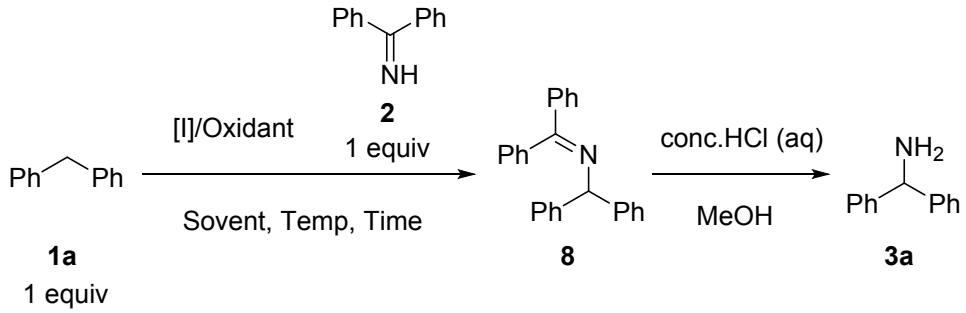
Supporting information

Optimization of reaction conditions for amination of diarylmethanes 1

Table 1s. Optimization of reaction conditions for amination of diarylmethanes **1**.



Entry	[I] or [M]	Oxidant	Solvent	Temp	Time (h)	Yield%[b]
1	$FeBr_3$	DTBP	-	90ºC	48	79
2	Bu_4NI	TBHP[d]	H_2O	100ºC	24	35
3	Bu_4NI	DTBP	H_2O	100ºC	24	0
4	Bu_4NI	H_2O_2	H_2O	100ºC	24	0
5	Bu_4NBr	TBHP[d]	H_2O	100ºC	24	0
6	NIS	TBHP[d]	H_2O	100ºC	24	66
7	KI	TBHP[d]	H_2O	100ºC	24	67
8	PhI	TBHP[d]	H_2O	100ºC	24	0
9	I_2	TBHP[d]	H_2O	100ºC	24	44
10	-	TBHP[d]	H_2O	100ºC	24	0
11[c]	KI	TBHP[d]	H_2O	100ºC	48	80

[a] Unless otherwise noted, reactions were carried out with diphenylmethane **1a** (1 mmol), benzophenone imine **2** (1 mmol), "I" (0.1 mmol) and oxidant (2 mmol).

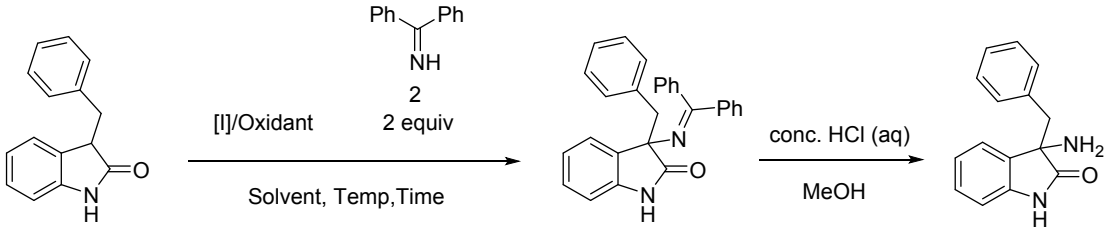
[b] Isolated yield of two steps.

[c] benzophenone imine **2** (2 mmol), oxidant (3 mmol) were used.

[d] 70% aqueous solution.

Optimization of reaction conditions for 3-substituted indolin-2-ones 4

Table 2s. Optimization of reaction conditions for 3-substituted indolin-2-ones **4**.



4a

1 equiv

9

6a

Entry	[I]	Oxidant	Solvent	Temp	Time（h）	Yield%[b]
1	Bu_4NI	TBHP[d]	Hexane	Reflux	6	79
2	Bu_4NI	TBHP[d]	H_2O	100ºC	6	45
3	Bu_4NI	TBHP[d]	H_2O	70ºC	6	72
4	Bu_4NI	DTBP	H_2O	70ºC	6	0
5	Bu_4NI	H_2O_2	H_2O	70ºC	6	0
6	Bu_4NBr	TBHP[d]	H_2O	70ºC	6	0
7	NIS	TBHP[d]	H_2O	70ºC	6	44
8	KI	TBHP[d]	H_2O	70ºC	6	34
9	PhI	TBHP[d]	H_2O	70ºC	6	0
10	I_2	TBHP[d]	H_2O	70ºC	6	49
11	-	TBHP[d]	H_2O	70ºC	6	0
12[c]	Bu_4NI	TBHP[d]	H_2O	70ºC	12	83

[a] Unless otherwise noted, reactions were carried out with 3-benzylindolin-2-one **4a** (1 mmol), benzophenone imine **2** (1 mmol), "I" (0.1 mmol) and oxidant (2 mmol).

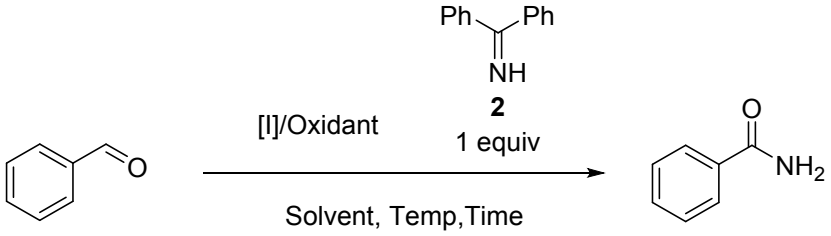
[b] Isolated yield of two steps.

[c] benzophenone imine (2 mmol), oxidant (3 mmol) were used.

[d] 70% aqueous solution.

Optimization of reaction conditions for amination of aldehydes 6

Table 3s. Optimization of reaction conditions for amination of aldehydes **6**.



6a 1 equiv **7a**

Entry	[I] or [M]	Oxidant	Solvent	Temp	Time (h)	Yield%[b]
1	CuBr	TBHP[d]	MeCN	Reflux	12	0
2	Bu_4NI	TBHP[d]	Hexane	Reflux	12	59
3	Bu_4NI	TBHP[d]	H_2O	100ºC	12	60
4	Bu_4NI	DTBP	H_2O	100ºC	12	0
5	Bu_4NI	H_2O_2	H_2O	100ºC	12	0
6	Bu_4NBr	TBHP[d]	H_2O	100ºC	12	27
7	NIS	TBHP[d]	H_2O	100ºC	12	24
8	KI	TBHP[d]	H_2O	100ºC	12	46
9	PhI	TBHP[d]	H_2O	100ºC	12	12
10	I_2	TBHP[d]	H_2O	100ºC	12	59
11	-	TBHP[d]	H_2O	100ºC	12	35
12	Bu_4NI	TBHP[d]	H_2O	50ºC	12	36
13	Bu_4NI	TBHP[d]	H_2O	25ºC	12	<5
14[c]	Bu_4NI	TBHP[d]	H_2O	100ºC	24	81

[a] Unless otherwise noted, reactions were carried out with benzaldehyde **6a** (1 mmol), benzophenone imine **2** (1 mmol), "I" (0.1 mmol) and oxidant (2 mmol).

[b] Isolated yield of two steps.

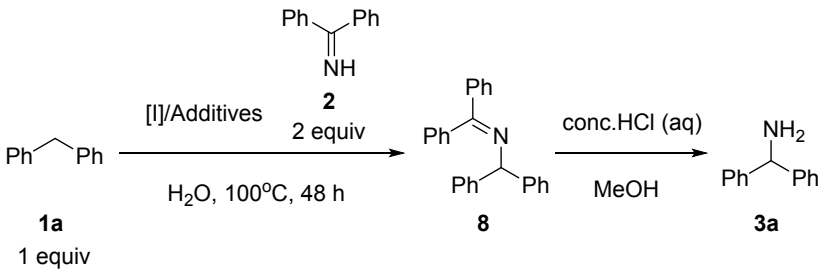
[c] benzophenone imine (2 mmol), oxidant (3 mmol) were used.

[d] 70% aqueous solution.

Control experiments for amination of diarylmethane 1a

When equivalent TEMPO (a radical-trapping reagent) was added in the amination of diphenylmethane **1a**, the desired amine product **3a** was obtained in trace quantity (Table 1, entry 1). The amination of **1a** did not occur in the presence of KI, I_2, KIO_3 or KIO_4 under neutral conditions (Table 1, entries 2, 3, 5 and 6). In contrast, desired product **3a** was obtained in 78% yield in the presence of I_2 under basic condition (Table1, entry 4). Hypoiodite anion (IO^-) might be generated from I_2 under basic condition, and then might be disproportioned to iodite anion (IO_2^-) and iodide anion (I^-).[1] Hence, hypoiodite (+1) or iodite (+3) might be catalytic species for our oxidative amination of diarylmethanes **1**.

Table 4s. Control experiments for amination of diarylmethane **1a**.



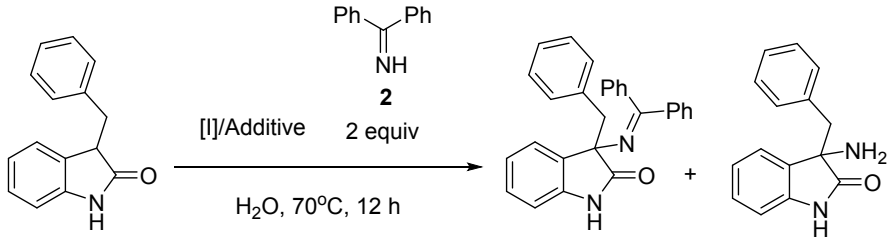
Entry	[I]	Oxidant	Additives	Yield% (**3a**)[b]
1	KI (0.1 eq)	TBHP (3 eq)	TEMPO (1 eq)	Trace
2	KI (1 eq)	-	-	0
3	I_2 (1 eq)	-	-	0
4	I_2 (1 eq)	-	KOH (2 eq)	78
5	KIO_3 (1 eq)	-	-	0
6	KIO_4 (1 eq)	-	-	0

[a] Unless otherwise noted, reactions were carried out with diphenylmethane **1a** (1 mmol) and benzophenone imine **2** (2 mmol).
[b] Isolated yield.

Control experiments for amination of 3-substituted indolin-2-one 4a

In the presence of equivalent TEMPO, the desired product **5a** and the imine intermediate **9** were obtained in trace quantity employing **4a** as starting material (Table 2, entry 1). No product was observed in the presence of TBAI under neutral condition. On the contrary, amination occured in the presence of I_2 under neutral condition and primary amine **5a** was obtained in 75% yield (Table 2, entry 3). Notably, in the presence of I_2 under basic condition, imine **9** rather than primary amine **5a** was generated (Table 2, entry 4) which probably resulted from elimination of hydrogen iodide under basic condition so that the imine **9** was not hydrolyzed. Iodate (+5) and periodate (+7) were inert under both neutral and basic conditions (Table 2, entries 5, 6, 7 and 8). Therefore, iodine (0), hypoiodite (+1) or iodite (+3) may be catalytic species for our oxidative amination of 3-substituted indolin-2-ones **4**.

Table 5s. Control experiments for amination of 3-substituted indolin-2-one **4a**.



4a 1 equiv | **9** | **5a**

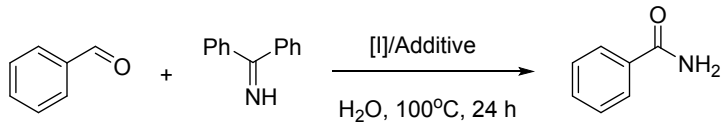
Entry	[I]	Oxidant	Additives	Yield%(**9**/**5a**)[b]
1	TBAI (0.1 eq)	TBHP (3 eq)	TEMPO (1 eq)	Trace
2	TBAI (1 eq)	-	-	0/0
3	I_2 (1 eq)	-	-	0/75
4	I_2 (1 eq)	-	Bu_4NOH (2 eq)	72/0
5	$NaIO_3$ (1 eq)	-	-	0/0
6	$NaIO_3$ (1 eq)	-	Bu_4NOH (1 eq)	0/0
7	$NaIO_4$ (1 eq)	-	-	0/0
8	$NaIO_4$ (1 eq)	-	Bu_4NOH (1 eq)	0/0

[a] Unless otherwise noted, reactions were carried out with 3-benzylindolin-2-one **4a** (1 mmol) and benzophenone imine **2** (2 mmol).
[b] Isolated yield.

Control experiment for amidation of aldehyde 6a

The radical-trapping experiment employing TEMPO suggested that a radical intermediate might be involved in our amidation of aldehydes (Table 3, entry 1). No product was observed in the presence of TBAI or I_2 under neutral condition (Table 3, entries 2 and 3). Hypoiodite (+1) and iodite (+3) were inert pre-catalyst for this reaction (Table 3， entry 4). On the contrary, in the presence of iodate (+5) and periodate (+7) amidation occured under basic conditions (Table 3，entries 6 and 8) in 27% and 49% yields, respectively. Thus, iodate (+5) and periodate (+7) might be catalytic species which promoted our oxidative amidation of aldehyde **6a**.

Table 6s. Control experiment for amidation of aldehyde **6a**.



6a 1 equiv　　**2** 2 equiv　　**7a**

Entry	[I](1 eq)	Oxidant	Additives	Yield%[b]
1	TBAI (0.1 eq)	TBHP (3 eq)	TEMPO (1 eq)	Trace
2	TBAI (1 eq)	-	-	0
3	I_2 (1 eq)	-	-	0
4	I_2 (1 eq)	-	Bu_4NOH (2 eq)	0
5	$NaIO_3$ (1 eq)	-	-	0
6	$NaIO_3$ (1 eq)	-	Bu_4NOH (1 eq)	27
7	$NaIO_4$ (1 eq)	-	-	0
8	$NaIO_4$ (1 eq)	-	Bu_4NOH (1 eq)	49

[a] Unless otherwise noted, reactions were carried out with benzaldehyde **6a** (1 mmol) and benzophenone imine **2** (2 mmol).
[b] Isolated yield.

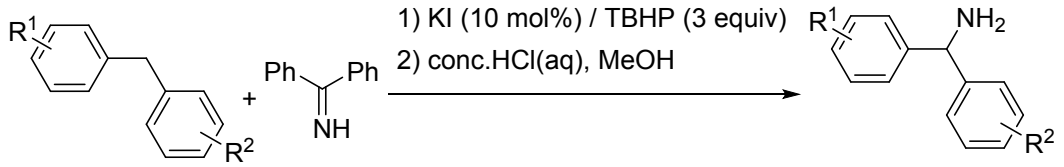
General methods

The reagents (chemicals) were purchased from J&K, Energy Chemical, Adamas, Accela and Shanghai Chemical Reagent Co. and used without further purification. Analytical thin-layer chromatography (TLC) was HSGF 254 (150−200 μm thickness; Yantai Huiyou Co., China). Nuclear magnetic resonance (NMR) spectroscopy was performed on a Bruker AMX-400 NMR (IS as TMS). Chemical shifts were reported in parts per million (ppm, δ) downfield from tetramethylsilane. Proton coupling patterns were described as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), and broad (br). High-resolution mass spectral analysis (HRMS) was performed on Thermo Fisher Scientific LTQ FT Ultra mass spectrometer at Shanghai Institute of Organic Chemistry, Chinese Academic of Sciences.

Preparation for the starting materials (1a-m and 4a-r)

The starting materials (**6a-p**) were purchased from J&K, Energy Chemical, Adamas, Accela and Shanghai Chemical Reagent Co. and used without further purification. **1a-m** and **4a-r** were prepared as the previously reported methods in the literatures. [2, 3]

General procedure for the amination of diarylmethanes (1)

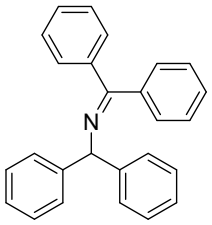


1a-m 1 equiv　　**2** 2 equiv　　**3a-m**

To a mixture of diarlymethane **1** (1 mmol), KI (16.6 mg, 0.1 mmol, 10 mmol%) and benzophenone imine **2** (362.2 mg, 2 mmol) was added TBHP (70% in water, 386 mg, 3 mmol) at room temperature. After stirring at 100 °C for 48 h, the reaction mixture was poured into saturated $Na_2S_2O_3$ (aqueous solution, 5 mL), extracted with ethyl acetate (3×5 mL) and washed with brine. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed in vacuo. The residue was dissolved in methanol (5 mL) without further purification. To the solution was added conc. HCl (aqueous solution, 0.5 mL).the reaction mixture was stirred at 50 °C. The reaction was monitored by TLC analysis. After the completion, solvents were removed in vacuo and 10 mL of water was added to the residue. The mixture was washed with diethyl ether (3×3 mL). The aqueous layer was separated and basified with saturated $NaHCO_3$ (aqueous solution) until pH=9. Then the mixture was extracted with ethyl acetate (3×5 mL) and washed with brine. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed in vacuo to afford analytically

pure primary amine **3**.

Characterization of intermediates 8

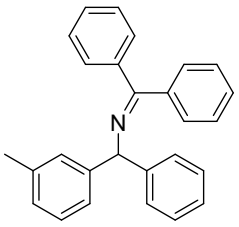


N-benzhydryl-1,1-diphenylmethanimine (8a)

^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 7.71 (d, *J* = 6.8 Hz, 2H), 7.62-7.55 (m, 3H), 7.49 (dt, *J* = 14.2, 6.7 Hz, 3H), 7.35 (d, *J* = 4.4 Hz, 8H), 7.26 (dq, *J* = 8.6, 4.1 Hz, 2H), 7.10 (dd, *J* = 6.4, 2.8 Hz, 2H), 5.54 (s, 1H).

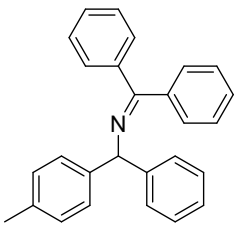
^{13}C NMR (101 MHz, DMSO-d_6) *δ:* 166.48, 144.55, 139.09, 135.79, 130.37, 128.79, 128.69, 128.42, 128.29, 128.14, 127.13, 127.04, 126.73, 69.27.

HRMS (ESI) *m/z* calcd $C_{26}H_{22}N$ $[M+H]^+$ 348.1747, found 348.1749.



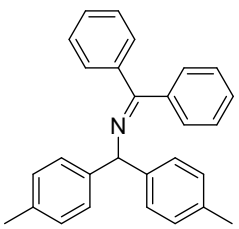
1,1-diphenyl-N-(phenyl(m-tolyl)methyl)methanimine (8b)

MS (ESI) *m/z* calcd $C_{27}H_{24}N$ $[M+H]^+$ 362.2, found 362.0.



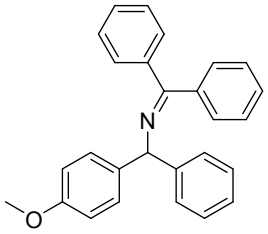
1,1-diphenyl-N-(phenyl(p-tolyl)methyl)methanimine (8c)

MS (ESI) *m/z* calcd $C_{27}H_{24}N$ $[M+H]^+$ 362.2, found 362.0.



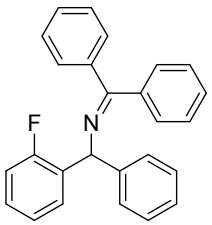
N-(di-p-tolylmethyl)-1,1-diphenylmethanimine (8d)

MS (ESI) *m/z* calcd $C_{28}H_{26}N$ $[M+H]^+$ 376.2, found 376.0.



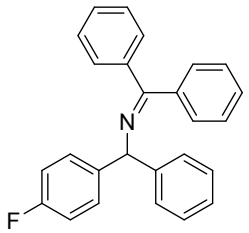
N-((4-methoxyphenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8e)

MS (ESI) *m/z* calcd $C_{27}H_{24}NO$ $[M+H]^+$ 378.2, found 378.0.



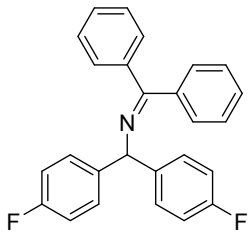
N-((2-fluorophenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8f)

MS (ESI) *m/z* calcd $C_{26}H_{21}FN$ $[M+H]^+$ 366.2, found 366.0.



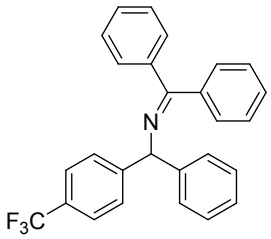
N-((4-fluorophenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8g)

MS (ESI) *m/z* calcd $C_{26}H_{21}FN$ $[M+H]^+$ 366.2, found 366.0.



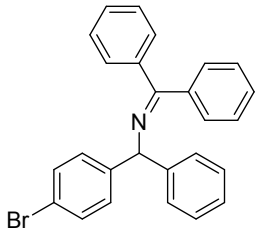
N-(bis(4-fluorophenyl)methyl)-1,1-diphenylmethanimine (8h)

MS (ESI) *m/z* calcd $C_{26}H_{20}F_2N$ $[M+H]^+$ 384.2, found 383.9.



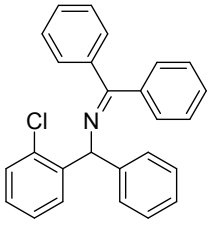
1,1-diphenyl-N-(phenyl(4-(trifluoromethyl)phenyl)methyl)methanimine (8i)

MS (ESI) *m/z* calcd $C_{27}H_{21}F_3N$ $[M+H]^+$ 416.2, found 416.0.



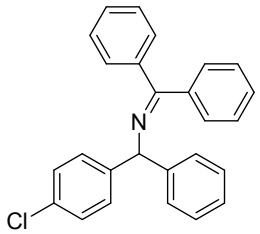
N-((4-bromophenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8j)

MS (ESI) *m/z* calcd $C_{26}H_{21}BrN$ $[M+H]^+$ 426,1, found 425.9.



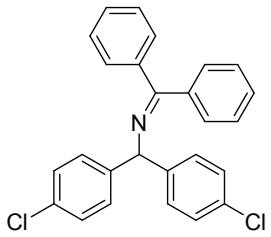
N-((2-chlorophenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8k)

MS (ESI) *m/z* calcd $C_{26}H_{21}ClN$ $[M+H]^+$ 382.1, found 382.0.



N-((4-chlorophenyl)(phenyl)methyl)-1,1-diphenylmethanimine (8l)

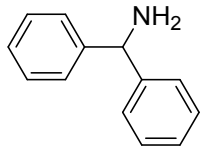
MS (ESI) *m/z* calcd $C_{26}H_{21}ClN$ $[M+H]^+$ 382.1, found 382.0.



N-(bis(4-chlorophenyl)methyl)-1,1-diphenylmethanimine (8m)

MS (ESI) *m/z* calcd $C_{26}H_{20}Cl_2N$ $[M+H]^+$ 416.1, found 415.9.

Characterization of diarylmethylamines (3)

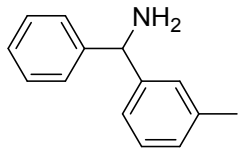


Diphenylmethanamine (3a): 80% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.36 (d, *J* = 7.7 Hz, 4H), 7.29 (t, *J* = 7.5 Hz, 4H), 7.21 (q, *J* = 6.9 Hz, 2H), 5.18 (s, 1H), 1.83 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 145.61, 128.53, 127.00, 126.94, 59.78.

HRMS (ESI) *m/z* calcd $C_{13}H_{14}N$ $[M+H]^+$ 184.1121, found 184.1121.

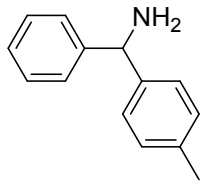


Phenyl(m-tolyl)methanamine (3b): 82% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.37 (d, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.25-7.13 (m, 4H), 7.04 (d, *J* = 7.0 Hz, 1H), 5.19 (s, 1H), 2.52 (s, 2H), 2.32 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 144.30, 144.21, 136.99, 127.39, 127.32, 126.69, 126.52, 125.86, 125.82, 122.89, 58.56, 20.41.

HRMS (ESI) *m/z* calcd $C_{14}H_{16}N$ $[M+H]^+$ 198.1277, found 198.1276.



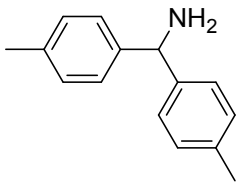
Phenyl(p-tolyl)methanamine (3c): 75% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.37 (d, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 2H), 7.27-7.21 (t,

3H), 7.11 (d, 2H), 5.20 (s, 1H), 2.74 (s, 2H), 2.32 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ:* 144.60, 141.52, 135.45, 128.09, 127.39, 125.82, 125.77, 125.73, 58.35, 19.98.

HRMS (ESI) *m/z* calcd $C_{14}H_{16}N$ $[M+H]^+$ 198.1277, found 198.1278.

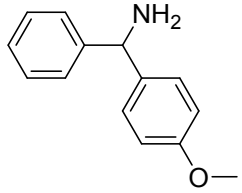


Di-p-tolylmethanamine (3d): 74% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.25 (d, *J* = 9.3 Hz, 4H), 7.10 (d, *J* = 7.8 Hz, 4H), 5.20 (s, 1H), 2.32 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 142.74, 136.52, 129.21, 126.82, 59.19, 21.12.

HRMS (ESI) *m/z* calcd $C_{15}H_{18}N$ $[M+H]^+$ 212.1434, found 212.1433.

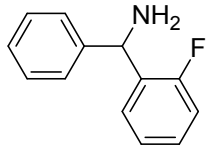


(4-Methoxyphenyl)(phenyl)methanamine (3e): 79% yield. Yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.36 (d, *J* = 7.5 Hz, 2H), 7.30 (dd, *J* = 13.5, 7.9 Hz, 4H), 7.23 (t, *J* = 7.1 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 2H), 5.19 (s, 1H), 3.78 (s, 3H), 2.74 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 157.45, 144.64, 136.59, 127.37, 126.90, 125.79, 125.72, 112.73, 57.96, 54.10.

HRMS (ESI) *m/z* calcd $C_{14}H_{16}NO$ $[M+H]^+$ 214.1226, found 214.1226.



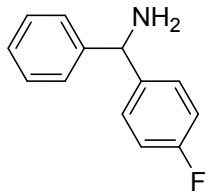
(2-Fluorophenyl)(phenyl)methanamine (3f): 58% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.46 (t, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.22 (dd, *J* = 12.2, 6.6 Hz, 2H), 7.12 (t, *J* = 7.5 Hz, 1H), 7.05-6.97 (m, 1H), 5.52 (s, 1H), 2.29 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 160.46, 158.02, 143.07, 131.62, 131.48, 127.52, 127.43, 126.95, 126.91, 126.06, 125.75, 123.24, 123.21, 114.51, 114.29, 52.08, 52.05.

^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -118.36.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}NF$ $[M+H]^+$ 202.1027, found 202.1026.



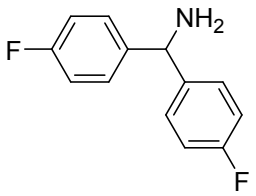
(4-Fluorophenyl)(phenyl)methanamine (3g): 85% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.33 (dd, *J* = 16.4, 7.7 Hz, 6H), 7.24 (s, 1H), 6.99 (t, *J* = 8.4 Hz, 2H), 5.24 (s, 1H), 2.65 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 161.97, 159.54, 144.00, 139.88, 129.01, 127.52, 127.47, 127.39, 127.22, 126.10, 125.73, 114.27, 114.06, 57.91.

^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -115.73.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}NF$ $[M+H]^+$ 202.1027, found 202.1026.



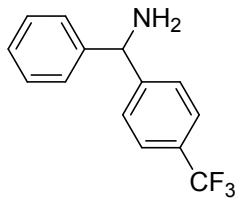
Bis(4-fluorophenyl)methanamine (3h): 83% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.32 (dd, *J* = 8.6, 5.5 Hz, 4H), 7.00 (t, *J* = 8.7 Hz, 4H), 5.20 (s, 1H), 1.81 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 162.02, 159.58, 140.06, 140.03, 127.34, 127.26, 114.37, 114.16, 57.35.

^{19}F NMR (376 MHz, $CDCl_3$) *δ* -115.7.

HRMS (ESI) *m/z* calcd $C_{13}H_{12}NF_2$ $[M+H]^+$ 220.0932, found 220.0931.



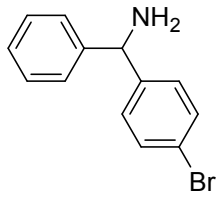
Phenyl(4-(trifluoromethyl)phenyl)methanamine (3i): 70% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.61-7.49 (m, 4H), 7.33 (dt, *J* = 15.8, 8.0 Hz, 5H), 5.31 (s, 1H), 2.27 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 148.16, 143.53, 129.05, 127.69, 127.26, 126.39, 126.21, 125.84, 124.46, 124.42, 124.38, 124.35, 58.41.

^{19}F NMR (376 MHz, DMSO-d_6) *δ*: -67.14.

HRMS (ESI) *m/z* calcd $C_{14}H_{13}NF_3$ $[M+H]^+$ 252.0995, found 252.0994.

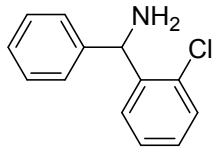


(4-Bromophenyl)(phenyl)methanamine (3j): 82% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.43 (d, *J* = 8.4 Hz, 2H), 7.36-7.29 (m, 4H), 7.26 (t, *J* = 7.0 Hz, 4H), 5.18 (s, 1H), 2.14 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 145.10, 144.56, 131.57, 128.74, 128.68, 127.27, 126.86, 120.81, 59.24.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}NBr$ $[M+H]^+$ 262.0226, found 262.0225.

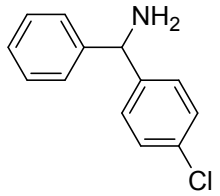


(2-Chlorophenyl)(phenyl)methanamine (3k): 54% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.53 (d, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 2H), 7.33 (dd, *J* = 12.7, 7.1 Hz, 3H), 7.25 (s, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 5.66 (s, 1H), 2.33 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 143.80, 142.81, 133.19, 132.47, 130.11, 129.68, 128.46, 128.35, 128.32, 128.23, 127.23, 127.14, 127.12, 55.87.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}NCl$ $[M+H]^+$ 218.0731, found 218.0732.

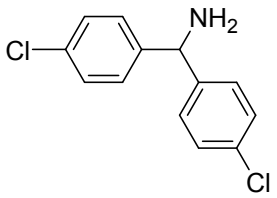


(4-Chlorophenyl)(phenyl)methanamine (3l): 84% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.36-7.29 (m, 6H), 7.25 (dd, *J* = 14.8, 7.8 Hz, 3H), 5.20 (s, 1H), 2.18 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 145.16, 144.03, 132.67, 128.67, 128.62, 128.36, 127.26, 126.86, 59.18.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}NCl$ $[M+H]^+$ 218.0731, found 218.0729.



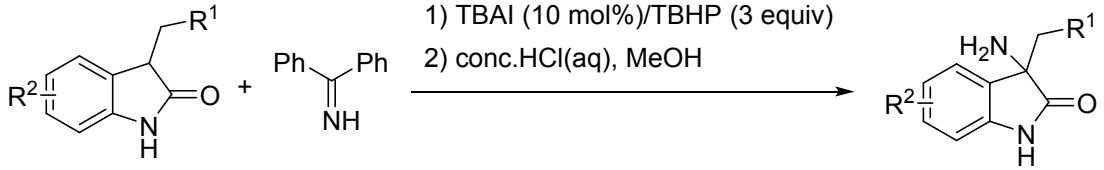
Bis(4-chlorophenyl)methanamine (3m): 73% yield. Light yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.29 (s, 8H), 5.17 (s, 1H), 1.72 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 143.60, 132.91, 128.72, 128.20, 58.57.

HRMS (ESI) *m/z* calcd $C_{13}H_{12}NCl_2$ $[M+H]^+$ 252.0341, found 252.0340.

General procedure for the amination of 3-substituted indolin-2-ones (4)



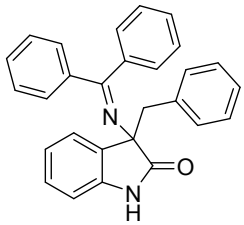
4a-r 1 equiv

2 2 equiv

5a-r

To a mixture of 3-substituted indolin-2-ones **4** (1 mmol), TBAI (36.9 mg, 0.1 mmol, 10 mmol%) and benzophenone imine **2** (362.2 mg, 2 mmol) was added TBHP (70% in water, 386 mg, 3 mmol) at room temperature. After stirring at 70 ^{o}C for 12 h, the reaction mixture was poured into saturated $Na_2S_2O_3$ (aqueous solution, 5 mL), extracted with ethyl acetate (3×5 mL) and washed with brine. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed in vacuo. The residue was dissolved in methanol (5 mL) without further purification. To the solution was added conc. HCl (aqueous solution, 0.5 mL).the reaction mixture was stirred at 50 ^{o}C. The reaction was monitored by TLC analysis. After the completion, solvents were removed in vacuo and 10 mL of water was added to the residue. The mixture was washed with diethyl ether (3×3 mL). The aqueous layer was separated and basified with saturated $NaHCO_3$ (aqueous solution) until pH=9. Then the mixture was extracted with ethyl acetate (3×5 mL) and washed with brine. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed in vacuo to afford analytically pure primary amine **5**.

Characterization of intermediates 9

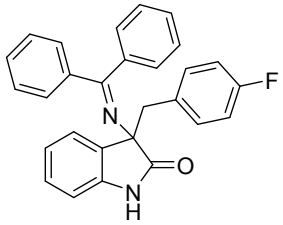


3-benzyl-3-((diphenylmethylene)amino)indolin-2-one (9a)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.59 (s, 1H), 7.57-7.49 (m, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.19 (t, J = 7.5 Hz, 1H), 7.15-6.94 (m, 8H), 6.90 (d, J = 6.7 Hz, 1H), 6.87-6.79 (m, 1H), 6.46 (s, 2H), 6.20 (d, J = 7.7 Hz, 1H), 3.65 (d, J = 12.7 Hz, 1H), 3.27 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 177.92, 169.43, 141.37, 139.72, 135.90, 135.39, 132.91, 130.56, 130.47, 128.12, 128.08, 127.99, 127.74, 127.36, 127.21, 126.98, 126.36, 123.97, 120.95, 109.11, 70.34, 47.08.

HRMS (ESI) *m/z* calcd $C_{28}H_{23}N_2O$ $[M+H]^+$ 403.1805, found 403.1810.

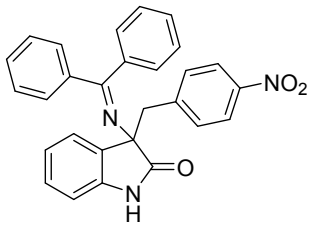


3-((diphenylmethylene)amino)-3-(4-fluorobenzyl)indolin-2-one (9b)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.61 (s, 1H), 7.56-7.48 (m, 2H), 7.48-7.42 (m, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.19 (t, J = 7.5 Hz, 1H), 6.98 (tdd, J = 25.2, 19.3, 14.2 Hz, 8H), 6.87-6.81 (m, 1H), 6.43 (s, 2H), 6.22 (d, J = 7.7 Hz, 1H), 3.62 (d, J = 12.8 Hz, 1H), 3.26 (d, J = 12.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 177.85, 169.59, 141.32, 139.66, 135.89, 132.80, 132.31, 132.23, 131.57, 131.54, 130.50, 128.17, 128.13, 127.99, 127.77, 127.35, 126.99, 123.94, 121.05, 114.06, 113.85, 109.15, 70.27, 46.08.

HRMS (EI) *m/z* calcd $C_{28}H_{21}FN_2O$ (M^+) 420.1638, found 420.1639.

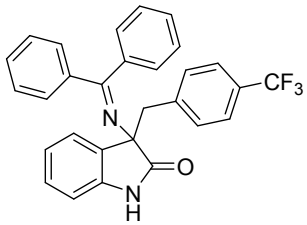


3-((diphenylmethylene)amino)-3-(4-nitrobenzyl)indolin-2-one (9c)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.73 (s, 1H), 8.04 (d, J = 8.6 Hz, 2H), 7.52 (d, J = 7.3 Hz, 2H), 7.46 (t, J = 7.2 Hz, 1H), 7.39 (t, J = 7.3 Hz, 2H), 7.31 (d, J = 8.6 Hz, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.03 (t, J = 7.3 Hz, 3H), 6.87 (q, J = 7.1 Hz, 2H), 6.46 (s, 2H), 6.25 (d, J = 7.7 Hz, 1H), 3.73 (d, J = 12.5 Hz, 1H), 3.39 (d, J = 12.6 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.08, 170.47, 146.77, 144.43, 141.65, 140.03, 136.25, 132.96, 132.47, 131.13, 128.93, 128.70, 128.55, 128.36, 127.83, 127.55, 124.54, 122.76, 121.76, 109.83, 70.48, 46.89.

HRMS (EI) *m/z* calcd $C_{28}H_{21}N_3O_3$ (M^+) 447.1583, found 447.1586.

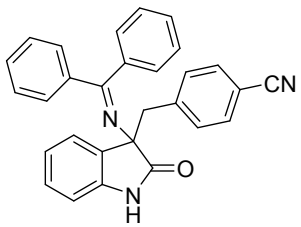


3-((diphenylmethylene)amino)-3-(4-(trifluoromethyl)benzyl)indolin-2-one (9d)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.68 (s, 1H), 7.49 (dt, J = 14.7, 7.1 Hz, 6H), 7.39 (t, J = 7.4 Hz, 2H), 7.21 (dd, J = 12.5, 7.7 Hz, 3H), 7.02 (t, J = 7.5 Hz, 3H), 6.92 (d, J = 7.1 Hz, 1H), 6.86 (t, J = 7.4 Hz, 1H), 6.45 (s, 1H), 6.24 (d, J = 7.7 Hz, 1H), 3.70 (d, J = 12.6 Hz, 1H), 3.37 (d, J = 12.5 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.15, 170.31, 141.73, 140.90, 140.08, 136.30, 133.11, 131.89, 131.07, 128.83, 128.66, 128.52, 128.31, 127.83, 127.74, 127.52, 127.43, 124.53, 124.49, 124.45, 121.68, 109.74, 70.55, 47.09.

HRMS (EI) *m/z* calcd $C_{29}H_{21}F_3N_2O$ (M^+) 470.1606, found 470.1602.

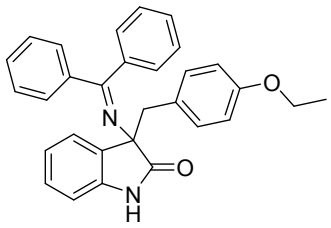


4-((3-((diphenylmethylene)amino)-2-oxoindolin-3-yl)methyl)benzonitrile (9e)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.64 (s, 1H), 7.51 (d, J = 7.3 Hz, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.24 – 7.15 (m, 3H), 7.00 (dd, J = 15.0, 7.9 Hz, 5H), 6.92 (d, J = 7.1 Hz, 1H), 6.85 (t, J = 7.4 Hz, 1H), 6.44 (s, 2H), 6.23 (d, J = 7.7 Hz, 1H), 3.62 (d, J = 12.7 Hz, 1H), 3.27 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.26, 170.16, 141.79, 140.11, 136.35, 134.92, 133.21, 132.83, 131.69, 131.04, 128.74, 128.65, 128.51, 128.29, 127.84, 127.70, 127.51, 124.45, 121.62, 109.69, 70.68, 46.72.

HRMS (EI) *m/z* calcd $C_{29}H_{21}N_3O$ (M^+) 427.1685, found 427.1688.

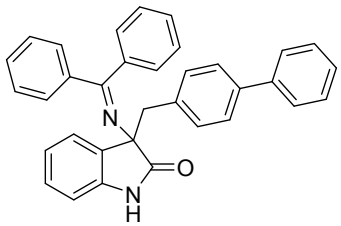


3-((diphenylmethylene)amino)-3-(4-ethoxybenzyl)indolin-2-one (9f)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.56 (s, 1H), 7.50 (d, *J* = 7.6 Hz, 2H), 7.48-7.41 (m, 1H), 7.38 (t, *J* = 7.3 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.00 (dd, *J* = 16.8, 9.2 Hz, 3H), 6.91 (d, *J* = 7.3 Hz, 1H), 6.84 (t, *J* = 8.3 Hz, 3H), 6.64 (d, *J* = 7.6 Hz, 2H), 6.45 (s, 2H), 6.20 (d, *J* = 7.7 Hz, 1H), 3.89 (q, *J* = 6.8 Hz, 2H), 3.57 (d, *J* = 12.8 Hz, 1H), 3.20 (d, *J* = 12.9 Hz, 1H), 1.26 (t, *J* = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.50, 169.85, 157.57, 141.90, 140.22, 136.43, 133.57, 131.98, 130.96, 128.63, 128.53, 128.47, 128.23, 127.84, 127.56, 127.48, 124.42, 121.46, 113.56, 109.57, 70.94, 63.13, 46.78, 15.14.

HRMS (EI) *m/z* calcd $C_{30}H_{26}N_2O_2$ (M^+) 446.1994, found 446.1996.

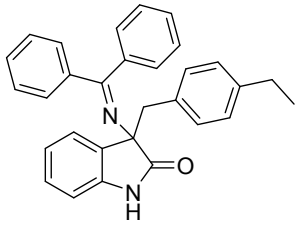


3-([1,1'-biphenyl]-4-ylmethyl)-3-((diphenylmethylene)amino)indolin-2-one (9g)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.63 (s, 1H), 7.60 (d, *J* = 7.4 Hz, 2H), 7.53 (d, *J* = 7.2 Hz, 2H), 7.50-7.35 (m, 7H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.16-6.91 (m, 6H), 6.86 (t, *J* = 7.4 Hz, 1H), 6.46 (s, 2H), 6.22 (d, *J* = 7.7 Hz, 1H), 3.67 (d, *J* = 12.7 Hz, 1H), 3.31 (d, *J* = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.39, 170.01, 141.89, 140.20, 140.15, 138.44, 136.39, 135.29, 133.48, 131.70, 131.02, 129.34, 128.66, 128.51, 128.27, 127.85, 127.71, 127.51, 126.85, 125.88, 124.50, 121.57, 109.67, 70.79, 47.18.

HRMS (EI) *m/z* calcd $C_{34}H_{26}N_2O$ (M^+) 478.2045, found 478.2044.

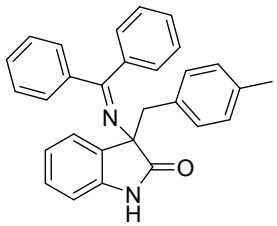


3-((diphenylmethylene)amino)-3-(4-ethylbenzyl)indolin-2-one (9h)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.57 (s, 1H), 7.51 (d, *J* = 7.8 Hz, 2H), 7.45 (t, *J* = 7.1 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.00 (dd, *J* = 16.4, 8.8 Hz, 3H), 6.88 (ddd, *J* = 18.6, 16.6, 7.7 Hz, 6H), 6.43 (s, 2H), 6.20 (d, *J* = 7.7 Hz, 1H), 3.60 (d, *J* = 12.7 Hz, 1H), 3.23 (d, *J* = 12.7 Hz, 1H), 2.50-2.44 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.44, 169.85, 142.04, 141.89, 140.22, 136.40, 133.52, 133.05, 131.00, 128.62, 128.56, 128.48, 128.23, 127.84, 127.48, 127.12, 124.44, 121.45, 109.60, 70.83, 47.23, 28.15, 15.90.

HRMS (EI) *m/z* calcd $C_{30}H_{26}N_2O$ (M^+) 430.2045, found 430.2046.

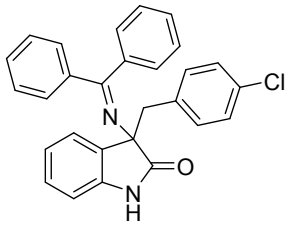


3-((diphenylmethylene)amino)-3-(4-methylbenzyl)indolin-2-one (9i)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.55 (s, 1H), 7.51 (d, *J* = 7.4 Hz, 2H), 7.45 (t, *J* = 7.0 Hz, 1H), 7.37 (t, *J* = 7.4 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.00 (dd, *J* = 17.2, 9.6 Hz, 3H), 6.91 (dd, *J* = 12.7, 7.6 Hz, 3H), 6.84 (t, *J* = 7.0 Hz, 3H), 6.44 (s, 2H), 6.20 (d, *J* = 7.7 Hz, 1H), 3.60 (d, *J* = 12.7 Hz, 1H), 3.24 (d, *J* = 12.7 Hz, 1H), 2.18 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.42, 169.85, 141.89, 140.21, 136.41, 135.71, 133.50, 132.72, 130.97, 130.91, 128.62, 128.56, 128.47, 128.36, 128.23, 127.84, 127.48, 124.43, 121.46, 109.60, 70.88, 47.24, 21.09.

HRMS (EI) *m/z* calcd $C_{29}H_{24}N_2O$ (M^+) 416.1889, found 416.1885.

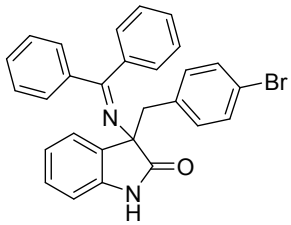


3-(4-chlorobenzyl)-3-((diphenylmethylene)amino)indolin-2-one (9j)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.63 (s, 1H), 7.50 (d, J = 7.4 Hz, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.19 (t, J = 9.1 Hz, 3H), 7.00 (dd, J = 16.0, 8.0 Hz, 5H), 6.92 (d, J = 7.2 Hz, 1H), 6.85 (t, J = 7.4 Hz, 1H), 6.43 (s, 2H), 6.22 (d, J = 7.7 Hz, 1H), 3.61 (d, J = 12.7 Hz, 1H), 3.27 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.25, 170.16, 141.78, 140.11, 136.34, 134.92, 133.21, 132.83, 131.68, 131.05, 130.10, 129.06, 128.74, 128.66, 128.50, 128.30, 127.84, 127.70, 127.51, 124.45, 121.62, 109.69, 70.68, 46.71.

HRMS (EI) *m/z* calcd $C_{28}H_{21}ClN_2O$ (M^+) 436.1342, found 436.1344.

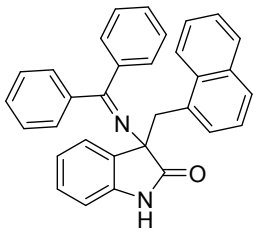


3-(4-bromobenzyl)-3-((diphenylmethylene)amino)indolin-2-one (9k)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.64 (s, 1H), 7.51 (d, J = 7.3 Hz, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.31 (d, J = 8.3 Hz, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.01 (t, J = 7.6 Hz, 3H), 6.93 (d, J = 8.2 Hz, 3H), 6.85 (t, J = 7.4 Hz, 1H), 6.44 (s, 2H), 6.23 (d, J = 7.7 Hz, 1H), 3.60 (d, J = 12.7 Hz, 1H), 3.26 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.24, 170.17, 141.78, 140.11, 136.34, 135.33, 133.23, 133.20, 131.04, 130.61, 130.10, 129.05, 128.75, 128.65, 128.51, 128.29, 127.84, 127.51, 124.45, 121.62, 120.30, 109.70, 70.61, 46.78.

HRMS (EI) *m/z* calcd $C_{28}H_{21}BrN_2O$ (M^+) 480.0837, found 480.0839.

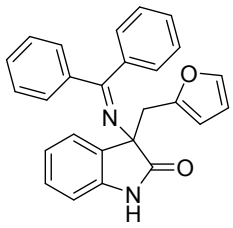


3-((diphenylmethylene)amino)-3-(naphthalen-1-ylmethyl)indolin-2-one (9l)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.72 (s, 1H), 8.12 (d, J = 8.3 Hz, 1H), 7.83 (d, J = 7.8 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.48-7.23 (m, 9H), 7.18 (t, J = 7.4 Hz, 1H), 7.13-6.88 (m, 3H), 6.66 (d, J = 4.2 Hz, 2H), 6.36 (d, J = 79.4 Hz, 2H), 6.25 (d, J = 7.7 Hz, 1H), 4.07 (d, J = 13.5 Hz, 1H), 3.68 (d, J = 13.5 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 179.00, 170.11, 141.71, 140.21, 136.33, 133.68, 133.60, 133.36, 132.64, 130.93, 129.85, 128.61, 128.50, 128.19, 127.93, 127.64, 127.44, 125.80, 125.53, 125.49, 125.10, 125.02, 121.24, 109.67, 70.83, 42.75.

HRMS (EI) *m/z* calcd $C_{32}H_{24}N_2O$ (M^+) 452.1889, found 452.1906.

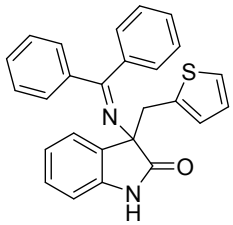


3-((diphenylmethylene)amino)-3-(furan-2-ylmethyl)indolin-2-one (9m)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.76 (s, 1H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.48-7.41 (m, 1H), 7.38 (dd, *J* = 13.2, 5.3 Hz, 3H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.15-6.96 (m, 3H), 6.90-6.79 (m, 2H), 6.49 (s, 2H), 6.34-6.24 (m, 2H), 5.98 (s, 1H), 3.66 (d, *J* = 14.3 Hz, 1H), 3.28 (d, *J* = 14.3 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.19, 170.13, 151.02, 142.00, 141.94, 140.07, 136.19, 133.49, 131.04, 130.10, 129.06, 128.74, 128.63, 128.50, 128.32, 127.79, 127.53, 124.14, 121.70, 110.86, 109.70, 108.53, 69.39.

HRMS (EI) *m/z* calcd $C_{26}H_{20}N_2O_2$ (M^+) 392.1525, found 392.1529.

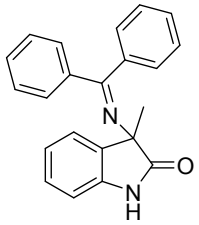


3-((diphenylmethylene)amino)-3-(thiophen-2-ylmethyl)indolin-2-one (9n)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.79 (s, 1H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.46 (t, *J* = 7.1 Hz, 1H), 7.40 (t, *J* = 7.3 Hz, 2H), 7.32 (d, *J* = 5.1 Hz, 1H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.02 (t, *J* = 7.7 Hz, 3H), 6.86 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.77 (t, *J* = 7.5 Hz, 1H), 6.64 (t, *J* = 5.7 Hz, 2H), 6.38 (d, *J* = 47.7 Hz, 2H), 6.31 (d, *J* = 7.7 Hz, 1H), 3.74 (d, *J* = 14.0 Hz, 1H), 3.33 (d, *J* = 14.1 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.25, 170.44, 141.98, 139.92, 138.04, 136.15, 133.14, 131.13, 128.80, 128.65, 128.30, 127.83, 127.54, 126.23, 126.00, 124.25, 121.54, 109.79, 69.88, 41.87.

HRMS (EI) *m/z* calcd $C_{26}H_{20}N_2OS$ (M^+) 408.1296, found 408.1298.

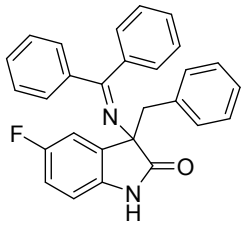


3-((diphenylmethylene)amino)-3-methylindolin-2-one (9o)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.81 (s, 1H), 7.47 (d, J = 7.6 Hz, 2H), 7.43 (t, J = 7.2 Hz, 1H), 7.35 (t, J = 7.5 Hz, 2H), 7.24 (t, J = 7.4 Hz, 1H), 7.16 (d, J = 7.3 Hz, 1H), 7.08 (t, J = 7.6 Hz, 3H), 6.90 (t, J = 7.5 Hz, 1H), 6.43 (d, J = 7.7 Hz, 3H), 1.71 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 179.65, 169.90, 141.24, 140.04, 136.28, 136.22, 130.92, 128.57, 128.51, 128.42, 128.37, 127.90, 127.54, 123.26, 122.03, 109.90, 66.20, 29.35.

HRMS (EI) *m/z* calcd $C_{22}H_{18}N_2O$ (M^+) 326.1419, found 326.1420.

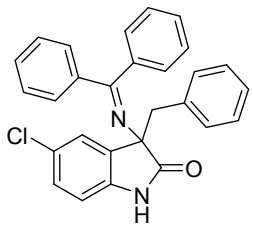


3-benzyl-3-((diphenylmethylene)amino)-5-fluoroindolin-2-one (9p)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.66 (s, 1H), 7.53 (d, J = 7.3 Hz, 2H), 7.46 (t, J = 7.2 Hz, 1H), 7.39 (t, J = 7.4 Hz, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.10 (t, J = 8.5 Hz, 5H), 7.00 (d, J = 2.8 Hz, 2H), 6.83-6.74 (m, 2H), 6.55 (s, 2H), 6.14 (dd, J = 8.3, 4.3 Hz, 1H), 3.67 (d, J = 12.7 Hz, 1H), 3.30 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.39, 170.50, 159.22, 156.87, 140.02, 138.02, 136.36, 135.57, 134.64, 134.56, 131.13, 130.98, 130.10, 129.06, 128.66, 128.57, 128.37, 127.83, 127.60, 127.00, 114.93, 114.69, 112.39, 112.15, 110.22, 110.14, 71.17, 47.42.

HRMS (EI) *m/z* calcd $C_{28}H_{21}FN_2O$ (M^+) 420.1638, found 420.1642.

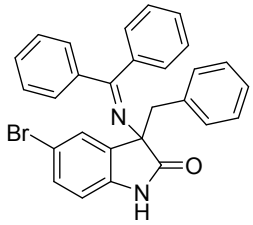


3-benzyl-5-chloro-3-((diphenylmethylene)amino)indolin-2-one (9q)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.78 (s, 1H), 7.54 (d, J = 7.3 Hz, 2H), 7.47 (t, J = 7.2 Hz, 1H), 7.39 (t, J = 7.4 Hz, 2H), 7.21 (t, J = 7.4 Hz, 1H), 7.18-6.95 (m, 8H), 6.90 (d, J = 1.8 Hz, 1H), 6.50 (s, 2H), 6.19 (d, J = 8.3 Hz, 1H), 3.68 (d, J = 12.7 Hz, 1H), 3.28 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.16, 170.53, 140.71, 139.98, 136.34, 135.54, 135.03, 131.16, 131.02, 128.67, 128.59, 128.45, 128.37, 127.83, 127.64, 127.05, 125.47, 124.70, 110.91, 70.91, 47.30.

HRMS (EI) *m/z* calcd $C_{28}H_{21}ClN_2O$ (M^+) 436.1342, found 436.1347.



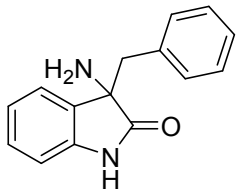
3-benzyl-5-bromo-3-((diphenylmethylene)amino)indolin-2-one (9r)

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.78 (s, 1H), 7.53 (d, J = 7.3 Hz, 2H), 7.47 (t, J = 7.1 Hz, 1H), 7.40 (t, J = 7.4 Hz, 2H), 7.26-7.01 (m, 7H), 7.02-6.96 (m, 3H), 6.51 (s, 2H), 6.15 (d, J = 8.3 Hz, 1H), 3.67 (d, J = 12.7 Hz, 1H), 3.26 (d, J = 12.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 178.03, 170.52, 141.09, 139.98, 136.33, 135.56, 135.44, 131.21, 131.05, 128.69, 128.59, 128.47, 127.83, 127.65, 127.45, 127.06, 113.08, 111.46, 70.84, 47.26.

HRMS (EI) *m/z* calcd $C_{28}H_{21}BrN_2O$ (M^+) 480.0837, found 480.0844.

Characterization of 3-aminoindolin-2-ones (5)

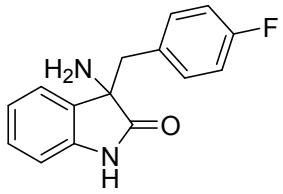


3-Amino-3-benzylindolin-2-one (5a): 83% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.02 (s, 1H), 7.24 (d, J = 7.3 Hz, 1H), 7.13-6.99 (m, 4H), 6.93 (t, J = 7.4 Hz, 1H), 6.84 (dd, J = 6.4, 2.7 Hz, 2H), 6.59 (d, J = 7.7 Hz, 1H), 2.99 (dd, J = 42.5, 12.7 Hz, 2H), 2.21 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.37, 142.14, 136.27, 132.70, 130.34, 128.68, 127.86, 126.68, 124.76, 121.55, 109.58, 62.96, 45.37.

HRMS (ESI) *m/z* calcd $C_{15}H_{15}N_2O$ $[M+H]^+$ 239.1179, found 239.1178.



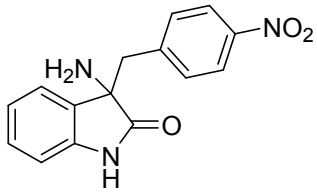
3-Amino-3-(4-fluorobenzyl)indolin-2-one (5b): 75% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.03 (s, 1H), 7.23 (d, J = 7.3 Hz, 1H), 7.10 (t, J = 7.6 Hz, 1H), 6.99-6.80 (m, 5H), 6.61 (d, J = 7.7 Hz, 1H), 2.98 (dd, J = 46.4, 12.8 Hz, 2H), 2.21 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.34, 162.58, 160.17, 142.09, 132.51, 132.44, 132.41, 132.14, 132.06, 128.78, 124.77, 121.62, 114.69, 114.48, 109.62, 62.93, 44.36.

^{19}F NMR (376 MHz, DMSO-d_6) δ: -116.80.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OF$ $[M+H]^+$ 257.1085, found 257.1084.

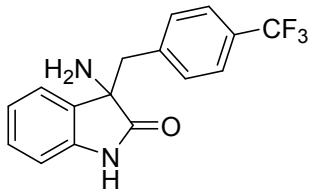


3-Amino-3-(4-nitrobenzyl)indolin-2-one (5c): 64% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.14 (s, 1H), 7.97 (d, J = 8.3 Hz, 2H), 7.22 (t, J = 8.6 Hz, 1H), 7.19-7.07 (m, 3H), 6.95 (t, J = 7.4 Hz, 1H), 6.65 (d, J = 7.6 Hz, 1H), 3.14 (dd, J = 56.8, 12.5 Hz, 2H), 2.37 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.00, 146.61, 144.66, 141.93, 132.04, 131.72, 129.00, 124.86, 122.91, 121.78, 109.79, 62.81, 44.74.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_3O_3$ $[M+H]^+$ 284.1030, found 284.1030.



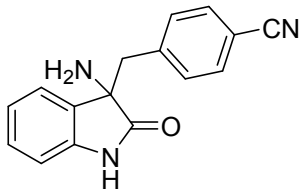
3-Amino-3-(4-(trifluoromethyl)benzyl)indolin-2-one (5d): 70% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.09 (s, 1H), 7.45 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 7.3 Hz, 1H), 7.10 (dd, J = 19.3, 7.8 Hz, 3H), 6.95 (t, J = 7.5 Hz, 1H), 6.63 (d, J = 7.7 Hz, 1H), 3.09 (dd, J = 50.5, 12.6 Hz, 2H), 2.28 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.10, 142.00, 141.22, 132.28, 131.16, 128.92, 127.59, 127.28, 126.15, 124.80, 124.67, 124.64, 123.44, 121.71, 109.69, 62.82, 44.90.

^{19}F NMR (376 MHz, DMSO-d_6) δ: -60.81.

HRMS (ESI) *m/z* calcd $C_{16}H_{14}N_2OF_3$ $[M+H]^+$ 307.1053, found 307.1052.

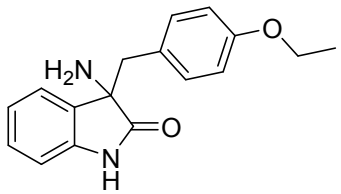


4-((3-Amino-2-oxoindolin-3-yl)methyl)benzonitrile (5e): 74% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.06 (s, 1H), 7.24 (d, J = 7.3 Hz, 1H), 7.11 (t, J = 9.1 Hz, 3H), 6.94 (t, J = 7.4 Hz, 1H), 6.85 (d, J = 8.3 Hz, 2H), 6.63 (d, J = 7.7 Hz, 1H), 2.99 (dd, J = 47.3, 12.7 Hz, 2H), 2.24 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.24, 142.06, 135.29, 132.40, 132.14, 131.51, 128.84, 127.83, 124.78, 121.67, 109.67, 62.88, 44.51.

HRMS (ESI) *m/z* calcd $C_{16}H_{14}N_3O$ $[M+H]^+$ 264.1131, found 264.1131.

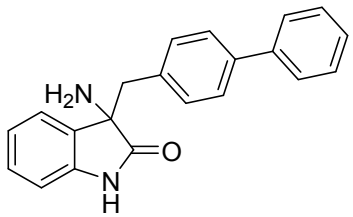


3-Amino-3-(4-ethoxybenzyl)indolin-2-one (5f): 82% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 9.99 (s, 1H), 7.24 (d, J = 7.3 Hz, 1H), 7.09 (t, J = 7.6 Hz, 1H), 6.93 (t, J = 7.4 Hz, 1H), 6.73 (d, J = 8.5 Hz, 2H), 6.60 (t, J = 7.8 Hz, 3H), 3.86 (q, J = 6.9 Hz, 2H), 2.92 (dd, J = 41.6, 12.9 Hz, 2H), 2.16 (s, 2H), 1.24 (t, J = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.50, 157.43, 142.18, 132.84, 131.30, 128.64, 127.98, 124.71, 121.55, 113.71, 109.56, 63.01, 44.55, 15.13.

HRMS (ESI) *m/z* calcd $C_{17}H_{19}N_2O_2$ $[M+H]^+$ 283.1441, found 283.1440.



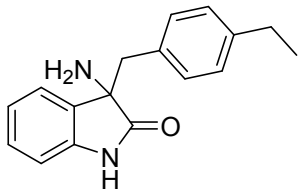
3-([1,1'-Biphenyl]-4-ylmethyl)-3-aminoindolin-2-one (5g): 84% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.06 (s, 1H), 7.57 (d, J = 7.5 Hz, 2H), 7.39 (dd, J = 12.0, 5.2 Hz, 4H), 7.35 – 7.25 (m, 2H), 7.11 (t, J = 7.6 Hz, 1H), 6.96 (dd, J = 10.5, 8.0 Hz, 3H), 6.63 (d, J = 7.6 Hz, 1H), 3.05 (dd, J = 45.0, 12.7 Hz, 2H), 2.26 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.34, 142.17, 140.12, 138.30, 135.63, 132.74, 130.98,

129.32, 128.78, 127.69, 126.82, 126.05, 124.80, 121.66, 109.65, 62.92, 44.93.

HRMS (ESI) *m/z* calcd $C_{21}H_{19}N_2O$ $[M+H]^+$ 315.1492, found 315.1490.

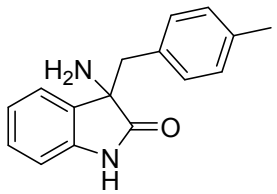


3-Amino-3-(4-ethylbenzyl)indolin-2-one (5h): 78% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.74 (s, 1H), 7.29 (s, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 2H), 6.82 (d, *J* = 7.2 Hz, 2H), 6.71 (d, *J* = 7.6 Hz, 1H), 3.09 (dd, *J* = 45.7, 12.7 Hz, 2H), 2.53 (q, *J* = 7.5 Hz, 2H), 2.05 (s, 2H), 1.15 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 182.59, 142.64, 140.54, 131.85, 131.60, 130.23, 128.94, 127.26, 124.65, 122.47, 110.08, 62.79, 44.70, 28.37, 15.40.

HRMS (ESI) *m/z* calcd $C_{17}H_{19}N_2O$ $[M+H]^+$ 267.1492, found 267.1491.

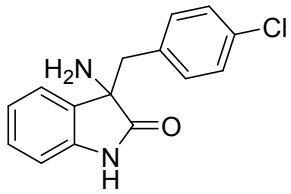


3-Amino-3-(4-methylbenzyl)indolin-2-one (5i): 79% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.79 (s, 1H), 7.29-7.21 (m, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.83 (dd, *J* = 32.9, 7.7 Hz, 4H), 6.72 (d, *J* = 7.7 Hz, 1H), 3.04 (dd, *J* = 43.1, 13.0 Hz, 2H), 2.19 (s, 3H), 1.96 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 182.52, 140.54, 136.28, 131.59, 131.55, 130.12, 128.95, 128.48, 124.62, 122.48, 110.06, 62.85, 44.72, 21.04.

HRMS (ESI) *m/z* calcd $C_{16}H_{17}N_2O$ $[M+H]^+$ 253.1335, found 253.1334.



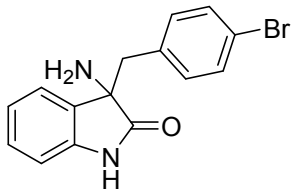
3-Amino-3-(4-chlorobenzyl)indolin-2-one (5j): 80% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 10.06 (s, 1H), 7.24 (d, *J* = 7.3 Hz, 1H), 7.11 (t, *J* = 9.0 Hz, 3H), 6.94 (t, *J* = 7.4 Hz, 1H), 6.85 (d, *J* = 8.3 Hz, 2H), 6.63 (d, *J* = 7.7 Hz, 1H), 2.99 (dd, *J* = 47.3, 12.7 Hz, 2H), 2.24 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 181.24, 142.06, 135.29, 132.40, 132.14, 131.50, 128.84,

127.83, 124.78, 121.67, 109.67, 62.88, 44.51.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OCl$ $[M+H]^+$ 273.0789, found 273.0789.

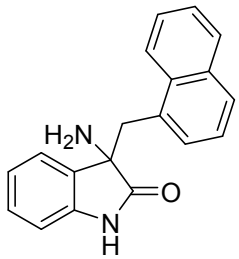


3-Amino-3-(4-bromobenzyl)indolin-2-one (5k): 82% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.04 (s, 1H), 7.25 (t, J = 7.9 Hz, 3H), 7.10 (t, J = 7.4 Hz, 1H), 6.94 (t, J = 7.4 Hz, 1H), 6.79 (d, J = 8.3 Hz, 2H), 6.62 (d, J = 7.7 Hz, 1H), 2.96 (dd, J = 47.2, 12.7 Hz, 2H), 2.27 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.18, 142.05, 135.71, 132.54, 132.38, 130.76, 128.84, 124.78, 121.68, 120.07, 109.67, 62.81, 44.55.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OBr$ $[M+H]^+$ 317.0284, found 317.0282.

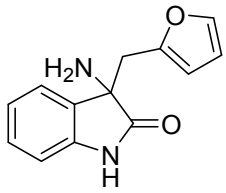


3-Amino-3-(naphthalen-1-ylmethyl)indolin-2-one (5l): 78% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.03 (s, 1H), 8.12-8.02 (m, 1H), 7.82-7.73 (m, 1H), 7.67 (d, J = 8.1 Hz, 1H), 7.42-7.33 (m, 2H), 7.27-7.16 (m, 2H), 7.10 (d, J = 7.0 Hz, 1H), 7.01 (t, J = 7.5 Hz, 1H), 6.79 (t, J = 7.5 Hz, 1H), 6.56 (d, J = 7.7 Hz, 1H), 3.53 (s, 2H), 2.32 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.70, 142.04, 133.58, 132.96, 132.76, 132.63, 128.64, 128.55, 127.41, 125.59, 125.58, 125.21, 125.16, 121.39, 109.52, 63.05.

HRMS (ESI) *m/z* calcd $C_{19}H_{17}N_2O$ $[M+H]^+$ 289.1335, found 289.1334.



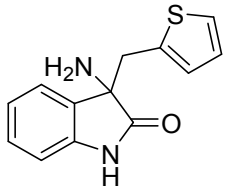
3-Amino-3-(furan-2-ylmethyl)indolin-2-one (5m): 54% yield. Light yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.10 (s, 1H), 7.31-7.17 (m, 2H), 7.14 (d, J = 7.3 Hz, 1H), 7.01 (t, J = 7.5 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 6.18 (s, 1H), 5.90 (d, J = 2.5 Hz, 1H), 3.14 (dd, J =

42.9, 14.6 Hz, 2H), 2.04 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 182.21, 149.98, 141.77, 140.47, 131.56, 129.09, 124.41, 122.66, 110.29, 110.20, 108.38, 61.43, 37.28.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}N_2O_2$ $[M+H]^+$ 229.0972, found 229.0971.

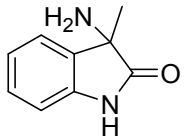


3-Amino-3-(thiophen-2-ylmethyl)indolin-2-one (5n): 80% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.55 (t, *J* = 21.3 Hz, 1H), 7.30 (d, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 7.6 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.03 (d, *J* = 4.9 Hz, 1H), 6.78 (t, *J* = 6.1 Hz, 2H), 6.63 (s, 1H), 3.45-3.25 (m, 2H), 1.86 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 181.85, 140.71, 136.38, 131.07, 129.35, 127.47, 126.34, 124.76, 124.61, 122.81, 110.09, 62.38, 39.20.

HRMS (ESI) *m/z* calcd $C_{13}H_{13}N_2OS$ $[M+H]^+$ 245.0743, found 245.0742.

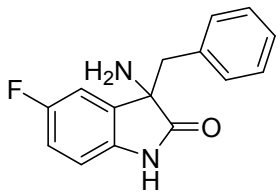


3-Amino-3-methylindolin-2-one (5o): 86% yield. Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.32 (s, 1H), 7.40 (d, *J* = 7.3 Hz, 1H), 7.24 (d, *J* = 7.7 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 7.7 Hz, 1H), 1.84 (s, 2H), 1.49 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 182.64, 141.55, 135.40, 128.58, 123.80, 121.93, 109.89, 58.06, 26.26.

HRMS (ESI) *m/z* calcd $C_9H_{11}N_2O$ $[M+H]^+$ 163.0866, found 163.0865.



3-Amino-3-benzyl-5-fluoroindolin-2-one (5p): 78% yield. White solid.

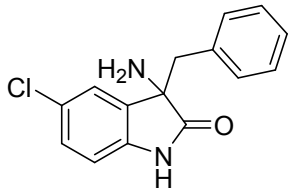
^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 10.05 (s, 1H), 7.13 (dd, *J* = 8.4, 2.6 Hz, 1H), 7.11-7.03 (m, 3H), 6.89 (ddd, *J* = 8.0, 7.5, 2.5 Hz, 3H), 6.57 (dd, *J* = 8.4, 4.4 Hz, 1H), 3.02 (dd, *J* = 41.8, 12.7 Hz, 2H), 2.34 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 181.26, 159.44, 157.09, 138.23, 138.22, 136.01, 134.77,

134.70, 130.30, 127.96, 126.79, 114.92, 114.69, 112.67, 112.43, 110.25, 110.17, 63.57, 63.56, 45.23.

^{19}F NMR (376 MHz, DMSO-d_6) δ: -122.22.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OF$ $[M+H]^+$ 257.1085, found 257.1084.

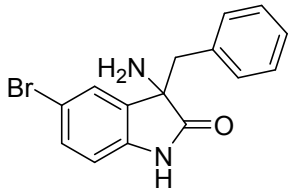


3-Amino-3-benzyl-5-chloroindolin-2-one (5q): 79% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.13 (s, 1H), 7.29 (d, *J* = 2.0 Hz, 1H), 7.12 (dd, *J* = 8.2, 2.1 Hz, 1H), 7.11-7.04 (m, 3H), 6.86 (dd, *J* = 6.2, 2.8 Hz, 2H), 6.59 (d, *J* = 8.2 Hz, 1H), 3.01 (dd, *J* = 48.5, 12.7 Hz, 2H), 2.31 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 181.00, 140.99, 135.96, 135.05, 130.30, 128.43, 127.98, 126.83, 125.64, 125.00, 110.92, 63.36, 45.17.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OCl$ $[M+H]^+$ 273.0789, found 273.0788.



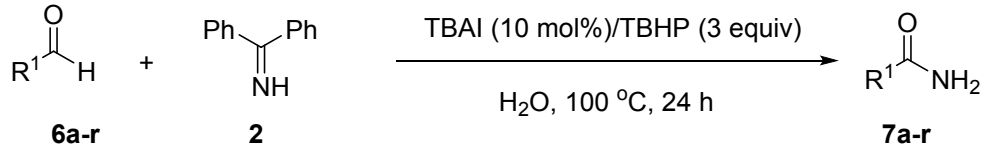
3-Amino-3-benzyl-5-bromoindolin-2-one (5r): 81% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.14 (s, 1H), 7.40 (s, 1H), 7.25 (dd, *J* = 8.2, 1.9 Hz, 1H), 7.17-6.98 (m, 3H), 6.95-6.78 (m, 2H), 6.55 (d, *J* = 8.2 Hz, 1H), 3.00 (dd, *J* = 50.3, 12.7 Hz, 2H), 2.31 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 180.87, 141.40, 135.96, 135.46, 131.28, 130.32, 127.97, 127.75, 126.84, 113.35, 111.48, 63.33, 45.18.

HRMS (ESI) *m/z* calcd $C_{15}H_{14}N_2OBr$ $[M+H]^+$ 317.0284, found 317.0282.

General procedure for the amination of aldehydes (6)

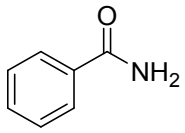


6a-r 1 equiv | **2** 2 equiv | **7a-r**

To a mixture of aldehydes **6** (1 mmol), TBAI (36.9 mg, 0.1 mmol, 10 mmol%) and benzophenone imine **2** (362.2 mg, 2 mmol) was added TBHP (70% in water, 386 mg, 3 mmol)

at room temperature. After stirring at 100 °C for 24 h, the reaction mixture was poured into saturated $Na_2S_2O_3$ (aqueous solution, 5 mL), extracted with ethyl acetate (3×5 mL) and washed with brine. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed in vacuo. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate as eluent) to afford analytically pure primary amine **7**.

Characterization of amides (7)

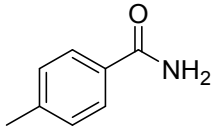


Benzamide (7a): 81% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.04 (s, 1H), 7.92 (d, J = 7.9 Hz, 2H), 7.58-7.50 (m, 1H), 7.46 (dd, J = 11.4, 4.1 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 168.46, 134.71, 131.69, 128.67, 127.94.

HRMS (ESI) *m/z* calcd C_7H_8NO $[M+H]^+$ 122.0600, found 122.0601.

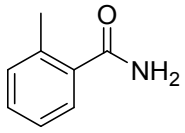


4-Methylbenzamide (7b): 79% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.72 (d, J = 8.1 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 6.02 (d, J = 105.1 Hz, 2H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.62, 142.61, 130.38, 129.30, 127.42, 21.51.

HRMS (ESI) *m/z* calcd $C_8H_{10}NO$ $[M+H]^+$ 136.0757, found 136.0756.

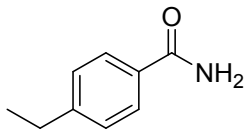


2-Methylbenzamide (7c): 81% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, J = 7.6 Hz, 1H), 7.35 (t, J = 7.5 Hz, 1H), 7.26-7.19 (m, 2H), 5.97 (s, 2H), 2.51 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.41, 136.31, 135.22, 131.21, 130.29, 126.98, 125.75, 19.99.

HRMS (ESI) *m/z* calcd $C_8H_{10}NO$ $[M+H]^+$ 136.0757, found 136.0756.

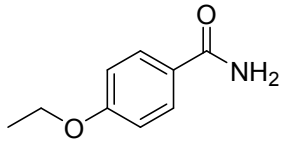


4-Ethylbenzamide (7d): 83% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 7.9 Hz, 2H), 5.94 (d, J = 149.4 Hz, 2H), 2.71 (q, J = 7.6 Hz, 2H), 1.26 (t, J = 7.6 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.70, 148.77, 130.70, 129.81, 128.12, 127.51, 126.12, 28.81, 15.29.

HRMS (ESI) *m/z* calcd $C_9H_{12}NO$ $[M+H]^+$ 150.0913, found 150.0913.

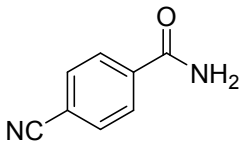


4-Ethoxybenzamide (7e): 84% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.80 (d, J = 8.8 Hz, 2H), 6.94 (d, J = 8.8 Hz, 2H), 6.28 (s, 2H), 4.10 (q, J = 7.0 Hz, 2H), 1.45 (t, J = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 167.92, 161.33, 129.82, 126.80, 114.21, 63.70, 15.01.

HRMS (ESI) *m/z* calcd $C_9H_{12}NO_2$ $[M+H]^+$ 166.0863, found 166.0862.

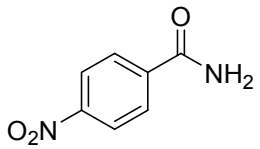


4-Cyanobenzamide (7f): 50% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.24 (s, 1H), 8.00 (dd, J = 31.4, 8.3 Hz, 4H), 7.70 (s, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 166.91, 138.75, 132.84, 128.72, 118.84, 114.11.

HRMS (ESI) *m/z* calcd $C_8H_7N_2O$ $[M+H]^+$ 147.0553, found 147.0553.

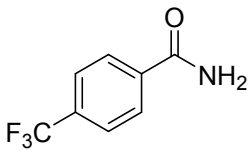


4-Nitrobenzamide (7g): 38% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.31 (d, J = 8.4 Hz, 3H), 8.12 (d, J = 8.5 Hz, 2H), 7.75 (s, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 166.68, 149.51, 140.44, 129.37, 123.90.

HRMS (ESI) *m/z* calcd $C_7H_7N_2O_3$ $[M+H]^+$ 167.0451, found 167.0451.



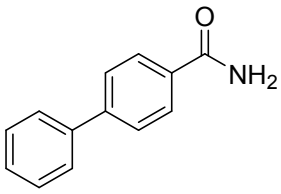
4-(Trifluoromethyl)benzamide (7h): 53% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 (s, 2H), 7.73 (d, J = 8.2 Hz, 2H), 5.99 (d, J = 122.0 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 167.15, 138.54, 132.10, 131.78, 131.46, 131.15, 128.78, 125.73, 125.69.

^{19}F NMR (376 MHz, DMSO-d_6) δ: -61.38.

HRMS (ESI) *m/z* calcd $C_8H_7NOF_3$ $[M+H]^+$ 190.0474, found 190.0473.

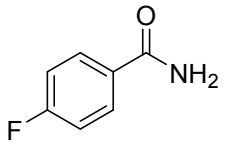


[1,1'-Biphenyl]-4-carboxamide (7i): 89% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.92 (d, J = 8.3 Hz, 2H), 7.69 (d, J = 8.3 Hz, 2H), 7.62 (d, J = 7.5 Hz, 2H), 7.48 (t, J = 7.5 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H), 6.19 (d, J = 143.5 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 168.06, 143.23, 139.70, 133.57, 129.48, 128.65, 128.48, 127.34, 126.92.

HRMS (ESI) *m/z* calcd $C_{13}H_{12}NO$ $[M+H]^+$ 198.0913, found 198.0912.



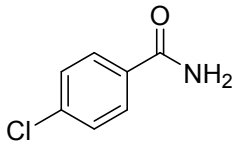
4-Fluorobenzamide (7j): 77% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (dd, J = 8.6, 5.3 Hz, 2H), 7.14 (t, J = 8.5 Hz, 2H), 6.09 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 167.31, 165.62, 163.16, 131.21, 131.18, 130.62, 130.53, 115.65, 115.43.

^{19}F NMR (376 MHz, DMSO-d_6) δ: -109.56.

HRMS (ESI) *m/z* calcd $C_8H_7NOF_3$ $[M+H]^+$ 190.0474, found 190.0473.

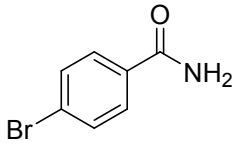


4-Chlorobenzamide (7k): 64% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.77 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 6.03 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 167.30, 136.56, 133.48, 129.87, 128.75.

HRMS (ESI) *m/z* calcd C_7H_7NOCl $[M+H]^+$ 156.0211, found 156.0209.

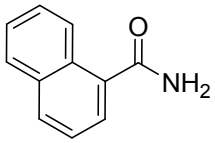


4-Bromobenzamide (7l): 65% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.70 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 5.98 (d, *J* = 142.0 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 167.39, 133.86, 131.70, 130.07, 125.49.

HRMS (ESI) *m/z* calcd C_7H_7NOBr $[M+H]^+$ 199.9706, found 199.9704.

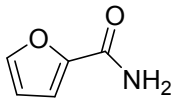


1-Naphthamide (7m): 70% yield. White solid.

^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 8.34 (d, *J* = 7.6 Hz, 1H), 8.09-7.94 (m, 3H), 7.70-7.61 (m, 2H), 7.56 (ddd, *J* = 20.2, 10.9, 6.4 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 171.12, 135.13, 133.68, 130.26, 130.21, 128.65, 127.08, 126.60, 126.08, 125.63, 125.41.

HRMS (ESI) *m/z* calcd $C_{11}H_{10}NO$ $[M+H]^+$ 172.0757, found 172.0756.

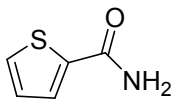


Furan-2-carboxamide (7n): 51%yield. Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.48 (d, *J* = 0.8 Hz, 1H), 7.18 (d, *J* = 3.5 Hz, 1H), 6.53 (dd, *J* = 3.4, 1.7 Hz, 1H), 6.05 (d, *J* = 207.7 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) *δ*: 159.89, 148.50, 145.46, 114.08, 112.24.

HRMS (ESI) *m/z* calcd $C_5H_6NO_2$ $[M+H]^+$ 112.0393, found 112.0393.

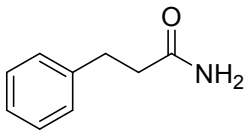


Thiophene-2-carboxamide (7o): 82% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, J = 3.7 Hz, 1H), 7.55 (d, J = 4.9 Hz, 1H), 7.11 (dd, J = 4.8, 3.9 Hz, 1H), 5.95 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ: 163.35, 140.78, 131.44, 129.13, 128.35.

HRMS (ESI) *m/z* calcd C_5H_6NOS $[M+H]^+$ 128.0165, found 128.0164.



3-Phenylpropanamide (7p): 70% yield. White solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30 (t, J = 7.4 Hz, 2H), 7.22 (d, J = 6.9 Hz, 3H), 5.58 (d, J = 54.8 Hz, 2H), 2.98 (t, J = 7.7 Hz, 2H), 2.55 (t, J = 7.8 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 174.00, 139.65, 127.54, 127.28, 125.27, 36.48, 30.36.

HRMS (ESI) *m/z* calcd $C_9H_{12}NO$ $[M+H]^+$ 150.0913, found 150.0913.

References:

[1] S. Yamada, D. Morizono and K. Yamamoto, *Tetrahedron Lett.* 1992, **33**, 4629.

[2] L. Cheng, L. Liu, D. Wang, Y. Chen, *Org. Lett.* 2009, **11**, 3874-3877.

[3] Y. Cheng, W. Dong, L. Wang, K. Parthasarathy, C. Bolm, *Org. Lett.* 2014, **16**, 2000-2002

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Acq. Operator     : jd701092                            Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
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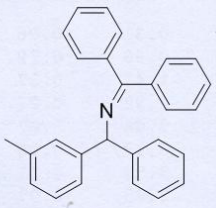
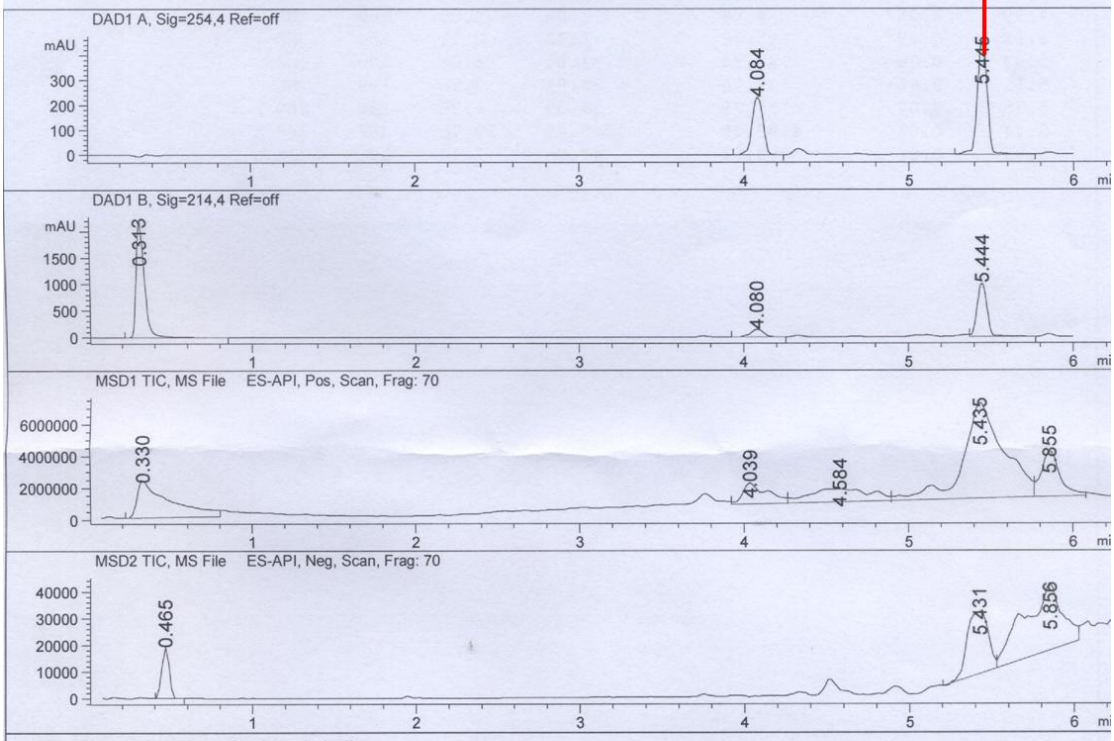


8b

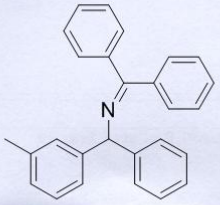


Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	34.99	8.23	1.05	169	ND
2.91	0.11	3.50	0.46	0.10	126	ND
3.03	0.07	3.47	0.75	0.10	126	ND
3.17	0.12	4.33	0.50	0.13	126	ND
3.47	0.14	6.99	0.70	0.21	126	ND
3.76	0.13	9.13	0.94	0.27	100	ND
3.90	0.09	5.52	0.87	0.17	100	ND
4.08	0.07	1074.58	232.01	32.16	196	ND
4.33	0.08	119.37	24.98	3.57	126	ND
4.54	0.11	19.86	2.58	0.59	126	ND
4.69	0.11	30.37	3.92	0.91	100	ND
4.94	0.10	15.47	2.29	0.46	100	ND
5.09	0.13	30.04	3.26	0.90	100	ND
5.24	0.08	14.53	2.83	0.43	100	ND
5.44	0.07	1915.56	455.64	57.32	362	280
5.85	0.09	54.14	9.04	1.62	391	113

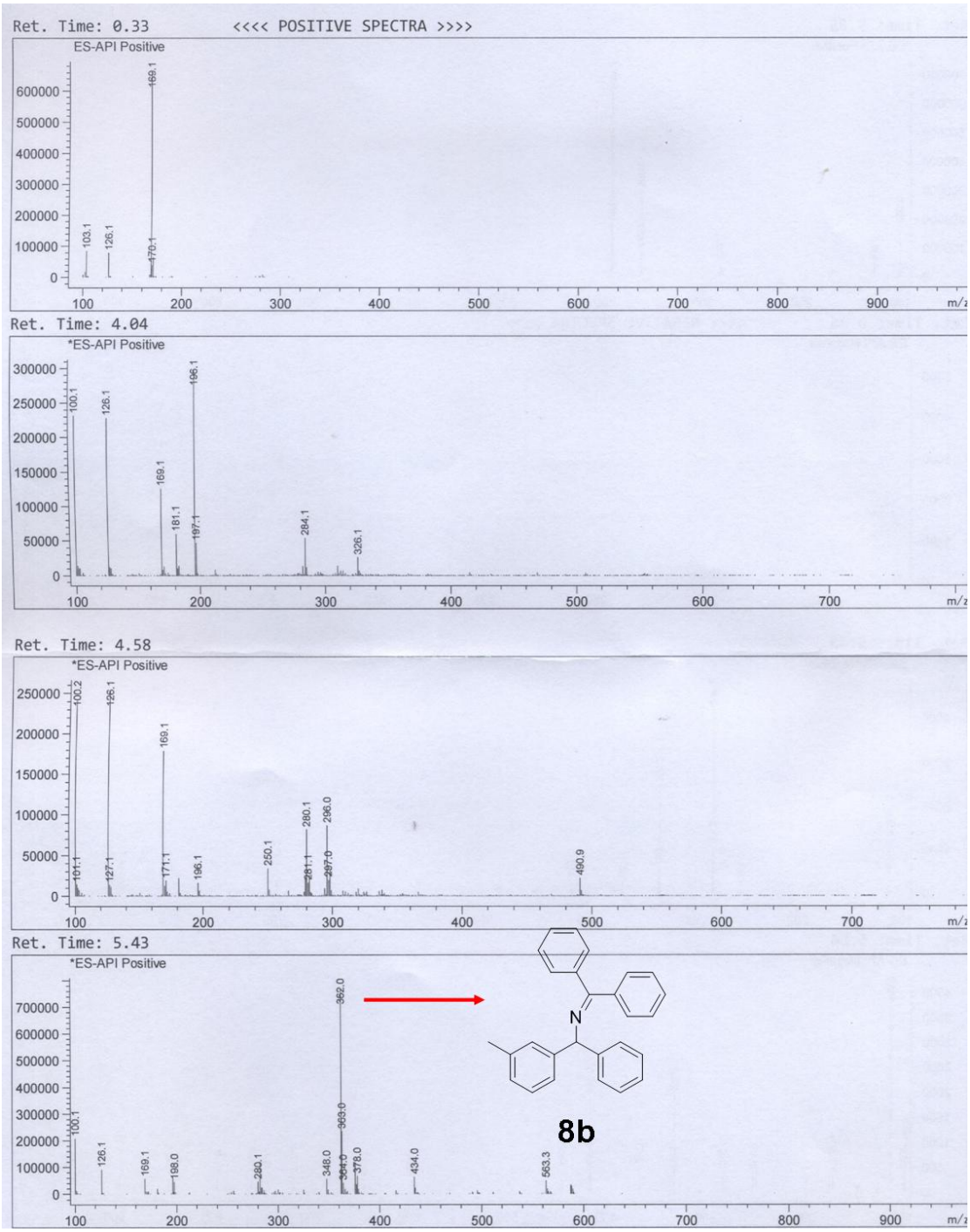
Integration Results for DAD1 B, Sig=214,4 Ref=off

0.31	0.06	7726.45	2186.95	54.87	169	ND
0.98	0.29	102.36	4.56	0.73	169	ND
1.47	0.37	158.96	5.33	1.13	169	ND
2.92	0.07	3.65	0.81	0.03	126	ND
3.04	0.07	2.87	0.62	0.02	126	ND
3.17	0.06	2.08	0.51	0.01	126	ND
3.54	0.11	5.09	0.66	0.04	126	ND
3.75	0.07	104.82	20.95	0.74	100	ND
3.84	0.06	27.96	6.72	0.20	100	ND
4.08	0.09	826.07	145.28	5.87	196	ND
4.33	0.08	130.33	26.67	0.93	126	ND
4.53	0.11	40.27	5.14	0.29	126	ND
4.68	0.11	68.95	9.64	0.49	100	ND
4.79	0.05	6.66	1.86	0.05	100	ND
4.92	0.09	15.32	2.42	0.11	100	ND
5.07	0.06	143.24	31.64	1.02	100	ND
5.11	0.06	139.18	32.81	0.99	198	ND
5.35	0.07	187.29	39.39	1.33	196	280
5.44	0.07	4190.15	1009.45	29.75	362	280
5.85	0.07	200.68	47.06	1.43	391	113



8b

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)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Ret. Time: 4.04
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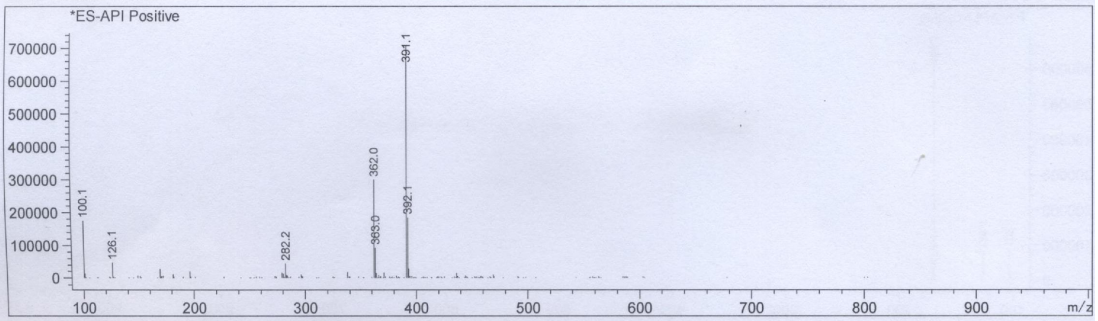
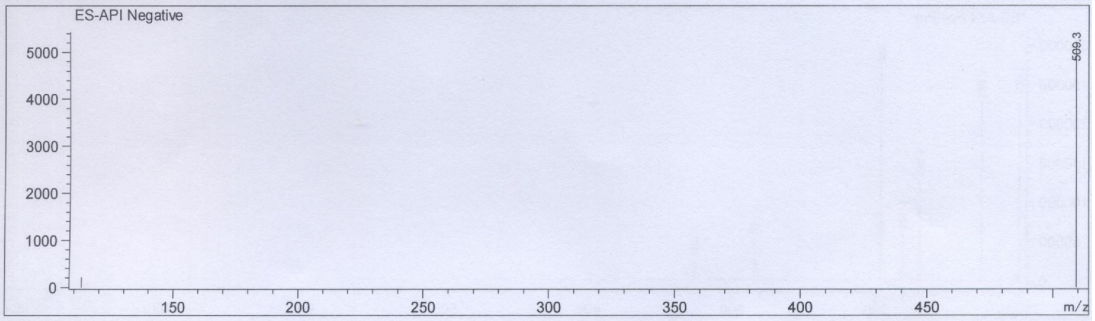
Ret. Time: 4.58
*ES-API Positive
Ret. Time: 5.43
*ES-API Positive
N
8b

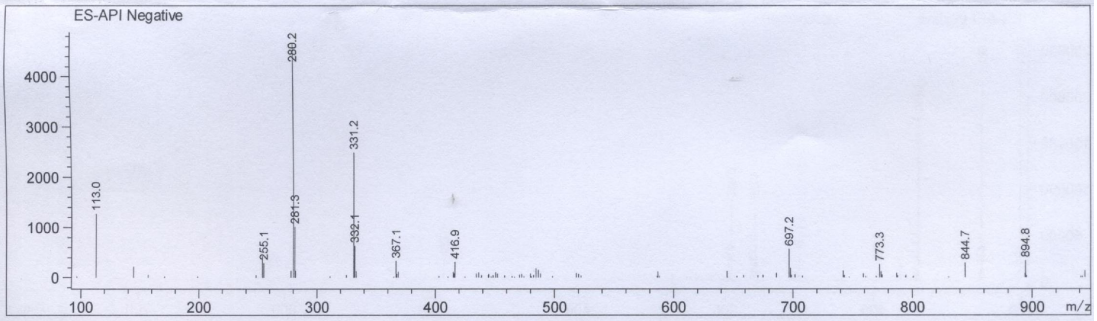
Ret. Time: 5.85



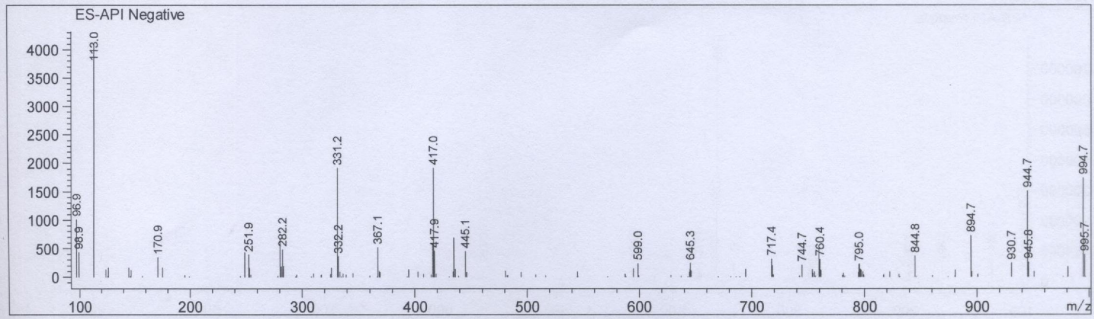
Ret. Time: 0.46 <<<< NEGATIVE SPECTRA >>>>



Ret. Time: 5.43



Ret. Time: 5.86



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Injection Date    : 03-Aug-15, 09:41:19                Seq. Line : 0
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Acq. Operator     : jd701092                            Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
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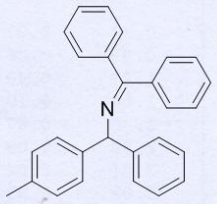

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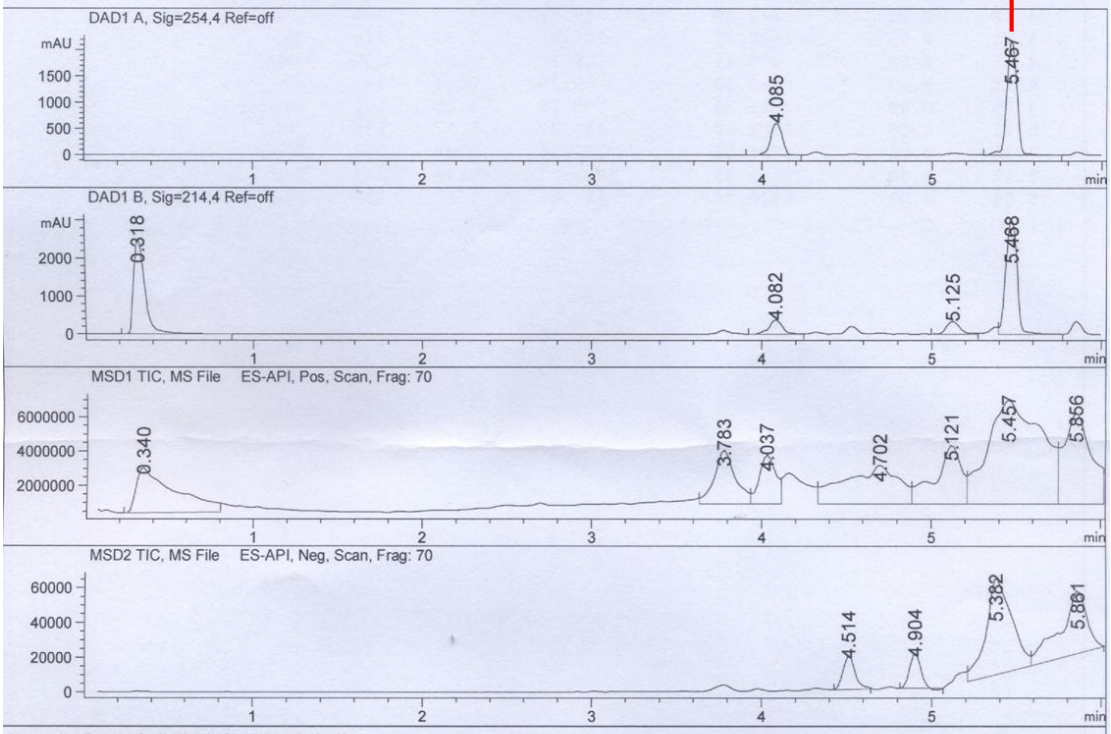

Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
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1.57	0.30	11.37	0.47	0.09	169	ND
2.91	0.11	50.89	7.07	0.39	169	ND
3.29	0.11	12.06	1.58	0.09	126	ND
3.40	0.10	11.88	1.80	0.09	126	ND
3.54	0.09	7.18	1.15	0.05	126	ND
3.77	0.13	31.14	3.30	0.24	181	ND
4.09	0.08	2881.76	608.32	21.99	196	ND
4.32	0.08	249.43	53.93	1.90	126	ND
4.53	0.08	101.30	19.04	0.77	126	255
4.70	0.11	51.69	6.84	0.39	126	ND
4.95	0.08	24.99	4.37	0.19	126	283
5.13	0.14	286.48	31.99	2.19	286	ND
5.47	0.07	9086.40	2175.66	69.34	362	280
5.85	0.07	232.17	54.69	1.77	391	113

Integration Results for DAD1 B, Sig=214,4 Ref=off

0.32	0.07	11685.82	2509.90	34.04	169	ND
0.99	0.26	148.62	7.39	0.43	169	ND
1.47	0.40	222.14	6.72	0.65	169	ND
2.54	0.10	7.06	1.07	0.02	169	ND
2.70	0.07	2.04	0.53	0.01	169	ND
2.90	0.09	28.84	5.09	0.08	169	ND
3.01	0.04	2.32	0.88	0.01	126	ND
3.09	0.09	12.59	1.86	0.04	126	ND
3.27	0.10	12.05	1.94	0.04	126	ND
3.40	0.09	10.73	1.66	0.03	126	ND
3.54	0.08	12.09	2.05	0.04	126	ND
3.77	0.08	591.52	101.82	1.72	181	ND
4.08	0.09	2168.19	376.37	6.32	196	ND
4.32	0.08	247.88	49.40	0.72	126	ND
4.53	0.08	1005.77	207.86	2.93	126	255
4.69	0.09	170.61	28.22	0.50	250	ND
4.81	0.09	63.33	9.25	0.18	126	283
4.95	0.08	51.87	9.38	0.15	126	283
5.13	0.09	1981.50	339.21	5.77	286	ND
5.38	0.05	685.55	194.54	2.00	378	280
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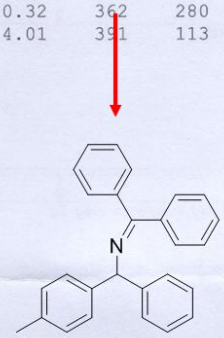
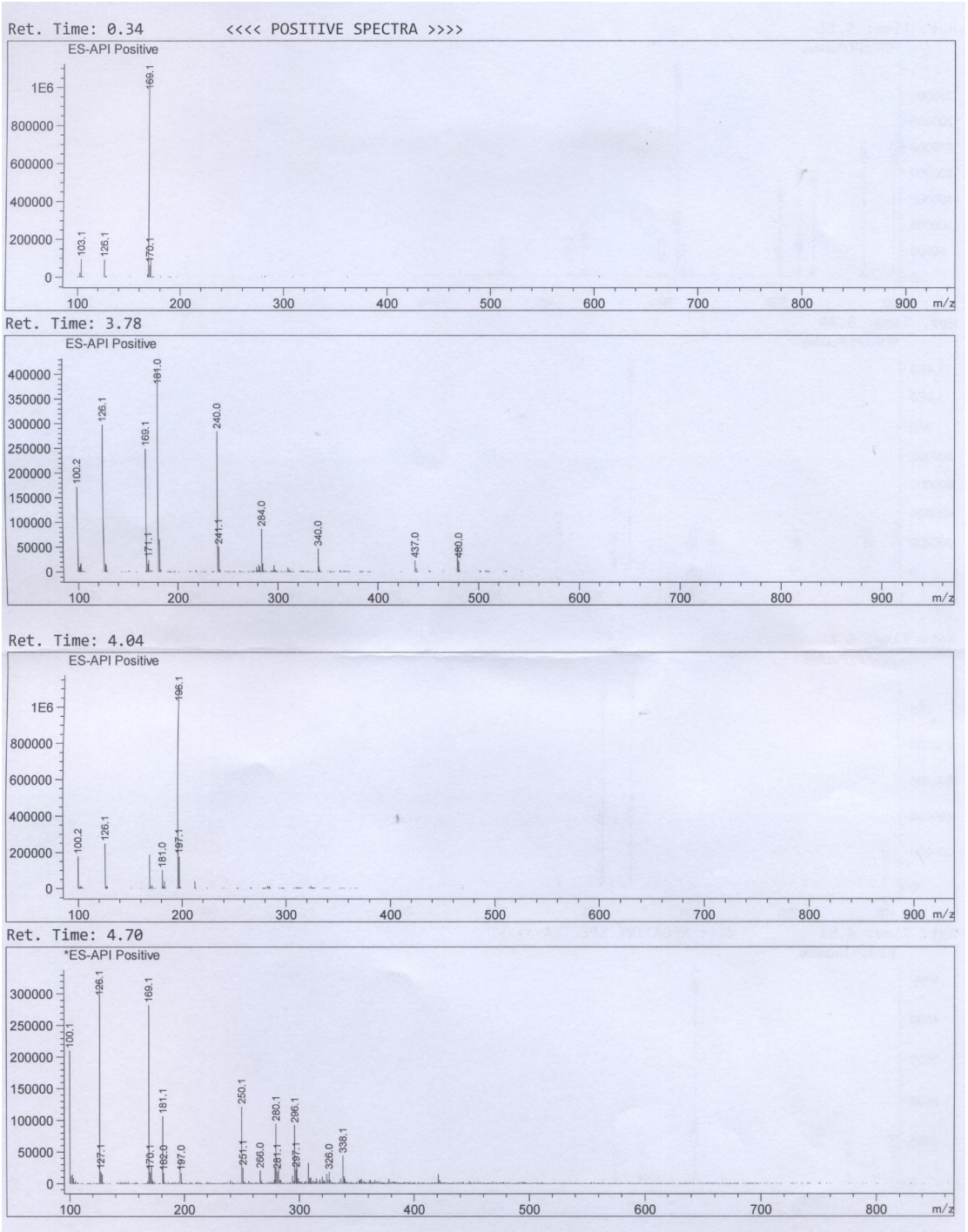


8c

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)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126.1
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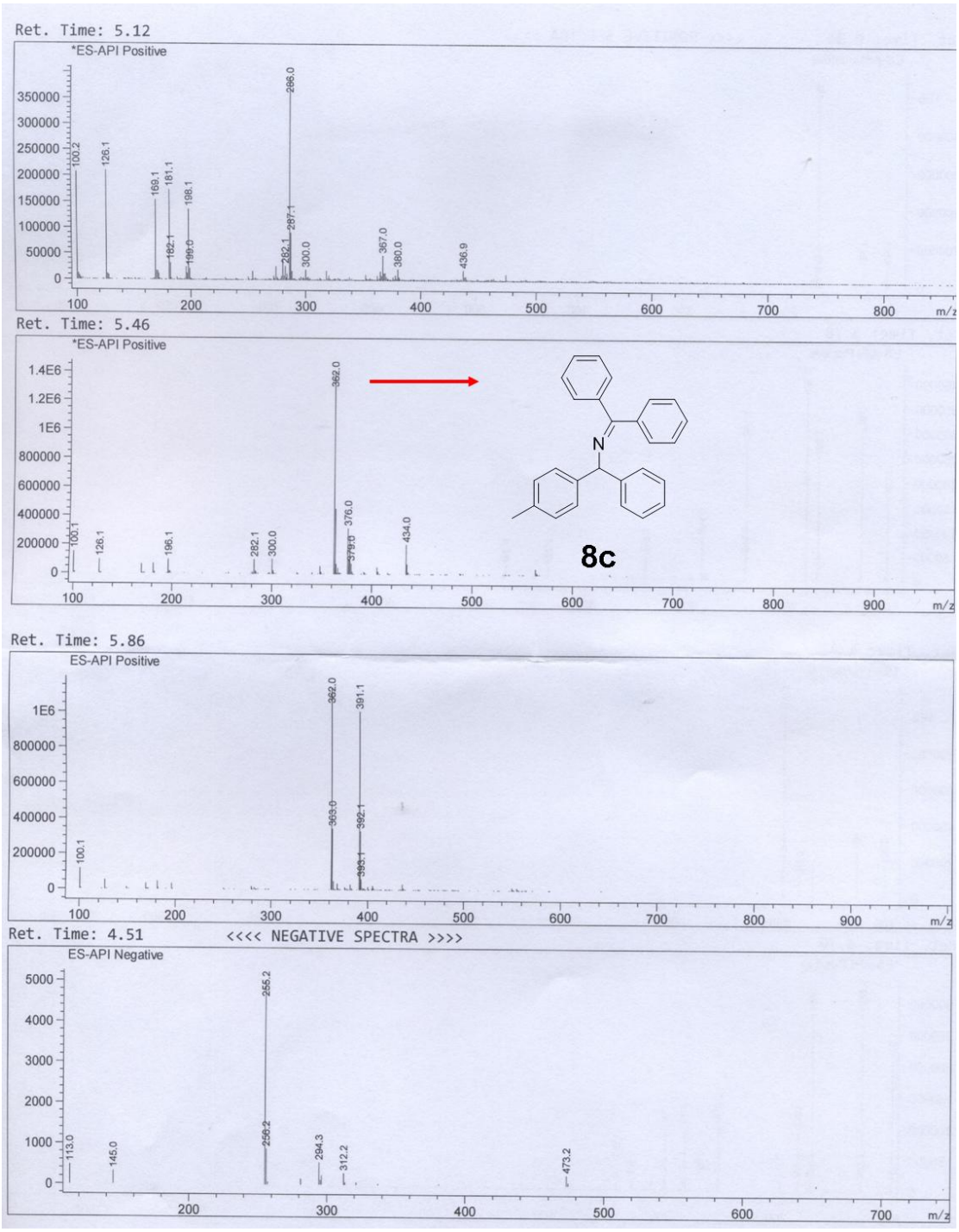
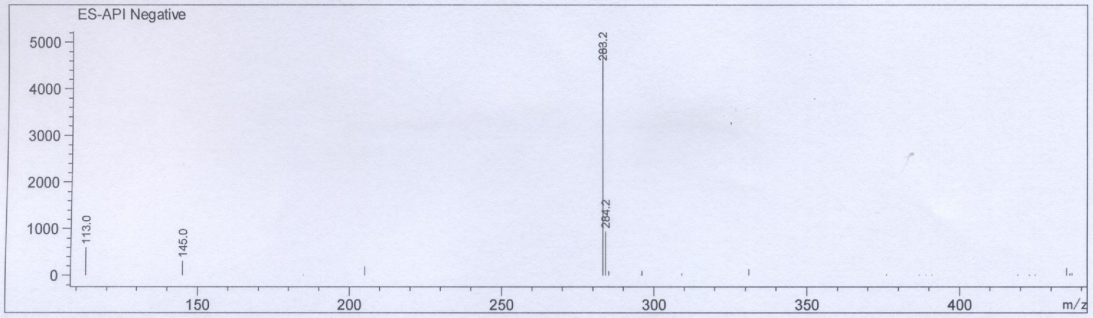
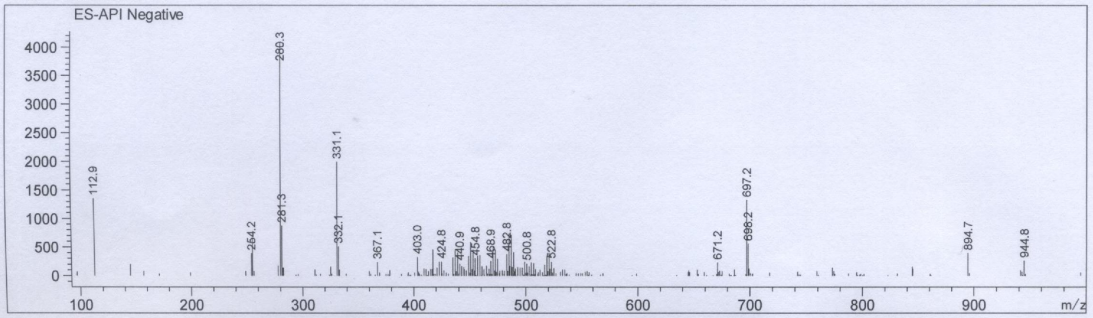
181.0
126.1
169.1
240.0
100.2
284.0
241.1
340.0
171.1
437.0
480.0
Ret. Time: 4.04
ES-API Positive
196.1
100.2
126.1
181.0
197.1
Ret. Time: 4.70
*ES-API Positive
126.1
169.1
100.1
250.1
181.1
280.1
296.1
127.1
170.1
182.0
197.0
251.1
266.0
281.1
297.1
326.0
338.1
m/z


Ret. Time: 5.12
*ES-API Positive
Ret. Time: 5.46
*ES-API Positive
8c
Ret. Time: 5.86
ES-API Positive
Ret. Time: 4.51
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative

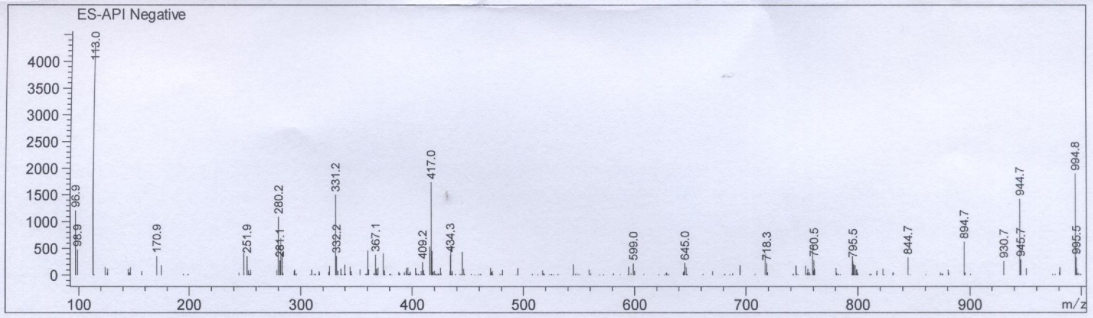
Ret. Time: 4.90



Ret. Time: 5.38



Ret. Time: 5.86



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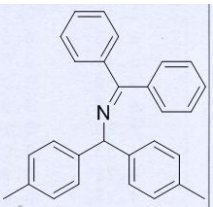
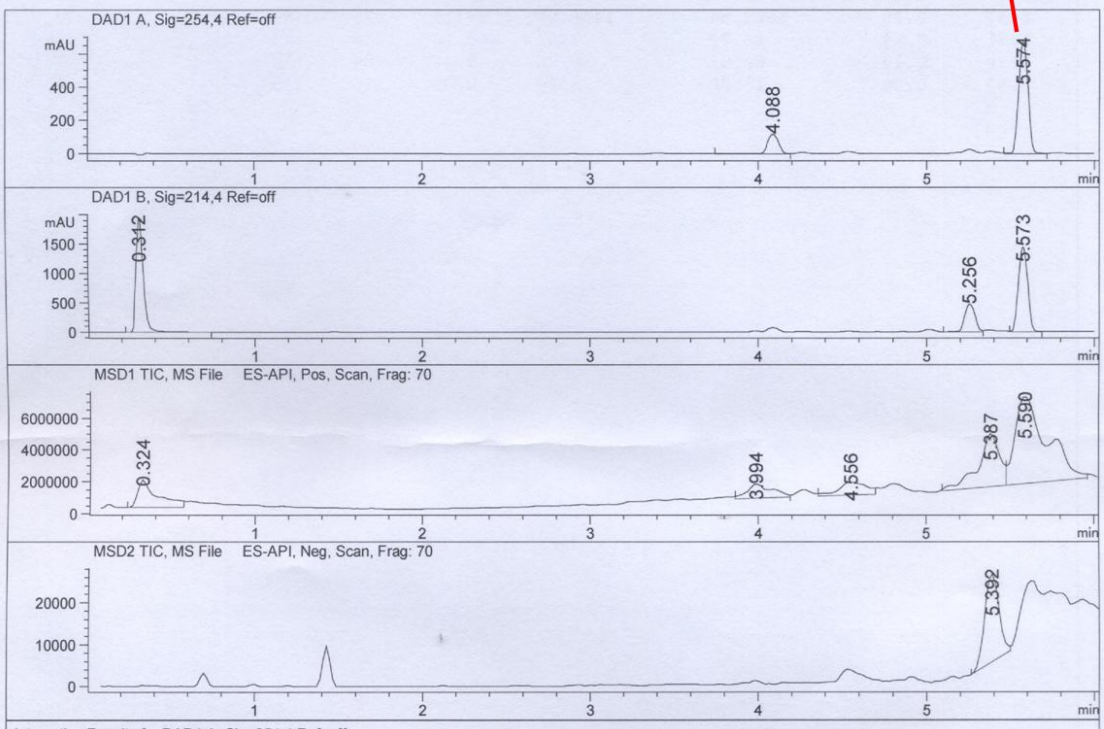
Injection Date    : 03-Aug-15, 10:22:53                Seq. Line : 0
Sample Name       : 3D                              Location  : P1-D-04
Acq. Operator     : jd701092                           Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8d



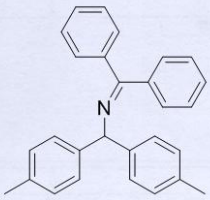
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	51.85	11.22	1.40	169	ND
2.90	0.11	3.36	0.47	0.09	100	ND
3.41	0.11	36.78	5.08	0.99	126	ND
4.09	0.07	532.86	117.42	14.35	126	ND
4.26	0.10	47.21	7.47	1.27	100	ND
4.53	0.08	71.19	13.49	1.92	100	ND
4.72	0.11	23.03	2.93	0.62	100	ND
4.94	0.16	37.17	2.98	1.00	100	ND
5.25	0.09	143.07	24.82	3.85	195	ND
5.38	0.08	75.08	14.72	2.02	314	280
5.57	0.06	2645.69	676.16	71.23	376	ND
5.78	0.12	47.15	5.30	1.27	376	113

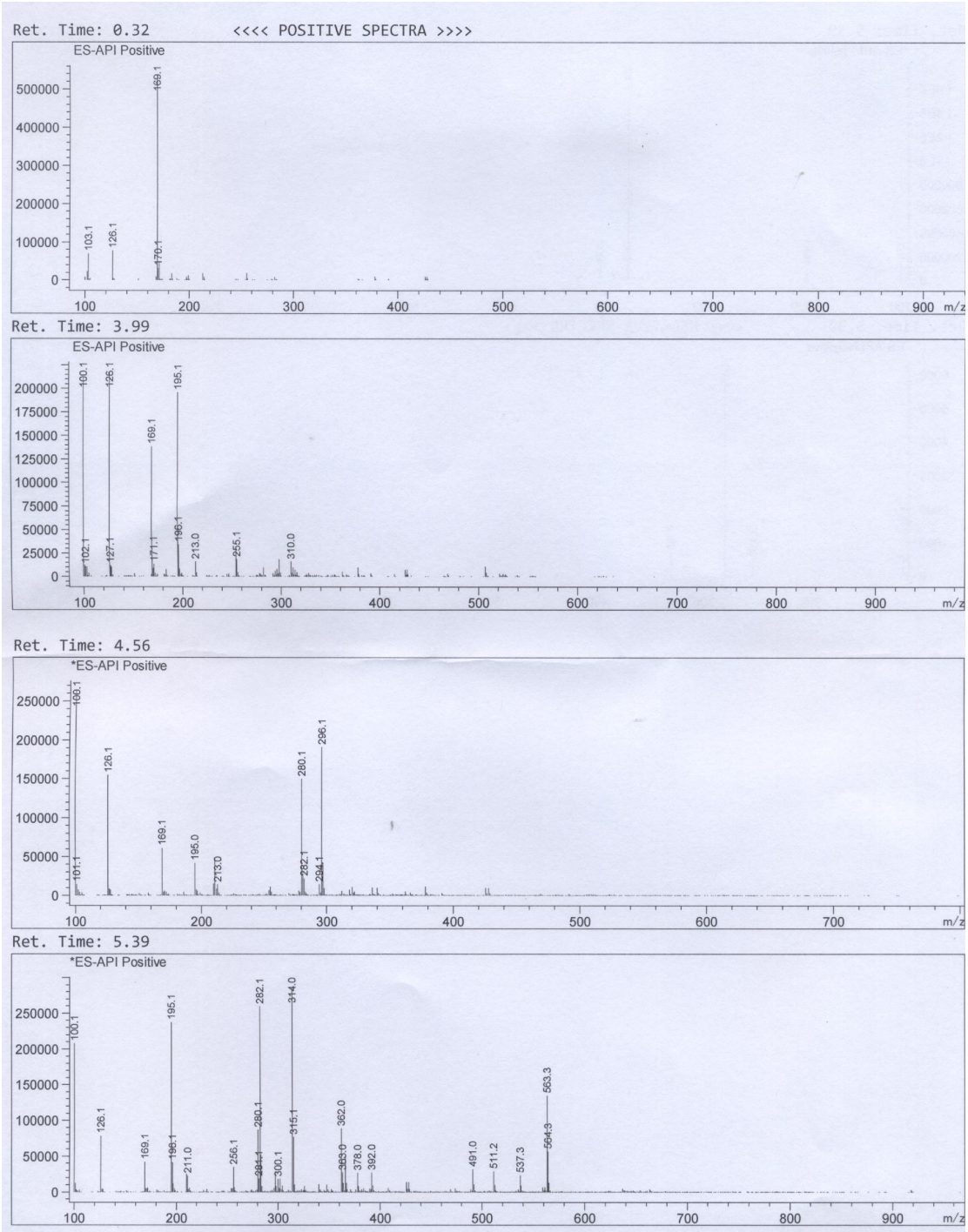
Integration Results for DAD1 B, Sig=214,4 Ref=off

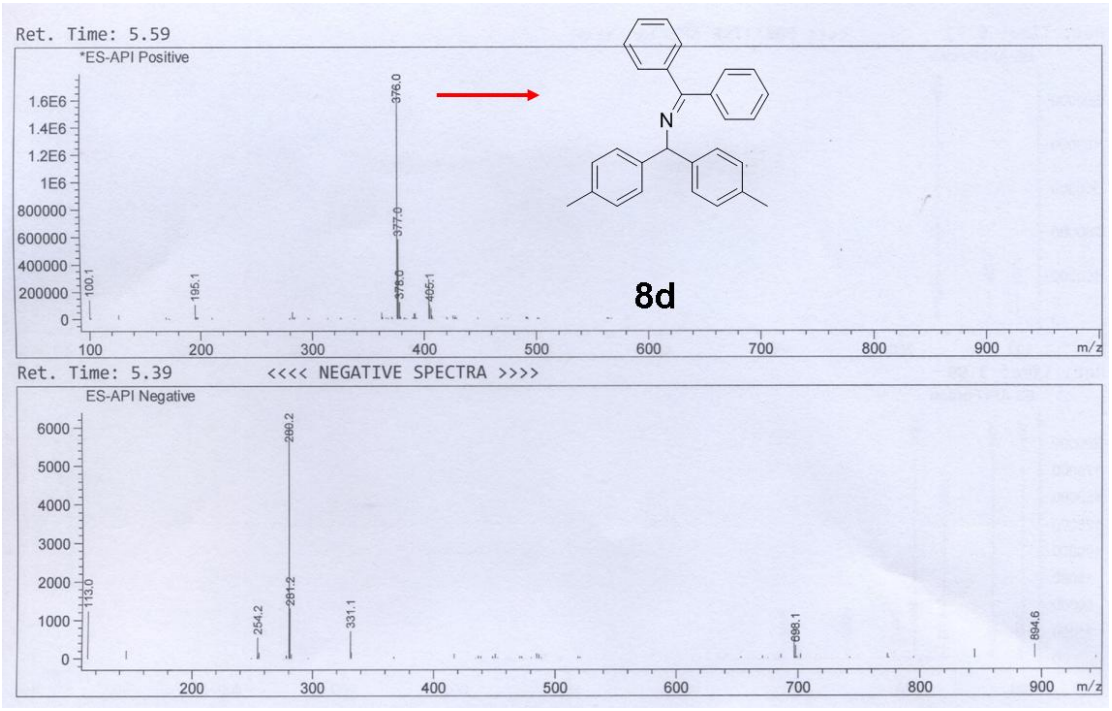
RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.31	0.05	5950.29	1932.71	39.52	169	ND
0.97	0.27	72.82	3.49	0.48	169	ND

1.47	0.36	150.31	5.12	1.00	126	ND
2.85	0.09	4.72	0.71	0.03	100	ND
3.41	0.09	20.95	3.83	0.14	126	ND
3.99	0.07	86.40	20.20	0.57	126	ND
4.09	0.08	361.69	79.18	2.40	100	ND
4.26	0.10	72.68	10.38	0.48	100	ND
4.53	0.09	93.18	16.64	0.62	100	ND
4.71	0.08	27.69	4.74	0.18	100	ND
4.77	0.03	5.20	3.11	0.03	100	ND
4.86	0.10	32.88	4.63	0.22	100	ND
5.01	0.08	196.74	42.51	1.31	100	ND
5.26	0.07	2060.02	474.97	13.68	195	ND
5.37	0.07	160.40	33.12	1.07	314	280
5.47	0.04	30.81	12.27	0.20	362	280
5.57	0.06	5629.54	1446.00	37.39	376	ND
5.71	0.04	18.72	7.54	0.12	376	113
5.78	0.10	68.91	9.96	0.46	404	113
5.97	0.08	13.90	3.09	0.09	376	113

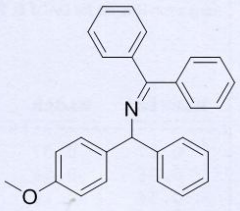


8d

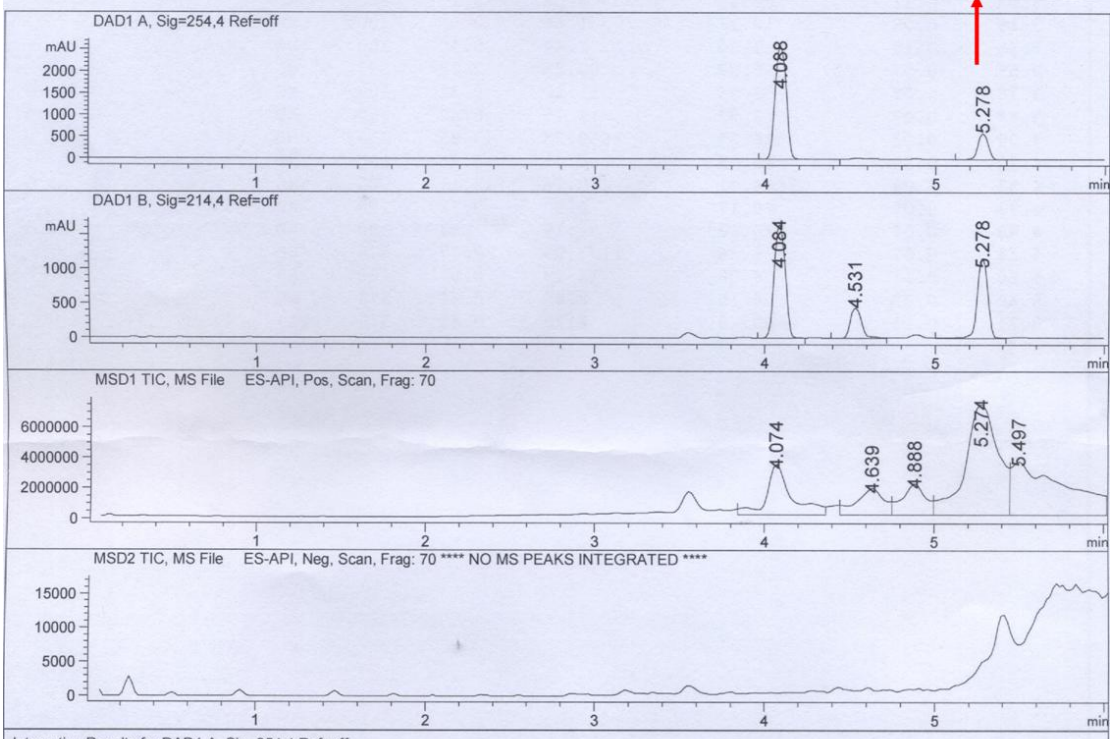

Ret. Time: 0.32
<<<< POSITIVE SPECTRA >>>>
ES-API Positive
169.1
103.1
126.1
170.1
Ret. Time: 3.99
ES-API Positive
100.1
126.1
195.1
169.1
102.1
127.1
171.1
196.1
213.0
255.1
310.0
Ret. Time: 4.56
*ES-API Positive
100.1
126.1
169.1
195.0
213.0
280.1
296.1
101.1
282.1
294.1
Ret. Time: 5.39
*ES-API Positive
100.1
126.1
169.1
195.1
196.1
211.0
256.1
280.1
282.1
284.1
300.1
314.0
315.1
362.0
363.0
378.0
392.0
491.0
511.2
537.3
563.3
564.3


Ret. Time: 5.59
*ES-API Positive
376.0
377.0
100.1
196.1
405.1
8d
N
Ret. Time: 5.39
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative
280.2
281.2
113.0
254.2
331.1
698.1
894.6
m/z

```
Injection Date    : 03-Aug-15, 11:20:19                Seq. Line : 0
Sample Name       : 3E                              Location  : P1-E-02
Acq. Operator     : jd701092                           Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8e

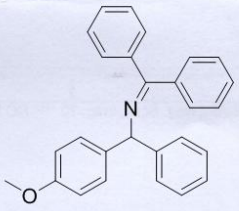


Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.26	0.08	44.30	8.40	0.27	102	ND
0.37	0.07	67.68	14.09	0.41	102	ND
0.97	0.55	454.78	10.16	2.74	102	ND
1.48	0.76	593.91	9.40	3.58	100	ND
2.61	0.20	82.26	5.23	0.50	100	ND
2.92	0.27	117.11	5.57	0.71	100	ND
3.25	0.15	52.85	4.68	0.32	100	ND
3.39	0.10	30.83	4.33	0.19	100	ND
3.55	0.10	70.74	10.69	0.43	197	ND
3.70	0.10	28.33	4.26	0.17	100	ND
3.88	0.11	66.30	8.87	0.40	100	ND
4.09	0.08	11960.30	2612.25	72.00	213	ND
4.55	0.13	268.32	27.80	1.62	100	ND
4.88	0.09	107.64	17.49	0.65	197	ND
5.07	0.09	31.15	4.79	0.19	100	ND
5.28	0.07	2533.98	594.16	15.25	378	ND
5.50	0.11	47.15	6.30	0.28	450	280
5.63	0.19	53.35	3.59	0.32	378	113

Integration Results for DAD1 B, Sig=214,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.27	0.07	54.59	12.16	0.32	102	ND
0.36	0.06	63.84	15.26	0.37	102	ND
0.54	0.18	177.57	13.08	1.03	102	ND
0.96	0.40	385.21	11.94	2.24	102	ND
1.48	0.53	600.55	14.32	3.49	100	ND
2.61	0.16	33.69	2.76	0.20	100	ND
2.91	0.10	29.88	4.04	0.17	100	ND
3.03	0.11	39.35	5.06	0.23	100	ND
3.19	0.08	10.37	1.66	0.06	100	ND
3.35	0.10	17.20	2.44	0.10	100	ND
3.55	0.07	365.09	80.83	2.12	197	ND
3.70	0.08	52.98	11.22	0.31	100	ND
3.87	0.09	72.97	12.75	0.42	100	ND
4.08	0.08	7546.27	1660.73	43.83	213	ND
4.30	0.10	45.03	6.41	0.26	100	ND
4.53	0.08	2084.71	440.76	12.11	100	ND
4.73	0.05	20.17	5.90	0.12	100	ND
4.89	0.08	270.22	51.19	1.57	100	ND
5.28	0.07	5126.36	1173.99	29.77	378	ND
5.50	0.11	98.92	13.79	0.57	450	280
5.62	0.09	54.15	8.45	0.31	378	ND
5.71	0.13	69.79	6.78	0.41	378	113



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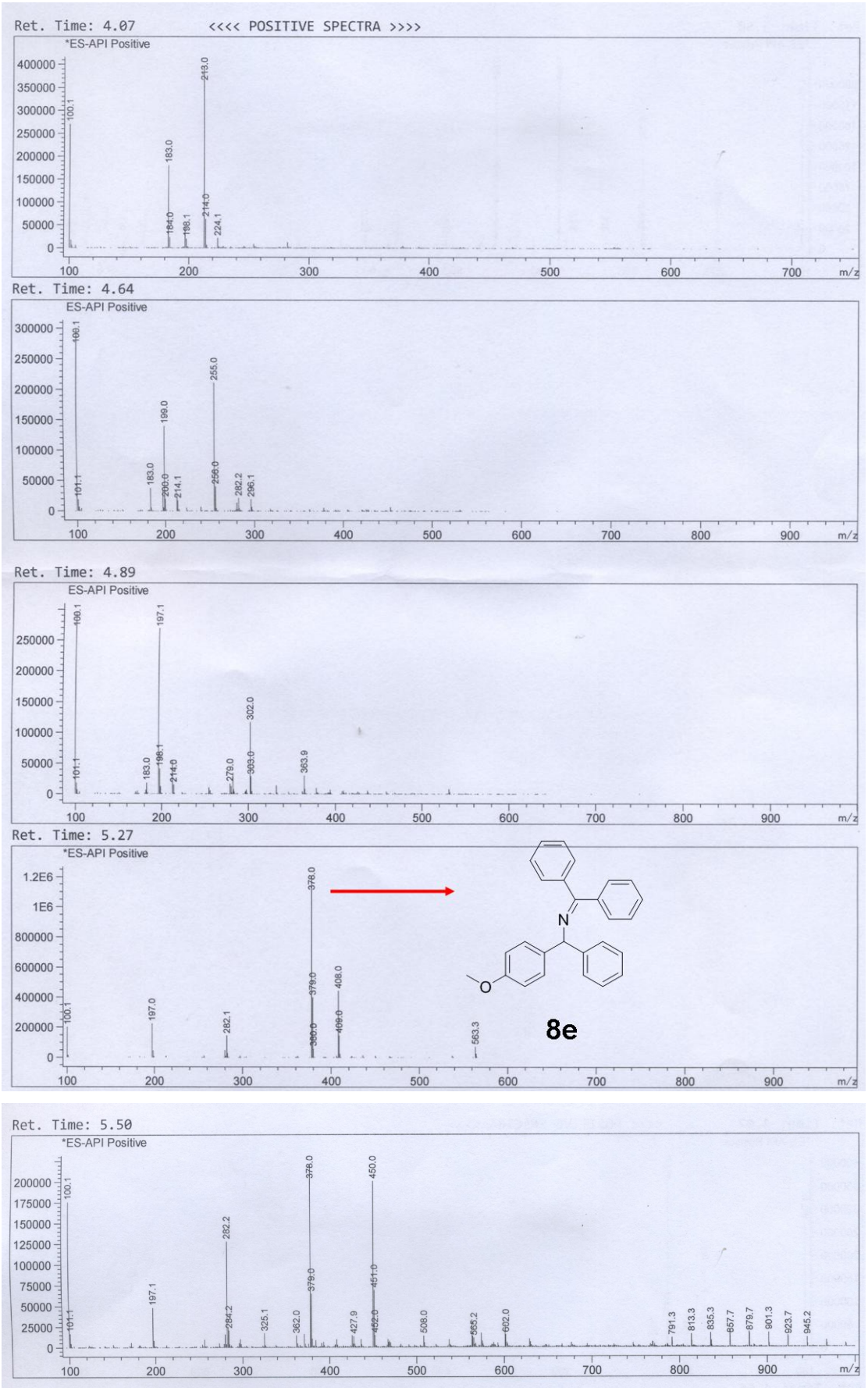
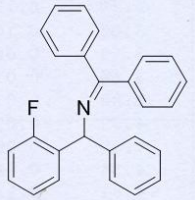
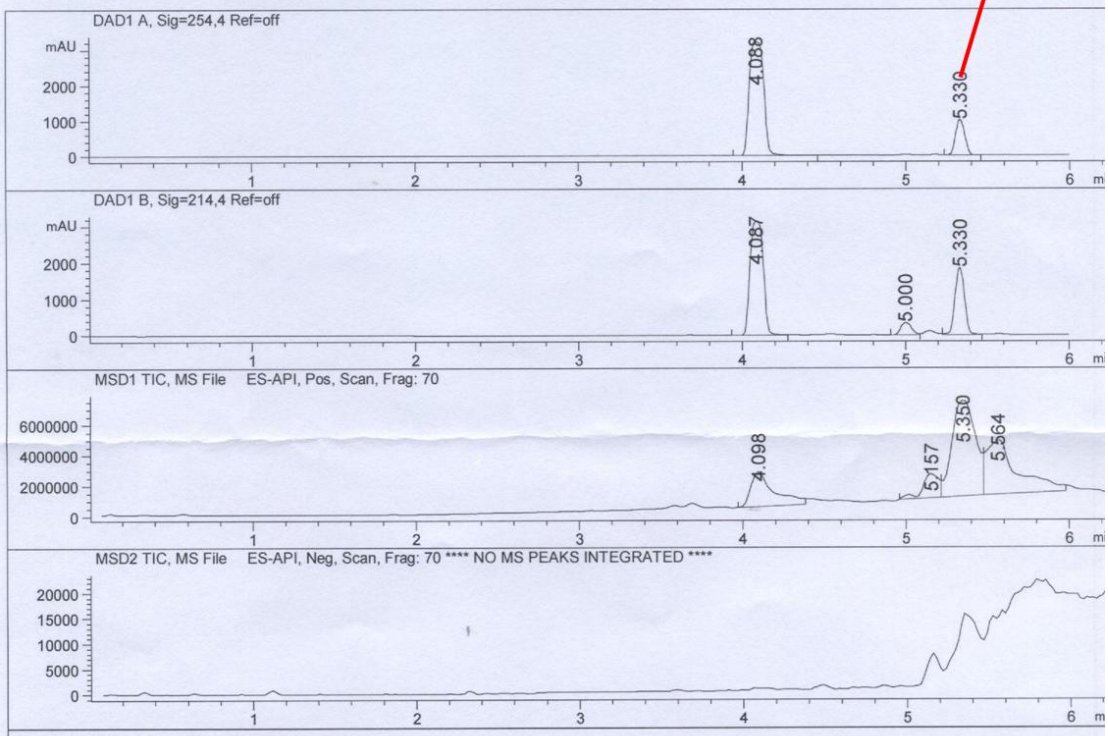

Ret. Time: 4.07
<<<< POSITIVE SPECTRA >>>>
*ES-API Positive
Ret. Time: 4.64
ES-API Positive
Ret. Time: 4.89
ES-API Positive
Ret. Time: 5.27
*ES-API Positive
8e
Ret. Time: 5.50
*ES-API Positive
m/z

Injection Date : 31-Jul-15, 10:05:56 Seq. Line : 0
Sample Name : 3F Location : P2-D-09
Acq. Operator : jd701092 Inj : 0
Spec. Reported : MS Integration Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%' Custom 1: Custom 2:
Method Info : Flow: 2.0 mL/min.
Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
Column Temperature: 50 degree.
MS Scan Range: m/z 100 - 1000. Scan Rate: 1.06 sec/cycle.
Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.



8f



Integration Results for DAD1 A, Sig=254,4 Ref=off

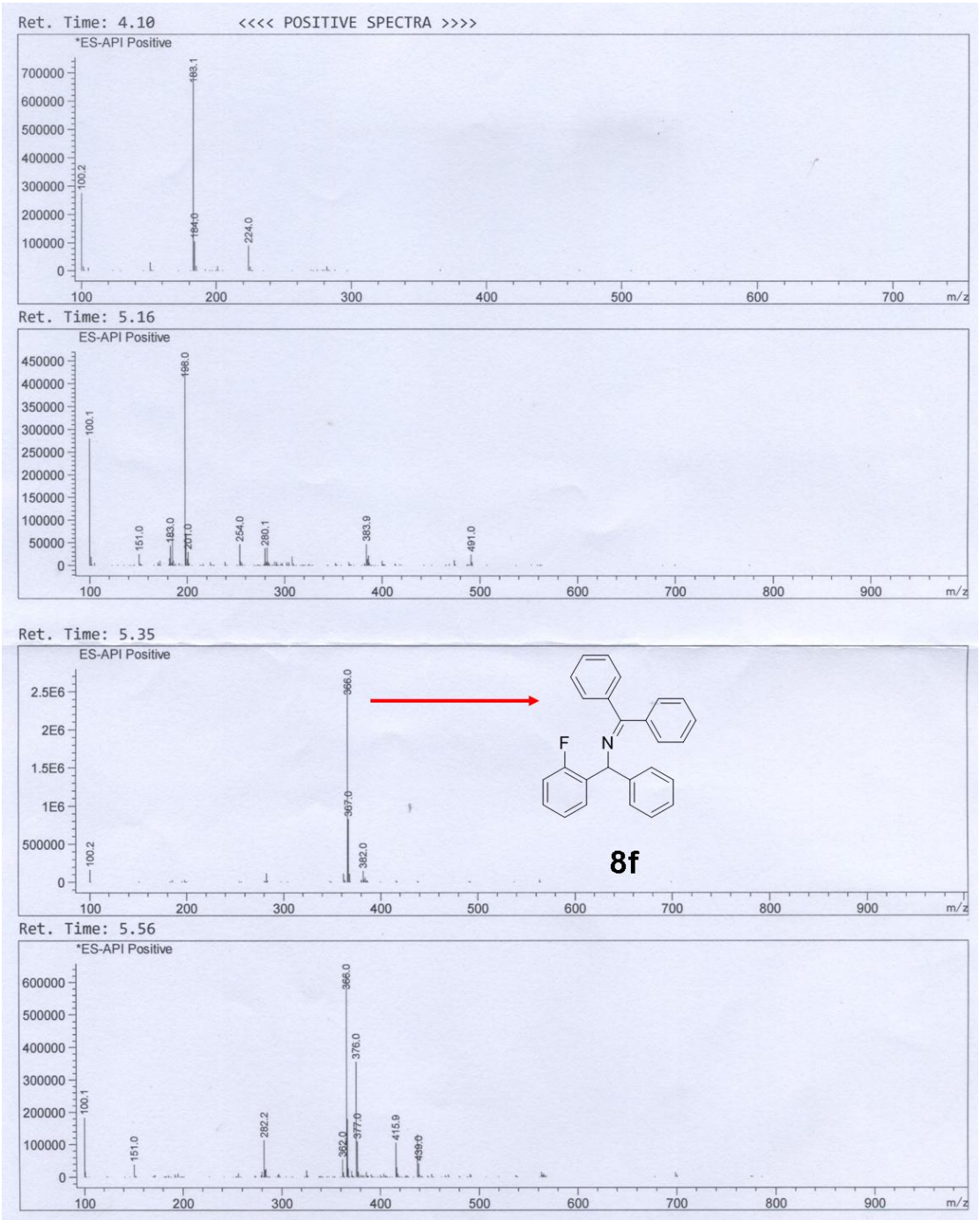
RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.36	0.06	40.57	10.39	0.17	151	ND
4.09	0.10	19553.84	3216.17	81.54	183	ND
4.54	0.11	122.32	15.07	0.51	100	ND
5.00	0.08	125.31	26.09	0.52	100	ND
5.17	0.08	86.67	17.75	0.36	198	ND
5.33	0.06	3984.56	1010.76	16.62	366	ND
5.57	0.11	64.52	8.35	0.27	366	ND
5.77	0.09	2.87	0.44	0.01	366	113

Integration Results for DAD1 B, Sig=214,4 Ref=off

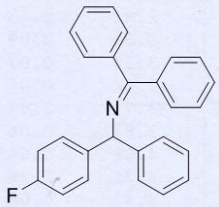
RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.28	0.11	89.02	12.10	0.31	151	ND
0.36	0.05	54.22	14.06	0.19	151	ND
0.53	0.19	209.47	14.27	0.72	151	ND
0.95	0.36	379.04	13.40	1.31	151	ND
1.46	0.23	248.92	13.86	0.86	151	ND
1.89	0.36	343.23	12.86	1.19	100	ND

2.79	0.13	11.33	1.20	0.04	100	ND
3.03	0.10	11.75	1.84	0.04	100	ND
3.58	0.06	17.76	4.54	0.06	100	ND
3.68	0.07	38.33	9.05	0.13	100	ND
3.88	0.07	3.70	0.81	0.01	100	ND
4.09	0.09	17469.09	3140.74	60.44	183	ND
4.30	0.08	50.05	9.88	0.17	183	ND
4.54	0.12	207.97	25.14	0.72	100	ND
4.81	0.12	45.98	5.40	0.16	100	ND
5.00	0.07	1556.35	343.46	5.38	100	ND
5.14	0.08	501.81	104.28	1.74	198	ND
5.33	0.07	7514.16	1847.53	26.00	366	ND
5.57	0.10	139.71	19.35	0.48	316	ND
5.75	0.11	10.46	1.30	0.04	366	113

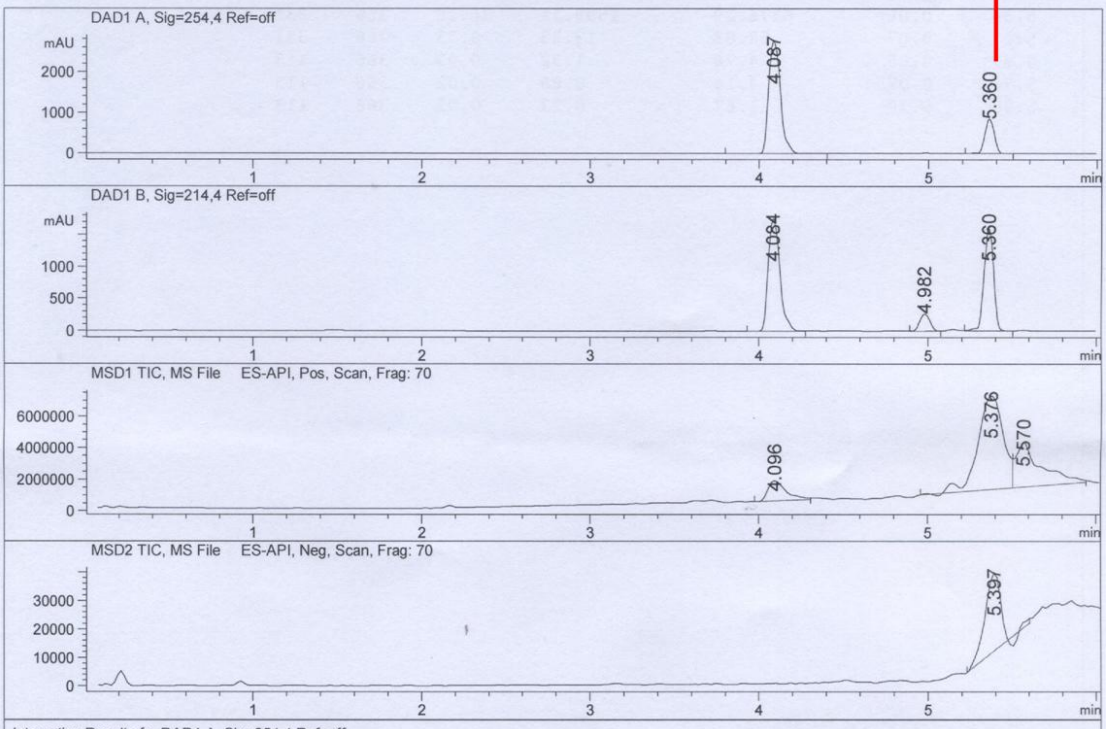
8f


Ret. Time: 4.10
<<<< POSITIVE SPECTRA >>>>
*ES-API Positive
Ret. Time: 5.16
ES-API Positive
Ret. Time: 5.35
ES-API Positive
F
N
8f
Ret. Time: 5.56
*ES-API Positive

```
Injection Date    : 31-Jul-15, 09:33:56                Seq. Line : 0
Sample Name       : 3G                                 Location  : P2-D-05
Acq. Operator     : jd701092                           Inj       : 0
Spec. Reported    : MS Integration                     Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8g



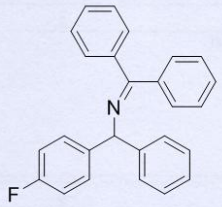
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.36	0.06	41.76	10.24	0.25	151	ND
4.09	0.08	13369.70	2762.86	79.30	100	ND
4.56	0.12	20.40	2.48	0.12	100	ND
4.70	0.08	7.20	1.26	0.04	100	ND
4.98	0.07	67.15	14.70	0.40	100	ND
5.17	0.07	24.30	5.74	0.14	198	ND
5.36	0.06	3304.70	844.39	19.60	366	331
5.57	0.07	24.03	5.41	0.14	366	331

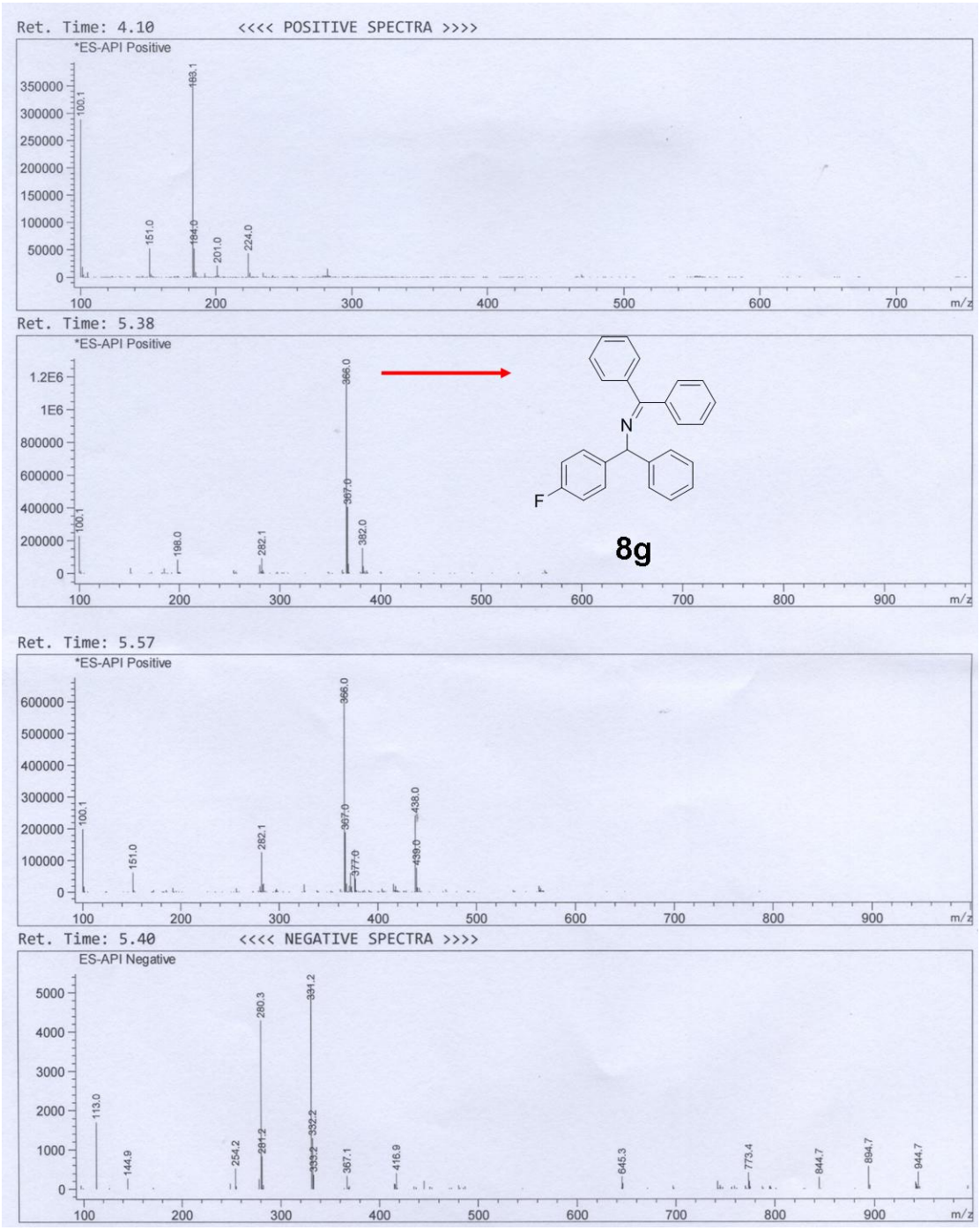
Integration Results for DAD1 B, Sig=214,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.28	0.11	89.70	11.70	0.51	151	ND
0.36	0.05	55.74	13.94	0.32	151	ND
0.54	0.19	204.03	14.04	1.16	151	ND
0.78	0.12	109.44	12.21	0.62	151	ND
0.95	0.30	302.51	13.13	1.72	151	ND
1.46	0.33	340.28	13.66	1.93	151	ND

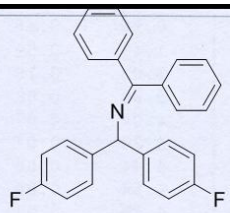
1.88	0.37	302.23	10.95	1.71	151	ND
2.81	0.13	12.68	1.27	0.07	100	ND
3.02	0.09	4.49	0.70	0.03	100	ND
3.14	0.07	1.53	0.31	0.01	100	ND
3.62	0.06	8.84	2.30	0.05	100	ND
3.71	0.07	26.02	6.01	0.15	100	ND
3.87	0.06	1.26	0.33	0.01	100	ND
4.08	0.08	8411.88	1761.98	47.69	183	ND
4.31	0.08	14.32	2.69	0.08	100	ND
4.47	0.08	27.87	5.07	0.16	100	ND
4.55	0.07	17.99	3.82	0.10	100	ND
4.83	0.11	18.59	2.50	0.11	100	ND
4.98	0.07	1125.89	265.23	6.38	100	ND
5.15	0.08	116.32	23.58	0.66	198	ND
5.36	0.06	6376.29	1598.11	36.15	366	331
5.57	0.07	58.83	13.33	0.33	366	331
5.67	0.05	4.76	1.32	0.03	366	113
5.76	0.07	4.14	0.85	0.02	366	113
5.90	0.10	1.67	0.23	0.01	366	113



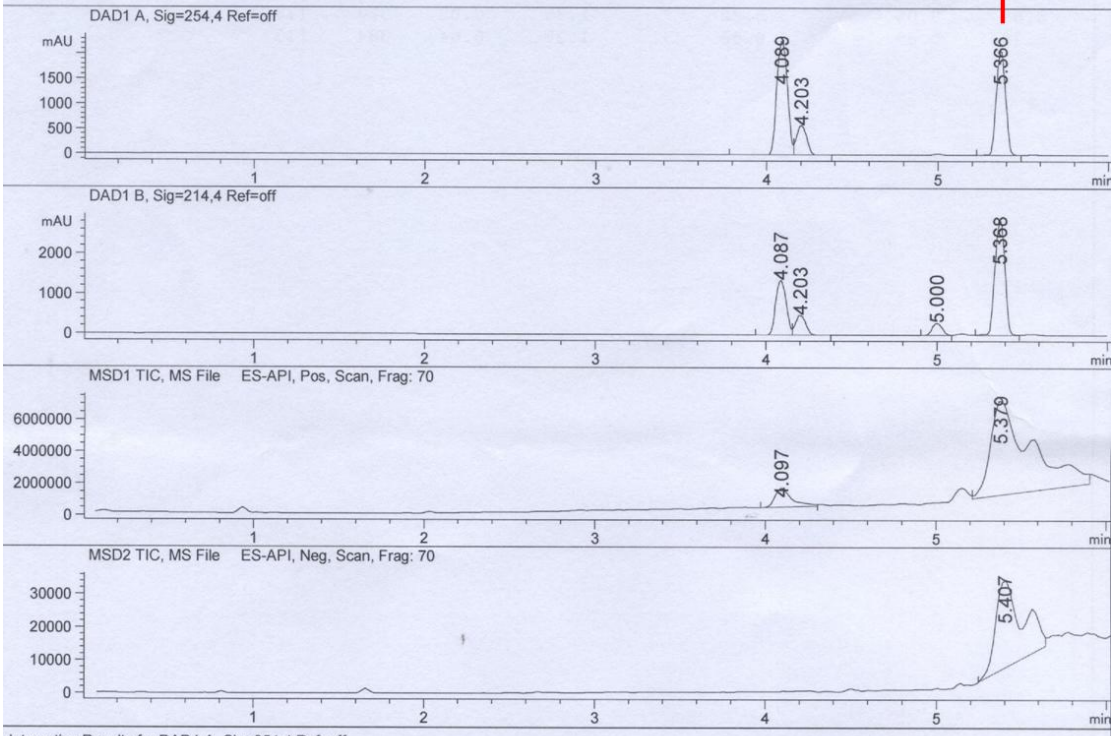
8g


Ret. Time: 4.10
<<<< POSITIVE SPECTRA >>>>
*ES-API Positive
Ret. Time: 5.38
*ES-API Positive
N
F
8g
Ret. Time: 5.57
*ES-API Positive
Ret. Time: 5.40
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative

Injection Date : 31-Jul-15, 11:02:01 Seq. Line : 0
Sample Name : 3H Location : P2-E-07
Acq. Operator : jd701092 Inj : 0
Spec. Reported : MS Integration Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%' Custom 1: Custom 2:
Method Info : Flow: 2.0 mL/min.
Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
Column Temperature: 50 degree.
MS Scan Range: m/z 100 - 1000. Scan Rate: 1.06 sec/cycle.
Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.



8h



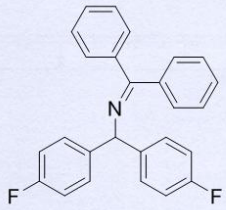
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	43.20	10.44	0.21	151	ND
4.09	0.07	9914.14	2286.56	48.07	100	ND
4.20	0.06	2415.52	587.66	11.71	100	ND
4.43	0.08	13.81	2.62	0.07	100	ND
4.56	0.10	29.57	4.43	0.14	100	ND
4.76	0.10	7.96	1.11	0.04	100	ND
5.00	0.07	101.77	22.05	0.49	100	ND
5.17	0.08	26.86	5.19	0.13	198	ND
5.37	0.06	7969.64	2093.46	38.64	384	280
5.56	0.09	69.95	11.15	0.34	384	331
5.78	0.17	31.07	2.38	0.15	384	113

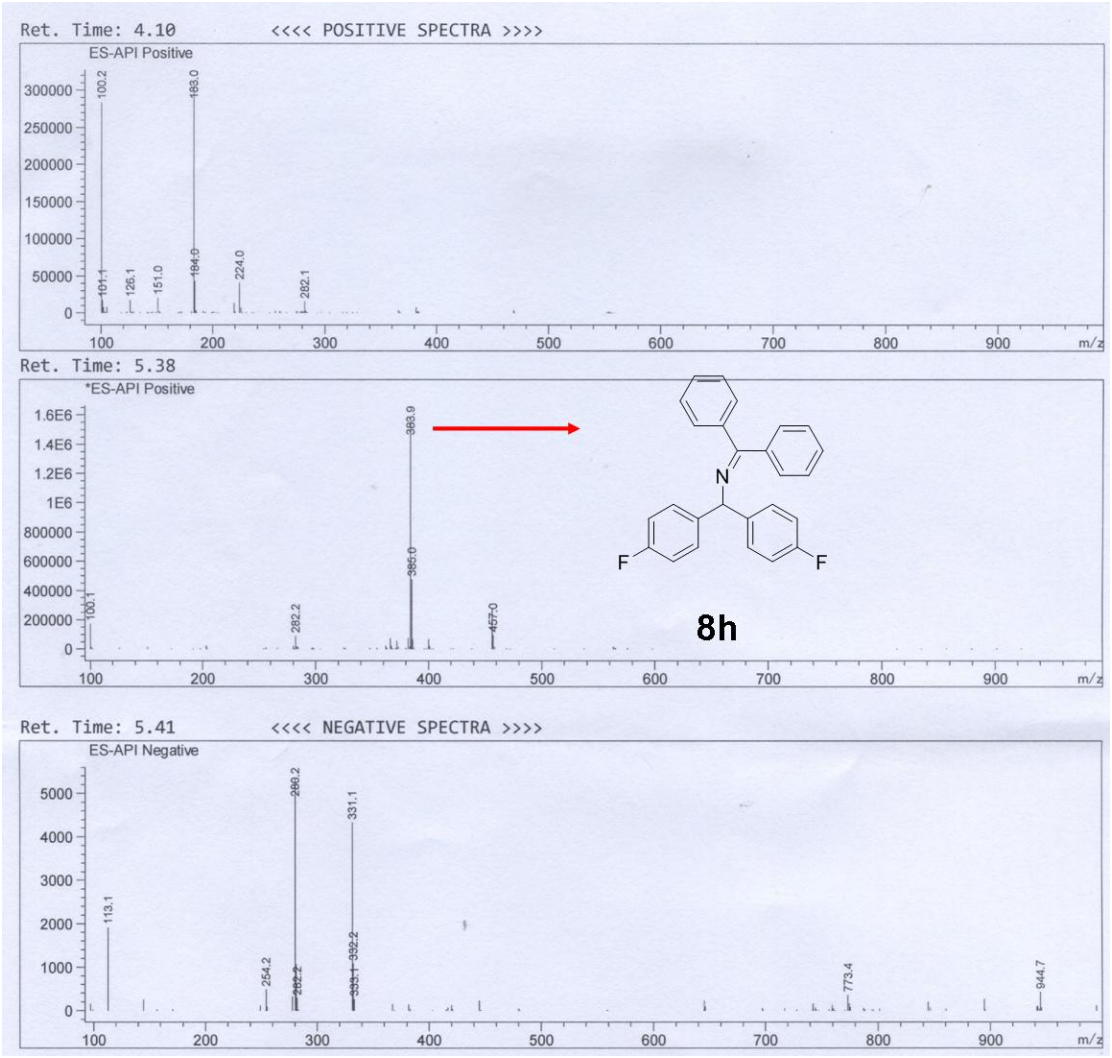
Integration Results for DAD1 B, Sig=214,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.29	0.07	48.69	11.05	0.22	102	ND
0.37	0.05	55.10	13.99	0.24	151	ND
0.54	0.18	201.85	14.12	0.89	151	ND

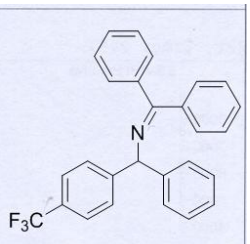
0.96	0.46	493.20	13.44	2.19	648	ND
1.48	0.59	649.73	13.94	2.88	100	ND
3.03	0.16	8.79	0.74	0.04	100	ND
3.80	0.08	5.04	0.93	0.02	100	ND
4.09	0.07	5822.75	1357.06	25.81	183	ND
4.20	0.07	2045.40	488.65	9.07	100	ND
4.44	0.06	10.71	2.55	0.05	100	ND
4.54	0.09	29.39	5.27	0.13	100	ND
4.76	0.07	7.79	1.77	0.03	100	ND
4.88	0.06	1.60	0.44	0.01	100	ND
5.00	0.07	1201.79	294.12	5.33	100	ND
5.15	0.07	134.46	29.38	0.60	198	ND
5.37	0.07	11702.71	2852.22	51.88	384	280
5.56	0.09	124.60	21.79	0.55	384	331
5.69	0.06	5.22	1.38	0.02	384	113
5.78	0.09	8.58	1.39	0.04	384	113



8h


Ret. Time: 4.10
<<<< POSITIVE SPECTRA >>>>
ES-API Positive
Ret. Time: 5.38
*ES-API Positive
N
F
F
8h
Ret. Time: 5.41
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative
m/z

Injection Date : 30-Jul-15, 16:26:06 Seq. Line : 0
Sample Name : 3I-7 Location : P1-B-05
Acq. Operator : jd701092 Inj : 0
Spec. Reported : MS Integration Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\METHODS\5MIN_POS.M
Analysis Method : C:\Chem32\1\METHODS\5MIN_POS.M
Sample Info : Easy-Access Method: '5MIN_5%-95%_POS.M'
Method Info : Flow: 1.5 ml/min.
Gradient: B%: 0.0 min, 5%; 4.5 min, 95%; 5.0 min, 95%.
Column: Agilent SB-C18, 1.8 um, 4.6*30 mm.
Column Temperature: 50 degree.
MS Scan Range: m/z 100 - 1000.
Scan Rate: 1.06 sec/cycle.



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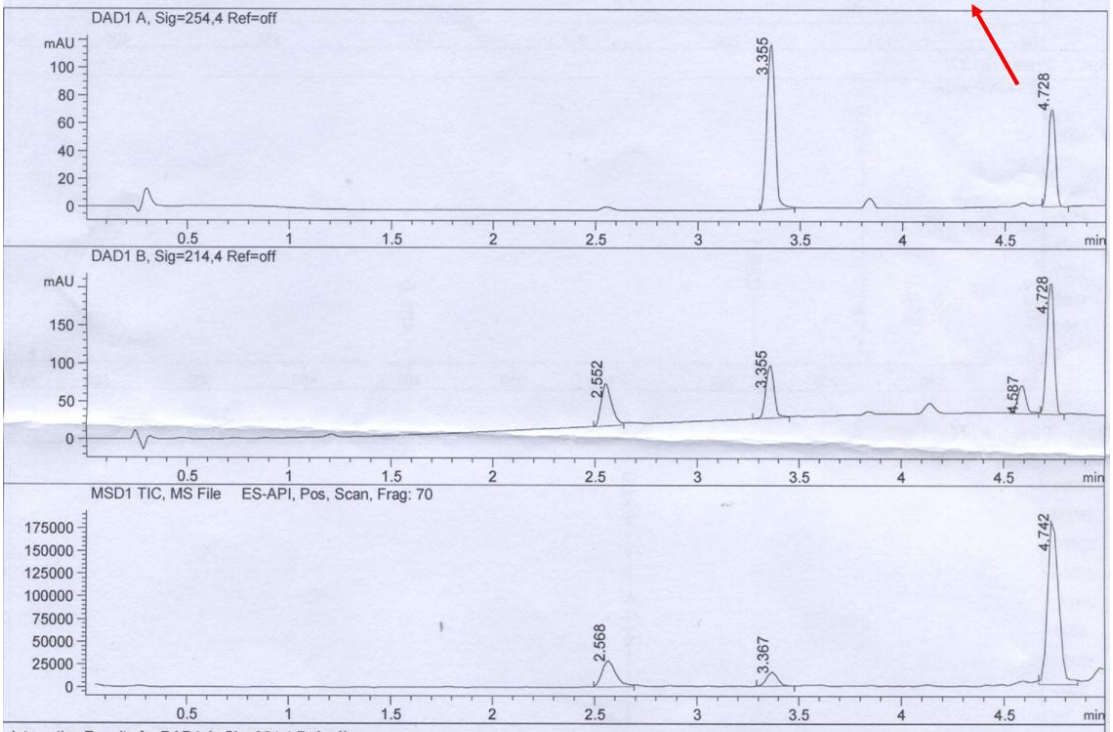
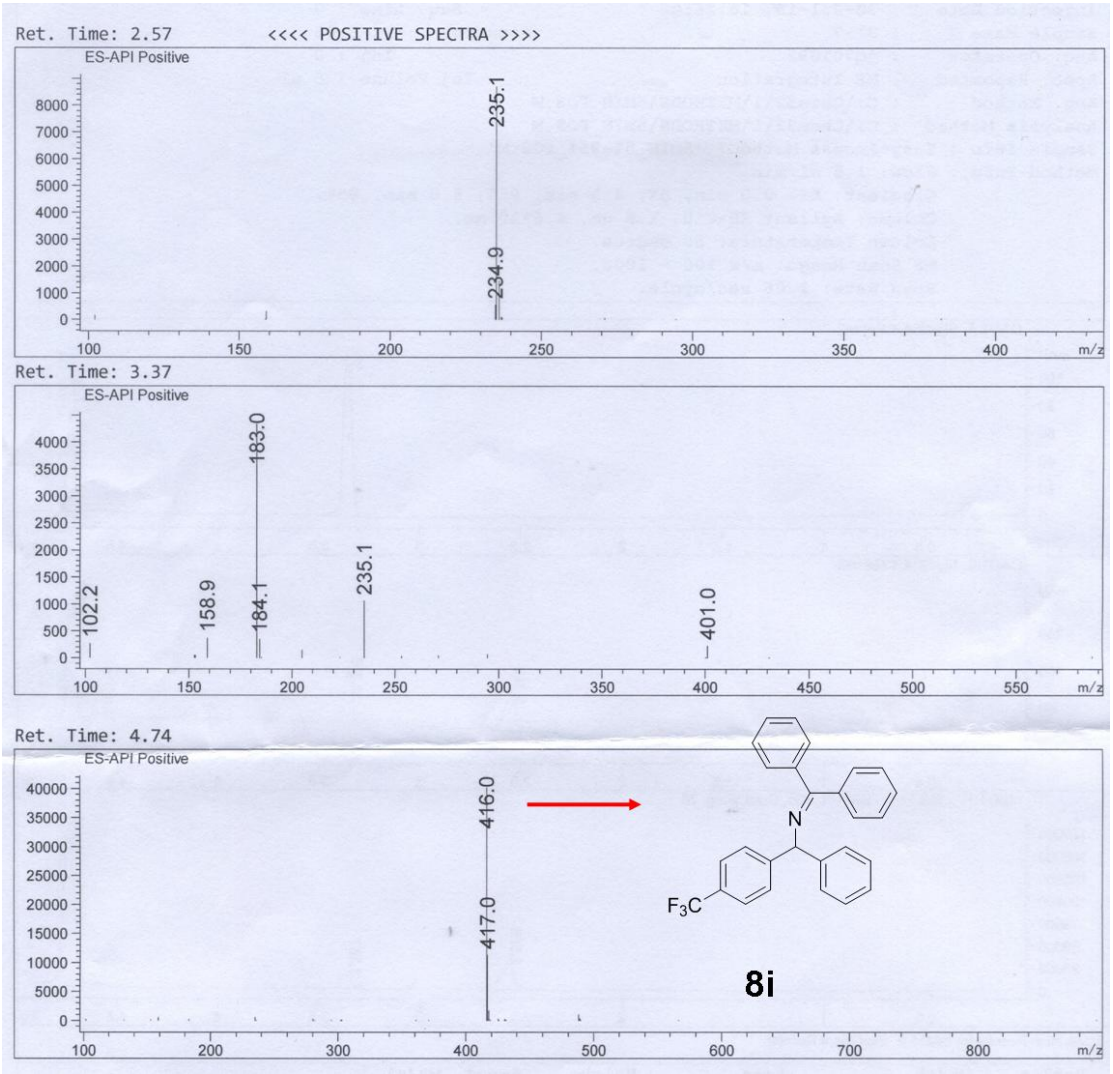


Integration Results for DAD1 A, Sig=254,4 Ref=off

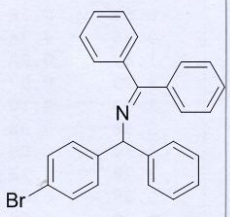
RetTim	Width	Area	Height	Area%	MS(+)
2.55	0.06	9.36	2.62	1.70	235
3.36	0.04	331.34	119.23	60.34	183
3.84	0.04	18.34	7.07	3.34	ND
4.59	0.06	10.43	2.57	1.90	ND
4.73	0.04	179.66	70.46	32.72	416

Integration Results for DAD1 B, Sig=214,4 Ref=off

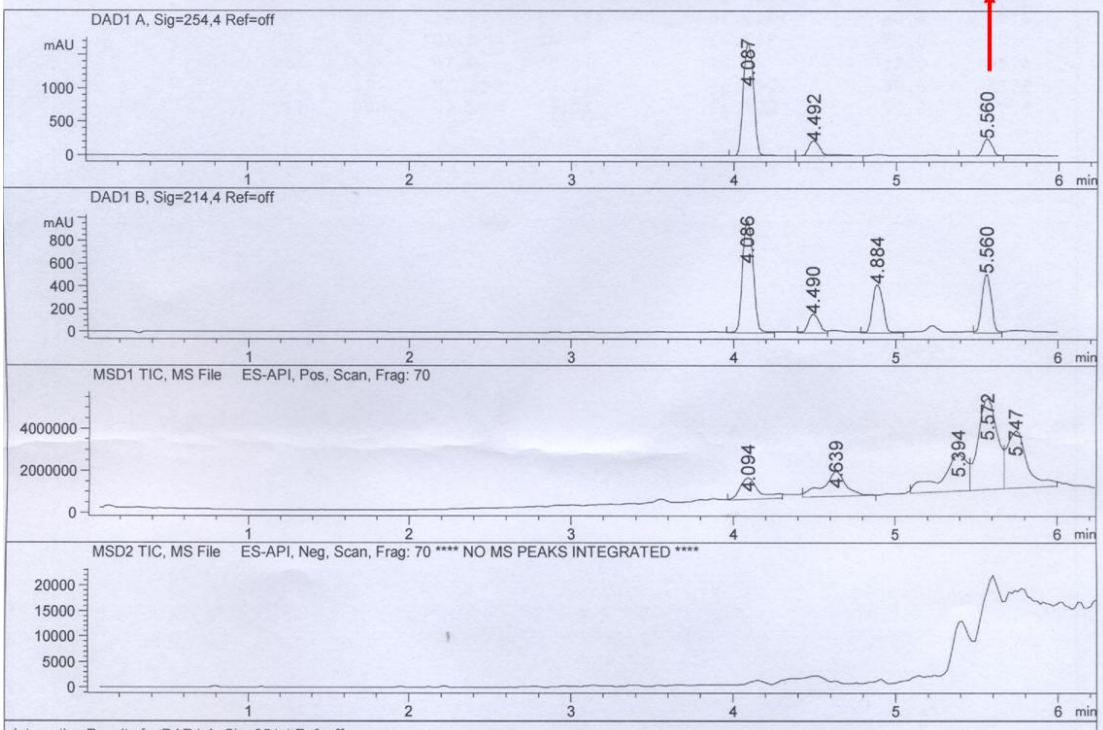
RetTim	Width	Area	Height	Area%	MS(+)
1.21	0.05	11.90	3.71	1.16	ND
2.55	0.06	202.37	55.70	19.70	235
3.36	0.04	194.88	69.02	18.97	183
3.84	0.05	14.03	4.40	1.37	ND
4.14	0.05	47.20	13.68	4.59	ND
4.59	0.05	106.89	36.86	10.40	ND
4.73	0.04	443.19	173.20	43.14	416
4.83	0.06	6.95	1.55	0.68	416


Ret. Time: 2.57
<<<< POSITIVE SPECTRA >>>>
ES-API Positive
235.1
234.9
m/z
Ret. Time: 3.37
ES-API Positive
183.0
235.1
102.2
158.9
184.1
401.0
m/z
Ret. Time: 4.74
ES-API Positive
416.0
417.0
N
F3C
8i
m/z

```
Injection Date    : 03-Aug-15, 11:12:19                Seq. Line : 0
Sample Name       : 3J                              Location  : P1-E-01
Acq. Operator     : jd701092                            Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8j



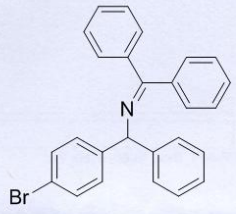
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.26	0.08	47.29	8.33	0.48	102	ND
0.37	0.07	68.26	13.98	0.69	102	ND
3.21	0.14	4.68	0.45	0.05	100	ND
3.55	0.10	10.61	1.49	0.11	100	ND
3.78	0.10	9.28	1.37	0.09	100	ND
3.88	0.09	14.63	2.59	0.15	100	ND
4.09	0.07	7430.37	1679.30	74.82	100	ND
4.49	0.08	1113.13	217.13	11.21	100	ND
4.89	0.09	84.14	14.96	0.85	100	ND
5.22	0.14	58.66	5.75	0.59	100	ND
5.56	0.06	999.63	248.32	10.07	428	280
5.74	0.14	90.84	8.68	0.91	500	113

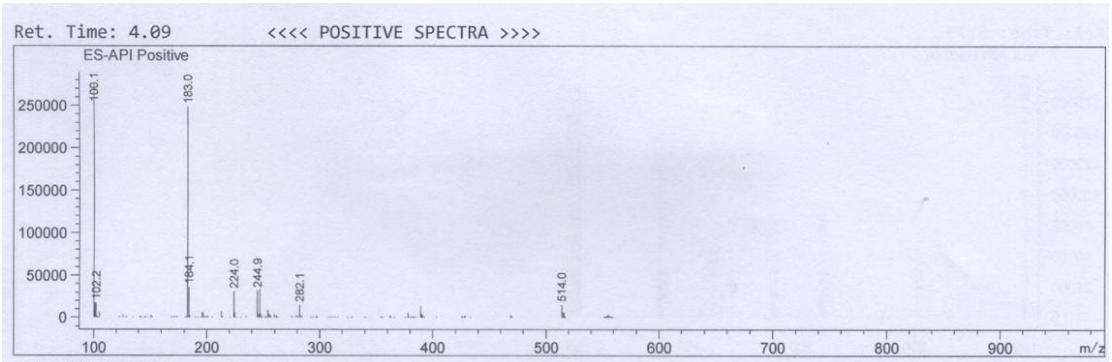
Integration Results for DAD1 B, Sig=214,4 Ref=off

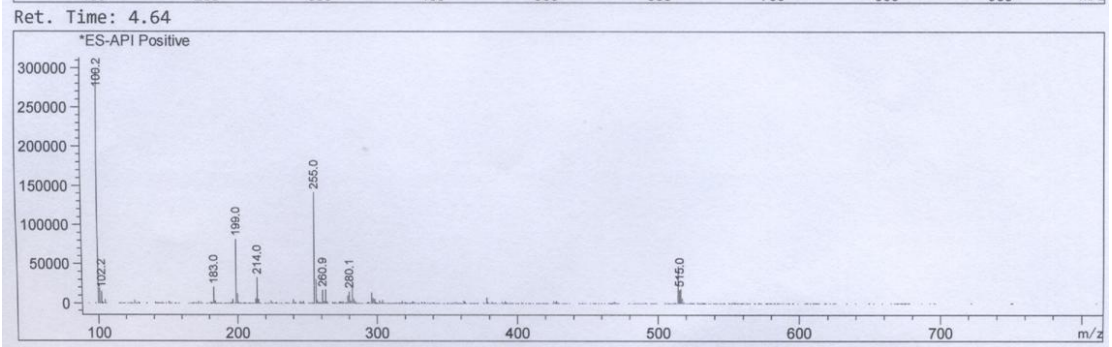
RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.05	0.18	19.54	1.34	0.18	102	ND
0.27	0.08	56.18	11.10	0.52	102	ND

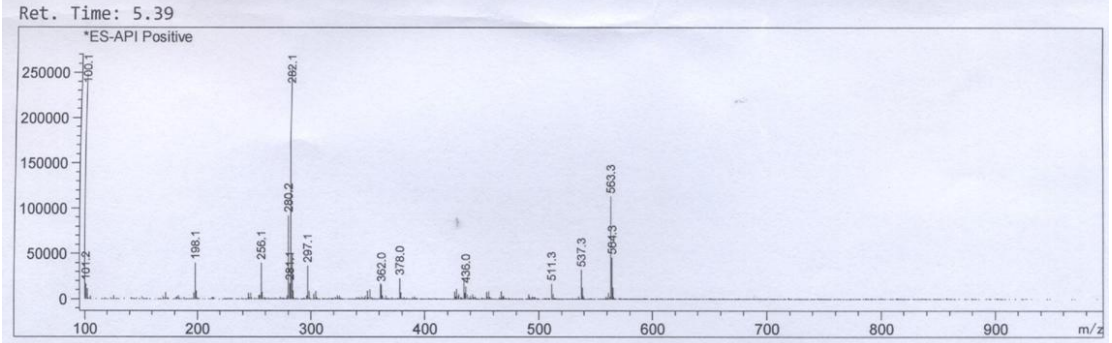
0.36	0.06	63.21	15.46	0.58	102	ND
0.54	0.18	169.08	12.38	1.55	102	ND
0.96	0.38	270.02	9.02	2.48	102	ND
1.47	0.22	117.31	6.69	1.08	102	ND
2.90	0.07	3.58	0.89	0.03	100	ND
3.03	0.10	6.85	0.89	0.06	100	ND
3.38	0.08	3.78	0.68	0.03	100	ND
3.54	0.08	37.47	7.72	0.34	100	ND
3.78	0.10	19.13	2.85	0.18	100	ND
3.88	0.09	32.81	5.75	0.30	100	ND
4.09	0.08	4487.42	985.72	41.17	183	ND
4.30	0.11	43.68	5.34	0.40	100	ND
4.49	0.08	766.85	160.80	7.04	100	ND
4.62	0.10	137.94	19.65	1.27	100	ND
4.88	0.08	1919.10	422.13	17.61	100	ND
5.22	0.09	347.79	61.02	3.19	100	ND
5.46	0.11	83.04	10.35	0.76	282	280
5.56	0.06	2046.55	512.05	18.78	428	331
5.74	0.17	267.77	20.81	2.46	500	113

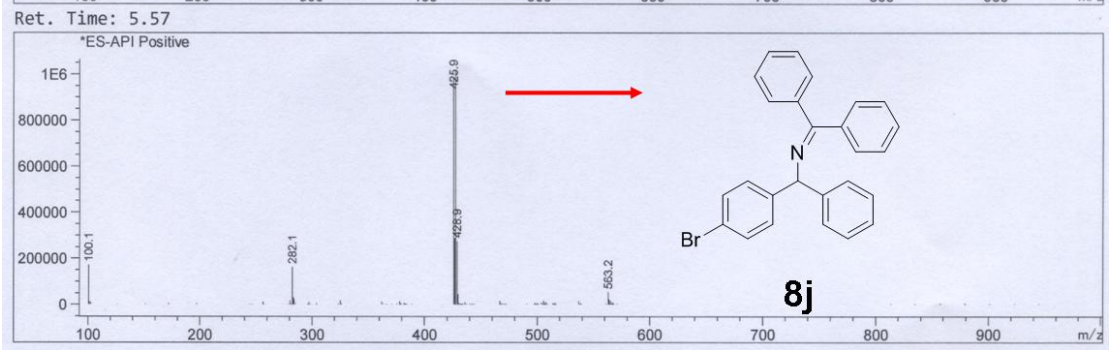


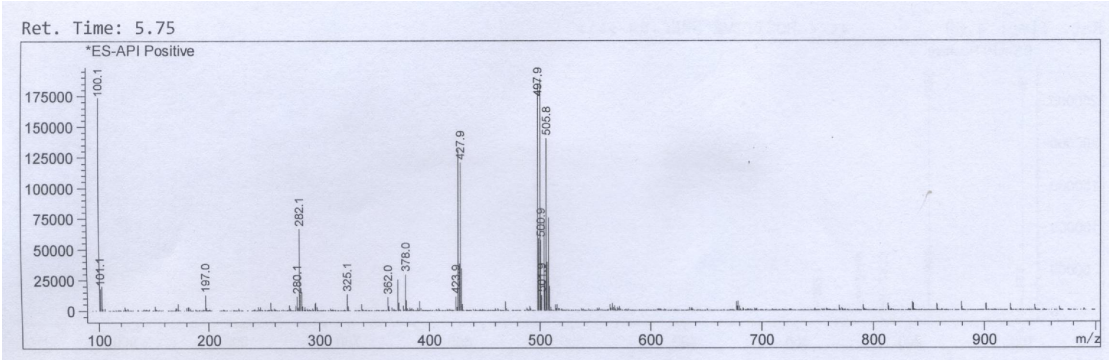
8j


Ret. Time: 4.09
<<<< POSITIVE SPECTRA >>>>
ES-API Positive
m/z

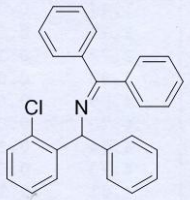

Ret. Time: 4.64
*ES-API Positive
m/z


Ret. Time: 5.39
*ES-API Positive
m/z

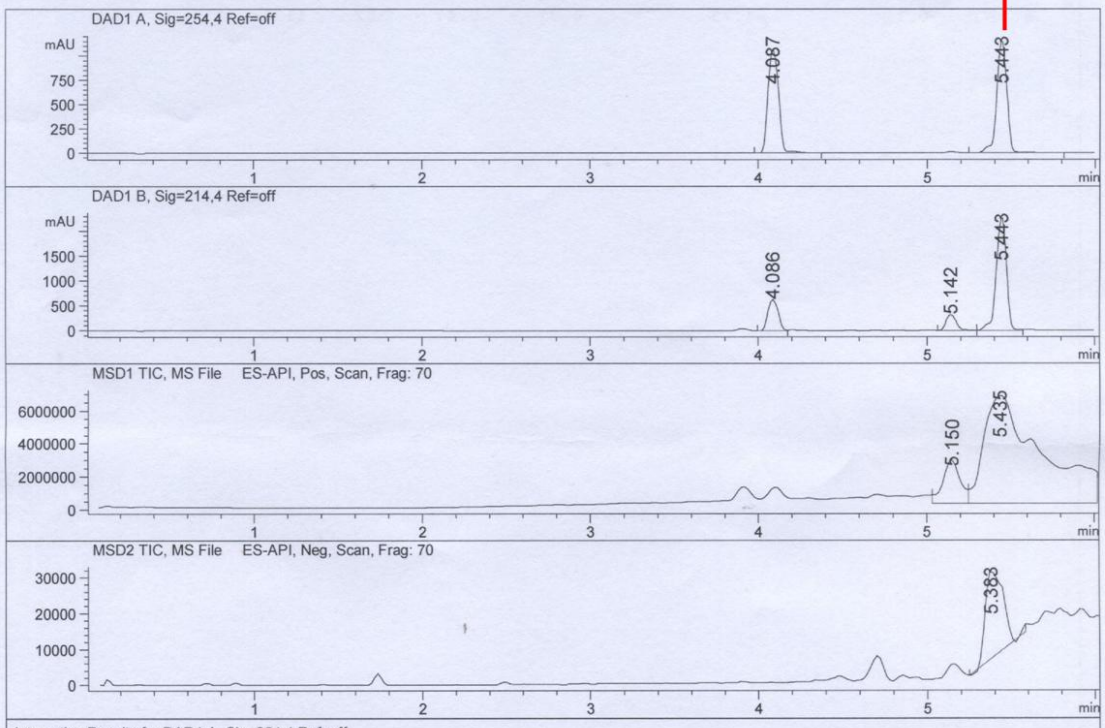

Ret. Time: 5.57
*ES-API Positive
Br
N
8j
m/z


Ret. Time: 5.75
*ES-API Positive
m/z

```
Injection Date    : 31-Jul-15, 10:21:57                Seq. Line : 0
Sample Name       : 3K                              Location  : P2-E-02
Acq. Operator     : jd701092                            Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8k



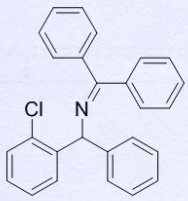
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	42.04	10.04	0.43	102	ND
3.91	0.09	4.99	0.88	0.05	201	ND
4.09	0.07	4659.49	1070.79	48.07	100	ND
4.55	0.15	35.15	3.14	0.36	100	ND
4.70	0.09	10.11	1.70	0.10	100	ND
4.84	0.12	8.36	1.00	0.09	100	ND
5.00	0.09	9.54	1.50	0.10	100	ND
5.14	0.08	54.81	11.07	0.57	198	ND
5.44	0.07	4845.15	1159.02	49.99	382	ND
5.91	0.13	23.17	2.30	0.24	382	113

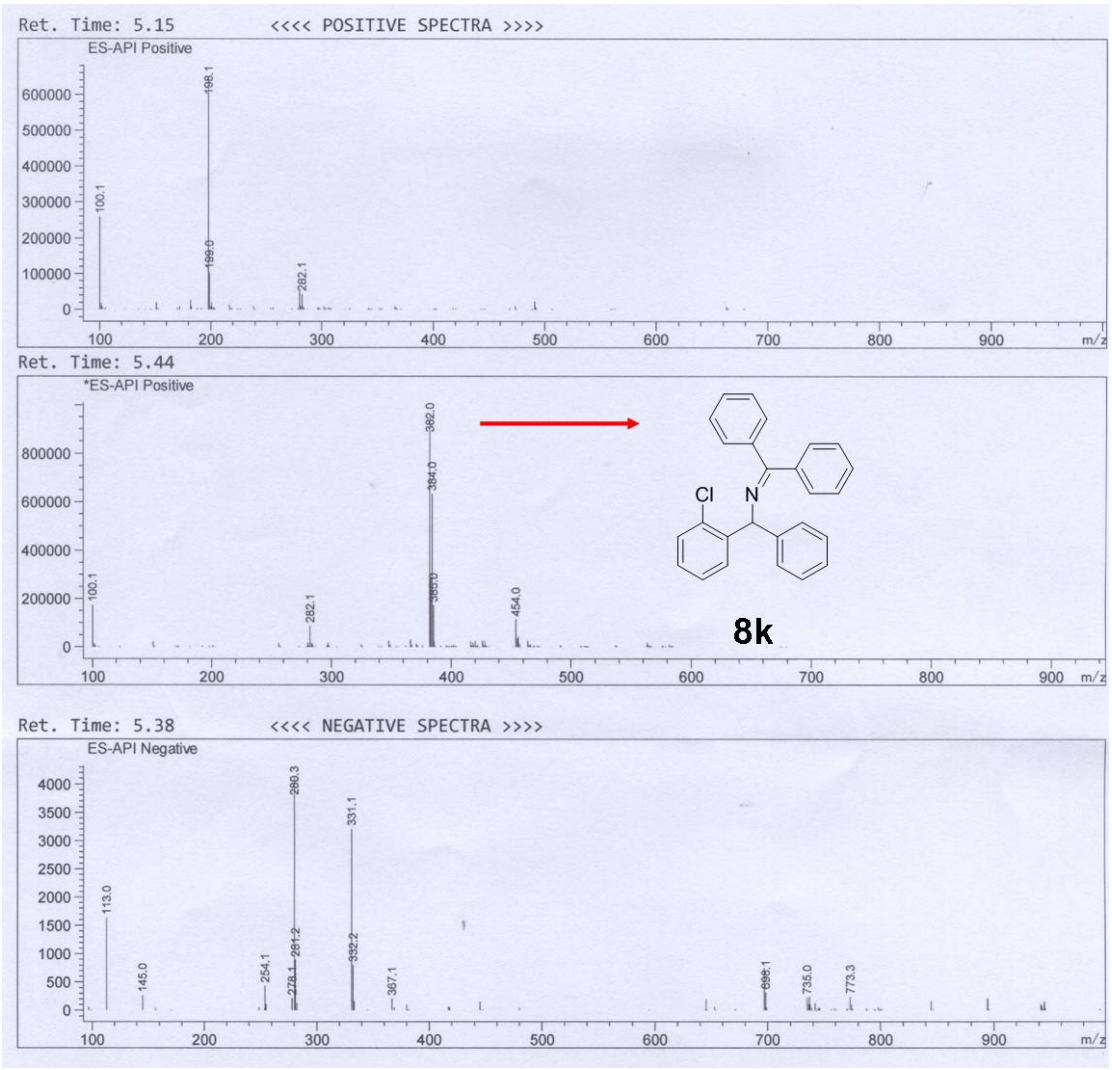
Integration Results for DAD1 B, Sig=214,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.28	0.06	47.18	11.36	0.30	102	ND
0.36	0.05	55.22	14.10	0.35	102	ND
0.54	0.18	199.27	14.26	1.25	102	ND
0.95	0.45	494.54	13.52	3.10	151	ND

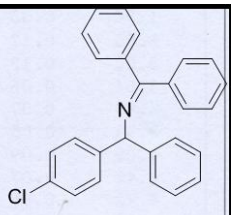
1.47	0.55	632.94	13.93	3.96	100	ND
2.79	0.14	12.14	1.12	0.08	100	ND
3.72	0.11	7.30	0.96	0.05	100	ND
3.90	0.08	186.01	40.13	1.17	100	ND
4.09	0.07	2676.51	624.43	16.77	183	ND
4.20	0.09	99.04	16.07	0.62	100	ND
4.54	0.14	62.62	6.52	0.39	100	ND
4.70	0.08	38.01	7.22	0.24	100	ND
4.81	0.11	58.63	7.30	0.37	100	ND
5.00	0.08	60.06	11.39	0.38	100	ND
5.14	0.07	1396.47	321.84	8.75	198	ND
5.44	0.07	9667.93	2278.12	60.56	382	280
5.59	0.08	131.79	22.17	0.83	382	113
5.73	0.10	73.68	10.26	0.46	382	113
5.92	0.11	64.79	8.31	0.41	382	113



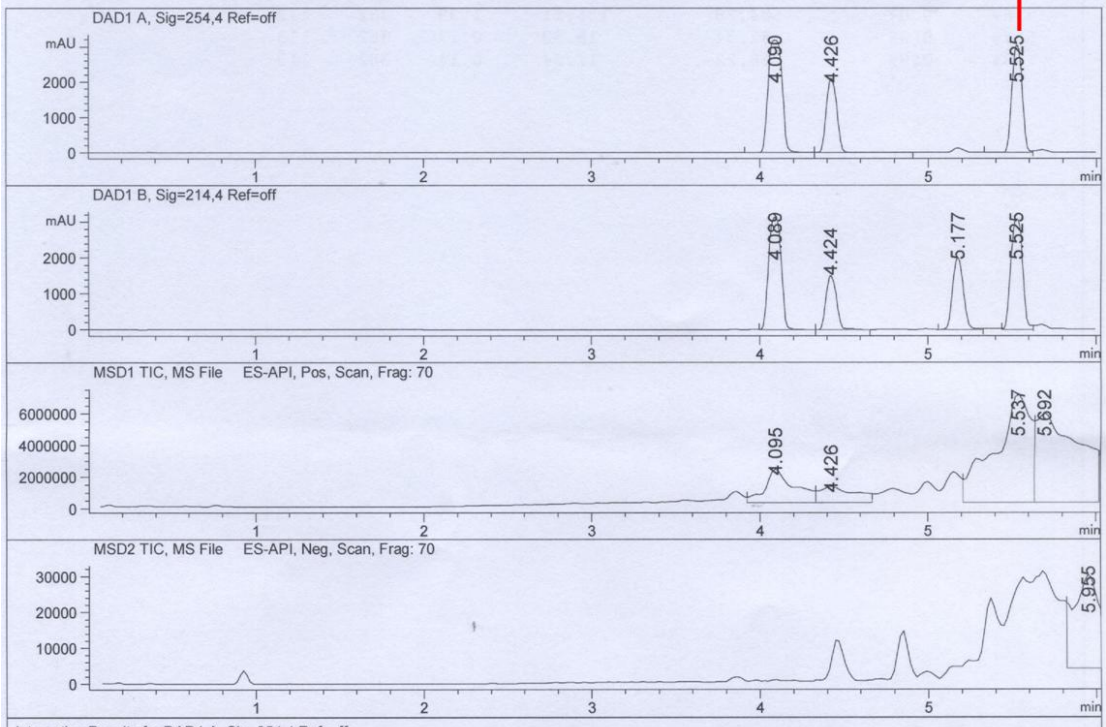
8k


Ret. Time: 5.15
<<<< POSITIVE SPECTRA >>>>
ES-API Positive
198.1
100.1
199.0
282.1
Ret. Time: 5.44
*ES-API Positive
382.0
384.0
100.1
282.1
454.0
Cl
N
8k
Ret. Time: 5.38
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative
280.3
331.1
113.0
145.0
254.1
279.1
281.2
332.2
367.1
688.1
735.0
773.3
m/z

```
Injection Date    : 31-Jul-15, 10:29:57                Seq. Line : 0
Sample Name       : 3L                              Location  : P2-E-03
Acq. Operator     : jd701092                            Inj : 0
Spec. Reported    : MS Integration                  Inj Volume : 5 ul
Acq. Method       : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method   : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%'  Custom 1:   Custom 2:
Method Info : Flow: 2.0 mL/min.
              Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
              Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
              Column Temperature: 50 degree.
              MS Scan Range: m/z 100 - 1000.   Scan Rate: 1.06 sec/cycle.
              Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.
```



8l



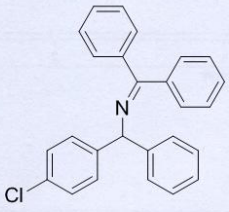
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	40.97	10.42	0.10	151	ND
3.55	0.14	6.96	0.66	0.02	100	ND
3.85	0.13	6.85	0.72	0.02	100	ND
4.09	0.09	18149.02	3186.46	43.98	183	ND
4.43	0.07	9199.00	2100.12	22.29	100	ND
4.99	0.09	52.54	8.30	0.13	100	ND
5.18	0.08	583.68	122.79	1.41	378	ND
5.53	0.07	12802.23	3052.28	31.02	382	331
5.67	0.09	429.68	73.03	1.04	382	113

Integration Results for DAD1 B, Sig=214,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.27	0.07	51.31	10.83	0.10	151	ND
0.36	0.05	54.03	13.85	0.11	151	ND
0.54	0.17	189.28	13.94	0.39	151	ND
0.97	0.45	481.67	13.42	0.98	151	ND
1.47	0.30	327.20	13.77	0.67	151	ND

1.90	0.35	338.61	12.72	0.69	100	ND
2.80	0.12	11.90	1.33	0.02	100	ND
3.03	0.12	11.58	1.46	0.02	100	ND
3.54	0.06	11.70	2.99	0.02	100	ND
3.85	0.07	53.35	12.76	0.11	100	ND
3.97	0.05	55.20	16.86	0.11	100	ND
4.09	0.09	16185.86	3134.99	32.94	183	ND
4.42	0.07	6557.46	1507.54	13.34	100	ND
4.72	0.05	16.80	5.27	0.03	100	ND
4.81	0.10	78.50	11.35	0.16	100	ND
4.99	0.07	76.60	16.94	0.16	453	ND
5.18	0.07	8841.89	2007.42	17.99	378	ND
5.42	0.08	145.29	25.09	0.30	282	280
5.53	0.08	14816.15	3070.52	30.15	382	331
5.67	0.07	684.28	135.51	1.39	382	113
5.78	0.08	81.34	15.52	0.17	382	113
5.94	0.09	68.22	11.64	0.14	382	113



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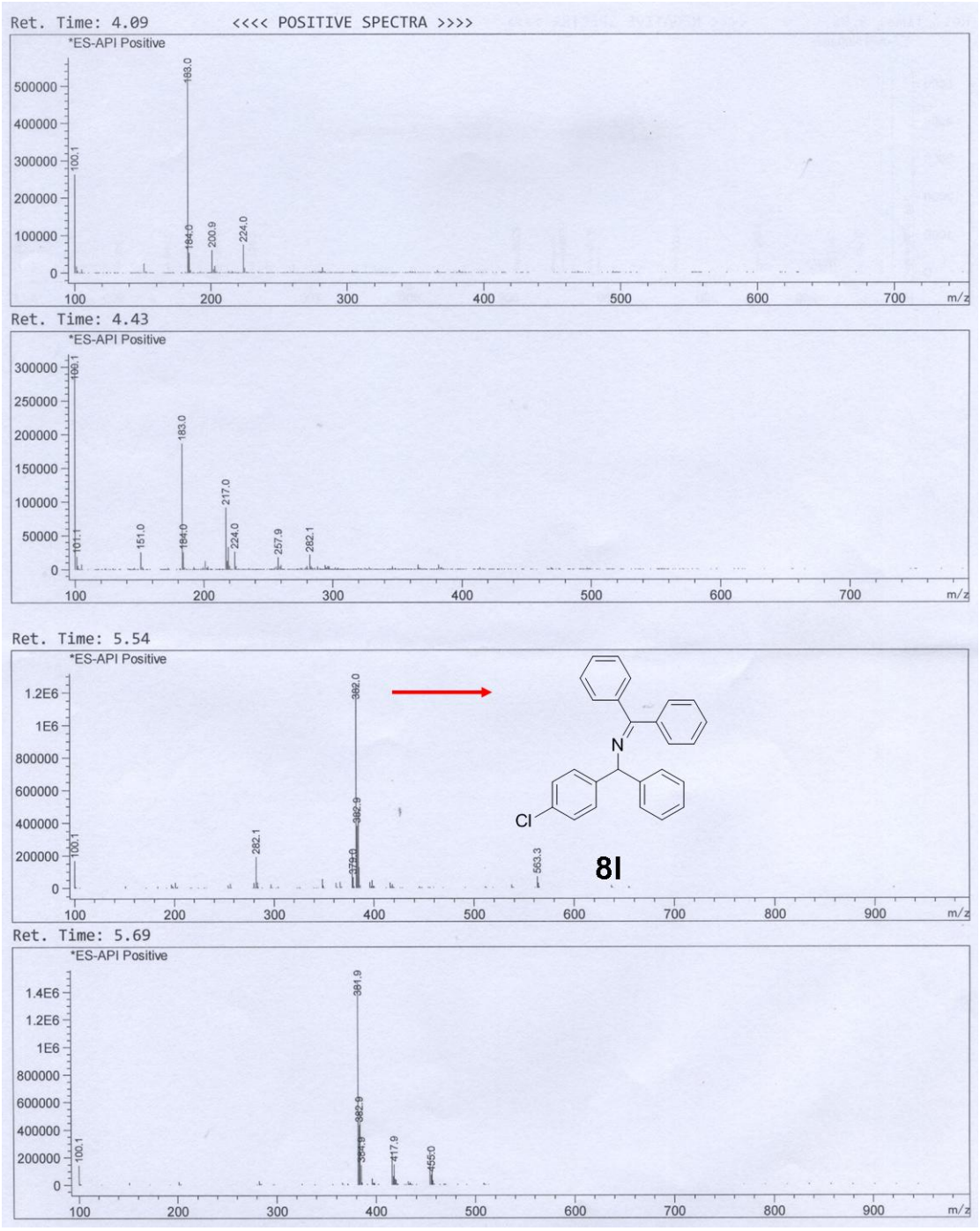
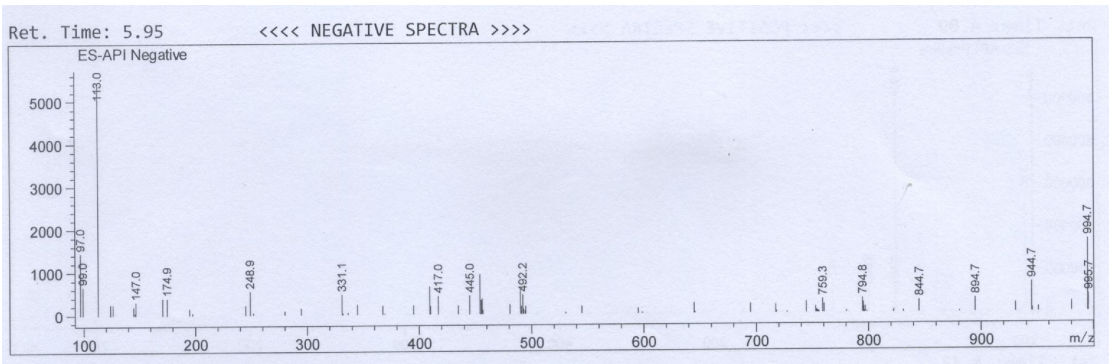
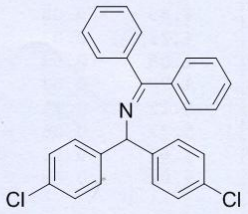
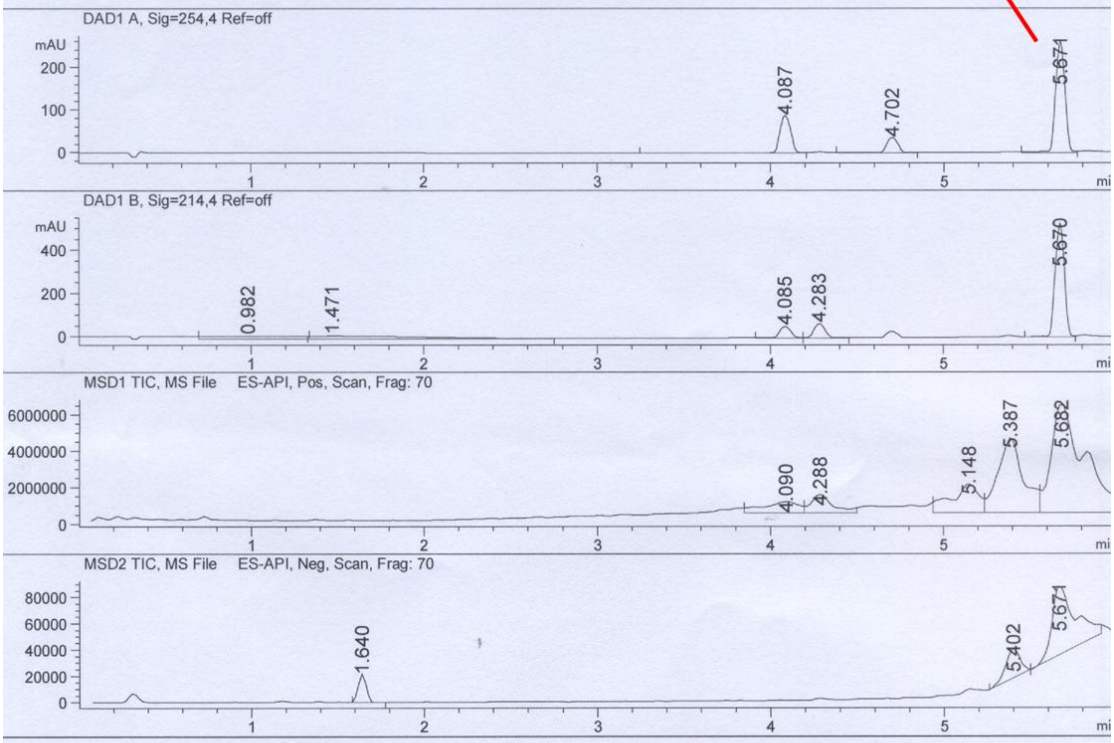

Ret. Time: 4.09
<<<< POSITIVE SPECTRA >>>>
*ES-API Positive
Ret. Time: 4.43
*ES-API Positive
Ret. Time: 5.54
*ES-API Positive
N
Cl
8l
Ret. Time: 5.69
*ES-API Positive
m/z


Ret. Time: 5.95
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative
m/z

Injection Date : 31-Jul-15, 09:13:33 Seq. Line : 0
Sample Name : 3M Location : P2-D-03
Acq. Operator : jd701092 Inj : 0
Spec. Reported : MS Integration Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Analysis Method : C:\Chem32\1\METHODS\5_95_5MIN_NEG.M
Sample Info : Easy-Access Method: '5MIN_5%-95%' Custom 1: Custom 2:
Method Info : Flow: 2.0 mL/min.
Gradient: B%: 1.0 min, 5%; 5.0 min, 95%; 6.0 min, 95%.
Column: Agilent SB-C18, 3.5 um, 4.6*50 mm.
Column Temperature: 50 degree.
MS Scan Range: m/z 100 - 1000. Scan Rate: 1.06 sec/cycle.
Instrument information: Agilent 1200 Series + 6140 Quadrupole LC/MS.



8m



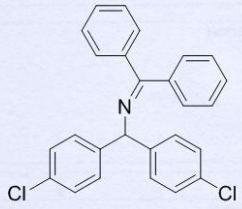
Integration Results for DAD1 A, Sig=254,4 Ref=off

RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.37	0.06	40.75	9.78	2.35	151	ND
4.09	0.07	395.40	87.64	22.85	100	ND
4.28	0.09	17.86	3.00	1.03	100	ND
4.70	0.08	178.80	36.58	10.33	100	ND
5.37	0.34	57.22	2.07	3.31	100	113
5.67	0.06	994.81	262.57	57.48	416	113
5.82	0.13	45.84	4.59	2.65	488	113

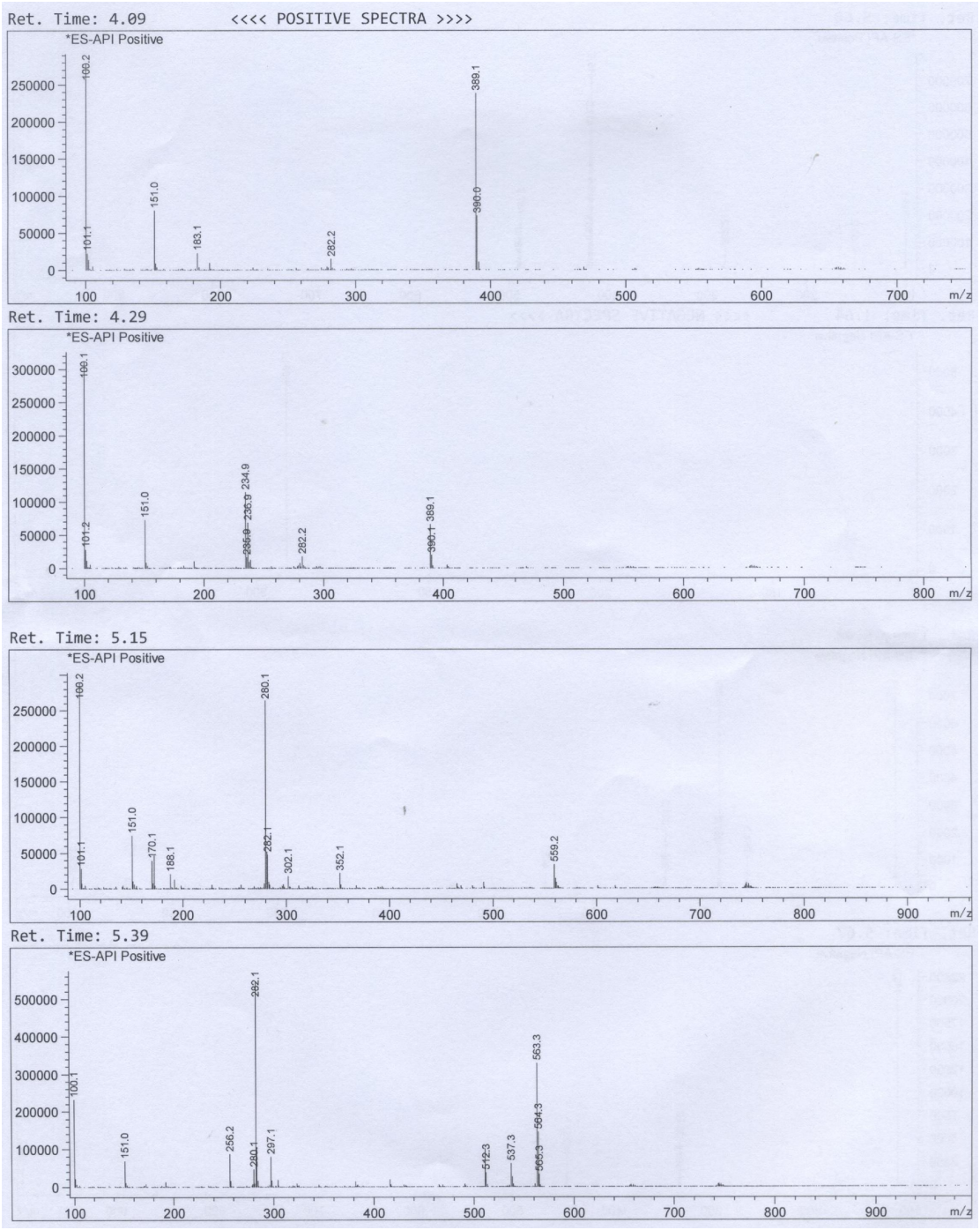
Integration Results for DAD1 B, Sig=214,4 Ref=off

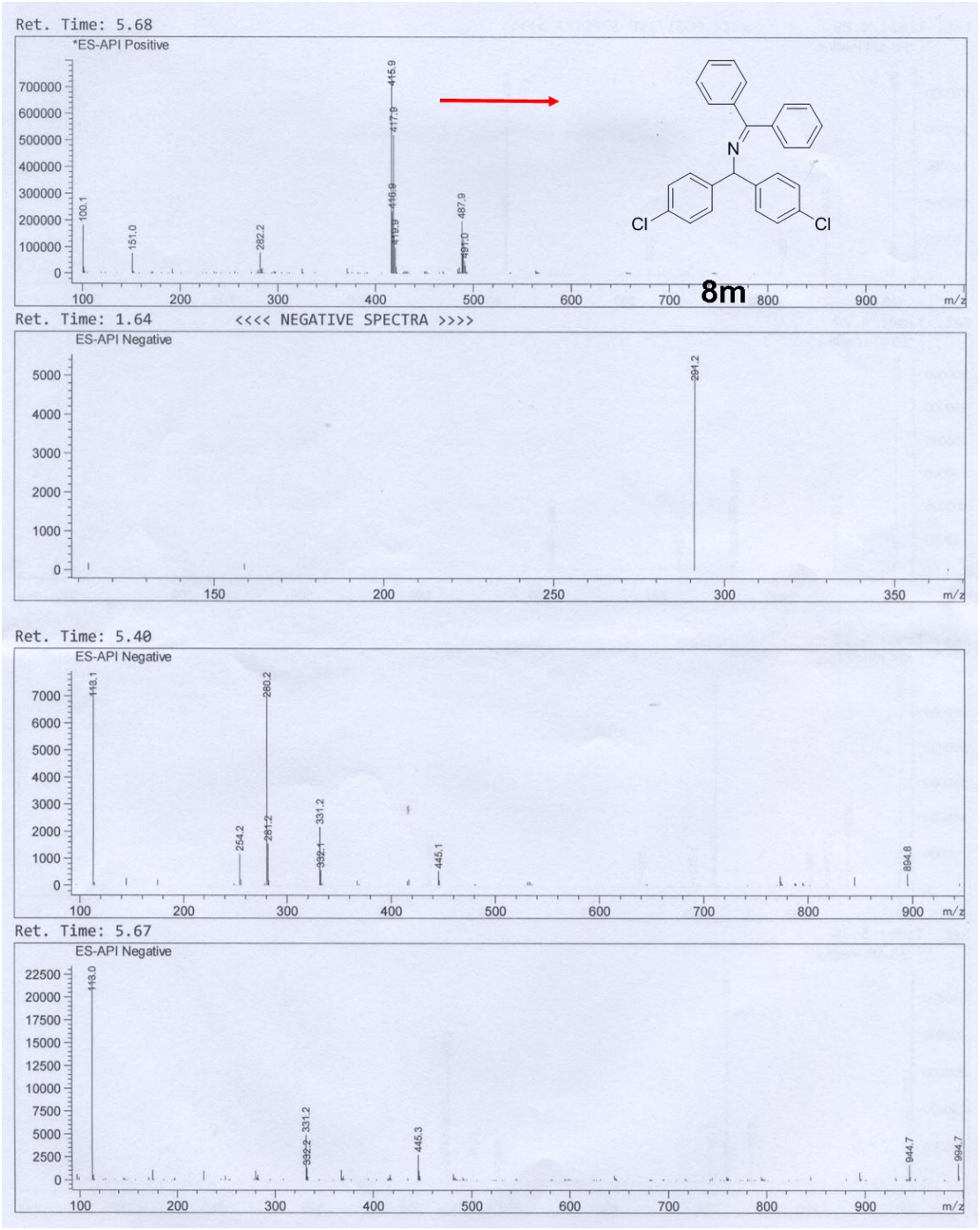
RetTim	Width	Area	Height	Area%	MS(+)	MS(-)
0.29	0.07	43.55	10.06	1.07	151	ND
0.36	0.05	51.83	13.01	1.27	151	ND
0.54	0.19	190.88	12.45	4.68	151	ND
0.98	0.43	428.12	12.30	10.50	151	ND
1.47	0.54	576.36	12.89	14.14	151	ND
2.80	0.12	9.25	1.06	0.23	100	ND
3.14	0.17	3.71	0.27	0.09	100	ND

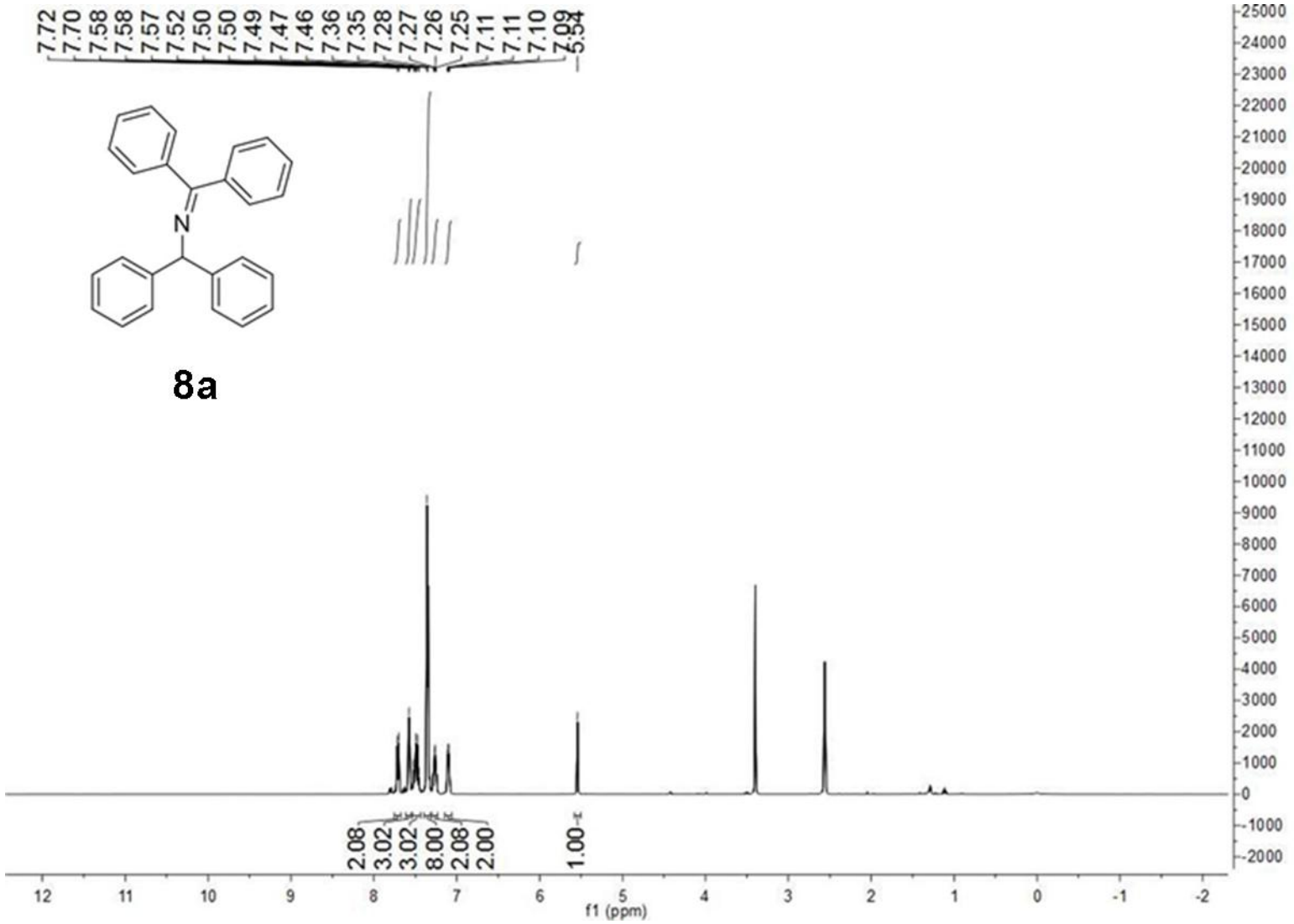
3.88	0.16	23.53	1.91	0.58	100	ND
4.09	0.08	256.96	54.19	6.30	100	ND
4.28	0.07	296.48	66.92	7.27	100	ND
4.55	0.08	1.83	0.31	0.04	100	ND
4.70	0.07	130.45	30.26	3.20	100	ND
5.15	0.11	14.66	1.85	0.36	100	113
5.37	0.08	40.14	7.57	0.99	282	280
5.67	0.06	1958.63	529.79	48.06	416	113
5.82	0.09	49.19	7.58	1.21	438	113

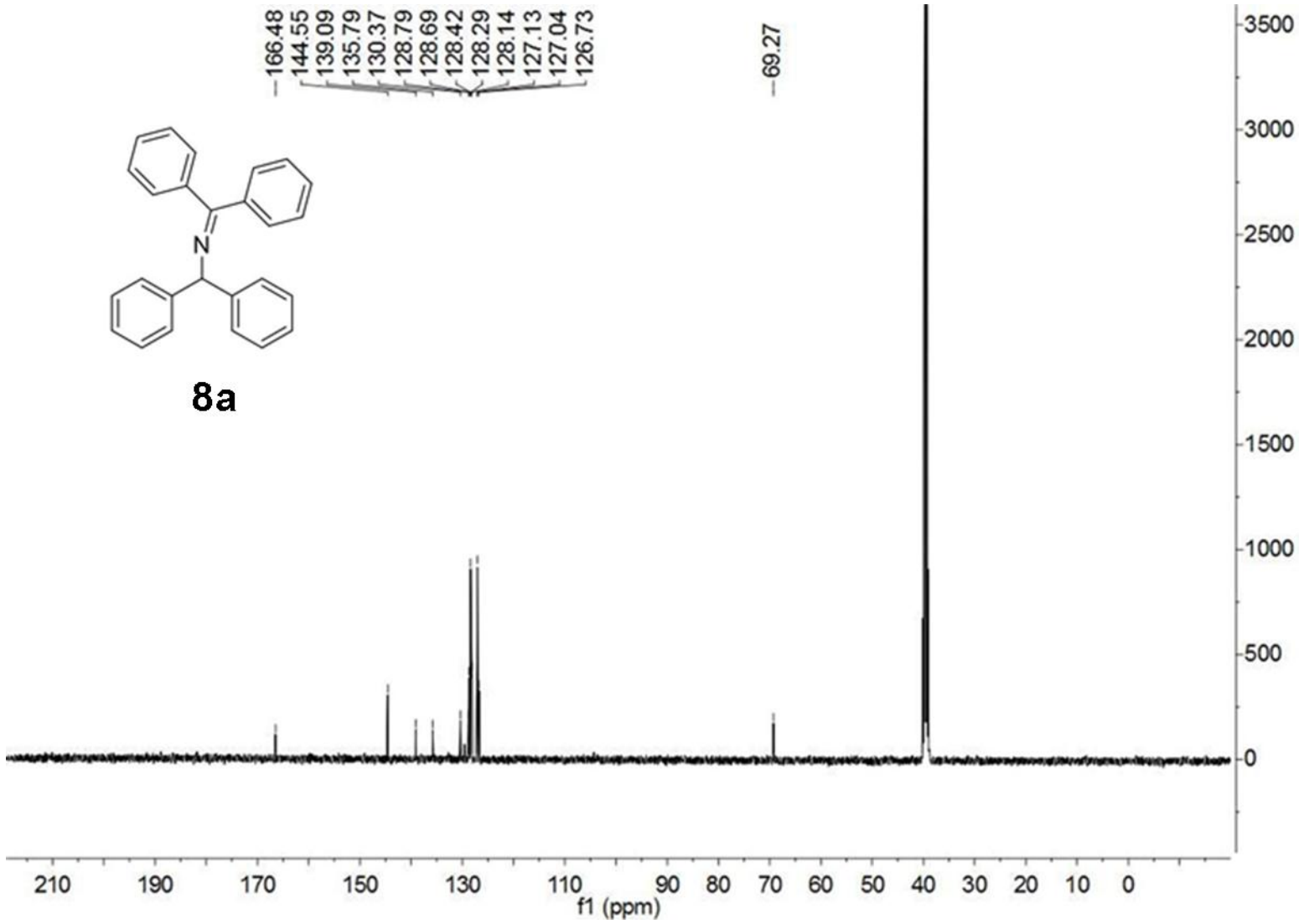


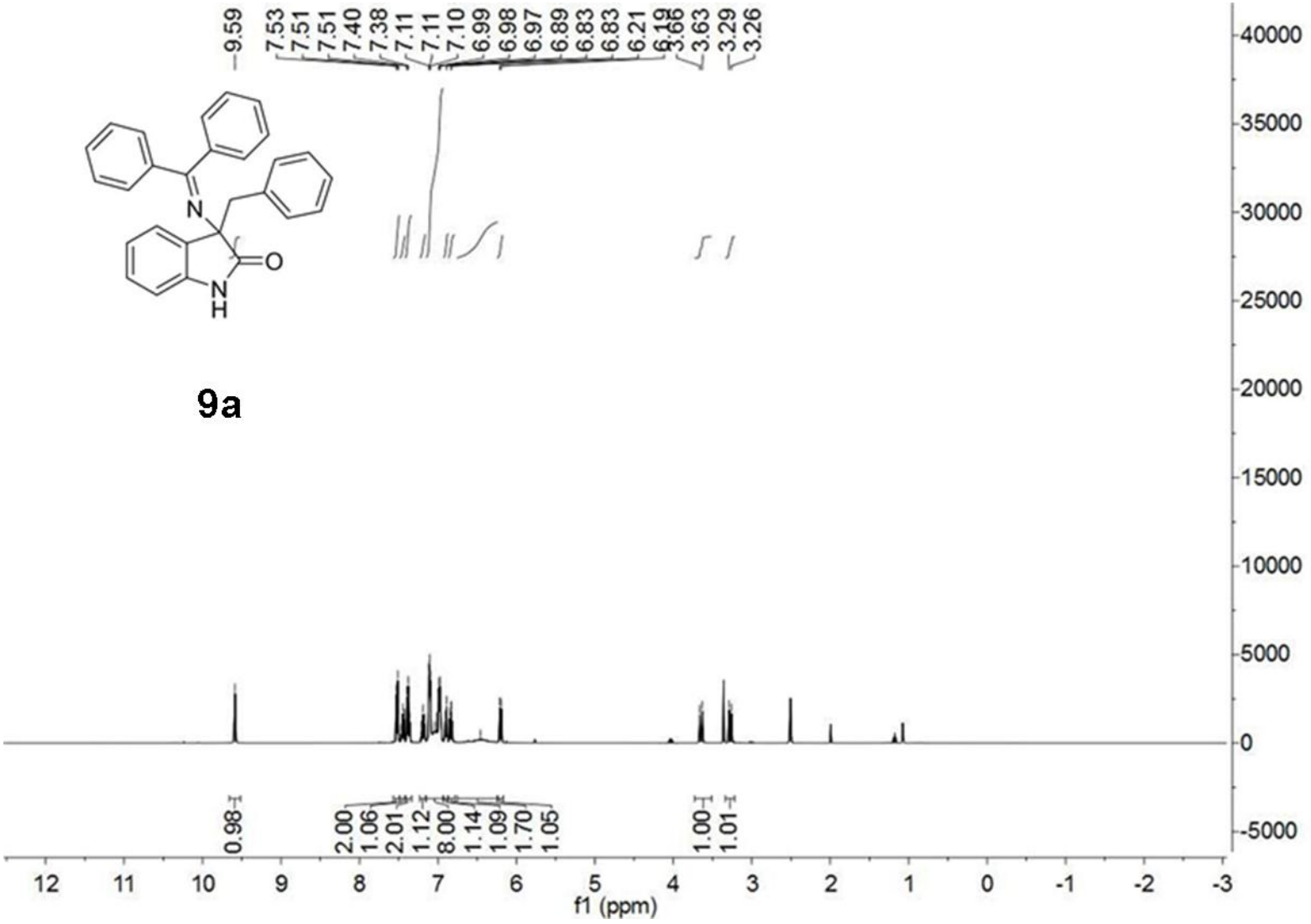
8m

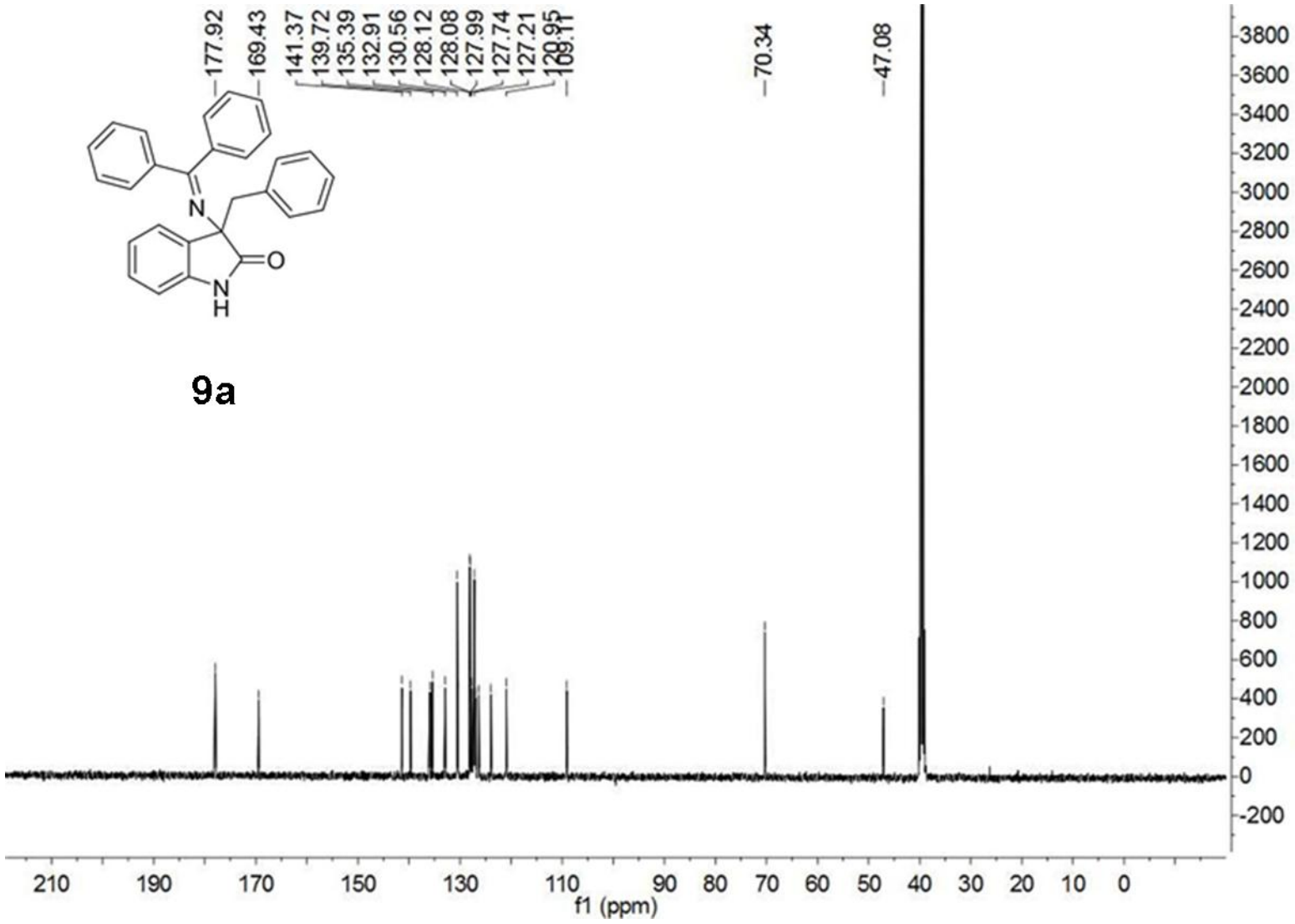

Ret. Time: 4.09
<<<< POSITIVE SPECTRA >>>>
*ES-API Positive
100.2
389.1
151.0
390.0
101.1
183.1
282.2
m/z
Ret. Time: 4.29
*ES-API Positive
100.1
234.9
151.0
101.2
282.2
389.1
m/z
Ret. Time: 5.15
*ES-API Positive
100.2
280.1
151.0
101.1
170.1
188.1
282.1
302.1
352.1
559.2
m/z
Ret. Time: 5.39
*ES-API Positive
282.1
563.3
100.1
151.0
256.2
280.1
297.1
564.3
537.3
m/z

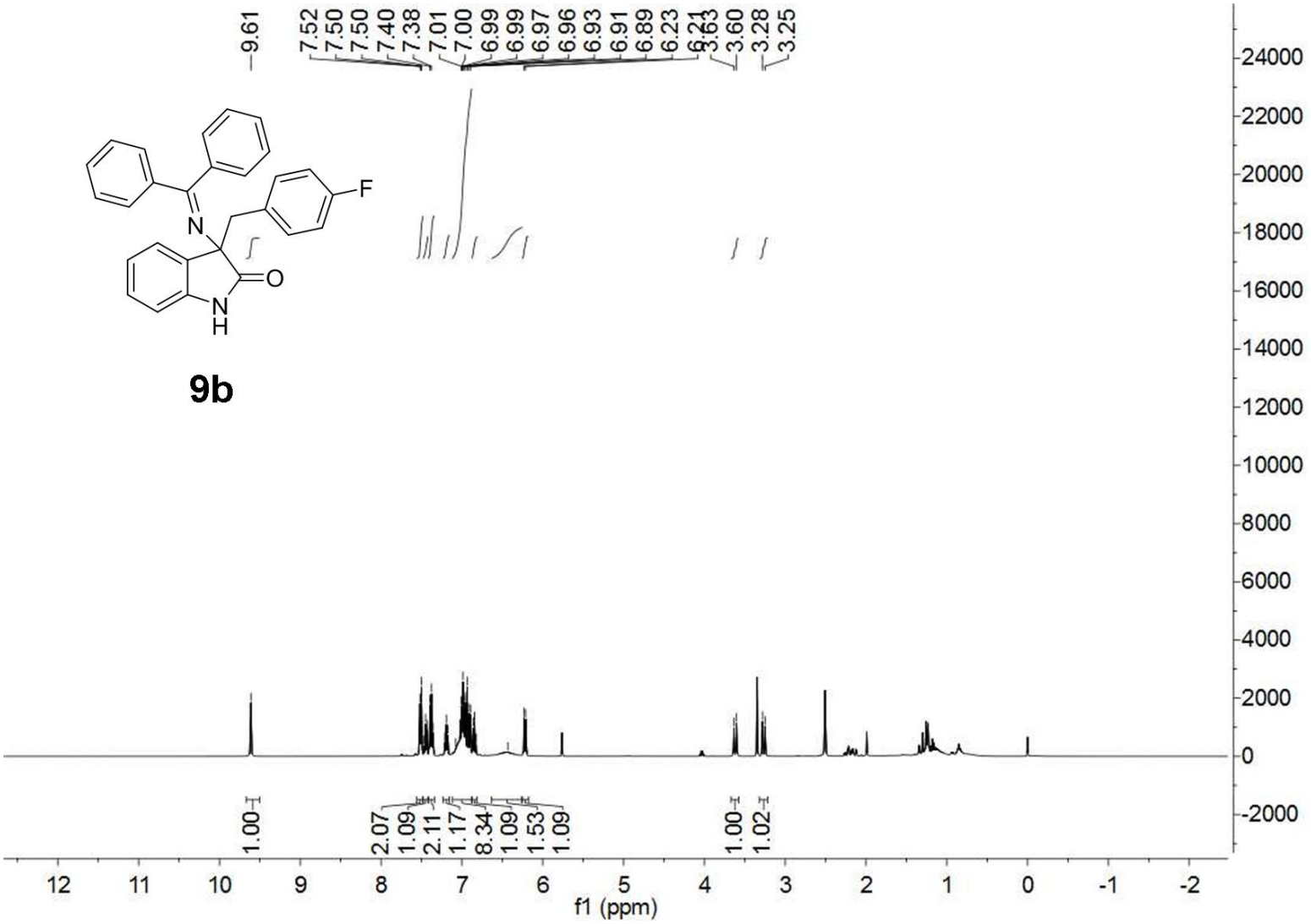

Ret. Time: 5.68
*ES-API Positive
100.1
151.0
282.2
415.9
417.9
416.9
487.9
491.0
N
Cl
Cl
8m
Ret. Time: 1.64
<<<< NEGATIVE SPECTRA >>>>
ES-API Negative
291.2
Ret. Time: 5.40
ES-API Negative
113.1
280.2
254.2
281.2
331.2
332.1
445.1
894.8
Ret. Time: 5.67
ES-API Negative
113.0
331.2
332.2
445.3
944.7
994.7
m/z


7.72
7.70
7.58
7.58
7.57
7.52
7.50
7.50
7.49
7.47
7.46
7.36
7.35
7.28
7.27
7.26
7.25
7.11
7.11
7.10
7.09
5.54
N
8a
2.08
3.02
3.02
8.00
2.08
2.00
1.00
f1 (ppm)

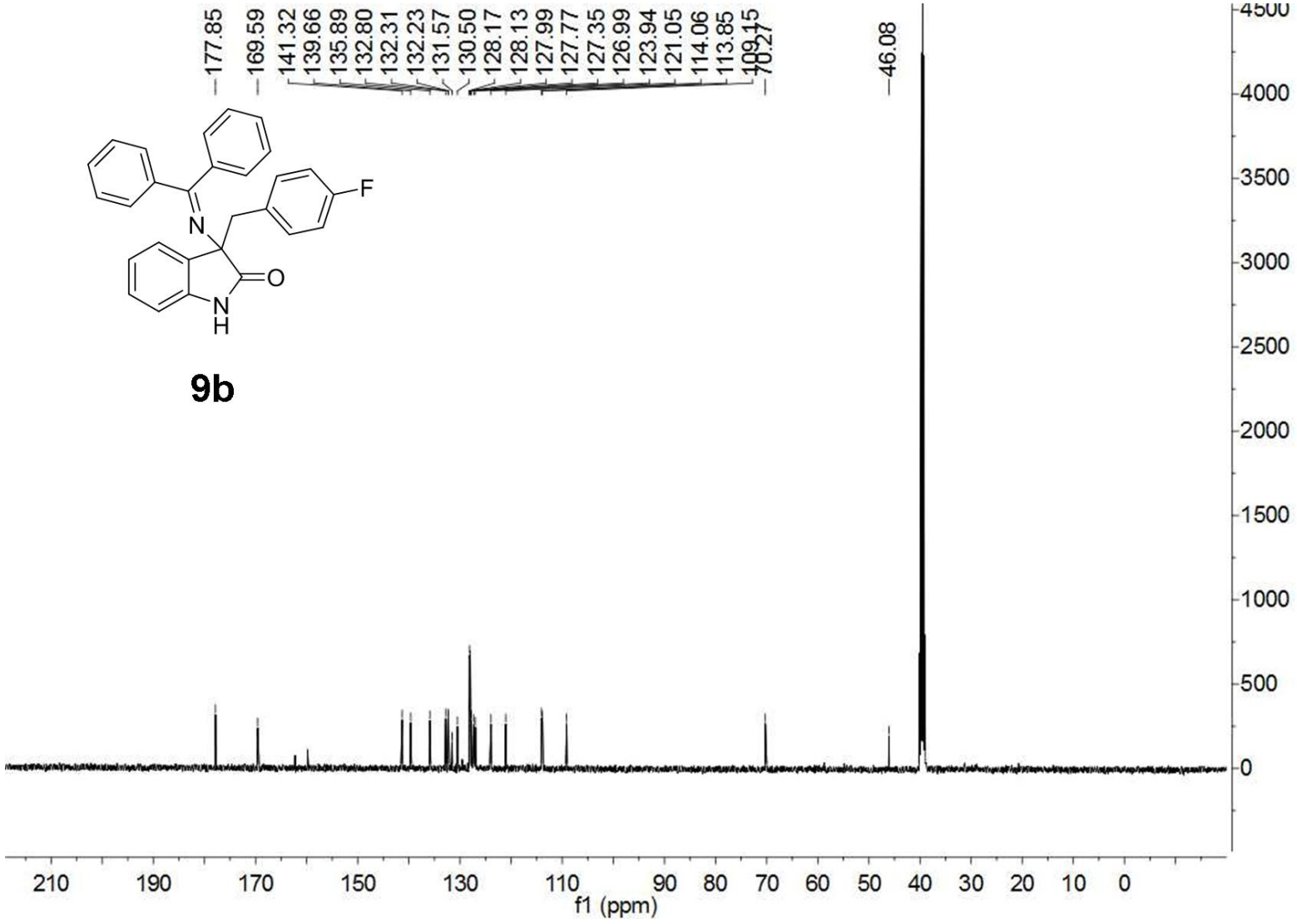

166.48
144.55
139.09
135.79
130.37
128.79
128.69
128.42
128.29
128.14
127.13
127.04
126.73
69.27
N
8a
3500
3000
2500
2000
1500
1000
500
0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

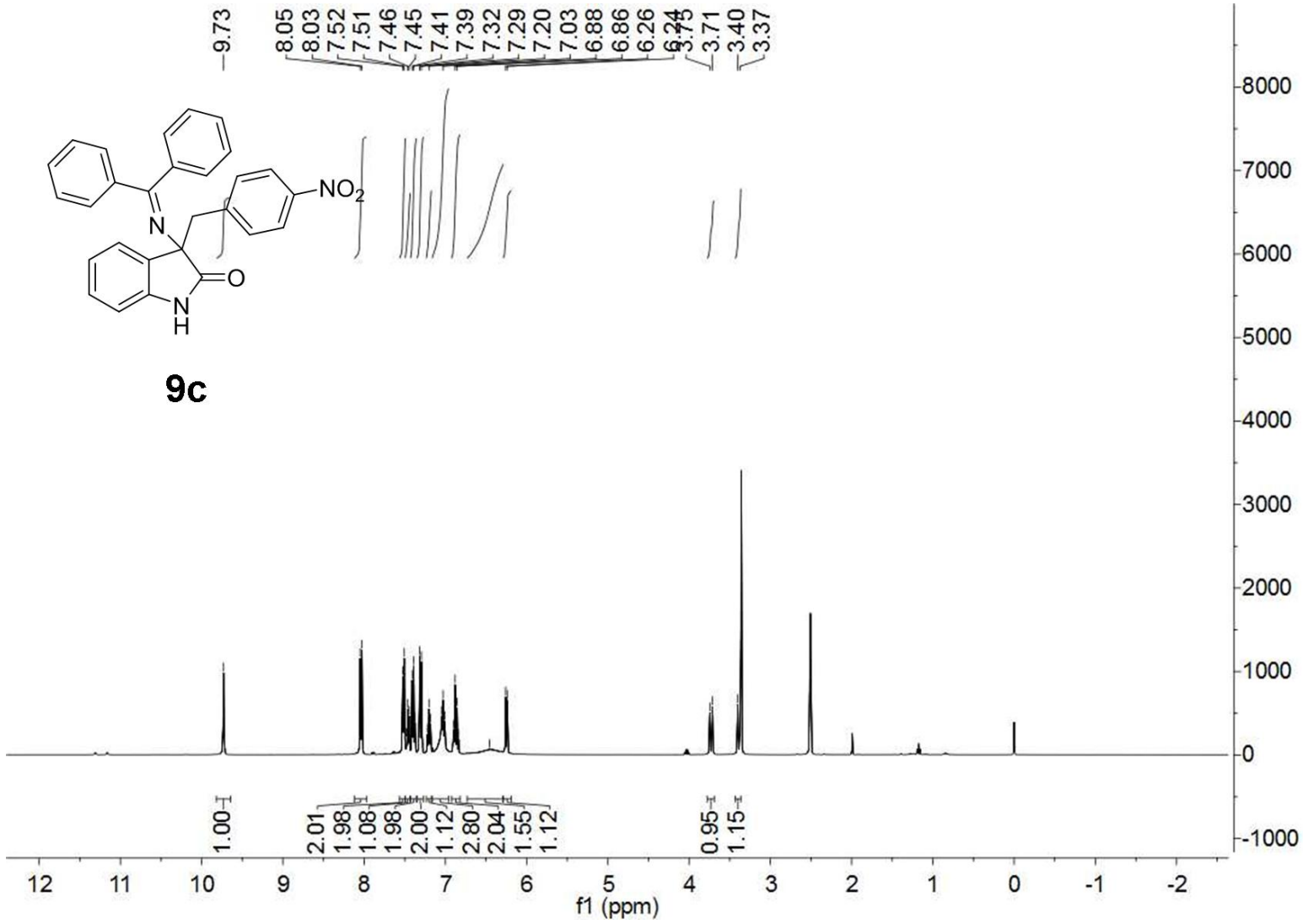

9.59
7.53
7.51
7.51
7.40
7.38
7.11
7.11
7.10
6.99
6.98
6.97
6.89
6.83
6.83
6.21
6.19
6.16
3.63
3.29
3.26
N
O
N
H
9a
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
0.98
2.00
1.06
2.01
1.12
8.00
1.14
1.09
1.70
1.05
1.00
1.01
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

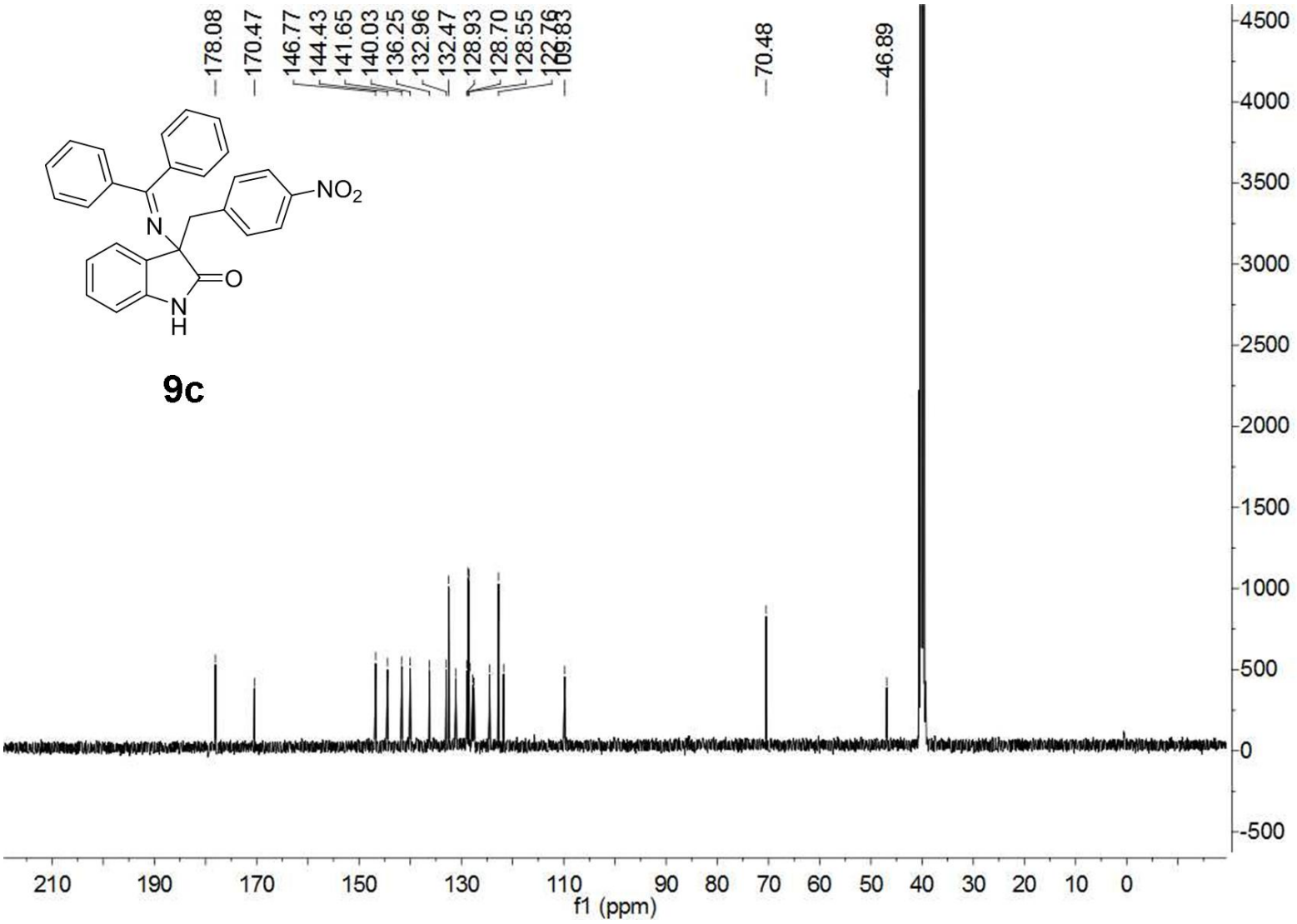

177.92
169.43
141.37
139.72
135.39
132.91
130.56
128.12
128.08
127.99
127.74
127.21
120.95
109.11
70.34
47.08
N
O
N
H
9a
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.61
7.52
7.50
7.50
7.40
7.38
7.01
7.00
6.99
6.99
6.97
6.96
6.93
6.91
6.89
6.23
6.21
3.63
3.60
3.28
3.25
F
N
O
N
H
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
1.00
2.07
1.09
2.11
1.17
8.34
1.09
1.53
1.09
1.00
1.02
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

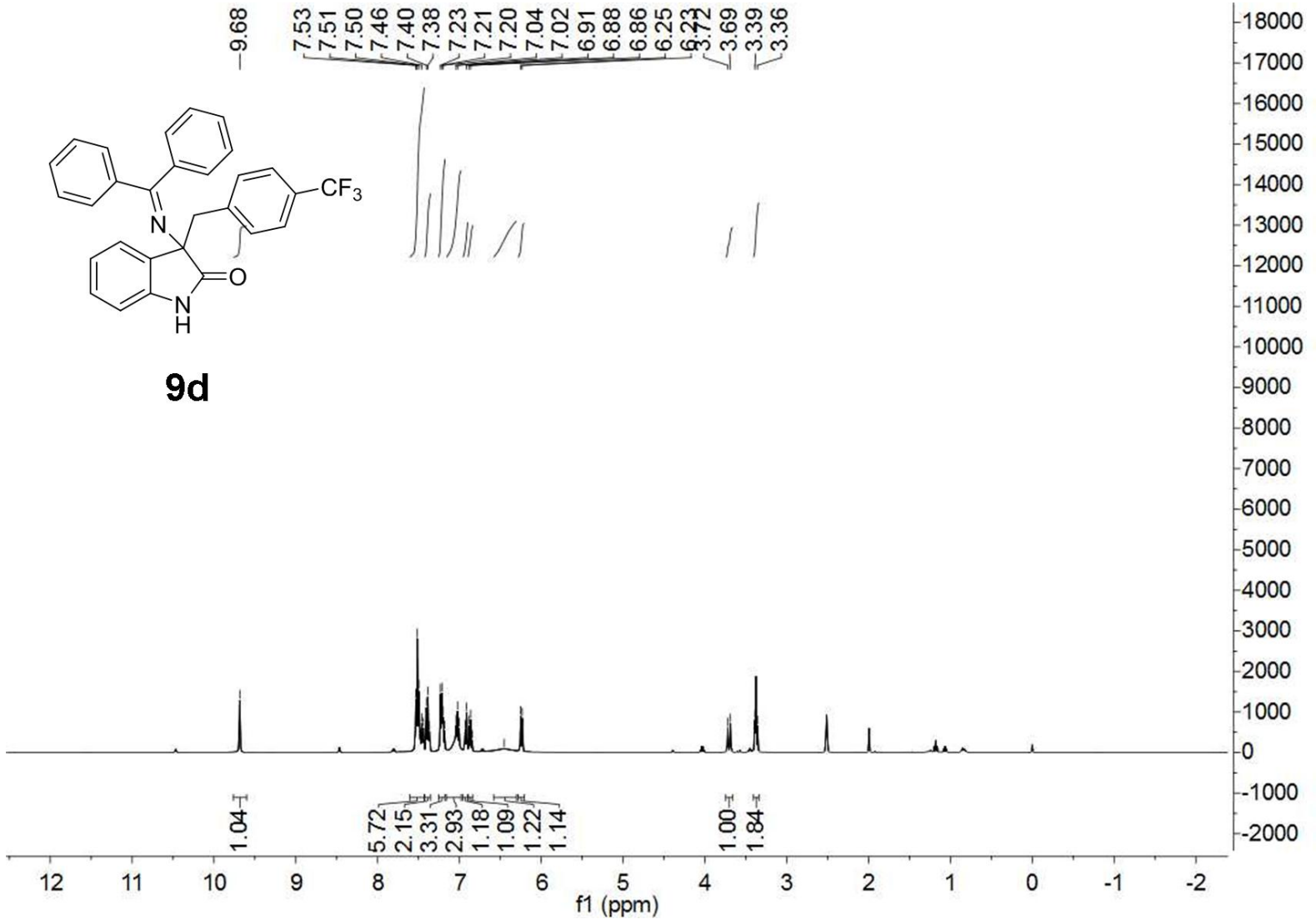
9b

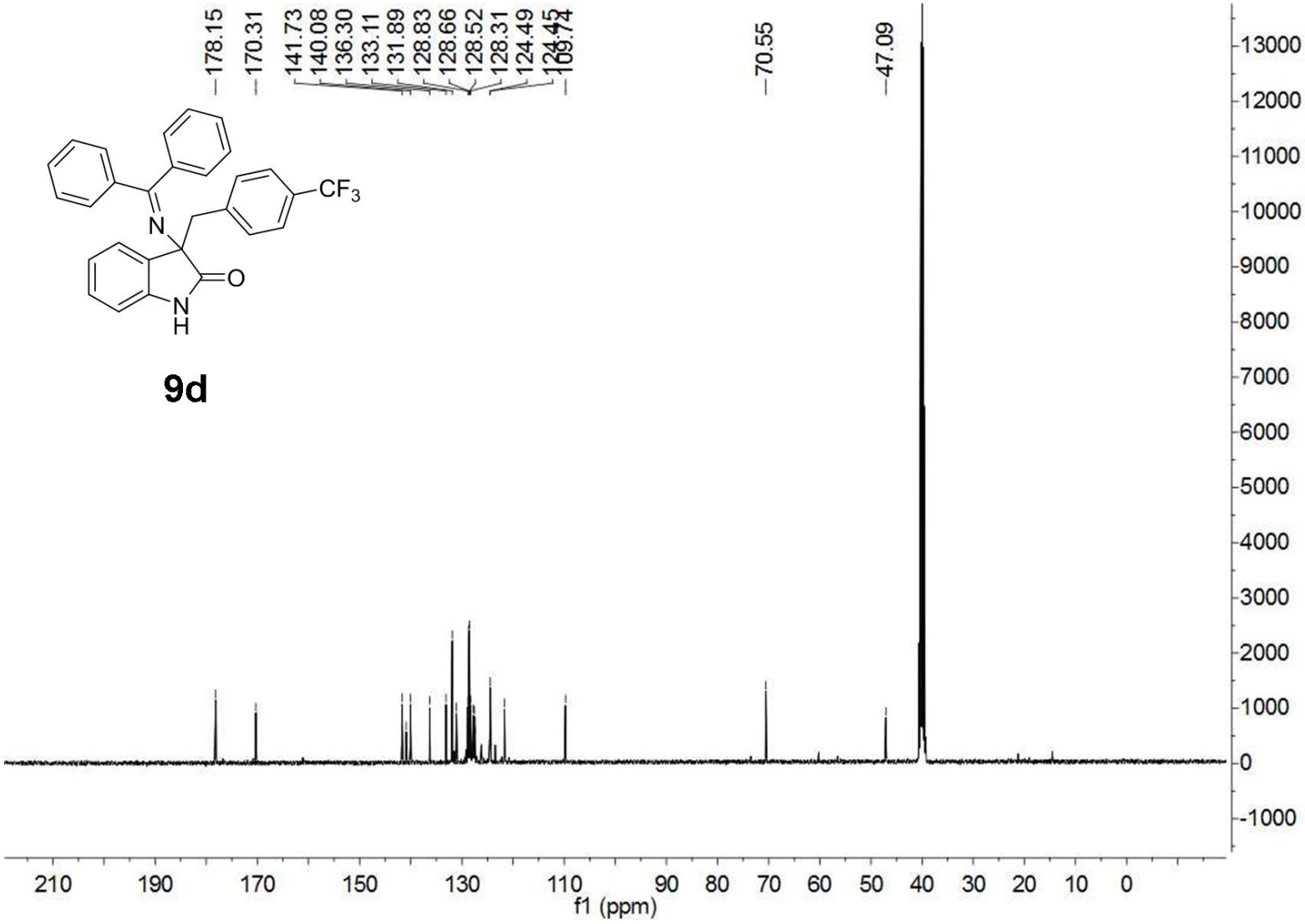

177.85
169.59
141.32
139.66
135.89
132.80
132.31
132.23
131.57
130.50
128.17
128.13
127.99
127.77
127.35
126.99
123.94
121.05
114.06
113.85
109.15
70.27
46.08
F
N
O
N
H
9b
4500
4000
3500
3000
2500
2000
1500
1000
500
0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.73
8.05
8.03
7.52
7.51
7.46
7.45
7.41
7.39
7.32
7.29
7.20
7.03
6.88
6.86
6.26
6.24
3.75
3.71
3.40
3.37
NO2
N
O
N
H
9c
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.00
2.01
1.98
1.08
1.98
2.00
1.12
2.80
2.04
1.55
1.12
0.95
1.15
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

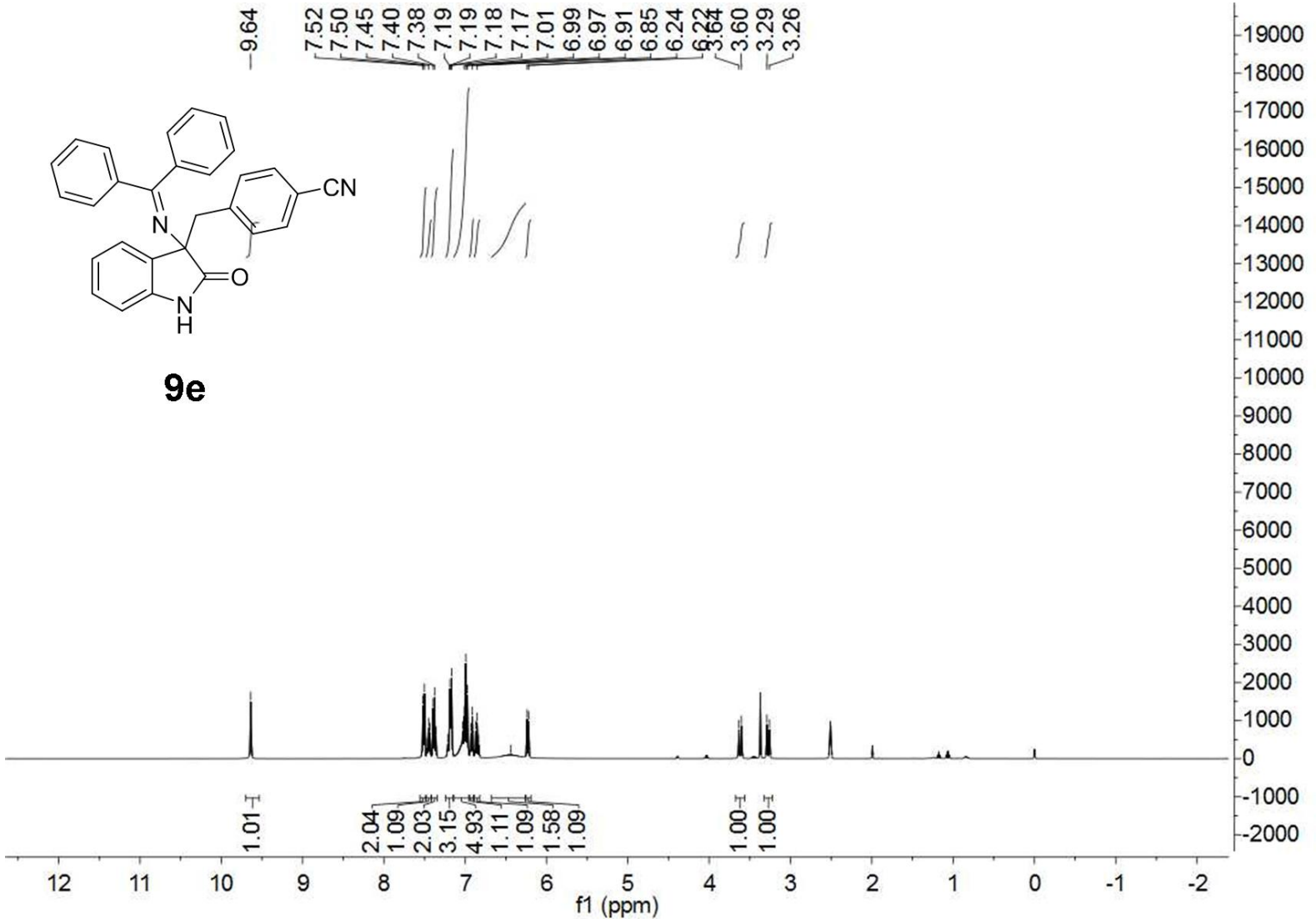

178.08
170.47
146.77
144.43
141.65
140.03
136.25
132.96
132.47
128.93
128.70
128.55
70.48
46.89
NO2
N
O
N
H
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9c


9.68
7.53
7.51
7.50
7.46
7.40
7.38
7.23
7.21
7.20
7.04
7.02
6.91
6.88
6.86
6.25
6.23
3.72
3.69
3.39
3.36
CF3
N
O
N
H
9d
1.04
5.72
2.15
3.31
2.93
1.18
1.09
1.22
1.14
1.00
1.84
f1 (ppm)


178.15
170.31
141.73
140.08
136.30
133.11
131.89
128.83
128.66
128.52
128.31
124.49
124.45
109.74
70.55
47.09
CF3
N
O
N
H
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9d


9.64
7.52
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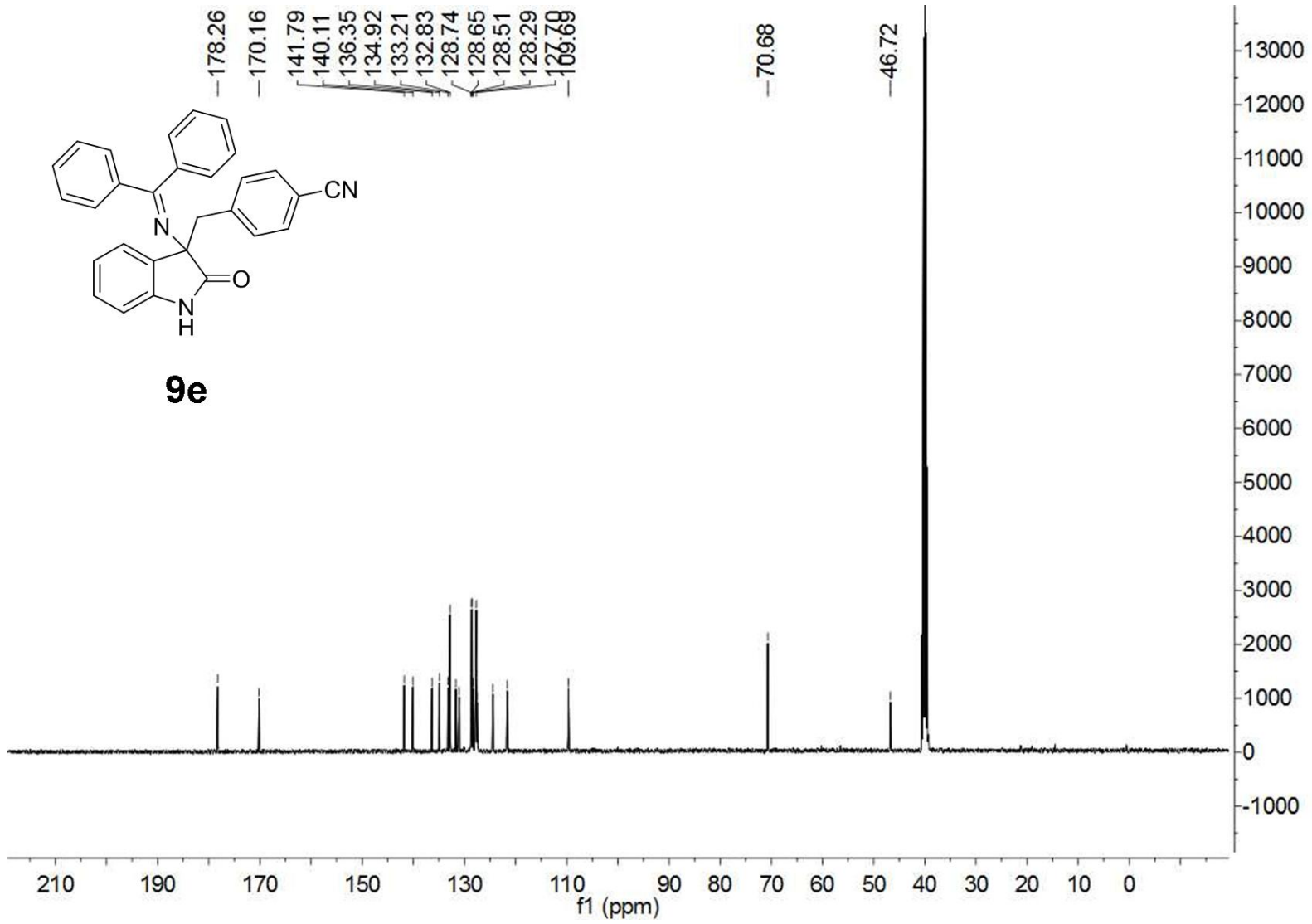
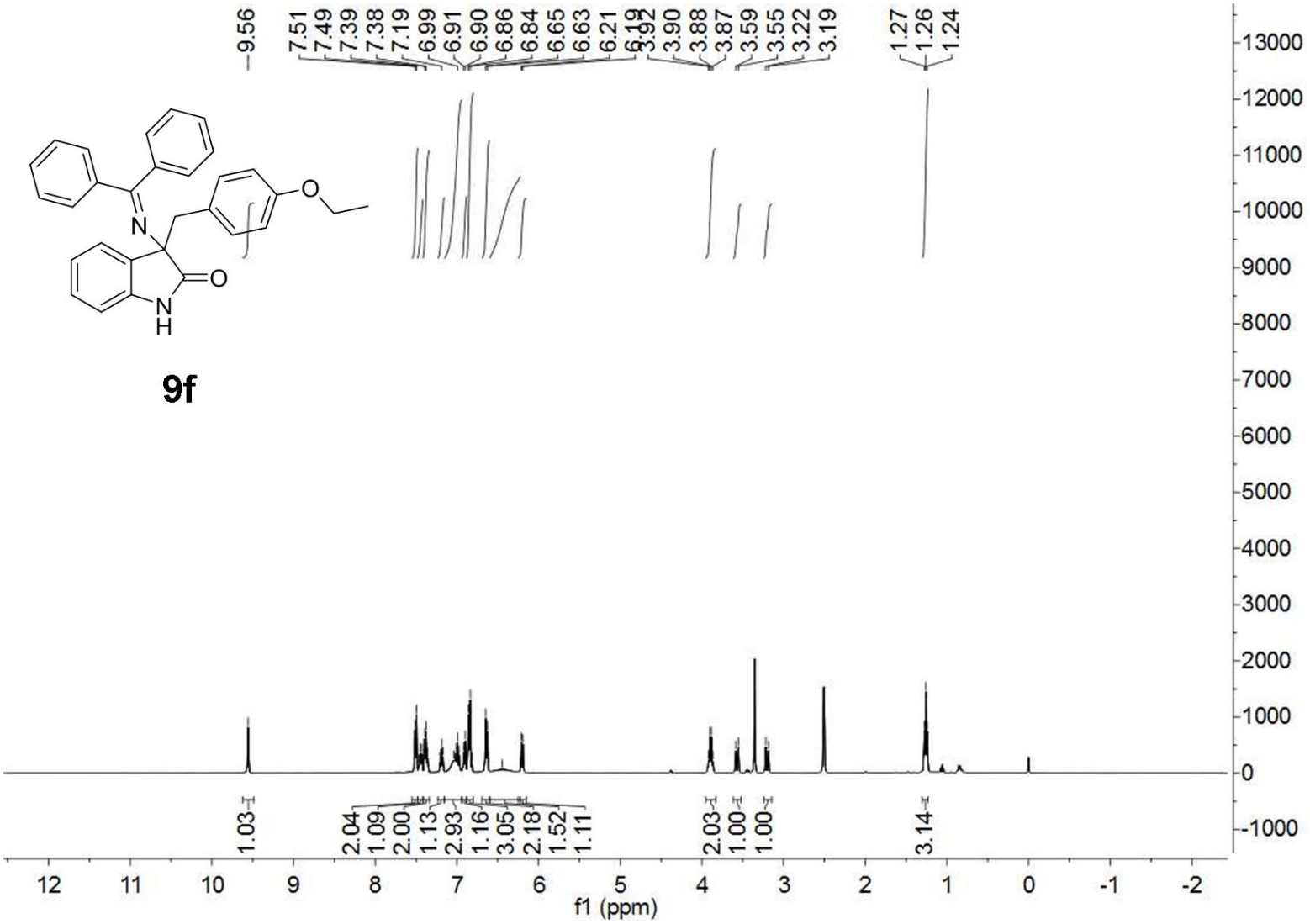
7.45
7.40
7.38
7.19
7.19
7.18
7.17
7.01
6.99
6.97
6.91
6.85
6.24
6.22
3.64
3.60
3.29
3.26
CN
N
O
N
H
1.01
2.04
1.09
2.03
3.15
4.93
1.11
1.09
1.58
1.09
1.00
1.00
f1 (ppm)

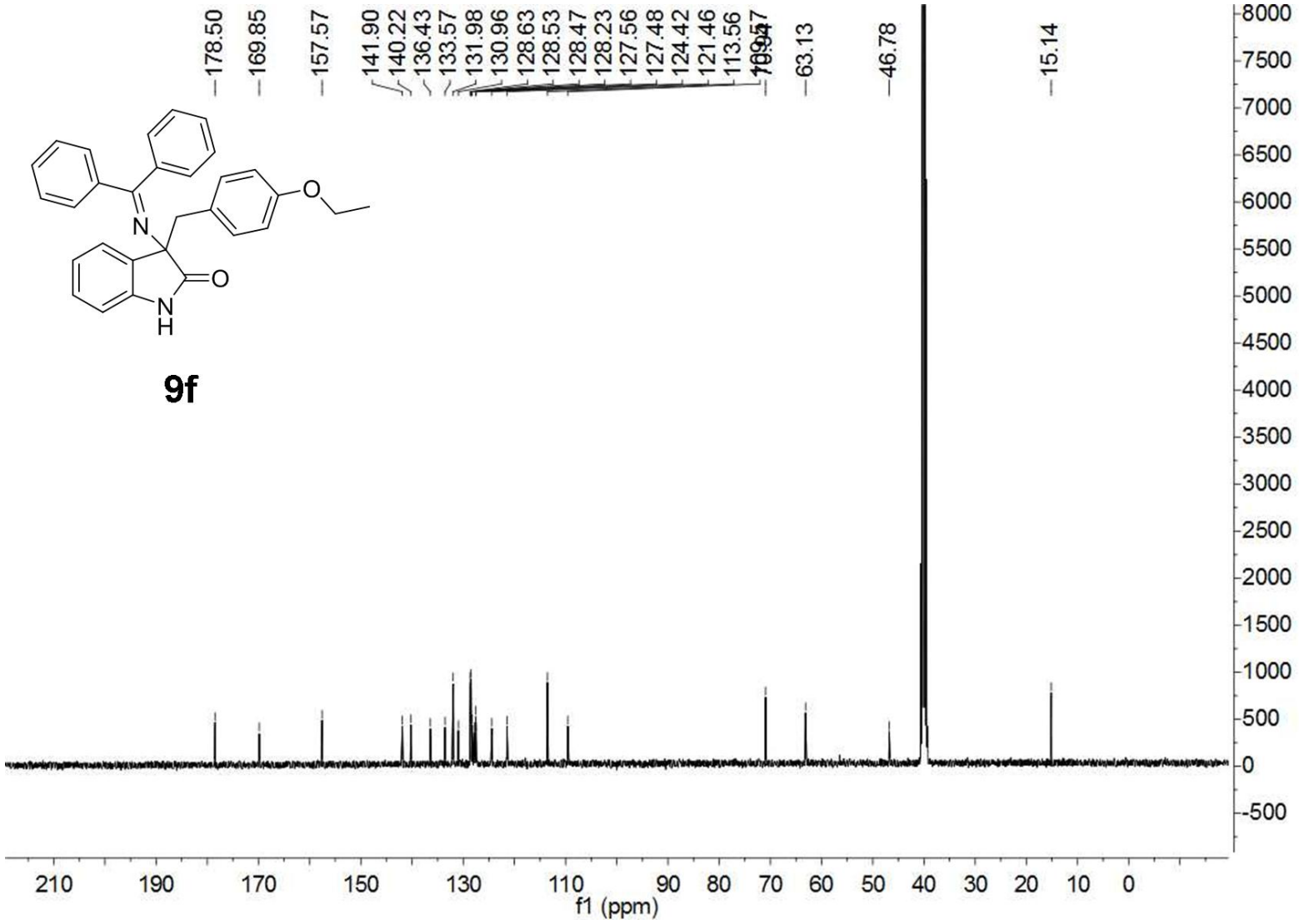
9e


178.26
170.16
141.79
140.11
136.35
134.92
133.21
132.83
128.74
128.65
128.51
128.29
70.68
46.72
CN
N
O
N
H
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9e


9.56
7.51
7.49
7.39
7.38
7.19
6.99
6.91
6.90
6.86
6.84
6.65
6.63
6.21
6.19
3.92
3.90
3.88
3.87
3.59
3.55
3.22
3.19
1.27
1.26
1.24
O
N
O
N
H
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.03
2.04
1.09
2.00
1.13
2.93
1.16
3.05
2.18
1.52
1.11
2.03
1.00
1.00
3.14
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

9f


178.50
169.85
157.57
141.90
140.22
136.43
133.57
131.98
130.96
128.63
128.53
128.47
128.23
127.56
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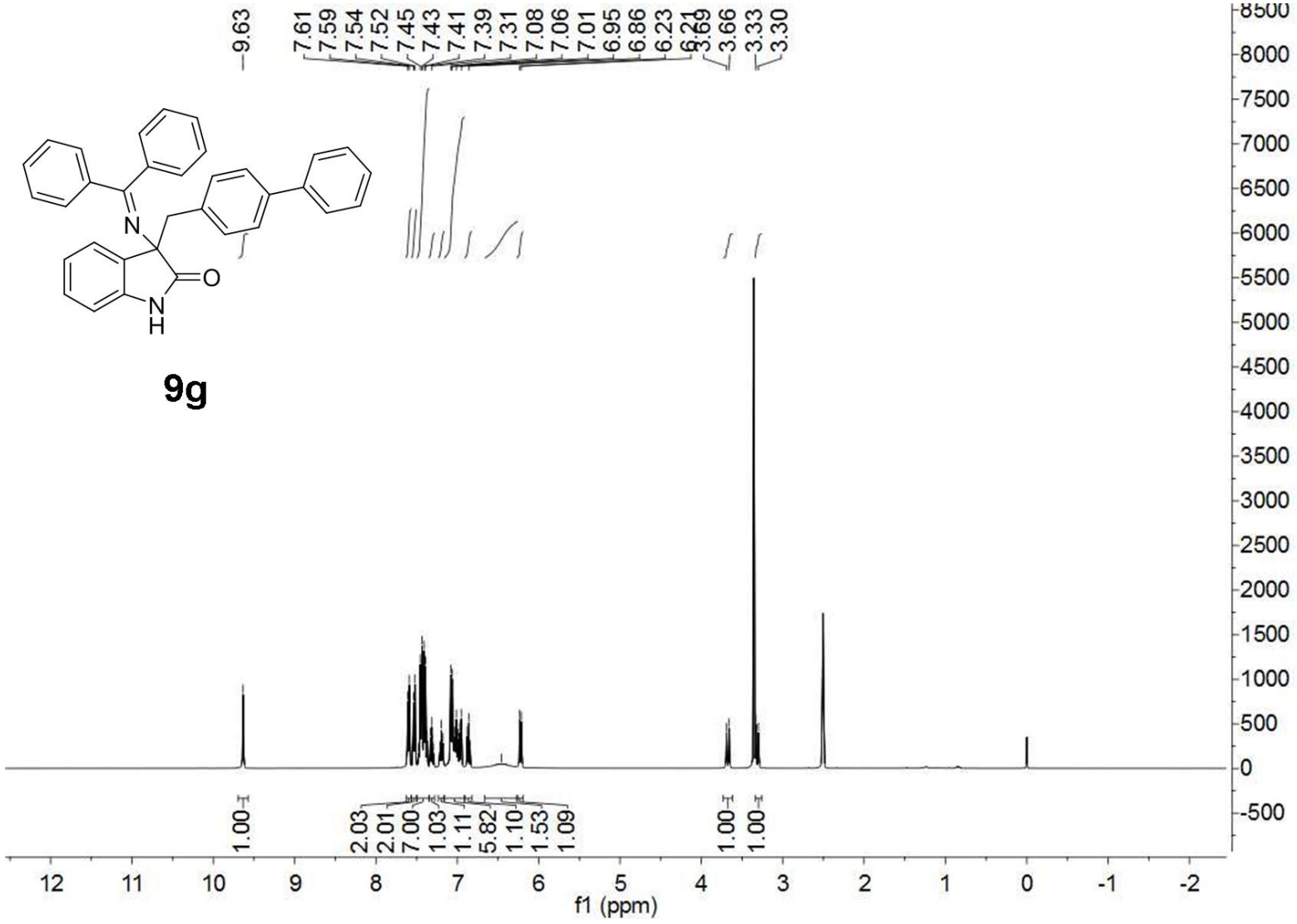
N
H
f1 (ppm)

9f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7.31
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7.06
7.01
6.95
6.86
6.23
6.21
3.69
3.66
3.33
3.30
N
O
N
H
1.00
2.03
2.01
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1.10
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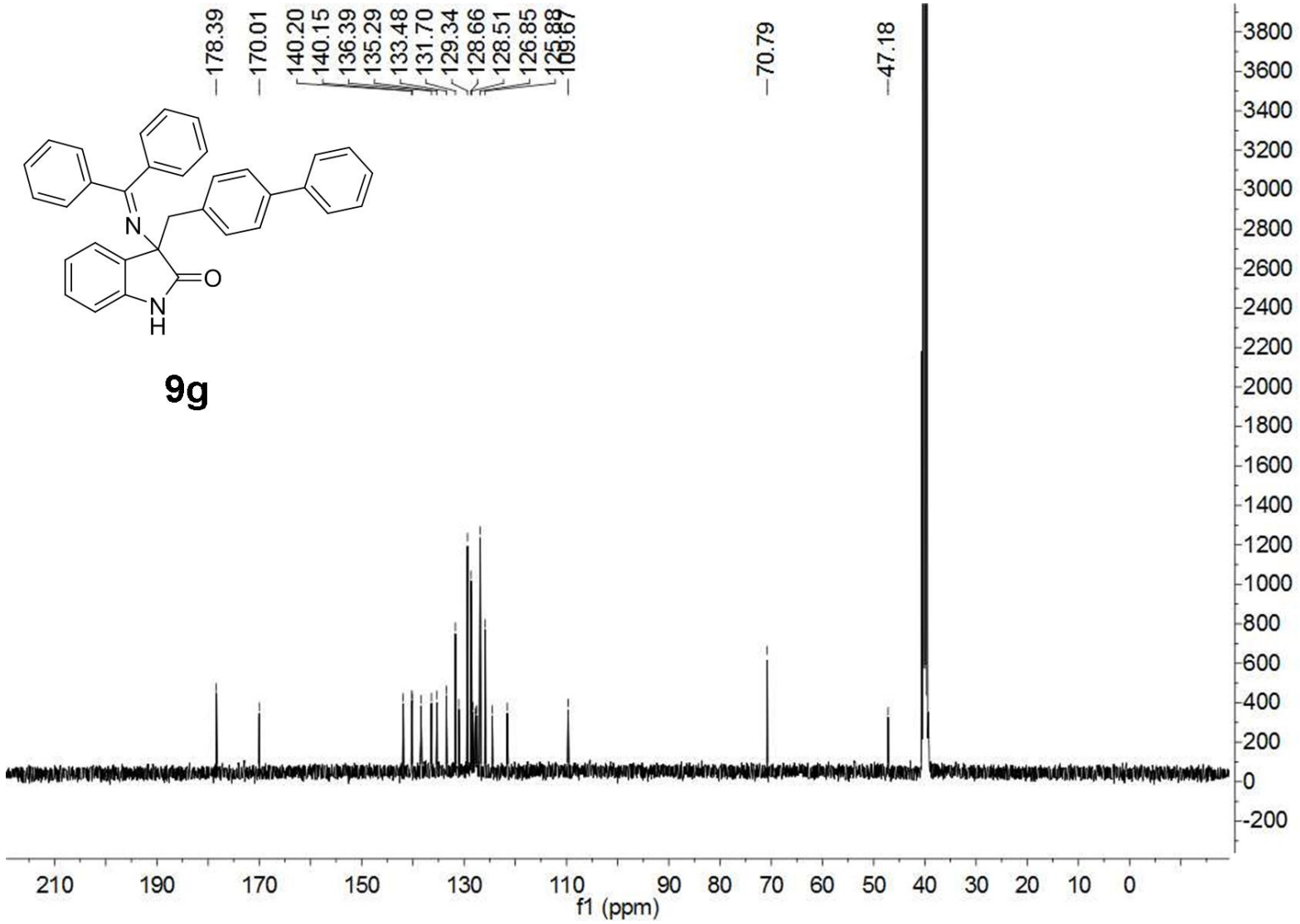
1.00
f1 (ppm)

9g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135.29
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128.51
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N
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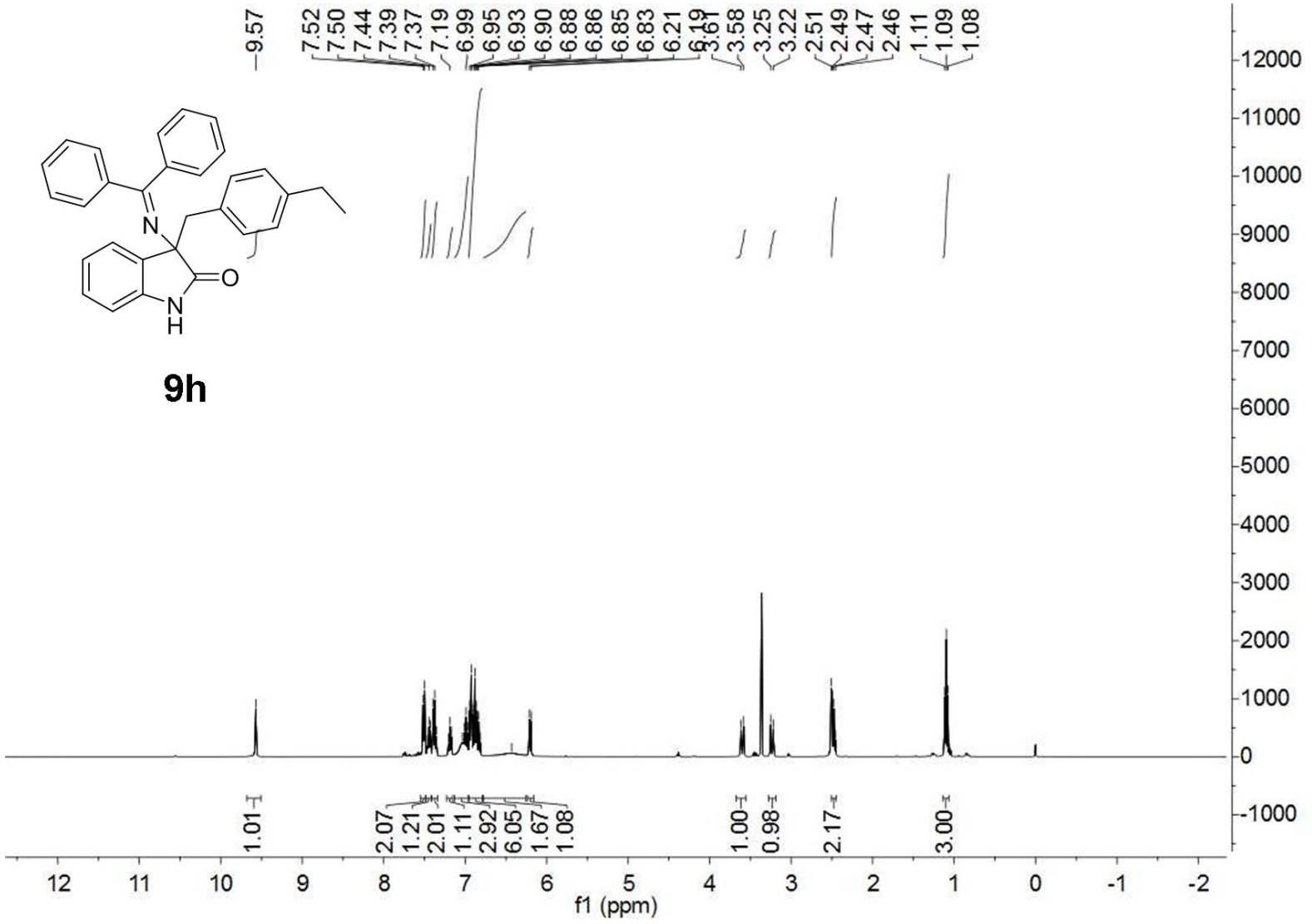
N
H
f1 (ppm)

9g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6.88
6.86
6.85
6.83
6.21
6.19
3.61
3.58
3.25
3.22
2.51
2.49
2.47
2.46
1.11
1.09
1.08
N
O
N
H
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.01
2.07
1.21
2.01
1.11
2.92
6.05
1.67
1.08
1.00
0.98
2.17
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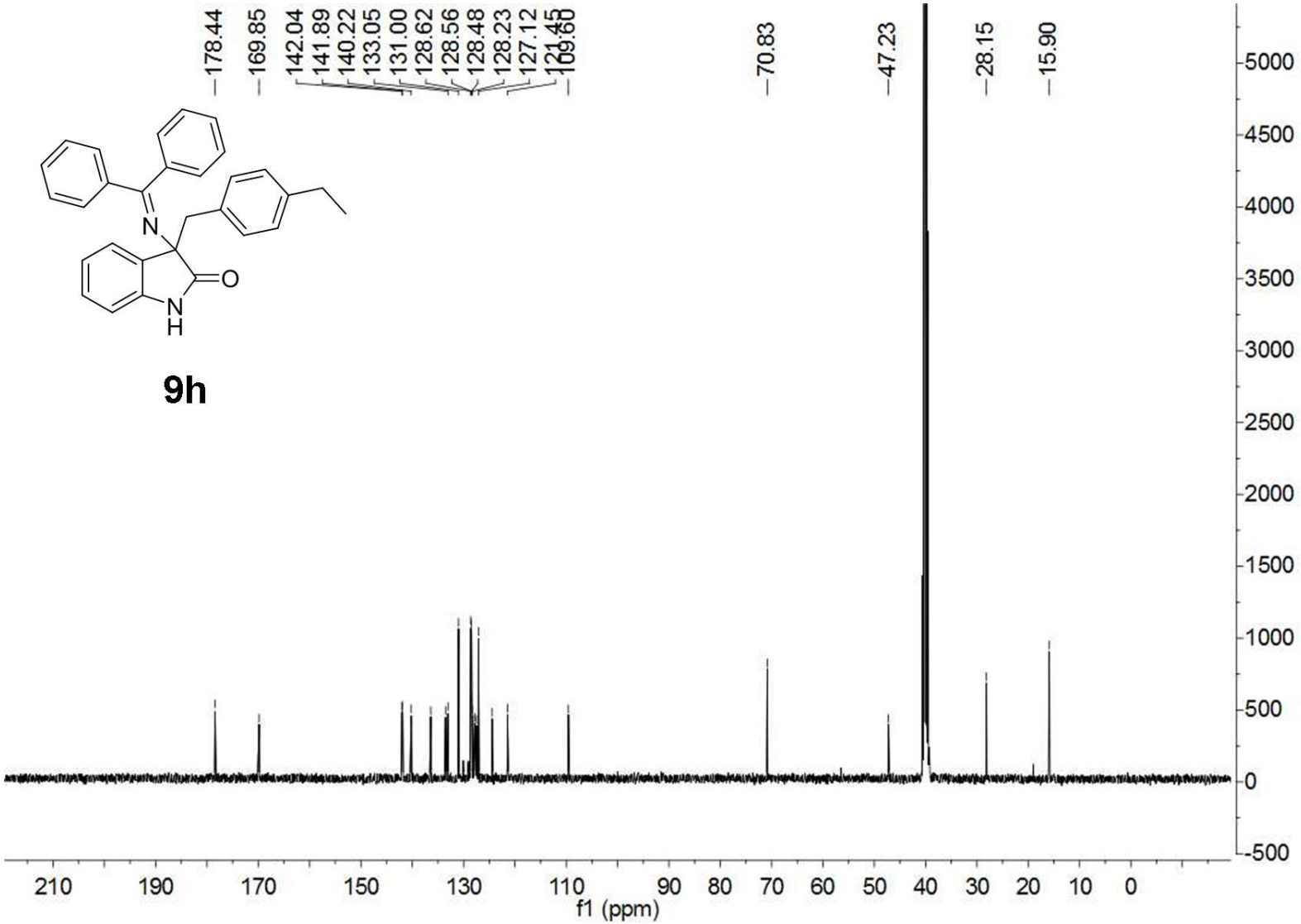
-2
f1 (ppm)

9h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127.12
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109.60
70.83
47.23
28.15
15.90
N
O
N
H
9h
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
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90
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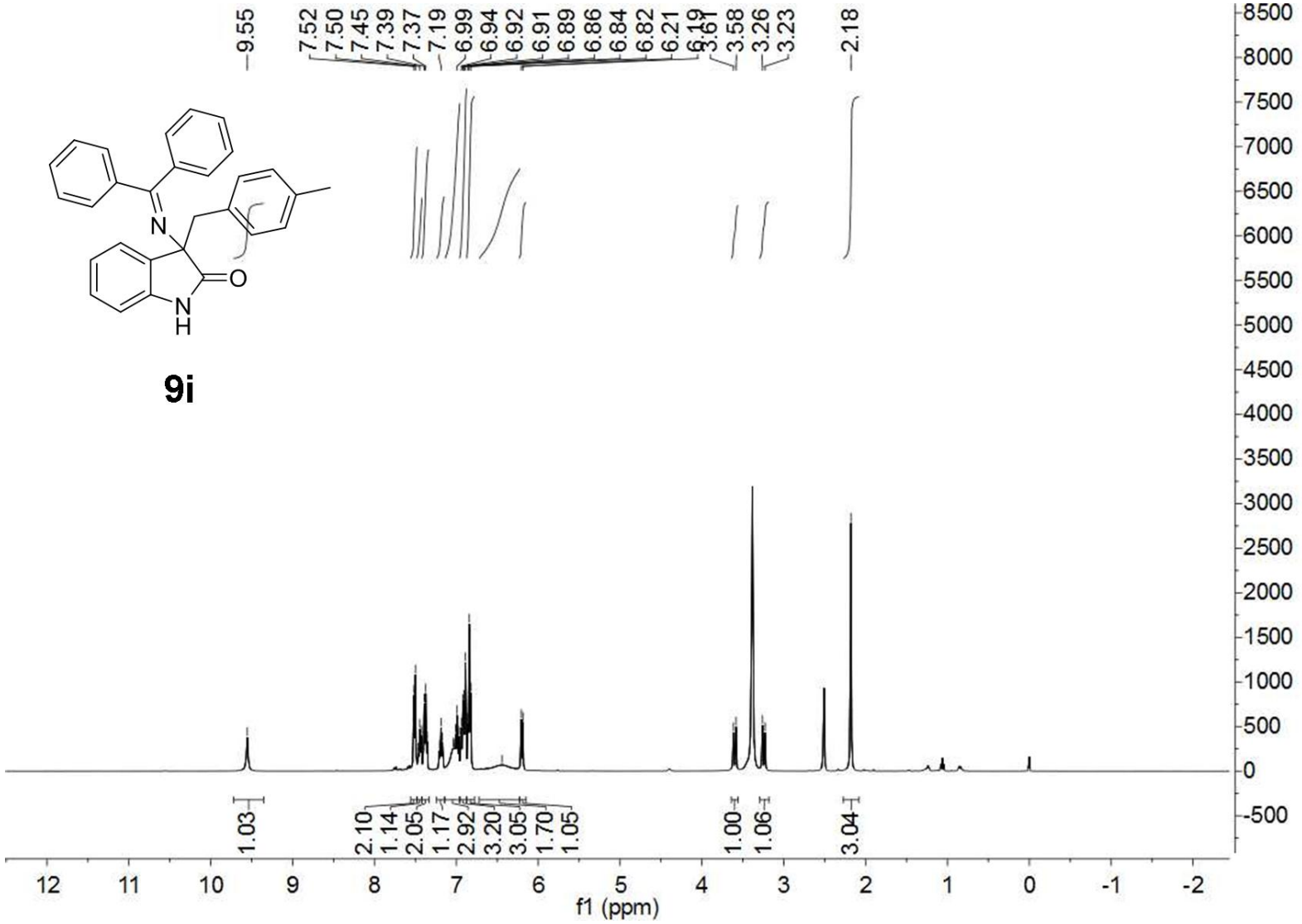
f1 (ppm)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6.89
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6.84
6.82
6.21
6.19
3.61
3.58
3.26
3.23
2.18
N
O
N
H
1.03
2.10
1.14
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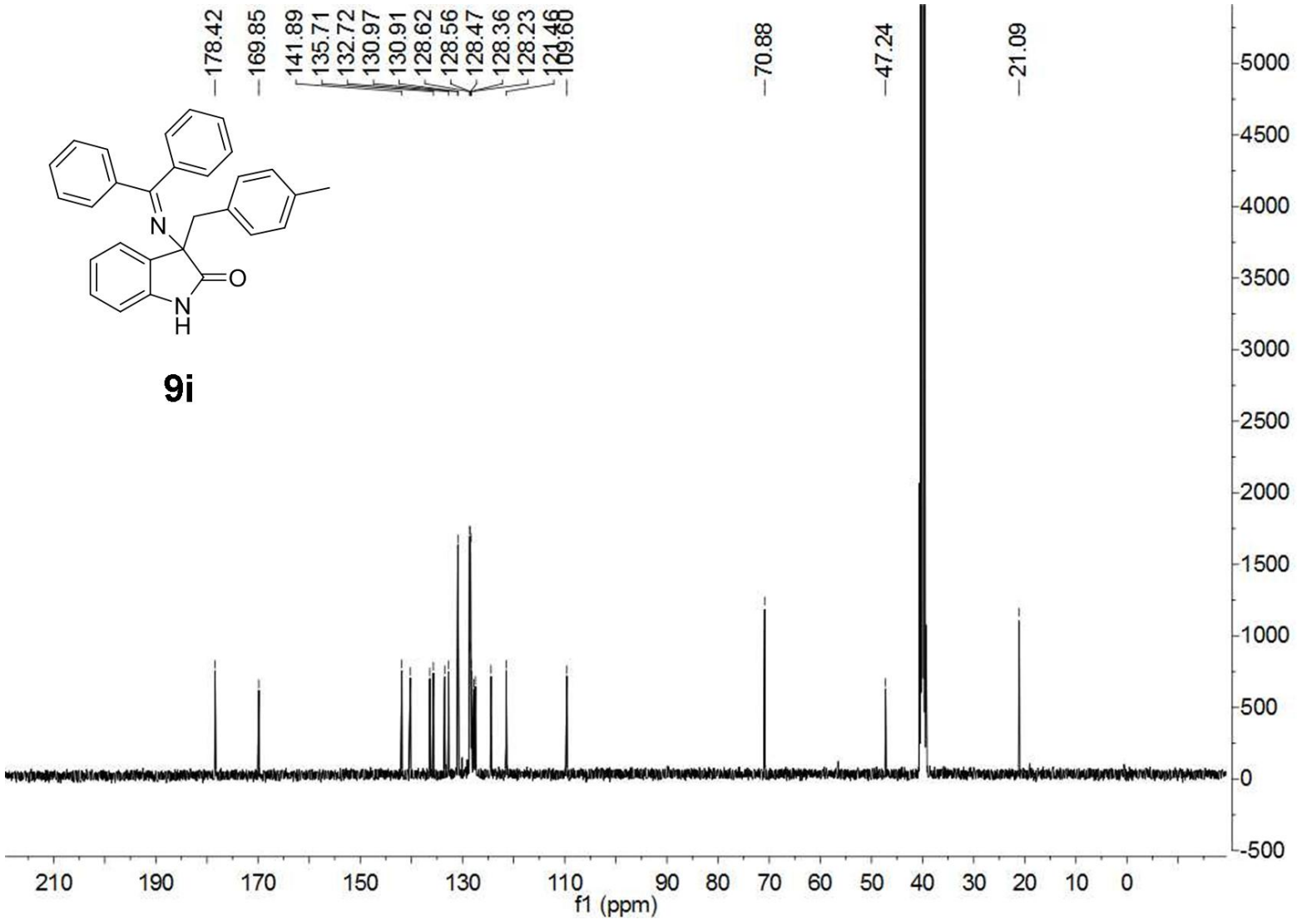
1.06
3.04
f1 (ppm)

9i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128.36
128.23
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109.60
70.88
47.24
21.09
N
O
N
H
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4500
4000
3500
3000
2500
2000
1500
1000
500
0
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30
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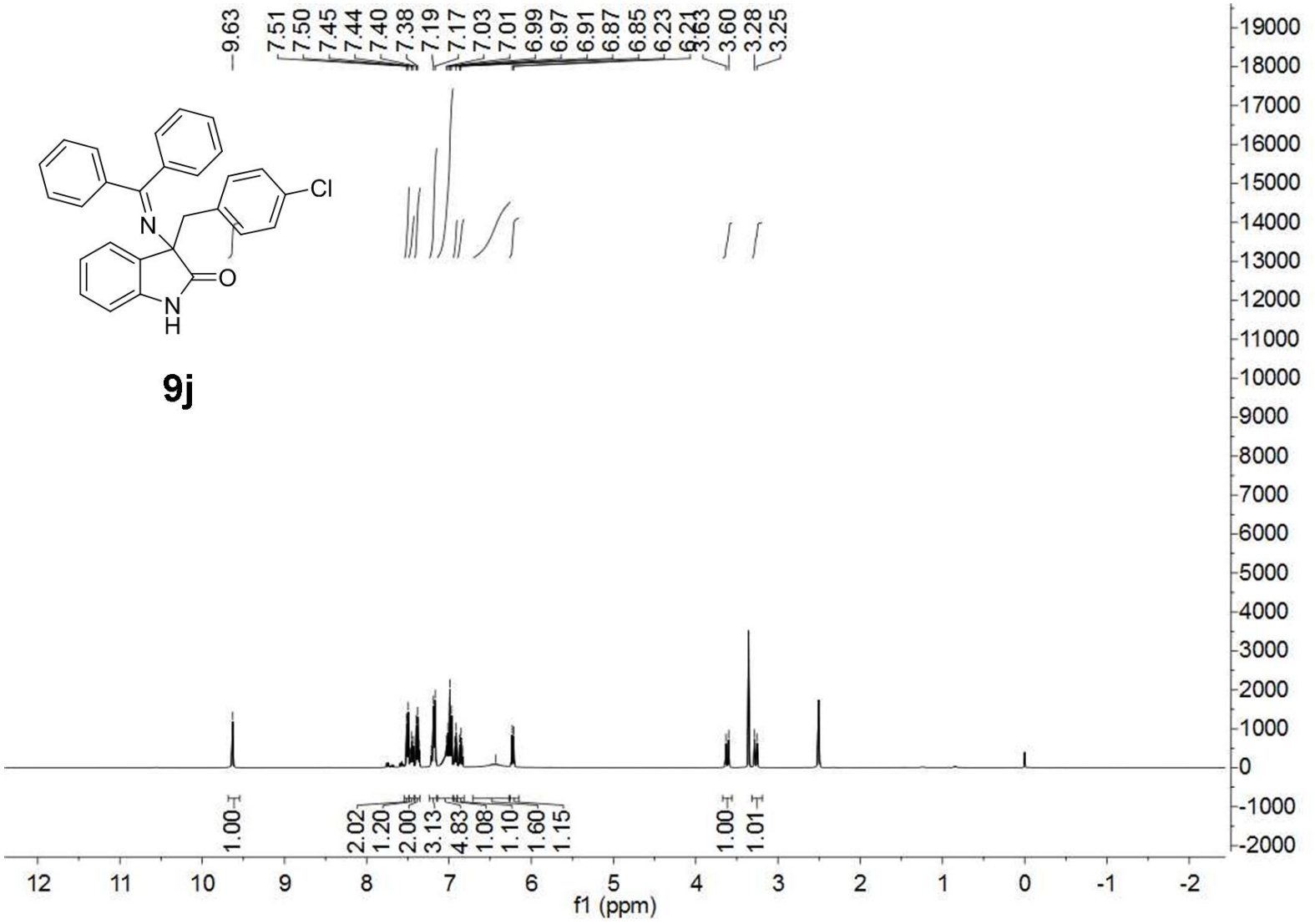
f1 (ppm)

9i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7.03
7.01
6.99
6.97
6.91
6.87
6.85
6.23
6.21
3.63
3.60
3.28
3.25
Cl
N
O
N
H
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
1.00
2.02
1.20
2.00
3.13
4.83
1.08
1.10
1.60
1.15
1.00
1.01
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9
8
7
6
5
4
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2
1
0
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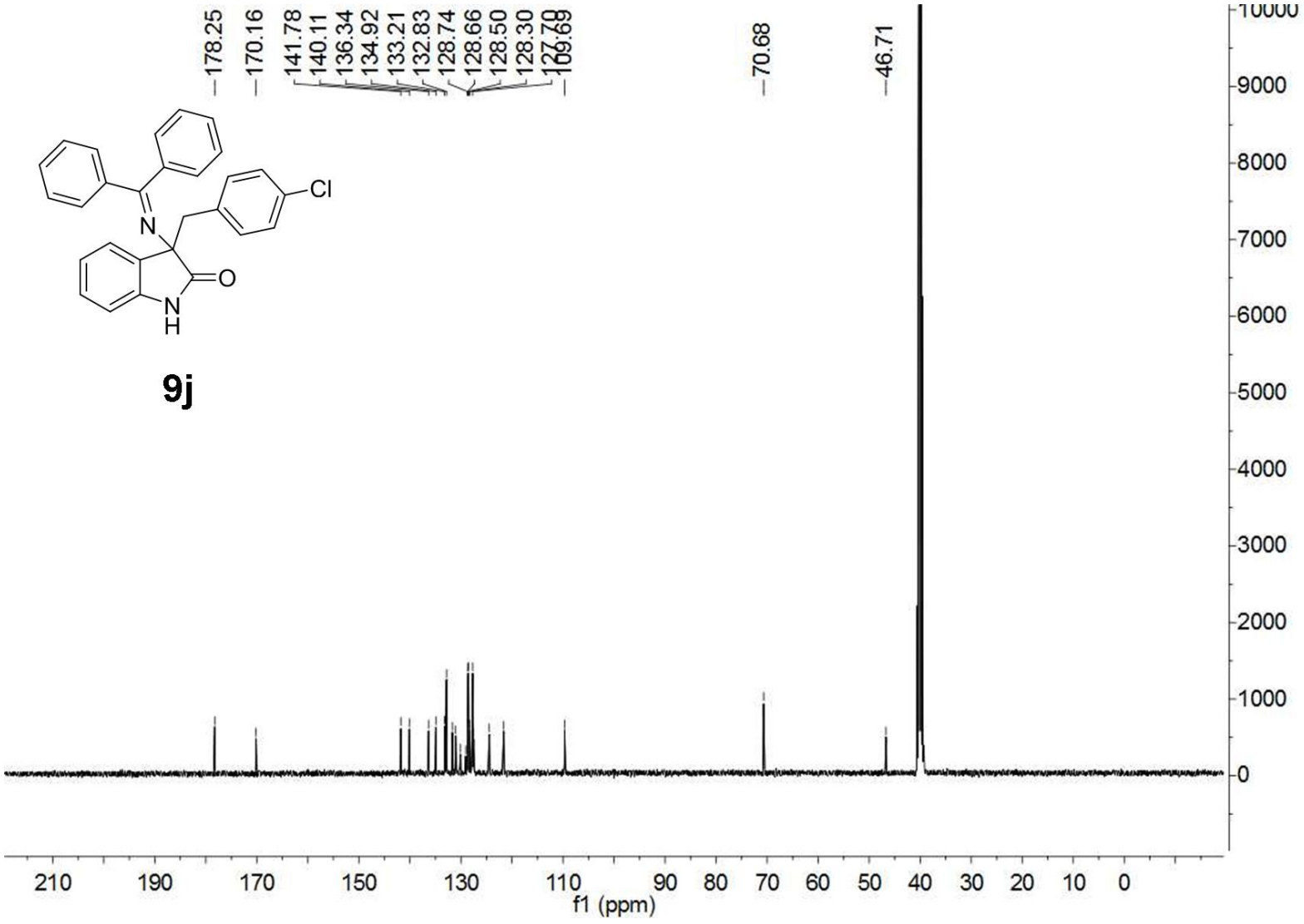
-2
f1 (ppm)

9j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170.16
141.78
140.11
136.34
134.92
133.21
132.83
128.74
128.66
128.50
128.30
70.68
46.71
Cl
N
O
N
H
9j
10000
9000
8000
7000
6000
5000
4000
3000
2000
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0
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170
150
130
110
90
80
70
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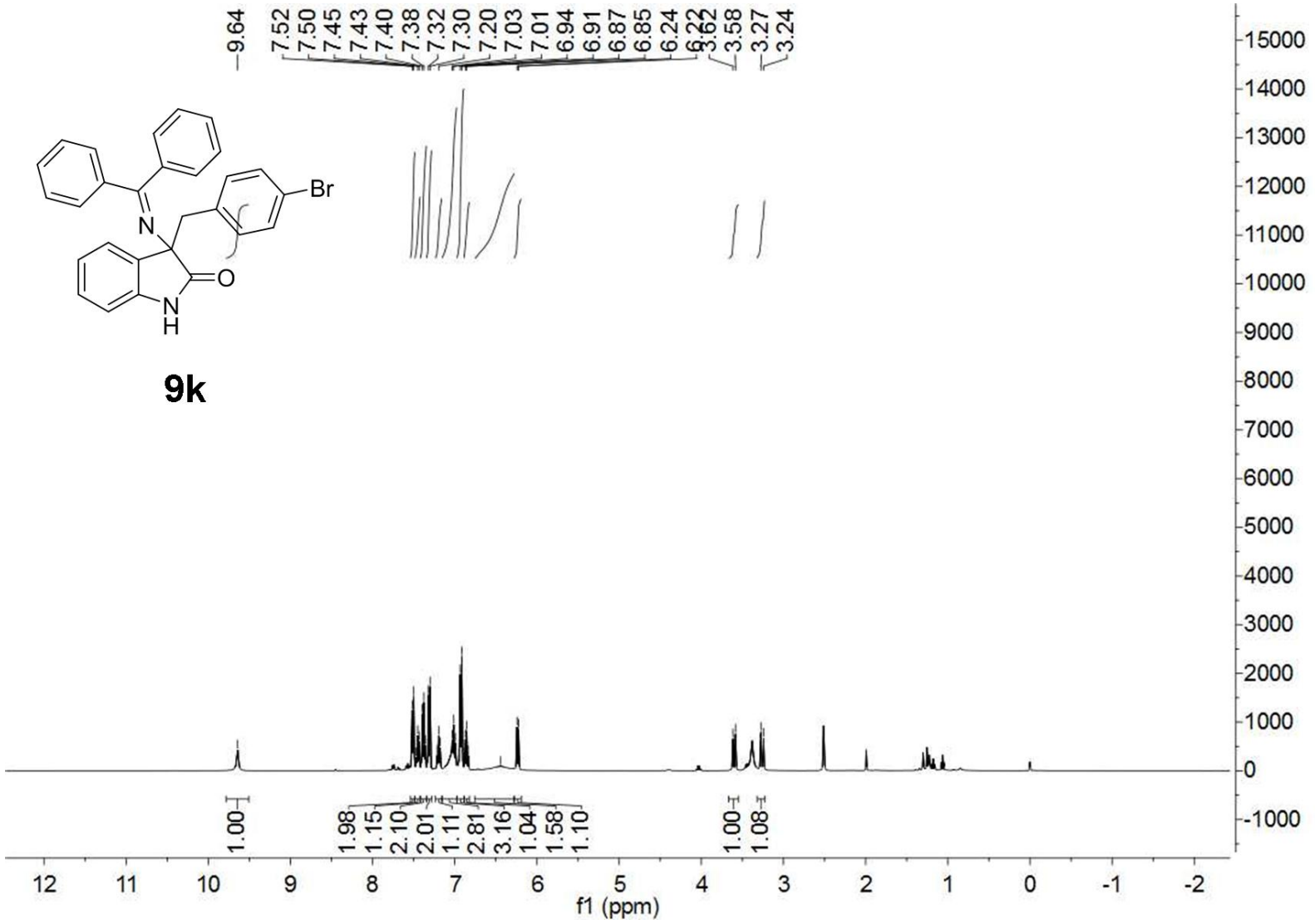
50
40
30
20
10
0
f1 (ppm)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7.32
7.30
7.20
7.03
7.01
6.94
6.91
6.87
6.85
6.24
6.22
3.62
3.58
3.27
3.24
Br
N
O
N
H
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.00
1.98
1.15
2.10
2.01
1.11
2.81
3.16
1.04
1.58
1.10
1.00
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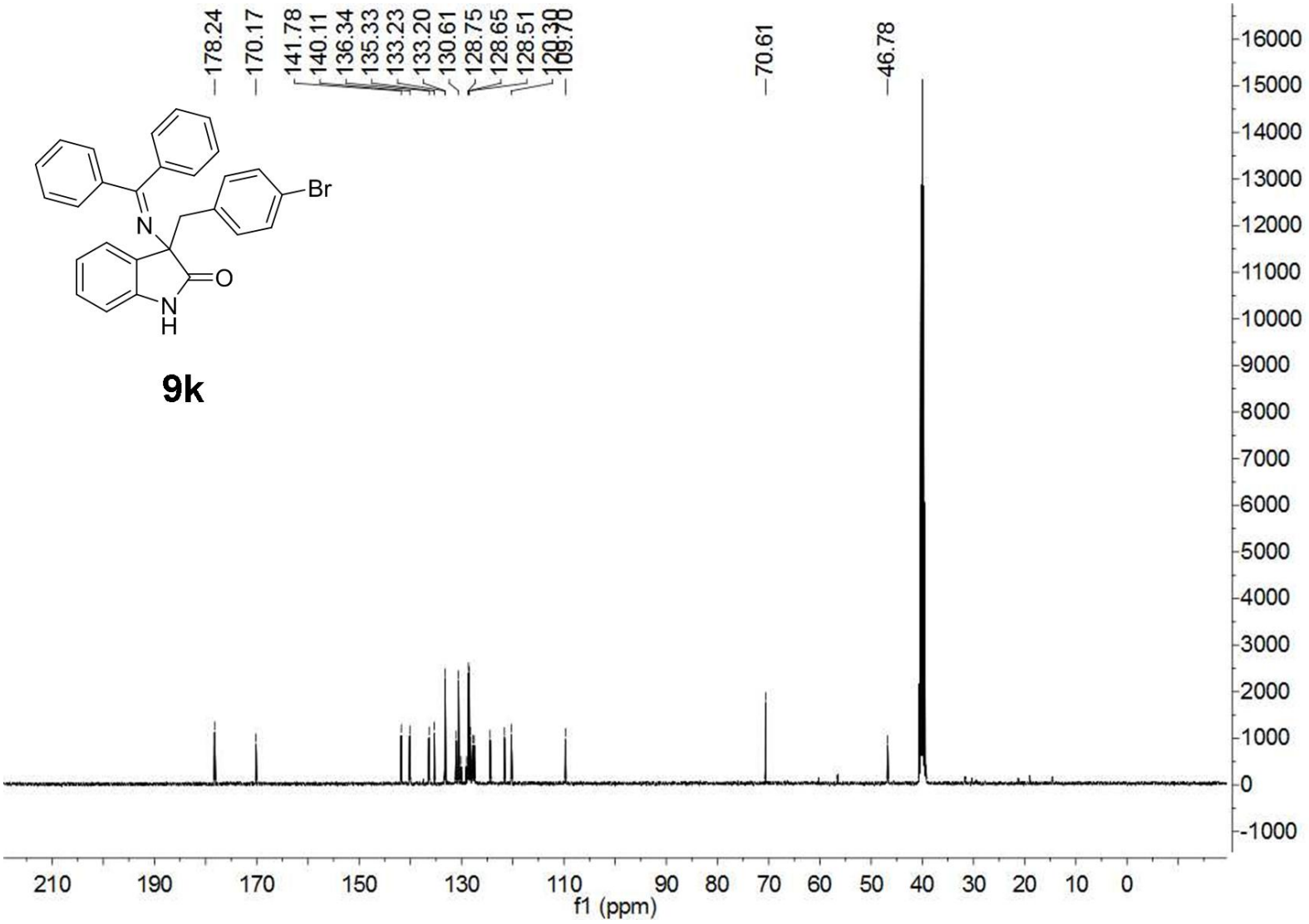
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

9k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141.78
140.11
136.34
135.33
133.23
133.20
130.61
128.75
128.65
128.51
70.61
46.78
Br
N
O
N
H
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
190
170
150
130
110
90
80
70
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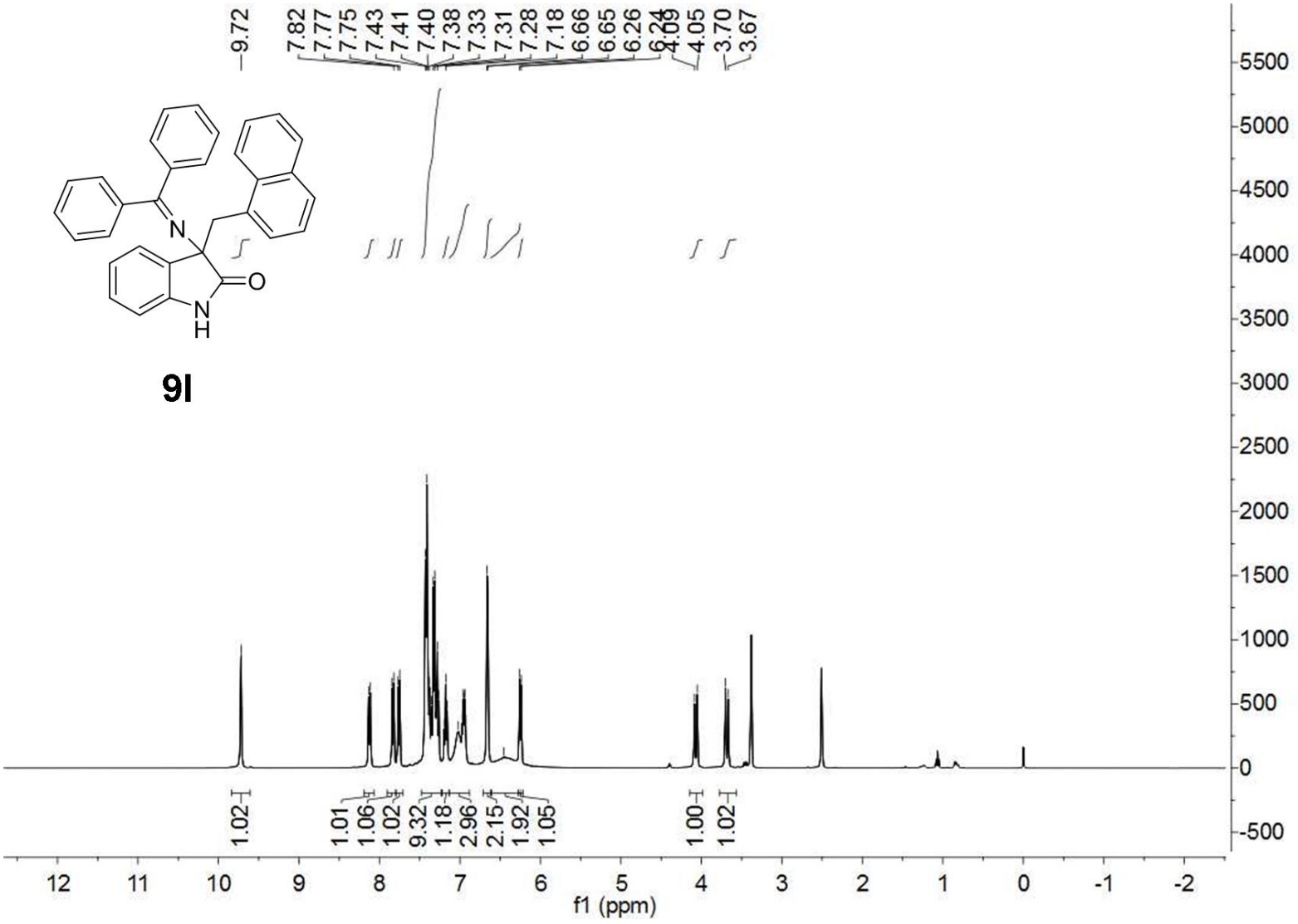
50
40
30
20
10
0
f1 (ppm)

9k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7.38
7.33
7.31
7.28
7.18
6.66
6.65
6.26
6.24
4.09
4.05
3.70
3.67
N
O
N
H
1.02
1.01
1.06
1.02
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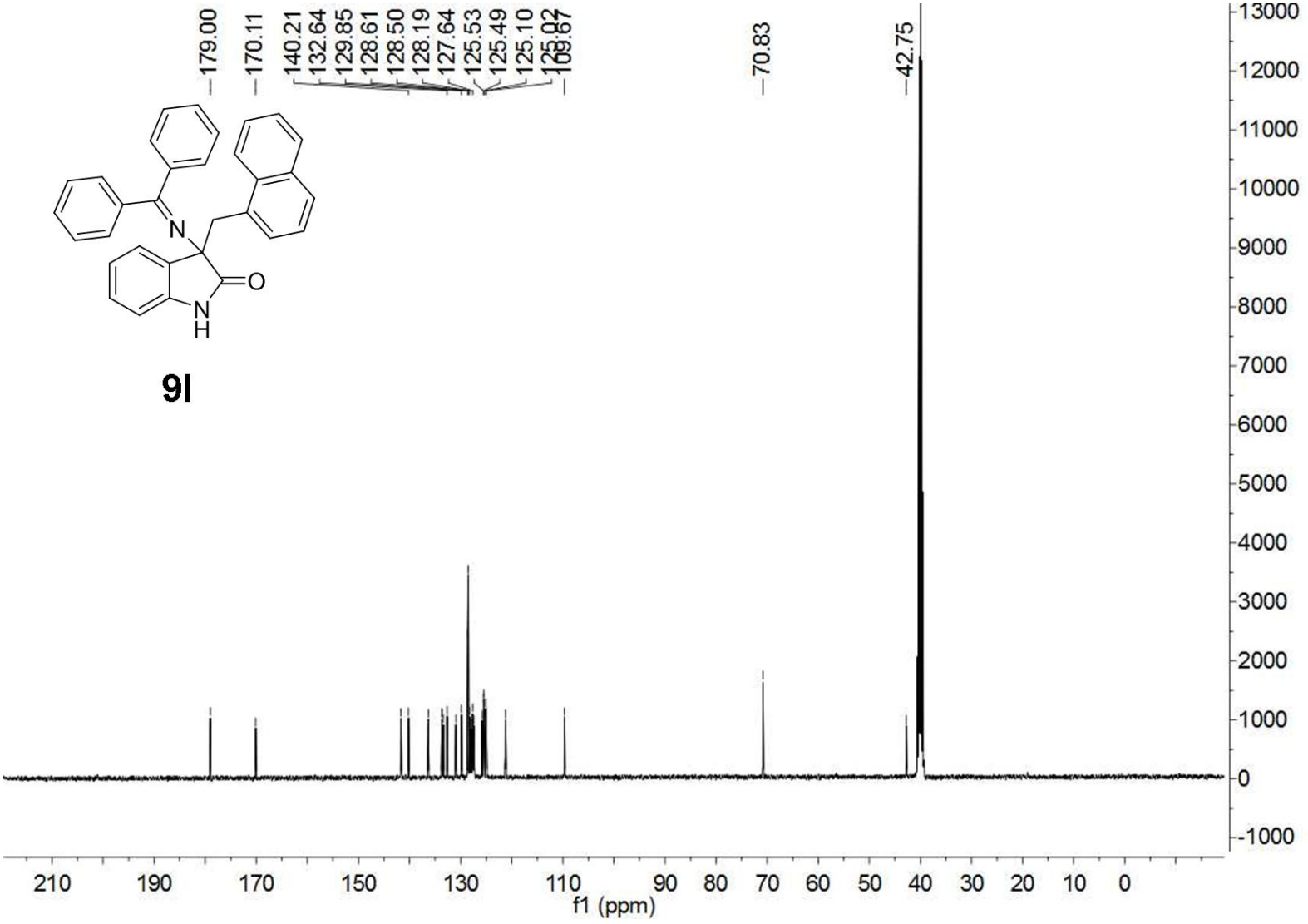
1.18
2.96
2.15
1.92
1.05
1.00
1.02
f1 (ppm)

9l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170.11
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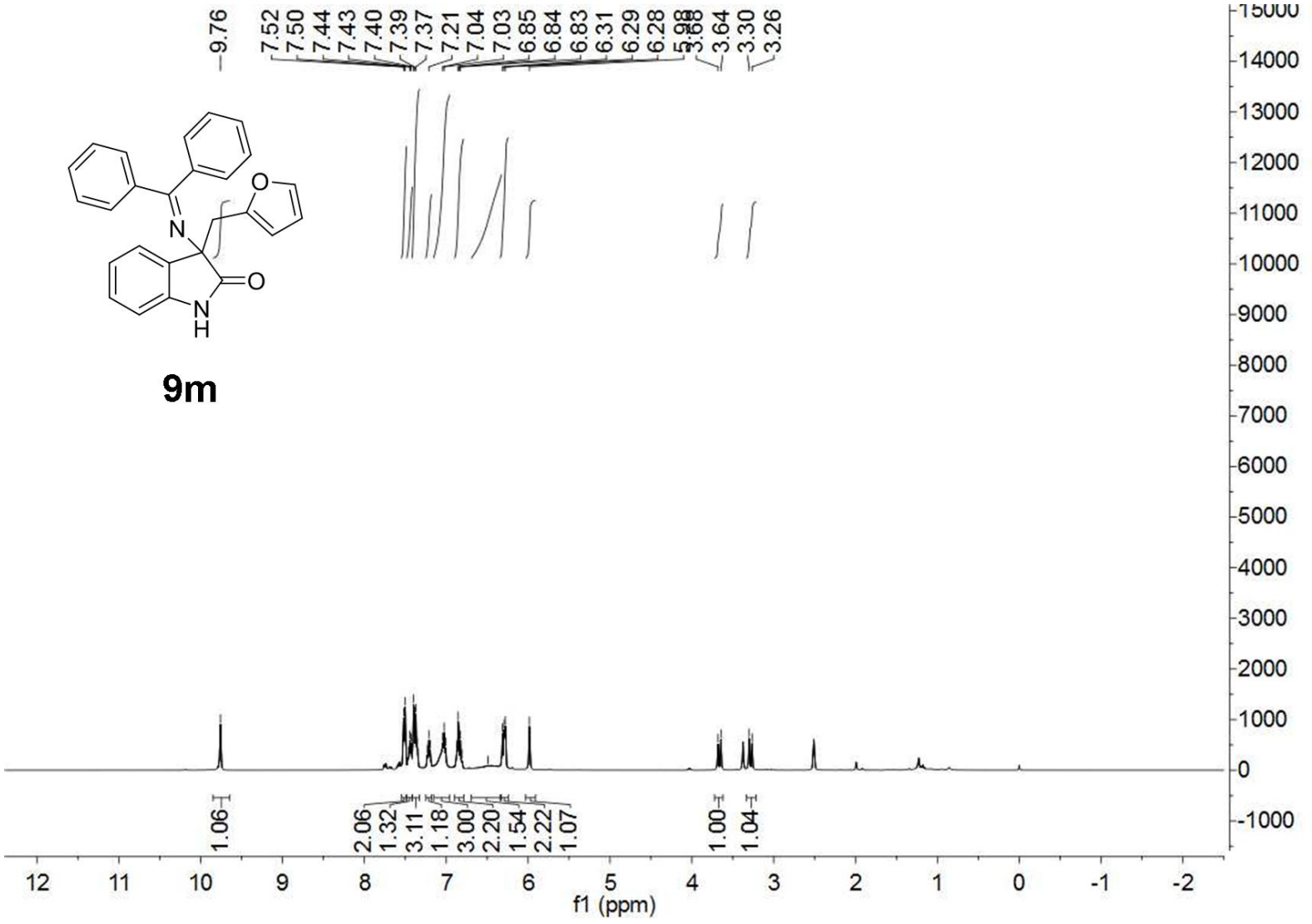
129.85
128.61
128.50
128.19
127.64
125.53
125.49
125.10
70.83
42.75
N
O
N
H
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9l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7.21
7.04
7.03
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6.84
6.83
6.31
6.29
6.28
3.64
3.30
3.26
O
N
O
N
H
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.06
2.06
1.32
3.11
1.18
3.00
2.20
1.54
2.22
1.07
1.00
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9
8
7
6
5
4
3
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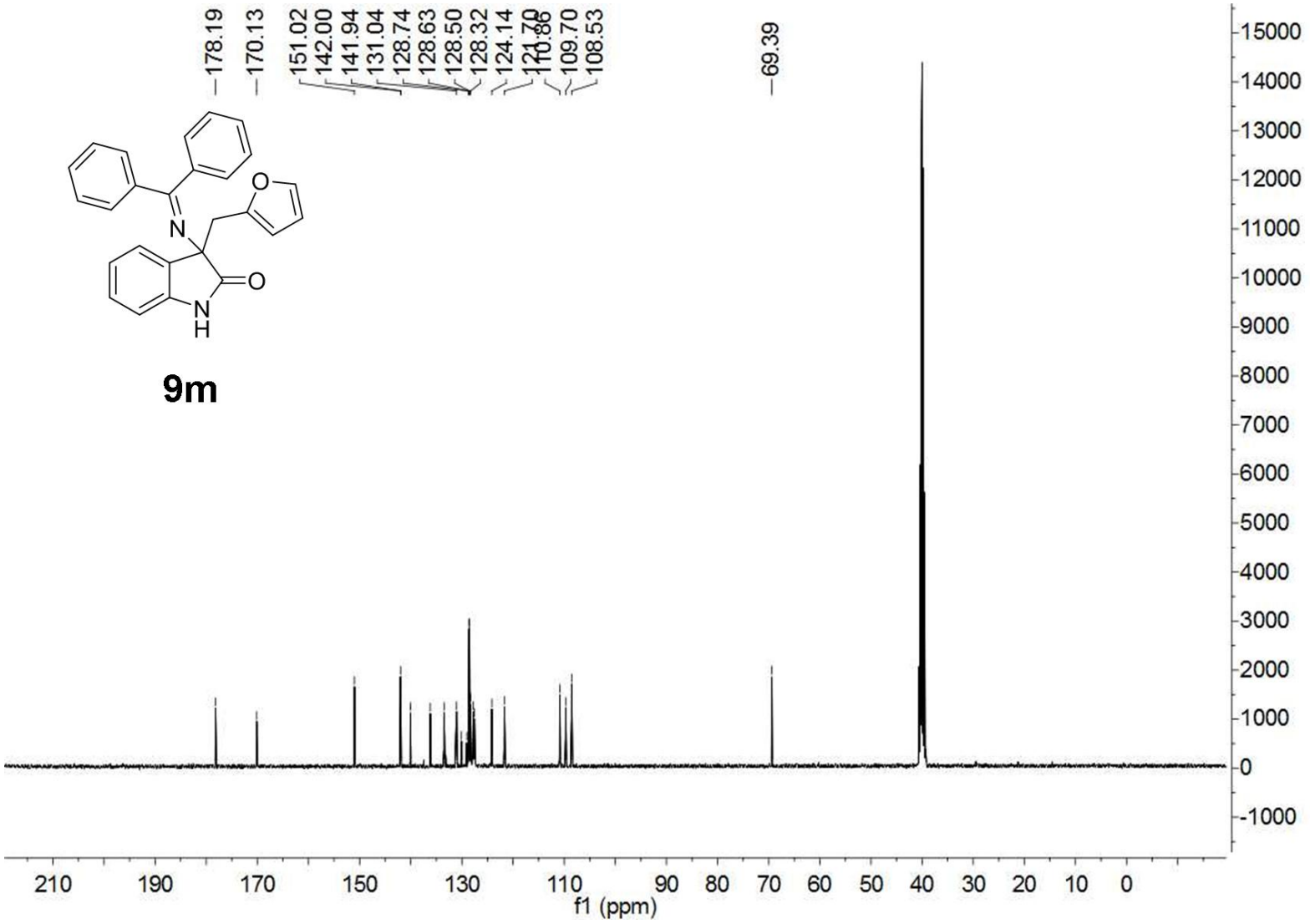
1
0
-1
-2
f1 (ppm)

9m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141.94
131.04
128.74
128.63
128.50
128.32
124.14
121.70
110.88
109.70
108.53
69.39
O
N
O
N
H
9m
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
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170
150
130
110
90
80
70
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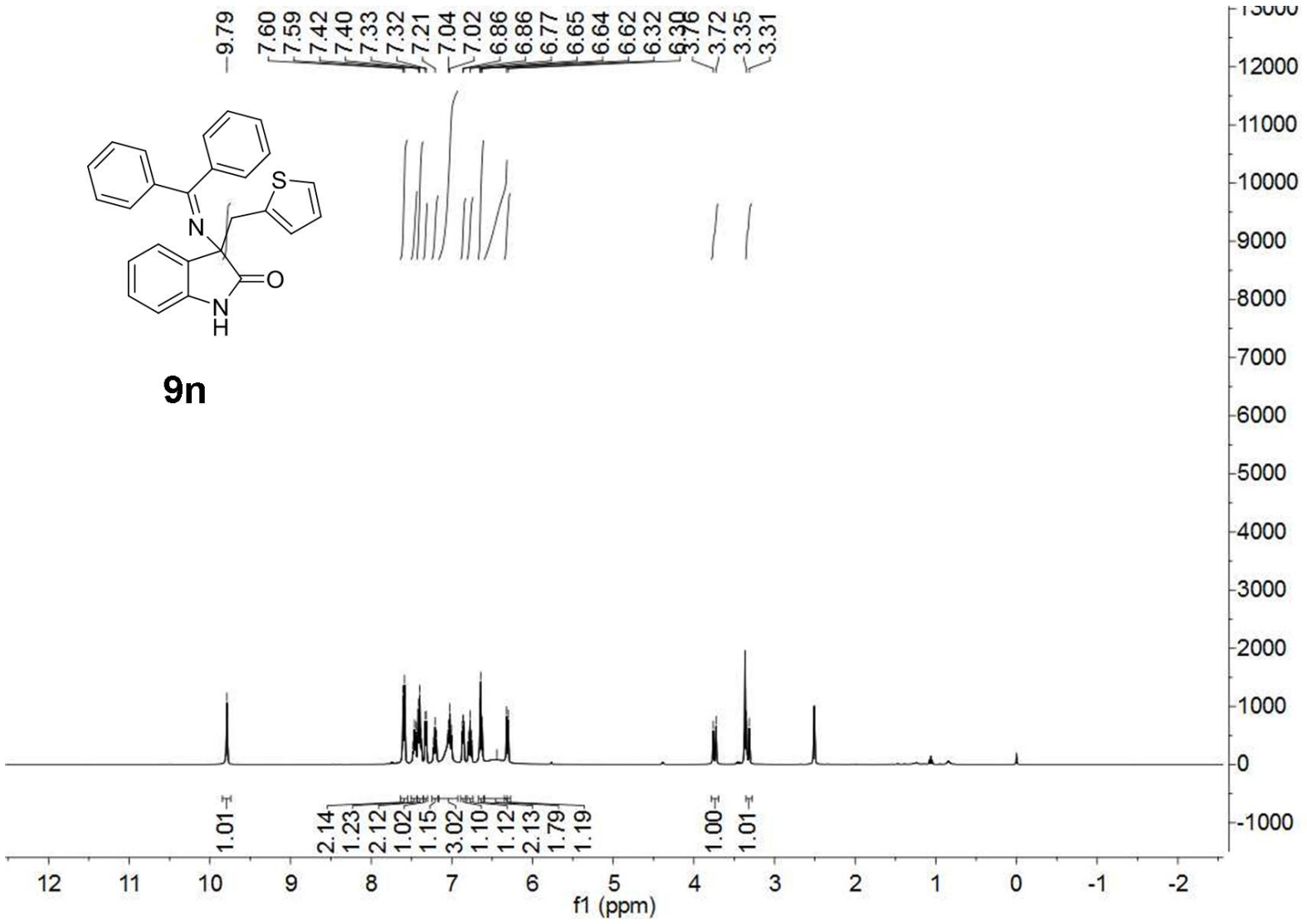
50
40
30
20
10
0
f1 (ppm)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7.02
6.86
6.86
6.77
6.65
6.64
6.62
6.32
3.72
3.35
3.31
S
N
O
N
H
9n
1.01
2.14
1.23
2.12
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3.02
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1.12
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1.19
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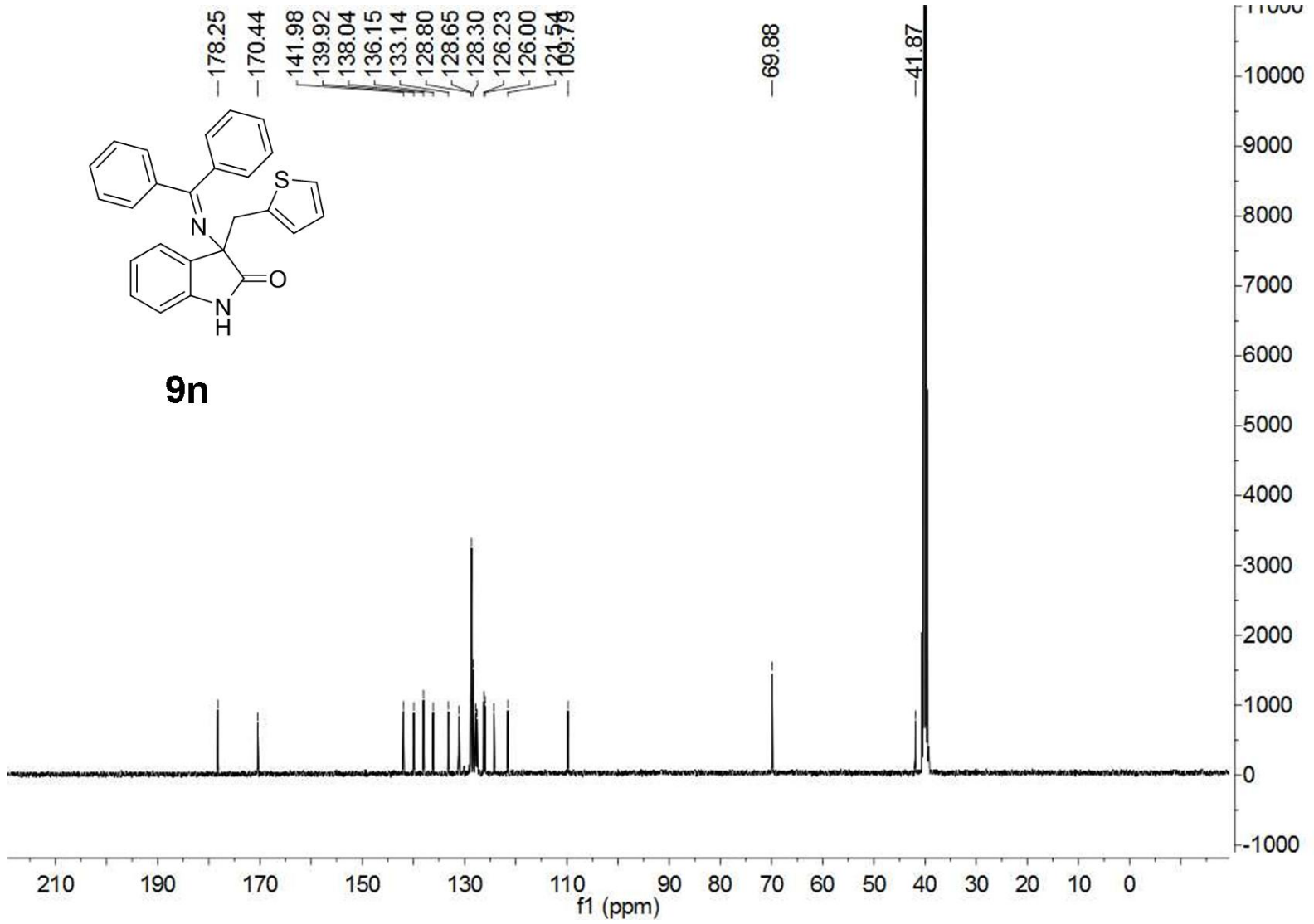
1.01
f1 (ppm)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128.80
128.65
128.30
126.23
126.00
121.54
109.79
69.88
41.87
S
N
O
N
H
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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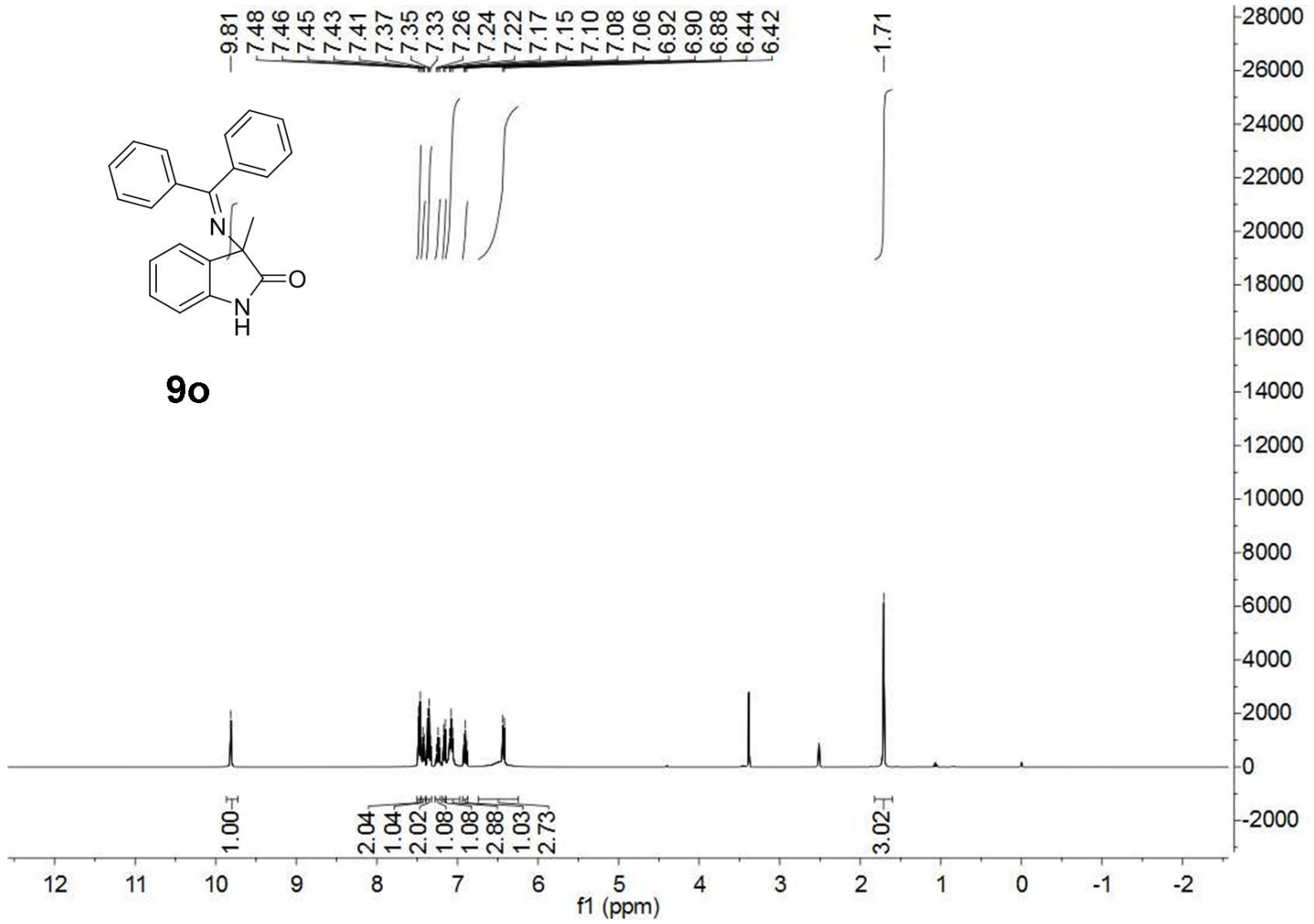
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9n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7.35
7.33
7.26
7.24
7.22
7.17
7.15
7.10
7.08
7.06
6.92
6.90
6.88
6.44
6.42
1.71
N
O
N
H
9o
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
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1.00
2.04
1.04
2.02
1.08
1.08
2.88
1.03
2.73
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10
9
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4
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2
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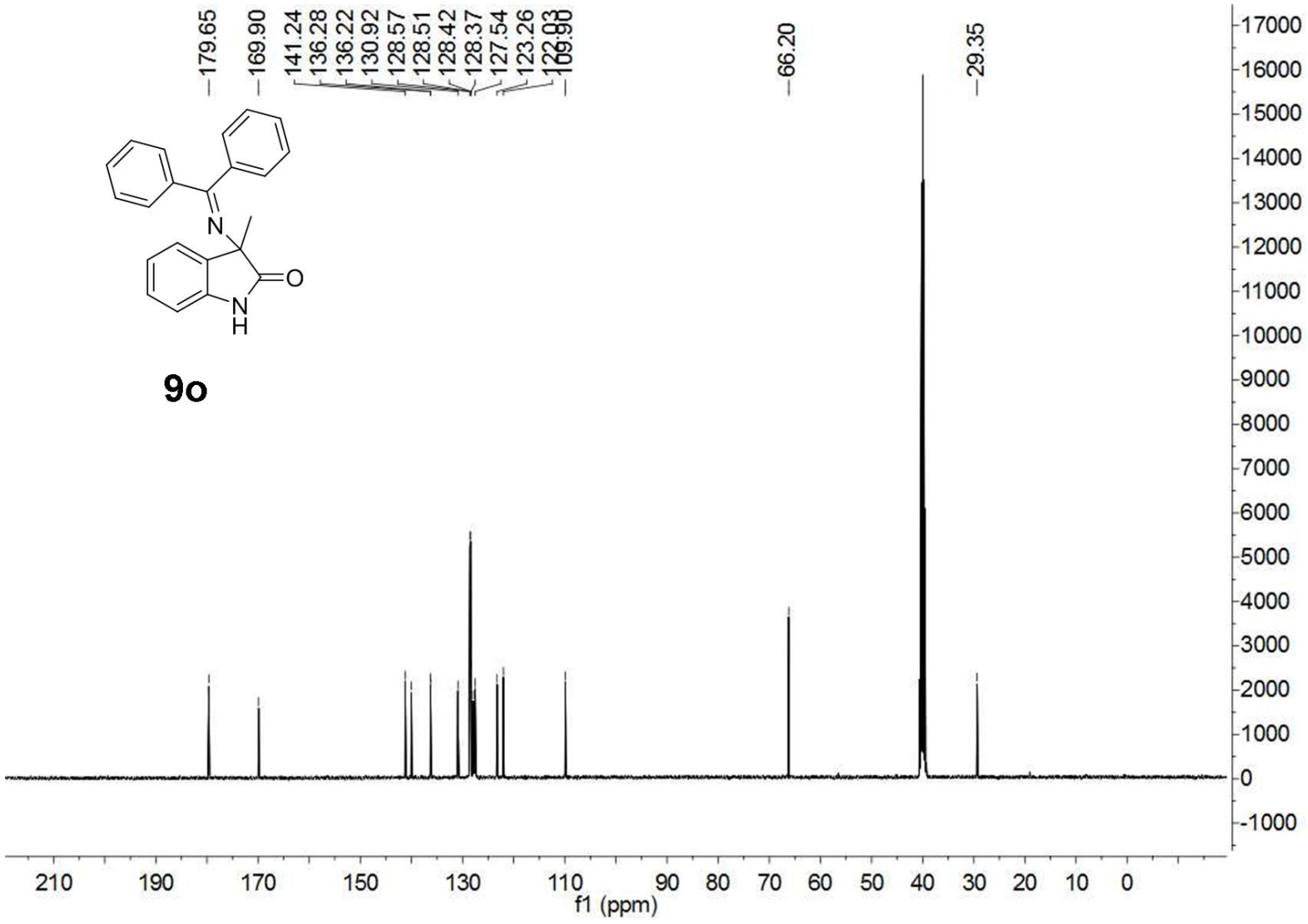
-2
f1 (ppm)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130.92
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128.51
128.42
128.37
127.54
123.26
66.20
29.35
N
O
N
H
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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190
170
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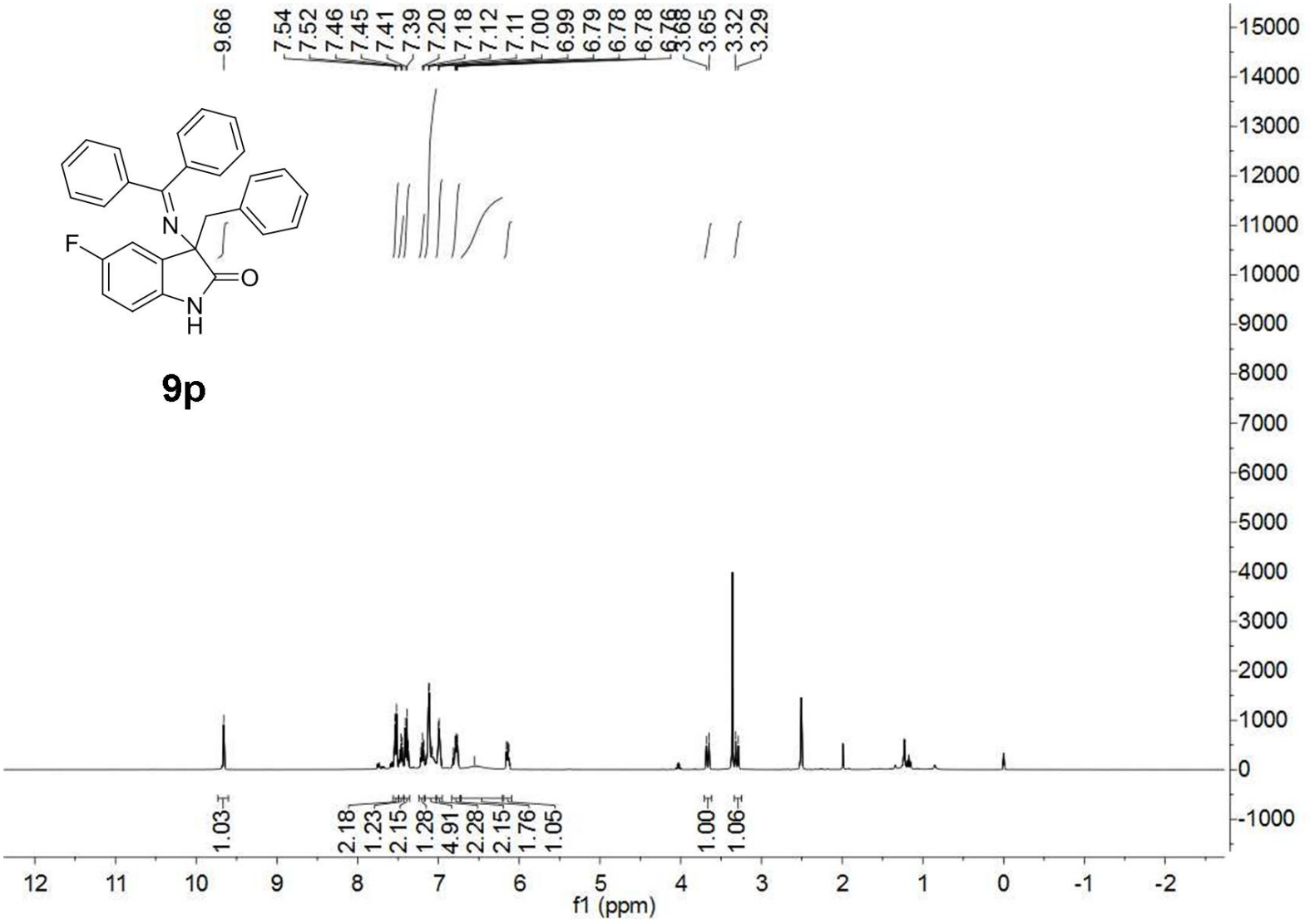
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9o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7.39
7.20
7.18
7.12
7.11
7.00
6.99
6.79
6.78
6.78
3.65
3.32
3.29
F
N
O
N
H
9p
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.03
2.18
1.23
2.15
1.28
4.91
2.28
2.15
1.76
1.05
1.00
1.06
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10
9
8
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5
4
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2
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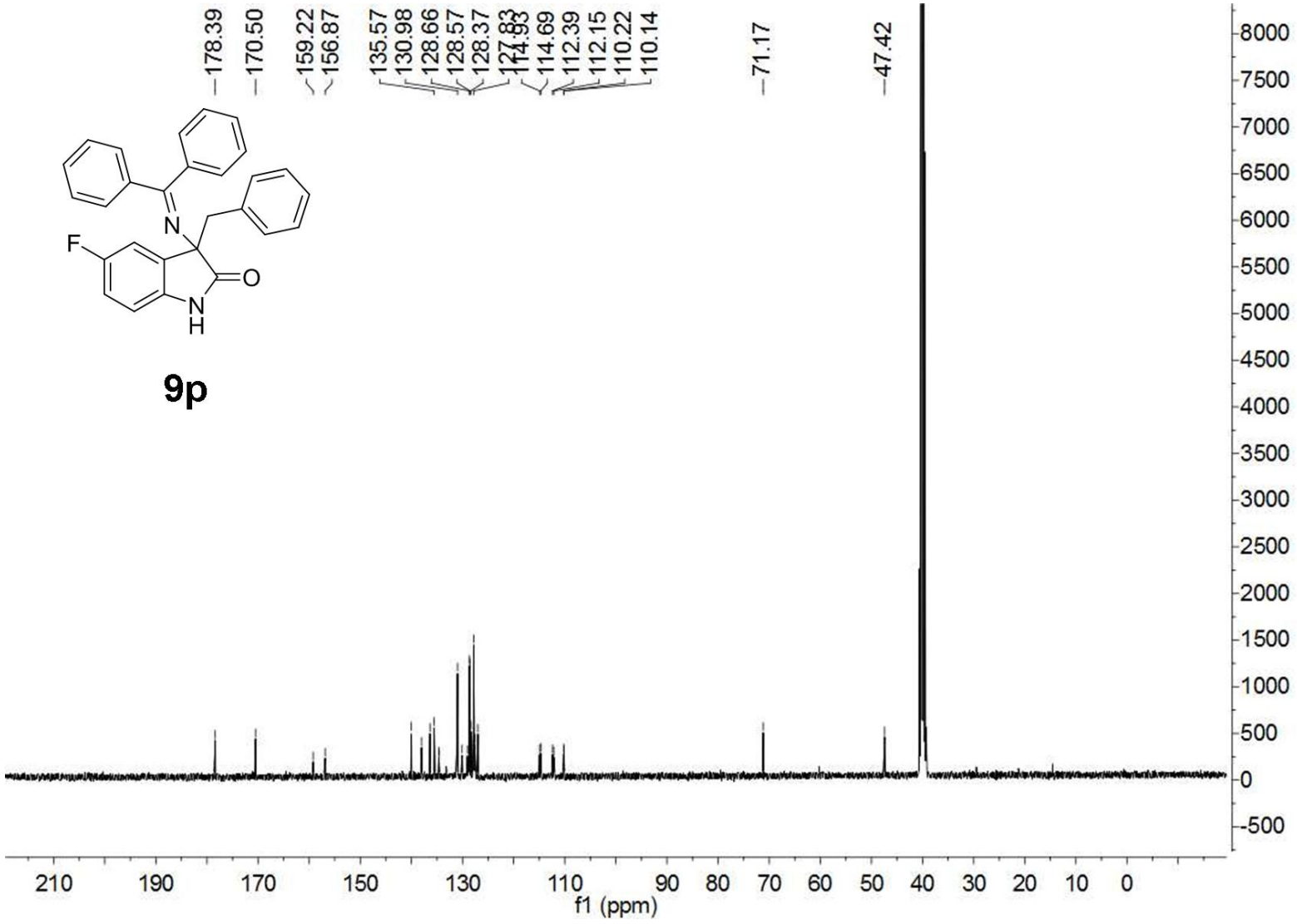
-1
-2
f1 (ppm)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128.66
128.57
128.37
114.69
112.39
112.15
110.22
110.14
71.17
47.42
F
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O
N
H
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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190
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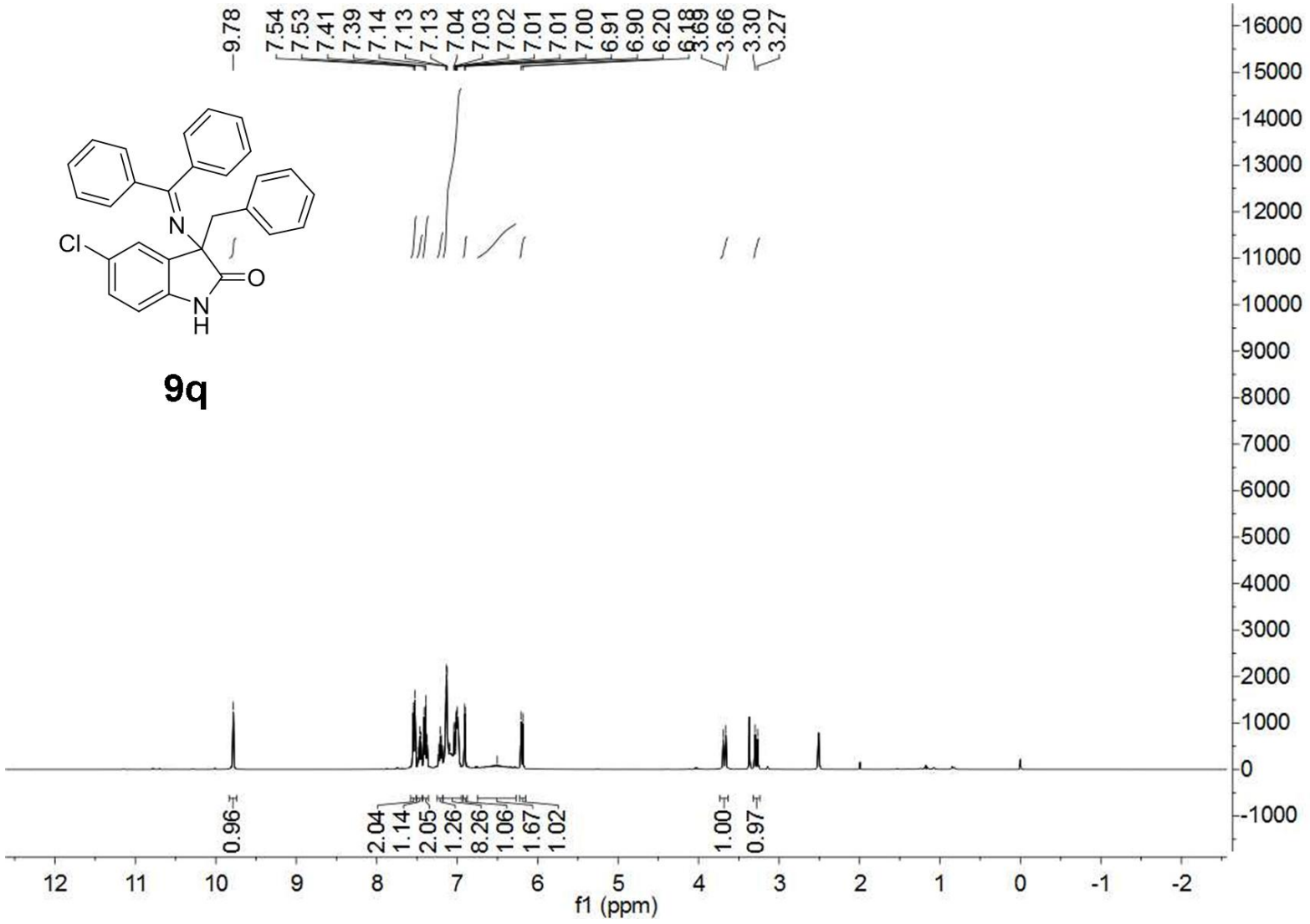
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

9p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7.14
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7.13
7.04
7.03
7.02
7.01
7.01
7.00
6.91
6.90
6.20
6.18
3.69
3.66
3.30
3.27
Cl
N
O
N
H
9q
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0.96
2.04
1.14
2.05
1.26
8.26
1.06
1.67
1.02
1.00
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9
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4
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2
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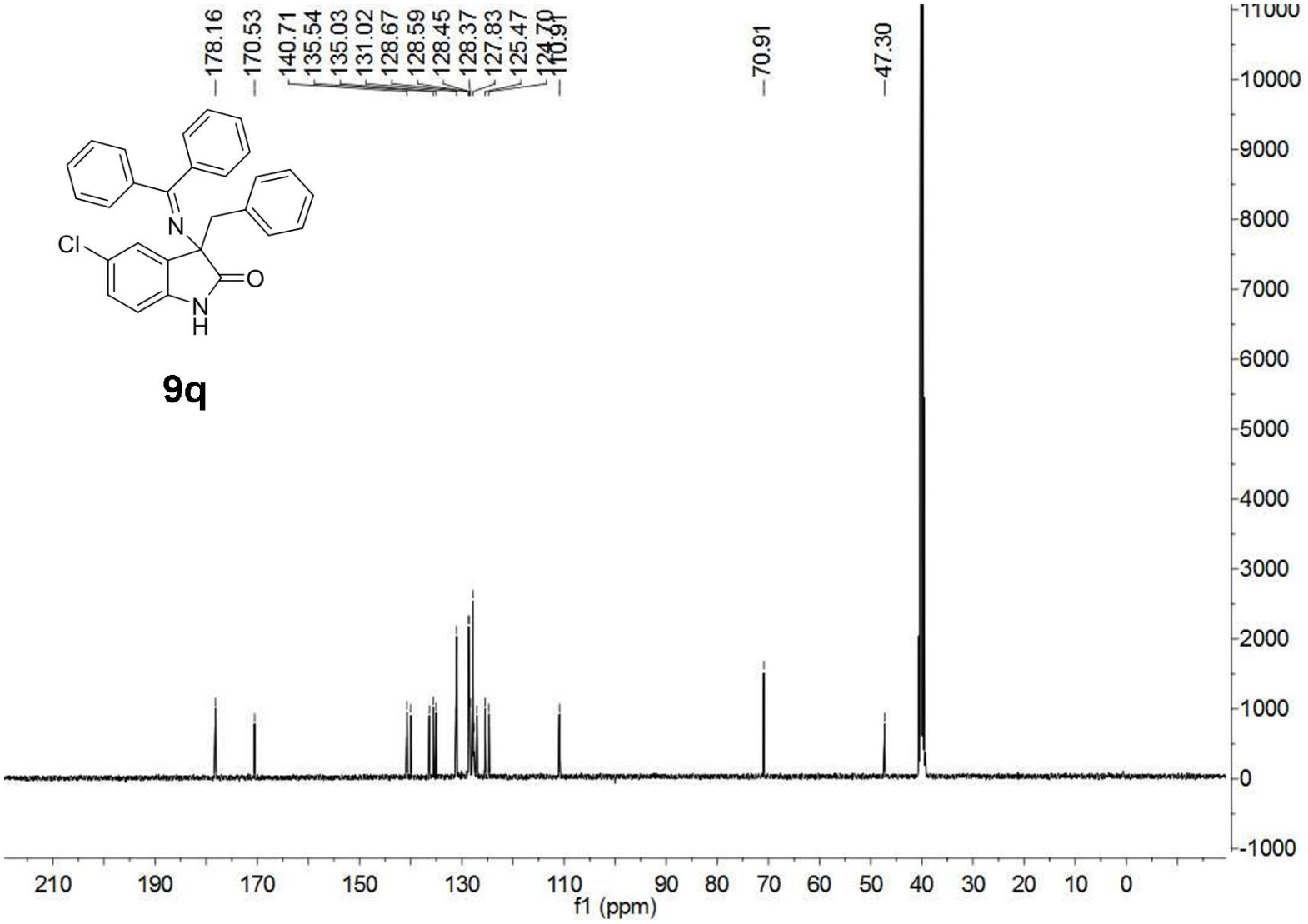
-1
-2
f1 (ppm)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135.54
135.03
131.02
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128.59
128.45
128.37
127.83
125.47
124.70
110.91
70.91
47.30
Cl
N
O
N
H
9q
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
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150
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90
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70
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50
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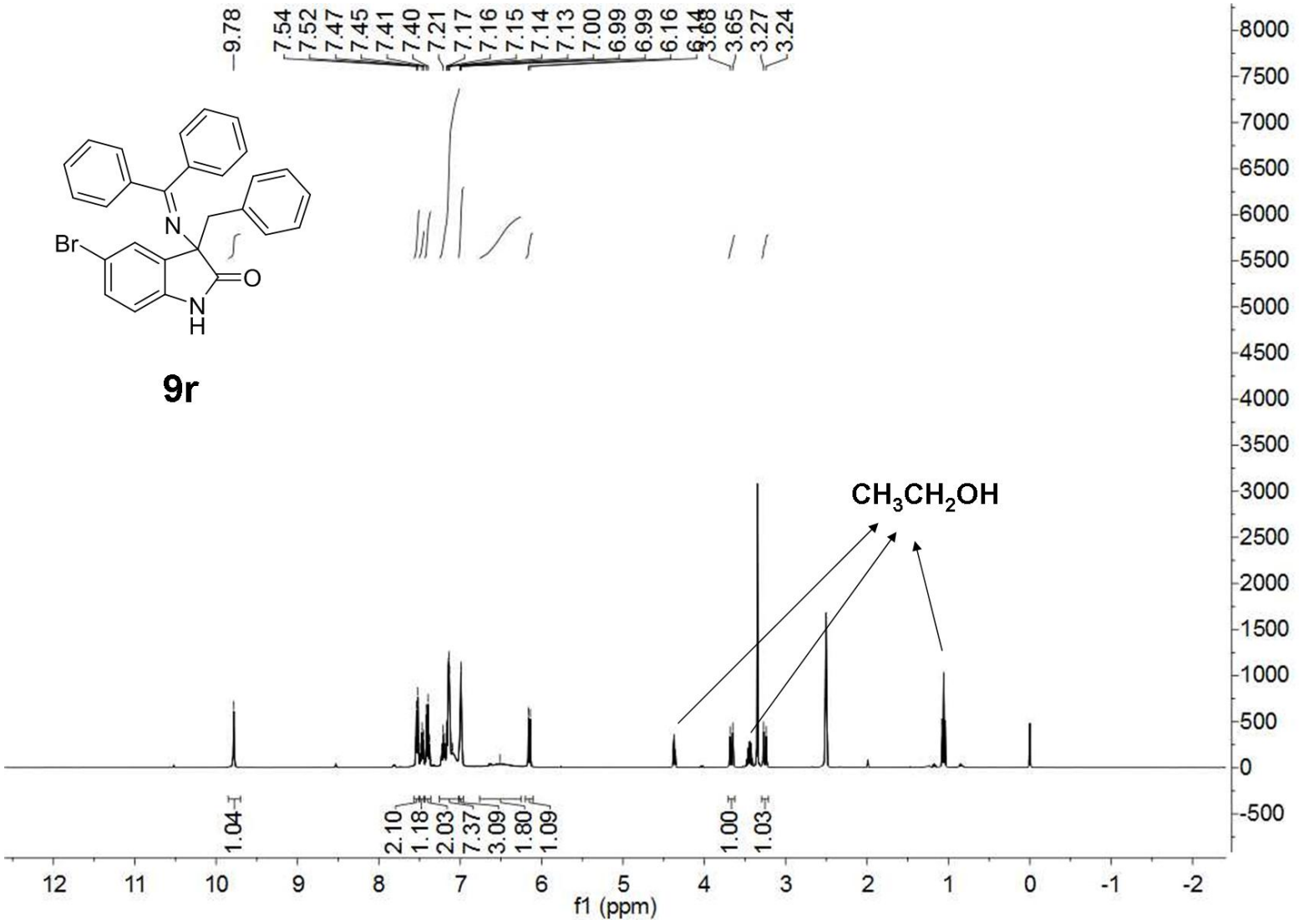
10
0
f1 (ppm)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7.47
7.45
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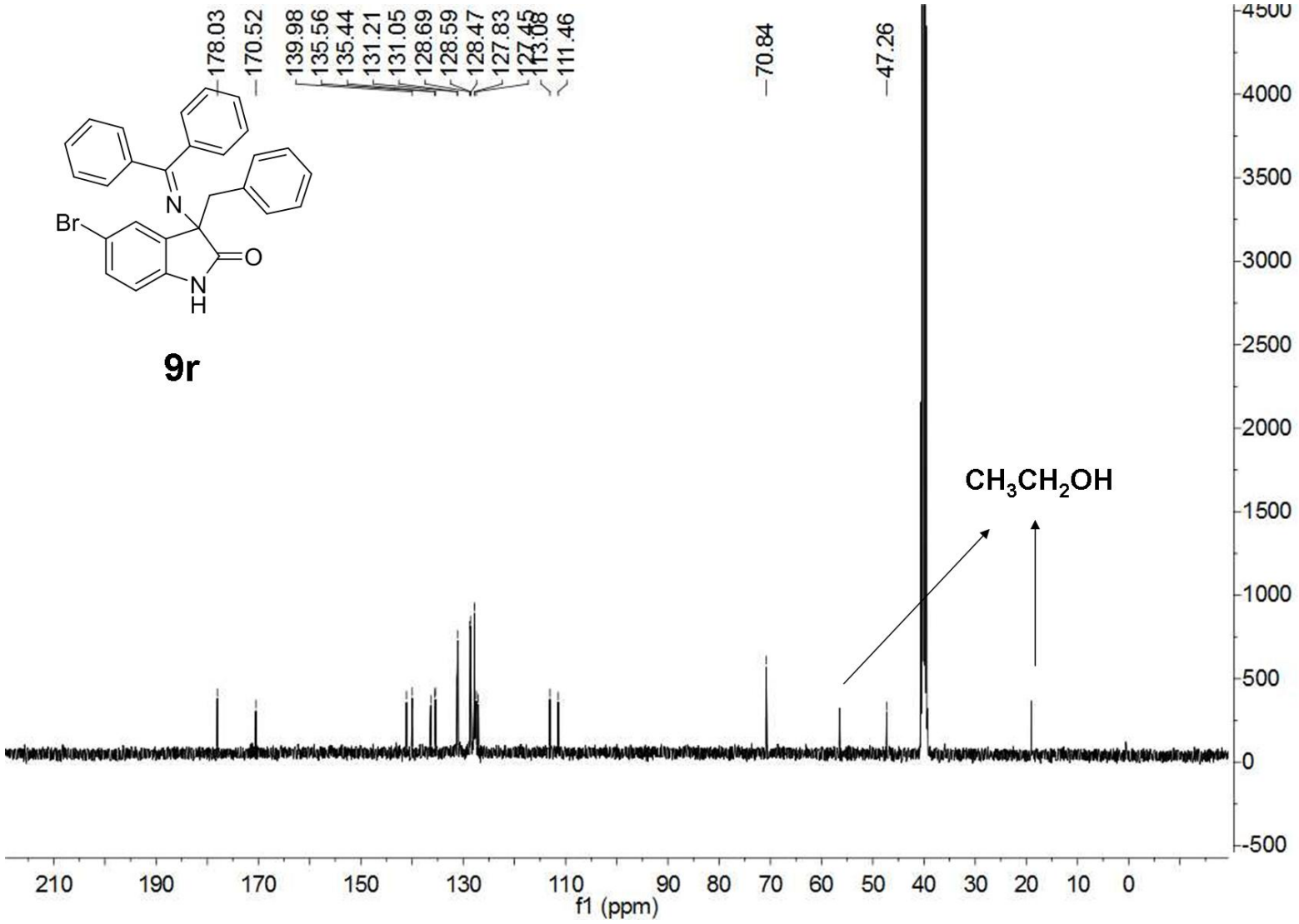
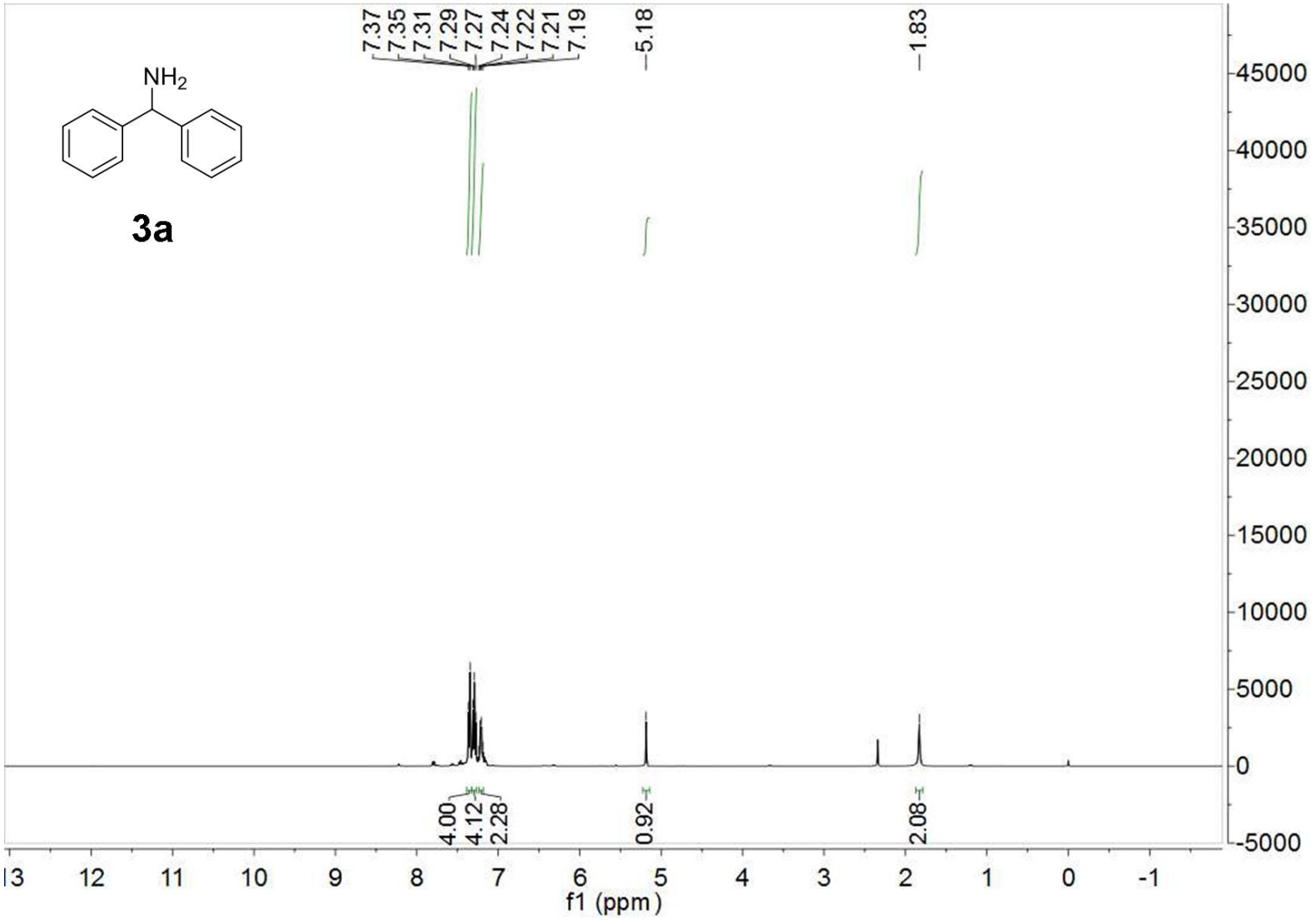
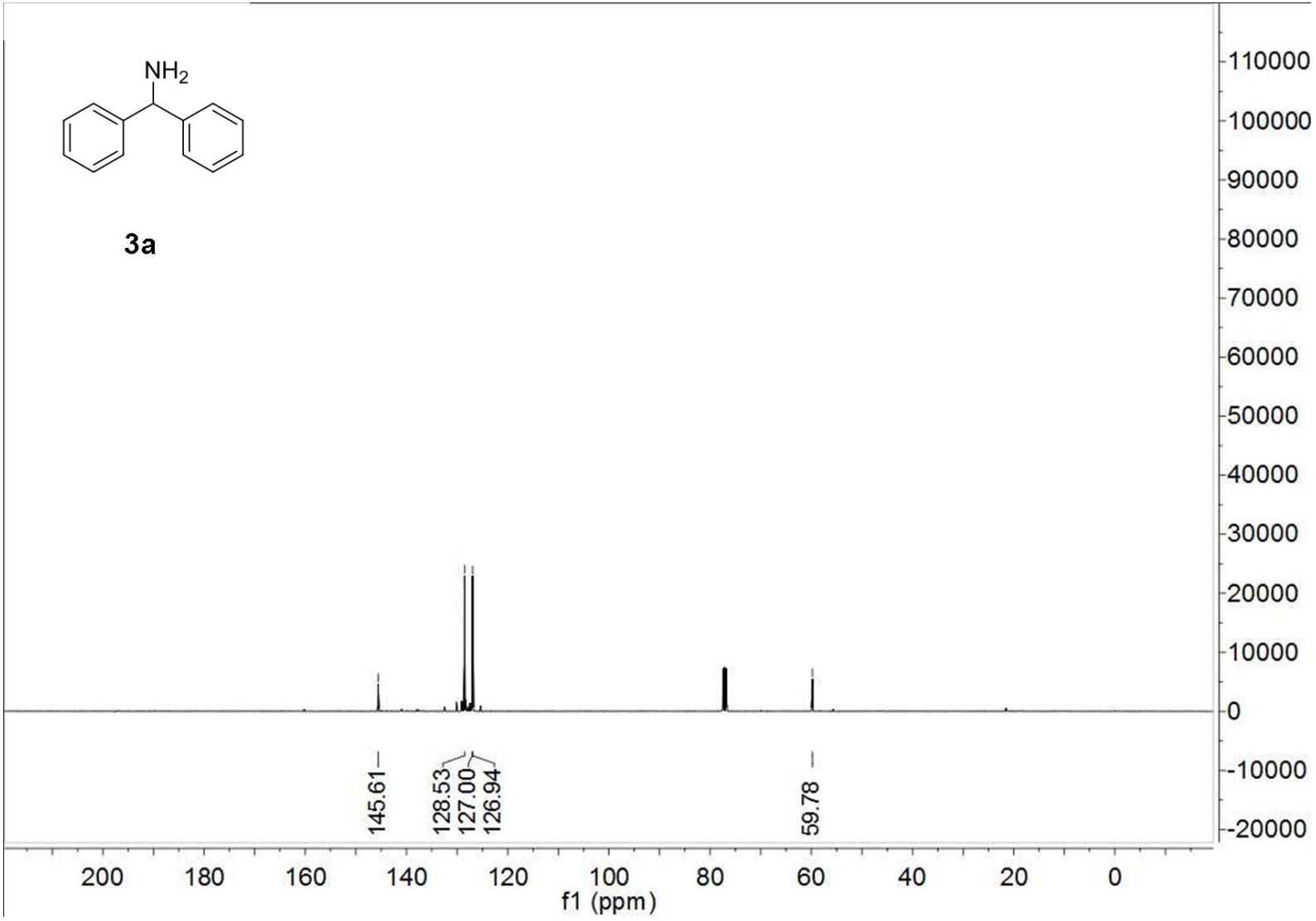
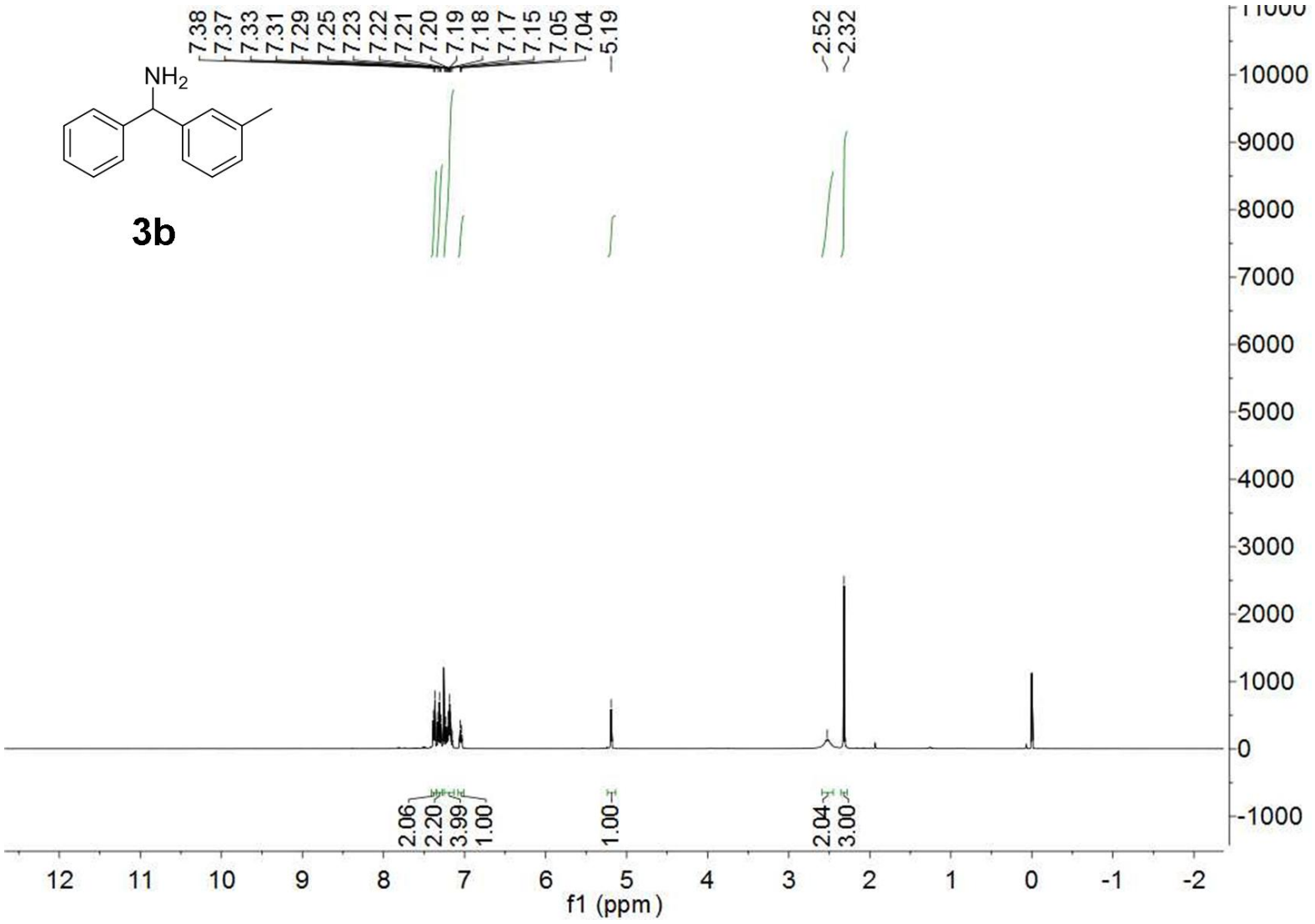
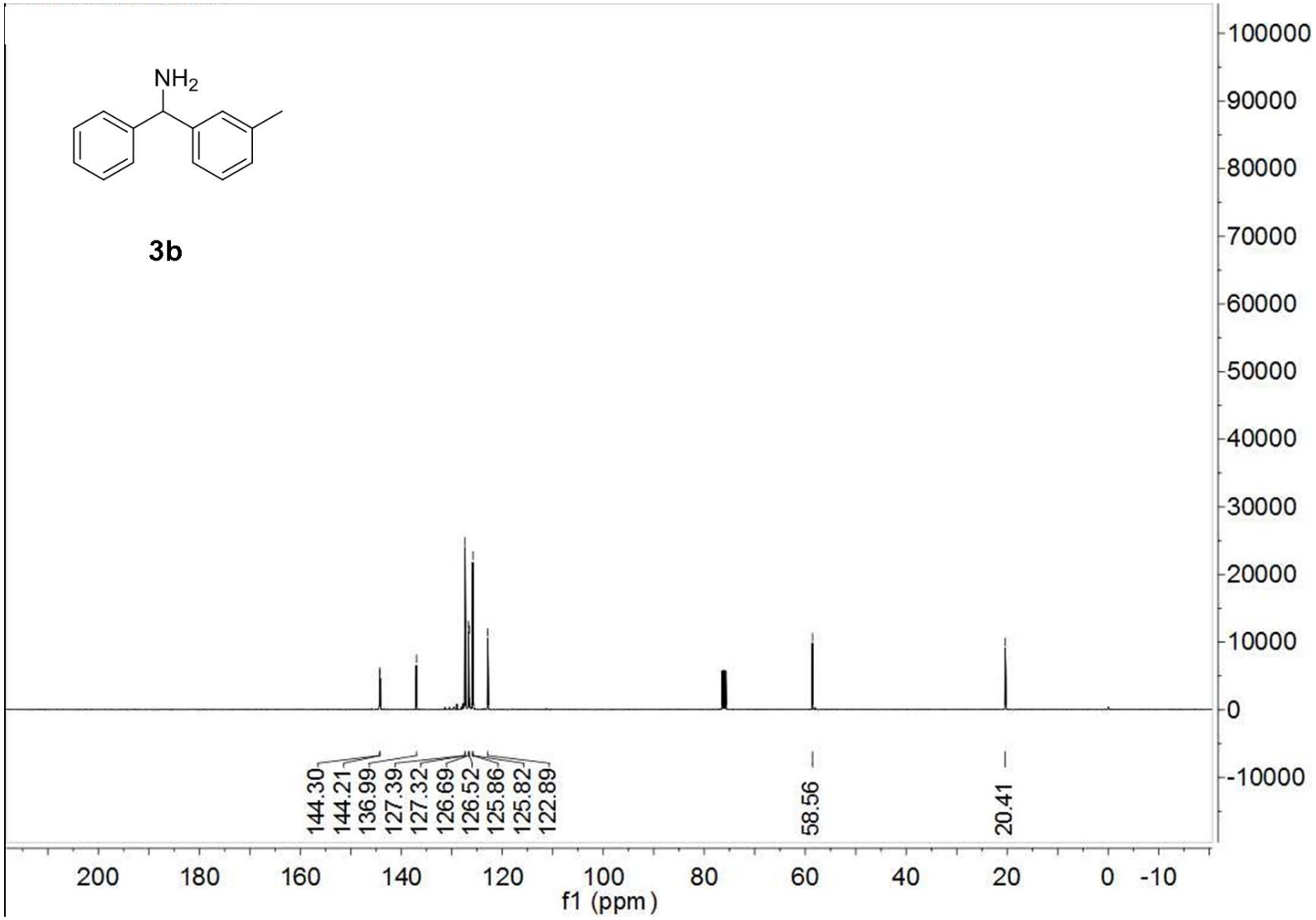
7.40
7.21
7.17
7.16
7.15
7.14
7.13
7.00
6.99
6.99
6.16
6.14
3.68
3.65
3.27
3.24
Br
N
O
N
H
9r
CH3CH2OH
1.04
2.10
1.18
2.03
7.37
3.09
1.80
1.09
1.00
1.03
f1 (ppm)

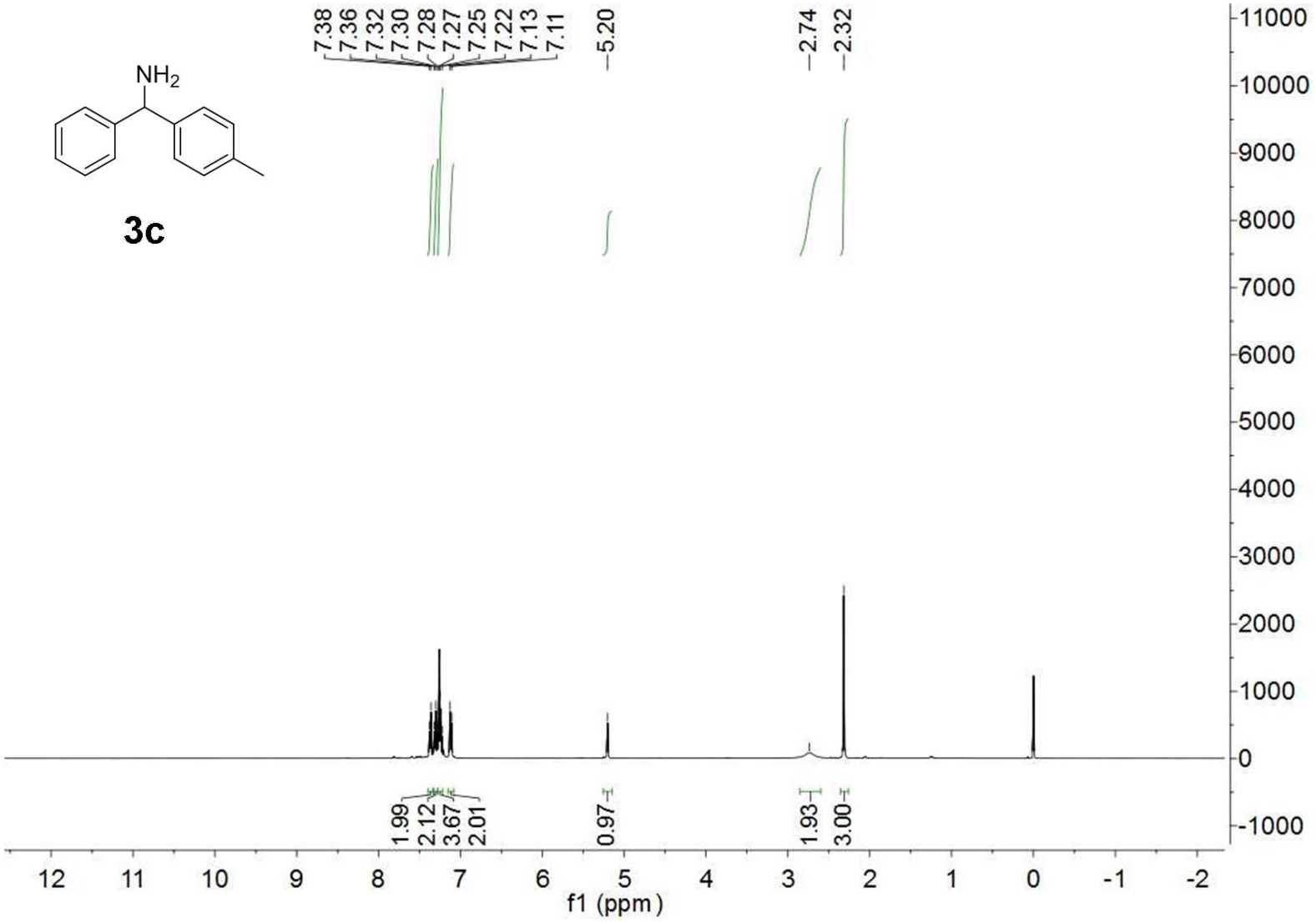

178.03
170.52
139.98
135.56
135.44
131.21
131.05
128.69
128.59
128.47
127.83
111.46
70.84
47.26
Br
N
O
N
H
9r
CH3CH2OH
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

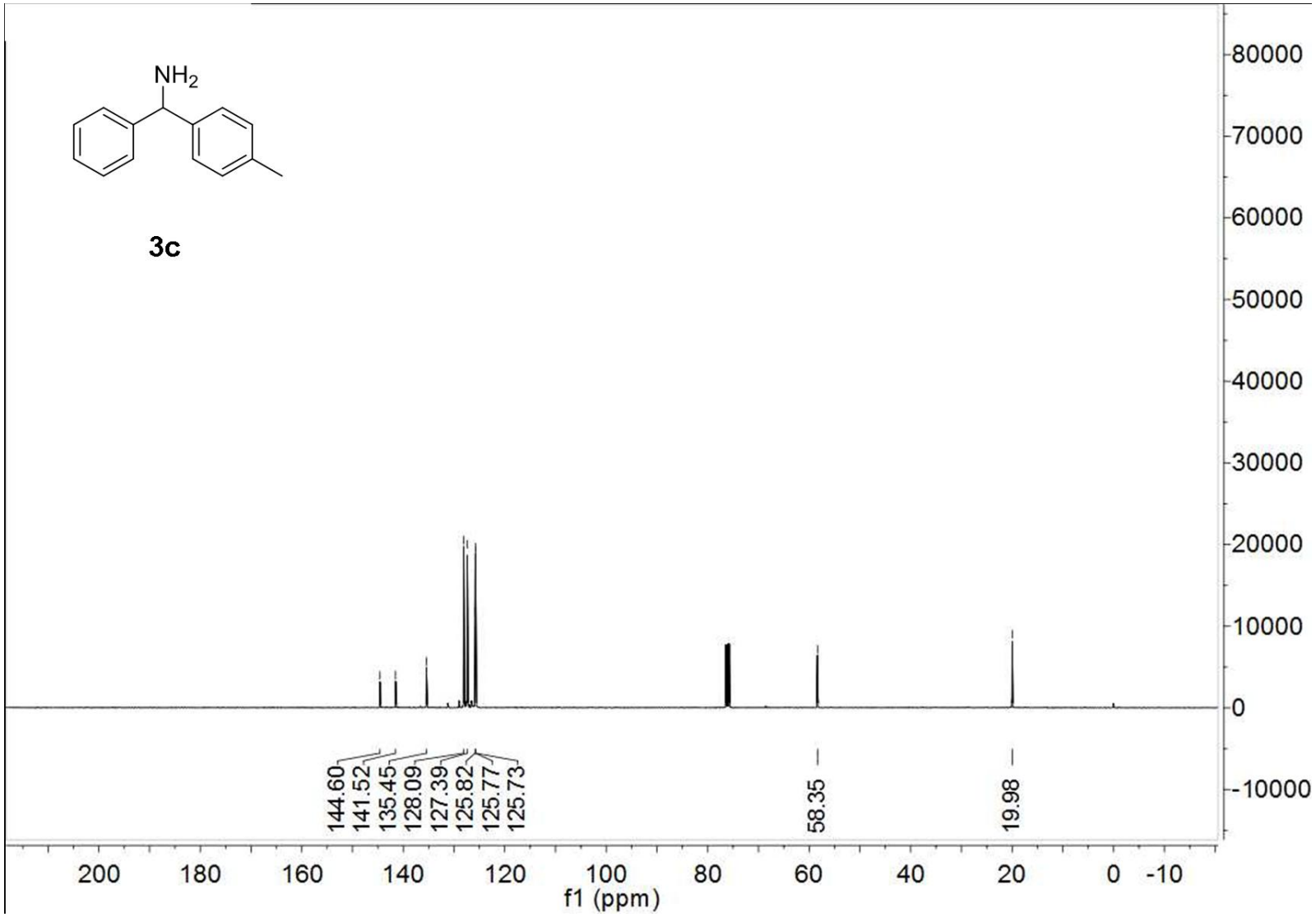

NH2
3a
7.37
7.35
7.31
7.29
7.27
7.24
7.22
7.21
7.19
5.18
1.83
4.00
4.12
2.28
0.92
2.08
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

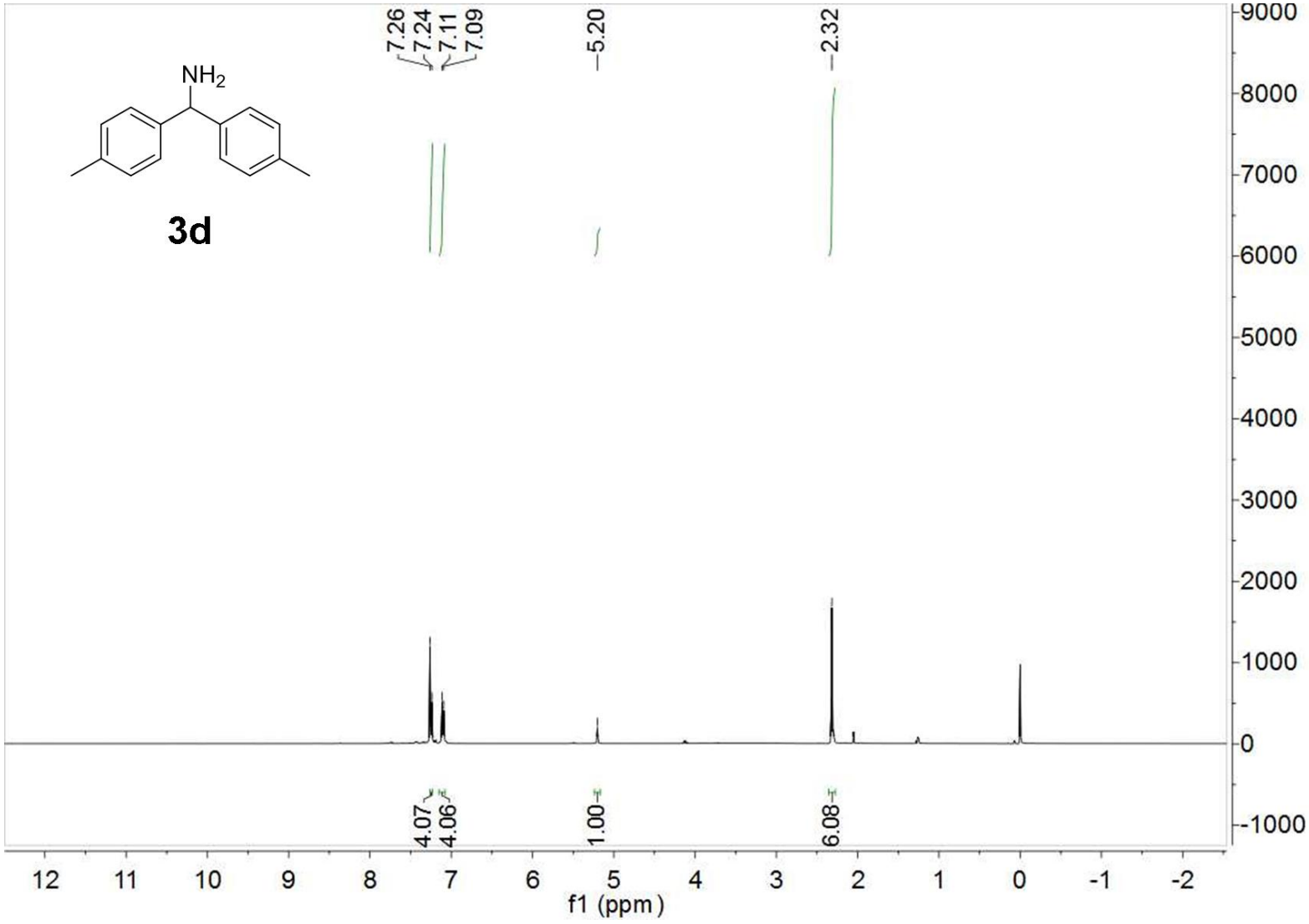

NH2
3a
145.61
128.53
127.00
126.94
59.78
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

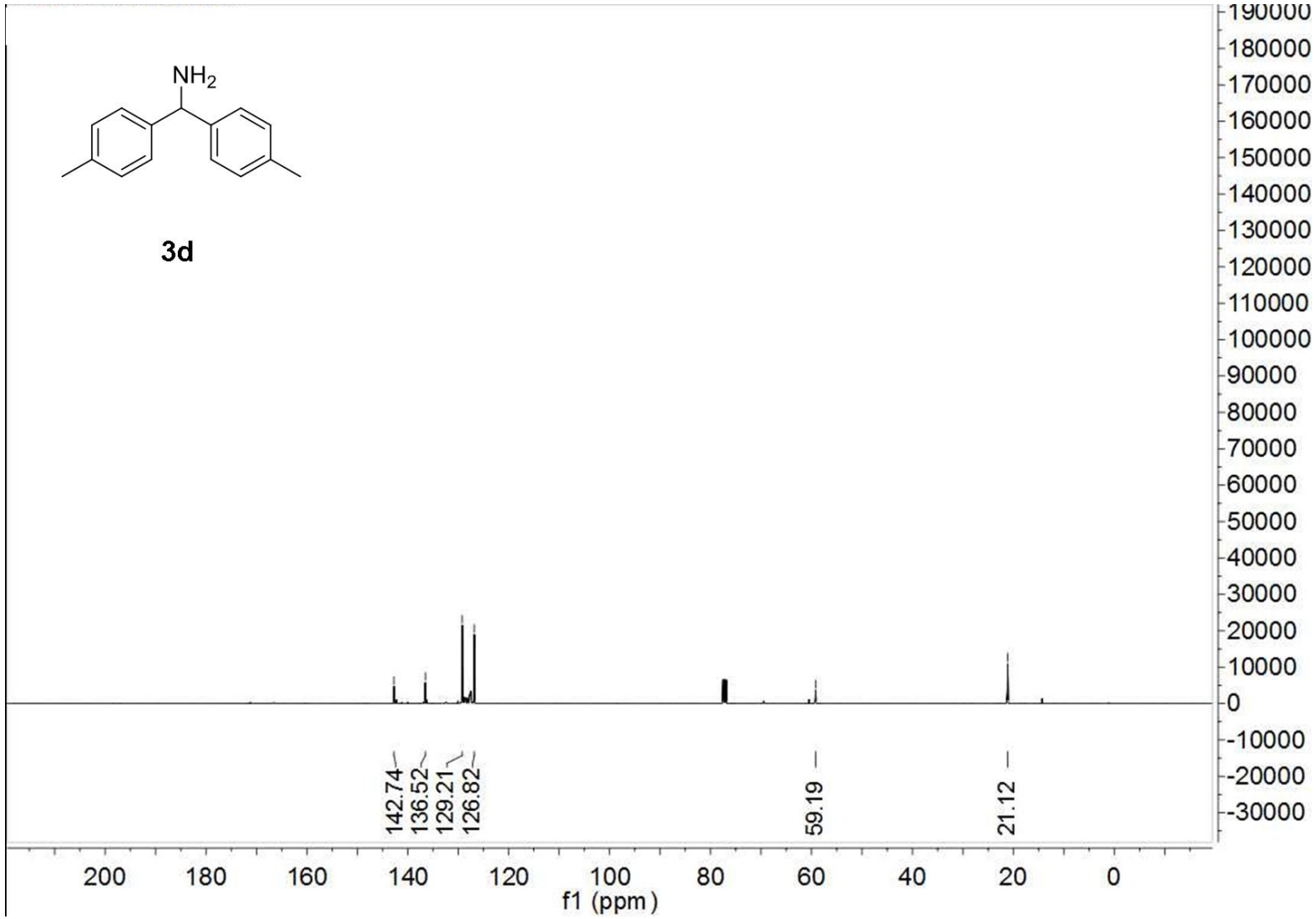

7.38
7.37
7.33
7.31
7.29
7.25
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.15
7.05
7.04
5.19
2.52
2.32
NH2
3b
2.06
2.20
3.99
1.00
1.00
2.04
3.00
f1 (ppm)

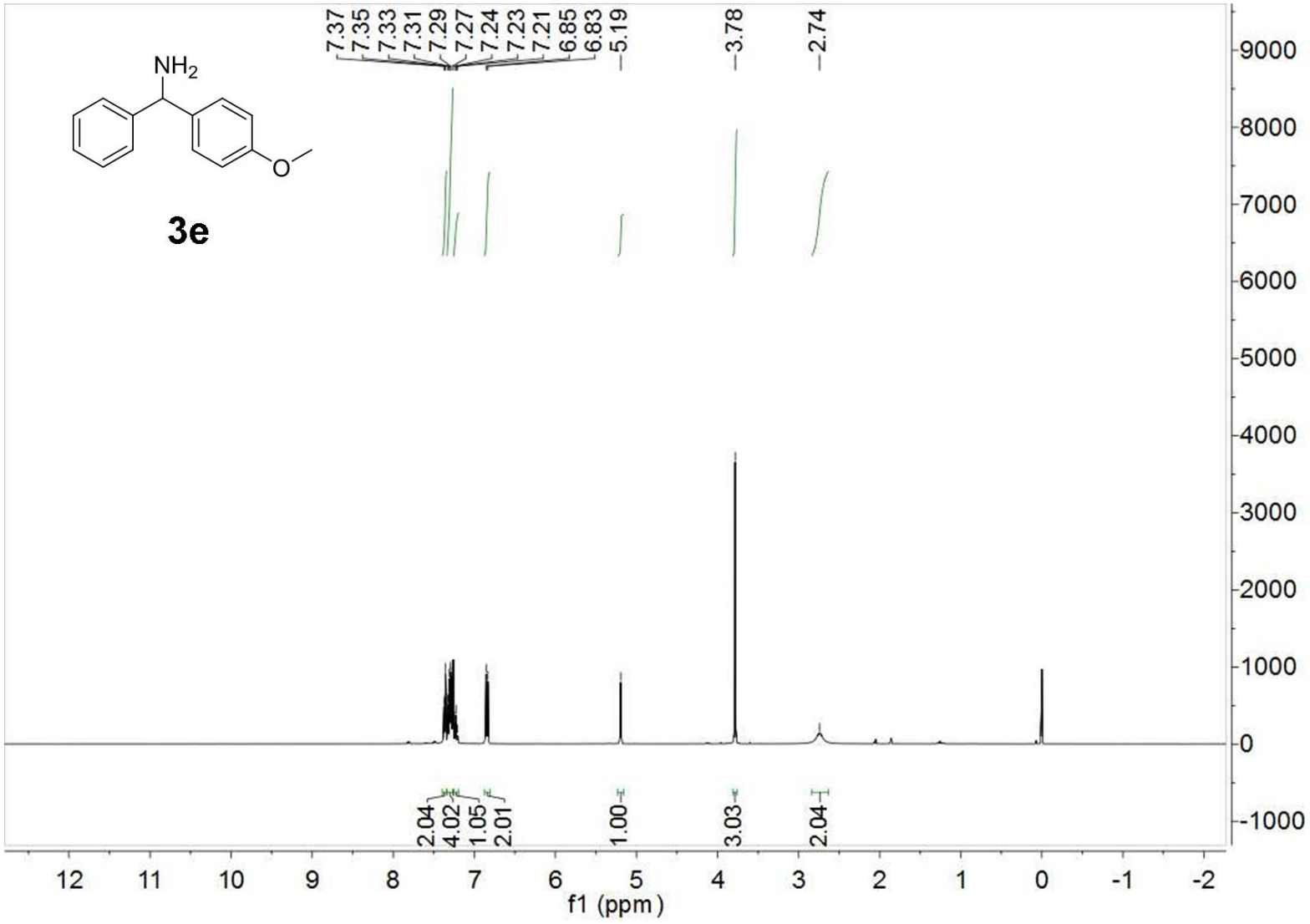

NH2
3b
144.30
144.21
136.99
127.39
127.32
126.69
126.52
125.86
125.82
122.89
58.56
20.41
f1 (ppm)

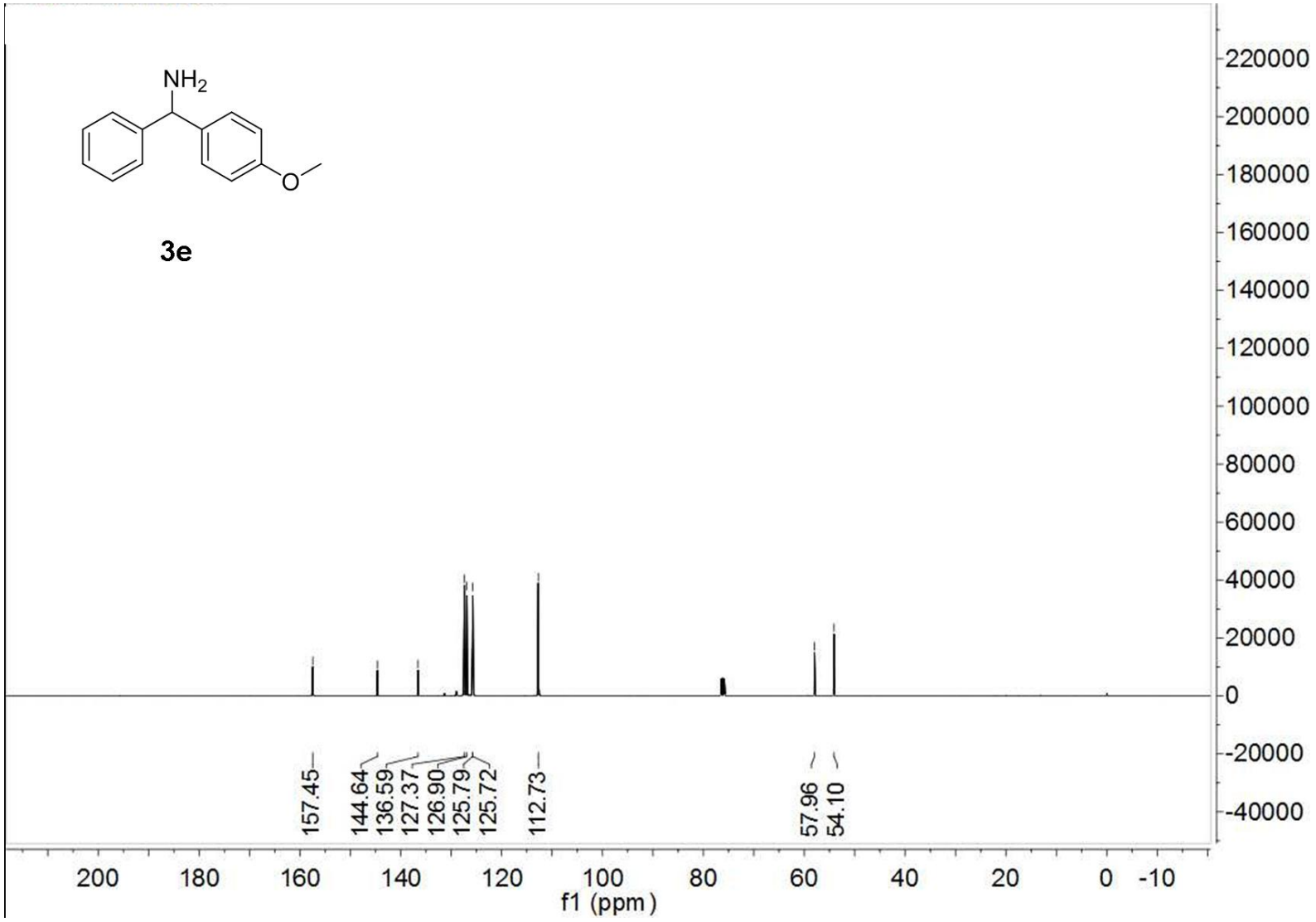

NH2
3c
7.38
7.36
7.32
7.30
7.28
7.27
7.25
7.22
7.13
7.11
5.20
2.74
2.32
1.99
2.12
3.67
2.01
0.97
1.93
3.00
f1 (ppm)

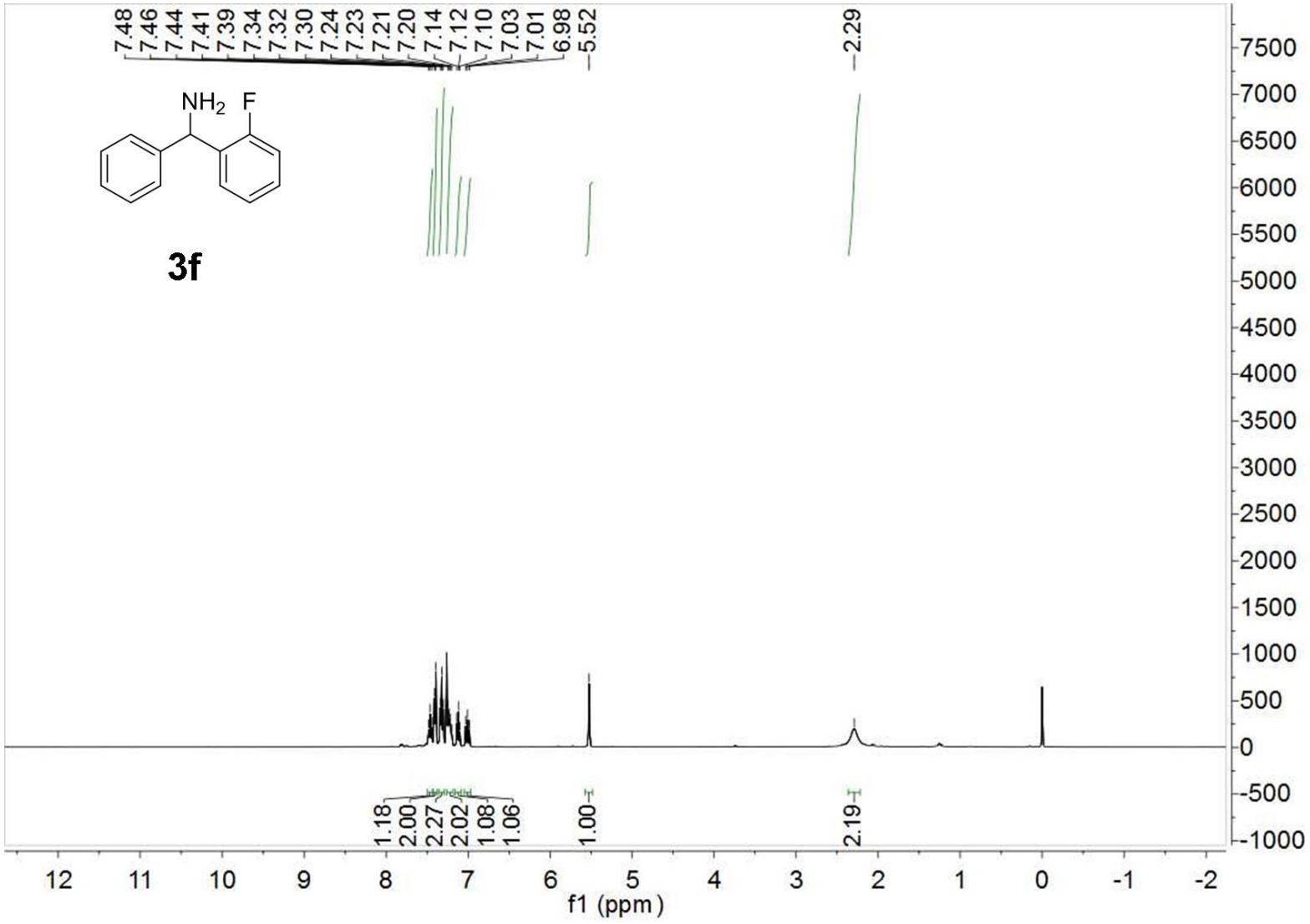

NH2
3c
144.60
141.52
135.45
128.09
127.39
125.82
125.77
125.73
58.35
19.98
f1 (ppm)

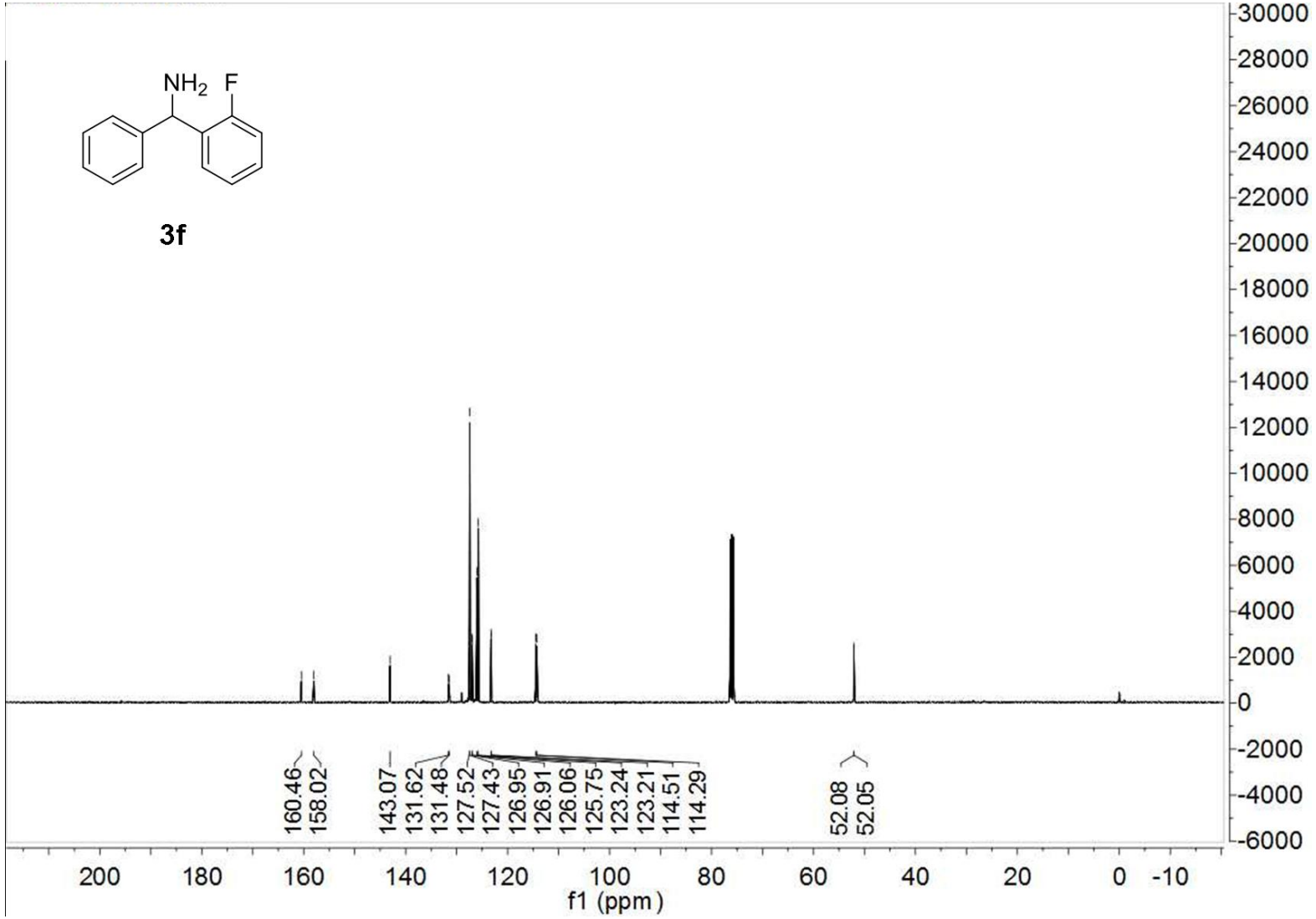

NH2
3d
7.26
7.24
7.11
7.09
5.20
2.32
4.07
4.06
1.00
6.08
f1 (ppm)

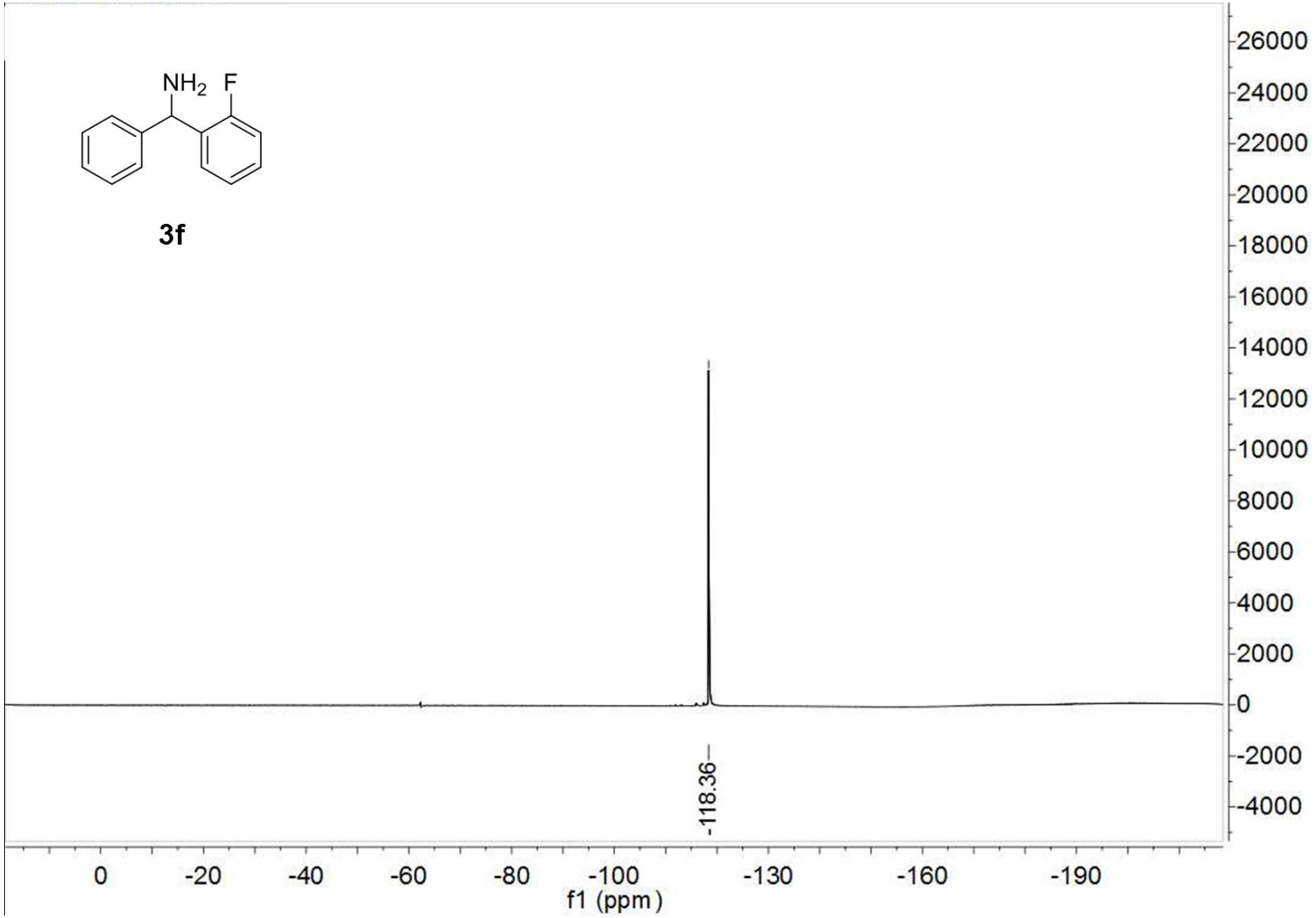

NH2
3d
142.74
136.52
129.21
126.82
59.19
21.12
f1 (ppm)

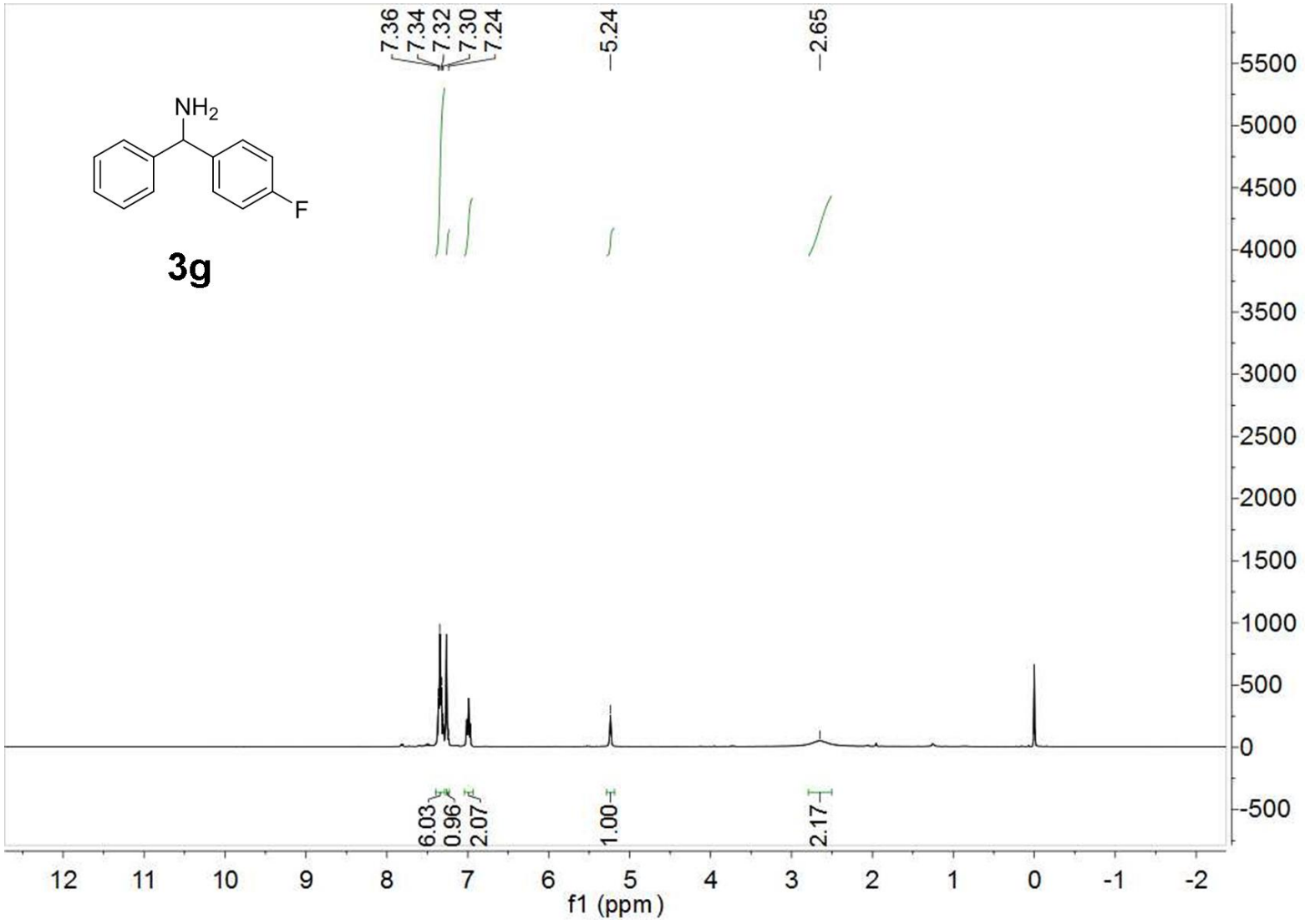

NH2
O
3e
7.37
7.35
7.33
7.31
7.29
7.27
7.24
7.23
7.21
6.85
6.83
5.19
3.78
2.74
2.04
4.02
1.05
2.01
1.00
3.03
2.04
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

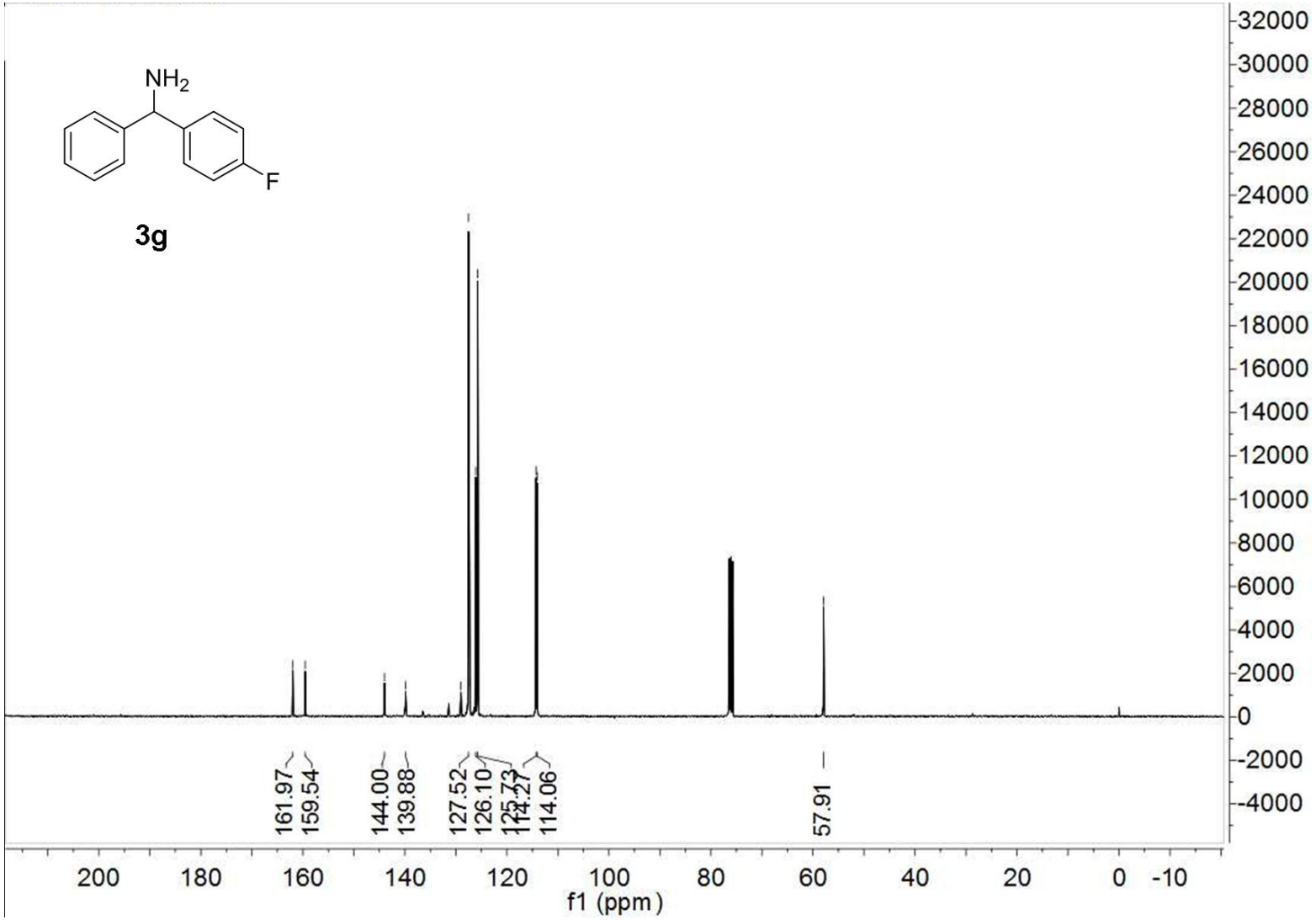

NH2
O
3e
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
157.45
144.64
136.59
127.37
126.90
125.79
125.72
112.73
57.96
54.10
200
180
160
140
120
100
80
60
40
20
0
-10
f1 (ppm)

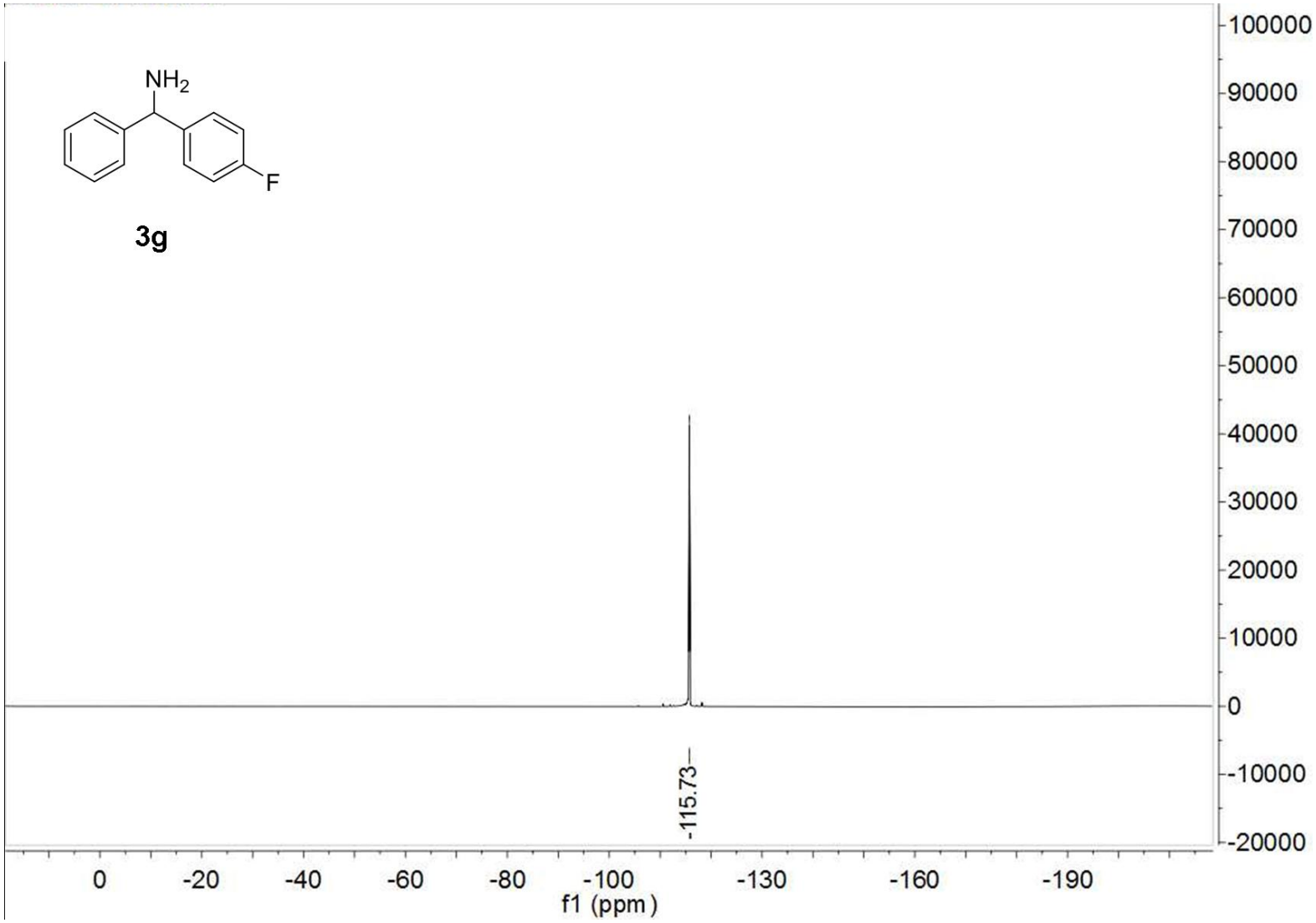

7.48
7.46
7.44
7.41
7.39
7.34
7.32
7.30
7.24
7.23
7.21
7.20
7.14
7.12
7.10
7.03
7.01
6.98
5.52
2.29
NH2
F
3f
1.18
2.00
2.27
2.02
1.08
1.06
1.00
2.19
f1 (ppm)

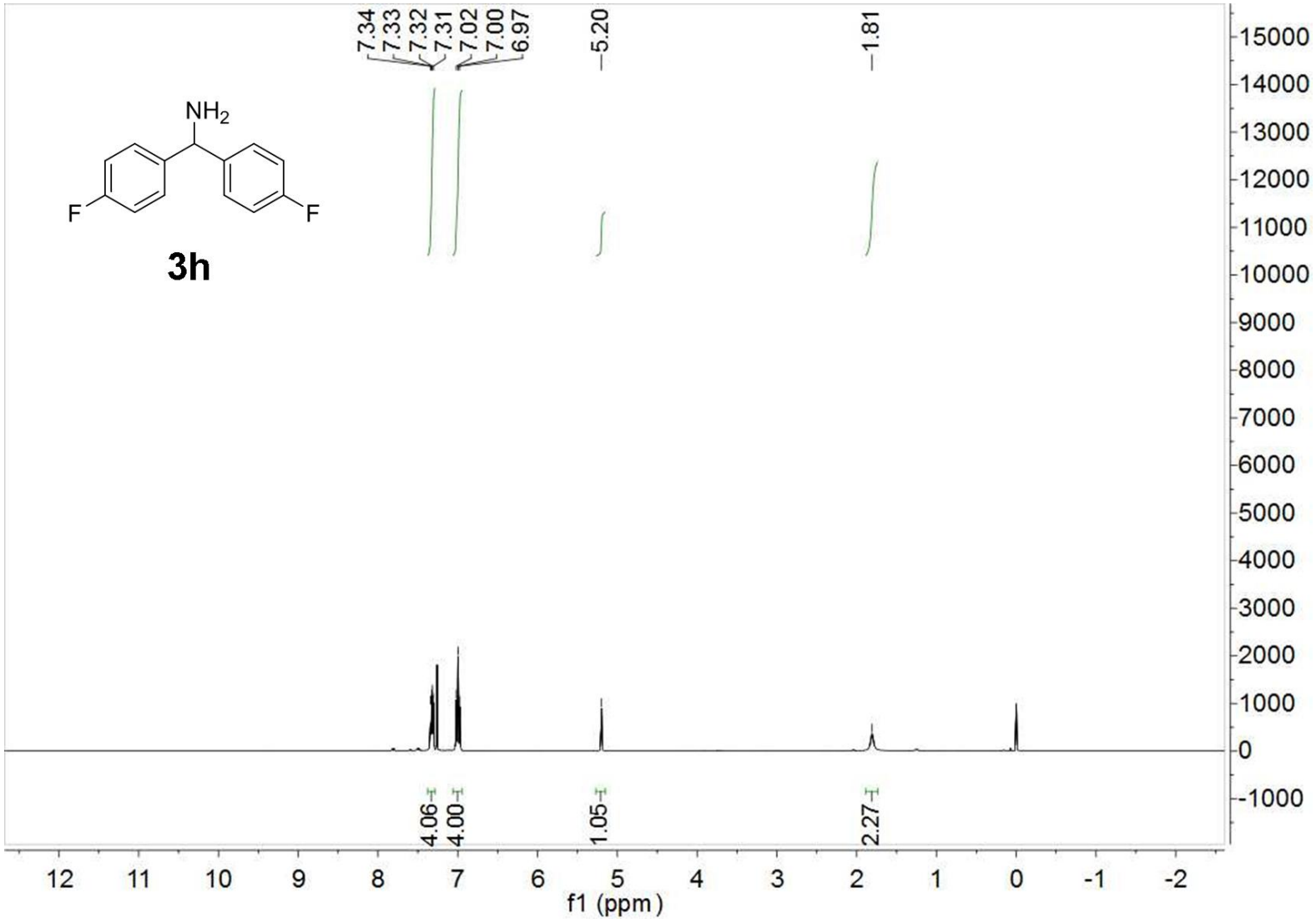

NH2 F
3f
160.46
158.02
143.07
131.62
131.48
127.52
127.43
126.95
126.91
126.06
125.75
123.24
123.21
114.51
114.29
52.08
52.05
f1 (ppm)

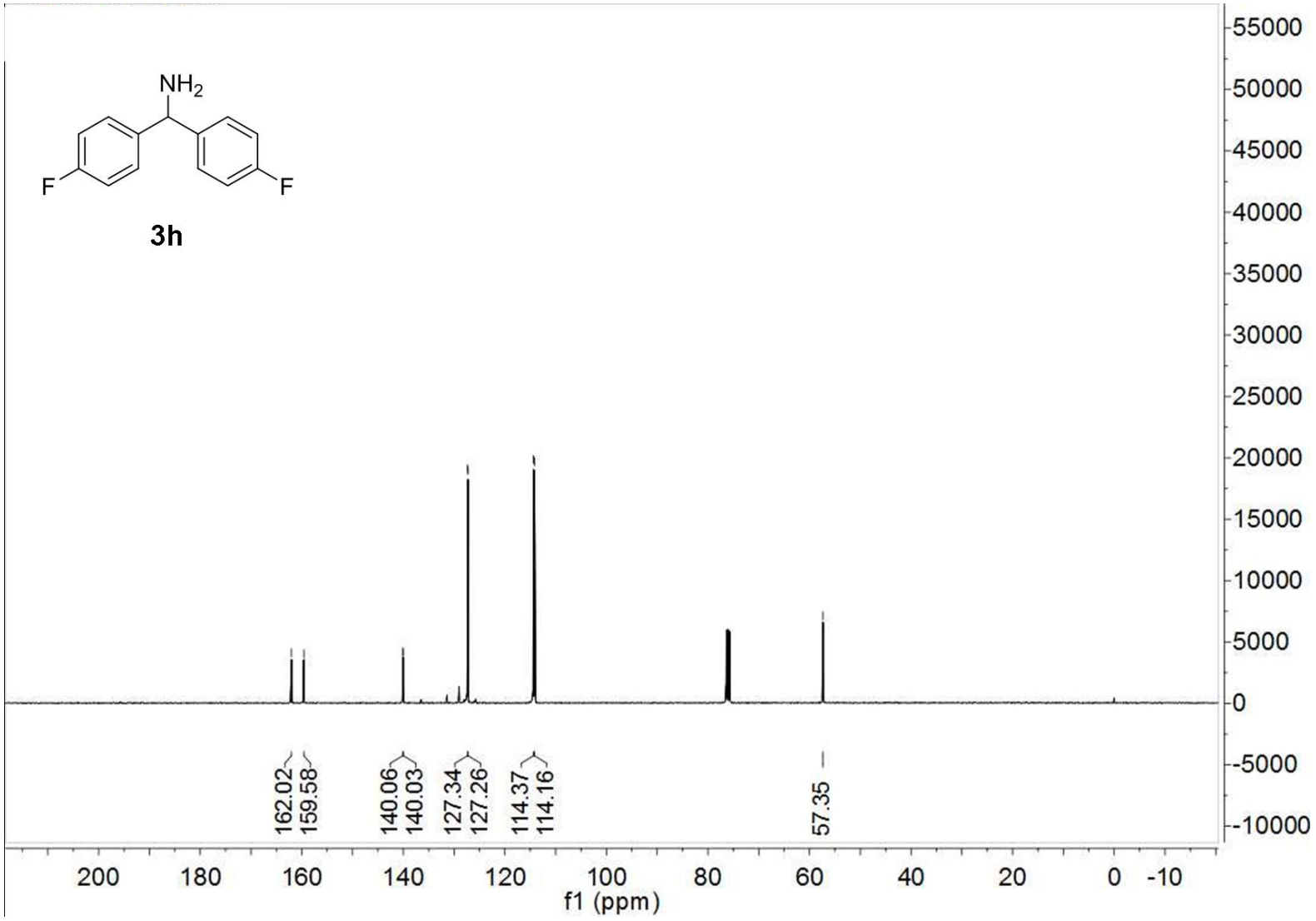

NH2 F
3f
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-118.36
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

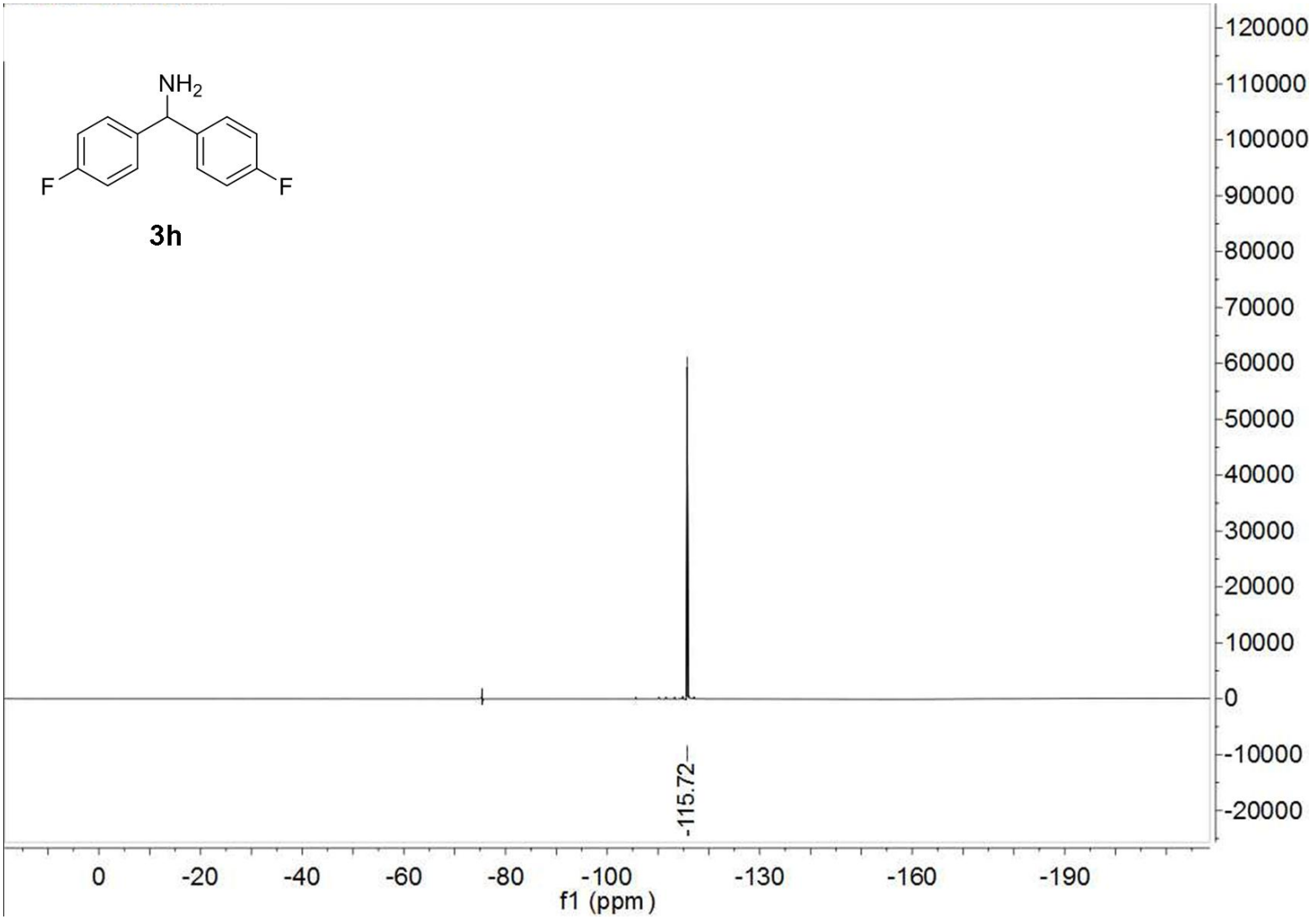

7.36
7.34
7.32
7.30
7.24
5.24
2.65
NH2
F
3g
6.03
0.96
2.07
1.00
2.17
f1 (ppm)

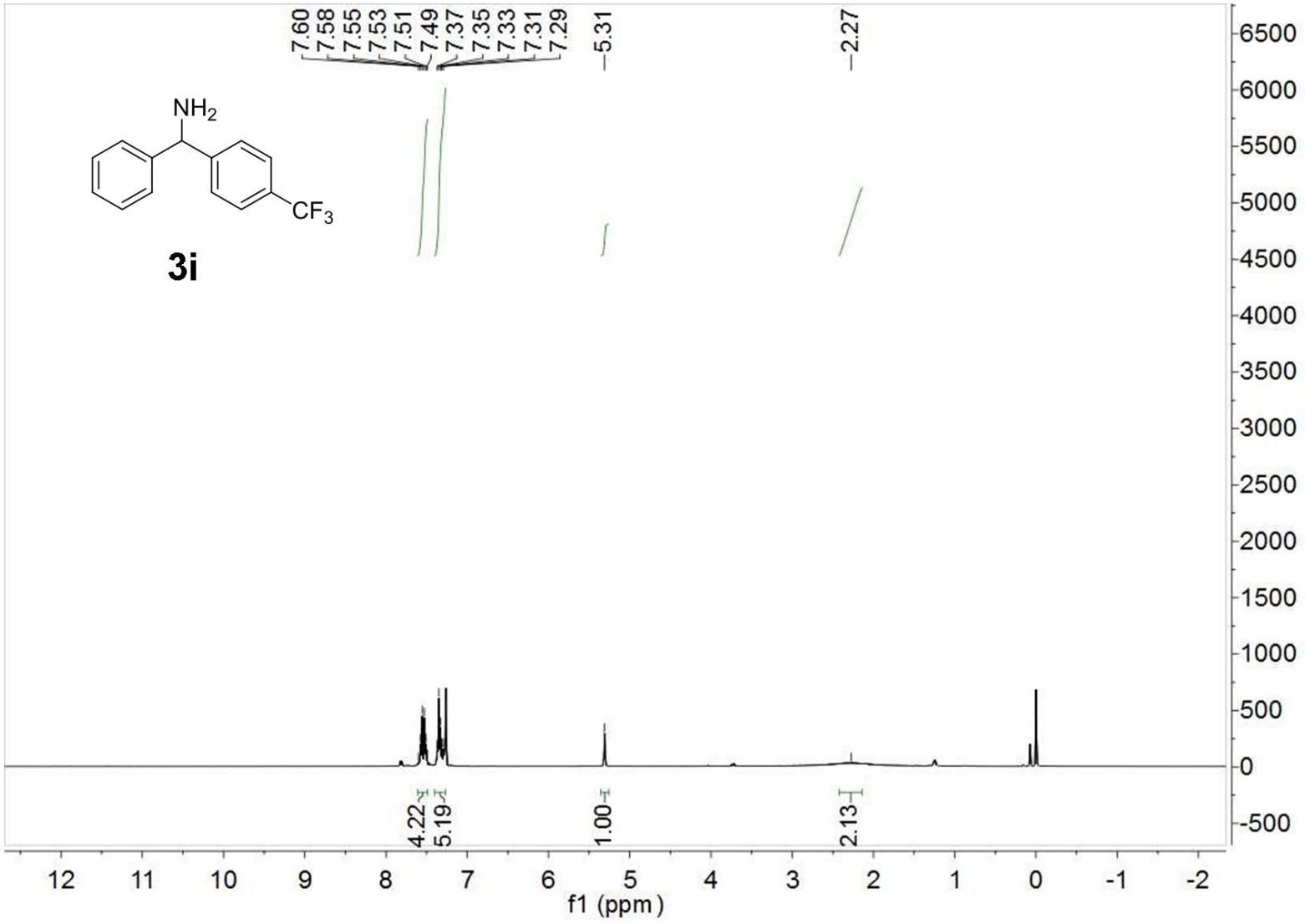

NH2
F
3g
161.97
159.54
144.00
139.88
127.52
126.10
125.73
114.27
114.06
57.91
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
200
180
160
140
120
100
80
60
40
20
0
-10
f1 (ppm)

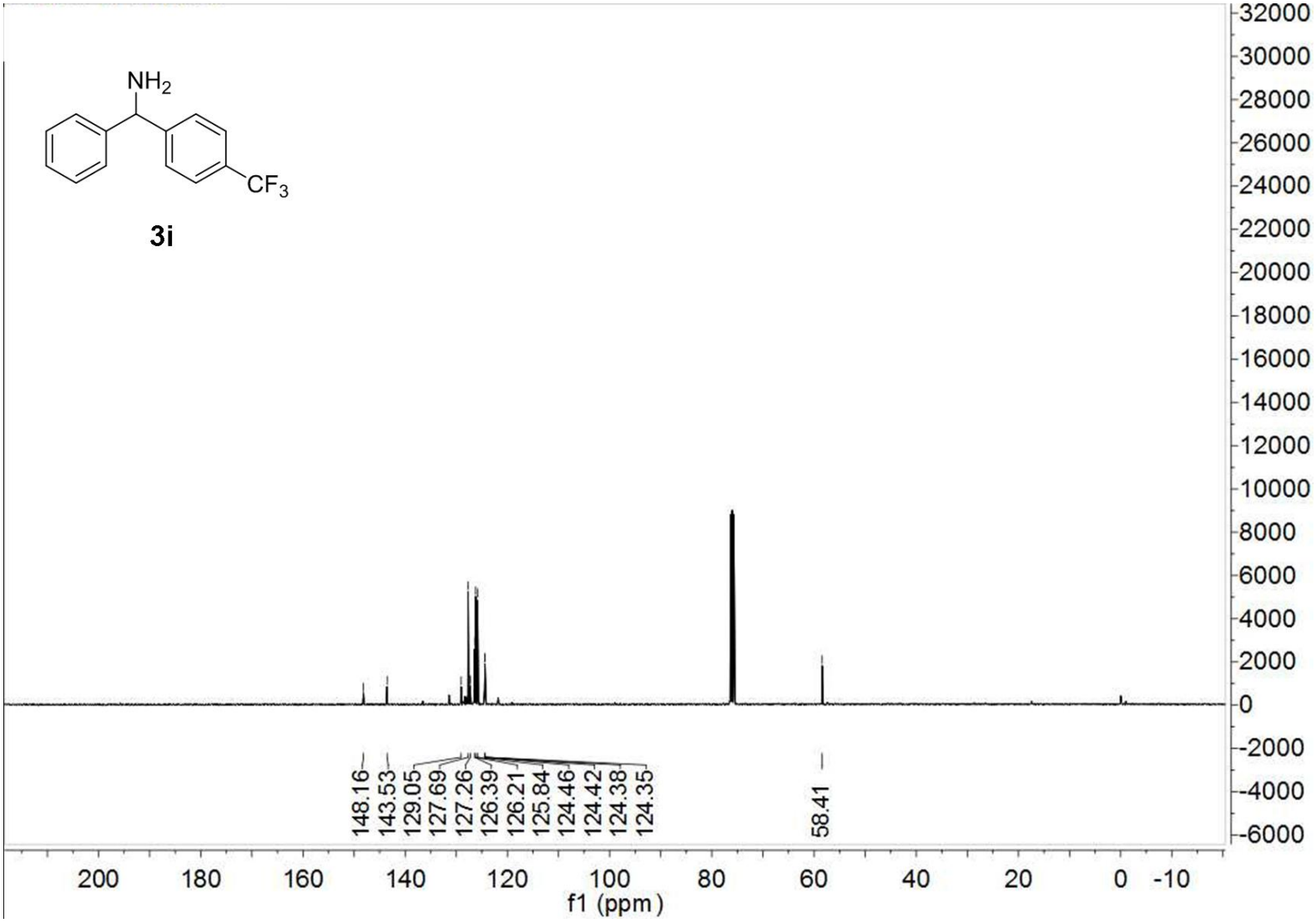

NH2
F
3g
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-115.73
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

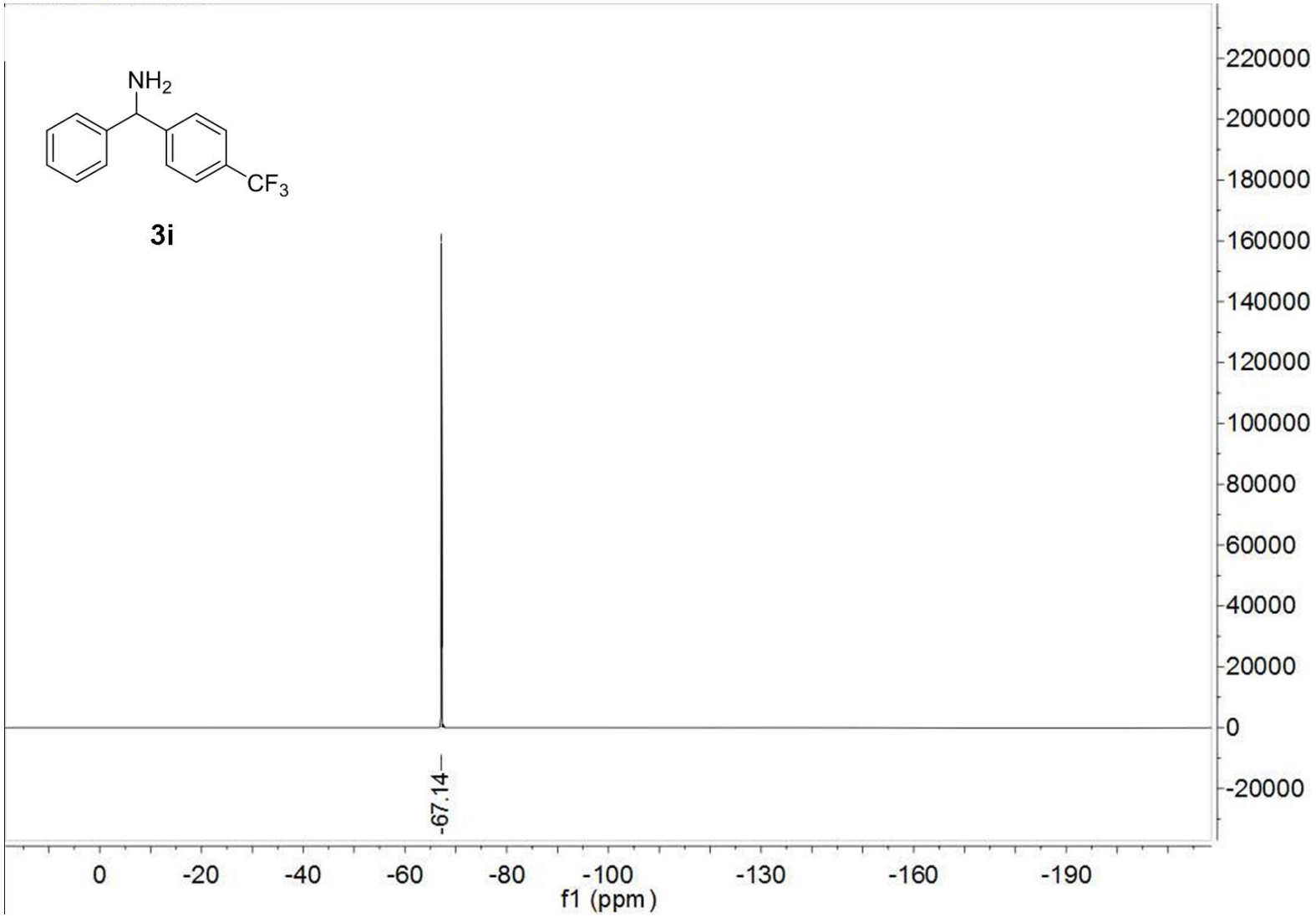

7.34
7.33
7.32
7.31
7.02
7.00
6.97
5.20
1.81
NH2
F
F
3h
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
4.06
4.00
1.05
2.27
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

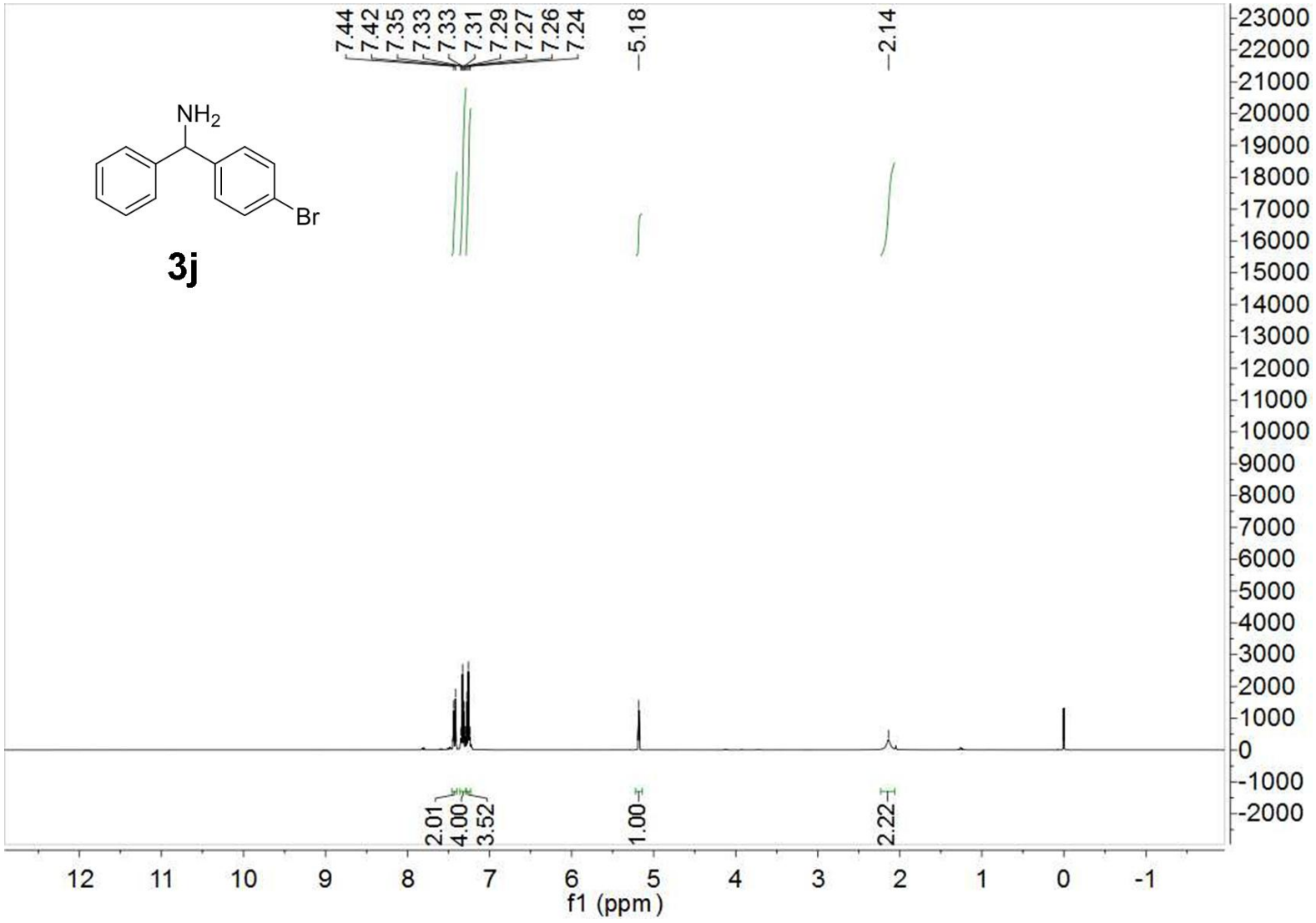

NH2
F
F
3h
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
162.02
159.58
140.06
140.03
127.34
127.26
114.37
114.16
57.35
200
180
160
140
120
100
80
60
40
20
0
-10
f1 (ppm)

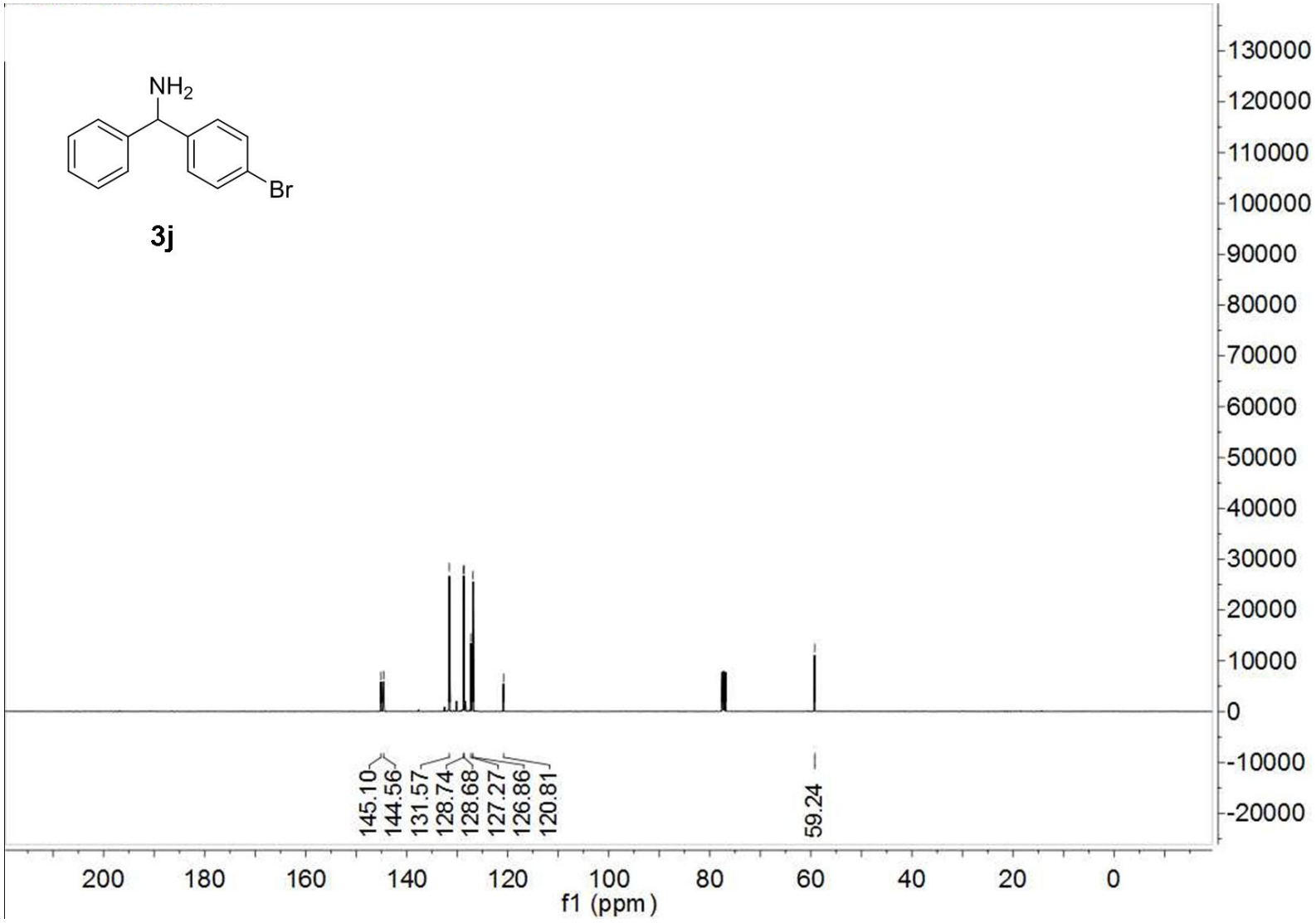

NH2
F
F
3h
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
-115.72
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

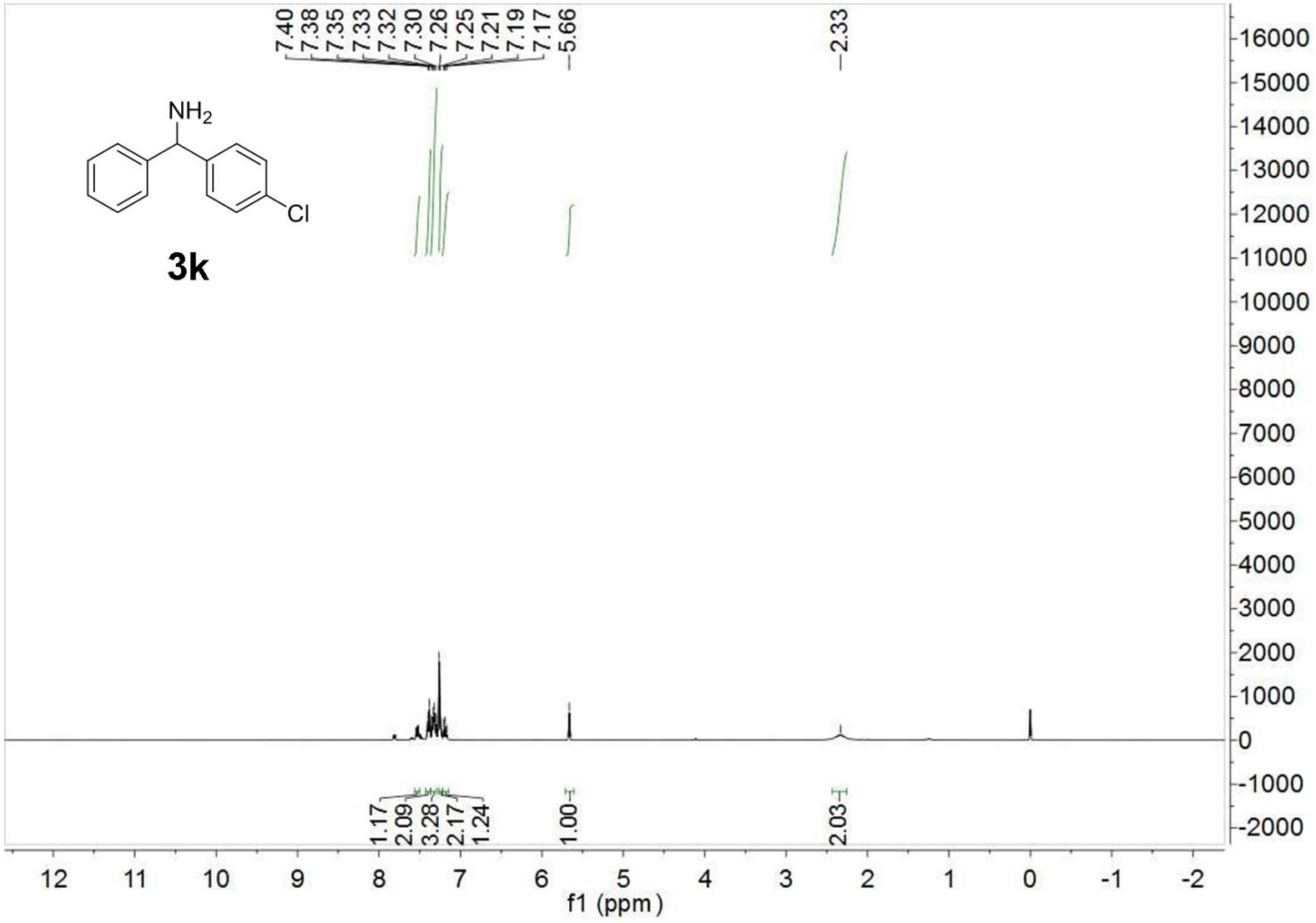

7.60
7.58
7.55
7.53
7.51
7.49
7.37
7.35
7.33
7.31
7.29
5.31
2.27
NH2
CF3
3i
4.22
5.19
1.00
2.13
f1 (ppm)

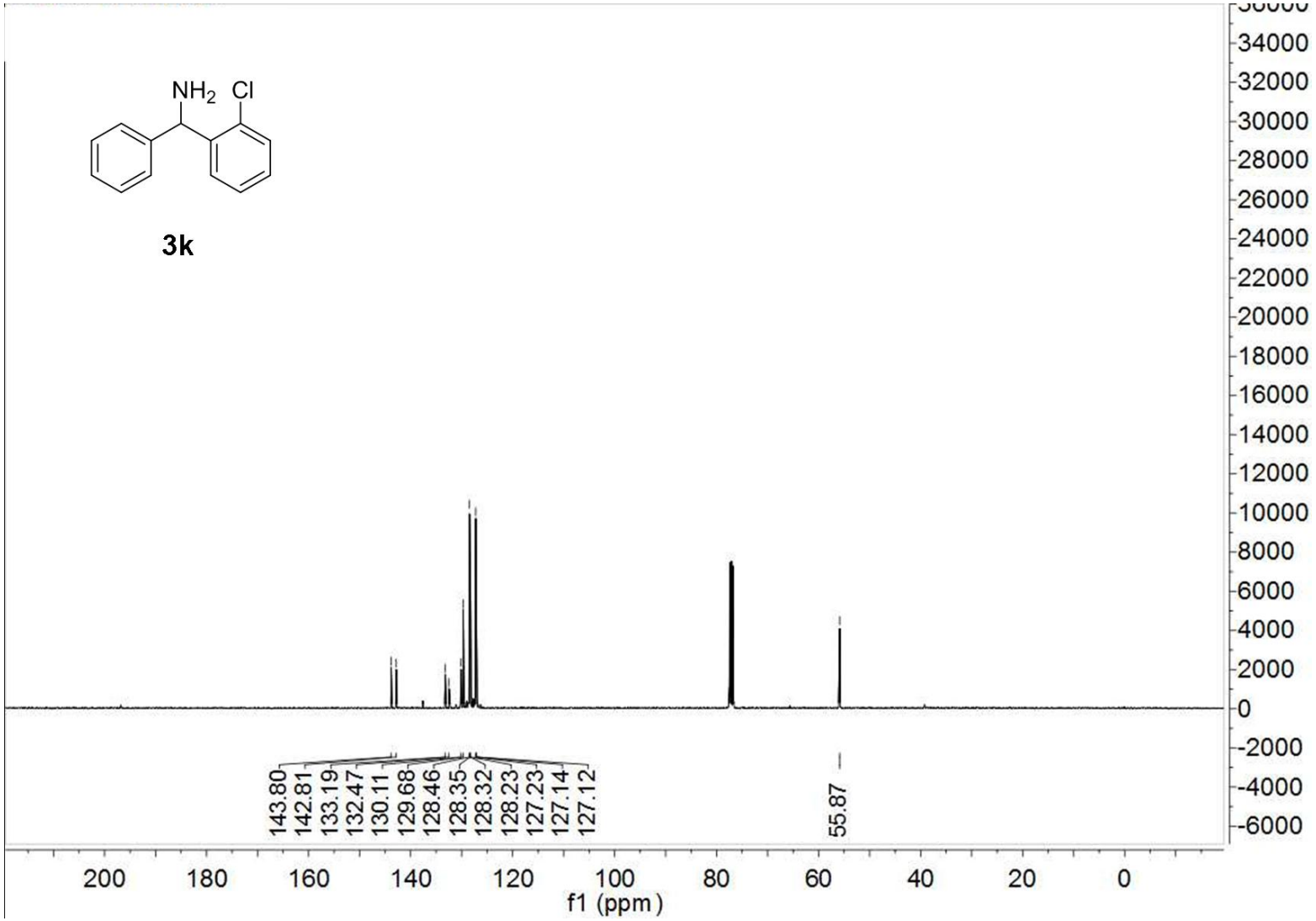

NH2
CF3
3i
148.16
143.53
129.05
127.69
127.26
126.39
126.21
125.84
124.46
124.42
124.38
124.35
58.41
f1 (ppm)

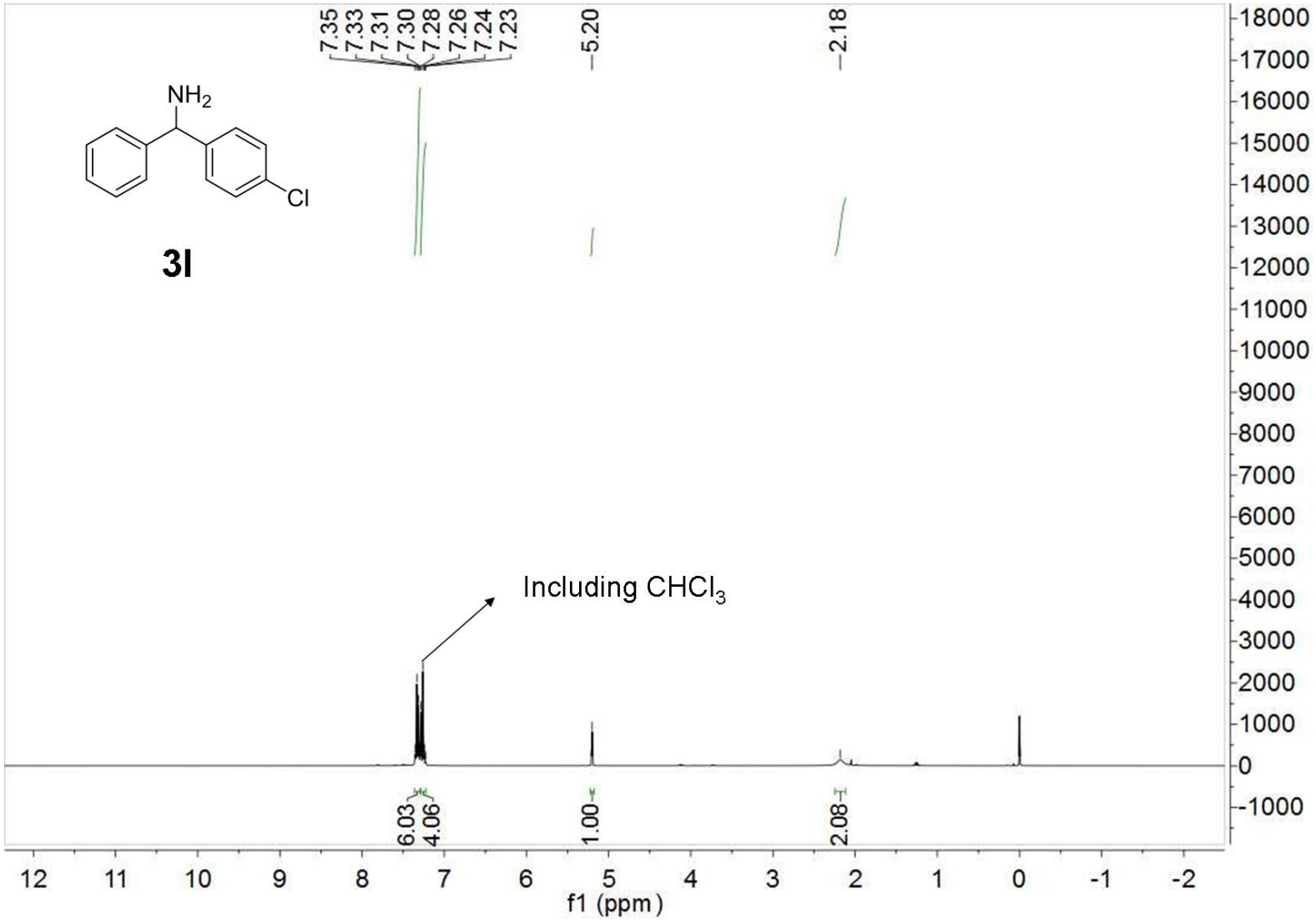

NH2
CF3
3i
-67.14
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

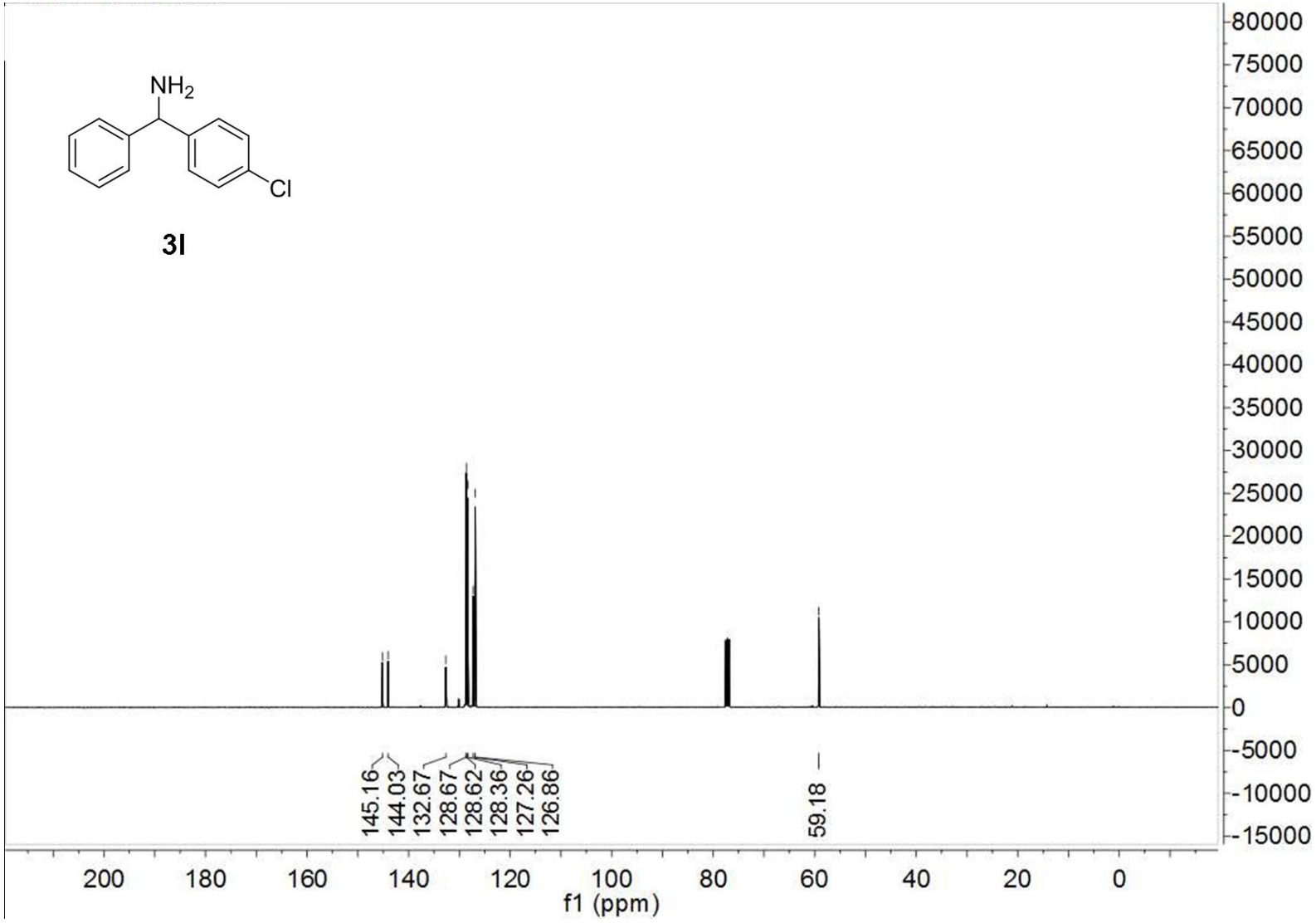

7.44
7.42
7.35
7.33
7.33
7.31
7.29
7.27
7.26
7.24
5.18
2.14
NH2
Br
3j
2.01
4.00
3.52
1.00
2.22
f1 (ppm)

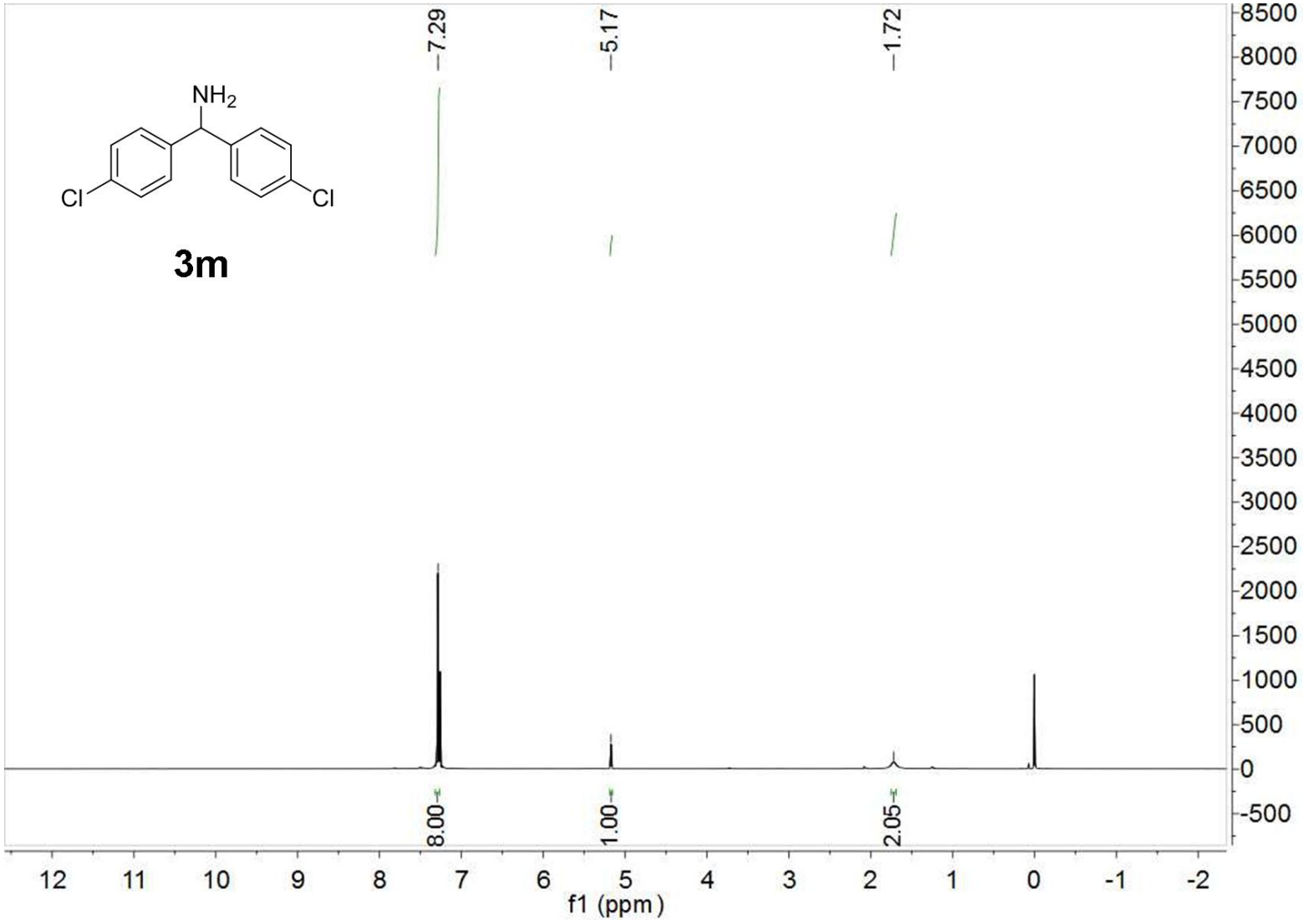

NH2
Br
3j
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
145.10
144.56
131.57
128.74
128.68
127.27
126.86
120.81
59.24
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

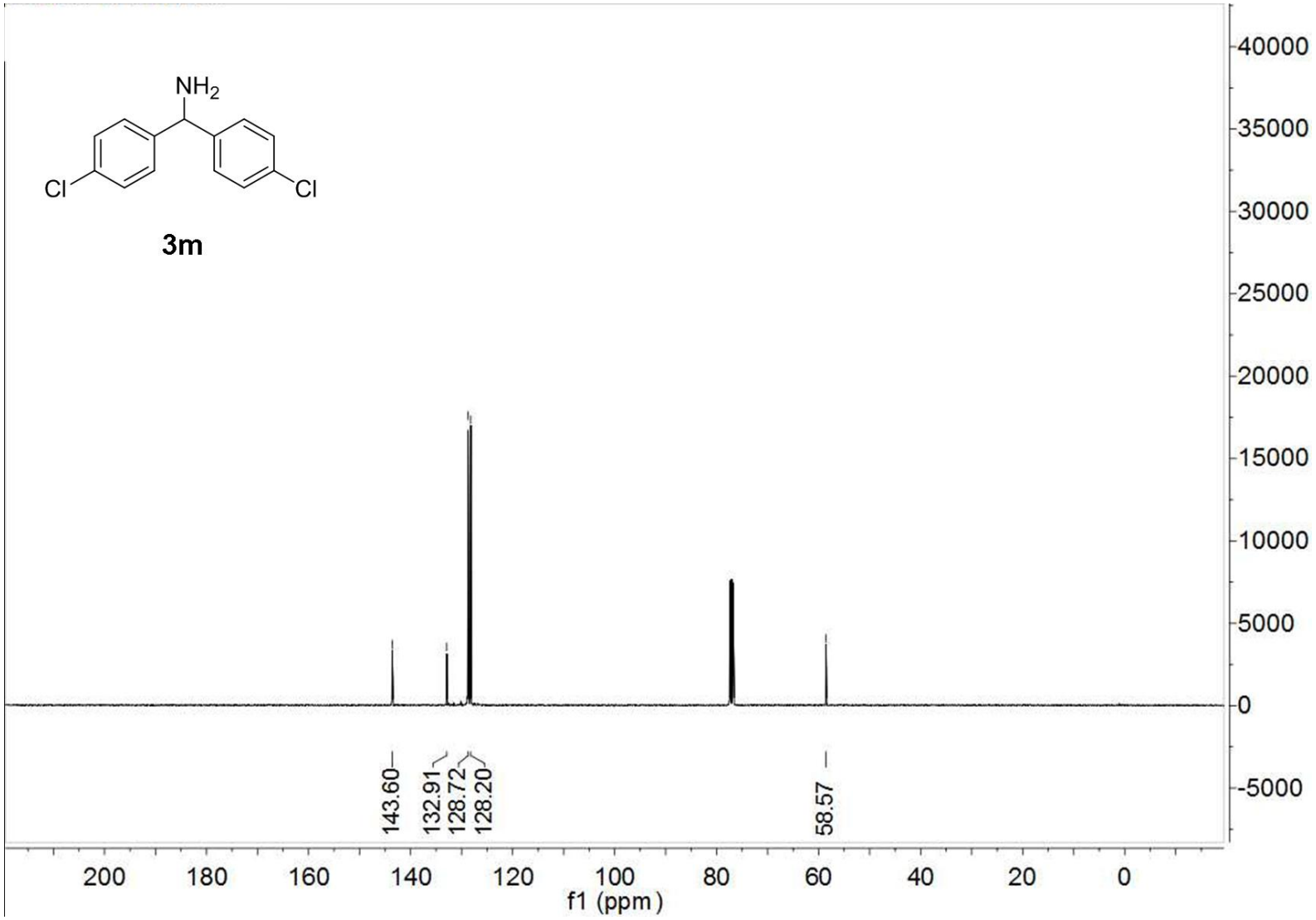

7.40
7.38
7.35
7.33
7.32
7.30
7.26
7.25
7.21
7.19
7.17
5.66
2.33
NH2
Cl
3k
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
1.17
2.09
3.28
2.17
1.24
1.00
2.03
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

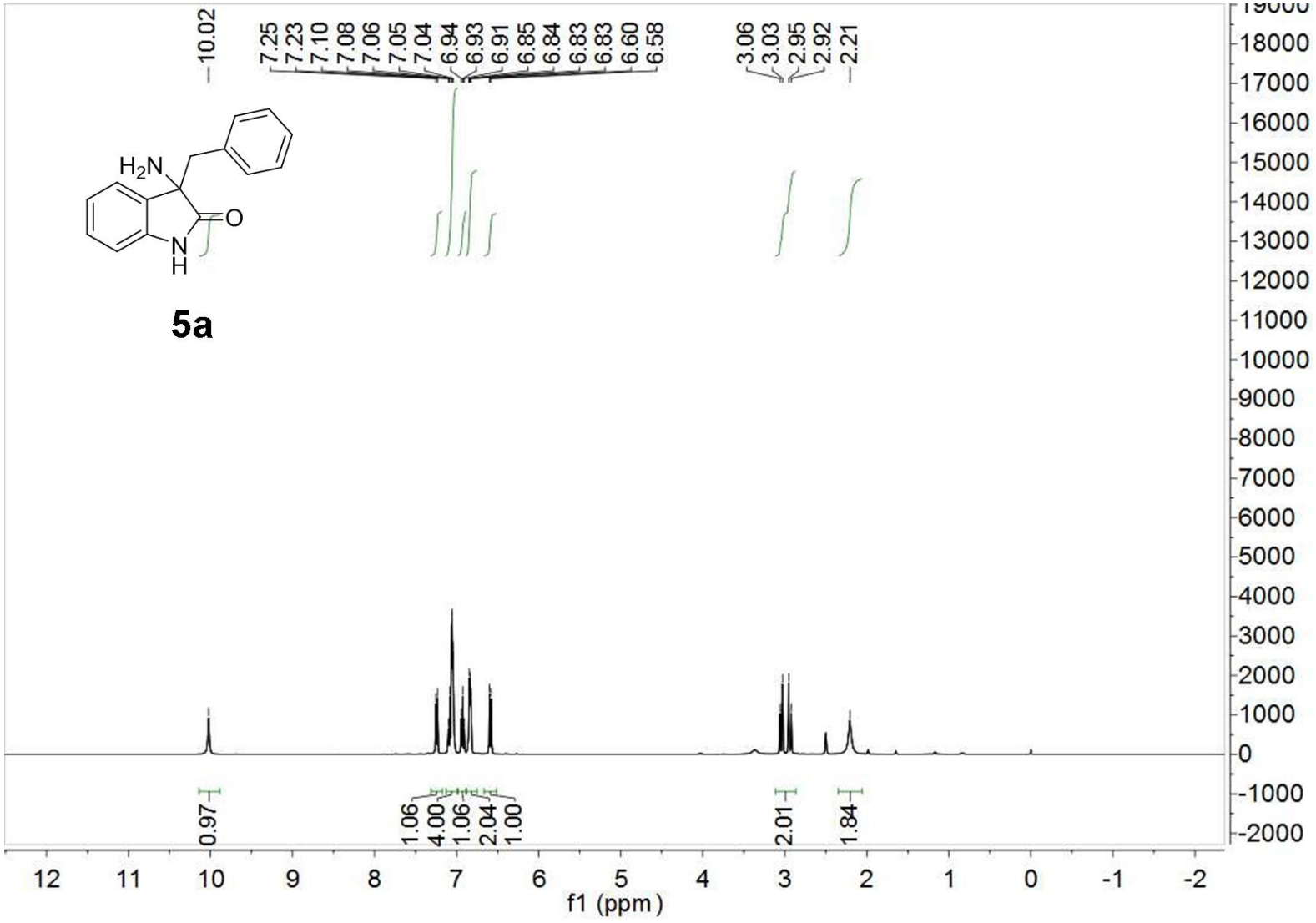

NH2 Cl
3k
143.80
142.81
133.19
132.47
130.11
129.68
128.46
128.35
128.32
128.23
127.23
127.14
127.12
55.87
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000

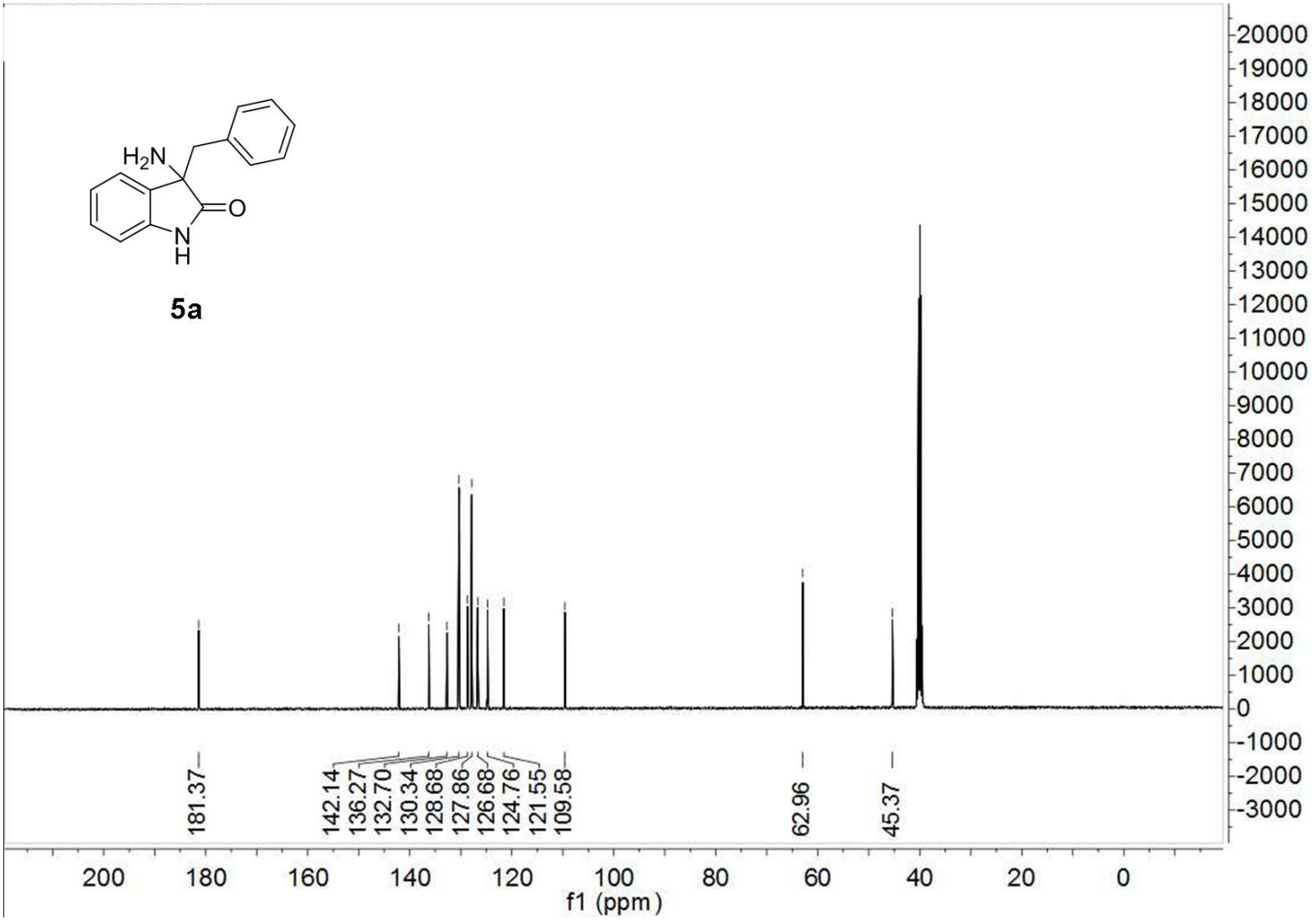

7.35
7.33
7.31
7.30
7.28
7.26
7.24
7.23
5.20
2.18
NH2
Cl
3l
Including CHCl3
6.03
4.06
1.00
2.08
f1 (ppm)

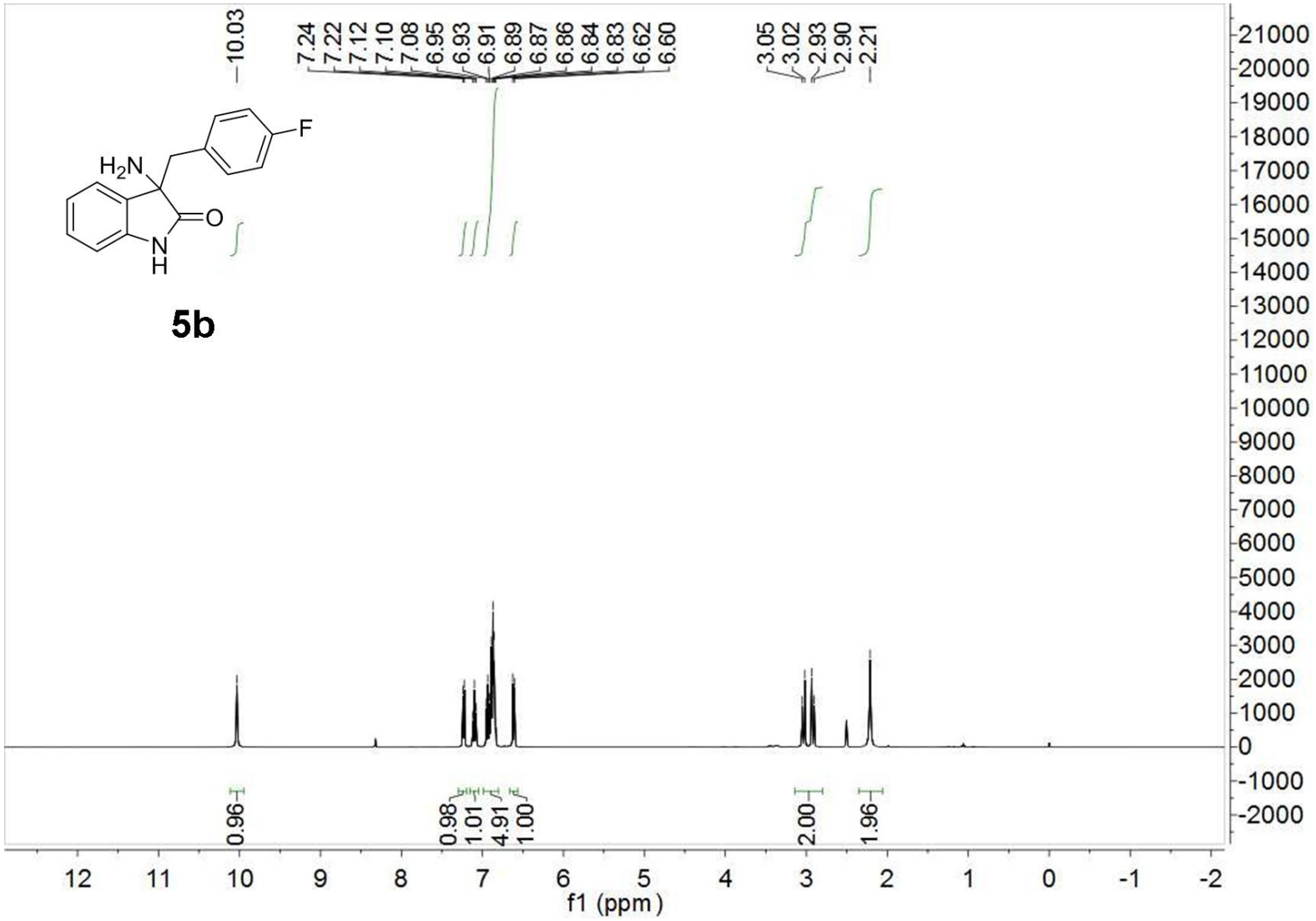

NH2
Cl
3l
145.16
144.03
132.67
128.67
128.62
128.36
127.26
126.86
59.18
f1 (ppm)

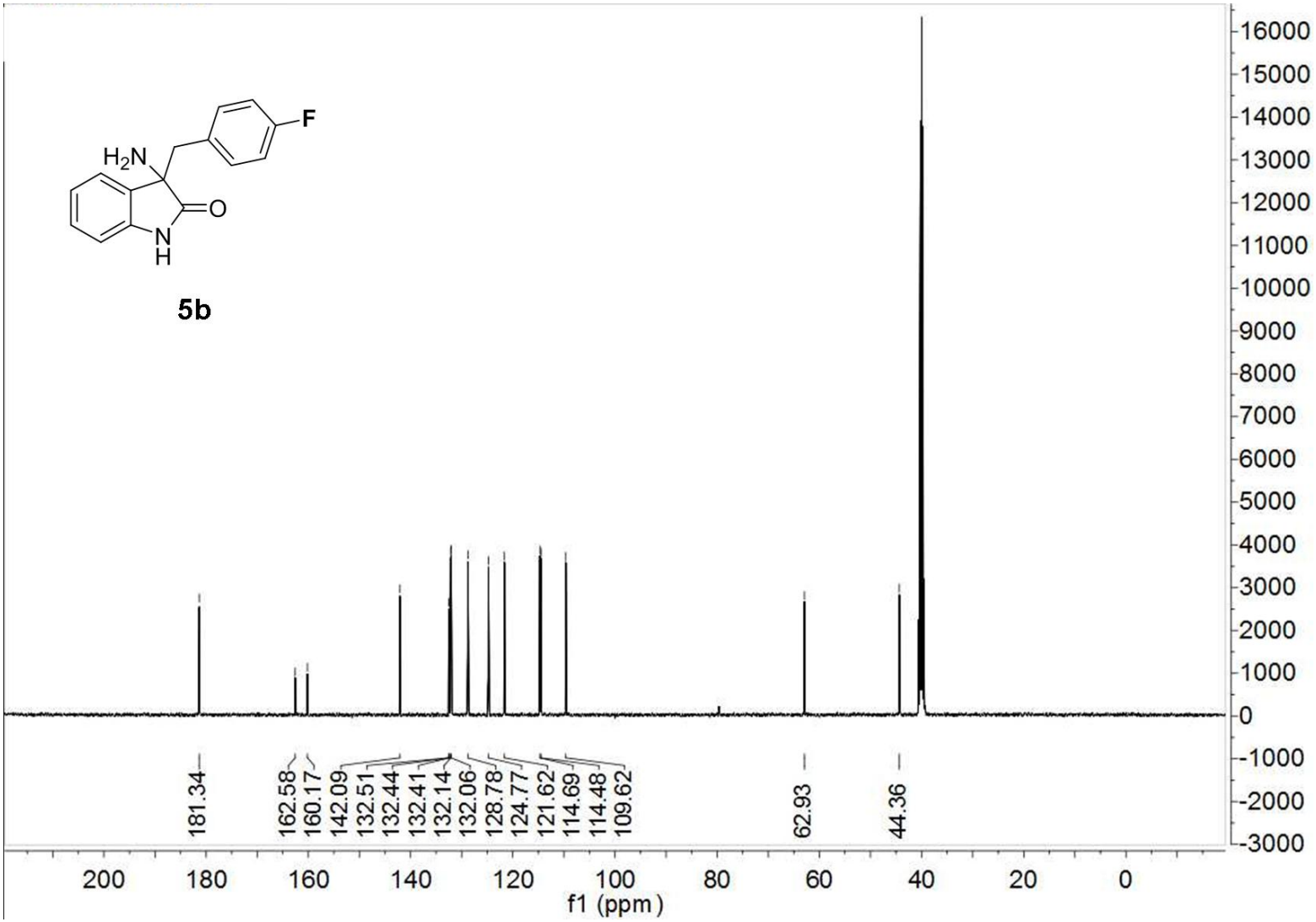

NH2
Cl
Cl
3m
7.29
5.17
1.72
8.00
1.00
2.05
f1 (ppm)

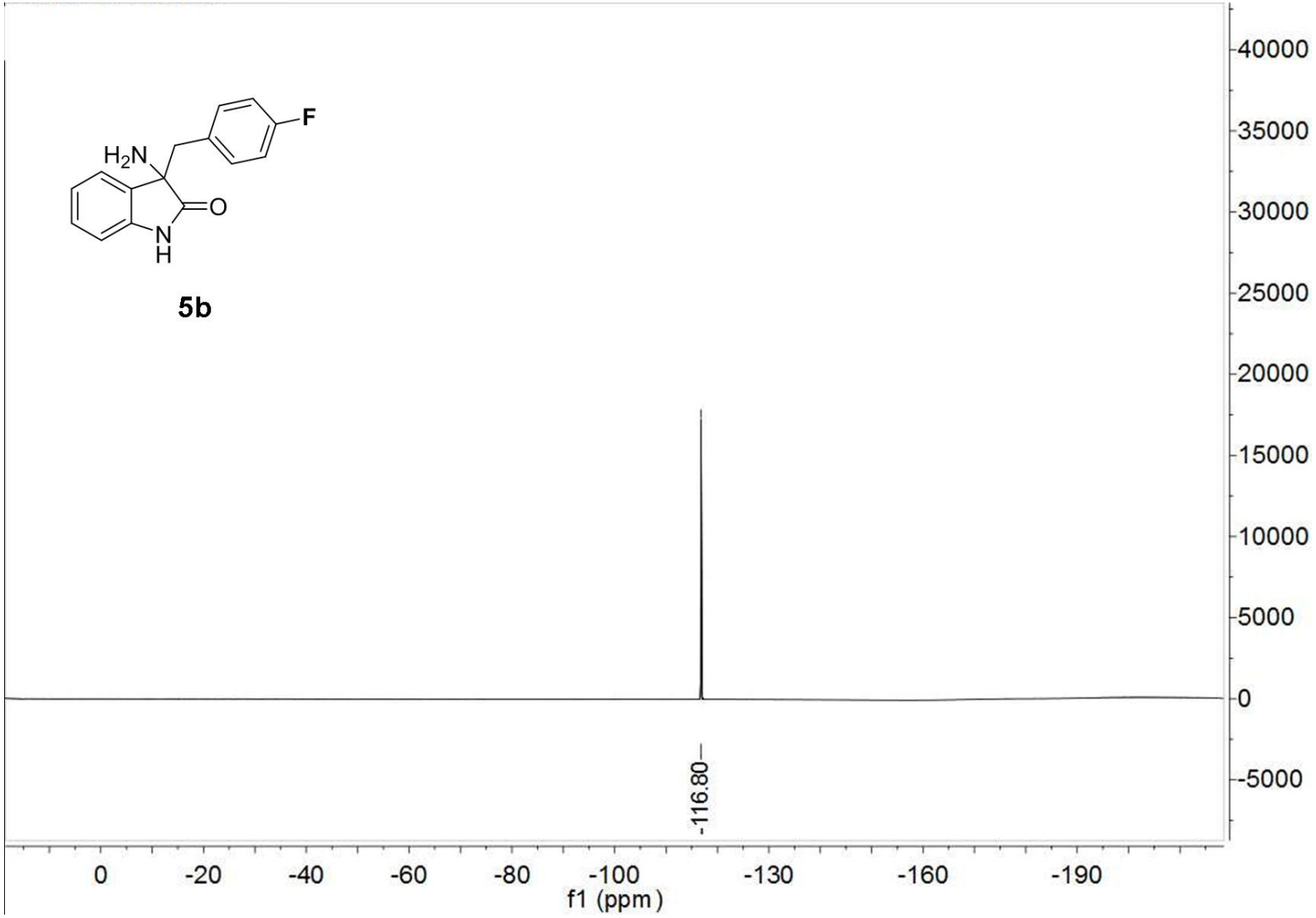

NH2
Cl
Cl
3m
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
143.60
132.91
128.72
128.20
58.57
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

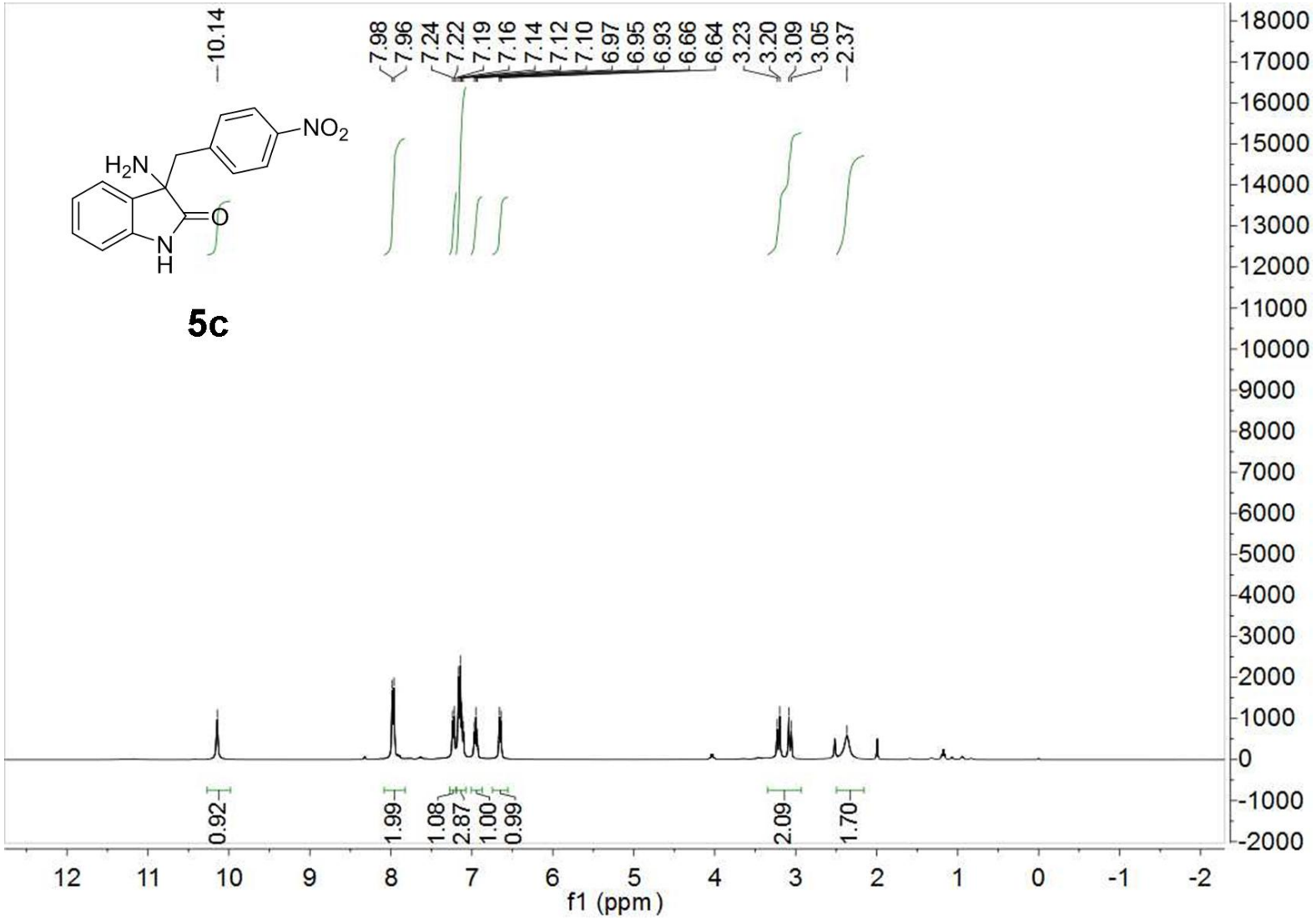

10.02
7.25
7.23
7.10
7.08
7.06
7.05
7.04
6.94
6.93
6.91
6.85
6.84
6.83
6.83
6.60
6.58
3.06
3.03
2.95
2.92
2.21
H2N
O
N
H
5a
0.97
1.06
4.00
1.06
2.04
1.00
2.01
1.84
f1 (ppm)

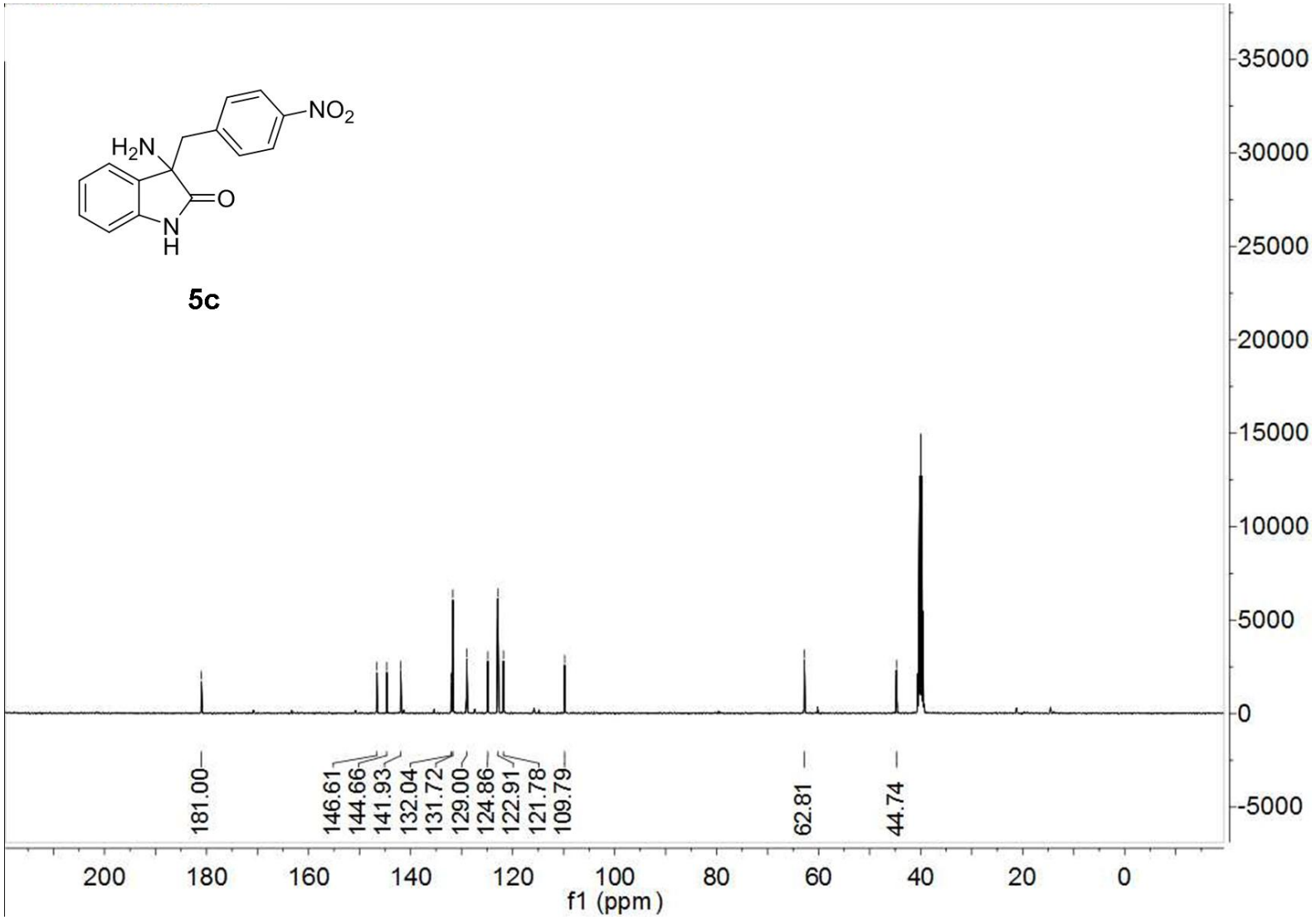

H2N
O
N
H
5a
181.37
142.14
136.27
132.70
130.34
128.68
127.86
126.68
124.76
121.55
109.58
62.96
45.37
f1 (ppm)

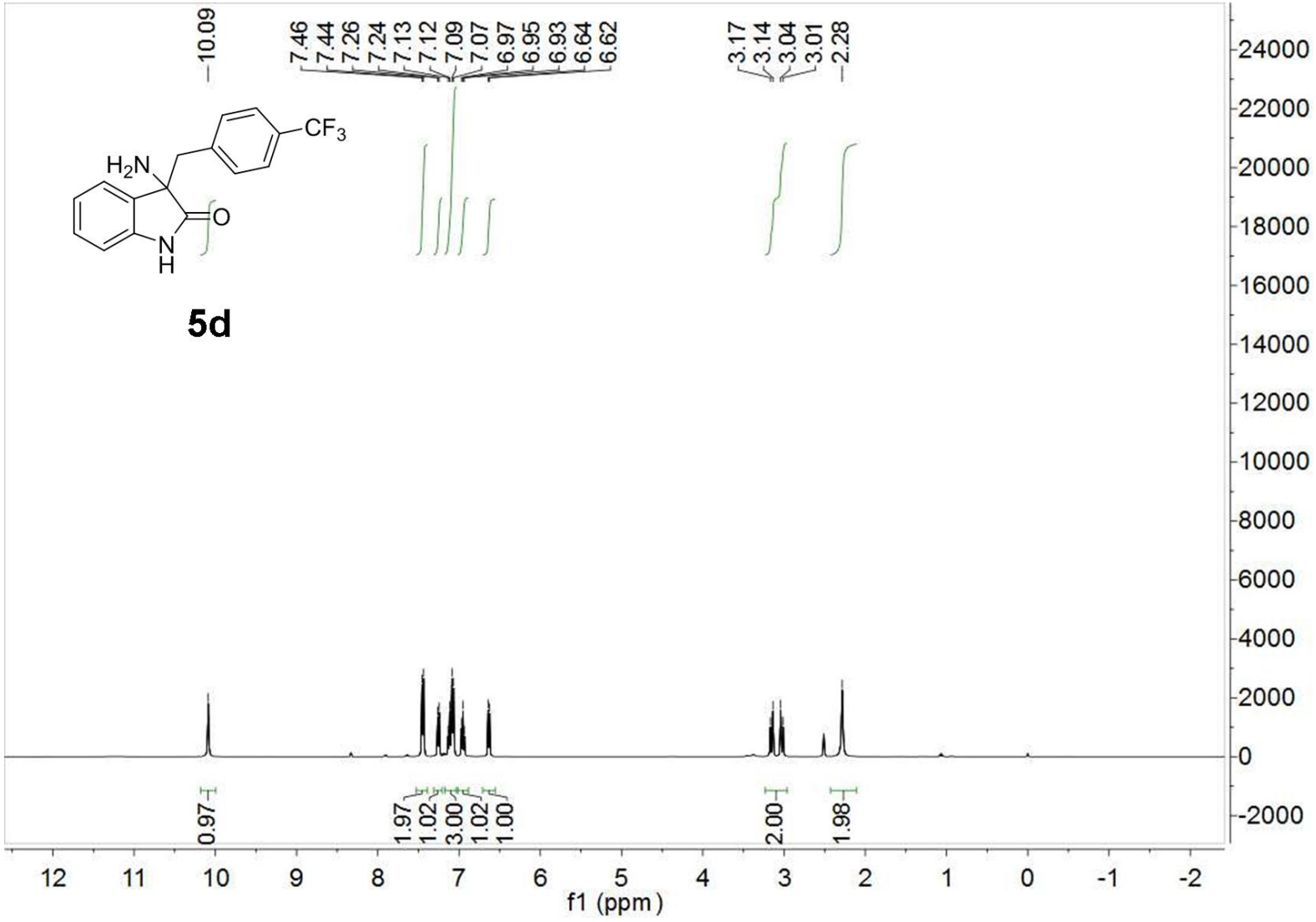

10.03
7.24
7.22
7.12
7.10
7.08
6.95
6.93
6.91
6.89
6.87
6.86
6.84
6.83
6.62
6.60
3.05
3.02
2.93
2.90
2.21
F
H2N
O
N
H
5b
0.96
0.98
1.01
4.91
1.00
2.00
1.96
f1 (ppm)

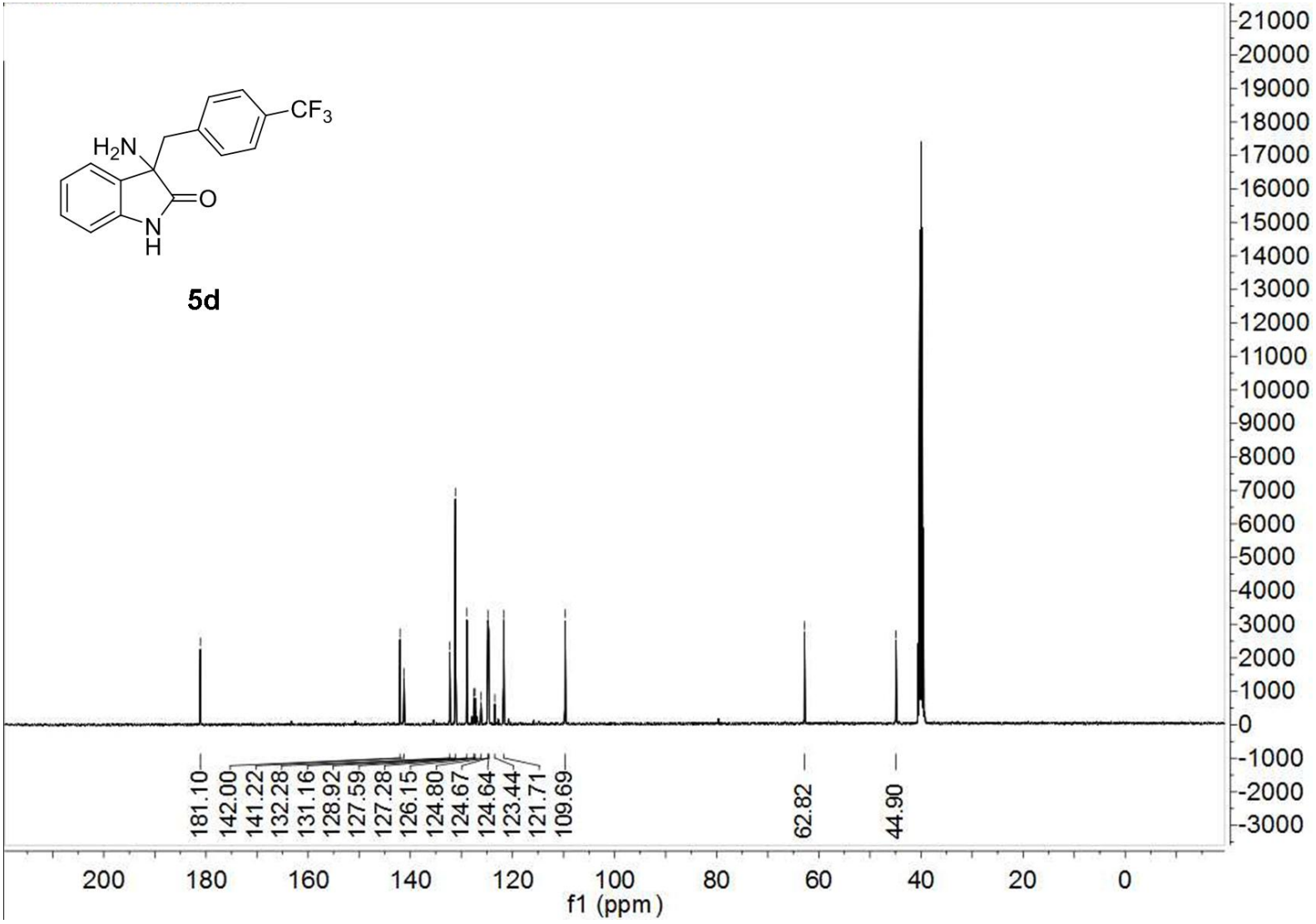

F
H2N
O
N
H
5b
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
181.34
162.58
160.17
142.09
132.51
132.44
132.41
132.14
132.06
128.78
124.77
121.62
114.69
114.48
109.62
62.93
44.36
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

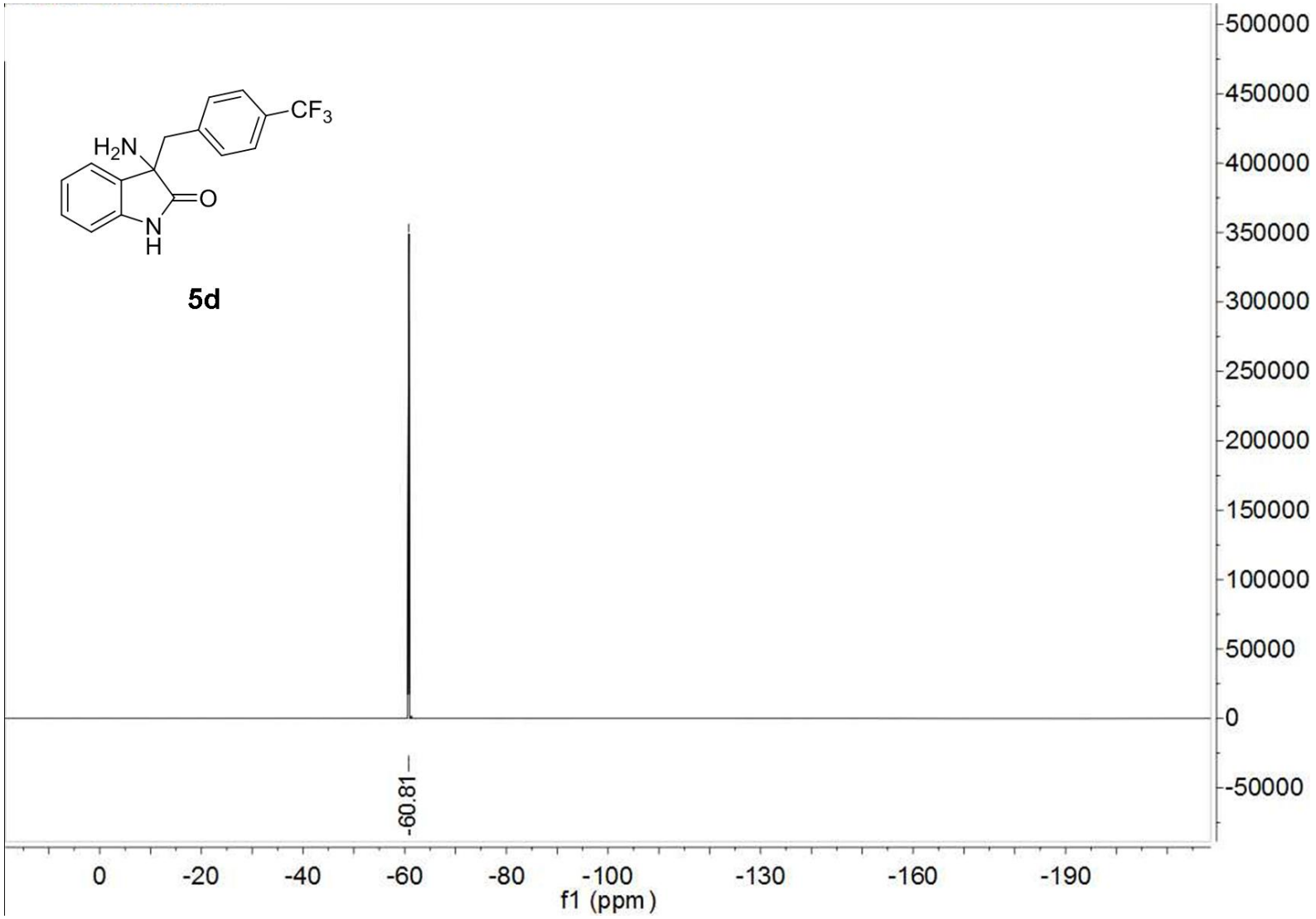

F
H2N
O
N
H
5b
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-116.80
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

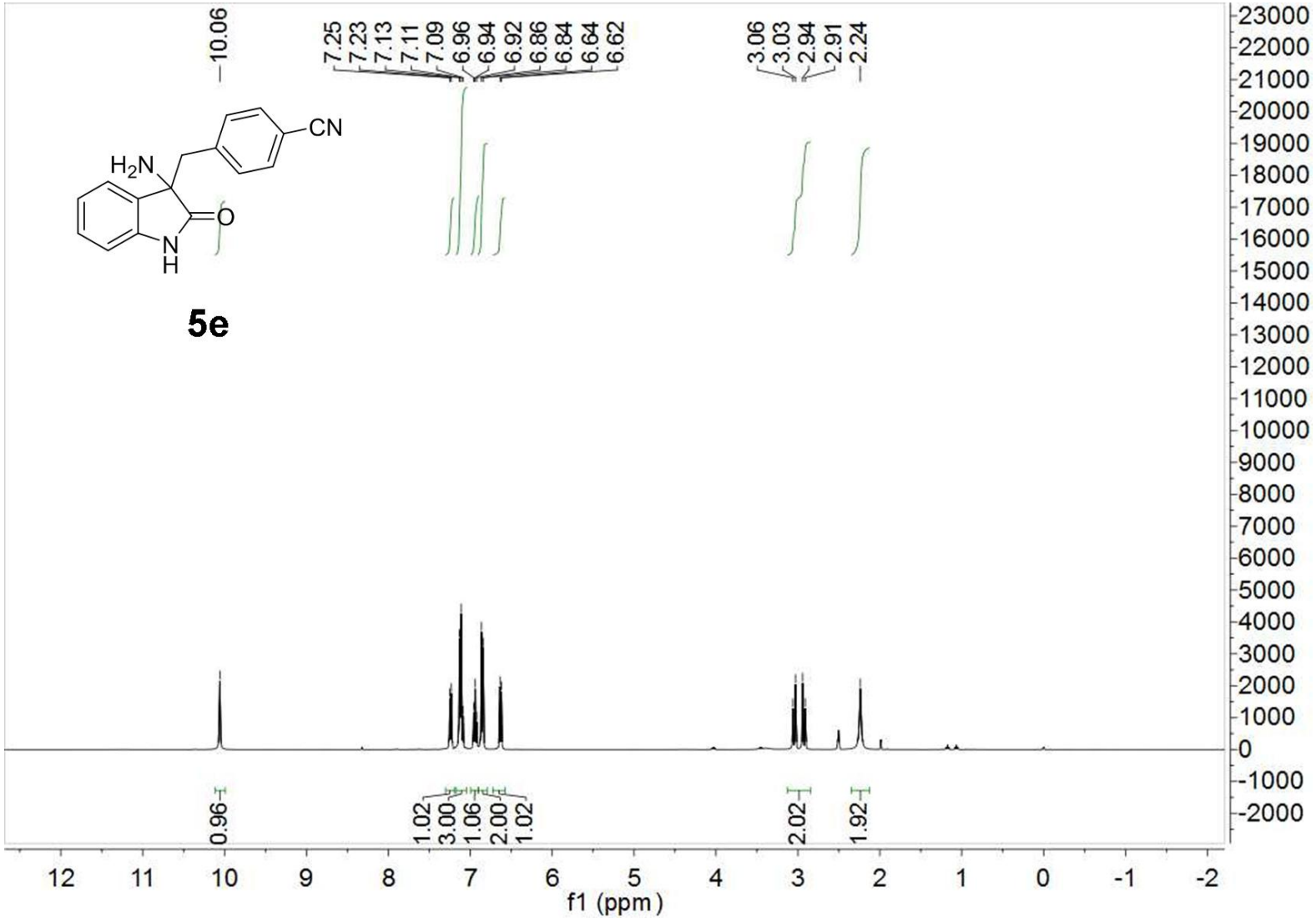

10.14
7.98
7.96
7.24
7.22
7.19
7.16
7.14
7.12
7.10
6.97
6.95
6.93
6.66
6.64
3.23
3.20
3.09
3.05
2.37
NO2
H2N
O
N
H
5c
0.92
1.99
1.08
2.87
1.00
0.99
2.09
1.70
f1 (ppm)

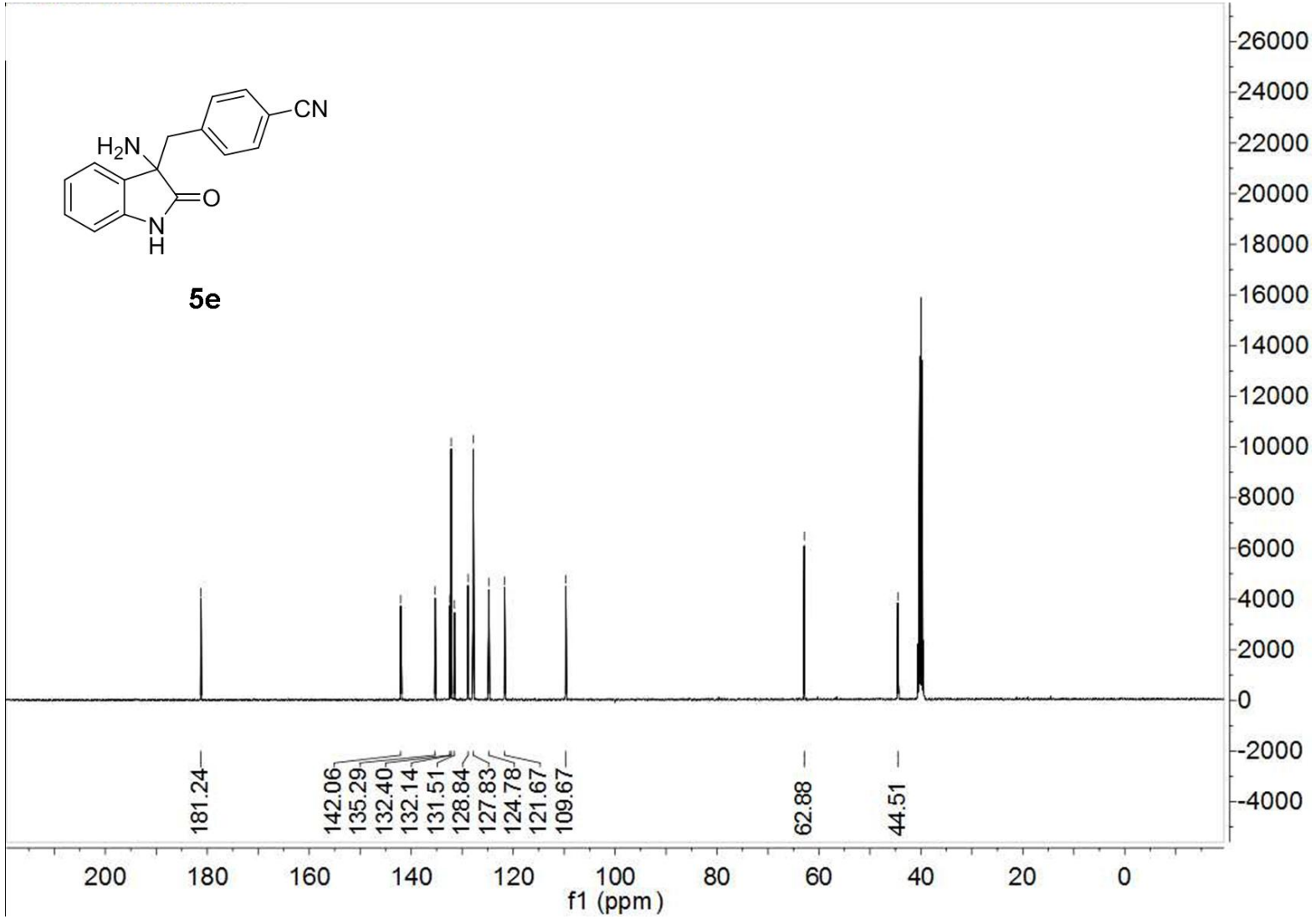

NO2
H2N
O
N
H
5c
35000
30000
25000
20000
15000
10000
5000
0
-5000
181.00
146.61
144.66
141.93
132.04
131.72
129.00
124.86
122.91
121.78
109.79
62.81
44.74
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

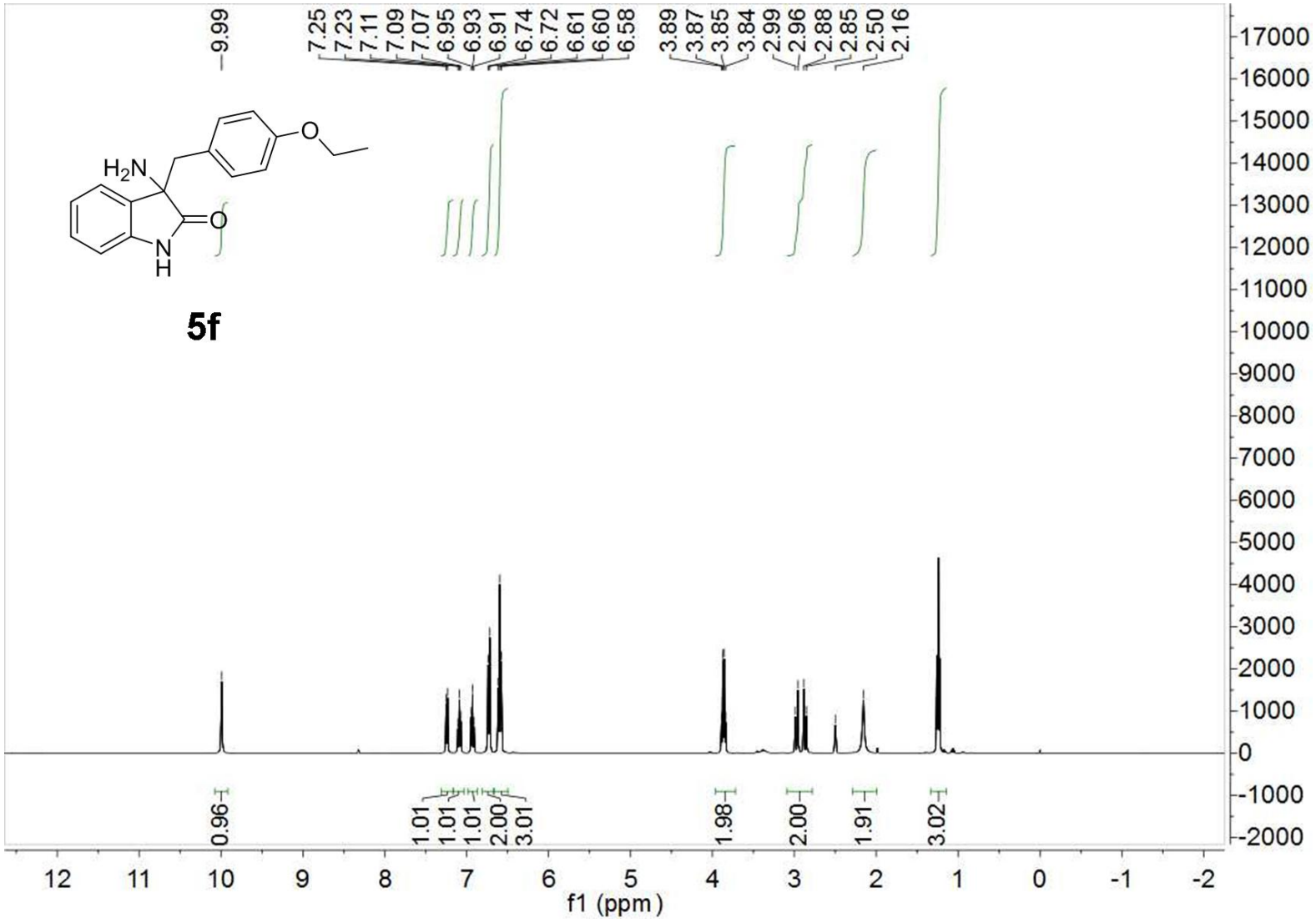

10.09
7.46
7.44
7.26
7.24
7.13
7.12
7.09
7.07
6.97
6.95
6.93
6.64
6.62
3.17
3.14
3.04
3.01
2.28
CF3
H2N
O
N
H
5d
0.97
1.97
1.02
3.00
1.02
1.00
2.00
1.98
f1 (ppm)

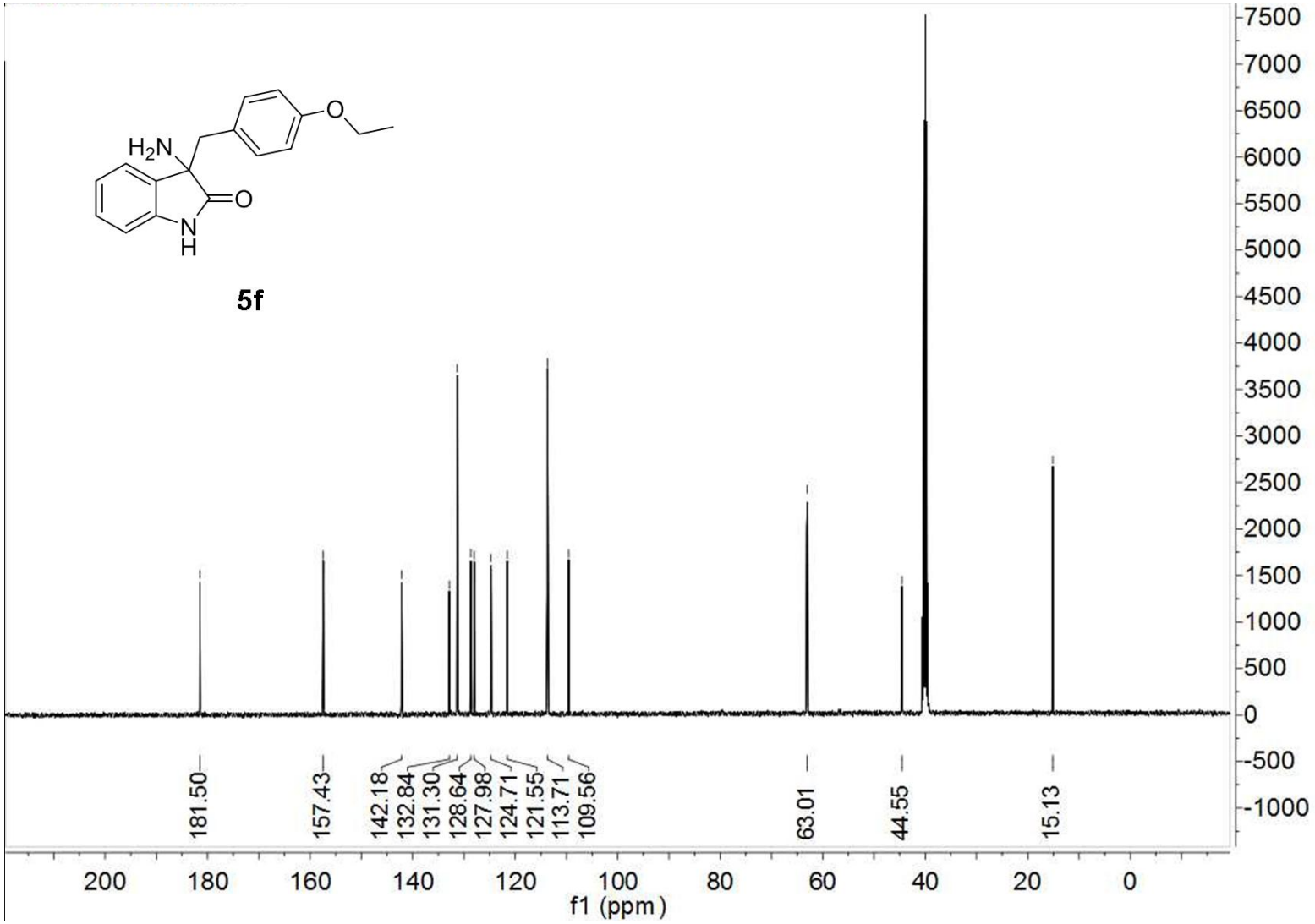

H_2N
CF_3
O
N
H
5d
181.10
142.00
141.22
132.28
131.16
128.92
127.59
127.28
126.15
124.80
124.67
124.64
123.44
121.71
109.69
62.82
44.90
f1 (ppm)

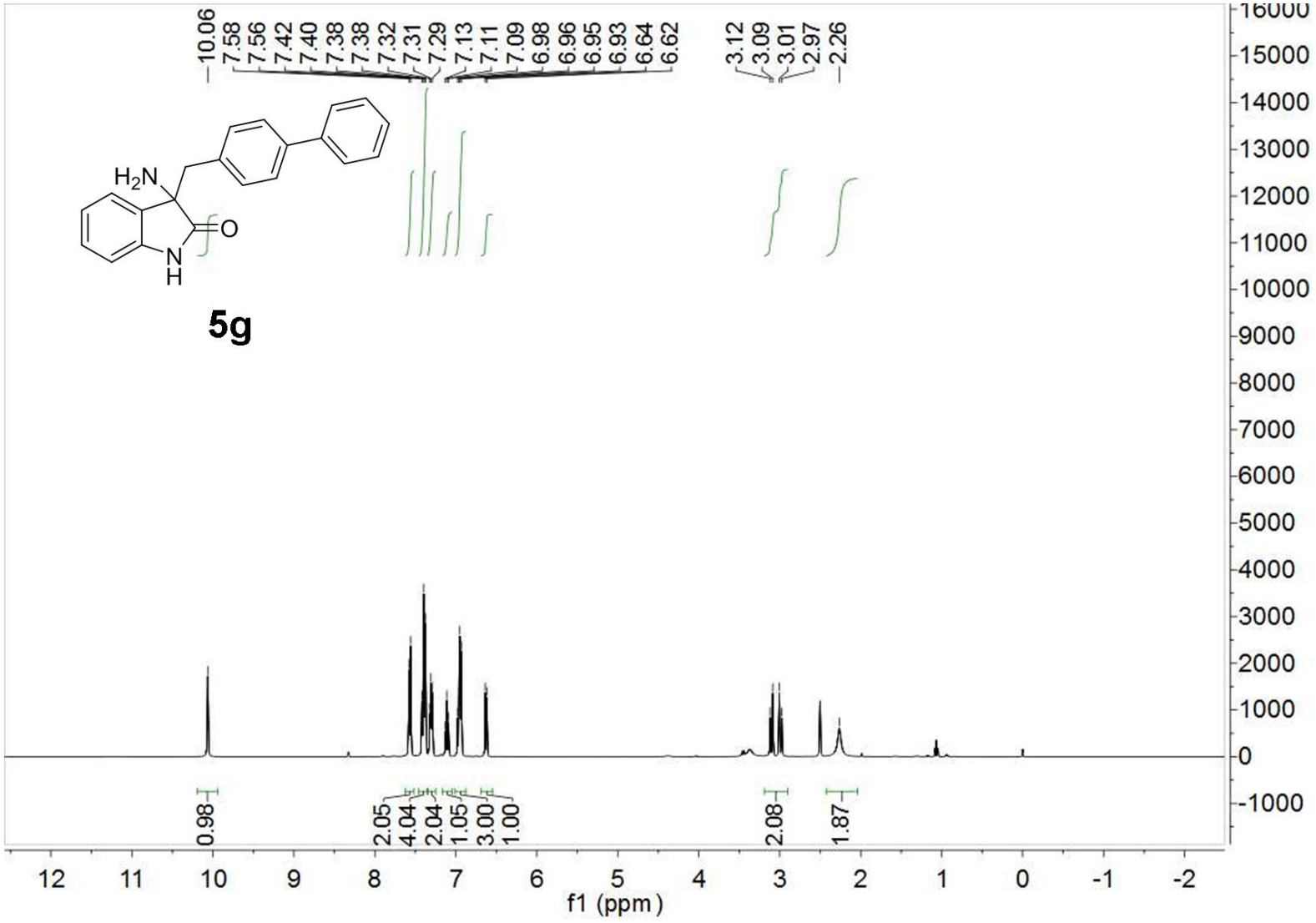

H_2N
CF_3
O
N
H
5d
-60.81
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

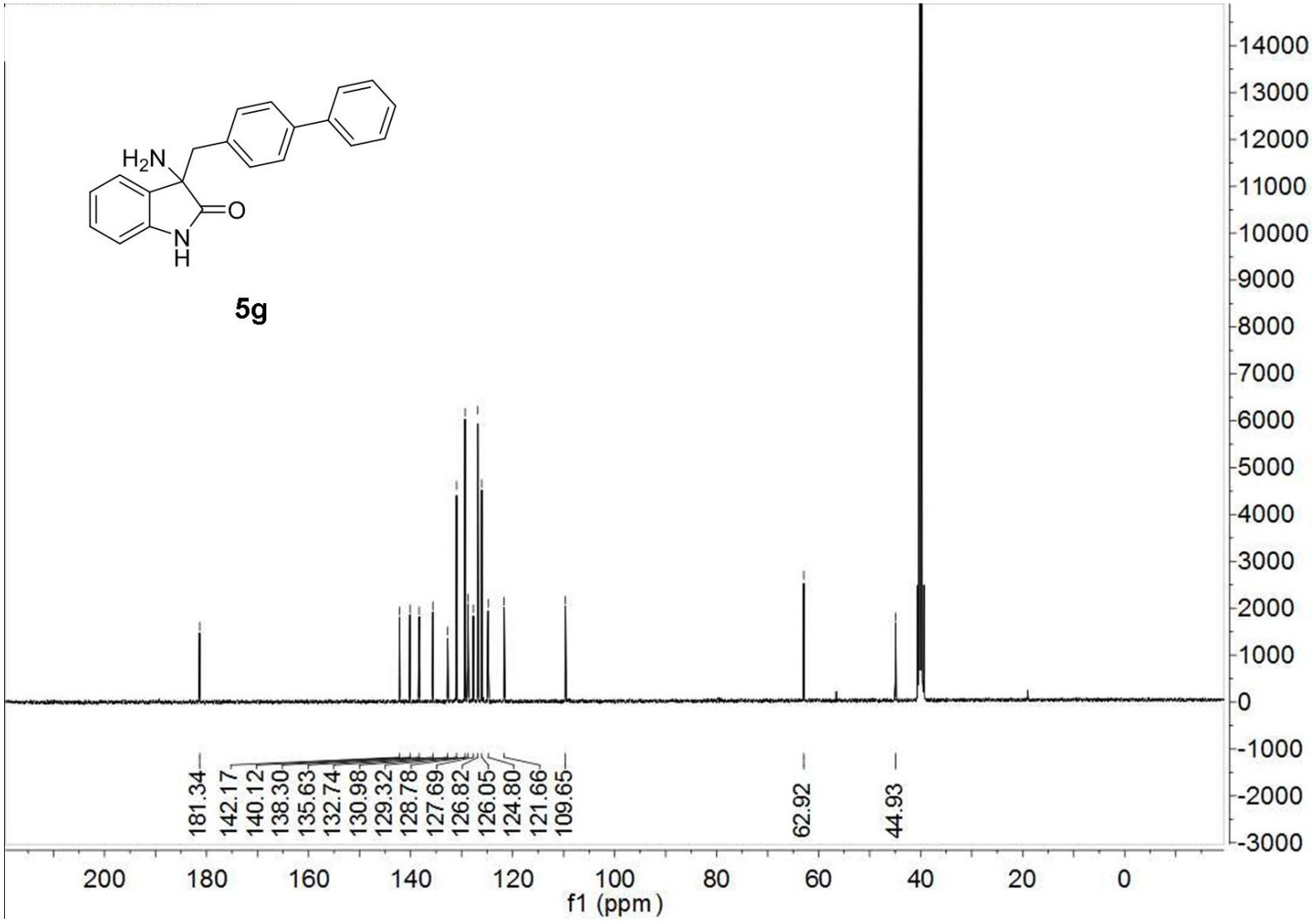

10.06
7.25
7.23
7.13
7.11
7.09
6.96
6.94
6.92
6.86
6.84
6.64
6.62
3.06
3.03
2.94
2.91
2.24
CN
H2N
O
N
H
5e
0.96
1.02
3.00
1.06
2.00
1.02
2.02
1.92
f1 (ppm)

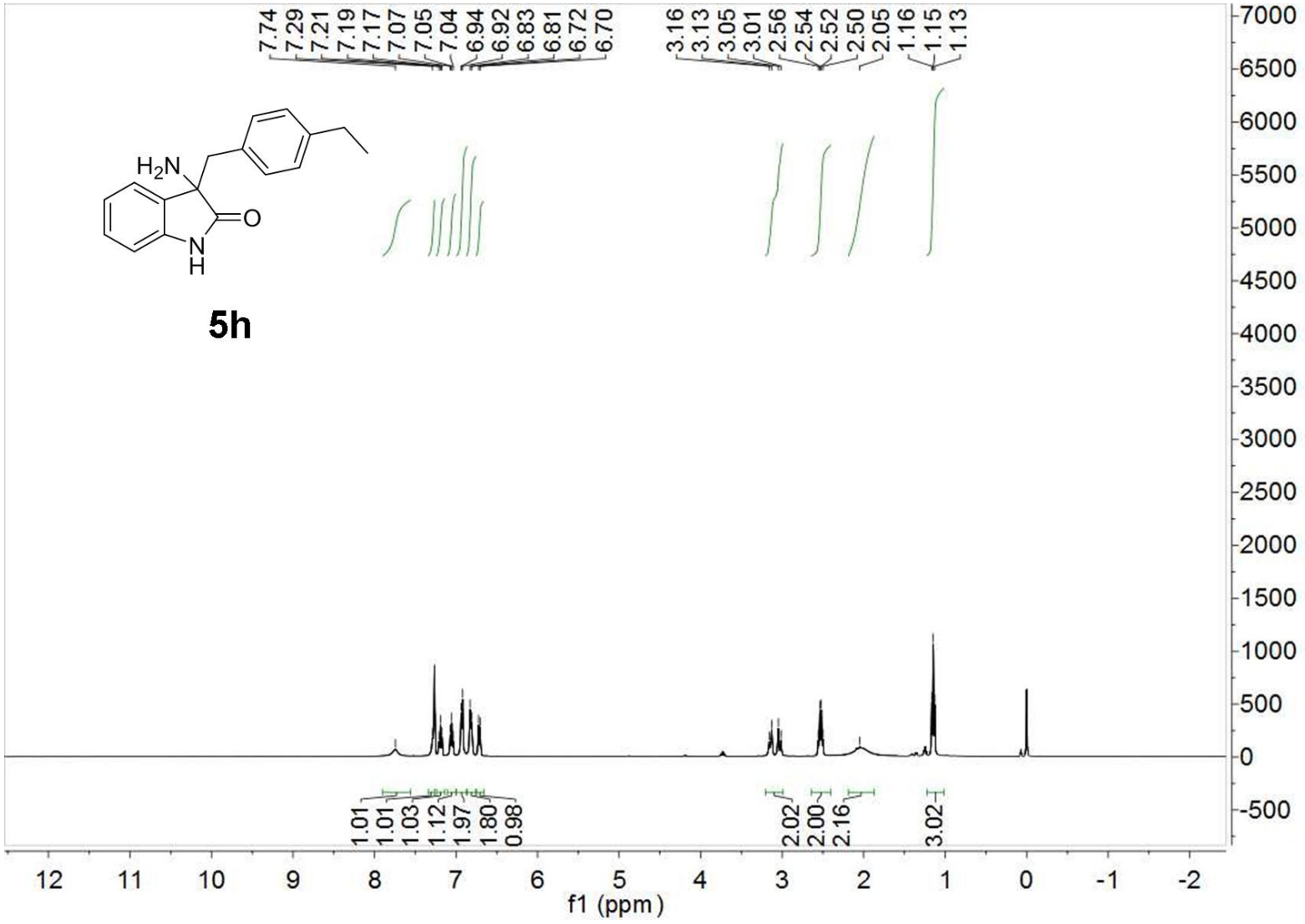

CN
H2N
O
N
H
5e
181.24
142.06
135.29
132.40
132.14
131.51
128.84
127.83
124.78
121.67
109.67
62.88
44.51
f1 (ppm)

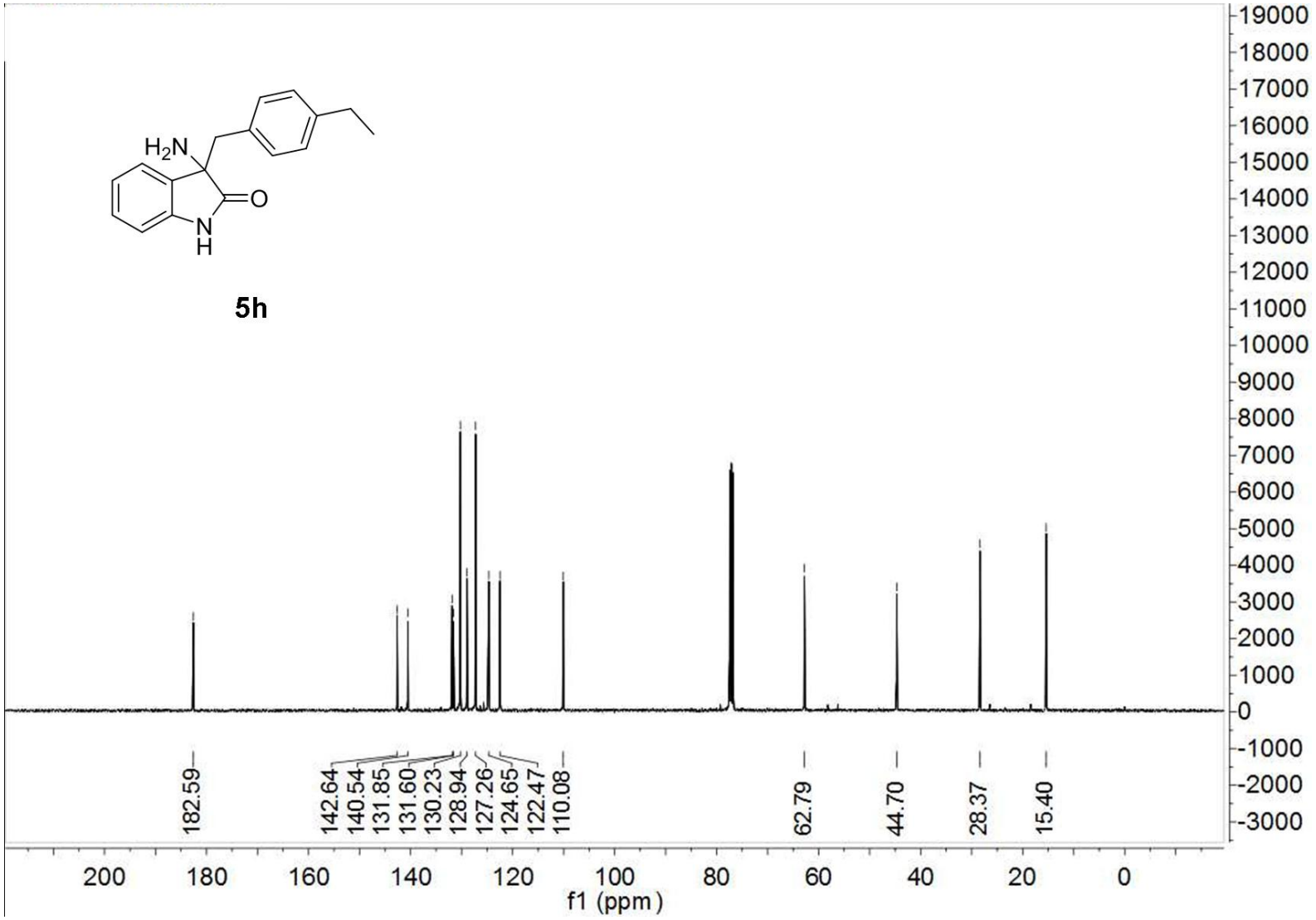

9.99
7.25
7.23
7.11
7.09
7.07
6.95
6.93
6.91
6.74
6.72
6.61
6.60
6.58
3.89
3.87
3.85
3.84
2.99
2.96
2.88
2.85
2.50
2.16
H2N
O
O
N
H
5f
0.96
1.01
1.01
1.01
2.00
3.01
1.98
2.00
1.91
3.02
f1 (ppm)

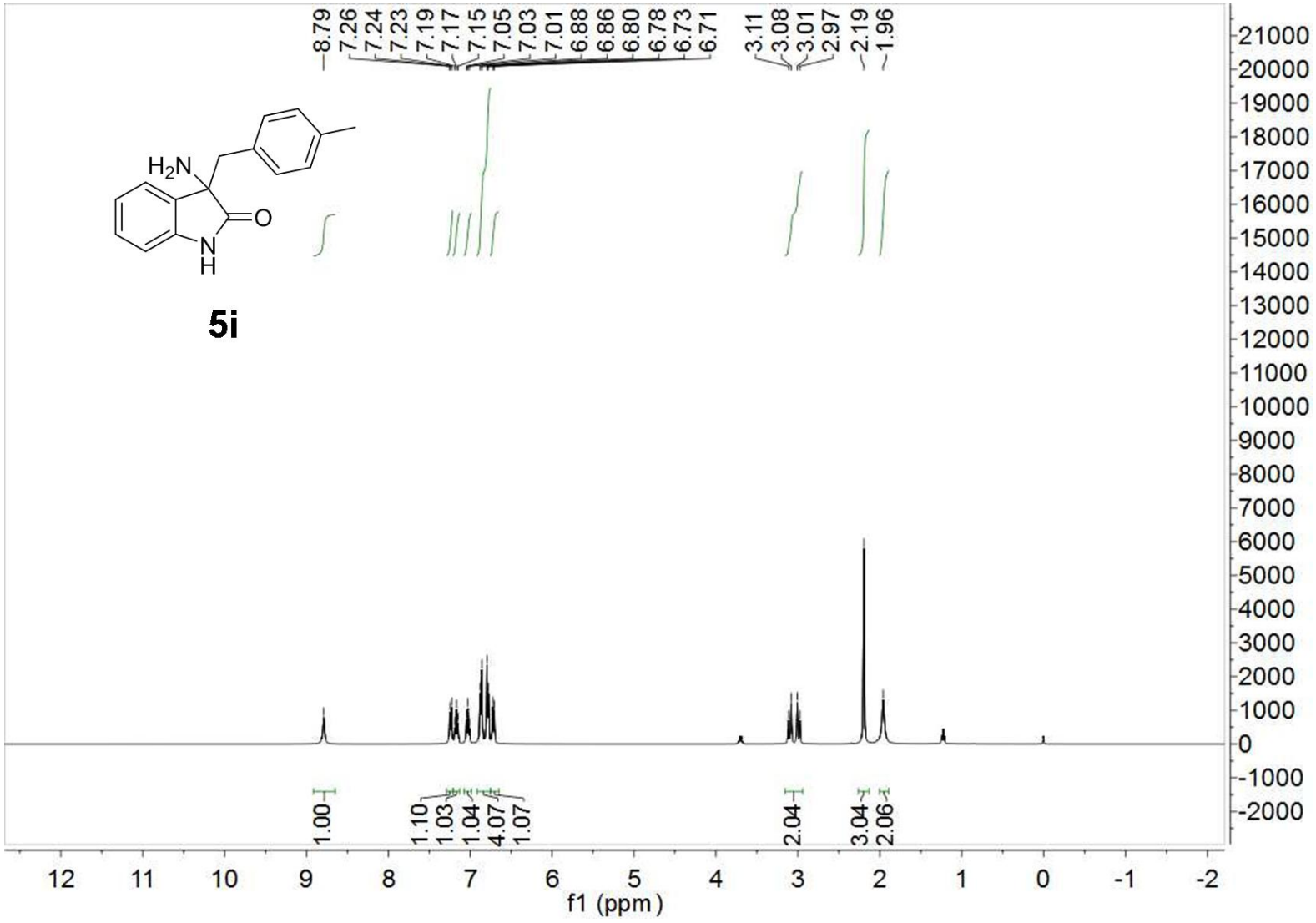

H2N
O
O
N
H
5f
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
181.50
157.43
142.18
132.84
131.30
128.64
127.98
124.71
121.55
113.71
109.56
63.01
44.55
15.13
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

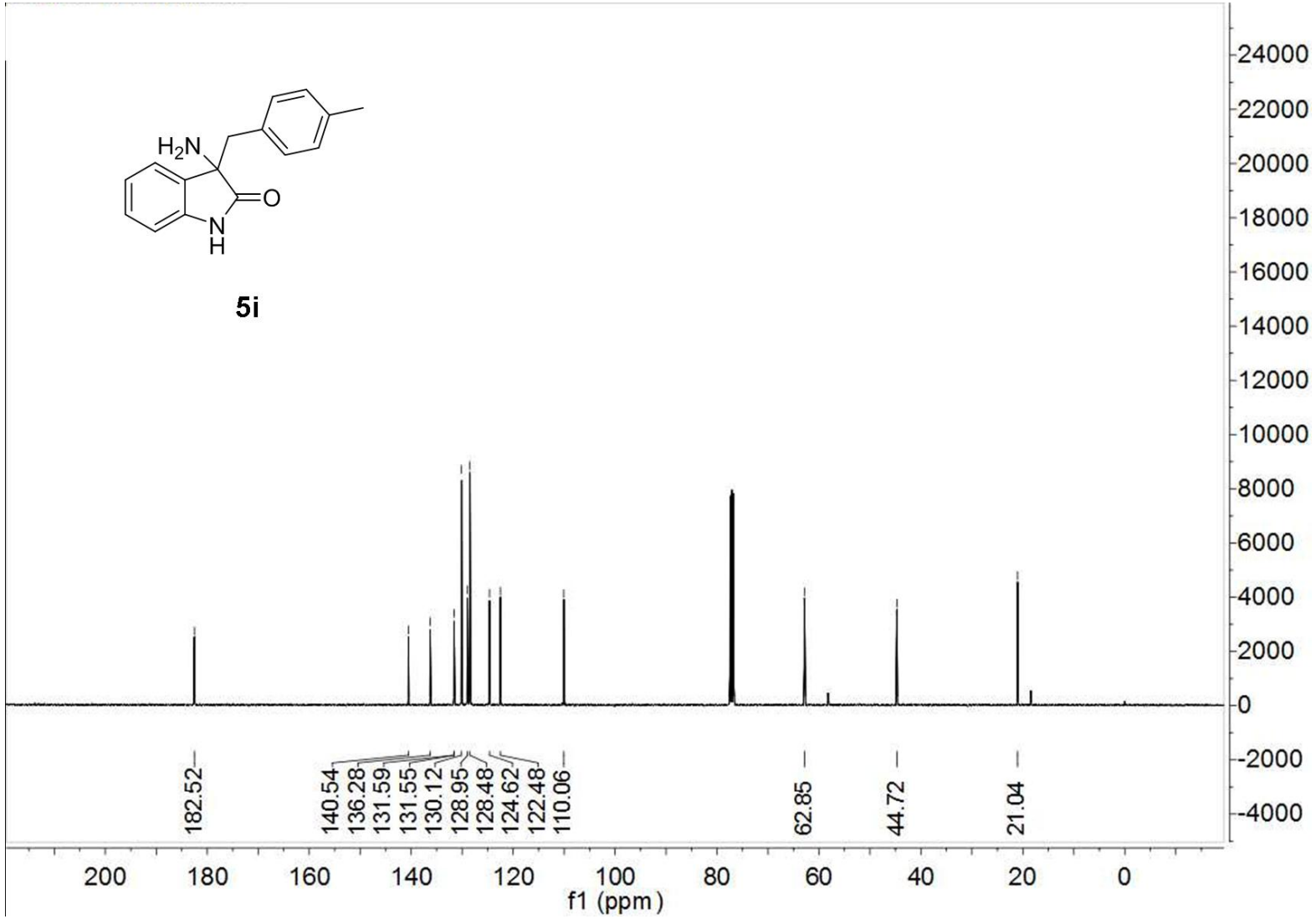

10.06
7.58
7.56
7.42
7.40
7.38
7.38
7.32
7.31
7.29
7.13
7.11
7.09
6.98
6.96
6.95
6.93
6.64
6.62
3.12
3.09
3.01
2.97
2.26
H2N
O
N
H
5g
0.98
2.05
4.04
2.04
1.05
3.00
1.00
2.08
1.87
f1 (ppm)

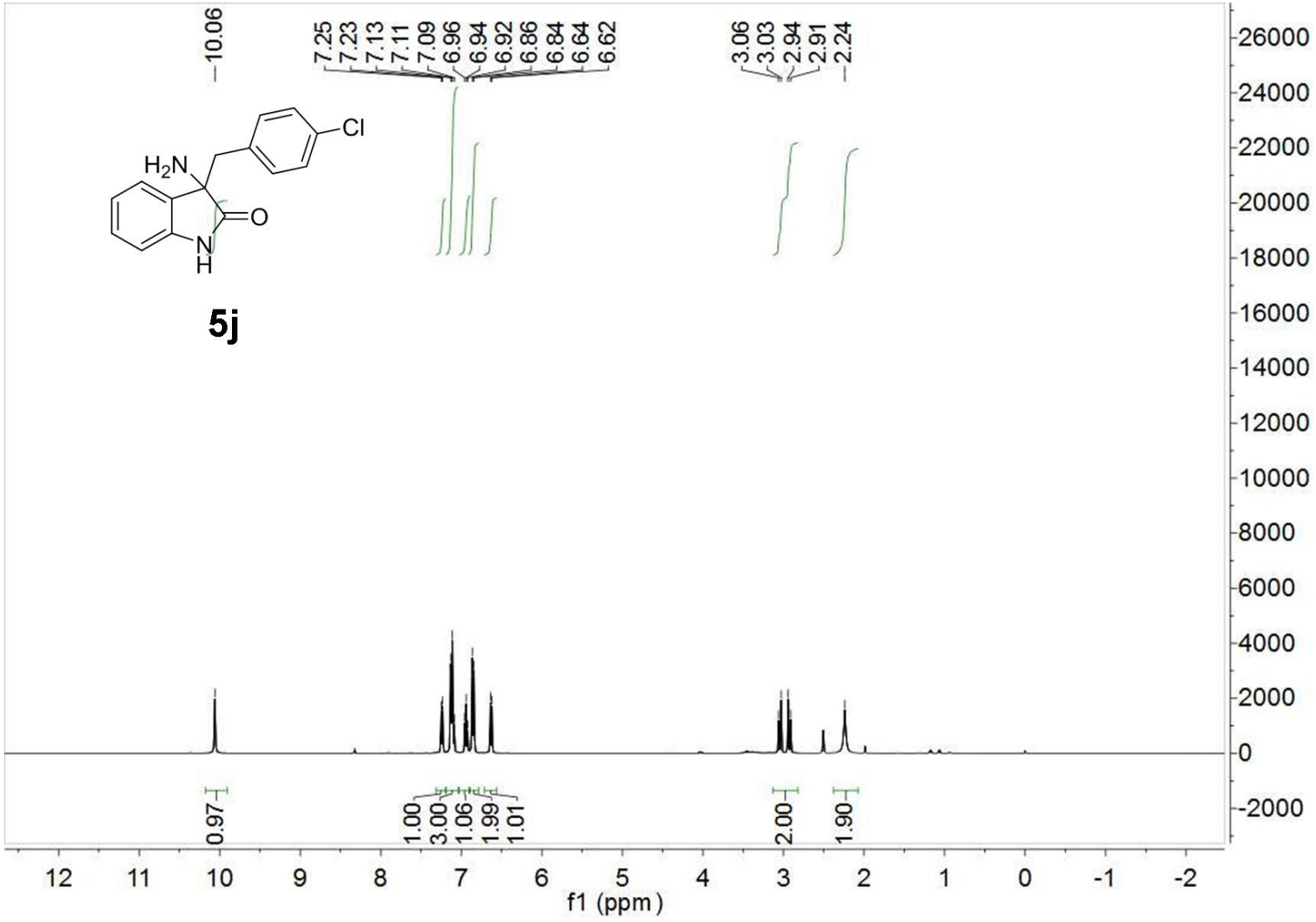

H2N
O
N
H
5g
181.34
142.17
140.12
138.30
135.63
132.74
130.98
129.32
128.78
127.69
126.82
126.05
124.80
121.66
109.65
62.92
44.93
f1 (ppm)

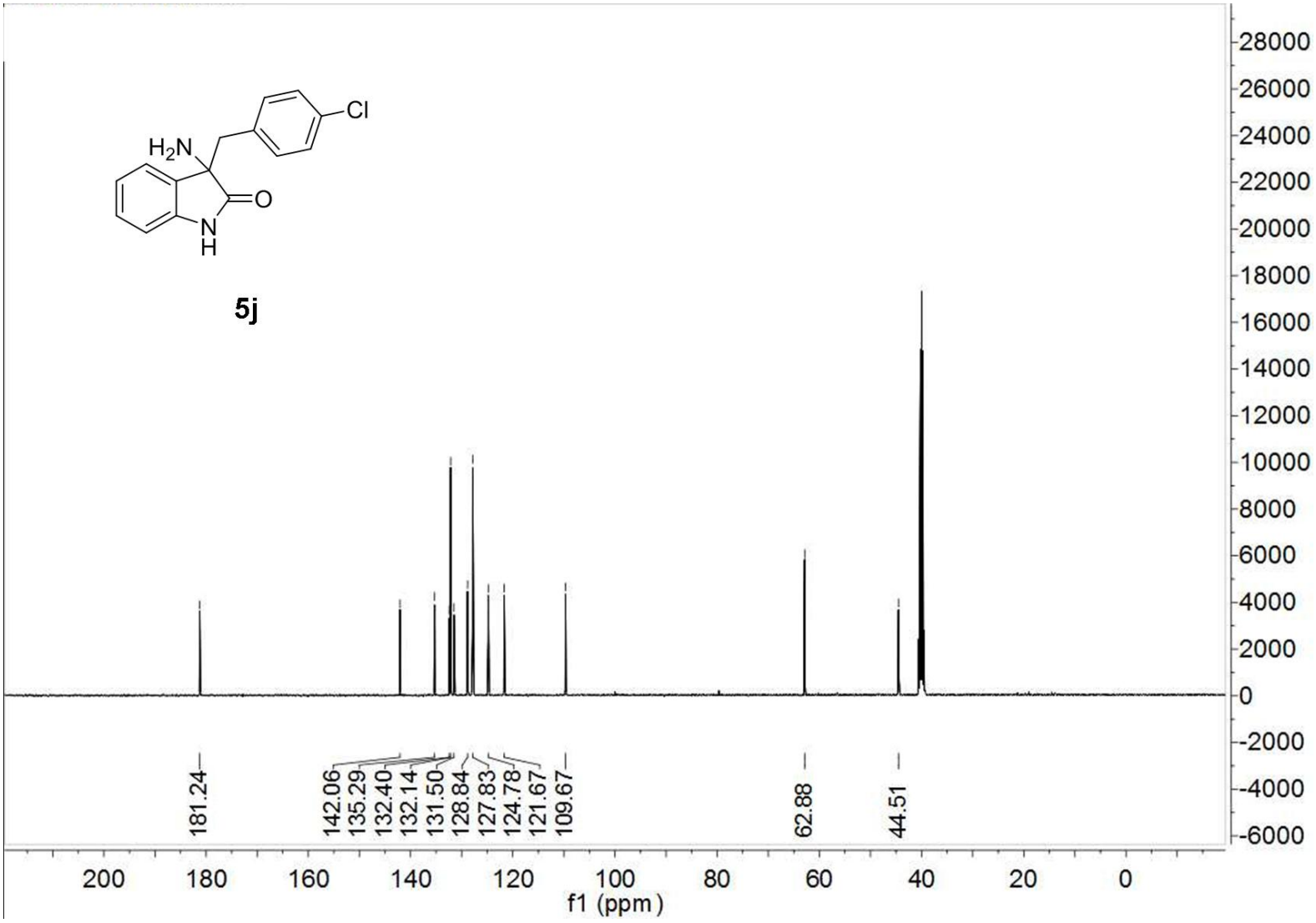

7.74
7.29
7.21
7.19
7.17
7.07
7.05
7.04
6.94
6.92
6.83
6.81
6.72
6.70
3.16
3.13
3.05
3.01
2.56
2.54
2.52
2.50
2.05
1.16
1.15
1.13
H2N
O
N
H
5h
1.01
1.01
1.03
1.12
1.97
1.80
0.98
2.02
2.00
2.16
3.02
f1 (ppm)

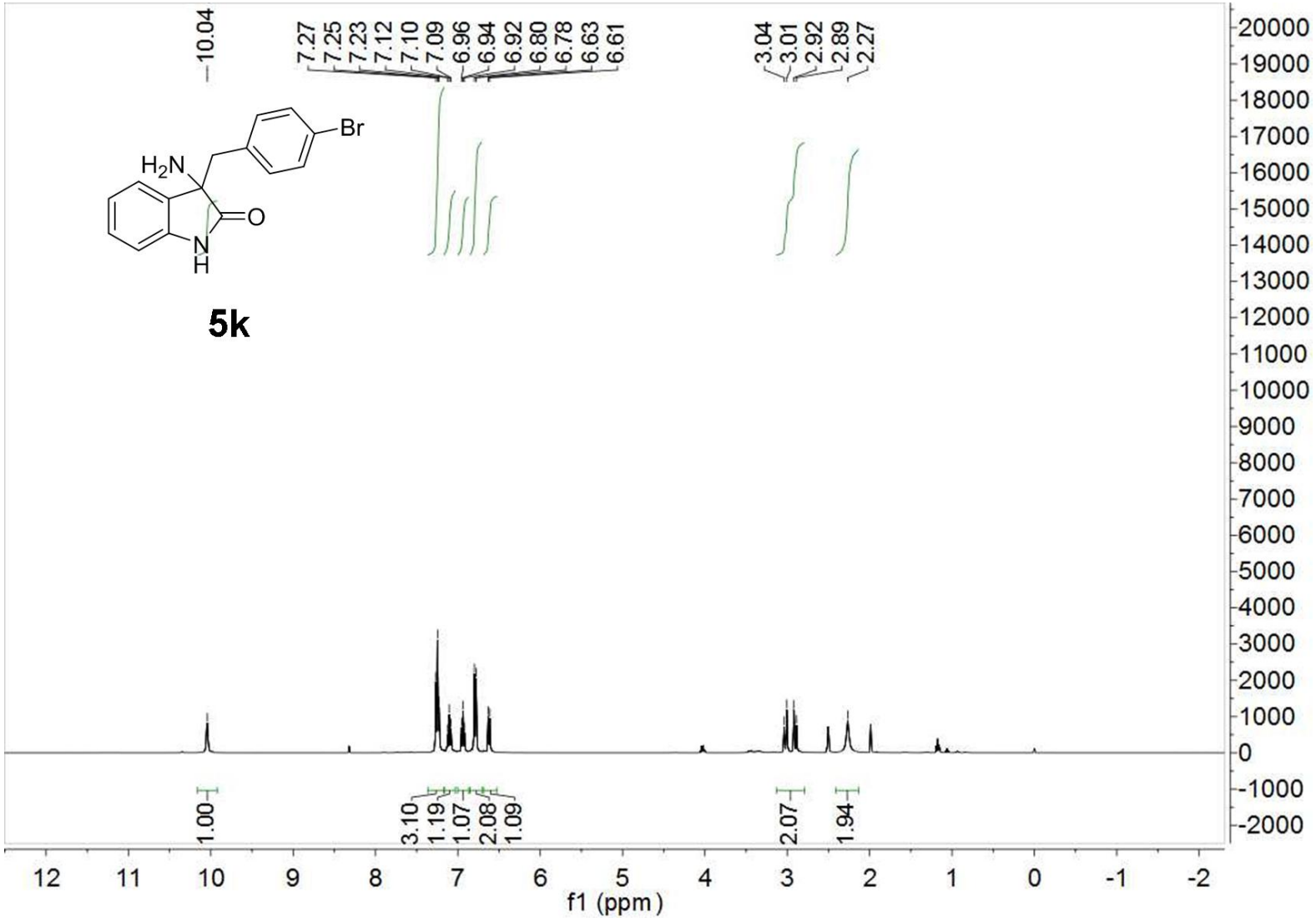

H2N
O
N
H
5h
182.59
142.64
140.54
131.85
131.60
130.23
128.94
127.26
124.65
122.47
110.08
62.79
44.70
28.37
15.40
f1 (ppm)

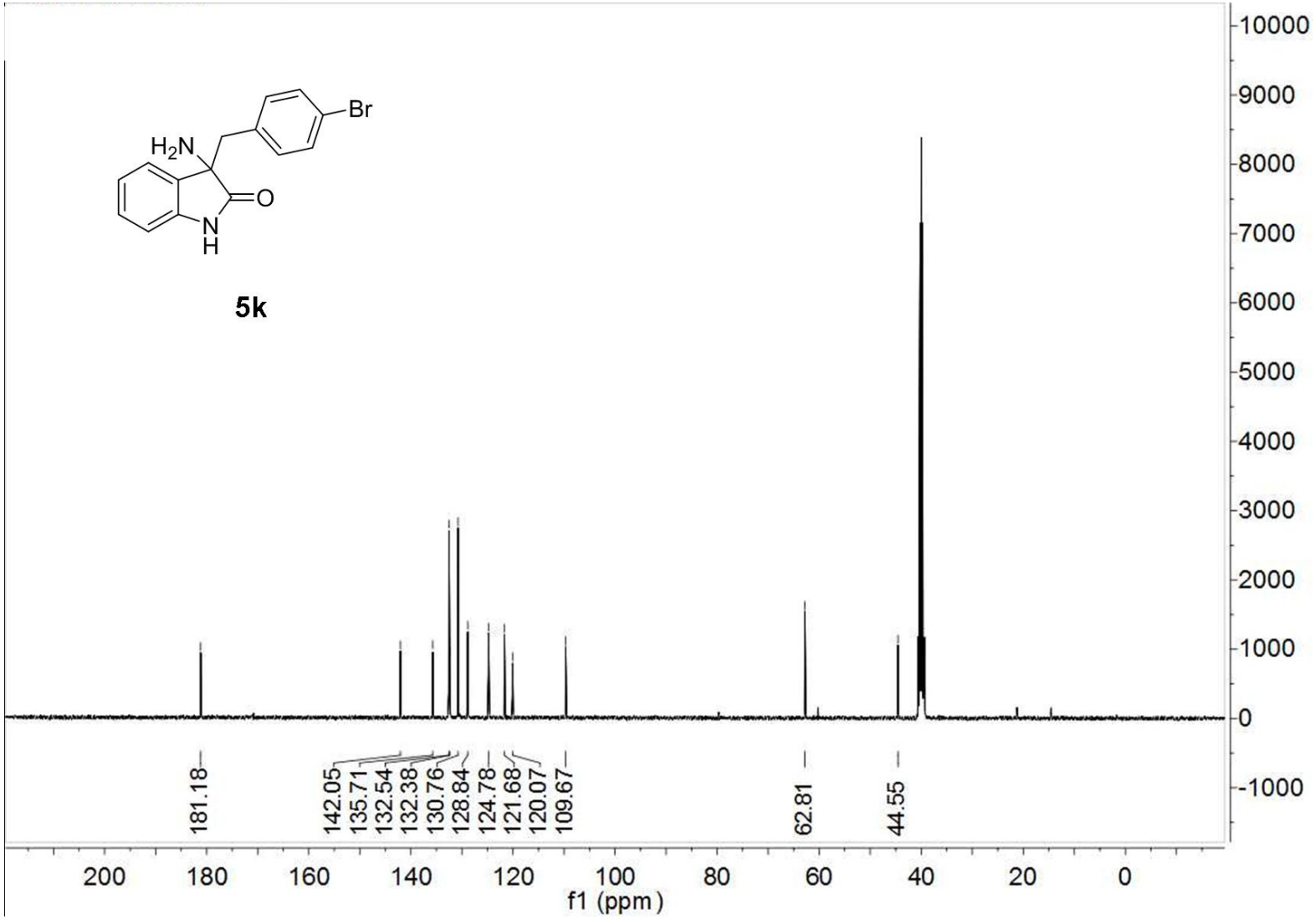

8.79
7.26
7.24
7.23
7.19
7.17
7.15
7.05
7.03
7.01
6.88
6.86
6.80
6.78
6.73
6.71
3.11
3.08
3.01
2.97
2.19
1.96
H2N
O
N
H
5i
1.00
1.10
1.03
1.04
4.07
1.07
2.04
3.04
2.06
f1 (ppm)

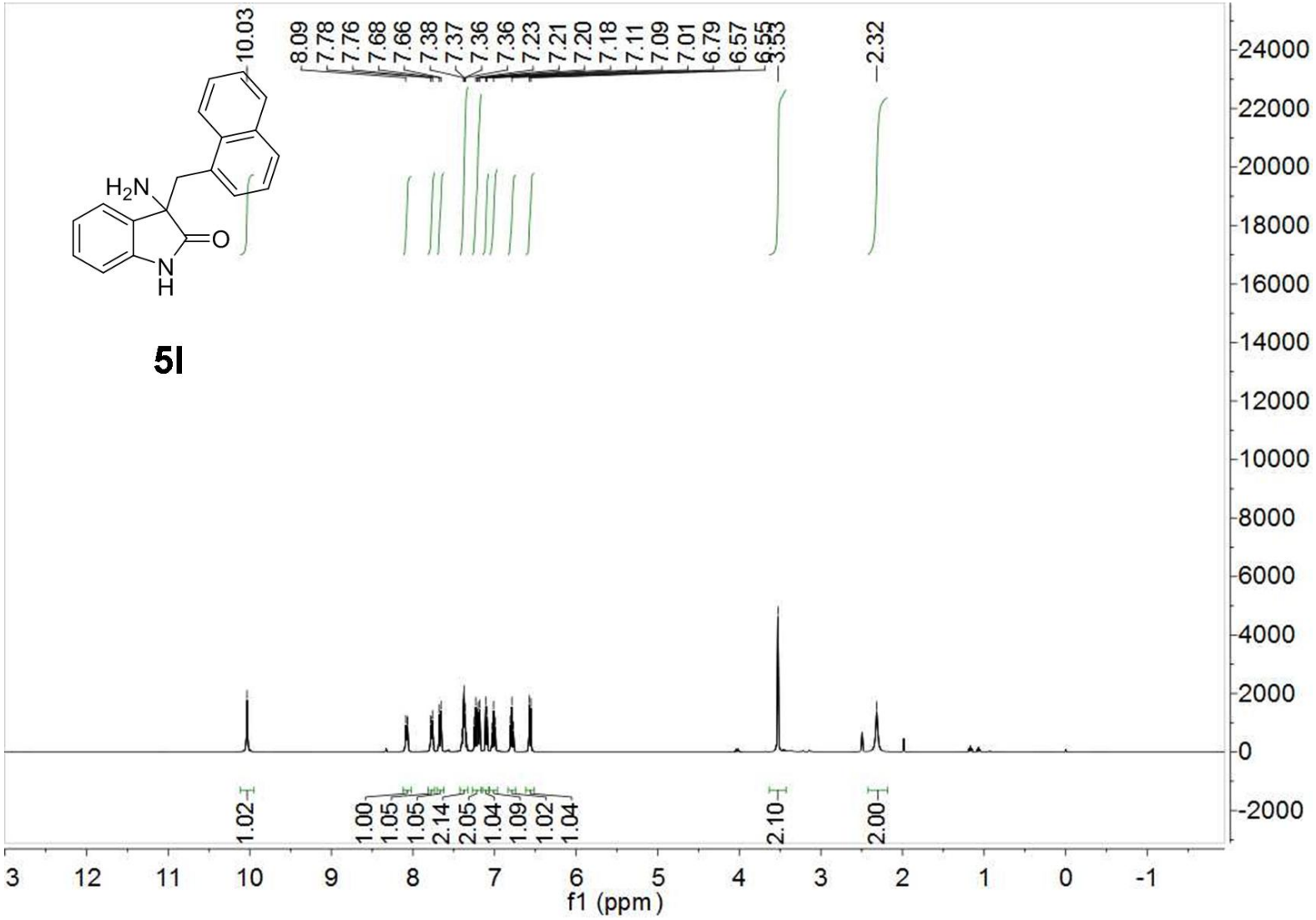

H2N
O
N
H
5i
182.52
140.54
136.28
131.59
131.55
130.12
128.95
128.48
124.62
122.48
110.06
62.85
44.72
21.04
f1 (ppm)

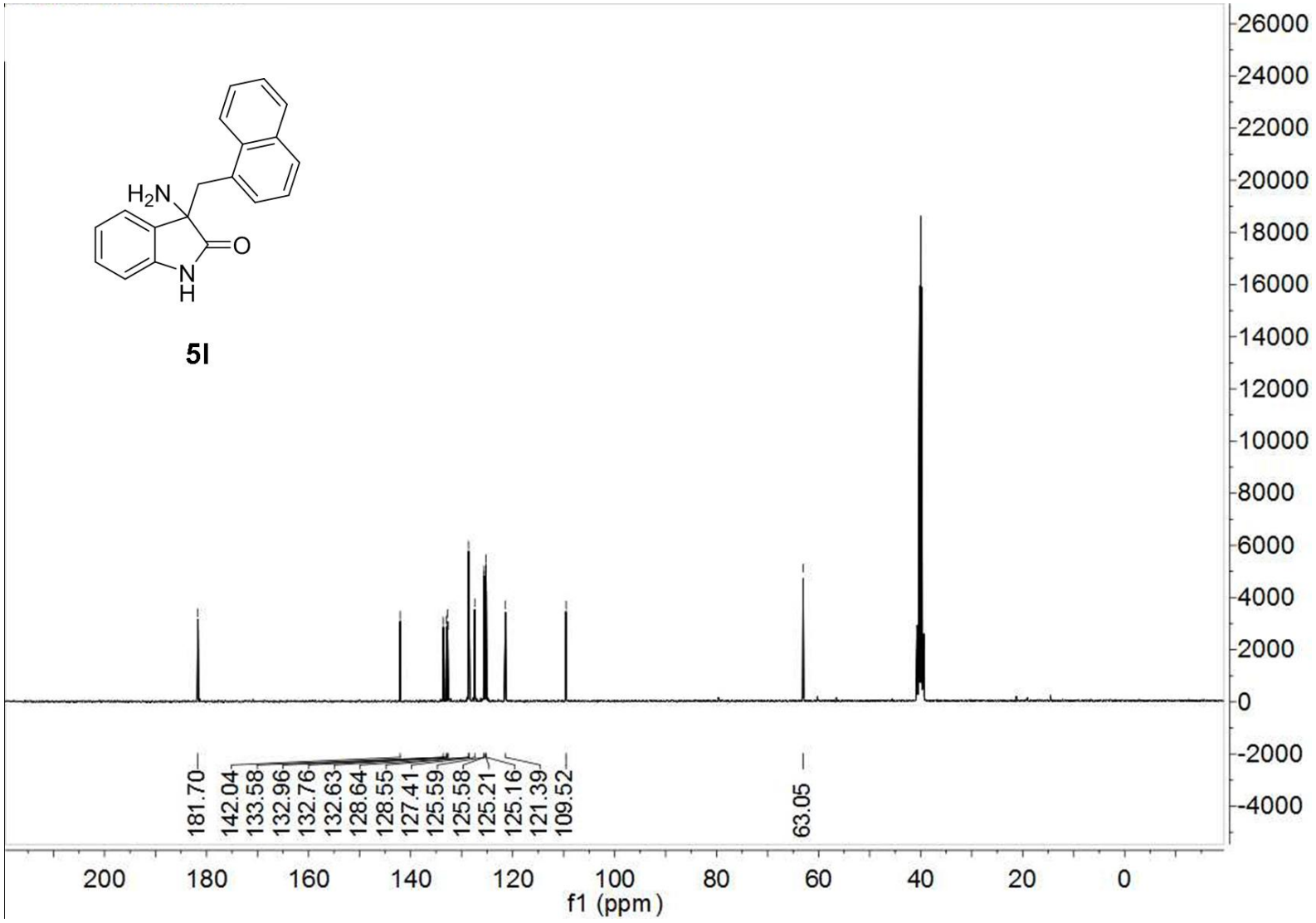

10.06
7.25
7.23
7.13
7.11
7.09
6.96
6.94
6.92
6.86
6.84
6.64
6.62
3.06
3.03
2.94
2.91
2.24
Cl
H2N
O
N
H
5j
0.97
1.00
3.00
1.06
1.99
1.01
2.00
1.90
f1 (ppm)

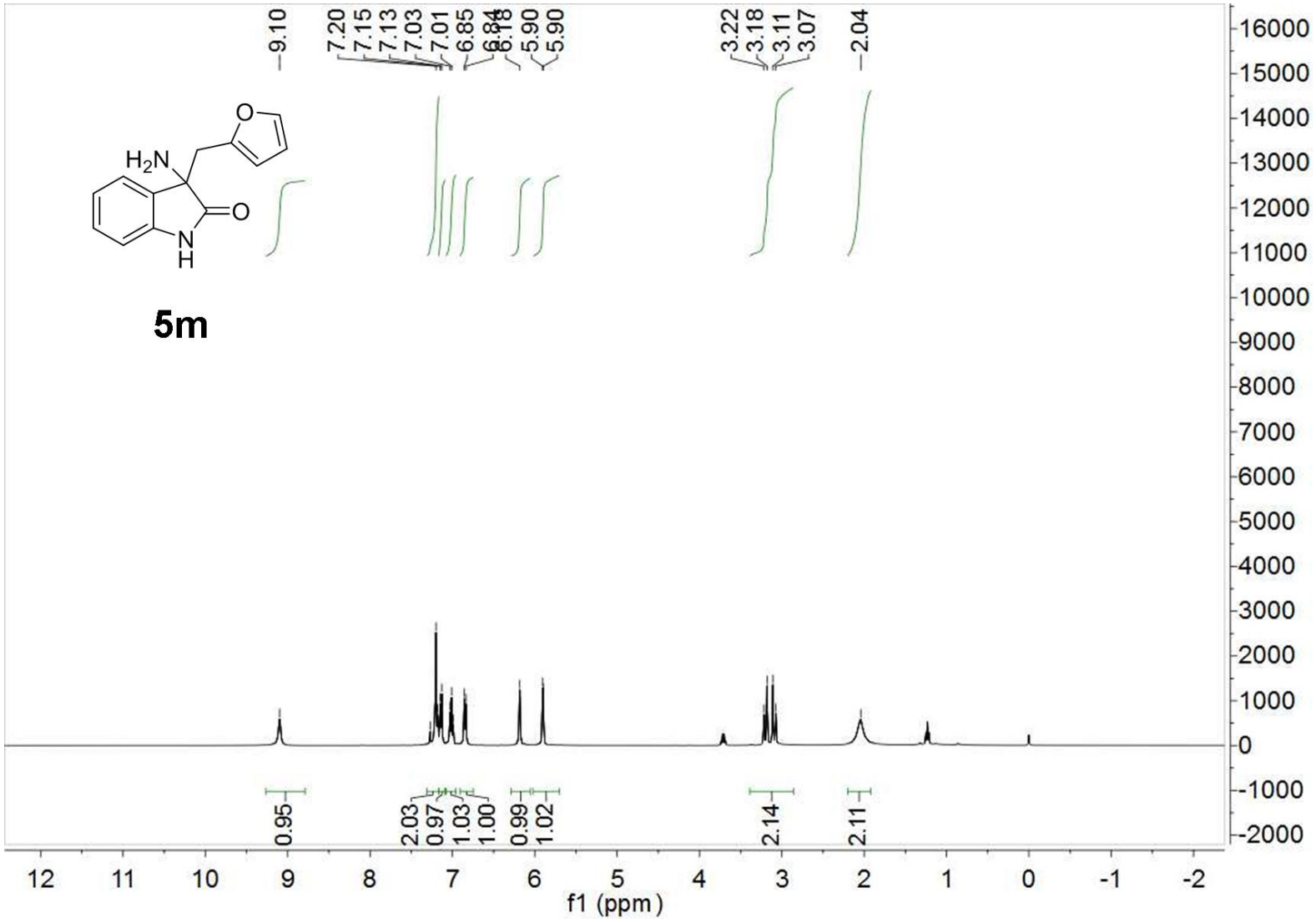

Cl
H2N
O
N
H
5j
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
181.24
142.06
135.29
132.40
132.14
131.50
128.84
127.83
124.78
121.67
109.67
62.88
44.51
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

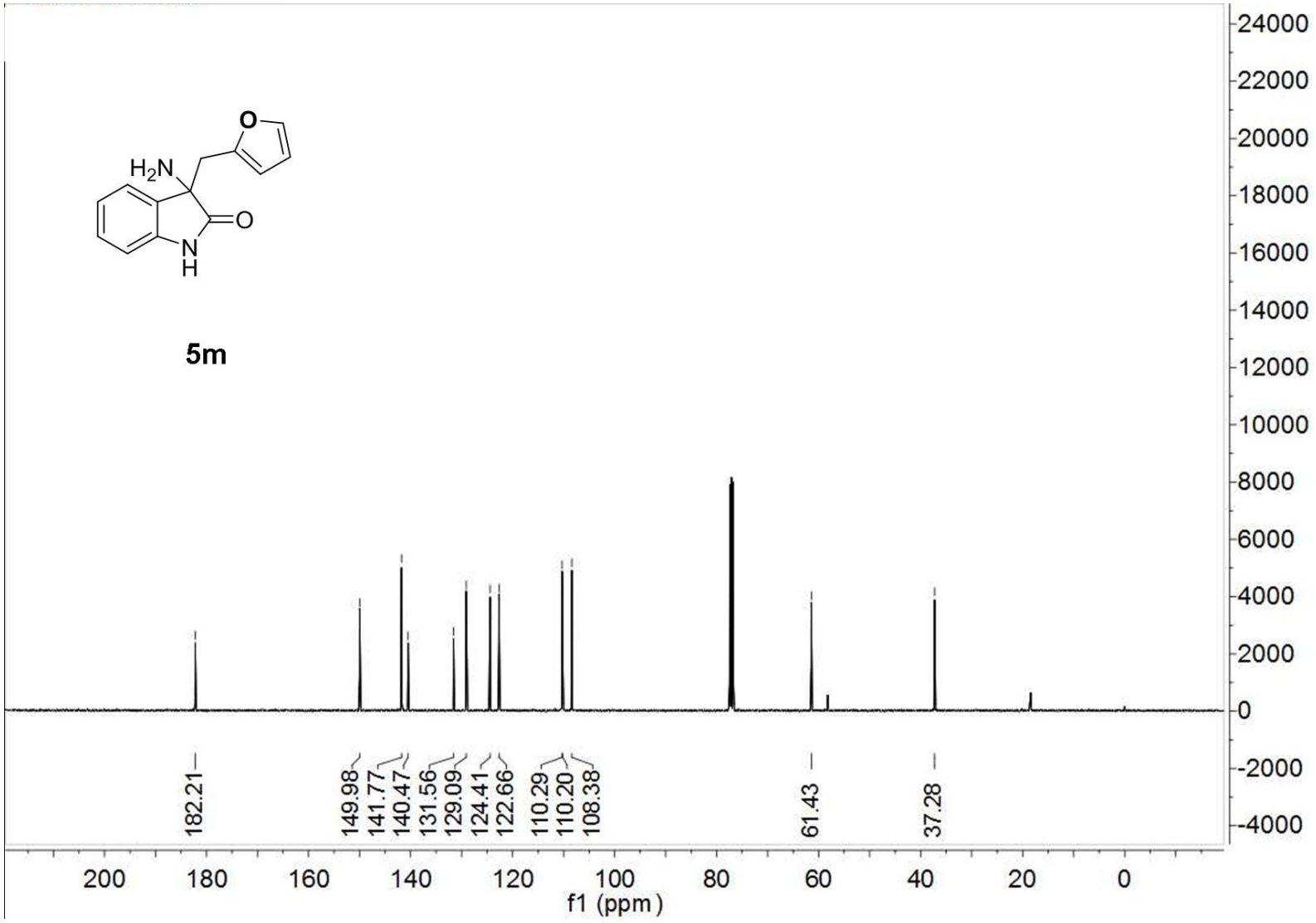

10.04
7.27
7.25
7.23
7.12
7.10
7.09
6.96
6.94
6.92
6.80
6.78
6.63
6.61
3.04
3.01
2.92
2.89
2.27
Br
H2N
O
N
H
5k
1.00
3.10
1.19
1.07
2.08
1.09
2.07
1.94
f1 (ppm)

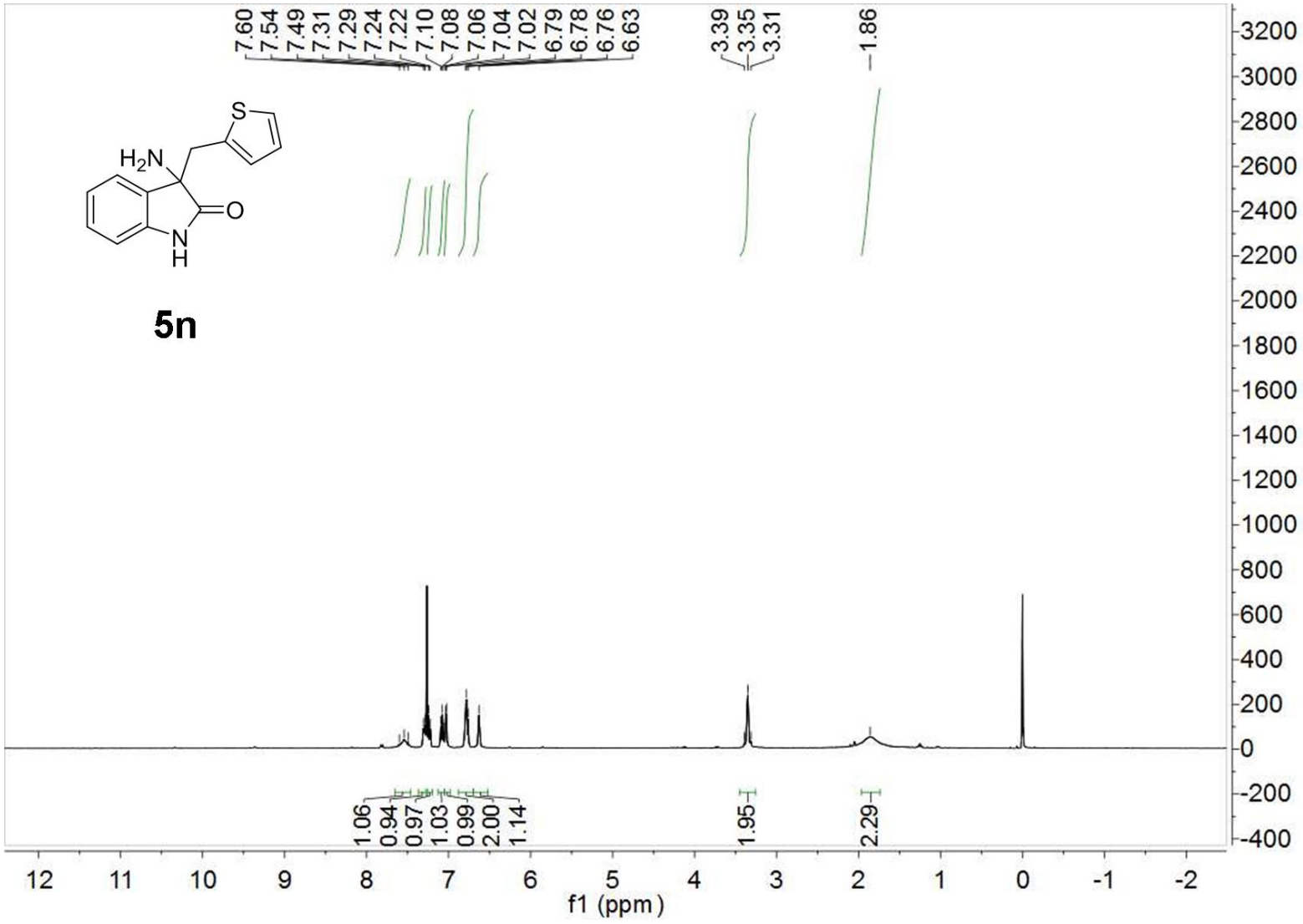

Br
H2N
O
N
H
5k
181.18
142.05
135.71
132.54
132.38
130.76
128.84
124.78
121.68
120.07
109.67
62.81
44.55
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

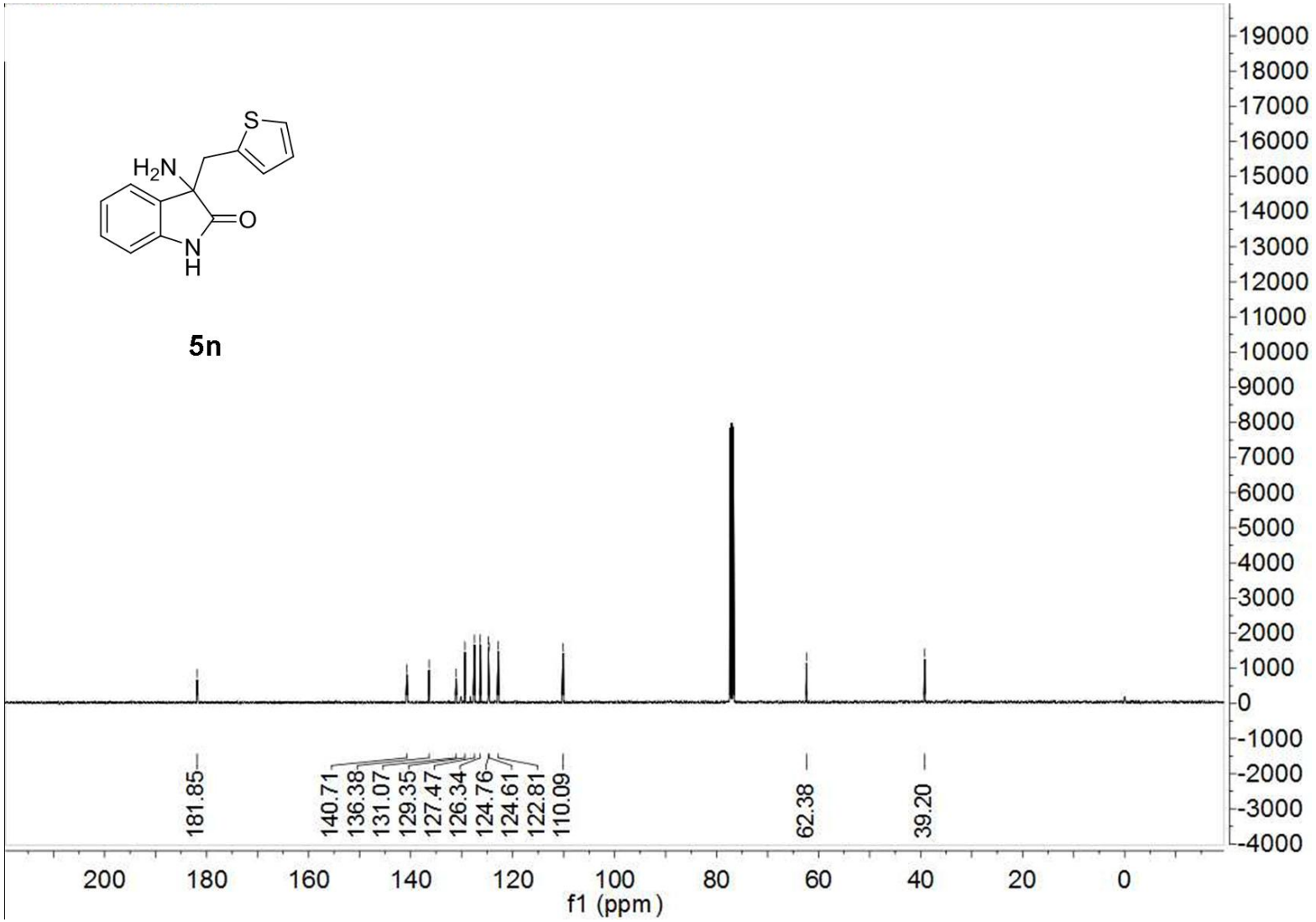

10.03
8.09
7.78
7.76
7.68
7.66
7.38
7.37
7.36
7.36
7.23
7.21
7.20
7.18
7.11
7.09
7.01
6.79
6.57
6.55
6.53
2.32
H2N
O
N
H
5l
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
1.02
1.00
1.05
1.05
2.14
2.05
1.04
1.09
1.02
1.04
2.10
2.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

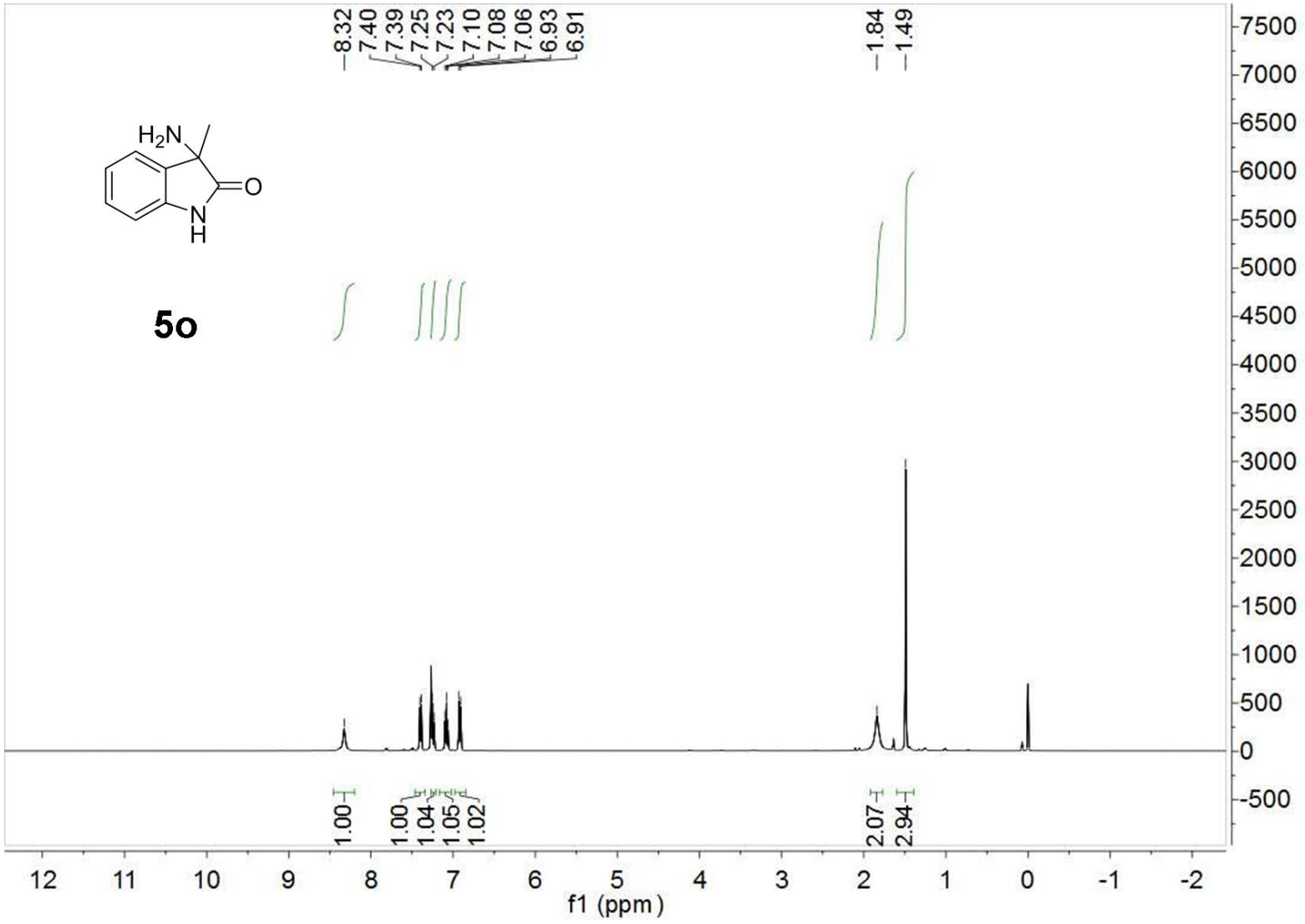

H2N
O
N
H
5l
181.70
142.04
133.58
132.96
132.76
132.63
128.64
128.55
127.41
125.59
125.58
125.21
125.16
121.39
109.52
63.05
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

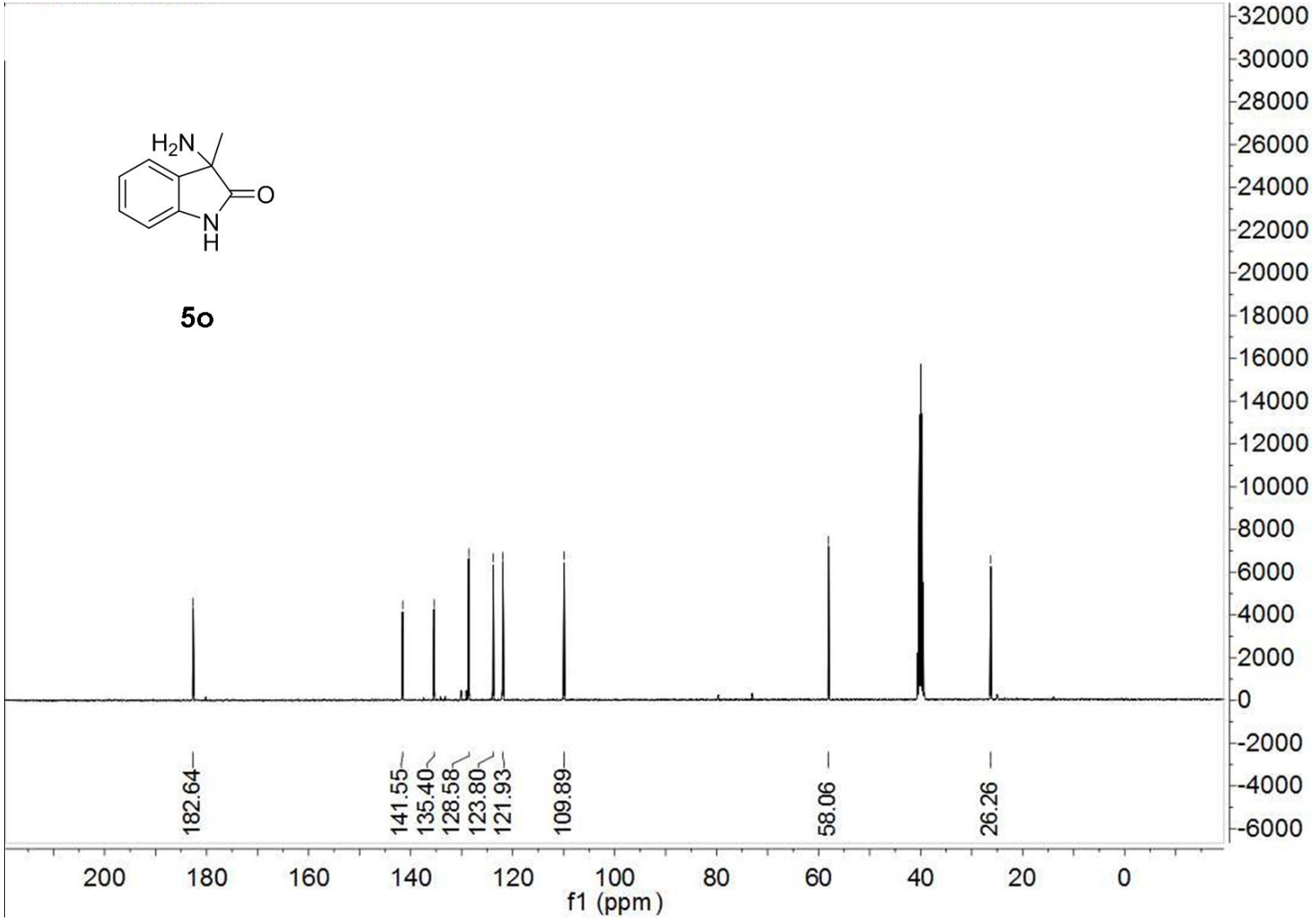

9.10
7.20
7.15
7.13
7.03
7.01
6.85
6.84
6.78
5.90
5.90
3.22
3.18
3.11
3.07
2.04
H2N
O
O
N
H
5m
0.95
2.03
0.97
1.03
1.00
0.99
1.02
2.14
2.11
f1 (ppm)

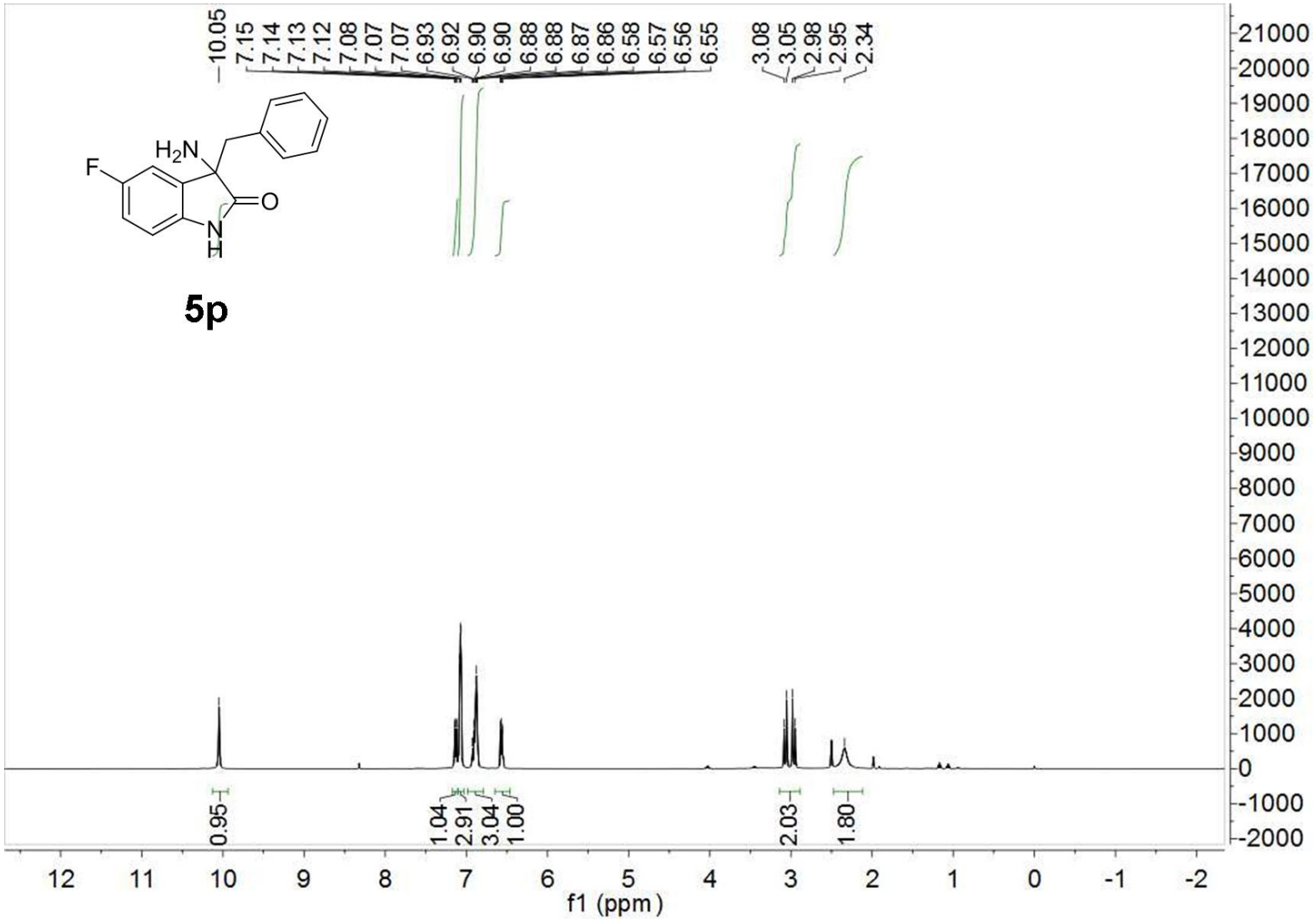

O
H2N
O
N
H
5m
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
182.21
149.98
141.77
140.47
131.56
129.09
124.41
122.66
110.29
110.20
108.38
61.43
37.28
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


7.60
7.54
7.49
7.31
7.29
7.24
7.22
7.10
7.08
7.06
7.04
7.02
6.79
6.78
6.76
6.63
3.39
3.35
3.31
1.86
S
H2N
O
N
H
5n
1.06
0.94
0.97
1.03
0.99
2.00
1.14
1.95
2.29
f1 (ppm)

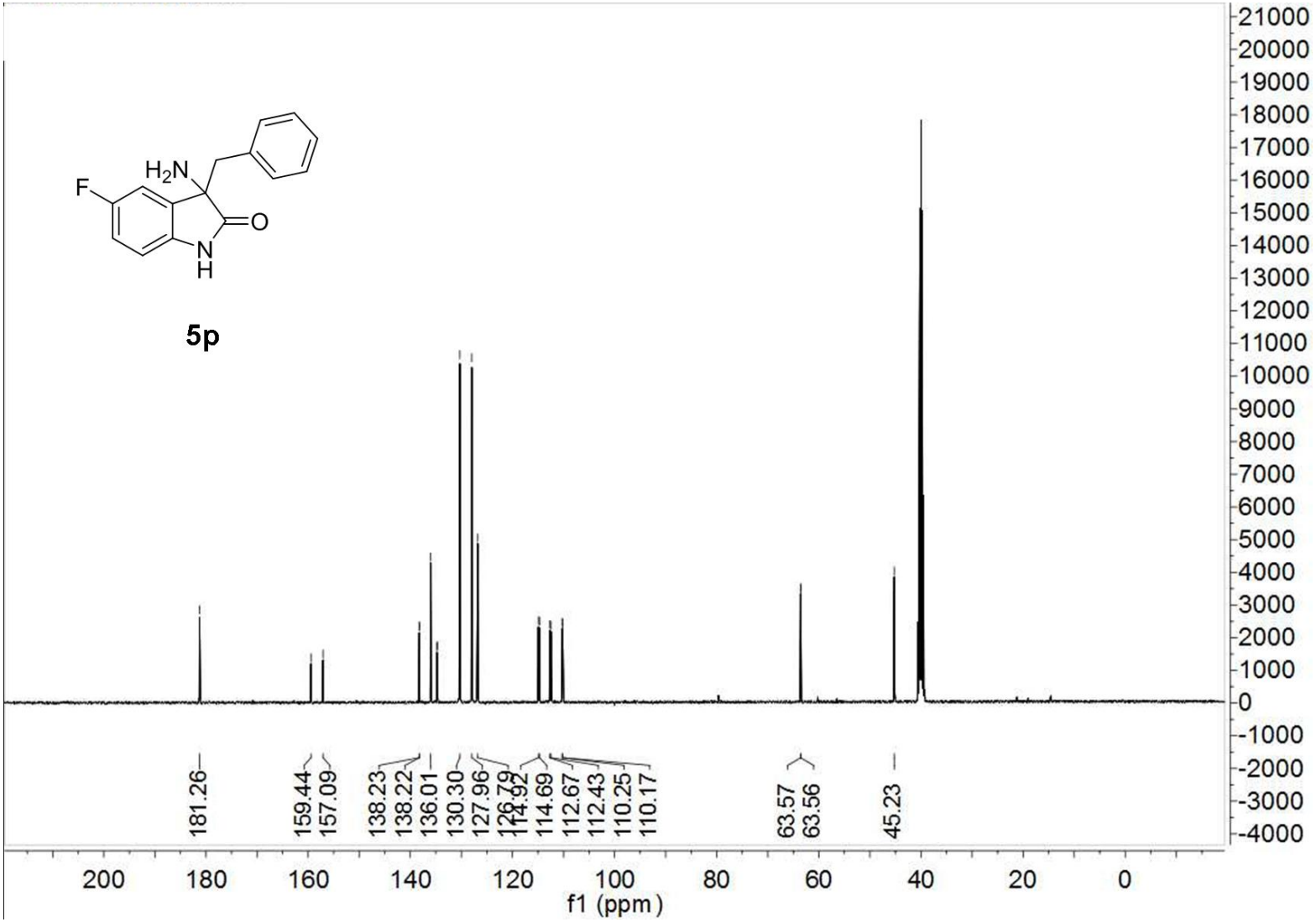

S
H2N
O
N
H
5n
181.85
140.71
136.38
131.07
129.35
127.47
126.34
124.76
124.61
122.81
110.09
62.38
39.20
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000

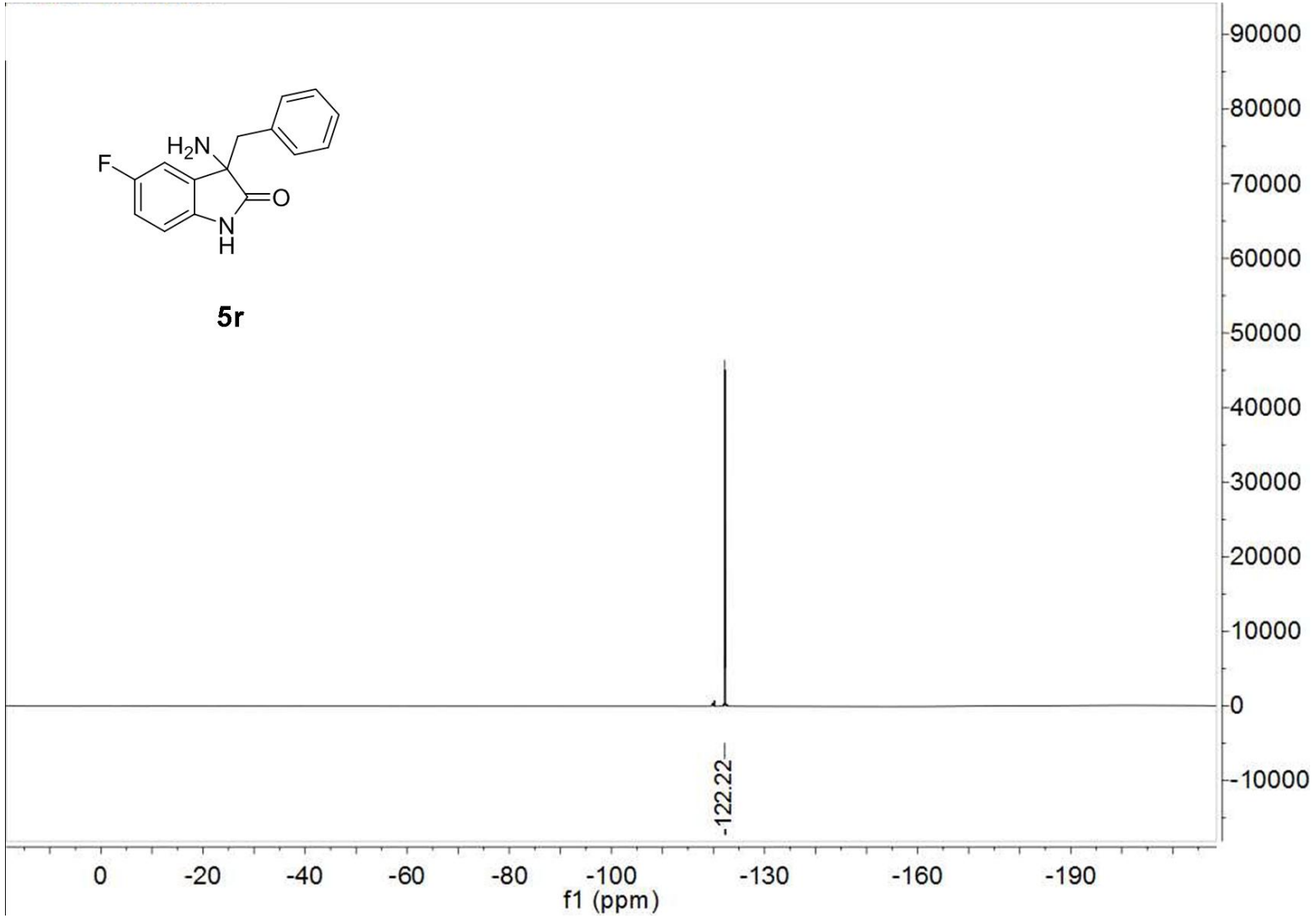

8.32
7.40
7.39
7.25
7.23
7.10
7.08
7.06
6.93
6.91
1.84
1.49
H2N
O
N
H
5o
1.00
1.00
1.04
1.05
1.02
2.07
2.94
f1 (ppm)

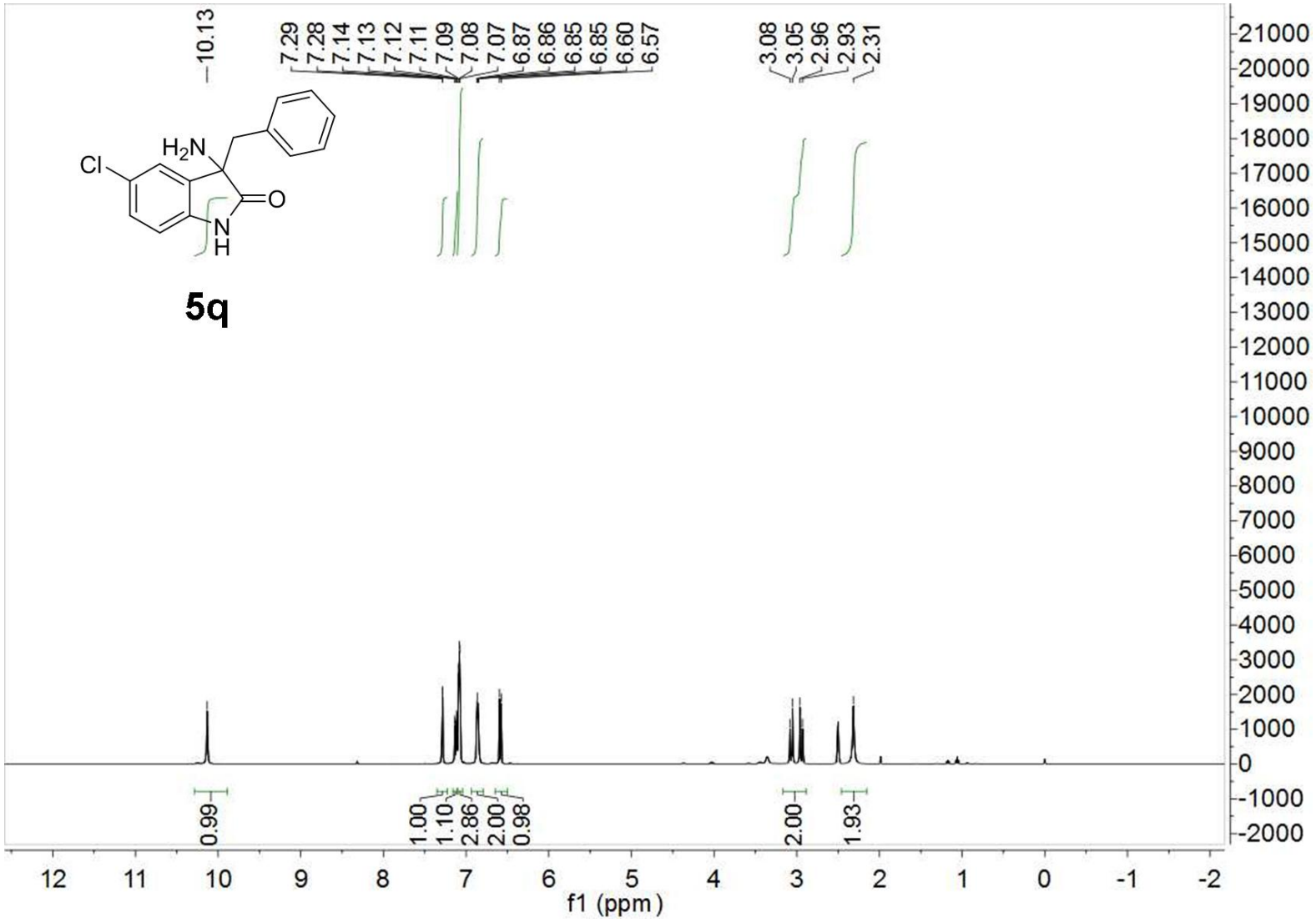

H2N
O
N
H
5o
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
182.64
141.55
135.40
128.58
123.80
121.93
109.89
58.06
26.26
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

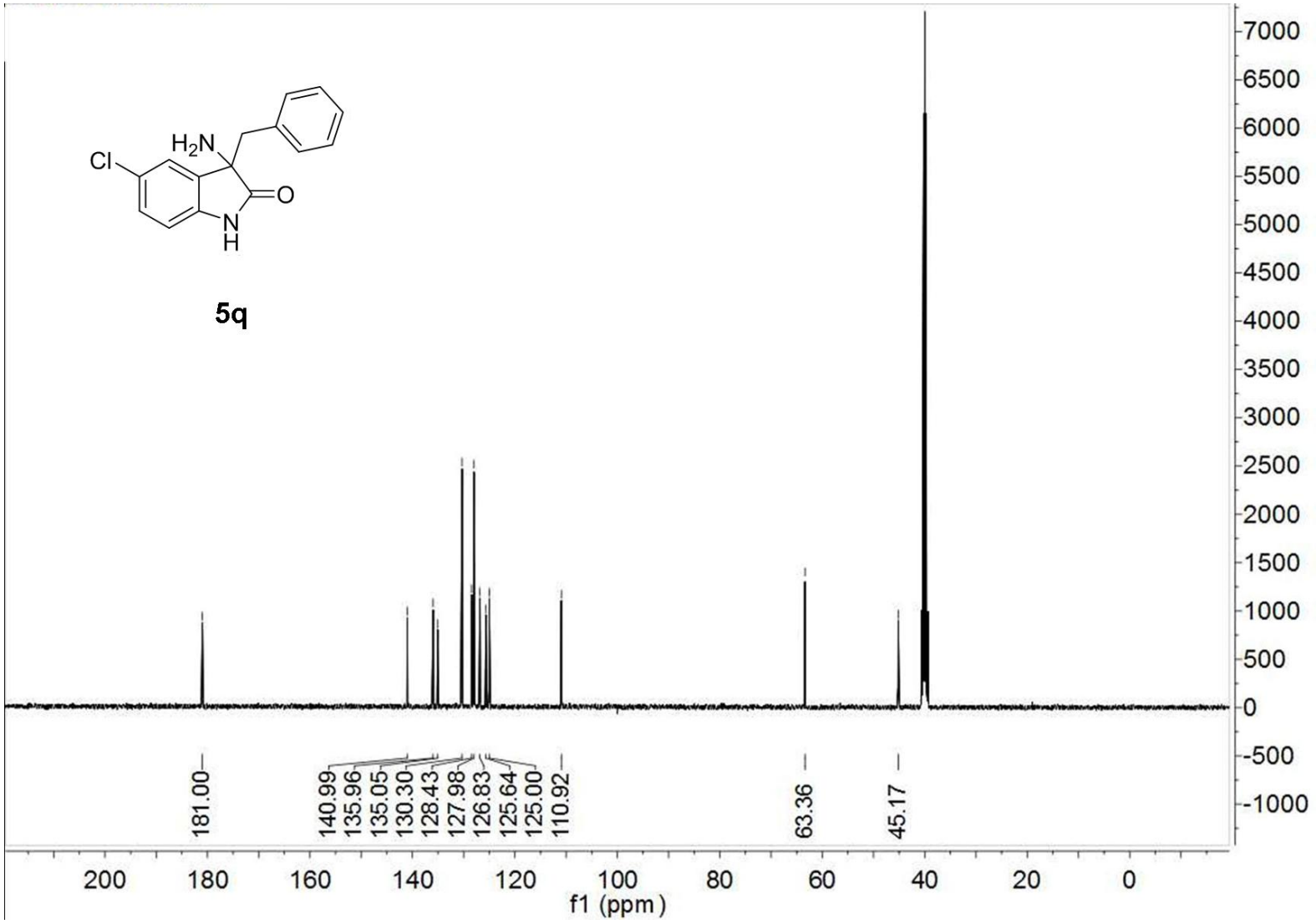

10.05
7.15
7.14
7.13
7.12
7.08
7.07
7.07
6.93
6.92
6.90
6.90
6.88
6.88
6.87
6.86
6.58
6.57
6.56
6.55
3.08
3.05
2.98
2.95
2.34
H2N
F
O
N
H
0.95
1.04
2.91
3.04
1.00
2.03
1.80
f1 (ppm)

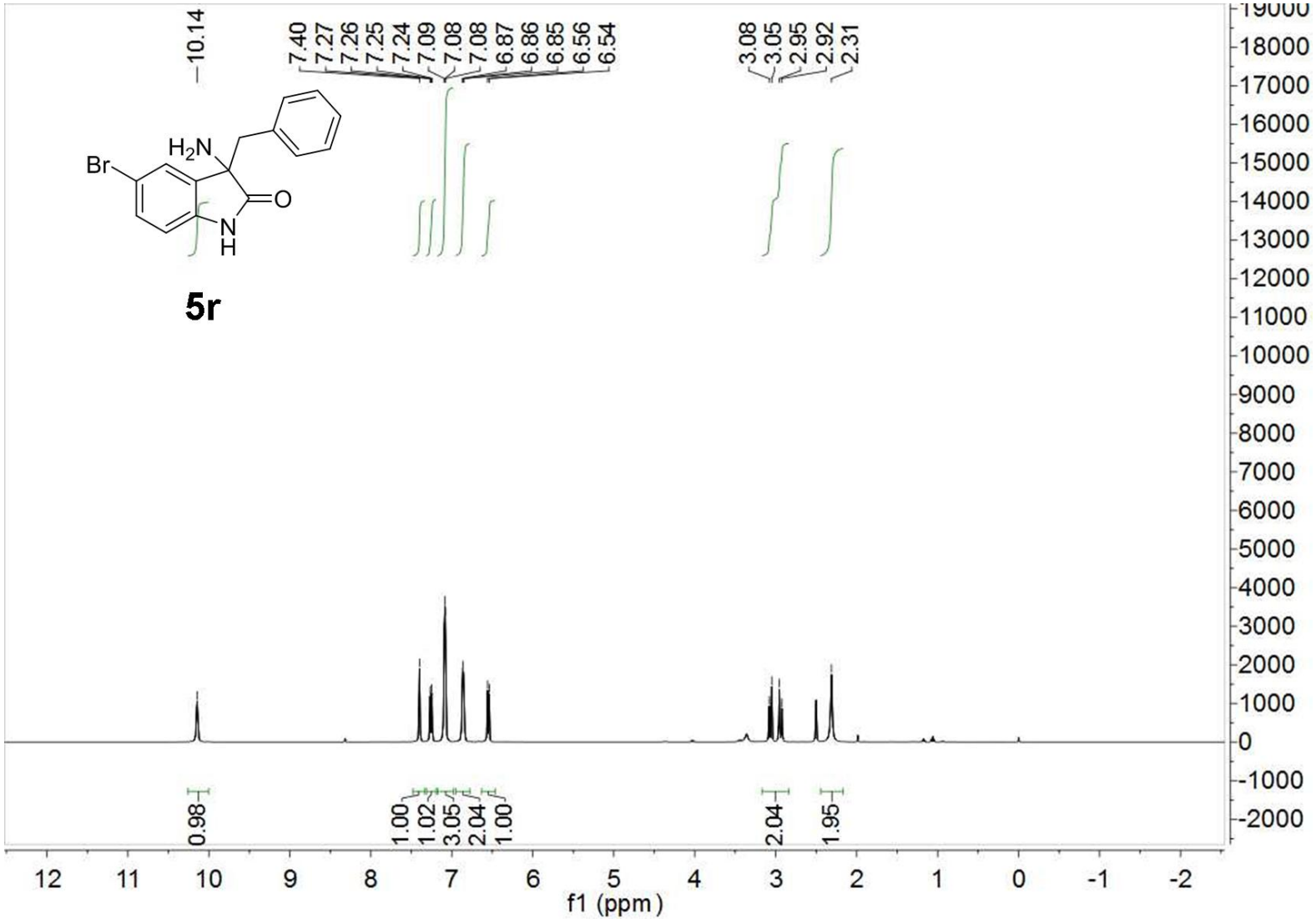
5p

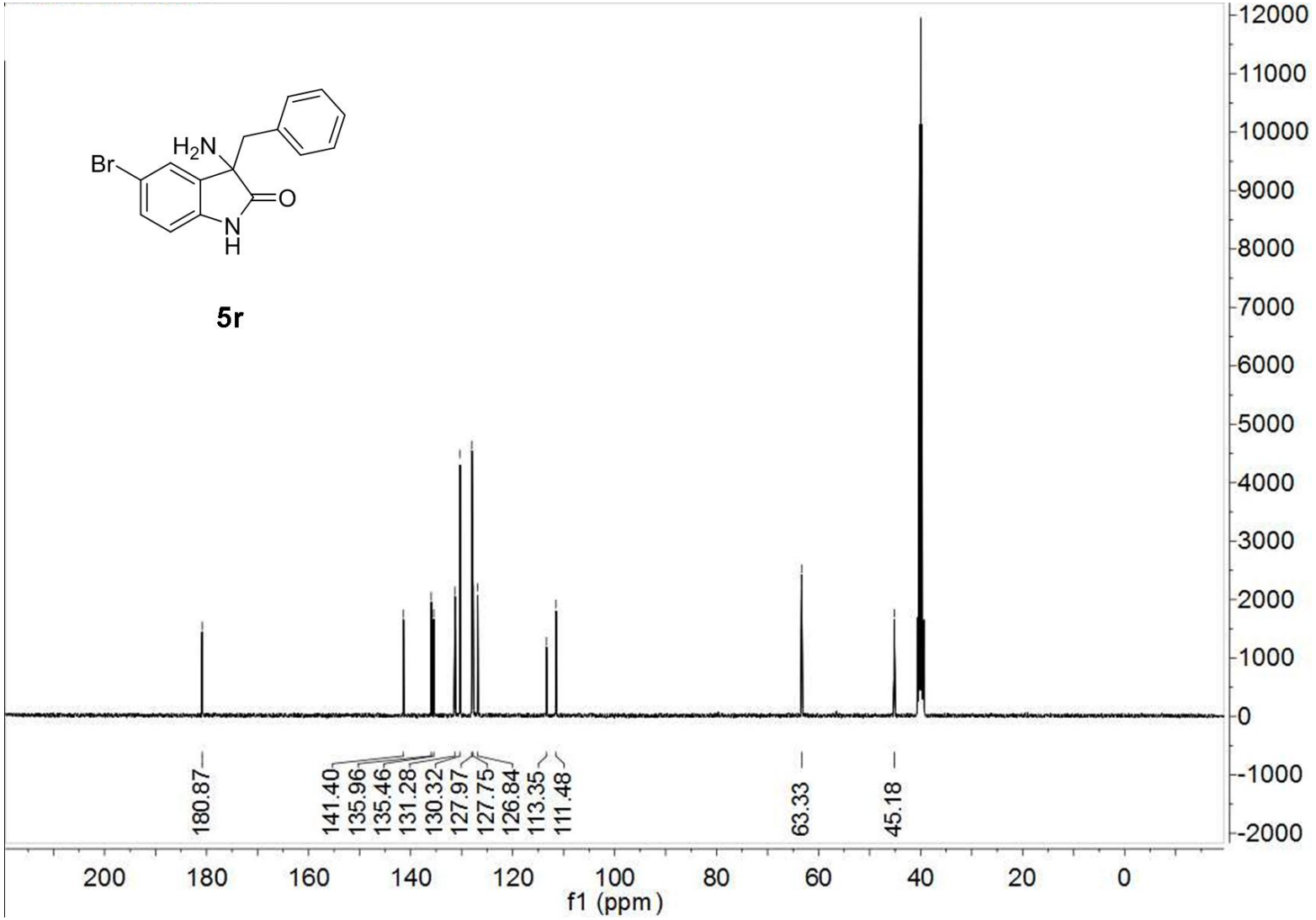

F
H2N
O
N
H
5p
181.26
159.44
157.09
138.23
138.22
136.01
130.30
127.96
126.79
114.92
114.69
112.67
112.43
110.25
110.17
63.57
63.56
45.23
f1 (ppm)

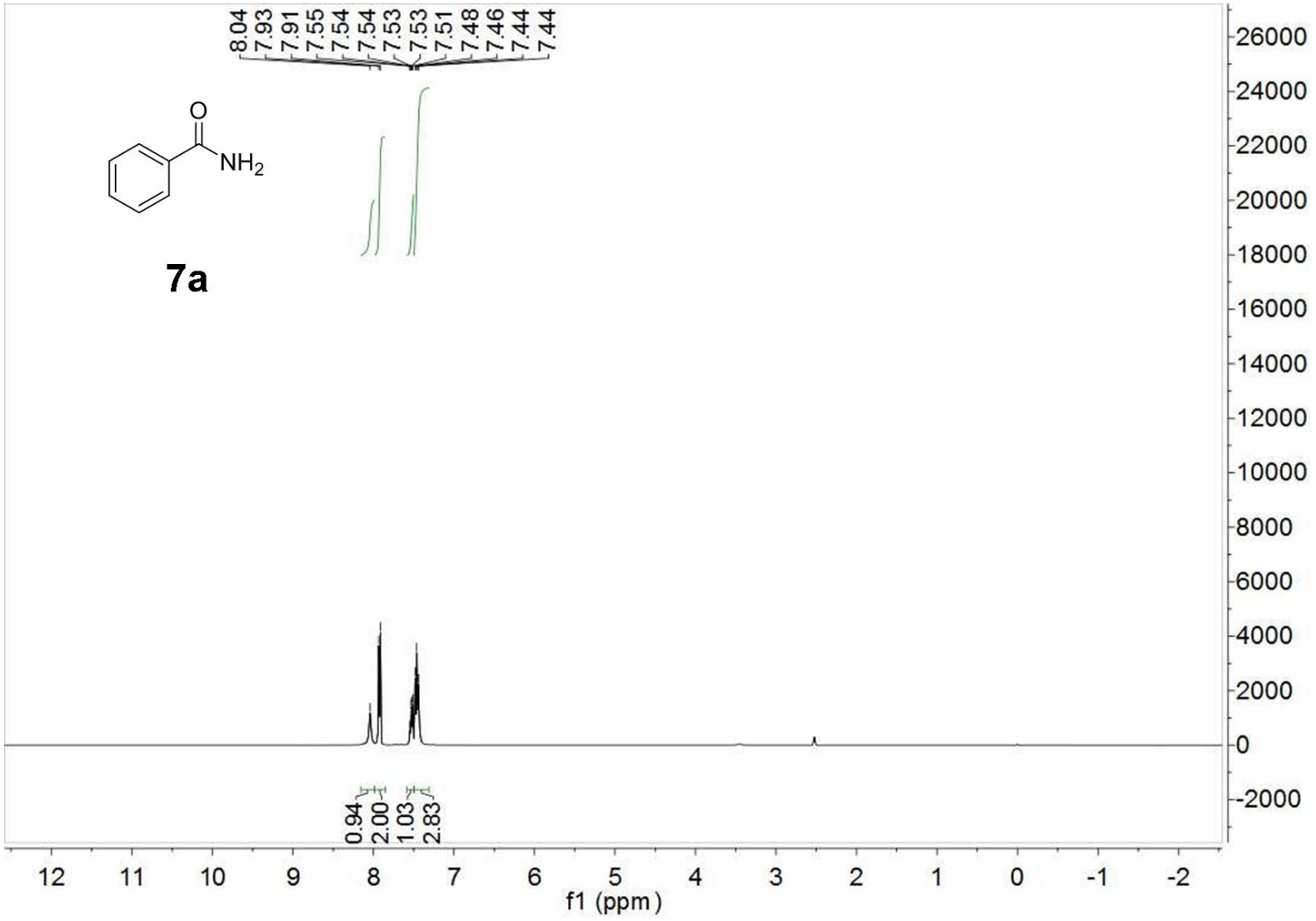

F
H2N
O
N
H
5r
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-122.22
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

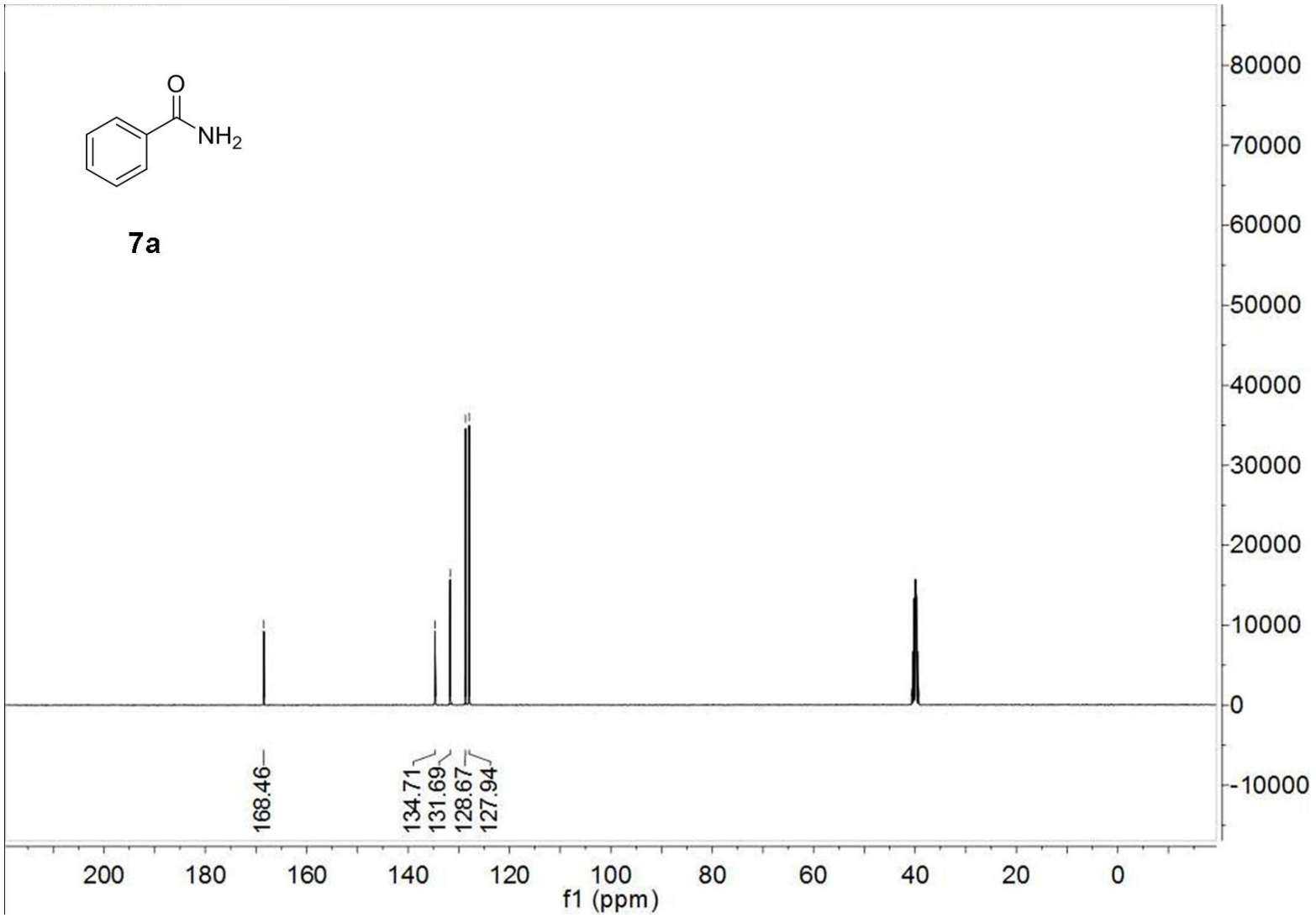

10.13
7.29
7.28
7.14
7.13
7.12
7.11
7.09
7.08
7.07
6.87
6.86
6.85
6.85
6.60
6.57
3.08
3.05
2.96
2.93
2.31
Cl
H2N
O
N
H
5q
0.99
1.00
1.10
2.86
2.00
0.98
2.00
1.93
f1 (ppm)

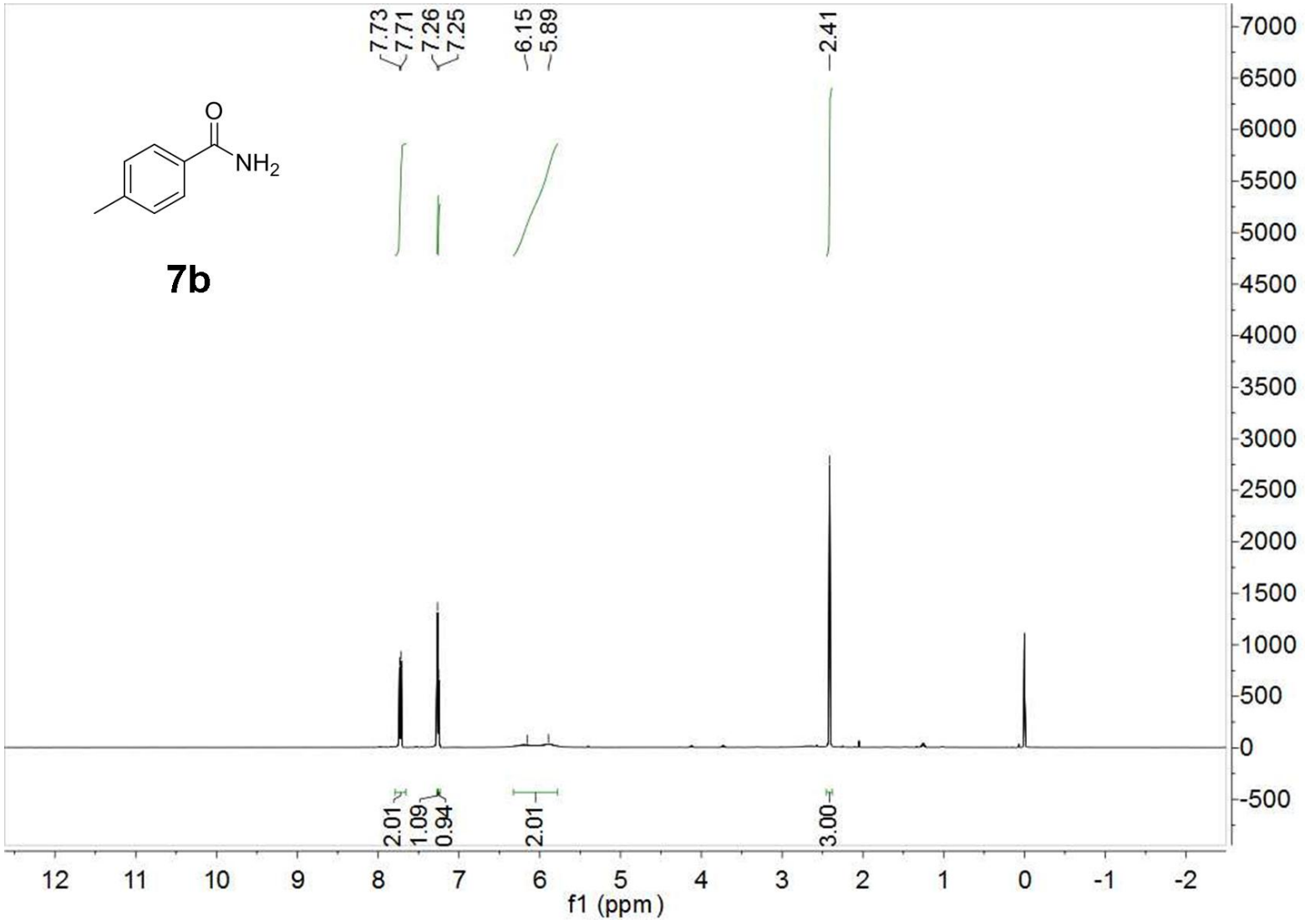

Cl
H2N
O
N
H
5q
181.00
140.99
135.96
135.05
130.30
128.43
127.98
126.83
125.64
125.00
110.92
63.36
45.17
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000

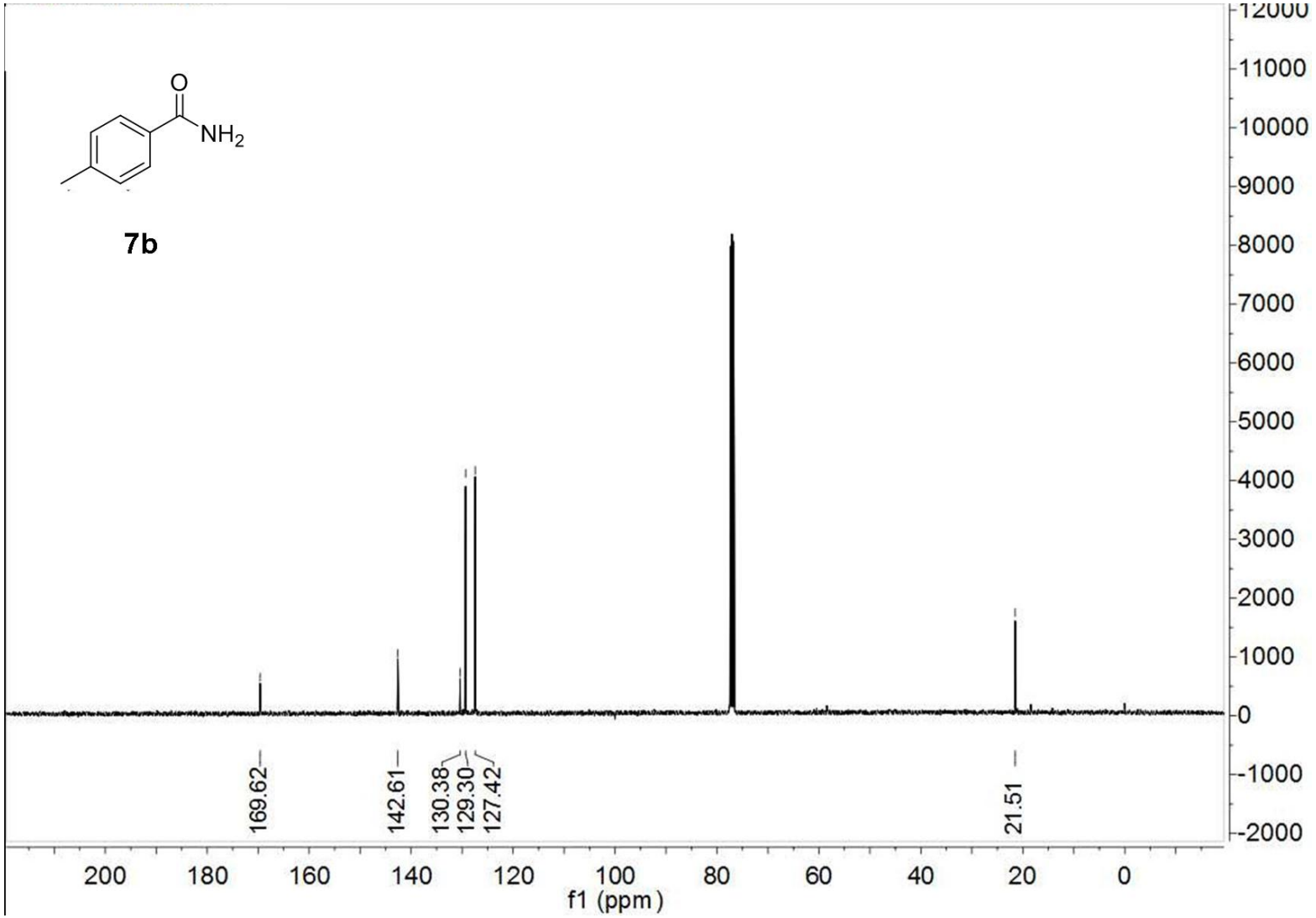

10.14
7.40
7.27
7.26
7.25
7.24
7.09
7.08
7.08
6.87
6.86
6.85
6.56
6.54
3.08
3.05
2.95
2.92
2.31
Br
H2N
O
N
H
5r
0.98
1.00
1.02
3.05
2.04
1.00
2.04
1.95
f1 (ppm)

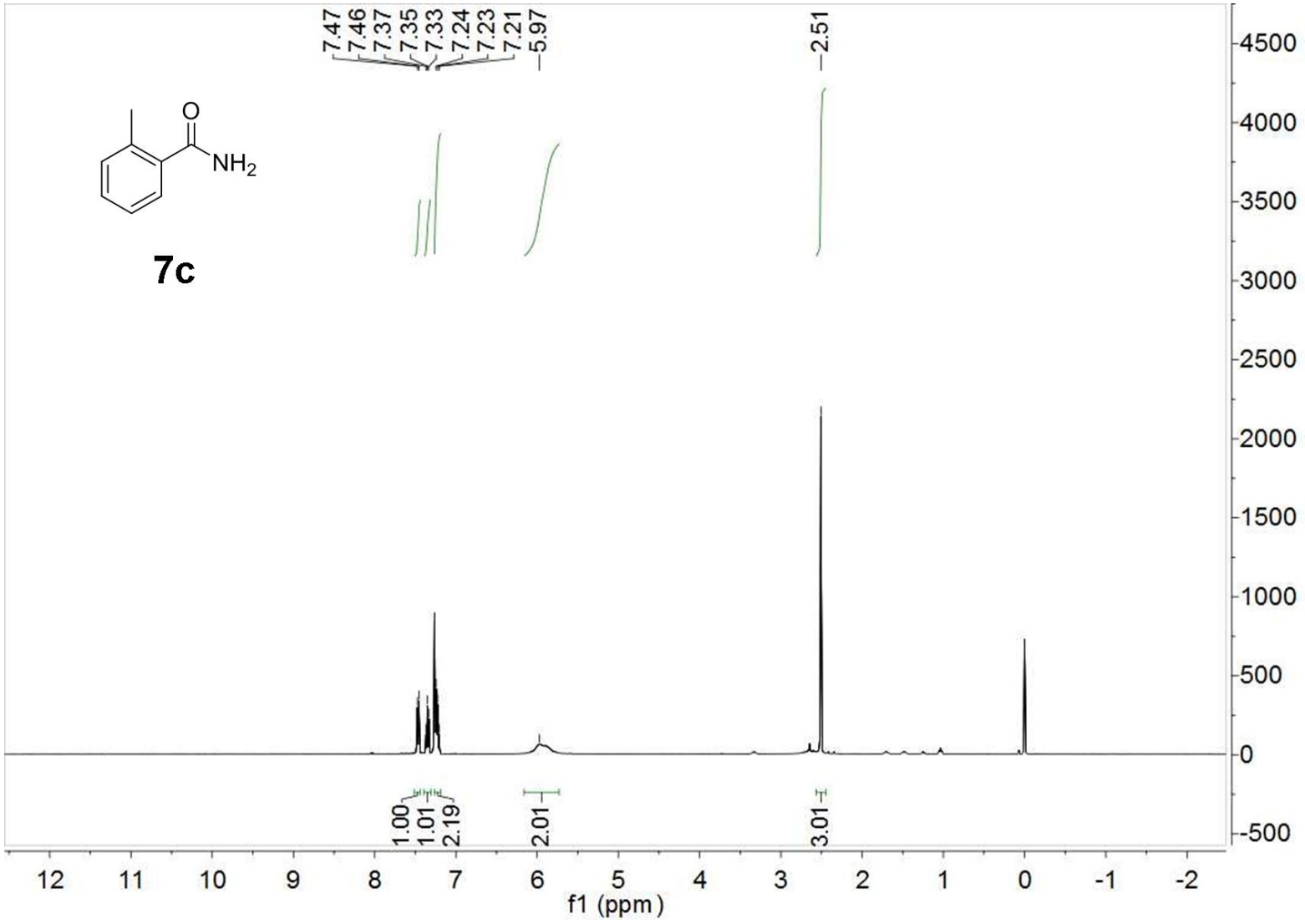

Br
H2N
O
N
H
5r
180.87
141.40
135.96
135.46
131.28
130.32
127.97
127.75
126.84
113.35
111.48
63.33
45.18
f1 (ppm)

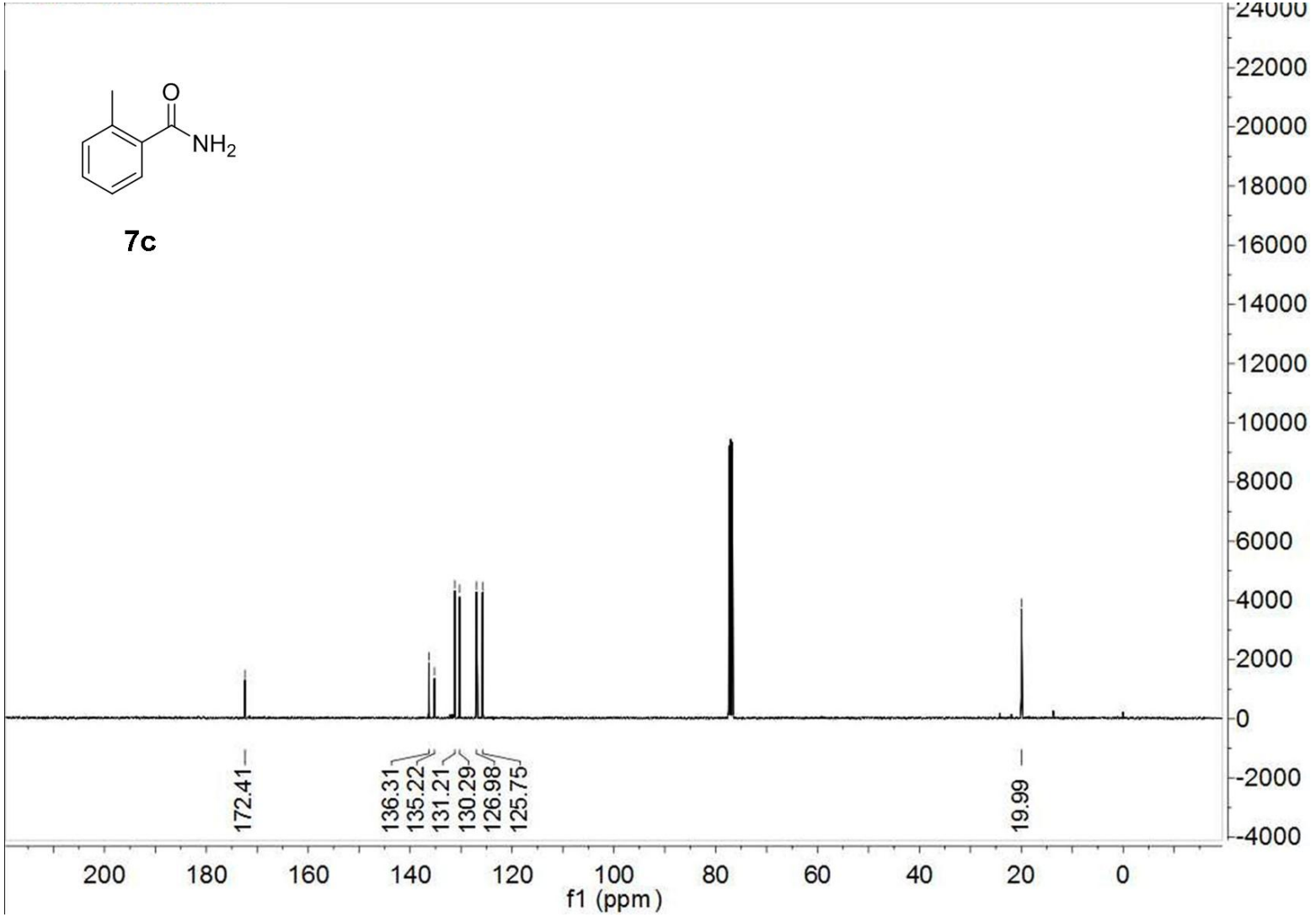

8.04
7.93
7.91
7.55
7.54
7.54
7.53
7.53
7.51
7.48
7.46
7.44
7.44
O
NH2
7a
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
0.94
2.00
1.03
2.83
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

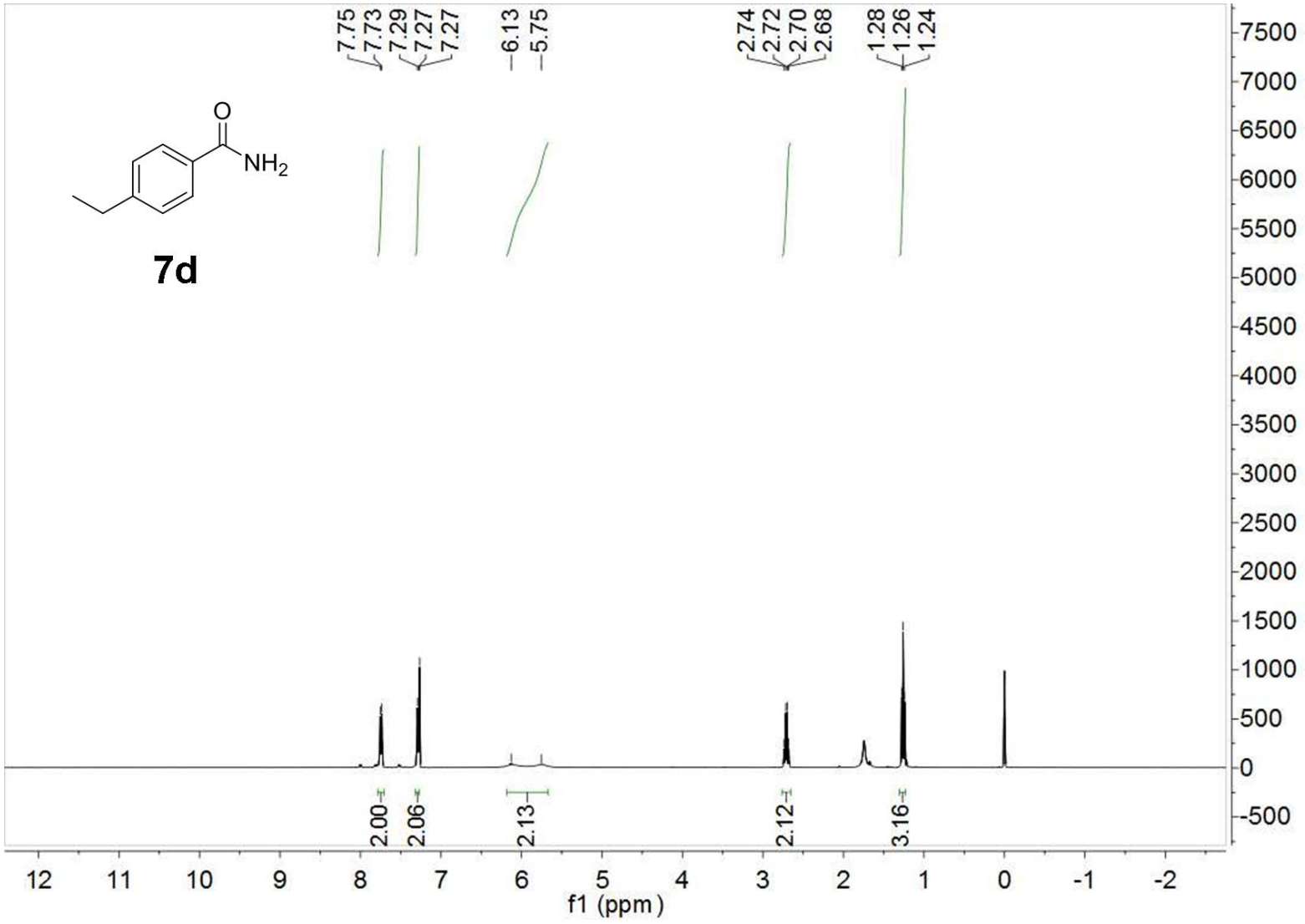

O
NH2
7a
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
168.46
134.71
131.69
128.67
127.94
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

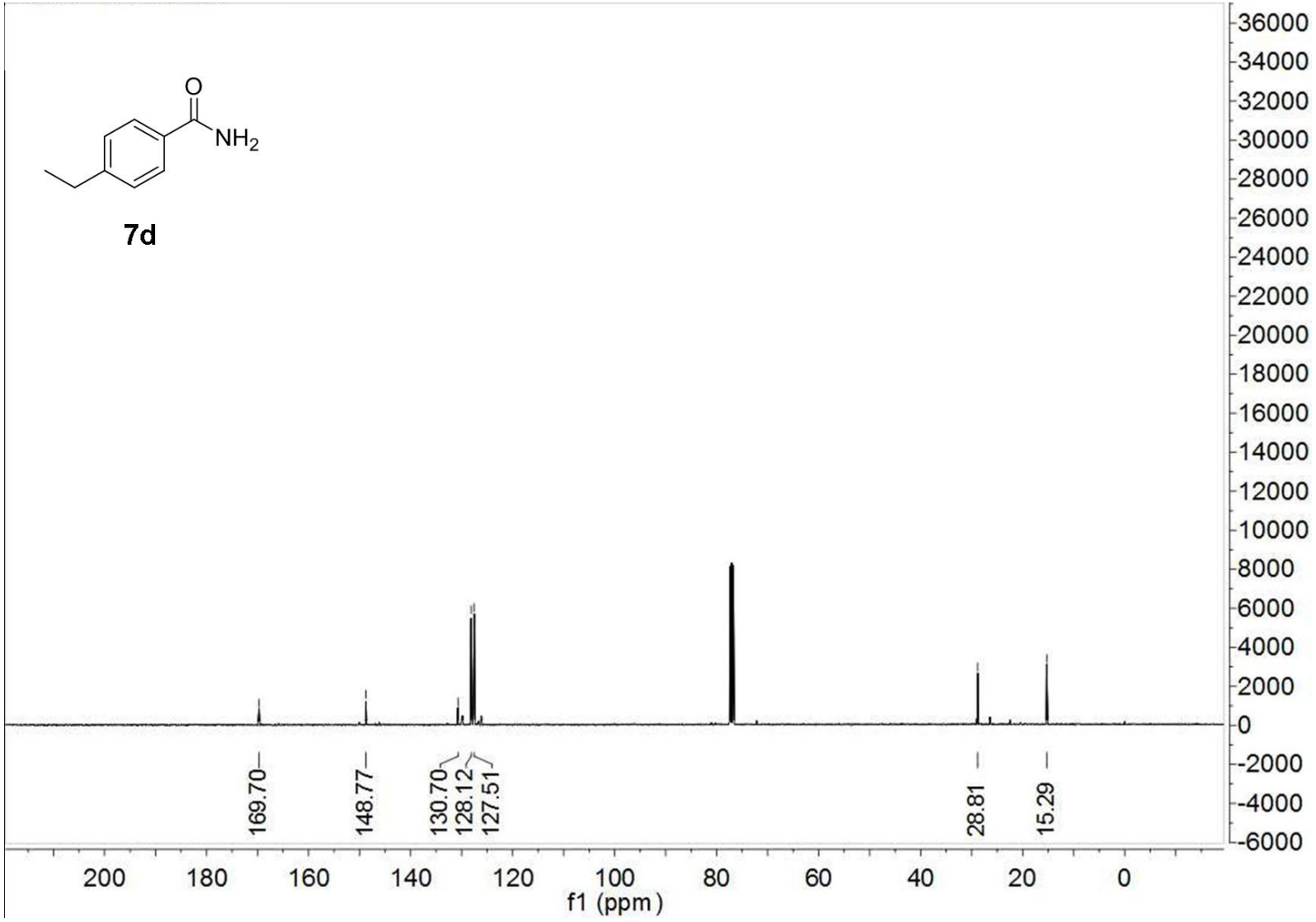

7.73
7.71
7.26
7.25
6.15
5.89
2.41
O
NH2
7b
2.01
1.09
0.94
2.01
3.00
f1 (ppm)

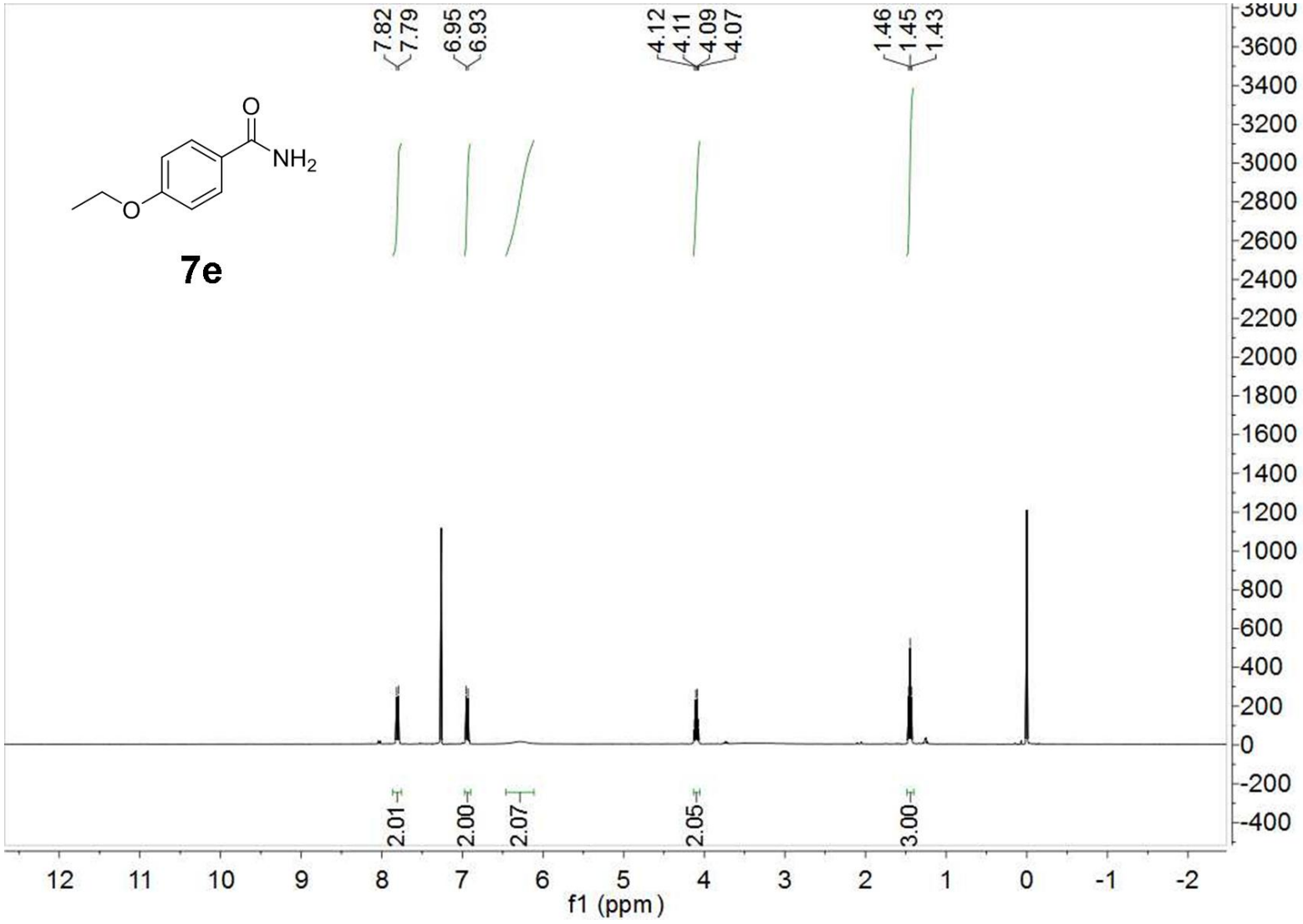

O
NH2
7b
169.62
142.61
130.38
129.30
127.42
21.51
f1 (ppm)

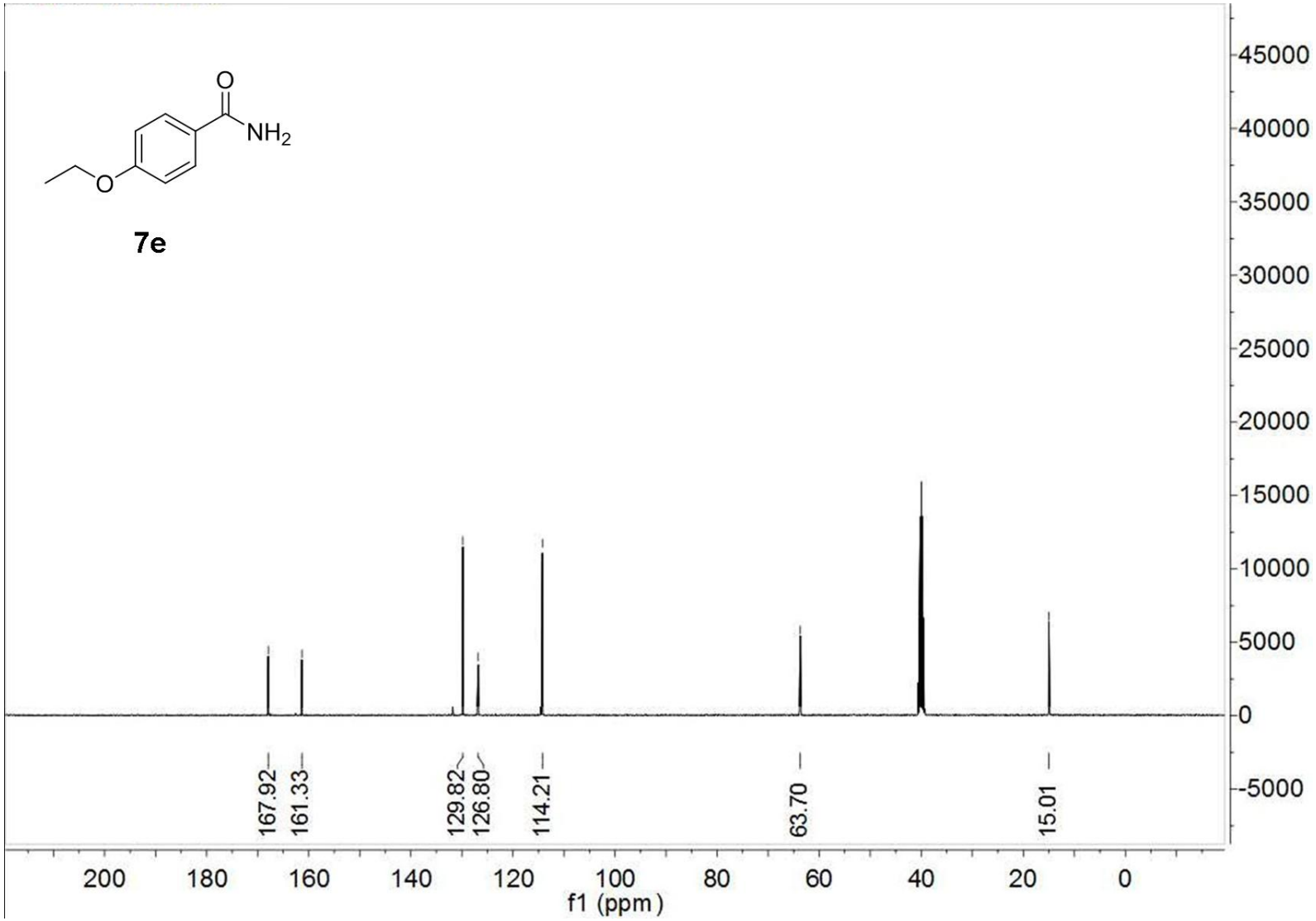

7.47
7.46
7.37
7.35
7.33
7.24
7.23
7.21
5.97
2.51
O
NH2
7c
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
1.00
1.01
2.19
2.01
3.01
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

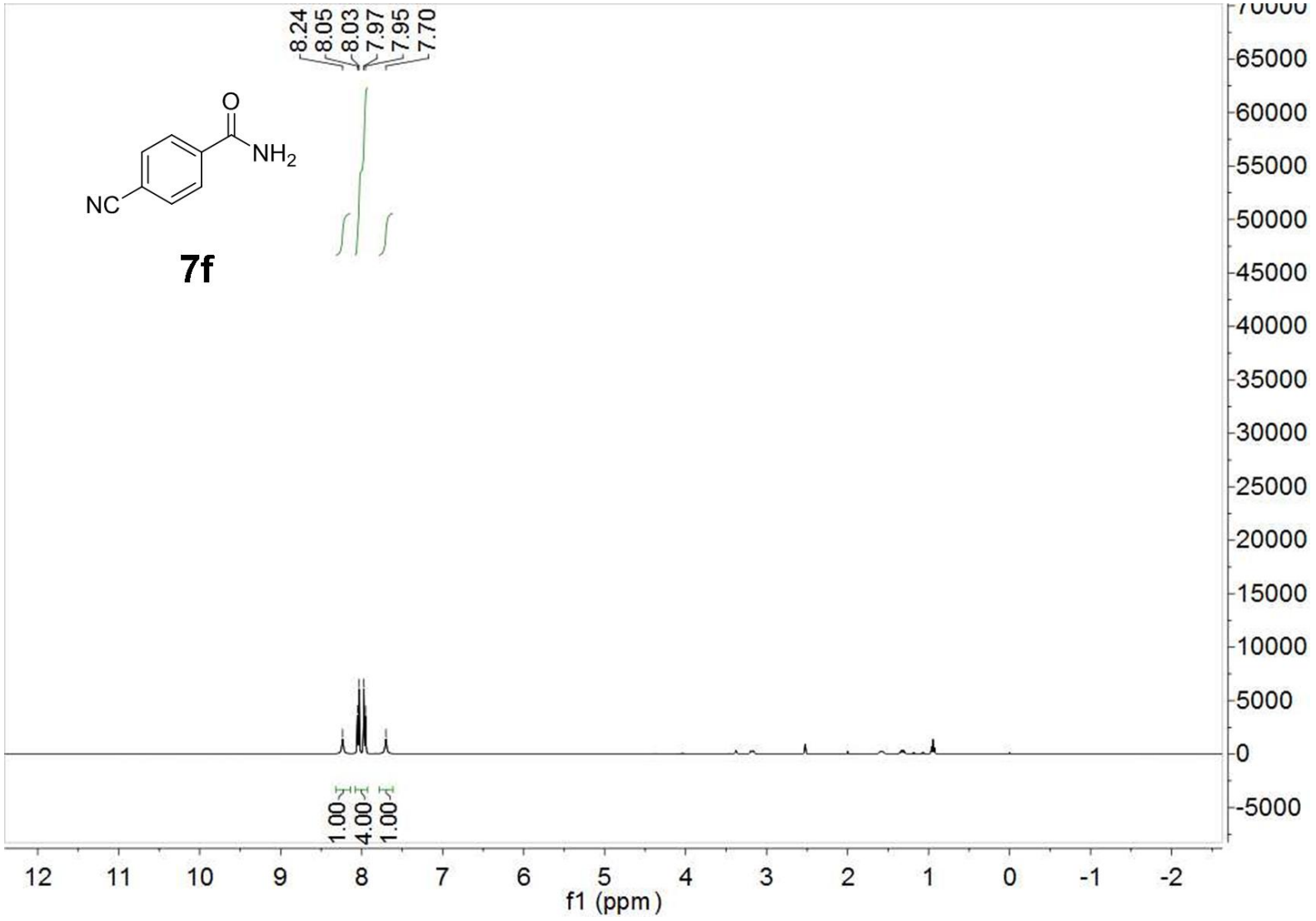

O
NH2
7c
172.41
136.31
135.22
131.21
130.29
126.98
125.75
19.99
f1 (ppm)

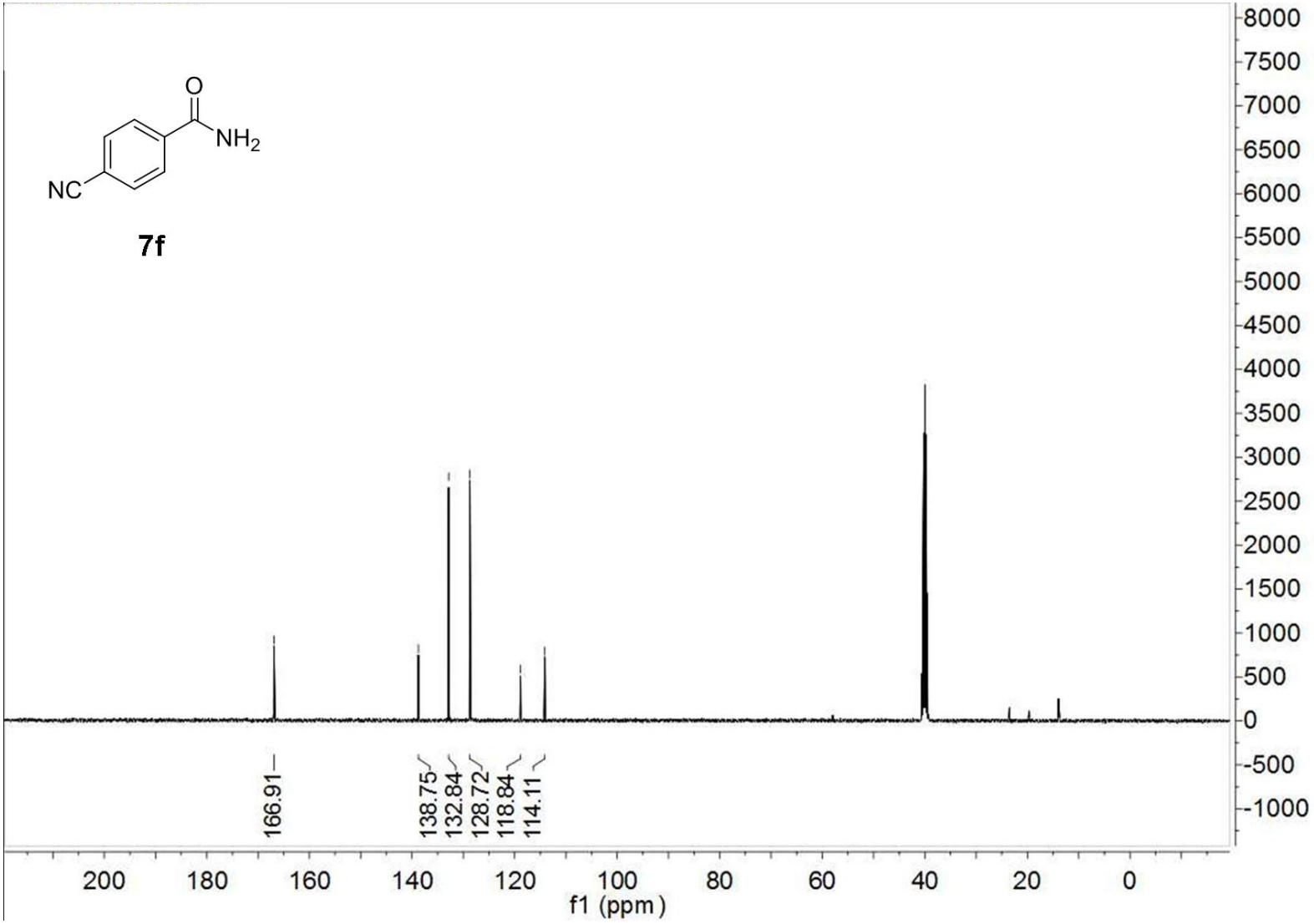

7.75
7.73
7.29
7.27
7.27
6.13
5.75
2.74
2.72
2.70
2.68
1.28
1.26
1.24
O
NH2
7d
2.00
2.06
2.13
2.12
3.16
f1 (ppm)

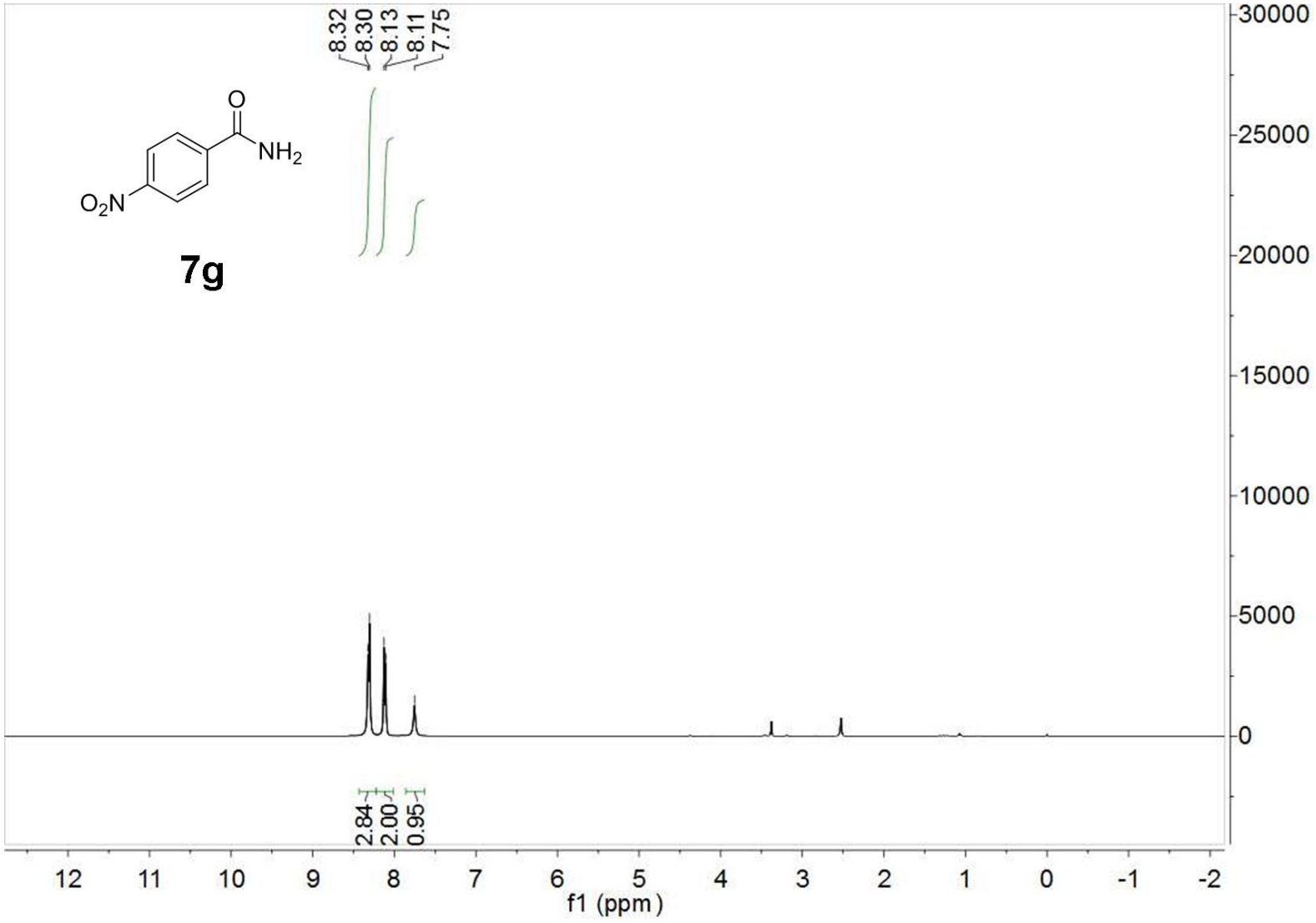

O
NH2
7d
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
169.70
148.77
130.70
128.12
127.51
28.81
15.29
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

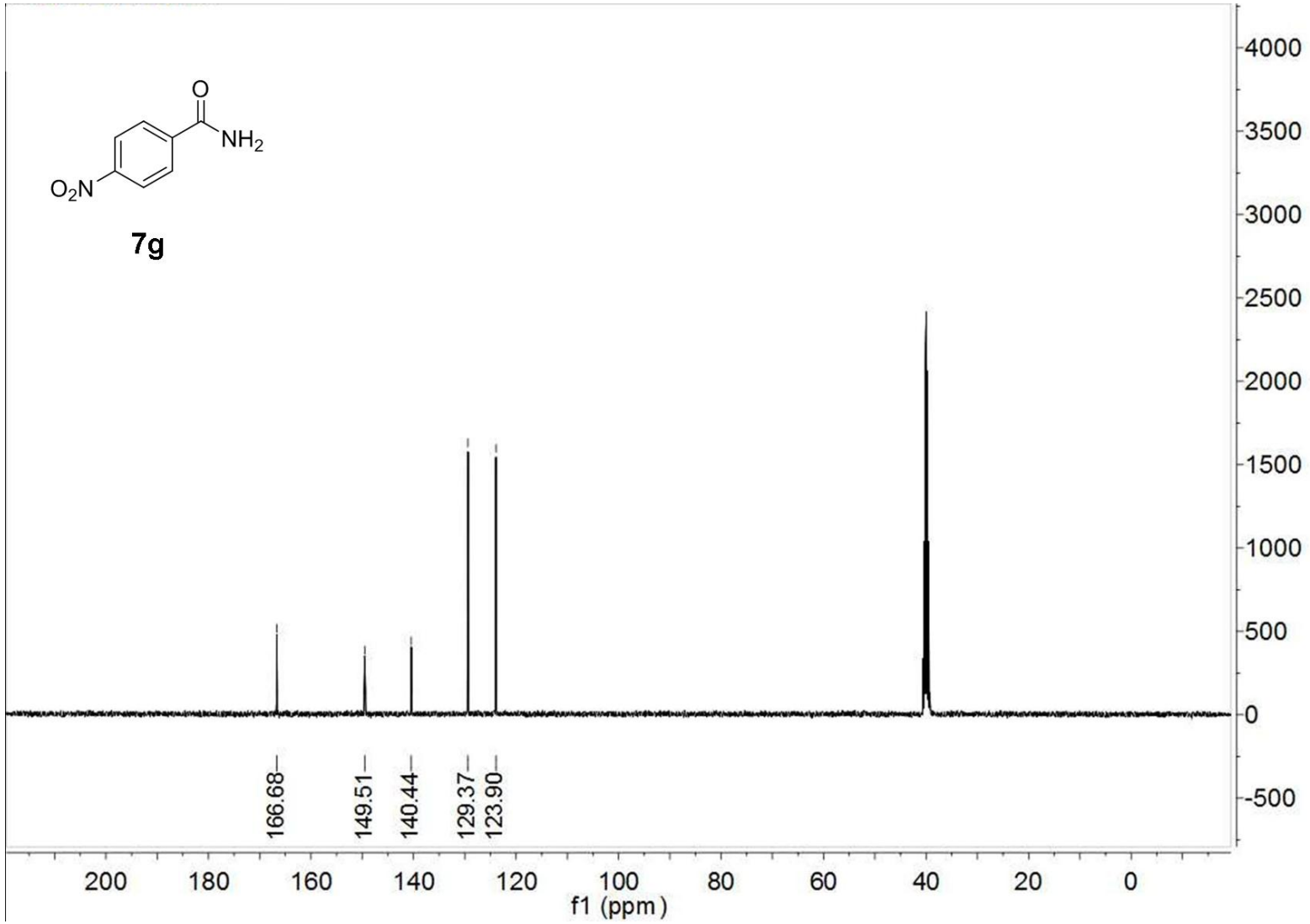

7.82
7.79
6.95
6.93
4.12
4.11
4.09
4.07
1.46
1.45
1.43
O
NH2
O
7e
2.01
2.00
2.07
2.05
3.00
f1 (ppm)

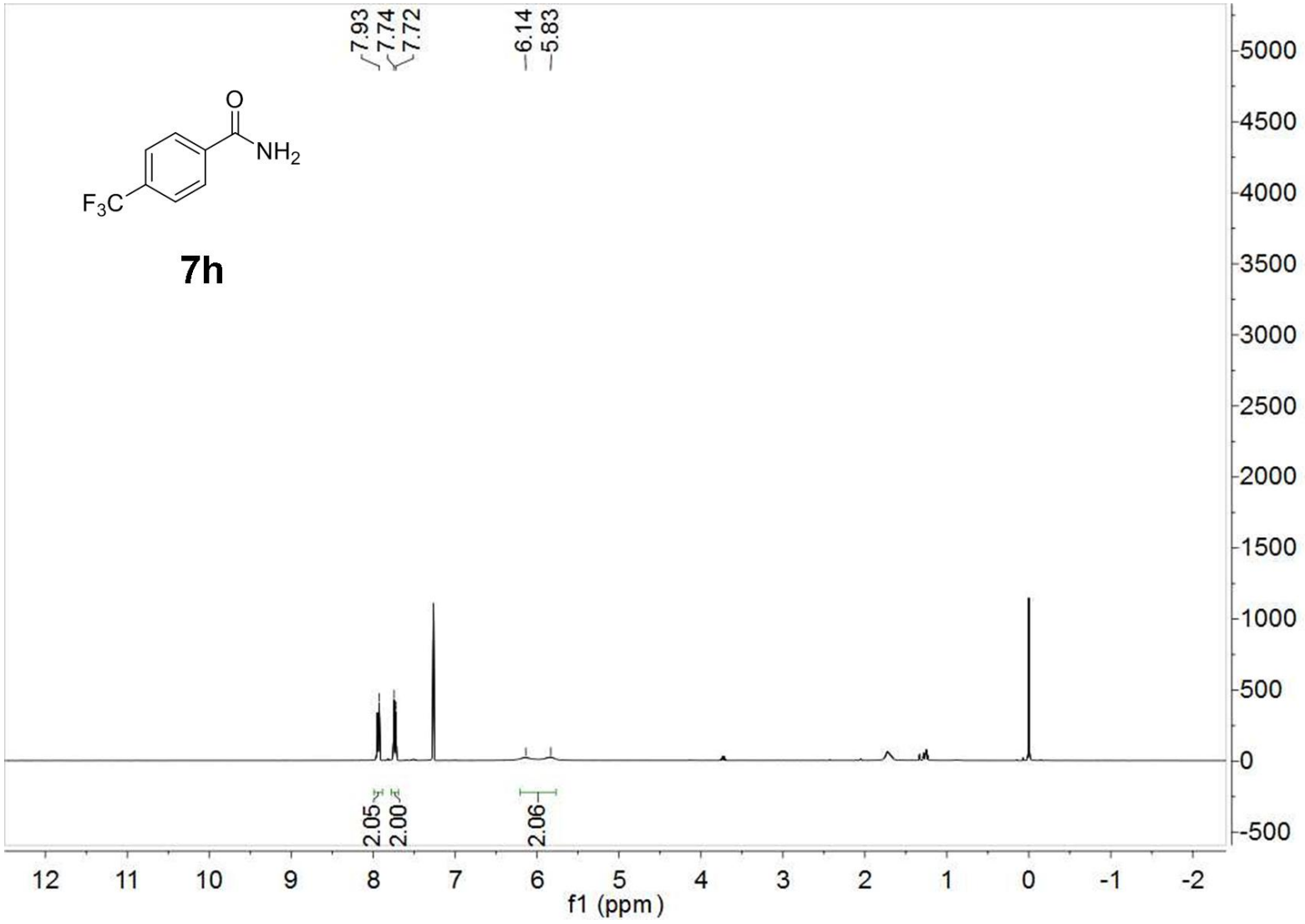

O
NH2
O
7e
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
167.92
161.33
129.82
126.80
114.21
63.70
15.01
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

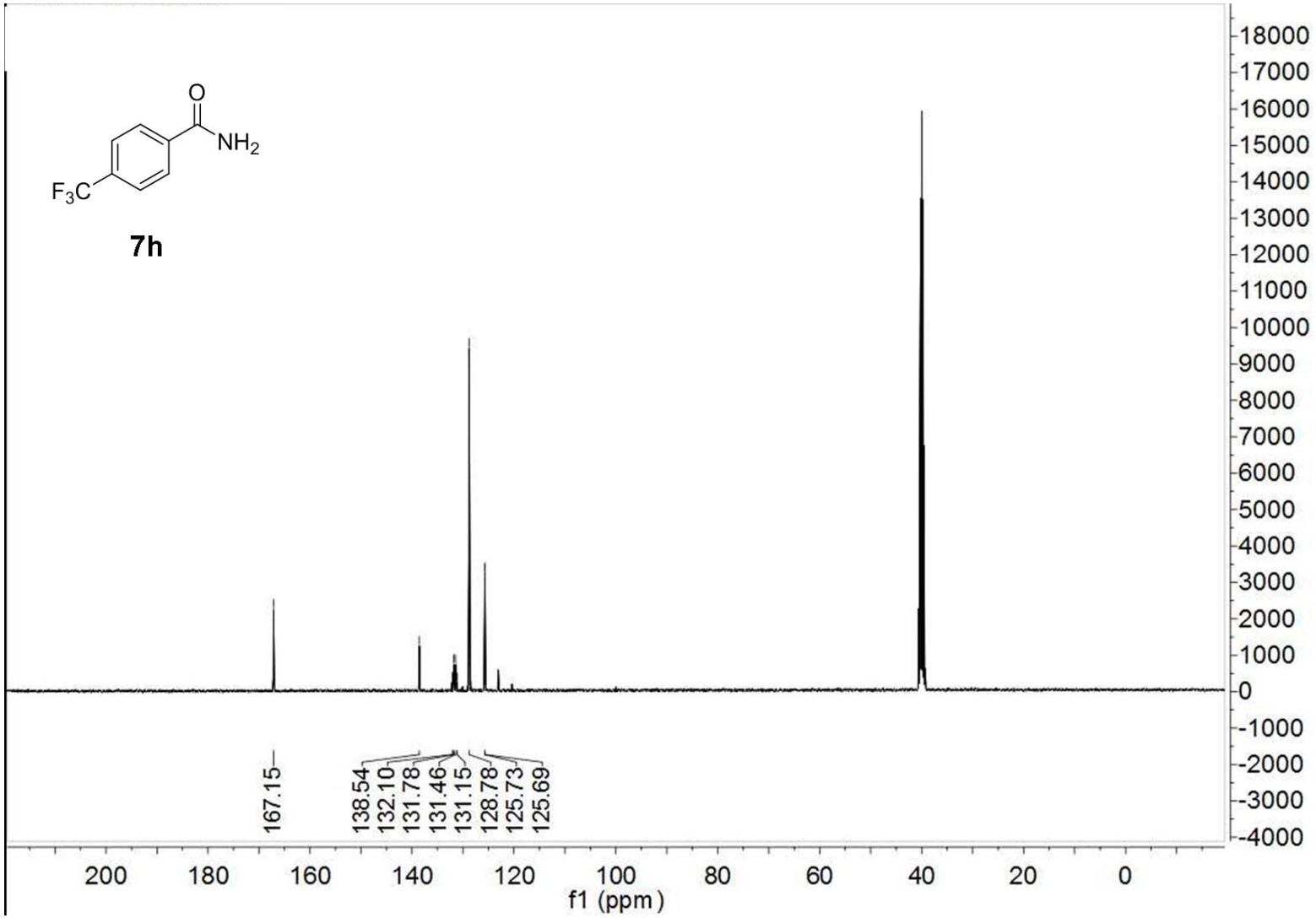

8.24
8.05
8.03
7.97
7.95
7.70
O
NH2
NC
7f
1.00
4.00
1.00
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000

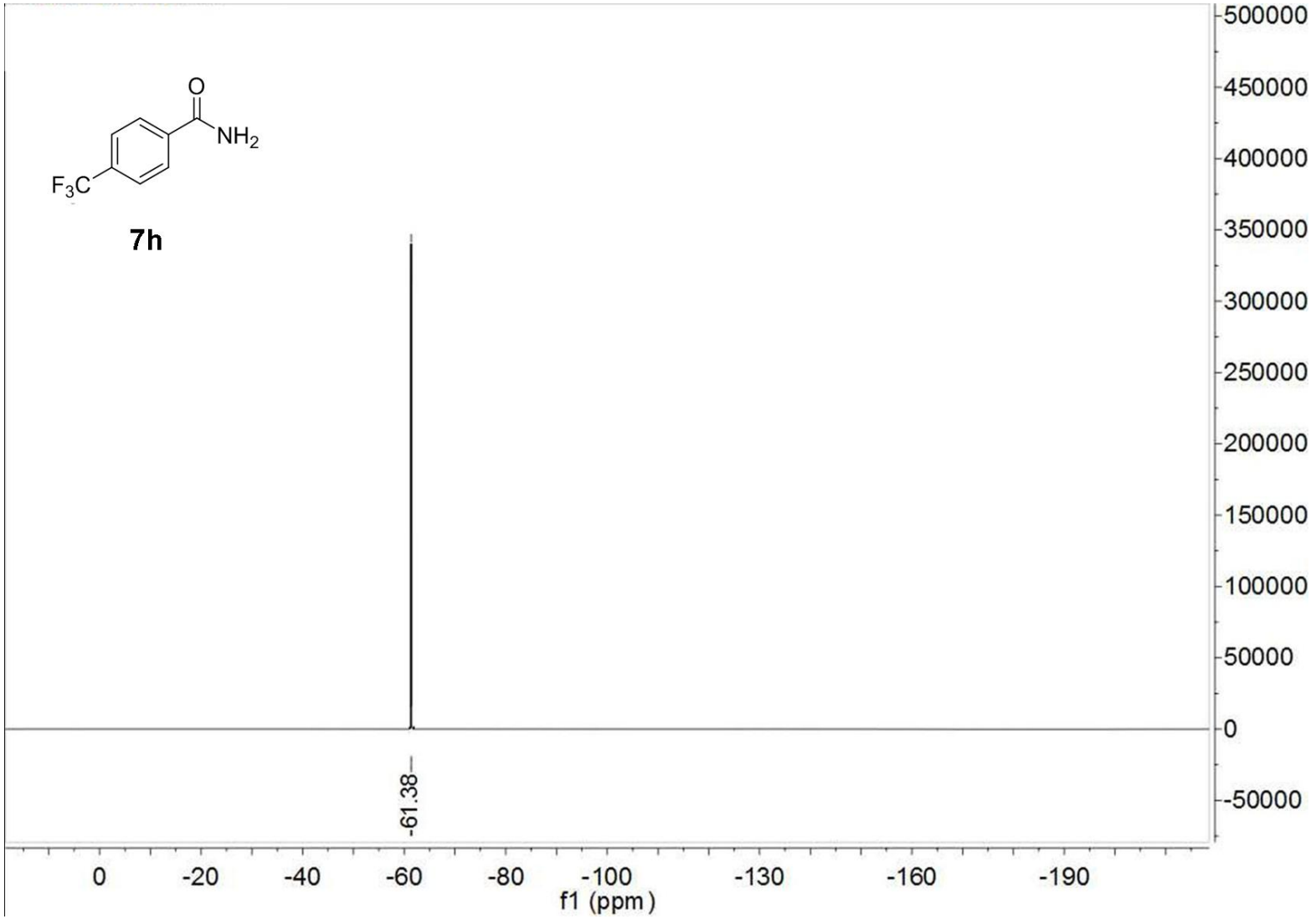

O
NH2
NC
7f
166.91
138.75
132.84
128.72
118.84
114.11
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000

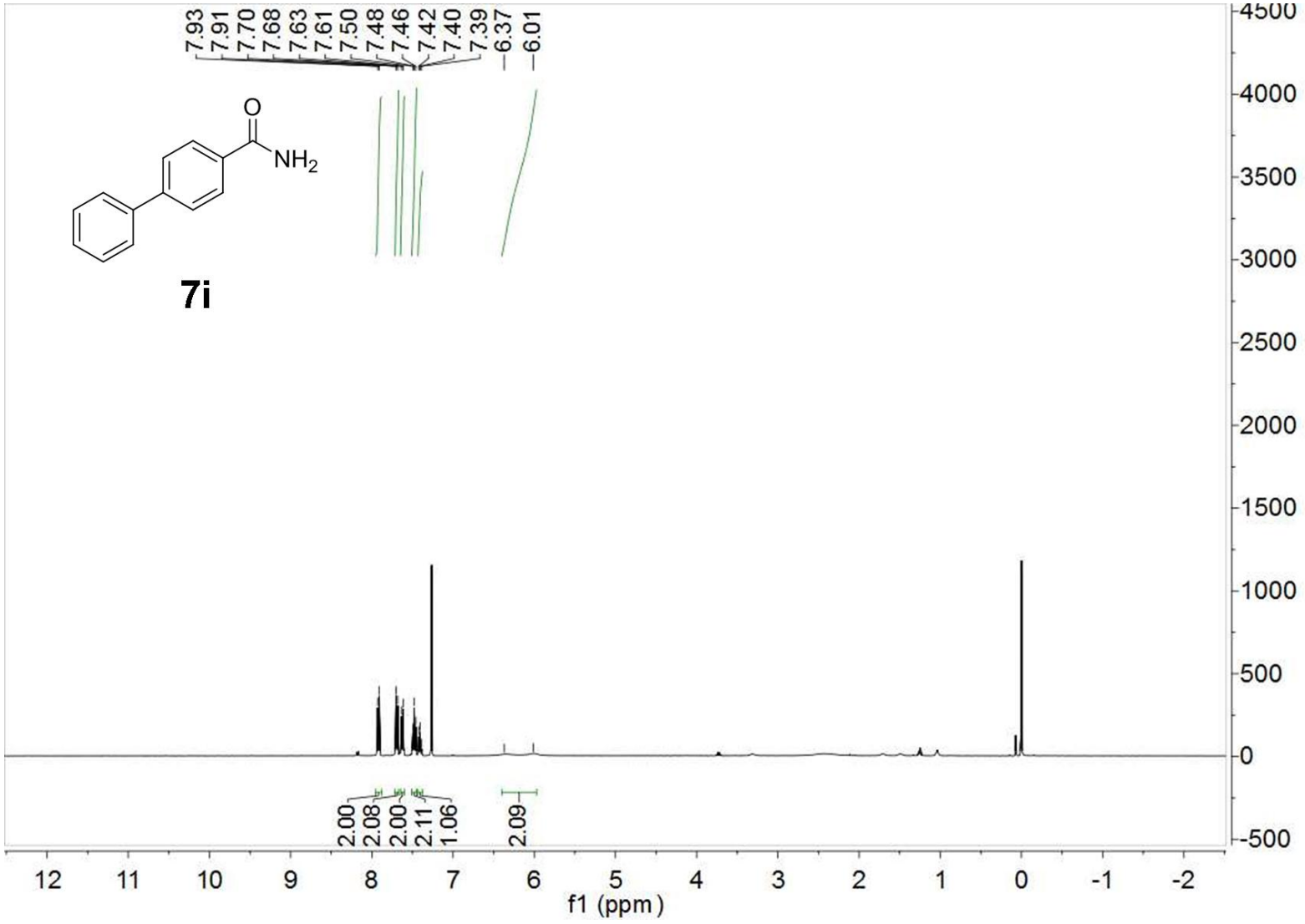

8.32
8.30
8.13
8.11
7.75
O
NH2
O2N
7g
30000
25000
20000
15000
10000
5000
0
2.84
2.00
0.95
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

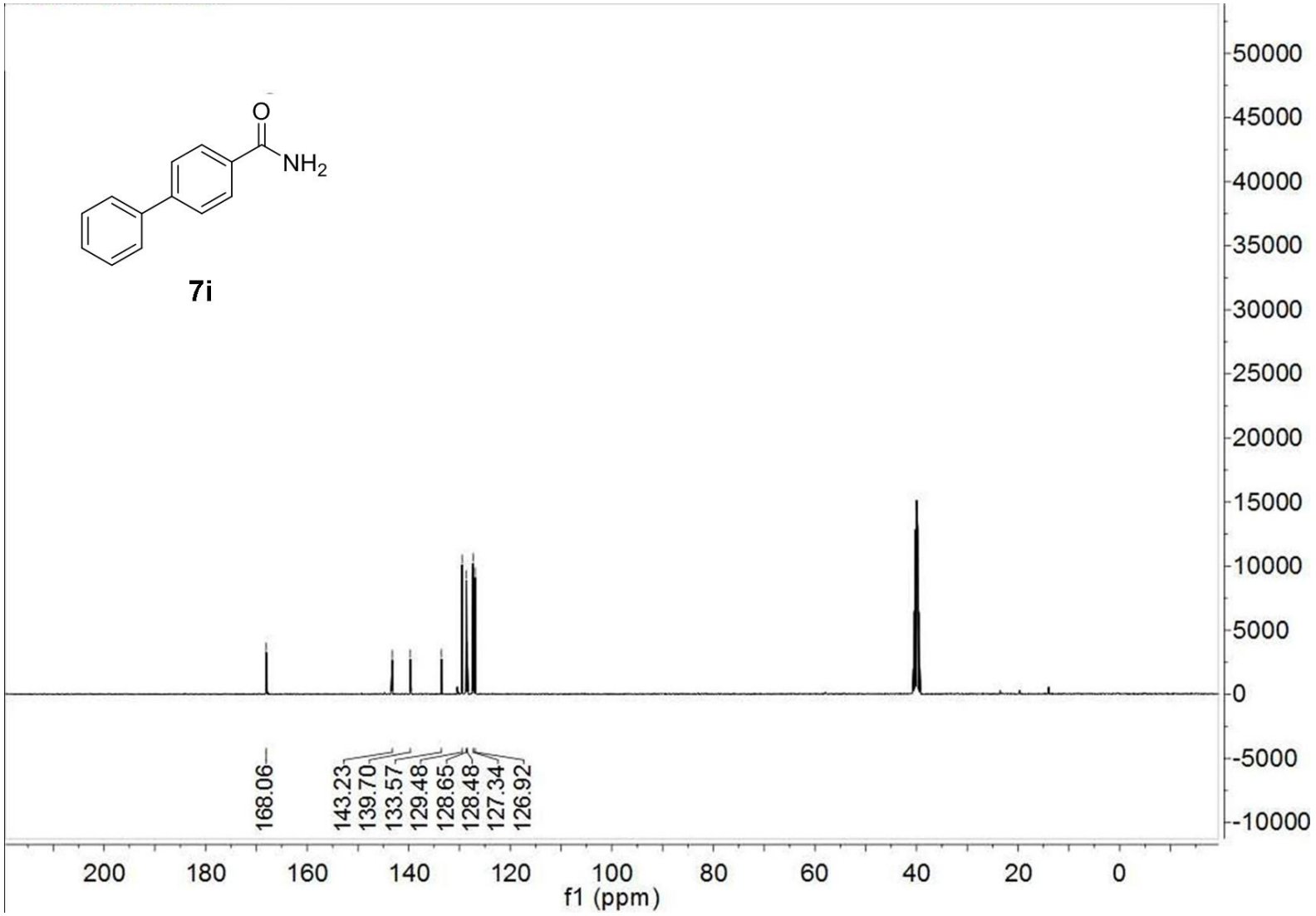

O
NH2
O2N
7g
4000
3500
3000
2500
2000
1500
1000
500
0
-500
166.68
149.51
140.44
129.37
123.90
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

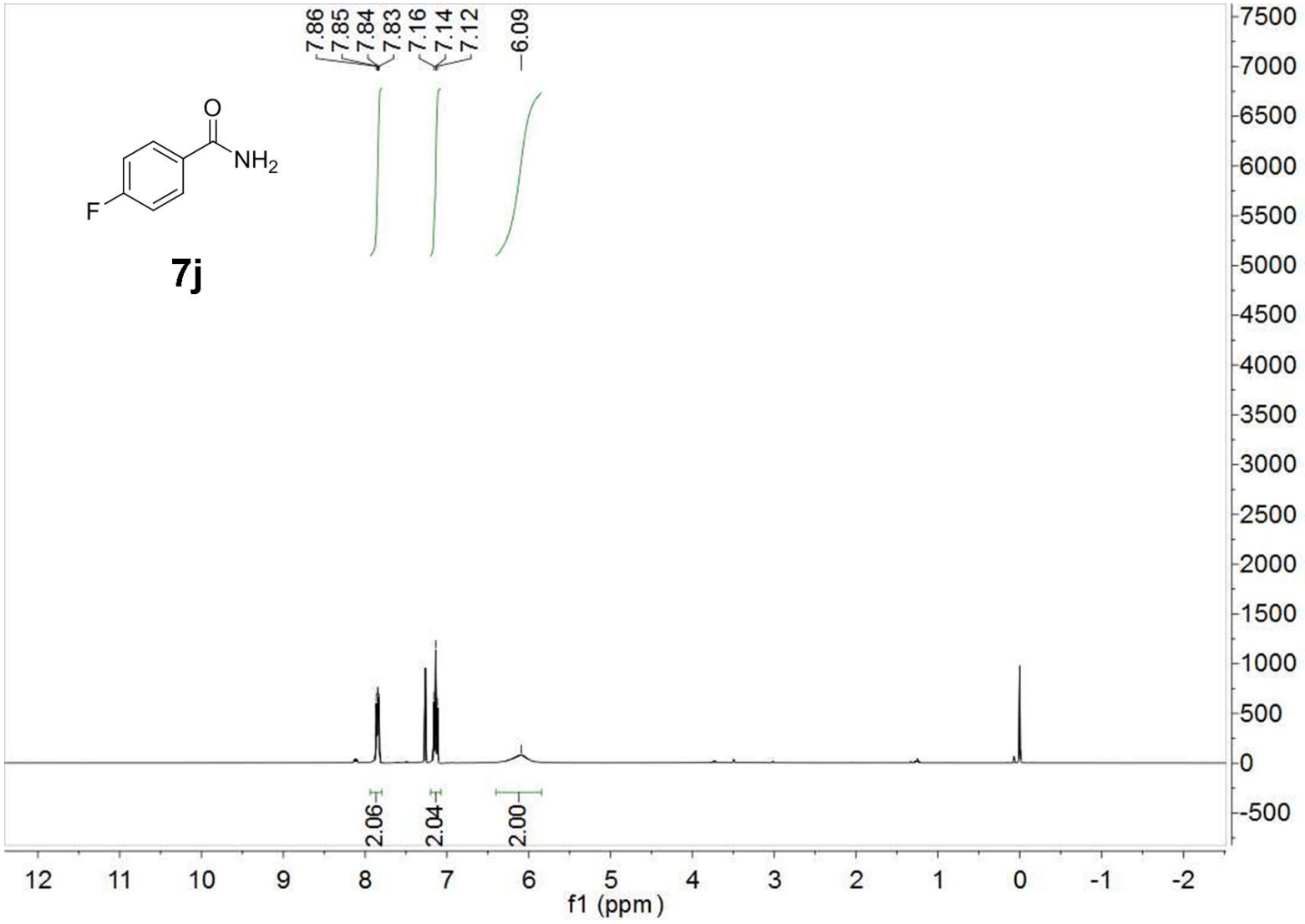

7.93
7.74
7.72
6.14
5.83
O
NH2
F3C
7h
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
2.05
2.00
2.06
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

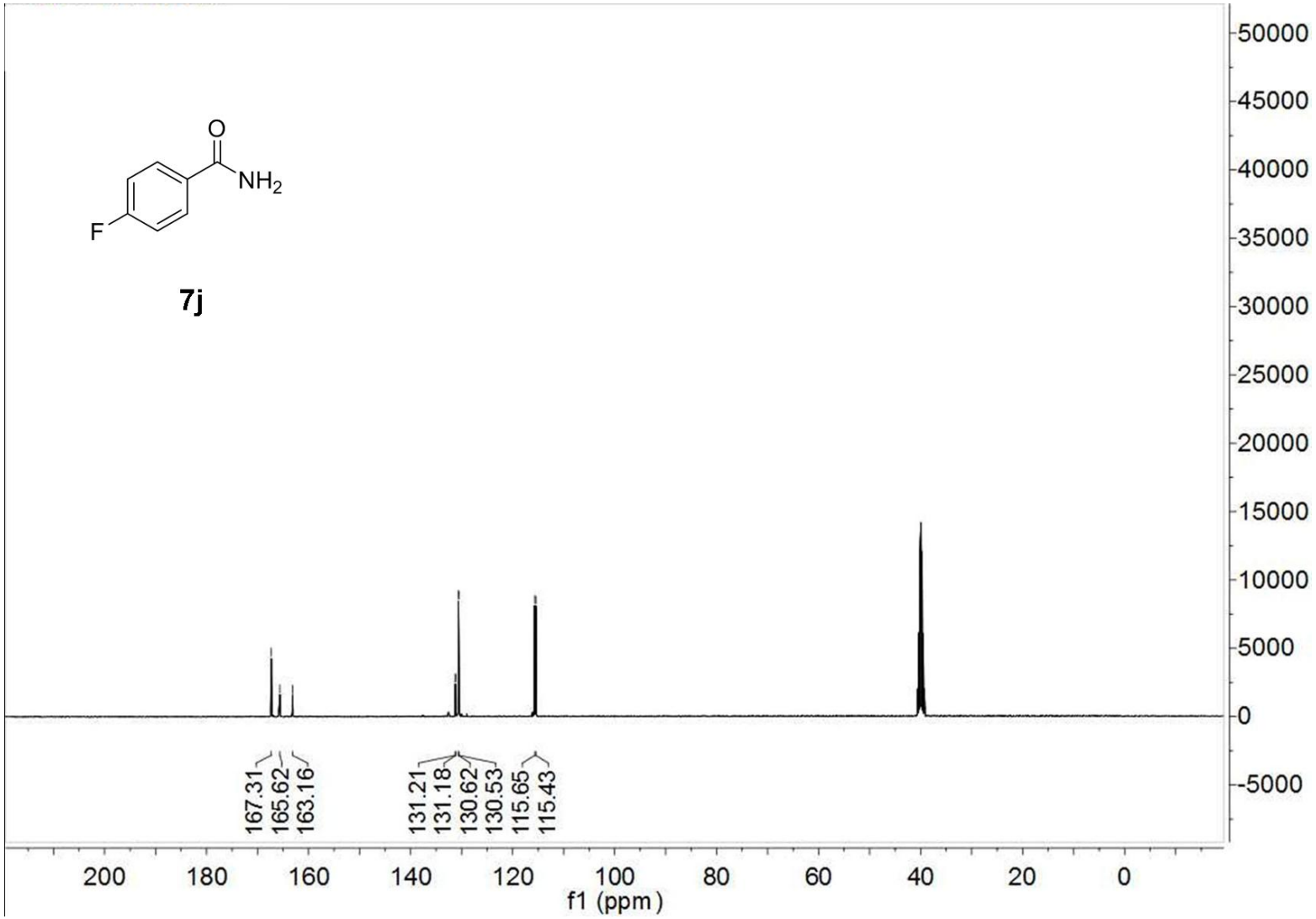

O
NH2
F3C
7h
167.15
138.54
132.10
131.78
131.46
131.15
128.78
125.73
125.69
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000

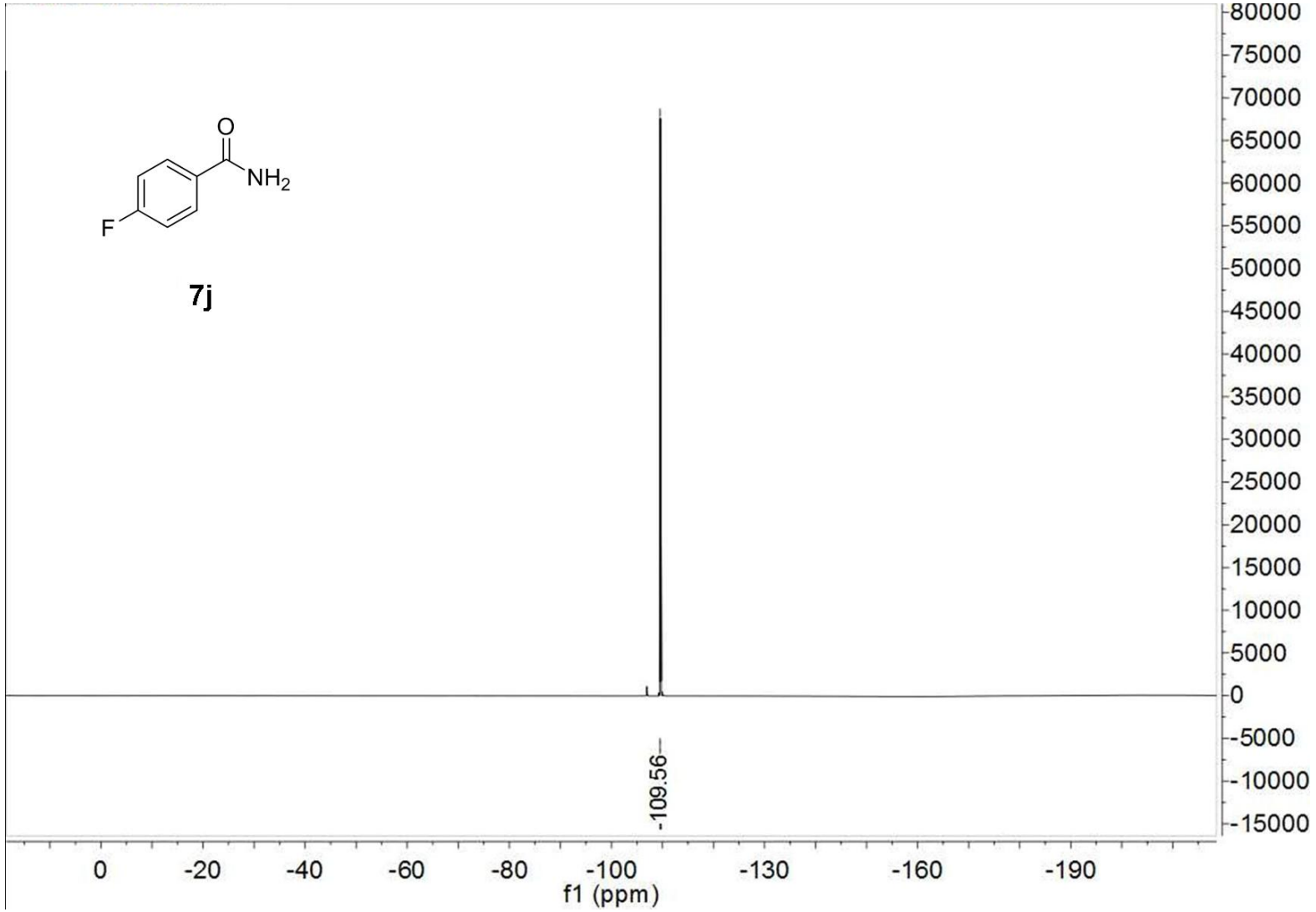

O
NH2
F3C
7h
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
-61.38
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)

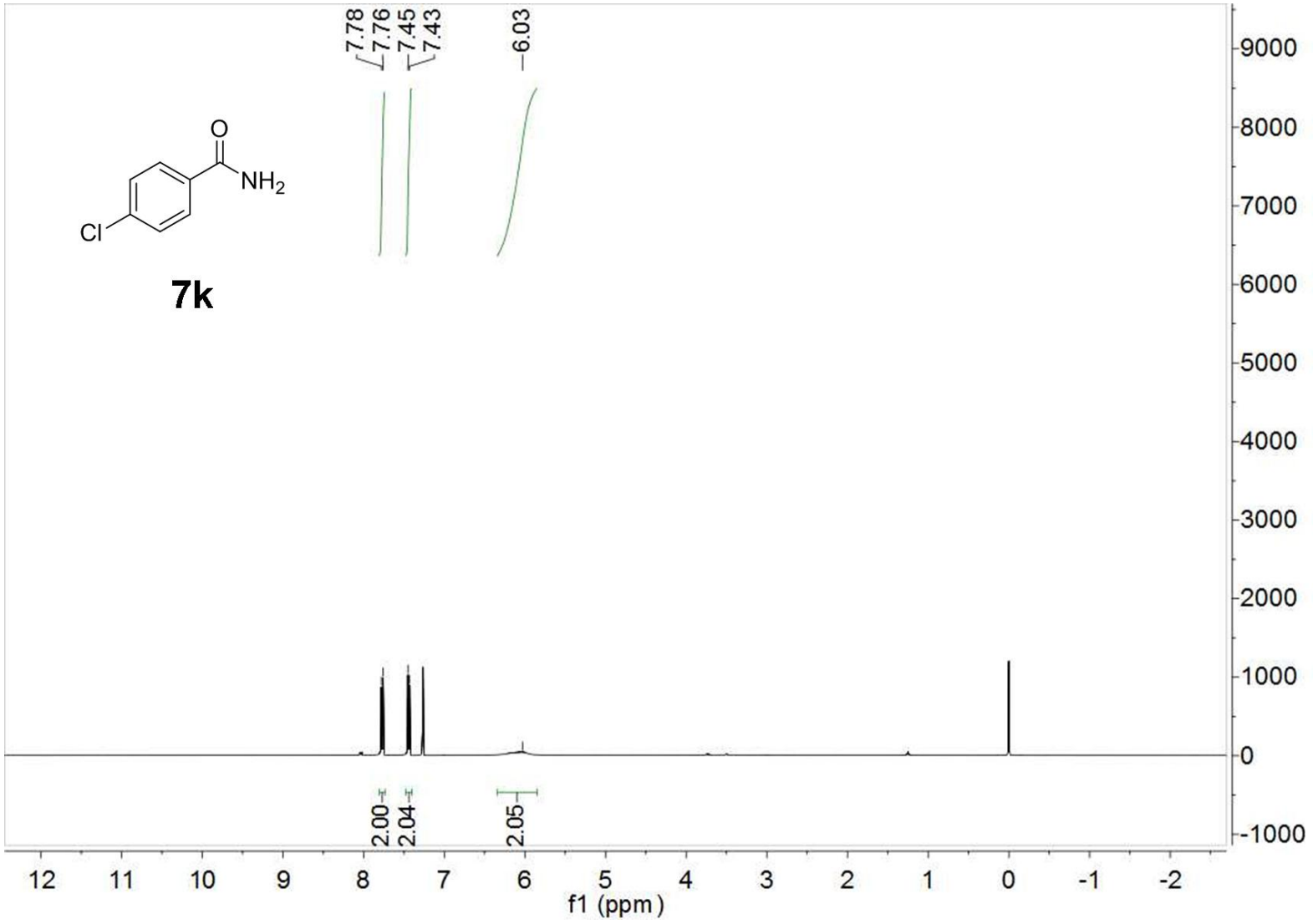

7.93
7.91
7.70
7.68
7.63
7.61
7.50
7.48
7.46
7.42
7.40
7.39
6.37
6.01
O
NH2
7i
2.00
2.08
2.00
2.11
1.06
2.09
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

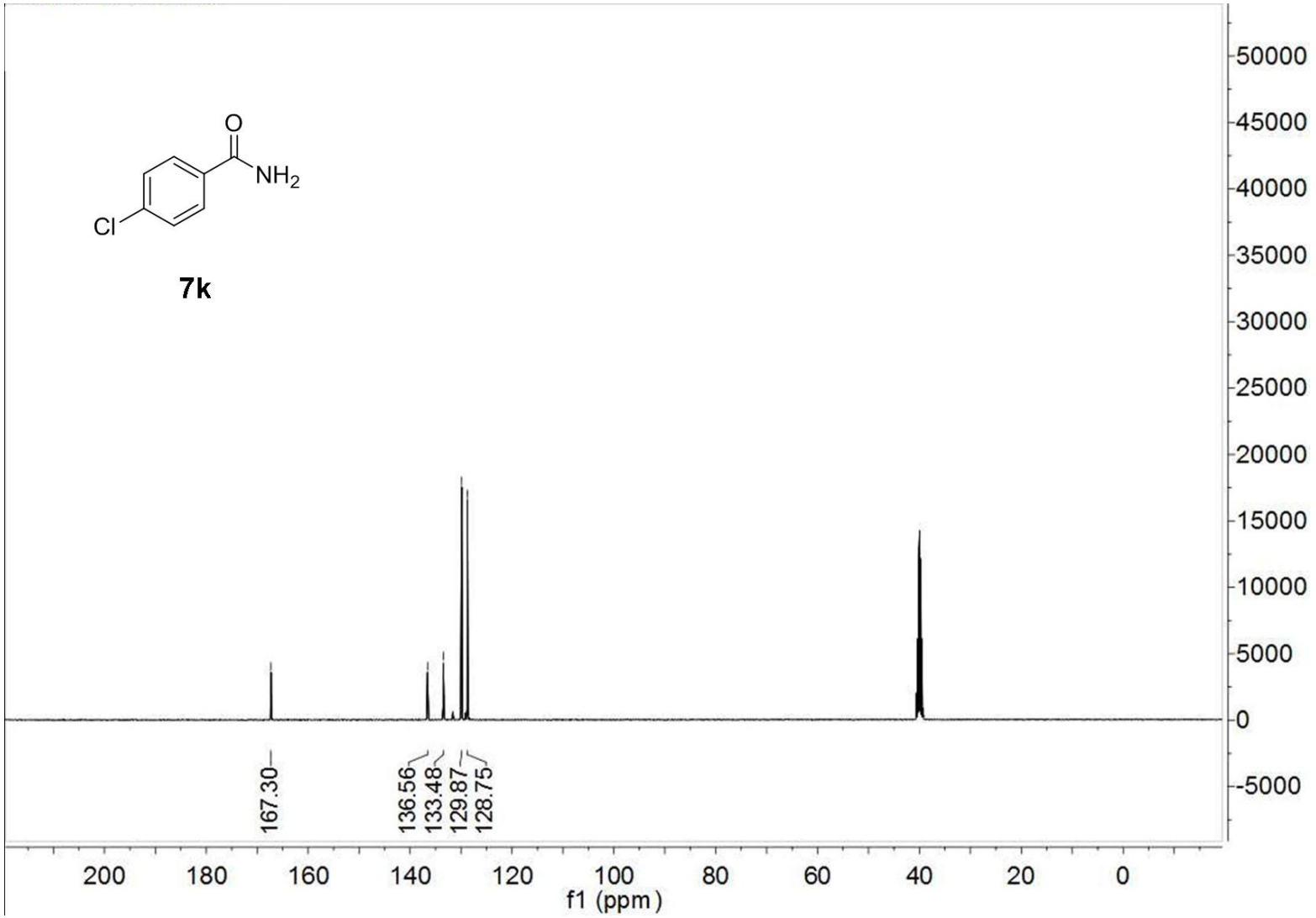

O
NH2
7i
168.06
143.23
139.70
133.57
129.48
128.65
128.48
127.34
126.92
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

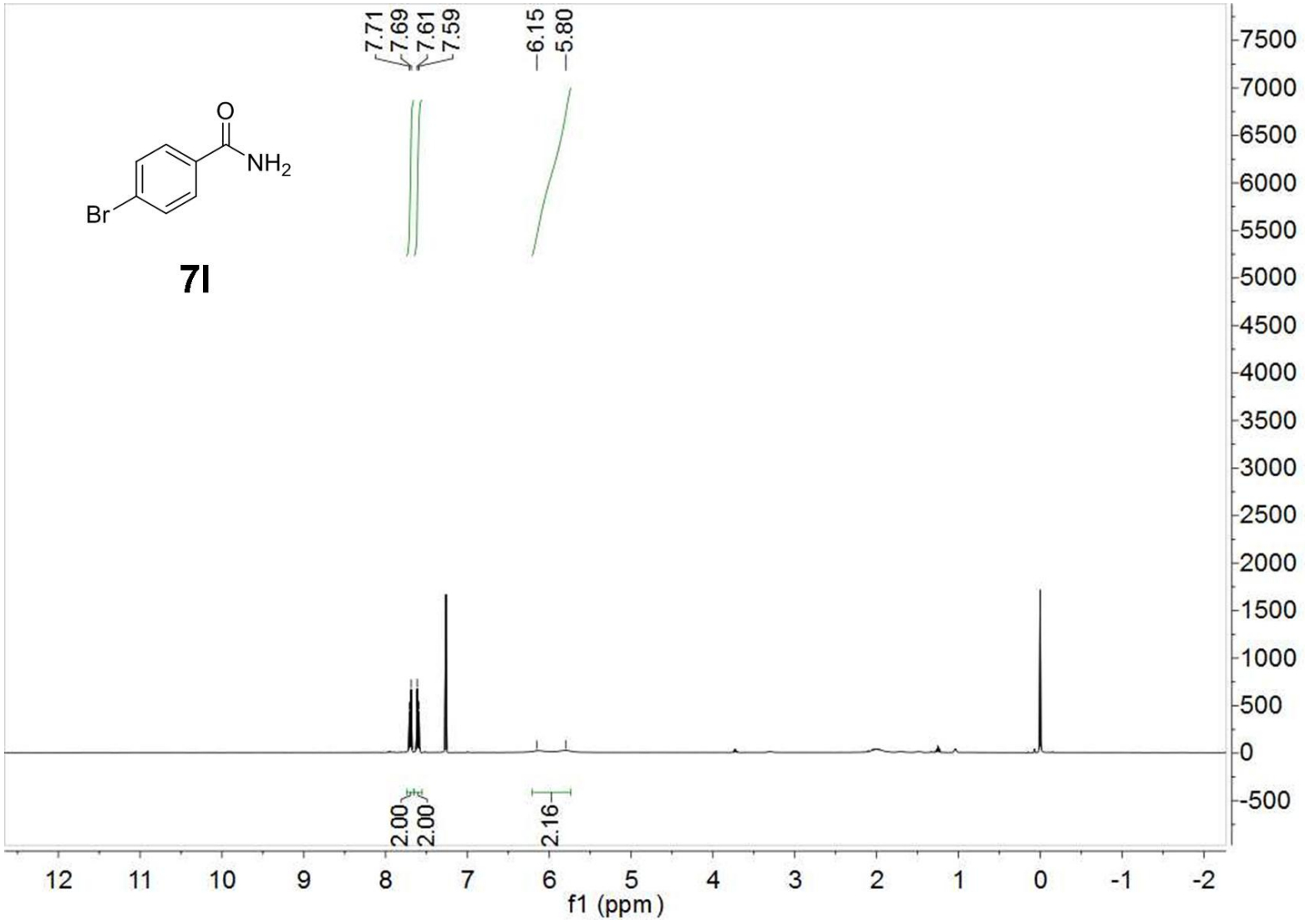

7.86
7.85
7.84
7.83
7.16
7.14
7.12
6.09
O
NH2
F
7j
2.06
2.04
2.00
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

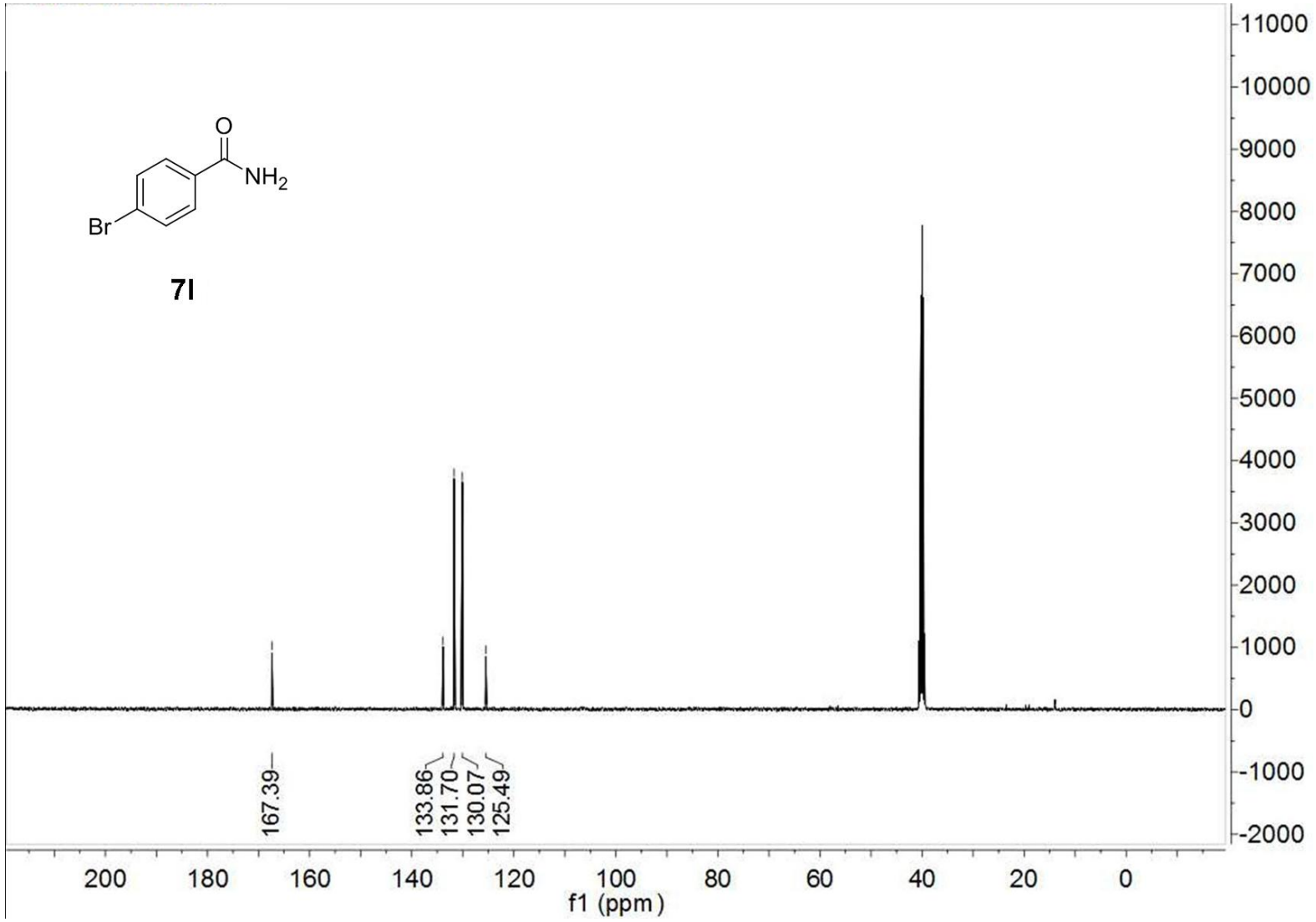

O
NH2
F
7j
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
167.31
165.62
163.16
131.21
131.18
130.62
130.53
115.65
115.43
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

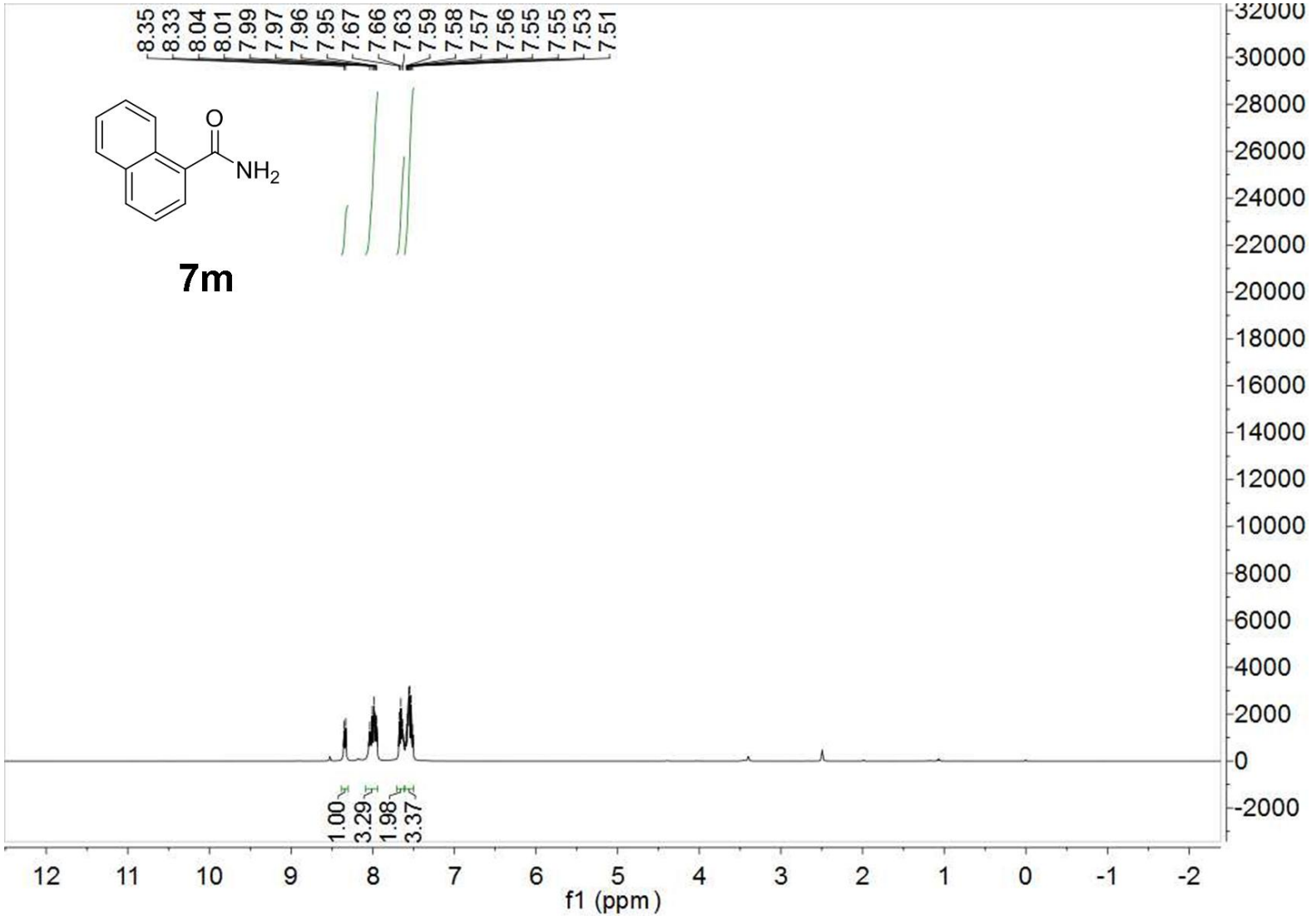

O
NH2
F
7j
-109.56
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
-15000

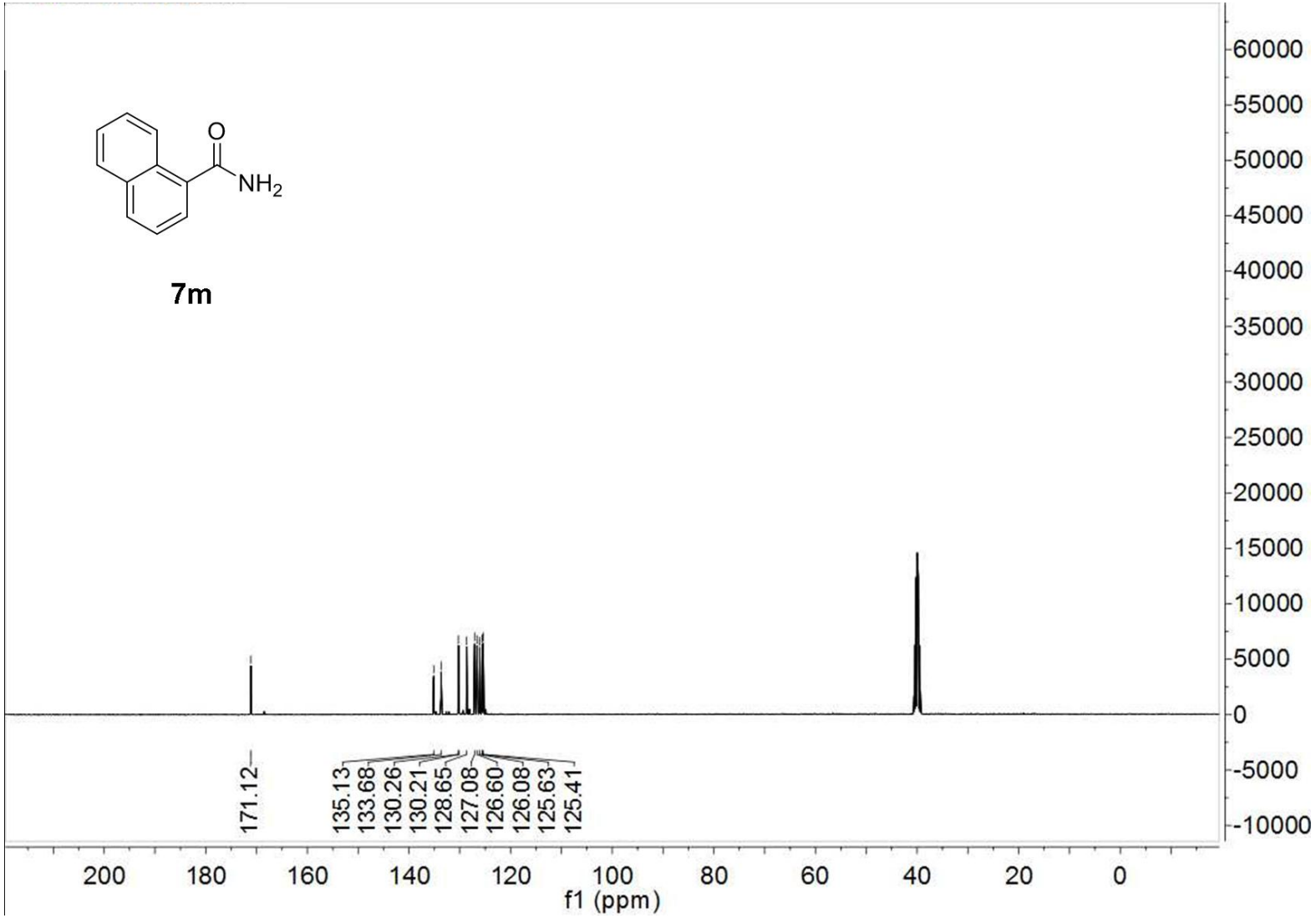

7.78
7.76
7.45
7.43
6.03
O
NH2
Cl
7k
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
2.00
2.04
2.05
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

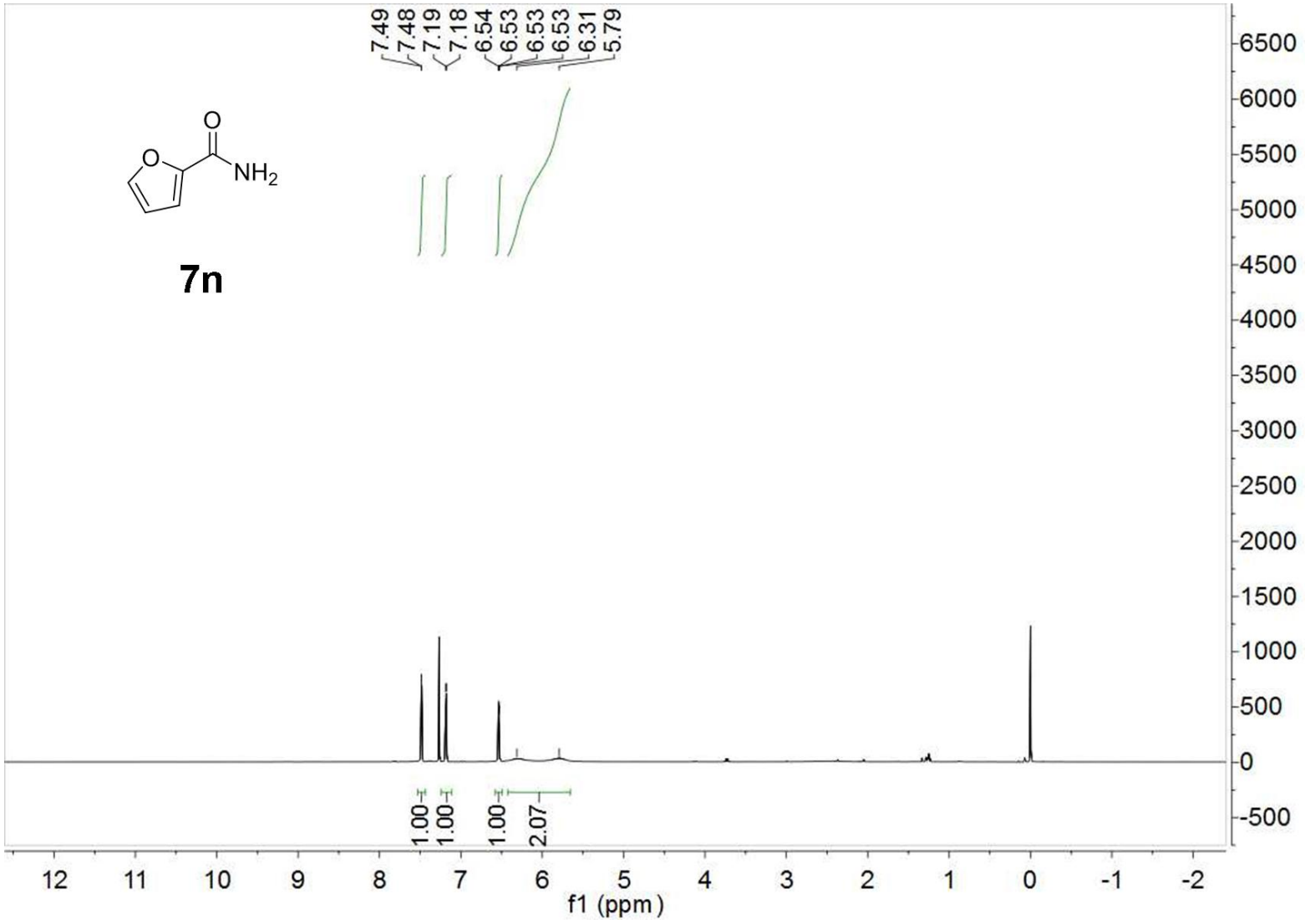

O
NH2
Cl
7k
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
167.30
136.56
133.48
129.87
128.75
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

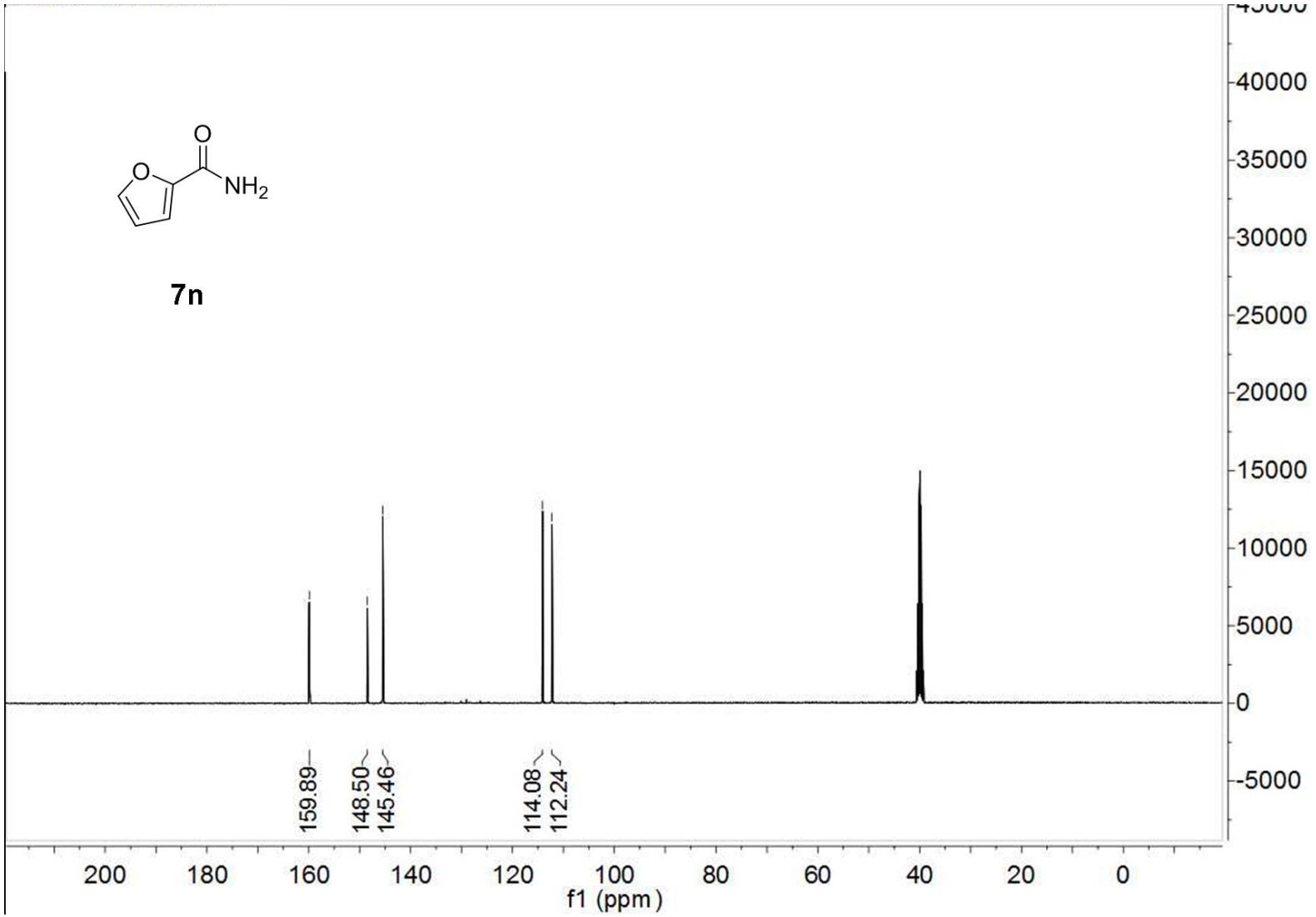

7.71
7.69
7.61
7.59
6.15
5.80
O
NH2
Br
7l
2.00
2.00
2.16
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

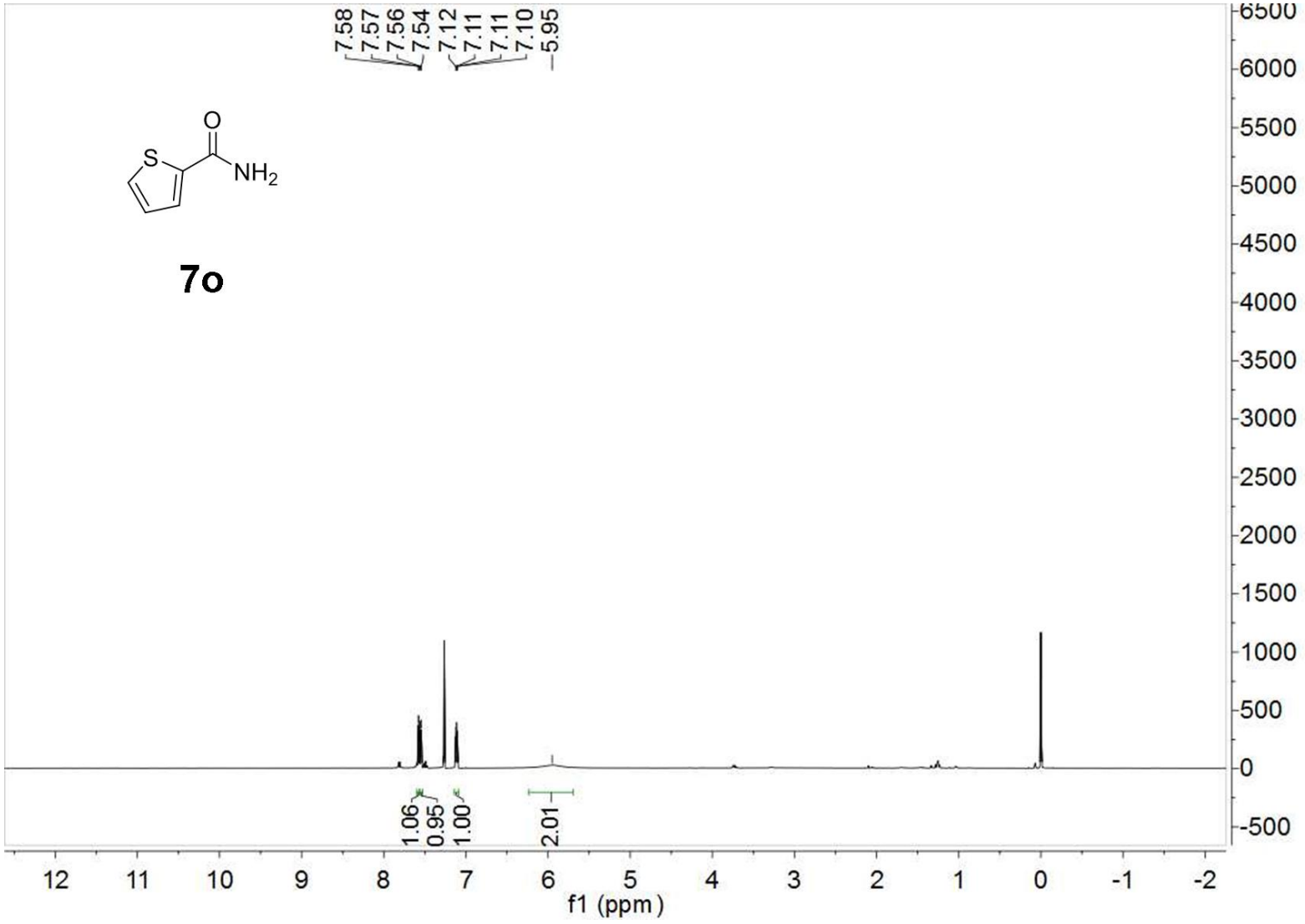

O
NH2
Br
7l
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
167.39
133.86
131.70
130.07
125.49
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

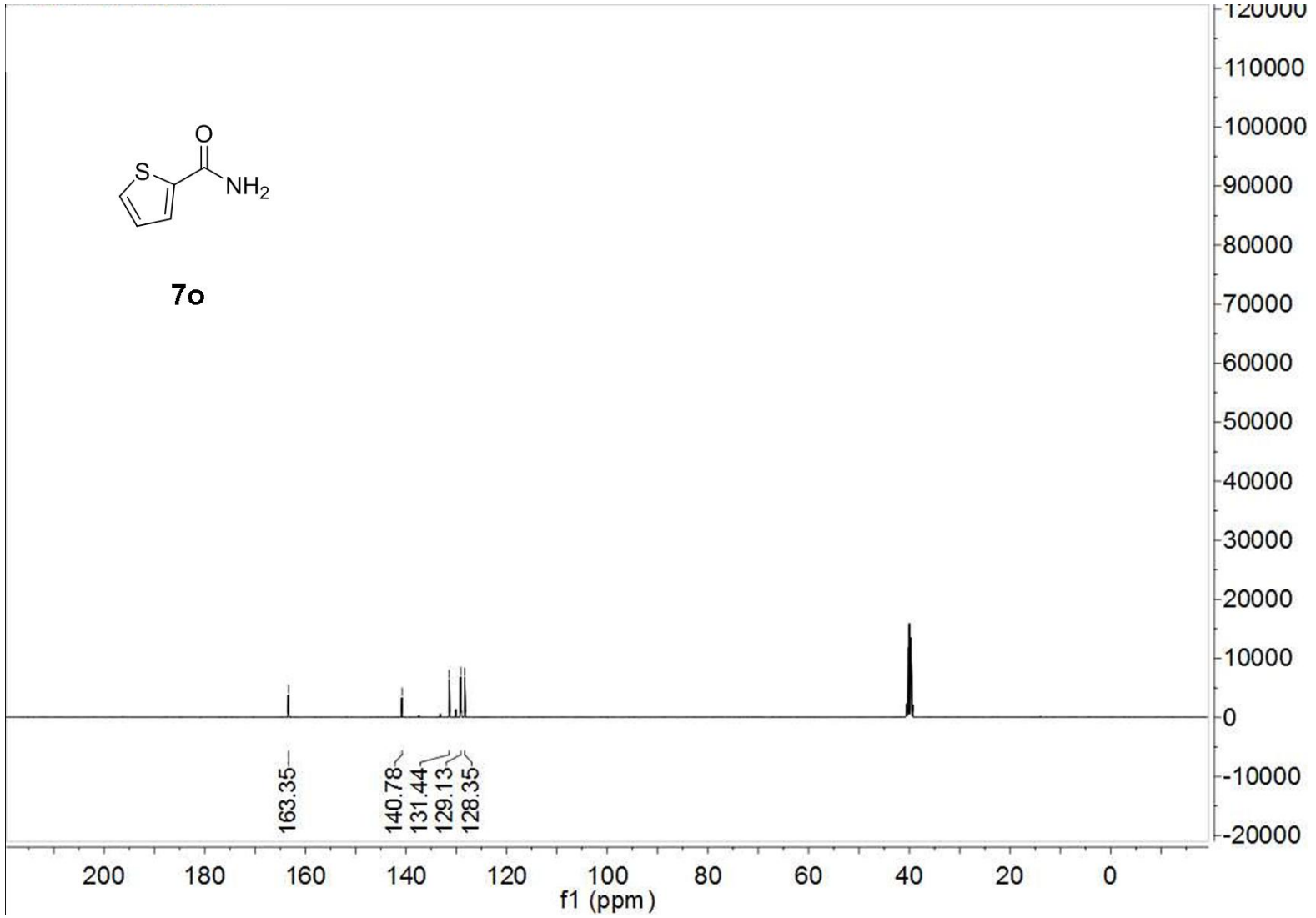

8.35
8.33
8.04
8.01
7.99
7.97
7.96
7.95
7.67
7.66
7.63
7.59
7.58
7.57
7.56
7.55
7.55
7.53
7.51
O
NH2
7m
1.00
3.29
1.98
3.37
f1 (ppm)

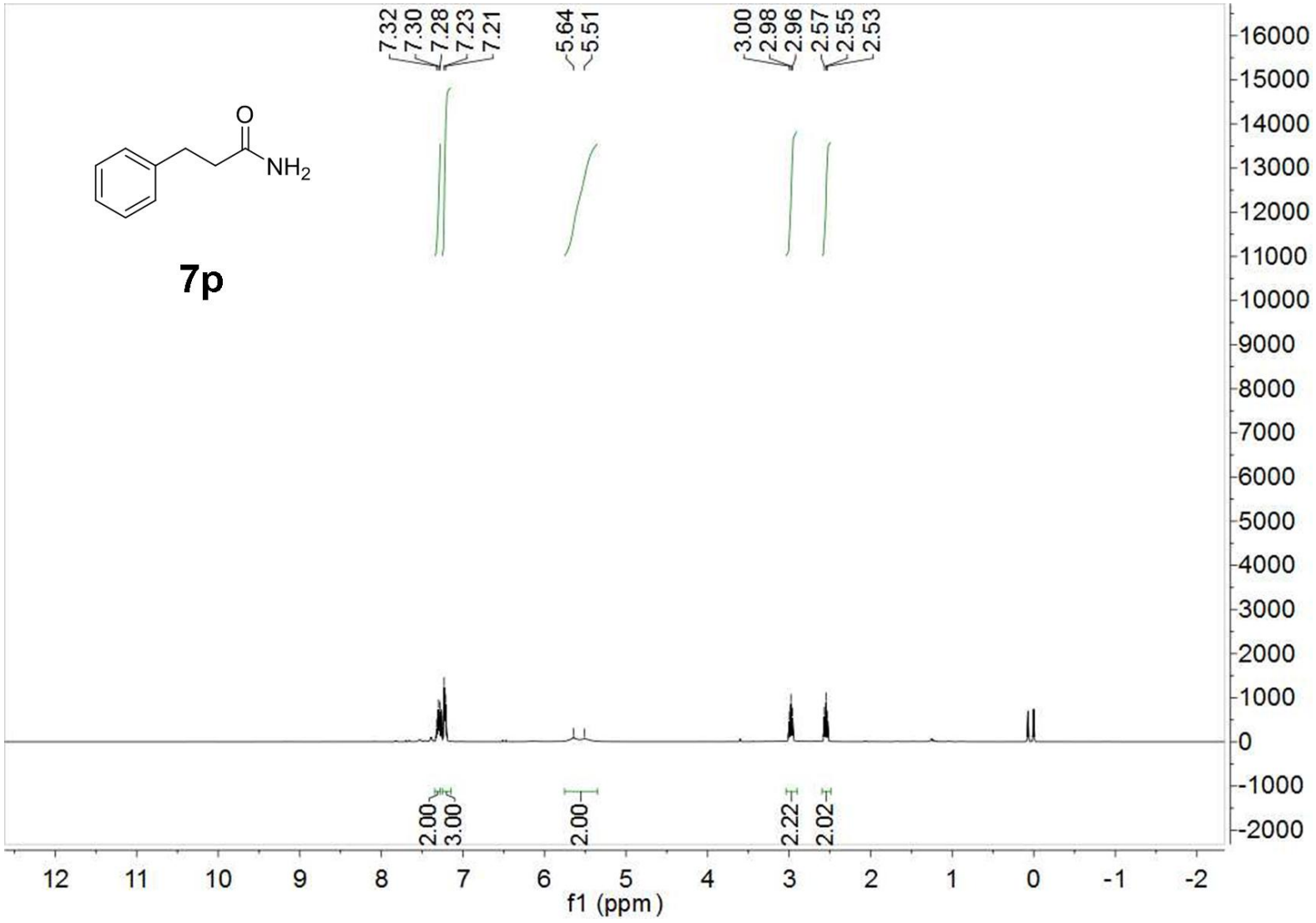

O
NH2
7m
171.12
135.13
133.68
130.26
130.21
128.65
127.08
126.60
126.08
125.63
125.41
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

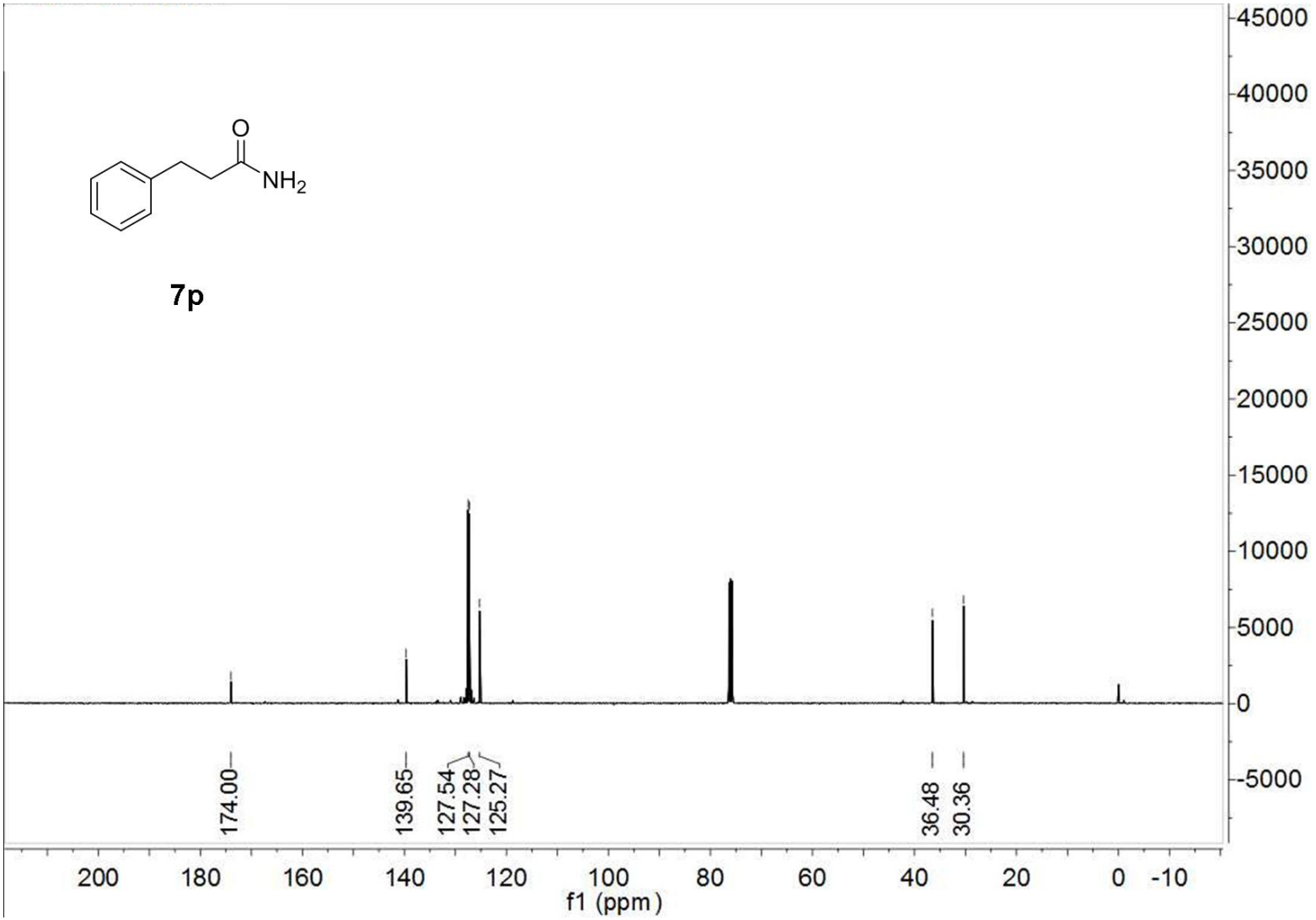

7.49
7.48
7.19
7.18
6.54
6.53
6.53
6.53
6.31
5.79
O
O
NH2
7n
1.00
1.00
1.00
2.07
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500


O
O
NH2
7n
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
159.89
148.50
145.46
114.08
112.24
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


7.58
7.57
7.56
7.54
7.12
7.11
7.11
7.10
5.95
O
S
NH2
7o
1.06
0.95
1.00
2.01
f1 (ppm)


O
S
NH2
7o
163.35
140.78
131.44
129.13
128.35
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000


7.32
7.30
7.28
7.23
7.21
5.64
5.51
3.00
2.98
2.96
2.57
2.55
2.53
O
NH2
7p
2.00
3.00
2.00
2.22
2.02
f1 (ppm)


O
NH2
7p
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
174.00
139.65
127.54
127.28
125.27
36.48
30.36
200
180
160
140
120
100
80
60
40
20
0
-10
f1 (ppm)