Supporting Information

Efficient Synthesis of Spirooxindolyl Oxazol-2(5H)-ones via Palladium(II)-Catalyzed Addition of Arylboronic Acids to Nitriles

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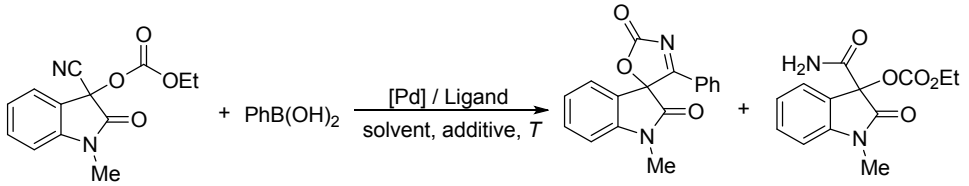
I. General Information

All reactions were carried out under inert atmospheric condition unless otherwise noted, and solvents were dried according to established procedures. Reactions were monitored by thin layer chromatography (TLC) visualizing with ultraviolet light (UV), $KMnO_4$, p-anisaldehyde stain, and phosphomolybdic acid (PMA) stain; column chromatography purifications were carried out using silica gel. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a 300 or 500 MHz spectrometer in $CDCl_3$, and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on 125 MHz spectrometer in $CDCl_3$ unless otherwise noted. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane (TMS) and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26 ppm). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane (TMS) and are referenced to the carbon resonances of the solvent residual peak ($CDCl_3$ = δ 77.16 ppm). NMR data are presented as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant in Hertz (Hz), integration. Mass spectra were recorded on the Bruker MicrOTOF Q II.

II. Reaction Condition Screening

Table S1. Optimized reaction conditions of Pd (II) complex catalyzed reaction [a]



1a **2a** **3a** **4a**

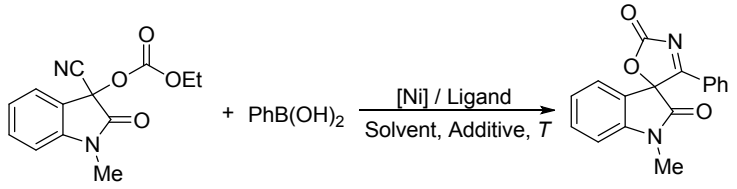
Entry	Cat.	Ligand	Solvent	Additive	*t* (h)	Yield **3a** (%)[b]	Yield **4b** (%)[b]
1	$Pd(OAc)_2$	**L1**	THF	AcOH (10 eq.)	24	77	8
2	$Pd(OAc)_2$	**L1**	THF	Cs_2CO_3 (0.2 eq.)	24	58	nd
3	$Pd(OAc)_2$	**L1**	THF	CSA (10 eq.)	24	24	17
4	$Pd(OAc)_2$	**L1**	THF	TsOH (10 eq.)	24	32	24
5	$Pd(OAc)_2$	**L1**	THF	MsOH (10 eq.)	24	26	20
6	$Pd(OAc)_2$	**L1**	1,4-dioxane	AcOH (10 eq.)	24	55	24
7	$Pd(OAc)_2$	**L1**	CH_3CN	AcOH (10 eq.)	24	40	23
8	$Pd(OAc)_2$	**L1**	DCE	AcOH (10 eq.)	24	48	13
9	$Pd(OAc)_2$	**L1**	Toluene	AcOH (10 eq.)	24	27	32
10	$Pd(OAc)_2$	**L1**	MeOH	AcOH (10 eq.)	24	49	17
11	$Pd(OAc)_2$	**L1**	NMA	AcOH (10 eq.)	24	47	30
12	Pd(OAc)2	**L1**	DMF	AcOH (10 eq.)	24	79	nd
13	$Pd(OAc)_2$	**L1**	DMA	AcOH (10 eq.)	24	78	nd
14	$Pd(OAc)_2$	**L1**	DMSO	AcOH (10 eq.)	24	71	nd
15	$Pd(OAc)_2$	**L1**	NMP	AcOH (10 eq.)	24	82	nd
16	$Pd(TFA)_2$	**L1**	NMP	AcOH (10 eq.)	24	77	nd
17	$Pd(acac)_2$	**L1**	NMP	AcOH (10 eq.)	24	88	nd
18	$Pd(acac)_2$	**L2**	NMP	AcOH (10 eq.)	24	86	nd
19	$Pd(acac)_2$	**L3**	NMP	AcOH (10 eq.)	24	73	nd
20	$Pd(acac)_2$	**L4**	NMP	AcOH (10 eq.)	24	88	nd
21	$Pd(acac)_2$	**L1**	NMP	AcOH (10 eq.)	24	70	nd
22	$Pd(acac)_2$	**L1**	NMP	AcOH (10 eq.)/KF (2 eq.)	24	45	nd
23	$Pd(acac)_2$	**L1**	NMP	AcOH (10 eq.)/CsF (2 eq.)	24	34	nd
24	$Pd(acac)_2$	**L1**	NMP	AcOH (5 eq.)	24	87	nd
25	$Pd(acac)_2$	**L1**	NMP	AcOH (5 eq.)	30	91	nd
26	$Pd(OAc)_2$	**L1**	NMP	AcOH (5 eq.)	36	92	nd
27[c]	$Pd(OAc)_2$	**L1**	NMP	AcOH (5 eq.)	36	91	nd
28[c]	$Pd(OAc)_2$	**L1**	NMP	-	36	83	nd
29	-	**L1**	NMP	-	24	nd	nd

30	$Pd(OAc)_2$	-	NMP	-	24	nd	nd
31	-	-	NMP	AcOH (5 eq.)	24	nd	nd
32	$Pd(OAc)_2$	**L1**	NMP	AcOH (5 eq.)	36	79	nd
33 [c,d]	$Pd(OAc)_2$	**L1**	NMP	AcOH (5 eq.)	35	76	nd

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), catalyst (10 mol %), ligand (12 mol %) and HOAc (10 equiv.) in solvent (1 mL) at 80 °C. [b] Isolated yield. [c] $Pd(OAc)_2$ (5 mol %) and bpy (6 mol %) were used. [d] Run at 100 °C.

$\mathbf{L_1}$: 2,2'-Bipyridine; $\mathbf{L_2}$: 4,4'-Dimethyl-2,2'-Bipyridine; $\mathbf{L_3}$: 5,5'-Dimethyl-2,2'-Bipyridine; $\mathbf{L_4}$: 1,10-Phenanthroline

Table SI-2. Optimized reaction conditions of Ni (II) complex catalyzed reaction [a]



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

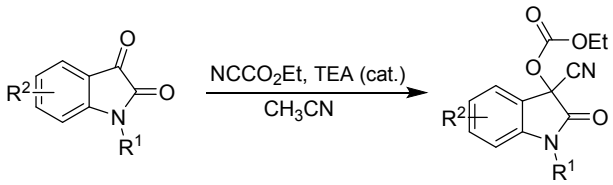
Entry	Cat.	Ligand	Solvent	Additive	*t* (h)	Yield (%) [b]
1	$Ni(acac)_2$	**L1**	Toluene	Cs_2CO_3	12	45
2 [c]	$Ni(acac)_2$	**L1**	Toluene	HOAc	12	< 1
3	$Ni(acac)_2$	**L1**	THF	Cs_2CO_3	12	15
4	$Ni(acac)_2$	**L1**	DMF	Cs_2CO_3	12	26
5	$Ni(acac)_2$	**L1**	DMSO	Cs_2CO_3	12	28
6	$Ni(acac)_2$	**L1**	DME	Cs_2CO_3	12	15
7	$Ni(acac)_2$	**L1**	NMP	Cs_2CO_3	12	27
8	$Ni(acac)_2$	**L1**	MTBE	Cs_2CO_3	12	56
9	$Ni(dppe)Cl_2$	none	MTBE	Cs_2CO_3	12	38
10	$Ni(PPh_3)_2Cl_2$	none	MTBE	Cs_2CO_3	12	25
11	$Ni(OAc)_2 \cdot 4H_2O$	**L1**	MTBE	Cs_2CO_3	12	33
12	$NiCl_2 \cdot 6H_2O$	**L1**	MTBE	Cs_2CO_3	12	17
13	$NiCl_2$(DME)	**L1**	MTBE	Cs_2CO_3	12	15
14	$Ni(ClO_4)_2$	**L1**	MTBE	Cs_2CO_3	12	51
15	$Ni(acac)_2$	-	MTBE	Cs_2CO_3	12	30
16	$Ni(acac)_2$	**L2**	MTBE	Cs_2CO_3	12	67 (66) [d]
17	$Ni(acac)_2$	**L3**	MTBE	Cs_2CO_3	12	50
18	$Ni(acac)_2$	**L4**	MTBE	Cs_2CO_3	12	60
19 [e]	$NiCl_2 \cdot 6H_2O$	**L2**	MTBE	Cs_2CO_3	12	53
20	$Ni(acac)_2$	**L2**	MTBE	Na_2CO_3	12	28
21	$Ni(acac)_2$	**L2**	MTBE	K_2CO_3	12	59

22	$Ni(acac)_2$	**L2**	MTBE	CsF	12	34
23[f]	$Ni(acac)_2$	**L2**	MTBE	Cs_2CO_3	12	45
24[g]	$Ni(acac)_2$	**L2**	MTBE	Cs_2CO_3	12	35

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), Ni (II) catalyst (10 mol %), ligand (12 mol %) and Cs_2CO_3 (0.2 equiv.) in solvent (C = 0.25 *M*) at 110 °C for 12 h. [b] Isolated yield. [c] HOAc (10 equiv.) instead of Cs_2CO_3 (0.2 equiv.). [d] Yield in parenthesis for 24 h. [e] AgOTf (0.2 eq.) was added. [f] Run at 120 °C. [g] Run at 130 °C.

L_1: 2,2'-Bipyridine; **L_2**: 4,4'-Dimethyl-2,2'-Bipyridine; **L_3**: 5,5'-Dimethyl-2,2'-Bipyridine; **L_4**: 1,10-Phenanthroline

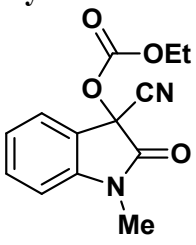
III. Preparation of Substrates



Compound **2a-2p** were prepared according to the known procedure.[1-3]

To a solution of isatin derivative **S1** (1.0 mmol) in CH_3CN (2.0 mL) was added Et_3N(0.3 mmol) and ethyl cyanoformate (1.2 mmol), and the mixture was stirred at room temperature overnight. Upon completion, the reaction mixture was then concentrated under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (60-90℃) to afford the cyano-ethoxycarbonylation product **1**. Compounds **4a-4d** were prepared according to the similar procedure.

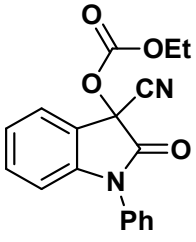
3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1a

Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 8.2 Hz, 1H), 7.52-7.47 (m, 1H), 7.20-7.16 (m, 1H), 6.92 (d, *J* = 7.9 Hz, 1H), 4.24-4.15 (m, 2H), 3.28 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.90, 151.75, 144.37, 132.98, 125.95, 124.31, 121.73, 113.08, 109.65, 71.24, 66.09, 27.37, 14.12.

3-cyano-2-oxo-1-phenylindolin-3-yl ethyl carbonate

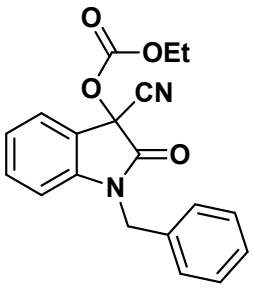


1b

Yellow solid, mp: 110.3-110.9 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.59-7.53 (m, 2H), 7.51-7.38 (m, 4H), 7.23-7.18 (m, 1H), 6.81 (d, *J* = 7.9 Hz, 1H), 4.31-4.11 (m, 2H), 1.31 (t, *J* = 7.3 Hz, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 165.47, 151.75, 144.90, 133.32, 132.85, 130.12,

129.31, 126.70, 125.80, 124.67, 121.41, 113.12, 110.93, 71.51, 66.18, 14.15. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_4^+$ ([M+H]$^+$): 323.1026, found 323.1027.

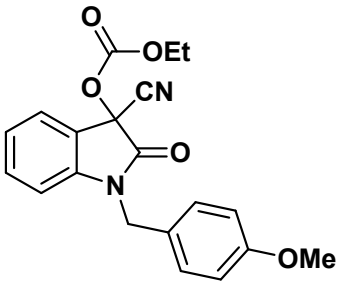
1-benzyl-3-cyano-2-oxoindolin-3-yl ethyl carbonate



1c

Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.4 Hz, 1H), 7.38-7.27 (m, 6H), 7.15-7.11 (m, 1H), 6.75 (d, *J* = 7.9 Hz, 1H), 5.01 (d, *J* = 15.8 Hz, 1H), 4.90 (d, *J* = 15.8 Hz, 1H), 4.27-4.14 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.15, 151.69, 143.41, 134.23, 132.78, 129.15, 128.24, 127.38, 125.83, 124.32, 121.74, 113.15, 110.79, 71.42, 66.16, 45.05, 14.13.

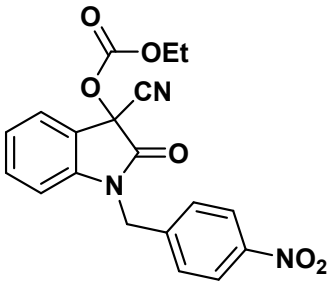
3-cyano-1-(4-methoxybenzyl)-2-oxoindolin-3-yl ethyl carbonate



1d

Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.5 Hz, 1H), 7.36-7.32 (m, 1H), 7.28 (d, *J* = 8.7 Hz, 2H), 7.14-7.10 (m, 1H), 6.89-6.86 (m, 2H), 6.77 (d, *J* = 7.9 Hz, 1H), 4.96 (d, J = 15.6 Hz, 1H), 4.82 (d, J = 15.6 Hz, 1H), 4.27-4.17 (m, 2H), 3.78 (s, 3H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.07, 159.55, 151.69, 143.44, 132.75, 128.86, 126.18, 125.80, 124.24, 121.76, 114.51, 113.17, 110.83, 71.44, 66.13, 55.39, 44.56, 14.13.

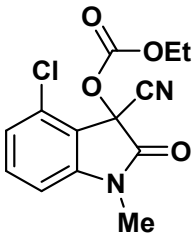
3-cyano-1-(4-nitrobenzyl)-2-oxoindolin-3-yl ethyl carbonate



1e

Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.7 Hz, 2H), 7.64 (d, *J* = 7.5 Hz, 1H), 7.55 (d, *J* = 8.7 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.67 (d, *J* = 7.9 Hz, 1H), 5.26 (d, *J* = 16.5 Hz, 1H), 4.88 (d, *J* = 16.5 Hz, 1H), 4.28-4.17 (m, 2H), 1.32 (t, *J* = 7.1 Hz, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 166.16, 151.46, 147.83, 142.56, 141.46, 132.78, 128.12, 125.66, 124.71, 124.27, 121.56, 112.78, 110.21, 71.28, 66.26, 44.20, 14.02.

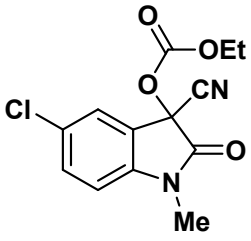
4-chloro-3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1f

Yellow solid, mp: 97.3-97.9 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (t, *J* = 8.1 Hz, 1H), 7.13 (d, *J* = 8.3 Hz, 1H), 6.84 (d, *J* = 7.9 Hz, 1H), 4.23-4.10 (m, 2H), 3.29 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.64, 151.25, 145.93, 133.89, 132.26, 124.82, 118.62, 111.58, 108.02, 71.09, 66.23, 27.67, 14.08. HRMS (ESI): calcd. for $C_{13}H_{12}ClN_2O_4^+$ ($[M+H]^+$): 295.0480, found 295.0481.

5-chloro-3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate

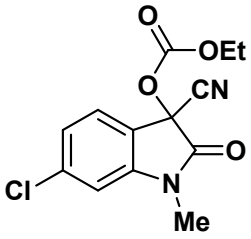


1g

Yellow solid, mp: 98.3-99.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 2.1 Hz, 1H), 7.47 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 4.25-4.18 (m, 2H), 3.28 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.48, 151.71, 142.88, 132.92, 129.76, 126.39, 123.04, 112.55,

110.72, 70.74, 66.38, 27.53, 14.12. HRMS (ESI): calcd. for $C_{13}H_{12}ClN_2O_4^+$ ([M+H]$^+$): 295.0480, found 295.0476.

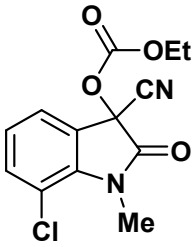
6-chloro-3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1h

Yellow solid, mp: 97.1-97.6 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.1 Hz, 1H), 7.18-7.15 (m, 1H), 6.93 (d, *J* = 1.8 Hz, 1H), 4.25-4.15 (m, 2H), 3.27 (s, 3H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.81, 151.73, 145.56, 139.20, 126.99, 124.25, 119.96, 112.64, 110.54, 70.61, 66.27, 27.50, 14.10. HRMS (ESI): calcd. for $C_{13}H_{11}ClN_2NaO_4^+$ ([M+Na]$^+$): 317.0300, found 317.0305.

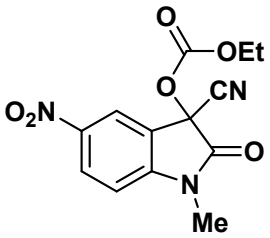
7-chloro-3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1i

Yellow solid, mp: 96.0-96.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55-7.51 (m, 1H), 7.44-7.39 (m, 1H), 7.10 (t, *J* = 7.9 Hz, 1H), 4.24-4.15 (m, 2H), 3.65 (s, 3H), 1.30 (t, *J* = 7.1 Hz, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 166.16, 151.41, 140.21, 135.08, 124.91, 124.10, 124.02, 116.99, 112.48, 70.57, 66.17, 30.83, 13.98. HRMS (ESI): calcd. for $C_{13}H_{12}ClN_2O_4^+$ ([M+H]$^+$): 295.0480, found 295.0478.

3-cyano-1-methyl-5-nitro-2-oxoindolin-3-yl ethyl carbonate

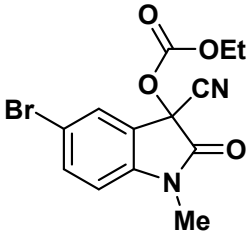


1j

Yellow solid, mp: 117.3-118.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.53 (d, *J* = 2.2 Hz, 1H), 8.46 (dd, *J* = 8.7, 2.2 Hz, 1H), 7.06 (d, *J* = 8.7 Hz, 1H), 4.27-4.17 (m, 2H), 3.37 (s, 3H), 1.32 (t, *J* = 7.1 Hz,

3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 165.97, 151.61, 149.51, 144.37, 129.50, 122.35, 121.77, 111.92, 109.67, 70.07, 66.75, 27.91, 14.06. HRMS (ESI): calcd. for $C_{13}H_{12}N_3O_6^+$ ([M+H]$^+$): 306.0721, found 306.0718.

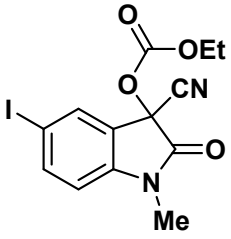
5-bromo-3-cyano-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1k

Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, J = 8.1 Hz, 1H), 7.18-7.15 (m, 1H), 6.93 (d, J = 1.8 Hz, 1H), 4.25-4.15 (m, 2H), 3.27 (s, 3H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.37, 151.68, 143.36, 135.82, 129.06, 123.32, 116.72, 112.55, 111.15, 70.63, 66.38, 27.50, 14.10.

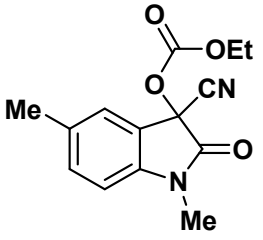
3-cyano-5-iodo-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1l

Yellow solid, mp: 115.9-116.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 1.7 Hz, 1H), 7.81 (dd, J = 8.3, 1.8 Hz, 1H), 6.70 (d, J = 8.3 Hz, 1H), 4.26-4.17 (m, 2H), 3.26 (s, 3H), 1.32 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.25, 151.67, 144.03, 141.74, 134.46, 123.59, 112.61, 111.62, 86.24, 70.49, 66.38, 27.46, 14.12. HRMS (ESI): calcd. for $C_{13}H_{12}IN_2O_4^+$ ([M+H]$^+$): 386.9836, found 386.9832.

3-cyano-1,5-dimethyl-2-oxoindolin-3-yl ethyl carbonate

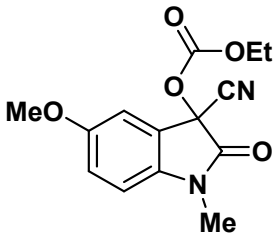


1m

Yellow solid, mp: 104.5-105.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.28 (d, J = 8.0 Hz, 1H), 6.80 (d, J = 7.9 Hz, 1H), 4.25-4.15 (m, 2H), 3.26 (s, 3H), 2.37 (s, 3H), 1.30 (t, J = 7.1 Hz,

3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 165.85, 151.76, 141.92, 134.26, 133.27, 126.53, 121.65, 113.24, 109.46, 71.41, 66.03, 27.37, 21.13, 14.13. HRMS (ESI): calcd. for $C_{14}H_{15}N_2O_3^+$ ([M+H]$^+$): 275.1026, found 275.1025.

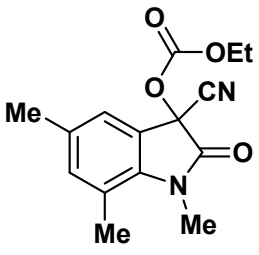
3-cyano-5-methoxy-1-methyl-2-oxoindolin-3-yl ethyl carbonate



1n

Yellow solid, mp: 109.2-109.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, *J* = 2.6 Hz, 1H), 7.02-6.99 (m, 1H), 6.84 (d, *J* = 8.6 Hz, 1H), 4.24-4.17 (m, 2H), 3.82 (s, 3H), 3.25 (s, 3H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.60, 156.98, 151.71, 137.50, 122.55, 117.68, 113.09, 112.57, 110.32, 71.47, 66.09, 56.06, 27.41, 14.10. HRMS (ESI): calcd. for $C_{14}H_{15}N_2O_5^+$ ([M+H]$^+$): 291.0975, found 291.0977.

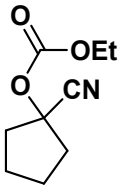
3-cyano-1,5,7-trimethyl-2-oxoindolin-3-yl ethyl carbonate



1o

Yellow solid, mp: 109.9-110.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (s, 1H), 7.01 (s, 1H), 4.24-4.13 (m, 2H), 3.51 (s, 3H), 2.53 (s, 3H), 2.30 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.72, 151.61, 139.53, 137.08, 134.07, 123.98, 122.32, 121.17, 113.34, 71.34, 65.95, 30.79, 20.80, 18.89, 14.13. HRMS (ESI): calcd. for $C_{17}H_{13}N_2O_3^+$ ([M+H]$^+$): 289.1183, found 289.1187.

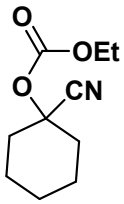
1-cyanocyclopentyl ethyl carbonate



4a

Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.26 (q, *J* = 7.1 Hz, 2H), 2.39-2.29(m, 4H), 1.88-1.82 (m, 4H), 1.34 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.02, 119.19, 78.82, 64.98, 39.05, 23.35, 14.22.

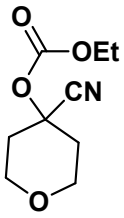
1-cyanocyclohexyl ethyl carbonate



4b

Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.26 (q, *J* = 7.1 Hz, 2H), 2.37-2.29 (m, 2H), 1.90-1.76 (m, 4H), 1.72-1.59 (m, 4H), 1.34 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.56, 118.41, 75.03, 64.87, 35.21, 24.52, 22.22, 14.27.

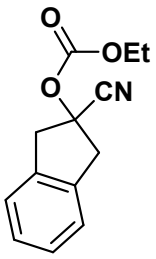
4-cyanotetrahydro-2H-pyran-4-yl ethyl carbonate



4c

Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.28 (q, *J* = 7.1 Hz, 2H), 3.99-3.92 (m, 2H), 3.78-3.70 (m, 2H), 2.43-2.3 (m, 2H), 2.13-2.05 (m, 2H), 1.35 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.37, 117.57, 72.08, 65.22, 63.69, 35.33, 14.22. HRMS (ESI): calcd. for $C_9H_{14}NO_4^+$ ($[M+H]^+$): 200.0917, found 200.0918.

2-cyano-2,3-dihydro-1H-inden-2-yl ethyl carbonate

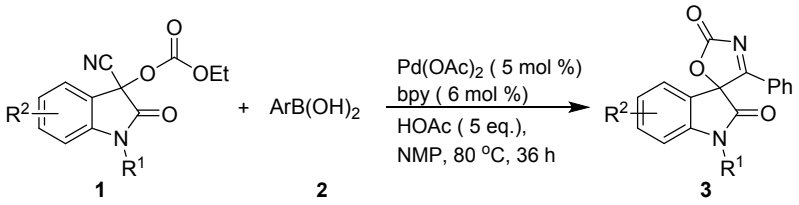


4d

White solid, mp: 81.9-82.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 4H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.76 (d, *J* = 17.1 Hz, 2H), 3.60 (d, *J* = 17.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.08, 137.06, 128.03, 124.78, 118.77, 77.69, 65.29, 45.24, 14.21. HRMS (ESI): calcd. for $C_{13}H_{14}NO_3^+$ ([M+H]$^+$): 232.0968, found 232.0969.

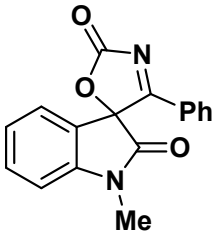
Ⅳ. General Procedure and Experimental Details of Pd-Catalyzed Addition/Cyclization Sequences

1) General Procedure and Experimental Details



Arylboronic acid **1** (3.0 equiv), substrate **2** (0.3 mmol), $Pd(OAc)_2$ (5 mol %), 2,2'-bipyridine (6 mol %), CH_3COOH (5.0 equiv) and NMP (0.2 *M*) were placed in a sealed tube under nitrogen atmosphere. The mixture was stirred vigorously at 80 °C for 36 hours. Upon completion, the mixture was cooled to room temperature, and then $NaHCO_3$ was added until no bubbles were generated. After the aqueous phase was extracted with ethyl acetate three times, the combined organic layers were washed with saturated $NaHCO_3$ and then brine, dried over anhydrous Na_2SO_4, filtered and finally concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (60-90℃) to afford the desired products **3**. Products **5a-5d** were prepared according to the similar procedure.

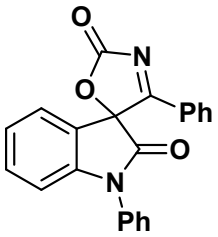
1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3aa

Yellow solid (79.8 mg, 91%), mp: 210.0-210.7 °C. 1H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.60-7.49 (m, 2H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.16-7.05 (m, 3H), 3.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.16, 168.14, 165.65, 144.36, 135.52, 132.80, 129.75, 129.56, 127.53, 125.29, 124.59, 122.80, 110.13, 88.24, 27.42. HRMS (ESI): calcd. for $C_{17}H_{13}N_2O_3^+$ ($[M+H]^+$): 293.0921, found 293.0918.

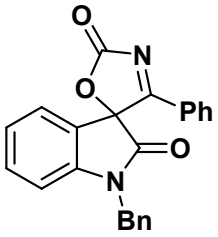
1,4'-diphenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ba

Yellow solid (95.6 mg, 90%), mp: 225.4-226.2 °C. ^1H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.8 Hz, 2H), 7.63-7.55 (m, 3H), 7.51-7.40 (m, 6H), 7.22 (d, *J* = 7.4 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.02 (d, *J* = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.04, 167.36, 165.62, 144.49, 135.63, 133.21, 132.66, 130.22, 129.87, 129.64, 129.27, 127.67, 126.21, 125.60, 125.05, 122.58, 111.39, 88.35. HRMS (ESI): calcd. for $C_{22}H_{15}N_2O_3^+$ ($[M+H]^+$): 355.1077, found 355.1076.

1-benzyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione

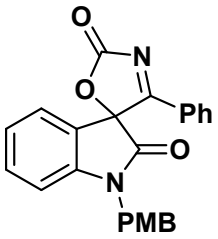


3ca

Yellow solid (101.6 mg, 92%), mp: 188.8-189.6 °C. ^1H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.8 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.36-7.30 (m, 5H), 7.29-7.24 (m, 2H), 7.13 (d, *J* = 7.4 Hz, 1H), 7.09-7.05 (m, 1H), 7.02 (d, *J* = 8.0 Hz, 1H), 5.16 (d, *J* = 15.3 Hz, 1H), 4.79 (d, *J* = 15.3 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.15, 168.19, 165.69, 143.46, 135.50, 134.55,

132.62, 129.96, 129.46, 129.21, 128.54, 128.10, 127.41, 125.38, 124.56, 122.87, 111.02, 88.27, 44.98. HRMS (ESI): calcd. for $C_{23}H_{17}N_2O_3^+$ ([M+H]$^+$): 369.1234, found 369.1239.

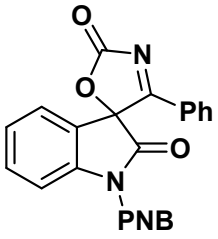
1-(4-methoxybenzyl)-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3da

Yellow solid (106.3 mg, 89%), mp: 182.7-183.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63-7.59 (m, 2H), 7.57-7.52 (m, 1H), 7.42 (td, *J* = 7.7, 1.5 Hz, 1H), 7.31-7.23 (m, 4H), 7.14-7.02 (m, 3H), 6.89-6.84 (m, 2H), 5.11 (d, *J* = 15.2 Hz, 1H), 4.72 (d, *J* = 15.1 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.17, 168.12, 165.73, 159.74, 143.50, 135.48, 132.57, 129.97, 129.55, 129.44, 127.42, 126.57, 125.37, 124.49, 122.93, 114.53, 111.04, 88.31, 55.47, 44.45. HRMS (ESI): calcd. for $C_{24}H_{19}N_2O_4^+$ ([M+H]$^+$): 399.1339, found 399.1335.

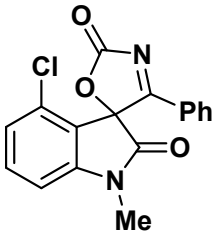
1-(4-nitrobenzyl)-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ea

Yellow solid (111.5 mg, 90%), mp: 211.1-211.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.1 Hz, 2H), 7.65-7.57 (m, 3H), 7.54-7.41 (m, 3H), 7.37-7.28 (m, 2H), 7.23-7.11 (m, 2H), 6.97-6.92 (m, 1H), 5.19 (d, *J* = 15.9 Hz, 1H), 4.98 (d, *J* = 15.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.83, 168.40, 165.42, 148.09, 142.88, 141.78, 135.77, 132.85, 129.85, 129.56, 128.72, 127.48, 125.82, 125.14, 124.46, 122.78, 110.55, 88.00, 44.26. HRMS (ESI): calcd. for $C_{23}H_{16}N_3O_5^+$ ([M+H]$^+$): 414.1084, found 414.1080.

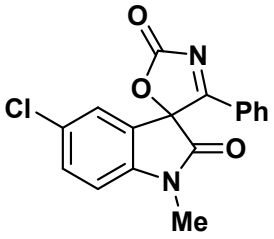
4-chloro-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3fa

Yellow solid (86.1 mg, 88%), mp: 207.9-208.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.66 (m, 2H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.47 (t, *J* = 8.1 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.05 (d, *J* = 8.3 Hz, 1H), 6.98 (d, *J* = 7.9 Hz, 1H), 3.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.28, 167.59, 165.39, 145.96, 135.62, 133.82, 133.11, 129.66, 129.32, 127.73, 125.09, 120.19, 108.35, 87.43, 27.69. HRMS (ESI): calcd. for $C_{17}H_{12}ClN_2O_3^+$ ($[M+H]^+$): 327.0531, found 327.0527.

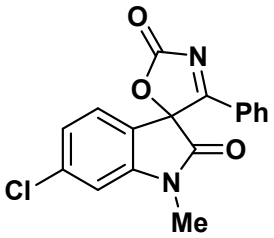
5-chloro-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ga

Yellow solid (91.0 mg, 93%), mp: 209.7-210.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.7 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.50 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 2.1 Hz, 1H), 7.02 (d, *J* = 8.4 Hz, 1H), 3.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.50, 167.81, 165.20, 142.91, 135.80, 132.73, 130.06, 129.77, 129.73, 127.33, 125.75, 124.35, 111.11, 87.57, 27.61. HRMS (ESI): calcd. for $C_{17}H_{12}ClN_2O_3^+$ ($[M+H]^+$): 327.0531, found 327.0532.

6-chloro-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione

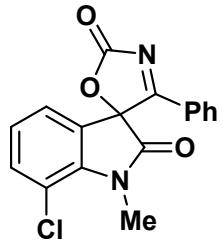


3ha

Yellow solid (88.0 mg, 90%), mp: 207.1-207.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.4 Hz, 2H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.14-7.05 (m, 3H), 3.35 (s, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 187.66, 168.13, 165.30, 145.59, 138.90, 135.73, 129.71, 127.38, 126.31, 124.54,

121.12, 111.00, 87.49, 27.59. HRMS (ESI): calcd. for $C_{17}H_{12}ClN_2O_3^+$ ([M+H]$^+$): 327.0531, found 327.0531.

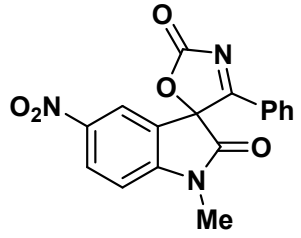
7-chloro-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ia

Yellow solid (87.8 mg, 90%), mp: 204.1-204.9 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.43 (m, 3H), 7.04 (d, *J* = 8.7 Hz, 2H), 3.71 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.52, 168.46, 165.25, 140.31, 135.74, 134.98, 129.76, 129.71, 127.32, 125.34, 123.88, 117.34, 87.31, 30.93. HRMS (ESI): calcd. for $C_{17}H_{12}ClN_2O_3^+$ ([M+H]$^+$): 327.0531, found 327.0532.

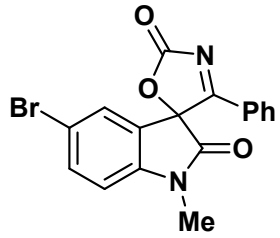
1-methyl-5-nitro-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ja

Yellow solid (87.6 mg, 87%), mp: 210.3-211.1 °C. ^{1}H NMR (500 MHz, DMSO) δ 8.76 (s, 1H), 8.52 (d, *J* = 8.9 Hz, 1H), 7.75-7.50 (m, 6H), 3.42 (s, 3H). ^{13}C NMR (125 MHz, DMSO) δ 186.39, 168.55, 164.74, 150.00, 143.95, 136.01, 130.08, 129.25, 126.50, 122.51, 122.37, 111.74, 86.55, 27.90. HRMS (ESI): calcd. for $C_{17}H_{12}N_3O_5^+$ ([M+H]$^+$): 338.0771, found 338.0771.

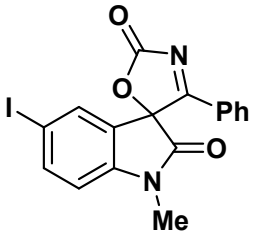
5-bromo-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ka

Yellow solid (95.4 mg, 86%), mp: 220.5-221.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69-7.58 (m, 4H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.28-7.24 (m, 1H), 6.97 (d, *J* = 8.4 Hz, 1H), 3.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.39, 167.60, 165.08, 143.28, 135.70, 135.53, 129.65, 129.62, 128.31, 127.19, 124.50, 116.94, 111.46, 87.38, 27.47. HRMS (ESI): calcd. for $C_{17}H_{12}BrN_2O_3^+$ ([M+H]$^+$): 371.0026, found 371.0027.

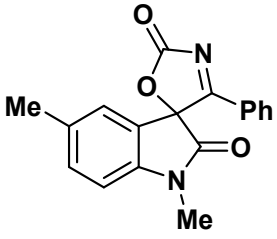
5-iodo-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3la

Yellow solid (100.3 mg, 80%), mp: 211.5-212.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.2 Hz, 1H), 7.68 (d, *J* = 7.7 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.45-7.34 (m, 3H), 6.86 (d, *J* = 8.2 Hz, 1H), 3.34 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.49, 167.58, 165.21, 144.05, 141.54, 135.79, 133.90, 131.07, 129.79, 129.74, 127.34, 124.89, 111.97, 86.64, 27.52. HRMS (ESI): calcd. for $C_{17}H_{12}IN_2O_3^+$ ([M+H]$^+$): 418.9887, found 418.9884.

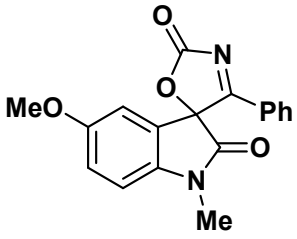
1,5-dimethyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ma

Yellow solid (82.6 mg, 90%), mp: 149.5-150.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.5 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 1H), 7.02-6.91 (m, 2H), 3.33 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.25, 168.11, 165.70, 141.89, 135.46, 134.54, 133.03, 129.79, 129.54, 127.59, 125.88, 122.76, 109.89, 88.45, 27.42, 21.05. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_3^+$ ([M+H]$^+$): 307.1077, found 307.1078.

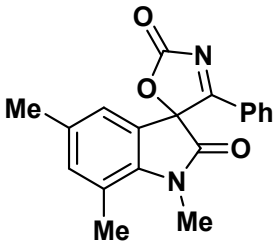
5-methoxy-1-methyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3na

Yellow solid (88.9 mg, 92%), mp: 205.3-206.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.06-6.96 (m, 2H), 6.72 (d, *J* = 2.5 Hz, 1H), 3.73 (s, 3H), 3.32 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.18, 167.90, 165.65, 157.19, 137.42, 135.54, 129.80, 129.57, 127.54, 123.79, 117.54, 111.78, 110.81, 88.52, 56.00, 27.48. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_4^+$ ([M+H]$^+$): 323.1026, found 323.1024.

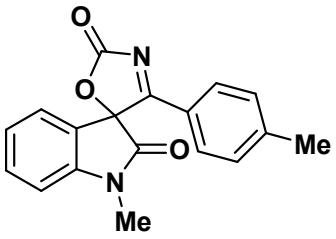
1,5,7-trimethyl-4'-phenyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3oa

Yellow solid (91.4 mg, 95%), mp: 216.9-217.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.7 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.42-7.36 (m, 2H), 7.04 (s, 1H), 6.75 (s, 1H), 3.57 (s, 3H), 2.63 (s, 3H), 2.21 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.29, 168.88, 165.75, 139.49, 136.88, 135.37, 134.33, 129.86, 129.52, 127.69, 123.70, 123.37, 121.56, 88.19, 30.73, 20.71, 18.94. HRMS (ESI): calcd. for $C_{19}H_{17}N_2O_3^+$ ([M+H]$^+$): 321.1234, found 321.1234.

1-methyl-4'-(p-tolyl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione

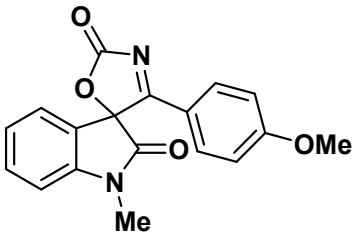


3ab

Yellow solid (86.4 mg, 94%), mp: 178.6-179.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.4 Hz, 2H), 7.54-7.49 (m, 1H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.15-7.10 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 1H), 3.35 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.90, 168.30, 165.89, 147.18, 144.33, 132.68,

130.34, 129.84, 125.25, 124.86, 124.53, 123.09, 110.03, 88.07, 27.37, 22.02. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_3^+$ ([M+H]$^+$): 307.1077, found 307.1078.

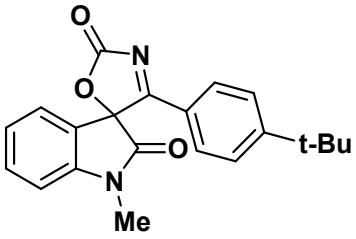
4'-(4-methoxyphenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ac

Yellow solid (87.0 mg, 90%), mp: 220.3-221.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.16-7.09 (m, 2H), 7.06 (d, *J* = 7.9 Hz, 1H), 6.84 (d, *J* = 8.5 Hz, 2H), 3.82 (s, 3H), 3.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 186.93, 168.52, 166.10, 165.65, 144.27, 132.62, 132.31, 125.29, 124.53, 123.42, 120.02, 115.12, 110.01, 87.76, 55.82, 27.36. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_4^+$ ([M+H]$^+$): 323.1026, found 323.1027.

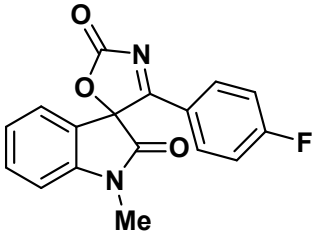
4'-(4-(tert-butyl)phenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ad

Yellow solid (91.9 mg, 88%), mp: 191.8-192.6°C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.7 Hz, 2H), 7.55-7.50 (m, 1H), 7.38 (d, *J* = 8.6 Hz, 2H), 7.16-7.09 (m, 2H), 7.07 (d, *J* = 7.9 Hz, 1H), 3.35 (s, 3H), 1.27 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.75, 168.37, 165.95, 160.01, 144.39, 132.66, 129.82, 126.68, 125.36, 124.80, 124.55, 123.18, 110.01, 88.08, 35.58, 30.93, 27.40. HRMS (ESI): calcd. for $C_{21}H_{21}N_2O_3^+$ ([M+H]$^+$): 349.1547, found 349.1551.

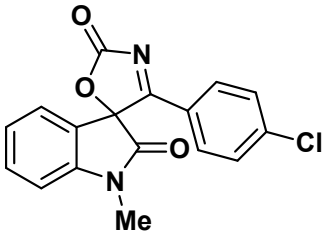
4'-(4-fluorophenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ae

Yellow solid (76.3 mg, 82%), mp: 194.4-195.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.68 (m, 2H), 7.58-7.51 (m, 1H), 7.17-7.03 (m, 5H), 3.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.76, 168.40, 168.07, 165.81, 165.42, 144.33, 132.94, 132.62, 132.53, 125.39, 124.73, 123.98, 123.95, 122.65, 117.26, 117.04, 110.18, 88.06, 27.48. HRMS (ESI): calcd. for $C_{17}H_{12}FN_2O_3^+$ ($[M+H]^+$): 311.0826, found 311.0826.

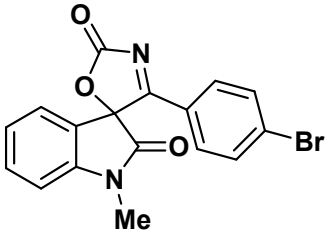
4'-(4-chlorophenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3af

Yellow solid (73.4 mg, 75%), mp: 180.6-181.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.59 (m, 2H), 7.57-7.51 (m, 1H), 7.37-7.34 (m, 2H), 7.14 (d, J = 4.4 Hz, 2H), 7.08 (d, J = 7.9 Hz, 1H), 3.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.03, 167.93, 165.33, 144.33, 142.34, 132.98, 130.95, 130.06, 125.93, 125.36, 124.73, 122.47, 110.21, 88.13, 27.48. HRMS (ESI): calcd. for $C_{17}H_{12}ClN_2O_3^+$ ($[M+H]^+$): 327.0531, found 327.0532.

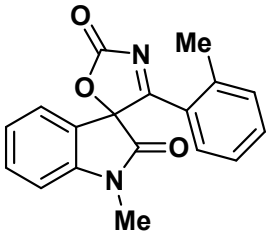
4'-(4-bromophenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ag

Yellow solid (69.9 mg, 63%), mp: 190.5-191.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56-7.51 (m, 5H), 7.14-7.12 (m, 2H), 7.07 (d, J = 8.0 Hz, 1H), 3.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.21, 167.91, 165.31, 144.35, 133.06, 132.97, 131.22, 130.94, 126.37, 125.39, 124.74, 122.48, 110.17, 88.13, 27.49. HRMS (ESI): calcd. for $C_{17}H_{12}BrN_2O_3^+$ ($[M+H]^+$): 371.0026, found 371.0027.

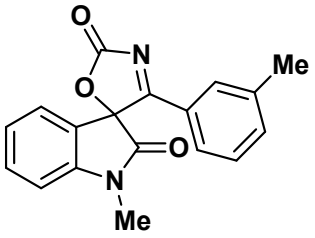
1-methyl-4'-(o-tolyl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3ai

Yellow solid (71.7 mg, 78%), mp: 199.4-200.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (t, *J* = 7.7 Hz, 1H), 7.41-7.30 (m, 2H), 7.18-7.10 (m, 2H), 7.05-6.98 (m, 2H), 6.88 (d, *J* = 8.0 Hz, 1H), 3.28 (s, 3H), 2.70 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.90, 168.34, 165.93, 144.43, 142.62, 133.62, 133.00, 132.60, 129.27, 126.83, 126.24, 124.93, 124.47, 122.59, 110.00, 89.66, 27.31, 23.32. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_3^+$ ($[M+H]^+$): 307.1077, found 307.1077.

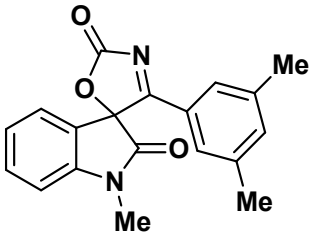
1-methyl-4'-(m-tolyl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3aj

Yellow solid (82.7 mg, 90%), mp: 183.7-184.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.55-7.50 (m, 1H), 7.38 (d, *J* = 7.4 Hz, 1H), 7.26 (d, *J* = 7.6 Hz, 1H), 7.21 (t, *J* = 7.7 Hz, 1H), 7.14-7.10 (m, 2H), 7.07 (d, *J* = 7.9 Hz, 1H), 3.35 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.31, 168.20, 165.70, 144.36, 139.54, 136.40, 132.74, 130.46, 129.34, 127.46, 126.70, 125.27, 124.57, 122.93, 109.99, 88.24, 27.38, 21.35. HRMS (ESI): calcd. for $C_{18}H_{15}N_2O_3^+$ ($[M+H]^+$): 307.1077, found 307.1078.

4'-(3,5-dimethylphenyl)-1-methyl-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione

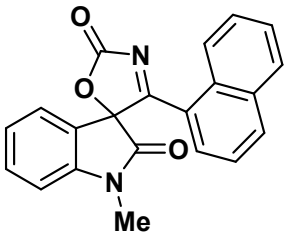


3ak

Yellow solid (88.3 mg, 92%), mp: 217.9-218.6 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55-7.50 (m, 1H), 7.27 (s, 2H), 7.19 (s, 1H), 7.15-7.10 (m, 2H), 7.06 (d, *J* = 7.9 Hz, 1H), 3.34 (s, 3H), 2.21 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.43, 168.28, 165.78, 144.38, 139.22, 137.34, 132.69, 127.53, 125.30,

124.57, 123.09, 109.79, 88.23, 27.34, 21.26. HRMS (ESI): calcd. for $C_{19}H_{17}N_2O_3^+$ ([M+H]$^+$): 321.1234, found 321.1238.

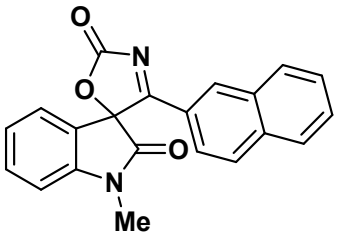
1-methyl-4'-(naphthalen-1-yl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3al

Yellow solid (92.4 mg, 90%), mp: 218.8-219.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.34 (d, *J* = 8.7 Hz, 1H), 8.00 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 8.1 Hz, 1H), 7.73-7.69 (m, 1H), 7.61-7.56 (m, 1H), 7.51-7.46 (m, 1H), 7.27-7.21 (m, 2H), 7.18 (d, *J* = 7.4 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 7.9 Hz, 1H), 3.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.21, 168.52, 165.84, 144.39, 136.09, 134.18, 132.63, 131.52, 130.54, 129.65, 129.01, 127.40, 126.70, 125.02, 124.55, 124.38, 123.15, 110.05, 89.80, 27.38. HRMS (ESI): calcd. for $C_{21}H_{15}N_2O_3^+$ ([M+H]$^+$): 343.1077, found 343.1073.

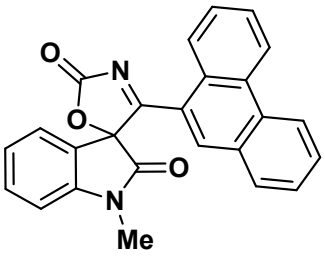
1-methyl-4'-(naphthalen-2-yl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3am

Yellow solid (83.1 mg, 81%), mp: 219.4-220.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.84-7.79 (m, 3H), 7.68-7.48 (m, 4H), 7.19 (d, *J* = 7.3 Hz, 1H), 7.15-7.10 (m, 2H), 3.39 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.86, 168.18, 144.36, 136.39, 132.69, 132.31, 129.94, 129.80, 129.43, 127.92, 127.38, 125.37, 124.86, 124.56, 124.33, 109.81, 88.17, 27.33. HRMS (ESI): calcd. for $C_{21}H_{15}N_2O_3^+$ ([M+H]$^+$): 343.1077, found 343.1074.

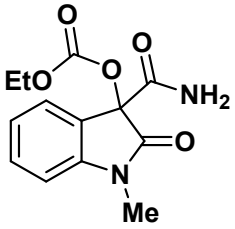
1-methyl-4'-(phenanthren-9-yl)-2'H-spiro[indoline-3,5'-oxazole]-2,2'-dione



3an

Yellow solid (97.6 mg, 83%), mp: 249.9-250.8 °C. ^{1}H NMR (400 MHz, DMSO) δ 9.09-9.04 (m, 1H), 8.94-8.89 (m, 1H), 8.85 (d, *J* = 8.3 Hz, 1H), 7.87-7.79 (m, 3H), 7.75-7.63 (m, 3H), 7.59-7.51 (m, 2H), 7.30 (d, *J* = 7.9 Hz, 1H), 7.13 (td, *J* = 7.6, 1.0 Hz, 1H), 3.36 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 188.10, 168.02, 165.24, 144.23, 132.78, 132.16, 131.86, 130.99, 130.45, 130.23, 128.52, 128.33, 128.24, 128.00, 127.67, 126.61, 125.81, 124.27, 123.57, 123.20, 123.12, 121.59, 110.89, 89.89, 27.33. HRMS (ESI): calcd. for $C_{25}H_{17}N_2O_3^+$ ([M+H]$^+$): 393.1234, found 393.1231.

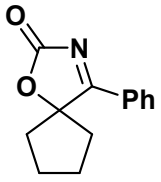
3-carbamoyl-1-methylindolin-3-yl ethyl carbonate



4a

White solid , mp: 180.1-180.9 °C. ^{1}H NMR (500 MHz, DMSO) δ 7.90 (br, 1H), 7.81 (br, 1H), 7.40 (t, *J* = 7.7 Hz, 1H), 7.33 (d, *J* = 7.3 Hz, 1H), 7.09-7.04 (m, 2H), 4.11 -4.02 (m, 2H), 3.14 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, DMSO) δ 170.63, 166.15, 151.29, 145.29, 130.66, 125.46, 122.63, 122.60, 109.08, 81.72, 64.87, 26.42, 13.86. HRMS (ESI): calcd. for $C_{13}H_{14}N_2NaO_5^+$ ([M+Na]$^+$): 301.0795, found 301.0790.

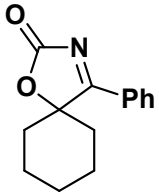
4-phenyl-1-oxa-3-azaspiro[4.4]non-3-en-2-one



6a

White solid (54.3 mg, 84%), mp: 102.1-102.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12-8.05 (m, 2H), 7.72-7.65 (m, 1H), 7.58-7.53 (m, 2H), 2.48-2.37 (m, 2H), 2.22-2.04 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.60, 165.57, 134.81, 130.05, 129.38, 127.97, 98.84, 39.24, 26.30. HRMS (ESI): calcd. for $C_{13}H_{14}NO_2^+$ ([M+H]$^+$): 216.1019, found 216.1015.

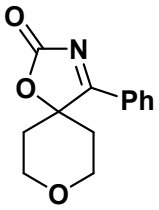
4-phenyl-1-oxa-3-azaspiro[4.5]dec-3-en-2-one



6b

White solid (58.4 mg, 85%), mp: 98.5-99.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.0 Hz, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 2H), 2.23-2.13 (m, 2H), 1.96-1.80 (m, 7H), 1.49-1.38 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.80, 165.40, 134.70, 130.42, 129.31, 128.37, 91.79, 34.49, 24.67, 22.22. HRMS (ESI): calcd. for $C_{14}H_{16}NO_2^+$ ([M+H]$^+$): 230.1176, found 230.1178.

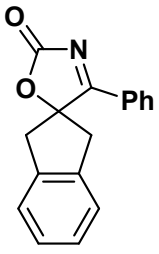
4-phenyl-1,8-dioxa-3-azaspiro[4.5]dec-3-en-2-one



6c

White solid (56.3 mg, 81%), mp: 140.2-141.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27-8.22 (m, 2H), 7.74-7.68 (m, 1H), 7.59 (t, *J* = 7.8 Hz, 2H), 4.14-4.05 (m, 2H), 3.95-3.87 (m, 2H), 2.65-2.55 (m, 2H), 1.76-1.68 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.82, 164.79, 135.15, 130.52, 129.50, 127.84, 88.57, 64.21, 34.36. HRMS (ESI): calcd. for $C_{13}H_{14}NO_3^+$ ([M+H]$^+$): 232.0968, found 232.0970.

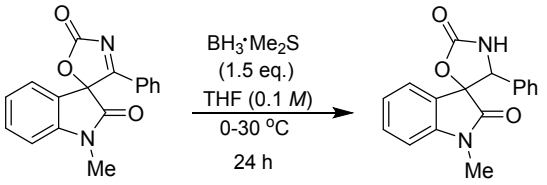
4'-phenyl-1,3-dihydro-2'H-spiro[indene-2,5'-oxazol]-2'-one



6d

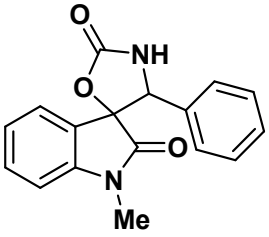
White solid (67.9 mg, 86%), mp: 144.1-144.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.92 (m, 2H), 7.68-7.62 (m, 1H), 7.50-7.44 (m, 2H), 7.37-7.29 (m, 4H), 3.85-3.79 (m, 2H), 3.59-3.5 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.27, 165.64, 138.46, 135.20, 130.09, 129.53, 128.10, 127.40, 125.09, 95.74, 45.09. HRMS (ESI): calcd. for $C_{17}H_{14}NO_2^+$ ([M+H]$^+$): 264.1019, found 264.1020.

V. Synthetic Transformation



To solution of **3aa** (0.25 mmol) in THF (0.1 M) was added BH_3 • Me_2S (1.5 equiv) dropwise at 0 °C.[4] After stirring at 30 °C for 24 hours, the reaction mixture was cooled to 0 °C and quenched by dropwise addition of a 1:1 mixture of THF:H_2O. The aqueous layer was extracted with dichloromethane three times. The combined organic layers were dried over anhydrous Na_2SO_4, filtered and evaporated under reduced pressure. The crude product was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (60-90℃) to afford the desired product **7**.

1-methyl-4'-phenylspiro[indoline-3,5'-oxazolidine]-2,2'-dione



7

White solid (64.7 mg, 88%) , mp: 229.6-230.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 7.28-7.20 (m, 4H), 7.08-7.05 (m, 2H), 6.72 (d, *J* = 7.8 Hz, 1H), 6.41 (br, 1H), 5.33 (s, 1H), 2.78 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.89, 158.24, 144.46, 132.65, 131.70, 129.06, 128.51, 126.23, 124.88, 124.51, 123.57, 108.62, 84.42, 65.21, 25.86. HRMS (ESI): calcd. for $C_{17}H_{15}N_2O_3^+$ ($[M+H]^+$): 295.1077, found 295.1078.

VI. References

1. A. Baeza, C. Nájera, M. G. Retamosa, J. M. Sansano. *Synthesis*, 2005, 16, 2787.

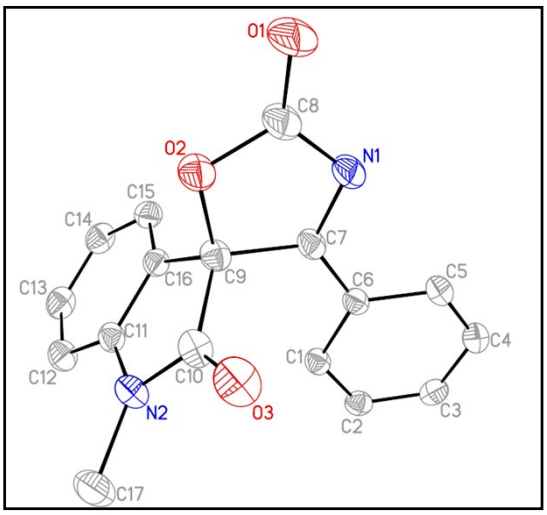
2. S. Aokia, S. Kotanib, M. Sugiuraa, M. Nakajimaa.*Tetrahedron Letters*, 2010, 51, 3547.

3. Y. Ogura, M. Akakura, A. Sakakura, and K. Ishihara. *Angew. Chem. Int. Ed.* 2013, 52, 8299.

4. L. Zhao, W.-W. Liao. *Org. Chem. Front.*, 2018, 5, 801.

VII. Crystal Data and Structure Refinement

1) Compound **3aa**



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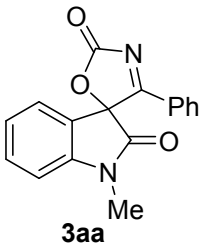


3aa

Table 1. Crystal data and structure refinement for 3aa.

Identification code	3aa	
Empirical formula	C17 H12 N2 O3	
Formula weight	292.29	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 9.3850(5) Å	α= 90°.
	b = 9.0474(4) Å	β= 97.418(2)°.
	c = 16.9250(9) Å	γ = 90°.
Volume	1425.07(12) $Å^3$	
Z	4	
Density (calculated)	1.362 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	608	
Crystal size	0.21 x 0.20 x 0.18 mm^3	
Theta range for data collection	3.05 to 27.48°.	
Index ranges	-12<=h<=12, -11<=k<=11, -21<=l<=21	
Reflections collected	28489	
Independent reflections	3257 [R(int) = 0.0453]	
Completeness to theta = 27.48°	99.8 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.9830 and 0.9803
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3257 / 0 / 200
Goodness-of-fit on F^2	1.018
Final R indices [I>2sigma(I)]	R1 = 0.0456, wR2 = 0.1157
R indices (all data)	R1 = 0.0758, wR2 = 0.1407
Largest diff. peak and hole	0.156 and -0.178 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for Y. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7314(2)	1043(2)	2539(1)	49(1)
C(2)	6974(2)	1293(2)	3292(1)	54(1)
C(3)	5872(2)	517(2)	3574(1)	62(1)
C(4)	5115(2)	-526(2)	3093(1)	70(1)
C(5)	5444(2)	-776(2)	2341(1)	62(1)
C(6)	6543(2)	3(2)	2048(1)	47(1)
C(7)	6867(2)	-349(2)	1245(1)	50(1)
C(8)	6731(2)	-1353(2)	71(1)	72(1)
C(9)	8167(2)	182(2)	884(1)	51(1)
C(10)	9594(2)	-376(2)	1361(1)	57(1)
C(11)	9770(2)	2120(2)	1225(1)	51(1)
C(12)	10287(2)	3541(2)	1287(1)	66(1)
C(13)	9402(3)	4651(2)	946(1)	72(1)
C(14)	8048(2)	4359(2)	557(1)	70(1)
C(15)	7529(2)	2916(2)	498(1)	59(1)
C(16)	8409(2)	1813(2)	831(1)	48(1)
C(17)	11845(2)	753(3)	2014(2)	83(1)
N(1)	6070(2)	-1207(2)	766(1)	67(1)
N(2)	10440(2)	820(2)	1542(1)	58(1)
O(1)	6311(2)	-2085(2)	-498(1)	102(1)
O(2)	7958(2)	-527(2)	117(1)	69(1)
O(3)	9873(2)	-1656(2)	1536(1)	82(1)

Table 3. Bond lengths [Å] and angles [°] for Y.

C(1)-C(2)	1.371(2)
C(1)-C(6)	1.395(2)
C(2)-C(3)	1.385(3)
C(3)-C(4)	1.382(3)
C(4)-C(5)	1.366(3)
C(5)-C(6)	1.393(2)
C(6)-C(7)	1.465(2)
C(7)-N(1)	1.290(2)
C(7)-C(9)	1.511(2)
C(8)-O(1)	1.193(2)
C(8)-O(2)	1.367(2)
C(8)-N(1)	1.406(3)
C(9)-O(2)	1.439(2)
C(9)-C(16)	1.497(2)
C(9)-C(10)	1.555(3)
C(10)-O(3)	1.216(2)
C(10)-N(2)	1.353(2)
C(11)-C(12)	1.374(2)
C(11)-C(16)	1.390(2)
C(11)-N(2)	1.407(2)
C(12)-C(13)	1.381(3)
C(13)-C(14)	1.380(3)
C(14)-C(15)	1.392(3)
C(15)-C(16)	1.370(2)
C(17)-N(2)	1.452(3)
C(2)-C(1)-C(6)	120.03(16)
C(1)-C(2)-C(3)	120.66(17)
C(4)-C(3)-C(2)	119.60(19)
C(5)-C(4)-C(3)	120.01(18)
C(4)-C(5)-C(6)	121.03(18)
C(5)-C(6)-C(1)	118.66(17)
C(5)-C(6)-C(7)	118.21(15)
C(1)-C(6)-C(7)	123.09(15)
N(1)-C(7)-C(6)	122.70(16)
N(1)-C(7)-C(9)	111.76(16)

C(6)-C(7)-C(9)	125.50(14)
O(1)-C(8)-O(2)	122.5(2)
O(1)-C(8)-N(1)	126.4(2)
O(2)-C(8)-N(1)	111.10(15)
O(2)-C(9)-C(16)	112.90(14)
O(2)-C(9)-C(7)	101.54(13)
C(16)-C(9)-C(7)	118.28(14)
O(2)-C(9)-C(10)	109.33(14)
C(16)-C(9)-C(10)	102.94(13)
C(7)-C(9)-C(10)	111.92(15)
O(3)-C(10)-N(2)	127.19(18)
O(3)-C(10)-C(9)	125.42(17)
N(2)-C(10)-C(9)	107.39(15)
C(12)-C(11)-C(16)	121.17(17)
C(12)-C(11)-N(2)	128.10(17)
C(16)-C(11)-N(2)	110.71(14)
C(11)-C(12)-C(13)	117.47(18)
C(14)-C(13)-C(12)	121.80(18)
C(13)-C(14)-C(15)	120.41(19)
C(16)-C(15)-C(14)	117.84(18)
C(15)-C(16)-C(11)	121.30(15)
C(15)-C(16)-C(9)	130.91(16)
C(11)-C(16)-C(9)	107.76(15)
C(7)-N(1)-C(8)	107.44(16)
C(10)-N(2)-C(11)	111.18(15)
C(10)-N(2)-C(17)	123.79(17)
C(11)-N(2)-C(17)	125.02(16)
C(8)-O(2)-C(9)	108.13(14)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Y. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	48(1)	40(1)	58(1)	1(1)	4(1)	-2(1)

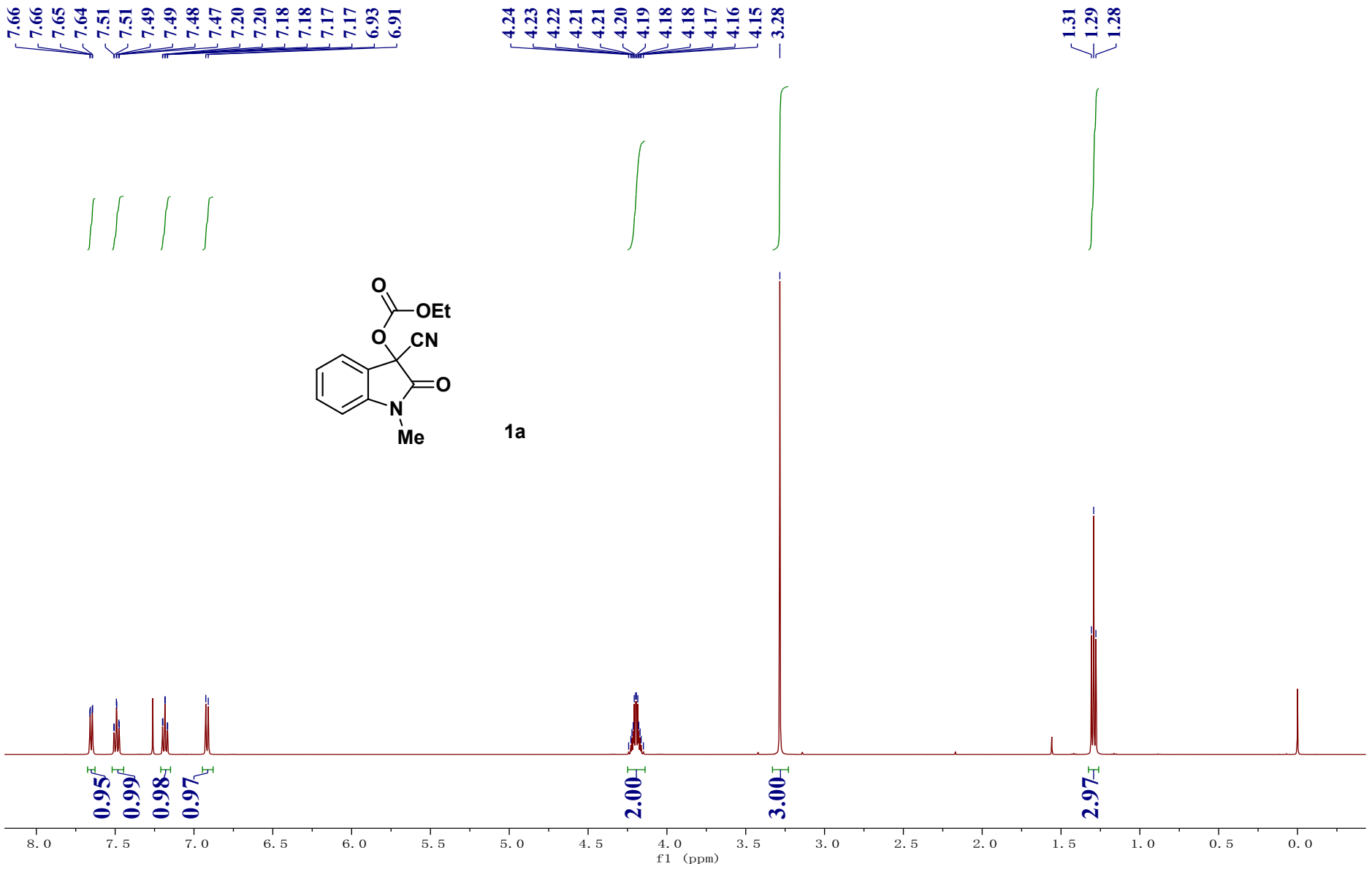
C(2)	54(1)	49(1)	58(1)	-1(1)	-2(1)	4(1)
C(3)	64(1)	69(1)	52(1)	8(1)	6(1)	9(1)
C(4)	63(1)	80(1)	67(1)	10(1)	10(1)	-15(1)
C(5)	58(1)	62(1)	65(1)	-2(1)	4(1)	-18(1)
C(6)	45(1)	38(1)	56(1)	3(1)	0(1)	-1(1)
C(7)	50(1)	37(1)	62(1)	-1(1)	3(1)	-6(1)
C(8)	81(1)	62(1)	72(1)	-20(1)	6(1)	-20(1)
C(9)	56(1)	42(1)	56(1)	-10(1)	7(1)	-6(1)
C(10)	54(1)	46(1)	72(1)	-8(1)	14(1)	1(1)
C(11)	53(1)	50(1)	52(1)	-5(1)	10(1)	-10(1)
C(12)	69(1)	56(1)	73(1)	-4(1)	6(1)	-22(1)
C(13)	97(2)	47(1)	74(1)	-2(1)	15(1)	-23(1)
C(14)	91(2)	50(1)	70(1)	11(1)	11(1)	-1(1)
C(15)	64(1)	56(1)	55(1)	4(1)	3(1)	-6(1)
C(16)	54(1)	43(1)	46(1)	-3(1)	8(1)	-8(1)
C(17)	55(1)	91(2)	99(2)	4(1)	-8(1)	-4(1)
N(1)	73(1)	60(1)	66(1)	-14(1)	6(1)	-22(1)
N(2)	46(1)	55(1)	72(1)	-4(1)	2(1)	-3(1)
O(1)	119(1)	102(1)	83(1)	-48(1)	10(1)	-38(1)
O(2)	76(1)	66(1)	65(1)	-24(1)	15(1)	-16(1)
O(3)	78(1)	47(1)	120(1)	-2(1)	9(1)	10(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Y.

	x	y	z	U(eq)
H(1)	8058	1568	2356	59
H(2)	7490	1990	3616	65
H(3)	5643	697	4083	74
H(4)	4380	-1058	3281	84
H(5)	4927	-1477	2021	75
H(12)	11197	3748	1550	79
H(13)	9729	5623	979	87
H(14)	7477	5130	333	84
H(15)	6615	2708	240	70

H(17A)	12049	-247	2182	125
H(17B)	11852	1376	2474	125
H(17C)	12563	1087	1700	125

VIII. ^{1}H and ^{13}C NMR Spectral Copies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4.20
4.19
4.18
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4.17
4.16
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1.29
1.28
O
OEt
O
CN
O
N
Me
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0.97
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3.00
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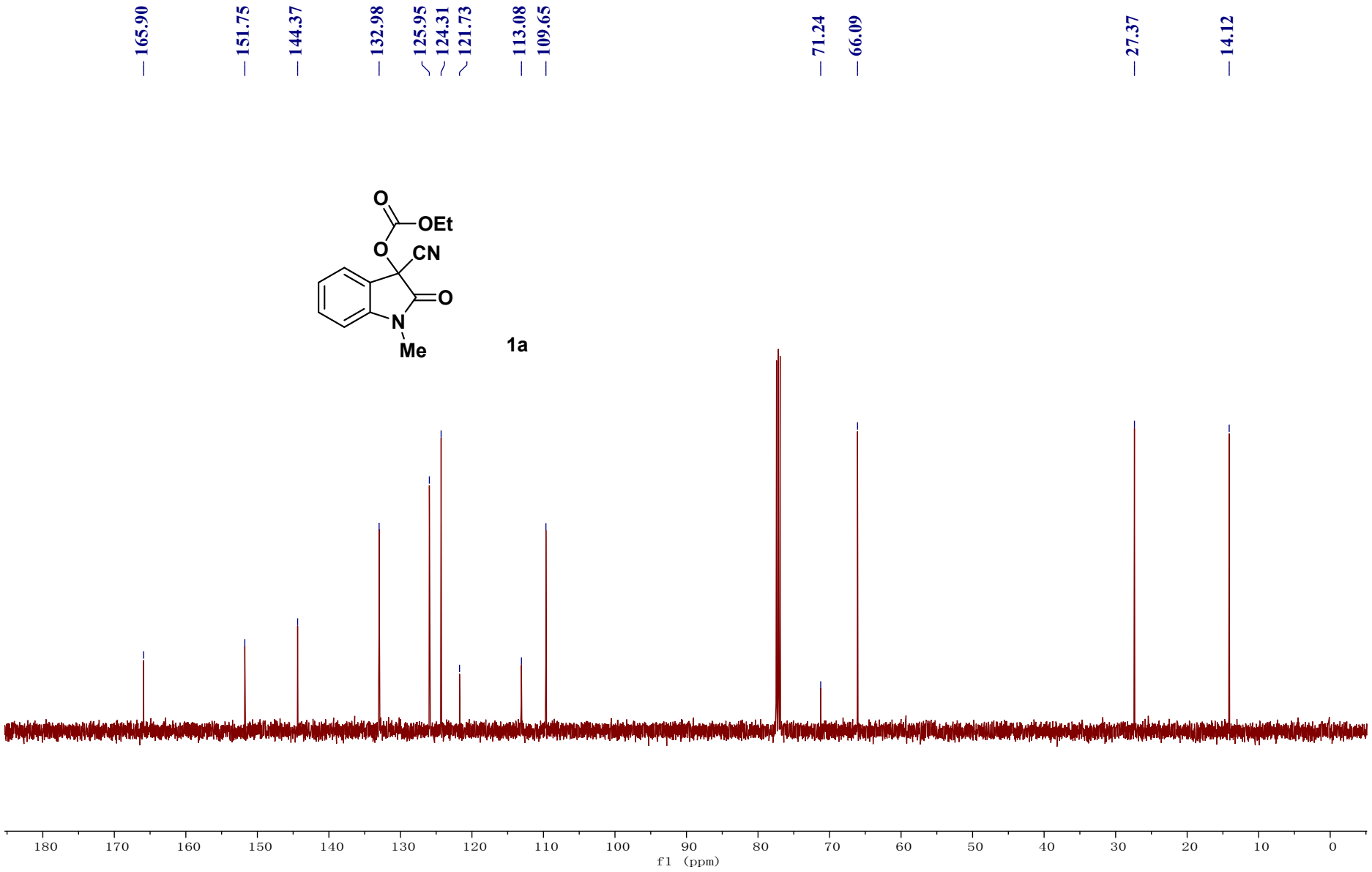
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O
OEt
O
CN
O
N
Me
1a
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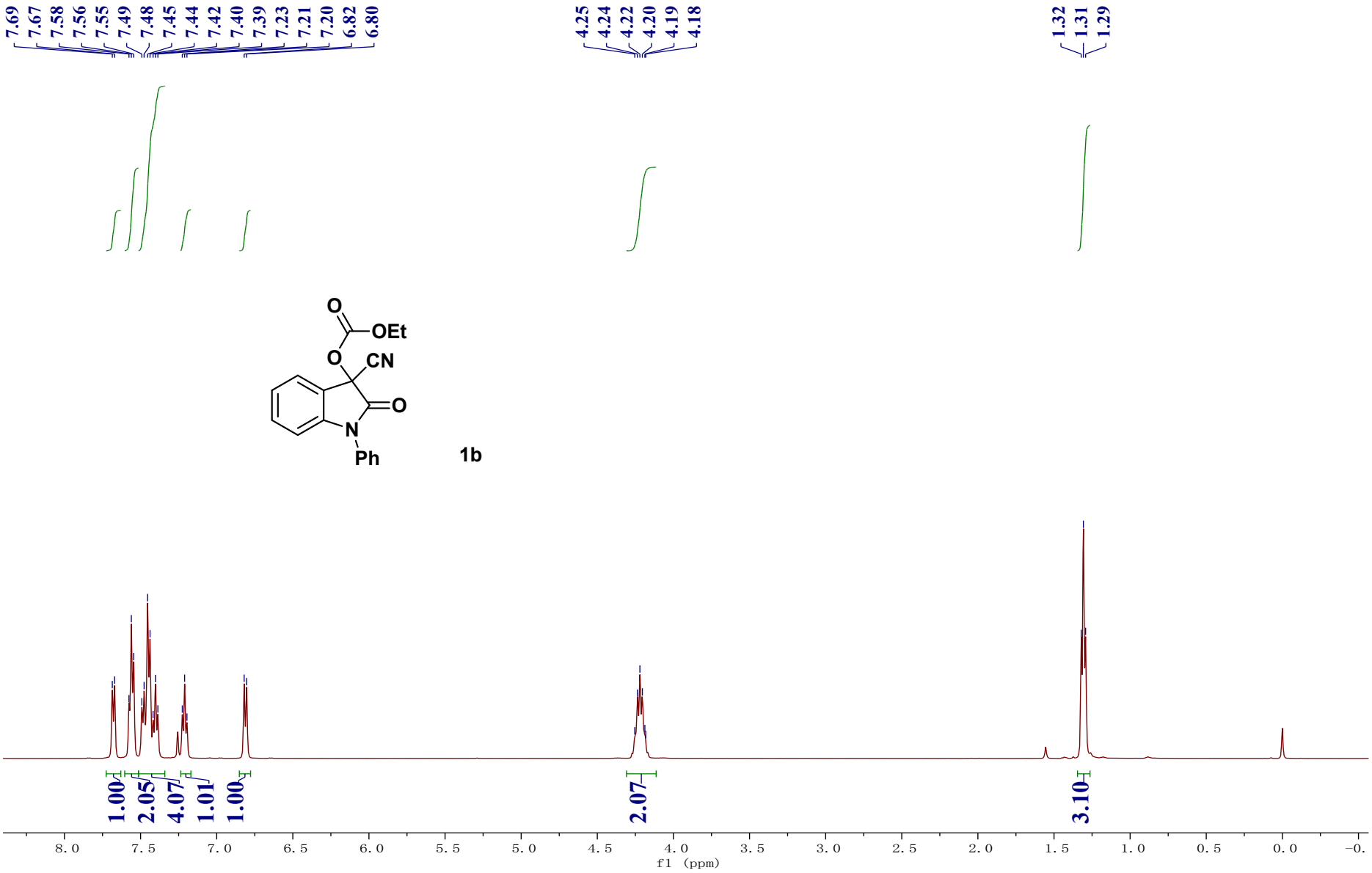
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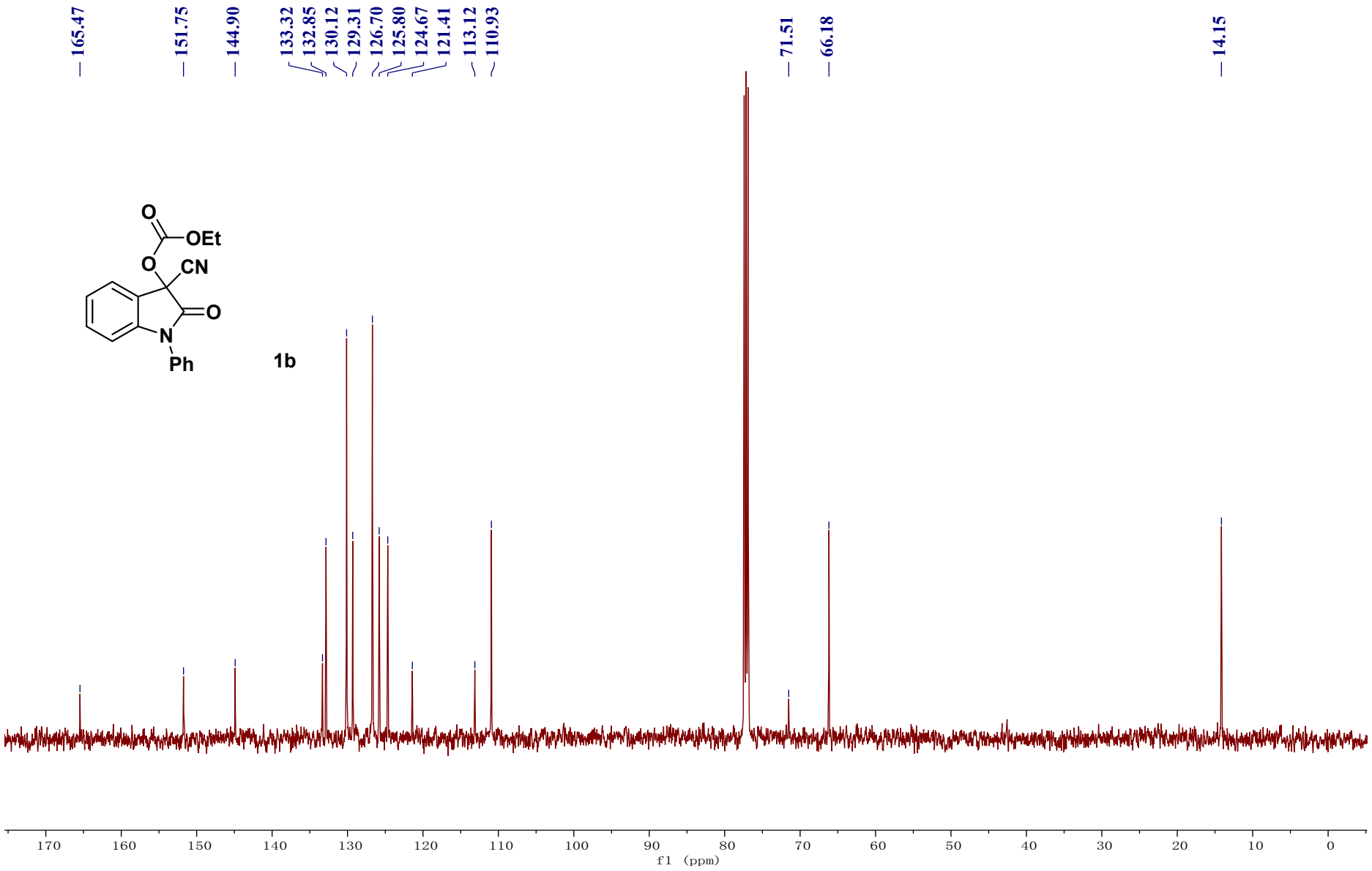
f1 (ppm)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Ph
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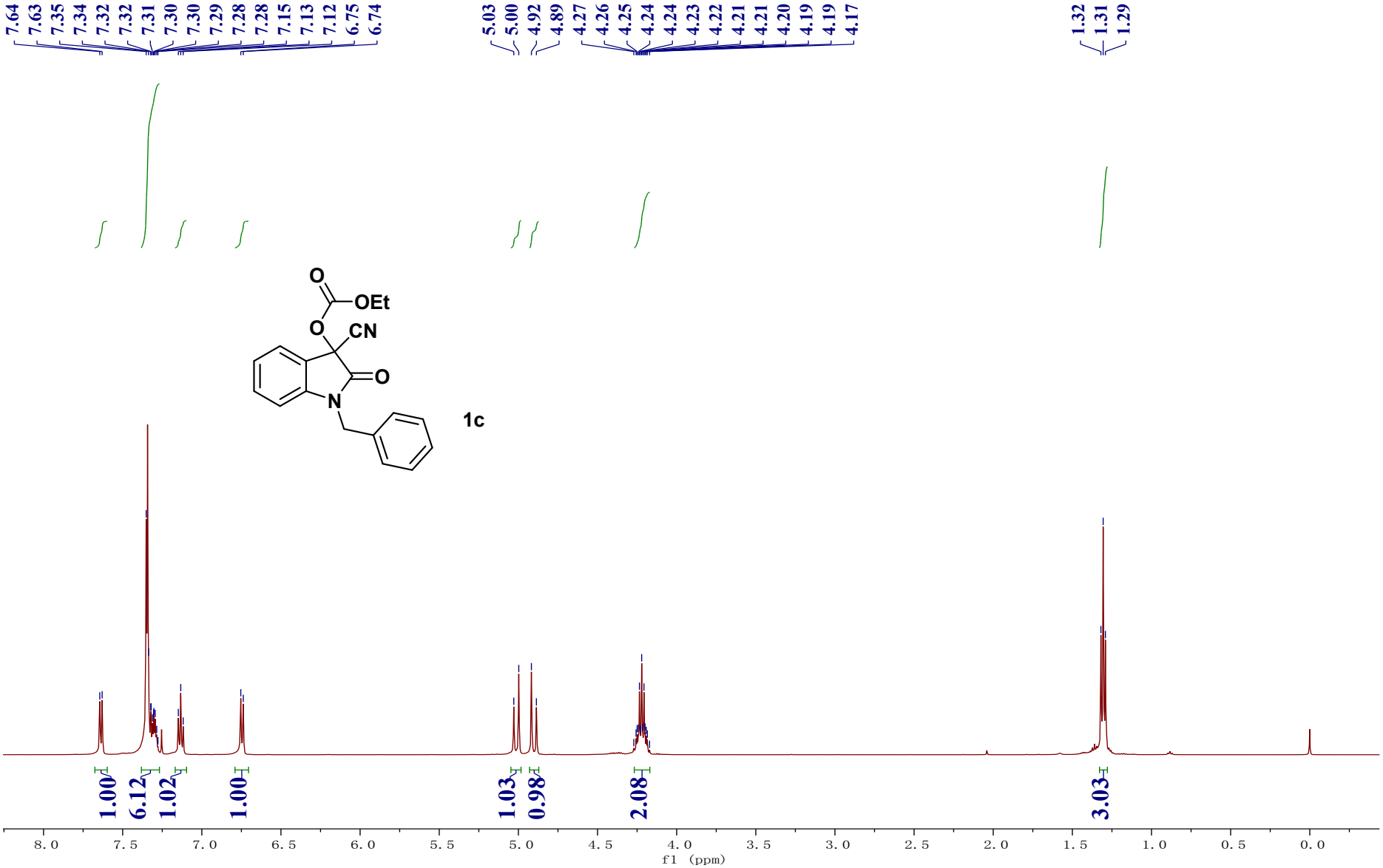
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5.00
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4.27
4.26
4.25
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4.23
4.22
4.21
4.21
4.20
4.19
4.19
4.17
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1.31
1.29
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O
CN
O
N
1c
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1.02
1.00
1.03
0.98
2.08
3.03
8.0
7.5
7.0
6.5
6.0
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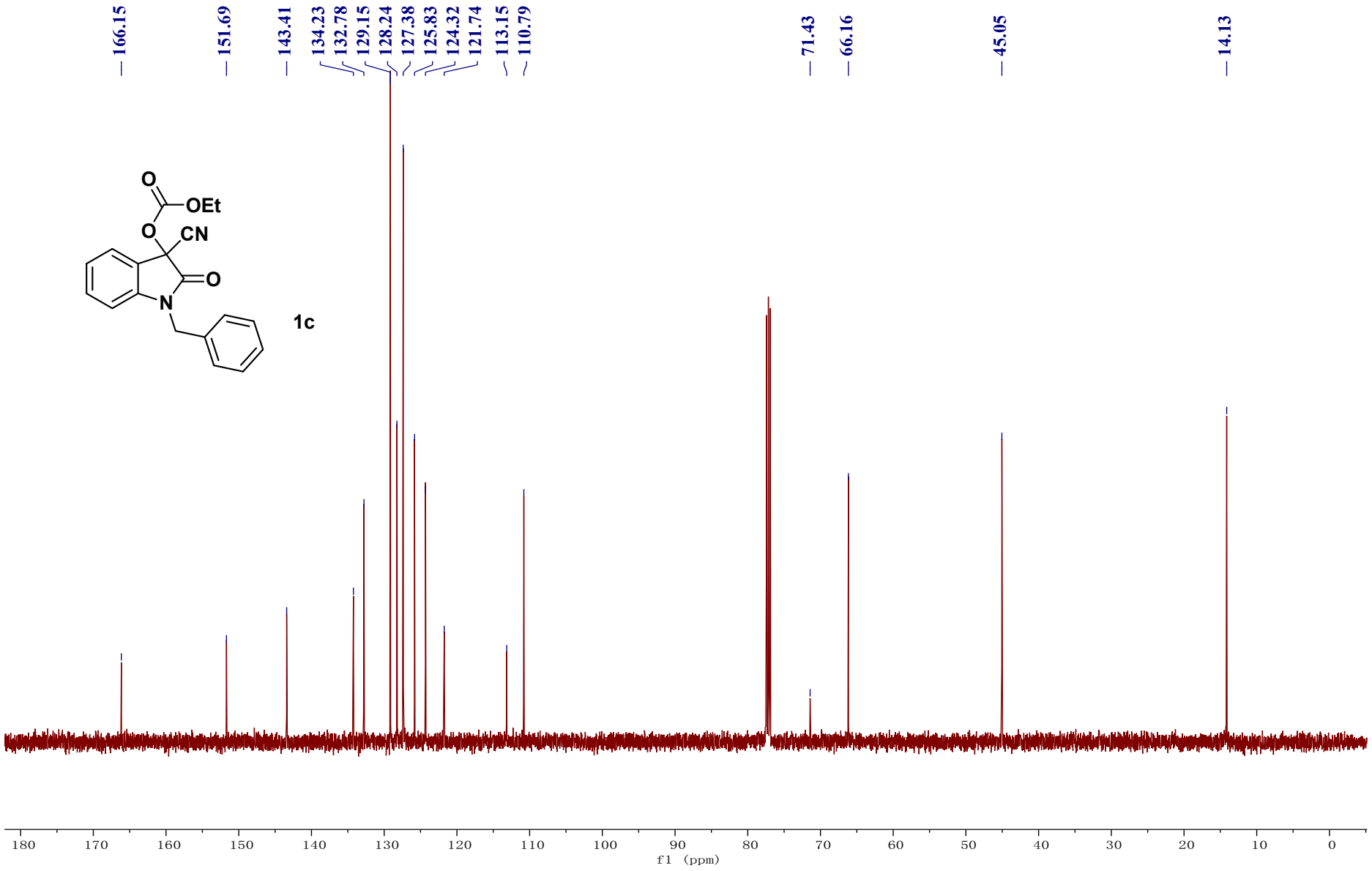
f1 (ppm)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71.43
66.16
45.05
14.13
O
OEt
O
CN
O
N
1c
180
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50
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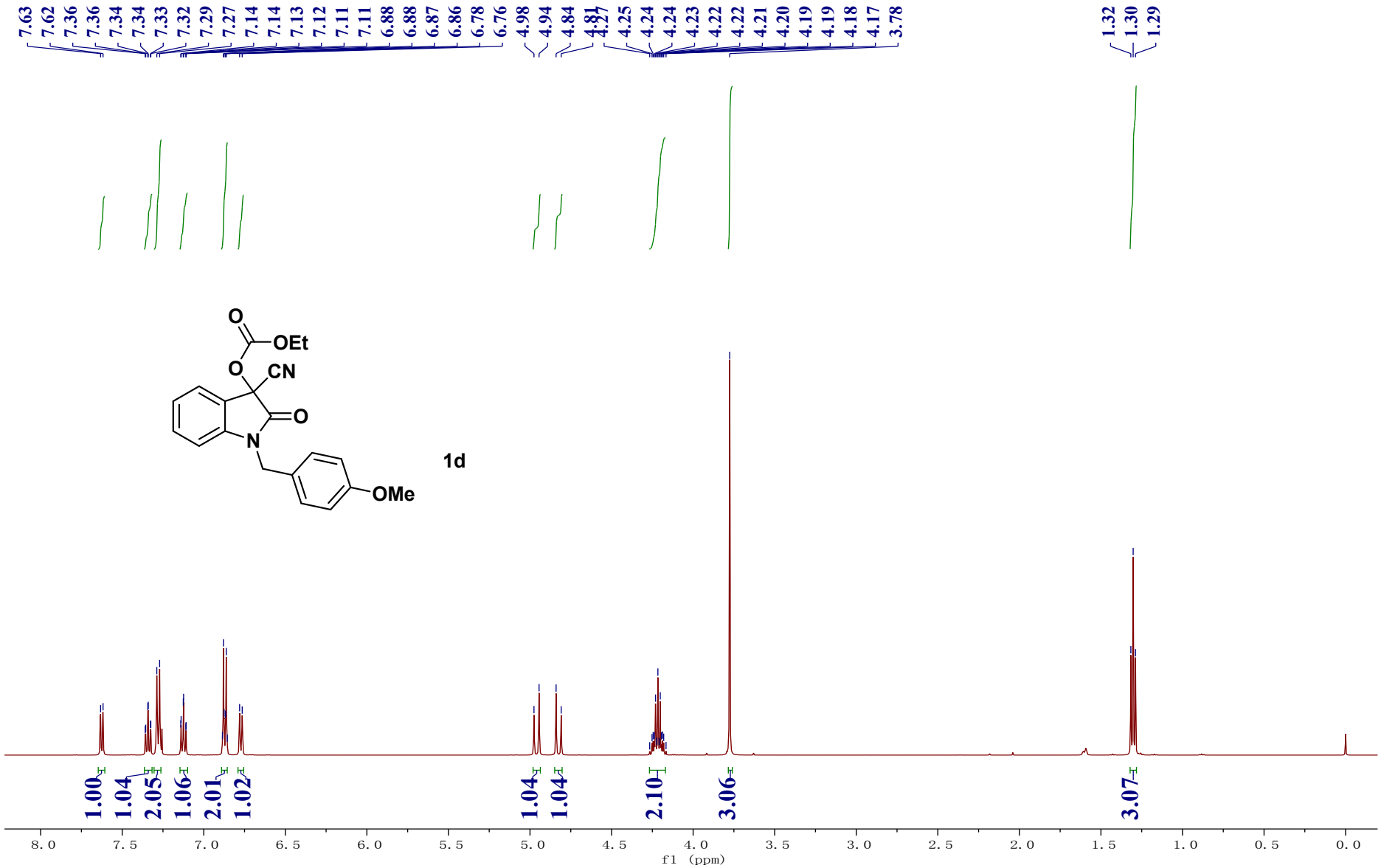
30
20
10
0
f1 (ppm)


7.63
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7.32
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7.11
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6.78
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4.19
4.18
4.17
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1.30
1.29
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OEt
O
CN
O
N
OMe
1d
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1.04
2.05
1.06
2.01
1.02
1.04
1.04
2.10
3.06
3.07
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7.0
6.5
6.0
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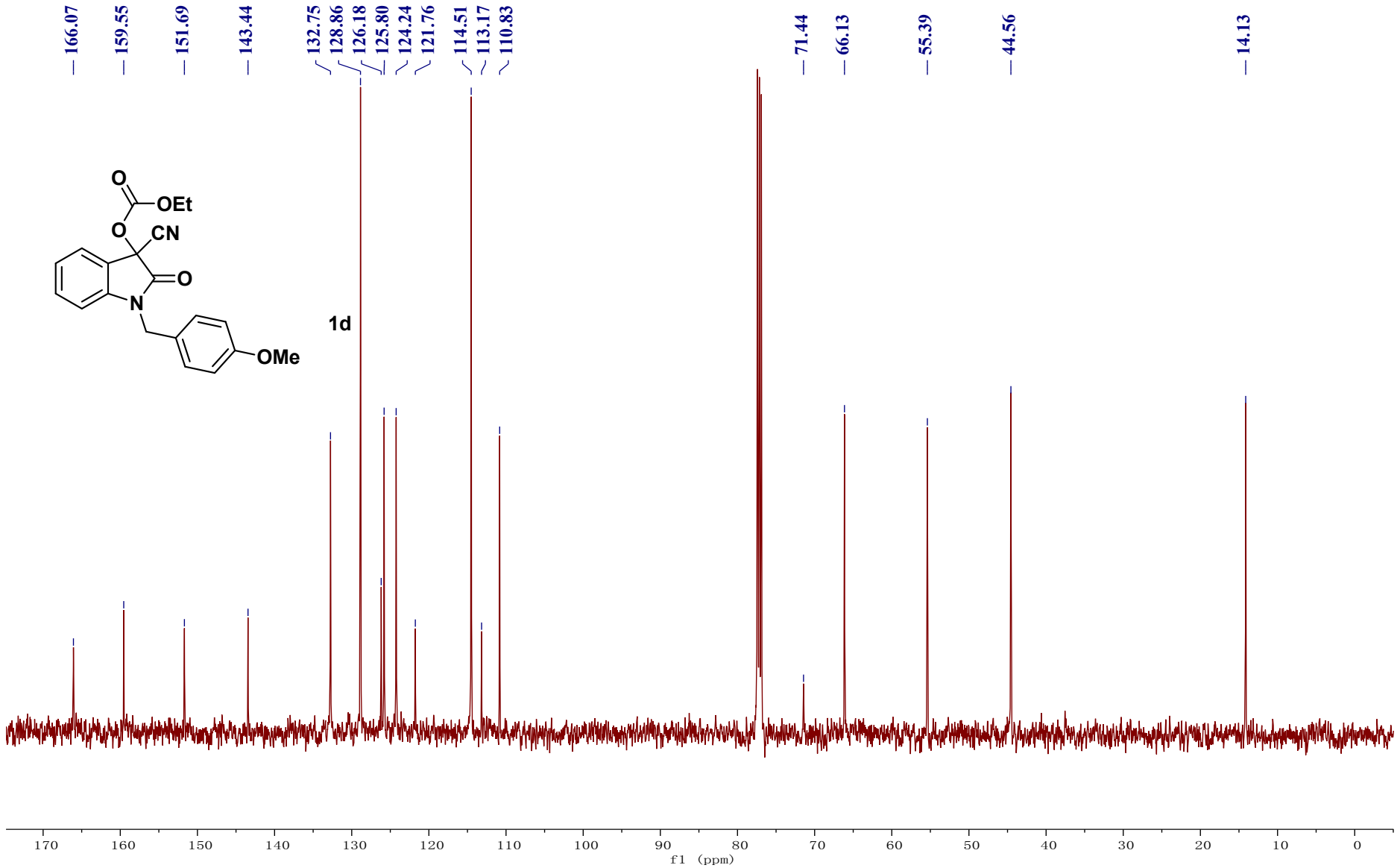
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71.44
66.13
55.39
44.56
14.13
O
OEt
O
CN
O
N
OMe
1d
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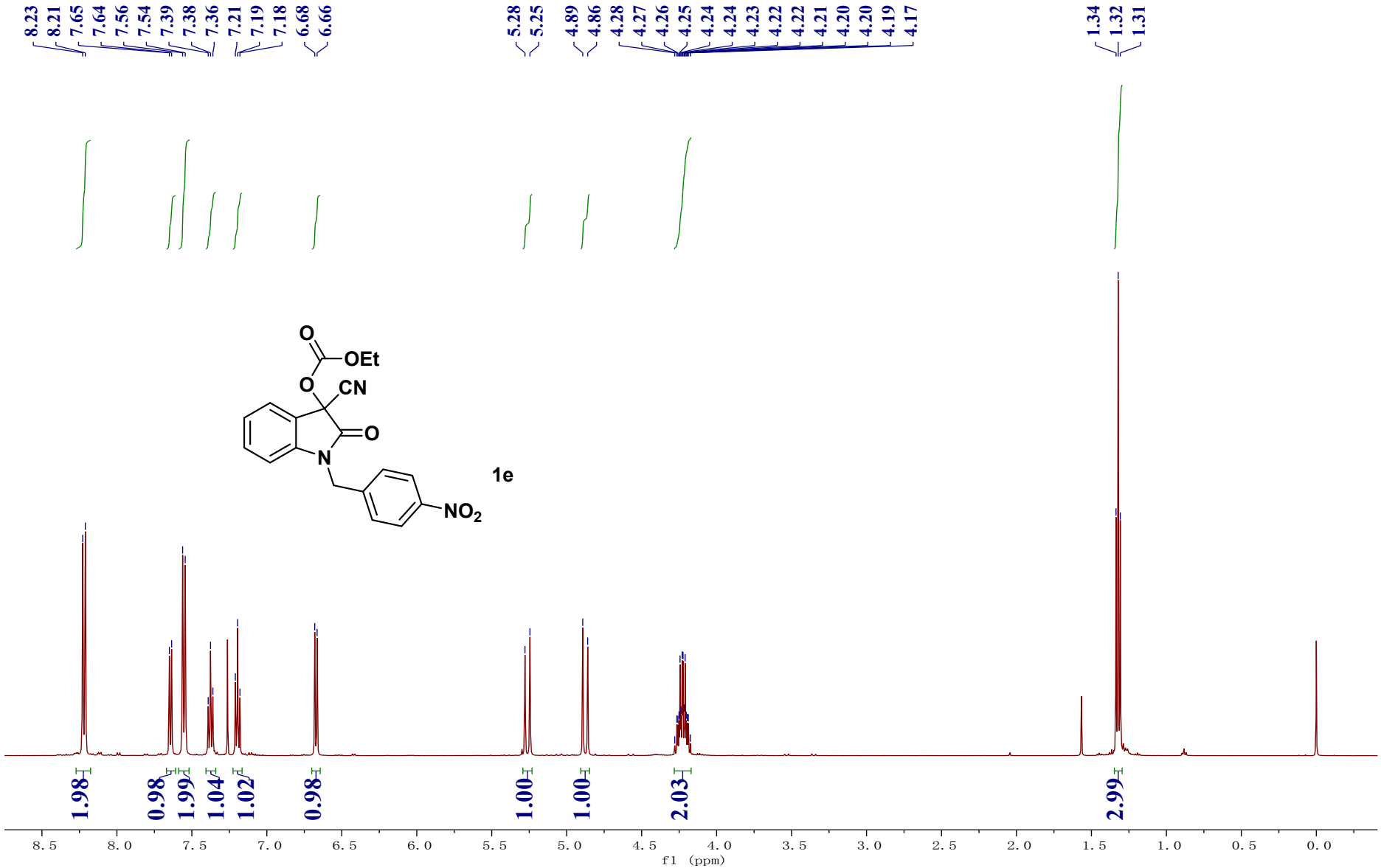
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5.25
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4.27
4.26
4.25
4.24
4.24
4.23
4.22
4.22
4.21
4.20
4.20
4.19
4.17
1.34
1.32
1.31
O
OEt
O
CN
O
N
NO2
1e
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0.98
1.99
1.04
1.02
0.98
1.00
1.00
2.03
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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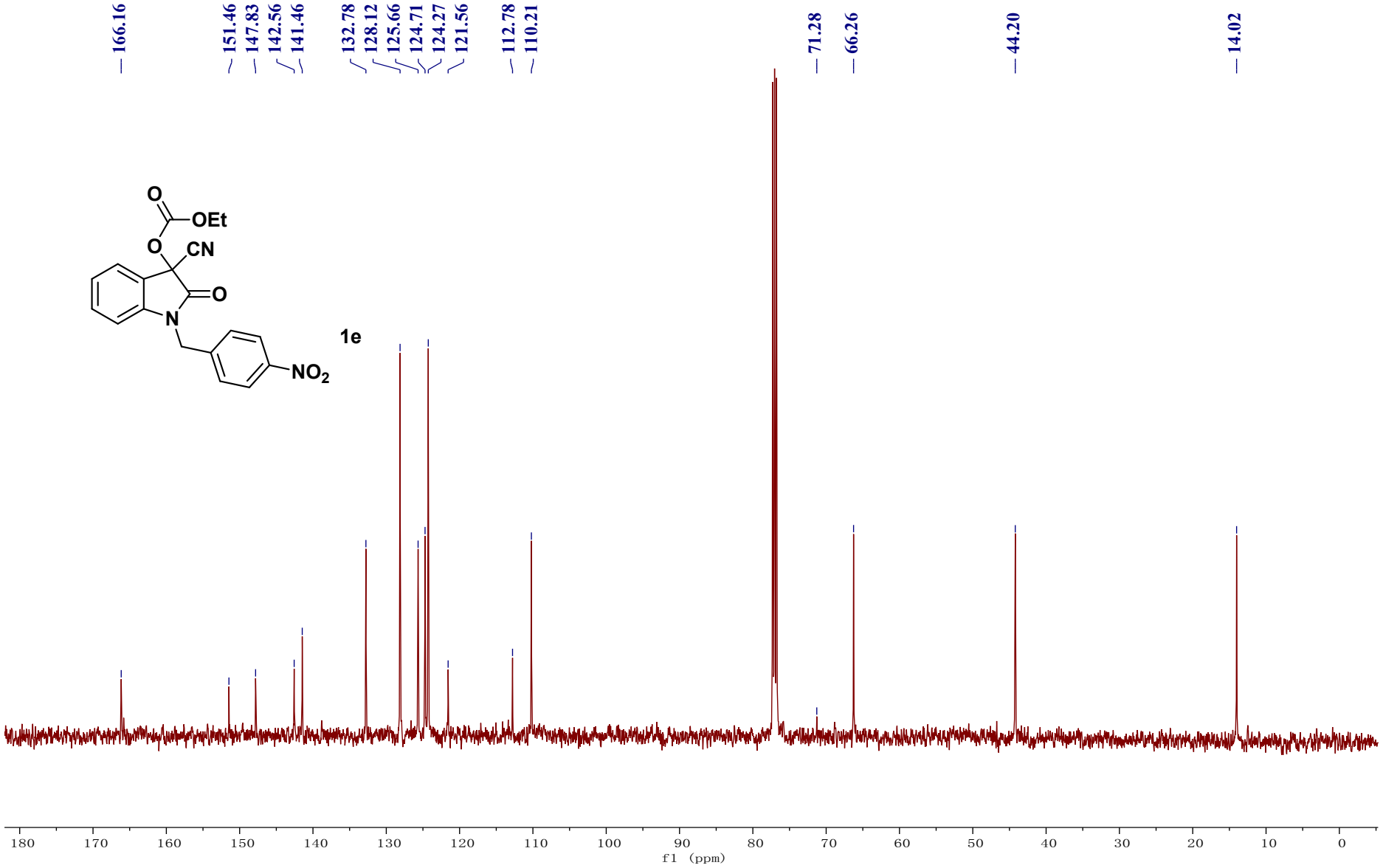
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124.27
121.56
112.78
110.21
71.28
66.26
44.20
14.02
O
OEt
O
CN
O
N
NO2
1e
180
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100
90
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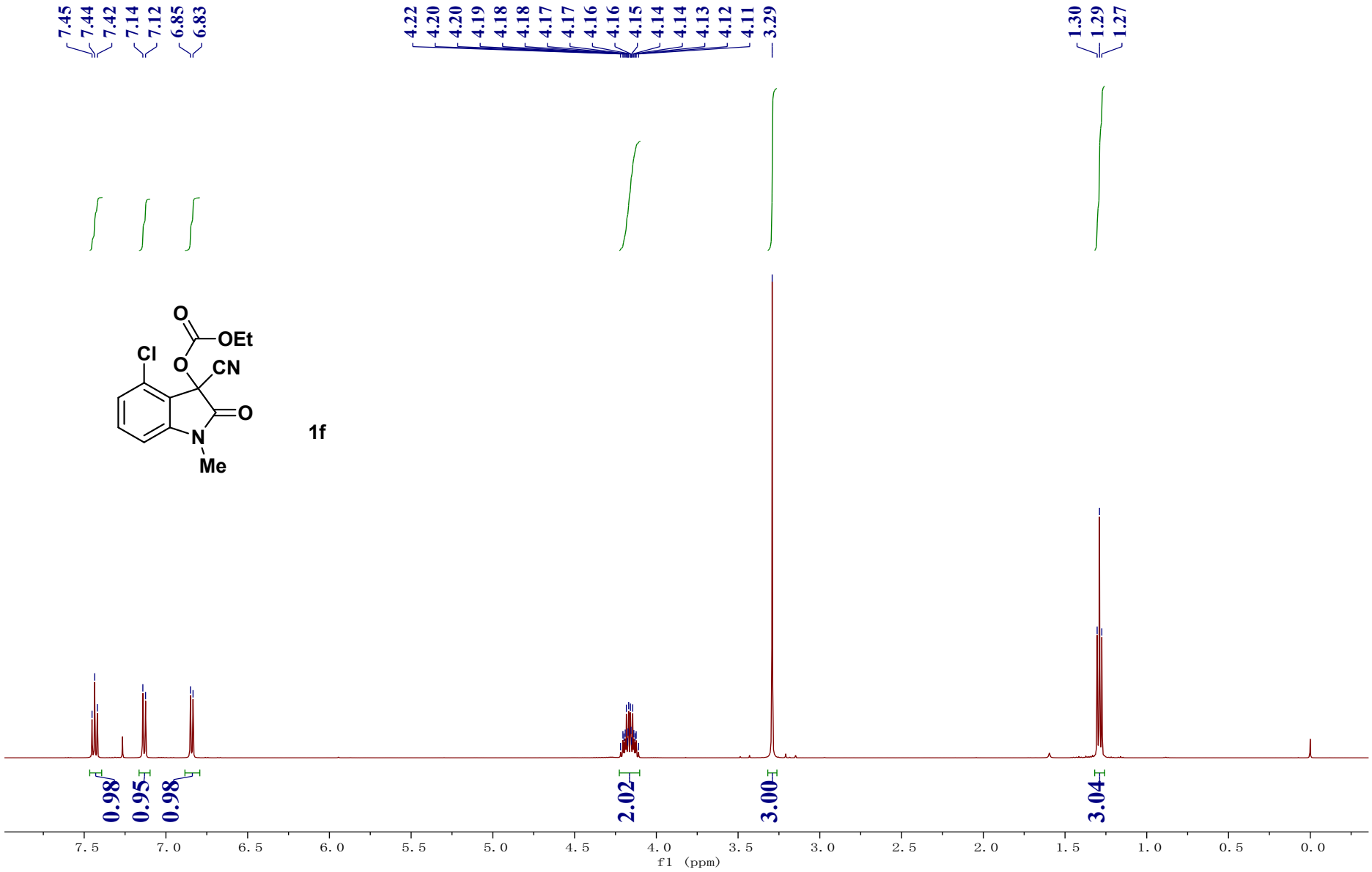
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4.17
4.17
4.16
4.16
4.15
4.14
4.14
4.13
4.12
4.11
3.29
1.30
1.29
1.27
O
OEt
Cl
O
CN
O
N
Me
1f
0.98
0.95
0.98
2.02
3.00
3.04
7.5
7.0
6.5
6.0
5.5
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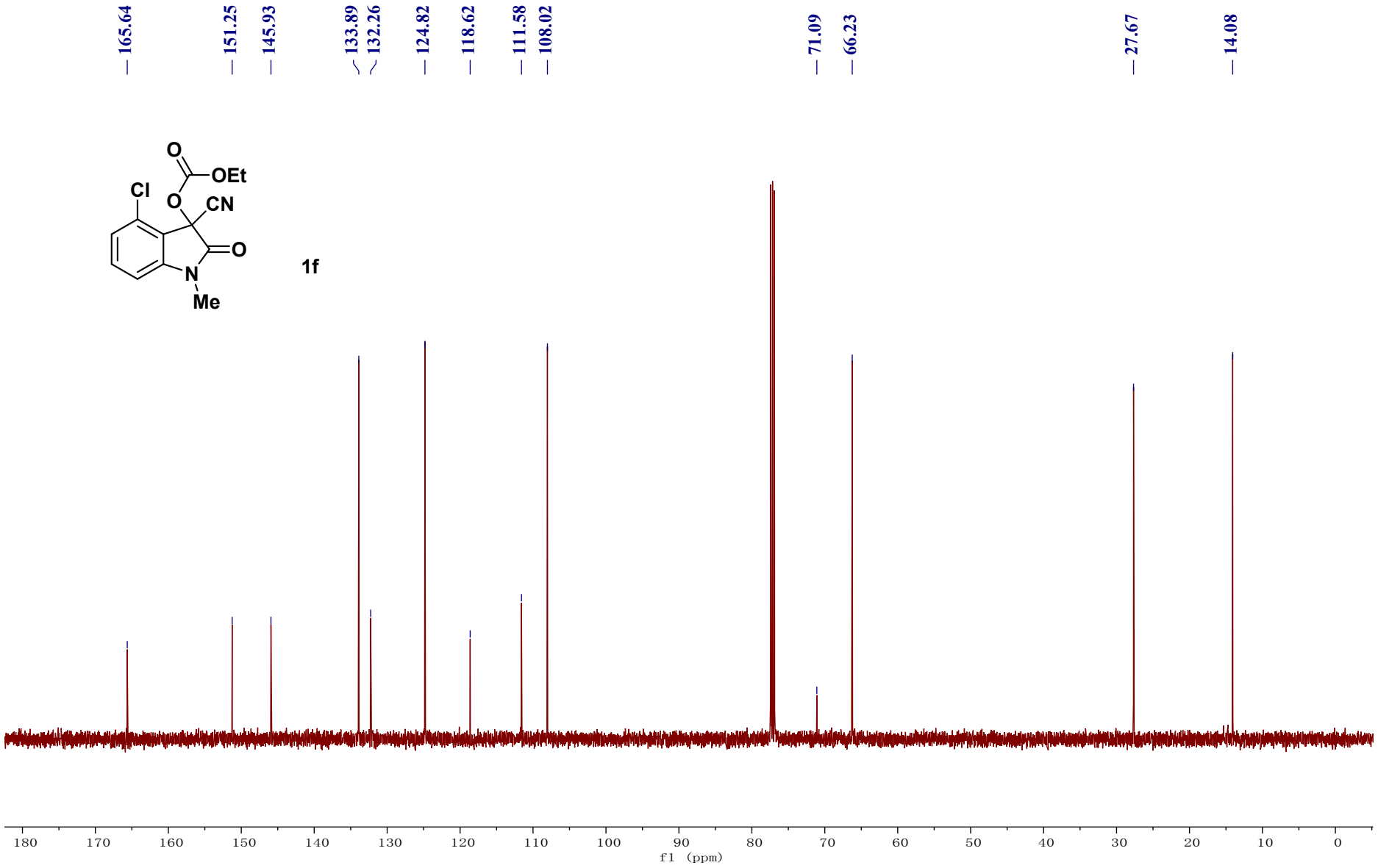
f1 (ppm)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14.08
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Cl
O
CN
O
N
Me
1f
180
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100
90
80
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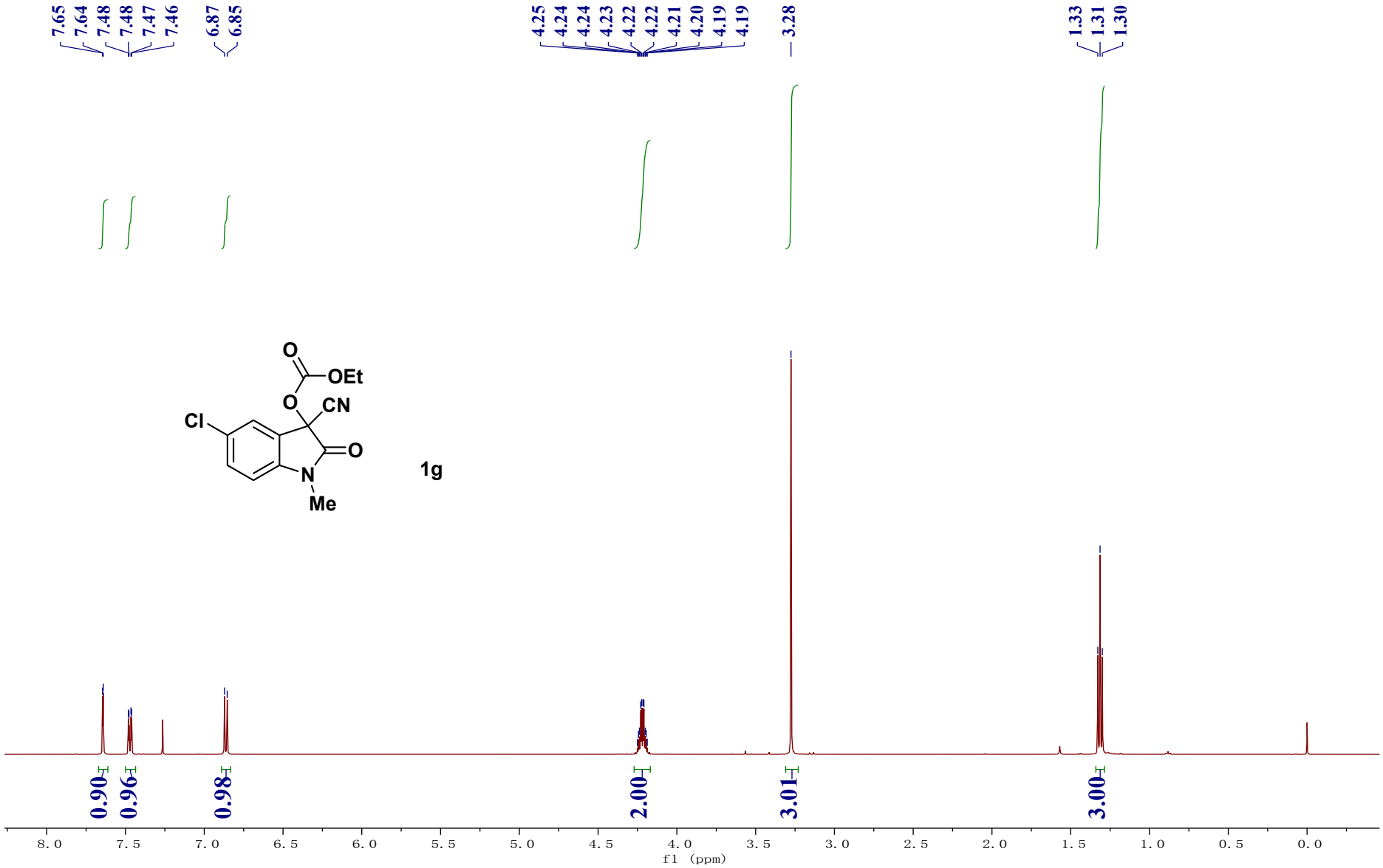
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4.22
4.21
4.20
4.19
4.19
3.28
1.33
1.31
1.30
O
OEt
O
CN
Cl
O
N
Me
1g
0.90
0.96
0.98
2.00
3.01
3.00
8.0
7.5
7.0
6.5
6.0
5.5
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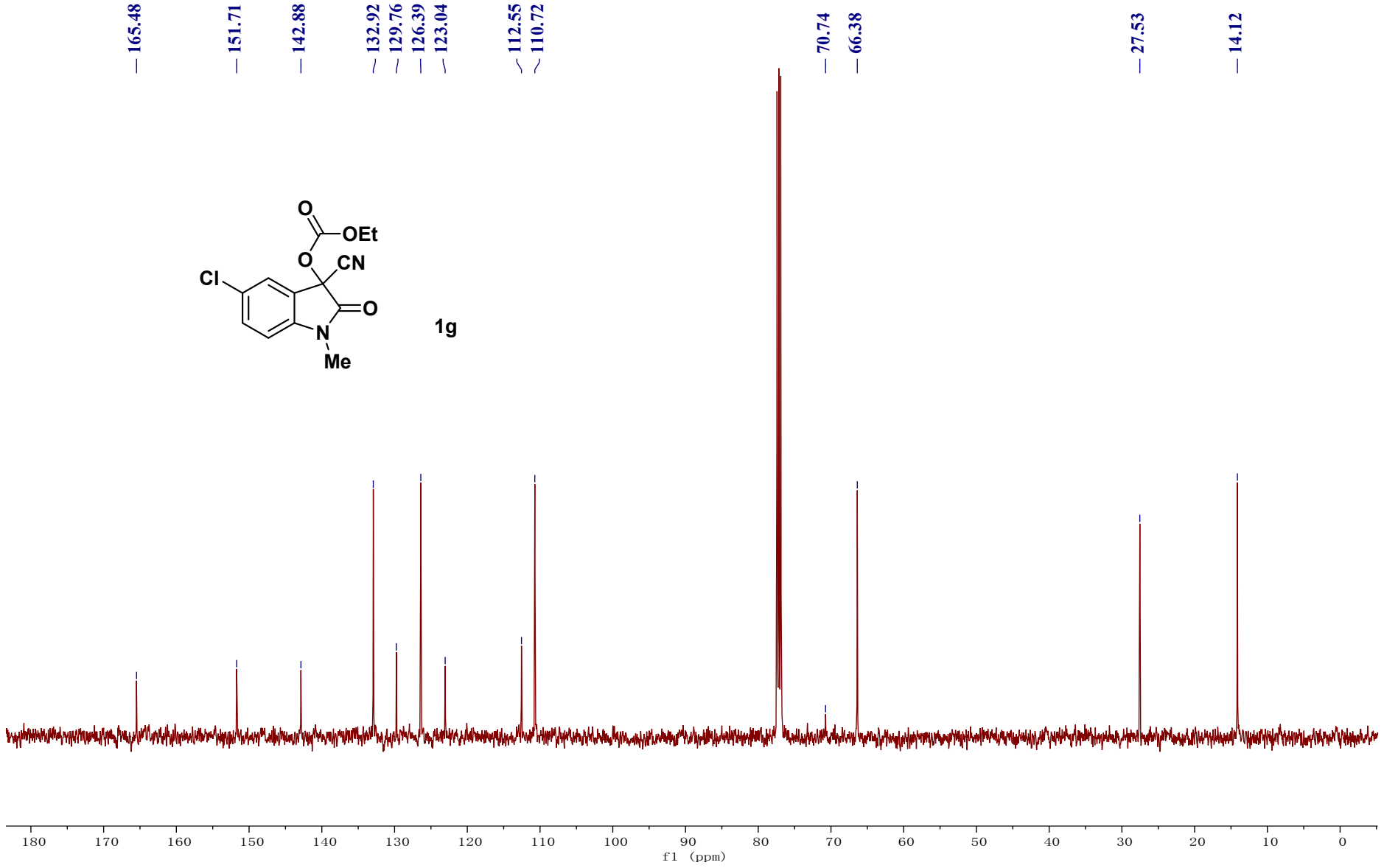
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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126.39
123.04
112.55
110.72
70.74
66.38
27.53
14.12
O
OEt
O
CN
Cl
O
N
Me
1g
180
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110
100
90
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50
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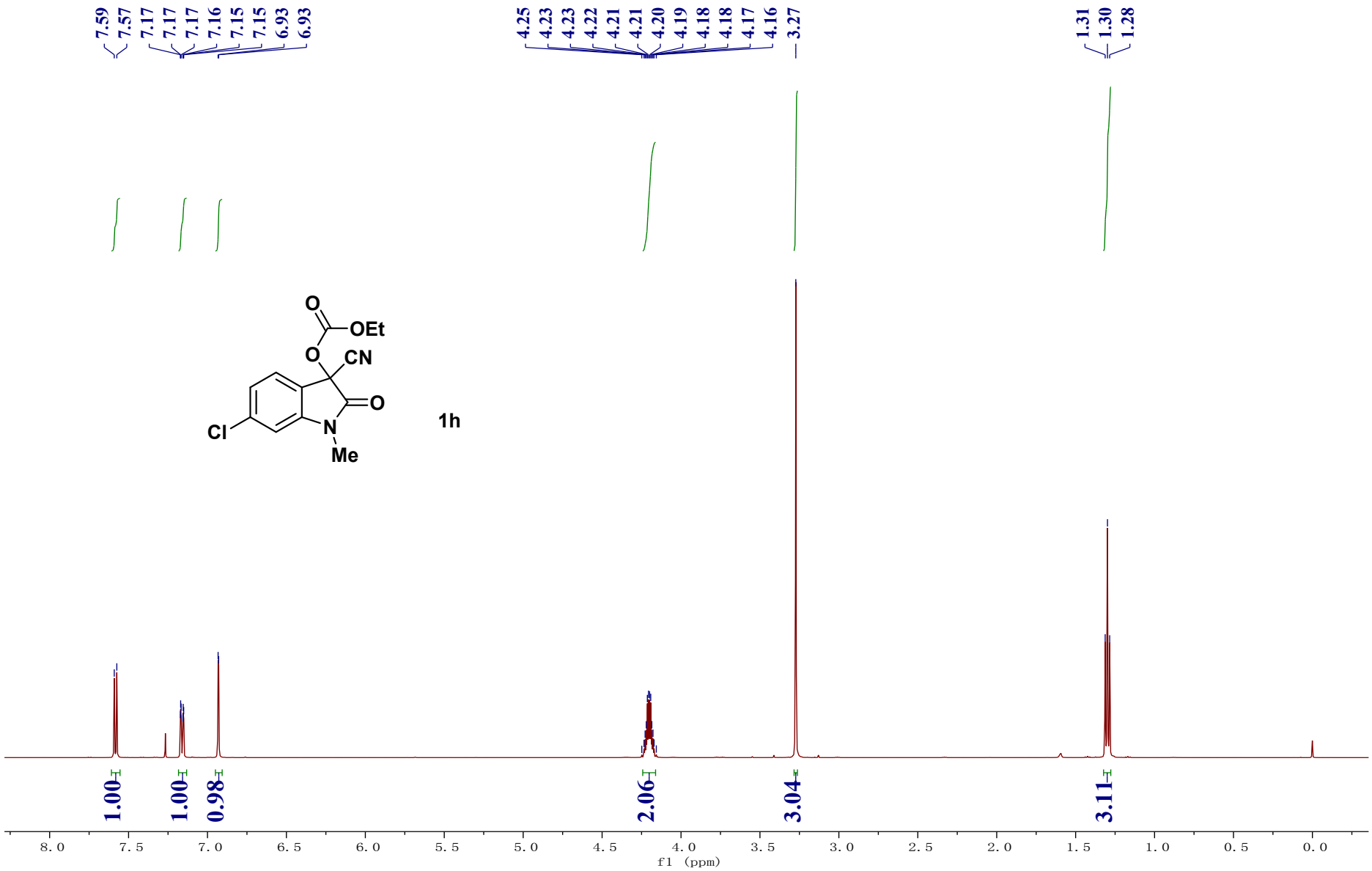
f1 (ppm)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4.21
4.21
4.20
4.19
4.18
4.18
4.17
4.16
3.27
1.31
1.30
1.28
O
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O
CN
O
Cl
N
Me
1h
1.00
1.00
0.98
2.06
3.04
3.11
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7.0
6.5
6.0
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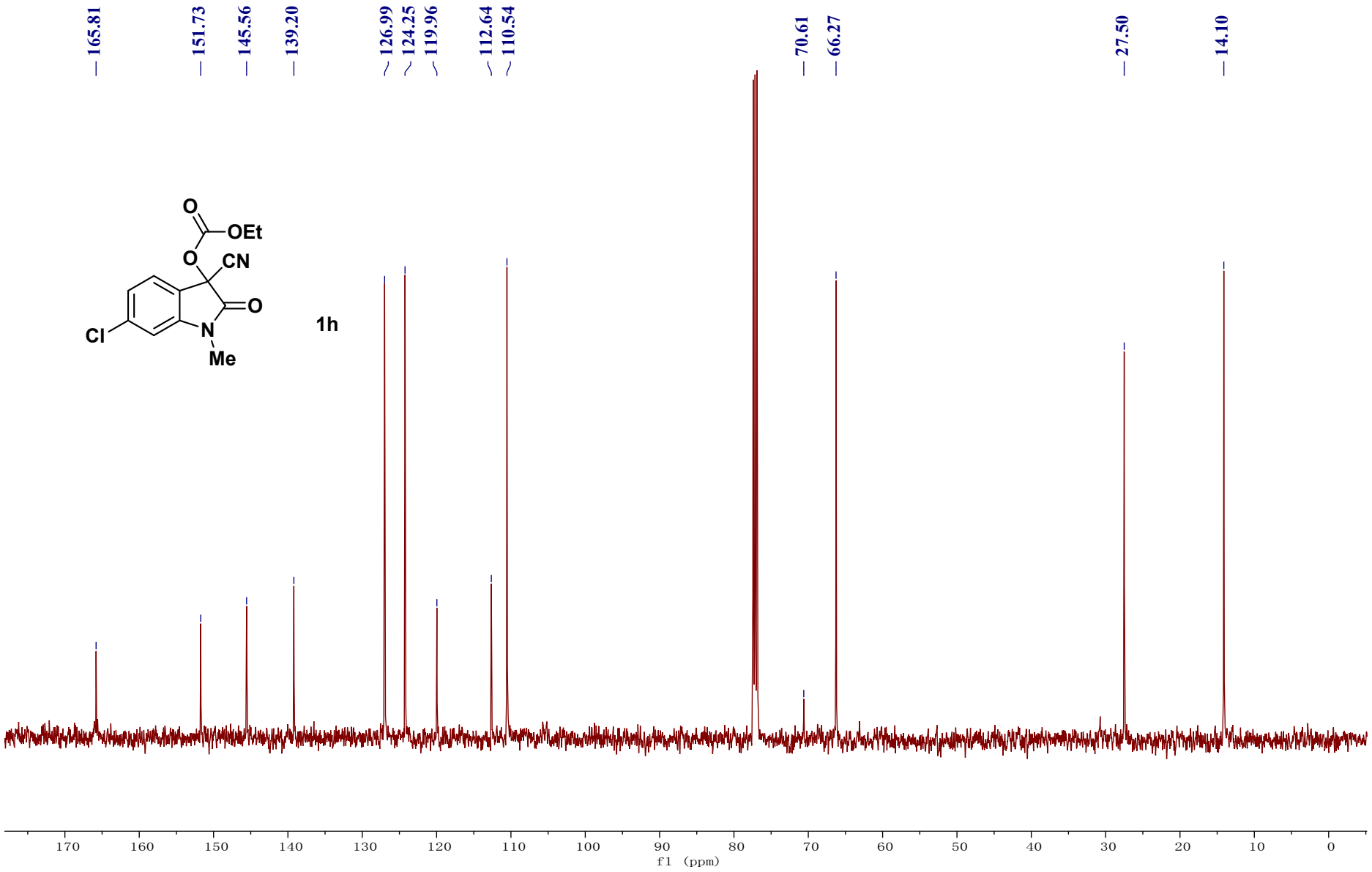
f1 (ppm)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66.27
27.50
14.10
O
OEt
O
CN
O
Cl
N
Me
1h
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120
110
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90
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50
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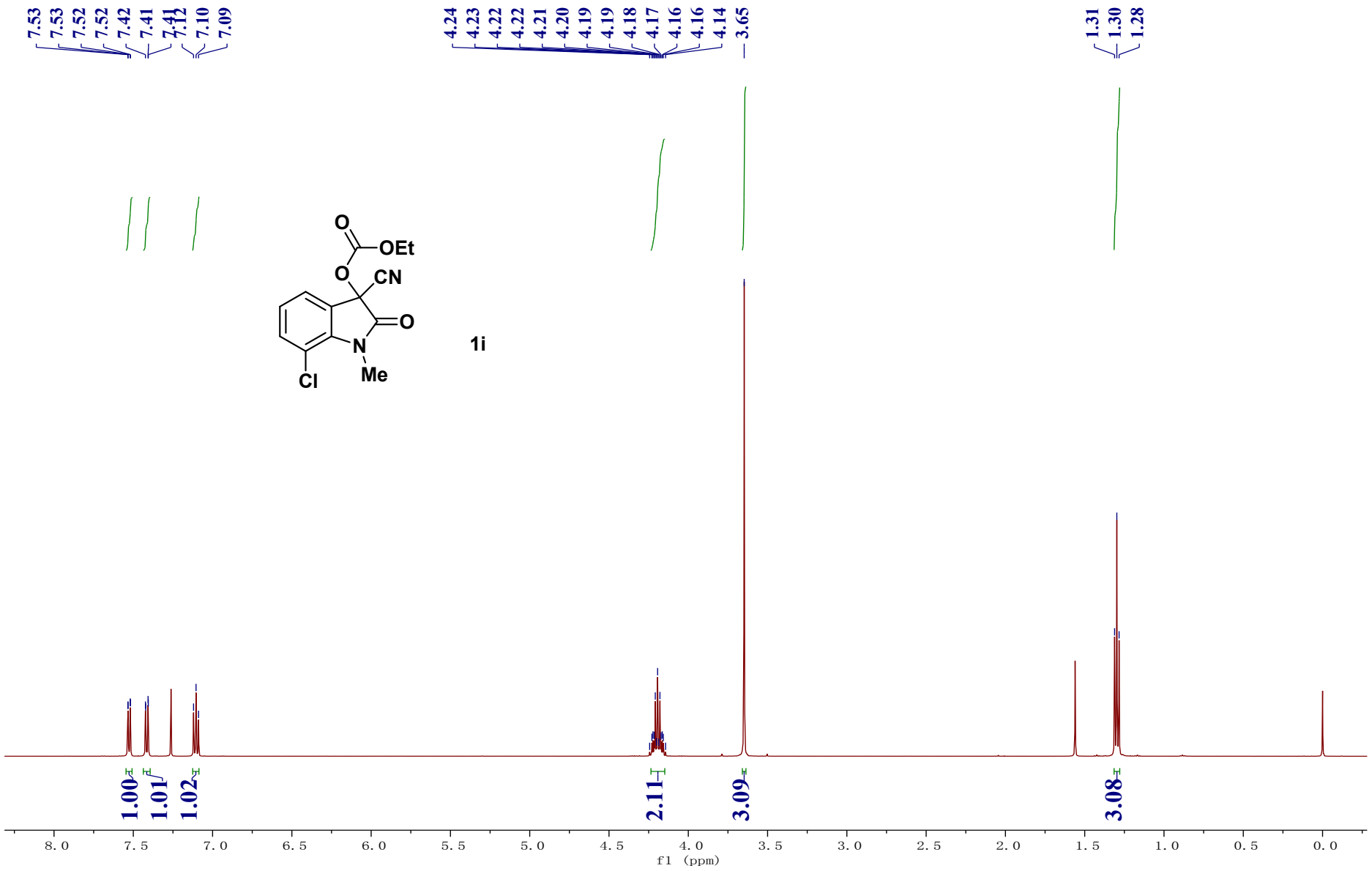
f1 (ppm)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4.22
4.21
4.20
4.19
4.19
4.18
4.17
4.16
4.16
4.14
3.65
1.31
1.30
1.28
O
OEt
O
CN
O
N
Me
Cl
1i
1.00
1.01
1.02
2.11
3.09
3.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
3.0
2.5
2.0
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1.0
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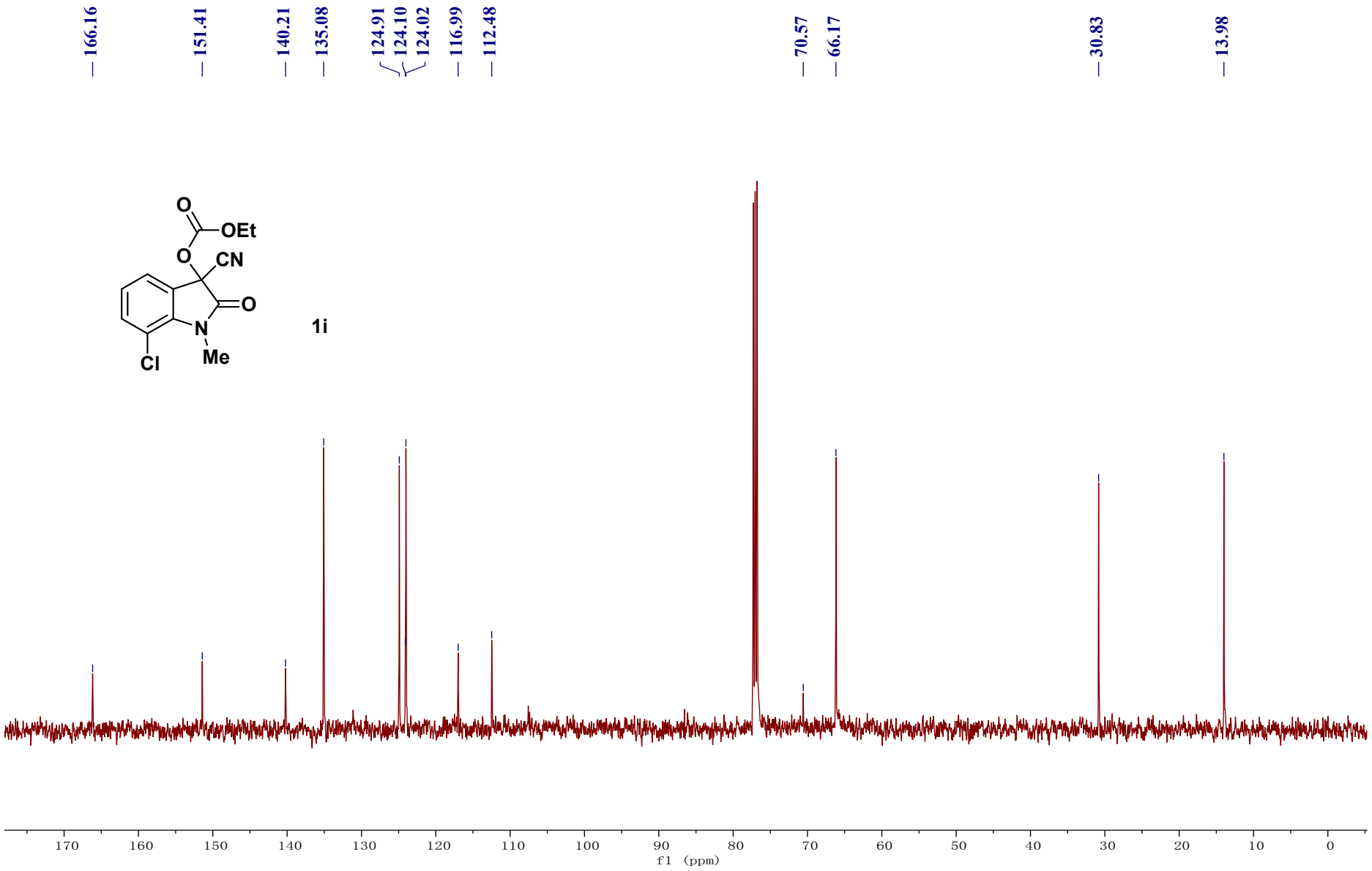
0.0
f1 (ppm)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70.57
66.17
30.83
13.98
O
OEt
O
CN
O
N
Me
Cl
1i
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130
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110
100
90
80
70
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50
40
30
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10
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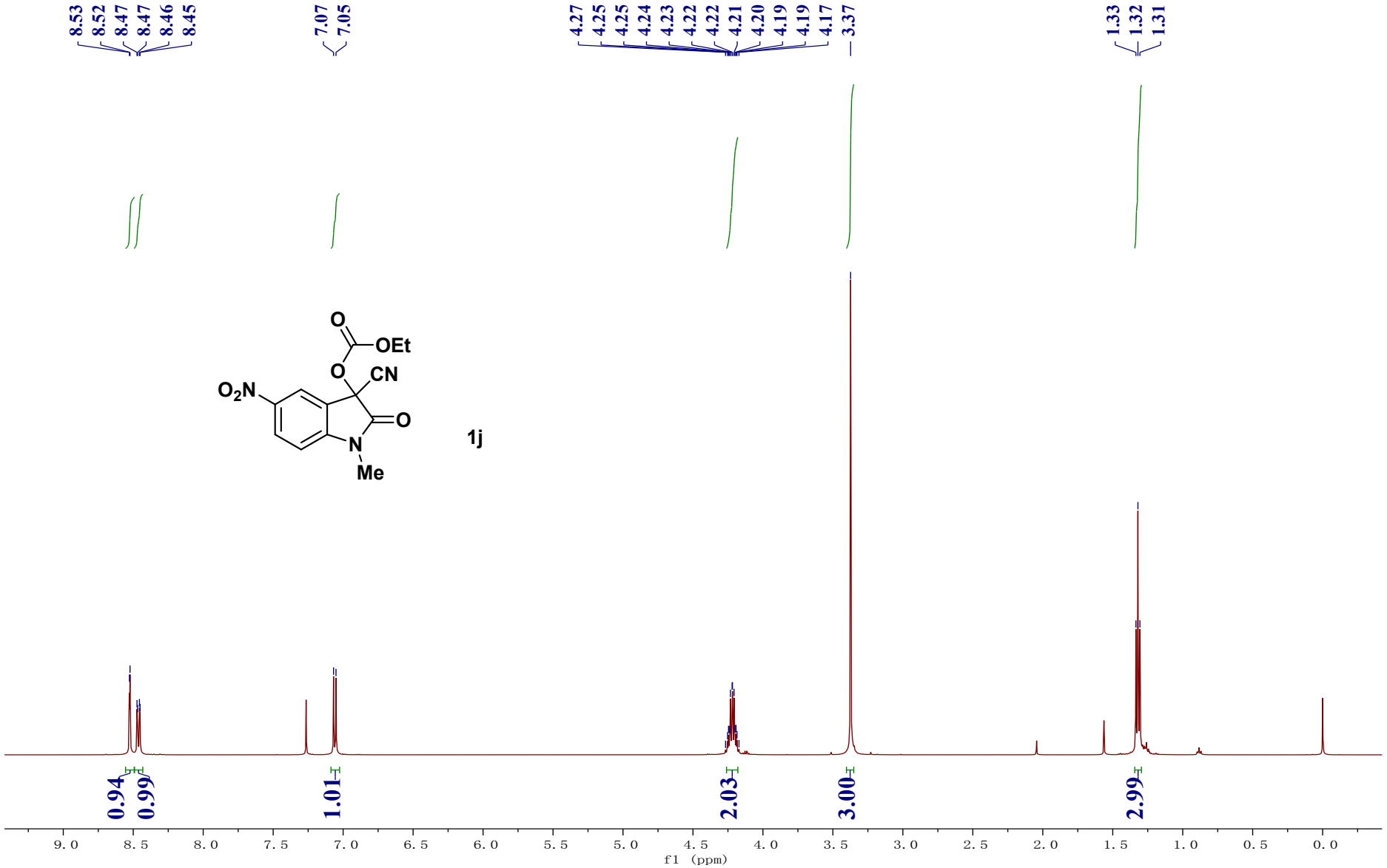
f1 (ppm)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4.24
4.23
4.22
4.22
4.21
4.20
4.19
4.19
4.17
3.37
1.33
1.32
1.31
O
OEt
O
CN
O2N
O
N
Me
1j
0.94
0.99
1.01
2.03
3.00
2.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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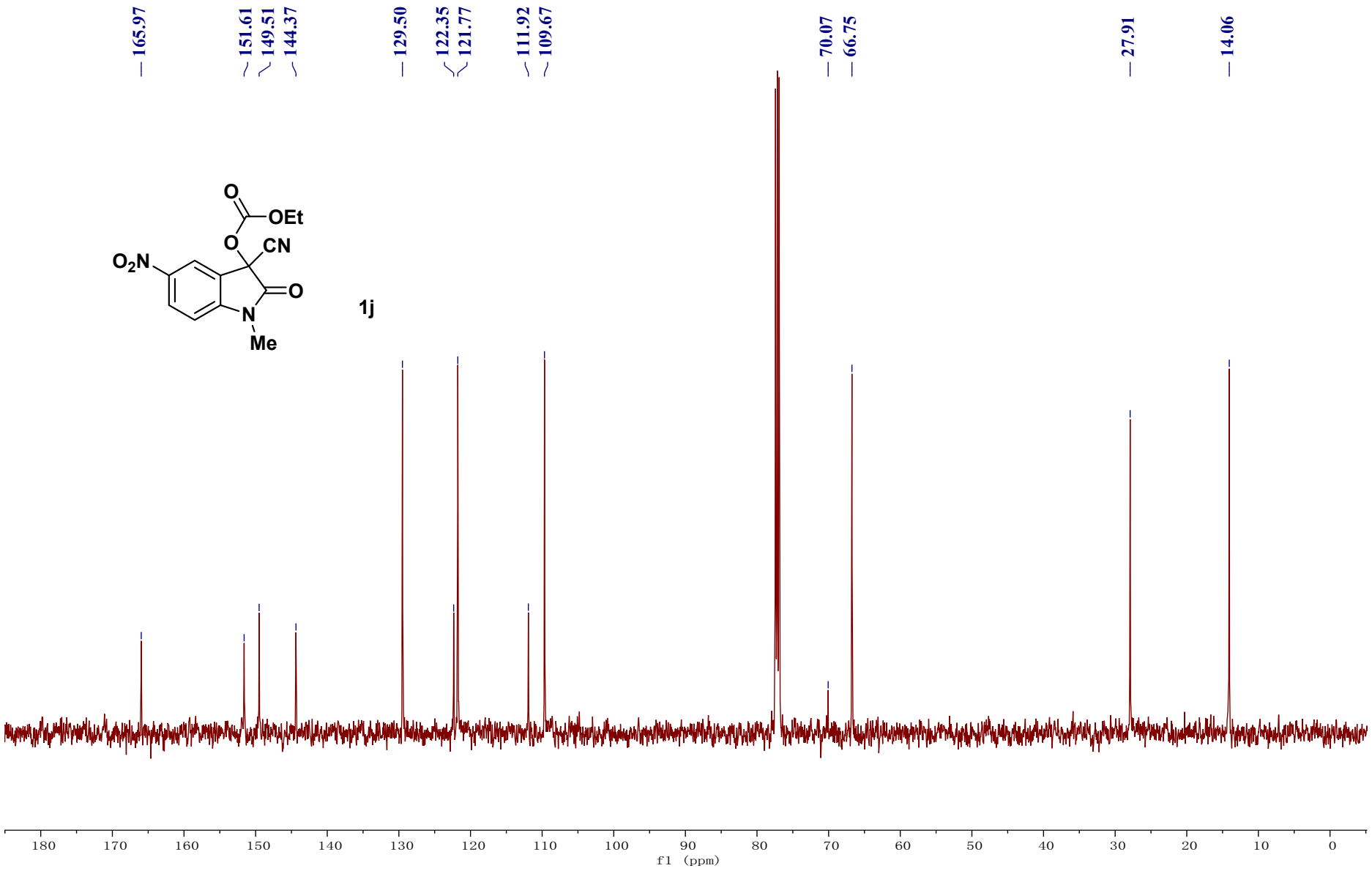
0.0
f1 (ppm)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111.92
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70.07
66.75
27.91
14.06
O
OEt
O
CN
O2N
O
N
Me
1j
180
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130
120
110
100
90
80
70
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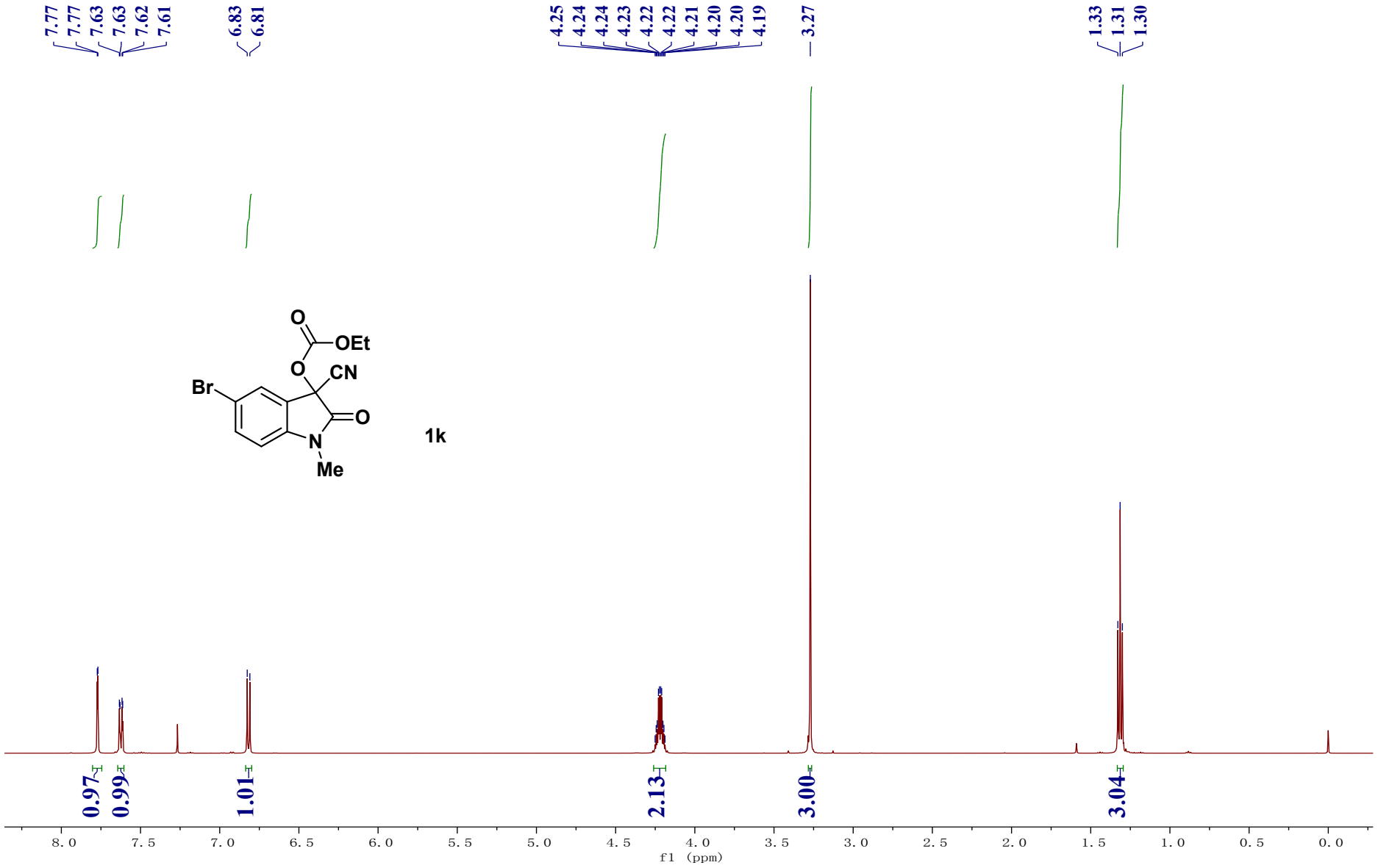
50
40
30
20
10
0
f1 (ppm)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4.22
4.21
4.20
4.20
4.19
3.27
1.33
1.31
1.30
O
OEt
O
CN
Br
O
N
Me
1k
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0.99
1.01
2.13
3.00
3.04
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
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1.0
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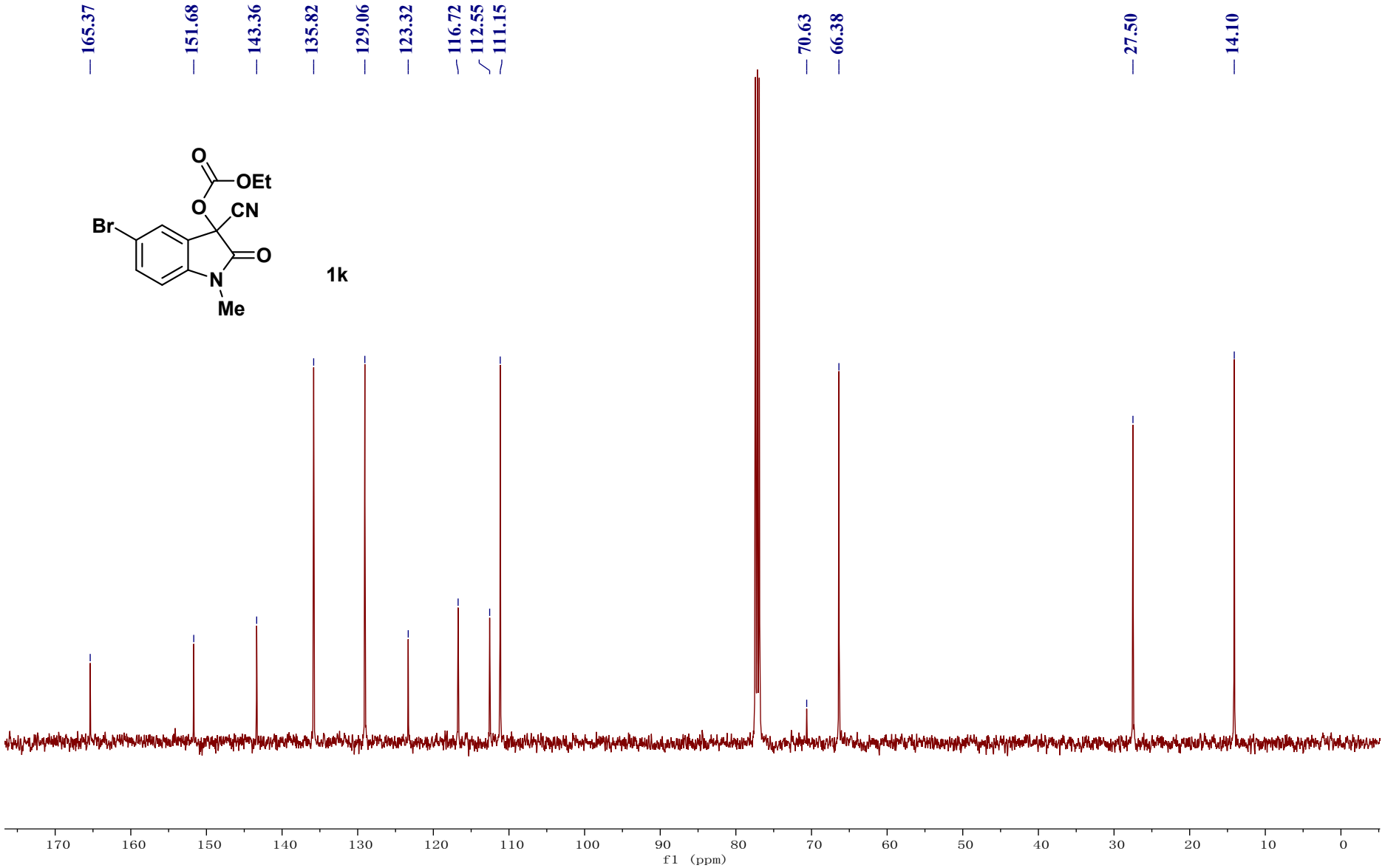
f1 (ppm)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66.38
27.50
14.10
O
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O
CN
Br
O
N
Me
1k
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130
120
110
100
90
80
70
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50
40
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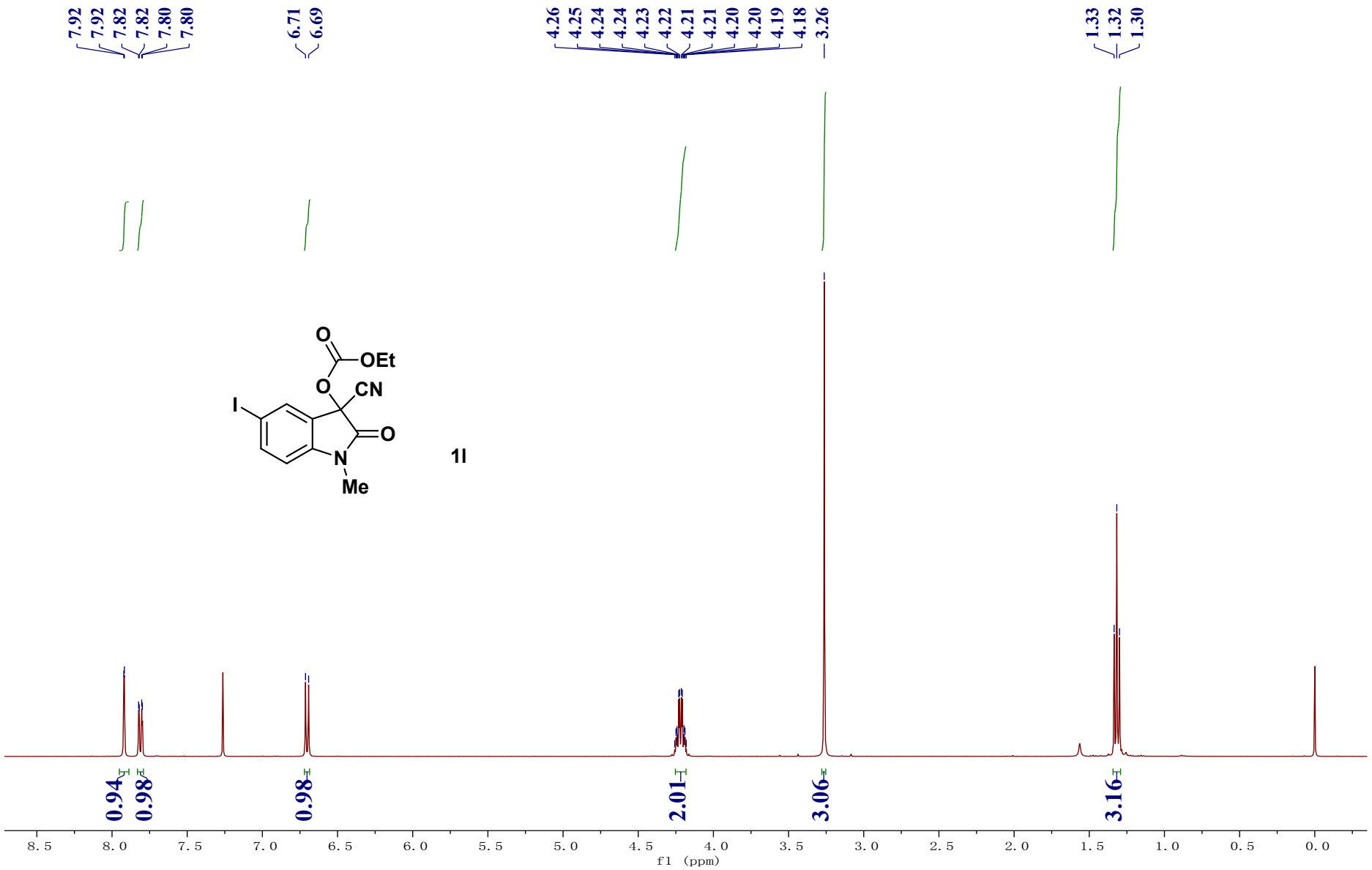
10
0
f1 (ppm)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4.23
4.22
4.21
4.21
4.20
4.20
4.19
4.18
3.26
1.33
1.32
1.30
O
OEt
O
CN
I
O
N
Me
1l
0.94
0.98
0.98
2.01
3.06
3.16
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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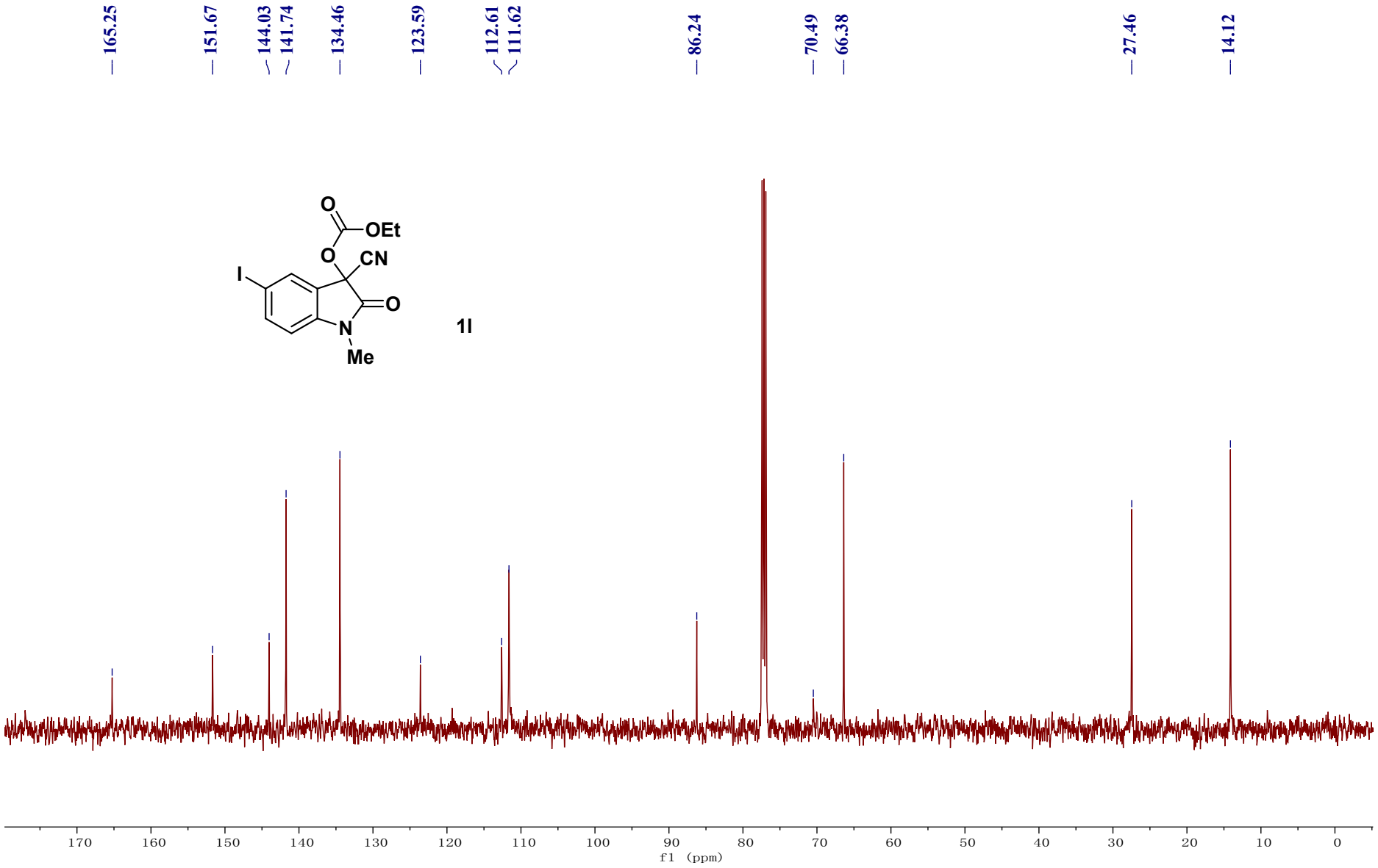
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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66.38
27.46
14.12
O
OEt
O
CN
I
O
N
Me
1l
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110
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90
80
70
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50
40
30
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10
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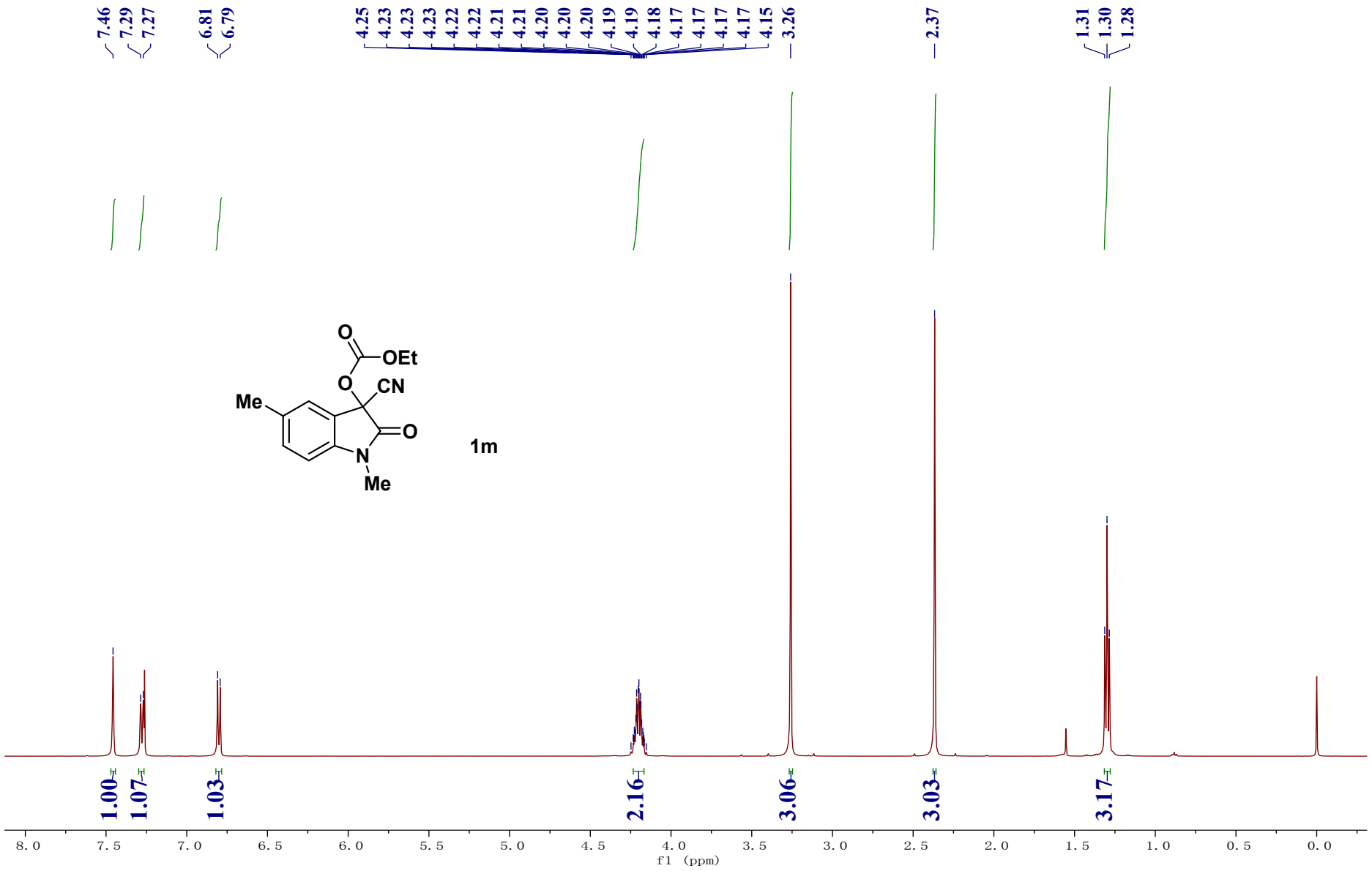
f1 (ppm)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4.20
4.20
4.19
4.19
4.18
4.17
4.17
4.17
4.17
4.15
3.26
2.37
1.31
1.30
1.28
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O
CN
Me
O
N
Me
1m
1.00
1.07
1.03
2.16
3.06
3.03
3.17
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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1.0
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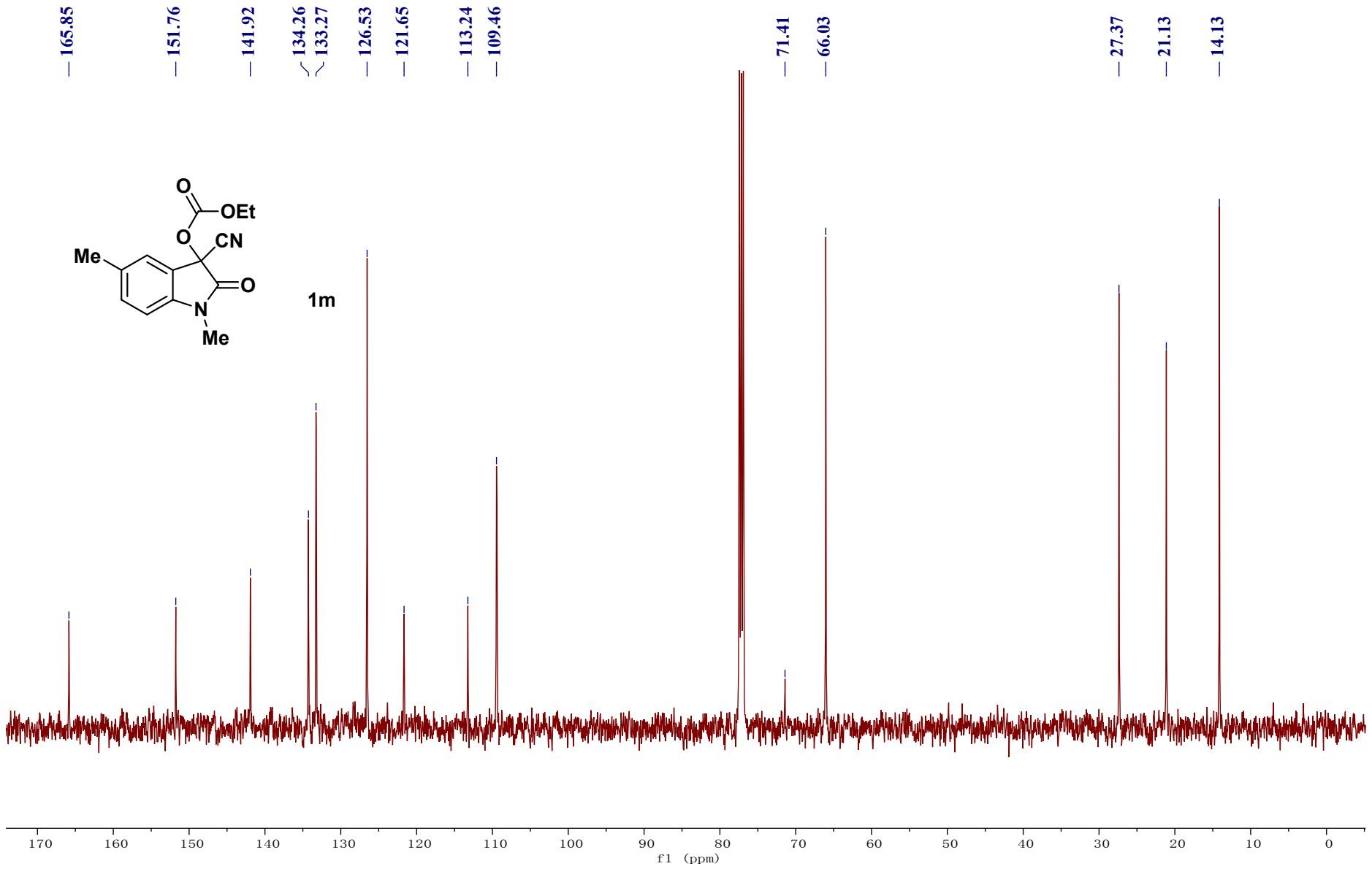
f1 (ppm)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21.13
14.13
O
OEt
O
CN
Me
O
N
Me
1m
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130
120
110
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90
80
70
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50
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10
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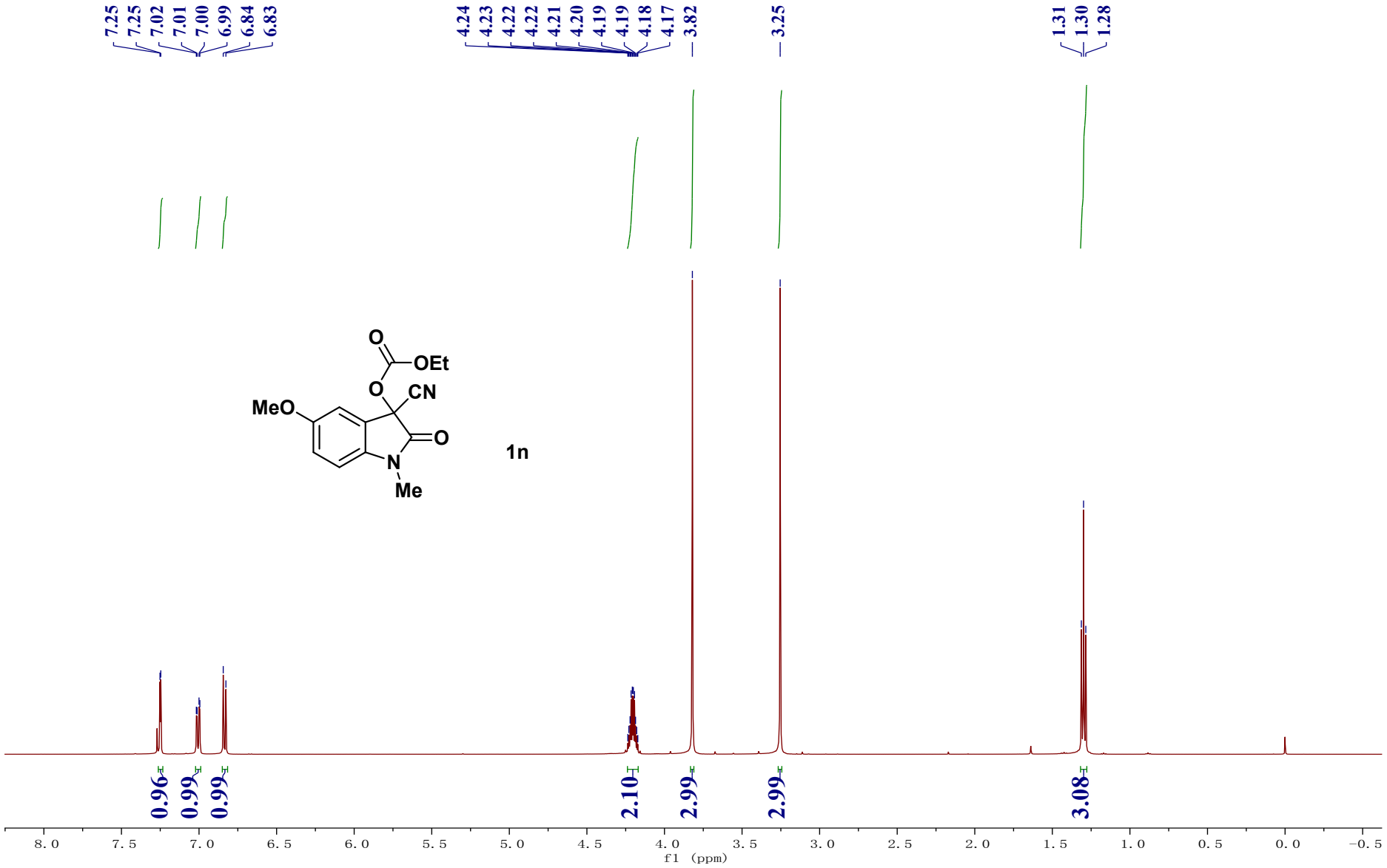
f1 (ppm)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4.22
4.21
4.20
4.19
4.19
4.18
4.17
3.82
3.25
1.31
1.30
1.28
O
OEt
O
CN
MeO
O
N
Me
1n
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0.99
0.99
2.10
2.99
2.99
3.08
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7.5
7.0
6.5
6.0
5.5
5.0
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2.0
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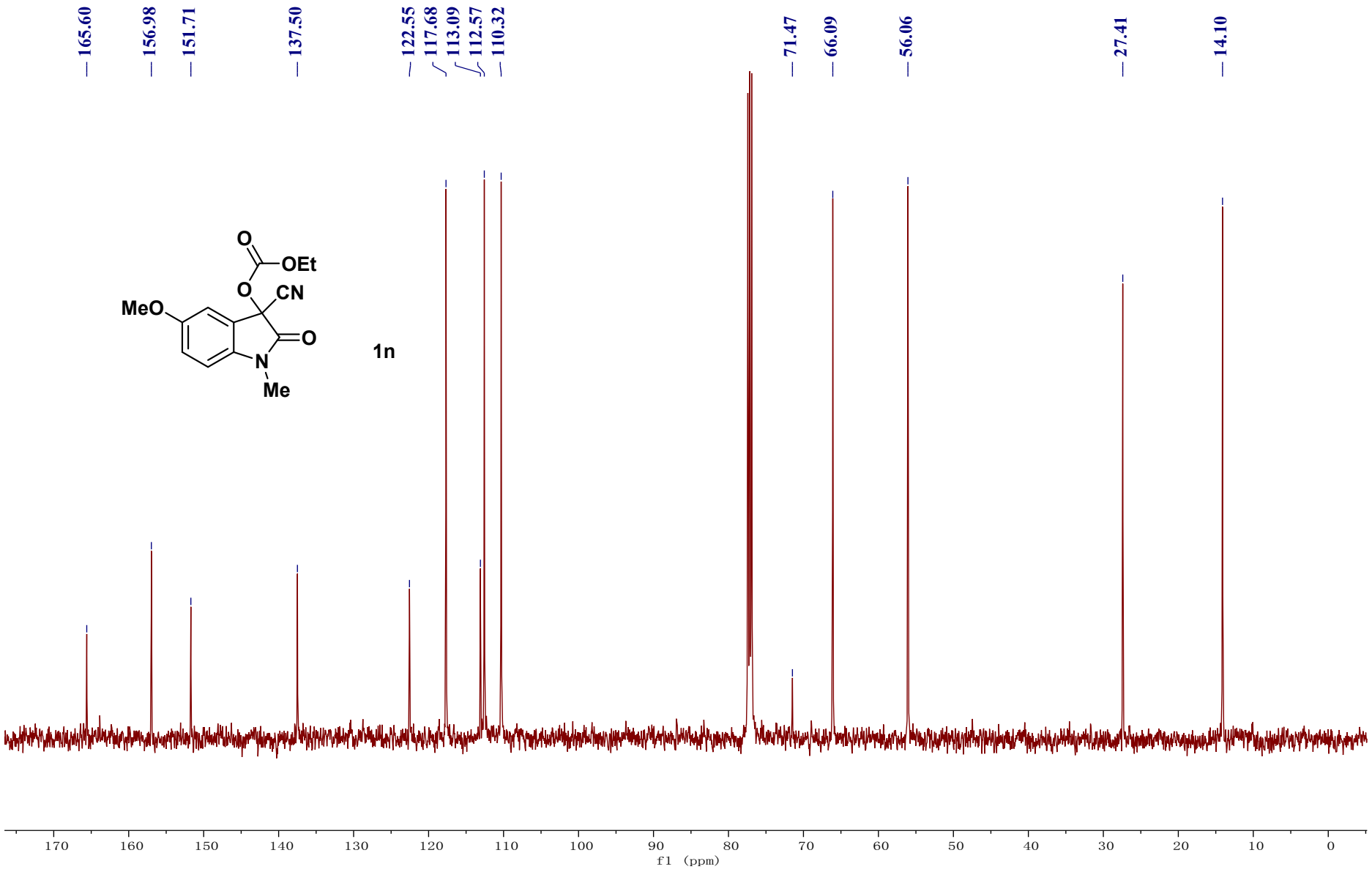
f1 (ppm)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56.06
27.41
14.10
O
OEt
O
CN
MeO
O
N
Me
1n
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100
90
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50
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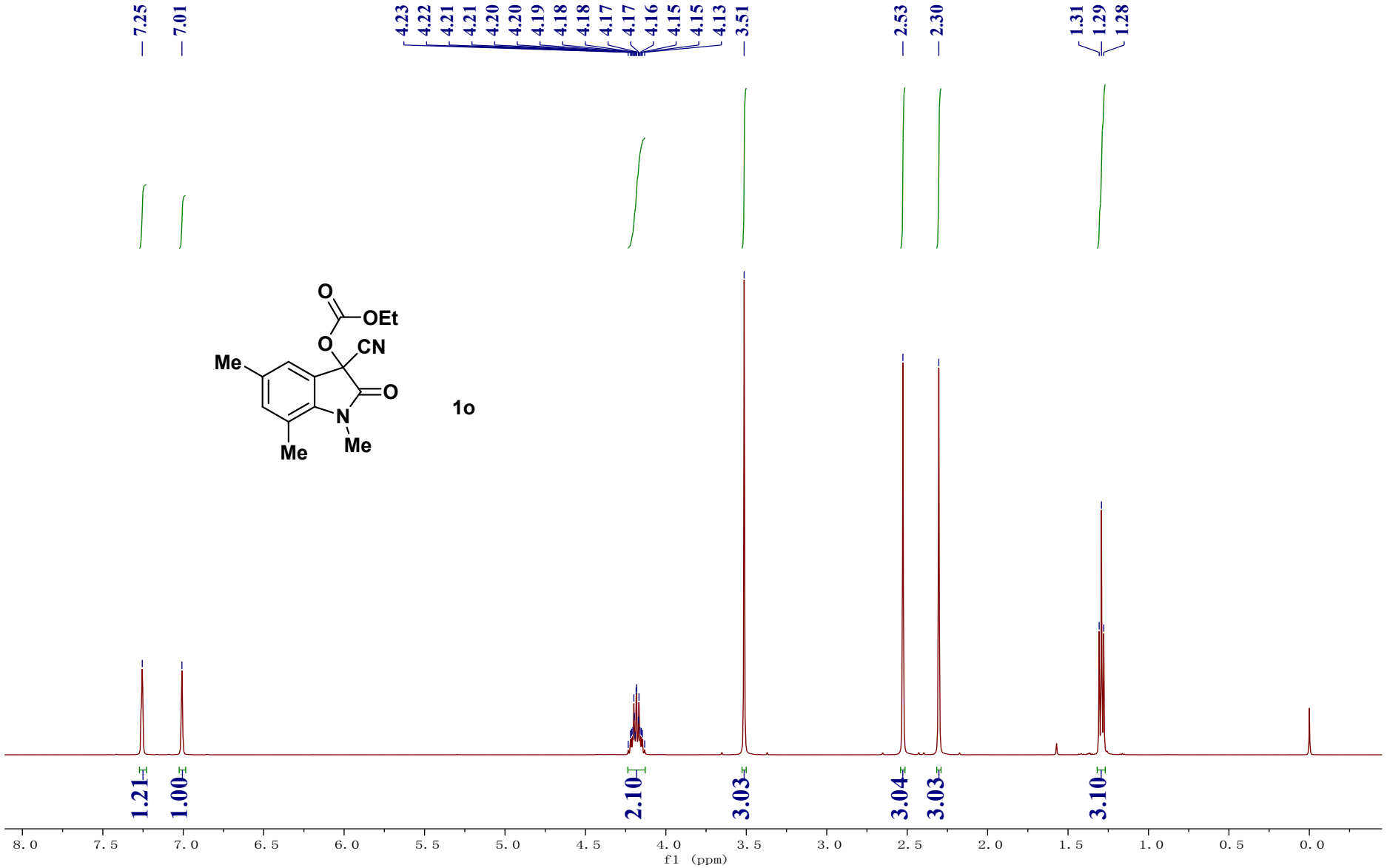
f1 (ppm)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4.16
4.15
4.15
4.13
3.51
2.53
2.30
1.31
1.29
1.28
O
OEt
O
CN
Me
O
N
Me
Me
1o
1.21
1.00
2.10
3.03
3.04
3.03
3.10
8.0
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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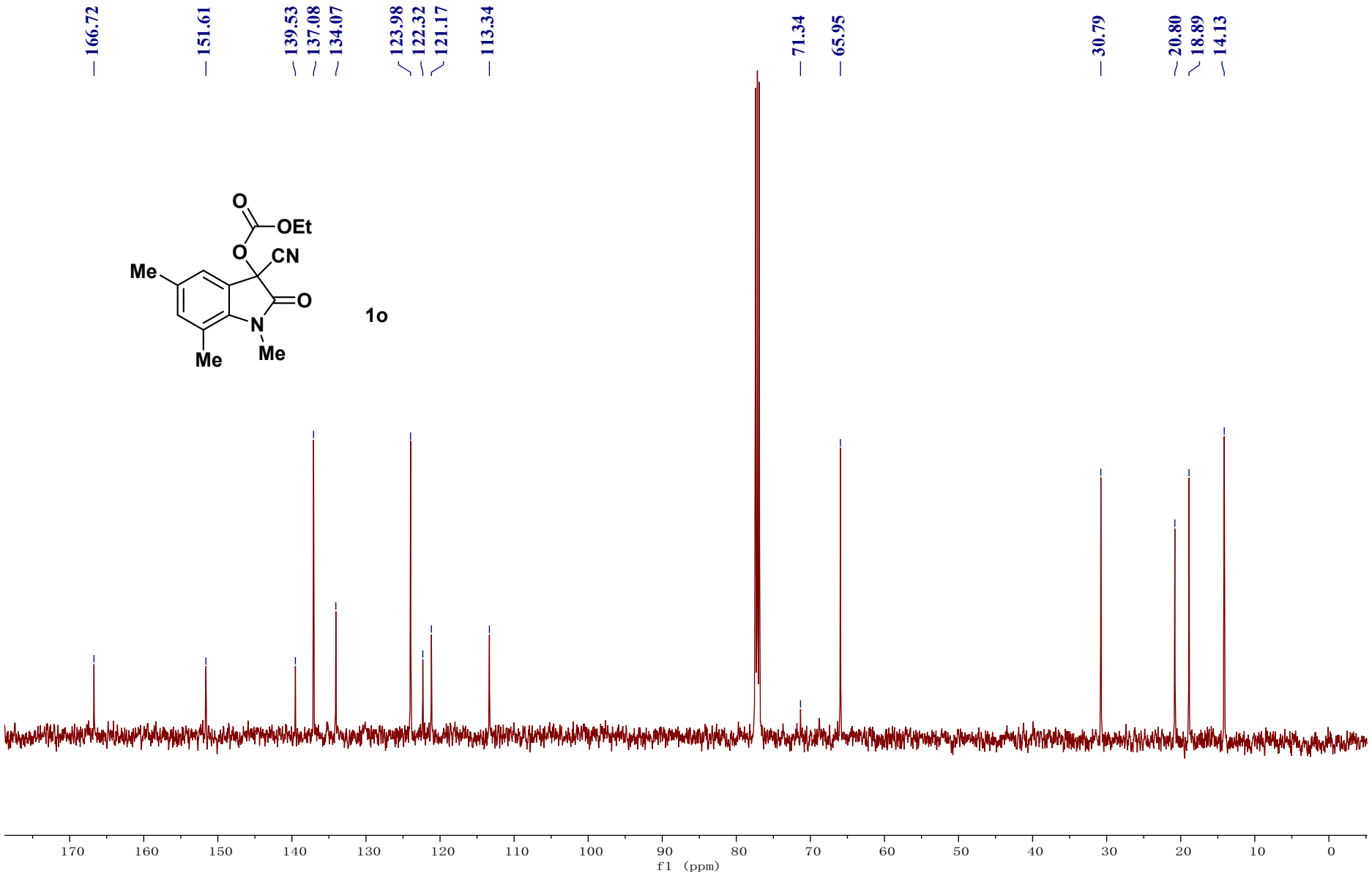
f1 (ppm)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122.32
121.17
113.34
71.34
65.95
30.79
20.80
18.89
14.13
O
OEt
O
CN
Me
O
N
Me
Me
1o
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130
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90
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10
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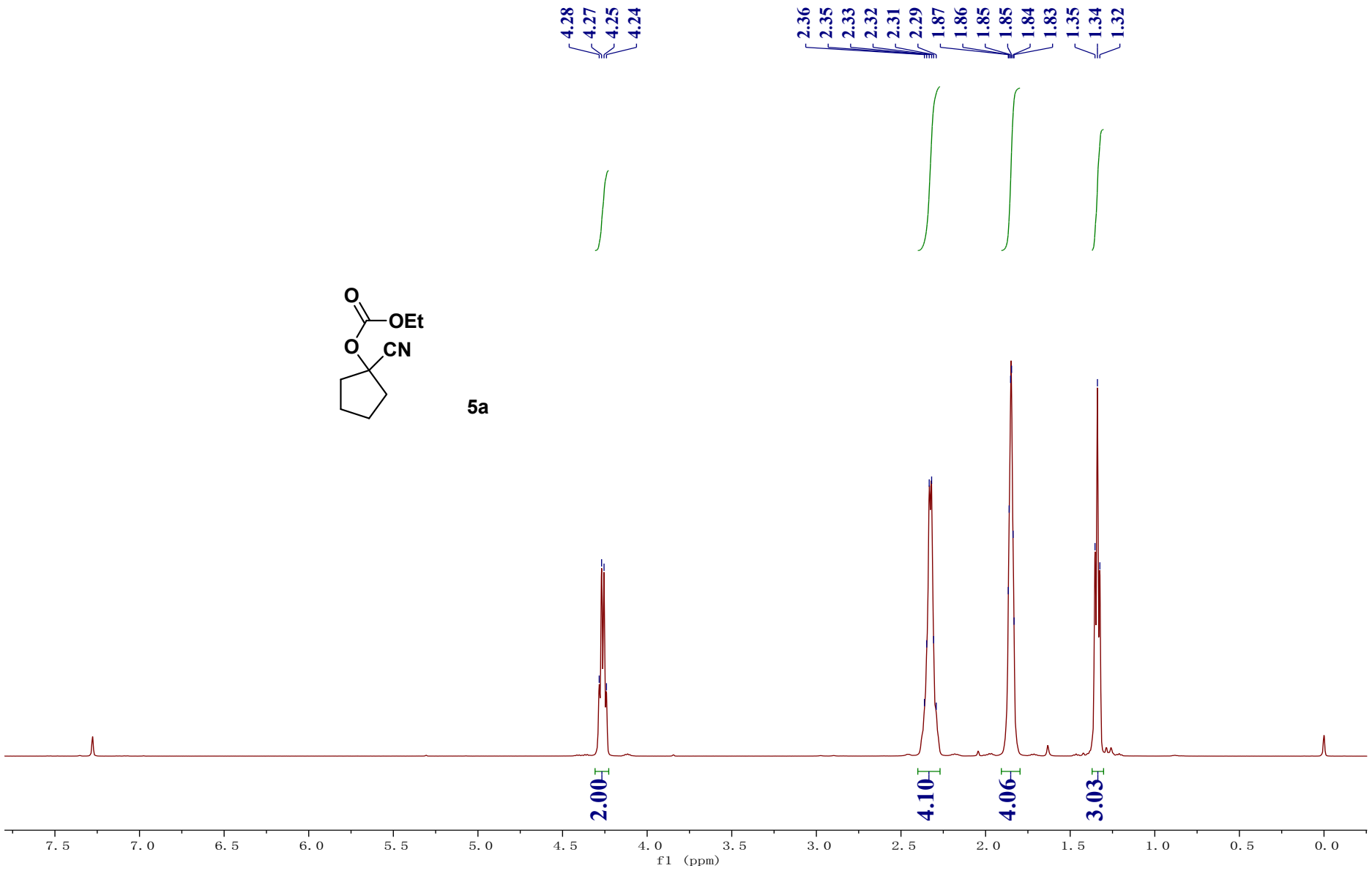
f1 (ppm)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2.29
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1.86
1.85
1.85
1.84
1.83
1.35
1.34
1.32
O
OEt
O
CN
5a
2.00
4.10
4.06
3.03
7.5
7.0
6.5
6.0
5.5
5.0
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2.5
2.0
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1.0
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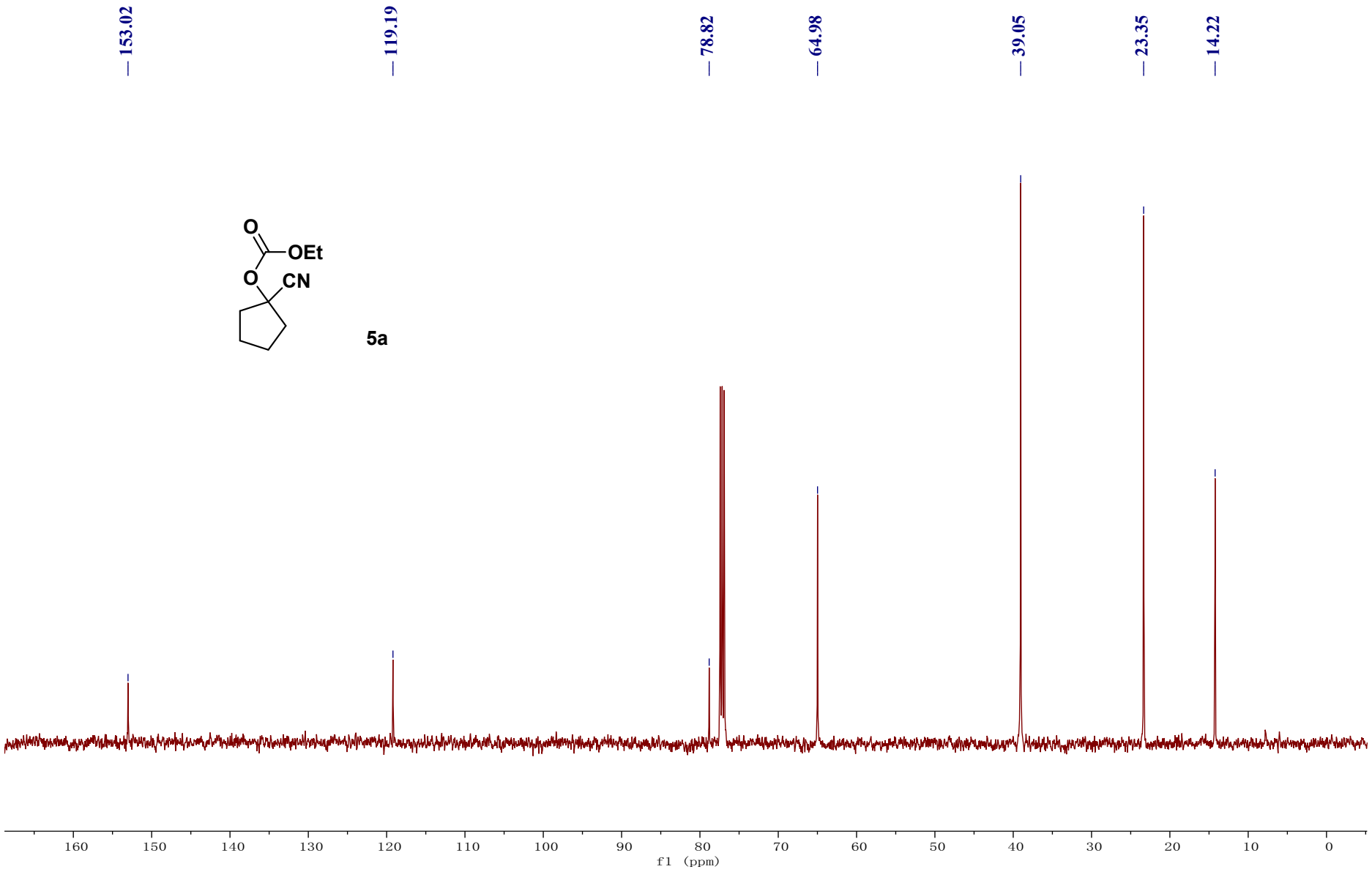
0.0
f1 (ppm)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23.35
14.22
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OEt
O
CN
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10
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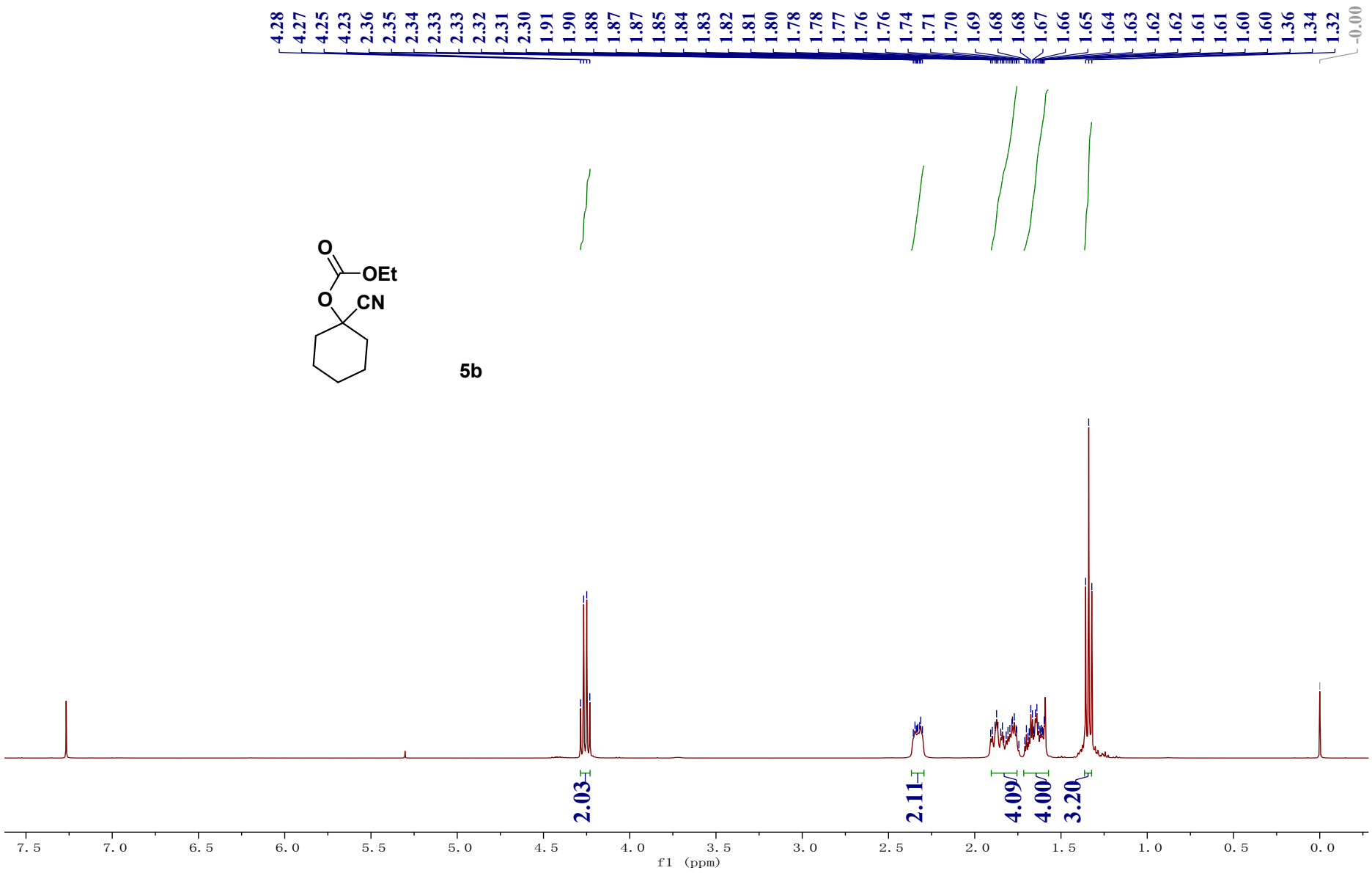
f1 (ppm)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O
OEt
O
CN
5b
2.03
2.11
4.09
4.00
3.20
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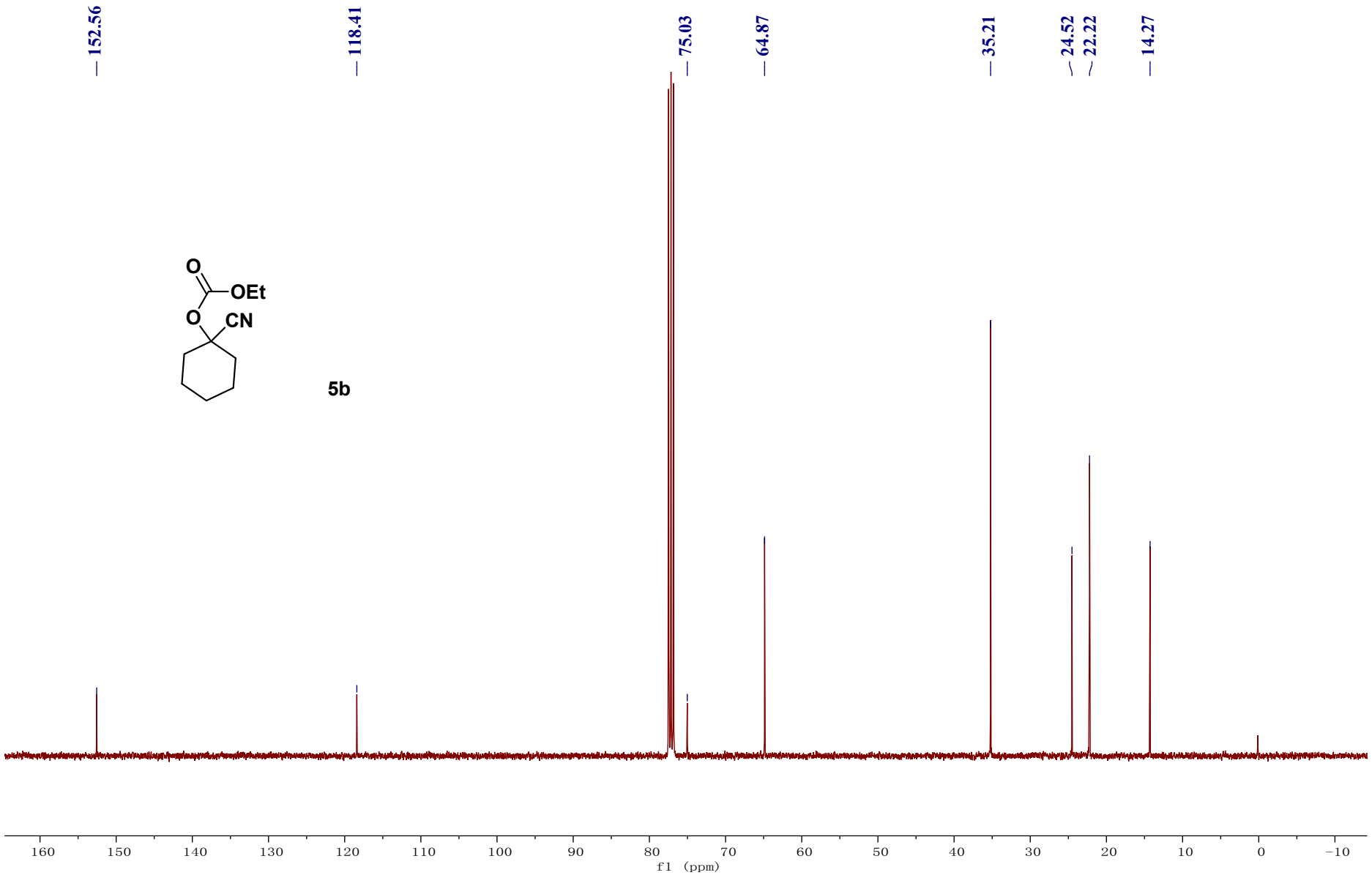
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118.41
75.03
64.87
35.21
24.52
22.22
14.27
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OEt
O
CN
5b
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120
110
100
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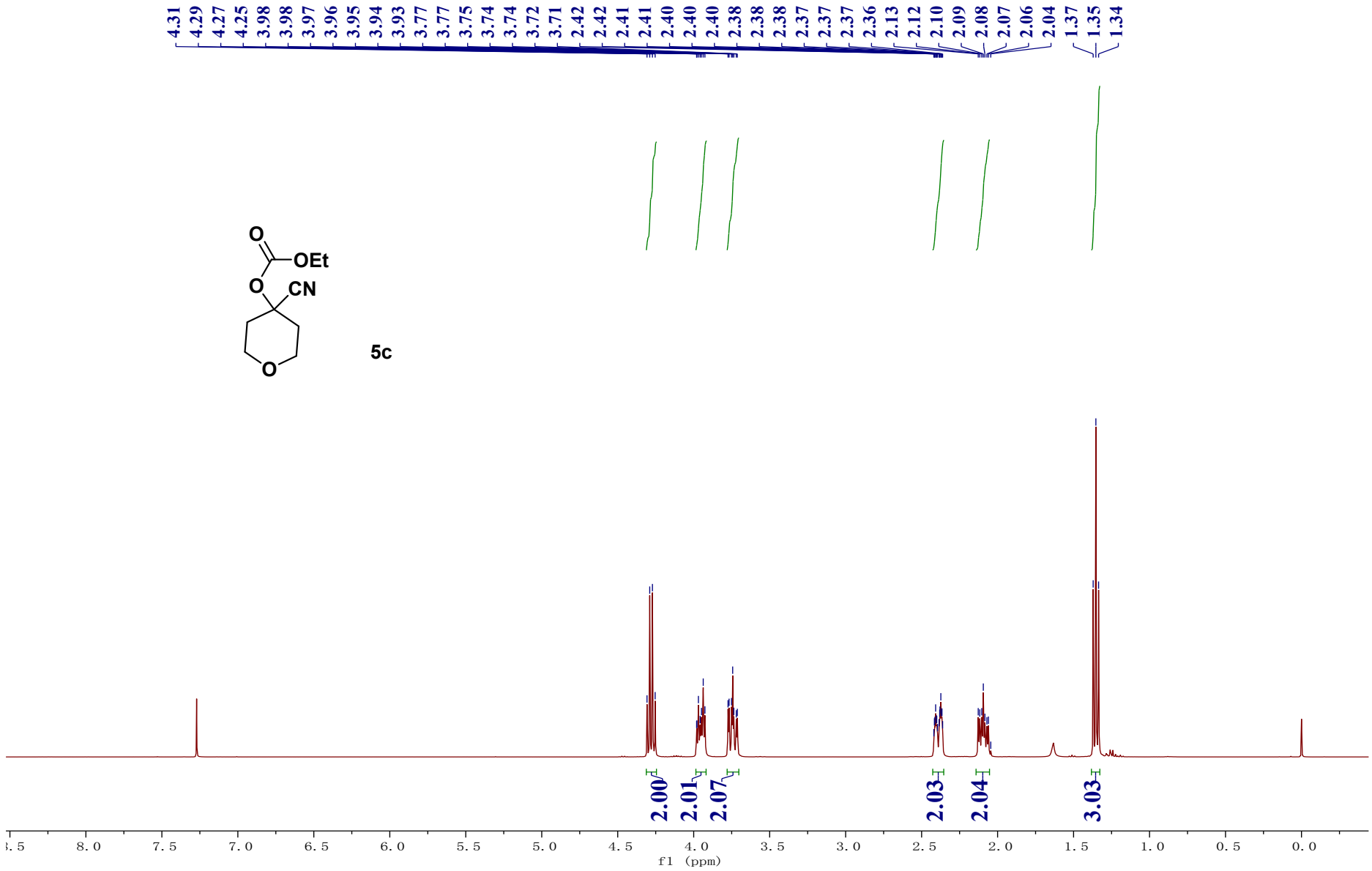
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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3.75
3.74
3.74
3.72
3.71
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2.42
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2.36
2.13
2.12
2.10
2.09
2.08
2.07
2.06
2.04
1.37
1.35
1.34
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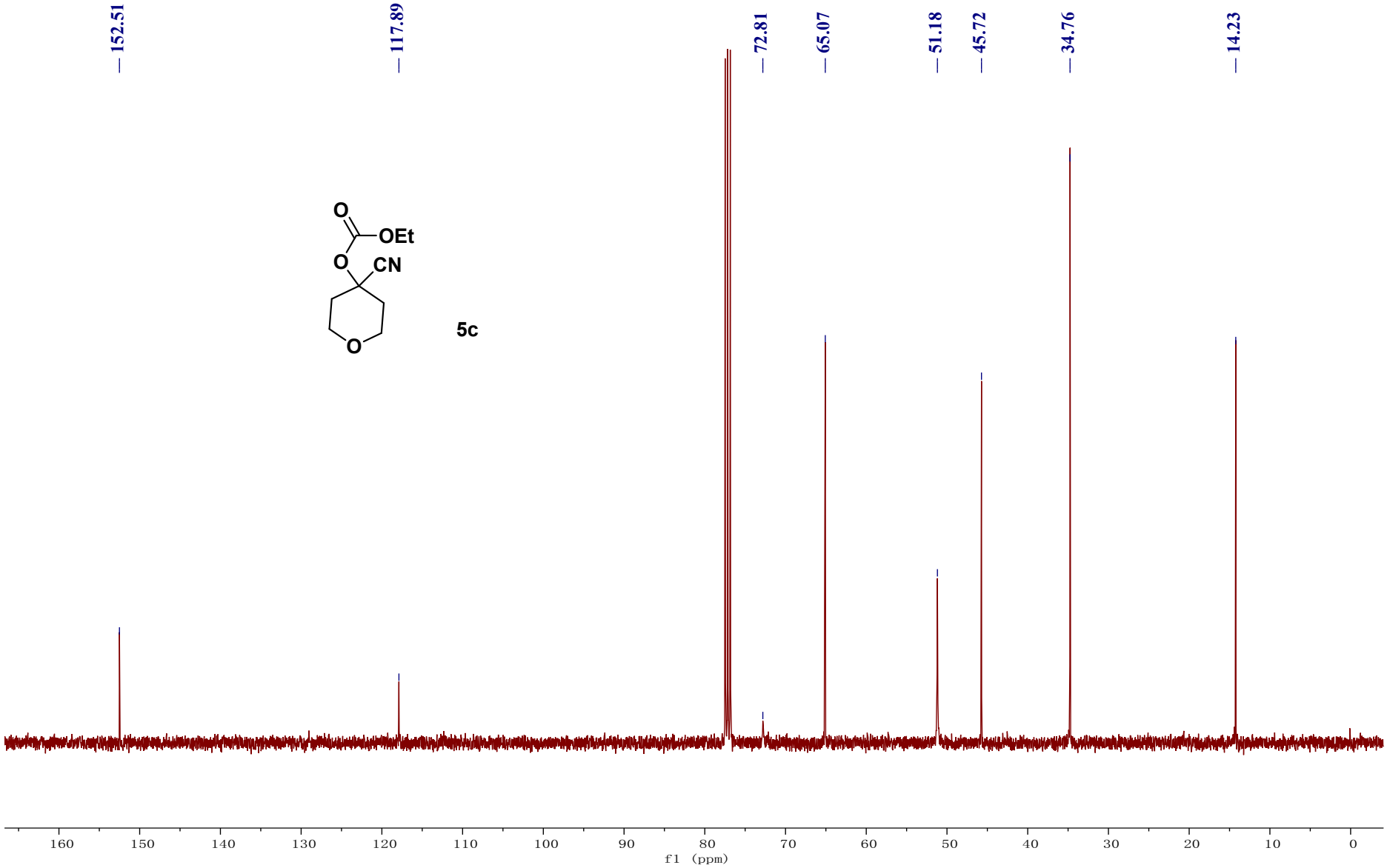
O
CN
O
5c
2.00
2.01
2.07
2.03
2.04
3.03
f1 (ppm)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51.18
45.72
34.76
14.23
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OEt
O
CN
O
5c
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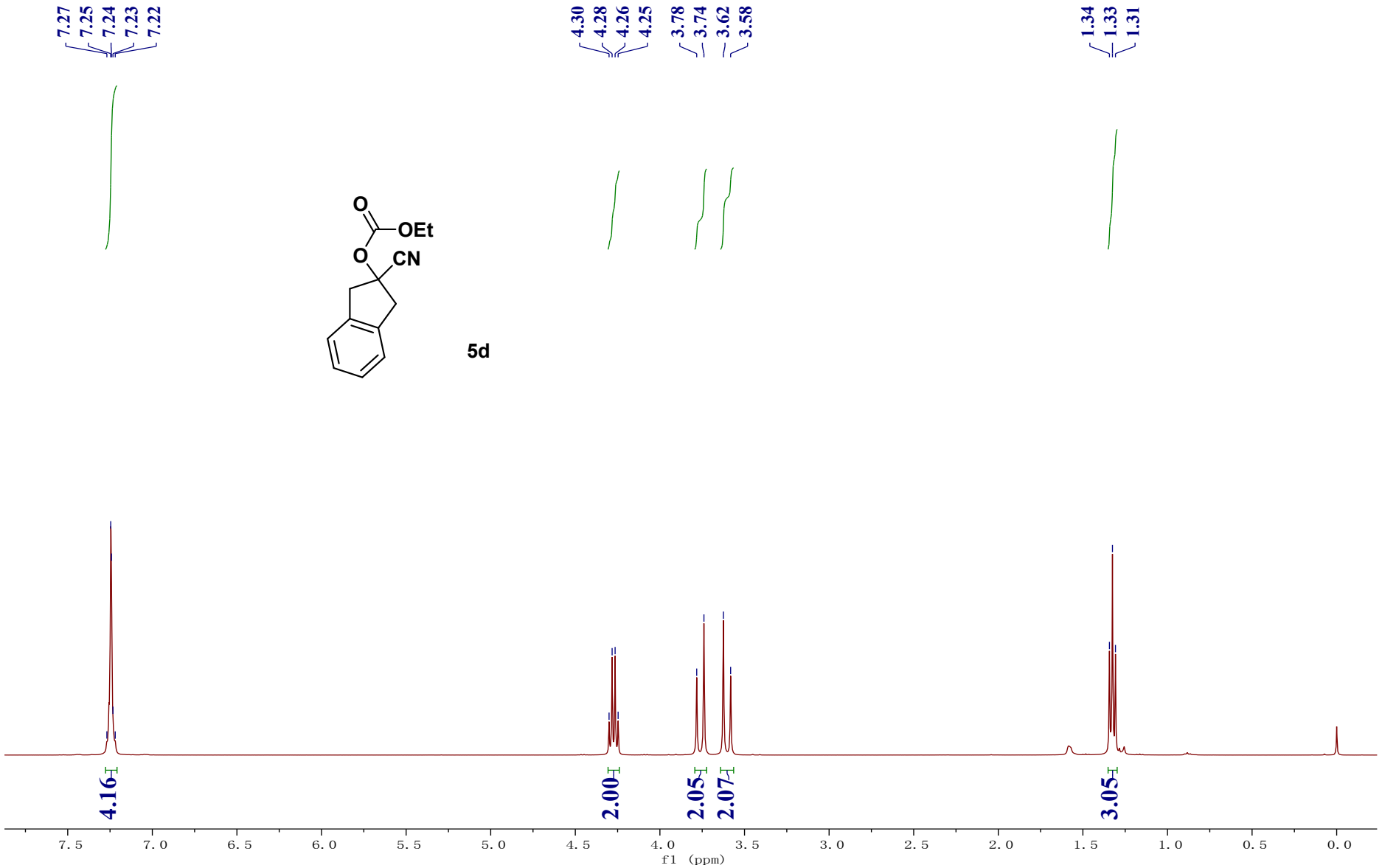
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.23
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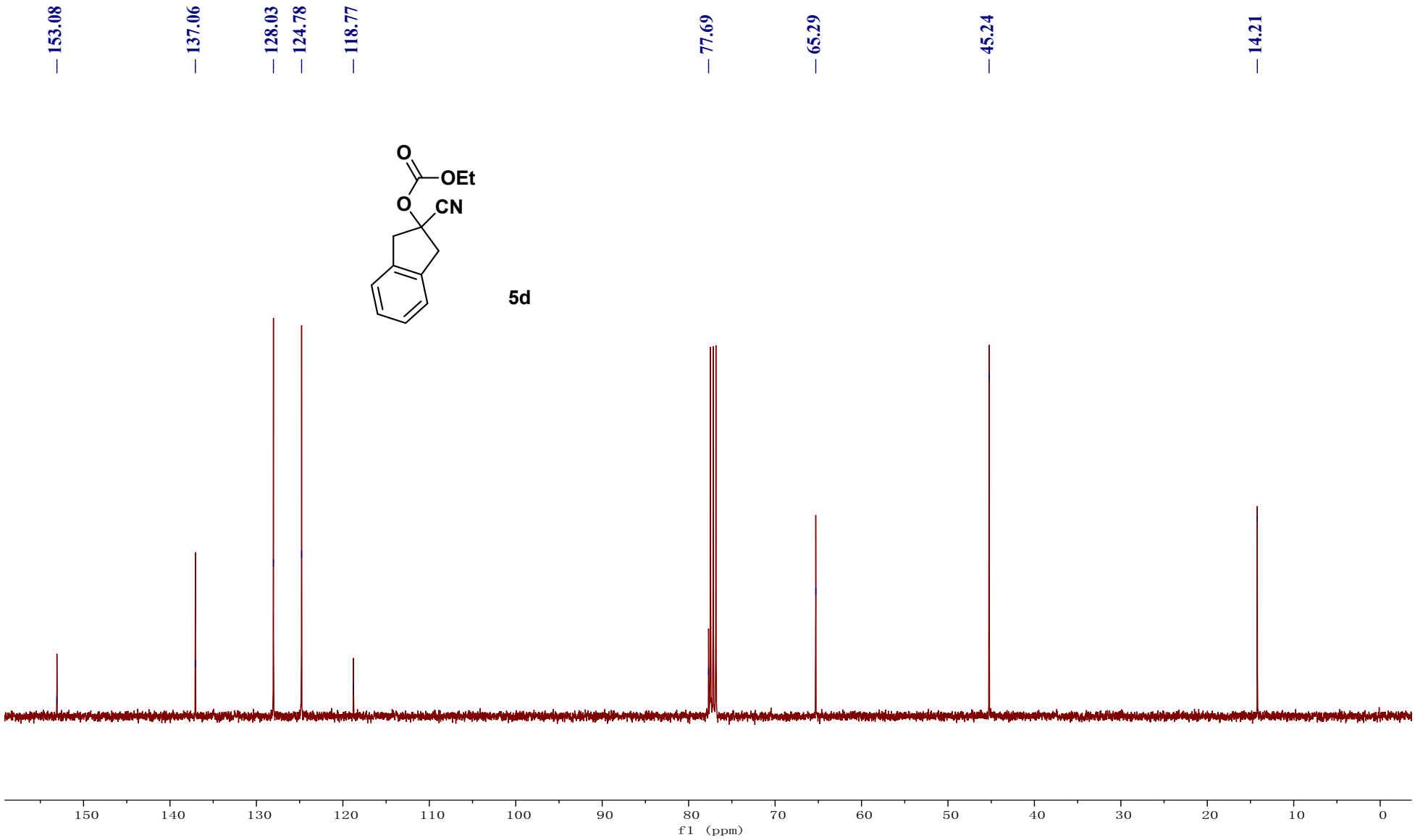
4.30
4.28
4.26
4.25
3.78
3.74
3.62
3.58
1.34
1.33
1.31
O
OEt
O
CN
5d
4.16
2.00
2.05
2.07
3.05
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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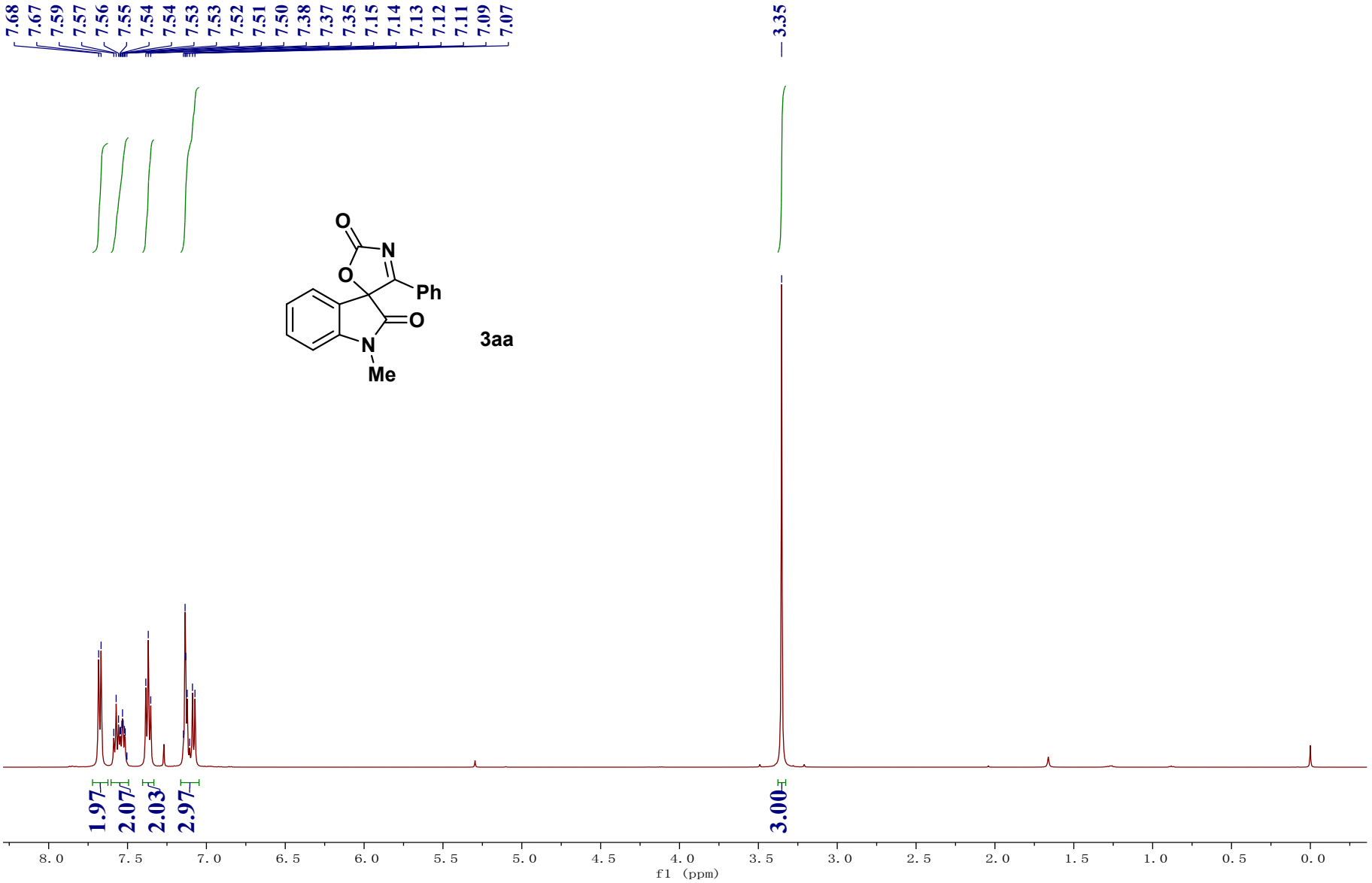
137.06
128.03
124.78
118.77
77.69
65.29
45.24
14.21
O
OEt
O
CN
5d
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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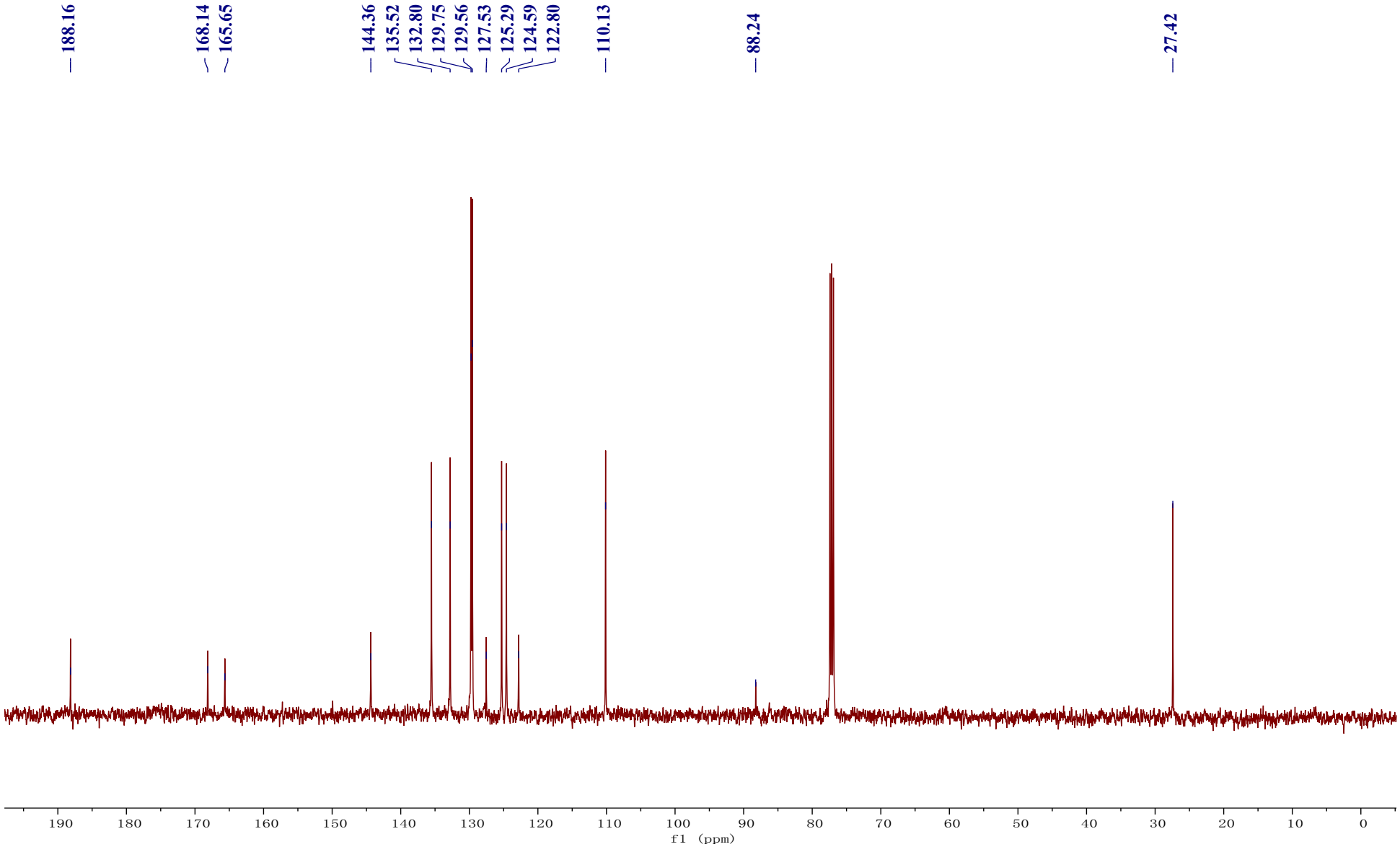
7.38
7.37
7.35
7.15
7.14
7.13
7.12
7.11
7.09
7.07
3.35
O
N
O
Ph
O
N
Me
3aa
1.97
2.07
2.03
2.97
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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144.36
135.52
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129.75
129.56
127.53
125.29
124.59
122.80
110.13
88.24
27.42
190
180
170
160
150
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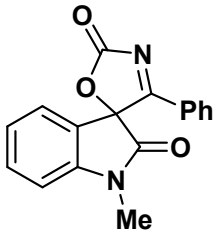
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



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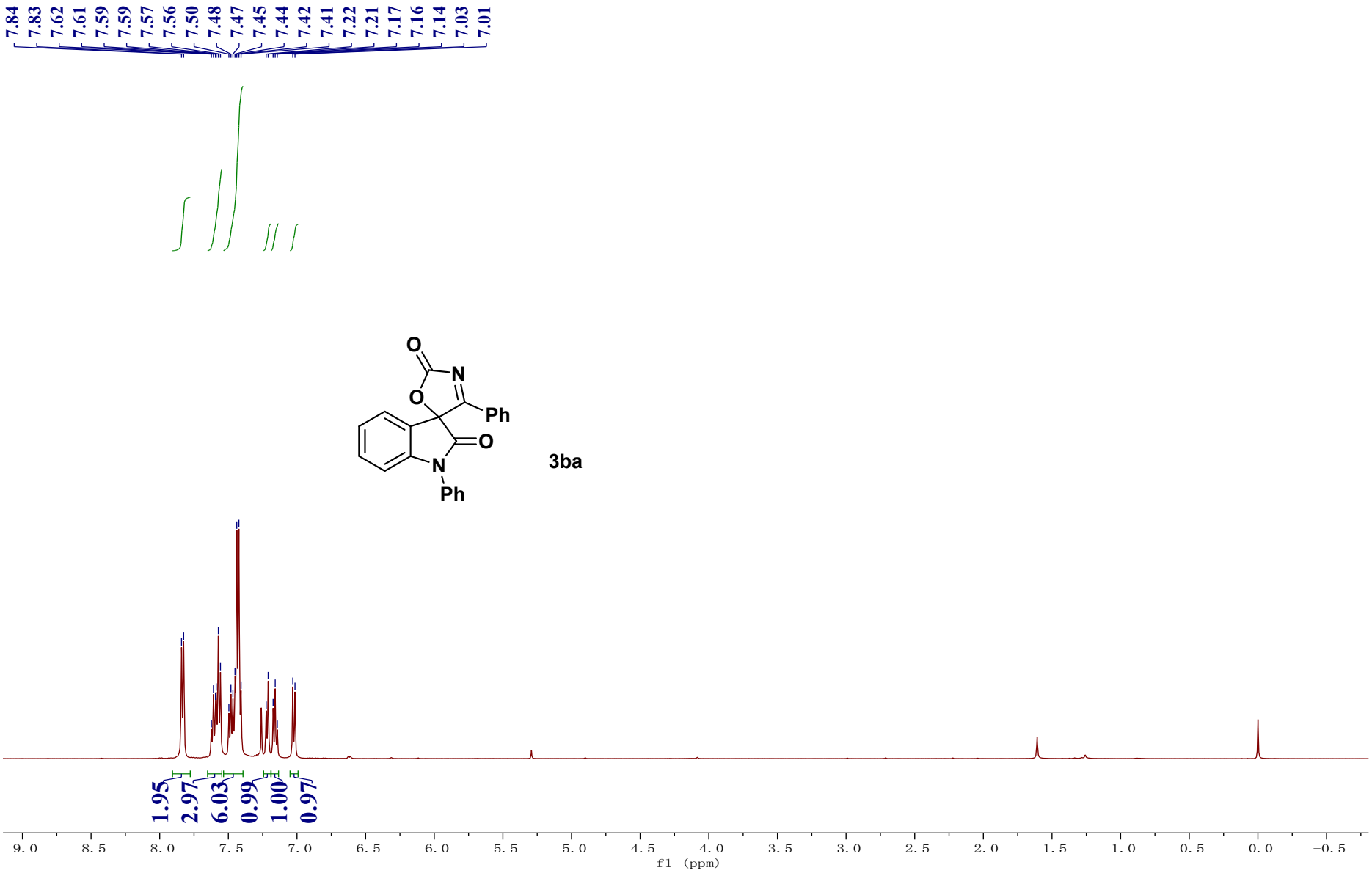
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7.57
7.56
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7.48
7.47
7.45
7.44
7.42
7.41
7.22
7.21
7.17
7.16
7.14
7.03
7.01
O
N
O
Ph
O
N
Ph
3ba
1.95
2.97
6.03
0.99
1.00
0.97
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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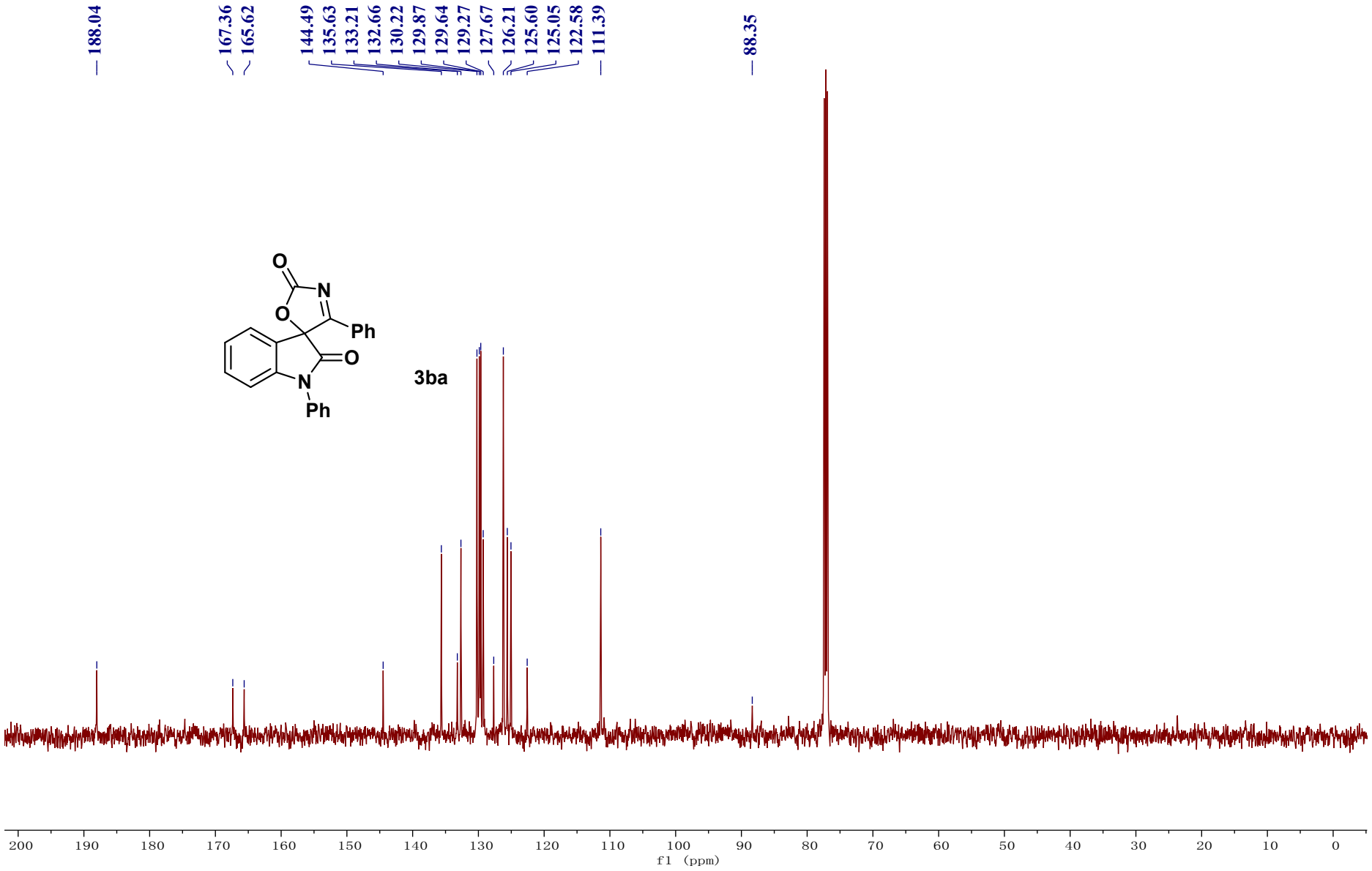
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)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132.66
130.22
129.87
129.64
129.27
127.67
126.21
125.60
125.05
122.58
111.39
88.35
O
N
O
Ph
O
N
Ph
3ba
200
190
180
170
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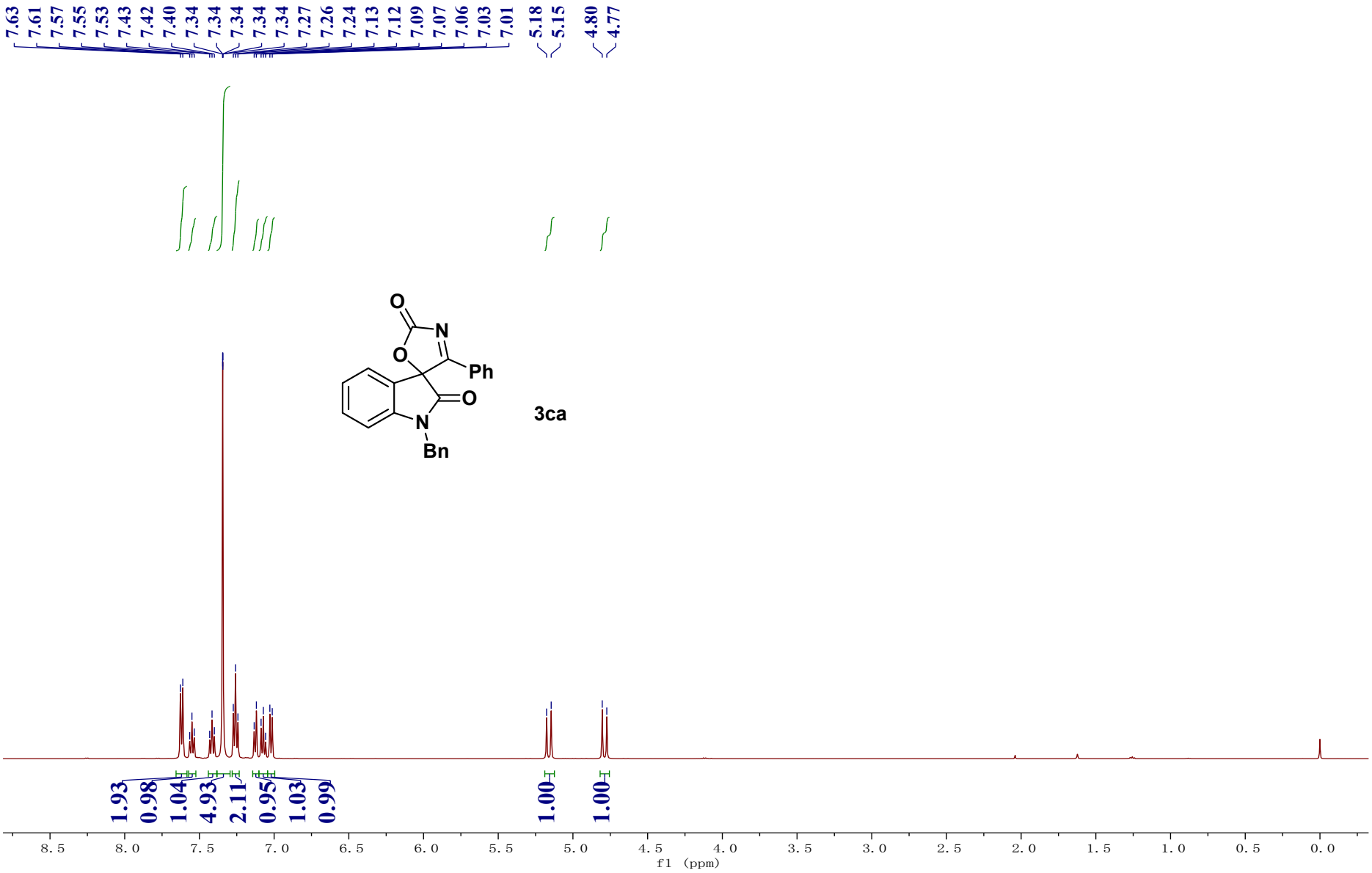
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.40
7.34
7.34
7.34
7.34
7.34
7.27
7.26
7.24
7.13
7.12
7.09
7.07
7.06
7.03
7.01
5.18
5.15
4.80
4.77
O
N
O
Ph
O
N
Bn
3ca
1.93
0.98
1.04
4.93
2.11
0.95
1.03
0.99
1.00
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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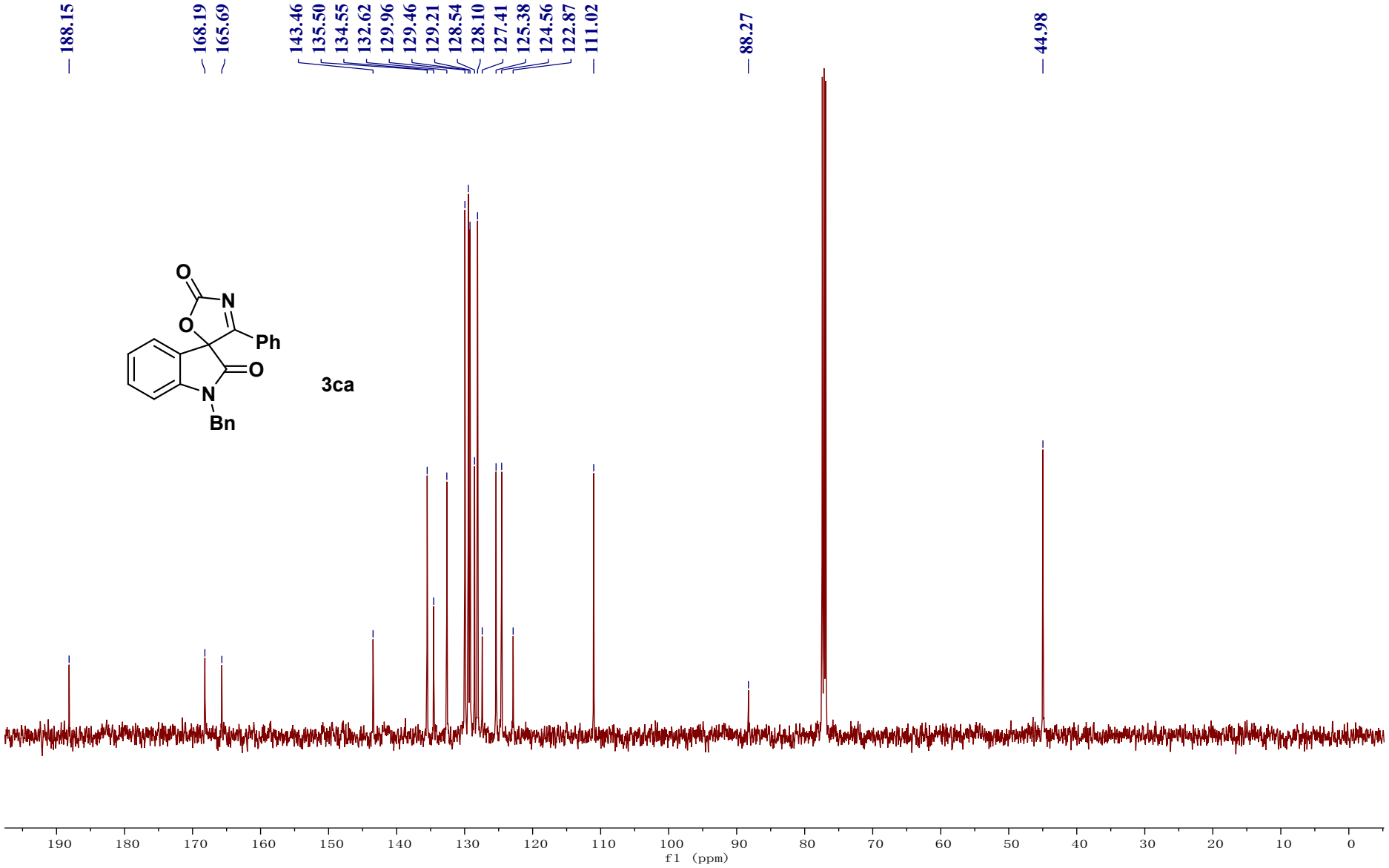
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.96
129.46
129.21
128.54
128.10
127.41
125.38
124.56
122.87
111.02
88.27
44.98
O
N
O
Ph
O
N
Bn
3ca
190
180
170
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150
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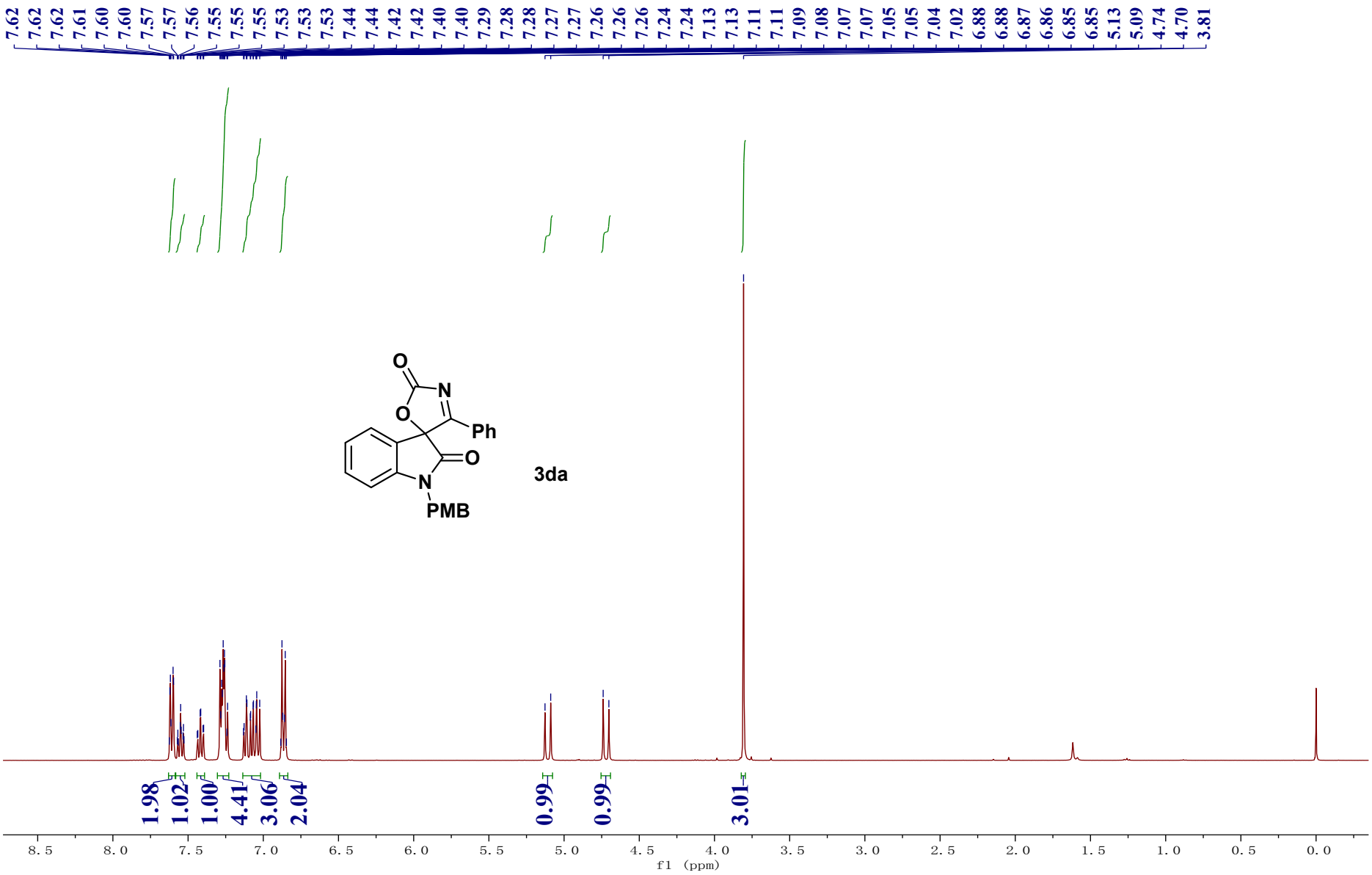
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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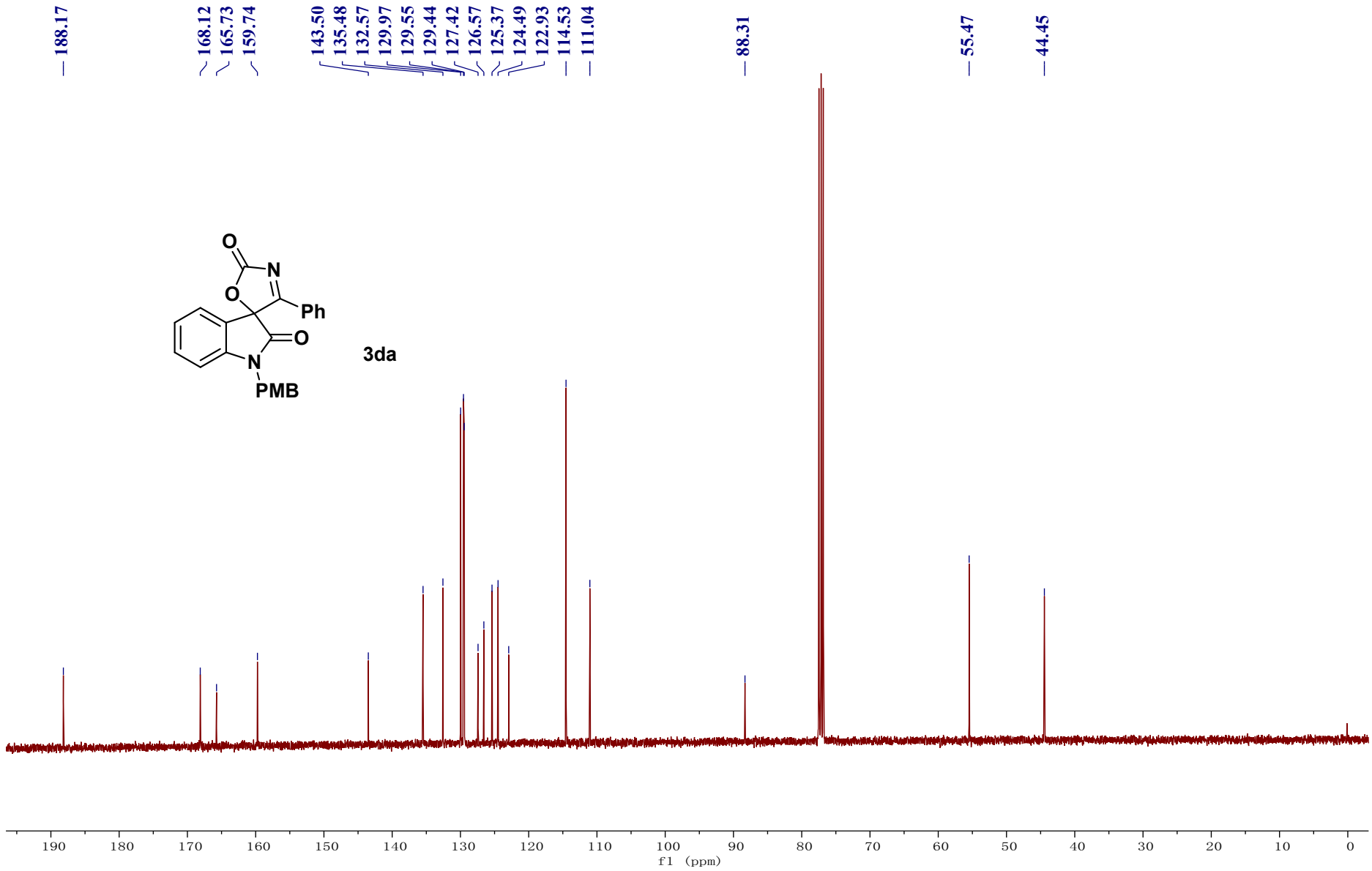
O
N
O
Ph
O
N
PMB
3da
1.98 1.02 1.00 4.41 3.06 2.04 0.99 0.99 3.01
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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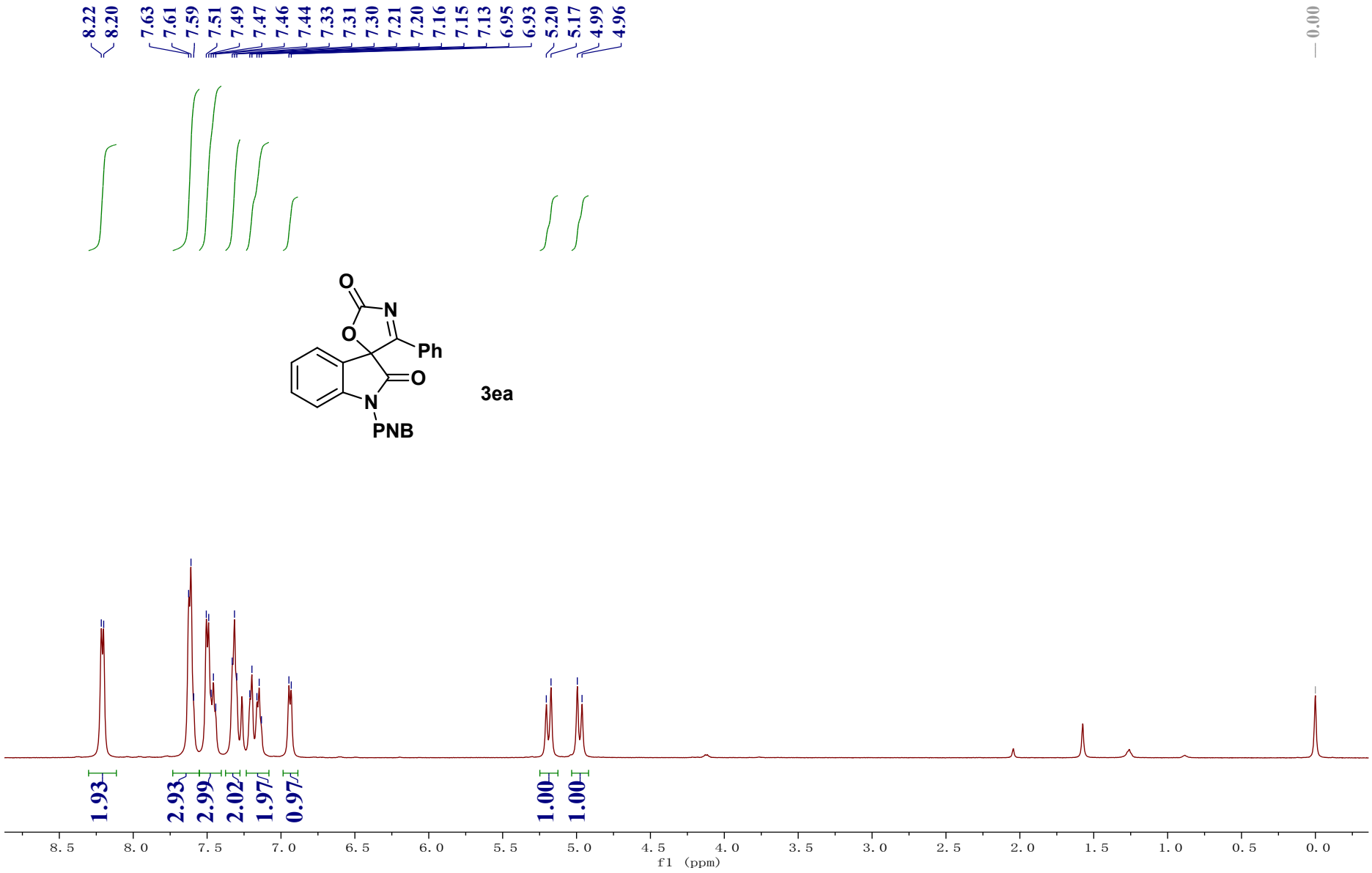
129.97
129.55
129.44
127.42
126.57
125.37
124.49
122.93
114.53
111.04
88.31
55.47
44.45
O
N
O
Ph
O
N
PMB
3da
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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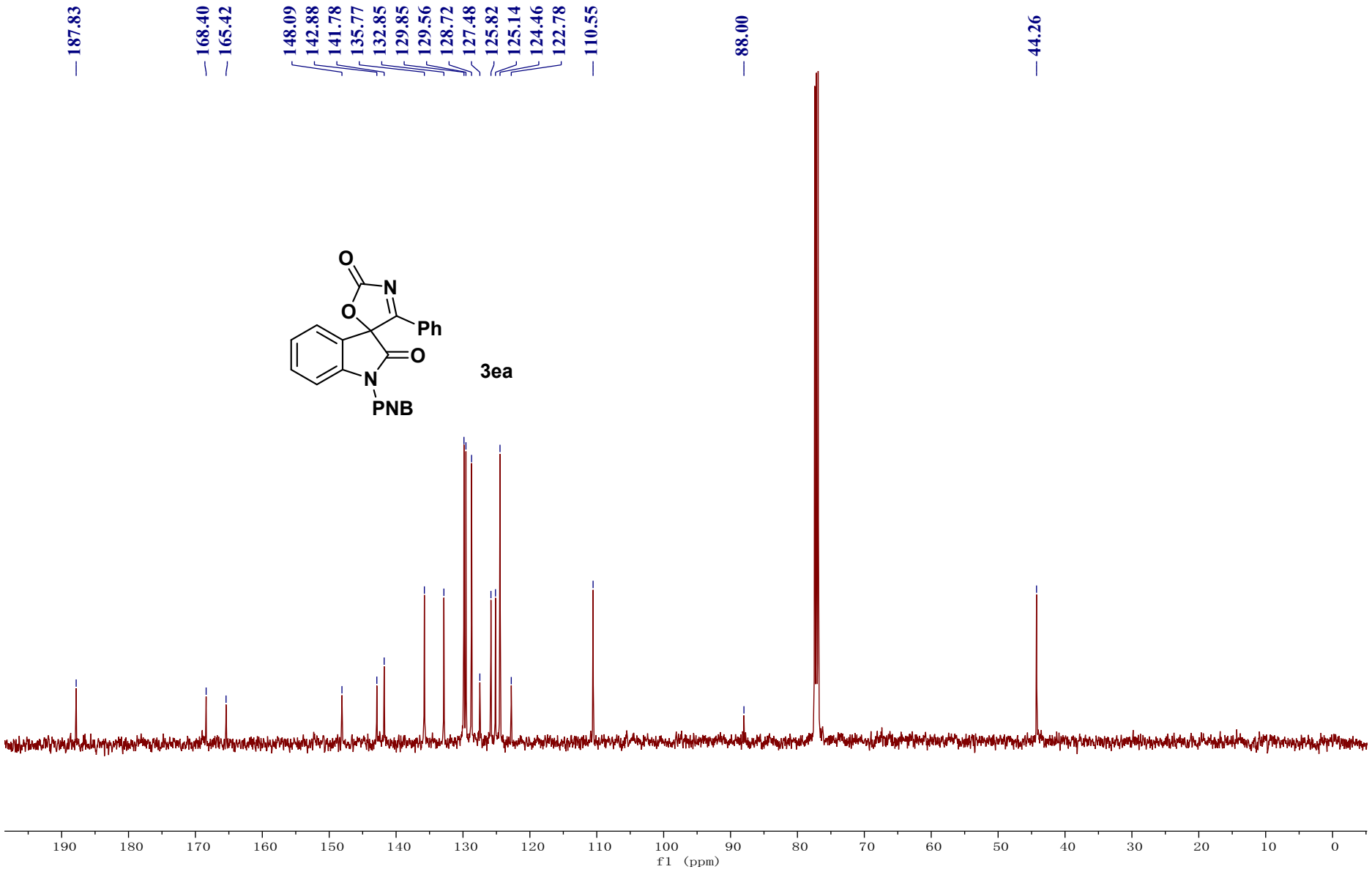
7.49
7.47
7.46
7.44
7.33
7.31
7.30
7.21
7.20
7.16
7.15
7.13
6.95
6.93
5.20
5.17
4.99
4.96
0.00
O
N
O
Ph
O
N
PNB
3ea
1.93
2.93
2.99
2.02
1.97
0.97
1.00
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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135.77
132.85
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129.56
128.72
127.48
125.82
125.14
124.46
122.78
110.55
88.00
44.26
O
N
O
Ph
O
N
PNB
3ea
190
180
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110
100
90
80
70
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50
40
30
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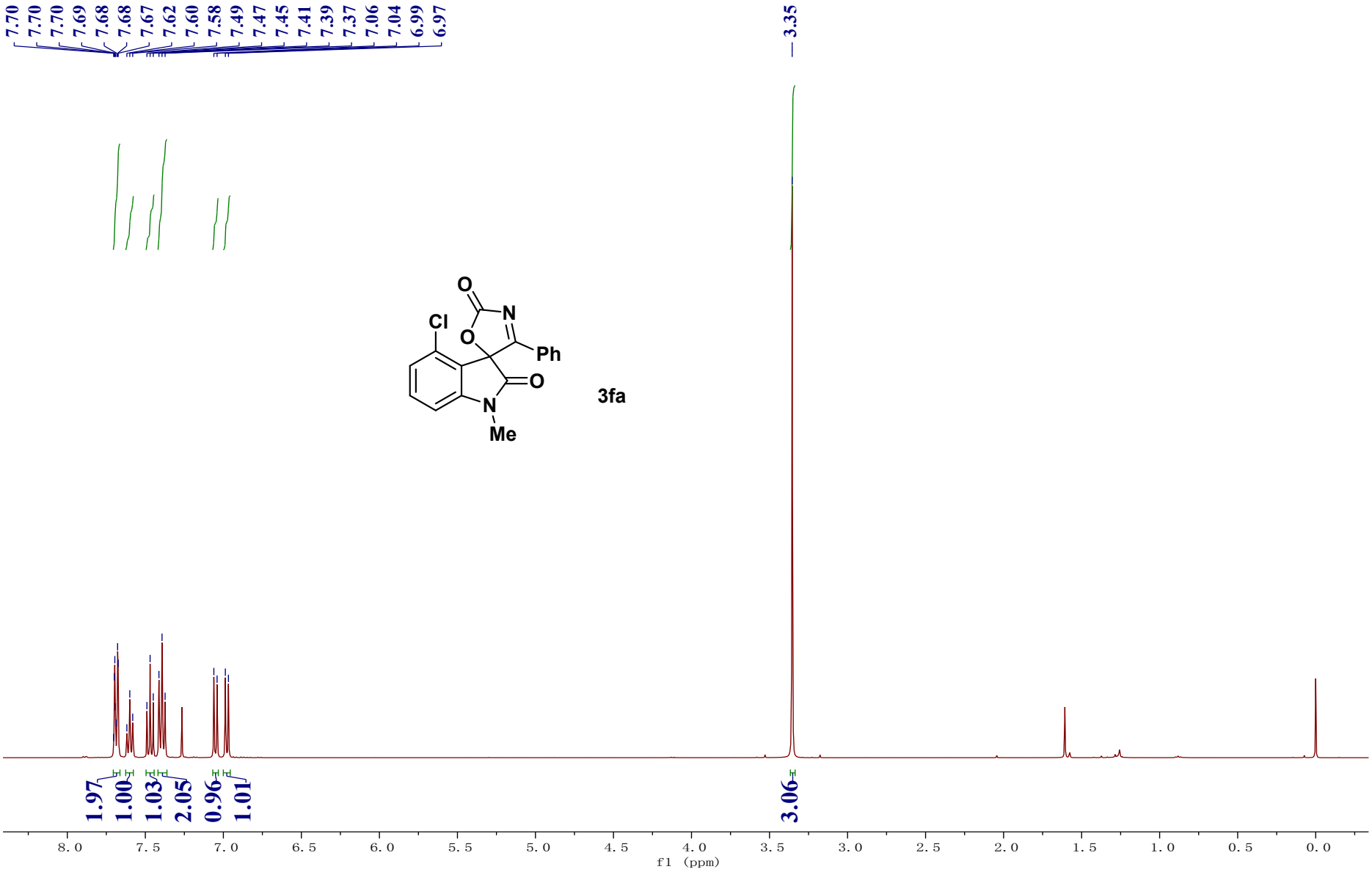
10
0
f1 (ppm)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7.58
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7.47
7.45
7.41
7.39
7.37
7.06
7.04
6.99
6.97
3.35
O
N
Cl
O
Ph
O
N
Me
3fa
1.97
1.00
1.03
2.05
0.96
1.01
3.06
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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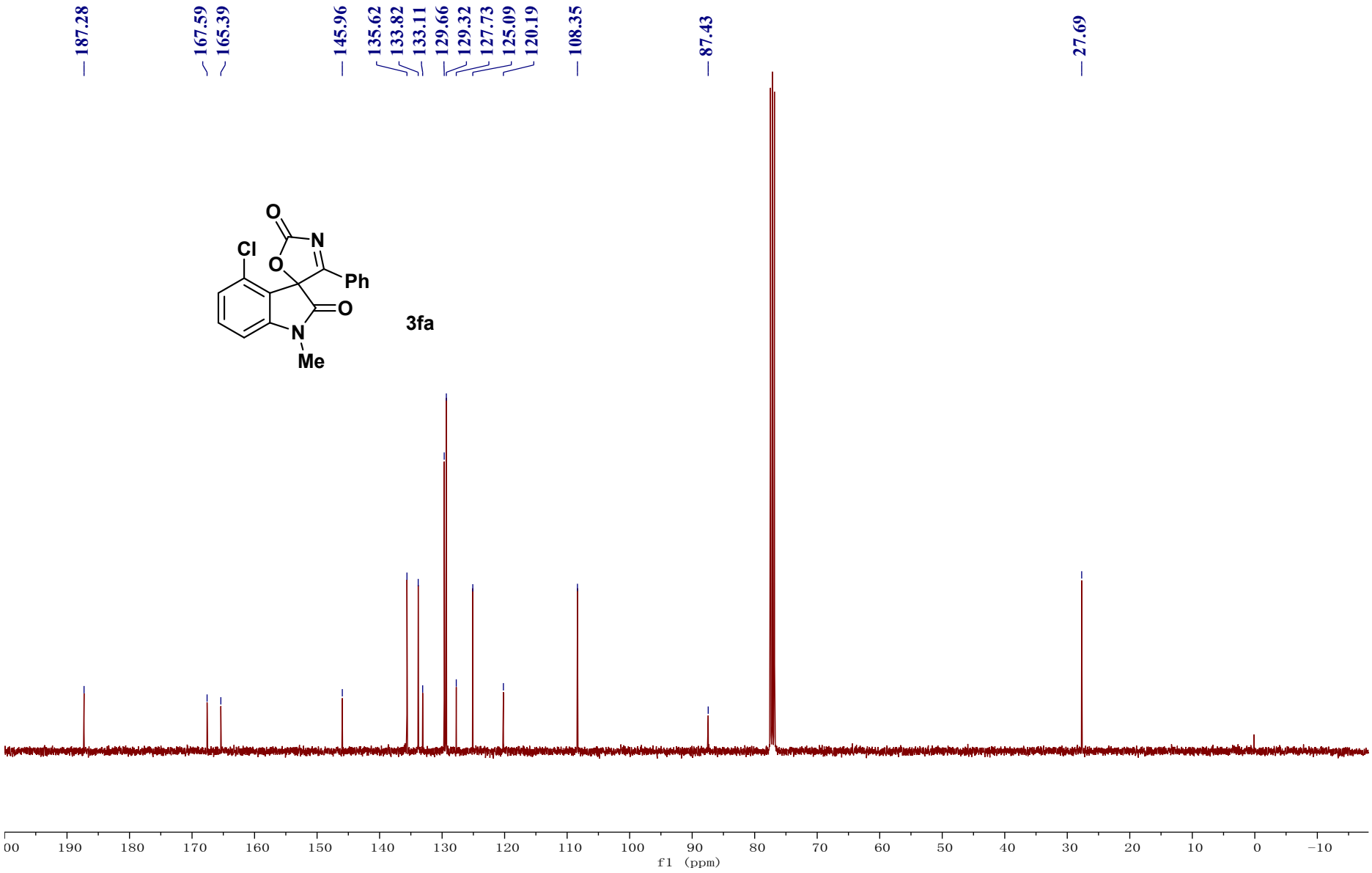
0.0
f1 (ppm)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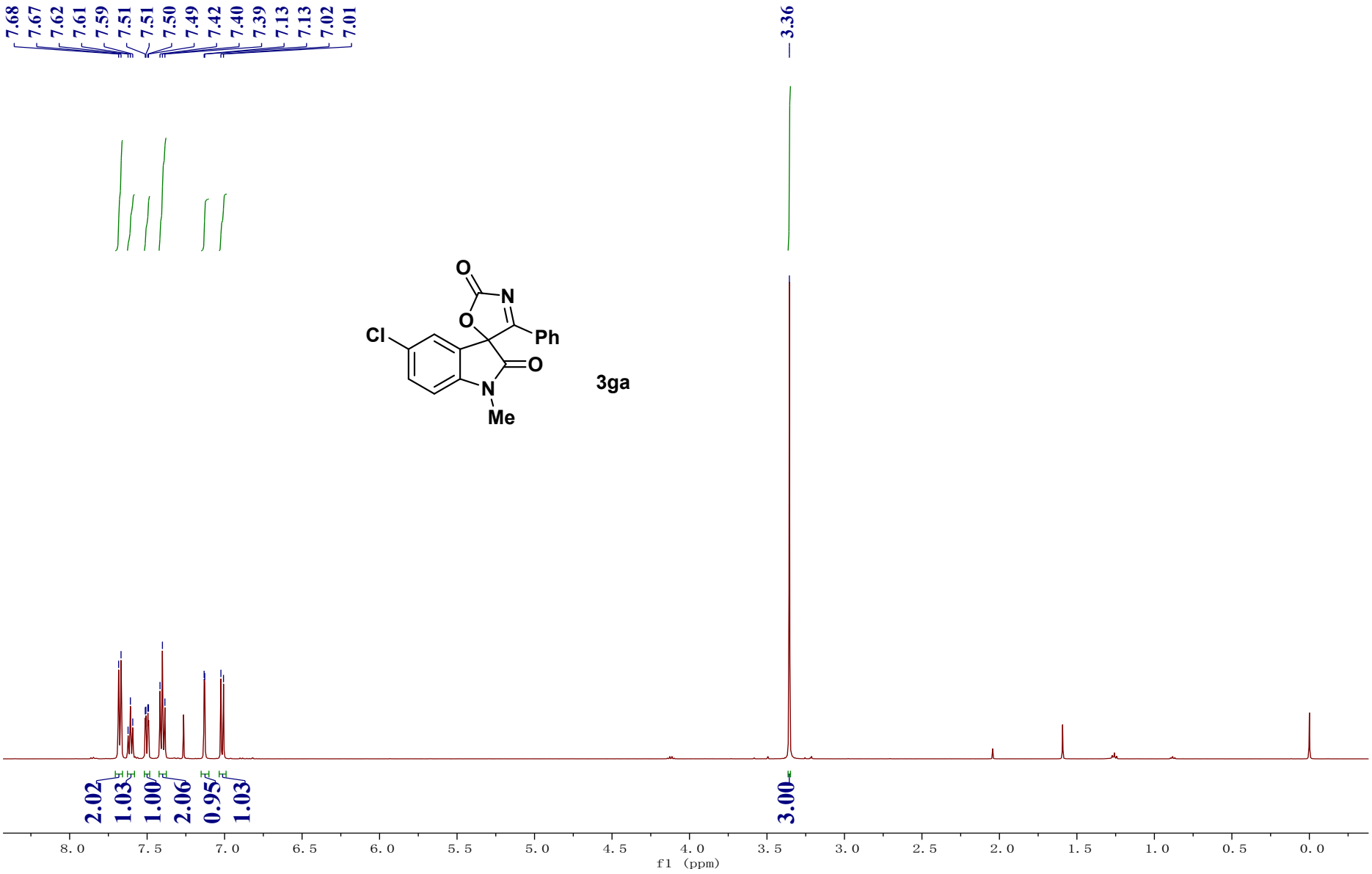
133.82
133.11
129.66
129.32
127.73
125.09
120.19
108.35
87.43
27.69
O
N
Cl
O
Ph
O
N
Me
3fa
f1 (ppm)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7.51
7.50
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7.42
7.40
7.39
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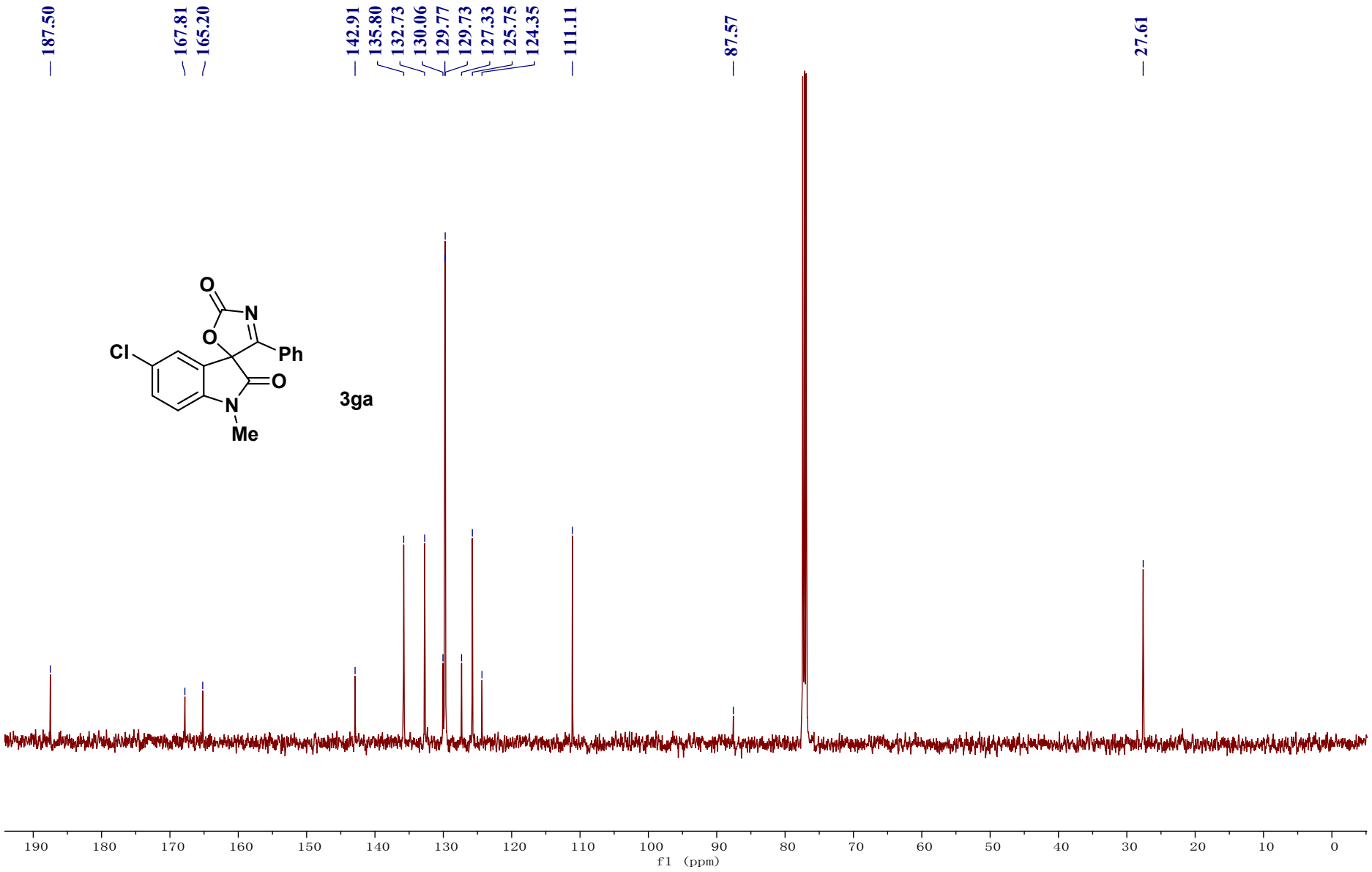
7.13
7.02
7.01
3.36
O
N
O
Cl
Ph
O
N
Me
3ga
2.02
1.03
1.00
2.06
0.95
1.03
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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130.06
129.77
129.73
127.33
125.75
124.35
111.11
87.57
27.61
O
N
O
Cl
Ph
O
N
Me
3ga
190
180
170
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150
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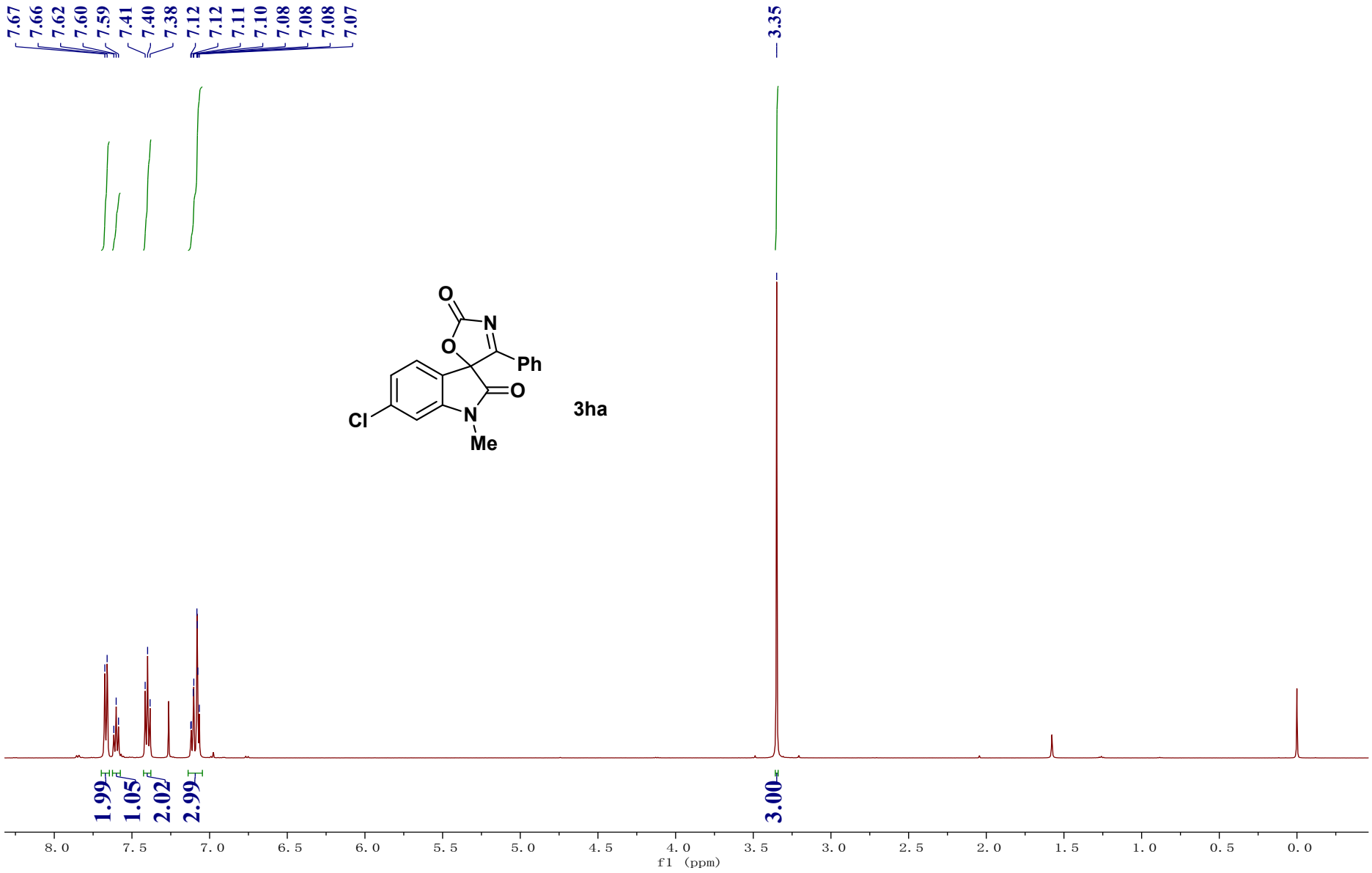
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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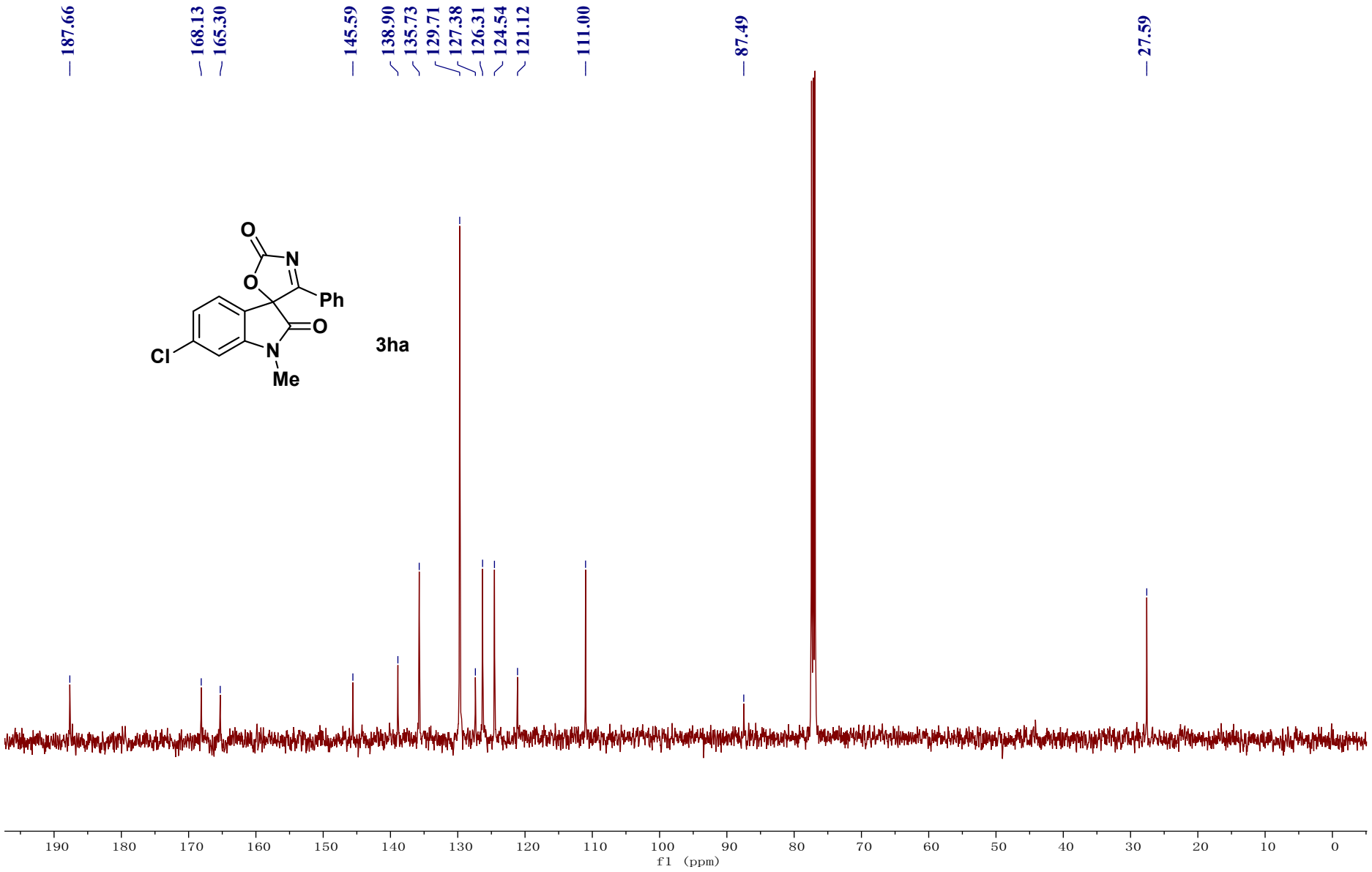
7.38
7.12
7.12
7.11
7.10
7.08
7.08
7.08
7.07
3.35
O
N
O
Ph
O
Cl
N
Me
3ha
1.99
1.05
2.02
2.99
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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O
Ph
O
Cl
N
Me
3ha
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180
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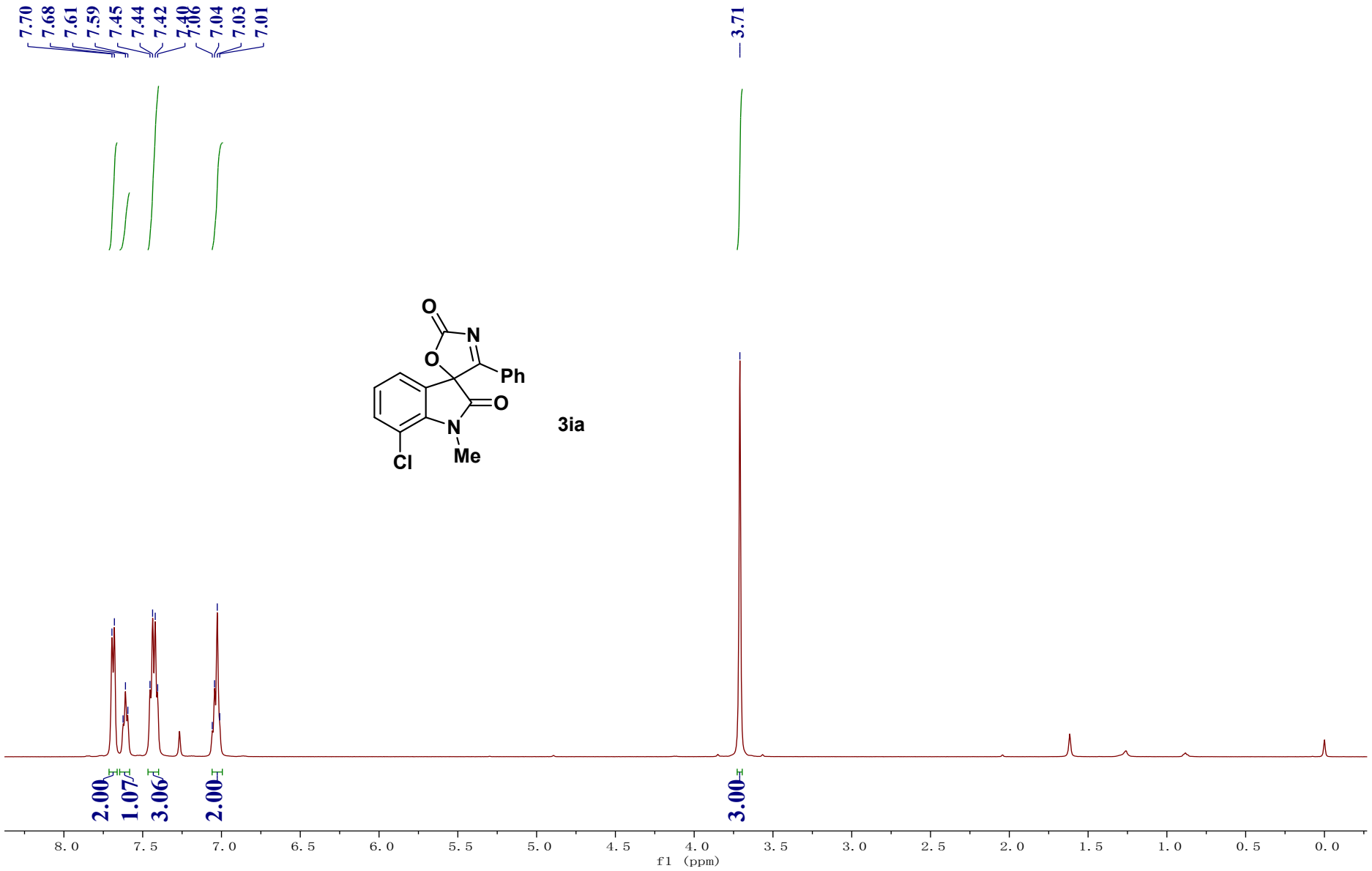
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3.71
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O
Ph
O
N
Me
Cl
3ia
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1.07
3.06
2.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
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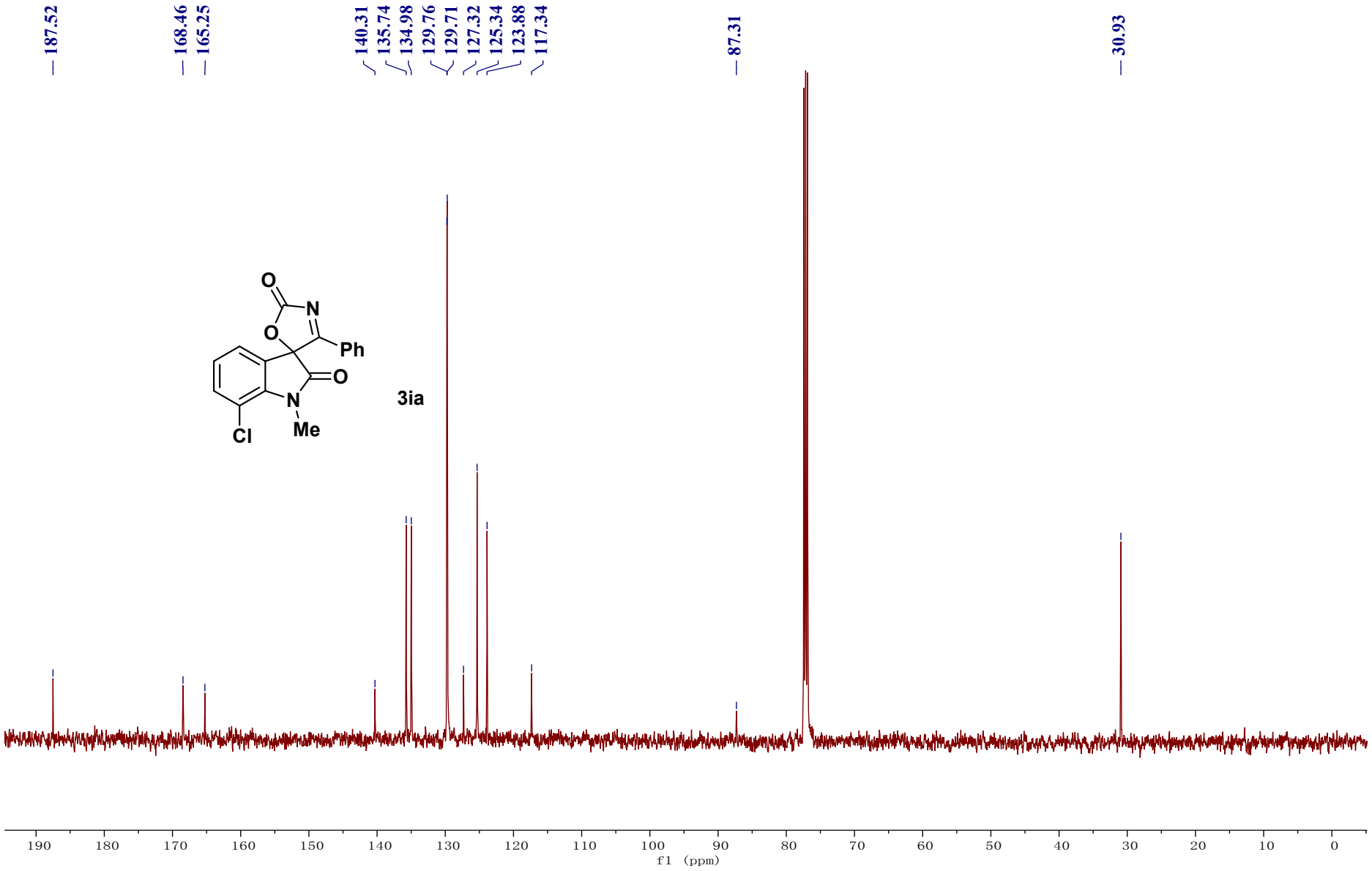
f1 (ppm)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129.71
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O
N
O
Ph
O
N
Me
Cl
3ia
190
180
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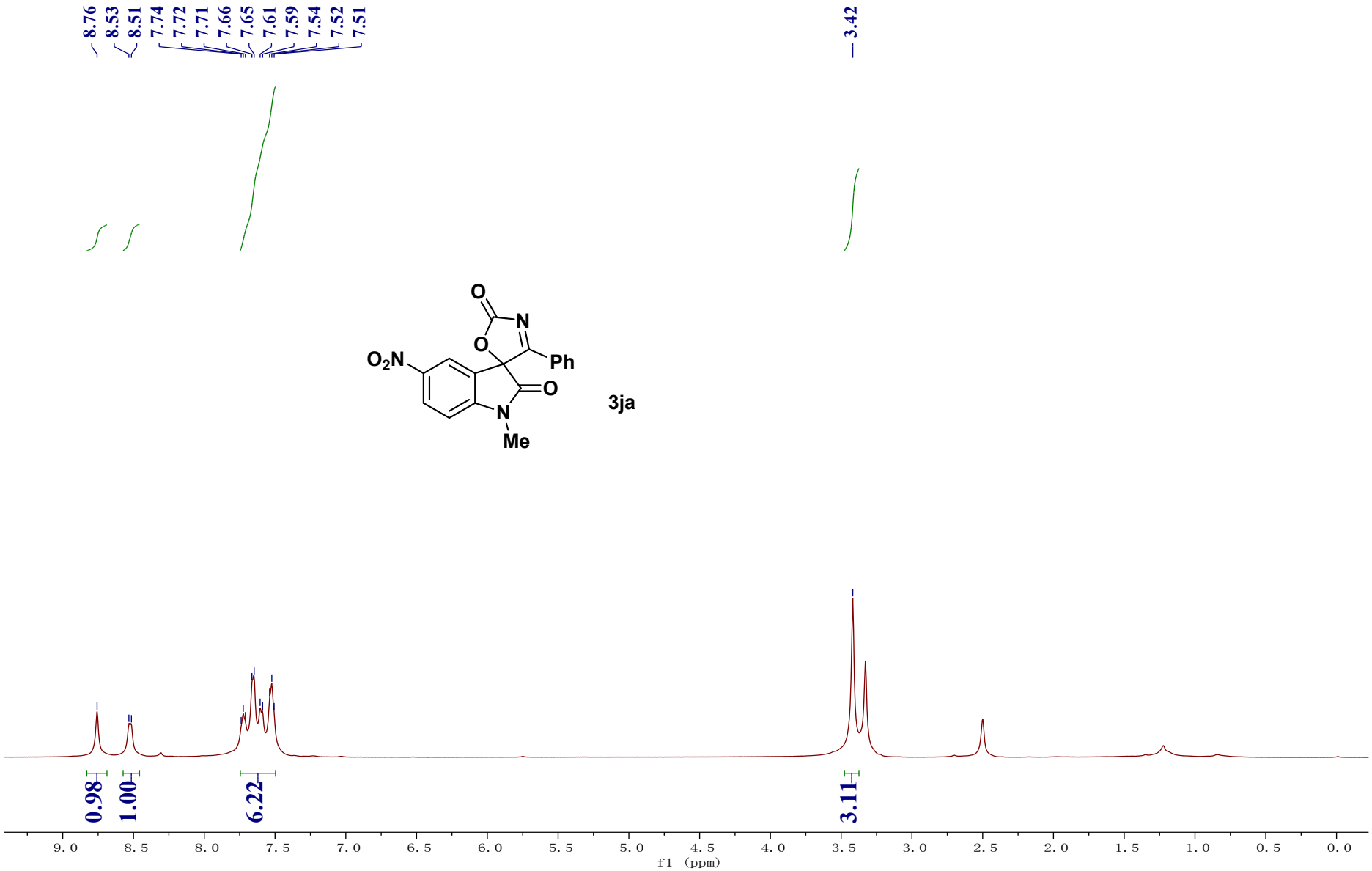
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7.51
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O
Ph
O2N
O
N
Me
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3.11
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7.0
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5.5
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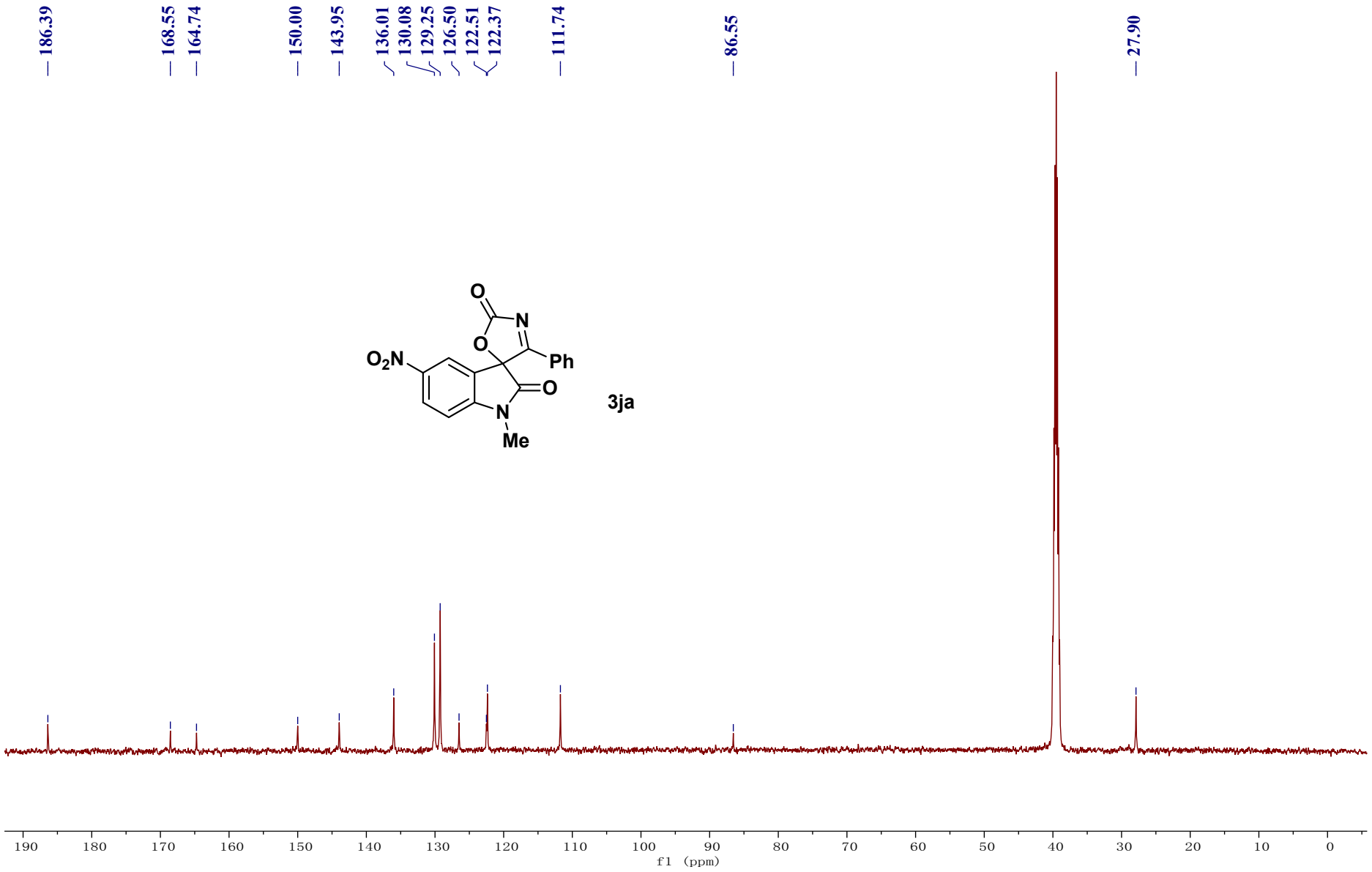
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O
N
O
Ph
O2N
O
N
Me
3ja
190
180
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90
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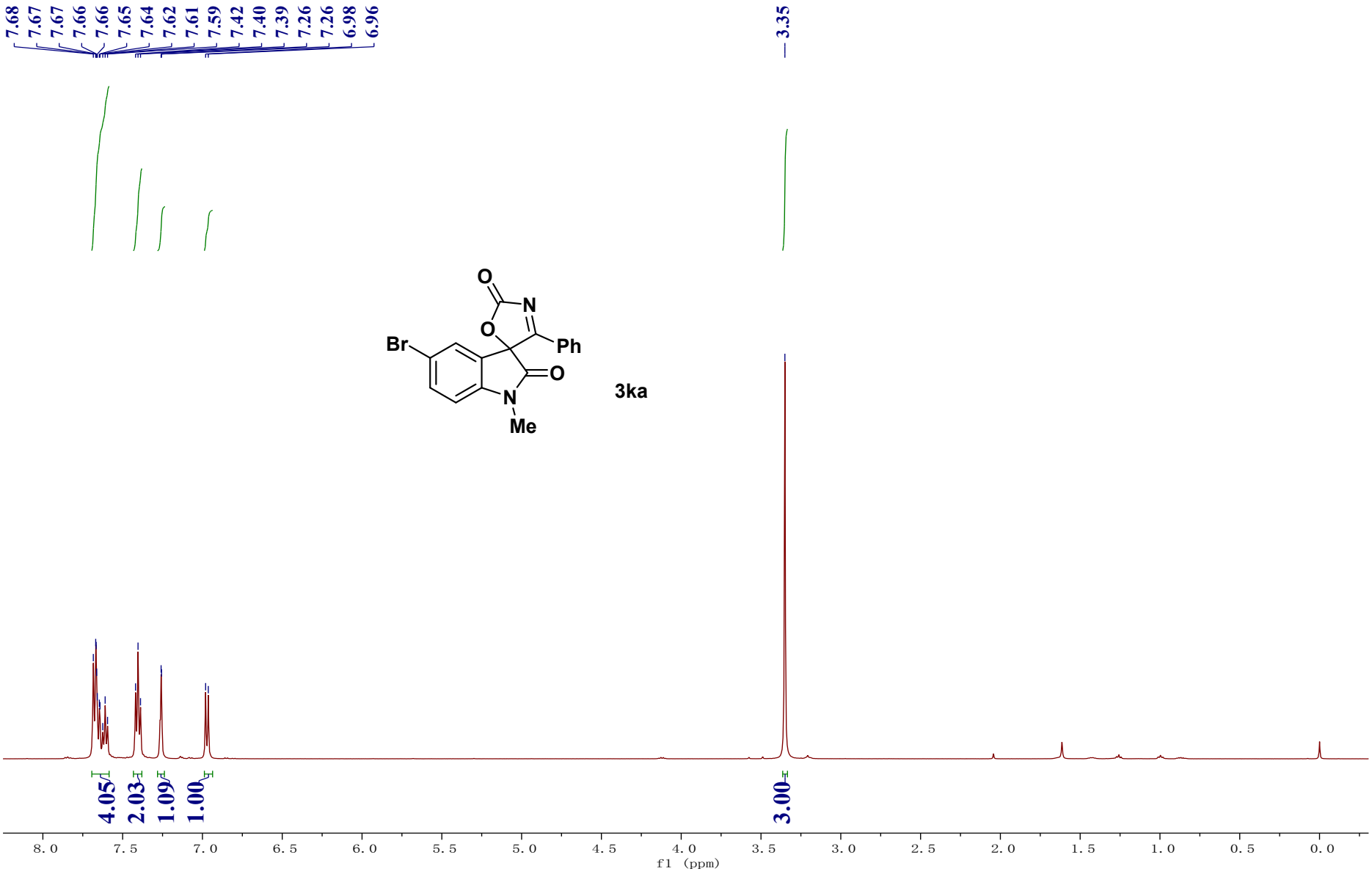
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7.26
6.98
6.96
3.35
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N
O
Ph
Br
O
N
Me
3ka
4.05
2.03
1.09
1.00
3.00
8.0
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7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
1.0
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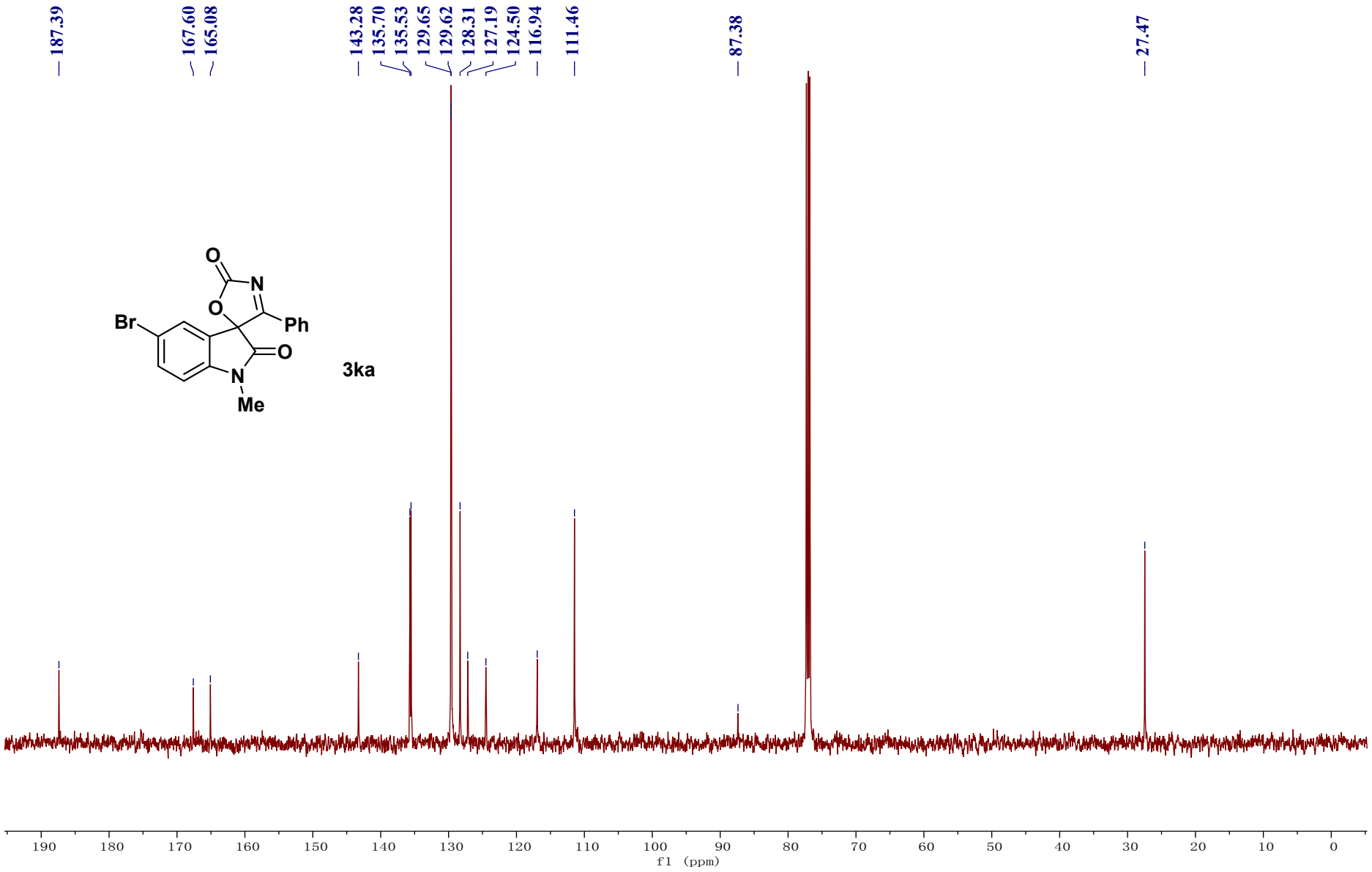
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27.47
Br
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N
O
Ph
O
N
Me
3ka
190
180
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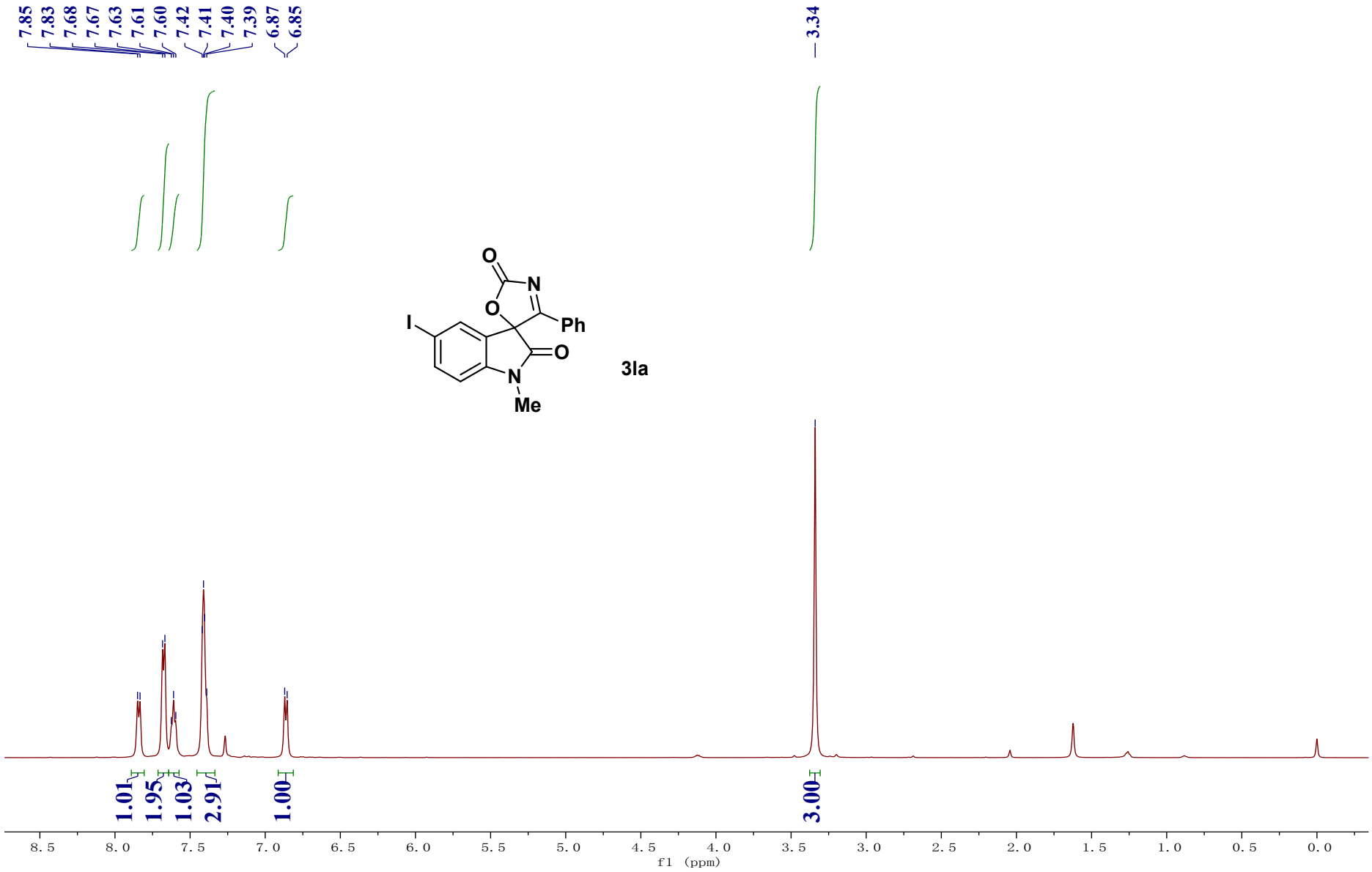
f1 (ppm)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6.85
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I
O
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Me
3Ia
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1.95
1.03
2.91
1.00
3.00
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6.0
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2.5
2.0
1.5
1.0
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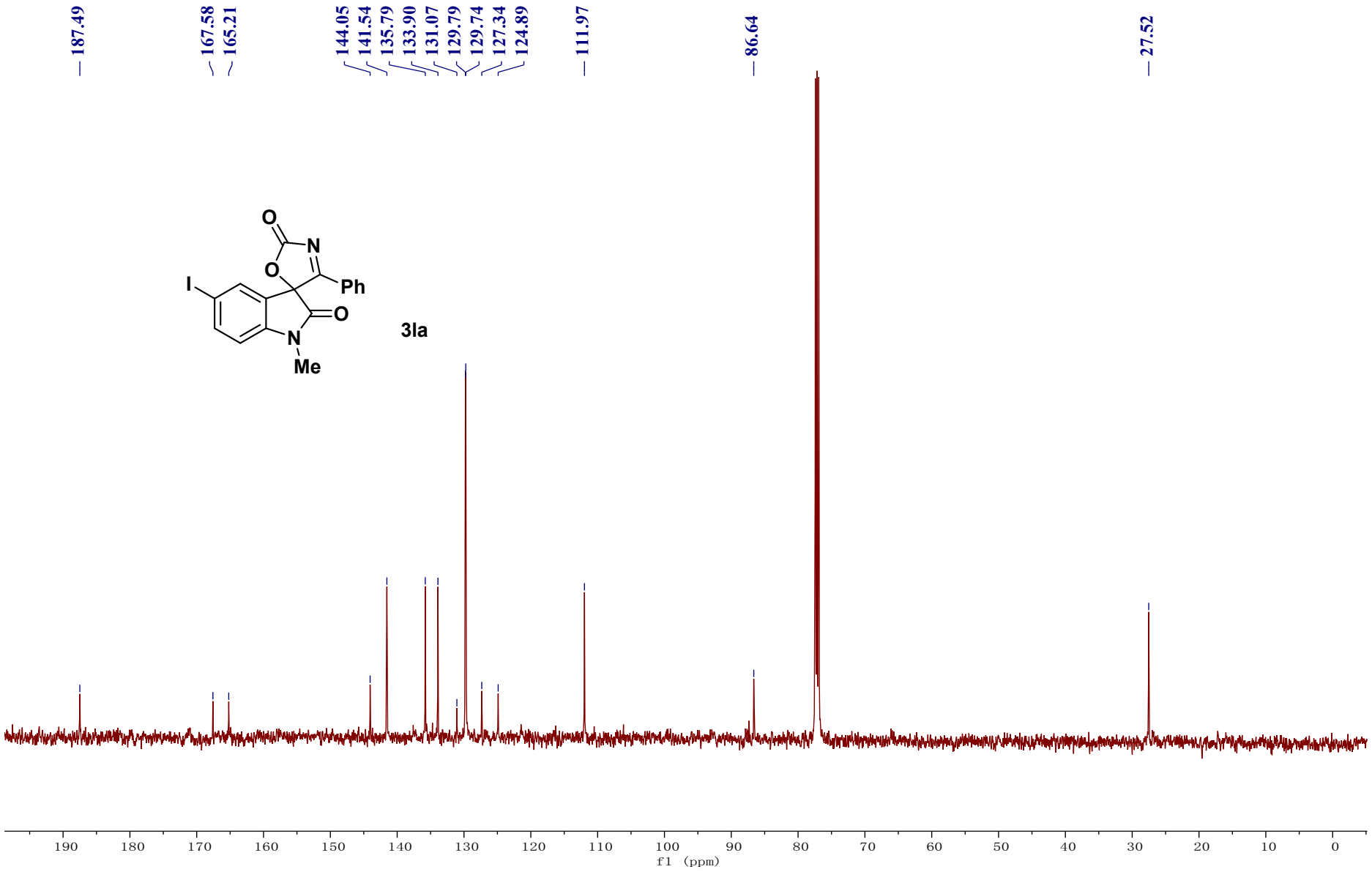
0.0
f1 (ppm)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I
O
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Me
3la
190
180
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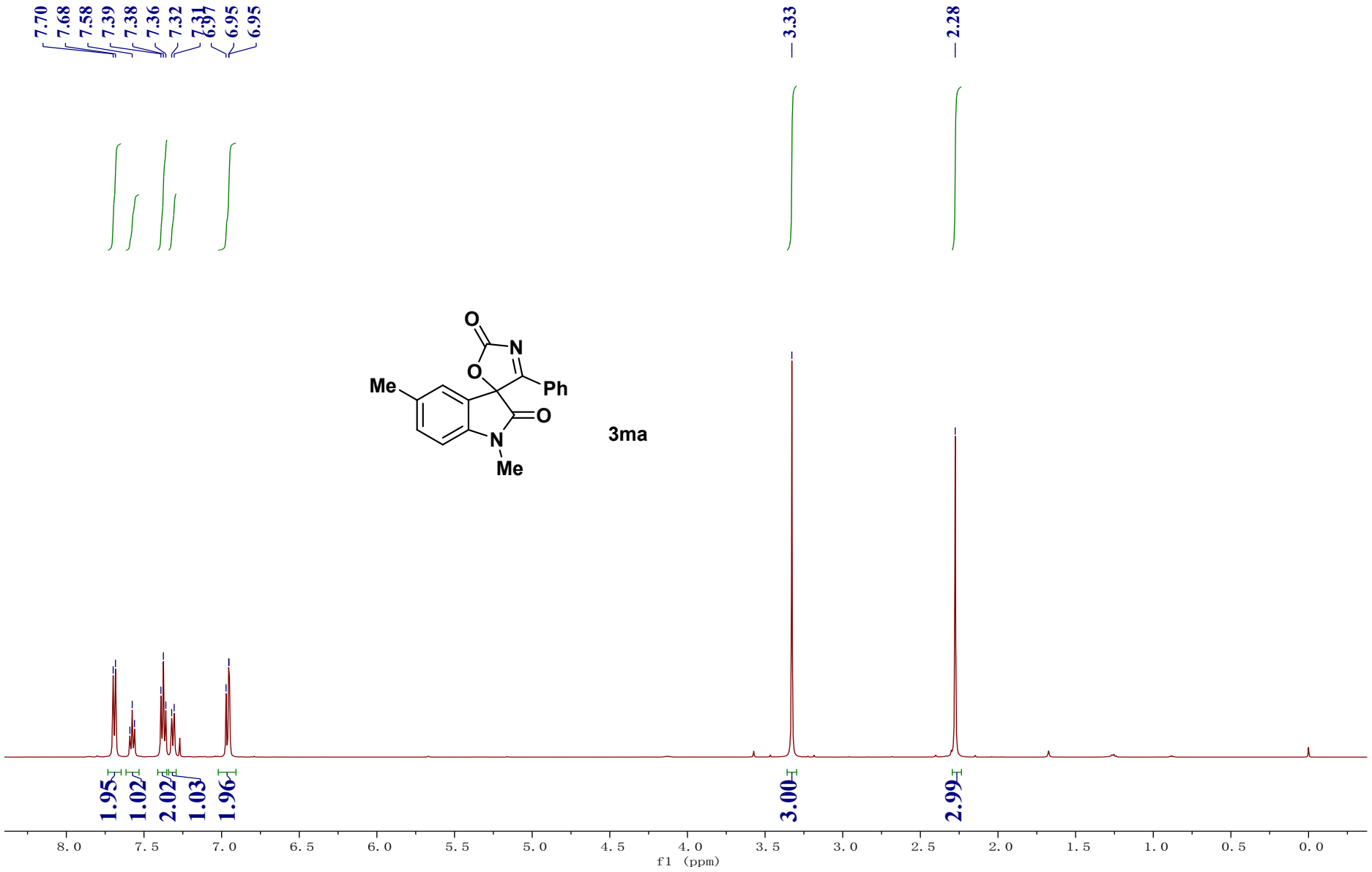
f1 (ppm)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6.95
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O
Ph
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O
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Me
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1.95
1.02
2.02
1.03
1.96
3.00
2.99
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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
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1.0
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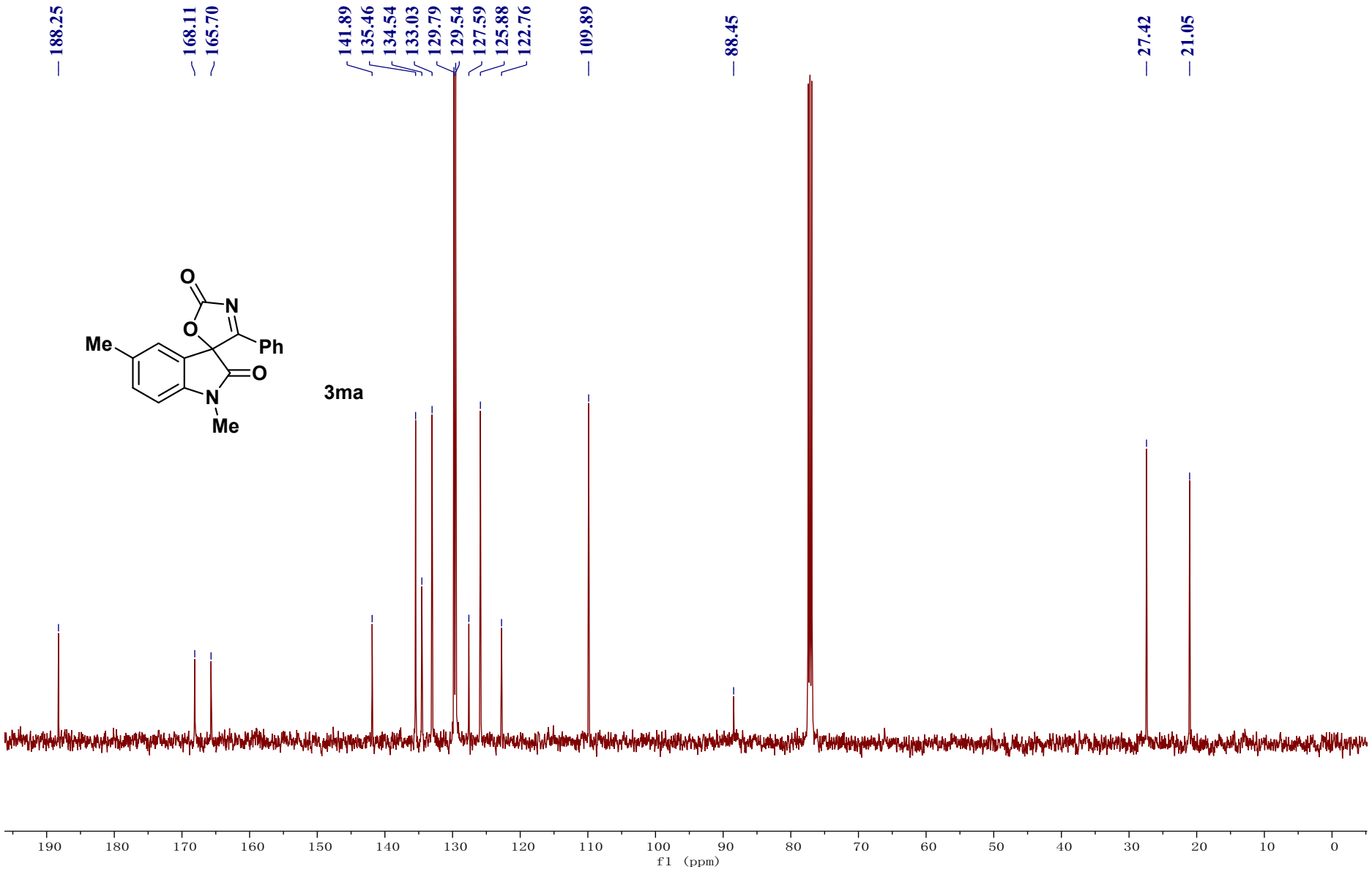
f1 (ppm)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O
N
O
Ph
Me
O
N
Me
3ma
190
180
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90
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50
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10
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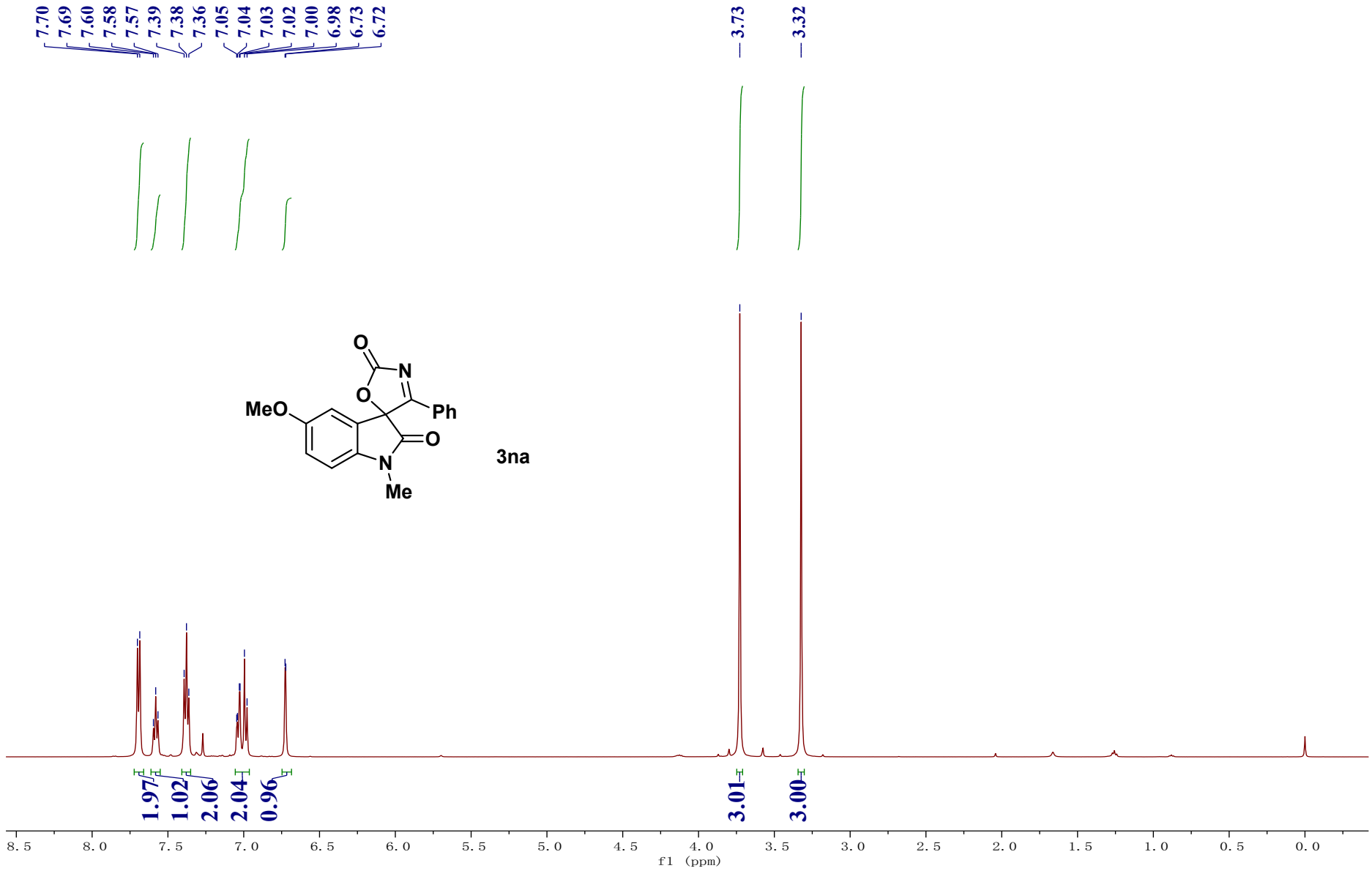
f1 (ppm)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7.03
7.02
7.00
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6.73
6.72
3.73
3.32
MeO
O
N
O
Ph
O
N
Me
3na
1.97
1.02
2.06
2.04
0.96
3.01
3.00
8.5
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7.0
6.5
6.0
5.5
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4.5
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2.0
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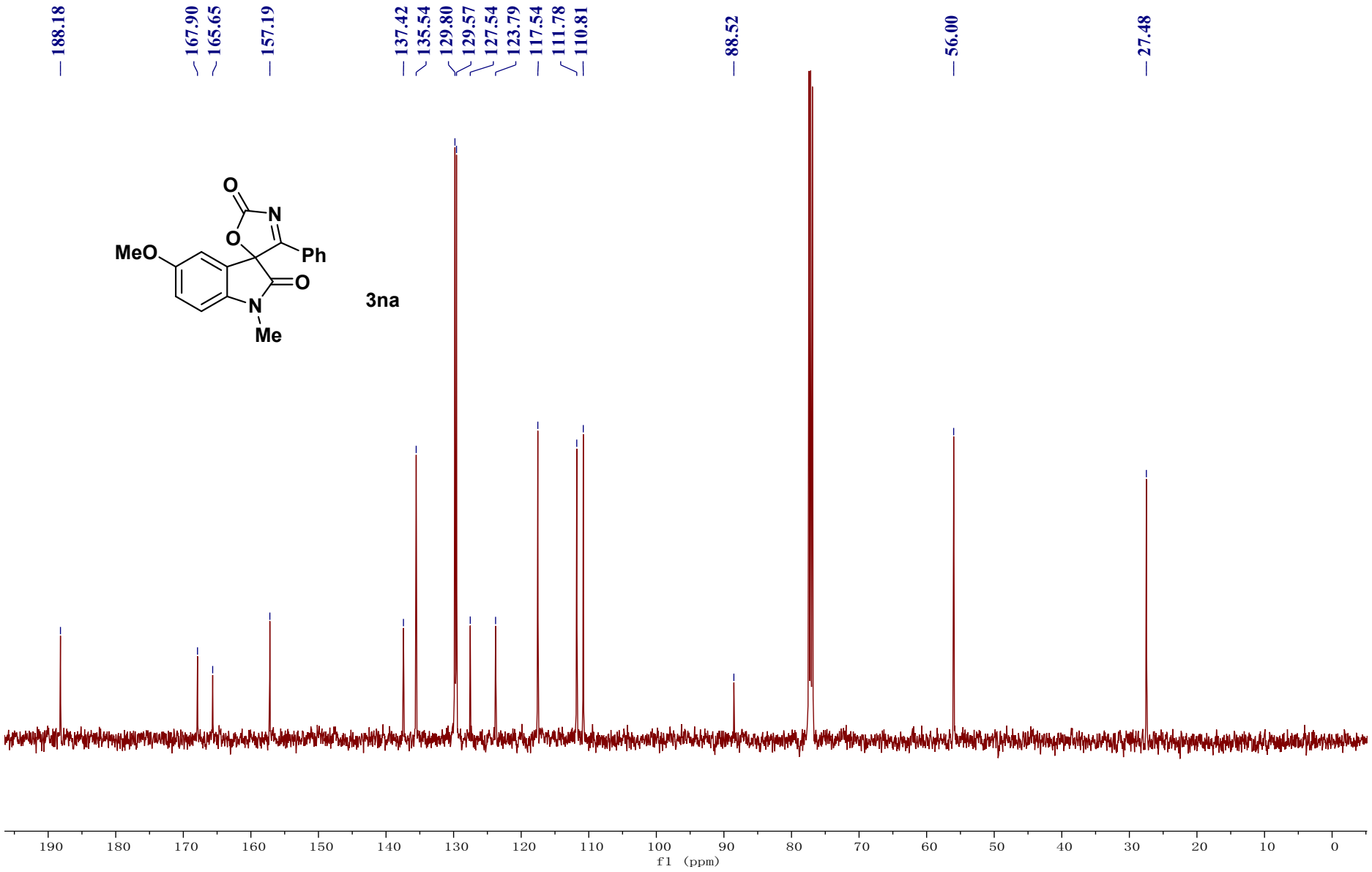
f1 (ppm)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27.48
O
N
O
Ph
MeO
O
N
Me
3na
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180
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90
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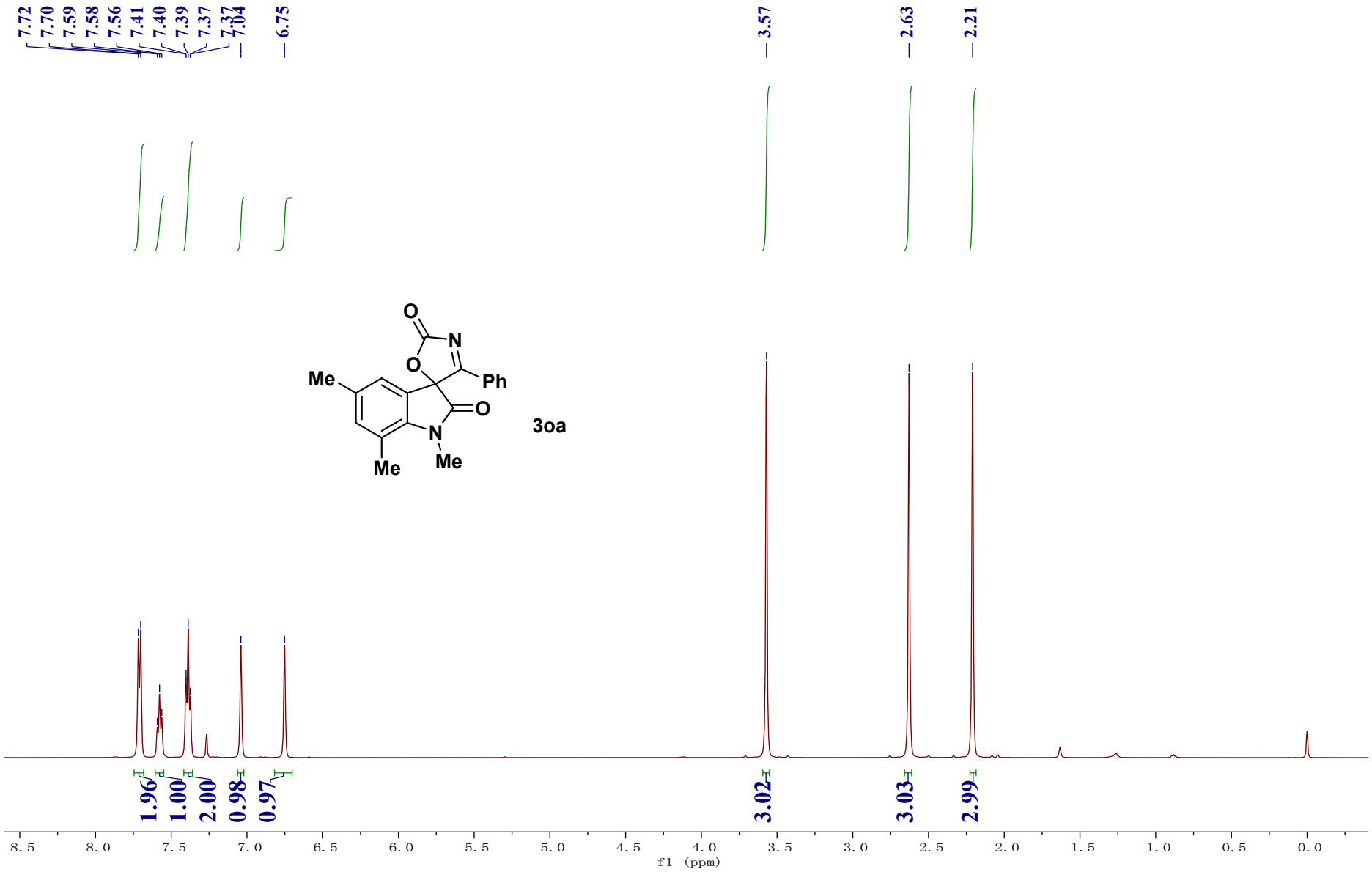
f1 (ppm)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Me
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3.03
2.99
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7.0
6.5
6.0
5.5
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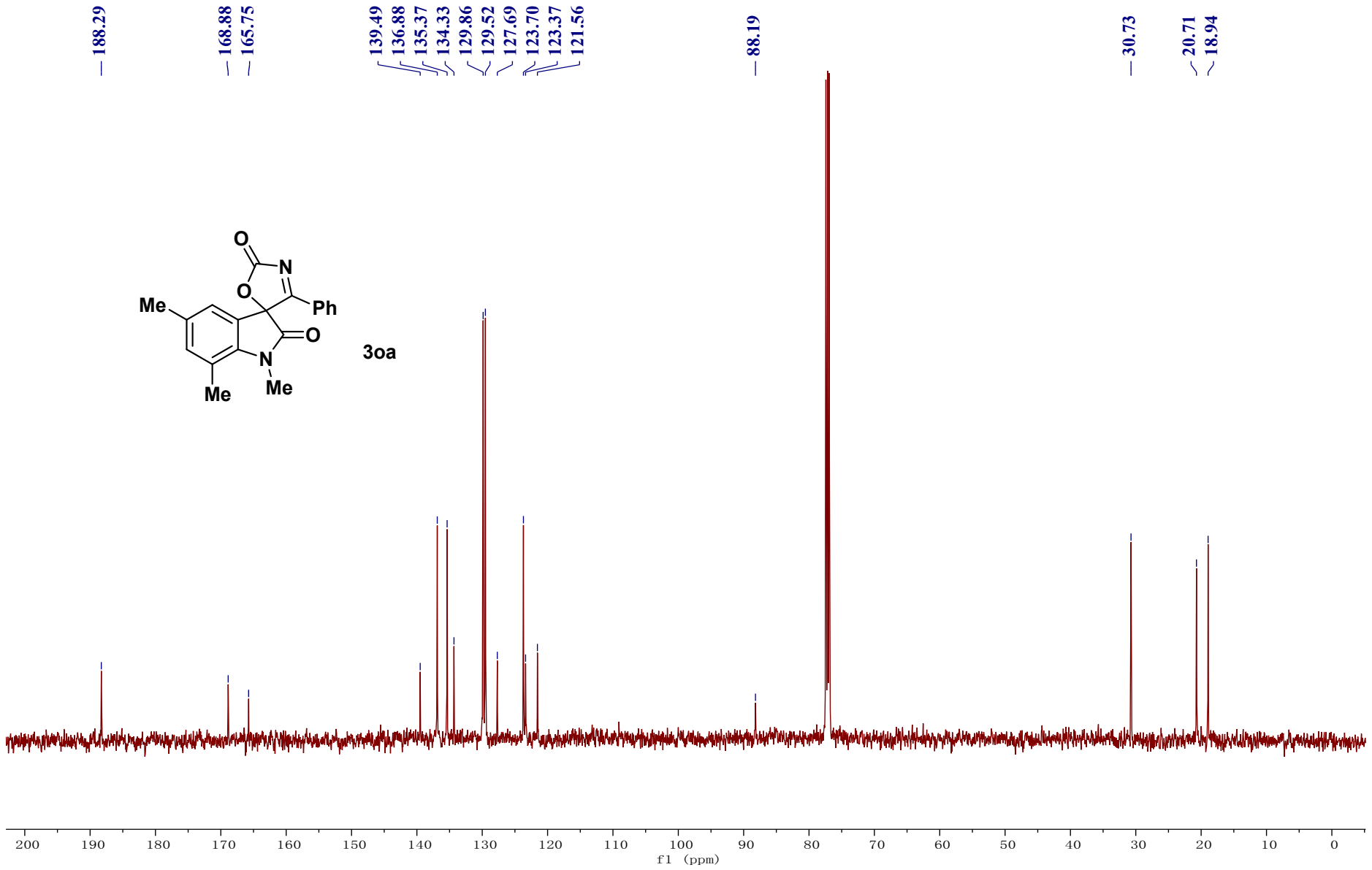
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88.19
30.73
20.71
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O
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Ph
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O
N
Me
Me
3oa
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180
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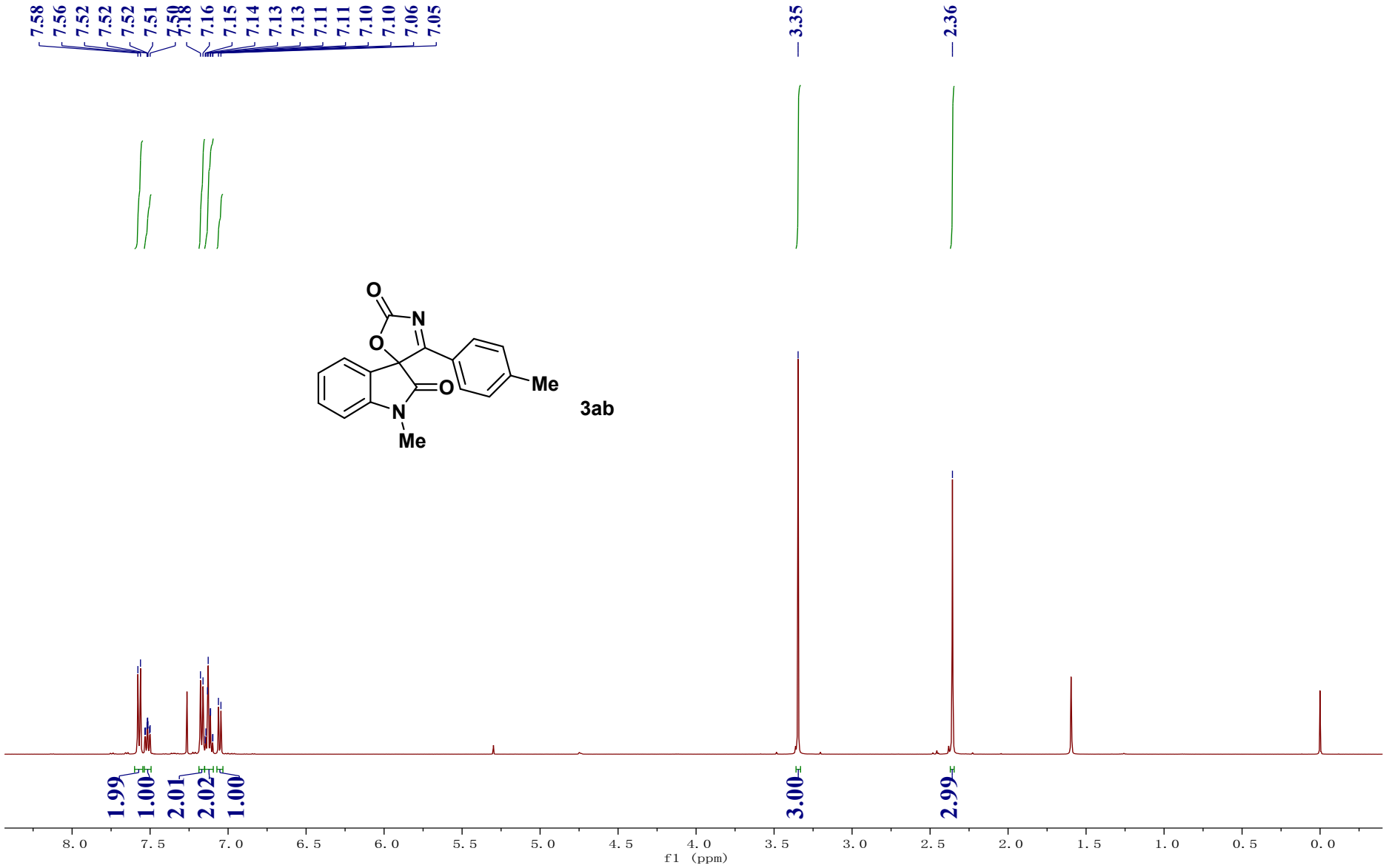
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7.14
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7.13
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7.11
7.10
7.10
7.06
7.05
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2.0
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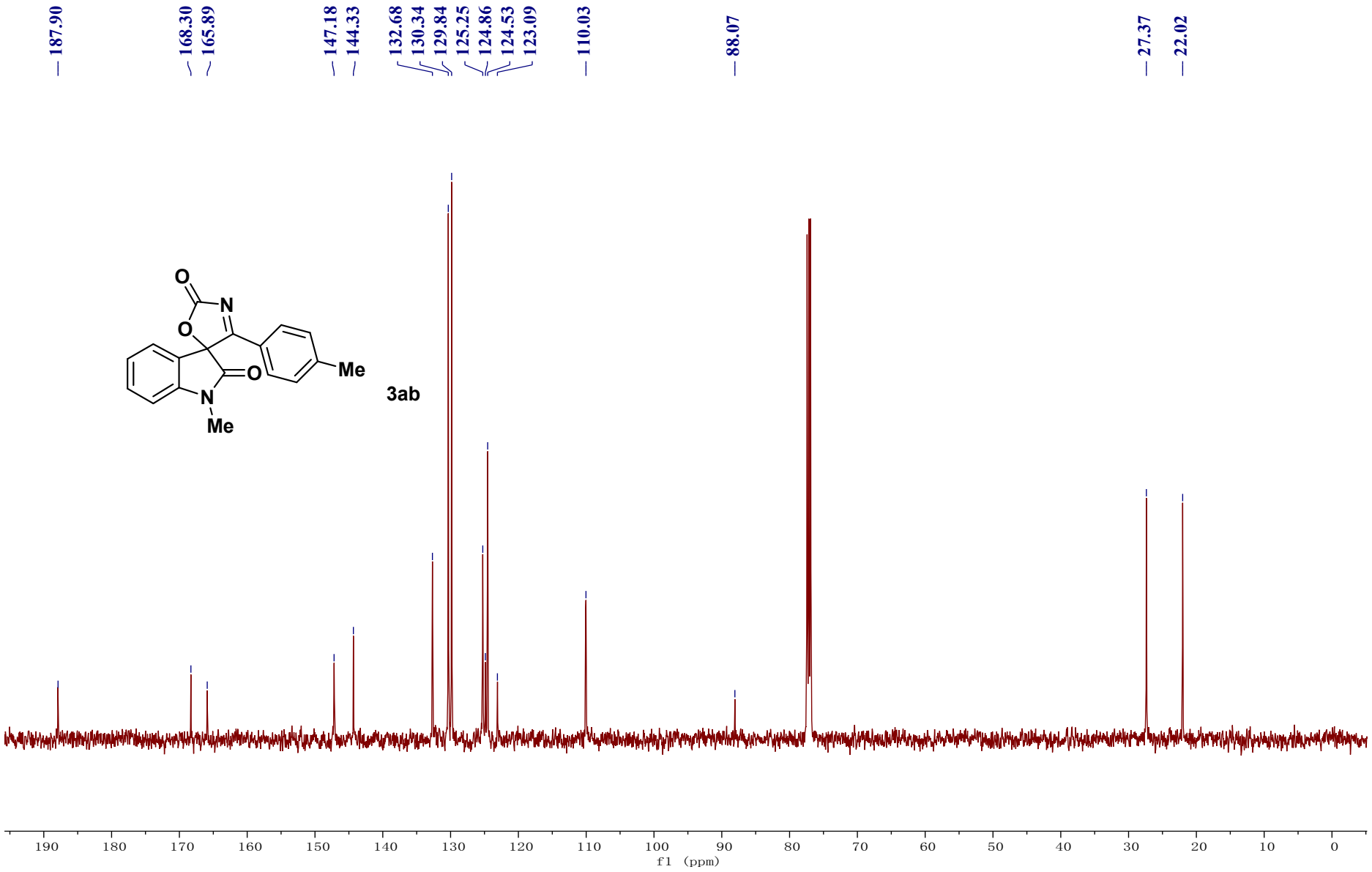
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123.09
110.03
88.07
27.37
22.02
O
N
O
O
N
Me
Me
3ab
190
180
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110
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50
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30
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10
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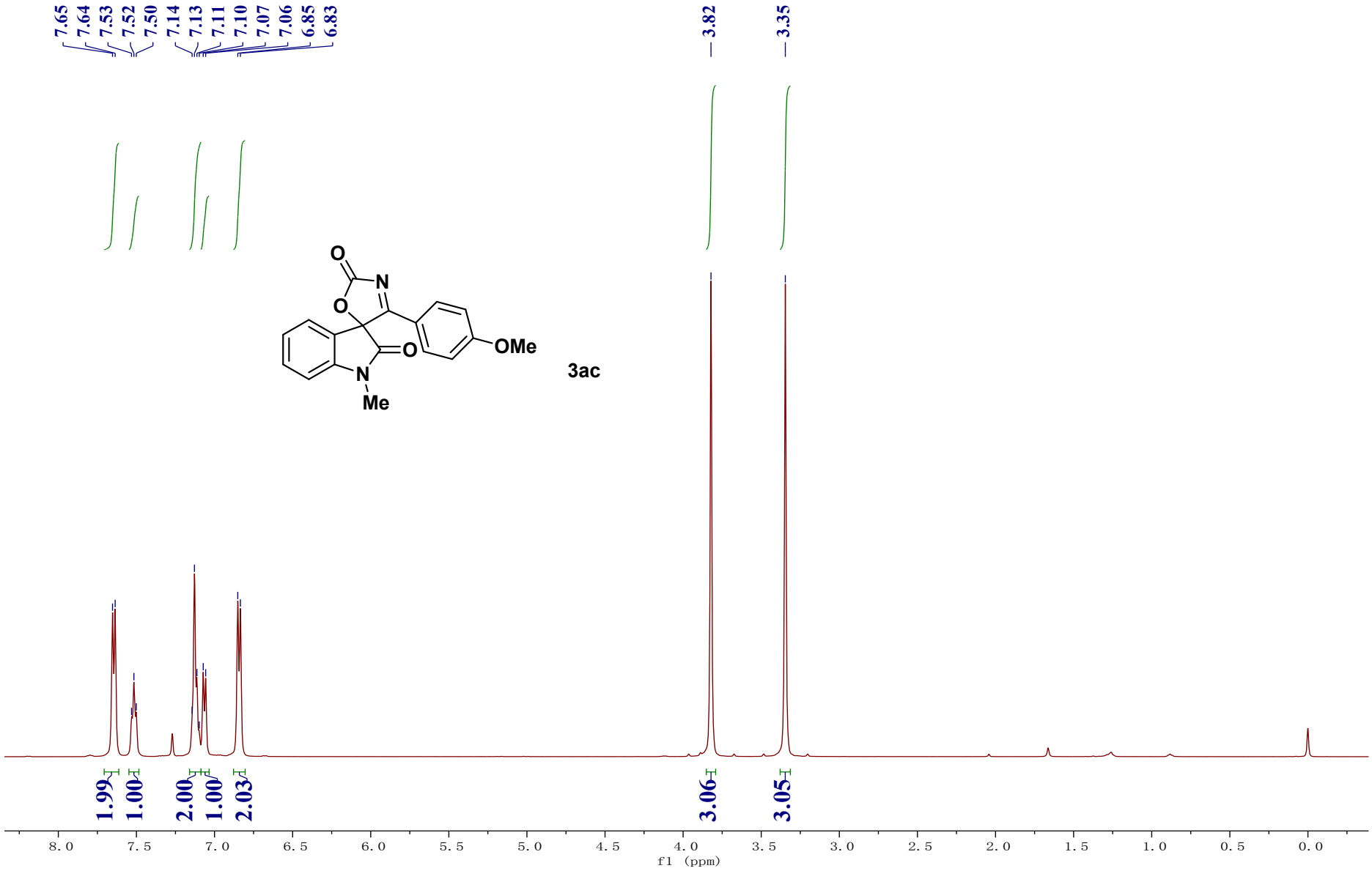
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6.83
3.82
3.35
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N
O
O
N
Me
OMe
3ac
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1.00
2.00
1.00
2.03
3.06
3.05
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7.0
6.5
6.0
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3.0
2.5
2.0
1.5
1.0
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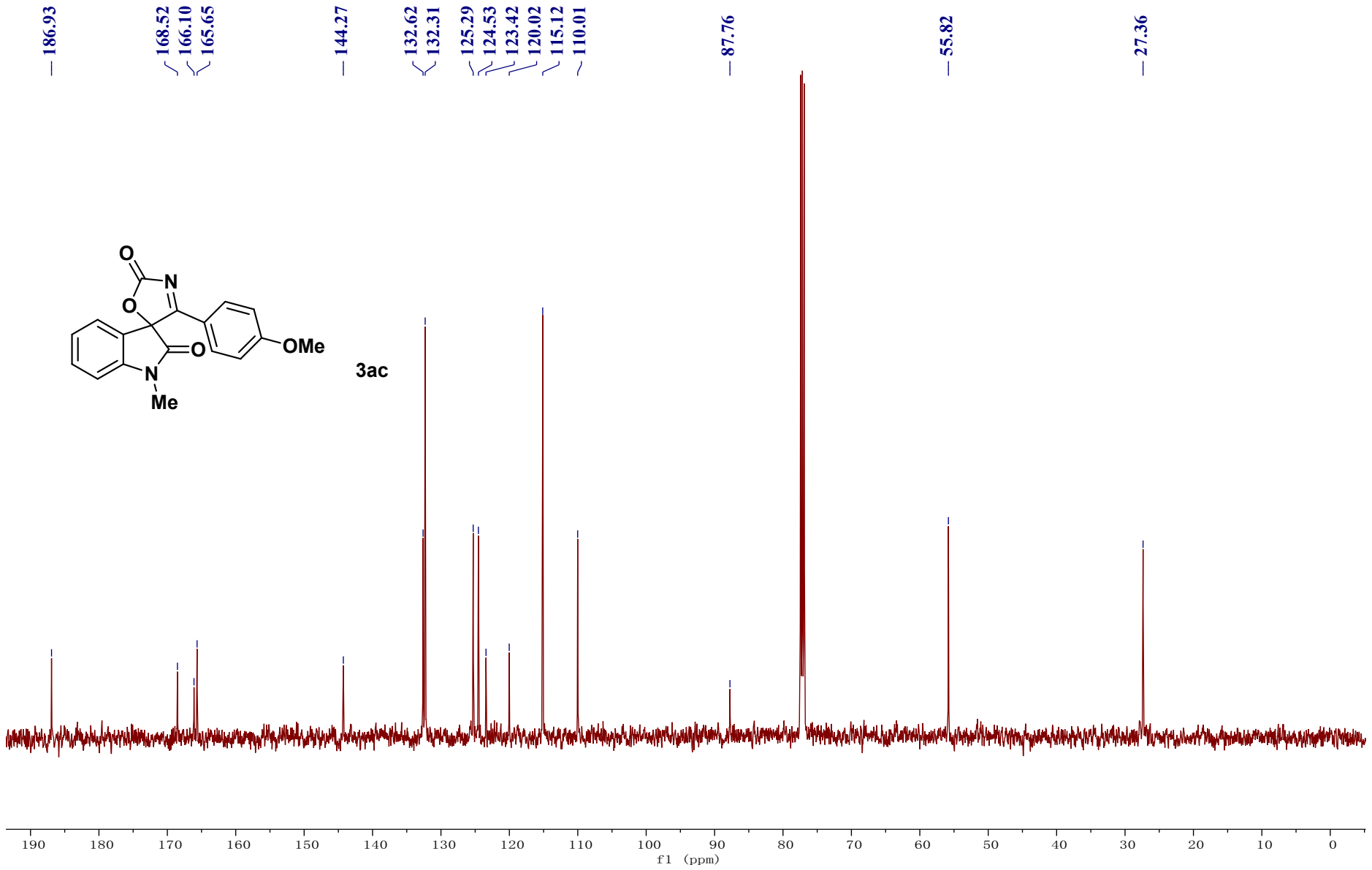
f1 (ppm)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123.42
120.02
115.12
110.01
87.76
55.82
27.36
O
N
O
O
N
Me
OMe
3ac
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180
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110
100
90
80
70
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50
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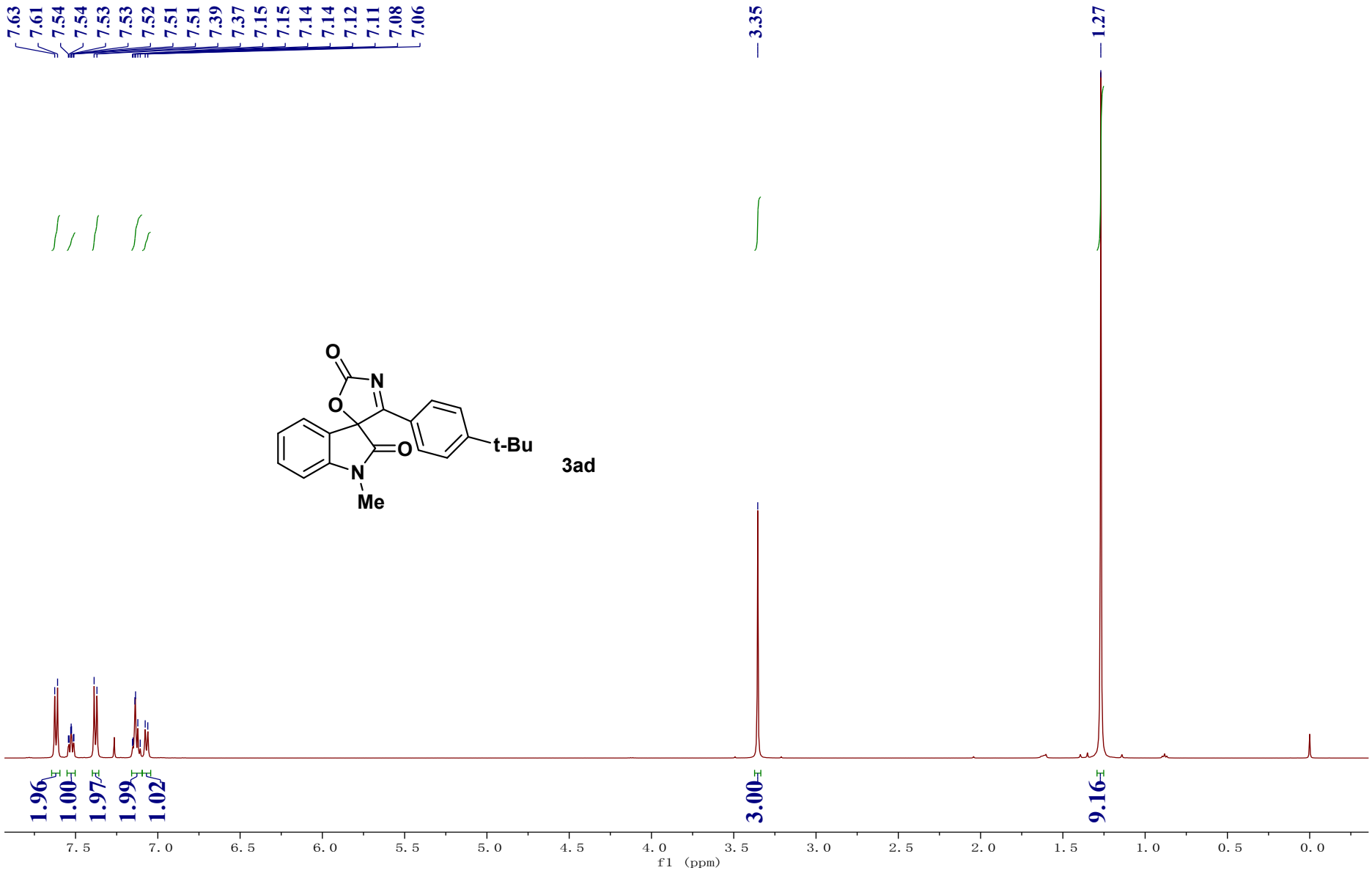
f1 (ppm)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7.14
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7.08
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1.99
1.02
3.00
9.16
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6.0
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1.0
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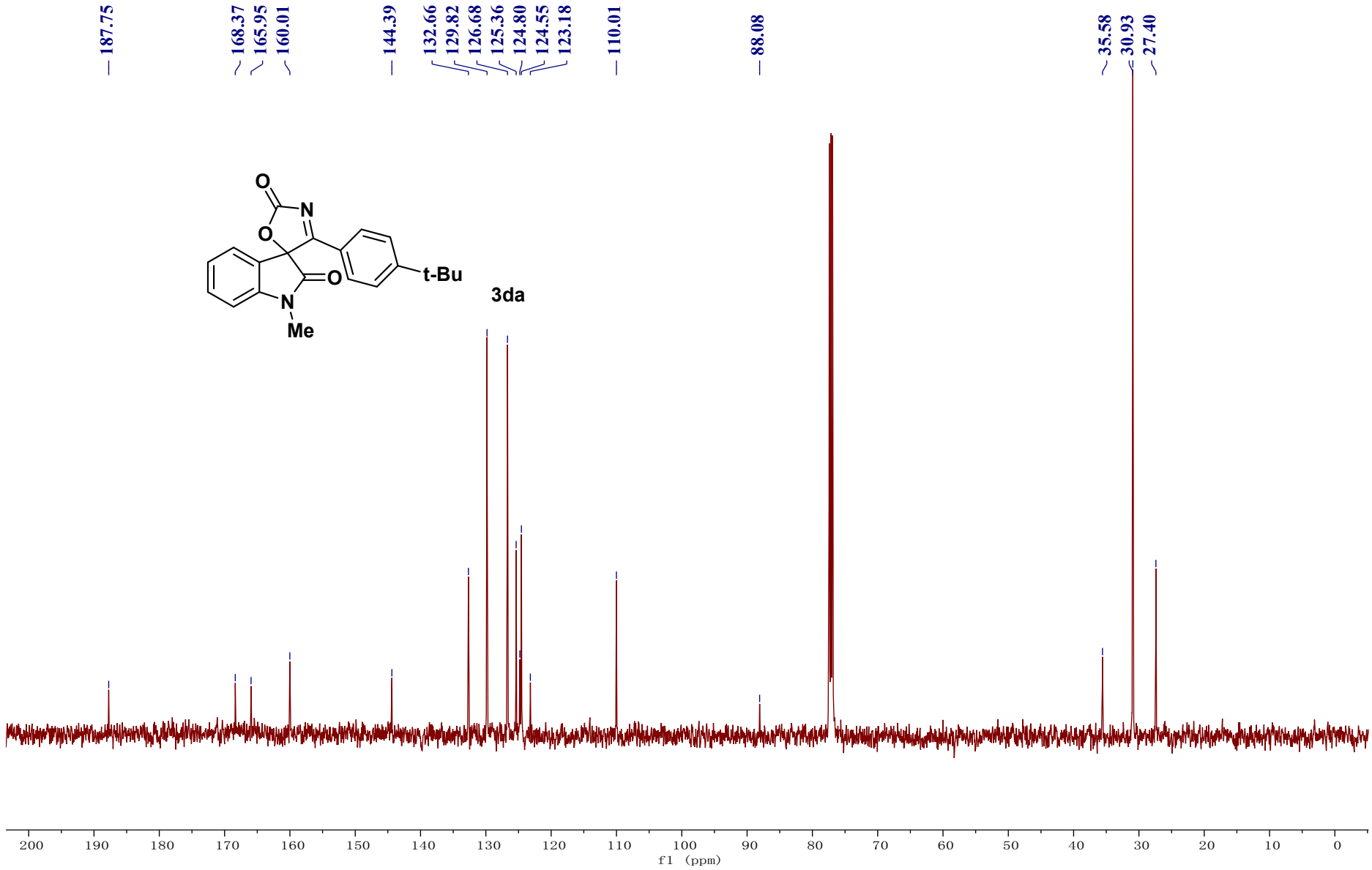
f1 (ppm)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88.08
35.58
30.93
27.40
O
N
O
O
N
Me
t-Bu
3da
200
190
180
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110
100
90
80
70
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50
40
30
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10
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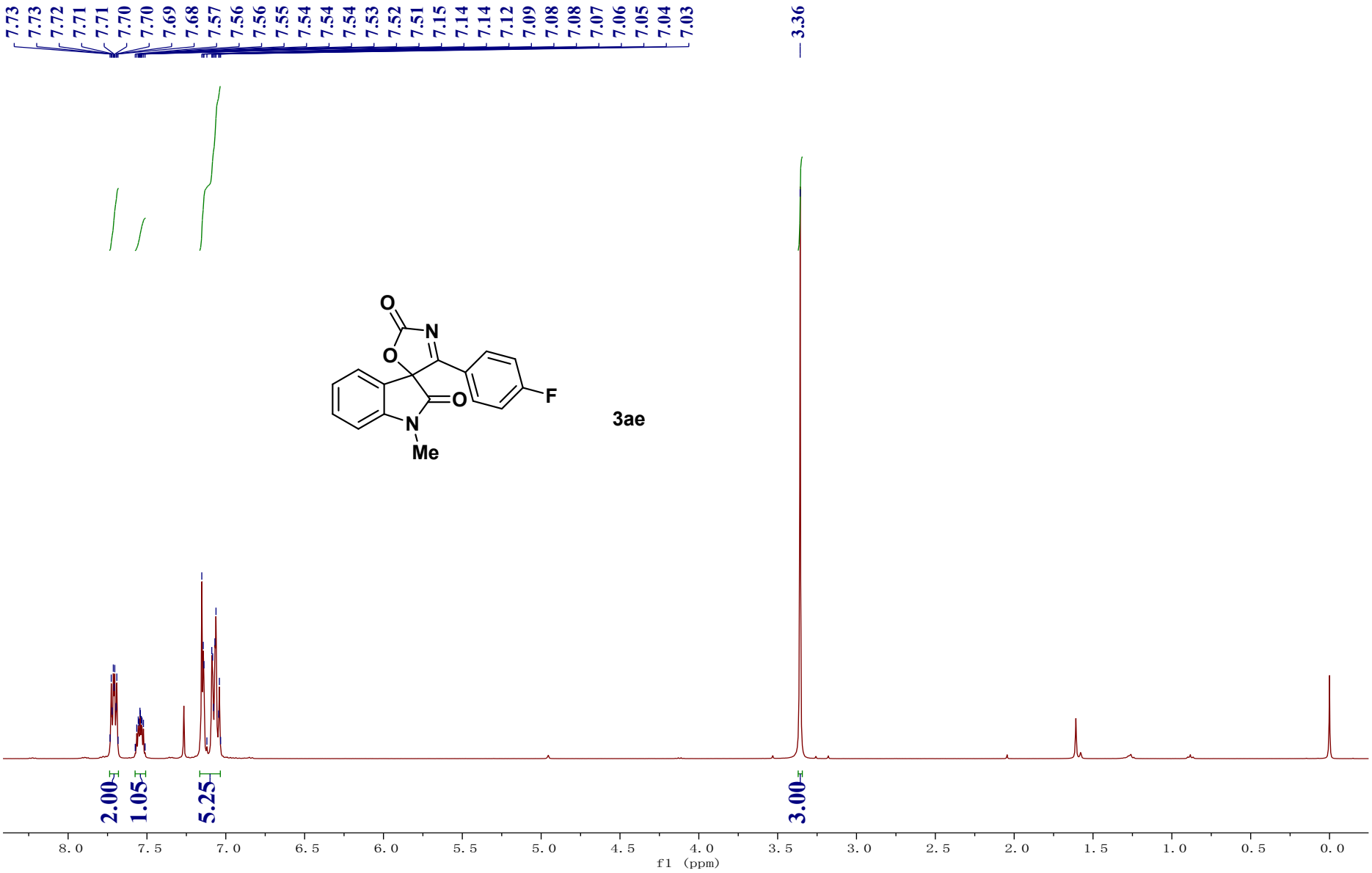
f1 (ppm)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7.09
7.08
7.08
7.07
7.06
7.05
7.04
7.03
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6.0
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3.0
2.5
2.0
1.5
1.0
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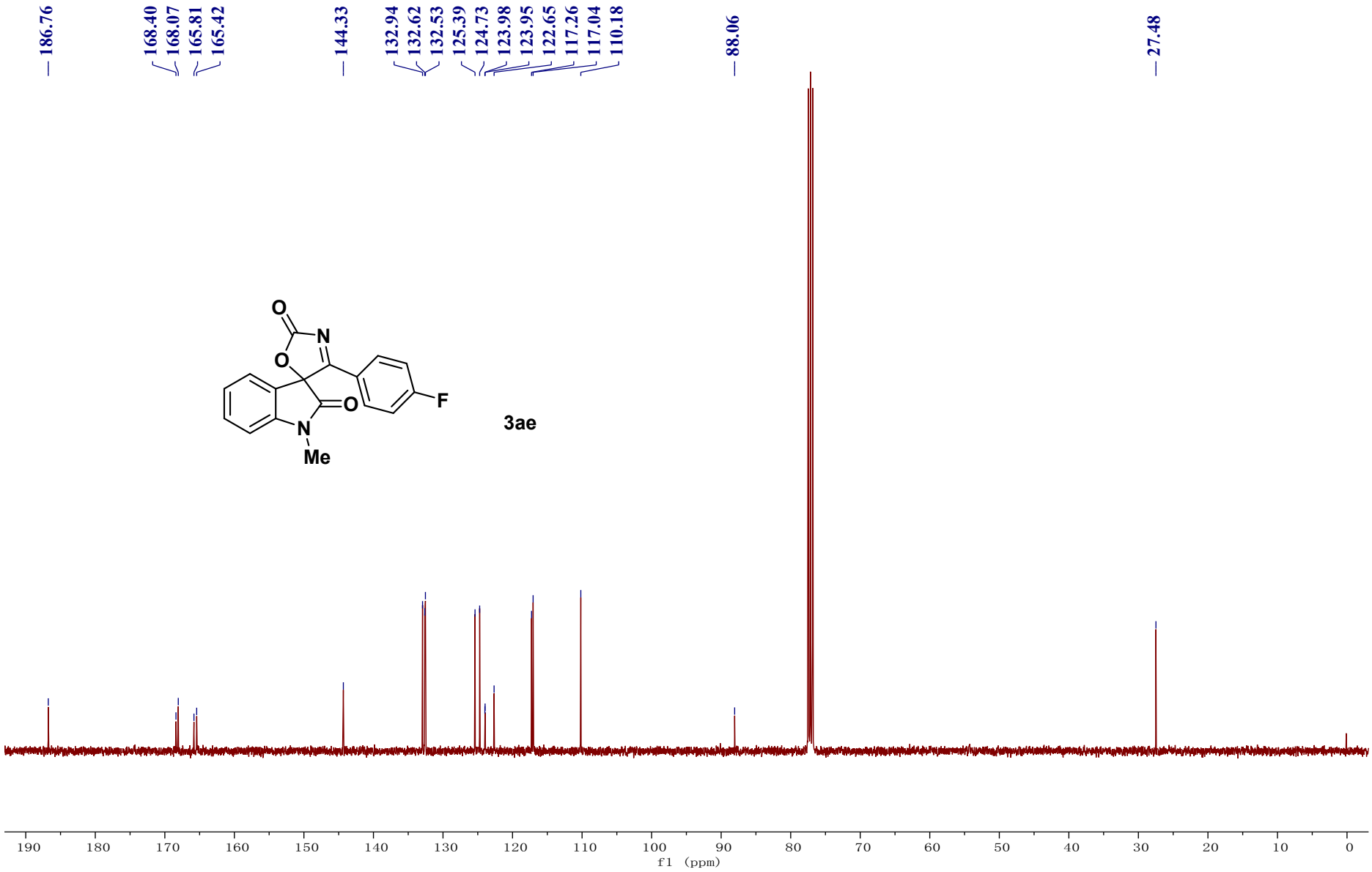
0.0
f1 (ppm)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123.95
122.65
117.26
117.04
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88.06
27.48
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N
O
O
F
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Me
3ae
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180
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100
90
80
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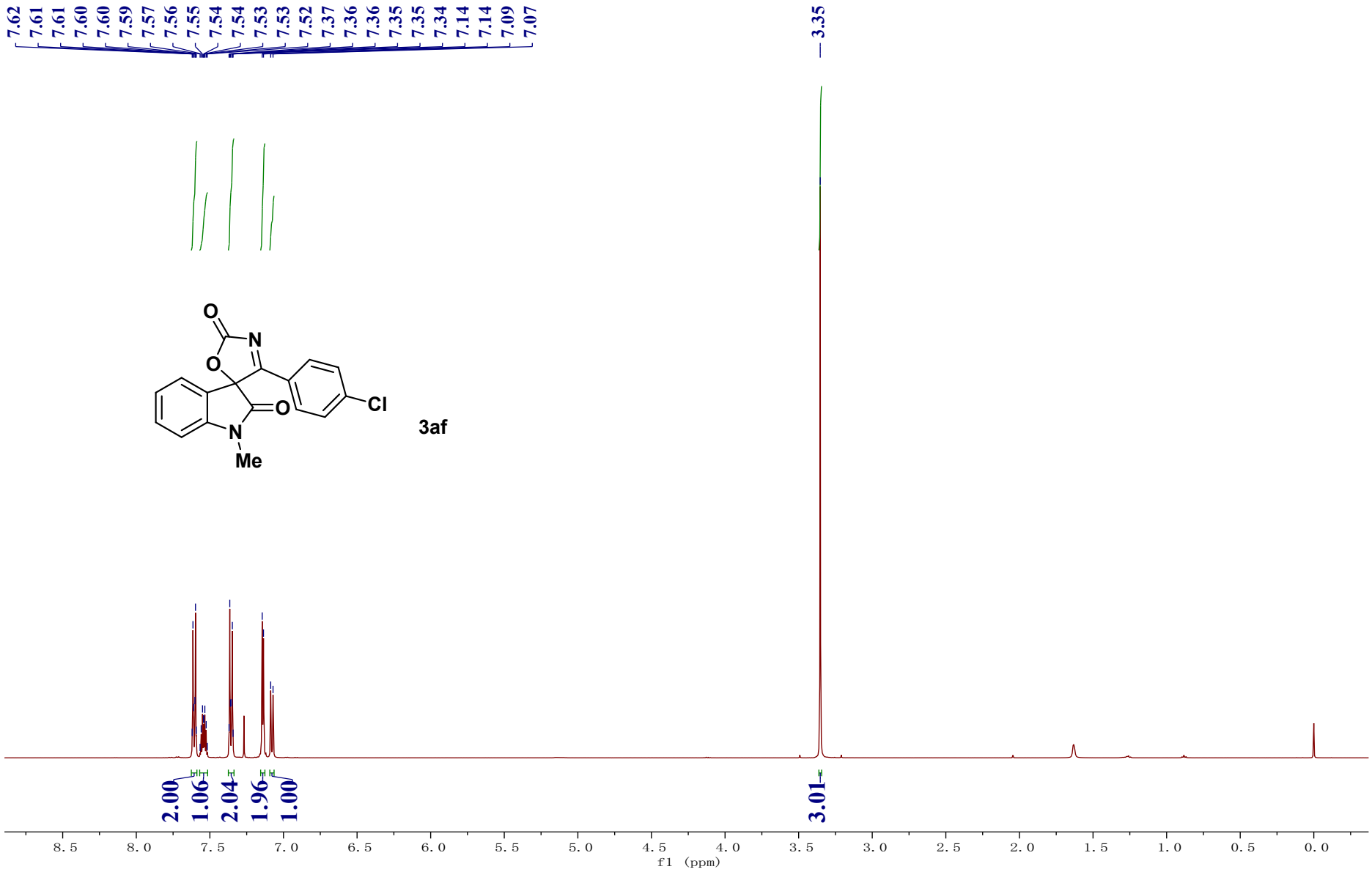
10
0
f1 (ppm)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7.07
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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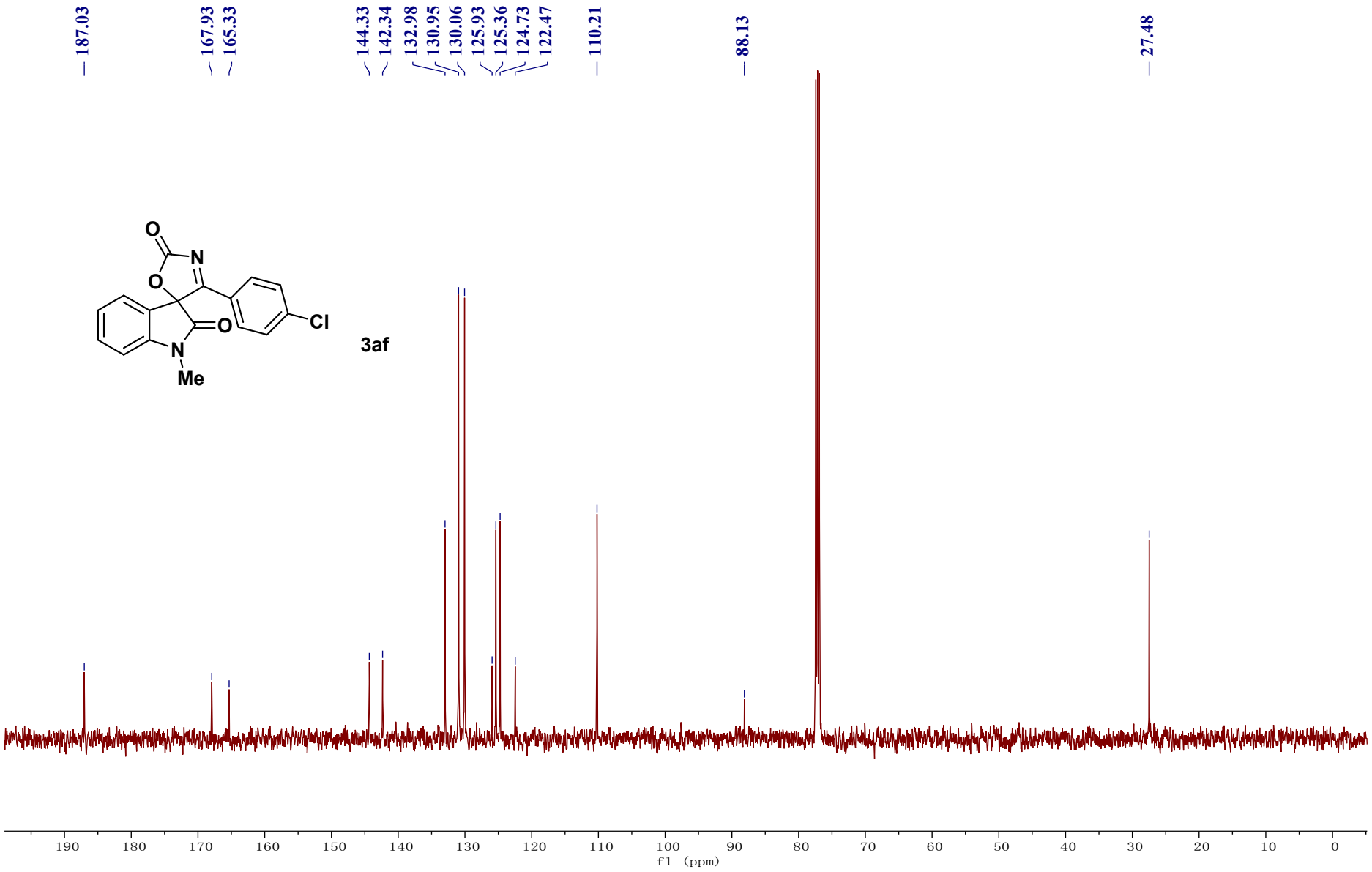
f1 (ppm)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125.93
125.36
124.73
122.47
110.21
88.13
27.48
O
N
O
O
N
Me
Cl
3af
190
180
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110
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90
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70
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50
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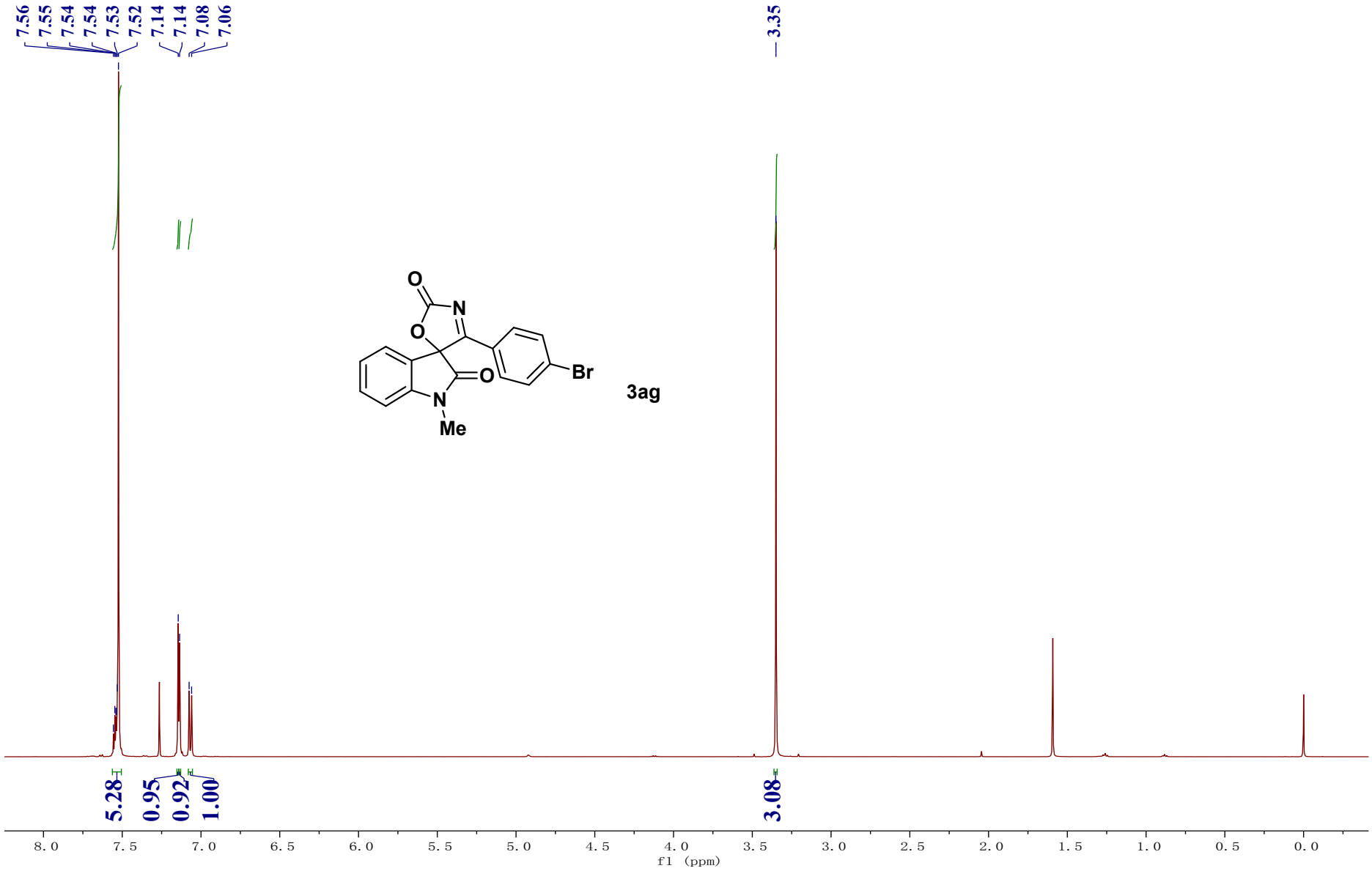
f1 (ppm)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O
O
N
Me
Br
3ag
5.28
0.95
0.92
1.00
3.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
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2.0
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1.0
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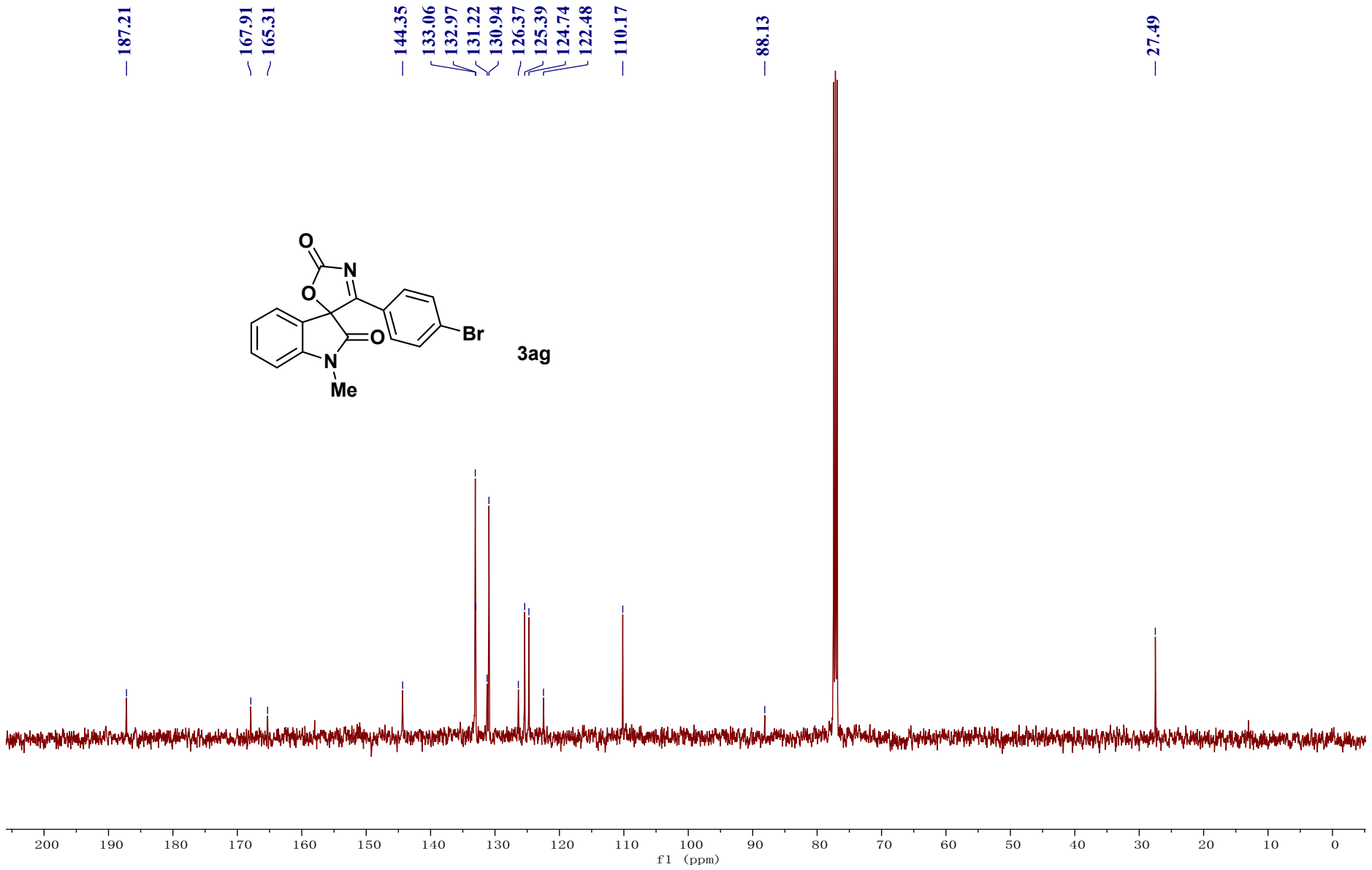
0.0
f1 (ppm)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131.22
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125.39
124.74
122.48
110.17
88.13
27.49
O
N
O
O
N
Me
Br
3ag
200
190
180
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100
90
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50
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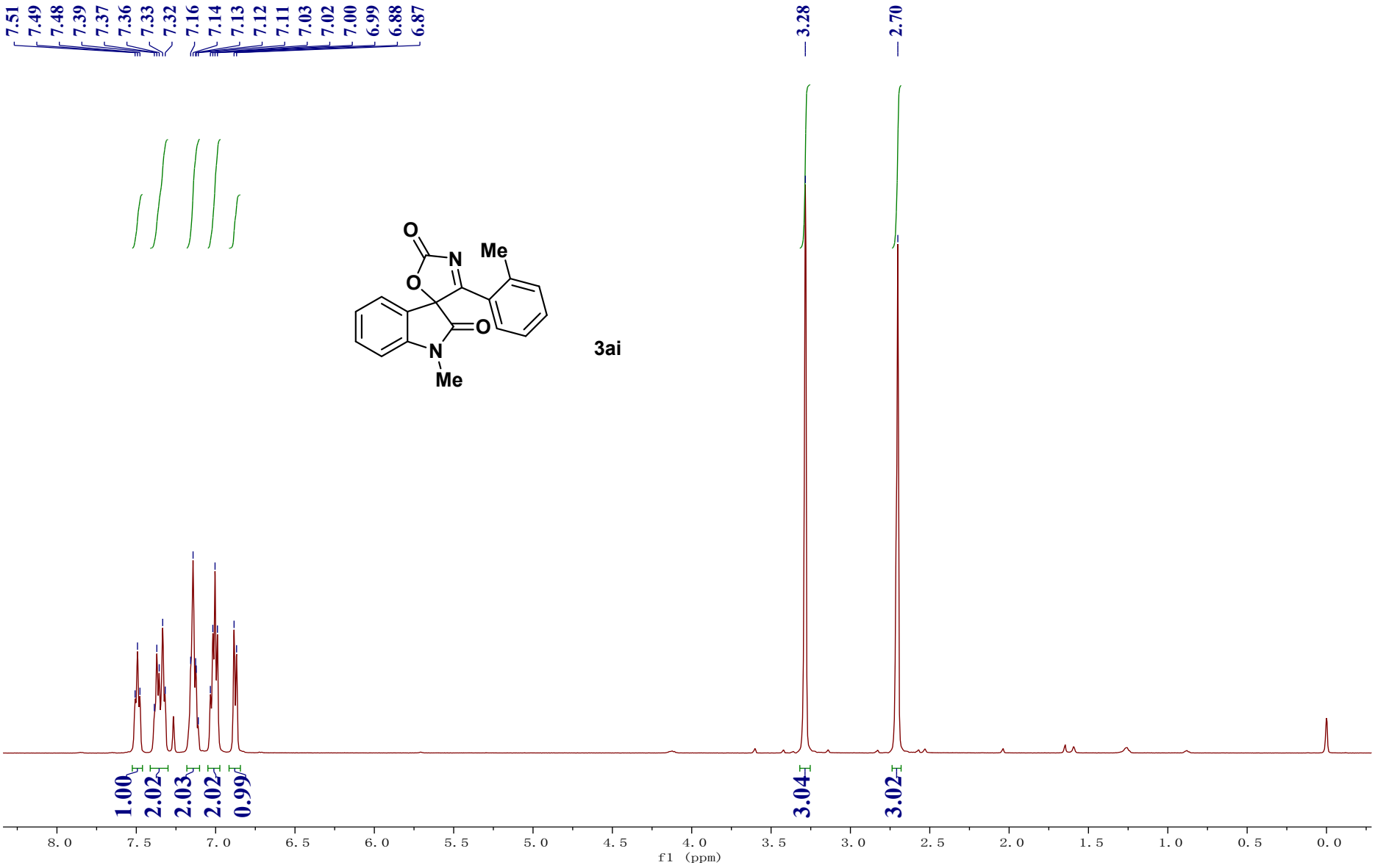
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7.32
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7.14
7.13
7.12
7.11
7.03
7.02
7.00
6.99
6.88
6.87
3.28
2.70
O
N
Me
O
O
N
Me
3ai
1.00
2.02
2.03
2.02
0.99
3.04
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
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1.0
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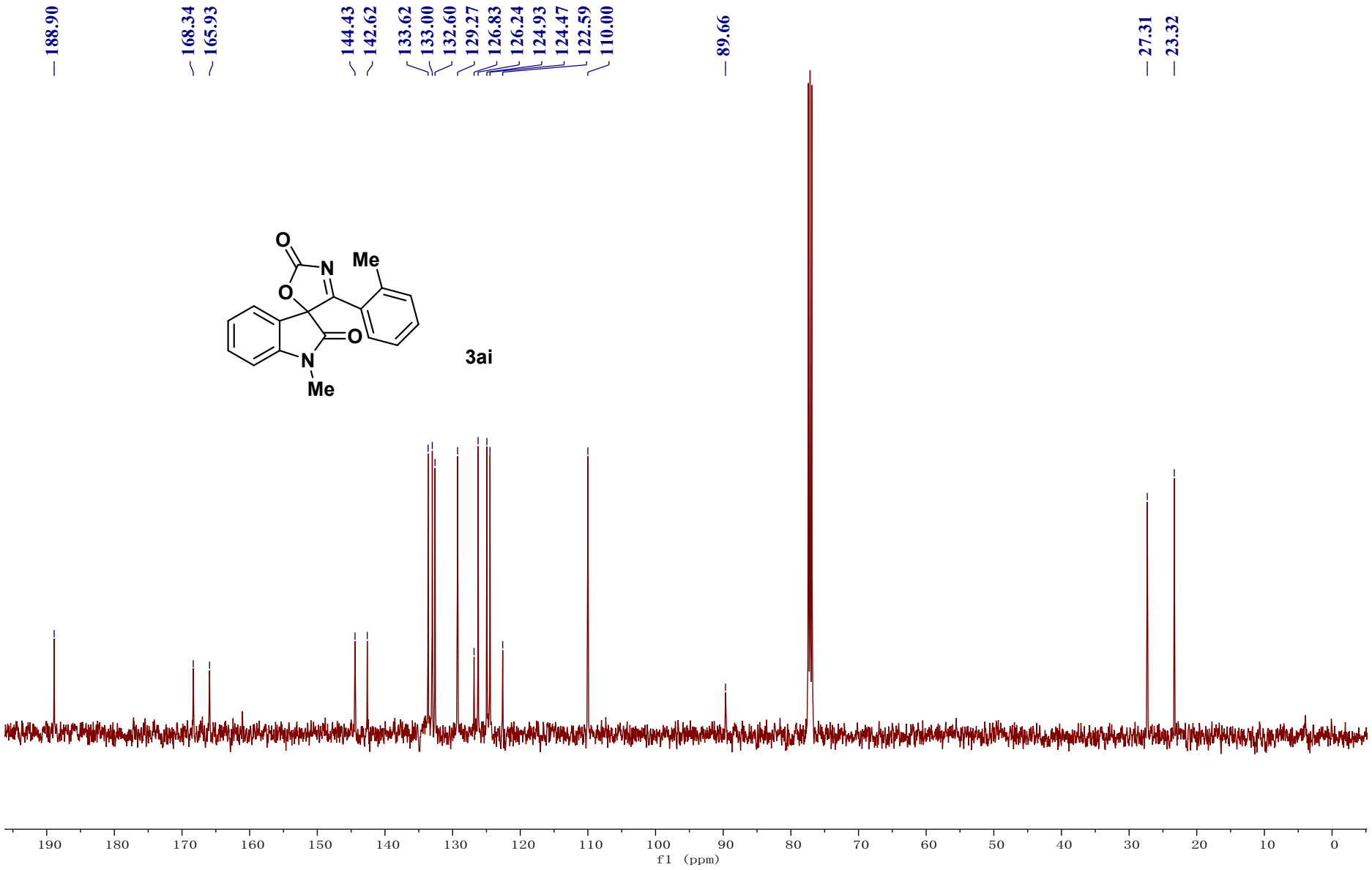
0.0
f1 (ppm)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126.24
124.93
124.47
122.59
110.00
89.66
27.31
23.32
O
N
Me
O
O
N
Me
3ai
190
180
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130
120
110
100
90
80
70
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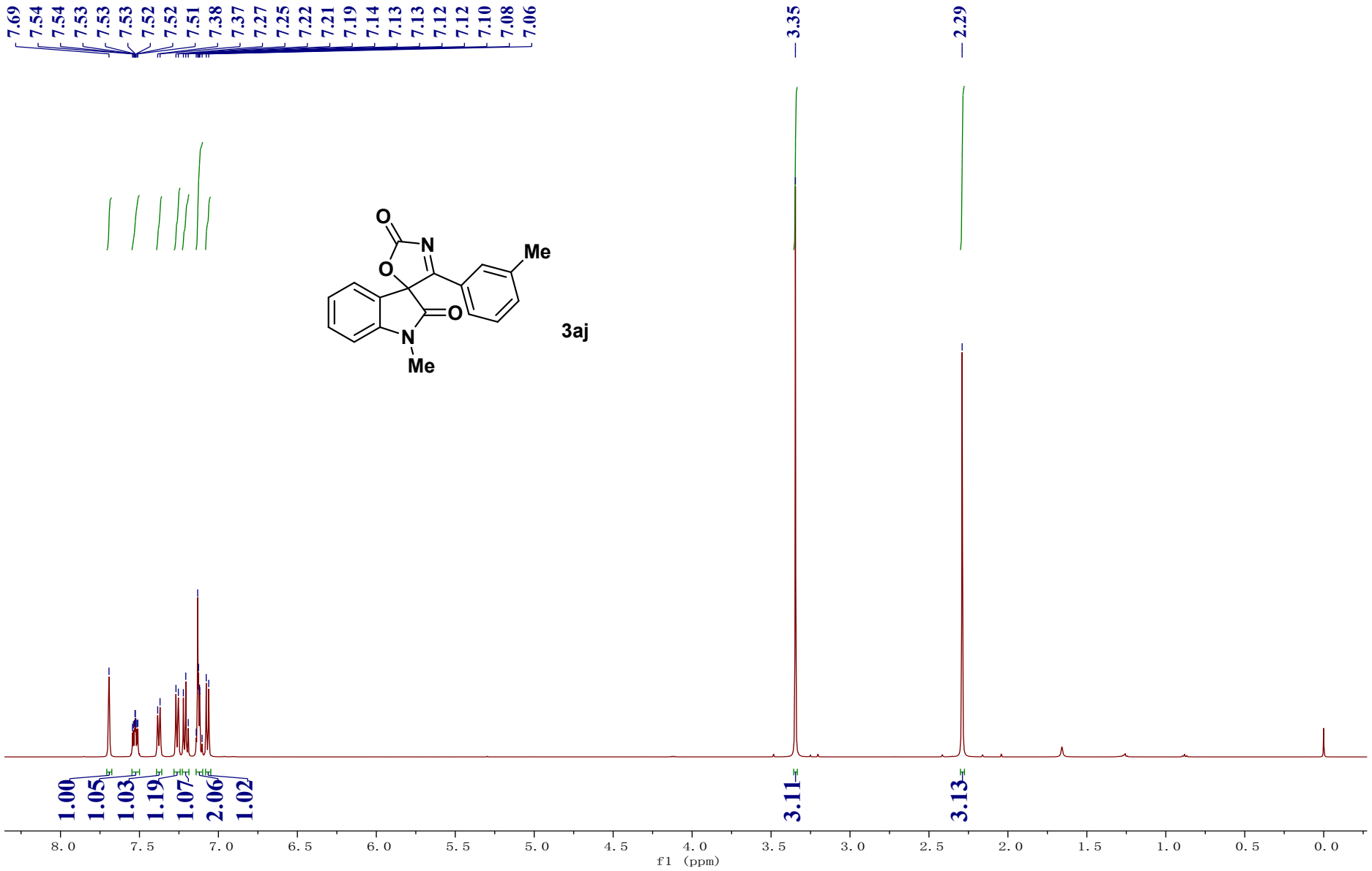
50
40
30
20
10
0
f1 (ppm)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7.27
7.25
7.22
7.21
7.19
7.14
7.13
7.13
7.12
7.12
7.10
7.08
7.06
3.35
2.29
O
N
O
Me
O
N
Me
3aj
1.00
1.05
1.03
1.19
1.07
2.06
1.02
3.11
3.13
8.0
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7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
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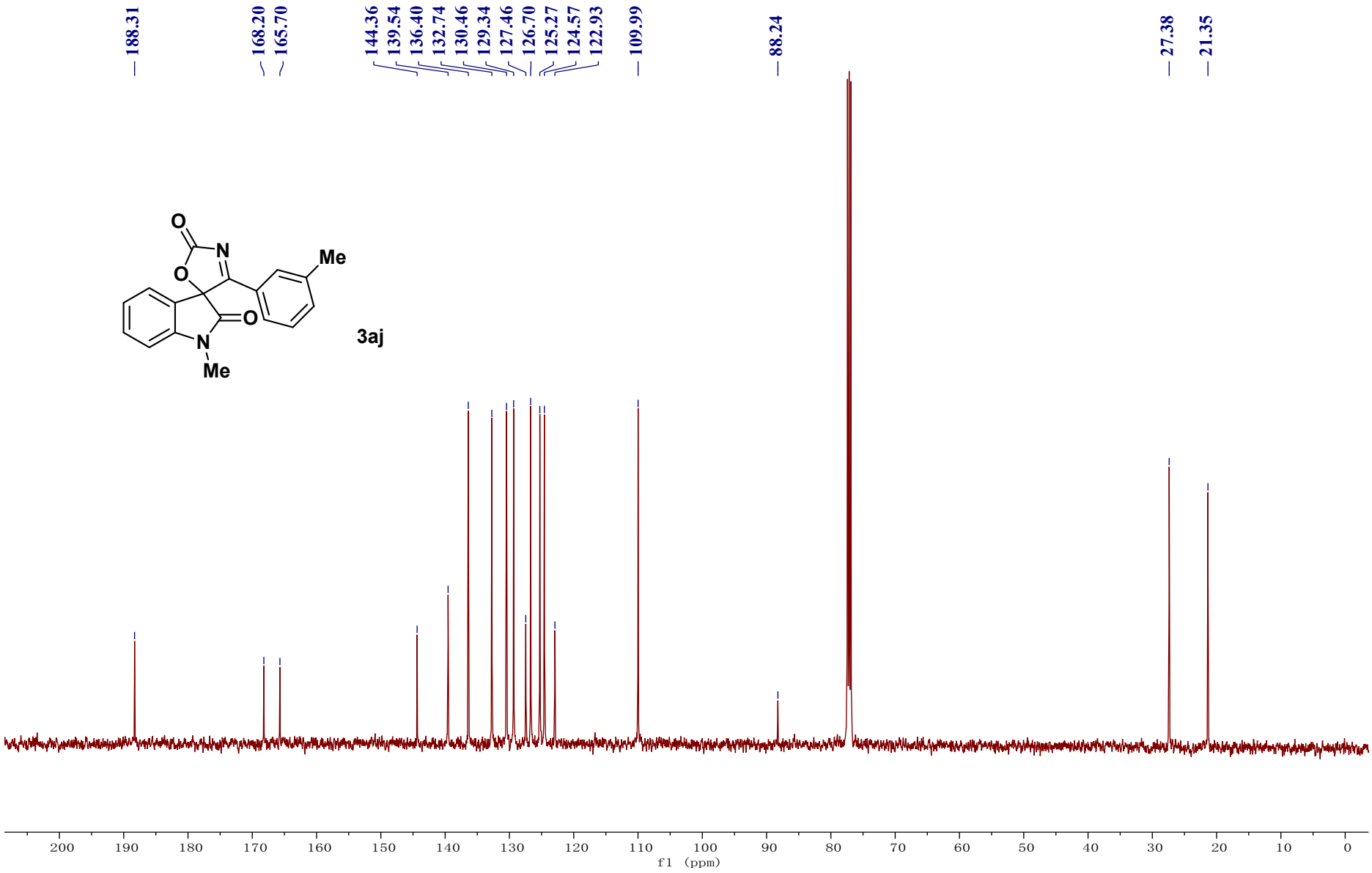
f1 (ppm)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130.46
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127.46
126.70
125.27
124.57
122.93
109.99
88.24
27.38
21.35
O
N
O
Me
O
N
Me
3aj
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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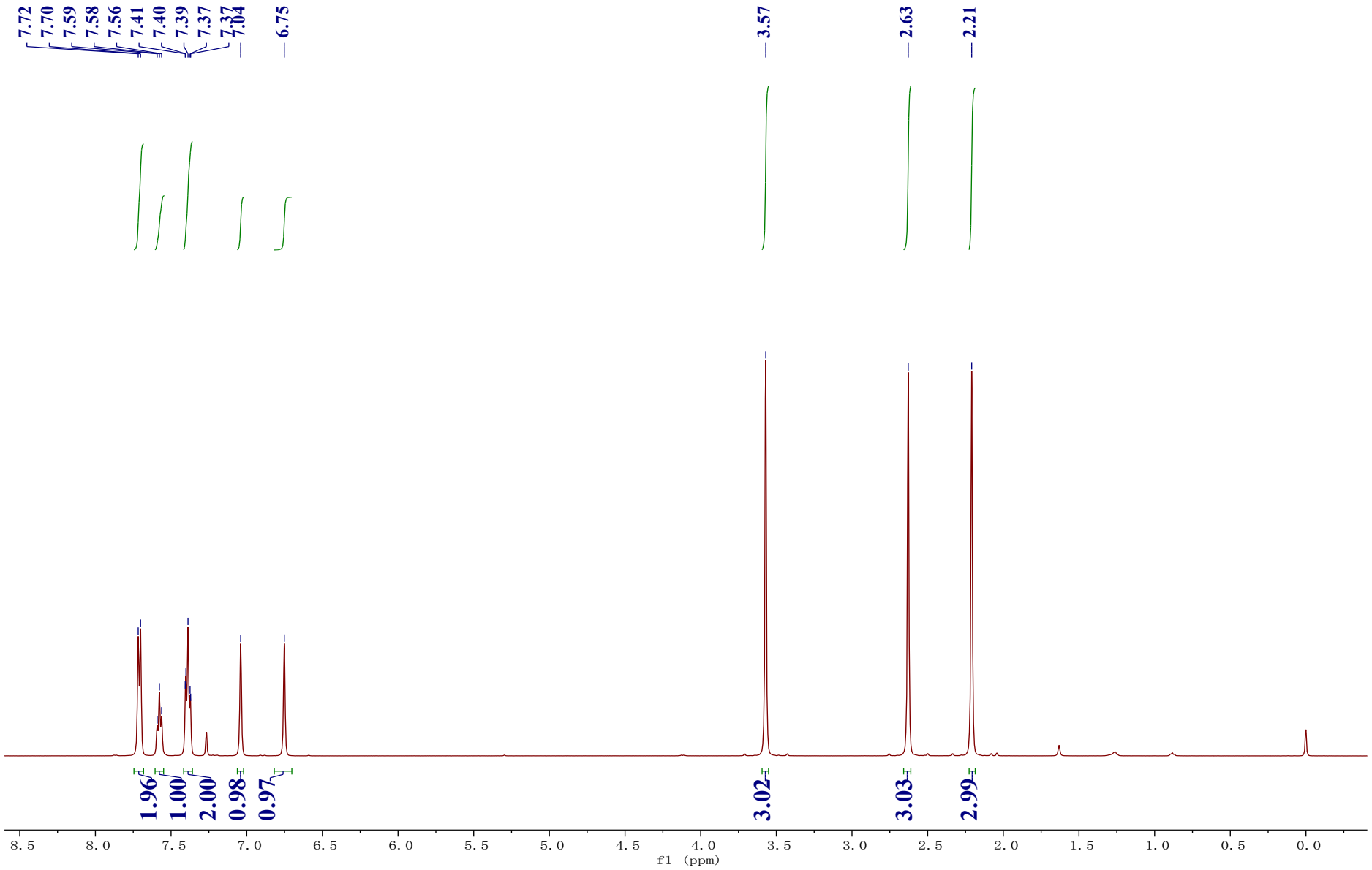
50
40
30
20
10
0
f1 (ppm)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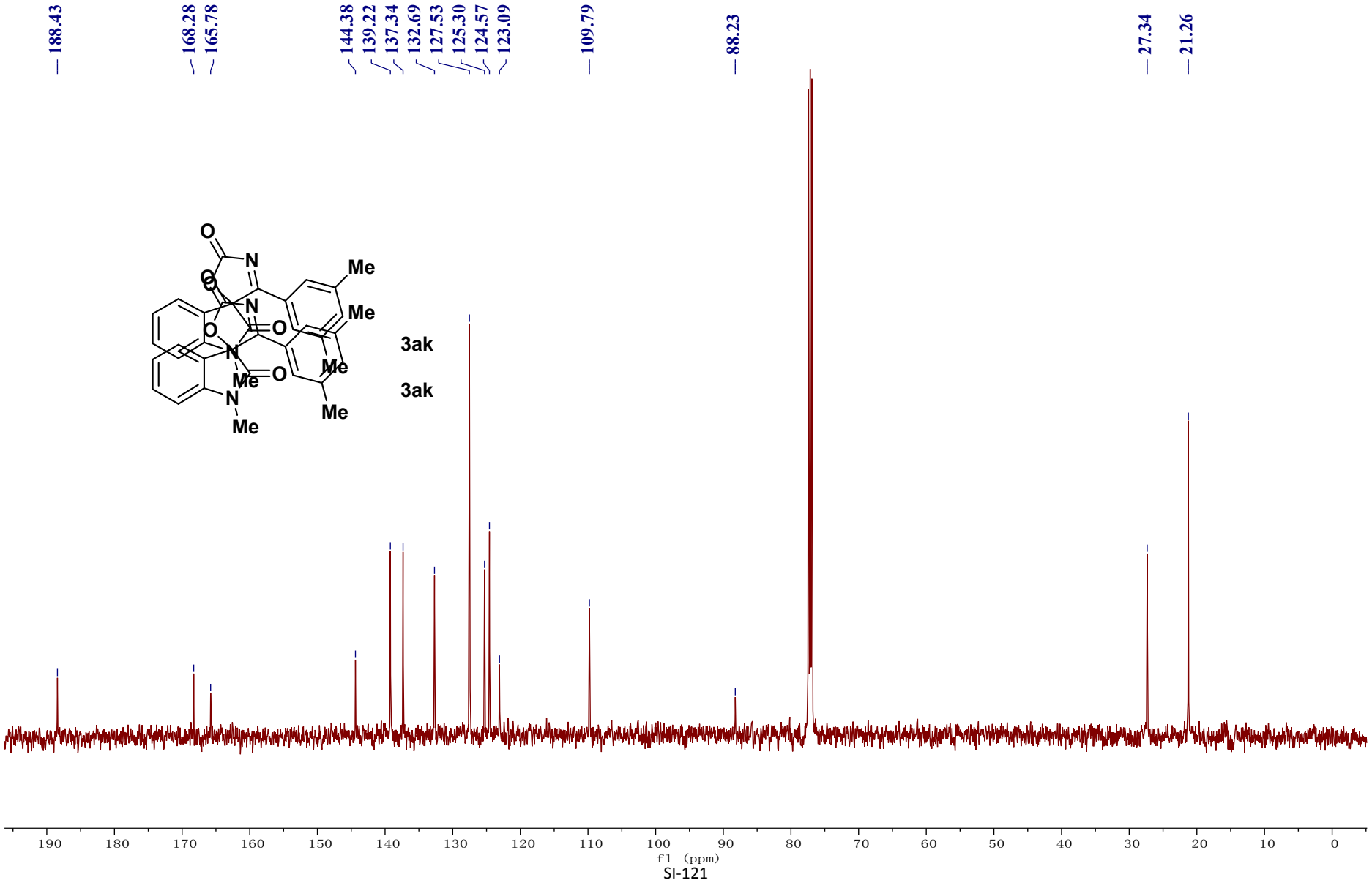
7.40
7.39
7.37
7.37
7.04
6.75
3.57
2.63
2.21
1.96
1.00
2.00
0.98
0.97
3.02
3.03
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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137.34
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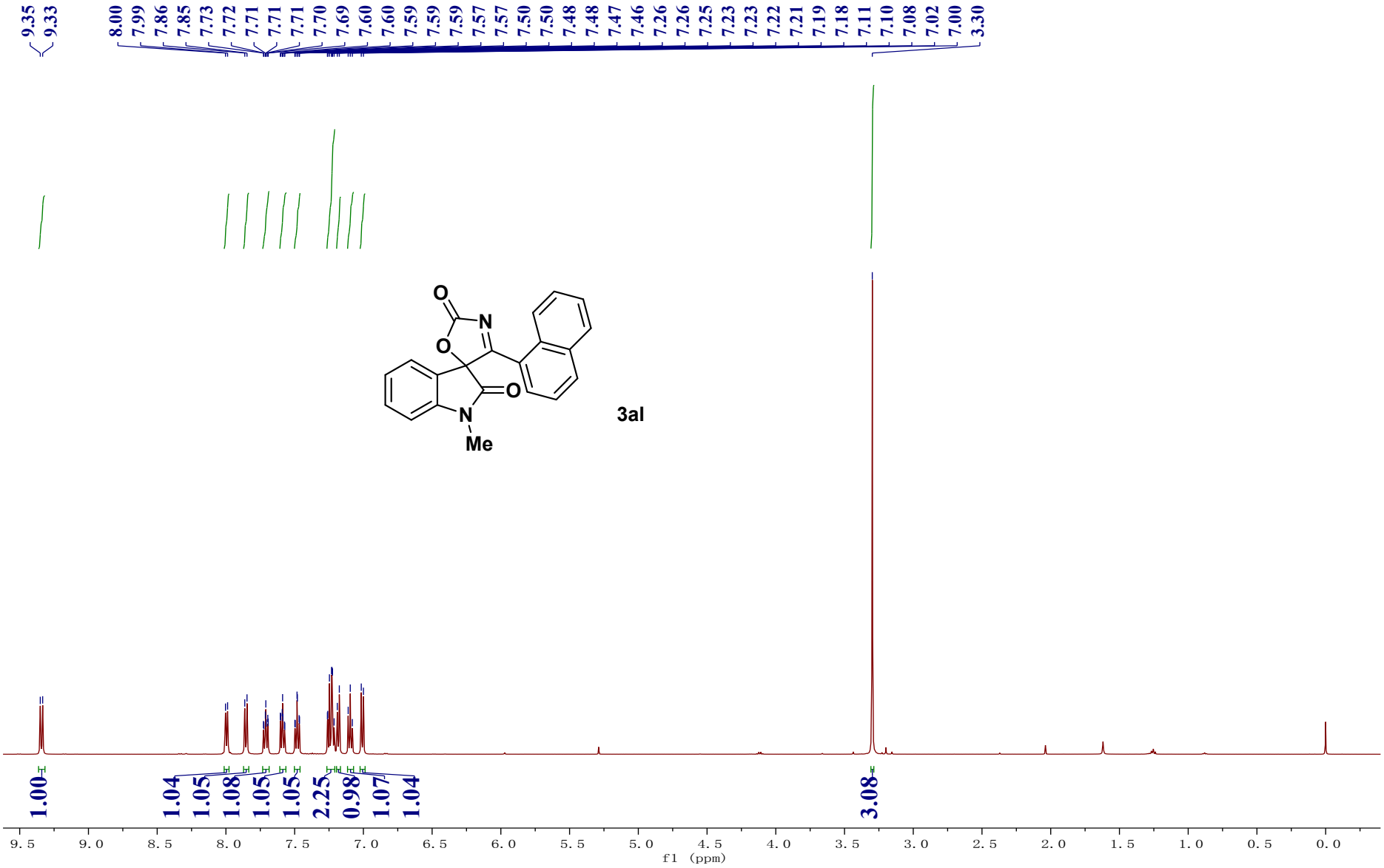
88.23
27.34
21.26
3ak
3ak
f1 (ppm)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7.85
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7.70
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7.60
7.59
7.59
7.59
7.57
7.57
7.50
7.50
7.48
7.48
7.47
7.46
7.26
7.26
7.25
7.23
7.23
7.22
7.21
7.19
7.18
7.11
7.10
7.08
7.02
7.00
3.30
O
N
O
O
N
Me
3al
1.00
1.04
1.05
1.08
1.05
1.05
2.25
0.98
1.07
1.04
3.08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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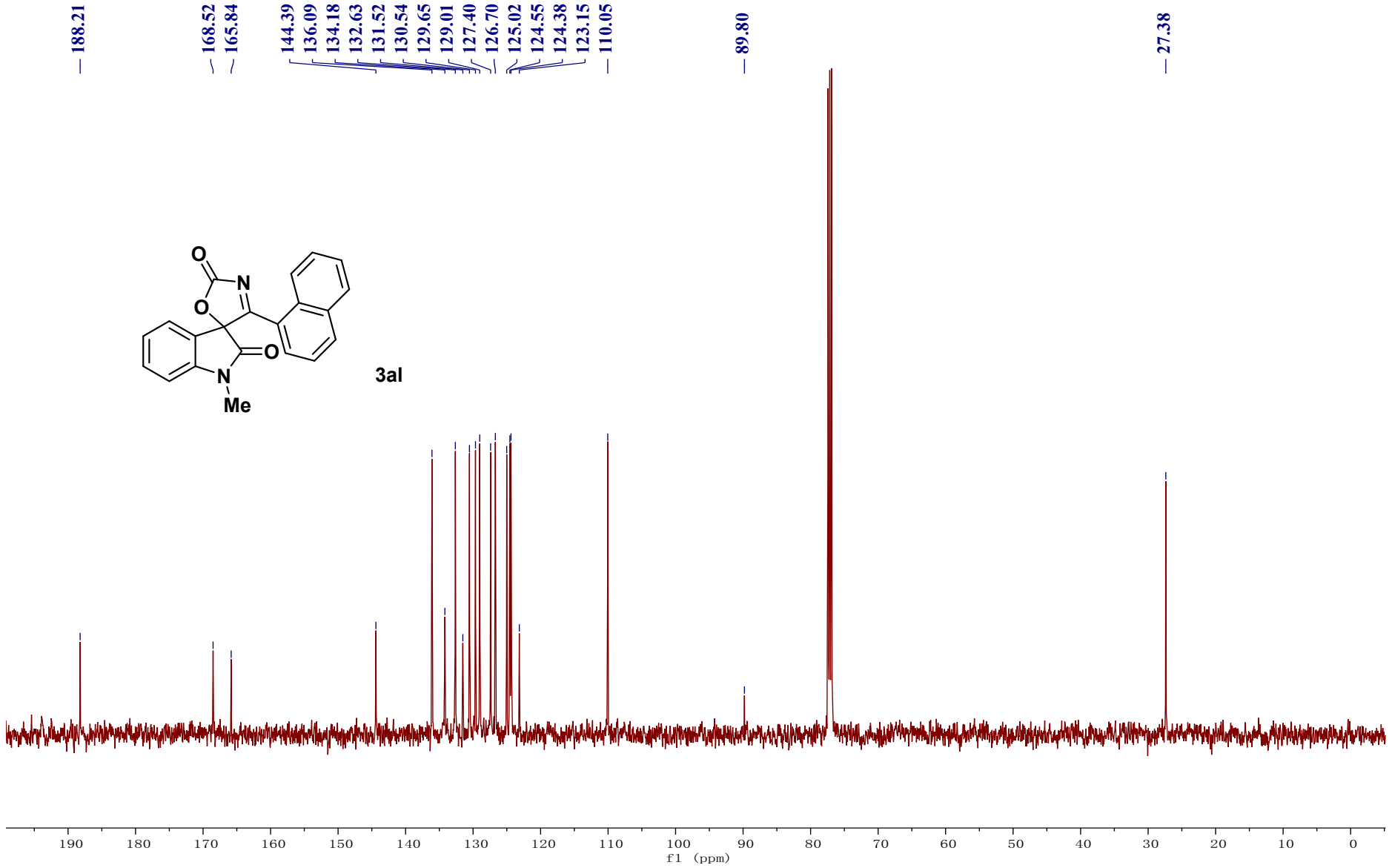
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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131.52
130.54
129.65
129.01
127.40
126.70
125.02
124.55
124.38
123.15
110.05
89.80
27.38
O
N
O
O
N
Me
3al
190
180
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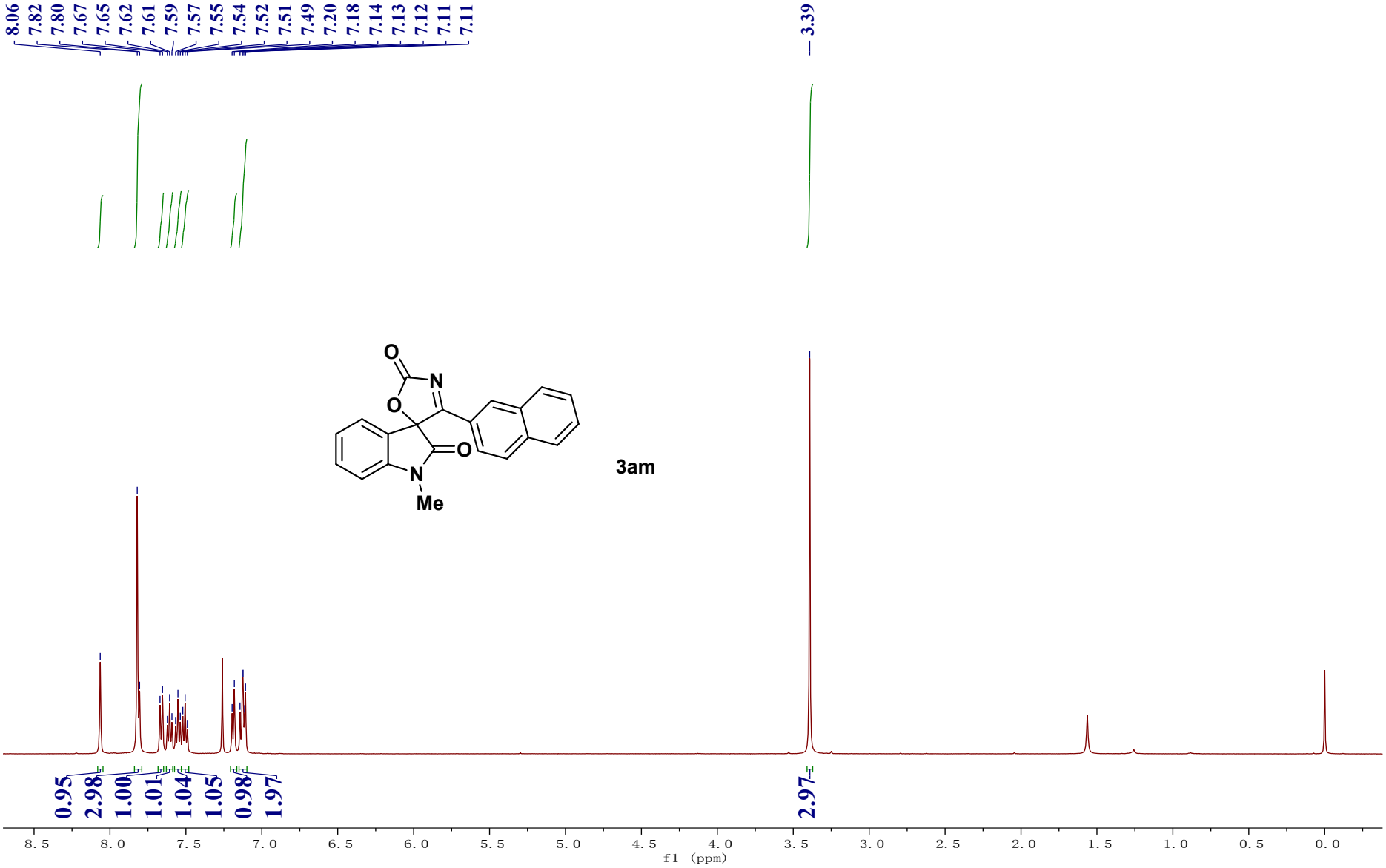
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.52
7.51
7.49
7.20
7.18
7.14
7.13
7.12
7.11
7.11
3.39
O
N
O
O
N
Me
3am
0.95
2.98
1.00
1.01
1.04
1.05
0.98
1.97
2.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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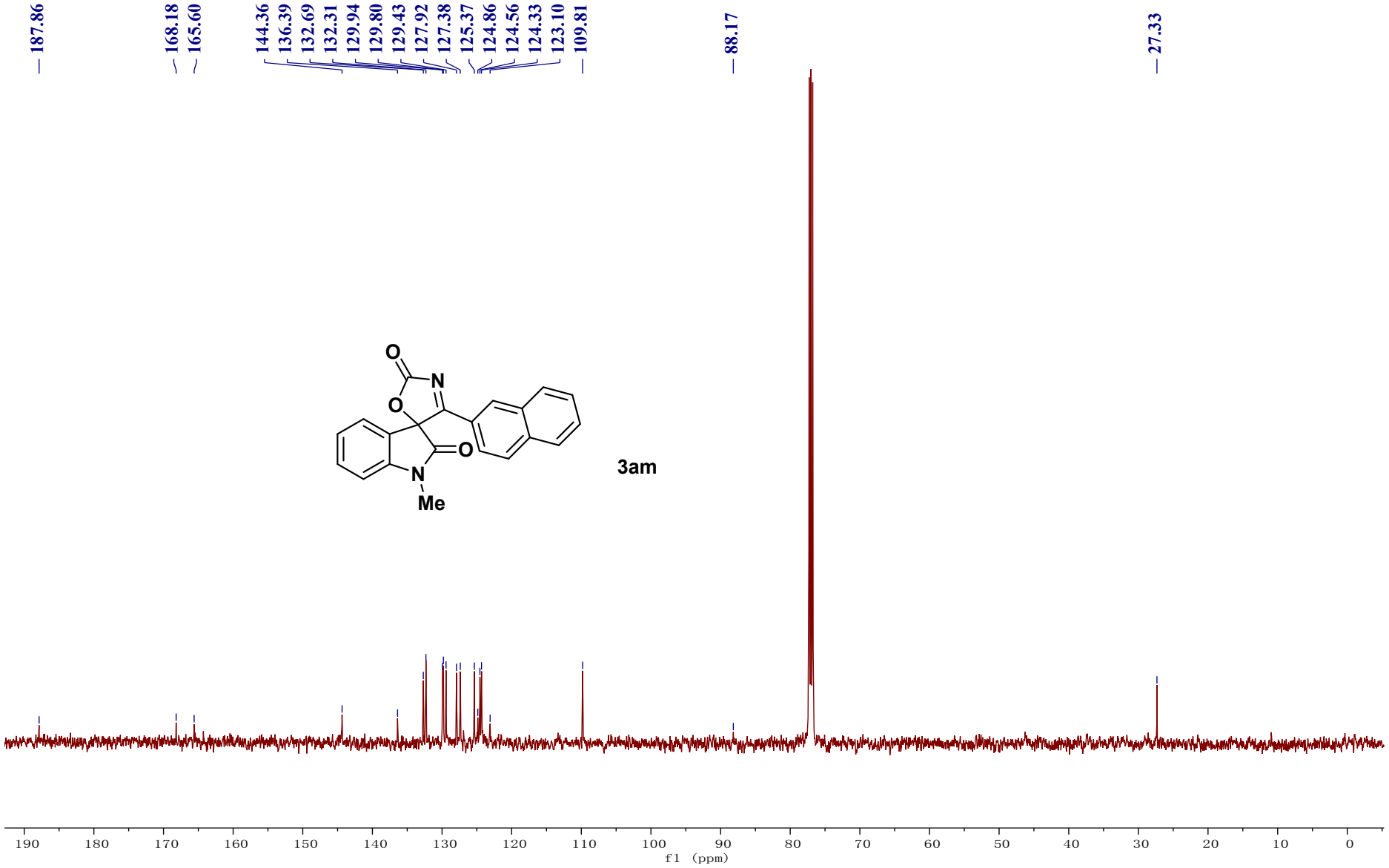
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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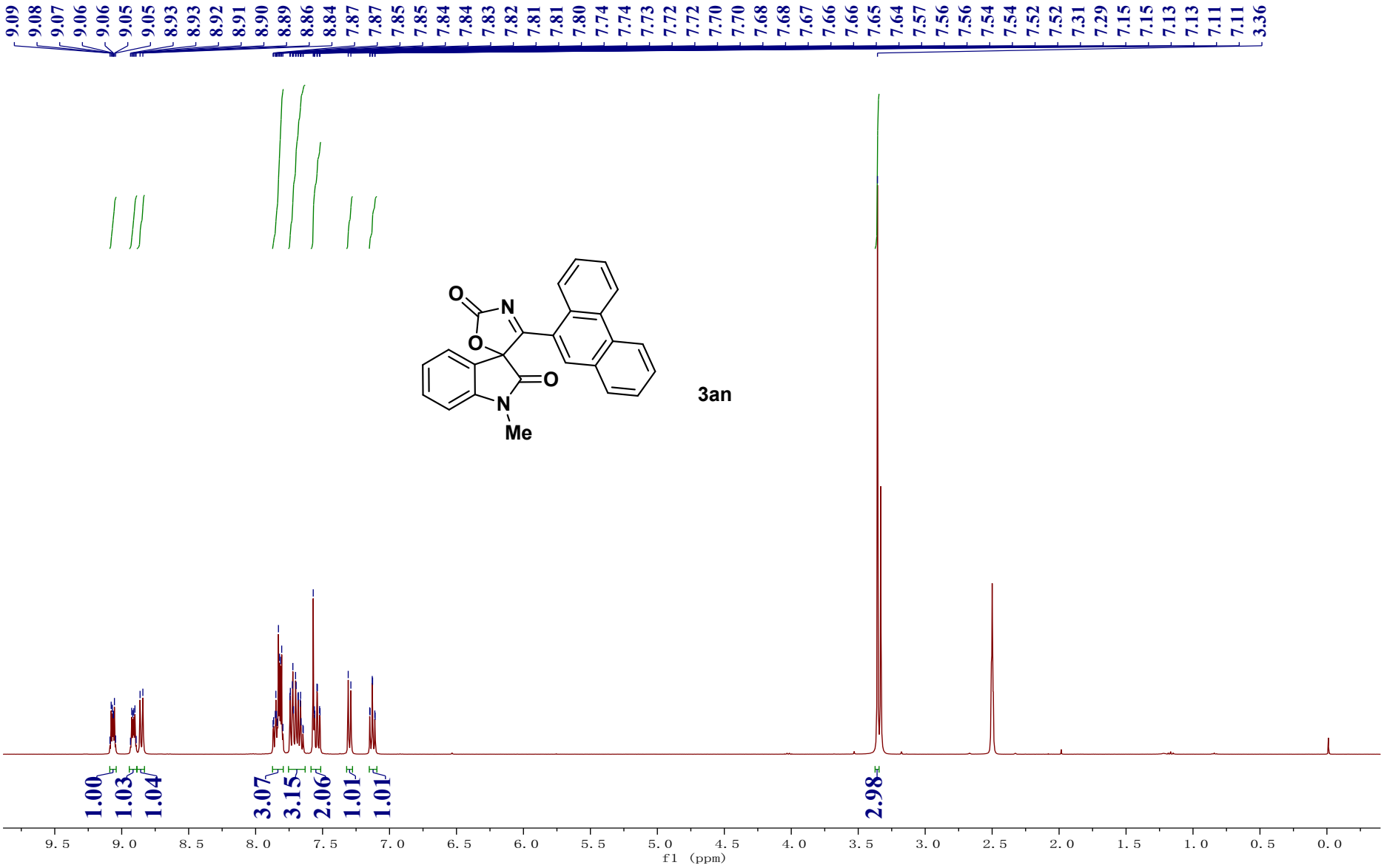
125.37
124.86
124.56
124.33
123.10
109.81
88.17
27.33
O
N
O
O
N
Me
3am
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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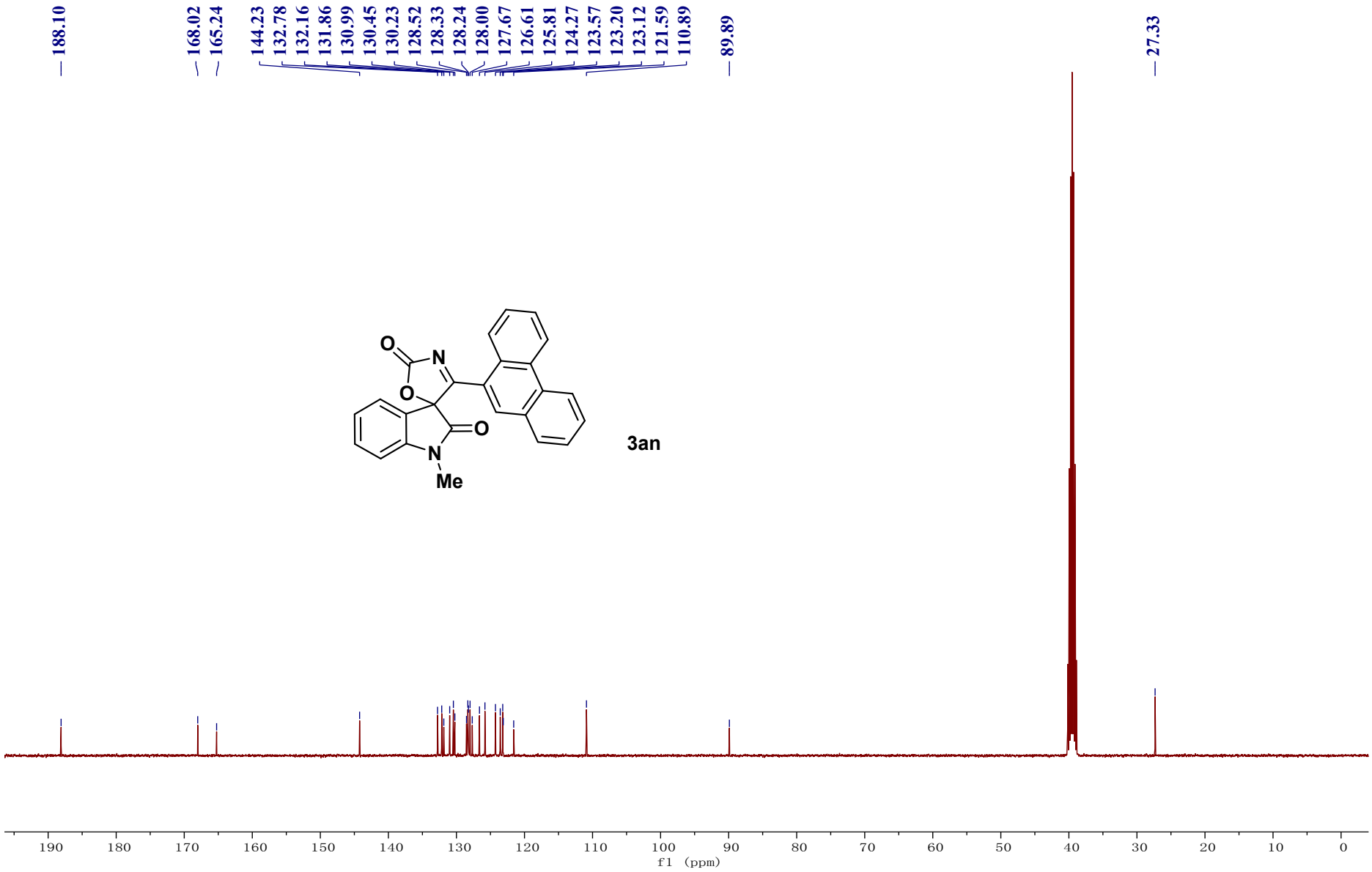
O
N
O
O
N
Me
3an
1.00 1.03 1.04 3.07 3.15 2.06 1.01 1.01 2.98
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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132.16
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130.99
130.45
130.23
128.52
128.33
128.24
128.00
127.67
126.61
125.81
124.27
123.57
123.20
123.12
121.59
110.89
89.89
27.33
O
N
O
O
N
Me
3an
190
180
170
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130
120
110
100
90
80
70
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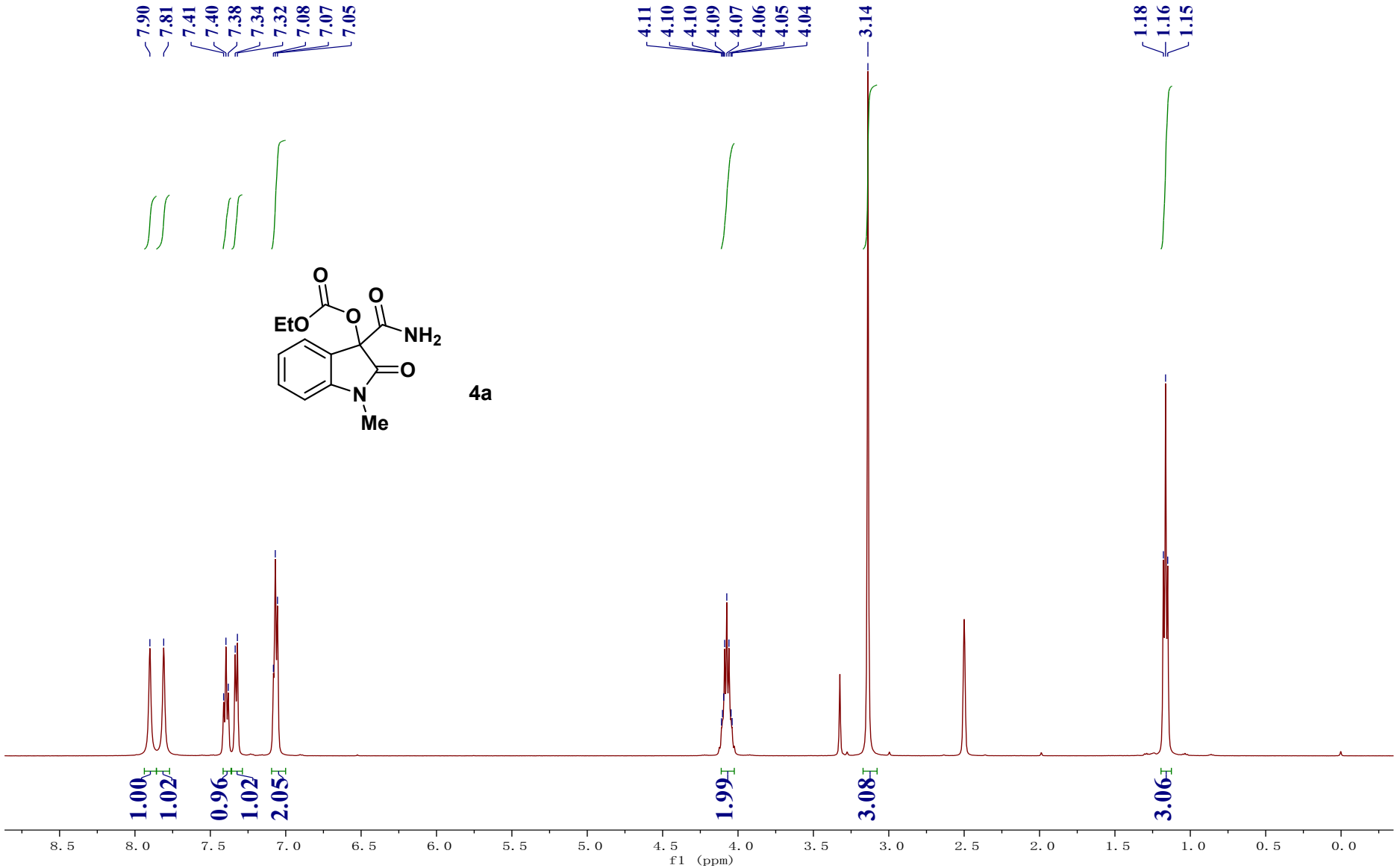
50
40
30
20
10
0
f1 (ppm)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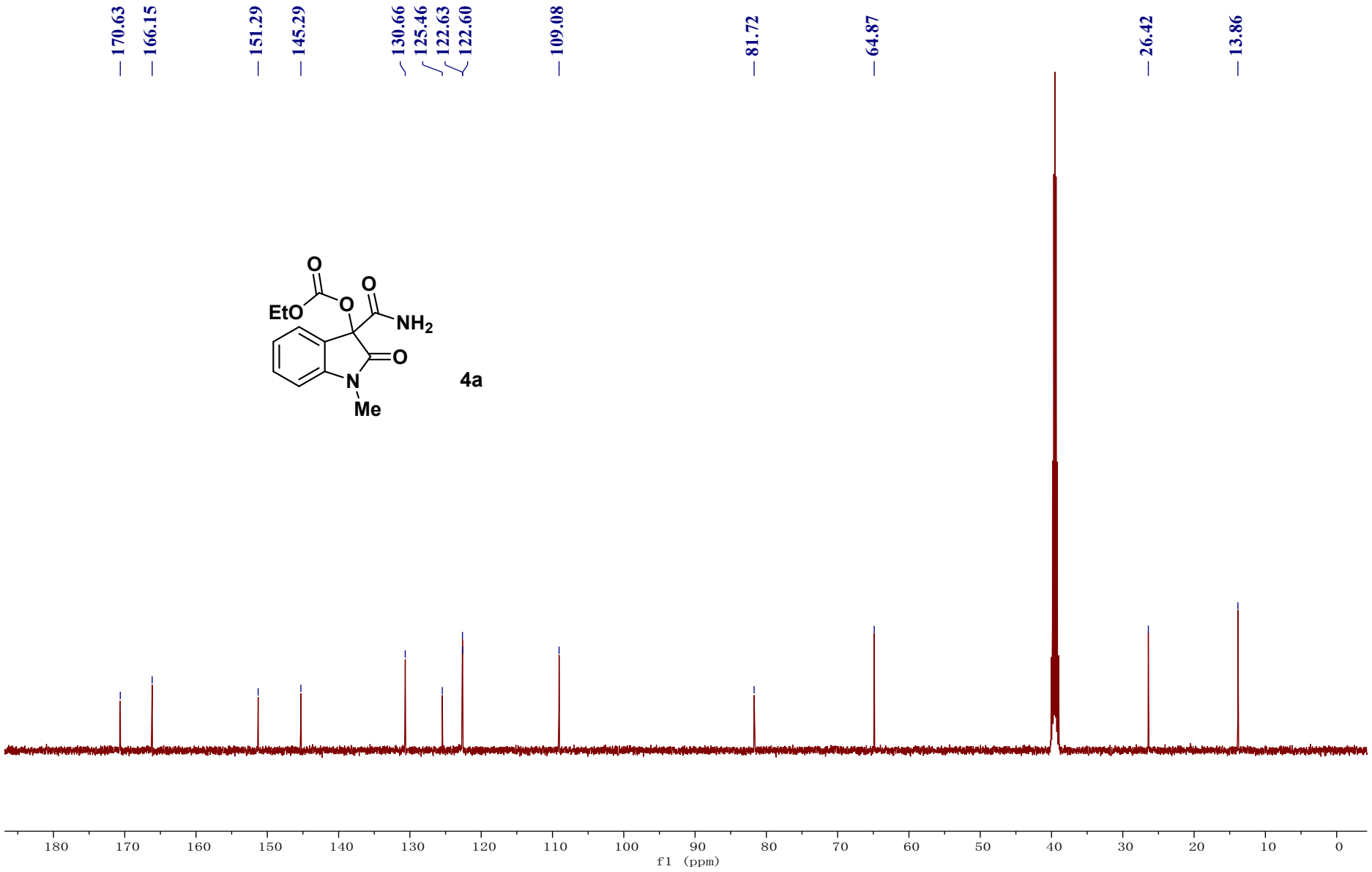
7.32
7.08
7.07
7.05
4.11
4.10
4.10
4.09
4.07
4.06
4.05
4.04
3.14
1.18
1.16
1.15
EtO
O
O
O
O
NH2
N
Me
4a
1.00
1.02
0.96
1.02
2.05
1.99
3.08
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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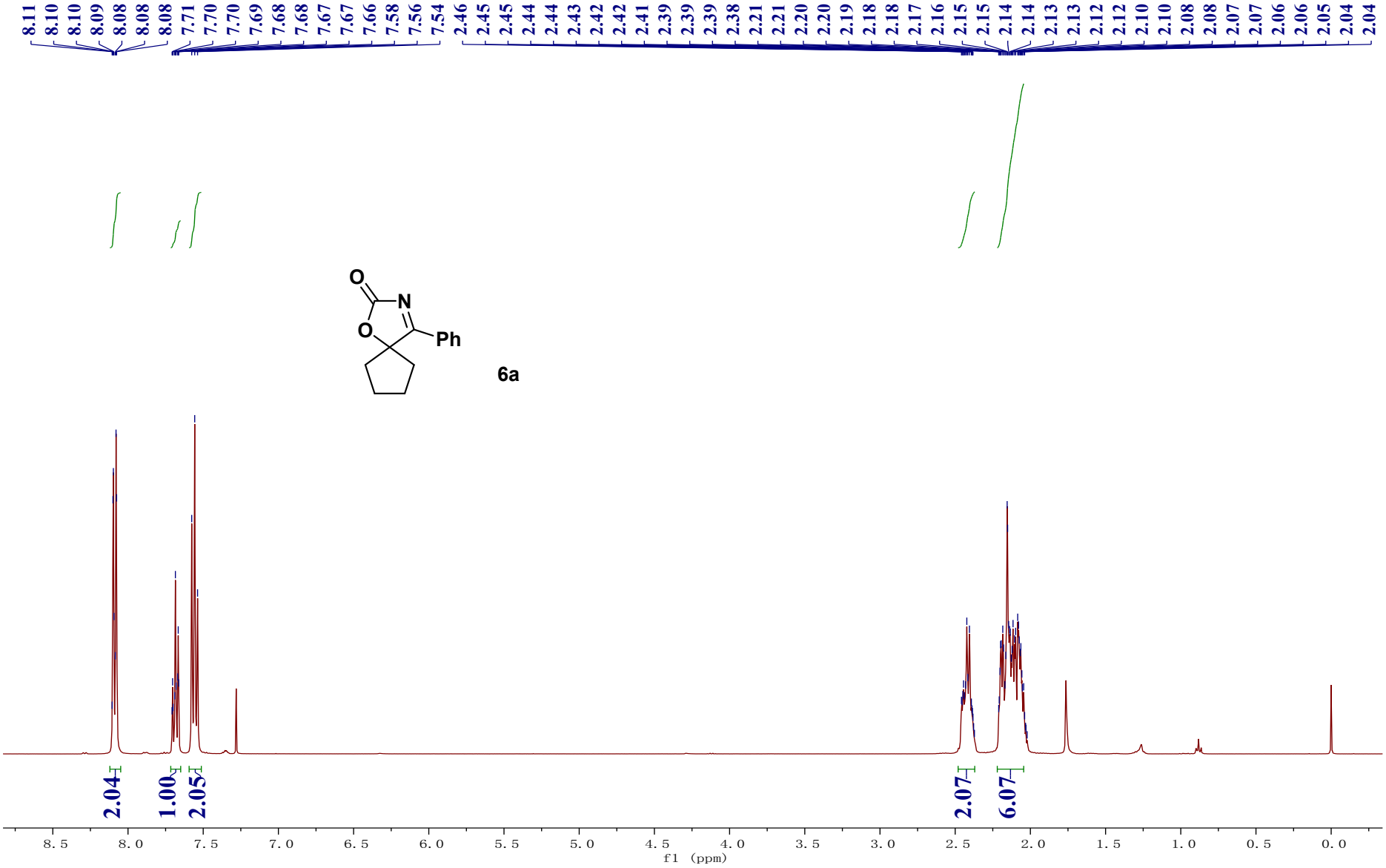
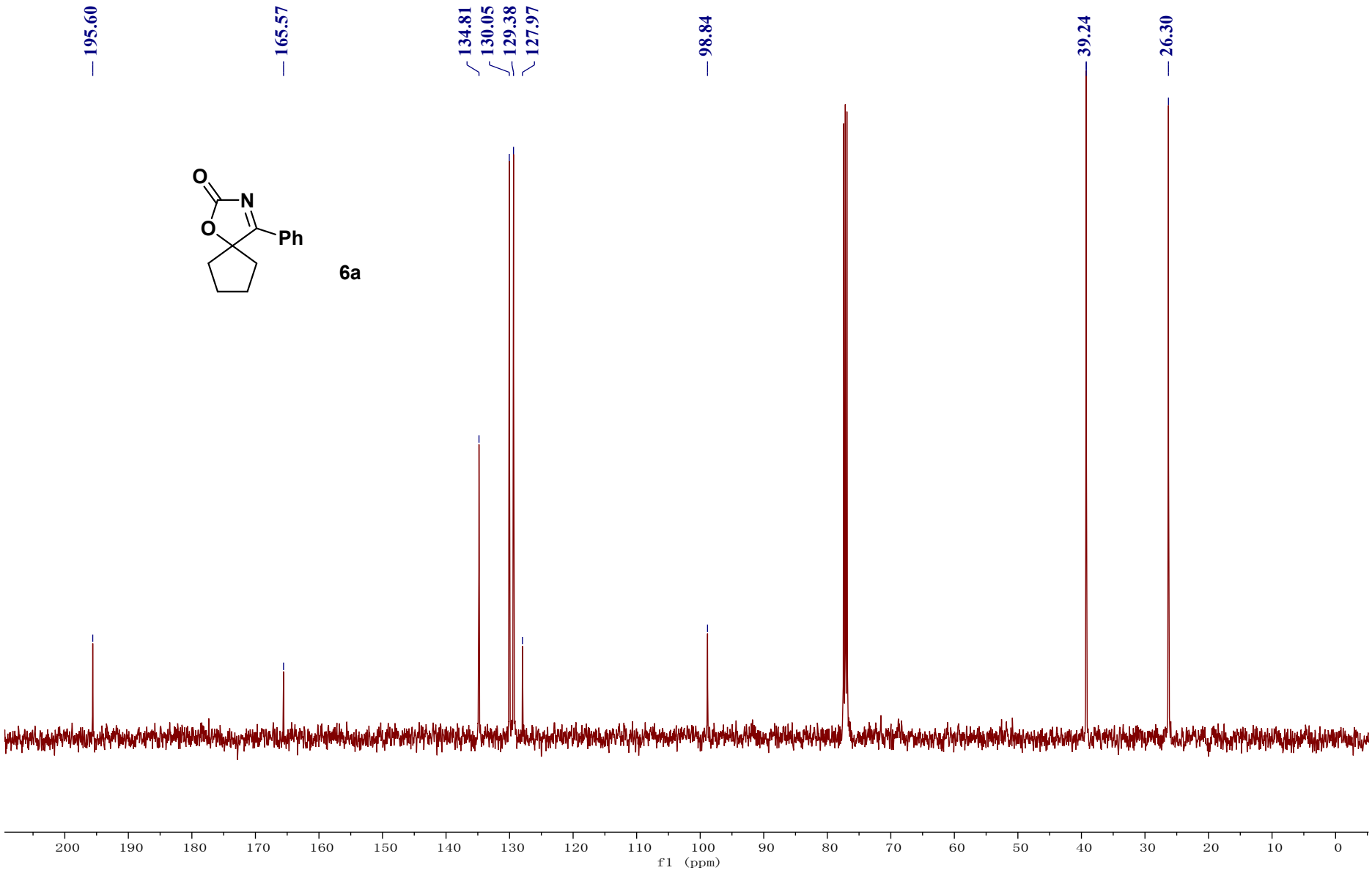
130.66
125.46
122.63
122.60
109.08
81.72
64.87
26.42
13.86
O
O
EtO
O
NH2
O
N
Me
4a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.11 8.10 8.10 8.09 8.08 8.08 8.08 7.71 7.70 7.70 7.69 7.68 7.68 7.67 7.67 7.66 7.58 7.56 7.54
2.46 2.45 2.45 2.44 2.44 2.43 2.42 2.42 2.41 2.39 2.39 2.39 2.38 2.21 2.21 2.20 2.20 2.19 2.18 2.18 2.17 2.16 2.15 2.15 2.14 2.14 2.13 2.13 2.12 2.12 2.10 2.10 2.08 2.08 2.07 2.07 2.06 2.06 2.05 2.04 2.04
O
N
O
Ph
6a
2.04
1.00
2.05
2.07
6.07
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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98.84
39.24
26.30
O
N
O
Ph
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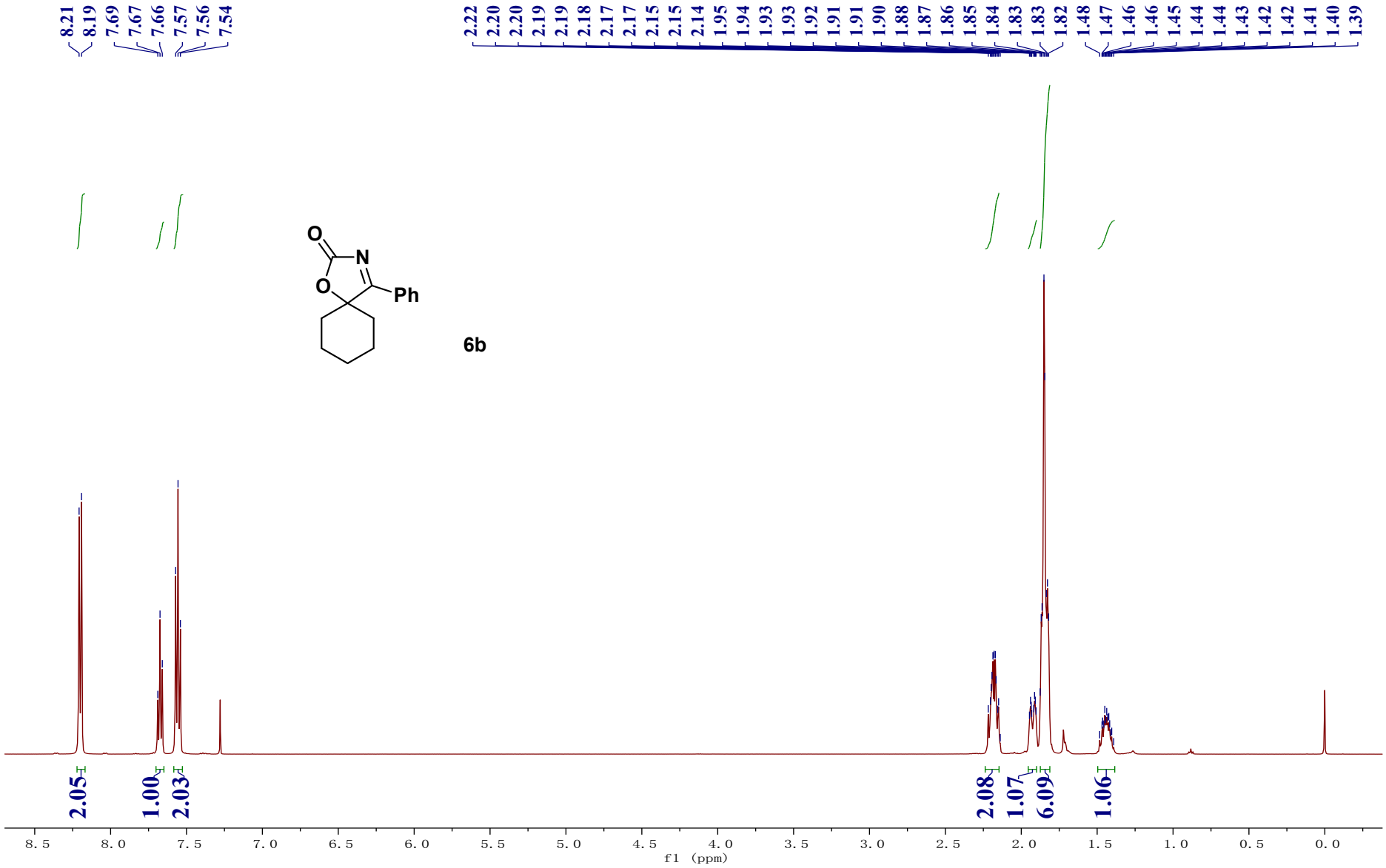
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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2.18
2.17
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2.15
2.15
2.14
1.95
1.94
1.93
1.93
1.92
1.91
1.91
1.90
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1.83
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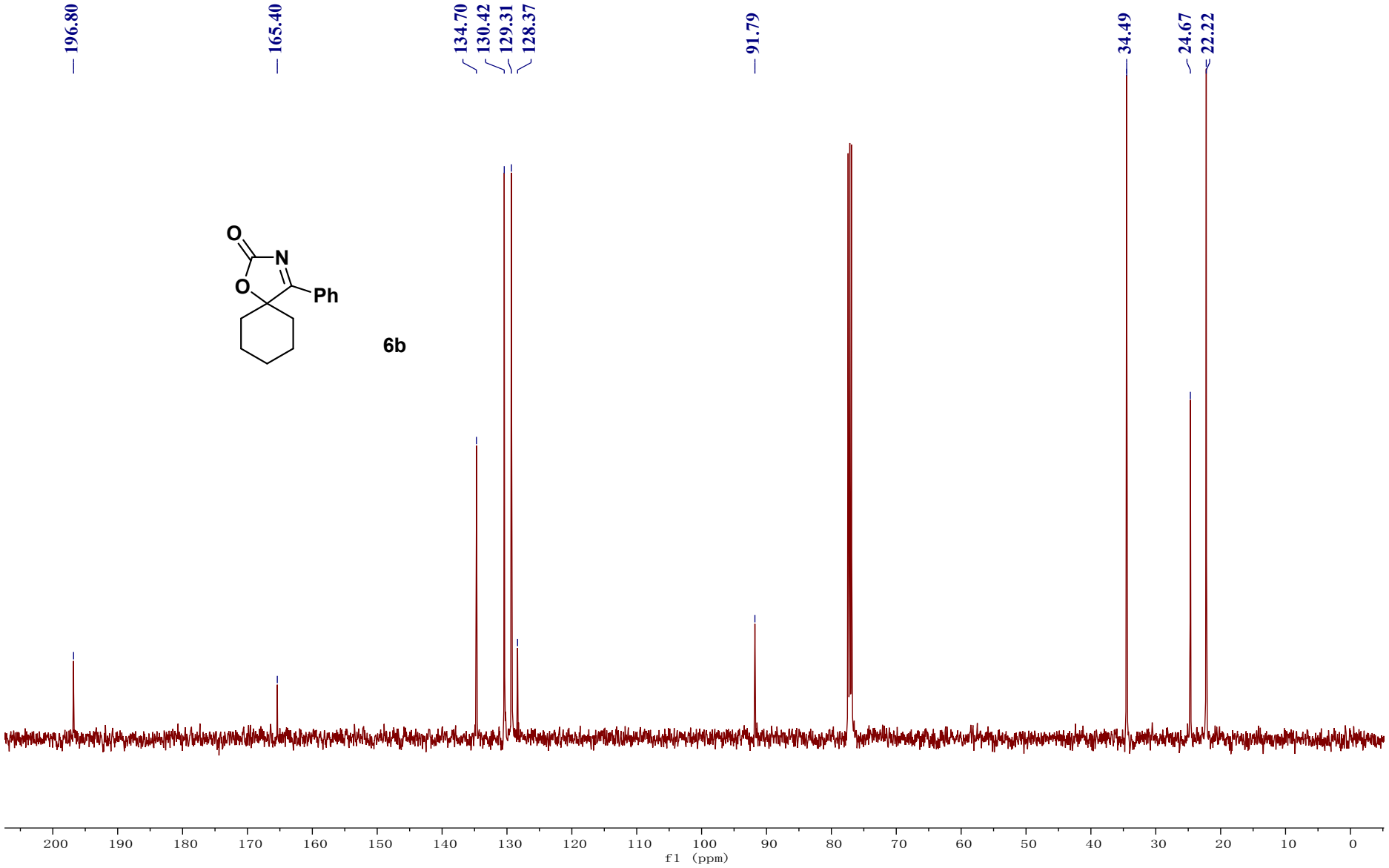
1.46
1.46
1.45
1.44
1.44
1.43
1.42
1.42
1.41
1.40
1.39
O
N
O
Ph
6b
2.05
1.00
2.03
2.08
1.07
6.09
1.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.31
128.37
91.79
34.49
24.67
22.22
O
N
O
Ph
6b
200
190
180
170
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150
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130
120
110
100
90
80
70
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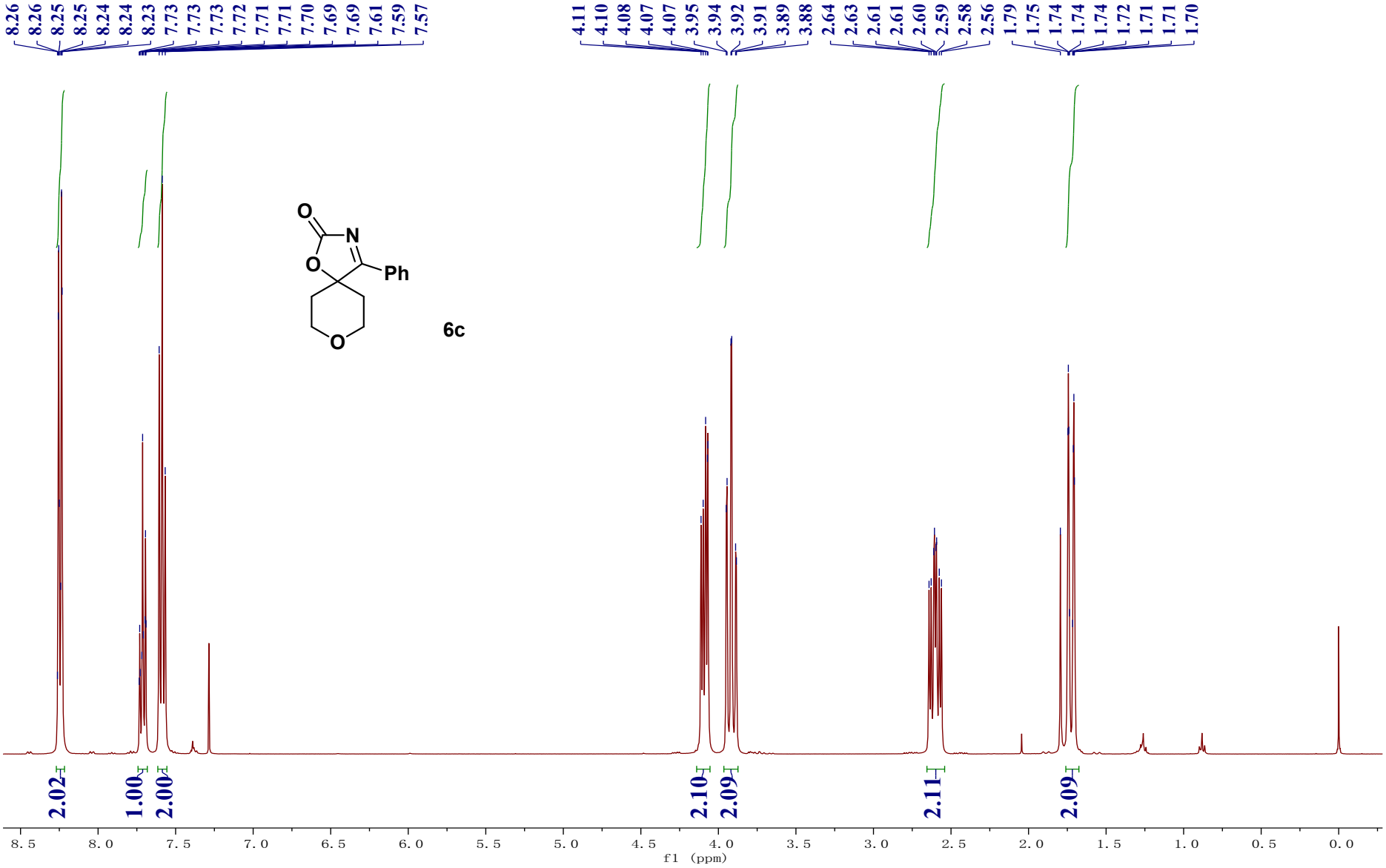
50
40
30
20
10
0
f1 (ppm)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7.73
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7.71
7.71
7.70
7.69
7.69
7.61
7.59
7.57
4.11
4.10
4.08
4.07
4.07
3.95
3.94
3.92
3.91
3.89
3.88
2.64
2.63
2.61
2.61
2.60
2.59
2.58
2.56
1.79
1.75
1.74
1.74
1.74
1.72
1.71
1.71
1.70
O
N
O
Ph
O
6c
2.02
1.00
2.00
2.10
2.09
2.11
2.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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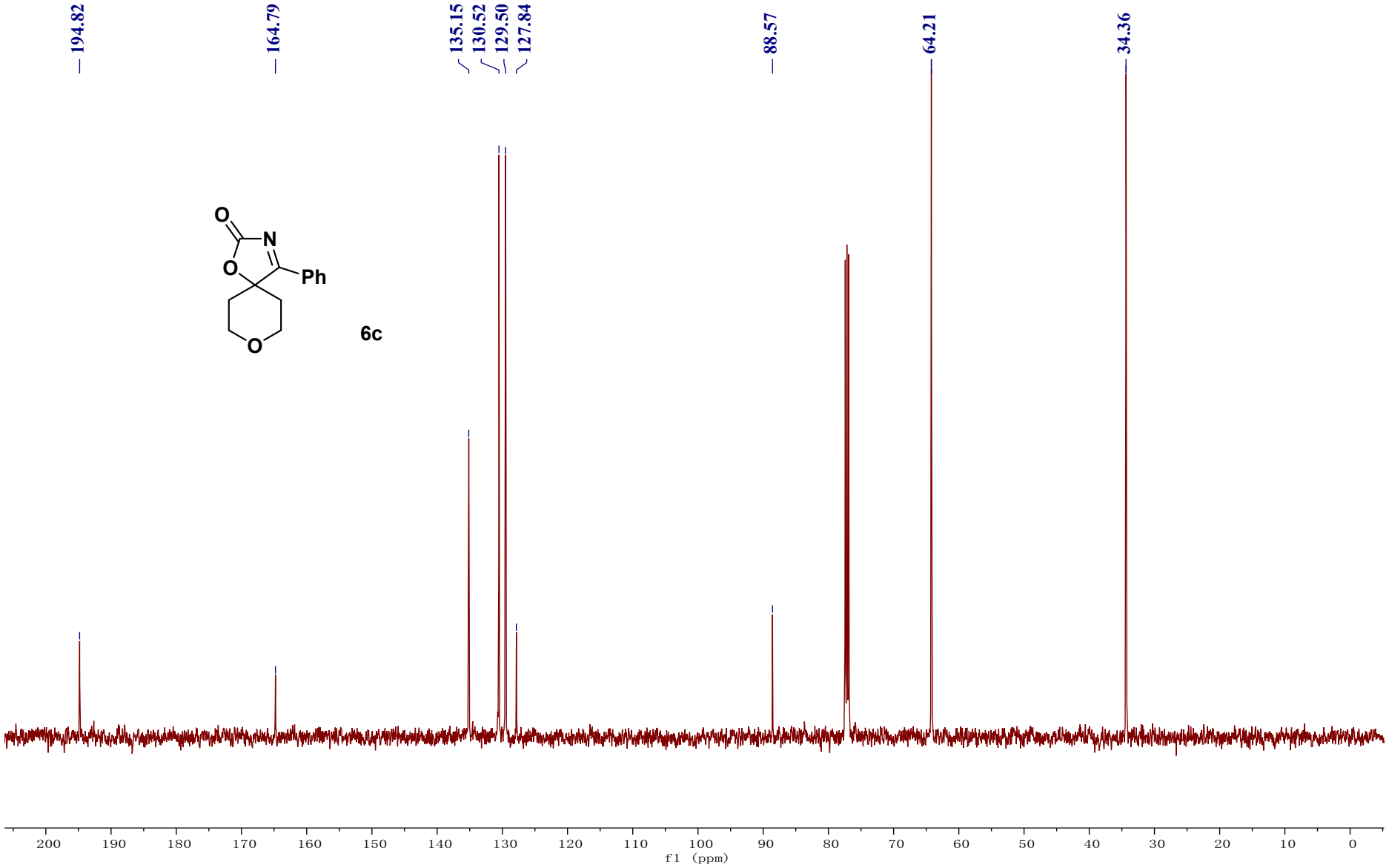
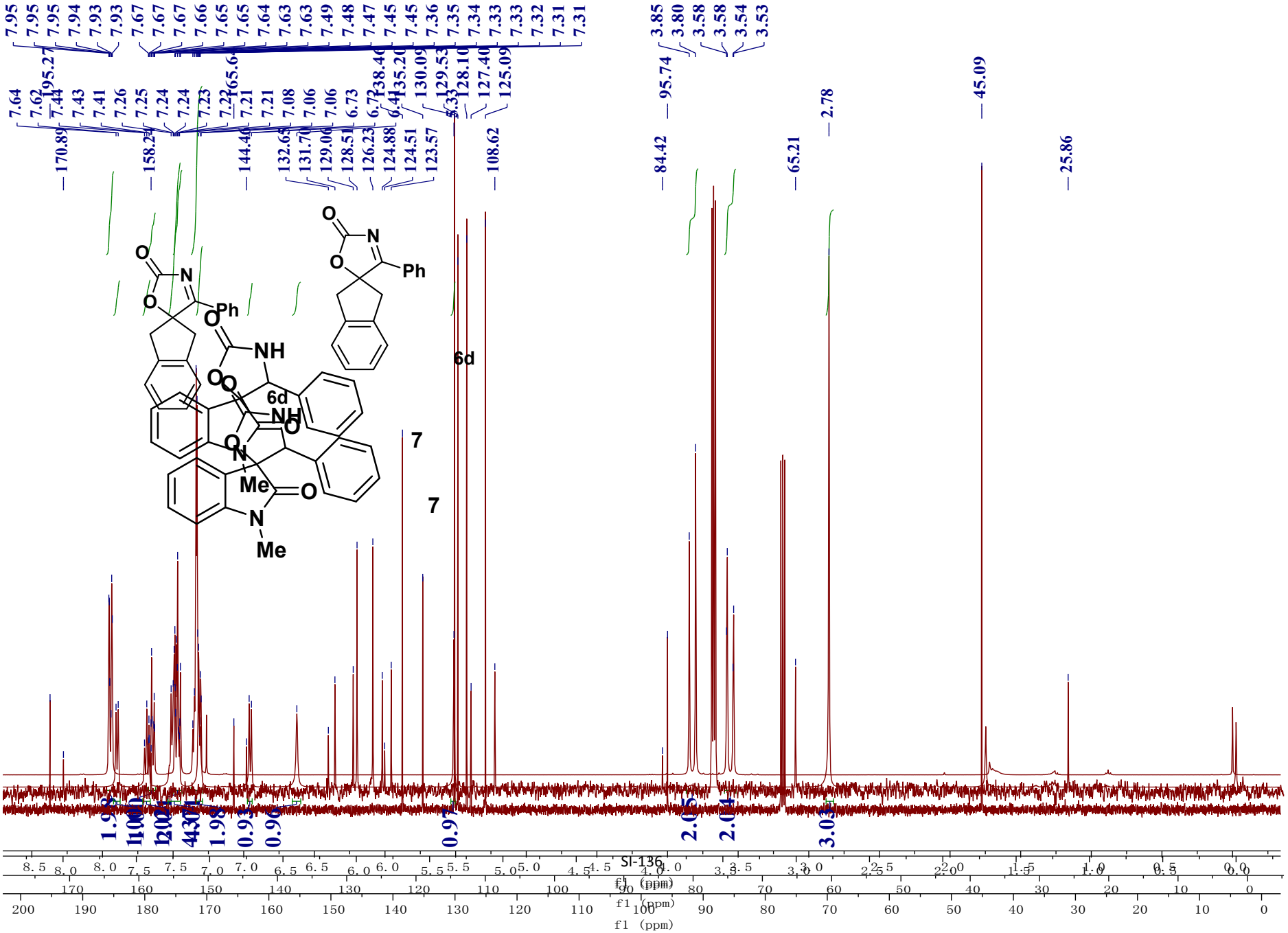
0.0
f1 (ppm)


194.82
164.79
135.15
130.52
129.50
127.84
88.57
64.21
34.36
O
N
O
Ph
O
6c
200
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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3.80
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6.72
6.41
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132.62
131.70
129.06
128.51
126.23
124.88
124.51
123.57
138.46
135.20
130.09
129.53
128.10
127.40
125.09
108.62
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95.74
65.21
2.78
45.09
25.86
6d
7
7
Ph
NH
Me
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0.93
0.96
0.97
2.05
2.04
3.03
SI-136
f1 (ppm)