H), 2.32 (s, 3H), 2.17 – 2.09 (m, 1H), 2.02 – 1.92 (m, 1H), 1.73 – 1.66 (m, 2H), 1.47 – 1.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.35, 144.73, 143.36, 136.94, 135.54, 135.08, 132.90, 130.60, 129.75, 128.53, 128.40, 127.99, 127.76, 126.84, 126.67, 126.38, 124.55, 123.00, 122.61, 120.09, 113.71, 42.51, 38.38, 35.31, 29.16, 27.59, 24.10, 21.51 ppm; IR (neat): υ_{max} 3059, 2927, 2859, 1682, 1595, 1445, 1367, 1269, 1174, 1125, 1020, 971, 911, 805, 744, 686, 576, 535 cm^{-1}; HRMS (ESI) calcd for $C_{34}H_{33}NNaO_3S$ $[M+Na]^+$ 558.2073, found 558.2080.

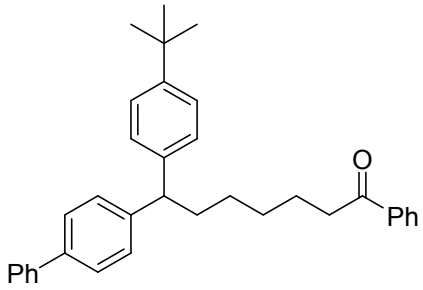


(8S,9S,13S,14S)-3-(7-oxo-1,7-diphenylheptyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4m): colorless oil (60%, 62.3 mg, dr = 1:1); R_f 0.10 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 – 7.90 (m, 2H), 7.57 – 7.51 (m, 1H), 7.45 – 7.41 (m, 2H), 7.29 – 7.21 (m, 4H), 7.20 – 7.12 (m, 2H), 7.05 – 7.00 (m, 1H), 6.95 (s, 1H), 3.81 (t, *J* = 7.8 Hz, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.87 – 2.84 (m, 2H), 2.51 (d, *J* = 8.6 Hz, 0.5H), 2.46 (d, *J* = 8.6 Hz, 0.5H), 2.40 – 2.35 (m, 1H), 2.27 – 2.21 (m, 1H), 2.13 (d, *J* = 8.8 Hz, 0.5H), 2.09 (d, *J* = 8.8 Hz, 0.5H), 2.08 – 1.91 (m, 5H), 1.75 – 1.66 (m, 2H), 1.63 – 1.54 (m, 2H), 1.52 – 1.45 (m, 3H), 1.44 – 1.36 (m, 3H), 1.34 – 1.25 (m, 2H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 220.95, 200.38, 145.20, 142.62, 137.29, 136.94, 136.27, 136.25, 132.83, 128.47, 128.32, 128.28, 127.96, 127.75, 125.92, 125.27, 125.25, 125.04, 124.99, 50.88, 50.86, 50.41, 47.92, 44.20, 38.41, 38.03, 35.80, 35.49, 31.51, 29.41, 29.20, 27.83, 26.49, 25.55, 24.04, 21.50, 13.78 ppm; IR (neat): υ_{max} 3024, 2929, 2861, 1736, 1684, 1594, 1494, 1452, 1406, 1367, 1259, 1216, 1085, 1015, 912, 806, 733, 698 cm^{-1}; HRMS (ESI) calcd for $C_{37}H_{42}NaO_2$ $[M+Na]^+$ 541.3077, found 541.3078.

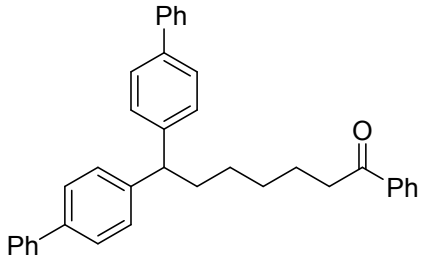


7-([1,1'-biphenyl] -4-yl) -7- (4-methoxyphenyl) -1-phenylheptan-1-one (5a): colorless oil (63%, 56.4 mg); R_f 0.30 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.53 (m, 3H), 7.52 – 7.50 (m, 2H), 7.47 – 7.41 (m, 4H), 7.36 – 7.28 (m, 3H), 7.21 – 7.19 (m, 2H), 6.87 – 6.84 (m, 2H), 3.90 (t, *J* = 7.8 Hz, 1H), 3.78 (s, 3H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.10 – 2.06 (m, 2H),

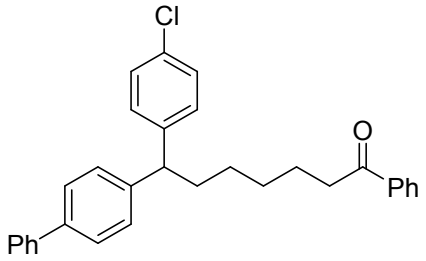
1.77 – 1.72 (m, 2H), 1.48 – 1.43 (m, 2H), 1.38 – 1.33 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.44, 157.86, 144.73, 140.96, 138.79, 137.19, 137.02, 132.85, 128.71, 128.65, 128.52, 128.09, 128.01, 127.08, 126.97, 126.95, 113.80, 55.16, 50.06, 38.42, 35.64, 29.21, 27.81, 24.09 ppm; IR (neat): υ_{max} 2918, 1677, 1597, 1500, 1450, 1248, 1177, 1082, 1025, 805, 748, 689 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO_2$ $[M+Na]^+$ 471.2295, found 471.2286.



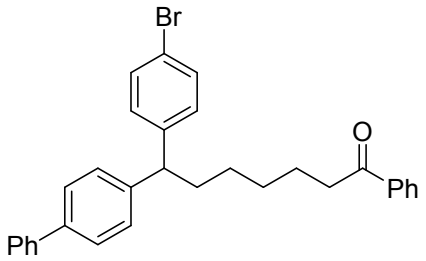
7-([1,1'-biphenyl]-4-yl)-7-(4-(tert-butyl)phenyl)-1-phenylheptan-1-one (5b): colorless oil (71%, 67.1 mg); R_f 0.41 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.61 – 7.50 (m, 5H), 7.47 – 7.42 (m, 4H), 7.35 – 7.32(m, 5H), 7.22 (d, *J* = 8.4 Hz, 2H), 3.93 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.5 Hz, 2H), 2.13 – 2.09 (m, 2H), 1.78 – 1.73 (m, 2H), 1.50 – 1.43 (m, 2H), 1.40 – 1.35 (m, 2H), 1.32 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.43, 148.73, 144.49, 142.01, 140.98, 138.81, 137.03, 132.84, 128.64, 128.51, 128.23, 128.00, 127.32, 127.07, 126.95, 125.29, 50.54, 38.43, 35.60, 34.29, 31.35, 29.24, 27.85, 24.09 ppm; IR (neat): υ_{max} 3029, 2951, 2862, 1684, 1481, 1453, 1405, 1362, 1263, 1214, 1105, 1013, 825, 756, 694 cm^{-1}; HRMS (ESI) calcd for $C_{35}H_{38}NaO$ $[M+Na]^+$ 497.2815, found 497.2822



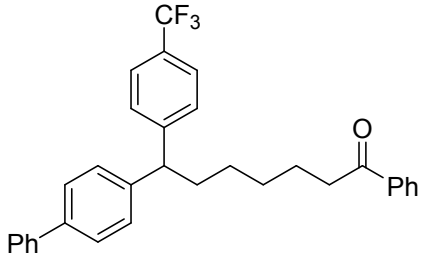
7,7-di([1,1'-biphenyl]-4-yl)-1-phenylheptan-1-one (5c): colorless oil (51%, 50.6 mg); R_f 0.31 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.60 – 7.58 (m, 4H), 7.57 – 7.54 (m, 5H), 7.48 – 7.41 (m, 6H), 7.39 – 7.31 (m, 6H), 4.00 (t, *J* = 7.8 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.17 – 2.14 (m, 2H), 1.79 – 1.74 (m, 2H), 1.51 – 1.46 (m, 2H), 1.44 – 1.39 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.41, 144.19, 140.91, 138.98, 137.02, 132.86, 128.67, 128.52, 128.21, 128.00, 127.17, 127.02, 126.96, 50.64, 38.41, 35.48, 29.22, 27.83, 24.07 ppm; IR (neat): υ_{max} 3030, 2929, 2858, 1683, 1595, 1519, 1484, 1452, 1406, 1364, 1262, 1216, 1086, 1018, 803, 753, 695 cm^{-1}; HRMS (ESI) calcd for $C_{37}H_{34}NaO$ $[M+Na]^+$ 517.2502, found 517.2506.



7-([1,1'-biphenyl]-4-yl)-7-(4-chlorophenyl)-1-phenylheptan-1-one (5d): white solid, mp: 59–60 °C (68%, 61.7 mg); R_f 0.33 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 7.2 Hz, 2H), 7.57 – 7.46 (m, 5H), 7.44 – 7.38 (m, 4H), 7.33 – 7.27 (m, 1H), 7.26 – 7.21 (m, 4H), 7.20 – 7.15 (m, 2H), 3.89 (t, *J* = 7.8 Hz, 1H), 2.91 (t, *J* = 7.4 Hz, 2H), 2.11 – 1.97 (m, 2H), 1.74 – 1.67 (m, 2H), 1.46 – 1.37 (m, 2H), 1.36 – 1.27 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.32, 143.70, 143.53, 140.76, 139.09, 136.95, 132.87, 131.76, 129.16, 128.67, 128.52, 128.51, 128.08, 127.97, 127.17, 127.07, 126.93, 50.25, 38.34, 35.33, 29.13, 27.72, 24.00 ppm; IR (neat): υ_{max} 3030, 2929, 2858, 1682, 1592, 1486, 1451, 1405, 1363, 1260, 1215, 1087, 1011, 819, 754, 694 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{29}ClNaO$ $[M+Na]^+$ 475.1799, found 475.1800.

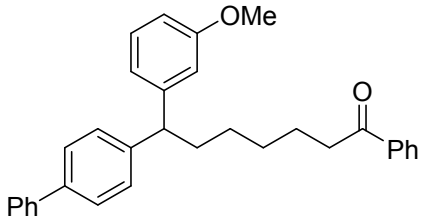


7-([1,1'-biphenyl]-4-yl)-7-(4-bromophenyl)-1-phenylheptan-1-one (5e): colorless oil (41%, 40.7 mg); R_f 0.32 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 6.8 Hz, 2H), 7.60 – 7.49 (m, 5H), 7.49 – 7.39 (m, 6H), 7.36 – 7.31 (m, 1H), 7.29 – 7.27 (m, 2H), 7.16 – 7.14 (m, 2H), 3.90 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.14 – 2.00 (m, 2H), 1.77 – 1.70 (m, 2H), 1.50 – 1.40 (m, 2H), 1.39 – 1.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.34, 144.06, 143.60, 140.76, 139.12, 136.94, 132.89, 131.48, 129.58, 128.68, 128.52, 128.08, 127.98, 127.19, 127.08, 126.94, 119.86, 50.32, 38.35, 35.27, 29.13, 27.72, 24.00 ppm; IR (neat): υ_{max} 3026, 2927, 2857, 1682, 1519, 1483, 1451, 1405, 1364, 1261, 1215, 1078, 1012, 805, 754, 694 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{29}BrNaO$ $[M+Na]^+$ 519.1294, found 519.1295.

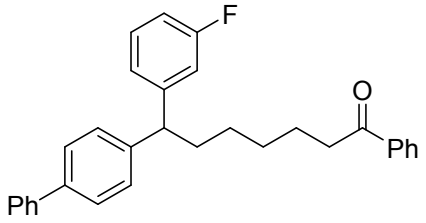


7-([1,1'-biphenyl] -4-yl) -7- (4-trifluoromethylphenyl) -1-phenylheptan-1-one (5f)：colorless oil (34%, 33.1 mg); R_f 0.61 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 6.8 Hz, 2H), 7.58 – 7.52 (m, 7H), 7.49 – 7.37 (m, 6H), 7.36 – 7.33 (m, 1H), 7.32 – 7.29 (m, 2H), 4.01 (t, *J* = 7.8 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.15 – 2.07 (m, 2H), 1.78 – 1.70 (m, 2H), 1.49 – 1.42 (m, 2H), 1.40 – 1.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.32, 149.15, 143.14, 140.70, 139.32, 136.95, 132.92, 128.70, 128.54, 128.15, 128.13, 127.98, 127.28, 127.15, 126.96, 125.40 (q, *J* = 3.7 Hz), 124.23 (q, *J* = 270.1 Hz), 50.77, 38.34, 35.23, 29.14, 27.73,

23.98 ppm; IR (neat): υ_{max} 3031, 2929, 2859, 1683, 1609, 1481, 1453, 1411, 1366, 1324, 1261, 1217, 1164, 1119, 1070, 1015, 830, 755, 694 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{29}F_3NaO$ $[M+Na]^+$ 509.2063, found 509.2070.

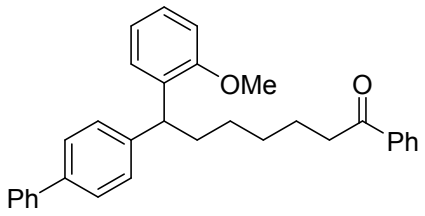


7-([1,1'-biphenyl] -4-yl) -7- (3-methoxyphenyl) -1-phenylheptan-1-one (5g): colorless oil (64%, 57.3 mg); R_f 0.23 (EtOAc/petroleum ether = 1:20); ^1H NMR (600 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 6.6 Hz, 2H), 7.59 – 7.53 (m, 3H), 7.52 – 7.51(m, 2H), 7.47 – 7.41 (m, 4H), 7.35 – 7.30 (m, 3H), 7.23 (t, *J* = 7.8 Hz, 1H), 6.90 – 6.89 (m, 1H), 6.85 – 6.84 (m, 1H), 6.75 – 6.73 (m, 1H), 3.92 (t, *J* = 7.8 Hz, 1H), 3.79 (s, 3H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.12 – 2.08 (m, 2H), 1.77 – 1.72 (m, 2H), 1.48 – 1.43 (m, 2H), 1.40 – 1.35 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.42, 159.61, 146.73, 144.13, 140.93, 138.93, 137.01, 132.84, 129.36, 128.65, 128.51, 128.14, 128.00, 127.11, 126.99, 126.95, 120.29, 114.03, 110.90, 55.09, 50.97, 38.42, 35.41, 29.21, 27.81, 24.08 ppm; IR (neat): υ_{max} 3024, 2921, 2850, 1677, 1589, 1479, 1448, 1362, 1255, 1142, 1082, 1036, 798, 741, 692 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO_2$ $[M+Na]^+$ 471.2295, found 471.2301.

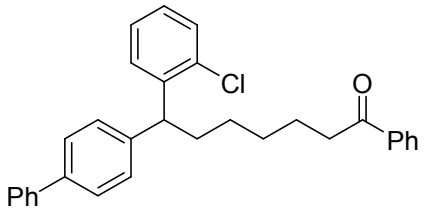


7-([1,1'-biphenyl]-4-yl)-7-(3-fluorophenyl)-1-phenylheptan-1-one (5h): white solid, mp: 72–73 °C (64%, 56.2 mg); R_f 0.39 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.94 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.51 (m, 5H), 7.49 – 7.39 (m, 4H), 7.33 – 7.23 (m, 4H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.98 – 6.95 (m, 1H), 6.88 (td, *J* = 8.4, 2.4 Hz, 1H), 3.94 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.09 – 2.04 (m, 2H),

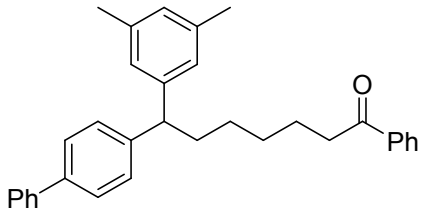
1.77 – 1.70 (m, 2H), 1.50 – 1.40 (m, 2H), 1.39 – 1.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.36, 162.94 (d, *J* = 243.9 Hz), 147.76 (d, *J* = 6.6 Hz), 143.51, 140.81, 139.19, 137.01, 132.88, 129.81 (d, *J* = 8.2 Hz), 128.69, 128.53, 128.14, 128.00, 127.22, 127.09, 126.97, 123.55 (d, *J* = 1.4 Hz), 114.64 (d, *J* = 10.6 Hz), 112.99 (d, *J* = 21.0 Hz), 50.70, 38.39, 35.34, 29.18, 27.75, 24.06 ppm; IR (neat): υ_{max} 3031, 2931, 2859, 1682, 1590, 1520, 1483, 1448, 1407, 1363, 1253, 1137, 1078, 1009, 967, 839, 750, 694 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{29}FNaO$ $[M+Na]^+$ 459.2095, found 459.2010.



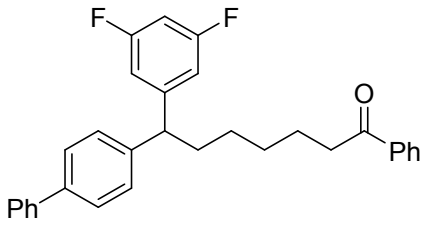
7-([1,1'-biphenyl] -4-yl) -7- (2-methoxyphenyl) -1-phenylheptan-1-one (5i): colorless oil (61%, 54.7 mg); R_f 0.27 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.61 – 7.53 (m, 3H), 7.52 – 7.50 (m, 2H), 7.47 – 7.41 (m, 4H), 7.38 – 7.28 (m, 4H), 7.21 – 7.18 (m, 1H), 6.97 – 6.95 (m, 1H), 6.87 – 6.86 (m, 1H), 4.47 (t, *J* = 7.8 Hz, 1H), 3.81 (s, 3H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.10 – 2.06 (m, 2H), 1.78 – 1.73 (m, 2H), 1.49 – 1.44 (m, 2H), 1.41 – 1.36 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.49, 156.98, 144.31, 141.08, 138.49, 137.02, 133.51, 132.81, 128.61, 128.49, 128.00, 127.54, 126.99, 126.92, 126.87, 126.80, 120.52, 110.67, 55.42, 42.66, 38.47, 34.76, 29.25, 27.74, 24.12 ppm; IR (neat): υ_{max} 2932, 1683, 1592, 1485, 1452, 1364, 1241, 1022, 802, 753, 695 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO_2$ $[M+Na]^+$ 471.2295, found 471.2298.



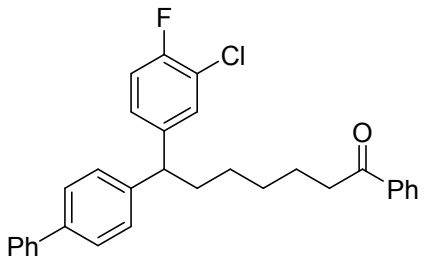
7-([1,1'-biphenyl]-4-yl)-7-(2-chlorophenyl)-1-phenylheptan-1-one (5j): white solid, mp: 94–95 ^{o}C (62%, 55.9 mg); R_f 0.38 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.61 – 7.50 (m, 5H), 7.49 – 7.40 (m, 4H), 7.39 – 7.30 (m, 5H), 7.25 (td, *J* = 7.6, 1.6 Hz, 1H), 7.14 (td, *J* = 7.6, 1.6 Hz, 1H), 4.55 (t, *J* = 7.8 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.14 – 2.05 (m, 2H), 1.79 – 1.72 (m, 2H), 1.54 – 1.35 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.37, 142.76, 142.31, 140.83, 139.00, 136.97, 134.18, 132.85, 129.63, 128.65, 128.50, 128.41, 127.99, 127.26, 127.02, 126.94, 46.20, 38.40, 35.20, 29.18, 27.56, 24.05 ppm; IR (neat): υ_{max} 3060, 2929, 2858, 1683, 1592, 1477, 1454, 1407, 1364, 1260, 1214, 1083, 1033, 802, 752, 694 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{29}ClNaO$ $[M+Na]^+$ 475.1799, found 475.1802.



7-([1,1'-biphenyl]-4-yl)-7-(3,5-dimethylphenyl)-1-phenylheptan-1-one (5k): white solid, mp: 75–76 ^{o}C (70%, 61.9 mg); R_f 0.45 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.97 (d, *J* = 7.2 Hz, 2H), 7.62 – 7.52 (m, 5H), 7.50 – 7.42 (m, 4H), 7.38 – 7.31 (m, 3H), 6.93 (s, 2H), 6.86 (s, 1H), 3.89 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.32 (s, 6H), 2.14 – 2.08 (m, 2H), 1.80 – 1.73 (m, 2H), 1.52 – 1.43 (m, 2H), 1.43 – 1.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.42, 144.96, 144.49, 140.96, 138.74, 137.76, 136.98, 132.83, 128.63, 128.49, 128.14, 127.98, 127.81, 127.06, 126.92, 125.59, 50.91, 38.42, 35.47, 29.23, 27.85, 24.06, 21.38 ppm; IR (neat): υ_{max} 3023, 2927, 2859, 1684, 1598, 1480, 1453, 1407, 1365, 1261, 1216, 1083, 1016, 838, 740, 695 cm^{-1}; HRMS (ESI) calcd for $C_{33}H_{34}NaO$ $[M+Na]^+$ 469.2502, found 469.2512.

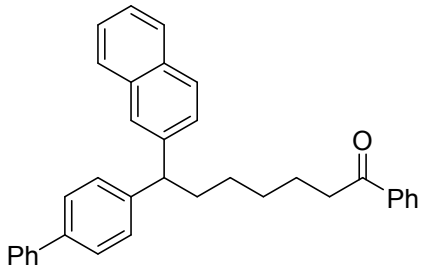


7-([1,1'-biphenyl]-4-yl)-7-(3,5-difluorophenyl)-1-phenylheptan-1-one (5l): colorless oil (51%, 46.6 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 7.2 Hz, 2H), 7.60 – 7.50 (m, 5H), 7.47 – 7.41 (m, 4H), 7.37 – 7.31 (m, 1H), 7.29 – 7.27 (m, 2H), 6.84 – 6.76 (m, 2H), 6.66 – 6.60 (m, 1H), 3.91 (t, *J* = 7.8 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.12 – 1.99 (m, 2H), 1.77 – 1.70 (m, 2H), 1.48 – 1.41 (m, 2H), 1.38 – 1.31(m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.31, 161.95 (d, *J* = 246.5 Hz), 161.92 (d, *J* = 246.5 Hz), 149.21 (t, *J* = 8.2 Hz), 142.68, 140.67, 139.48, 136.97, 132.91, 128.71, 128.54, 128.09, 127.99, 127.33, 127.18, 126.98, 110.62 (dd, *J* = 24.7, 11.7 Hz, 2C), 101.58 (t, *J* = 25.2 Hz), 50.67 (t, *J* = 1.75 Hz), 38.35, 35.13, 29.12, 27.67, 24.00 ppm; IR (neat): υ_{max} 2927, 2857, 1793, 1681, 1591, 1519, 1450, 1308, 1262, 1210, 1114, 1081, 981, 839, 803, 749, 692 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{28}F_2NaO$ $[M+Na]^+$ 477.2000, found 477.1999.

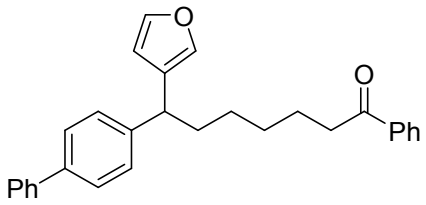


7-([1,1'-biphenyl]-4-yl)-7-(3-chloro-4-fluorophenyl)-1-phenylheptan-1-one (5m): colorless oil (55%, 52.1 mg); R_f 0.35 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.93 (d, *J* =7.2 Hz, 2H), 7.58 – 7.49 (m, 5H), 7.45 – 7.40 (m, 4H), 7.34 – 7.29 (m, 1H), 7.28 (dd, *J* = 7.2, 2.4 Hz, 1H), 7.26 (s, 1H), 7.24 (d, *J* = 3.0 Hz, 1H), 7.12 – 7.09 (m, 1H), 7.03 (t, *J* = 8.7 Hz, 1H), 3.88 (t, *J* = 7.8 Hz, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.10 – 1.97 (m, 2H), 1.75 – 1.70 (m, 2H), 1.45 – 1.40 (m, 2H), 1.37 – 1.29 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 200.31, 156.53 (d, *J* = 246.0 Hz), 143.21, 142.21 (d, *J* = 4.4 Hz), 140.70, 139.33, 136.99, 132.90, 129.73, 128.71,

128.53, 128.06, 127.98, 127.40 (d, J = 7.5 Hz), 127.29, 127.15, 126.96, 120.69 (d, J = 17.6 Hz), 116.41 (d, J = 20.9 Hz), 50.00, 38.33, 35.38, 29.10, 27.69, 23.99 ppm; IR (neat): υ_{max} 3032, 2931, 2859, 1741, 1682, 1592, 1493, 1452, 1405, 1364, 1253, 1065, 1010, 884, 828, 749, 694 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{28}ClFNaO$ $[M+Na]^+$ 493.1705, found 493.1702.

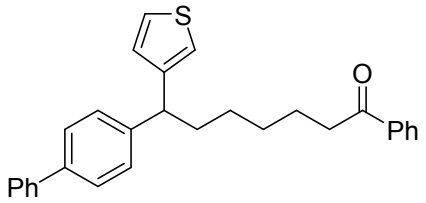


7-([1,1'-biphenyl]-4-yl)-7-(naphthalen-2-yl)-1-phenylheptan-1-one (5n): white solid, mp: 106–107 °C (50%, 46.8 mg); R_f 0.37 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, J = 6.8 Hz, 2H), 7.87 – 7.75 (m, 4H), 7.61 – 7.51 (m, 5H), 7.51 – 7.31 (m, 10H), 4.13 (t, J = 7.8 Hz, 1H), 2.95 (t, J = 7.4 Hz, 2H), 2.29 – 2.16 (m, 2H), 1.80 – 1.72 (m, 2H), 1.54 – 1.38 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.39, 144.14, 142.44, 140.87, 138.94, 136.97, 133.49, 132.83, 132.11, 128.65, 128.49, 128.30, 128.08, 127.97, 127.67, 127.54, 127.11, 127.00, 126.94, 126.69, 125.91, 125.86, 125.33, 50.94, 38.39, 35.21, 29.22, 27.82, 24.05 ppm; IR (neat): υ_{max} 3053, 2929, 2858, 1682, 1594, 1491, 1452, 1406, 1365, 1262, 1216, 1085, 1017, 809, 750, 694 cm^{-1}; HRMS (ESI) calcd for $C_{35}H_{32}NaO$ $[M+Na]^+$ 491.2345, found 491.2351.

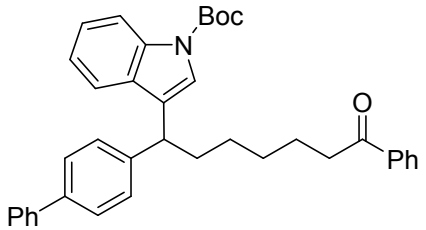


7-([1,1'-biphenyl]-4-yl)-7-(furan-3-yl)-1-phenylheptan-1-one (5o): colorless oil (65%, 53.5 mg); R_f 0.33 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.93 (d, J = 8.4 Hz, 2H), 7.60 – 7.49 (m, 5H), 7.45 – 7.39 (m, 4H), 7.35 – 7.23 (m, 5H), 6.26 (s, 1H), 3.78 (t, J = 7.6 Hz, 1H), 2.92 (t, J = 7.2 Hz, 2H), 2.04 – 1.87 (m,

2H), 1.75 – 1.68 (m, 2H), 1.50 – 1.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.38, 143.72, 142.89, 140.88, 139.07, 138.80, 136.97, 132.85, 129.11, 128.66, 128.51, 128.11, 127.98, 127.08, 127.02, 126.95, 110.32, 41.91, 38.39, 35.73, 29.12, 27.56, 24.06 ppm; IR (neat): υ_{max} 3027, 2927, 2857, 1680, 1586, 1490, 1449, 1407, 1362, 1257, 1210,1148, 1074, 1016, 865, 795, 741, 691 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{28}NaO_2$ $[M+Na]^+$ 431.1982, found 431.1989.

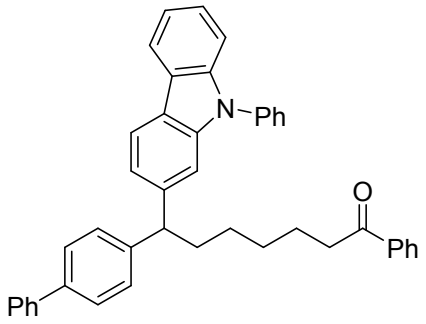


7-([1,1'-biphenyl]-4-yl)-1-phenyl-7-(thiophen-3-yl)heptan-1-one (5p): colorless oil (42%, 35.7 mg); R_f 0.29 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.93 (d, *J* = 6.8 Hz, 2H), 7.58 – 7.48 (m, 5H), 7.45 – 7.39 (m, 4H), 7.34 – 7.22 (m, 4H), 7.01 – 7.00 (m, 1H), 6.94 (dd, *J* = 4.8, 1.2 Hz, 1H), 3.99 (t, *J* = 7.8 Hz, 1H), 2.92 (t, *J* = 7.4 Hz, 2H), 2.15 – 1.96 (m, 2H), 1.75 – 1.68 (m, 2H), 1.46 – 1.29 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.40, 146.03, 143.87, 140.88, 138.99, 136.97, 132.86, 128.66, 128.51, 128.17, 127.99, 127.67, 127.11, 127.01, 126.94, 125.40, 120.00, 46.52, 38.40, 35.95, 29.15, 27.69, 24.06 ppm; IR (neat): υ_{max} 2920, 2853, 1677, 1518, 1481, 1449, 1404, 1260, 1208, 1137, 1078, 1012, 751, 689 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{28}NaOS$ $[M+Na]^+$ 447.1753, found 447.1753.

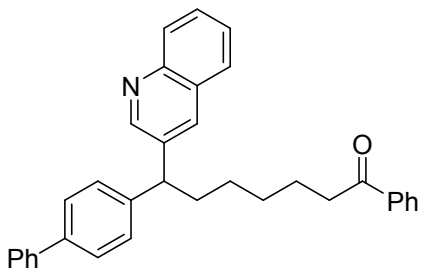


tert-butyl-3-(1-([1,1'-biphenyl]-4-yl)-7-oxo-7-phenylheptyl)-1H-indole-1-carboxylate (5q): colorless oil (53%, 58.6 mg); R_f 0.20 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.09 (s, 1H), 7.94 (d, *J* = 6.8 Hz, 2H), 7.59 – 7.48 (m, 6H), 7.46 – 7.26 (m, 9H), 7.17 – 7.11 (m, 1H), 4.12 (t, *J* = 7.6 Hz, 1H), 2.94

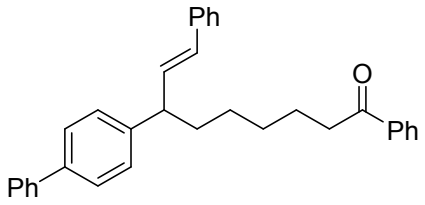
(t, *J* = 7.2 Hz, 2H), 2.26 – 2.18 (m, 1H), 2.09 – 2.03 (m, 1H), 1.80 – 1.72 (m, 2H), 1.70 (s, 9H), 1.54 – 1.39 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.41, 143.15, 140.88, 139.06, 136.98, 132.86, 128.65, 128.51, 128.25, 128.00, 127.10, 126.99, 126.94, 124.47, 124.21, 122.28, 119.68, 115.17, 42.19, 38.42, 35.38, 29.22, 28.22, 27.69, 24.11 ppm; IR (neat): υ_{max} 2929, 1727, 1685, 1452, 1371, 1308, 1258, 1218, 1159, 1087, 1022, 802, 753, 695 cm^{-1}; HRMS (ESI) calcd for $C_{38}H_{39}NNaO_3$ $[M+Na]^+$ 580.2822, found 580.2831.



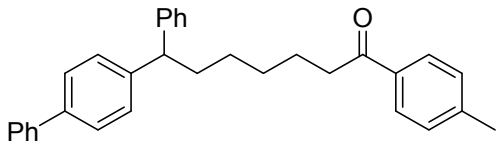
7-([1,1'-biphenyl]-4-yl)-1-phenyl-7-(9-phenyl-9H-carbazol-2-yl)heptan-1-one (5r): yellow oil (62%, 72.3 mg): R_f 0.53 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.20 – 8.18 (m, 1H), 8.10 (s, 1H), 7.97 – 7.95 (m, 2H), 7.63 – 7.53 (m, 9H), 7.49 – 7.39 (m, 9H), 7.38 – 7.29 (m, 4H), 4.19 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.30 – 2.24 (m, 2H), 1.83 – 1.75 (m, 2H), 1.57 – 1.45 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.43, 145.13, 141.01, 140.96, 139.44, 138.71, 137.70, 136.95, 136.75, 132.80, 129.75, 128.62, 128.47, 128.17, 127.97, 127.22, 127.09, 126.94, 126.90, 126.16, 125.78, 123.37, 123.25, 120.24, 119.73, 119.06, 109.75, 109.69, 50.87, 38.41, 35.89, 29.25, 27.93, 24.08 ppm; IR (neat): υ_{max} 3054, 2928, 2858, 1683, 1595, 1492, 1453, 1407, 1360, 1328, 1229, 1078, 1015, 911, 806, 741, 696 cm^{-1}; HRMS (ESI) calcd for $C_{43}H_{37}NNaO$ $[M+Na]^+$ 606.2767, found 606.2774.



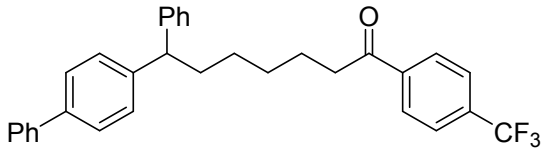
7-([1,1'-biphenyl]-4-yl)-1-phenyl-7-(quinolin-3-yl)heptan-1-one (5s): colorless oil (37%, 34.7 mg); R_f 0.60 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.86 (d, *J* = 2.4 Hz, 1H), 8.08 (d, *J* = 8.8 Hz, 1H), 8.01 (d, *J* = 2.4 Hz, 1H), 7.95 – 7.92 (m, 2H), 7.81 – 7.78 (m, 1H), 7.69 – 7.65 (m, 1H), 7.60 – 7.50 (m, 6H), 7.46 – 7.40 (m, , 4H), 7.38 – 7.30 (m, 3H), 4.16 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.26 – 2.20 (m, 2H), 1.78 – 1.71 (m, 2H), 1.52 – 1.37 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.29, 151.82, 146.86, 142.78, 140.66, 139.40, 137.68, 136.91, 133.34, 132.88, 129.07, 128.82, 128.69, 128.51, 128.28, 128.04, 127.96, 127.59, 127.35, 127.14, 126.96, 126.62, 48.55, 38.33, 35.16, 29.14, 27.76, 23.98 ppm; IR (neat): υ_{max} 3032, 2928, 2858, 1682, 1592, 1489, 1453, 1409, 1369, 1463, 1217, 1085, 1017, 911, 797, 751, 695 cm^{-1}; HRMS (ESI) calcd for $C_{34}H_{32}NO$ $[M+H]^+$ 470.2478, found 470.2485.



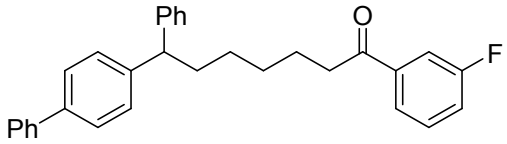
(E)-7-([1,1'-biphenyl]-4-yl)-1,9-diphenylnon-8-en-1-one (5t): colorless oil (46%, 40.5 mg); R_f 0.40 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.63 – 7.53 (m, 5H), 7.47 – 7.43 (m, 4H), 7.40 – 7.28 (m, 7H), 7.24 – 7.18 (m, 1H), 6.46 (d, *J* = 15.6 Hz, 1H), 6.37 (dd, *J* = 16.0, 7.6 Hz, 1H), 3.48 (q, *J* = 7.4 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.91 – 1.85 (m, 2H), 1.80 – 1.72 (m, 2H), 1.51 – 1.33 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.42, 143.63, 140.94, 139.08, 137.44, 136.96, 134.07, 132.85, 129.41, 128.68, 128.51, 128.44, 127.99, 127.97, 127.21, 127.03, 127.02, 126.98, 126.11, 48.75, 38.44, 35.61, 29.18, 27.42, 24.10 ppm; IR (neat): υ_{max} 3026, 2926, 2857, 1682, 1593, 1487, 1451, 1405, 1365, 1261, 1214, 1078, 1015, 967, 833, 802, 750, 692 cm^{-1}; HRMS (ESI) calcd for $C_{33}H_{32}NaO$ $[M+Na]^+$ 467.2345, found 467.2351.



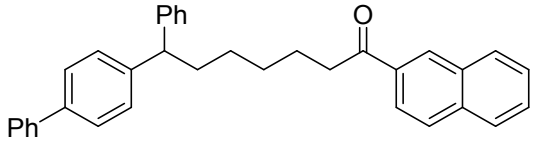
7-([1,1'-biphenyl]-4-yl)-7-phenyl-1-(p-tolyl)heptan-1-one (6a): white solid, mp: 69–70 °C (46%, 39.5 mg); R_f 0.47 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.82 (d, *J* = 8.4 Hz, 2H), 7.58 – 7.51 (m, 2H), 7.50 – 7.48 (m, 2H), 7.42 – 7.38 (m, 2H), 7.33 – 7.19 (m, 9H), 7.19 – 7.15 (m, 2H), 3.92 (t, *J* = 7.8 Hz, 1H), 2.88 (t, *J* = 8.0 Hz, 2H), 2.39 (s, 3H), 2.11 – 2.05 (m, 2H), 1.74 – 1.67 (m, 2H), 1.46 – 1.39 (m, 2H), 1.39 – 1.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.12, 145.05, 144.29, 143.57, 140.91, 138.85, 134.50, 129.18, 128.65, 128.42, 128.18, 128.13, 127.82, 127.09, 126.98, 126.95, 126.08, 50.94, 38.31, 35.46, 29.23, 27.82, 24.19, 21.59 ppm; IR (neat): υ_{max} 3025, 2923, 2856, 1677, 1603, 1483, 1451, 1406, 1365, 1213, 1181, 1077, 1015, 807, 758, 696 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO$ $[M+Na]^+$ 455.2345, found 455.2352



7-([1,1'-biphenyl]-4-yl)-1-(4-trifluoromethylphenyl)-7-phenylheptan-1-one (6b): white solid, mp: 55–56 °C (63%, 61.7 mg); R_f 0.42 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.04 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.61 – 7.56 (m, 2H), 7.55 – 7.53 (m, 2H), 7.46 – 7.42 (m, 2H), 7.37 – 7.29 (m, 7H), 7.22 – 7.19 (m, 1H), 3.97 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.16 – 2.10 (m, 2H), 1.80 – 1.72 (m, 2H), 1.52 – 1.35 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 199.27, 144.97, 144.22, 140.83, 139.56, 138.88, 134.11 (q, *J* = 32.3 Hz), 128.66, 128.43, 128.29, 128.17, 127.79, 127.08, 127.02, 126.91, 126.12, 125.59 (q, *J* = 3.7 Hz), 123.58 (q, *J* = 271.0 Hz), 50.92, 38.65, 35.41, 29.07, 27.75, 23.77 ppm; IR (neat): υ_{max} 3029, 2931, 2860, 1691, 1593, 1491, 1453, 1408, 1323, 1170, 1127, 1068, 1014, 832, 761, 700 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{29}F_3NaO$ $[M+Na]^+$ 509.2063, found 509.2064.

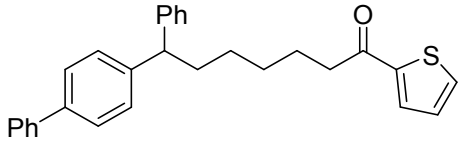


7-([1,1'-biphenyl]-4-yl)-1-(3-fluorophenyl)-7-phenylheptan-1-one (6c): white solid, mp: 76–77 °C (68%, 59.7 mg); R_f 0.39 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.70 – 7.76 (m, 1H), 7.62 – 7.59 (m, 1H), 7.57 – 7.52 (m, 2H), 7.50 – 7.48 (m, 2H), 7.41 – 7.36 (m, 3H), 7.33 – 7.23 (m, 7H), 7.23 – 7.14 (m, 2H), 3.92 (t, *J* = 7.8 Hz, 1H), 2.88 (t, *J* = 7.2 Hz, 2H), 2.11 – 2.05 (m, 2H), 1.74 – 1.67 (m, 2H), 1.47 – 1.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 198.99 (d, *J* = 2.0 Hz), 162.78 (d, *J* = 246.2 Hz), 144.98, 144.23, 140.85, 139.03 (d, *J* = 5.9 Hz), 138.86, 130.15 (d, *J* = 7.6 Hz), 128.64, 128.42, 128.16, 127.79, 127.07, 126.98, 126.91, 126.10, 123.71 (d, *J* = 2.9 Hz), 119.84 (d, *J* = 21.3 Hz), 114.70 (d, *J* = 21.9 Hz), 50.92, 38.52, 35.42, 29.11, 27.78, 23.88 ppm; IR (neat): υ_{max} 3029, 2930, 2859, 1687, 1590, 1486, 1446, 1362, 1252, 1160, 1079, 1015, 836, 761, 696 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{29}FNaO$ $[M+Na]^+$ 459.2095, found 459.2094.

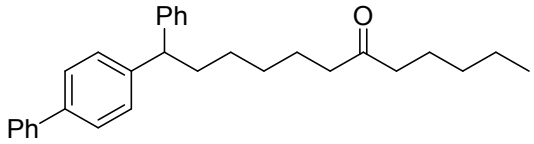


7-([1,1'-biphenyl]-4-yl)-1-(naphthalen-2-yl)-7-phenylheptan-1-one (6d): white solid, mp: 158–159 °C (53%, 49.4 mg); R_f 0.33 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.46 (s, 1H), 8.05 – 8.02 (m, 1H), 7.97 – 7.95 (m, 1H), 7.93 – 7.86 (m, 2H), 7.64 – 7.49 (m, 6H), 7.44 – 7.41 (m, 2H), 7.36 – 7.28 (m, 7H), 7.23 – 7.18 (m, 1H), 3.97 (t, *J* = 7.8 Hz, 1H), 3.08 (t, *J* = 7.4 Hz, 2H), 2.16 – 2.10 (m, 2H), 1.84 – 1.77 (m, 2H), 1.54 – 1.47 (m, 2H), 1.45 – 1.35 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.38, 145.04, 144.28, 140.89, 138.86, 135.45, 134.30, 132.48, 129.57, 129.49, 128.65, 128.43, 128.36, 128.30, 128.19, 127.82, 127.72, 127.09, 126.98, 126.94, 126.68, 126.09, 123.89, 50.94, 38.48, 35.47, 29.23, 27.83, 24.22 ppm; IR (neat): υ_{max} 3029, 2927, 2858, 1678, 1594, 1480, 1456, 1366, 1266, 1177, 1085,

1022, 908, 811, 754, 700 cm^{-1}; HRMS (ESI) calcd for $C_{35}H_{32}NaO$ $[M+Na]^+$ 491.2345, found 491.2342.

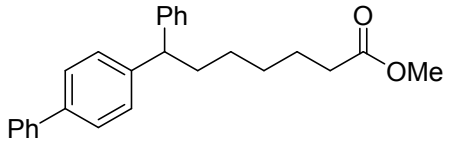


7-([1,1'-biphenyl]-4-yl)-7-phenyl-1-(thiophen-2-yl)heptan-1-one (6e): colorless oil (48%, 40.9 mg); R_f 0.25 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.68 (dd, *J* = 4.0, 1.2 Hz, 1H), 7.62 – 7.56 (m, 3H), 7.53 – 7.51 (m, 2H), 7.46 – 7.40 (m, 2H), 7.36 – 7.27 (m, 7H), 7.22 – 7.18 (m, 1H), 7.11 (dd, *J* = 4.8, 3.6 Hz, 1H), 3.95 (t, *J* = 7.8 Hz, 1H), 2.87 (t, *J* = 7.4 Hz, 2H), 2.14 – 2.08 (m, 2H), 1.79 – 1.71 (m, 2H), 1.52 – 1.41 (m, 2H), 1.41 – 1.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 193.37, 145.00, 144.41, 144.26, 140.89, 138.85, 133.34, 131.65, 128.64, 128.42, 128.17, 127.99, 127.80, 127.08, 126.98, 126.93, 126.09, 50.92, 39.18, 35.43, 29.16, 27.75, 24.47 ppm; IR (neat): υ_{max} 3027, 2927, 2858, 1661, 1597, 1485, 1453, 1412, 1362, 1259, 1022, 912, 804, 758, 727 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{28}NaOS$ $[M+Na]^+$ 447.1753, found 447.1752.

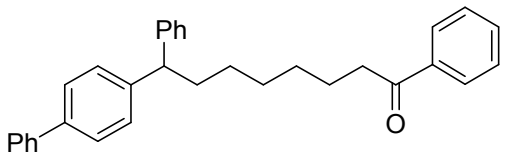


12-([1,1'-biphenyl]-4-yl)-12-phenyldodecan-6-one (6f): colorless oil (86%, 71.2 mg); R_f 0.44 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.57 – 7.55 (m, 2H), 7.52 – 7.49 (m, 2H), 7.44 – 7.40 (m, 2H), 7.34 – 7.26 (m, 7H), 7.21 – 7.16 (m, 1H), 3.92 (t, *J* = 7.8 Hz, 1H), 2.35 (t, *J* = 8.2 Hz, 4H), 2.10 – 2.04 (m, 2H), 1.58 – 1.51 (m, 4H), 1.38 – 1.24 (m, 8H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 211.62, 145.04, 144.30, 140.92, 138.89, 128.67, 128.44, 128.18, 127.82, 127.11, 127.01, 126.96, 126.11, 50.95, 42.78, 42.64, 35.48, 31.41, 29.17, 27.79, 23.64, 23.53, 22.45, 13.93 ppm; IR (neat): υ_{max} 3027, 2956, 2920, 2857, 1708,

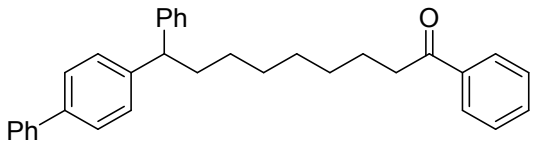
1597, 1453, 1408, 1368, 1261, 1083, 1022, 802, 757, 695 cm^{-1}; HRMS (ESI) calcd for $C_{30}H_{36}NaO$ $[M+Na]^+$ 435.2658, found 435.2658.



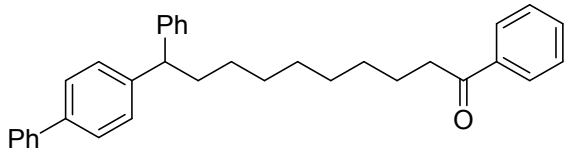
methyl 7-([1,1'-biphenyl]-4-yl)-7-phenylheptanoate (6g): colorless oil (60%, 44.6 mg); R_f 0.45 (EtOAc/petroleum ether = 1:30); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.56 – 7.54 (m, 2H), 7.51 – 7.49 (m, 2H), 7.42 – 7.39 (m, 2H), 7.32 – 7.25 (m, 7H), 7.21 – 7.15 (m, 1H), 3.92 (t, *J* = 7.8 Hz, 1H), 3.64 (s, 3H), 2.27 (t, *J* = 7.5 Hz, 2H), 2.10 – 2.04 (m, 2H), 1.63 – 1.58 (m, 2H), 1.41 – 1.28 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 174.19, 145.03, 144.28, 140.94, 138.93, 128.66, 128.44, 128.18, 127.83, 127.12, 127.01, 126.97, 126.12, 51.43, 50.97, 35.48, 34.01, 29.10, 27.66, 24.77 ppm; IR (neat): υ_{max} 3057, 3026, 2999, 2856, 1601, 1581, 1562, 1543, 1520, 1362, 1331, 1308, 1115, 1088, 1074, 1030, 912, 644, 627 cm^{-1} HRMS (ESI) calcd for $C_{26}H_{28}NaO_2$ $[M+Na]^+$ 395.1982, found 395.1977.



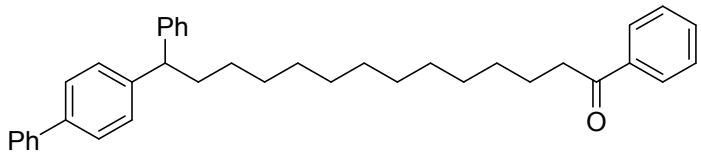
8-([1,1'-biphenyl]-4-yl)-1,8-diphenyloctan-1-one (6h): white solid, mp: 74–75 °C (34%, 29.2 mg); R_f 0.35 (EtOAc/petroleum ether = 1:20); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.50 (m, 5H), 7.49 – 7.40 (m, 4H), 7.36 – 7.27 (m, 7H), 7.23 – 7.17 (m, 1H), 3.94 (t, *J* = 7.8 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.12 – 2.07 (m, 2H), 1.77 – 1.68 (m, 2H), 1.46 – 1.30 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.49, 145.11, 144.37, 140.92, 138.84, 136.98, 132.85, 128.65, 128.51, 128.41, 128.17, 128.00, 127.82, 127.09, 126.98, 126.95, 126.07, 51.00, 38.50, 35.64, 29.46, 29.17, 27.89, 24.26 ppm; IR (neat): υ_{max} 3027, 2926, 2857, 1682, 1593, 1486, 1452, 1406, 1364, 1262, 1214, 1079, 1017, 914, 806, 759, 695 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO$ $[M+Na]^+$ 455.2345, found 455.2346.



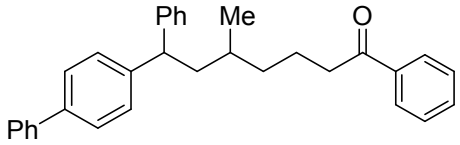
9-([1,1'-biphenyl]-4-yl)-1,9-diphenylnonan-1-one (6i): white solid (62%, 55.4 mg); R_f 0.37 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.98 (d, *J* = 7.2 Hz, 2H), 7.61 – 7.51 (m, 5H), 7.50 – 7.40 (m, 4H), 7.36 – 7.28 (m, 7H), 7.23 – 7.19 (m, 1H), 3.96 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.13 – 2.07 (m, 2H), 1.78 – 1.70 (m, 2H), 1.39 – 1.32 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.51, 145.15, 144.40, 140.91, 138.81, 136.98, 132.83, 128.64, 128.50, 128.40, 128.18, 127.99, 127.81, 127.06, 126.97, 126.93, 126.04, 50.99, 38.52, 35.65, 29.44, 29.27, 29.26, 27.95, 24.23 ppm; IR (neat): υ_{max} 3023, 2921, 2852, 1680, 1520, 1484, 1450, 1373, 1259, 1207, 1082, 1019, 802, 755, 694 cm^{-1}; HRMS (ESI) calcd for $C_{33}H_{34}NaO$ $[M+Na]^+$ 469.2502, found 469.2508.



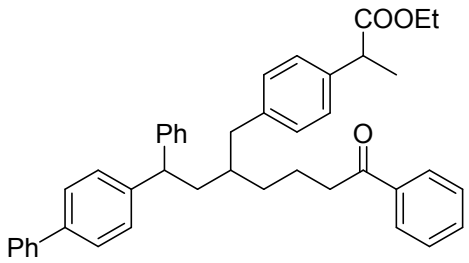
10-([1,1'-biphenyl]-4-yl)-1,10-diphenyldecan-1-one (6j): white solid, mp: 83–84 ^{o}C (36%, 33mg); R_f 0.50 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.97 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.51 (m, 5H), 7.48 – 7.40 (m, 4H), 7.35 – 7.27 (m, 7H), 7.21 – 7.17 (m, 1H), 3.94 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.11 – 2.05 (m, 2H), 1.76 – 1.69 (m, 2H), 1.40 – 1.28 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.57, 145.19, 144.45, 140.94, 138.82, 137.01, 132.84, 128.65, 128.51, 128.40, 128.19, 128.01, 127.84, 127.07, 126.97, 126.95, 126.05, 51.01, 38.57, 35.68, 29.56, 29.40, 29.29, 27.98, 24.29 ppm; IR (neat): υ_{max} 3022, 2921, 2851, 1681, 1519, 1484, 1450, 1365, 1264, 1213, 1012, 807, 753, 694 cm^{-1}; HRMS (ESI) calcd for $C_{34}H_{36}NaO$ $[M+Na]^+$ 483.2658, found 483.2660.



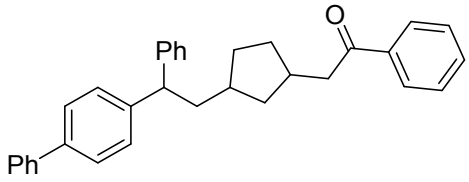
14-([1,1'-biphenyl]-4-yl)-1,14-diphenyltetradecan-1-one (6k): white solid, mp: 84–85 °C (37%, 38.2 mg); R_f 0.53 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.38 (m, 9H), 7.35 – 7.26 (m, 7H), 7.21 – 7.16 (m, 1H), 3.93 (t, *J* = 7.8 Hz, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.10 – 2.04 (m, 2H), 1.77 – 1.69 (m, 2H), 1.38 – 1.24 (m, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.63, 145.23, 144.49, 140.96, 138.82, 137.04, 132.84, 128.66, 128.52, 128.40, 128.20, 128.03, 127.85, 127.07, 126.98, 126.96, 126.04, 51.03, 38.61, 35.72, 29.64, 29.60, 29.58, 29.47, 29.35, 28.03, 24.35 ppm; IR (neat): υ_{max} 3026, 2720, 2852, 1683, 1593, 1452, 1365, 1260, 1210, 1084, 1021, 802, 753, 694 cm^{-1}; HRMS (ESI) calcd for $C_{38}H_{44}NaO$ $[M+Na]^+$ 539.3284, found 539.3286.



7-([1,1'-biphenyl]-4-yl)-5-methyl-1,7-diphenylheptan-1-one (6l): colorless oil (64%, 55.1 mg, dr = 1:1); R_f 0.41 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 8.4 Hz, 2H), 7.60 – 7.50 (m, 5H), 7.49 – 7.40 (m, 4H), 7.38 – 7.29 (m, 7H), 7.24 – 7.17 (m, 1H), 4.13 (dd, *J* = 9.2, 6.8 Hz, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.23 – 2.16 (m, 1H), 1.92 – 1.66 (m, 3H), 1.55 – 1.38 (m, 2H), 1.37 – 1.27 (m, 1H), 1.00 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.37, 145.38, 144.62, 144.57, 143.83, 140.87, 140.84, 138.87, 138.82, 136.96, 132.84, 128.63, 128.49, 128.45, 128.41, 128.31, 128.11, 127.96, 127.95, 127.73, 127.10, 127.07, 126.98, 126.93, 126.91, 126.13, 126.03, 48.24, 48.21, 42.81, 42.79, 38.55, 36.49, 36.46, 29.97, 21.27, 21.24, 19.63, 19.58 ppm; IR (neat): υ_{max} 3028, 2921, 1683, 1594, 1487, 1452, 1367, 1289, 1210, 1079, 1014, 830, 758, 696 cm^{-1}; HRMS (ESI) calcd for $C_{32}H_{32}NaO$ $[M+Na]^+$ 455.2345, found 455.2358.

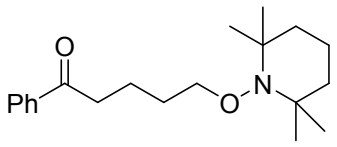


Ethyl-2-(4-(2-(2-([1,1'-biphenyl]-4-yl)-2-phenylethyl)-6-oxo-6-phenylhexyl)phenyl)propanoate (6m): colorless oil (60%, 73.1 mg, dr = 1:1); R_f 0.15 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.90 (d, *J* = 6.8 Hz, 2H), 7.56 – 7.51 (m, 3H), 7.50 – 7.37 (m, 6H), 7.33 – 7.28 (m, 1H), 7.26 – 7.09 (m, 9H), 7.01 – 6.99 (m, 2H), 4.18 – 4.07 (m, 2H), 4.03 (t, *J* = 8.2 Hz, 1H), 3.68 (q, *J* = 7.2 Hz, 1H), 2.84 (t, *J* = 7.0 Hz, 2H), 2.60 (d, *J* = 6.8 Hz, 2H), 2.10 – 1.97 (m, 2H), 1.79 – 1.73 (m, 2H), 1.64 – 1.56 (m, 1H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.44 – 1.38 (m, 2H), 1.22 – 1.16 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.22, 174.69, 144.94, 144.20, 143.65, 140.85, 140.82, 139.67, 138.90, 138.87, 138.85, 138.11, 138.09, 136.93, 132.88, 129.44, 129.43, 128.65, 128.51, 128.46, 128.39, 128.21, 127.97, 127.95, 127.84, 127.22, 127.10, 127.03, 127.00, 126.92, 126.15, 126.11, 60.63, 48.31, 48.29, 45.13, 45.11, 40.09, 40.05, 39.33, 38.54, 36.92, 36.88, 32.90, 20.63, 18.61, 18.57, 14.10 ppm; IR (neat): υ_{max} 3026, 2925, 1729, 1684, 1594, 1484, 1452, 1369, 1325, 1256, 1170, 1082, 1021, 910, 805, 750, 698 cm^{-1}; HRMS (ESI) calcd for $C_{43}H_{44}NaO_3$ $[M+Na]^+$ 631.3183, found 631.3187.

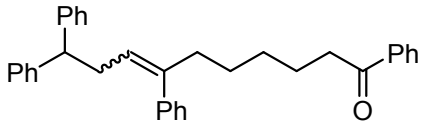


2-(3-(2-([1,1'-biphenyl]-4-yl)-2-phenylethyl)cyclopentyl)-1-phenylethan-1-one (6n): colorless oil (65%, 57.6 mg, dr = 1:1); R_f 0.42 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.98 – 7.95 (m, 2H), 7.61 – 7.50 (m, 5H), 7.50 – 7.40 (m, 4H), 7.37 – 7.27 (m, 7H), 7.21 (m, 1H), 4.01 (t, *J* = 7.8 Hz, 1H), 3.02 (d, *J* = 7.2, 1H), 2.96 (dd, *J* = 7.0, 3.0 Hz, 1H), 2.61 – 2.53(m, 0.5H), 2.42 – 2.34 (m, 0.5H), 2.17 – 2.11 (m, 2.5H), 2.02 – 1.76 (m, 3H), 1.71 – 1.64 (m, 0.5H), 1.53 – 1.46 (m, 0.5H),

1.37 – 1.25 (m, 2H), 1.23 – 1.11 (m, 0.5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.21, 200.15, 145.06, 145.03, 144.97, 144.34, 144.30, 144.28, 144.23, 140.88, 138.85, 138.82, 137.09, 137.04, 132.83, 128.64, 128.48, 128.41, 128.18, 128.16, 128.12, 128.04, 128.02, 127.82, 127.80, 127.76, 127.08, 127.06, 126.97, 126.93, 126.92, 126.08, 126.06, 49.70, 49.60, 45.11, 42.62, 42.56, 40.41, 40.38, 38.39, 37.46, 36.27, 36.26, 35.83, 34.66, 32.97, 32.84, 32.81, 31.53, 31.35, 31.31 ppm; IR (neat): υ_{max} 3028, 2928, 2858, 1682, 1594, 1487, 1450, 1404, 1370, 1273, 1211, 1078, 1005, 910, 831, 750, 696 cm^{-1}; HRMS (ESI) calcd for $C_{33}H_{32}NaO$ $[M+Na]^+$ 467.2345, found 467.2348.



1-phenyl-5-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)pentan-1-one (known compound) (7): colorless oil (83%, 105.3 mg); R_f 0.39 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 (d, *J* = 7.0 Hz, 2H), 7.58 – 7.51 (m, 1H), 7.47 – 7.43 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 3.01 (t, *J* = 7.4 Hz, 2H), 1.87 – 1.80 (m, 2H), 1.65 – 1.58 (m, 2H), 1.54 – 1.40 (m, 5H), 1.34 – 1.27 (m, 1H), 1.15 (s, 6H), 1.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.28, 136.99, 132.84, 128.50, 127.99, 76.43, 59.60, 39.52, 38.55, 33.04, 28.40, 21.42, 20.08, 17.07 ppm; IR (neat): υ_{max} 2933, 1687, 1593, 1455, 1365, 1256, 1134, 1065, 967, 795, 746, 696 cm^{-1}.



1,7,10,10-tetraphenyldec-7-en-1-one (9): colorless oil (58%, 53.0 mg, Z/E = 6.7 : 1); R_f 0.45 (EtOAc/petroleum ether = 1:30); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 7.2 Hz, 2H), 7.56 – 7.51 (m, 1H), 7.45 – 7.42 (m, 2H), 7.29 – 7.16 (m, 15H), 7.14 – 7.09 (m, 1H), 5.55 (t, *J* = 7.0 Hz, 1H), 4.06 (t, *J* = 7.8 Hz, 1H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.87 (t, *J* = 7.4 Hz, 2H), 2.43 (t, *J* = 7.4 Hz, 2H), 1.70 – 1.62 (m, 2H), 1.34 –

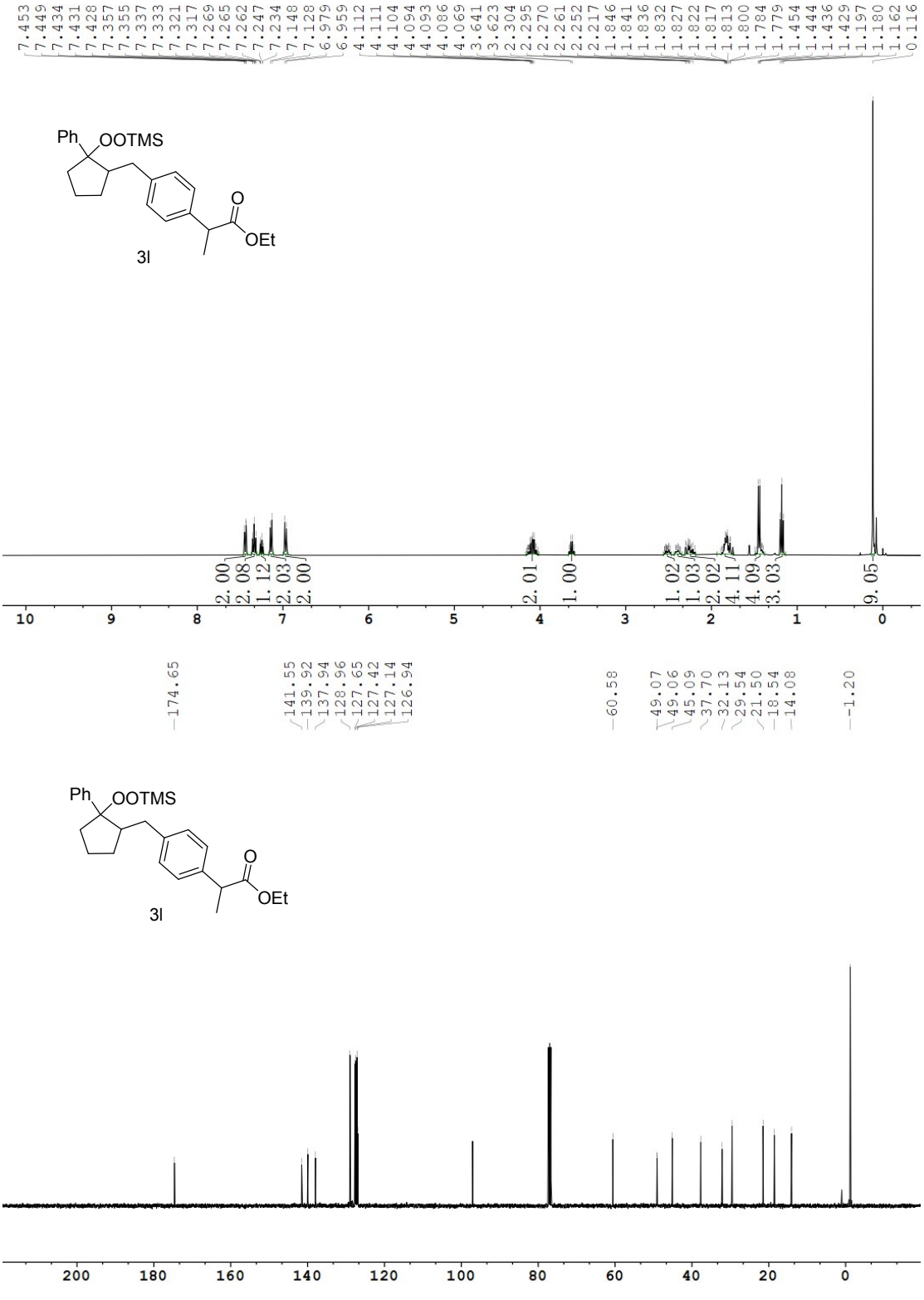
1.25 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 200.38, 144.63, 143.11, 141.20, 137.04, 132.85, 128.52, 128.38, 128.29, 128.22, 128.13, 128.08, 128.02, 126.82, 126.52, 126.41, 126.37, 126.18, 126.01, 51.69, 51.52, 38.43, 34.76, 29.80, 29.25, 28.17, 24.13; IR (neat): υ_{max} 3026, 2928, 2857, 1952, 1883, 1808, 1681, 1591, 1490, 1447, 1360, 1263, 1218, 1080, 1022, 914, 801, 746, 692 cm^{-1}; HRMS (ESI) calcd for $C_{34}H_{34}NaO$ $[M+Na]^+$ 481.2502, found 481.2507.

References

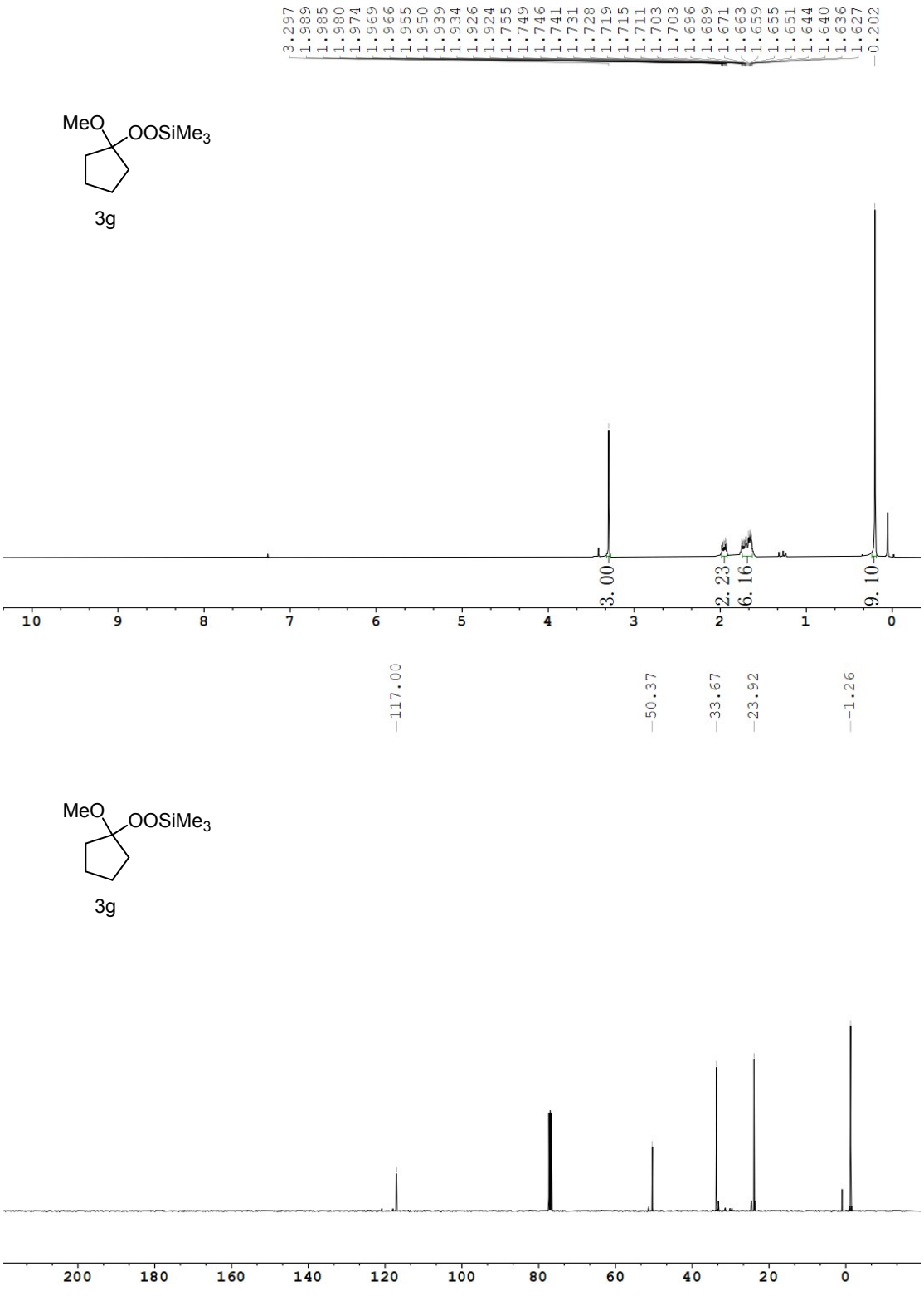
(1) (a) R. Sakamoto, S. Sakurai and K. Maruoka, *Chem.-Eur. J.*, 2017, **23**, 9030. (b) R. Sakamoto, T. Kato, S. Sakurai and K. Maruoka, *Org. Lett*., 2018, **20**, 1400. (c) P. C. Too, Y. L. Tnay and S. Chiba, *Beilstein J. Org. Chem.*, 2013, **9**, 1217. (d) T. Seihara, S. Sakurai, T. Kato, R. Sakamoto and K. Maruoka, *Org. Lett.*, 2019, **21**, 2477. (e) J.-C. Yang, L. Chen, F. Yang, P. Li and L.-N. Guo, *Org. Chem. Front*., 2019, **6**, 2792. (f) L. Chen, J.-C. Yang, P. Xu, J.-J. Zhang, X.-H. Duan and L.-N. Guo, *J. Org. Chem*., 2020, **85**, 7515. (g) S. Sakurai, S. Tsuzuki, R. Sakamoto and K. Maruoka, *J. Org. Chem*., 2020, **85**, 3973. (h) K. Zmitek, M. Zupan, S. Stavber and J. Iskra, *J. Org. Chem.,* 2007, **72**, 6534.

^{1}H NMR and ^{13}C NMR Spectra of Cycloalkylsilyl Peroxides

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of cycloalkylsilyl peroxide 3l (known compound)

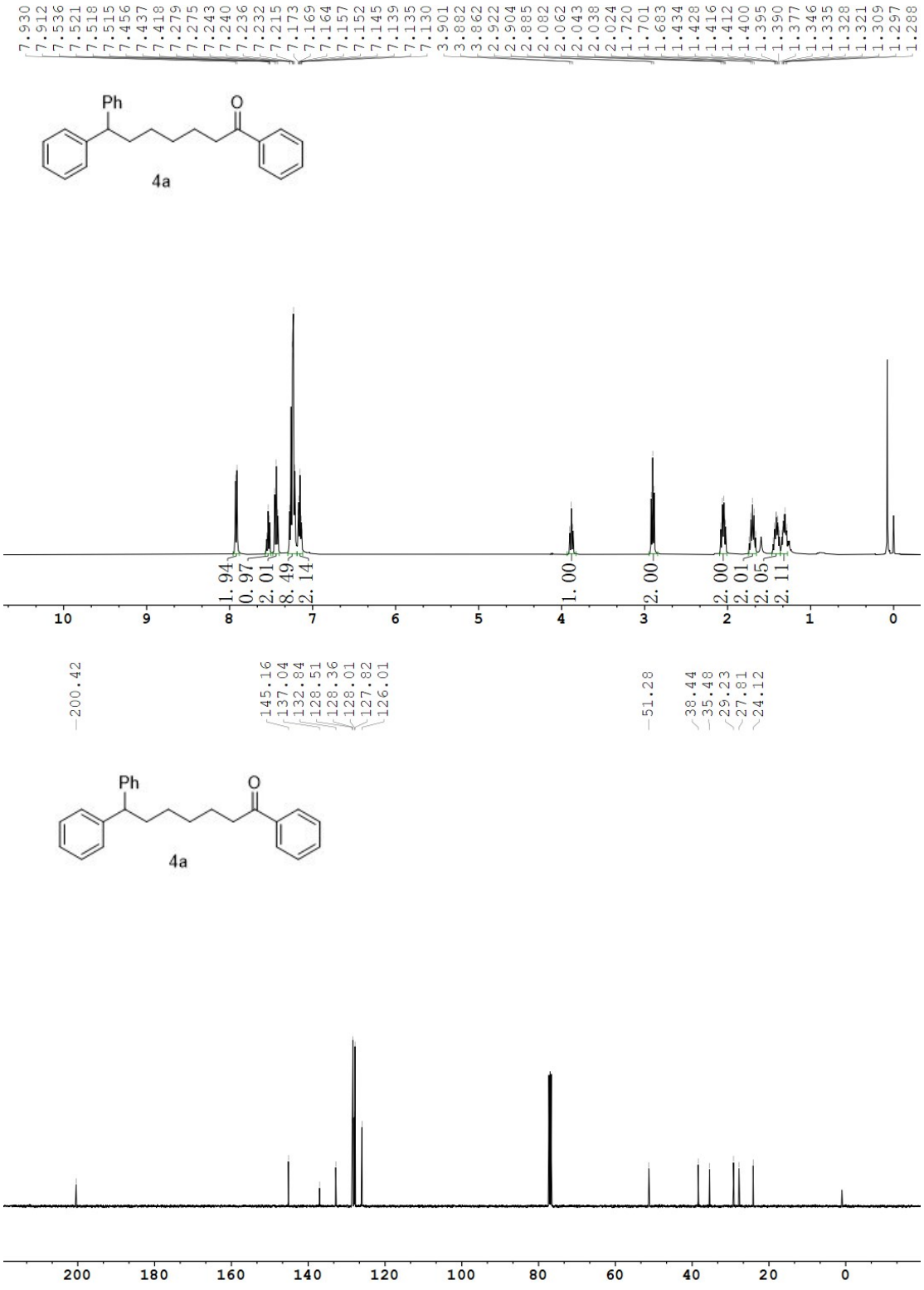


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of cycloalkylsilyl peroxide 3g

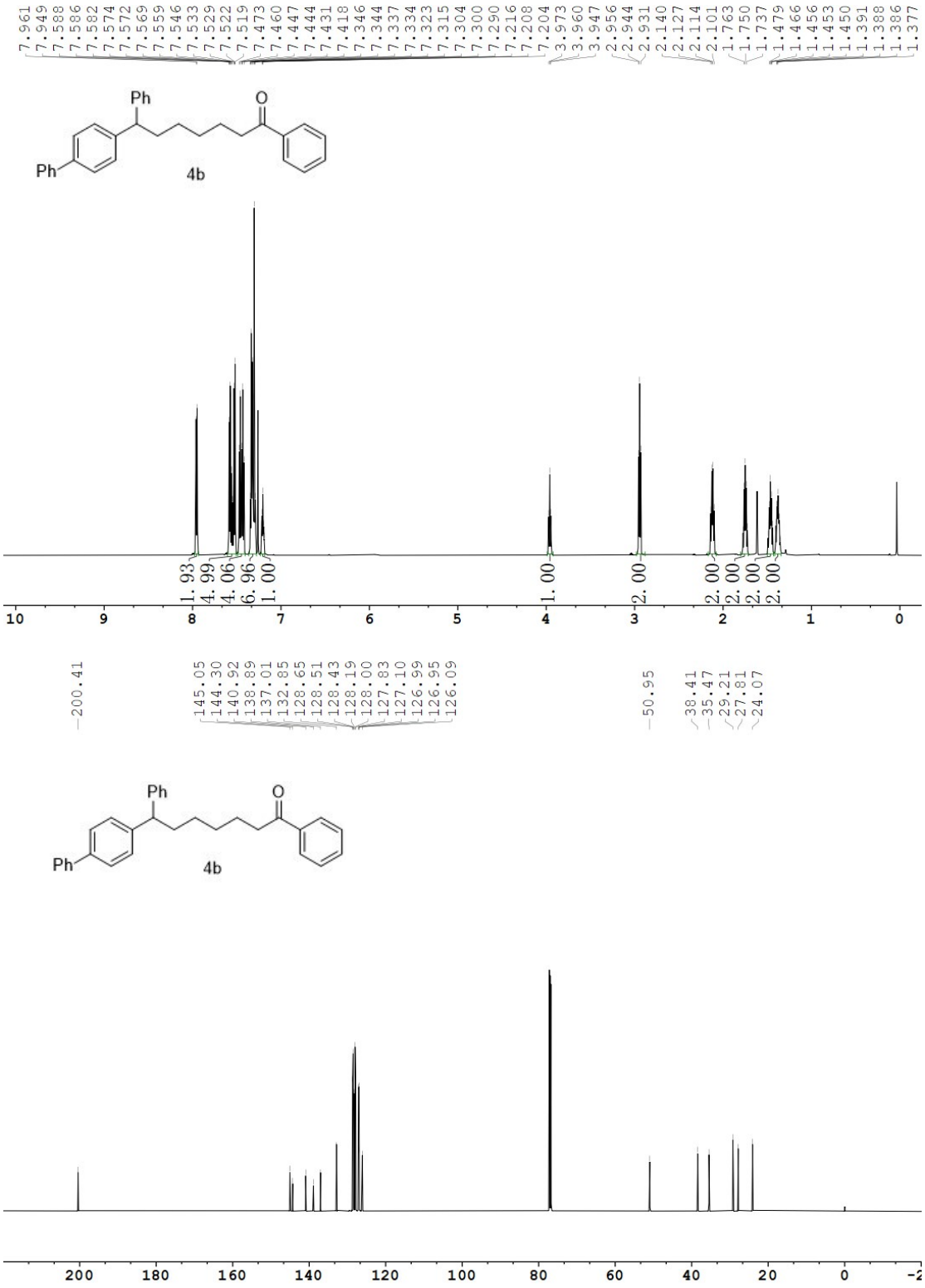


^{1}H NMR and ^{13}C NMR Spectra of the Products 4, 5, 6, 7 and 9

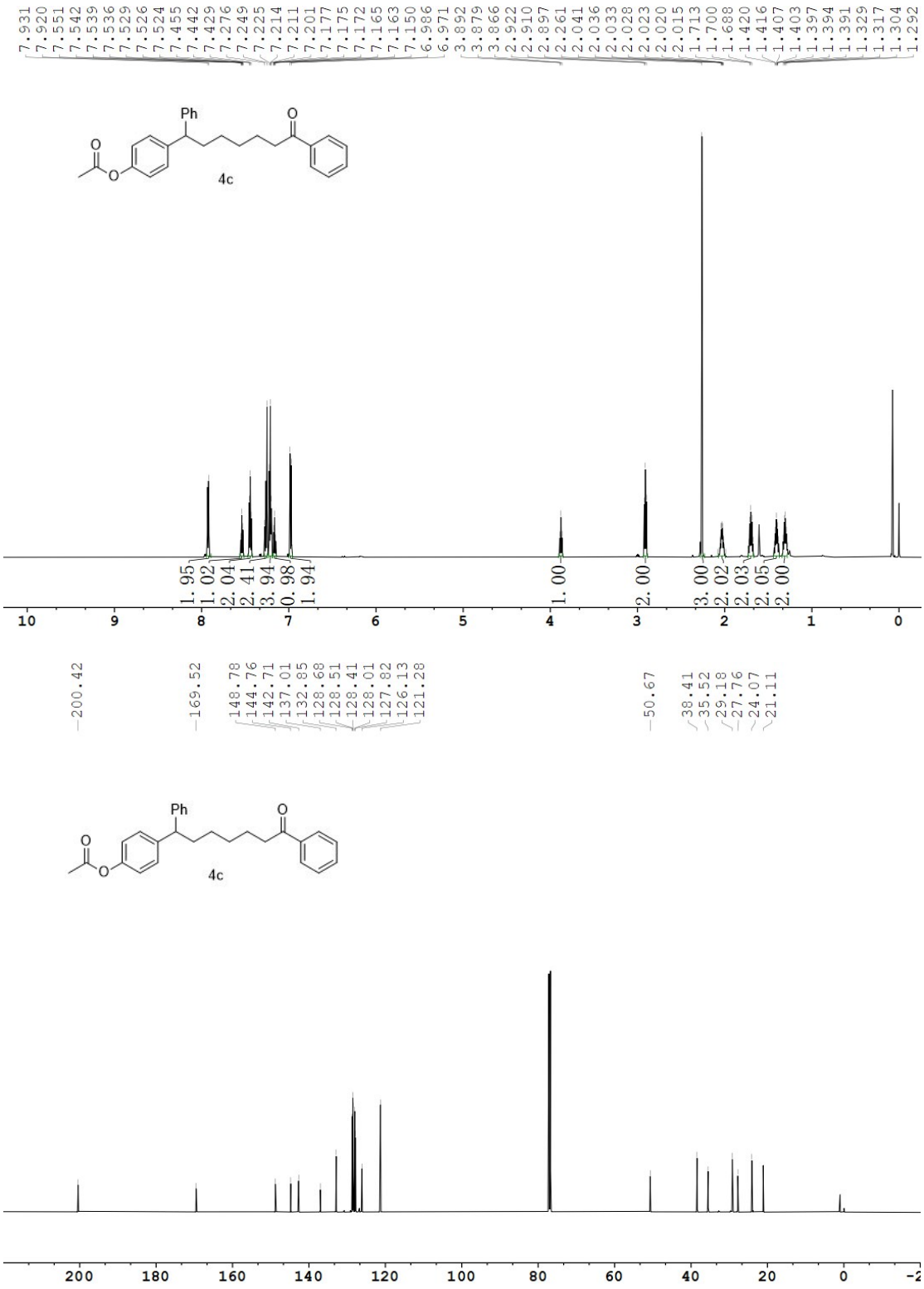
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 4a



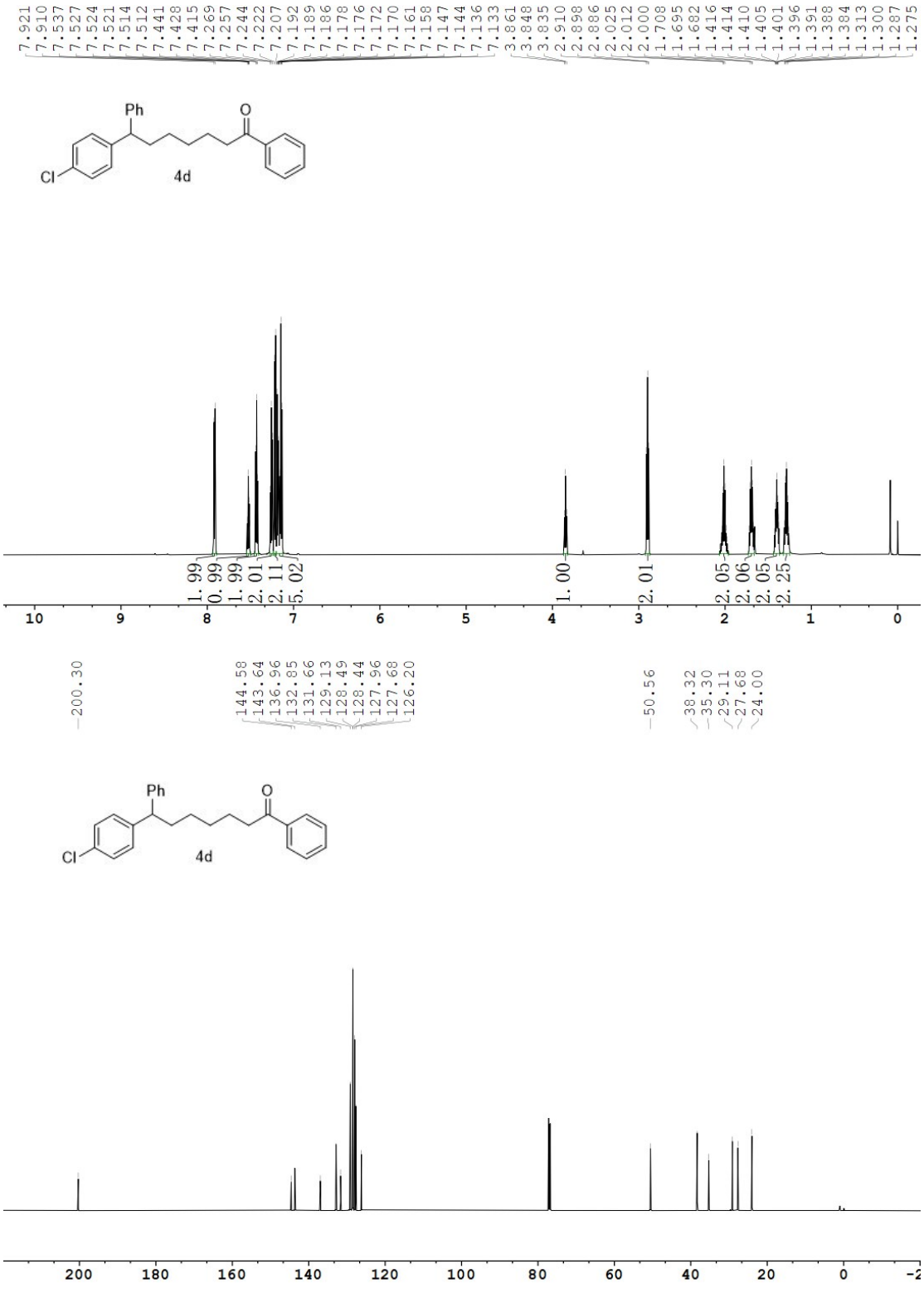
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4b



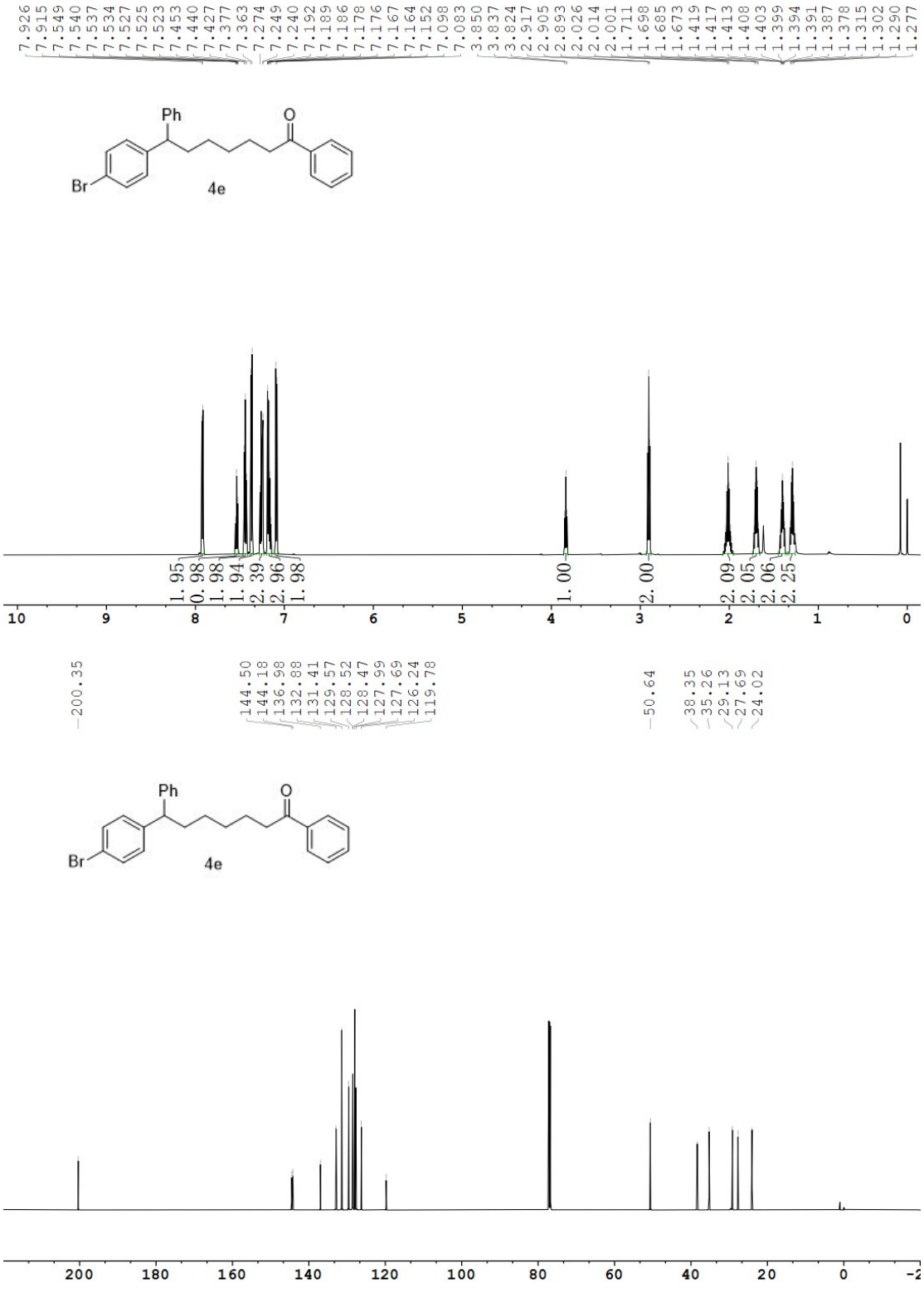
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4c



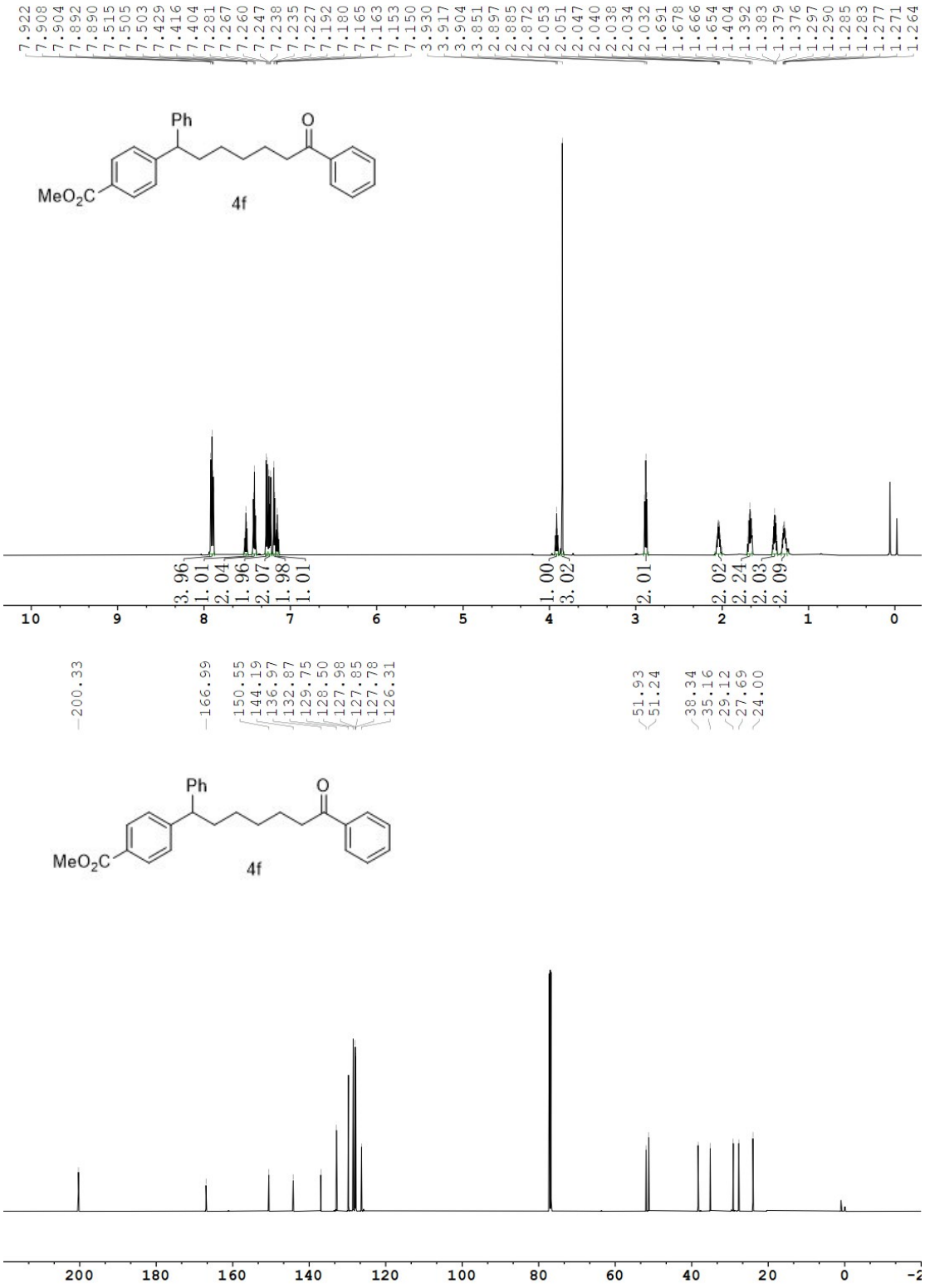
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4d



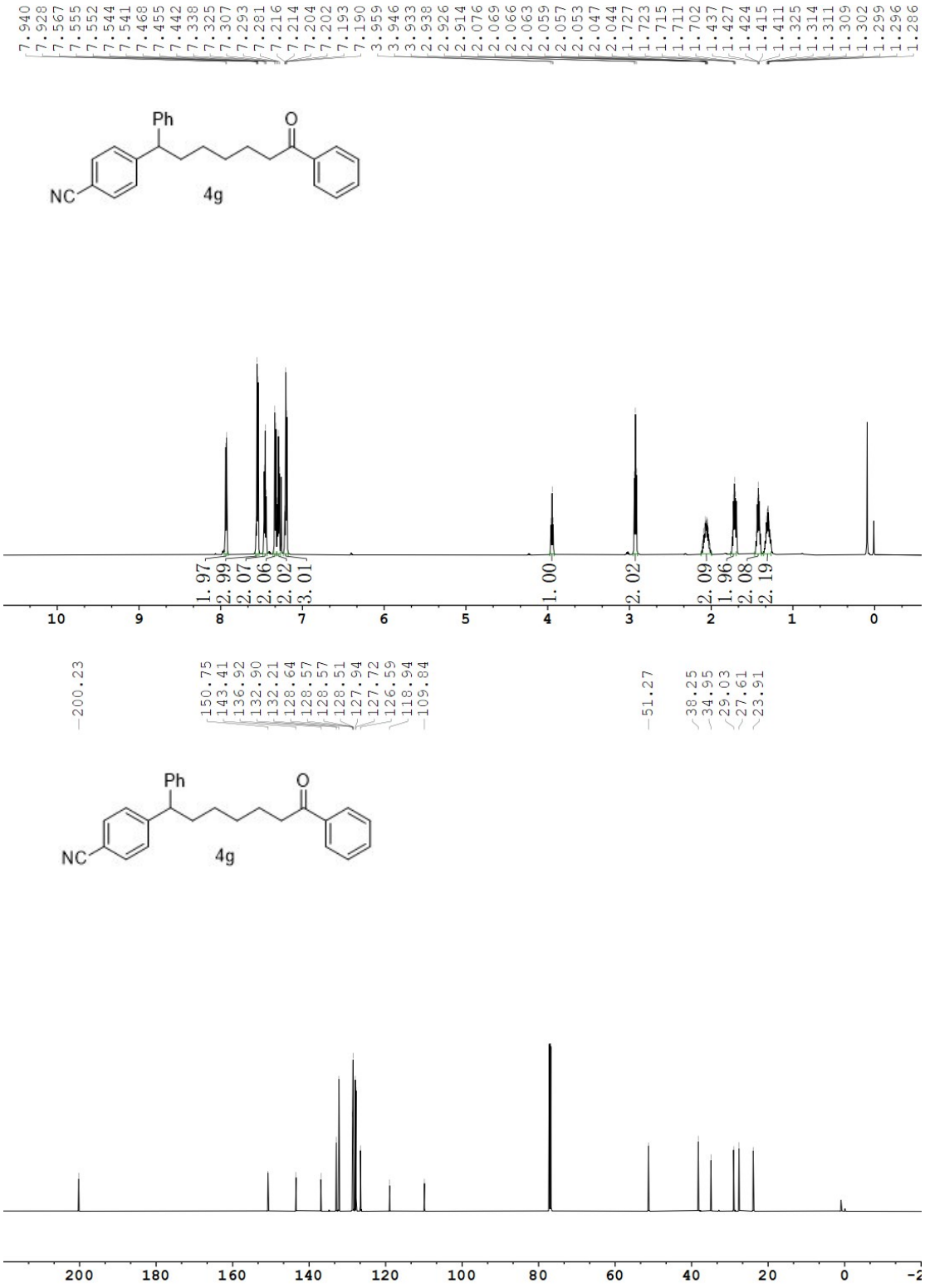
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4e



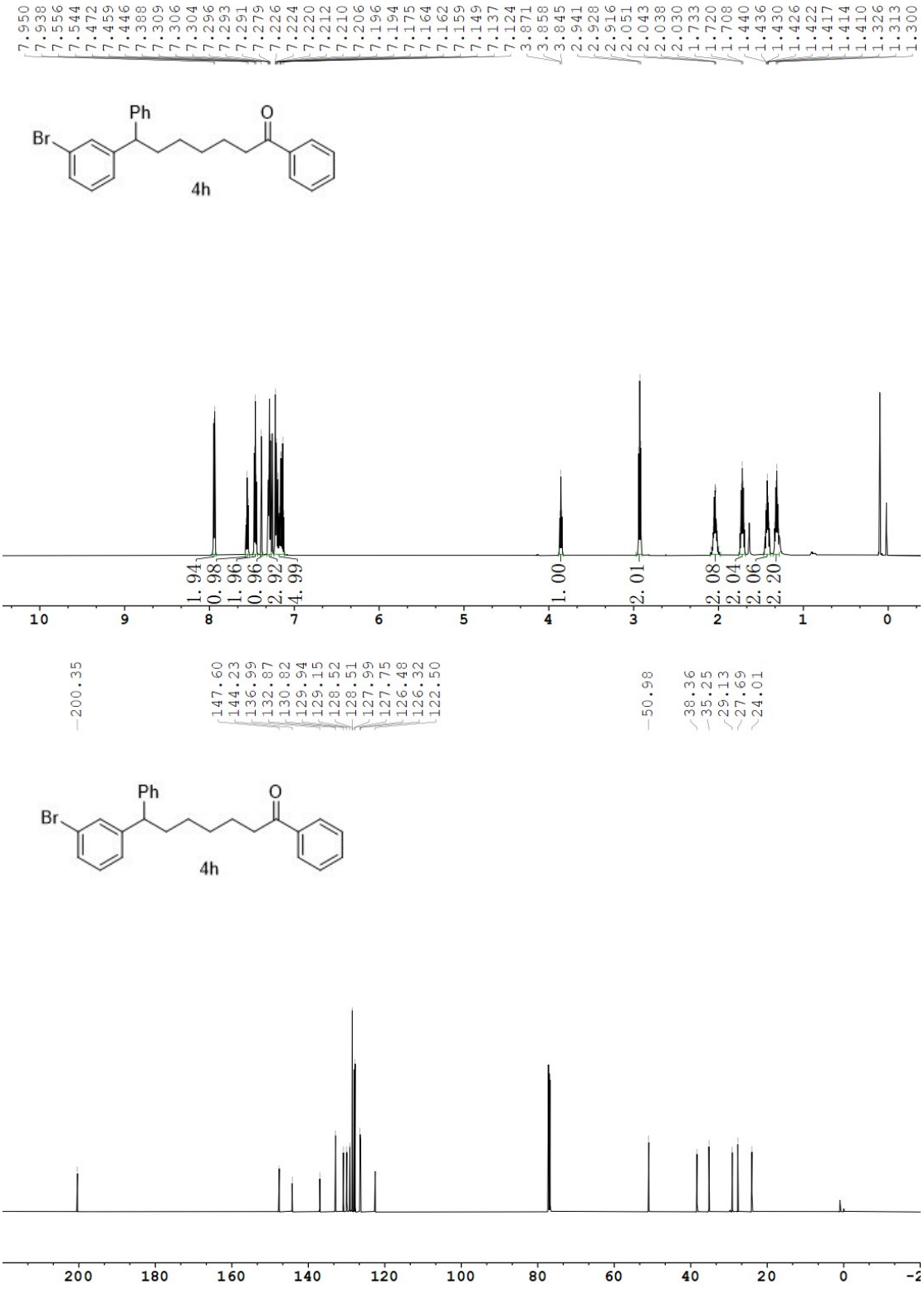
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4f



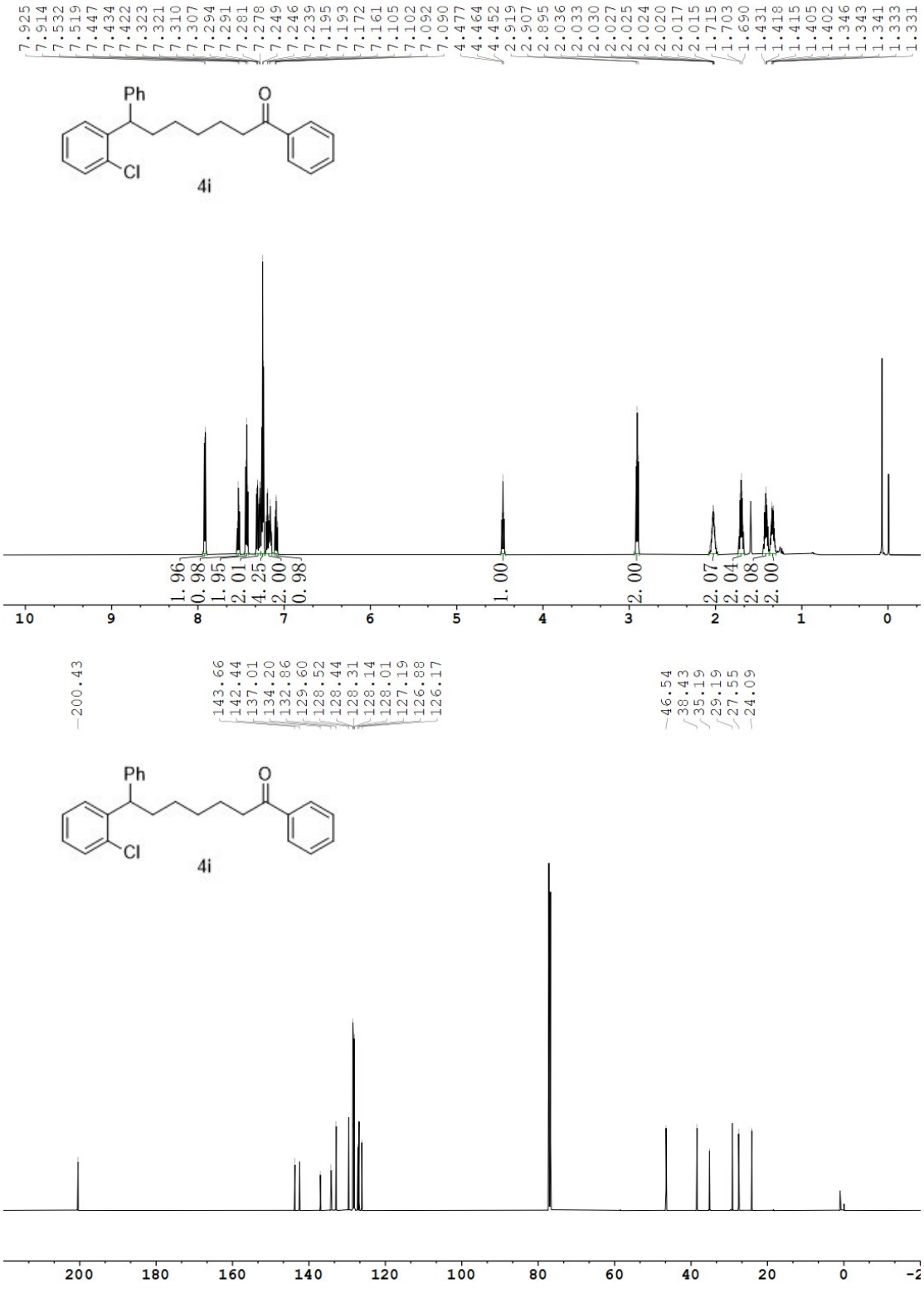
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4g



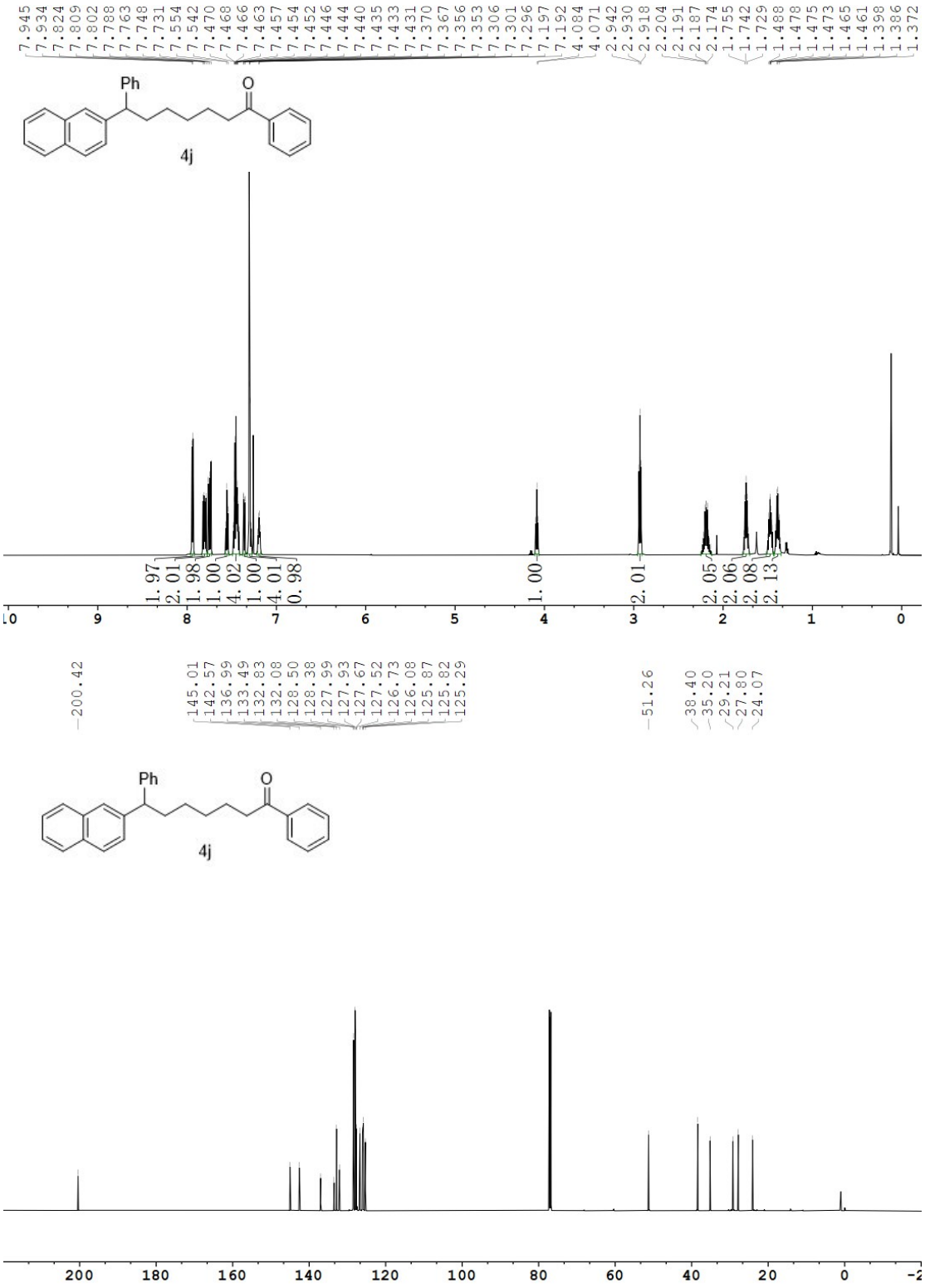
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4h



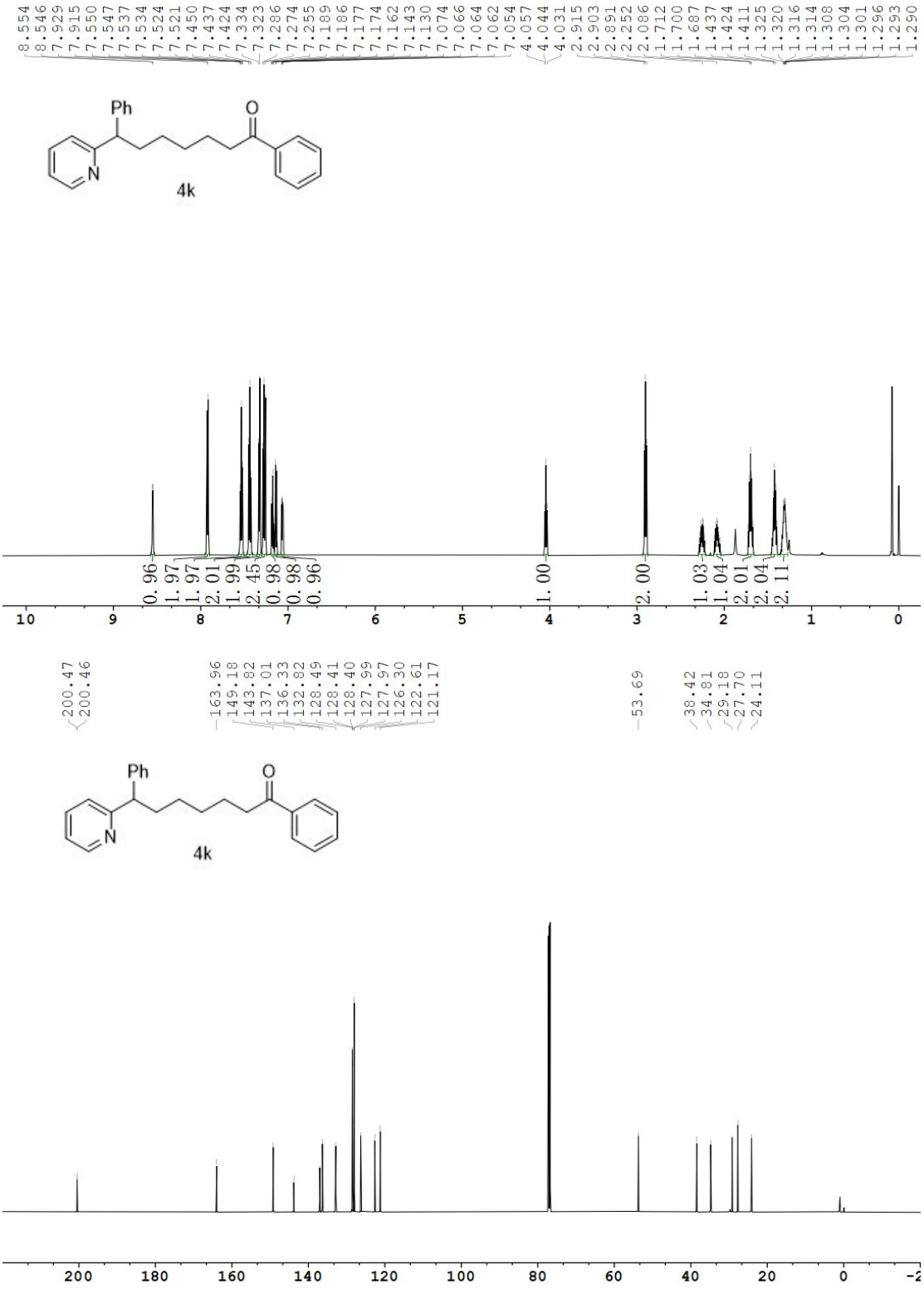
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4i



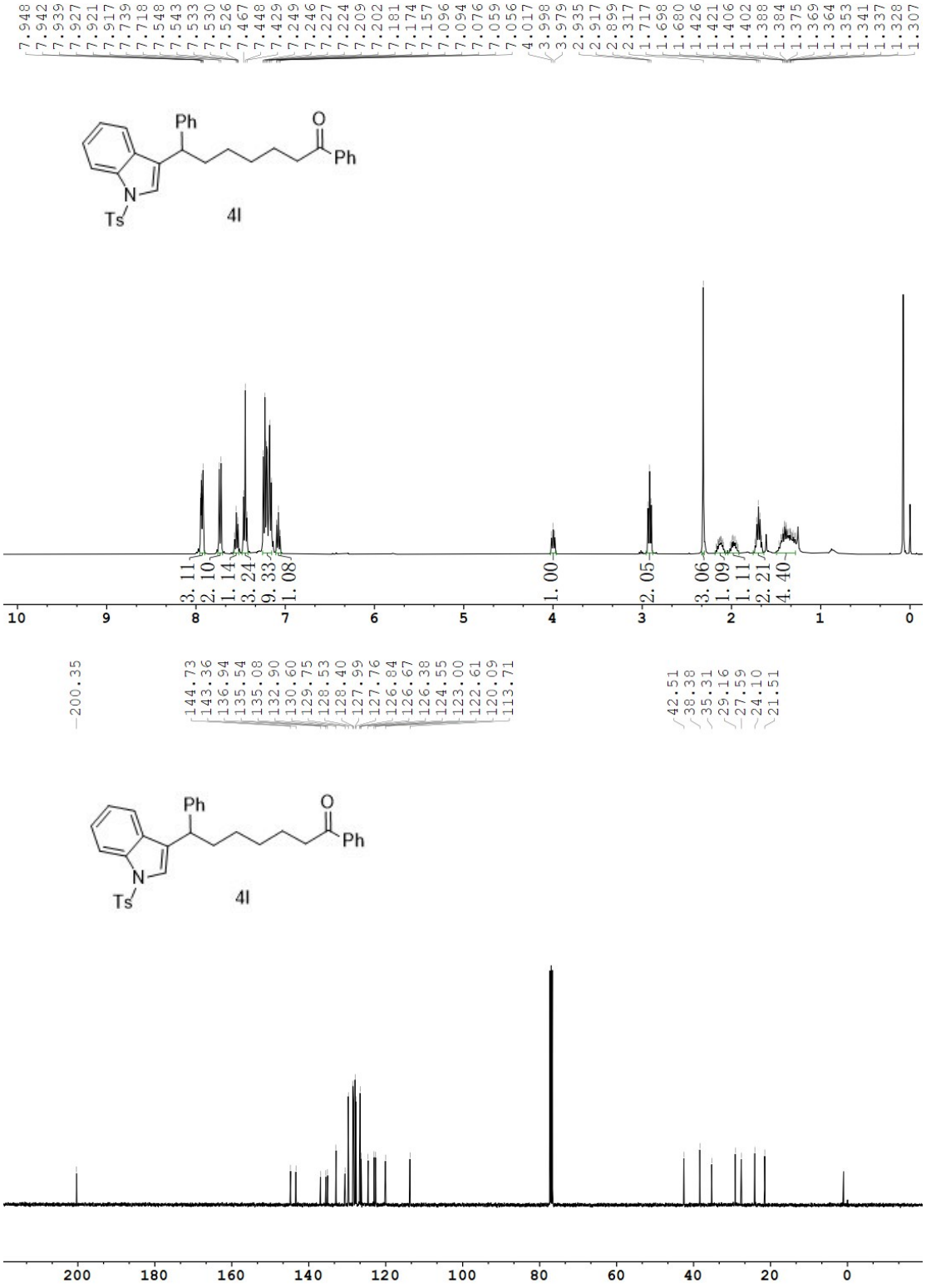
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4j



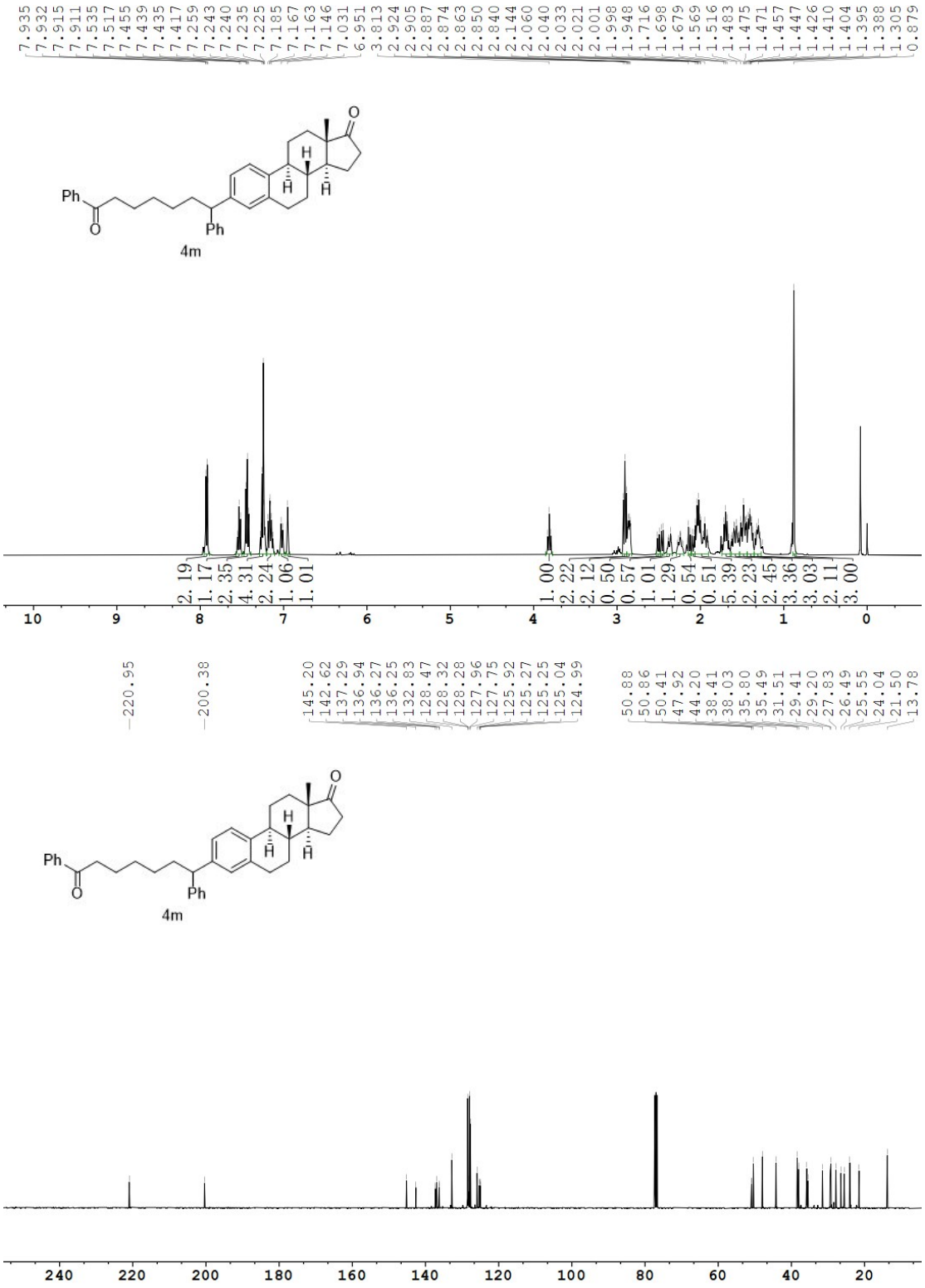
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 4k



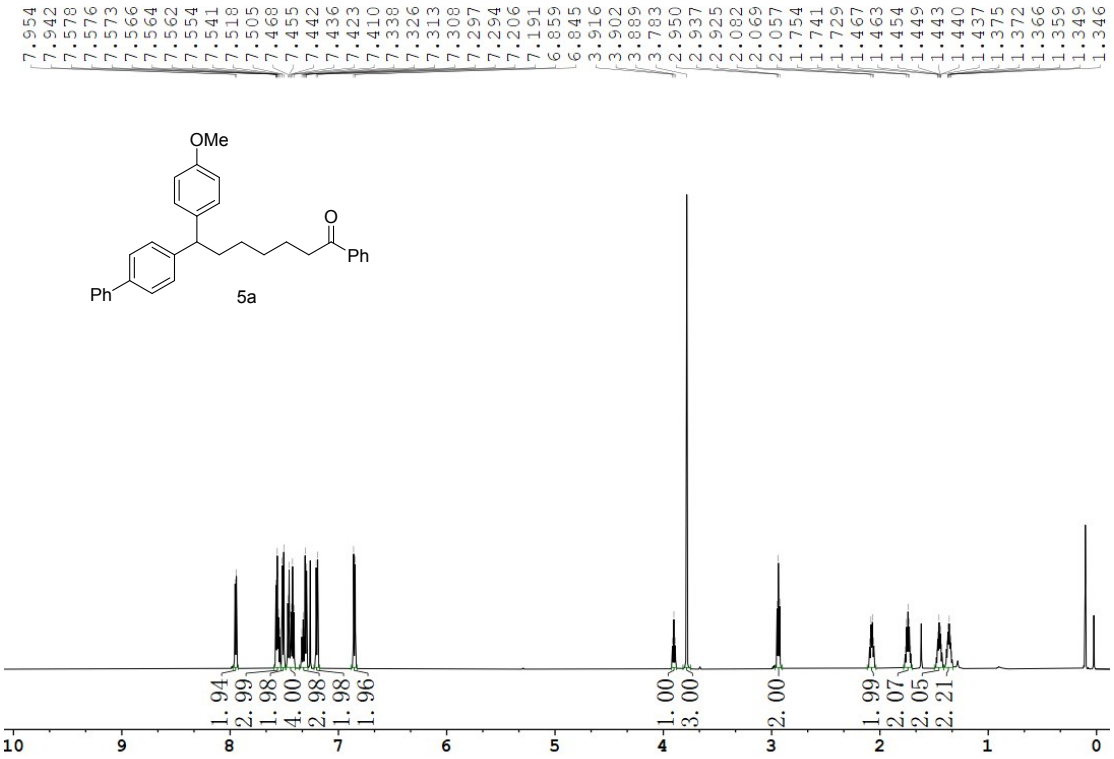
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 4l

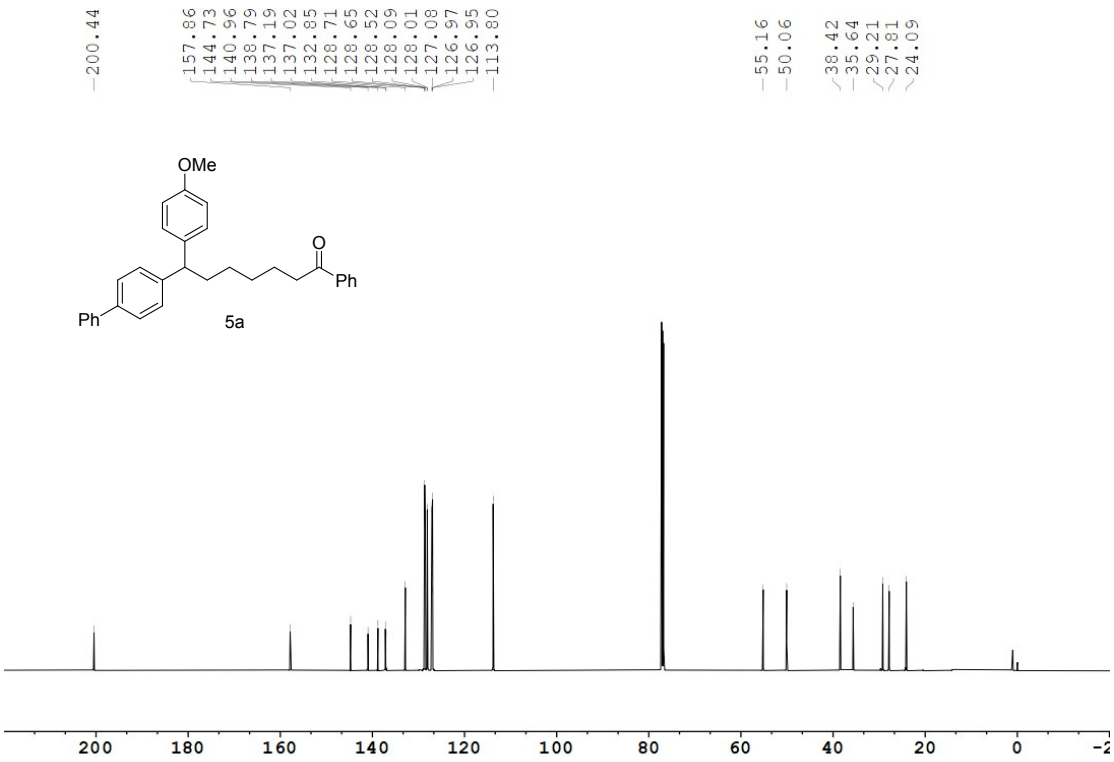


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 4m

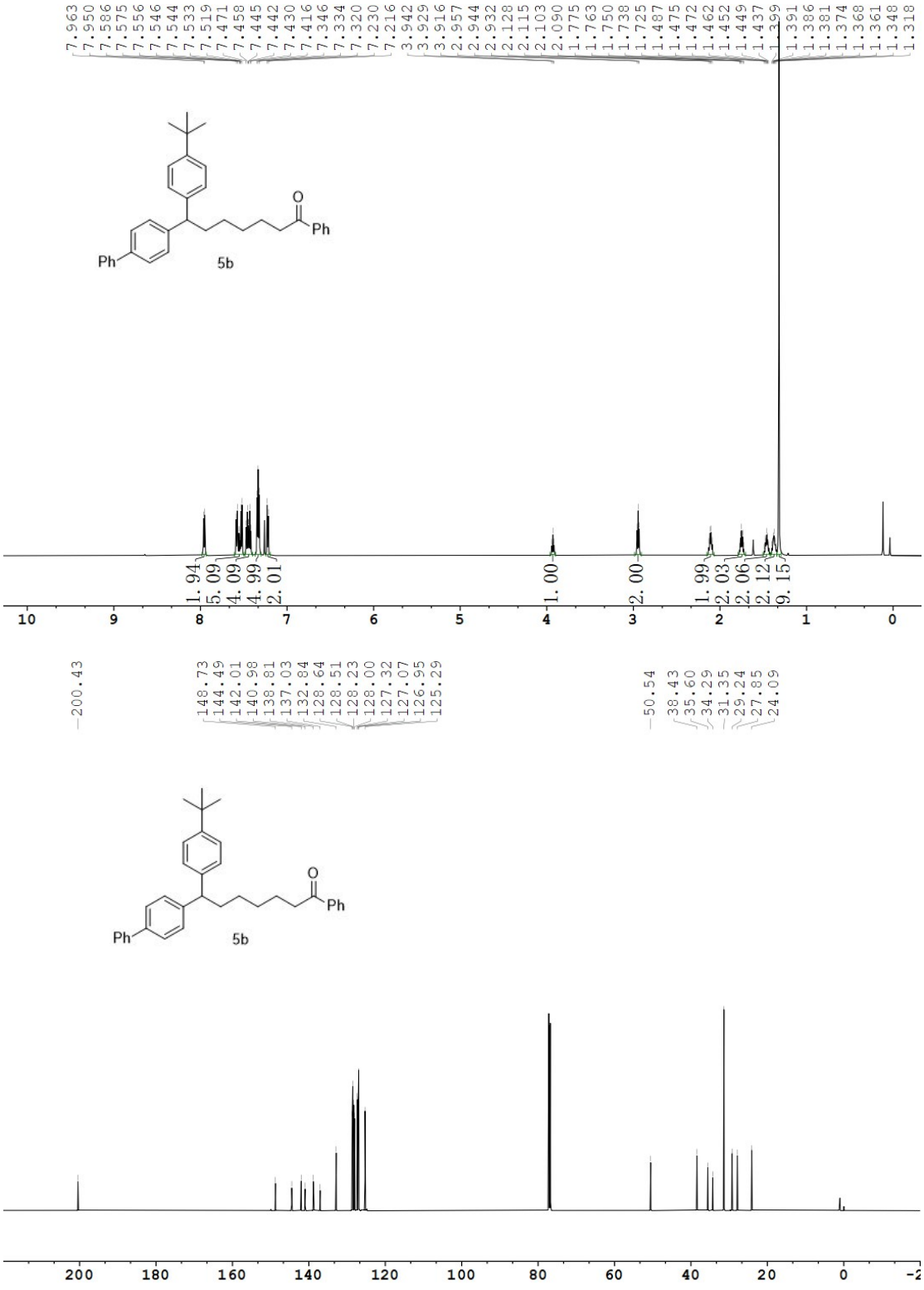


^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5a

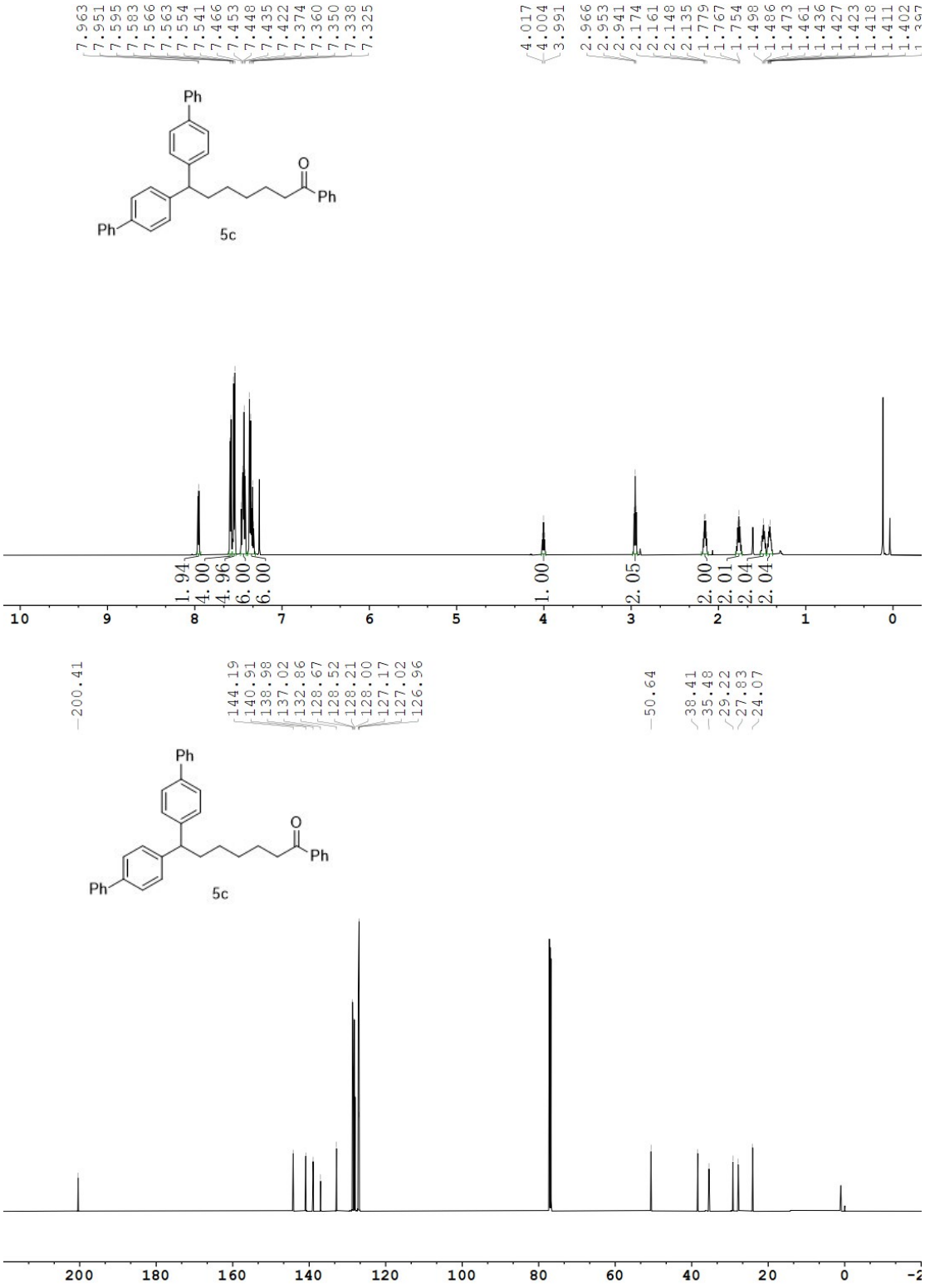




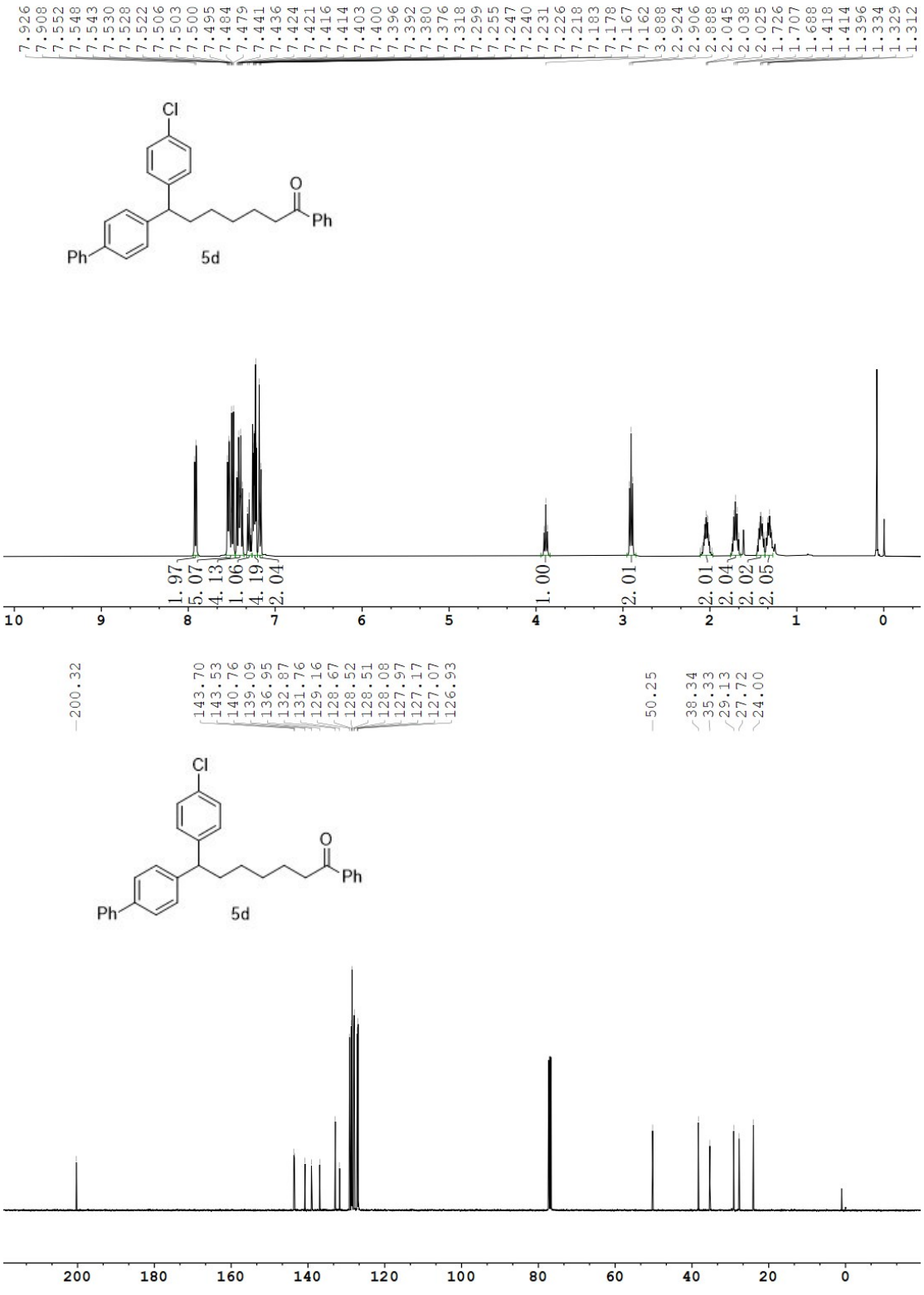
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5b



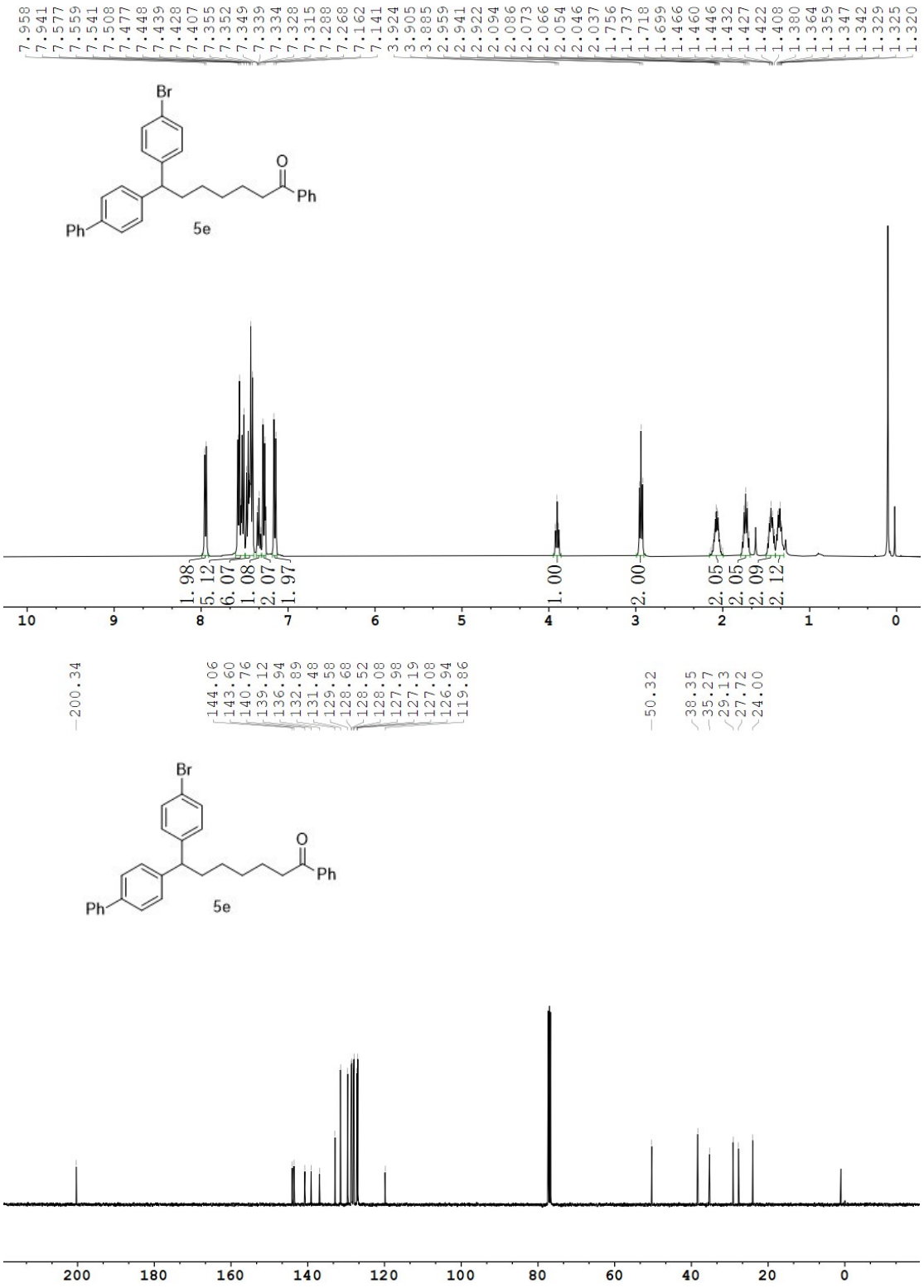
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5c



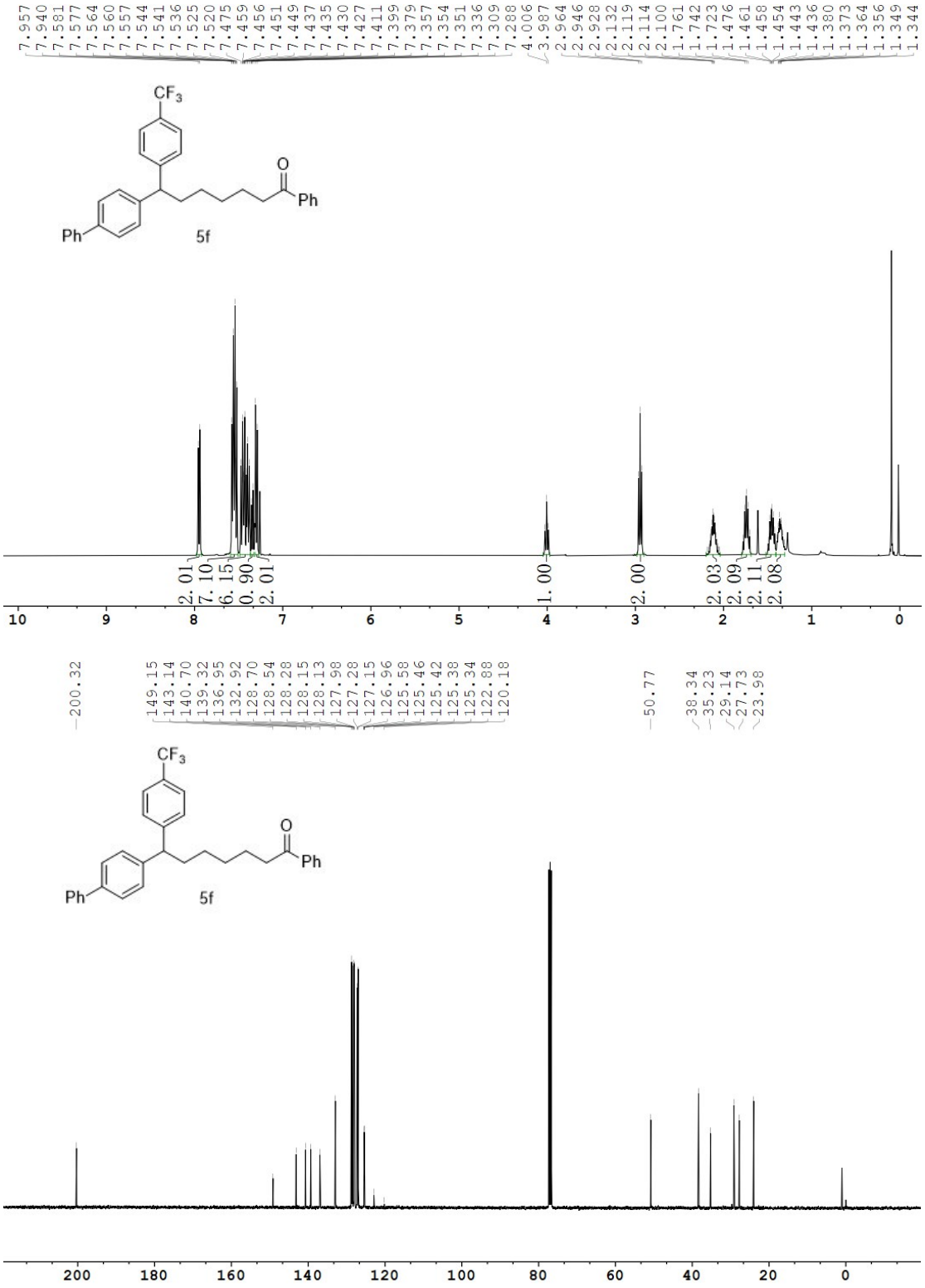
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5d



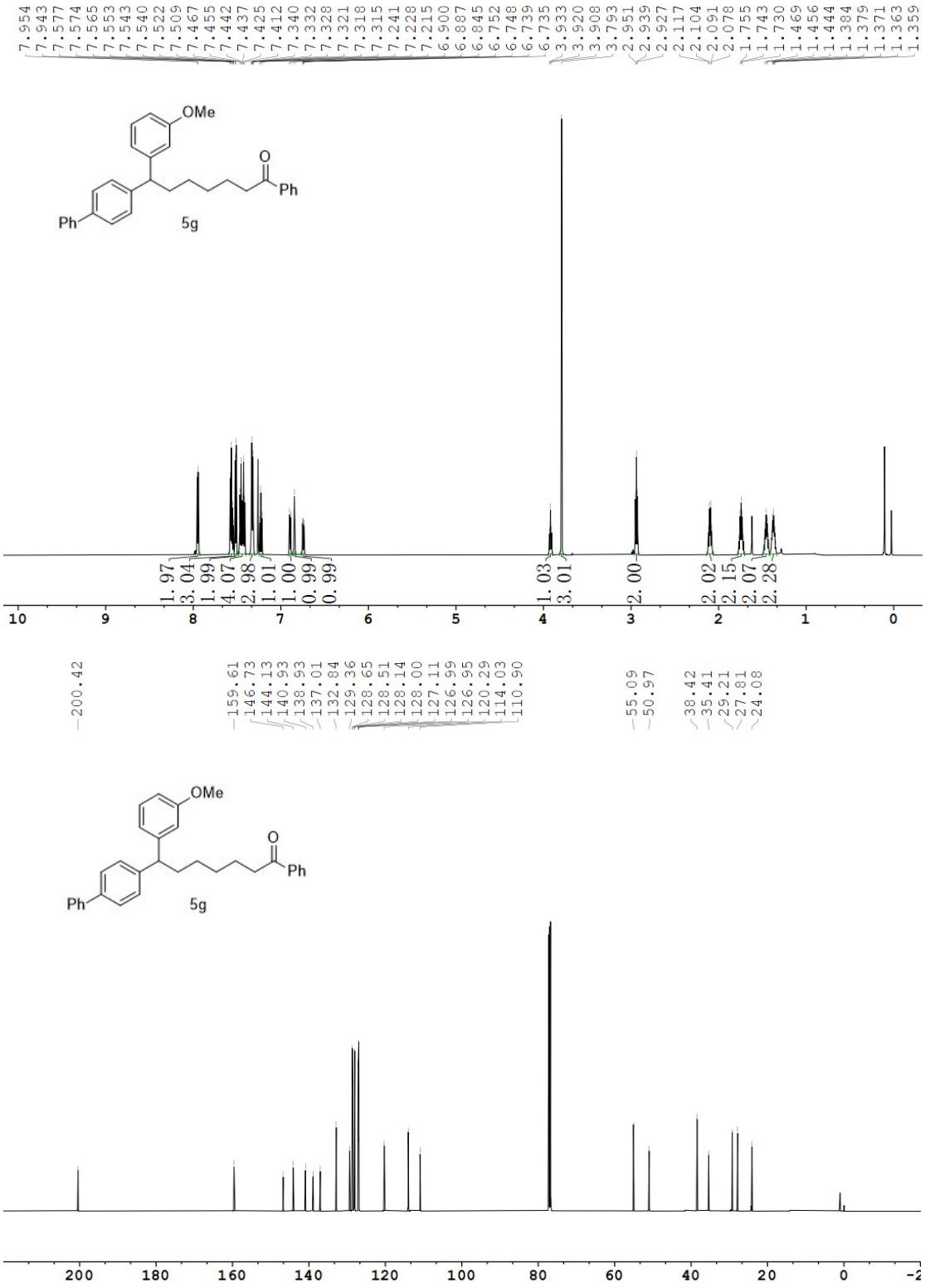
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5e



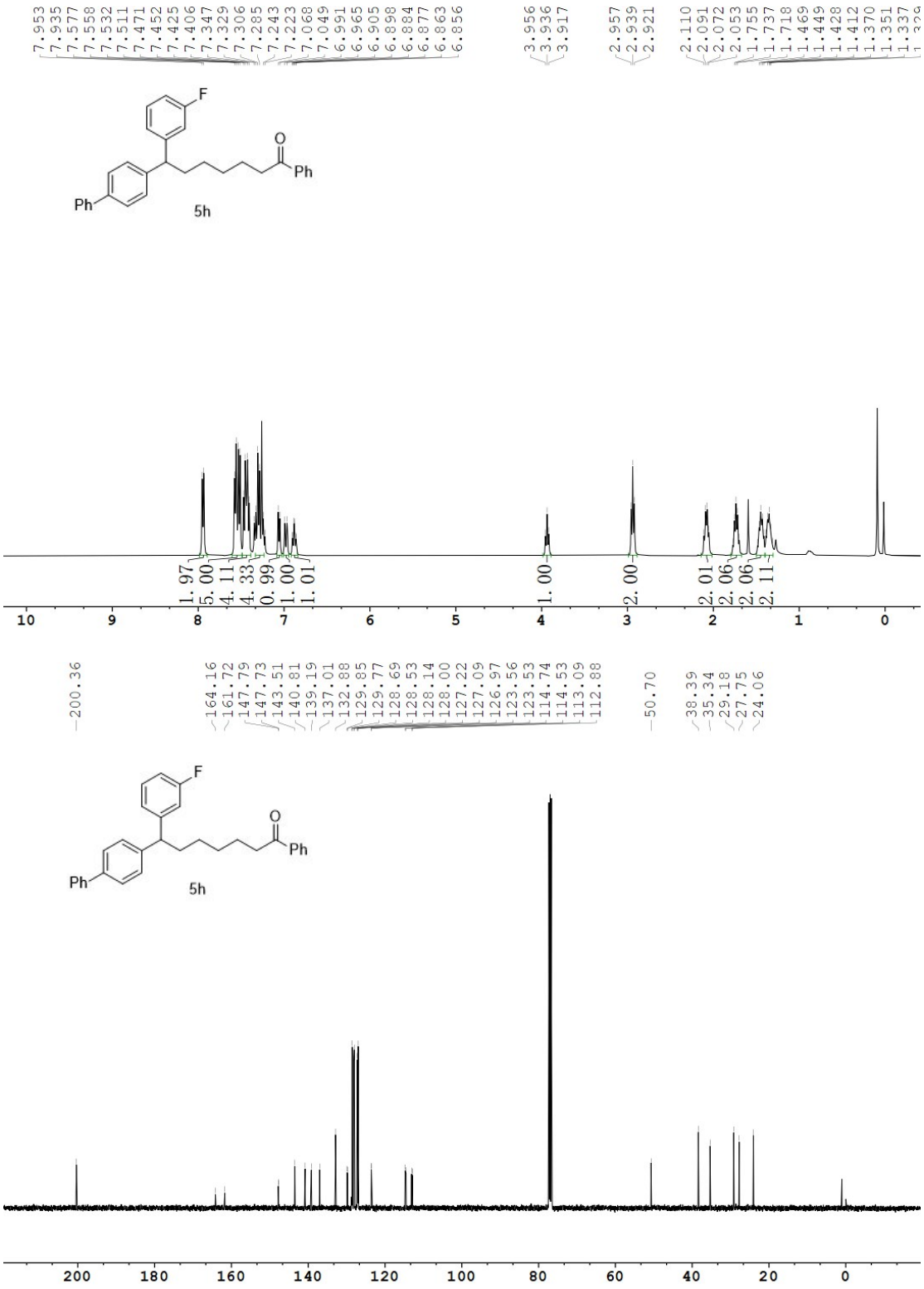
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5f



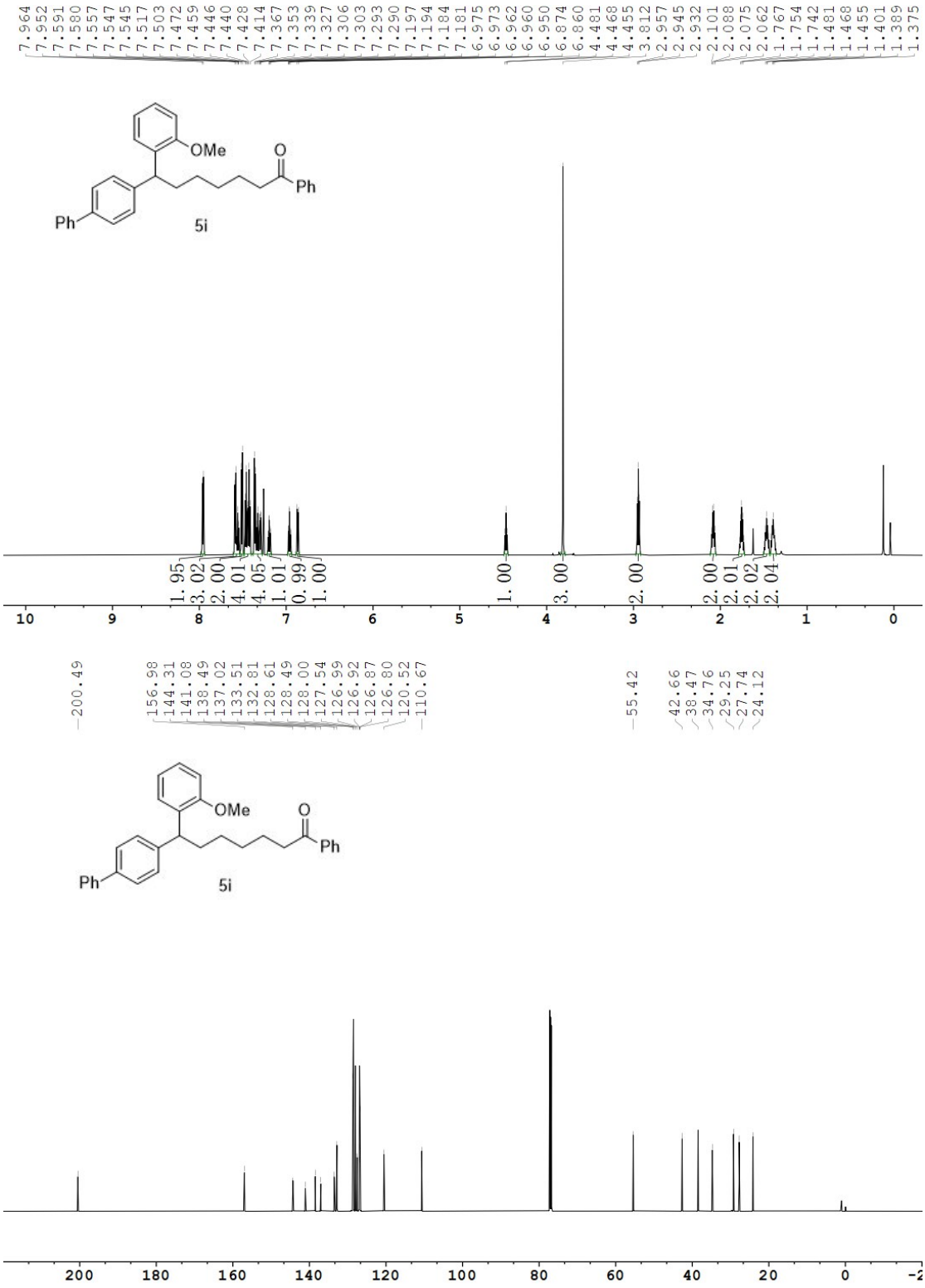
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5g



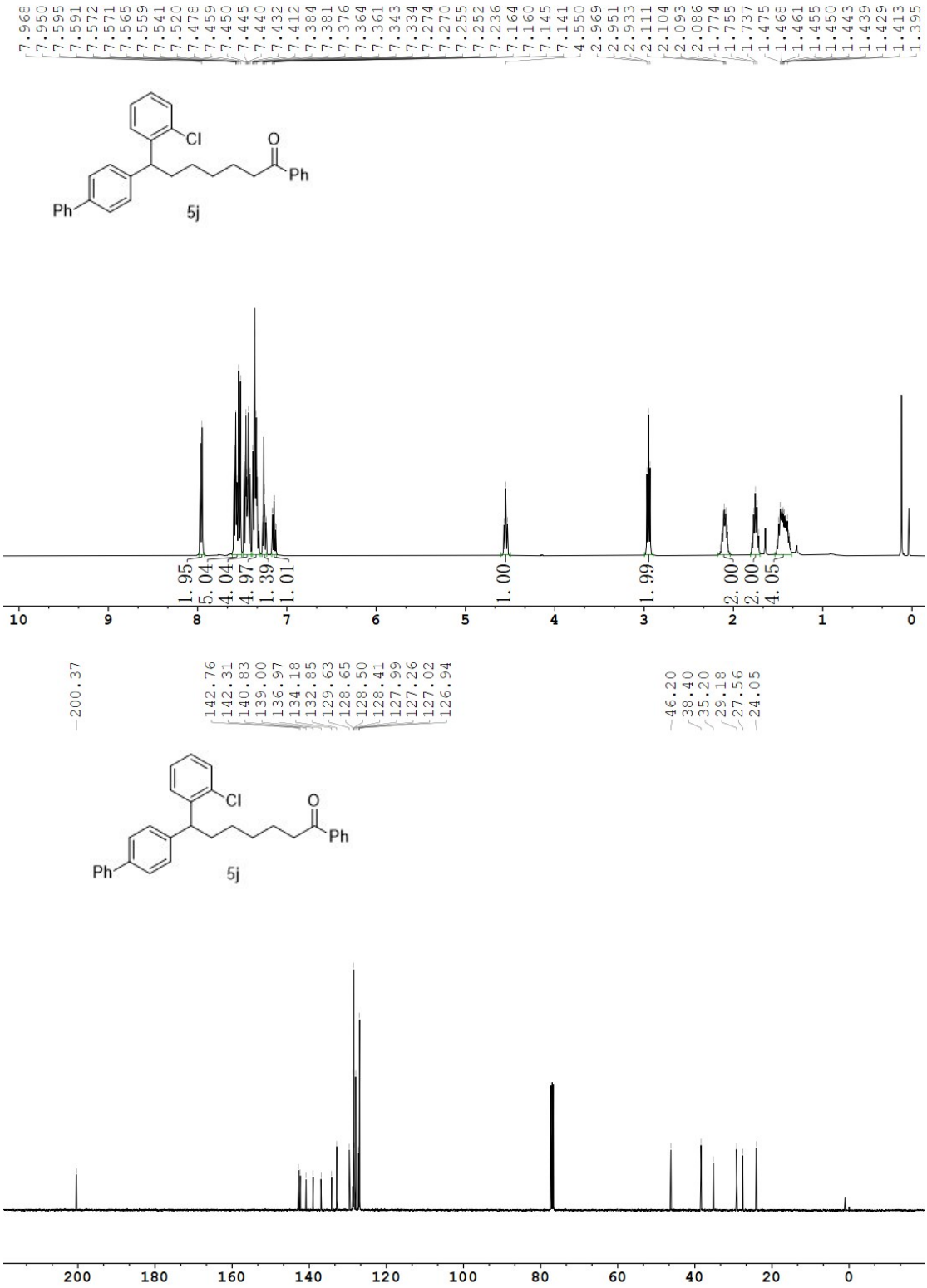
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5h



^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5i

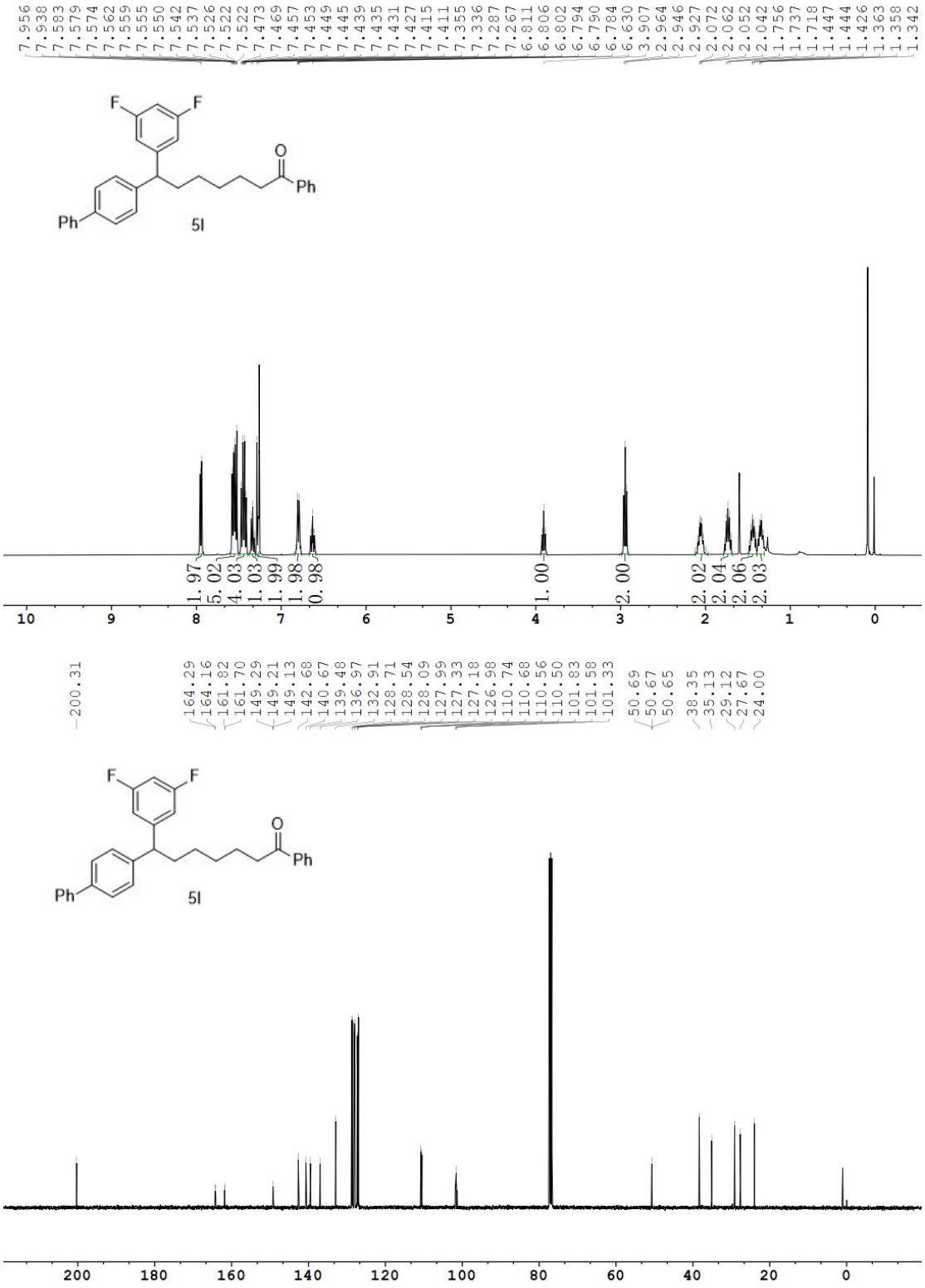


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5j

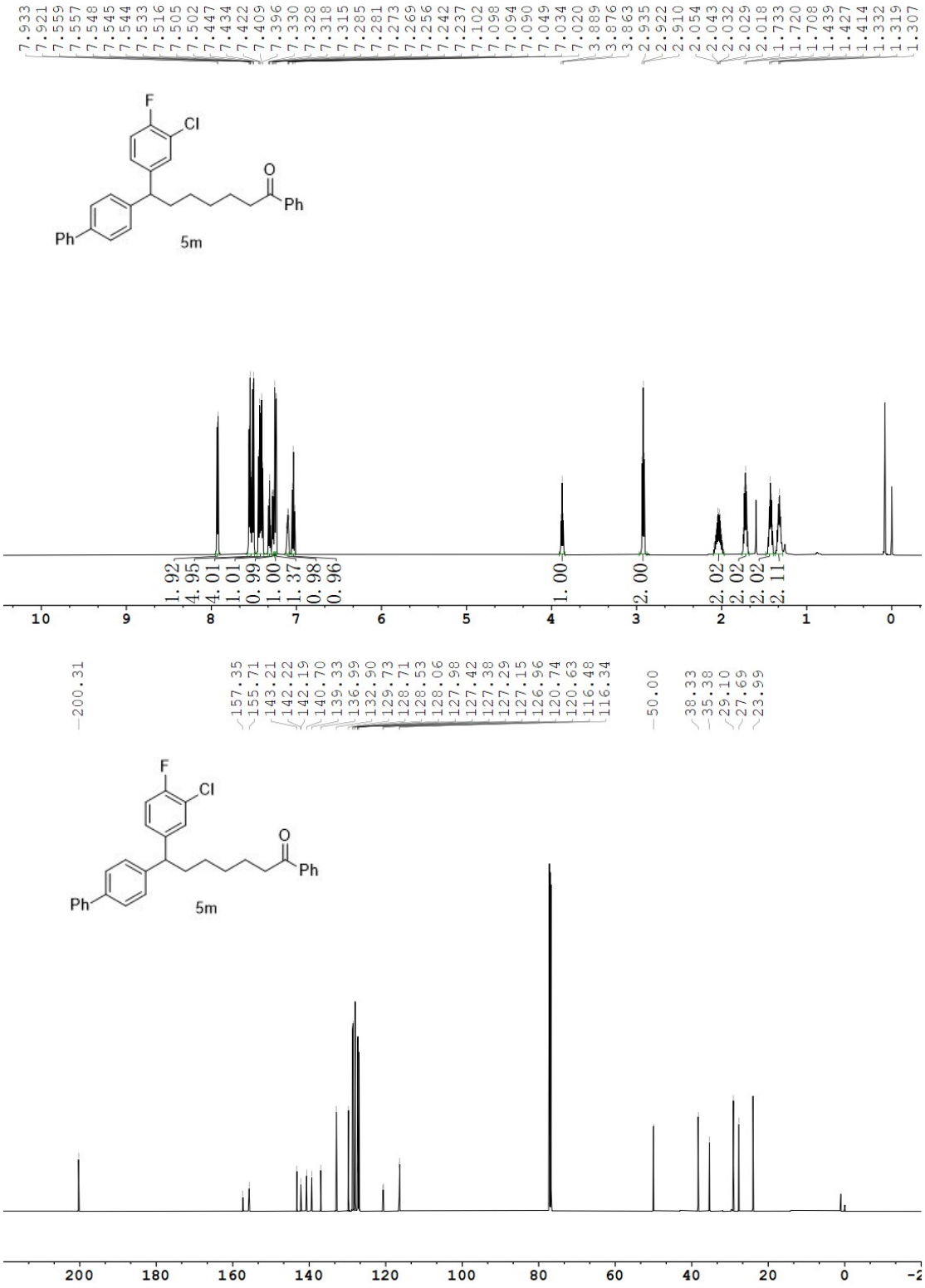


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5k

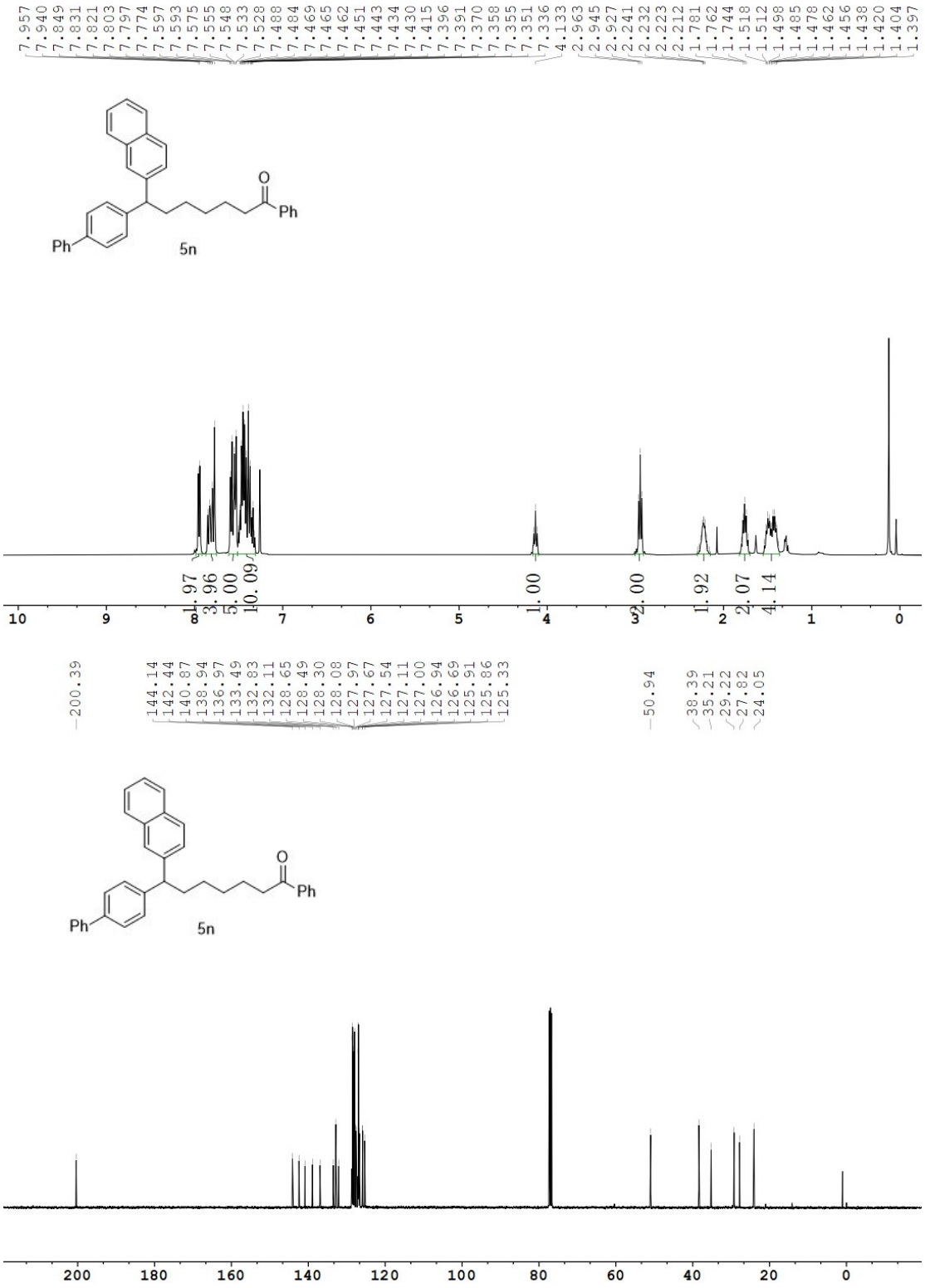
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5l



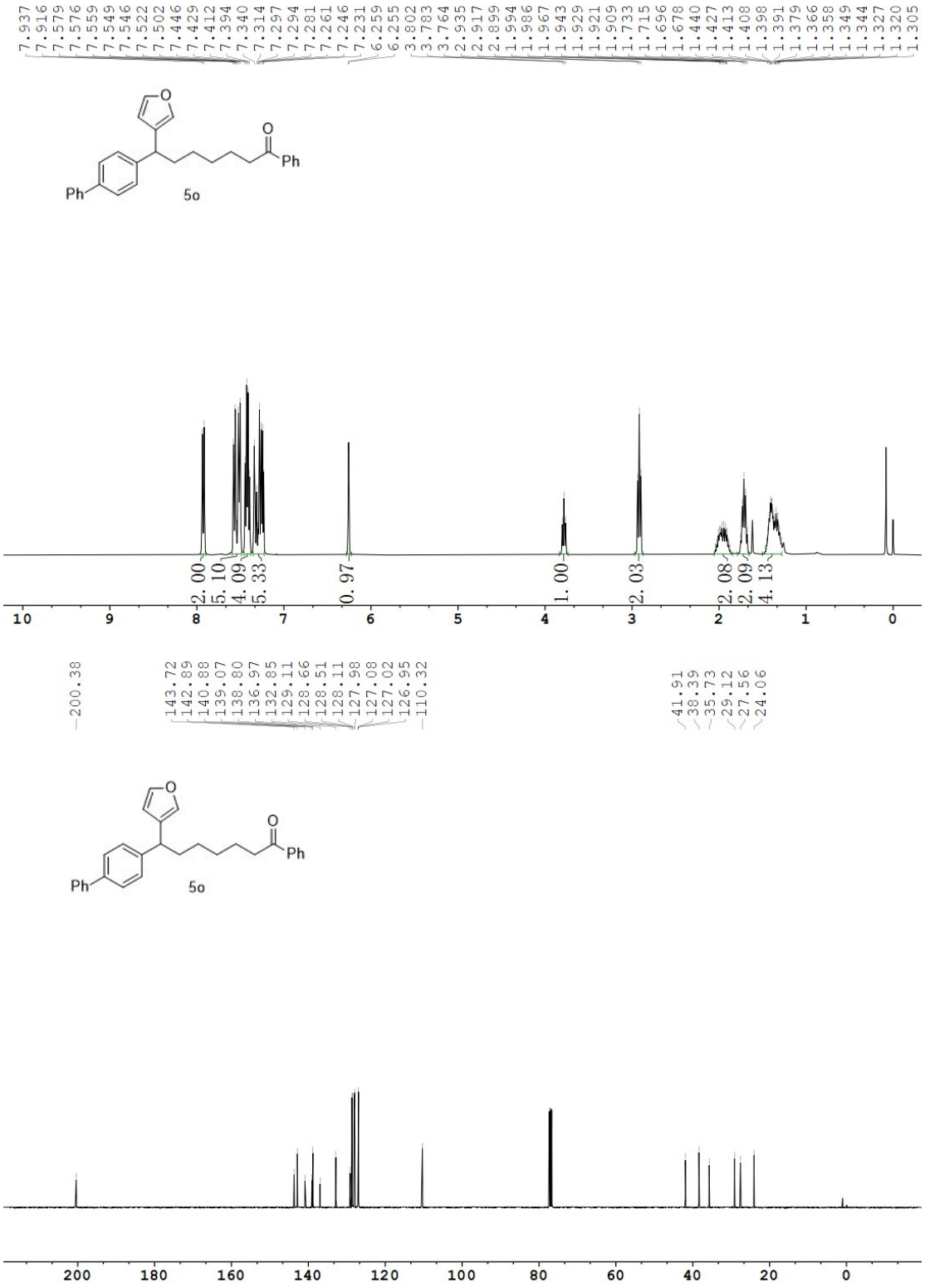
^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$) spectra of product 5m



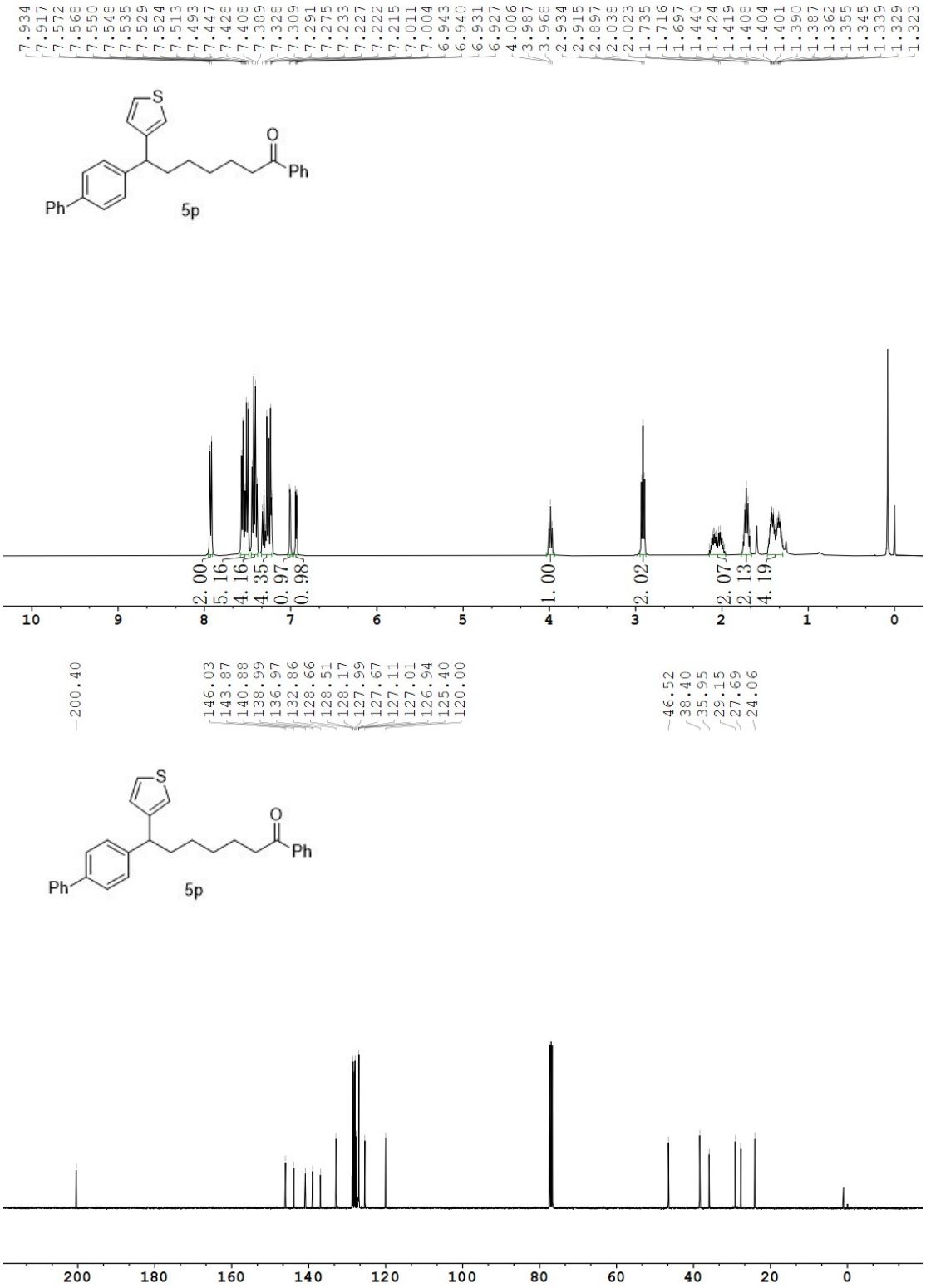
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5n



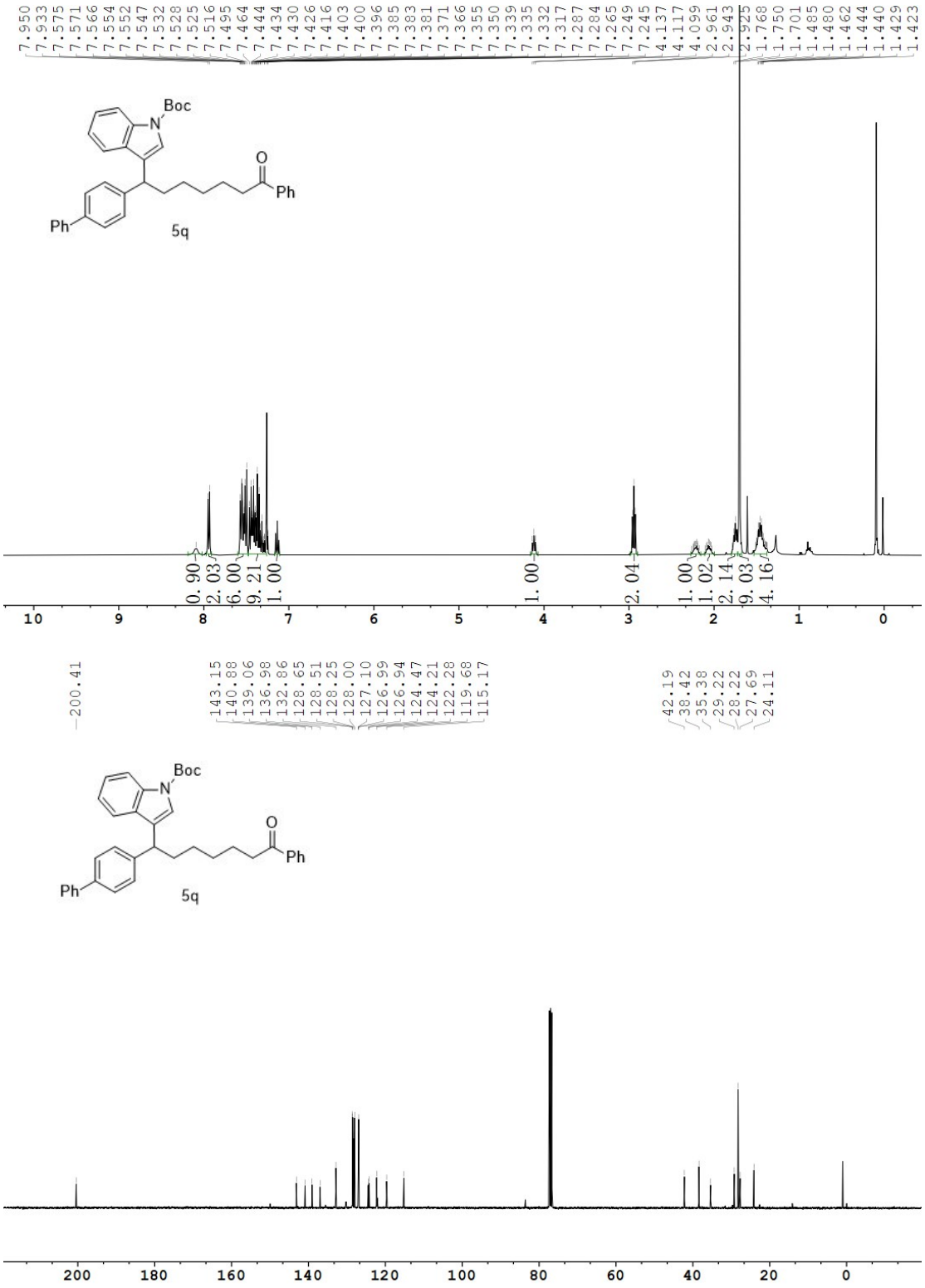
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5o



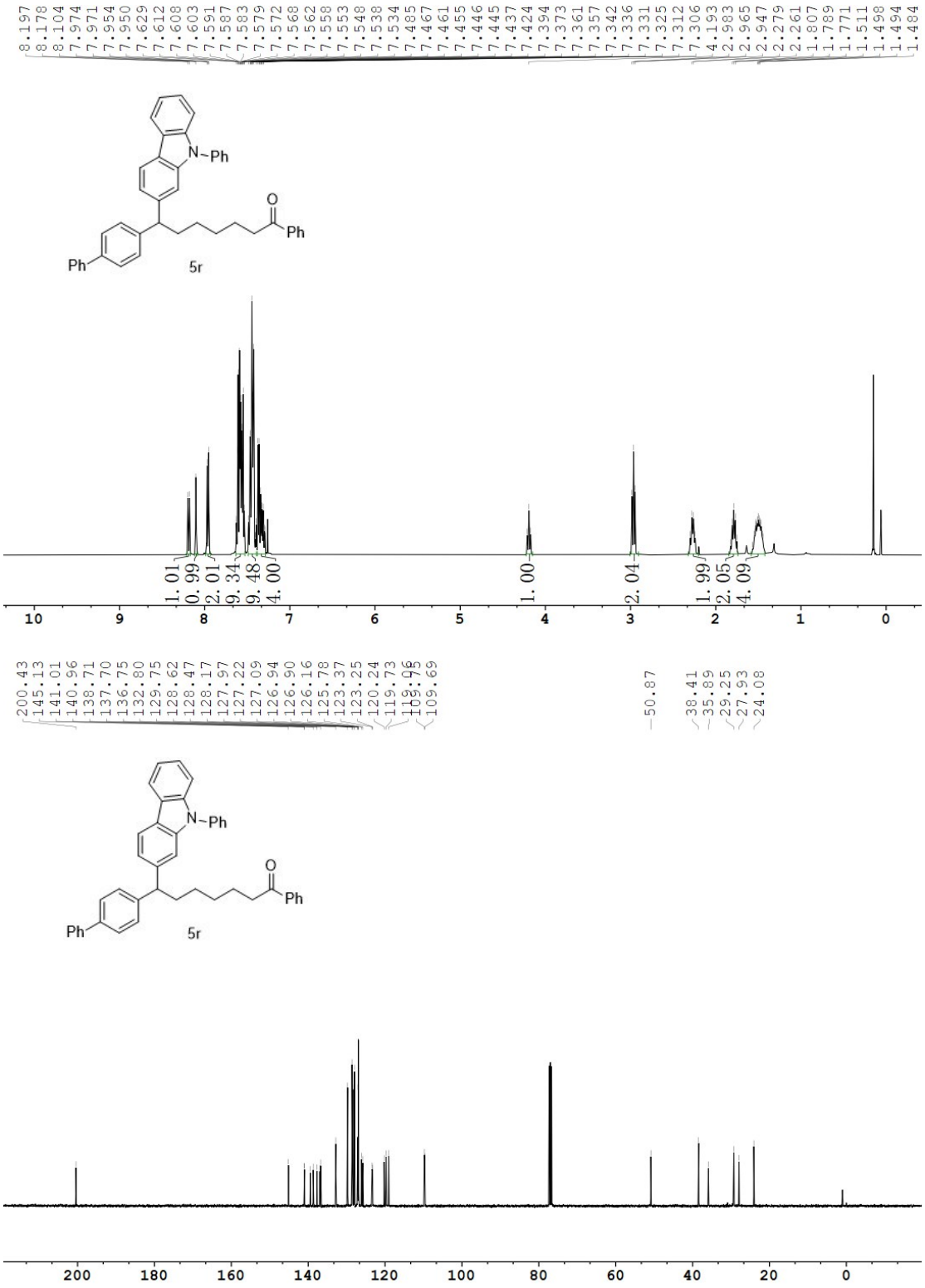
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5p



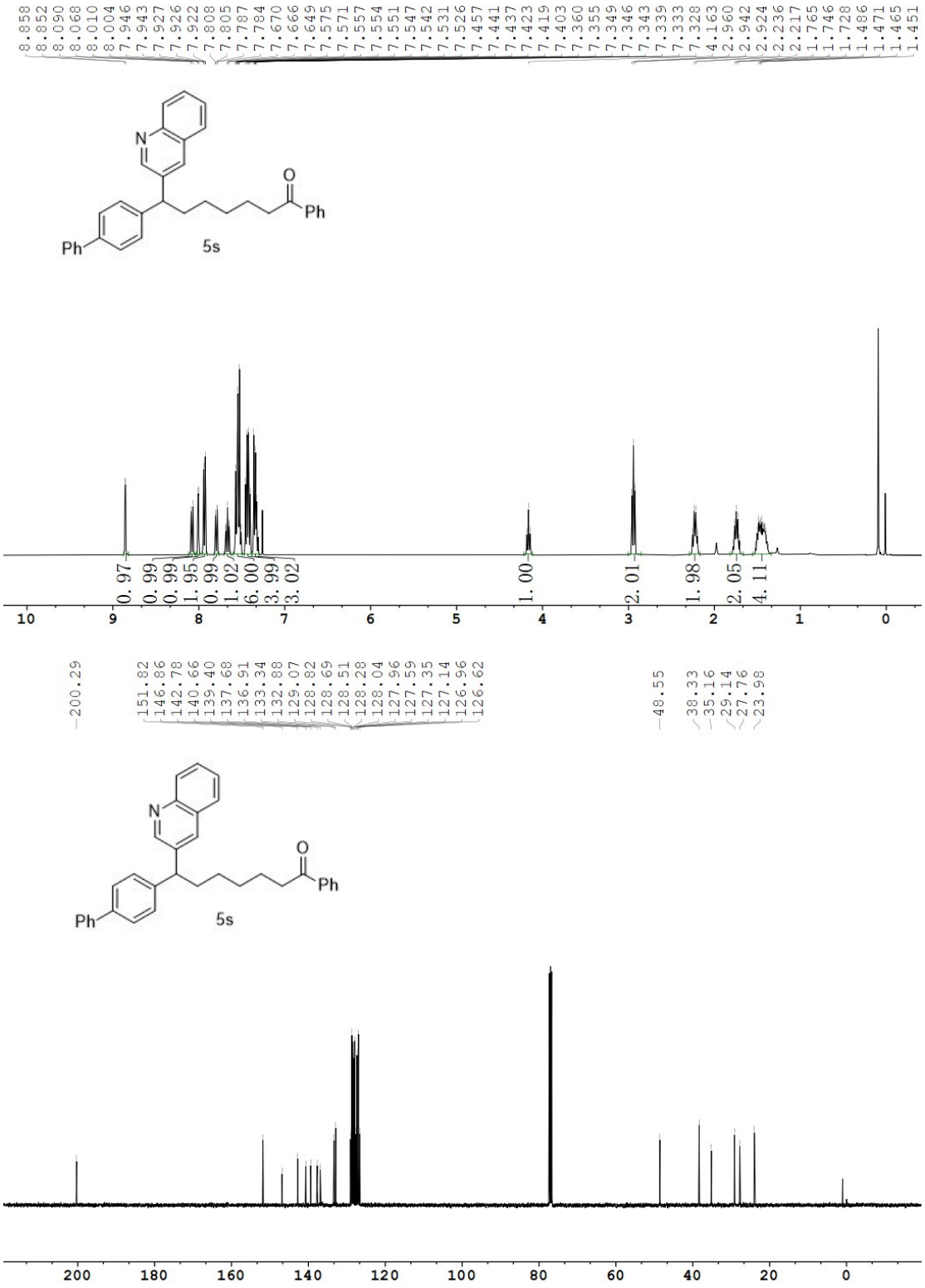
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5q



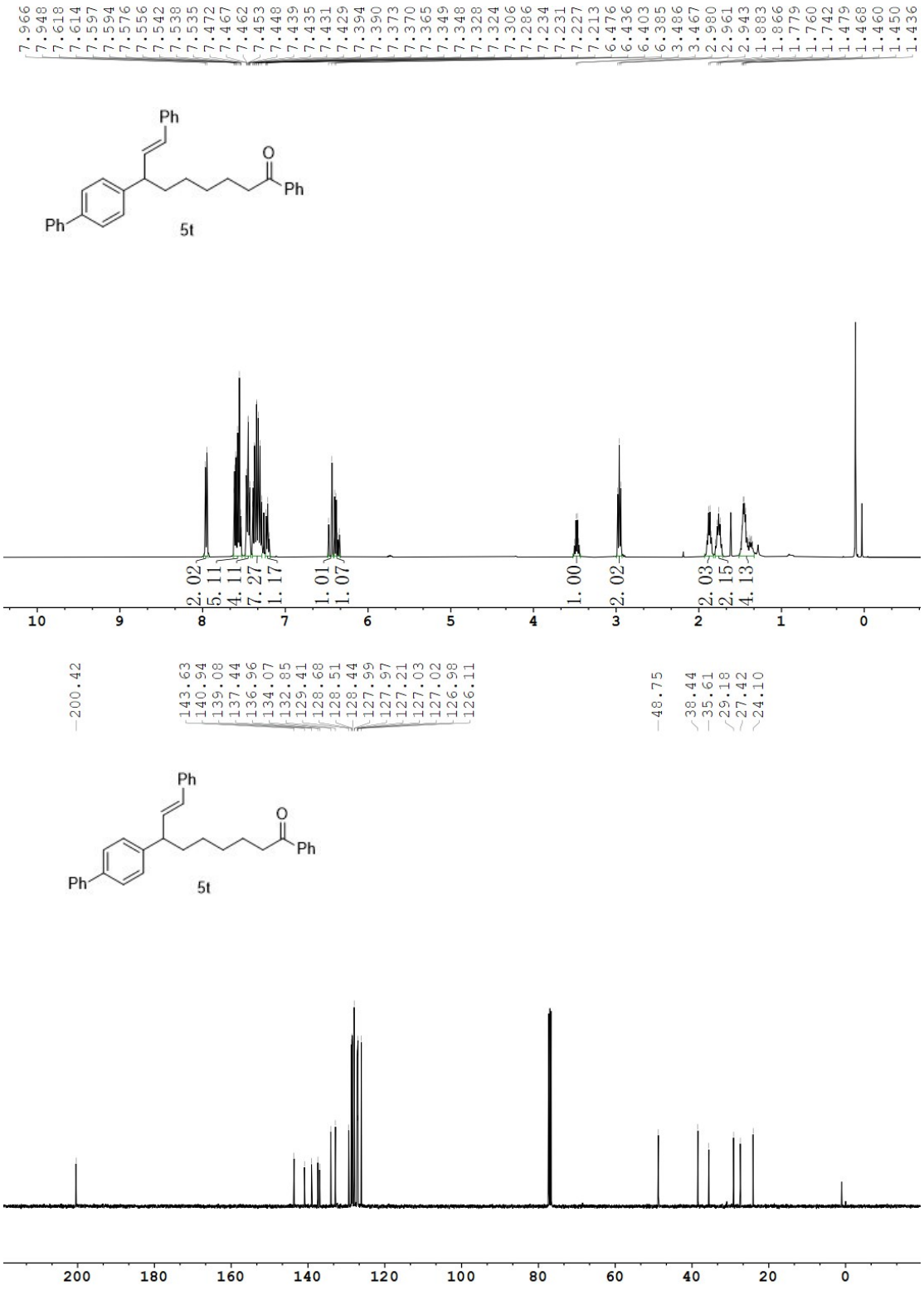
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5r



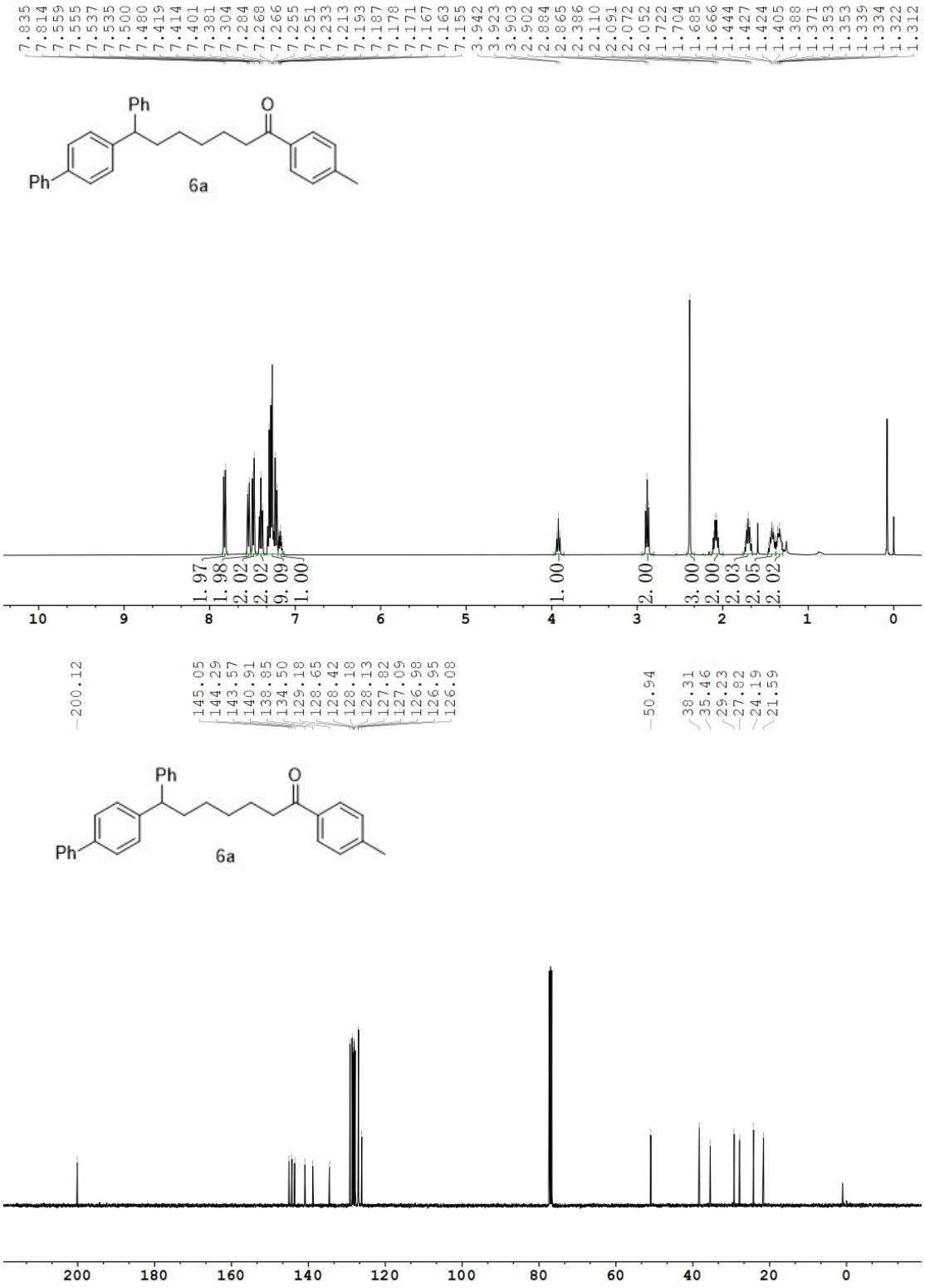
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5s



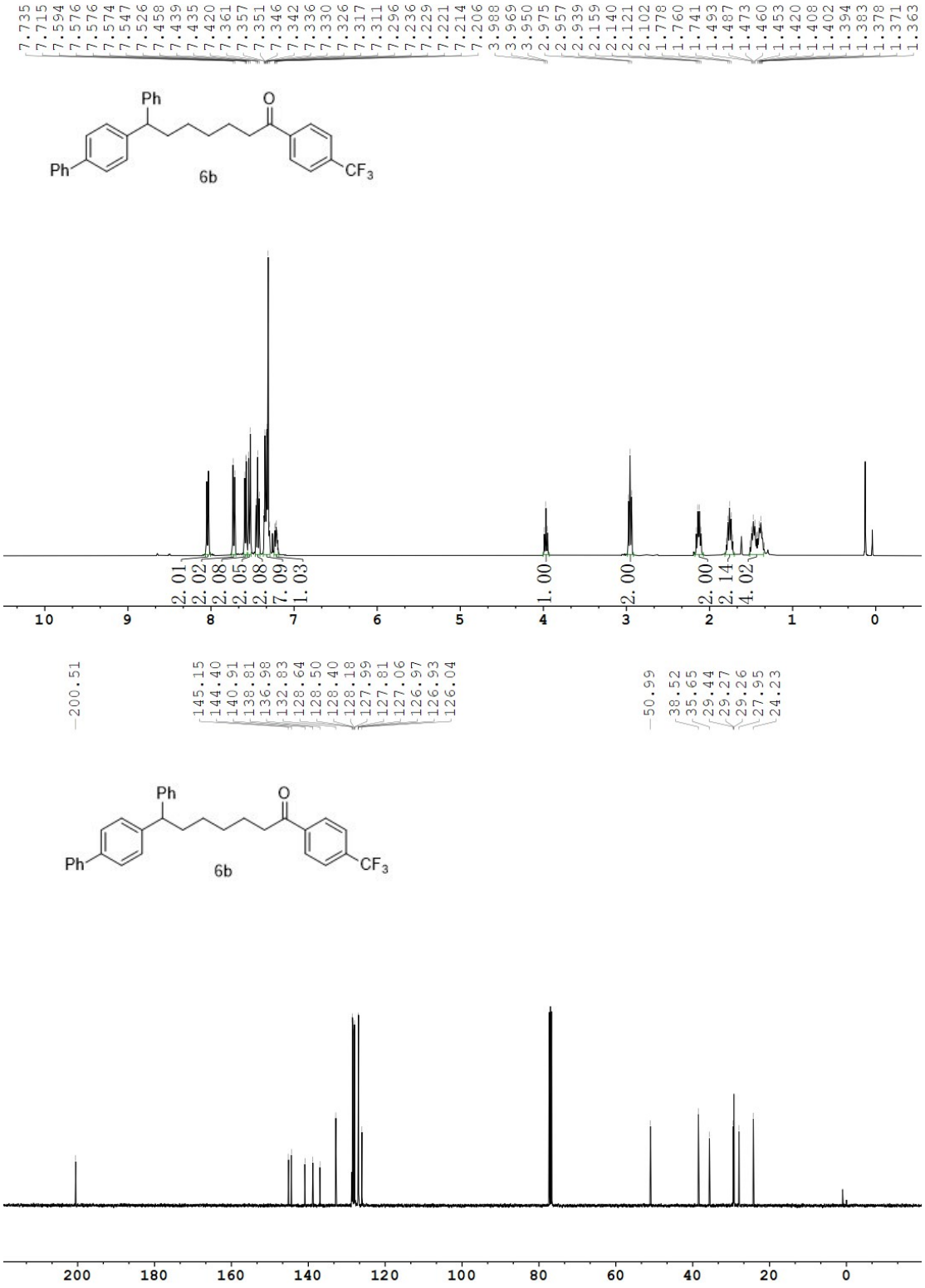
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 5t



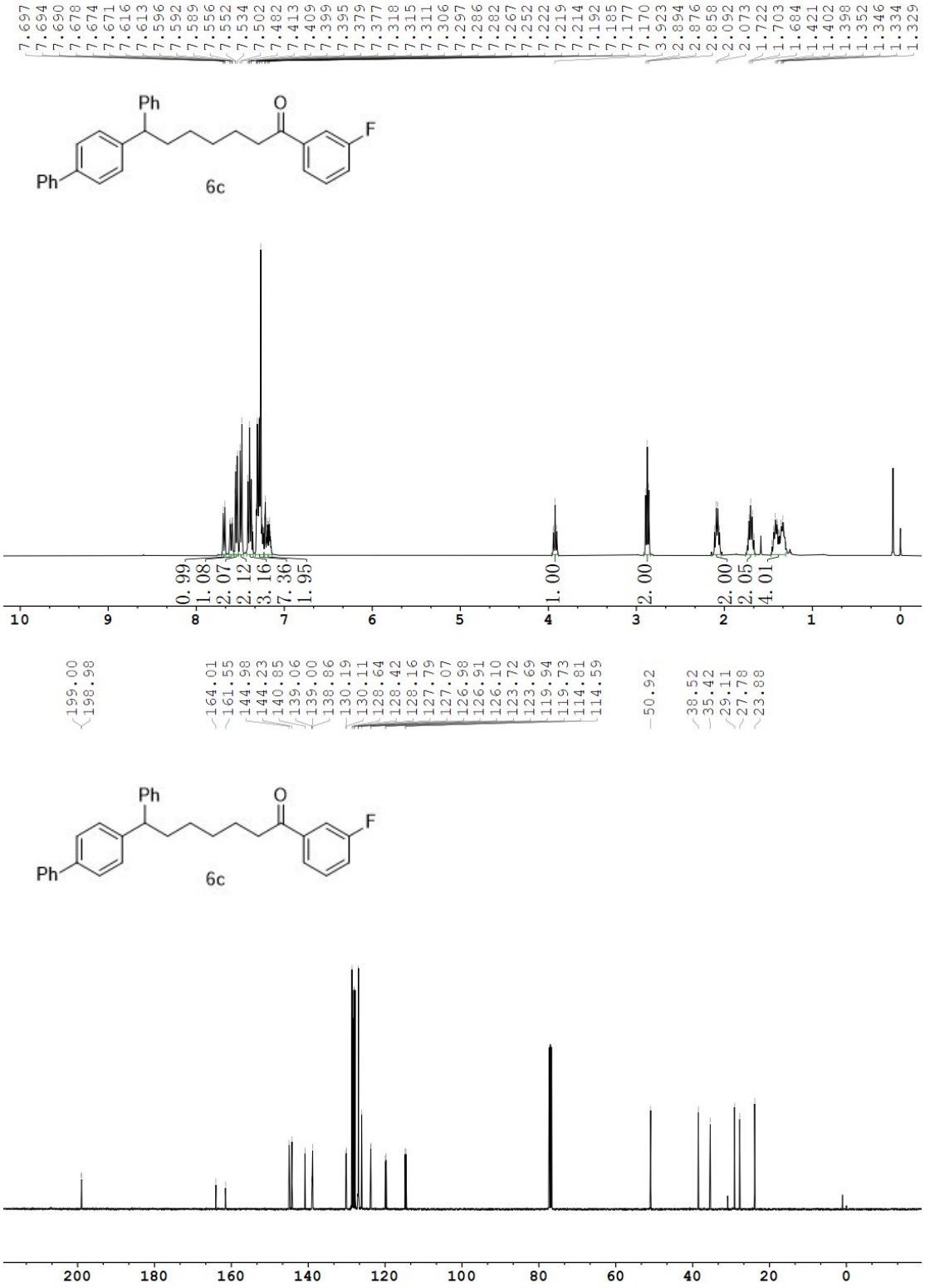
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6a



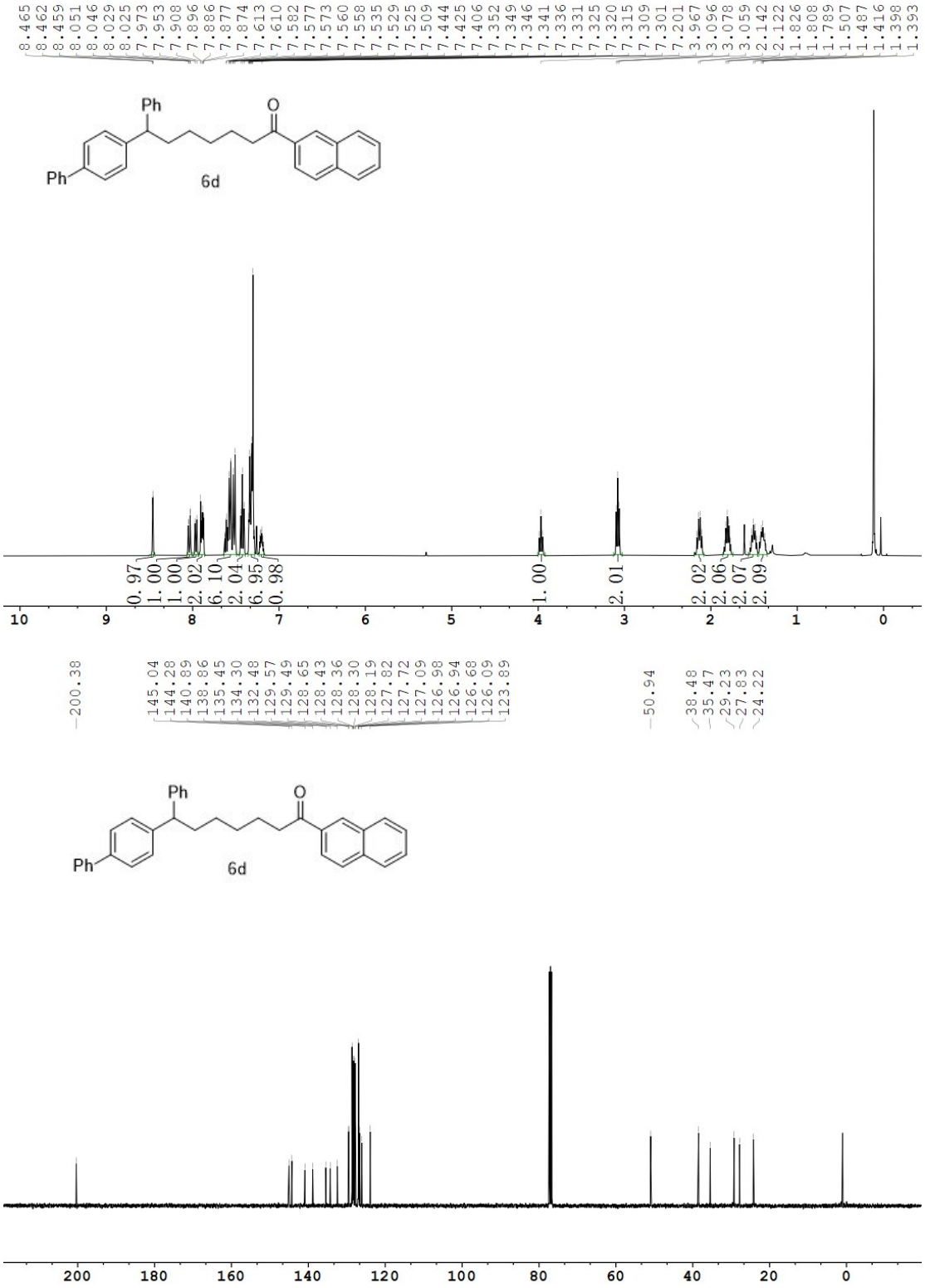
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6b



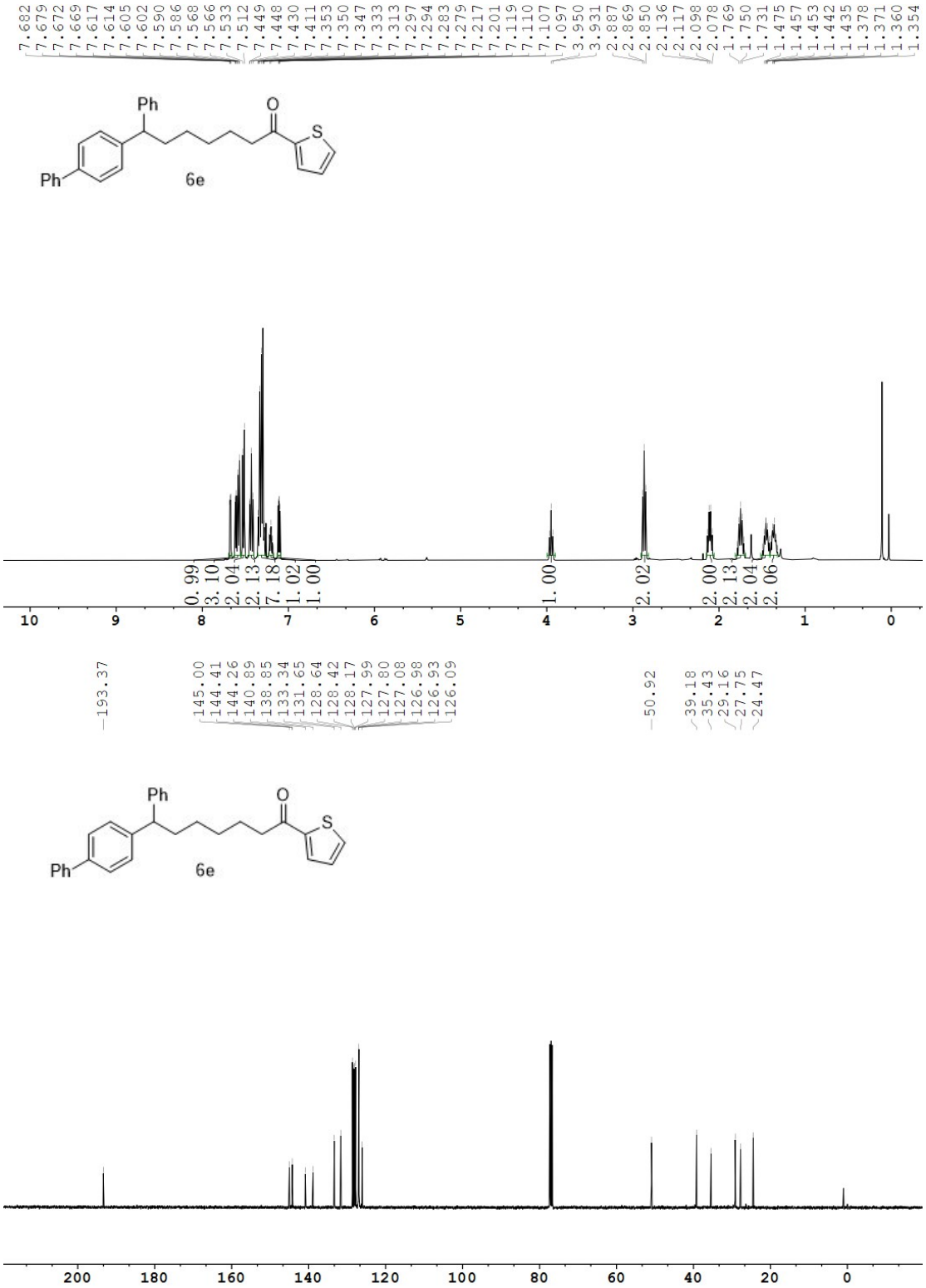
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6c



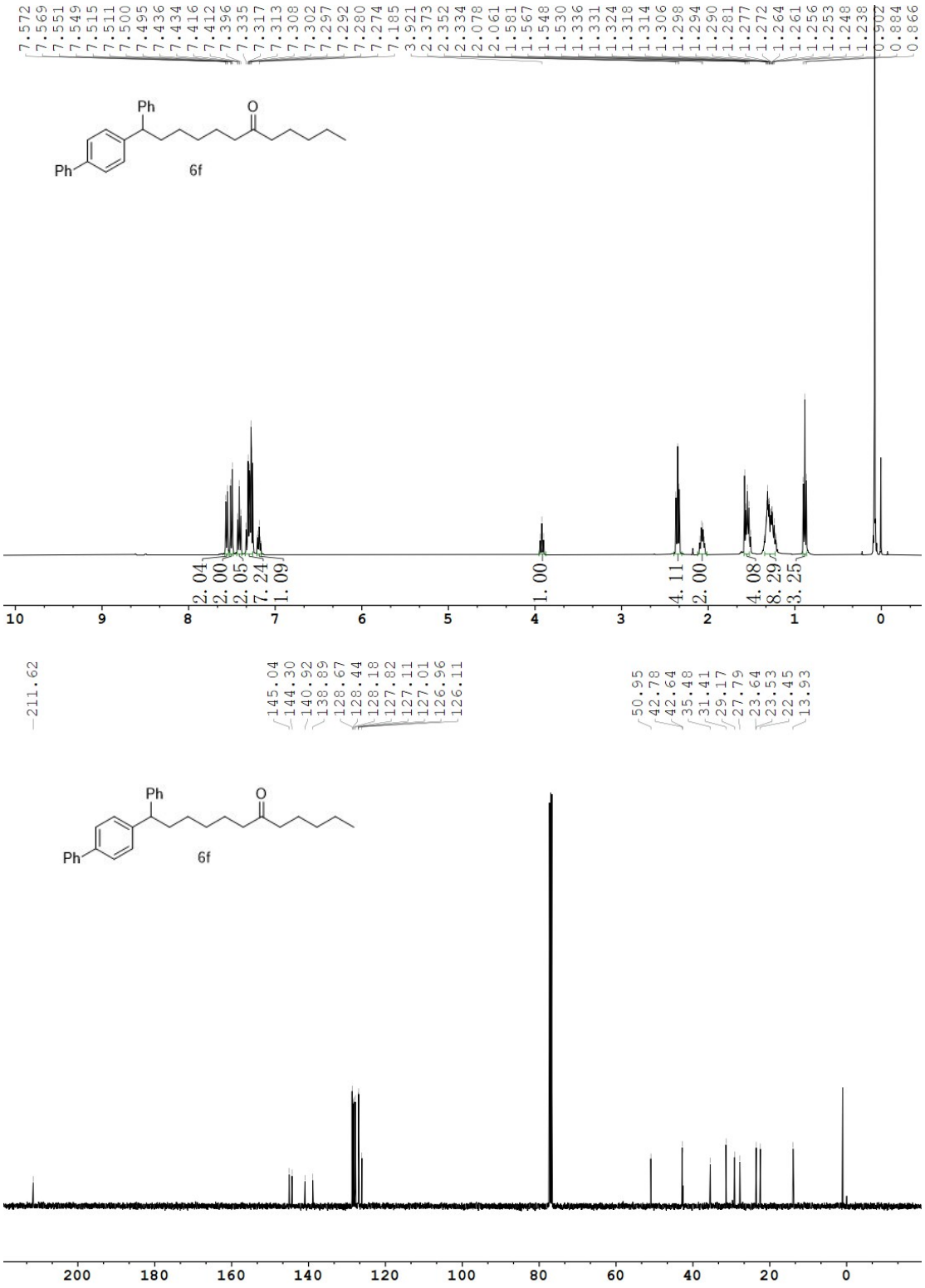
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6d



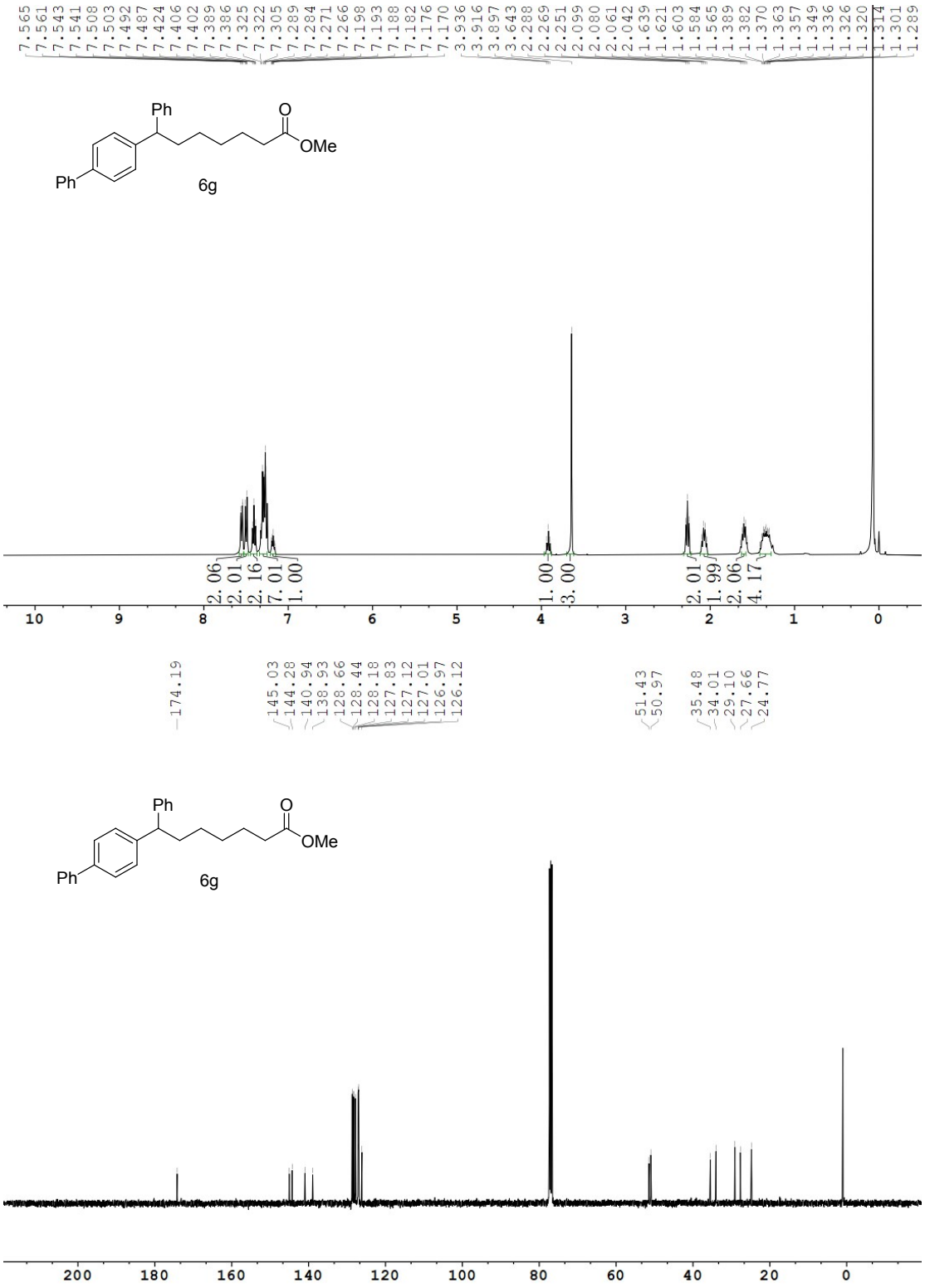
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6e



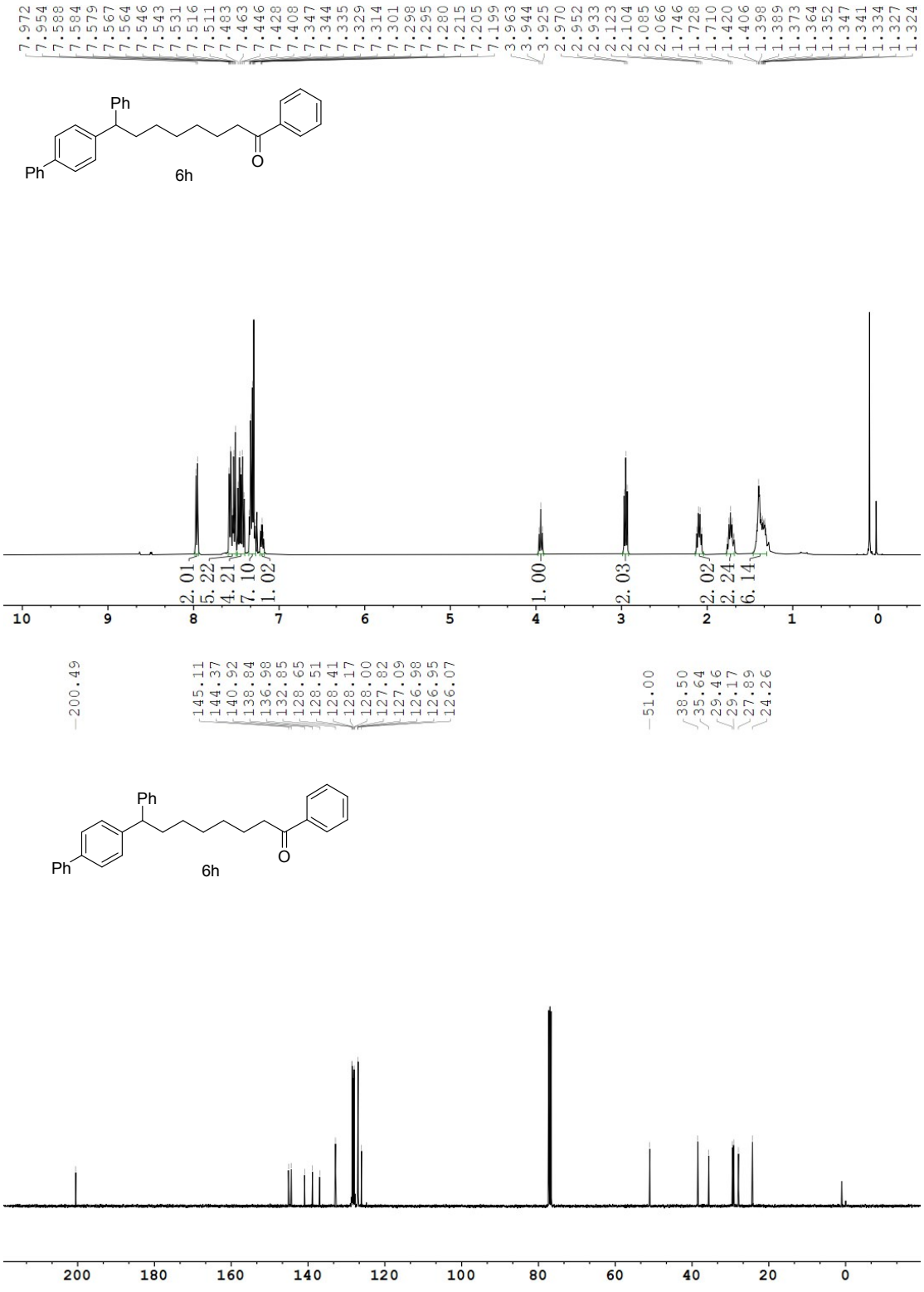
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6f



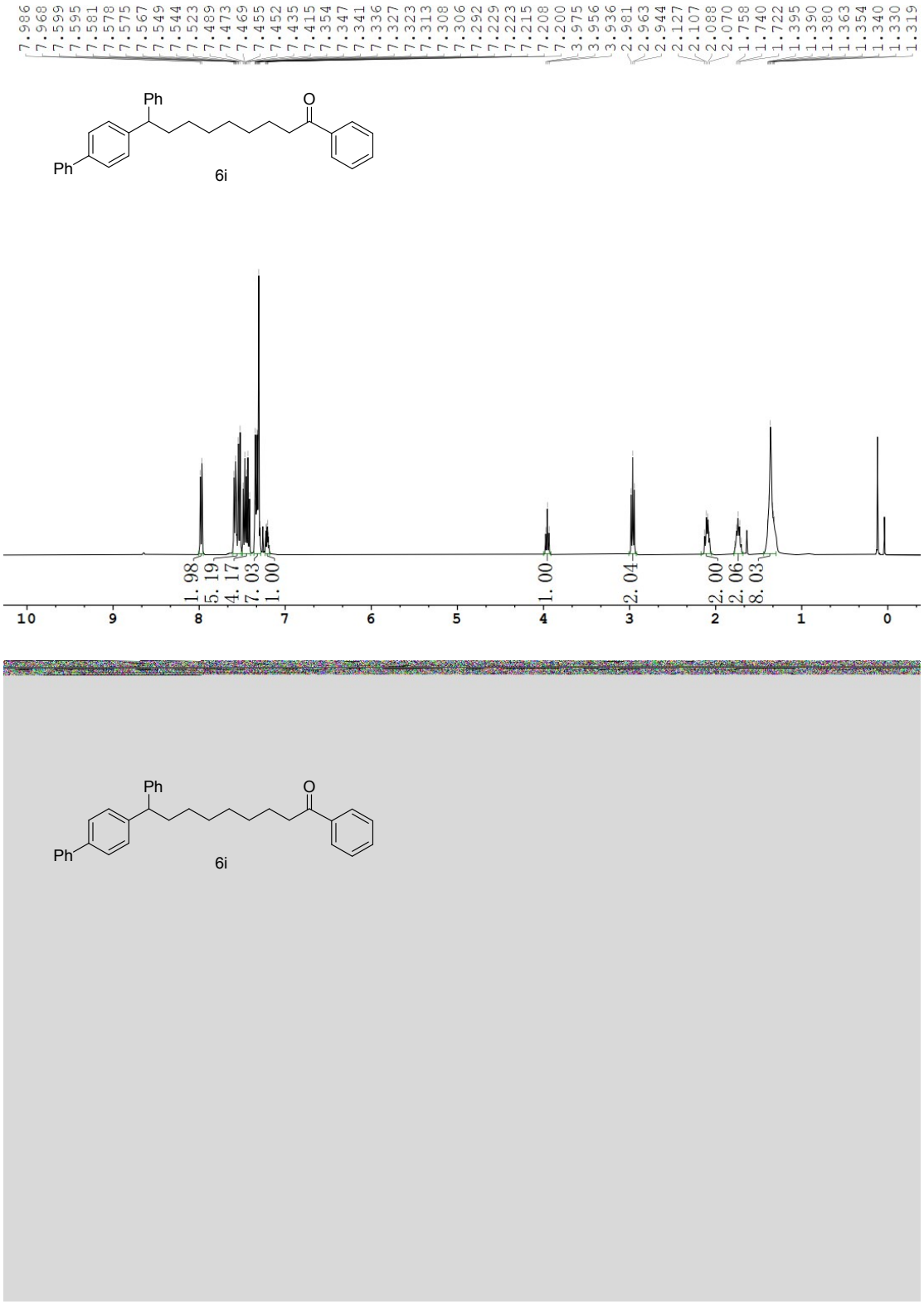
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6g



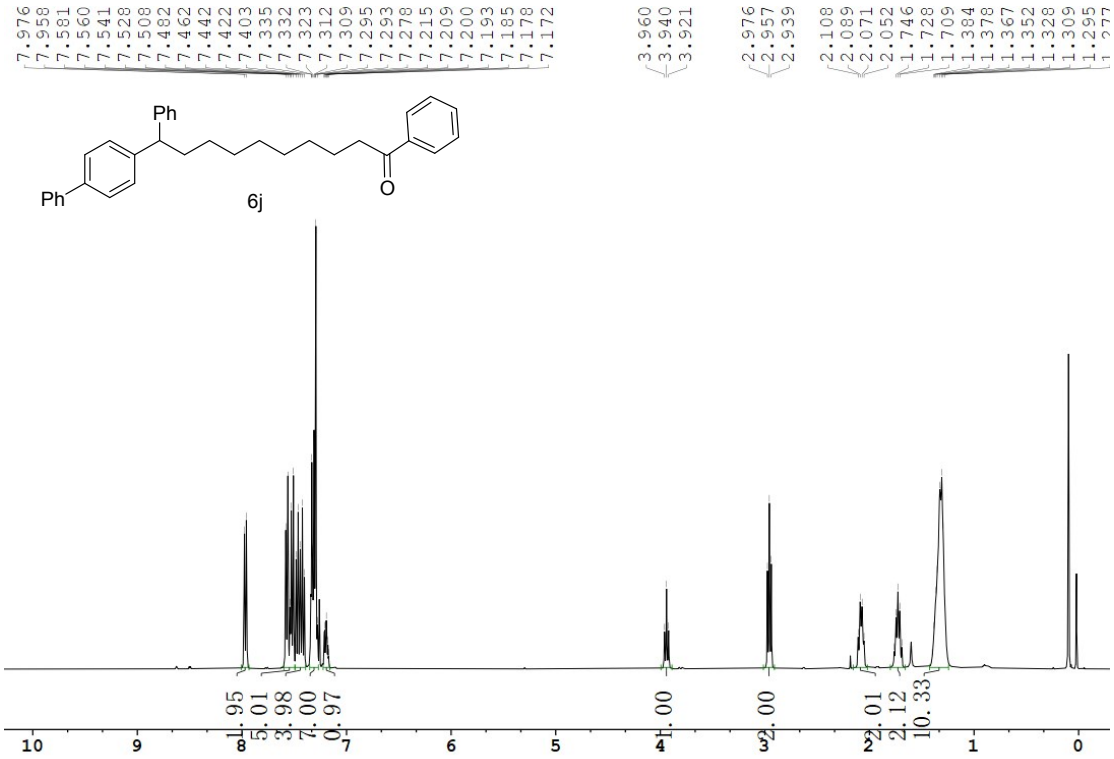
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6h

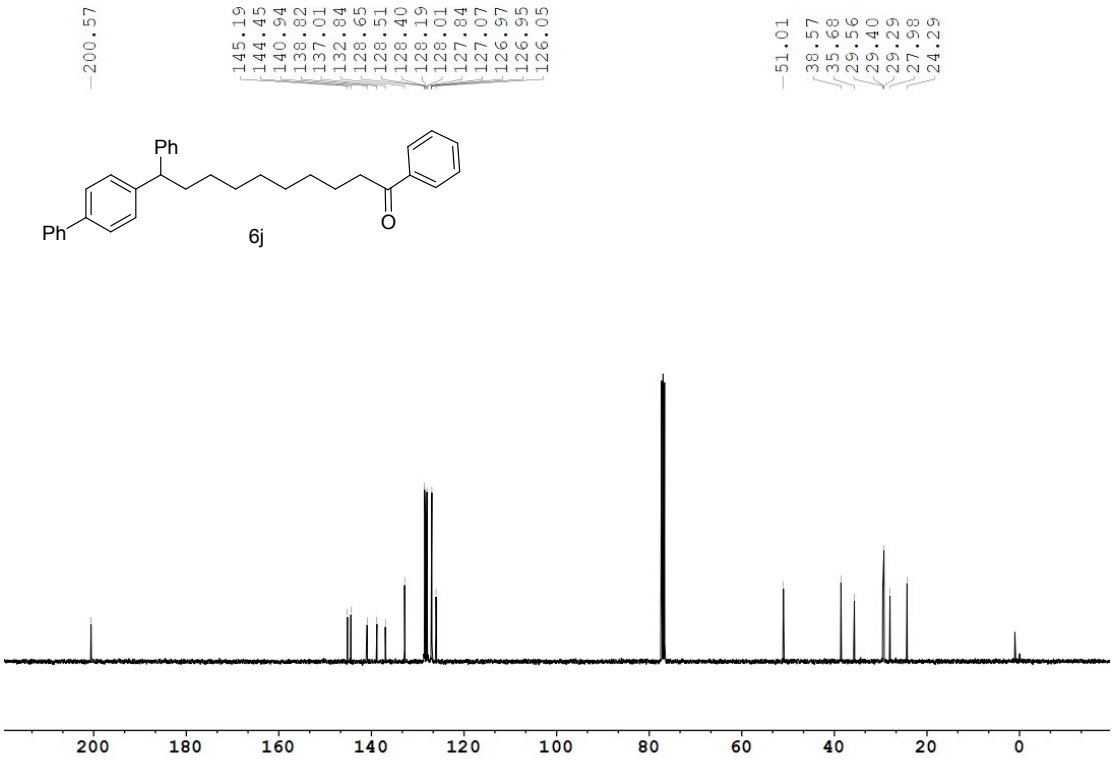


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6i

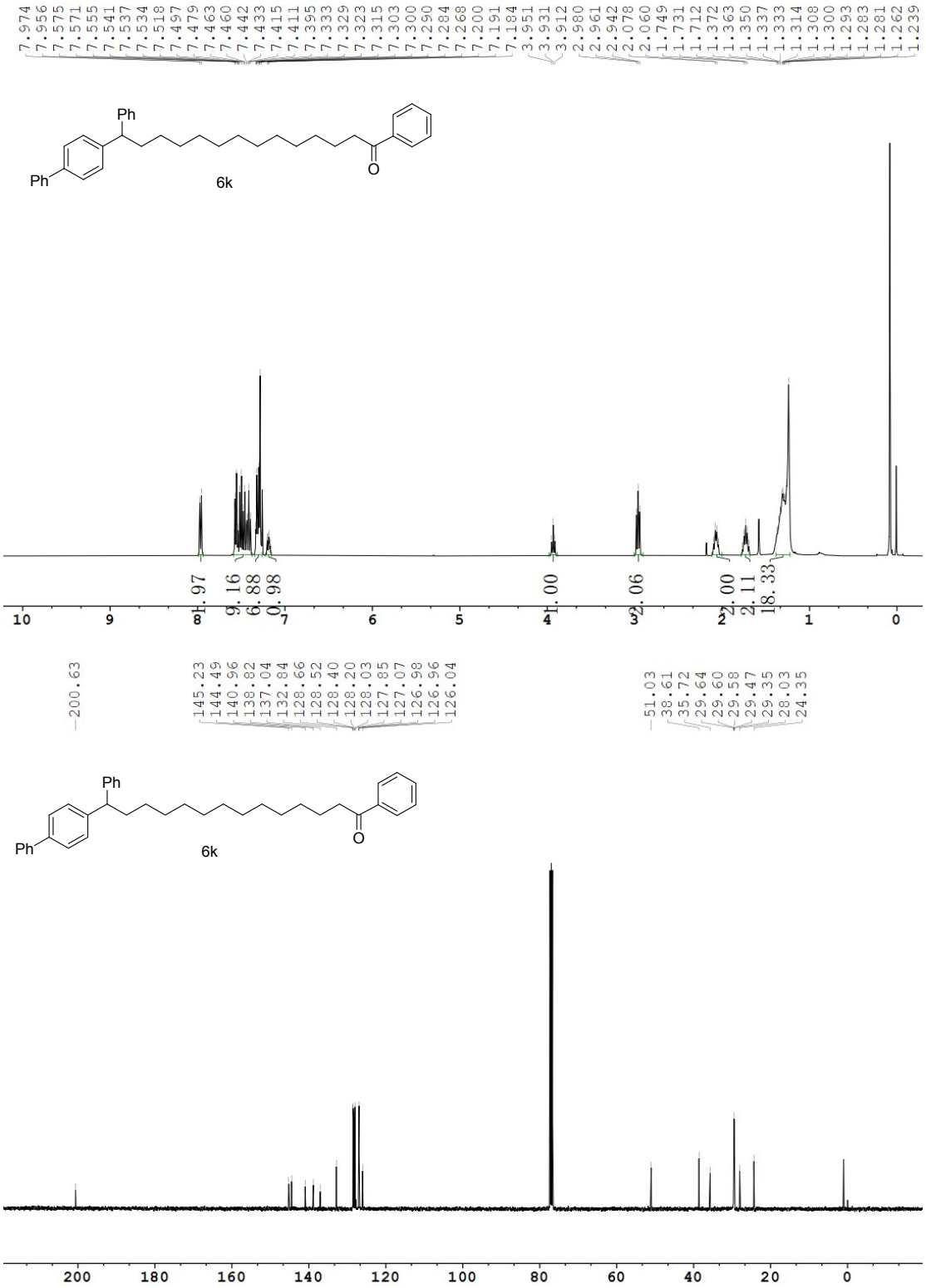


^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6j

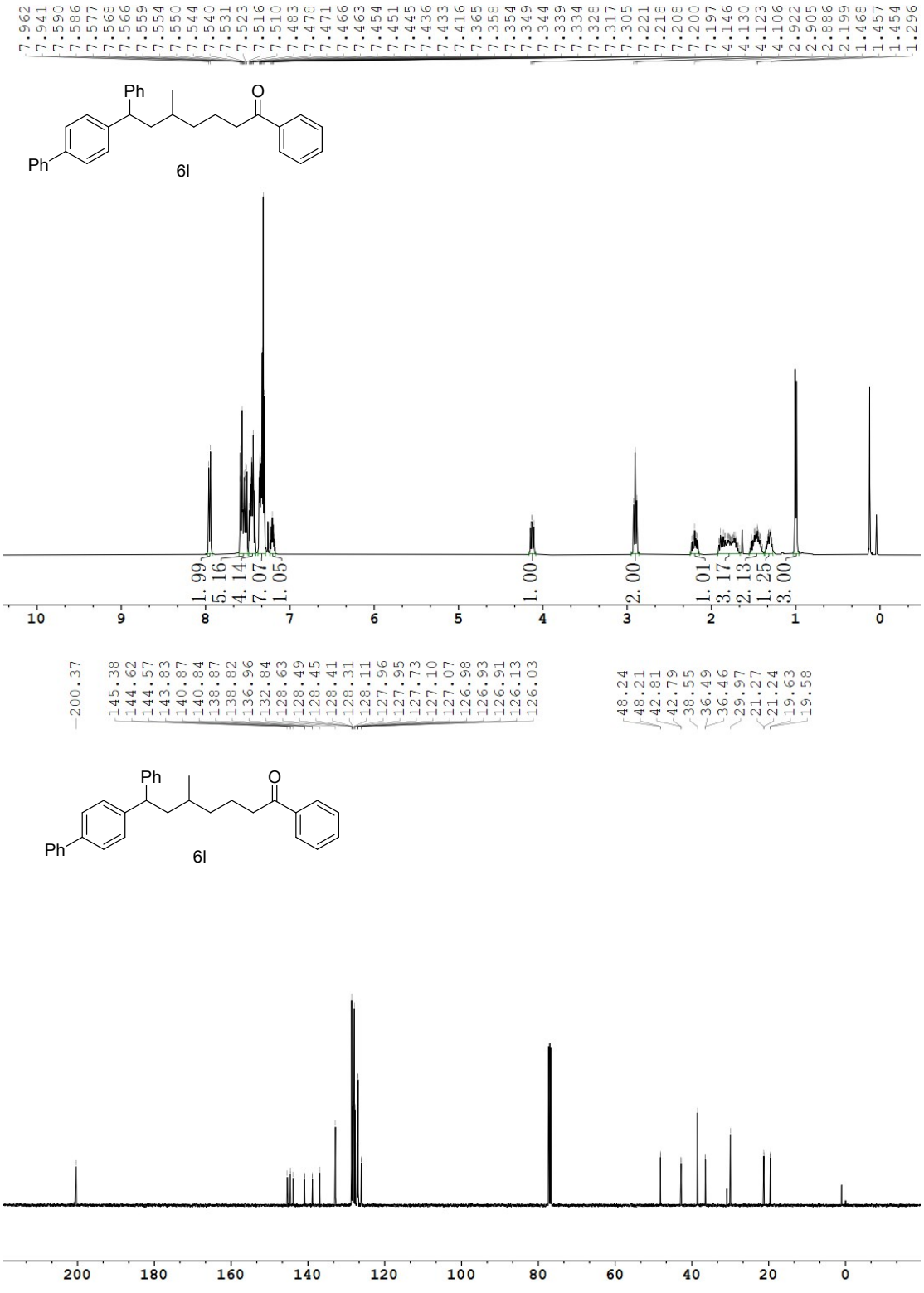




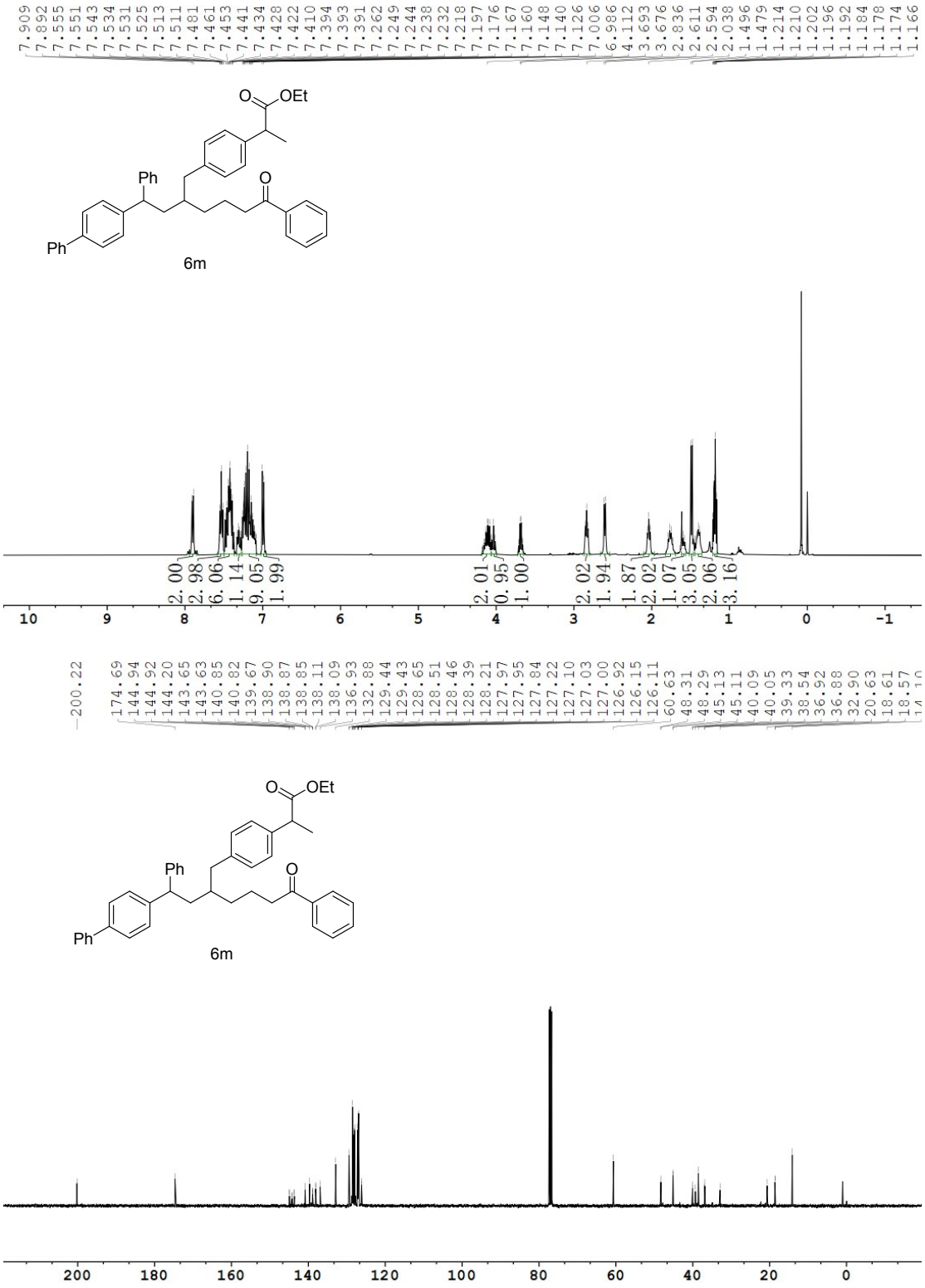
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6k



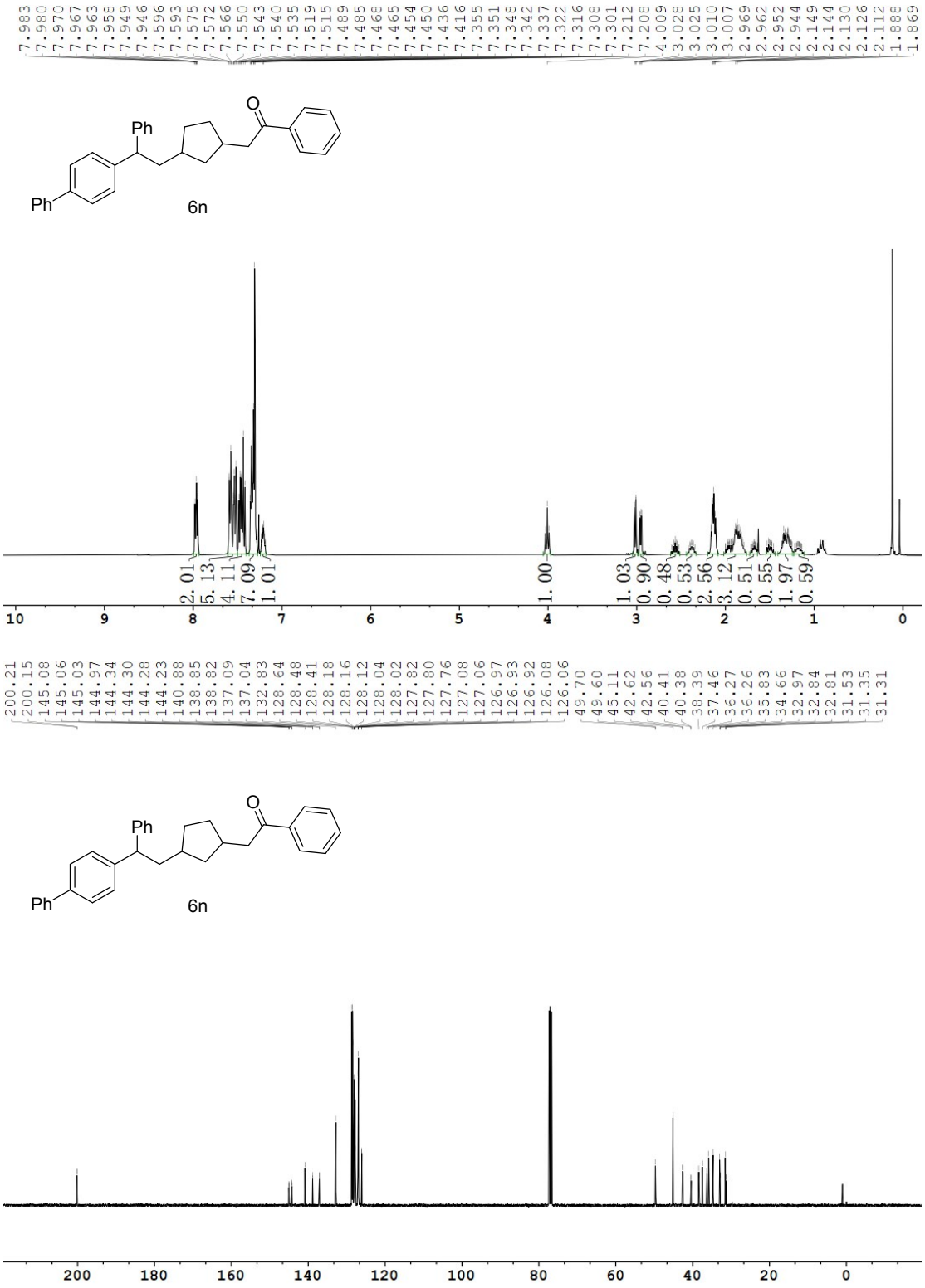
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6l



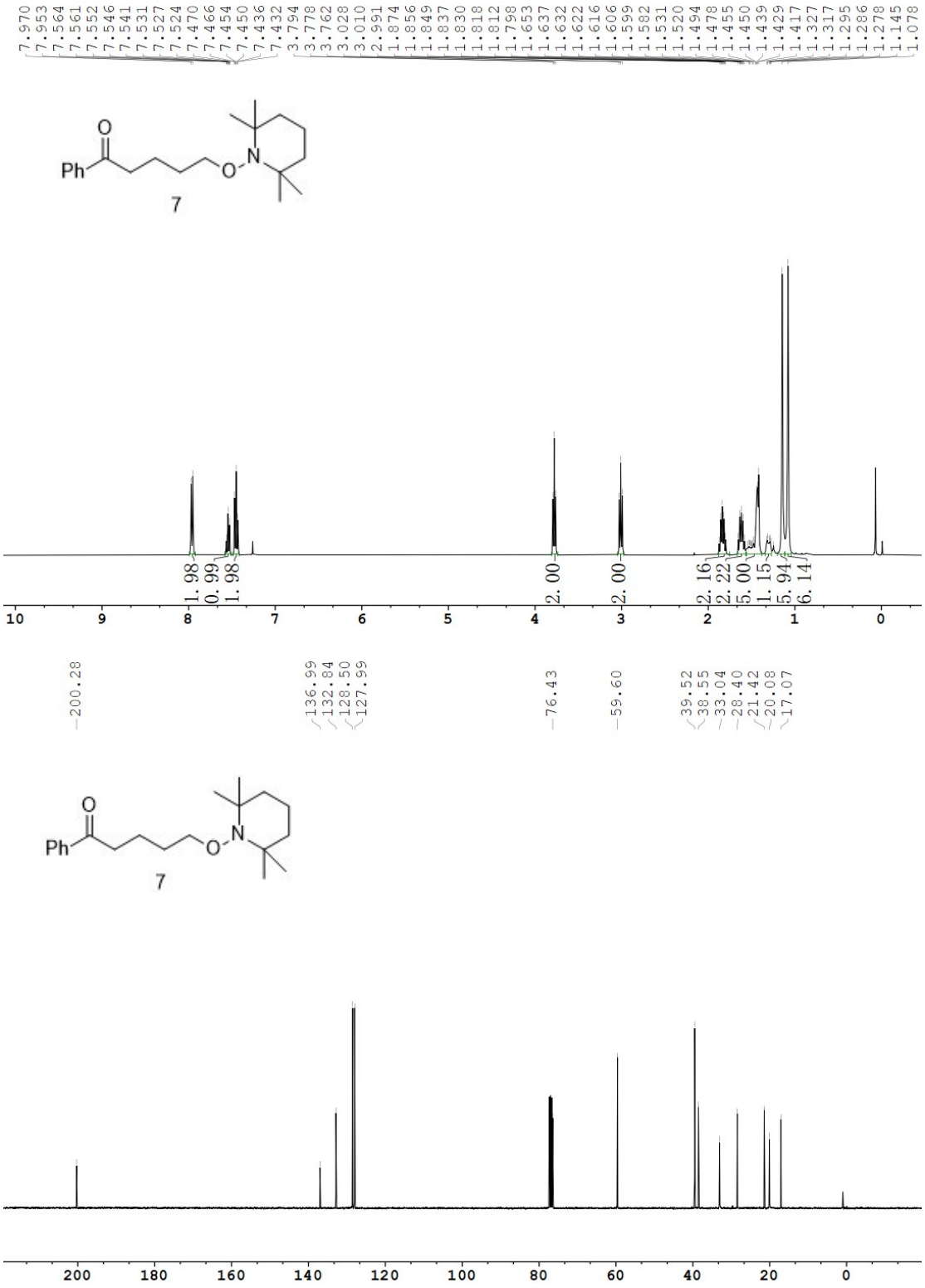
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6m



^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 6n



^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 7



^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$) spectra of product 9