Fast Construction of Isoquinolin-1(2*H*)-ones by Direct Intramolecular C-H/N-H Functionalization under Metal-Free Conditions

Li-Rong Wen, Guang-Yi Ren, Rui-Sen Geng, Lin-Bao Zhang,* Ming Li

**State Key Laboratory Base of Eco-Chemical Engineering, College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, P. R. China*

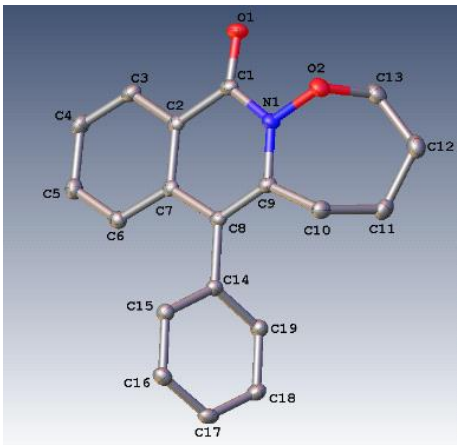
* E-mail: zhang_linbao@126.com,

Contents

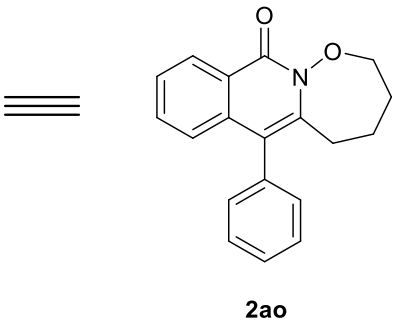
General methods

Unless noted, all commercial reagents and solvents were used without further purification. Melting points were recorded on a RY-1 microscopic melting apparatus and uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded at 500 MHz (or 400 MHz) and 125 MHz (or 100 MHz), ^{19}F NMR spectra was recorded at 376 MHz, respectively, with TMS as internal standard. Mass spectra were obtained on an Ultima Global spectrometer with an ESI source. Silica gel (200–300 mesh) for column chromatography and silica GF254 for TLC were produced by Qingdao Marine Chemical Company (China).

Molecular structure and crystallographic data of 2ao



CCDC:1876764



2ao

Figure S1. X-ray crystal structure of **2ao**

Table S1. Crystal data and structure refinement for **2ao**

Empirical formula	$C_{19}H_{17}NO_2$
CCDC number	1876764
Formula weight	291.33
Temperature	170.01(10) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 9.07170(10) Å
	b = 15.9713(2) Å
	c = 9.99690(10) Å

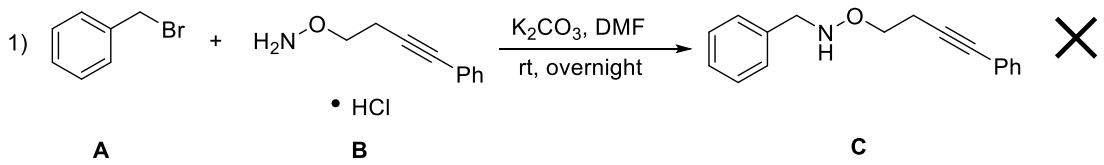
Volume	1430.55(3) $Å^3$
Z	4
Density (calculated)	1.353 Mg/m^3
Absorption coefficient	0.699 mm^{-1}
F(000)	616
Crystal size	0.475 x 0.472 x 0.345 mm^3
Theta range for data collection	5.267 to 66.542°.
Index ranges	-10<=h<=10, -16<=k<=19, -11<=l<=11
Reflections collected	12432
Independent reflections	2497 [R(int) = 0.0193]
Completeness to theta = 66.542°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.51589
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2497 / 0 / 199
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0472, wR2 = 0.1171
R indices (all data)	R1 = 0.0495, wR2 = 0.1190
Extinction coefficient	n/a
Largest diff. peak and hole	0.801 and -0.257 $e.Å^{-3}$

Preparation of starting materials

Substrates **1** were synthesized according to the literature.[1]

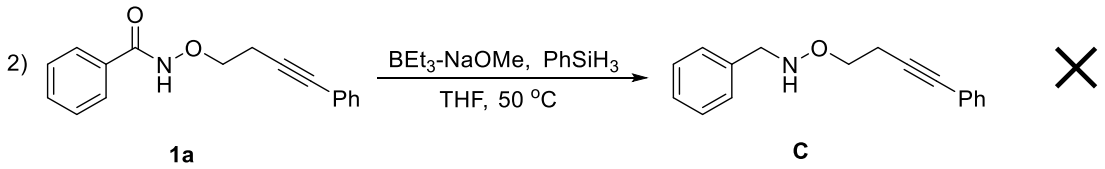
Unsuccessful substrate synthesis

It is difficult to synthesize alkyl alkyne tethered *N*-hydroxylamines derivatives. After many attempts we failed to synthesize the corresponding compounds.



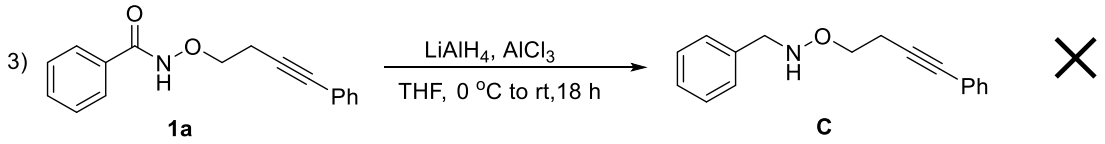
A **B** **C**

B (39.5 mg, 0.2 mmol) was dissolved in DMF (1.5 mL). Potassium carbonate (110.6 mg, 0.8 mmol) was added to the flask followed by benzyl bromide (30 μL, 0.26 mmol). The mixture was stirred overnight. The result showed that **C** was not obtained.



1a **C**

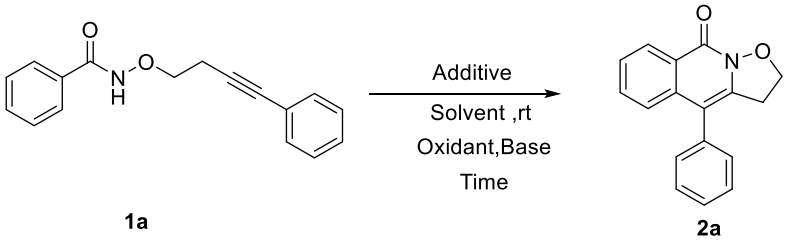
In an argon-filled glovebox, a dried Schlenk tube was charged with NaOMe (0.01 mmol, 2.5 mol %), THF (1 mL), and BEt_3 (0.01 mmol, 1 M in THF, 2.5 mol %). After the resulting mixture was stirred at RT for 3 min, **1a** (0.4 mmol, 1 equiv) and $PhSiH_3$ (0.8 mmol, 2 equiv) were added by syringe. The reaction tube was sealed tightly with a teflon plug and the mixture was stirred at 50 °C for 48 h. The result showed that **C** was not obtained.



1a **C**

A suspension of $AlCl_3$ (266.7 mg, 2 mmol) in THF (15 ml) was added via cannula to a suspension of $LiAlH_4$ (98.8 mg, 2.6 mmol) in THF (15 mL) both at 0 °C. After stirring for 10 min at 0 °C, **1a** (53.1 mg, 0.2 mmol) in THF (15 mL) was then added via cannula slowly with evolution of gas. The ice was removed and it was allowed to stir at rt for 18 h. The result showed that **C** was not obtained.

Optimization of reaction conditions



1a **2a**

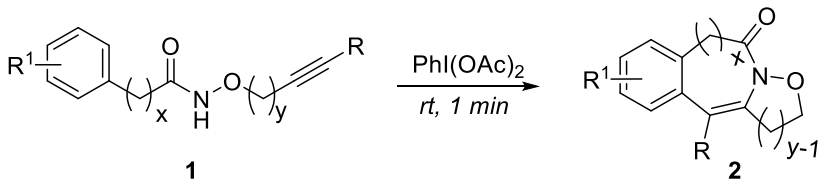
Entry	Solvent	Oxidant	Additive	Time	Yield (%)[b]
1	HFIP	PIDA	NaOAc	1 min	91
2	HFIP	PIDA	---	1 min	92
3	HFIP	---	---	1 min	NR
4[c]	HFIP	*m*-CPBA	PhI	10 h	60
5	HFIP	$PhI(AdCOO)_2$	---	1 min	82
6	HFIP	$PhI(CF_3COO)_2$	---	1 min	74
7	HFIP	Ph-EBX	---	1 min	trace

8	HFIP	HTIB	---	1 min	30
9	MeOH	PIDA	---	1 min	trace
10	CH_3CH_2OH	PIDA	---	1 min	trace
11	CF_3CH_2OH	PIDA	---	1 min	80
12	*t*-AmOH	PIDA	---	1 min	trace
13	DCE	PIDA	---	1 min	NR
14	$CHCl_3$	PIDA	---	1 min	NR
15	toluene	PIDA	---	1 min	NR
16	DMF	PIDA	---	1 min	NR
17	MeCN	PIDA	---	1 min	32
18[d]	HFIP	PIDA	---	1 min	45
19[d]	CH_3CH_2OH	PIDA	---	1 min	trace
20[d]	MeCN	PIDA	---	1 min	trace
21[d]	DCE	PIDA	---	1 min	NR

[a] Reaction conditions: **1** (0.20 mmol), Oxidant (0.2 mmol), additive (0.2 mmol), solvent (4 mL), rt. [b] Isolated yields. [c] PhI (0.02 mmol). [d] PIDA (0.1 mmol). PIDA = $PhI(OAc)_2$, PIFA = $PhI(OCOCF_3)_2$, HFIP = hexafluoro-2-propanol, TFE = trifluoroethanol, HTIB = hydroxyl(phenyl) iodotosylate.

Synthesis of compounds 2

Substrates **1** (0.2 mmol, 1.0 equiv), PIDA (0.2 mmol, 1.0 equiv) and HFIP (4 mL) were added to a 25 mL round bottom flask which was equipped with a stirring bar. The mixture was stirred at room temperature open to air until the starting material was completely consumed. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 1/1) to give the corresponding product **2**.



1 **2**

Gram-scale reaction

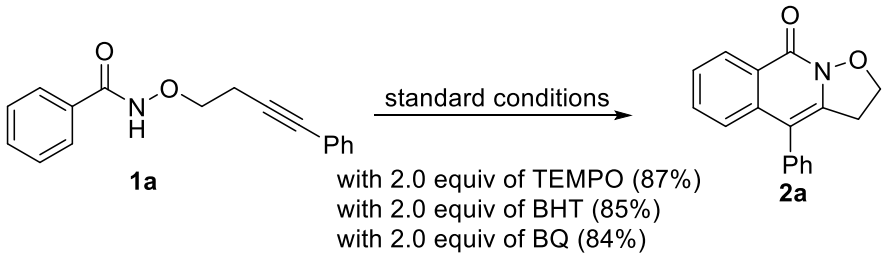
Substrates **1** (5 mmol, 1.0 equiv), PIDA (5 mmol, 1.0 equiv) and HFIP (100 mL) were added to a 250 mL round bottom flask which was equipped with a stirring bar. The mixture was stirred at room temperature open to air until the starting material was completely consumed. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 1/1) to give the corresponding product **2** (1.05 g, 80%).

HFIP recycling

1a (0.5 mmol, 1.0 equiv), PIDA (0.5 mmol, 1.0 equiv) and HFIP (10 mL) were added to a 25 mL round bottom flask which was equipped with a stirring bar. The mixture was stirred at room temperature open to air until the starting material was completely consumed, then the solvent was collected by vacuum distillation. Subsequently, **1a** (0.2 mmol, 1.0 equiv), PIDA (0.2 mmol, 1.0 equiv) and recycled HFIP (4 mL) were added to a 25 mL round bottom flask and reacted until the starting material was completely consumed. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 1/1) to give product **2a** in 90% yield.

Control experiments

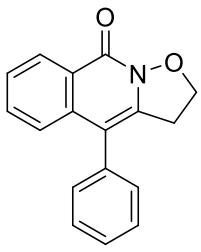
TEMPO/BHT/BQ inhibiting experiment:



Substrates **1** (0.2 mmol, 1.0 equiv), TEMPO (0.4 mmol, 2.0 equiv) or BHT (0.4 mmol, 2.0 equiv) or BQ (0.4 mmol, 2.0 equiv) and HFIP (4 mL) were added to a 25 mL round bottom flask which was equipped with a stirring bar, then PIDA (0.2 mmol, 1.0 equiv) was added. The mixture was stirred at room temperature open to air until the starting material was completely consumed. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 1/1) to give the corresponding product.

Characterization of compounds 2

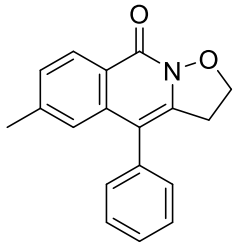
7-phenyl-8,9-dihydro-12*H*-benzo[*h*]isoxazolo[2,3-*b*]isoquinolin-12-one (2a).[2]



White solid, 48 mg, 92% yield, M.p. = 206-208 °C;

^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.54 (d, J = 7.1 Hz, 1H), 7.59-7.41 (m, 5H), 7.36 (t, J = 7.7 Hz, 3H), 4.58 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 153.95, 136.20, 135.02, 132.52, 131.63, 130.31, 128.93, 128.10, 127.47, 126.32, 124.71, 113.03, 69.55, 32.46.

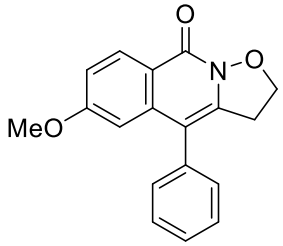
6-methyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2b). [2]



White solid, 55 mg, 99% yield, M.p. = 219-221 °C;

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.34 (d, 1H), 7.50 (t, J = 7.5 Hz, 2H), 7.44 (t, J = 6.9 Hz, 1H), 7.36 (d, J = 8.5 Hz, 1H), 7.34 (d, J = 7.8 Hz, 2H), 7.29 – 7.25 (m, 1H), 4.57 (t, J = 7.5 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H), 2.48 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 153.90, 136.51, 135.17, 133.90, 133.17, 131.51, 130.27, 128.86, 128.00, 127.00, 126.23, 124.65, 113.02, 109.98, 69.58, 32.32, 21.20.

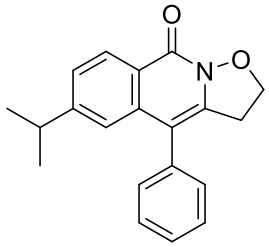
6-methoxy-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2c). [2]



White solid, 57 mg, 98% yield, M.p. = 160-162 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90 (d, J = 2.7 Hz, 1H), 7.47 (t, J = 7.3 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H), 7.35 – 7.30 (m, 2H), 7.30 – 7.25 (m, 1H), 7.13 (dd, J = 9.0, 2.7 Hz, 1H), 4.56 (t, J = 7.5 Hz, 2H), 3.91 (s, 3H), 3.36 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 158.33, 153.43, 135.12, 130.19,130.07, 128.81, 127.97, 127.53, 126.28, 122.21, 113.01, 107.13, 69.71, 55.71, 32.13.

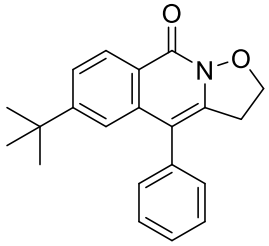
6-isopropyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2d).



White solid, 60 mg, 98% yield, M.p. = 100-102 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.39 (d, 1H), 7.50 (t, J = 7.3 Hz, 2H), 7.47 – 7.41 (m, 2H), 7.34 (d, J = 7.1 Hz, 2H), 7.31 (d, J = 8.4 Hz, 1H), 4.57 (t, J = 7.5 Hz, 2H), 3.38 (t, J = 7.5 Hz, 2H), 3.10-3.03 (m, 1H), 1.31 (d, J = 6.9 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.05, 147.48, 135.21, 134.28, 131.55, 130.76, 130.27, 128.86, 128.00, 126.32, 124.80, 124.54, 113.02, 99.99, 69.58, 34.06, 32.32, 23.89; **HRMS** (ESI) m/z calcd for $C_{20}H_{19}NNaO_2^+$ $[M+Na]^+$ 328.1313, found 328.1318.

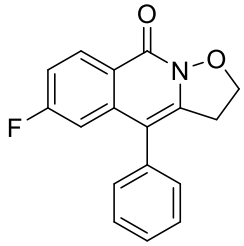
6-(tert-butyl)-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2e).



White solid, 63 mg, 99% yield, M.p. = 206-208 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.52 (d, J = 2.0 Hz, 1H), 7.61 (dd, J = 8.6, 2.1 Hz, 1H), 7.49 (t, J = 7.3 Hz, 2H), 7.44 (t, J = 7.3 Hz, 1H), 7.38 – 7.29 (m, 3H), 4.56 (t, J = 7.5 Hz, 2H), 3.38 (t, J = 7.5 Hz, 2H), 1.38 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.10, 149.69, 135.13, 133.83, 131.63, 130.22, 129.64, 128.80, 127.94, 125.99, 124.49, 123.29, 112.58, 69.52, 34.94, 32.27, 31.24. **HRMS** (ESI) m/z calcd for $C_{21}H_{21}NNaO_2^+$ $[M+Na]^+$ 342.1470, found 342.1470.

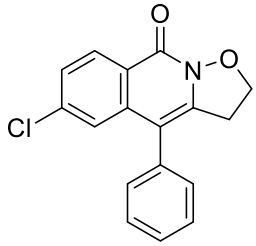
6-fluoro-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2f).



White solid, 48 mg, 86% yield, M.p. =230-232 °C;

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.16-8.13 (m, 1H), 7.53 – 7.48 (m, 2H), 7.48 – 7.42 (m, 1H), 7.38 – 7.31(m, 3H), 7.28 – 7.24 (m, 1H), 4.58 (t, J = 7.6 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 161.10 (d, $^1J_{C,F}$ = 248.2 Hz), 152.94, 134.69, 132.76, 131.90, 130.15, 128.98, 128.24, 127.87 (d, $^3J_{C,F}$ = 7.6 Hz), 127.14 (d, $^3J_{C,F}$ = 7.4 Hz), 120.31 (d, $^2J_{C,F}$ = 23.5 Hz), 112.66, 112.46 (d, $^2J_{C,F}$ = 23.2 Hz), 69.74, 32.25; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -109.27; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}FNNaO_2^+$ $[M+Na]^+$ 304.0750, found 304.0754.

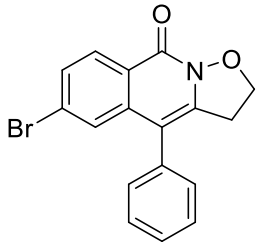
6-chloro-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one(2g).[2]



White solid, 52 mg, 87% yield, M.p. = 236-238 °C;

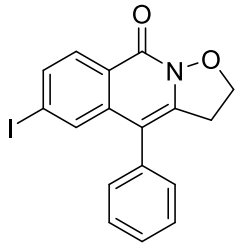
^{1}H NMR (500 MHz $CDCl_3$): δ 8.47 (d, J = 3.6 Hz, 1H), 7.51 (t, J = 7.3 Hz, 2H), 7.48 – 7.42 (m, 2H), 7.31 (m, 3H), 4.58 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 152.76, 134.51, 134.43, 132.89, 132.53, 132.02, 130.14, 129.01, 128.29, 127.29, 126.71, 126.34, 112.60, 69.69, 32.36.

6-bromo-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2h).



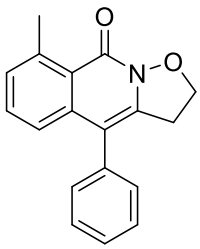
White solid, 61 mg, 89% yield, M.p. = 204-206 °C;
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.65 (d, J = 2.1 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.55 – 7.43 (m, 3H), 7.36 – 7.30 (m, 2H), 7.23 (d, J = 8.7 Hz, 1H), 4.59 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 152.72, 134.90, 134.81, 134.43, 133.12, 130.20, 129.91, 129.08, 128.36, 127.60, 126.52, 120.50, 112.73, 69.74, 32.47; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}BrNNaO_2^+$ $[M+Na]^+$ 363.9949, found 363.9949.

6-bromo-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2i).



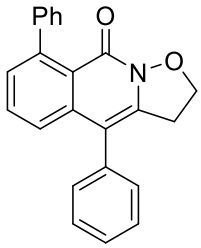
White solid, 64 mg, 64% yield, M.p. = 218-220 °C
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.87 (d, J = 1.7 Hz, 1H), 7.85 – 7.74 (m, 1H), 7.51 (t, J = 7.4 Hz, 2H), 7.46 (t, J = 7.3 Hz, 1H), 7.32 (d, J = 7.1 Hz, 2H), 7.09 (d, J = 8.6 Hz, 1H), 4.59 (t, J = 7.6 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 150.61, 138.42, 134.21, 133.40, 132.46, 131.39, 128.27, 127.15, 126.43, 125.79, 124.54, 110.85, 89.54, 67.78, 30.59; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}INNaO_2^+$ $[M+Na]^+$ 411.9810, found 411.9808.

8-methyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2j).



White solid, 55 mg, 99% yield, M.p. =180-182 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.49 (d, J = 8.7 Hz, 1H), 7.46 – 7.40 (m, 3H), 7.38 – 7.32 (m, 2H), 7.33 – 7.28 (m, 2H), 4.53 (t, J = 7.6 Hz, 2H), 3.22 (t, J = 7.6 Hz, 2H), 1.87 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.15, 139.23, 135.59, 134.72, 134.30, 133.43, 130.22, 128.59, 127.84, 127.51, 126.18, 126.11, 113.06, 69.41, 33.09, 23.87; **HRMS** (ESI) m/z calcd for $C_{18}H_{15}NNaO_2^+$ $[M+Na]^+$ 300.1000, found 300.1002.

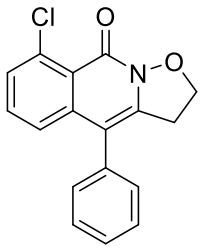
4,8-diphenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2k).



White solid, 67 mg, 99% yield, M.p. =197-199 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.52 (d, J = 8.0 Hz, 1H), 7.61-7.52 (m, 3H), 7.50 – 7.44 (m, 3H), 7.36 (d, J = 7.5 Hz, 1H), 7.19-7.13 (m, 3H), 7.07 (d, J = 6.9 Hz, 2H), 4.35 (q, J = 7.8 Hz, 1H), 4.14 (q, J = 7.7 Hz, 1H), 3.04–2.98 (m, 1H), 2.73–2.67 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.88, 142.59, 140.60, 137.01, 132.72, 132.04, 131.80, 130.70, 128.89, 128.41, 128.25, 127.59, 127.52, 127.22, 126.24, 126.11, 124.83, 112.08, 100.01, 69.36, 32.22; **HRMS** (ESI) m/z calcd for $C_{23}H_{17}NNaO_2^+$ $[M+Na]^+$ 362.1157, found 362.1158.

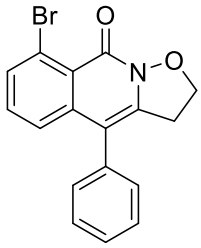
8-chloro-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2l). [2]



White solid, 45 mg, 75% yield, M.p. = 207-209 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.51 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 7.6 Hz, 1H), 7.40-7.42 (m, 4H), 7.27 (d, J = 6.7 Hz, 2H), 4.54 (t, J = 7.6 Hz, 2H), 3.24 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.12, 137.66, 135.25, 135.03, 132.48, 130.51, 130.18, 128.78, 128.24, 127.67, 126.94, 126.47, 111.44, 69.50, 33.19.

8-bromo-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2m).

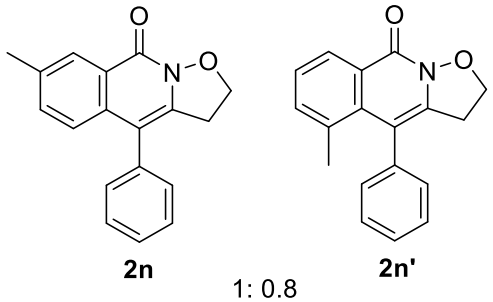


White solid, 57 mg, 84% yield, M.p. = 196-198°C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.57 (d, J = 8.0 Hz, 1H), 7.90 – 7.73 (m, 1H), 7.40-7.38 (m, 3H), 7.29-7.24 (m, 3H), 4.53 (t, J = 7.7 Hz, 2H), 3.23 (t, J = 7.7 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.15, 139.20, 137.14, 135.40, 133.55, 130.80, 128.96, 128.34, 127.88, 127.65, 126.78, 118.83, 112.30, 69.52, 33.37; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}BrNNaO_2^+$ $[M+Na]^+$ 363.9949, found

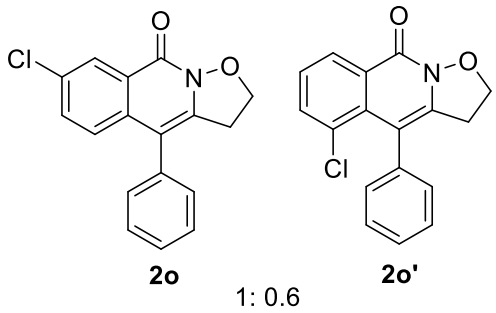
363.9947.

7-methyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-b]isoquinolin-9-one (2n). [2]
5-methyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-b]isoquinolin-9-one (2n'). [2]



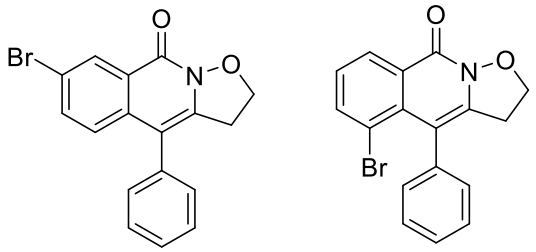
White solid; 94% yield.
^{1}H NMR (500 MHz $CDCl_3$): δ 8.47 (d, J = 7.0 Hz, 0.77H), 8.33 (s, 1H), 7.51-7.25 (m, 12.6H), 4.56 (t, J = 7.3 Hz, 2H), 4.51 (t, J = 7.6 Hz, 1.61H), 3.37 (t, J = 7.4 Hz, 2H), 3.20 (t, J = 7.4 Hz, 1.61H), 2.46 (s, 3H), 1.85 (s, 2.34H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.07, 153.83, 139.16, 136.44, 135.52, 135.10, 134.67, 134.24, 133.83, 133.40, 133.12, 131.46, 130.21, 128.81, 128.53, 127.95, 127.78, 127.44, 126.94, 126.10, 126.03, 124.60, 112.96, 69.55, 69.37, 33.03, 32.28, 23.83, 21.17.

7-chloro-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2o). [2]
5-chloro-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2o'). [2]



White solid; 99% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.60-8.50 (m, 0.56H) , 8.51 (d, J = 2.2 Hz, 1H), 7.62– 7.25(m, 11.3H), 4.59 (t, J = 7.6 Hz, 2H), 4.55 (t , 1.20H), 3.39 (t, J = 7.6 Hz, 2H), 3.25 (t, 1.20H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.18, 152.82, 137.68, 134.53, 134.48, 132.87, 132.62, 132.53, 132.07, 130.56, 130.19, 130.15, 129.02, 128.83, 128.29, 128.27, 69.67, 69.50, 33.21, 32.38.

7-bromo-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2p).
5-bromo-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2p').

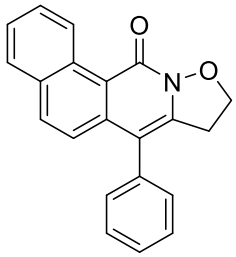


2p 1: 0.5 **2p'**

White solid; 84%.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (d, J = 2.0 Hz, 1H), 8.59 (dd, J = 8.0, 1.1 Hz, 0.47H), 7.84 (dd, J =7.6, 1.1 Hz, 0.48H), 7.61 (dd, J = 8.7, 2.1 Hz, 1H), 7.53-7.46 (m, 3H), 7.40-7.42 (m,1.49H), 7.34-7.328(m, 3H), 7.27 – 7.23 (m, 1.53H), 4.59 (t, J = 7.6 Hz, 2H), 4.54 (t, J = 7.9 Hz, 1H), 3.39 (t, J = 7.6 Hz, 2H), 3.26 (t, J = 7.7 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 153.22, 152.74, 139.27, 137.21, 135.46, 134.91, 134.82, 134.47, 133.64, 133.11, 130.87, 130.20, 129.96, 129.07, 128.41, 128.35, 127.94, 127.72, 127.65, 126.83, 126.50, 120.52, 118.90, 112.70, 112.37, 69.71, 69.56, 33.44, 32.47; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}BrNNaO_2^+$ $[M+Na]^+$ 363.9949, found 363.9943.

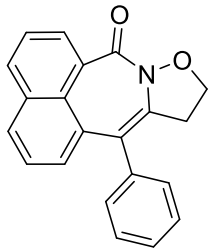
12-phenyl-10,11-dihydro-7*H*-isoxazolo[2,3-*a*]naphtho[1,8-*de*]azepin-7-one (2q).



White solid,36 mg, 57% yield, M.p. = 232-234 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 10.33 (d, J = 8.7 Hz, 1H), 7.86 (d, J = 8.8 Hz, 2H), 7.75 (t, J = 7.6 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.3 Hz, 2H), 7.47 (t, J = 7.3 Hz, 1H), 7.41 – 7.30 (m, 3H), 4.63 (t, J = 7.8 Hz, 2H), 3.46 (t, J = 7.7 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.74, 137.27, 135.58, 133.58, 132.83, 132.19, 131.76, 130.56, 129.00, 128.28, 128.13, 127.90, 127.57, 126.50, 122.64, 119.64, 112.87, 100.00, 69.36, 32.96; **HRMS** (ESI) m/z calcd for $C_{21}H_{15}NNaO_2^+$ $[M+Na]^+$ 336.1000, found 336.1002.

7-phenyl-8,9-dihydro-12*H*-benzo[*h*]isoxazolo[2,3-*b*]isoquinolin-12-one (2q').

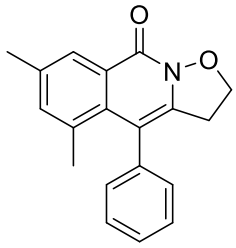


White solid, 25 mg, 40% yield, M.p. = 230-232 °C;

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.53 (d, J = 8.8 Hz, 1H), 7.82 (t, J = 7.6 Hz, 2H), 7.52-7.43 (m, 5H), 7.33 (d, J = 6.1 Hz, 2H), 7.06 (t, J = 7.8 Hz, 1H), 4.60 (t, J = 7.7 Hz, 2H), 3.34 (t, J = 7.8 Hz, 2H); **^{13}C**

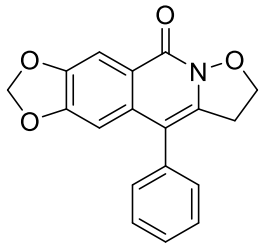
NMR (125 MHz, $CDCl_3$): δ 153.87, 139.51, 135.92, 134.78, 134.03, 129.78, 129.58, 129.12, 128.72, 128.07, 127.80, 127.45, 125.33, 125.12, 123.45, 113.13, 69.66, 33.52; **HRMS** (ESI) m/z calcd for $C_{21}H_{15}NNaO_2^+$ $[M+Na]^+$ 336.1000, found 336.1002.

5,7-dimethyl-4-phenyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2r).



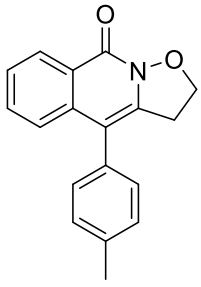
White solid, 57 mg, 97% yield, M.p. = 179-181 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.29 (s, 1H), 7.41 (q, J = 7.2, 6.7 Hz, 3H), 7.30 – 7.26 (m, 2H), 7.17 (s, 1H), 4.53-4.48 (m, 2H), 3.19 (t, J = 7.6 Hz, 2H), 2.42 (s, 3H), 1.82 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.03, 139.27, 137.10, 136.04, 134.51, 132.36, 131.98, 130.14, 128.49, 127.70, 127.48, 125.67, 112.98, 69.38, 32.88, 23.65, 20.85; **HRMS** (ESI) m/z calcd for $C_{19}H_{17}NNaO_2^+$ $[M+Na]^+$ 314.1157, found 314.1158.

10-phenyl-8,9-dihydro-5*H*-[1,3]dioxolo[4,5-*g*]isoxazolo[2,3-*b*]isoquinolin-5-one (2s).



White solid, 55 mg, 90% yield, M.p. = 236-238 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83 (s, 1H), 7.48 (t, J = 7.3 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H), 7.33 – 7.28 (m, 2H), 6.66 (s, 1H), 6.01 (s, 2H), 4.54 (t, J = 7.6 Hz, 2H), 3.33 (t, J = 7.6 Hz, 2H) ; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.13, 151.56, 147.38, 135.18, 133.47, 131.19, 130.16, 128.91, 128.05, 121.83, 112.66, 105.11, 102.76, 101.82, 69.61, 32.39; **HRMS** (ESI) m/z calcd for $C_{18}H_{13}NNaO_4^+$ $[M+Na]^+$ 330.0742, found 330.0743.

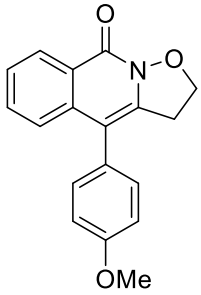
4-(p-tolyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2t). [2]



White solid, 52 mg, 94% yield, M.p. = 195-197 °C;

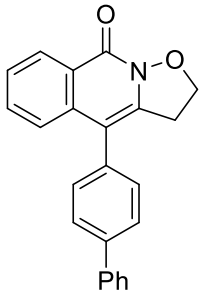
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.52 (d, J = 8.0 Hz, 1H), 7.55-7.51 (m, 1H), 7.46 (t, J = 7.2 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 7.30 (d, J = 7.9 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 4.56 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H), 2.44 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.91, 137.87, 136.34, 132.44, 131.93, 131.53, 130.13, 129.59, 127.40, 126.28, 124.76, 112.99, 69.54, 32.44,21.27;

4-(4-methoxyphenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2u). [2]



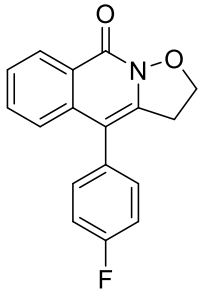
White solid, 36 mg, 62% yield, M.p. =238-240°C;
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.54 (d, J = 8.0 Hz, 1H), 7.58 – 7.53 (m, 1H), 7.52 – 7.45 (m, 1H), 7.39 (d, J = 7.9 Hz, 1H), 7.28-7.26 (m, 2H), 7.06 – 6.99 (m, 2H), 4.57 (t, J = 7.6 Hz, 2H), 3.89 (s, 3H), 3.39 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 159.37, 153.96, 136.54, 132.53, 131.58, 131.42, 127.47, 127.08, 126.29, 126.26, 124.75, 114.34, 112.72, 69.55, 55.38, 32.46.

4-([1,1'-biphenyl]-4-yl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2v).



White solid, 62 mg, 92% yield, M.p. = 146-148 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.54 (d, J = 8.1 Hz, 1H), 7.72 (d, J = 8.2 Hz, 2H), 7.67 (d, J = 8.5 Hz, 2H), 7.60 – 7.53 (m, 1H), 7.51 – 7.46 (m, 4H), 7.44-7.37 (m, 3H), 4.58 (t, J = 7.6 Hz, 2H), 3.44 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.95, 140.91, 140.38, 136.16, 133.92, 132.65, 131.69, 130.75, 128.95, 127.68, 127.58, 127.48, 127.08, 126.35, 126.31, 124.75, 112.68, 69.63, 32.55; **HRMS** (ESI) m/z calcd for $C_{23}H_{17}NNaO_2^+$ $[M+Na]^+$ 362.1157, found 362.1156.

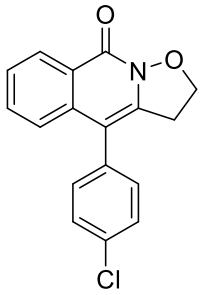
4-(4-fluorophenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2w).



White solid, 46 mg, 81% yield, M.p. = 218-220 °C;

^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.53 (d, J = 8.0 Hz, 1H), 7.59 – 7.53 (m, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.37 – 7.28 (m, 3H), 7.21 (m, 2H), 4.58 (t, J = 7.6 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 162.51 (d, $^1J_{C,F}$ = 247.9 Hz), 153.93, 136.17, 132.77, 132.05 (d, $^3J_{C,F}$ = 8.1 Hz), 131.75, 130.88 (d, $^4J_{C,F}$ =3.5 Hz), 127.54, 126.42, 126.27, 124.47, 116.04 (d, $^2J_{C,F}$ = 21.5 Hz), 111.94, 69.53, 32.44; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.39; **HRMS** (ESI) m/z calcd for $C_{17}H_{12}FNNaO_2^+$ $[M+Na]^+$ 304.0750, found 304.0753.

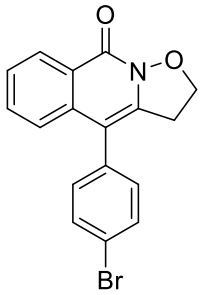
4-(4-chlorophenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2x). [2]



White solid, 58 mg, 97% yield, M.p. = 233-235 °C;

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.51 (d, J = 8.0 Hz, 1H), 7.58-7.53 (m, 1H), 7.50-7.45 (m, 3H), 7.33-7.27 (m, 3H), 4.58 (t, J = 7.6 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 125 MHz): δ 153.87, 135.88, 134.18, 133.43, 132.75, 131.77, 131.69, 129.22, 127.52, 126.44, 126.24, 124.39, 111.72, 69.53, 32.43.

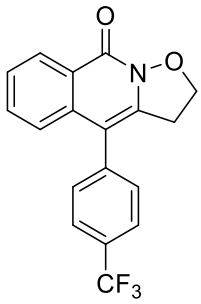
4-(4-bromophenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2y). [2]



White solid, 64 mg, 94% yield, M.p. =224-226°C;

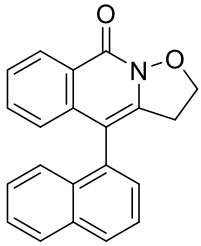
^{1}H NMR ($CDCl_3$, 400MHz): δ 8.63 – 8.43 (m, 1H), 7.71 – 7.60 (m, 2H), 7.59 – 7.44 (m, 2H), 7.35 – 7.28 (m, 1H), 7.26-7.23 (m, 2H), 4.67 – 4.46 (m, 2H), 3.38 (t, J = 7.5 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 153.89, 135.80, 133.93, 132.70, 132.21, 132.01, 131.81, 127.55, 126.47, 126.25, 124.39, 122.34, 111.75, 69.54, 32.45.

4-(4-(trifluoromethyl)phenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2z).



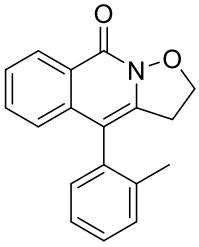
White solid, 66 mg, 99% yield, M.p. = 199-201 °C;
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.55 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.60 – 7.55 (m, 1H), 7.54 – 7.46 (m, 3H), 7.30 (d, J = 8.7 Hz, 1H), 4.60 (t, J = 7.5 Hz, 2H), 3.40 (t, J = 7.6 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 153.95, 138.94, 135.61, 132.94, 131.95, 130.84, 130.40 (q, $^2J_{C,F}$ = 32.5 Hz), 127.70, 126.64, 126.33, 125.98 (q, $^3J_{C,F}$ = 3.6 Hz), 124.28, 124.01 (q, $^1J_{C,F}$ = 270.1 Hz), 111.57, 69.54, 32.49; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.62; **HRMS** (ESI) m/z calcd for $C_{18}H_{12}F_3NNaO_2^+$ $[M+Na]^+$ 354.0718, found 354.0718.

4-(naphthalen-1-yl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2aa). [2]



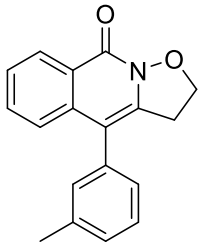
White solid, 62 mg, 99% yield, M.p. = 222-224 °C;
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59-8.56 (m, 1H), 7.97 (t, J = 7.8 Hz, 2H), 7.62-7.58 (m, 1H), 7.56 – 7.34 (m, 6H), 7.07 – 6.95 (m, 1H), 4.61-4.50 (m, 2H), 3.34-3.26 (m, 1H), 3.14-3.06 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 154.17, 136.86, 133.96, 133.50, 132.41, 132.28, 131.74, 129.00, 128.70, 128.66, 127.41, 126.80, 126.37, 126.32, 126.19, 125.73, 125.26, 125.10, 110.85, 69.55, 32.26.

4-(*o*-tolyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ab). [2]



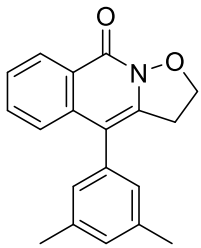
White solid, 55 mg, 99% yield, M.p. = 140-142 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.54-8.51 (m, 1H), 7.54 – 7.42 (m, 2H), 7.39-7.34 (m ,2H), 7.31-7.28 (m, 1H), 7.19 (d, J = 7.3 Hz, 1H), 7.05 (d, J = 7.8 Hz, 1H), 4.57 (t, J = 7.7 Hz, 2H), 3.37 – 3.27 (m, 1H), 3.19-3.12 (m, 1H), 2.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 154.00, 137.59, 136.08, 134.05, 132.35, 131.69, 130.58, 130.46, 128.60, 127.40, 126.41, 126.24, 124.50, 112.21, 69.45, 32.12, 19.63.

4-(*m*-tolyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ac).



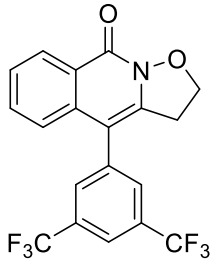
White solid, 52 mg, 94% yield, M.p. = 189-191 °C;
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.53 (d, J = 8.1 Hz, 1H), 7.59 – 7.51 (m, 1H), 7.52 – 7.43 (m, 1H), 7.39 (t, J = 7.7 Hz, 2H), 7.26 (d, J = 7.5 Hz, 1H), 7.17-7.12 (m, 2H), 4.57 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H), 2.43 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz): δ 153.95, 138.63, 136.27, 134.91, 132.40, 131.59, 130.90, 128.84, 128.80, 127.42, 127.33, 126.26, 124.81, 113.20, 69.58, 32.46, 21.47; **HRMS** (ESI) m/z calcd for $C_{18}H_{15}NNaO_2^+$ $[M+Na]^+$ 300.1000, found 300.1003.

4-(3,5-dimethylphenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ad). [2]



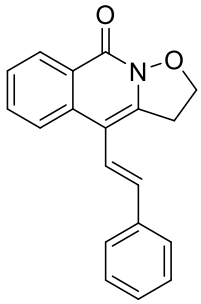
White solid, 55 mg, 95% yield, M.p. = 249-251 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.53 (d, J = 7.9 Hz, 1H), 7.54 (t, J = 7.1 Hz, 1H), 7.47 (t, J = 7.3 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 7.08 (s, 1H), 6.95 (s, 2H), 4.57 (t, J = 7.6 Hz, 2H), 3.40 (t, J = 7.6 Hz, 2H), 2.38 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.94, 138.47, 136.35, 134.82, 132.28, 131.54, 129.69, 127.97, 127.37, 126.20, 124.89, 113.34, 69.58, 32.46, 21.35.

4-(3,5-bis(trifluoromethyl)phenyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ae).



White solid, 72 mg, 90% yield, M.p. = 238-240 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.54 (d, J = 8.8 Hz, 1H), 7.99 (s, 1H), 7.87 (s, 2H), 7.62-7.58 (m, 1H), 7.52 (t, J = 7.5 Hz, 1H), 7.19 (d, J = 8.1 Hz, 1H), 4.62 (t, J = 7.6 Hz, 2H), 3.41 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.82, 137.43, 135.09, 133.63, 132.53 (q, $^2J_{C,F}$ = 32.5 Hz) ,132.27, 130.58, 127.87, 126.88, 126.25, 123.58, 123.02 (d, $^1J_{C,F}$ = 272.3 Hz), 109.95, 109.78, 69.41, 32.44; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.78; **HRMS** (ESI) m/z calcd for $C_{19}H_{11}F_6NNaO_2^+$ $[M+Na]^+$ 422.0592, found 422.0588.

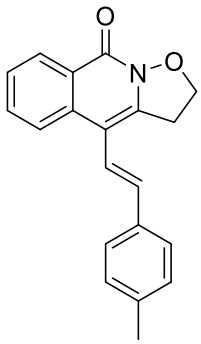
(*E*)-4-styryl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2af).



White solid, 44 mg, 75% yield, M.p. = 206-208 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.52 (d, J = 7.3 Hz, 1H), 7.82 (d, J = 8.2 Hz, 1H), 7.69 – 7.64 (m, 1H), 7.54-7.48 (m, 3H), 7.41 (t, J = 7.6 Hz, 2H), 7.33 (t, J = 7.4 Hz, 1H), 7.22 (d, J = 16.3 Hz, 1H), 6.76 (d, J = 16.2 Hz, 1H), 4.60 (t, J = 7.5 Hz, 2H), 3.69 (t, J = 7.5 Hz, 2H).; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.89, 136.81, 135.39, 134.10, 132.37, 131.83, 128.88, 128.21, 127.71, 126.56, 126.39, 126.20, 123.52, 121.45, 109.31, 69.97, 32.20; **HRMS** (ESI) m/z calcd for $C_{19}H_{15}NNaO_2^+$ $[M+Na]^+$ 312.1000, found 312.1004.

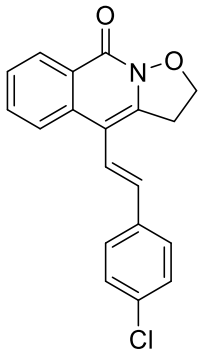
(*E*)-4-(4-methylstyryl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ag).



White solid, 49 mg, 80% yield, M.p. = 210-212 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.51 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 8.2 Hz, 1H), 7.66 (t, J = 7.6 Hz, 1H), 7.51 (t, J = 7.6 Hz, 1H), 7.42 (d, J = 7.7 Hz, 2H), 7.21 (d, J = 7.8 Hz, 2H), 7.17 (d, J = 16.2 Hz, 1H), 6.73 (d, J = 16.2 Hz, 1H), 4.59 (t, J = 7.4 Hz, 2H), 3.68 (t, J = 7.5 Hz, 2H), 2.39 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.87, 138.23, 135.45, 134.07, 132.19, 131.78, 129.56, 127.68, 126.51, 126.30, 126.20, 123.55, 120.39, 69.45, 109.51, 33.02, 21.28; **HRMS** (ESI) m/z calcd for $C_{20}H_{17}NNaO_2^+$ $[M+Na]^+$ 326.1157, found 326.1164.

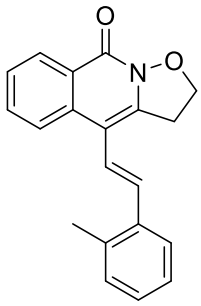
(*E*)-4-(4-chlorostyryl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ah).



White solid, 55 mg, 84% yield, M.p. = 202-204 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.49 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 8.2 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.44 (d, J = 8.1 Hz, 2H), 7.36 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 16.2 Hz, 1H), 6.70 (d, J = 16.2 Hz, 1H), 4.59 (t, J = 7.3 Hz, 2H), 3.67 (t, J = 7.5 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.79, 135.27, 135.18, 133.78, 132.62, 132.49, 131.82, 128.97, 127.67, 127.49, 126.55, 126.13, 123.36, 122.02, 108.92, 69.37, 33.02; **HRMS** (ESI) m/z calcd for $C_{19}H_{14}ClNNaO_2^+$ $[M+Na]^+$ 346.0611, found 346.0612.

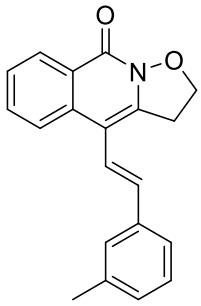
(*E*)-4-(2-methylstyryl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ai).



White solid, 55 mg, 84% yield, M.p. = 222-224 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.51 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 8.2 Hz, 1H), 7.69 – 7.61 (m, 2H), 7.50 (t, J = 7.5 Hz, 1H), 7.29-7.20 (m, 3H), 7.09 (d, J = 16.1 Hz, 1H), 6.99 (d, J = 16.1 Hz, 1H), 4.60 (t, J = 7.4 Hz, 2H), 3.69 (t, J = 7.5 Hz, 2H), 2.41 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.86, 136.05, 135.64, 135.41, 132.34, 132.18, 131.81, 130.57, 128.12, 127.67, 126.52, 126.41, 126.19, 125.44, 123.50, 122.75, 109.65, 69.43, 33.08, 19.98 ; **HRMS** (ESI) m/z calcd for $C_{20}H_{17}NNaO_2^+$ $[M+Na]^+$ 326.1157, found 326.1163.

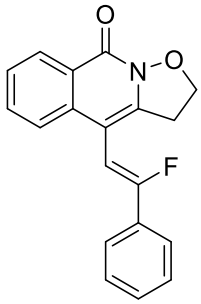
(*E*)-4-(3-methylstyryl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2aj).



White solid, 50 mg, 82% yield, M.p. = 228-230°C;

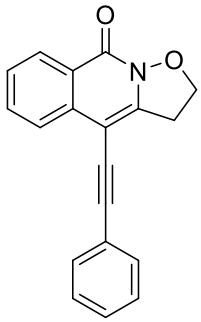
^{1}H NMR (500 MHz $CDCl_3$): δ 8.50 (d, J = 8.1 Hz, 1H), 7.80 (d, J = 8.2 Hz, 1H), 7.68 – 7.61 (m, 1H), 7.53 – 7.46 (m, 1H), 7.36 – 7.27 (m, 3H), 7.18 (d, J = 16.3 Hz, 1H), 7.14 (d, J = 7.2 Hz, 1H), 6.72 (d, J = 16.3 Hz, 1H), 4.58 (t, J = 7.5 Hz, 2H), 3.66 (t, J = 7.5 Hz, 2H), 2.40 (s, 3H).; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.81, 138.45, 136.70, 135.34, 134.15, 132.26, 131.73, 128.96, 128.72, 127.61, 127.02, 126.46, 126.12, 123.49, 121.13, 109.32, 69.39, 32.96, 21.39; **HRMS** (ESI) m/z calcd for $C_{20}H_{17}NNaO_2^+$ $[M+Na]^+$ 326.1157, found 326.1160.

(*Z*)-4-(2-fluoro-2-phenylvinyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ak).



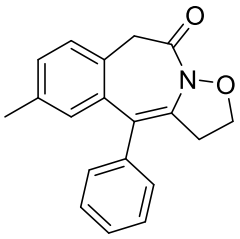
White solid, 49 mg, 79% yield, M.p. = 200-202 °C;
^{1}H NMR (500 MHz $CDCl_3$): δ 8.51 (d, J = 8.1 Hz, 1H), 7.76 – 7.60 (m, 4H), 7.55 – 7.39 (m, 4H), 6.48 (d, J = 37.1 Hz, 1H), 4.61 (t, J = 7.6 Hz, 2H), 3.55 (t, J = 7.6 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 157.88 (d, $^1J_{C,F}$ = 255.5 Hz), 153.99, 135.47, 134.02, 131.75, 131.51, 129.73, 128.76, 127.62, 126.43, 125.99, 124.54 (d, $^3J_{C,F}$ = 7.2 Hz), 123.57, 103.50, 98.27 (d,$^2J_{C,F}$ = 15.6 Hz), 69.57, 33.10; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -109.27; **HRMS** (ESI) m/z calcd for $C_{19}H_{14}FNNaO_2$ $[M+Na]^+$ 330.0906, found 330.0914.

4-(phenylethynyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2al).



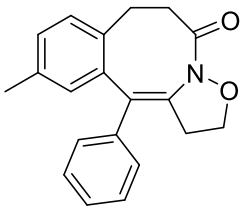
White solid, 40 mg, 70% yield, M.p. = 158-160 °C;
^{1}H NMR (500 MHz $CDCl_3$): δ 8.48 (d, J = 9.6 Hz, 1H), 8.03 (d, J = 8.1 Hz, 1H), 7.73 (t, J = 7.6 Hz, 1H), 7.63 – 7.57 (m, 2H), 7.54 (t, J = 7.6 Hz, 1H), 7.47 – 7.37 (m, 3H), 4.68 (t, J = 7.8 Hz, 2H), 3.78 (t, J = 7.8 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.78, 138.68, 135.38, 132.68, 131.42, 128.63, 128.54, 127.42, 126.90, 125.55, 124.76, 122.84, 96.07, 95.19, 82.54, 69.46, 33.21; **HRMS** (ESI) m/z calcd for $C_{19}H_{13}NNaO_2^+$ $[M+Na]^+$ 310.0844, found 310.0850.

6-methyl-4-phenyl-3,9-dihydrobenzo[*d*]isoxazolo[2,3-*a*]azepin-10(2*H*)-one (2am).



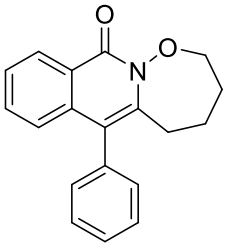
White solid, 34 mg, 56% yield, M.p. =178-180 °C;
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44 (t, J = 7.4 Hz, 2H), 7.37 (t, J = 7.3 Hz, 1H), 7.28 (d, J = 7.3 Hz, 2H), 7.19 (d, J = 7.8 Hz, 1H), 7.11 (d, J = 7.8 Hz, 1H), 6.70 (s, 1H), 4.18 (t, J = 7.0 Hz, 2H), 3.63 (s, 2H), 3.13 (t, J = 7.0 Hz, 2H), 2.21 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 169.68, 139.52, 137.13, 136.46, 132.38, 129.98, 129.78, 129.52, 129.33, 128.76, 128.59, 128.43, 128.01, 127.70, 122.10, 69.09, 42.48, 32.26, 21.04; **HRMS** (ESI) m/z calcd for $C_{19}H_{17}NNaO_2^+$ $[M+Na]^+$ 314.1157, found 314.1161.

(*Z*)-10-methyl-12-phenyl-1,2,6,7-tetrahydro-5*H*-benzo[*d*]isoxazolo[2,3-*a*]azocin-5-one (2an).



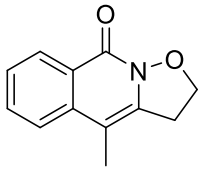
White solid, 34 mg, 54% yield, M.p. = 196-198 °C;
^{1}H NMR (500 MHz $CDCl_3$): δ 7.42 – 7.37 (m, 2H), 7.34-7.30 (m, 3H), 7.16 (d, J = 7.8 Hz, 1H), 7.06 (d, J = 9.0 Hz, 1H), 6.88 (s, 1H), 4.11 (t, J = 7.4 Hz, 2H), 3.10 – 2.98 (m, 6H), 2.24 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 165.05, 134.27,1 32.88, 130.62, 130.36, 127.20, 123.95, 123.39 ,123.08, 122.77, 122.31, 61.82, 32.72, 27.24, 23.52, 15.65; **HRMS** (ESI) m/z calcd for $C_{20}H_{19}NNaO_2^+$ $[M+Na]^+$ 328.1313, found 328.1316.

6-phenyl-2,3,4,5-tetrahydro-11*H*-[1,2]oxazepino[2,3-*b*]isoquinolin-11-one (2ao).



White solid, 47 mg, 80% yield, M.p. = 212-214 °C;
^{1}H NMR (500 MHz $CDCl_3$): δ 8.51 (d, J = 6.9 Hz, 1H), 7.53 – 7.46 (m, 3H), 7.46 – 7.40 (m, 2H), 7.26 (d, J = 6.6 Hz, 2H), 7.04 (d, J = 8.1 Hz, 1H), 4.76 (s, 1H), 4.05 (s, 1H), 2.78 (d, 2H), 2.00 (s, 2H), 1.86 (s, 1H), 1.56 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 158.29, 142.31, 136.83, 136.31, 132.03, 128.80, 127.80, 127.78, 126.00, 125.58, 125.40, 115.32, 78.61, 28.90, 28.64, 25.71; **HRMS** (ESI) m/z calcd for $C_{19}H_{17}NNaO_2^+$ $[M+Na]^+$ 314.1157, found 314.1163.

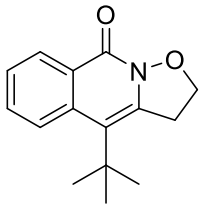
4-methyl-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2ap).



White solid, 29 mg, 71% yield, M.p. = 140-142 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.46 (d, J = 12.5 Hz, 1H), 7.69 – 7.50 (m, 2H), 7.49-7.40 (m, 1H), 4.64 – 4.48 (m, 2H), 3.57 – 3.41 (m, 2H), 2.30-2.20 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.69, 136.33, 131.48, 127.42, 126.29, 125.99, 122.78, 105.59, 69.19, 31.66, 13.65; **HRMS** (ESI) m/z calcd for $C_{12}H_{11}NNaO_2^+$ $[M+Na]^+$ 224.0687, found 224.0688.

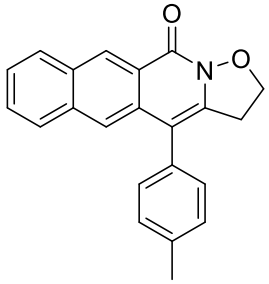
4-(tert-butyl)-2,3-dihydro-9*H*-isoxazolo[2,3-*b*]isoquinolin-9-one (2aq).



White solid, 16 mg, 32% yield, M.p. = 118-120 °C;

^{1}H NMR (500 MHz $CDCl_3$): δ 8.56 (d, J = 8.0 Hz, 1H), 8.20 (d, J = 8.6 Hz, 1H), 7.62 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.5 Hz, 1H), 4.46 (t, J = 7.4 Hz, 2H), 3.83 (t, J = 7.5 Hz, 2H), 1.63 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.62, 135.71, 130.46, 130.14, 127.85, 127.35, 126.91, 125.63, 118.83, 68.45, 35.98, 35.41, 32.94; **HRMS** (ESI) m/z calcd for $C_{15}H_{17}NNaO_2^+$ $[M+Na]^+$ 266.1157, found 266.1160.

4-(p-tolyl)-2,3-dihydro-11*H*-benzo[g]isoxazolo[2,3-*b*]isoquinolin-11-one (1ar)



White solid, 51 mg, 78% yield, M.p. = 229-231 °C;

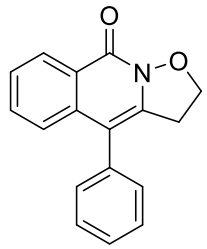
^{1}H NMR (500 MHz $CDCl_3$): δ 8.57 – 8.49 (m, 1H), 7.85-7.79 (m, 2H), 7.60 (dd, J = 9.0, 4.4 Hz, 1H), 7.49-7.44 (m, 1H), 7.31 (d, J = 5.4 Hz, 2H), 7.24 – 7.16 (m, 2H), 7.13 – 7.05 (m, 1H), 4.59 (td, J = 7.8, 2.9 Hz, 2H), 3.34 (td, J = 7.8, 3.1 Hz, 2H), 2.48 (s, 3H).; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 153.88, 137.83, 136.44, 135.92, 134.80, 134.19, 130.29, 129.58, 129.24, 128.70, 127.84, 127.74, 127.45, 125.32, 125.08, 123.48, 113.16, 69.66, 33.54, 21.39; **HRMS** (ESI) m/z calcd for $C_{22}H_{17}NNaO_2^+$ $[M+Na]^+$ 350.1157, found 350.1157.

References

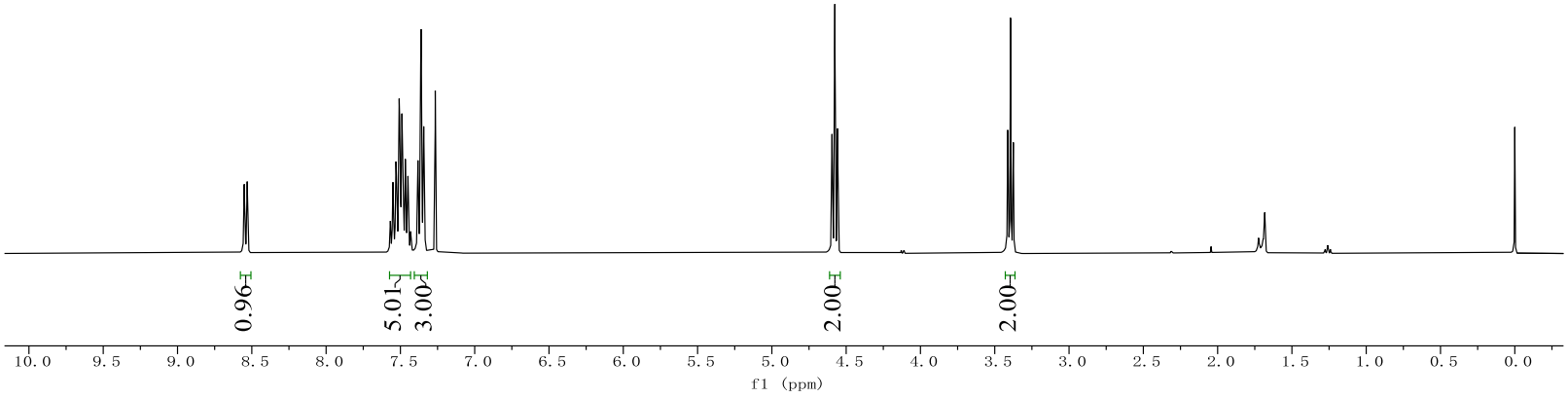
[1] X. Xu, Y. Liu, C.-M. Park, *Angew. Chem., Int. Ed.* **2012**, *51*, 9372–9376.

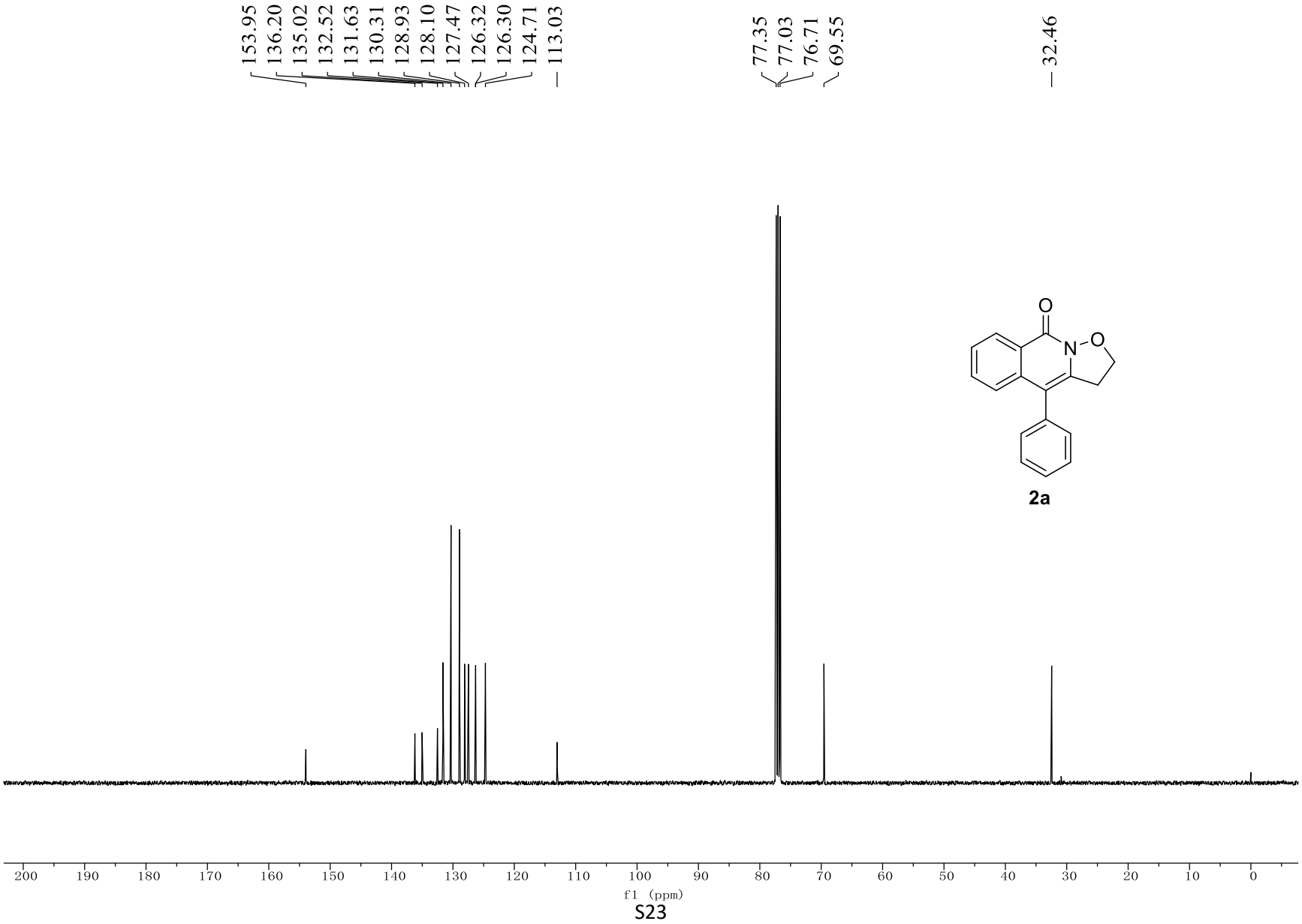
[2] F. Chen, S.-Q. Lai, F.-F. Zhu, Q. Meng, Y. Jiang, W. Yu, B. Han, *ACS Catal.* **2018**, *8*, 8925−8931

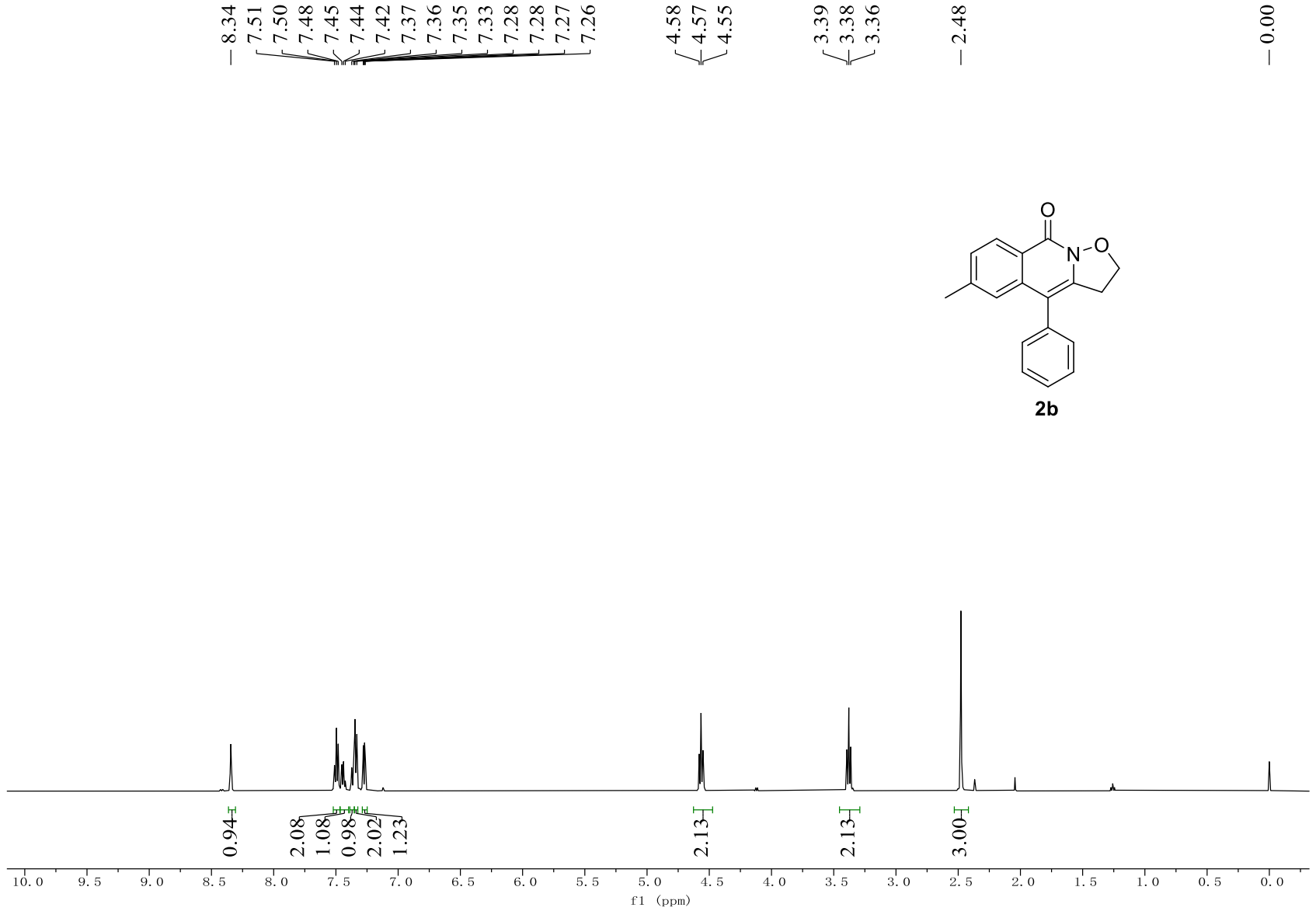
^{1}H NMR ,^{13}C NMR and ^{19}F NMR spectra of 2.



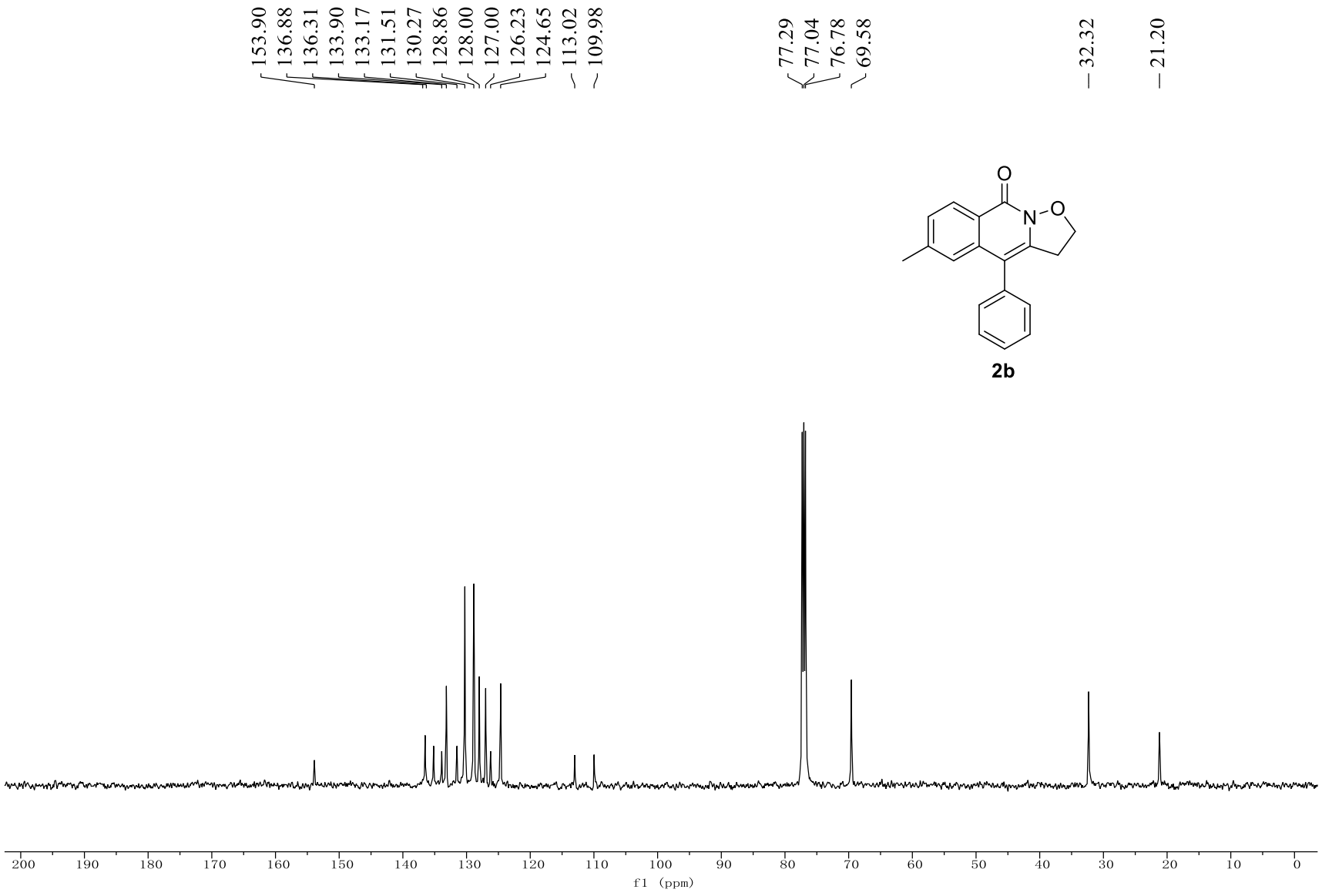
2a

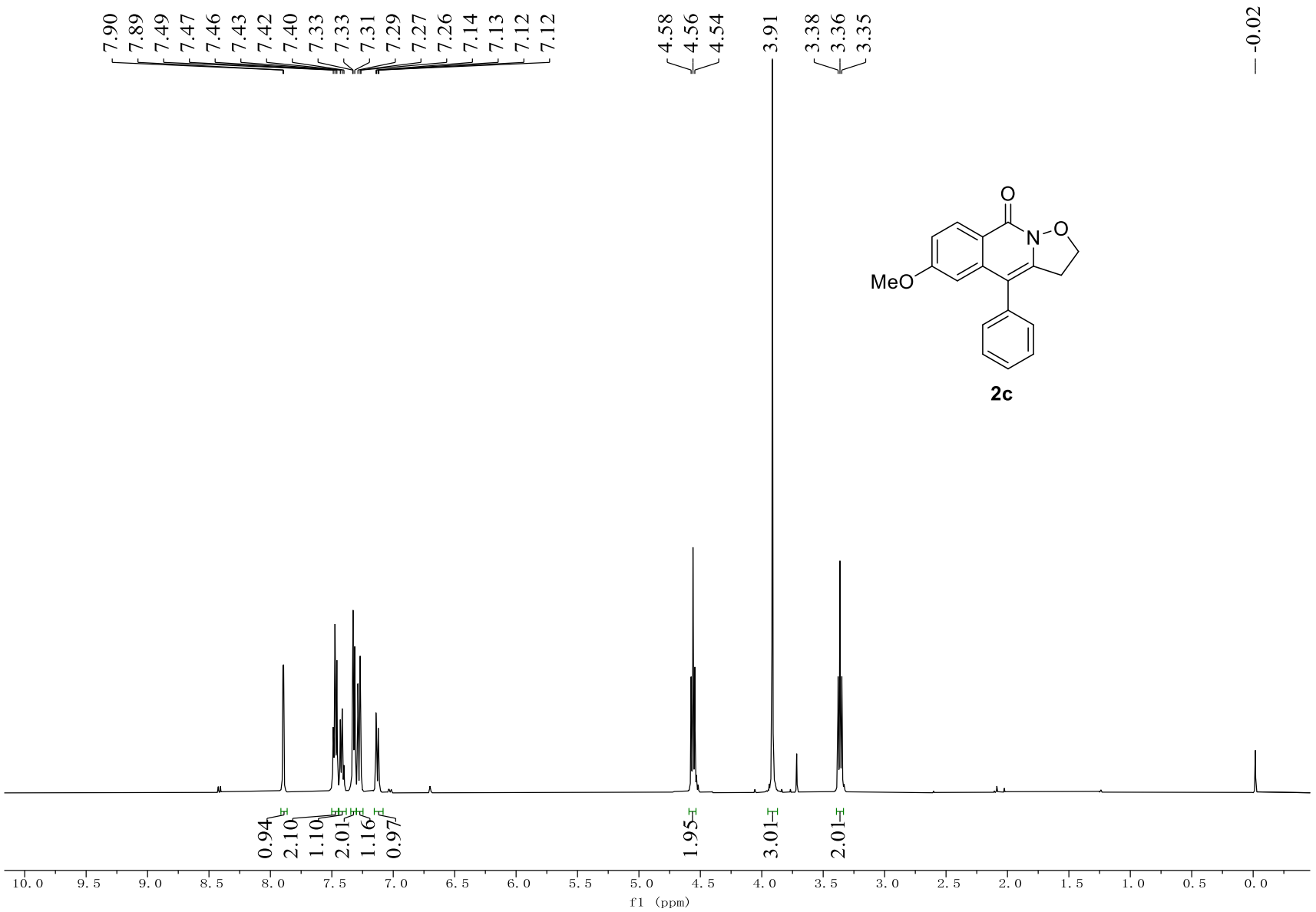


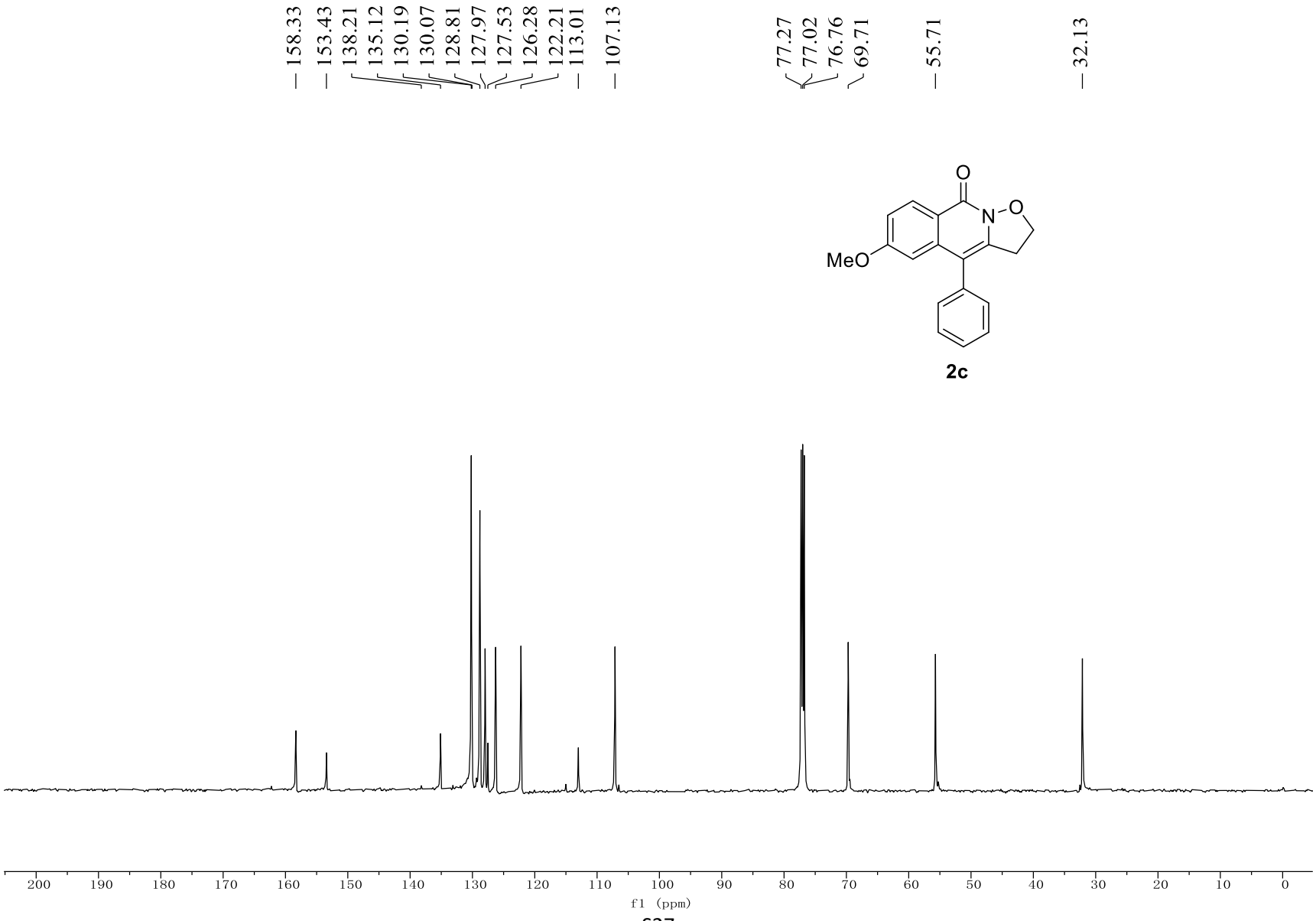

153.95
136.20
135.02
132.52
131.63
130.31
128.93
128.10
127.47
126.32
126.30
124.71
113.03
77.35
77.03
76.71
69.55
32.46
O
N
O
2a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

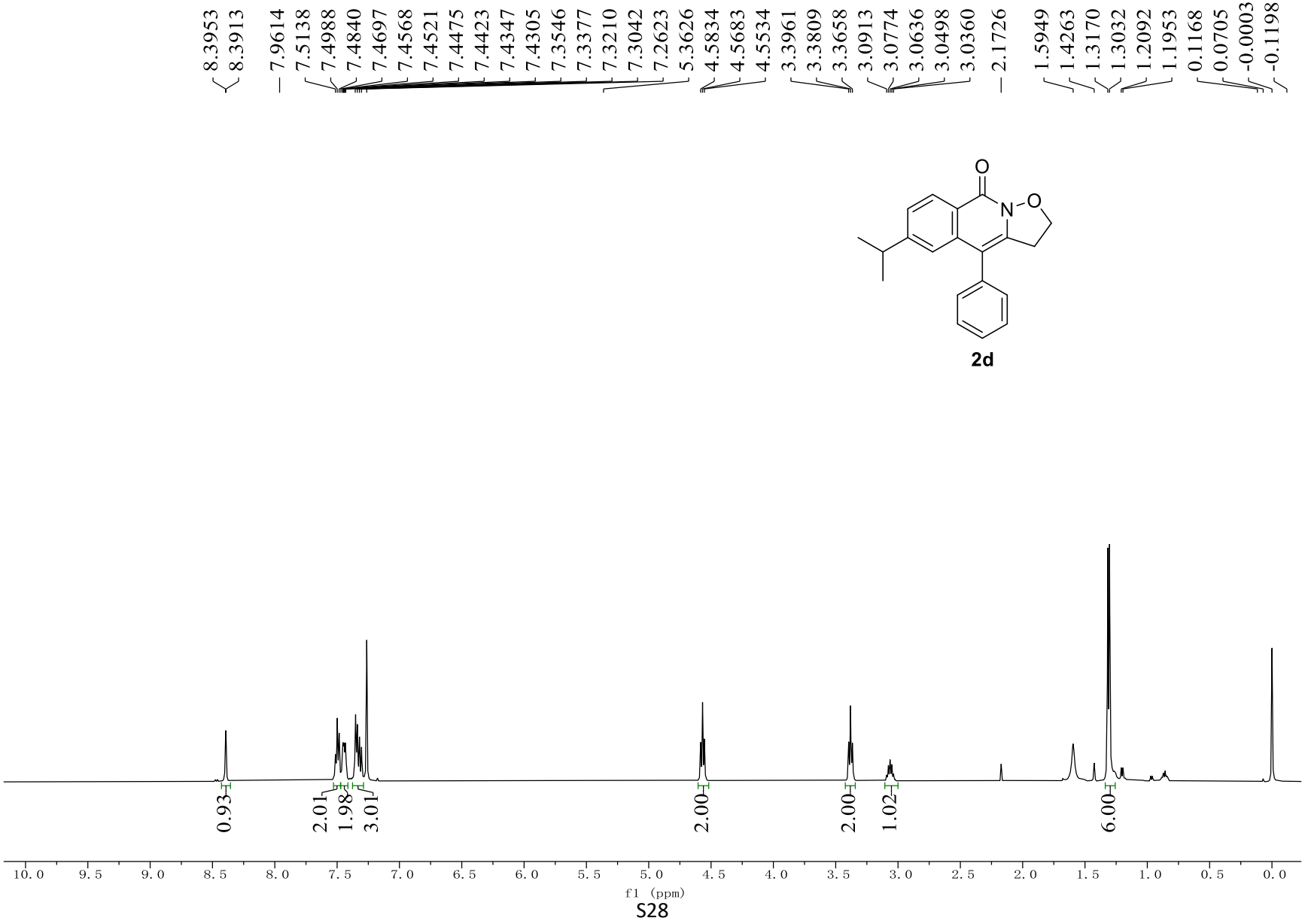

8.34
7.51
7.50
7.48
7.45
7.44
7.42
7.37
7.36
7.35
7.33
7.28
7.28
7.27
7.26
4.58
4.57
4.55
3.39
3.38
3.36
2.48
0.00
O
N
O
0.94
2.08
1.08
0.98
2.02
1.23
2.13
2.13
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

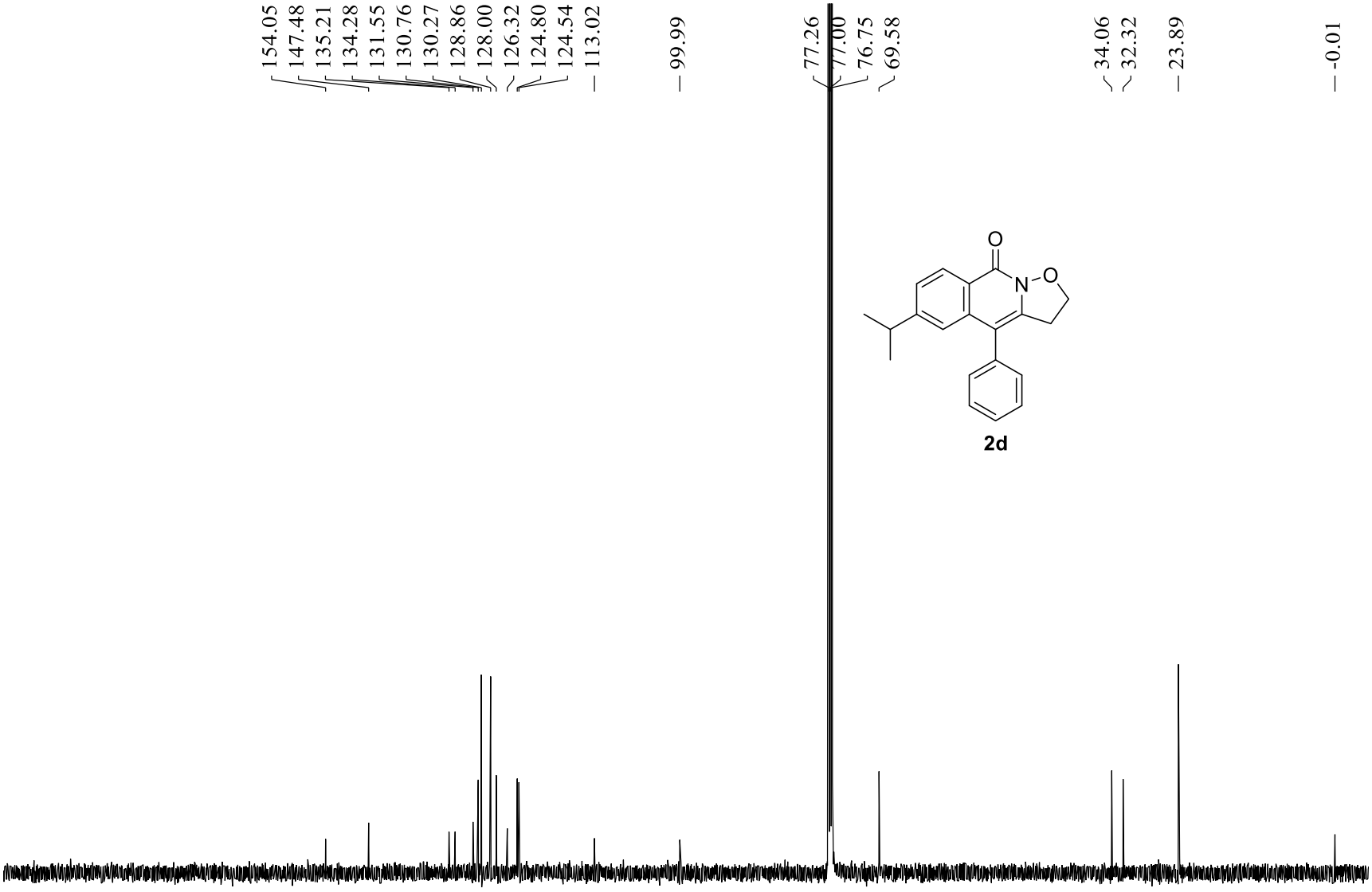
2b

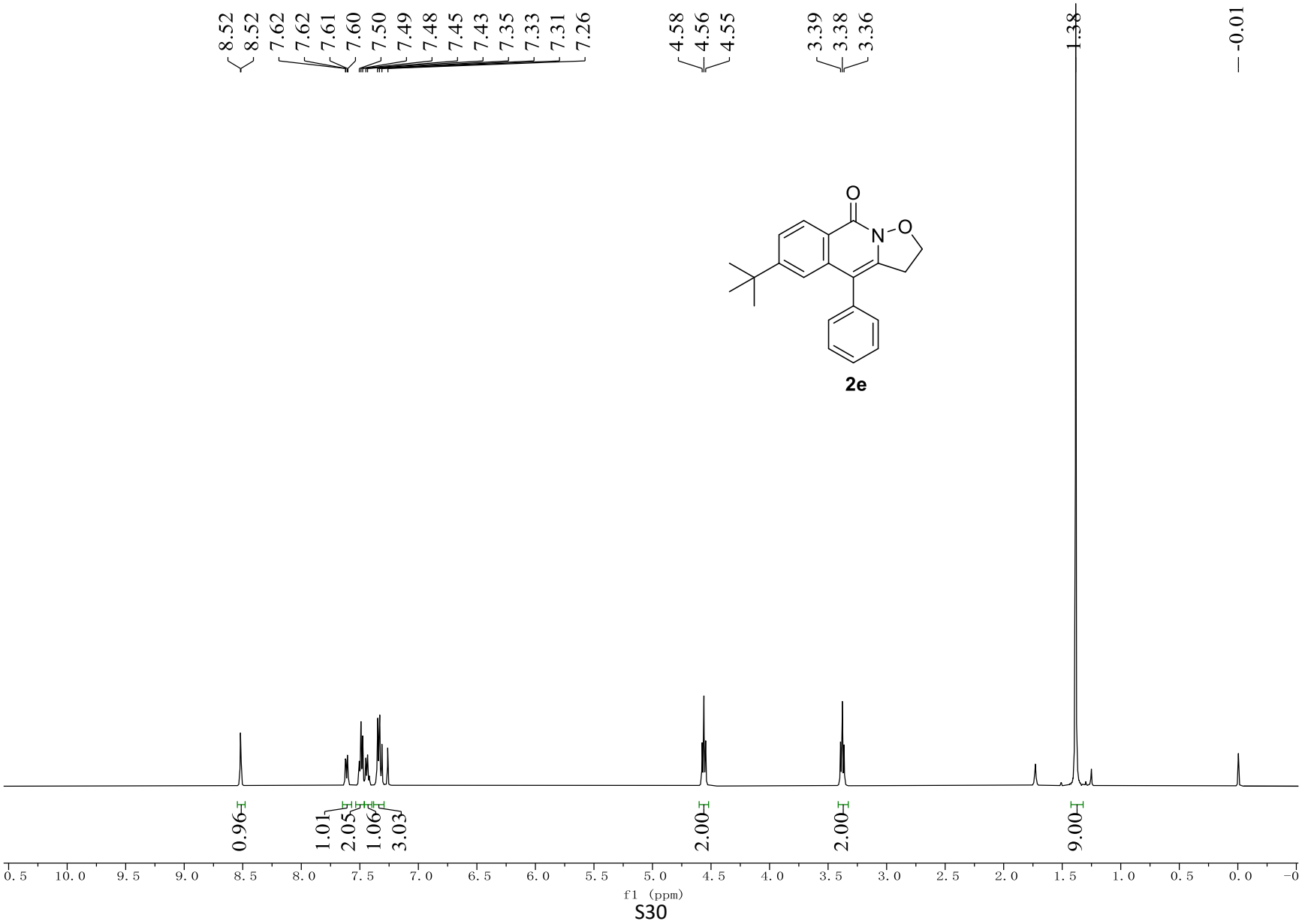

153.90
136.88
136.31
133.90
133.17
131.51
130.27
128.86
128.00
127.00
126.23
124.65
113.02
109.98
77.29
77.04
76.78
69.58
32.32
21.20
O
N
O
2b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

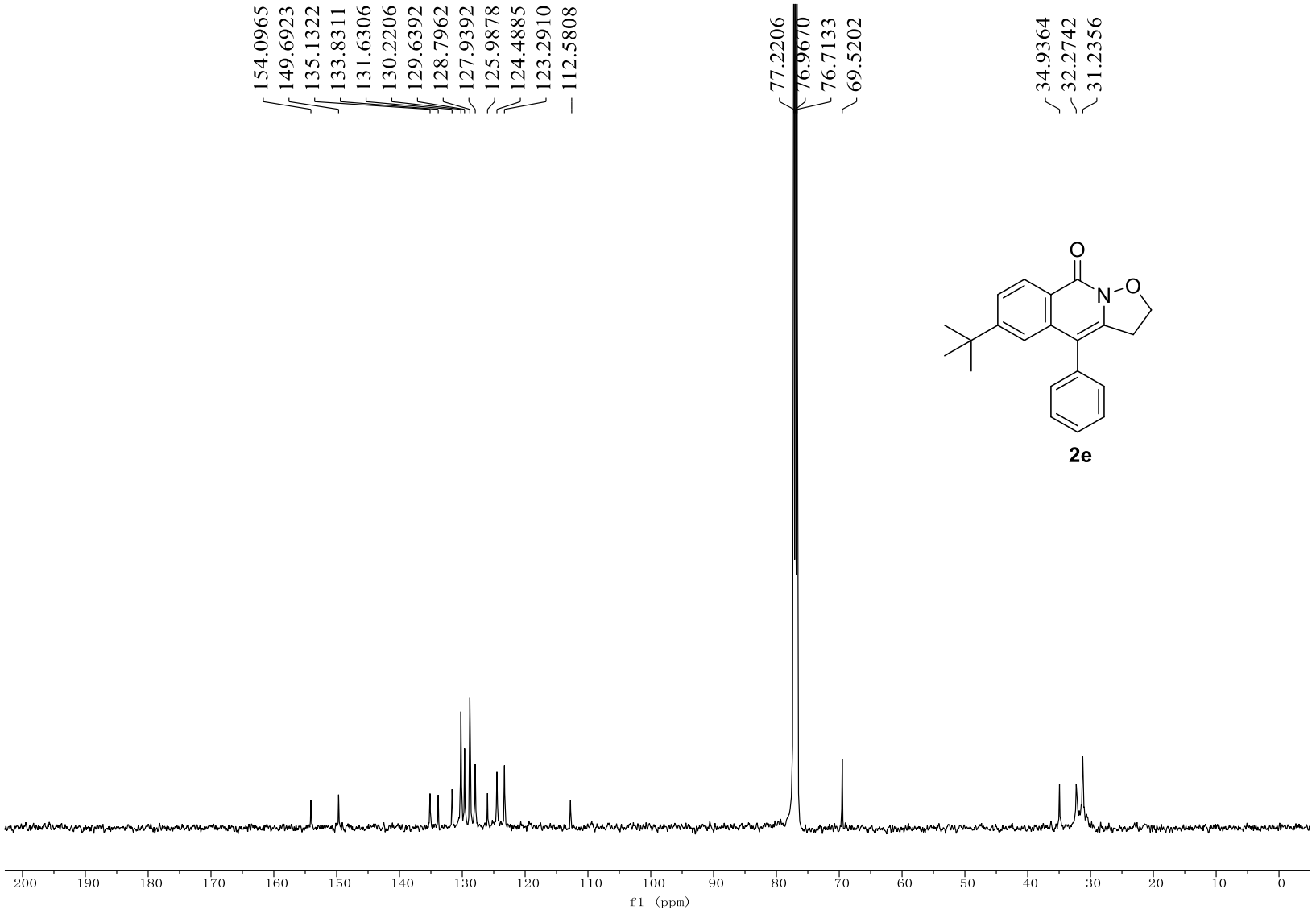

7.90
7.89
7.49
7.47
7.46
7.43
7.42
7.40
7.33
7.33
7.31
7.29
7.27
7.26
7.14
7.13
7.12
7.12
4.58
4.56
4.54
3.91
3.38
3.36
3.35
-0.02
O
N
O
MeO
2c
0.94
2.10
1.10
2.01
1.16
0.97
1.95
3.01
2.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

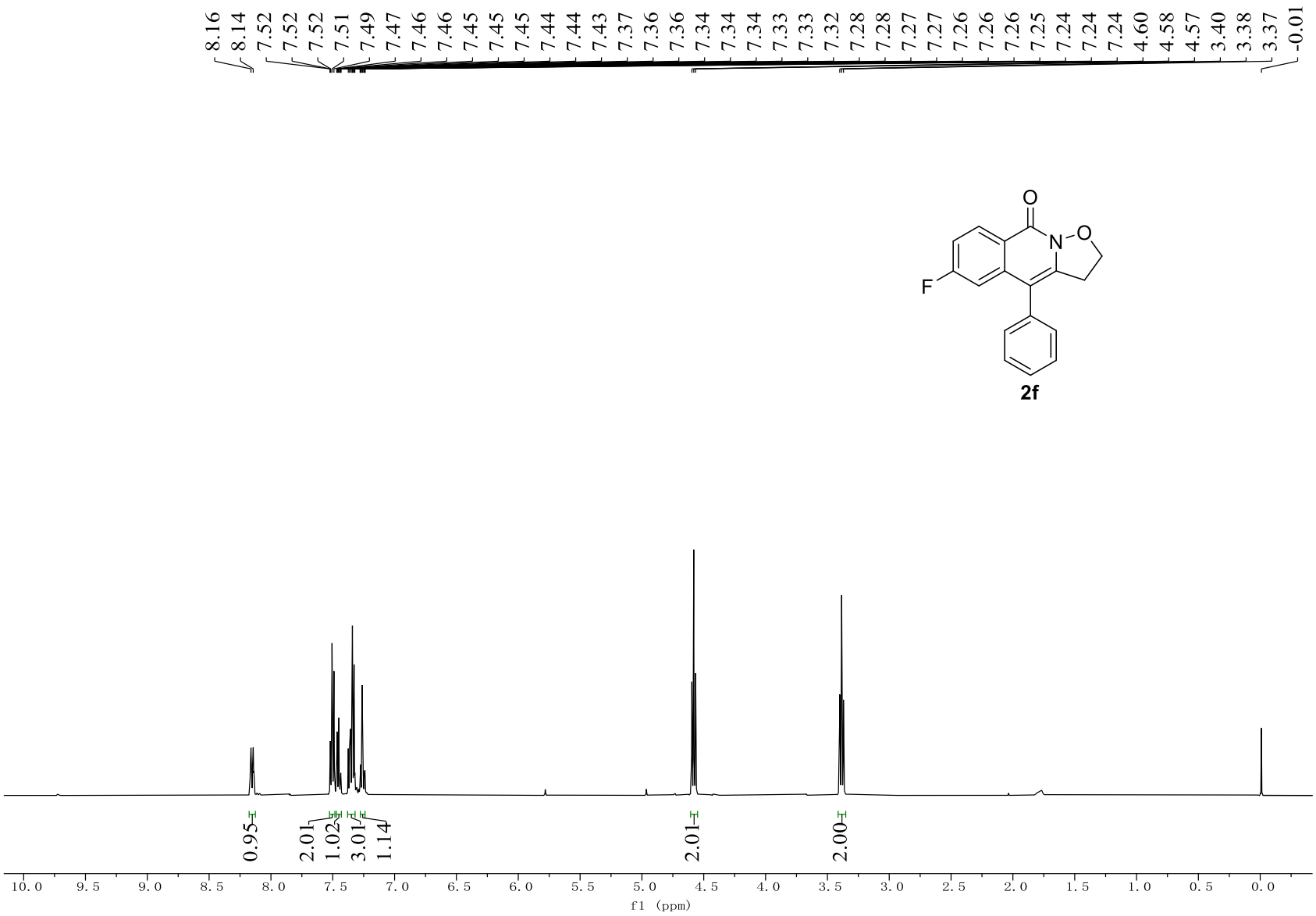

158.33
153.43
138.21
135.12
130.19
130.07
128.81
127.97
127.53
126.28
122.21
113.01
107.13
77.27
77.02
76.76
69.71
55.71
32.13
O
N
O
MeO
2c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

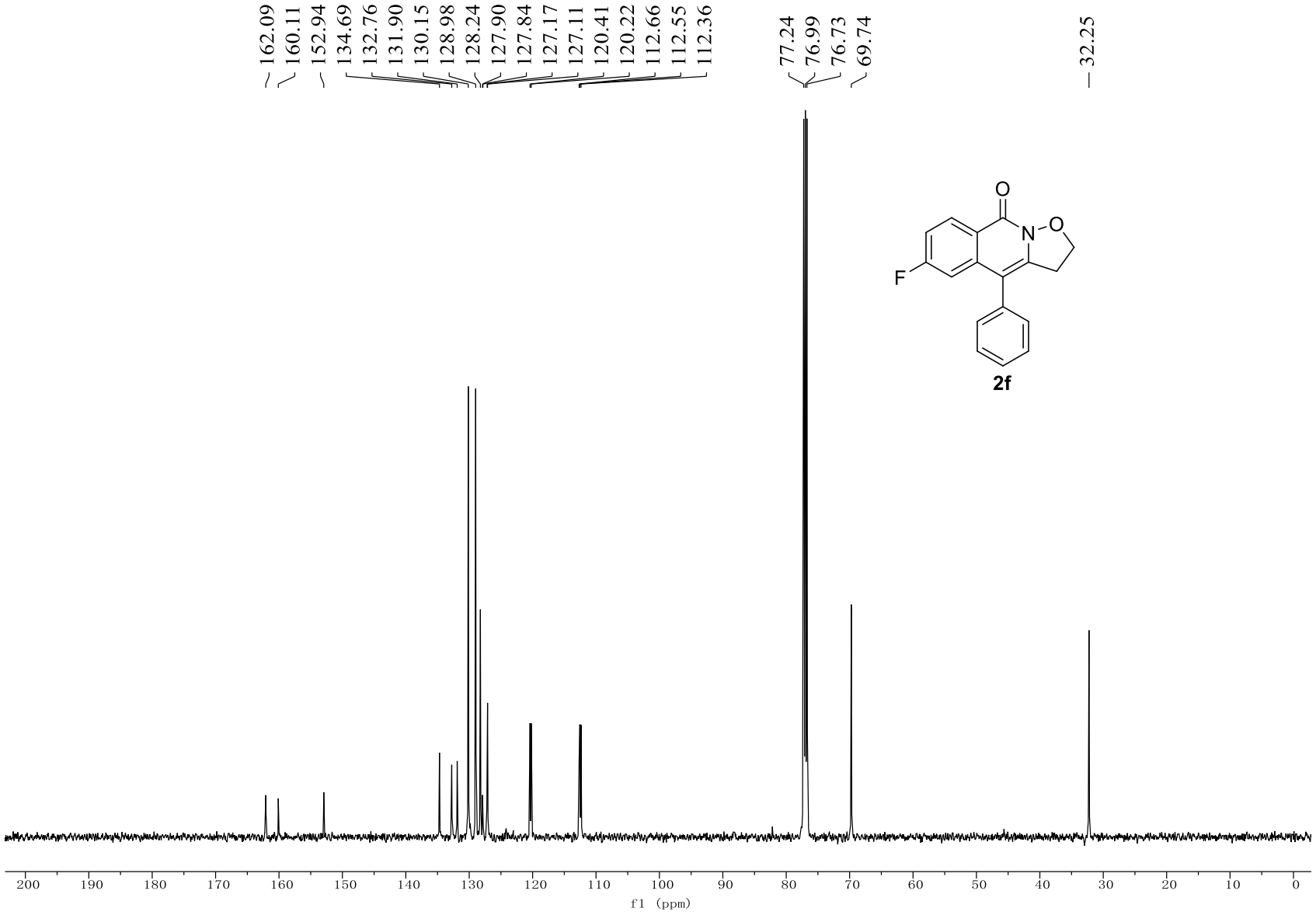

8.3953
8.3913
7.9614
7.5138
7.4988
7.4840
7.4697
7.4568
7.4521
7.4475
7.4423
7.4347
7.4305
7.3546
7.3377
7.3210
7.3042
7.2623
5.3626
4.5834
4.5683
4.5534
3.3961
3.3809
3.3658
3.0913
3.0774
3.0636
3.0498
3.0360
2.1726
1.5949
1.4263
1.3170
1.3032
1.2092
1.1953
0.1168
0.0705
-0.0003
-0.1198
O
N
O
2d
0.93
2.01
1.98
3.01
2.00
2.00
1.02
6.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

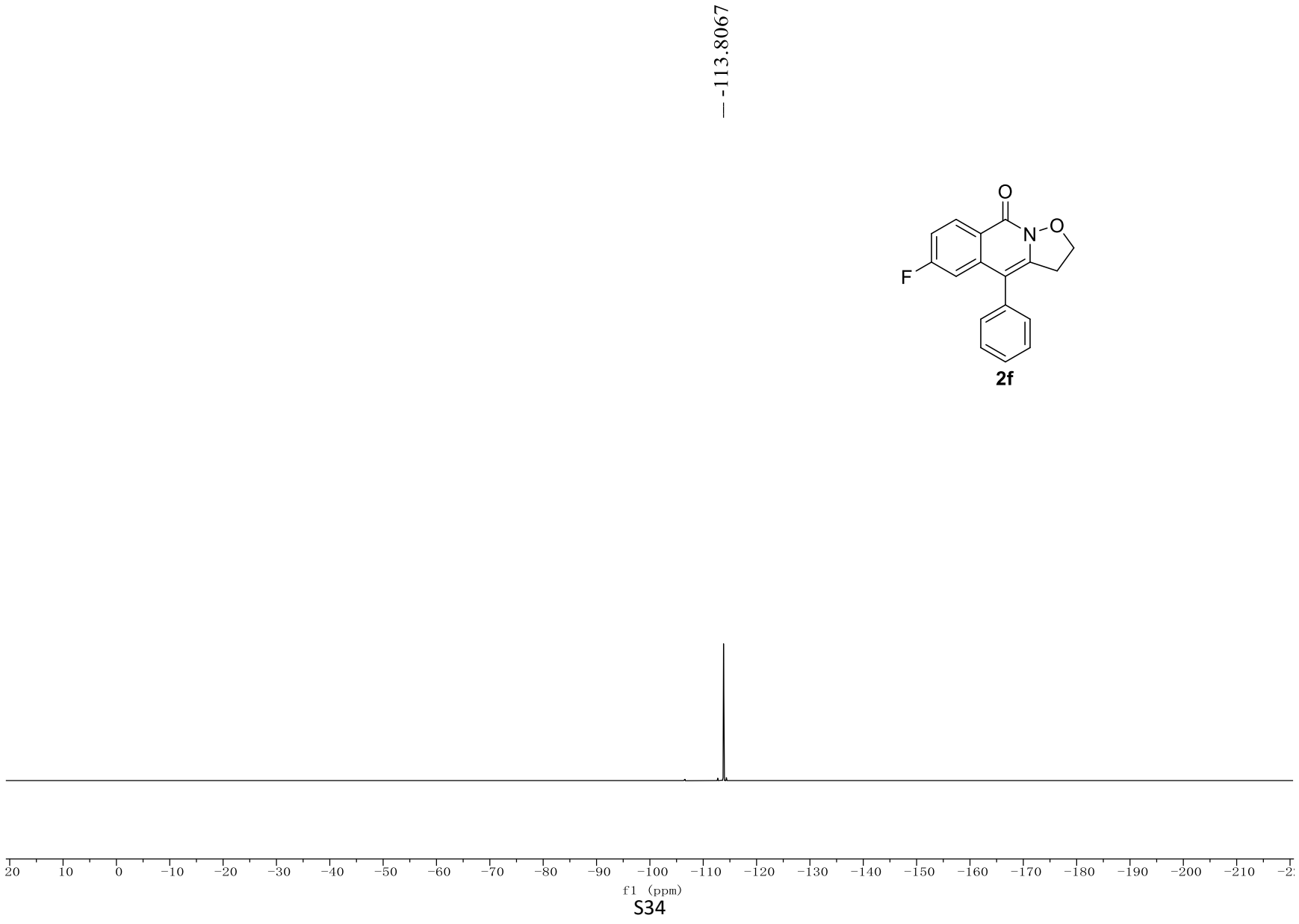

154.05
147.48
135.21
134.28
131.55
130.76
130.27
128.86
128.00
126.32
124.80
124.54
113.02
99.99
77.26
77.00
76.75
69.58
34.06
32.32
23.89
-0.01
O
N
O
2d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.52
8.52
7.62
7.62
7.61
7.60
7.50
7.49
7.48
7.45
7.43
7.35
7.33
7.31
7.26
4.58
4.56
4.55
3.39
3.38
3.36
1.38
-0.01
O
N
O
2e
0.96
1.01
2.05
1.06
3.03
2.00
2.00
9.00
f1 (ppm)

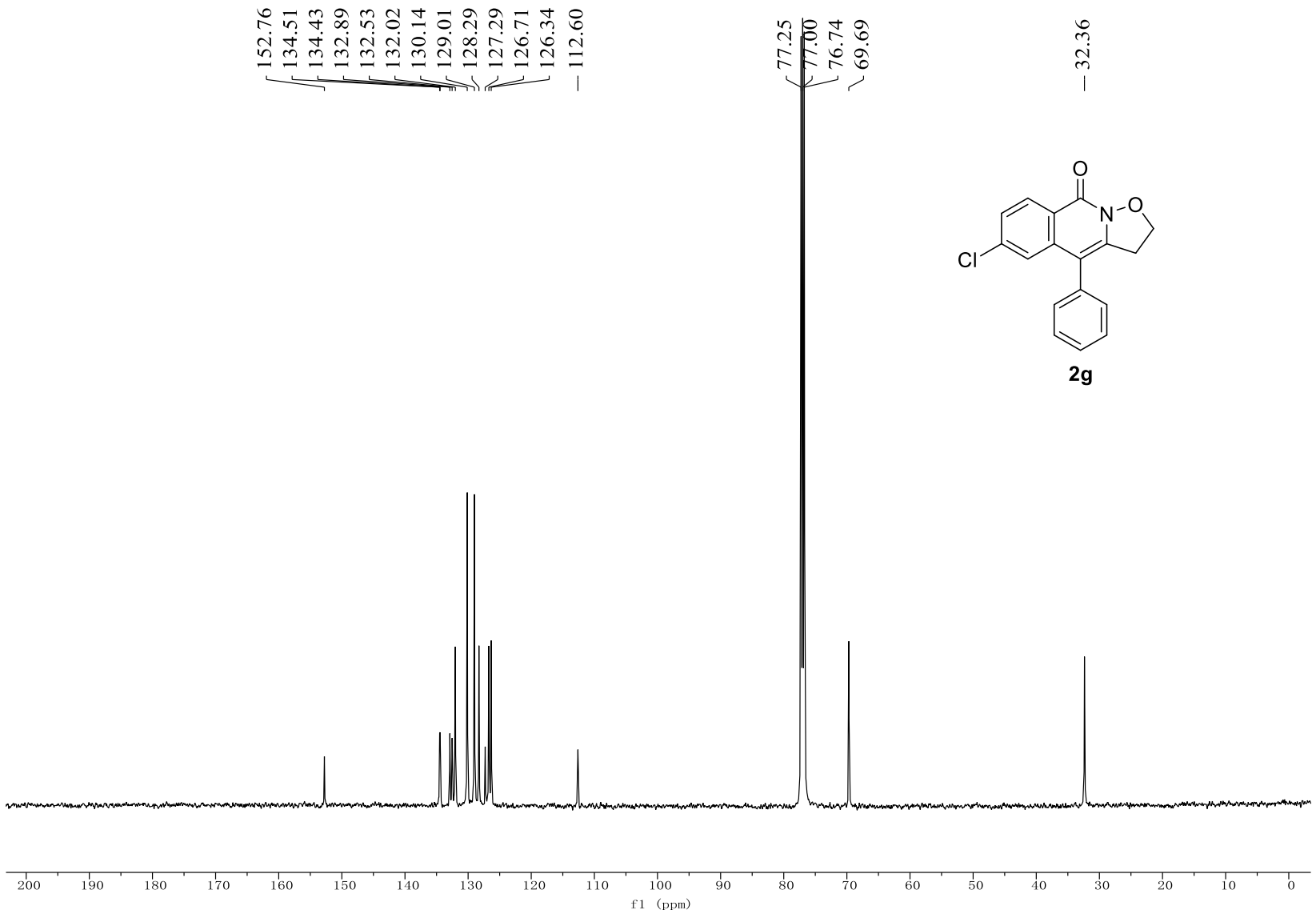

154.0965
149.6923
135.1322
133.8311
131.6306
130.2206
129.6392
128.7962
127.9392
125.9878
124.4885
123.2910
112.5808
77.2206
76.9670
76.7133
69.5202
34.9364
32.2742
31.2356
O
N
O
2e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

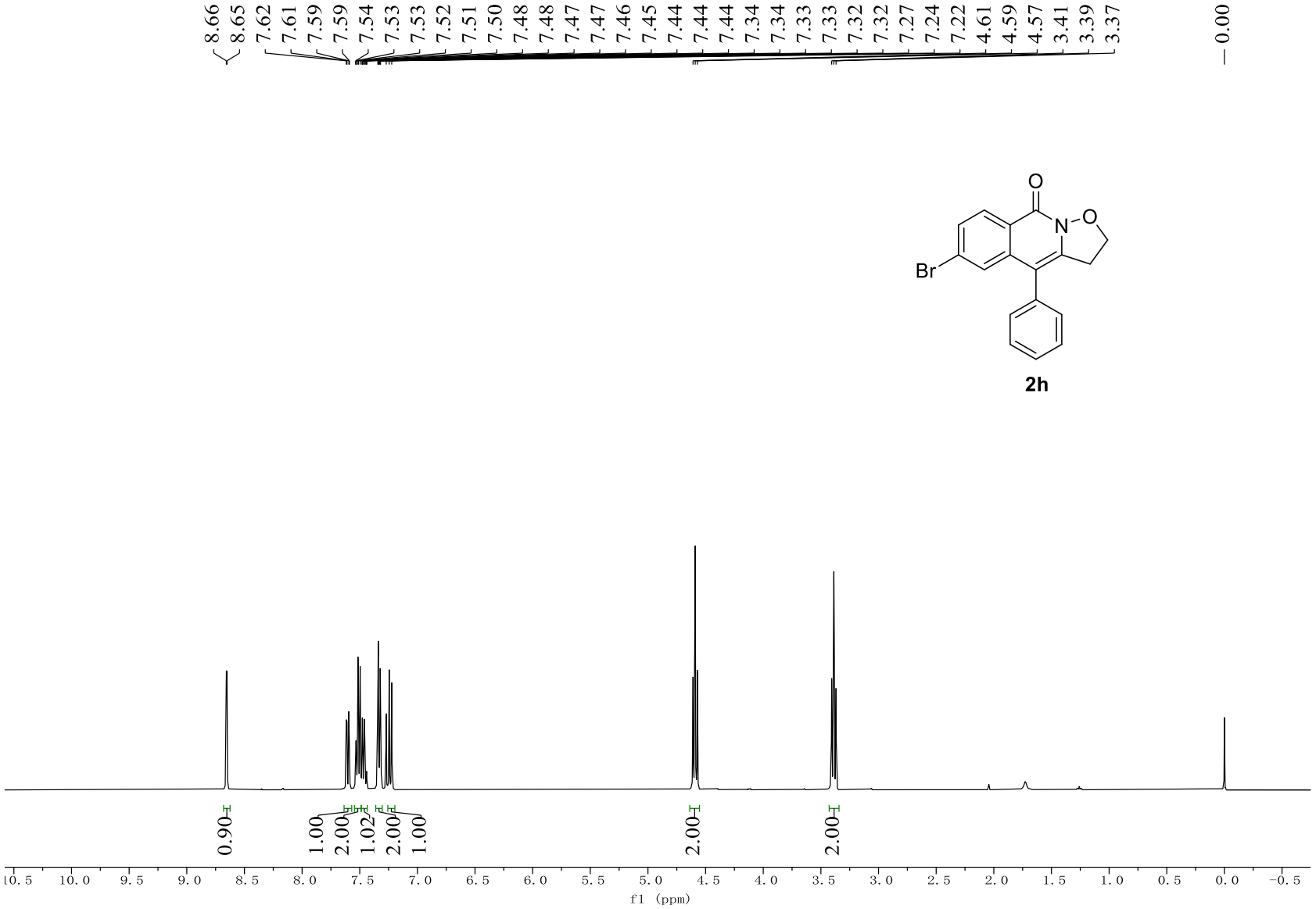

8.16
8.14
7.52
7.52
7.52
7.51
7.49
7.47
7.46
7.46
7.45
7.45
7.45
7.44
7.44
7.43
7.37
7.36
7.36
7.34
7.34
7.34
7.33
7.33
7.32
7.28
7.28
7.27
7.27
7.26
7.26
7.26
7.25
7.24
7.24
7.24
4.60
4.58
4.57
3.40
3.38
3.37
-0.01
O
N
O
F
2f
0.95
2.01
1.02
3.01
1.14
2.01
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

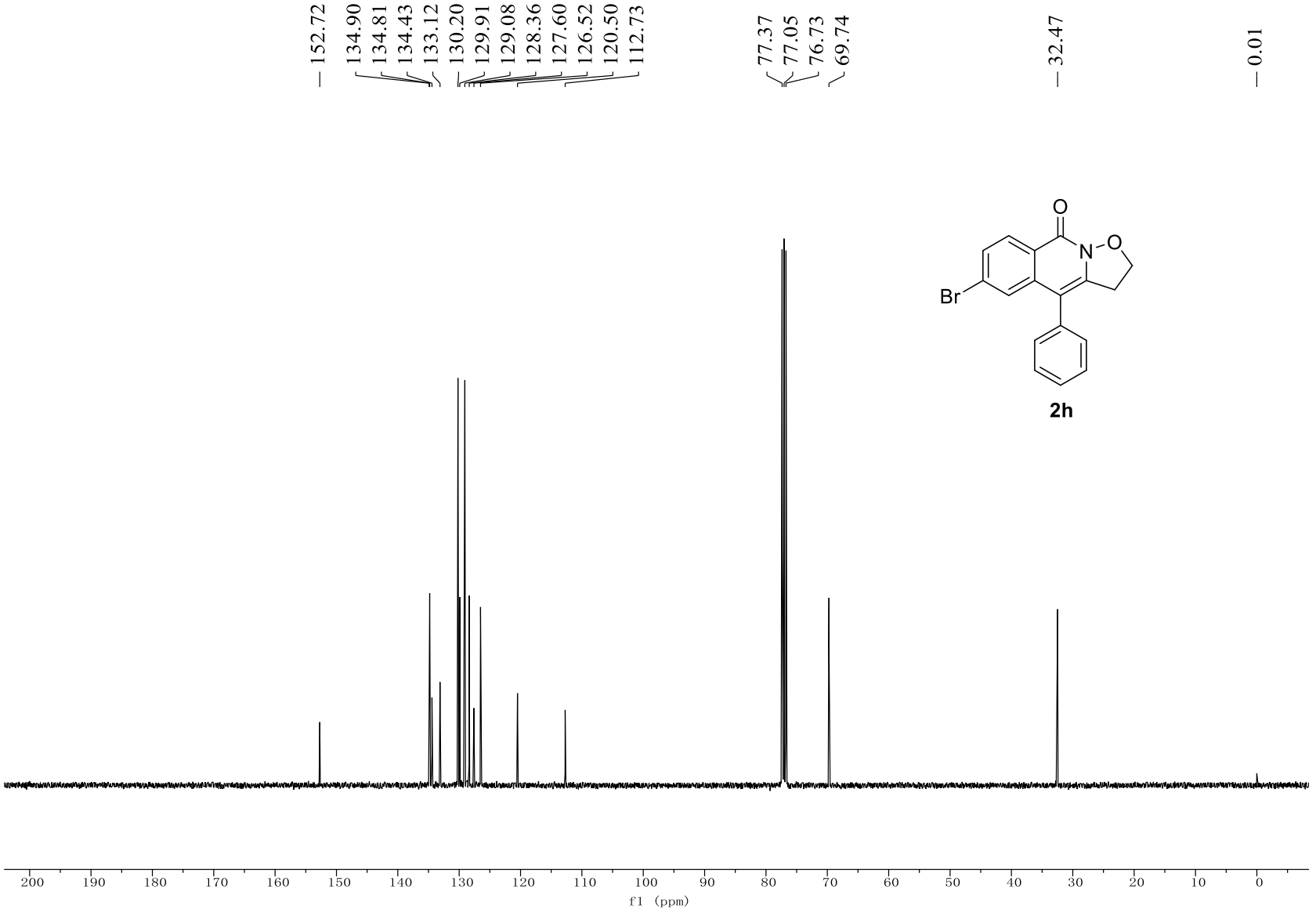

162.09
160.11
152.94
134.69
132.76
131.90
130.15
128.98
128.24
127.90
127.84
127.17
127.11
120.41
120.22
112.66
112.55
112.36
77.24
76.99
76.73
69.74
32.25
O
N
O
F
2f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

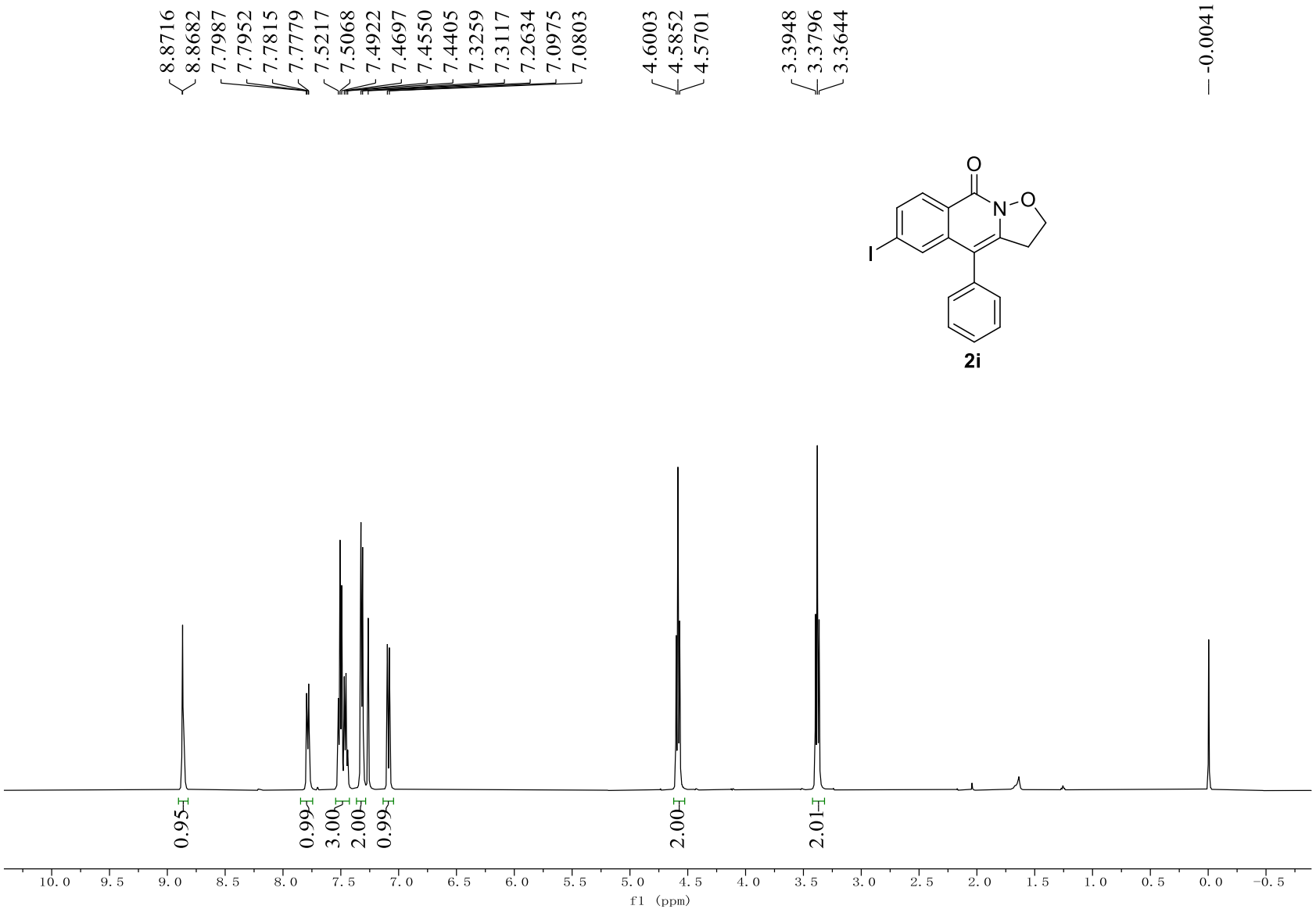

-113.8067
O
N
O
F
2f
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

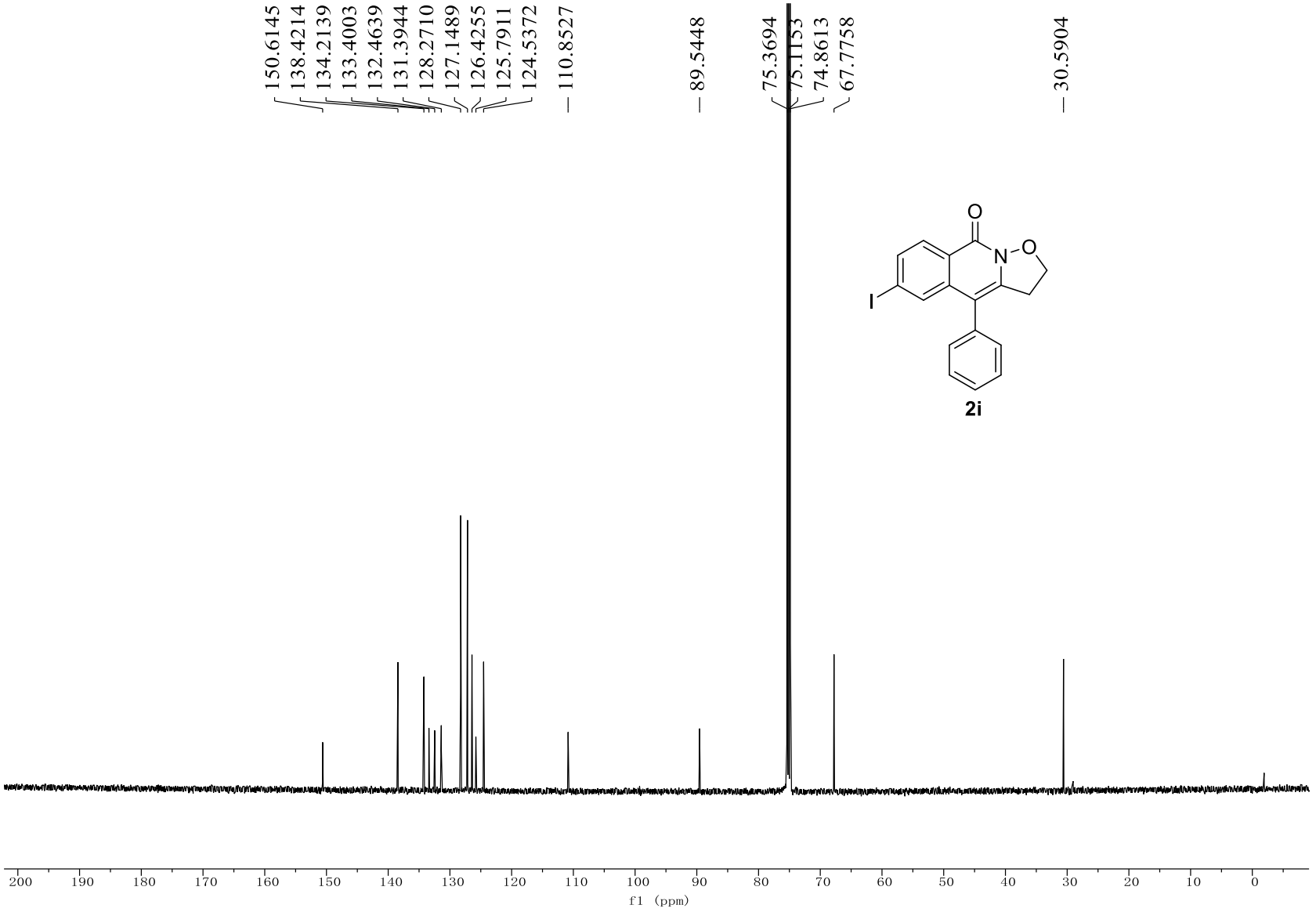

8.48
7.52
7.51
7.49
7.47
7.45
7.45
7.33
7.32
7.31
7.29
7.26
4.60
4.58
4.57
3.41
3.39
3.38
-0.01
O
N
O
Cl
2g
1.00
1.96
2.04
3.00
2.00
2.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

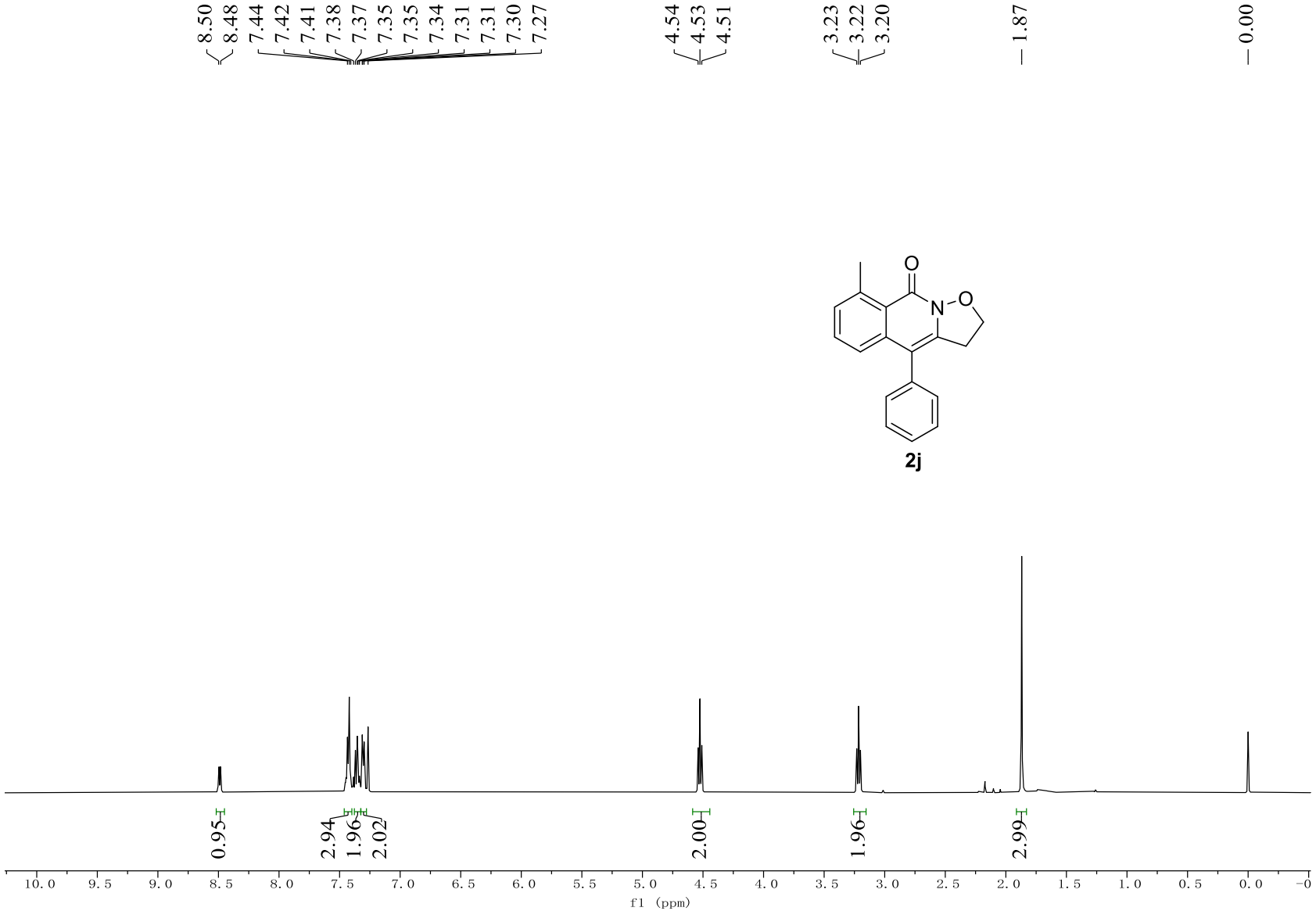

152.76
134.51
134.43
132.89
132.53
132.02
130.14
129.01
128.29
127.29
126.71
126.34
112.60
77.25
77.00
76.74
69.69
32.36
O
N
O
Cl
2g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

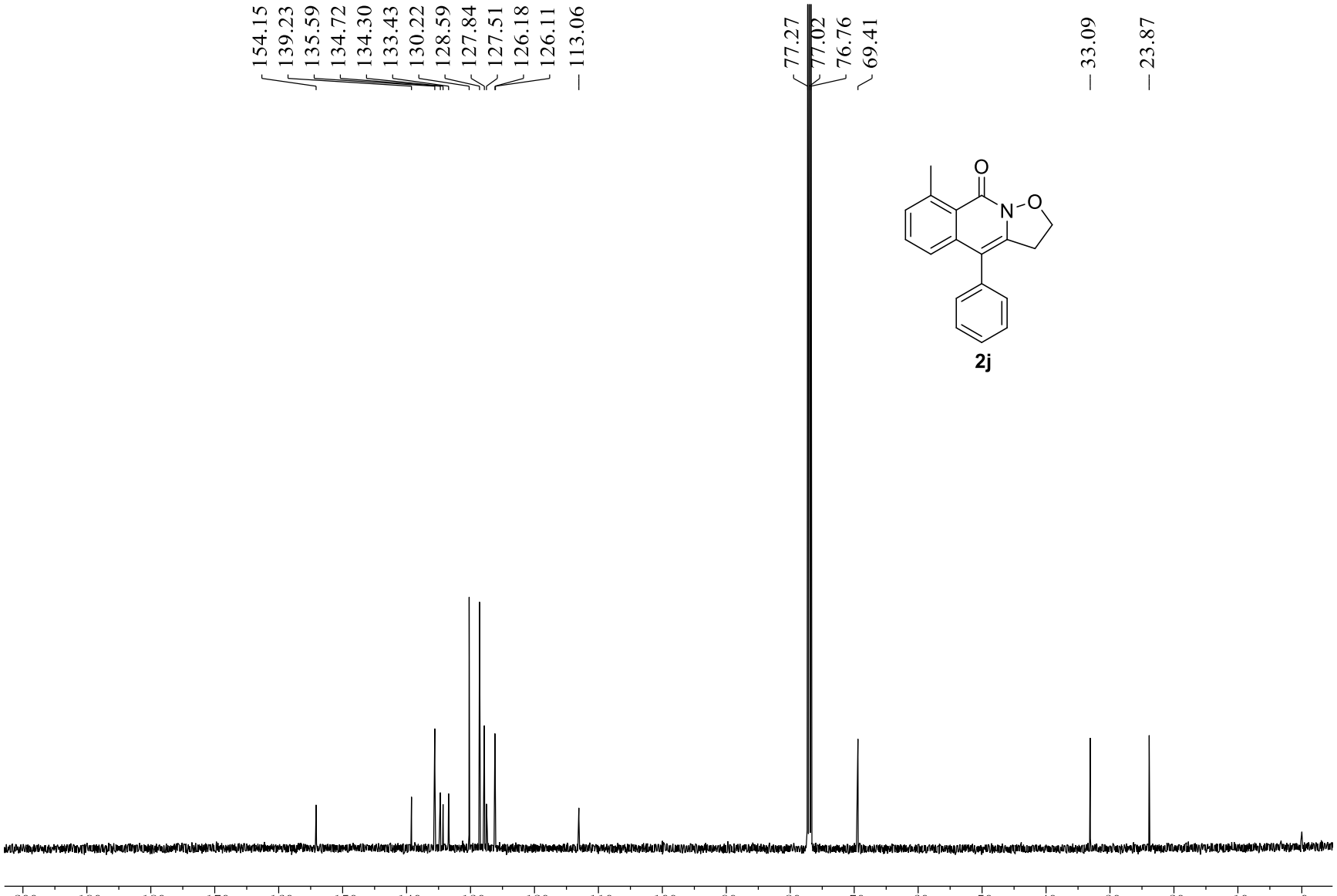

8.66
8.65
7.62
7.61
7.59
7.59
7.54
7.53
7.53
7.52
7.51
7.50
7.48
7.48
7.47
7.47
7.46
7.45
7.44
7.44
7.44
7.34
7.34
7.33
7.33
7.32
7.32
7.27
7.24
7.22
4.61
4.59
4.57
3.41
3.39
3.37
0.00
O
N
O
Br
2h
0.90
1.00
2.00
1.02
2.00
1.00
2.00
2.00
f1 (ppm)

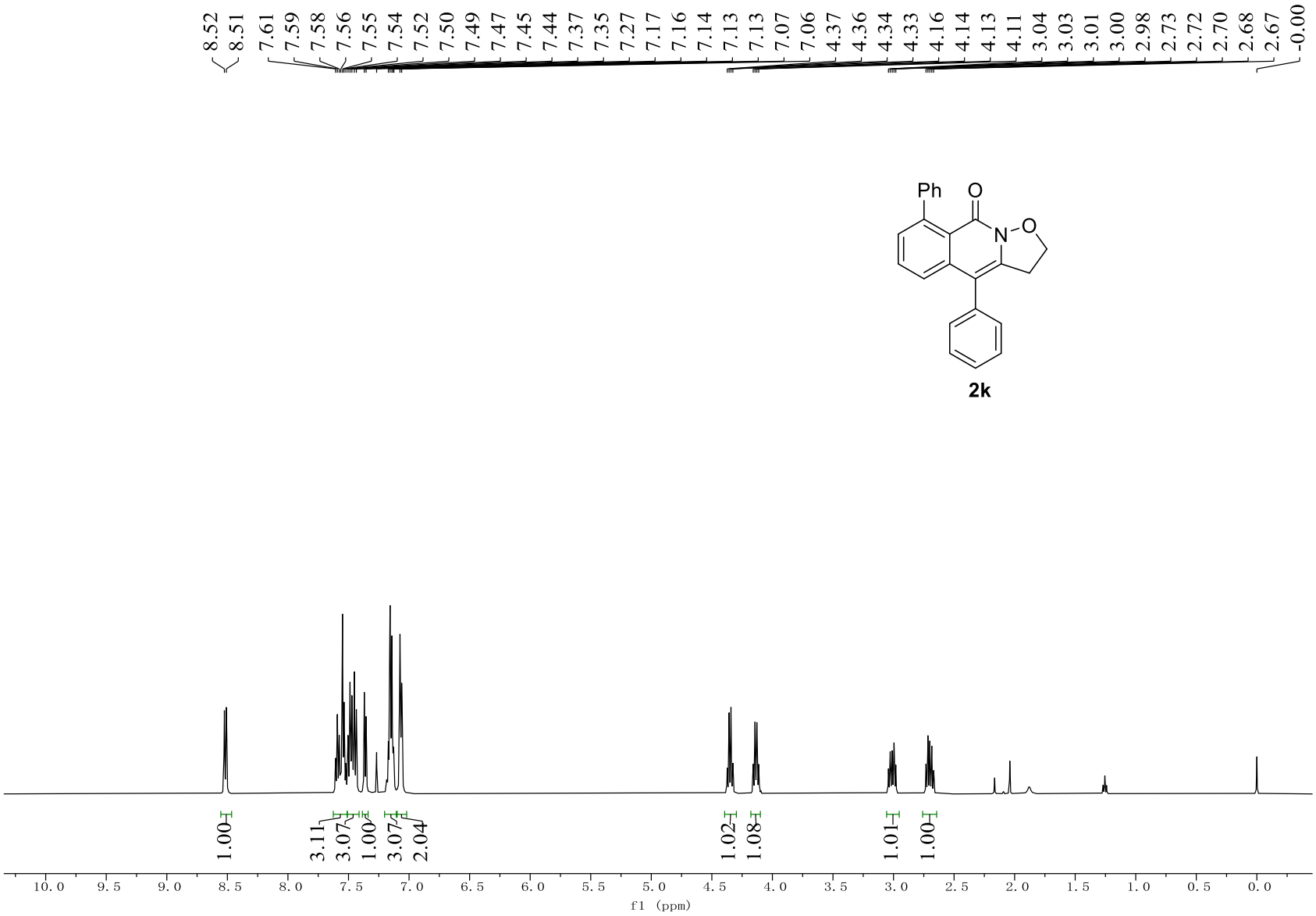

152.72
134.90
134.81
134.43
133.12
130.20
129.91
129.08
128.36
127.60
126.52
120.50
112.73
77.37
77.05
76.73
69.74
32.47
0.01
O
N
O
Br
2h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

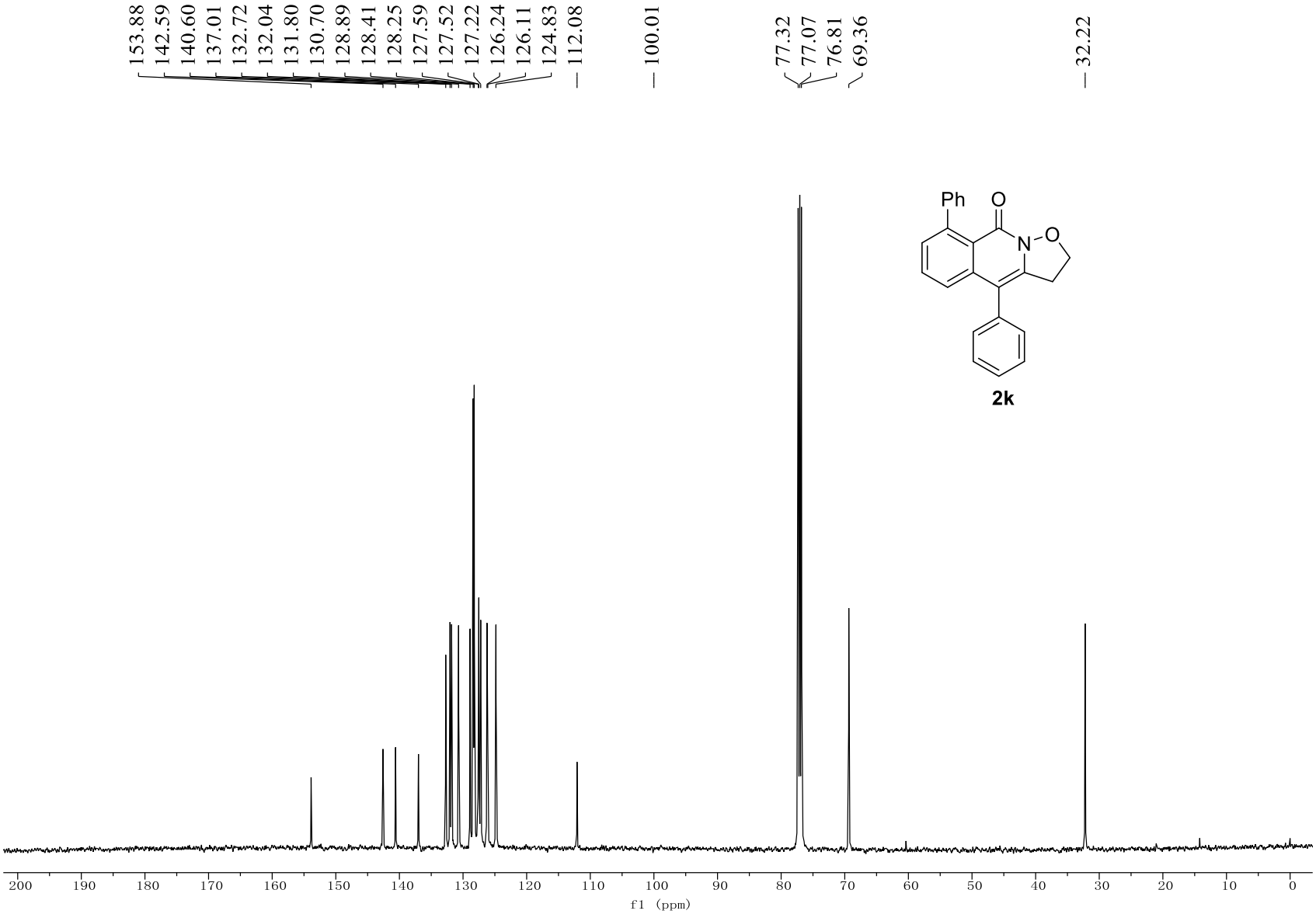

8.8716
8.8682
7.7987
7.7952
7.7815
7.7779
7.5217
7.5068
7.4922
7.4697
7.4550
7.4405
7.3259
7.3117
7.2634
7.0975
7.0803
4.6003
4.5852
4.5701
3.3948
3.3796
3.3644
-0.0041
O
N
O
I
2i
0.95
0.99
3.00
2.00
0.99
2.00
2.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

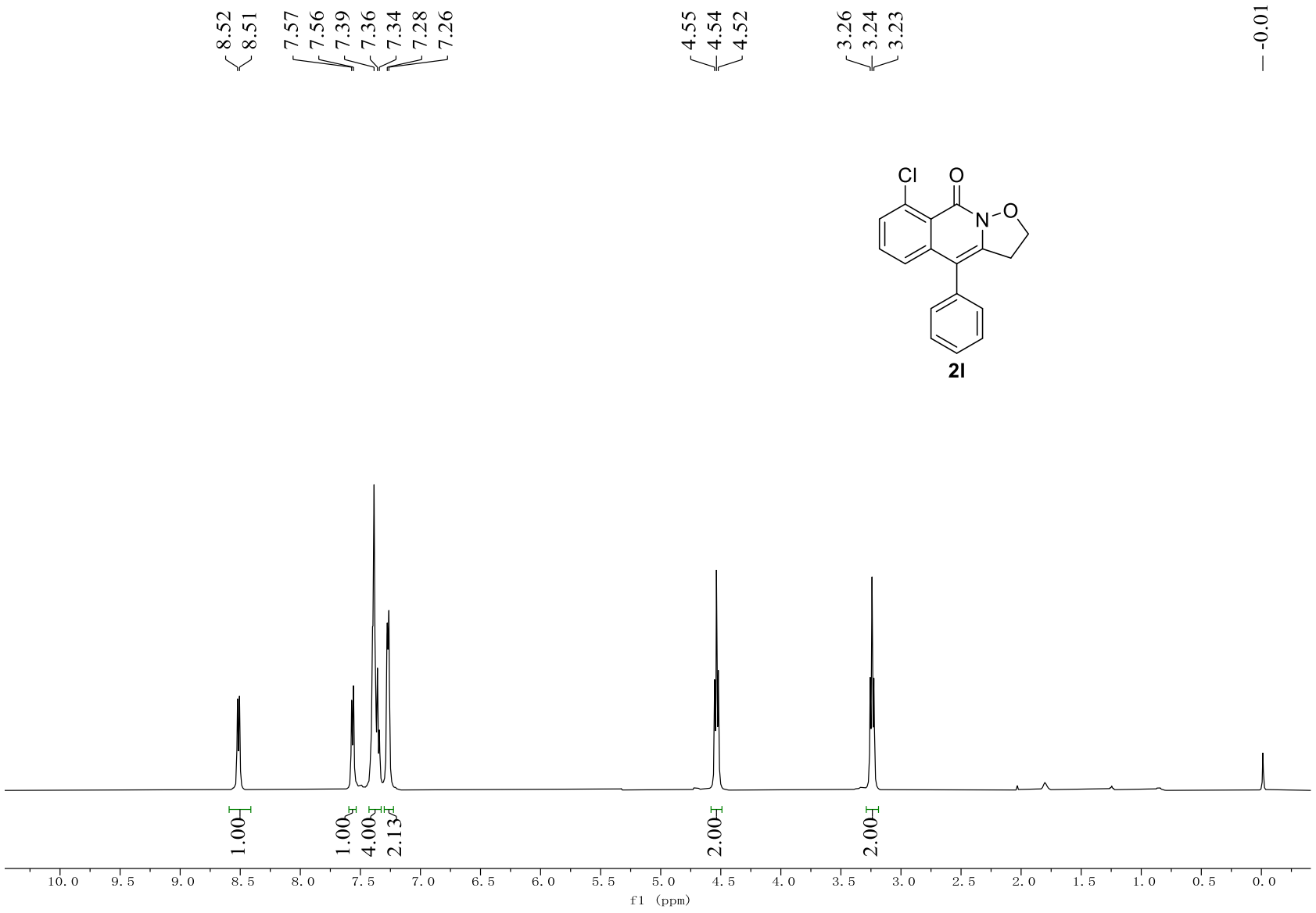

150.6145
138.4214
134.2139
133.4003
132.4639
131.3944
128.2710
127.1489
126.4255
125.7911
124.5372
110.8527
89.5448
75.3694
75.1153
74.8613
67.7758
30.5904
O
N
O
I
2i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

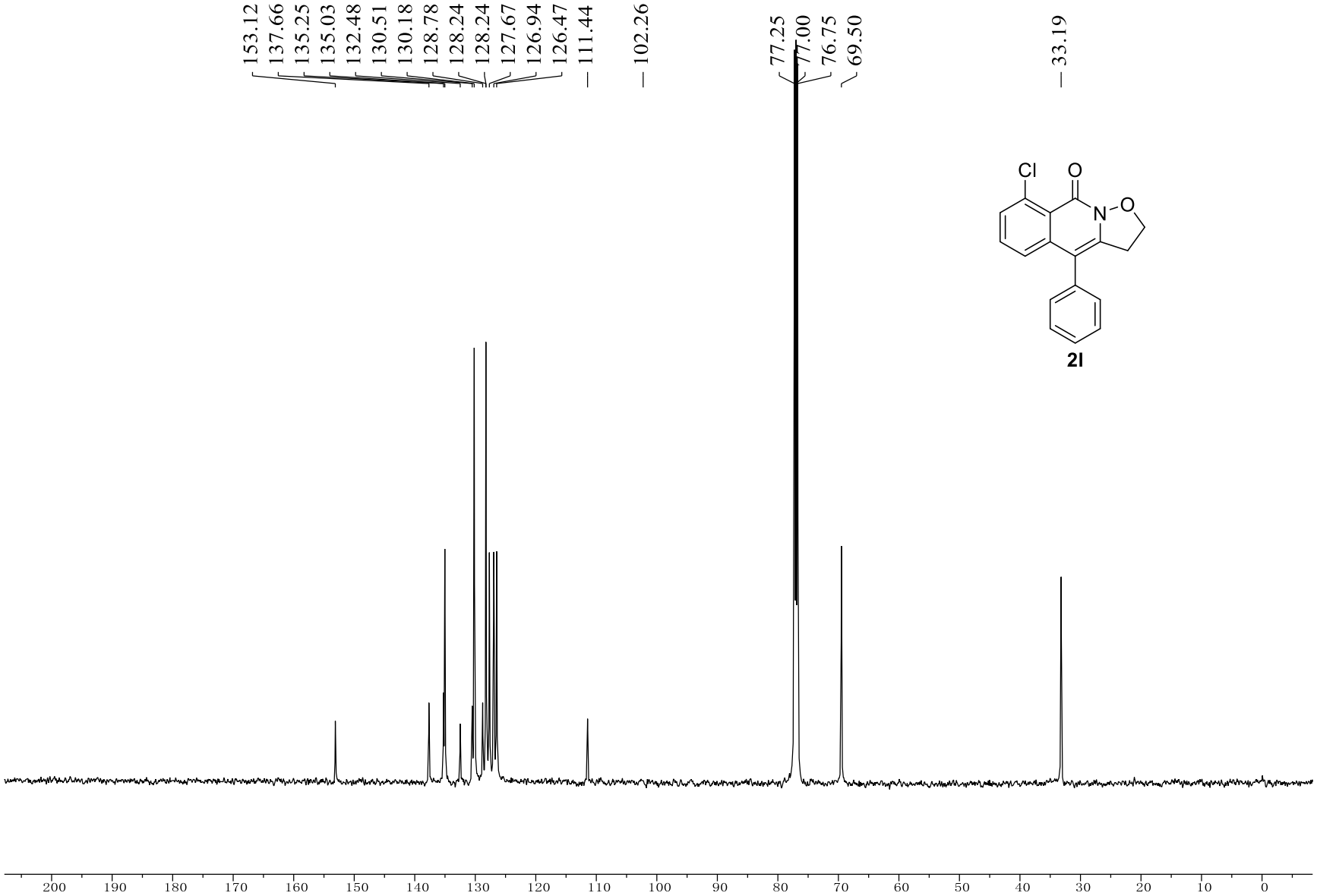

8.50
8.48
7.44
7.42
7.41
7.38
7.37
7.35
7.35
7.34
7.31
7.31
7.30
7.27
4.54
4.53
4.51
3.23
3.22
3.20
1.87
0.00
O
N
O
2j
0.95
2.94
1.96
2.02
2.00
1.96
2.99
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0
f1 (ppm)


154.15
139.23
135.59
134.72
134.30
133.43
130.22
128.59
127.84
127.51
126.18
126.11
113.06
77.27
77.02
76.76
69.41
33.09
23.87
O
N
O
2j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

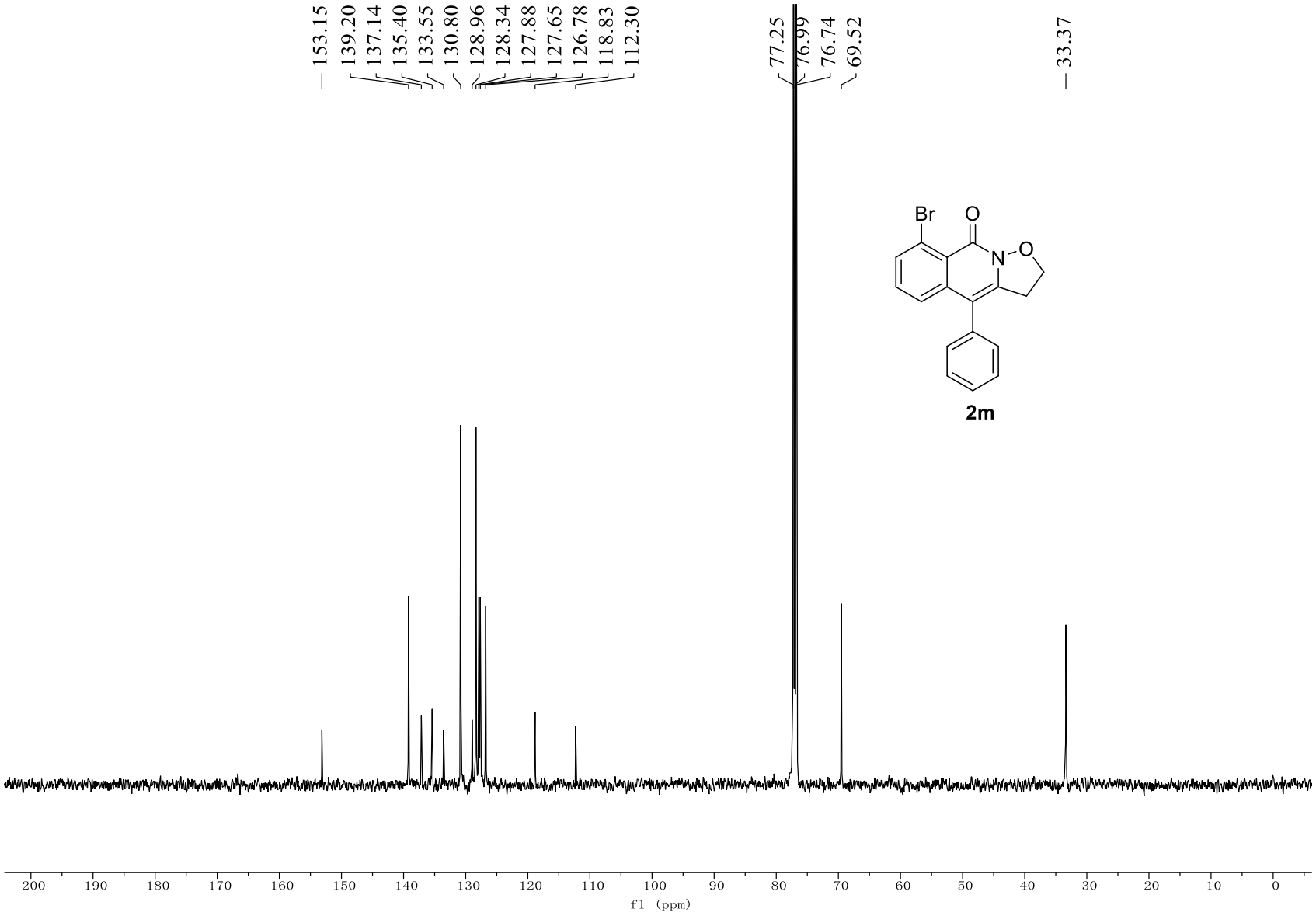

8.52
8.51
7.61
7.59
7.58
7.56
7.55
7.54
7.52
7.50
7.49
7.47
7.45
7.44
7.37
7.35
7.27
7.17
7.16
7.14
7.13
7.13
7.07
7.06
4.37
4.36
4.34
4.33
4.16
4.14
4.13
4.11
3.04
3.03
3.01
3.00
2.98
2.73
2.72
2.70
2.68
2.67
-0.00
Ph
O
N
O
2k
1.00
3.11
3.07
1.00
3.07
2.04
1.02
1.08
1.01
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

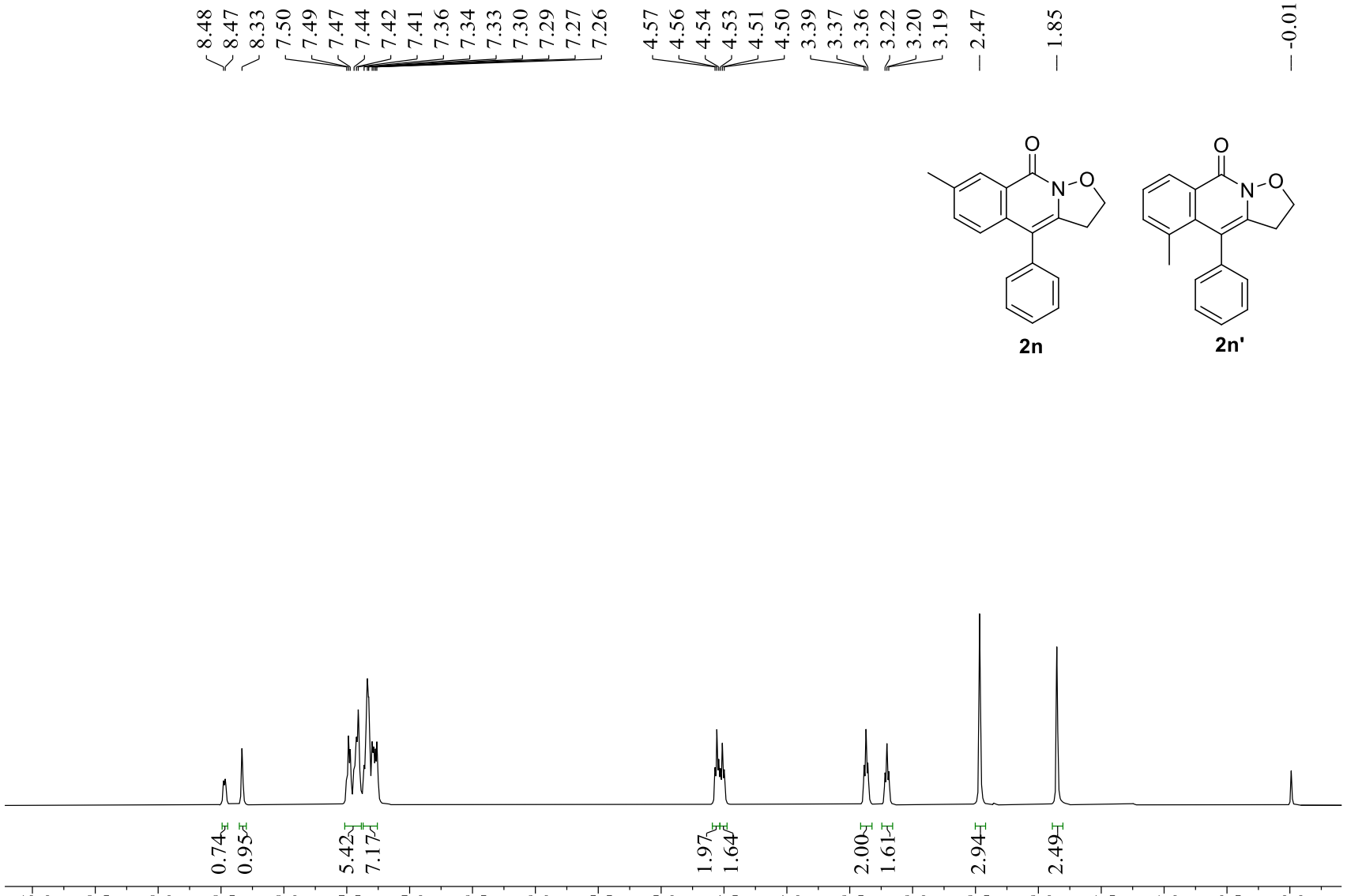

153.88
142.59
140.60
137.01
132.72
132.04
131.80
130.70
128.89
128.41
128.25
127.59
127.52
127.22
126.24
126.11
124.83
112.08
100.01
77.32
77.07
76.81
69.36
32.22
Ph
O
N
O
2k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

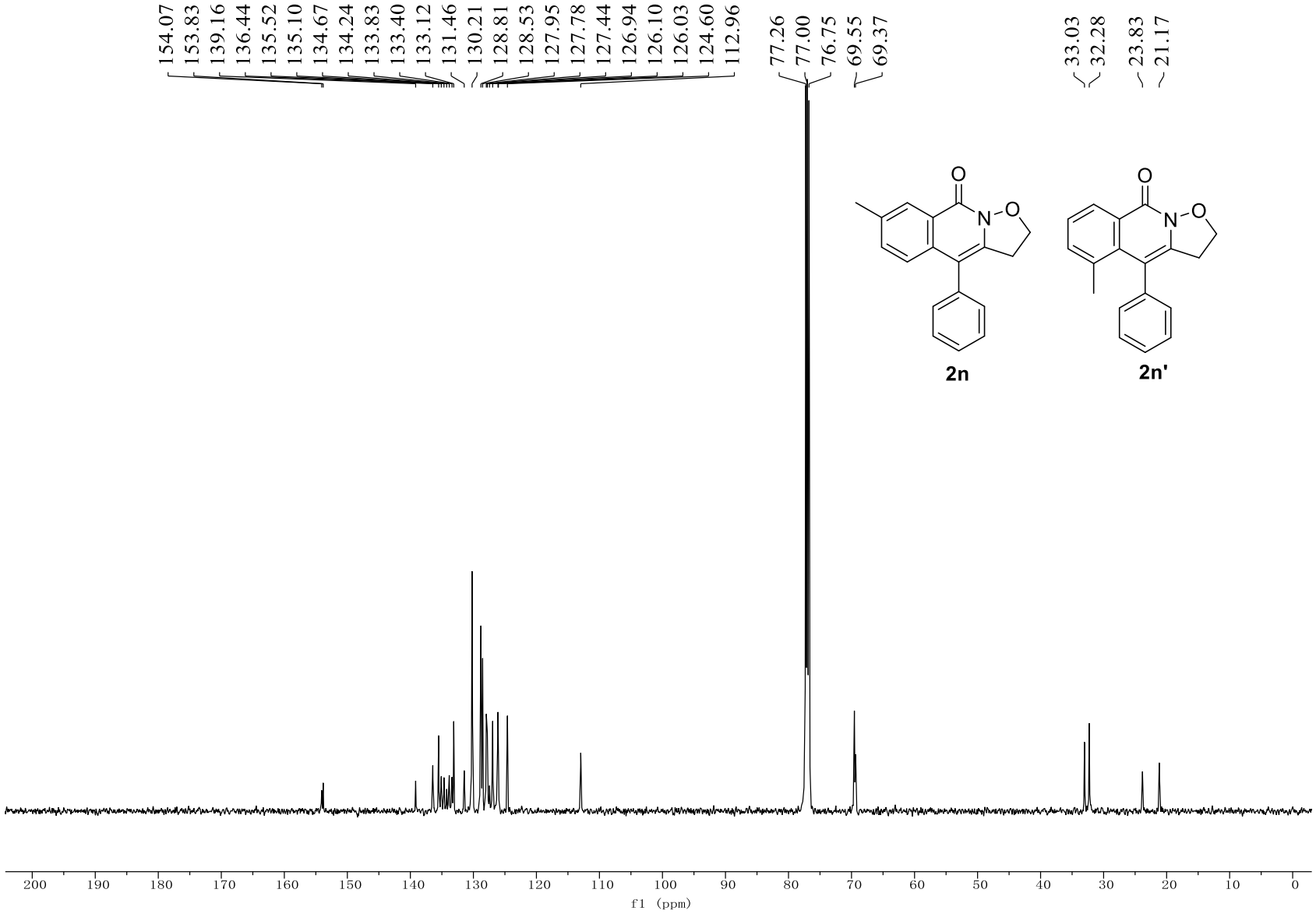

8.52
8.51
7.57
7.56
7.39
7.36
7.34
7.28
7.26
4.55
4.54
4.52
3.26
3.24
3.23
-0.01
Cl
O
N
O
2l
1.00
1.00
4.00
2.13
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

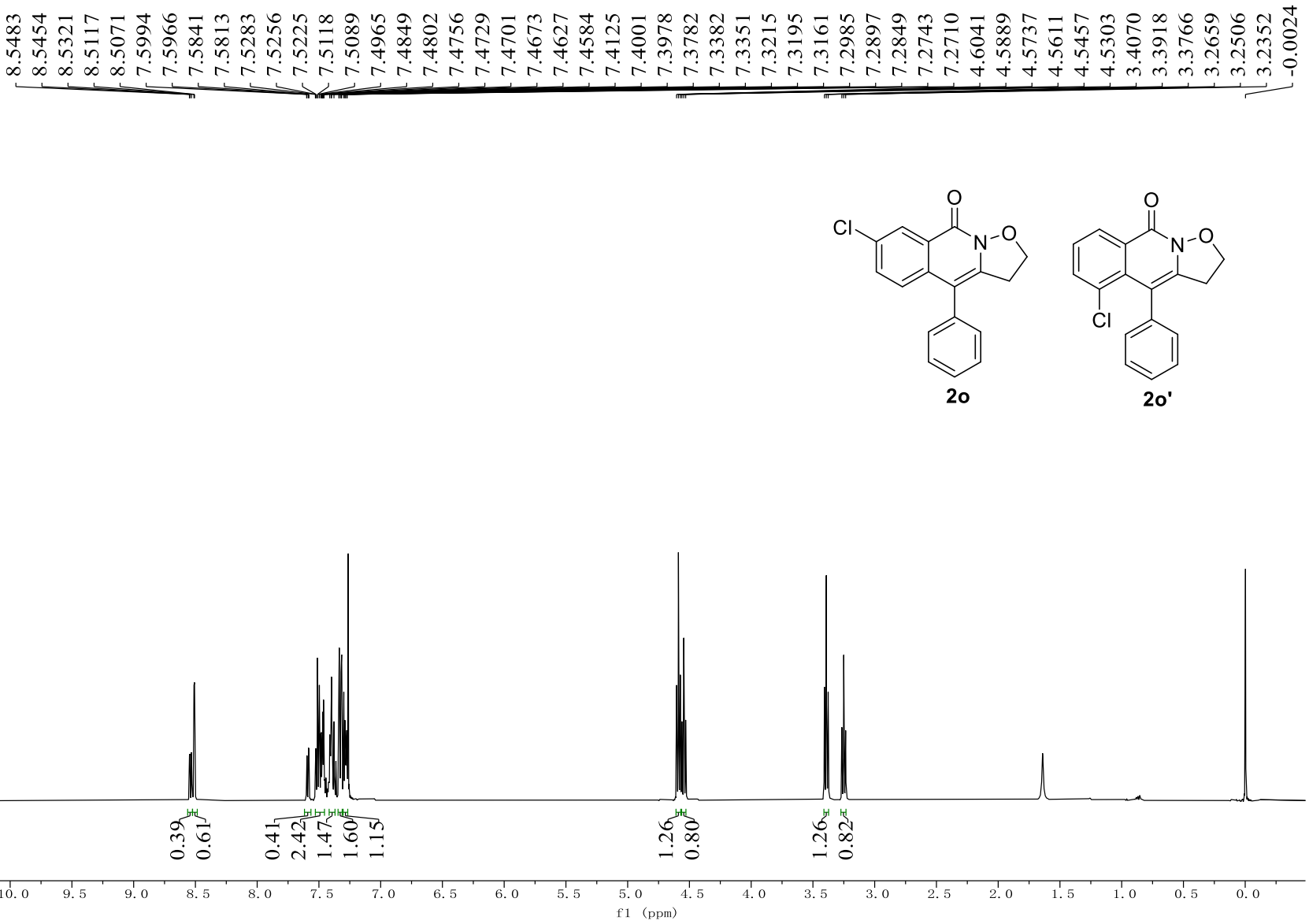

153.12
137.66
135.25
135.03
132.48
130.51
130.18
128.78
128.24
128.24
127.67
126.94
126.47
111.44
102.26
77.25
77.00
76.75
69.50
33.19
Cl
O
N
O
2l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

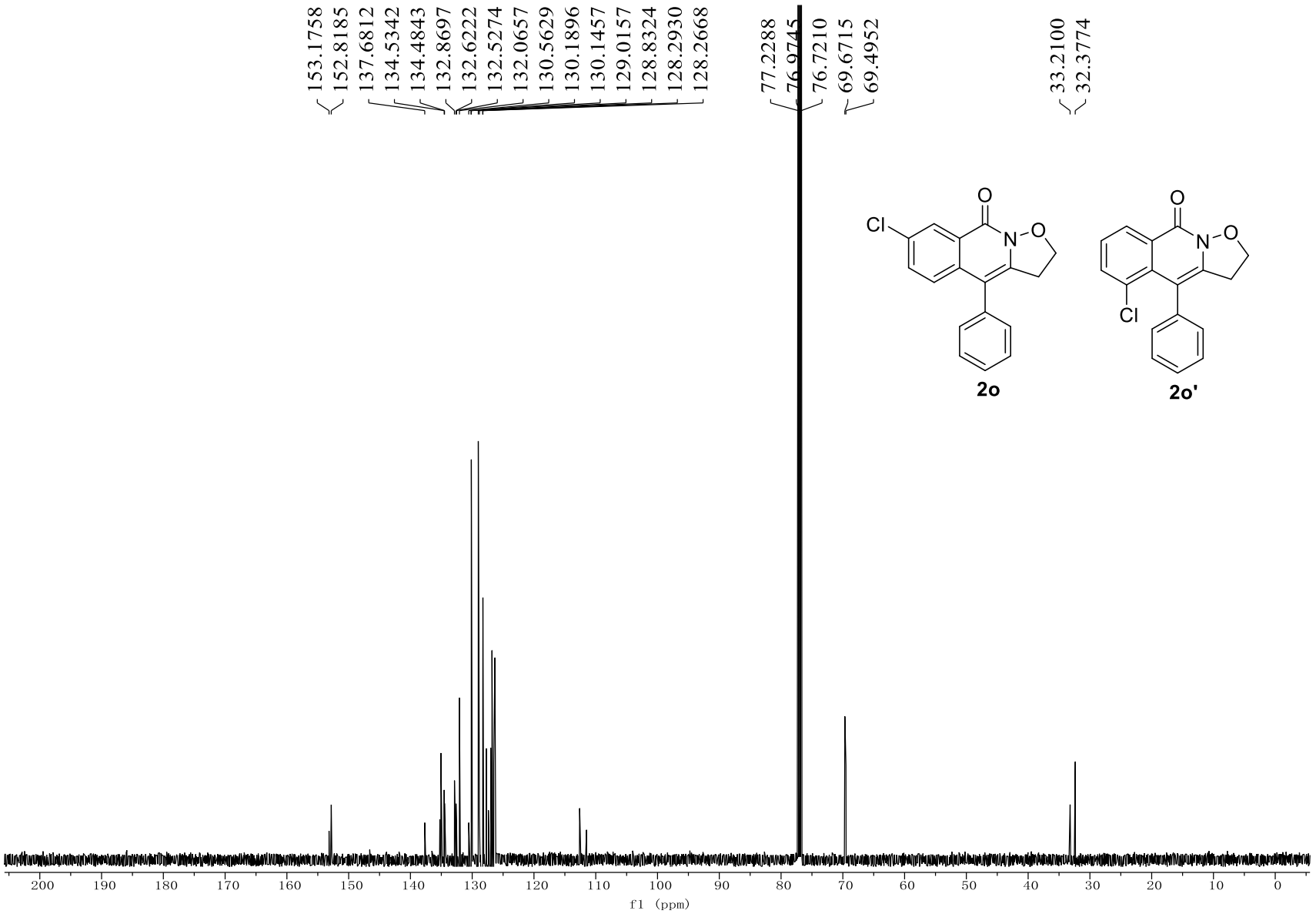

8.58
8.56
7.82
7.82
7.81
7.80
7.40
7.39
7.39
7.38
7.28
7.26
7.25
4.54
4.53
4.51
3.25
3.23
3.22
-0.02
Br
O
N
O
2m
1.00
1.00
3.00
3.21
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

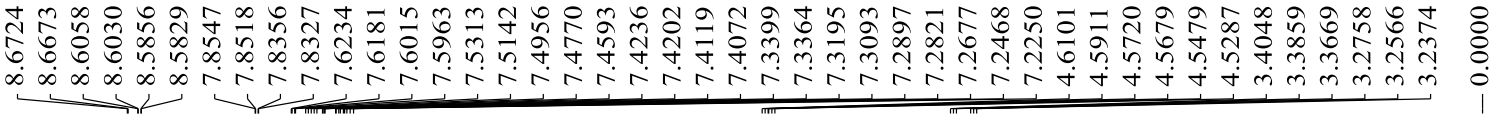

153.15
139.20
137.14
135.40
133.55
130.80
128.96
128.34
127.88
127.65
126.78
118.83
112.30
77.25
76.99
76.74
69.52
33.37
Br
O
N
O
2m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

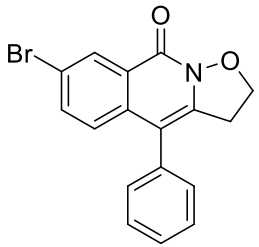

8.48
8.47
8.33
7.50
7.49
7.47
7.44
7.42
7.41
7.36
7.34
7.33
7.30
7.29
7.27
7.26
4.57
4.56
4.54
4.53
4.51
4.50
3.39
3.37
3.36
3.22
3.20
3.19
2.47
1.85
-0.01
O
N
O
2n
2n'
0.74
0.95
5.42
7.17
1.97
1.64
2.00
1.61
2.94
2.49
f1 (ppm)


154.07
153.83
139.16
136.44
135.52
135.10
134.67
134.24
133.83
133.40
133.12
131.46
130.21
128.81
128.53
127.95
127.78
127.44
126.94
126.10
126.03
124.60
112.96
77.26
77.00
76.75
69.55
69.37
33.03
32.28
23.83
21.17
O
N
O
2n
2n'
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

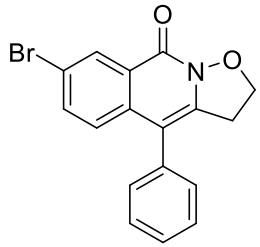

8.5483
8.5454
8.5321
8.5117
8.5071
7.5994
7.5966
7.5841
7.5813
7.5283
7.5256
7.5225
7.5118
7.5089
7.4965
7.4849
7.4802
7.4756
7.4729
7.4701
7.4673
7.4627
7.4584
7.4125
7.4001
7.3978
7.3782
7.3382
7.3351
7.3215
7.3195
7.3161
7.2985
7.2897
7.2849
7.2743
7.2710
4.6041
4.5889
4.5737
4.5611
4.5457
4.5303
3.4070
3.3918
3.3766
3.2659
3.2506
3.2352
-0.0024
Cl
O
N
O
2o
Cl
O
N
O
2o'
0.39
0.61
0.41
2.42
1.47
1.60
1.15
1.26
0.80
1.26
0.82
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.1758
152.8185
137.6812
134.5342
134.4843
132.8697
132.6222
132.5274
132.0657
130.5629
130.1896
130.1457
129.0157
128.8324
128.2930
128.2668
77.2288
76.9745
76.7210
69.6715
69.4952
33.2100
32.3774
Cl
O
N
O
2o
Cl
O
N
O
2o'
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

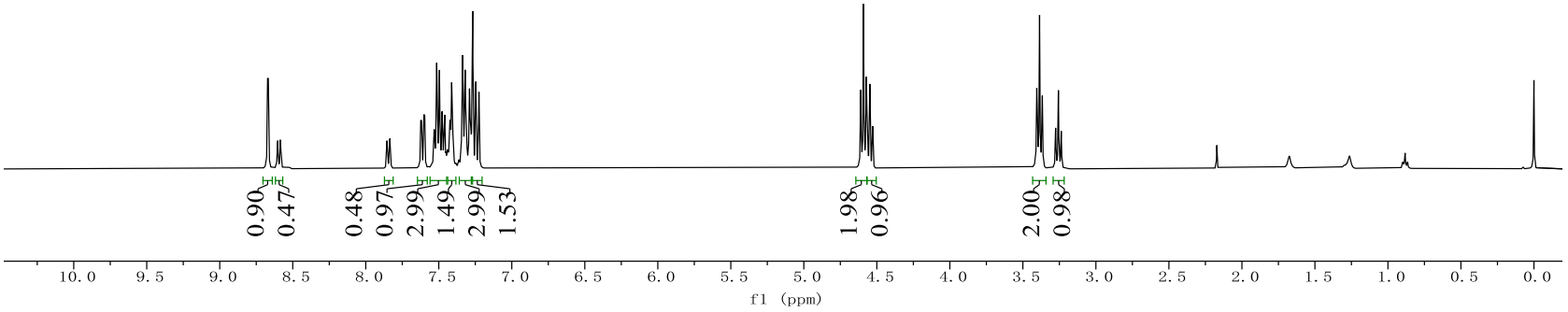

8.6724
8.6673
8.6058
8.6030
8.5856
8.5829
7.8547
7.8518
7.8356
7.8327
7.6234
7.6181
7.6015
7.5963
7.5313
7.5142
7.4956
7.4770
7.4593
7.4236
7.4202
7.4119
7.4072
7.3399
7.3364
7.3195
7.3093
7.2897
7.2821
7.2677
7.2468
7.2250
4.6101
4.5911
4.5720
4.5679
4.5479
4.5287
4.4048
3.3859
3.3669
3.2758
3.2566
3.2374
0.0000

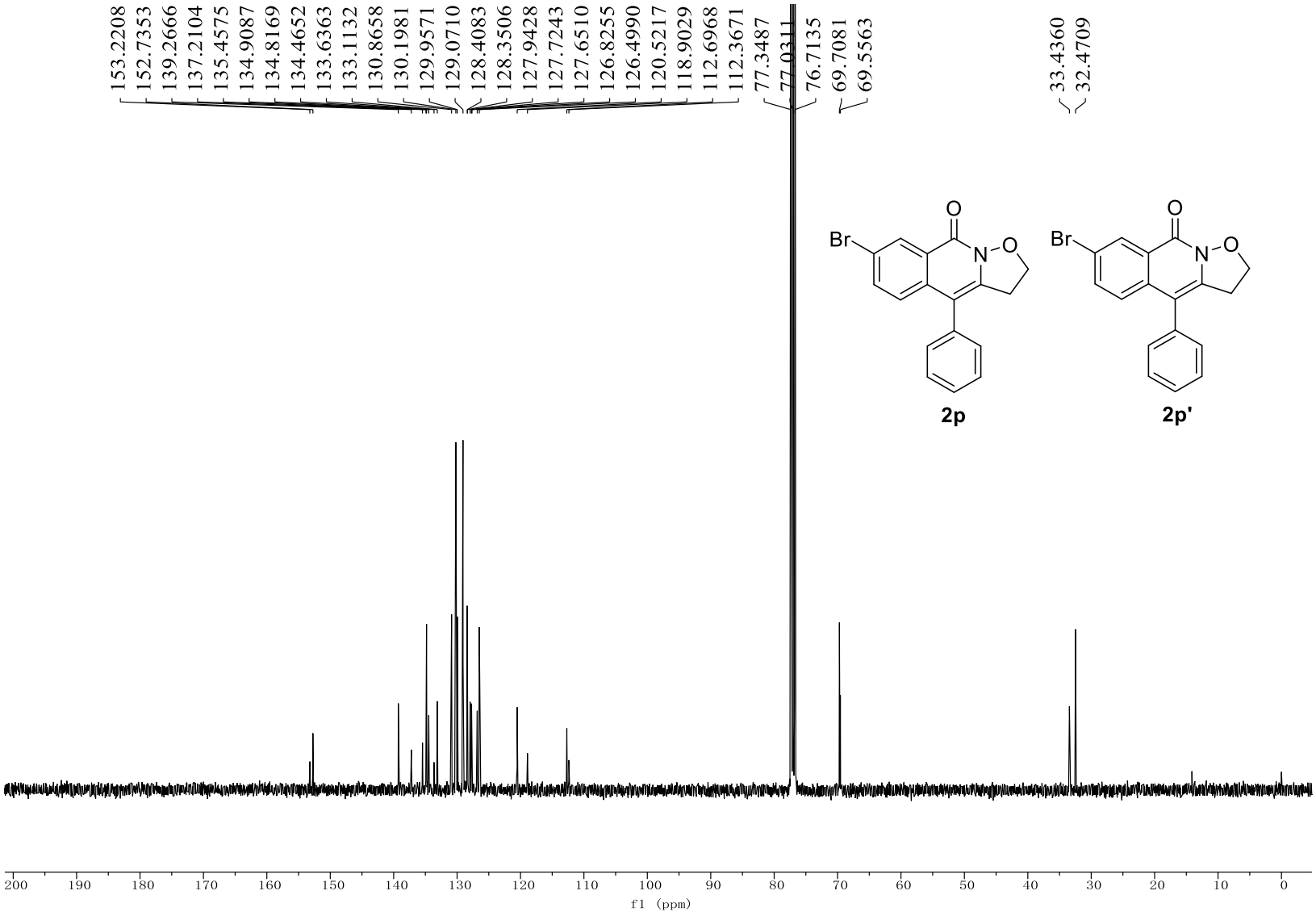

Br
O
N
O

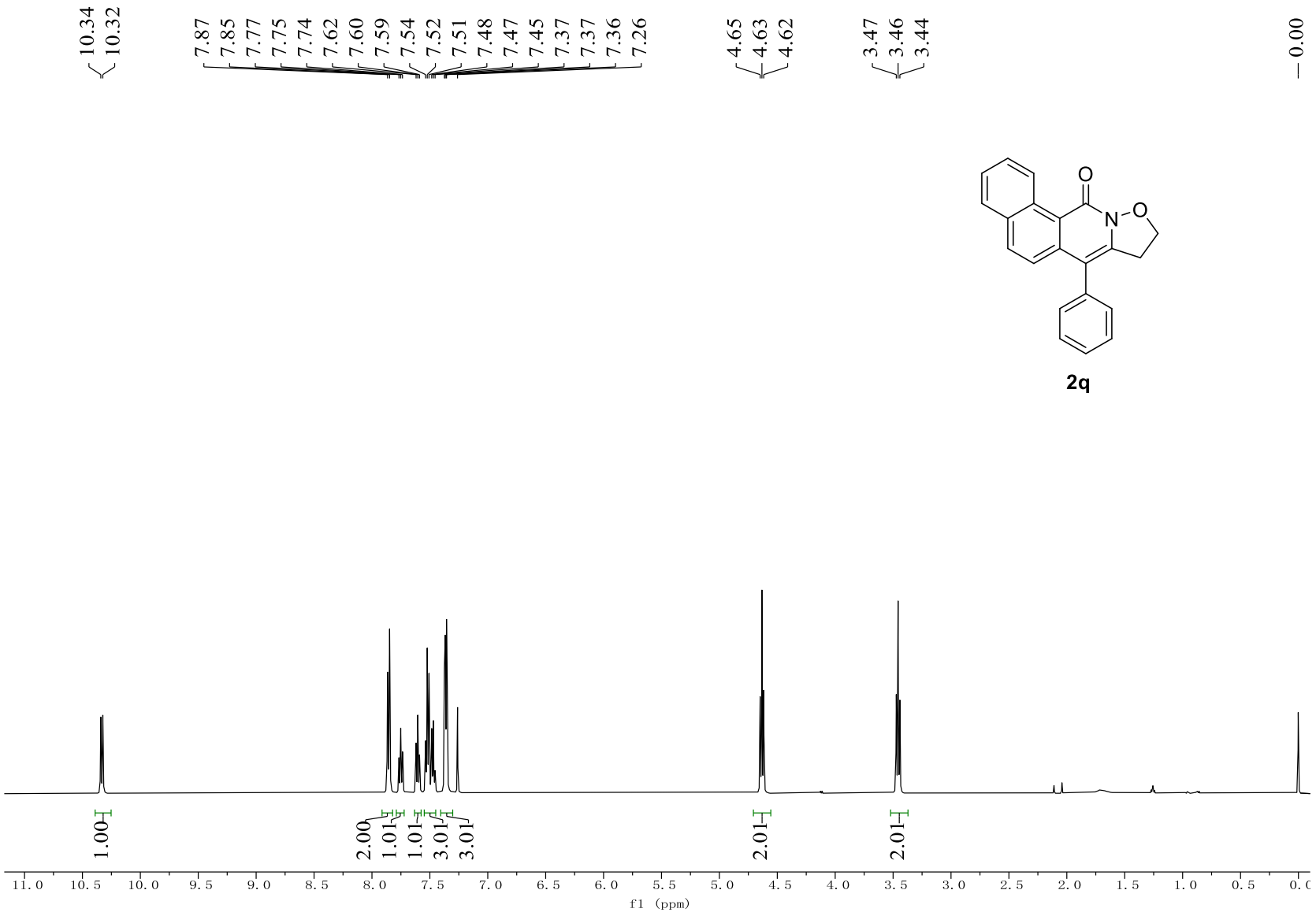
2p

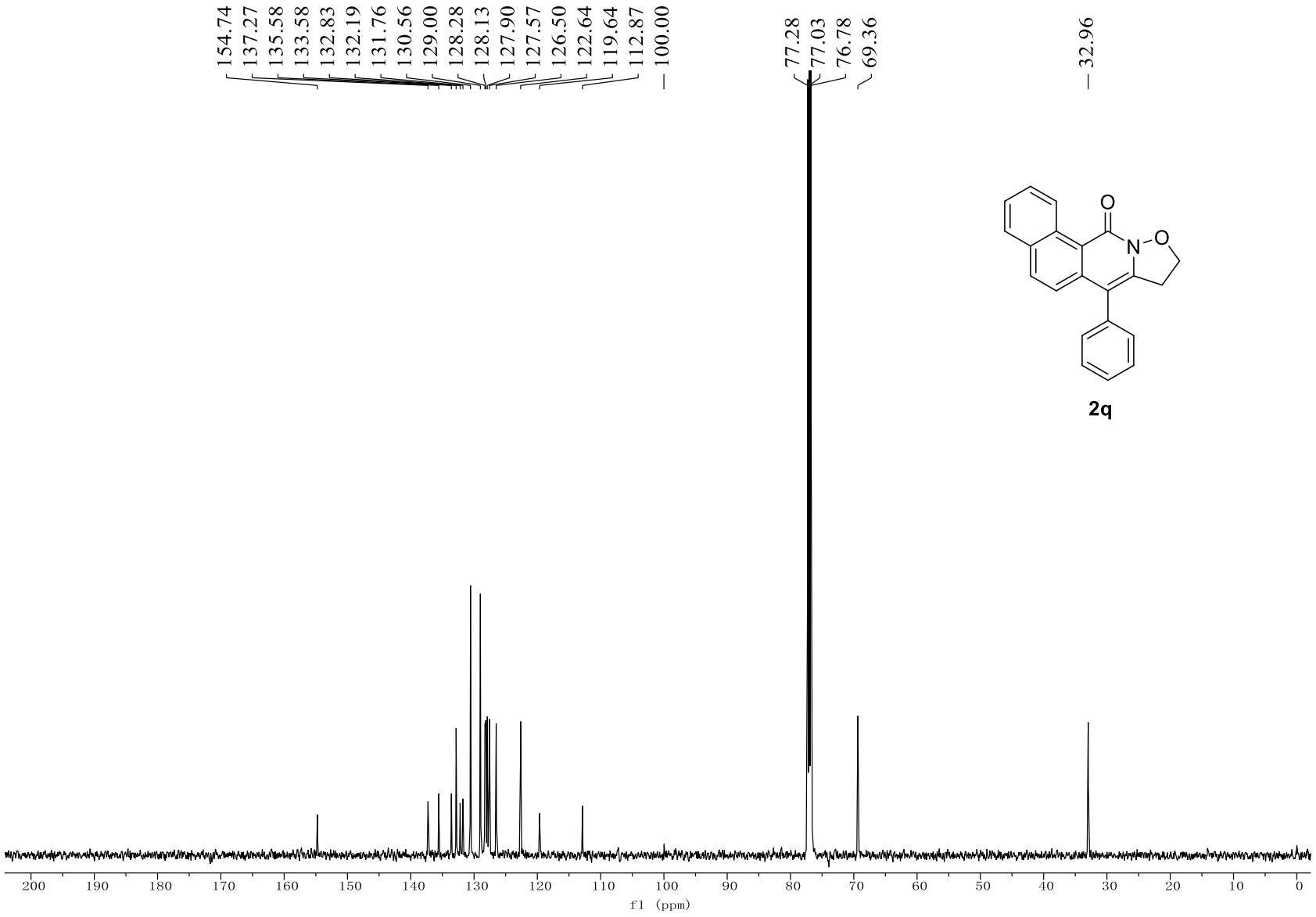

Br
O
N
O

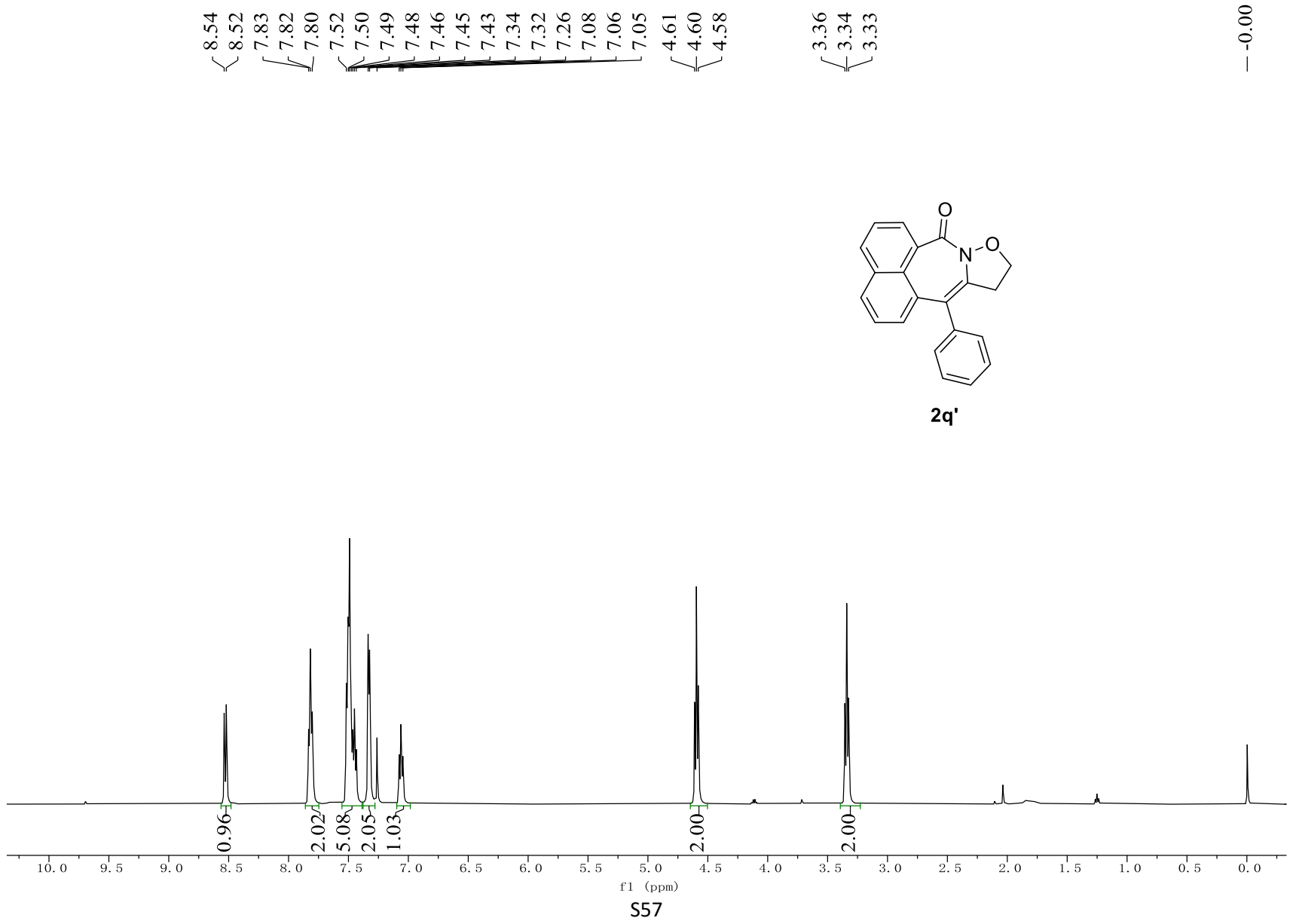
2p'

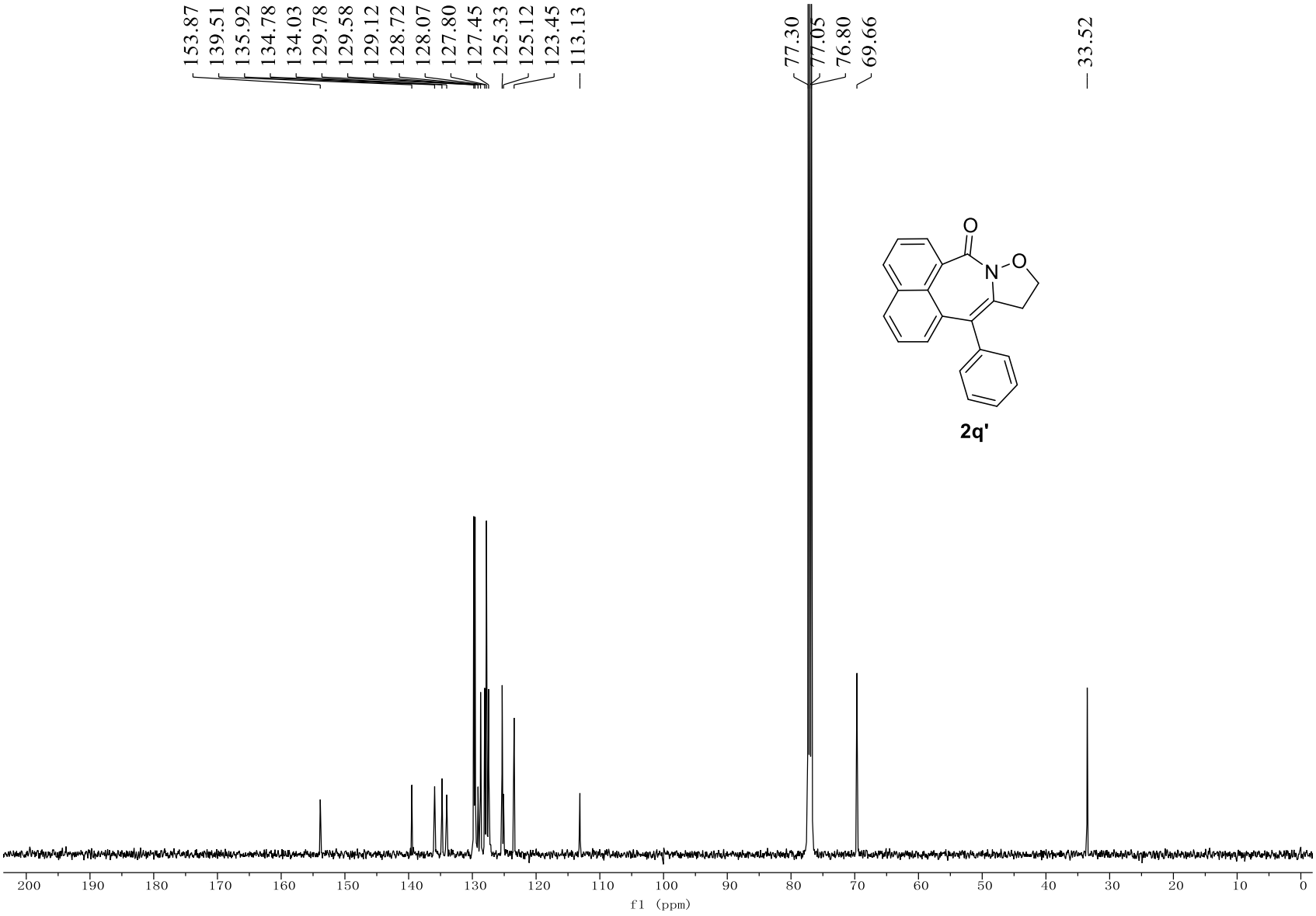

0.90
0.47
0.48
0.97
2.99
1.49
2.99
1.53
1.98
0.96
2.00
0.98
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

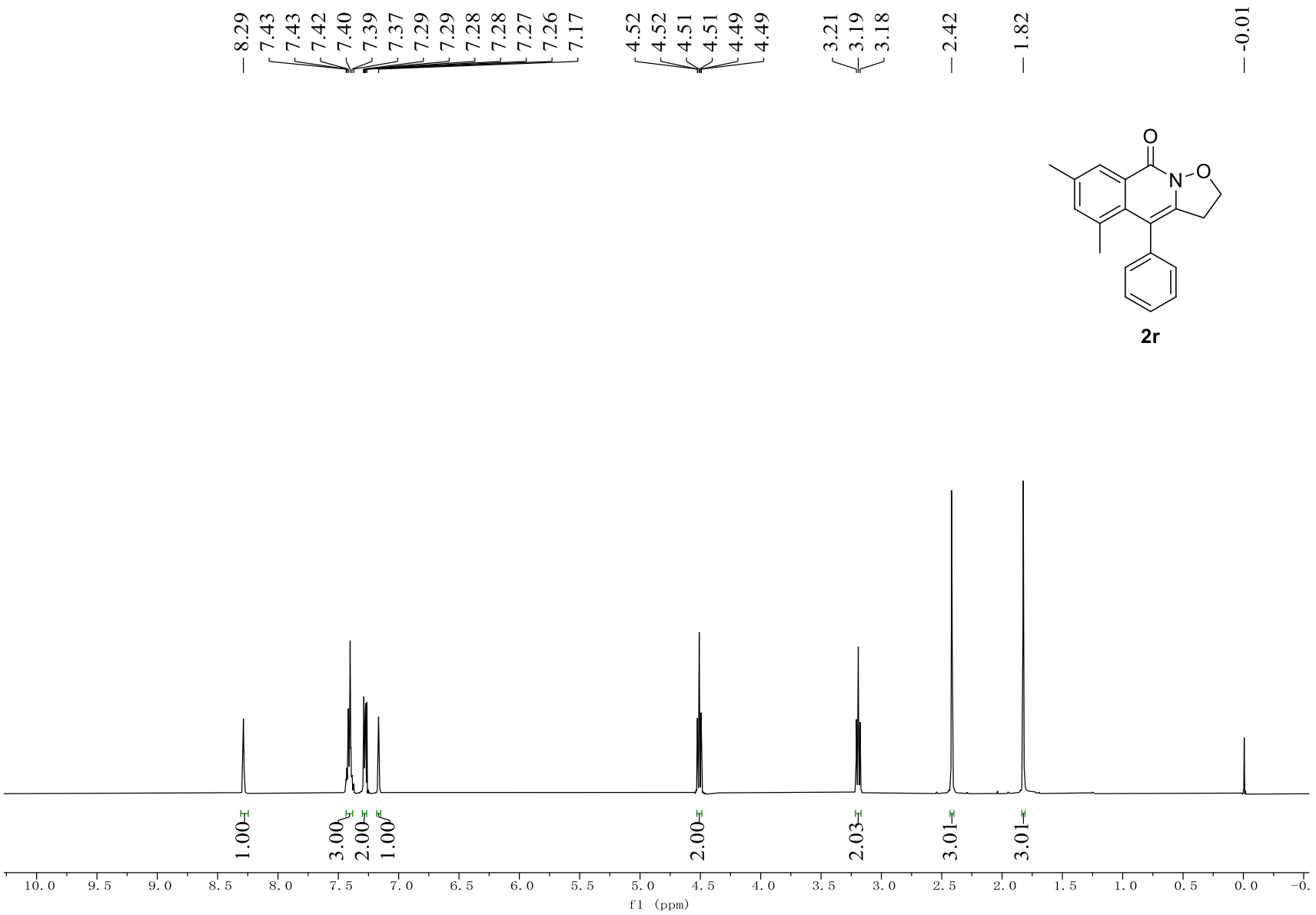

153.2208
152.7353
139.2666
137.2104
135.4575
134.9087
134.8169
134.4652
133.6363
133.1132
130.8658
130.1981
129.9571
129.0710
128.4083
128.3506
127.9428
127.7243
127.6510
126.8255
126.4990
120.5217
118.9029
112.6968
112.3671
77.3487
77.0311
76.7135
69.7081
69.5563
33.4360
32.4709
Br
O
N
O
2p
Br
O
N
O
2p'
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

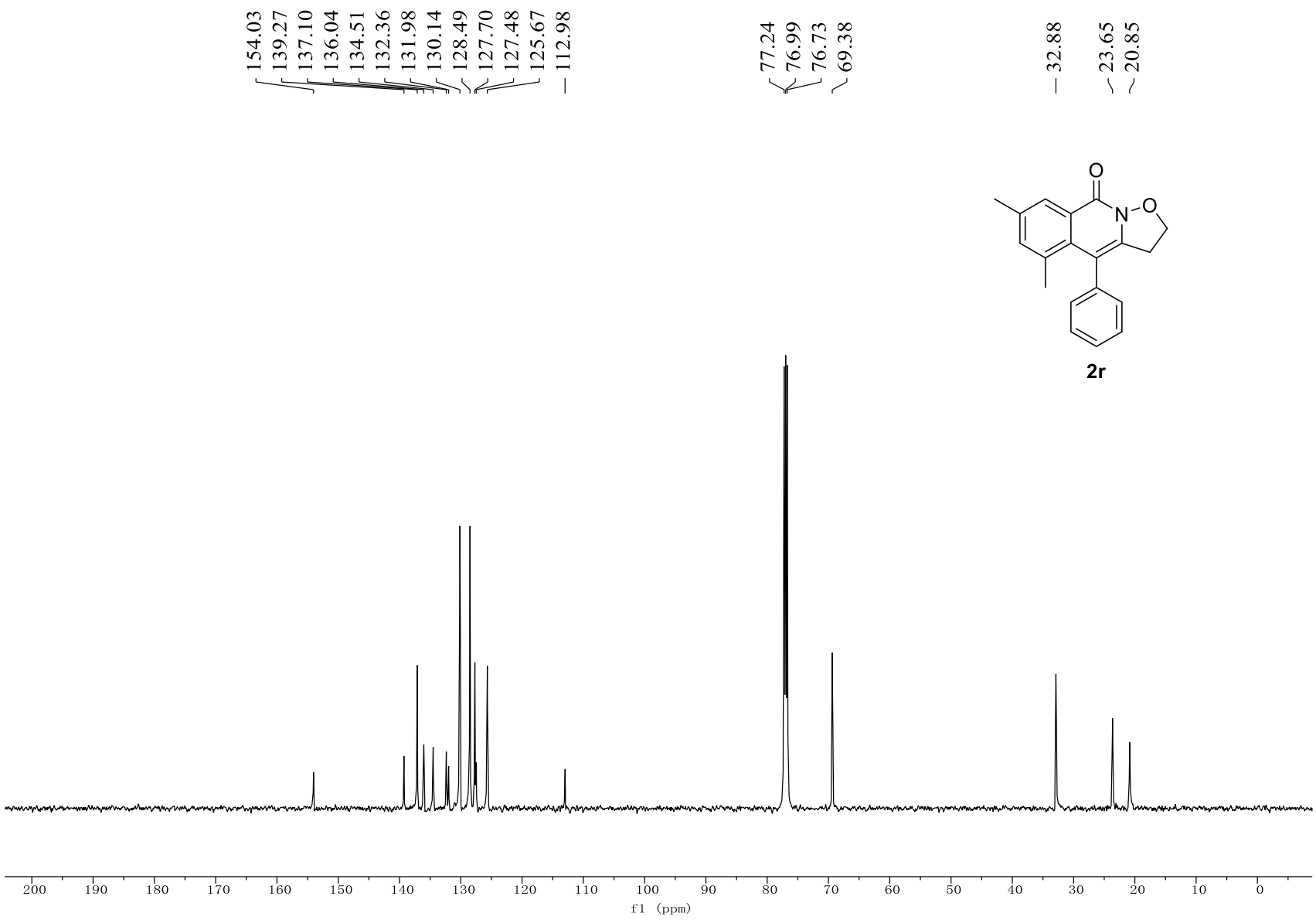

10.34
10.32
7.87
7.85
7.77
7.75
7.74
7.62
7.60
7.59
7.54
7.52
7.51
7.48
7.47
7.45
7.37
7.37
7.36
7.26
4.65
4.63
4.62
3.47
3.46
3.44
0.00
O
N
O
2q
1.00
2.00
1.01
1.01
3.01
3.01
2.01
2.01
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

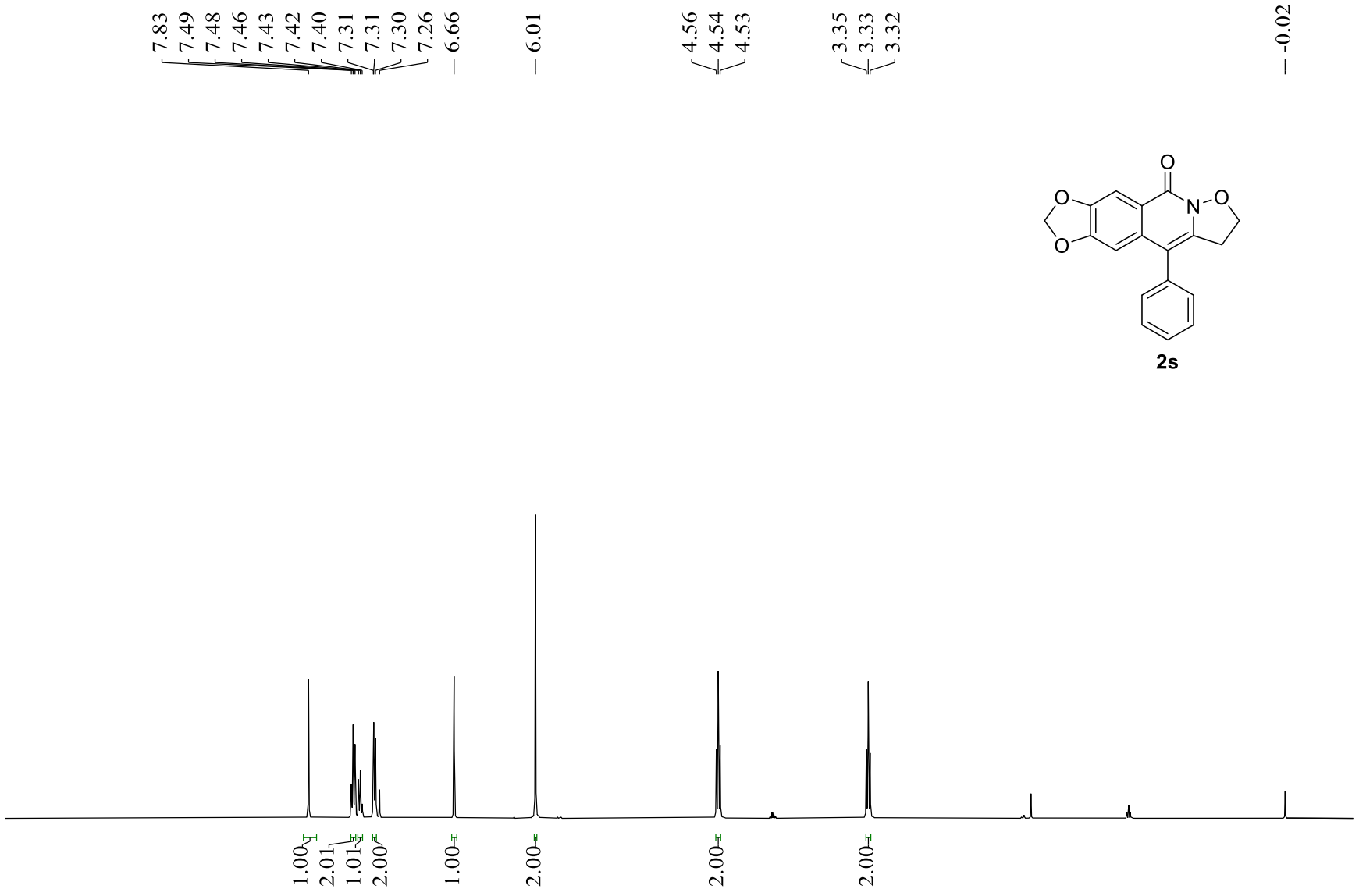

154.74
137.27
135.58
133.58
132.83
132.19
131.76
130.56
129.00
128.28
128.13
127.90
127.57
126.50
122.64
119.64
112.87
100.00
77.28
77.03
76.78
69.36
32.96
O
N
O
2q
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

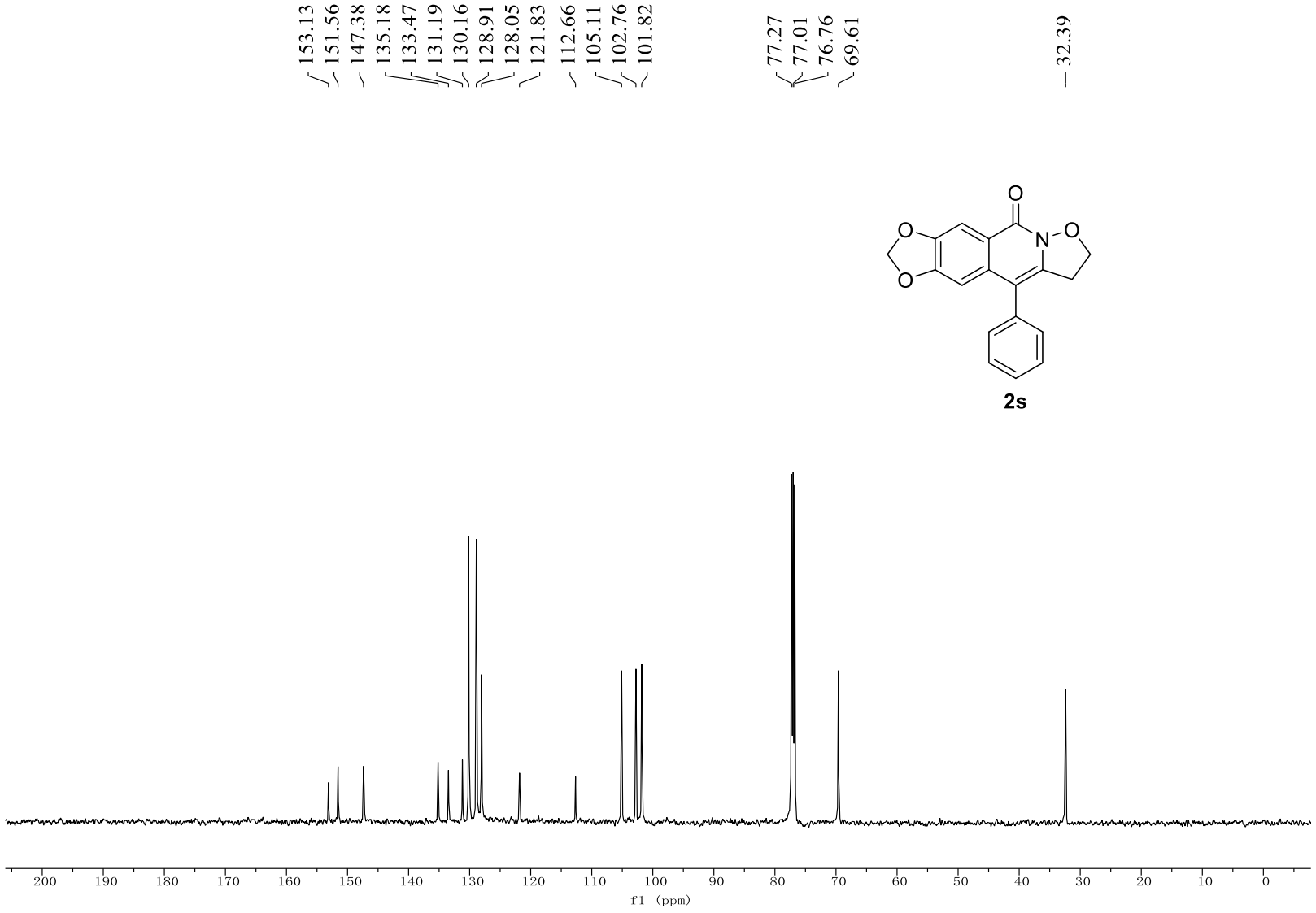

8.54
8.52
7.83
7.82
7.80
7.52
7.50
7.49
7.48
7.46
7.45
7.43
7.34
7.32
7.26
7.08
7.06
7.05
4.61
4.60
4.58
3.36
3.34
3.33
-0.00
O
N
O
2q'
0.96
2.02
5.08
2.05
1.03
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

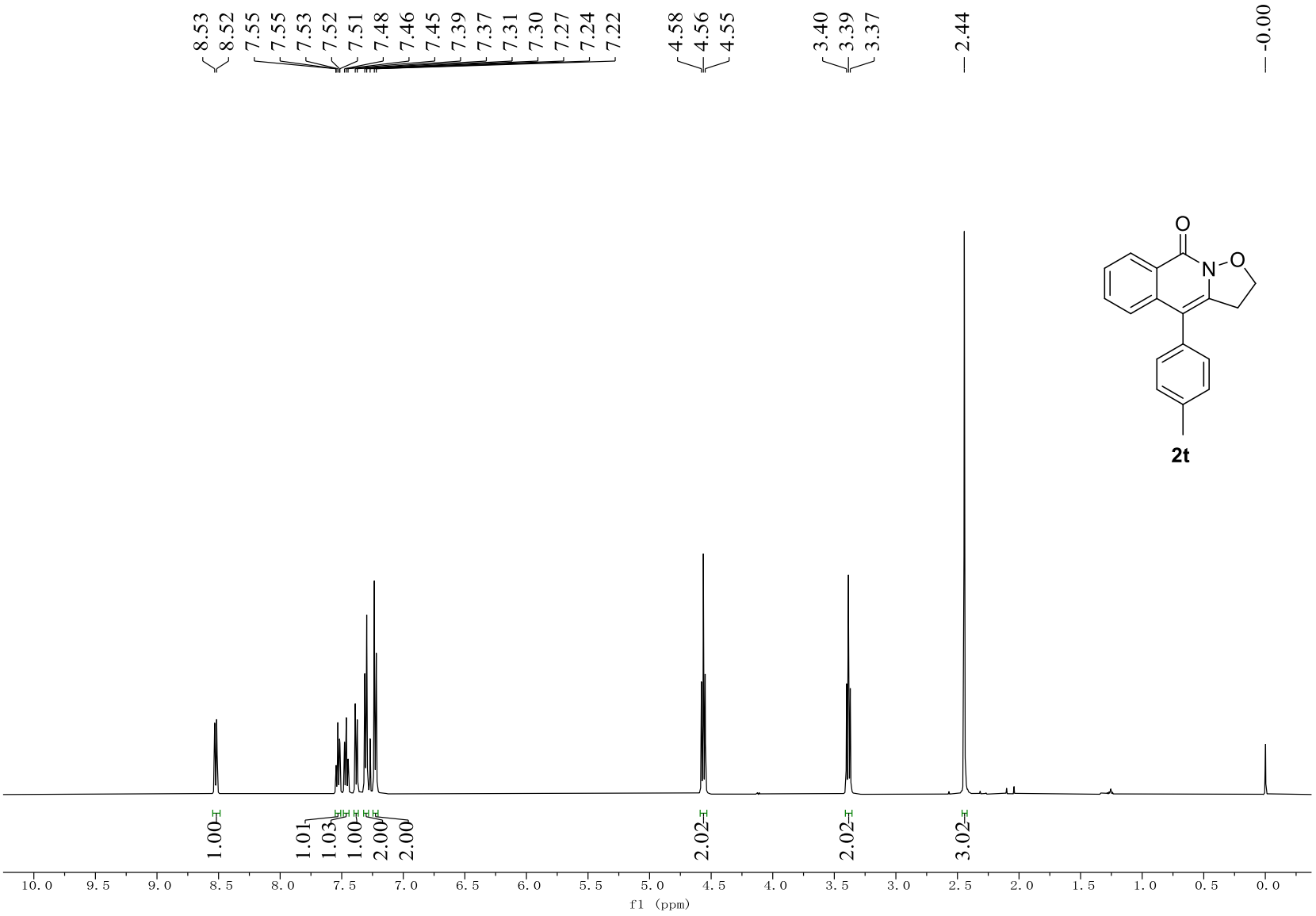

153.87
139.51
135.92
134.78
134.03
129.78
129.58
129.12
128.72
128.07
127.80
127.45
125.33
125.12
123.45
113.13
77.30
77.05
76.80
69.66
33.52
O
N
O
2q'
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

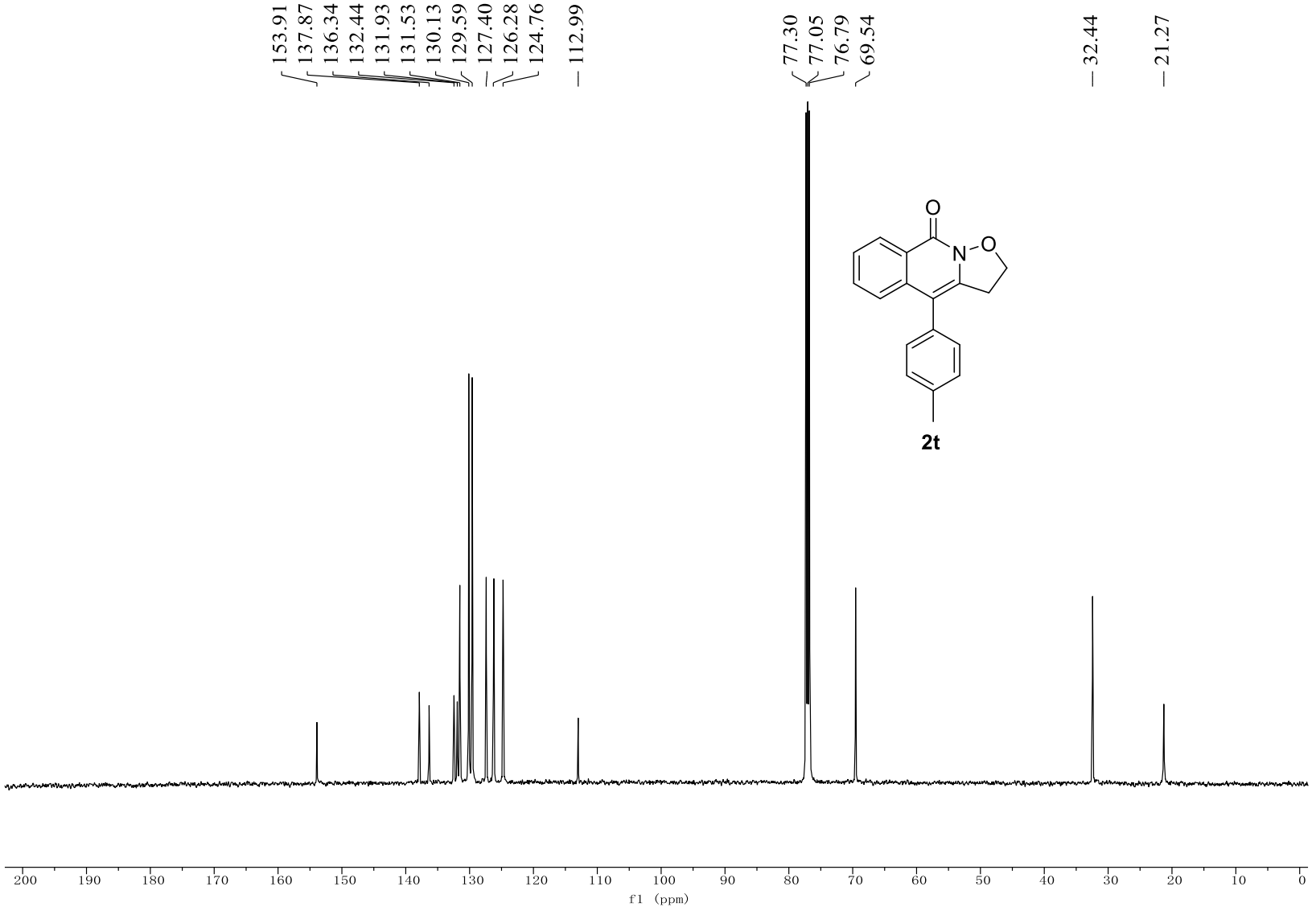

8.29
7.43
7.43
7.42
7.40
7.39
7.37
7.29
7.29
7.28
7.28
7.27
7.26
7.17
4.52
4.52
4.51
4.51
4.49
4.49
3.21
3.19
3.18
2.42
1.82
-0.01
O
N
O
2r
1.00
3.00
2.00
1.00
2.00
2.03
3.01
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

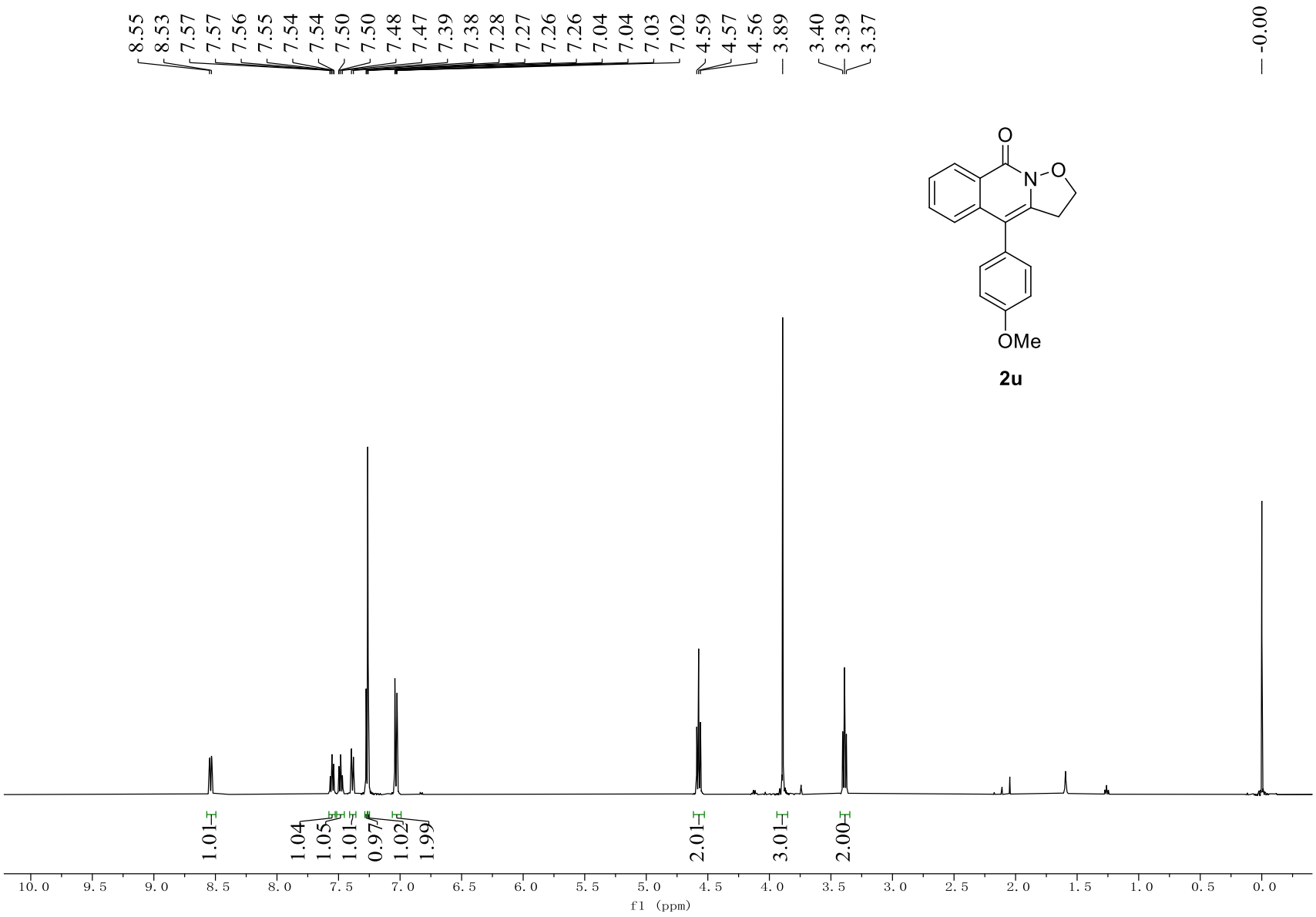

154.03
139.27
137.10
136.04
134.51
132.36
131.98
130.14
128.49
127.70
127.48
125.67
112.98
77.24
76.99
76.73
69.38
32.88
23.65
20.85
O
N
O
2r
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

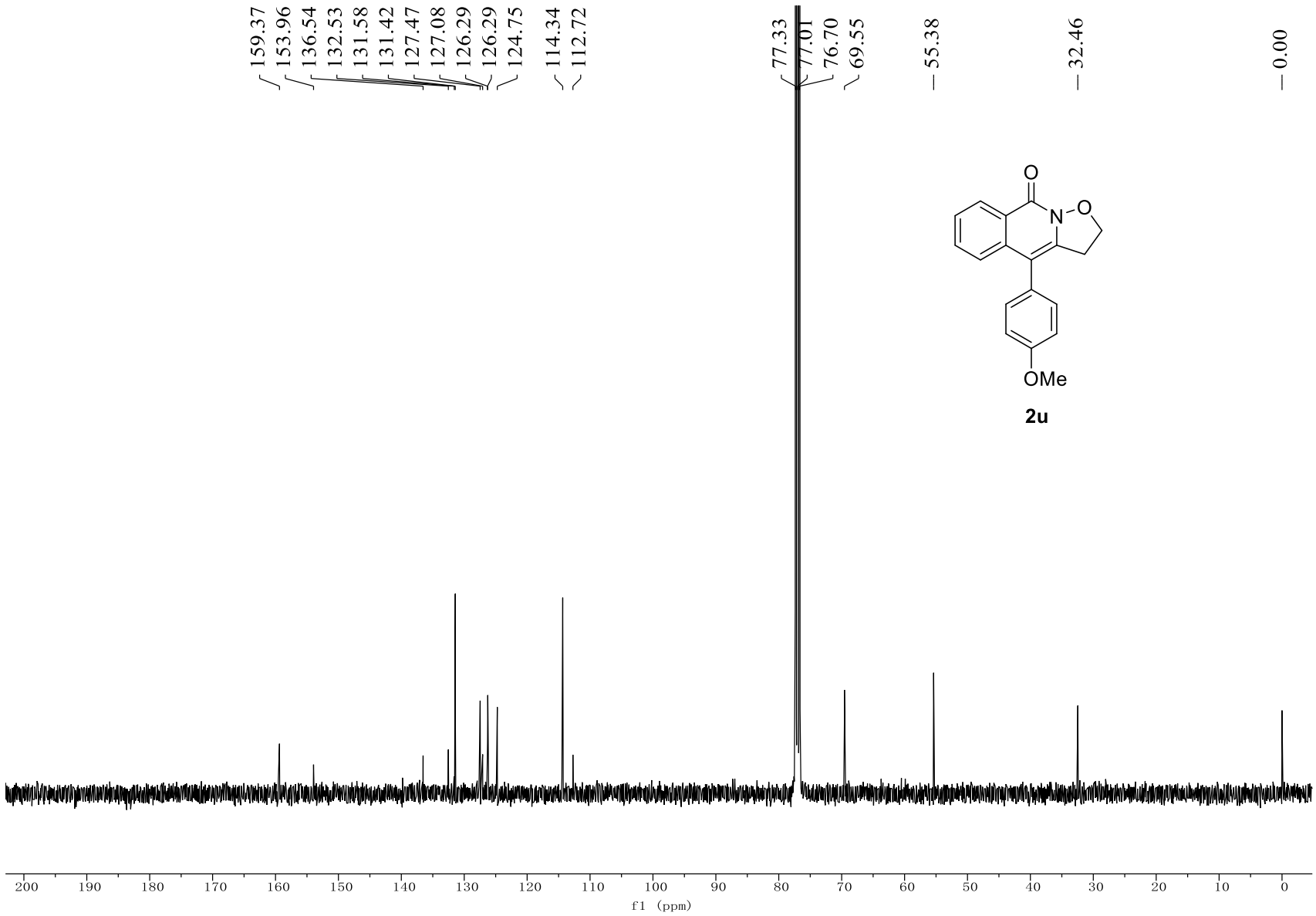

7.83
7.49
7.48
7.46
7.43
7.42
7.40
7.31
7.31
7.30
7.26
6.66
6.01
4.56
4.54
4.53
3.35
3.33
3.32
-0.02
O
O
O
N
O
2s
1.00
2.01
1.01
2.00
1.00
2.00
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

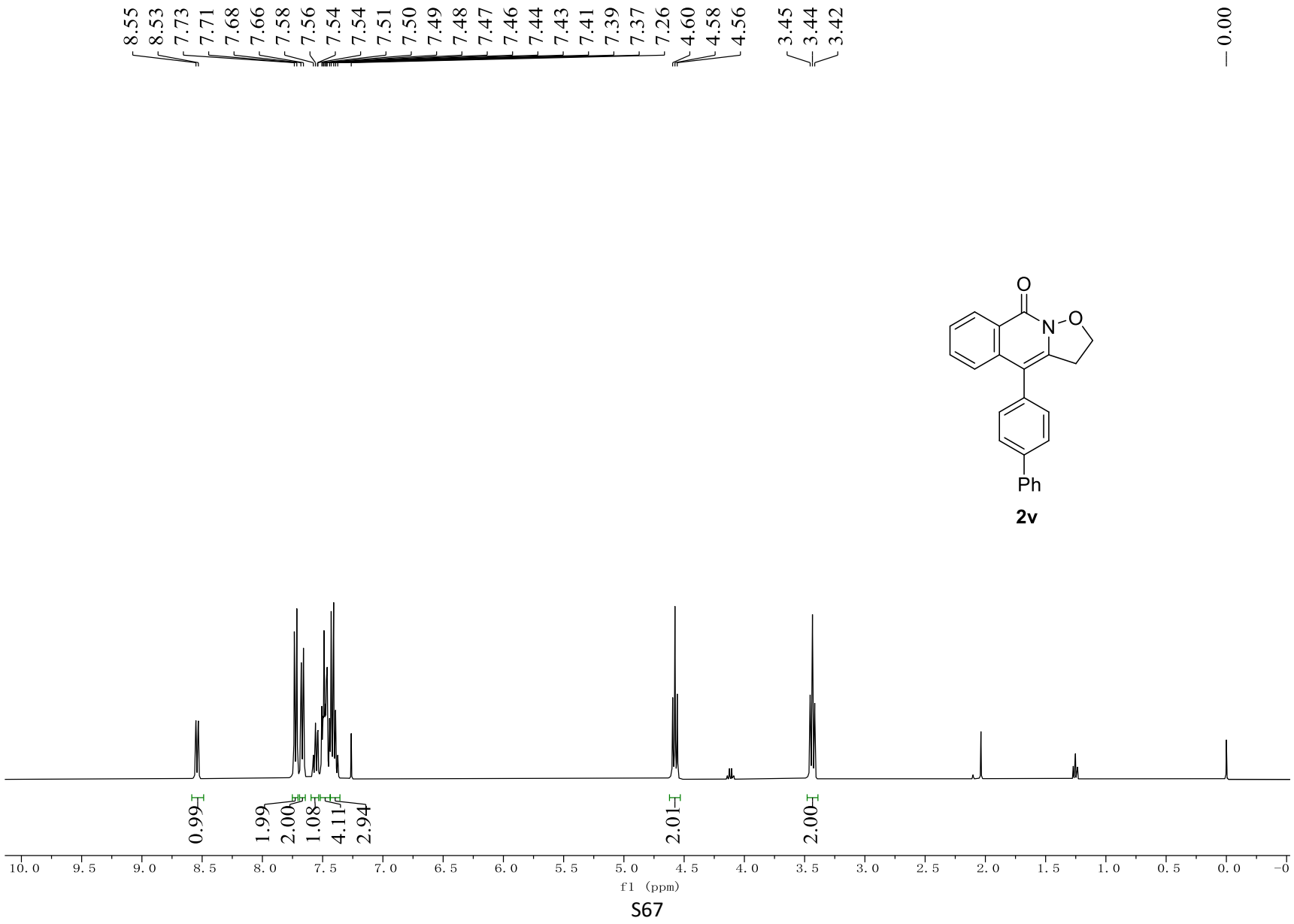

153.13
151.56
147.38
135.18
133.47
131.19
130.16
128.91
128.05
121.83
112.66
105.11
102.76
101.82
77.27
77.01
76.76
69.61
32.39
O
O
O
N
O
2s
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

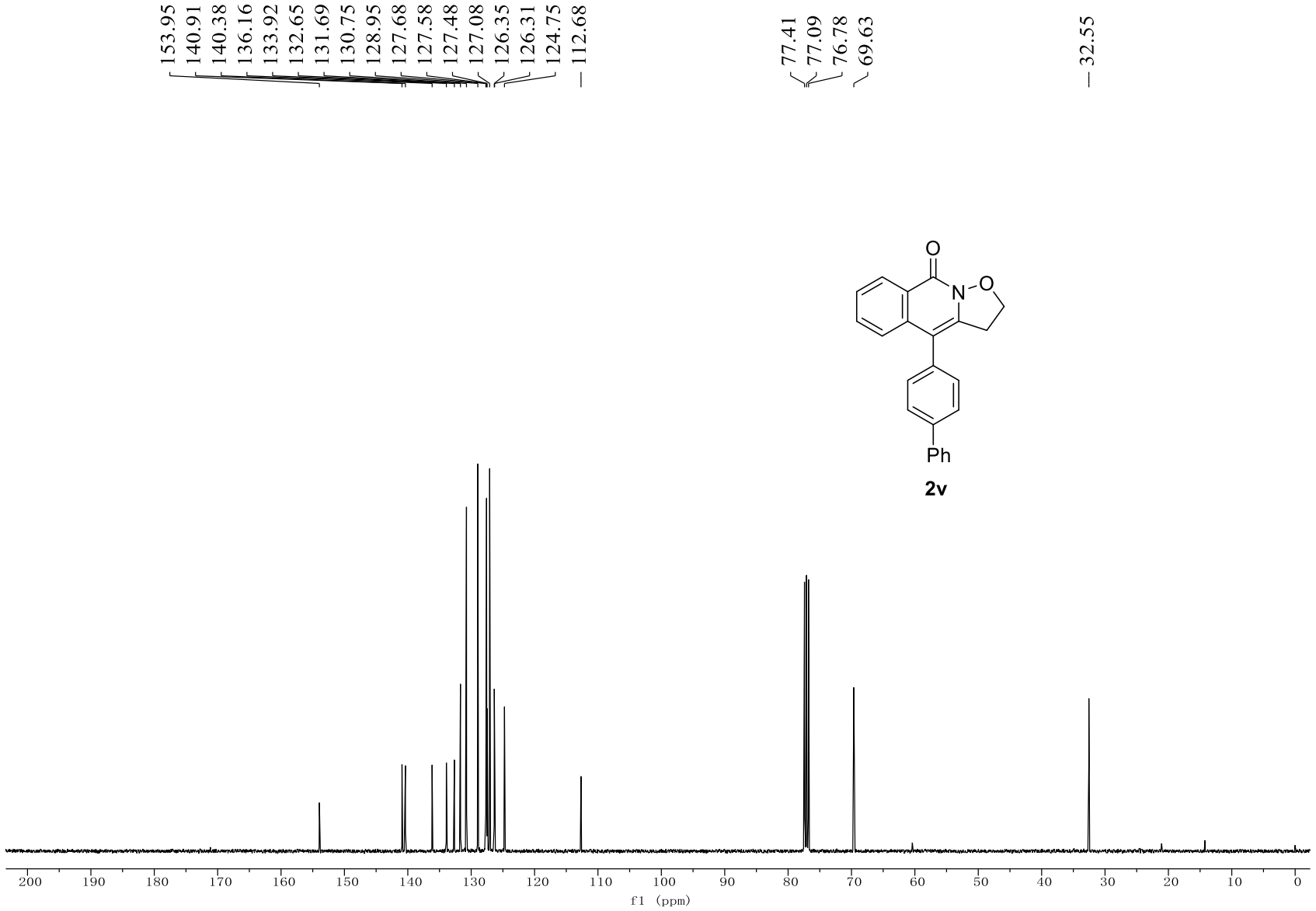

8.53
8.52
7.55
7.55
7.53
7.52
7.51
7.48
7.46
7.45
7.39
7.37
7.31
7.30
7.27
7.24
7.22
4.58
4.56
4.55
3.40
3.39
3.37
2.44
-0.00
O
N
O
2t
1.00
1.01
1.03
1.00
2.00
2.00
2.02
2.02
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

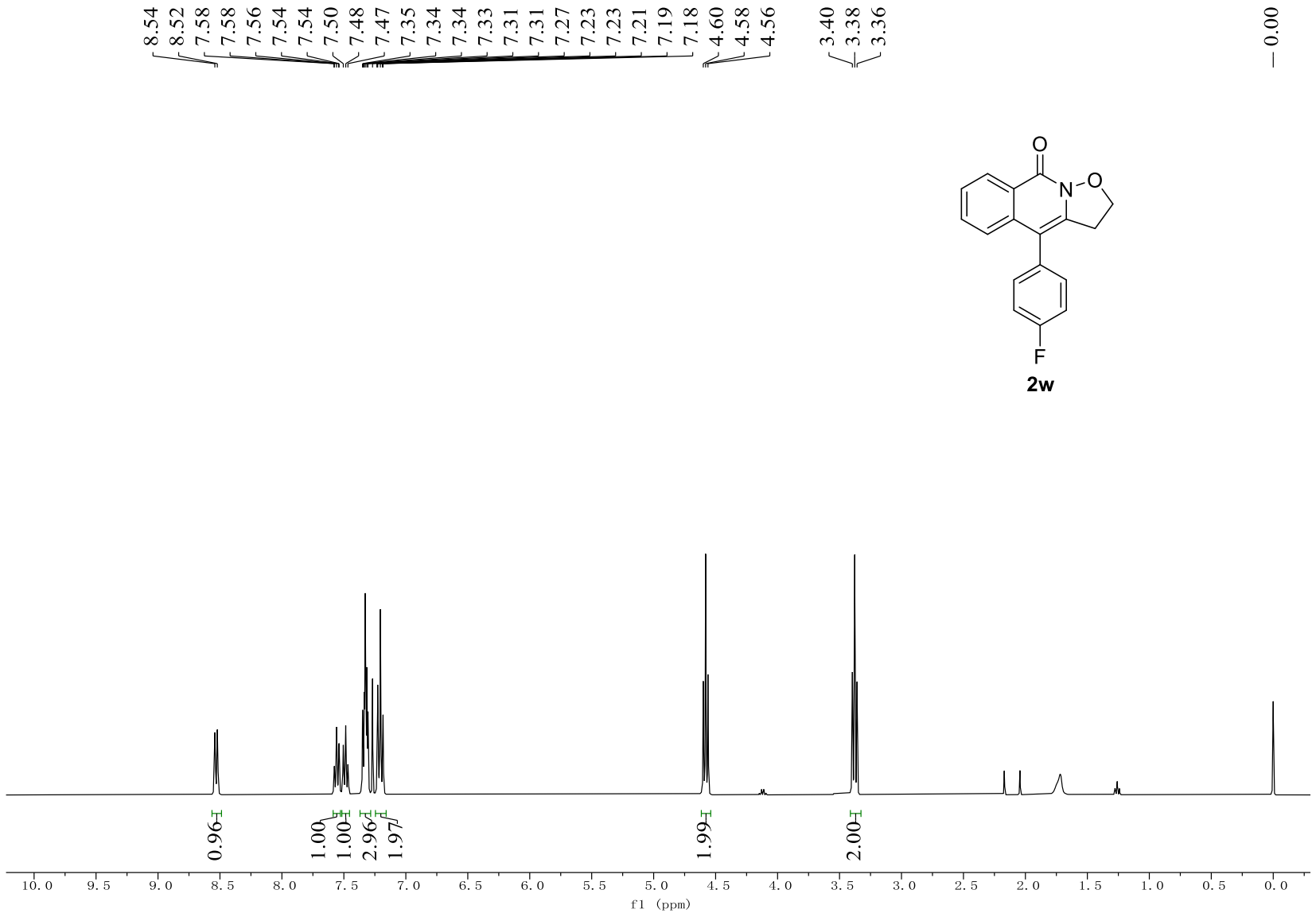

153.91
137.87
136.34
132.44
131.93
131.53
130.13
129.59
127.40
126.28
124.76
112.99
77.30
77.05
76.79
69.54
32.44
21.27
O
N
O
2t
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

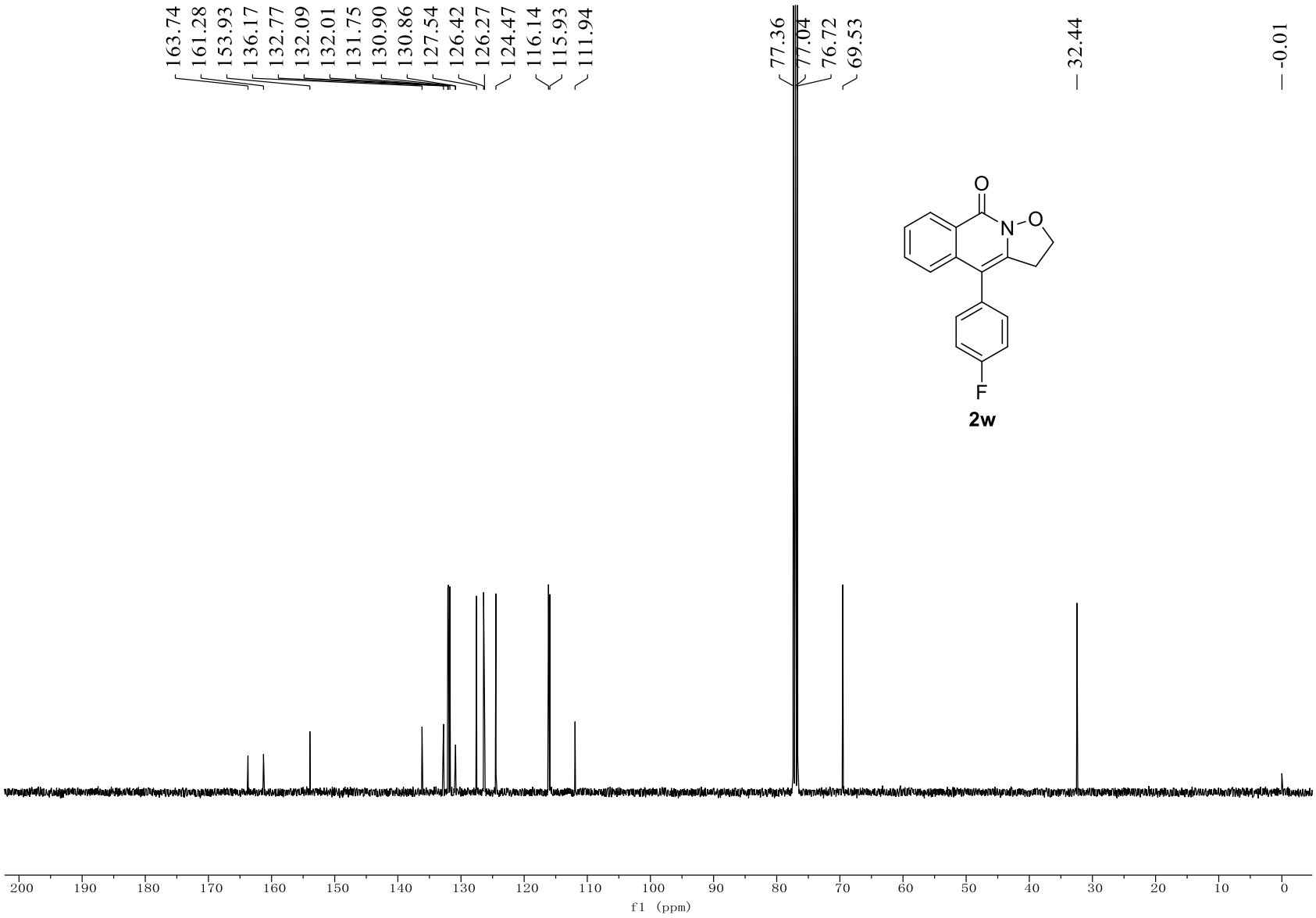

8.55
8.53
7.57
7.57
7.56
7.55
7.54
7.54
7.50
7.50
7.48
7.47
7.39
7.38
7.28
7.27
7.26
7.26
7.04
7.04
7.03
7.02
4.59
4.57
4.56
3.89
3.40
3.39
3.37
0.00
O
N
O
OMe
2u
1.01
1.04
1.05
1.01
0.97
1.02
1.99
2.01
3.01
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

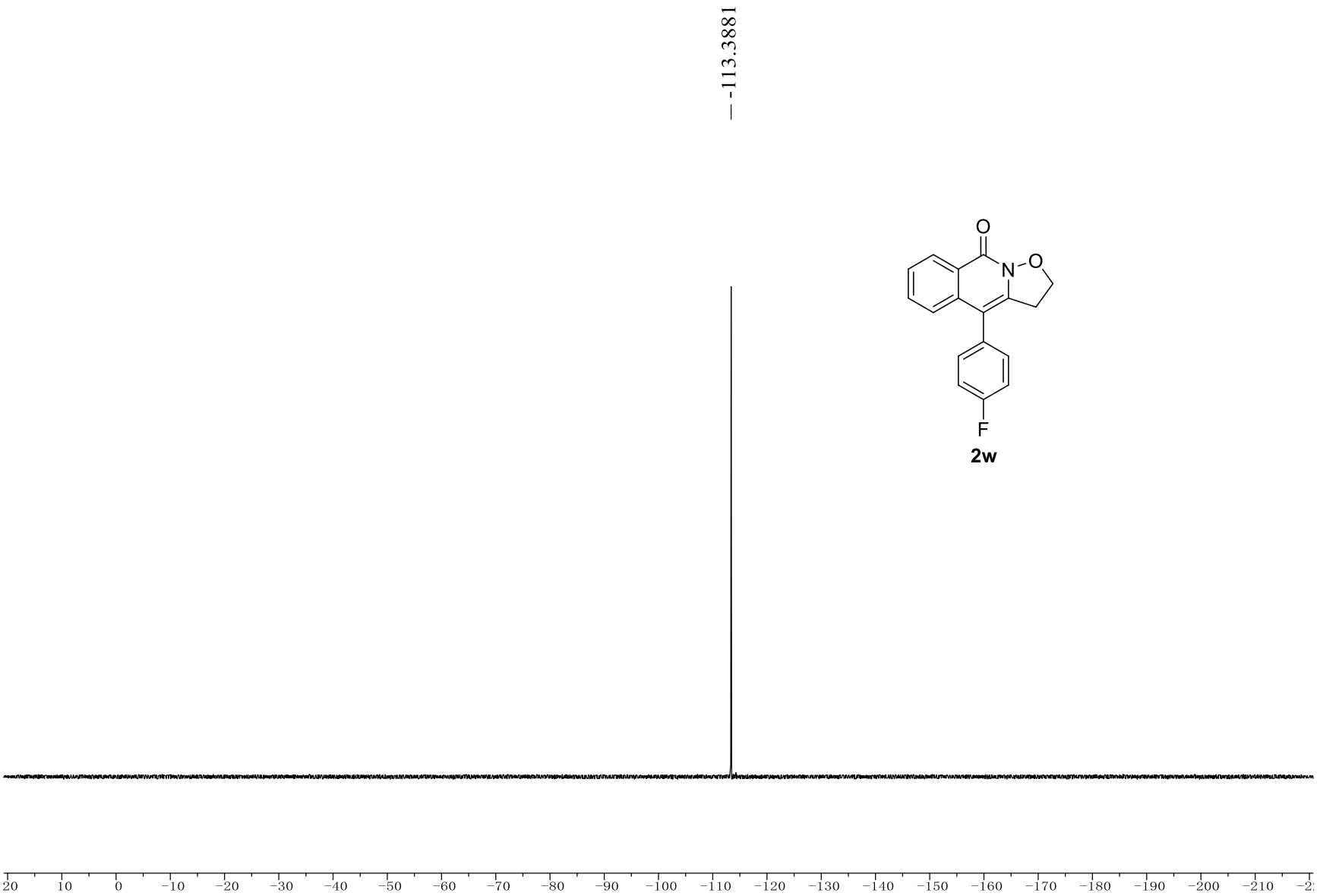

159.37
153.96
136.54
132.53
131.58
131.42
127.47
127.08
126.29
126.29
124.75
114.34
112.72
77.33
77.01
76.70
69.55
55.38
32.46
0.00
O
N
O
OMe
2u
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

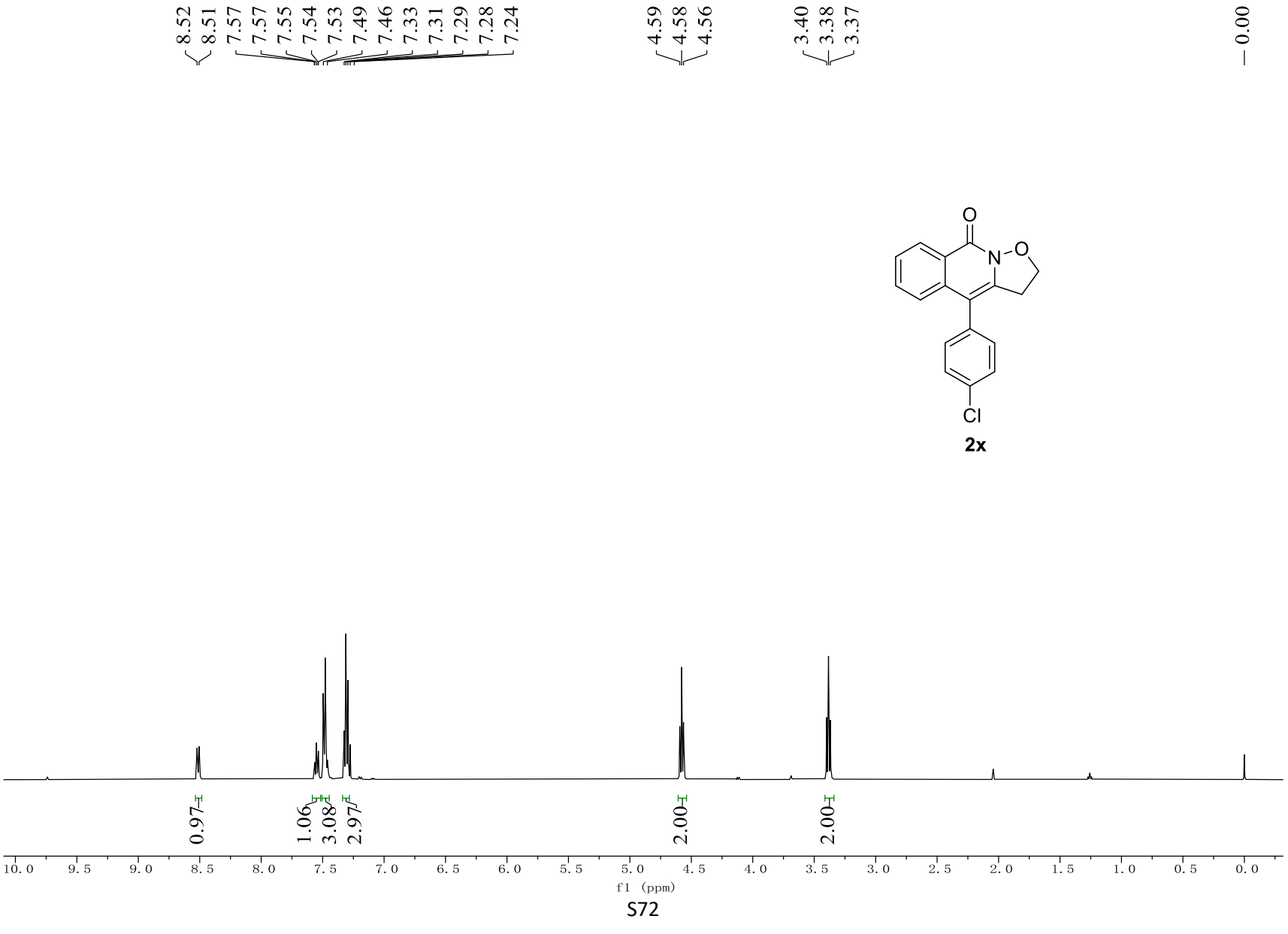

8.55
8.53
7.73
7.71
7.68
7.66
7.58
7.56
7.54
7.54
7.51
7.50
7.49
7.48
7.47
7.46
7.44
7.43
7.41
7.39
7.37
7.26
4.60
4.58
4.56
3.45
3.44
3.42
0.00
O
N
O
Ph
2v
0.99
1.99
2.00
1.08
4.11
2.94
2.01
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

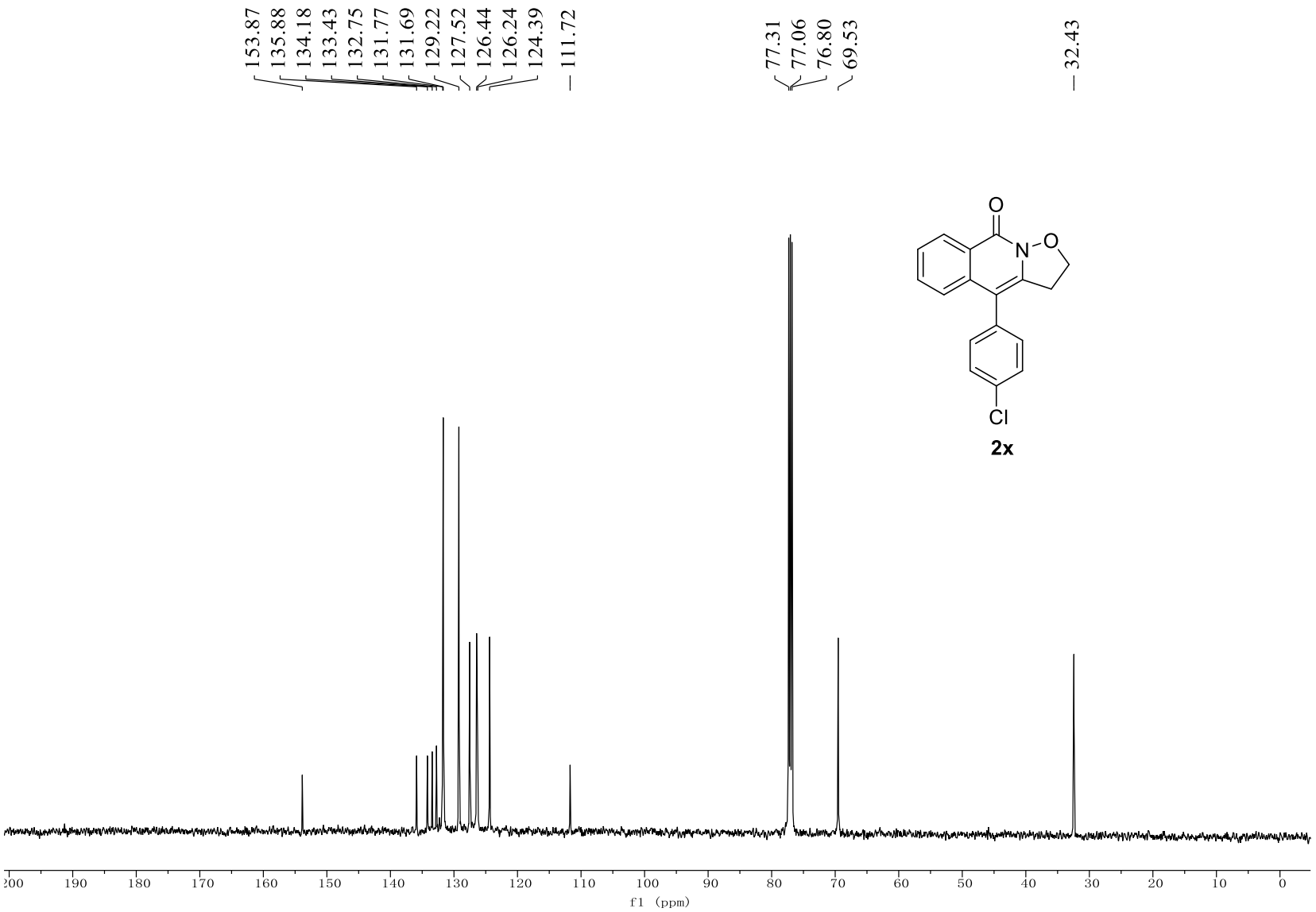

153.95
140.91
140.38
136.16
133.92
132.65
131.69
130.75
128.95
127.68
127.58
127.48
127.08
126.35
126.31
124.75
112.68
77.41
77.09
76.78
69.63
32.55
O
N
O
Ph
2v
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

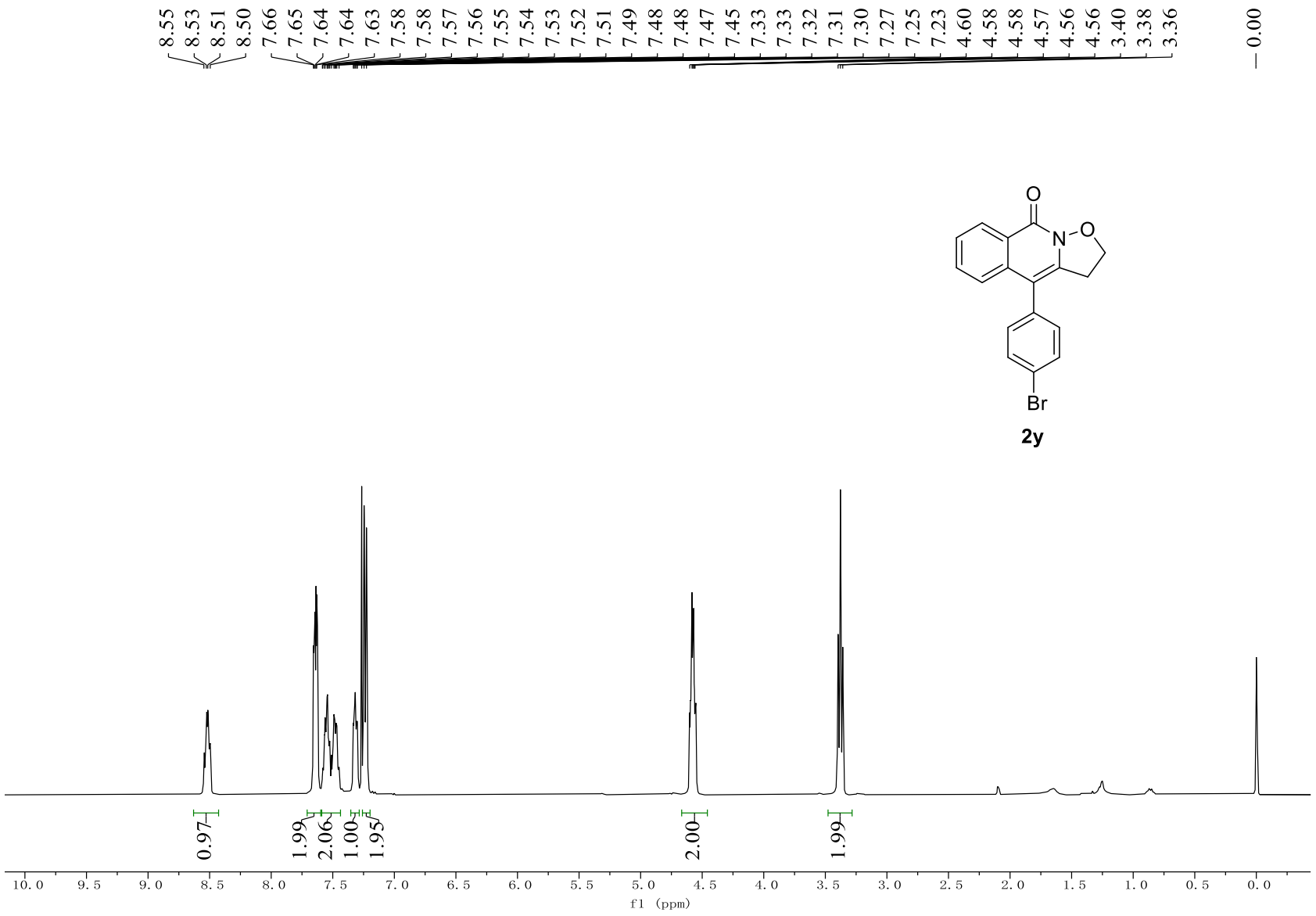

8.54
8.52
7.58
7.58
7.56
7.54
7.54
7.50
7.48
7.47
7.35
7.34
7.34
7.33
7.31
7.31
7.27
7.23
7.23
7.21
7.19
7.18
4.60
4.58
4.56
3.40
3.38
3.36
0.00
O
N
O
F
2w
0.96
1.00
1.00
2.96
1.97
1.99
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

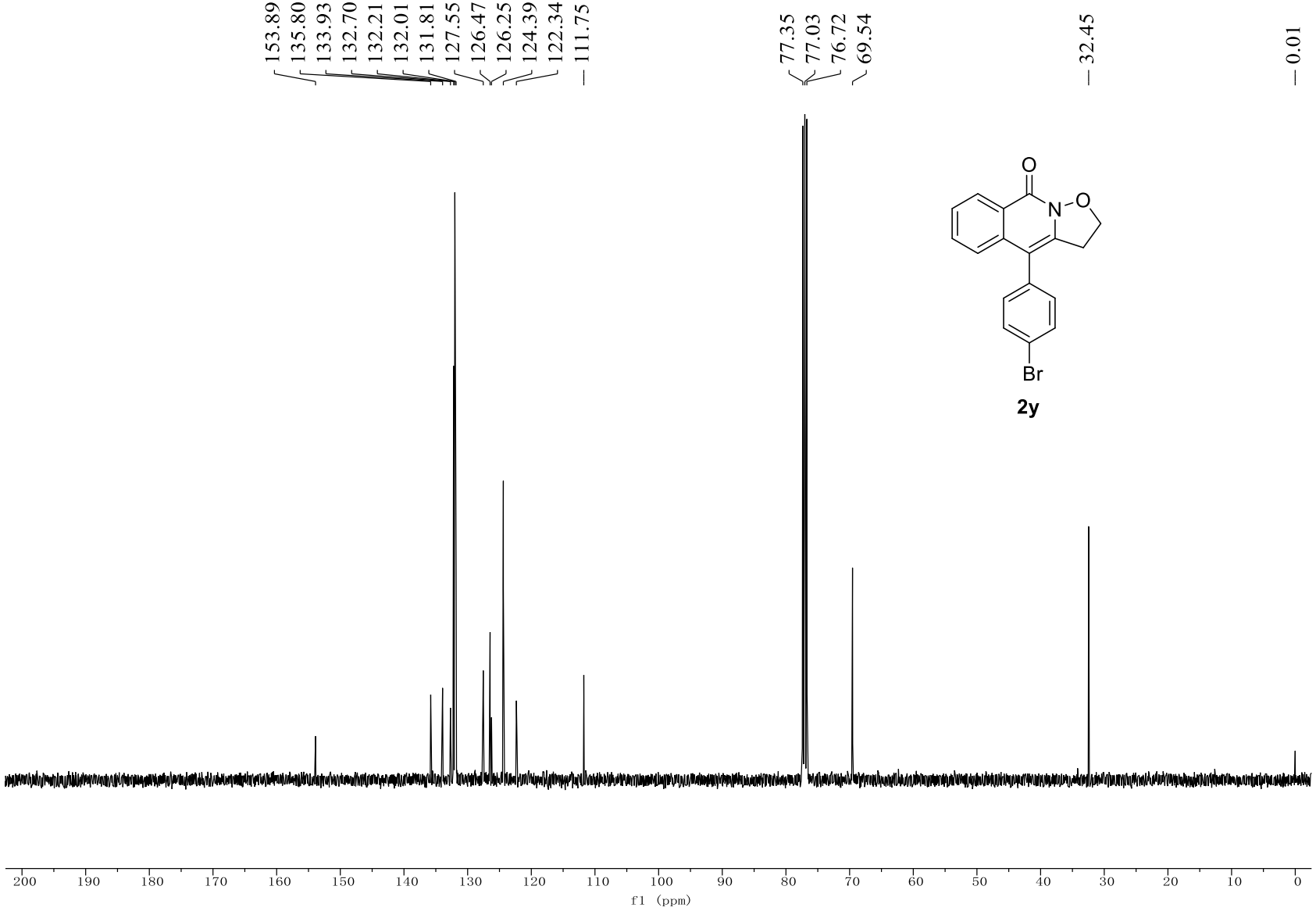

163.74
161.28
153.93
136.17
132.77
132.09
132.01
131.75
130.90
130.86
127.54
126.42
126.27
124.47
116.14
115.93
111.94
77.36
77.04
76.72
69.53
32.44
-0.01
O
N
O
F
2w
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-113.3881
O
N
O
F
2w
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)


8.52
8.51
7.57
7.57
7.55
7.54
7.53
7.49
7.46
7.33
7.31
7.29
7.28
7.24
4.59
4.58
4.56
3.40
3.38
3.37
0.00
O
N
O
Cl
2x
0.97
1.06
3.08
2.97
2.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.87
135.88
134.18
133.43
132.75
131.77
131.69
129.22
127.52
126.44
126.24
124.39
111.72
77.31
77.06
76.80
69.53
32.43
O
N
O
Cl
2x
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.55
8.53
8.51
8.50
7.66
7.65
7.64
7.64
7.63
7.58
7.58
7.57
7.56
7.55
7.54
7.53
7.52
7.51
7.49
7.48
7.48
7.47
7.45
7.33
7.33
7.32
7.31
7.30
7.27
7.25
7.23
4.60
4.58
4.58
4.57
4.56
4.56
3.40
3.38
3.36
0.00
O
N
O
Br
2y
0.97
1.99
2.06
1.00
1.95
2.00
1.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

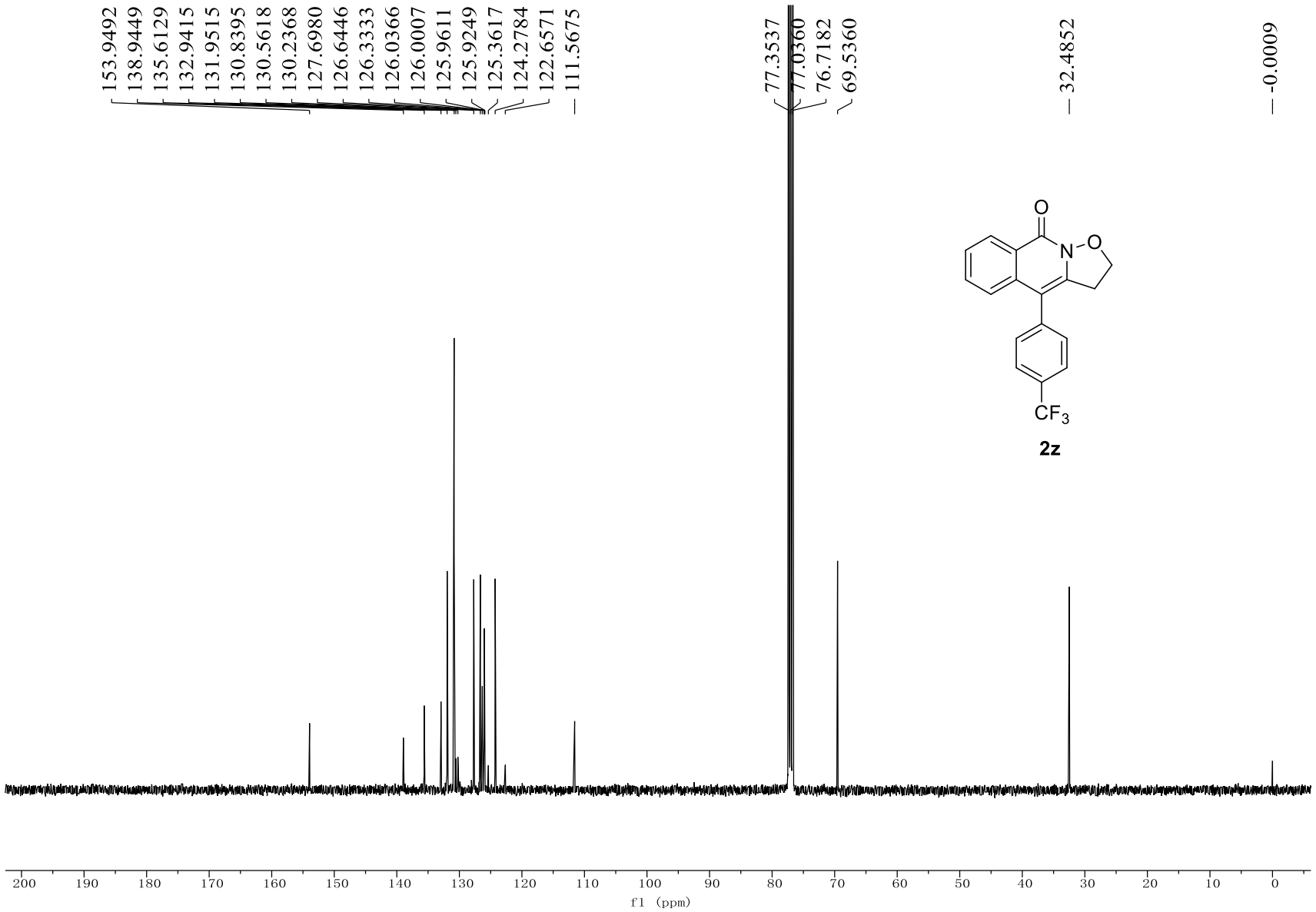

153.89
135.80
133.93
132.70
132.21
132.01
131.81
127.55
126.47
126.25
124.39
122.34
111.75
77.35
77.03
76.72
69.54
32.45
0.01
O
N
O
Br
2y
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

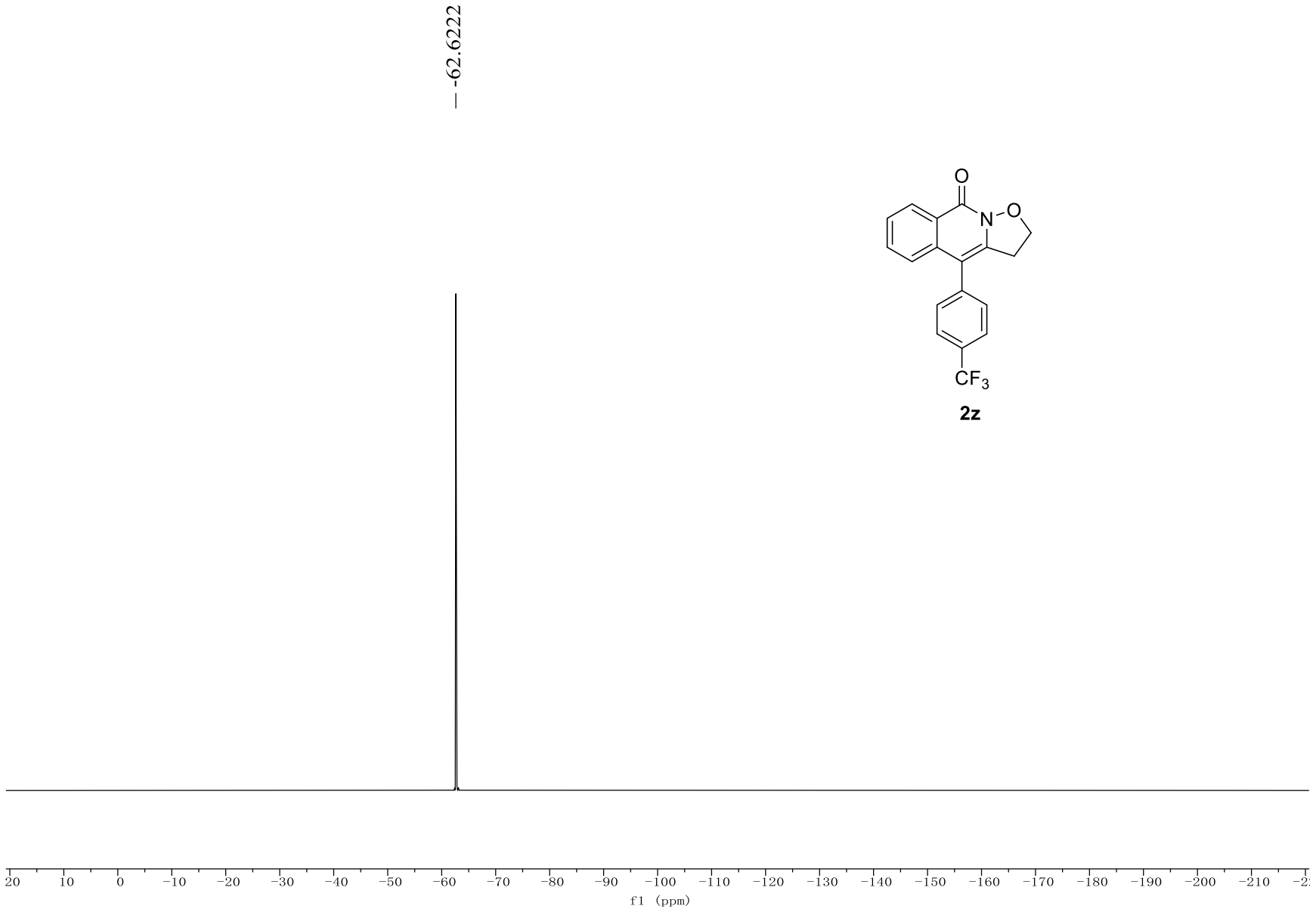
8.5600
8.5400
7.7942
7.7743
7.5936
7.5898
7.5759
7.5724
7.5558
7.5517
7.5218
7.5032
7.4888
7.4857
7.3137
7.2921
7.2657
4.6148
4.5959
4.5771
3.4147
3.3958
3.3768
-0.0001

O
N
O
CF3

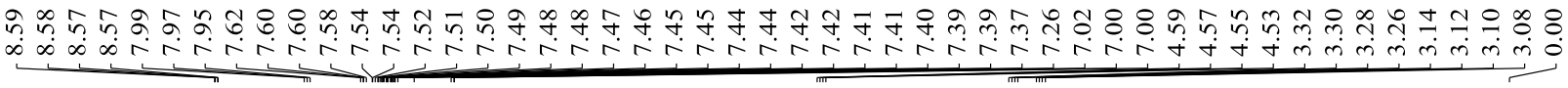
2z

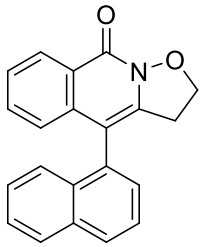
0.93
1.96
1.01
2.91
0.98
1.96
1.99
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


153.9492
138.9449
135.6129
132.9415
131.9515
130.8395
130.5618
130.2368
127.6980
126.6446
126.3333
126.0366
126.0007
125.9611
125.9249
125.3617
124.2784
122.6571
111.5675
77.3537
77.0360
76.7182
69.5360
32.4852
-0.0009
O
N
O
CF3
2z
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

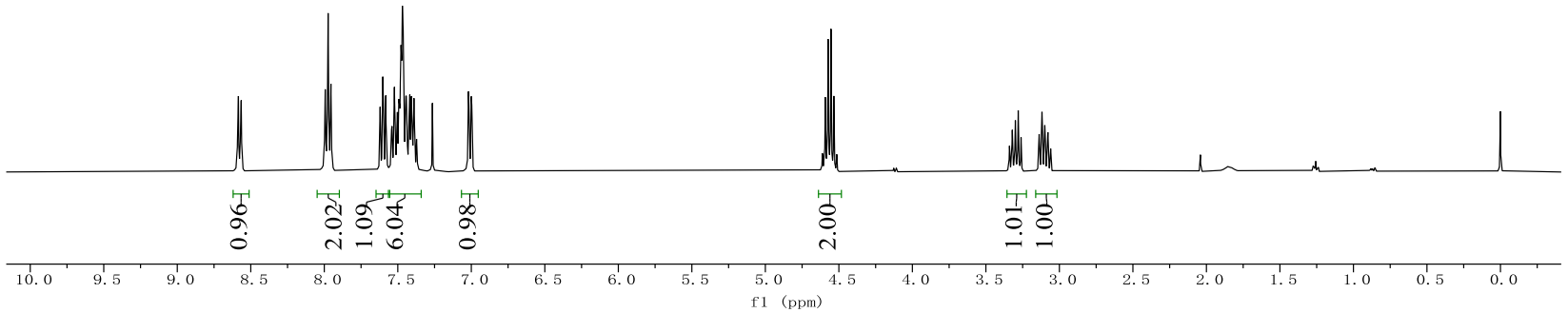

-62.6222
O
N
O
CF3
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

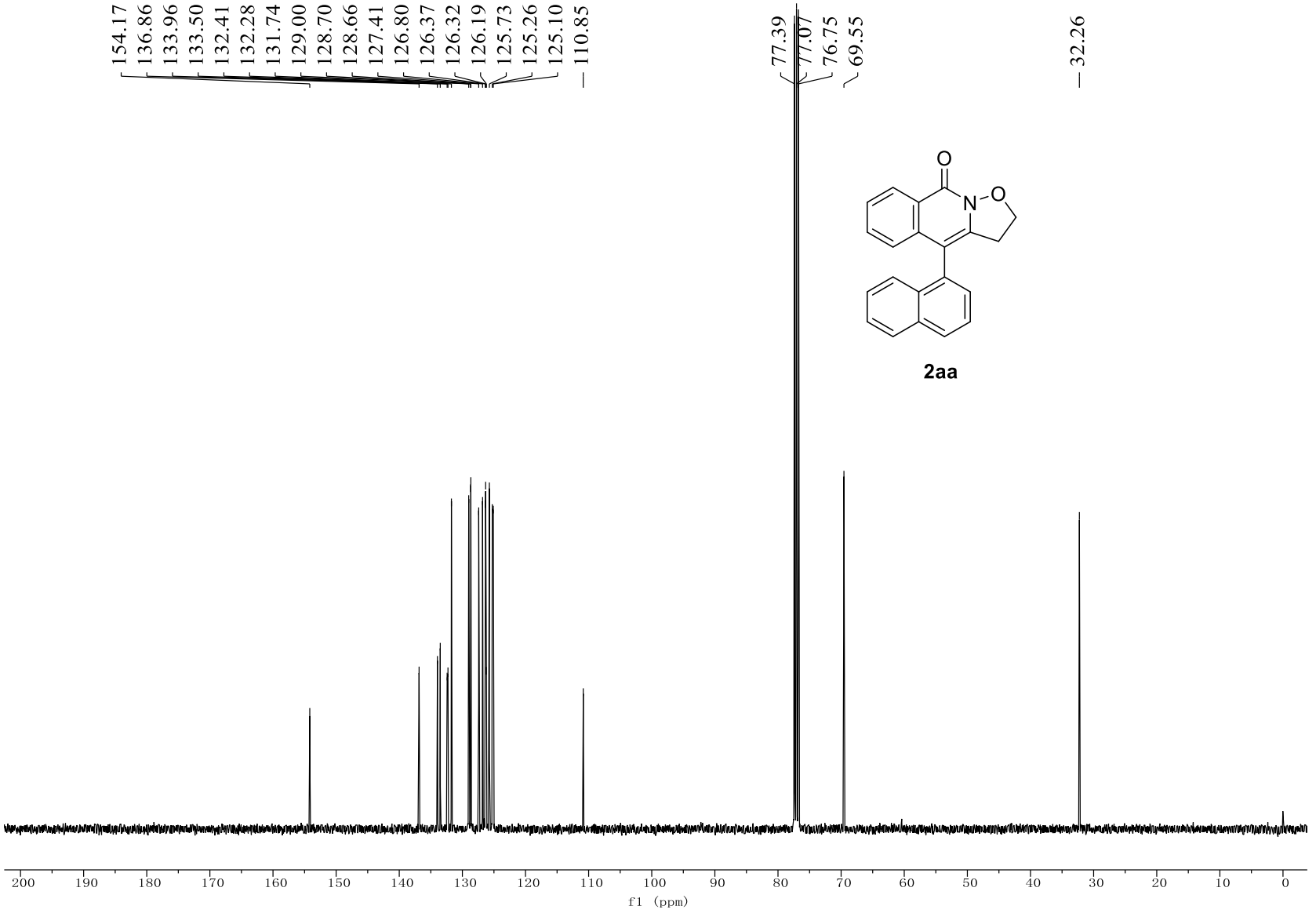
2z

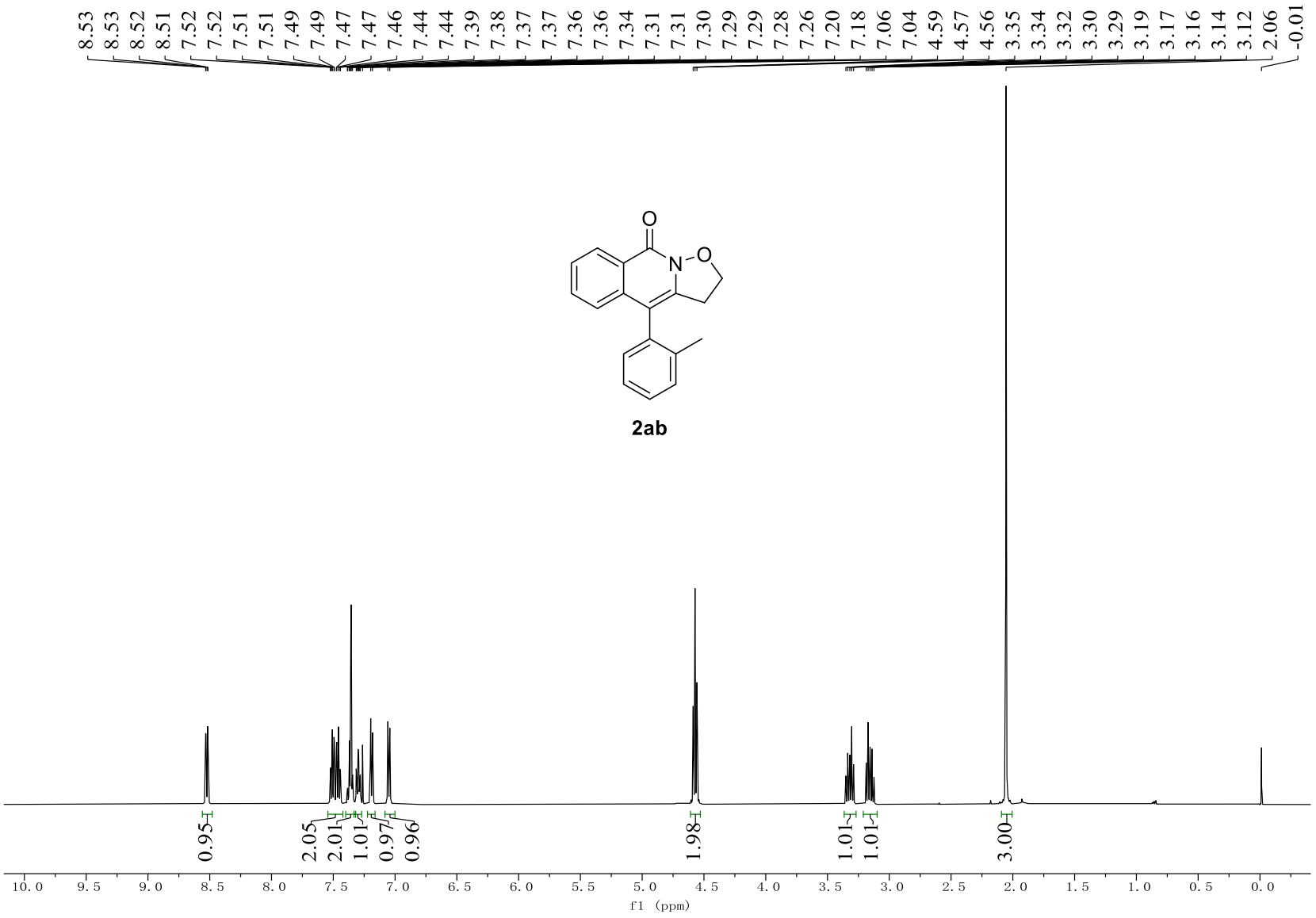

8.59
8.58
8.57
8.57
7.99
7.97
7.95
7.62
7.60
7.60
7.58
7.54
7.54
7.52
7.51
7.50
7.49
7.48
7.48
7.47
7.46
7.45
7.45
7.44
7.44
7.42
7.42
7.41
7.41
7.40
7.39
7.39
7.37
7.26
7.02
7.00
7.00
4.59
4.57
4.55
4.53
3.32
3.30
3.28
3.26
3.14
3.12
3.10
3.08
0.00

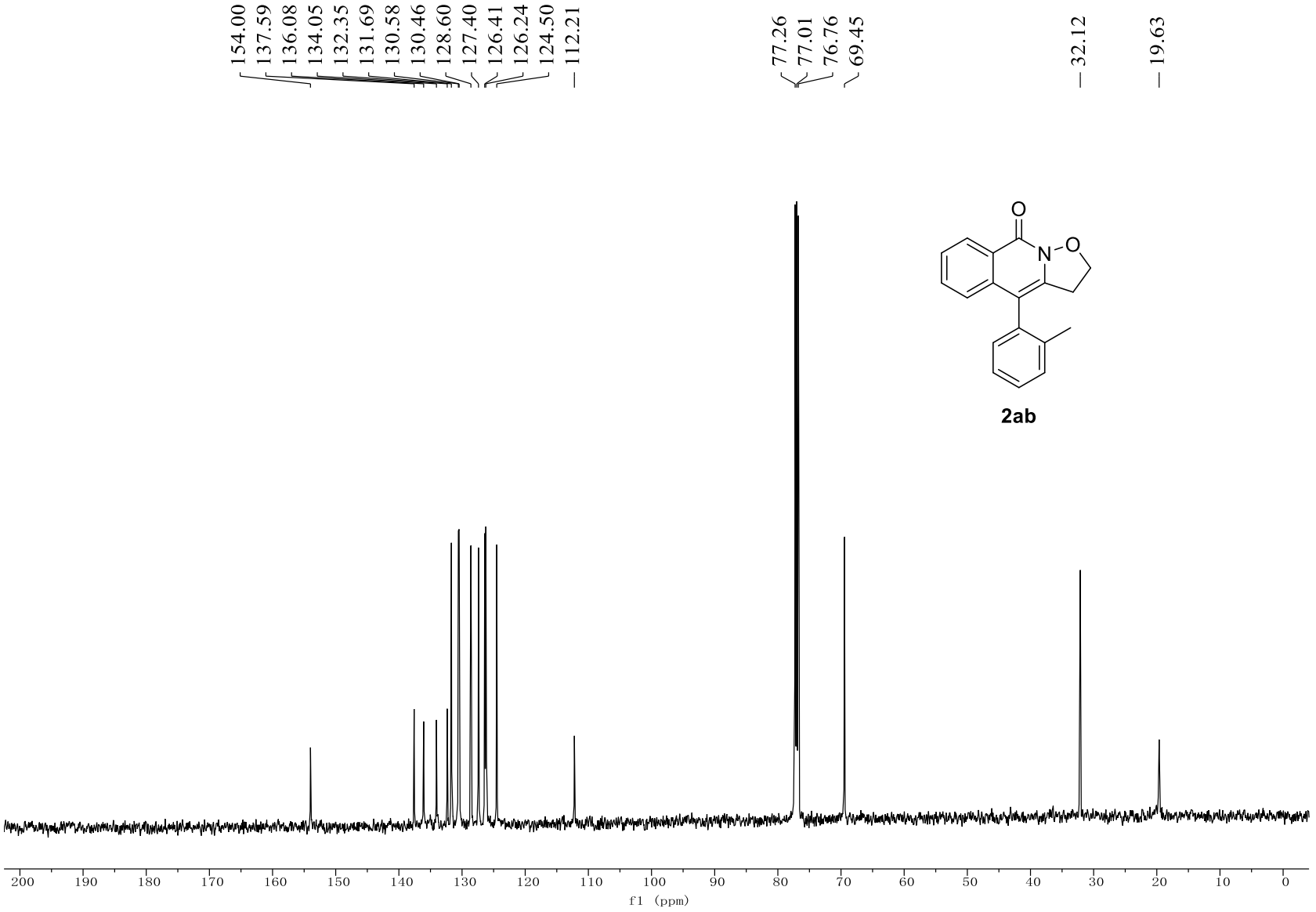

O
N
O

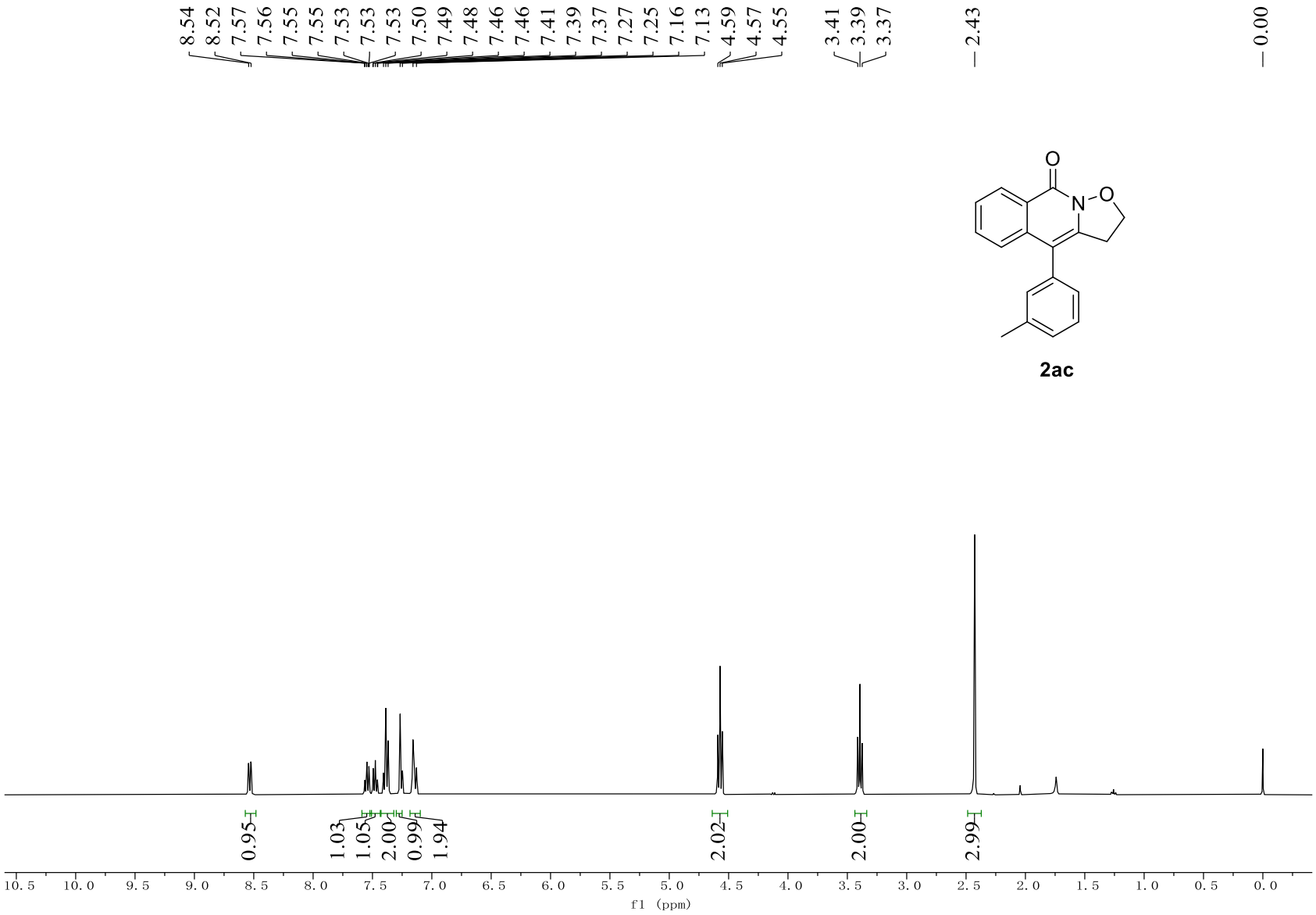
2aa

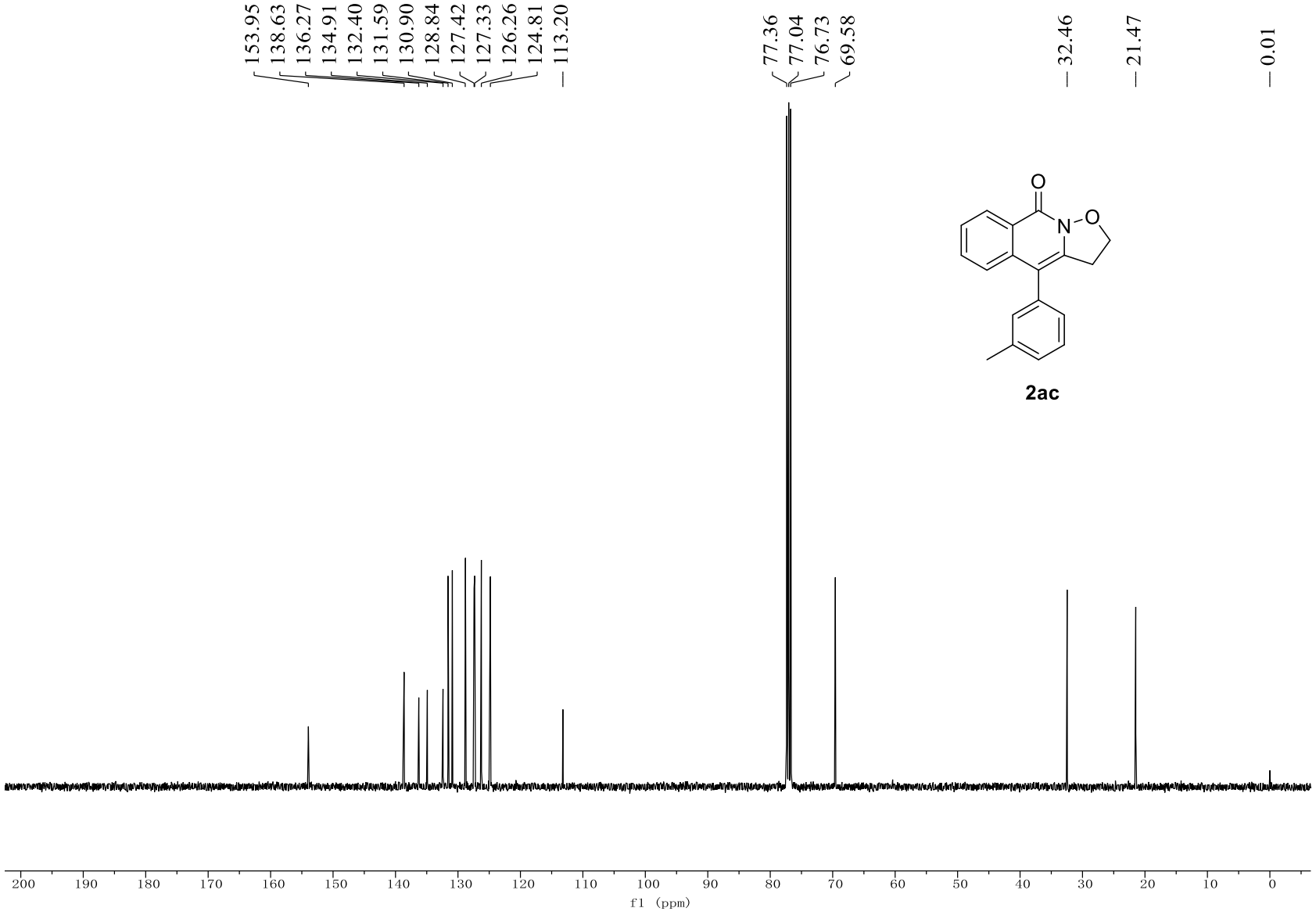

0.96
2.02
1.09
6.04
0.98
2.00
1.01
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

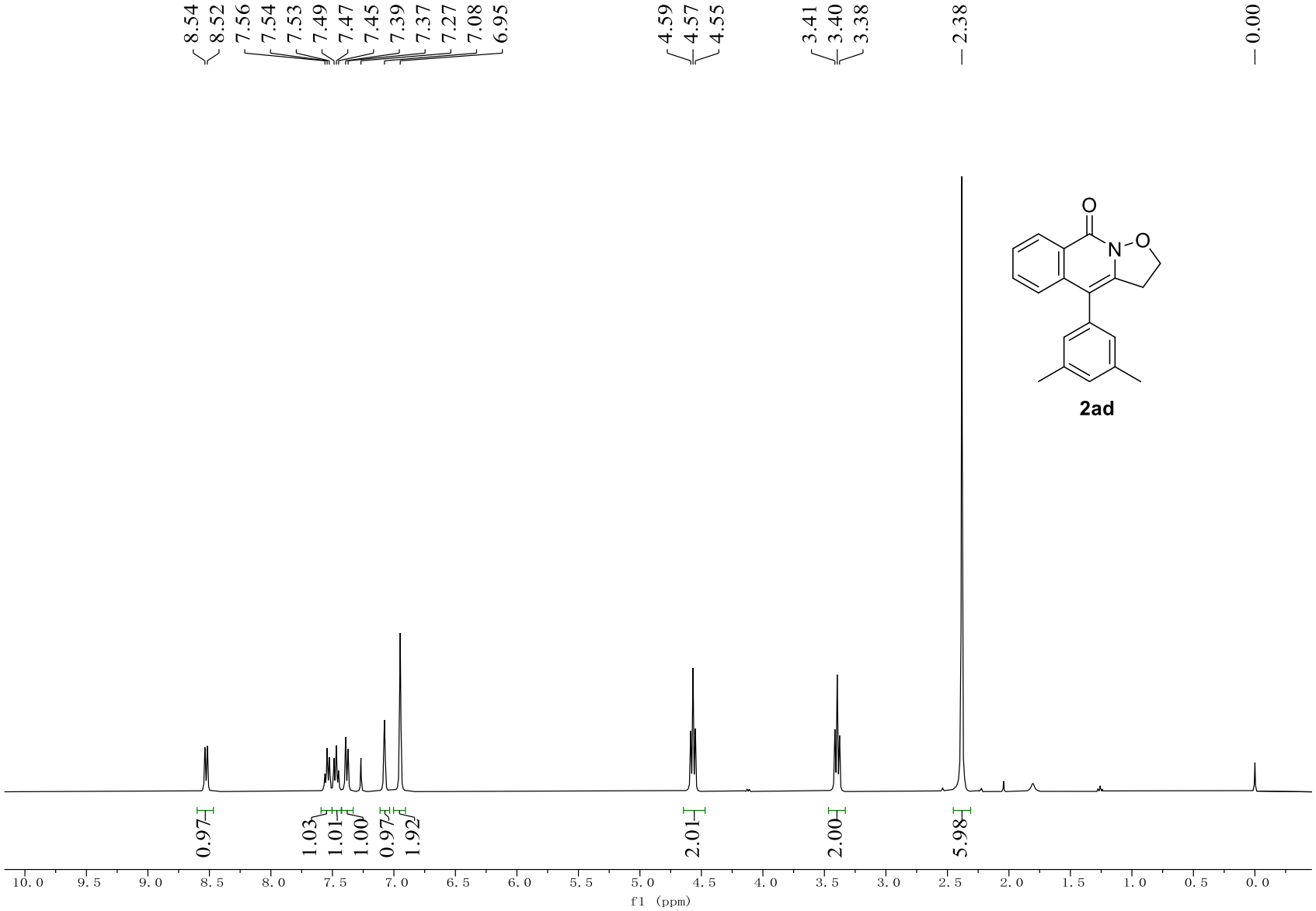

154.17
136.86
133.96
133.50
132.41
132.28
131.74
129.00
128.70
128.66
127.41
126.80
126.37
126.32
126.19
125.73
125.26
125.10
110.85
77.39
77.07
76.75
69.55
32.26
O
N
O
2aa
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

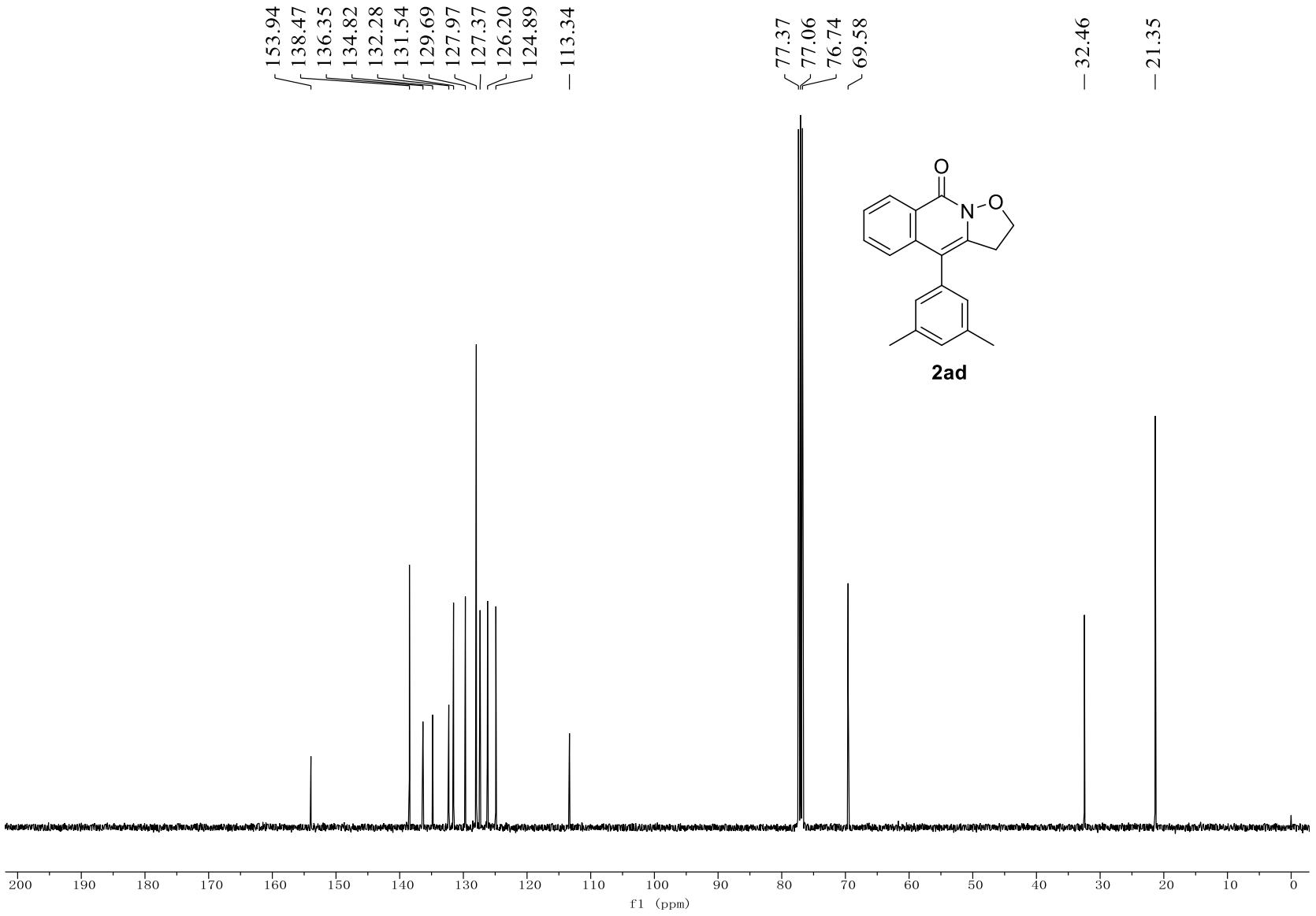

8.53
8.53
8.52
8.51
7.52
7.52
7.51
7.51
7.49
7.49
7.47
7.47
7.46
7.44
7.44
7.39
7.38
7.37
7.37
7.36
7.36
7.34
7.31
7.31
7.30
7.29
7.29
7.28
7.26
7.20
7.18
7.06
7.04
4.59
4.57
4.56
3.35
3.34
3.32
3.30
3.29
3.19
3.17
3.16
3.14
3.12
2.06
-0.01
O
N
O
2ab
0.95
2.05
2.01
1.01
0.97
0.96
1.98
1.01
1.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

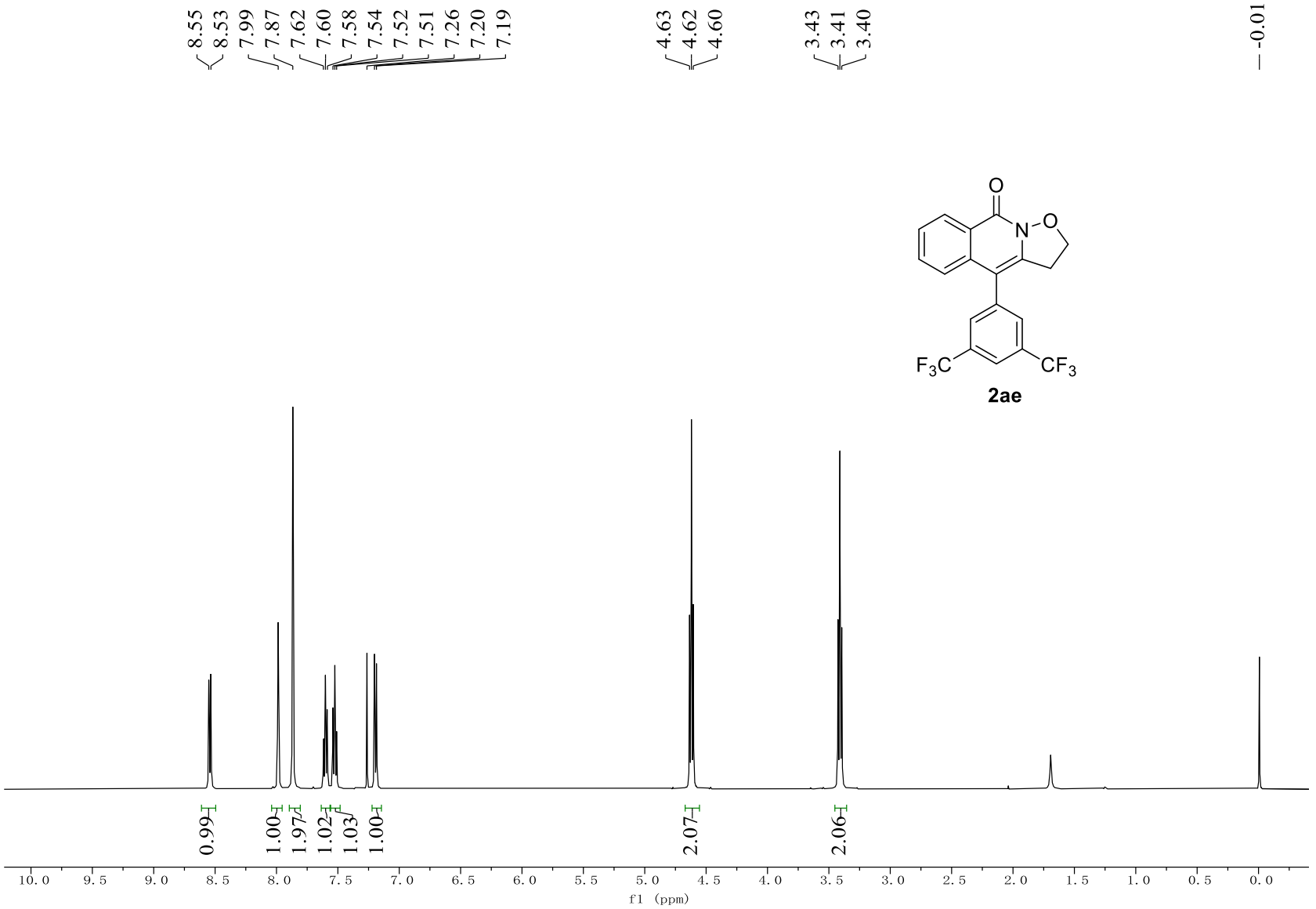

154.00
137.59
136.08
134.05
132.35
131.69
130.58
130.46
128.60
127.40
126.41
126.24
124.50
112.21
77.26
77.01
76.76
69.45
32.12
19.63
O
N
O
2ab
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

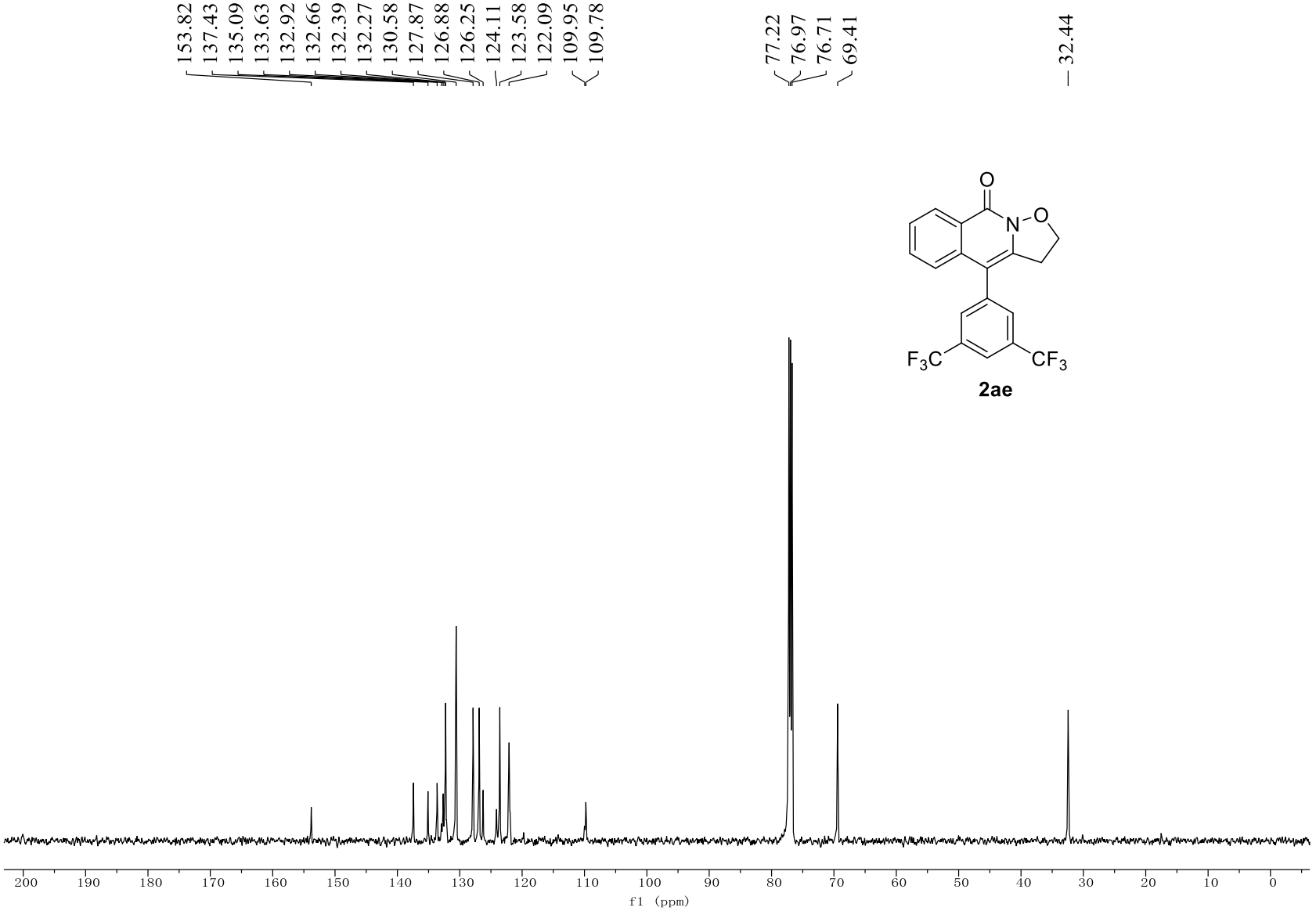

8.54
8.52
7.57
7.56
7.55
7.55
7.53
7.53
7.53
7.50
7.49
7.48
7.46
7.46
7.41
7.39
7.37
7.27
7.25
7.16
7.13
4.59
4.57
4.55
3.41
3.39
3.37
2.43
0.00
O
N
O
2ac
0.95
1.03
1.05
2.00
0.99
1.94
2.02
2.00
2.99
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

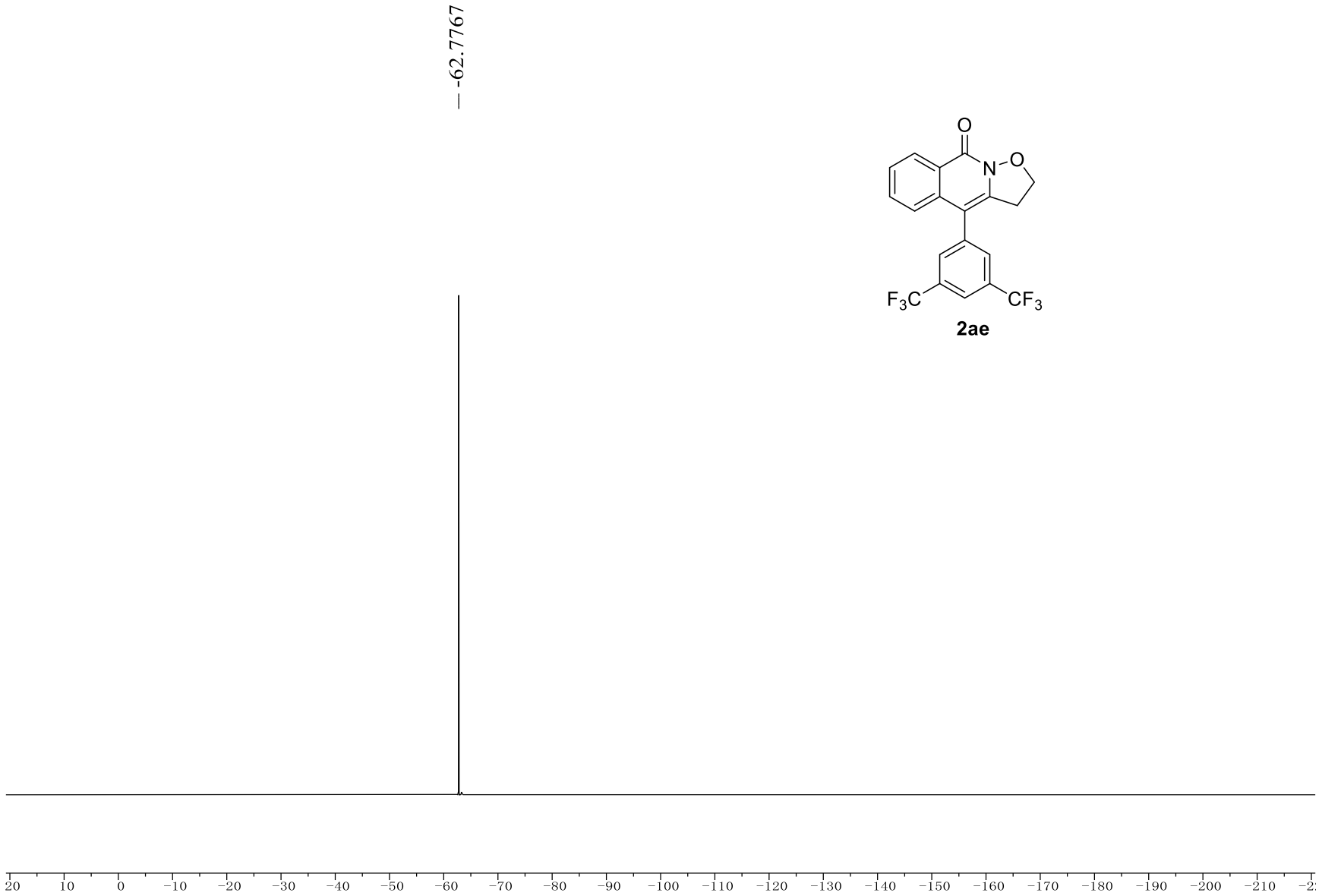

153.95
138.63
136.27
134.91
132.40
131.59
130.90
128.84
127.42
127.33
126.26
124.81
113.20
77.36
77.04
76.73
69.58
32.46
21.47
0.01
O
N
O
2ac
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

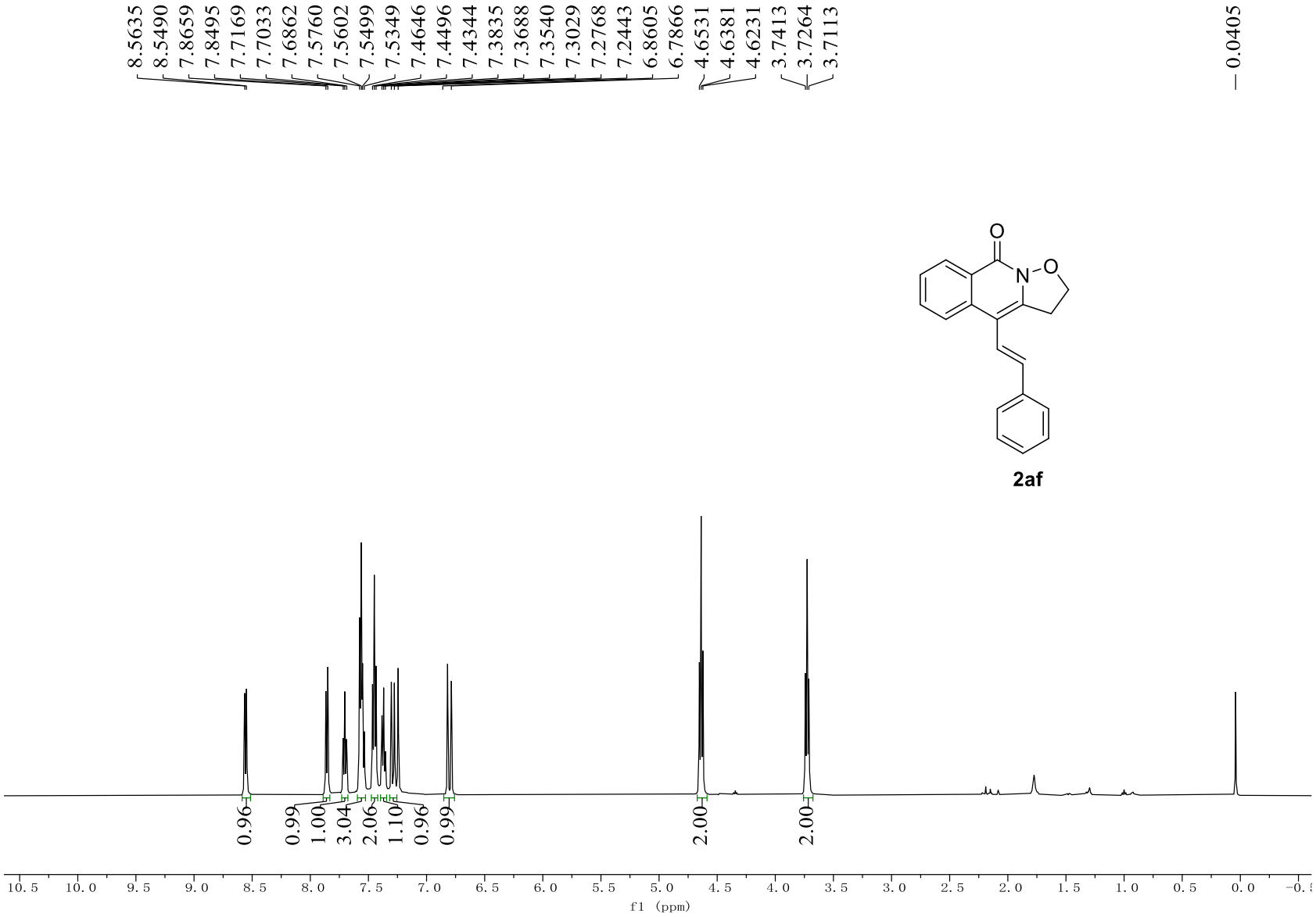

8.54
8.52
7.56
7.54
7.53
7.49
7.47
7.45
7.39
7.37
7.27
7.08
6.95
4.59
4.57
4.55
3.41
3.40
3.38
2.38
0.00
O
N
O
2ad
0.97
1.03
1.01
1.00
0.97
1.92
2.01
2.00
5.98
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

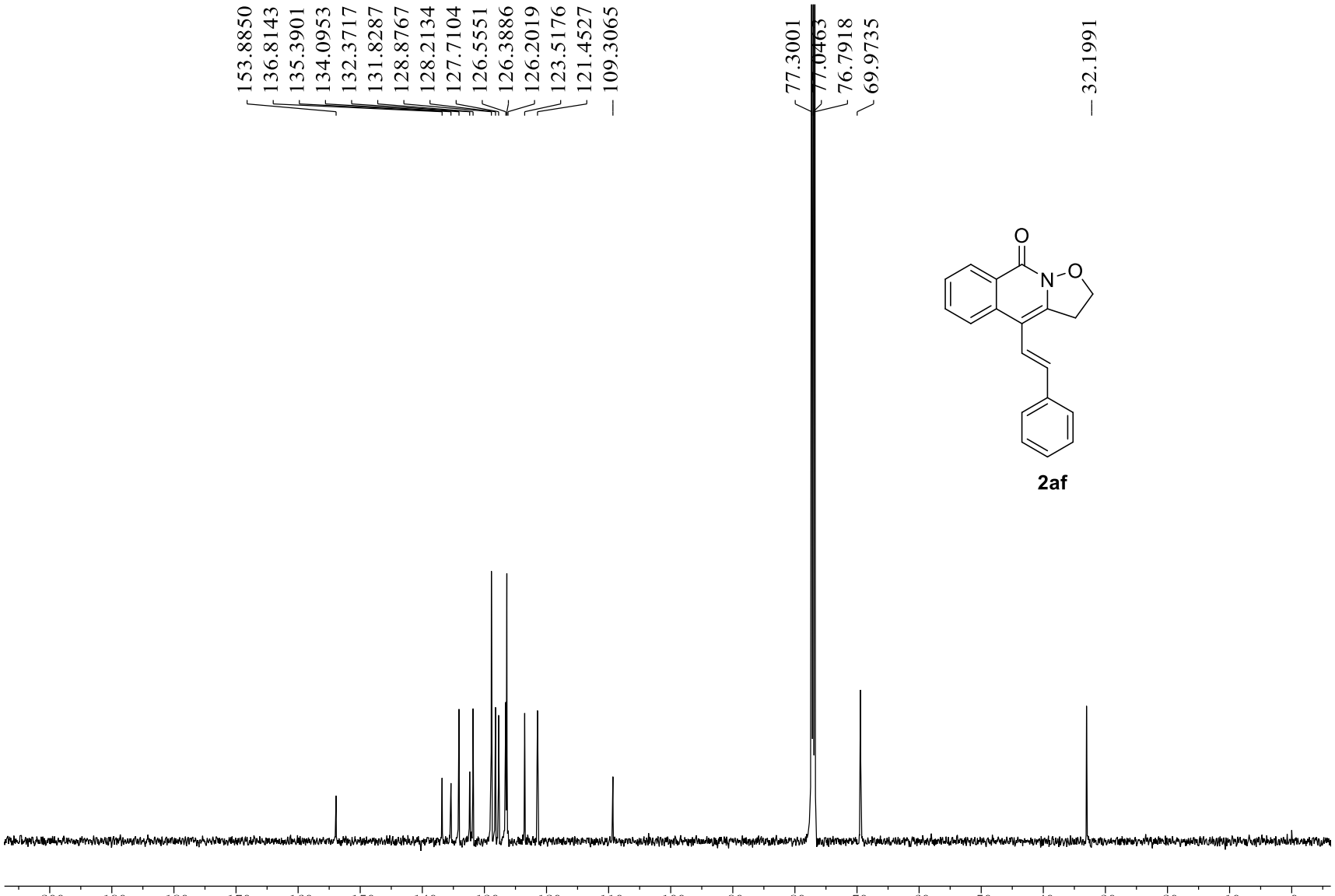

153.94
138.47
136.35
134.82
132.28
131.54
129.69
127.97
127.37
126.20
124.89
113.34
77.37
77.06
76.74
69.58
32.46
21.35
O
N
O
2ad
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

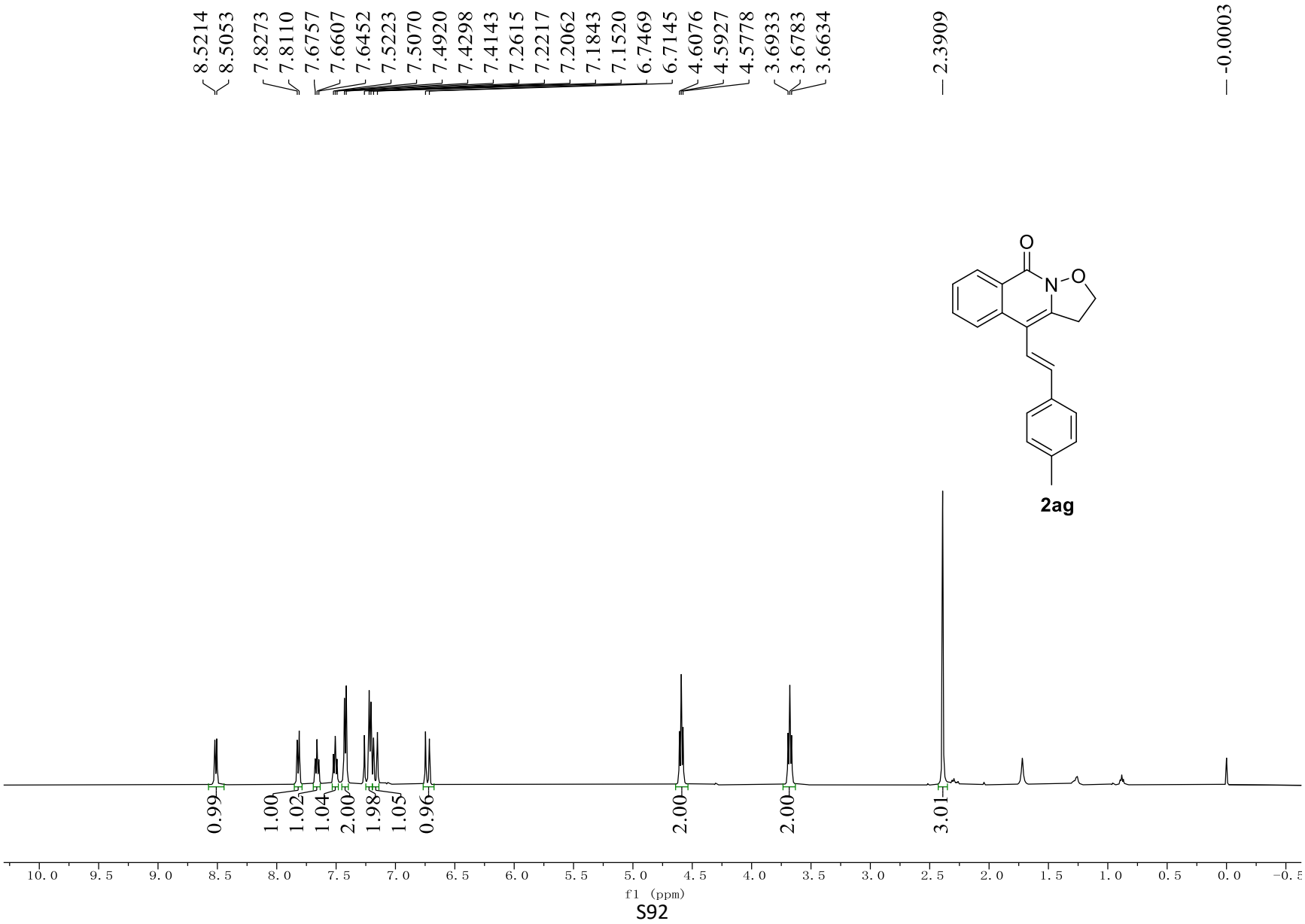

8.55
8.53
7.99
7.87
7.62
7.60
7.58
7.54
7.52
7.51
7.26
7.20
7.19
4.63
4.62
4.60
3.43
3.41
3.40
-0.01
O
N
O
F3C
CF3
2ae
0.99
1.00
1.97
1.02
1.03
1.00
2.07
2.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

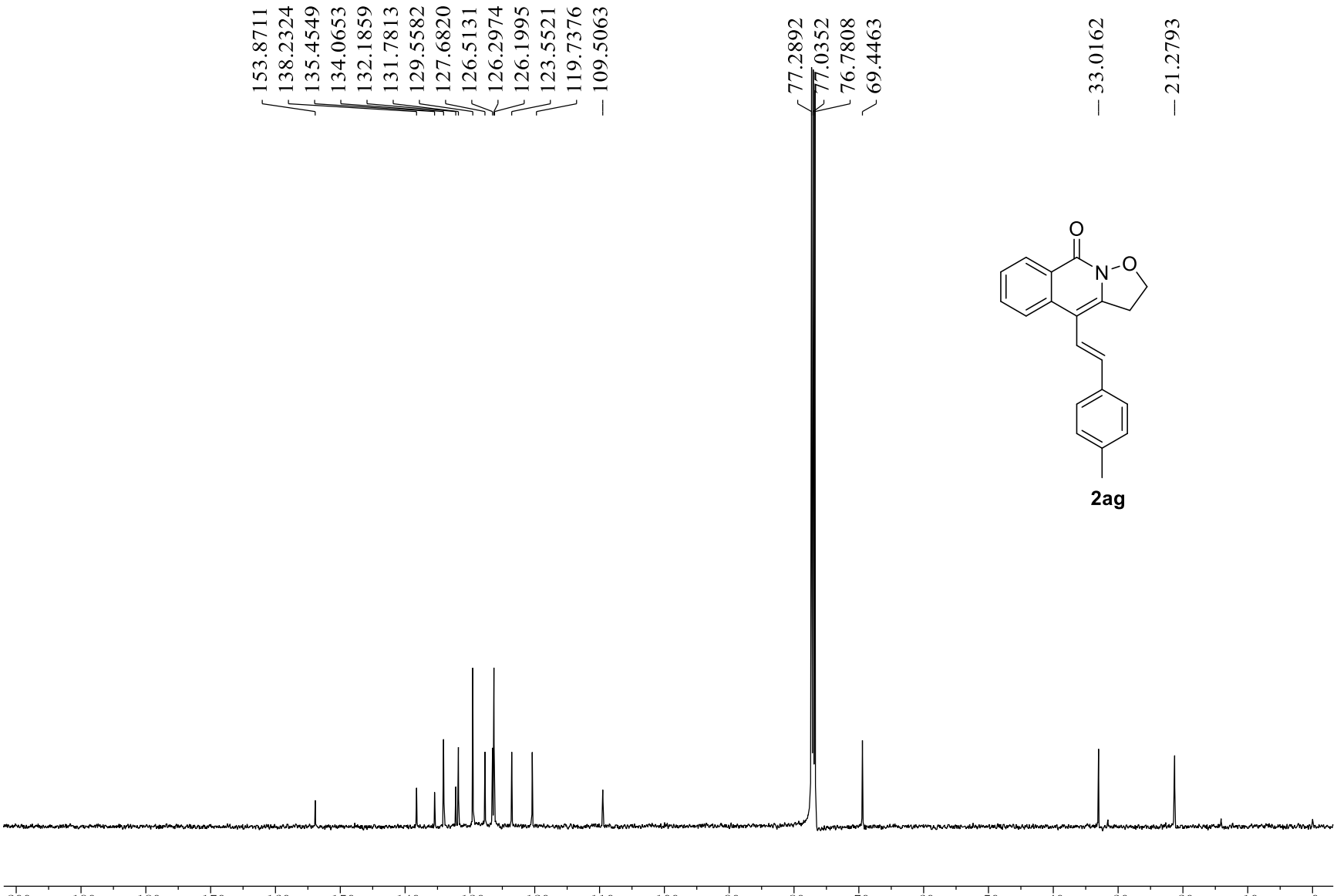

153.82
137.43
135.09
133.63
132.92
132.66
132.39
132.27
130.58
127.87
126.88
126.25
124.11
123.58
122.09
109.95
109.78
77.22
76.97
76.71
69.41
32.44
O
N
O
F3C
CF3
2ae
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

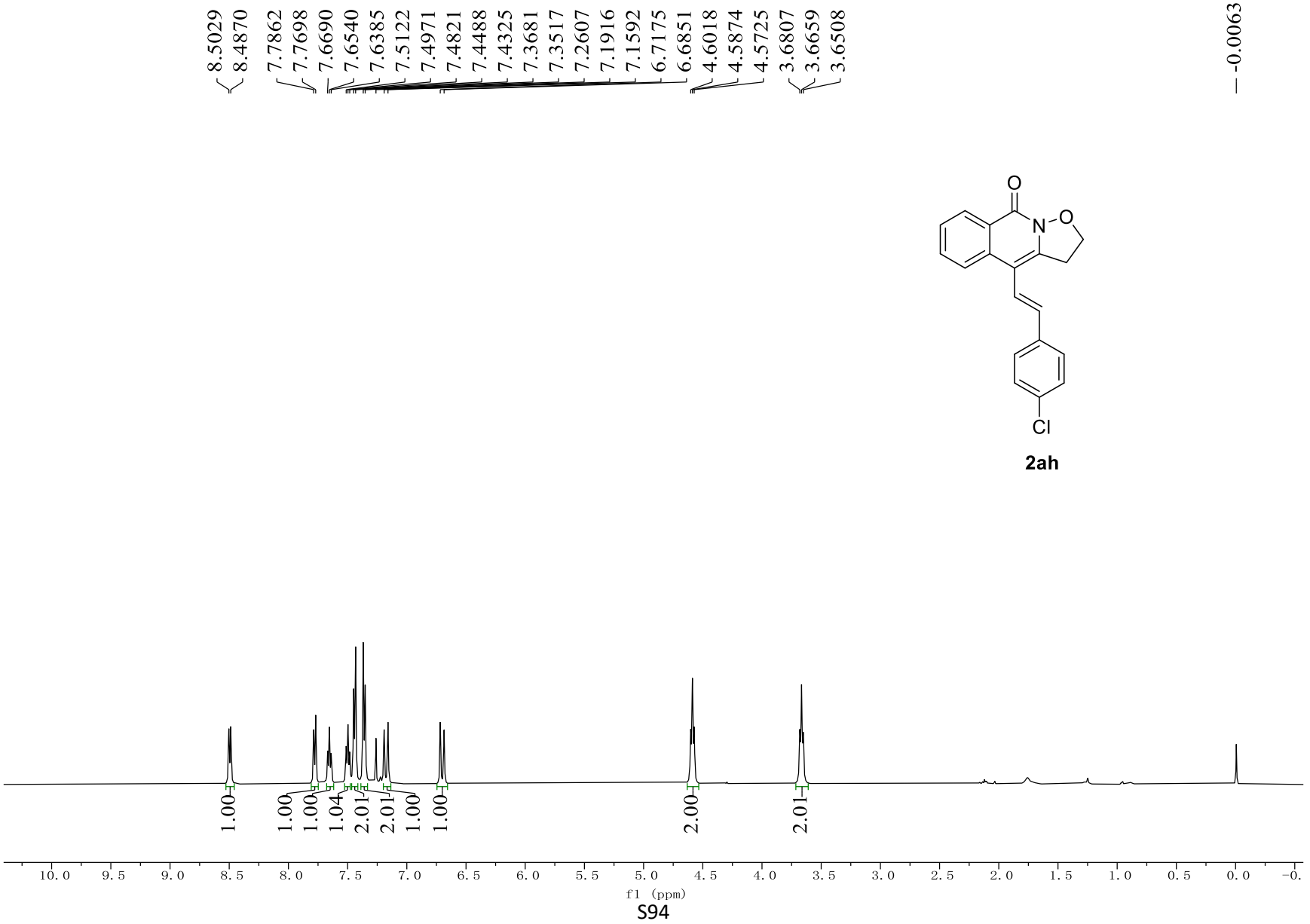

-62.7767
O
N
O
F3C
CF3
2ae
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

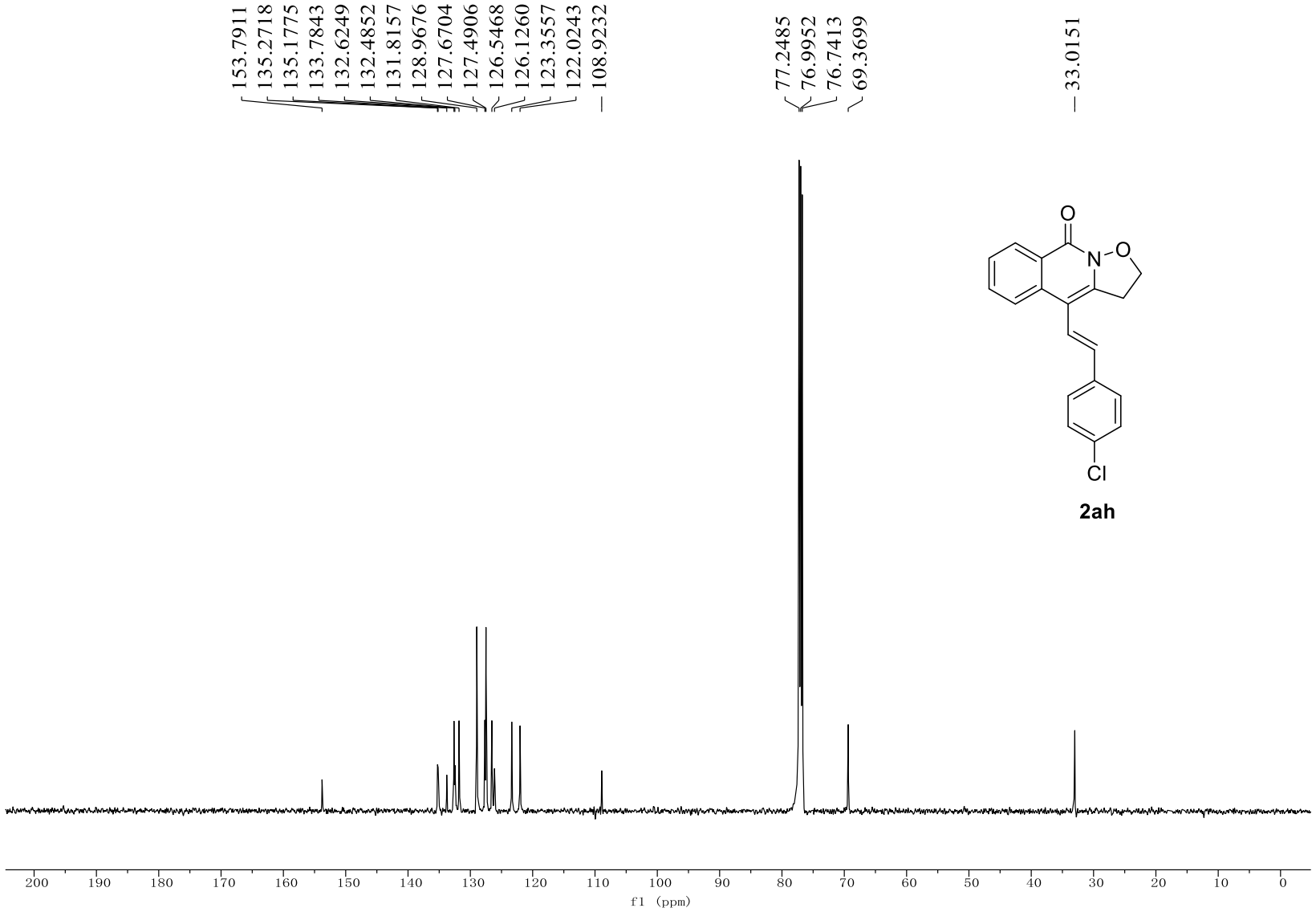

8.5635
8.5490
7.8659
7.8495
7.7169
7.7033
7.6862
7.5760
7.5602
7.5499
7.5349
7.4646
7.4496
7.4344
7.3835
7.3688
7.3540
7.3029
7.2768
7.2443
6.8605
6.7866
4.6531
4.6381
4.6231
3.7413
3.7264
3.7113
0.0405
O
N
O
2af
0.96
0.99
1.00
3.04
2.06
1.10
0.96
0.99
2.00
2.00
f1 (ppm)

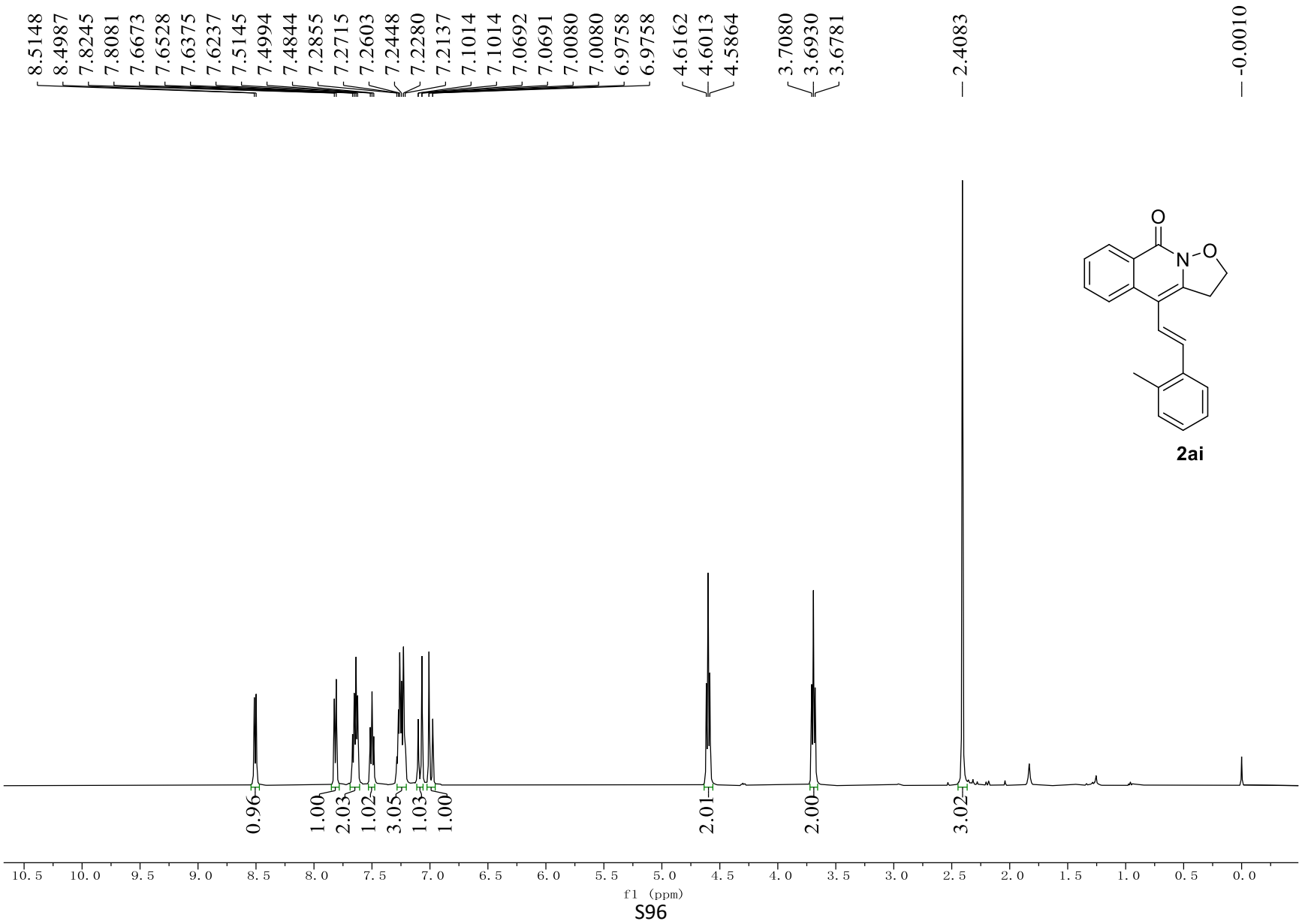

153.8850
136.8143
135.3901
134.0953
132.3717
131.8287
128.8767
128.2134
127.7104
126.5551
126.3886
126.2019
123.5176
121.4527
109.3065
77.3001
77.0463
76.7918
69.9735
32.1991
O
N
O
2af
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

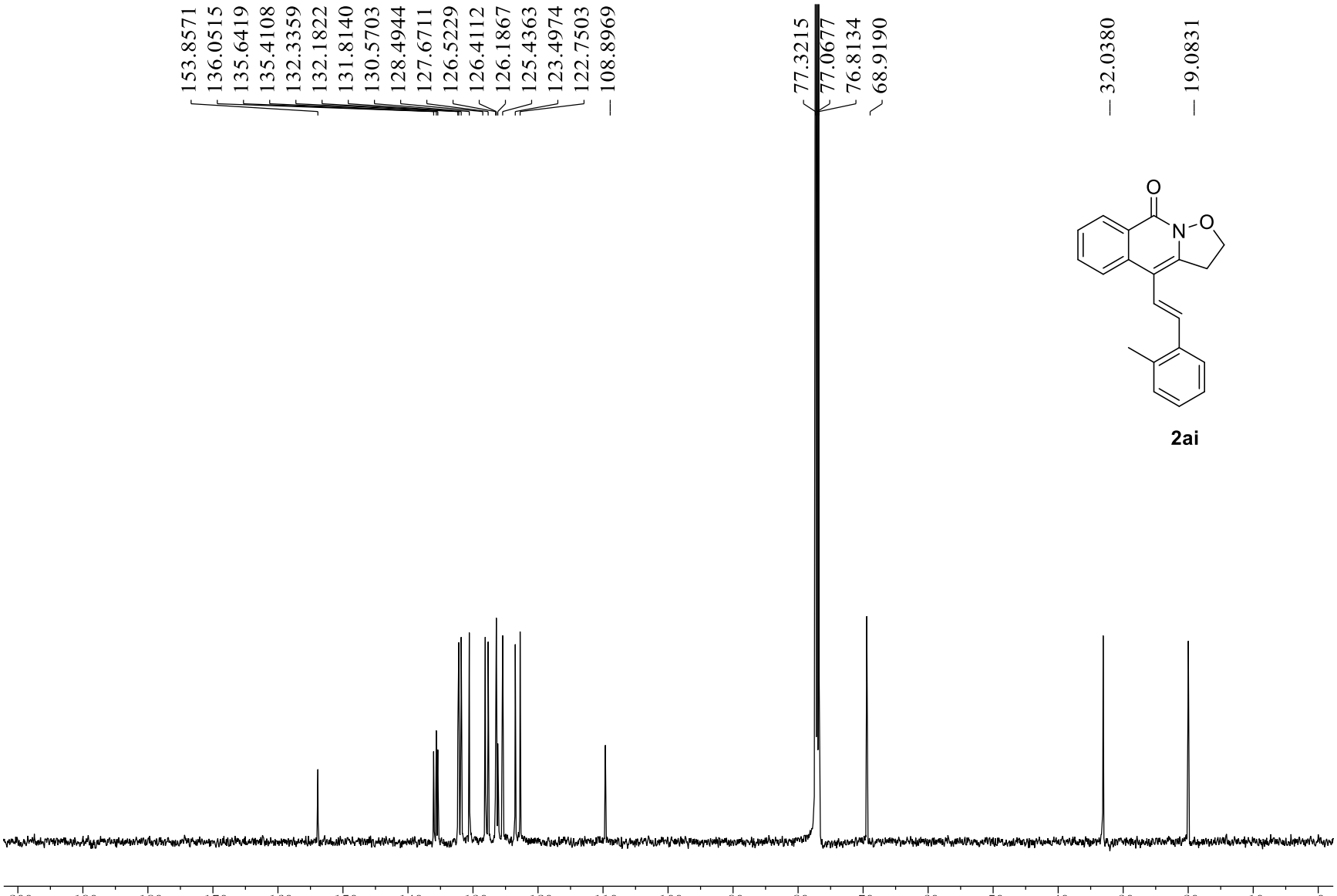

8.5214
8.5053
7.8273
7.8110
7.6757
7.6607
7.6452
7.5223
7.5070
7.4920
7.4298
7.4143
7.2615
7.2217
7.2062
7.1843
7.1520
6.7469
6.7145
4.6076
4.5927
4.5778
3.6933
3.6783
3.6634
2.3909
-0.0003
O
N
O
2ag
0.99
1.00
1.02
1.04
2.00
1.98
1.05
0.96
2.00
2.00
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

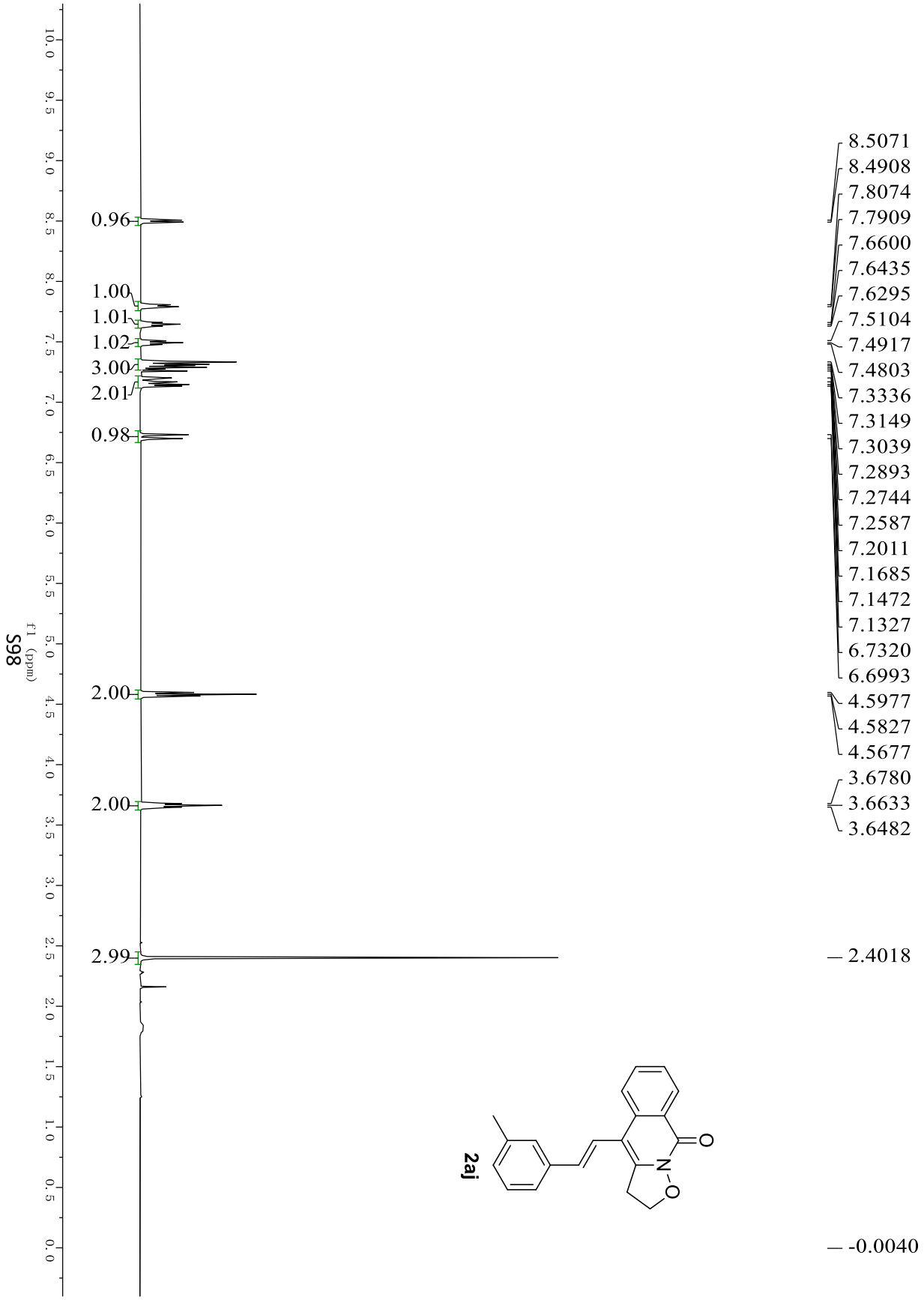

153.8711
138.2324
135.4549
134.0653
132.1859
131.7813
129.5582
127.6820
126.5131
126.2974
126.1995
123.5521
119.7376
109.5063
77.2892
77.0352
76.7808
69.4463
33.0162
21.2793
O
N
O
2ag
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

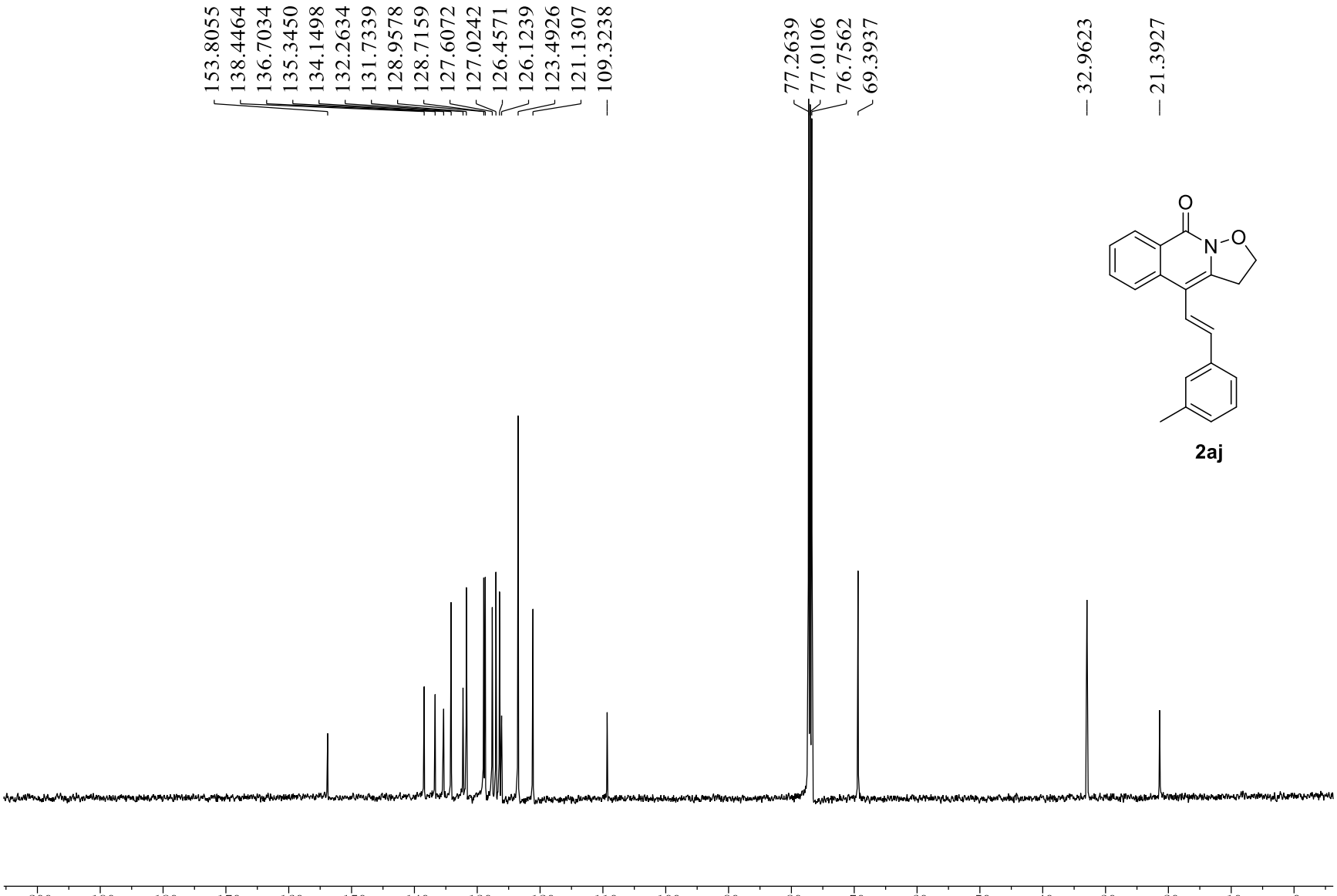

8.5029
8.4870
7.7862
7.7698
7.6690
7.6540
7.6385
7.5122
7.4971
7.4821
7.4488
7.4325
7.3681
7.3517
7.2607
7.1916
7.1592
6.7175
6.6851
4.6018
4.5874
4.5725
3.6807
3.6659
3.6508
-0.0063
O
N
O
Cl
2ah
1.00
1.00
1.00
1.04
2.01
2.01
1.00
1.00
2.00
2.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

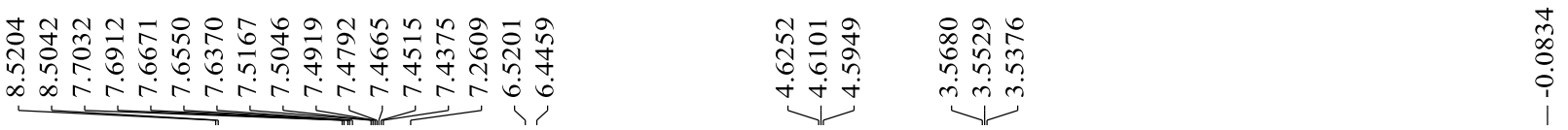

153.7911
135.2718
135.1775
133.7843
132.6249
132.4852
131.8157
128.9676
127.6704
127.4906
126.5468
126.1260
123.3557
122.0243
108.9232
77.2485
76.9952
76.7413
69.3699
33.0151
O
N
O
Cl
2ah
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

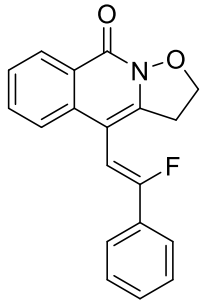

8.5148
8.4987
7.8245
7.8081
7.6673
7.6528
7.6375
7.6237
7.5145
7.4994
7.4844
7.2855
7.2715
7.2603
7.2448
7.2280
7.2137
7.1014
7.1014
7.0692
7.0691
7.0080
7.0080
6.9758
6.9758
4.6162
4.6013
4.5864
3.7080
3.6930
3.6781
2.4083
-0.0010
O
N
O
2ai
0.96
1.00
2.03
1.02
3.05
1.03
1.00
2.01
2.00
3.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.8571
136.0515
135.6419
135.4108
132.3359
132.1822
131.8140
130.5703
128.4944
127.6711
126.5229
126.4112
126.1867
125.4363
123.4974
122.7503
108.8969
77.3215
77.0677
76.8134
68.9190
32.0380
19.0831
O
N
O
2ai
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

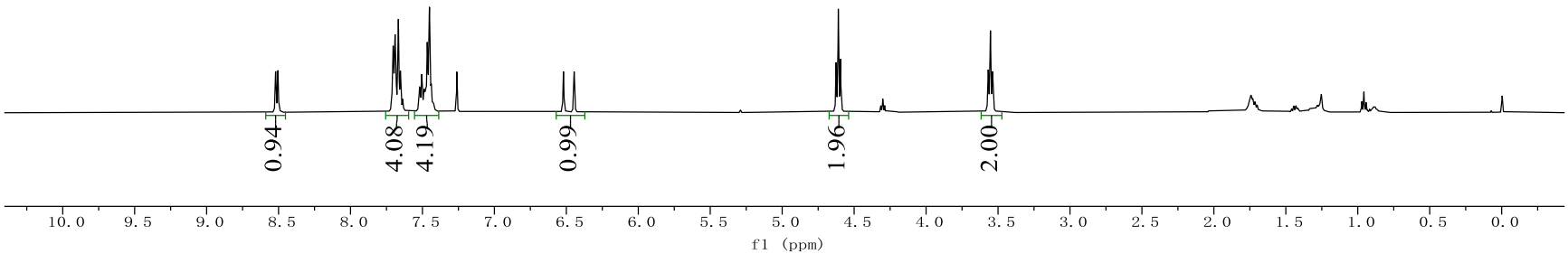

S98
f1 (ppm)
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0.96
1.00
1.01
1.02
3.00
2.01
0.98
2.00
2.00
2.99
8.5071
8.4908
7.8074
7.7909
7.6600
7.6435
7.6295
7.5104
7.4917
7.4803
7.3336
7.3149
7.3039
7.2893
7.2744
7.2587
7.2011
7.1685
7.1472
7.1327
6.7320
6.6993
4.5977
4.5827
4.5677
3.6780
3.6633
3.6482
2.4018
-0.0040
2aj
O
N
O

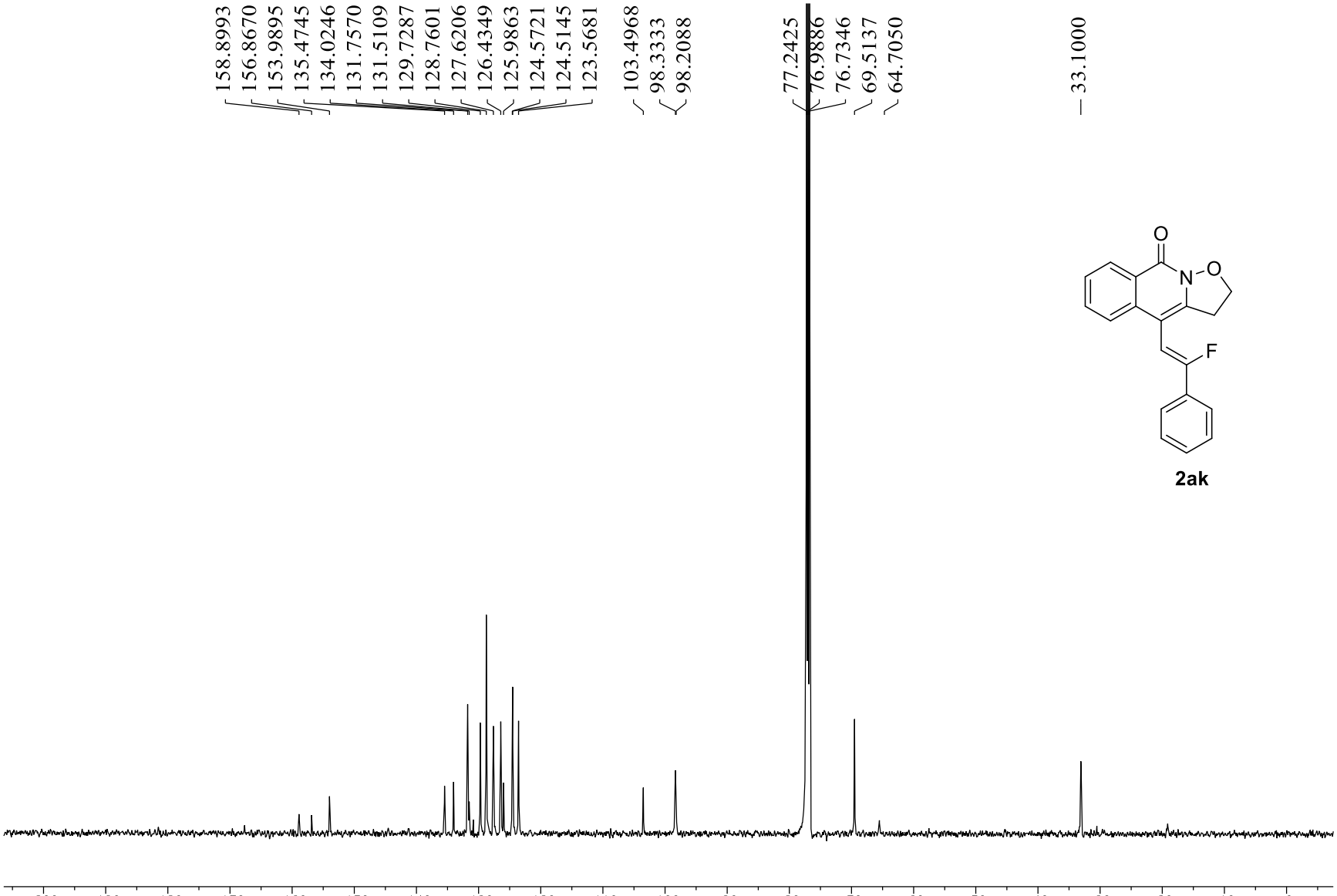

153.8055
138.4464
136.7034
135.3450
134.1498
132.2634
131.7339
128.9578
128.7159
127.6072
127.0242
126.4571
126.1239
123.4926
121.1307
109.3238
77.2639
77.0106
76.7562
69.3937
32.9623
21.3927
O
N
O
2aj
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.5204
8.5042
7.7032
7.6912
7.6671
7.6550
7.6370
7.5167
7.5046
7.4919
7.4792
7.4665
7.4515
7.4375
7.2609
6.5201
6.4459
4.6252
4.6101
4.5949
3.5680
3.5529
3.5376
-0.0834

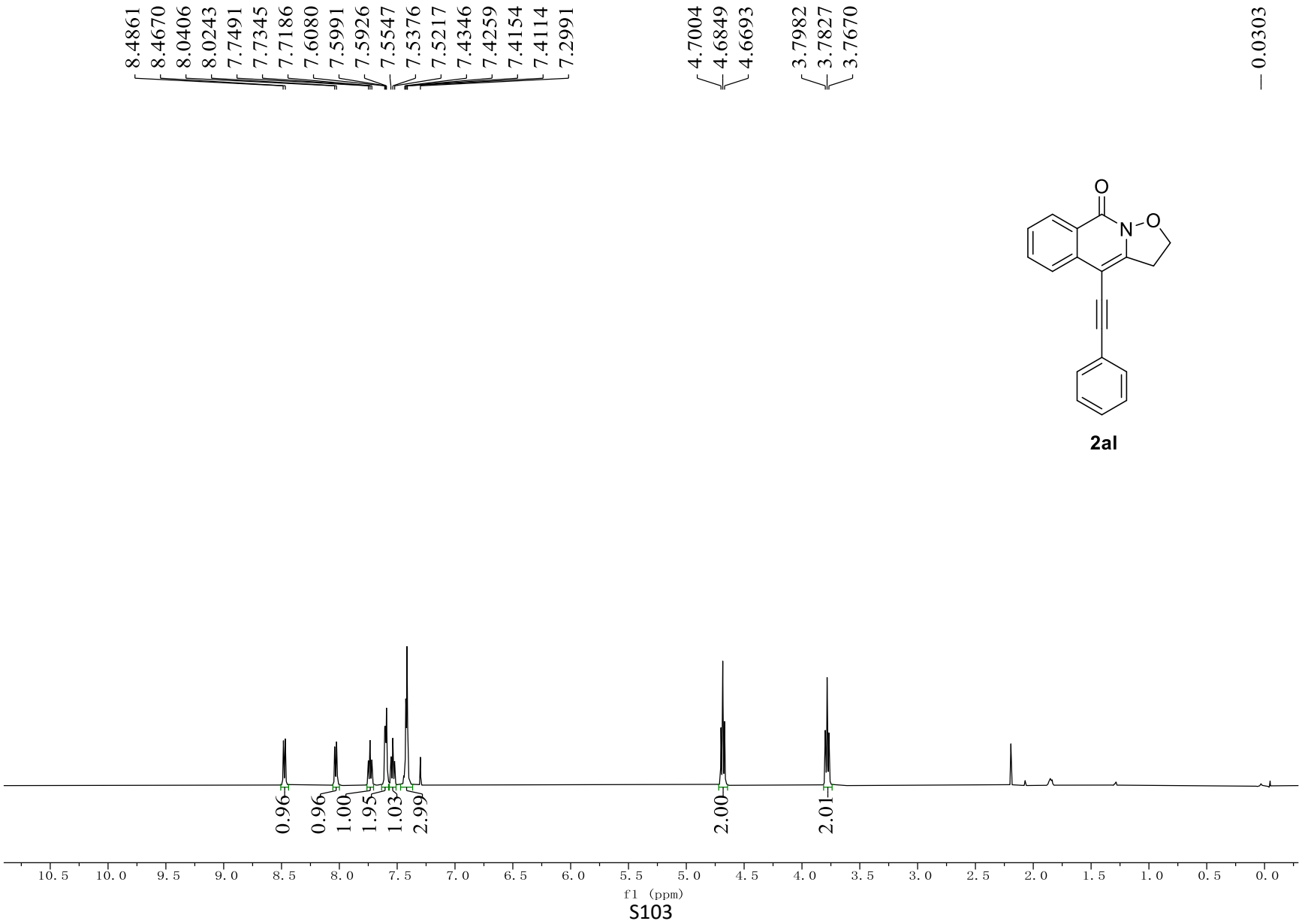

O
N
O
F

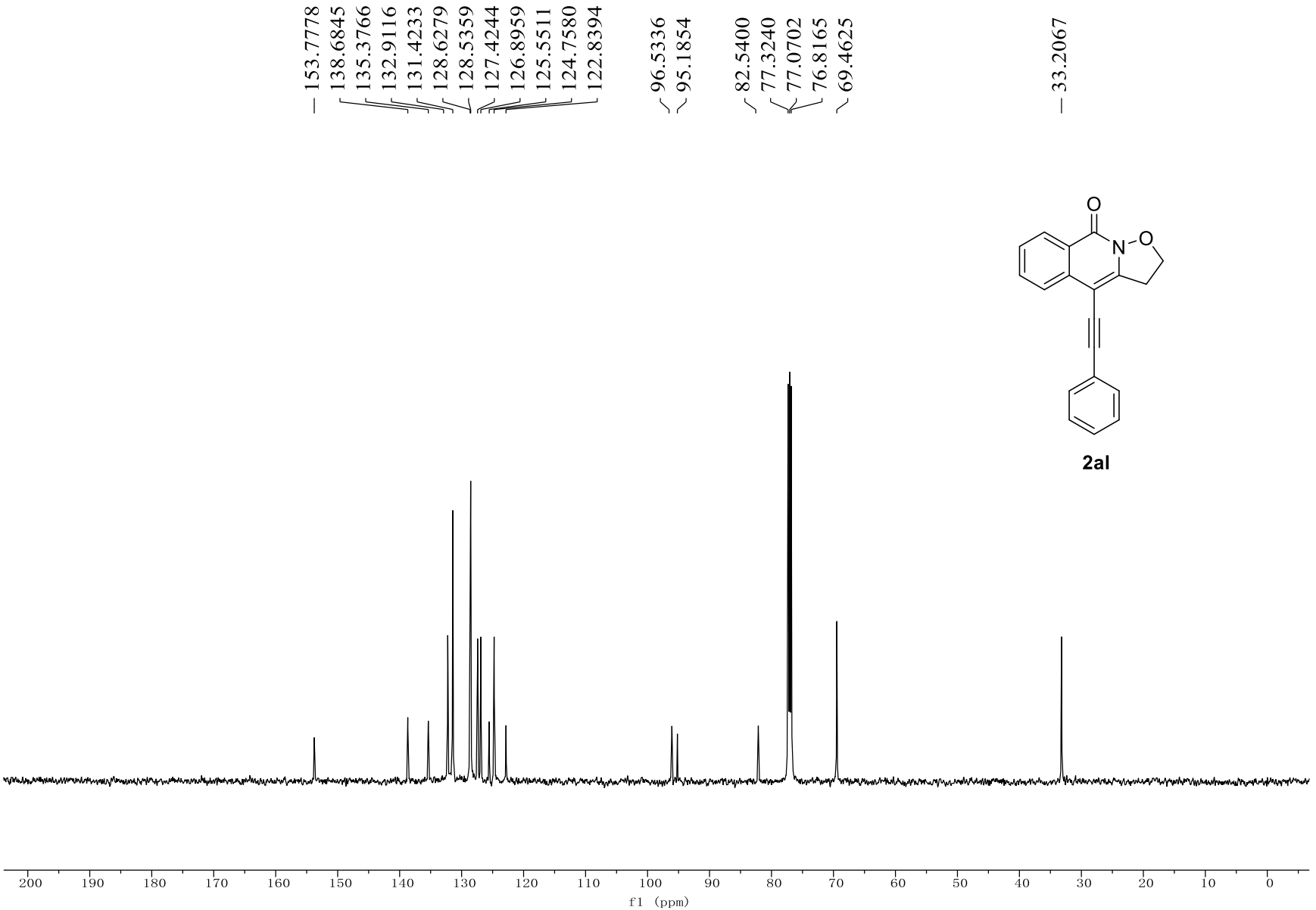
2ak

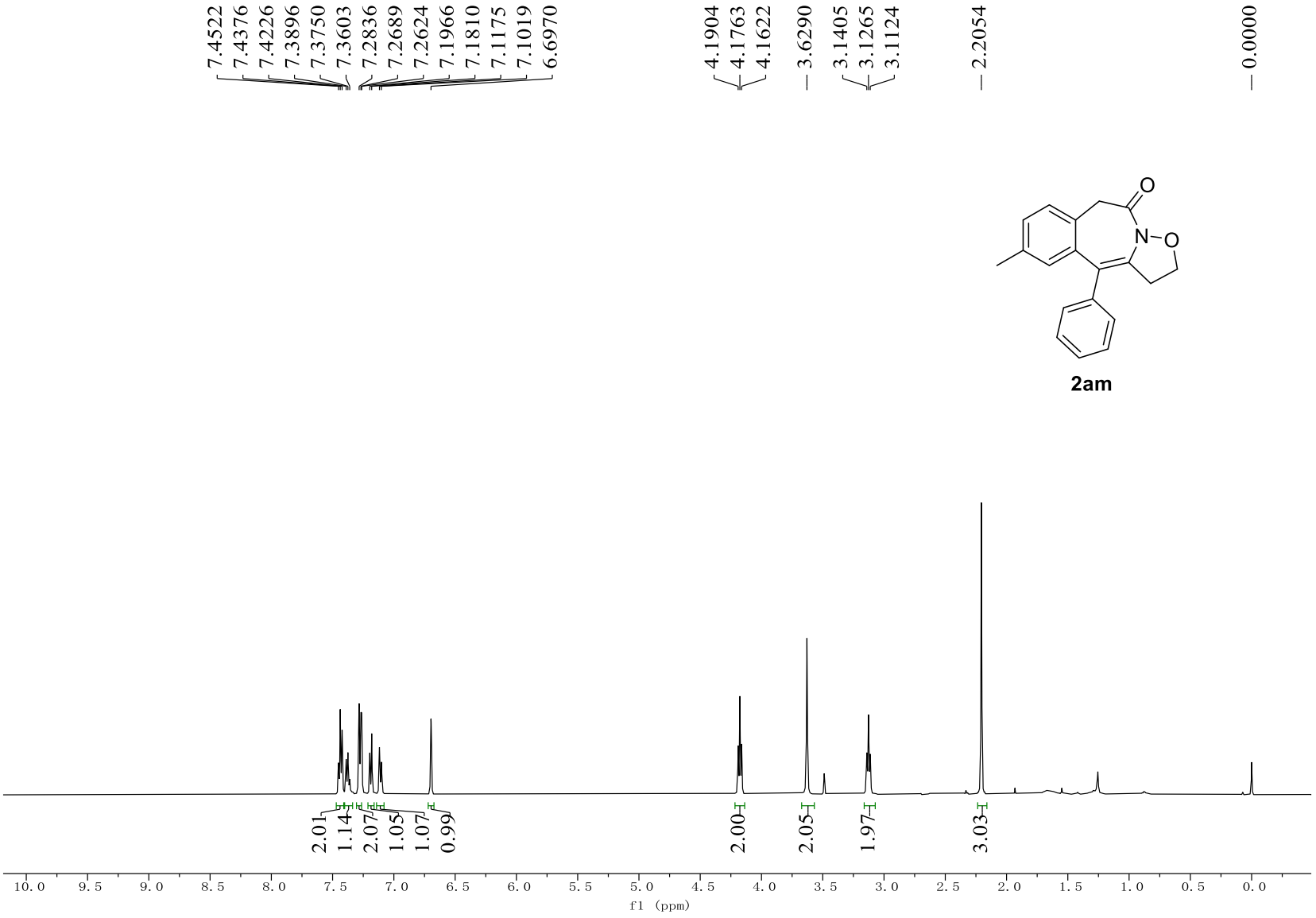

0.94
4.08
4.19
0.99
1.96
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

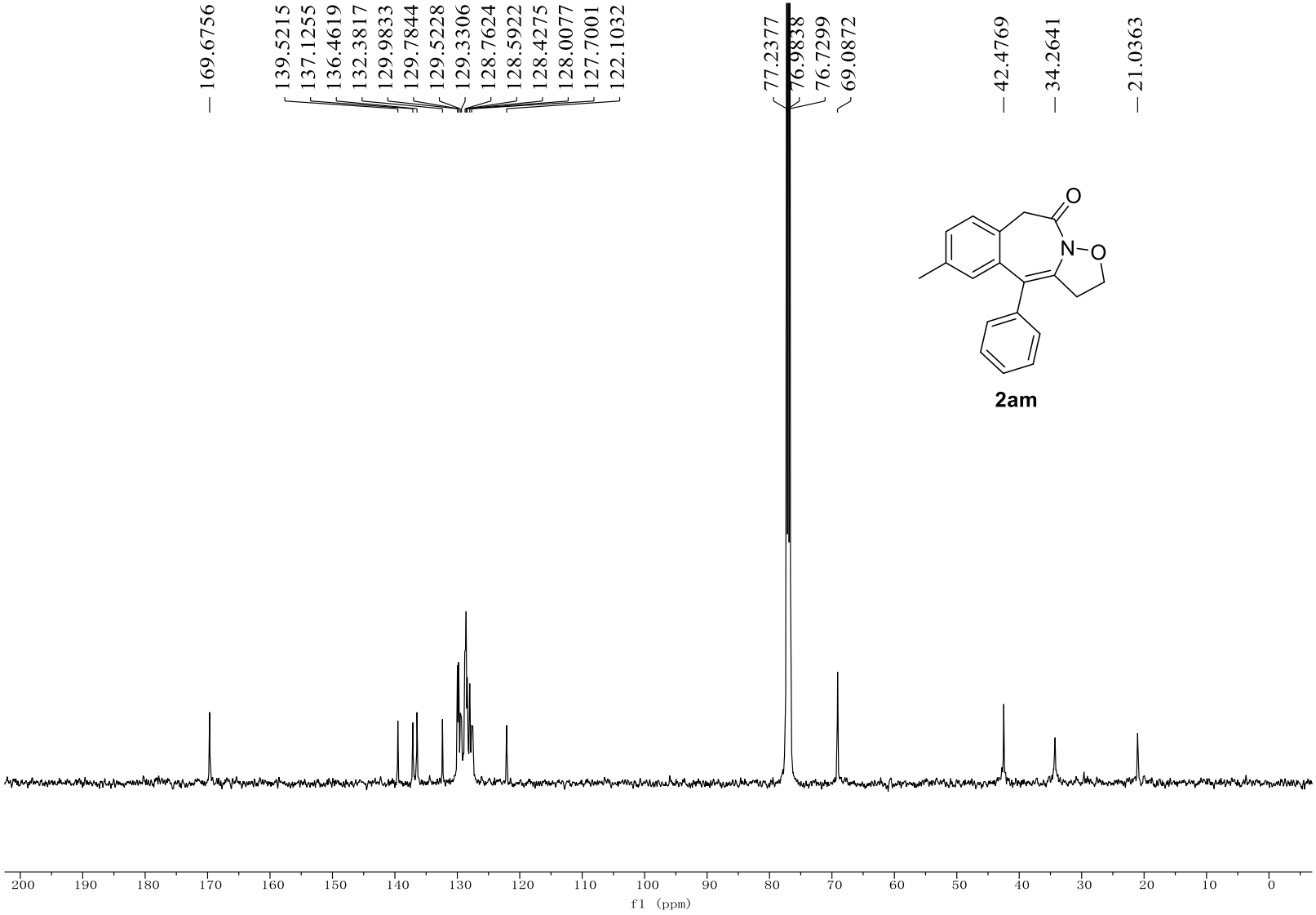

158.8993
156.8670
153.9895
135.4745
134.0246
131.7570
131.5109
129.7287
128.7601
127.6206
126.4349
125.9863
124.5721
124.5145
123.5681
103.4968
98.3333
98.2088
77.2425
76.9886
76.7346
69.5137
64.7050
33.1000
O
N
O
F
2ak
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

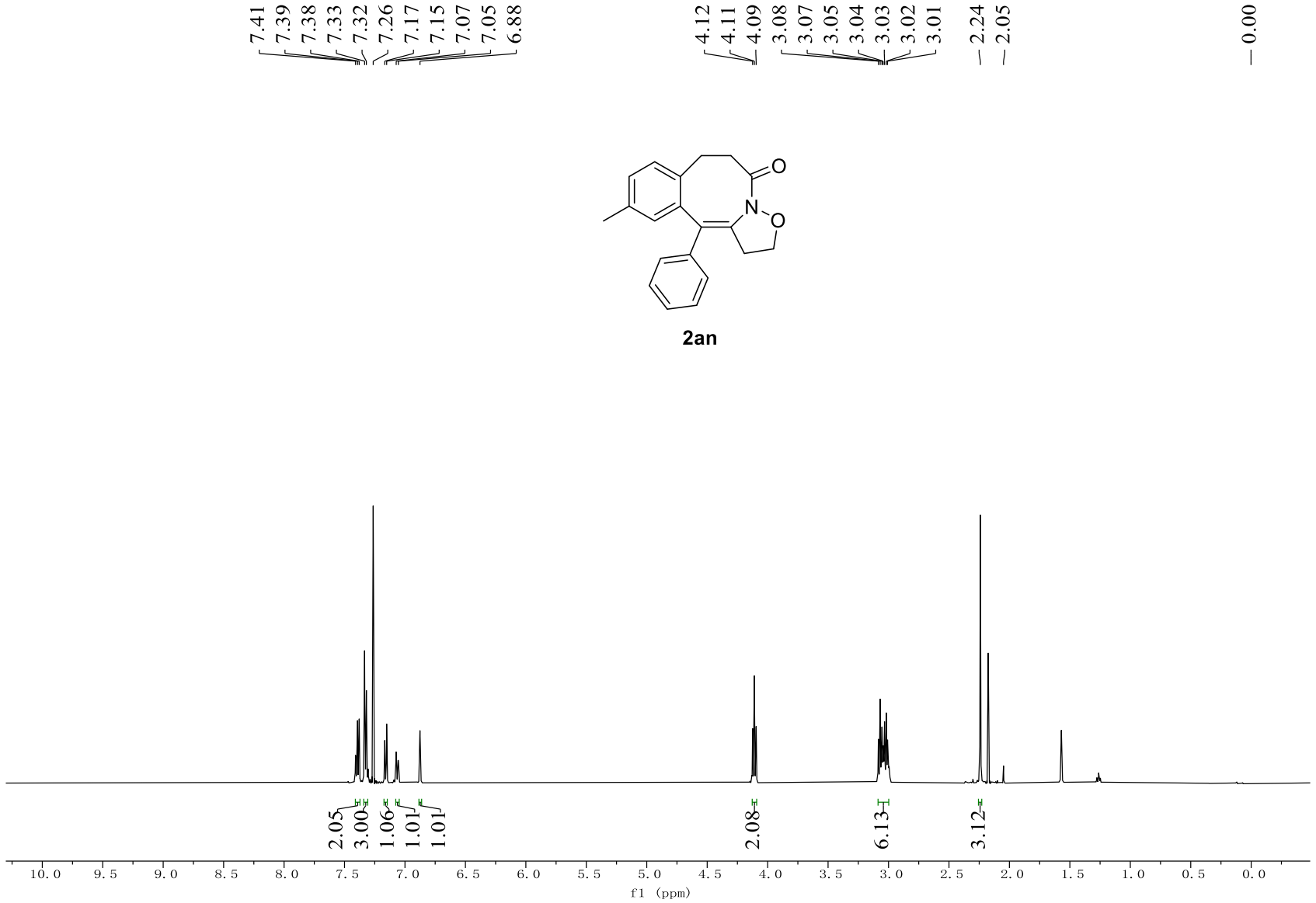

-109.2654
O
N
O
F
2ak
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)

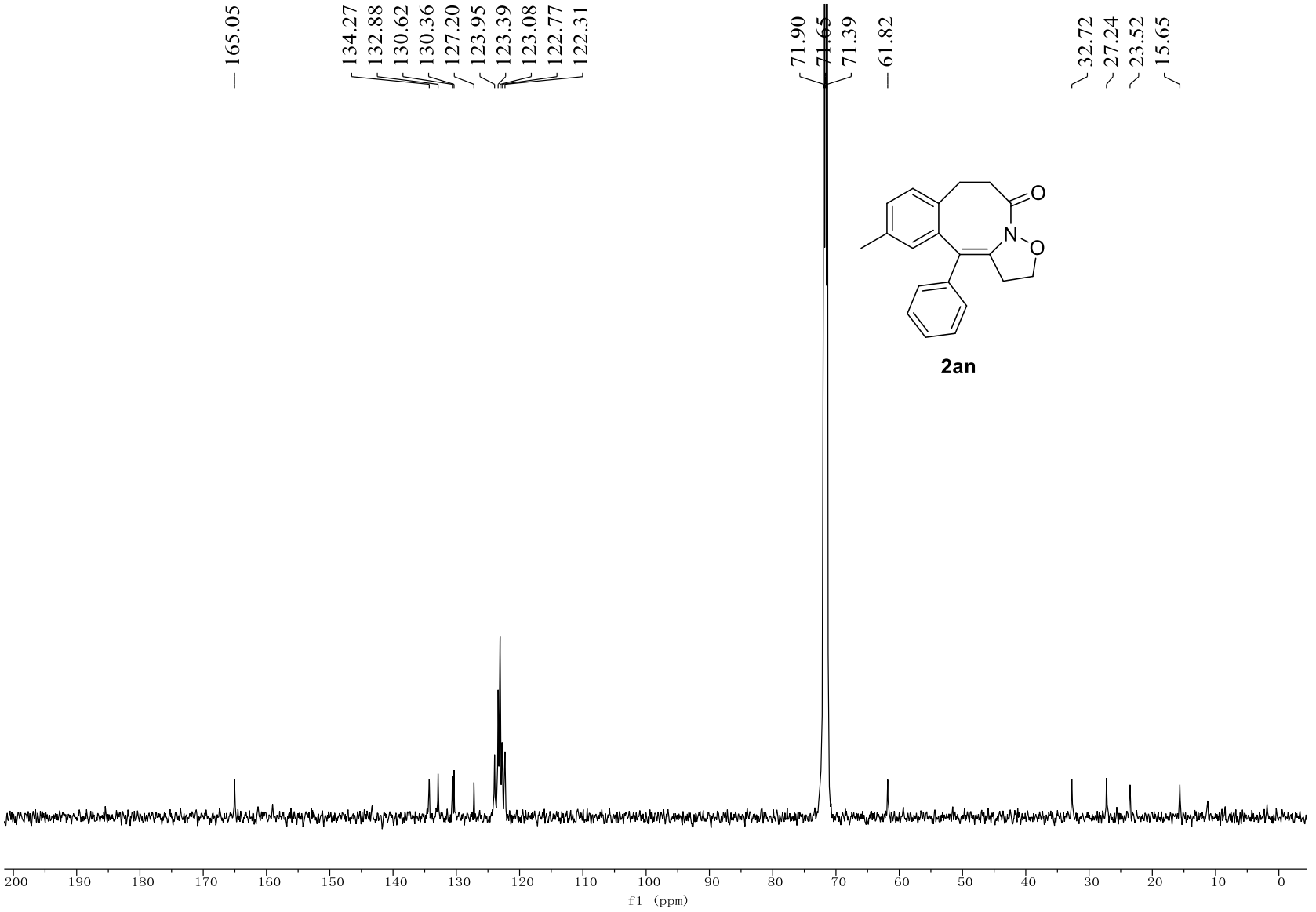

8.4861
8.4670
8.0406
8.0243
7.7491
7.7345
7.7186
7.6080
7.5991
7.5926
7.5547
7.5376
7.5217
7.4346
7.4259
7.4154
7.4114
7.2991
4.7004
4.6849
4.6693
3.7982
3.7827
3.7670
0.0303
O
N
O
2al
0.96
0.96
1.00
1.95
1.03
2.99
2.00
2.01
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

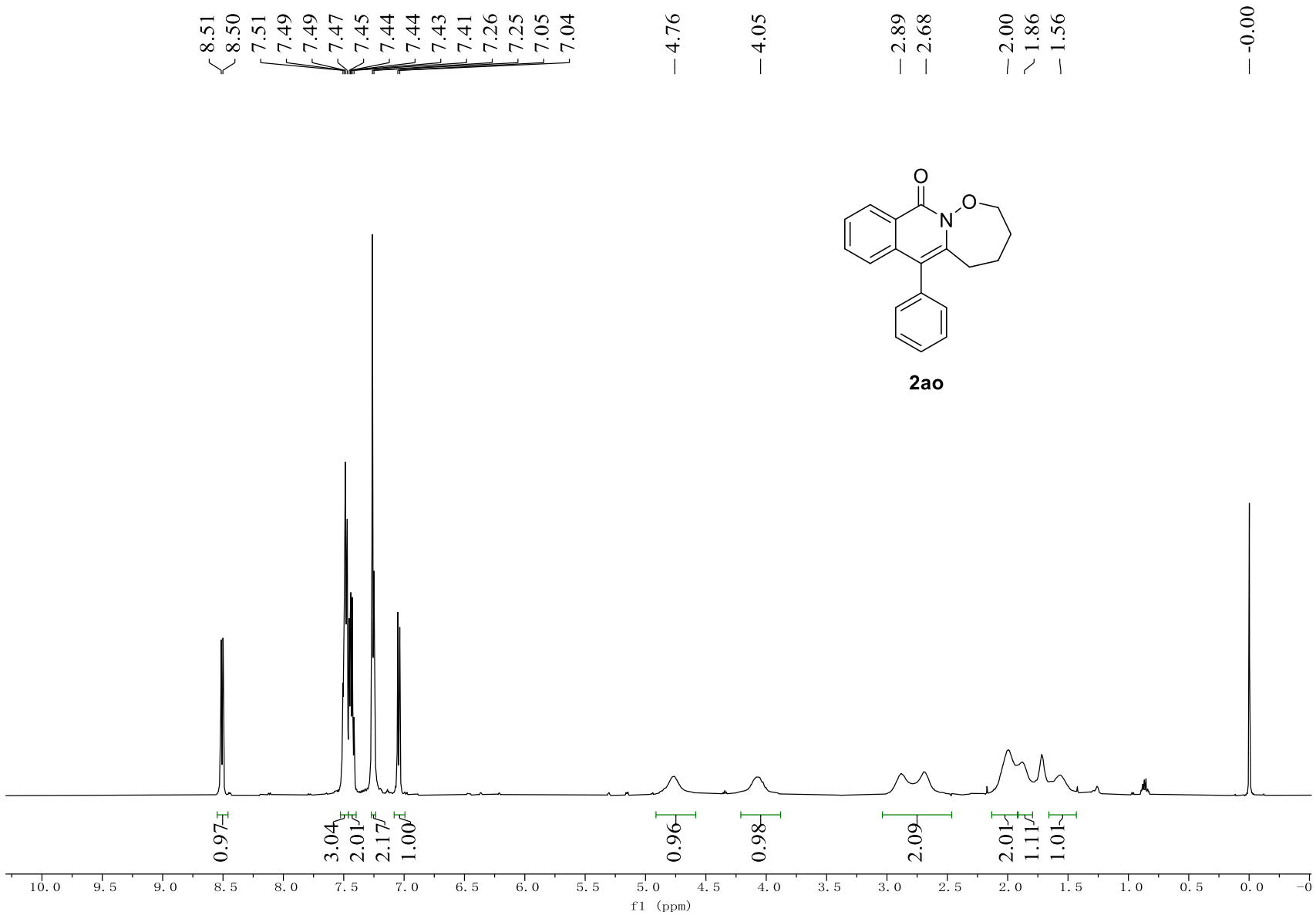

153.7778
138.6845
135.3766
132.9116
131.4233
128.6279
128.5359
127.4244
126.8959
125.5511
124.7580
122.8394
96.5336
95.1854
82.5400
77.3240
77.0702
76.8165
69.4625
33.2067
O
N
O
2al
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

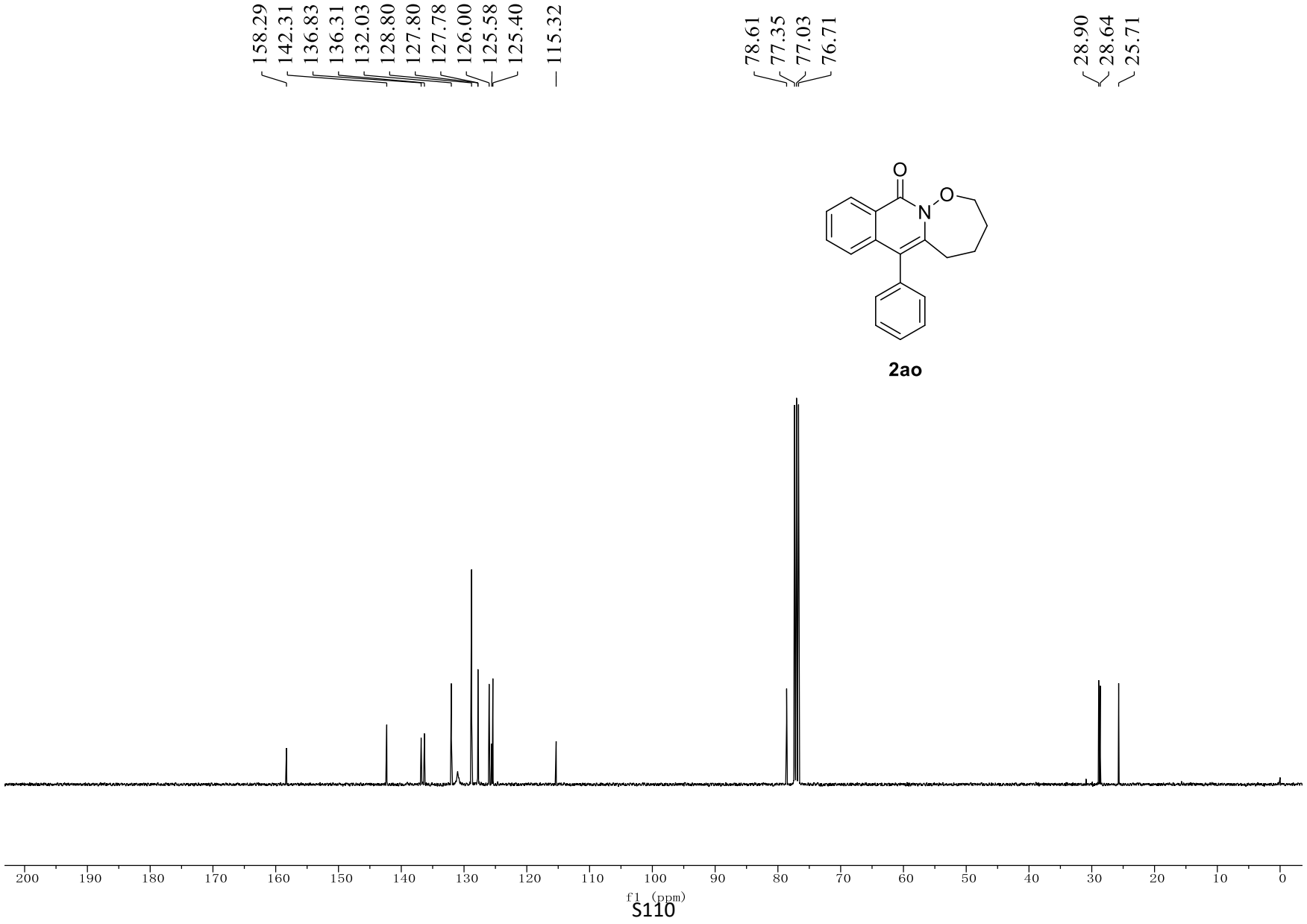

7.4522
7.4376
7.4226
7.3896
7.3750
7.3603
7.2836
7.2689
7.2624
7.1966
7.1810
7.1175
7.1019
6.6970
4.1904
4.1763
4.1622
3.6290
3.1405
3.1265
3.1124
2.2054
0.0000
O
N
O
2am
2.01
1.14
2.07
1.05
1.07
0.99
2.00
2.05
1.97
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

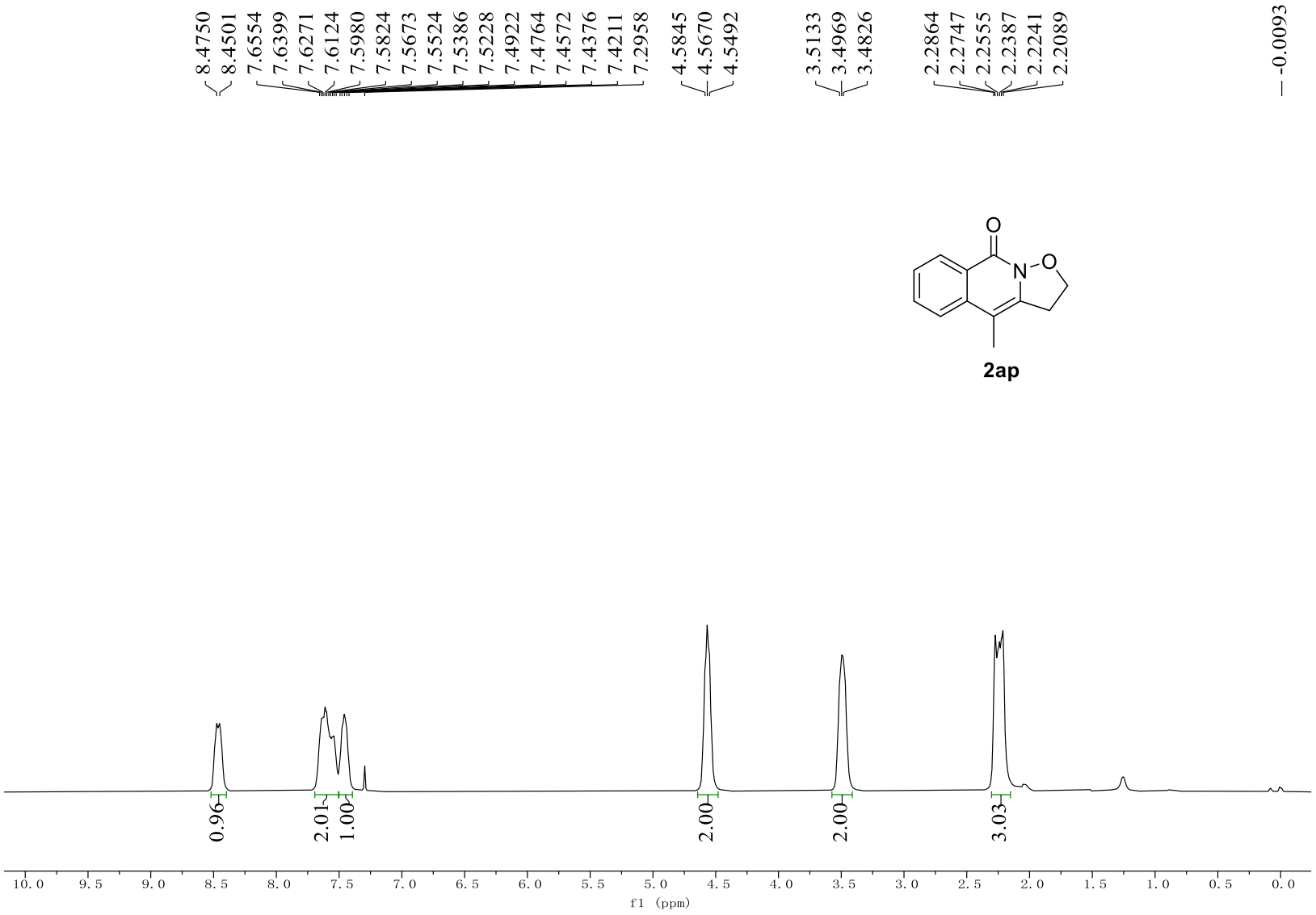

169.6756
139.5215
137.1255
136.4619
132.3817
129.9833
129.7844
129.5228
129.3306
128.7624
128.5922
128.4275
128.0077
127.7001
122.1032
77.2377
76.9838
76.7299
69.0872
42.4769
34.2641
21.0363
O
N
O
2am
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

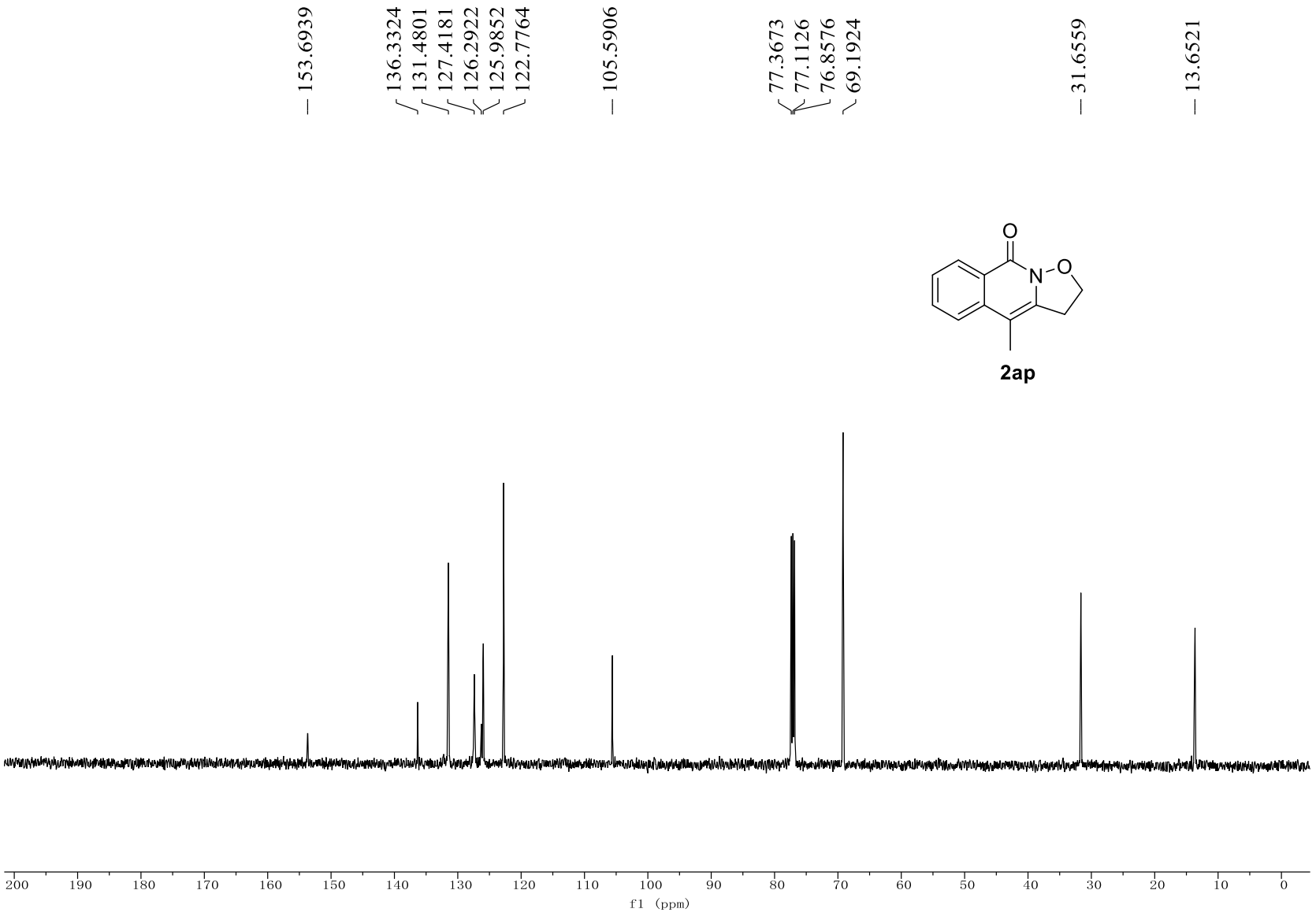

7.41
7.39
7.38
7.33
7.32
7.26
7.17
7.15
7.07
7.05
6.88
4.12
4.11
4.09
3.08
3.07
3.05
3.04
3.03
3.02
3.01
2.24
2.05
0.00
O
N
O
2an
2.05
3.00
1.06
1.01
1.01
2.08
6.13
3.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

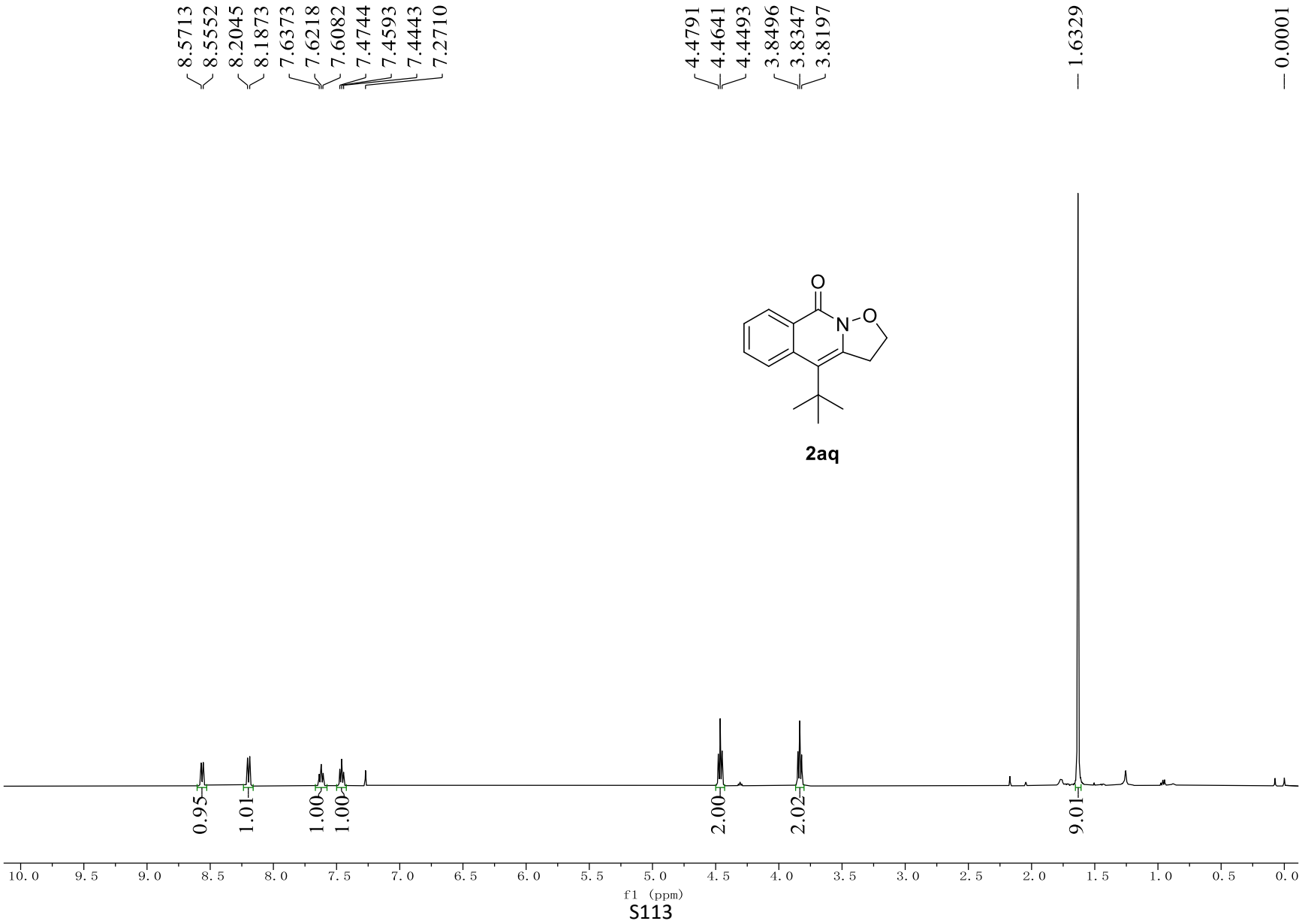

165.05
134.27
132.88
130.62
130.36
127.20
123.95
123.39
123.08
122.77
122.31
71.90
71.65
71.39
61.82
32.72
27.24
23.52
15.65
O
N
O
2an
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

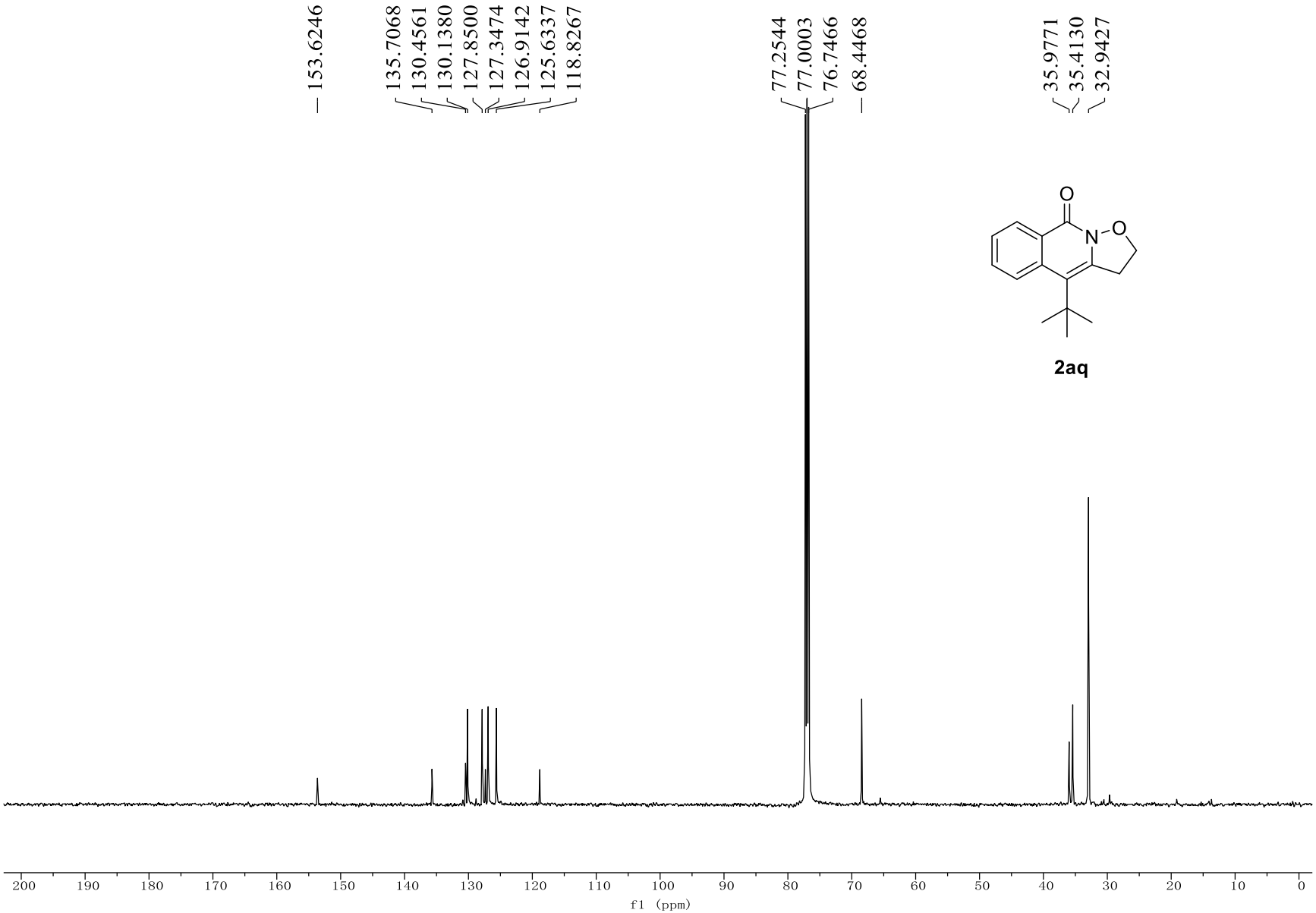

8.51
8.50
7.51
7.49
7.49
7.47
7.45
7.44
7.44
7.43
7.41
7.26
7.25
7.05
7.04
4.76
4.05
2.89
2.68
2.00
1.86
1.56
-0.00
O
N
O
2ao
0.97
3.04
2.01
2.17
1.00
0.96
0.98
2.09
2.01
1.11
1.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0
f1 (ppm)

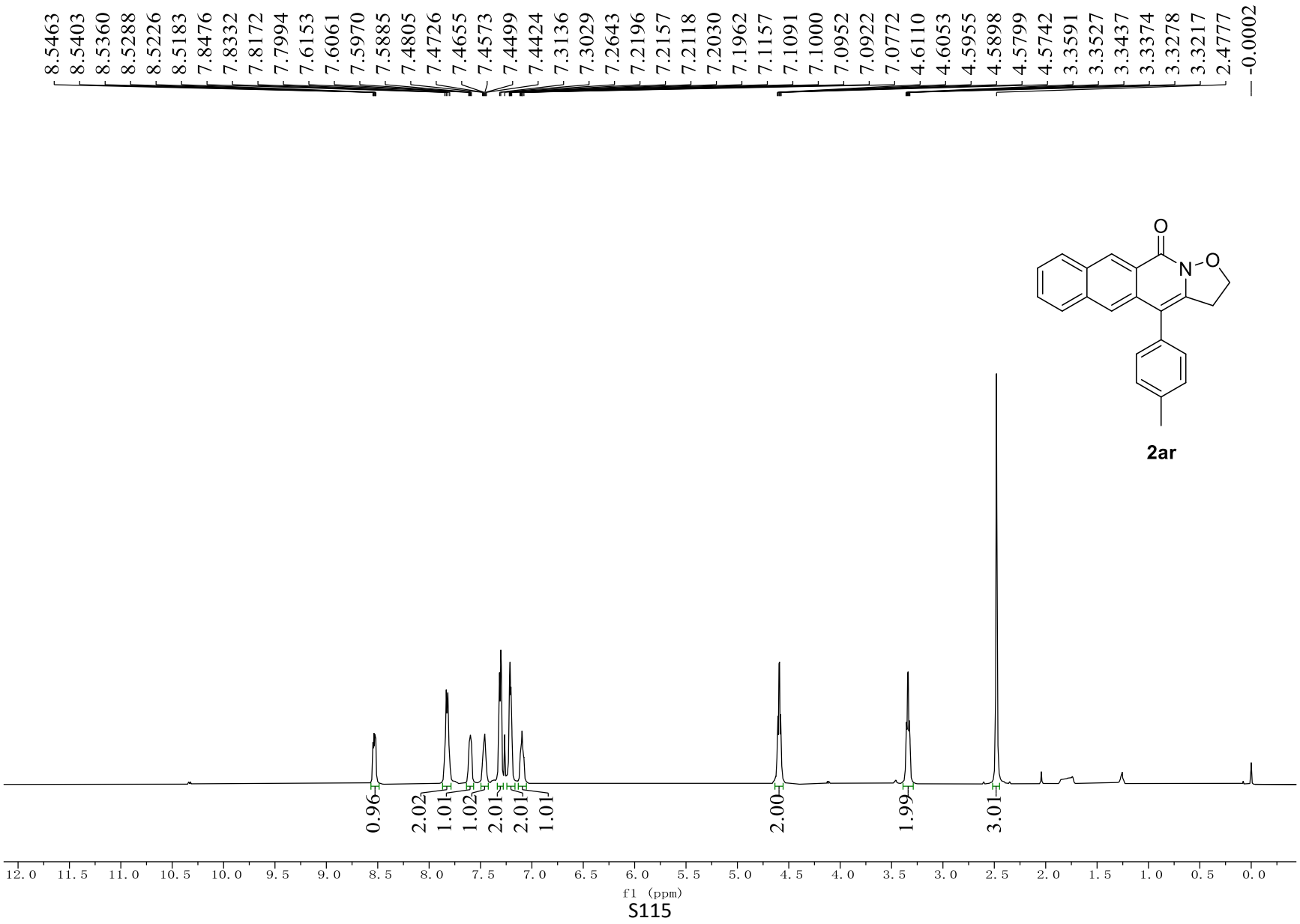

158.29
142.31
136.83
136.31
132.03
128.80
127.80
127.78
126.00
125.58
125.40
115.32
78.61
77.35
77.03
76.71
28.90
28.64
25.71
O
N
O
2ao
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

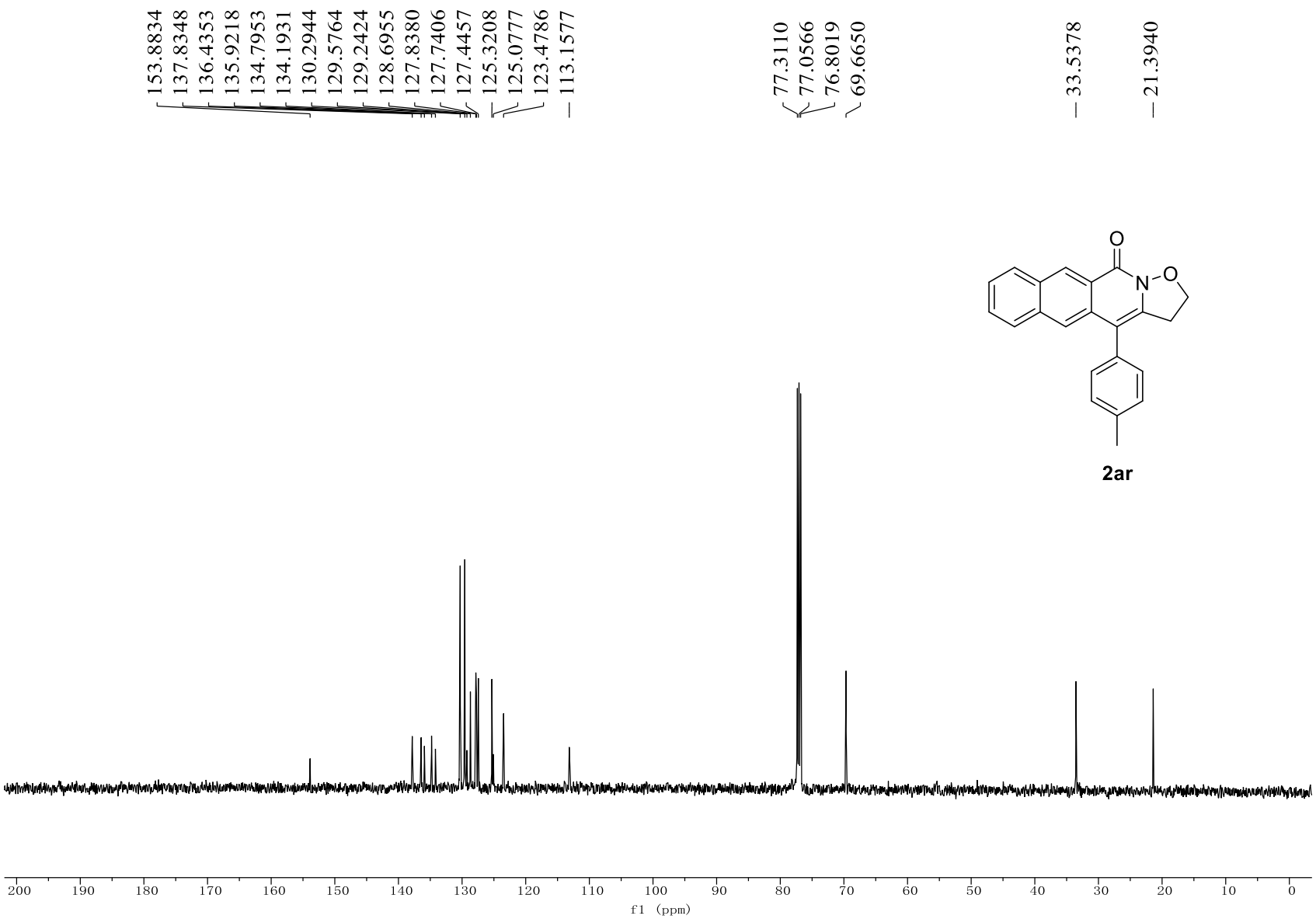

8.4750
8.4501
7.6554
7.6399
7.6271
7.6124
7.5980
7.5824
7.5673
7.5524
7.5386
7.5228
7.4922
7.4764
7.4572
7.4376
7.4211
7.2958
4.5845
4.5670
4.5492
3.5133
3.4969
3.4826
2.2864
2.2747
2.2555
2.2387
2.2241
2.2089
-0.0093
O
N
O
2ap
0.96
2.01
1.00
2.00
2.00
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.6939
136.3324
131.4801
127.4181
126.2922
125.9852
122.7764
105.5906
77.3673
77.1126
76.8576
69.1924
31.6559
13.6521
O
N
O
2ap
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.5713
8.5552
8.2045
8.1873
7.6373
7.6218
7.6082
7.4744
7.4593
7.4443
7.2710
4.4791
4.4641
4.4493
3.8496
3.8347
3.8197
1.6329
0.0001
O
N
O
2aq
0.95
1.01
1.00
1.00
2.00
2.02
9.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.6246
135.7068
130.4561
130.1380
127.8500
127.3474
126.9142
125.6337
118.8267
77.2544
77.0003
76.7466
68.4468
35.9771
35.4130
32.9427
O
N
O
2aq
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.5463
8.5403
8.5360
8.5288
8.5226
8.5183
7.8476
7.8332
7.8172
7.7994
7.6153
7.6061
7.5970
7.5885
7.4805
7.4726
7.4655
7.4573
7.4499
7.4424
7.3136
7.3029
7.2643
7.2196
7.2157
7.2118
7.2030
7.1962
7.1157
7.1091
7.1000
7.0952
7.0922
7.0772
4.6110
4.6053
4.5955
4.5898
4.5799
4.5742
3.3591
3.3527
3.3437
3.3374
3.3278
3.3217
2.4777
-0.0002
O
N
O
2ar
0.96
2.02
1.01
1.02
2.01
2.01
1.01
2.00
1.99
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


153.8834
137.8348
136.4353
135.9218
134.7953
134.1931
130.2944
129.5764
129.2424
128.6955
127.8380
127.7406
127.4457
125.3208
125.0777
123.4786
113.1577
77.3110
77.0566
76.8019
69.6650
33.5378
21.3940
O
N
O
2ar
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)