Supporting Information

One-Pot Synthesis of Pyrrolo[3,2,1-*de*]phenanthridines from 7-Phenylindoles via Tandem C-H Olefination/Aza-Michael addition

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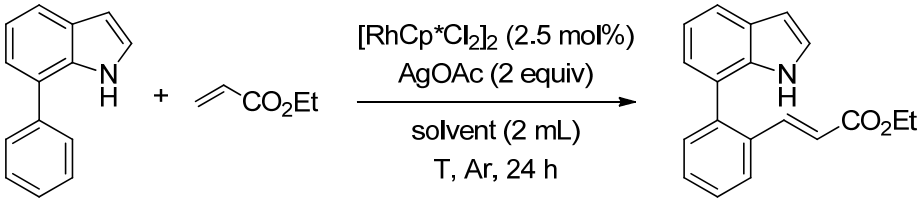
Contents:

1. Optimization of the C-H olefination of 7-phenyl-1*H*-indole 1a with ethyl acrylate 2a

Table S1 Optimization results of the C-H olefination of 7-phenyl-1*H*-indole **1a** with ethyl acrylate **2a**[a]



1a **2a** **3a**

entry	solvent	T(°C)	Yield[b] (%)
1	EtOAc	100°C	35
2	acetone	100°C	84
3	DCM	100°C	40
4	DCE	100°C	48
5	1.4-dioxane	100°C	75
6	DMSO	100°C	23
7	acetone	60°C	73
8	acetone	80°C	100
9	acetone	120°C	70
10	acetone	140°C	76
11[c]	acetone	80°C	99(97)

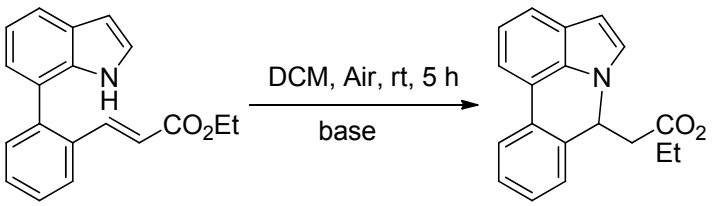
[a] Conditions: **1a** (0.3 mmol), **2a** (0.75 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol%), AgOAc (2 equiv), solvent (2 mL), T °C, Ar, 24 h.

[b] ^{1}H NMR Yield on the basis of the amount of 1a used, Number in parenthesis is isolated yield.

[c] Air atmosphere

2. Optimization of the intramolecular aza-Michael addition of (*E*)-ethyl 3-(2-(1*H*-indol-7-yl)phenyl)acrylate 3a

Table S2 Optimization results of the intramolecular aza-Michael addition [a]



3a **4a**

entry	base (equiv.)	Yield[b] (%)
1	KOH (0.3)	-

2	KO^tBu (0.3)	100
3	DMAP (0.3)	-
4	Et_3N (0.3)	-
5	Piperidine (0.3)	-
6	2,3-Dimethylquinoline (0.3)	-
7	$^nBu_4NOAc$ (10)	-
8	Me_4NOAc (10)	-
9[c]	Me_4NOAc (10)	
10	$^nBu_4NBF_4$ (10)	-
11	KPF_6 (10)	-
12	KOAc (10)	-
13	NaOAc (10)	-
14	CsOAc (10)	-
15	LiOAc (10)	-

[a] Conditions: **3a** (0.2 mmol), base, DCM (1.5 mL), Air, 5 h.

[b] Isolate yield.

[c] 1.5 mL MeCN

3. Single Crystal X-ray Structure Determination. Data collection and structural analysis of crystal were collected on an Agilent Technologies SuperNova Single Crystal Diffractometer equipped with graphite monochromatic Cu Kα radiation (λ = 1.54184 Å). The crystal was kept at 293 (10) K during data collection. Using Olex2[1], the structure was solved with the Superflip structure solution program using Charge Flipping and refined with the ShelXL refinement package using Least Squares minimisation. The hydrogen atoms on the ligands were placed in idealized positions and refined using a riding model. The detailed crystallographic data and structure refinement parameters for these compounds are summarized in **Table S1-S2.** CCDC 1943455 and 1943456 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(1) Single crystal structure of 3a (CCDC 1943455)

Table S1. Crystal data and structure refinement for **3i**

Identification code	Y-117
Empirical formula	$C_{19}H_{16}FNO2$
Formula weight	309.33
Temperature/K	294.50(10)
Crystal system	orthorhombic
Space group	Fdd2

a/Å	9.0639(3)
b/Å	37.0554(13)
c/Å	19.0348(9)
α/°	90
β/°	90
γ/°	90
Volume/$Å^3$	6393.2(4)
Z	16
$\rho_{calc}g/cm^3$	1.286
μ/mm^{-1}	0.750
F(000)	2592.0
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.548 to 145.422
Index ranges	$-8 \leq h \leq 11$, $-45 \leq k \leq 34$, $-23 \leq l \leq 20$
Reflections collected	8640
Independent reflections	2807 [R_{int} = 0.0226, R_{sigma} = 0.0182]
Data/restraints/parameters	2807/1/209
Goodness-of-fit on F^2	1.068
Final R indexes [I>=2σ (I)]	R_1 = 0.0482, wR_2 = 0.1273
Final R indexes [all data]	R_1 = 0.0540, wR_2 = 0.1336
Largest diff. peak/hole / e $Å^{-3}$	0.23/-0.17

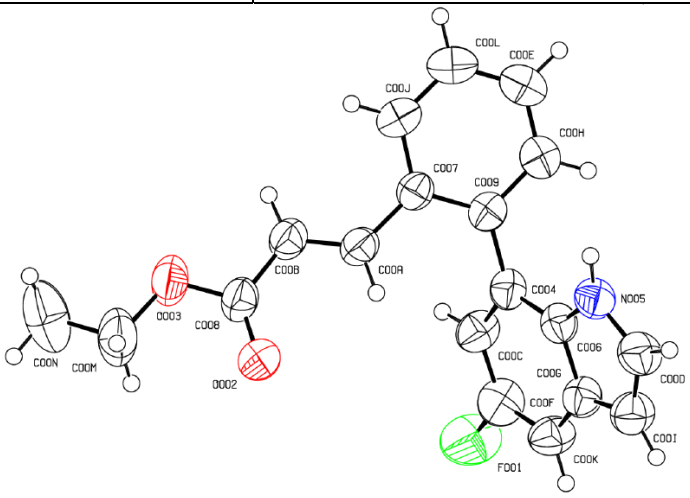


Figure S1 X-Ray structure of **3i** with 50% probability ellipsoids

(2) Single crystal structure of 3m (CCDC 1943456)

Table S2. Crystal data and structure refinement for **3m**

Identification code	Y-I_164
Empirical formula	2(C21H19NO4)
Formula weight	698.74
Temperature/K	295.42(10)
Crystal system	monoclicnic

Space group	$P2_1/n$
a/Å	10.7100(8)
b/Å	13.5614(18)
c/Å	12.7131(8)
α/°	90
β/°	98.322(7)
γ/°	90
Volume/Å^3	1827.0(3)
Z	2
ρ_{calc}g/cm^3	1.270
μ/mm^{-1}	0.720
F(000)	736.0
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.028 to 145.174
Index ranges	$-8 \le h \le 13$, $-12 \le k \le 16$, $-15 \le l \le 14$
Reflections collected	8673
Independent reflections	5544 [R_{int} = 0.0496, R_{sigma} = 0.0685]
Data/restraints/parameters	5544/1/473
Goodness-of-fit on F^2	1.017
Final R indexes [I>=2σ (I)]	R_1 = 0.0582, wR_2 = 0.1264
Final R indexes [all data]	R_1 = 0.1065, wR_2 = 0.1598
Largest diff. peak/hole / e Å^{-3}	0.17/-0.21

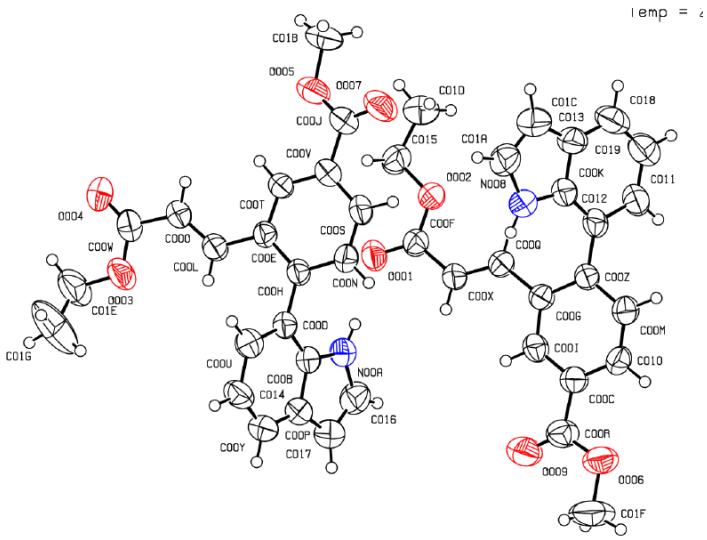
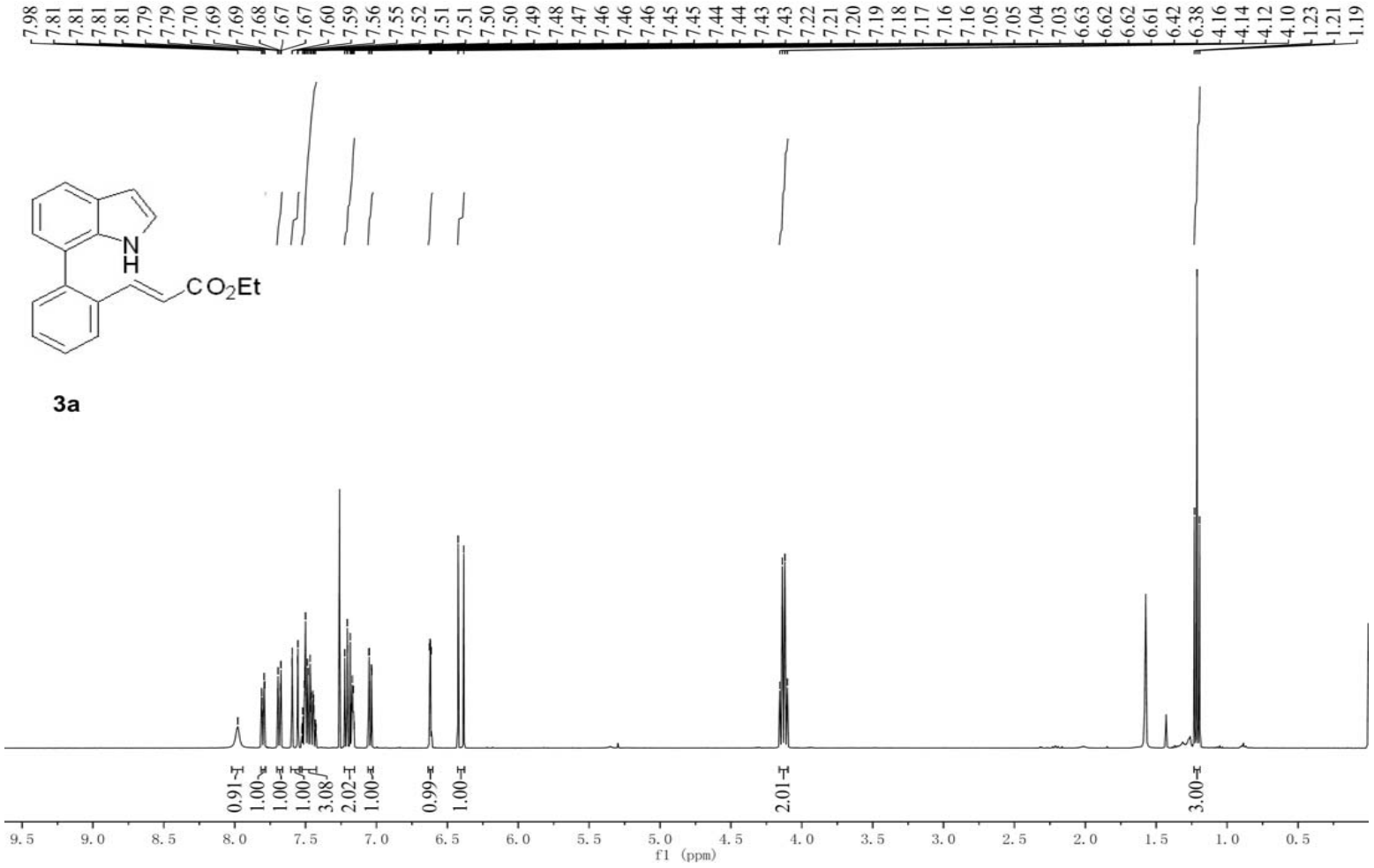


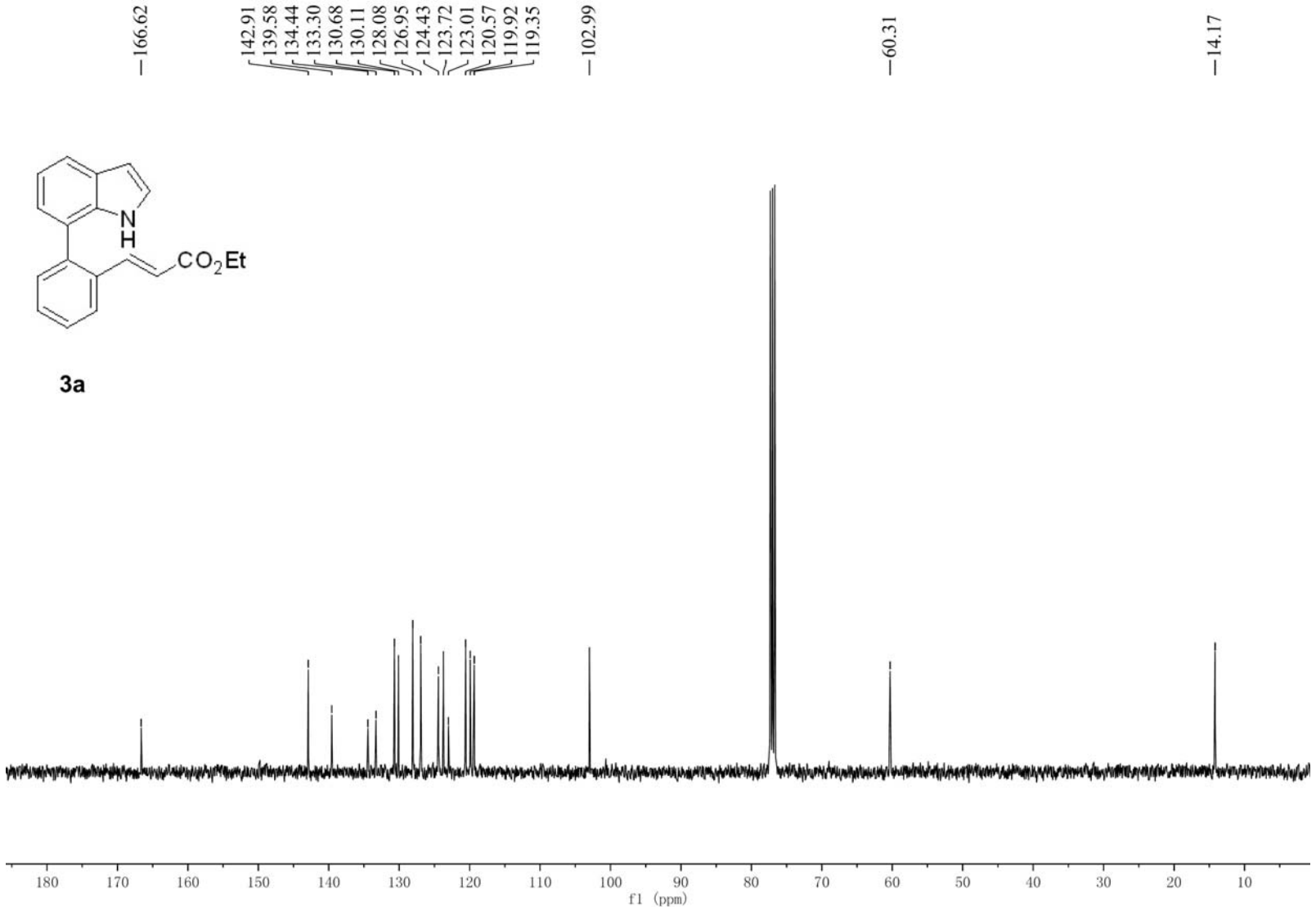
Figure S2 X-Ray structure of **3m** with 50% probability ellipsoids

References:

(1) (a) Sheldrick, G. M. *Acta Crystallogr., Sect. A: Found. Crystallogr.* **2008**, 64, 112-122. (b) Dolomanov, A. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschman, H. *J. Appl. Crystallogr.* **2009**, 42, 339-341.

4. Copies of ^{1}H and ^{13}C-NMR charts of products

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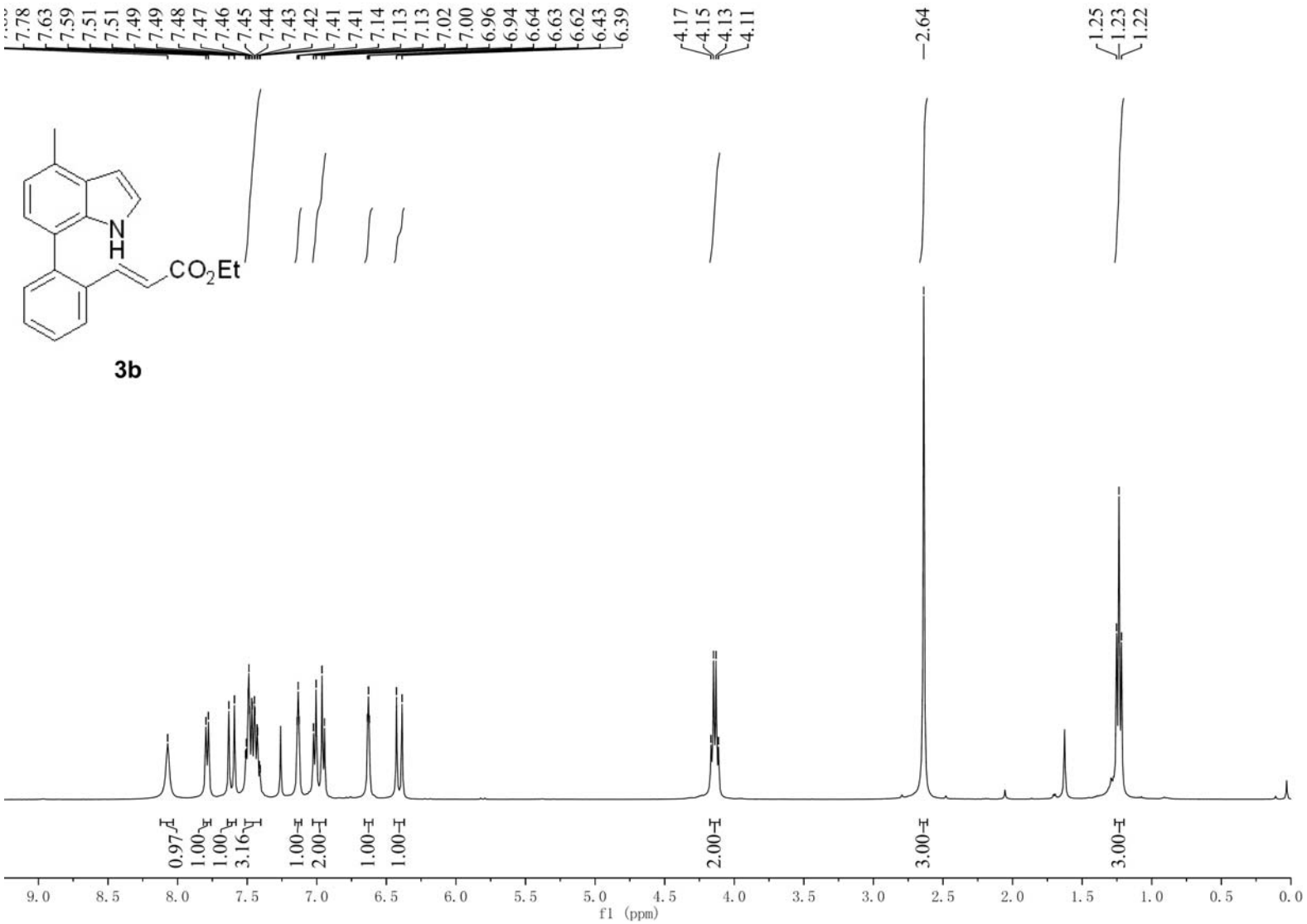
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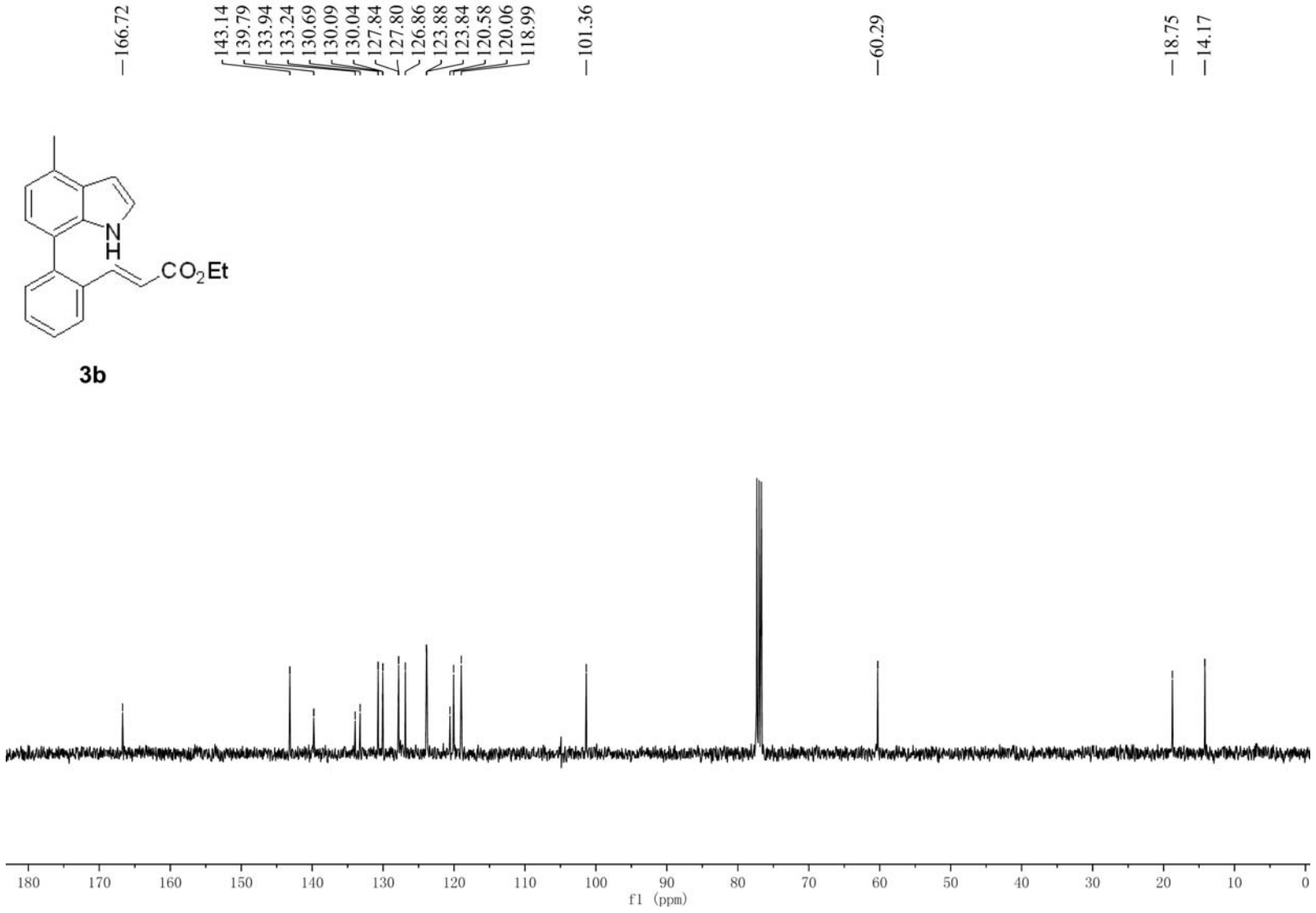
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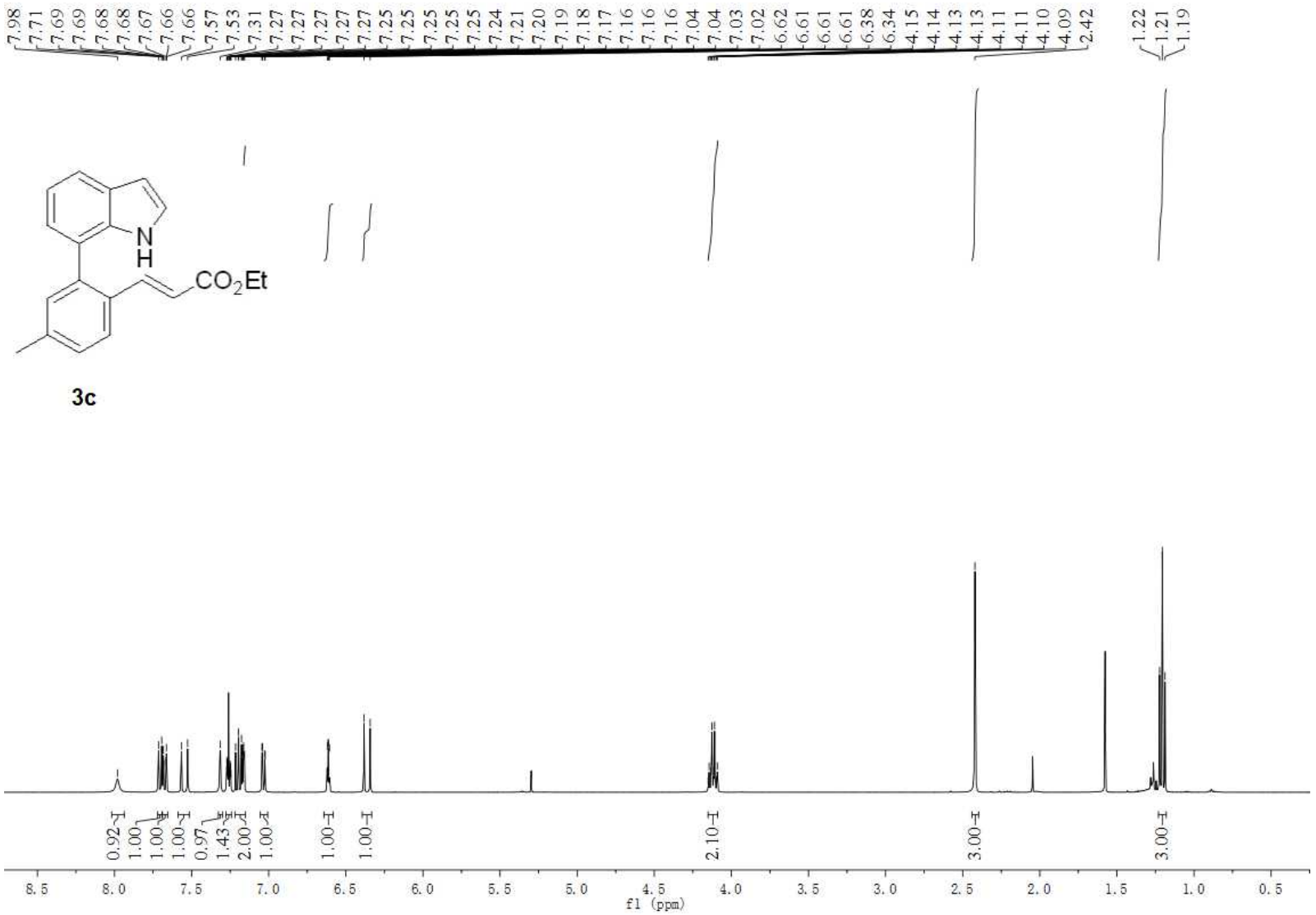
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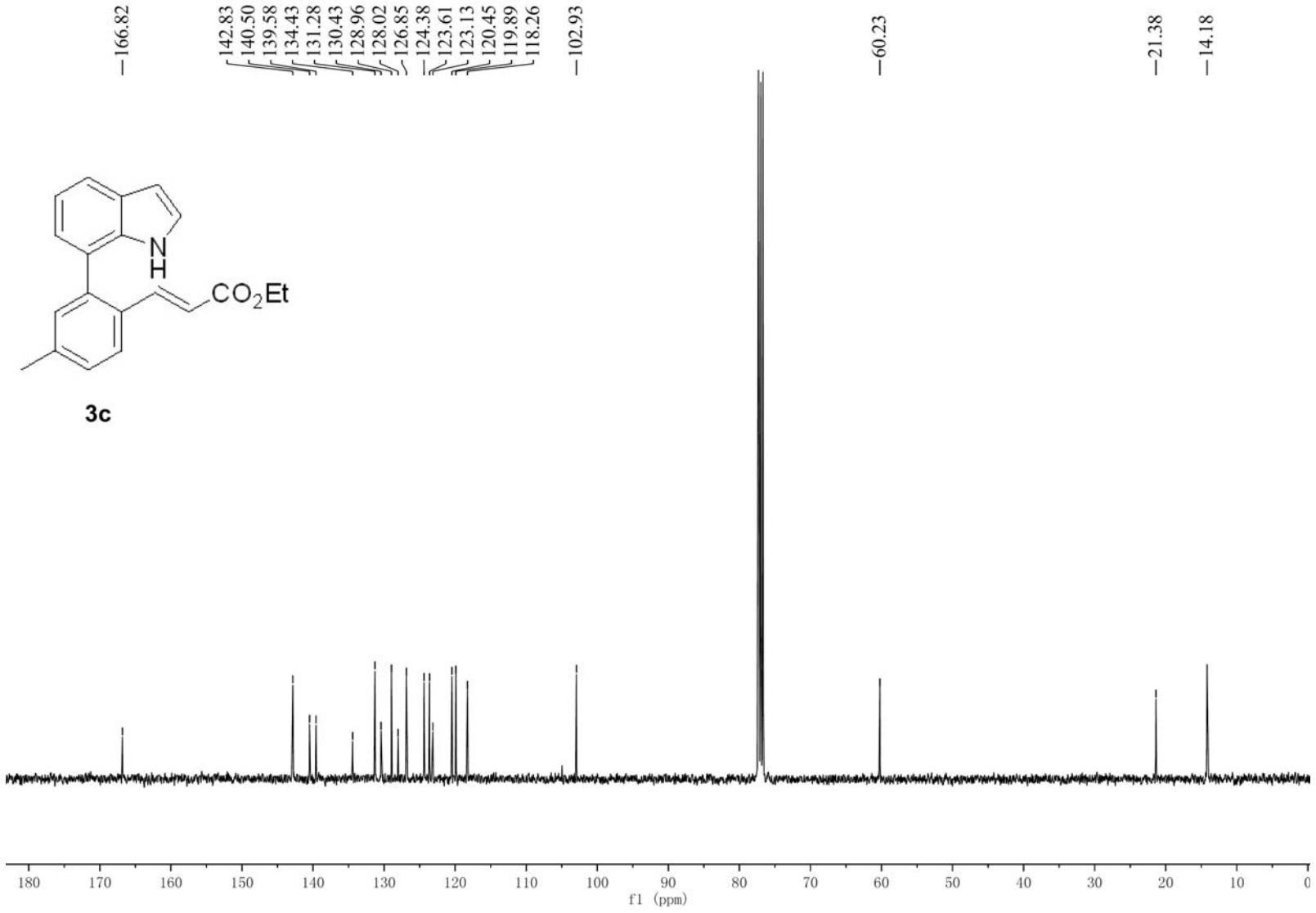
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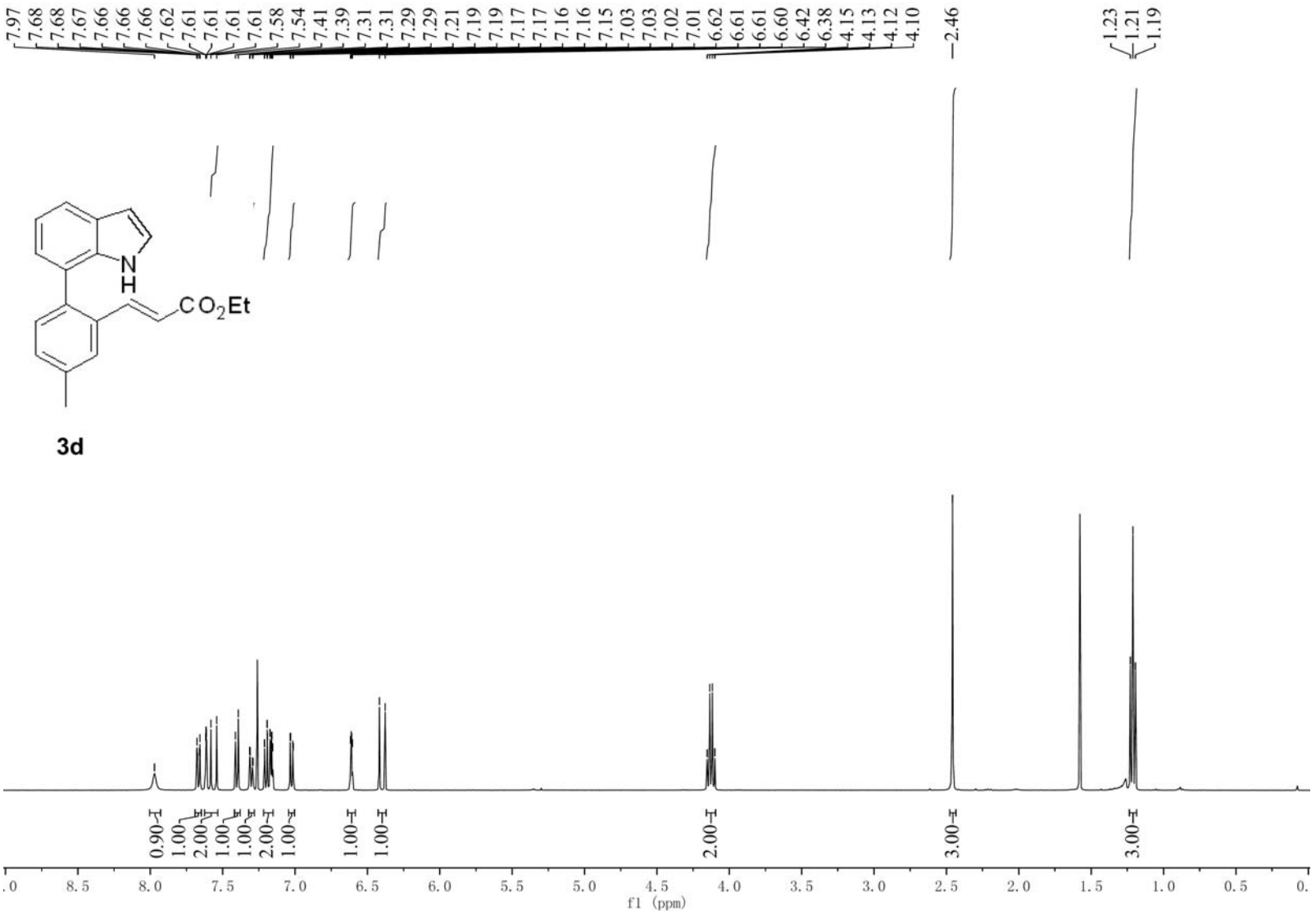
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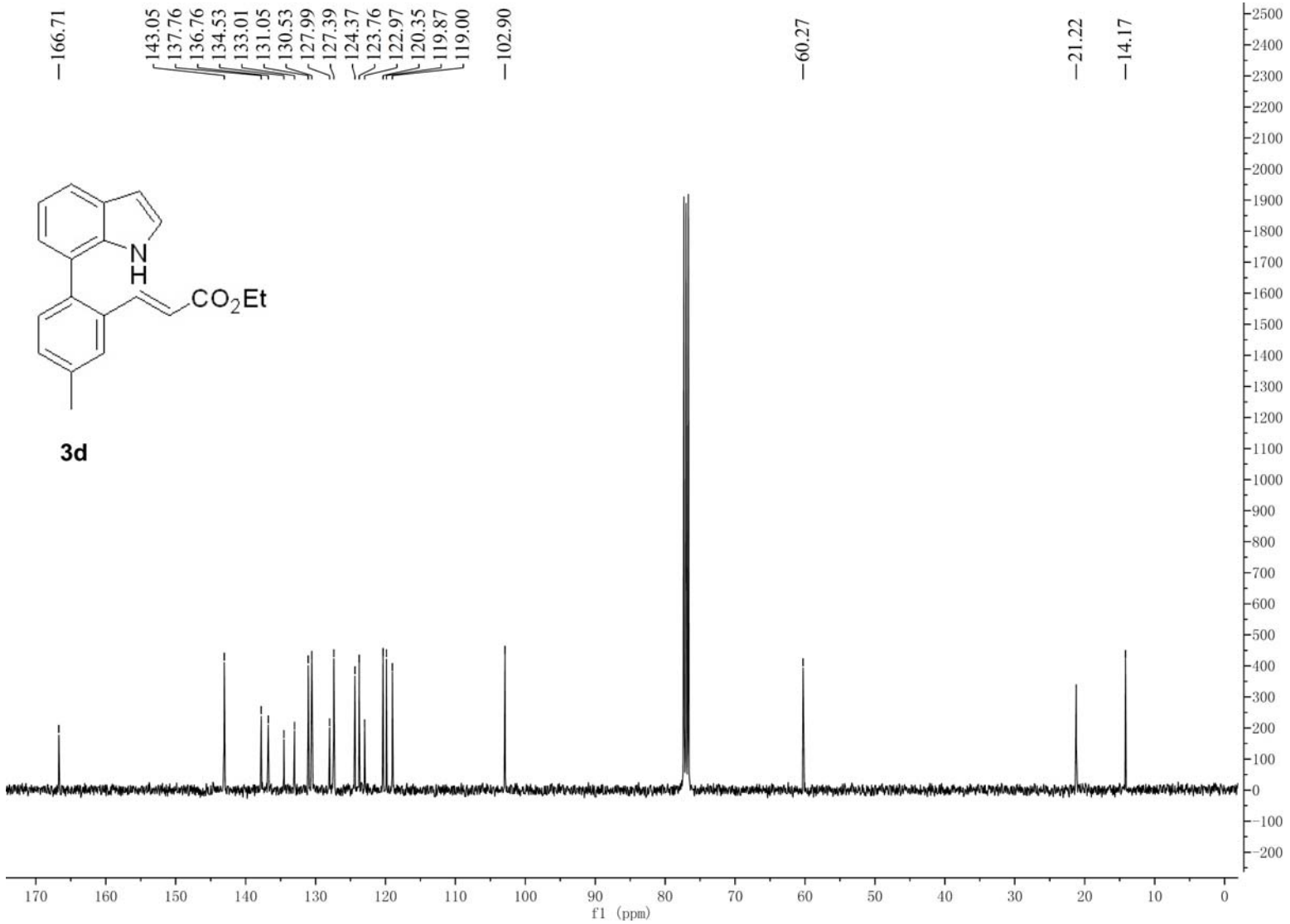
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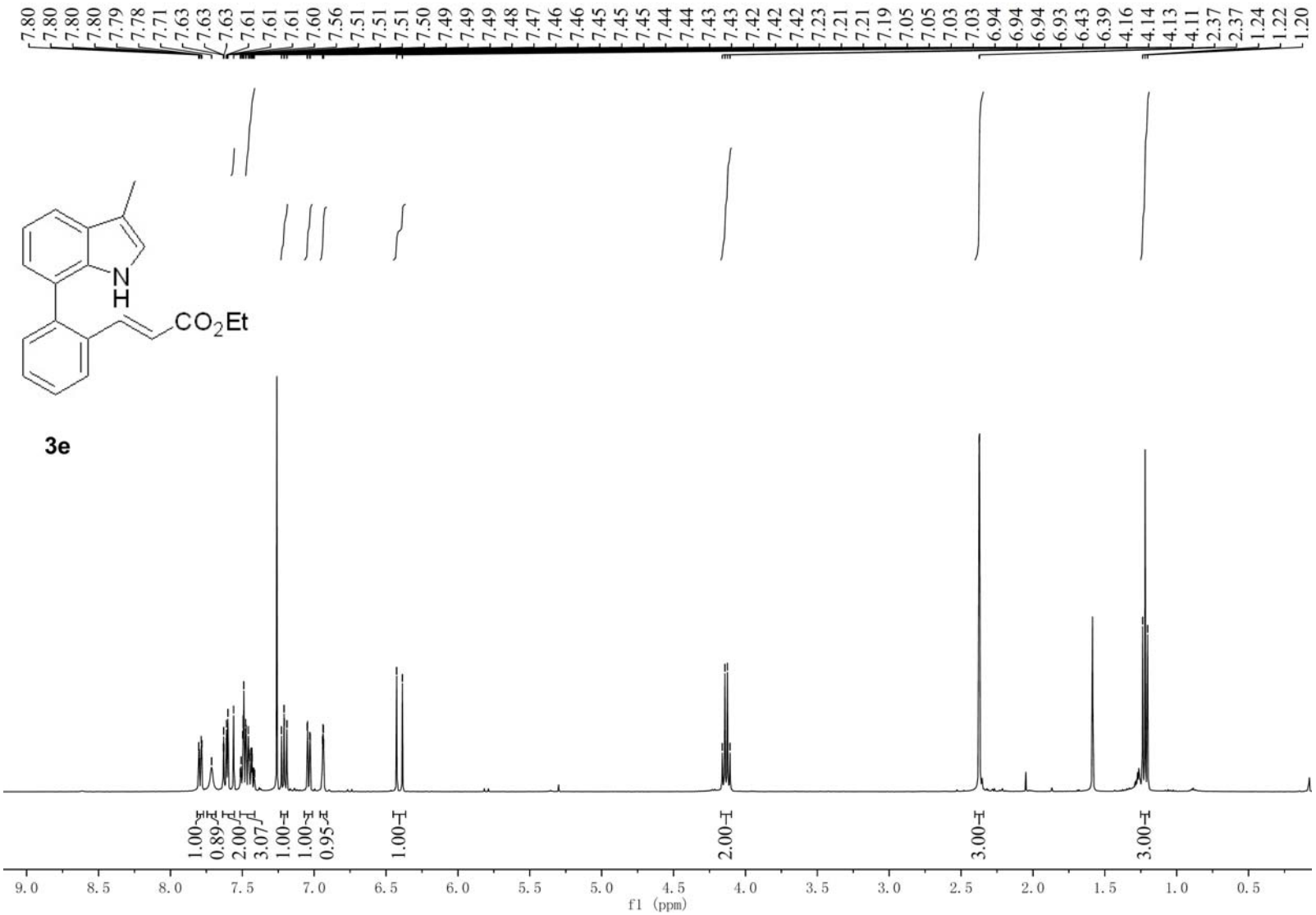
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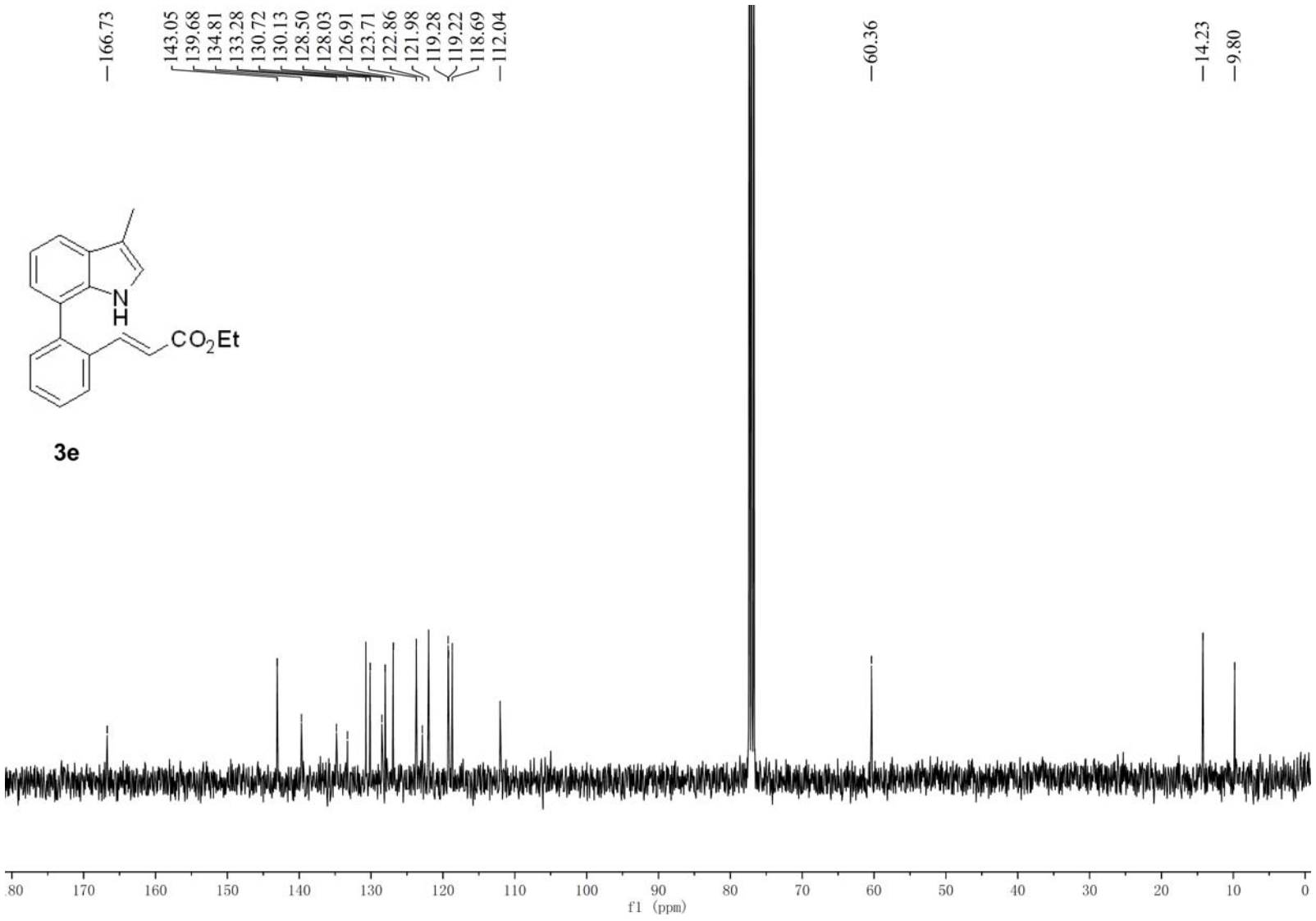
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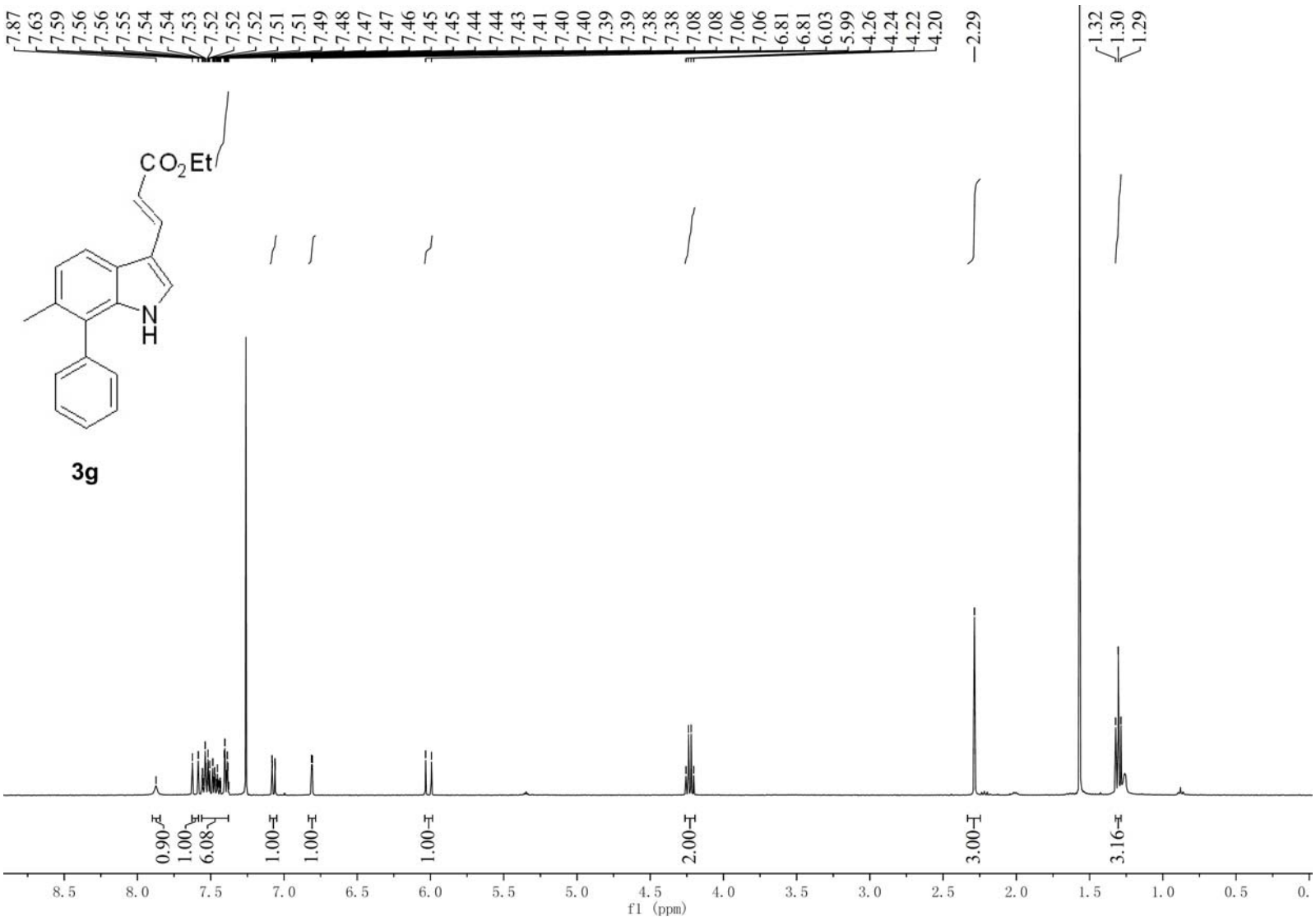
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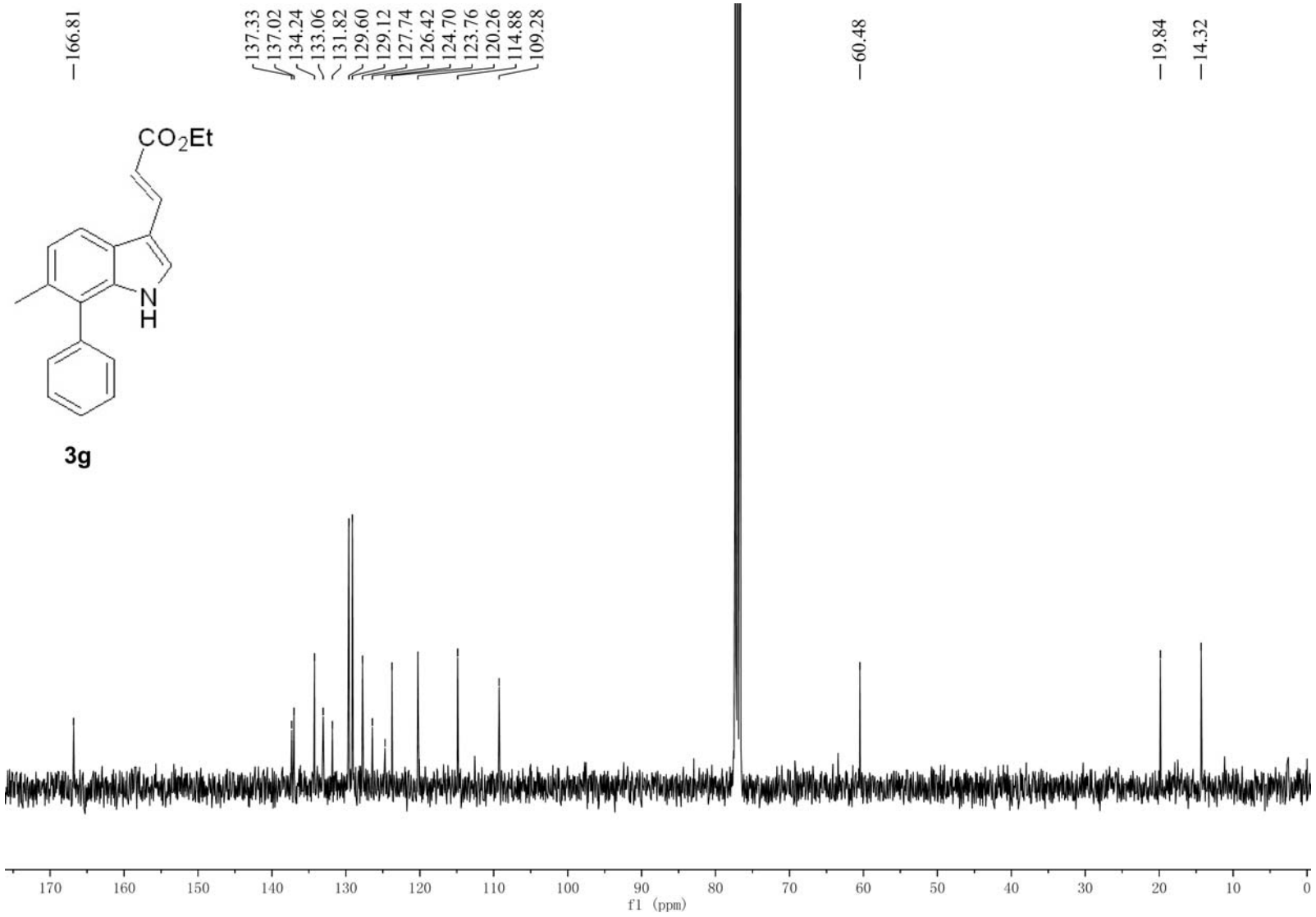
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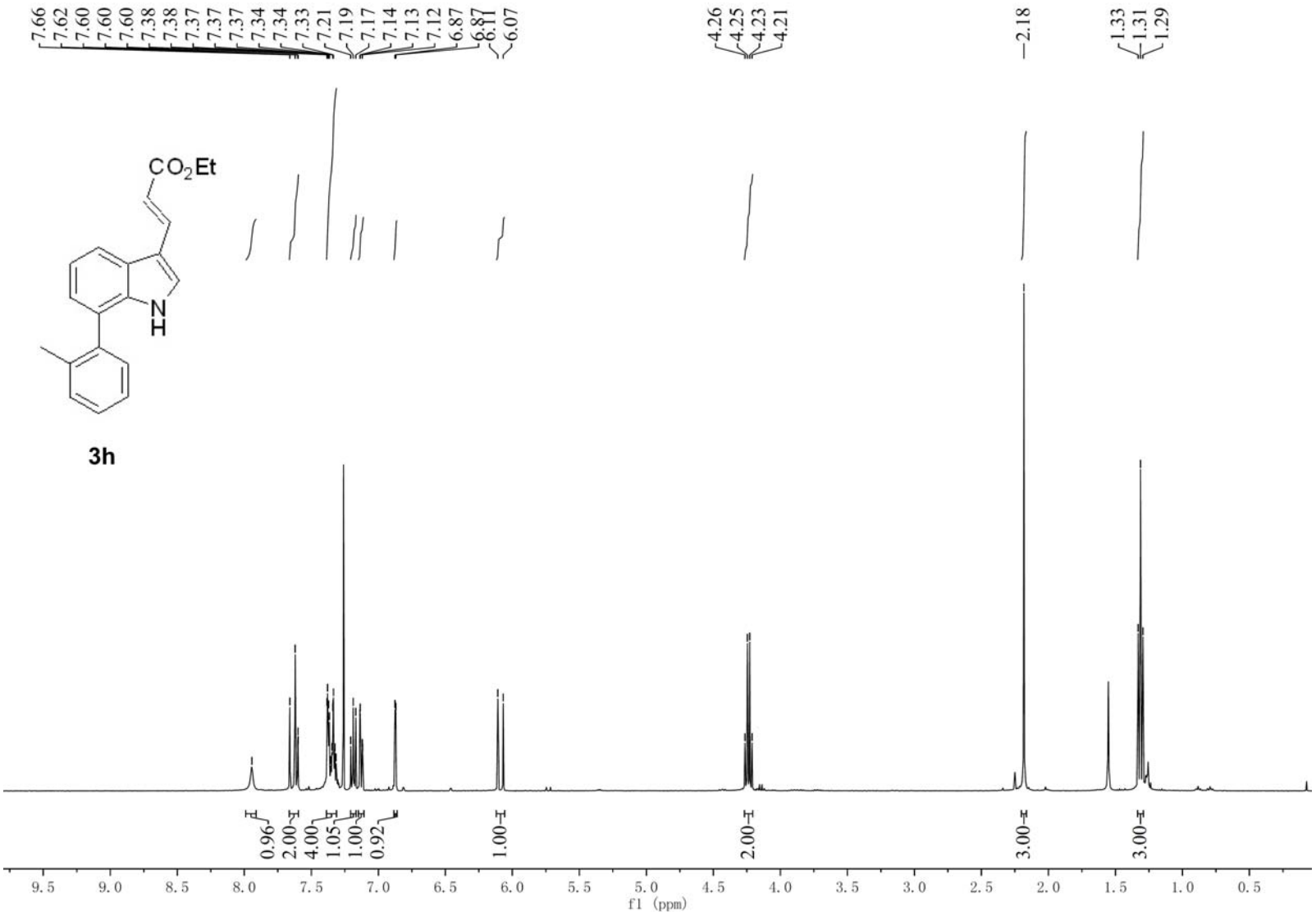
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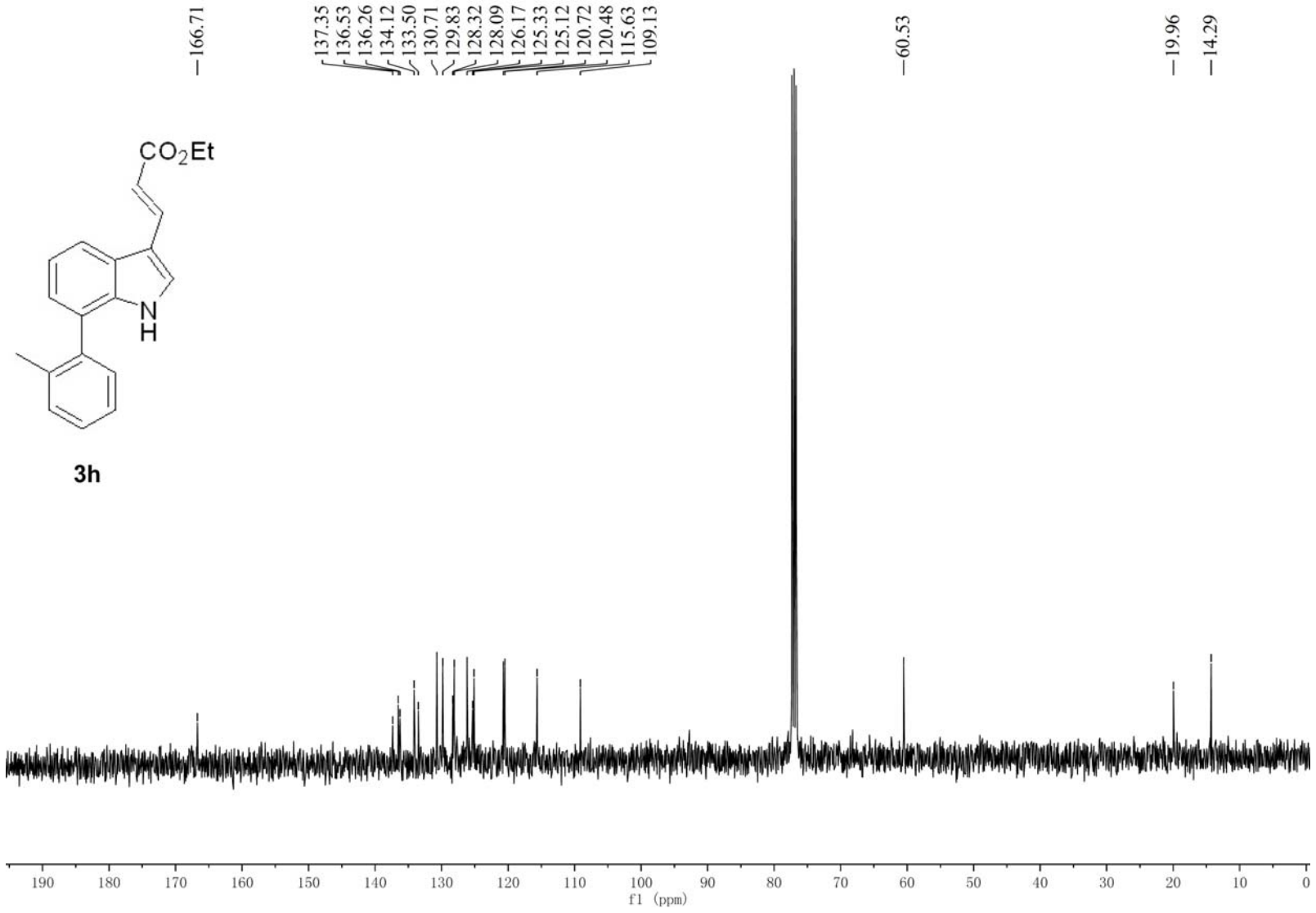
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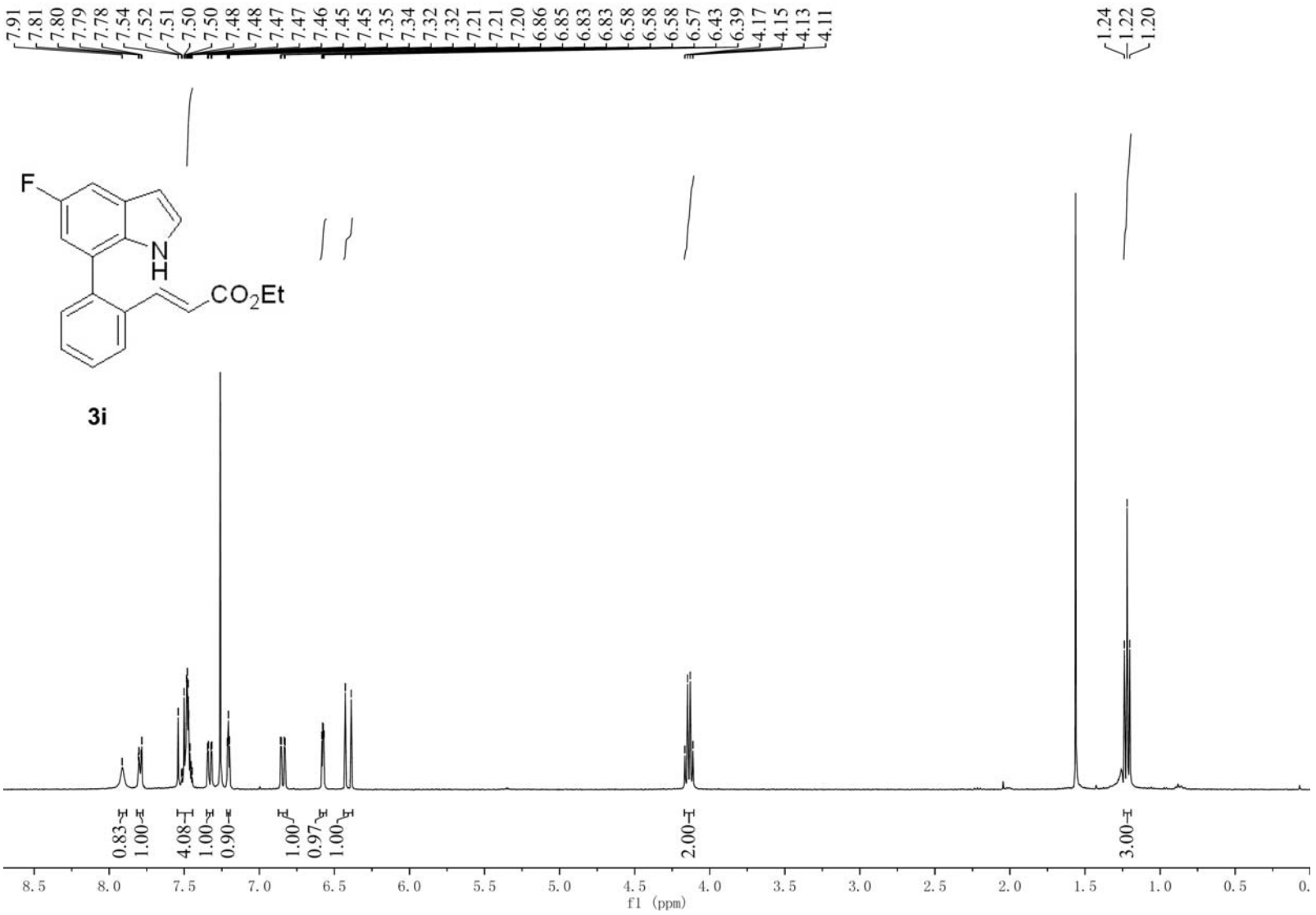
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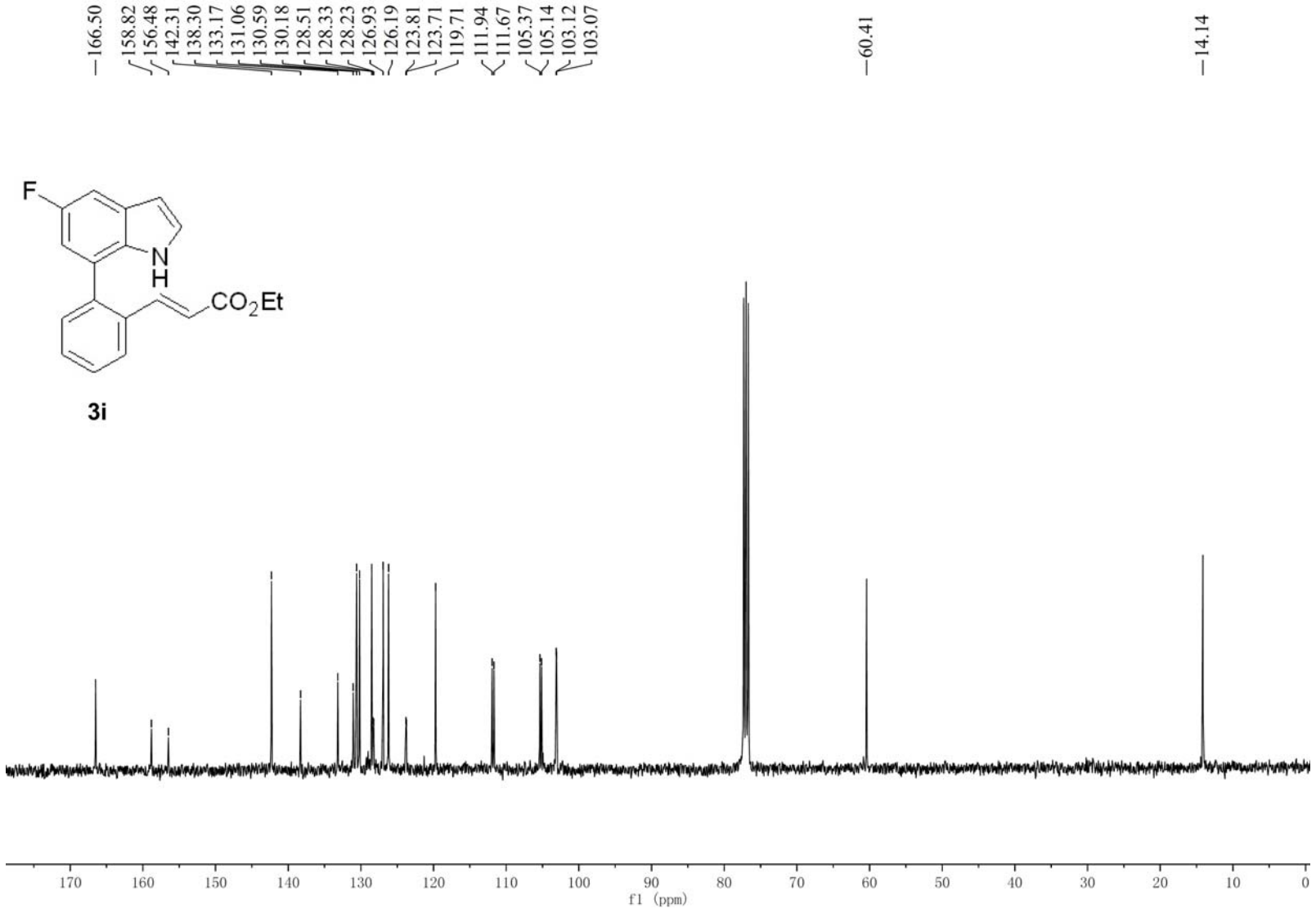
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128.33
128.23
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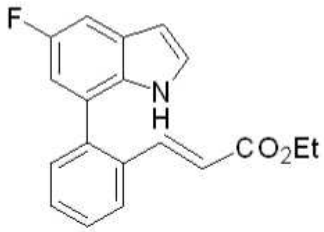
126.19
123.81
123.71
119.71
111.94
111.67
105.37
105.14
103.12
103.07
60.41
14.14
F
N
H
CO2Et
3i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


F
N
H
CO2Et

3i

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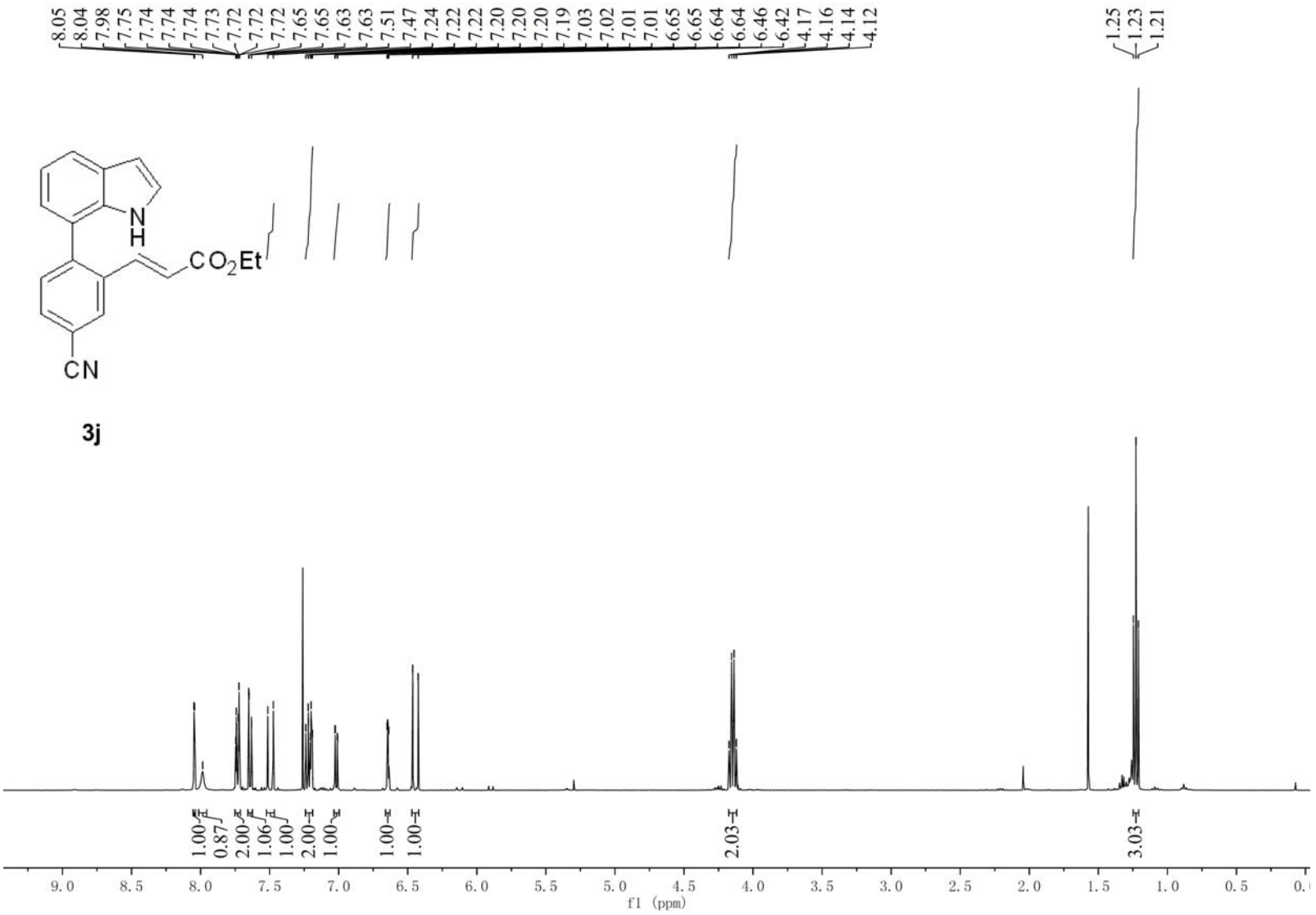
-124.83
-124.86
-15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155
f1 (ppm)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7.72
7.72
7.65
7.65
7.63
7.63
7.51
7.47
7.24
7.22
7.22
7.20
7.20
7.20
7.19
7.03
7.02
7.01
7.01
6.65
6.65
6.64
6.64
6.46
6.42
4.17
4.16
4.14
4.12
1.25
1.23
1.21
N
H
CO2Et
CN
3j
1.00
0.87
2.00
1.06
1.00
2.00
1.00
1.00
1.00
2.03
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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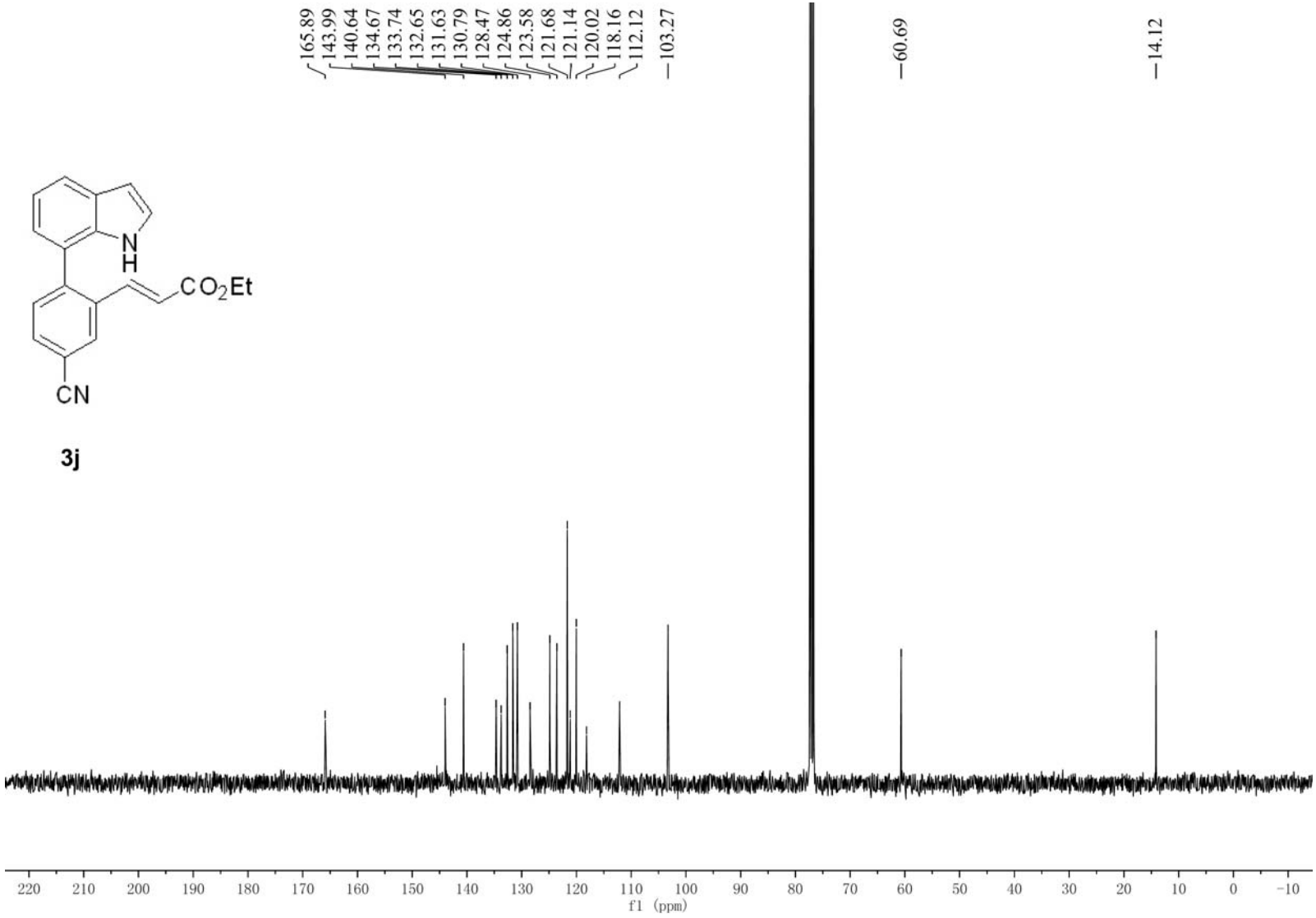
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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130.79
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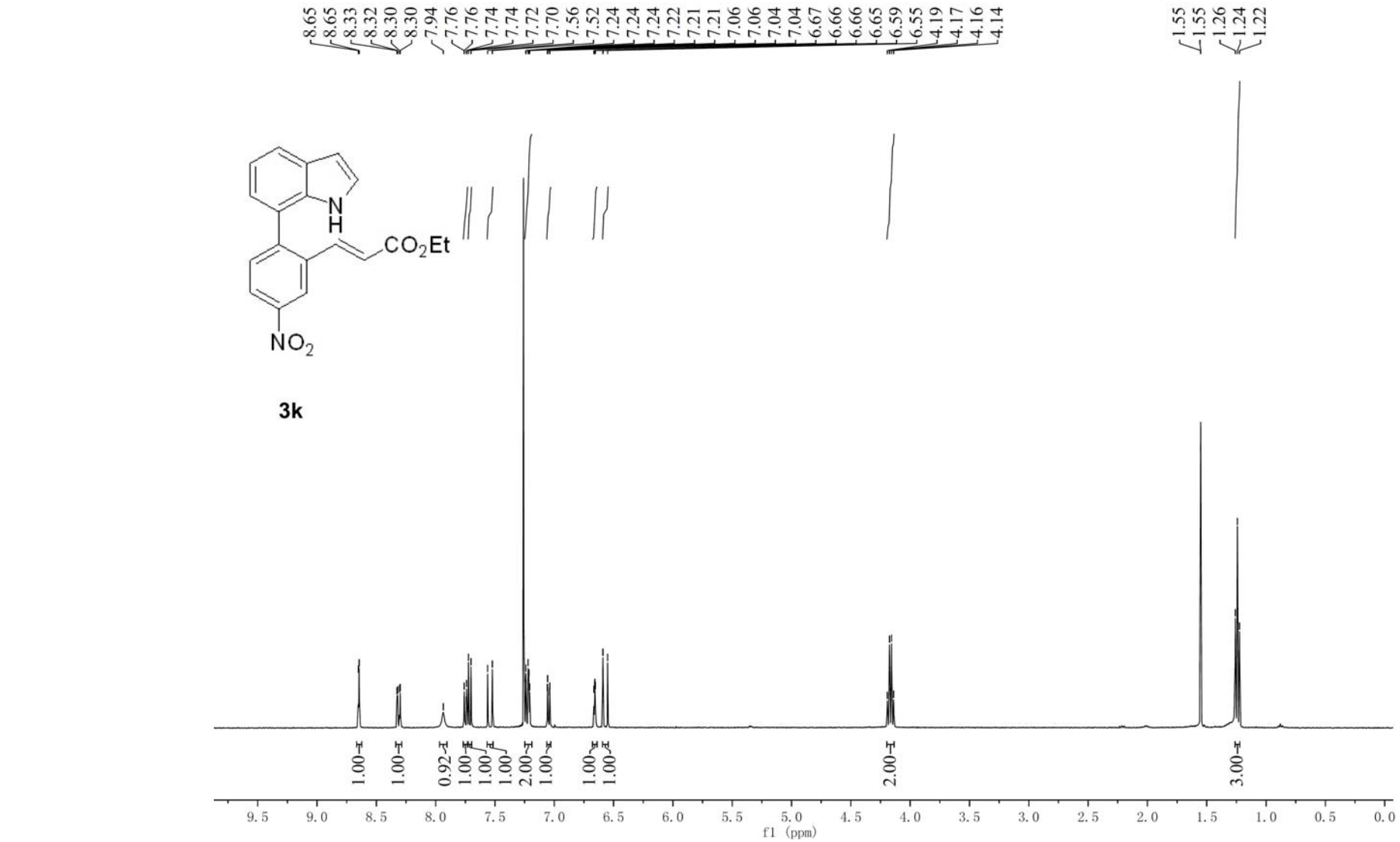
123.58
121.68
121.14
120.02
118.16
112.12
103.27
60.69
14.12
N
H
CO2Et
CN
3j
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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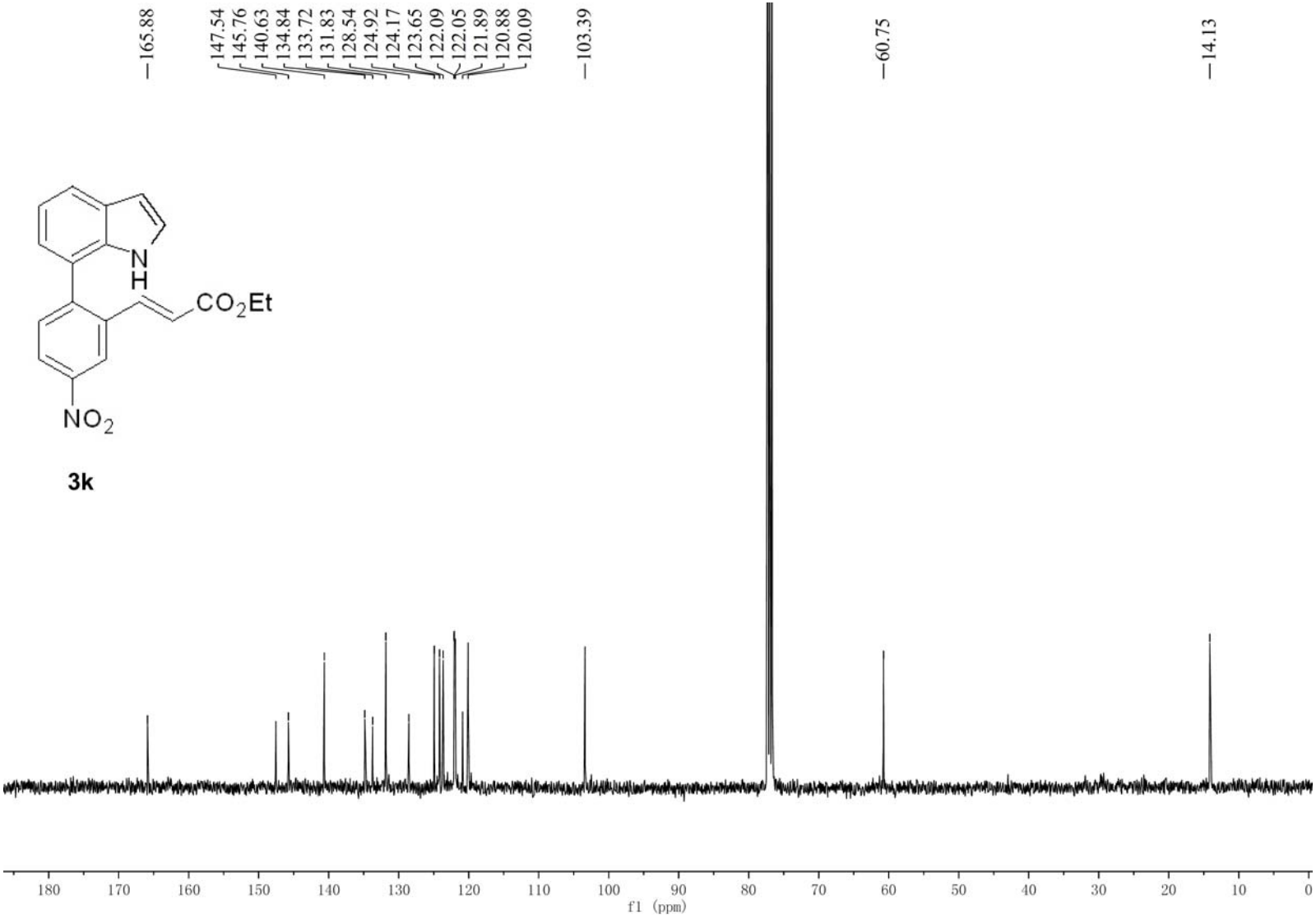
7.76
7.76
7.74
7.74
7.72
7.70
7.56
7.52
7.24
7.24
7.24
7.22
7.21
7.21
7.06
7.06
7.04
7.04
6.67
6.66
6.66
6.65
6.59
6.55
4.19
4.17
4.16
4.14
1.55
1.55
1.26
1.24
1.22
N
H
CO2Et
NO2
3k
1.00
1.00
0.92
1.00
1.00
1.00
2.00
1.00
1.00
1.00
2.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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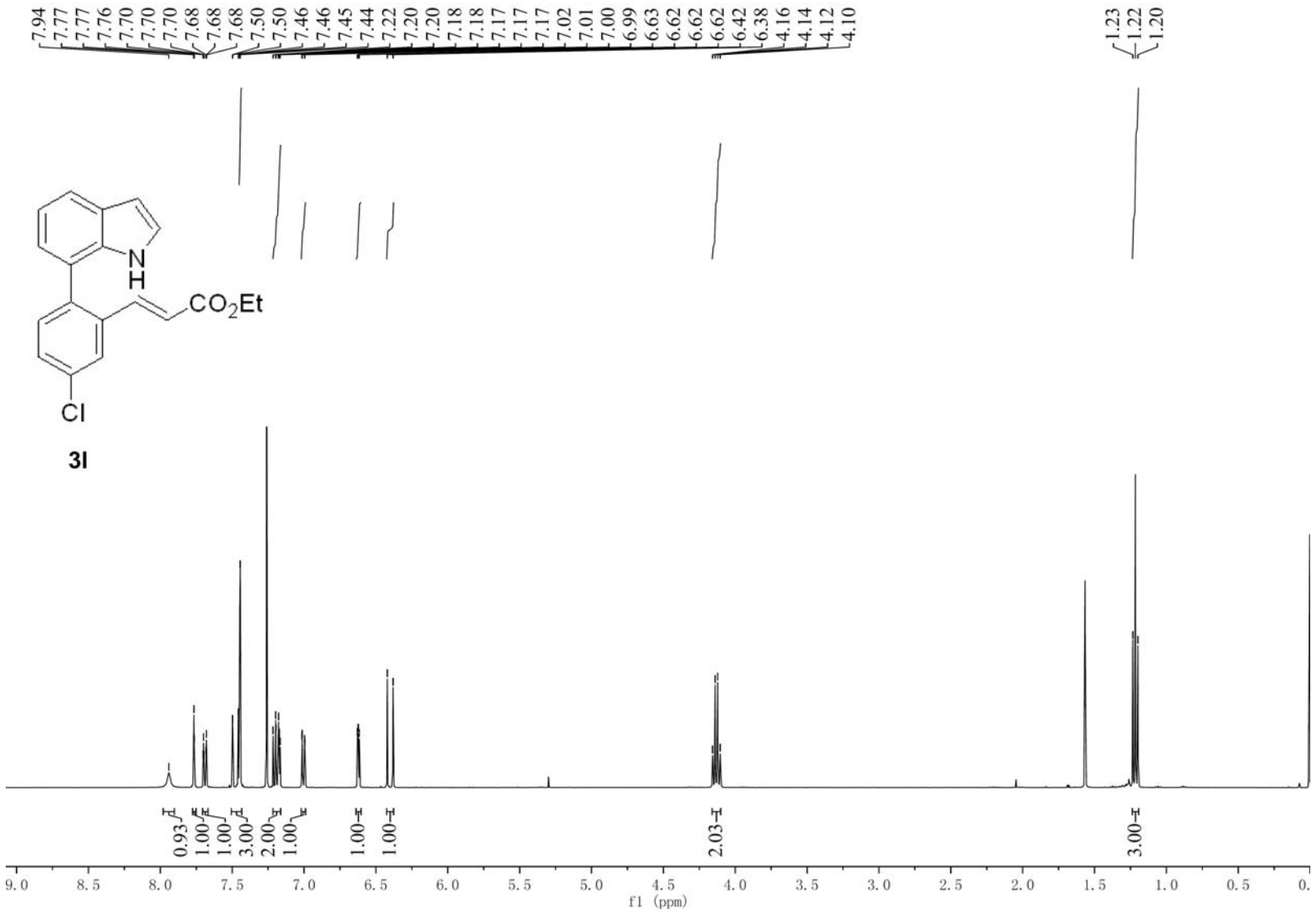
124.17
123.65
122.09
122.05
121.89
120.88
120.09
103.39
60.75
14.13
N
H
CO2Et
NO2
3k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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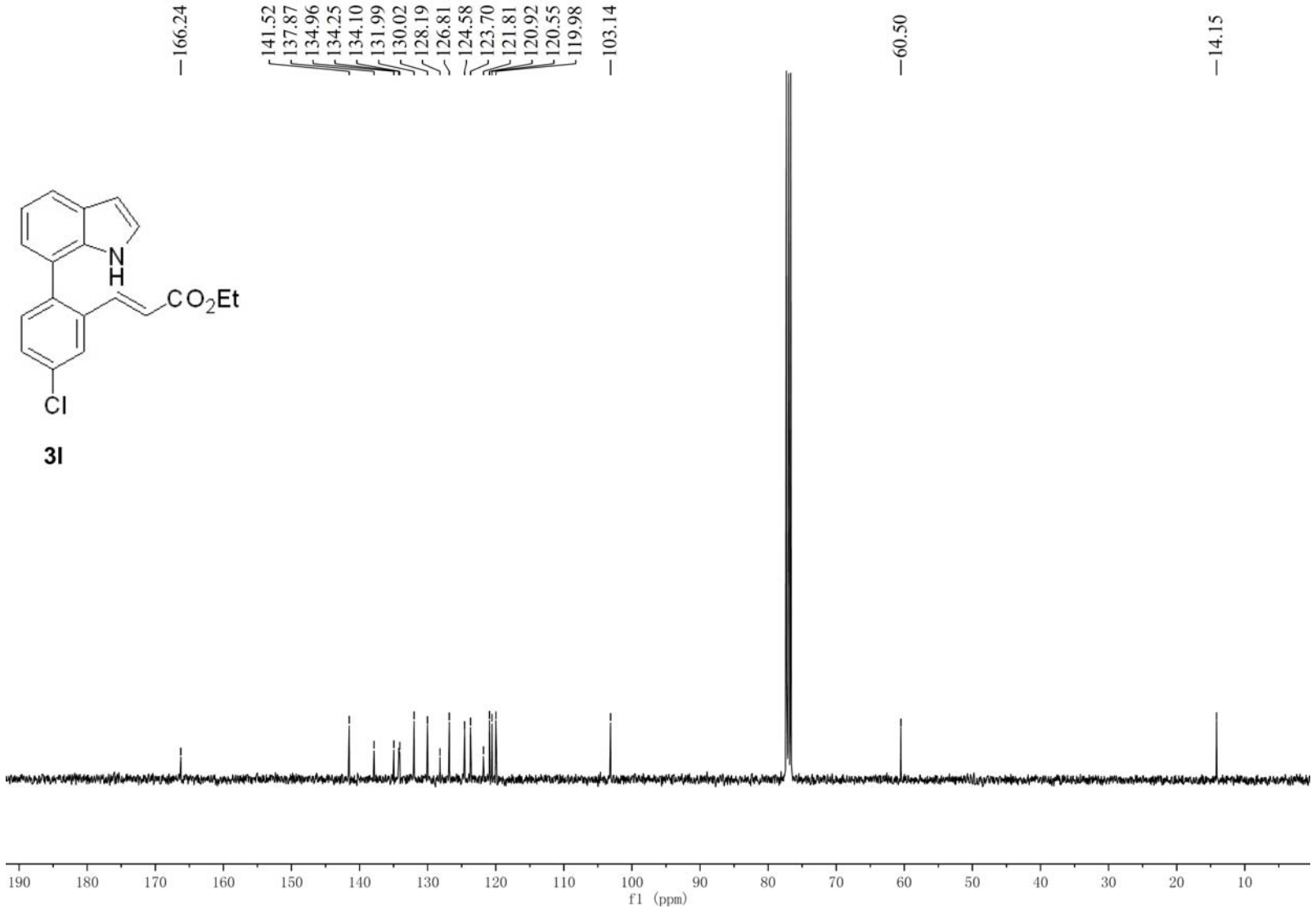
7.68
7.68
7.68
7.50
7.50
7.46
7.46
7.45
7.44
7.22
7.20
7.20
7.18
7.18
7.17
7.17
7.17
7.02
7.01
7.00
6.99
6.63
6.62
6.62
6.62
6.42
6.38
4.16
4.14
4.12
4.10
1.23
1.22
1.20
N
H
CO2Et
Cl
3l
0.93
1.00
1.00
3.00
2.00
1.00
1.00
1.00
2.03
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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134.10
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130.02
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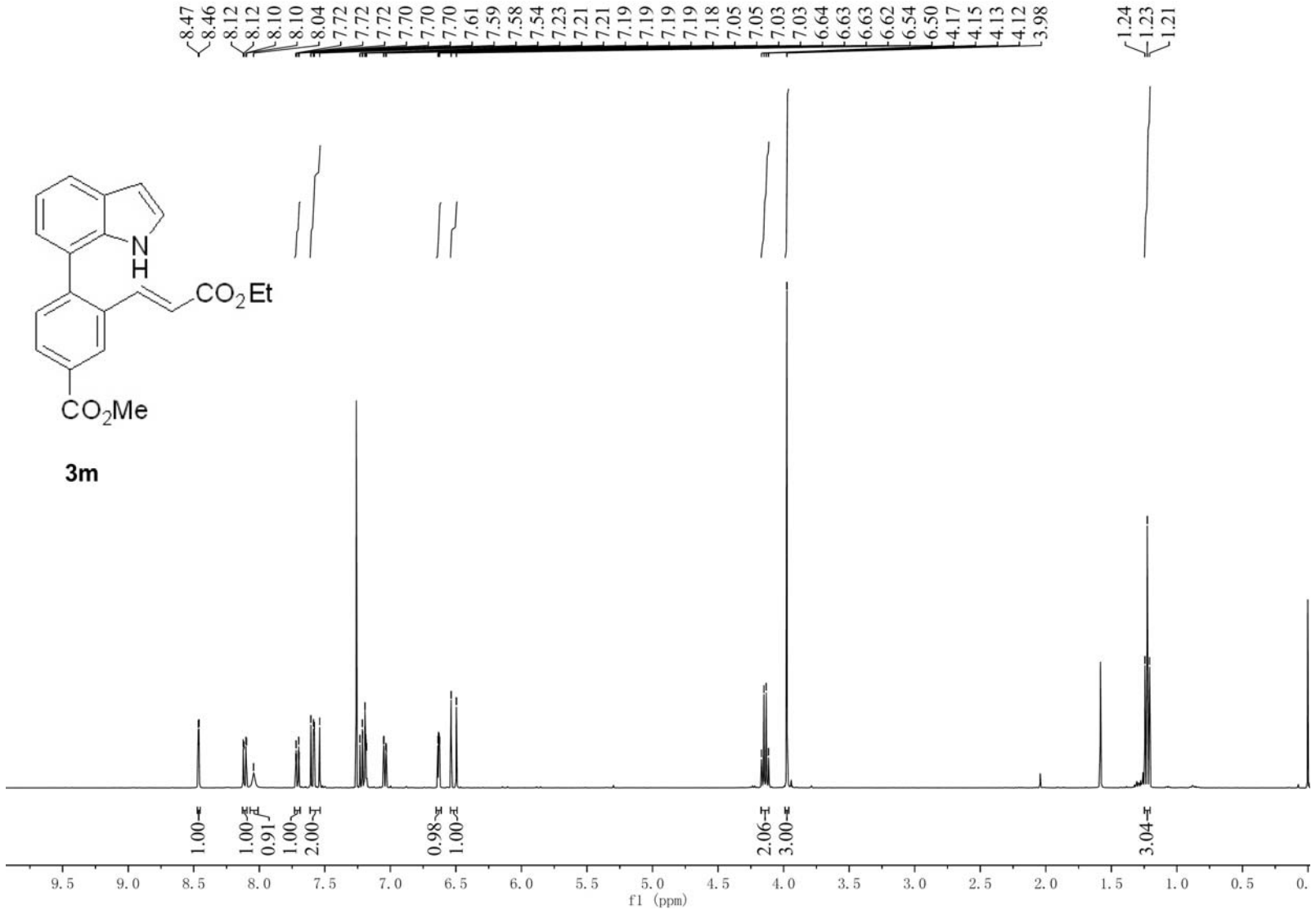
126.81
124.58
123.70
121.81
120.92
120.55
119.98
103.14
60.50
14.15
N
H
CO2Et
Cl
3l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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8.12
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8.10
8.04
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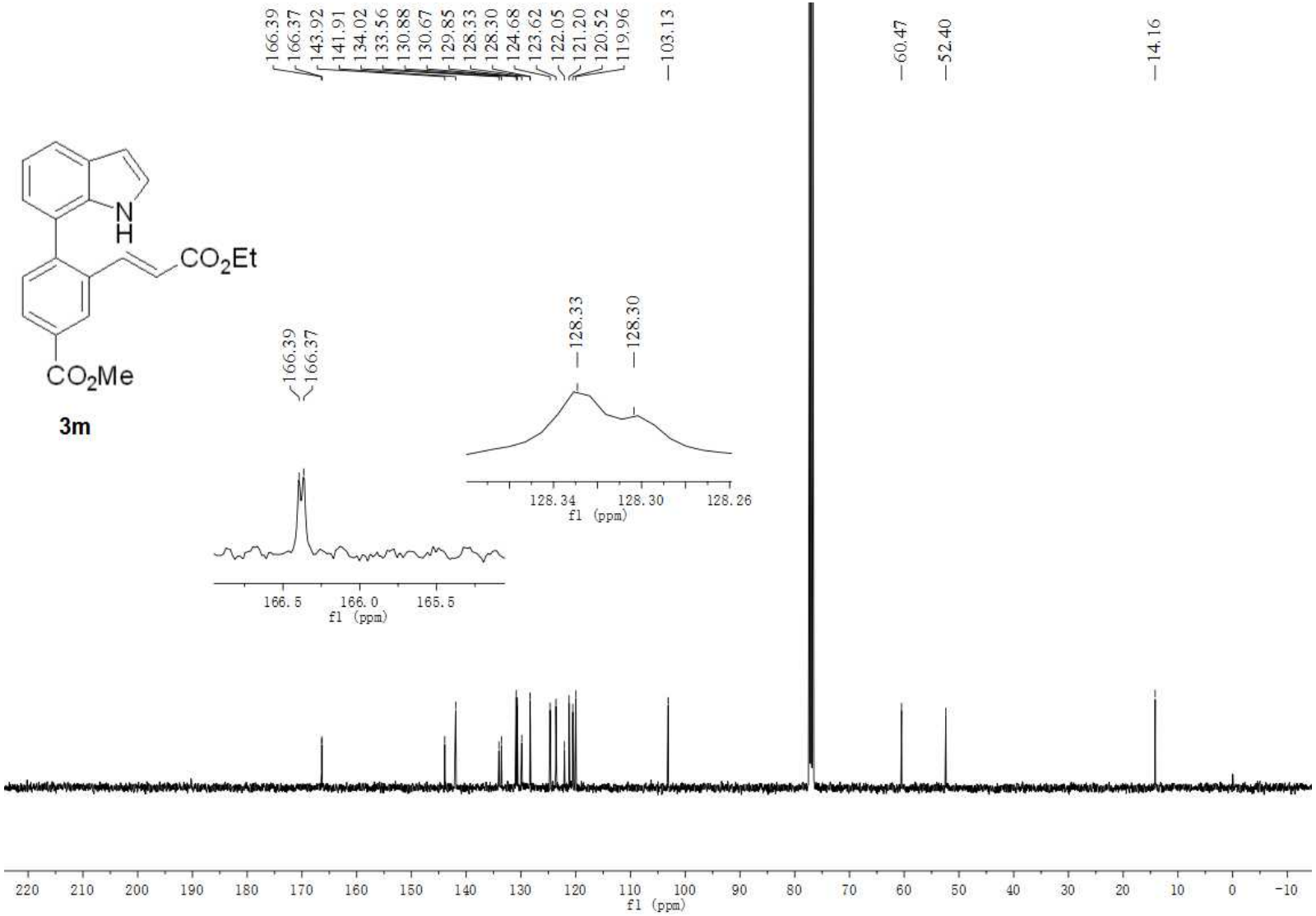
7.72
7.72
7.70
7.70
7.70
7.61
7.59
7.58
7.54
7.23
7.21
7.21
7.19
7.19
7.19
7.19
7.18
7.05
7.05
7.03
7.03
6.64
6.63
6.63
6.62
6.54
6.50
4.17
4.15
4.13
4.12
3.98
1.24
1.23
1.21
N
H
CO2Et
CO2Me
3m
1.00
1.00
0.91
1.00
2.00
0.98
1.00
2.06
3.00
3.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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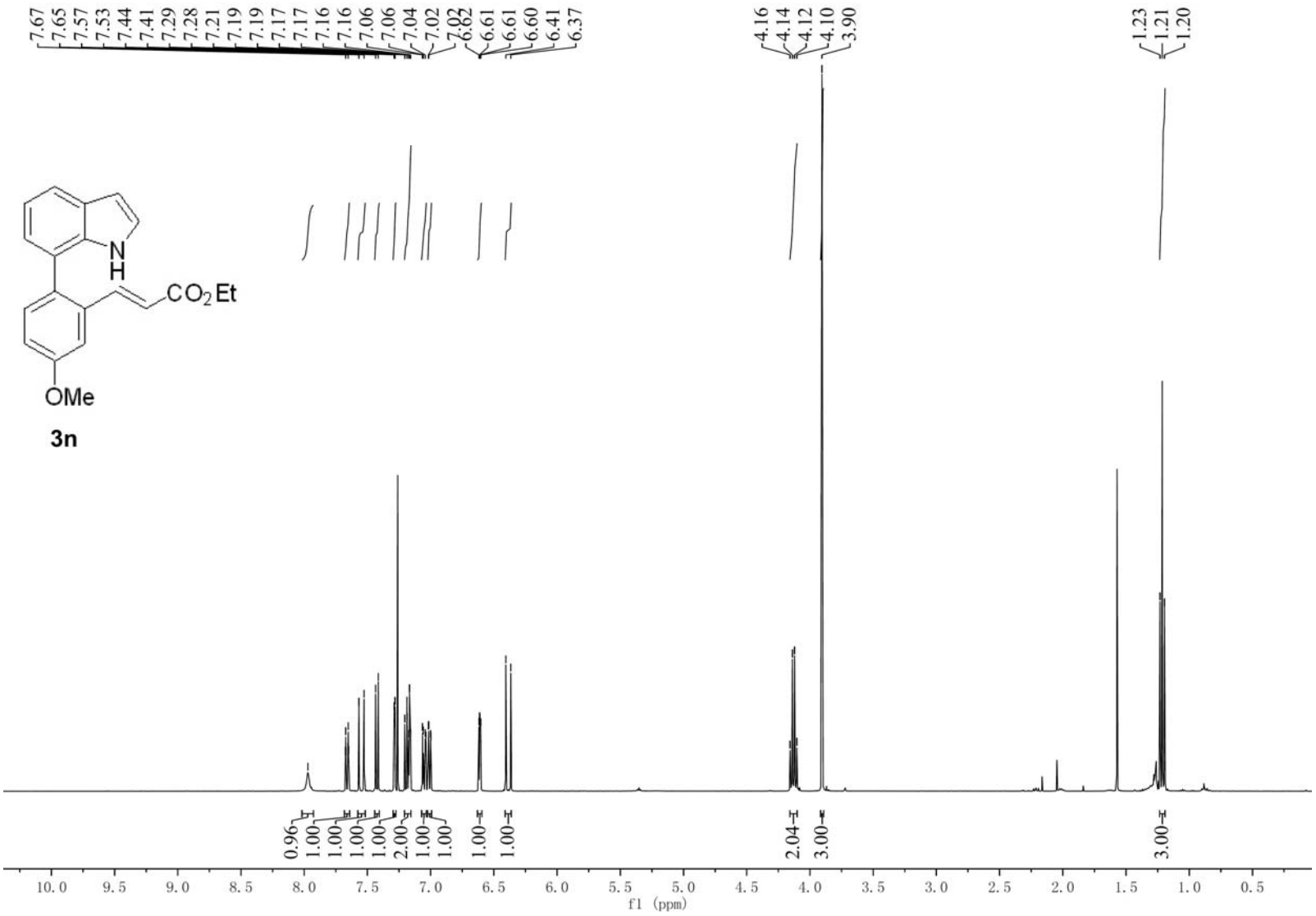
124.68
123.62
122.05
121.20
120.52
119.96
103.13
60.47
52.40
14.16
N
H
CO2Et
CO2Me
3m
166.39
166.37
166.5
166.0
165.5
f1 (ppm)
128.33
128.30
128.34
128.30
128.26
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.19
7.19
7.17
7.17
7.16
7.16
7.06
7.06
7.04
7.02
7.02
6.61
6.61
6.60
6.41
6.37
4.16
4.14
4.12
4.10
3.90
1.23
1.21
1.20
N
H
CO2Et
OMe
3n
0.96
1.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
2.04
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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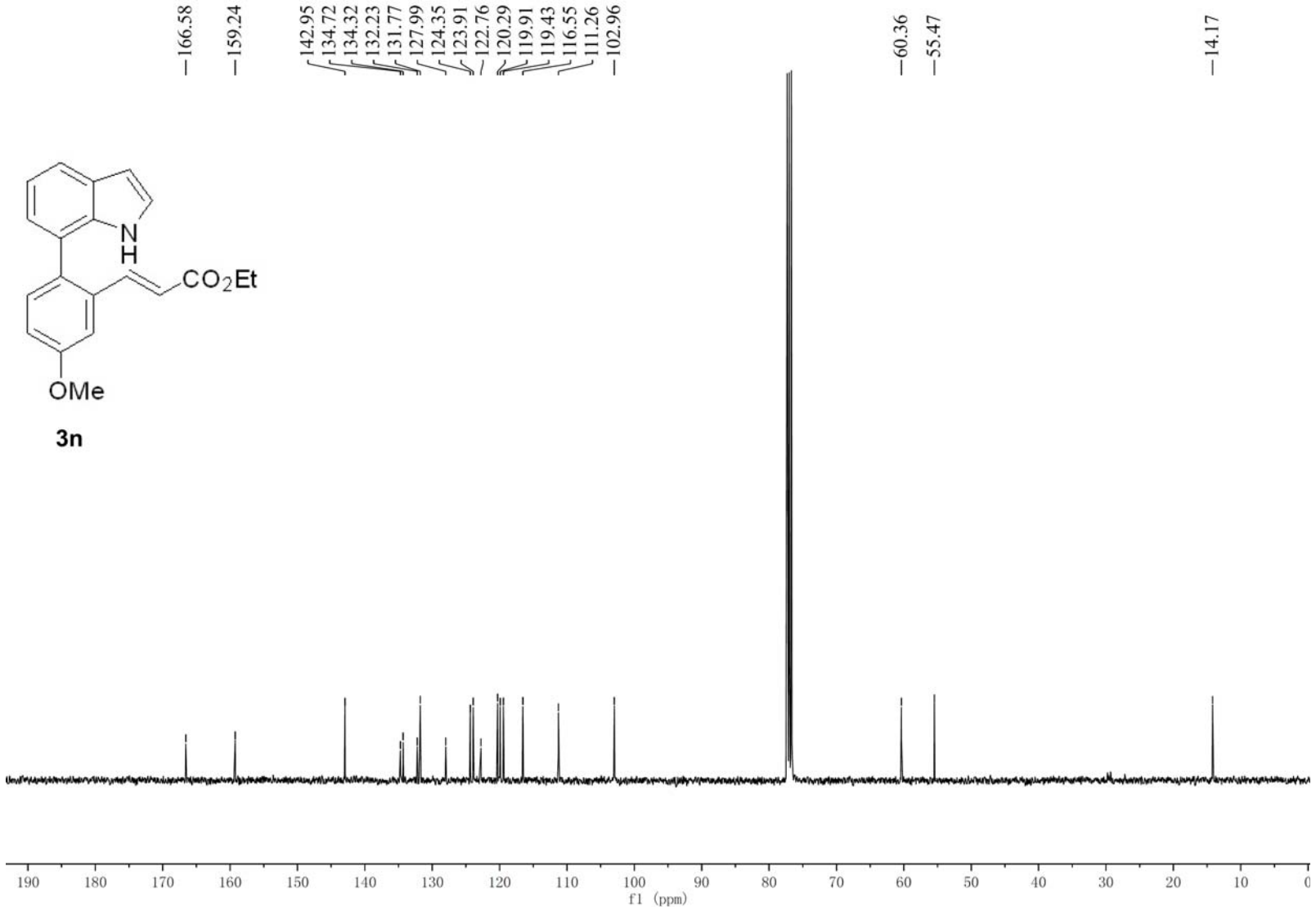
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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131.77
127.99
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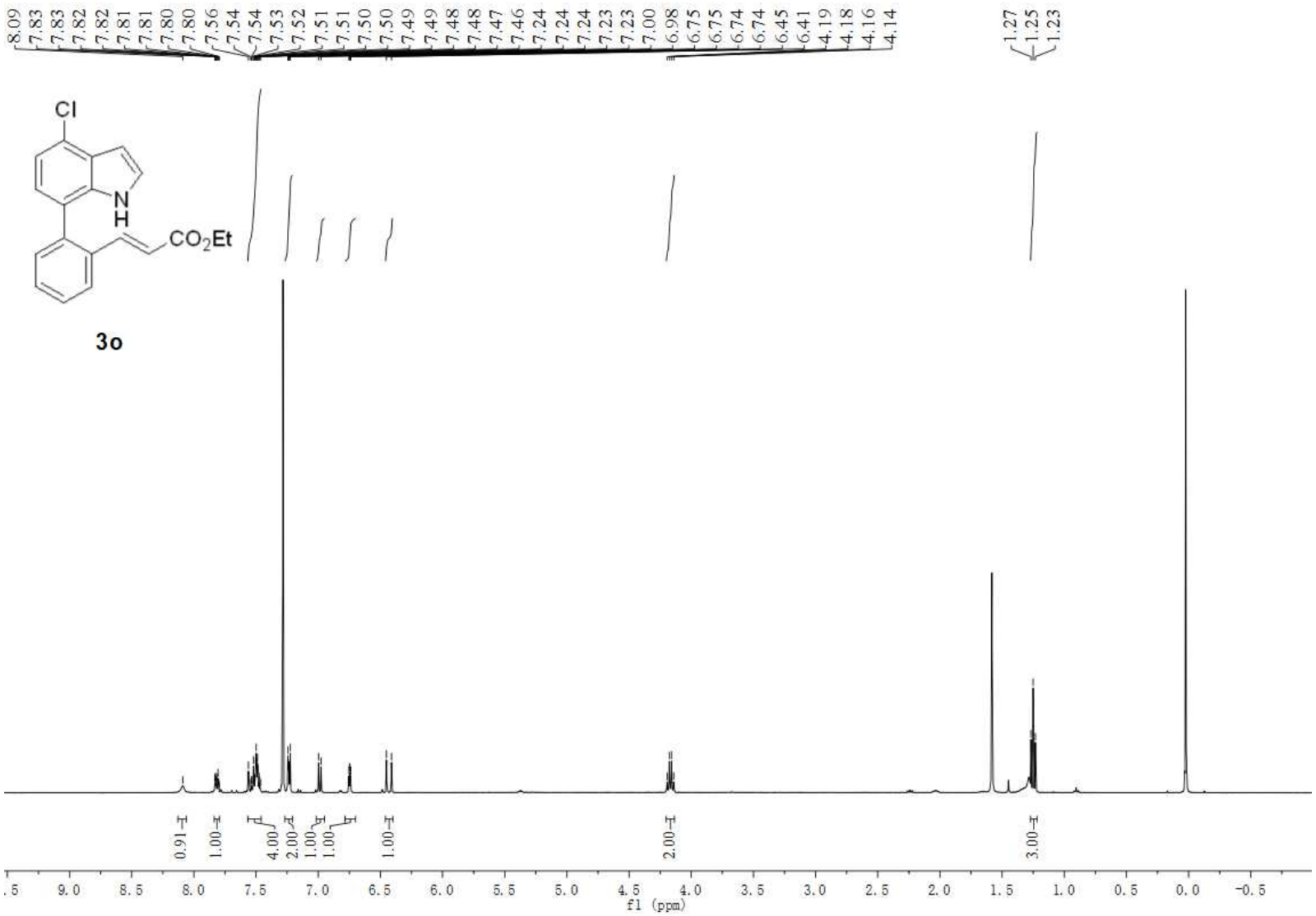
123.91
122.76
120.29
119.91
119.43
116.55
111.26
102.96
60.36
55.47
14.17
N
H
CO2Et
OMe
3n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.53
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7.51
7.51
7.50
7.50
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7.49
7.48
7.48
7.47
7.46
7.24
7.24
7.24
7.23
7.23
7.00
6.98
6.75
6.75
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6.74
6.45
6.41
4.19
4.18
4.16
4.14
1.27
1.25
1.23
Cl
N
H
CO2Et
3o
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4.00
2.00
1.00
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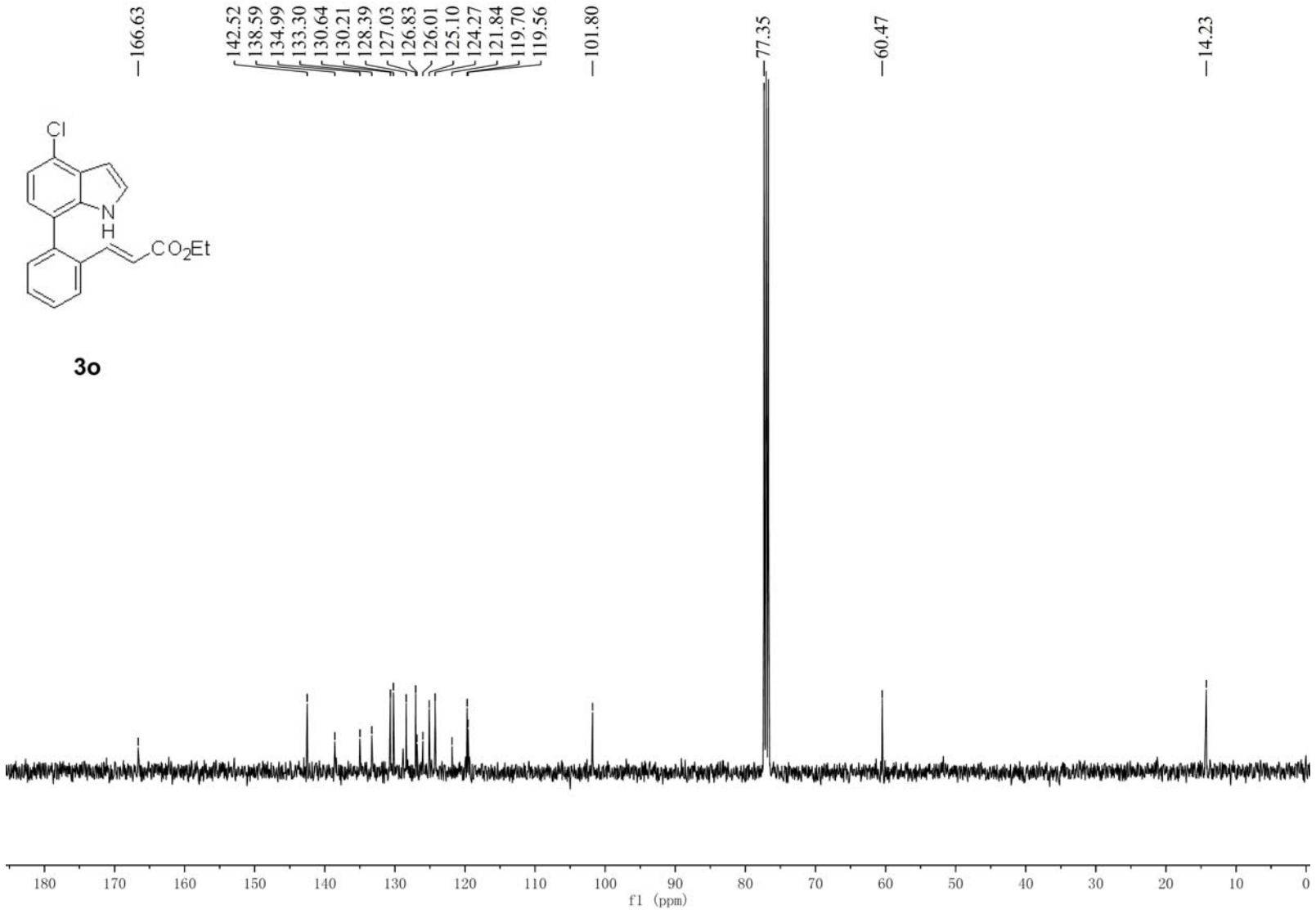
1.00
2.00
3.00
f1 (ppm)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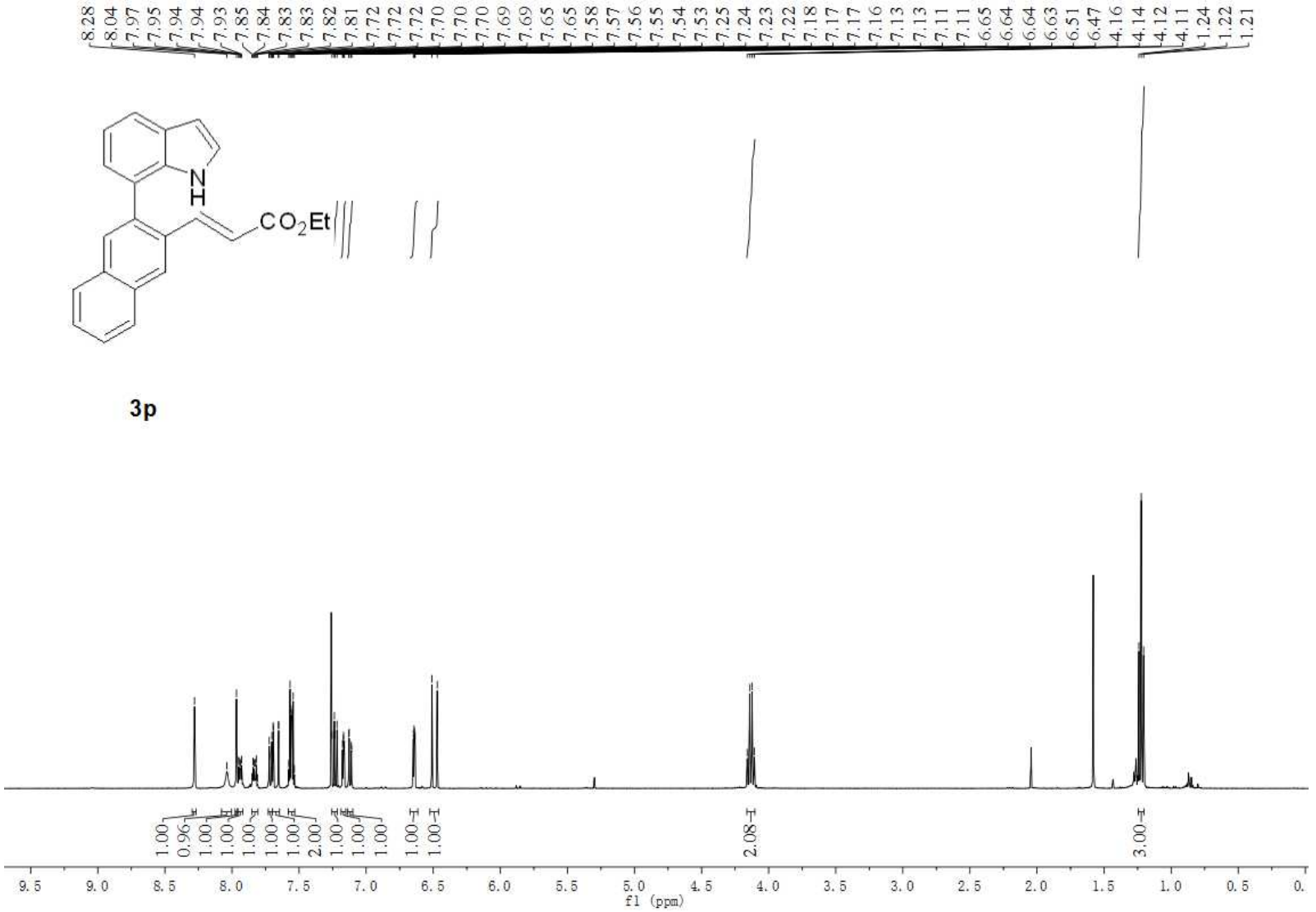
126.83
126.01
125.10
124.27
121.84
119.70
119.56
101.80
77.35
60.47
14.23
Cl
N
H
CO2Et
3o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.83
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7.69
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7.24
7.23
7.22
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7.17
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7.16
7.13
7.13
7.11
7.11
6.65
6.64
6.64
6.63
6.51
6.47
4.16
4.14
4.12
4.11
1.24
1.22
1.21
N
H
CO2Et
1.00
0.96
1.00
1.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
2.08
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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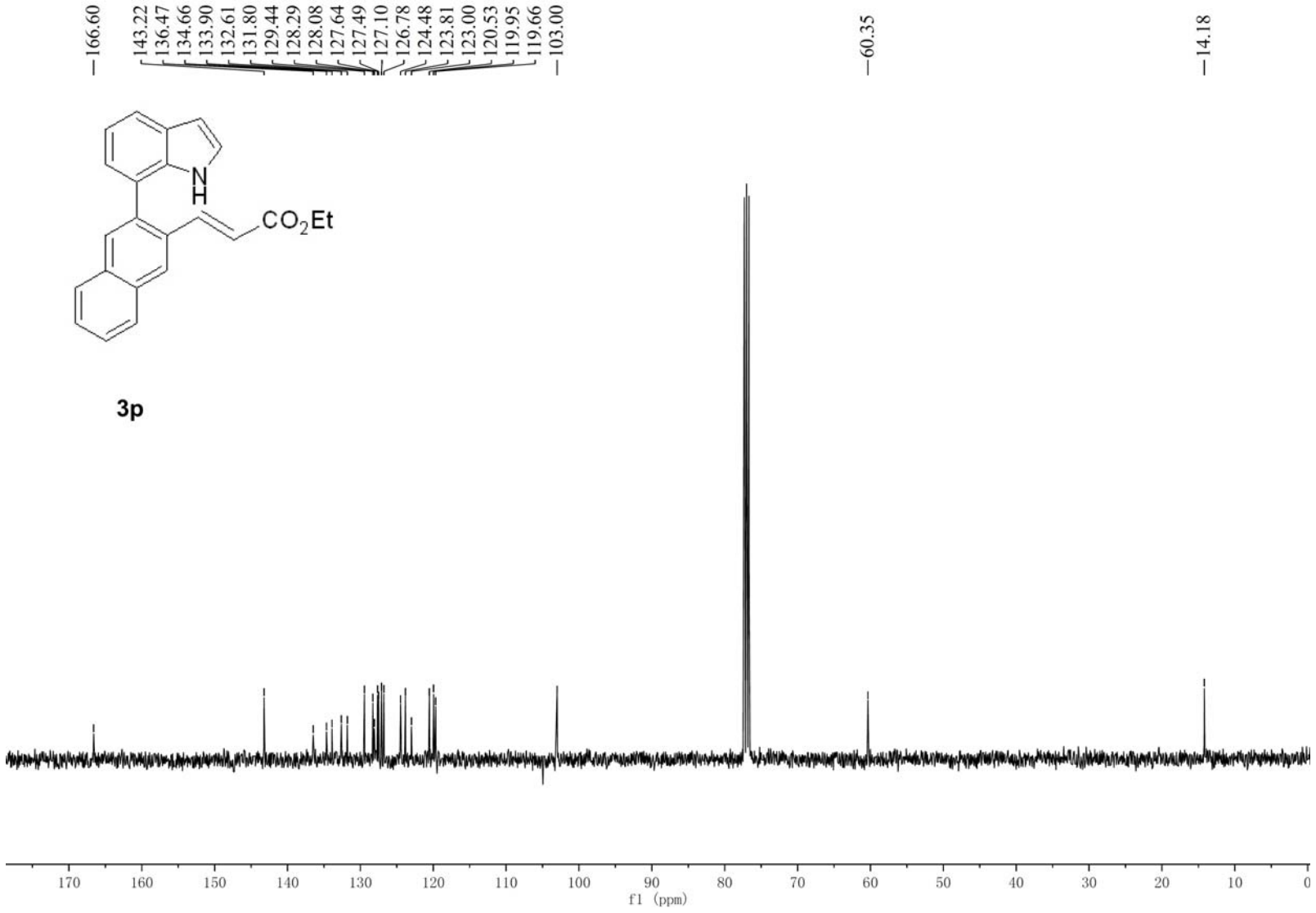
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

3p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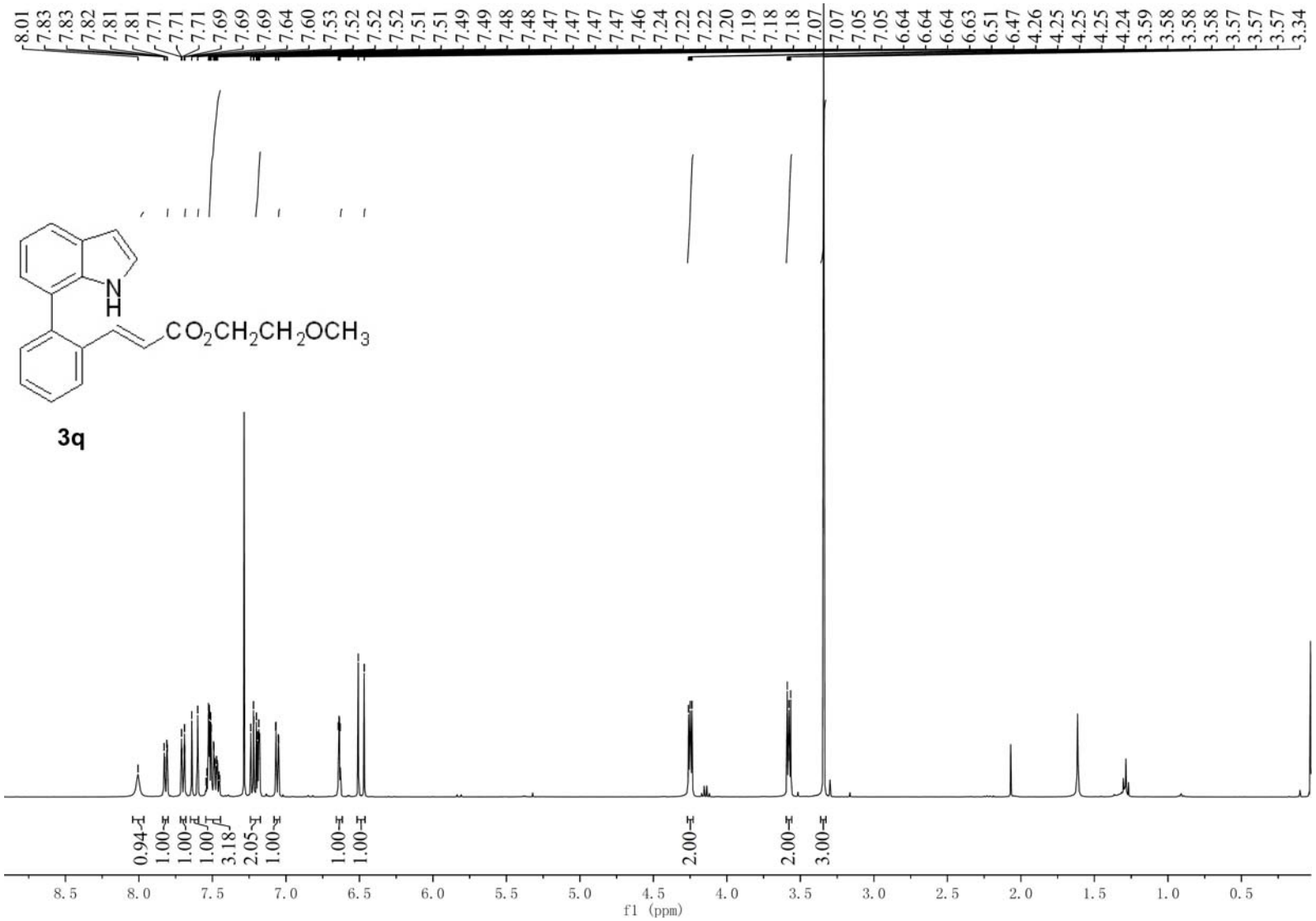
128.29
128.08
127.64
127.49
127.10
126.78
124.48
123.81
123.00
120.53
119.95
119.66
103.00
60.35
14.18
N
H
CO2Et
3p
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.64
7.60
7.53
7.52
7.52
7.52
7.51
7.51
7.49
7.49
7.48
7.48
7.47
7.47
7.47
7.47
7.46
7.24
7.22
7.22
7.20
7.19
7.18
7.18
7.07
7.07
7.05
7.05
6.64
6.64
6.64
6.63
6.51
6.47
4.26
4.25
4.25
4.25
4.24
3.59
3.58
3.58
3.58
3.57
3.57
3.57
3.34
N
H
CO2CH2CH2OCH3
3q
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1.00
1.00
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2.05
1.00
1.00
1.00
2.00
2.00
3.00
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8.0
7.5
7.0
6.5
6.0
5.5
5.0
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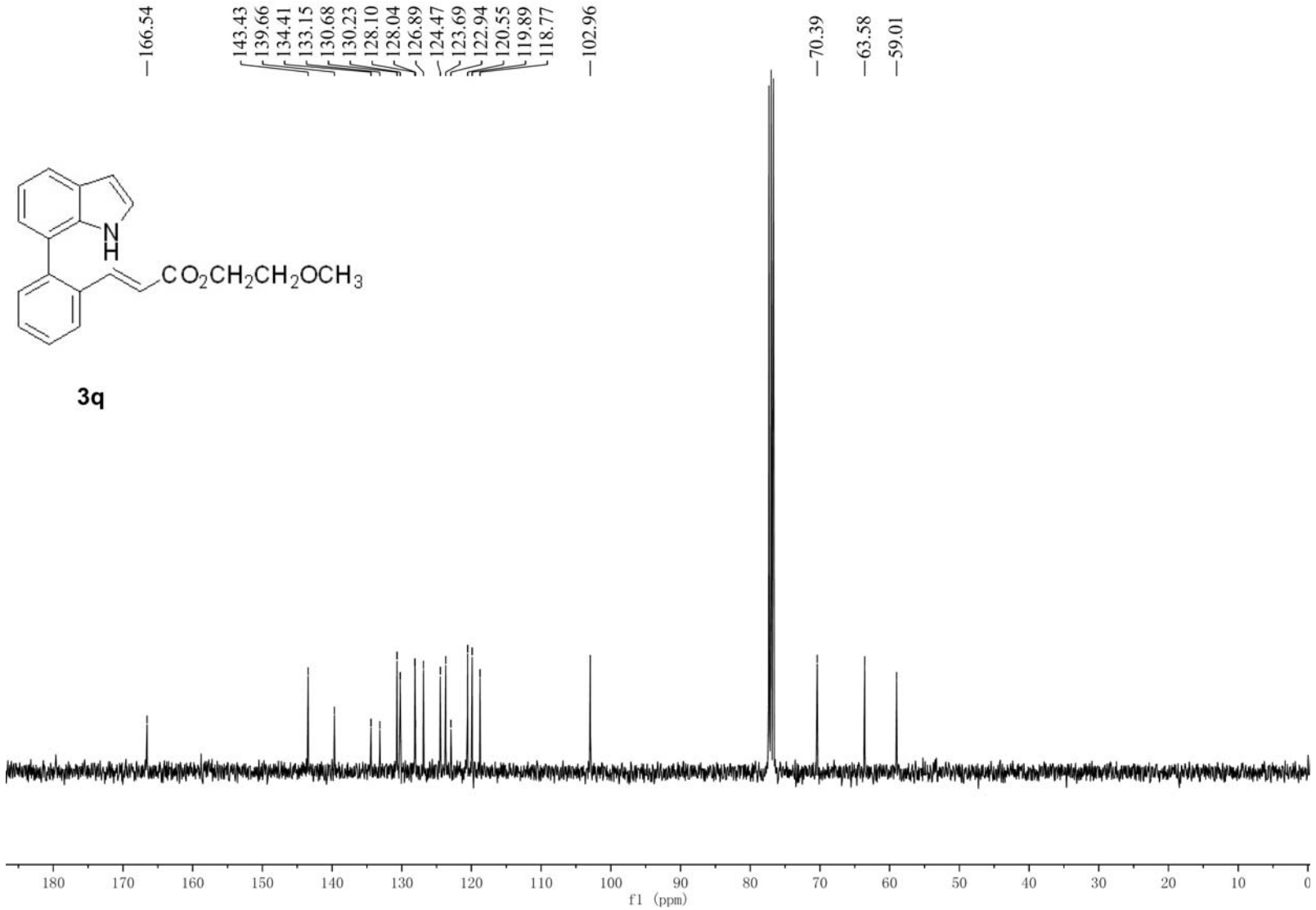
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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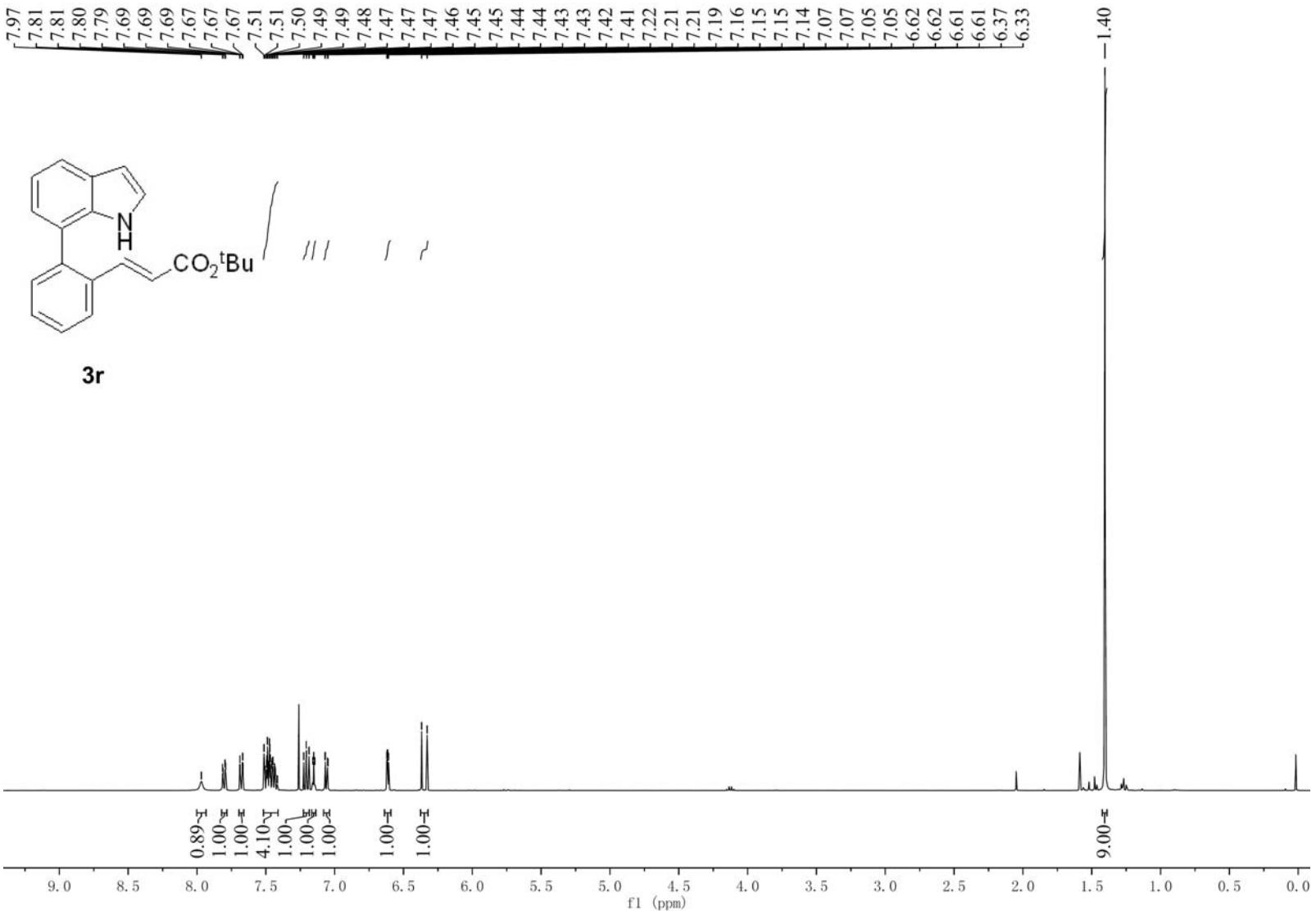
128.10
128.04
126.89
124.47
123.69
122.94
120.55
119.89
118.77
102.96
70.39
63.58
59.01
N
H
CO2CH2CH2OCH3
3q
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.67
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7.50
7.49
7.49
7.48
7.47
7.47
7.47
7.46
7.45
7.45
7.44
7.44
7.43
7.43
7.42
7.41
7.22
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7.21
7.19
7.16
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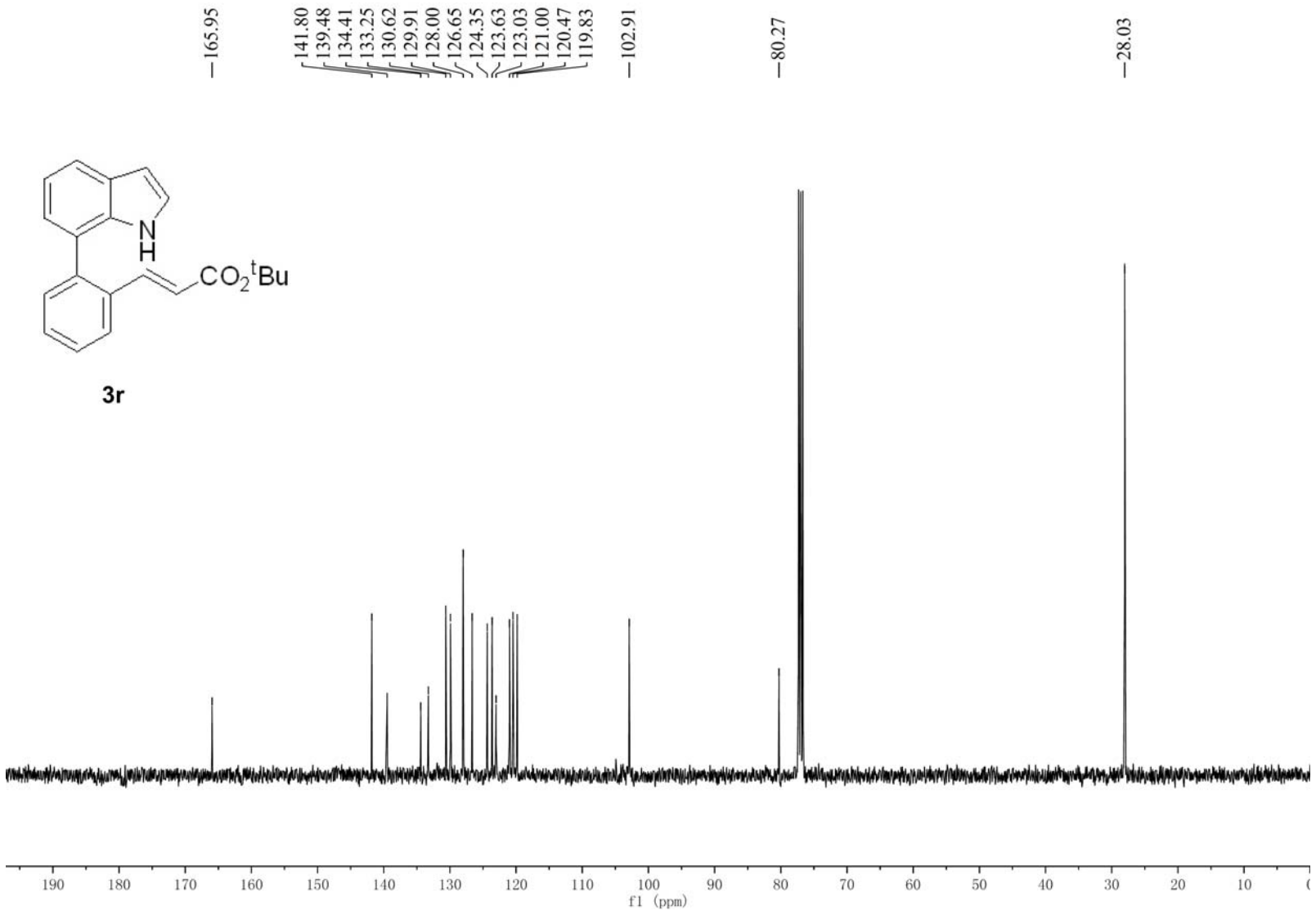
7.14
7.07
7.07
7.05
7.05
6.62
6.62
6.61
6.61
6.37
6.33
1.40
N
H
CO2tBu
3r
0.89
1.00
1.00
4.10
1.00
1.00
1.00
1.00
1.00
9.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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134.41
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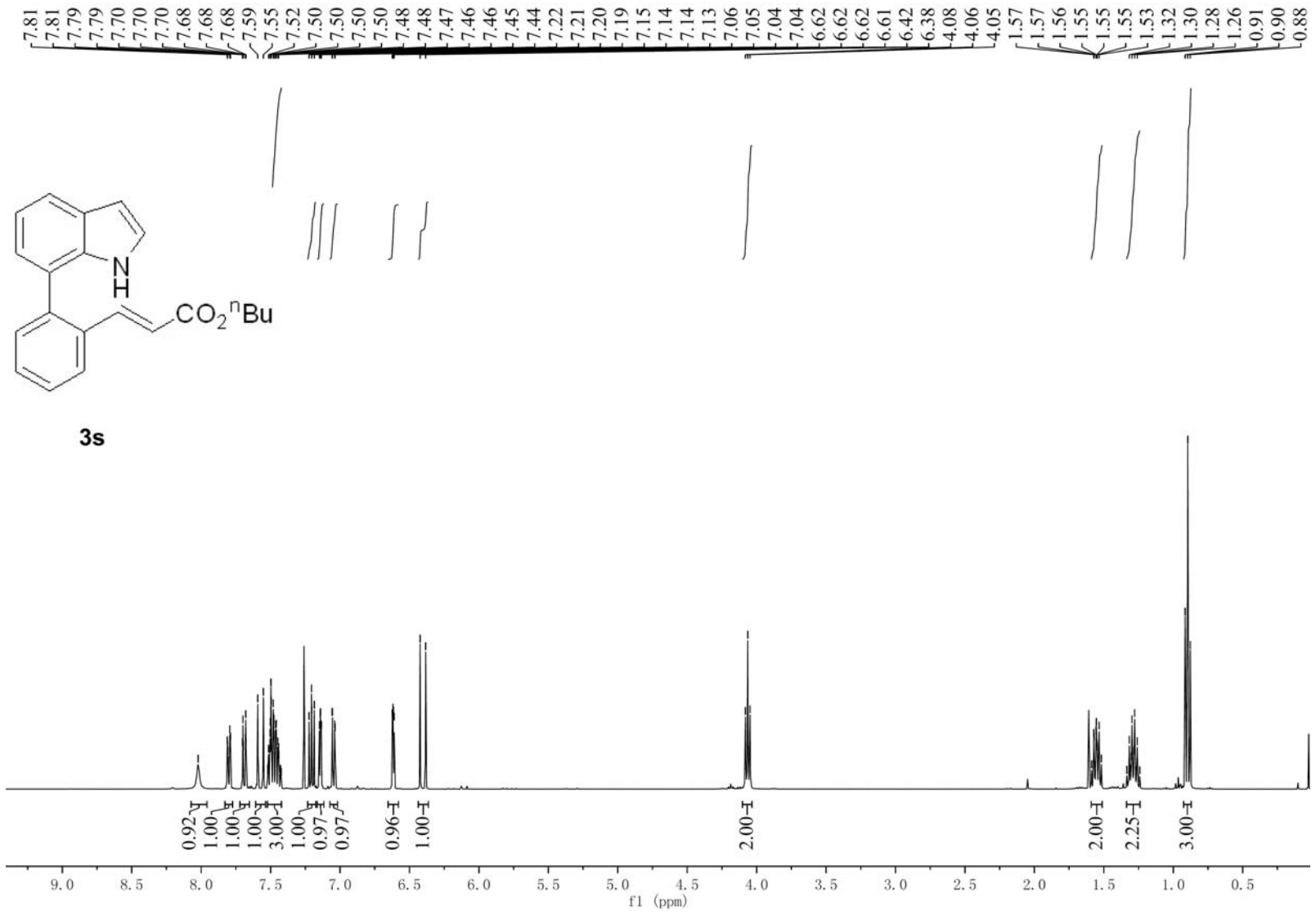
129.91
128.00
126.65
124.35
123.63
123.03
121.00
120.47
119.83
102.91
80.27
28.03
N
H
CO2tBu
3r
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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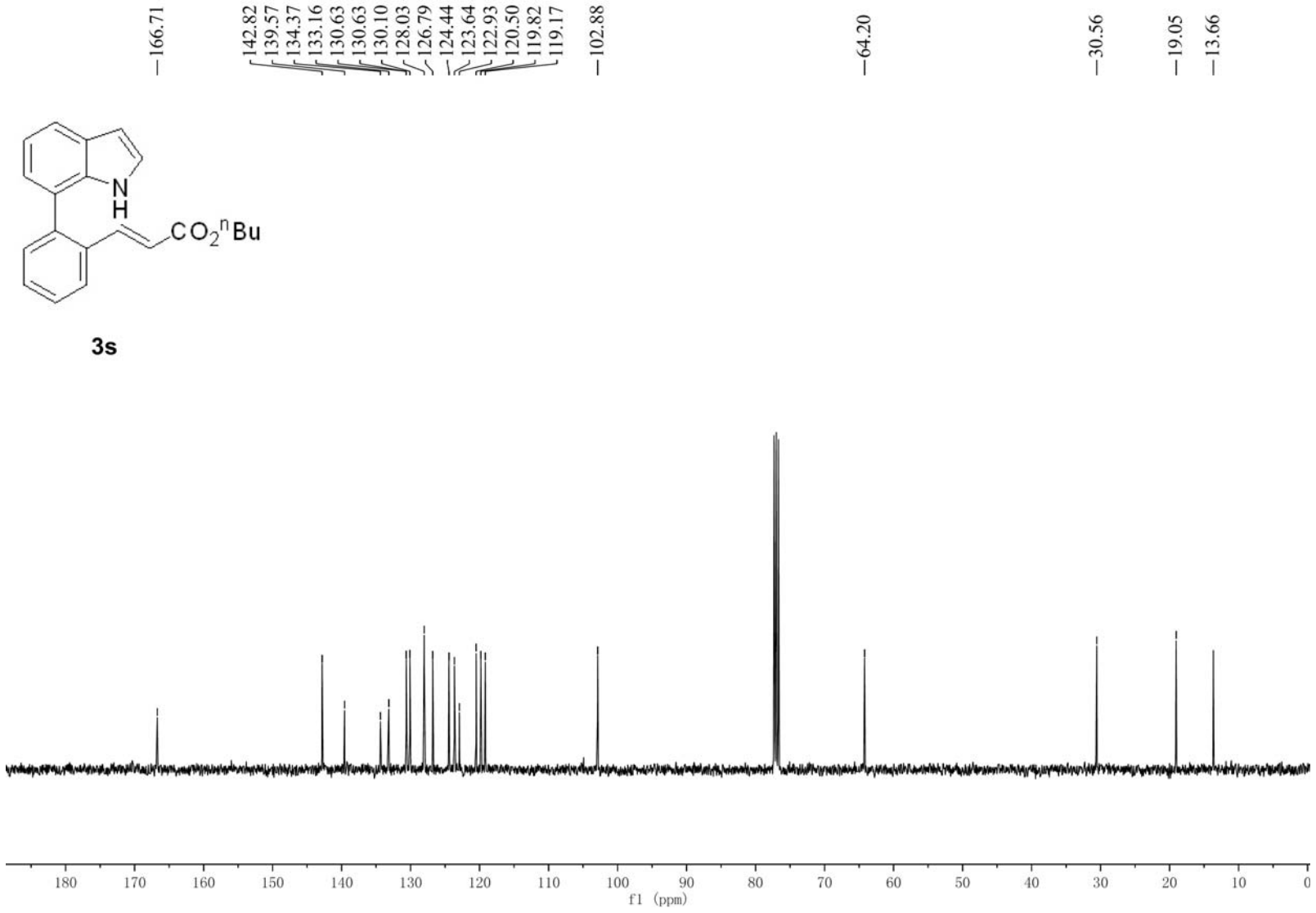
N
H
CO2nBu
3s
0.92 1.00 1.00 1.00 3.00 1.00 0.97 0.97 0.96 1.00 2.00 2.00 2.25 3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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124.44
123.64
122.93
120.50
119.82
119.17
102.88
64.20
30.56
19.05
13.66
N
H
CO2nBu
3s
180
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130
120
110
100
90
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70
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50
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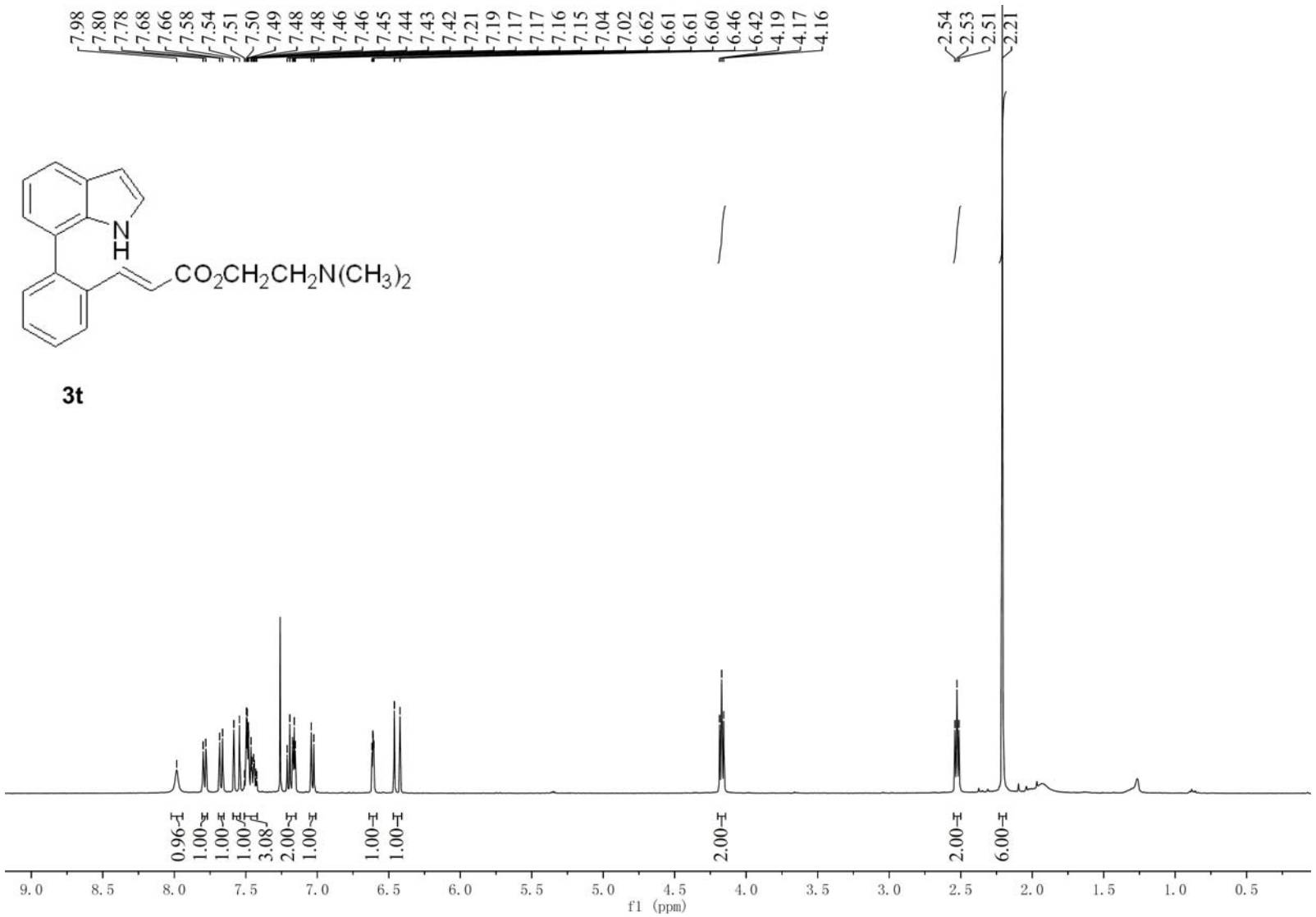
0
f1 (ppm)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7.44
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7.42
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7.17
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7.16
7.15
7.04
7.02
6.62
6.61
6.61
6.60
6.46
6.42
4.19
4.17
4.16
2.54
2.53
2.51
2.21
N
H
CO2CH2CH2N(CH3)2
3t
0.96
1.00
1.00
1.00
3.08
2.00
1.00
1.00
1.00
2.00
2.00
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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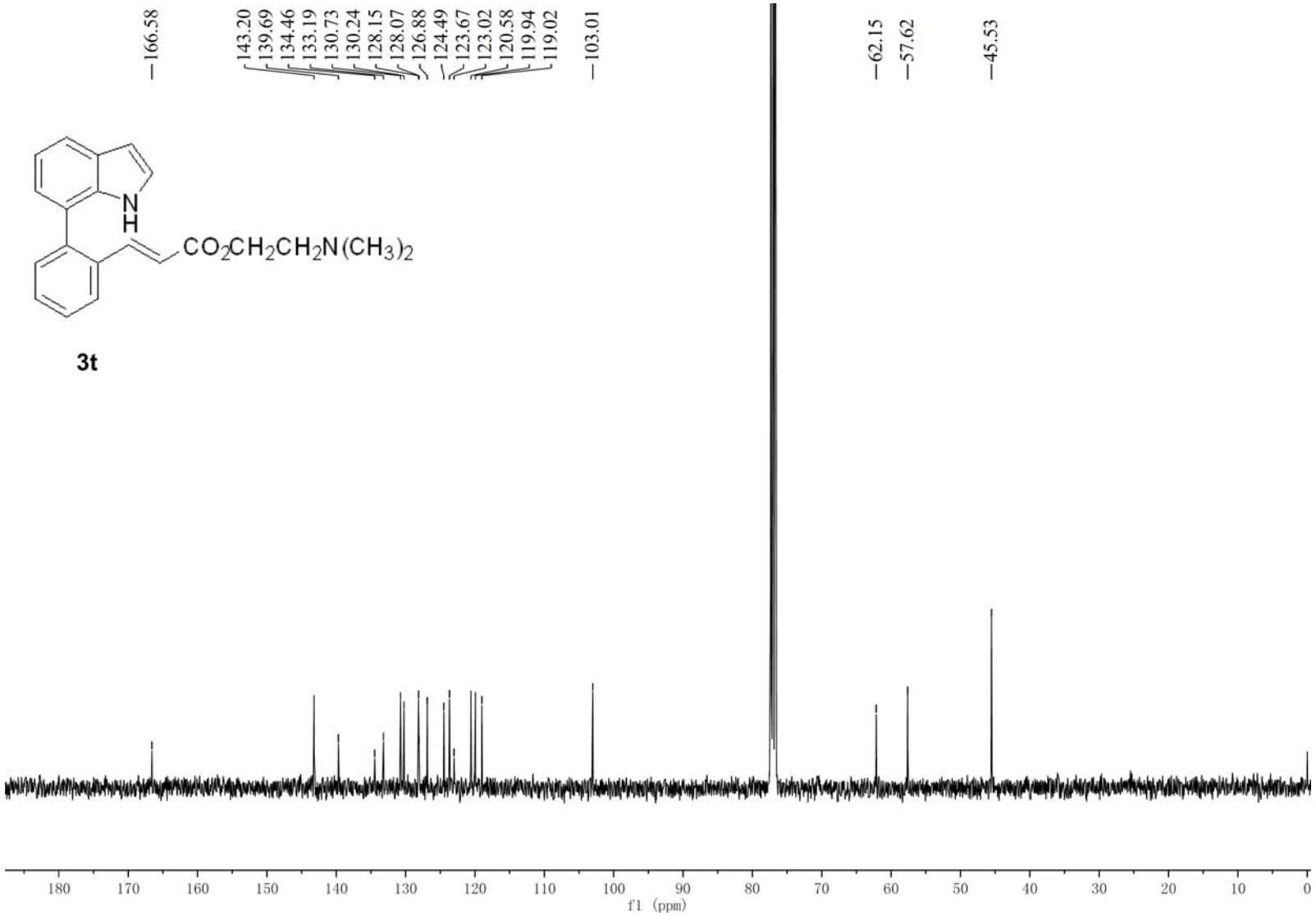
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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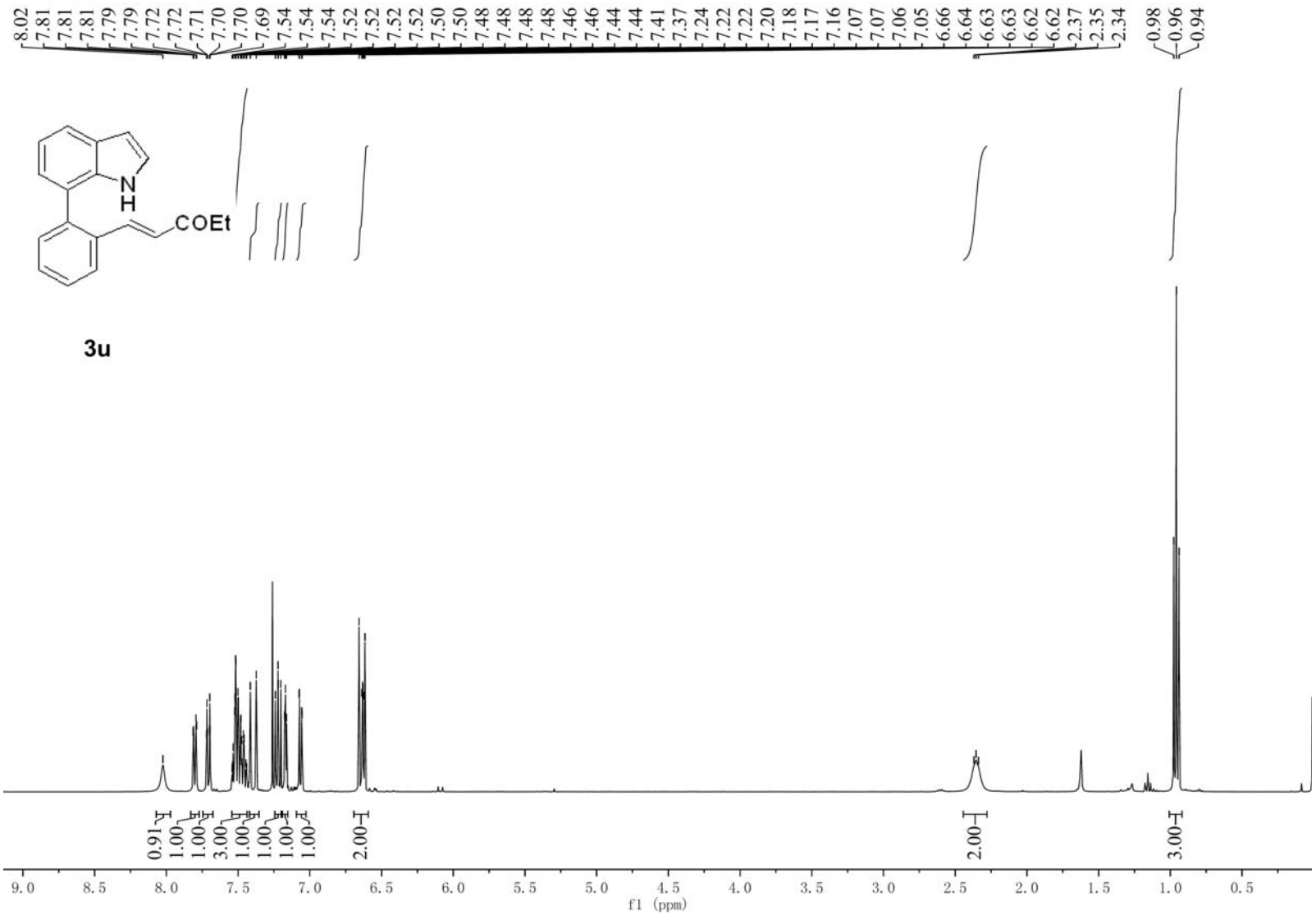
123.02
120.58
119.94
119.02
103.01
62.15
57.62
45.53
N
H
CO2CH2CH2N(CH3)2
3t
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.48
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7.46
7.44
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7.24
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7.17
7.16
7.07
7.07
7.06
7.05
6.66
6.64
6.63
6.63
6.62
6.62
2.37
2.35
2.34
0.98
0.96
0.94
N
H
COEt
3u
0.91
1.00
1.00
3.00
1.00
1.00
1.00
1.00
2.00
2.00
3.00
9.0
8.5
8.0
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7.0
6.5
6.0
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3.0
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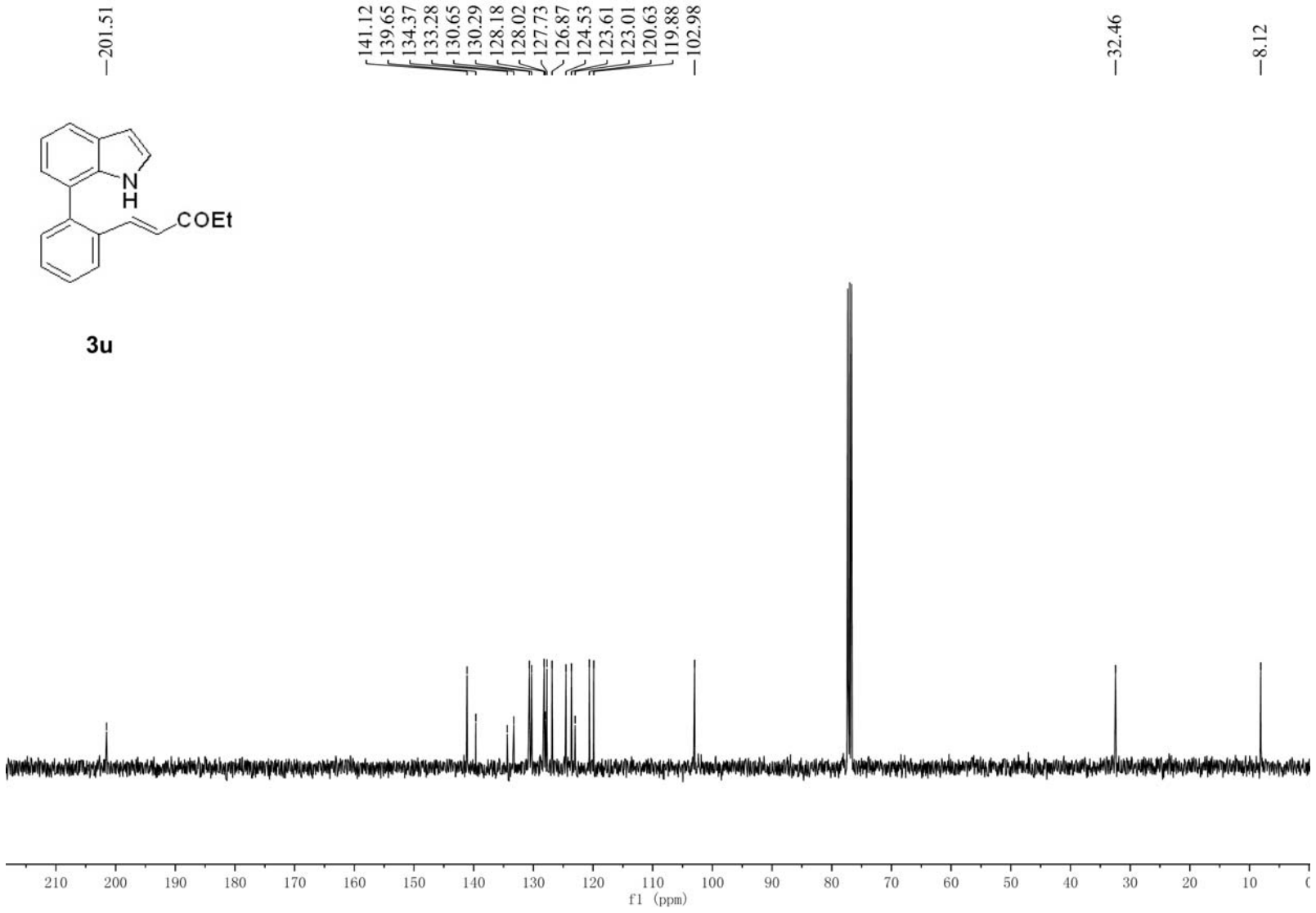
0.5
f1 (ppm)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128.18
128.02
127.73
126.87
124.53
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123.01
120.63
119.88
102.98
32.46
8.12
N
H
COEt
3u
210
200
190
180
170
160
150
140
130
120
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100
90
80
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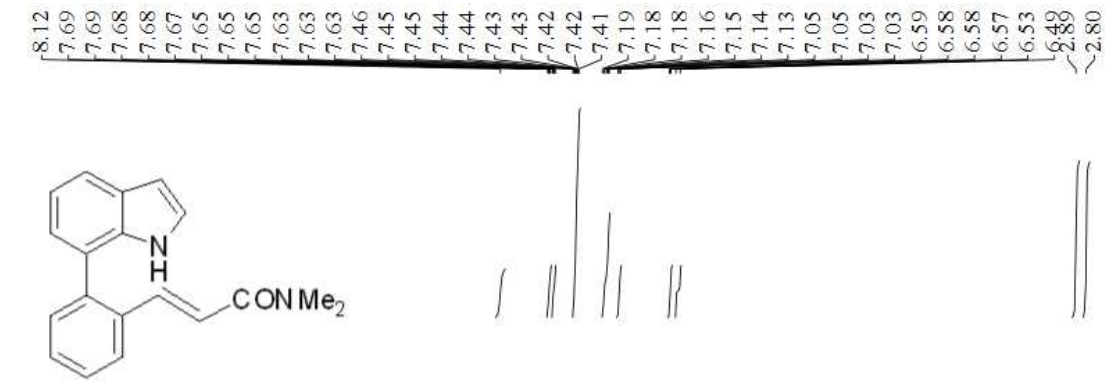
60
50
40
30
20
10
f1 (ppm)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7.05
7.05
7.03
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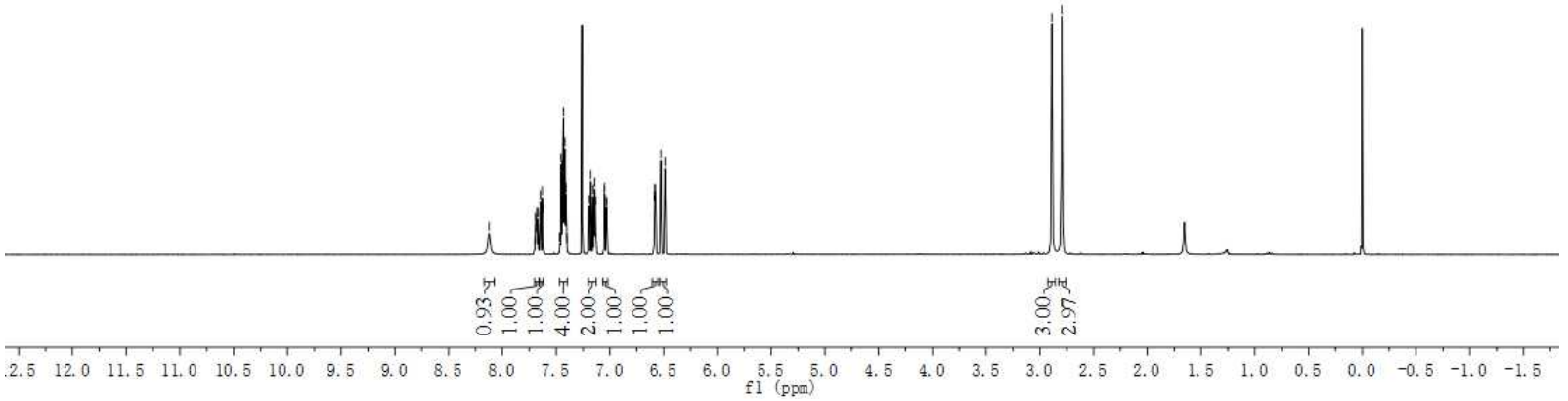
N
H
CONMe2

3v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1.00
1.00
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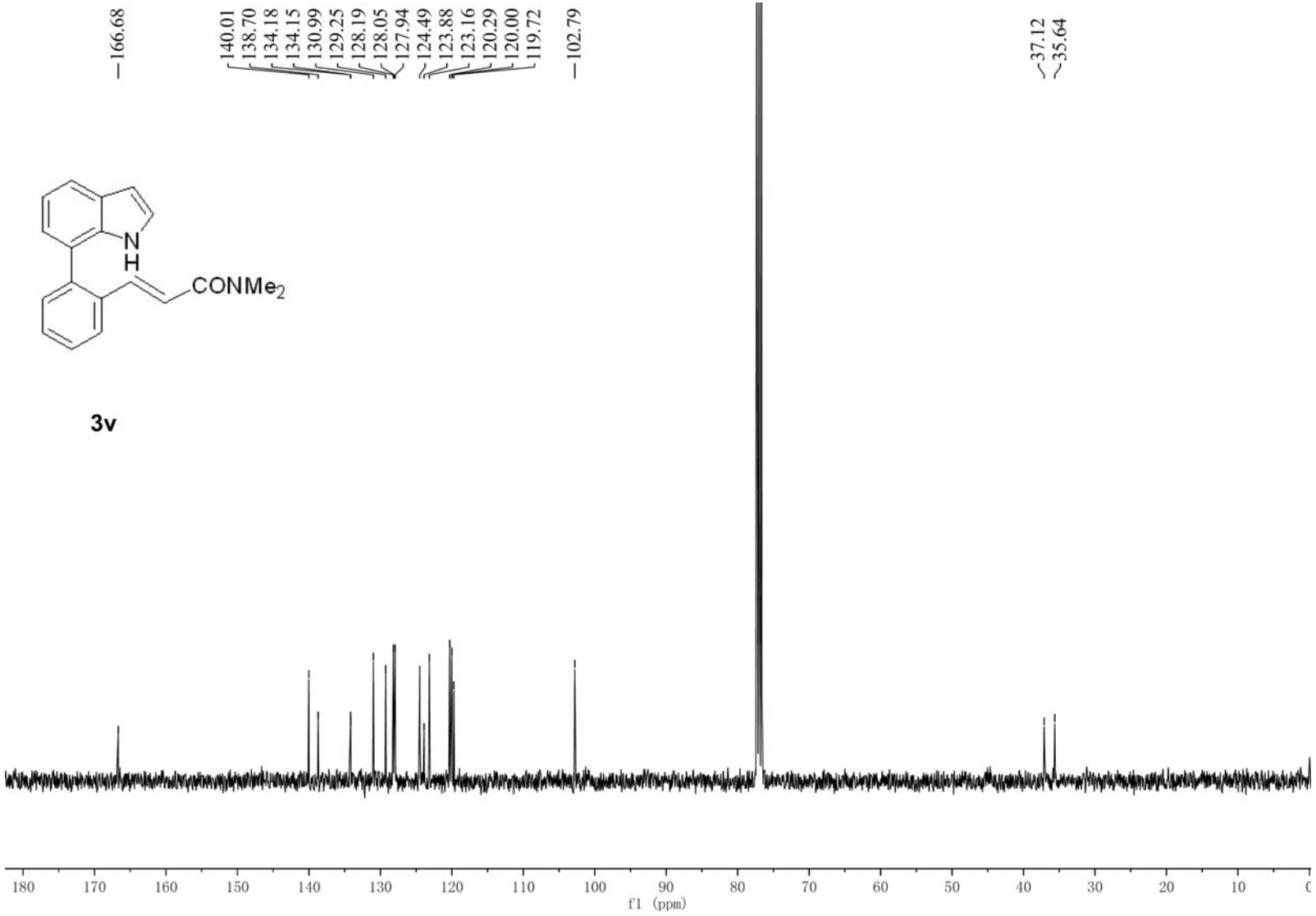
2.97
f1 (ppm)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128.19
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123.16
120.29
120.00
119.72
102.79
37.12
35.64
N
H
CONMe2
3v
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140
130
120
110
100
90
80
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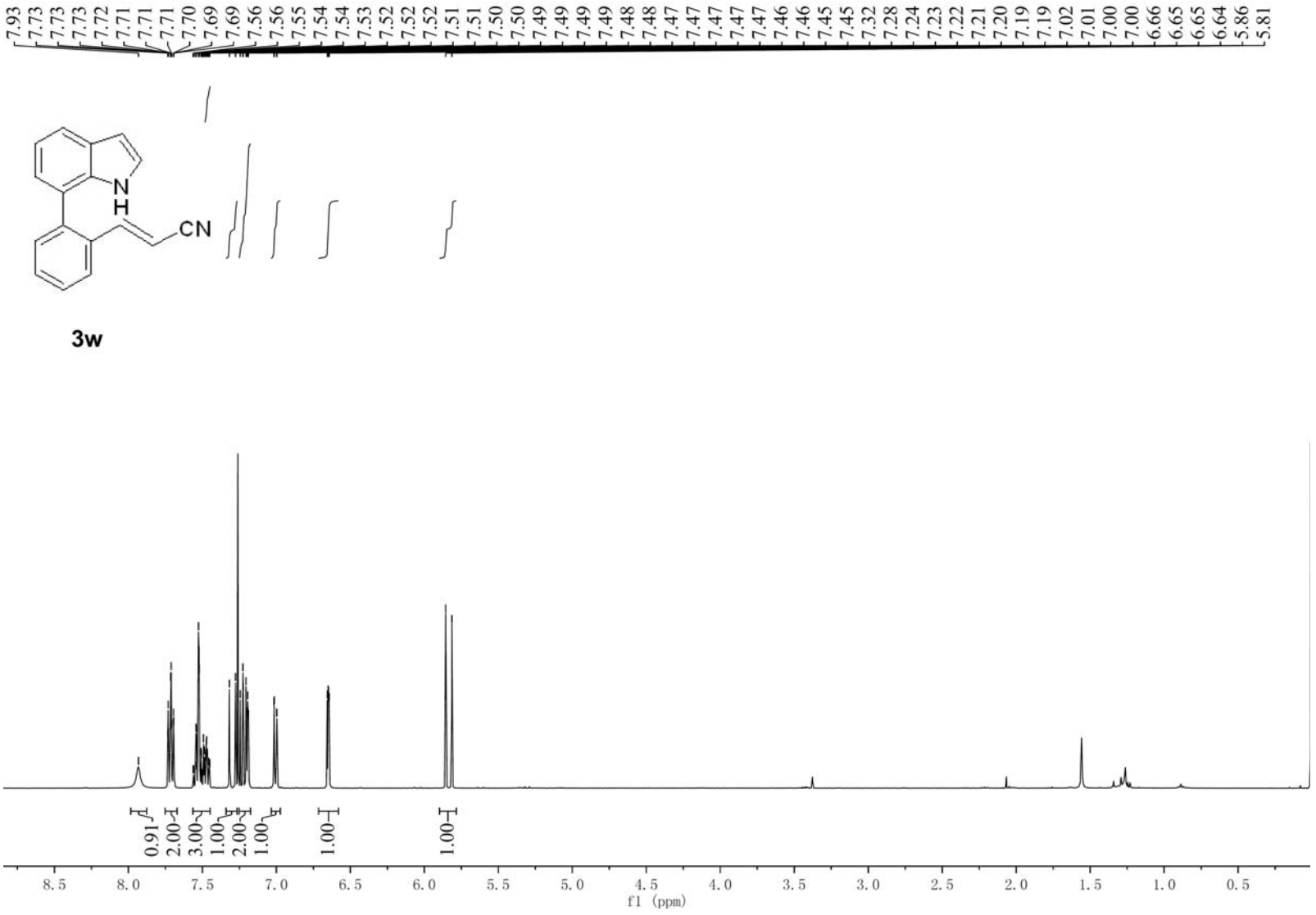
60
50
40
30
20
10
f1 (ppm)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7.19
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7.00
7.00
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6.65
6.65
6.64
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5.81
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H
CN
0.91
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1.00
2.00
1.00
1.00
1.00
8.5
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7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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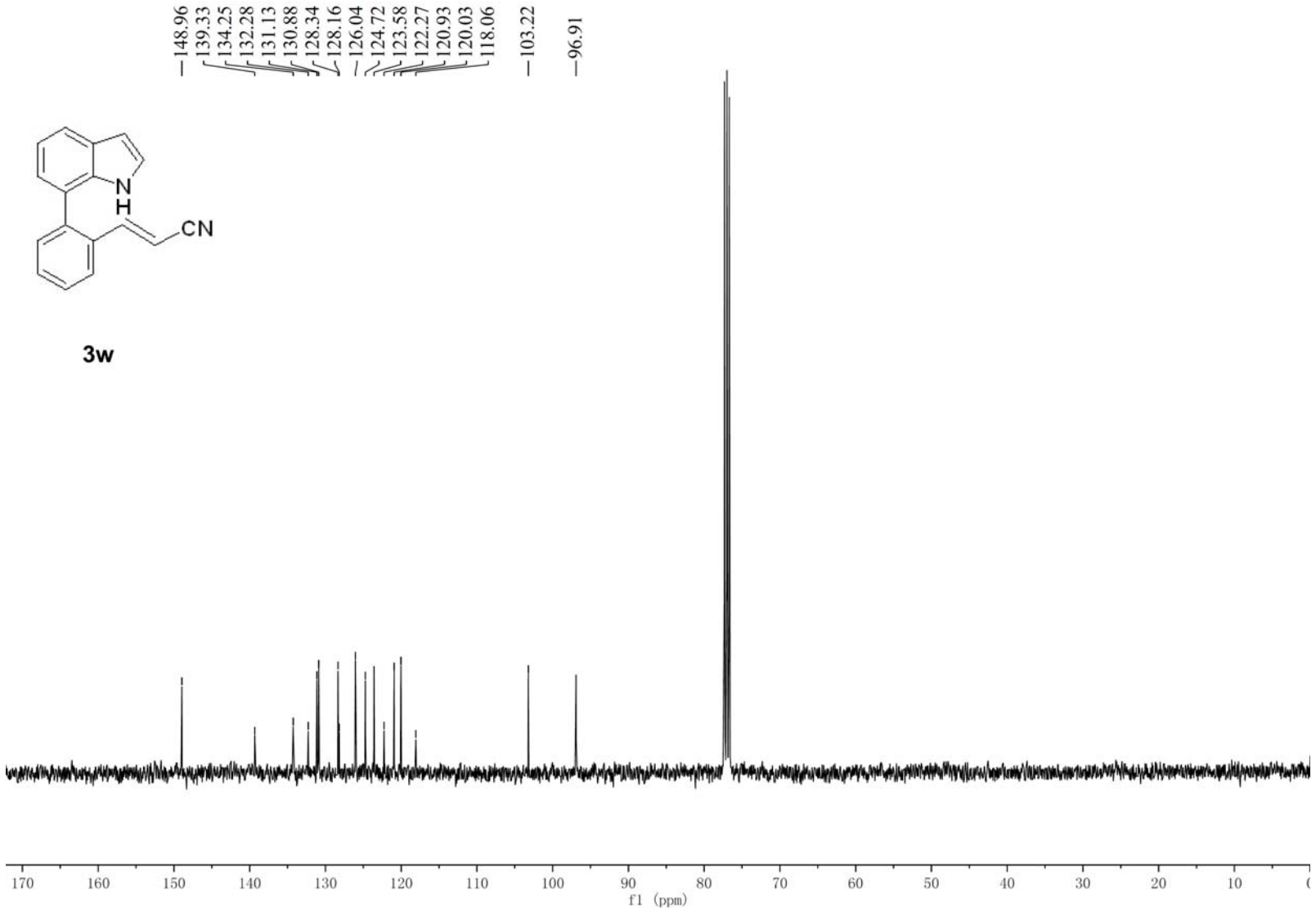
1.0
0.5
f1 (ppm)

3w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123.58
122.27
120.93
120.03
118.06
103.22
96.91
N
H
CN
3w
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140
130
120
110
100
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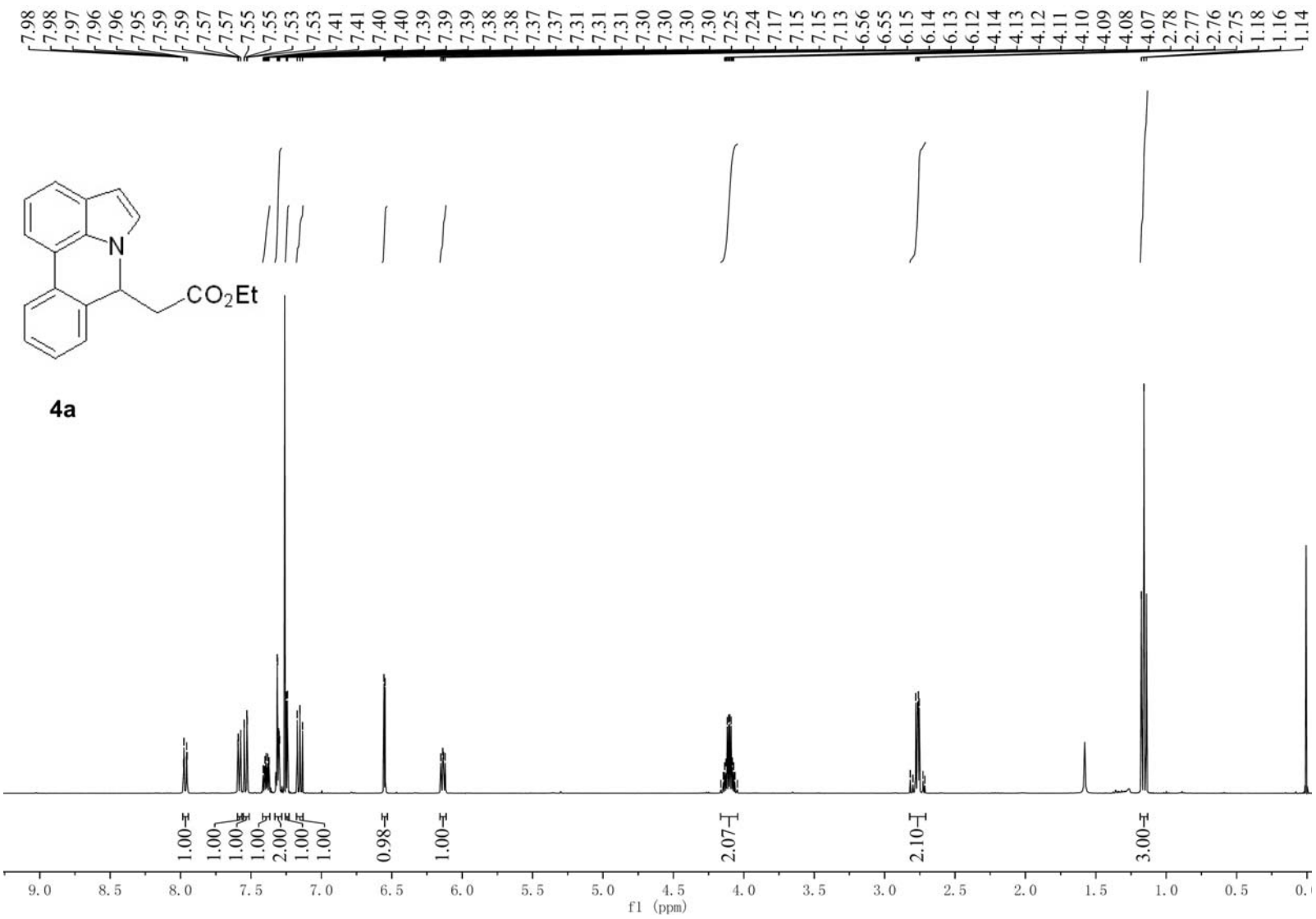
60
50
40
30
20
10
f1 (ppm)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7.37
7.31
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7.24
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7.15
7.13
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6.15
6.14
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6.12
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4.10
4.09
4.08
4.07
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2.77
2.76
2.75
1.18
1.16
1.14
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CO2Et
4a
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1.00
1.00
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1.00
1.00
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1.00
2.07
2.10
3.00
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8.0
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3.0
2.5
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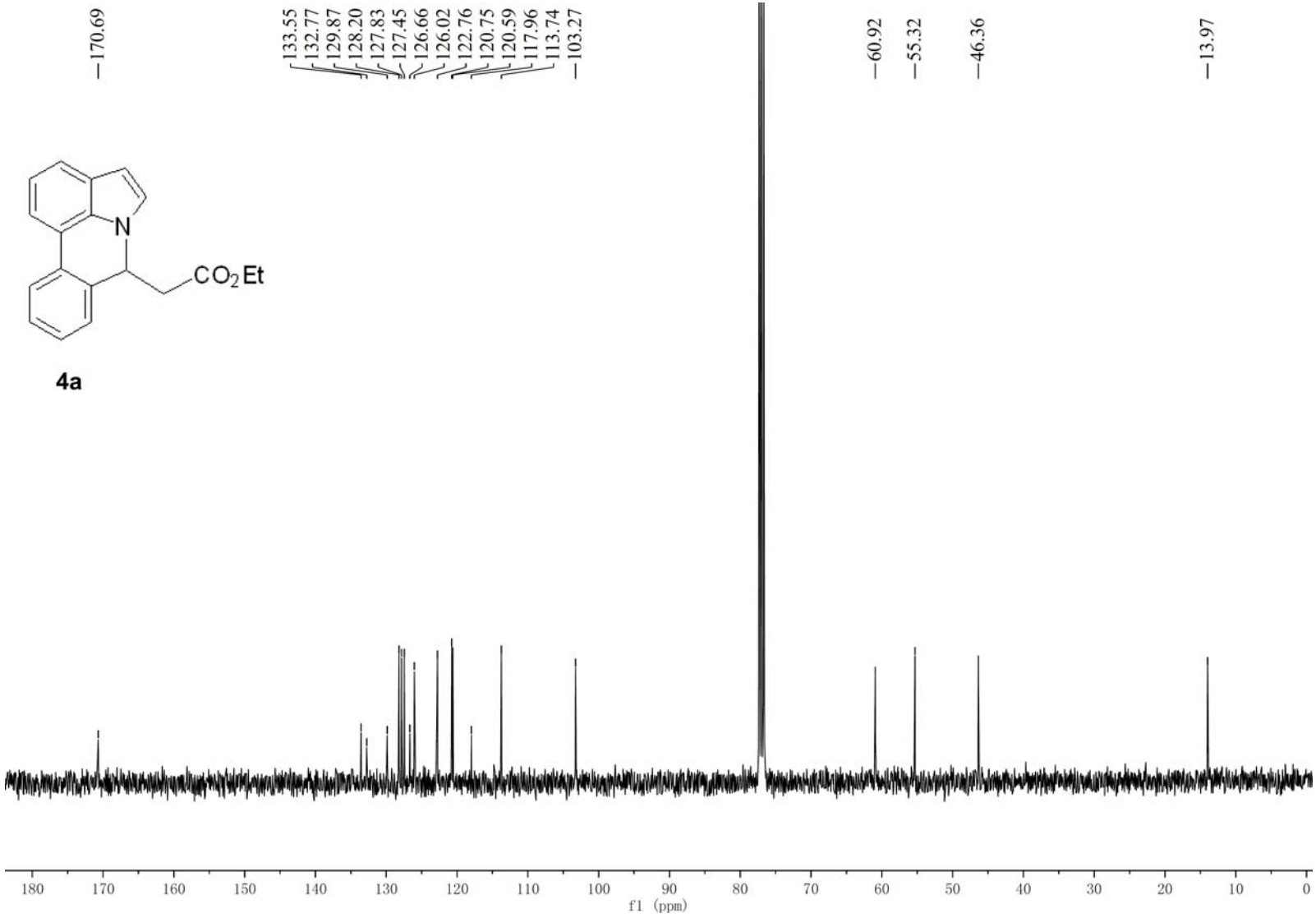
0.5
f1 (ppm)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120.59
117.96
113.74
103.27
60.92
55.32
46.36
13.97
N
CO2Et
4a
180
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110
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90
80
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50
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10
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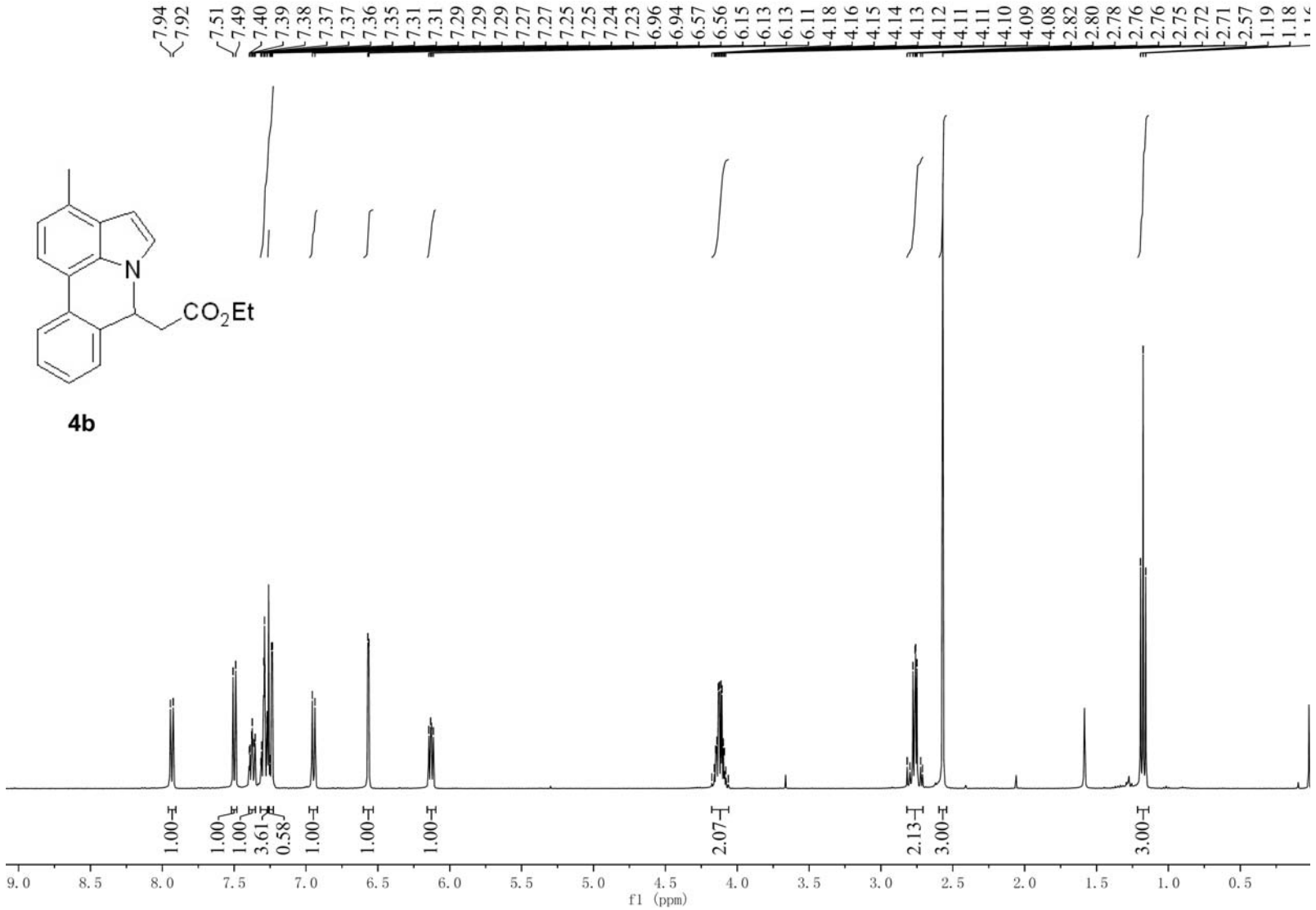
f1 (ppm)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4.13
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3.00
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3.0
2.5
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1.5
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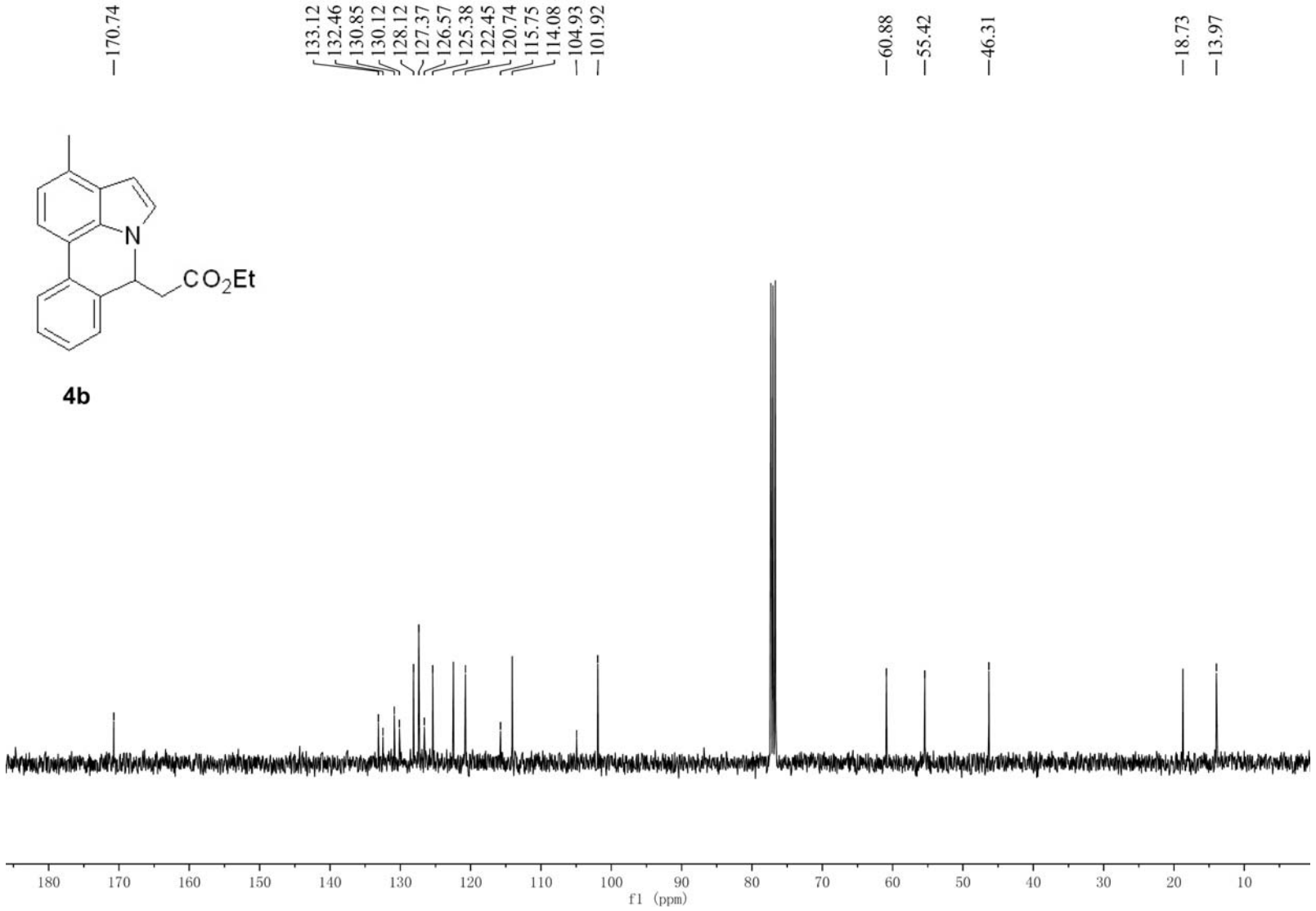
0.5
f1 (ppm)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114.08
104.93
101.92
60.88
55.42
46.31
18.73
13.97
N
CO2Et
4b
180
170
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140
130
120
110
100
90
80
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60
50
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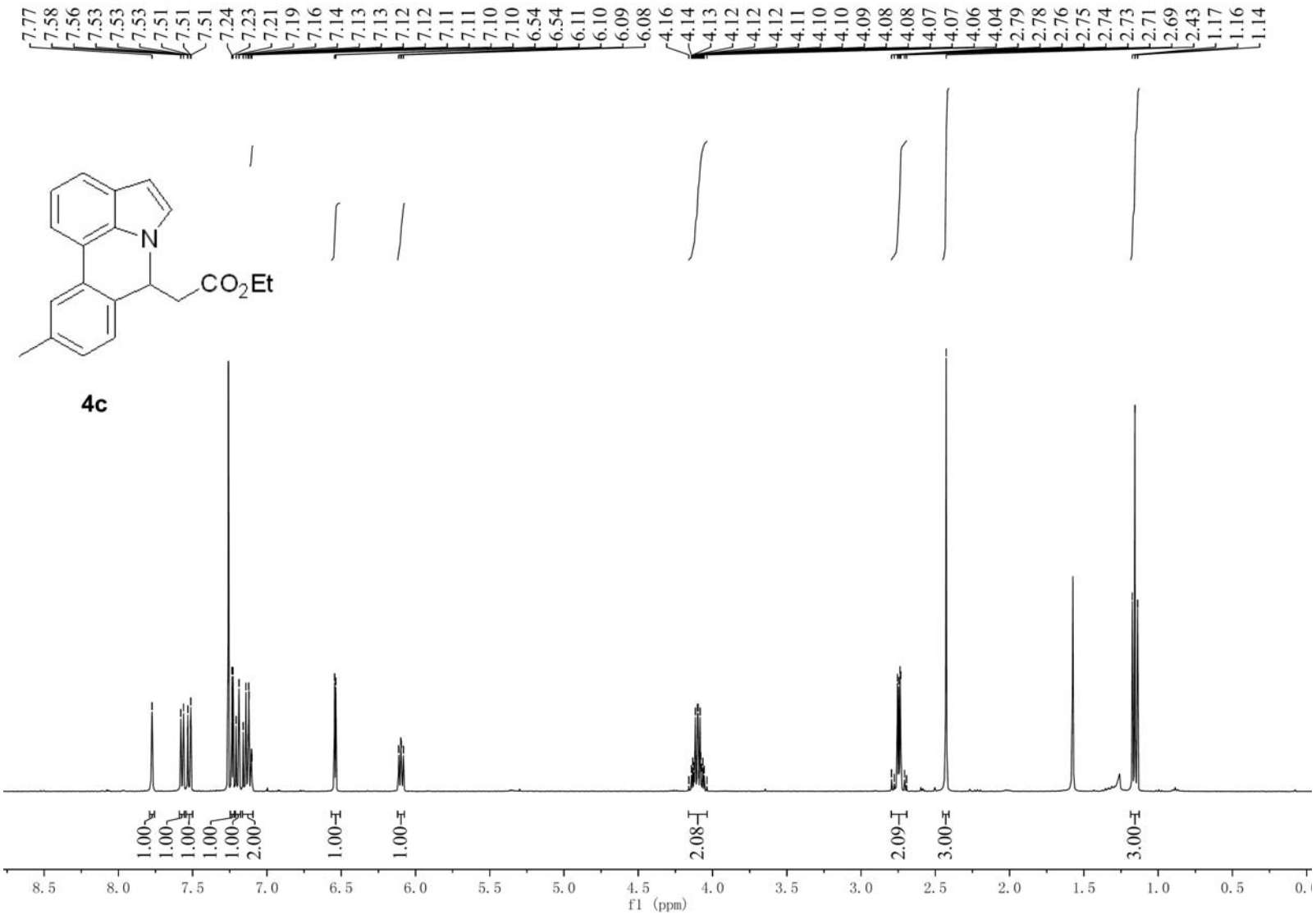
30
20
10
f1 (ppm)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4.10
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2.43
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CO2Et
4c
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1.00
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1.00
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3.0
2.5
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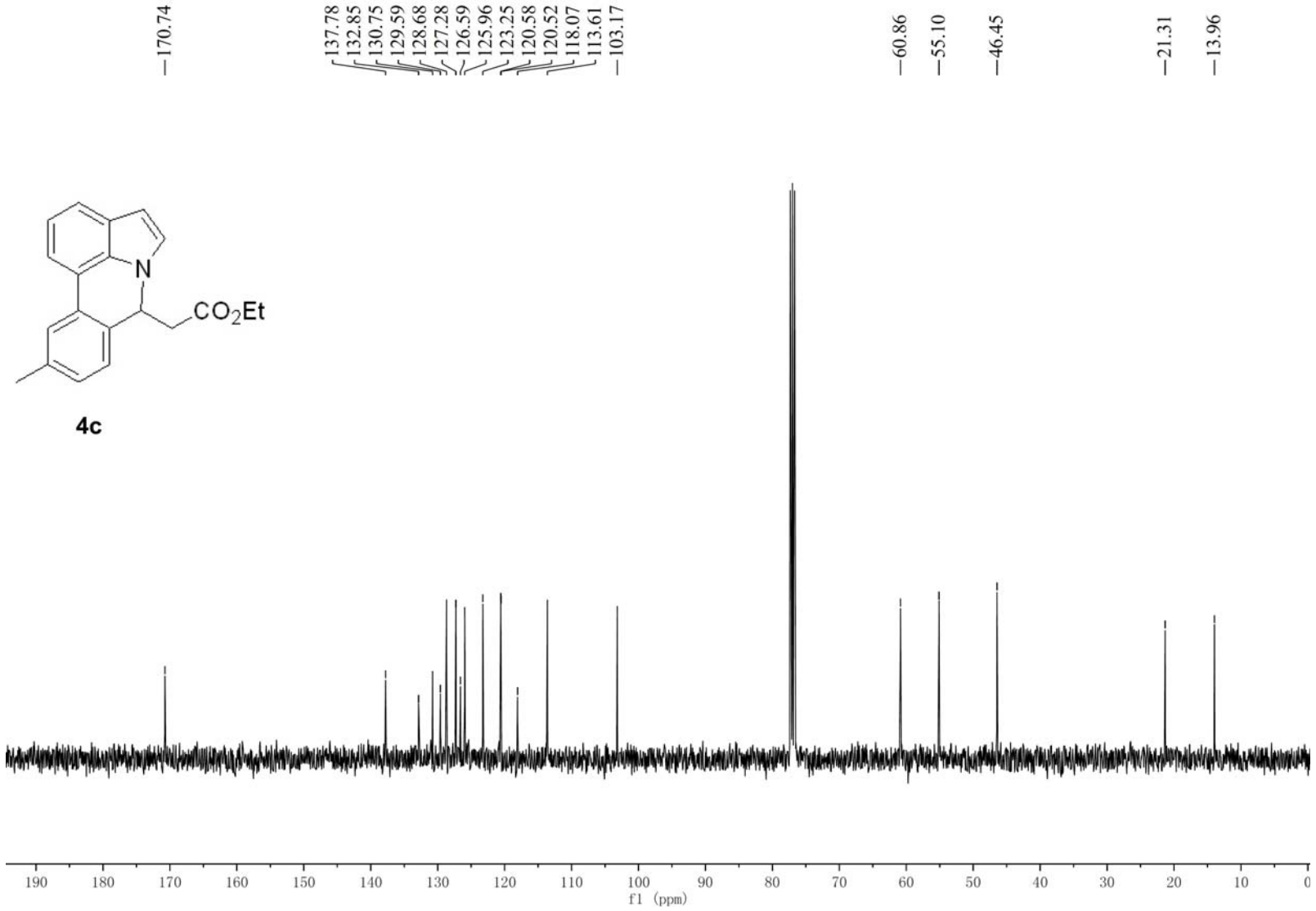
f1 (ppm)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55.10
46.45
21.31
13.96
N
CO2Et
4c
190
180
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80
70
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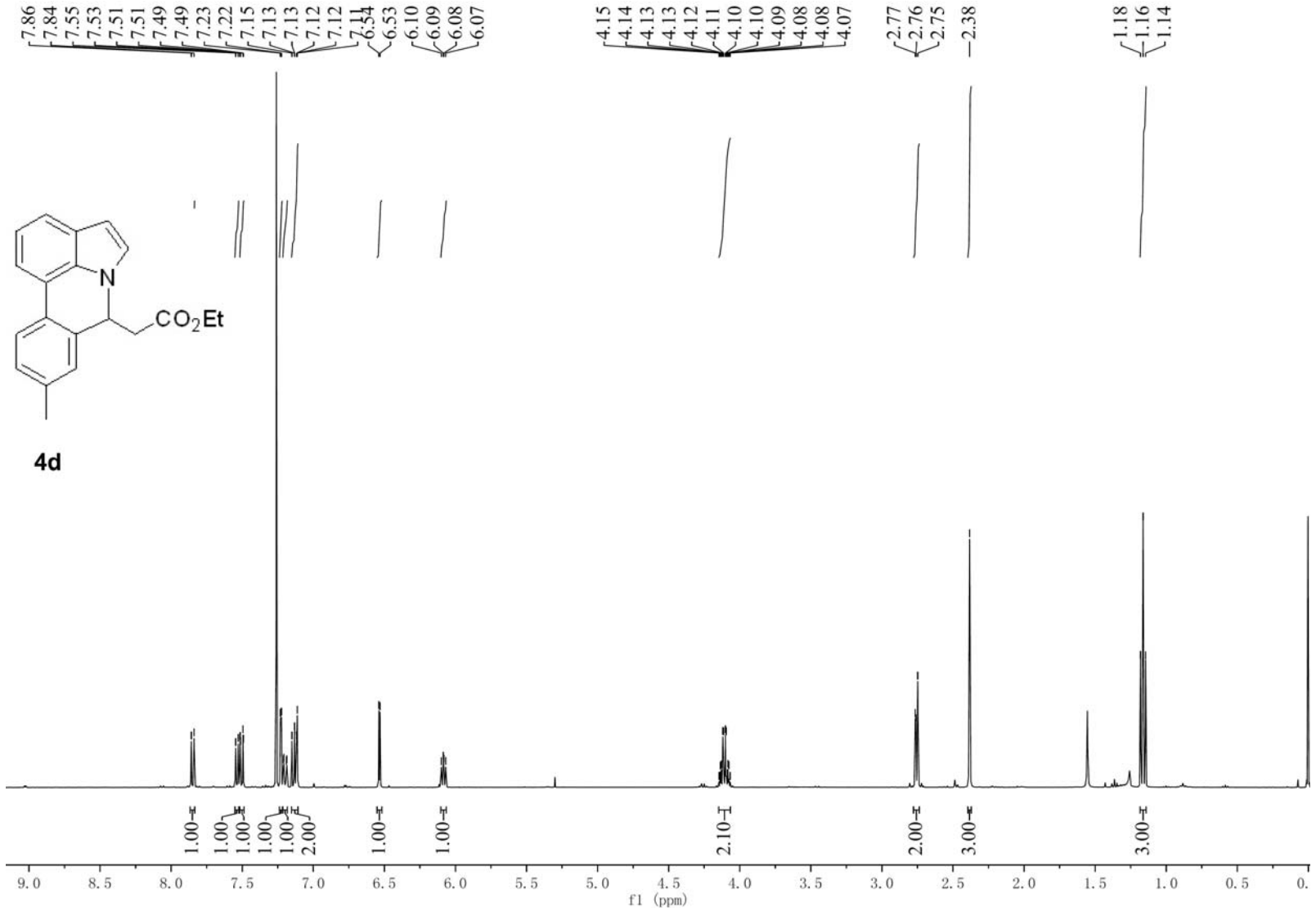
50
40
30
20
10
0
f1 (ppm)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4.10
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4.08
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1.16
1.14
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1.00
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2.0
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1.0
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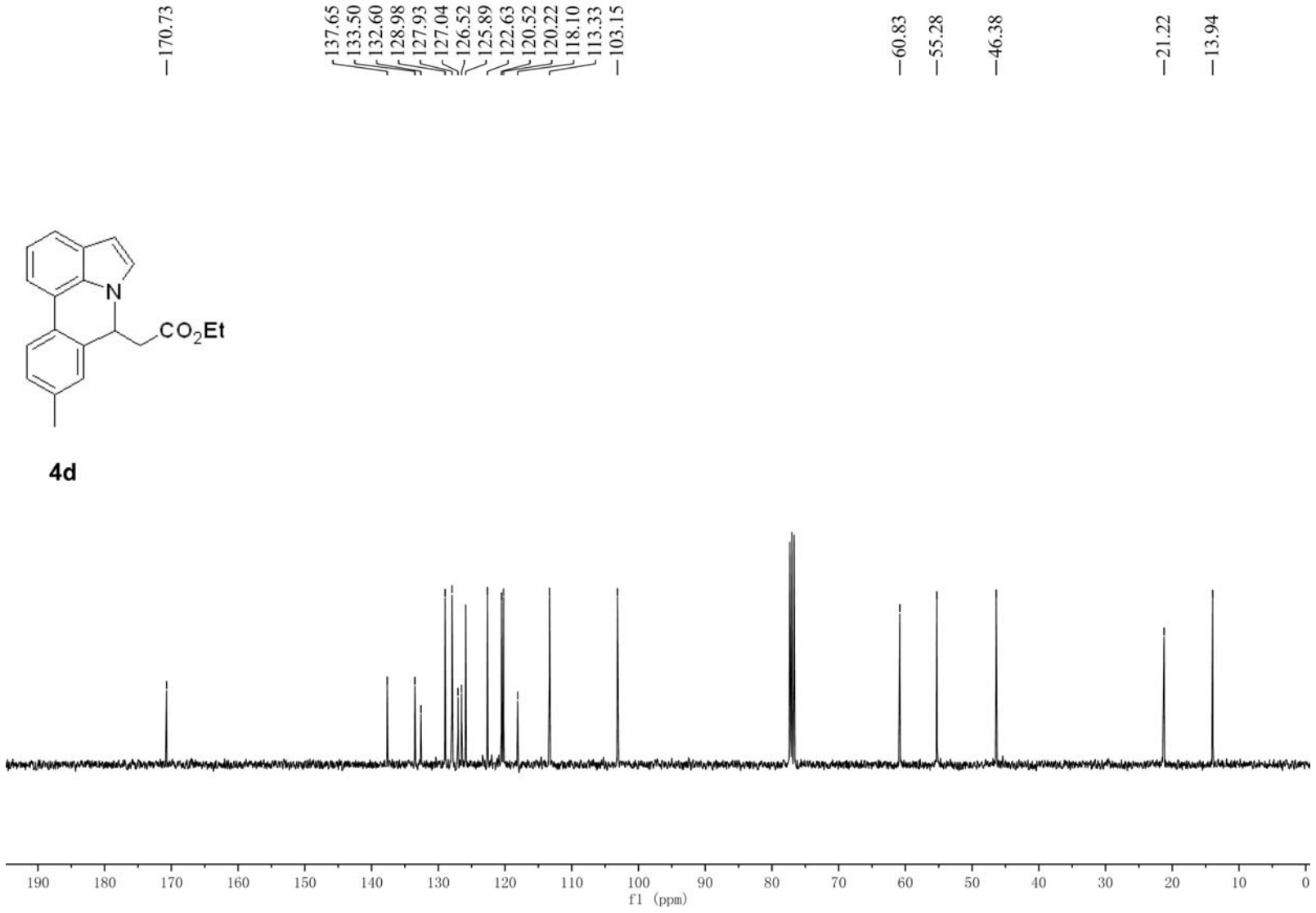
0.
f1 (ppm)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113.33
103.15
60.83
55.28
46.38
21.22
13.94
N
CO2Et
4d
190
180
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110
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90
80
70
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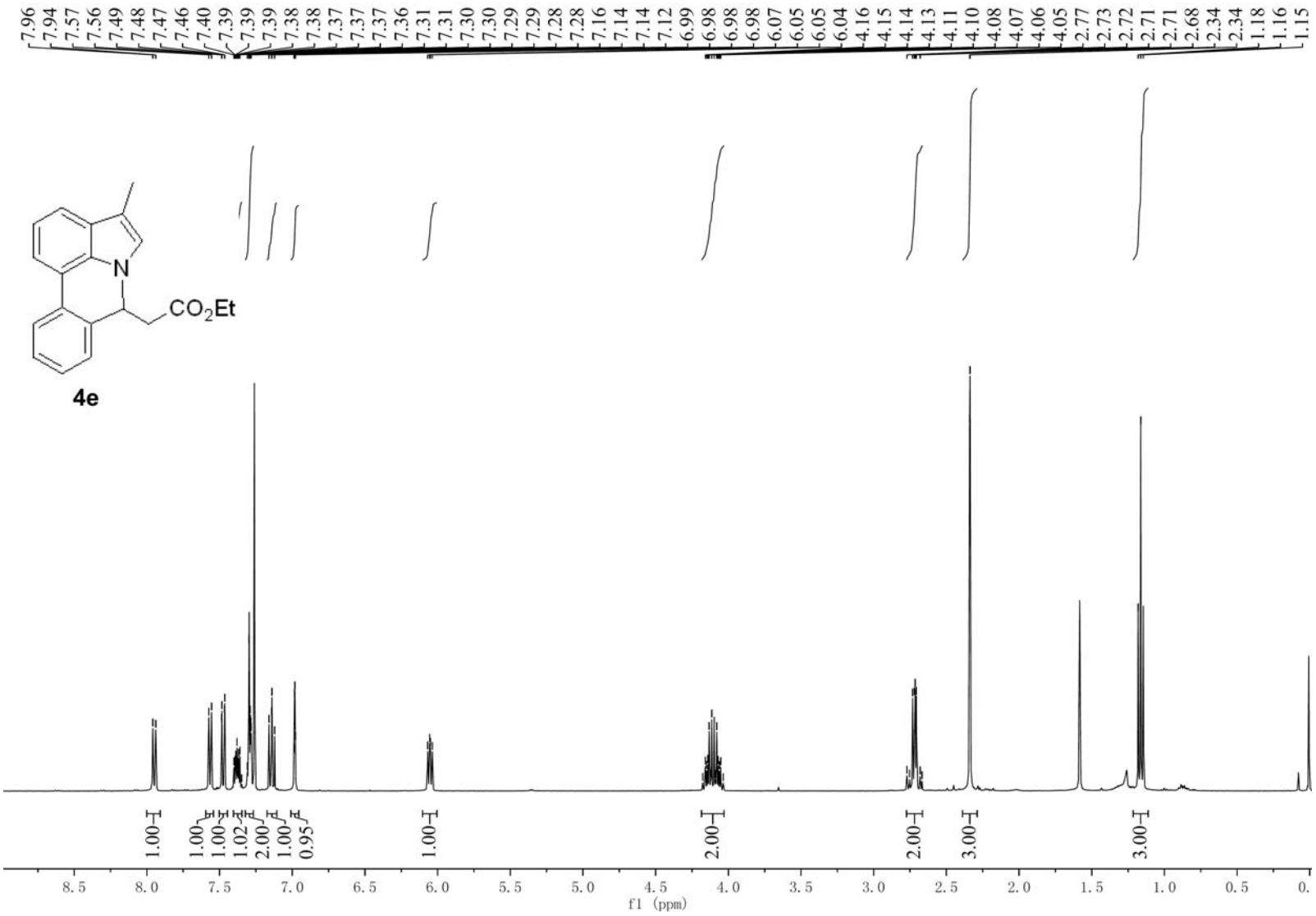
50
40
30
20
10
0
f1 (ppm)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2.34
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1.16
1.15
N
CO2Et
4e
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1.00
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2.00
1.00
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1.00
2.00
2.00
3.00
3.00
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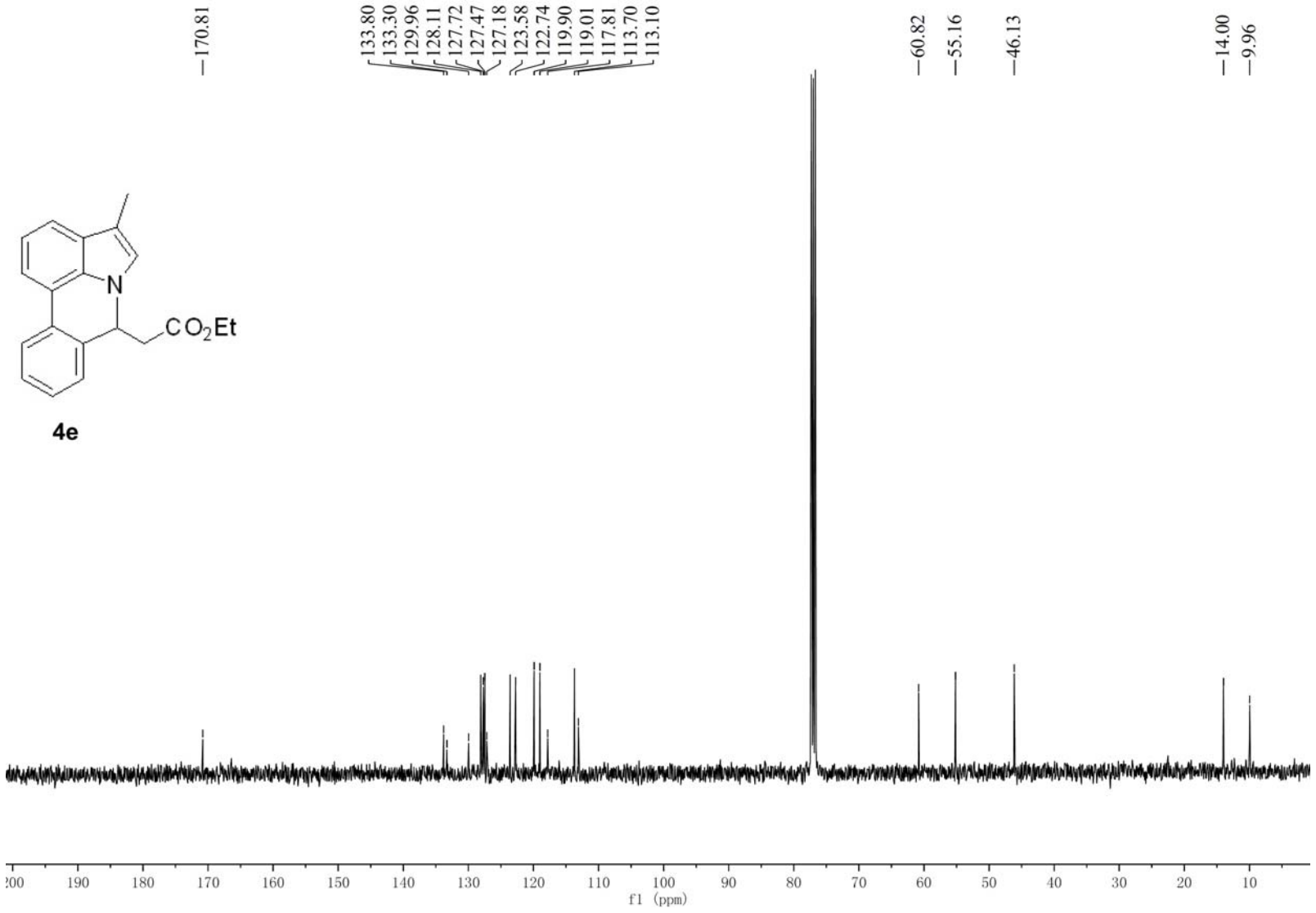
f1 (ppm)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119.01
117.81
113.70
113.10
60.82
55.16
46.13
14.00
9.96
N
CO2Et
4e
200
190
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140
130
120
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100
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80
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60
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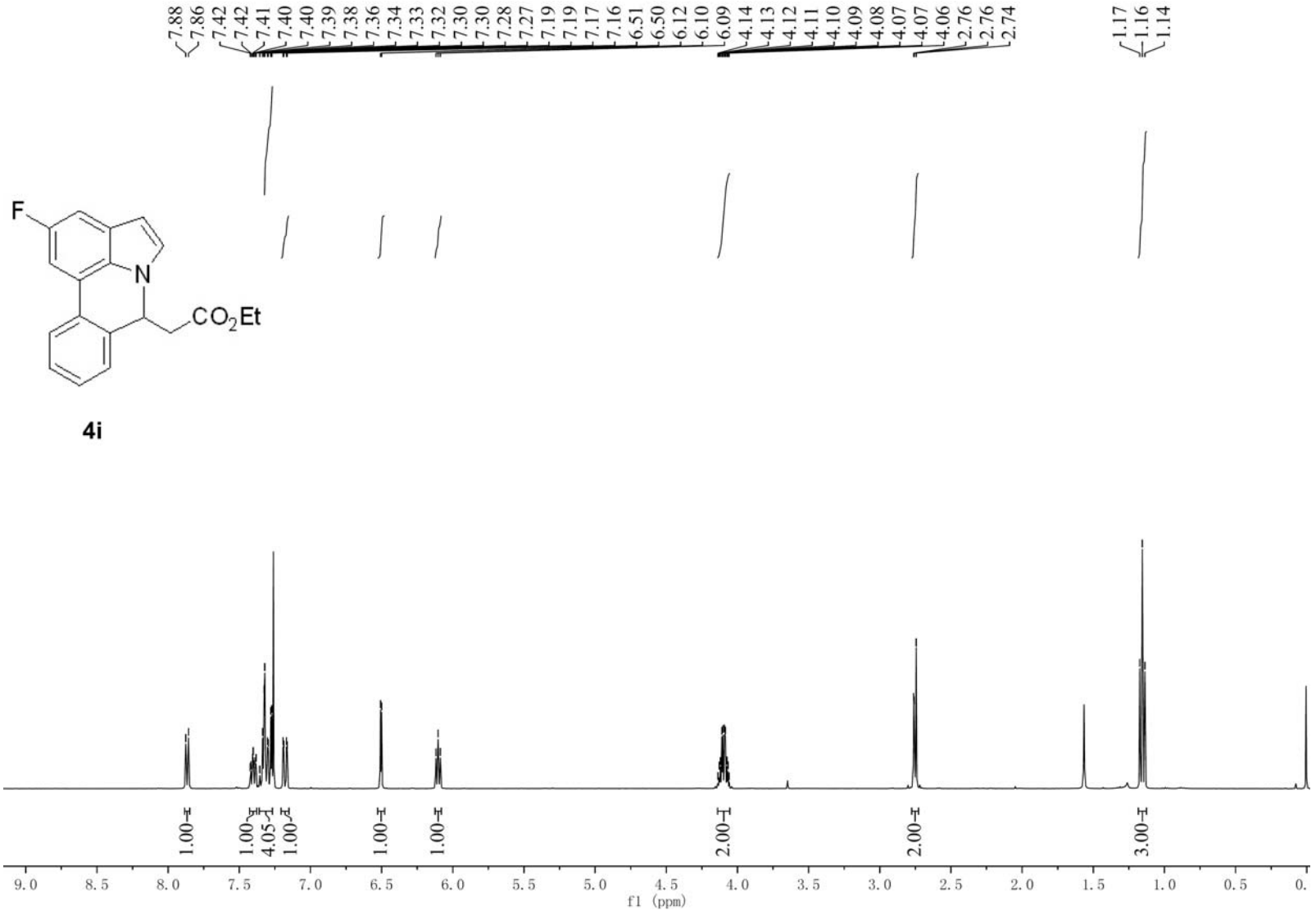
f1 (ppm)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7.30
7.30
7.28
7.27
7.19
7.19
7.17
7.16
6.51
6.50
6.12
6.10
6.09
4.14
4.13
4.12
4.11
4.10
4.09
4.08
4.07
4.07
4.06
2.76
2.76
2.74
1.17
1.16
1.14
F
N
CO2Et
1.00
1.00
4.05
1.00
1.00
1.00
2.00
2.00
3.00
9.0
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8.0
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7.0
6.5
6.0
5.5
5.0
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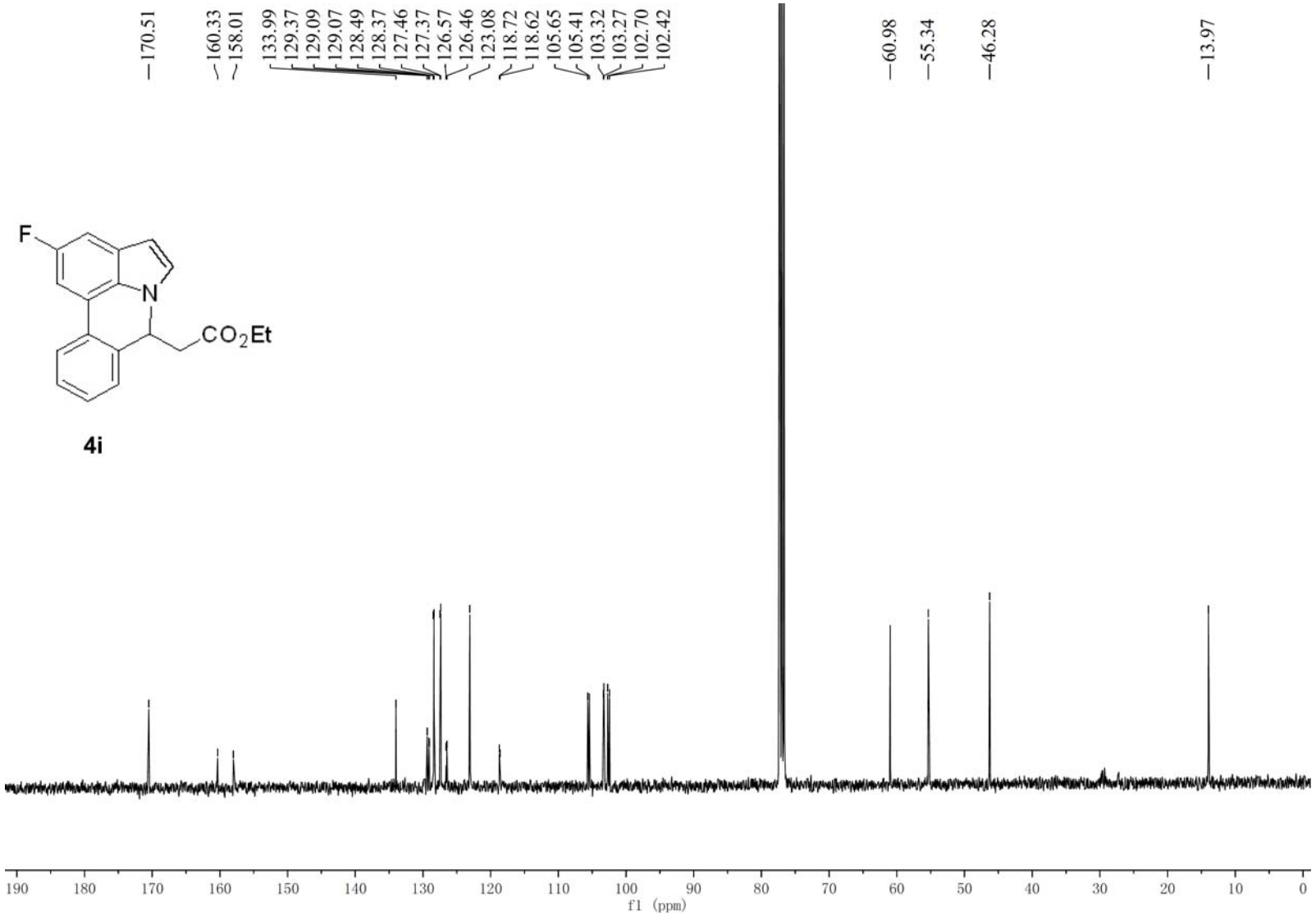
2.0
1.5
1.0
0.5
0.
f1 (ppm)

4i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128.49
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127.37
126.57
126.46
123.08
118.72
118.62
105.65
105.41
103.32
103.27
102.70
102.42
60.98
55.34
46.28
13.97
F
N
CO2Et
4i
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
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30
20
10
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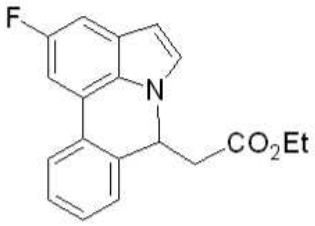
f1 (ppm)


F
N
CO2Et

4i

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-123.46
-123.49
-30
-40
-50
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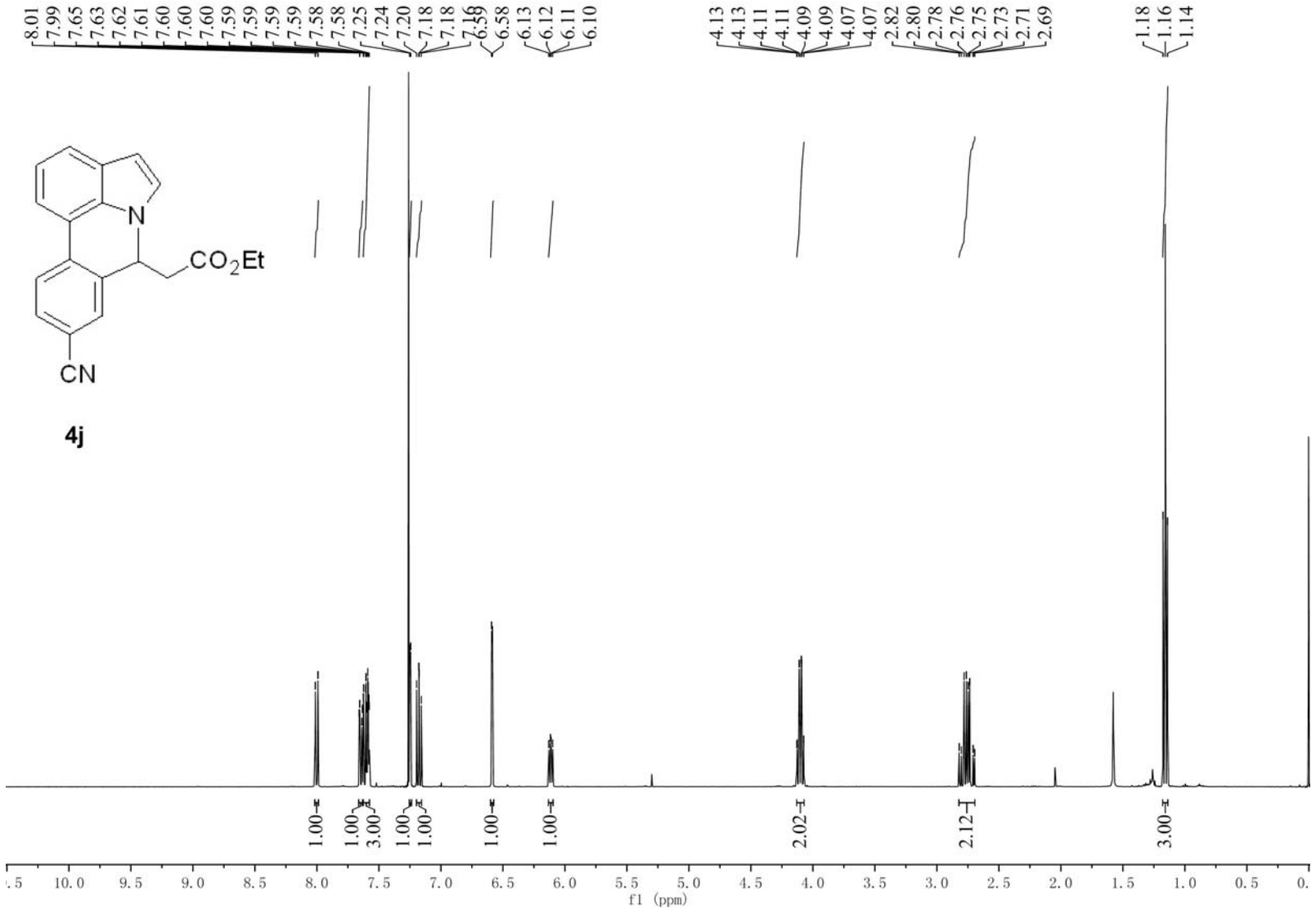
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
f1 (ppm)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7.60
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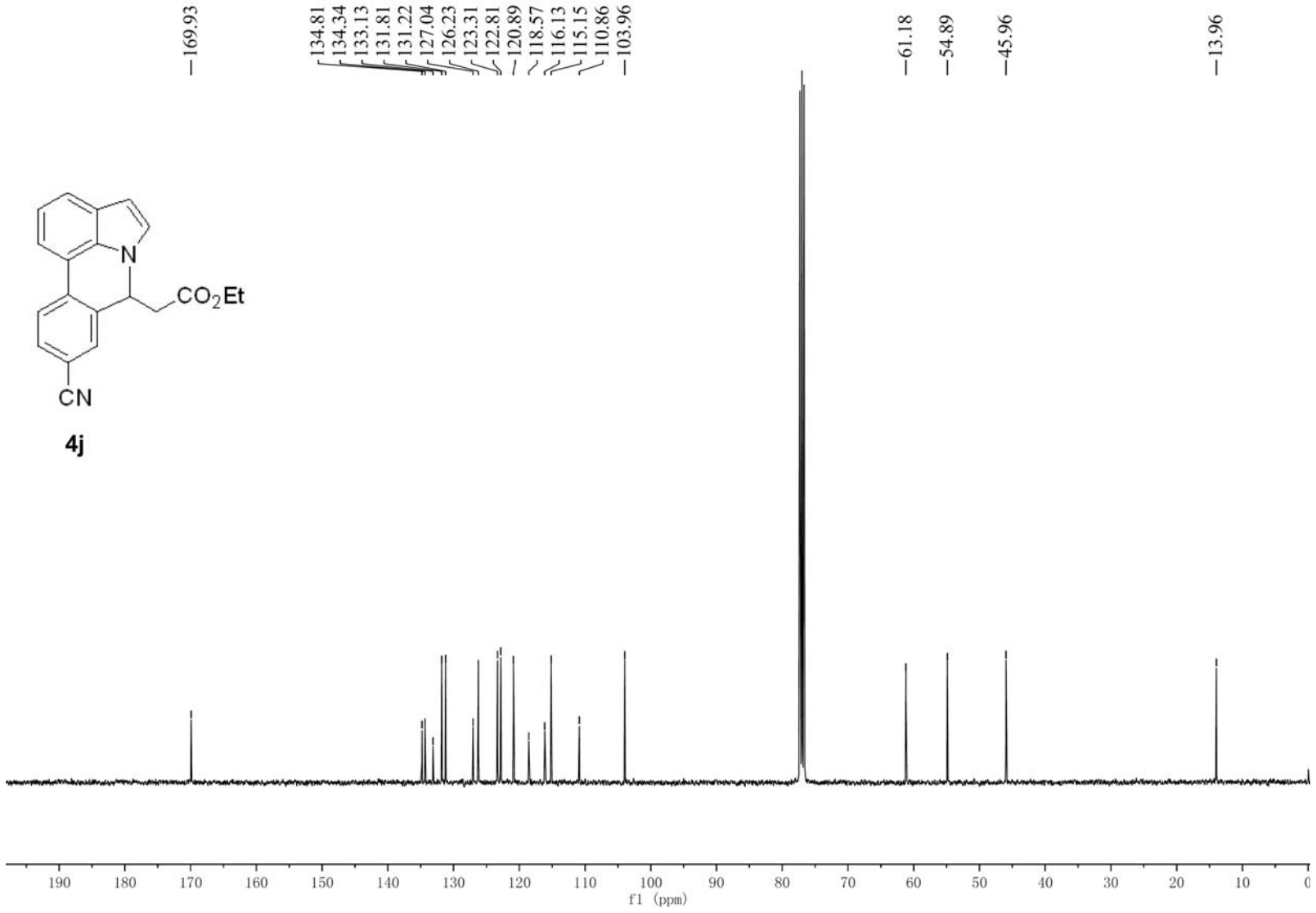
7.59
7.59
7.59
7.58
7.58
7.25
7.24
7.20
7.18
7.18
6.58
6.13
6.12
6.11
6.10
4.13
4.13
4.11
4.11
4.09
4.09
4.07
4.07
2.82
2.80
2.78
2.76
2.75
2.73
2.71
2.69
1.18
1.16
1.14
N
CO2Et
CN
4j
1.00
1.00
3.00
1.00
1.00
1.00
1.00
2.02
2.12
3.00
f1 (ppm)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126.23
123.31
122.81
120.89
118.57
116.13
115.15
110.86
103.96
61.18
54.89
45.96
13.96
N
CO2Et
CN
4j
190
180
170
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130
120
110
100
90
80
70
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50
40
30
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10
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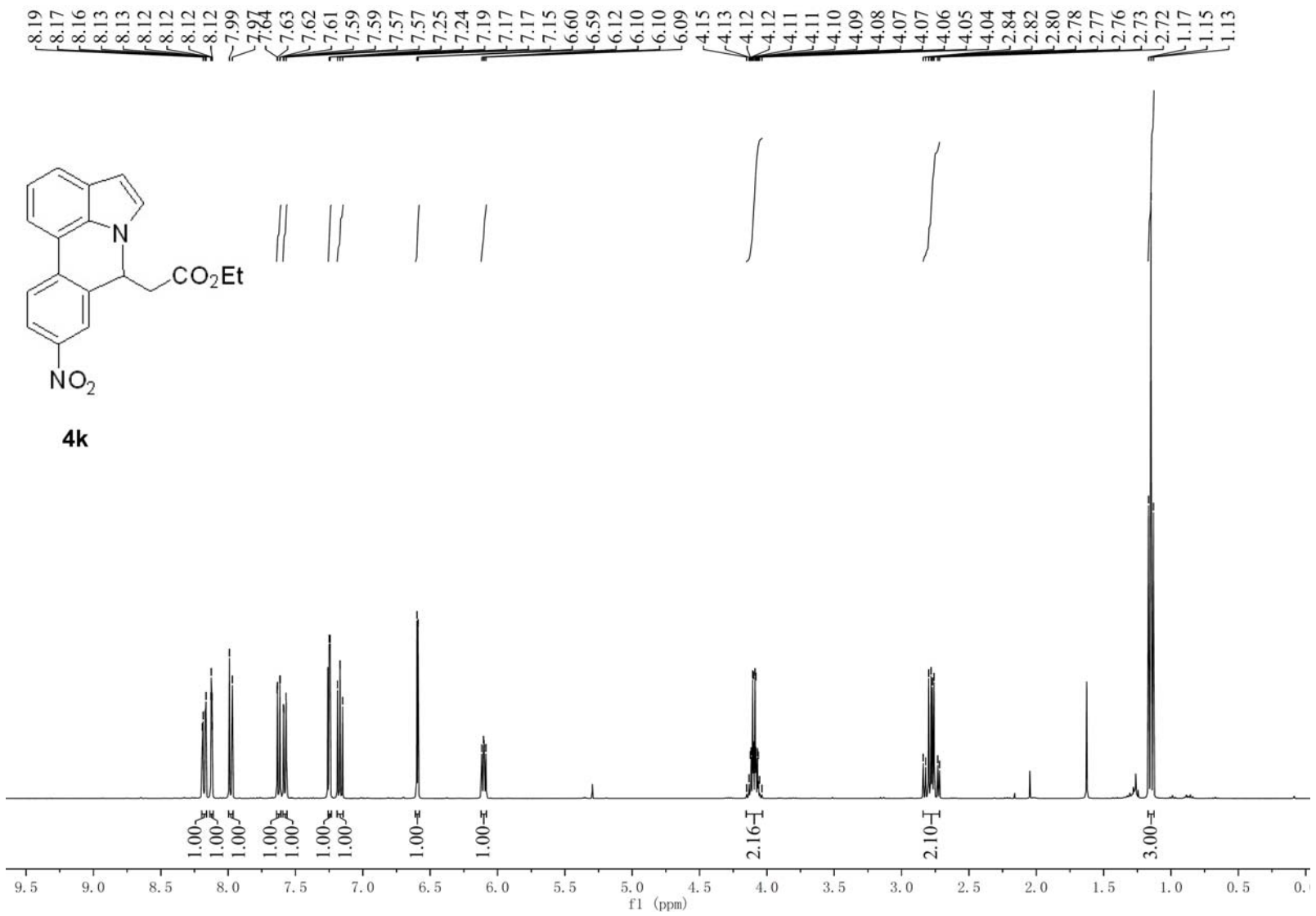
f1 (ppm)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8.13
8.13
8.12
8.12
8.12
8.12
7.99
7.97
7.84
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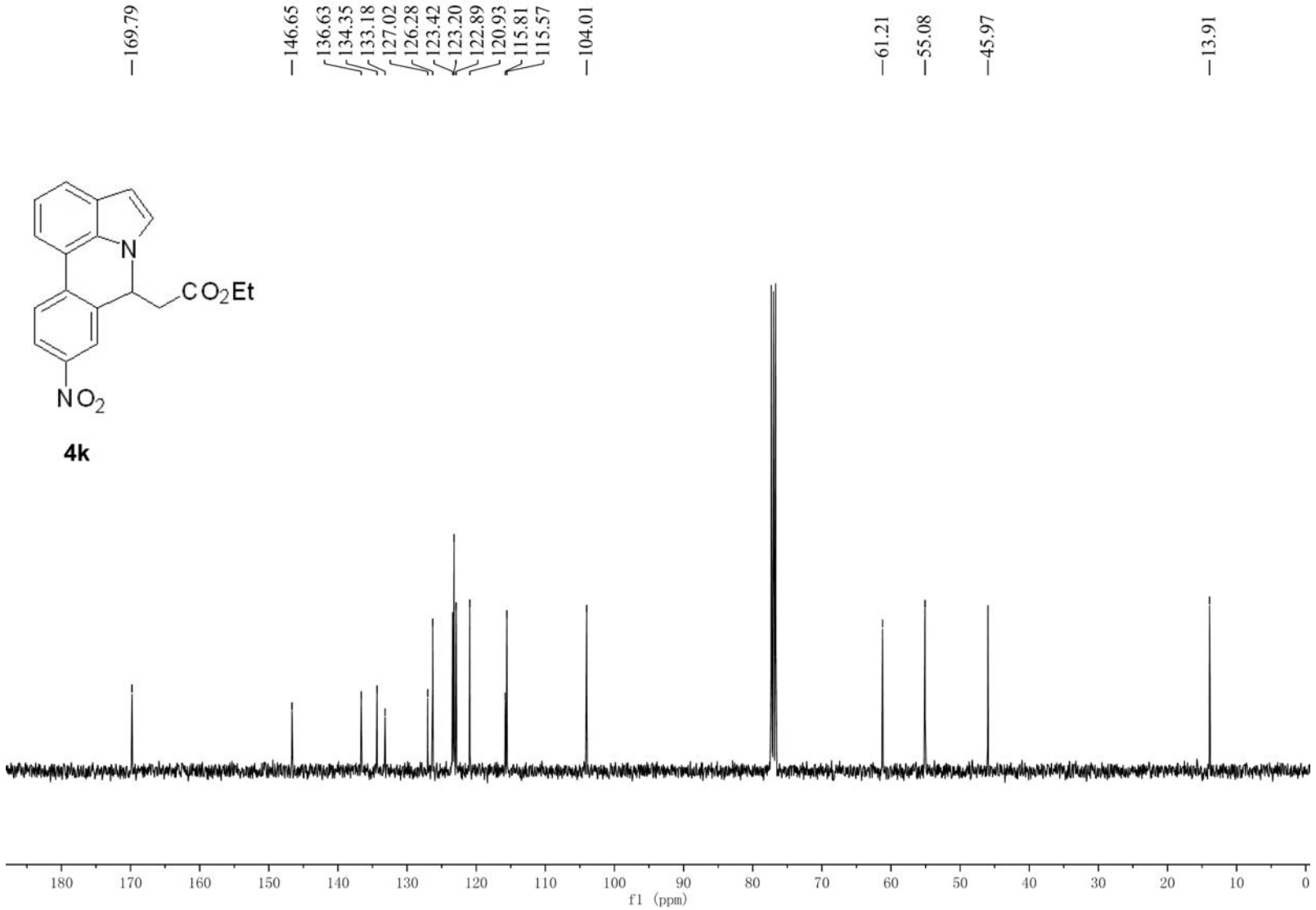
7.62
7.61
7.59
7.59
7.57
7.57
7.25
7.24
7.19
7.17
7.17
7.15
6.60
6.59
6.12
6.10
6.10
6.09
4.15
4.13
4.12
4.12
4.11
4.11
4.10
4.09
4.08
4.07
4.07
4.06
4.05
4.04
2.84
2.82
2.80
2.78
2.77
2.76
2.73
2.72
1.17
1.15
1.13
N
CO2Et
NO2
4k
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
2.16
2.10
3.00
9.5
9.0
8.5
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)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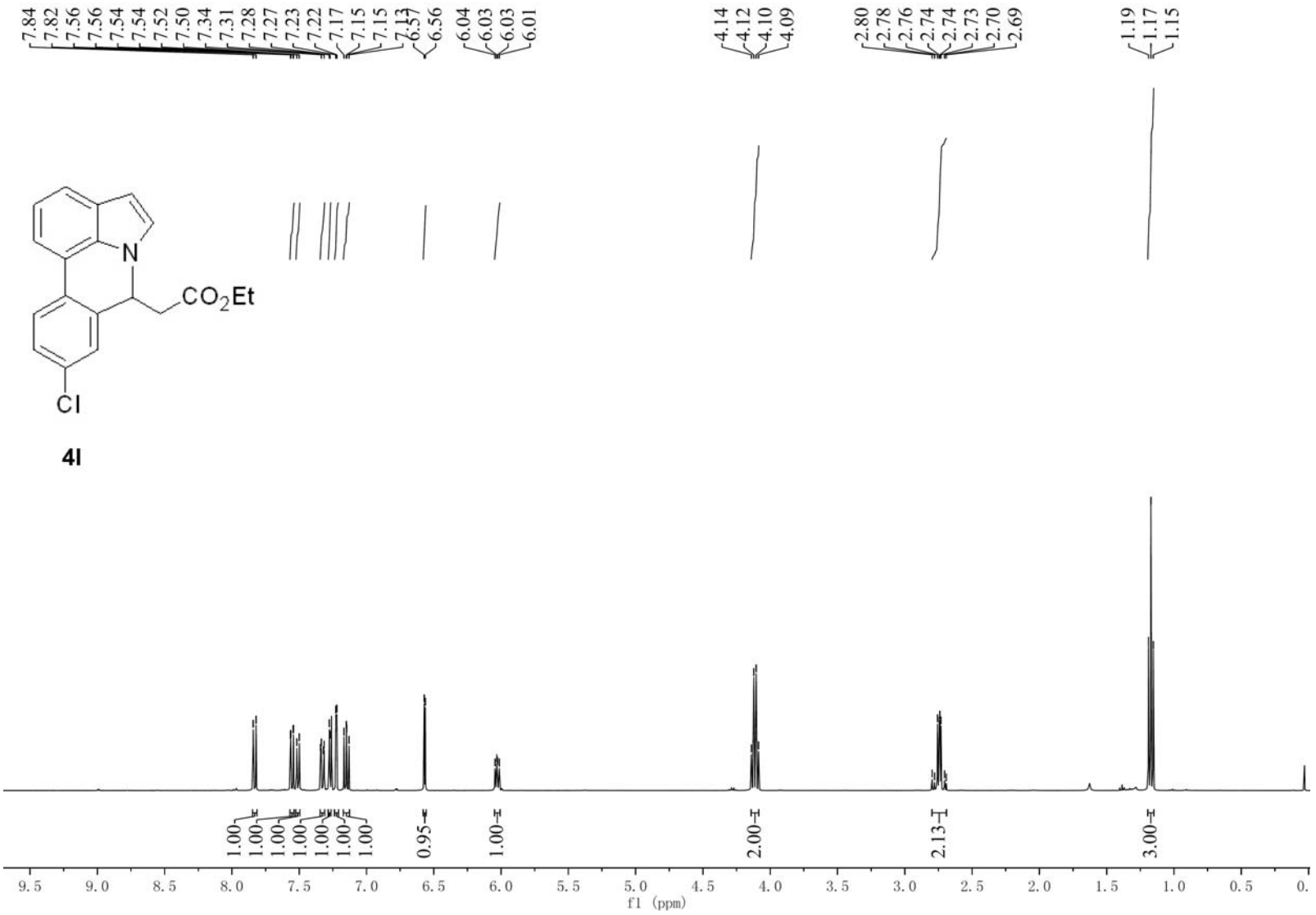
122.89
120.93
115.81
115.57
104.01
61.21
55.08
45.97
13.91
N
CO2Et
NO2
4k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.82
7.56
7.56
7.54
7.54
7.52
7.50
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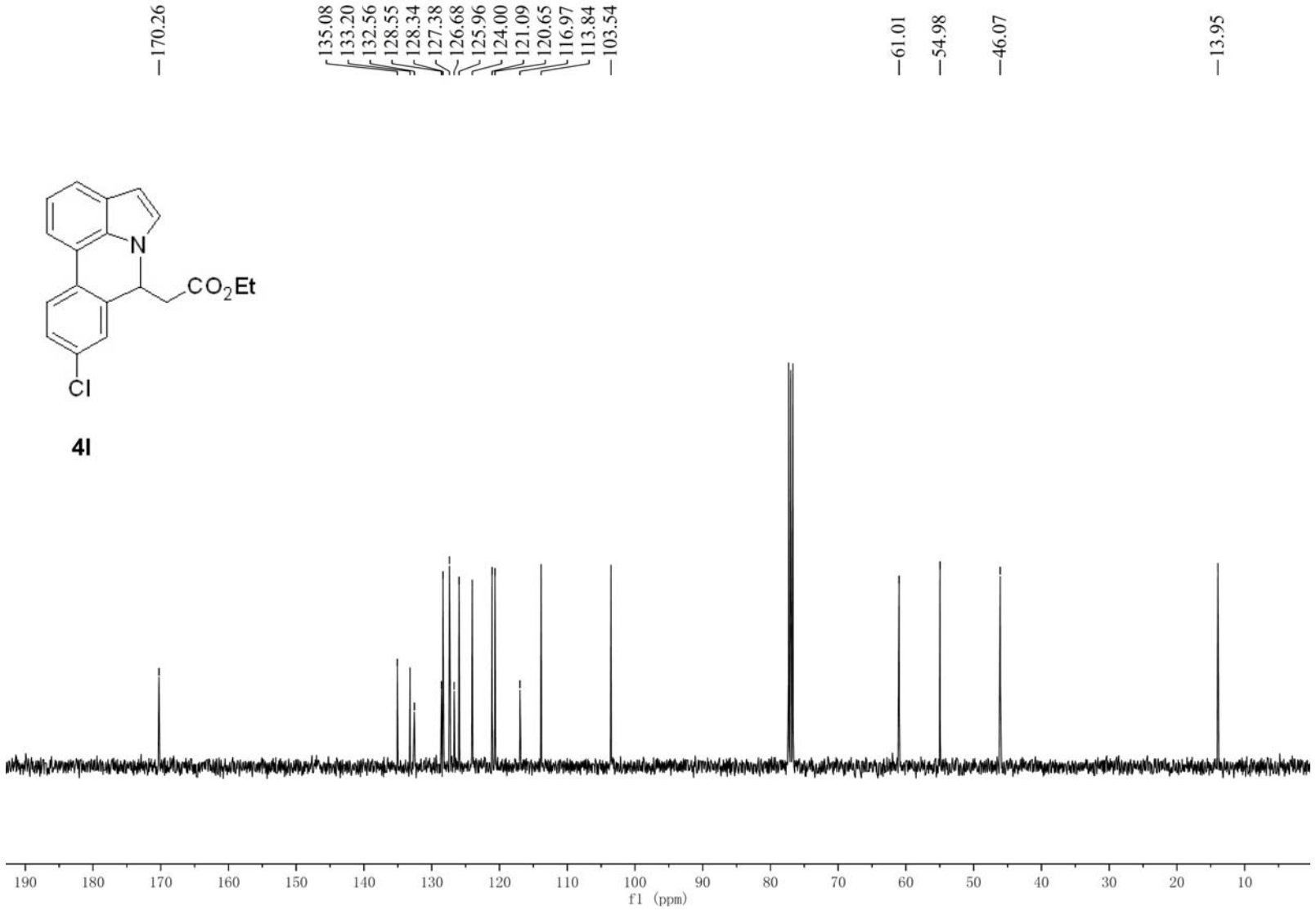
7.31
7.28
7.27
7.23
7.22
7.17
7.15
7.15
7.13
6.57
6.56
6.04
6.03
6.03
6.01
4.14
4.12
4.10
4.09
2.80
2.78
2.76
2.74
2.74
2.73
2.70
2.69
1.19
1.17
1.15
N
CO2Et
Cl
1.00
1.00
1.00
1.00
1.00
1.00
1.00
0.95
1.00
2.00
2.13
3.00
f1 (ppm)

4l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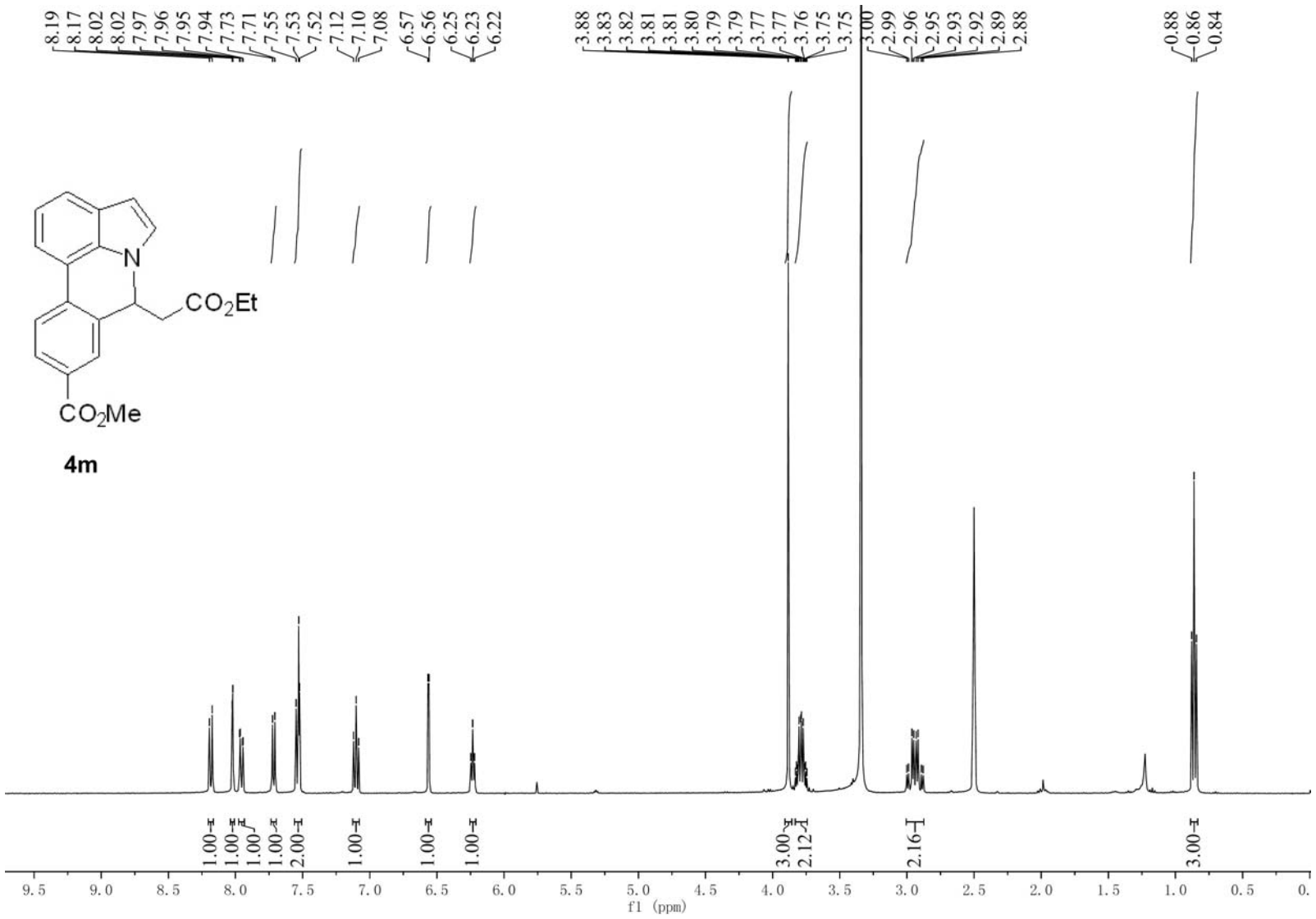
135.08
133.20
132.56
128.55
128.34
127.38
126.68
125.96
124.00
121.09
120.65
116.97
113.84
103.54
61.01
54.98
46.07
13.95
N
CO2Et
Cl
4l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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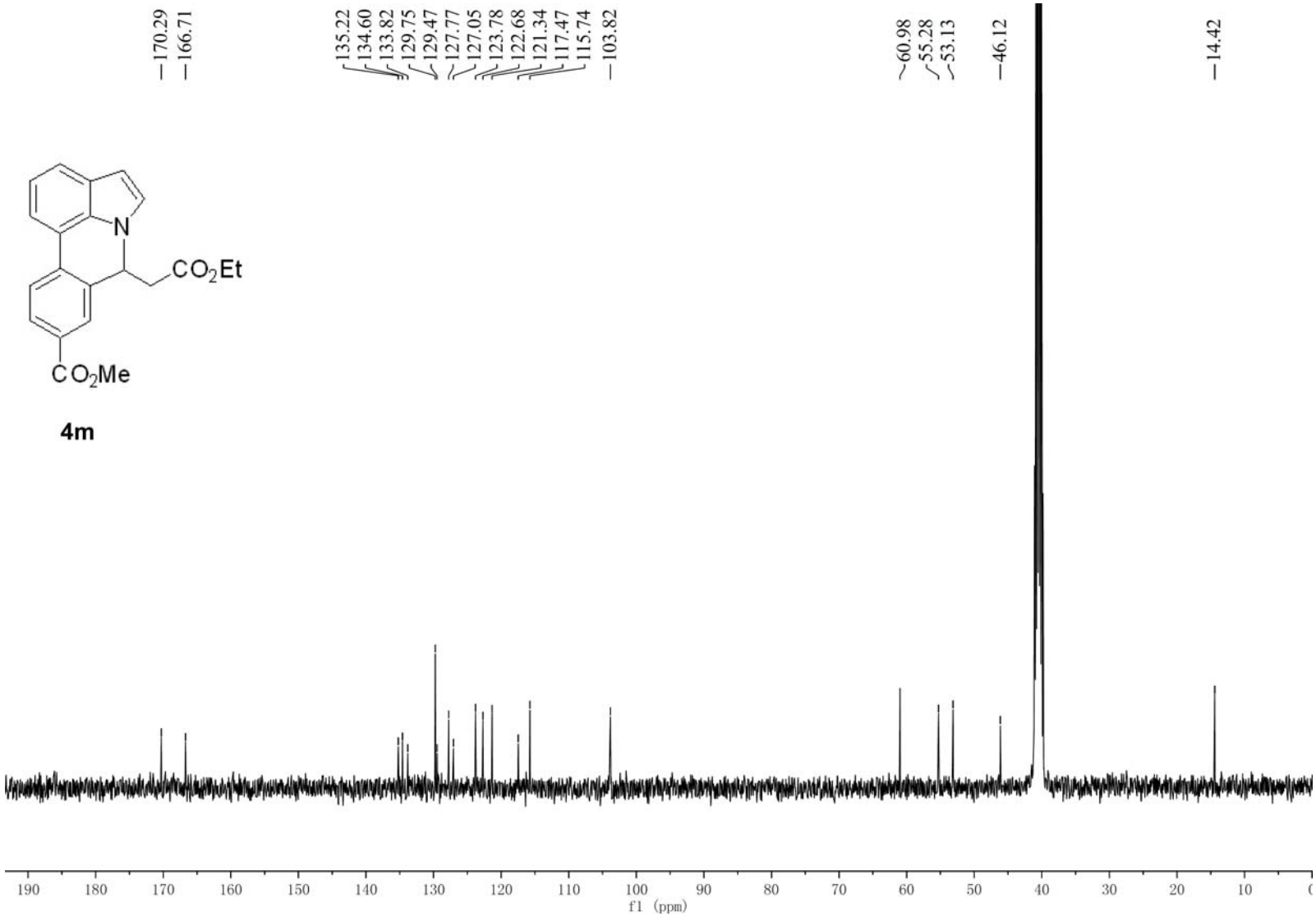
8.17
8.02
8.02
7.97
7.96
7.95
7.94
7.73
7.71
7.55
7.53
7.52
7.12
7.10
7.08
6.57
6.56
6.25
6.23
6.22
3.88
3.83
3.82
3.81
3.81
3.80
3.79
3.79
3.77
3.77
3.76
3.75
3.75
3.00
2.99
2.96
2.95
2.93
2.92
2.89
2.88
0.88
0.86
0.84
N
CO2Et
CO2Me
4m
1.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
3.00
2.12
2.16
3.00
9.5
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.
f1 (ppm)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135.22
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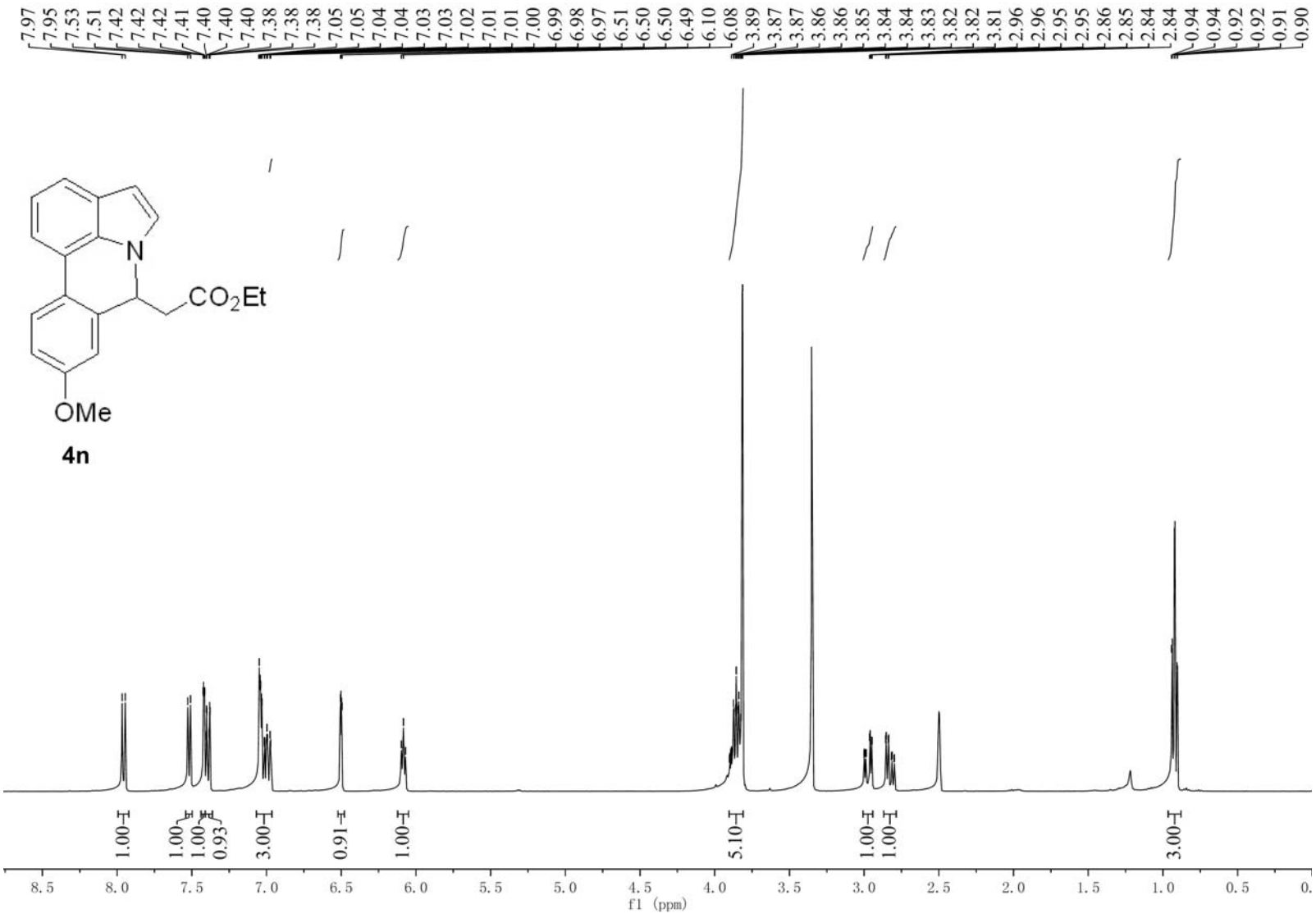
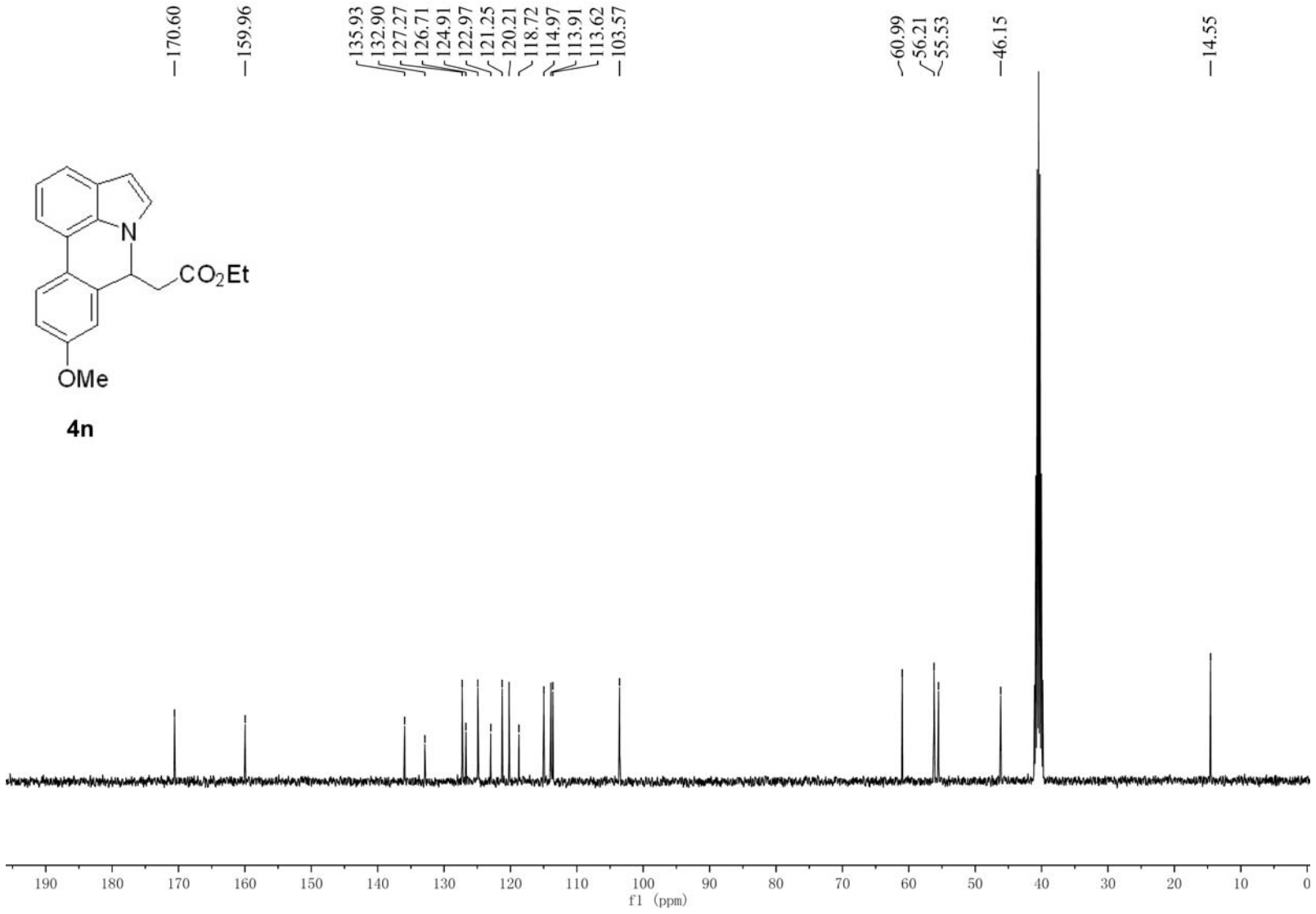
133.82
129.75
129.47
127.77
127.05
123.78
122.68
121.34
117.47
115.74
103.82
60.98
55.28
53.13
46.12
14.42
N
CO2Et
CO2Me
4m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.97 7.95 7.53 7.51 7.42 7.42 7.42 7.41 7.40 7.40 7.40 7.38 7.38 7.38 7.05 7.05 7.04 7.04 7.03 7.03 7.02 7.01 7.01 7.00 6.99 6.98 6.97 6.51 6.50 6.50 6.49 6.10 6.08 3.89 3.87 3.87 3.86 3.86 3.85 3.84 3.84 3.83 3.82 3.82 3.81 2.96 2.96 2.95 2.95 2.86 2.85 2.84 2.84 0.94 0.94 0.92 0.92 0.91 0.90
N
CO2Et
OMe
4n
1.00 1.00 1.00 0.93 3.00 0.91 1.00 5.10 1.00 1.00 3.00
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.
f1 (ppm)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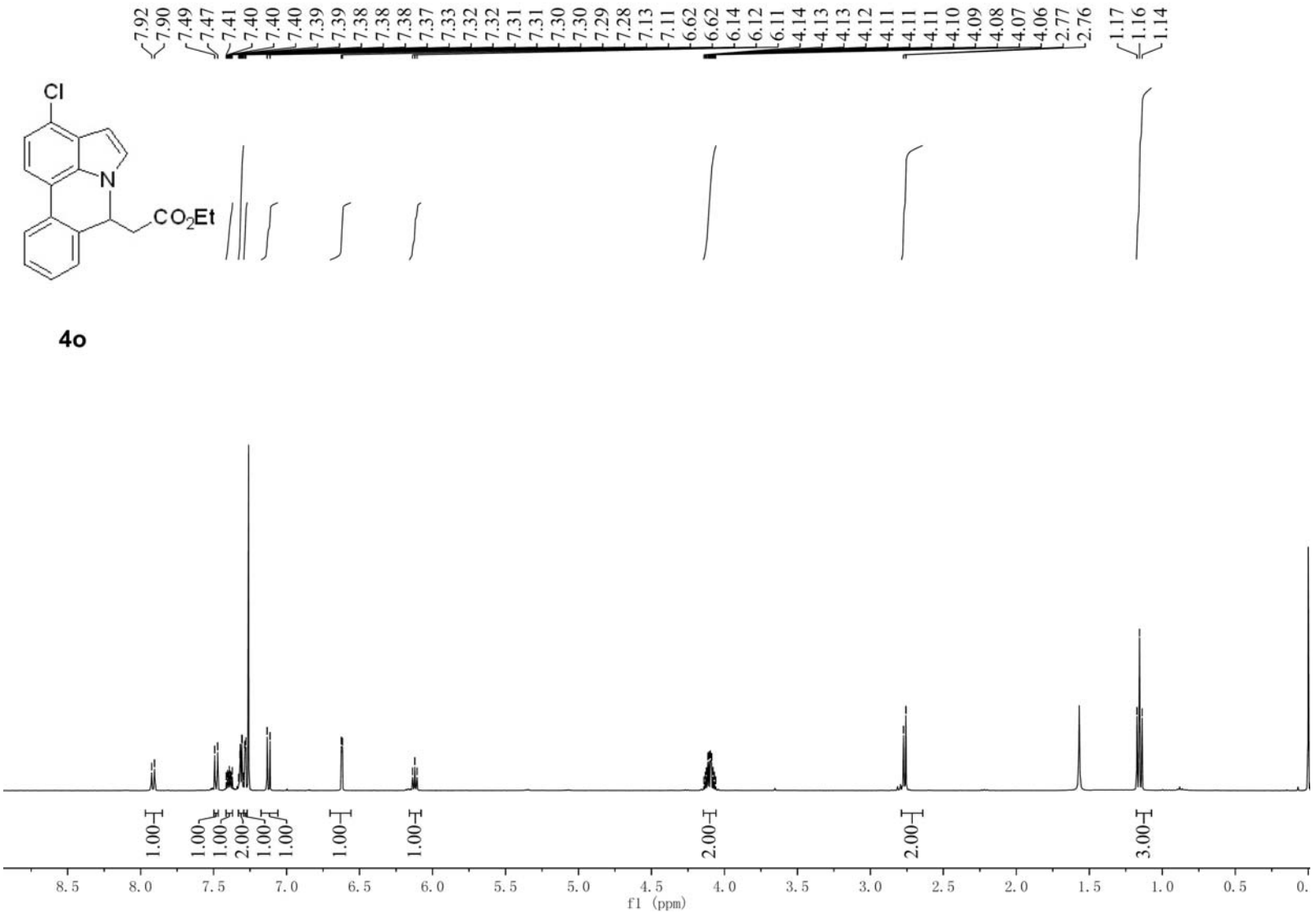
122.97
121.25
120.21
118.72
114.97
113.91
113.62
103.57
60.99
56.21
55.53
46.15
14.55
N
CO2Et
OMe
4n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.38
7.38
7.38
7.37
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.13
7.11
6.62
6.62
6.14
6.12
6.11
4.14
4.13
4.13
4.12
4.11
4.11
4.11
4.10
4.09
4.08
4.07
4.06
2.77
2.76
1.17
1.16
1.14
Cl
N
CO2Et
4o
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
2.00
2.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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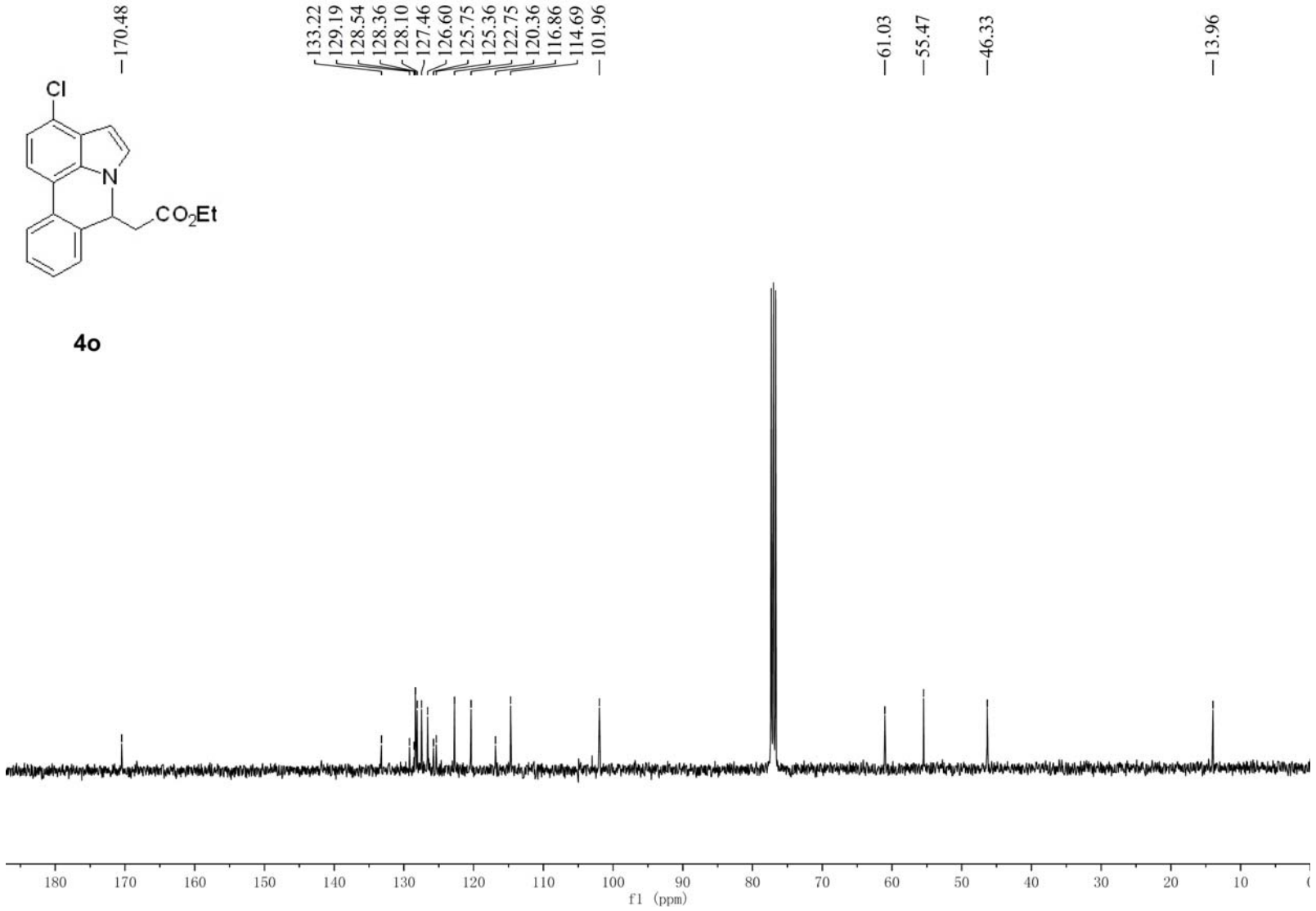
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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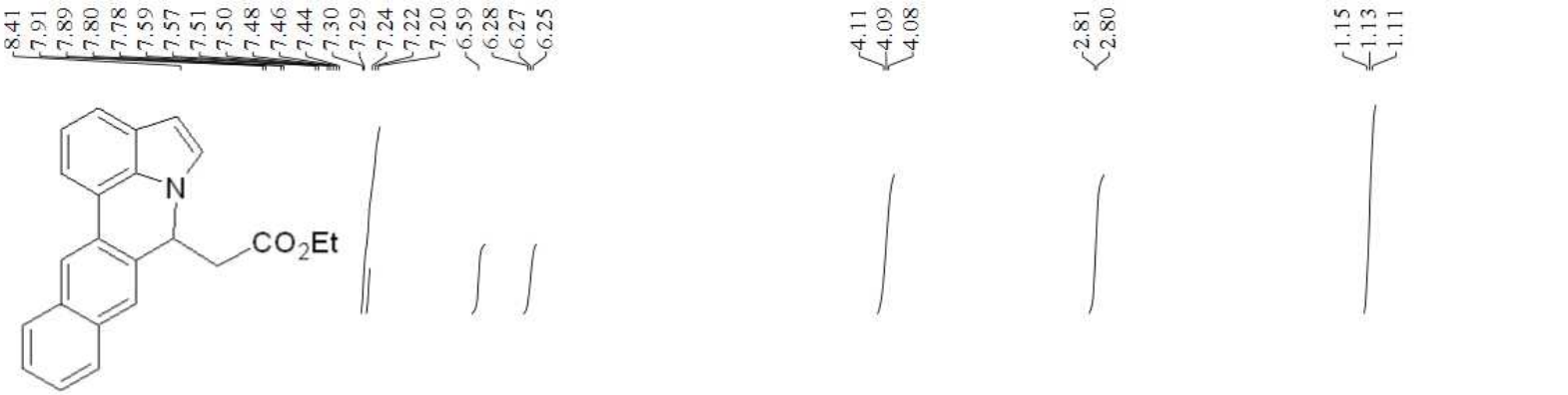
126.60
125.75
125.36
122.75
120.36
116.86
114.69
101.96
61.03
55.47
46.33
13.96
Cl
N
CO2Et
4o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.80
7.78
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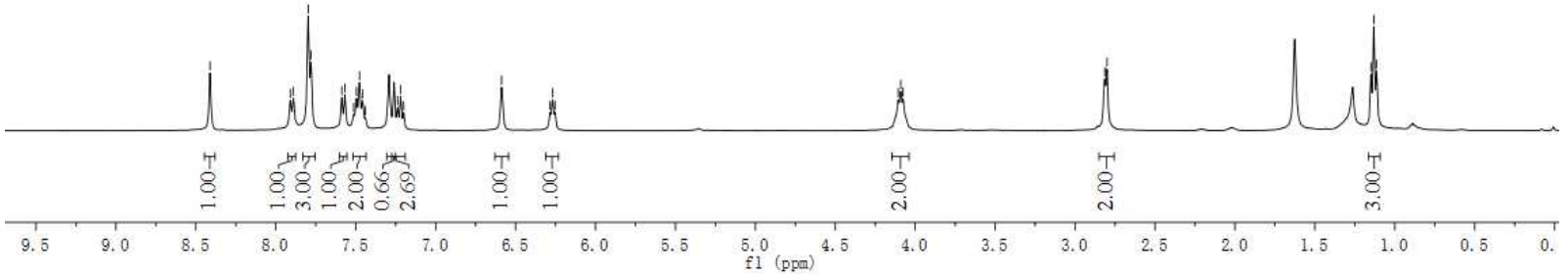
7.51
7.50
7.48
7.46
7.44
7.30
7.29
7.24
7.22
7.20
6.59
6.28
6.27
6.25
4.11
4.09
4.08
2.81
2.80
1.15
1.13
1.11
N
CO2Et

4p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1.00
3.00
1.00
2.00
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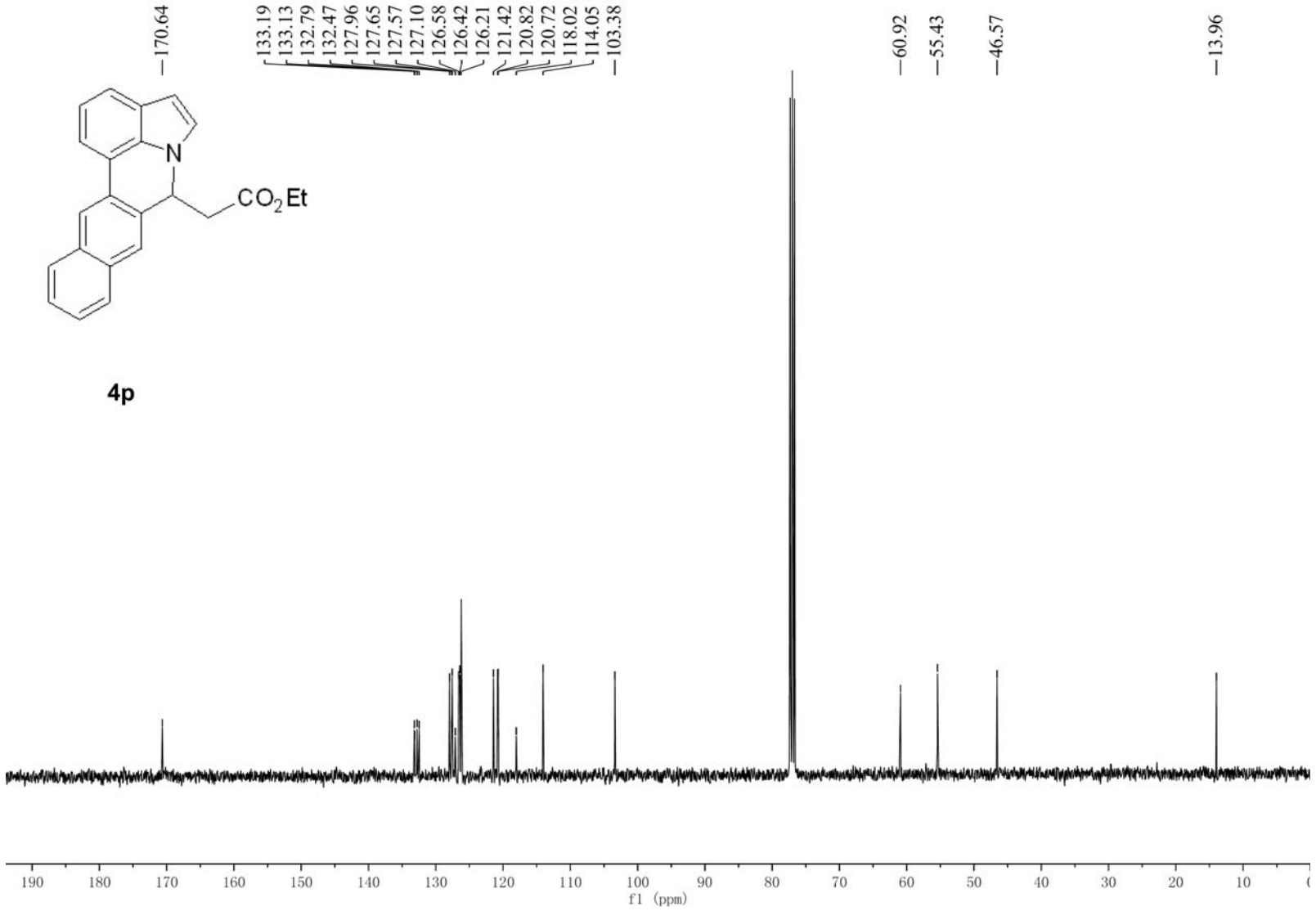
2.69
1.00
1.00
2.00
2.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)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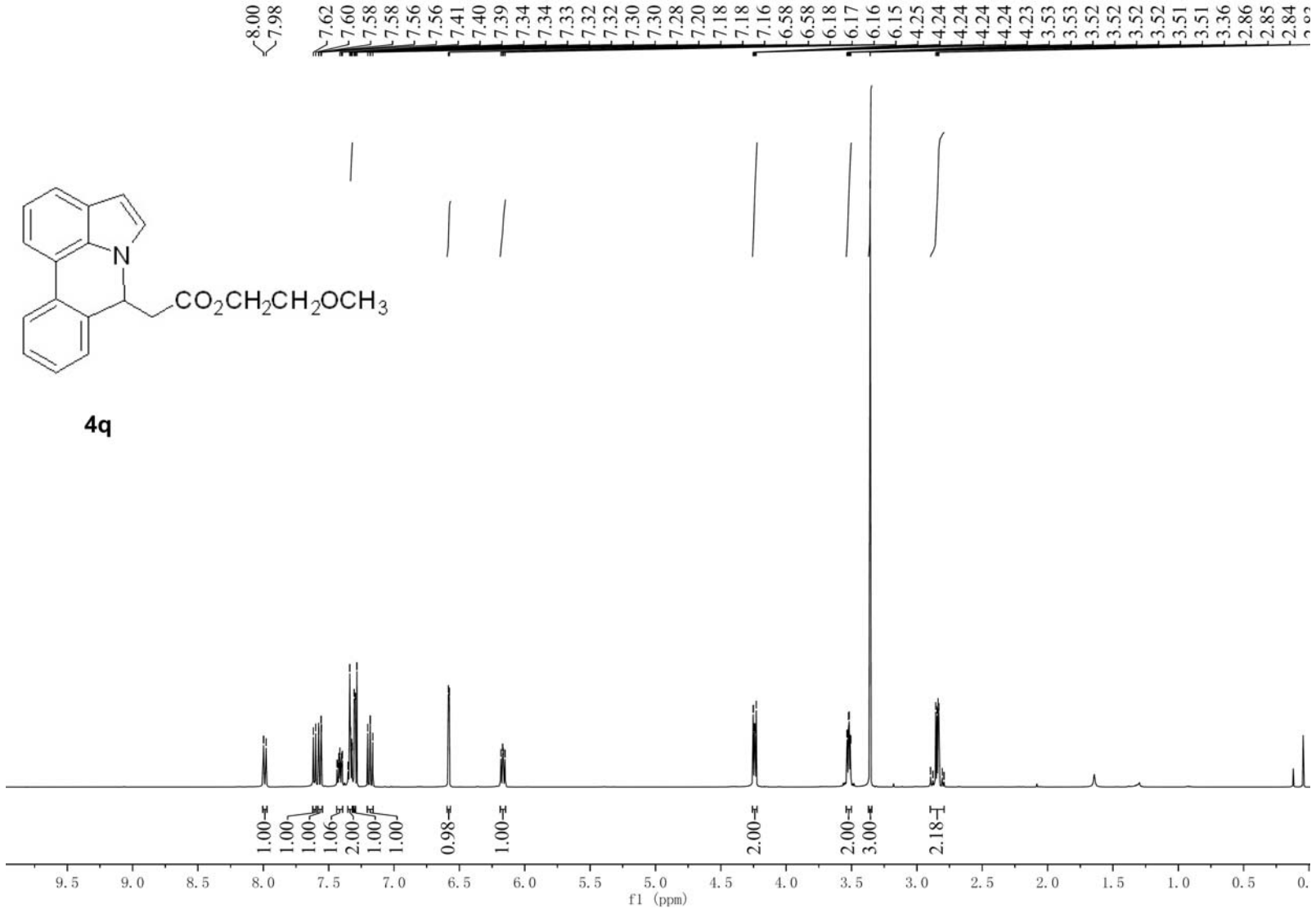
127.57
127.10
126.58
126.42
126.21
121.42
120.82
120.72
118.02
114.05
103.38
60.92
55.43
46.57
13.96
N
CO2Et
4p
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.56
7.41
7.40
7.39
7.34
7.34
7.33
7.32
7.32
7.30
7.30
7.28
7.20
7.18
7.18
7.16
6.58
6.58
6.18
6.17
6.16
6.15
4.25
4.24
4.24
4.24
4.23
3.53
3.53
3.52
3.52
3.52
3.52
3.51
3.51
3.36
2.86
2.85
2.84
N
CO2CH2CH2OCH3
4q
1.00
1.00
1.00
1.06
2.00
1.00
1.00
0.98
1.00
2.00
2.00
3.00
2.18
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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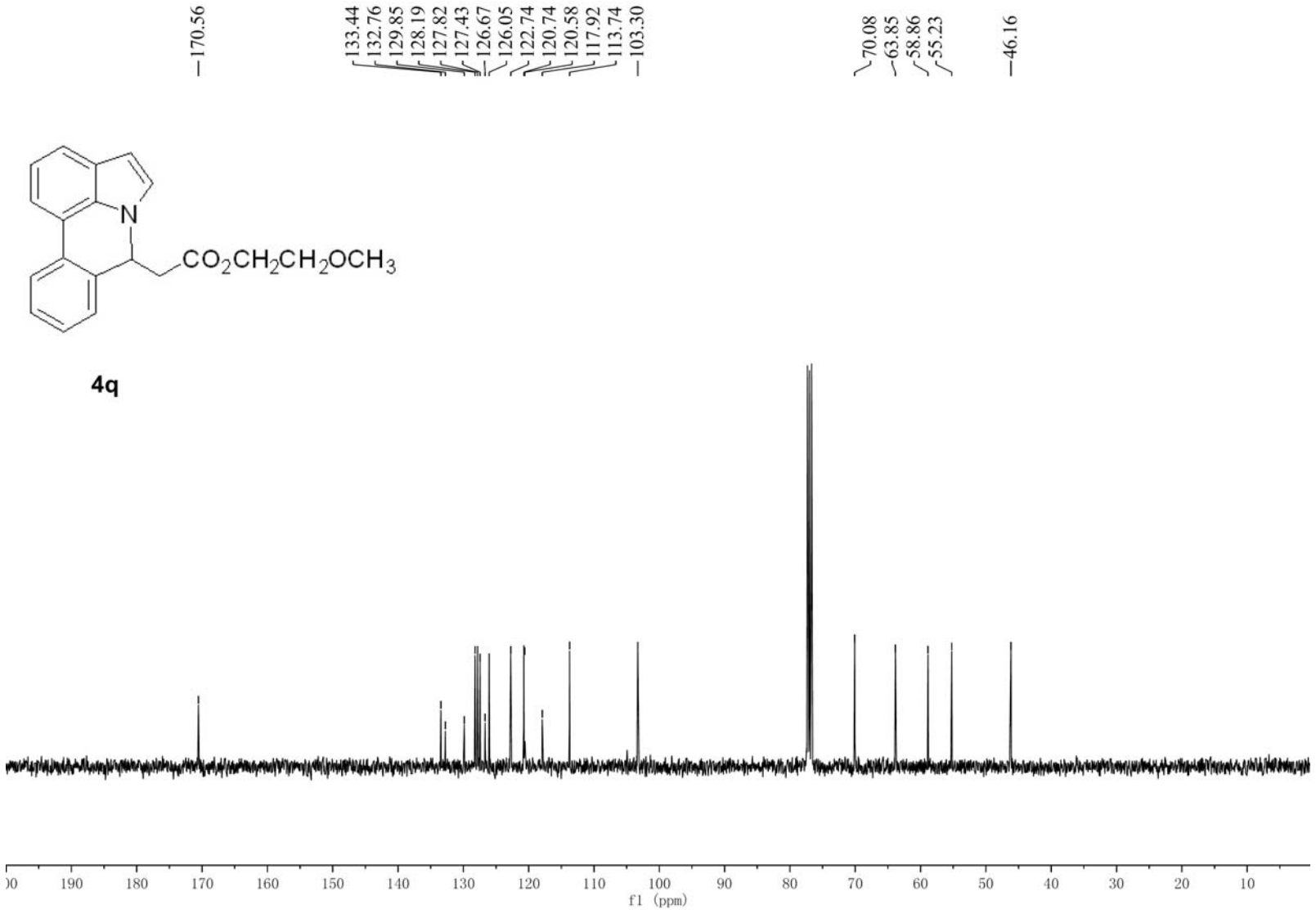
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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127.82
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126.67
126.05
122.74
120.74
120.58
117.92
113.74
103.30
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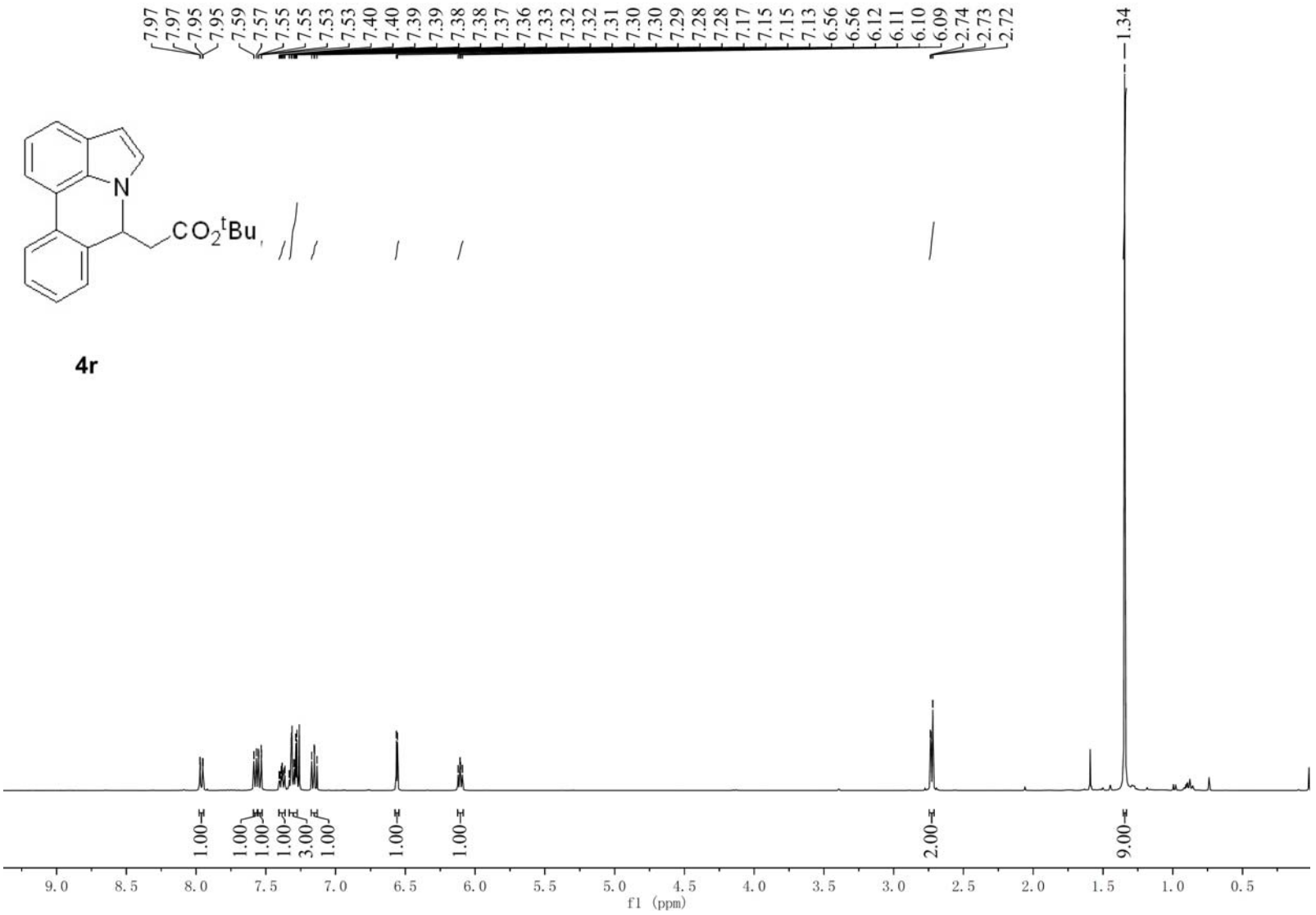
63.85
58.86
55.23
46.16
N
CO2CH2CH2OCH3
4q
f1 (ppm)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7.95
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7.55
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7.53
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7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.28
7.28
7.17
7.15
7.15
7.13
6.56
6.56
6.12
6.11
6.10
6.09
2.74
2.73
2.72
1.34
N
CO2tBu
1.00
1.00
1.00
3.00
1.00
1.00
1.00
2.00
9.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
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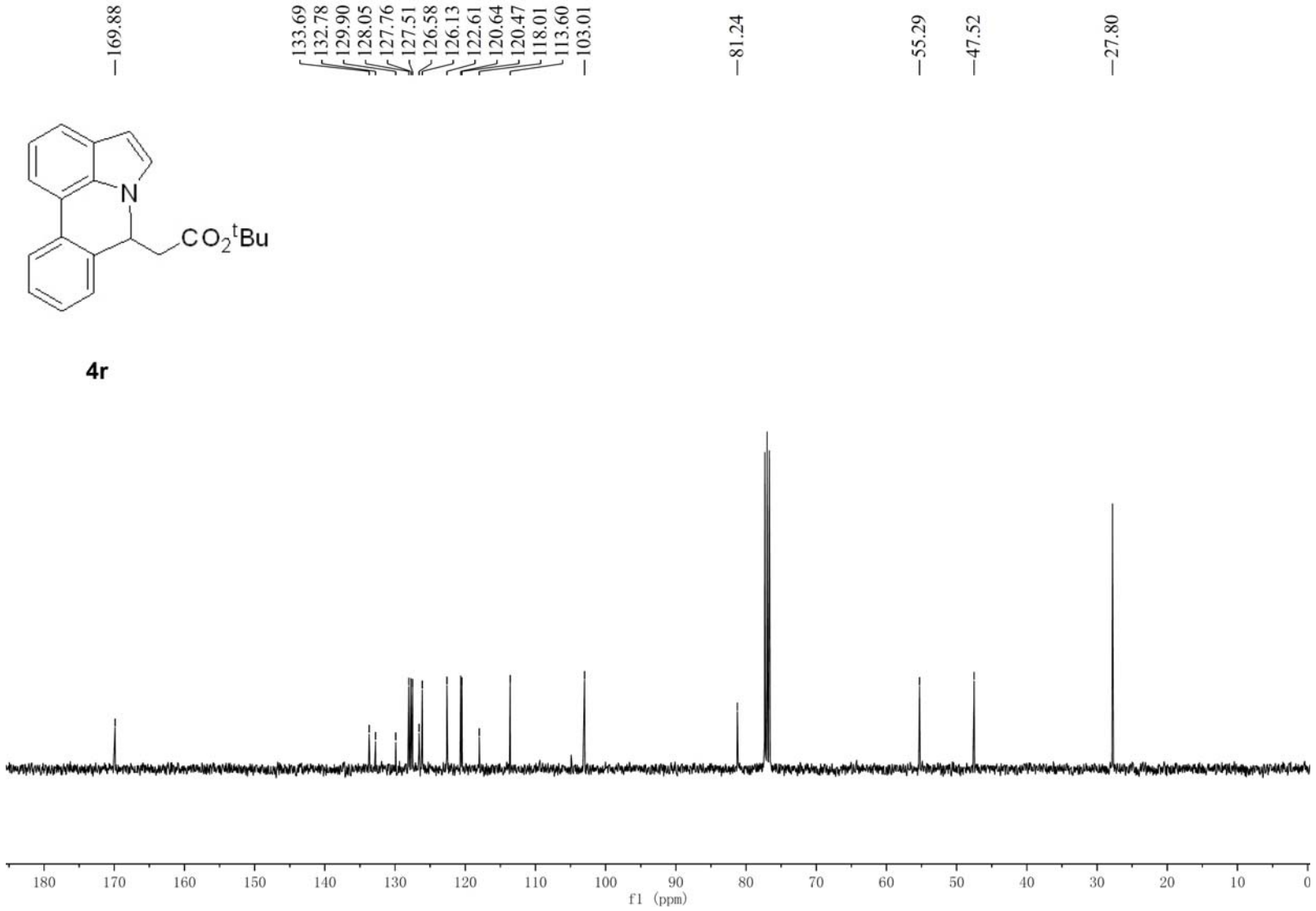
1.0
0.5
f1 (ppm)

4r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127.76
127.51
126.58
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120.64
120.47
118.01
113.60
103.01
81.24
55.29
47.52
27.80
N
CO2tBu
4r
180
170
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130
120
110
100
90
80
70
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50
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30
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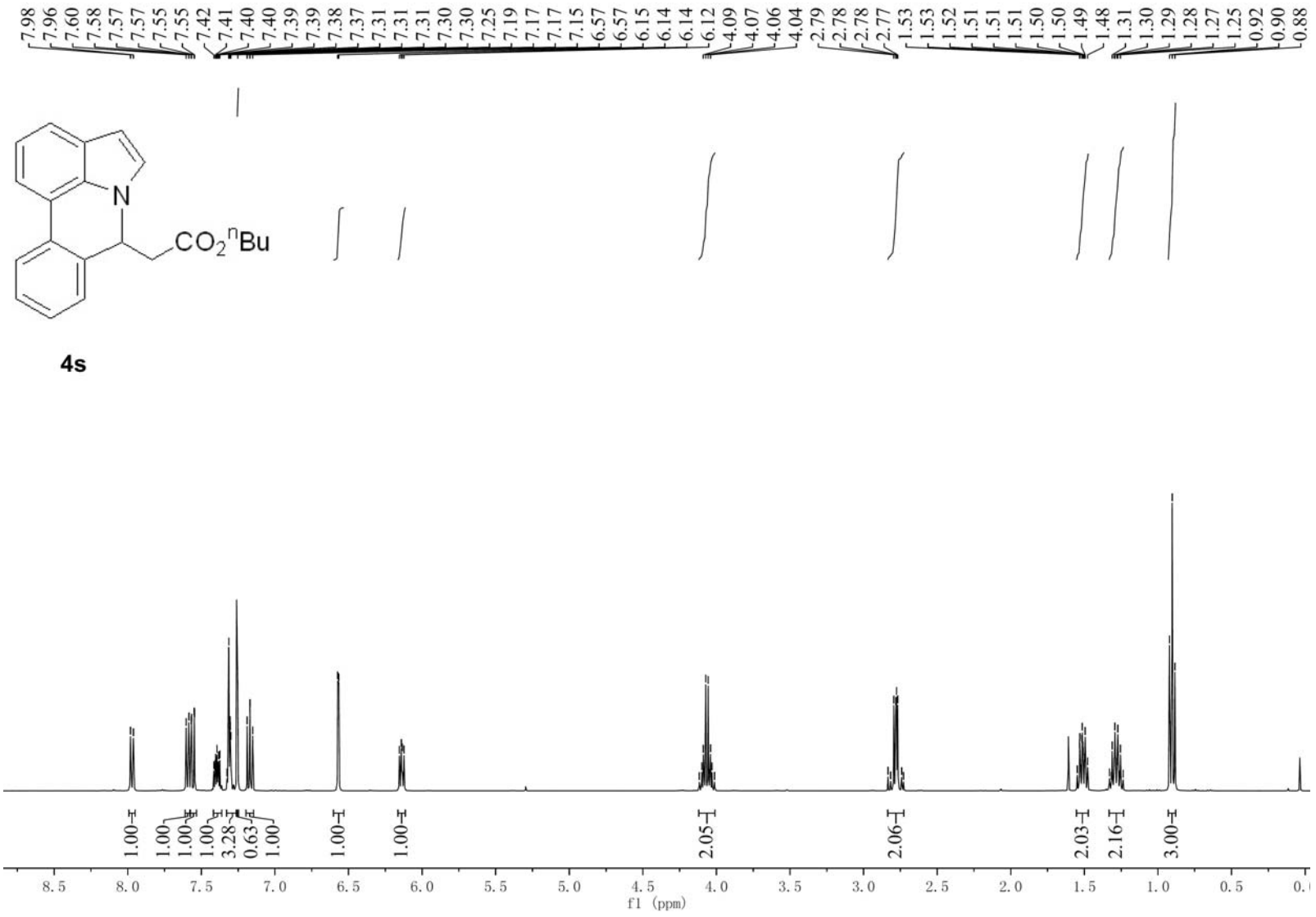
f1 (ppm)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7.39
7.38
7.37
7.31
7.31
7.31
7.30
7.30
7.25
7.19
7.17
7.17
7.15
6.57
6.57
6.15
6.14
6.14
6.12
4.09
4.07
4.06
4.04
2.79
2.78
2.78
2.77
1.53
1.53
1.52
1.51
1.51
1.51
1.50
1.50
1.49
1.48
1.31
1.30
1.29
1.28
1.27
1.25
0.92
0.90
0.88
N
CO2nBu
4s
1.00
1.00
1.00
1.00
3.28
0.63
1.00
1.00
1.00
2.05
2.06
2.03
2.16
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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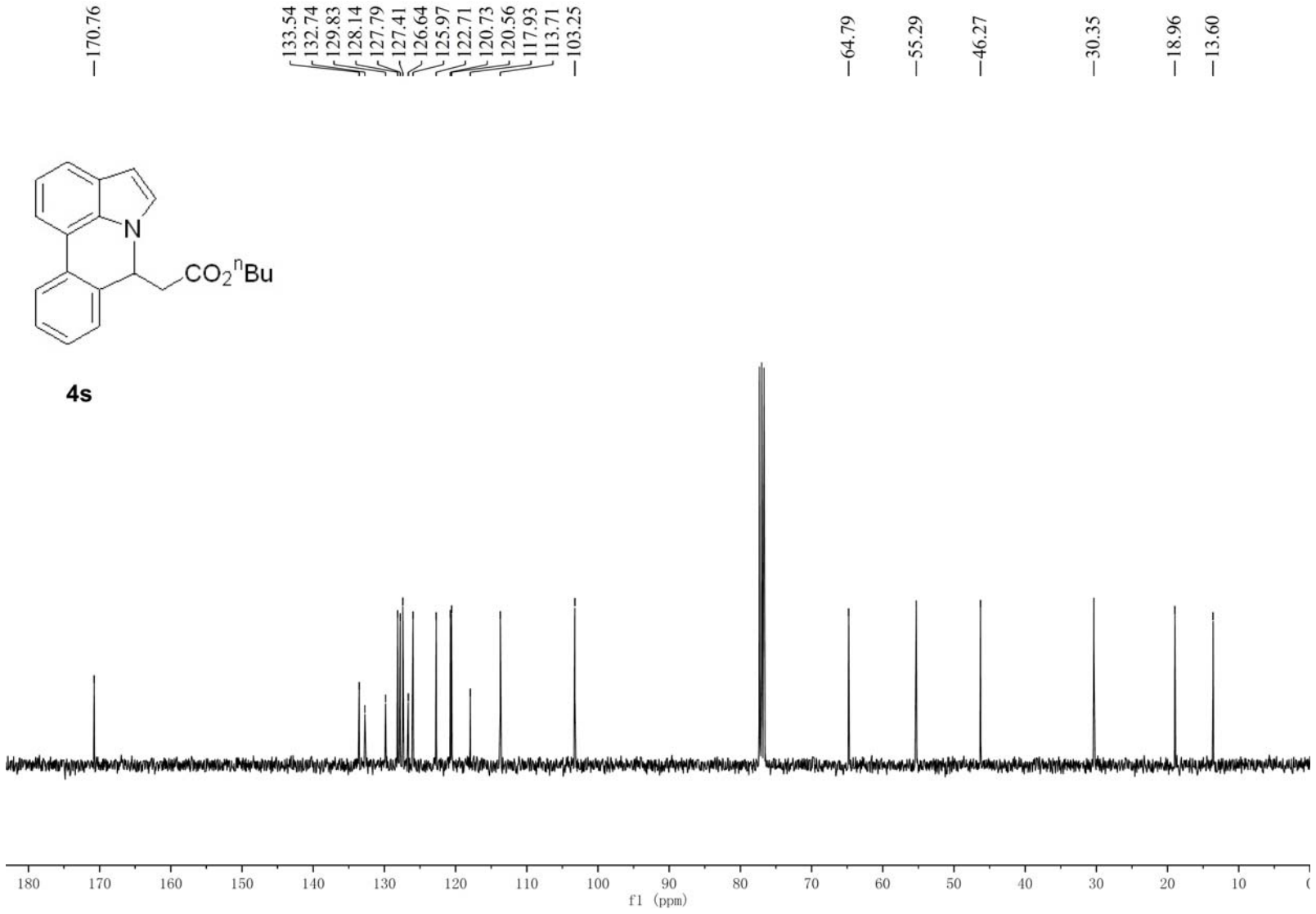
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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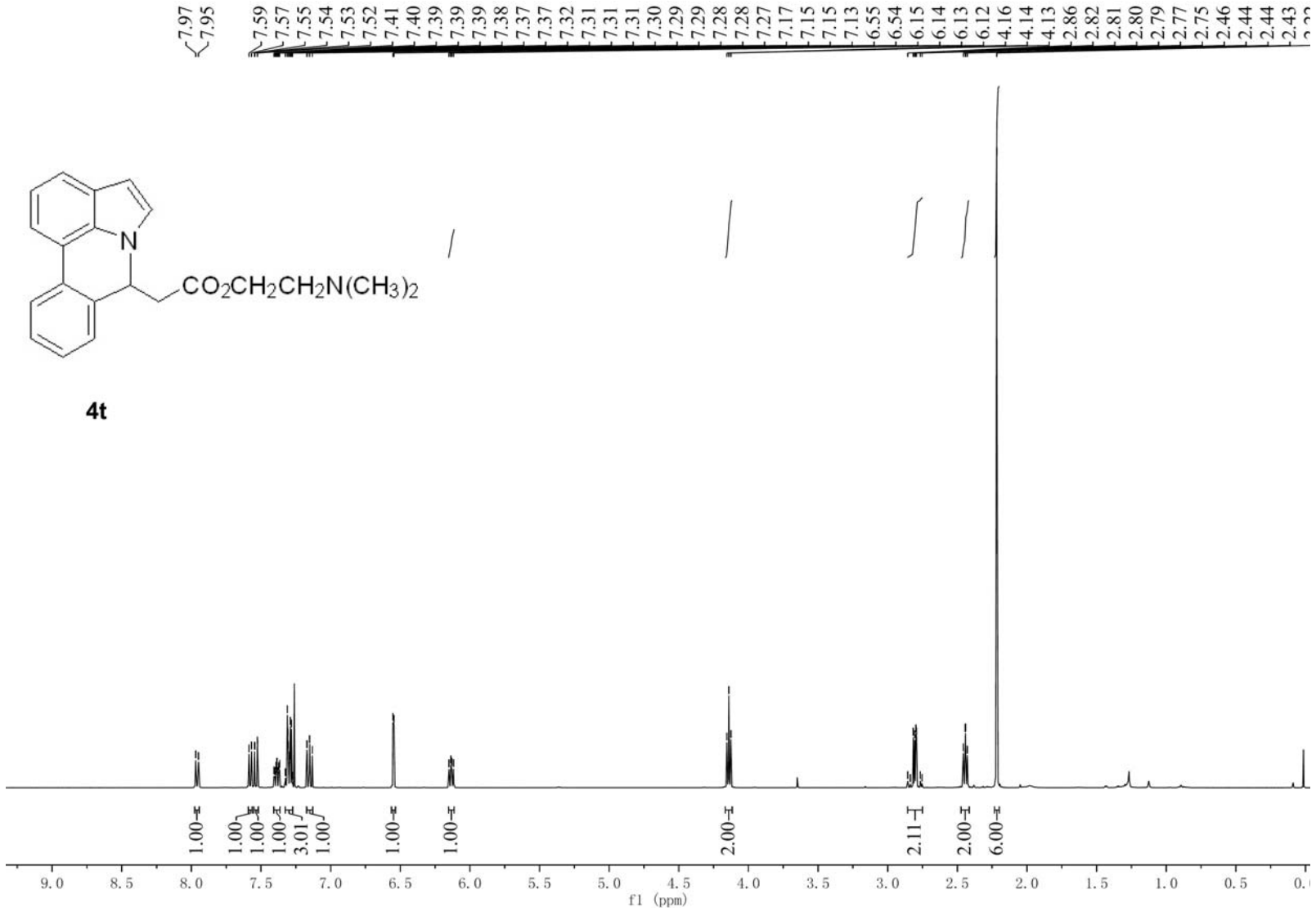
127.41
126.64
125.97
122.71
120.73
120.56
117.93
113.71
103.25
64.79
55.29
46.27
30.35
18.96
13.60
N
CO2nBu
4s
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.39
7.39
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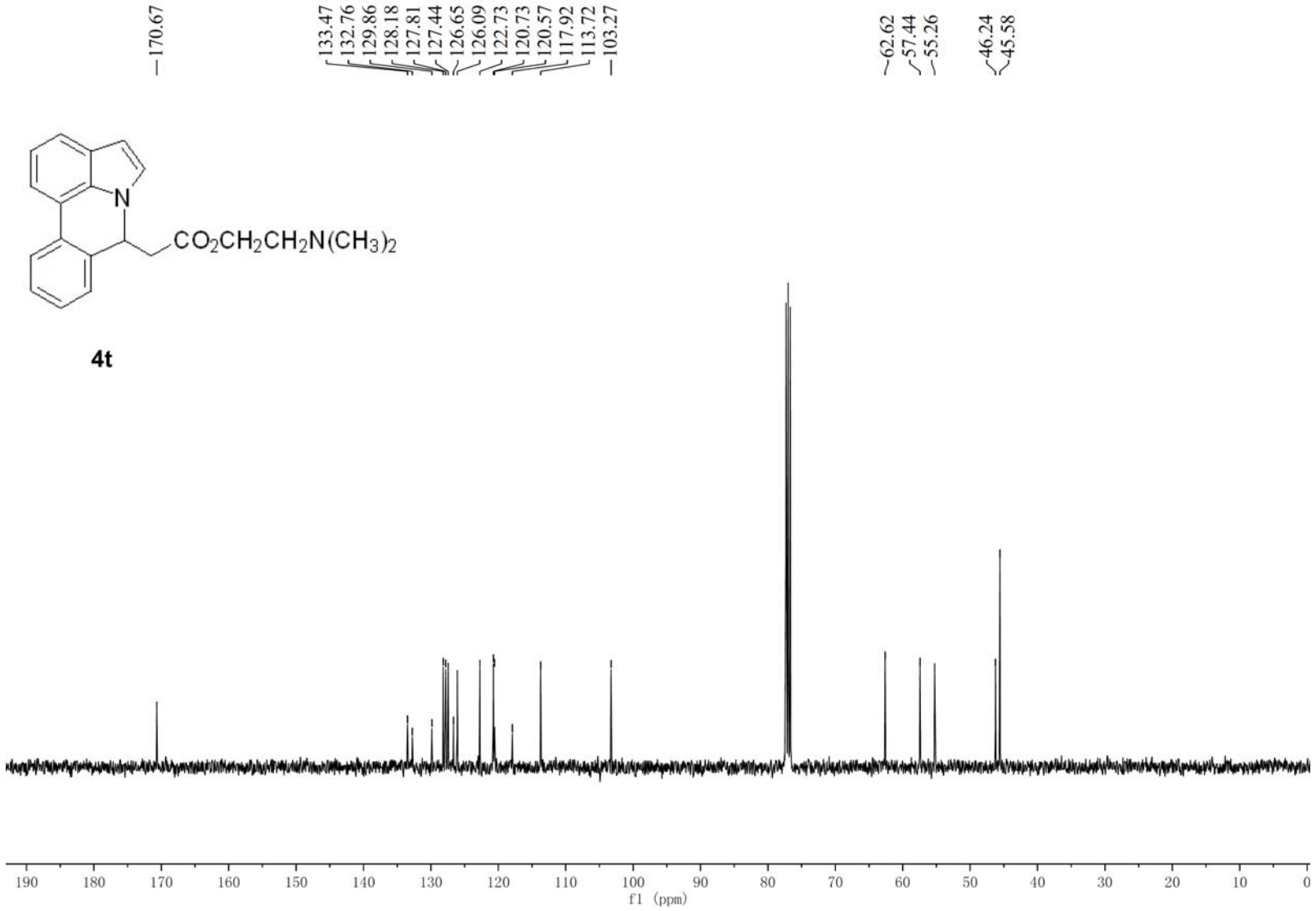
7.38
7.37
7.37
7.32
7.31
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.17
7.15
7.15
7.13
6.55
6.54
6.15
6.14
6.13
6.12
4.16
4.14
4.13
2.86
2.82
2.81
2.80
2.79
2.77
2.75
2.46
2.44
2.44
2.43
N
CO2CH2CH2N(CH3)2
4t
1.00
1.00
1.00
1.00
3.01
1.00
1.00
1.00
2.00
2.11
2.00
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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127.81
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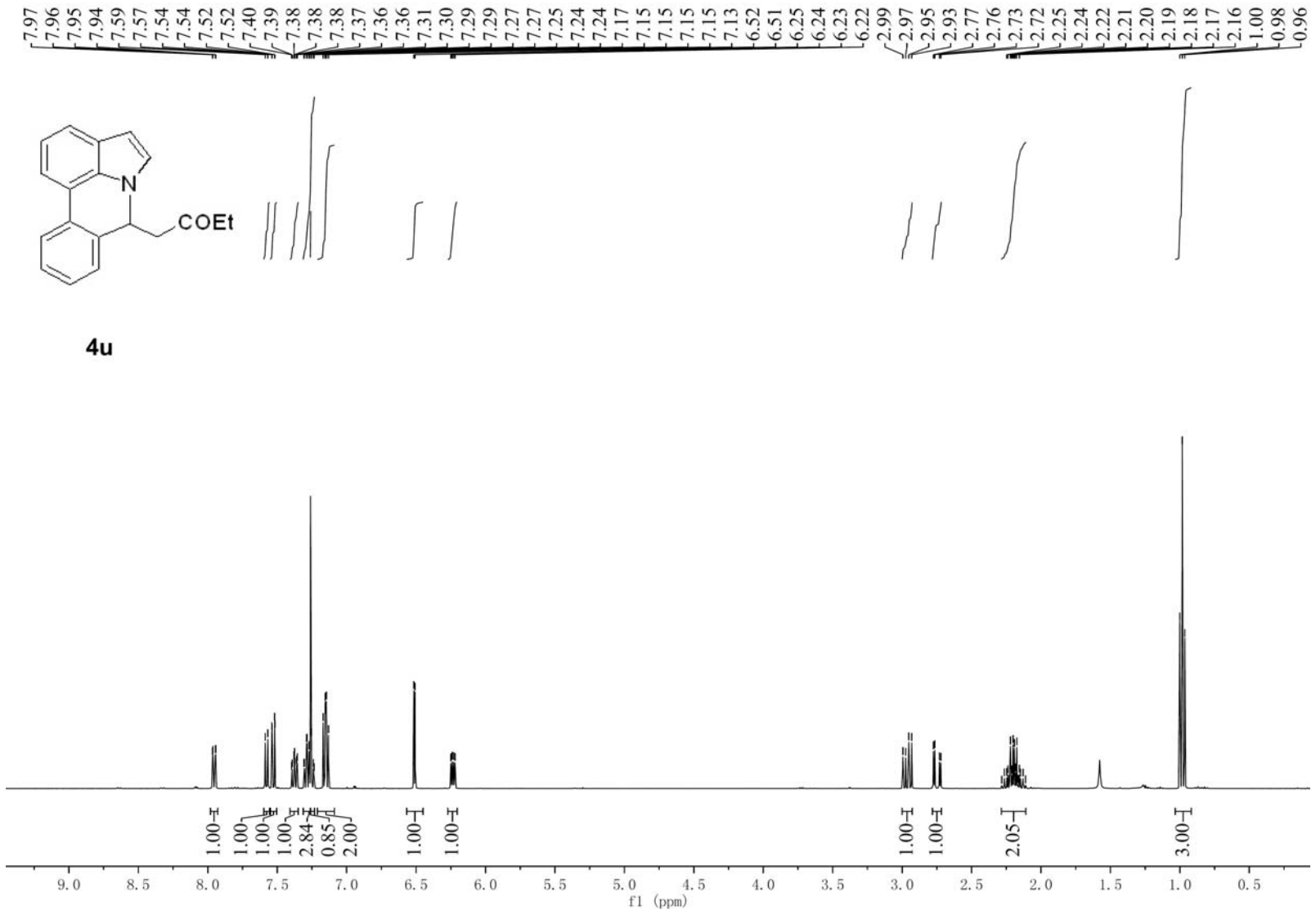
126.65
126.09
122.73
120.73
120.57
117.92
113.72
103.27
62.62
57.44
55.26
46.24
45.58
N
CO2CH2CH2N(CH3)2
4t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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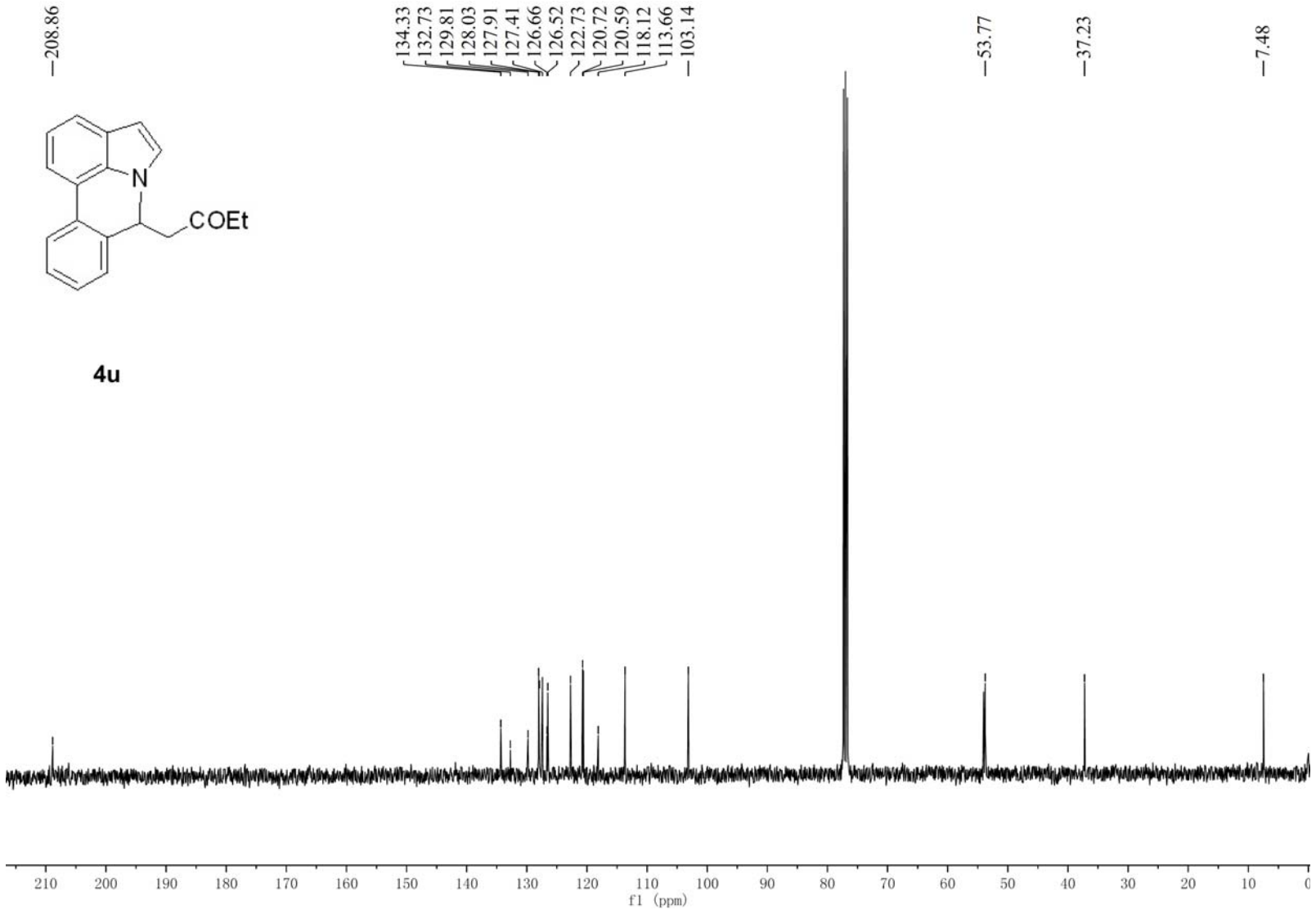
7.52
7.40
7.39
7.38
7.38
7.38
7.37
7.36
7.36
7.31
7.30
7.29
7.29
7.27
7.27
7.25
7.24
7.24
7.17
7.15
7.15
7.15
7.15
7.13
6.52
6.51
6.25
6.24
6.23
6.22
2.99
2.97
2.95
2.93
2.77
2.76
2.73
2.72
2.25
2.24
2.22
2.21
2.20
2.19
2.18
2.17
2.16
1.00
0.98
0.96
N
COEt
4u
1.00
1.00
1.00
1.00
2.84
0.85
2.00
1.00
1.00
1.00
1.00
2.05
3.00
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)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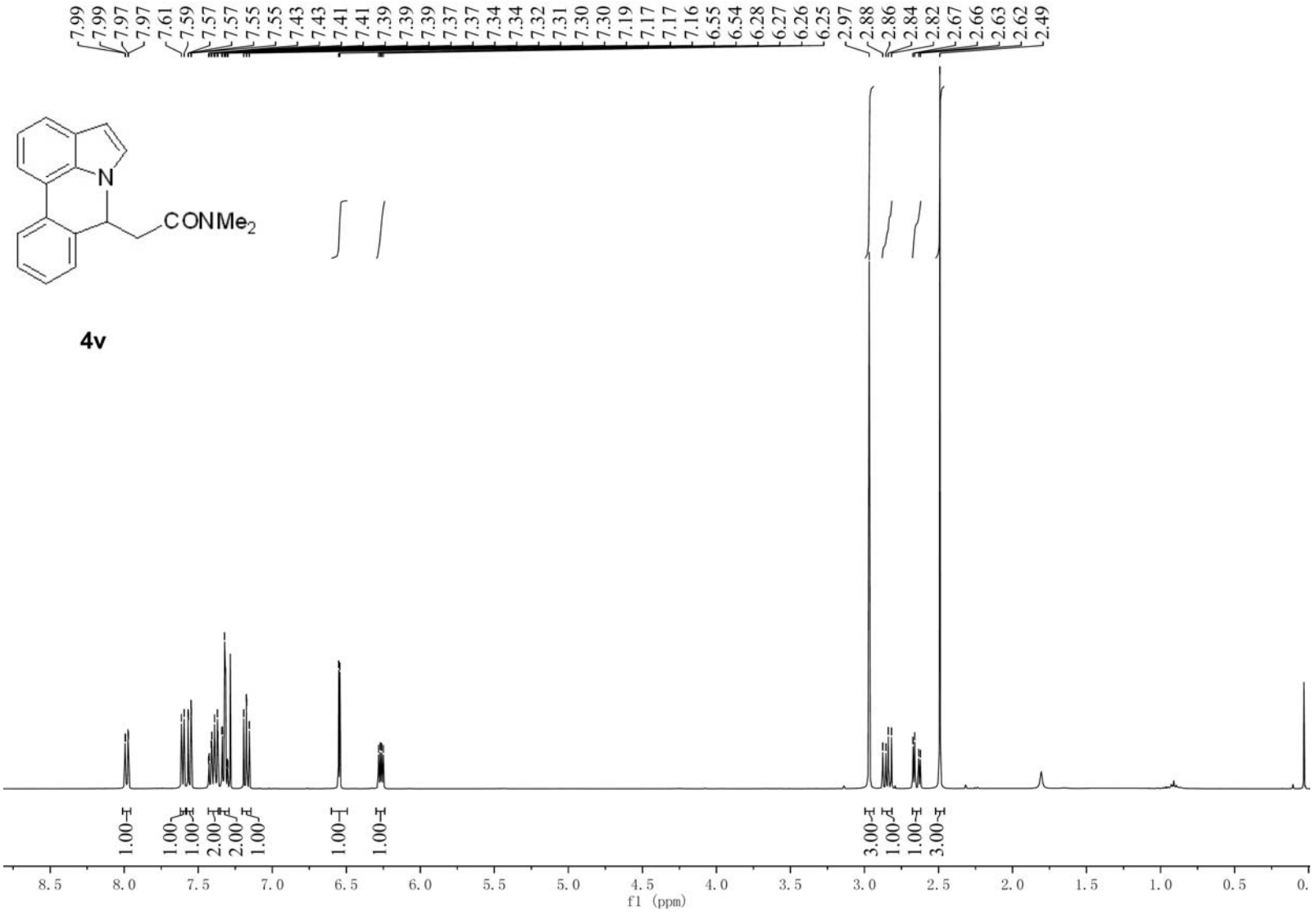
127.41
126.66
126.52
122.73
120.72
120.59
118.12
113.66
103.14
53.77
37.23
7.48
N
COEt
4u
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.97
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7.61
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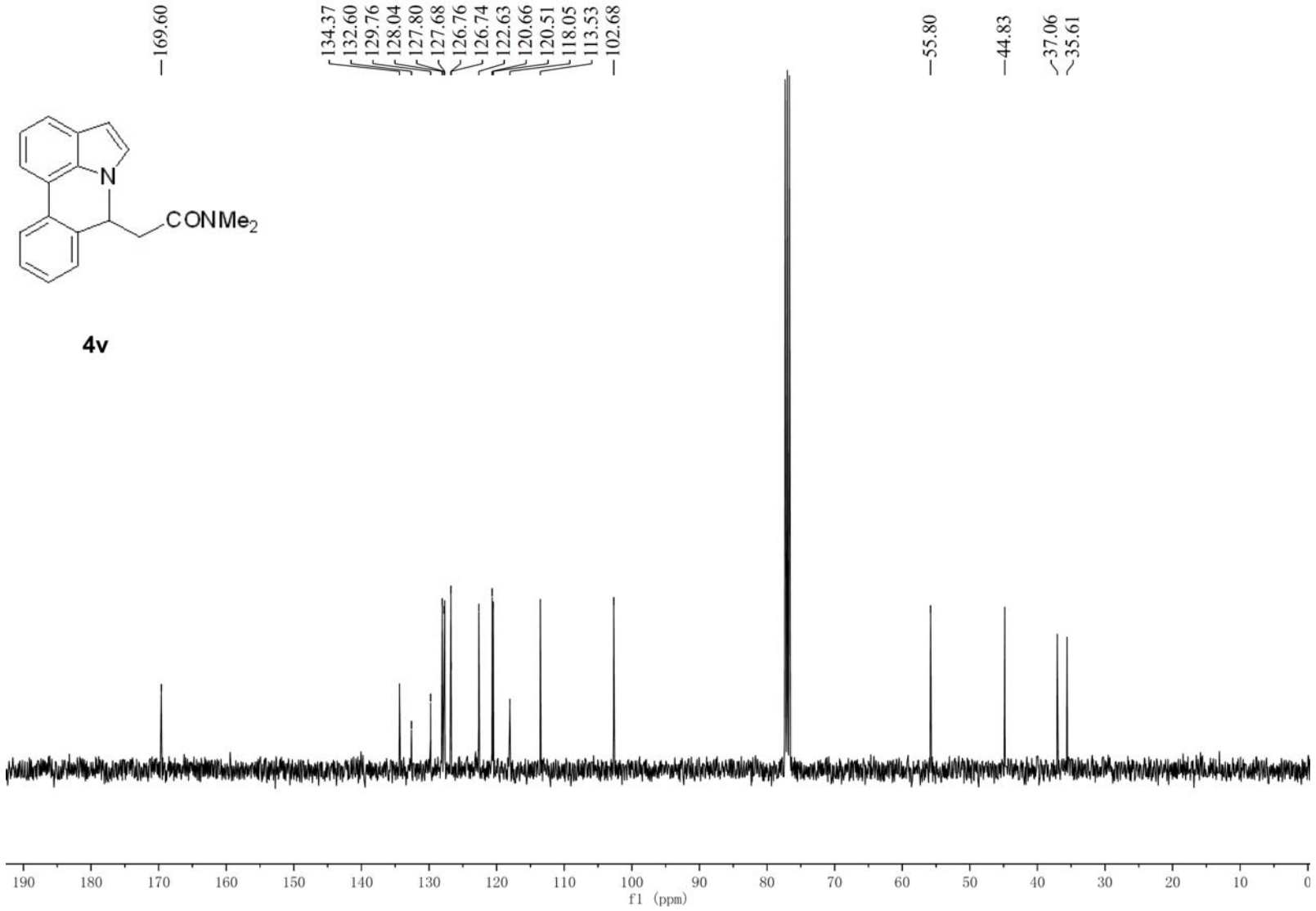
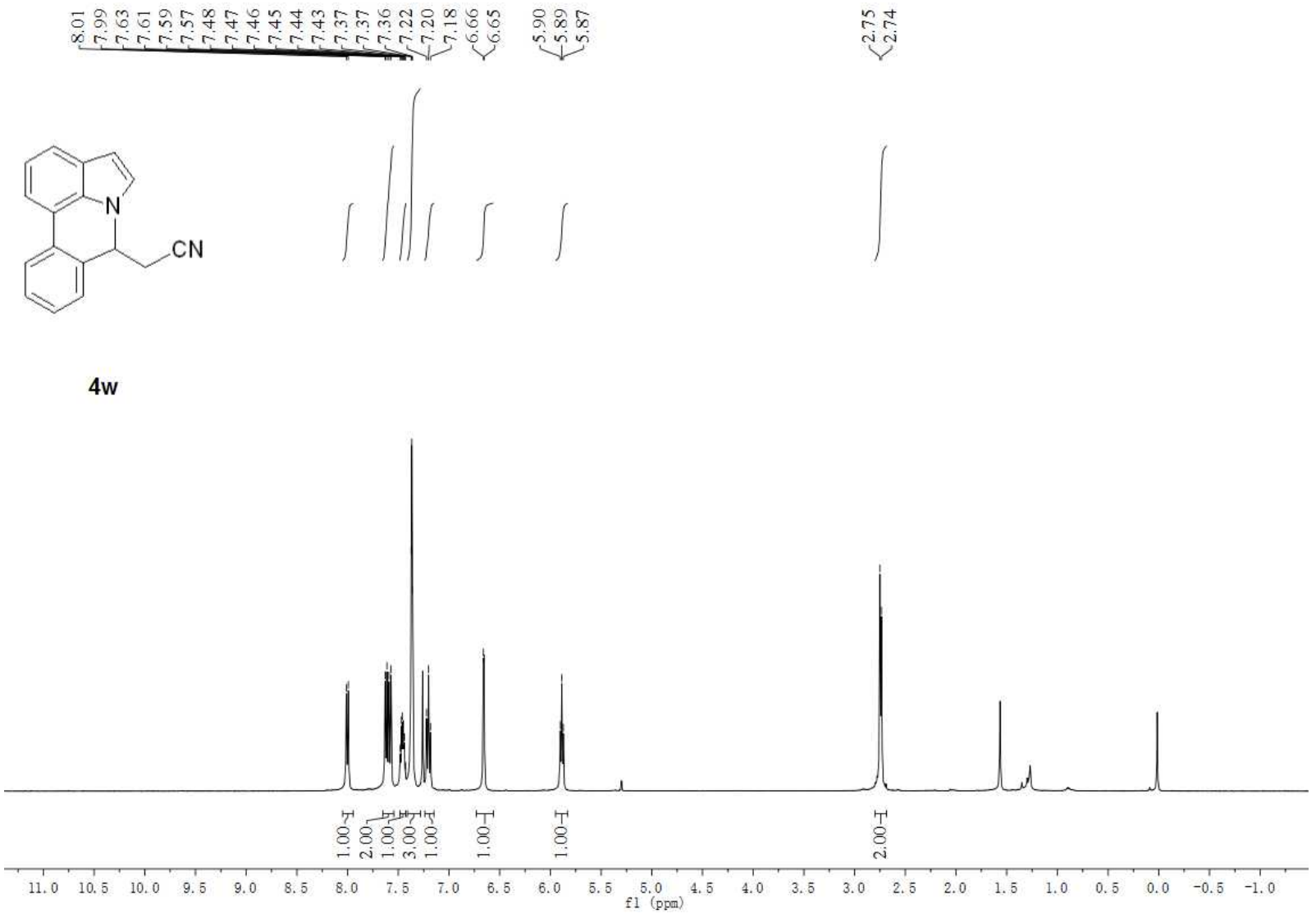
7.57
7.57
7.55
7.55
7.43
7.43
7.41
7.41
7.39
7.39
7.39
7.37
7.37
7.34
7.34
7.32
7.31
7.30
7.30
7.19
7.17
7.17
7.16
6.55
6.54
6.28
6.27
6.26
6.25
2.97
2.88
2.86
2.84
2.82
2.67
2.66
2.63
2.62
2.49
N
CONMe2
4v
1.00
1.00
1.00
2.00
2.00
1.00
1.00
1.00
3.00
1.00
1.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)


169.60
134.37
132.60
129.76
128.04
127.80
127.68
126.76
126.74
122.63
120.66
120.51
118.05
113.53
102.68
55.80
44.83
37.06
35.61
N
CONMe2
4v
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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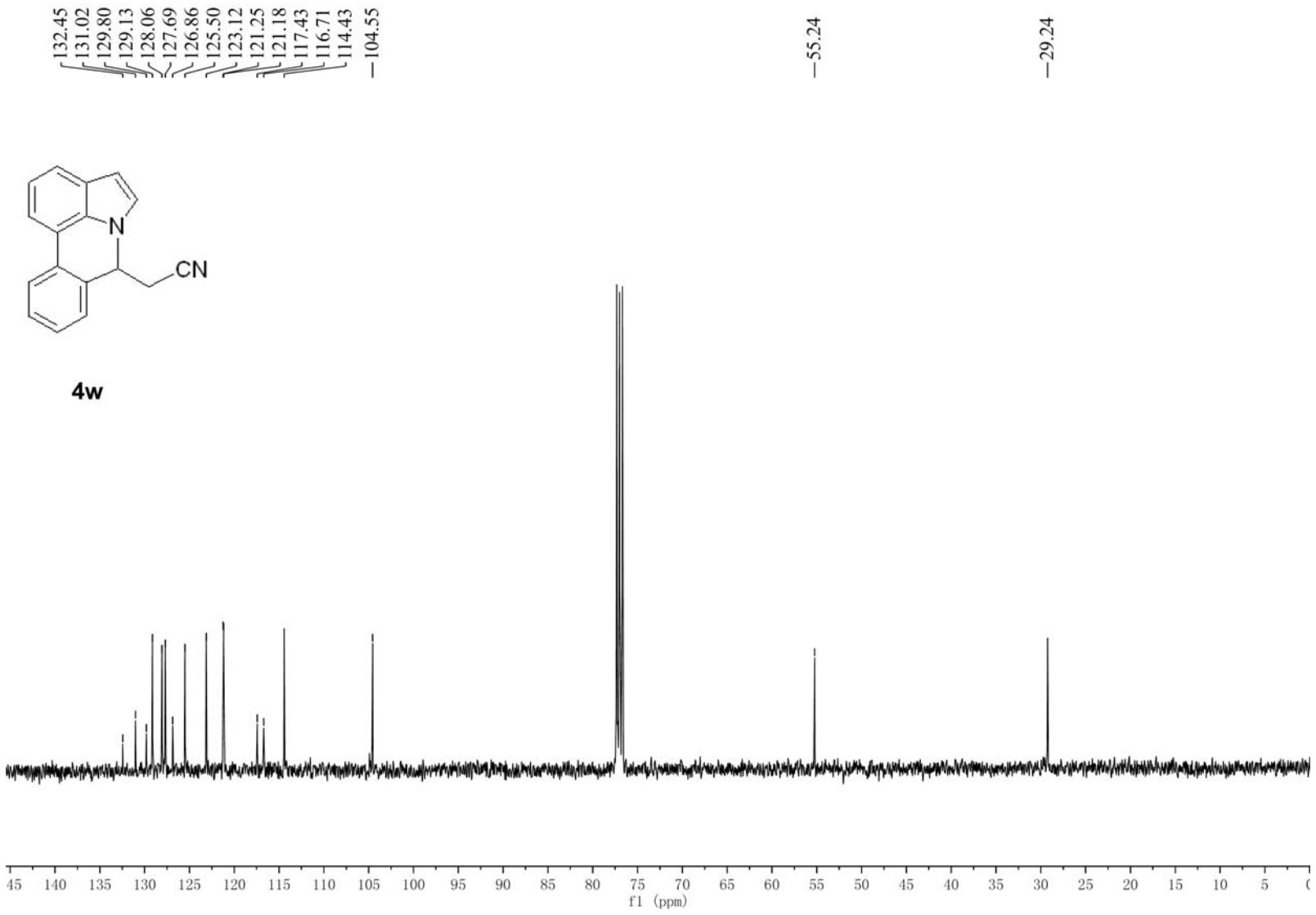
7.47
7.46
7.45
7.44
7.43
7.37
7.37
7.36
7.22
7.20
7.18
6.66
6.65
5.90
5.89
5.87
2.75
2.74
N
CN
1.00
2.00
1.00
3.00
1.00
1.00
1.00
2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

4w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131.02
129.80
129.13
128.06
127.69
126.86
125.50
123.12
121.25
121.18
117.43
116.71
114.43
104.55
55.24
29.24
N
CN
4w
45 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)