Supporting Information

Reductive Coupling of Benzyl Oxalates with Highly Functionalized Alkyl Bromides by Nickel Catalysis

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1. General Information

Reagents and solvents:

Unless otherwise noted, all chemicals used in the preparation of starting materials were commercially available and were used as received without further purifications. All nickel catalysts, reductants, ligands were purchased from *Acros*, *Alfa Aesar*, *Aldrich*, *Ark Pharm*, and *Strem*. Other chemicals were purchased from *TCI*, *Adamas*, and *Energy chemicals*, and were directly used without further purifications.

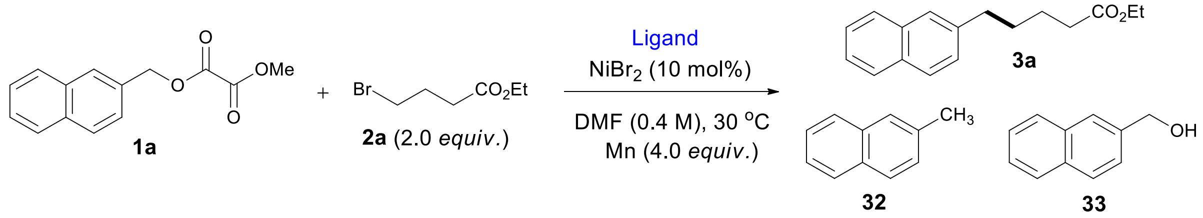
Anhydrous *N*,*N*-dimethylformamide (DMF), *N*,*N*-dimethylacetamide (DMA), Tetrahydrofuran (THF) and Acetonitrile (CH_3CN) were purified using a solvent-purification system that contained activated alumina and molecular sieves. Other solvents were dried and purified according to the procedure from ‘‘Purification of Laboratory Chemicals’’.[1]

Analytical methods:

^{1}H and ^{13}C NMR spectra were collected on a Bruker AVANCE III 400MHz spectrometer at room temperature. All ^{1}H NMR spectra are reported in parts per million (ppm) downfield of tetramethylsilane (TMS) and were referenced to the signal of TMS (0 ppm). All ^{13}C NMR spectra were reported in ppm relative to residual $CHCl_3$ (77 ppm). Coupling constants, *J*, are reported in hertz (Hz). ^{19}F NMR and ^{31}P NMR spectra were also collected on Bruker AVANCE III 400 MHz spectrometers at room temperature. Melting points were determined on a microscopic apparatus. IR spectra were collected with Bruker-TENSOR27 spectrometer and only major peaks were reported in cm^{-1}. HRMS was performed on Bruker Apex II FT-ICR mass instrument (ESI). GC analysis was performed on Thermo Scientific TRACE 1300. GC-MS data was collected on Thermo Scientific TRACE DSQ GC-MS. HPLC analysis was recorded on Thermo Scientific UltiMate 3000 equipped with Chiralpak IC column. Optical rotation was measured by the Perkin Elmer 341 polarimeter. Thin layer chromatography were carried out using XINNUO SGF254 TLC plates. Flash chromatography was performed with XINNUO silica gel (200-300 mesh). The yield reported in the manuscript refer to isolated yields and represent an average of at least two independent runs.

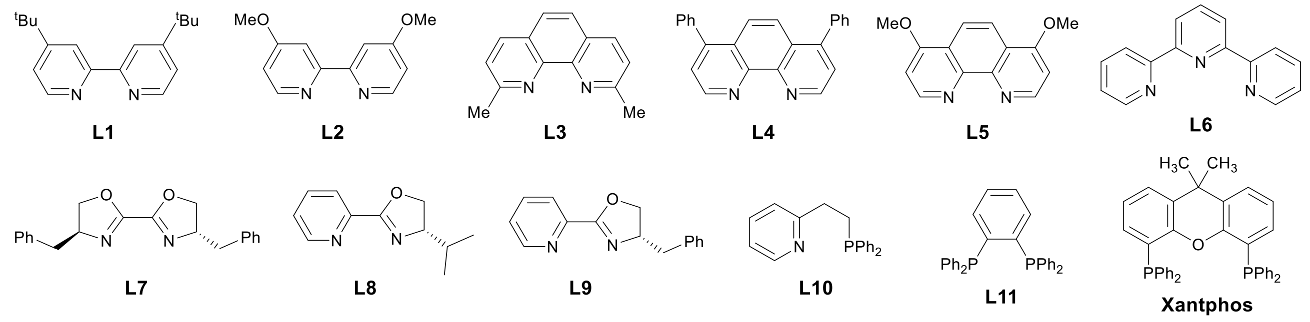
2. Optimization of Reaction Conditions

Table S1. Optimization of ligands.[a]



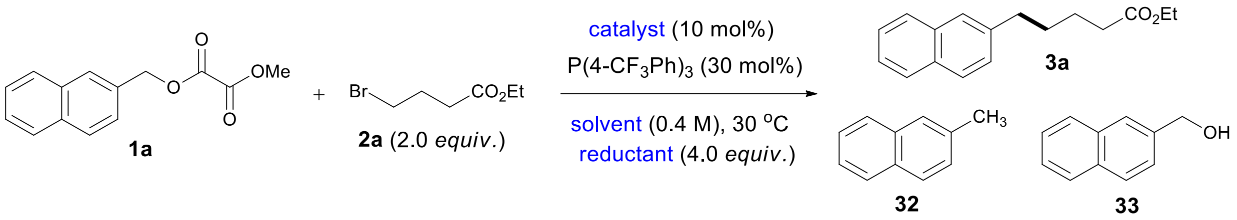
entry	ligand (mol%)	yield of products (%)		
		3a	**32**	**33**
1	bpy (15)	20	36	15
2	L1 (15)	4	7	0
3	L2 (15)	3	8	34
4	phen (15)	19	33	14
5	L3 (15)	3	29	33
6	L4 (15)	18	51	0
7	L5 (15)	2	5	23
8	L6 (15)	0	46	20
9	L7 (15)	5	7	48
10	L8 (15)	30	35	12
11	L9 (15)	32	30	16
12	L10 (15)	5	18	29
13	PPh_3 (30)	34	17	26
14	$P(2\text{-}MeOPh)_3$ (30)	0	9	33
15	$P(4\text{-}MeOPh)_3$ (30)	17	18	38
16	$P(2\text{-}MePh)_3$ (30)	0	4	28
17	$P(4\text{-}MePh)_3$ (30)	16	13	21
18	$P(4\text{-}FPh)_3$ (30)	50	14	14
19	**$P(4\text{-}CF_3Ph)_3$ (30)**	**58**	**7**	**25**
20	dppe (15)	21	26	32
21	dppp (15)	11	9	27
22	dppb (15)	15	12	19

23	dppf (15)	8	0	29
24	(R)-BINAP (15)	4	25	21
25	Xantphos (15)	6	15	40
26	L11 (15)	0	0	35



[a] Oxalate **1a** (0.2 mmol) was used and reacted for 24 h, yields were determined by ^{1}H NMR using anisole as an internal standard. phen: phenanthroline, dppe: bis(diphenylphosphino)ethane, dppp: 1,3-bis(diphenylphosphino)propane, dppb: 1,4-bis(diphenylphosphino)butane, dppf: 1,1'-bis(diphenylphosphino)ferrocene, (R)-BINAP: (R)-(+)-2,2'-bis(diphenylphosphino)-1,1'-binaphthalene.

Table S2. Optimization of solvent, catalyst and reductant[a]

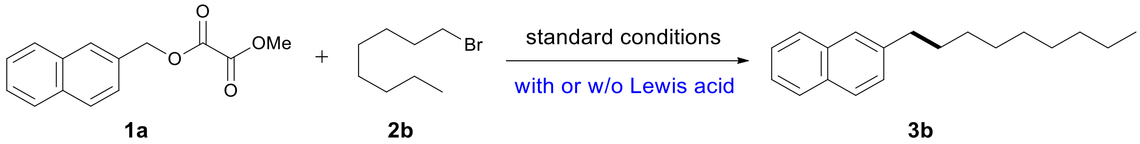


entry	catalyst	reductant	solvent (0.4 M)	yield of products (%)		
				3a	**32**	**33**
1	$NiBr_2$	Mn	DMF	58	7	25
2	$NiBr_2$	Mn	DMA	52	0	21
3	$NiBr_2$	Mn	DMPU	16	0	22
4	$NiBr_2$	Mn	DMI	28	0	17
5	$NiBr_2$	Mn	CH_3CN	3	8	21
6	$NiBr_2$	Mn	THF	51	9	33
7	$NiBr_2$	Mn	DME	10	4	35
8	**$NiBr_2$**	**Mn**	**DMSO**	**73 (79)[b]**	**6**	**7**
9	**$NiBr_2$**	**Mn**	**DMSO/DMF (1:1)**	**70 (75)[b]**	**3**	**9**
10	$NiBr_2$	Mn	DMSO/DMA (1:1)	68 (73)[b]	3	10
11	$NiBr_2$	Mn	DMSO/THF (1:1)	56	10	20
12	$NiBr_2$(diglyme)	Mn	DMSO	72	7	7
13	$NiCl_2$(DME)	Mn	DMSO	70	4	5
14	$NiCl_2$	Mn	DMSO	68	5	5
15	NiI_2	Mn	DMSO	46	3	14
16	Ni(acac)$_2$	Mn	DMSO	4	1	15
17	$NiBr_2$	Zn	DMSO	13	9	30
18	-	Mn	DMSO	0	-	-
19	$NiBr_2$	-	DMSO	0	-	-

[a] Oxalate **1a** (0.2 mmol) was used and reacted for 24 h, yields were determined by ^{1}H NMR using anisole as an internal standard. [b] Isolated yield.

3. Effect of Acidic Metals

Liebeskind,[2] Javor[3] and Martin[4] have demonstrated that the chelation of Lewis acidic metals to bidentate leaving groups would weaken the C–O bonds, thus accelerating the rate of oxidative addition process. As the presence of Mn^{2+} in our catalytic systems, we supposed that the leaving ability of oxalate might be enhanced by the same principle. In order to verify this concept, we then studied the effect of Lewis acid on this reaction. The reaction of oxalate **1a** with alkyl bromide **2b** was significantly accelerated when additional 25 mol% of $MnBr_2$ was used (Figure S1). Further, the reactivity of oxalate **1a** was dramatically improved in the presence of $Al(OTf)_3$ (25 mol%) (Figure S2). These results suggested that oxalate might be activated by acidic metals. Unfortunately, the use of extra Lewis acid such as $MnBr_2$, $Mn(OTf)_2$, $Al(OTf)_3$, $Mg(OTf)_2$, $MgBr_2$, $ZnBr_2$, $Sc(OTf)_3$, $Hf(OTf)_4$ did not lead to an improved yield of desired product.



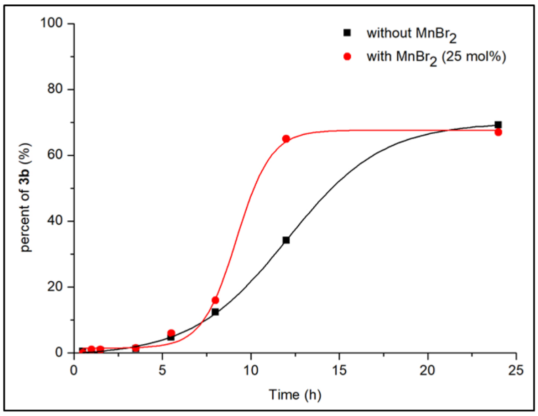


Figure S1. The effect of $MnBr_2$ on the formation of product **3b**. **2b** (2.0 equiv.) was used.

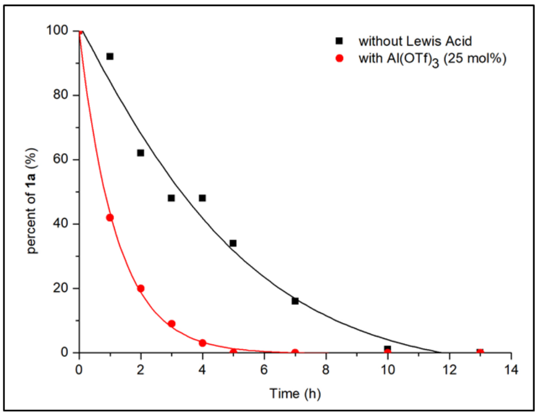
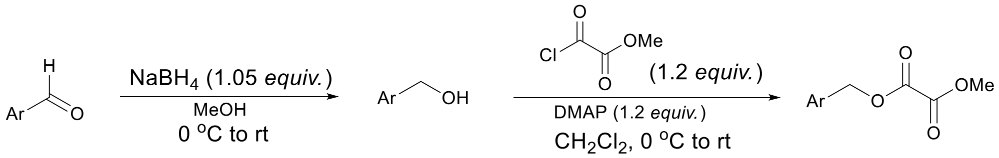


Figure S2. The effect of $Al(OTf)_3$ on the reactivity of oxalate **1a**. **2b** (1.0 equiv.), $Ni(COD)_2$ (10 mol%) were used. **1a** and **2b** were added after 12 h.

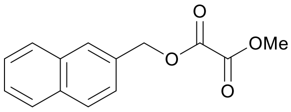
4. Preparation of Starting Materials

4.1 General Procedure for the synthesis of carboxylates and ethers



To a solution of aldehyde (2.5 mmol) in MeOH (10 mL) was added $NaBH_4$ (2.63 mmol, 1.05 equiv.) at 0 ^{o}C. The reaction mixture was stirred at room temperature until the reaction was completed (monitored by TLC). The reaction mixture was then quenched with water (10 mL) and extracted twice with EtOAc. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was directly used for the next step without further purification.

To a solution of DMAP (3.0 mmol, 1.2 equiv.) in CH_2Cl_2 (10 mL) was dropwise added methyl chlorooxoacetate (3.0 mmol, 1.2 equiv.) at 0 ^{o}C. After stirring at room temperature for 5 min, a solution of benzyl alcohol (about 2.5 mmol) in CH_2Cl_2 (5 mL) was dropwise added. The reaction mixture was stirred for 10 min and then quenched with water (10 mL), extracted twice with CH_2Cl_2. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the oxalates.



Methyl (naphthalen-2-ylmethyl) oxalate (1a)

This compound was synthesized according to the General Procedure, but 2-naphthalenemethanol (3.16 g, 20.0 mmol) was used directly.

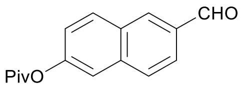
4.54 g (93% yield), white solid, Mp. 89-91 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.83 (m, 4 H), 7.52-7.48 (m, 3 H), 5.47 (s, 2 H), 3.89 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 157.4, 133.3, 133.1, 131.4, 128.6, 128.3, 128.1, 127.7, 126.6, 126.4, 125.9, 68.8, 53.6.

IR (neat, cm^{-1}): 1742, 1231, 935, 899, 865, 833, 795, 742.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{14}H_{12}NaO_4$ 267.0628, found 267.0629.



6-Formylnaphthalen-2-yl pivalate (1b-1)

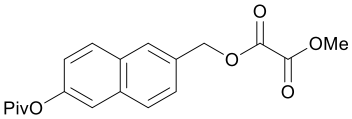
To a solution of 6-hydroxy-2-naphthaldehyde (0.86 g, 5.0 mmol) and Et_3N (1.40 mL, 10.0 mmol) in CH_2Cl_2 (20 mL) was dropwise added pivaloyl chloride (0.68 mL, 5.5 mmol) at 0 ℃. The reaction mixture was then allowed to warm to room temperature and stirred for 3-4 hours. The reaction was then quenched with water and the mixture extracted twice with CH_2Cl_2. The combine organic layers was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (1.18 g, 92% yield), Mp. 87-89 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 10.12 (s, 1 H), 8.29 (s, 1 H), 7.98-7.92 (m, 2 H), 7.85 (d, *J* = 8.8 Hz, 1 H), 7.59 (d, *J* = 2.0 Hz, 1 H), 7.29 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 1.41 (s, 9 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 191.9, 176.9, 151.3, 137.0, 134.0, 133.8, 130.9, 130.4, 128.6, 123.4, 122.5, 118.7, 39.2, 27.1.

IR (neat, cm^{-1}): 2982, 2360, 1744, 1693, 1472, 1255, 1166, 1146, 1120, 1104, 1033, 913, 816.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{16}H_{16}NaO_3$ 279.0992, found 279.1001.



Methyl ((6-(pivaloyloxy)naphthalen-2-yl)methyl) oxalate (**1b**)

This compound was synthesized according to the General Procedure, but the above aldehyde (*6-formylnaphthalen-2-yl pivalate*) (0.64 g, 2.5 mmol) was used.

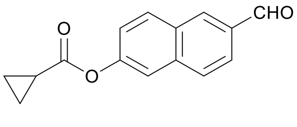
0.71 g (83% yield for 2 steps), white solid, Mp. 74-76 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.82-7.75 (m, 3 H), 7.52 (d, *J* = 2.0 Hz, 1 H), 7.45 (d, *J* = 8.4 Hz, 1 H), 7.21 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 5.40 (s, 2 H), 3.85 (s, 3 H), 1.39 (s, 9 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 176.9, 157.8, 157.2, 149.2, 133.5, 131.1, 130.8, 129.3, 128.0, 127.9, 126.5, 121.7, 118.2, 68.4, 53.4, 38.9, 27.0.

IR (neat, cm^{-1}): 1747, 1478, 1313, 1201, 1145, 1108.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{19}H_{20}NaO_6$ 367.1152, found 367.1153.



6-Formylnaphthalen-2-yl cyclopropanecarboxylate (**1c-1**)

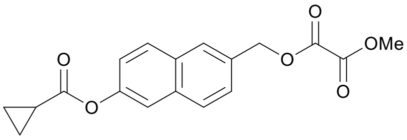
To a solution of 6-hydroxy-2-naphthaldehyde (0.86 g, 5.0 mmol), cyclopropanecarboxylic acid (0.44 mL, 5.5 mmol) and DMAP (61 mg, 0.5 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.14 g, 5.5 mmol) in CH_2Cl_2 (10 mL). After stirring at room temperature for 12 h, the reaction mixture was filtered through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a white solid (1.02 g, 85% yield), Mp. 77-79 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 10.10 (s, 1 H), 8.26 (s, 1 H), 7.95-7.90 (m, 2 H), 7.82 (d, J = 8.4 Hz, 1 H), 7.60 (s, 1 H), 7.32 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 1.93-1.87 (m, 1 H), 1.23-1.19 (m, 2 H), 1.09-1.04 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 191.8, 173.1, 150.9, 136.9, 133.9, 133.8, 130.8, 130.3, 128.6, 123.3, 122.4, 118.7, 13.0, 9.4.

IR (neat, cm^{-1}): 2360, 1746, 1693, 1629, 1474, 1378, 1245, 1145, 1094.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{15}H_{12}NaO_3$ 263.0679, found 263.0686.



(6-((Cyclopropanecarbonyl)oxy)naphthalen-2-yl)methyl methyl oxalate (1c)

This compound was synthesized according to the General Procedure, but the above aldehyde (*6-formylnaphthalen-2-yl cyclopropanecarboxylate*) (0.60 g, 2.5 mmol) was used.

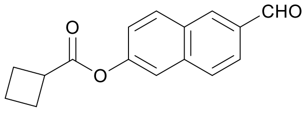
0.65 g (79% yield for 2 steps), white solid, Mp. 95-97 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.85-7.77 (m, 3 H), 7.56 (d, J = 2.0 Hz, 1 H), 7.47 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 7.27-7.24 (m, 1 H), 5.43 (s, 2 H), 3.87 (s, 3 H), 1.91-1.85 (m, 1 H), 1.22-1.18 (m, 2 H), 1.06-1.01 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.4, 157.9, 157.3, 149.0, 133.6, 131.2, 130.9, 129.4, 128.2, 128.0, 126.6, 121.8, 118.3, 68.5, 53.5, 12.9, 9.2.

IR (neat, cm^{-1}): 1743, 1387, 1221, 1153, 891, 825.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{18}H_{16}NaO_6$ 351.0839, found 351.0830.



6-Formylnaphthalen-2-yl cyclobutanecarboxylate (1d-1)

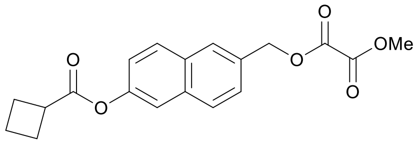
To a solution of 6-hydroxy-2-naphthaldehyde (0.86 g, 5.0 mmol), cyclobutanecarboxylic acid (0.53 mL, 5.5 mmol) and DMAP (61 mg, 0.5 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.14 g, 5.5 mmol) in CH_2Cl_2 (10 mL). After stirring at room temperature for 12 h, the reaction mixture was filtered through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a white solid (1.10 g, 87% yield), Mp. 60-62 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 10.14 (s, 1 H), 8.32 (s, 1 H), 8.00 (d, *J* = 8.8 Hz, 1 H), 7.96 (d, *J* = 1.6 Hz, 1 H), 7.94 (d, *J* = 1.2 Hz, 1 H), 7.63 (d, *J* = 2.0 Hz, 1 H), 7.34 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 3.50-3.41 (m, 1 H), 2.54-2.41 (m, 2 H), 2.41-2.33 (m, 2 H), 2.15-1.98 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 191.9, 173.7, 151.0, 137.1, 134.0, 133.9, 131.0, 130.5, 128.7, 123.5, 122.5, 118.8, 38.1, 25.3, 18.4.

IR (neat, cm^{-1}): 2948, 1752, 1694, 1629, 1473, 1336, 1258, 1144, 1032, 896, 814, 777.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{16}H_{14}NaO_3$ 277.0835, found 277.0847.



(6-((Cyclobutanecarbonyl)oxy)naphthalen-2-yl)methyl methyl oxalate (**1d**)

This compound was synthesized according to the General Procedure, but the above aldehyde (*6-formylnaphthalen-2-yl cyclobutanecarboxylate*) (0.64 g, 2.5 mmol) was used.

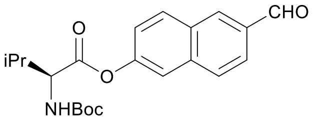
0.65 g (76% yield for 2 steps), white solid, Mp. 69-71 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.85-7.77 (m, 3 H), 7.55 (d, *J* = 2.0 Hz, 1 H), 7.48 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 7.24 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 5.43 (s, 2 H), 3.87 (s, 3 H), 3.47-3.39 (m, 1 H), 2.52-2.43 (m, 2 H), 2.39-2.30 (m, 2 H), 2.12-1.95 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.8, 157.9, 157.3, 149.0, 133.6, 131.2, 130.9, 129.4, 128.2, 128.0, 126.6, 121.8, 118.3, 68.5, 53.5, 38.0, 25.2, 18.3.

IR (neat, cm^{-1}): 2956, 1752, 1221, 1176, 894, 823.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{19}H_{18}NaO_6$ 365.0996, found 365.0986.



6-Formylnaphthalen-2-yl (*tert*-butoxycarbonyl)-*L*-valinate (**1e-1**)

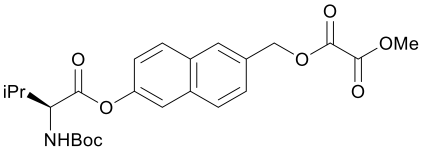
To a solution of *N*-Boc-*L*-valine[5] (1.19 g, 5.5 mmol), 6-hydroxy-2-naphthaldehyde (0.86 g, 5.0 mmol) and DMAP (61 mg, 0.5 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.14 g, 5.5 mmol) in CH_2Cl_2 (10 mL). After stirring at room temperature for 12 h, the reaction mixture was filtered through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a viscous oil (1.52 g, 82% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 10.14 (s, 1 H), 8.32 (s, 1 H), 8.02-7.95 (m, 2 H), 7.88 (d, *J* = 8.4 Hz, 1 H), 7.63 (s, 1 H), 7.35 (dd, J_1 = 1.6 Hz, J_2 = 8.8 Hz, 1 H), 5.19-5.01 (m, 1 H), 4.55-4.33 (m, 1 H), 2.41-2.37 (m, 1 H), 1.49 (s, 9 H), 1.14 (d, *J* = 7.2 Hz, 3 H), 1.08 (d, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 191.8, 171.0, 155.7, 150.5, 136.9, 134.0, 134.0, 131.1, 130.6, 128.7, 123.6, 122.2, 118.8, 80.1, 58.8, 31.2, 28.3, 19.1, 17.7.

IR (neat, cm^{-1}): 2970, 2360, 1763, 1697, 1630, 1508, 1473, 1366, 1257, 1158, 974, 776.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{21}H_{25}NNaO_5$ 394.1625, found 394.1636.



(6-(((*tert*-Butoxycarbonyl)-*L*-valyl)oxy)naphthalen-2-yl)methyl methyl oxalate (1e)

This compound was synthesized according to the General Procedure, but the above aldehyde (*6-formylnaphthalen-2-yl (tert-butoxycarbonyl)-L-valinate*) (0.93 g, 2.5 mmol) was used.

0.87 g (76% yield for 2 steps), white solid, Mp. 79-81 ^{o}C.

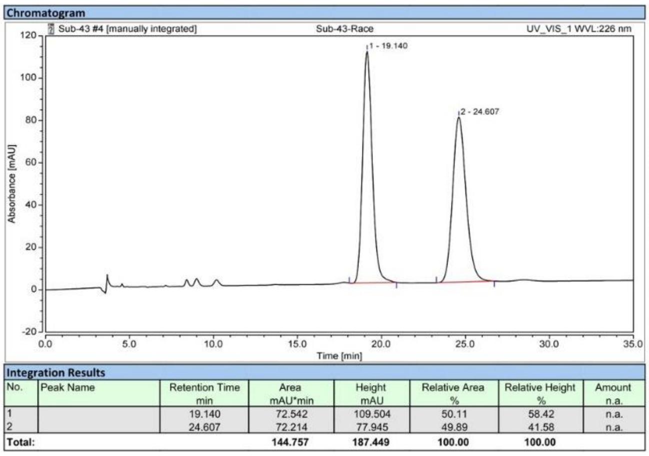
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.74 (m, 3 H), 7.54 (s, 1 H), 7.45 (d, *J* = 8.0 Hz, 1 H), 7.24 (d, *J* = 8.8 Hz, 1 H), 5.40 (s, 2 H), 5.30-5.28 (m, 1 H), 4.53-4.30 (m, 1 H), 3.85 (s, 3 H), 2.37-2.36 (m, 1 H), 1.48 (s, 9 H), 1.11 (d, *J* = 6.8 Hz, 3 H), 1.06 (d, *J* = 6.8 Hz, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 170.9, 157.7, 157.2, 155.5, 148.5, 133.4, 131.4, 130.9, 129.4, 128.0, 127.7, 126.5, 121.3, 118.1, 79.7, 68.2, 58.6, 53.3, 31.0, 28.1, 18.9, 17.5.

IR (neat, cm^{-1}): 2968, 1748, 1714, 1508, 1367, 1312, 1152.

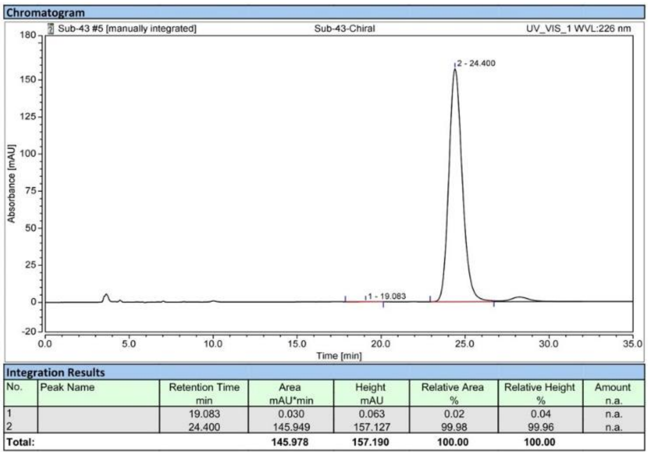
HRMS (ESI): [M+Na]$^+$ calcd. for $C_{24}H_{29}NNaO_8$ 482.1785, found 482.1774.

HPLC analysis: 99.9% ee; Chiralpak IC column (Hexane/*i*-PrOH = 75:25, flow rate = 1.0 mL/min, wave length = 226 nm), t_R = 19.083 min (minor), t_R = 24.400 min (major). $[\alpha]^{23}_D$ = -23.000 (c = 10.0 mg/mL, CH_2Cl_2).



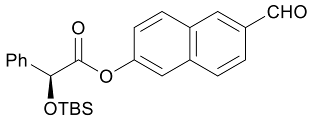
Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		19.140	72.542	109.504	50.11	58.42	n.a.
2		24.607	72.214	77.945	49.89	41.58	n.a.
Total:			**144.757**	**187.449**	**100.00**	**100.00**	

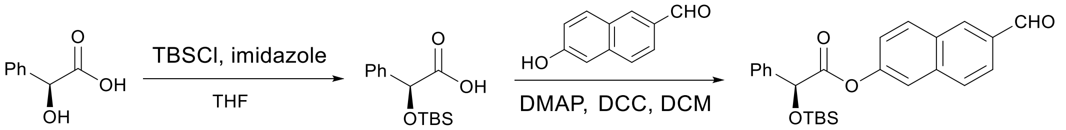


Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		19.083	0.030	0.063	0.02	0.04	n.a.
2		24.400	145.949	157.127	99.98	99.96	n.a.
Total:			**145.978**	**157.190**	**100.00**	**100.00**	



6-Formylnaphthalen-2-yl (*S*)-2-((*tert*-butyldimethylsilyl)oxy)-2-phenylacetate (1f-1)



Step 1: Following the literature procedure[6], *L*-(+)-Mandelic acid (0.31 g, 2.0 mmol) was dissolved in THF (15 mL) and cooled to 0 ℃ (ice bath). TBSCl (0.60 g, 4.0 mmol) and imidazole (0.33 g, 4.8 mmol) were added and the reaction mixture was stirred at 0 ^{o}C for 30 min. After stirring at room temperature for 24 h, the reaction mixture was filtered and concentrated in vacuum. The residue was dissolved in aqueous NaOH (3.0 mL, 1M) and stirred for 1.5 h. The mixture was diluted with H_2O (3.0 mL) and extracted twice with Et_2O. The aqueous phase was then acidified with 10% HCl until a pH of 3.5 and subsequently extracted twice with Et_2O. The combined organic solution was dried over anhydrous $MgSO_4$, filtered, and concentrated to afford the corresponding acid for next step without purification.

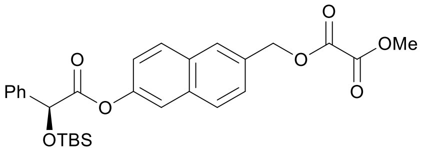
Step 2: To a solution of above residue, 6-hydroxy-2-naphthaldehyde (0.35 g, 2.0 mmol) and DMAP (25 mg, 0.2 mmol) in CH_2Cl_2 (15 mL) was added a solution of DCC (0.45 g, 2.2 mmol) in CH_2Cl_2 (5 mL). After stirring at room temperature for 12 h, the reaction mixture was filtered through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a viscous oil (0.71 g, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1 H), 8.21 (s, 1 H), 7.89-7.87 (m, 2 H), 7.76 (d, J = 8.4 Hz, 1 H), 7.66-7.64 (m, 2 H), 7.51 (s, 1 H), 7.44-7.41 (m, 2 H), 7.38-7.34 (m, 1 H), 7.18 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 5.55 (s, 1 H), 1.00 (s, 9 H), 0.23 (s, 3 H), 0.14 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.7, 170.4, 150.6, 138.4, 136.8, 133.9, 133.8, 130.9, 130.4, 128.55, 128.52, 128.45, 126.4, 123.4, 121.8, 118.4, 74.5, 25.6, 18.2, -5.0, -5.1.

IR (neat, cm^{-1}): 2930, 2857, 1775, 1697, 1630, 1473, 1336, 1257, 1146, 1105, 870, 838, 777, 720.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{25}H_{28}NaO_4Si$ 443.1649, found 443.1660.



(*S*)-(6-(2-((*tert*-Butyldimethylsilyl)oxy)-2-phenylacetoxy)naphthalen-2-yl)methyl methyl oxalate (1f)

This compound was synthesized according to the General Procedure, but the above aldehyde (6-formylnaphthalen-2-yl (S)-2-((tert-butyldimethylsilyl)oxy)-2-phenylacetate) (0.53 g, 1.25 mmol) was used.

0.46 g (72% yield for 2 steps), white solid, Mp. 78-80 ^{o}C.

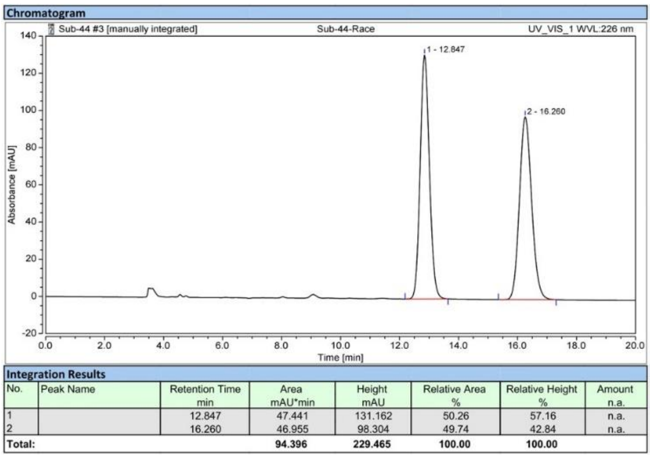
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.84-7.75 (m, 3 H), 7.65 (d, *J* = 7.2 Hz, 2 H), 7.49-7.42 (m, 4 H), 7.39-7.36 (m, 1 H), 7.14 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 5.54 (s, 1 H), 5.43 (s, 2 H), 3.88 (s, 3 H), 1.01 (s, 9 H), 0.24 (s, 3 H), 0.15 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 170.6, 157.9, 157.3, 148.8, 138.6, 133.5, 131.4, 131.0, 129.5, 128.5, 128.4, 128.2, 127.9, 126.6, 126.5, 121.2, 118.0, 74.5, 68.4, 53.5, 25.6, 18.3, -5.0, -5.1.

IR (neat, cm^{-1}): 2955, 1772, 1747, 1474, 1313, 1200, 1147, 871, 838, 781.

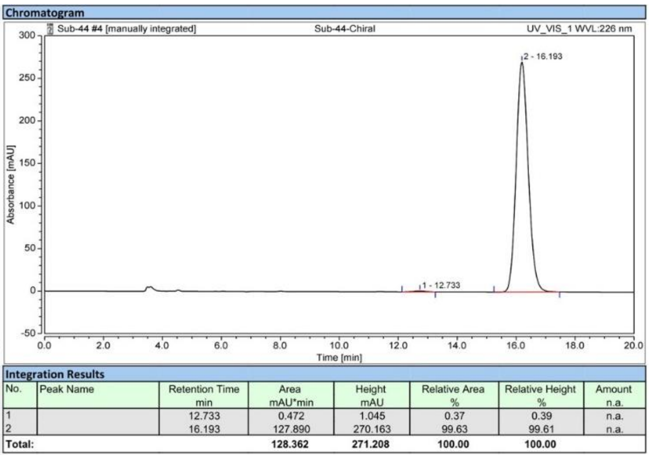
HRMS (ESI): [M+Na]$^+$ calcd. for $C_{28}H_{32}NaO_7Si$ 531.1810, found 531.1795.

HPLC analysis: 99.2% ee; Chiralpak IC column (Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, wave length = 226 nm), t_R = 12.733 min (minor), t_R = 16.193 min (major). $[\alpha]^{23}_D$ = +32.000 (c = 10.0 mg/mL, CH_2Cl_2).



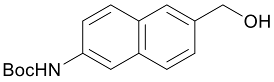
Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		12.847	47.441	131.162	50.26	57.16	n.a.
2		16.260	46.955	98.304	49.74	42.84	n.a.
Total:			94.396	229.465	100.00	100.00	



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		12.733	0.472	1.045	0.37	0.39	n.a.
2		16.193	127.890	270.163	99.63	99.61	n.a.
Total:			128.362	271.208	100.00	100.00	



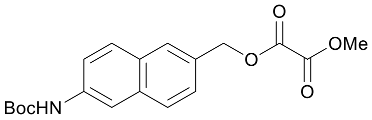
***tert*-Butyl (6-(hydroxymethyl)naphthalen-2-yl)carbamate** (**1g-1**)

This compound was synthesized according to the literature procedure.[7] To a mixture of 2-(6-aminonaphthyl)methanol[8] (0.69 g, 4.0 mmol), aqueous NaOH (5 mL, 1 M), water (4 mL) and dioxane (4 mL) was added Boc_2O (1.60 mL, 1.53 g, 7.0 mmol) at 0 ^{o}C. After stirring at room temperature for 6 h, dioxane was removed under reduced pressure and the residue was extracted twice with EtOAc. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title alcohol as a light brown solid (0.82 g, 80% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.97 (s, 1 H), 7.73-7.69 (m, 3 H), 7.41 (dd, J_1 = 1.2 Hz, J_2 = 8.4

Hz, 1 H), 7.31 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 6.69 (s, 1 H), 4.79 (s, 2H), 1.95 (s, 1H), 1.55 (s, 9H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 152.8, 136.9, 135.9, 133.5, 129.9, 128.7, 127.8, 125.9, 125.2, 119.5, 114.5, 80.7, 65.4, 28.4.



(6-((*tert*-Butoxycarbonyl)amino)naphthalen-2-yl)methyl methyl oxalate (**1g**)

This compound was synthesized according to the General Procedure, but the above alcohol (tert-butyl (6-(hydroxymethyl)naphthalen-2-yl)carbamate) (0.68 g, 2.5 mmol) was used directly.

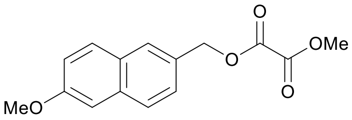
0.82 g (91% yield), white solid, Mp. 99-101 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00 (s, 1 H), 7.74-7.67 (m, 3 H), 7.41 (d, J = 8.4 Hz, 1 H), 7.33 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 6.88 (s, 1 H), 5.40 (s, 2 H), 3.87 (s, 3 H), 1.54 (s, 9 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 158.0, 157.4, 152.7, 136.6, 134.0, 129.8, 129.4, 128.8, 128.0, 127.9, 126.6, 119.6, 114.1, 80.6, 68.8, 53.5, 28.2.

IR (neat, cm^{-1}): 2979, 2256, 1747, 1637, 1541, 1498, 1239, 1051, 1026, 845, 814, 735.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{19}H_{21}NNaO_6$ 382.1261, found 382.1251.



(6-Methoxynaphthalen-2-yl)methyl methyl oxalate (**1h**)

This compound was synthesized according to the General Procedure, but 6-methoxy-2-naphthaldehyde (0.47 g, 2.5 mmol) was used.

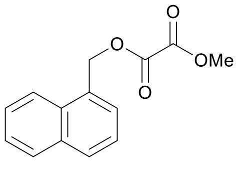
0.53 g (78% yield for 2 steps), white solid, Mp. 109-111 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80 (s, 1 H), 7.74-7.71 (m, 2 H), 7.45 (d, J = 8.8 Hz, 1 H), 7.17-7.12 (m, 2 H), 5.42 (s, 2 H), 3.90 (s, 3 H), 3.87 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 158.2, 158.0, 157.4, 134.6, 129.5, 129.0, 128.5, 128.4, 127.3, 126.7, 119.3, 105.6, 68.9, 55.2, 53.5.

IR (neat, cm^{-1}): 1750, 1611, 1488, 1217, 1179, 1030, 893, 859.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{15}H_{14}NaO_5$ 297.0733, found 297.0735.



Methyl (naphthalen-1-ylmethyl) oxalate (**1i**)

This compound was synthesized according to the General Procedure, but 1-naphthalenemethanol (0.40 g, 2.5 mmol) was used directly.

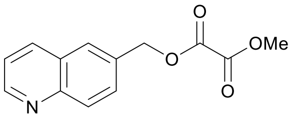
0.59 g (97% yield), white solid, Mp. 55-57 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.99 (d, *J* = 8.4 Hz, 1 H), 7.85 (dd, J_1 = 4.8 Hz, J_2 = 8.0 Hz, 2 H), 7.57-7.48 (m, 3 H), 7.42 (t, *J* = 8.0 Hz, 1 H), 5.73 (s, 2 H), 3.81 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.9, 157.4, 133.6, 131.4, 129.8, 129.5, 128.7, 128.2, 126.8, 126.0, 125.1, 123.2, 66.7, 53.4.

IR (neat, cm^{-1}): 2360, 1769, 1744, 1312, 1200, 1156, 793.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{14}H_{12}NaO_4$ 267.0628, found 267.0629.



Methyl (quinolin-6-ylmethyl) oxalate (1j)

This compound was synthesized according to the General Procedure, but 6-quinolinecarbaldehyde (0.39 g, 2.5 mmol) was used.

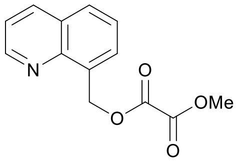
0.43 g (70% yield for 2 steps), white solid, Mp. 88-90 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.95 (dd, J_1 = 1.6 Hz, J_2 = 4.0 Hz, 1 H), 8.19-8.12 (m, 2 H), 7.88 (d, *J* = 1.2 Hz, 1 H), 7.74 (dd, J_1 = 2.0 Hz, J_2 = 4.8 Hz, 1 H), 7.44 (dd, J_1 = 4.0 Hz, J_2 = 8.0 Hz, 1 H), 5.50 (s, 2 H), 3.92 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.8, 157.3, 151.1, 148.1, 136.2, 132.3, 130.1, 129.4, 127.9, 127.9, 121.6, 68.1, 53.7.

IR (neat, cm^{-1}): 2391, 1735, 1229, 892, 833, 798.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{13}H_{12}NO_4$ 246.0761, found 246.0762.



Methyl (quinolin-8-ylmethyl) oxalate (1k)

This compound was synthesized according to the General Procedure, but 8-quinolinecarbaldehyde (0.39 g, 2.5 mmol) was used.

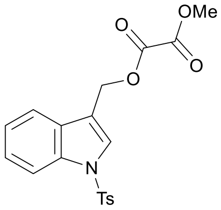
0.43 g (71% yield for 2 steps), white solid, Mp. 79-81 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.94 (dd, J_1 = 1.6 Hz, J_2 = 4.0 Hz, 1 H), 8,17 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1 H), 7.84-7.81 (m, 2 H), 7.55 (t, *J* = 8.0 Hz, 1 H), 7.44 (dd, J_1 = 4.0 Hz, J_2 = 8.4 Hz, 1 H), 6.06 (s, 2 H), 3.90 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 158.2, 157.5, 150.0, 146.0, 136.1, 132.4, 129.0, 128.7, 128.1, 126.1, 121.4, 65.2, 53.5.

IR (neat, cm^{-1}): 2360, 1741, 1501, 1313, 1203, 1169, 830, 792, 668.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{13}H_{12}NO_4$ 246.0761, found 246.0764.



Methyl ((1-tosyl-1*H*-indol-3-yl)methyl) oxalate (**1l**)

This compound was synthesized according to the General Procedure, but indole-3-carboxaldehyde[9] (0.44 g, 3.0 mmol) was used.

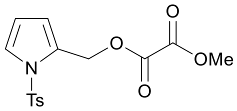
0.87 g (75% yield for two steps), white solid, Mp. 94-96 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.97 (d, *J* = 8.4 Hz, 1 H), 7.77 (d, *J* = 8.0 Hz, 2 H), 7.72 (s, 1 H), 7.59 (d, *J* = 7.6 Hz, 1 H), 7.33 (t, *J* = 7.2 Hz, 1 H), 7.24 (t, *J* = 7.2 Hz, 1 H), 7.18 (d, *J* = 8.0 Hz, 2 H), 5.44 (s, 2 H), 3.82 (s, 3 H), 2.26 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.6, 157.2, 145.1, 134.7, 134.6, 129.8, 128.9, 126.7, 126.6, 125.0, 123.4, 119.5, 115.3, 113.4, 59.8, 53.4, 21.3.

IR (neat, cm^{-1}): 2360, 1745, 1447, 1374, 1311, 1173, 1122, 974, 747, 671.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{19}H_{17}NNaO_6S$ 410.0669, found 410.0671.



Methyl ((1-tosyl-1*H*-pyrrol-2-yl)methyl) oxalate (**1m**)

This compound was synthesized according to the General Procedure, but 1-tosyl-pyrrole-2-carbaldehyde[9] (0.29 g, 3.0 mmol) was used.

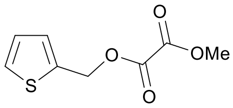
0.80 g (79% yield for two steps), white solid, Mp. 131-133 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.83 (d, *J* = 8.4 Hz, 2 H), 7.33-7.29 (m, 3 H), 6.43 (dd, J_1 = 1.6 Hz, J_2 = 3.2 Hz, 1 H), 6.25 (t, *J* = 3.2 Hz, 1 H), 5.46 (s, 2 H), 3.87 (s, 3 H), 2.41 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.6, 156.9, 145.2, 135.9, 130.0, 127.2, 126.8, 124.8, 119.0, 111.7, 59.7, 53.5, 21.6.

IR (neat, cm^{-1}): 1744, 1358, 1199, 1171, 1152, 755, 671.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{15}H_{15}NNaO_6S$ 360.0512, found 360.0517.



Methyl (thiophen-2-ylmethyl) oxalate (**1n**)

This compound was synthesized according to the General Procedure, but 2-thiophenemethanol (0.24 mL, 0.29 g, 2.5 mmol) was used directly.

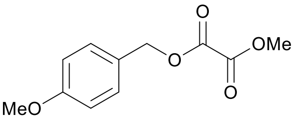
0.48 g (96% yield), colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 (dd, J_1 = 0.8 Hz, J_2 = 4.8 Hz, 1 H), 7.18 (d, J = 3.2 Hz, 1 H), 7.00 (dd, J_1 = 3.6 Hz, J_2 = 4.8 Hz, 1 H), 5.46 (s, 2 H), 3.88 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.8, 157.1, 135.5, 129.7, 127.8, 127.0, 62.5, 53.6.

IR (neat, cm^{-1}): 2395, 1743, 1311, 1200, 1152, 711.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_8H_8NaO_4S$ 223.0036, found 223.0038.



4-Methoxybenzyl methyl oxalate (**1o**).

This compound was synthesized according to the General Procedure, but 4-methoxybenzyl alcohol (0.31 mL, 0.35 g, 2.5 mmol) was used directly.

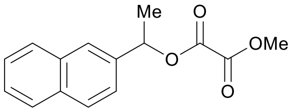
0.53 g (95% yield), white solid, Mp. 50-52 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.35 (d, J = 8.8 Hz, 2 H), 6.70 (d, J = 8.8 Hz, 2 H), 5.25 (s, 2 H), 3.88 (s, 3 H), 3.81 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 160.1, 158.1, 157.4, 130.7, 126.2, 114.0, 68.5, 55.3, 53.5.

IR (neat, cm^{-1}): 2962, 1767, 1614, 1518, 1440, 1385, 1237, 1187, 1031, 917, 892, 820, 786.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{11}H_{12}NaO_5$ 247.0577, found 247.0569.



Methyl (1-(naphthalen-2-yl)ethyl) oxalate (**1p**)

This compound was synthesized according to the General Procedure, but 2-acetonaphthone (0.43 g, 2.5 mmol) was used.

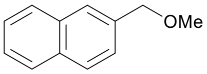
0.44 g (68% yield for 2 steps), white solid, Mp. 43-45 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.86-7.81 (m, 4 H), 7.52-7.46 (m, 3 H), 6.19 (q, J = 6.8 Hz, 1 H), 3.88 (s, 3 H), 1.74 (d, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 158.3, 156.9, 137.1, 133.2, 133.0, 128.6, 128.1, 127.6, 126.4, 126.4, 125.6, 123.8, 75.8, 53.5, 21.8.

IR (neat, cm^{-1}): 1744, 1438, 1307, 1204, 1166, 1059, 819, 749.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{15}H_{14}NaO_4$ 281.0784, found 281.0778.

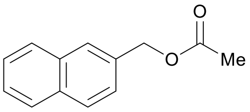


2-(Methoxymethyl)naphthalene (**4**, known compound)

To a suspension of NaH (0.40 g, 10.0 mmol, 60% in mineral oil) in dry THF (25 mL) was added a solution of 2-naphthalenemethanol (0.79 g, 5.0 mmol) in THF (5 mL) at 0 ℃. After stirring at room temperature for 1 h, MeI (0.62 mL, 10.0 mmol) was added. The reaction mixture was stirred at room temperature for 15 h, then quenched with water and extracted twice with EtOAc. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (0.81 g, 94% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 10.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 3 H), 7.78 (s, 1 H), 7.49-7.44 (m, 3 H), 4.62 (s, 2 H), 3.42 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 133.3, 133.0, 128.1, 127.8, 127.7, 126.4, 126.1, 125.8, 125.7, 74.7, 58.1.

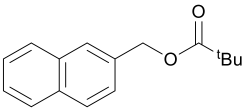


Naphthalen-2-ylmethyl acetate (**5**, known compound)

A round-bottom flask was charged with 2-naphthalenemethanol (0.79 g, 5.0 mmol) and dissolved with CH_2Cl_2 (20 mL). Et_3N (1.40 mL, 10.0 mmol) and acetyl chloride (0.39 mL, 5.5 mmol) were subsequently added dropwise to the reaction vessel at 0 ℃. The mixture was then allowed to warm to room temperature, and stirred for 3-4 hours. The reaction was then quenched with water and the mixture was extracted twice with CH_2Cl_2. The combine organic layers were washed with brine, dried over Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (0.89 g, 89% yield). Mp. 48-50 ^{o}C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 4.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 4 H), 7.51-7.44 (m, 3 H), 5.26 (s, 2 H), 2.12 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 133.3, 133.2, 133.1, 128.4, 127.9, 127.7, 127.3, 126.3, 126.2, 125.9, 66.4, 21.0.

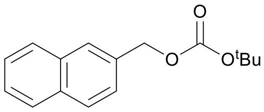


Naphthalen-2-ylmethyl pivalate (**6**, known compound)

A round-bottom flask was charged with 2-naphthalenemethanol (0.79 g, 5.0 mmol) and dissolved with CH_2Cl_2 (20 mL). Et_3N (1.40 mL, 10.0 mmol) and pivaloyl chloride (0.68 mL, 5.5 mmol) were subsequently added dropwise to the reaction vessel at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for 3-4 hours. The reaction was quenched with water and the mixture was extracted twice with CH_2Cl_2. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (1.10 g, 91% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 4.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.80 (m, 4 H), 7.51-7.43 (m, 3 H), 5.26 (s, 2 H), 1.25 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 133.8, 133.2, 133.0, 128.3, 127.9, 127.7, 126.8, 126.2, 126.1, 125.6, 66.2, 38.8, 27.2.



***tert*-Butyl (naphthalen-2-ylmethyl) carbonate** (**7**)

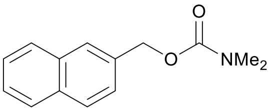
To a solution of 2-naphthalenemethanol (0.79 g, 5.0 mmol) in THF (30 mL) was dropwise added *n*-BuLi (2.0 mL, 5.0 mmol, 2.5 M hexane solution) at -78 ^{o}C. After stirring at -78 °C for 30 min, Boc_2O (1.15 mL, 1.09 g, 5.0 mmol) was then dropwise added. The reaction mixture was stirred at room temperature for 2 h and then quenched with water, extracted twice with EtOAc. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (1.12 g, 87% yield). Mp. 50-52 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.81 (m, 4 H), 7.48-7.46 (m, 3 H), 5.25 (s, 2 H), 1.50 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.4, 133.1, 133.0, 128.3, 128.0, 127.6, 127.3, 126.2, 125.8, 82.3, 68.8, 27.7.

IR (neat, cm^{-1}): 2980, 1739, 1369, 1279, 1254, 1161, 853, 818.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{16}H_{18}NaO_3$ 281.1148, found 281.1150.



Naphthalen-2-ylmethyl dimethylcarbamate (**8**)

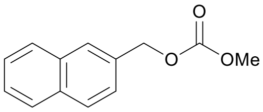
To a suspension of NaH (0.24 g, 6.0 mmol, 60% in mineral oil) in dry THF (15 mL) was dropwise added a solution of 2-naphthalenemethanol (0.79 g, 5.0 mmol) in THF (5 mL) at 0 ℃. After stirring for 30 min, dimethylcarbamoyl chloride (0.55 mL, 6.0 mmol) was slowly added. The reaction mixture was stirred at room temperature overnight and was then carefully quenched with saturated NH_4Cl, extracted twice with EtOAc. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (0.99 g, 79% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.83-7.81 (m, 4 H), 7.49-7.44 (m, 3 H), 5.28 (s, 2 H), 2.93 (s, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 156.4, 134.3, 133.1, 133.0, 128.2, 127.9, 127.6, 126.8, 126.1, 126.0, 125.7, 67.1, 36.5, 35.9.

IR (neat, cm^{-1}): 1703, 1493, 1400, 1183, 1053, 818, 749.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{14}H_{15}NNaO_2$ 252.0995, found 252.0994.

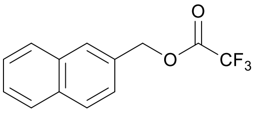


Methyl (naphthalen-2-ylmethyl) carbonate (**9**, known compound)

To a solution of 2-naphthalenemethanol (0.79 g, 5.0 mmol) in CH_2Cl_2 (20 mL) was successively added methyl chloroformate (0.58 mL, 7.5 mmol) and 4-dimethylaminopyridine (1.22 g, 10.0 mmol) at 0 °C. The reaction mixture was stirred at room temperature for 4 h and then quenched with saturated aqueous $NaHCO_3$ at 0 ℃. The organic layer was separated and the aqueous layer was extracted twice with EtOAc. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (0.92 g, 85% yield). Mp. 49-51 °C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 11.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.85-7.83 (m, 4 H), 7.51-7.47 (m, 3 H), 5.32 (s, 2 H), 3.80 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 155.7, 133.2, 133.1, 132.6, 128.4, 128.0, 127.7, 127.5, 126.4, 126.3, 125.7, 69.7, 54.9.



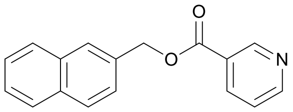
Naphthalen-2-ylmethyl 2,2,2-trifluoroacetate (**10**, known compound)

Under argon, 2-naphthalenemethanol (1.58 g, 10.0 mmol) and dry Et_2O (20 mL) were charged into a dry round-bottom flask containing a magnetic stir bar. Trifluoroacetic anhydride (2.78 mL, 20.0 mmol) was added dropwise at 0 °C. The resulting mixture was stirred at 0 °C for 2 h. At the end of the reaction (monitored by TLC), the reaction mixture was washed by saturated $NaHCO_3$ (3 × 30 mL) and dried over anhydrous $MgSO_4$, and then evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (2.42 g, 95% yield). Mp. 61-63 °C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 12.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.84 (m, 4 H), 7.53-7.51 (m, 2 H), 7.46 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 5.49 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4 (q, $J_{C\text{–}F}$ = 42.0 Hz), 133.4, 133.0, 130.5, 128.8, 128.3, 127.7, 126.8, 126.6, 125.6, 114.6 (q, $J_{C\text{–}F}$ = 284.0 Hz), 69.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.79.



Naphthalen-2-ylmethyl nicotinate (**11**)

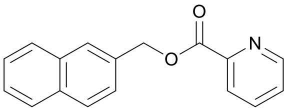
To a solution of 2-naphthalenemethanol (0.63 g, 4.0 mmol), nicotinic acid (0.99 g, 8.0 mmol) and DMAP (49 mg, 0.4 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.65 g, 8.0 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred at room temperature for 12 h and then filtered through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a white solid (0.92 g, 87% yield). Mp. 79-81 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 (d, J = 1.6 Hz, 1 H), 8.76 (dd, J_1 = 1.6 Hz, J_2 = 4.8 Hz, 1 H), 8.31 (dt, J_1 = 2.0 Hz, J_2 = 8.0 Hz, 1 H), 7.90-7.82 (m, 4 H), 7.55-7.47 (m, 3 H), 7.35 (dd, J_1 = 4.8 Hz, J_2 = 8.0 Hz, 1 H), 5.53 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 153.4, 150.9, 137.0, 133.02, 132.98, 132.7, 128.4, 127.9, 127.6, 127.5, 126.3, 125.8, 125.7, 123.2, 67.2.

IR (neat, cm^{-1}): 1723, 1590, 1452, 1280, 1110, 1024, 856, 817, 740, 701.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{17}H_{14}NO_2$ 264.1019, found 264.1015.



Naphthalen-2-ylmethyl picolinate (**12**)

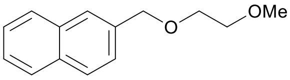
To a solution of 2-naphthalenemethanol (0.63 g, 4.0 mmol), picolinic acid (0.99 g, 8.0 mmol) and DMAP (49 mg, 0.4 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.65 g, 8.0 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred at room temperature for 12 h and then filtered through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a white solid (0.87 g, 83% yield). Mp. 69-71 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.77 (dd, J_1 = 0.8 Hz, J_2 = 4.8 Hz, 1 H), 8.14 (d, J = 7.6 Hz, 1 H), 7.95 (s, 1 H), 7.86-7.78 (m, 4 H), 7.59 (dd, J_1 = 2.0 Hz, J_2 = 8.4 Hz, 1 H), 7.50-7.43 (m, 3 H), 5.62 (s, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 165.0, 149.9, 147.9, 136.9, 133.11, 133.10, 132.9, 128.4, 128.0, 127.8, 127.6, 126.9, 126.3, 126.2, 126.1, 125.2, 67.6.

IR (neat, cm^{-1}): 2360, 1718, 1436, 1303, 1243, 1125, 817, 746, 704.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{17}H_{14}NO_2$ 264.1019, found 264.1021.



2-((2-Methoxyethoxy)methyl)naphthalene (**13**)

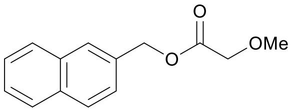
This compound was synthesized according to the literature procedure.[3] To a suspension of NaH (0.43 g, 10.8 mmol, 60% in mineral oil) in DMF (5.0 mL) was added a solution of 2-naphthalenemethanol (0.57 g, 3.6 mmol) in DMF (5.5 mL). After stirring for 30 min, a solution of bromoethyl methyl ether (0.38 mL, 0.56 g, 4.0 mmol) in DMF (4.0 mL) was added over 30 min. After stirring for 1 hour, a second portion of bromoethyl methyl ether (0.38 mL, 4.0 mmol) in DMF (4.0 mL) was slowly added over 30 min. The reaction mixture was stirred for two hours and then quenched with saturated aqueous NH_4Cl (15 mL), extracted twice with EtOAc. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The crude residue was purified by silica gel column to give the title product as a light yellow oil (0.52 g, 67% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.83-7.79 (m, 4H), 7.50-7.43 (m, 3H), 4.74 (s, 2H), 3.66-3.64 (m, 2H), 3.60-3.58 (m, 2H), 3.40 (s, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 135.6, 133.2, 132.9, 128.1, 127.8, 127.6, 126.5, 126.0, 125.78, 125.76, 73.4, 72.0, 69.2, 59.1.

IR (neat, cm^{-1}): 2920, 1634, 1509, 1470, 1362, 1198, 1124, 1097, 855, 817, 750.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{14}H_{17}O_2$ 217.1223, found 217.1227.

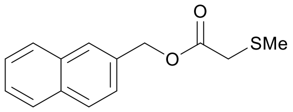


Naphthalen-2-ylmethyl 2-methoxyacetate (**14**, known compound)

To a solution of 2-naphthalenemethanol (0.63 g, 4.0 mmol), methoxyacetic acid (0.61 mL, 0.72 g, 8.0 mmol) and DMAP (49 mg, 0.4 mmol) in CH_2Cl_2 (25 mL) was dropwise added a solution of DCC (1.65 g, 8.0 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred at room temperature for 12 h and was then filtered through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a colorless oil (0.88 g, 96% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 13.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 4 H), 7.51-7.45 (m, 3 H), 5.36 (s, 2 H), 4.10 (s, 2 H), 3.46 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 133.13, 133.10, 132.7, 128.4, 128.0, 127.68, 127.67, 126.4, 126.3, 125.9, 69.8, 66.7, 59.4.



Naphthalen-2-ylmethyl 2-(methylthio)acetate (**15**)

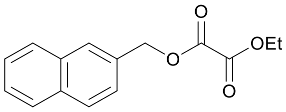
To a solution of 2-naphthalenemethanol (0.63 g, 4.0 mmol), (methylthio)acetic acid (0.70 mL, 0.85 g, 8.0 mmol) and DMAP (49 mg, 0.4 mmol) in CH_2Cl_2 (25 mL) was added a solution of DCC (1.65 g, 8.0 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred at room temperature for 12 h and was then filtered through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a colorless oil (0.86 g, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.82 (m, 4 H), 7.50-7.45 (m, 3 H), 5.33 (s, 2 H), 3.25 (s, 2 H), 2.19 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 133.1, 133.1, 133.0, 128.4, 127.9, 127.7, 127.4, 126.3, 126.3, 125.8, 67.1, 35.7, 16.3.

IR (neat, cm^{-1}): 2919, 1732, 1509, 1275, 1129, 972, 855, 816, 752.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{14}H_{15}O_2S$ 247.0787, found 247.0782.



Ethyl (naphthalen-2-ylmethyl) oxalate (**16**)

This compound was synthesized according to the General Procedure, but 2-naphthalenemethanol (0.40 g, 2.5 mmol) and ethyl chlorooxoacetate (3.0 mmol, 1.2 equiv.) were used directly.

0.62 g (96% yield), white solid, Mp. 40-42 ^{o}C.

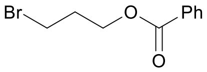
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.86-7.80 (m, 4 H), 7.50-7.47 (m, 3 H), 5.44 (s, 2 H), 4.32 (q, *J* = 7.2 Hz, 2 H), 1.33 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 157.7, 157.6, 133.2, 133.0, 131.5, 128.5, 128.2, 128.0, 127.6, 126.5, 126.4, 125.9, 68.6, 63.1, 13.8.

IR (neat, cm^{-1}): 1758, 1750, 1216, 1201, 870, 822, 731.

HRMS (ESI): [M+Na]$^+$ calcd for $C_{15}H_{14}NaO_4$ 281.0784, found 281.0785.

4.2 Preparation of alkyl bromides (other alkyl bromides are commercial available)

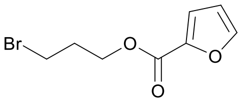


3-Bromopropyl benzoate (**2i**, known compound)

To a stirred solution of 3-bromo-1-propanol (0.45 mL, 0.69 g, 5.0 mmol) and Et_3N (0.80 mL, 5.5 mmol) in Et_2O (10 mL) was added benzoyl chloride (0.58 mL, 0.70 g, 5.0 mmol) at 0 ℃. After stirring at 0 ℃ for 1 h and then at room temperature for 8.5 h, the reaction mixture was quenched with water and extracted twice with EtOAc. The combined organic layers was dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (1.09 g, 90% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 14.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.05-8.03 (m, 2 H), 7.59-7.55 (m, 1 H), 7.45 (t, *J* = 7.6 Hz, 2 H), 4.47 (t, *J* = 6.0 Hz, 2 H), 3.55 (t, *J* = 6.4 Hz, 2 H), 2.36-2.29 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 166.3, 133.0, 130.0, 129.5, 128.4, 62.6, 31.8, 29.4.

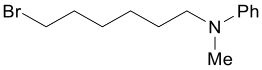


3-Bromopropyl furan-2-carboxylate (**2j**, known compound)

To a solution of 3-bromo-1-propanol (0.36 mL, 0.56 g, 4.0 mmol), 2-furoic acid (0.68 mL, 0.90 g, 8.0 mmol) and DMAP (49 mg, 0.4 mmol) in CH_2Cl_2 (25 mL) was added a solution of DCC (1.65 g, 8.0 mmol) in CH_2Cl_2 (10 mL). After stirring at room temperature for 12 h, the reaction mixture was filtered through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a colorless oil (0.87 g, 93% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 15.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (s, 1 H), 7.19 (d, J = 3.2 Hz, 1 H), 6.52 (dd, J_1 = 1.6 Hz, J_2 = 3.6 Hz, 1 H), 4.45 (t, J = 6.0 Hz, 2 H), 3.53 (t, J = 6.4 Hz, 2 H), 2.34-2.27 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 146.4, 144.3, 118.1, 111.8, 62.5, 31.7, 29.2.



_N_-(6-Bromohexyl)-_N_-methylaniline (**2l**)

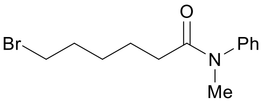
This compound was synthesized according to the literature procedure.[16] To a 100 mL round-bottom flask fitted with a reflux condenser were added *N*-methylaniline (1.08 mL, 10.0 mmol), K_2CO_3 (2.76 g, 20.0 mmol), and 1,6-dibromohexane (2.30 mL, 3.66 g, 15.0 mmol) in CH_3CN (40 mL). The reaction mixture was refluxed for 24 h and then cooled to room temperature. The mixture was quenched with water and extracted twice with EtOAc. The combined organic solution was washed with saturated sodium bicarbonate solution, water, saturated sodium chloride solution, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a yellow oil (1.70 g, 63% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, J = 8.0 Hz, 2 H), 6.69-6.65 (m, 3 H), 3.38 (t, J = 6.8 Hz, 2 H), 3.29 (t, J = 7.2 Hz, 2 H), 2.90 (s, 3 H), 1.88-1.76 (m, 2 H), 1.61-1.53 (m, 2 H), 1.49-1.42 (m, 2 H), 1.36-1.29 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 149.2, 129.1, 115.8, 112.0, 52.6, 38.2, 33.8, 32.7, 28.0, 26.5, 26.3.

IR (neat, cm^{-1}): 2932, 1598, 1506, 1367, 747, 691.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{13}H_{21}BrN$ 270.0852, found 270.0853.



6-Bromo-_N_-methyl-_N_-phenylhexanamide (**2o**)

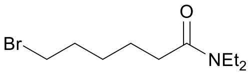
This compound was synthesized according to the literature procedure.[17] To a solution of *N*-methylaniline (1.08 mL, 10.0 mmol), DMAP (122 mg, 1.0 mmol) and Et_3N (2.08 mL, 15.0 mmol) in CH_2Cl_2 (50 mL) was dropwise added 6-bromohexanoyl chloride (1.53 mL, 2.14 g, 10.0 mmol) at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred overnight. The resulting suspension was passed through a pad of silica gel. The filtrate was concentrated, and the crude residue was purified by silica gel column to give the title product as a colorless oil (2.68 g, 95% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 (t, *J* = 7.6 Hz, 2 H), 7.35 (t, *J* = 7.2 Hz, 1 H), 7.18 (d, *J* = 7.6 Hz, 2 H), 3.34 (t, *J* = 6.8 Hz, 2 H), 3.26 (s, 3 H), 2.09 (t, *J* = 7.6 Hz, 2 H), 1.81-1.74 (m, 2 H), 1.64-1.56 (m, 2 H), 1.38-1.30 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 172.5, 143.9, 129.6, 127.6, 127.1, 37.1, 33.6, 33.5, 32.3, 27.6, 24.4.

IR (neat, cm^{-1}): 2935, 1657, 1595, 1496, 1384, 1117, 701.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{13}H_{19}BrNO$ 284.0645, found 284.0647.

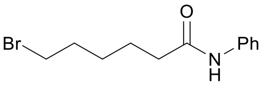


6-Bromo-*N*,*N*-diethylhexanamide (**2p**, known compound)

To a solution of Et_2NH (1.04 mL, 10.0 mmol), Et_3N (2.08 mL, 15.0 mmol) and DMAP (122 mg, 1.0 mmol) in CH_2Cl_2 (50 mL) was dropwise added a solution of 6-Bromohexanoyl chloride (1.53 mL, 2.14 g, 10.0 mmol) in CH_2Cl_2 (10 mL) at 0 °C. The mixture was then allowed to warm to room temperature, and stirred overnight. The resulting suspension was passed through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a colorless oil (2.38 g, 95% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 17.

^{1}H NMR (400 MHz, CDCl$_3$) δ 3.44-3.28 (m, 6 H), 2.32 (t, *J* = 7.6 Hz, 2 H), 1.91-1.86 (m, 2 H), 1.72-1.64 (m, 2 H), 1.53-1.47 (m, 2 H), 1.18 (t, *J* = 7.2 Hz, 3 H), 1.11 (t, *J* = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 171.5, 41.7, 39.8, 33.5, 32.6, 32.4, 27.8, 24.3, 14.2, 12.9.



6-Bromo-*N*-phenylhexanamide (**2q**)

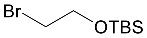
This compound was synthesized according to the literature procedure.[17] To a solution of aniline (0.91 mL, 10.0 mmol), Et_3N (2.08 mL, 15.0 mmol) and DMAP (122 mg, 1.0 mmol) in CH_2Cl_2 (50 mL) was dropwise added a solution of 6-bromohexanoyl chloride (1.53 mL, 2.14 g, 10.0 mmol) in CH_2Cl_2 (10 mL) at 0 °C. The mixture was then allowed to warm to room temperature, and stirred overnight. The resulting suspension was passed through a pad of silica gel. The filtrate was concentrated and the crude residue was purified by silica gel column to give the title product as a yellow solid (2.21 g, 82% yield). Mp. 75-77 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.08 (s, 1 H), 7.52 (d, *J* = 7.6 Hz, 2 H), 7.29-7.25 (m, 2 H), 7.10-7.06 (m, 1 H), 3.36 (t, *J* = 6.4 Hz, 2 H), 2.34 (t, *J* = 7.6 Hz, 2 H), 1.86-1.79 (m, 2 H), 1.74-1.66 (m, 2 H), 1.49-1.42 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 171.5, 137.9, 128.8, 124.1, 120.1, 37.1, 33.5, 32.3, 27.6, 24.6.

IR (neat, cm^{-1}): 3336, 2944, 1664, 1596, 1521, 1439, 1417, 754, 691.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{12}H_{17}BrNO$ 270.0488, found 270.0482.

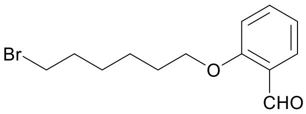


(2-Bromoethoxy)(*tert*-butyl)dimethylsilane (**2t**, known compound)

To a 50 mL round-bottom flask with stirbar was added 2-bromoethanol (1.0 g, 8.0 mmol), Et_3N (1.3 mL, 8.8 mmol), DMAP (50 mg, 0.4 mmol) and CH_2Cl_2 (25 mL). TBSCl (1.32 g, 8.8 mmol) was added at room temperature with constant stirring. After 12 h, water was added (30 mL), and the aqueous layer was extracted twice with CH_2Cl_2. The combine organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (1.49 g, 78%). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 18.

^{1}H NMR (400 MHz, CDCl$_3$) δ 3.89 (t, *J* = 6.4 Hz, 2 H), 3.40 (t, *J* = 6.4 Hz, 2 H), 0.91 (s, 9 H), 0.09 (s, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 63.5, 33.3, 25.8, 18.3, -5.3.



2-((6-Bromohexyl)oxy)benzaldehyde (**2v**)

This compound was synthesized according to the literature procedure.[19] To a solution of salicylaldehyde (0.53 mL, 0.61 g, 5.0 mmol) in DMF (15 mL) was added K_2CO_3 (1.38 g, 10.0 mmol). After stirring at room temperature for 1 h, a solution of 1,6-dibromohexane (1.15 mL, 1.83 g, 7.5 mmol) in DMF (5 mL) was then added and the mixture was stirred overnight. The reaction mixture was poured into water (30 mL) and extracted twice with EtOAc. The organic solution was washed with 2% NaOH (3 × 30 mL), water (2 × 30 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a colorless oil (1.03 g, 73% yield).

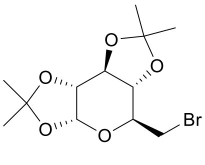
^{1}H NMR (400 MHz, CDCl$_3$) δ 10.51 (d, *J* = 0.8 Hz, 1 H), 7.82 (dd, J_1 = 1.6 Hz, J_2 = 7.6 Hz, 1

H), 7.55-7.51 (m, 1 H), 7.03-6.96 (m, 2 H), 4.08 (t, *J* = 6.4 Hz, 2 H), 3.43 (t, *J* = 6.8 Hz, 2 H), 1.92-1.86 (m, 4 H), 1.55-1.50 (m, 4 H).

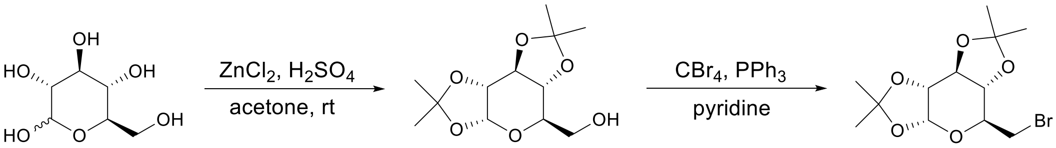
^{13}C NMR (100 MHz, CDCl$_3$) δ 189.7, 161.4, 135.9, 128.2, 124.8, 120.5, 112.4, 68.2, 33.6, 32.5, 28.9, 27.8, 25.2.

IR (neat, cm^{-1}): 2938, 1687, 1598, 1458, 1387, 1286, 1243, 1189, 1161, 1103, 1042, 840, 758, 654.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{13}H_{18}BrO_2$ 285.0485, found 285.0479.



(3a*R*,5*S*,5a*S*,8a*S*,8b*R*)-5-(Bromomethyl)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran (17)



Step 1: Following the literature procedure[20], to a solution of anhydrous $ZnCl_2$ (2.04 g, 15.0 mmol) in acetone (40 mL) was added H_2SO_4 (0.07 mL, 1.3 mmol) and D-(+)-Glucose (1.80 g, 10.0 mmol) at room temperature. The mixture was stirred at room temperature for 16 h, and then quenched with sat. aq. $NaHCO_3$ (30 mL). The resulted suspension was filtrated through a pad of Celite, then the Celite pad was washed with acetone. The filtrate was concentrated under reduced pressure to remove excess acetone and extracted with diethyl ether (3 × 30 mL). The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure. the residue was obtained as a white solid (2.47 g, 95% yield) and used directly without further purification.

Step 2: Following the literature procedure[21], to a solution of the above alcohol (1.30 g, 5.0 mmol) in anhydrous pyridine (30 mL) was added PPh_3 (2.62 g, 10.0 mmol) and CBr_4 (2.49 g, 7.5 mmol). The solution was warmed to 35 ℃ and stirred for 16 h, and then at 50 ℃ for an additional 48 h. The pyridine was evaporated in vacuum, and the resulting syrup was extracted with hexane (3 × 30 mL). The residue was dissolved in CH_2Cl_2 (50 mL), filtered, and the filtrate was washed with water (3 × 50 mL). The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure. The crude residue was purified by silica gel column to give the title product as a light yellow viscous oil (1.02 g, 63% yield).

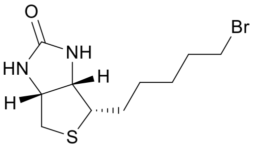
^{1}H NMR (400 MHz, CDCl$_3$) δ 6.01 (d, *J* = 3.6 Hz, 1 H), 4.60 (d, *J* = 4.0 Hz, 1 H), 4.33 (dd, J_1 =

7.2 Hz, J_2 = 3.6 Hz, 1 H), 4.24 (d, J = 3.6 Hz, 1 H), 3.75 (dt, J_1 = 7.6 Hz, J_2 = 3.2 Hz, 1 H), 3.63 (dd, J_1 = 11.2 Hz, J_2 = 3.2 Hz, 1 H), 3.45 (dd, J_1 = 10.8 Hz, J_2 = 7.6 Hz, 1 H), 1.50 (s, 3H), 1.39 (s, 3 H), 1.38 (s, 3 H), 1.34 (s, 3H).

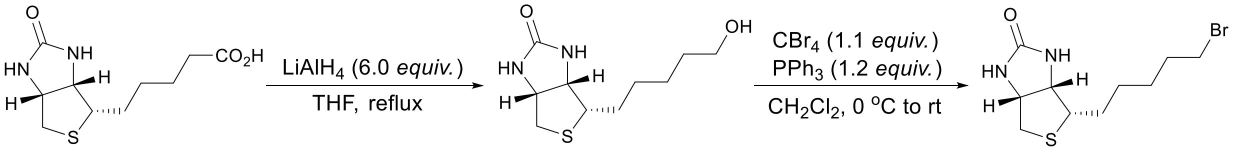
^{13}C NMR (100 MHz, CDCl$_3$) δ 112.3, 106.4, 101.4, 83.8, 81.5, 75.0, 71.9, 33.1, 27.1, 26.5, 23.9, 23.8.

IR (neat, cm^{-1}): 2988, 2937, 1379, 1221, 1161, 1076, 1023, 886, 792.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{12}H_{19}BrNaO_5$ 345.0308, found 345.0303.



(3a*S*,4*S*,6a*R*)-4-(5-Bromopentyl)tetrahydro-1*H*-thieno[3,4-*d*]imidazol-2(3*H*)-one (**19**, known compound)

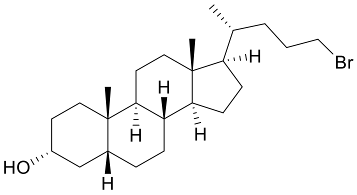


Step 1: Following the literature procedure[22], the solution of D-Biotin (1.22 g, 5.0 mmol) in dry THF (60 mL) was added dropwise to a suspension of powdered $LiAlH_4$ (1.14 g, 30.0 mmol) in dry THF (30 mL) at room temperature. The reaction mixture was refluxed and stirred overnight. To this mixture was carefully added 60 mL of water at room temperature and 50 mL of H_2SO_4 (1 M), stirring for 1 h. The mixture was extracted with EtOAc (20 × 60 mL). After drying with anhydrous Na_2SO_4 and evaporating the solvent, the alcohol was obtained as a white solid (0.98 g, 85% yield) and directly used without further purification.

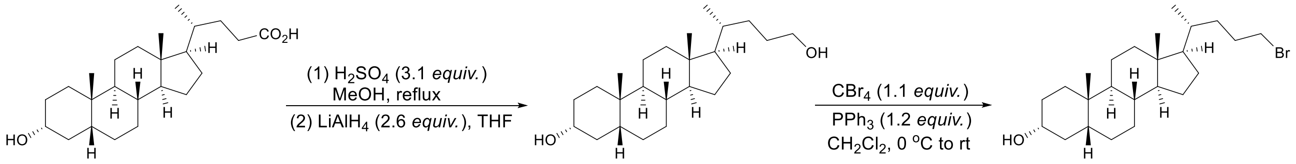
Step 2: To a solution of the above alcohol (0.69 g, 3.0 mmol) and PPh_3 (0.95 g, 3.6 mmol) in CH_2Cl_2 (20 mL) was added CBr_4 (1.10 g, 3.3 mmol) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred overnight. The reaction was then quenched with water and the mixture extracted twice with CH_2Cl_2. The combine organic layers was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (0.67 g, 69% yield). **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 23.

^{1}H NMR (400 MHz, CD$_3$OD) δ 4.59 (dd, J_1 = 7.6 Hz, J_2 = 4.8 Hz, 1 H), 4.40 (dd, J_1 = 8.0 Hz, J_2 = 4.4 Hz, 1 H), 3.55 (t, J = 6.8 Hz, 2 H), 3.41 (s, 2 H), 3.03 (dd, J_1 = 12.8 Hz, J_2 = 4.8 Hz, 1 H), 2.80 (d, J = 12.8 Hz, 1 H), 2.00-1.93 (m, 2 H), 1.88-1.80 (m, 1H), 1.73-1.50 (m, 5 H).

^{13}C NMR (100 MHz, CD$_3$OD) δ 166.3, 63.6, 61.8, 57.3, 41.2, 34.5, 34.0, 29.8, 29.7, 29.3.



(3*R*,5*R*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-17-((*R*)-5-Bromopentan-2-yl)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-ol (**21**)



Step 1: To a solution of Lithocholic acid (1.88 g, 5 mmol) in MeOH (15 mL) was added H_2SO_4 (0.9 mL) and the mixture was refluxed for 2 h. After removing the volatiles, the crude mixture was extracted with EtOAc (2 × 20 ml). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The resulting solid was then dissolved in THF (20 mL) and $LiAlH_4$ (0.49 g, 13.0 mmol) was slowly added. After stirring for 2 h, the reaction was quenched by addition of water (20 mL) and the crude mixture was extracted with EtOAc (2 × 20 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give alcohol as a white solid (1.69 g, 93% yield).

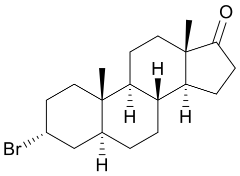
Step 2: To a solution of the above alcohol (1.09 g, 3.0 mmol) and PPh_3 (0.95 g, 3.6 mmol) in CH_2Cl_2 (20 mL) was added CBr_4 (1.10 g, 3.3 mmol) at 0 ^{o}C. The reaction mixture was then allowed to warm to room temperature and stirred overnight. The reaction was then quenched with water and the mixture was extracted twice with CH_2Cl_2. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (0.83 g, 65% yield). Mp. 136-138 ^{o}C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 3.66-3.60 (m, 1 H), 3.42-3.35 (m, 2 H), 1.97-1.72 (m, 7 H), 1.68-1.64 (m, 2 H), 1.57-1.48 (m, 3 H), 1.42-1.32 (m, 7 H), 1.27-1.00 (m, 10 H), 0.93-0.91 (m, 6 H), 0.64 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 71.8, 56.4, 55.9, 42.6, 42.0, 40.3, 40.1, 36.4, 35.8, 35.3, 35.2, 34.6, 34.5, 34.4, 30.5, 29.5, 28.2, 27.1, 26.4, 24.2, 23.3, 20.8, 18.6, 12.0.

IR (neat, cm^{-1}): 3311, 2934, 2862, 1576, 1436, 1038, 734.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{24}H_{41}BrNaO$ 447.2233, found 447.2223.



(3*R*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-3-Bromo-10,13-dimethylhexadecahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (**23**, known compound)

This compound was synthesized according to the literature procedure.[24] To a solution of PPh_3 (0.66 g, 2.5 mmol) in CH_2Cl_2 (24 mL) was dropwise added bromine (77 μL, 1.5 mmol) at 0 ^{o}C. Et_3N (0.21 mL, 1.5 mmol), and *p*-toluenesulfonyl chloride (42 mg, 0.2 mmol) were then added and the reaction. After stirring for 10 minutes, a solution of *trans*-androsterone (0.33 g, 1.1 mmol) in CH_2Cl_2 (5 mL) was dropwise added and the reaction mixture was allowed to stir at room temperature overnight. The reaction was quenched with water, extracted twice with EtOAc. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the title product as a white solid (0.34 g, 88% yield). Mp. 127-129 ^{o}C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 24.

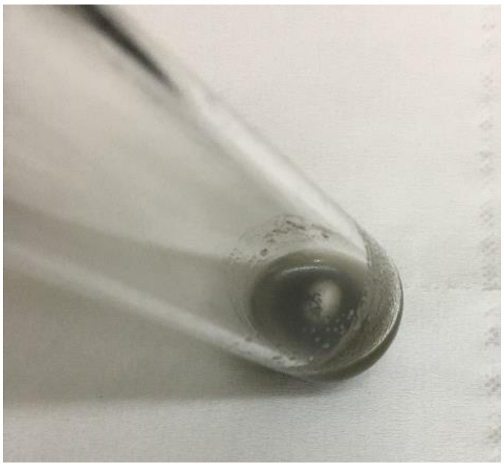
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.73 (m, 1 H), 2.44 (dd, J_1 = 8.8 Hz, J_2 = 8.4 Hz, 1 H), 2.12-2.03 (m, 1 H), 1.97-1.91 (m, 3 H), 1.83-1.76 (m, 4 H), 1.73-1.68 (m, 2 H), 1.62-1.45 (m, 4 H), 1.35-1.22 (m, 5 H), 1.12-1.01 (m, 1 H), 0.92-0.86 (m, 4 H), 0.82 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 221.0, 55.5, 53.8, 51.2, 47.6, 40.0, 37.0, 36.2, 35.7, 34.8, 32.7, 31.3, 30.8, 30.5, 27.4, 21.6, 19.9, 13.7, 12.2.

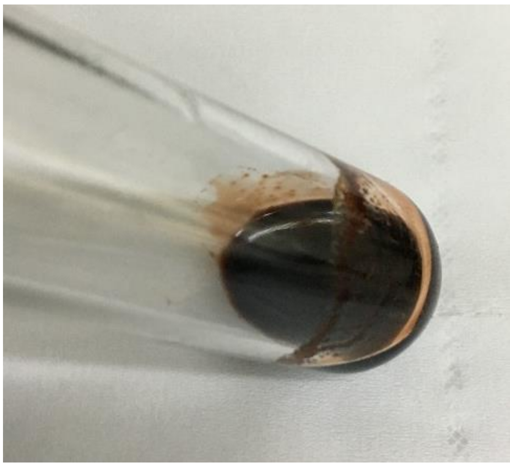
5. Cross-Coupling of Benzyl Oxalates with Alkyl Bromides

5.1 General Procedure

To an oven-dried reaction tube containing $NiBr_2$ (4.4 mg, 10 mol%), P(4-CF_3Ph)$_3$ (28 mg, 30 mol%), Mn (44 mg, 4.0 equiv.) and oxalates (0.2 mmol) was added a solution of alkyl bromide (0.4 mmol) in DMSO/DMF (1:1, 0.5 mL) or DMSO (0.5 mL). The reaction mixture was then frozen in a liquid nitrogen bath, vacuumed and refilled with argon for three times. The mixture was stirred at 30 ^{o}C for 24 h and then quenched with water (20 mL), extracted with ethyl acetate (3 × 10 mL). The combined organic layers were washed with brine (20 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated to dryness. The crude residue was purified by silica gel column to give the desired product.

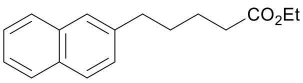


before reaction



after 6 h

5.2 Characterization Data



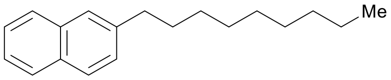
Ethyl 5-(naphthalen-2-yl)pentanoate (**3a**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 25.

First run: 38.2 mg, 74.6% yield. **Second run**: 38.3 mg, 74.8% yield. **Third run**: 40.4 mg, 78.9% yield (*DMSO as the solvent*).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.46-7.38 (m, 2 H), 7.31 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 4.11 (q, J = 7.2 Hz, 2 H), 2.79 (t, J = 7.2 Hz, 2 H), 2.33 (t, J = 7.2 Hz, 2 H), 1.78-1.66 (m, 4 H), 1.23 (t, J = 7.2 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 139.6, 133.6, 132.0, 127.8, 127.6, 127.4, 127.2, 126.3, 125.8, 125.1, 60.2, 35.7, 34.2, 30.7, 24.6, 14.2.



2-Nonylnaphthalene (**3b**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil.

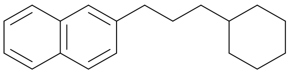
First run: 39.2 mg, 77.2% yield. **Second run**: 38.6 mg, 76.0% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.45-7.38 (m, 2 H), 7.32 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 2.76 (t, J = 7.6 Hz, 2 H), 1.73-1.66 (m, 2 H), 1.33-1.26 (m, 12 H), 0,87 (t, J = 6.8 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 133.6, 131.9, 127.7, 127.6, 127.5, 127.4, 126.3, 125.8, 125.0, 36.1, 31.9, 31.4, 29.6, 29.4, 29.3, 22.7, 14.1.

IR (neat, cm^{-1}): 2925, 2853, 2359, 1464, 814, 744.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{19}H_{27}$ 255.2107, found 255.2109.



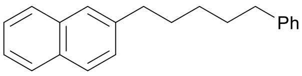
2-(3-Cyclohexylpropyl)naphthalene (**3c**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 26.

First run: 41.7 mg, 82.7% yield. **Second run**: 43.1 mg, 85.6% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.74 (m, 3 H), 7.59 (s, 1 H), 7.45-7.37 (m, 2 H), 7.32 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 2.73 (t, J = 7.6 Hz, 2 H), 1.72-1.62 (m, 7 H), 1.26-1.10 (m, 6 H), 0.90-0.84 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 133.6, 131.9, 127.7, 127.6, 127.4, 127.4, 126.2, 125.8, 124.9, 37.6, 37.2, 36.4, 33.4, 28.7, 26.7, 26.4.



2-(5-Phenylpentyl)naphthalene (**3d**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil.

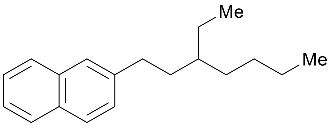
First run: 41.7 mg, 75.8% yield. **Second run**: 42.0 mg, 76.6% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.59 (s, 1 H), 7.46-7.38 (m, 2 H), 7.32-7.24 (m, 3 H), 7.18-7.15 (m, 3 H), 2.76 (t, *J* = 7.6 Hz, 2 H), 2.60 (t, *J* = 7.6 Hz, 2 H), 1.77-1.63 (m, 4 H), 1.46-1.38 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 142.7, 140.3, 133.6, 131.9, 128.4, 128.2, 127.38, 127.37, 127.6, 127.4, 126.3, 125.8, 125.6, 125.0, 36.0, 35.9, 31.4, 31.2, 28.9.

IR (neat, cm^{-1}): 2928, 2854, 2360, 1507, 1455, 853, 816, 745, 698.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{23}$ 275.1794, found 275.1794.



2-(3-Ethylheptyl)naphthalene (**3e**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil.

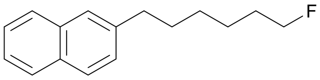
First run: 31.8 mg, 62.5% yield. **Second run**: 30.0 mg, 59.2% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.61 (s, 1 H), 7.45-7.38 (m, 2 H), 7.33 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 2.76-2.72 (m, 2 H), 1.67-1.62 (m, 2 H), 1.40-1.29 (m, 9 H), 0.92-0.86 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.8, 133.7, 131.9, 127.7, 127.6, 127.4, 127.3, 126.1, 125.8, 124.9, 38.6, 35.1, 33.3, 32.7, 28.9, 25.8, 23.2, 14.2, 10.8.

IR (neat, cm^{-1}): 2957, 2925, 2360, 1507, 1457, 851, 814, 743.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{19}H_{27}$ 255.2107, found 255.2110.



2-(6-Fluorohexyl)naphthalene (**3f**)

The title compound was prepared according to the General Procedure. The reaction was

conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a yellow oil.

First run: 30.4 mg, 66.0% yield. **Second run**: 29.9 mg, 64.7% yield.

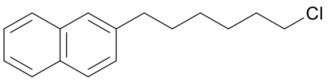
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.75 (m, 3 H), 7.60 (s, 1 H), 7.46-7.39 (m, 2 H), 7.33 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 4.43 (dt, J = 47.0, 6.0 Hz, 2 H), 2.78 (t, J = 7.2 Hz, 2 H), 1.76-1.63 (m, 4 H), 1.49-1.36 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.1, 133.6, 131.9, 127.8, 127.6, 127.37, 127.35, 126.3, 125.8, 125.0, 84.2 (d, $J_{C–F}$ = 163.0 Hz), 35.9, 31.2, 30.3 (d, $J_{C–F}$ = 19.0 Hz), 28.9, 25.0 (d, $J_{C–F}$ = 5.0 Hz).

^{19}F NMR (376 MHz, CDCl$_3$) δ -218.11.

IR (neat, cm^{-1}): 2360, 1734, 1717, 1699, 1684, 1653, 1635, 1558, 1540, 1507, 1457.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{16}H_{20}F$ 231.1544, found 231.1542.



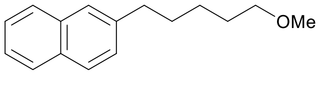
2-(6-Chlorohexyl)naphthalene (**3g**, known compound).

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a light yellow oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 27.

First run: 30.3 mg, 61.5% yield. **Second run**: 31.2 mg, 63.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.75 (m, 3 H), 7.59 (s, 1 H), 7.45-7.38 (m, 2 H), 7.31 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 3.51 (t, J = 6.8 Hz, 2 H), 2.77 (t, J = 7.2 Hz, 2 H), 1.79-1.67 (m, 4 H), 1.51-1.41 (m, 2 H), 1.39-1.34 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.0, 133.6, 131.9, 127.8, 127.6, 127.4, 127.3, 126.3, 125.8, 125.0, 45.1, 35.9, 32.5, 31.1, 28.5, 26.7.



2-(5-Methoxypentyl)naphthalene (**3h**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (200:1 petroleum ether/ethyl acetate) to give the product as a light yellow oil.

First run: 35.8 mg, 78.5% yield. **Second run**: 37.5 mg, 82.2% yield.

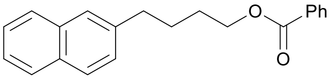
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.45-7.38 (m, 2 H), 7.32 (dd, J_1

= 1.6 Hz, J_2 = 8.4 Hz, 1 H), 3.36 (t, J = 6.4 Hz, 2 H), 3.32 (s, 3 H), 2.77 (t, J = 7.6 Hz, 2 H), 1.76-1.68 (m, 2 H), 1.65-1.58 (m, 2 H), 1.46-1.38 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1, 133.6, 131.9, 127.7, 127.6, 127.4, 127.4, 126.3, 125.8, 125.0, 72.8, 58.5, 36.0, 31.2, 29.5, 25.8.

IR (neat, cm^{-1}): 2930, 2856, 1507, 1457, 1118, 816, 746.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{16}H_{21}O$ 229.1587, found 229.1586.



4-(Naphthalen-2-yl)butyl benzoate (**3i**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (200:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

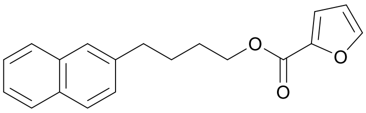
First run: 37.6 mg, 61.8% yield. **Second run**: 35.5 mg, 58.4% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 7.6 Hz, 2 H), 7.81-7.76 (m, 3 H), 7.62 (s, 1 H), 7.56-7.52 (m, 1 H), 7.46-7.39 (m, 4 H), 7.34 (d, J = 8.4 Hz, 1 H), 4.36 (t, J = 6.4 Hz, 2 H), 2.85 (t, J = 6.8 Hz, 2 H), 1.89-1.86 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 139.5, 133.6, 132.8, 132.0, 130.4, 129.5, 128.3, 127.9, 127.6, 127.4, 127.2, 126.4, 125.9, 125.1, 64.8, 35.6, 28.3, 27.6.

IR (neat, cm^{-1}): 2928, 1717, 1601, 1508, 1451, 1314, 1273, 1175, 1114, 1026, 855, 817, 746, 711.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{21}H_{20}NaO_2$ 327.1356, found 327.1353.



4-(Naphthalen-2-yl)butyl furan-2-carboxylate (**3j**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 to 30:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

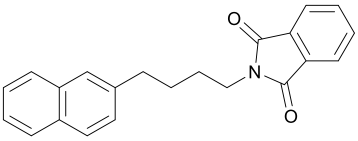
First run: 38.3 mg, 65.1% yield. **Second run**: 38.0 mg, 64.6% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81-7.76 (m, 3 H), 7.62 (s, 1 H), 7.56 (d, J = 0.8 Hz, 1 H), 7.47-7.39 (m, 2 H), 7.33 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 7.16 (d, J = 3.6 Hz, 1 H), 6.49 (dd, J_1 = 1.6 Hz, J_2 = 7.6 Hz, 1 H), 4.34 (t, J = 6.4 Hz, 2 H), 2.84 (t, J = 7.2 Hz, 2 H), 1.89-1.77 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 146.2, 144.8, 139.4, 133.5, 132.0, 127.9, 127.6, 127.4, 127.2, 126.4, 125.9, 125.1, 117.8, 111.8, 64.8, 35.5, 28.2, 27.5.

IR (neat, cm^{-1}): 2928, 1725, 1475, 1296, 1179, 1119, 762.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{19}H_{18}NaO_3$ 317.1148, found 317.1146.



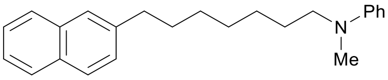
2-(4-(Naphthalen-2-yl)butyl)isoindoline-1,3-dione (**3k**, known compound).

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (20:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 101-103 °C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 28.

First run: 37.9 mg, 57.6% yield. **Second run**: 36.3 mg, 55.2% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.83-7.73 (m, 5 H), 7.69-7.67 (m, 2 H), 7.60 (s, 1 H), 7.44-7.38 (m, 2 H), 7.32-7.30 (m, 1 H), 3.74-3.73 (m, 2 H), 2.82 (m, 2 H), 1.76-1.74 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 168.4, 139.4, 133.8, 133.6, 132.1, 132.0, 127.9, 127.6, 127.4, 127.2, 126.4, 125.8, 125.1, 123.1, 37.8, 35.5, 28.5, 28.2.



***N*-Methyl-*N*-(7-(naphthalen-2-yl)heptyl)aniline** (**3l**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (300:1 petroleum ether/ethyl acetate) to give the product as a light yellow oil.

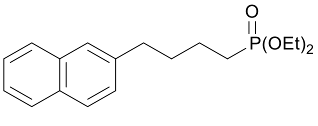
First run: 39.6 mg, 59.8% yield. **Second run**: 40.1 mg, 60.6% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.75 (m, 3 H), 7.60 (s, 1 H), 7.46-7.39 (m, 2 H), 7.32 (d, J = 8.4 Hz, 1 H), 7.23-7.19 (m, 2 H), 6.69-6.65 (m, 3 H), 3.28 (t, J = 7.6 Hz, 2 H), 2.90 (s, 3 H), 2.76 (t, J = 7.6 Hz, 2 H), 1.70 (m, 2 H), 1.57-1.54 (m, 2 H), 1.37 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 149.4, 140.3, 133.6, 131.9, 129.1, 127.7, 127.6, 127.39, 127.37, 126.3, 125.8, 125.0, 115.8, 112.1, 52.8, 38.3, 36.1, 31.3, 29.4, 29.3, 27.1, 26.6.

IR (neat, cm^{-1}): 2925, 2853, 2360, 1598, 1506, 814, 746, 690.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{24}H_{30}N$ 332.2373, found 332.2375.



Diethyl (4-(naphthalen-2-yl)butyl)phosphonate (3m)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (4:1 to 1:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

First run: 50.3 mg, 78.6% yield. **Second run**: 52.9 mg, 82.7% yield.

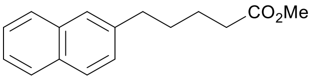
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.75 (m, 3 H), 7.60 (s, 1 H), 7.46-7.39 (m, 2 H), 7.31 (d, J = 8.4 Hz, 1 H), 4.11-4.03 (m, 4 H), 2.79 (t, J = 7.2 Hz, 2 H), 1.84-1.67 (m, 6 H), 1.29 (t, J = 7.2 Hz, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 133.5, 131.9, 127.8, 127.5, 127.3, 127.2, 126.3, 125.8, 125.1, 61.4 (d, J_{C-P} = 6.5 Hz), 35.5, 32.1 (d, J_{C-P} = 16.7 Hz), 25.5 (d, J_{C-P} = 139.9 Hz), 22.1 (d, J_{C-P} = 5.0 Hz), 16.4 (d, J_{C-P} = 6.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ 32.21 ppm.

IR (neat, cm^{-1}): 2930, 1243, 1025, 958, 816.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{18}H_{26}O_3P$ 321.1614, found 321.1613.



Methyl 5-(naphthalen-2-yl)pentanoate (3n)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a light yellow oil.

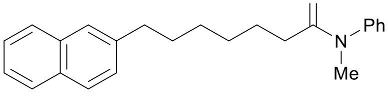
First run: 37.5 mg, 77.5% yield. **Second run**: 37.0 mg, 76.4% yield. **Third run**: 38.8 mg, 80.2% yield (*DMSO as the solvent*).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.46-7.38 (m, 2 H), 7.31 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 3.65 (s, 3 H), 2.78 (t, J = 7.2 Hz, 2 H), 2.34 (t, J = 7.2 Hz, 2 H), 1.78-1.66 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 139.6, 133.6, 131.9, 127.8, 127.6, 127.4, 127.2, 126.3, 125.8, 125.1, 51.5, 35.7, 33.9, 30.7, 24.5.

IR (neat, cm^{-1}): 2947, 1737, 1435, 1172, 816, 747.

HRMS (ESI): $[M+Na]^+$ calcd. for $C_{16}H_{18}NaO_2$ 265.1199, found 265.1192.



N-Methyl-7-(naphthalen-2-yl)-*N*-phenylheptanamide (3o)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

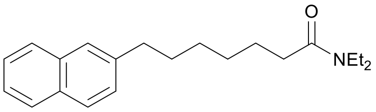
First run: 45.3 mg, 65.7% yield. **Second run**: 45.9 mg, 66.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.73 (m, 3 H), 7.57 (s, 1 H), 7.46-7.36 (m, 4 H), 7.33-7.28 (m, 2 H), 7.14 (d, J = 7.6 Hz, 2 H), 3.25 (s, 3 H), 2.71 (t, J = 7.6 Hz, 2 H), 2.05 (t, J = 7.2 Hz, 2 H), 1.67-1.55 (m, 4 H), 1.25 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.2, 144.3, 140.2, 133.6, 131.9, 129.6, 127.7, 127.6, 127.5, 127.4, 127.34, 127.27, 126.2, 125.8, 124.9, 37.3, 36.0, 34.0, 31.1, 29.1, 28.9, 25.4.

IR (neat, cm^{-1}): 2925, 2853, 2360, 1655, 1595, 1495, 1458, 1381, 1118, 816, 700.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{24}H_{28}NO$ 346.2165, found 346.2168.



***N,N*-Diethyl-7-(naphthalen-2-yl)heptanamide** (**3p**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

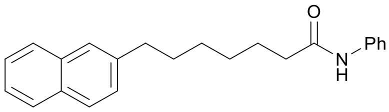
First run: 47.8 mg, 76.8% yield. **Second run**: 49.4 mg, 79.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.46-7.38 (m, 2 H), 7.32 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 3.36 (q, J = 7.2 Hz, 2 H), 3.26 (q, J = 7.2 Hz, 2 H), 2.77 (t, J = 7.6 Hz, 2 H), 2.67 (t, J = 7.6 Hz, 2 H), 1.74-1.62 (m, 4 H), 1.40-1.38 (m, 4 H), 1.15-1.08 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 172.1, 140.2, 133.6, 131.9, 127.7, 127.5, 127.4, 127.3, 126.2, 125.7, 124.9, 41.9, 39.9, 36.0, 33.0, 31.1, 29.3, 29.1, 25.3, 14.3, 13.1.

IR (neat, cm^{-1}): 2927, 1641, 1460, 816.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{30}NO$ 312.2322, found 312.2322.



7-(Naphthalen-2-yl)-*N*-phenylheptanamide (**3q**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (20:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 97-99 ^{o}C.

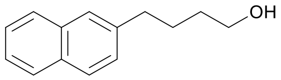
First run: 49.1 mg, 74.2% yield. **Second run**: 46.9 mg, 70.9% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.73 (m, 3 H), 7.58 (s, 1 H), 7.50-7.48 (m, 2 H), 7.45-7.38 (m, 2 H), 7.32-7.27 (m, 4 H), 7.07 (t, *J* = 7.2 Hz, 1 H), 2.75 (t, *J* = 7.6 Hz, 2 H), 2.30 (t, *J* = 7.6 Hz, 2 H), 1.72-1.65 (m, 4 H), 1.40-1.38 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 171.3, 140.1, 137.9, 133.6, 131.9, 128.9, 127.7, 127.6, 127.4, 126.3, 125.8, 125.0, 124.1, 119.8, 37.7, 35.9, 31.0, 29.1, 28.9, 25.5.

IR (neat, cm^{-1}): 3299, 3053, 2929, 2855, 1660, 1600, 1544, 1499, 1442, 1309, 1252, 817, 754, 692.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{23}H_{26}NO$ 332.2009, found 332.2003.



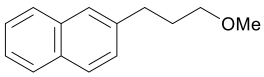
4-(Naphthalen-2-yl)butan-1-ol (**3r**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (100:1 to 10:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 34-36 °C. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 29.

First run: 25.2 mg, 62.5% yield. **Second run**: 24.0 mg, 59.9% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.76 (m, 3 H), 7.62 (s, 1 H), 7.47-7.40 (m, 2 H), 7.33 (d, *J* = 7.6 Hz, 1 H), 3.68 (t, *J* = 6.4 Hz, 2 H), 2.81 (t, *J* = 7.6 Hz, 2 H), 1.83-1.75 (m, 2 H), 1.68-1.61 (m, 2 H), 1.30 (s, 1 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.8, 133.6, 132.0, 127.8, 127.6, 127.4, 127.3, 126.4, 125.9, 125.1, 62.8, 35.7, 32.3, 27.4.



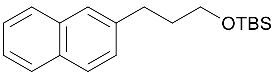
2-(3-Methoxypropyl)naphthalene (**3s**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (300:1 petroleum ether/ethyl acetate) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 30.

First run: 33.5 mg, 83.8% yield. **Second run**: 30.9 mg, 77.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.79-7.74 (m, 3 H), 7.61 (s, 1 H), 7.45-7.38 (m, 2 H), 7.32 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 3.39 (t, *J* = 6.4 Hz, 2 H), 3.33 (s, 3 H), 2.84 (t, *J* = 7.2 Hz, 2 H), 2.00-1.93 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.4, 133.6, 131.9, 127.8, 127.5, 127.4, 127.3, 126.4, 125.8, 125.1, 71.8, 58.5, 32.4, 31.1.



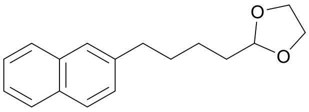
***tert*-Butyldimethyl(3-(naphthalen-2-yl)propoxy)silane** (**3t**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 °C for 24 h. The crude residue was purified by flash chromatography (300:1 petroleum ether/ethyl acetate) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 31.

First run: 45.1 mg, 75.2% yield. **Second run**: 49.4 mg, 82.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.85-7.79 (m, 3 H), 7.66 (s, 1 H), 7.50-7.42 (m, 2 H), 7.38 (dd, J_1 = 2.0 Hz, J_2 = 8.4 Hz, 1 H), 3.71 (t, J = 6.4 Hz, 2 H), 2.88 (t, J = 7.6 Hz, 2 H), 2.00-1.93 (m, 2 H), 0.97 (s, 9 H), 0.11 (s, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.7, 133.6, 131.9, 127.8, 127.6, 127.43, 127.37, 126.4, 125.8, 125.0, 62.3, 34.3, 32.2, 26.0, 18.3, -5.3.



2-(4-(Naphthalen-2-yl)butyl)-1,3-dioxolane (**3u**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 °C for 24 h. The crude residue was purified by flash chromatography (100:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

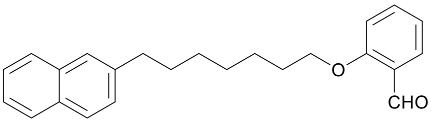
First run: 37.1 mg, 72.5% yield. **Second run**: 37.6 mg, 73.4% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.60 (s, 1 H), 7.45-7.38 (m, 2 H), 7.32 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 4.83 (t, J = 4.8 Hz, 1 H), 3.98-3.90 (m, 2 H), 3.86-3.78 (m, 2 H), 2.78 (t, J = 7.6 Hz, 2 H), 1.79-1.68 (m, 4 H), 1.54-1.46 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.0, 133.6, 131.9, 127.7, 127.5, 127.4, 127.3, 126.3, 125.8, 125.0, 104.5, 64.8, 36.0, 33.7, 31.3, 23.8.

IR (neat, cm^{-1}): 2943, 1507, 1409, 1132, 1032, 945, 852, 817, 748.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{17}H_{20}NaO_2$ 279.1356, found 279.1357.



2-((7-(Naphthalen-2-yl)heptyl)oxy)benzaldehyde (**3v**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (200:1 to 100:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

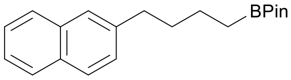
First run: 30.9 mg, 44.7% yield. **Second run**: 32.5 mg, 46.9% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 10.51 (d, *J* = 0.8 Hz, 1 H), 7.84-7.75 (m, 4 H), 7.60 (s, 1 H), 7.53-7.48 (m, 1 H), 7.46-7.38 (m, 2 H), 7.32 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 6.99 (t, *J* = 7.6 Hz, 1 H), 6.94 (d, *J* = 8.4 Hz, 1 H), 4.04 (t, *J* = 6.4 Hz, 2 H), 2.77 (t, *J* = 7.6 Hz, 2 H), 1.86-1.81 (m, 2 H), 1.79-1.68 (m, 2 H), 1.50-1.38 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 189.9, 161.5, 140.2, 135.9, 133.6, 131.9, 128.2, 127.7, 127.6, 127.4, 127.4, 126.3, 125.8, 125.0, 124.9, 120.4, 112.5, 68.5, 36.0, 31.2, 29.2, 29.2, 29.0, 26.0.

IR (neat, cm^{-1}): 2929, 2855, 1687, 1599, 1458, 1386, 1286, 1242, 1160, 1102, 817, 756.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{24}H_{27}O_2$ 347.2006, found 347.1999.



4,4,5,5-Tetramethyl-2-(4-(naphthalen-2-yl)butyl)-1,3,2-dioxaborolane (**3w**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a light yellow oil.

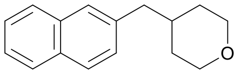
First run: 37.8 mg, 61.0% yield. **Second run**: 36.3 mg, 58.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.79-7.73 (m, 3 H), 7.60 (s, 1 H), 7.44-7.37 (m, 2 H), 7.32 (d, *J* = 8.4 Hz, 1 H), 2.77 (t, *J* = 7.6 Hz, 2 H), 1.76-1.68 (m, 2 H), 1.55-1.47 (m, 2 H), 1.23 (s, 12H), 0.84 (t, *J* = 7.6 Hz, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.4, 133.6, 131.9, 127.7, 127.5, 127.44, 127.36, 126.2, 125.7, 124.9, 82.9, 35.9, 34.0, 24.8, 23.7.

IR (neat, cm^{-1}): 2929, 1377, 1321, 1145, 967, 816, 745.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{20}H_{27}BNaO_2$ 333.1996, found 333.1995.



4-(Naphthalen-2-ylmethyl)tetrahydro-*2H*-pyran (**3x**)

The title compound was prepared according to the General Procedure but DMSO was used as a solvent. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (200:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

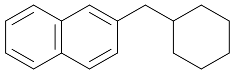
First run: 29.2 mg, 64.6% yield. **Second run**: 28.2 mg, 62.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.75 (m, 3 H), 7.58 (s, 1 H), 7.47-7.39 (m, 2 H), 7.29 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 3.94 (dd, J_1 = 3.6 Hz, J_2 = 10.8 Hz, 2 H), 3.36-3.30 (m, 2 H), 2.70 (d, J = 7.2 Hz, 2 H), 1.91-1.80 (m, 1 H), 1.59-1.56 (m, 2 H), 1.43-1.33 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 137.6, 133.5, 132.0, 127.75, 127.74, 127.6, 127.4, 127.3, 125.9, 125.2, 68.0, 43.6, 37.1, 33.0.

IR (neat, cm^{-1}): 2923, 2842, 1599, 1508, 1445, 1385, 1232, 1134, 1090, 1014, 982, 857, 821, 794, 746.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{16}H_{19}O$ 227.1430, found 227.1427.



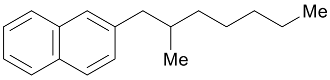
2-(Cyclohexylmethyl)naphthalene (**3y**, known compound).

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 10.

First run: 26.7 mg, 59.6% yield. **Second run**: 27.8 mg, 62.0% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.56 (s, 1 H), 7.45-7.38 (m, 2 H), 7.30 (dd, J_1 = 0.8 Hz, J_2 = 8.4 Hz, 1 H), 2.64 (d, J = 7.2 Hz, 2 H), 1.71-1.57 (m, 6 H), 1.25-1.13 (m, 3 H), 1.03-0.94 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 138.9, 133.5, 131.9, 128.0, 127.6, 127.5, 127.4, 127.2, 125.8, 124.9, 44.3, 39.8, 33.2, 26.6, 26.3.



2-(2-Methylheptyl)naphthalene (**3z**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil.

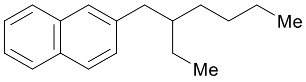
First run: 25.0 mg, 52.1% yield. **Second run**: 23.4 mg, 48.7% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.57 (s, 1 H), 7.46-7.38 (m, 2 H), 7.30 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 2.79 (dd, J_1 = 6.0 Hz, J_2 = 13.6 Hz, 1 H), 2.50 (dd, J_1 = 8.4 Hz, J_2 = 13.2 Hz, 1 H), 1.83-1.79 (m, 1 H), 1.39-1.16 (m, 8 H), 0.90-0.86 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.3, 133.5, 131.9, 128.0, 127.6, 127.5, 127.4, 127.3, 125.7, 125.0, 43.9, 36.8, 35.0, 32.1, 26.8, 22.7, 19.5, 14.1.

IR (neat, cm^{-1}): 2925, 2854, 1508, 1460, 1376, 809, 746.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{18}H_{25}$ 241.1951, found 241.1946.



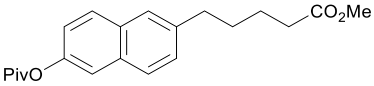
2-(2-Ethylhexyl)naphthalene (**3aa**, known compound)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil. **^{1}H NMR** and **^{13}C NMR** data are consistent with those reported in ref. 32.

First run: 20.6 mg, 43% yield. **Second run**: 23.1 mg, 48.1% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81-7.70 (m, 3 H), 7.58 (s, 1 H), 7.47-7.39 (m, 2 H), 7.31 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 2.73-2.64 (m, 2 H), 1.71-1.65 (m, 1 H), 1.36-1.25 (m, 8 H), 0.91-0.86 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.5, 133.5, 131.8, 128.0, 127.6, 127.5, 127.4, 127.3, 125.7, 124.9, 41.0, 40.3, 32.3, 28.8, 25.3, 23.0, 14.2, 10.8.



Methyl 5-(6-(pivaloyloxy)naphthalen-2-yl)pentanoate (**3ab**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 to 30:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 75-77 °C.

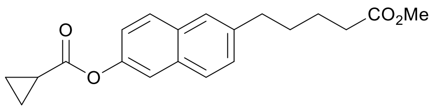
First run: 53.2 mg, 77.8% yield. **Second run**: 54.4 mg, 79.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.76 (d, J = 9.2 Hz, 1 H), 7.70 (d, J = 8.4 Hz, 1 H), 7.59 (s, 1 H), 7.47 (d, J = 2.0 Hz, 1 H), 7.31 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 7.15 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 3.65 (s, 3 H), 2.77 (t, J = 7.2 Hz, 2 H), 2.34 (t, J = 7.2 Hz, 2 H), 1.77-1.65 (m, 4 H), 1.39 (s, 9 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 177.2, 173.9, 148.2, 139.3, 132.2, 131.5, 128.7, 127.9, 127.5, 126.2, 121.1, 118.1, 51.4, 39.0, 35.5, 33.9, 30.6, 27.1, 24.5.

IR (neat, cm^{-1}): 2937, 1747, 1607, 1475, 1367, 1279, 1142, 1109, 1029, 905, 813, 735, 653.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{21}H_{26}NaO_4$ 365.1723, found 365.1717.



6-(5-Methoxy-5-oxopentyl)naphthalen-2-yl cyclopropanecarboxylate (**3ac**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 100-102 °C.

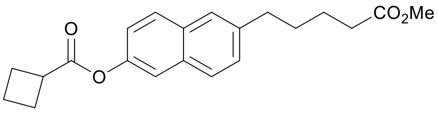
First run: 54.4 mg, 83.4% yield. **Second run**: 53.0 mg, 81.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.75 (d, J = 8.8 Hz, 1 H), 7.70 (d, J = 8.4 Hz, 1 H), 7.59 (s, 1 H), 7.51 (d, J = 2.0 Hz, 1 H), 7.31 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 7.20 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 3.65 (s, 3 H), 2.77 (t, J = 7.2 Hz, 2 H), 2.34 (t, J = 7.2 Hz, 2 H), 1.91-1.85 (m, 1 H), 1.77-1.65 (m, 4 H), 1.22-1.18 (m, 2 H), 1.05-1.00 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.0, 173.6, 147.9, 139.4, 132.2, 131.5, 128.7, 127.9, 127.6, 126.2, 121.2, 118.2, 51.4, 35.5, 33.9, 30.6, 24.5, 13.0, 9.2.

IR (neat, cm^{-1}): 3465, 2949, 1744, 1439, 1383, 1159, 891, 816, 737, 648.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{20}H_{23}O_4$ 327.1591, found 327.1586.



6-(5-Methoxy-5-oxopentyl)naphthalen-2-yl cyclobutanecarboxylate (**3ad**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 72-74 °C.

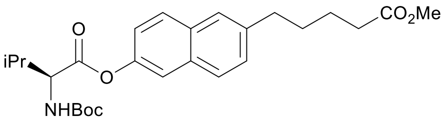
First run: 58.4 mg, 85.9% yield. **Second run**: 56.1 mg, 82.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.77 (d, J = 8.8 Hz, 1 H), 7.71 (d, J = 8.4 Hz, 1 H), 7.60 (s, 1 H), 7.50 (d, J = 2.0 Hz, 1 H), 7.32 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 7.18 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 3.66 (s, 3 H), 3.47-3.39 (m, 1 H), 2.78 (t, J = 7.2 Hz, 2 H), 2.53-2.43 (m, 2 H), 2.40-2.31 (m, 4 H), 2.13-1.96 (m, 2 H), 1.78-1.66 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.1, 174.0, 148.0, 139.4, 132.2, 131.6, 128.8, 127.9, 127.6, 126.3, 121.2, 118.2, 51.5, 38.2, 35.6, 33.9, 30.7, 25.3, 24.6, 18.4.

IR (neat, cm^{-1}): 2946, 1748, 1359, 1147, 895, 815.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{25}O_4$ 341.1747, found 341.1740.



Methyl 5-(6-(((*tert*-butoxycarbonyl)-*L*-valyl)oxy)naphthalen-2-yl)pentanoate (3ae)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (20:1 petroleum ether/ethyl acetate) to give the product as a light yellow oil.

First run: 83.2 mg, 91.0% yield. **Second run**: 81.6 mg, 89.3% yield.

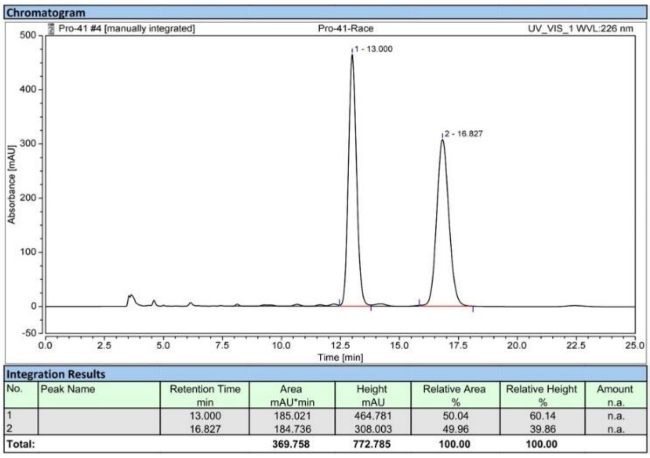
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.75 (d, *J* = 9.2 Hz, 1 H), 7.71 (d, *J* = 8.4 Hz, 1 H), 7.60 (s, 1 H), 7.51 (d, *J* = 2.0 Hz, 1 H), 7.33 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 1 H), 7.19 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1 H), 5.16-4.92 (m, 1 H), 4.54-4.29 (m, 1 H), 3.65 (s, 3 H), 2.78 (t, *J* = 7.2 Hz, 2 H), 2.40-2.33 (m, 3 H), 1.76-1.65 (m, 4H), 1.48 (s, 9H), 1.12 (d, *J* = 7.2 Hz, 3H), 1.06 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.9, 171.3, 155.7, 147.5, 139.6, 132.1, 131.7, 128.9, 128.1, 127.6, 126.2, 120.8, 118.1, 79.9, 58.7, 51.4, 35.5, 33.8, 31.4, 30.6, 28.3, 24.5, 19.1, 17.7.

IR (neat, cm^{-1}): 3382, 2966, 1717, 1606, 1507, 1366, 1151, 1088, 1015, 974, 880, 810, 734.

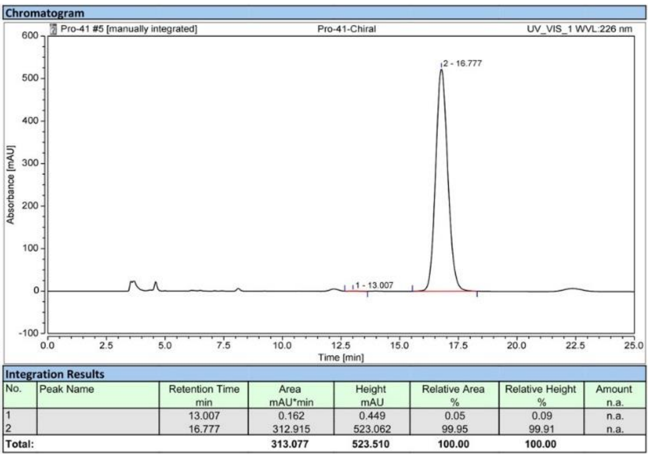
HRMS (ESI): [M+H]$^+$ calcd. for $C_{26}H_{36}NO_6$ 458.2537, found 458.2531.

HPLC analysis: 99.9% ee; Chiralpak IC column (Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, wave length = 226 nm), t_R = 13.003 min (minor), t_R = 16.777 min (major). $[\alpha]^{23}_D$ = -26.000 (c = 10.0 mg/mL, CH_2Cl_2).



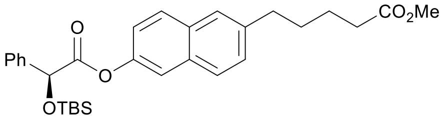
Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		13.000	185.021	464.781	50.04	60.14	n.a.
2		16.827	184.736	308.003	49.96	39.86	n.a.
Total:			369.758	772.785	100.00	100.00	



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		13.007	0.162	0.449	0.05	0.09	n.a.
2		16.777	312.915	523.062	99.95	99.91	n.a.
Total:			313.077	523.510	100.00	100.00	



Methyl (*S*)-5-(6-(2-((*tert*-butyldimethylsilyl)oxy)-2-phenylacetoxy)naphthalen-2-yl)pentanoate (3af)

The title compound was prepared according to the General Procedure. The reaction was

conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

First run: 87.9 mg, 86.9% yield. **Second run**: 90.5 mg, 89.4% yield.

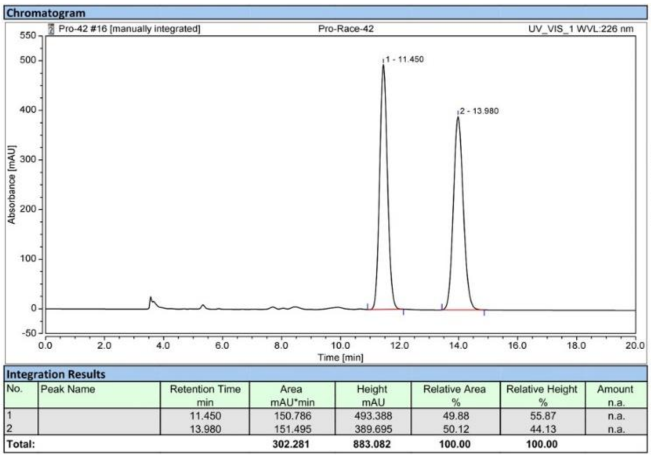
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.75-7.65 (m, 4 H), 7.59 (s, 1 H), 7.46-7.42 (m, 3 H), 7.40-7.36 (m, 1 H), 7.32 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 7.09 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 5.53 (s, 1 H), 3.67 (s, 3 H), 2.79 (t, J = 7.2 Hz, 2 H), 2.36 (t, J = 7.2 Hz, 2 H), 1.79-1.66 (m, 4 H), 1.00 (s, 9 H), 0.24 (s, 3 H), 0.15 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.0, 170.8, 147.7, 139.5, 138.7, 132.1, 131.6, 128.8, 128.5, 128.4, 128.0, 127.6, 126.5, 126.2, 120.6, 117.9, 74.6, 51.4, 35.6, 33.9, 30.6, 25.7, 24.5, 18.3, -4.9, -5.1.

IR (neat, cm^{-1}): 2930, 2857, 1738, 1606, 1462, 1361, 1145, 971, 872, 838, 780, 729, 696.

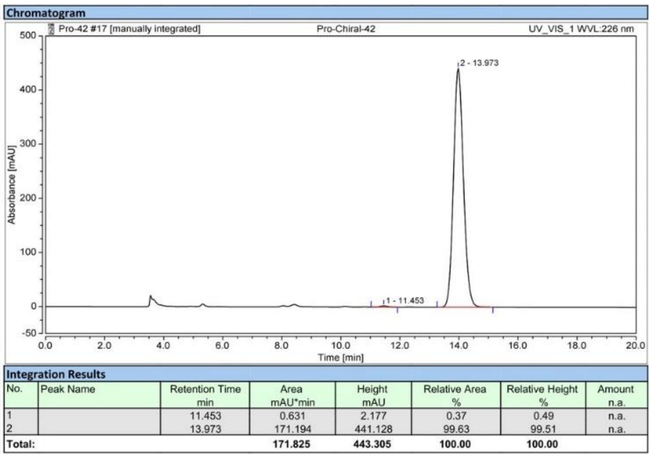
HRMS (ESI): [M+H]$^+$ calcd. for $C_{30}H_{39}O_5Si$ 507.2561, found 507.2552.

HPLC analysis: 99.2% ee; Chiralpak IC column (Hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, wave length = 226 nm), t_R = 11.453 min (minor), t_R = 13.973 min (major). $[\alpha]^{23}_D$ = +33.000 (c = 10.0 mg/mL, CH_2Cl_2).



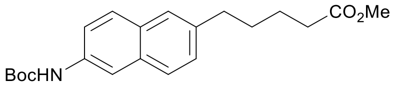
Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.450	150.786	493.388	49.88	55.87	n.a.
2		13.980	151.495	389.695	50.12	44.13	n.a.
Total:			302.281	883.082	100.00	100.00	



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.453	0.631	2.177	0.37	0.49	n.a.
2		13.973	171.194	441.128	99.63	99.51	n.a.
Total:			171.825	443.305	100.00	100.00	



Methyl 5-(6-((*tert*-butoxycarbonyl)amino)naphthalen-2-yl)pentanoate (**3ag**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (30:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

First run: 50.8 mg, 71.2% yield. **Second run**: 48.9 mg, 68.5% yield.

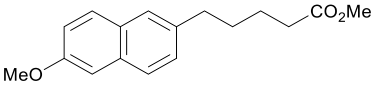
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 (s, 1 H), 7.66 (d, J = 8.4 Hz, 2 H), 7.50 (s, 1 H), 7.32-7.25

(m, 2 H), 6.72 (s, 1 H), 3.65 (s, 3 H), 2.74 (t, *J* = 6.8 Hz, 2 H), 2.34 (t, *J* = 6.8 Hz, 2 H), 1.76-1.66 (m, 4 H), 1.54 (s, 9 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.1, 152.9, 138.1, 135.2, 132.4, 130.1, 128.1, 127.8, 127.3, 126.0, 119.2, 114.4, 80.5, 51.4, 35.5, 33.9, 30.7, 28.3, 24.5.

IR (neat, cm^{-1}): 3346, 2933, 1727, 1611, 1540, 1498, 1367, 1239, 1159, 1050, 883, 812, 736.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{28}NO_4$ 358.2013, found 358.2007.



Methyl 5-(6-methoxynaphthalen-2-yl)pentanoate (**3ah**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (100:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 55-57 °C.

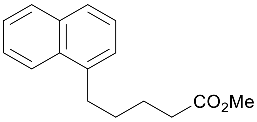
First run: 35.7 mg, 65.6% yield. **Second run**: 36.3 mg, 66.7% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.67-7.64 (m, 2 H), 7.52 (s, 1 H), 7.27 (dd, J_1 = 2.0 Hz, J_2 = 8.4 Hz, 1 H), 7.13-7.10 (m, 2 H), 3.89 (s, 3 H), 3.65 (s, 3 H), 2.74 (t, *J* = 7.2 Hz, 2 H), 2.34 (t, *J* = 7.2 Hz, 2 H), 1.74-1.67 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.0, 157.1, 137.2, 132.9, 129.0, 128.8, 127.7, 126.7, 126.2, 118.6, 105.6, 55.2, 51.4, 35.4, 33.9, 30.8, 24.5.

IR (neat, cm^{-1}): 3447, 2948, 1736, 1607, 1505, 1484, 1436, 1391, 1265, 1229, 1174, 1032, 852, 811.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{17}H_{20}NaO_3$ 295.1305, found 295.1298.



Methyl 5-(naphthalen-1-yl)pentanoate (**3ai**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

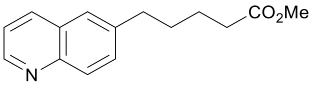
First run: 30.8 mg, 63.6% yield. **Second run**: 31.1 mg, 64.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.01 (d, *J* = 8.4 Hz, 1 H), 7.85-7.69 (m, 1 H), 7.70 (d, *J* = 8.4 Hz, 1 H), 7.52-7.44 (m, 2 H), 7.39 (t, *J* = 8.0 Hz, 1 H), 7.31 (d, *J* = 6.8 Hz, 1 H), 3.66 (s, 3 H), 3.09 (t, *J* = 7.2 Hz, 2 H), 2.36 (t, *J* = 7.2 Hz, 2 H), 1.83-1.74 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.0, 138.2, 133.9, 131.8, 128.8, 126.6, 125.9, 125.7, 125.5, 125.4, 123.7, 51.5, 33.9, 32.7, 30.2, 25.0.

IR (neat, cm^{-1}): 2948, 1736, 1509, 1435, 1167, 778.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{16}H_{18}NaO_2$ 265.1199, found 265.1194.



Methyl 5-(quinolin-6-yl)pentanoate (**3aj**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (20:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

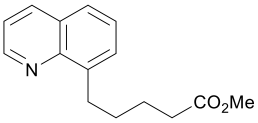
First run: 30.3 mg, 62.3% yield. **Second run**: 32.0 mg, 65.9 % yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.86 (dd, J_1 = 1.6 Hz, J_2 = 4.0 Hz, 1 H), 8.09 (d, J = 8.0 Hz, 1 H), 8.02 (d, J = 8.4 Hz, 1 H), 7.58-7.54 (m, 2 H), 7.36 (dd, J_1 = 4.4 Hz, J_2 = 8.0 Hz, 1 H), 3.66 (s, 3 H), 2.82 (t, J = 7.2 Hz, 2 H), 2.36 (t, J = 7.2 Hz, 2 H), 1.80-1.67 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.9, 149.7, 147.1, 140.5, 135.5, 130.8, 129.3, 128.3, 126.1, 121.0, 51.5, 35.5, 33.8, 30.6, 24.5.

IR (neat, cm^{-1}): 2948, 1735, 1594, 1500, 1436, 1173, 837.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{15}H_{18}NO_2$ 244.1332, found 244.1327.



Methyl 5-(quinolin-8-yl)pentanoate (**3ak**).

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (50:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

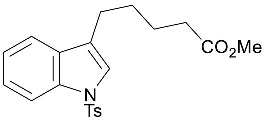
First run: 36.1 mg, 74.3%. **Second run**: 37.8 mg, 77.8%.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.92 (dd, J_1 = 1.6 Hz, J_2 = 4.4 Hz, 1H), 8.12 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1H), 7.65 (dd, J_1 = 1.2 Hz, J_2 = 8.0 Hz, 1H), 7.55 (d, J = 6.4 Hz, 1H), 7.45 (t, J = 7.2 Hz, 1H), 7.37 (dd, J_1 = 4.4 Hz, J_2 = 8.0 Hz, 1H), 3.64 (s, 3H), 3.30 (t, J = 6.8 Hz, 2H), 2.38 (t, J = 7.2 Hz, 2H), 1.88-1.73 (m, 4H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.2, 149.2, 146.8, 140.8, 136.3, 128.7, 128.4, 126.2, 126.0, 120.8, 51.4, 34.0, 31.0, 30.0, 24.9.

IR (neat, cm^{-1}): 2949, 1736, 1596, 1498, 1435, 1365, 1172, 795.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{15}H_{18}NO_2$ 244.1332, found 244.1325.



Methyl 5-(1-tosyl-*1H*-indol-3-yl)pentanoate (**3al**).

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (100:1 petroleum ether/ethyl acetate) to give the product as a yellow oil.

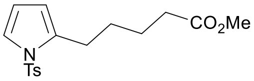
First run: 48.9 mg, 63.5% yield. **Second run**: 49.5 mg, 64.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.97 (d, *J* = 8.4 Hz, 1 H), 7.73 (d, *J* = 8.4 Hz, 2 H), 7.45 (d, *J* = 7.6 Hz, 1 H), 7.32-7.28 (m, 2 H), 7.23-7.18 (m, 3 H), 3.67 (s, 3 H), 2.67 (t, *J* = 6.8 Hz, 2 H), 2.36-2.31 (m, 5 H), 1.76-1.66 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.9, 144.6, 135.34, 135.30, 131.0, 129.7, 126.7, 124.6, 122.93, 122.87, 122.7, 119.4, 113.7, 51.5, 33.7, 28.3, 24.6, 24.5, 21.5.

IR (neat, cm^{-1}): 2949, 1735, 1597, 1447, 1367, 1173, 1121, 974, 813, 747, 703, 670.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{24}NO_4S$ 386.1421, found 386.1413.



Methyl 5-(1-tosyl-*1H*-pyrrol-2-yl)pentanoate (**3am**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (100:1 petroleum ether/ethyl acetate) to give the product as a brown oil.

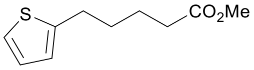
First run: 28.7 mg, 44.7% yield. **Second run**: 30.6 mg, 45.7% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.63 (d, *J* = 8.4 Hz, 2 H), 7.29-7.27 (m, 3 H), 6.19 (t, *J* = 3.2 Hz, 1 H), 5.99 (dd, J_1 = 1.6 Hz, J_2 = 3.2 Hz, 1 H), 3.66 (s, 3 H), 2.66 (t, *J* = 7.2 Hz, 2 H), 2.40 (s, 3 H), 2.29 (t, *J* = 7.2 Hz, 2 H), 1.69-1.55 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.9, 144.7, 136.5, 135.2, 129.9, 126.7, 122.3, 112.0, 111.3, 51.5, 33.7, 28.2, 26.8, 24.5, 21.6.

IR (neat, cm^{-1}): 2951, 1735, 1363, 1174, 1090, 813, 670.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{17}H_{21}NNaO_4S$ 358.1083, found 358.1076.



Methyl 5-(thiophen-2-yl)pentanoate (**3an**)

The title compound was prepared according to the General Procedure. The reaction was

conducted at 45 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

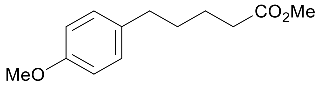
First run: 26.3 mg, 66.4% yield. **Second run**: 24.6 mg, 62.1% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.10 (dd, J_1 = 1.2 Hz, J_2 = 4.8 Hz, 1 H), 6.90 (dd, J_1 = 3.2 Hz, J_2 = 5.2 Hz, 1 H), 6.78-6.77 (m, 1 H), 3.66 (s, 3 H), 2.86-2.83 (m, 2 H), 2.36-2.32 (m, 2 H), 1.75-1.68 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 173.9, 144.9, 126.6, 124.1, 122.9, 51.5, 33.7, 31.1, 29.5, 24.3.

IR (neat, cm^{-1}): 2948, 1738, 1436, 1167, 695.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{10}H_{15}O_2S$ 199.0787, found 199.0783.



Methyl 5-(4-methoxyphenyl)pentanoate (**3ao**)

To a mixture of NiBr$_2$(diglyme) (7.1 mg, 10 mol%), dppb (17.1 mg, 20 mol%), Mn (44 mg, 4.0 equiv.), MgBr$_2$ (55.2 mg, 150 mol%), and oxalate **1o** (0.2 mmol) was added a solution of methyl 4-bromobutyrate (52 μL, 2.0 equiv.) and 3-trifluoromethylpyridine (1.5 μL, 5 mol%) in DMSO/CH$_3$CN (4:1, 0.5 mL, 0.4 M). The reaction mixture was quickly frozen by submersion in a liquid nitrogen bath, vacuumed and filled with argon for three times. The resulting solution was stirred at 45 °C for 36 h and then quenched with water (20 mL), extracted with ethyl acetate (3 × 10 mL). The combined organic layers were washed with brine (20 mL), dried over anhydrous Na$_2$SO$_4$, filtered, and evaporated to dryness. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a colorless oil.

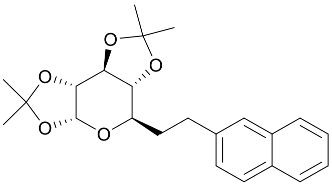
First run: 27.7 mg, 62.3% yield. **Second run**: 28.5 mg, 64.1% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.08 (d, J = 8.4 Hz, 2 H), 6.82 (d, J = 8.4 Hz, 2 H), 3.78 (s, 3 H), 3.66 (s, 3 H), 2.57 (t, J = 7.2 Hz, 2 H), 2.32 (t, J = 7.2 Hz, 2 H), 1.70-1.58 (m, 4 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.0, 157.7, 134.1, 129.2, 113.7, 55.2, 51.4, 34.6, 33.9, 31.1, 24.5.

IR (neat, cm^{-1}): 2932, 1737, 1612, 1513, 1437, 1246, 1176, 1035, 819.

HRMS (ESI): [M+Na]$^+$ calcd. for $C_{13}H_{18}NaO_3$ 245.1148, found 245.1140.



(5*R*,5a*R*,8a*S*,8b*R*)-2,2,7,7-Tetramethyl-5-(2-(naphthalen-2-yl)ethyl)tetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran (**18**)

The title compound was prepared according to the General Procedure. Alkyl bromide (0.2 mmol, 1.0 *equiv.*) and oxalate (0.4 mmol, 2.0 *equiv.*) were used. The reaction was conducted at 45 ℃ for 24 h. The crude residue was purified by flash chromatography (40:1 petroleum ether/ethyl acetate) to give the product as a colorless viscous oil.

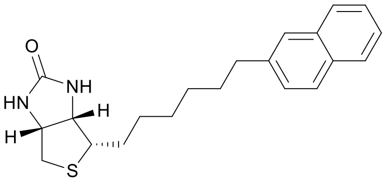
First run: 39.3 mg, 51.2% yield. **Second run**: 44.1 mg, 57.4% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.75 (m, 3 H), 7.64 (s, 1 H), 7.47-7.40 (m, 2 H), 7.35 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 5.97 (d, J = 3.6 Hz, 1 H), 4.56 (d, J = 3.6 Hz, 1 H), 4.25-4.21 (m, 2 H), 3.54-3.49 (m, 1 H), 3.01-2.94 (m, 1 H), 2.86-2.79 (m, 1 H), 2.17-2.11 (m, 1 H), 2.01-1.94 (m, 1 H), 1.49 (s, 3 H), 1.39 (s, 3 H), 1.33 (s, 3 H), 1.32 (s, 3 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.0, 133.5, 132.0, 127.9, 127.6, 127.4, 127.3, 126.5, 125.8, 125.1, 112.0, 106.3, 100.8, 83.9, 83.6, 74.9, 71.0, 35.2, 31.5, 27.1, 26.5, 24.1, 24.0.

IR (neat, cm^{-1}): 2922, 2850, 1699, 1653, 1558, 1541, 1507, 1220, 1076, 1023, 855.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{23}H_{29}O_5$ 385.2010, found 385.2004.



(3a*S*,4*S*,6a*R*)-4-(6-(Naphthalen-2-yl)hexyl)tetrahydro-1*H*-thieno[3,4-*d*]imidazol-2(3*H*)-one (**20**)

The title compound was prepared according to the General Procedure. The reaction was conducted at 45 ℃ for 24 h. The crude residue was purified by flash chromatography (40:1 CH_2Cl_2/MeOH) to give the product as a white solid. Mp. 131-133 ^{o}C.

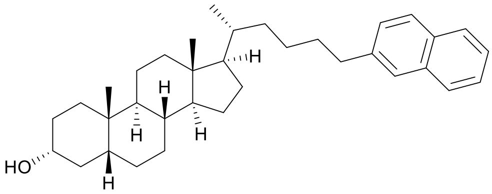
First run: 46.1 mg, 65.1% yield. **Second run**: 42.8 mg, 60.5% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.78-7.75 (m, 3 H), 7.60 (s, 1 H), 7.46-7.38 (m, 2 H), 7.32 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 5.88-5.86 (m, 2 H), 4.43-4.40 (m, 1 H), 4.23-4.20 (m, 1 H), 3.12-3.07 (m, 1 H), 2.85 (dd, J_1 = 12.8 Hz, J_2 = 5.2 Hz, 1 H), 2.77-2.67 (m, 3 H), 1.68-1.63 (m, 4 H), 1.36 (m, 6 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 163.8, 140.2, 133.5, 131.8, 127.7, 127.5, 127.4, 127.3, 126.2, 125.8, 125.0, 61.9, 60.0, 55.6, 40.5, 35.9, 31.2, 29.3, 29.0, 28.9, 28.5.

IR (neat, cm^{-1}): 3213, 2924, 2852, 1702, 1507, 1269, 950.

HRMS (ESI): [M+H]$^+$ calcd. for $C_{21}H_{27}N_2OS$ 355.1839, found 355.1839.



(3*R*,5*R*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-10,13-Dimethyl-17-((*R*)-6-(naphthalen-2-yl)hexan-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-ol (22)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (40:1 petroleum ether/ethyl acetate) to give the product as a colorless solid. Mp. 34-36 ^{o}C.

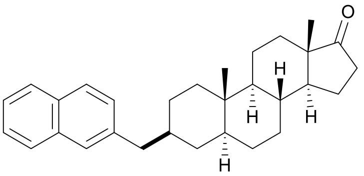
First run: 56.7 mg, 58.3% yield. **Second run**: 54.5 mg, 56.1% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.75 (m, 3 H), 7.61 (s, 1 H), 7.46-7.39 (m, 2 H), 7.33 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 3.61-3.60 (m, 1 H), 2.79-2.73 (m, 2 H), 1.97-1.94 (m, 1 H), 1.84-1.62 (m, 8 H), 1.52-1.30 (m, 10 H), 1.25-1.20 (m, 5 H), 1.15-0.98 (m, 7 H), 0.91-0.88 (m, 6 H), 0.62 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 133.5, 131.8, 127.7, 127.5, 127.4, 127.3, 126.2, 125.7, 124.9, 71.8, 56.4, 56.2, 42.6, 42.0, 40.3, 40.1, 36.3, 36.2, 35.8, 35.7, 35.3, 34.5, 31.9, 30.5, 28.3, 27.1, 26.4, 25.8, 24.2, 23.3, 20.8, 18.6, 12.0.

IR (neat, cm^{-1}): 3334, 2932, 2862, 1457, 1037, 908, 734.

HRMS (ESI): [M+Na]$^{+}$ calcd. for $C_{35}H_{50}NaO$ 509.3754, found 509.3748.



(*3S,5S,8R,9S,10S,13S,14S*)-10,13-dimethyl-3-(naphthalen-2-ylmethyl)hexadecahydro-*17H*-cyclopenta[*a*]phenanthren-17-one (24)

The title compound was prepared according to the General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (150:1 petroleum ether/ethyl acetate) to give the product as a white solid. Mp. 123-125 ^{o}C.

First run: 43.9 mg, 53.0% yield. **Second run**: 43.8 mg, 52.8% yield.

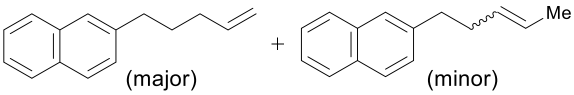
^{1}H NMR (400 MHz, $CDCl_3$, dr = 6.7:1) δ 7.80-7.74 (m, 3 H), 7.56 (s, 1 H), 7.46-7.38 (m, 2 H), 7.29 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 2.90-2.79 (m, 0.26 H, minor), 2.70-2.60 (m, 1.74 H, major), 2.48-2.37 (m, 1 H), 2.10-1.99 (m, 1 H), 1.96-1.85 (m, 1 H), 1.83-1.41 (m, 9 H), 1.34-1.15 (m, 6 H), 1.09-1.02 (m, 2 H), 0.96-0.83 (m, 5 H), 0.79 (s, 3 H), 0.70-0.64 (m, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$, dr = 6.7:1) δ 221.4, 138.8, 133.5, 131.9, 127.9, 127.6, 127.5, 127.4, 127.1, 125.8, 125.0, 54.9, 54.7, 51.6, 51.5, 47.8, 47.8, 46.6, 44.2, 40.6, 40.1, 38.5, 36.7, 36.2, 35.8, 35.3, 35.1, 34.9, 33.2, 32.5, 31.6, 31.6, 30.9, 28.8, 28.6, 21.7, 20.3, 20.1, 13.8, 12.3, 11.8.

IR (neat, cm^{-1}): 2918, 2852, 1739, 1452, 814, 747.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{30}H_{39}O$ 415.2995, found 415.2989.

5.3. Characterization Data of Mechanistic Investigation



The mixture of alkenes (28)

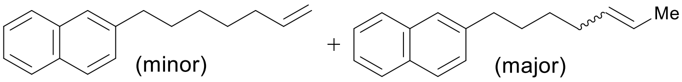
The title compounds were prepared according to the above General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give a mixture of alkenes (**28**, major/minor is 1.5:1) as a colorless oil. 19.7 mg, 50.3% yield.

^{1}H NMR (400 MHz, CDCl$_3$, mixture) δ 7.80-7.75 (m, 3 H), 7.62-7.60 (m, 1 H), 7.46-7.38 (m, 2 H), 7.36-7.31 (m, 1 H), 5.90-5.80 (m, 0.58 H, major CH), 5.55-5.43 (m, 0.77 H, minor CH=CH), 5.06-4.97 (m, 1.19 H, major CH_2), 2.84-2.76 (m, 2 H), 2.48-2.35 (m, 0.8 H, minor CH_2), 2.15-2.10 (m, 1.22 H, major CH_2), 1.85-1.77 (m, 1.24 H, minor CH_3), 1.65-1.54 (m, 1.21 H, major CH_2).

^{13}C NMR (100 MHz, CDCl$_3$, mixture) δ 140.0, 139.7, 138.6, 133.6, 132.0, 130.5, 129.6, 127.8, 127.8, 127.7, 127.6, 127.4, 127.4, 126.4, 126.4, 125.8, 125.5, 125.0, 124.6, 114.8, 36.2, 35.9, 35.4, 34.3, 33.3, 30.5, 28.6, 17.9, 12.8.

IR (neat, cm^{-1}): 3052, 2929, 2855, 1638, 1600, 1508, 1437, 963, 910, 854, 815, 744.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{15}H_{17}$ 197.1325, found 197.1321.



The mixture of alkenes (30)

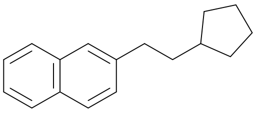
The title compounds were prepared according to the above General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give a mixture of alkenes (**30**, major/minor > 20:1) as a colorless oil. 27.3 mg, 60.9% yield.

^{1}H NMR (400 MHz, CDCl$_3$, major, the mixture of Z/E isomers) δ 7.79-7.74 (m, 3 H), 7.59 (s, 1 H), 7.45-7.37 (m, 2 H), 7.31 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1 H), 5.51-5.34 (m, 2 H), 2.79-2.74 (m, 2 H), 2.13-1.98 (m, 2 H), 1.80-1.59 (m, 5 H), 1.47-1.38 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$, major, the mixture of Z/E isomers) δ 140.29, 140.25, 133.6, 131.9, 131.3, 130.5, 127.7, 127.6, 127.41, 127.38, 126.3, 125.8, 125.0, 124.9, 123.9, 36.0, 35.97, 32.5, 30.9, 30.8, 29.24, 29.18, 26.7, 17.9, 12.8.

IR (neat, cm^{-1}): 2929, 2854, 1508, 1456, 965, 852, 815, 745.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{17}H_{21}$ 225.1638, found 225.1632.



2-(Cyclopentylmethyl)naphthalene (**31**)

The title compound was prepared according to the above General Procedure. The reaction was conducted at 30 ℃ for 24 h. The crude residue was purified by flash chromatography (petroleum ether) to give the product as a colorless oil. 5.9 mg, 13.2% yield.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.80-7.74 (m, 3 H), 7.61 (s, 1 H), 7.45-7.38 (m, 2 H), 7.33 (d, *J* = 7.6 Hz, 1 H), 2.78 (t, *J* = 8.0 Hz, 2 H), 1.81 (m, 3 H), 1.74-1.68 (m, 2 H), 1.62 (m, 2 H), 1.55-1.50 (m, 2 H), 1.16 (m, 2 H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 140.6, 133.6, 131.9, 127.7, 127.6, 127.4, 127.4, 126.2, 125.8, 124.9, 39.7, 38.0, 35.3, 32.7, 25.2.

IR (neat, cm^{-1}): 2947, 2857, 1508, 1453, 852, 814, 744.

HRMS (ESI): $[M+H]^+$ calcd. for $C_{17}H_{21}$ 225.1638, found 225.1631.

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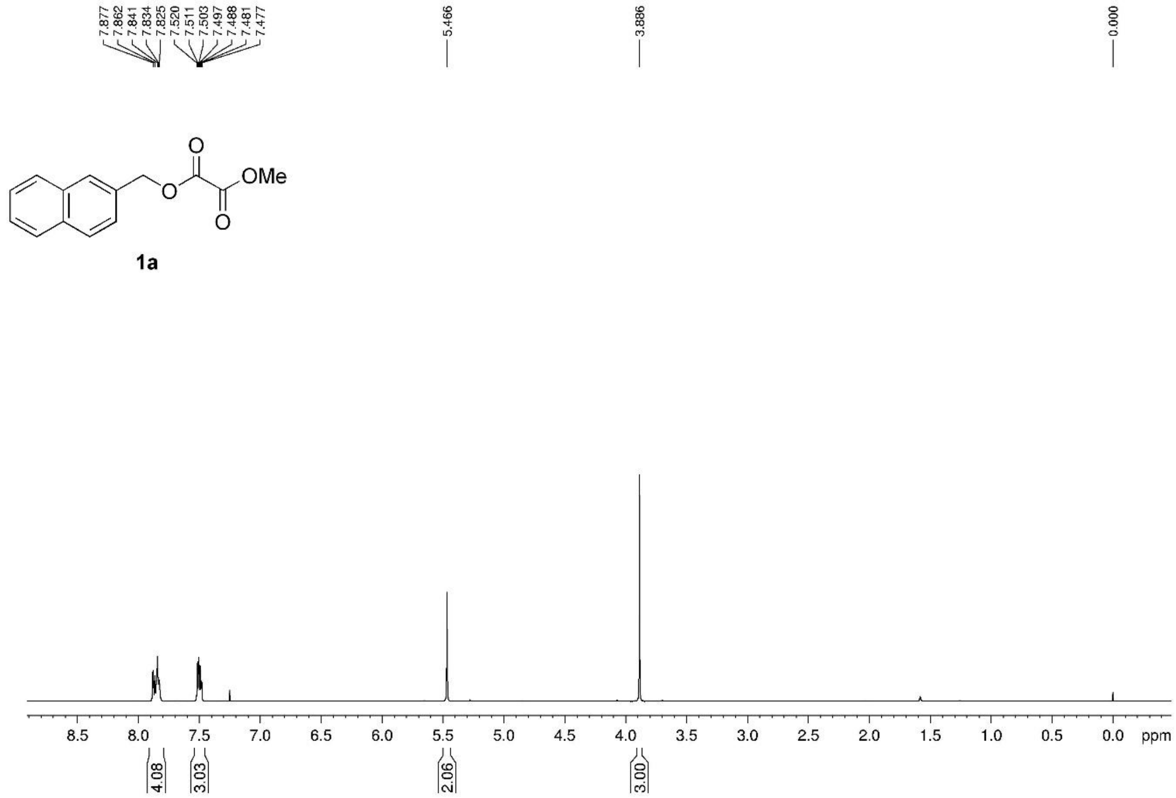
7. Copies of NMR Spectra for Compounds



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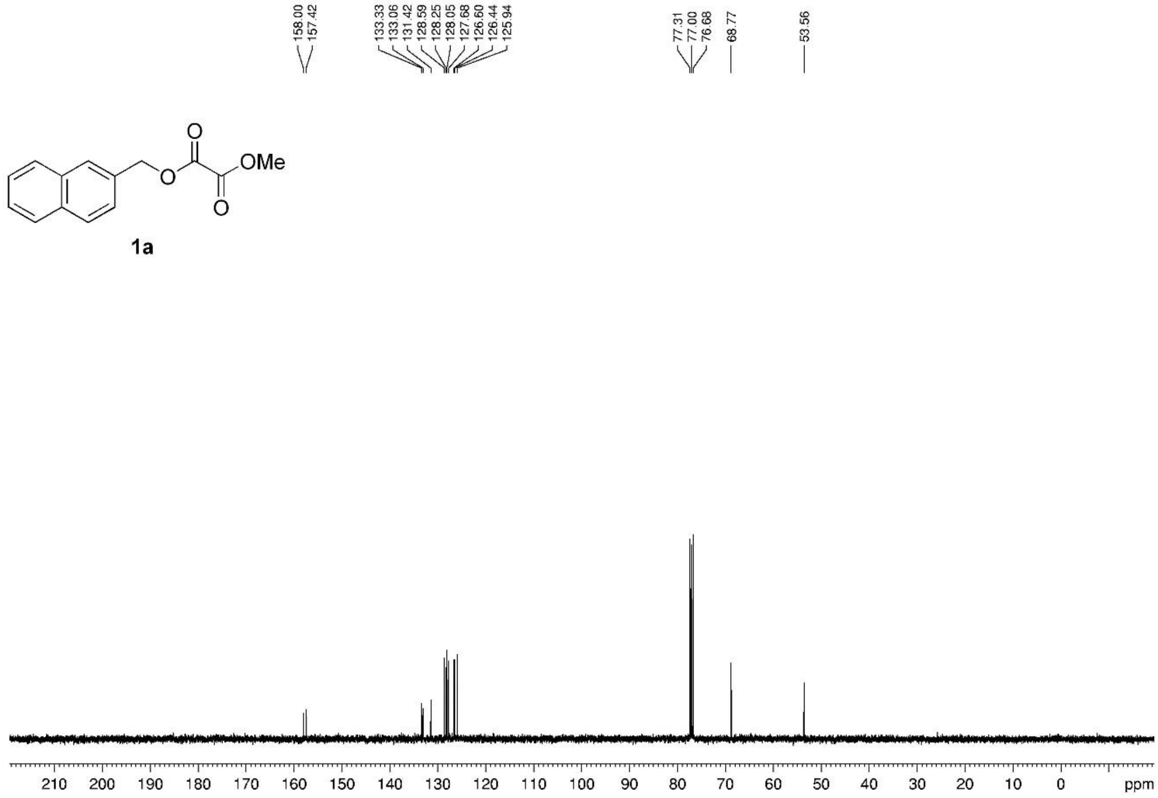

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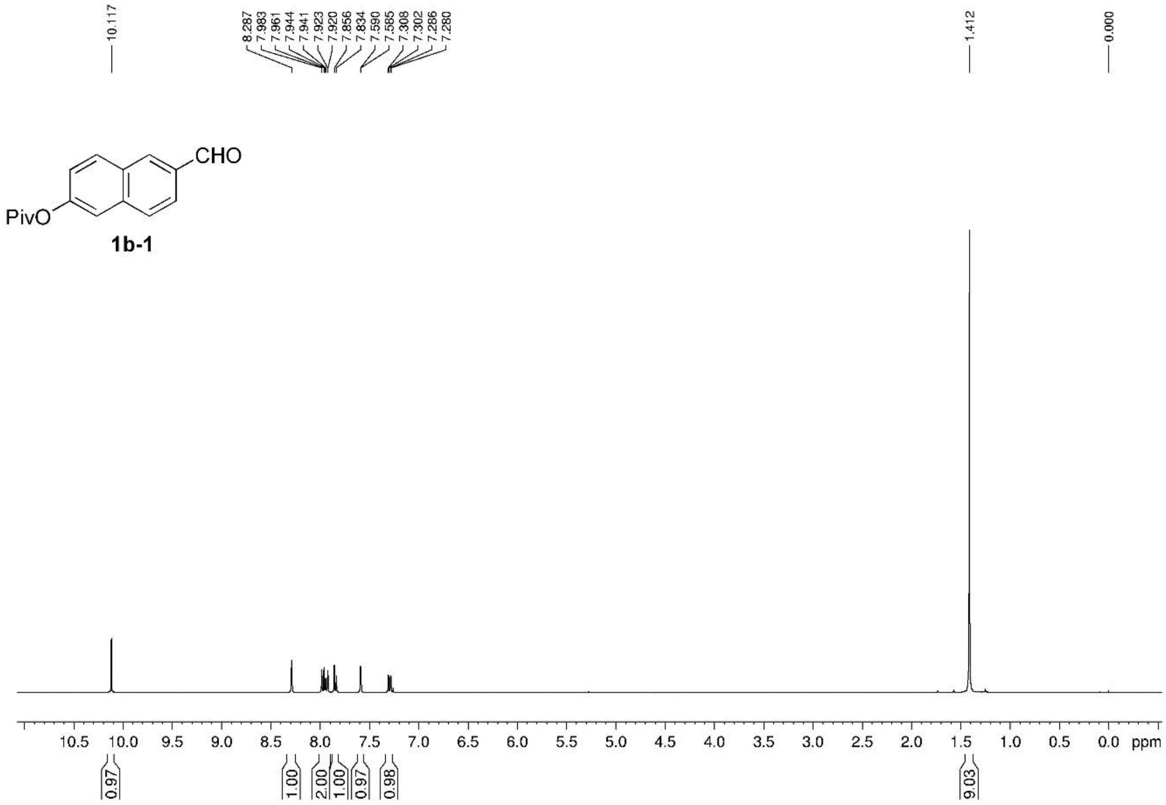
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7.302
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7.280
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PivO
1b-1
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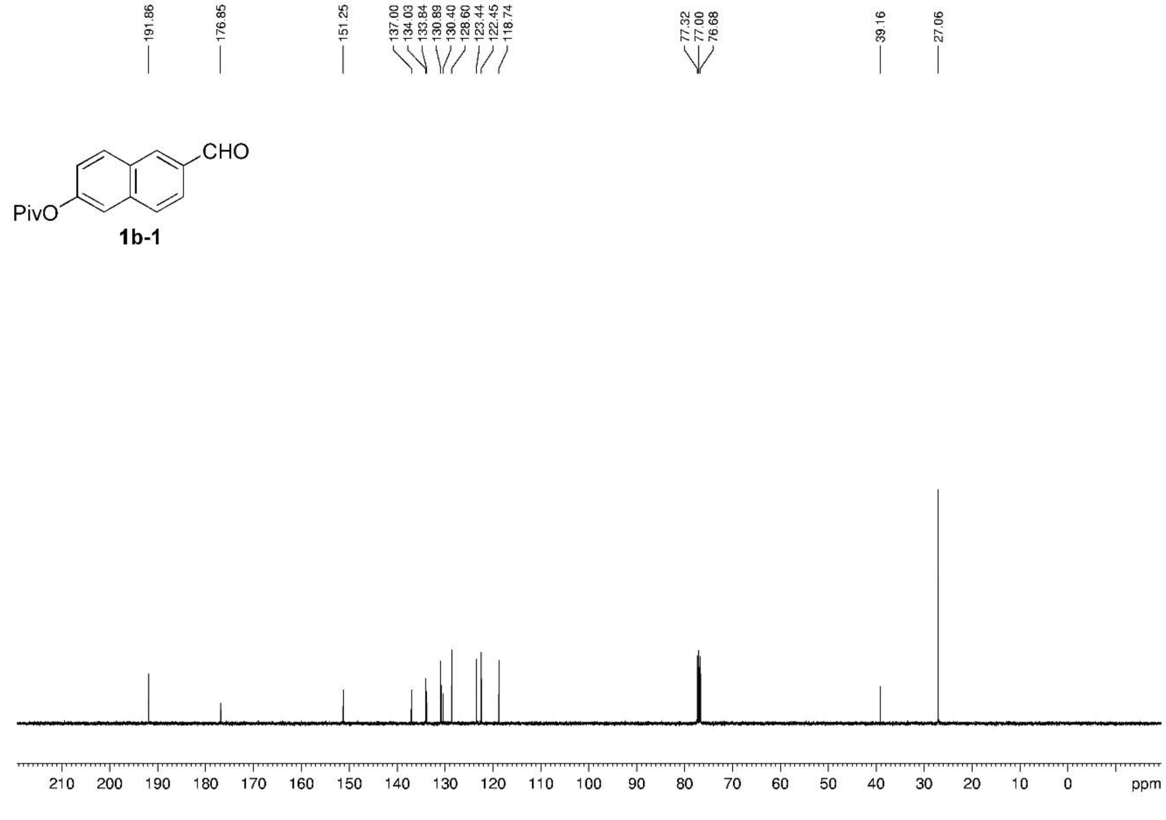
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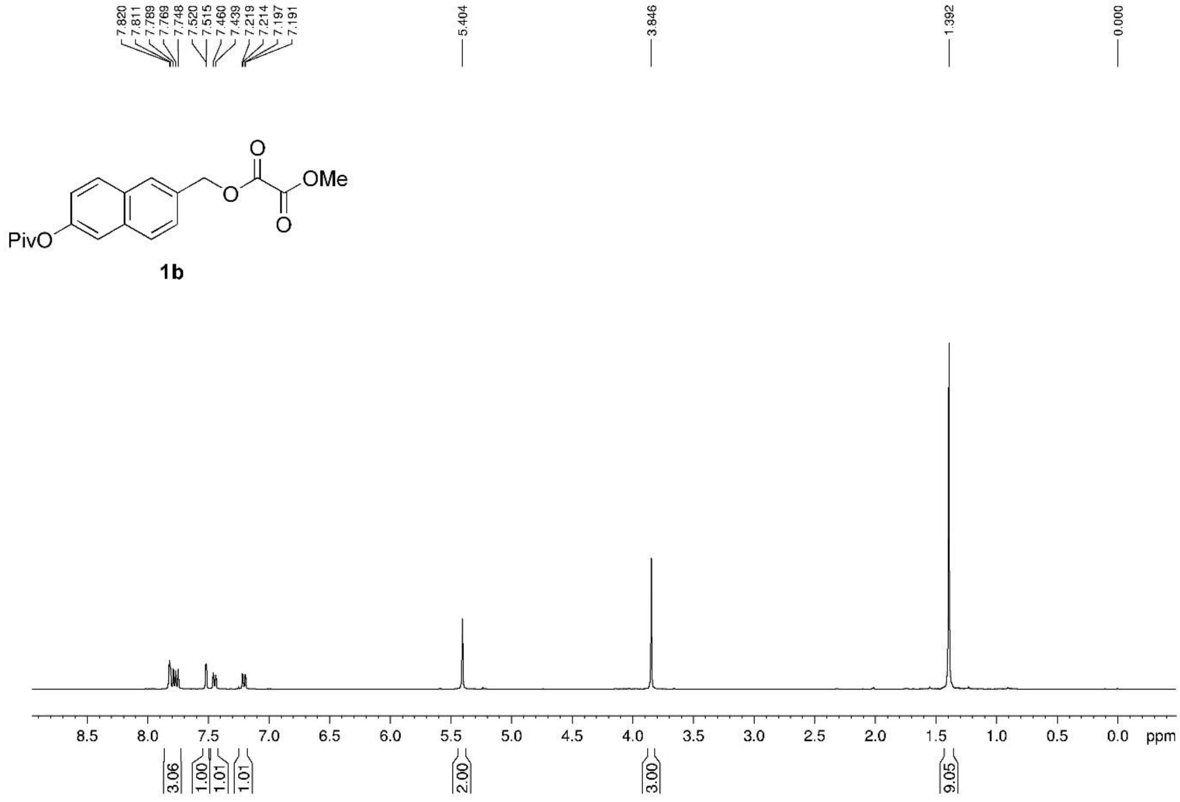

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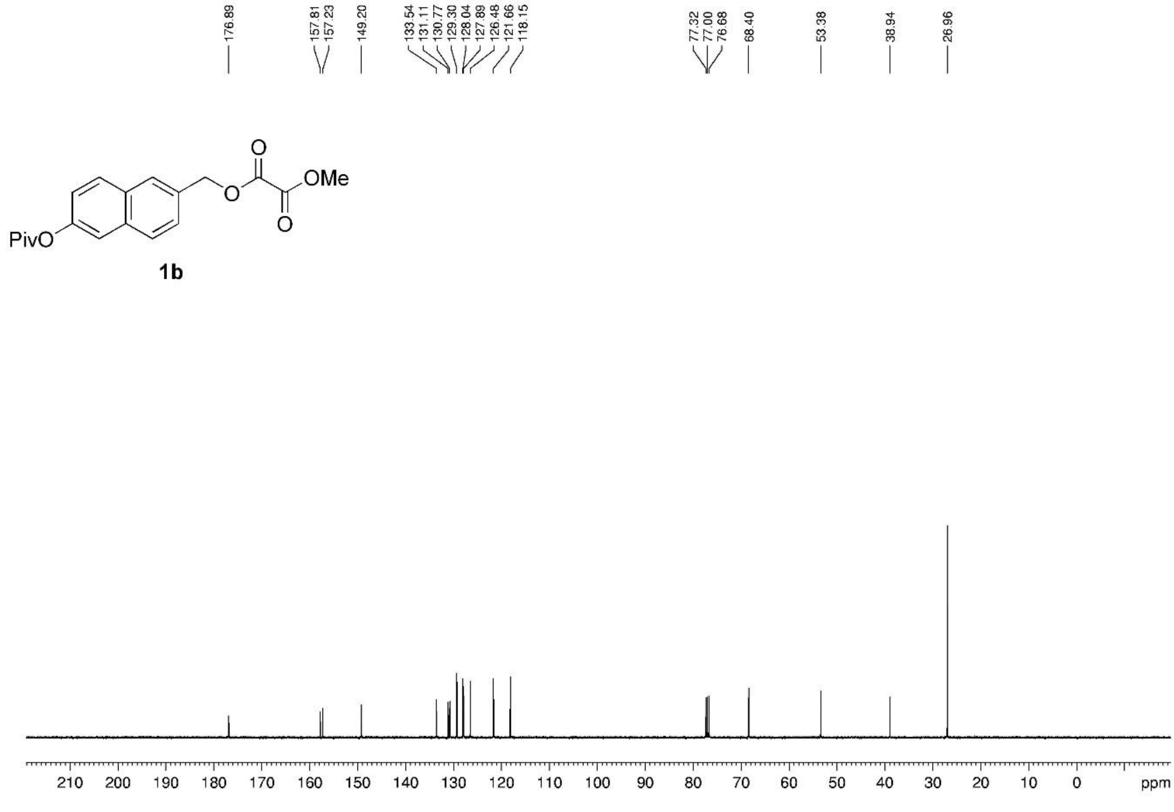

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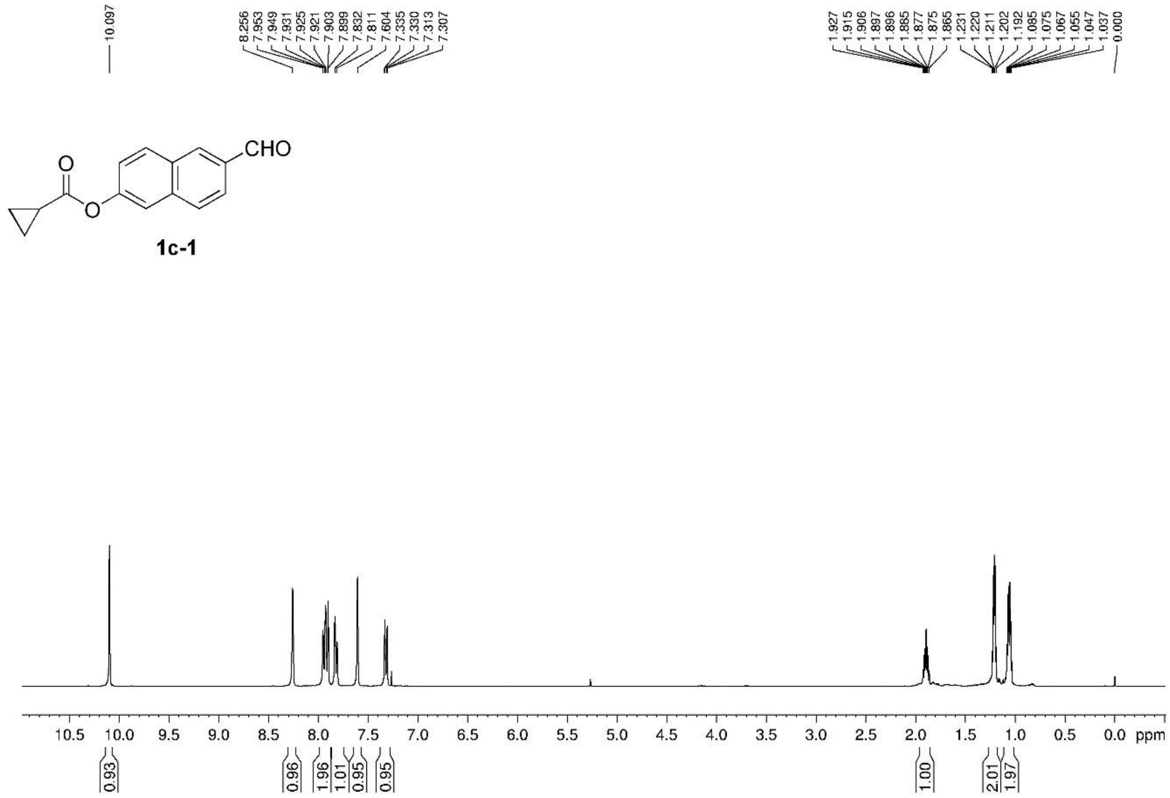
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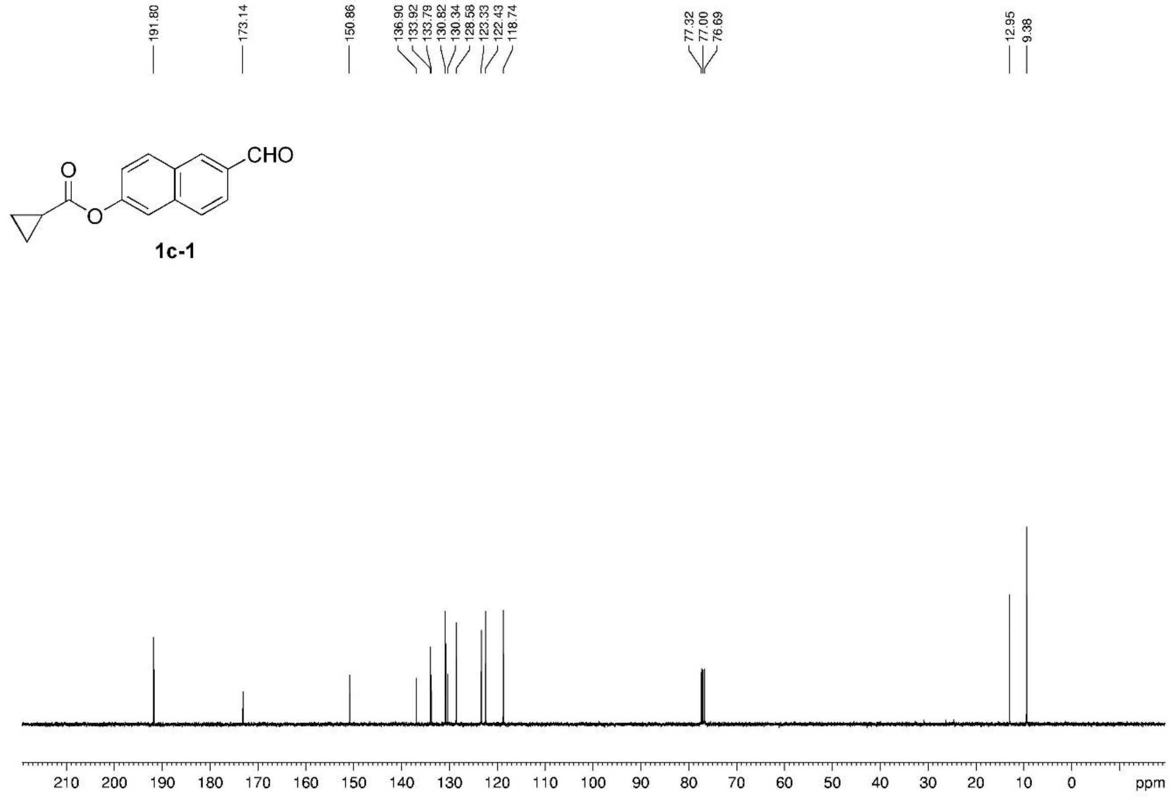
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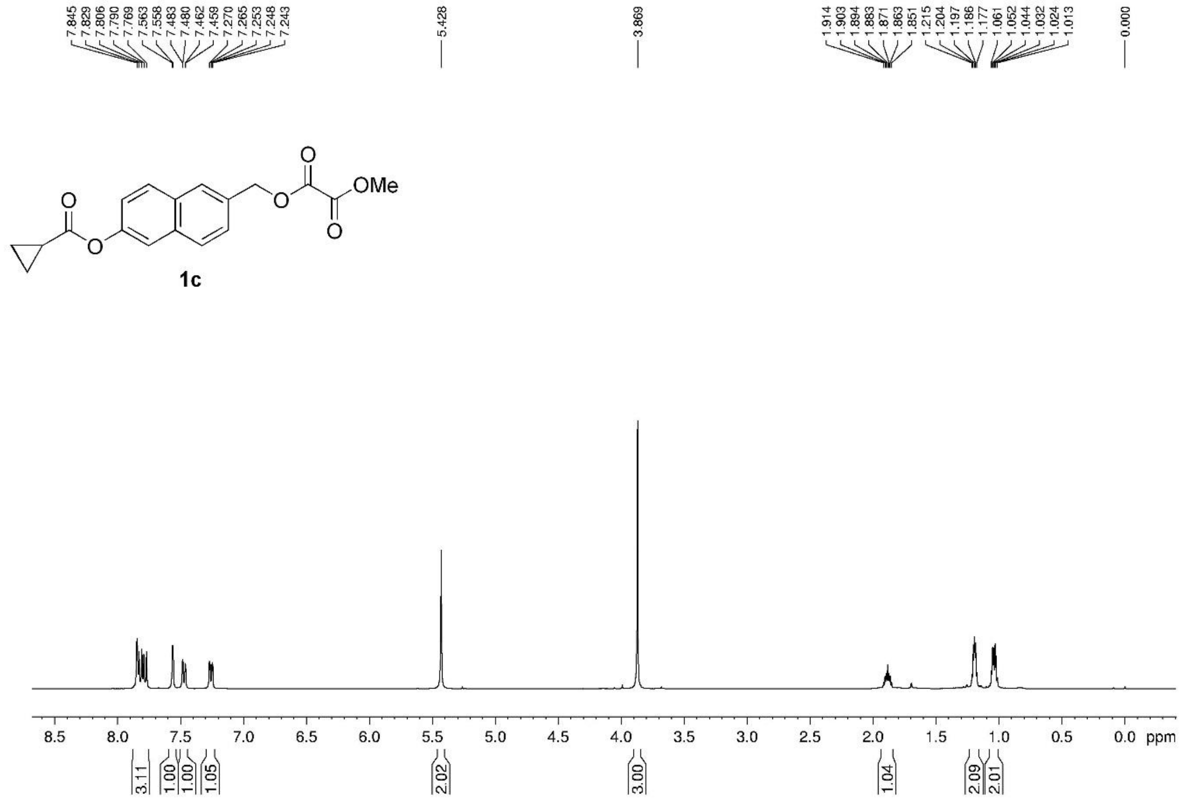
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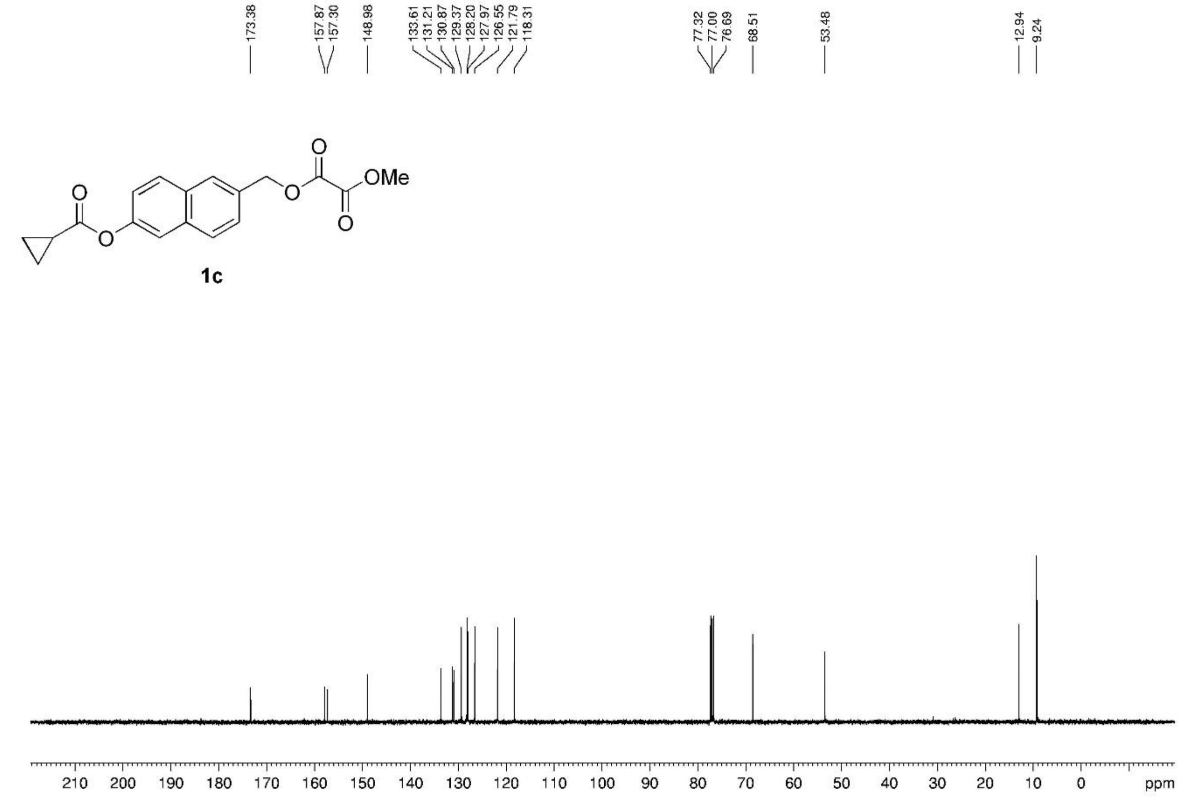
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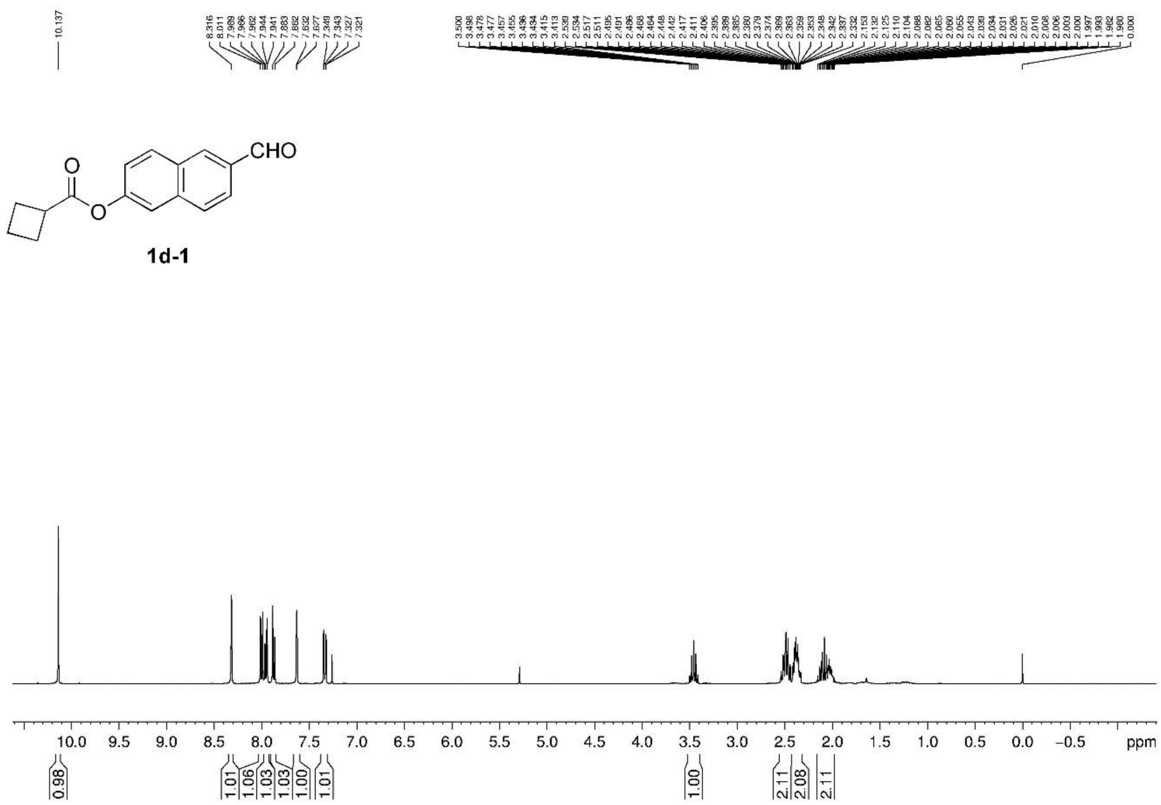
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
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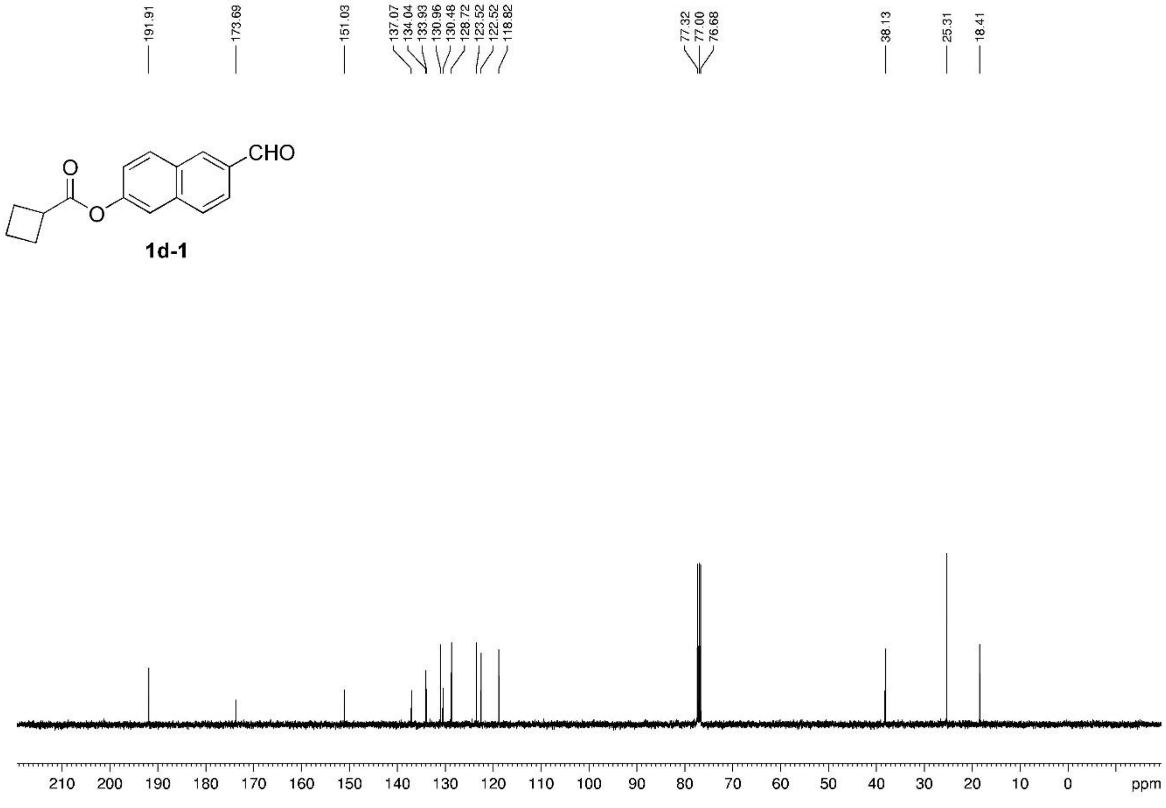
1.03
1.03
1.00
1.01
1.00
2.11
2.08
2.11

1d-1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122.52
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77.32
77.00
76.68
38.13
25.31
18.41
O
O
CHO
210
200
190
180
170
160
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130
120
110
100
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80
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40
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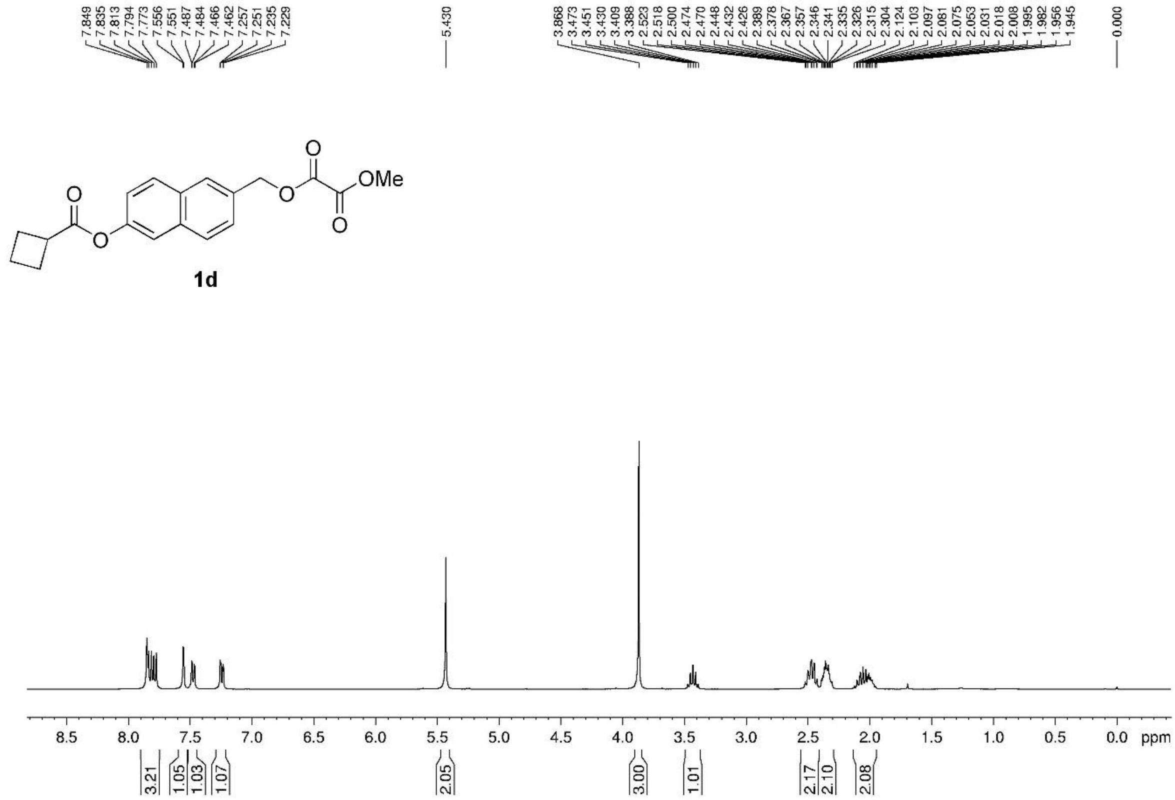
0
ppm

1d-1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7.251
7.235
7.229
5.430
3.868
3.473
3.451
3.430
3.409
3.388
2.523
2.518
2.500
2.474
2.470
2.448
2.432
2.426
2.389
2.378
2.367
2.357
2.346
2.341
2.335
2.326
2.315
2.304
2.124
2.103
2.097
2.081
2.075
2.053
2.031
2.018
2.008
1.995
1.982
1.956
1.945
0.000
O
O
O
OMe
O
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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ppm
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1.03
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1.01
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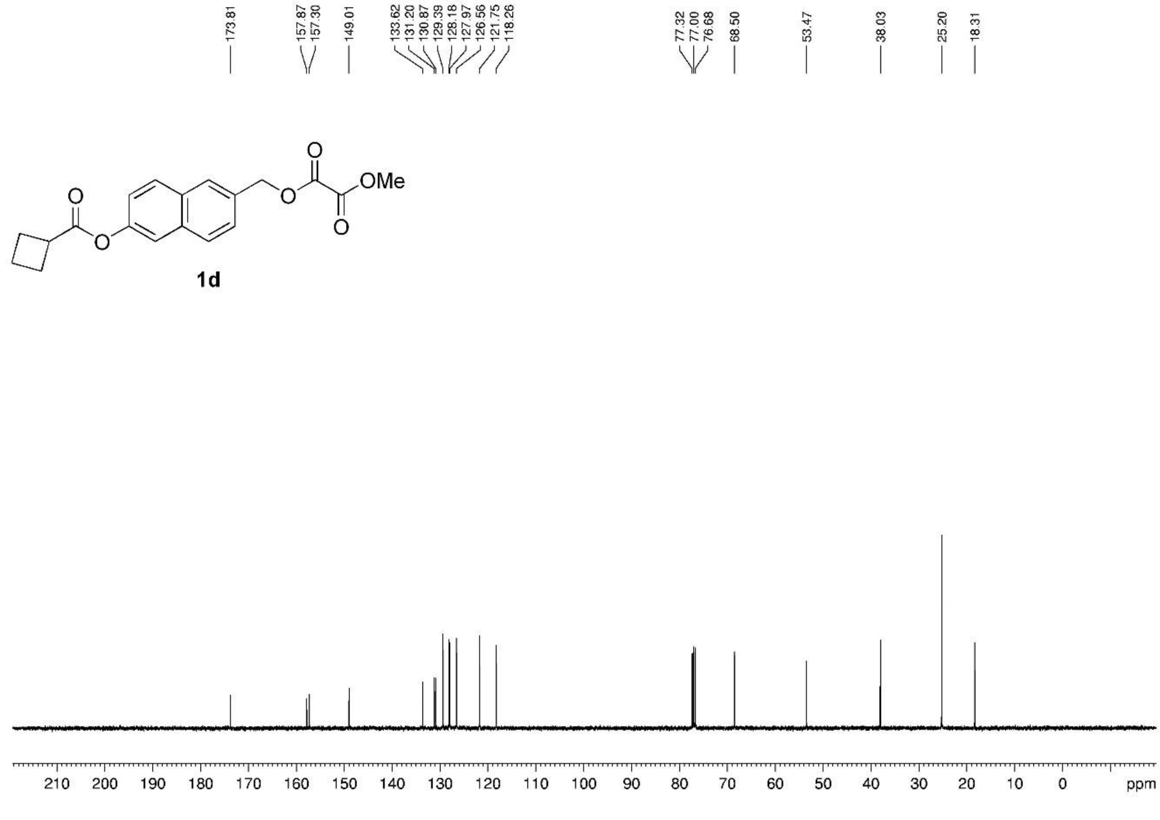
2.10
2.08

1d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118.26
77.32
77.00
76.68
68.50
53.47
38.03
25.20
18.31
O
O
O
OMe
O
O
210
200
190
180
170
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130
120
110
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90
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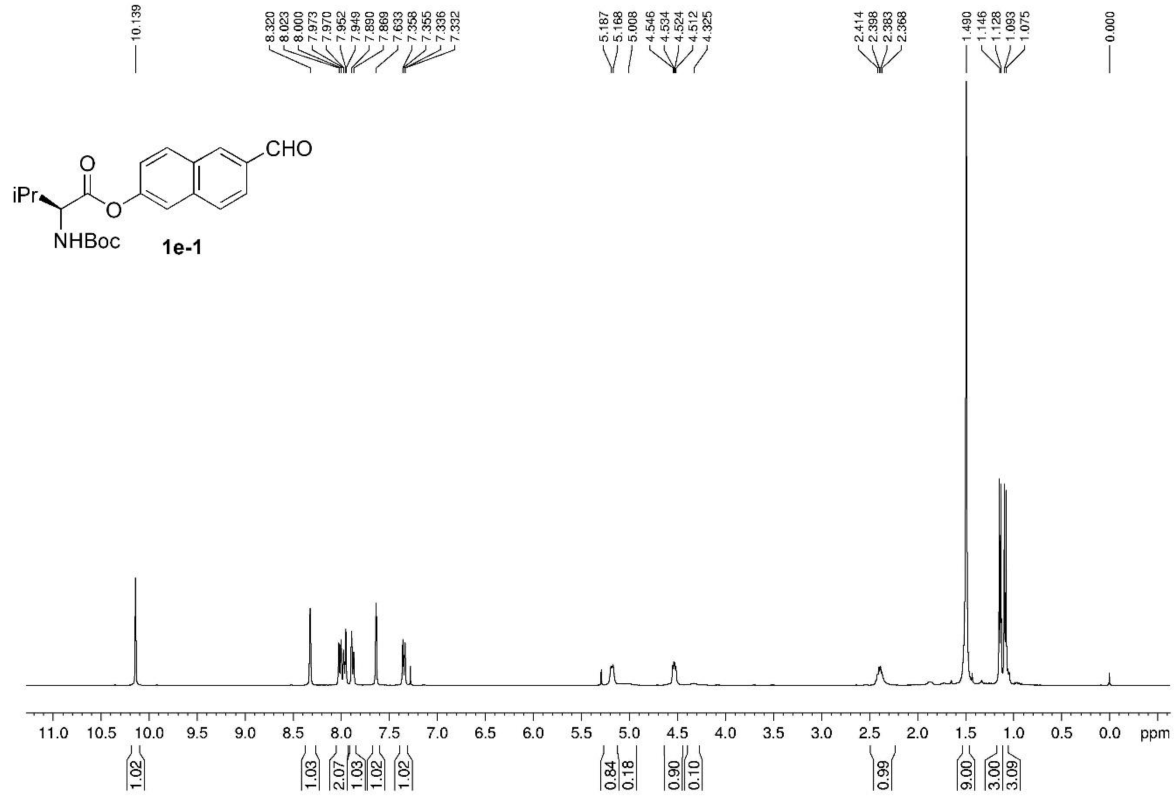
10
0
ppm

1d



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7.358
7.355
7.336
7.332
5.187
5.168
5.008
4.546
4.534
4.524
4.512
4.325
2.414
2.398
2.383
2.368
1.490
1.146
1.128
1.093
1.075
0.000
CHO
O
iPr
O
NHBoc
1e-1
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.02
1.03
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1.02
1.02
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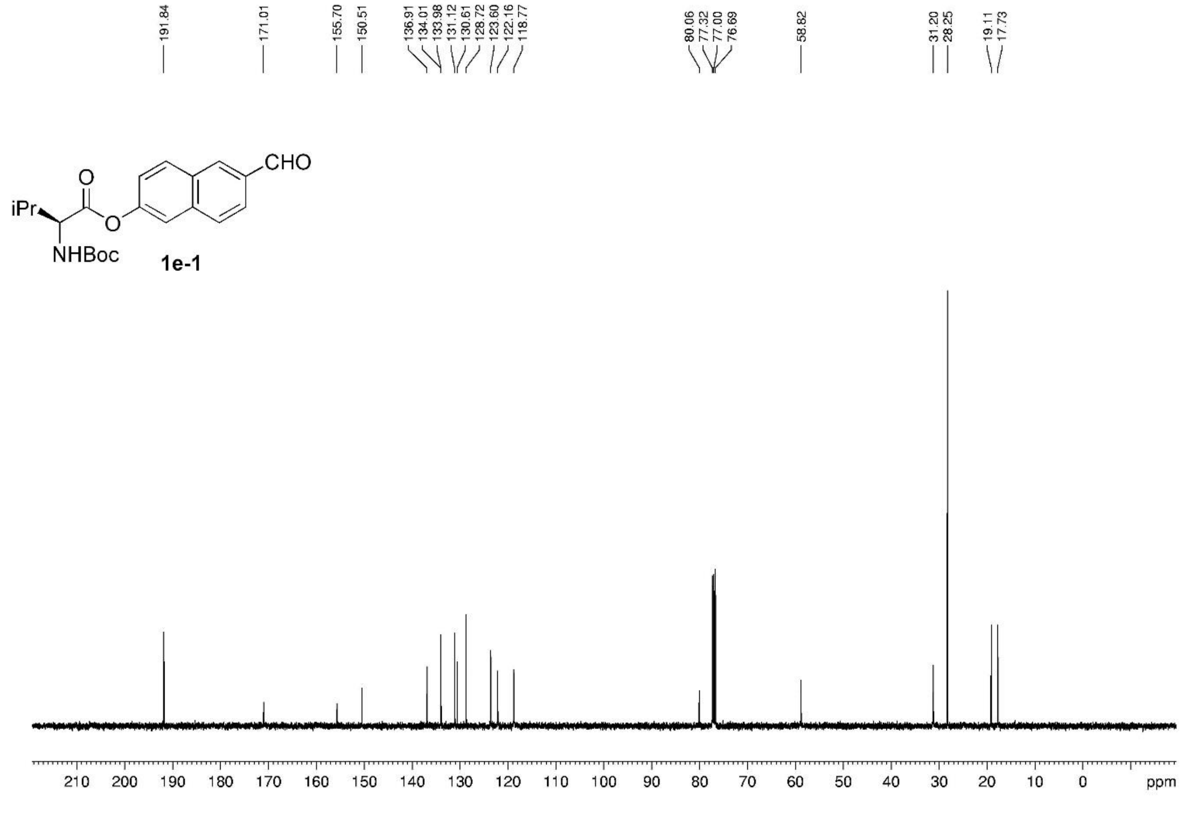

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128.72
123.60
122.16
118.77
80.06
77.32
77.00
76.69
58.82
31.20
28.25
19.11
17.73
CHO
O
iPr
O
NHBoc
1e-1
210
200
190
180
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140
130
120
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100
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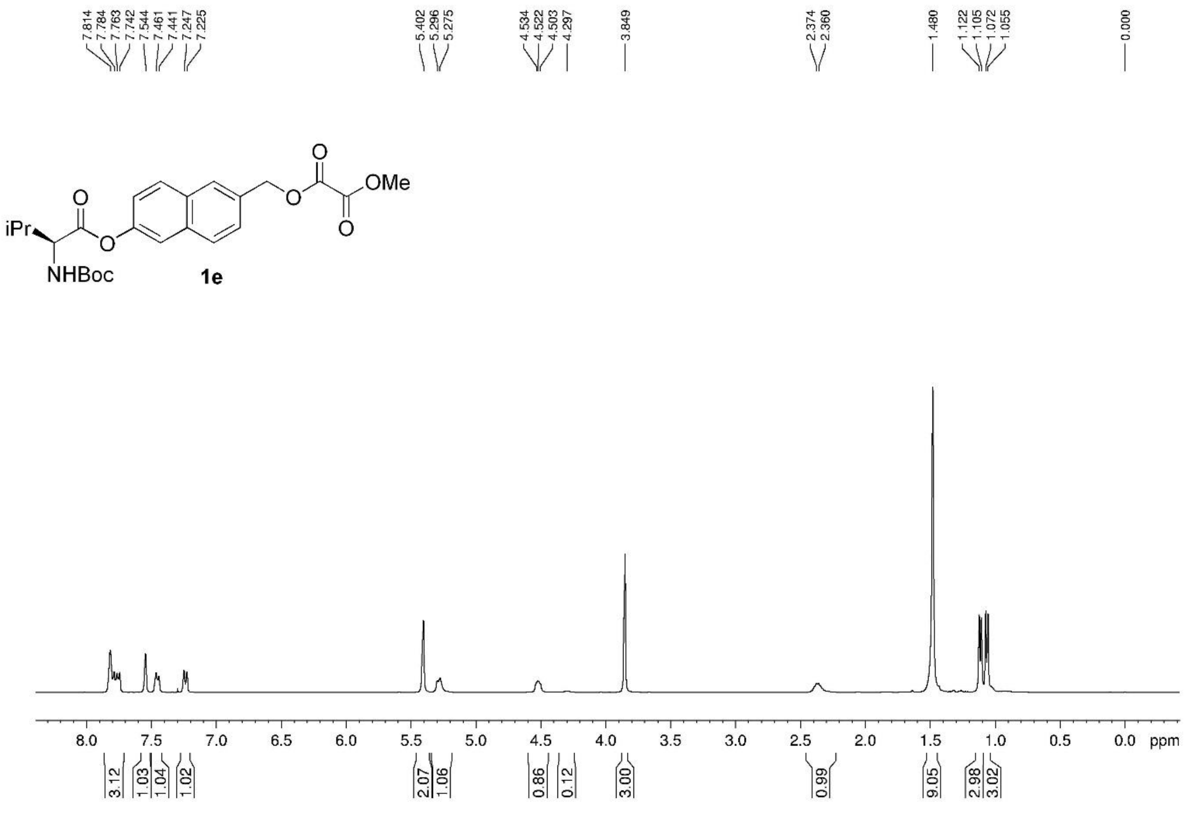
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7.247
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5.275
4.534
4.522
4.503
4.297
3.849
2.374
2.360
1.480
1.122
1.105
1.072
1.055
0.000
iPr
O
NHBoc
OMe
1e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.12
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1.04
1.02
2.07
1.06
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3.00
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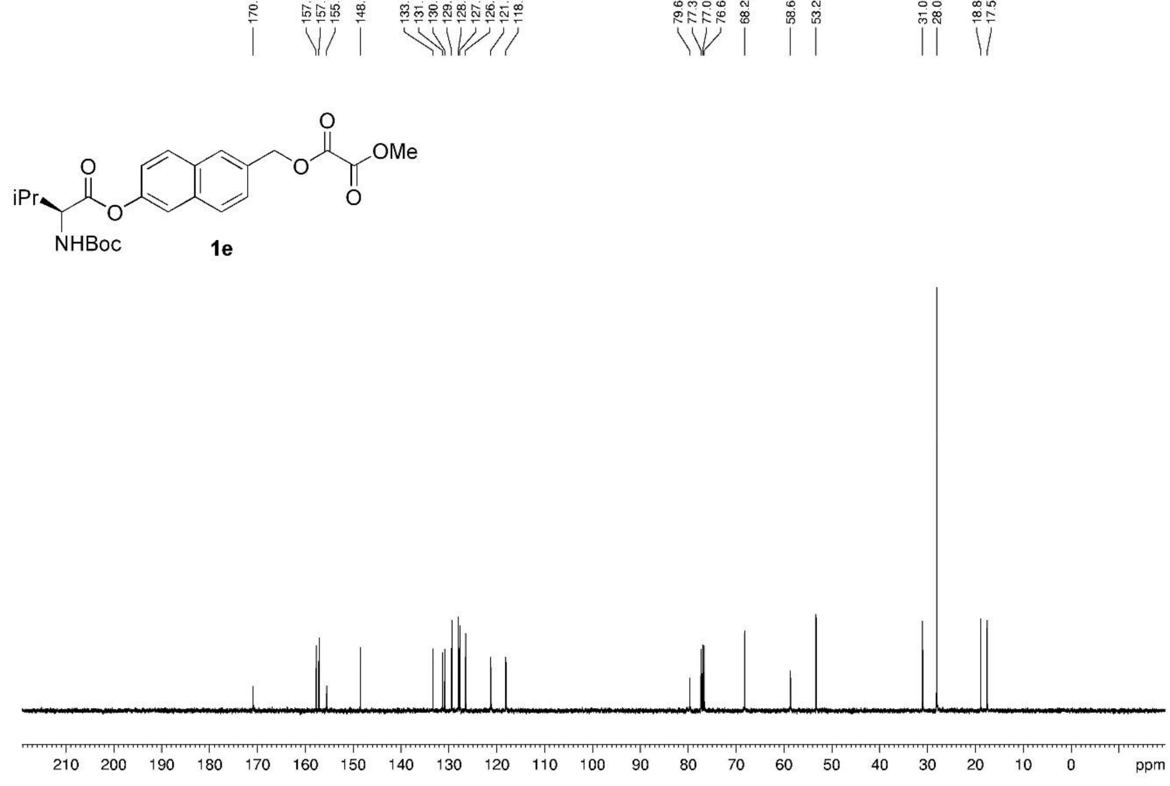
9.05
2.98
3.02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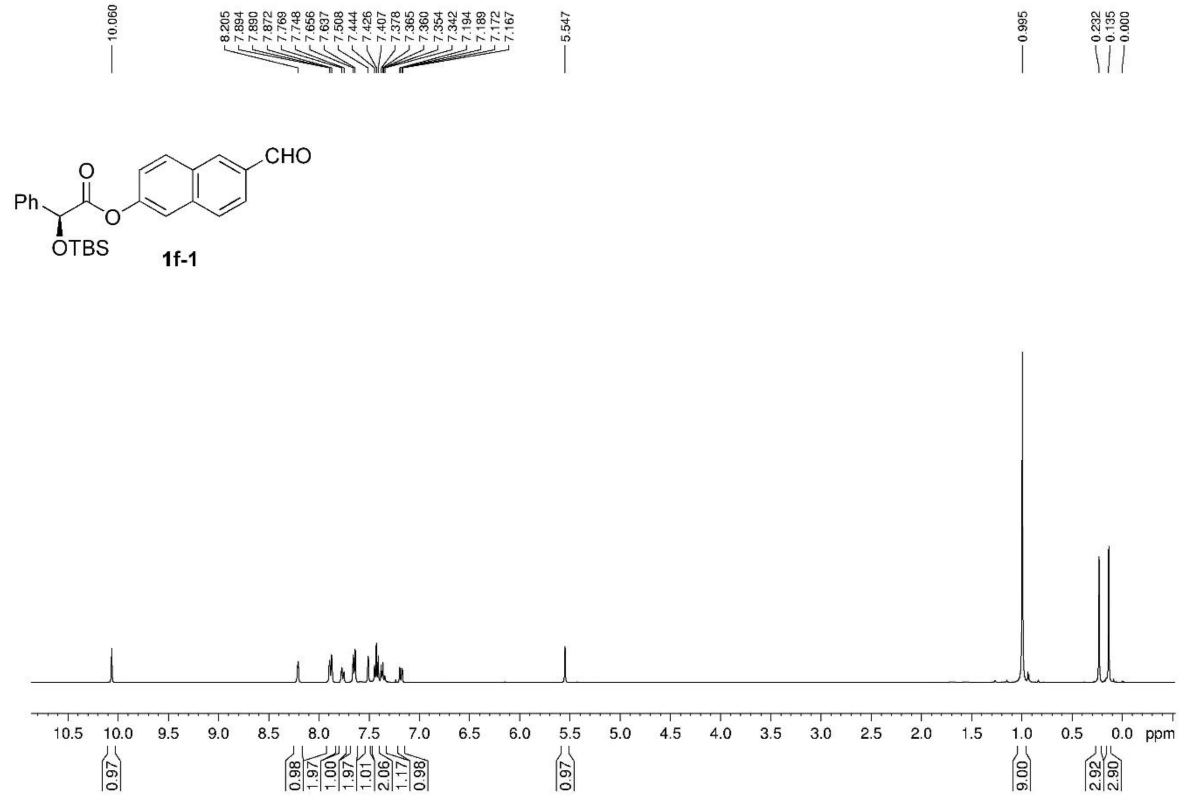
131.36
130.86
129.40
128.02
127.73
126.51
121.26
118.09
79.66
77.32
77.00
76.68
68.20
58.64
53.27
31.02
28.06
18.89
17.54
iPr
O
NHBoc
OMe
1e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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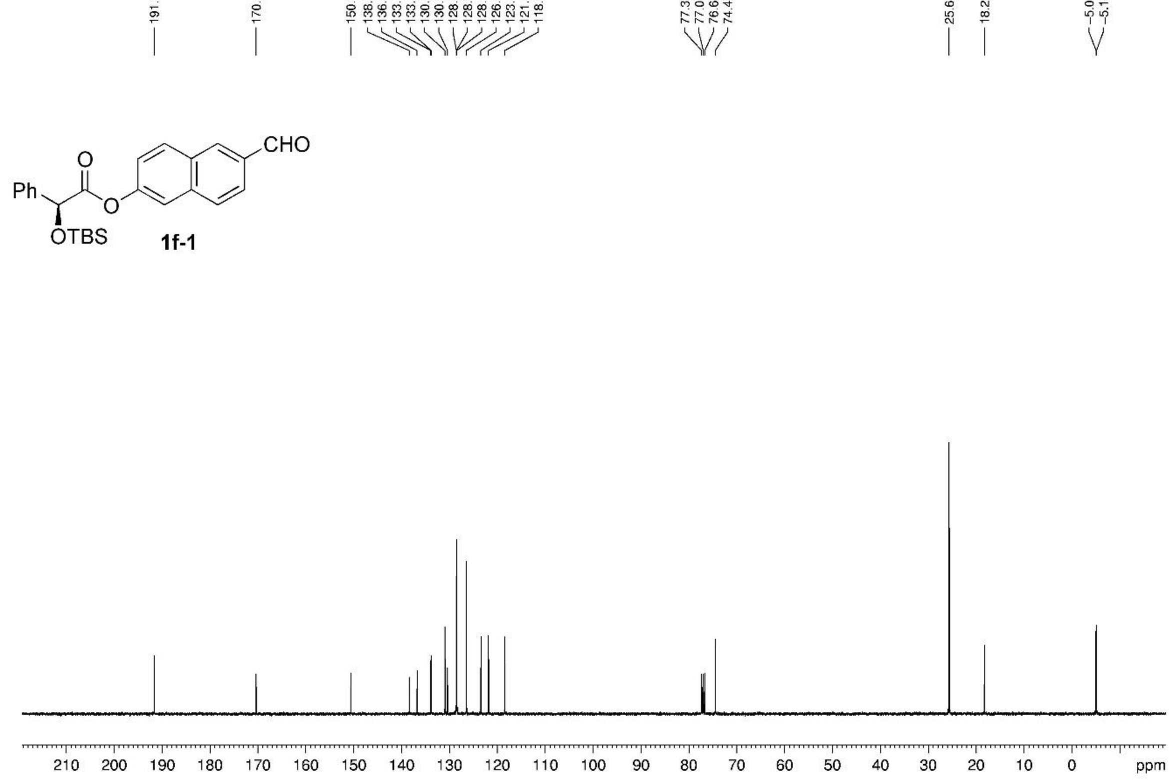
7.656
7.637
7.508
7.444
7.426
7.407
7.378
7.365
7.360
7.354
7.342
7.194
7.189
7.172
7.167
5.547
0.995
0.232
0.135
0.000
Ph
O
O
CHO
OTBS
1f-1
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.97
0.98
1.97
1.00
1.97
1.01
2.06
1.17
0.98
0.97
9.00
2.92
2.90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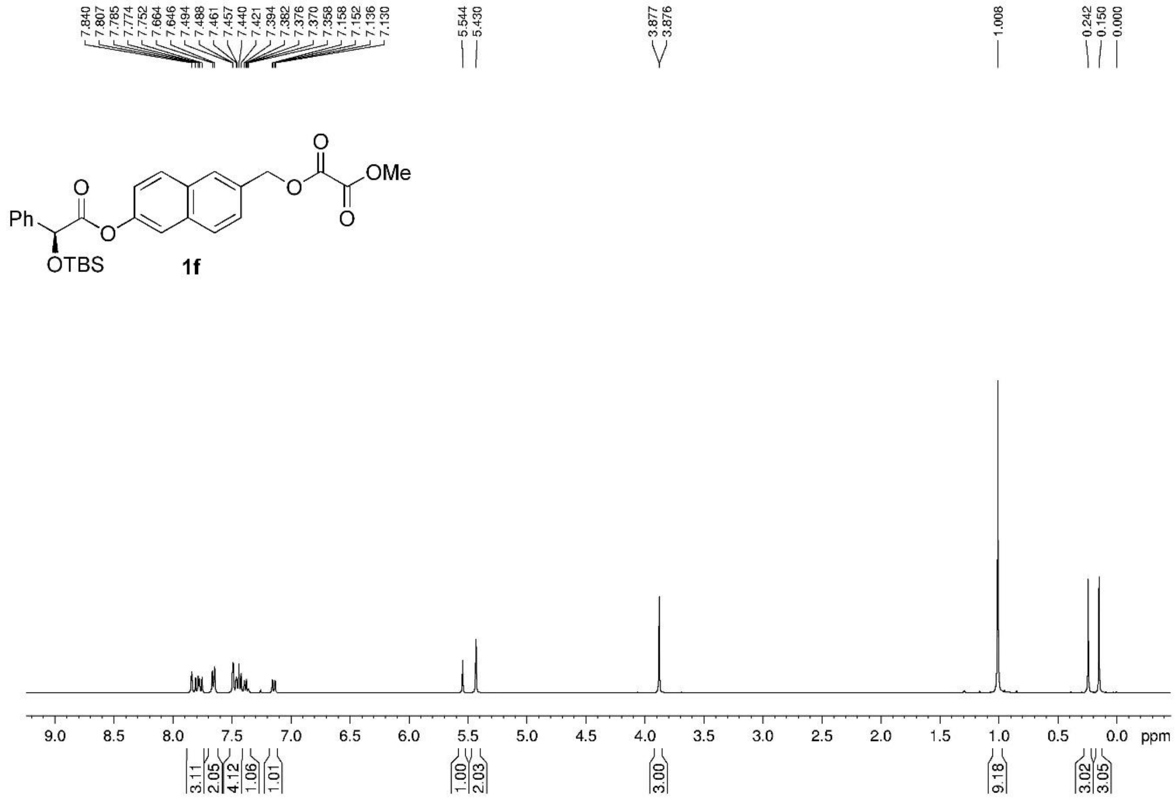
150.60
138.39
136.75
133.86
133.82
130.93
130.41
128.55
128.52
128.45
126.44
123.42
121.84
118.44
77.32
77.00
76.68
74.45
25.61
18.23
-5.04
-5.13
Ph
O
O
CHO
OTBS
1f-1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.752
7.664
7.646
7.494
7.488
7.461
7.457
7.440
7.421
7.394
7.382
7.376
7.370
7.358
7.158
7.152
7.136
7.130
5.544
5.430
3.877
3.876
1.008
0.242
0.150
0.000
O
OMe
Ph
O
O
O
O
OTBS
1f
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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ppm
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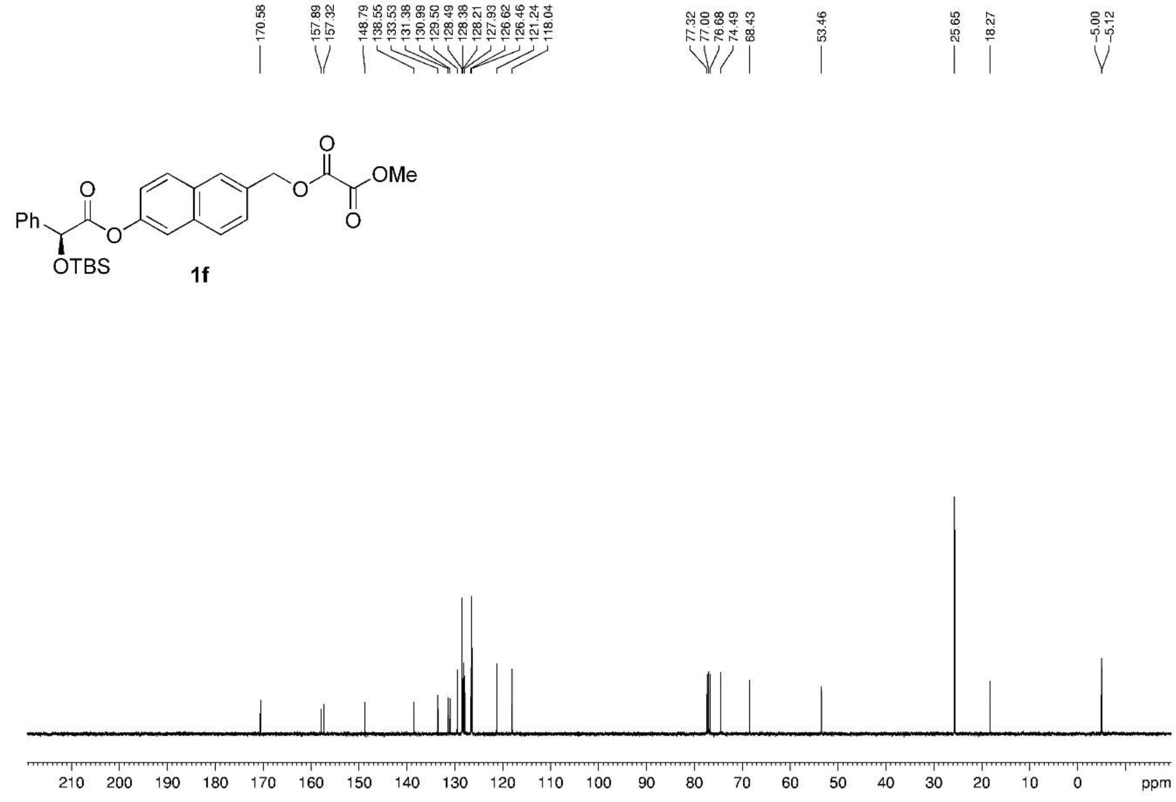
2.05
4.12
1.06
1.01
1.00
2.03
3.00
9.18
3.02
3.05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133.53
131.38
130.99
129.50
128.49
128.38
128.21
127.93
126.62
126.46
121.24
118.04
77.32
77.00
76.68
74.49
68.43
53.46
25.65
18.27
-5.00
-5.12
O
OMe
Ph
O
O
O
O
OTBS
1f
210
200
190
180
170
160
150
140
130
120
110
100
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80
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60
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20
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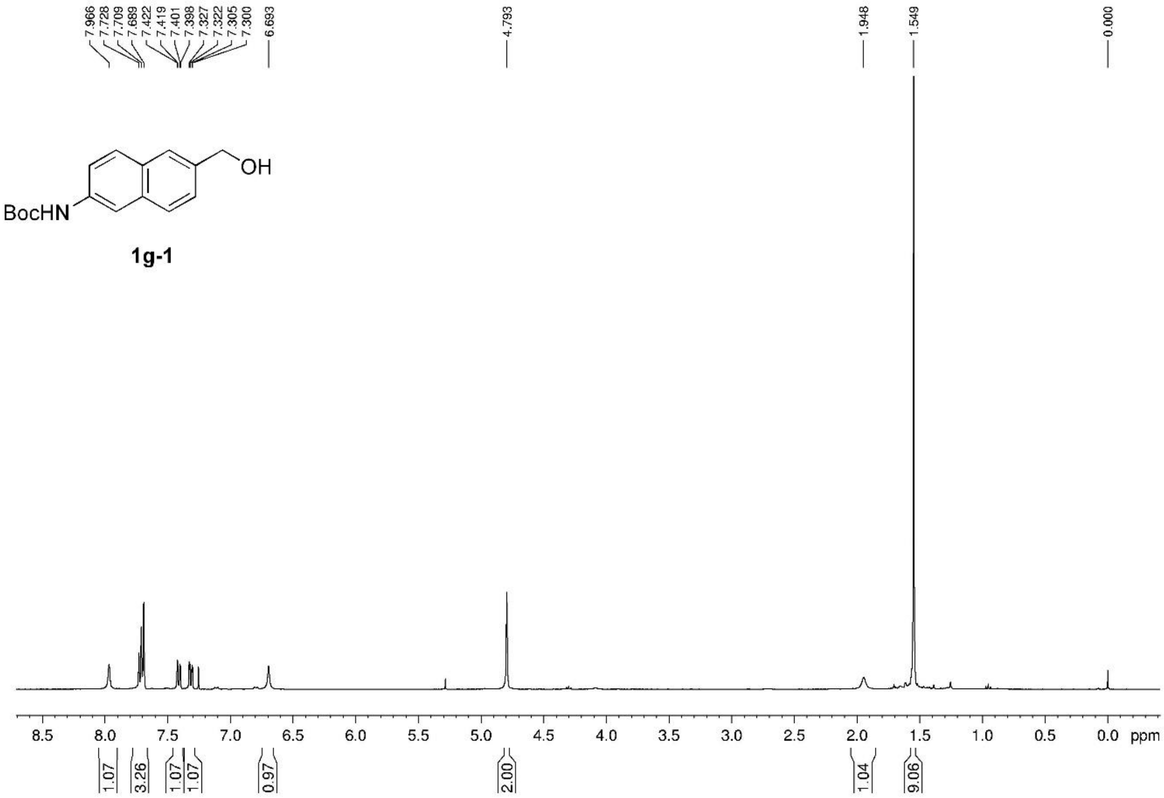
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7.419
7.401
7.398
7.327
7.322
7.305
7.300
6.693
4.793
1.948
1.549
0.000
OH
BocHN
1g-1
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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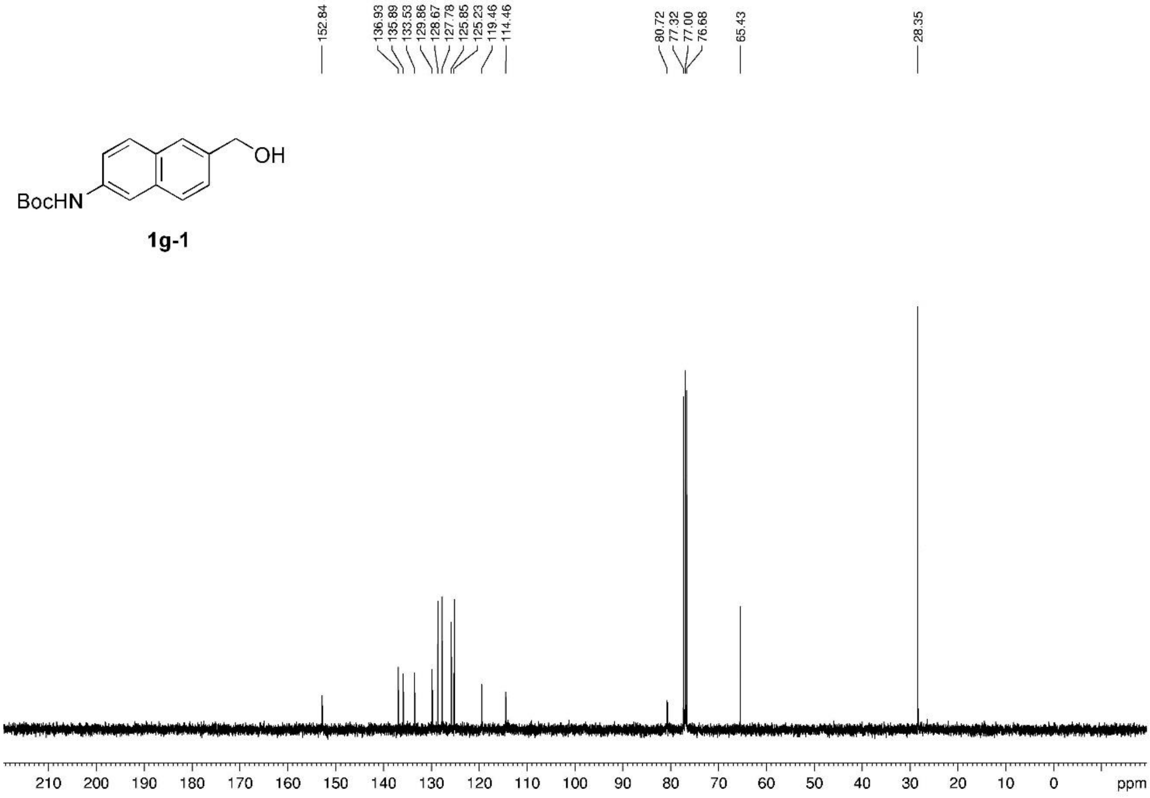
1.0
0.5
0.0
ppm
1.07
3.26
1.07
1.07
0.97
2.00
1.04
9.06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135.89
133.53
129.86
128.67
127.78
125.85
125.23
119.46
114.46
80.72
77.32
77.00
76.68
65.43
28.35
OH
BocHN
1g-1
210
200
190
180
170
160
150
140
130
120
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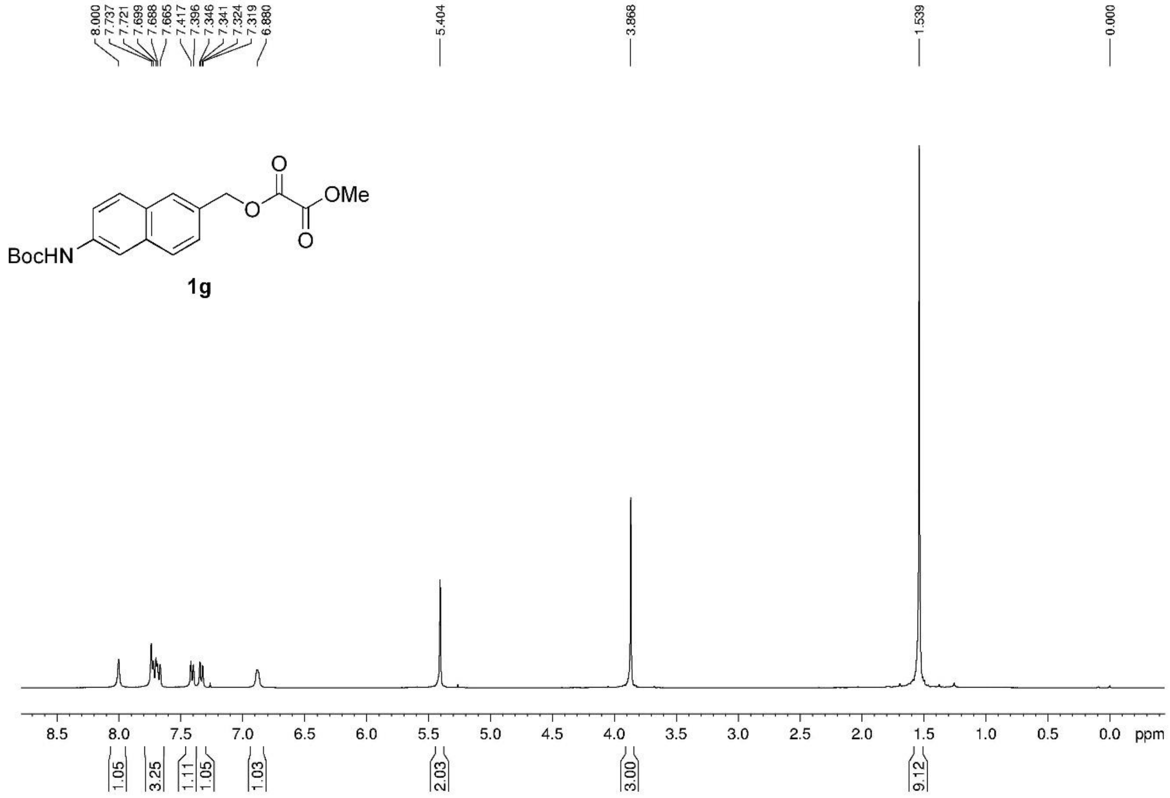
100
90
80
70
60
50
40
30
20
10
0
ppm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7.341
7.324
7.319
6.880
5.404
3.868
1.539
0.000
O
OMe
O
BocHN
1g
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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0.0
ppm
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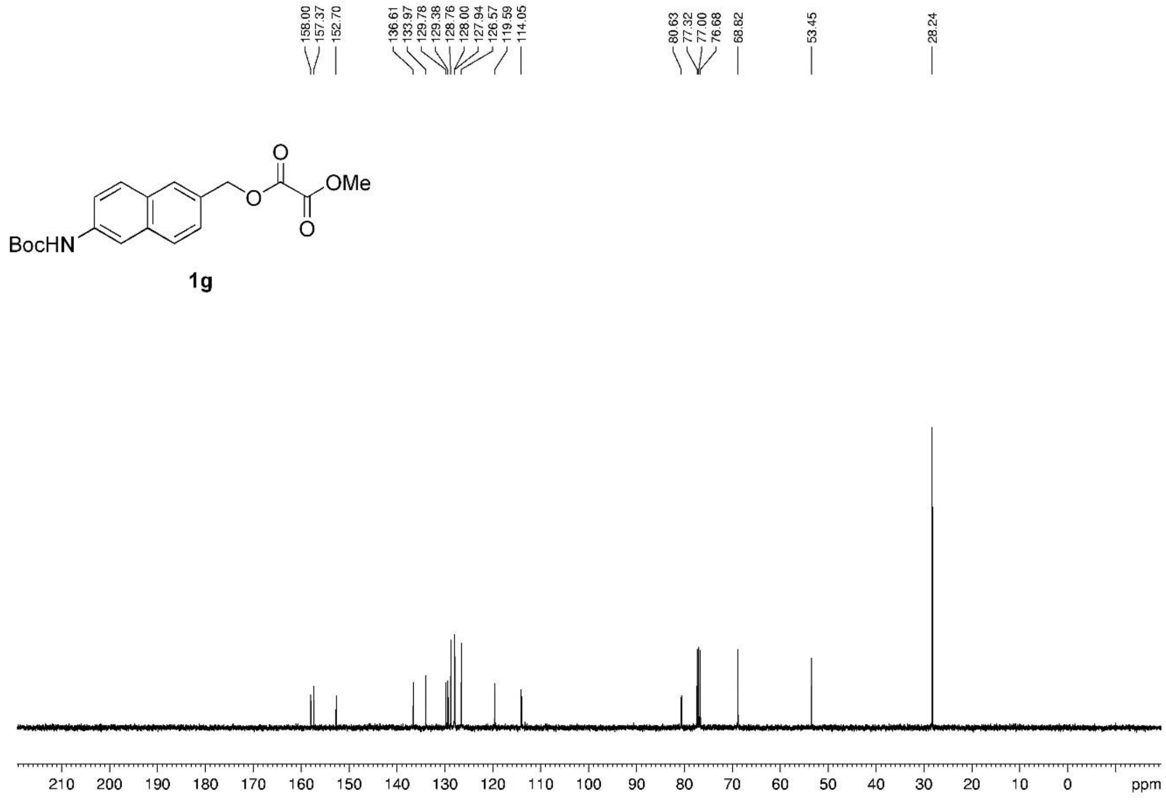
3.25
1.11
1.05
1.03
2.03
3.00
9.12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129.78
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128.76
128.00
127.94
126.57
119.59
114.05
80.63
77.32
77.00
76.68
68.82
53.45
28.24
O
OMe
O
BocHN
1g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
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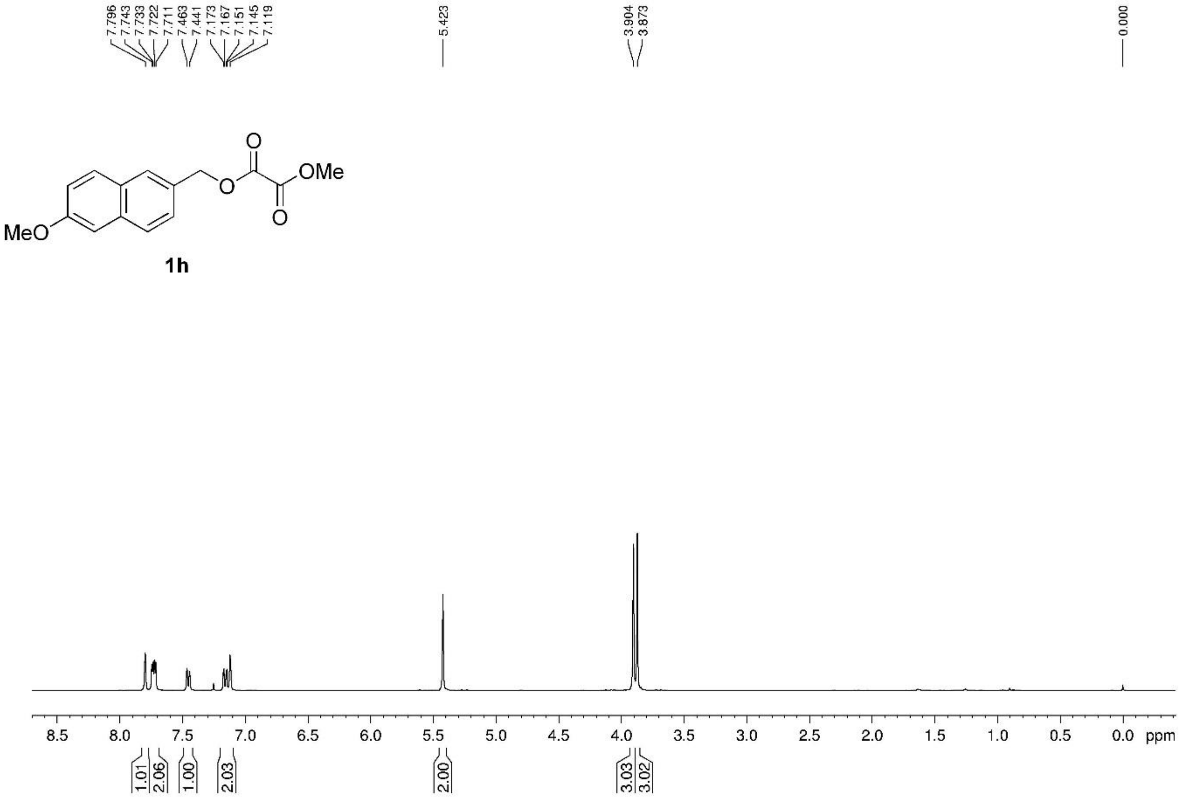
60
50
40
30
20
10
0
ppm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7.173
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7.151
7.145
7.119
5.423
3.904
3.873
0.000
O
OMe
MeO
O
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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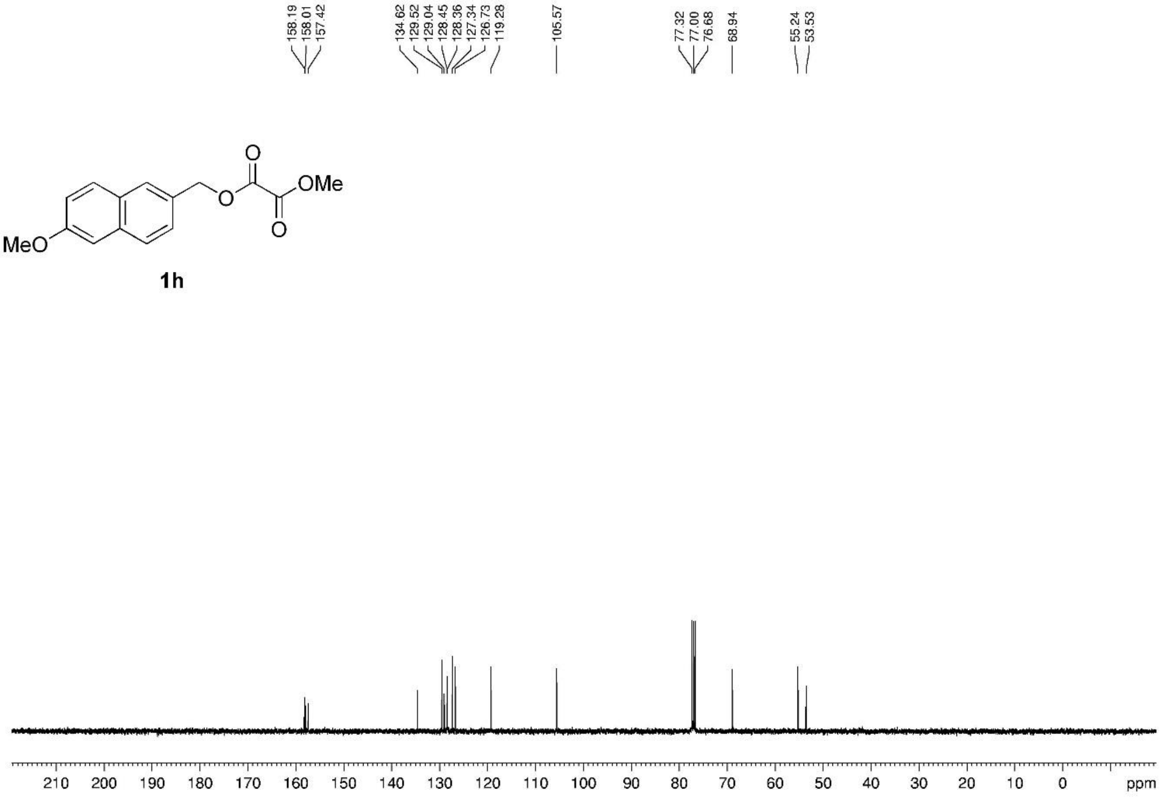
2.06
1.00
2.03
2.00
3.03
3.02

1h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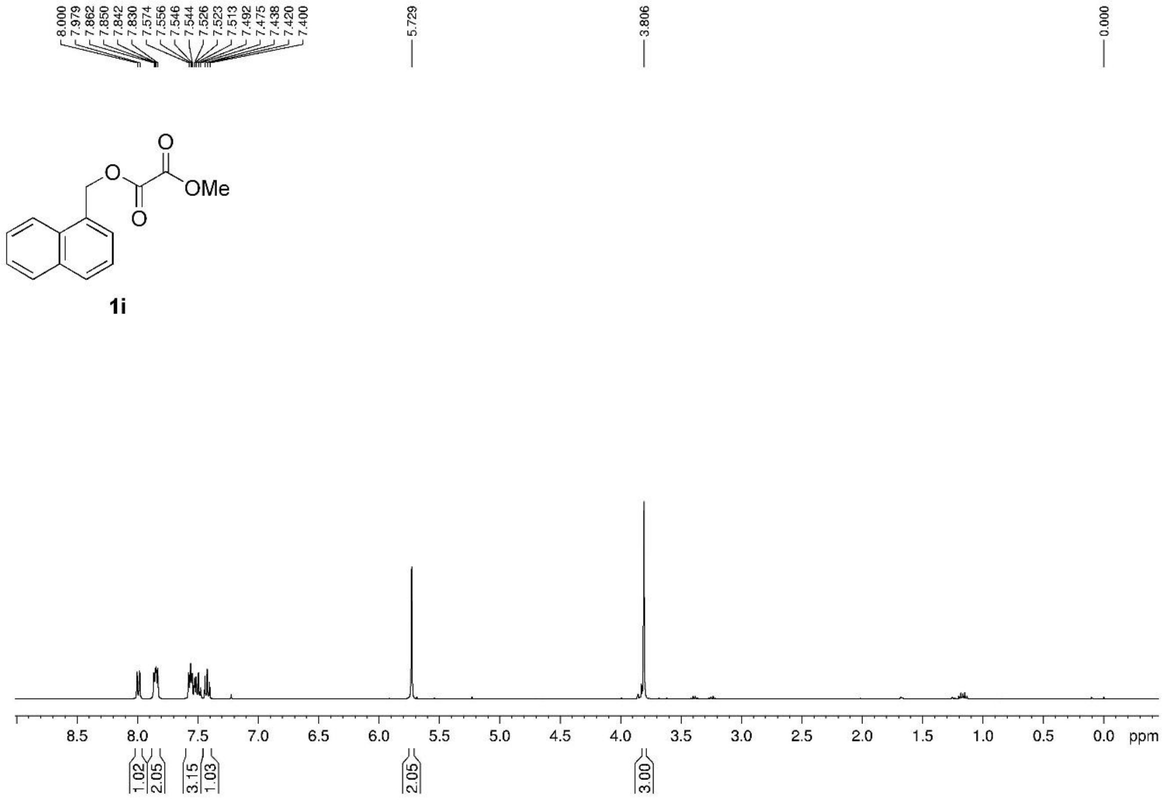
129.04
128.45
128.36
127.34
126.73
119.28
105.57
77.32
77.00
76.68
68.94
55.24
53.53
O
OMe
MeO
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

1h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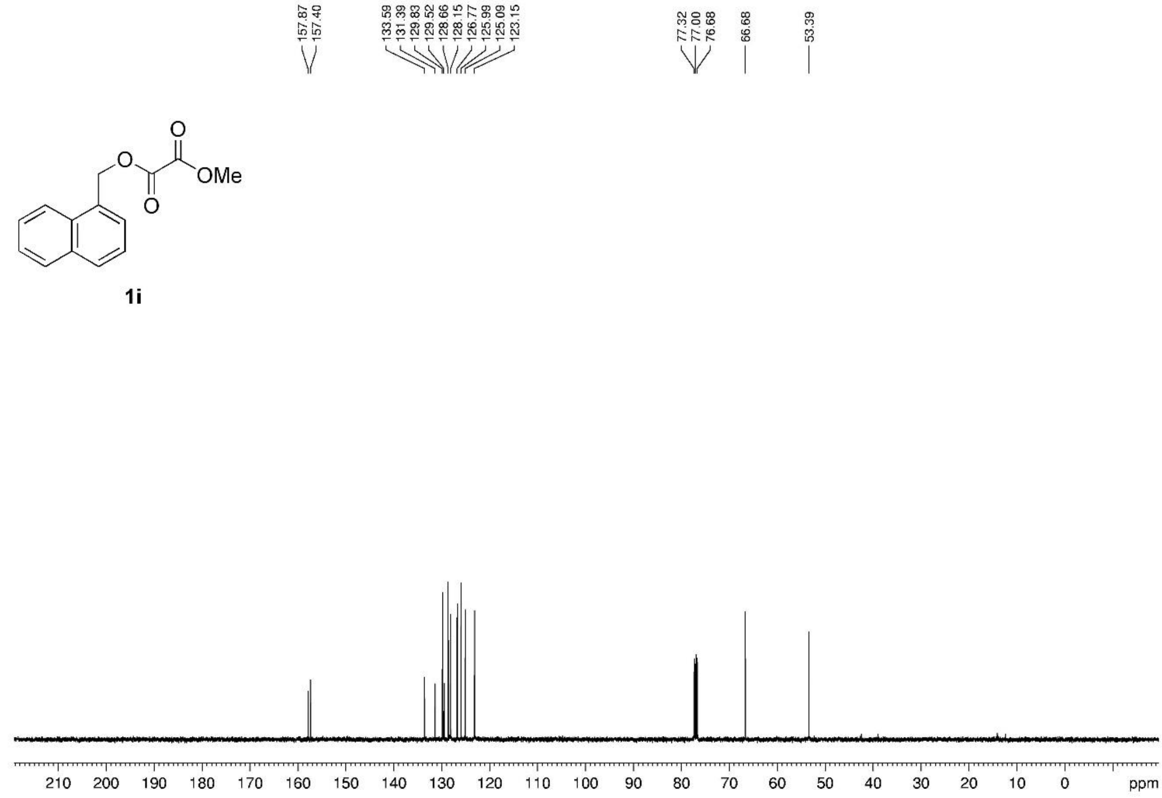
7.544
7.526
7.523
7.513
7.492
7.475
7.438
7.420
7.400
5.729
3.806
0.000
O
O
O
OMe
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.02
2.05
3.15
1.03
2.05
3.00

1i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129.52
128.66
128.15
126.77
125.99
125.09
123.15
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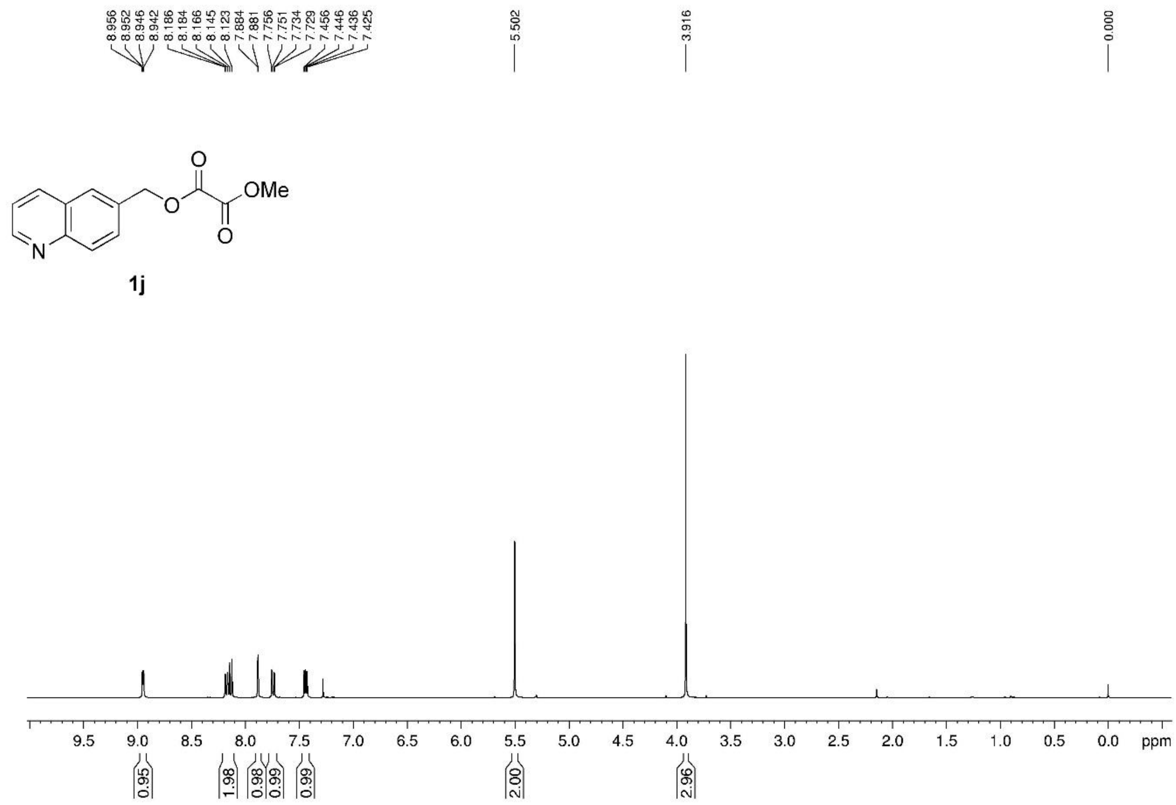
77.00
76.68
66.68
53.39
O
O
O
OMe
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

1i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8.145
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7.881
7.756
7.751
7.734
7.729
7.456
7.446
7.436
7.425
5.502
3.916
0.000
O
OMe
O
O
N
1j
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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0.5
0.0
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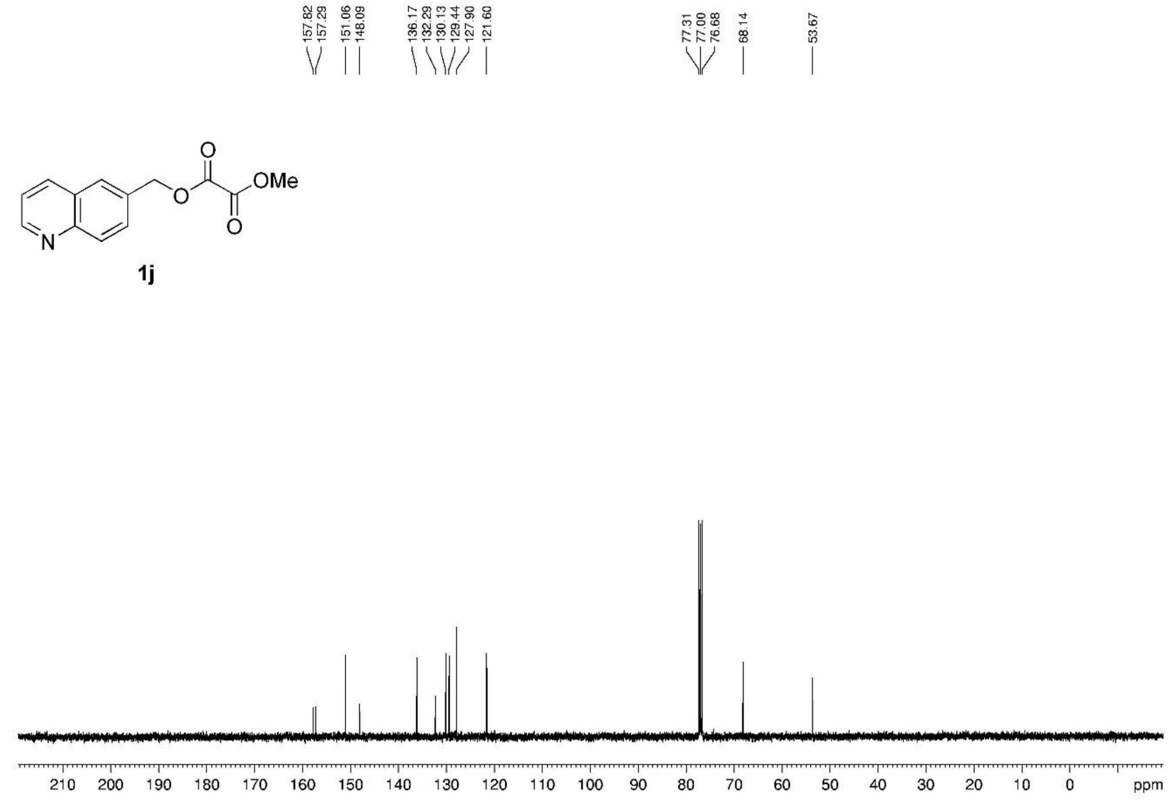
0.99
2.00
2.96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132.29
130.13
129.44
127.90
121.60
77.31
77.00
76.68
68.14
53.67
O
OMe
O
O
N
1j
210
200
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140
130
120
110
100
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80
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60
50
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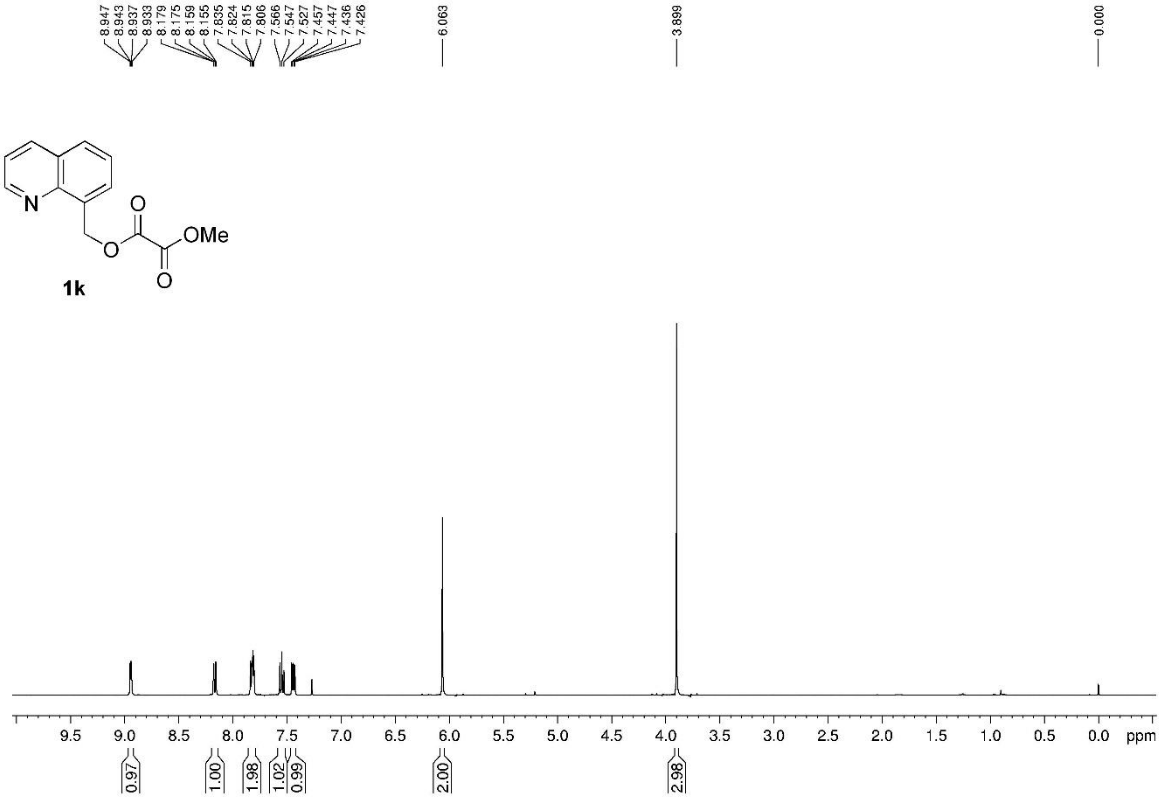
20
10
0
ppm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8.155
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7.824
7.815
7.806
7.566
7.547
7.527
7.457
7.447
7.436
7.426
6.063
3.899
0.000
N
O
OMe
O
O
1k
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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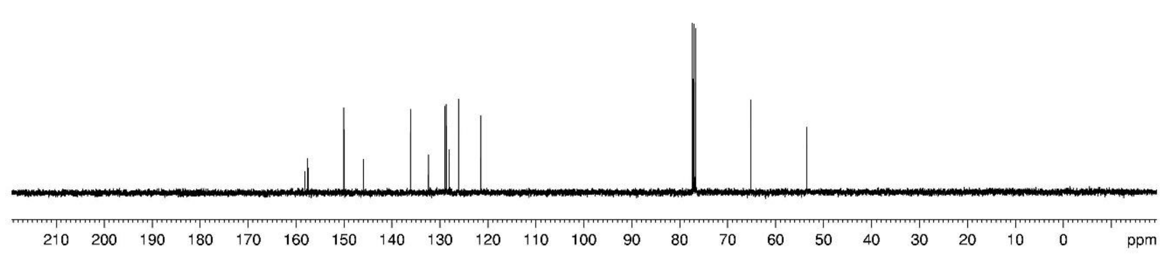
1.98
1.02
0.99
2.00
2.98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132.38
128.99
128.67
128.10
126.11
121.44
77.32
77.00
76.68
65.15
53.51
N
O
OMe
O
O
1k
210
200
190
180
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160
150
140
130
120
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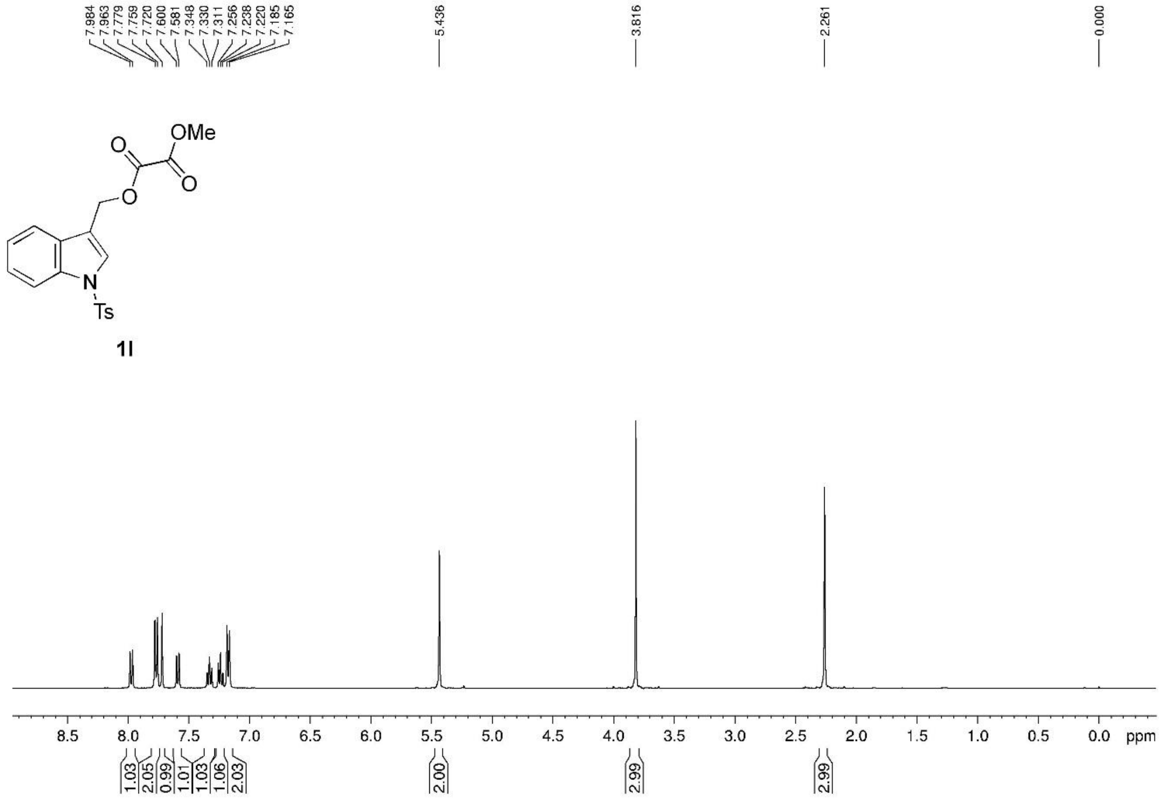
100
90
80
70
60
50
40
30
20
10
0
ppm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7.348
7.330
7.311
7.256
7.238
7.220
7.185
7.165
5.436
3.816
2.261
0.000
OMe
O
O
O
N
Ts
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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ppm
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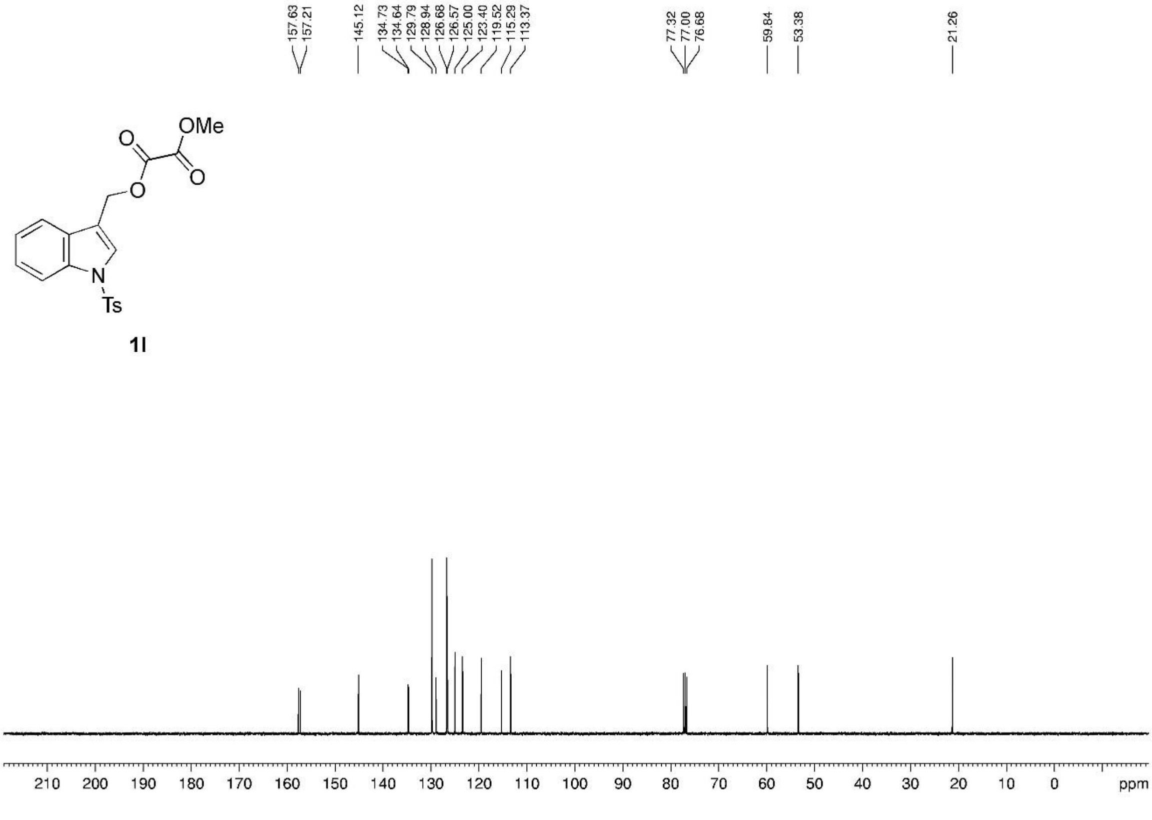
2.05
0.99
1.01
1.03
1.06
2.03
2.00
2.99
2.99

1l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134.64
129.79
128.94
126.68
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125.00
123.40
119.52
115.29
113.37
77.32
77.00
76.68
59.84
53.38
21.26
OMe
O
O
O
N
Ts
210
200
190
180
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40
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20
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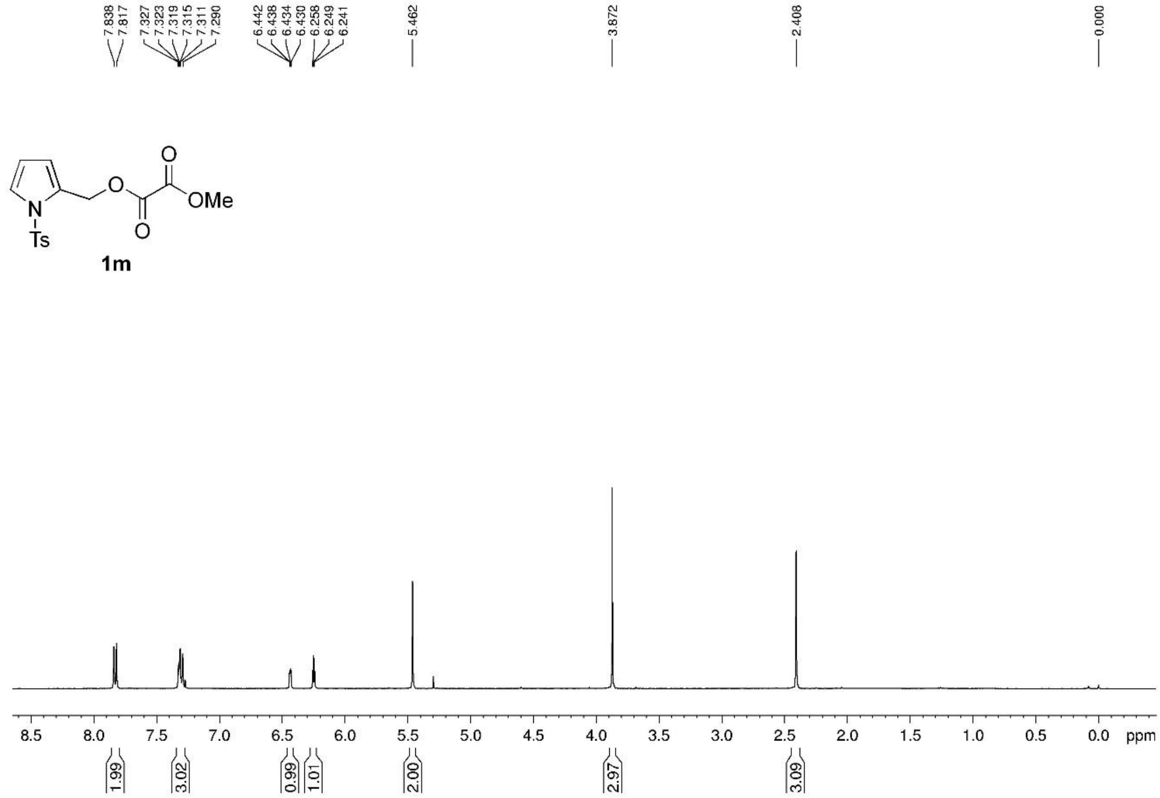
0
ppm

1l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7.327
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7.315
7.311
7.290
6.442
6.438
6.434
6.430
6.258
6.249
6.241
5.462
3.872
2.408
0.000
O
OMe
O
N
Ts
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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ppm
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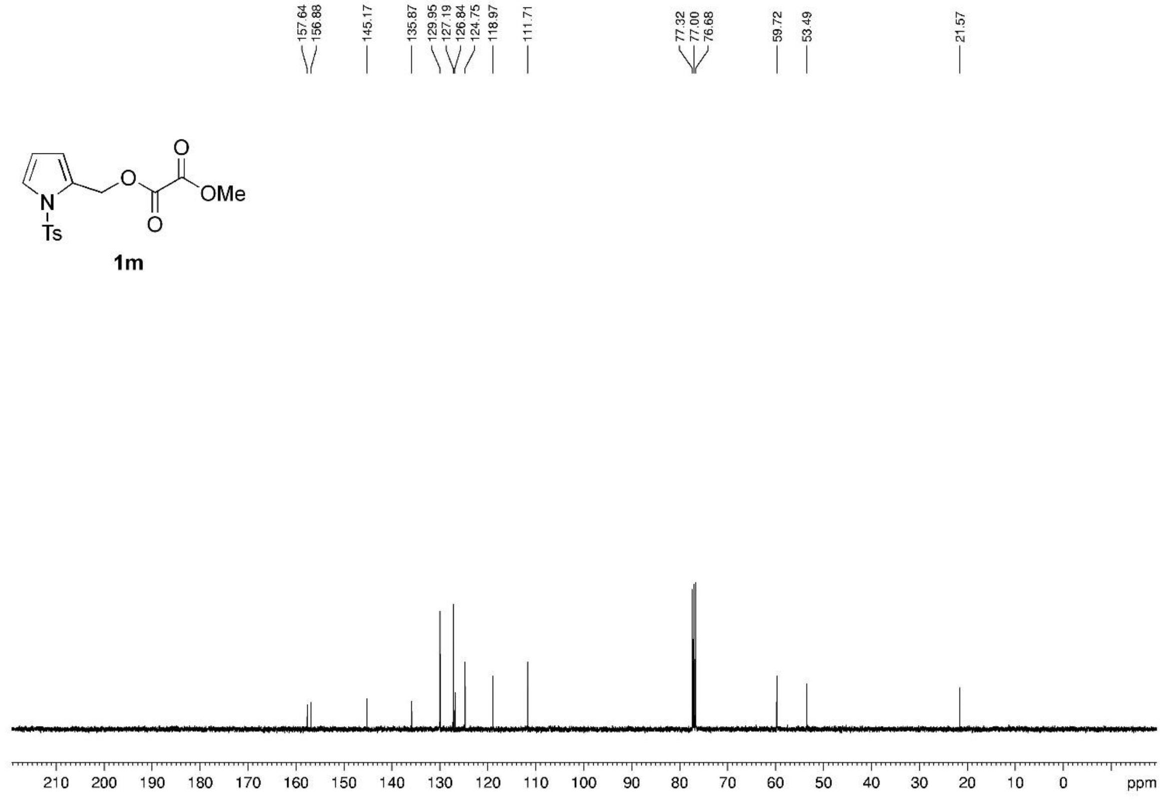
0.99
1.01
2.00
2.97
3.09

1m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129.95
127.19
126.84
124.75
118.97
111.71
77.32
77.00
76.68
59.72
53.49
21.57
O
OMe
O
N
Ts
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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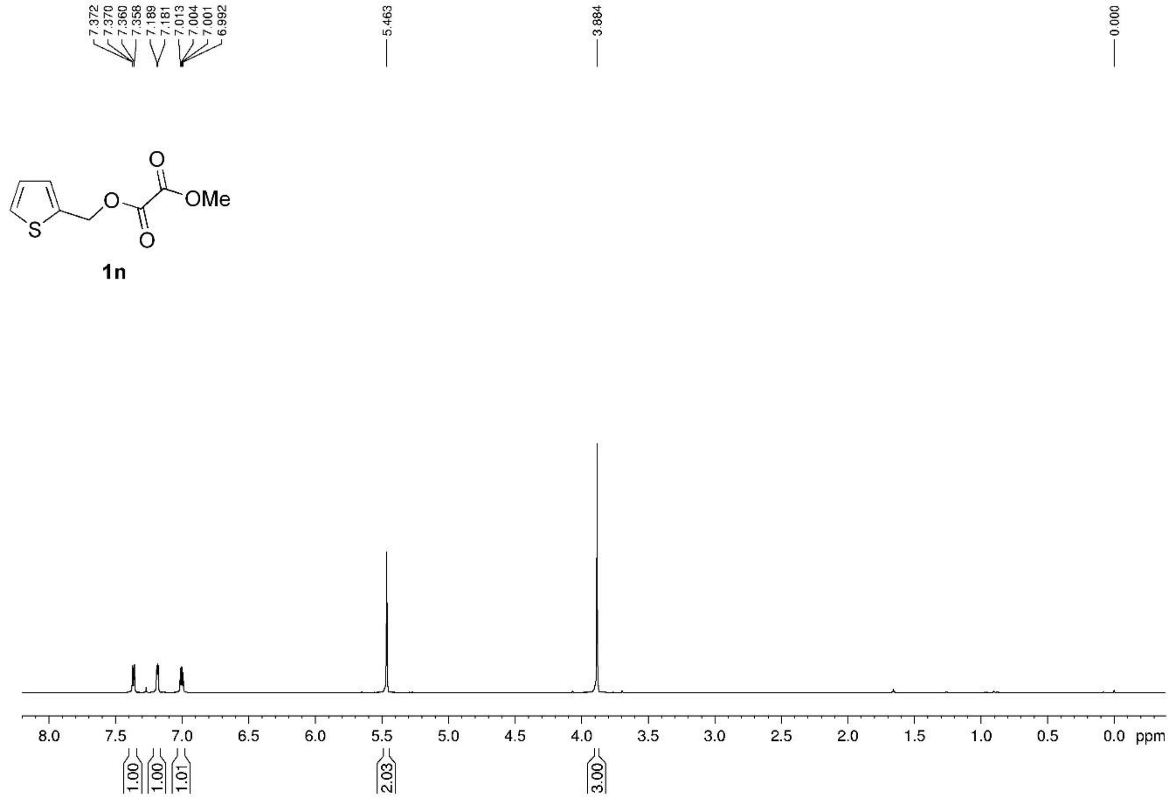
30
20
10
0
ppm

1m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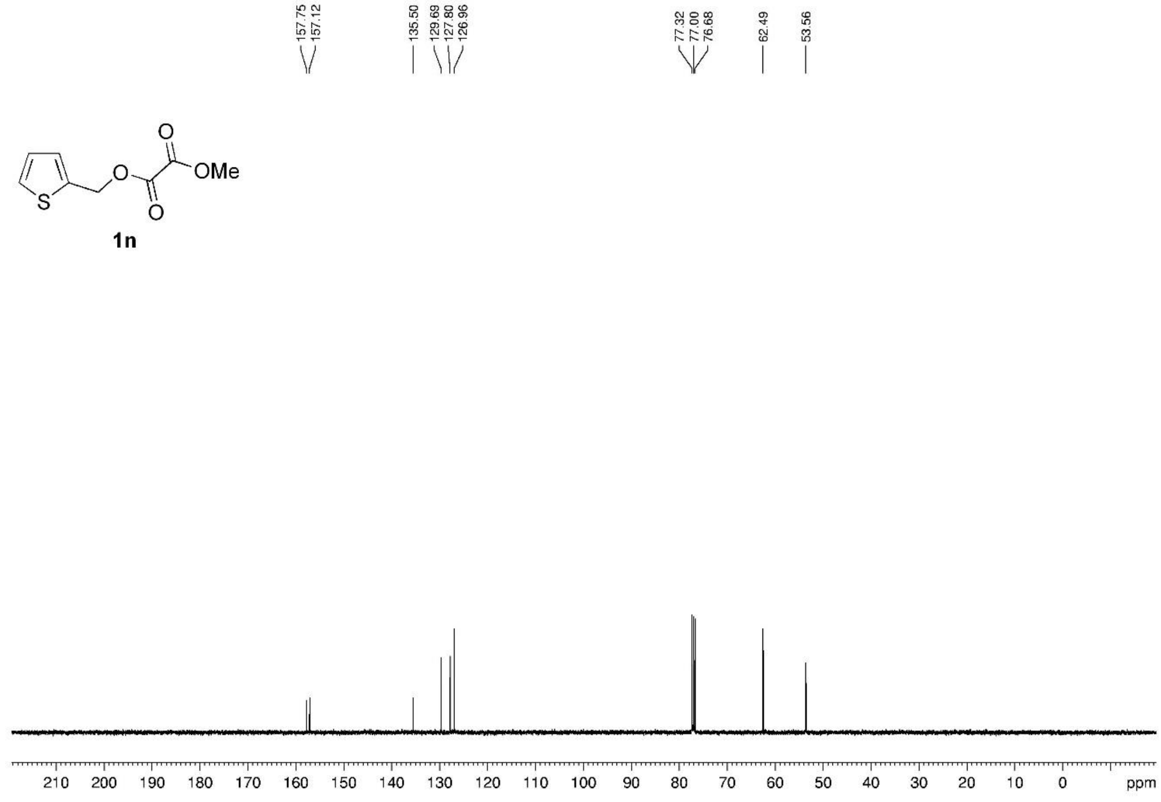
7.358
7.189
7.181
7.013
7.004
7.001
6.992
5.463
3.884
0.000
OMe
O
O
S
1n
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
1.00
1.00
1.01
2.03
3.00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127.80
126.96
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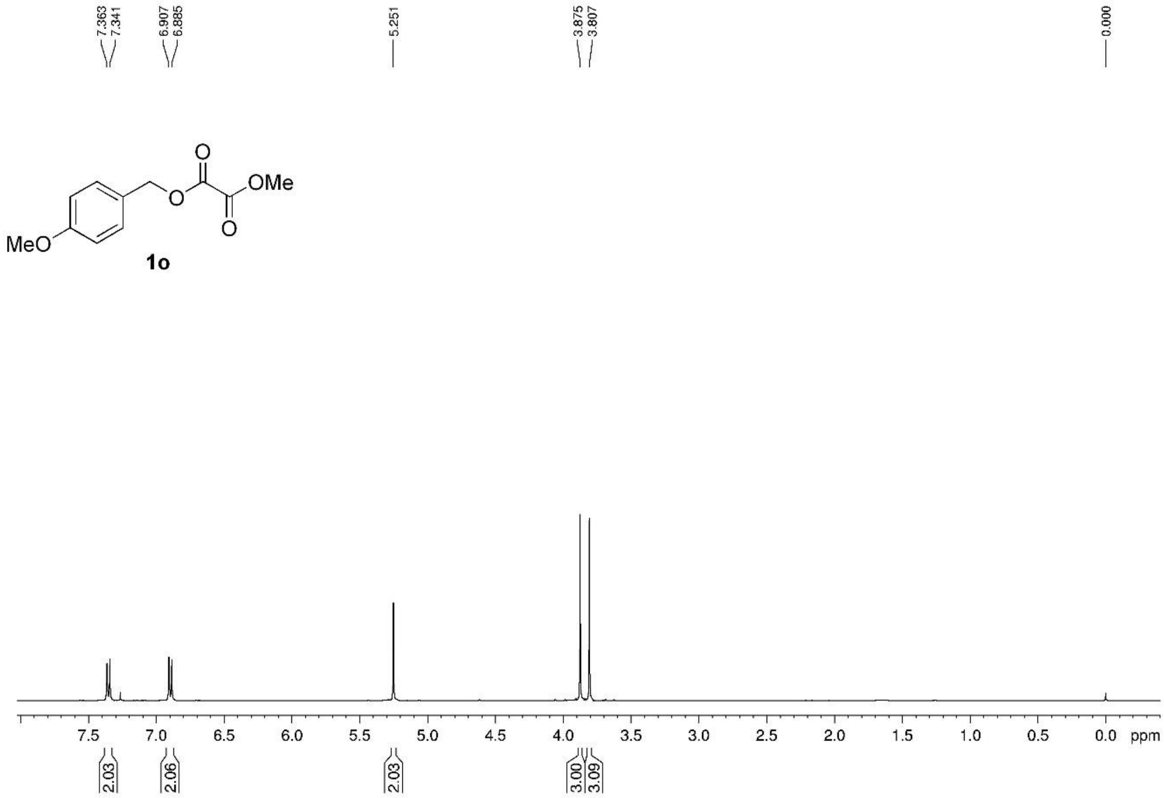
77.00
76.68
62.49
53.56
OMe
O
O
S
1n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.341
6.907
6.885
5.251
3.875
3.807
0.000
O
OMe
O
O
MeO
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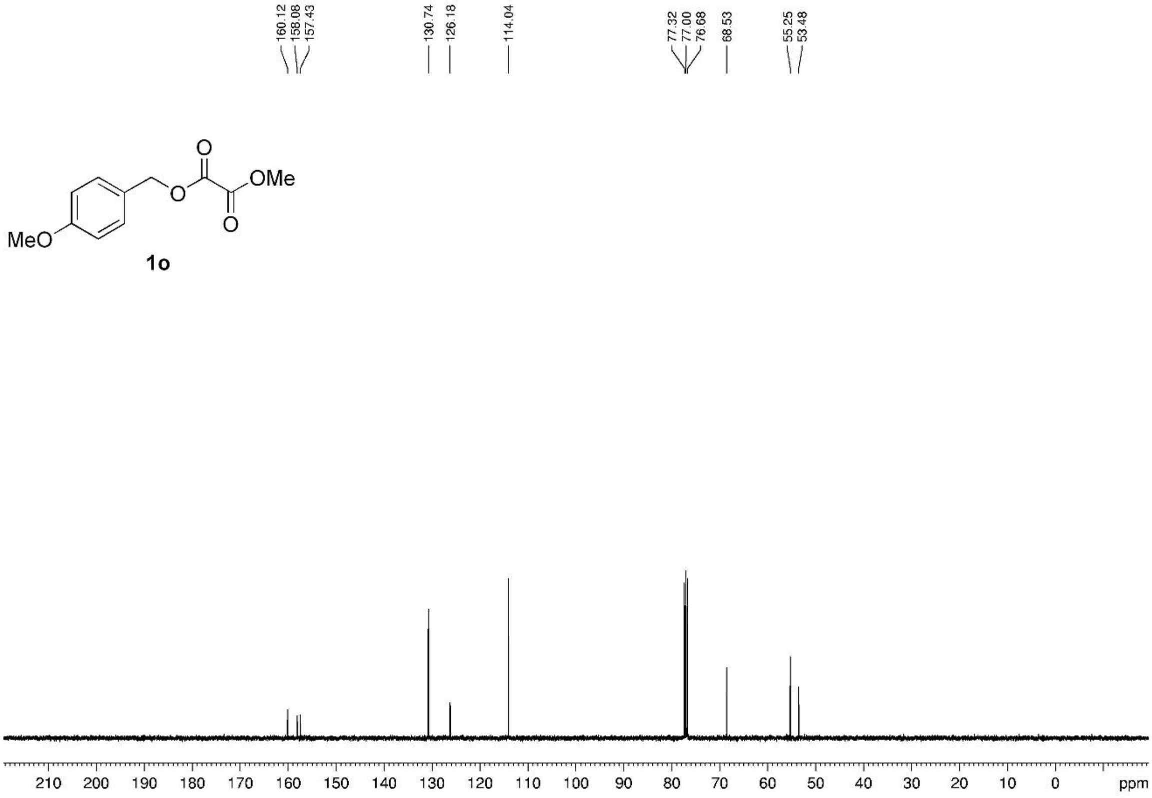
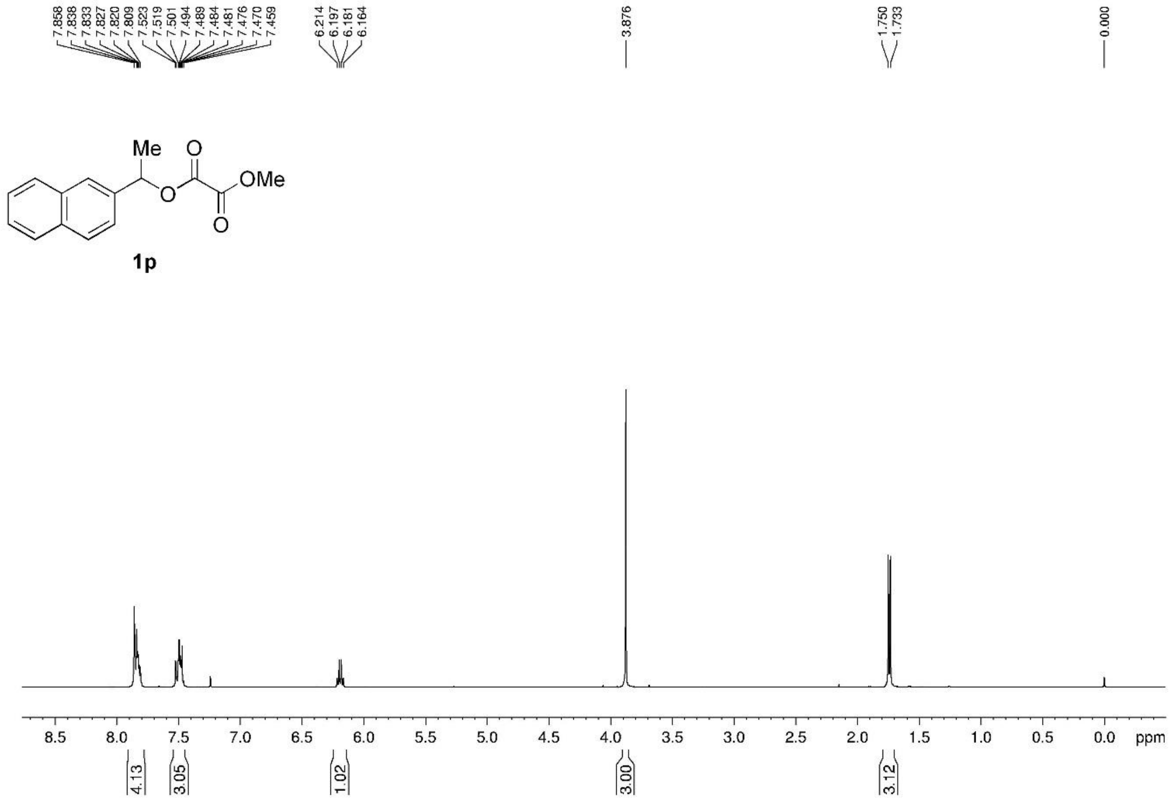
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.03
2.06
2.03
3.00
3.09


160.12
158.08
157.43
130.74
126.18
114.04
77.32
77.00
76.68
68.53
55.25
53.48
O
OMe
O
O
MeO
1o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.820
7.809
7.523
7.519
7.501
7.494
7.489
7.484
7.481
7.476
7.470
7.459
6.214
6.197
6.181
6.164
3.876
1.750
1.733
0.000
Me
O
OMe
O
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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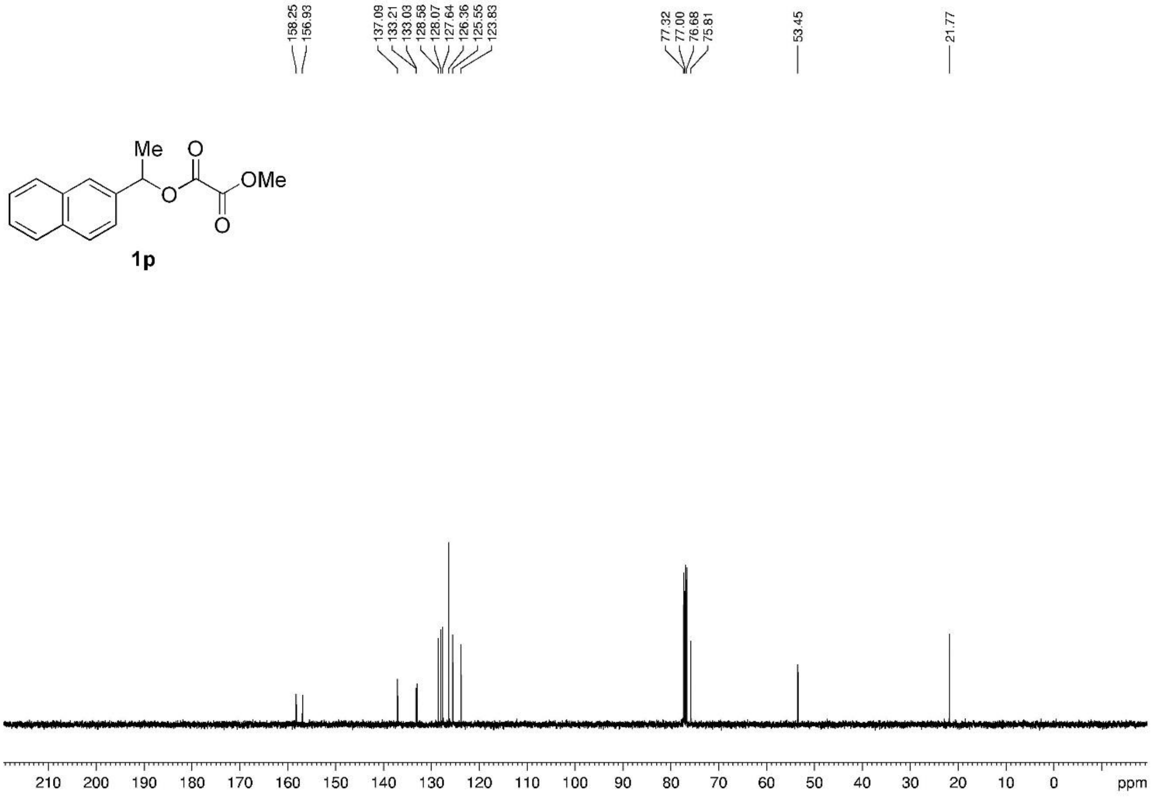
3.05
1.02
3.00
3.12

1p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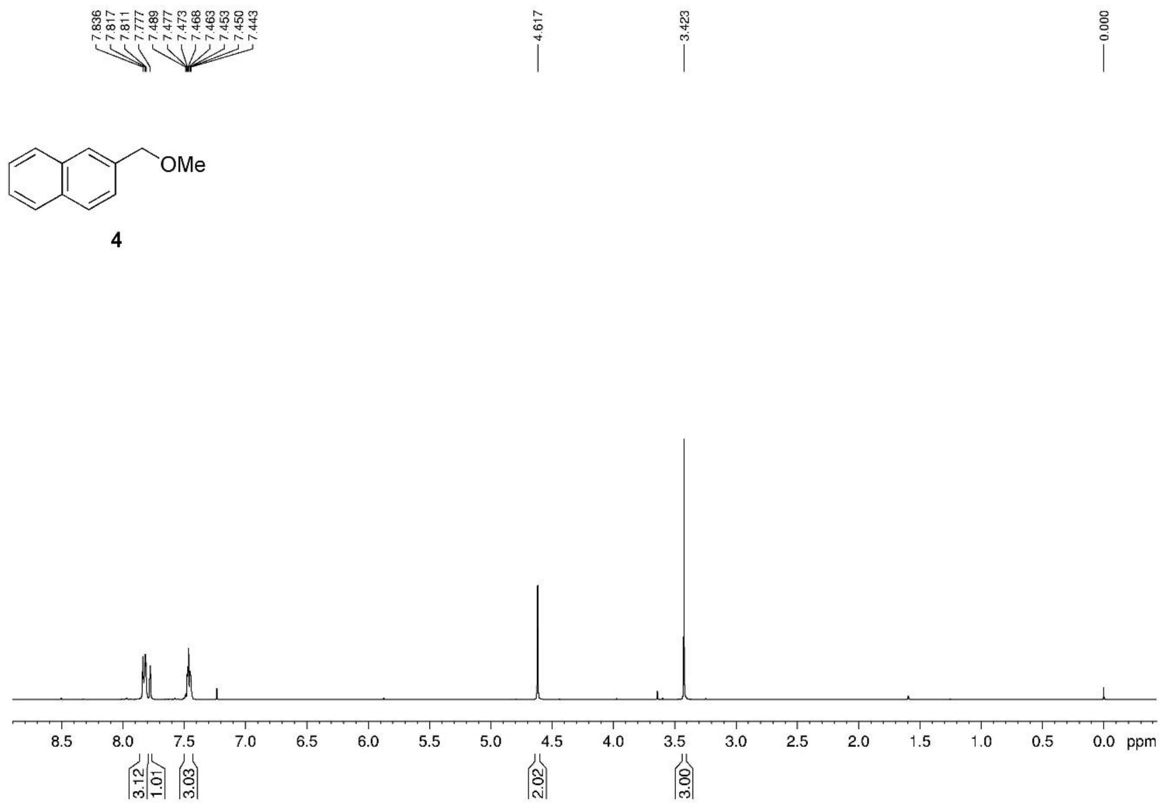
137.09
133.21
133.03
128.58
128.07
127.64
126.36
125.55
123.83
77.32
77.00
76.68
75.81
53.45
21.77
Me
O
OMe
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

1p



4

7.836
7.817
7.811
7.777
7.489
7.477
7.473
7.468
7.463
7.453
7.450
7.443
4.617
3.423
0.000
OMe
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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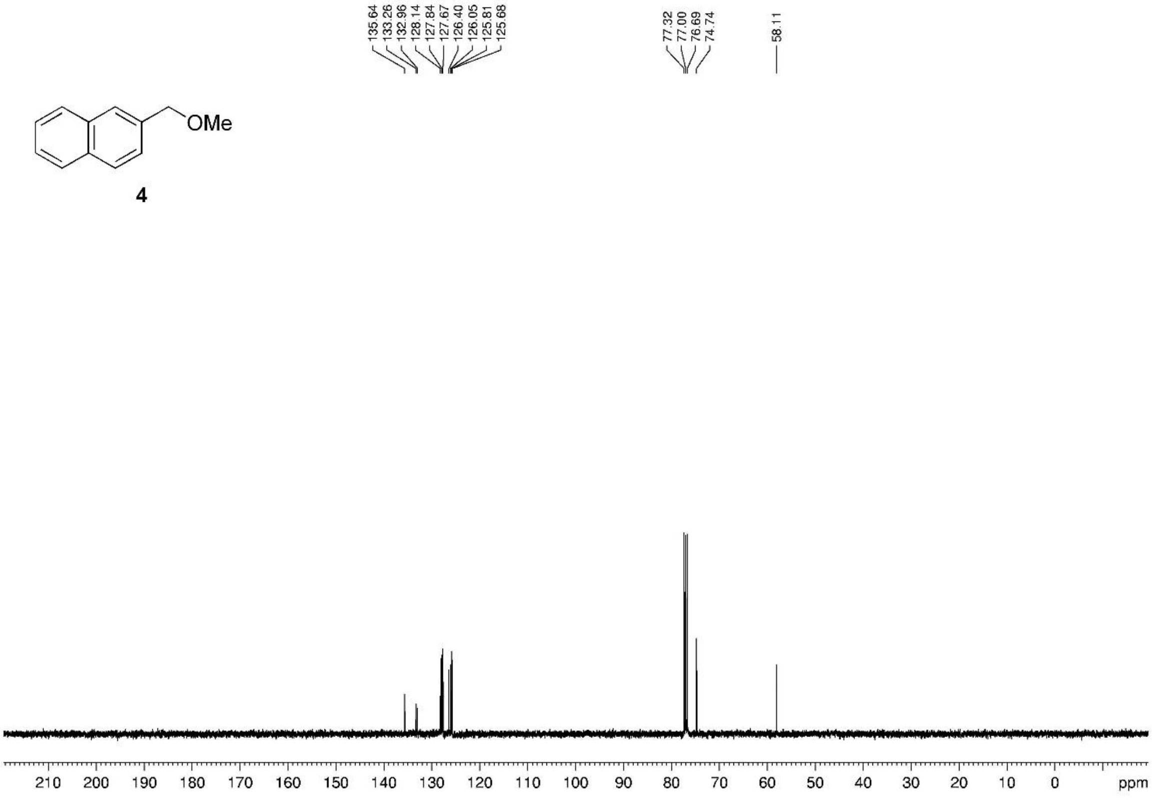
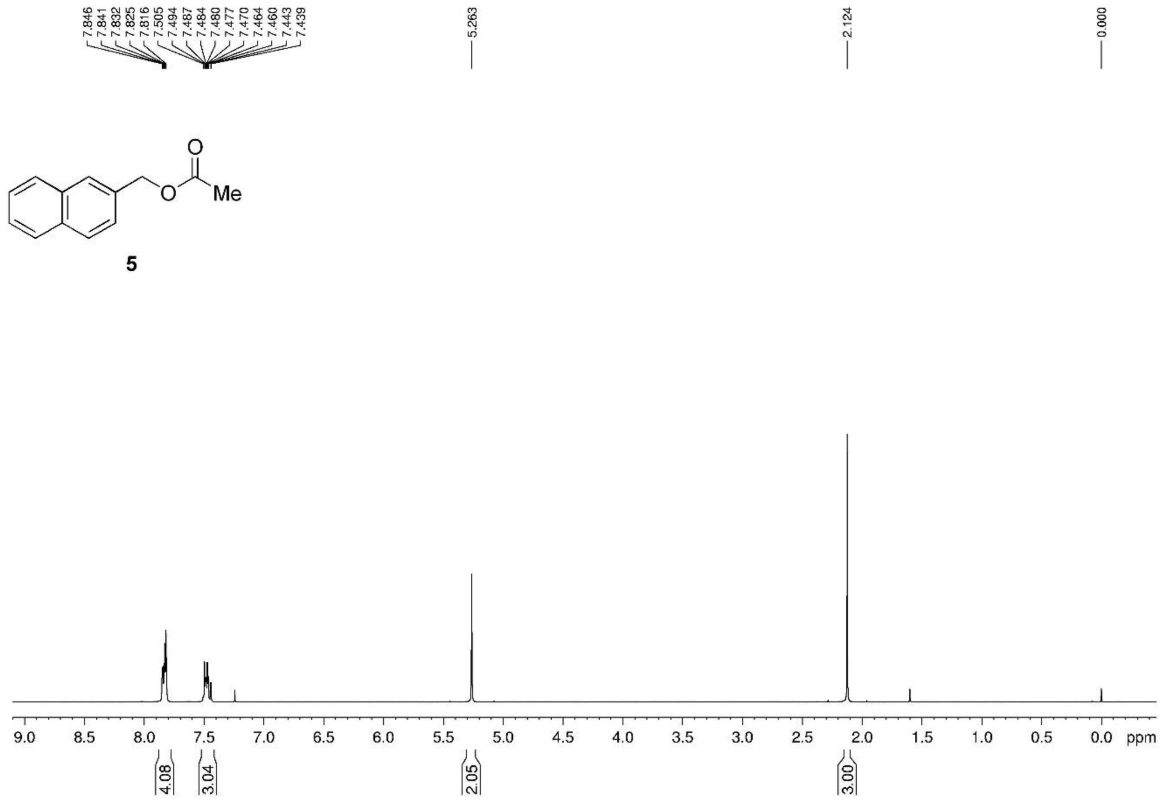
1.01
3.03
2.02
3.00



4

135.64
133.26
132.96
128.14
127.84
127.67
126.40
126.05
125.81
125.68
77.32
77.00
76.69
74.74
58.11
OMe
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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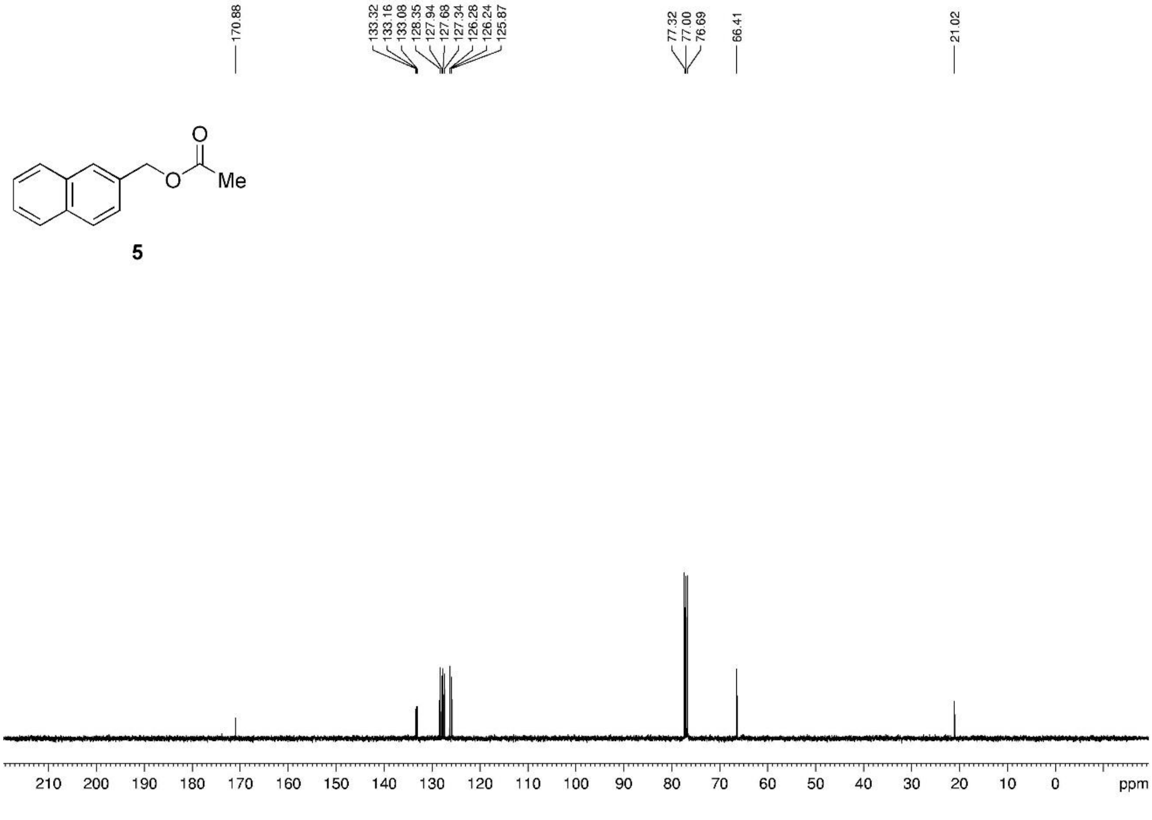
7.832
7.825
7.816
7.505
7.494
7.487
7.484
7.480
7.477
7.470
7.464
7.460
7.443
7.439
5.263
2.124
0.000
O
O
Me
5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.08
3.04
2.05
3.00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133.32
133.16
133.08
128.35
127.94
127.68
127.34
126.28
126.24
125.87
77.32
77.00
76.69
66.41
21.02
O
O
Me
5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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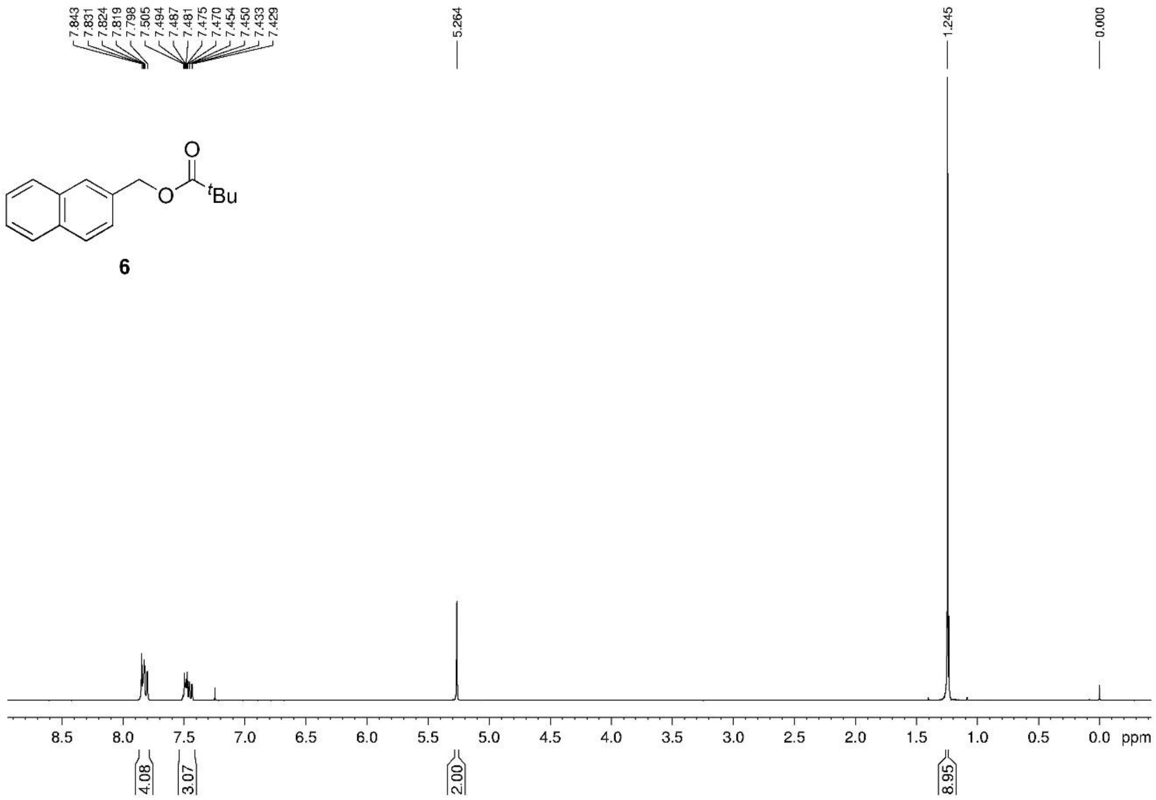
30
20
10
0
ppm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7.824
7.819
7.798
7.505
7.494
7.487
7.481
7.475
7.470
7.454
7.450
7.433
7.429
5.264
1.245
0.000
O
O
tBu
6
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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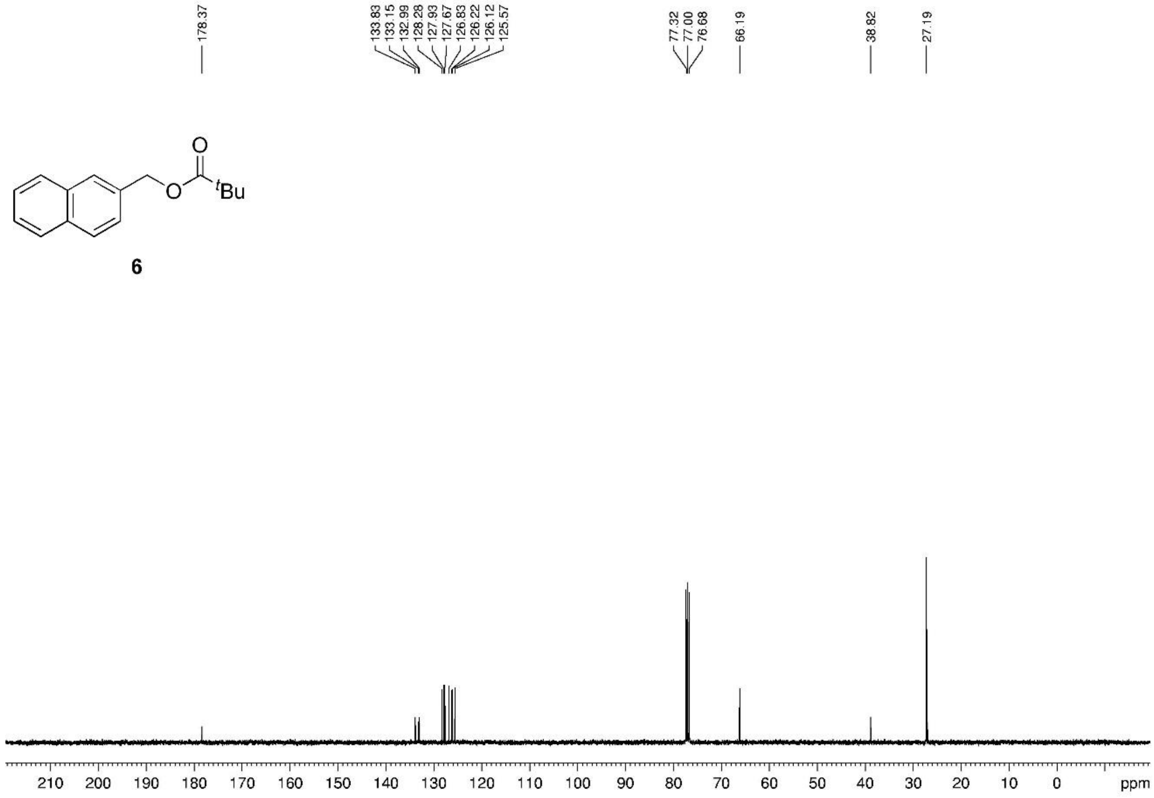
3.07
2.00
8.95


178.37
133.83
133.15
132.99
128.28
127.93
127.67
126.83
126.22
126.12
125.57
77.32
77.00
76.68
66.19
38.82
27.19
O
O
tBu
6
210
200
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180
170
160
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140
130
120
110
100
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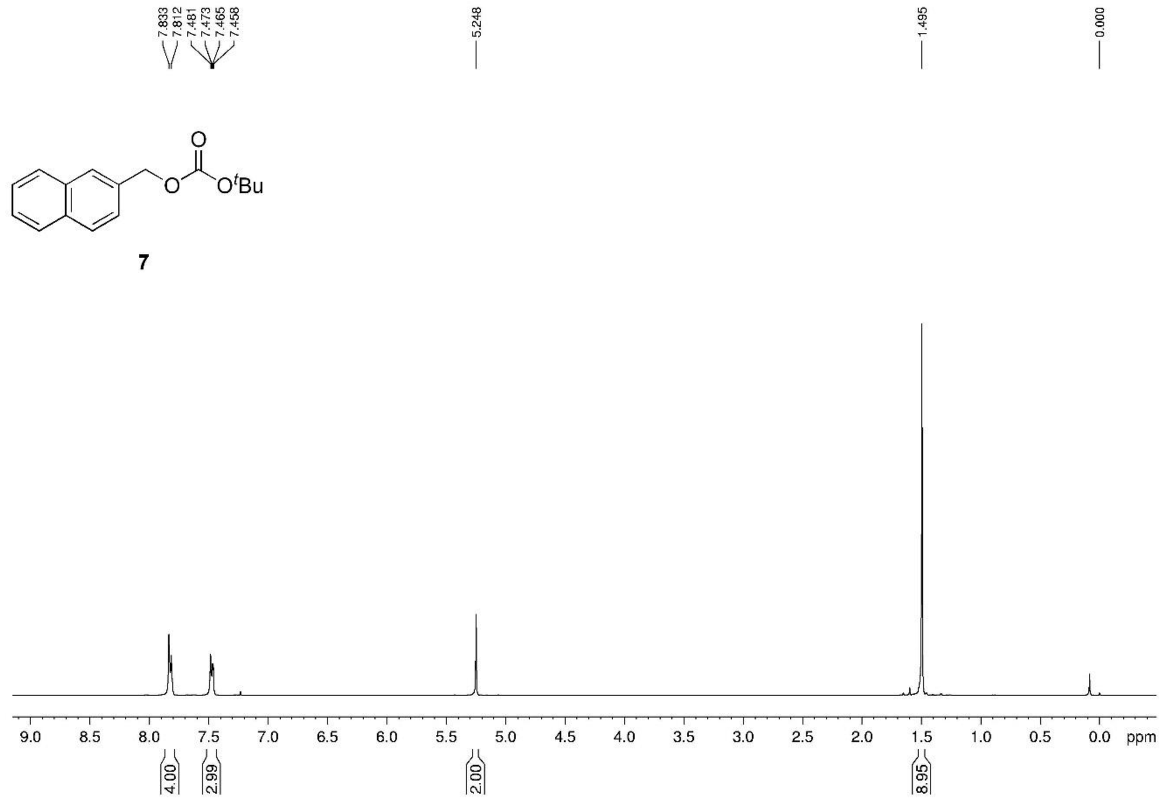
80
70
60
50
40
30
20
10
0
ppm



7

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7.481
7.473
7.465
7.458
5.248
1.495
0.000
O
O
O
tBu
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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ppm
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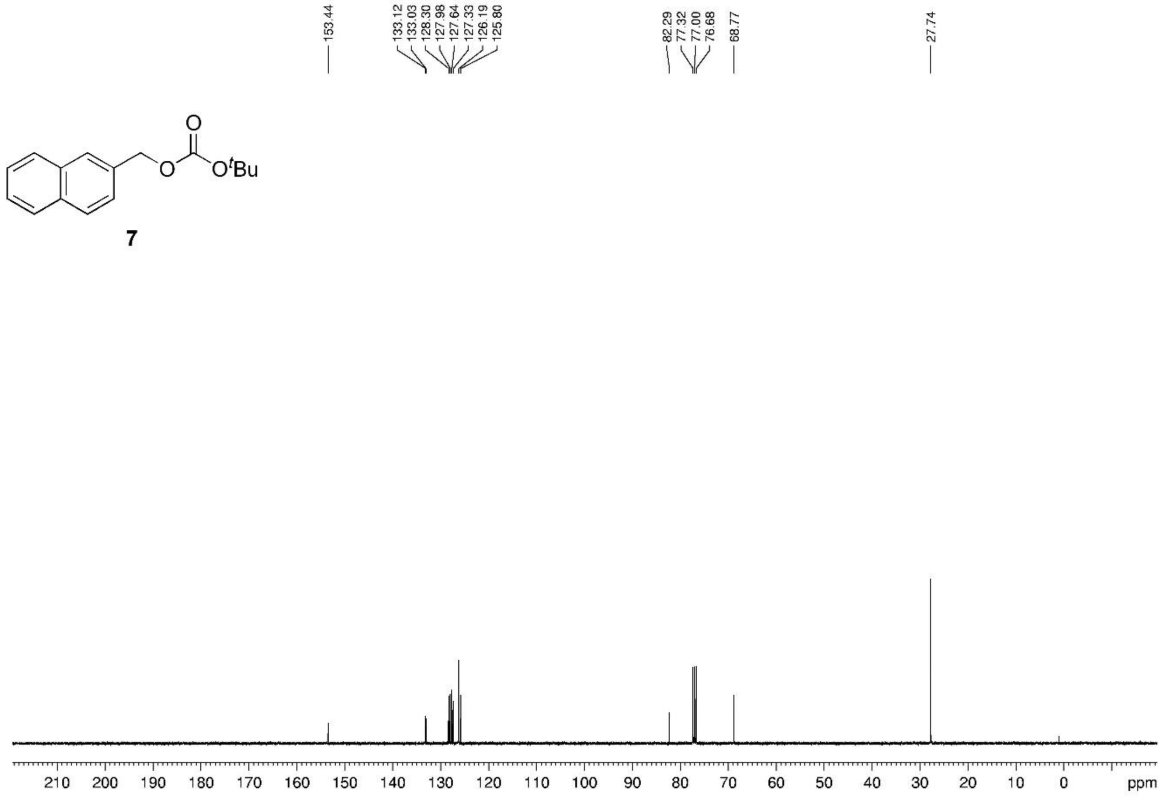
2.99
2.00
8.95



7

153.44
133.12
133.03
128.30
127.98
127.64
127.33
126.19
125.80
82.29
77.32
77.00
76.68
68.77
27.74
O
O
O
tBu
210
200
190
180
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140
130
120
110
100
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80
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20
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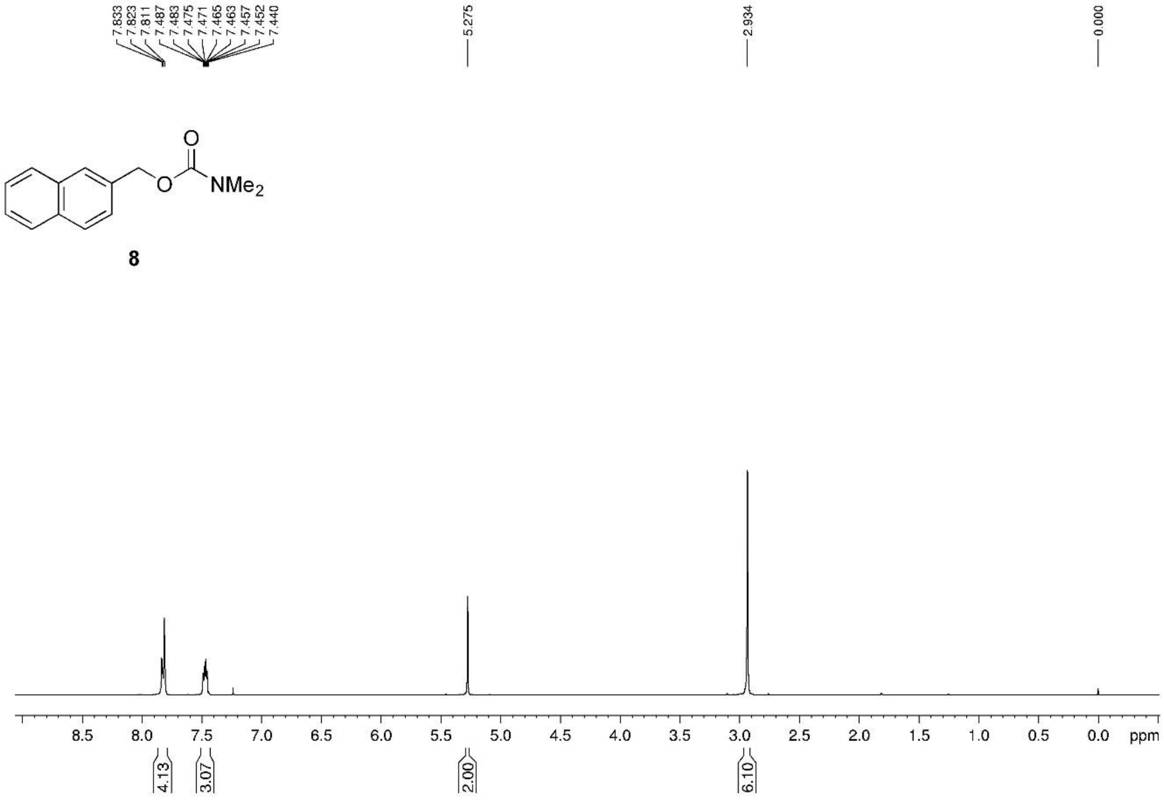
0
ppm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7.471
7.465
7.463
7.457
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7.440
5.275
2.934
0.000
O
O
NMe2
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.0
1.5
1.0
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ppm
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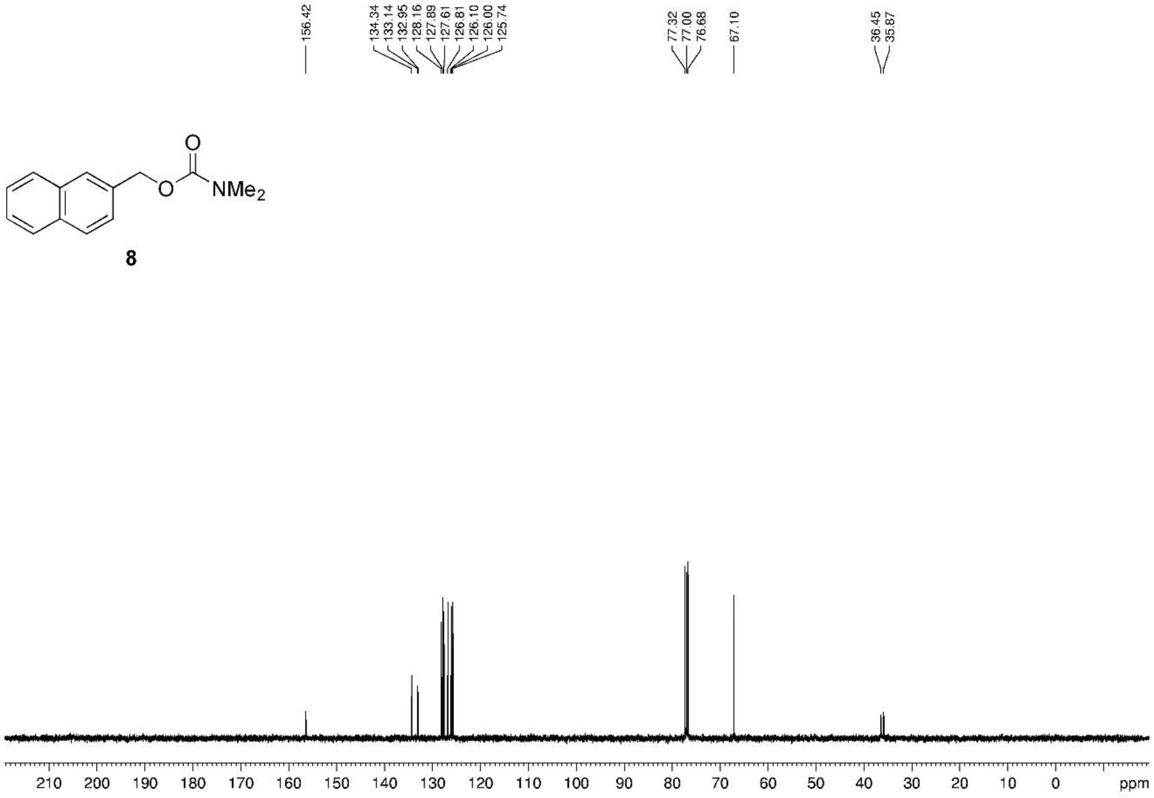
3.07
2.00
6.10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127.61
126.81
126.10
126.00
125.74
77.32
77.00
76.68
67.10
36.45
35.87
O
O
NMe2
8
210
200
190
180
170
160
150
140
130
120
110
100
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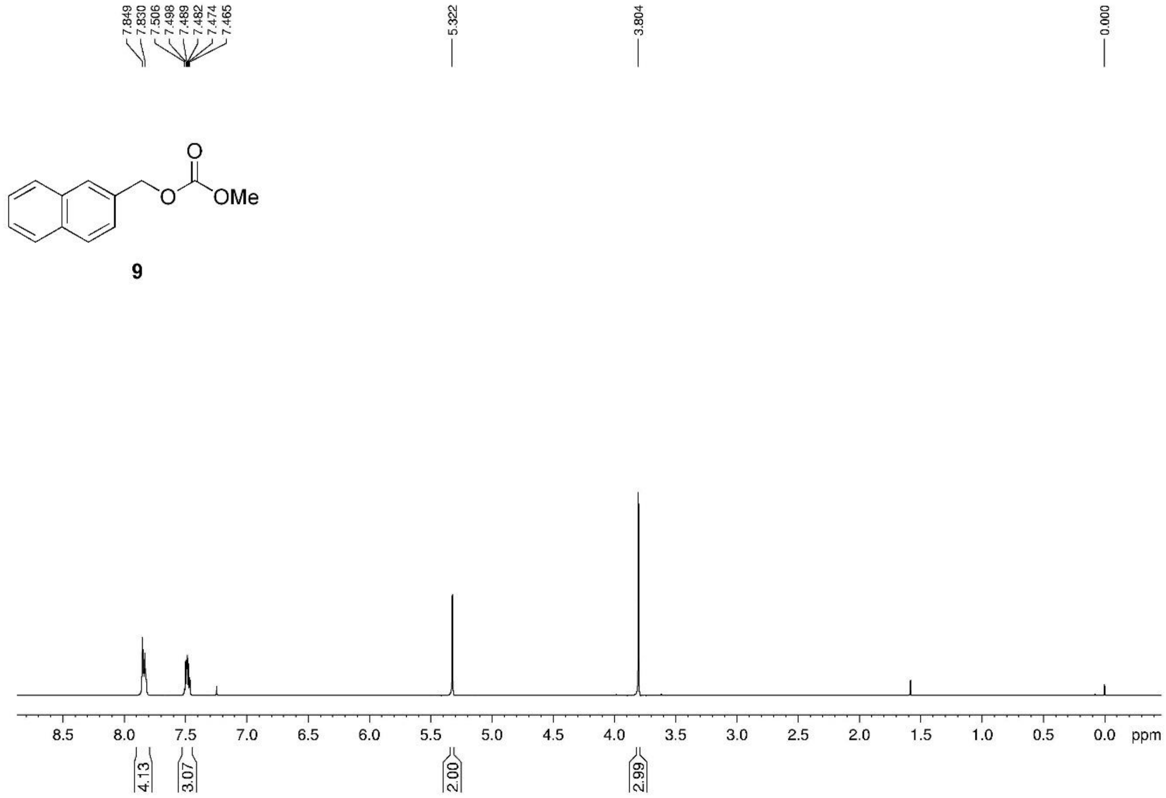
80
70
60
50
40
30
20
10
0
ppm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7.830
7.506
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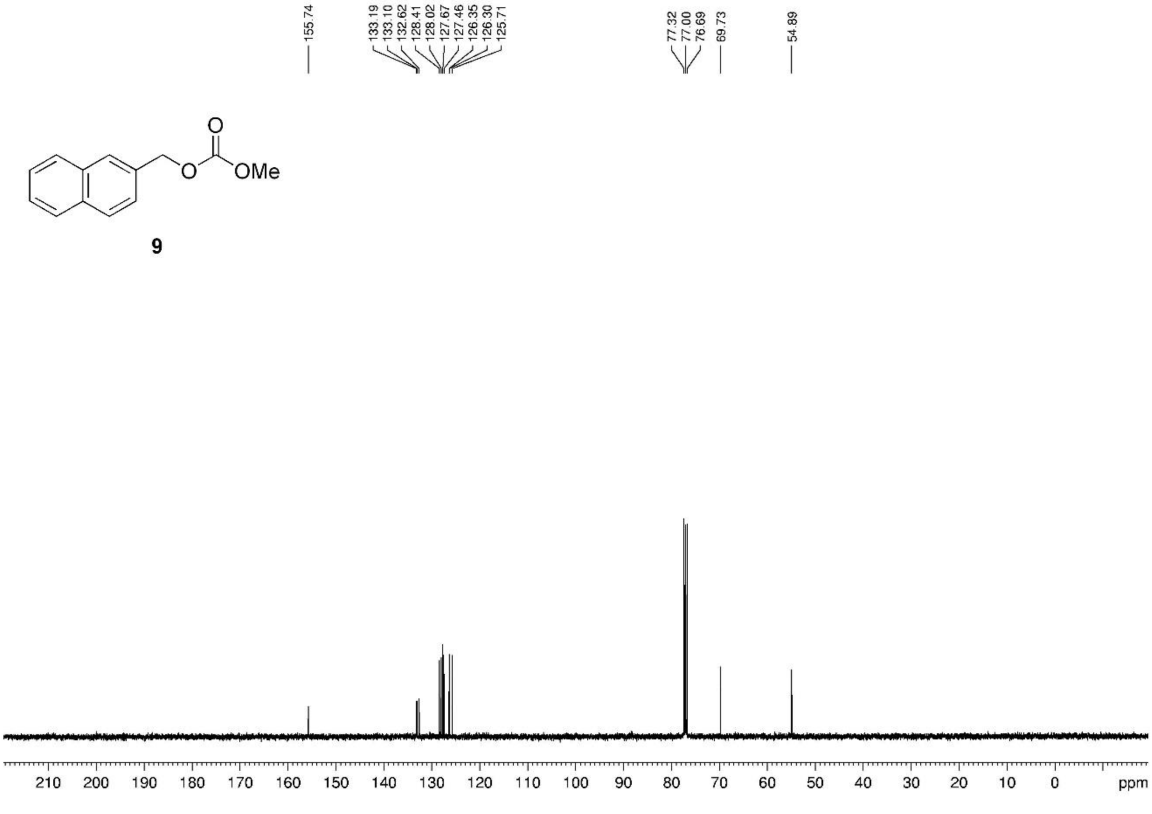
7.489
7.482
7.474
7.465
5.322
3.804
0.000
O
O
OMe
9
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.13
3.07
2.00
2.99


155.74
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133.10
132.62
128.41
128.02
127.67
127.46
126.35
126.30
125.71
77.32
77.00
76.69
69.73
54.89
O
O
OMe
9
210
200
190
180
170
160
150
140
130
120
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100
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80
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60
50
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20
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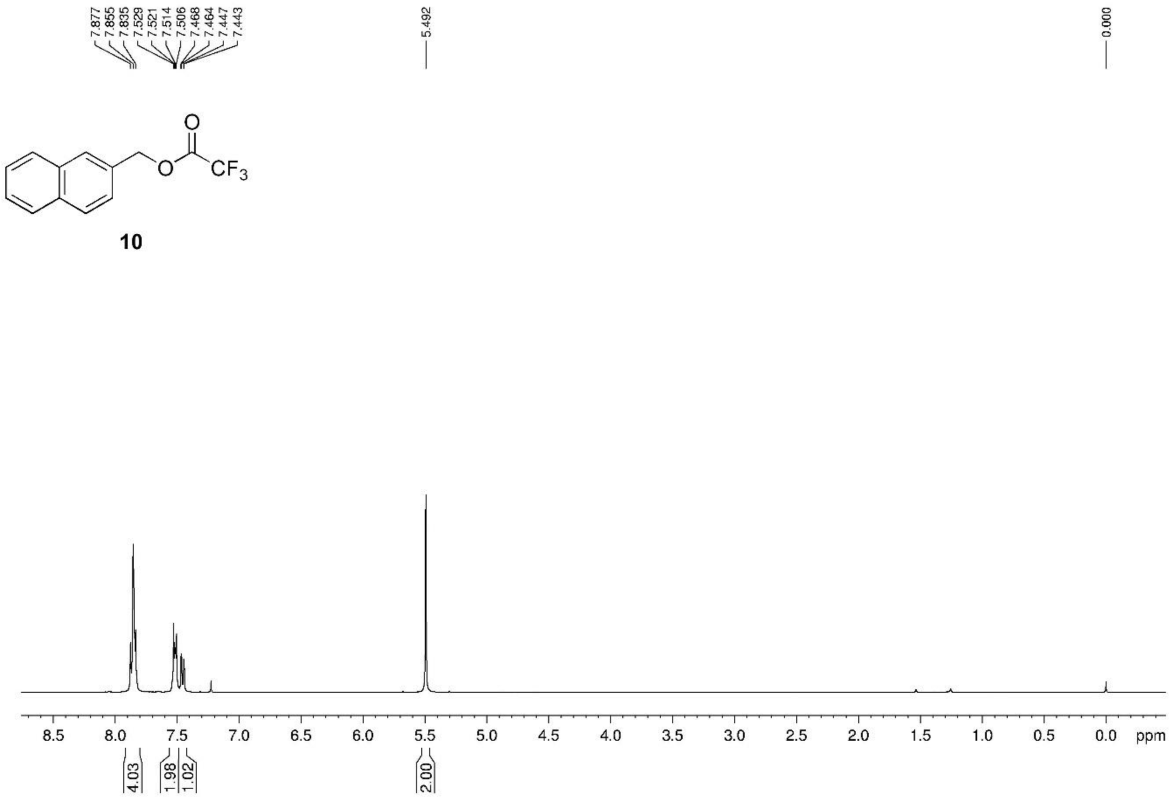
0
ppm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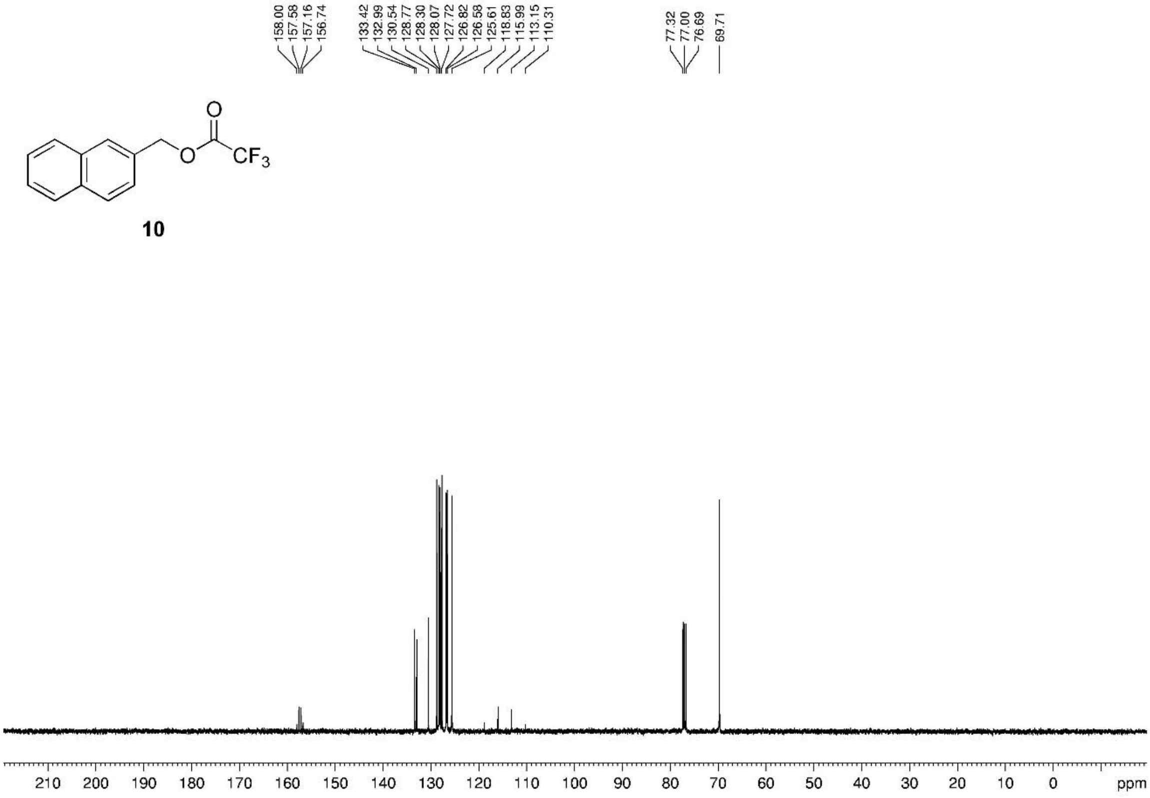
7.514
7.506
7.468
7.464
7.447
7.443
5.492
0.000
O
O
CF3
10
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.03
1.98
1.02
2.00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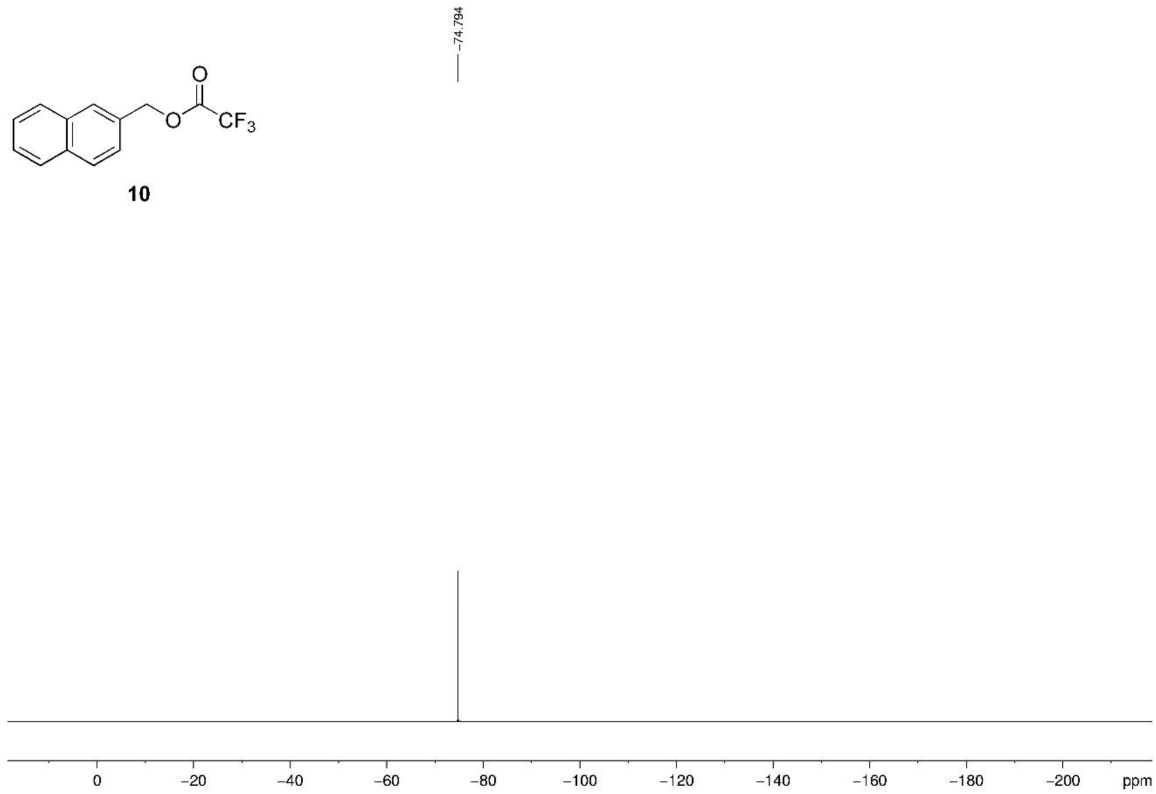
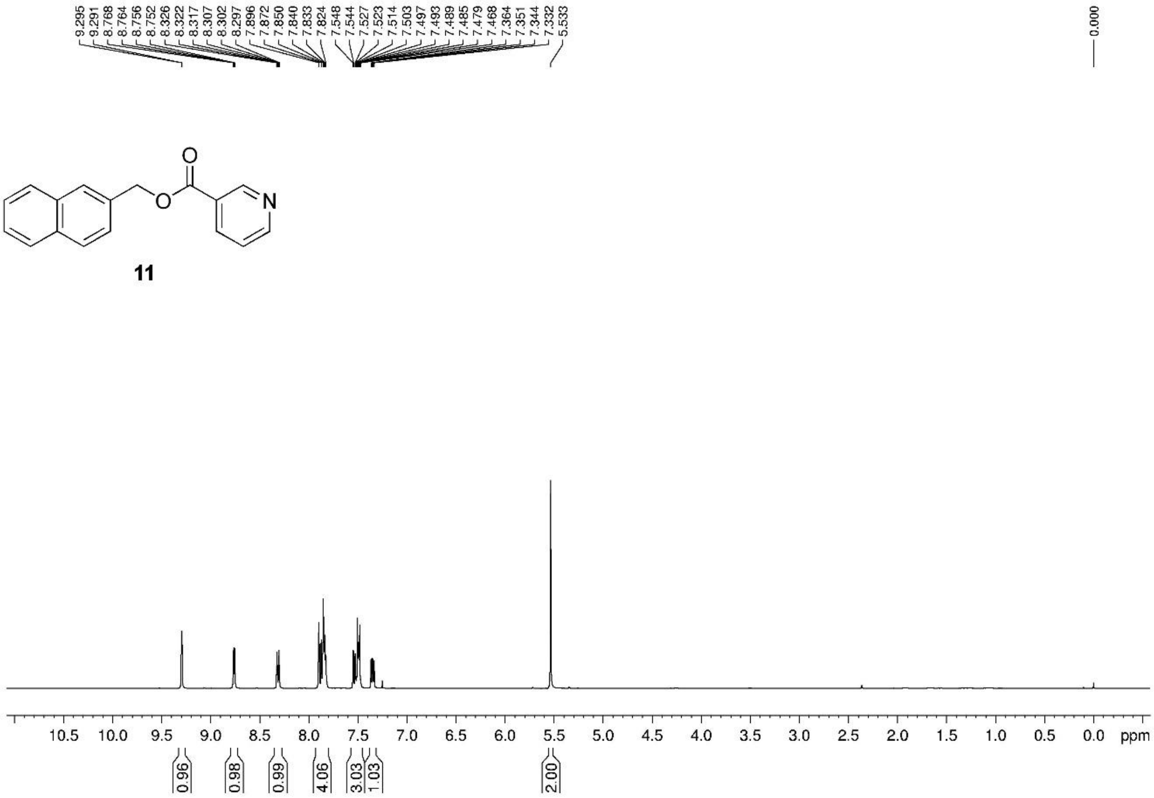
133.42
132.99
130.54
128.77
128.30
128.07
127.72
126.82
126.58
125.61
118.83
115.99
113.15
110.31
77.32
77.00
76.69
69.71
O
O
CF3
10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


–74.794
O
O
CF3
10
0
–20
–40
–60
–80
–100
–120
–140
–160
–180
–200
ppm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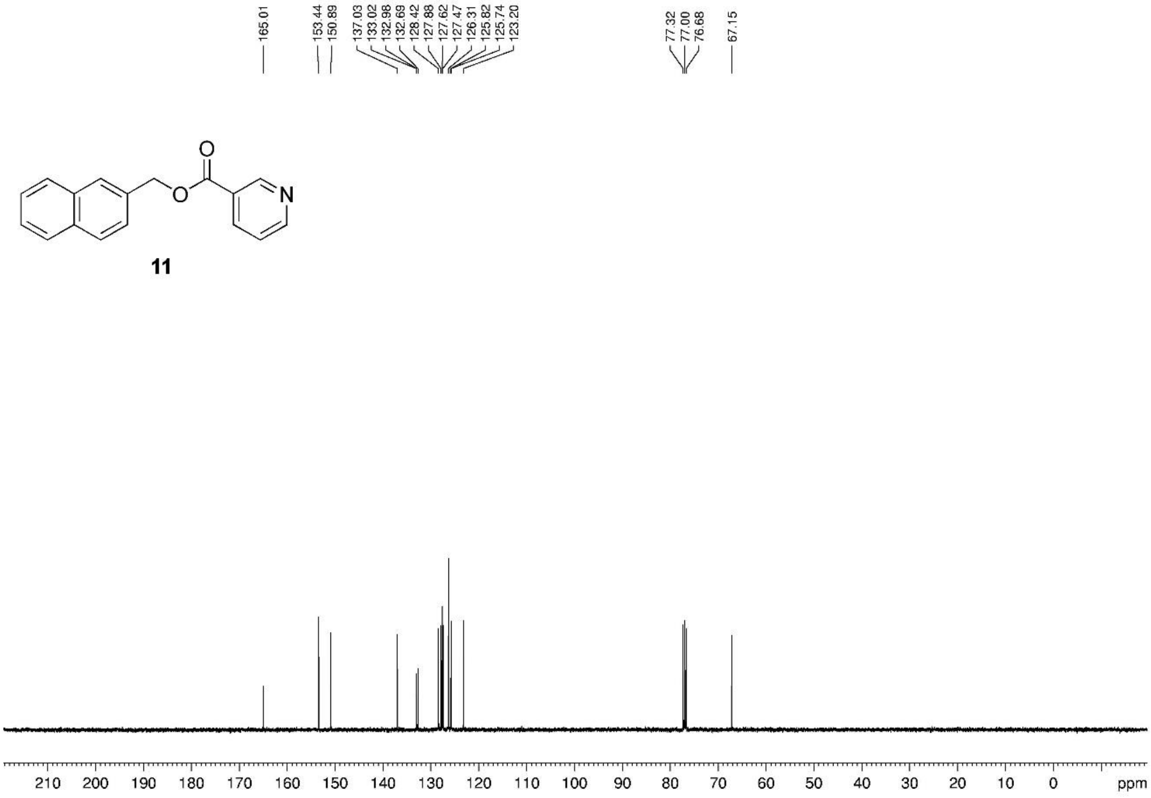
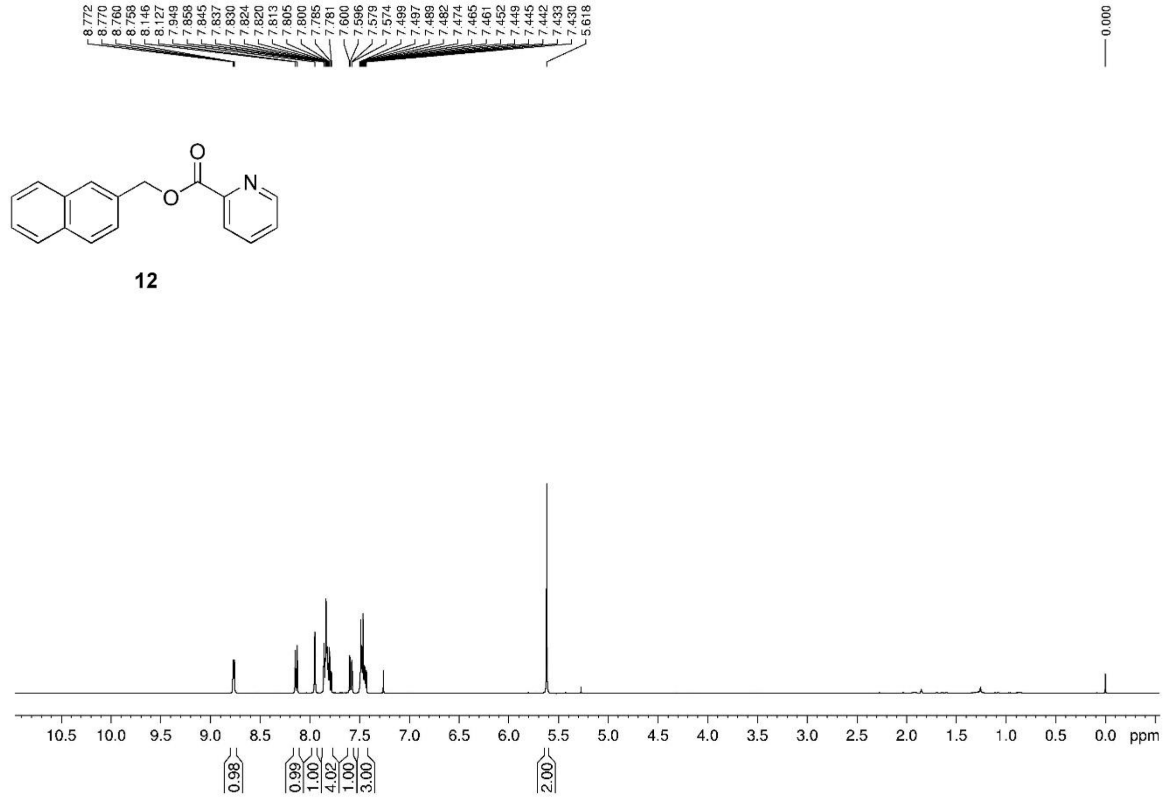
8.752
8.326
8.322
8.317
8.307
8.302
8.297
7.896
7.872
7.850
7.840
7.833
7.824
7.548
7.544
7.527
7.523
7.514
7.503
7.497
7.493
7.489
7.485
7.479
7.468
7.364
7.351
7.344
7.332
5.533
0.000
O
O
N
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.96
0.98
0.99
4.06
3.03
1.03
2.00

11


165.01
153.44
150.89
137.03
133.02
132.98
132.69
128.42
127.88
127.62
127.47
126.31
125.82
125.74
123.20
77.32
77.00
76.68
67.15
O
O
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

11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8.760
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8.146
8.127
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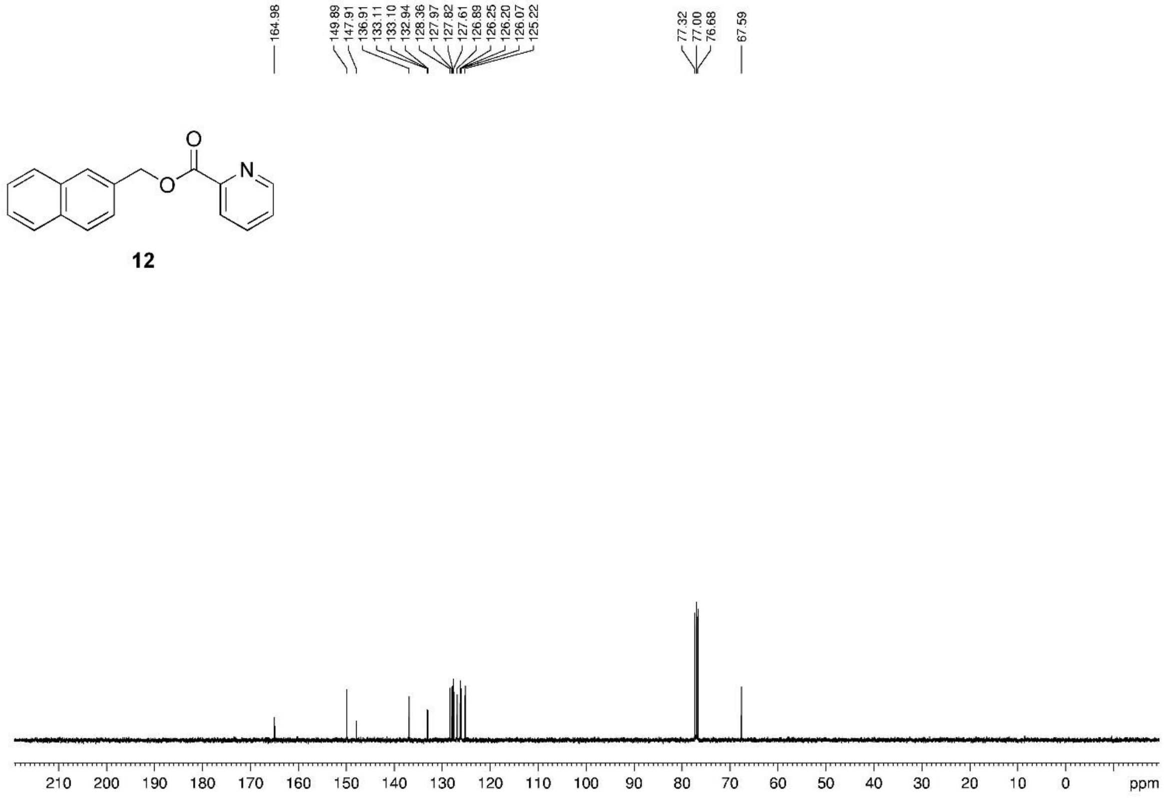
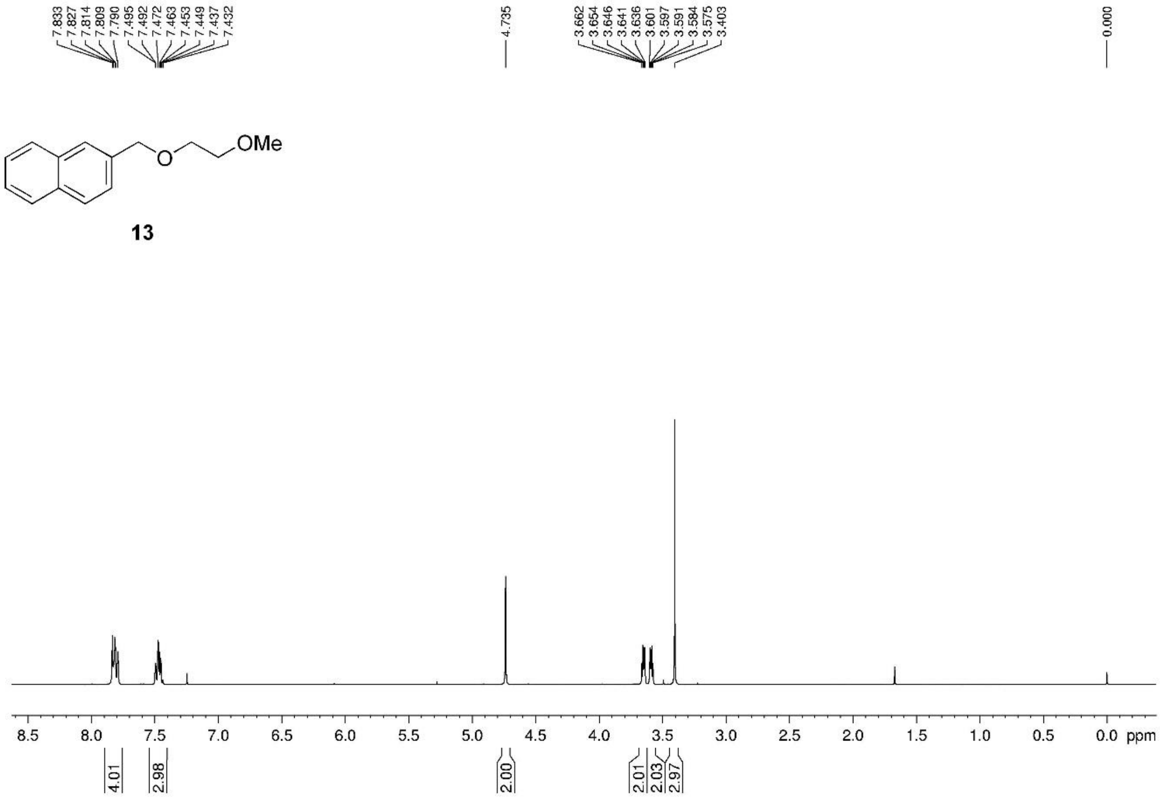
7.858
7.845
7.837
7.830
7.824
7.820
7.813
7.805
7.800
7.785
7.781
7.600
7.596
7.579
7.574
7.499
7.497
7.489
7.482
7.474
7.465
7.461
7.452
7.449
7.445
7.442
7.433
7.430
5.618
0.000
O
N
O
12
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
0.99
1.00
4.02
1.00
3.00
2.00


164.98
149.89
147.91
136.91
133.11
133.10
132.94
128.36
127.97
127.82
127.61
126.89
126.25
126.20
126.07
125.22
77.32
77.00
76.68
67.59
O
N
O
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.827
7.814
7.809
7.790
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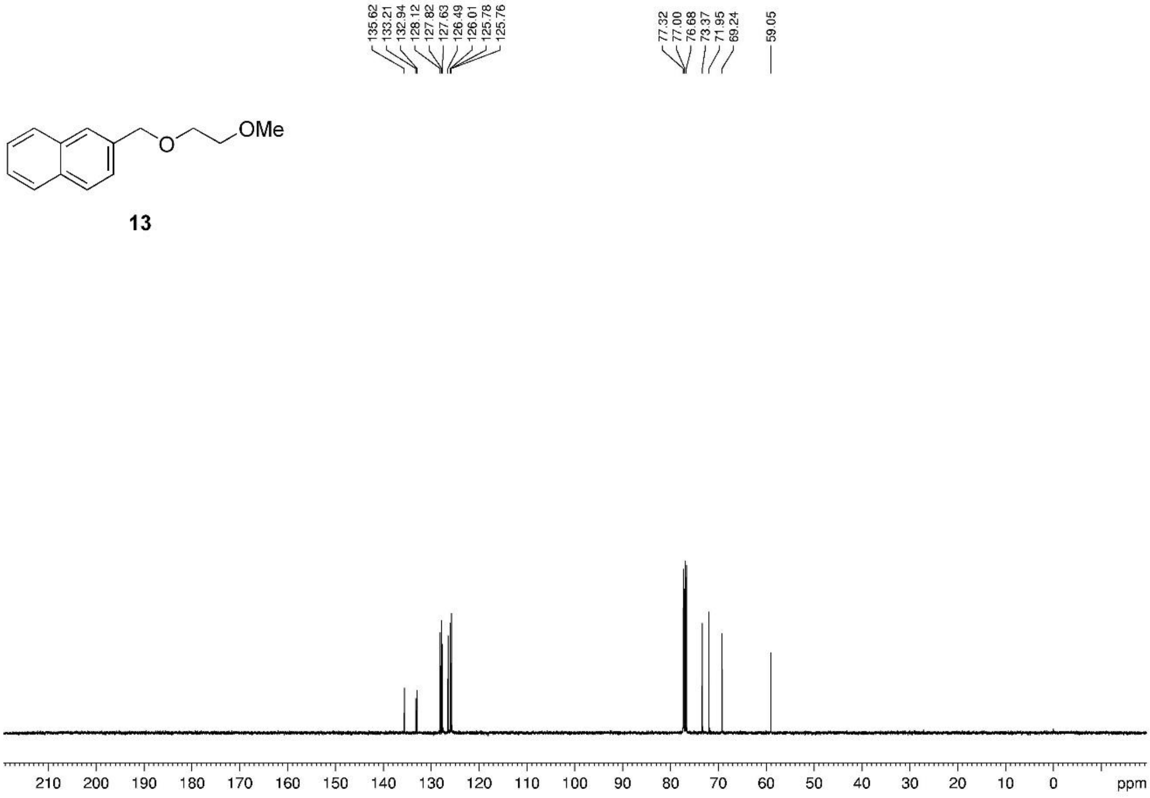
7.492
7.472
7.463
7.453
7.449
7.437
7.432
4.735
3.662
3.654
3.646
3.641
3.636
3.601
3.597
3.591
3.584
3.575
3.403
0.000
OMe
O
13
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
4.01
2.98
2.00
2.01
2.03
2.97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133.21
132.94
128.12
127.82
127.63
126.49
126.01
125.78
125.76
77.32
77.00
76.68
73.37
71.95
69.24
59.05
OMe
O
13
210
200
190
180
170
160
150
140
130
120
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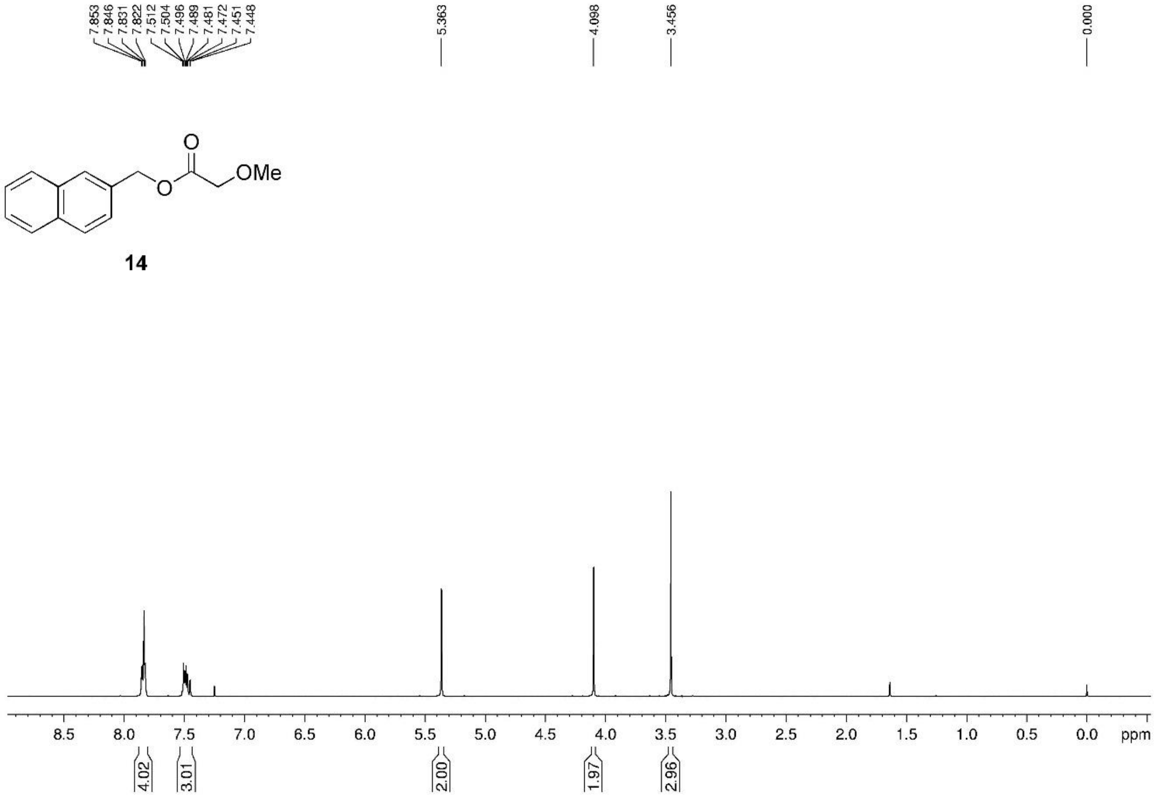
100
90
80
70
60
50
40
30
20
10
0
ppm



14

7.853
7.846
7.831
7.822
7.512
7.504
7.496
7.489
7.481
7.472
7.451
7.448
5.363
4.098
3.456
0.000
O
OMe
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.02
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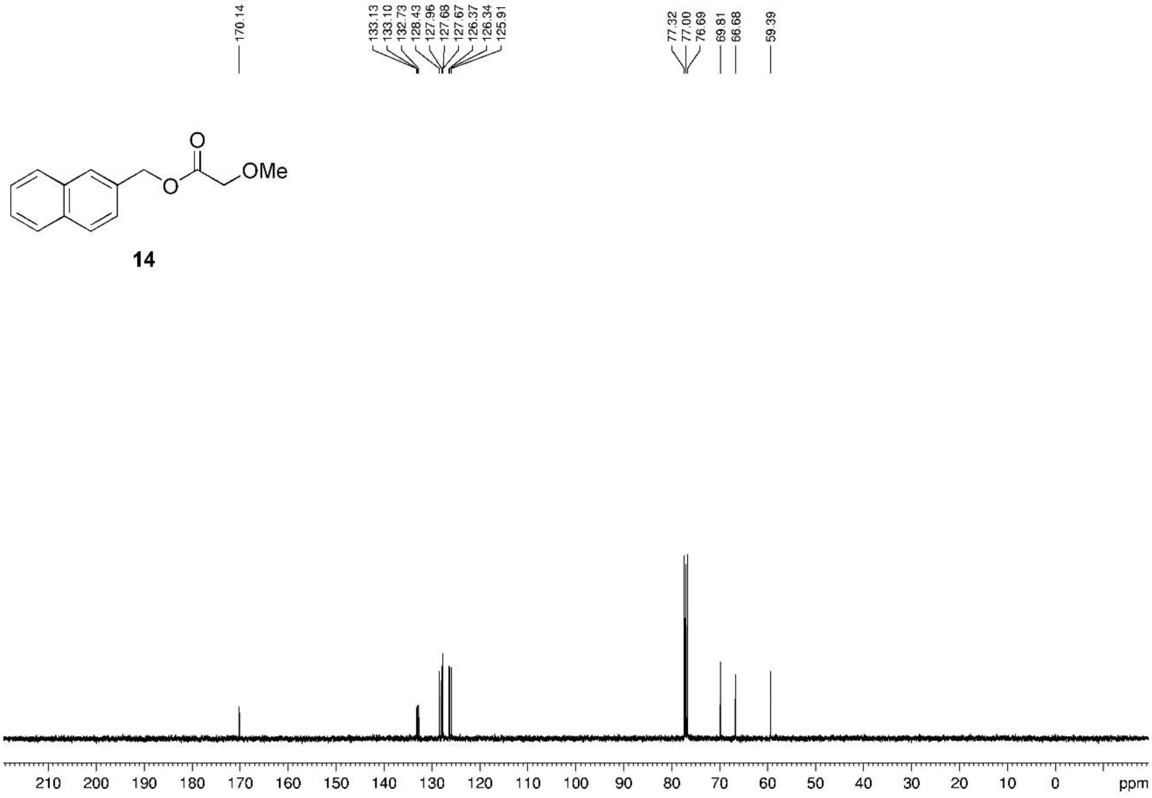
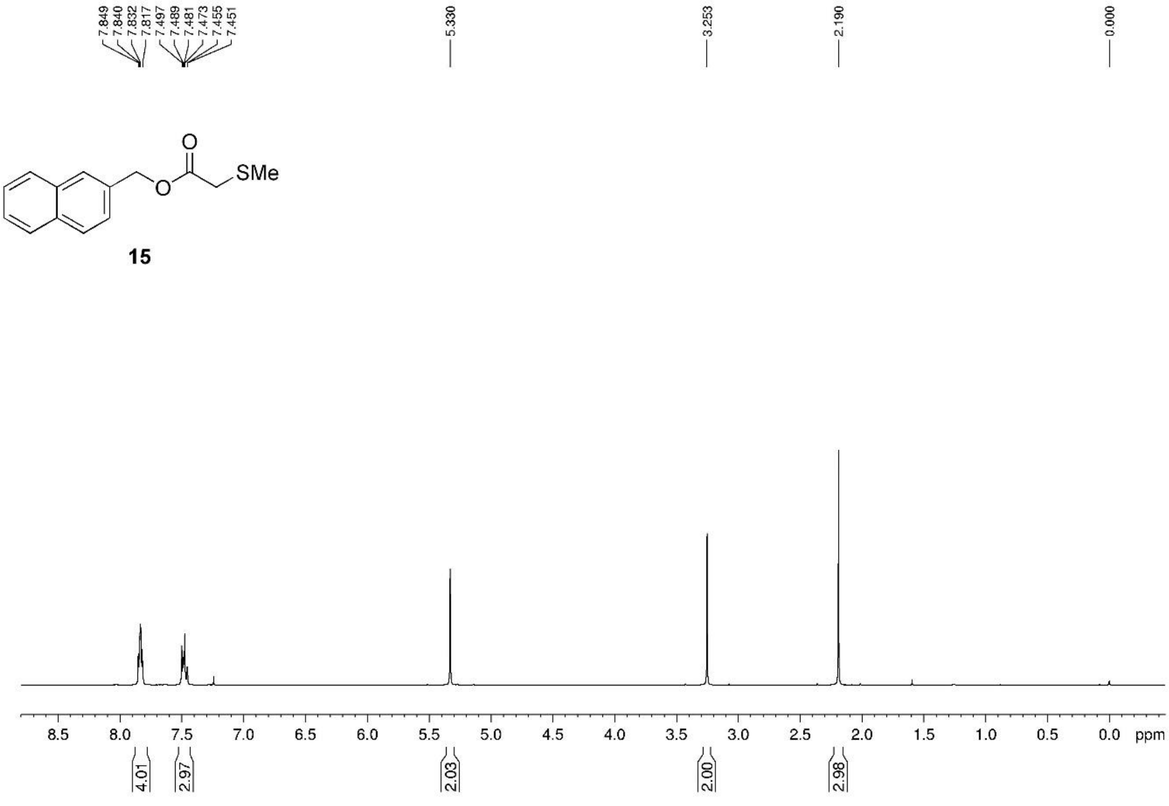
2.00
1.97
2.96



14

170.14
133.13
133.10
132.73
128.43
127.96
127.68
127.67
126.37
126.34
125.91
77.32
77.00
76.69
69.81
66.68
59.39
O
OMe
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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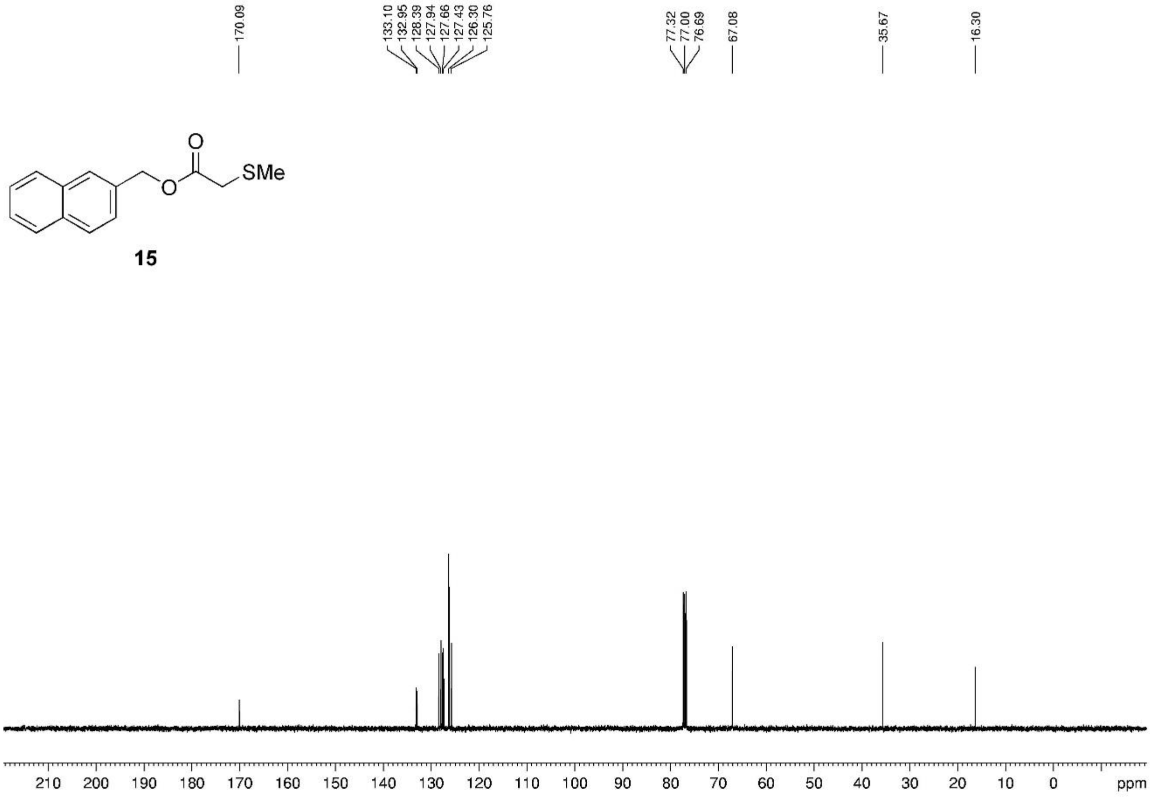
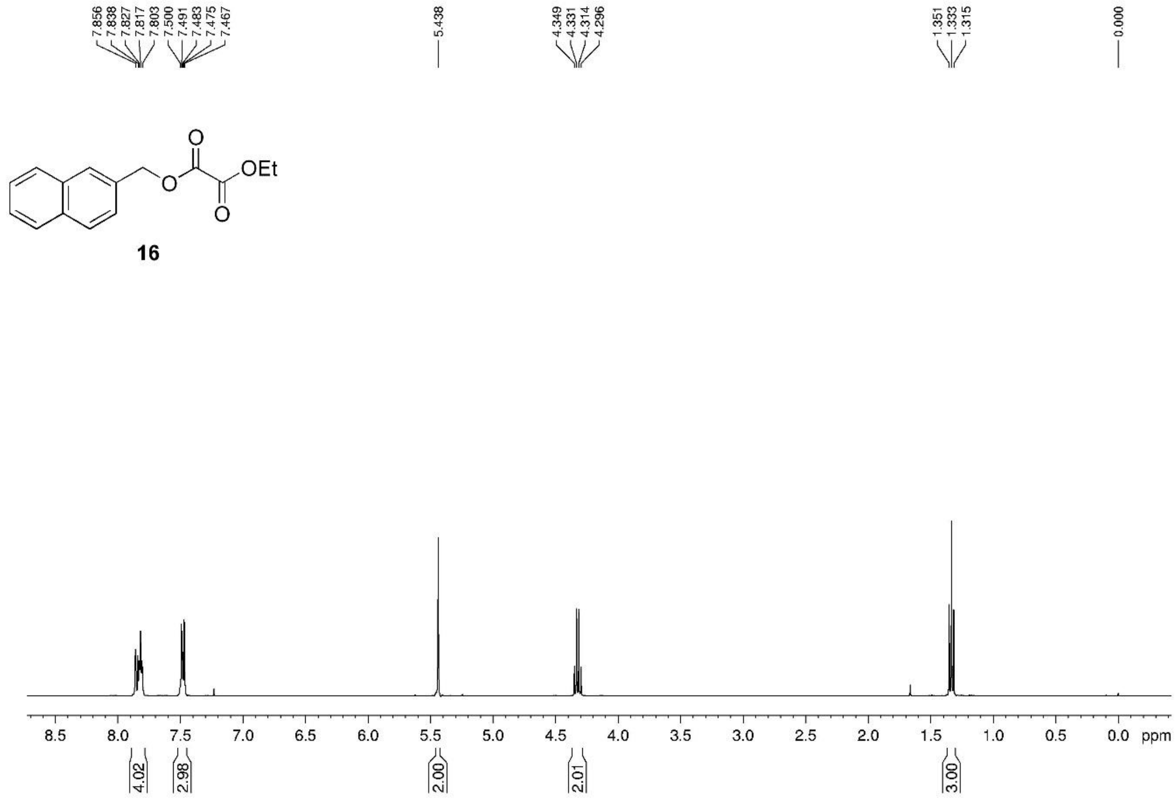
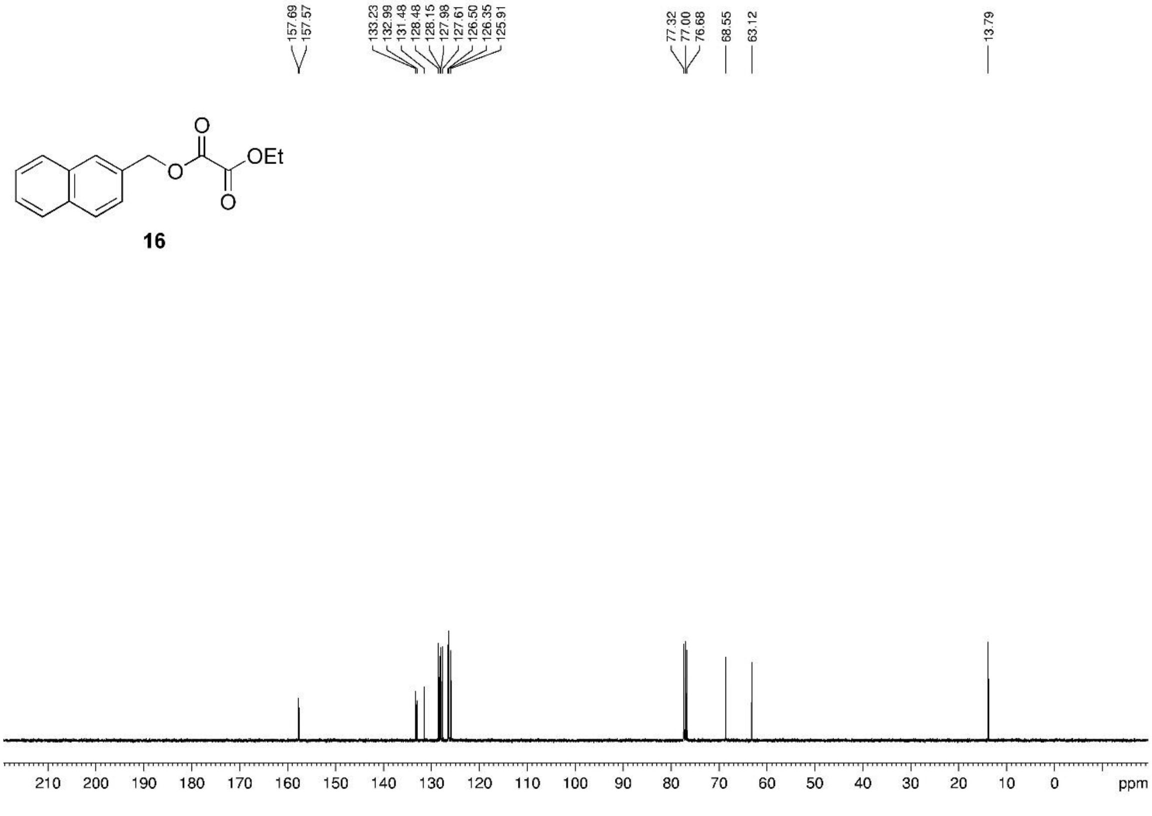
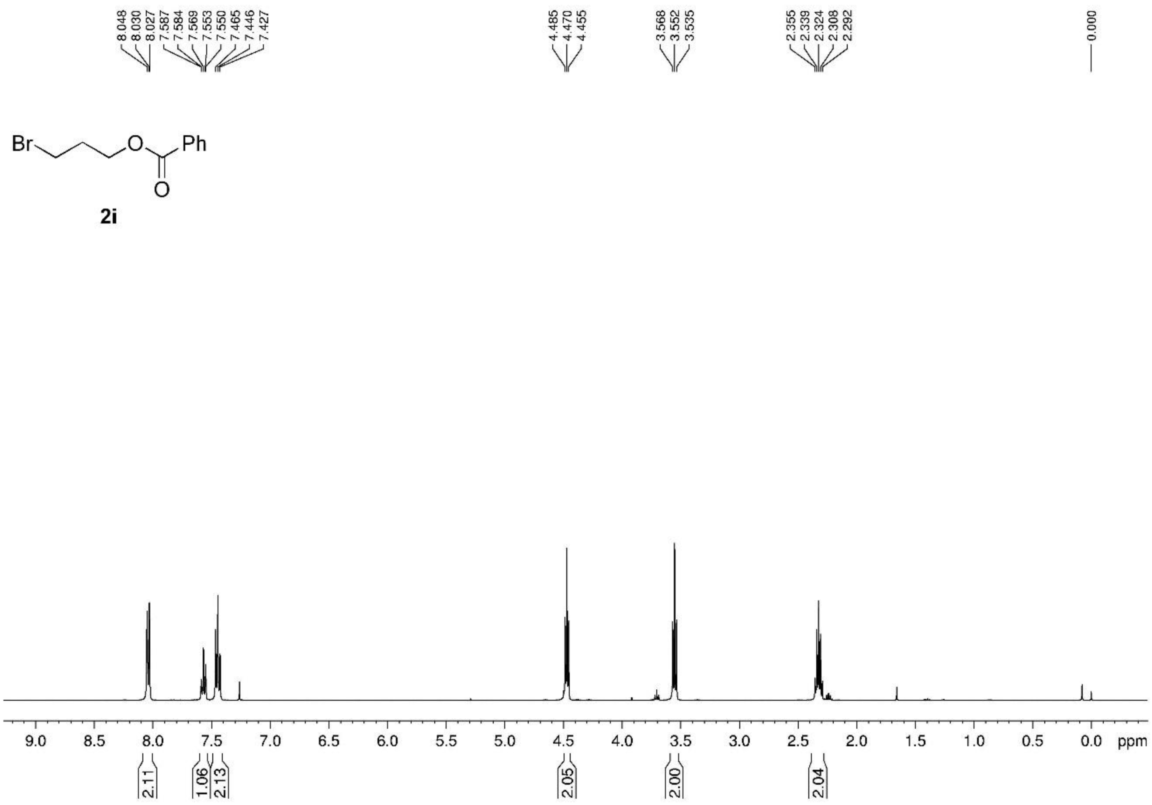
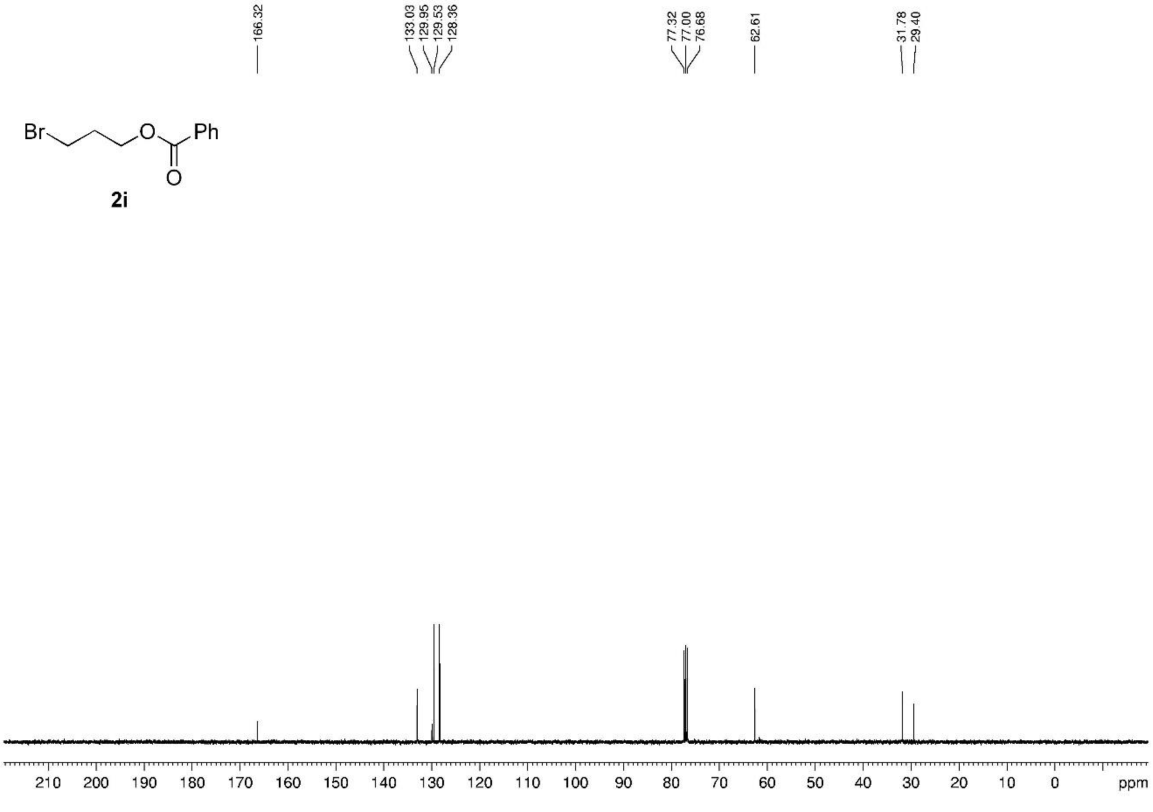
7.840
7.832
7.817
7.497
7.489
7.481
7.473
7.455
7.451
5.330
3.253
2.190
0.000
O
O
SMe
15
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.01
2.97
2.03
2.00
2.98

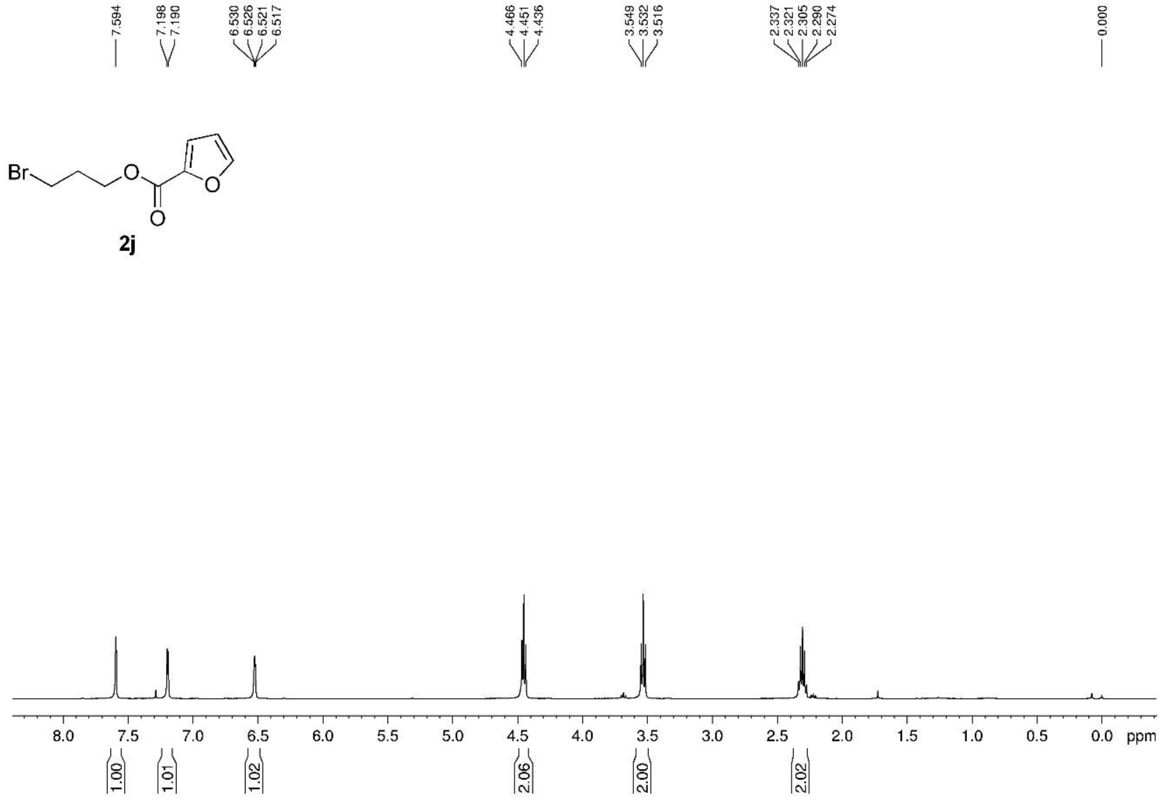

170.09
133.10
132.95
128.39
127.94
127.66
127.43
126.30
125.76
77.32
77.00
76.69
67.08
35.67
16.30
O
O
SMe
15
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.856
7.838
7.827
7.817
7.803
7.500
7.491
7.483
7.475
7.467
5.438
4.349
4.331
4.314
4.296
1.351
1.333
1.315
0.000
O
OEt
O
O
16
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.02
2.98
2.00
2.01
3.00

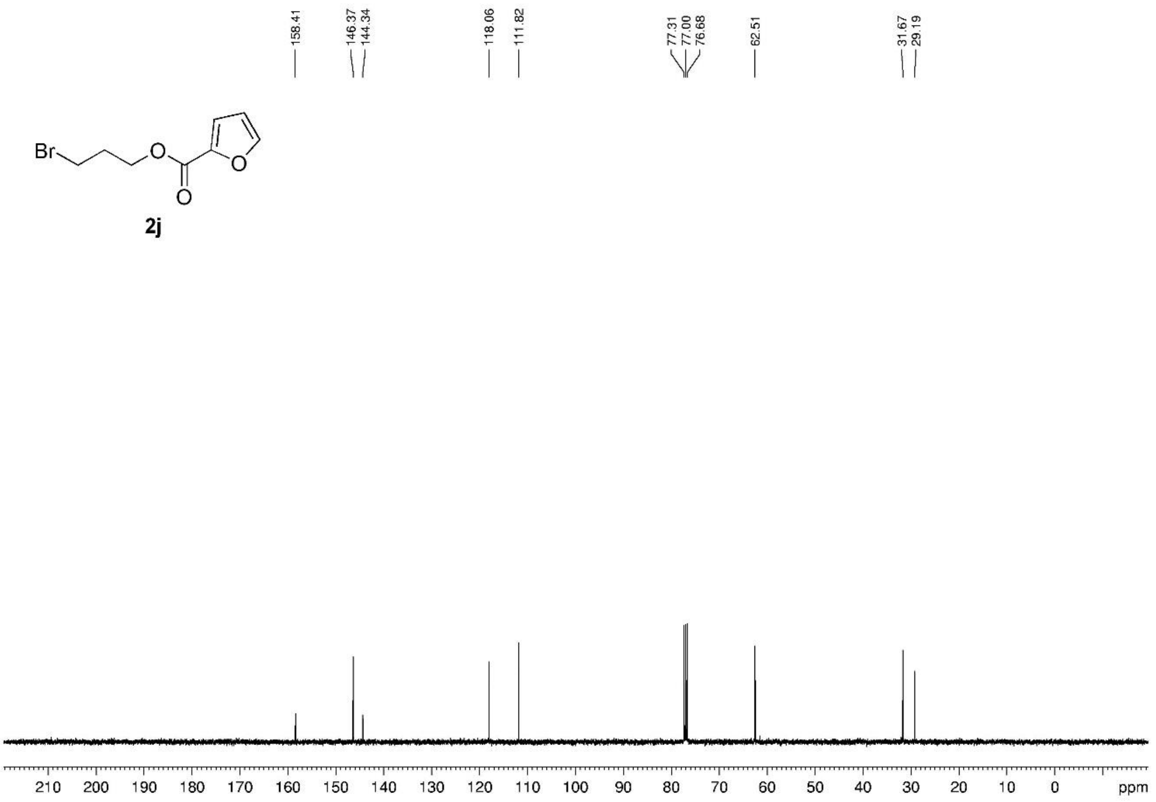

157.69
157.57
133.23
132.99
131.48
128.48
128.15
127.98
127.61
126.50
126.35
125.91
77.32
77.00
76.68
68.55
63.12
13.79
O
OEt
O
O
16
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.048
8.030
8.027
7.587
7.584
7.569
7.553
7.550
7.465
7.446
7.427
4.485
4.470
4.455
3.568
3.552
3.535
2.355
2.339
2.324
2.308
2.292
0.000
Br
O
Ph
O
2i
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.11
1.06
2.13
2.05
2.00
2.04

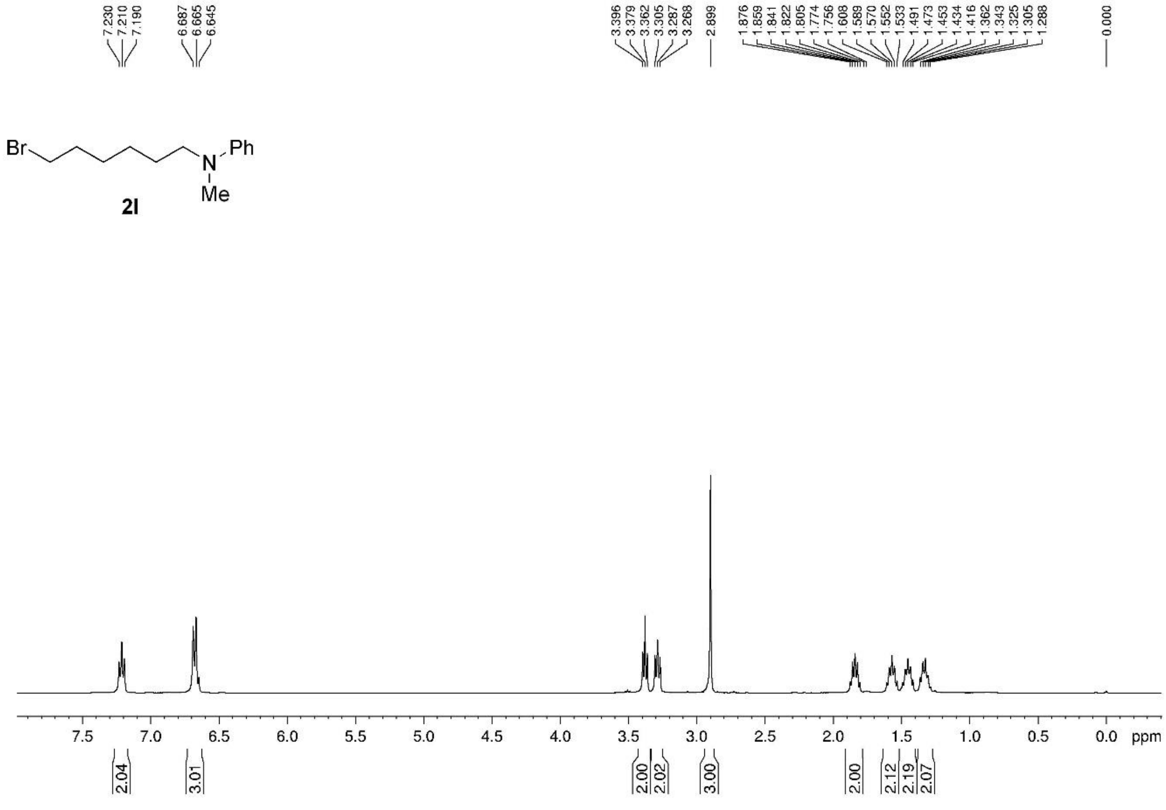

166.32
133.03
129.95
129.53
128.36
77.32
77.00
76.68
62.61
31.78
29.40
Br
O
Ph
O
2i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

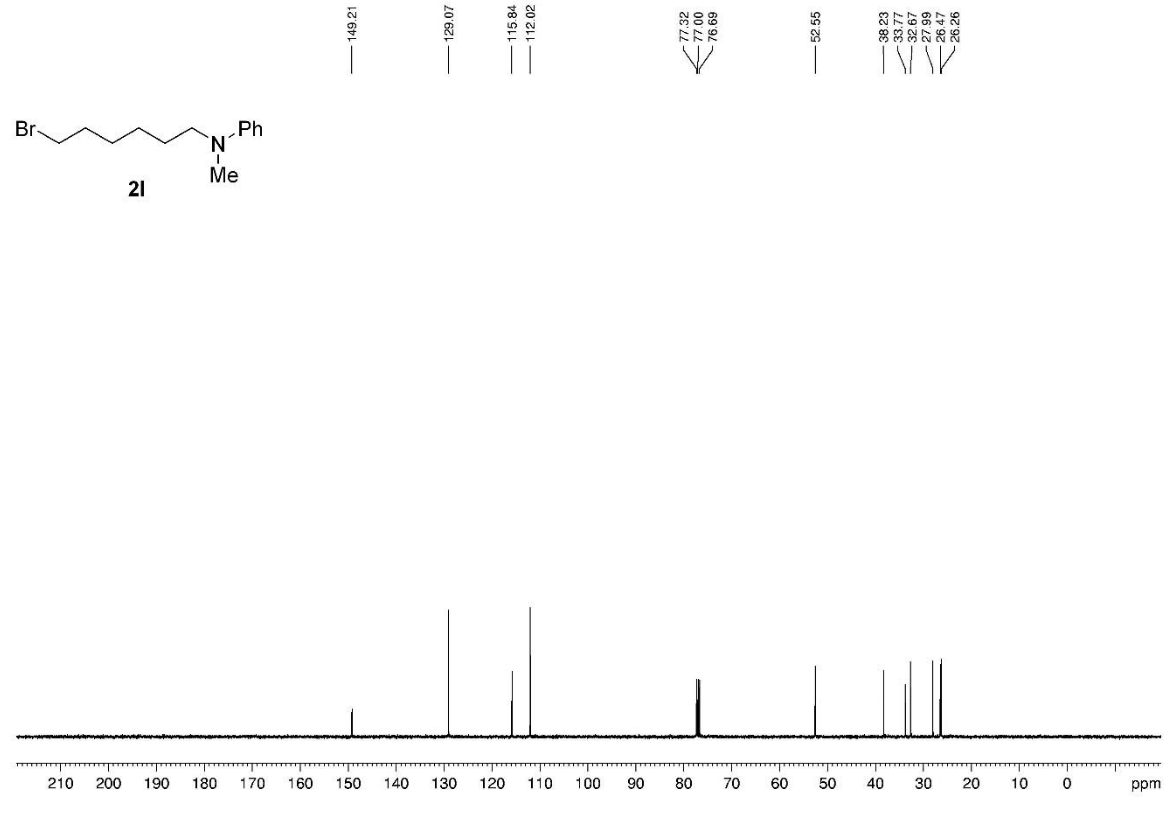

7.594
7.198
7.190
6.530
6.526
6.521
6.517
4.466
4.451
4.436
3.549
3.532
3.516
2.337
2.321
2.305
2.290
2.274
0.000
Br
O
O
O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
1.02
2.06
2.00
2.02

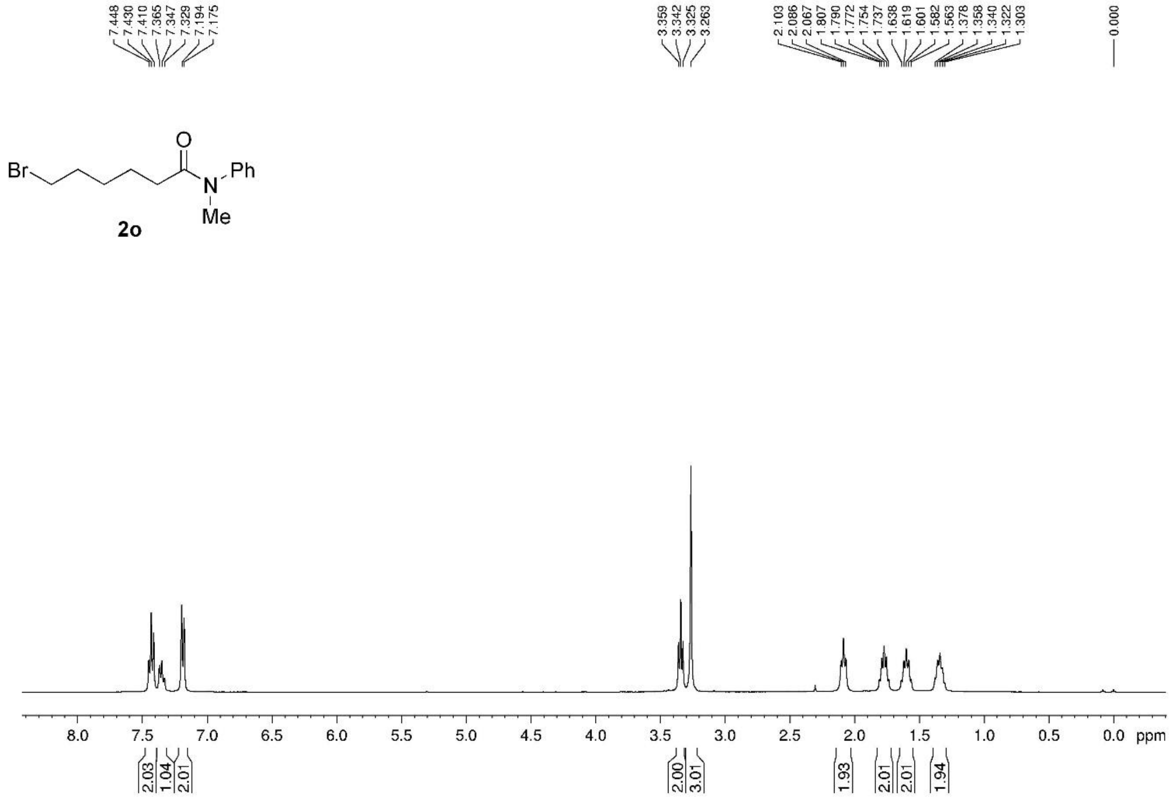
2j

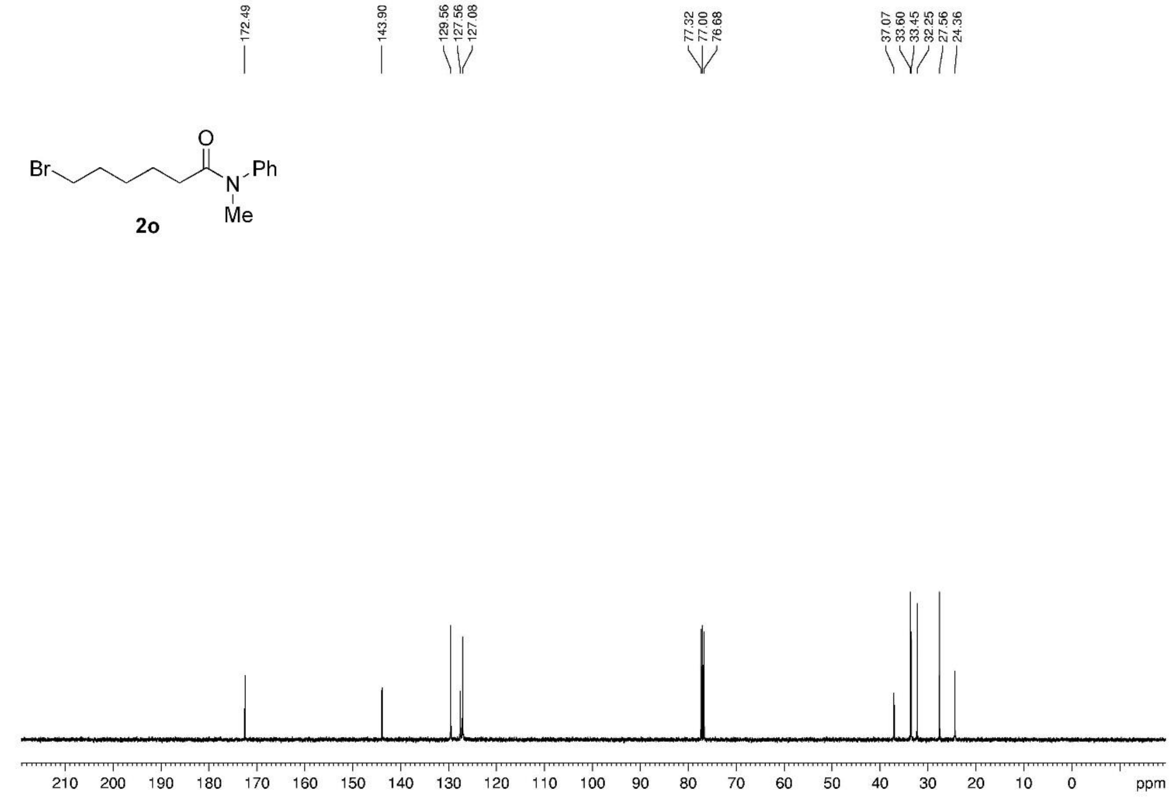

158.41
146.37
144.34
118.06
111.82
77.31
77.00
76.68
62.51
31.67
29.19
Br
O
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

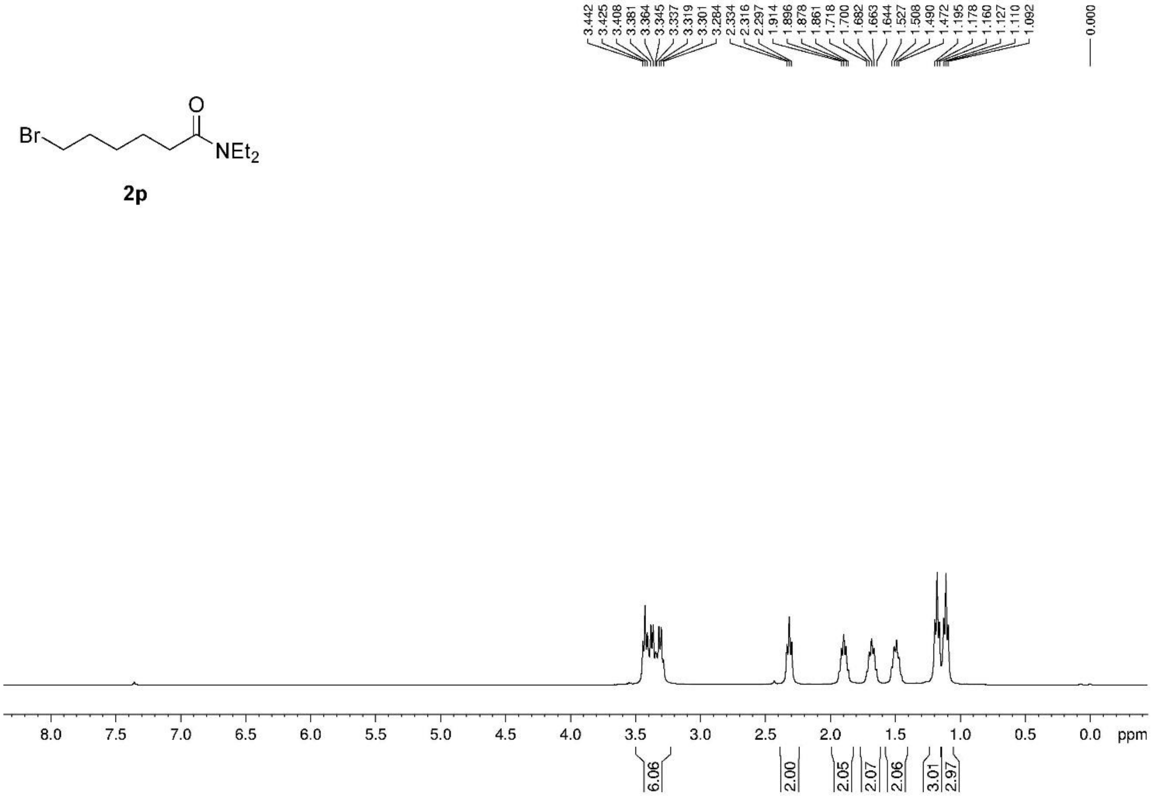
2j


7.230
7.210
7.190
6.687
6.665
6.645
3.396
3.379
3.362
3.305
3.287
3.268
2.899
1.876
1.859
1.841
1.822
1.805
1.774
1.756
1.608
1.589
1.570
1.552
1.533
1.491
1.473
1.453
1.434
1.416
1.362
1.343
1.325
1.305
1.288
0.000
Br
N
Ph
Me
2l
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.04
3.01
2.00
2.02
3.00
2.00
2.12
2.19
2.07

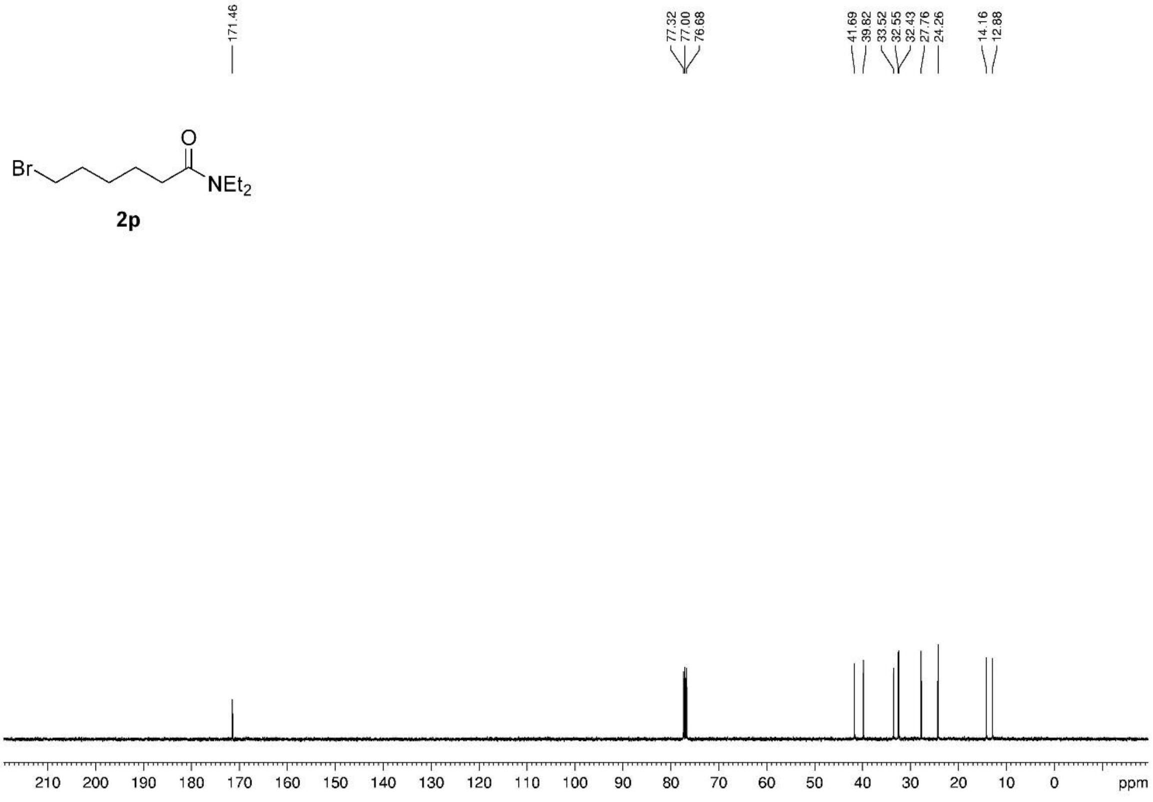

149.21
129.07
115.84
112.02
77.32
77.00
76.69
52.55
38.23
33.77
32.67
27.99
26.47
26.26
Br
N
Ph
Me
2l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.448
7.430
7.410
7.365
7.347
7.329
7.194
7.175
3.359
3.342
3.325
3.263
2.103
2.086
2.067
1.807
1.790
1.772
1.754
1.737
1.638
1.619
1.601
1.582
1.563
1.378
1.358
1.340
1.322
1.303
0.000
Br
O
N
Ph
Me
2o
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.03
1.04
2.01
2.00
3.01
1.93
2.01
2.01
1.94

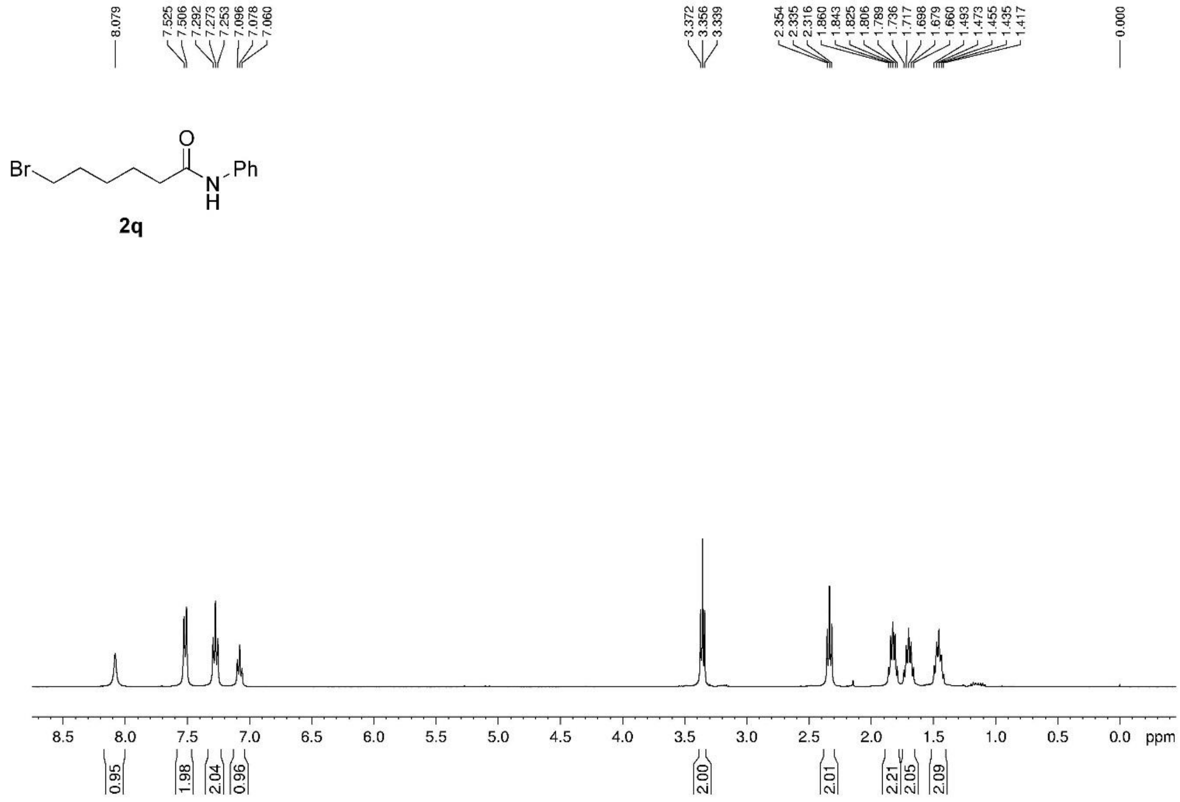

172.49
143.90
129.56
127.56
127.08
77.32
77.00
76.68
37.07
33.60
33.45
32.25
27.56
24.36
Br
O
N
Ph
Me
2o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



2p
3.442
3.425
3.408
3.381
3.364
3.345
3.337
3.319
3.301
3.284
2.334
2.316
2.297
1.914
1.896
1.878
1.861
1.718
1.700
1.682
1.663
1.644
1.527
1.508
1.490
1.472
1.195
1.178
1.160
1.127
1.110
1.092
0.000
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
6.06
2.00
2.05
2.07
2.06
3.01
2.97



2p
171.46
77.32
77.00
76.68
41.69
39.82
33.52
32.55
32.43
27.76
24.26
14.16
12.88
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


2q
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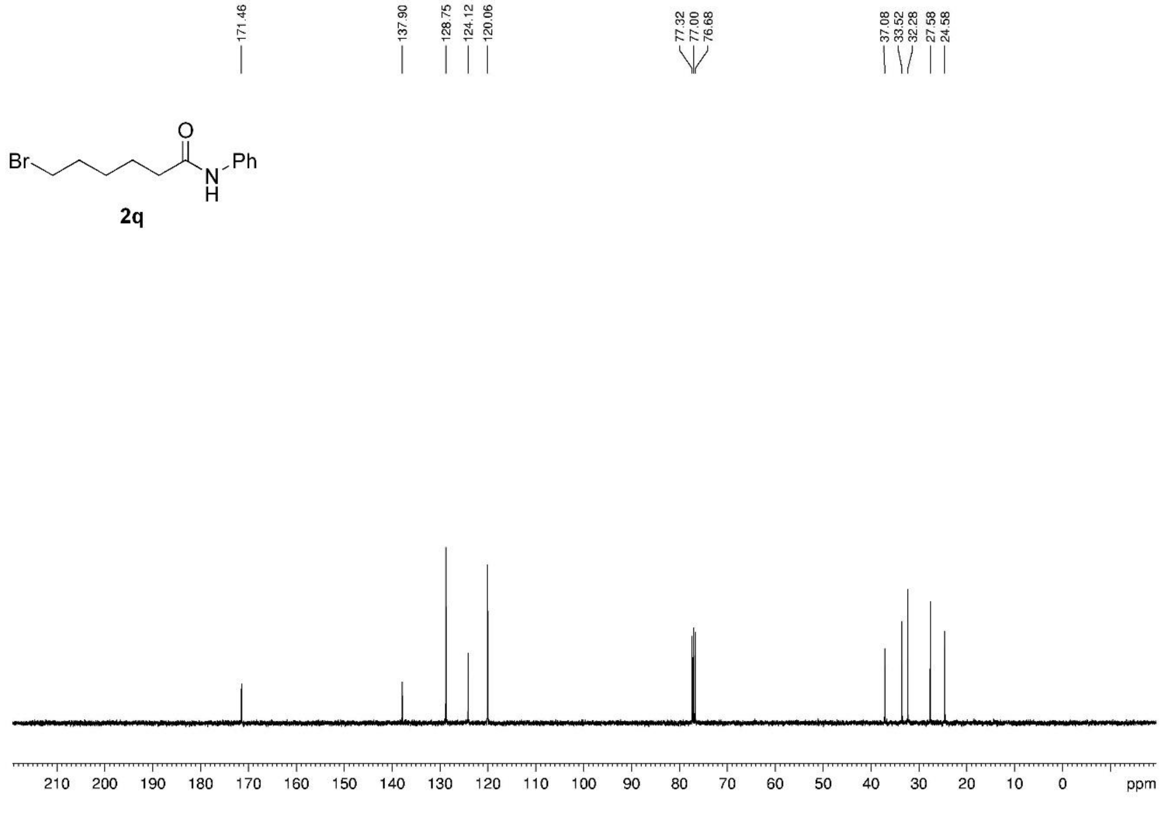
7.525
7.506
7.292
7.273
7.253
7.096
7.078
7.060
3.372
3.356
3.339
2.354
2.335
2.316
1.860
1.843
1.825
1.806
1.789
1.736
1.717
1.698
1.679
1.660
1.493
1.473
1.455
1.435
1.417
0.000
Br
O
N
H
Ph
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.95
1.98
2.04
0.96
2.00
2.01
2.21
2.05
2.09


2q
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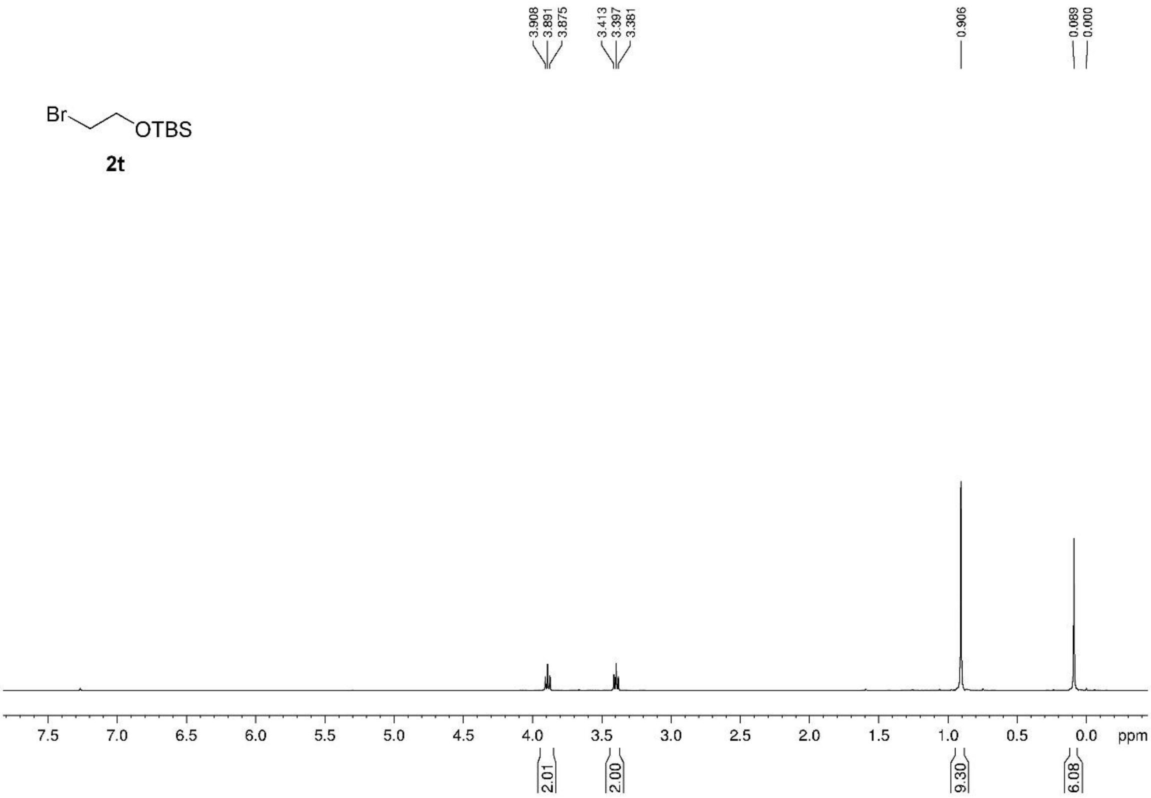
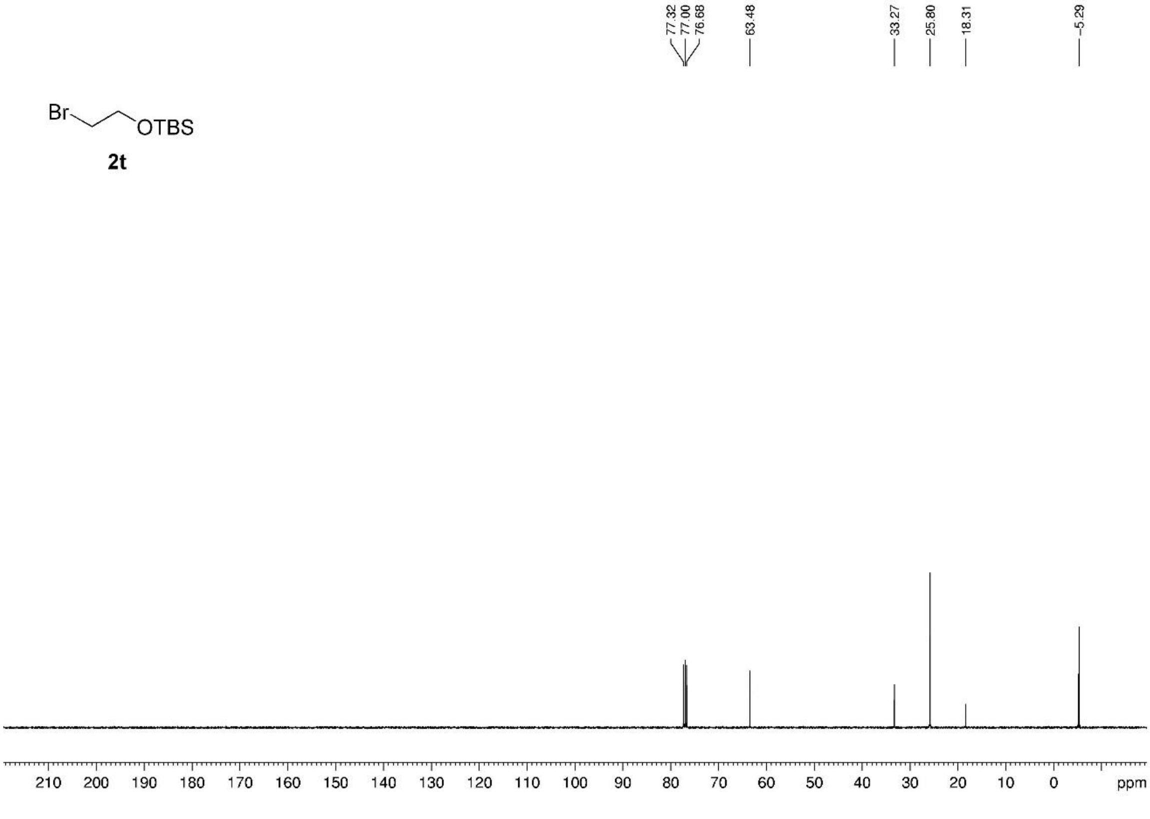
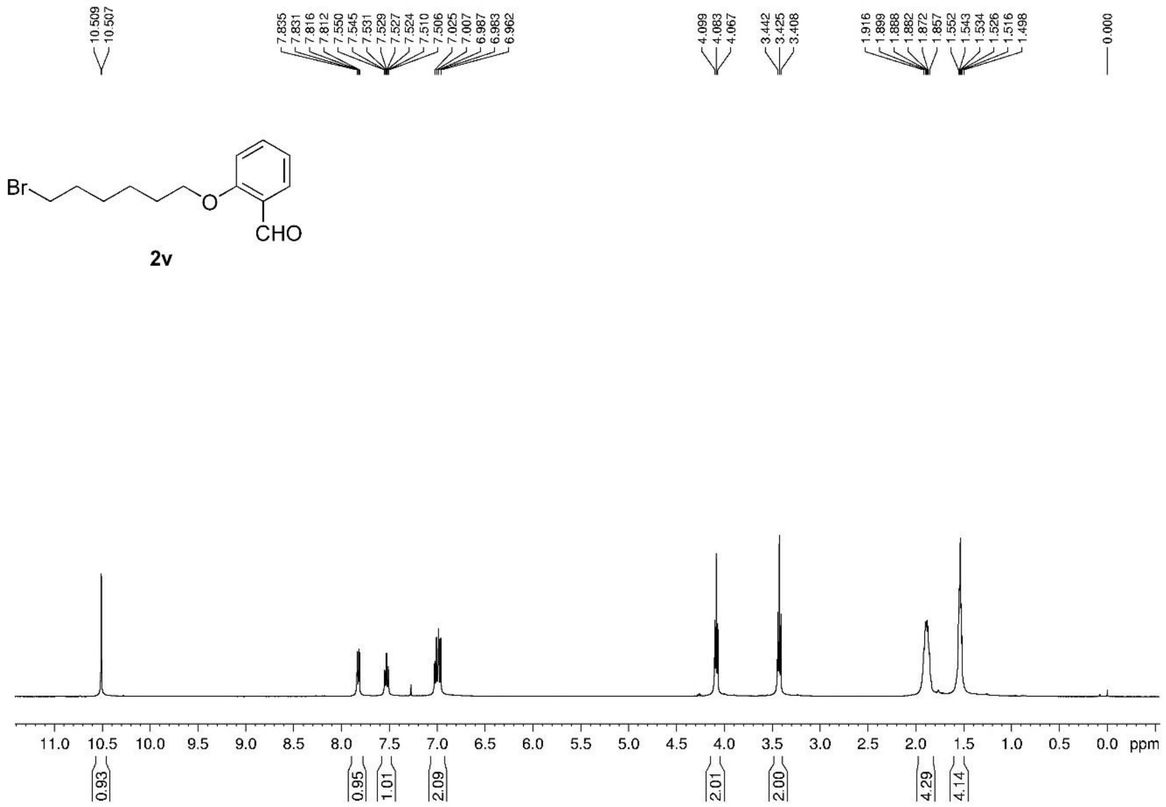
137.90
128.75
124.12
120.06
77.32
77.00
76.68
37.08
33.52
32.28
27.58
24.58
Br
O
N
H
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


3.908
3.891
3.875
3.413
3.397
3.381
0.906
0.089
0.000
Br
OTBS
2t
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.01
2.00
9.30
6.08


77.32
77.00
76.68
63.48
33.27
25.80
18.31
-5.29
Br
OTBS
2t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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10.507
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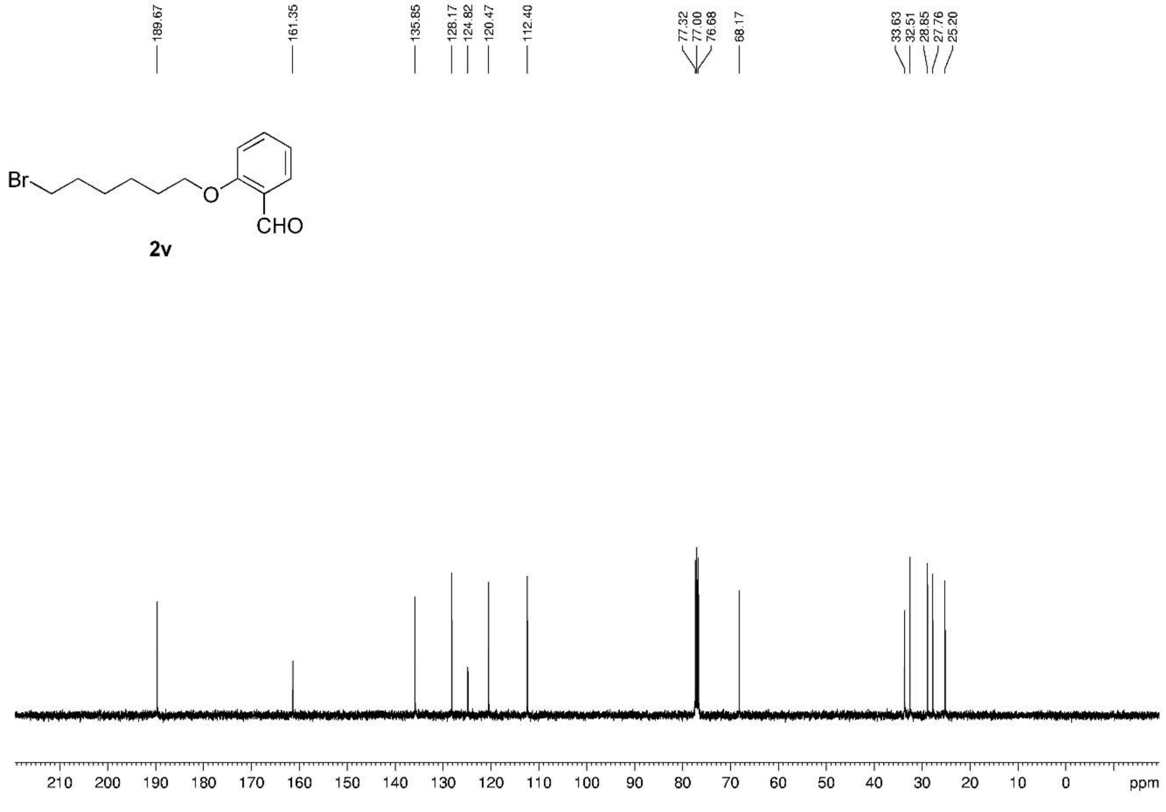
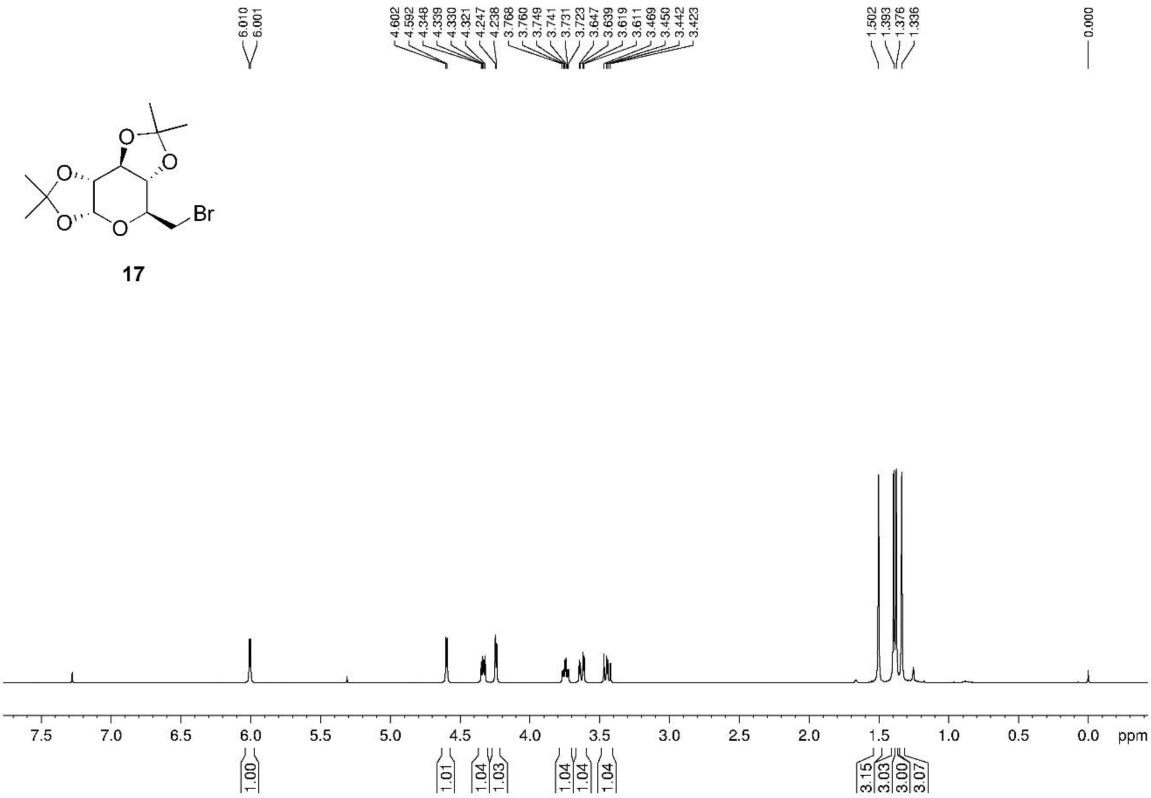
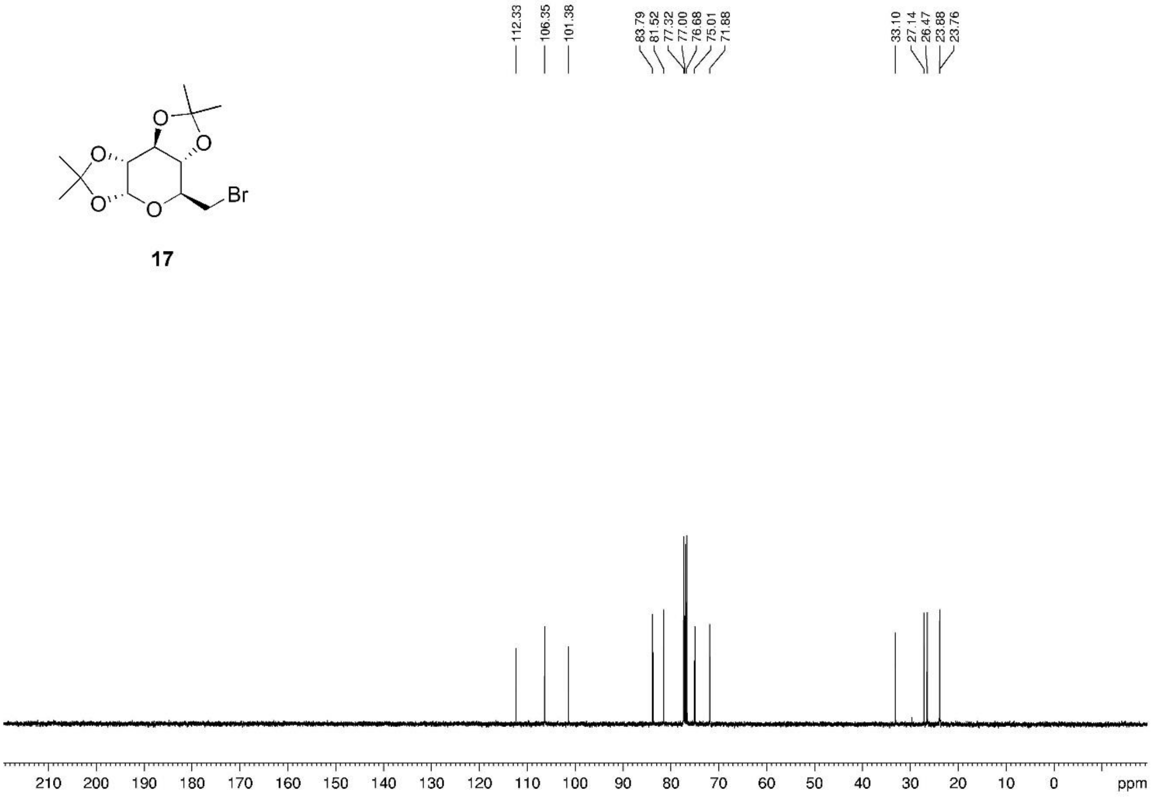
7.831
7.816
7.812
7.550
7.545
7.531
7.529
7.527
7.524
7.510
7.506
7.025
7.007
6.987
6.983
6.962
4.099
4.083
4.067
3.442
3.425
3.408
1.916
1.899
1.888
1.882
1.872
1.857
1.552
1.543
1.534
1.526
1.516
1.498
0.000
Br
O
CHO
2v
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.93
0.95
1.01
2.09
2.01
2.00
4.29
4.14


189.67
161.35
135.85
128.17
124.82
120.47
112.40
77.32
77.00
76.68
68.17
33.63
32.51
28.85
27.76
25.20
Br
O
CHO
2v
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


6.010
6.001
4.602
4.592
4.348
4.339
4.330
4.321
4.247
4.238
3.768
3.760
3.749
3.741
3.731
3.723
3.647
3.639
3.619
3.611
3.469
3.450
3.442
3.423
1.502
1.393
1.376
1.336
0.000
O
O
O
O
O
Br
17
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
1.04
1.03
1.04
1.04
1.04
3.15
3.03
3.00
3.07


112.33
106.35
101.38
83.79
81.52
77.32
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O
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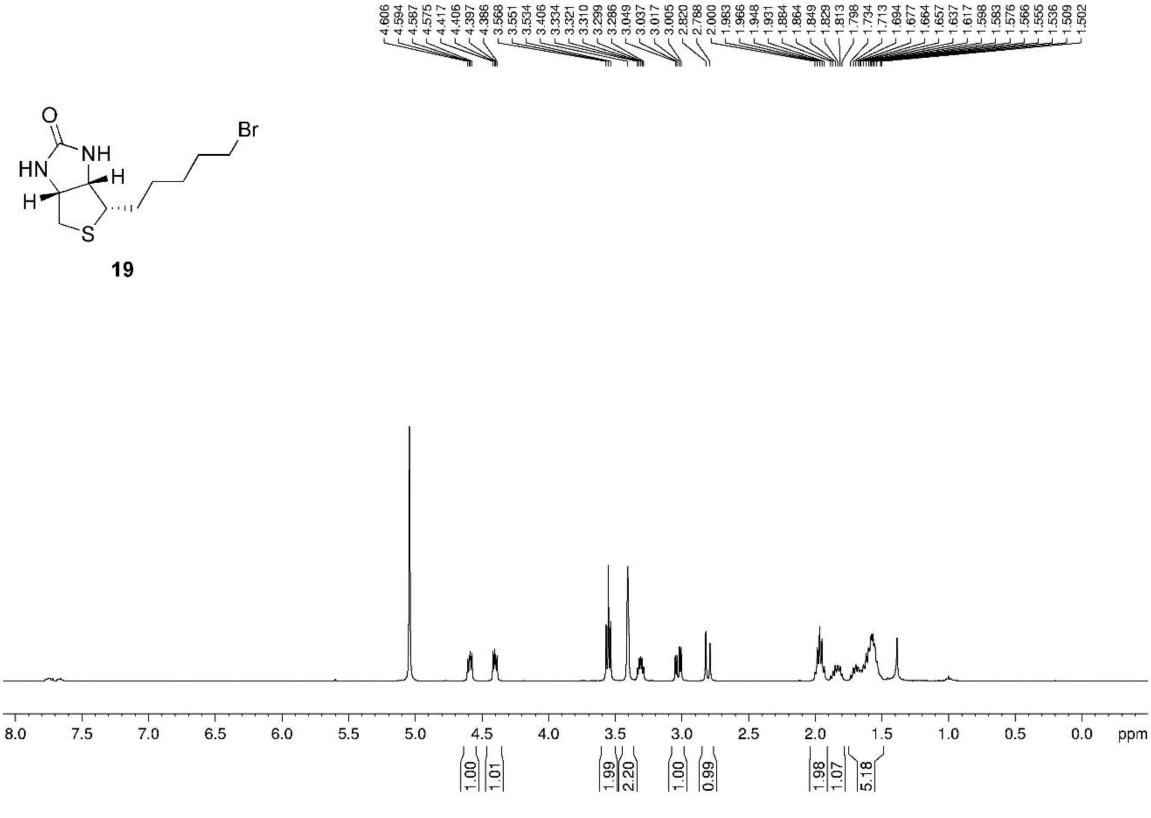
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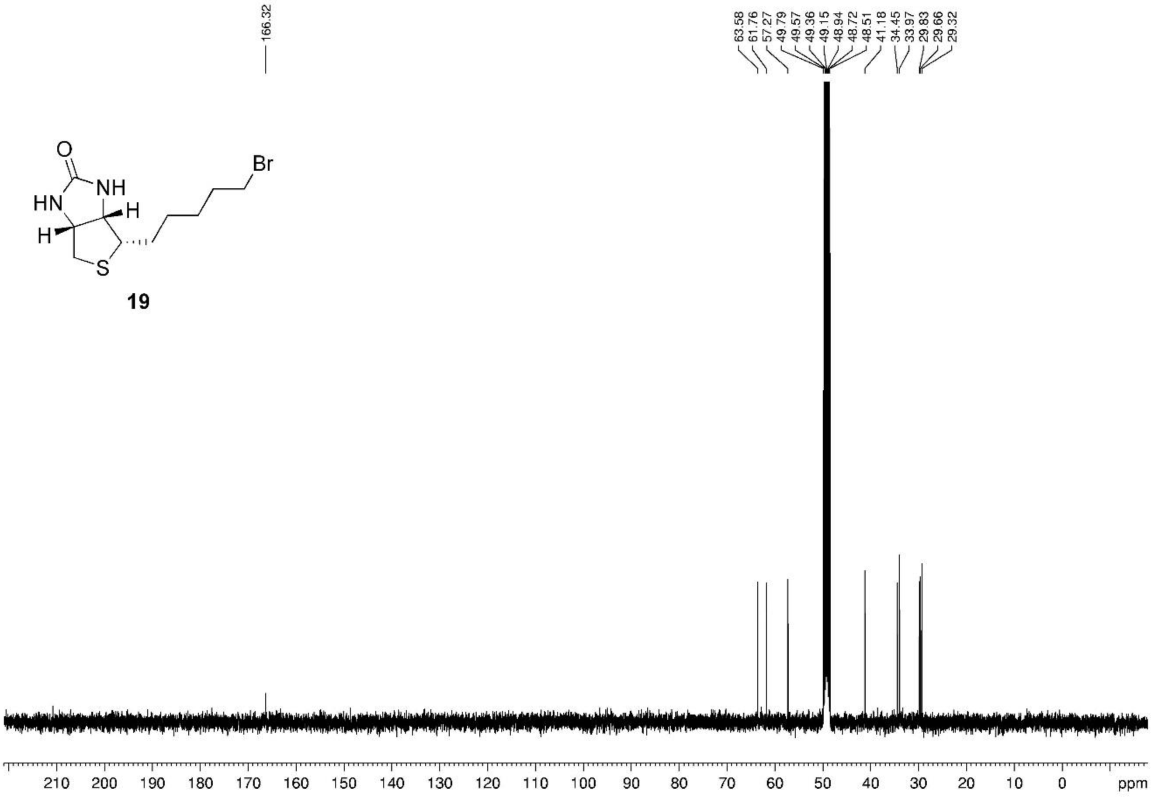
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H
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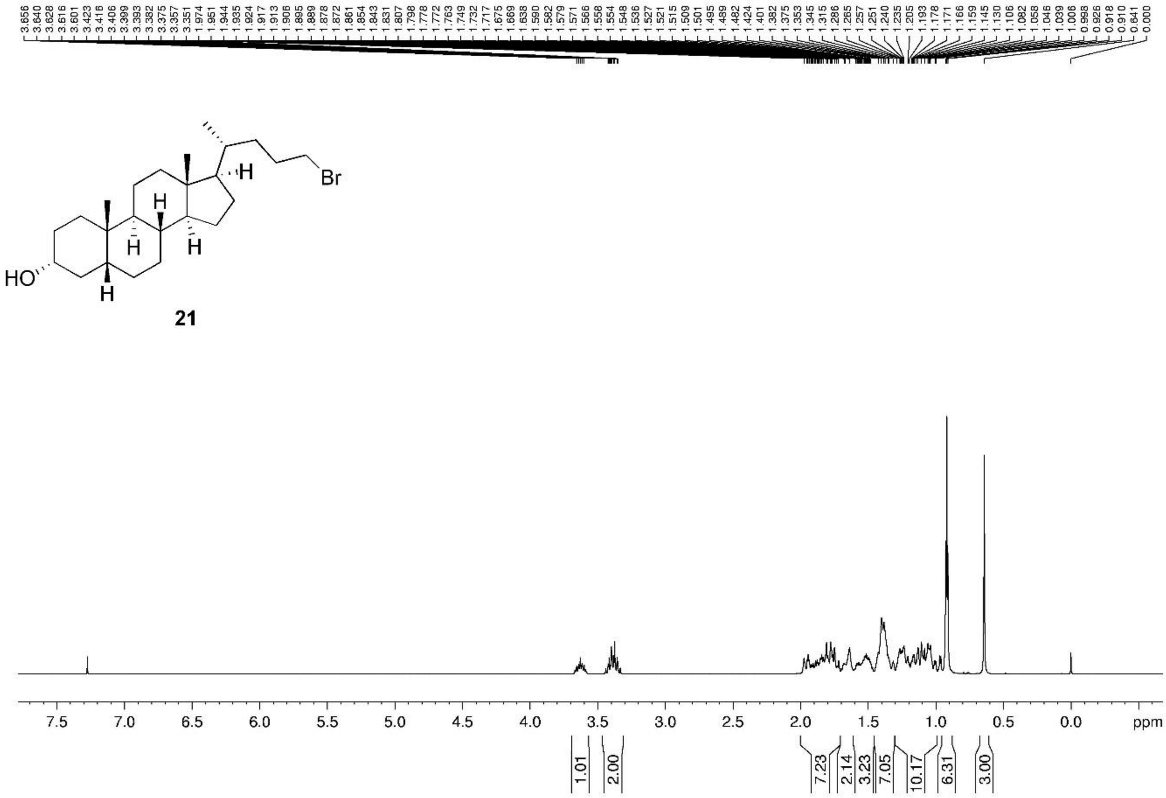
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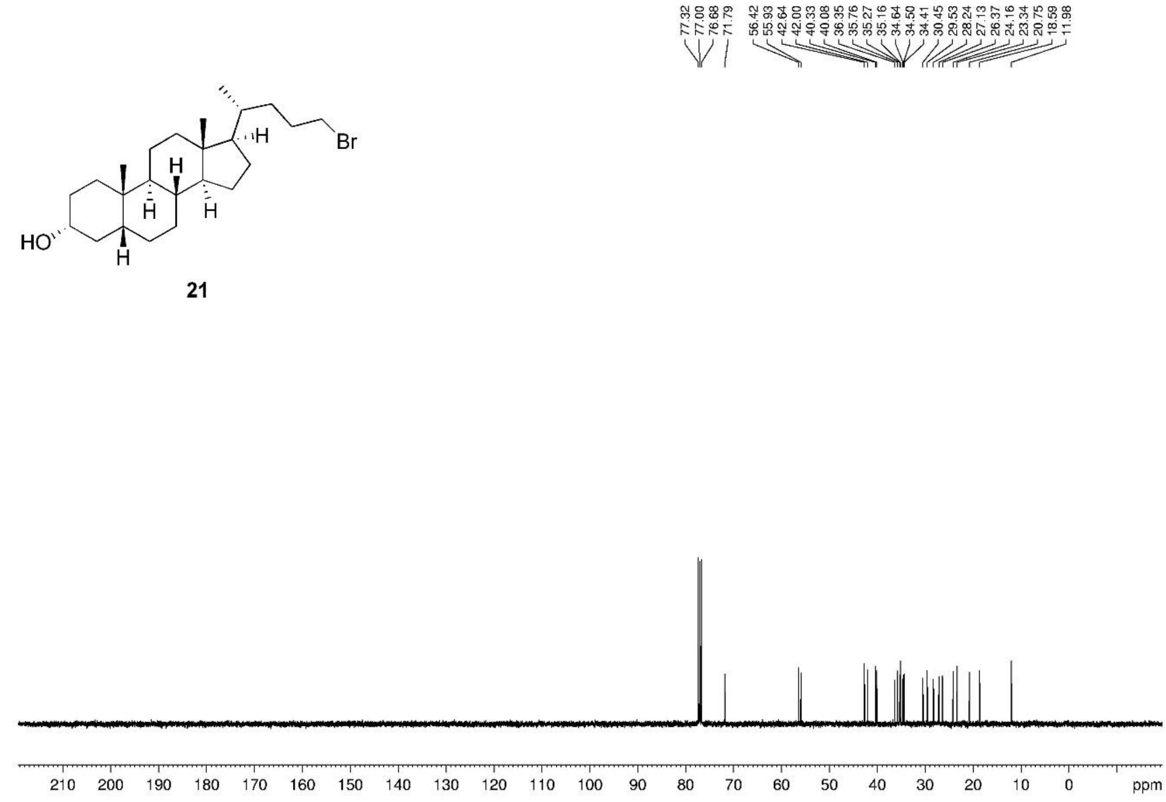
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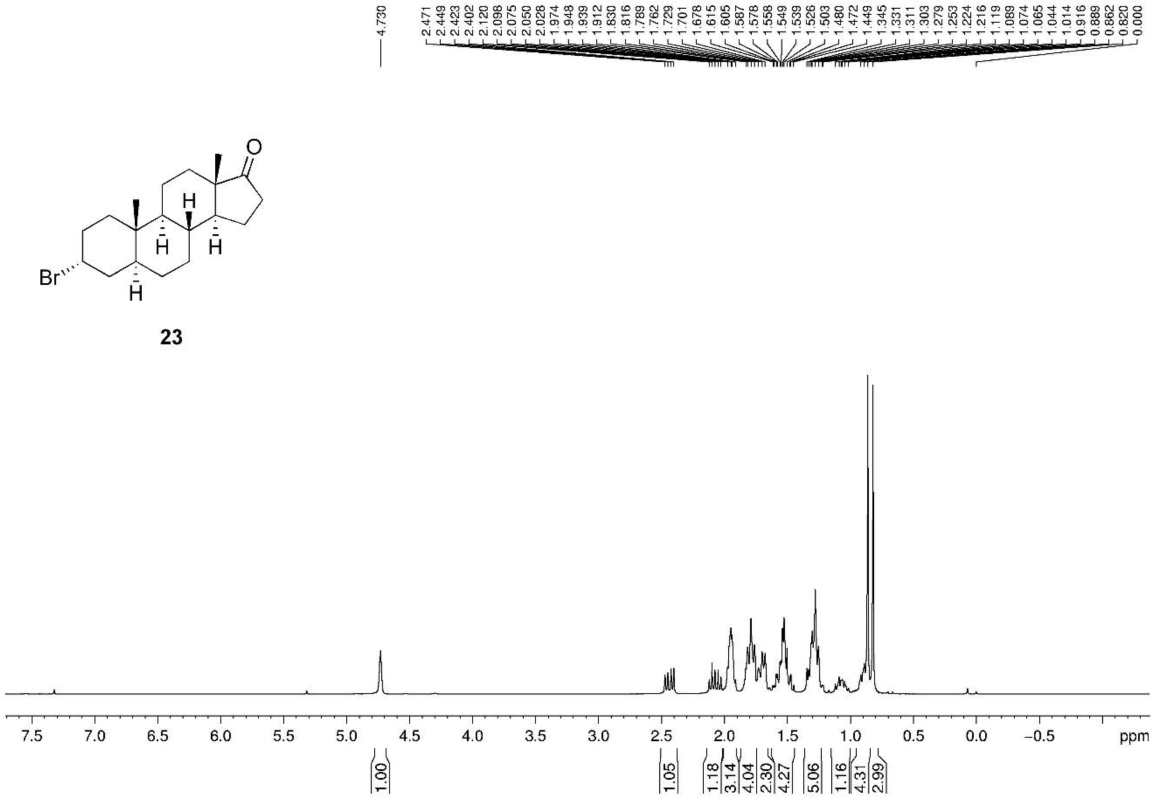

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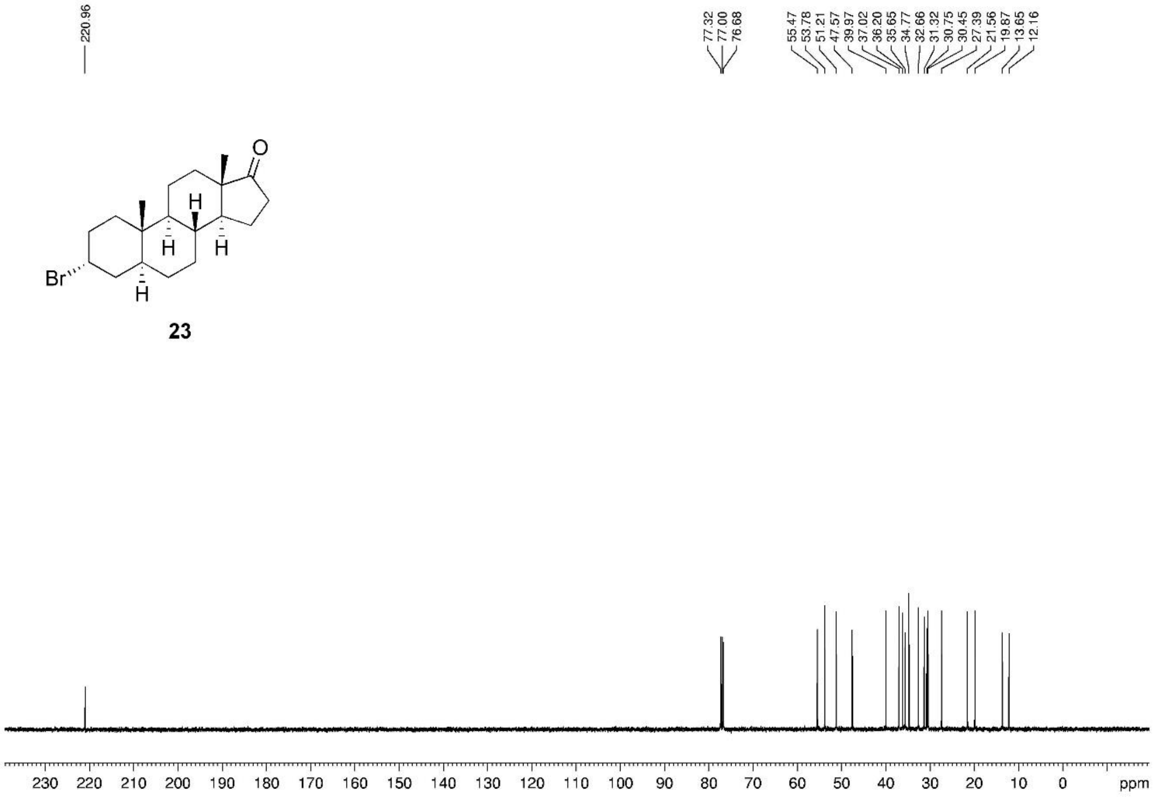

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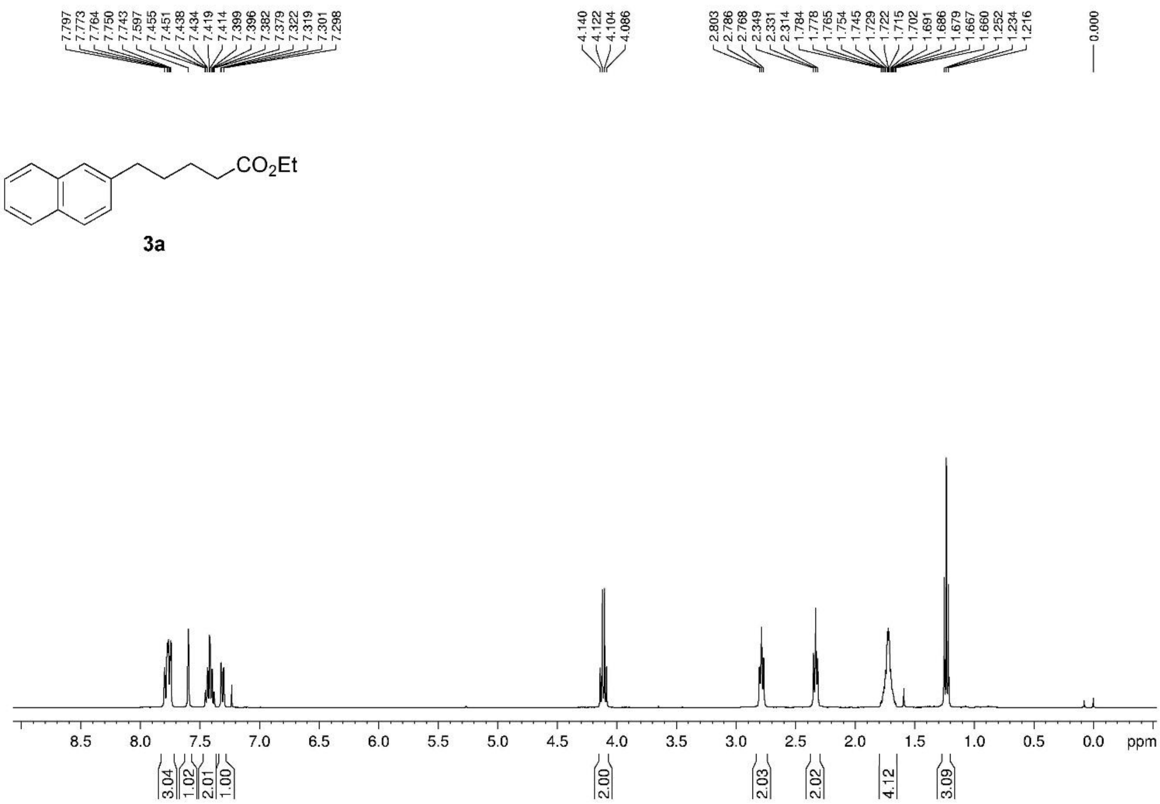
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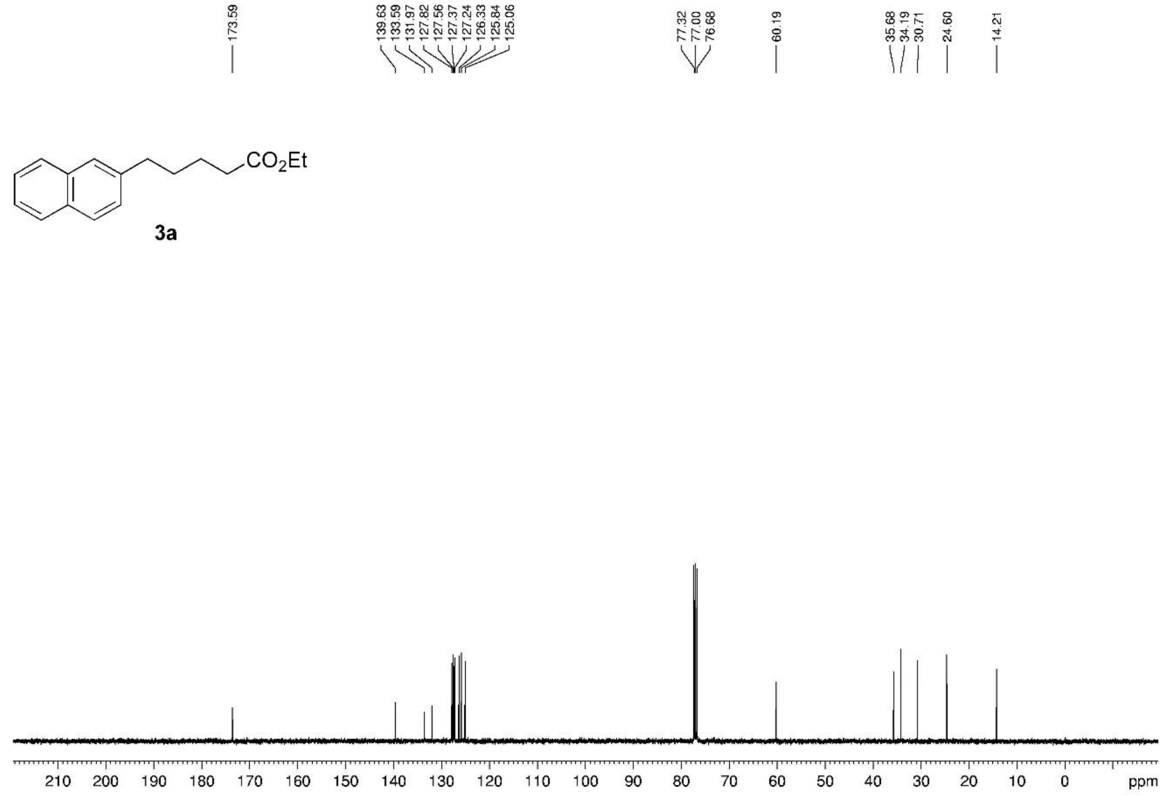
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CO2Et
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200
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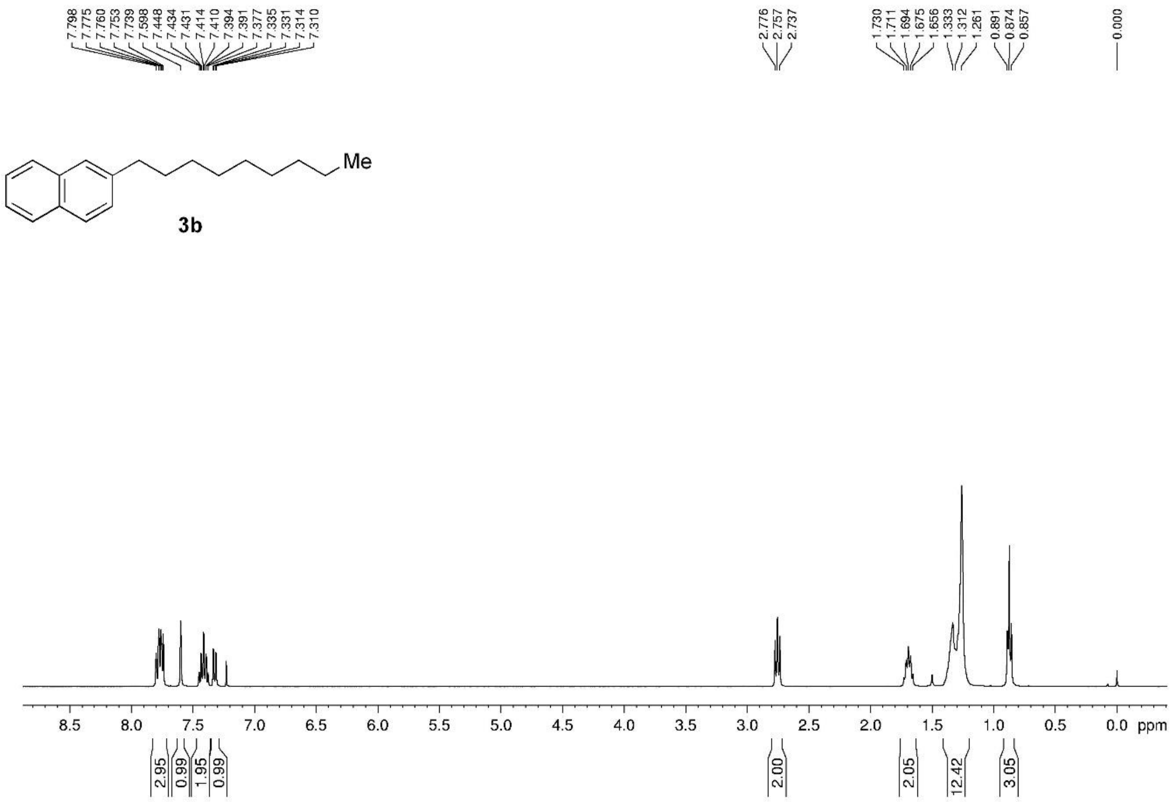
3a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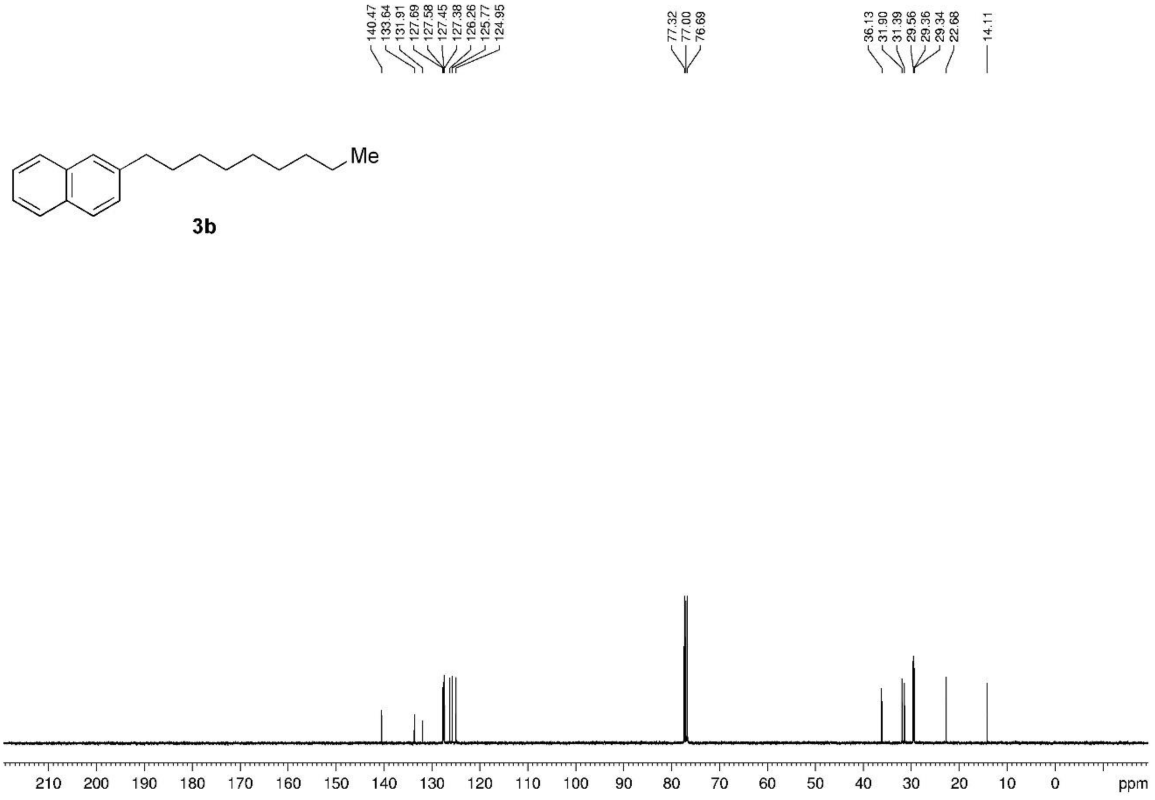
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Me
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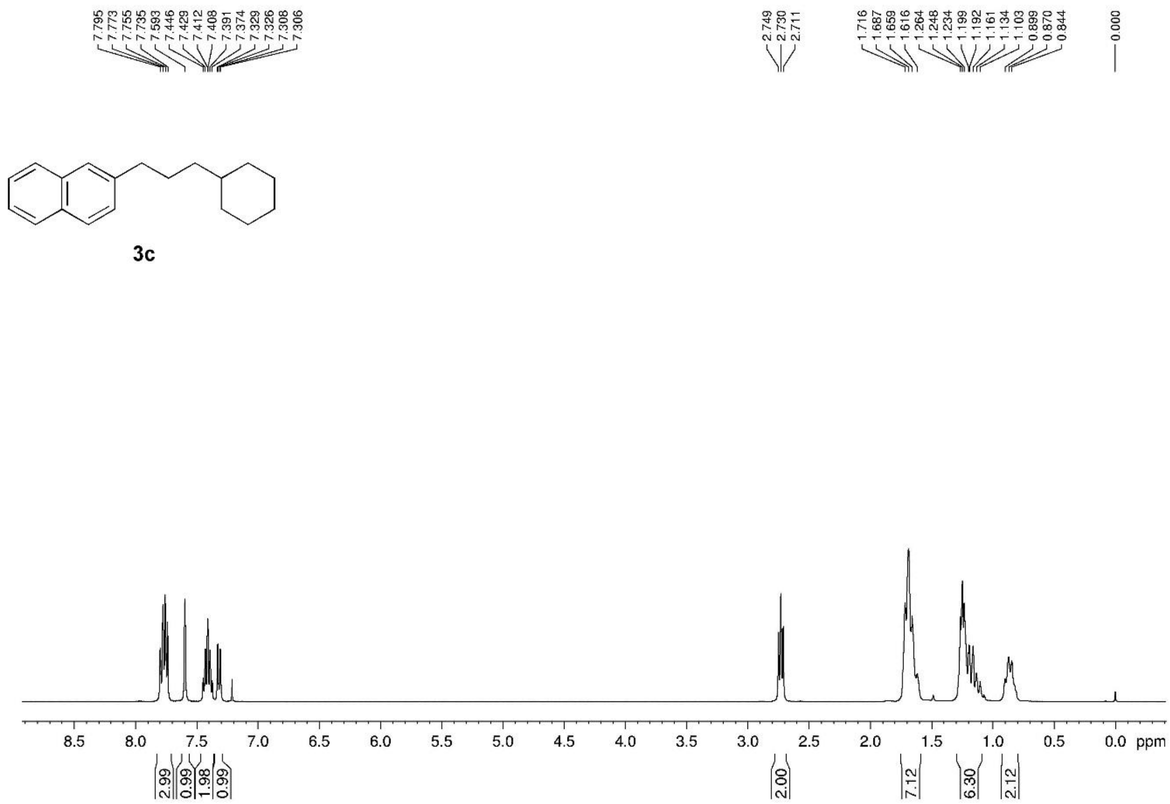
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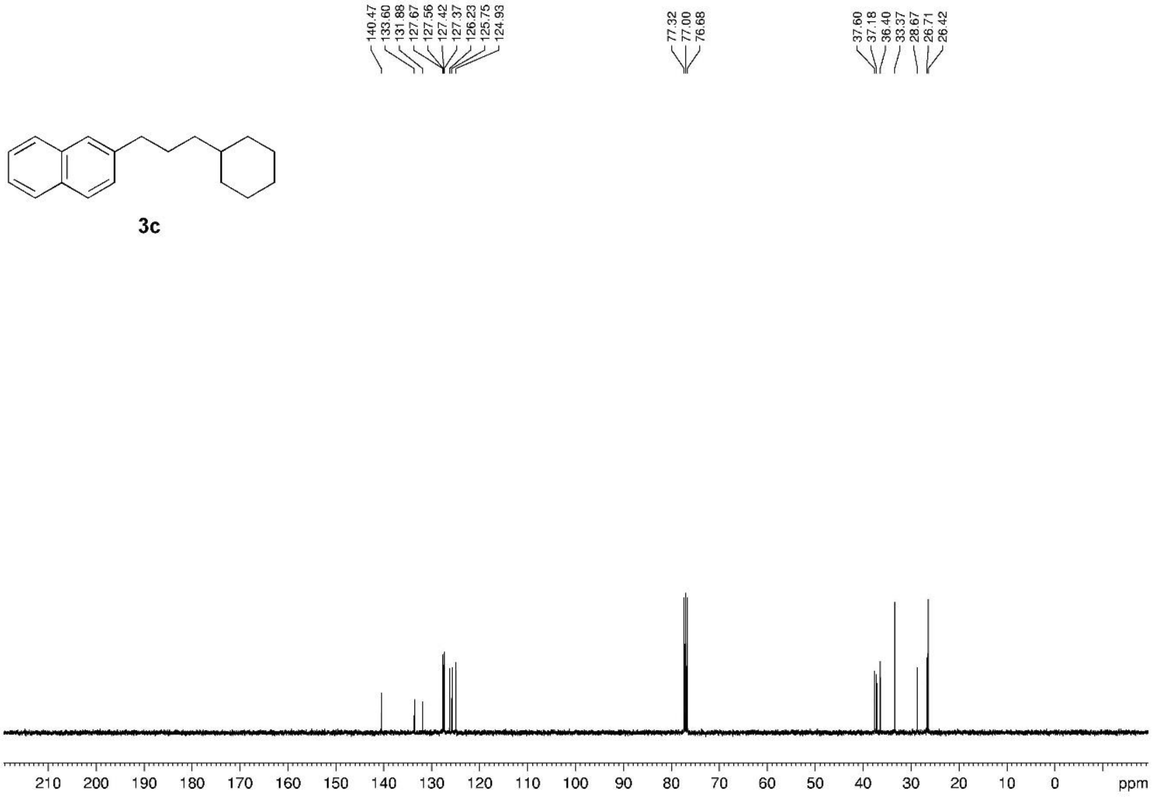
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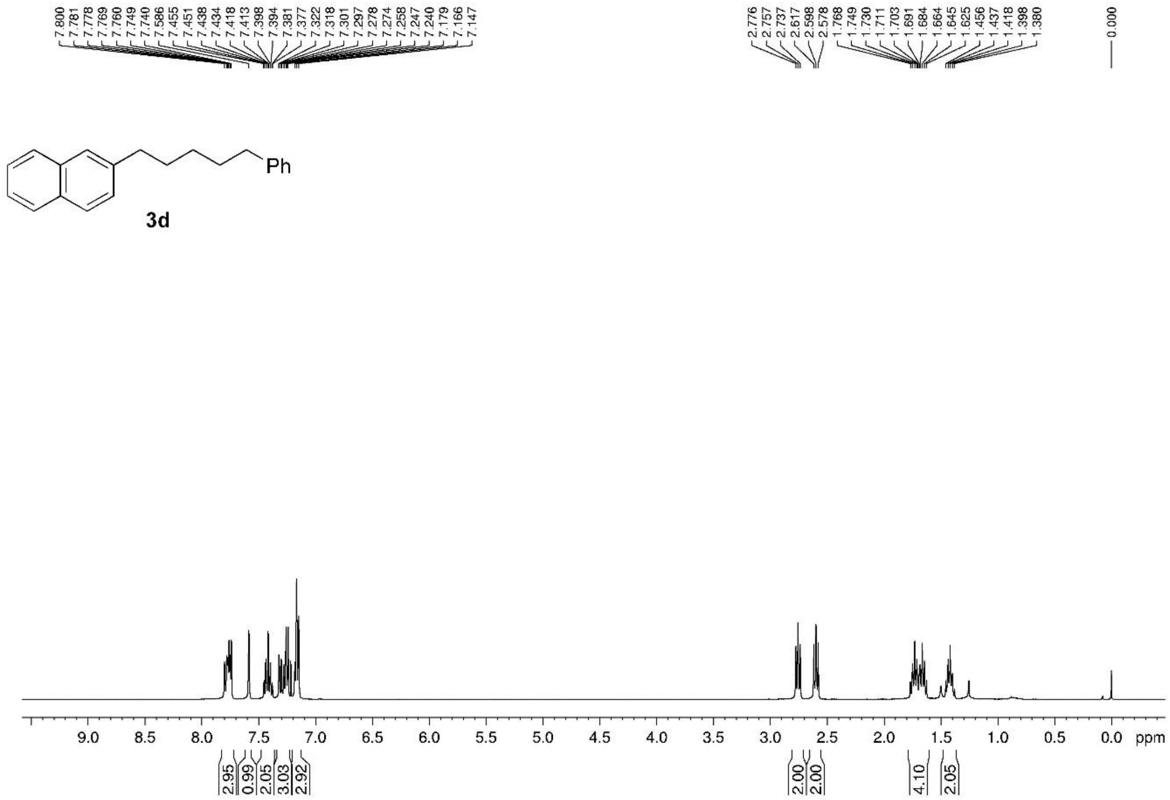
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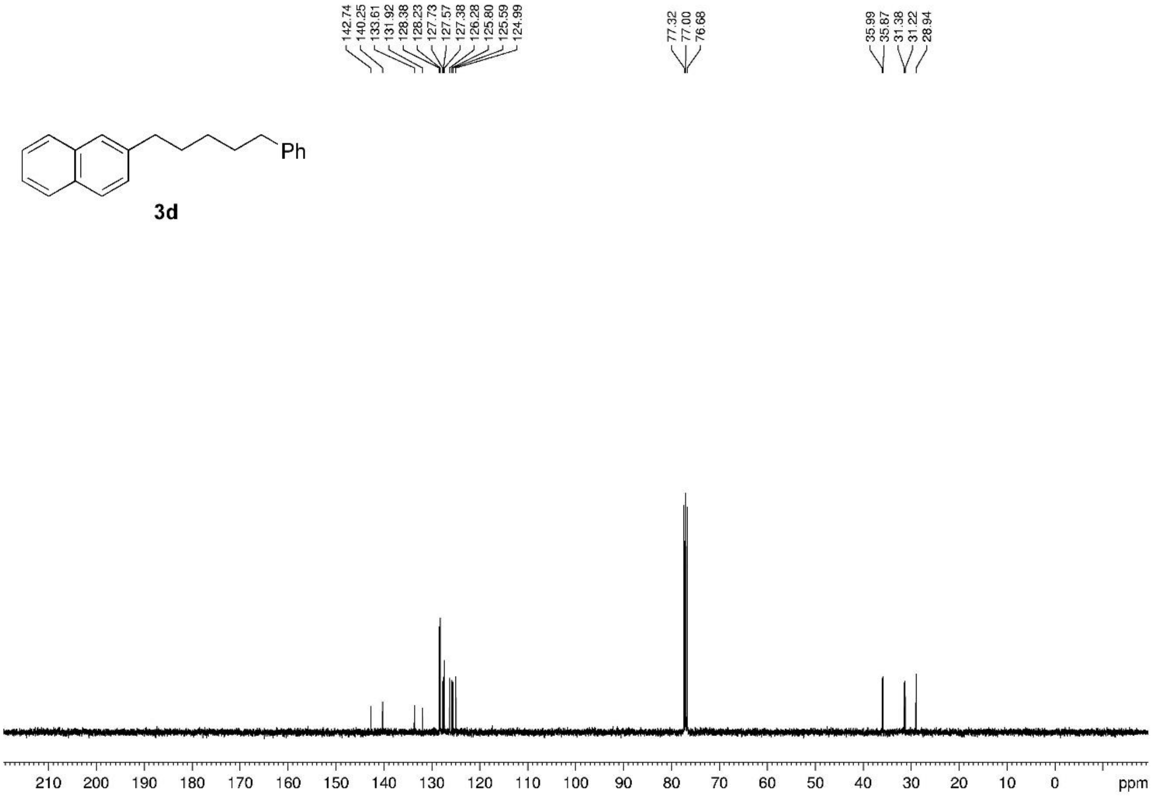
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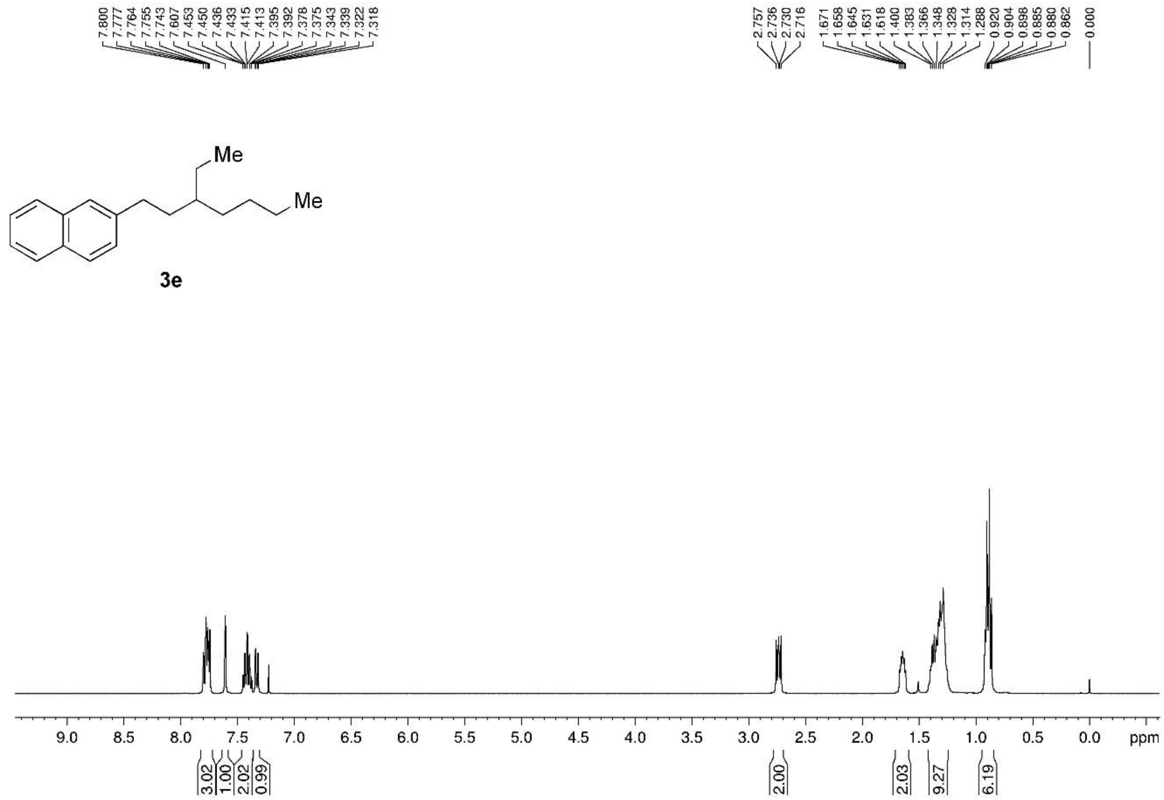
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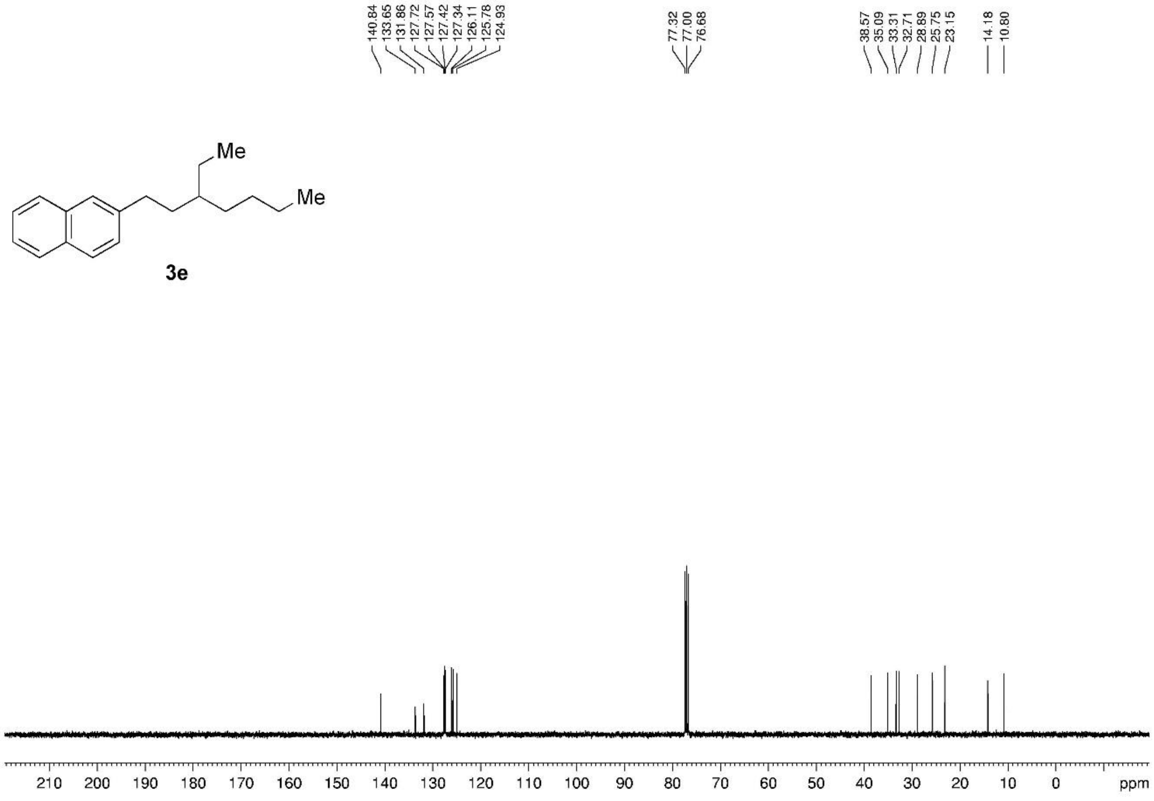
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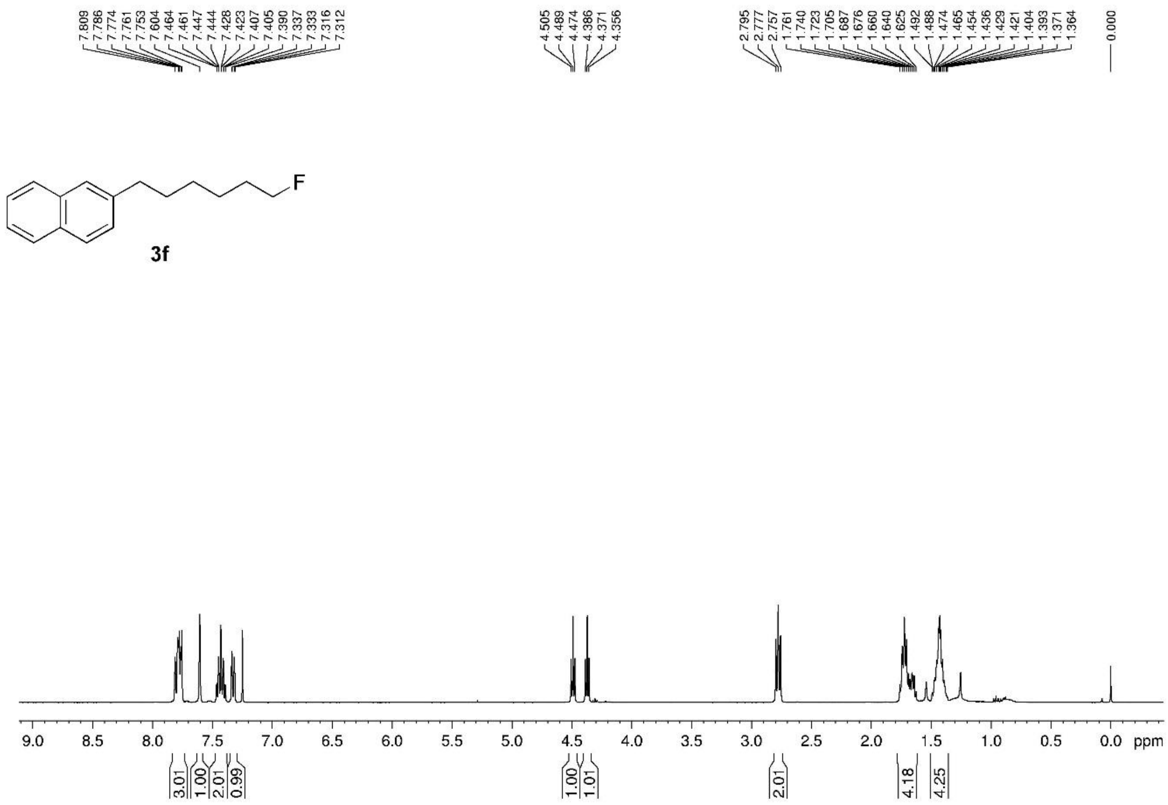
3e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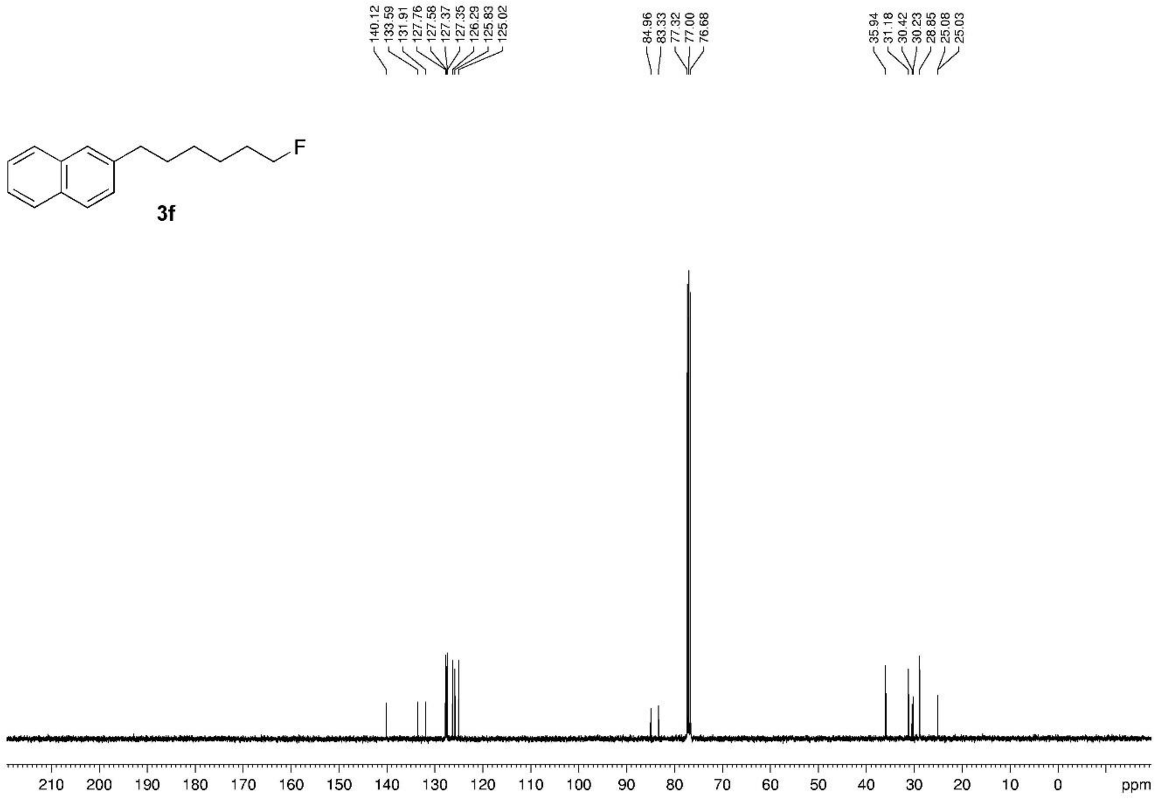
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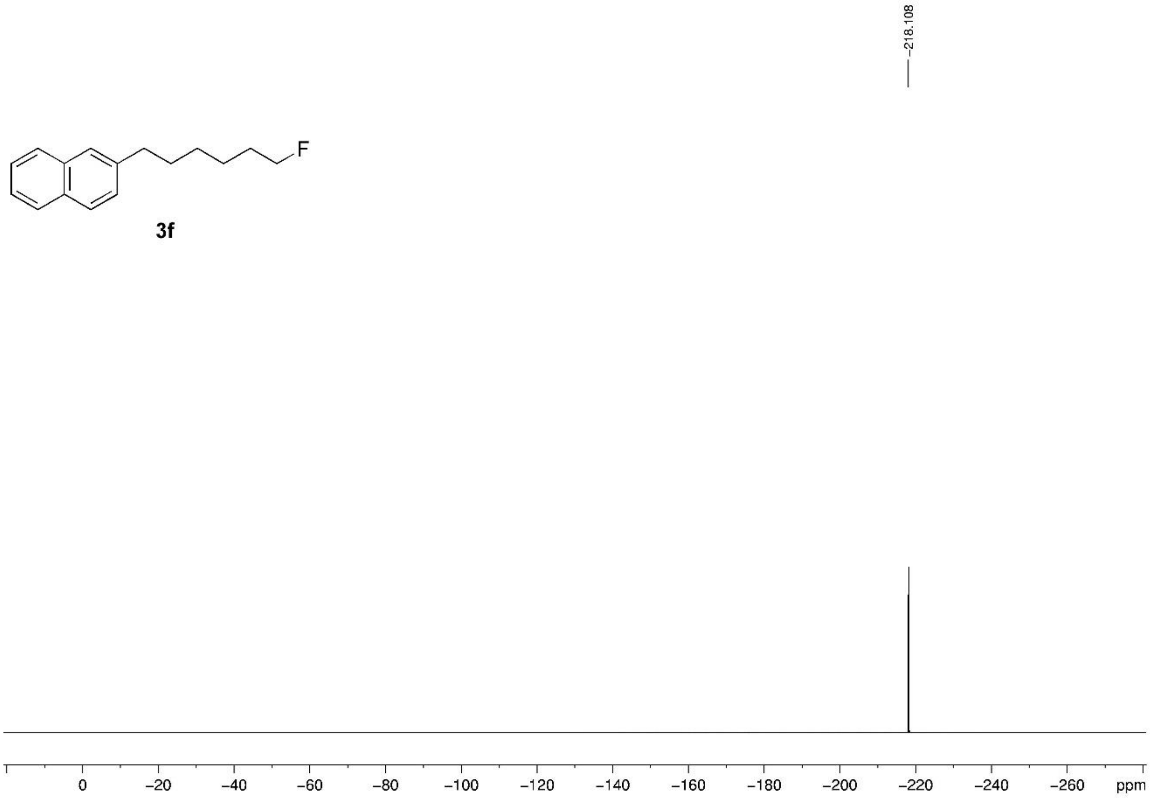

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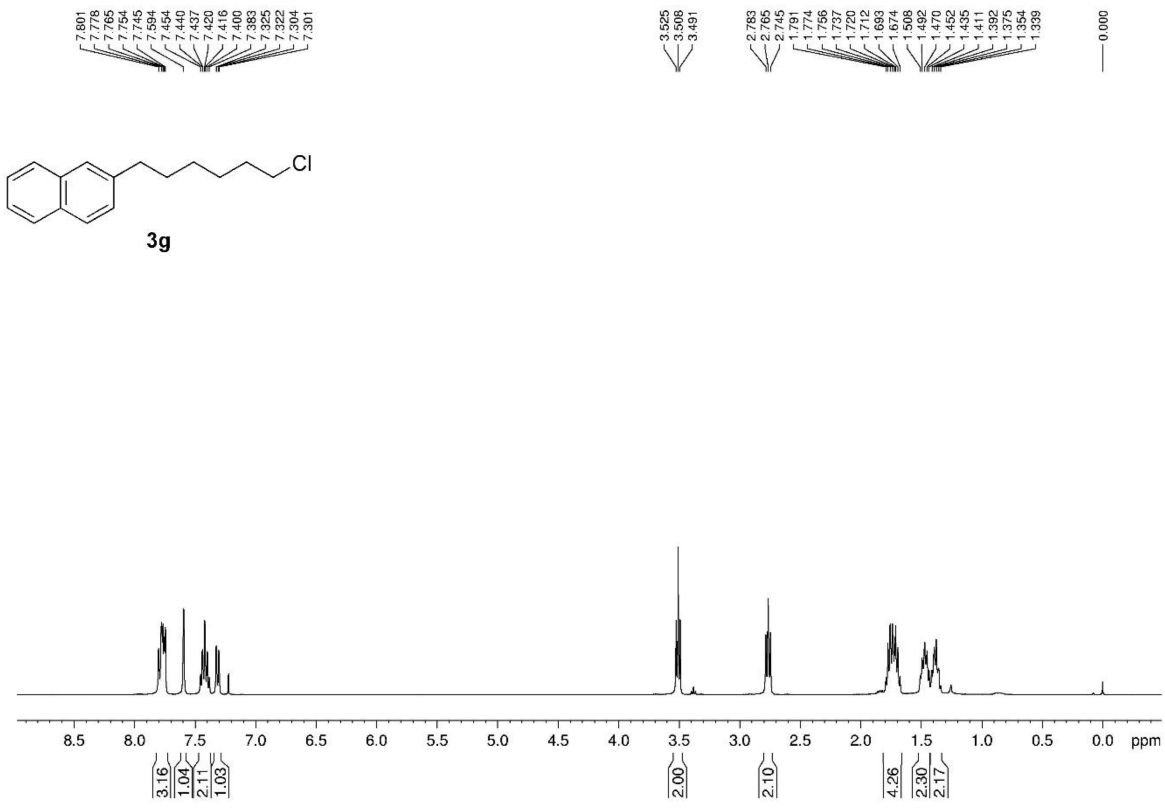
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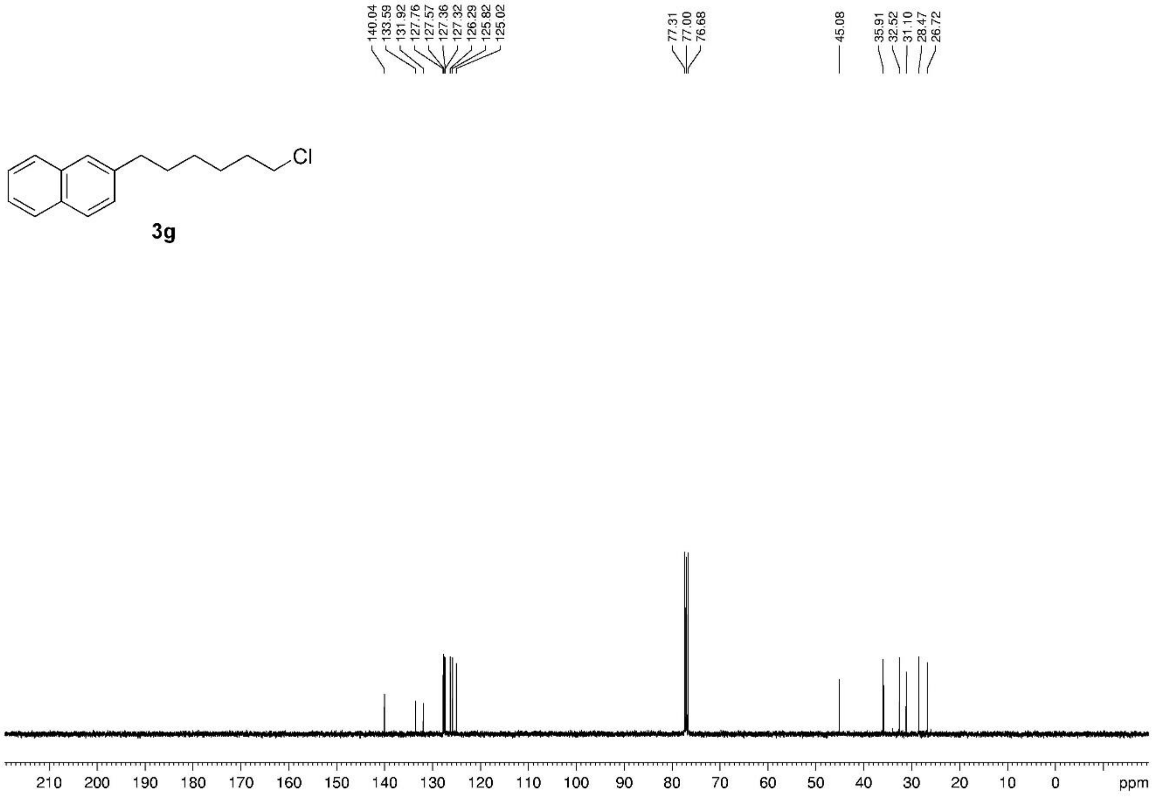
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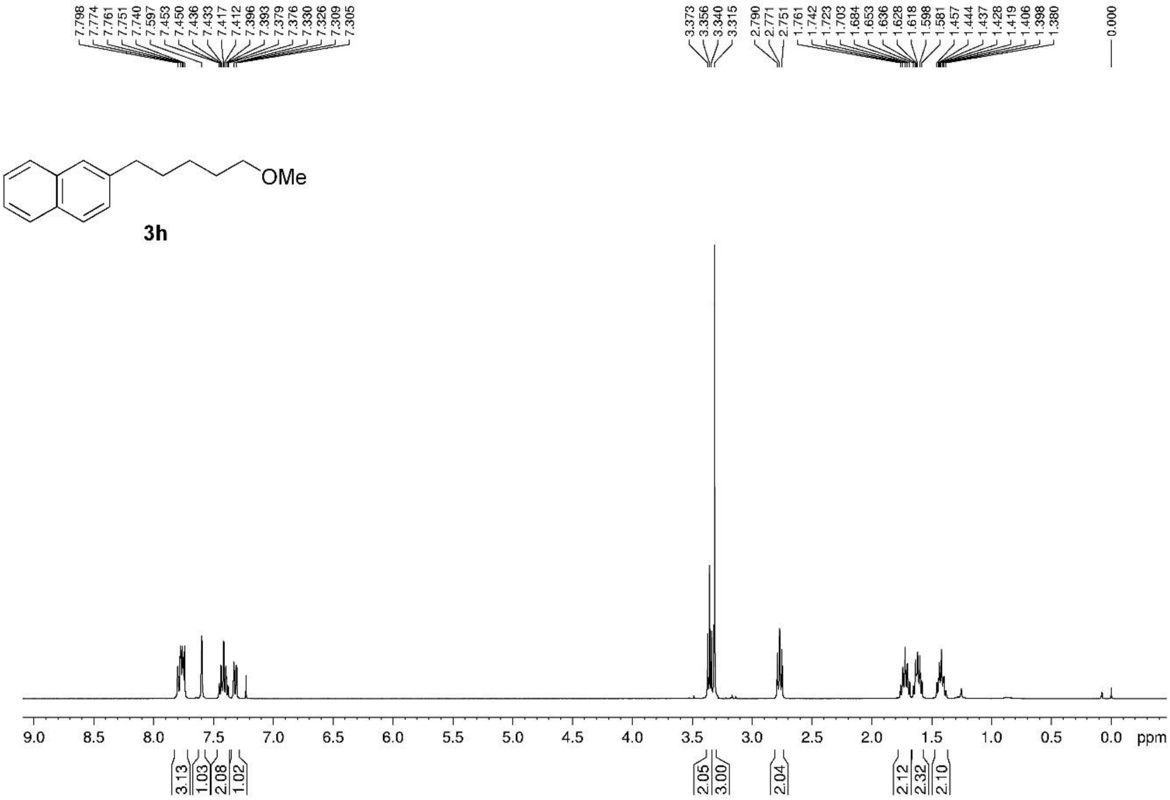
40
30
20
10
0
ppm

3g


3h

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7.774
7.761
7.751
7.740
7.597
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7.450
7.436
7.433
7.417
7.412
7.396
7.393
7.379
7.376
7.330
7.326
7.309
7.305
3.373
3.356
3.340
3.315
2.790
2.771
2.751
1.761
1.742
1.723
1.703
1.684
1.653
1.636
1.628
1.618
1.598
1.581
1.457
1.444
1.437
1.428
1.419
1.406
1.398
1.380
0.000
OMe
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
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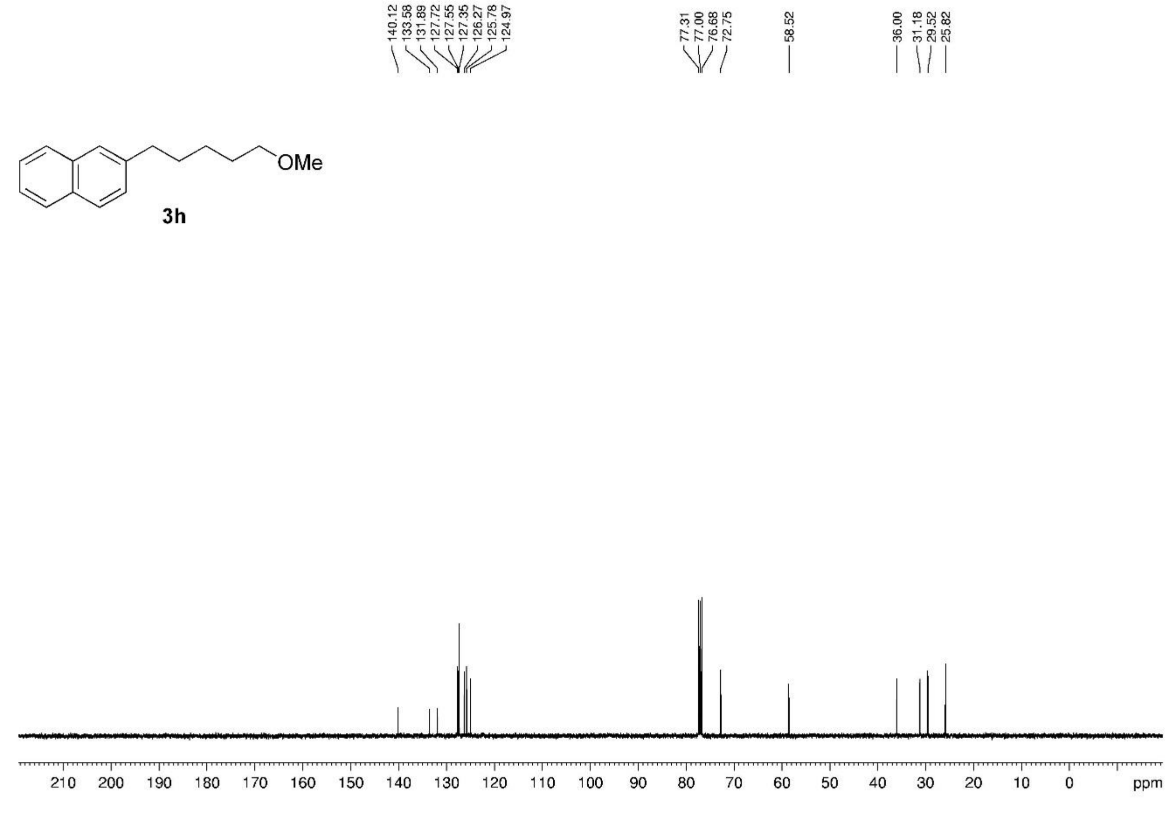
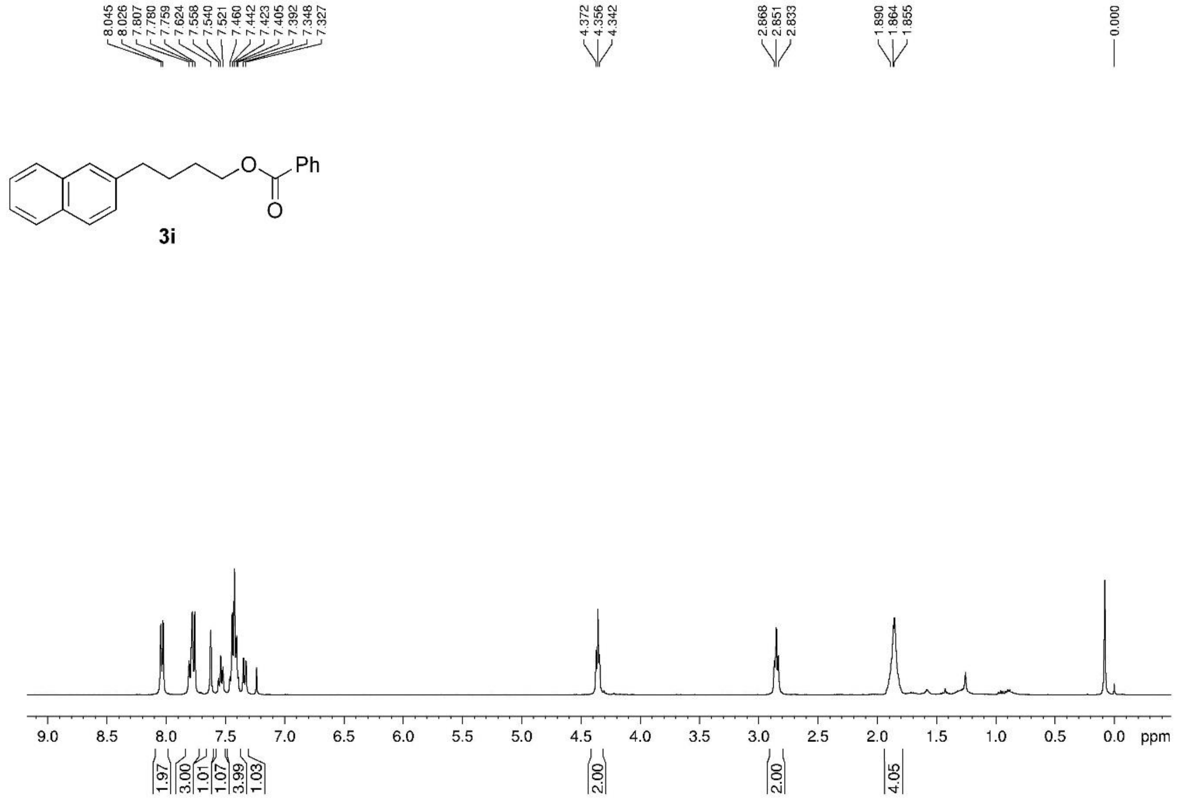
1.03
2.08
1.02
2.05
3.00
2.04
2.12
2.32
2.10


3h

140.12
133.58
131.89
127.72
127.55
127.35
126.27
125.78
124.97
77.31
77.00
76.68
72.75
58.52
36.00
31.18
29.52
25.82
OMe
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


3i

8.045
8.026
7.807
7.780
7.759
7.624
7.558
7.540
7.521
7.460
7.442
7.423
7.405
7.392
7.348
7.327
4.372
4.356
4.342
2.868
2.851
2.833
1.890
1.864
1.855
0.000
Ph
O
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
1.97
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1.01
1.07
3.99
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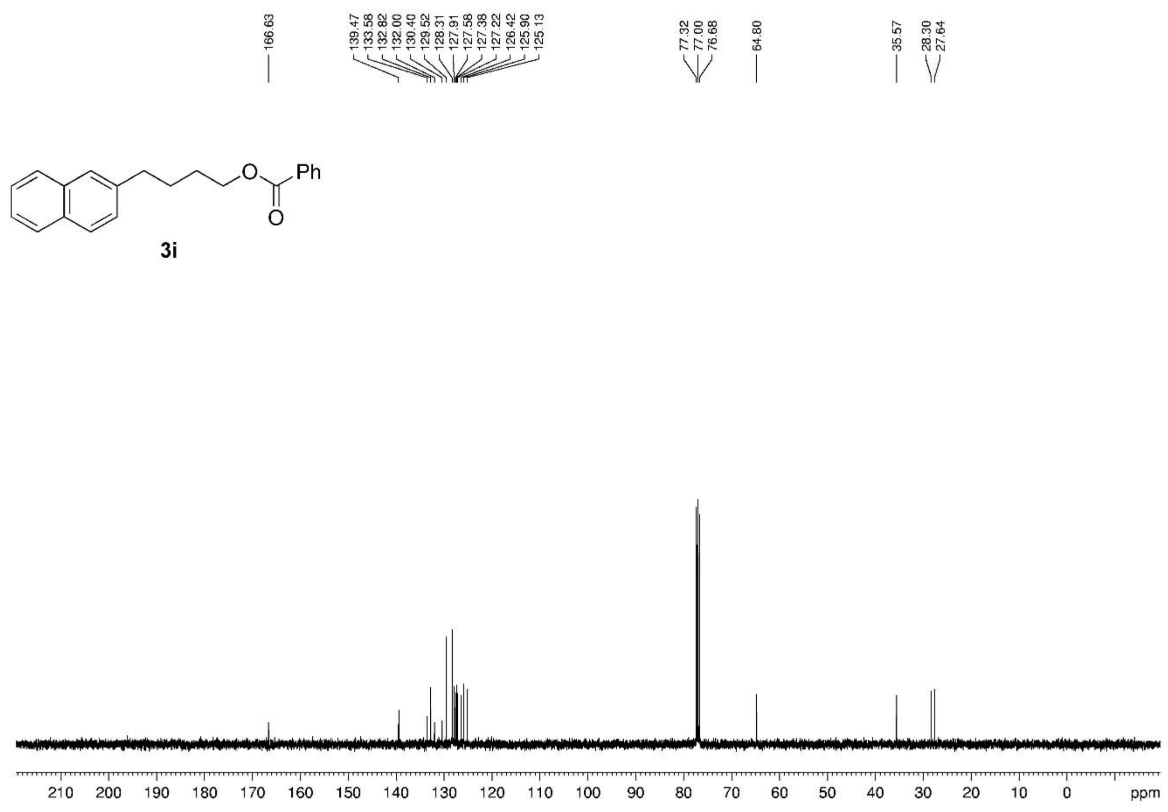
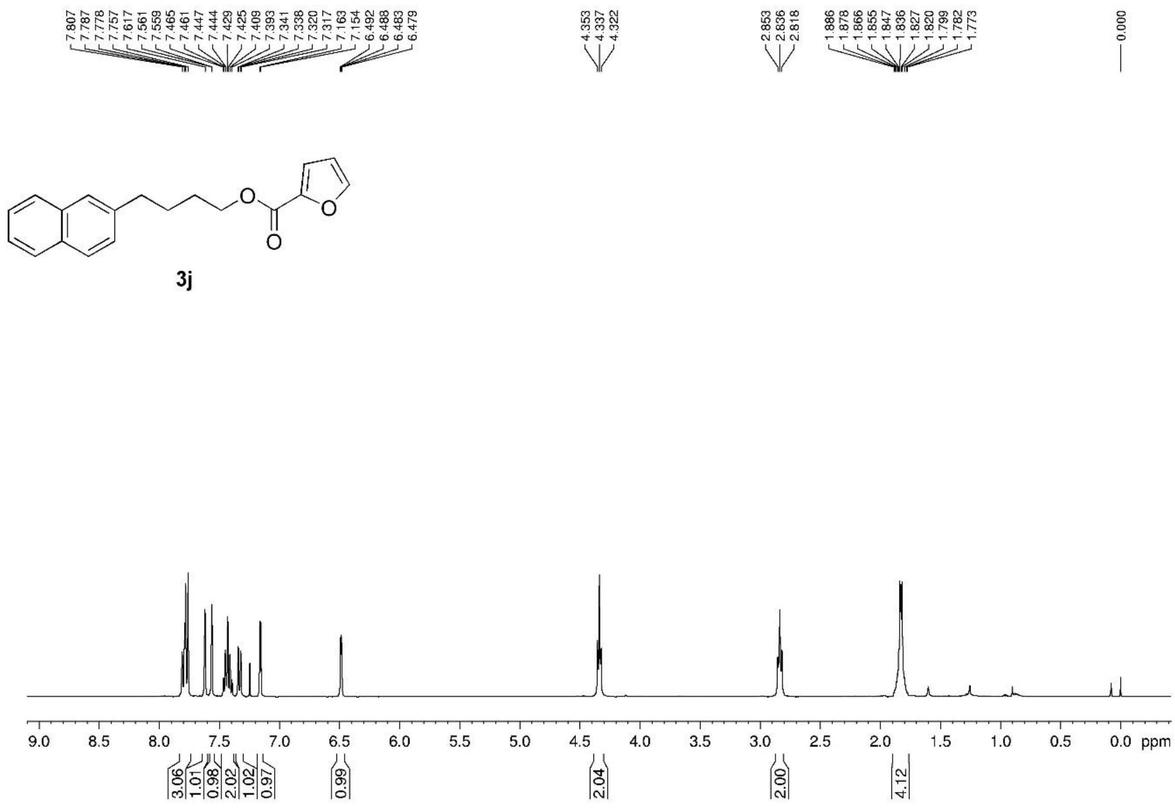
2.00
2.00
4.05


3i

166.63
139.47
133.58
132.82
132.00
130.40
129.52
128.31
127.91
127.58
127.38
127.22
126.42
125.90
125.13
77.32
77.00
76.68
64.80
35.57
28.30
27.64
Ph
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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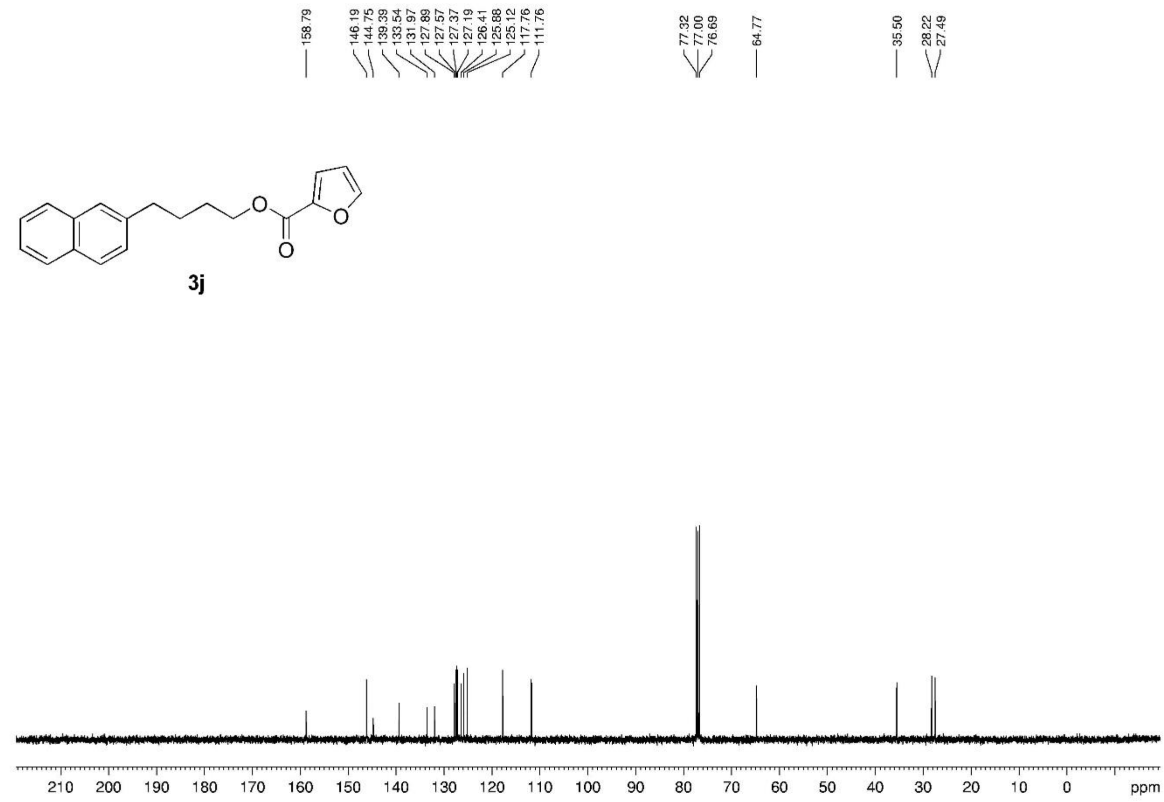
7.778
7.757
7.617
7.561
7.559
7.465
7.461
7.447
7.444
7.429
7.425
7.409
7.393
7.341
7.338
7.320
7.317
7.163
7.154
6.492
6.488
6.483
6.479
4.353
4.337
4.322
2.853
2.836
2.818
1.886
1.878
1.866
1.855
1.847
1.836
1.827
1.820
1.799
1.782
1.773
0.000
O
O
O
3j
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.06
1.01
0.98
2.02
1.02
0.97
0.99
2.04
2.00
4.12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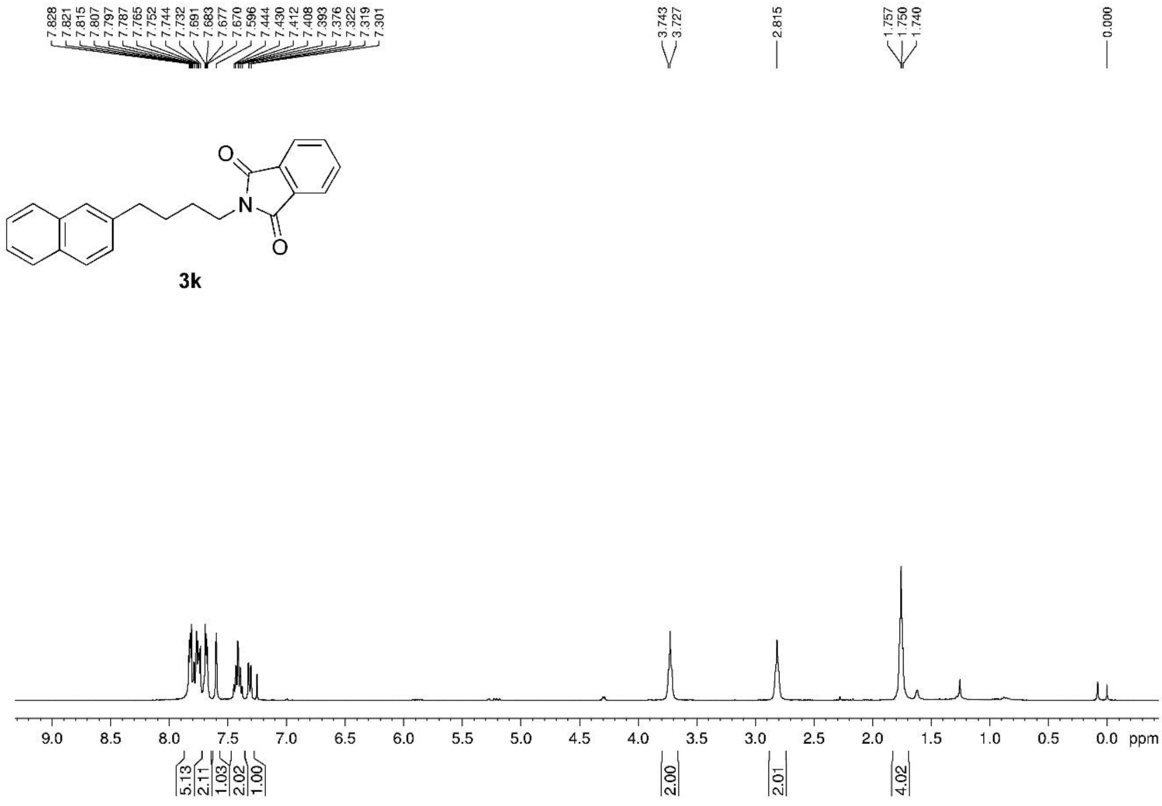
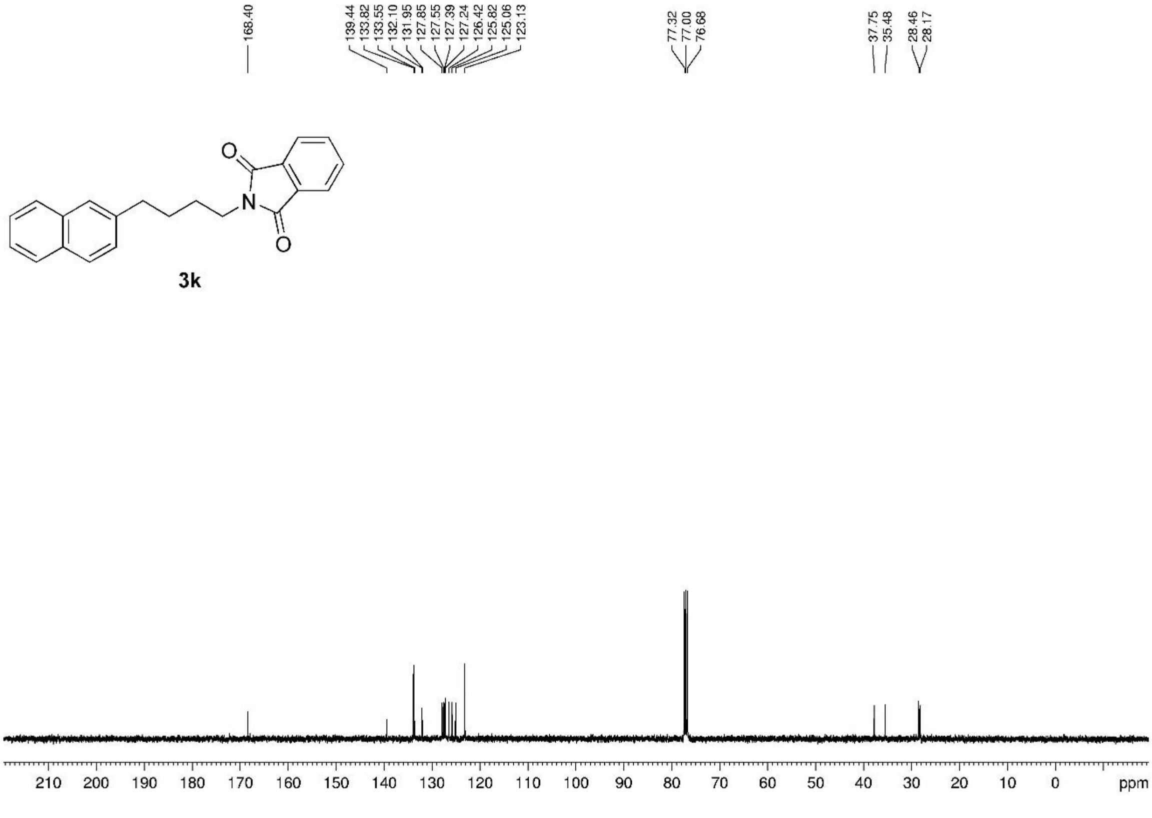
146.19
144.75
139.39
133.54
131.97
127.89
127.57
127.37
127.19
126.41
125.88
125.12
117.76
111.76
77.32
77.00
76.69
64.77
35.50
28.22
27.49
O
O
O
3j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.828
7.821
7.815
7.807
7.797
7.787
7.765
7.752
7.744
7.732
7.691
7.683
7.677
7.670
7.596
7.444
7.430
7.412
7.408
7.393
7.376
7.322
7.319
7.301
3.743
3.727
2.815
1.757
1.750
1.740
0.000
O
N
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.13
2.11
1.03
2.02
1.00
2.00
2.01
4.02

3k


168.40
139.44
133.82
133.55
132.10
131.95
127.85
127.55
127.39
127.24
126.42
125.82
125.06
123.13
77.32
77.00
76.68
37.75
35.48
28.46
28.17
O
N
O
210
200
190
180
170
160
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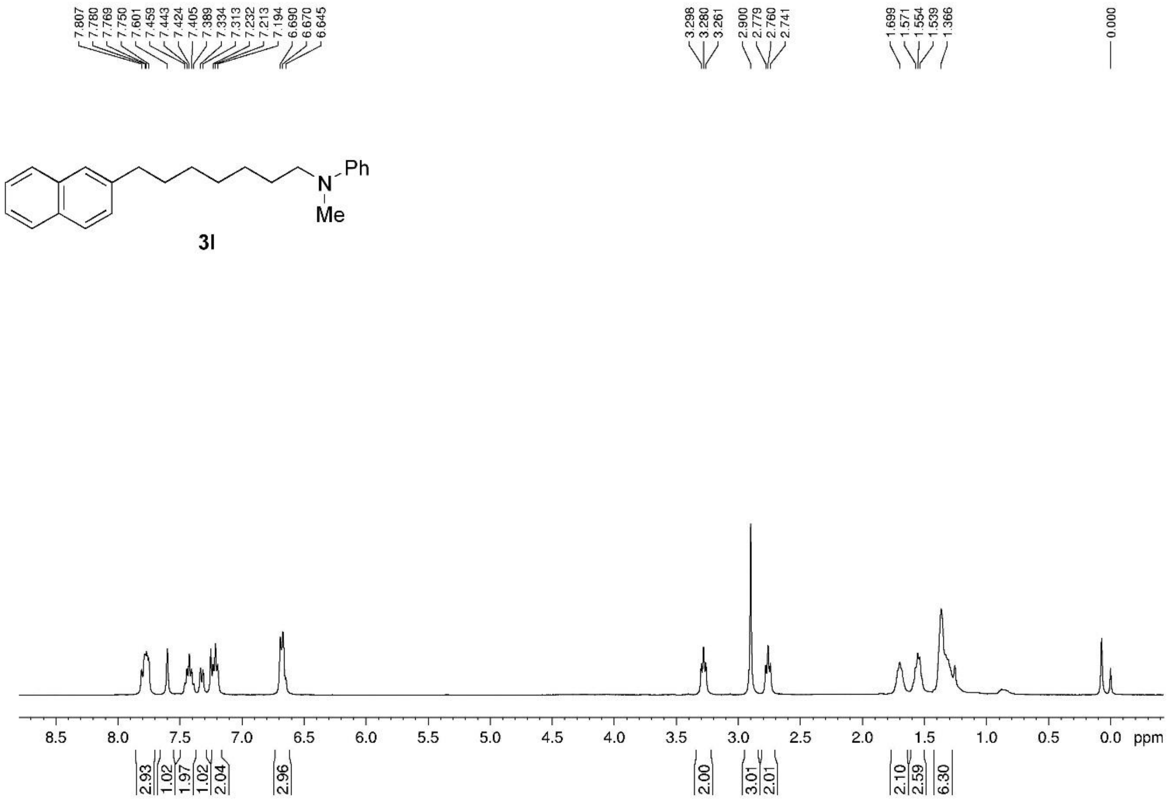
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3k



3l

7.807
7.780
7.769
7.750
7.601
7.459
7.443
7.424
7.405
7.389
7.334
7.313
7.232
7.213
7.194
6.690
6.670
6.645
3.298
3.280
3.261
2.900
2.779
2.760
2.741
1.699
1.571
1.554
1.539
1.366
0.000
N
Ph
Me
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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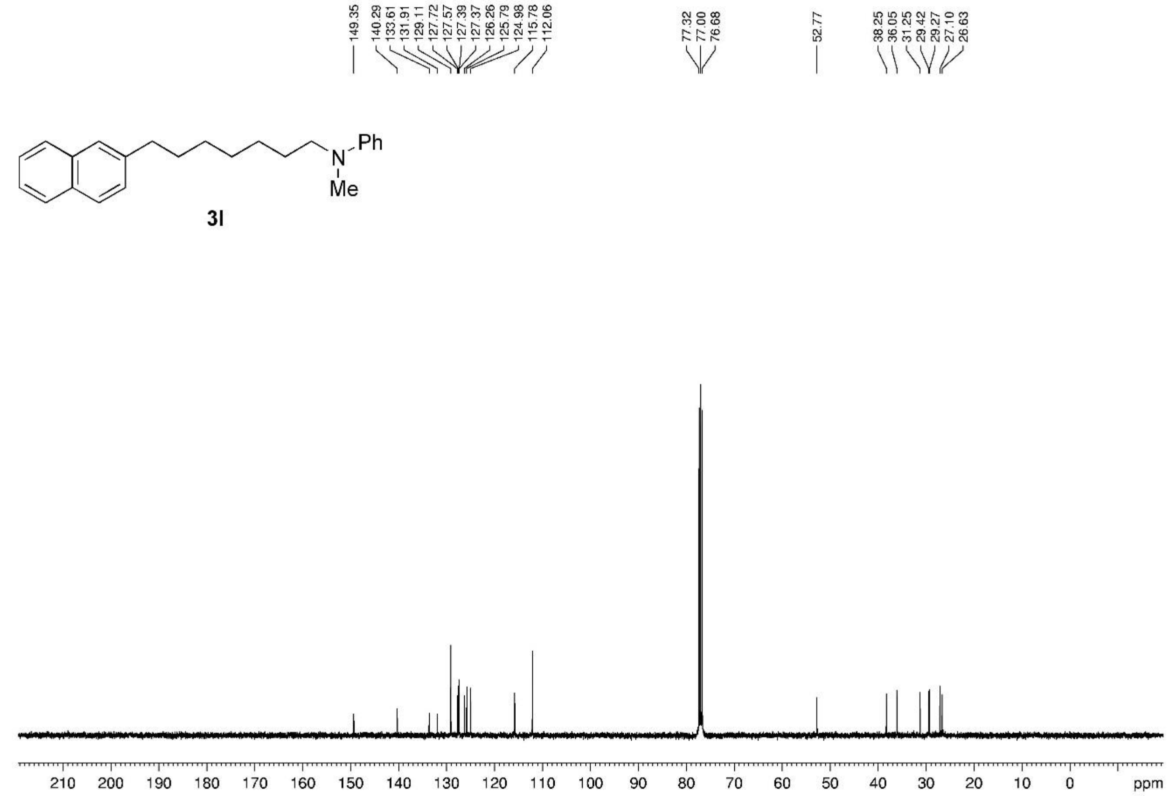
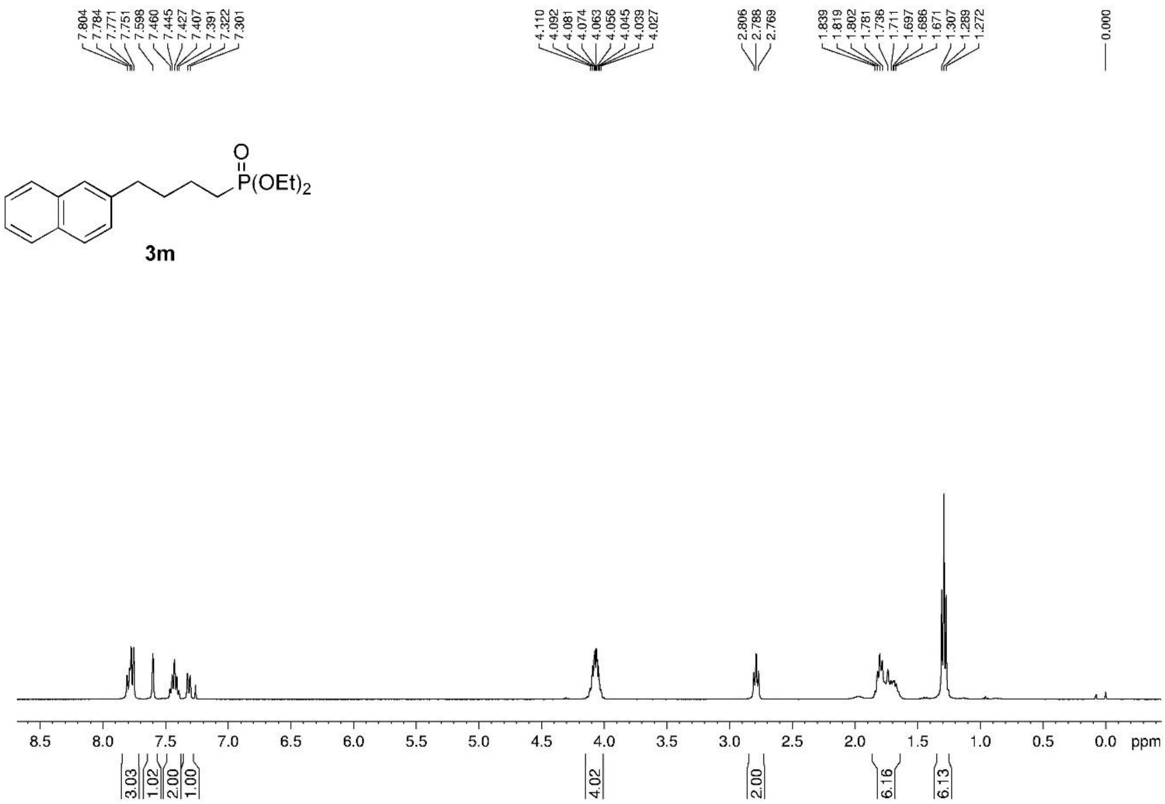
1.02
1.97
1.02
2.04
2.96
2.00
3.01
2.01
2.10
2.59
6.30



3l

149.35
140.29
133.61
131.91
129.11
127.72
127.57
127.39
127.37
126.26
125.79
124.98
115.78
112.06
77.32
77.00
76.68
52.77
38.25
36.05
31.25
29.42
29.27
27.10
26.63
N
Ph
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



3m

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7.598
7.460
7.445
7.427
7.407
7.391
7.322
7.301
4.110
4.092
4.081
4.074
4.063
4.056
4.045
4.039
4.027
2.806
2.788
2.769
1.839
1.819
1.802
1.781
1.736
1.711
1.697
1.686
1.671
1.307
1.289
1.272
0.000
O
P(OEt)2
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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0.0
ppm
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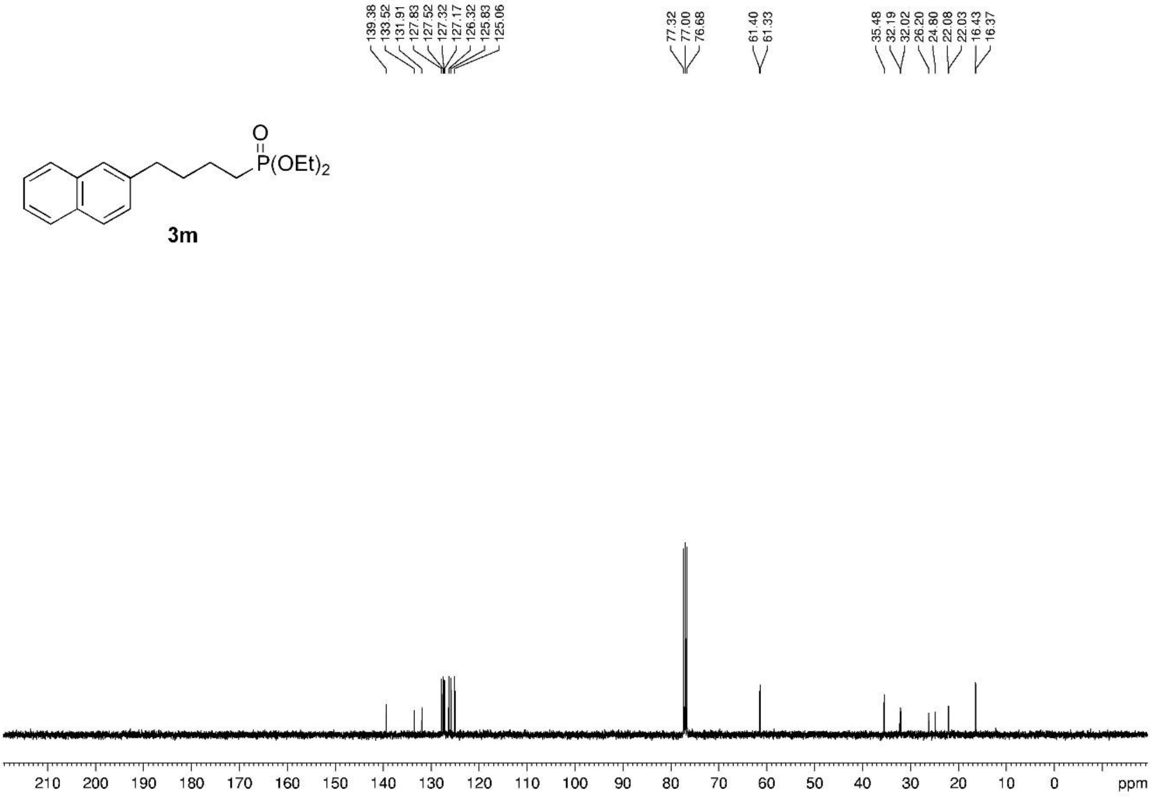
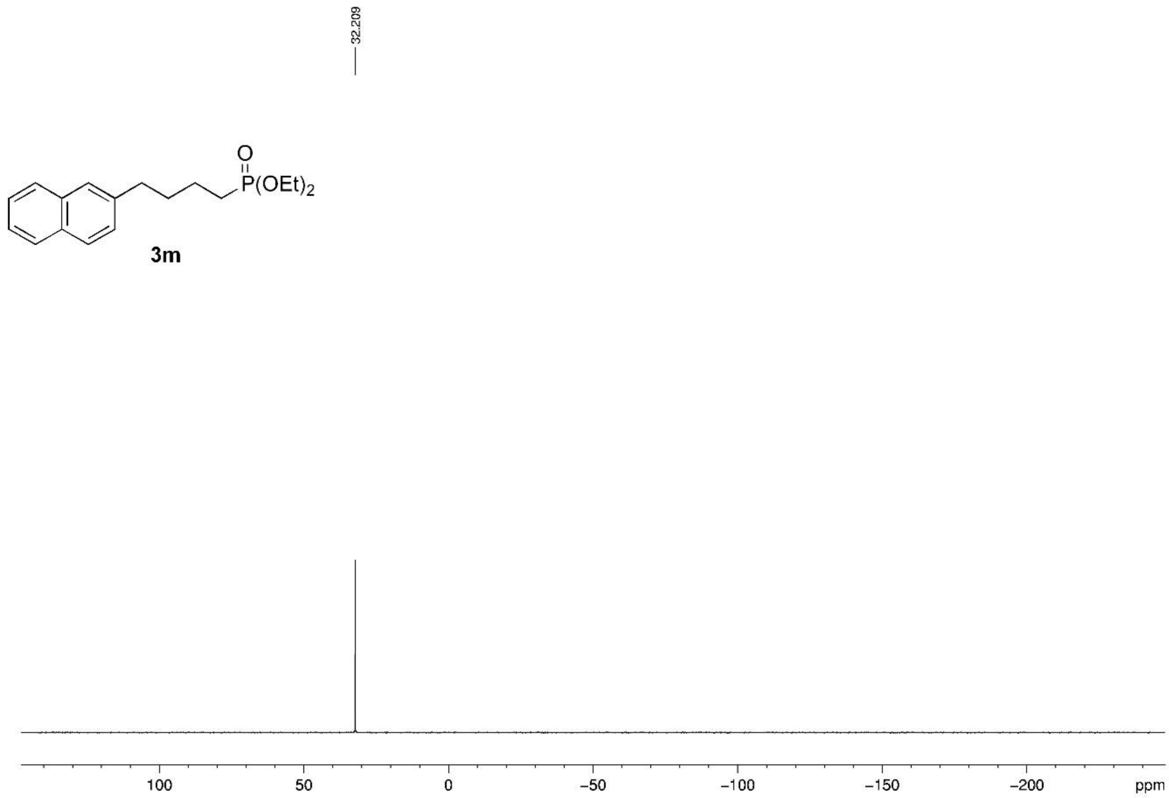
1.02
2.00
1.00
4.02
2.00
6.16
6.13

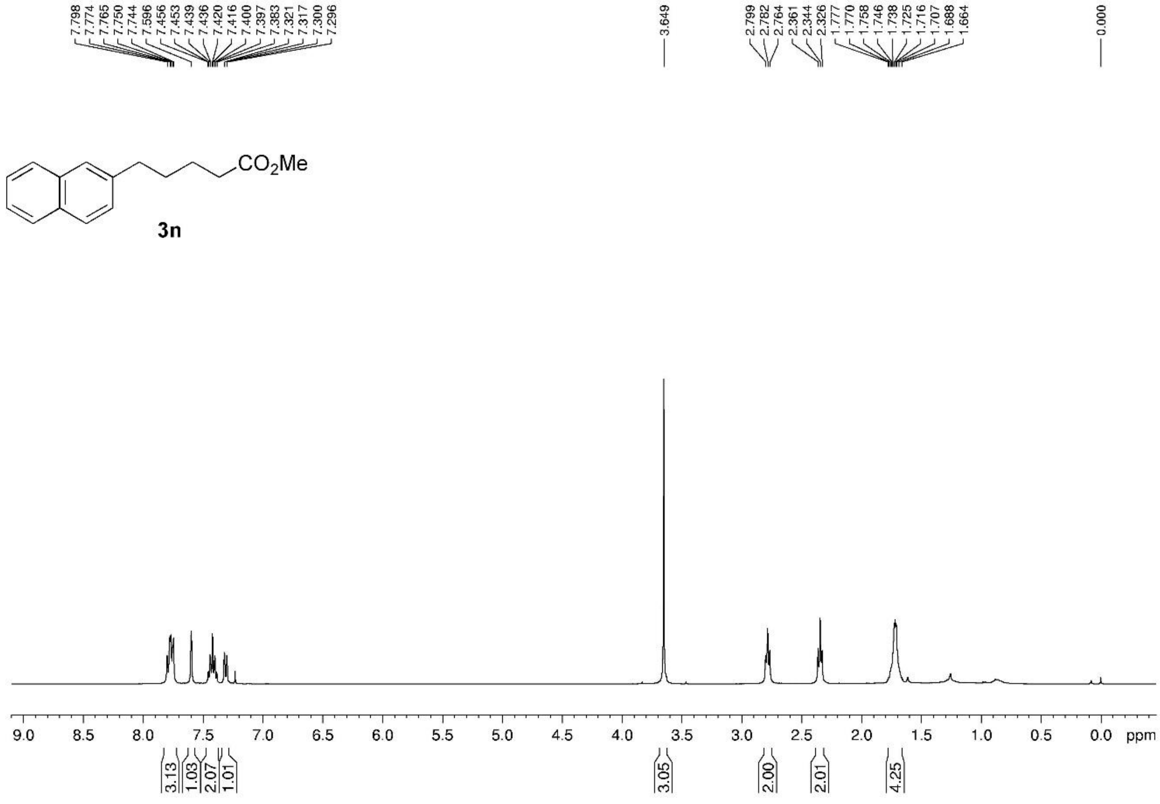


3m

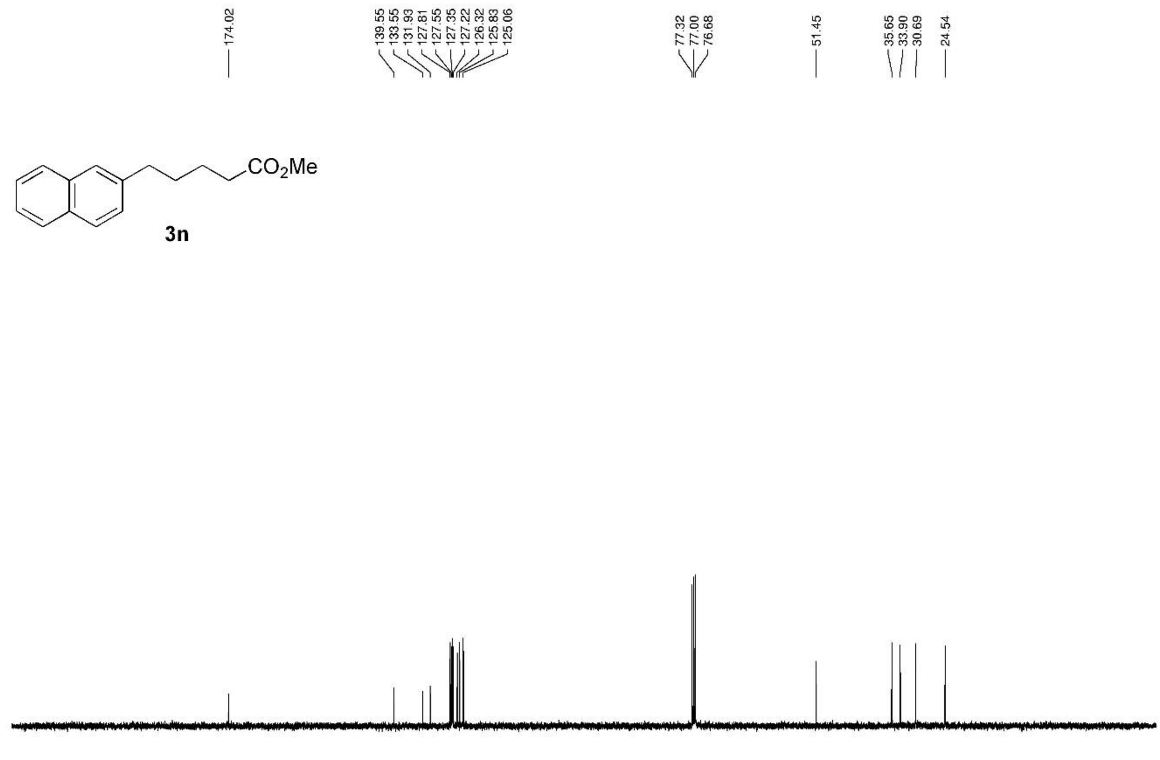
139.38
133.52
131.91
127.83
127.52
127.32
127.17
126.32
125.83
125.06
77.32
77.00
76.68
61.40
61.33
35.48
32.19
32.02
26.20
24.80
22.08
22.03
16.43
16.37
O
P(OEt)2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



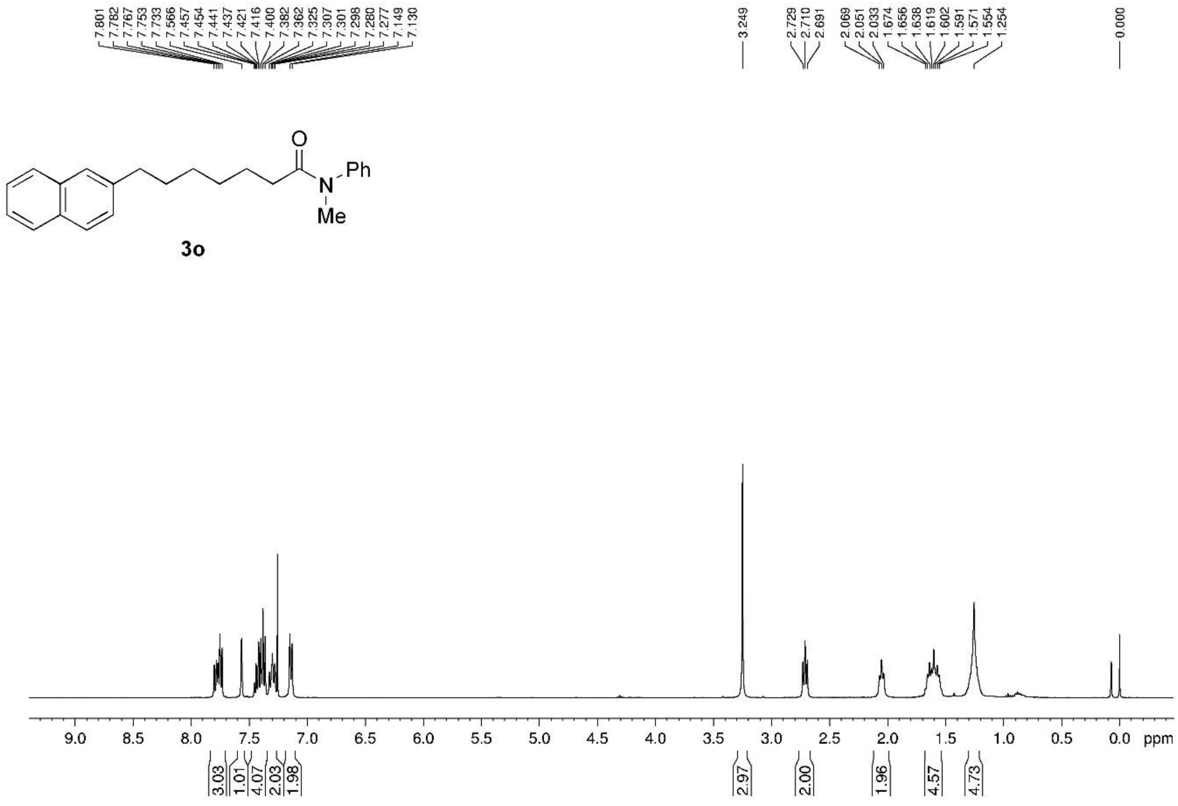
3m


3n

7.798
7.774
7.765
7.750
7.744
7.596
7.456
7.453
7.439
7.436
7.420
7.416
7.400
7.397
7.383
7.321
7.317
7.300
7.296
3.649
2.799
2.782
2.764
2.361
2.344
2.326
1.777
1.770
1.758
1.746
1.738
1.725
1.716
1.707
1.688
1.664
0.000
CO$_2$Me
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.13
1.03
2.07
1.01
3.05
2.00
2.01
4.25


3n

174.02
139.55
133.55
131.93
127.81
127.55
127.35
127.22
126.32
125.83
125.06
77.32
77.00
76.68
51.45
35.65
33.90
30.69
24.54
CO$_2$Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.566
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7.454
7.441
7.437
7.421
7.416
7.400
7.382
7.362
7.325
7.307
7.301
7.298
7.280
7.277
7.149
7.130
3.249
2.729
2.710
2.691
2.069
2.051
2.033
1.674
1.656
1.638
1.619
1.602
1.591
1.571
1.554
1.254
0.000
O
N
Ph
Me
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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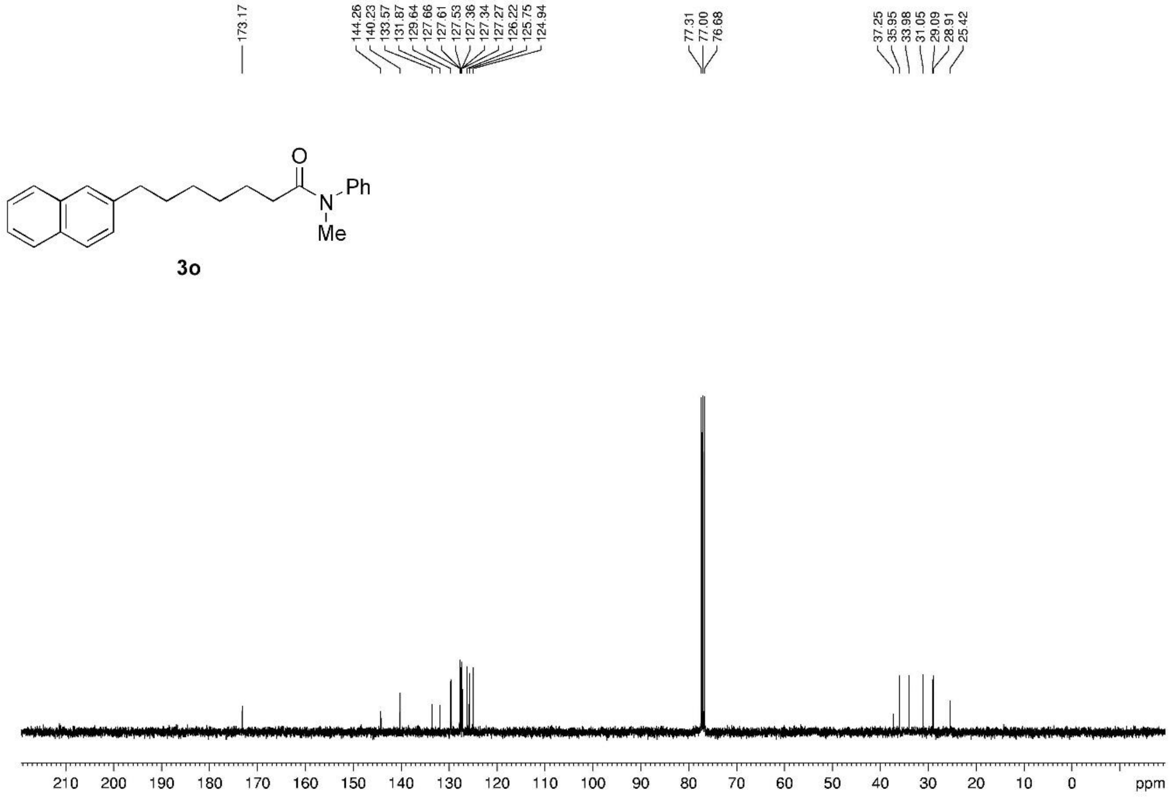
1.01
4.07
2.03
1.98
2.97
2.00
1.96
4.57
4.73

3o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144.26
140.23
133.57
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129.64
127.66
127.61
127.53
127.36
127.34
127.27
126.22
125.75
124.94
77.31
77.00
76.68
37.25
35.95
33.98
31.05
29.09
28.91
25.42
O
N
Ph
Me
210
200
190
180
170
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150
140
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120
110
100
90
80
70
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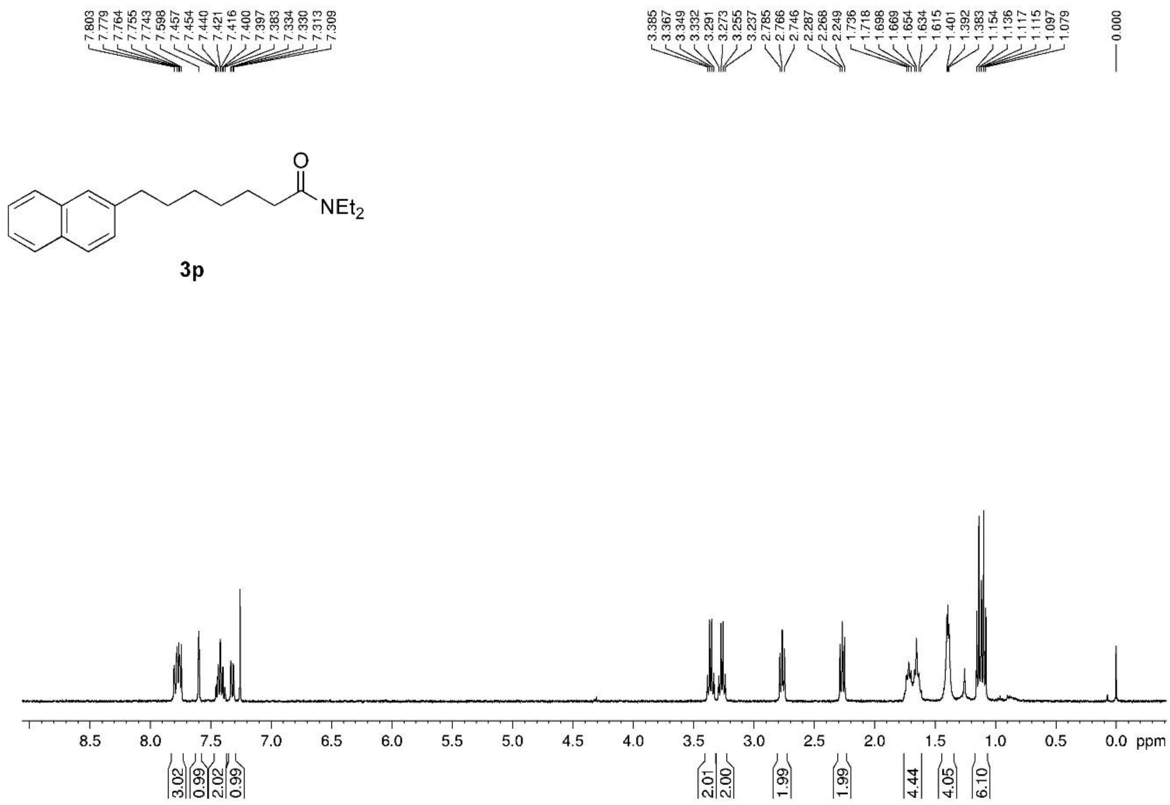
50
40
30
20
10
0
ppm

3o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7.743
7.598
7.457
7.454
7.440
7.421
7.416
7.400
7.397
7.383
7.334
7.330
7.313
7.309
3.385
3.367
3.349
3.332
3.291
3.273
3.255
3.237
2.785
2.766
2.746
2.287
2.268
2.249
1.736
1.718
1.698
1.669
1.654
1.634
1.615
1.401
1.392
1.383
1.154
1.136
1.117
1.115
1.097
1.079
0.000
O
NEt2
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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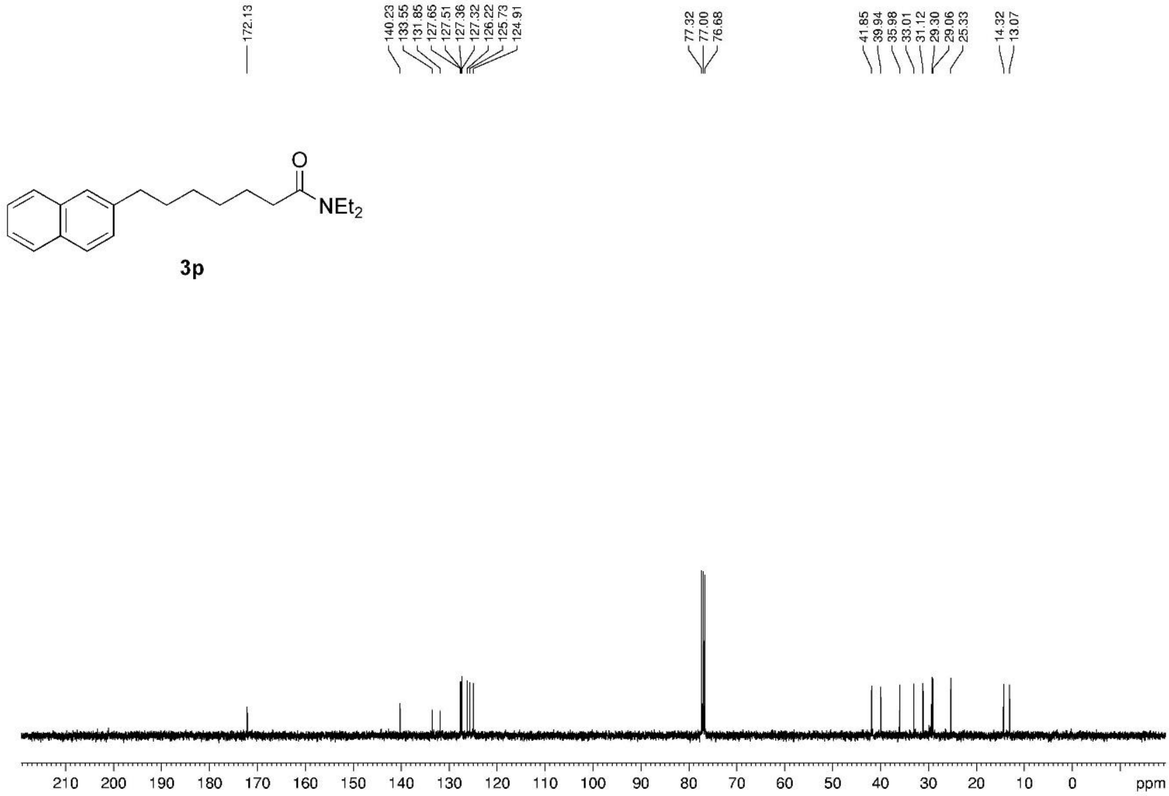
0.99
2.02
0.99
2.01
2.00
1.99
1.99
4.44
4.05
6.10


3p
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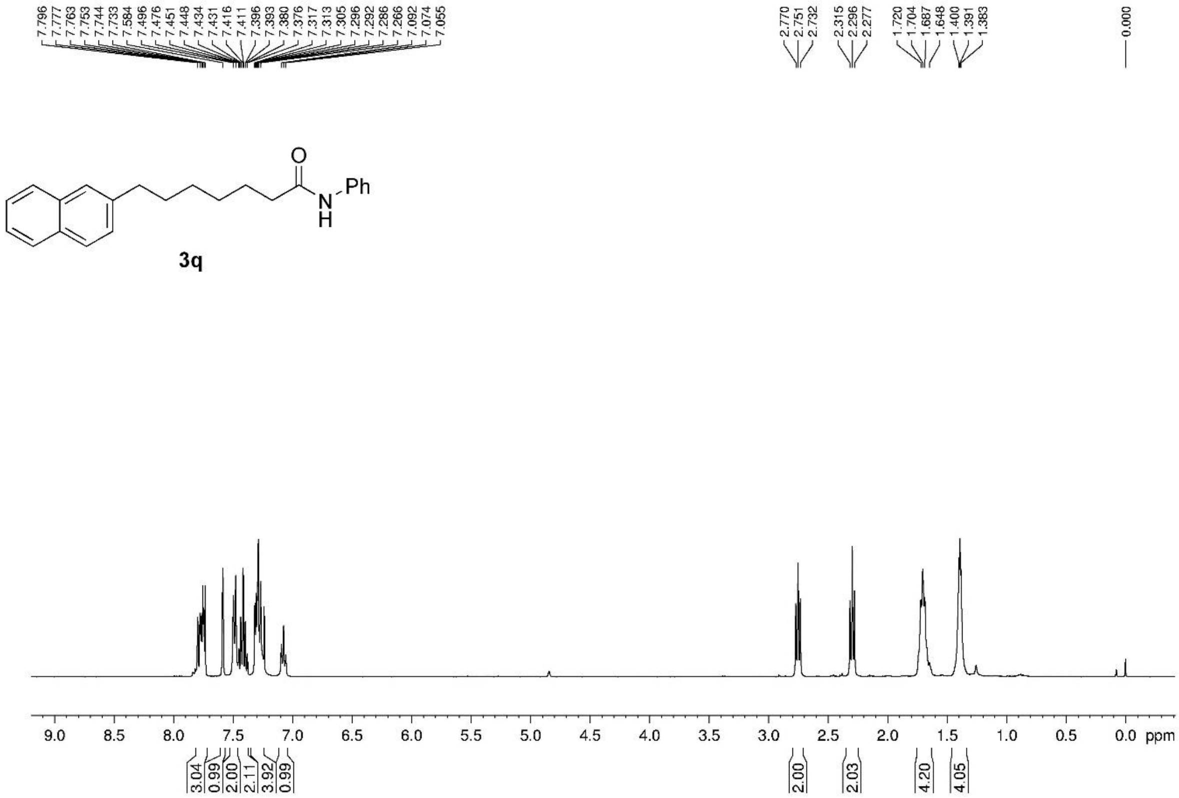
133.55
131.85
127.65
127.51
127.36
127.32
126.22
125.73
124.91
77.32
77.00
76.68
41.85
39.94
35.98
33.01
31.12
29.30
29.06
25.33
14.32
13.07
O
NEt2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.744
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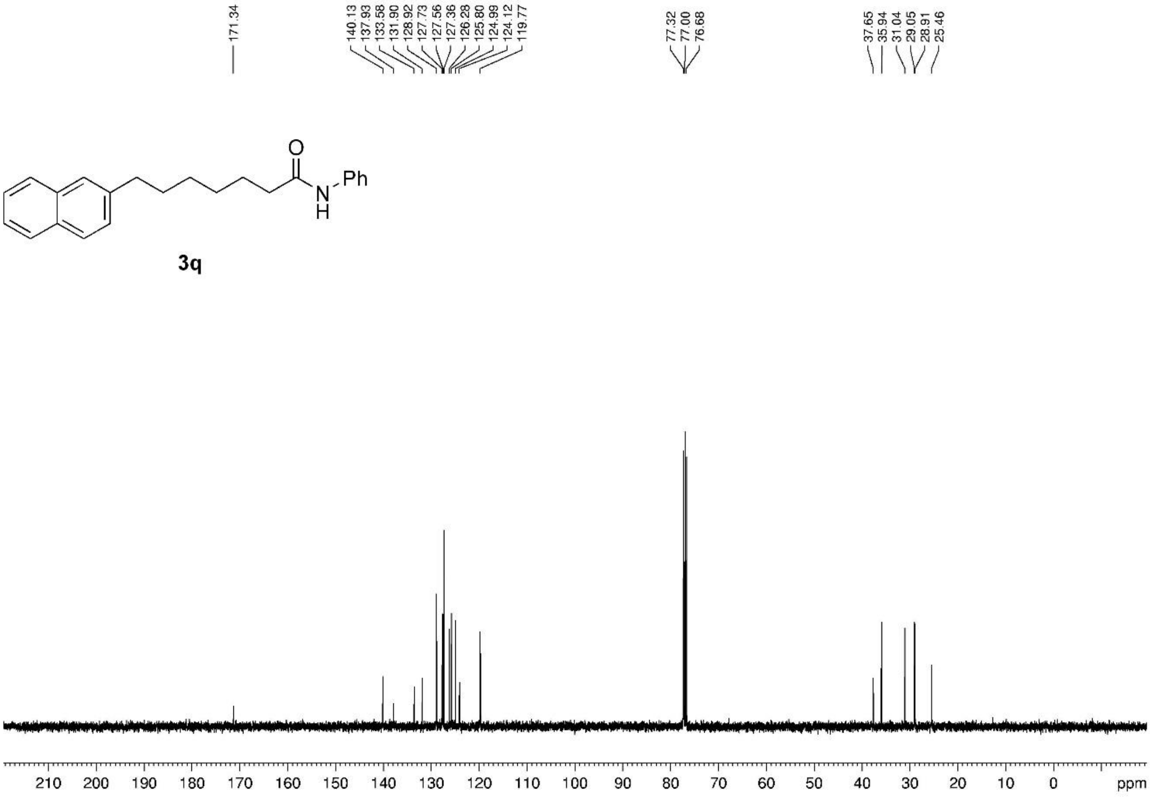
7.448
7.434
7.431
7.416
7.411
7.396
7.393
7.380
7.376
7.317
7.313
7.305
7.296
7.292
7.286
7.266
7.092
7.074
7.055
2.770
2.751
2.732
2.315
2.296
2.277
1.720
1.704
1.687
1.648
1.400
1.391
1.383
0.000
O
N
H
Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.04
0.99
2.00
2.11
3.92
0.99
2.00
2.03
4.20
4.05


3q
171.34
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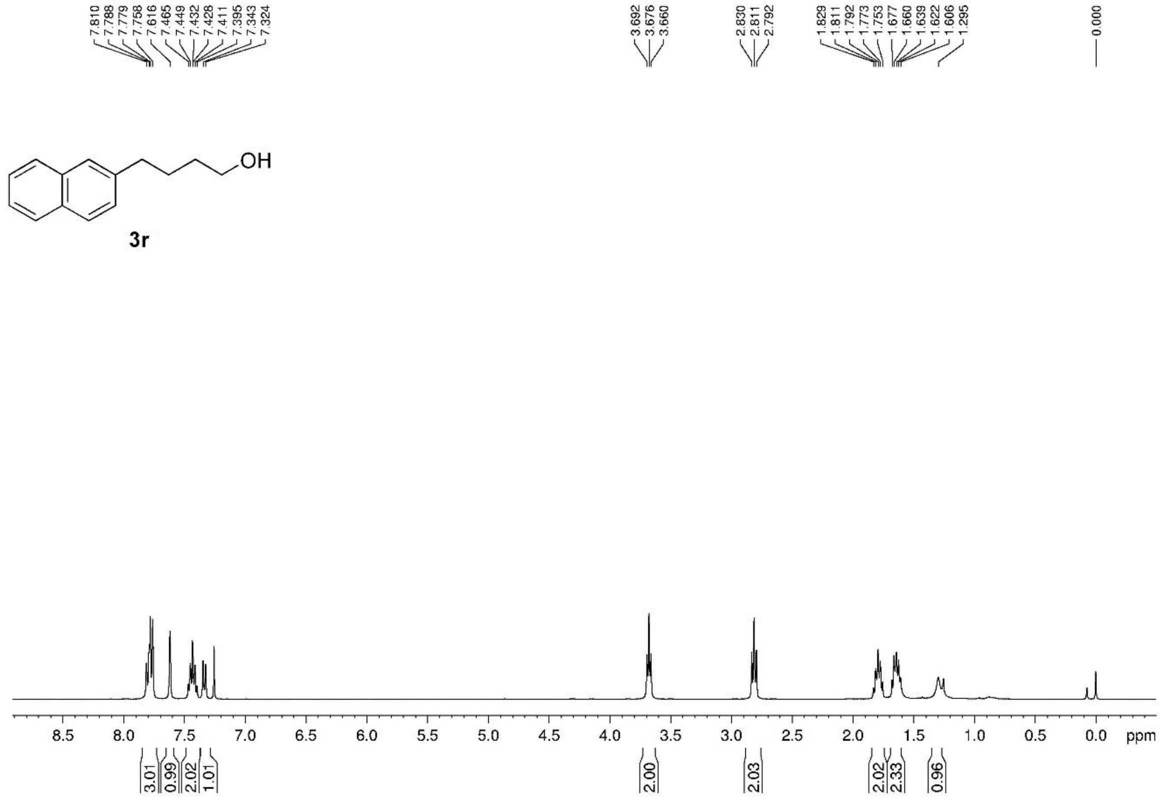
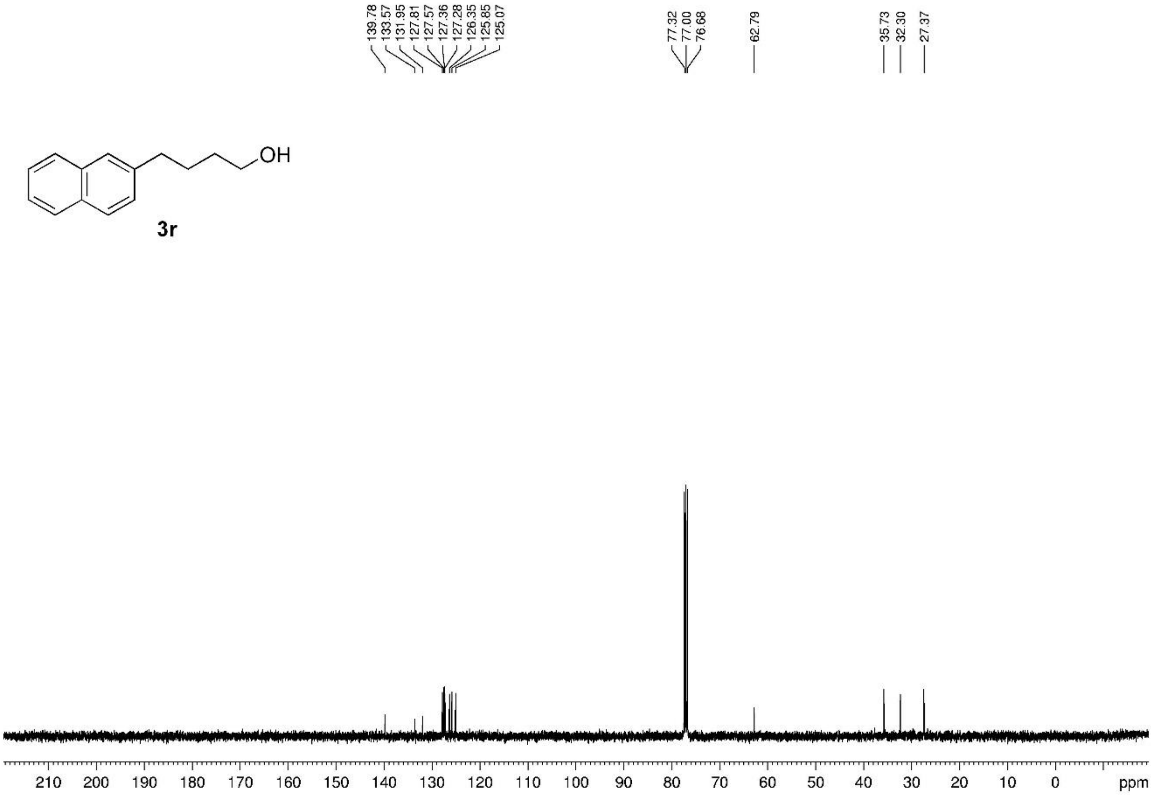
137.93
133.58
131.90
128.92
127.73
127.56
127.36
126.28
125.80
124.99
124.12
119.77
77.32
77.00
76.68
37.65
35.94
31.04
29.05
28.91
25.46
O
N
H
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


3r
7.810
7.788
7.779
7.758
7.616
7.465
7.449
7.432
7.428
7.411
7.395
7.343
7.324
3.692
3.676
3.660
2.830
2.811
2.792
1.829
1.811
1.792
1.773
1.753
1.677
1.660
1.639
1.622
1.606
1.295
0.000
OH
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.01
0.99
2.02
1.01
2.00
2.03
2.02
2.33
0.96


3r
139.78
133.57
131.95
127.81
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126.35
125.85
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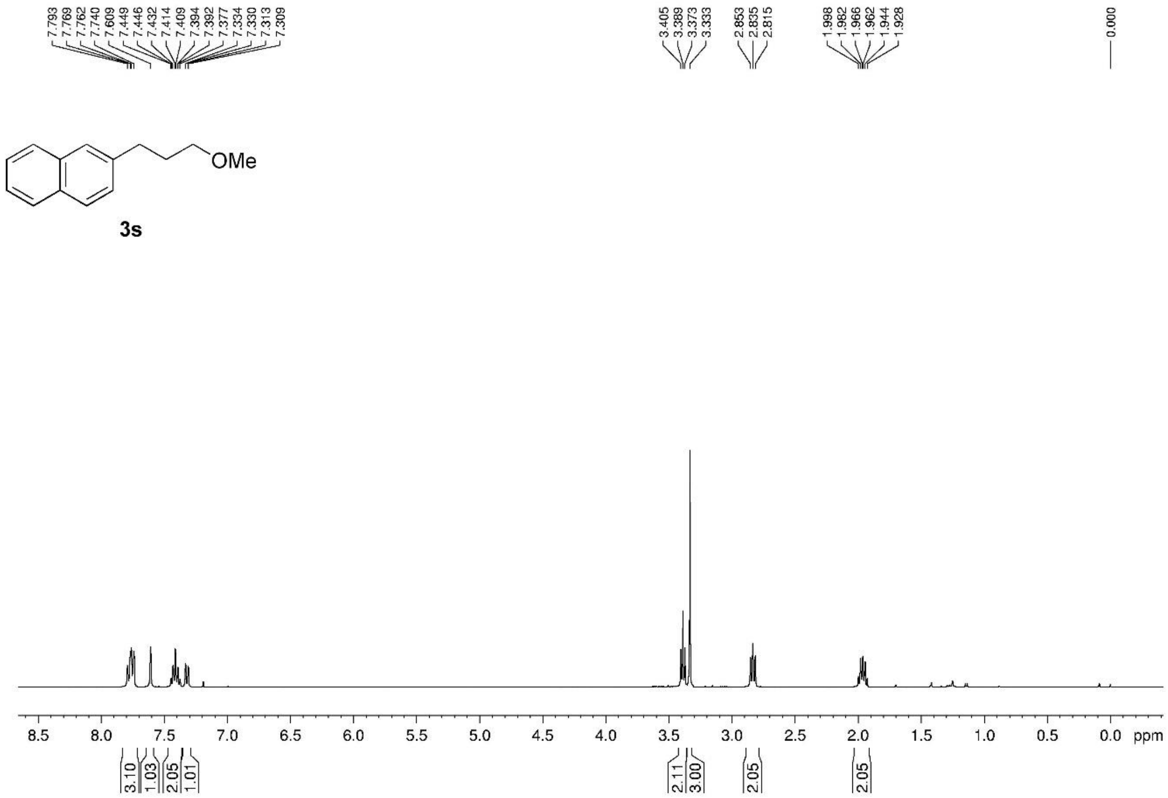
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7.313
7.309
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3.373
3.333
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2.835
2.815
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1.982
1.966
1.962
1.944
1.928
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OMe
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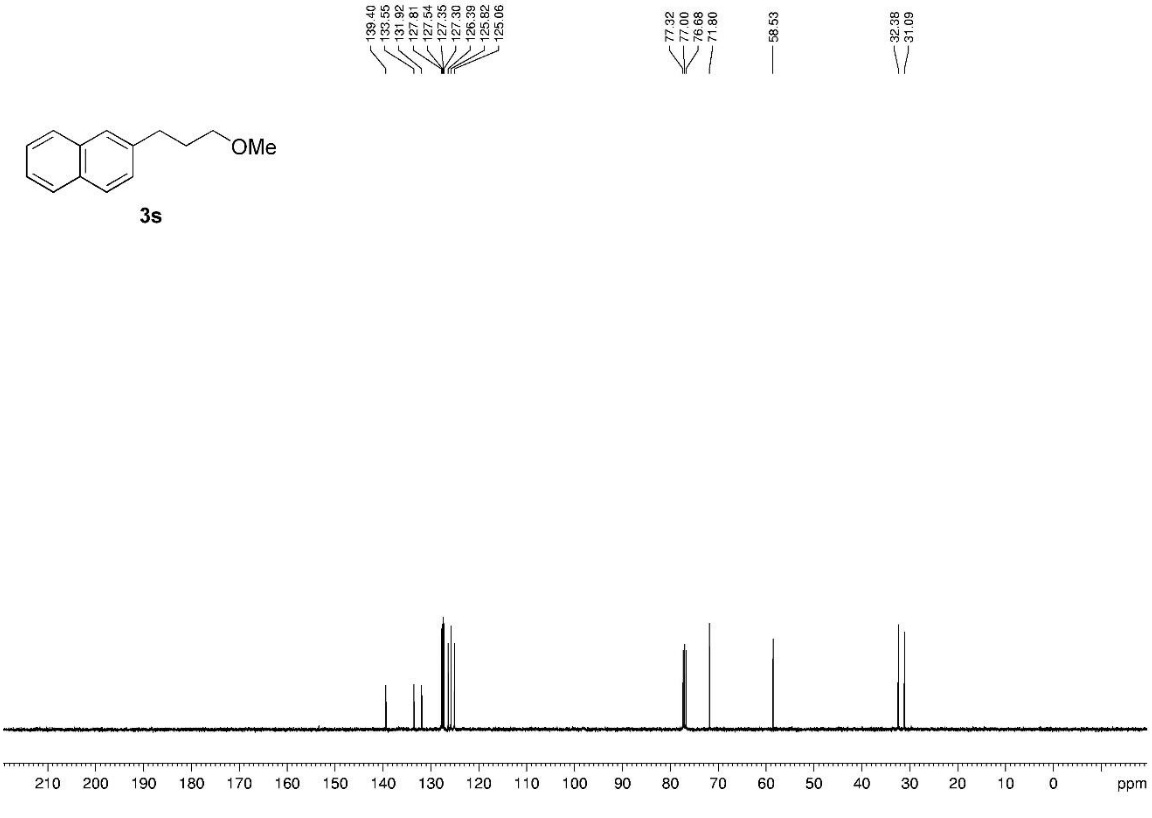
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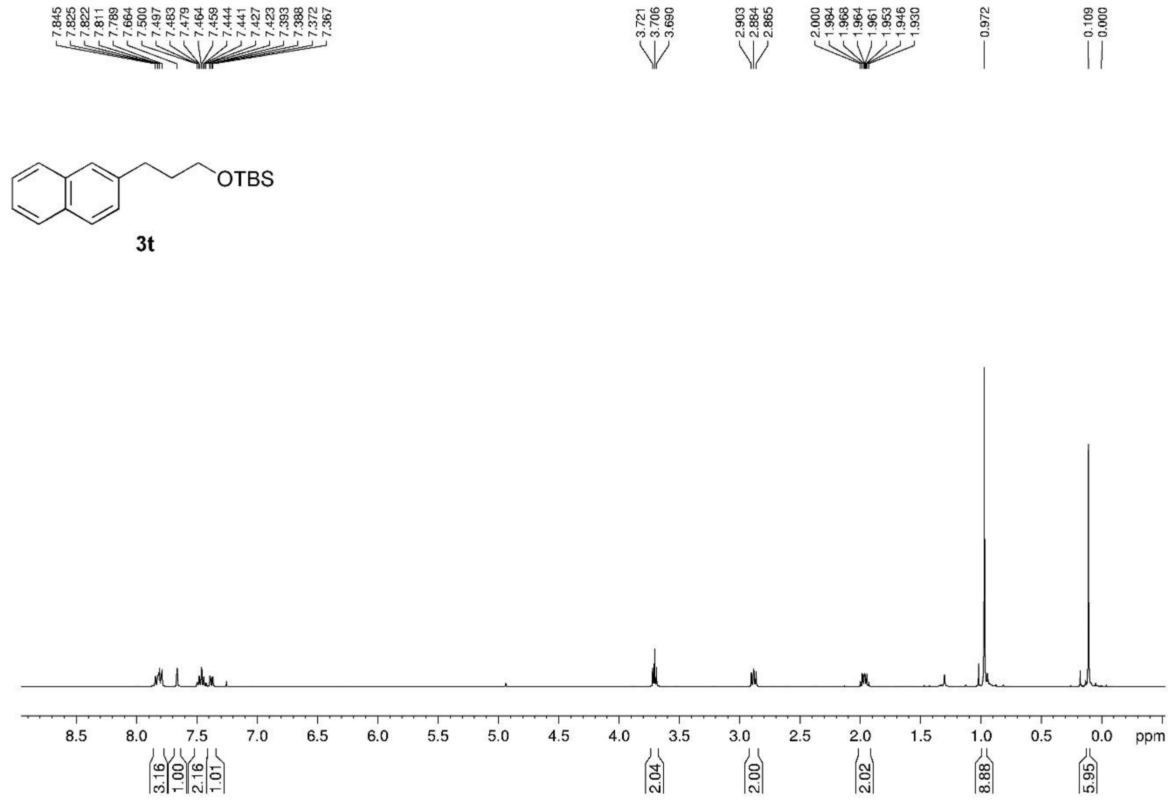
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7.441
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7.423
7.393
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3.690
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OTBS
3t
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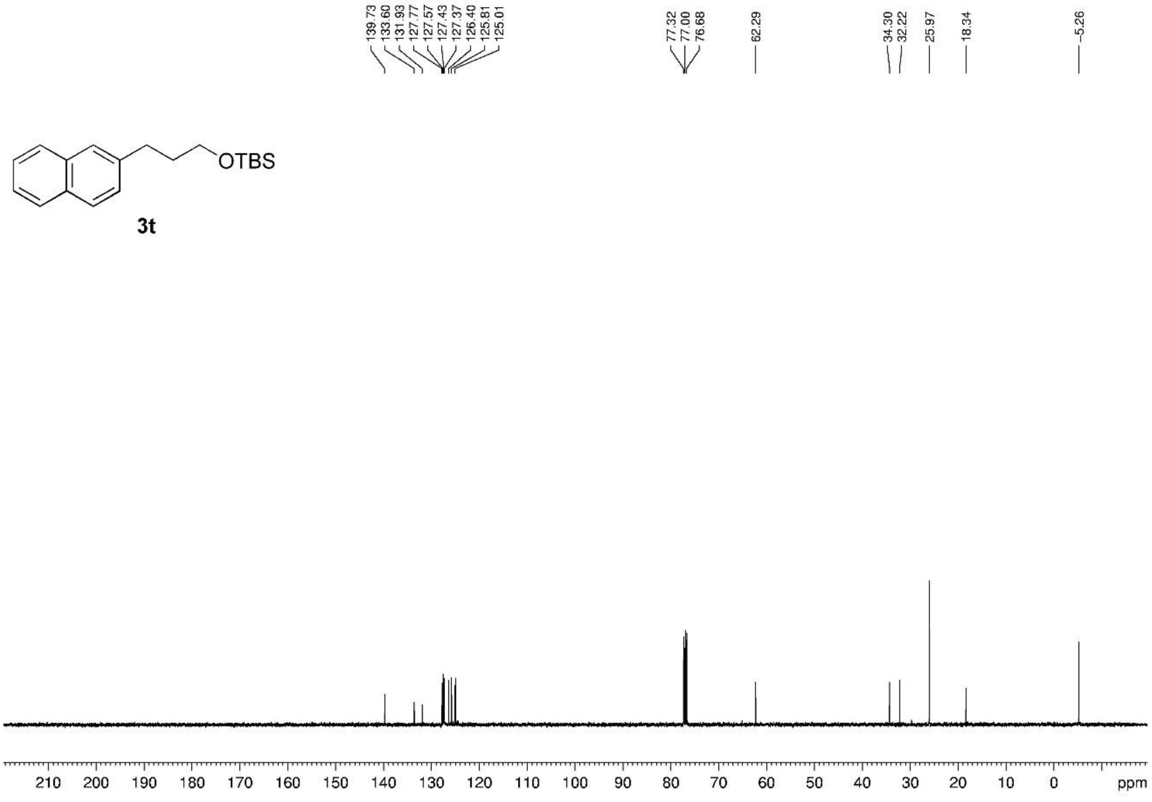
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3t
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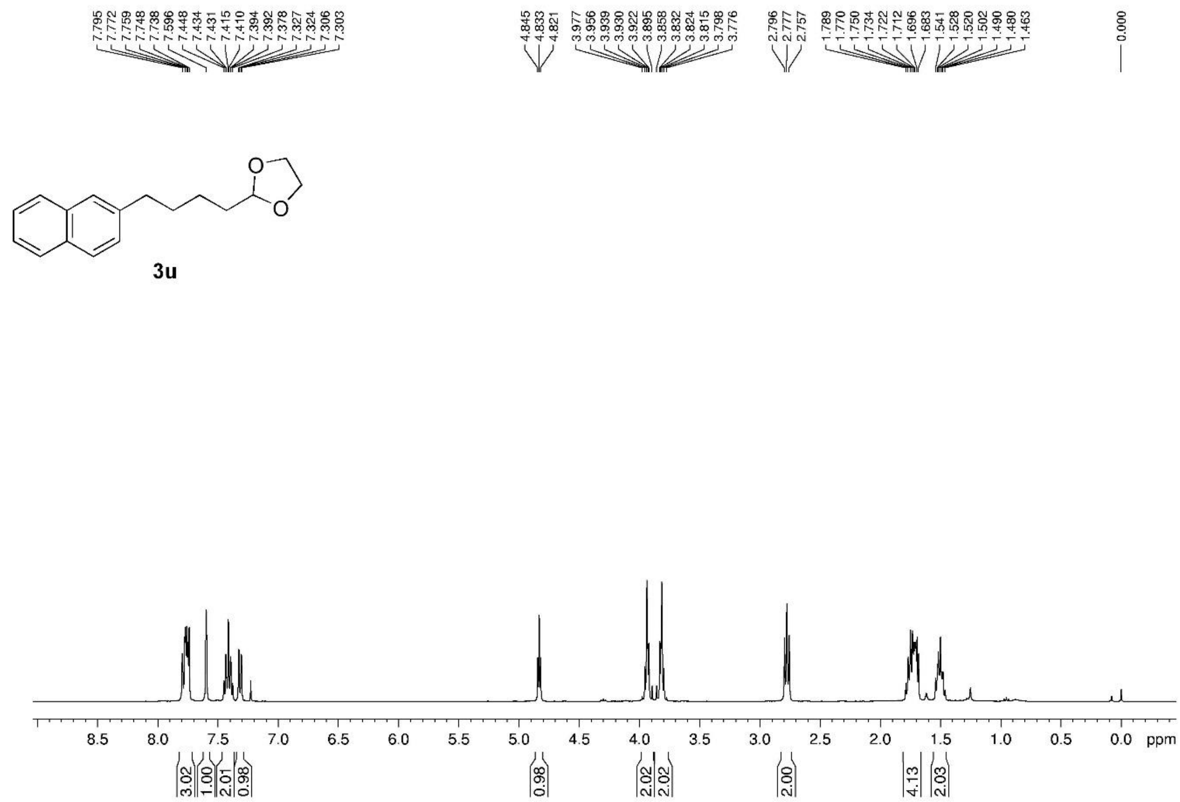
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1.520
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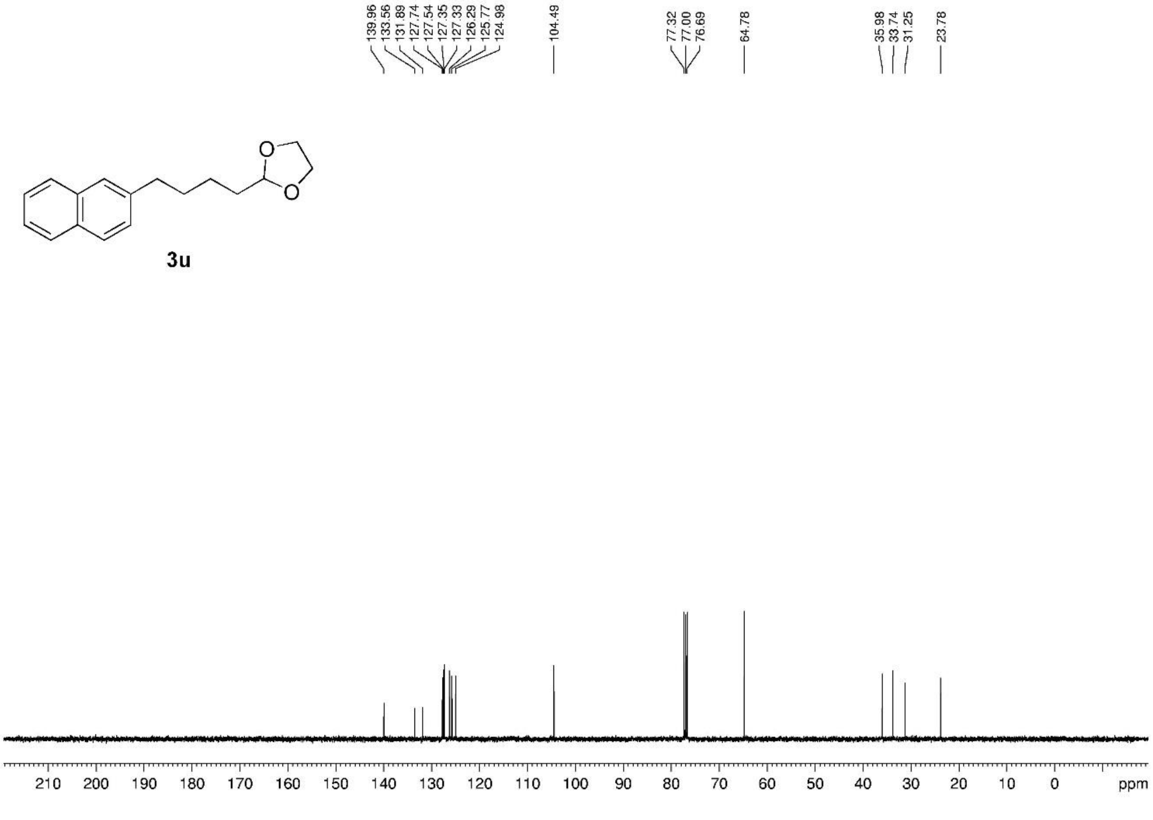
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64.78
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33.74
31.25
23.78
3u
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200
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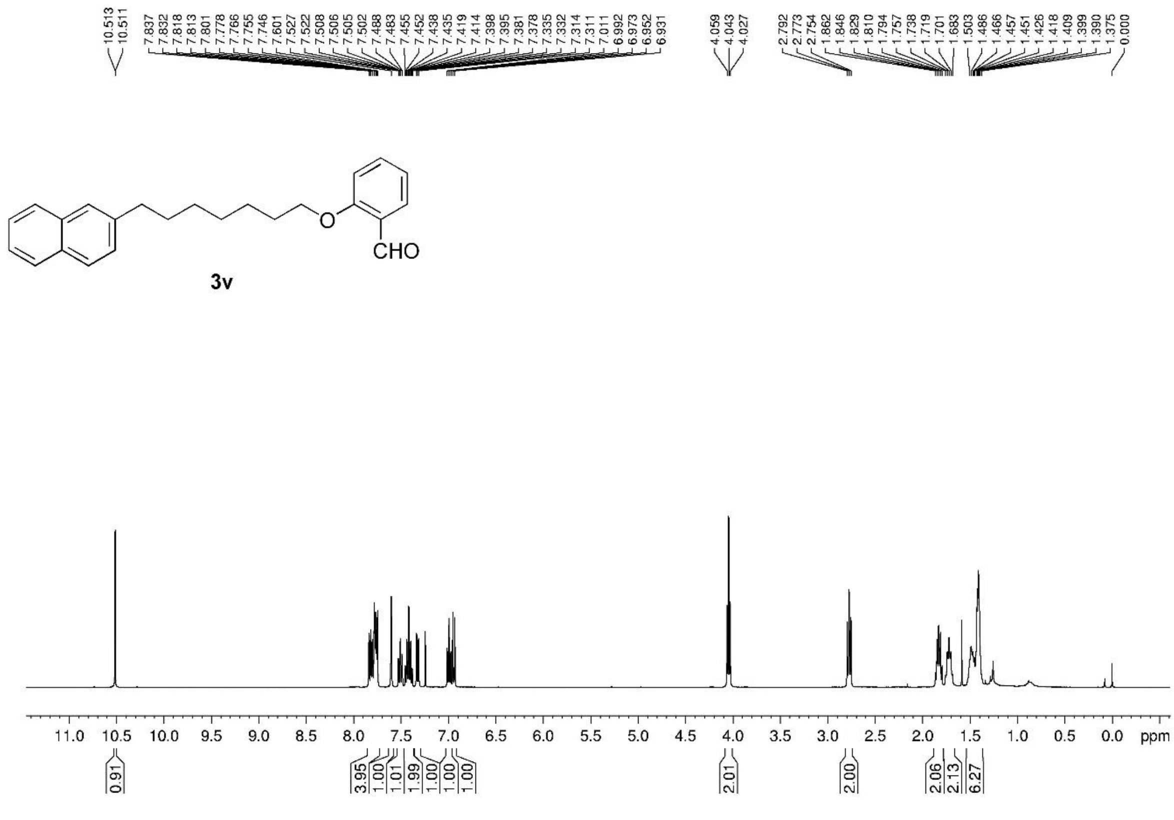
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7.522
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7.506
7.505
7.502
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7.435
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4.027
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1.757
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1.683
1.503
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1.466
1.457
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1.418
1.409
1.399
1.390
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O
CHO
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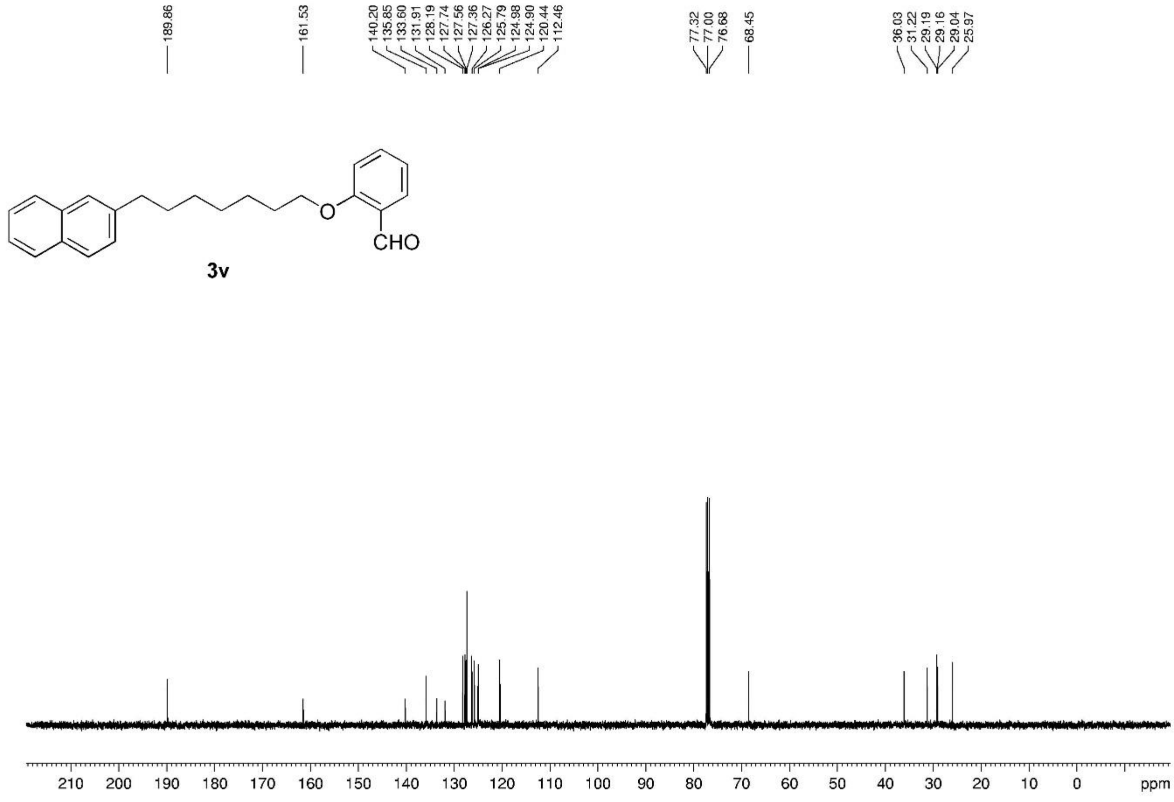
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120.44
112.46
77.32
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68.45
36.03
31.22
29.19
29.16
29.04
25.97
O
CHO
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200
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180
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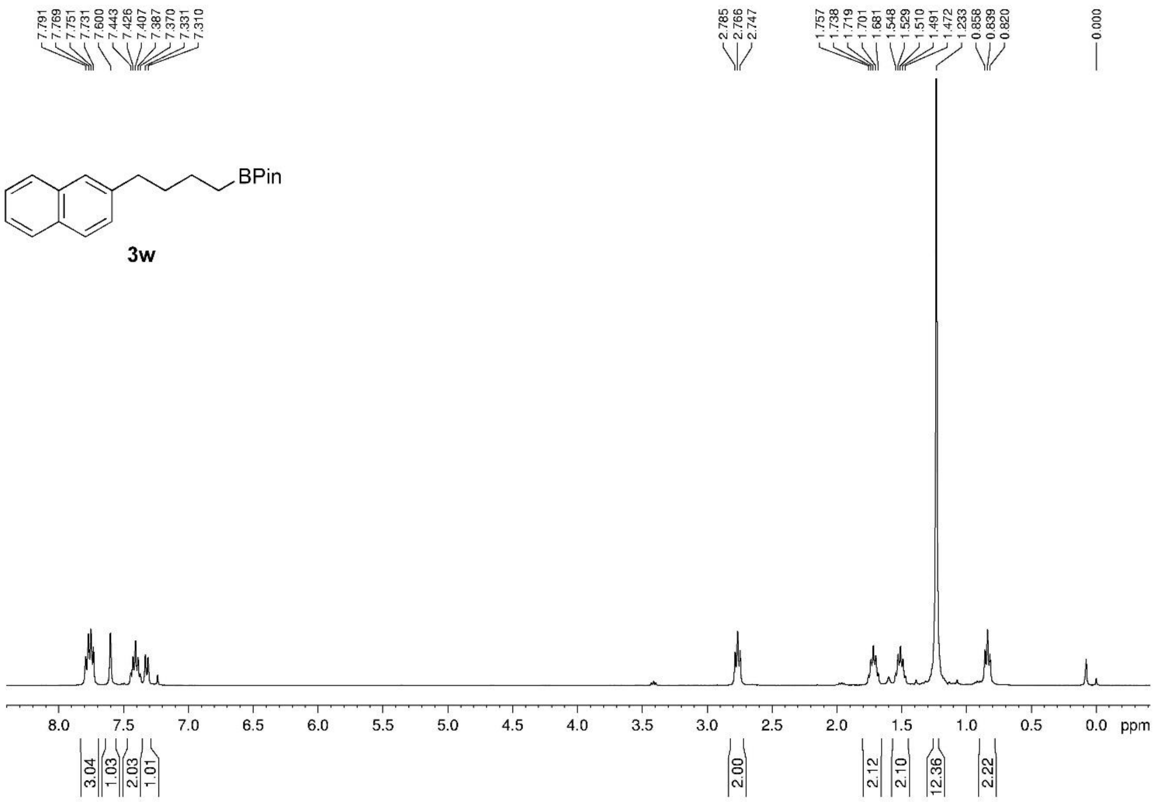
ppm

3v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2.766
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1.738
1.719
1.701
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1.548
1.529
1.510
1.491
1.472
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0.839
0.820
0.000
BPin
3w
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5.5
5.0
4.5
4.0
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2.0
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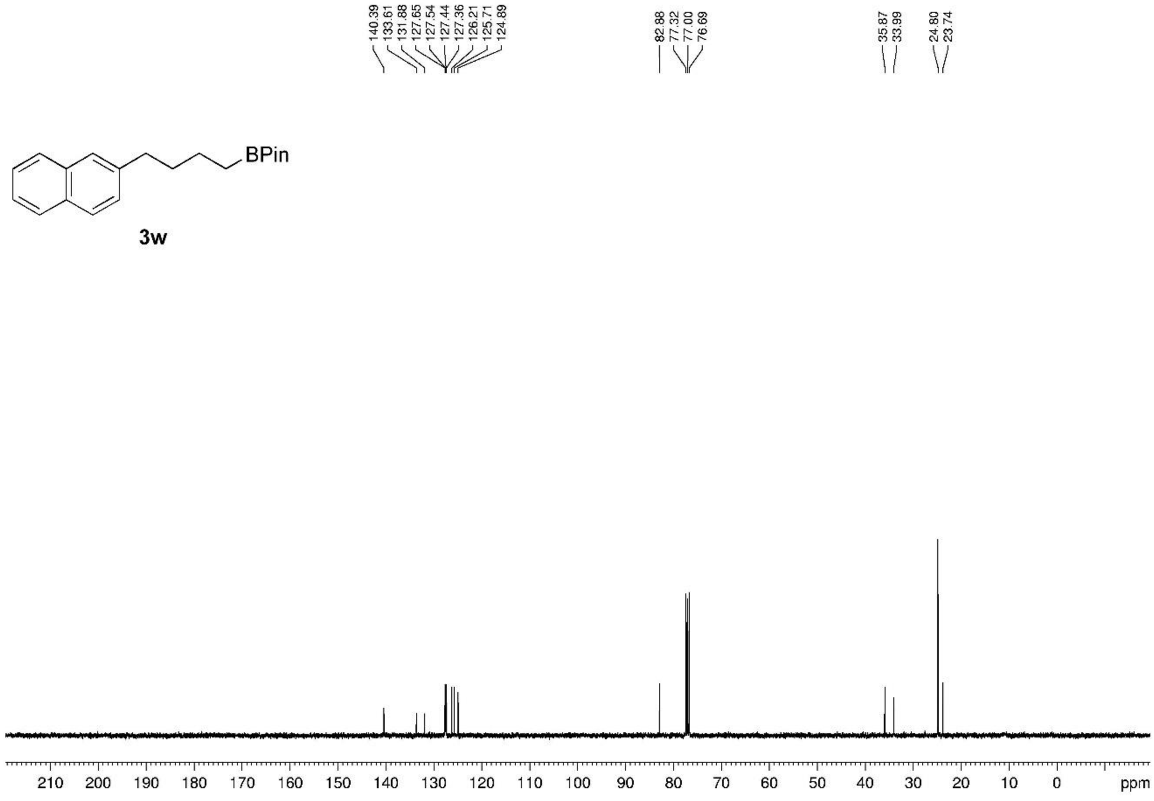
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35.87
33.99
24.80
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BPin
3w
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200
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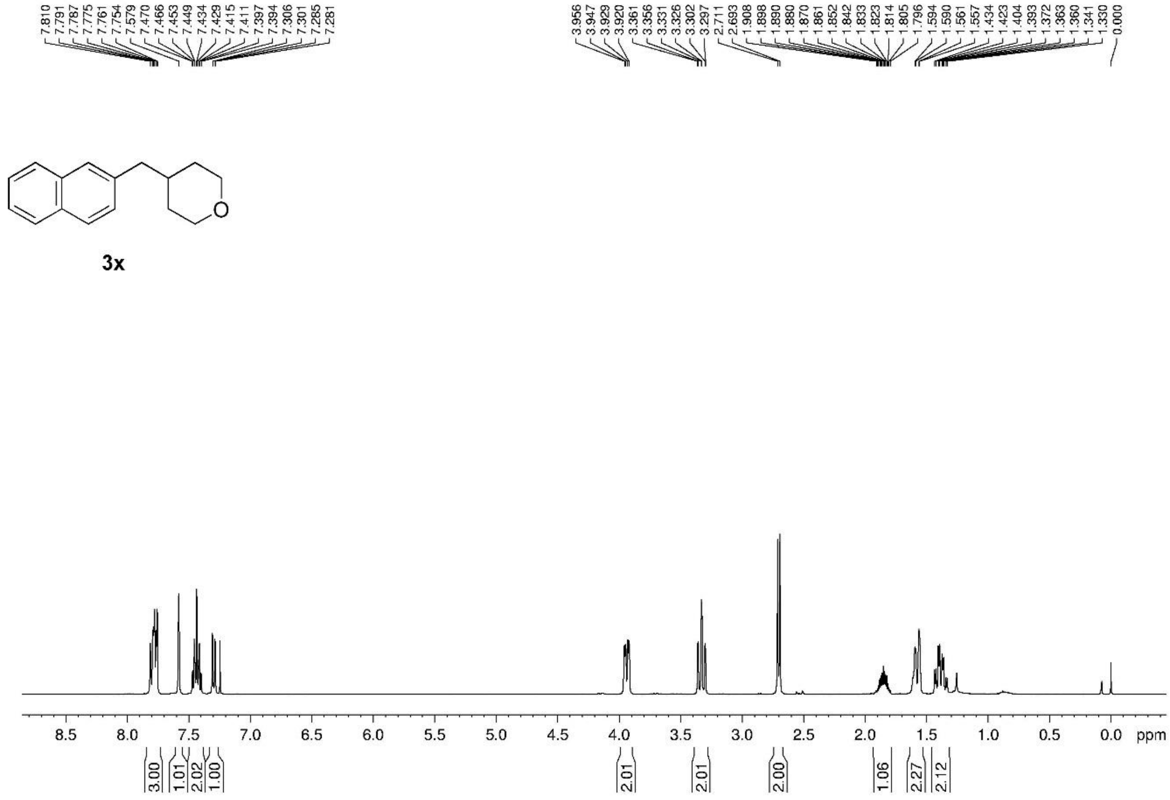
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1.557
1.434
1.423
1.404
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1.372
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1.330
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2.0
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2.27
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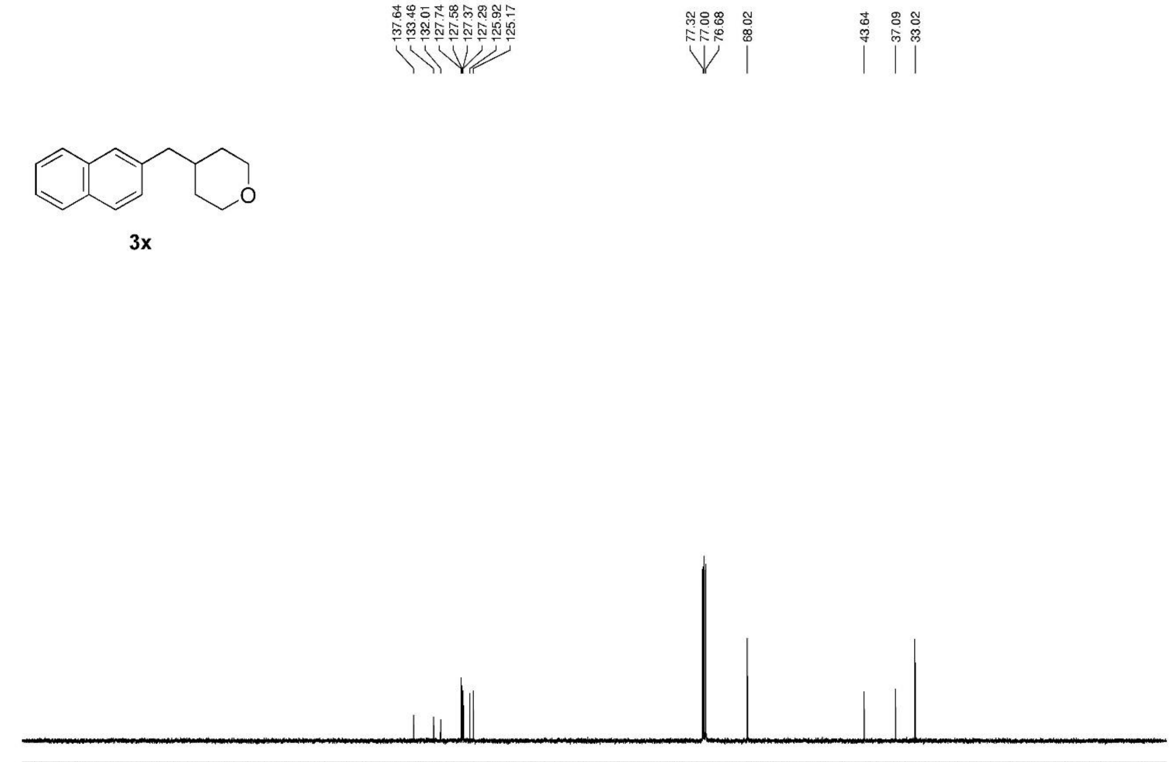
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68.02
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37.09
33.02
O
3x
210
200
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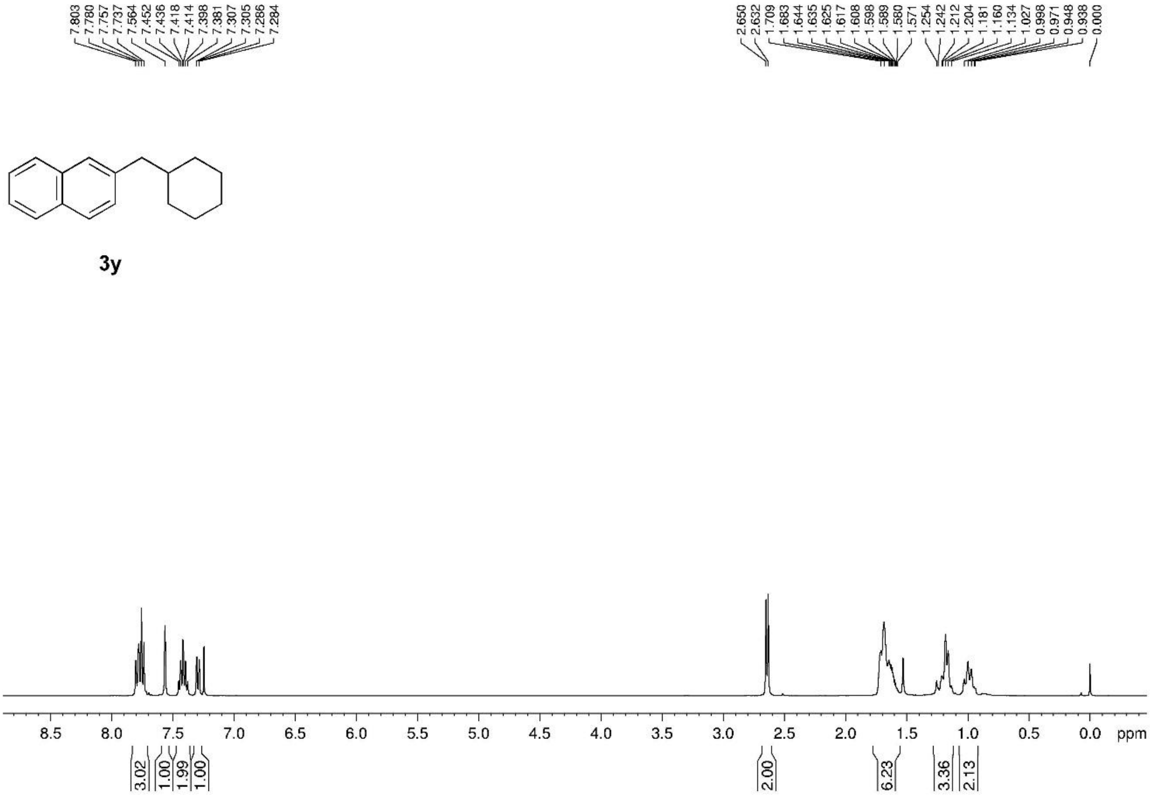
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1.580
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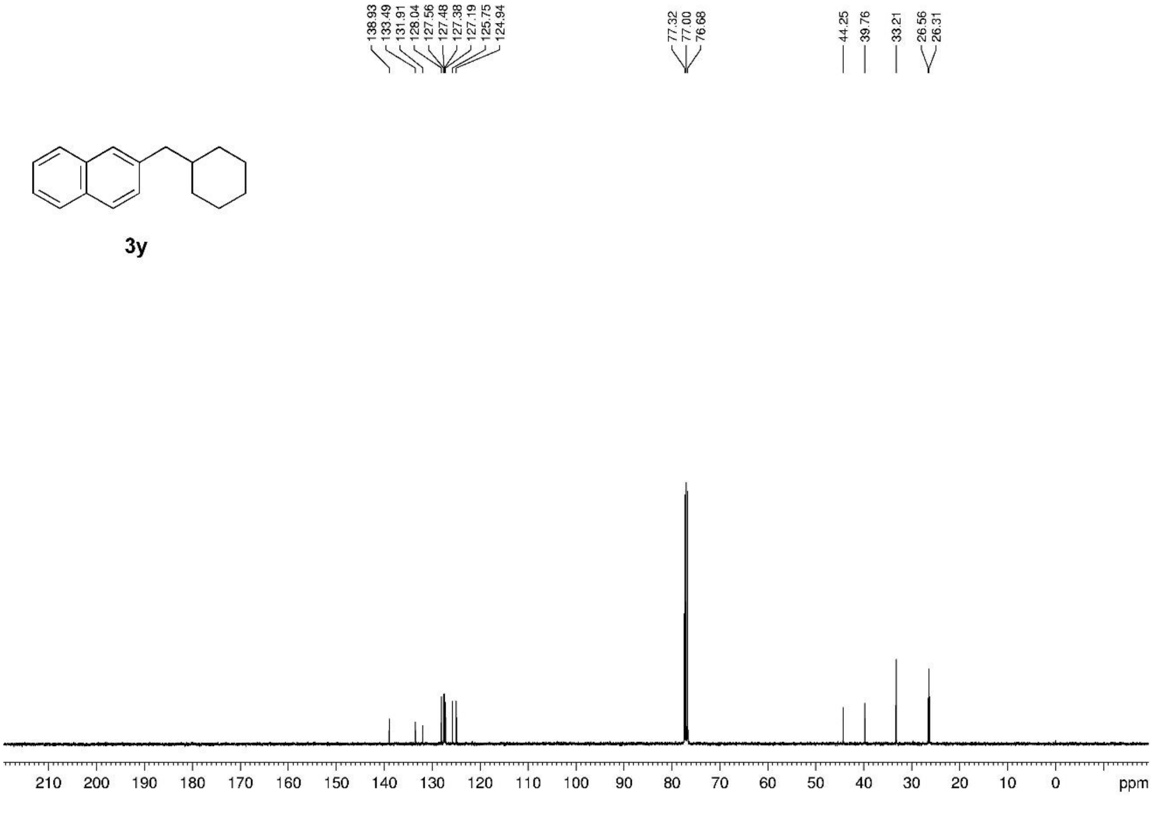
3y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200
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180
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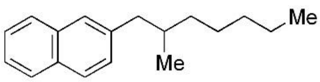
3y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Me

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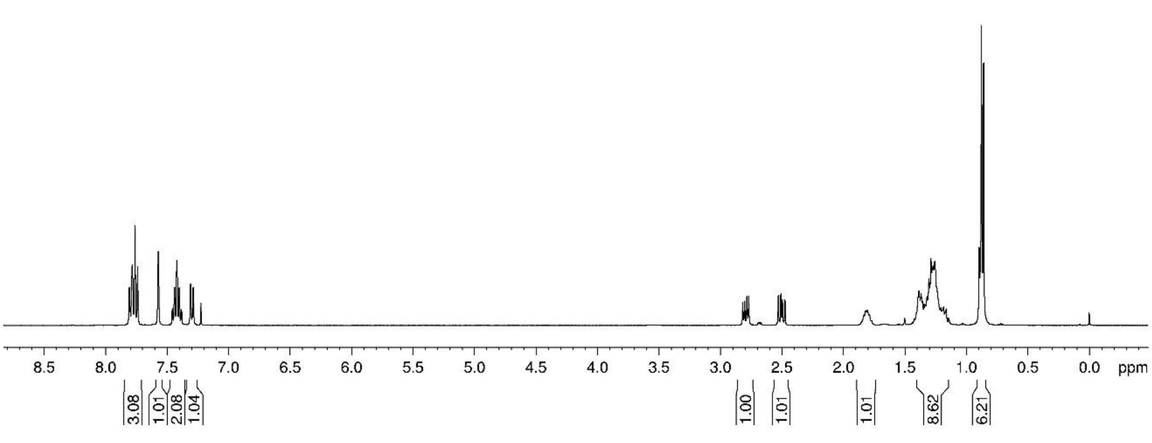
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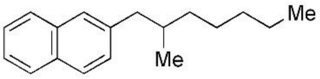
6.21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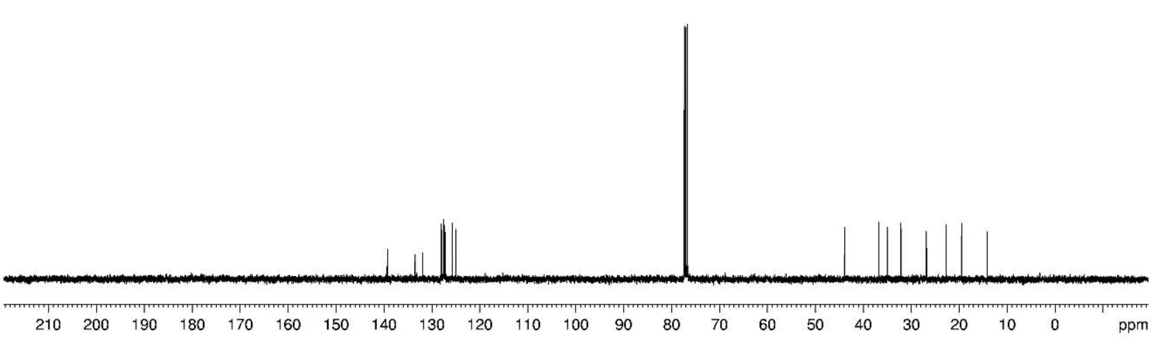
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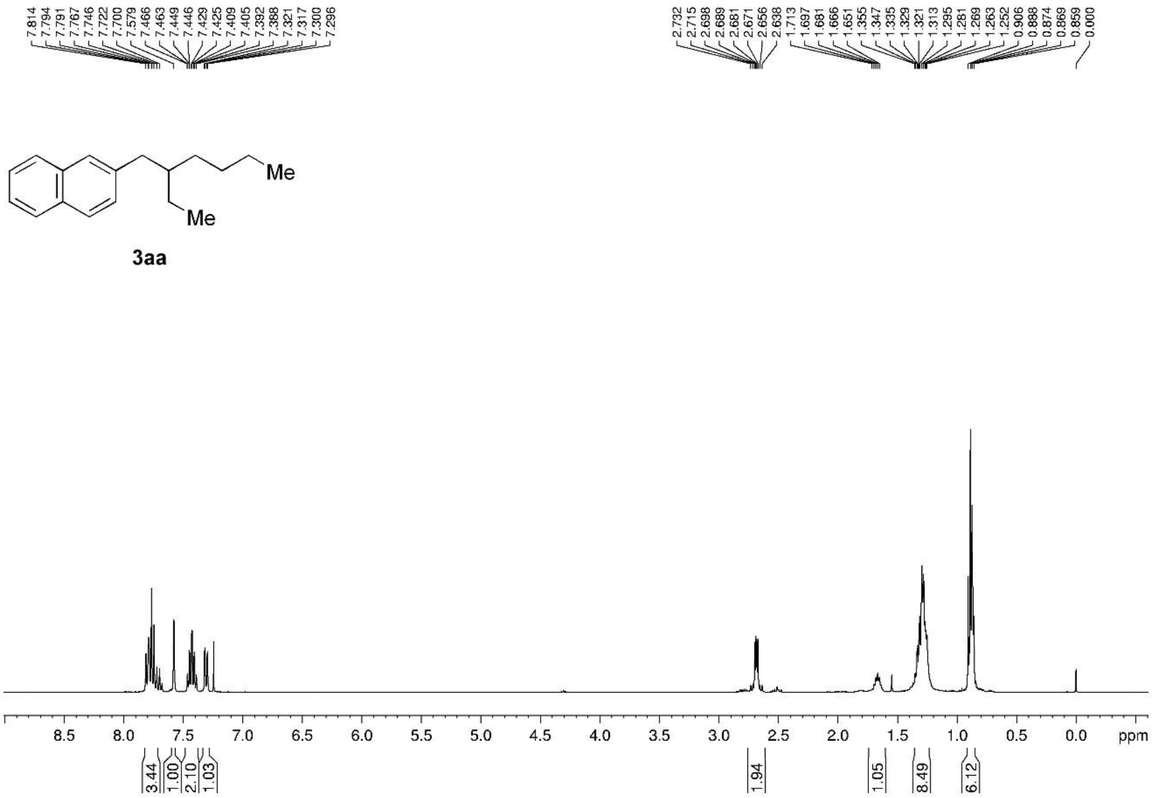
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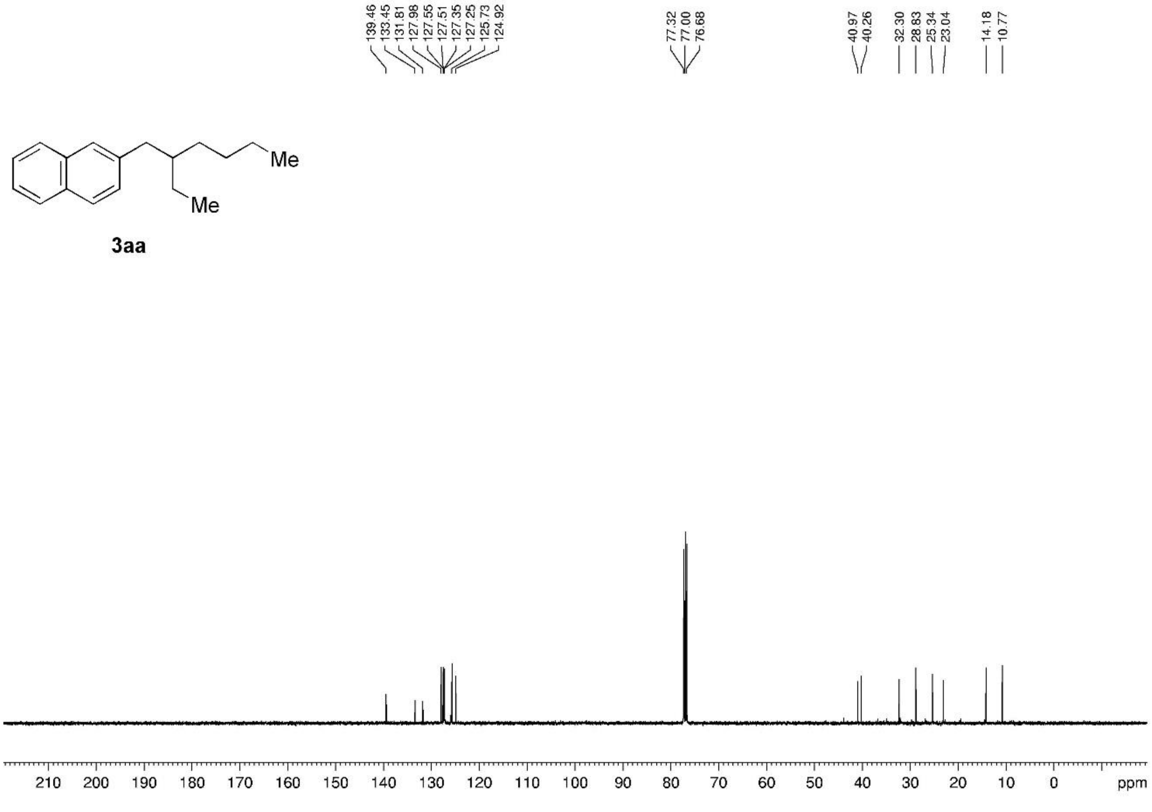
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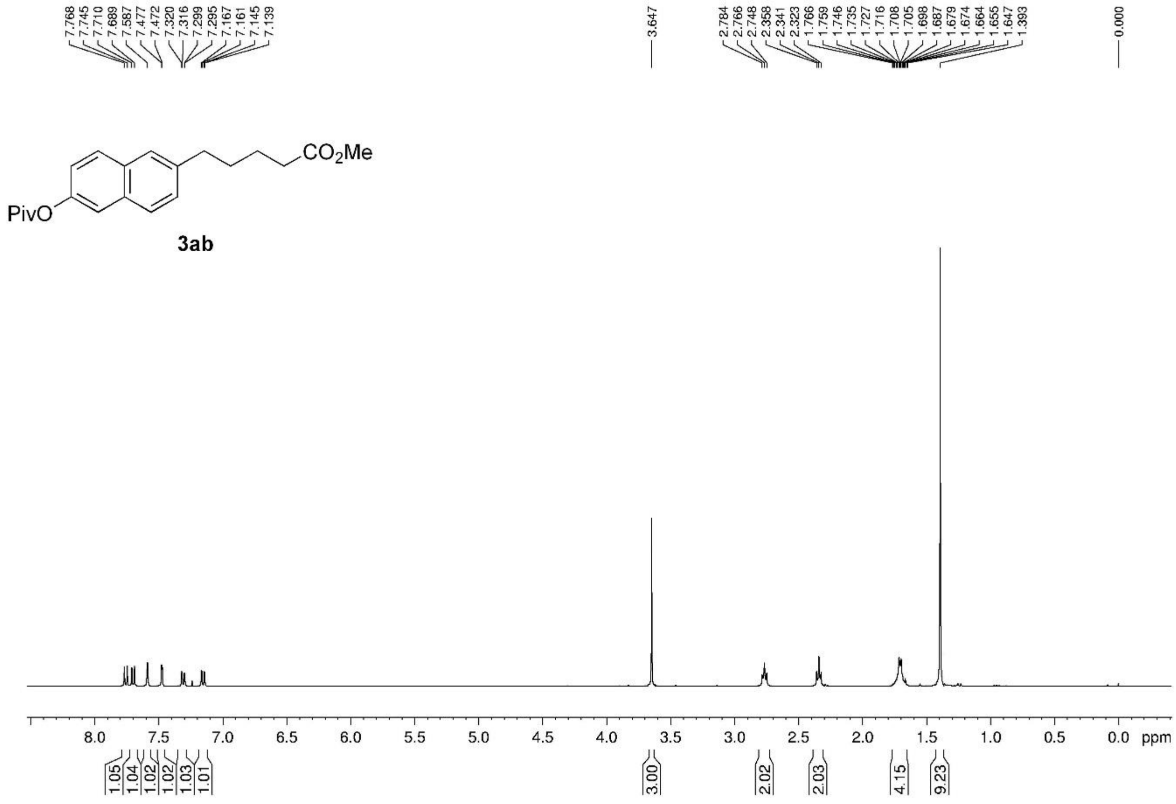
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CO2Me
PivO
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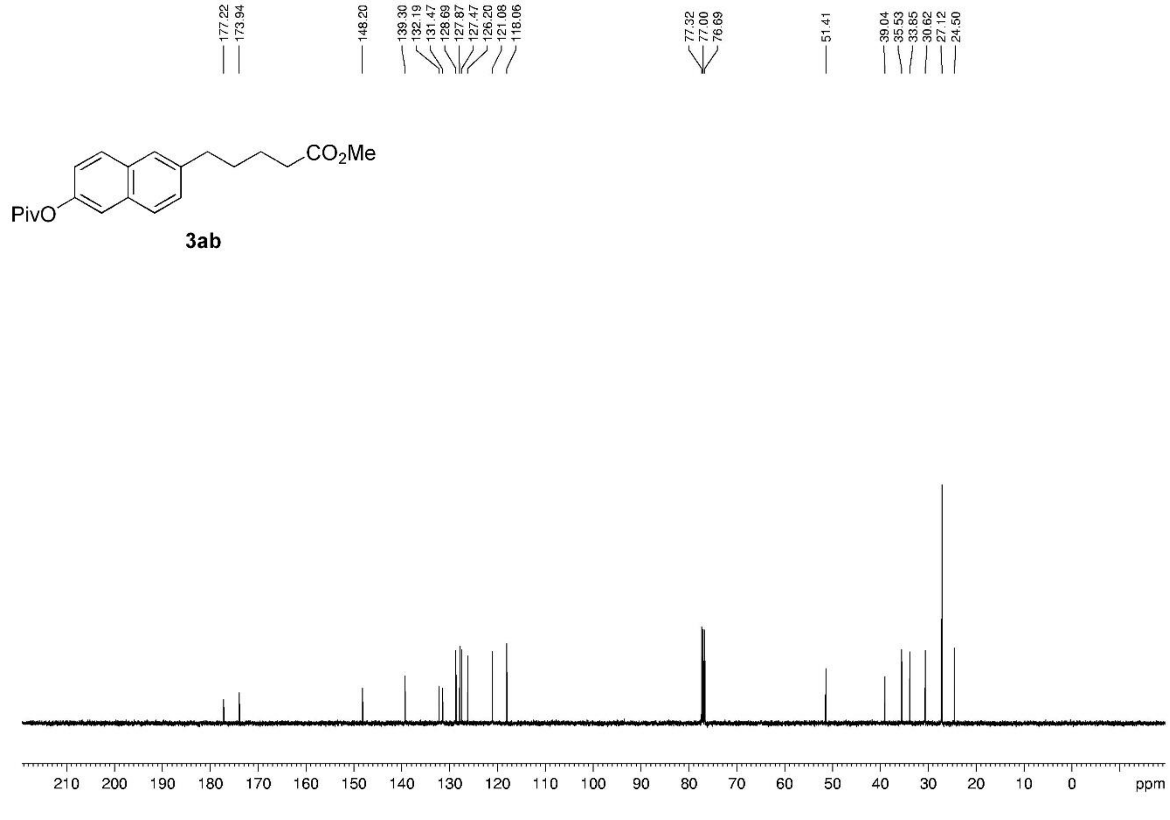
3ab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39.04
35.53
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30.62
27.12
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CO2Me
PivO
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30
20
10
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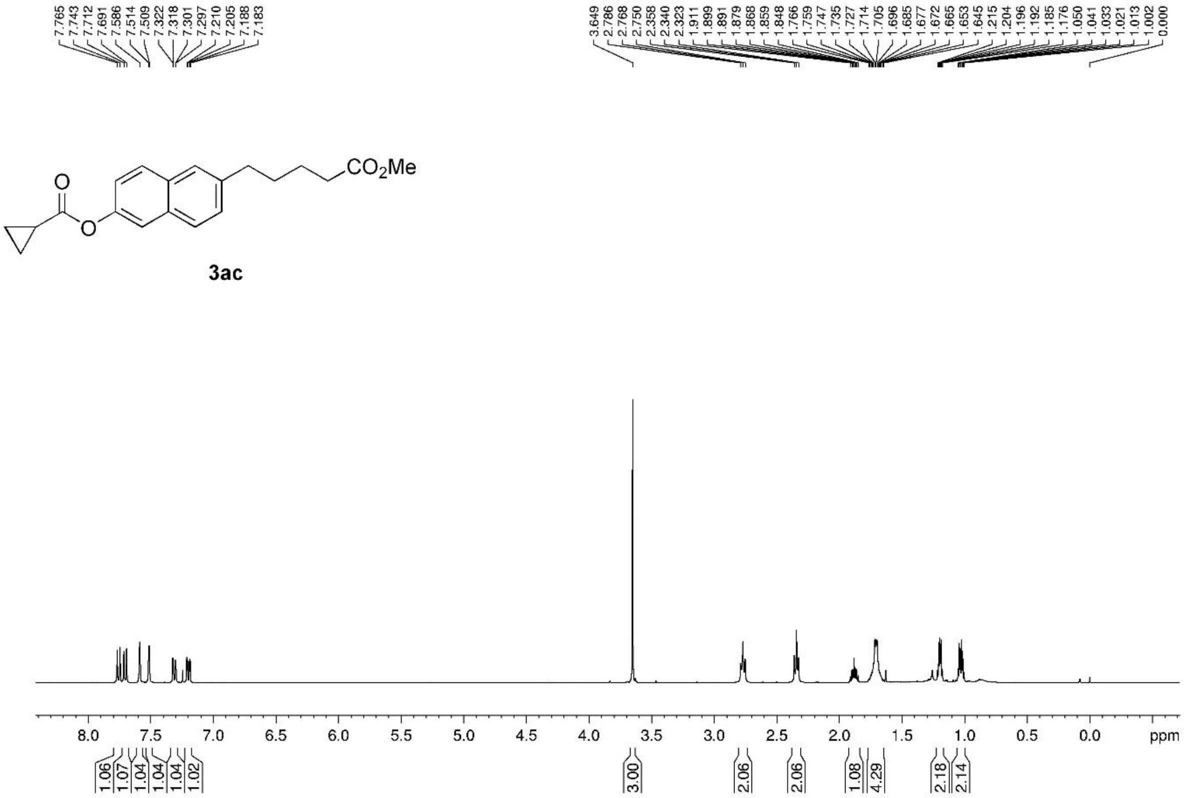
3ab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7.188
7.183
3.649
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2.358
2.340
2.323
1.911
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1.891
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1.766
1.759
1.747
1.735
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1.714
1.705
1.696
1.685
1.677
1.672
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1.653
1.645
1.215
1.204
1.196
1.192
1.185
1.176
1.050
1.041
1.033
1.021
1.013
1.002
0.000
O
O
CO_2Me
8.0
7.5
7.0
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6.0
5.5
5.0
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3.0
2.5
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0.0
ppm
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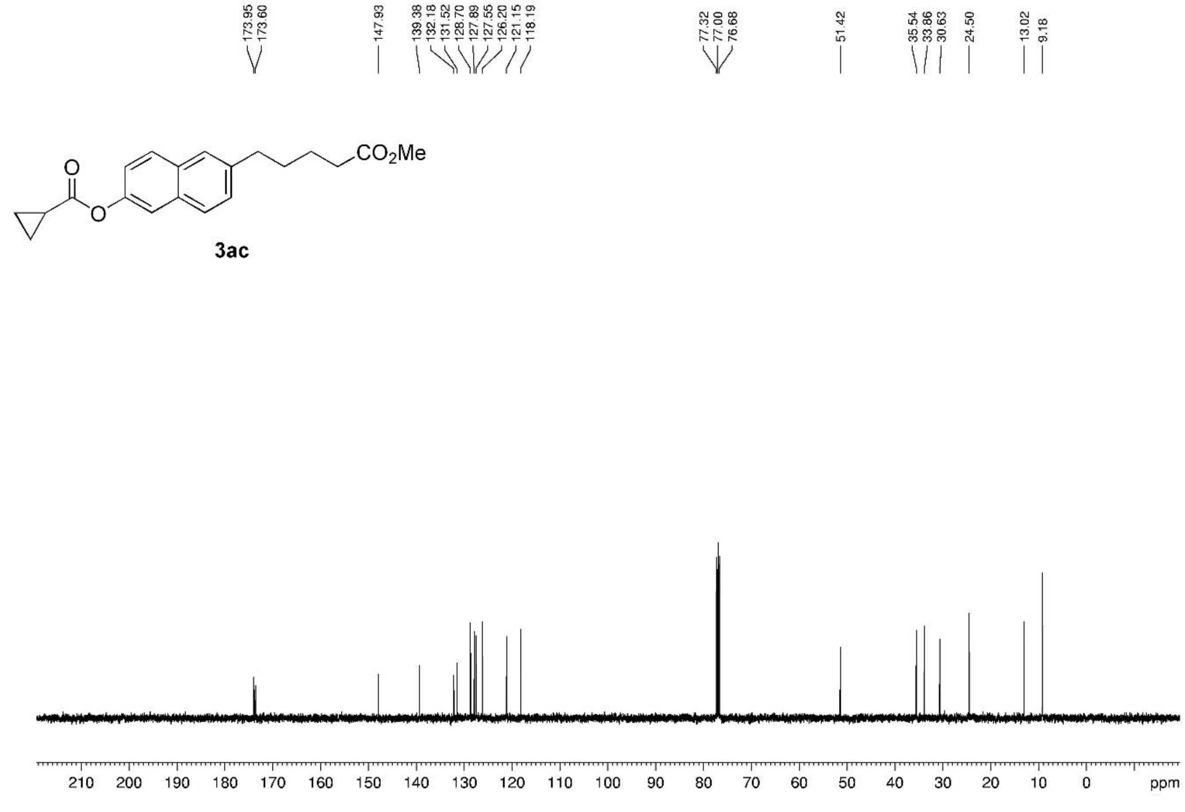
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77.00
76.68
51.42
35.54
33.86
30.63
24.50
13.02
9.18
O
O
CO_2Me
210
200
190
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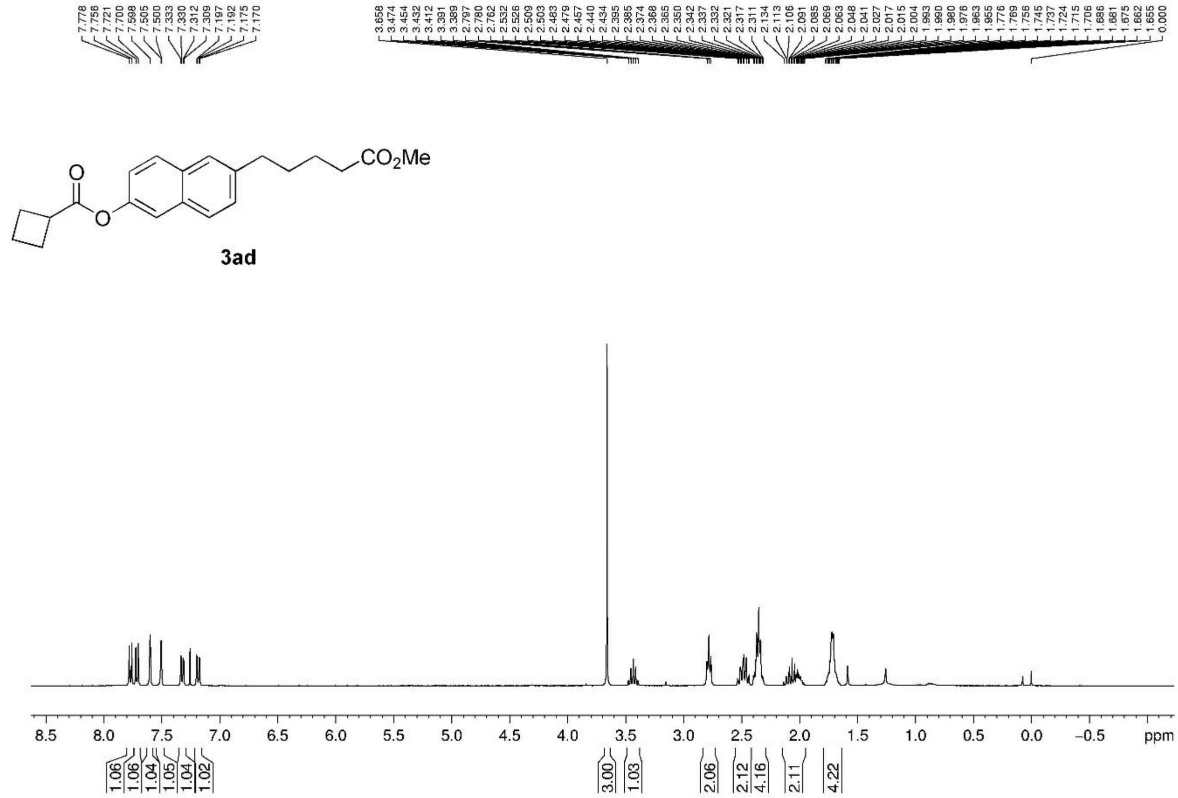
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3.389
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2.526
2.509
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2.434
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2.385
2.374
2.368
2.365
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2.337
2.332
2.321
2.317
2.311
2.134
2.113
2.106
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2.069
2.063
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2.041
2.027
2.017
2.015
2.004
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1.758
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1.737
1.724
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1.706
1.696
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1.655
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CO2Me
O
O
3ad
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7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
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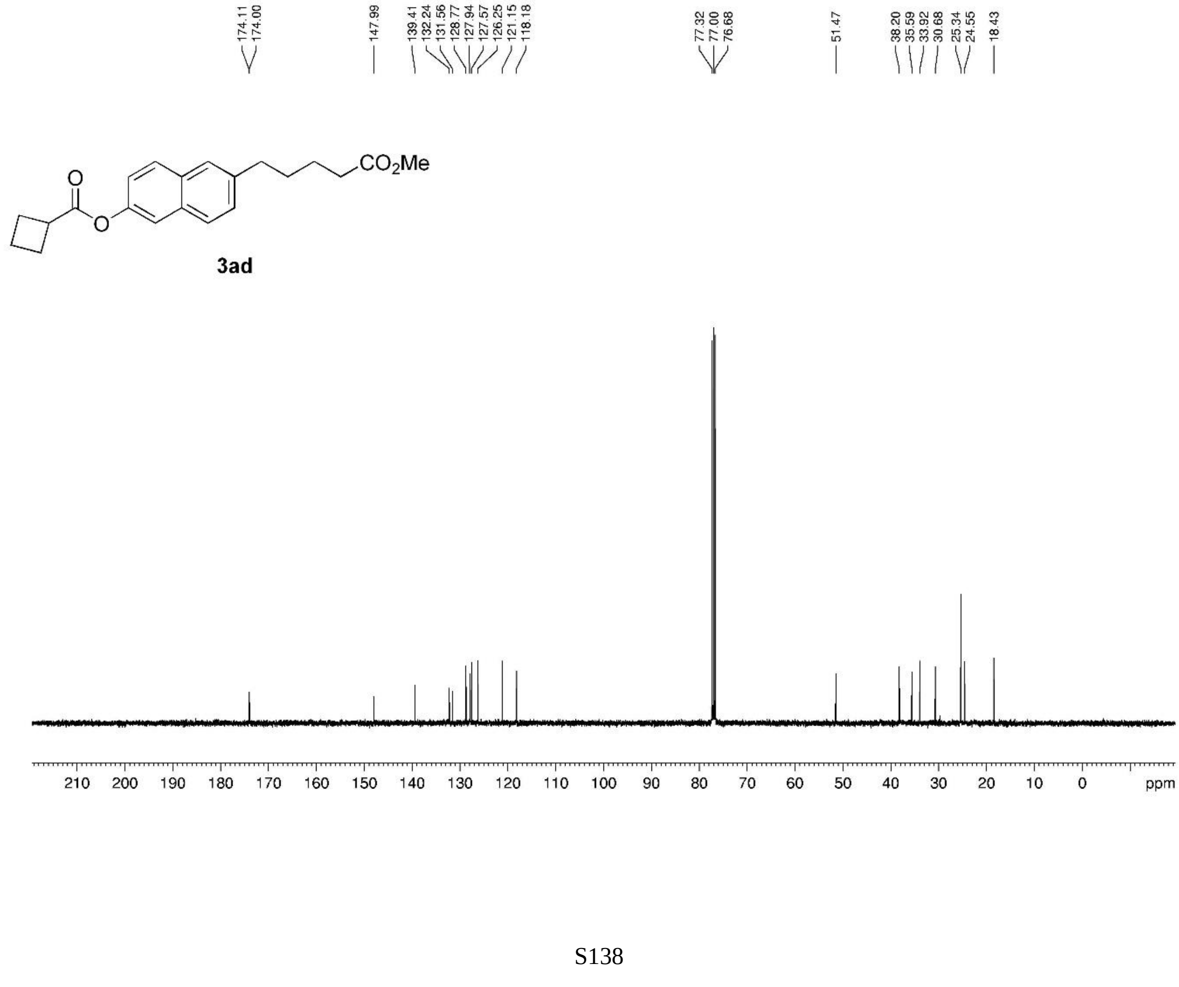
4.22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51.47
38.20
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33.92
30.68
25.34
24.55
18.43
CO2Me
O
O
3ad
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200
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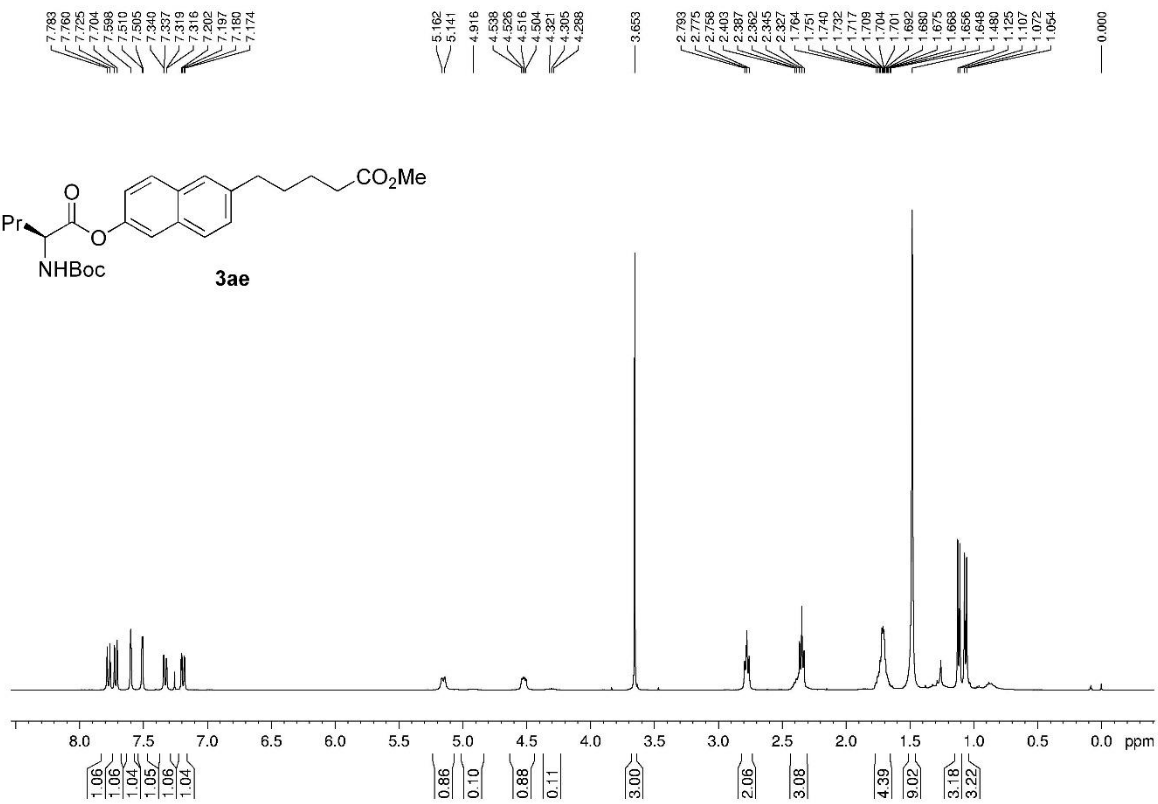
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7.316
7.202
7.197
7.180
7.174
5.162
5.141
4.916
4.538
4.526
4.516
4.504
4.321
4.305
4.288
3.653
2.793
2.775
2.758
2.403
2.387
2.362
2.345
2.327
1.764
1.751
1.740
1.732
1.717
1.709
1.704
1.701
1.692
1.680
1.675
1.668
1.656
1.648
1.480
1.125
1.107
1.072
1.054
0.000
iPr
O
O
CO2Me
NHBoc
3ae
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
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1.06
1.04
1.05
1.06
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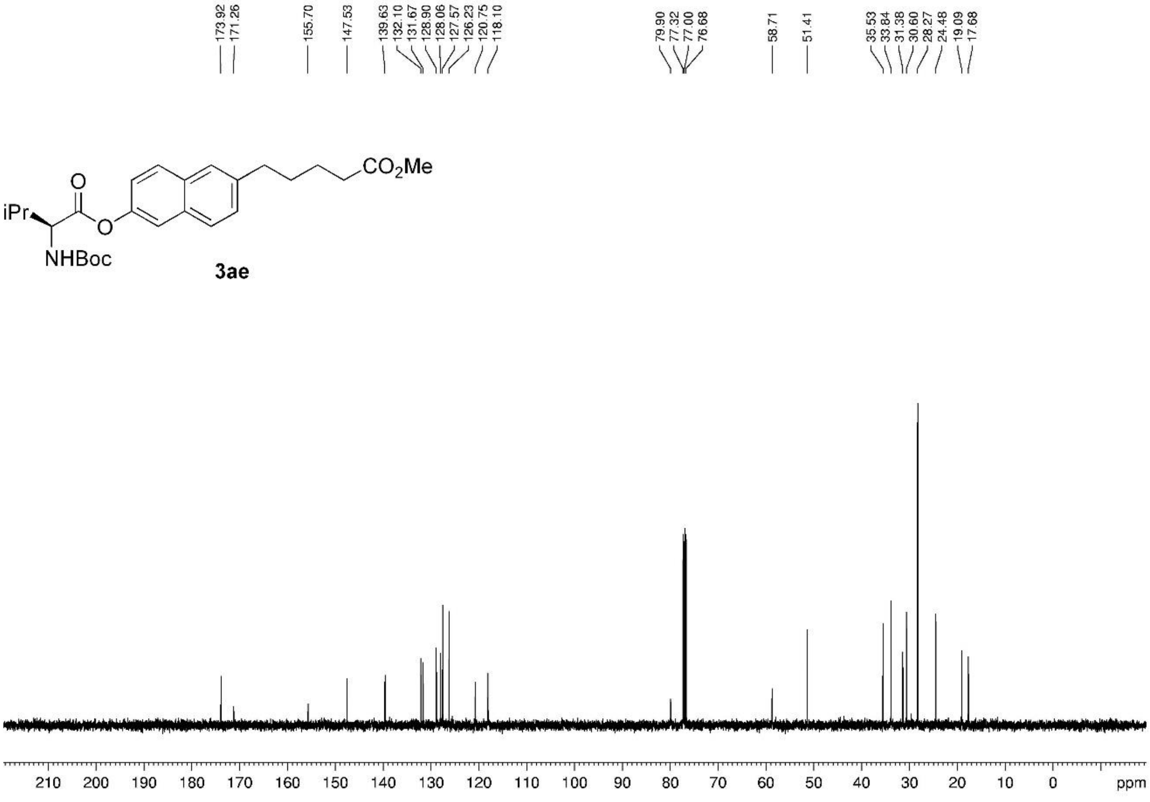
3.22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77.32
77.00
76.68
58.71
51.41
35.53
33.84
31.38
30.60
28.27
24.48
19.09
17.68
iPr
O
O
CO2Me
NHBoc
3ae
210
200
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140
130
120
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60
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30
20
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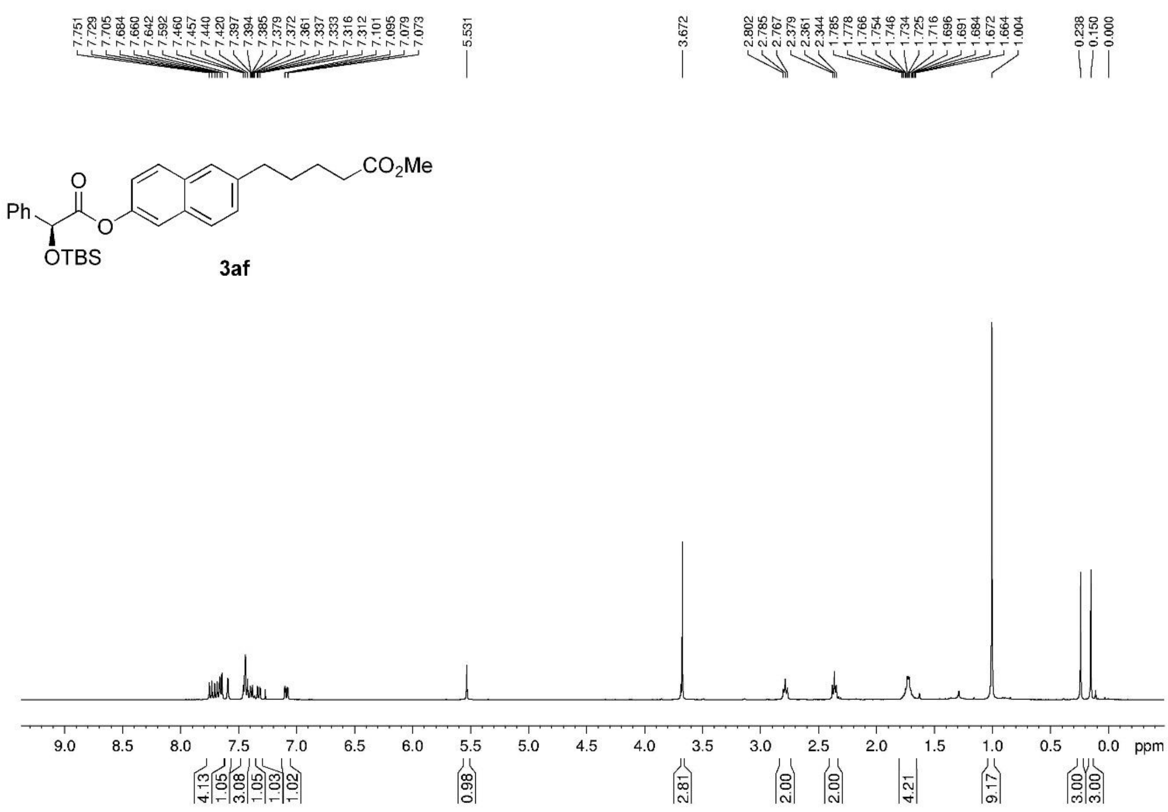
ppm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7.440
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7.385
7.379
7.372
7.361
7.337
7.333
7.316
7.312
7.101
7.095
7.079
7.073
5.531
3.672
2.802
2.785
2.767
2.379
2.361
2.344
1.785
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1.766
1.754
1.746
1.734
1.725
1.716
1.696
1.691
1.684
1.672
1.664
1.004
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0.150
0.000
Ph
O
O
OTBS
CO2Me
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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1.05
3.08
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1.03
1.02
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4.21
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3.00
3.00

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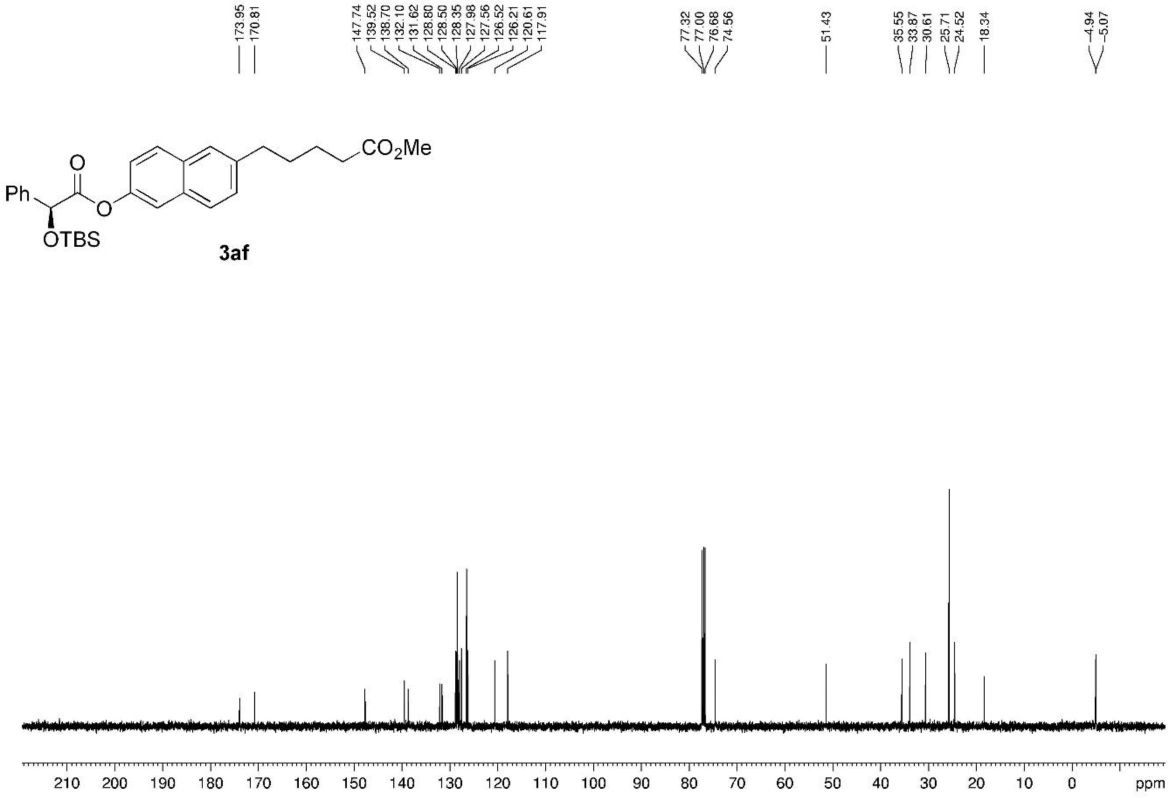
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126.52
126.21
120.61
117.91
77.32
77.00
76.68
74.56
51.43
35.55
33.87
30.61
25.71
24.52
18.34
-4.94
-5.07
Ph
O
O
OTBS
CO2Me
210
200
190
180
170
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150
140
130
120
110
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90
80
70
60
50
40
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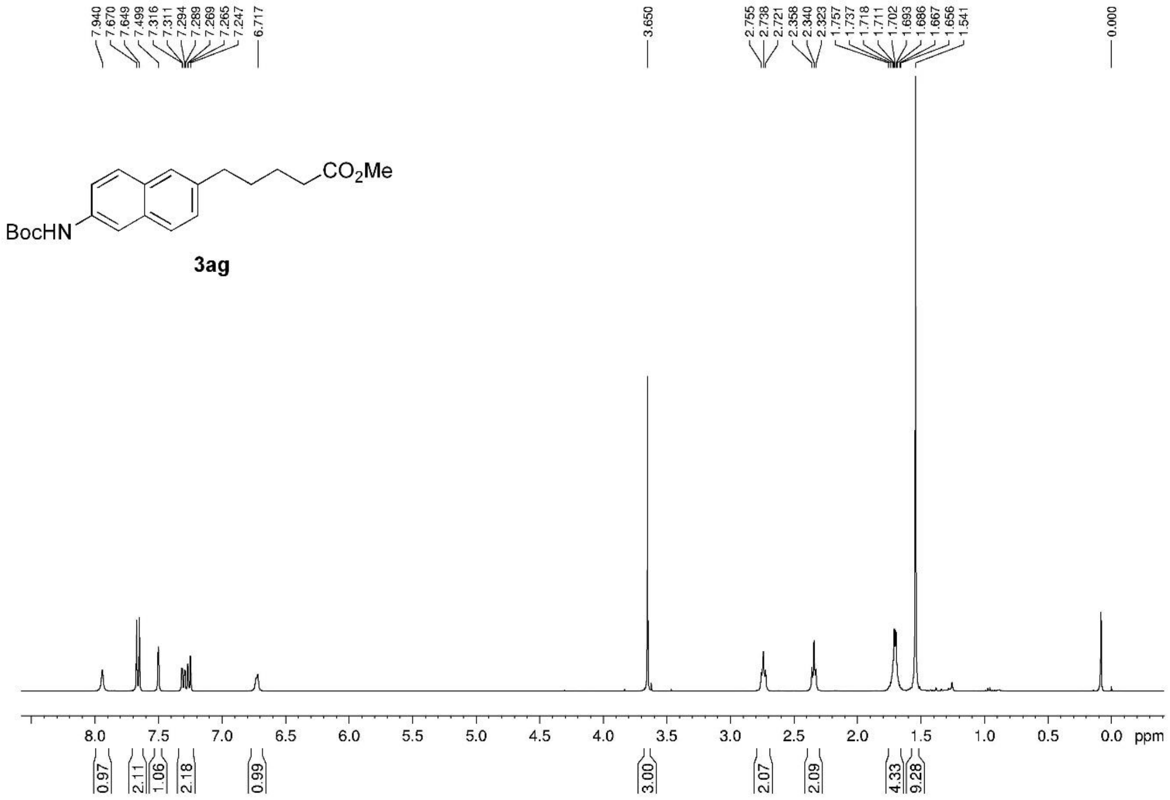
10
0
ppm

3af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7.265
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6.717
3.650
2.755
2.738
2.721
2.358
2.340
2.323
1.757
1.737
1.718
1.711
1.702
1.693
1.686
1.667
1.656
1.541
0.000
BocHN
CO2Me
3ag
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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2.09
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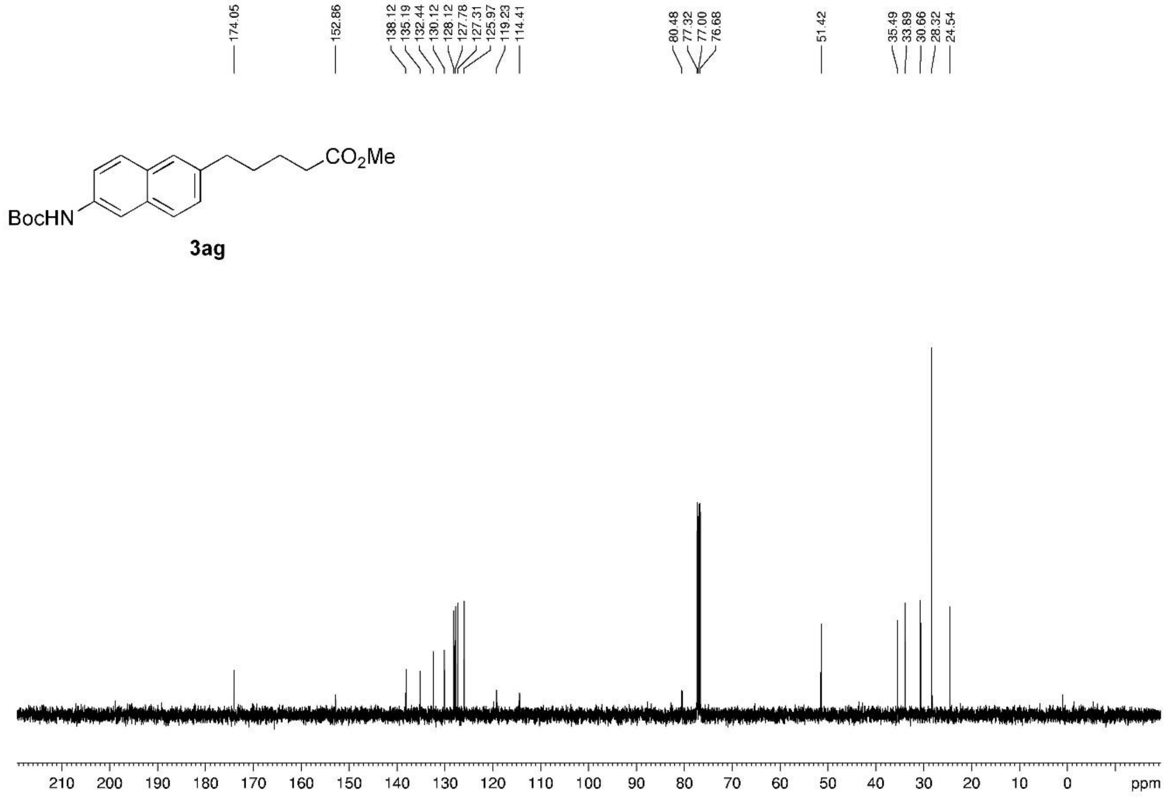
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80.48
77.32
77.00
76.68
51.42
35.49
33.89
30.66
28.32
24.54
BocHN
CO2Me
3ag
210
200
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180
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130
120
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100
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80
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60
50
40
30
20
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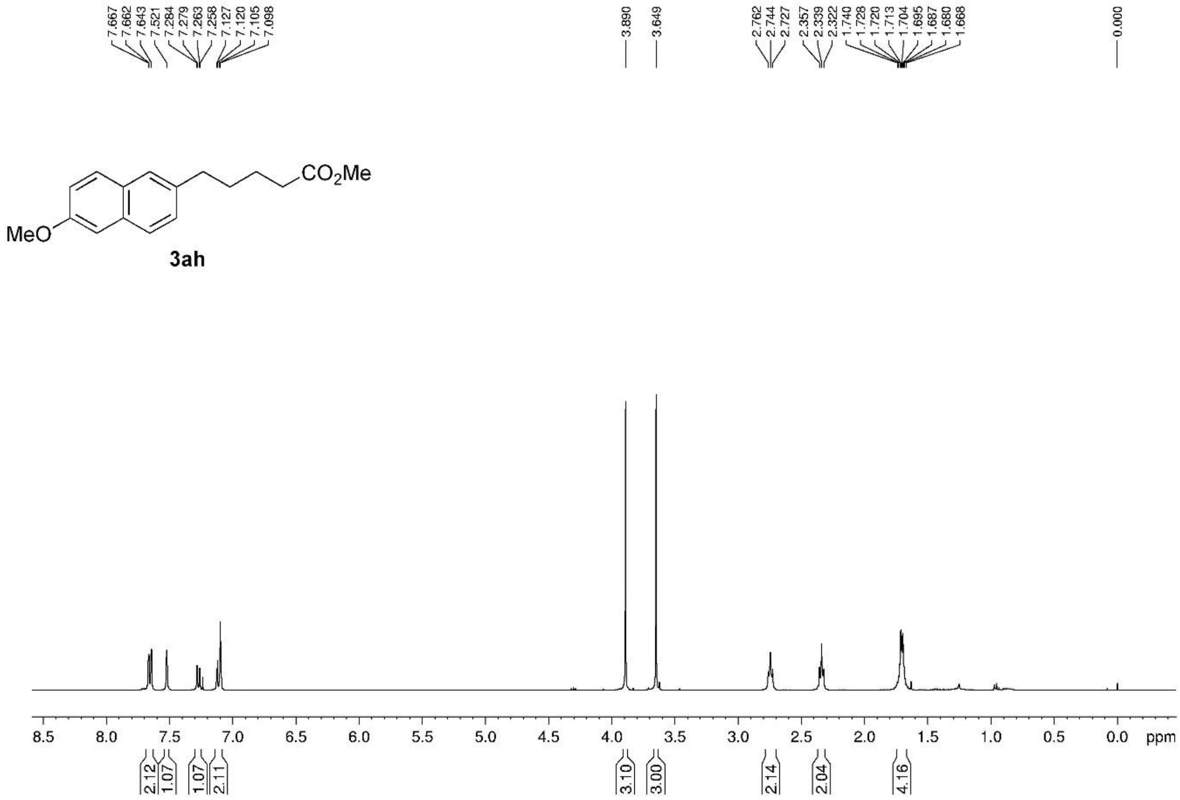
0
ppm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7.127
7.120
7.105
7.098
3.890
3.649
2.762
2.744
2.727
2.357
2.339
2.322
1.740
1.728
1.720
1.713
1.704
1.695
1.687
1.680
1.668
0.000
CO2Me
MeO
3ah
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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ppm
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1.07
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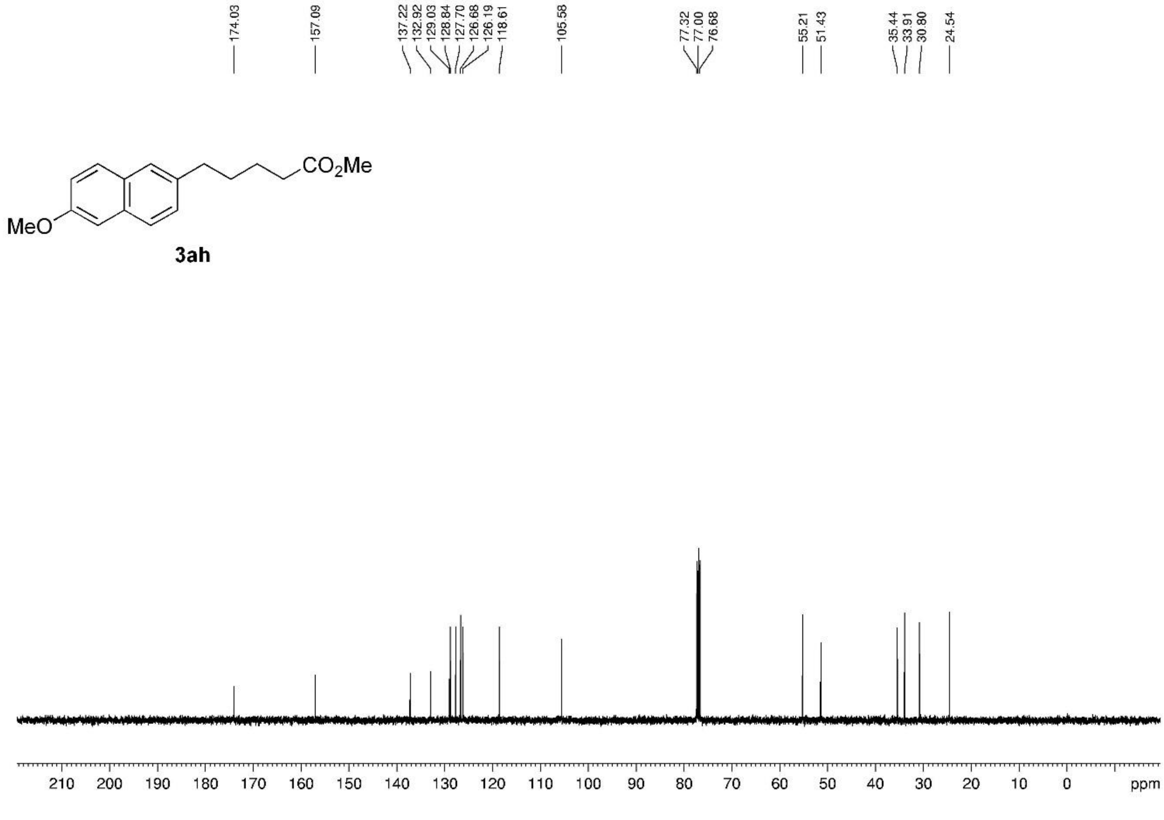
2.04
4.16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77.00
76.68
55.21
51.43
35.44
33.91
30.80
24.54
CO2Me
MeO
3ah
210
200
190
180
170
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130
120
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90
80
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60
50
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30
20
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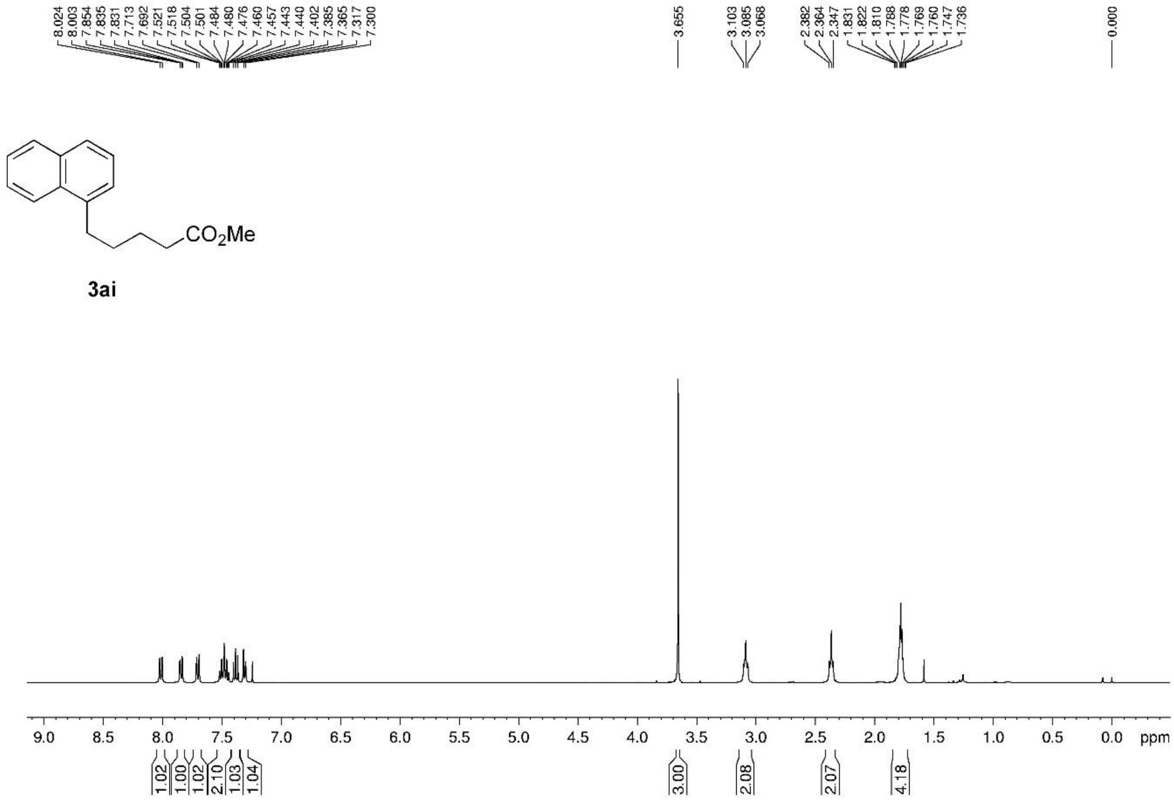
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7.457
7.443
7.440
7.402
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7.317
7.300
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3.103
3.085
3.068
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2.364
2.347
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1.822
1.810
1.788
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1.769
1.760
1.747
1.736
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8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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3.00
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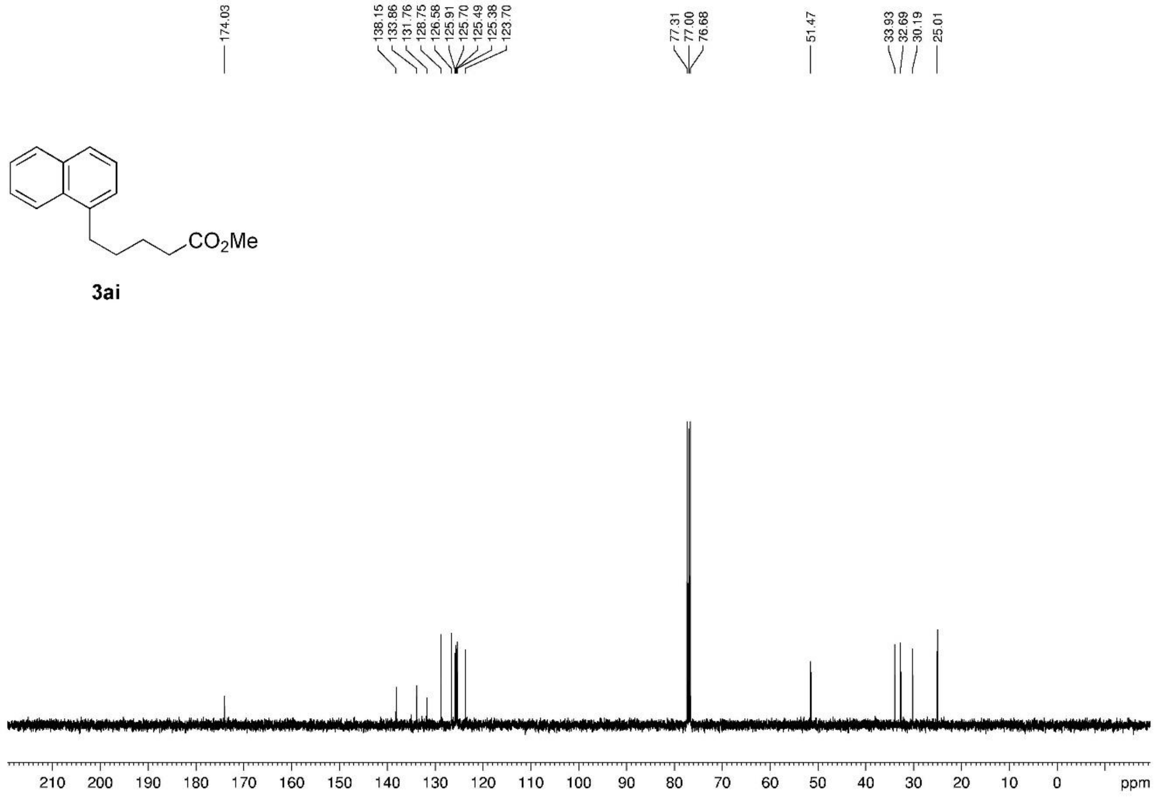
3ai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125.38
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76.68
51.47
33.93
32.69
30.19
25.01
CO2Me
210
200
190
180
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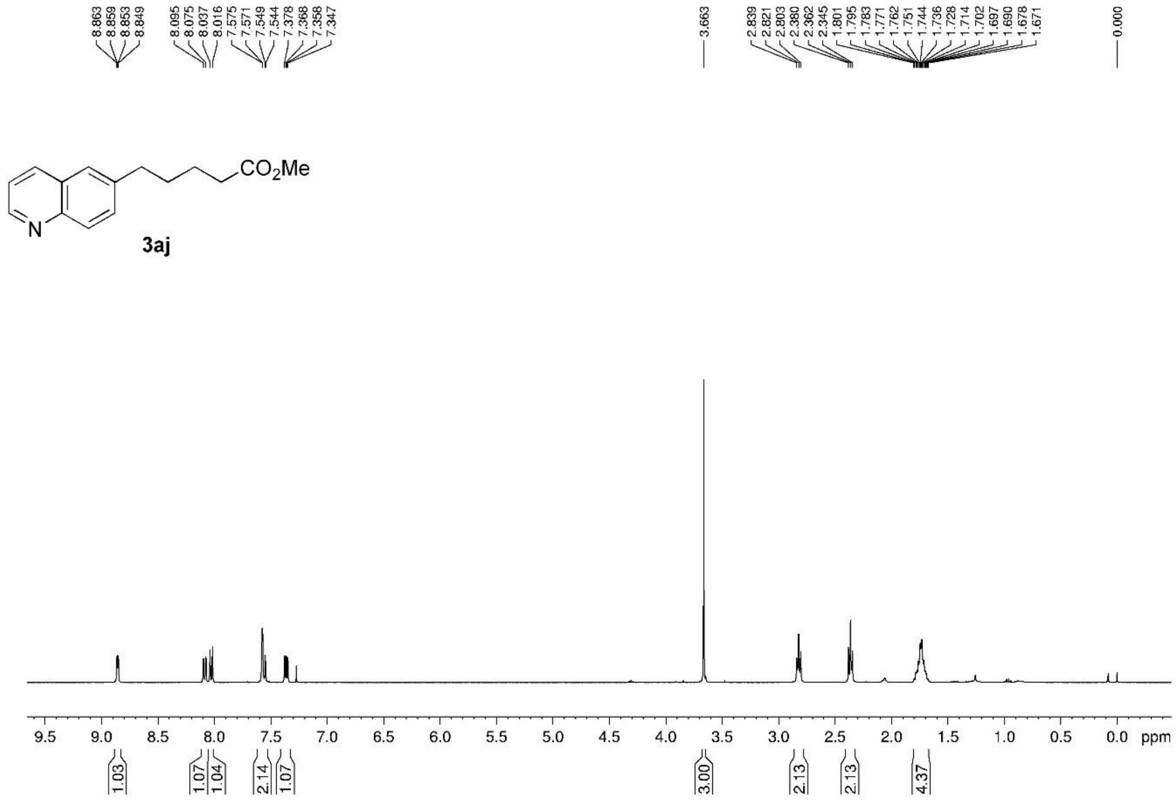
3ai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7.378
7.368
7.358
7.347
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1.795
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1.771
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1.744
1.736
1.728
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1.702
1.697
1.690
1.678
1.671
0.000
CO2Me
N
3aj
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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2.0
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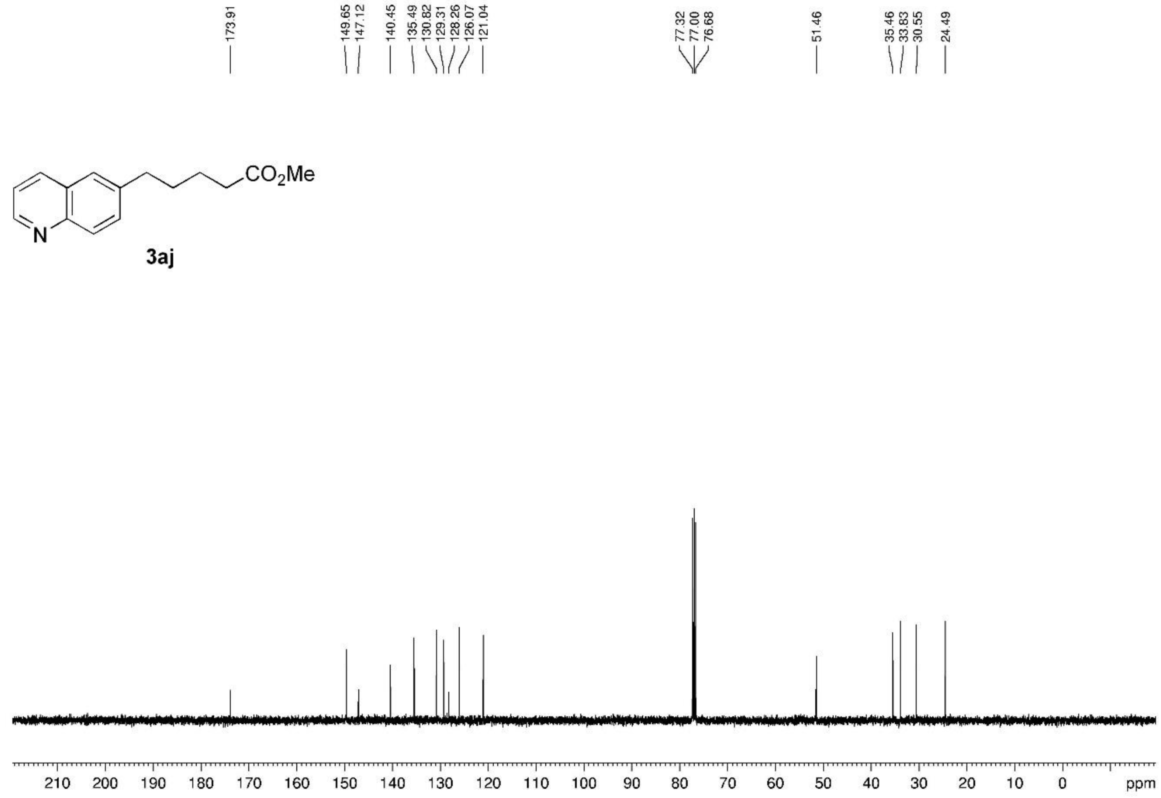
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77.00
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35.46
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30.55
24.49
CO2Me
N
3aj
210
200
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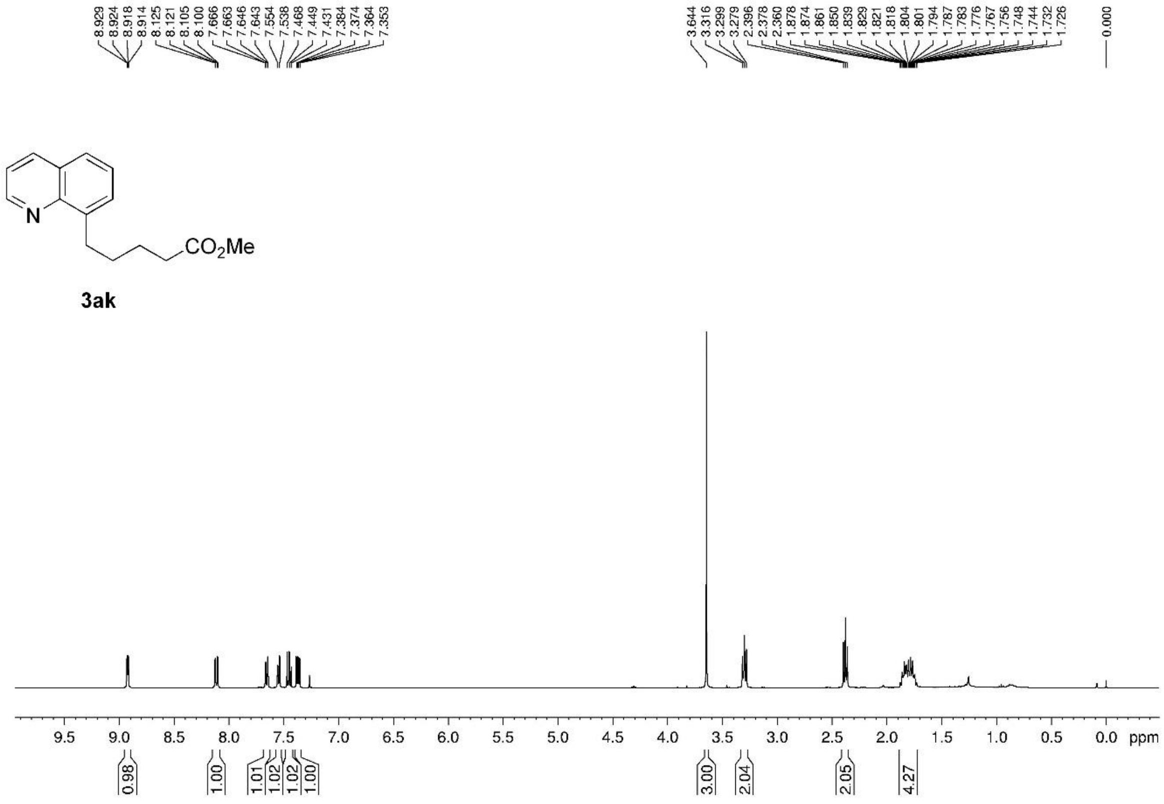
3ak

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1.767
1.756
1.748
1.744
1.732
1.726
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N
CO2Me
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8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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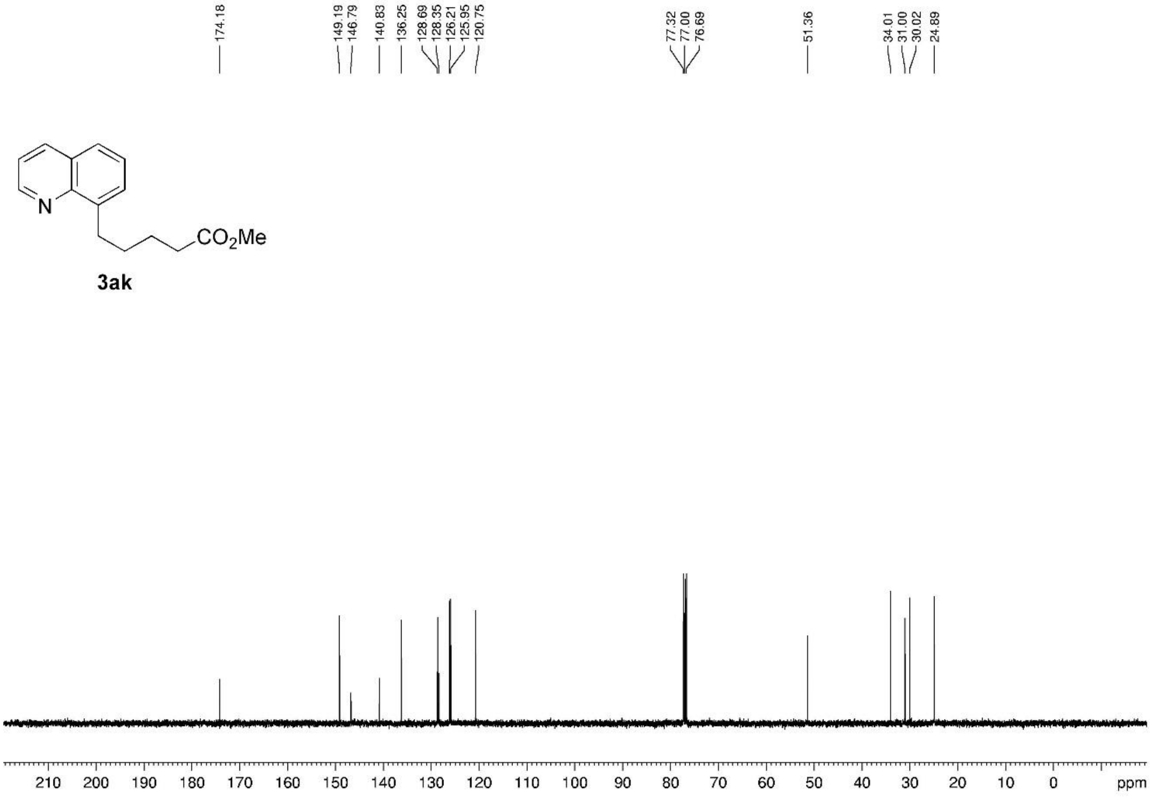
3ak

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76.69
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31.00
30.02
24.89
N
CO2Me
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200
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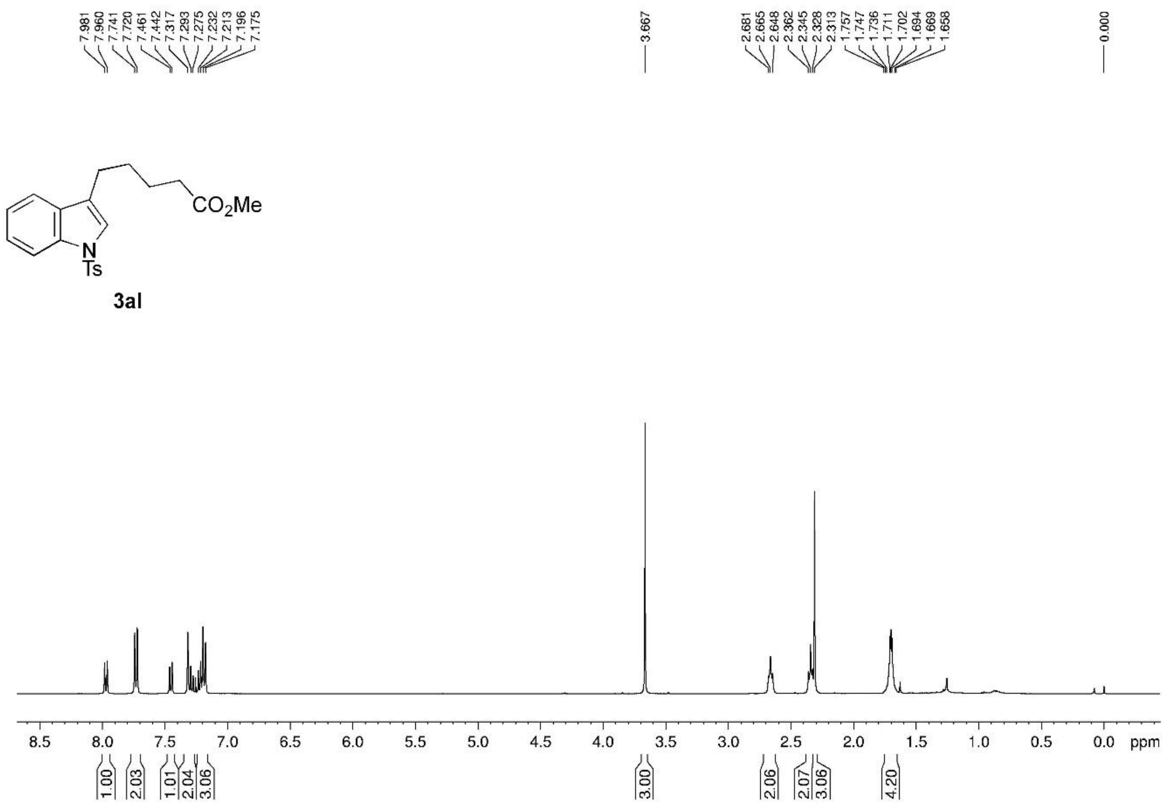
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7.175
3.667
2.681
2.665
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2.328
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1.747
1.736
1.711
1.702
1.694
1.669
1.658
0.000
CO2Me
N
Ts
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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1.01
2.04
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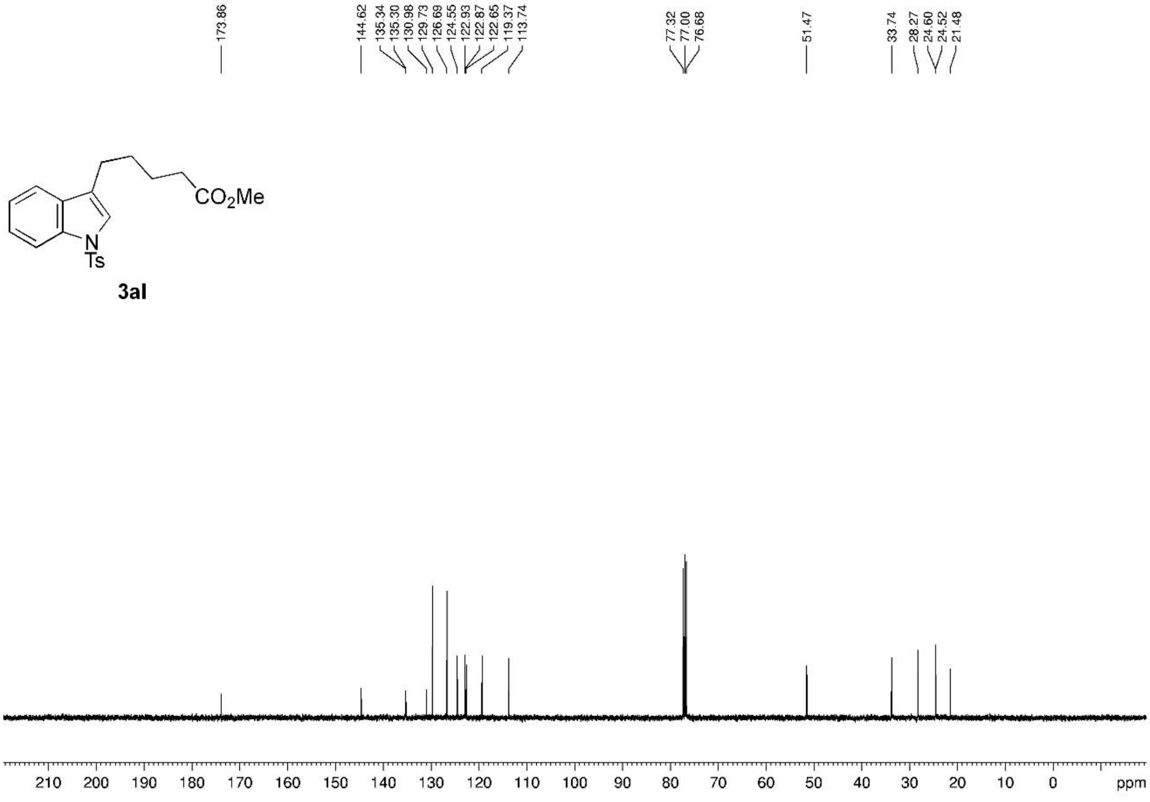
3.00
2.06
2.07
3.06
4.20

3al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113.74
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77.00
76.68
51.47
33.74
28.27
24.60
24.52
21.48
CO2Me
N
Ts
210
200
190
180
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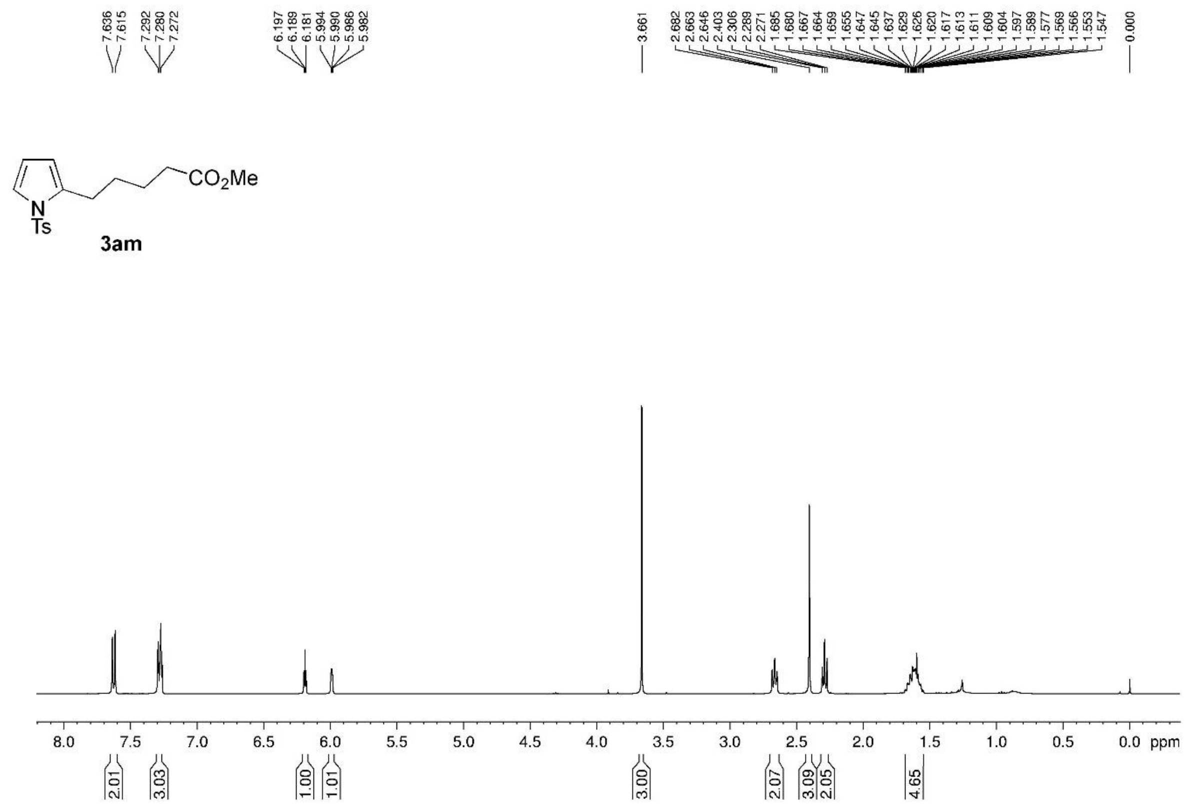
0
ppm

3al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2.663
2.646
2.403
2.306
2.289
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1.685
1.680
1.667
1.664
1.659
1.655
1.647
1.645
1.637
1.629
1.626
1.620
1.617
1.613
1.611
1.609
1.604
1.597
1.589
1.577
1.569
1.566
1.553
1.547
0.000
N
Ts
CO2Me
3am
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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1.01
3.00
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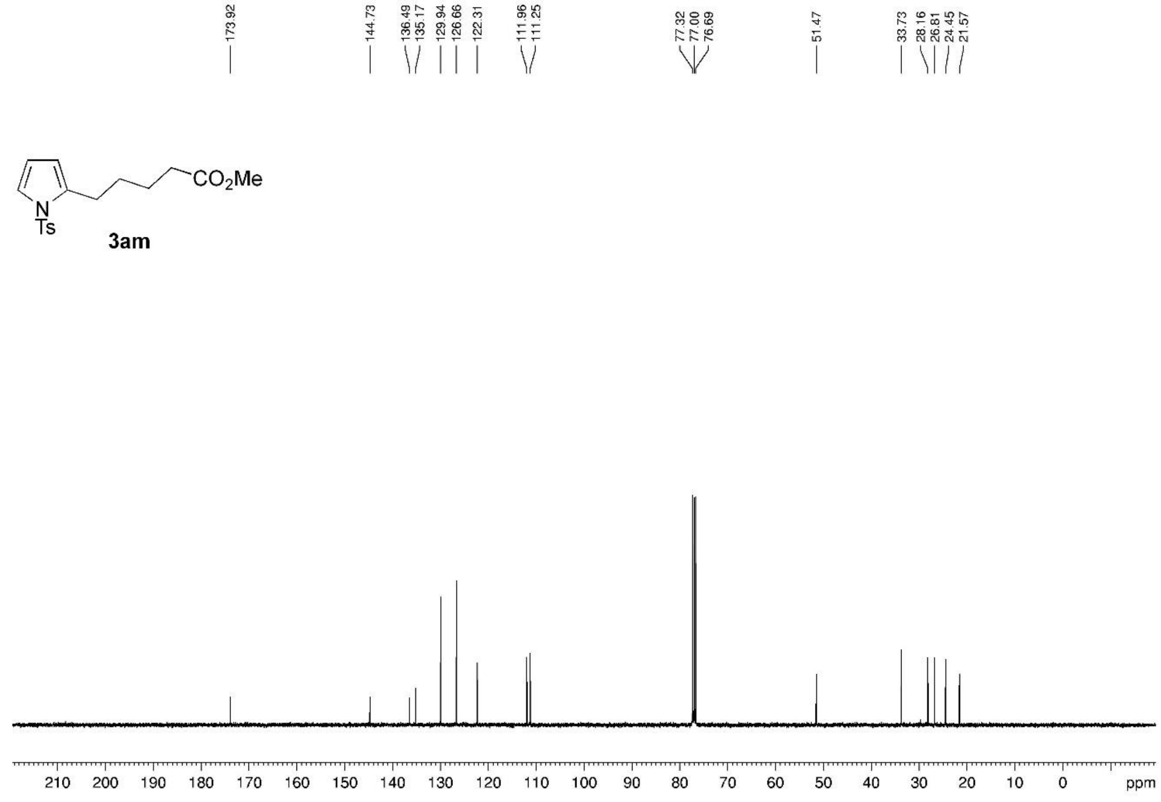
2.05
4.65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111.96
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51.47
33.73
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21.57
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Ts
CO2Me
3am
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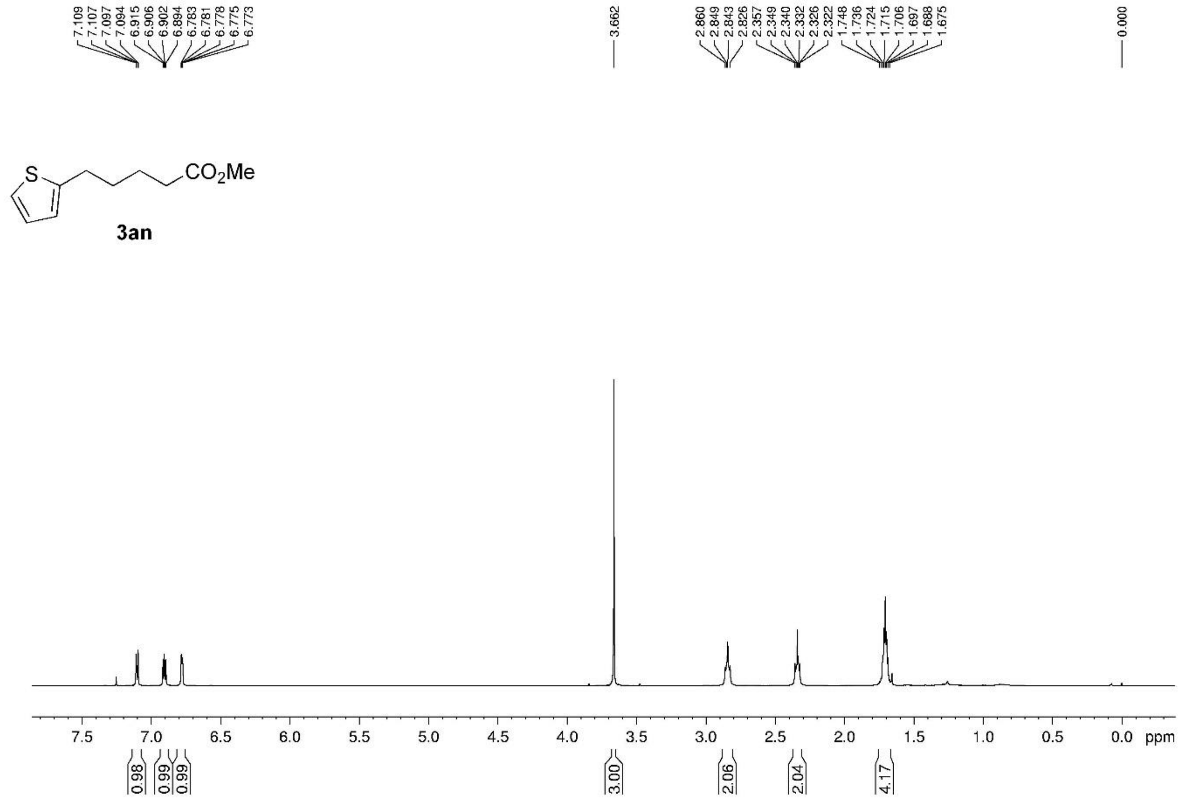
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6.775
6.773
3.662
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2.849
2.843
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2.357
2.349
2.340
2.332
2.326
2.322
1.748
1.736
1.724
1.715
1.706
1.697
1.688
1.675
0.000
S
CO2Me
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
2.0
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0.99
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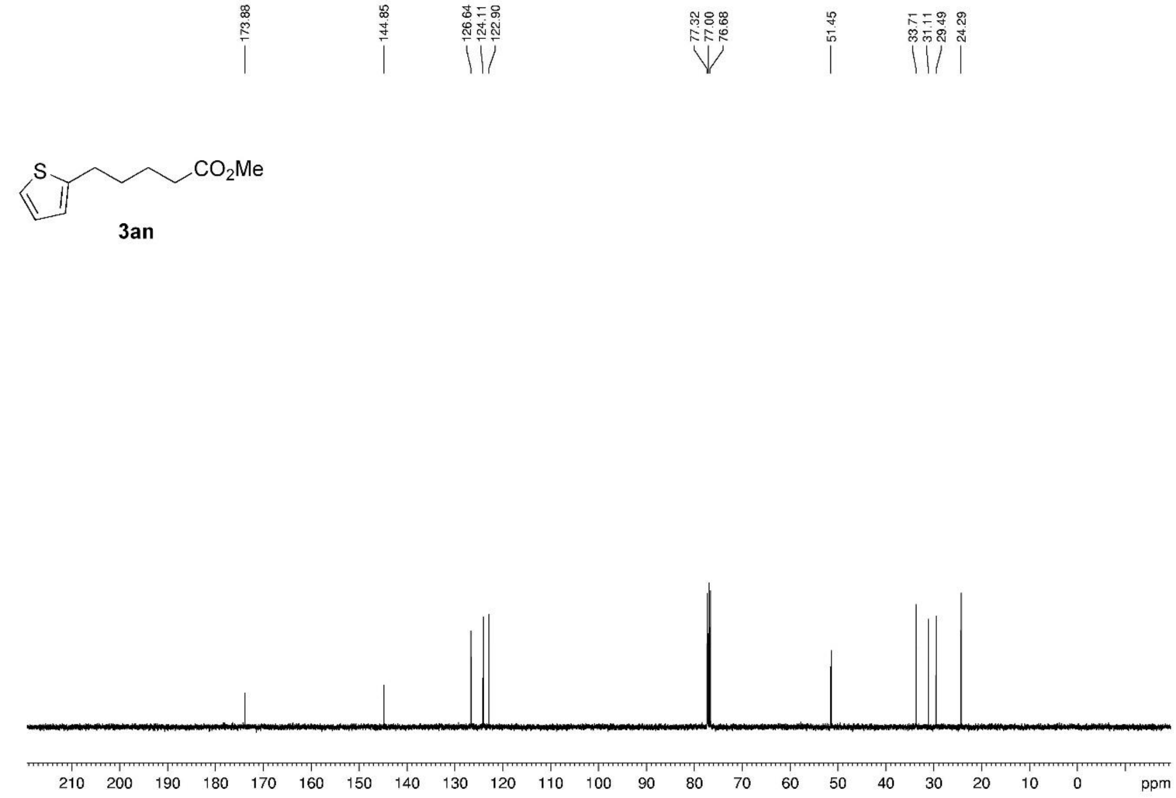
2.04
4.17

3an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24.29
S
CO2Me
210
200
190
180
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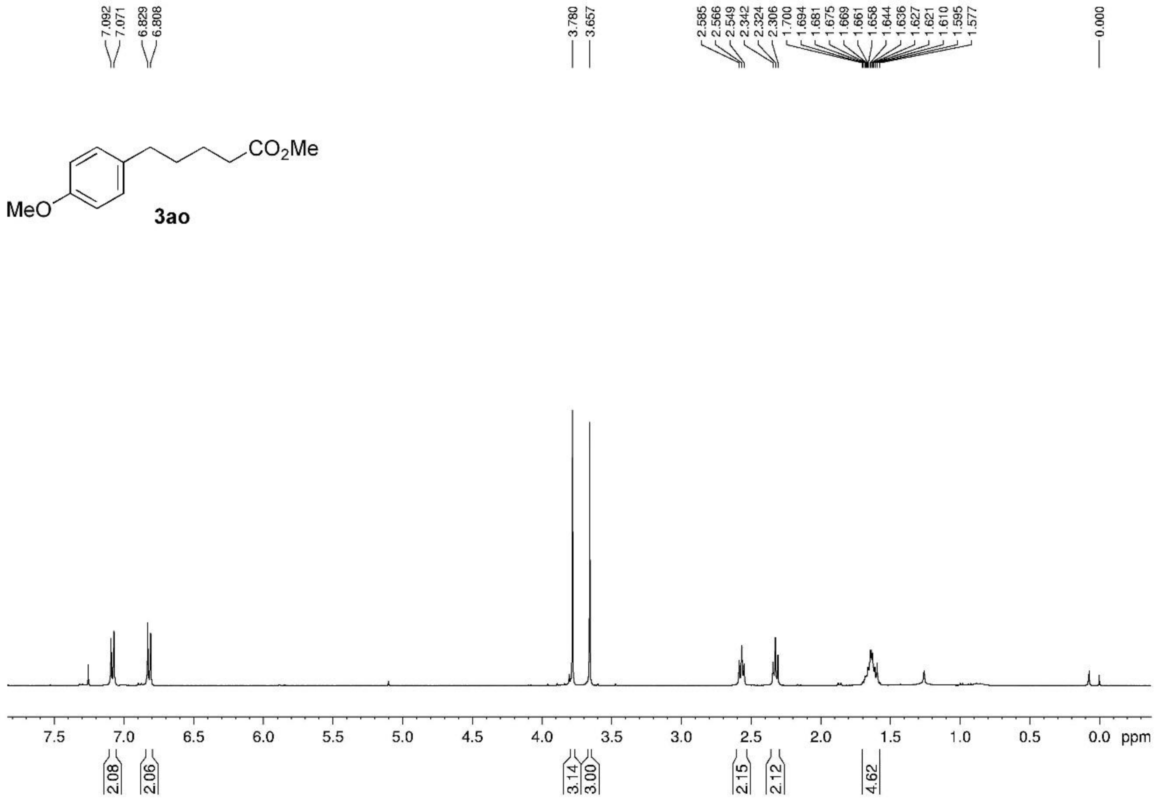
ppm

3an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1.700
1.694
1.681
1.675
1.669
1.661
1.658
1.644
1.636
1.627
1.621
1.610
1.595
1.577
0.000
CO2Me
MeO
3ao
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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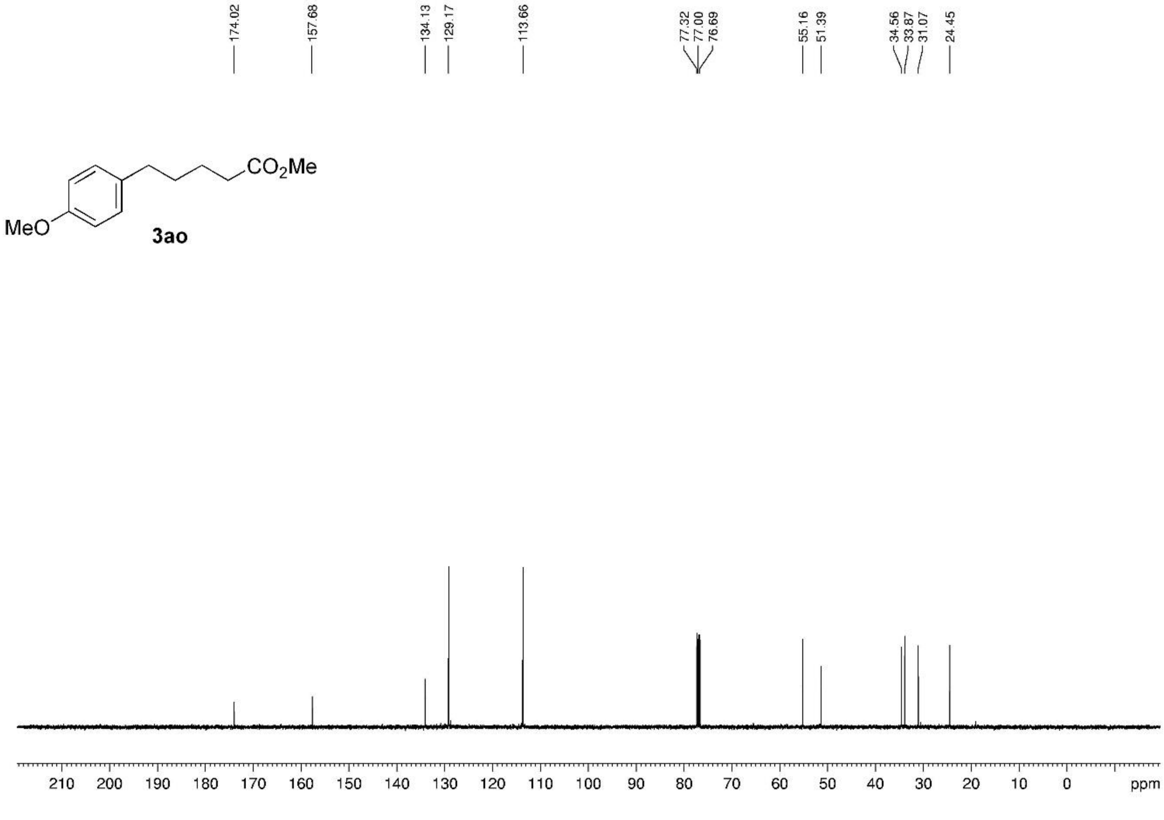
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51.39
34.56
33.87
31.07
24.45
CO2Me
MeO
3ao
210
200
190
180
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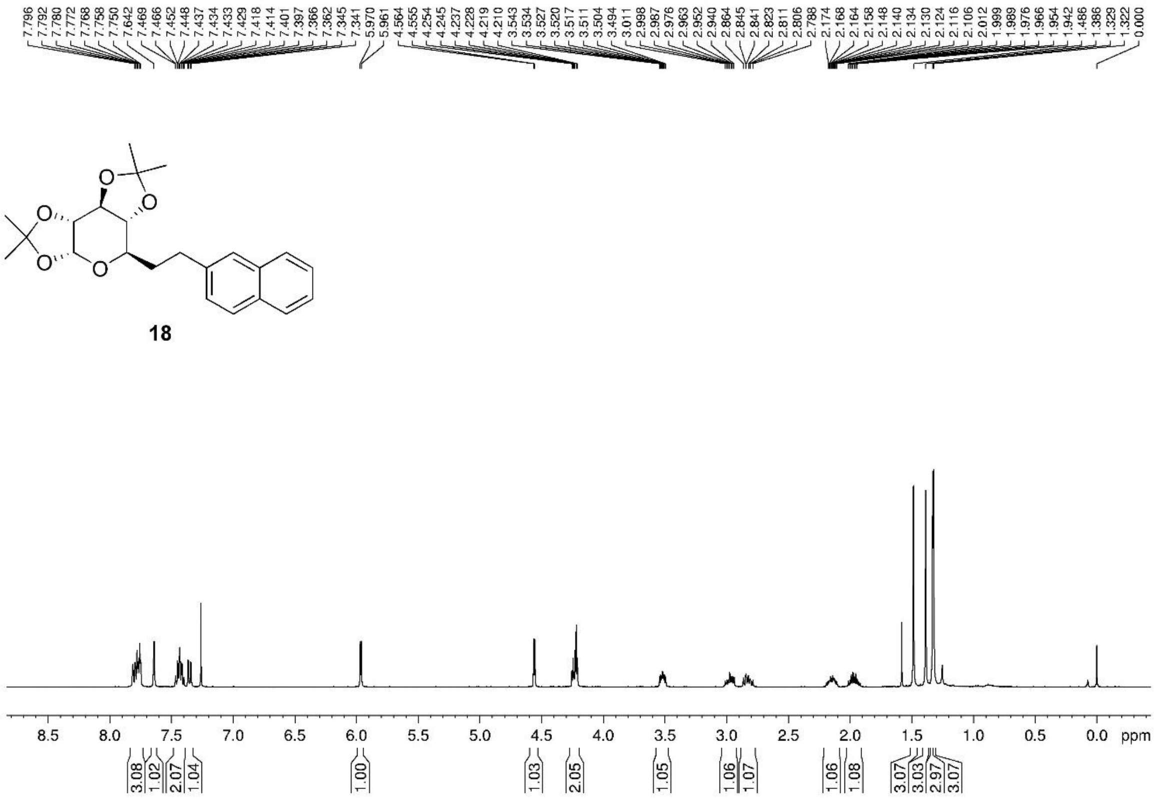
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.452
7.448
7.437
7.434
7.433
7.429
7.418
7.414
7.401
7.397
7.366
7.362
7.345
7.341
5.970
5.961
4.564
4.555
4.254
4.245
4.237
4.228
4.219
4.210
3.543
3.534
3.527
3.520
3.517
3.511
3.504
3.494
3.011
2.998
2.987
2.976
2.963
2.952
2.940
2.864
2.845
2.841
2.823
2.811
2.806
2.788
2.174
2.168
2.164
2.158
2.148
2.140
2.134
2.130
2.124
2.116
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1.989
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.08
1.02
2.07
1.04
1.00
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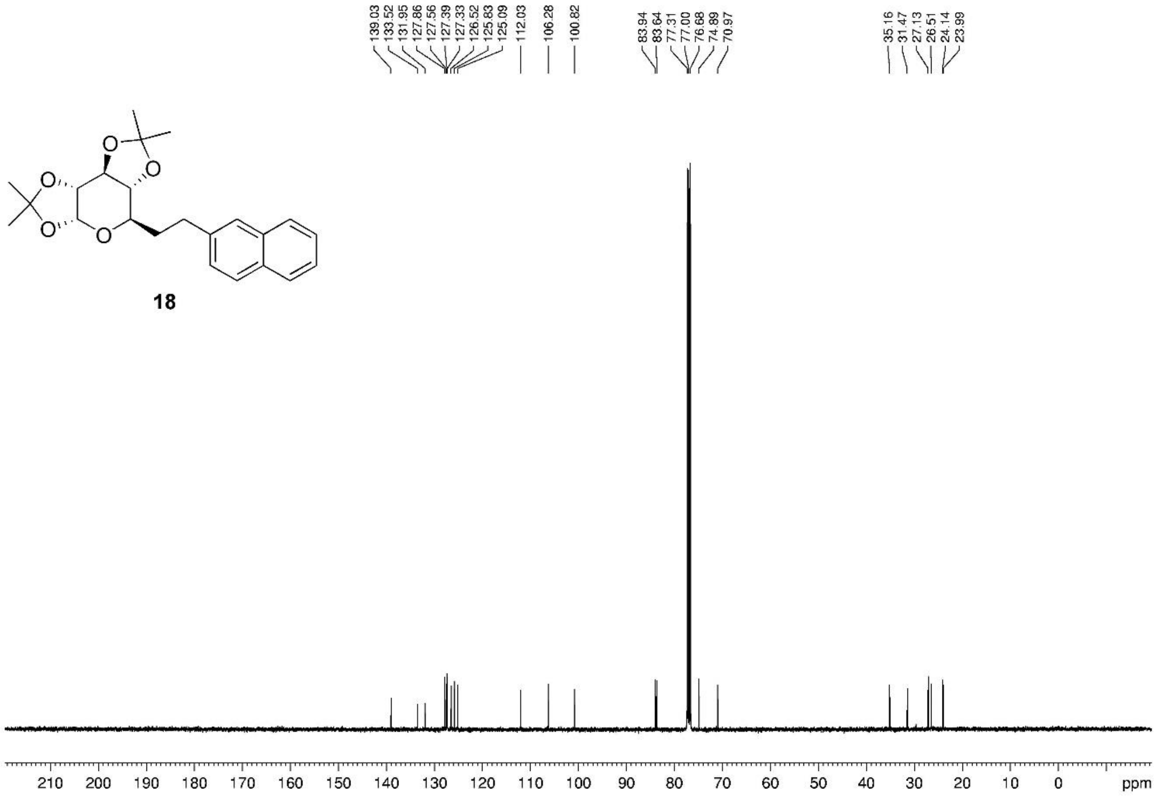
2.05
1.05
1.06
1.07
1.06
1.08
3.07
3.03
2.97
3.07

18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127.33
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125.09
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83.94
83.64
77.31
77.00
76.68
74.89
70.97
35.16
31.47
27.13
26.51
24.14
23.99
210
200
190
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50
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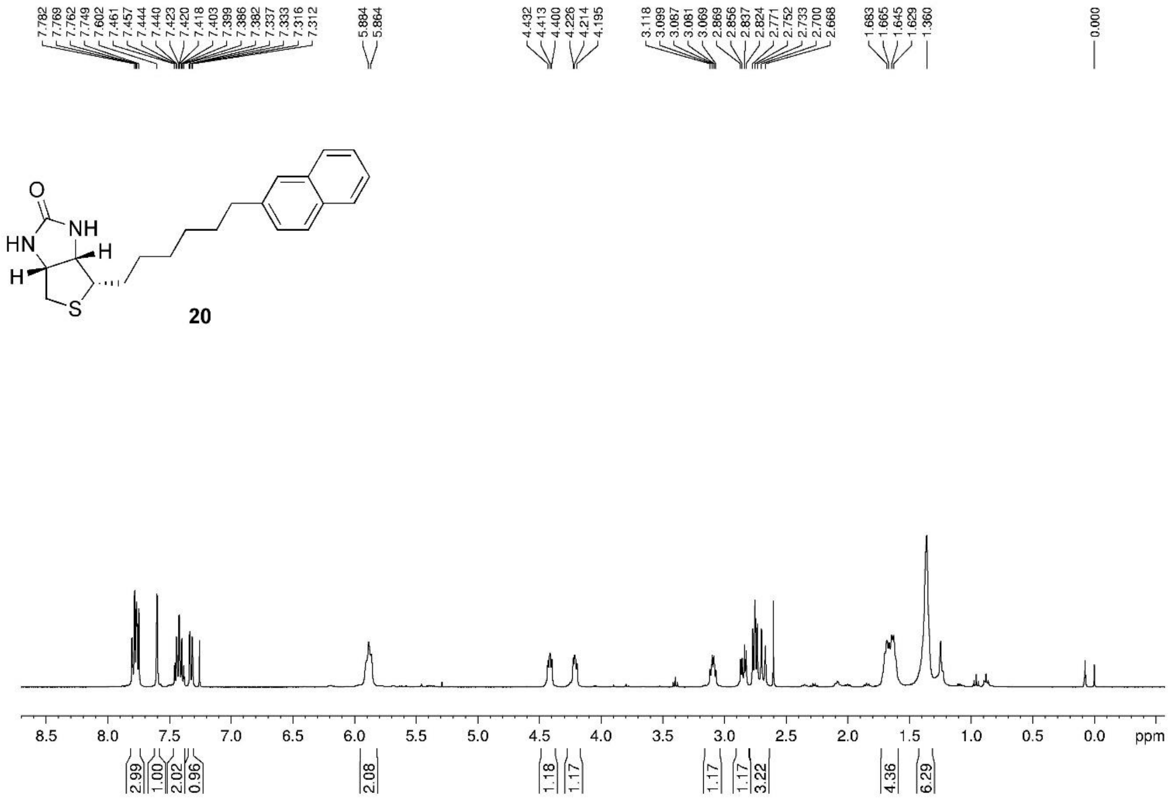
10
0
ppm

18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7.418
7.403
7.399
7.386
7.382
7.337
7.333
7.316
7.312
5.884
5.864
4.432
4.413
4.400
4.226
4.214
4.195
3.118
3.099
3.087
3.081
3.069
2.869
2.856
2.837
2.824
2.771
2.752
2.733
2.700
2.668
1.683
1.665
1.645
1.629
1.360
0.000
O
NH
HN
H
H
S
20
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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2.02
0.96
2.08
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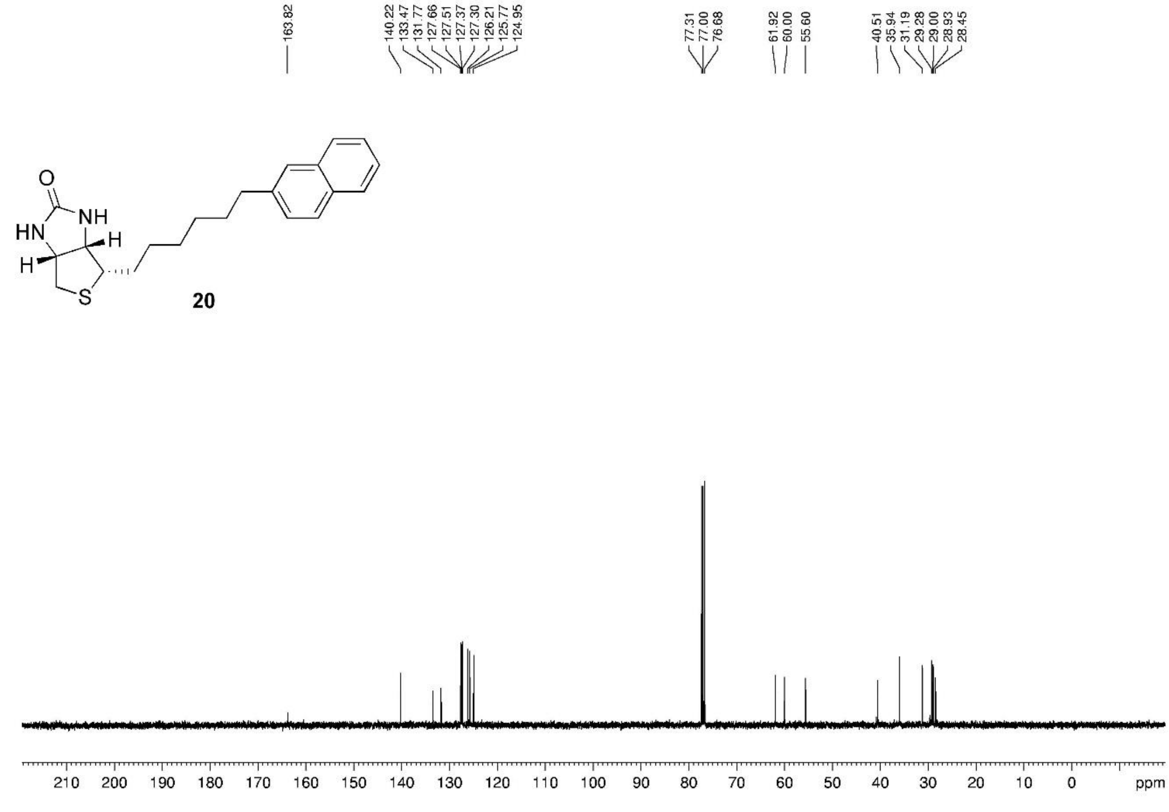
6.29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124.95
77.31
77.00
76.68
61.92
60.00
55.60
40.51
35.94
31.19
29.28
29.00
28.93
28.45
O
NH
HN
H
H
S
20
210
200
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130
120
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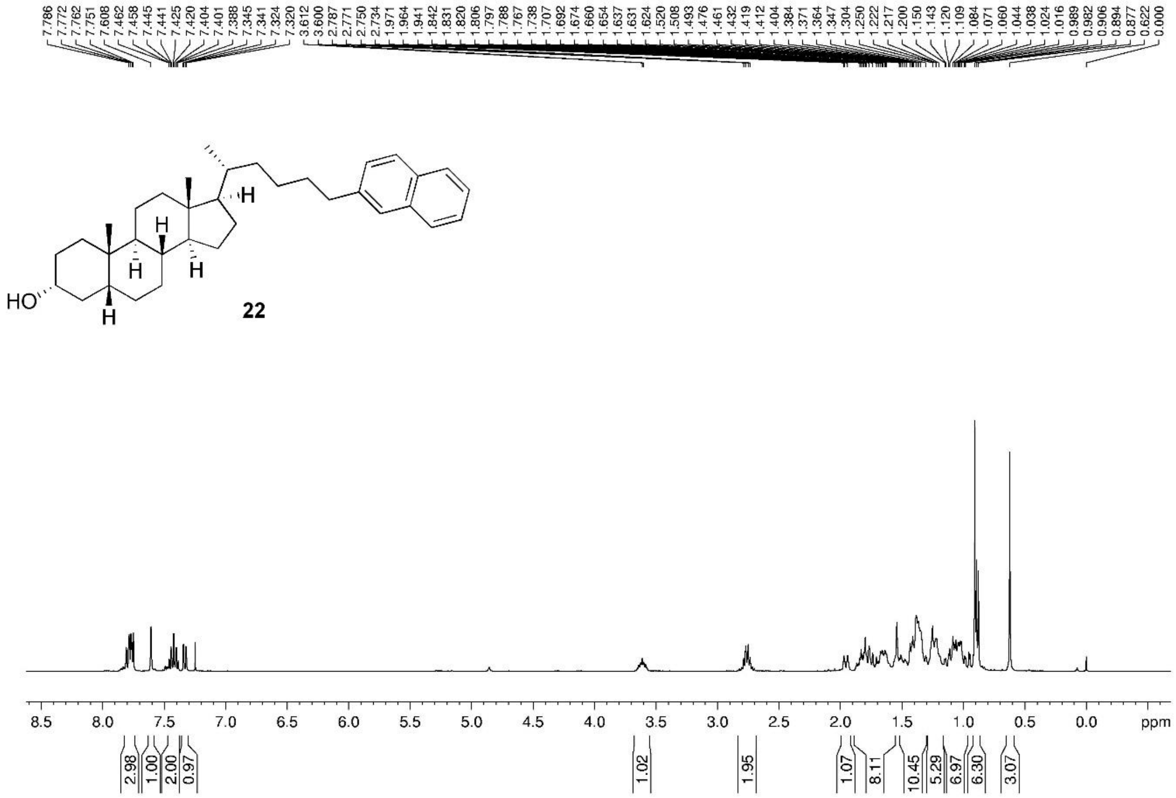
0
ppm


22

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7.772
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7.458
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7.441
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7.404
7.401
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7.341
7.324
7.320
3.612
3.600
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2.771
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1.820
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1.767
1.738
1.707
1.692
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1.637
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1.476
1.461
1.432
1.419
1.412
1.404
1.384
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1.364
1.347
1.304
1.250
1.222
1.217
1.200
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0.877
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H
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7.0
6.5
6.0
5.5
5.0
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3.5
3.0
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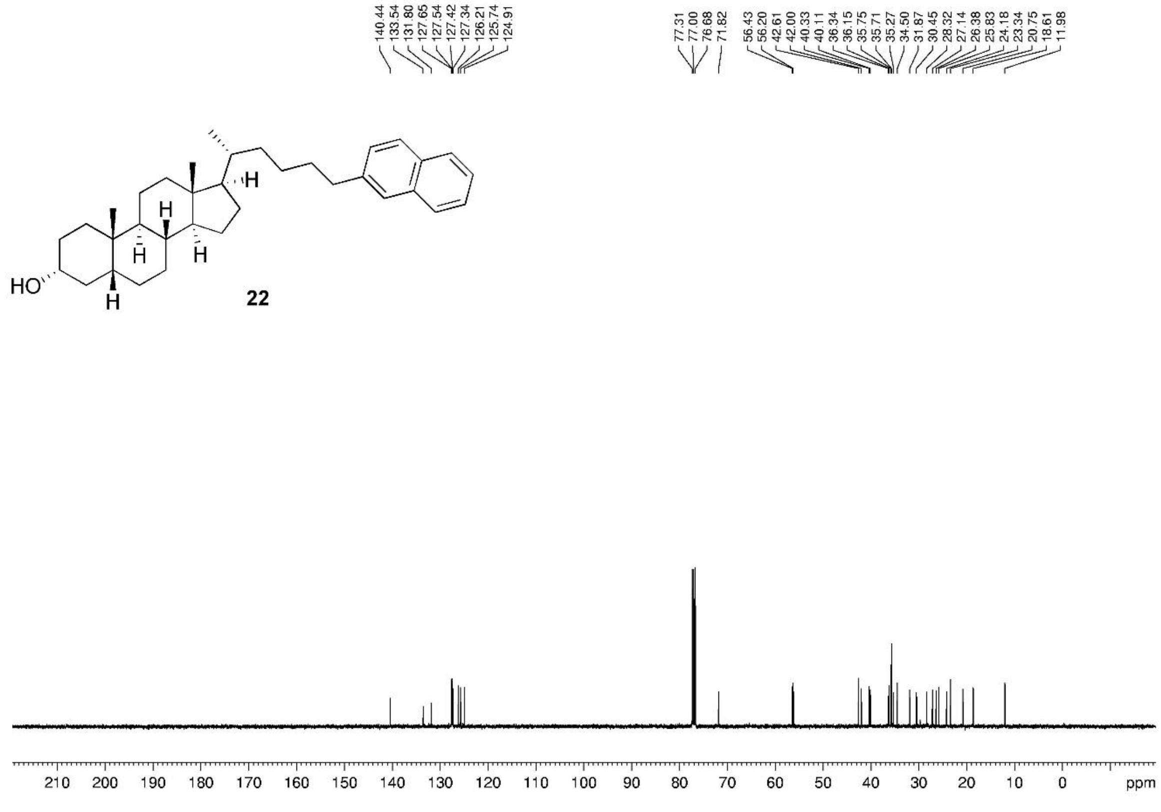
22

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35.71
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28.32
27.14
26.38
25.83
24.18
23.34
20.75
18.61
11.98
HO
H
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200
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20
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0
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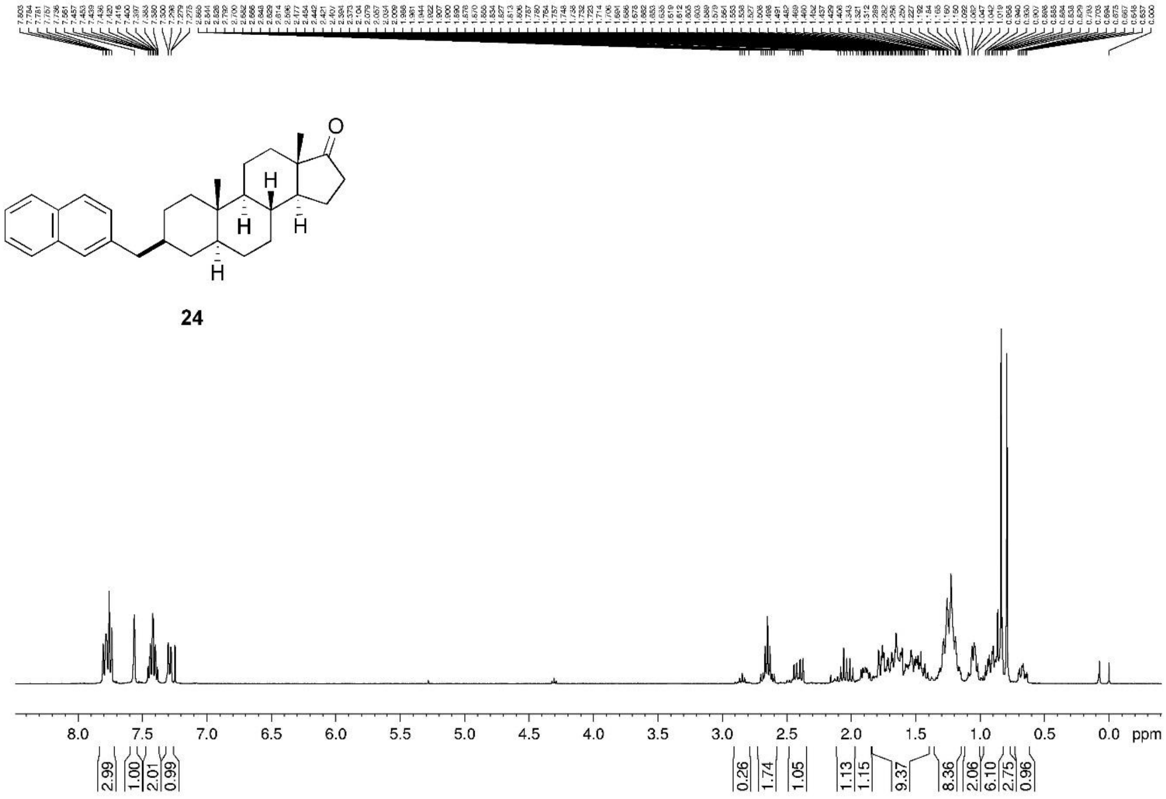
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0.675
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0.648
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0.000
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H
H
H
H
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6.5
6.0
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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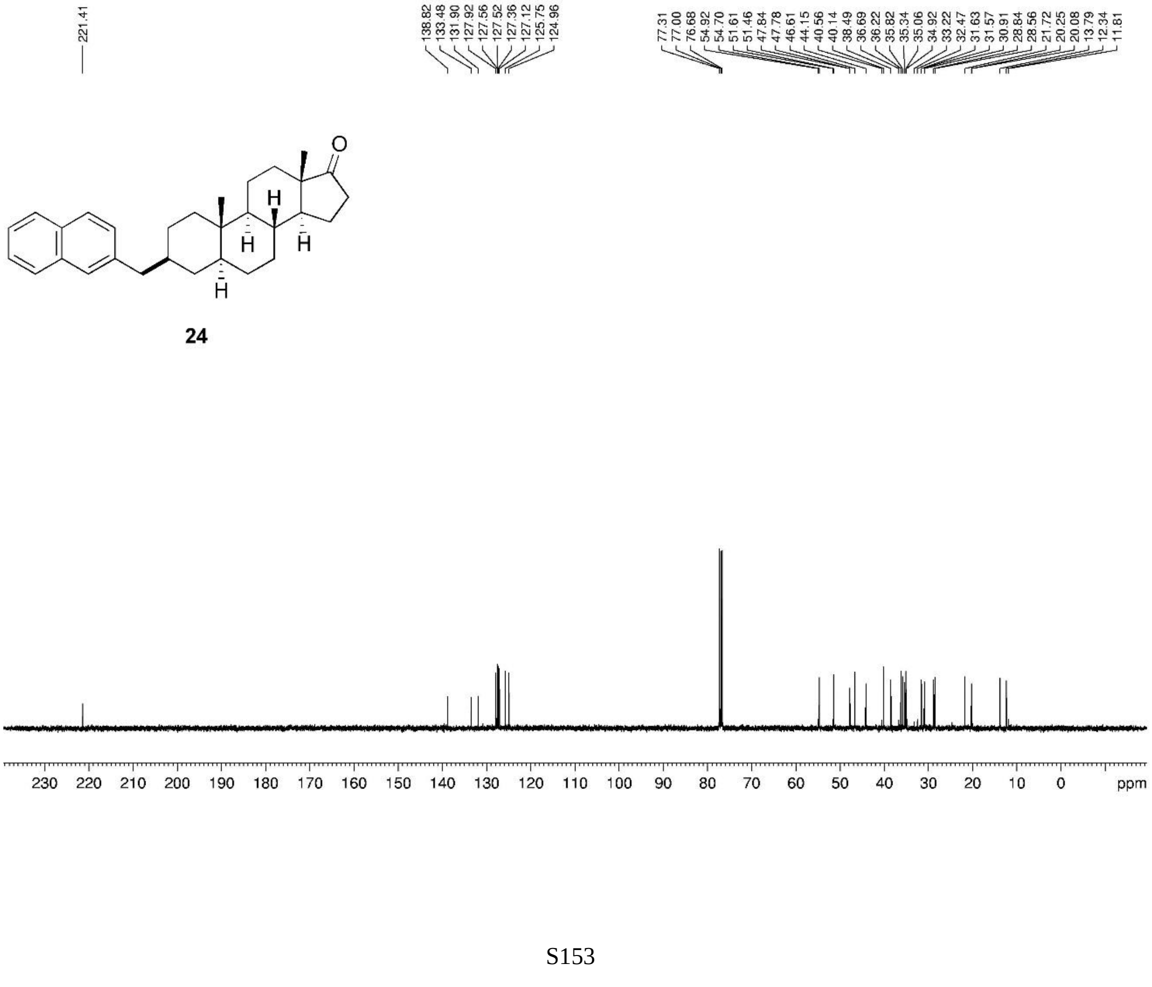
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47.78
46.61
44.15
40.56
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38.49
36.69
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35.82
35.34
35.06
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32.47
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30.91
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28.56
21.72
20.25
20.08
13.79
12.34
11.81
O
H
H
H
H
24
230
220
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200
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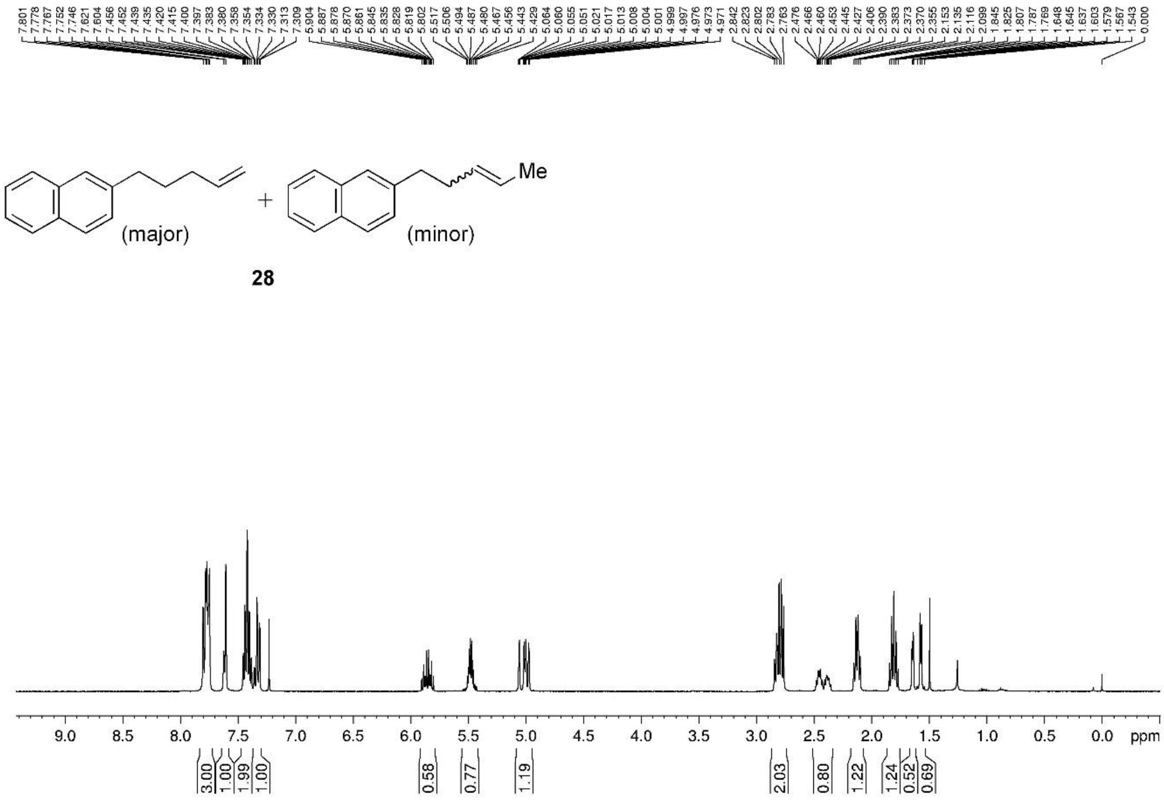
ppm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5.055
5.051
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5.017
5.013
5.008
5.004
5.001
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Me
(major)
+
(minor)
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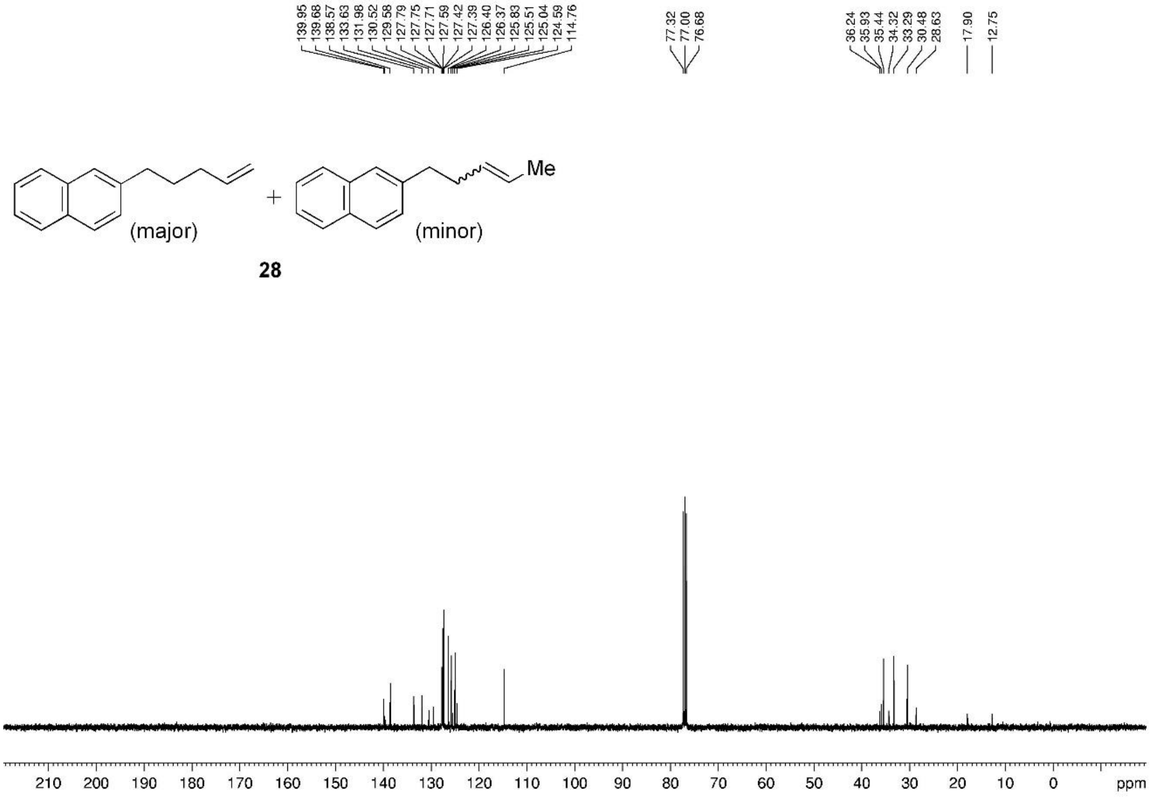
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126.40
126.37
125.83
125.51
125.04
124.59
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77.32
77.00
76.68
36.24
35.93
35.44
34.32
33.29
30.48
28.63
17.90
12.75
Me
(major)
+
(minor)
28
210
200
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100
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60
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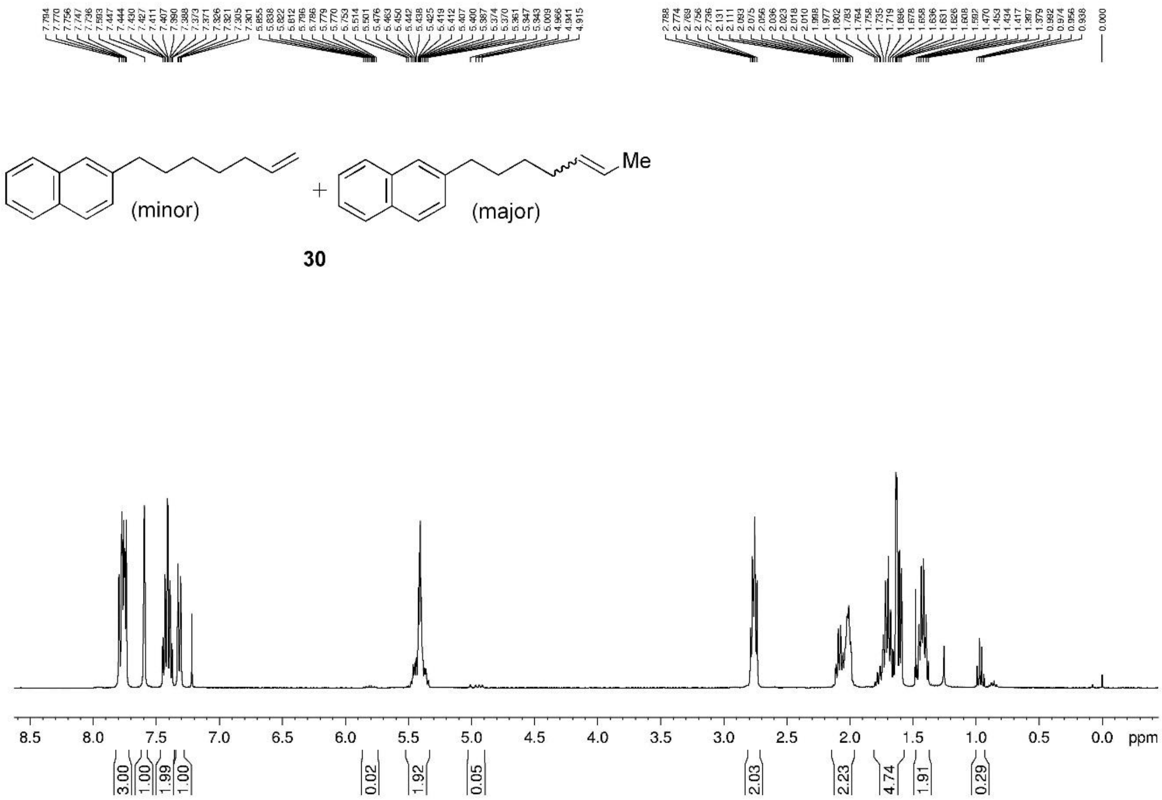
ppm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1.434
1.417
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1.379
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(minor)
+
Me
(major)
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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1.00
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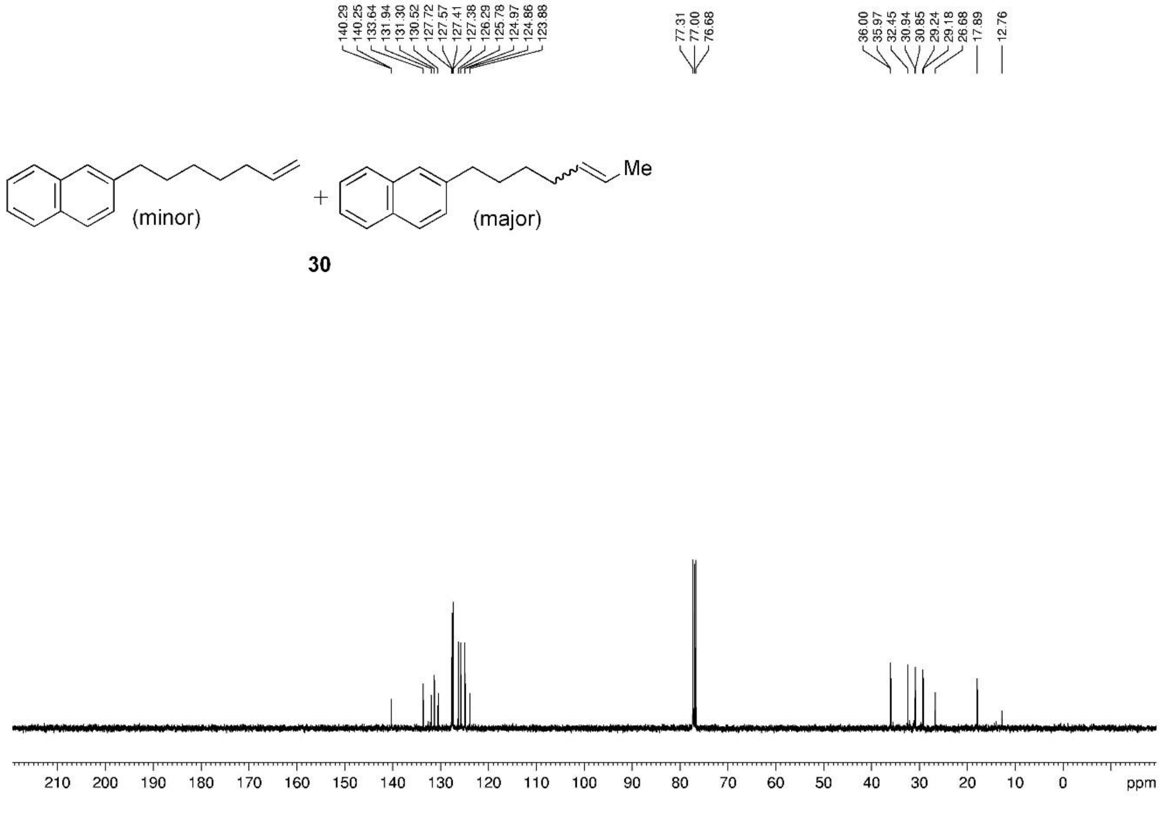
1.00
0.02
1.92
0.05
2.03
2.23
4.74
1.91
0.29

30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140.25
133.64
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131.30
130.52
127.72
127.57
127.41
127.38
126.29
125.78
124.97
124.86
123.88
77.31
77.00
76.68
36.00
35.97
32.45
30.94
30.85
29.24
29.18
26.68
17.89
12.76
(minor)
+
Me
(major)
210
200
190
180
170
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140
130
120
110
100
90
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50
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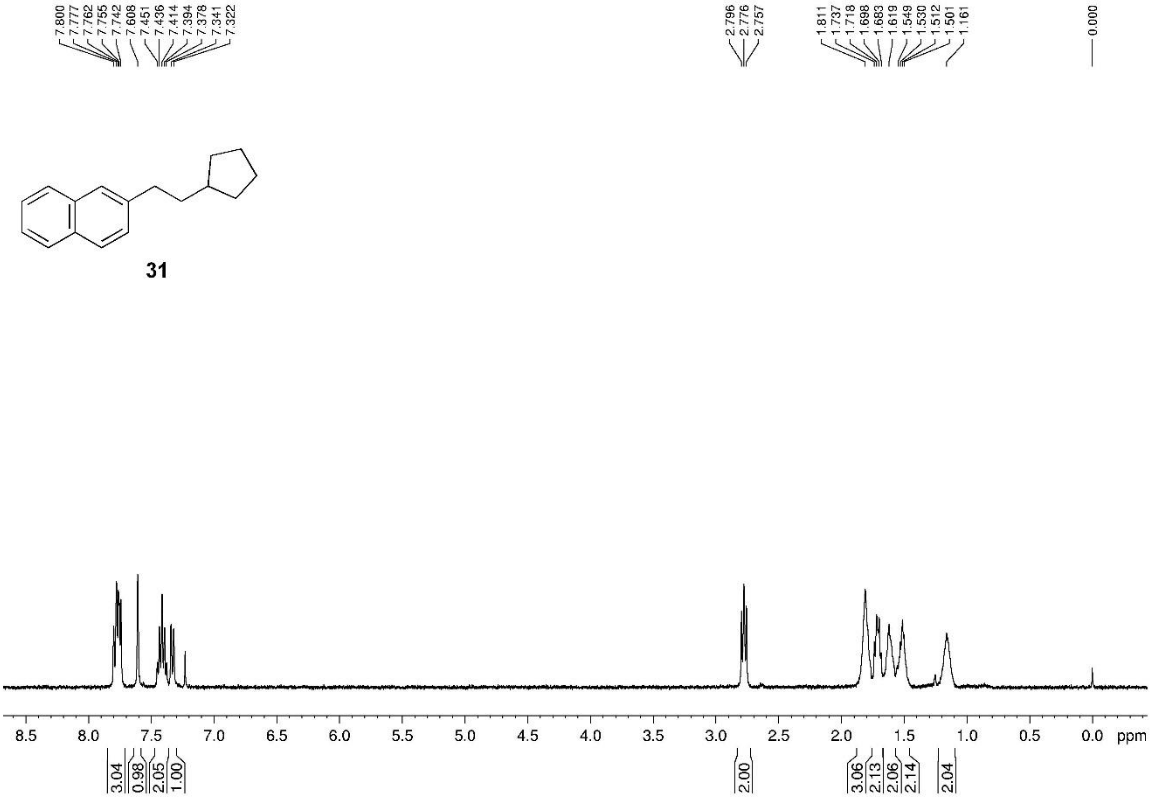
0
ppm

30



31

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7.608
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7.414
7.394
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7.341
7.322
2.796
2.776
2.757
1.811
1.737
1.718
1.698
1.683
1.619
1.549
1.530
1.512
1.501
1.161
0.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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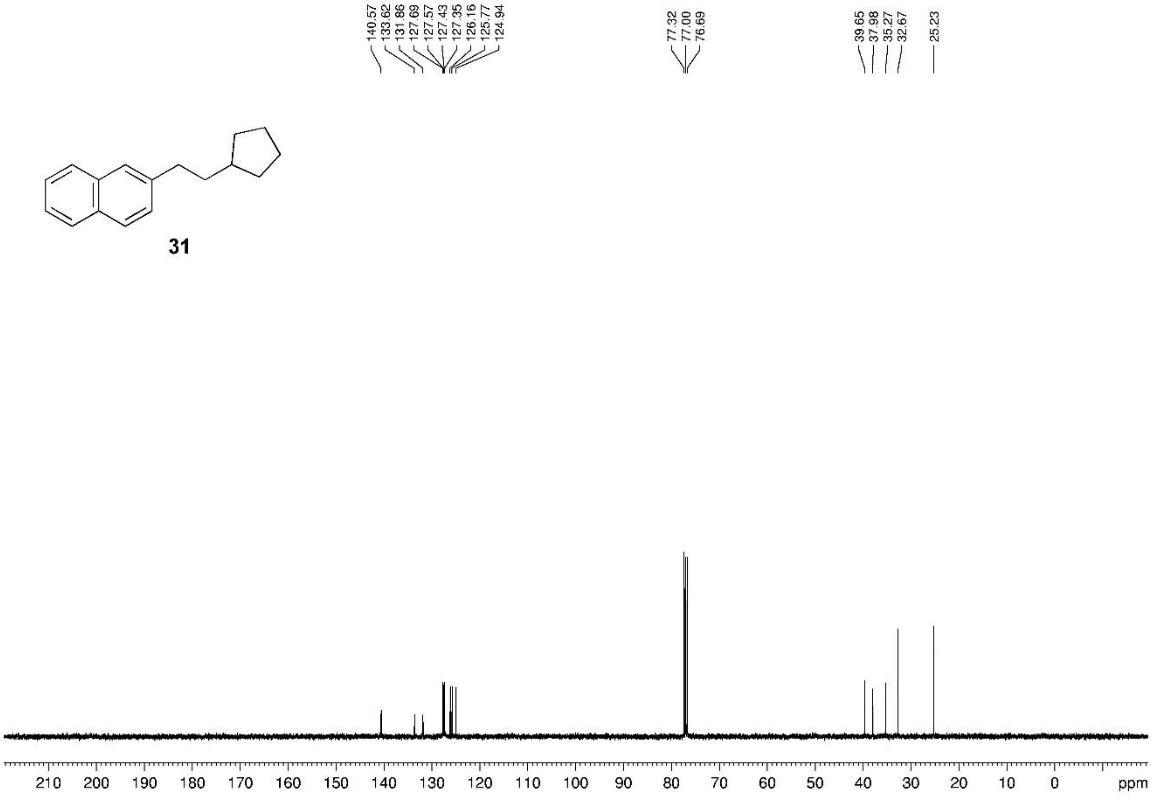
2.04



31

140.57
133.62
131.86
127.69
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127.43
127.35
126.16
125.77
124.94
77.32
77.00
76.69
39.65
37.98
35.27
32.67
25.23
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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40
30
20
10
0
ppm