

Electronic Supplementary Information

Construction of Dispirocyclohexanes via Amine-Catalyzed [2 + 2 + 2]

Annulations of Morita-Baylis-Hillman Acetates with Exocyclic Alkenes

Rongshun Chen,^a Silong Xu,^{b*} Xia Fan,^a Hanyuan Li,^a Yuhai Tang,^b and Zhengjie He^{a*}

^a The State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Collaborative Innovation Center of Chemical Science and Engineering (Tianjin), 94 Weijin Road, Tianjin 300071, P. R. China.

Fax: +86 22 23501520; e-mail: zhengjiehe@nankai.edu.cn

^b Department of Chemistry, School of Science, Xi'an Jiaotong University, Xi'an 710049, P. R. China.

Fax: +86 29 82655399; e-mail: silongxu@mail.xjtu.edu.cn

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I. ORTEP Representation of 3r and 5f

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

Identification code	3r
Empirical formula	C ₄₃ H ₃₂ F ₆ O ₆
Formula weight	758.69
Temperature	173(2) K
Wavelength	0.71073 Å,
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 12.174(2) Å α = 90 ° b = 21.206(4) Å β = 92.65(3) ° c = 14.570(3) Å, γ = 90 °
Volume	3757.4(13) Å ³
Z, Calculated density	4, 1.379 Mg/m ³
Absorption coefficient	0.145 mm ⁻¹
F(000)	16100
Crystal size	0.20 x 0.18 x 0.12 mm ³
Theta range for data collection	1.70 to 27.94 °
Limiting indices	-16 ≤ h ≤ 14, -27 ≤ k ≤ 27, -19 ≤ l ≤ 19
Reflections collected / unique	37451 / 8963 [R(int) = 0.0769]
Completeness to the θ = 25.02°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9828 and 0.9716
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8963 / 78 / 551
Goodness-of-fit on F ²	0.981
Final R indices[I > 2σ(I)]	R1 = 0.0613, wR2 = 0.1365
R indices (all data)	R1 = 0.1068, wR2 = 0.1590
Largest diff. peak and hole	0.296 and -0.324 e. Å ⁻³

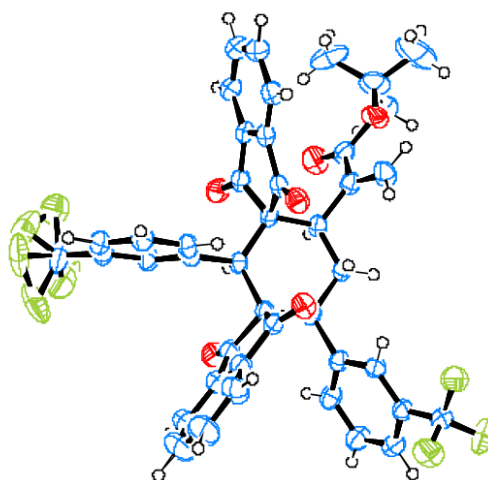
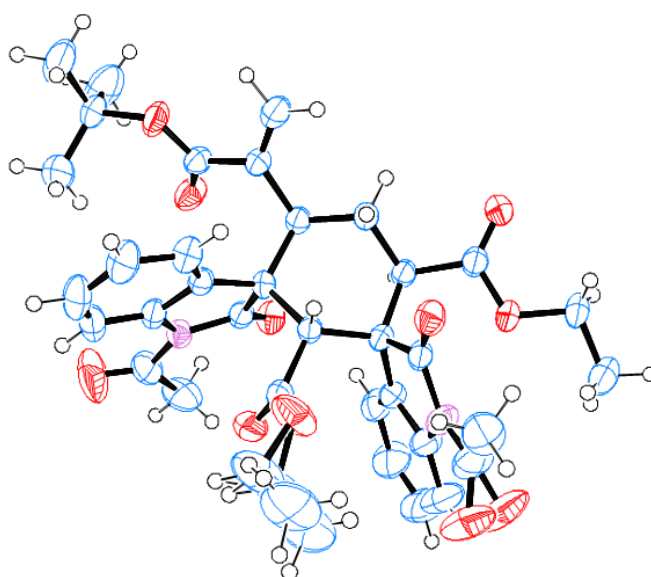


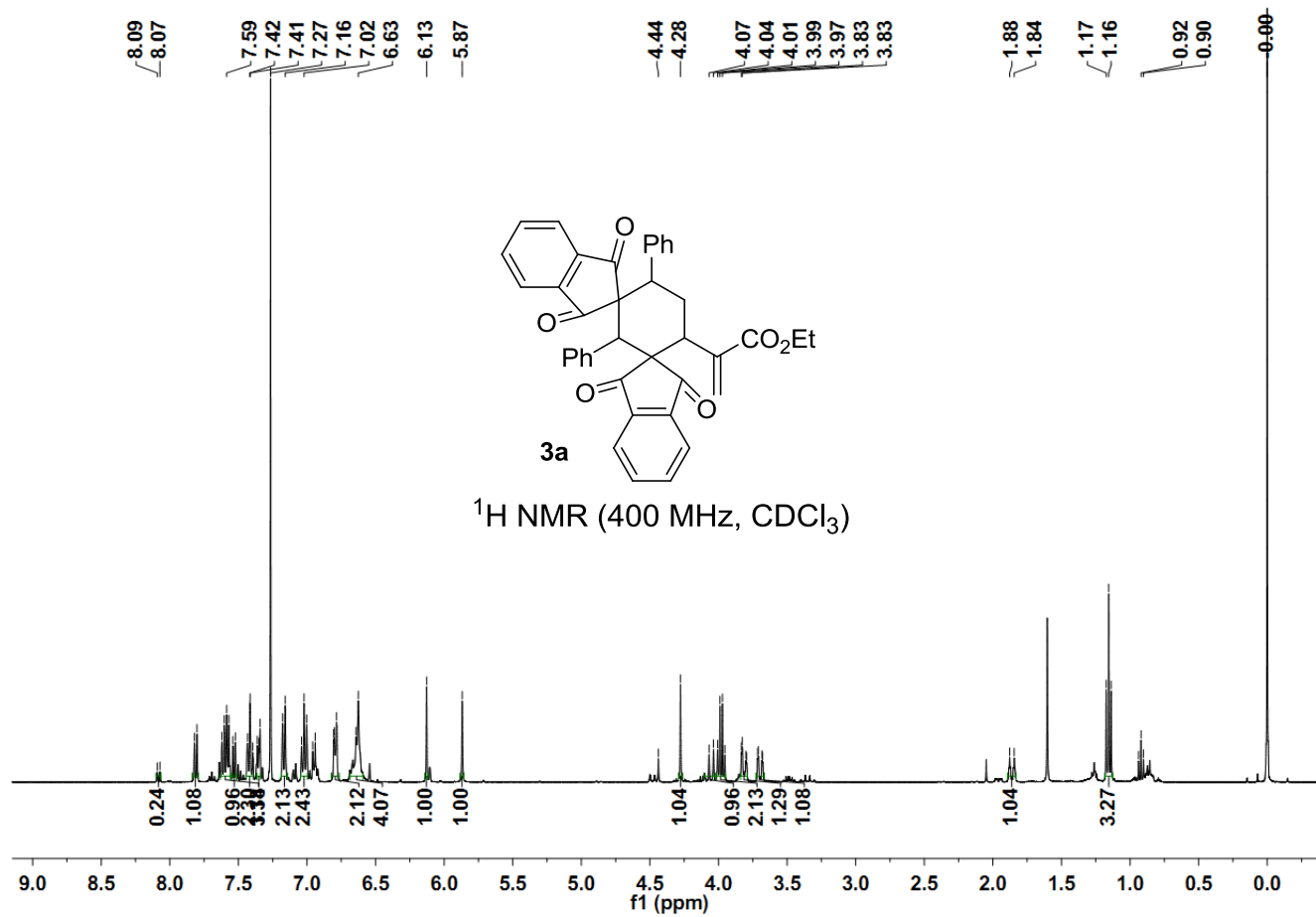
Figure S1. ORTEP Drawing for **3r**

Table S2. Crystal data and structure refinement for **5f**

Identification code	5f
Empirical formula	C ₃₇ H ₄₁ N ₂ O ₁₀
Formula weight	673.72
Temperature	293(2) K
Wavelength	0.71073 Å,
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 10.530(2) Å α = 65.69(3) ° b = 13.602(3) Å β = 80.28(3) ° c = 13.650(3) Å, γ = 81.84(3) °
Volume	1750.5(6) Å ³
Z, Calculated density	2, 1.278 Mg/m ³
Absorption coefficient	0.093 mm ⁻¹
F(000)	714
Crystal size	0.20 x 0.18 x 0.12 mm ³
Theta range for data collection	1.81 to 29.07 °
Limiting indices	-14 ≤ h ≤ 14, -18 ≤ k ≤ 14, -18 ≤ l ≤ 17
Reflections collected / unique	18820 / 8967 [R(int) = 0.0425]
Completeness to the θ = 25.02°	95.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9889 and 0.9816
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8967 / 84 / 472
Goodness-of-fit on F ²	0.998
Final R indices[I > 2σ(I)]	R1 = 0.0535, wR2 = 0.1128
R indices (all data)	R1 = 0.1238, wR2 = 0.1361
Largest diff. peak and hole	0.366 and -0.361 e. Å ⁻³

**Figure S2.** ORTEP Drawing for **5f**

II. NMR Spectra for 3 and 5



202.83
200.90
200.23
200.04

166.39

135.54
135.09
134.72
134.33

122.61
122.38
122.22
122.02

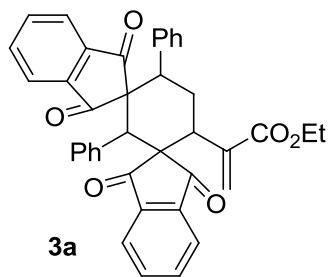
77.32
77.00
76.68

63.09
60.92

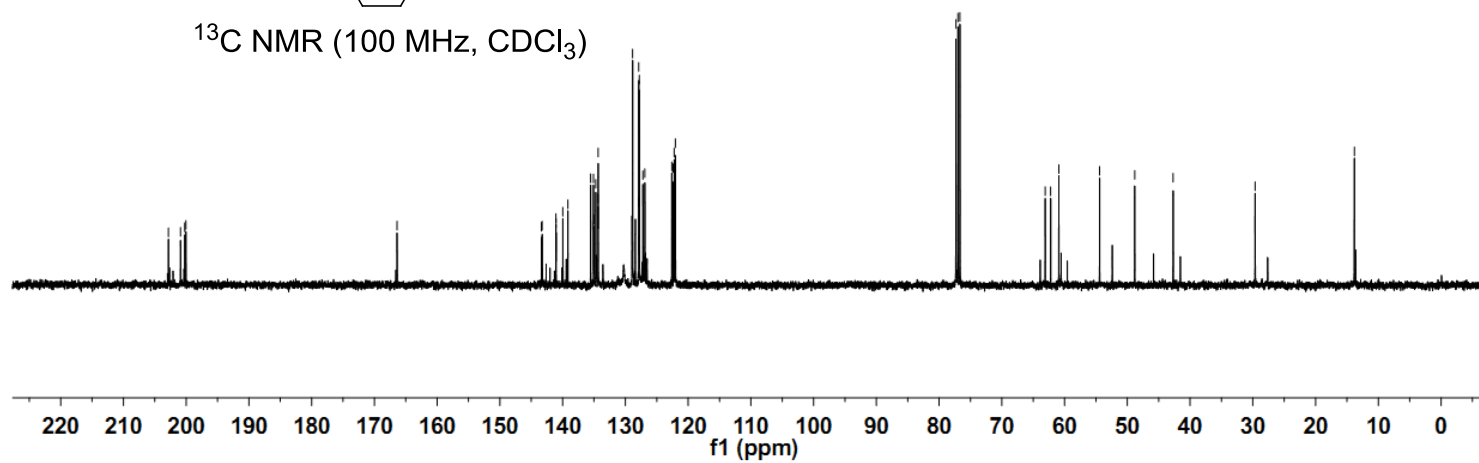
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48.83
42.70

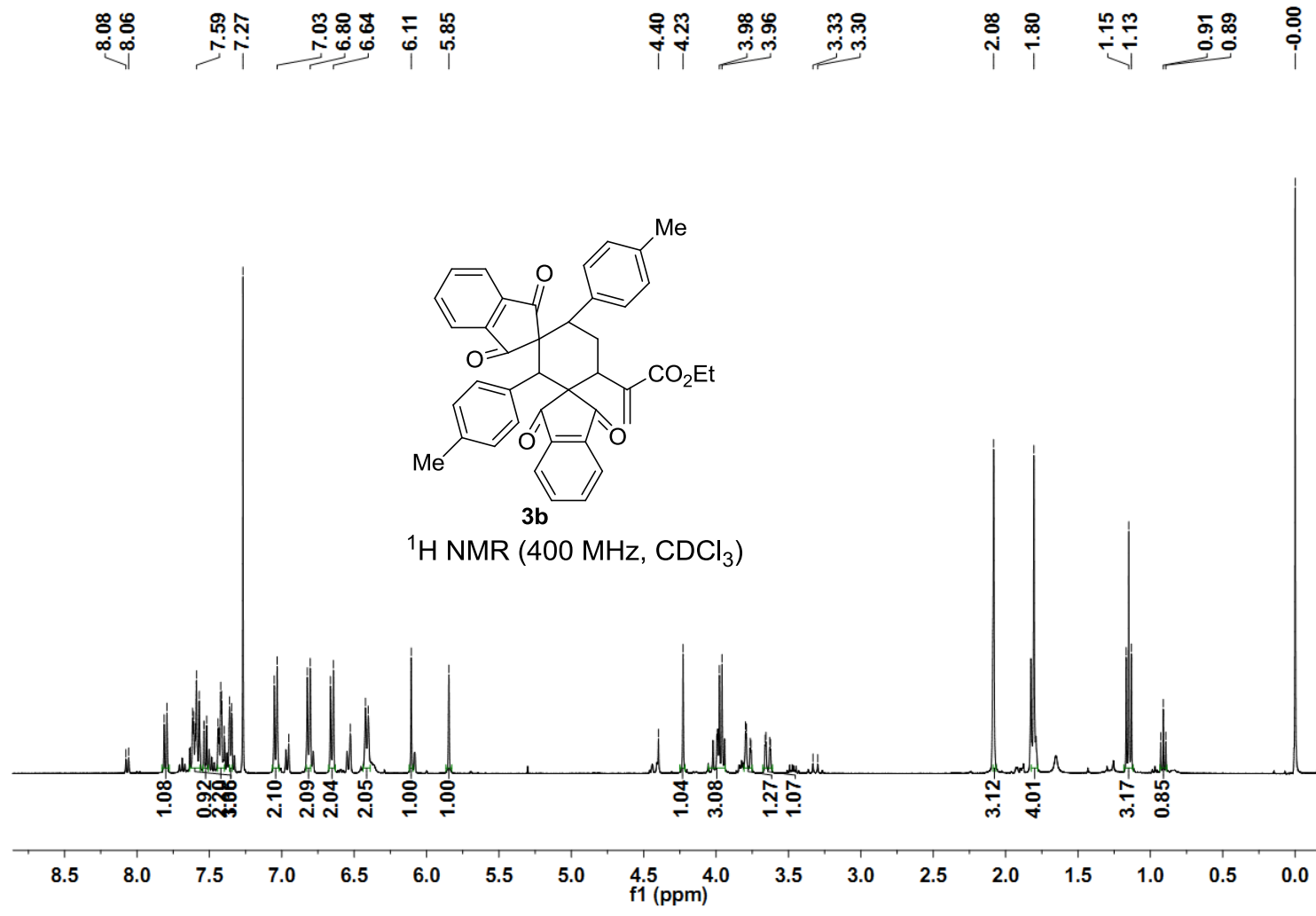
29.64

13.82



^{13}C NMR (100 MHz, CDCl_3)





203.08
201.05
200.42
200.14

166.41

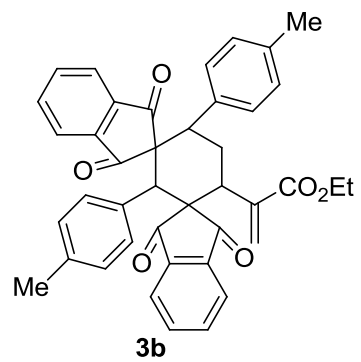
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128.69
128.54
128.42

122.59
122.37
122.21
122.01

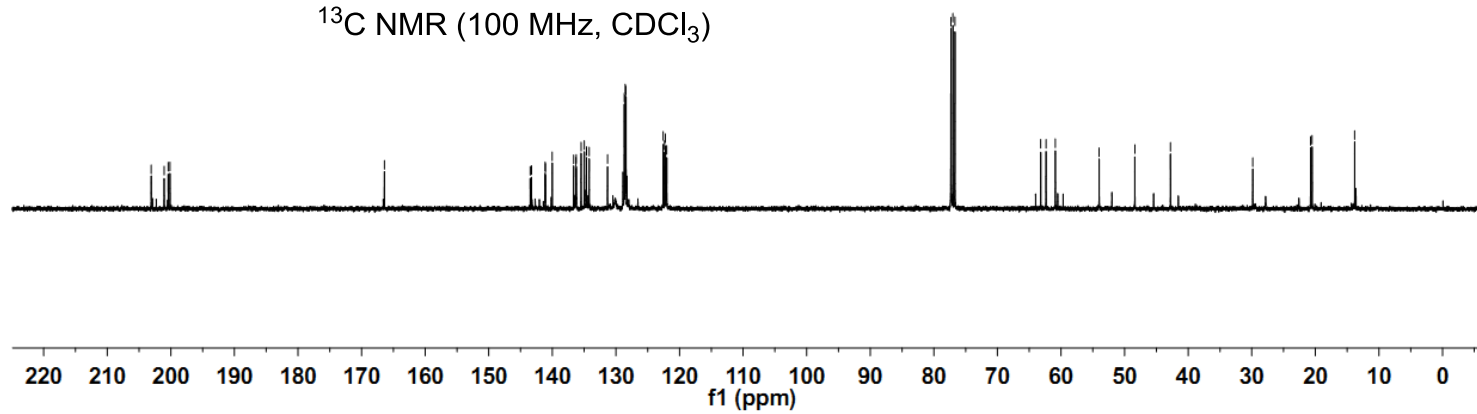
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76.68

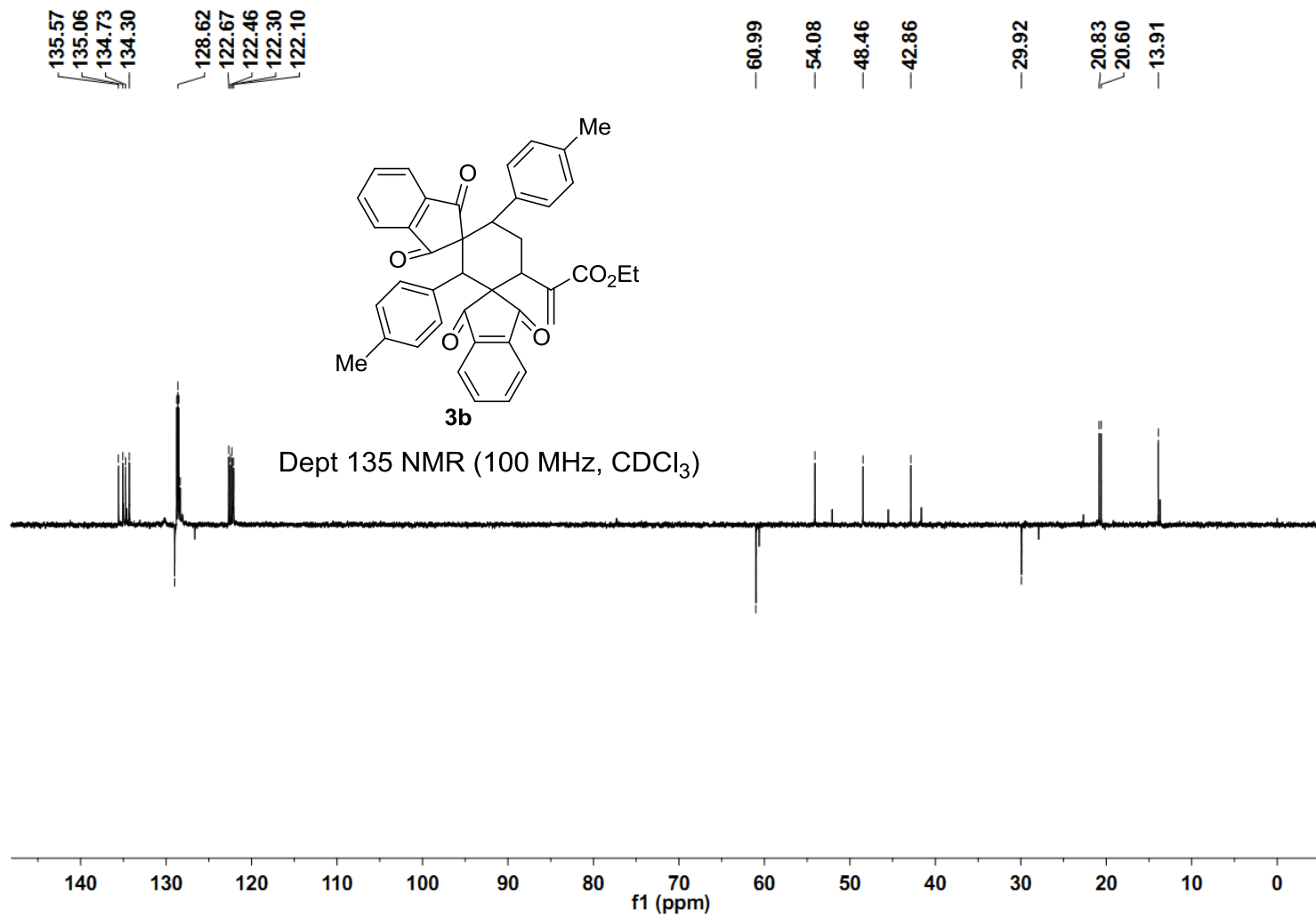
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48.39
42.78

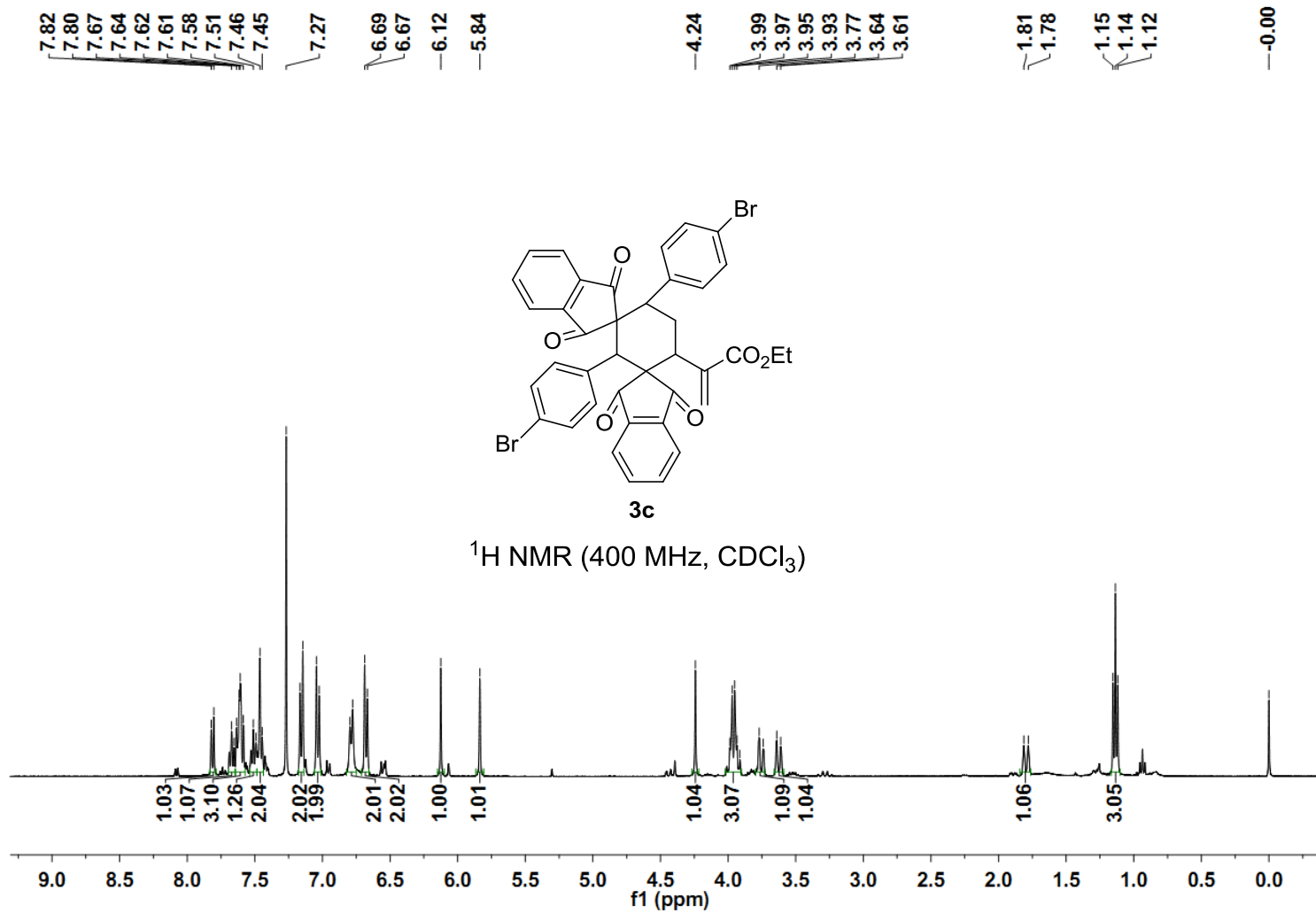
29.84
20.51
13.82

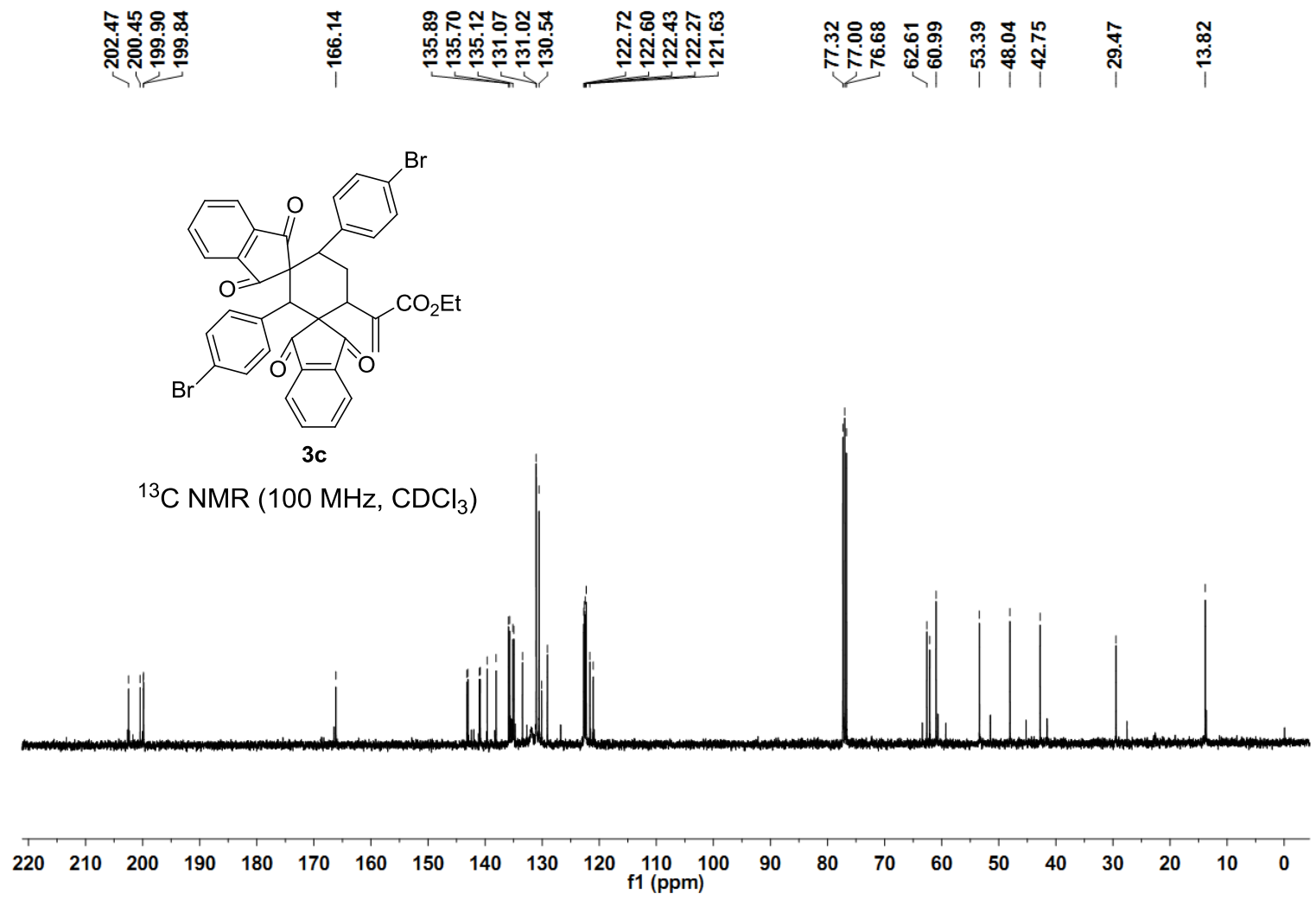


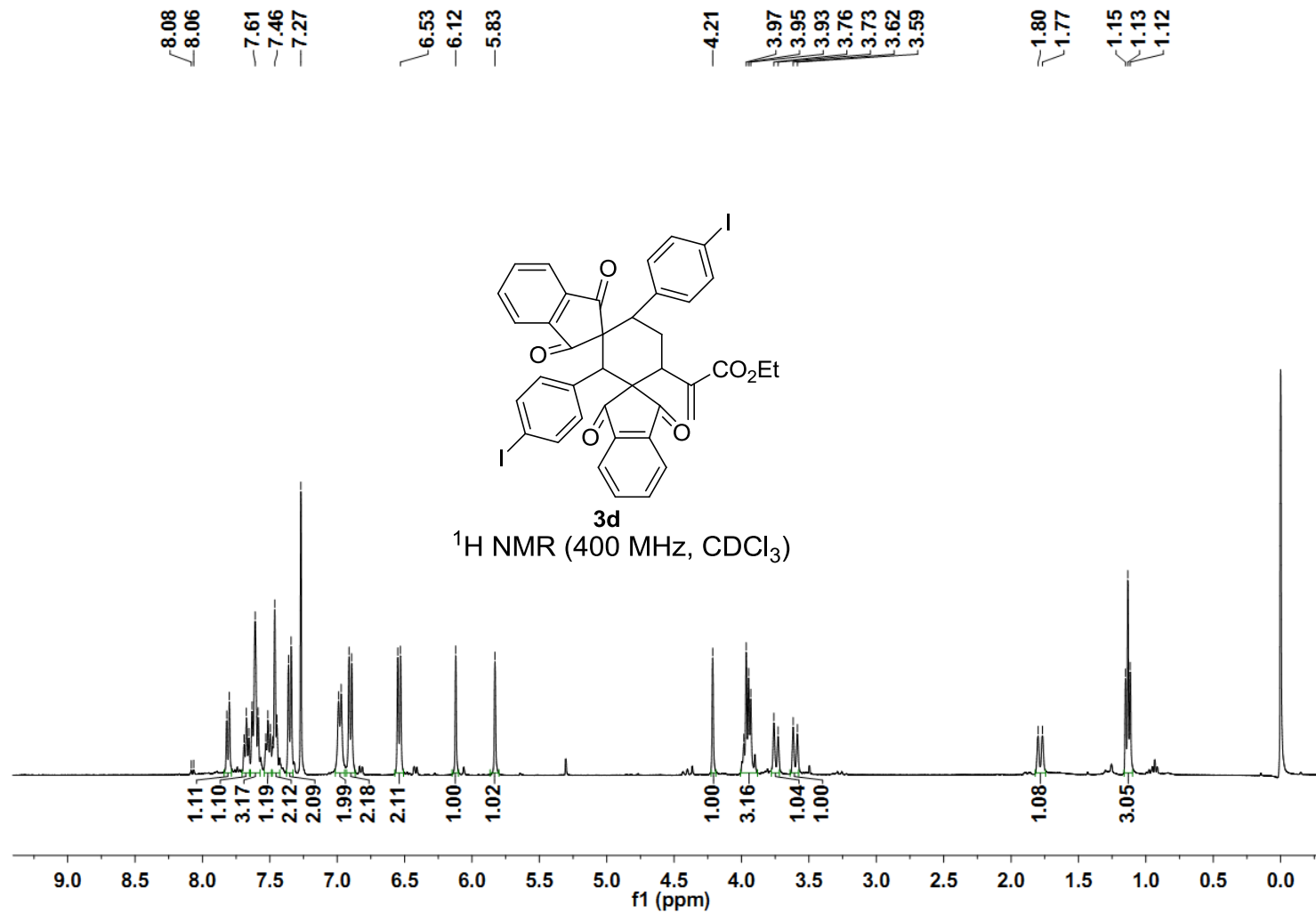
¹³C NMR (100 MHz, CDCl₃)

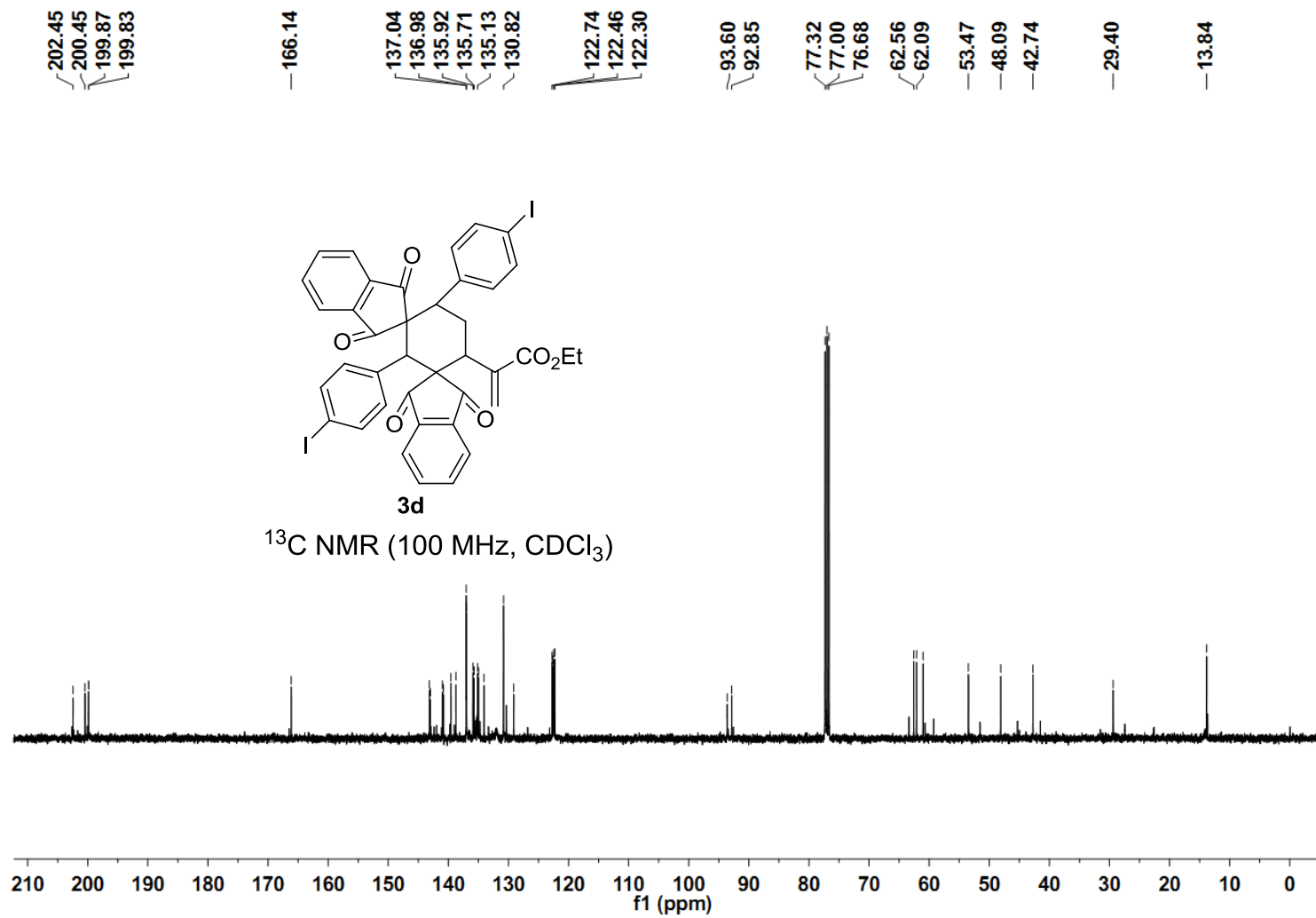


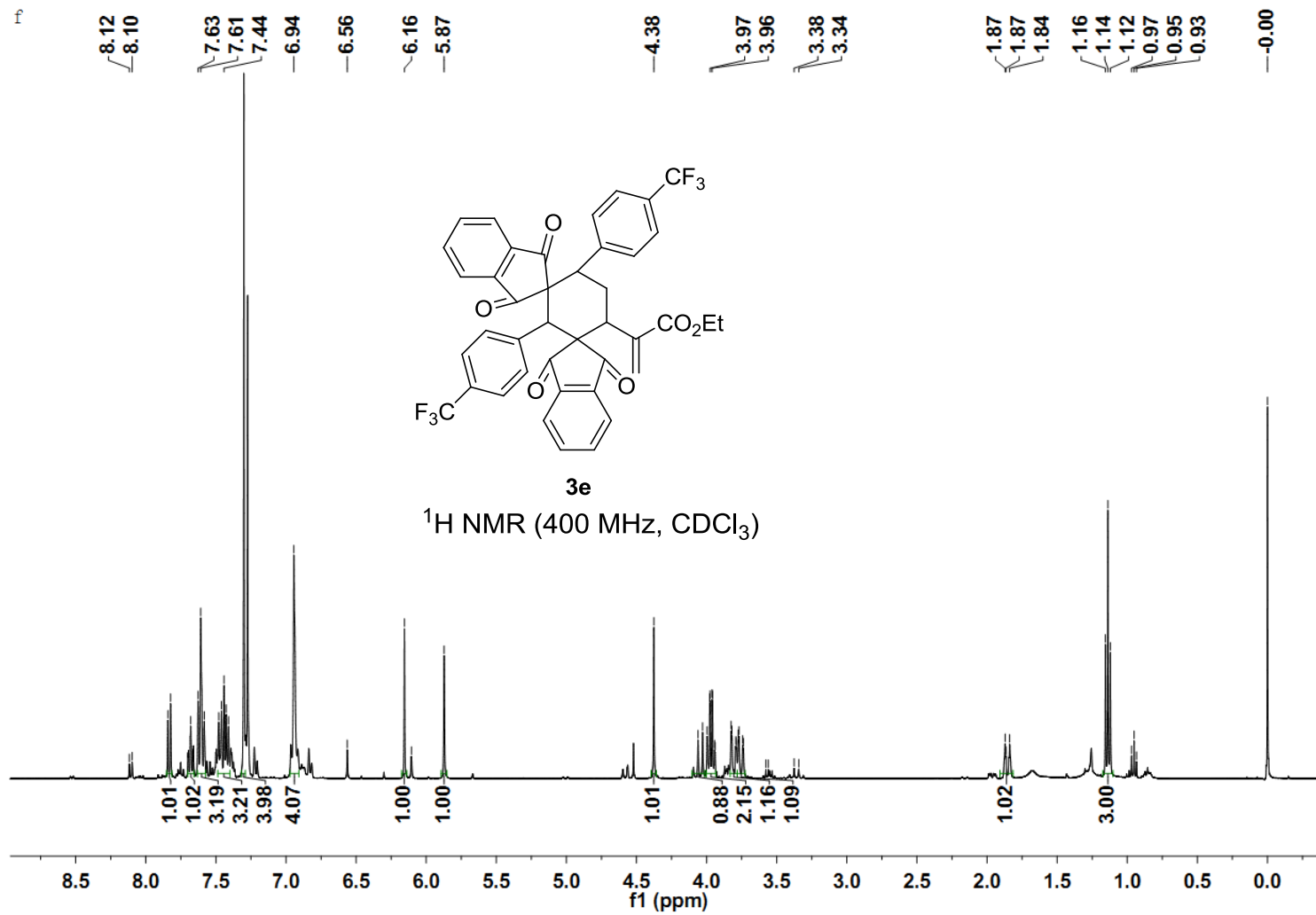


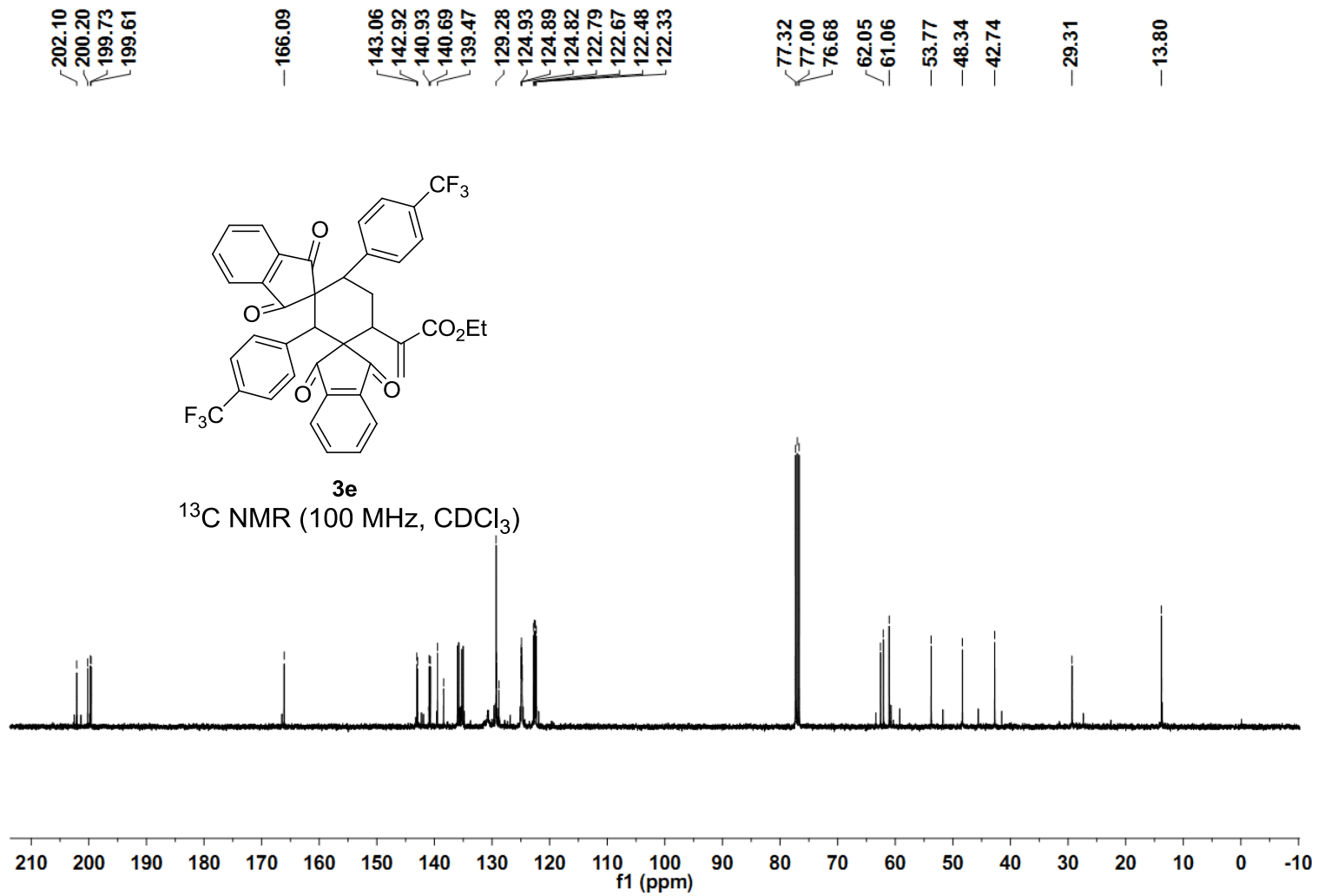


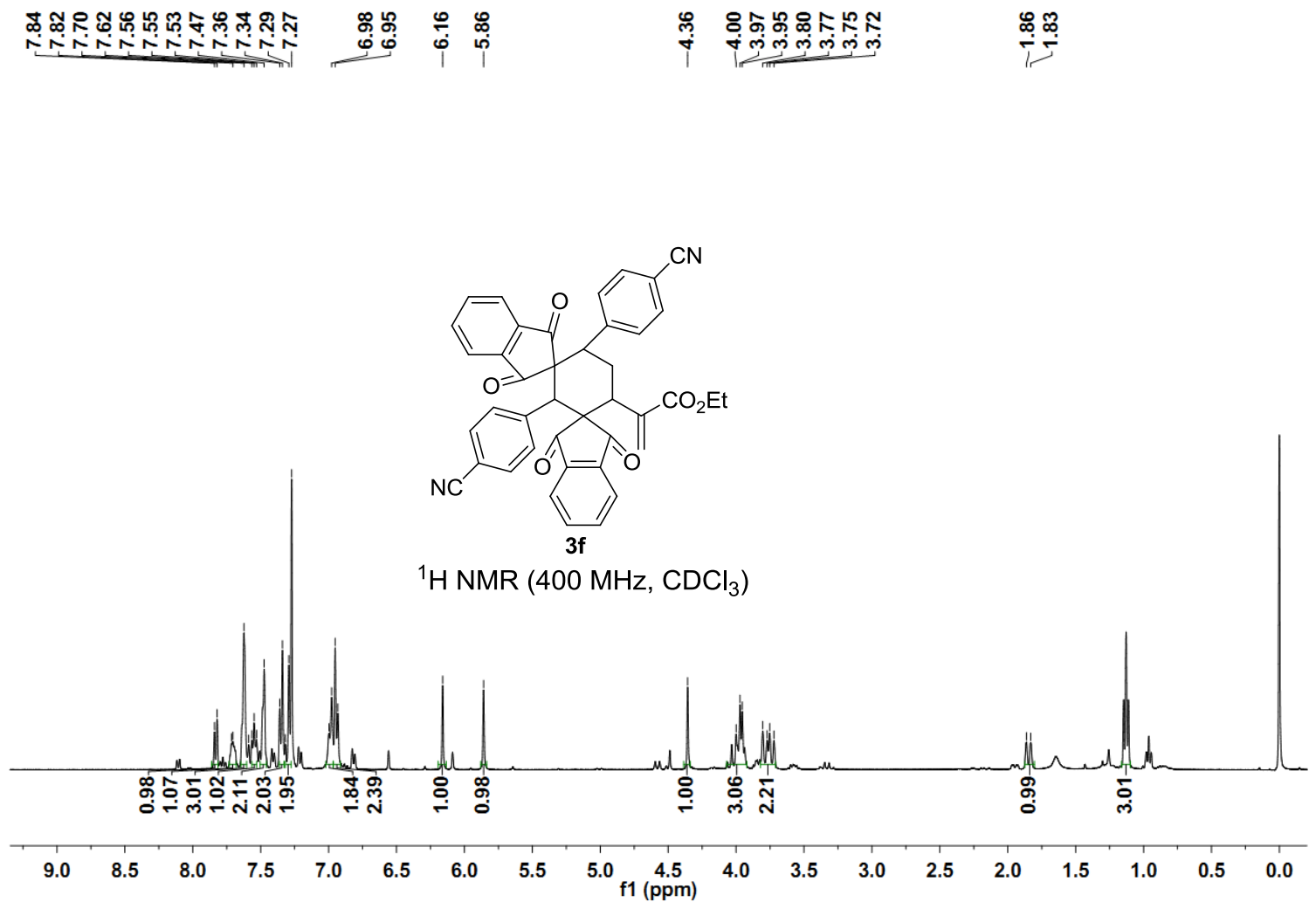


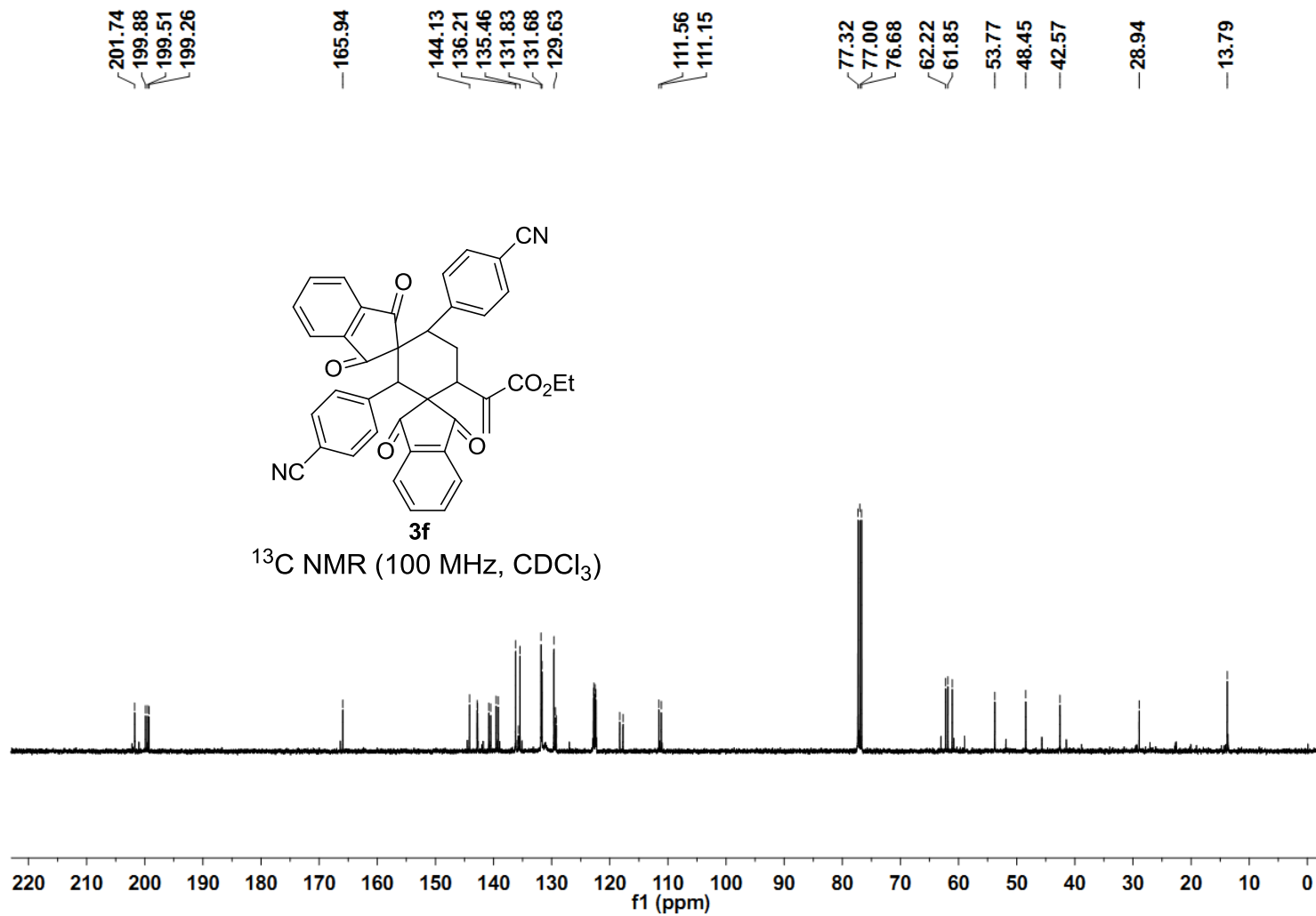


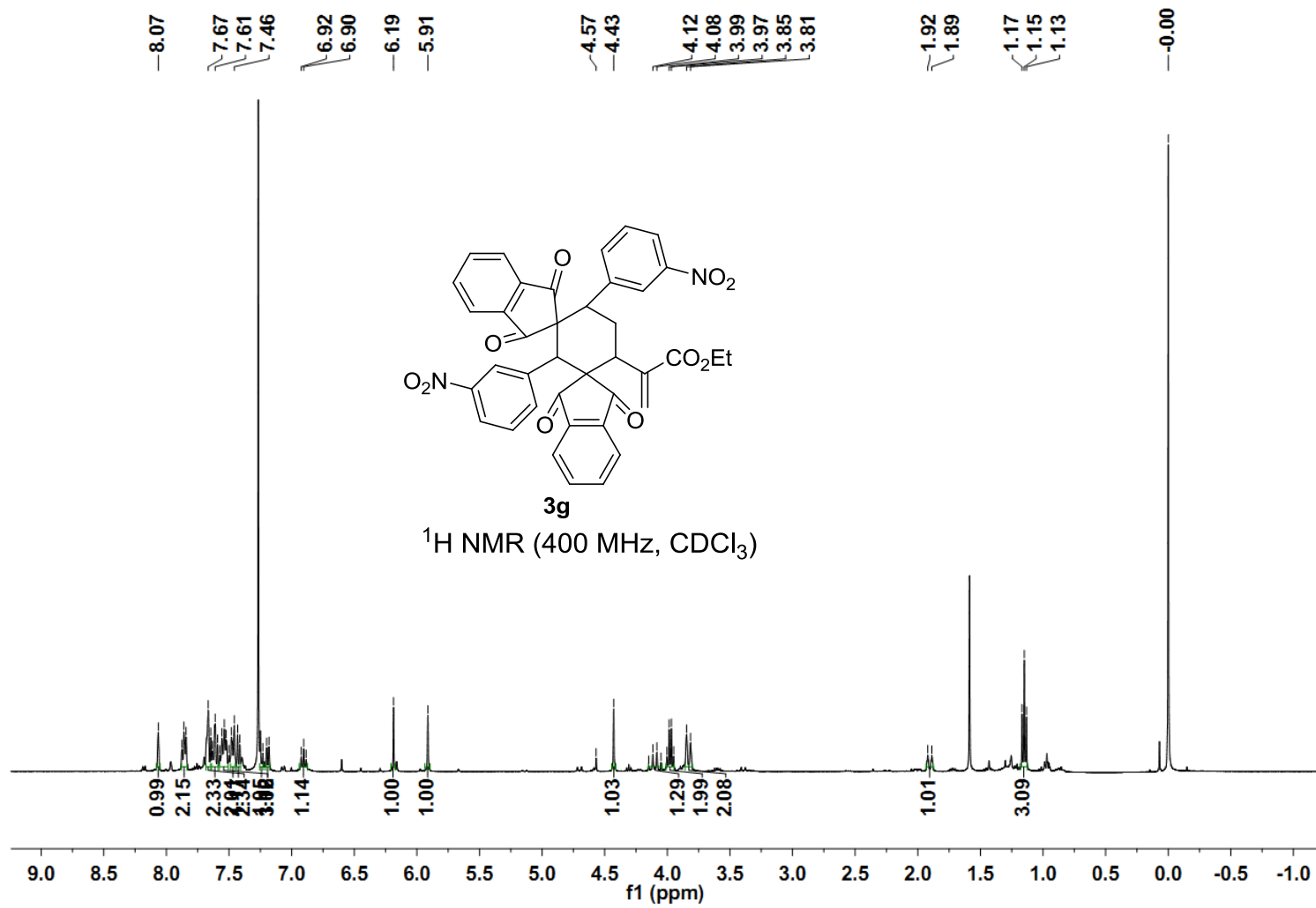












201.61
199.82
199.37
199.02

165.97

136.23
136.12
135.43
135.34
135.17

122.95
122.76
122.69
122.63
122.48

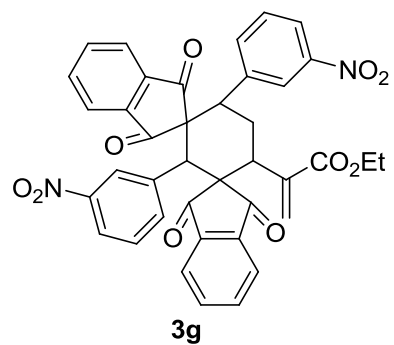
77.32
77.00
76.68

62.30

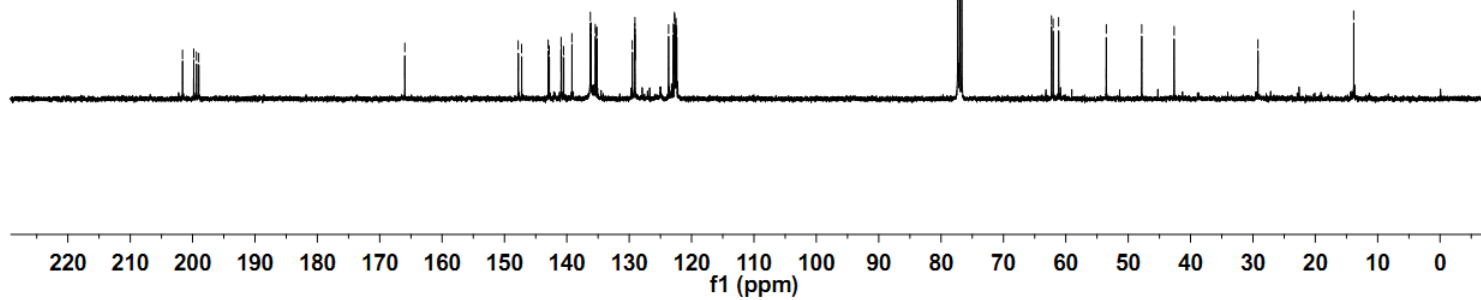
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47.84
42.64

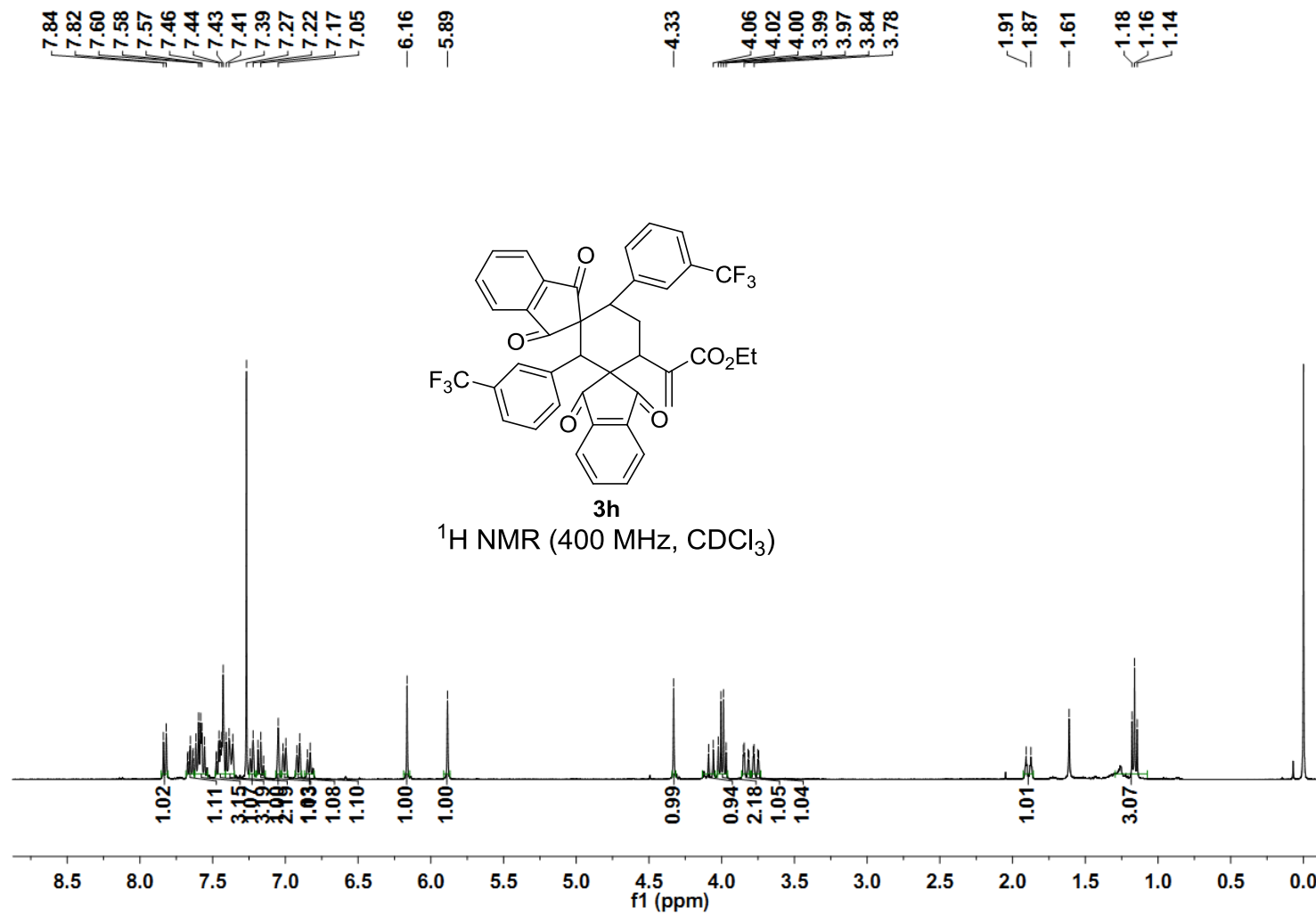
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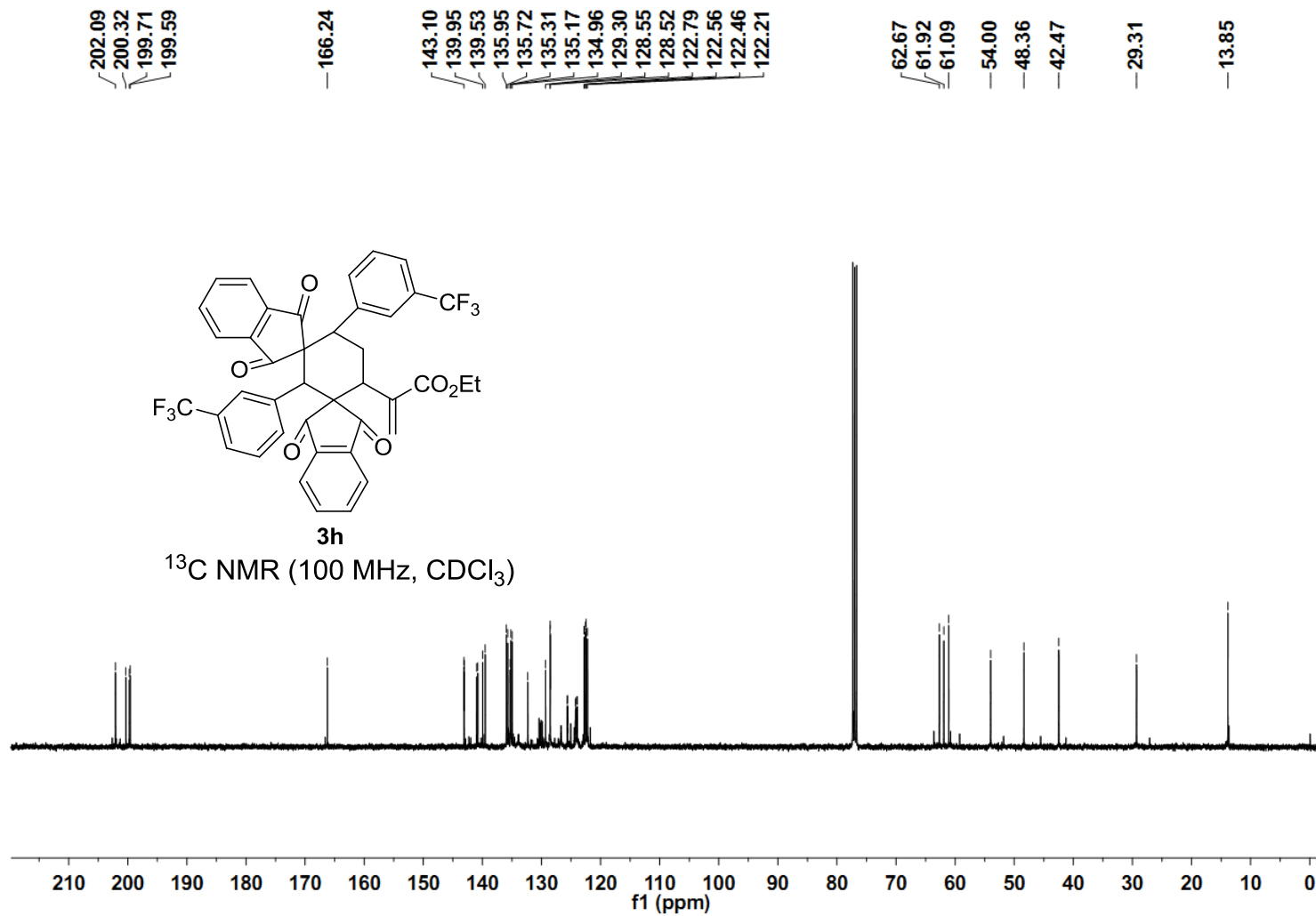
13.84

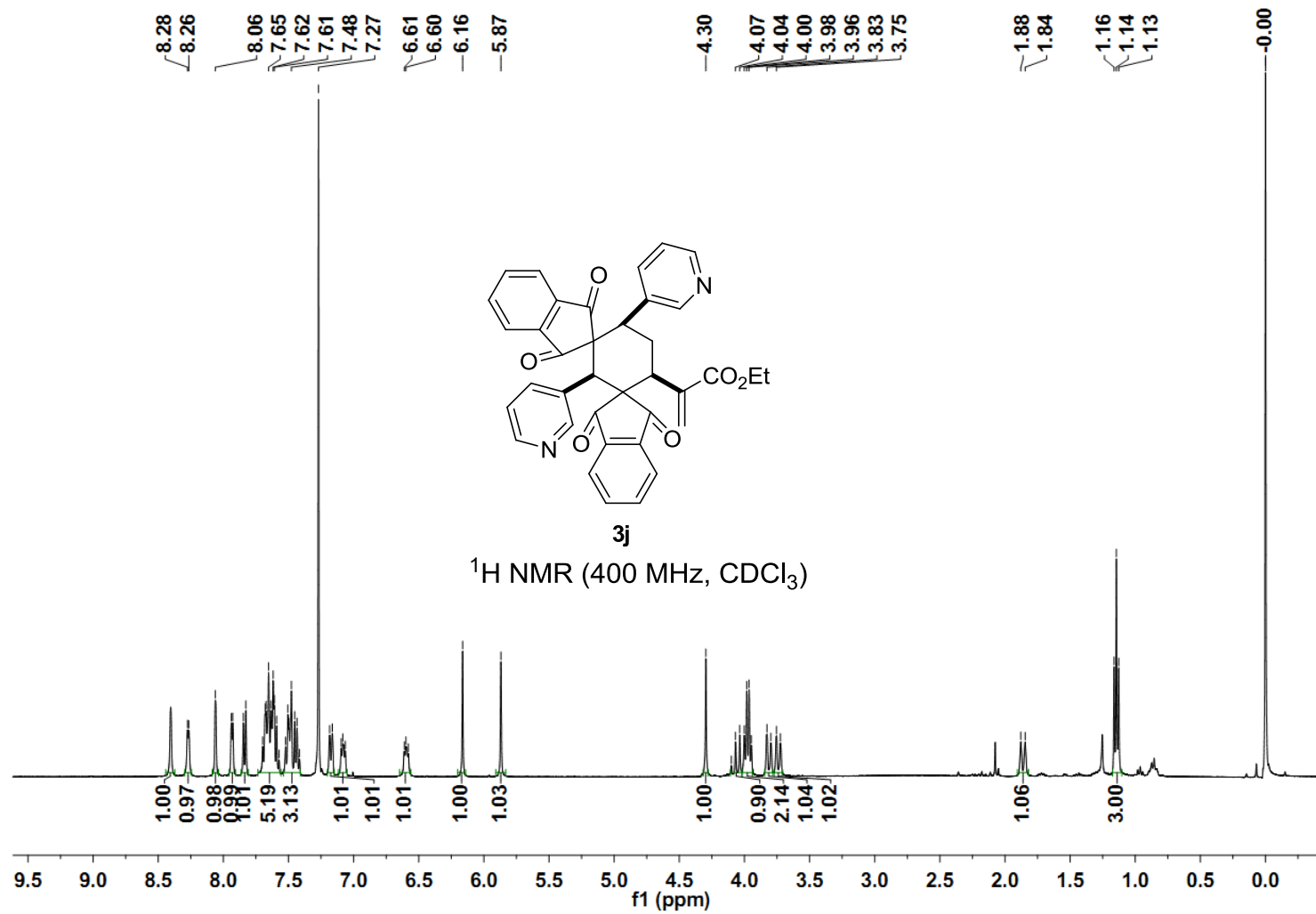


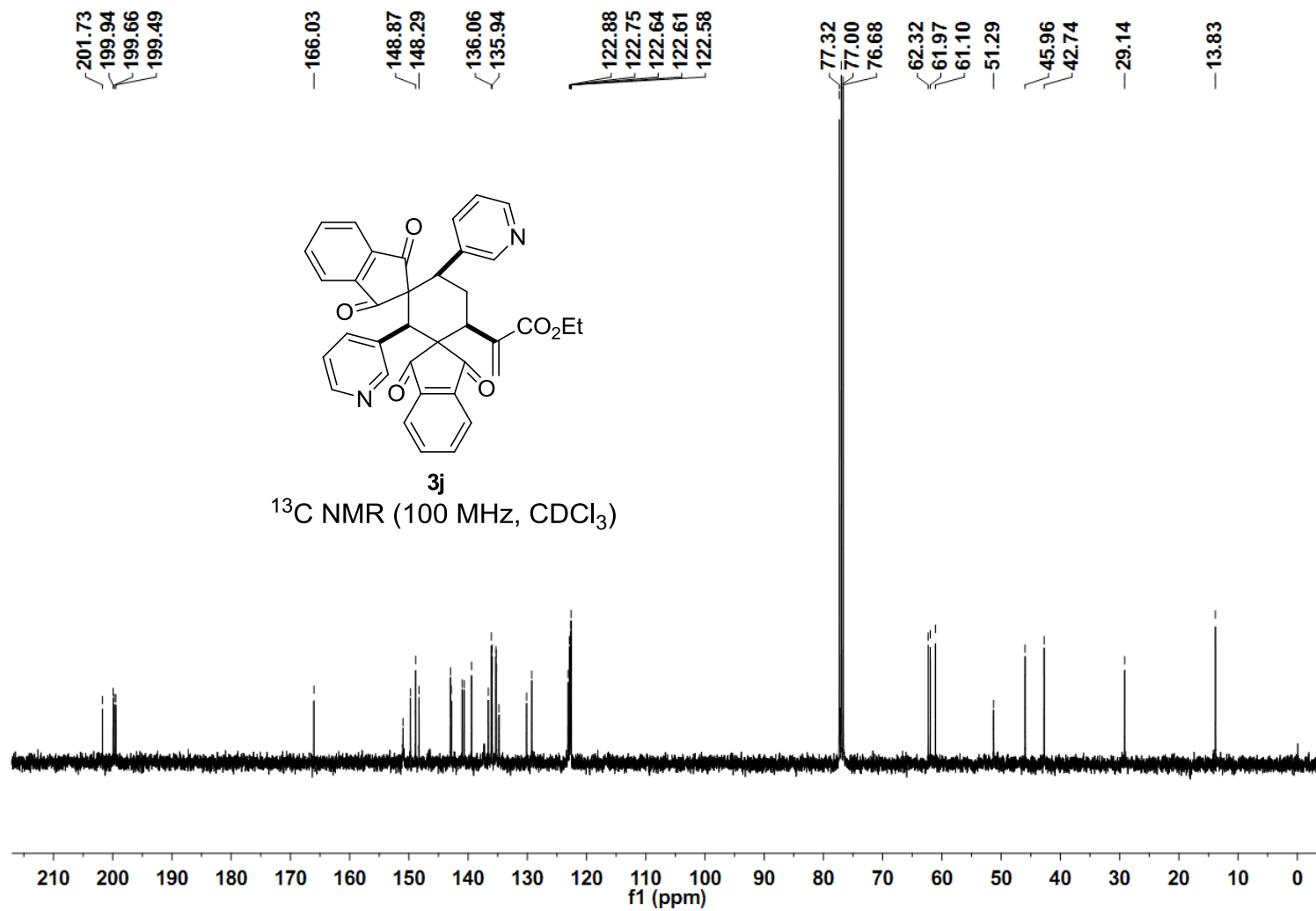
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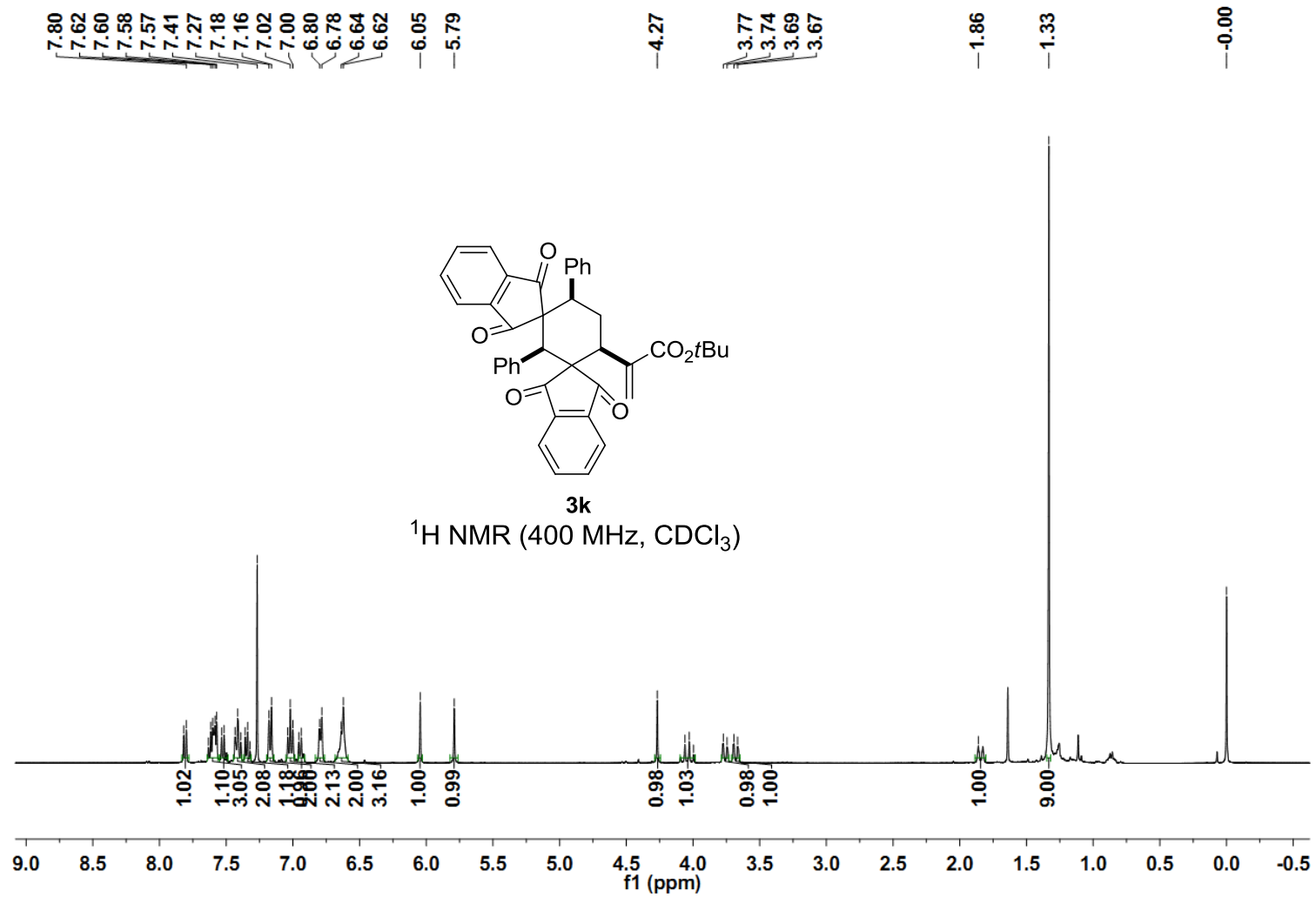












202.97
200.94
200.26
200.17

165.56

135.49
135.10
134.61
134.33

122.62
122.26
122.06

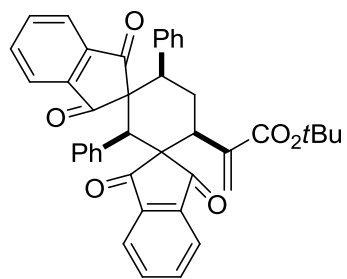
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63.17
62.29

54.49
48.96

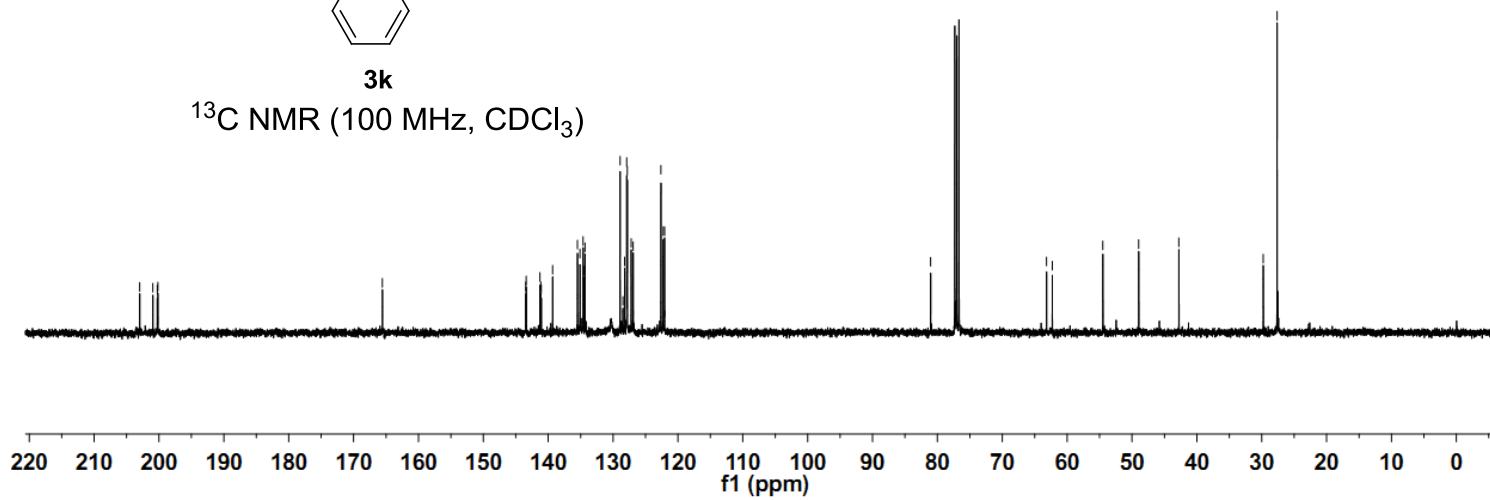
42.77

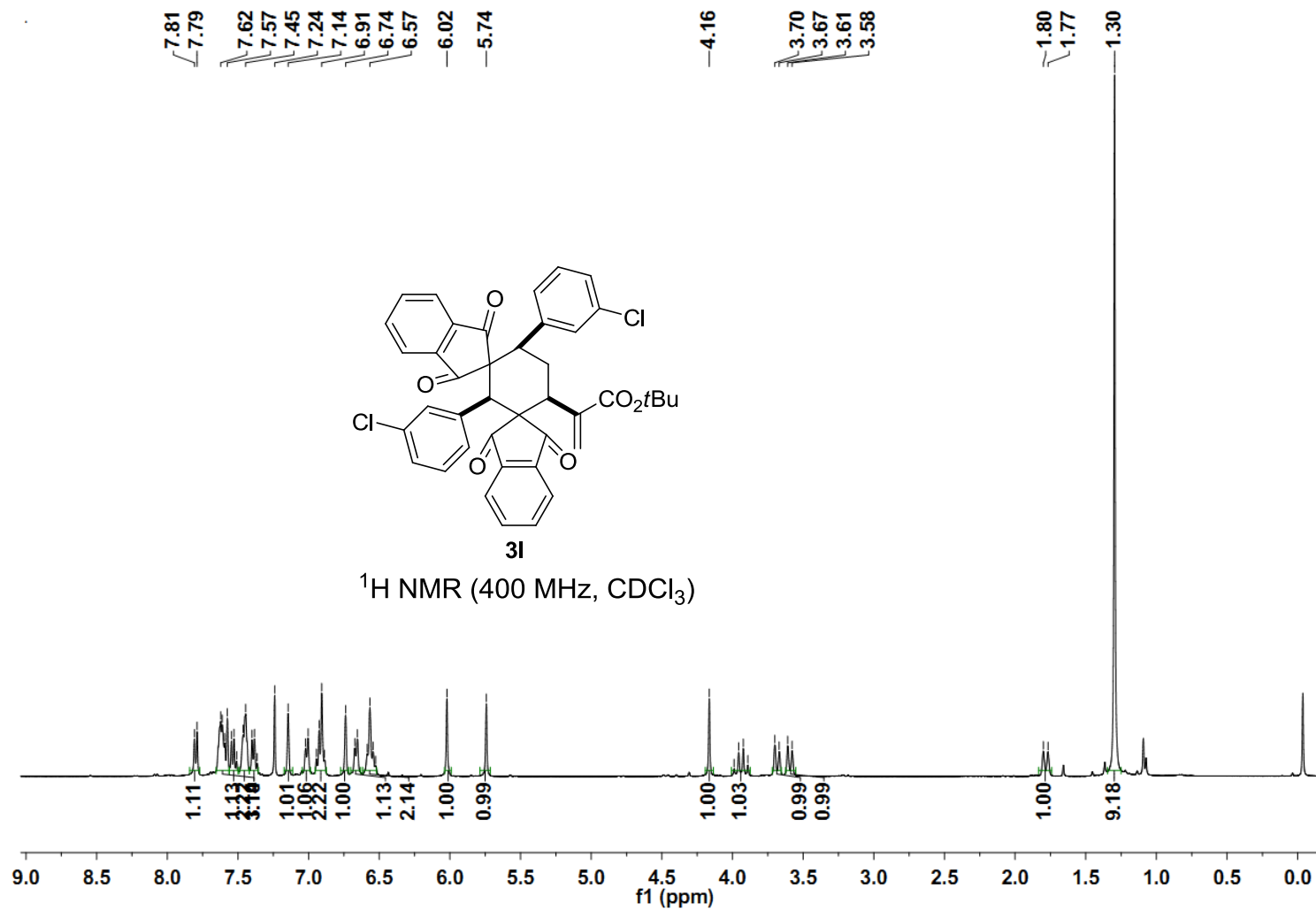
29.76
27.63



3k

^{13}C NMR (100 MHz, CDCl_3)





202.24
200.38
199.77
199.58

165.36

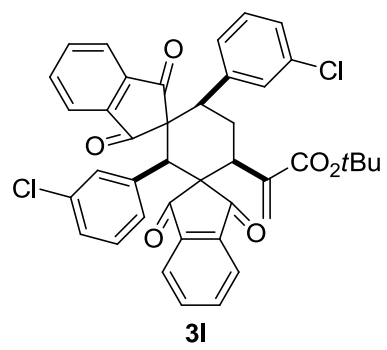
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140.91
140.86

122.81
122.72
122.45
122.31

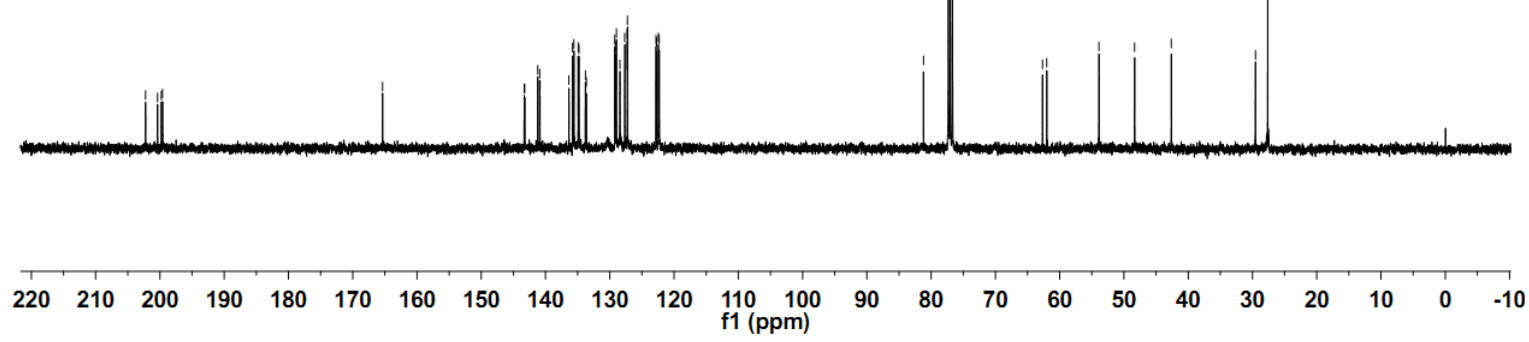
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77.00
76.68

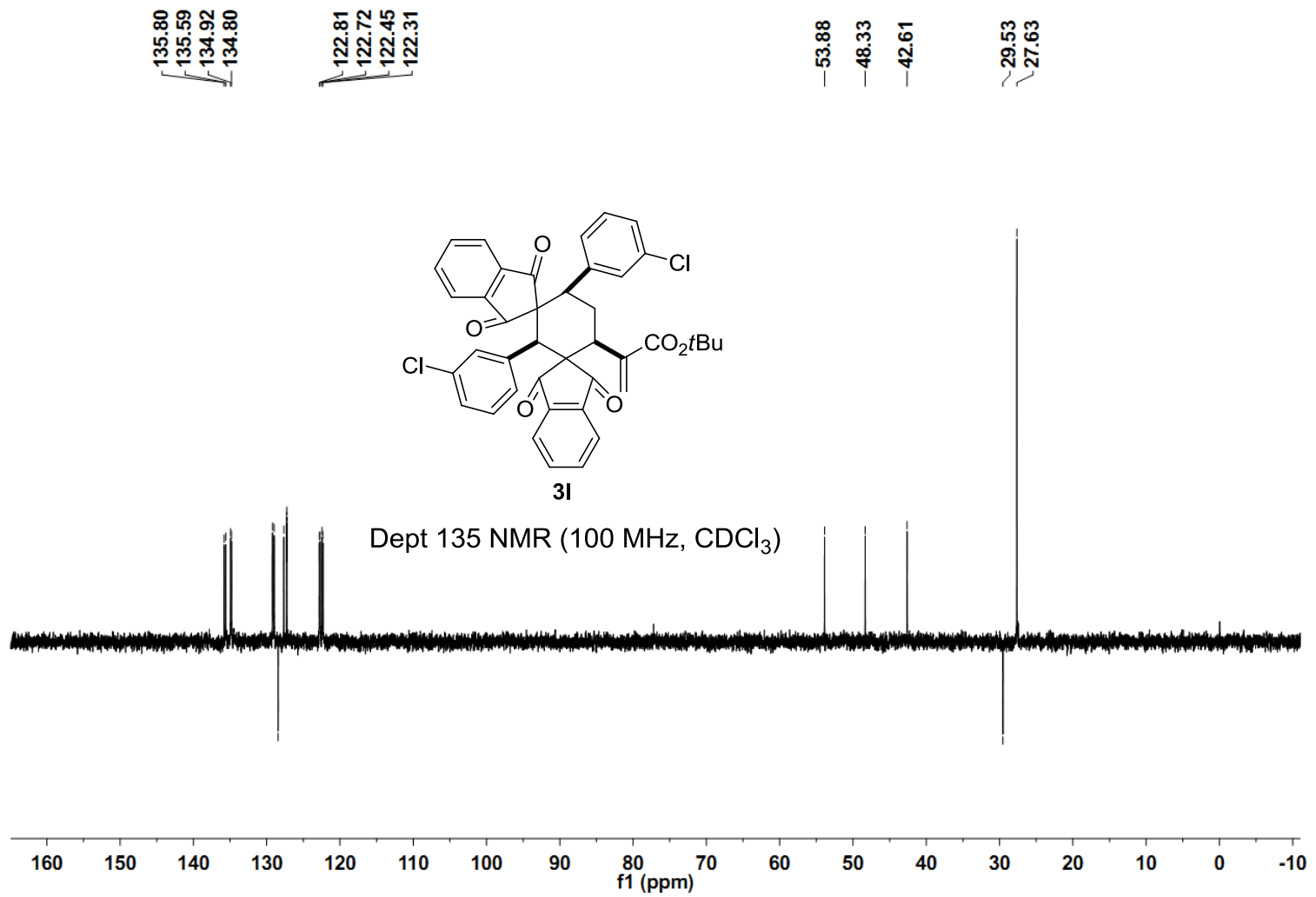
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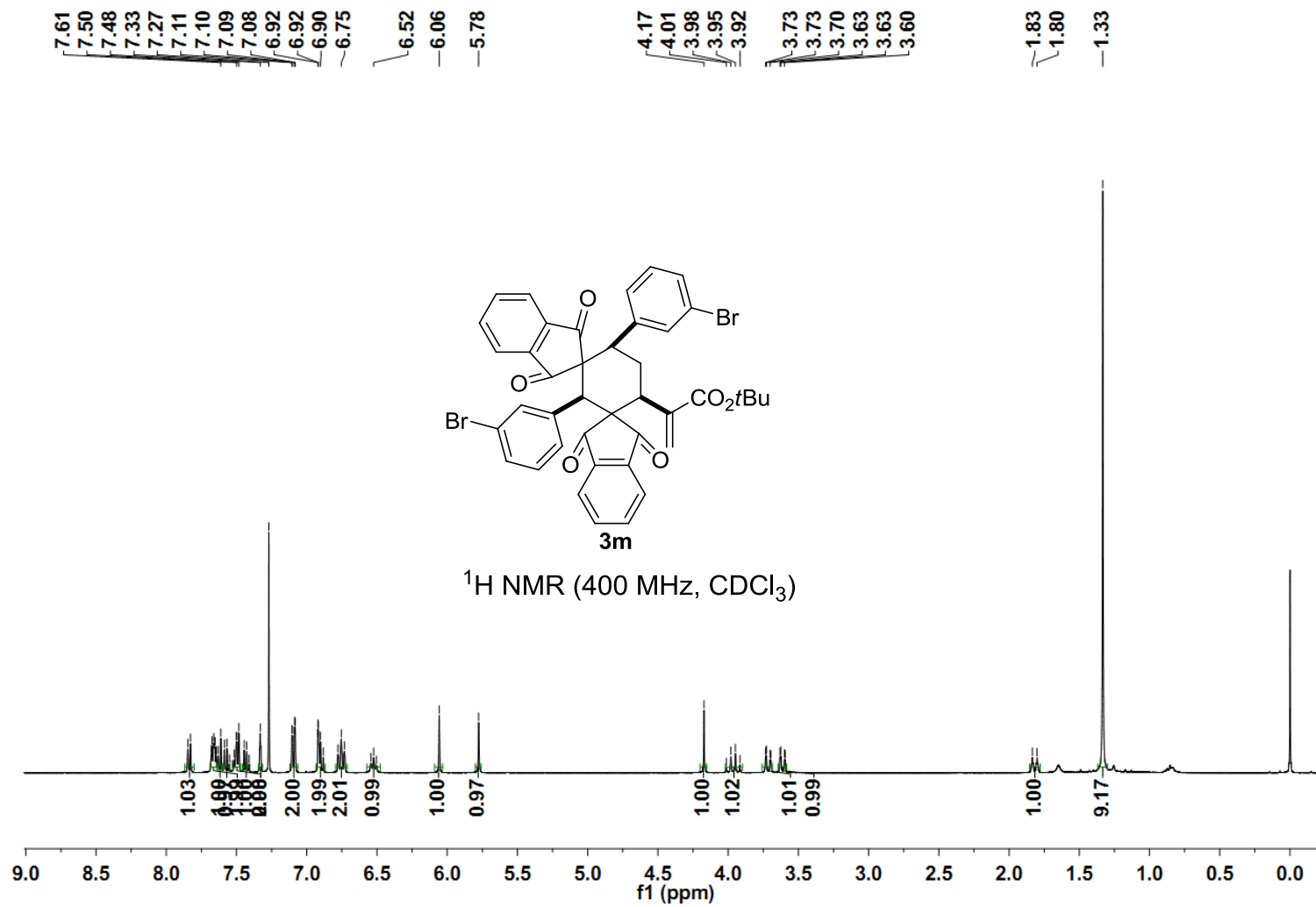
29.54
27.63

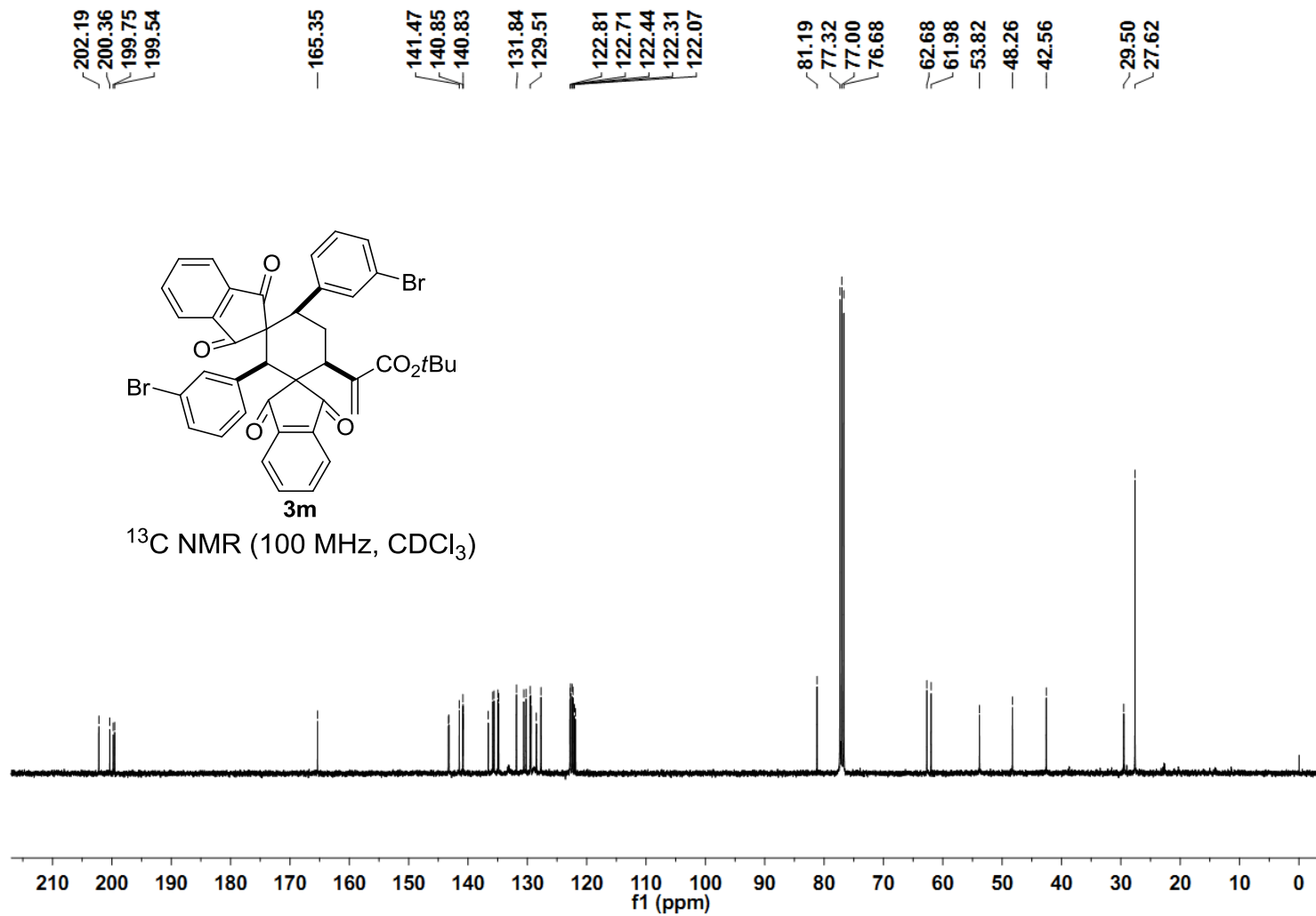


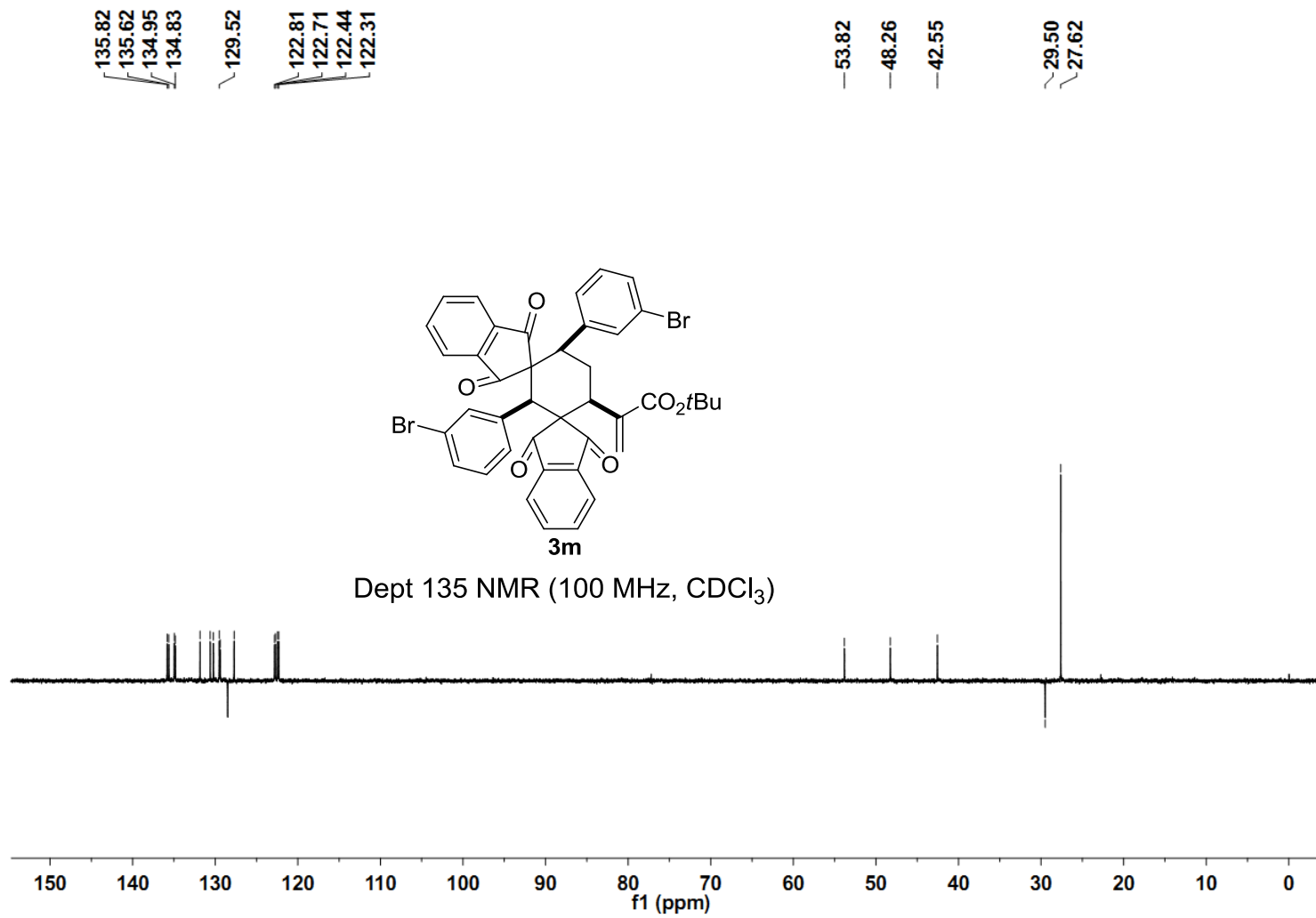
¹³C NMR (100 MHz, CDCl₃)

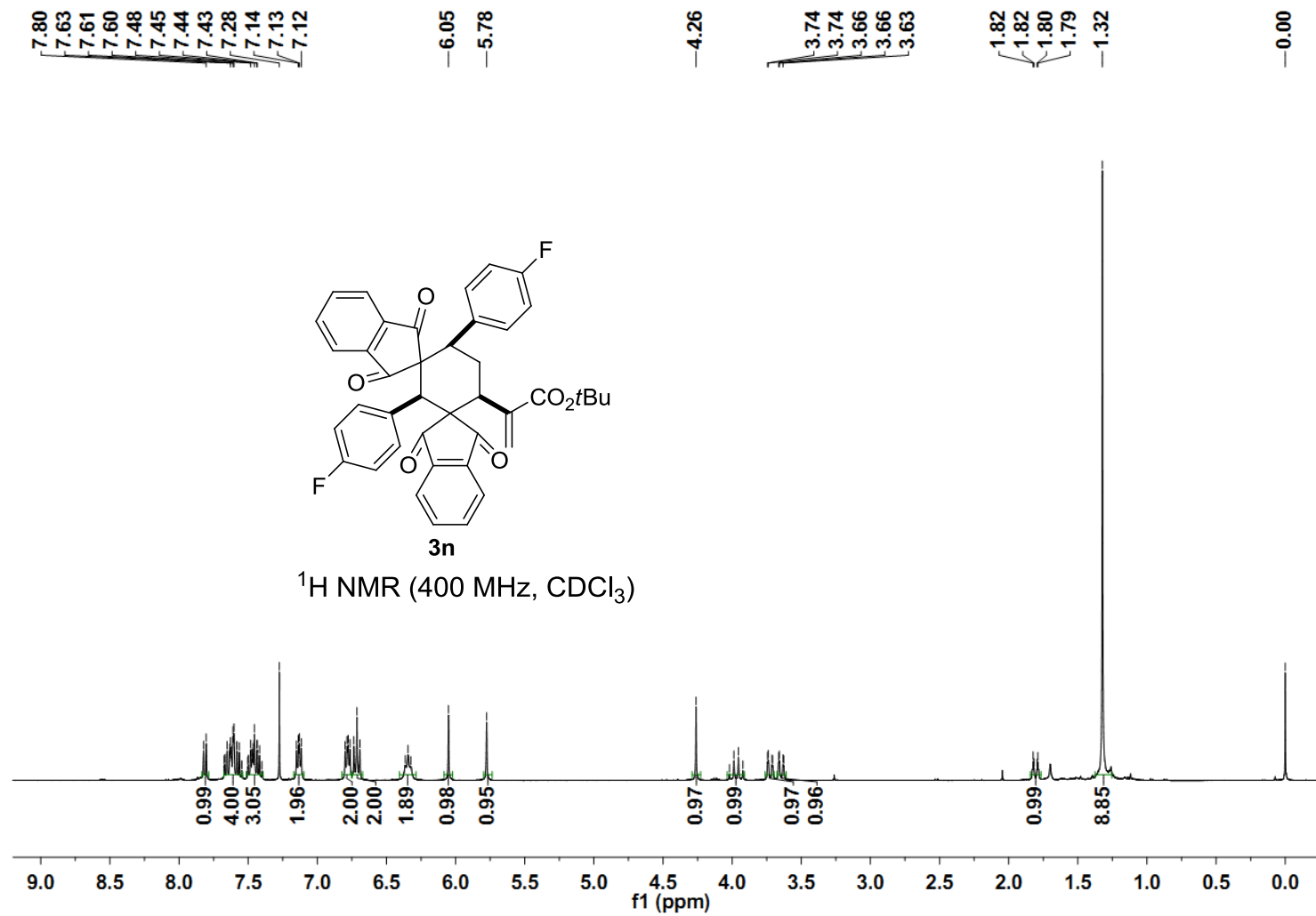


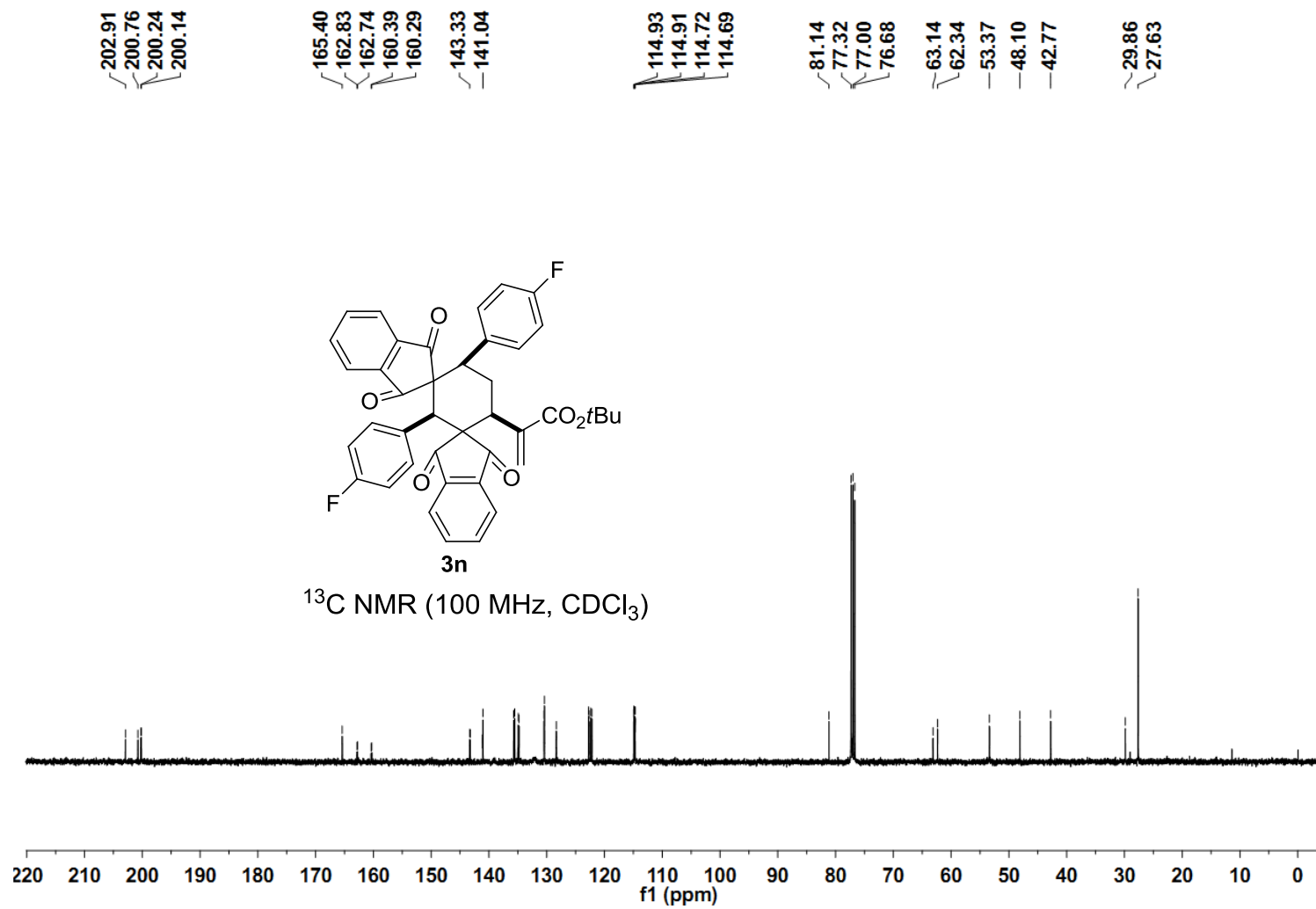


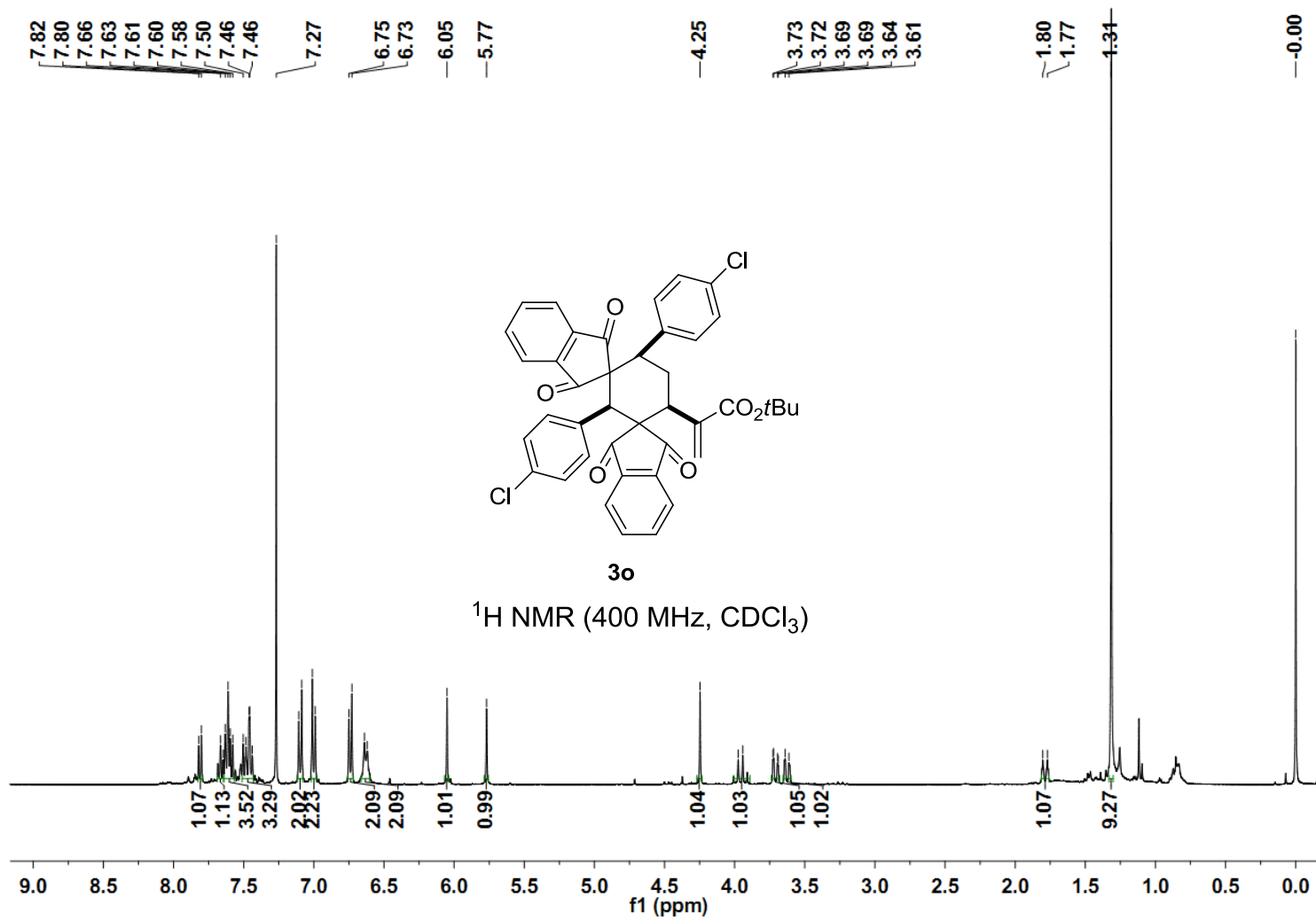


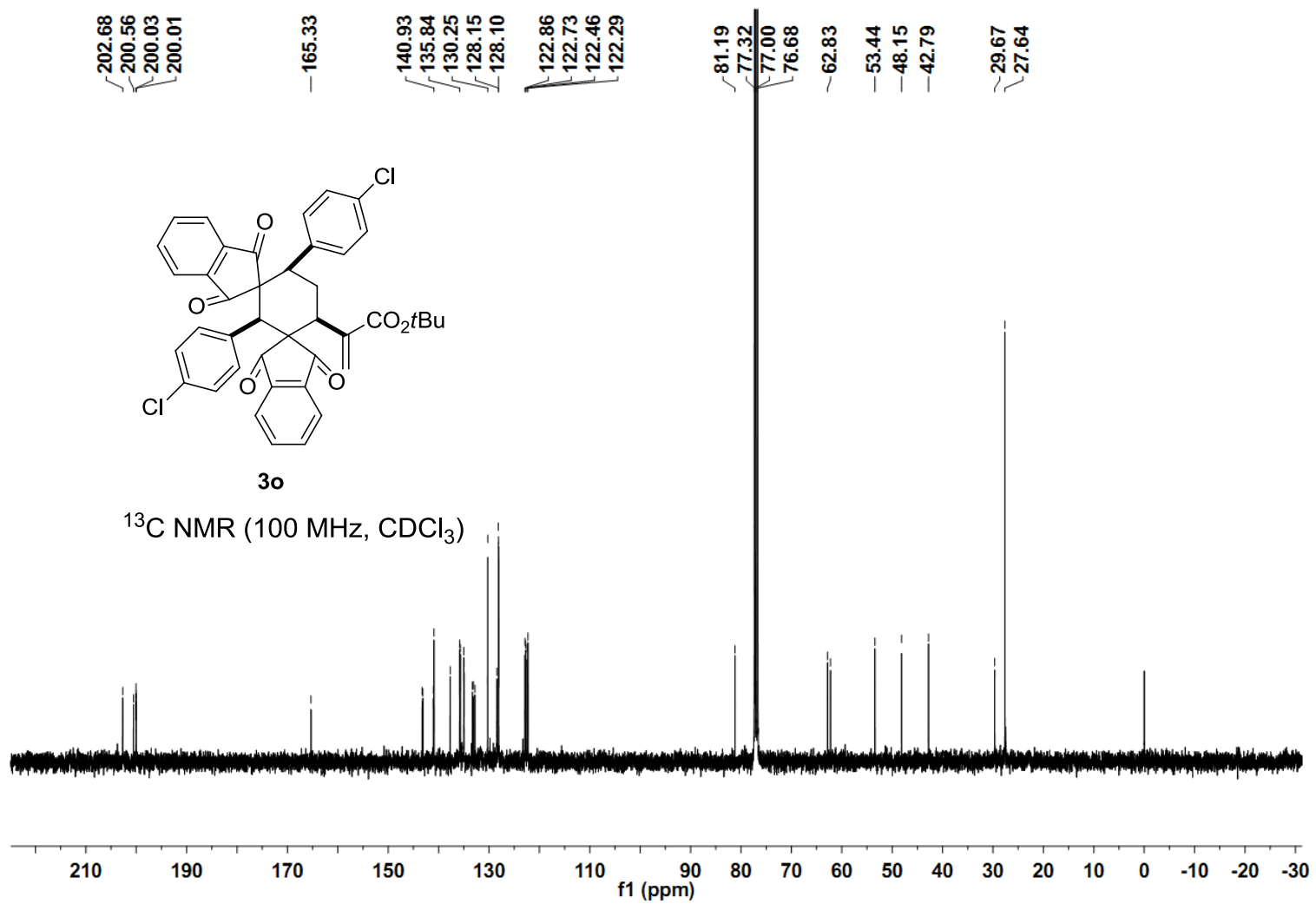


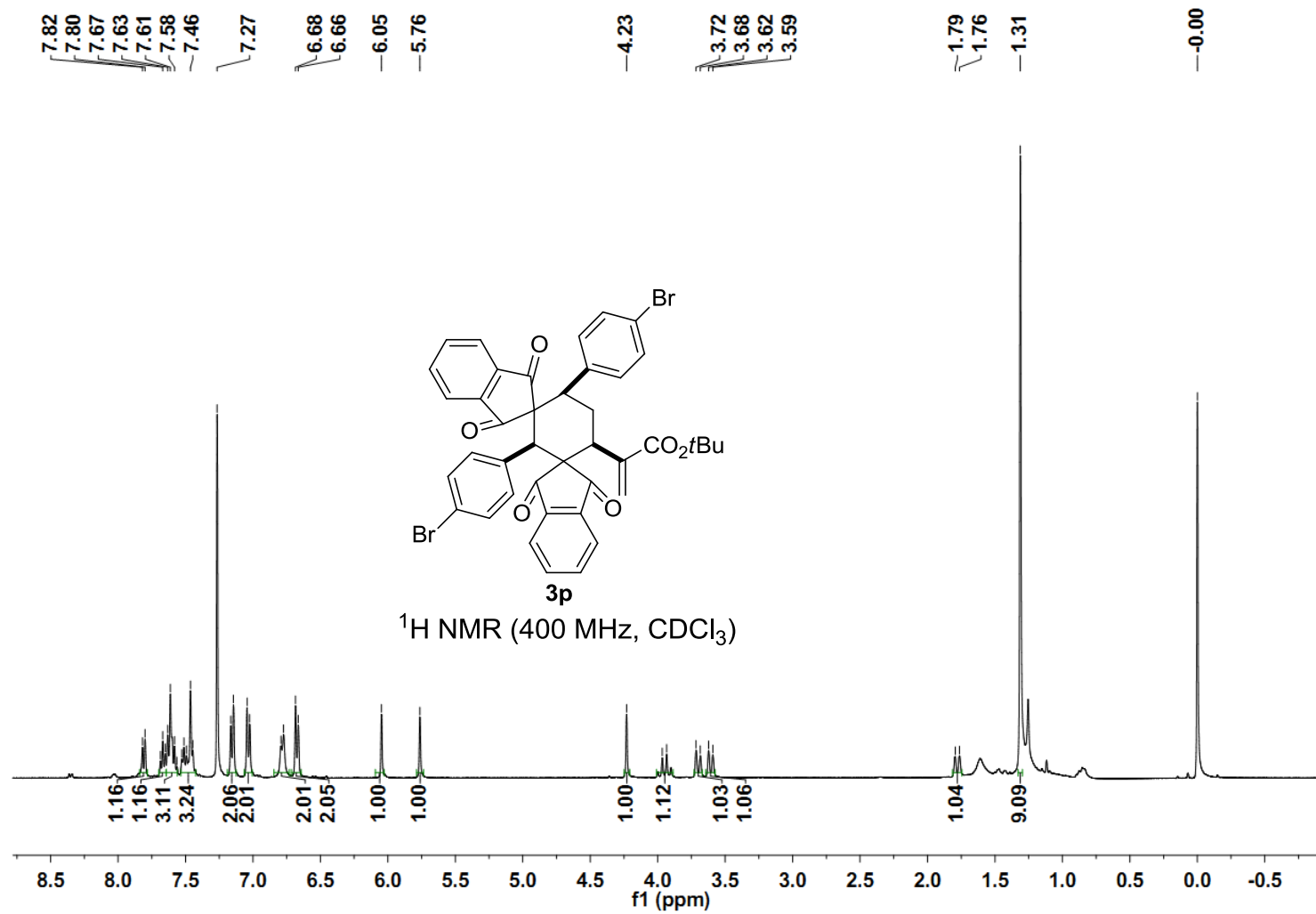


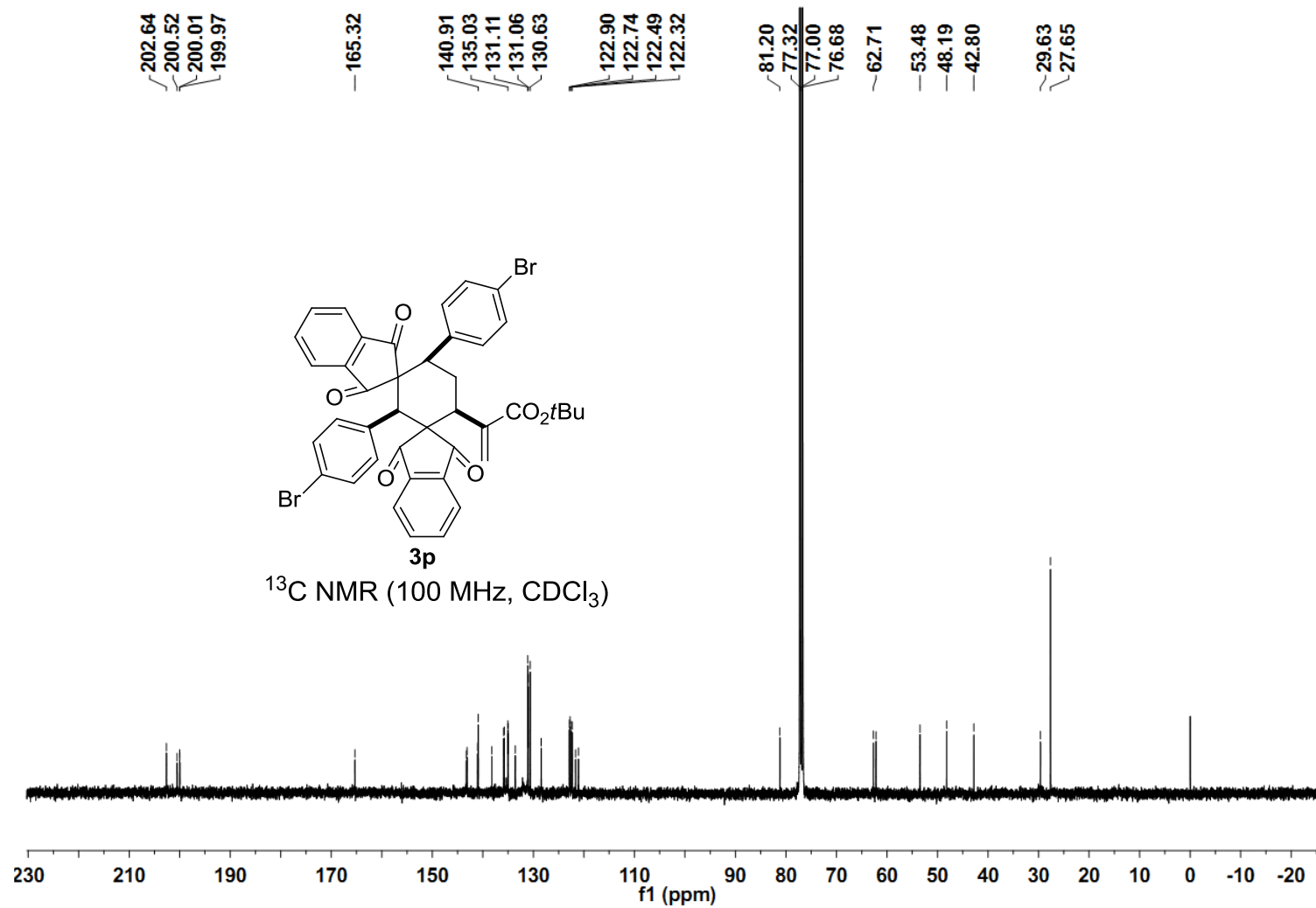


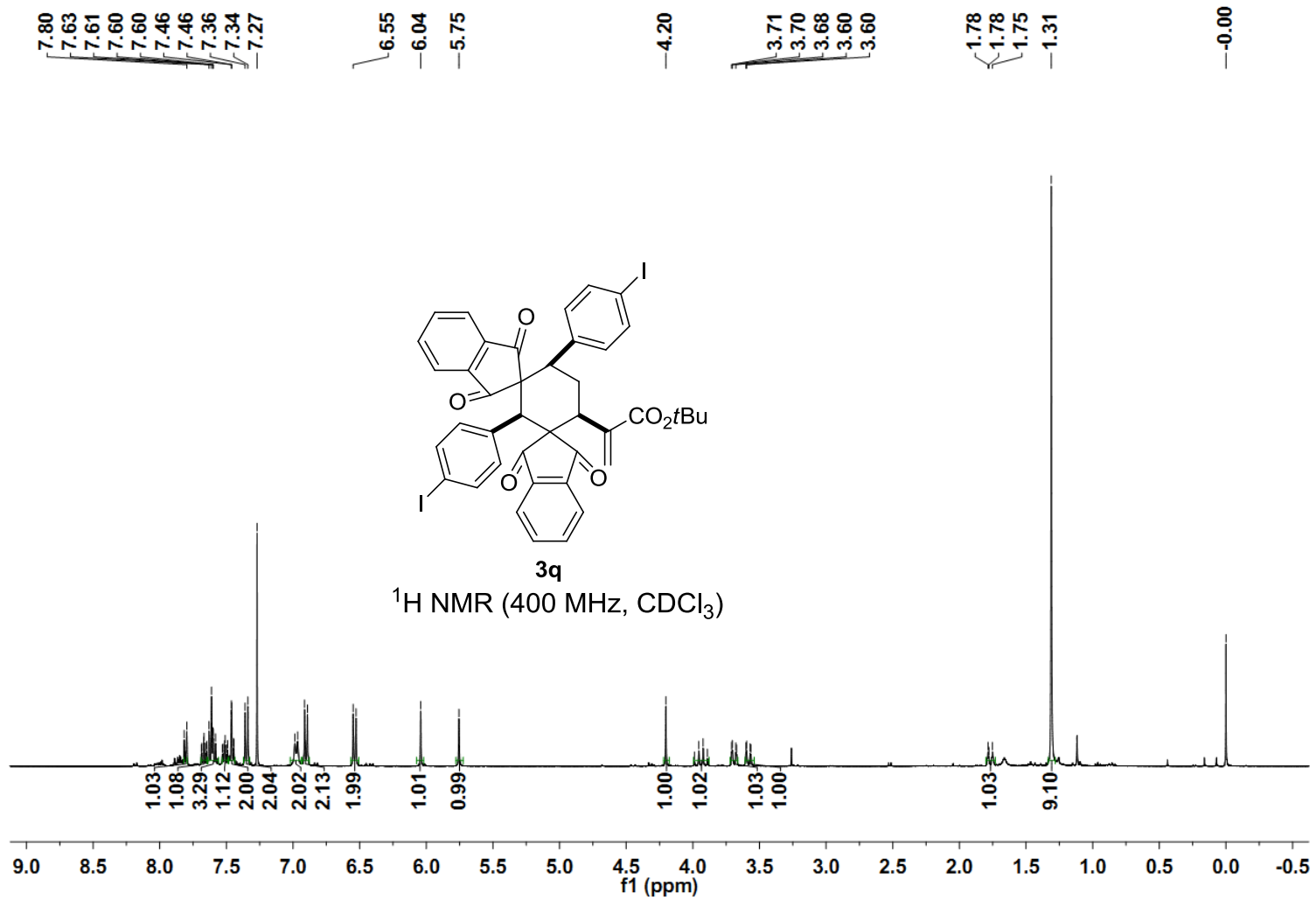


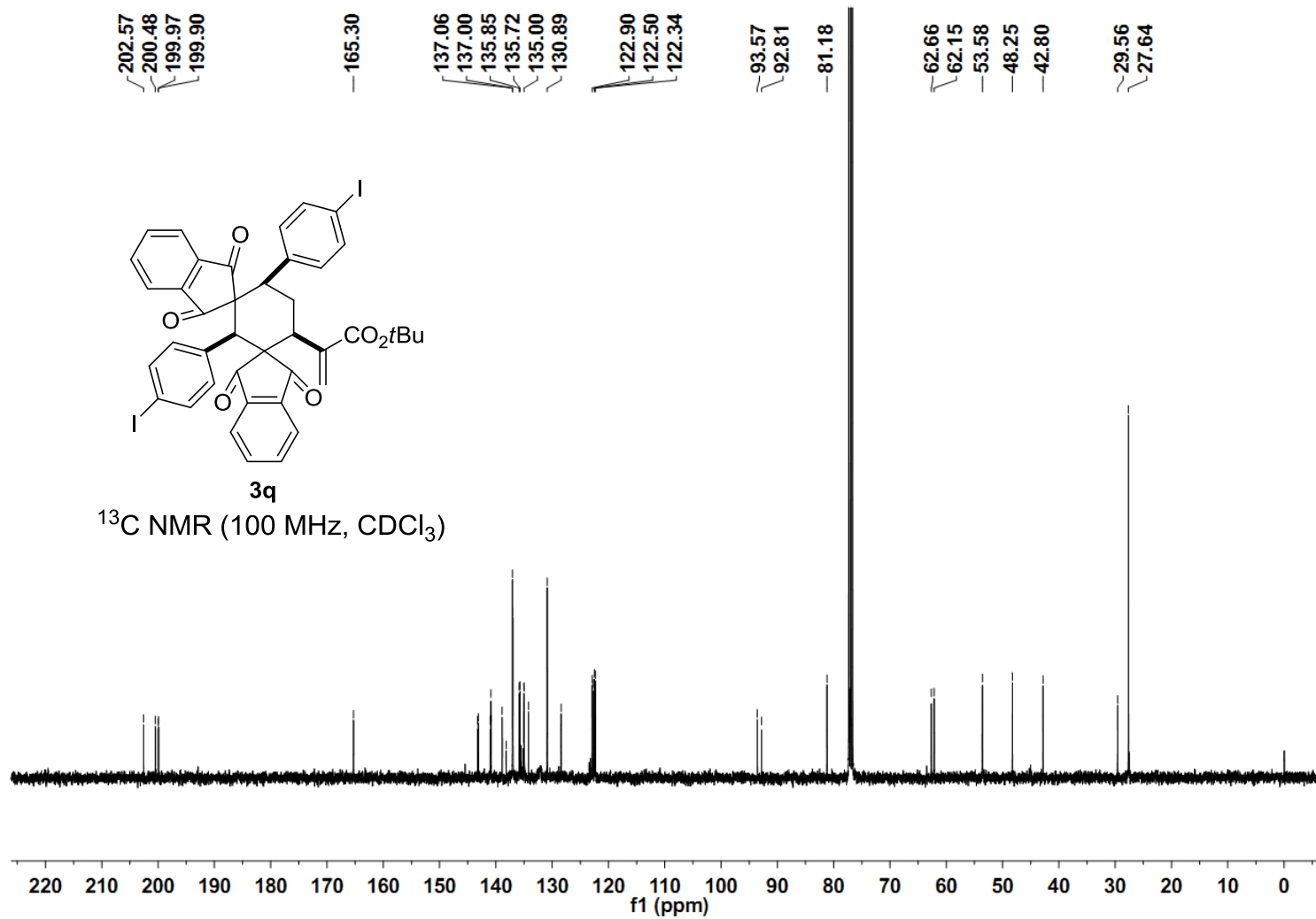


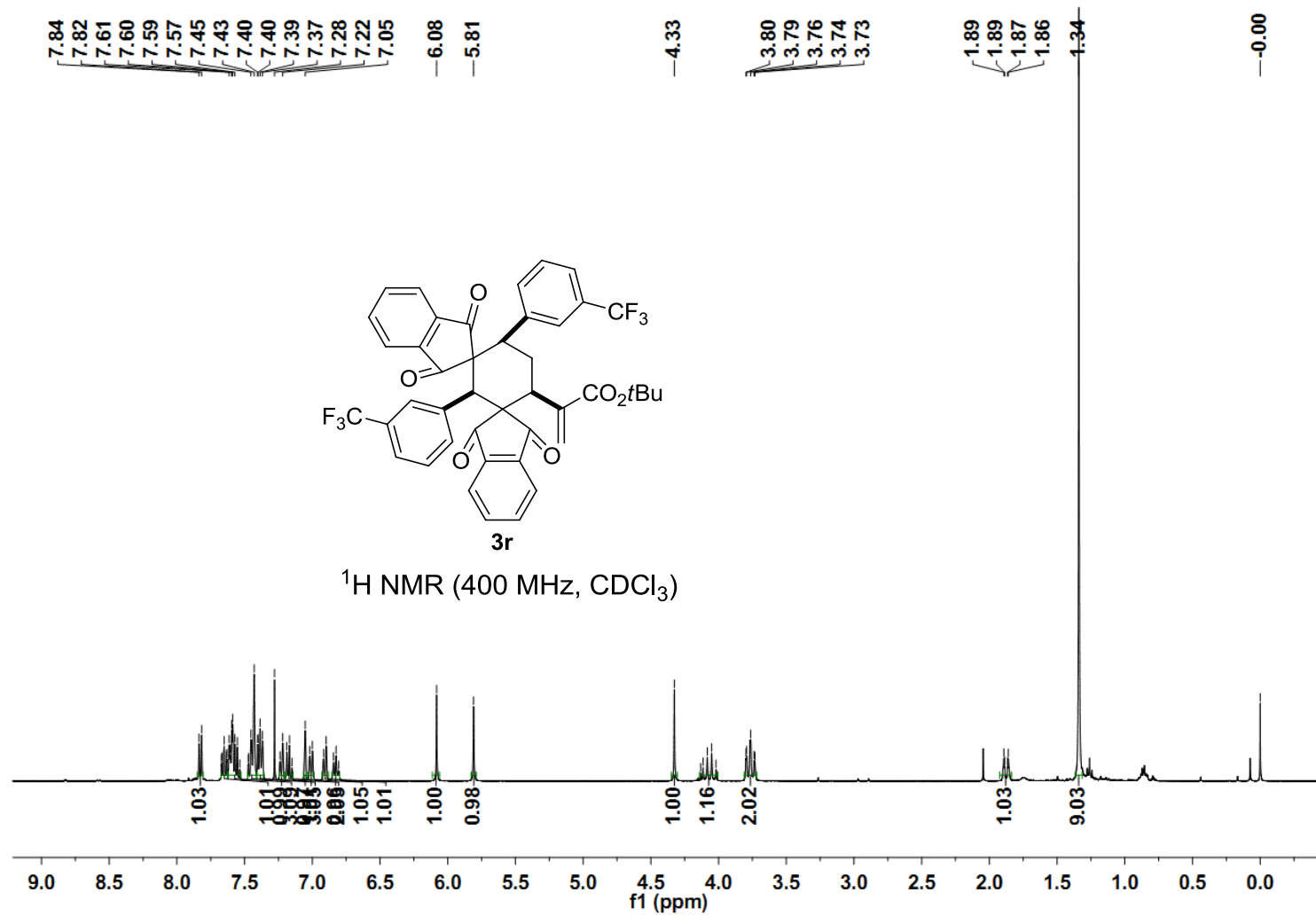


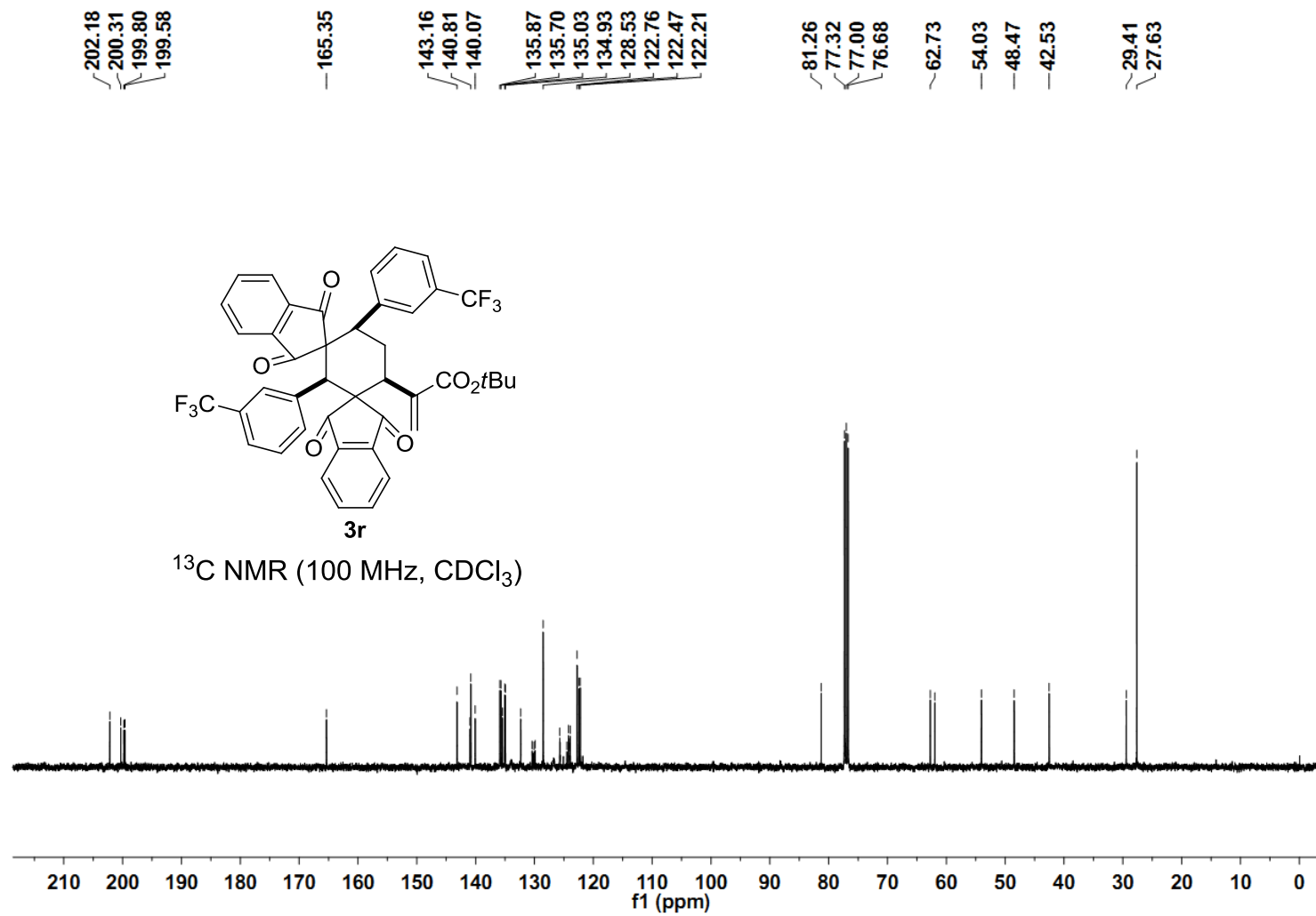


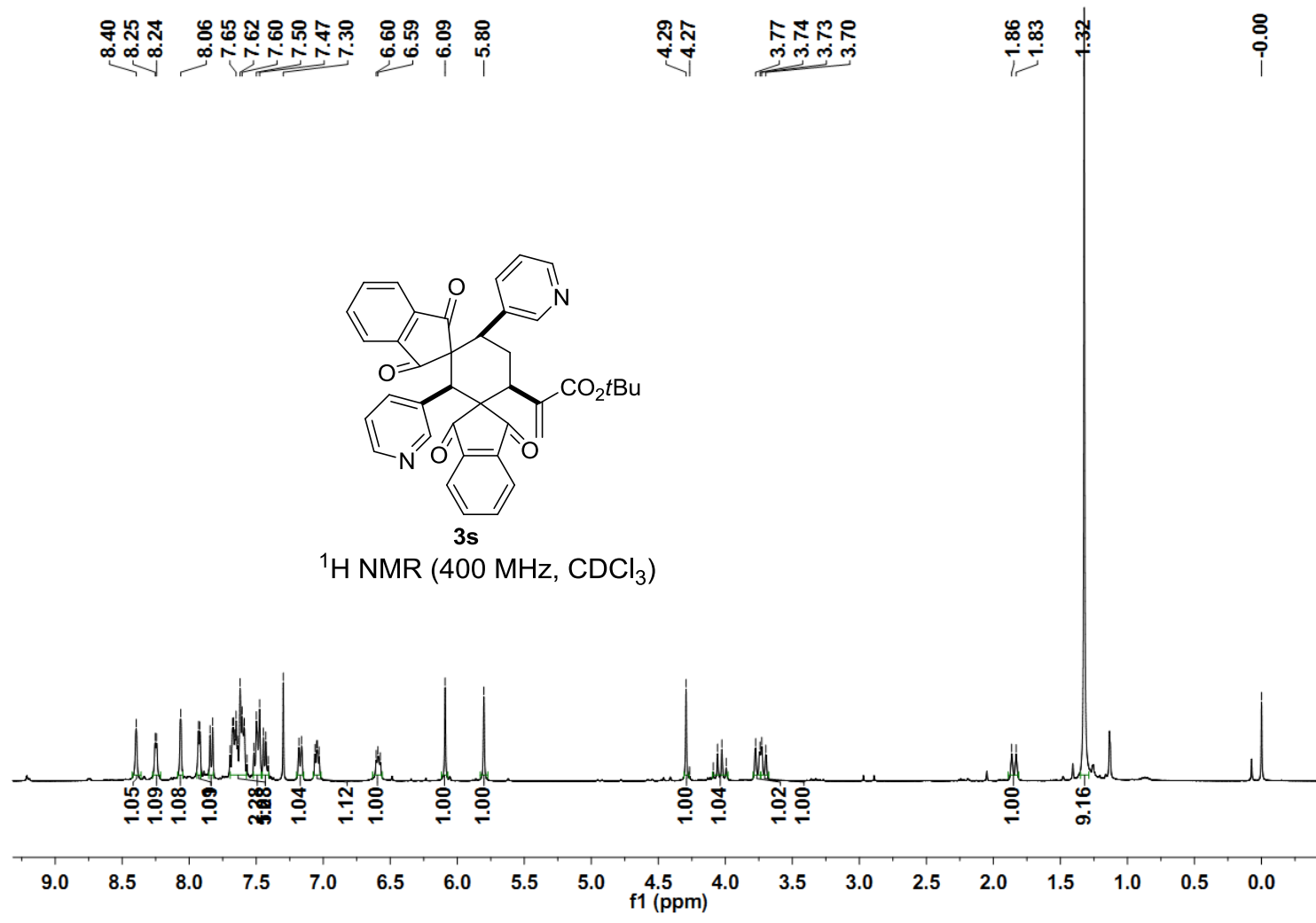


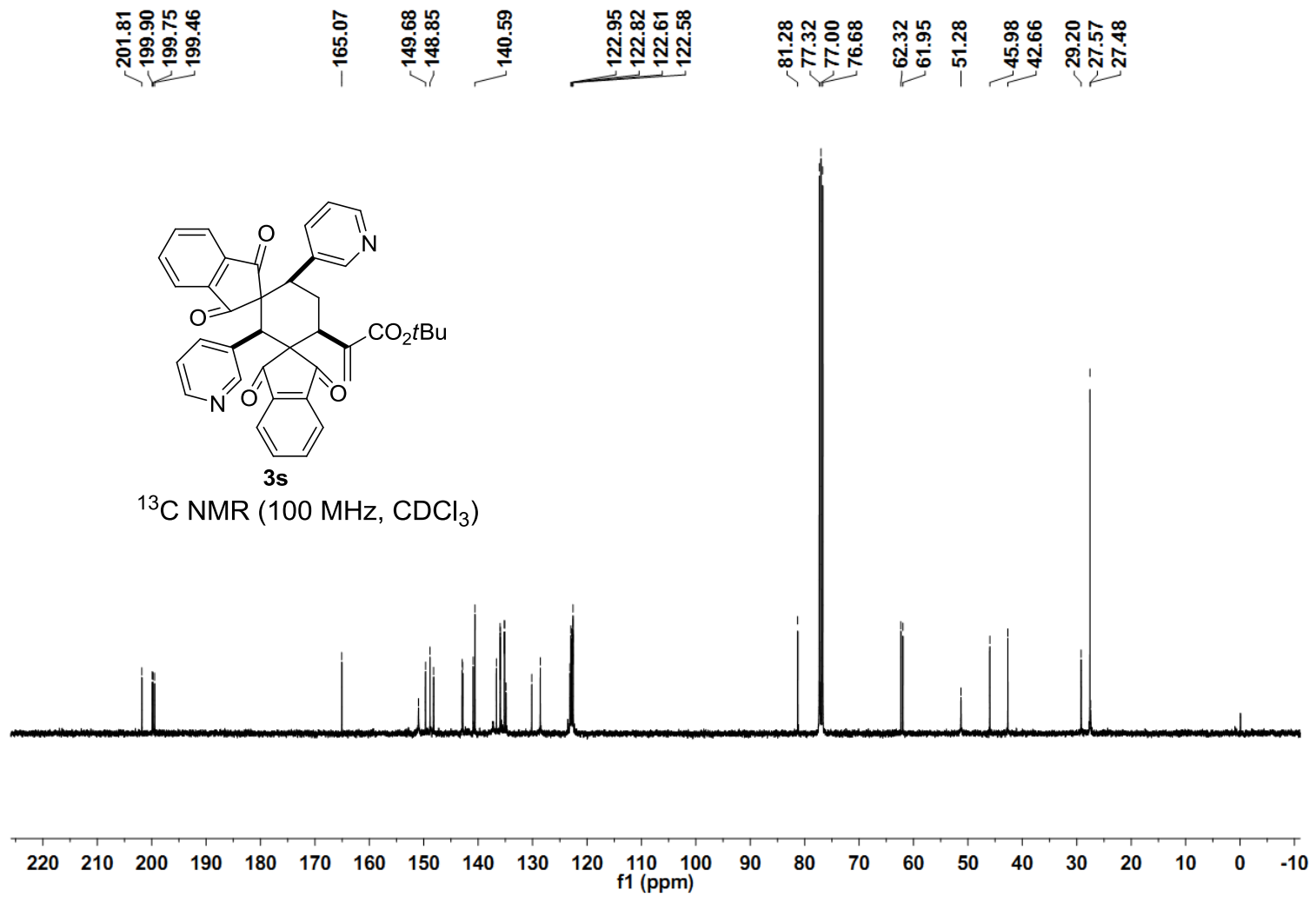


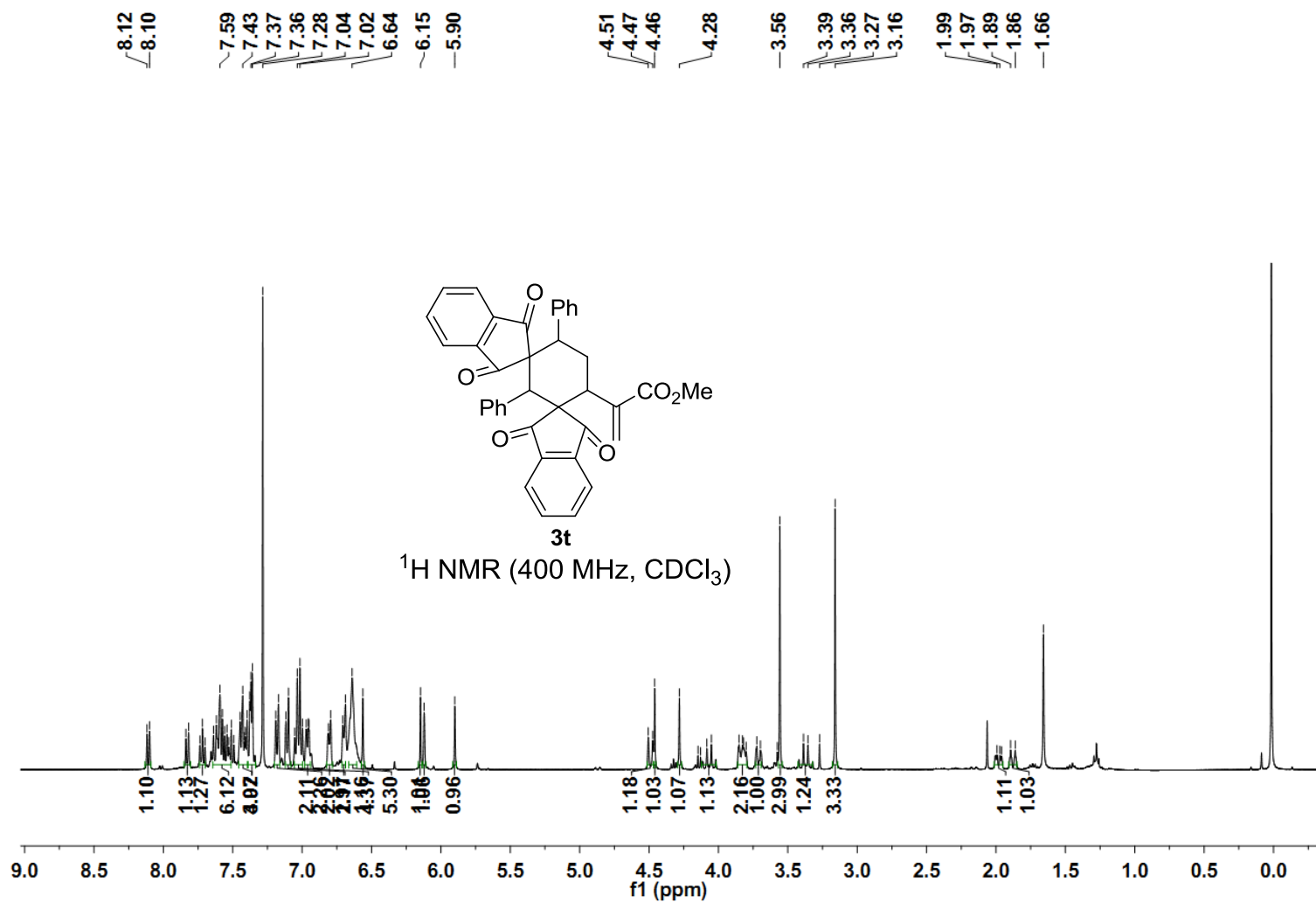


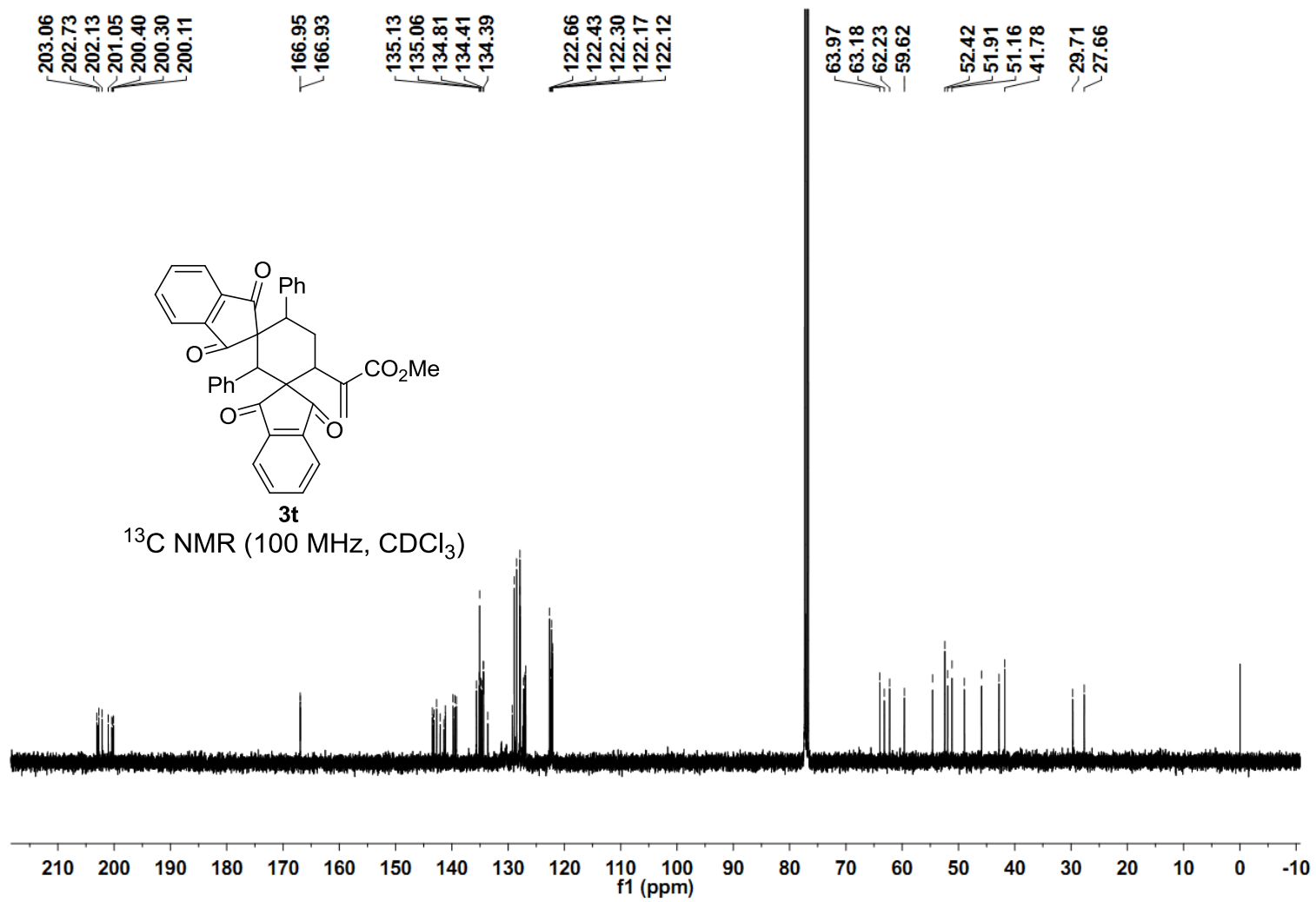


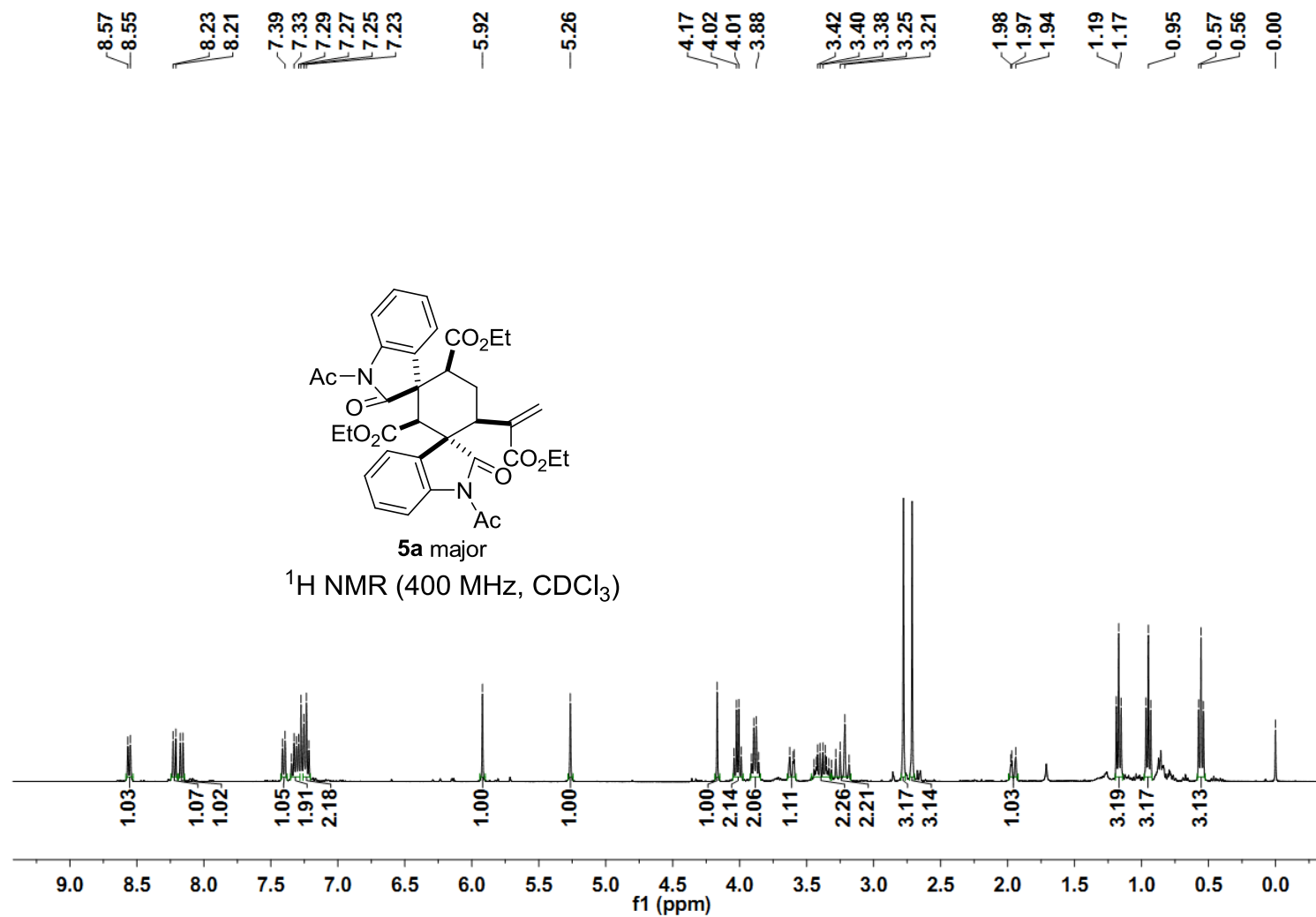




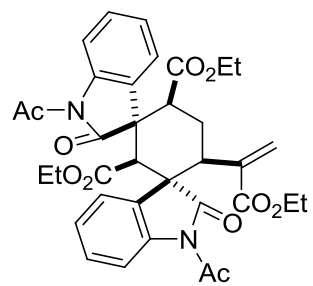






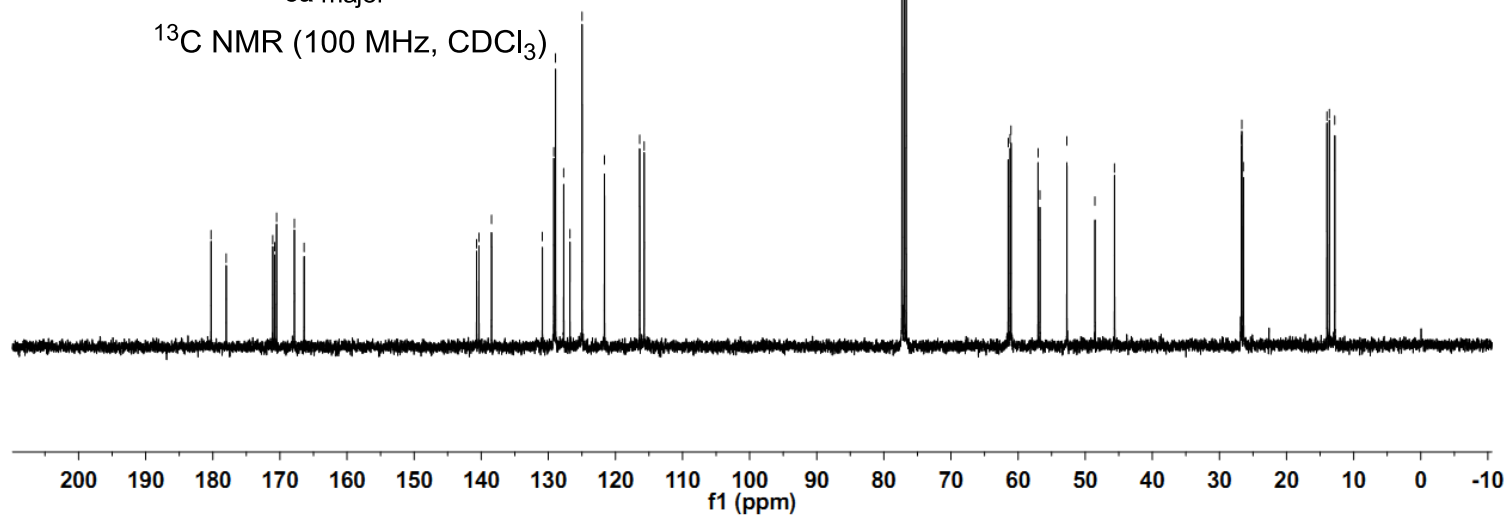


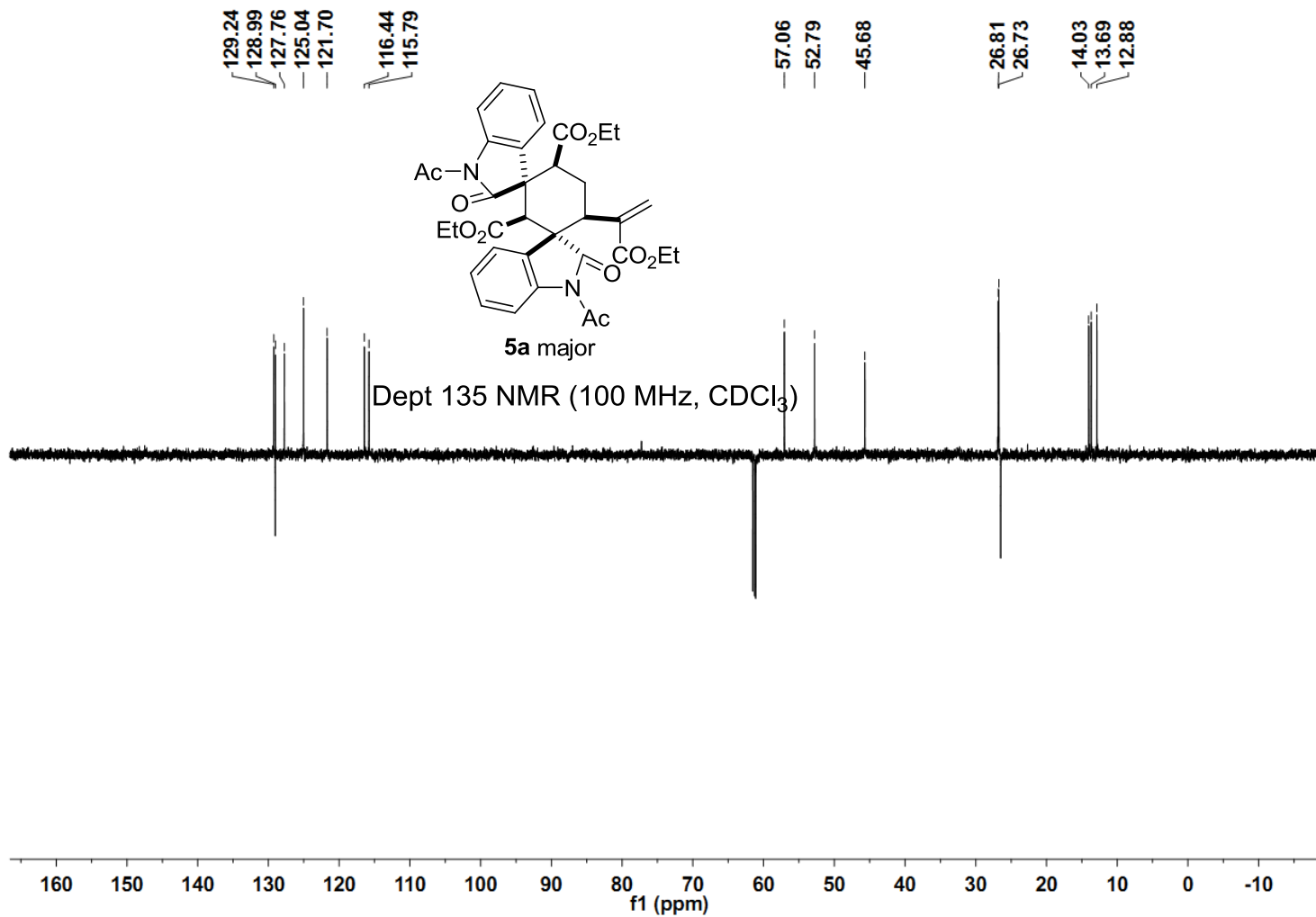
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170.50
167.85
140.70
140.34
138.46
128.94
124.98
116.39
115.73
77.32
77.00
76.68
61.20
61.06
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48.55
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26.74
26.66
26.42
13.97
13.63
12.82

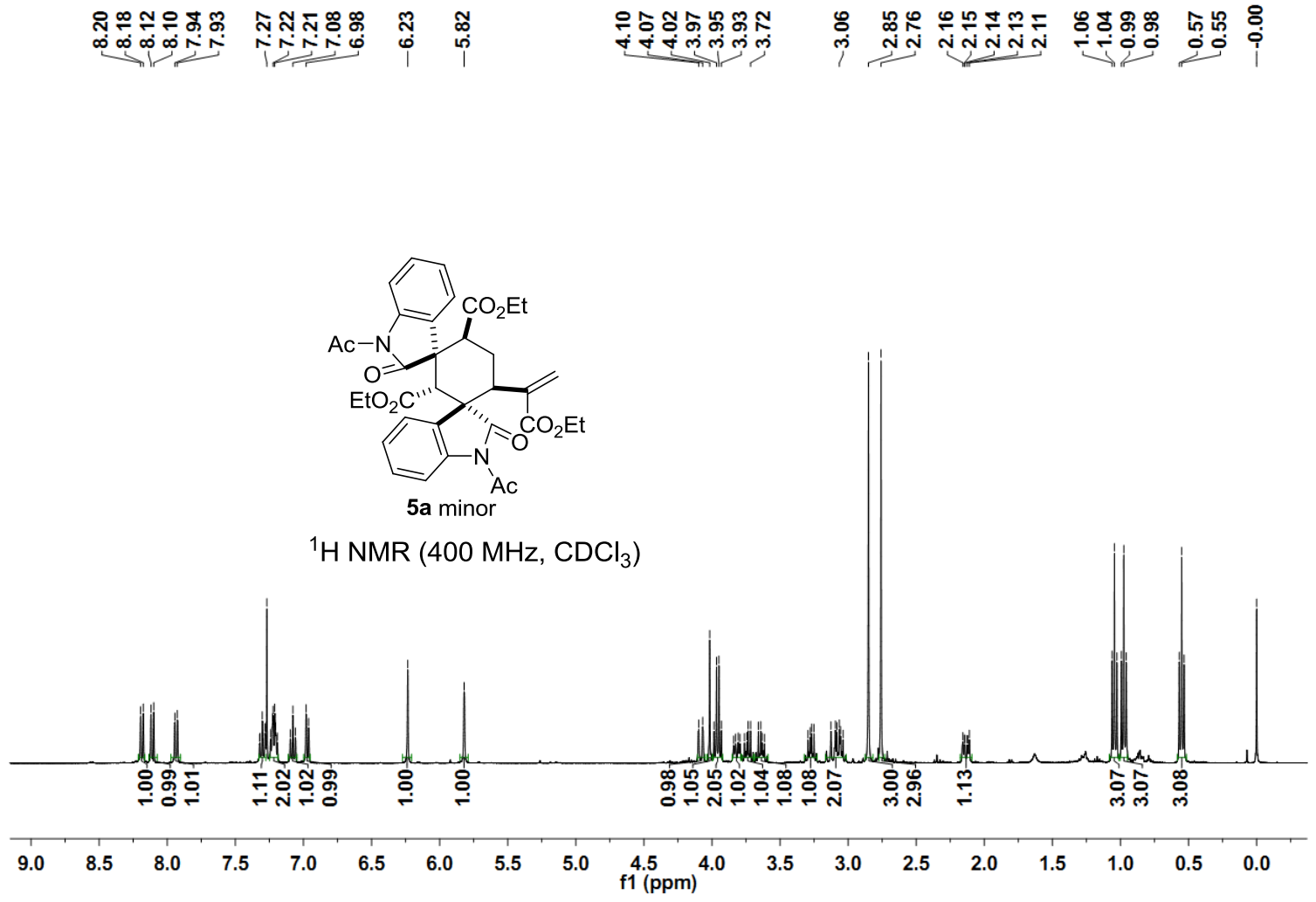


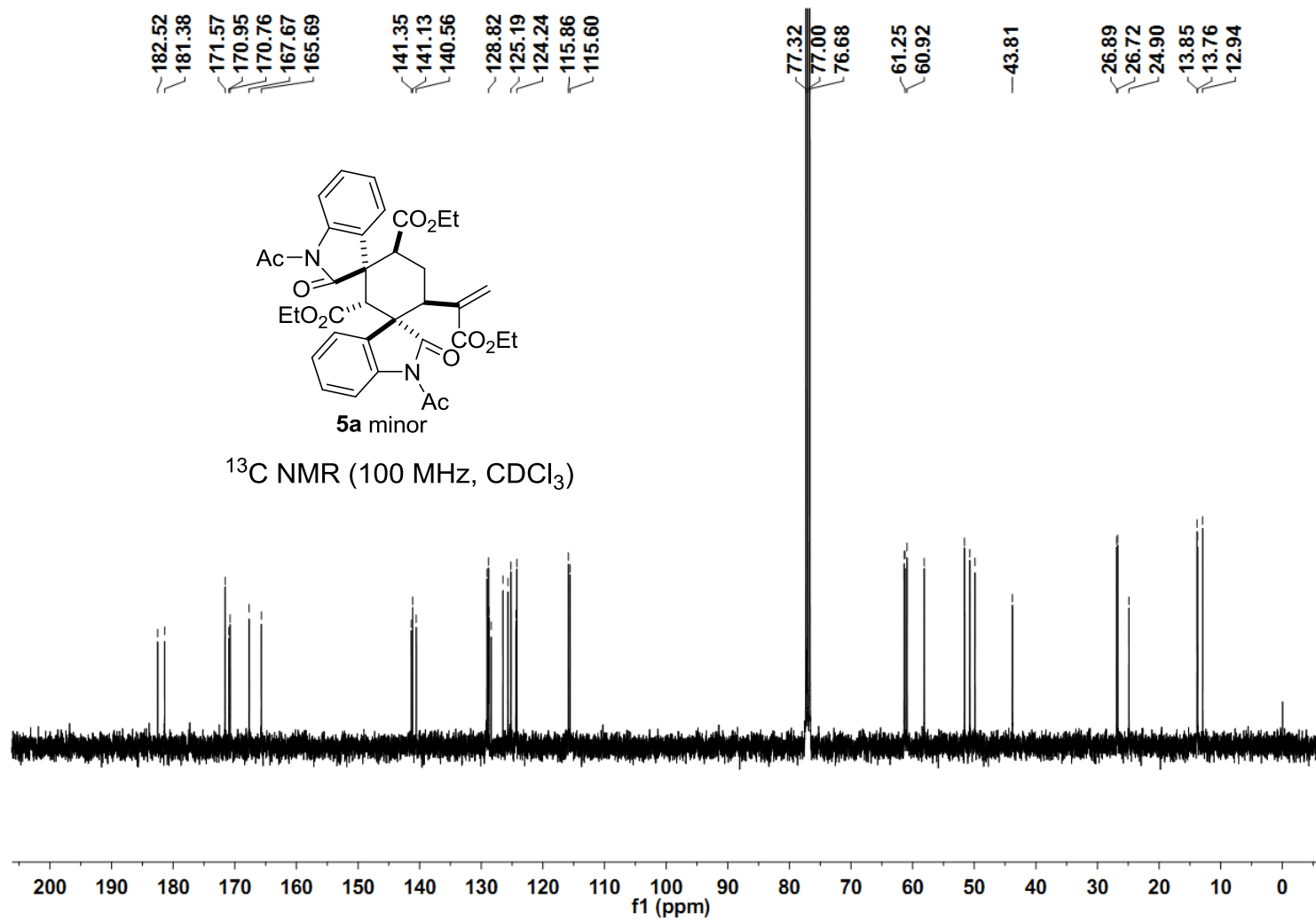
5a major

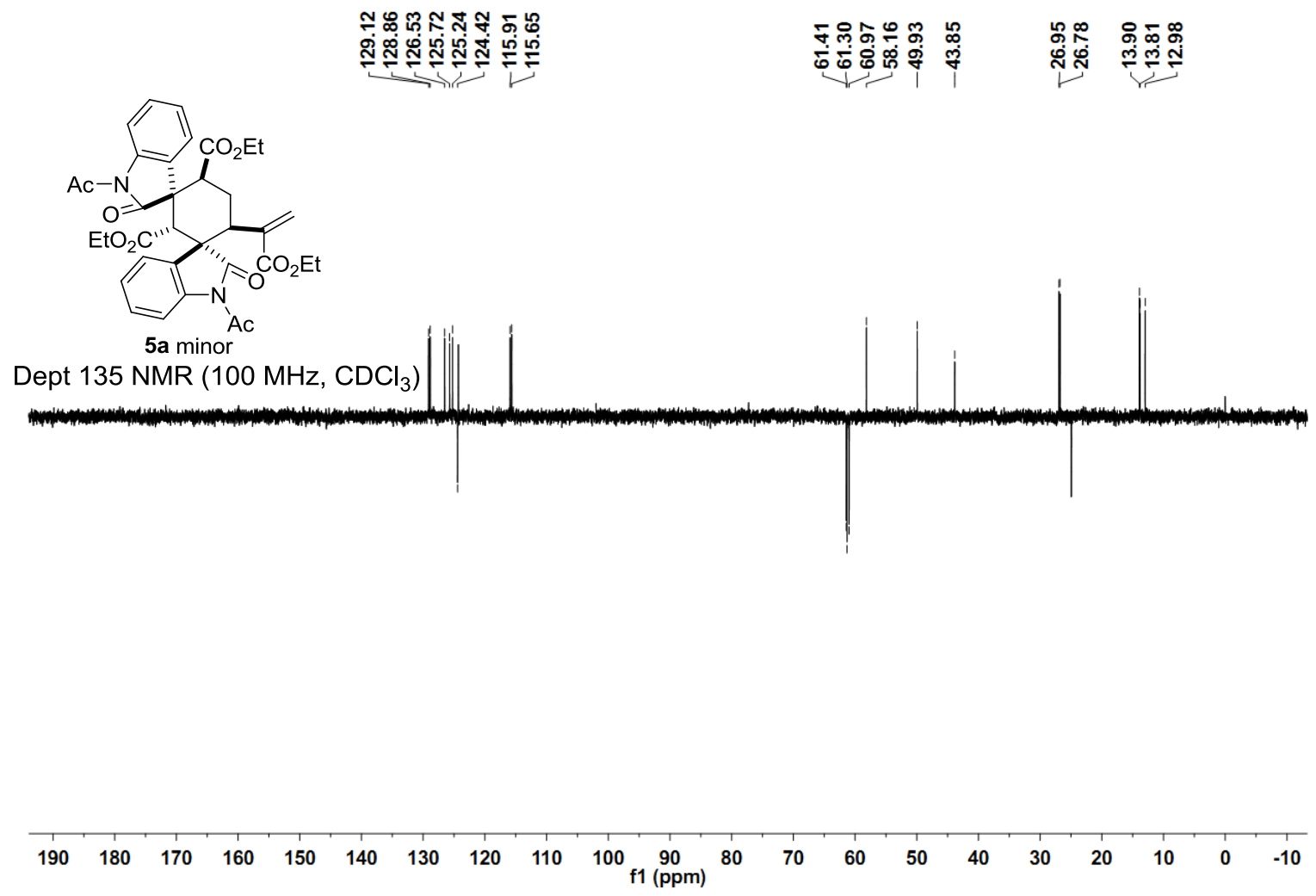
¹³C NMR (100 MHz, CDCl₃)

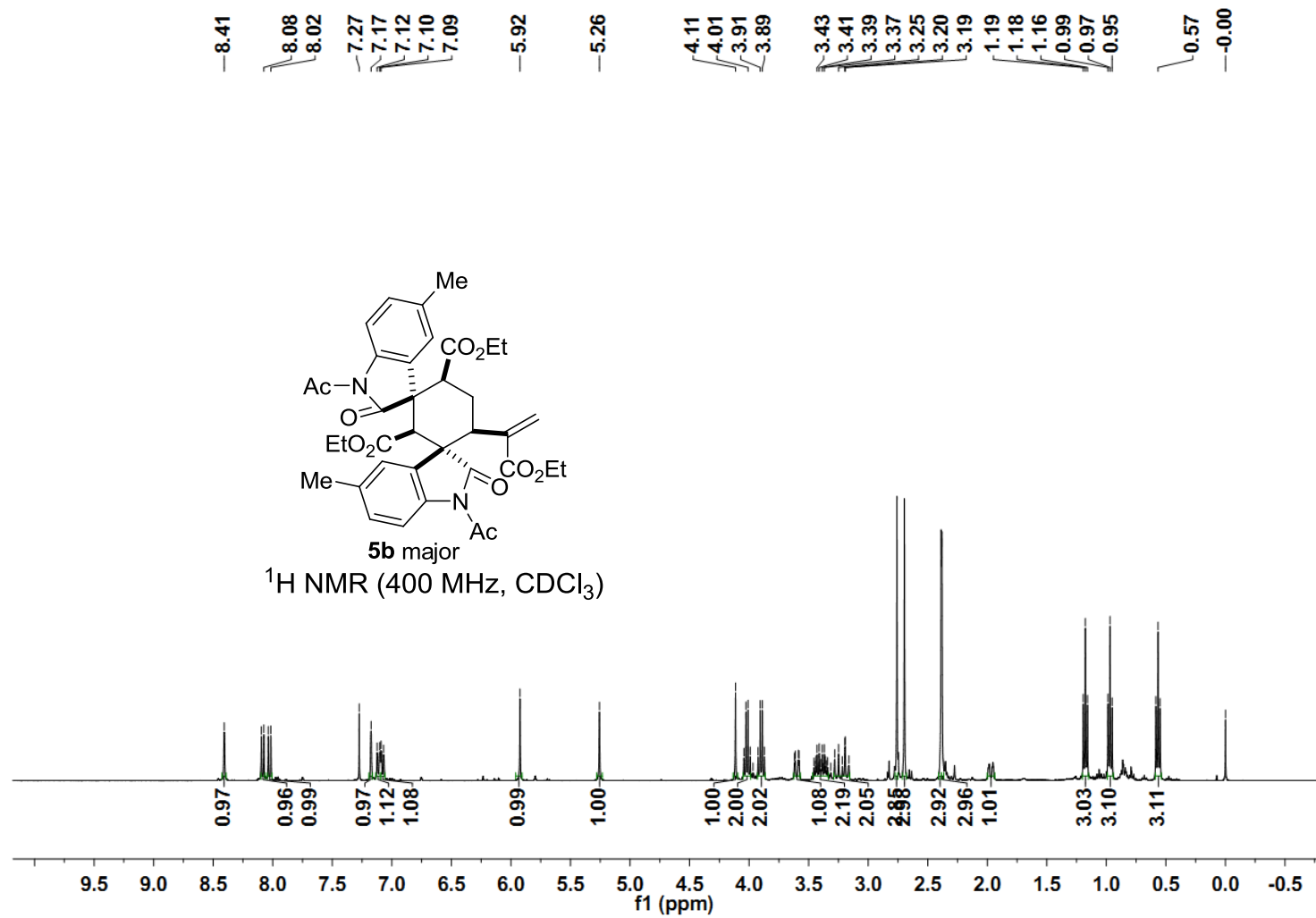


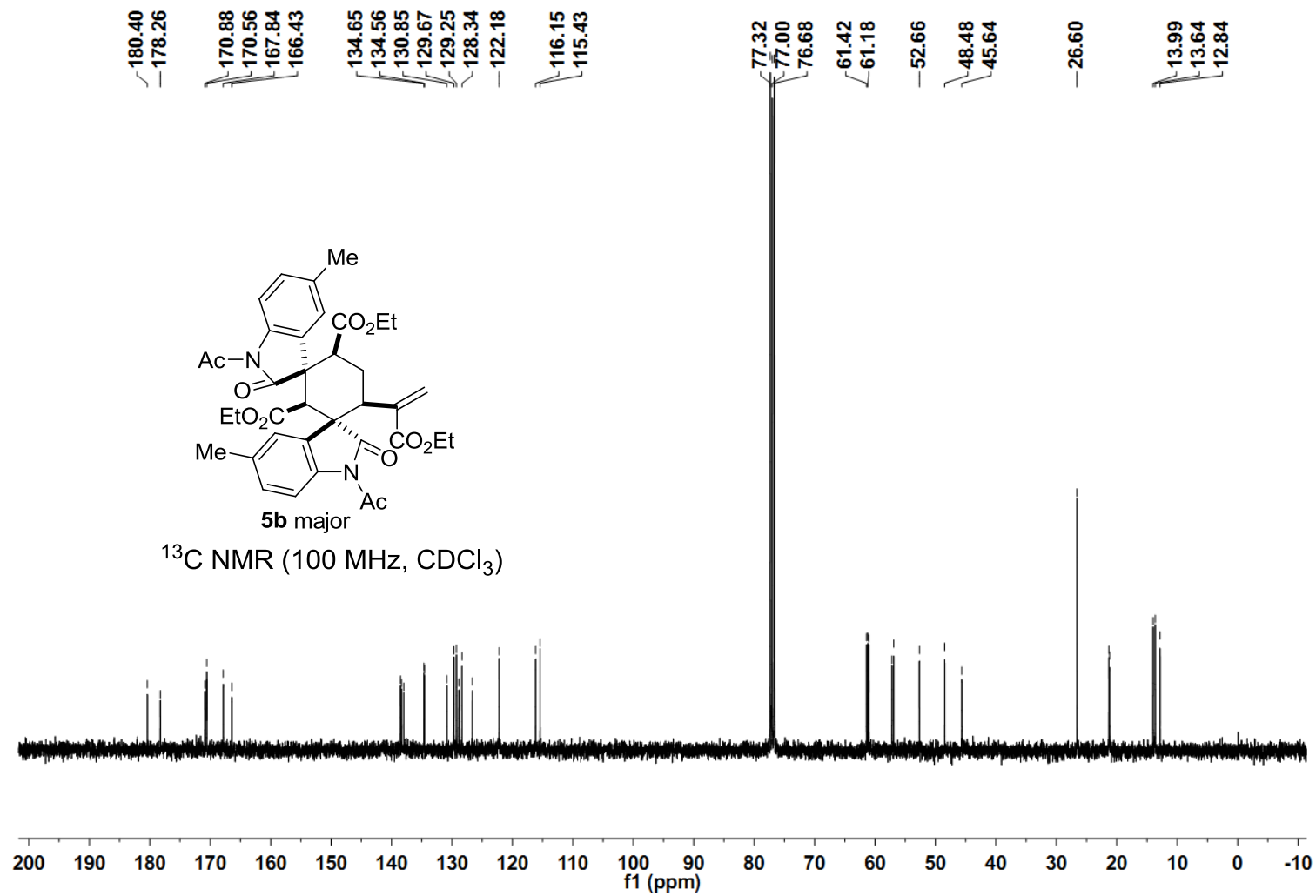


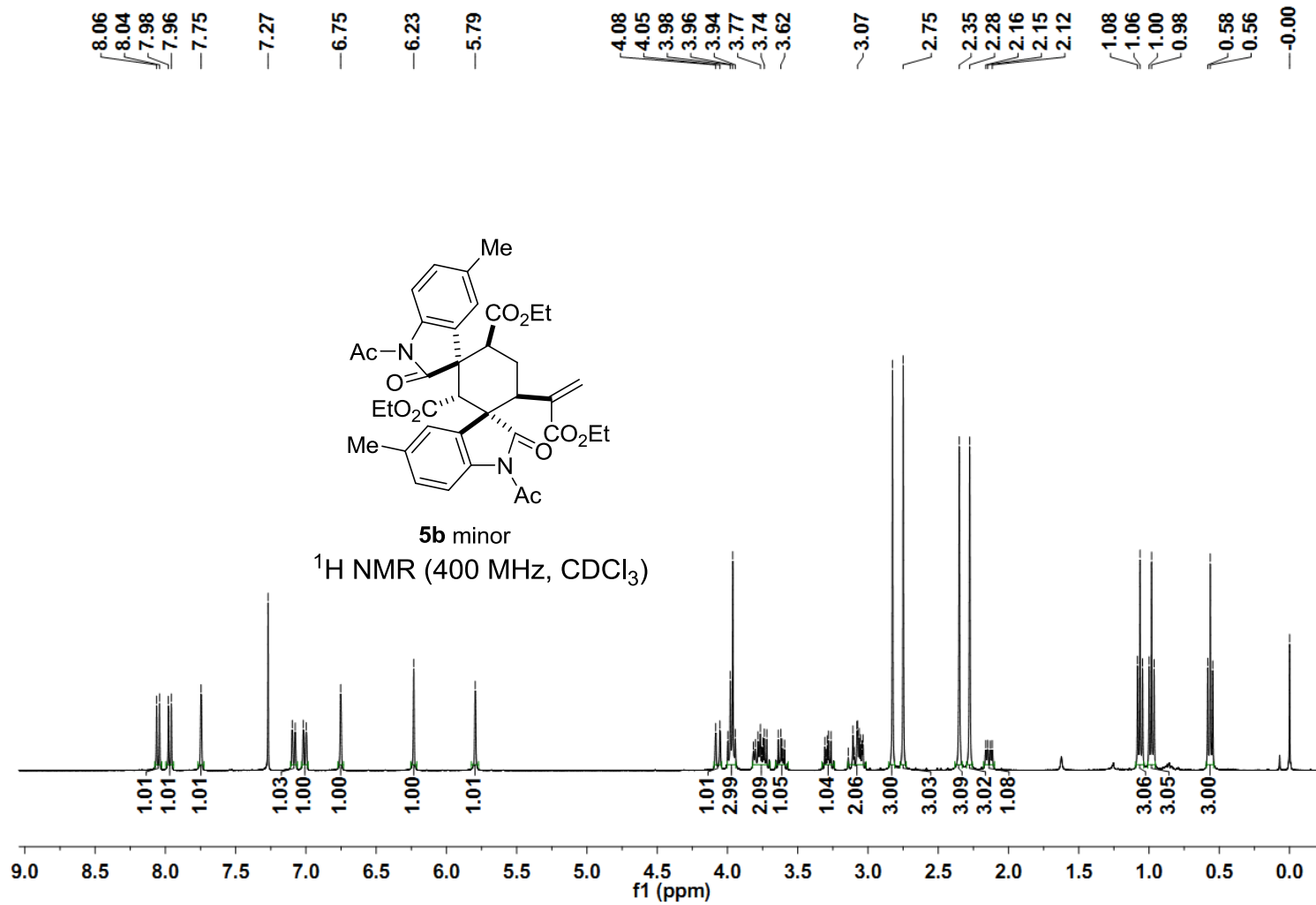


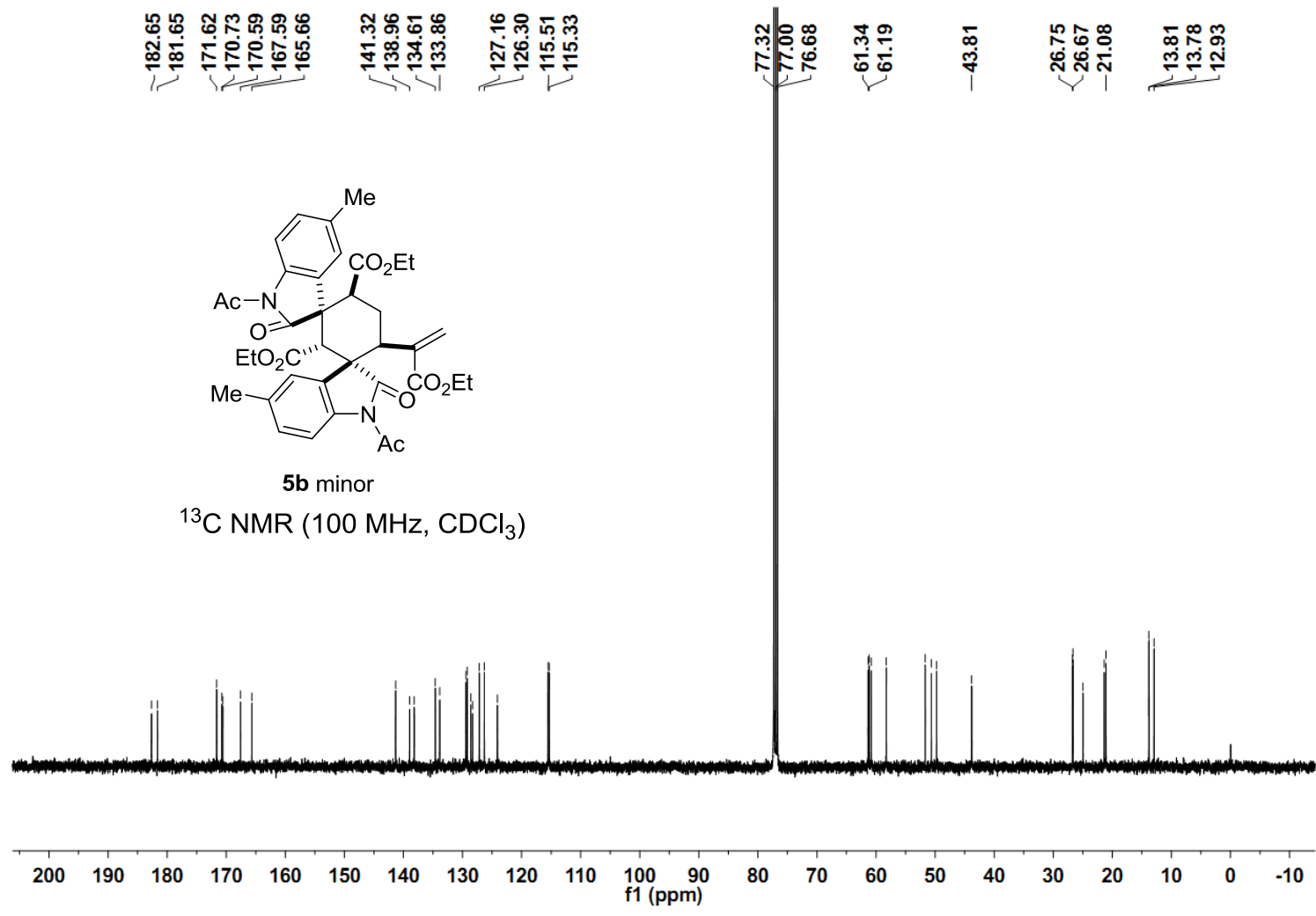


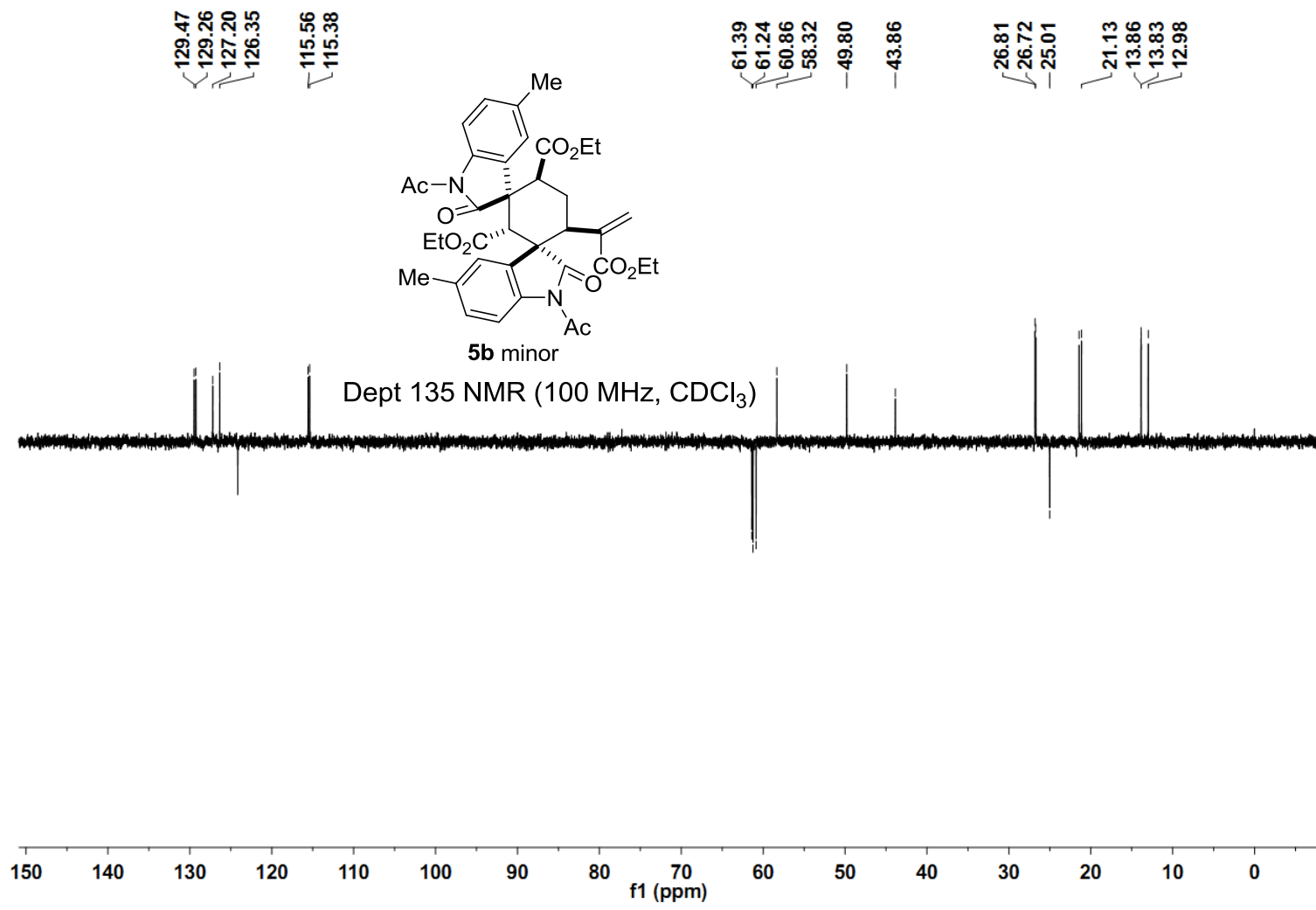


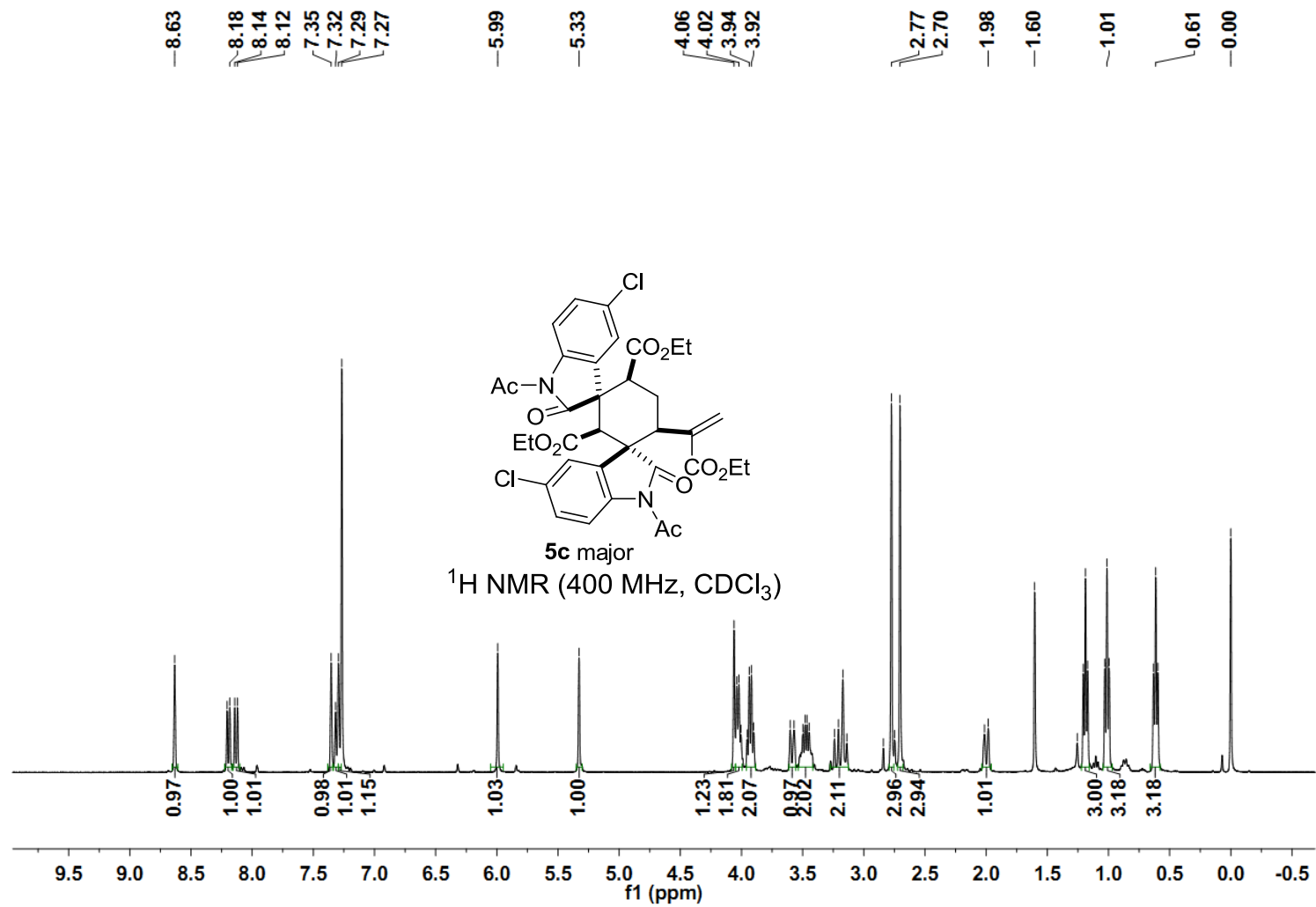


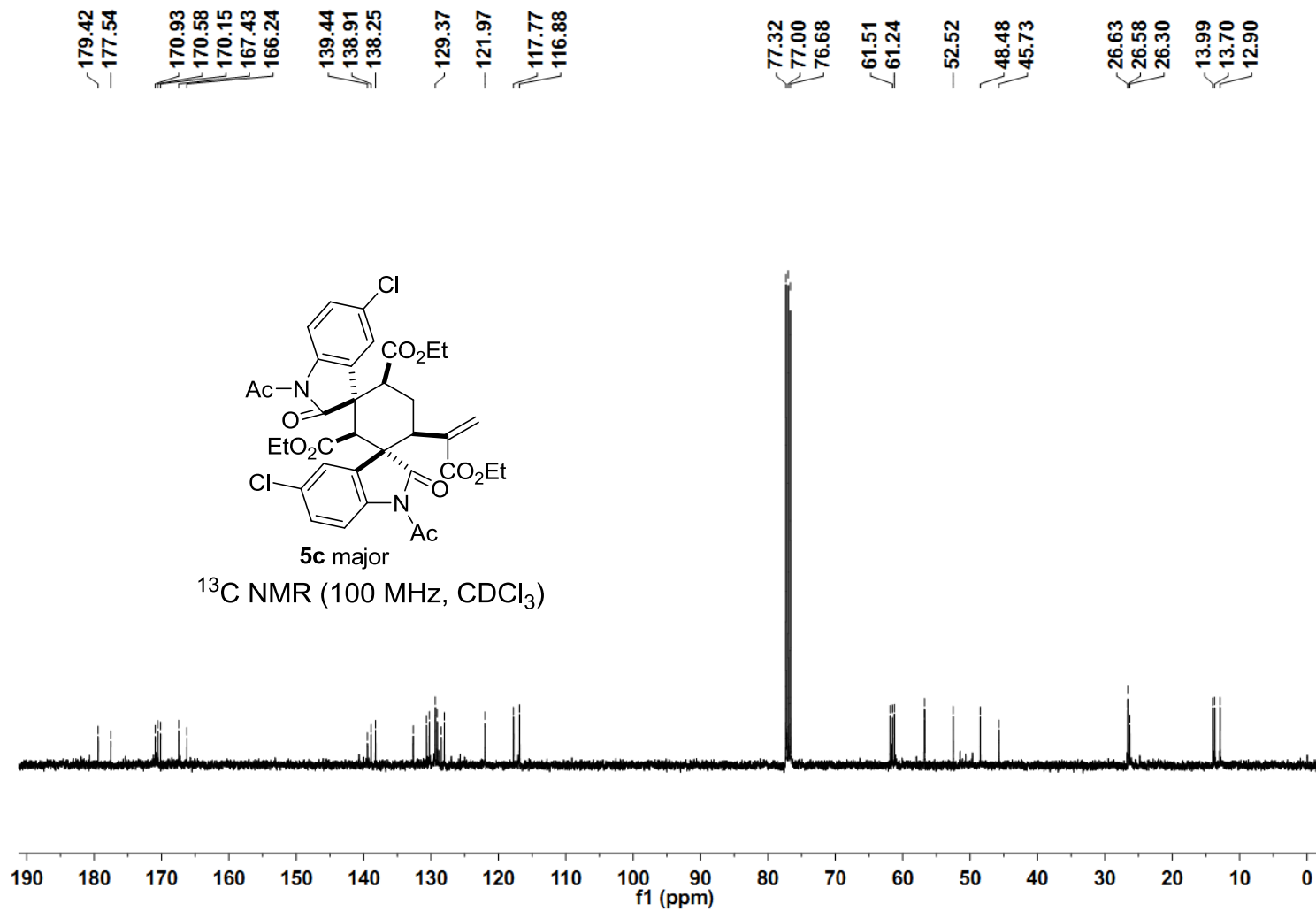


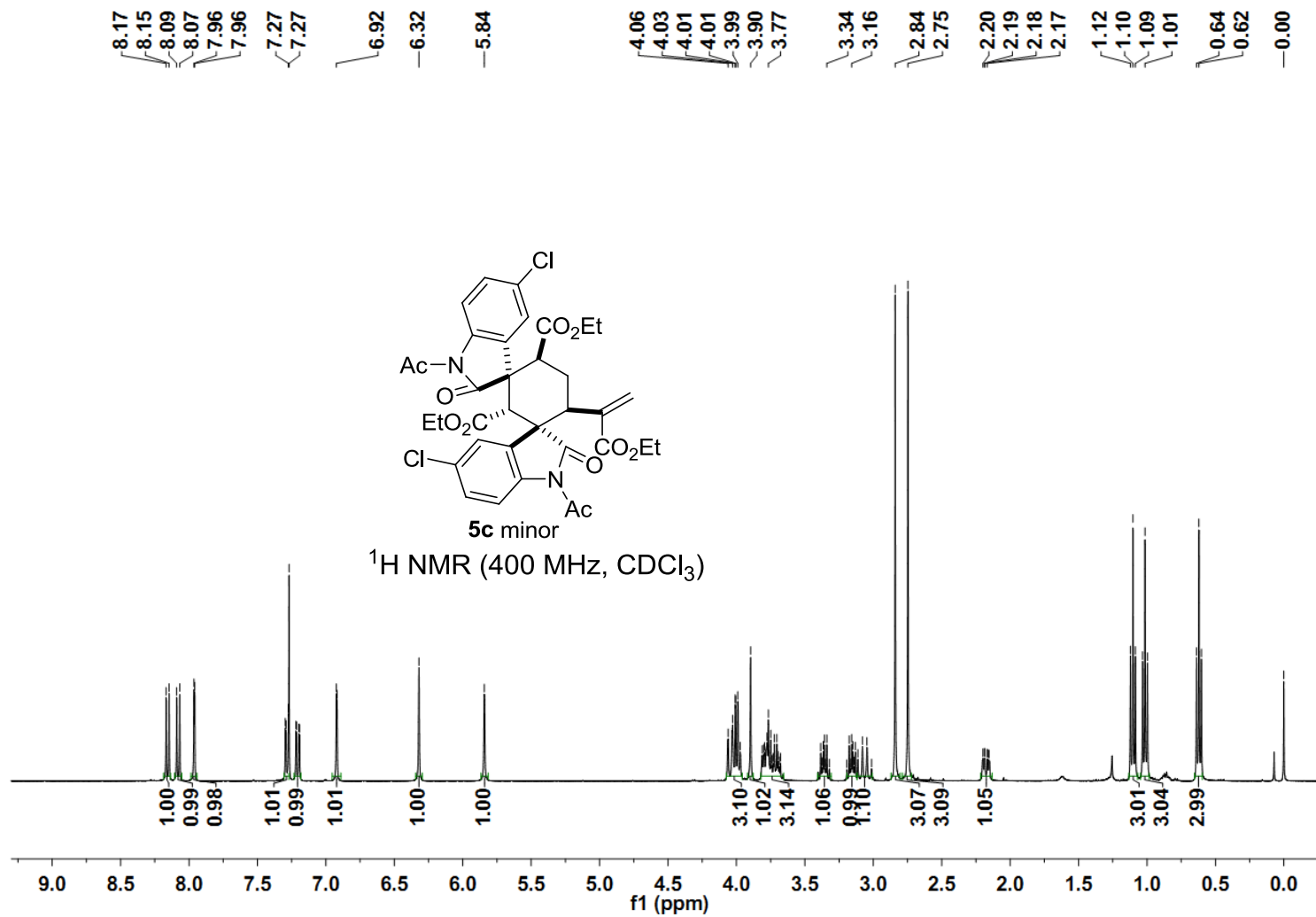












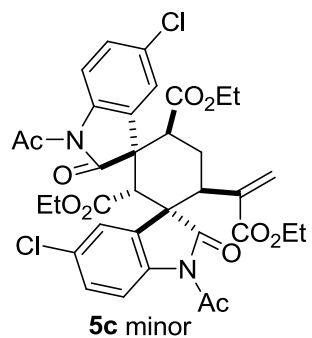
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180.69
171.20
170.77
170.53
167.26
165.71
140.68
140.00
139.26
128.88
126.99
125.67
117.07
116.85

77.32
77.00
76.68

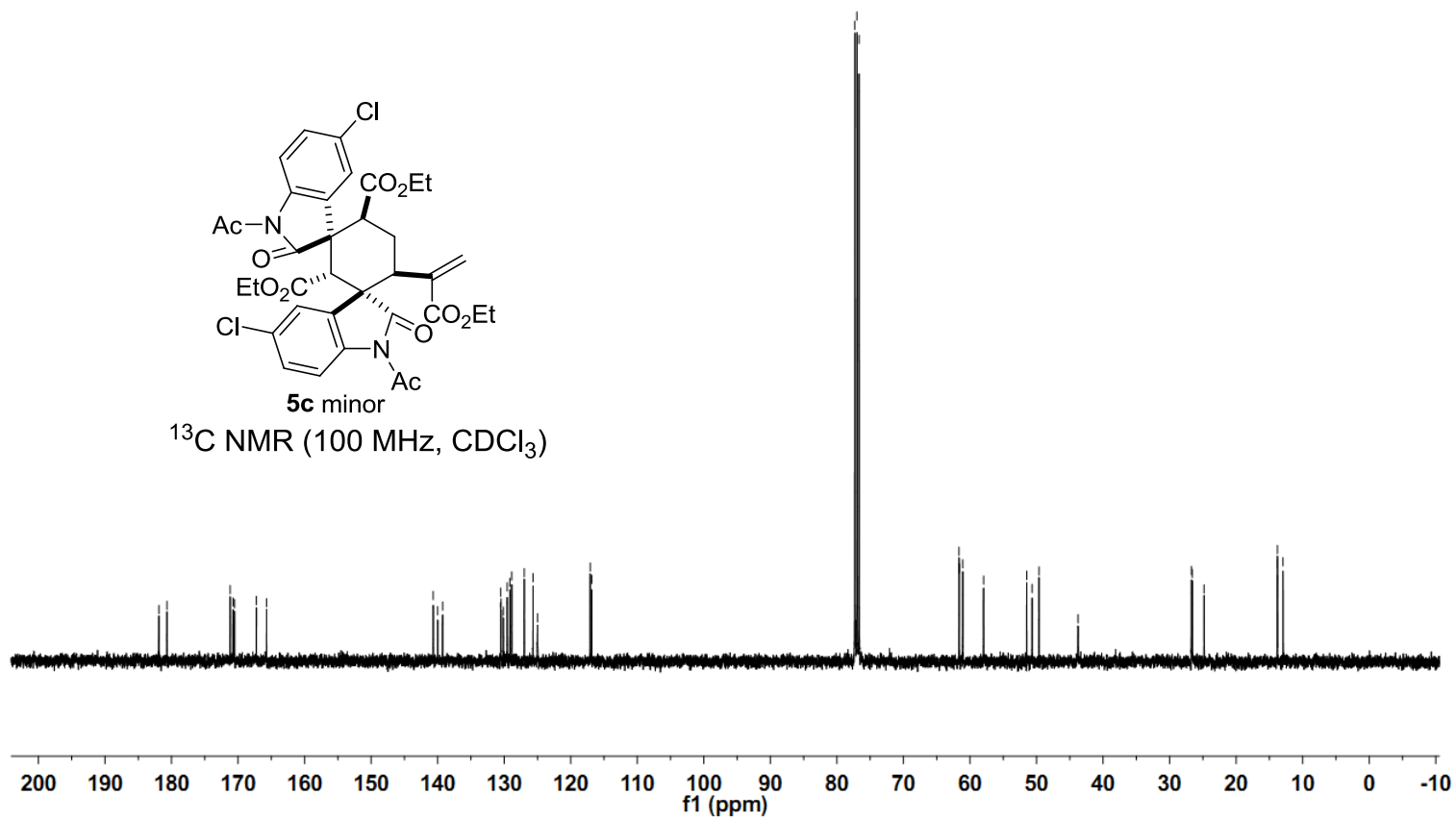
61.65
61.09

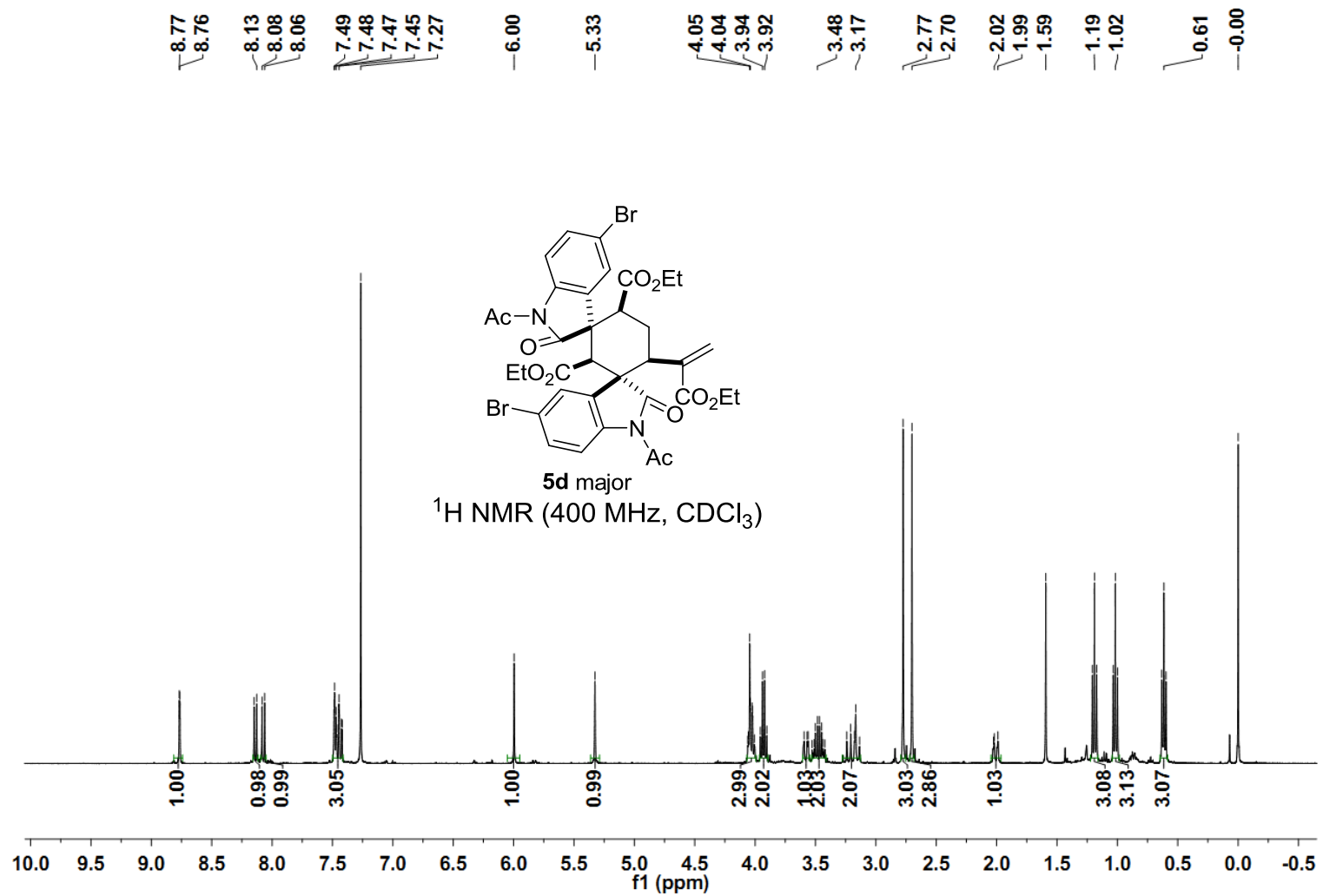
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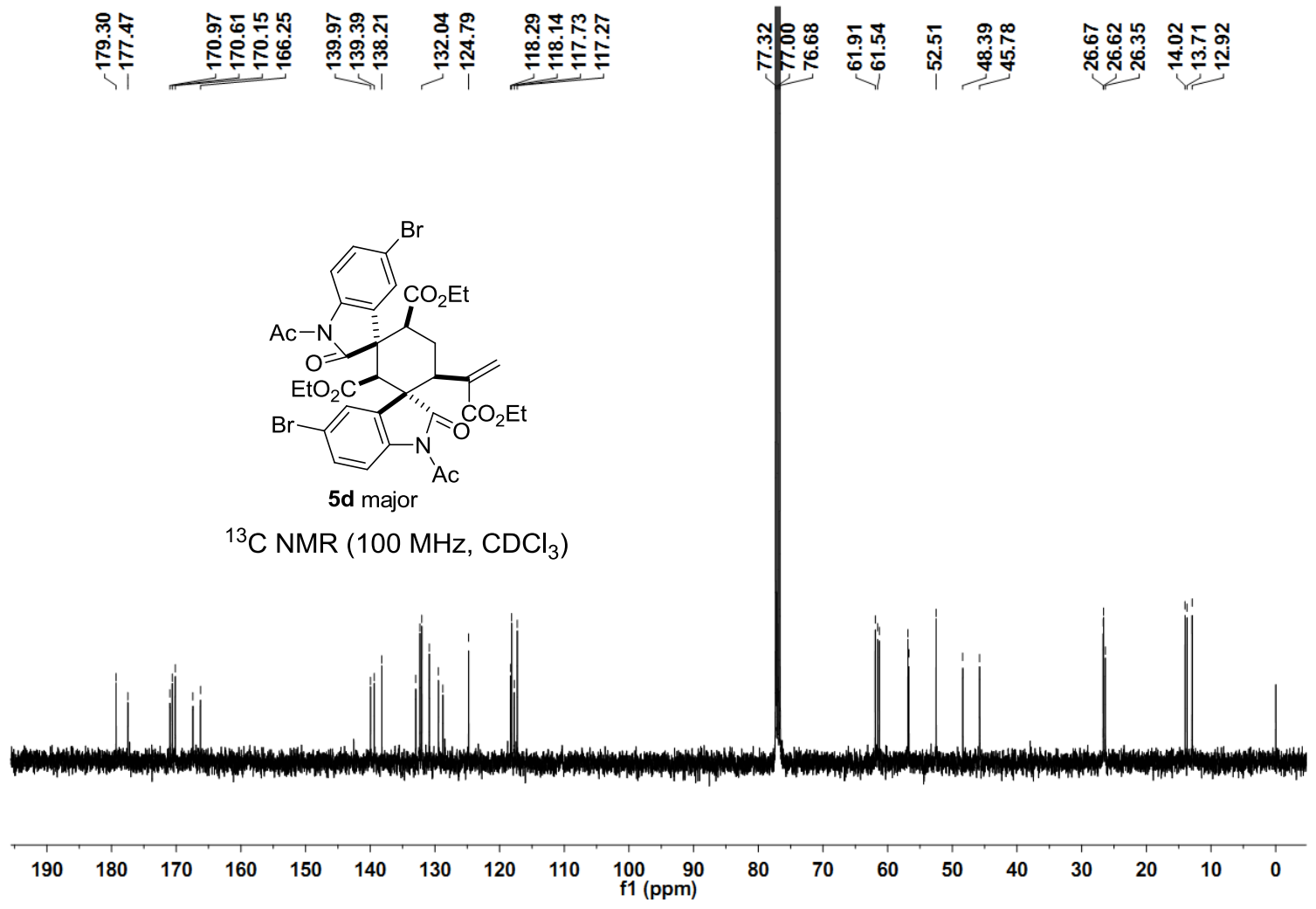
26.72
26.59
24.82
13.85
13.80
12.96

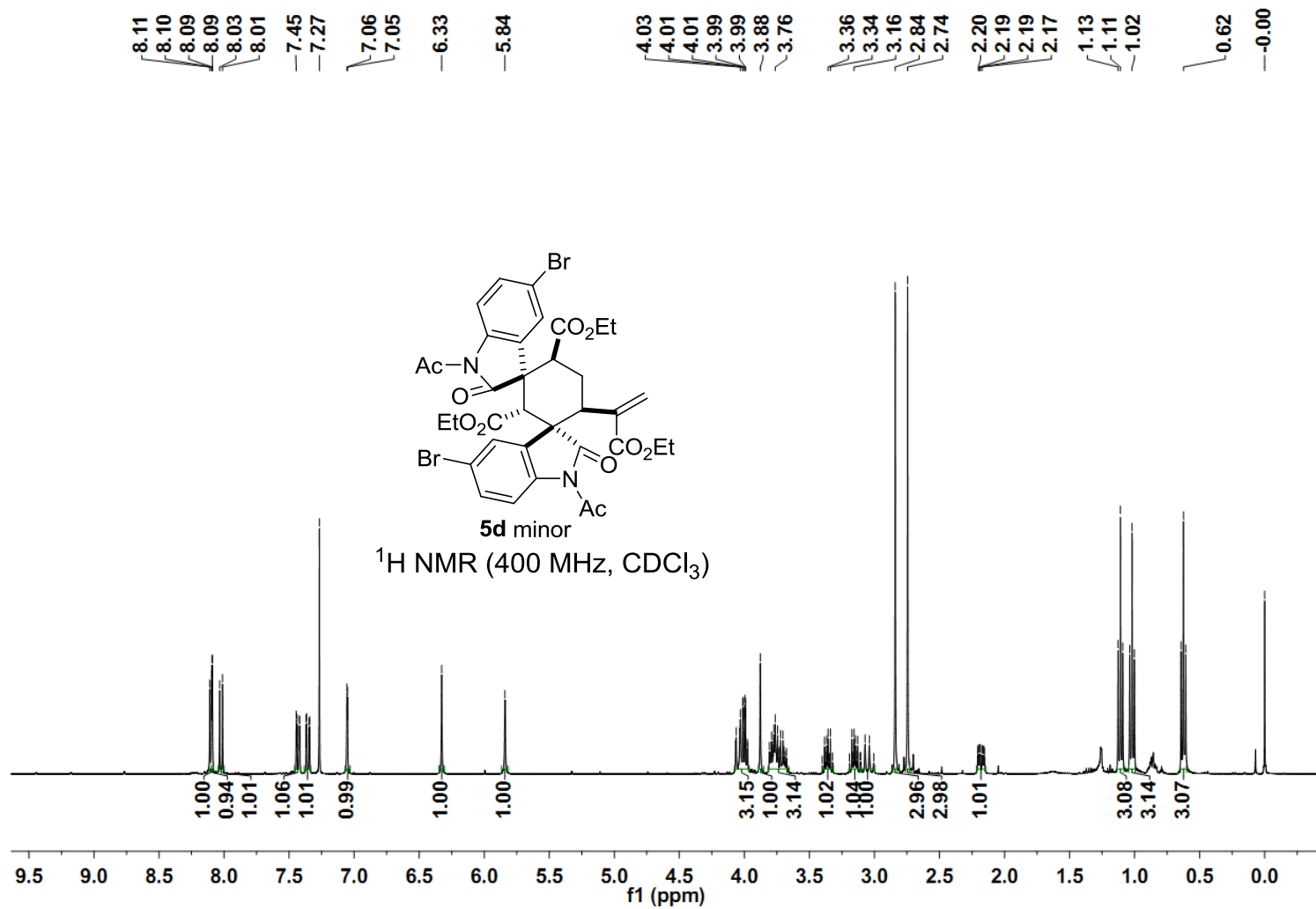


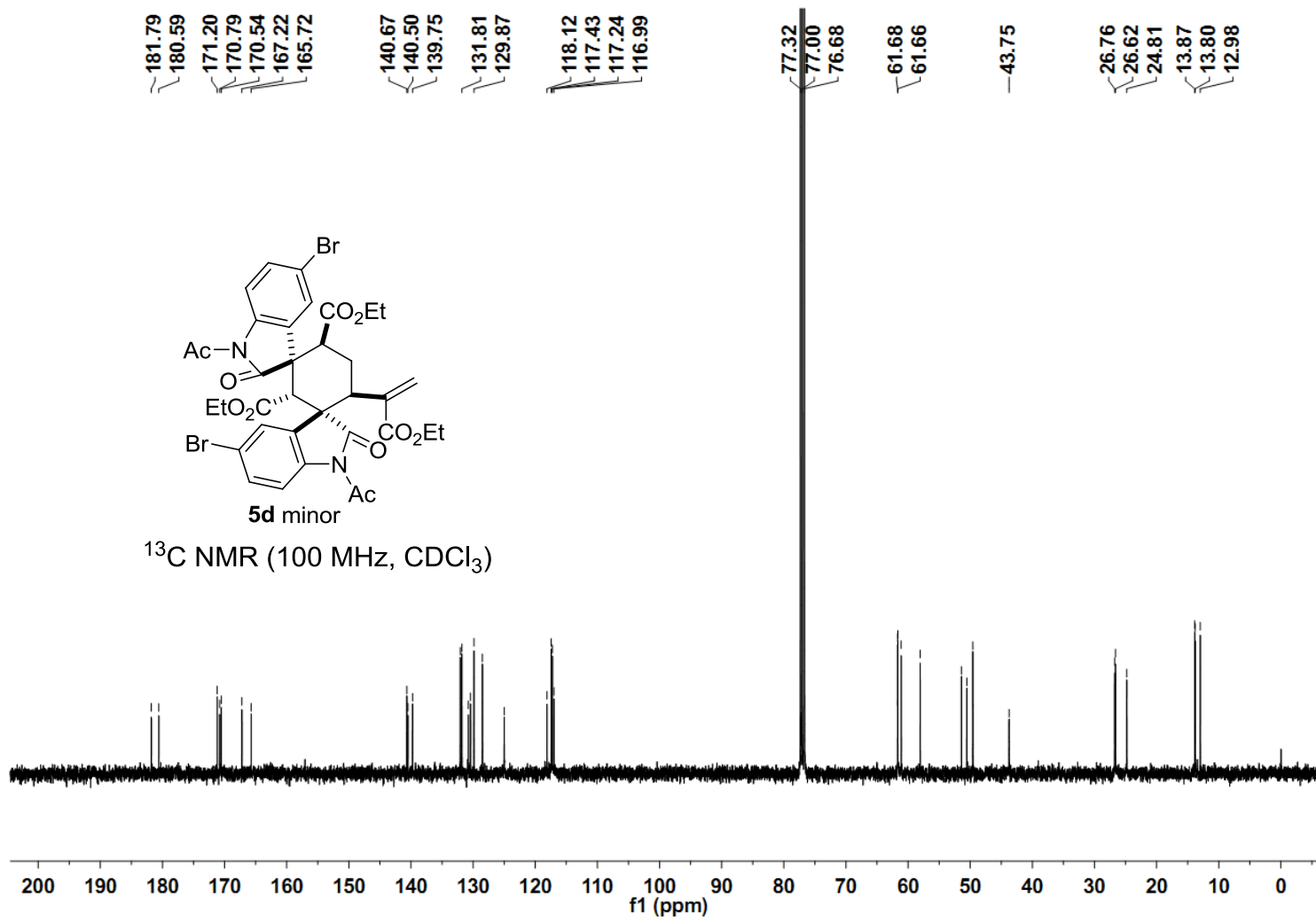
¹³C NMR (100 MHz, CDCl₃)











8.44
8.44
8.43
8.41
8.39
8.39

7.40
7.37
7.37
7.27
7.25
7.23

5.97

5.28

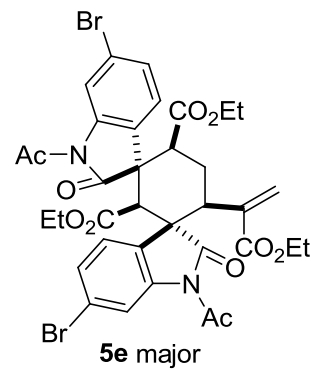
4.05
4.01
3.93
3.91

3.15
2.75
2.70

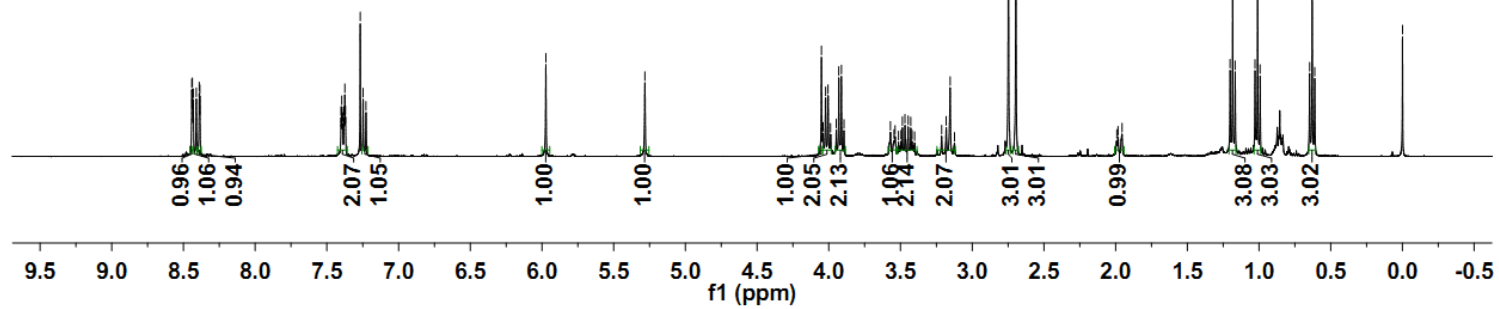
1.99
1.98
1.96

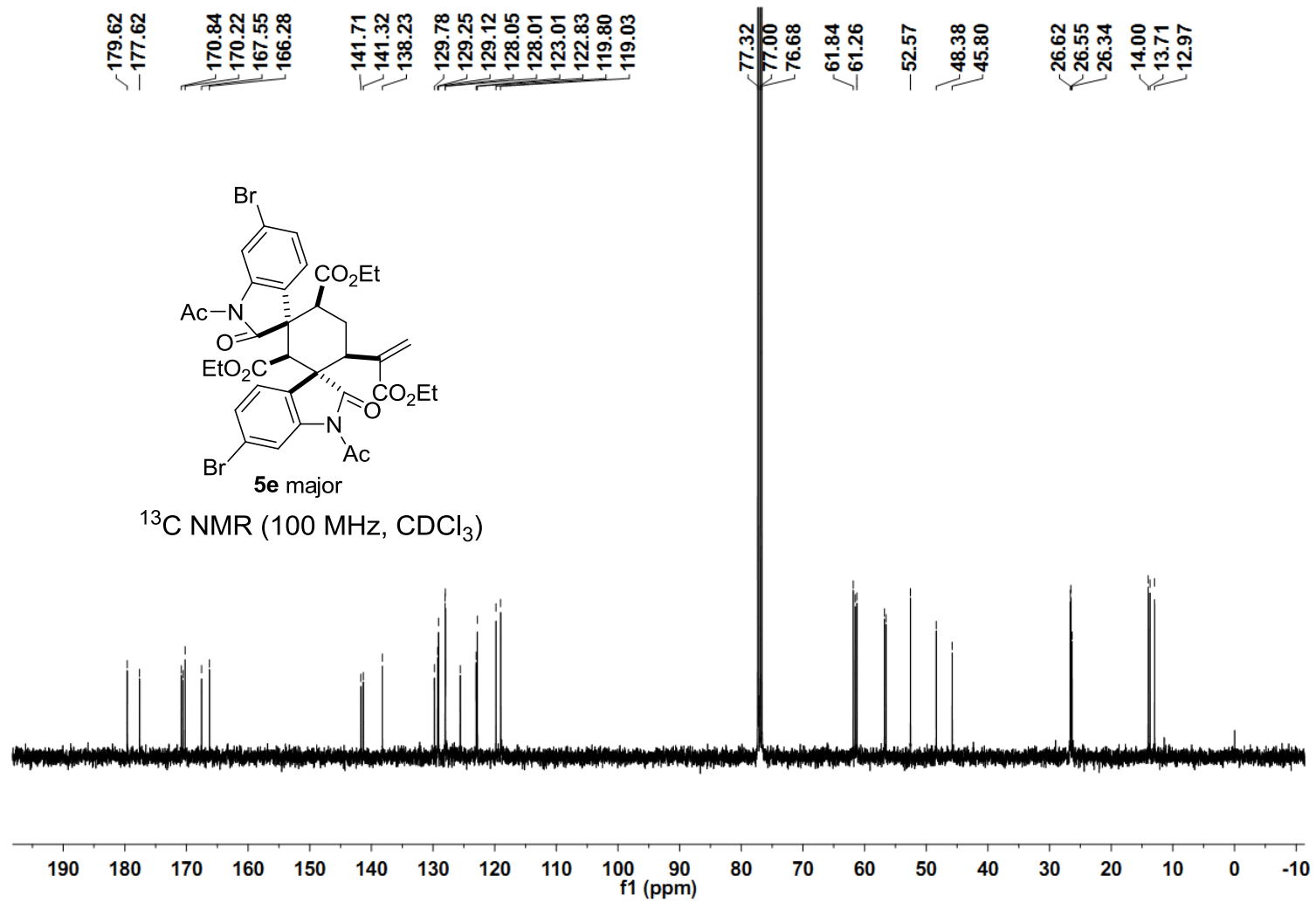
1.20
1.18
1.01

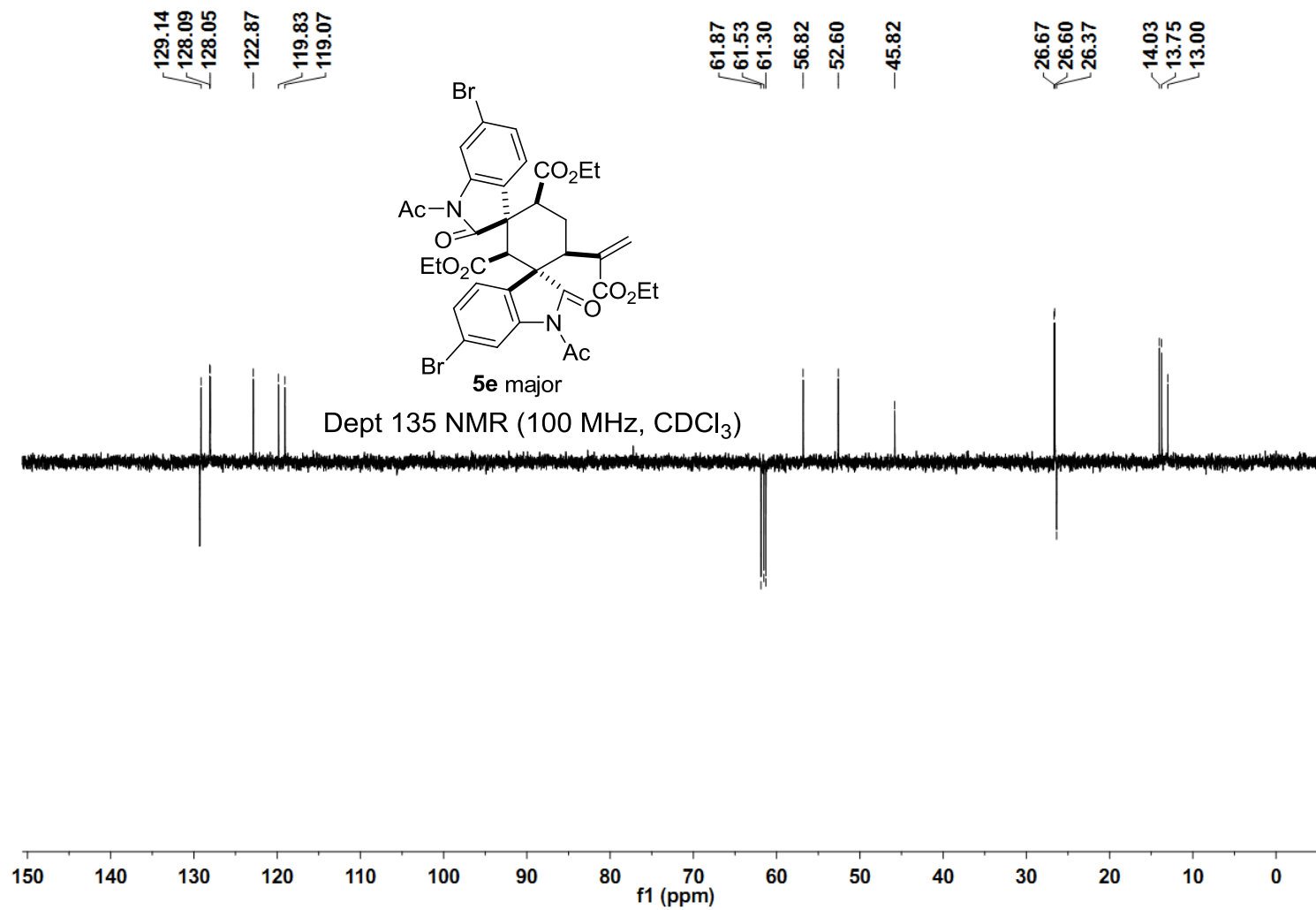
0.63
-0.00

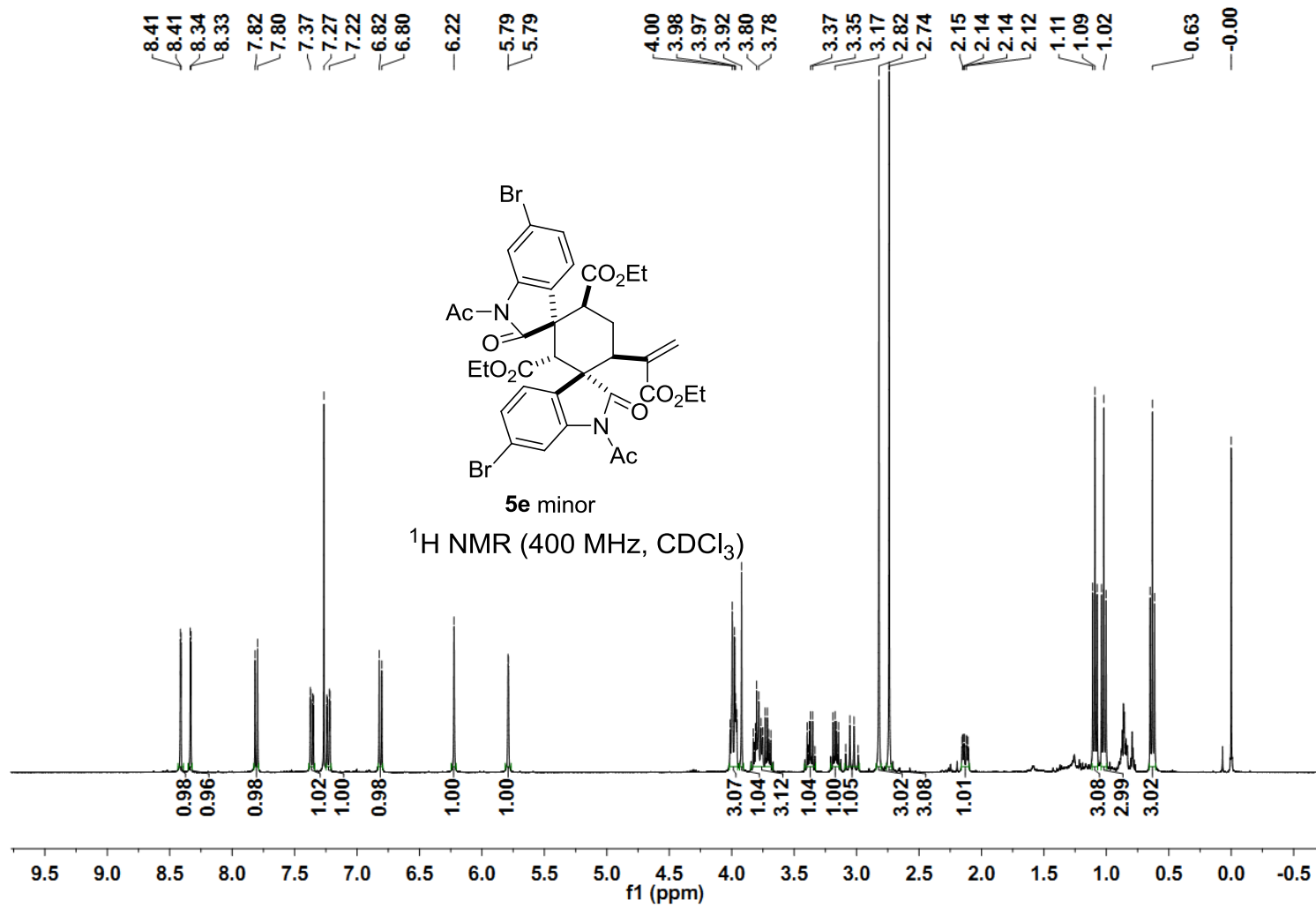


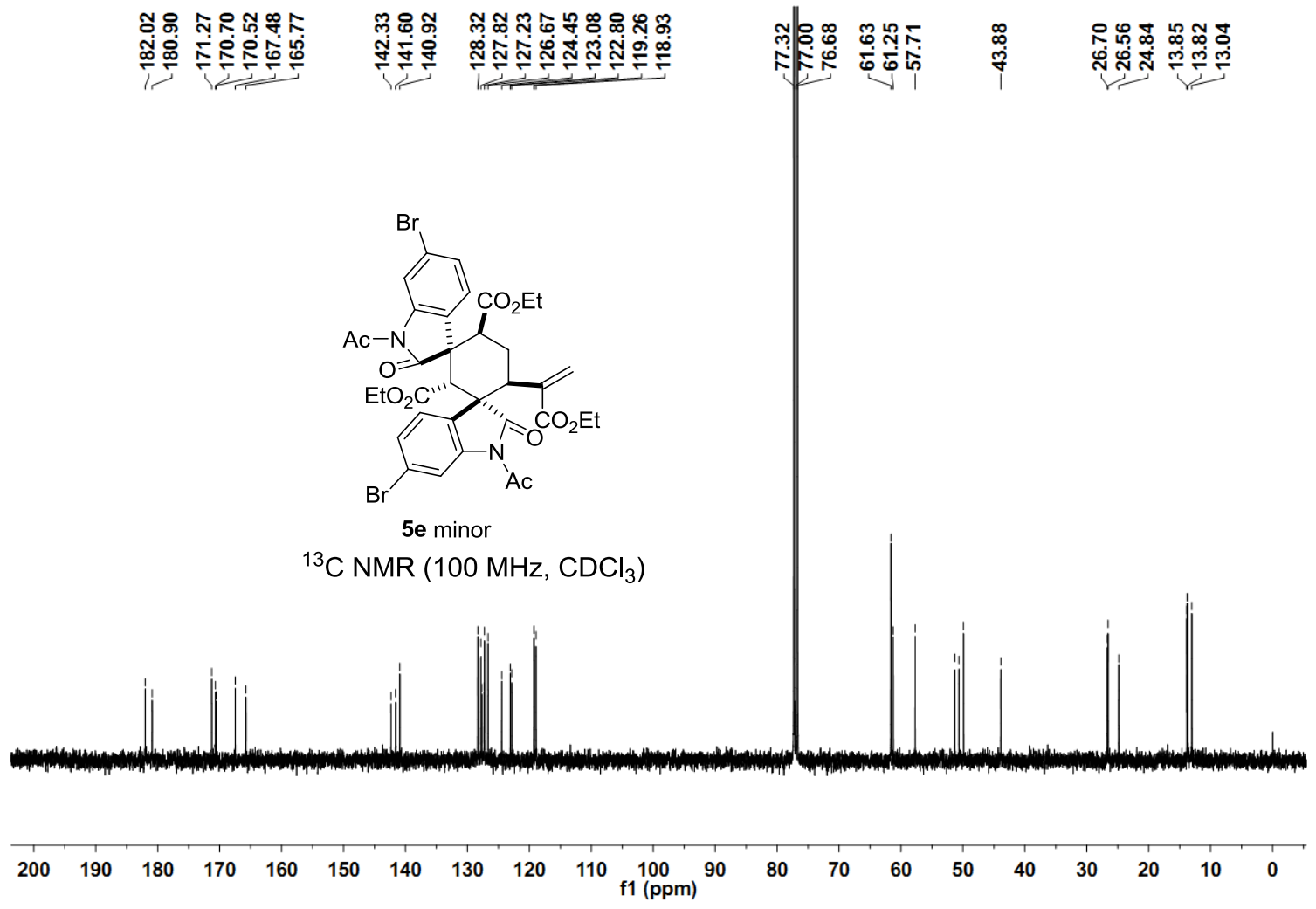
¹H NMR (400 MHz, CDCl₃)

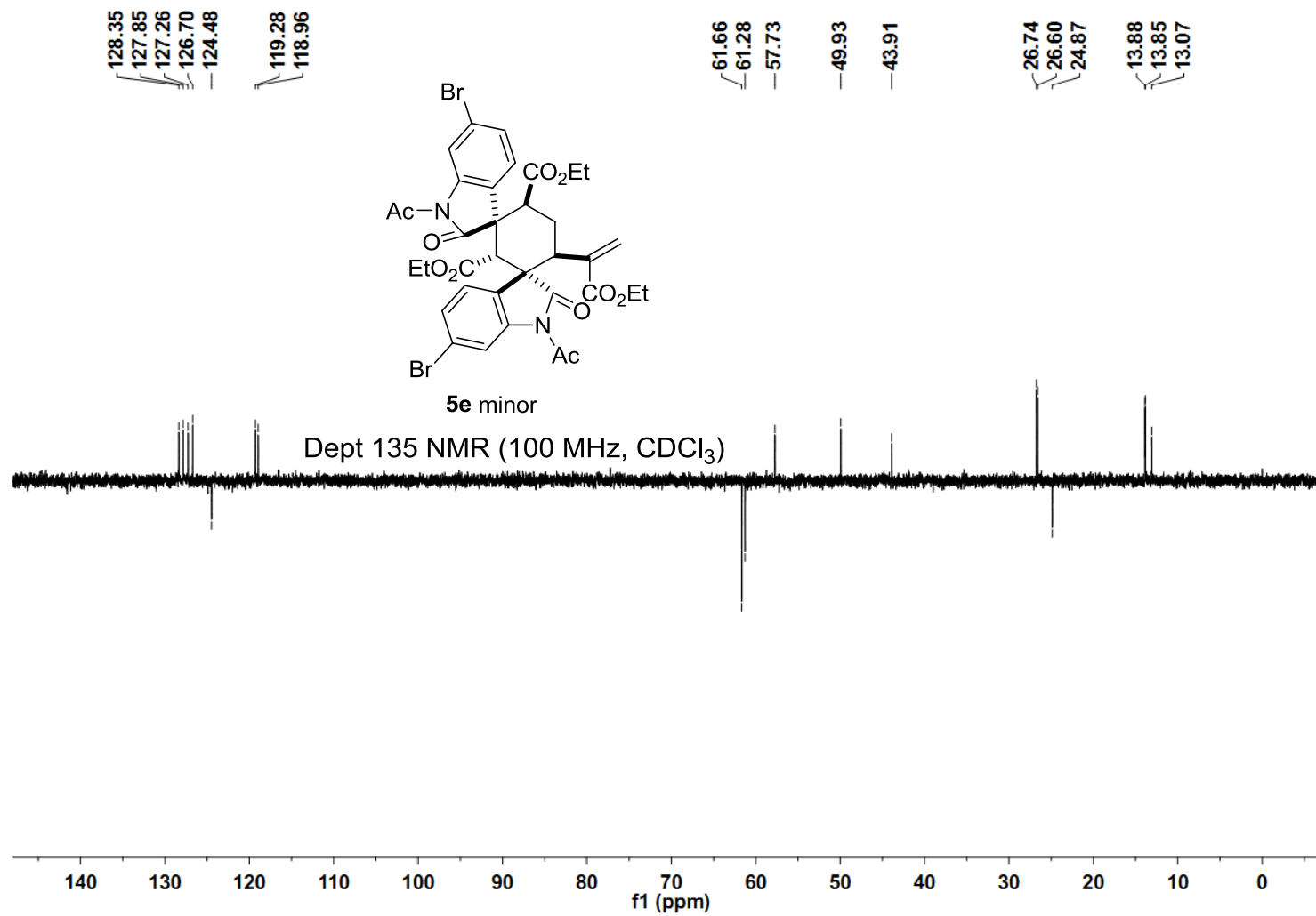


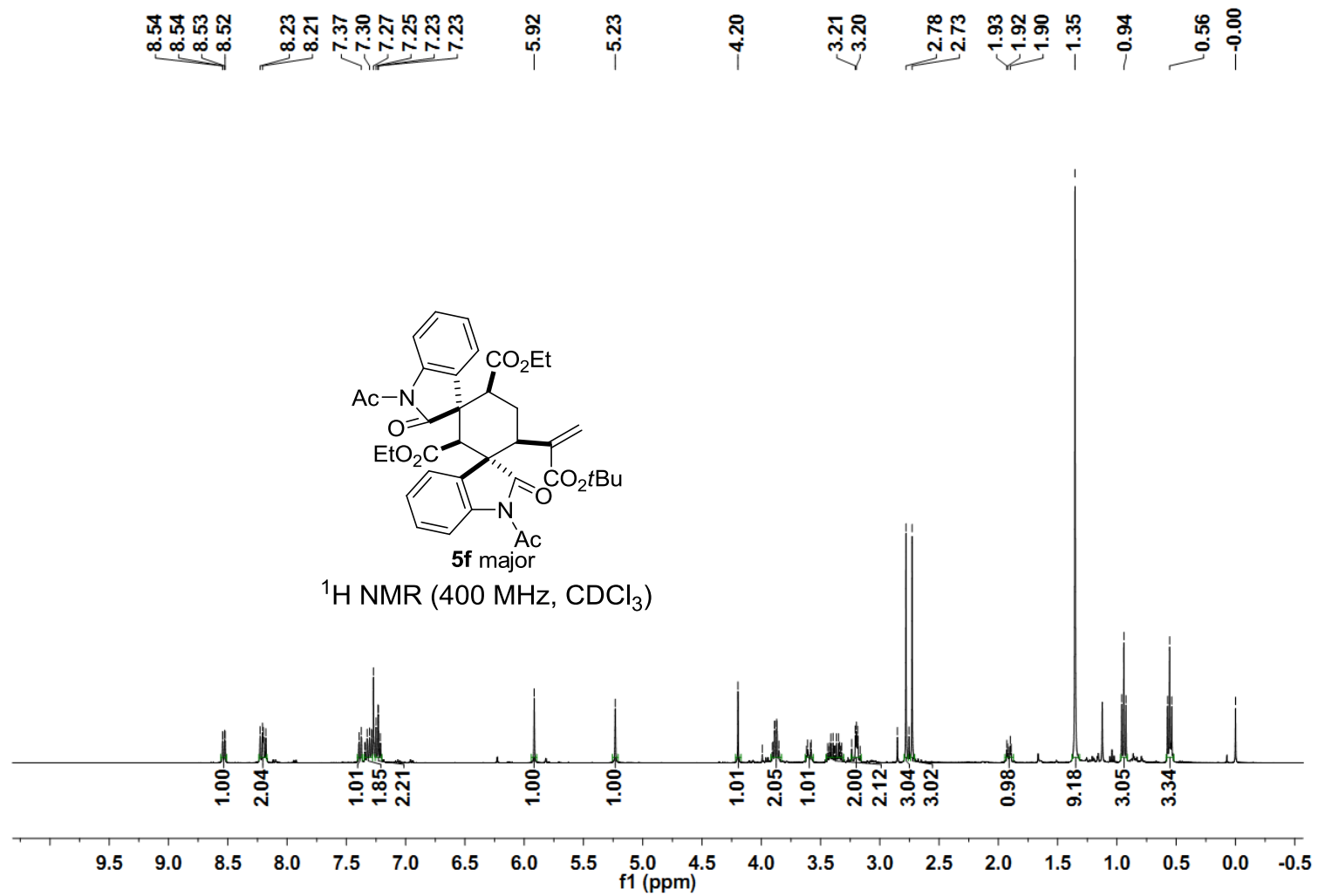


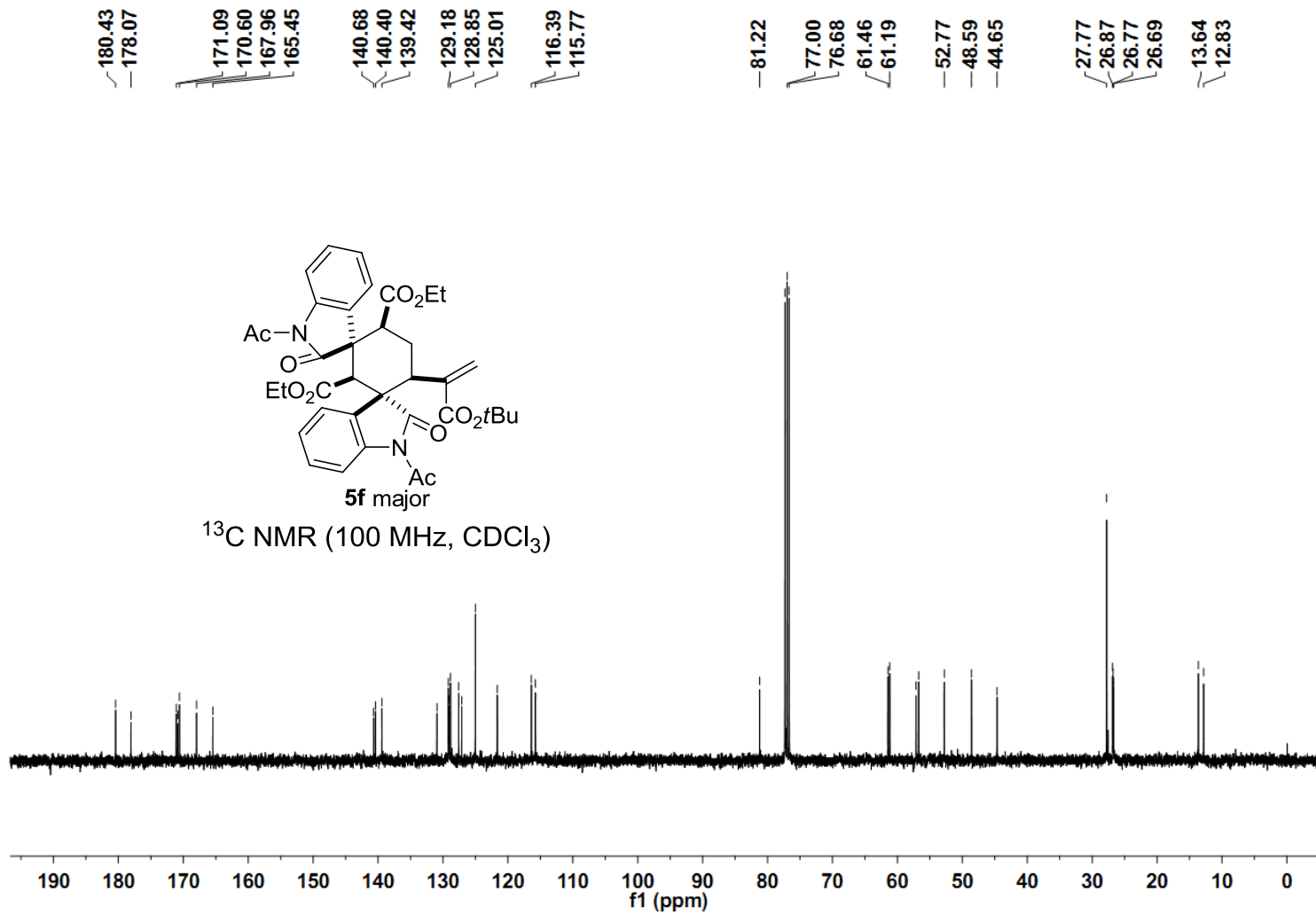


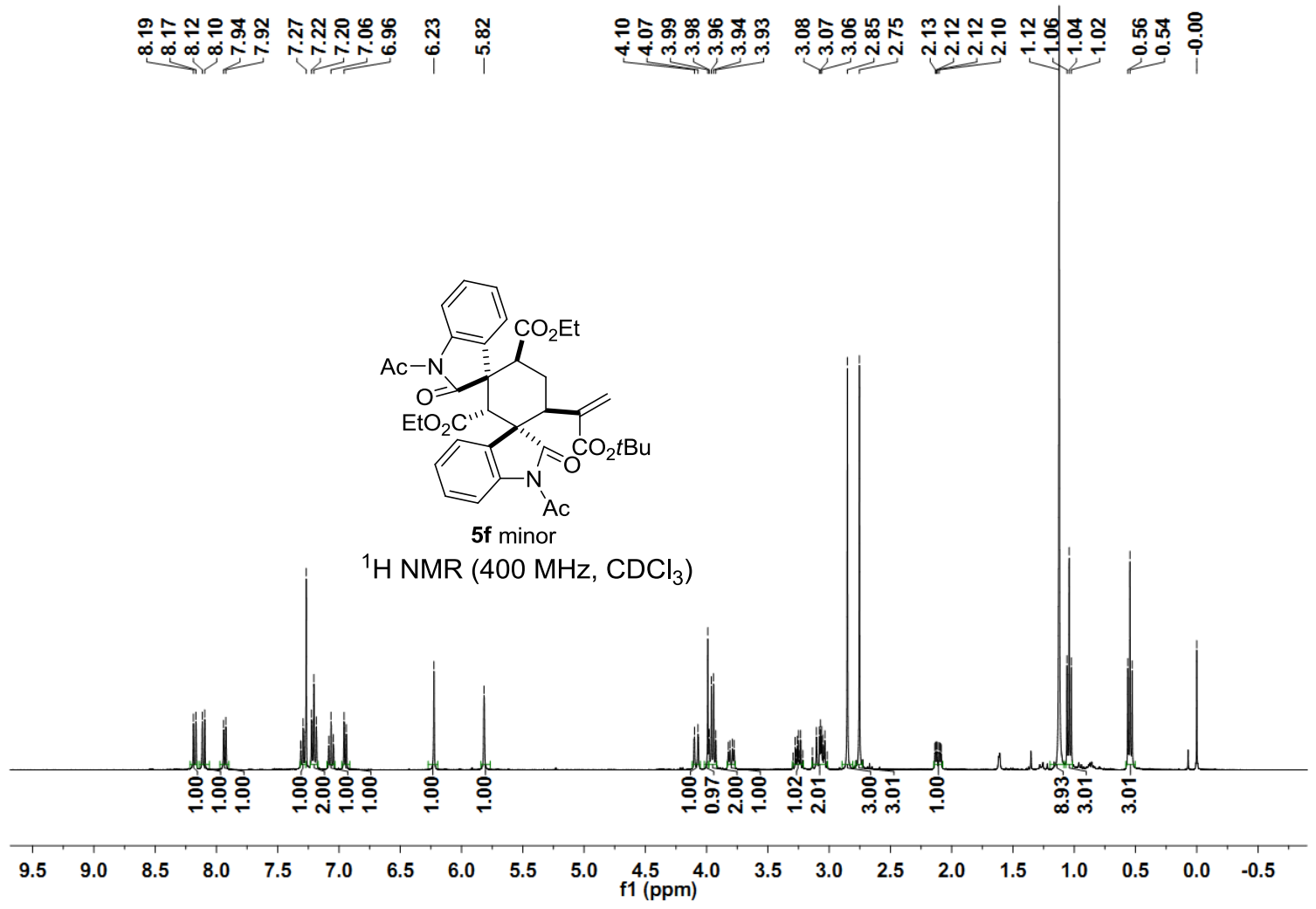


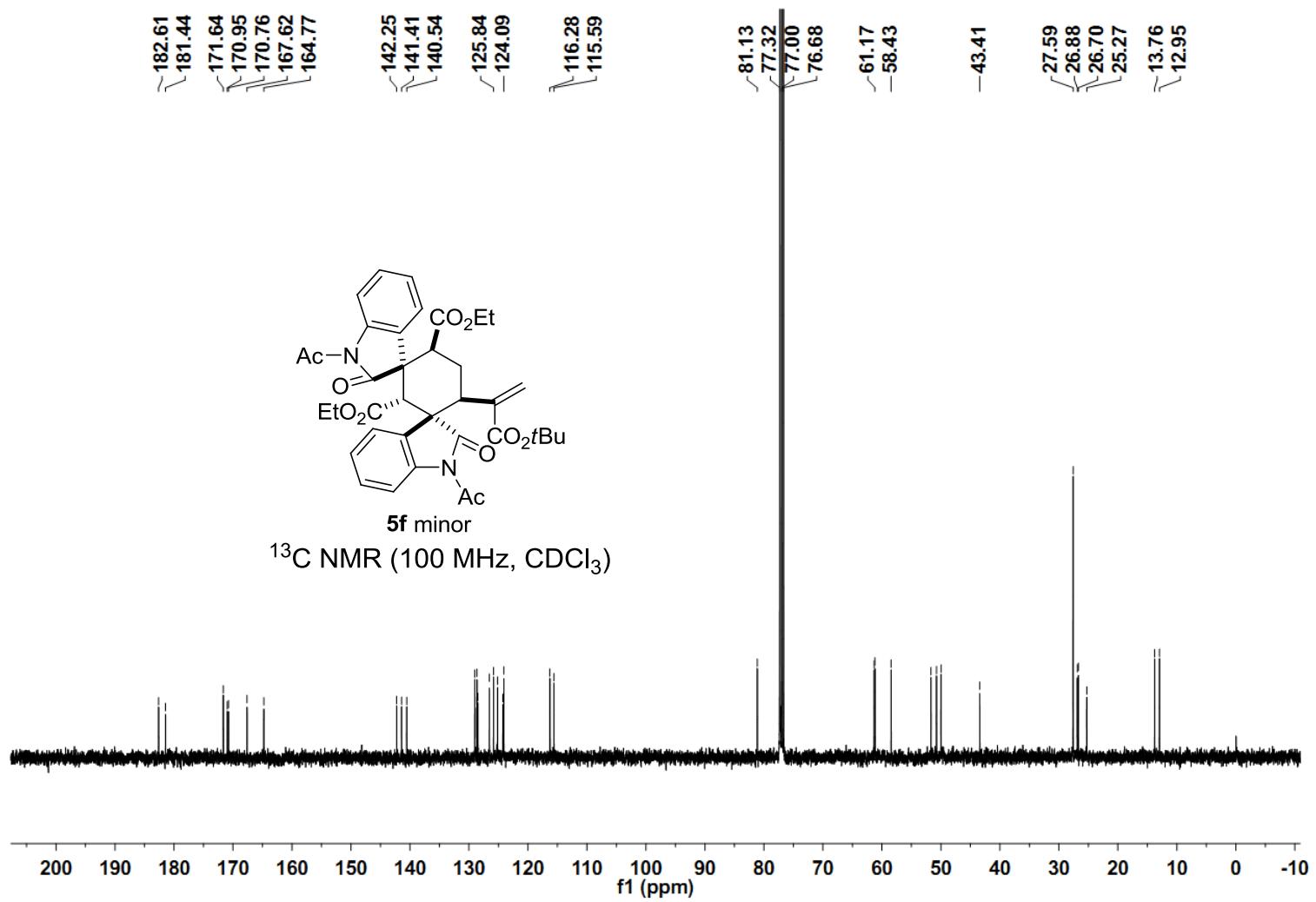


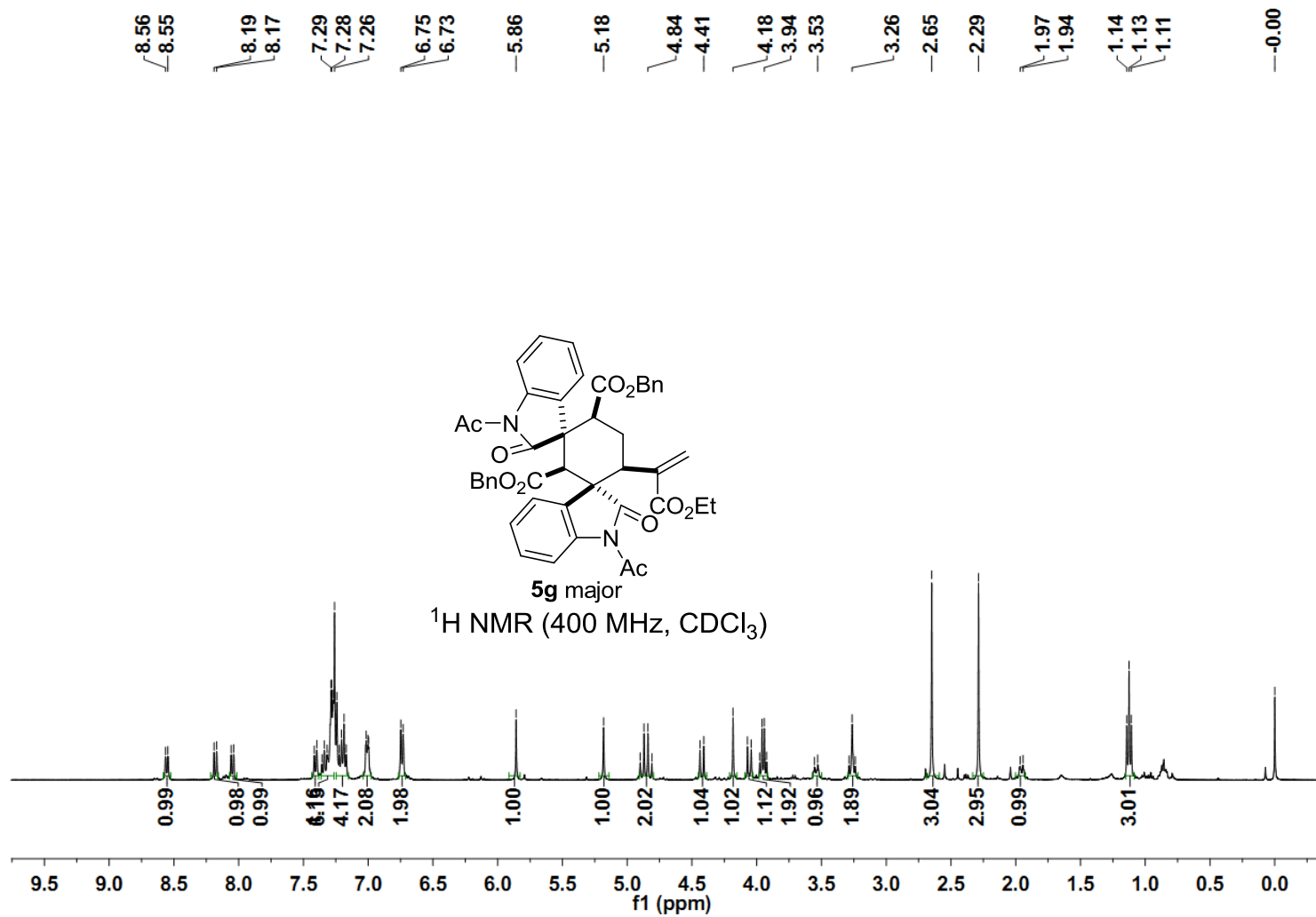


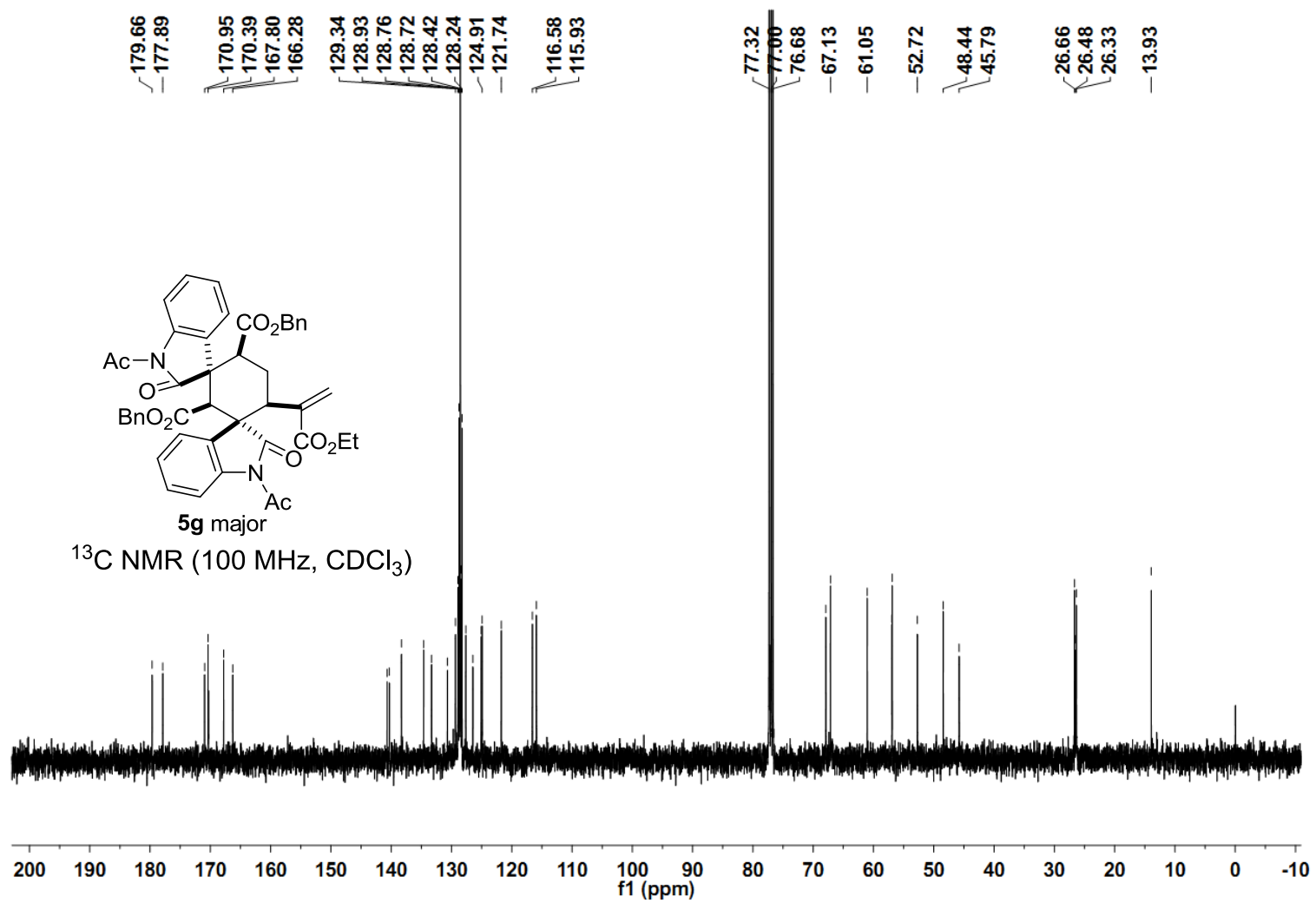


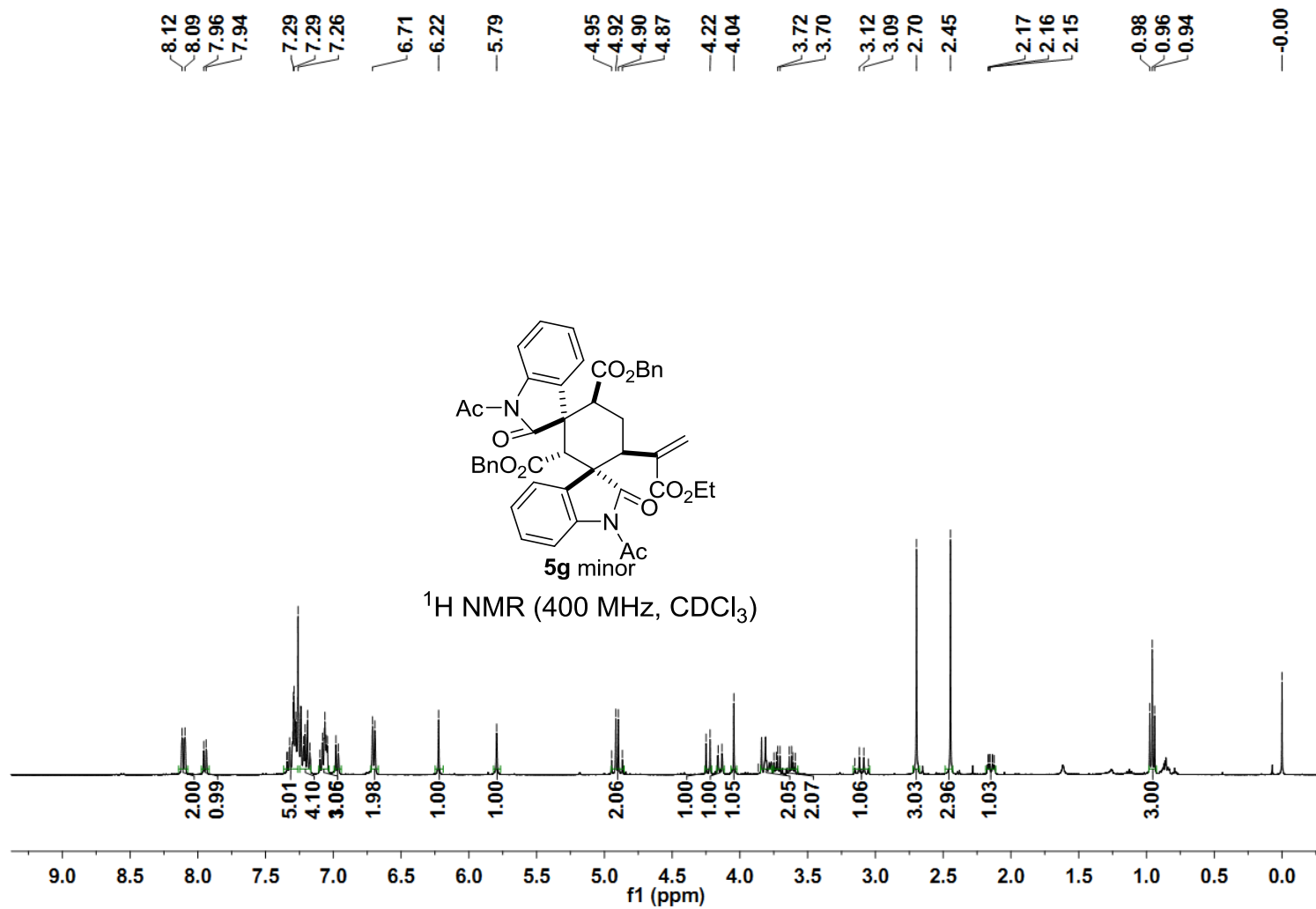


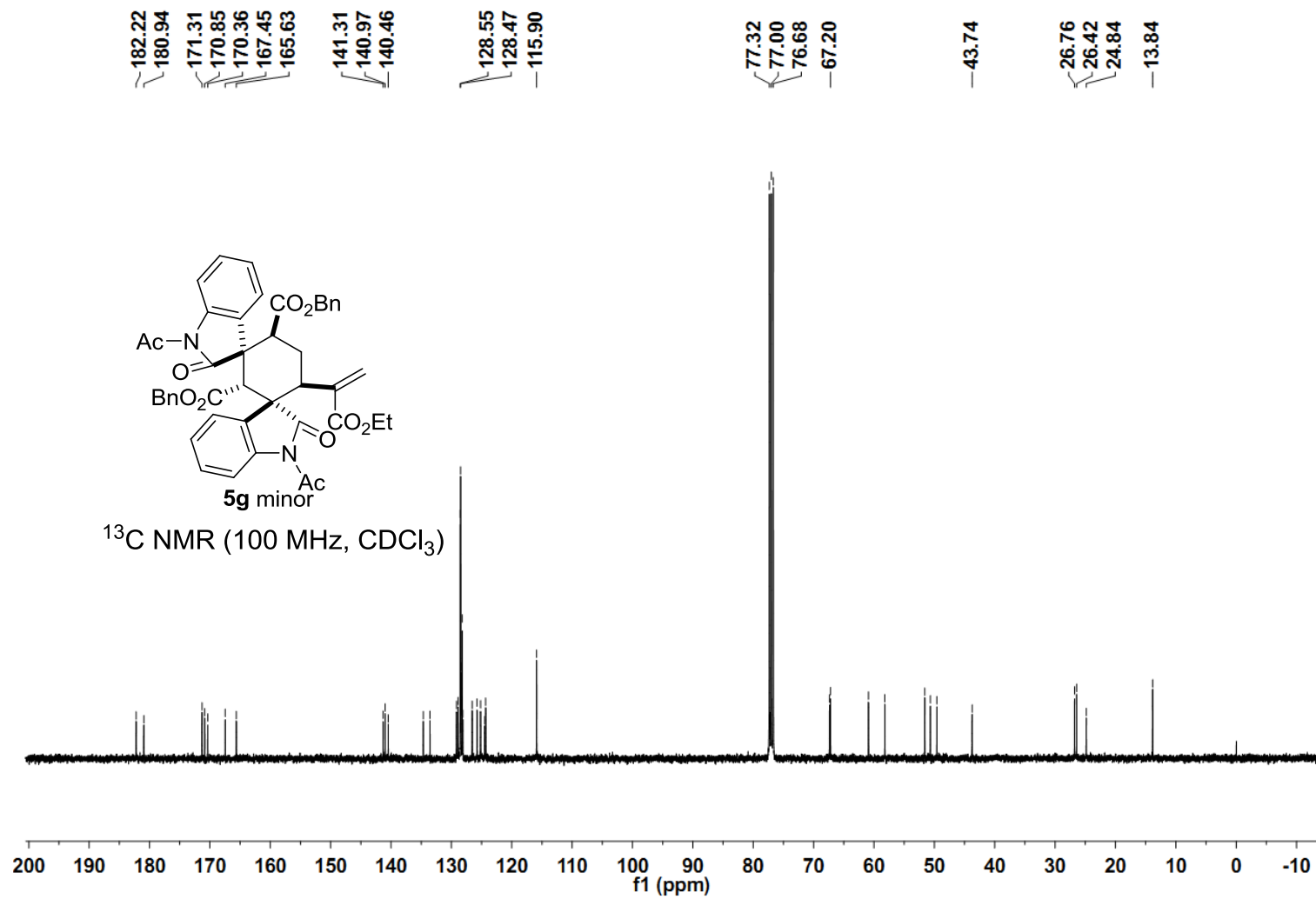


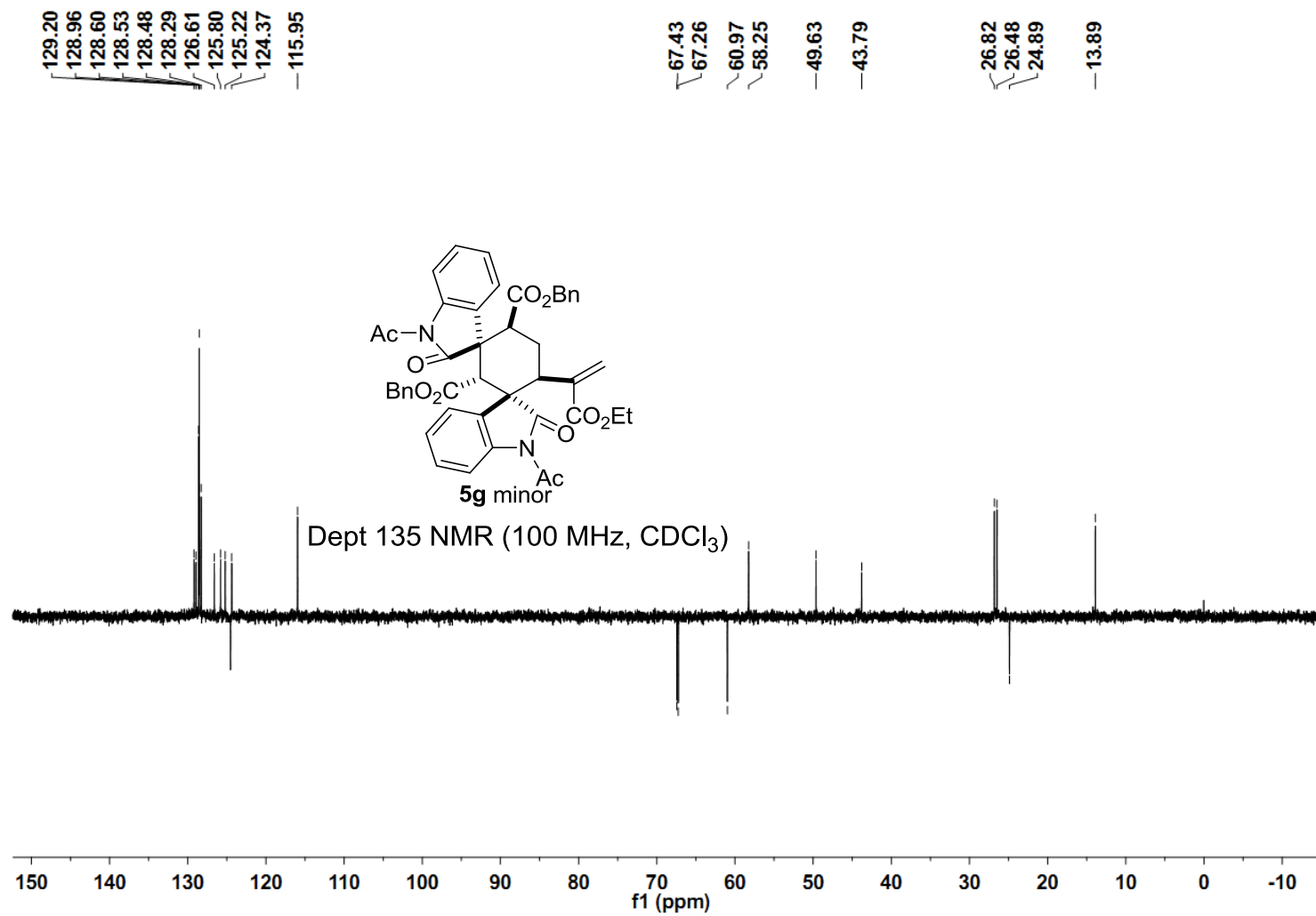


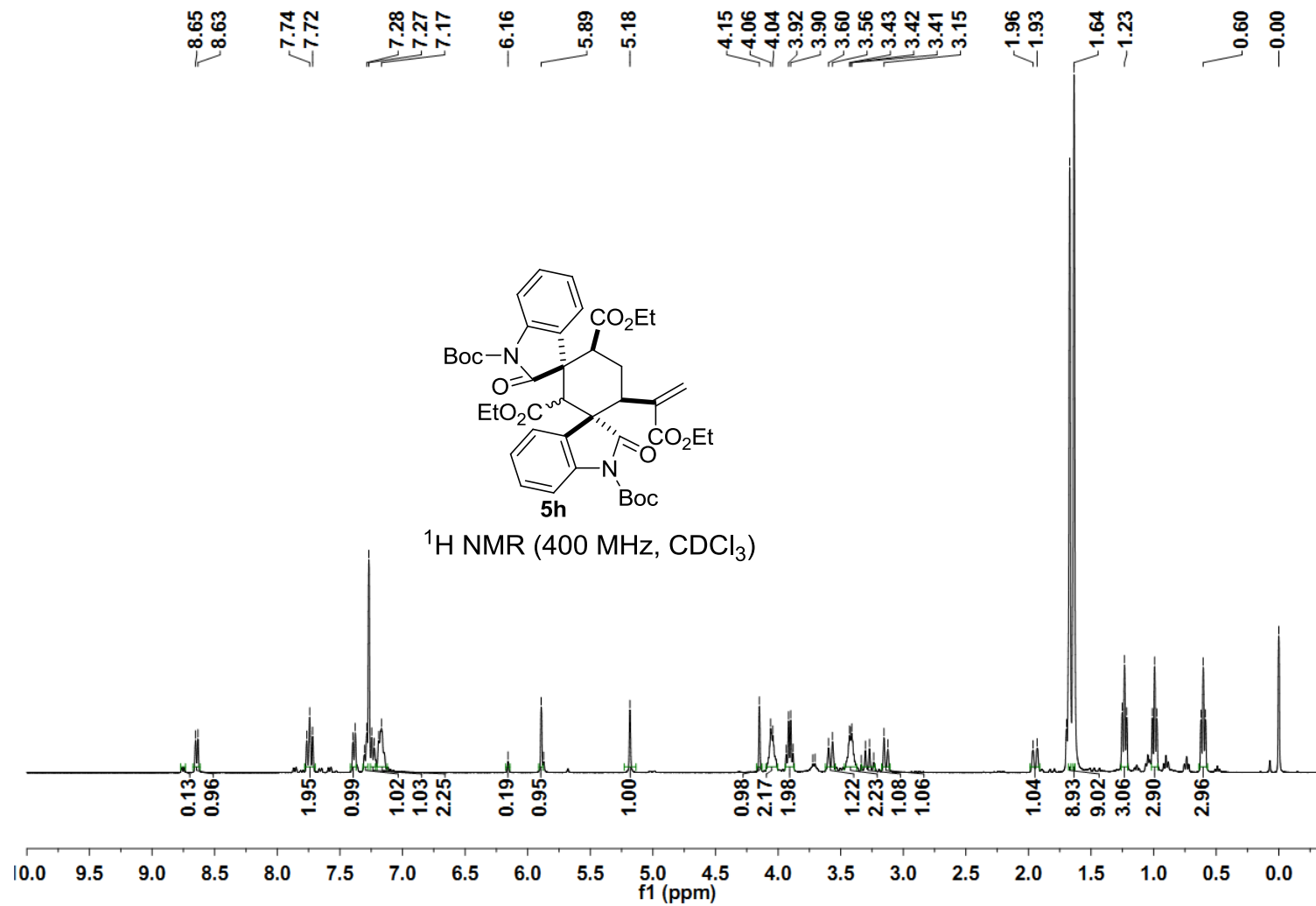




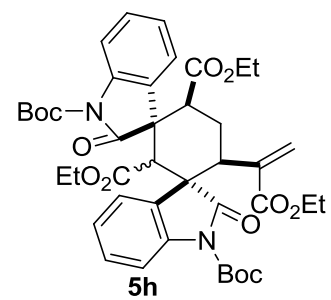




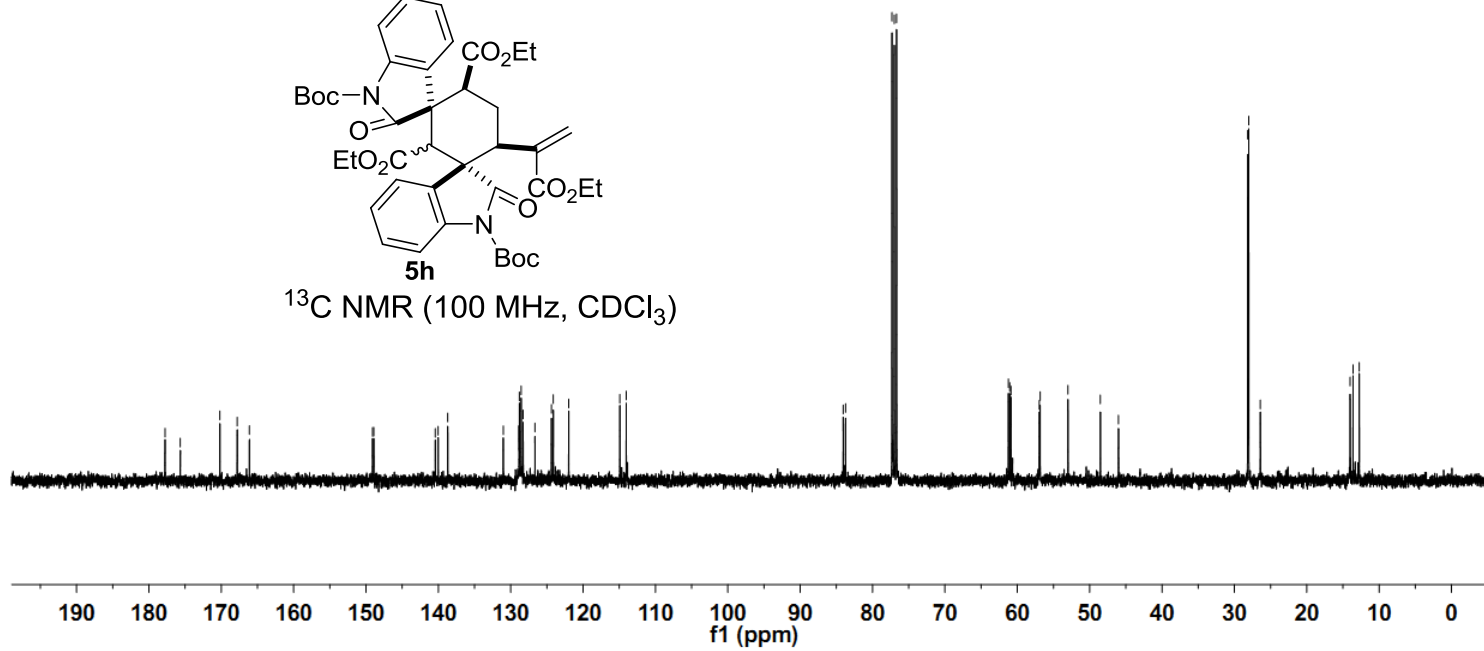


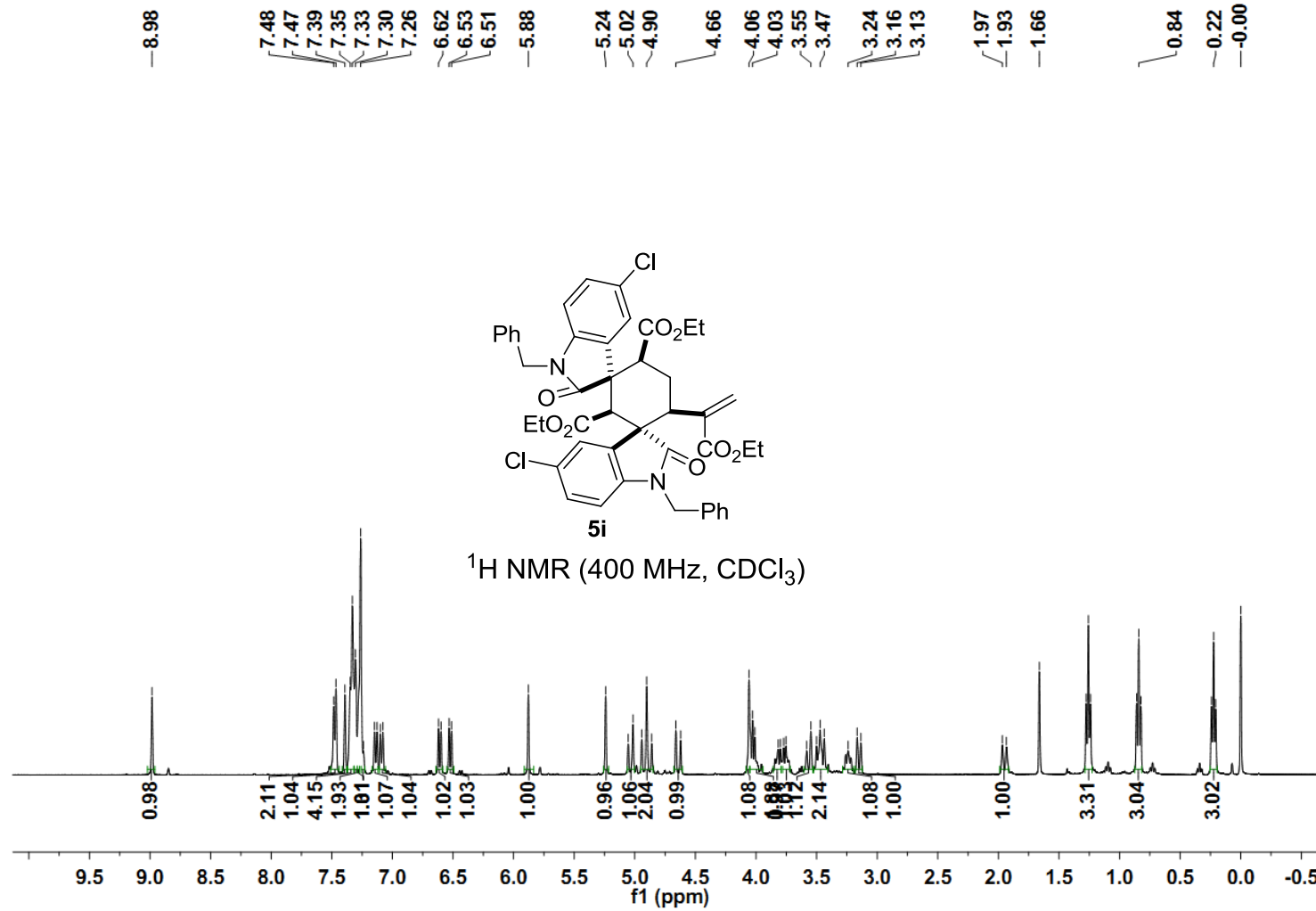


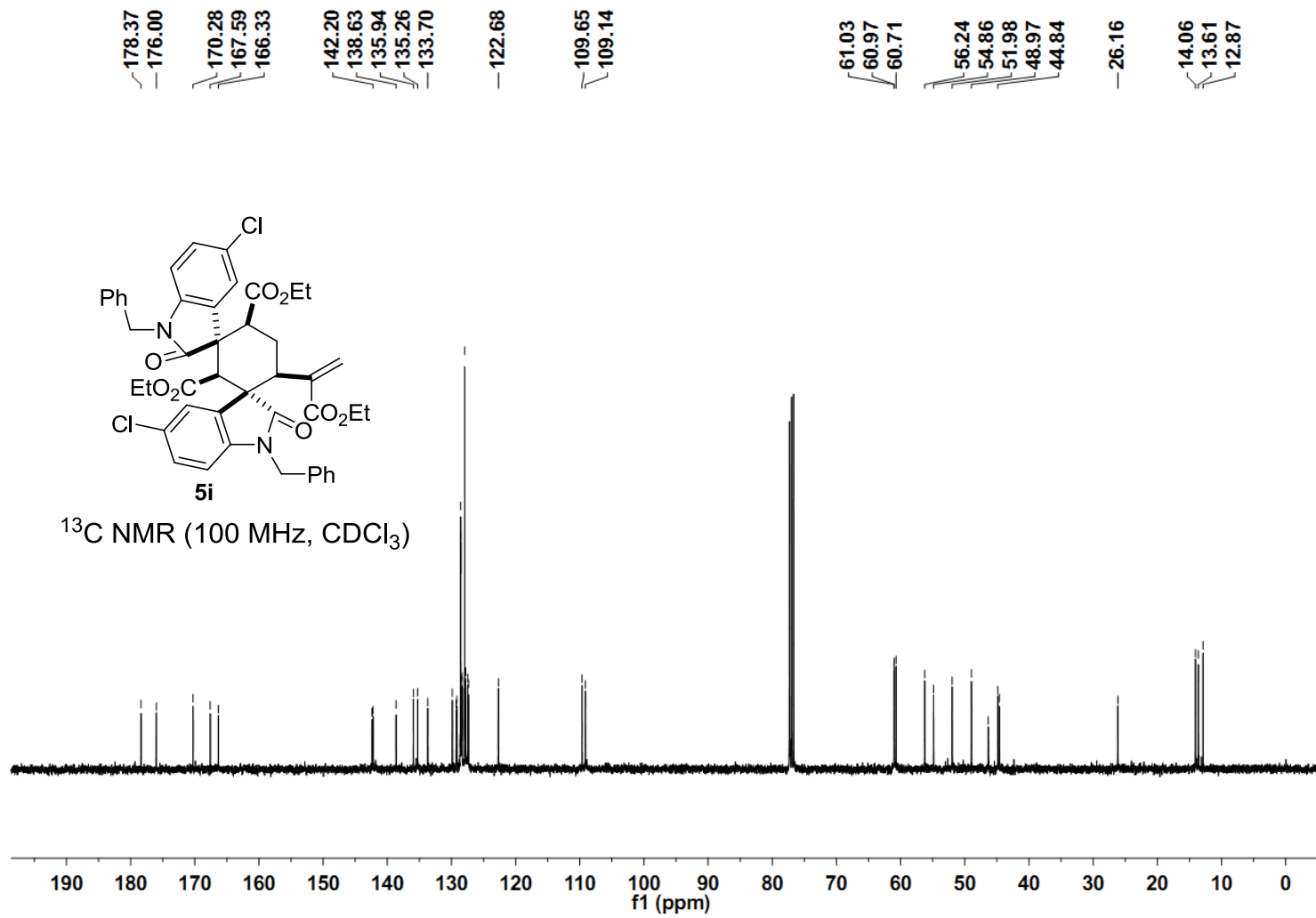
177.77
175.66
170.20
167.80
166.11
149.09
148.93
140.03
138.71
128.80
128.56
124.12
114.92
114.04
84.06
83.73
77.32
76.68
61.23
61.04
53.00
48.52
46.02
28.15
28.02
26.41
14.01
13.60
12.75

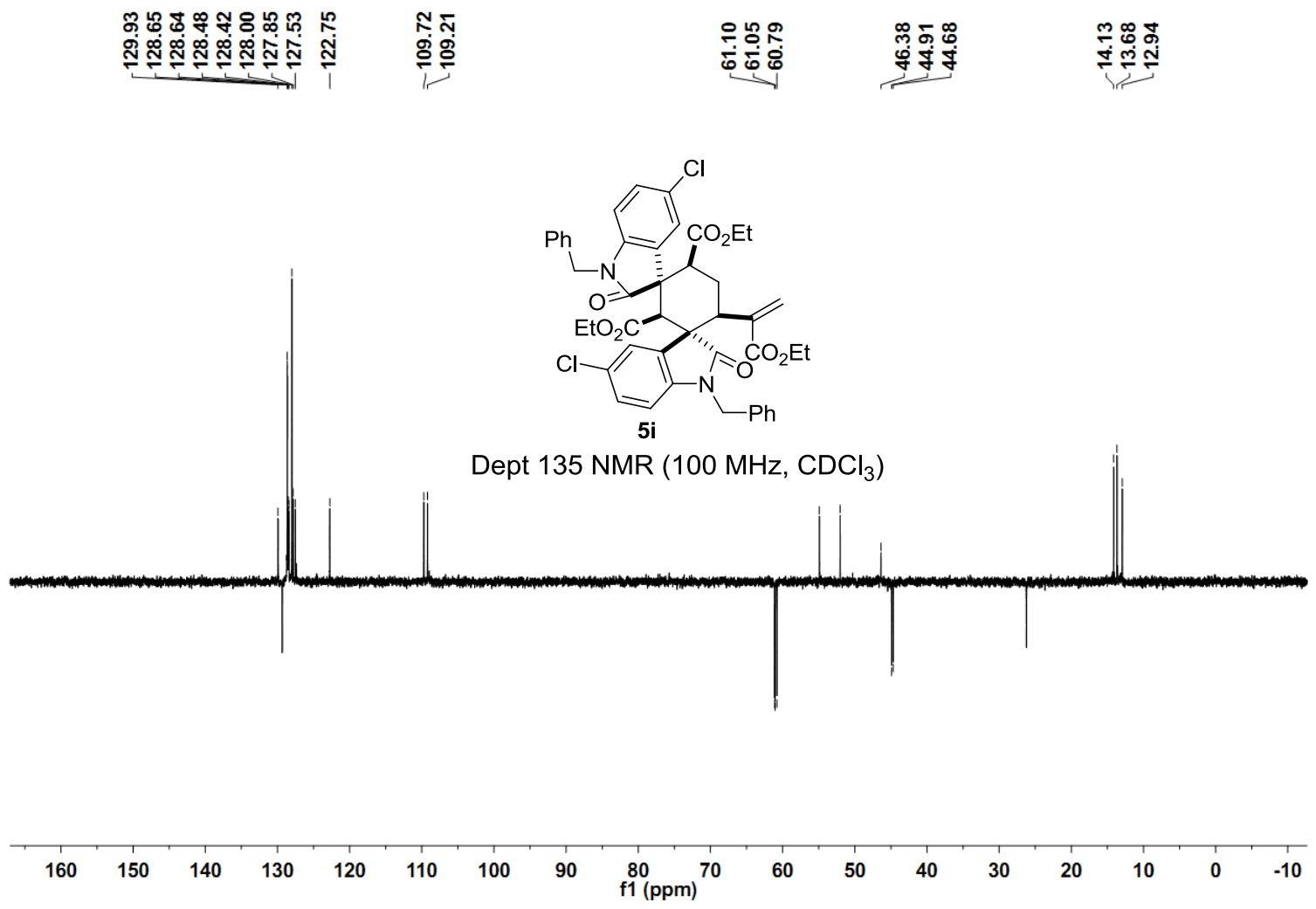


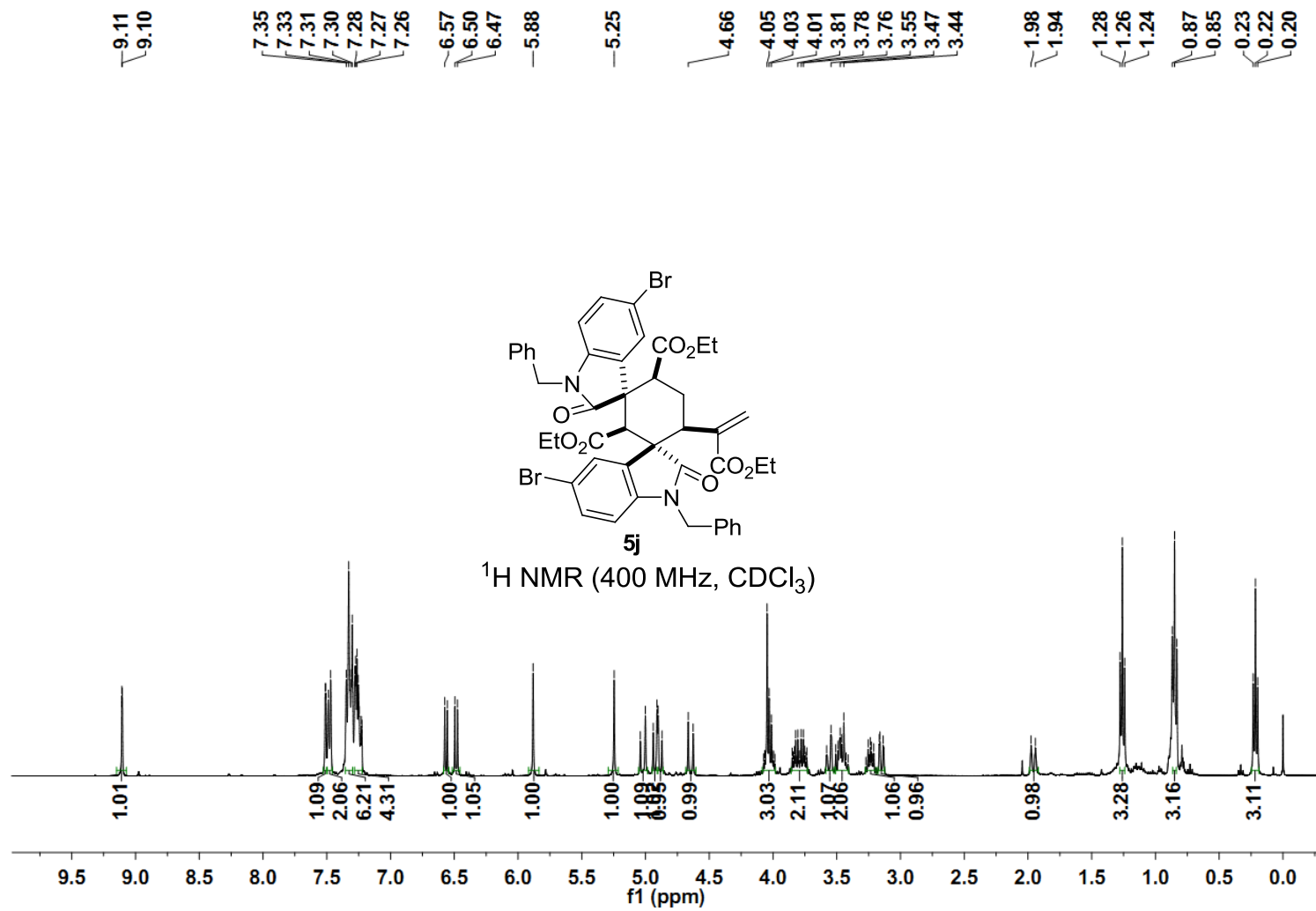
¹³C NMR (100 MHz, CDCl₃)



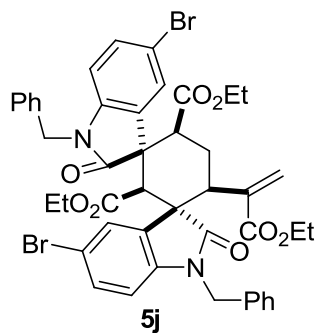




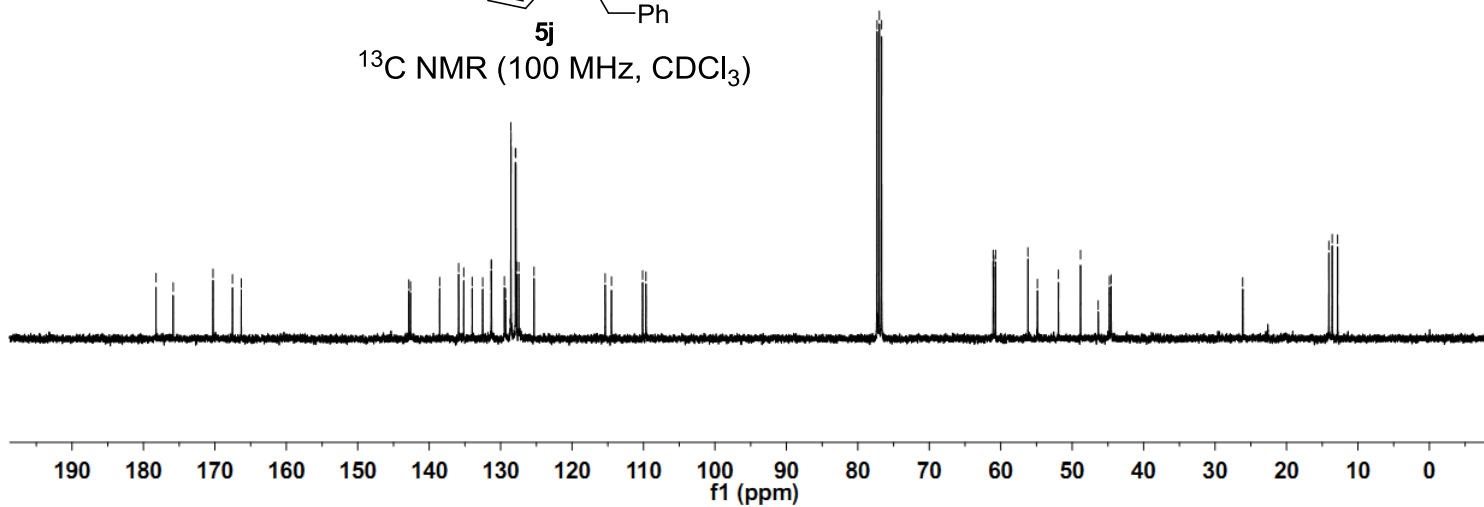


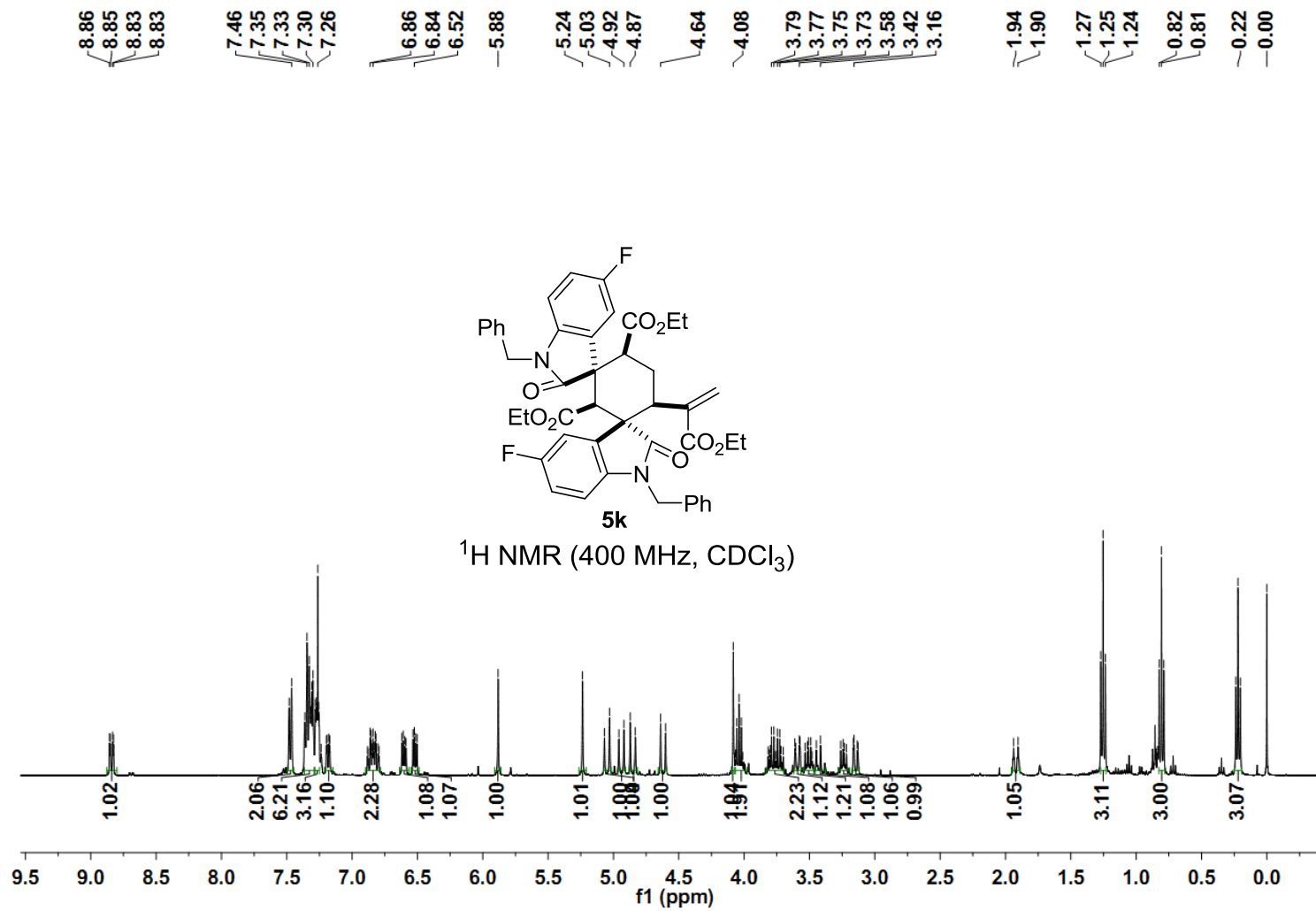


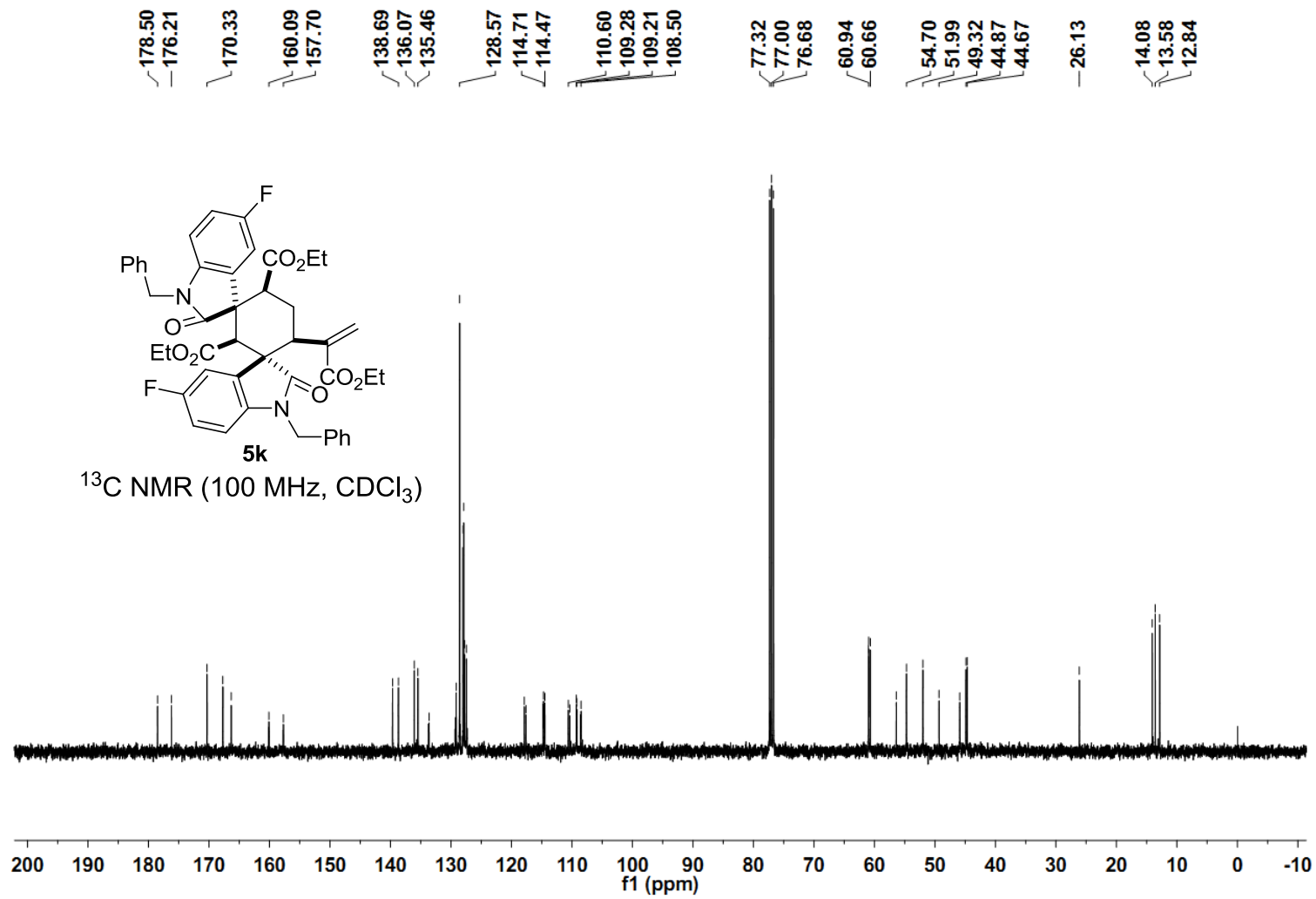
178.25
 175.85
 170.26
 167.54
 166.31
 142.85
 142.62
 128.57
 128.56
 127.93
 127.88
 115.37
 110.13
 109.67
 77.32
 77.00
 76.68
 61.05
 60.72
 56.18
 51.92
 48.83
 44.79
 44.55
 26.12
 14.07
 13.60
 12.85

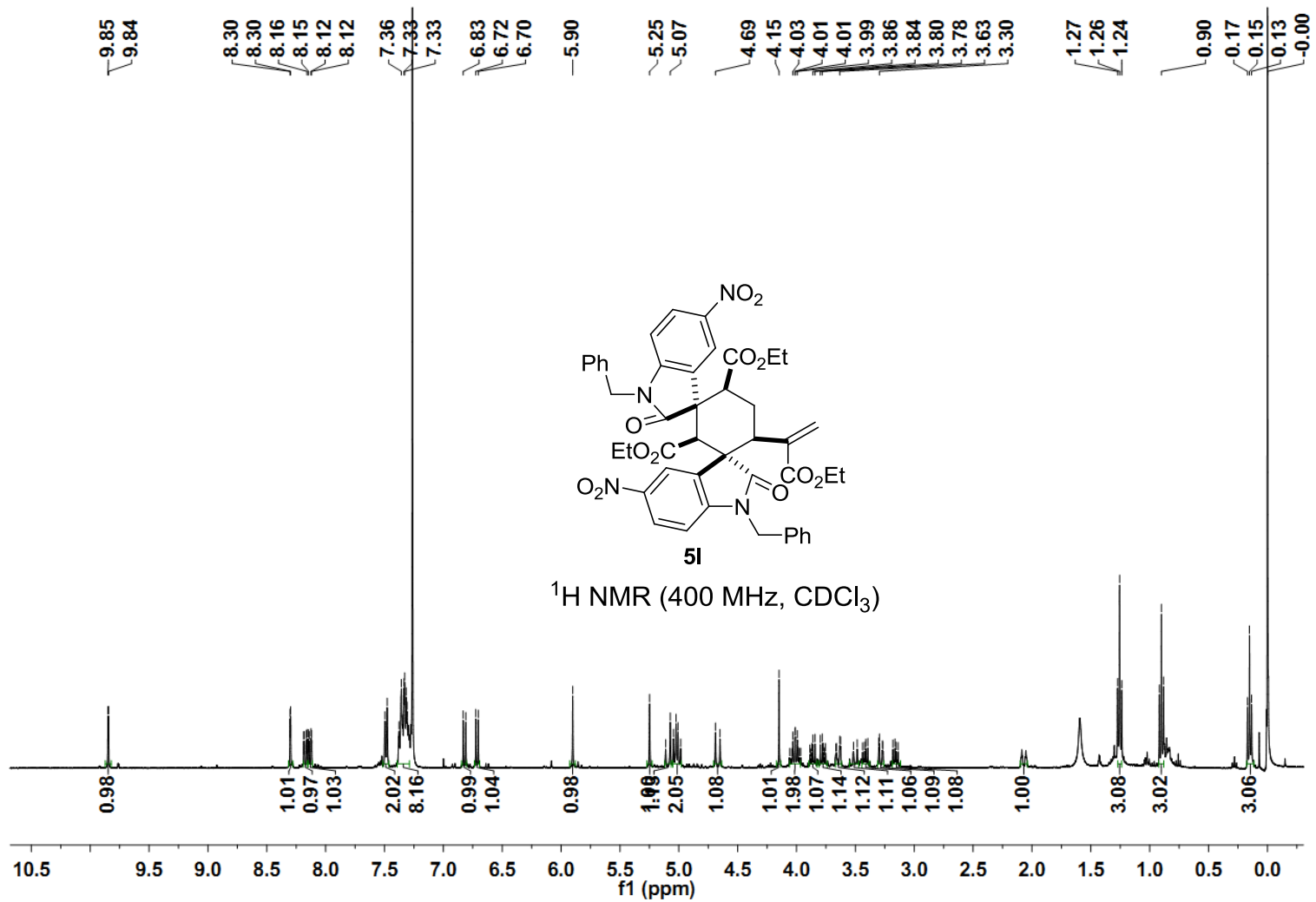


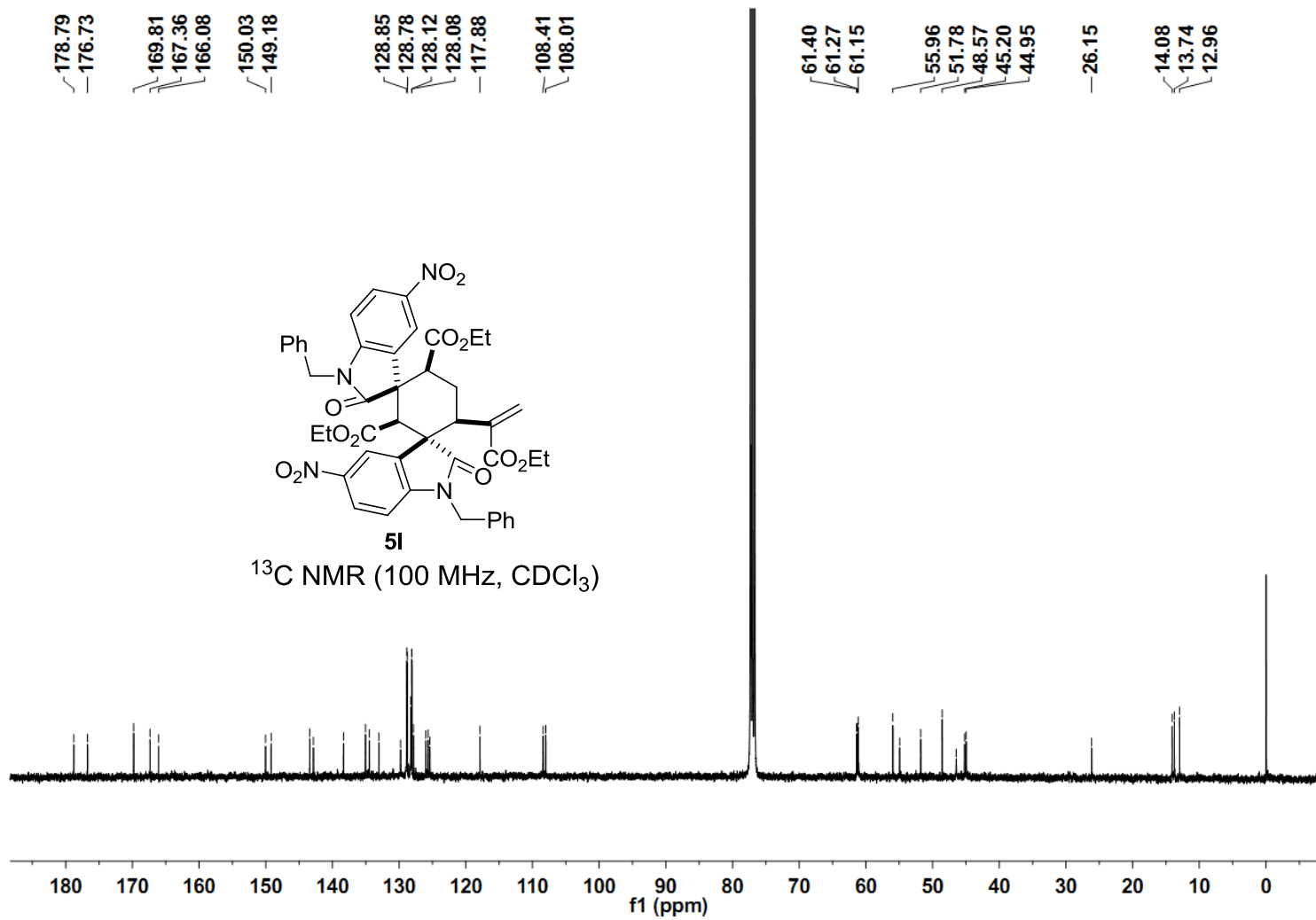
^{13}C NMR (100 MHz, CDCl_3)



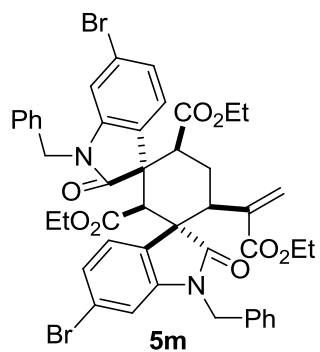




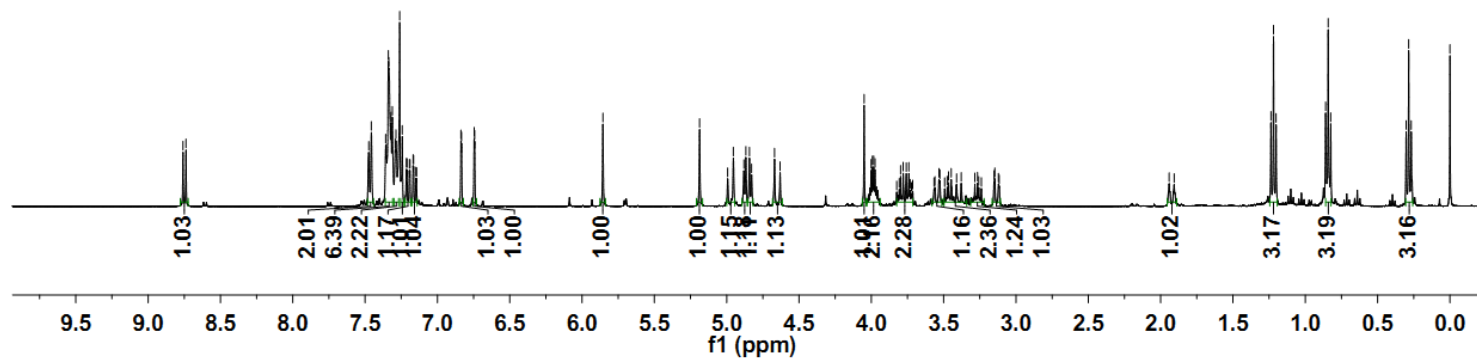


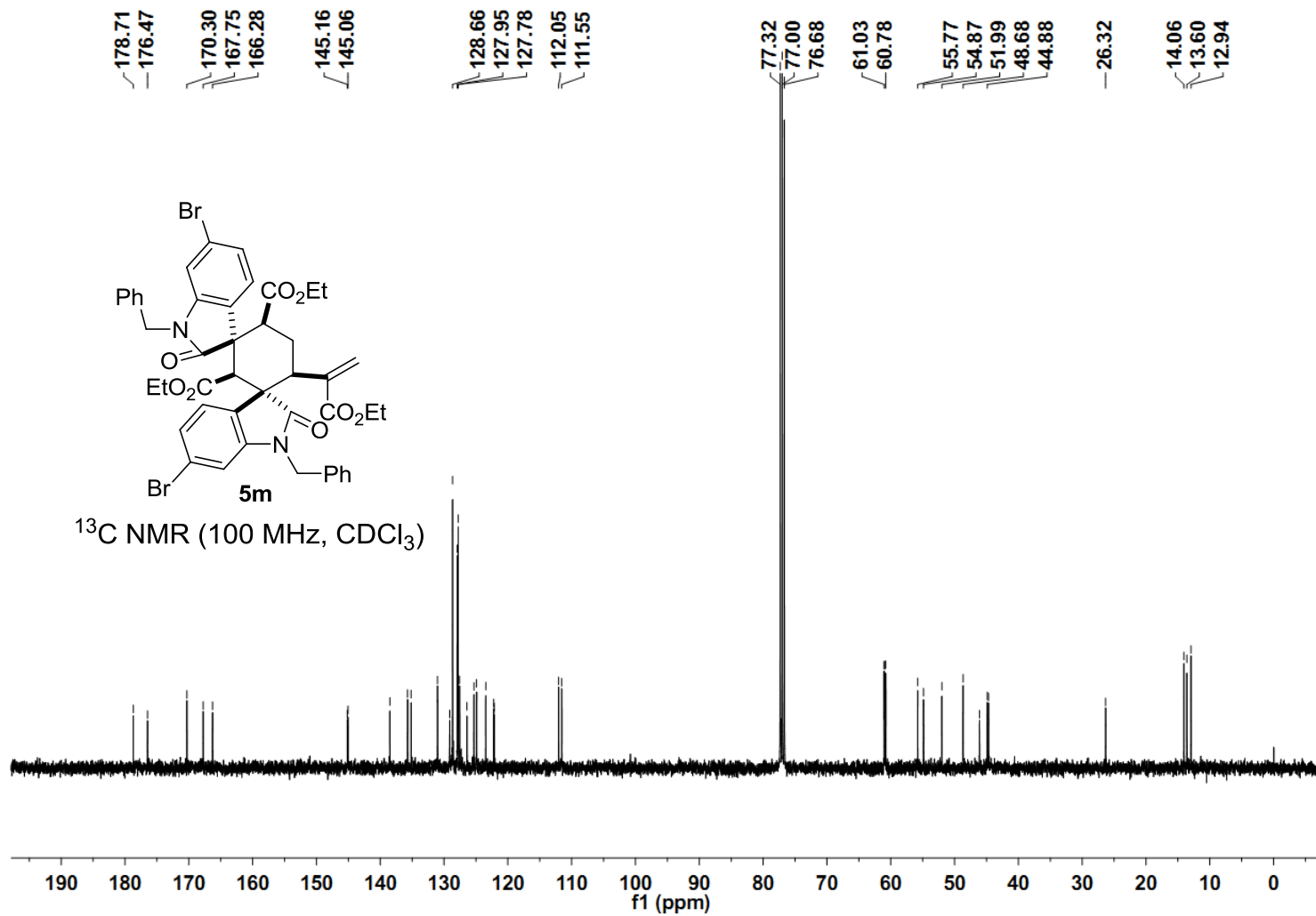


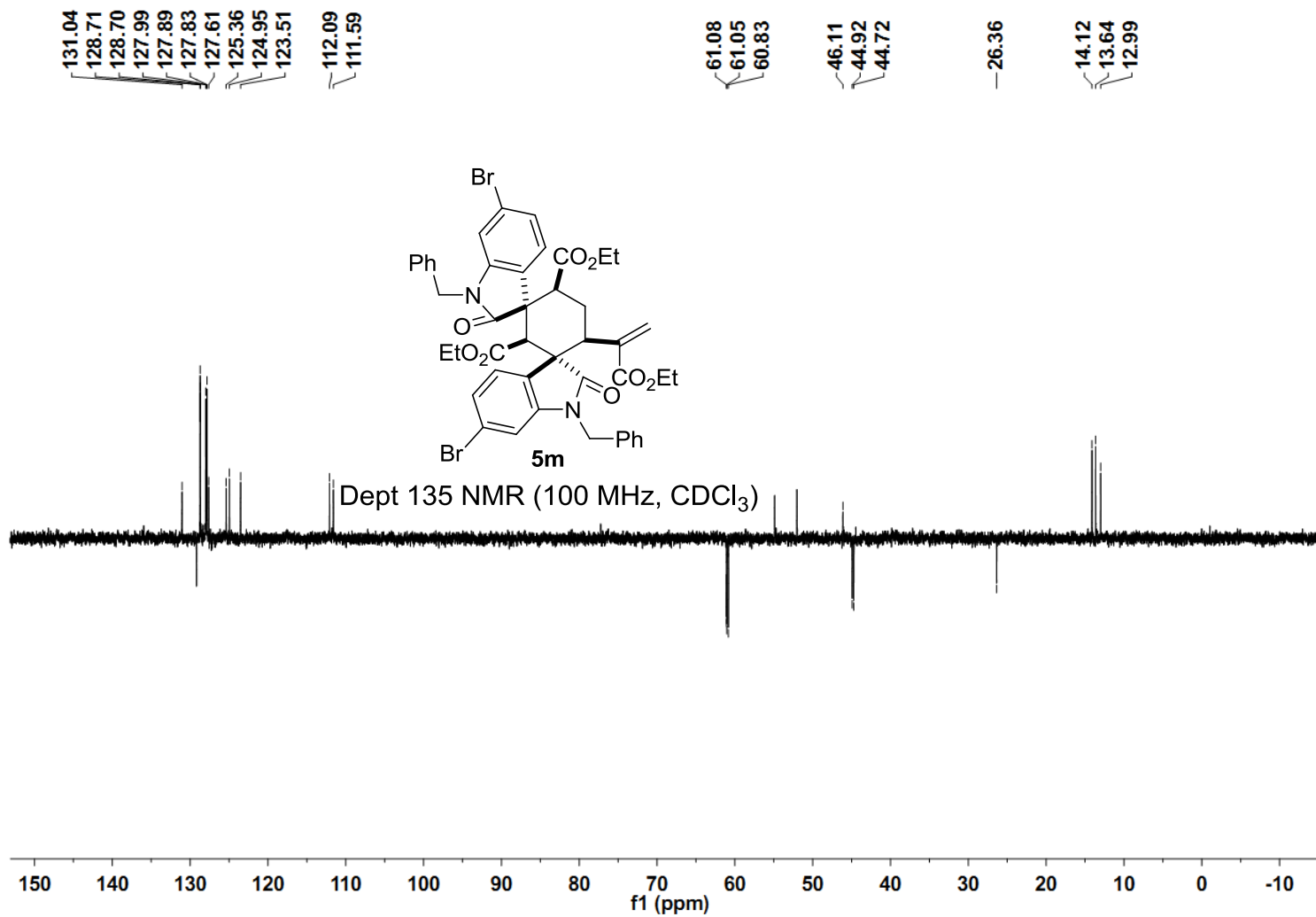
8.76
8.74
7.34
7.34
7.32
7.31
7.26
6.84
6.75
5.86
5.19
4.95
4.87
4.84
4.05
4.00
3.99
3.98
3.97
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3.78
3.76
3.74
1.94
1.91
1.24
1.22
0.84
0.28
0.00



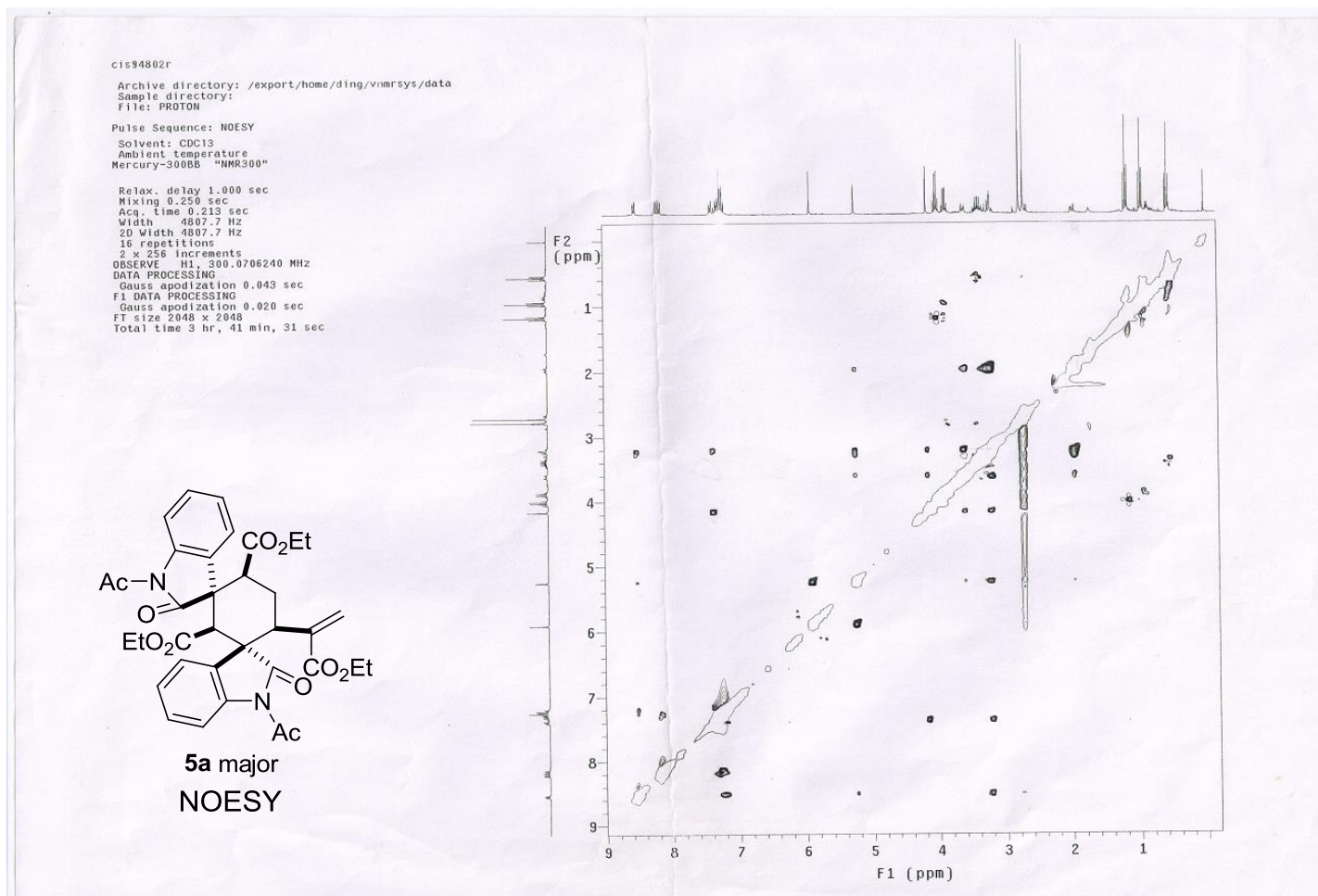
¹H NMR (400 MHz, CDCl₃)







III. NOESY Spectra for Compound 5a

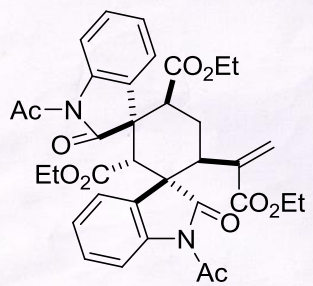


cis948016

Archive directory: /export/home/ding/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: NOESY
Solvent: CDCl3
Ambient temperature
Mercury-300BB "NMR300"

Relax. delay 1.000 sec
Mixing 0.250 sec
Acq. time 0.213 sec
Width 4807.7 Hz
2D Width 4807.7 Hz
16 repetitions
2 x 200 increments
OBSERVE H1, 300.0706261 MHz
DATA PROCESSING
Gauss apodization 0.047 sec
F1 DATA PROCESSING
Gauss apodization 0.020 sec
FT size 2048 x 2048
Total time 2 hr, 52 min, 39 sec



5a minor
NOESY

