

Supporting Information

Highly Efficient Iron-Catalyzed Conjugate Reduction of α,β -Unsaturated Ketones with Polymethylhydrosiloxane

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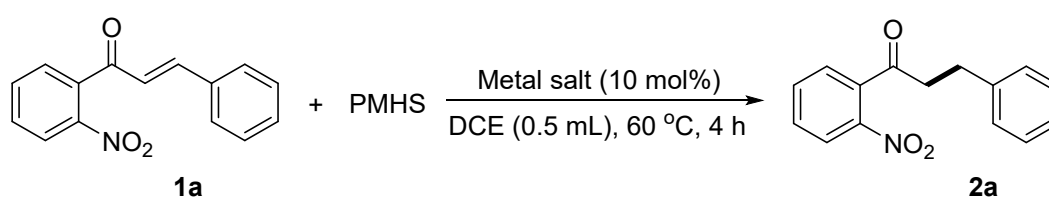
I. General Information

FeCl₃, PMHS, and (CF₃)₂CHOH were purchased from Adamas. All others reagents were purchased from commercial suppliers (Macklin, Knowles, KESHI, Aladdin and Energy Chemical) and used without further purification.

All compounds (starting materials and products) were characterized by ¹H NMR, ¹³C NMR, IR spectroscopy and high-resolution mass spectroscopy. ¹H NMR spectra were recorded on Bruker 300 or 400 MHz spectrometer and are referenced relative to residual CDCl₃ proton signals at δ 7.26 ppm. ¹⁹F NMR spectra were recorded on a Bruker 300 MHz spectrometer and are referenced to CFCl₃ (δ 0.0 ppm). Data for ¹H and ¹⁹F NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), integration, and coupling constant (Hz). ¹³C NMR spectra were recorded on a Bruker 300 MHz spectrometer and are referenced to CDCl₃ at δ 77.16 ppm. The ¹³C NMR spectra were obtained with ¹H decoupling. Data for ¹³C NMR are reported in terms of chemical shift and multiplicity where appropriate. IR spectra were obtained on a Bruker Alpha and was reported in terms of frequency of absorption (cm⁻¹). High Resolution Mass spectra were obtained from on an Agilent 6540 Q-TOF mass spectrometer, operating electrospray ionization (ESI) mode.

II. Optimization of the Reaction Conditions

Table S1. The screen of metal salts ^[a]



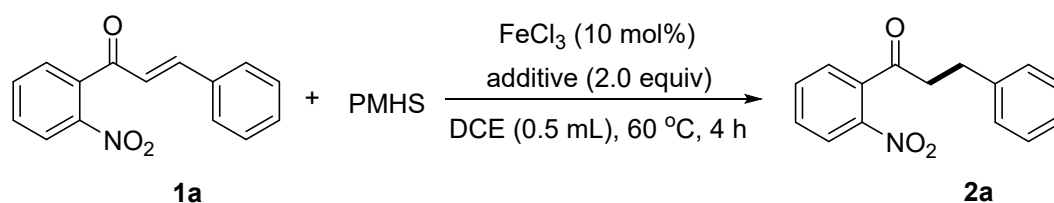
Entry	Metal salt	Yield (2a) ^[b]
1	none	0%
2	Co(acac) ₂	trace
3	VO(acac) ₂	0%
4	Ni(acac) ₂	0%
5	MoO ₂ (acac) ₂	0%
6	Mn(acac) ₃	trace

7	Fe(acac) ₃	0%
8	Cu(OAc) ₂	0%
9	Co(OAc) ₂	0%
10	Fe(OAc) ₂	0%
11	Zn(OAc) ₂	0%
12	CuPF ₆ ·4CH ₃ CN	14%
13	MnBr ₂ ·4H ₂ O	0%
14	Sm(OTf) ₃	16%
15	Ni(OTf) ₂	20%
16	Sc(OTf) ₃	25%
17	Cu(OTf) ₂	59%
18	FeCl ₃	84%

[a] Conditions: **1a** (50.7 mg, 0.20 mmol, 1.0 equiv), metal salt (0.020 mmol, 10 mol%), PMHS (100 μL, 2.2 equiv) in DCE (0.5 mL) at 60 °C for 4 h.

[b] Isolated yield.

Table S2. The screen of additive ^[a]



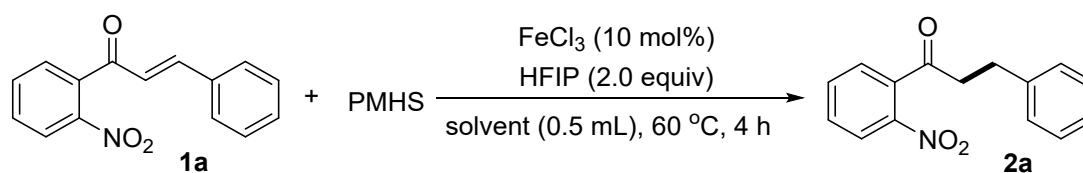
Entry	additive	Catalyst	Yield (2a) ^[b]
1	NH ₄ F	FeCl ₃	0%
2	CsF	FeCl ₃	0%
3	Cs ₂ CO ₃	FeCl ₃	0%
4	LiF	FeCl ₃	42%
5	NaF	FeCl ₃	63%
6	KF	FeCl ₃	0%
7	K ₃ PO ₄	FeCl ₃	0%

8	DABCO	FeCl ₃	0%
9	NH ₄ Cl	FeCl ₃	0%
10	NH ₄ OAc	FeCl ₃	0%
11	NaOAc	FeCl ₃	0%
12	TMG	FeCl ₃	< 5%
13	DBU	FeCl ₃	0%
14	DIPEA	FeCl ₃	0%
15	morpholine	FeCl ₃	trace
16	NaHCO ₃	FeCl ₃	0%
17	(CF ₃) ₂ CHOH	FeCl ₃	93%
18	(CF ₃) ₂ CHOH	-	0%

[a] Conditions: **1a** (50.7 mg, 0.20 mmol, 1.0 equiv), FeCl₃ (3.2 mg, 0.020 mmol, 10 mol%), additive (2.0 equiv), PMHS (100 μL, 2.2 equiv) in DCE (0.5 mL) at 60 °C for 4 h.

[b] Isolated yield.

Table S3. The screen of solvent [a]



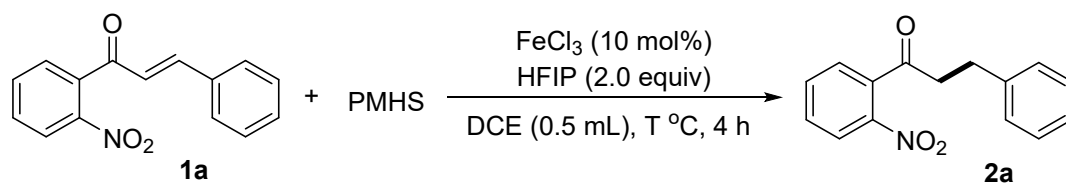
Entry	solvent	Yield (2a) ^[b]
1	DCE	93%
2	THF	0%
3	toluene	88%
4	CH ₃ CN	0%
5	CHCl ₃	47%
6	1,4-dioxane	0%
7	DMAc	0%
8	NMP	trace

9	EtOH	0%
10	PhOMe	86%
11	MTBE	0%
12	Ph ₂ O	84%
13	DMSO	0%

[a] Conditions: **1a** (50.7 mg, 0.20 mmol, 1.0 equiv), FeCl₃ (3.2 mg, 0.020 mmol, 10 mol%), (CF₃)₂CHOH (60 μL, 2.0 equiv), PMHS (100 μL, 2.2 equiv) in solvent (0.5 mL) at 60 °C for 4 h.

[b] Isolated yield.

Table S4. The screen of temperature [a]

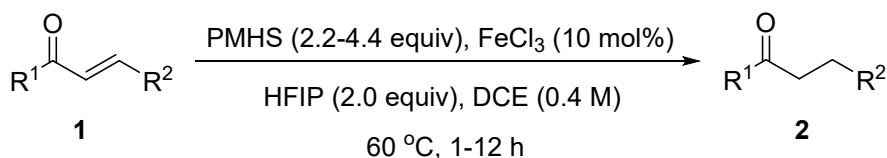


Entry	T	Yield (2a) ^[b]
1	25 °C	86%
2	35 °C	88%
3	60 °C	93%
4	90 °C	90%

[a] Conditions: **1a** (50.7 mg, 0.20 mmol, 1.0 equiv), FeCl₃ (3.2 mg, 0.020 mmol, 10 mol%), (CF₃)₂CHOH (60 μL, 2.0 equiv), PMHS (100 μL, 2.2 equiv) in DCE (0.5 mL) at T °C for 4 h.

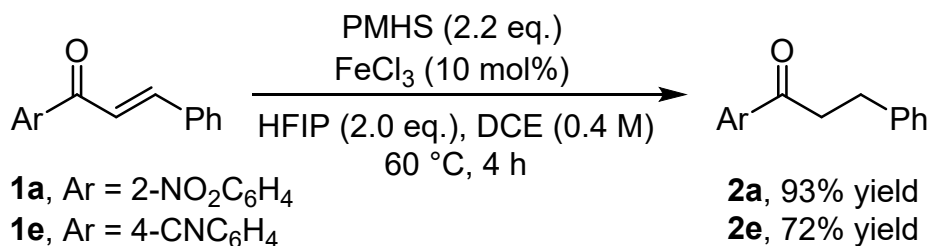
[b] Isolated yield.

III. General Procedure for Fe-Catalyzed Conjugate Reduction of Chalcone Derivatives

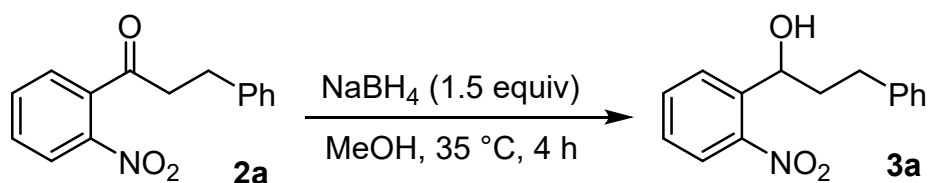


General procedure: Substituted enones **1** (0.20 mmol, 1.0 equiv) and FeCl₃ (3.2 mg, 0.020 mmol, 10 mol%) were dissolved in dry ClCH₂CH₂Cl (0.5 mL, 0.4 M solution). Then, (CF₃)₂CHOH (60.0 μL, 2.0 equiv) and PMHS (100.0-200.0 μL, 2.2-4.4 equiv) were added. The mixture was stirred in an oil bath at 60 °C for 1-12 h (the mixture was stirred at 400 rpm). After the reaction was complete, the mixture was extracted with EtOAc (5 mL × 3). The organic layer was concentrated to give the crude product. The mixture was purified by flash column chromatography (petroleum ether/EtOAc) to get the desired product **2**.

IV. Procedure for the scale-up reaction and the transformation of product.

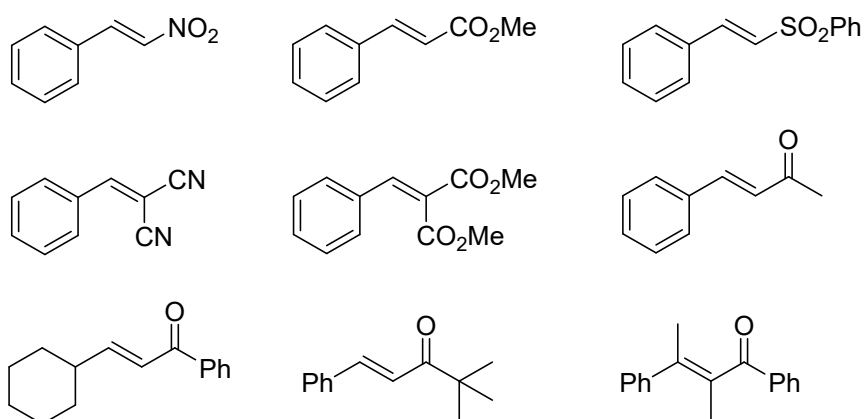


General procedure: An oven-dried 100 mL round-bottom flask was charged with **1a** (1.52 g, 6.0 mmol, 1.0 equiv), FeCl₃ (97.3 mg, 0.60 mmol, 10 mol%), (CF₃)₂CHOH (1.8 mL, 2.0 equiv), PMHS (3.0 mL, 2.2 equiv) and DCE (15 mL). The mixture was stirred in an oil bath at 60 °C for 4 h (the mixture was stirred at 400 rpm). After the reaction was complete, the mixture was extracted with EtOAc (20 mL × 3). The organic layer was concentrated to give the crude product. The product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound **2a** as a colorless liquid (1.42 g, 93% yield). The same procedure for substrate **1e**.

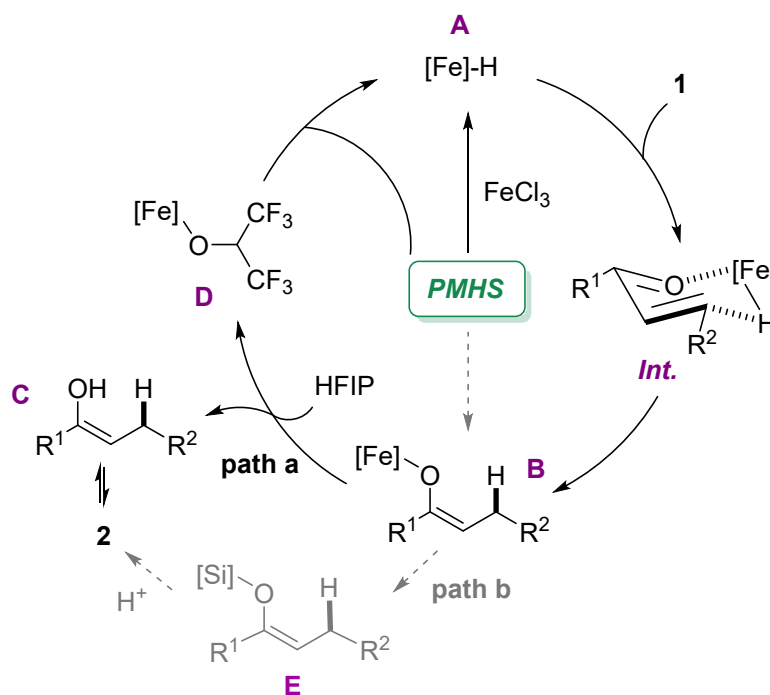


General procedure: Compound **2a** (153.2 mg, 0.60 mmol) was dissolved in MeOH (3 mL) and NaBH₄ (34.0 mg, 0.90 mmol, 1.5 equiv) was added. The mixture was stirred at 35 °C for 4 h, after the reaction was complete, the mixture was extracted with EtOAc. The organic layer was concentrated to give the crude product. The product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the derivative compound 138.0 mg of **3a** as a colorless liquid (90% yield).

V. Unsuccessful substrate scope



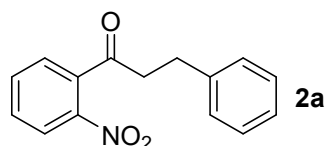
VI. Proposed mechanism



First, the precatalyst FeCl₃ could be reduced by PMHS to form iron hydride species **A**.

Then the α,β -unsaturated ketone **1** could coordinate with species **A** followed by the insertion into a Fe–H bond to generate species **B** via a possible six-membered ring intermediate. Later, HFIP induced the proto-demetalation to generate species **C** followed by tautomerization to deliver the desired product **2** (path a). Besides, without the additive, the iron enolate species **B** would react with PMHS via σ -bond metathesis to afford a silyl enolate **E**. Subsequently, the target product **2** was obtained by the hydrolysis of **E** in the quenching step (path b). Such an explanation is consistent with the experimental finding that a higher yield was observed when the HFIP was added which could accelerate the regeneration of reactive species **A**.

VII. The analytical and spectral characterization data of products.



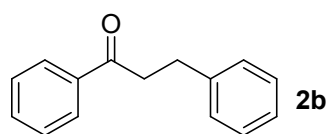
1-(2-Nitrophenyl)-3-phenylpropan-1-one. From **1a** (50.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μ L, 2.0 equiv), PMHS (100.0 μ L, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 4 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (47.2 mg, 93% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.18 – 8.11 (m, 1H), 7.77 – 7.68 (m, 1H), 7.62 – 7.57 (m, 1H), 7.32 – 7.26 (m, 6H), 3.17 – 3.13 (m, 4H).

¹³C NMR (75 MHz, CDCl₃) δ 201.8, 145.6, 140.5, 138.0, 134.4, 130.6, 128.6, 128.6, 128.4, 128.4, 127.3, 126.3, 124.4, 44.7, 30.1.

HRMS (ESI) m/z : [M + H]⁺ Calculated for [C₁₅H₁₄NO₃⁺]: 256.0968, found 256.0971.

IR (neat, cm⁻¹) 3028, 2866, 1703, 1604, 1573, 1523, 1453, 1344, 1205, 853, 788, 741, 697.



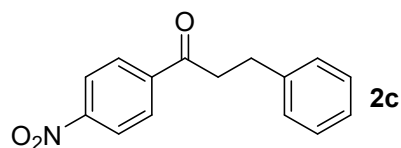
1,3-Diphenylpropan-1-one. From **1b** (41.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 4 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (29.2 mg, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.88 – 7.87 (m, 1H), 7.86 – 7.85 (m, 1H), 7.47 – 7.43 (m, 1H), 7.37 – 7.33 (m, 2H), 7.21 – 7.19 (m, 2H), 7.17 – 7.15 (m, 2H), 7.13 – 7.11 (m, 1H), 3.20 (t, *J* = 8.0 Hz, 2H), 2.98 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 199.2, 141.4, 136.9, 133.2, 128.7, 128.7, 128.7, 128.7, 128.5, 128.5, 128.1, 128.1, 126.2, 40.5, 30.2.

HRMS (ESI) *m/z*: [M + H]⁺ Calculated for [C₁₅H₁₅O⁺]: 211.1117, found 211.1120.

IR (neat, cm⁻¹) 3061, 3026, 2924, 1680, 1595, 1580, 1494, 1448, 1364, 1290, 1205, 972, 908, 741, 699.



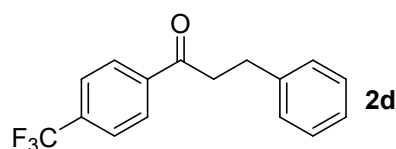
1-(4-Nitrophenyl)-3-phenylpropan-1-one. From **1c** (50.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (37.1 mg, 72% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.34 – 8.31 (m, 2H), 8.14 – 8.11 (m, 2H), 7.37 – 7.32 (m, 2H), 7.30 – 7.23 (m, 3H), 3.39 (t, *J* = 7.5 Hz, 2H), 3.13 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.7, 150.3, 141.2, 140.6, 129.1, 129.1, 128.7, 128.7, 128.4, 128.4, 126.4, 123.9, 123.9, 41.0, 29.9.

HRMS (ESI) *m/z*: [M + H]⁺ Calculated for [C₁₅H₁₄NO₃⁺]: 256.0968, found 256.0970.

IR (neat, cm⁻¹) 3028, 2923, 1692, 1603, 1524, 1496, 1453, 1344, 1198, 907, 853, 727.



1-(4-(Trifluoromethyl)phenyl)-3-phenylpropan-1-one. From **1d** (55.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (40.3 mg, 72% yield).

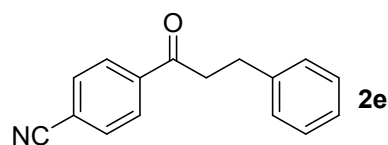
¹H NMR (400 MHz, CDCl₃) δ 7.96 – 7.94 (m, 2H), 7.63 – 7.61 (m, 2H), 7.23 – 7.20 (m, 2H), 7.17 – 7.14 (m, 2H), 7.12 – 7.07 (m, 1H), 3.23 (t, *J* = 8.0 Hz, 2H), 2.99 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 197.1, 139.8, 138.5, 133.3 (q, *J* = 32.3 Hz), 127.6, 127.6, 127.4, 127.4, 127.3, 127.3, 125.3, 124.6 (q, *J* = 4.0 Hz), 122.6 (q, *J* = 273.7 Hz), 39.7, 28.9.

¹⁹F NMR (282 MHz, CDCl₃) δ -63.1.

HRMS (ESI) *m/z*: [M + H]⁺ Calculated for [C₁₆H₁₄F₃O⁺]: 279.0991, found 279.0994.

IR (neat, cm⁻¹) 3029, 2930, 1691, 1604, 1584, 1496, 1454, 1410, 1323, 1166, 1125, 1065, 1015, 846, 746, 699.



4-(3-Phenylpropanoyl) benzonitrile. From **1e** (46.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 3 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (36.1 mg, 76% yield).

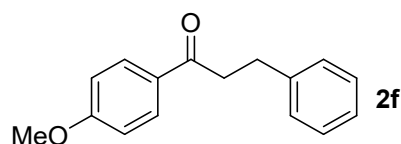
¹H NMR (400 MHz, CDCl₃) δ 7.93 – 7.92 (m, 1H), 7.91 – 7.90 (m, 1H), 7.65 – 7.64

(m, 1H), 7.63 – 7.62 (m, 1H), 7.22 – 7.18 (m, 2H), 7.15 – 7.13 (m, 2H), 7.12 – 7.09 (m, 1H), 3.21 (t, $J = 8.0$ Hz, 2H), 2.97 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.9, 140.7, 139.7, 132.6, 132.6, 128.7, 128.7, 128.5, 128.5, 128.4, 128.4, 126.4, 118.0, 116.3, 40.8, 29.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{16}\text{H}_{13}\text{NONa}^+]$: 258.0889, found 258.0891.

IR (neat, cm^{-1}) 3062, 3028, 2927, 2230, 1688, 1605, 1453, 1403, 1205, 800, 732, 698.



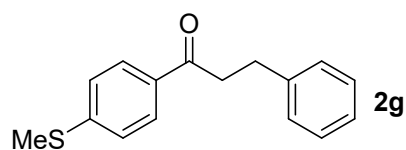
1-(4-Methoxyphenyl)-3-phenylpropan-1-one. From **1f** (47.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (200.0 μL , 4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 8 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (32.2 mg, 67% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.82 (m, 2H), 7.22 – 7.18 (m, 2H), 7.17 – 7.14 (m, 2H), 7.12 – 7.08 (m, 1H), 3.75 (s, 3H), 3.15 (t, $J = 8.0$ Hz, 2H), 2.96 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.9, 163.5, 141.5, 130.3, 130.3, 130.0, 128.5, 128.5, 128.5, 128.5, 126.1, 113.7, 113.7, 55.5, 40.2, 30.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{16}\text{H}_{16}\text{O}_2\text{Na}^+]$: 263.1043, found 263.1048.

IR (neat, cm^{-1}) 3027, 2962, 2933, 1668, 1600, 1574, 1509, 1453, 1419, 1258, 1026, 905, 839, 727, 697.



1-(4-(Methylthio)phenyl)-3-phenylpropan-1-one. From **1g** (50.9 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3

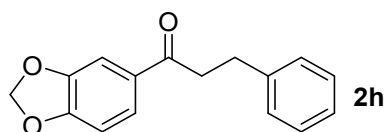
(3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (200.0 μL, 4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 12 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/20) to afford the title compound as a colorless liquid (33.9 mg, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.78 – 7.77 (m, 1H), 7.76 – 7.75 (m, 1H), 7.22 – 7.18 (m, 2H), 7.16 – 7.15 (m, 2H), 7.15 – 7.13 (m, 2H), 7.12 – 7.09 (m, 1H), 3.15 (t, *J* = 8.0 Hz, 2H), 2.96 (t, *J* = 8.0 Hz, 2H), 2.40 (s, 3H).

¹³C NMR (75 MHz, CDCl₃) δ 198.3, 145.9, 141.3, 133.1, 128.6, 128.6, 128.5, 128.5, 128.5, 128.5, 126.2, 125.0, 125.0, 40.3, 30.2, 14.8.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₆OSNa⁺]: 279.0814, found 279.0816.

IR (neat, cm⁻¹) 3063, 3027, 2922, 2858, 1661, 1588, 1554, 1490, 1451, 1399, 1209, 1076, 968, 908, 819, 773, 747, 697.



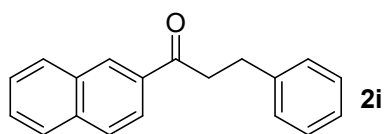
1-(benzo[*d*][1,3]dioxol-6-yl)-3-phenylpropan-1-one. From **1h** (50.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (200.0 μL, 4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 10 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (33.2 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.47 (m, 1H), 7.37 – 7.36 (m, 1H), 7.24 – 7.20 (m, 2H), 7.18 – 7.16 (m, 2H), 7.15 – 7.11 (m, 1H), 6.77 – 6.75 (m, 1H), 5.96 (s, 2H), 3.15 (t, *J* = 8.0 Hz, 2H), 2.97 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.3, 151.8, 148.2, 141.4, 131.8, 128.6, 128.6, 128.5, 128.5, 126.2, 124.3, 107.9, 107.9, 101.9, 40.2, 30.4.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₄O₃Na⁺]: 277.0835, found 277.0840.

IR (neat, cm^{-1}) 3026, 2921, 2857, 1675, 1604, 1503, 1487, 1439, 1352, 1243, 1096, 1035, 931, 840, 807, 748, 698.



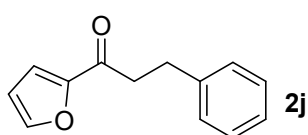
1-(Naphthalen-6-yl)-3-phenylpropan-1-one. From **1i** (51.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 9 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (31.2 mg, 60% yield).

^1H NMR (400 MHz, CDCl_3) δ 8.33 – 8.32 (m, 1H), 7.93 – 7.90 (m, 1H), 7.81 – 7.79 (m, 1H), 7.76 – 7.72 (m, 2H), 7.48 – 7.39 (m, 2H), 7.23 – 7.21 (m, 1H), 7.20 – 7.16 (m, 3H), 7.13 – 7.08 (m, 1H), 3.31 (t, $J = 8.0$ Hz, 2H), 3.02 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 199.2, 141.4, 135.6, 134.2, 132.6, 129.7, 129.6, 128.6, 128.6, 128.5, 128.5, 128.5, 127.8, 126.8, 126.2, 123.9, 40.6, 30.3.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{19}\text{H}_{17}\text{O}^+]$: 261.1274, found 261.1274.

IR (neat, cm^{-1}) 3059, 3026, 2923, 2851, 1679, 1627, 1598, 1453, 1260, 1123, 907, 731, 697.



1-(Furan-2-yl)-3-phenylpropan-1-one. From **1j** (39.6 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (150.0 μL , 3.3 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 4 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (26.1 mg, 65% yield).

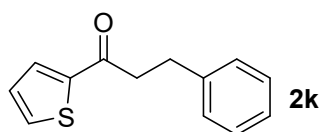
^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.45 (m, 1H), 7.21 – 7.17 (m, 2H), 7.15 – 7.13 (m, 2H), 7.12 – 7.08 (m, 1H), 7.07 – 7.06 (m, 1H), 6.42 – 6.40 (m, 1H), 3.05 (t, $J = 8.0$

Hz, 2H), 2.95 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 188.5, 152.6, 146.4, 141.0, 128.5, 128.5, 128.4, 128.4, 126.2, 117.1, 112.2, 40.2, 30.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{13}\text{H}_{12}\text{O}_2\text{Na}^+]$: 223.0730, found 223.0731.

IR (neat, cm^{-1}) 3062, 3027, 2928, 1672, 1603, 1568, 1467, 1394, 1260, 1012, 882, 754, 697.



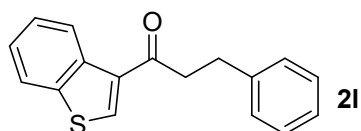
3-Phenyl-1-(thiophen-2-yl)propan-1-one. From **1k** (42.8 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (150.0 μL , 3.3 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 5 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (27.1 mg, 63% yield).

^1H NMR (300 MHz, CDCl_3) δ 7.74 – 7.72 (m, 1H), 7.67 – 7.65 (m, 1H), 7.35 – 7.28 (m, 4H), 7.25 – 7.23 (m, 1H), 7.16 – 7.14 (m, 1H), 3.28 (t, $J = 7.5$ Hz, 2H), 3.11 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 192.2, 144.2, 141.0, 133.6, 131.9, 128.6, 128.6, 128.5, 128.5, 128.1, 126.2, 41.2, 30.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{13}\text{H}_{12}\text{OSNa}^+]$: 239.0501, found 239.0503.

IR (neat, cm^{-1}) 3063, 3028, 2928, 1657, 1603, 1518, 1496, 1414, 906, 721, 698.



1-(Benzo[b]thiophen-2-yl)-3-phenylpropan-1-one. From **1l** (52.9 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (200.0 μL ,

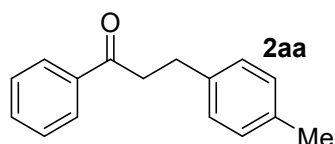
4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 8 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/20) to afford the title compound as a colorless liquid (32.8 mg, 63% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.96 – 7.86 (m, 3H), 7.49 – 7.41 (m, 2H), 7.36 – 7.31 (m, 5H), 3.38 (t, *J* = 7.5 Hz, 2H), 3.15 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 193.7, 143.5, 142.5, 140.9, 139.1, 129.0, 128.6, 128.6, 128.5, 128.5, 127.5, 126.3, 126.0, 125.0, 123.0, 41.1, 30.4.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₇H₁₄OSNa⁺]: 289.0658, found 289.0660.

IR (neat, cm⁻¹) 3024, 2954, 2920, 2863, 1665, 1600, 1566, 1496, 1453, 1290, 1210, 1160, 905, 844, 726, 698.



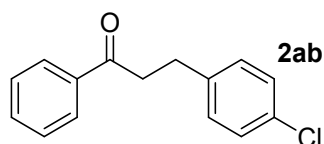
1-Phenyl-3-p-tolylpropan-1-one. From **1aa** (44.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (150.0 μL, 3.3 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 3 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (31.3 mg, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7.85 (m, 2H), 7.47 – 7.43 (m, 1H), 7.36 – 7.33 (m, 2H), 7.06 – 7.04 (m, 2H), 7.02 – 7.00 (m, 2H), 3.18 (t, *J* = 8.0 Hz, 2H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.23 (s, 3H).

¹³C NMR (75 MHz, CDCl₃) δ 199.3, 138.3, 136.9, 135.7, 133.1, 129.3, 129.3, 128.7, 128.7, 128.4, 128.4, 128.1, 128.1, 40.7, 29.8, 21.1.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₆ONa⁺]: 247.1093, found 247.1094.

IR (neat, cm⁻¹) 3022, 2922, 2861, 1683, 1597, 1580, 1514, 1448, 1203, 973, 810, 740, 688.



3-(4-Chlorophenyl)-1-phenylpropan-1-one. From **1ab** (48.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 8 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (34.7 mg, 72% yield).

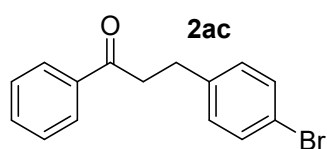
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.00 – 7.98 (m, 2H), 7.63 – 7.58 (m, 1H), 7.52 – 7.47 (m, 2H), 7.31 – 7.29 (m, 2H), 7.24 – 7.21 (m, 2H), 3.32 (t, $J = 7.5$ Hz, 2H), 3.08 (t, $J = 7.5$ Hz, 2H).

$^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 198.9, 139.8, 136.7, 133.2, 131.9, 129.9, 129.9, 128.7, 128.7, 128.6, 128.6, 128.0, 128.0, 40.2, 29.4.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{15}\text{H}_{14}^{35}\text{ClO}^+]$: 245.0728, found 245.0733.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{15}\text{H}_{14}^{37}\text{ClO}^+]$: 247.0698, found 247.0699.

IR (neat, cm^{-1}) 3027, 2931, 1684, 1597, 1580, 1492, 1448, 1091, 1015, 907, 814, 731, 688.



3-(4-Bromophenyl)-1-phenylpropan-1-one. From **1ac** (57.4 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 5 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (40.3 mg, 70% yield).

$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.00 – 7.97 (m, 2H), 7.60 – 7.57 (m, 1H), 7.51 – 7.43 (m, 4H), 7.18 – 7.15 (m, 2H), 3.31 (t, $J = 7.5$ Hz, 2H), 3.06 (t, $J = 7.5$ Hz, 2H).

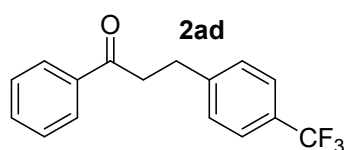
$^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 198.8, 140.3, 136.7, 133.2, 131.6, 131.6, 130.3, 130.3,

128.7, 128.7, 128.0, 128.0, 119.9, 40.1, 29.4.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{13}^{79}BrONa^+]$: 311.0042, found 311.0043.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{13}^{81}BrONa^+]$: 313.0022, found 313.0025.

IR (neat, cm^{-1}) 3060, 2926, 1682, 1597, 1580, 1487, 1448, 1403, 1205, 1071, 1011, 908, 811, 731, 688.



3-(4-(trifluoromethyl)phenyl)-1-phenylpropan-1-one. From **1ad** (55.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using $FeCl_3$ (3.2 mg, 0.020 mmol, 10 mol %), $(CF_3)_2CHOH$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^{\circ}C$ for 6 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (40.2 mg, 72% yield).

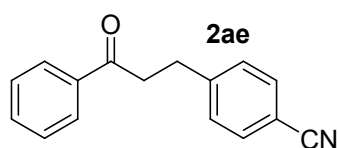
1H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.86 (m, 2H), 7.50 – 7.45 (m, 3H), 7.39 – 7.35 (m, 2H), 7.29 – 7.27 (m, 2H), 3.24 (t, $J = 8.0$ Hz, 2H), 3.05 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 144.4, 135.7, 132.2, 127.8, 127.8, 127.6, 127.6, 127.2 (q, $J = 32.3$ Hz), 127.0, 127.0, 124.4 (q, $J = 4.0$ Hz), 123.3 (q, $J = 273.7$ Hz), 38.8, 28.8.

^{19}F NMR (282 MHz, $CDCl_3$) δ -62.3.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{16}H_{13}F_3ONa^+]$: 301.0811, found 301.0816.

IR (neat, cm^{-1}) 3063, 2930, 1685, 1618, 1598, 1581, 1449, 1323, 1106, 1066, 1018, 908, 828, 742, 692.



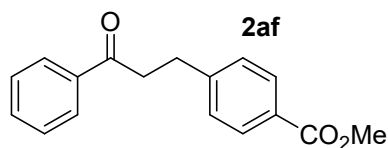
4-(3-Oxo-3-phenylpropyl)benzointrile. From **1ae** (46.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (150.0 μL, 3.3 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 10 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (31.2 mg, 66% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.99 – 7.97 (m, 2H), 7.63 – 7.60 (m, 3H), 7.52 – 7.47 (m, 2H), 7.41 – 7.39 (m, 2H), 3.36 (t, *J* = 7.5 Hz, 2H), 3.17 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 198.3, 147.0, 136.5, 133.4, 132.3, 132.3, 129.4, 129.4, 128.7, 128.7, 128.0, 128.0, 119.0, 110.1, 39.5, 30.0.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₃NONa⁺]: 258.0889, found 258.0893.

IR (neat, cm⁻¹) 3062, 3037, 2926, 2227, 1683, 1606, 1505, 1448, 1205, 1018, 909, 825, 729, 689.



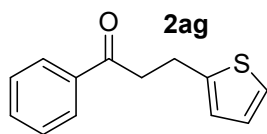
Methyl 4-(3-oxo-3-phenylpropyl)benzoate. From **1af** (53.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 4 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (36.2 mg, 68% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.89 – 7.87 (m, 2H), 7.87 – 7.86 (m, 2H), 7.49 – 7.45 (m, 1H), 7.39 – 7.35 (m, 2H), 7.25 – 7.23 (m, 2H), 3.81 (s, 3H), 3.24 (t, *J* = 8.0 Hz, 2H), 3.04 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 198.7, 167.0, 146.8, 136.7, 133.2, 129.9, 129.9, 128.7, 128.7, 128.5, 128.5, 128.0, 128.0, 121.6, 52.1, 39.8, 30.0.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₇H₁₆O₃Na⁺]: 291.0992, found 291.0993.

IR (neat, cm^{-1}) 3031, 2952, 1711, 1682, 1606, 1434, 1277, 1178, 1099, 907, 766, 731, 687.



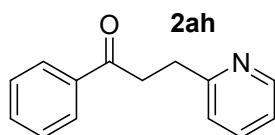
1-Phenyl-3-(thiophen-2-yl) propan-1-one. From **1ag** (42.9 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (200.0 μL , 4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 5 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (27.9 mg, 64% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 – 7.58 (m, 1H), 7.52 – 7.51 (m, 1H), 7.22 – 7.19 (m, 2H), 7.16 – 7.15 (m, 2H), 7.13 – 7.09 (m, 1H), 7.02 – 7.00 (m, 1H), 3.14 (t, $J = 8.0$ Hz, 2H), 2.98 (t, $J = 8.0$ Hz, 2H).

$^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 198.6, 143.9, 136.7, 133.2, 128.7, 128.7, 128.1, 128.1, 126.9, 124.7, 123.4, 40.6, 24.2.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{13}\text{H}_{12}\text{OSNa}^+]$: 239.0501, found 239.0501.

IR (neat, cm^{-1}) 3062, 2903, 2853, 1683, 1597, 1580, 1493, 1447, 1260, 1020, 798, 740, 687.



1-Phenyl-3-(pyridin-2-yl) propan-1-one. From **1ah** (41.8 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 8 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (19.1 mg, 46% yield).

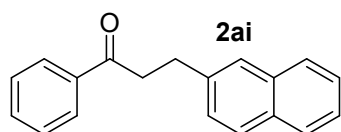
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.51 – 8.49 (m, 1H), 8.00 – 7.97 (m, 2H), 7.62 – 7.58

(m, 1H), 7.46 – 7.43 (m, 3H), 7.29 – 7.23 (m, 1H), 7.12 – 7.08 (m, 1H), 3.68 (t, $J = 7.5$ Hz, 2H), 3.51 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 198.6, 162.9, 149.2, 136.9, 136.4, 133.1, 128.6, 128.6, 128.1, 128.1, 124.2, 121.6, 43.6, 38.1.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{14}\text{H}_{14}\text{NO}^+]$: 212.1070, found 212.1075.

IR (neat, cm^{-1}) 3059, 2961, 1682, 1594, 1580, 1569, 1448, 1359, 1261, 1208, 1047, 837, 748, 688.



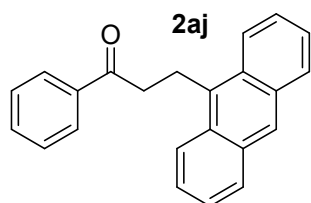
3-(Naphthalen-3-yl)-1-phenylpropan-1-one. From **1ai** (51.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 6 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (38.2 mg, 73% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.87 (m, 1H), 7.86 – 7.85 (m, 1H), 7.71 – 7.67 (m, 3H), 7.58 – 7.57 (m, 1H), 7.46 – 7.42 (m, 1H), 7.37 – 7.33 (m, 3H), 7.32 – 7.31 (m, 1H), 7.30 – 7.27 (m, 1H), 3.28 (t, $J = 8.0$ Hz, 2H), 3.13 (t, $J = 8.0$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 199.2, 138.8, 136.9, 133.7, 133.2, 132.1, 128.7, 128.7, 128.2, 128.1, 128.1, 127.7, 127.6, 127.2, 126.6, 126.1, 125.4, 40.4, 30.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{19}\text{H}_{16}\text{ONa}^+]$: 283.1093, found 283.1097.

IR (neat, cm^{-1}) 3055, 2961, 2927, 1682, 1597, 1579, 1507, 1448, 1204, 975, 811, 740, 688.



3-(Anthracen-9-yl)-1-phenylpropan-1-one. From **1aj** (61.6 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3

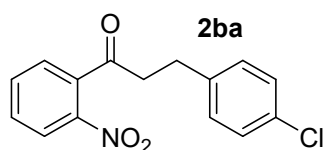
(3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (200.0 μL, 4.4 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 8 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (42.4 mg, 67% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.41 – 8.39 (m, 1H), 8.34 – 8.32 (m, 2H), 8.08 – 8.06 (m, 2H), 8.01 – 7.99 (m, 2H), 7.58 – 7.53 (m, 5H), 7.50 – 7.46 (m, 2H), 4.14 (t, *J* = 7.5 Hz, 2H), 3.50 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 199.4, 136.7, 133.5, 133.3, 131.7, 131.7, 129.6, 129.4, 129.4, 128.7, 128.7, 128.1, 128.1, 126.2, 126.2, 126.0, 126.0, 125.0, 125.0, 124.1, 124.1, 39.7, 22.0.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₂₃H₁₈ONa⁺]: 333.1250, found 333.1251.

IR (neat, cm⁻¹) 3053, 2964, 1681, 1622, 1596, 1579, 1446, 1347, 1260, 1203, 840, 728, 687.



3-(4-Chlorophenyl)-1-(2-nitrophenyl) propan-1-one. From **1ba** (57.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (52.1 mg, 90% yield).

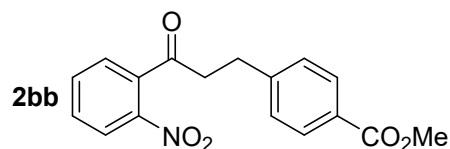
¹H NMR (300 MHz, CDCl₃) δ 8.13 – 8.10 (m, 1H), 7.74 – 7.69 (m, 1H), 7.63 – 7.59 (m, 1H), 7.34 – 7.31 (m, 1H), 7.27 – 7.24 (m, 2H), 7.19 – 7.16 (m, 2H), 3.10 – 3.08 (m, 4H).

¹³C NMR (75 MHz, CDCl₃) δ 201.3, 145.5, 139.0, 137.8, 134.4, 132.0, 130.7, 129.8, 129.8, 128.6, 128.6, 127.3, 124.5, 44.4, 29.3.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₅H₁₂NO₃³⁵ClNa⁺]: 312.0398, found 312.0401.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{12}NO_3^{37}ClNa^+]$: 314.0368, found 314.0374.

IR (neat, cm^{-1}) 2962, 2926, 2861, 1704, 1600, 1574, 1524, 1491, 1344, 1090, 1015, 853, 814, 787, 748, 699.



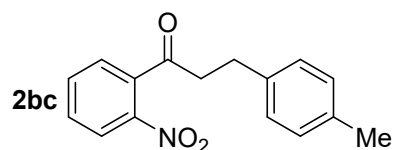
Methyl 4-(3-(2-nitrophenyl)-3-oxopropyl)benzoate. From **1bb** (62.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using $FeCl_3$ (3.2 mg, 0.020 mmol, 10 mol %), $(CF_3)_2CHOH$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^{\circ}C$ for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (54.4 mg, 86% yield).

1H NMR (300 MHz, $CDCl_3$) δ 8.15 – 8.12 (m, 1H), 7.98 – 7.96 (m, 2H), 7.74 – 7.69 (m, 1H), 7.64 – 7.59 (m, 1H), 7.33 – 7.30 (m, 3H), 3.91 (s, 3H), 3.22 – 3.10 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 167.0, 146.0, 145.5, 137.8, 134.4, 130.6, 129.9, 129.9, 128.5, 128.5, 128.3, 127.2, 124.5, 52.1, 44.1, 30.0.

HRMS (ESI) m/z : $[M + H]^+$ Calculated for $[C_{17}H_{16}NO_5^+]$: 314.1023, found 314.1024.

IR (neat, cm^{-1}) 3033, 2999, 2953, 1708, 1610, 1574, 1525, 1435, 1345, 1217, 1080, 1103, 1019, 984, 909, 855, 732.



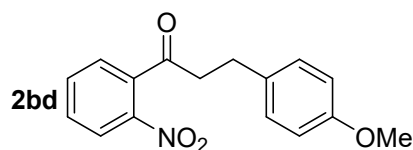
1-(2-Nitrophenyl)-3-p-tolylpropan-1-one. From **1bc** (53.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using $FeCl_3$ (3.2 mg, 0.020 mmol, 10 mol %), $(CF_3)_2CHOH$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^{\circ}C$ for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (48.3 mg, 89% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.14 – 8.11 (m, 1H), 7.75 – 7.70 (m, 1H), 7.64 – 7.59 (m, 1H), 7.36 – 7.34 (m, 1H), 7.19 – 7.16 (m, 4H), 3.13 – 3.12 (m, 4H), 2.37 (s, 3H).

¹³C NMR (75 MHz, CDCl₃) δ 201.9, 145.6, 138.0, 137.4, 135.8, 134.4, 130.6, 129.3, 129.3, 128.3, 128.3, 127.4, 124.4, 44.8, 29.7, 21.0.

HRMS (ESI) m/z: [M + Na]⁺ Calculated for [C₁₆H₁₅NO₃Na⁺]: 292.0944, found 292.0944.

IR (neat, cm⁻¹) 2964, 2922, 1705, 1527, 1345, 1260, 1084, 1046, 906, 853, 729, 699.



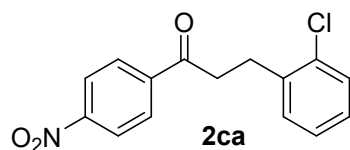
3-(4-Methoxyphenyl)-1-(2-nitrophenyl) propan-1-one. From **1bd** (37.4 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/6) to afford the title compound as a colorless liquid (47.9 mg, 84% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.97 (m, 1H), 7.59 – 7.55 (m, 1H), 7.49 – 7.45 (m, 1H), 7.19 – 7.17 (m, 1H), 7.04 – 7.01 (m, 2H), 6.73 – 6.70 (m, 2H), 3.66 (s, 3H), 2.98 – 2.92 (m, 4H).

¹³C NMR (75 MHz, CDCl₃) δ 201.9, 158.1, 145.6, 138.0, 134.4, 132.5, 130.6, 129.4, 129.4, 127.3, 124.4, 114.0, 114.0, 55.3, 44.9, 29.2.

HRMS (ESI) m/z: [M + Na]⁺ Calculated for [C₁₆H₁₅NO₄Na⁺]: 308.0893, found 308.0896.

IR (neat, cm⁻¹) 3033, 2959, 2934, 1707, 1611, 1575, 1527, 1511, 1345, 1244, 1032, 825, 729.



3-(2-Chlorophenyl)-1-(4-nitrophenyl) propan-1-one. From **1ca** (57.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (38.3 mg, 66% yield).

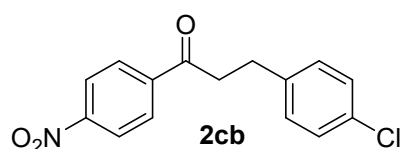
¹H NMR (300 MHz, CDCl₃) δ 8.34 – 8.31 (m, 2H), 8.15 – 8.12 (m, 2H), 7.41 – 7.38 (m, 1H), 7.35 – 7.32 (m, 1H), 7.24 – 7.22 (m, 1H), 7.21 – 7.18 (m, 1H), 3.39 (t, *J* = 7.5 Hz, 2H), 3.22 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.5, 150.3, 141.1, 138.2, 133.9, 130.9, 129.7, 129.1, 129.1, 128.1, 127.1, 123.9, 123.9, 39.0, 28.2.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₅H₁₂NO₃³⁵ClNa⁺]: 312.0398, found 312.0399.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₅H₁₂NO₃³⁷ClNa⁺]: 314.0368, found 314.0370.

IR (neat, cm⁻¹) 3072, 2962, 2859, 1692, 1602, 1522, 1475, 1443, 1343, 1199, 1107, 1051, 979, 853, 789, 740.



3-(4-Chlorophenyl)-1-(4-nitrophenyl) propan-1-one. From **1cb** (57.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (39.2 mg, 67% yield).

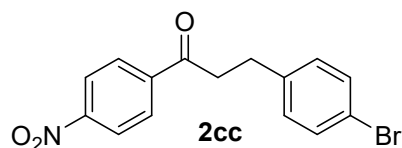
¹H NMR (300 MHz, CDCl₃) δ 8.31 – 8.28 (m, 2H), 8.13 – 8.10 (m, 2H), 7.28 – 7.19 (m, 4H), 3.36 (t, *J* = 7.5 Hz, 2H), 3.08 (t, *J* = 7.5 Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.3, 150.3, 141.1, 139.1, 132.1, 129.8, 129.8, 129.0, 129.0, 128.7, 128.7, 123.9, 123.9, 40.7, 29.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{15}\text{H}_{12}\text{NO}_3^{35}\text{ClNa}^+]$: 312.0398, found 312.0402.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{15}\text{H}_{12}\text{NO}_3^{37}\text{ClNa}^+]$: 314.0368, found 314.0372.

IR (neat, cm^{-1}) 3051, 2924, 2853, 1686, 1599, 1520, 1488, 1404, 1345, 1266, 1095, 906, 859, 811, 735.



3-(4-Bromophenyl)-1-(4-nitrophenyl) propan-1-one. From **1cc** (66.4 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (42.2 mg, 64% yield).

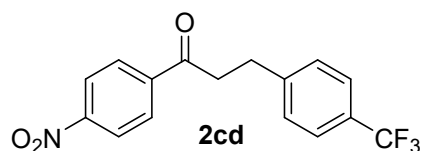
^1H NMR (300 MHz, CDCl_3) δ 8.32 – 8.29 (m, 2H), 8.13 – 8.10 (m, 2H), 7.44 – 7.41 (m, 2H), 7.17 – 7.14 (m, 2H), 3.36 (t, $J = 7.5$ Hz, 2H), 3.07 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.2, 150.3, 141.1, 139.6, 131.7, 131.7, 130.2, 130.2, 129.0, 129.0, 123.9, 123.9, 120.1, 40.6, 29.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{15}\text{H}_{12}\text{NO}_3^{79}\text{BrNa}^+]$: 355.9893, found 355.9897.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{15}\text{H}_{12}\text{NO}_3^{81}\text{BrNa}^+]$: 357.9872, found 357.9872.

IR (neat, cm^{-1}) 3111, 2926, 2854, 1687, 1600, 1521, 1487, 1404, 1343, 1261, 1196, 1071, 1010, 906, 852, 808, 729.



3-(4-(Trifluoromethyl) phenyl)-1-(4-nitrophenyl) propan-1-one. From **1cd** (64.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (47.7 mg, 74% yield).

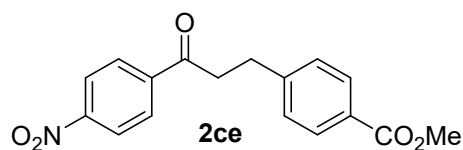
¹H NMR (400 MHz, CDCl₃) δ 8.22 – 8.20 (m, 2H), 8.03 – 8.00 (m, 2H), 7.48 – 7.46 (m, 2H), 7.30 – 7.28 (m, 2H), 3.30 (t, *J* = 8.0 Hz, 2H), 3.08 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.0, 150.4, 144.8, 141.0, 129.0, 129.0, 128.8, 128.8, 128.7 (q, *J* = 32.3 Hz), 125.5 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 270.0 Hz), 123.9, 123.9, 40.4, 29.5.

¹⁹F NMR (282 MHz, CDCl₃) δ -62.4.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₂F₃NO₃Na⁺]: 346.0661, found 346.0662.

IR (neat, cm⁻¹) 3112, 2962, 2856, 1689, 1600, 1523, 1413, 1346, 1319, 1260, 1161, 1105, 1085, 1016, 907, 789, 734.



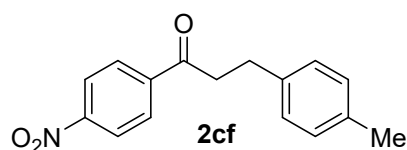
Methyl 4-(3-(4-nitrophenyl)-3-oxopropyl) benzoate. From **1ce** (62.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (40.0 mg, 65% yield).

^1H NMR (300 MHz, CDCl_3) δ 8.33 – 8.30 (m, 2H), 8.13 – 8.10 (m, 2H), 8.00 – 7.97 (m, 2H), 7.36 – 7.33 (m, 2H), 3.92 (s, 3H), 3.40 (t, $J = 7.5$ Hz, 2H), 3.17 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.1, 166.9, 150.4, 146.1, 141.0, 130.0, 130.0, 129.1, 129.1, 128.5, 128.5, 128.4, 123.9, 123.9, 52.1, 40.4, 29.7.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{17}\text{H}_{16}\text{NO}_5^+]$: 314.1023, found 314.1024.

IR (neat, cm^{-1}) 2955, 2852, 1715, 1695, 1607, 1525, 1436, 1345, 1279, 1107, 1020, 906, 854, 728.



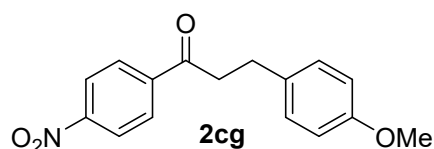
1-(4-Nitrophenyl)-3-p-tolylpropan-1-one. From **1cf** (53.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl_3 (3.2 mg, 0.020 mmol, 10 mol %), $(\text{CF}_3)_2\text{CHOH}$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^\circ\text{C}$ for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (35.5 mg, 66% yield).

^1H NMR (400 MHz, CDCl_3) δ 8.22 – 8.20 (m, 2H), 8.02 – 7.99 (m, 2H), 7.07 – 7.05 (m, 2H), 7.04 – 7.02 (m, 2H), 3.24 (t, $J = 8.0$ Hz, 2H), 2.97 (t, $J = 8.0$ Hz, 2H), 2.24 (s, 3H).

^{13}C NMR (75 MHz, CDCl_3) δ 197.8, 150.3, 141.3, 137.6, 135.9, 129.3, 129.3, 129.1, 129.1, 128.3, 128.3, 123.9, 123.9, 41.1, 29.5, 21.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calculated for $[\text{C}_{16}\text{H}_{15}\text{NO}_3\text{Na}^+]$: 292.0944, found 292.0945.

IR (neat, cm^{-1}) 3112, 2920, 2855, 1686, 1599, 1519, 1407, 1345, 1318, 1199, 973, 865, 810, 796, 731.



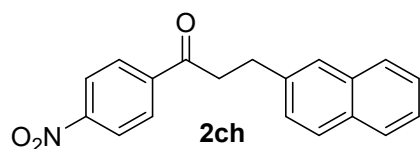
3-(4-Methoxyphenyl)-1-(4-nitrophenyl) propan-1-one. From **1cg** (56.7 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (36.8 mg, 64% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.31 – 8.28 (m, 2H), 8.12 – 8.09 (m, 2H), 7.20 – 7.17 (m, 2H), 6.87 – 6.84 (m, 2H), 3.80 (s, 3H), 3.34 (t, *J* = 7.5 Hz, 2H), 3.05 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.9, 158.1, 150.2, 141.3, 132.6, 129.4, 129.4, 129.1, 129.1, 123.9, 123.9, 114.0, 114.0, 55.3, 41.2, 29.0.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₆H₁₅NO₄Na⁺]: 308.0893, found 308.0897.

IR (neat, cm⁻¹) 3076, 2959, 2925, 1689, 1604, 1511, 1344, 1244, 1030, 820, 796.



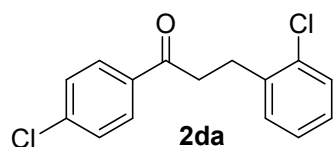
3-(Naphthalen-3-yl)-1-(4-nitrophenyl) propan-1-one. From **1ch** (37.4 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (37.3 mg, 61% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.17 – 8.16 (m, 1H), 8.15 – 8.14 (m, 1H), 7.99 – 7.97 (m, 1H), 7.96 – 7.95 (m, 1H), 7.71 – 7.66 (m, 3H), 7.58 – 7.57 (m, 1H), 7.36 – 7.32 (m, 2H), 7.29 – 7.26 (m, 1H), 3.31 (t, *J* = 8.0 Hz, 2H), 3.15 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.6, 150.3, 141.2, 138.1, 133.6, 132.2, 129.1, 129.1, 128.3, 127.7, 127.5, 127.0, 126.6, 126.2, 125.5, 123.9, 123.9, 40.9, 30.0.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{19}H_{15}NO_3Na^+]$: 328.0944, found 328.0945.

IR (neat, cm^{-1}) 3050, 2924, 2854, 1686, 1600, 1518, 1404, 1342, 1260, 1195, 1103, 1012, 907, 854, 804, 735.



3-(2-Chlorophenyl)-1-(4-chlorophenyl) propan-1-one. From **1da** (55.4 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using $FeCl_3$ (3.2 mg, 0.020 mmol, 10 mol %), $(CF_3)_2CHOH$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^{\circ}C$ for 1.5 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (31.0 mg, 55% yield).

1H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.90 (m, 2H), 7.45 – 7.41 (m, 2H), 7.38 – 7.36 (m, 1H), 7.32 – 7.31 (m, 1H), 7.21 – 7.19 (m, 2H), 3.31 – 3.27 (m, 2H), 3.20 – 3.18 (m, 2H).

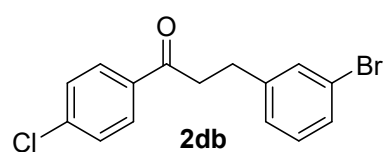
^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 139.5, 138.6, 135.0, 133.9, 130.8, 129.6, 129.5, 129.5, 128.9, 128.9, 127.9, 127.0, 38.4, 28.3.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{12}O^{35}Cl_2Na^+]$: 301.0157, found 301.0158.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{12}O^{35}Cl^{37}ClNa^+]$: 303.0128, found 303.0130.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{15}H_{12}O^{37}Cl_2Na^+]$: 305.0098, found 305.0101.

IR (neat, cm^{-1}) 3063, 2938, 1684, 1588, 1572, 1488, 1443, 1400, 1203, 1090, 1013, 976, 907, 831, 750, 729.



3-(3-Bromophenyl)-1-(4-chlorophenyl) propan-1-one. From **1db** (64.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (35.3 mg, 55% yield).

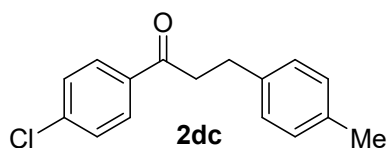
¹H NMR (400 MHz, CDCl₃) δ 7.81 – 7.80 (m, 1H), 7.79 – 7.78 (m, 1H), 7.35 – 7.34 (m, 1H), 7.33 – 7.31 (m, 2H), 7.26 – 7.23 (m, 1H), 7.10 – 7.06 (m, 2H), 3.16 (t, *J* = 8.0 Hz, 2H), 2.94 (t, *J* = 8.0 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 197.4, 143.5, 139.6, 135.0, 131.5, 130.1, 129.5, 129.5, 129.4, 129.0, 129.0, 127.2, 122.6, 40.0, 29.5.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₅H₁₂O³⁵Cl⁷⁹BrNa⁺]: 344.9652, found 344.9653.

HRMS (ESI) *m/z*: [M + Na]⁺ Calculated for [C₁₅H₁₂O³⁵Cl⁸¹BrNa⁺]: 346.9632, found 346.9632.

IR (neat, cm⁻¹) 3060, 2929, 1683, 1588, 1568, 1487, 1400, 1203, 1090, 978, 906, 778, 729.



1-(4-Chlorophenyl)-3-p-tolylpropan-1-one. From **1dc** (51.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 1 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (29.0 mg, 56% yield).

¹H NMR (300 MHz, CDCl₃) δ 7.95 – 7.92 (m, 2H), 7.48 – 7.45 (m, 2H), 7.21 – 7.18 (m, 4H), 3.30 (t, *J* = 7.5 Hz, 2H), 3.07 (t, *J* = 7.5 Hz, 2H), 2.38 (s, 3H).

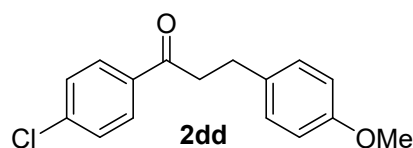
¹³C NMR (75 MHz, CDCl₃) δ 198.1, 139.5, 138.0, 135.8, 135.2, 129.5, 129.5, 129.3,

129.3, 128.9, 128.9, 128.3, 128.3, 40.6, 29.7, 21.1.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{16}H_{15}O^{35}ClNa^+]$: 281.0704, found 281.0706.

HRMS (ESI) m/z : $[M + Na]^+$ Calculated for $[C_{16}H_{15}O^{37}ClNa^+]$: 283.0674, found 283.0677.

IR (neat, cm^{-1}) 3023, 2915, 2857, 1674, 1587, 1570, 1515, 1486, 1200, 1092, 906, 806, 728.



1-(4-Chlorophenyl)-3-(4-methoxyphenyl) propan-1-one. From **1dd** (54.5 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using $FeCl_3$ (3.2 mg, 0.020 mmol, 10 mol %), $(CF_3)_2CHOH$ (60.0 μL , 2.0 equiv), PMHS (100.0 μL , 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 $^{\circ}C$ for 5 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (31.0 mg, 57% yield).

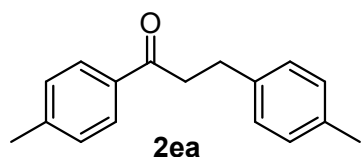
1H NMR (300 MHz, $CDCl_3$) δ 7.93 – 7.90 (m, 2H), 7.46 – 7.43 (m, 2H), 7.21 – 7.19 (m, 2H), 6.90 – 6.87 (m, 2H), 3.82 (s, 3H), 3.27 (t, $J = 7.5$ Hz, 2H), 3.04 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 198.1, 158.0, 139.5, 135.2, 133.1, 129.5, 129.5, 129.4, 129.4, 128.9, 128.9, 114.0, 114.0, 55.3, 40.7, 29.2.

HRMS (ESI) m/z : $[M + H]^+$ Calculated for $[C_{16}H_{16}O_2^{35}Cl^+]$: 275.0833, found 275.0833.

HRMS (ESI) m/z : $[M + H]^+$ Calculated for $[C_{16}H_{16}O_2^{37}Cl^+]$: 277.0804, found 277.0807.

IR (neat, cm^{-1}) 2999, 2933, 2835, 1684, 1611, 1588, 1511, 1464, 1441, 1244, 1090, 1034, 908, 818, 729.



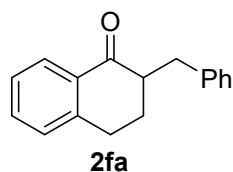
1,3-dip-tolylpropan-1-one. From **1ea** (47.3 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol%), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 2 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (26.1 mg, 54% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.78 – 7.76 (m, 2H), 7.16 – 7.14 (m, 2H), 7.06 – 7.04 (m, 2H), 7.02 – 7.00 (m, 2H), 3.16 (t, *J* = 8.0 Hz, 2H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.31 (s, 3H), 2.23 (s, 3H).

¹³C NMR (75 MHz, CDCl₃) δ 199.0, 143.8, 138.4, 135.6, 134.5, 129.3, 129.3, 129.3, 129.3, 128.4, 128.4, 128.2, 128.2, 40.6, 29.9, 21.7, 21.1.

HRMS (ESI) *m/z*: [M + H]⁺ Calculated for [C₁₇H₁₉O⁺]: 239.1430, found 239.1431.

IR (neat, cm⁻¹) 3022, 2916, 2861, 1678, 1605, 1572, 1514, 1447, 1179, 976, 908, 799, 730.



2-benzyl-3,4-dihydronaphthalen-1(2H)-one.

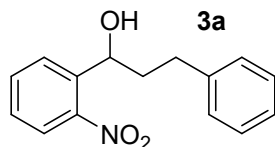
From **1fa** (46.8 mg, 0.20 mmol, 1.0 equiv), the title compound was prepared following the general procedure using FeCl₃ (3.2 mg, 0.020 mmol, 10 mol %), (CF₃)₂CHOH (60.0 μL, 2.0 equiv), PMHS (100.0 μL, 2.2 equiv) and DCE (0.5 mL). The reaction mixture was stirred at 60 °C for 4 h. The crude product was purified by column chromatography on silica gel (EtOAc/PE = 1/10) to afford the title compound as a colorless liquid (28.1 mg, 60% yield).

¹H NMR (300 MHz, CDCl₃) δ 8.15 – 8.10 (m, 1H), 7.54 – 7.49 (m, 1H), 7.39 – 7.35 (m, 3H), 7.30 – 7.26 (m, 4H), 3.59 – 3.53 (m, 1H), 2.98 – 2.95 (m, 2H), 2.84 – 2.67 (m, 2H), 2.19 – 2.12 (m, 1H), 1.87 – 1.77 (m, 1H).

^{13}C NMR (75 MHz, CDCl_3) δ 199.4, 144.1, 140.1, 133.3, 132.5, 129.3, 129.3, 128.8, 128.5, 128.5, 127.6, 126.7, 126.2, 49.5, 35.7, 28.7, 27.7.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{17}\text{H}_{17}\text{O}^+]$: 237.1274, found 237.1275.

IR (neat, cm^{-1}) 3062, 2927, 2861, 1679, 1599, 1495, 1453, 1291, 1219, 1156, 1029, 933, 739, 700.



1-(2-nitrophenyl)-3-phenylpropan-1-ol.

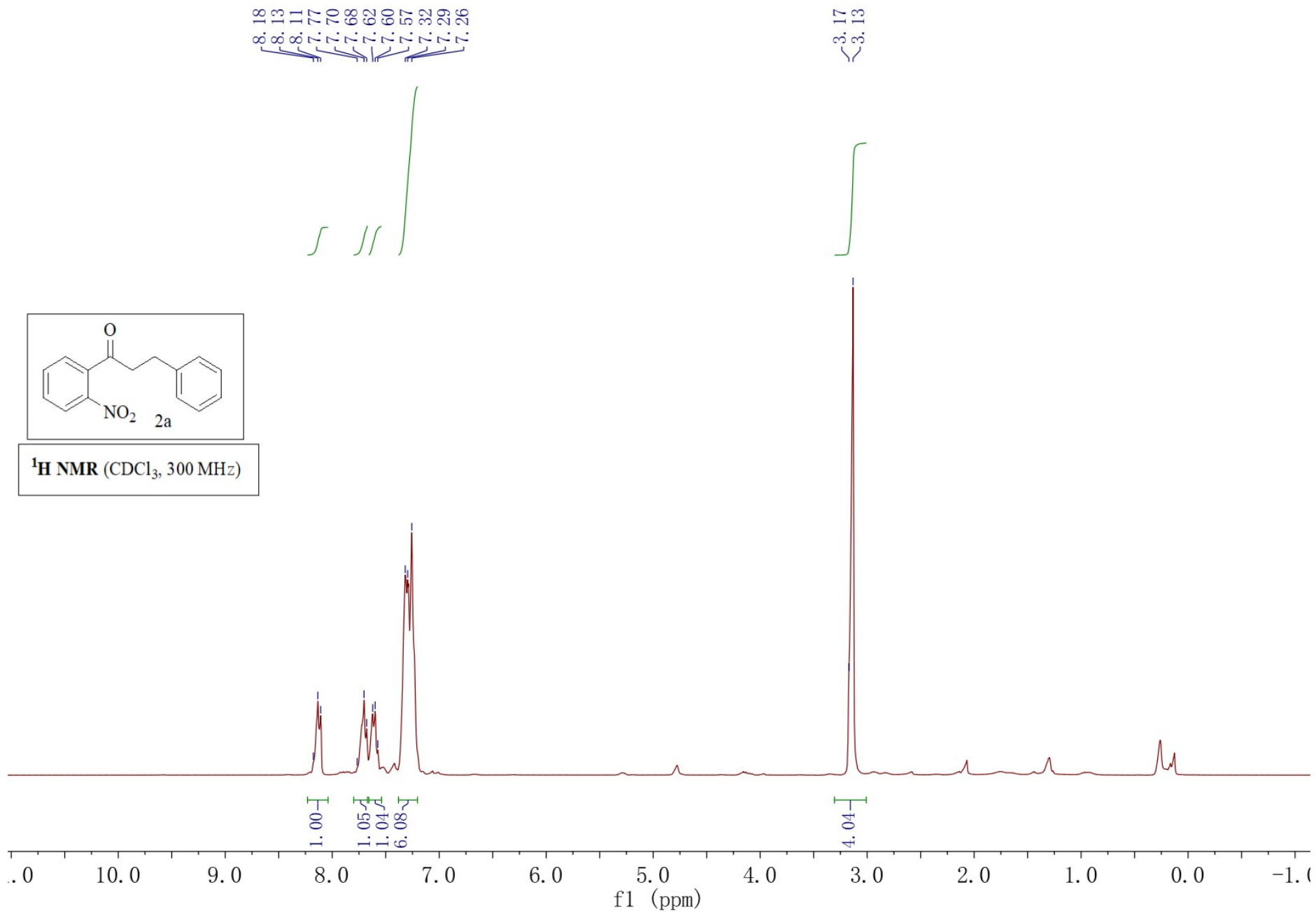
^1H NMR (300 MHz, CDCl_3) δ 7.94 – 7.91 (m, 1H), 7.85 – 7.83 (m, 1H), 7.67 – 7.62 (m, 1H), 7.45 – 7.40 (m, 1H), 7.36 – 7.31 (m, 2H), 7.27 – 7.24 (m, 3H), 5.28 (dd, $J = 6.0, 7.5$ Hz, 1H), 3.01 – 2.91 (m, 2H), 2.87 – 2.77 (m, 1H), 2.20 – 2.02 (m, 2H).

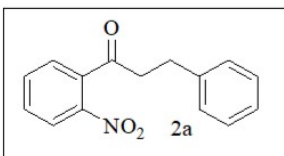
^{13}C NMR (75 MHz, CDCl_3) δ 147.7, 141.5, 140.2, 133.6, 128.5, 128.5, 128.5, 128.5, 128.2, 128.1, 126.1, 124.4, 69.0, 39.8, 32.6.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calculated for $[\text{C}_{15}\text{H}_{16}\text{NO}_3^+]$: 258.1125, found 258.1125.

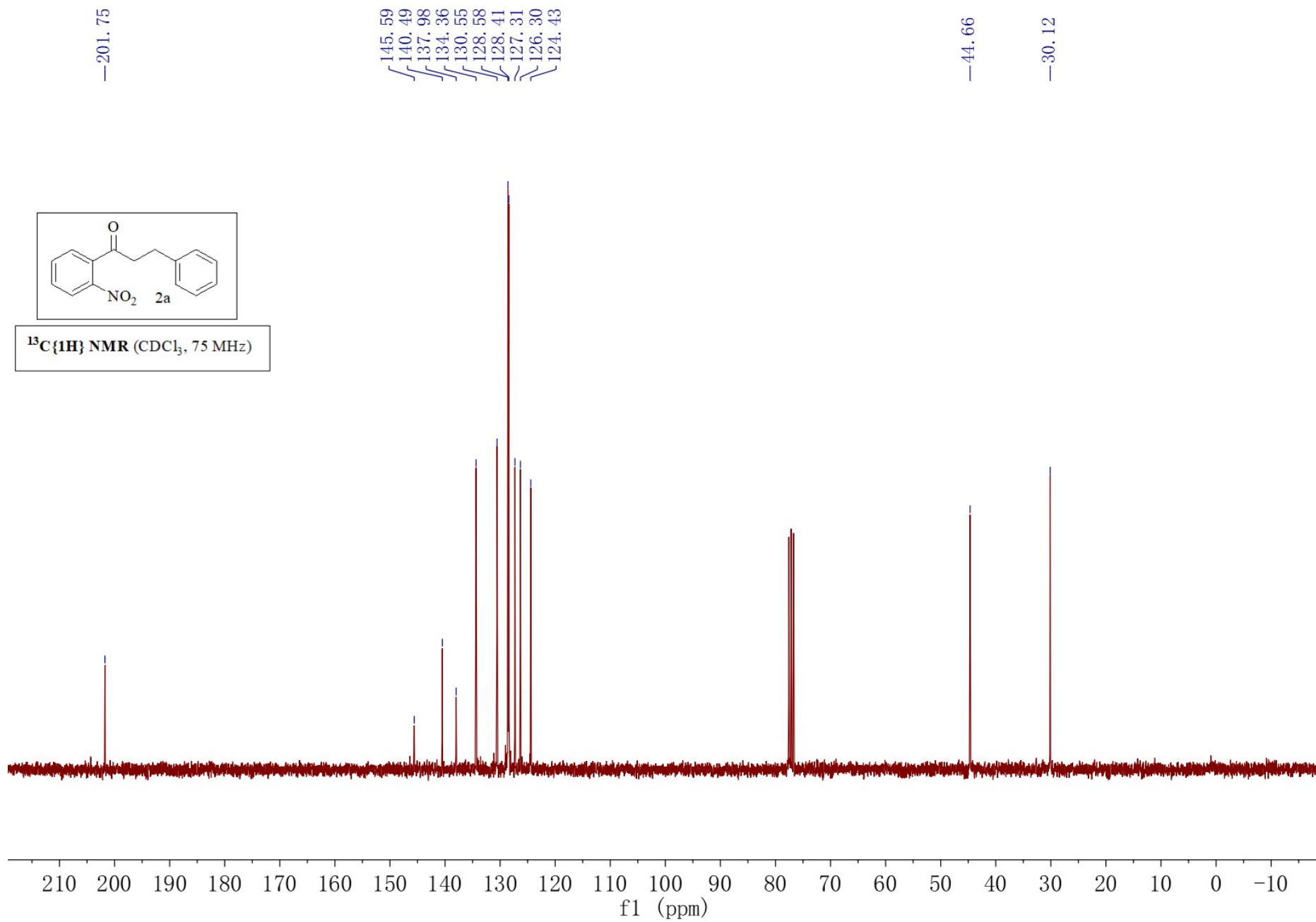
IR (neat, cm^{-1}) 3552, 3424, 3027, 2924, 2859, 1607, 1578, 1520, 1453, 1342, 1190, 1057, 909, 857, 787, 737, 698.

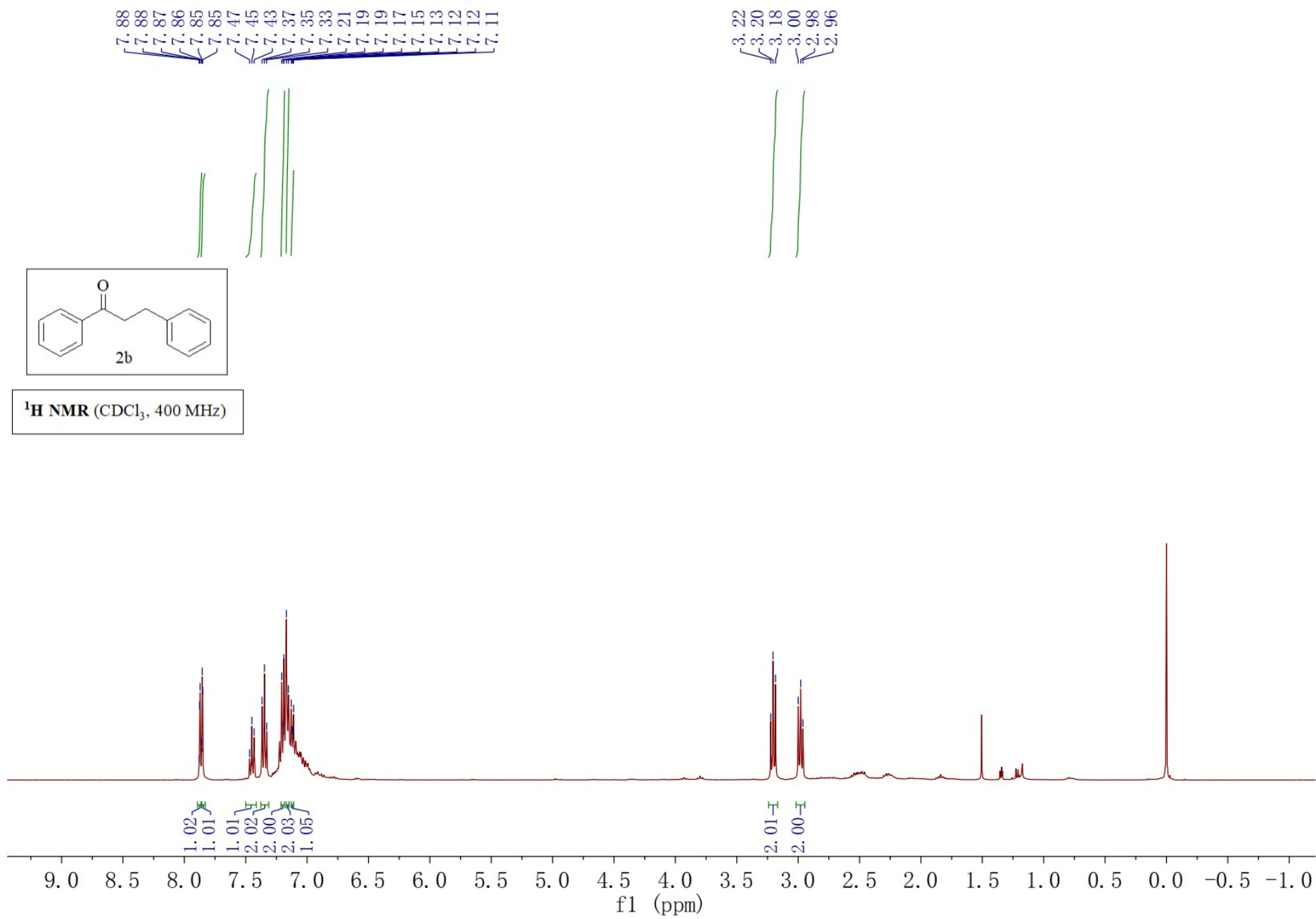
VIII. Spectroscopic Data (NMR and IR Spectrum)

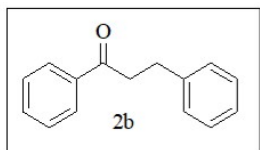




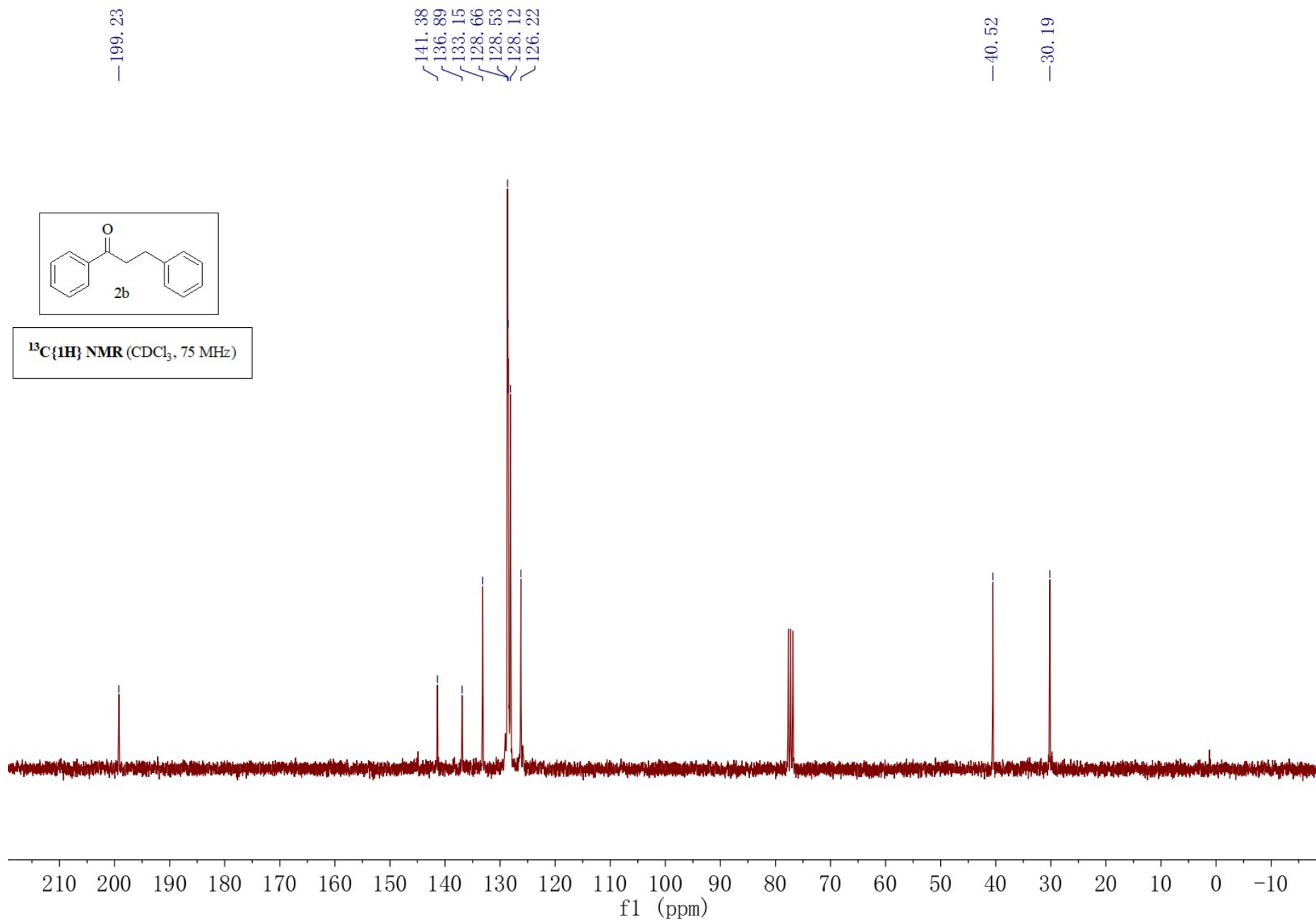
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

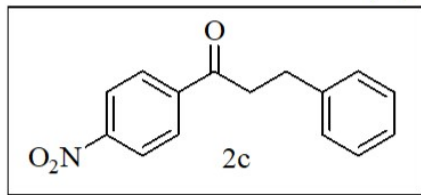




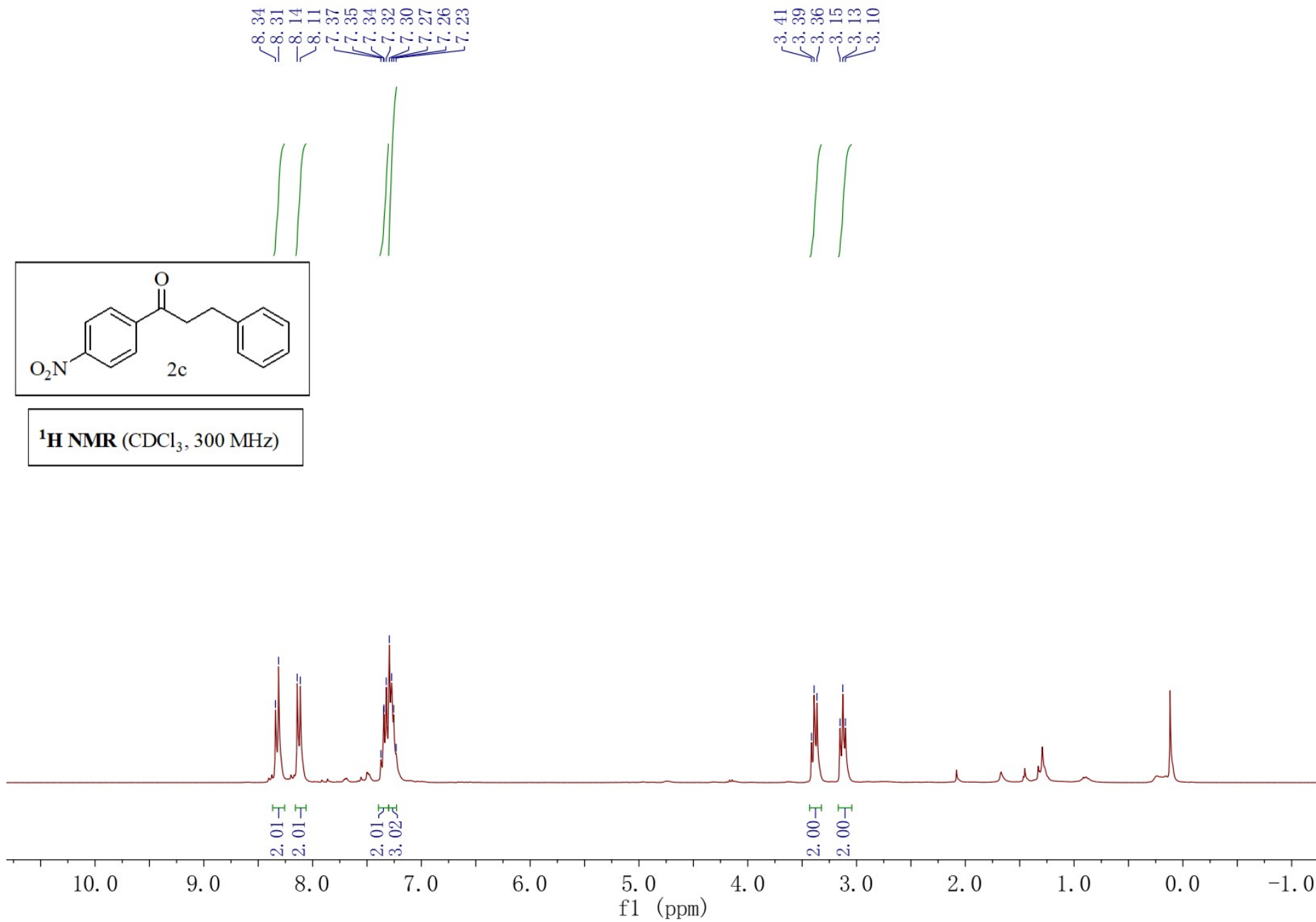


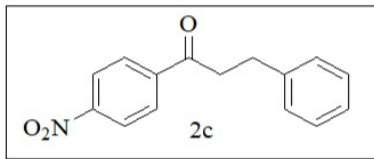
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



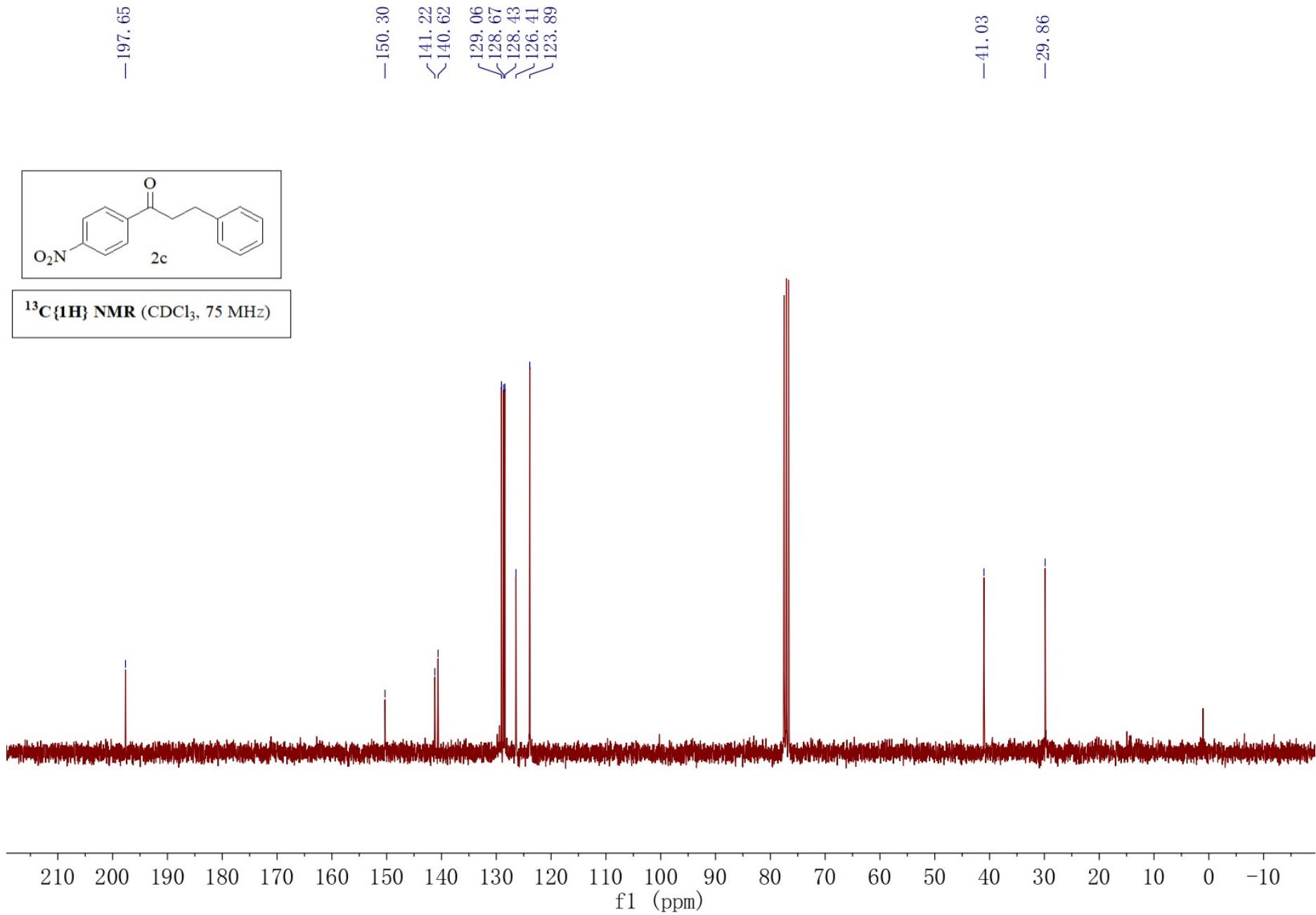


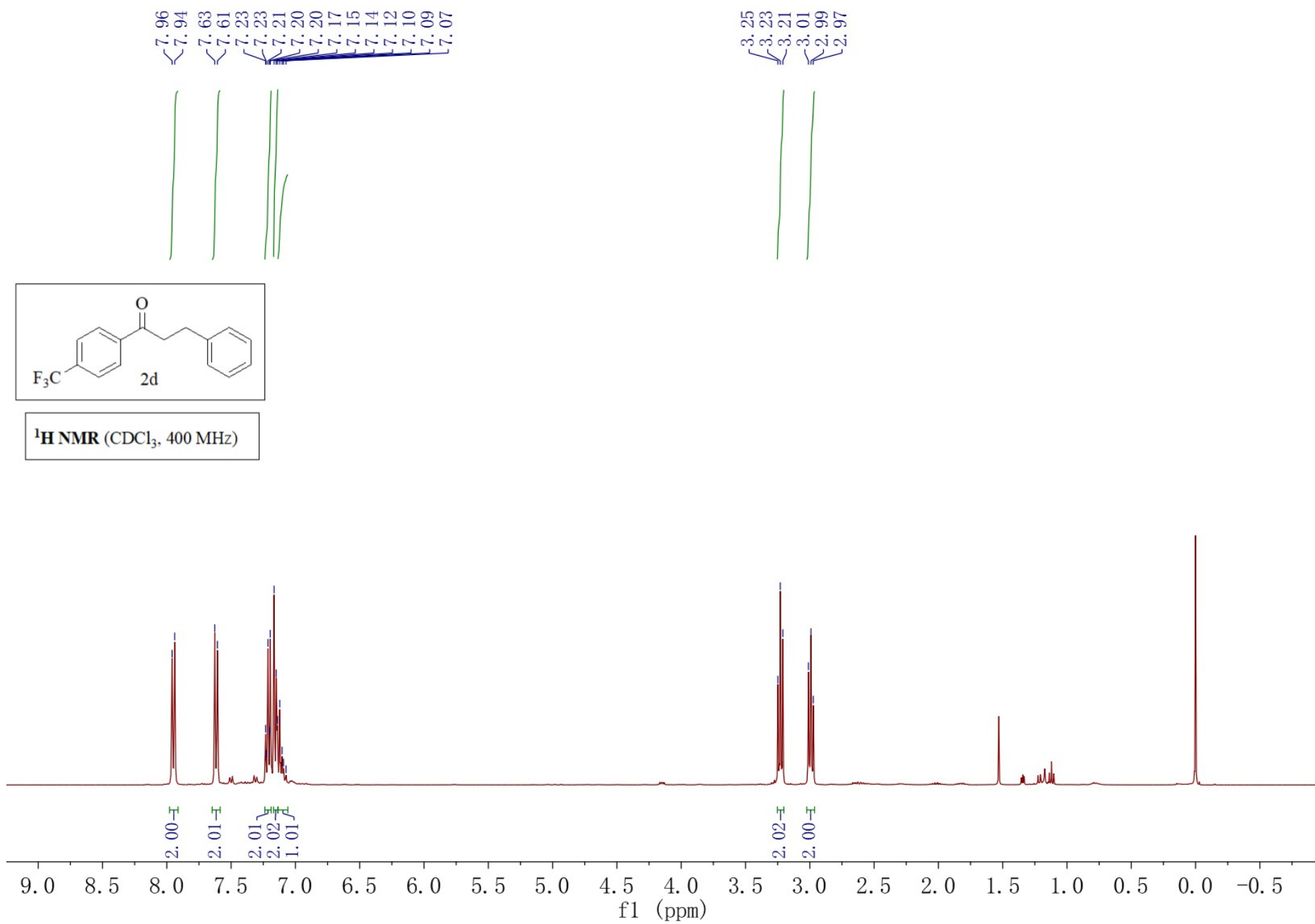
$^1\text{H NMR}$ (CDCl_3 , 300 MHz)



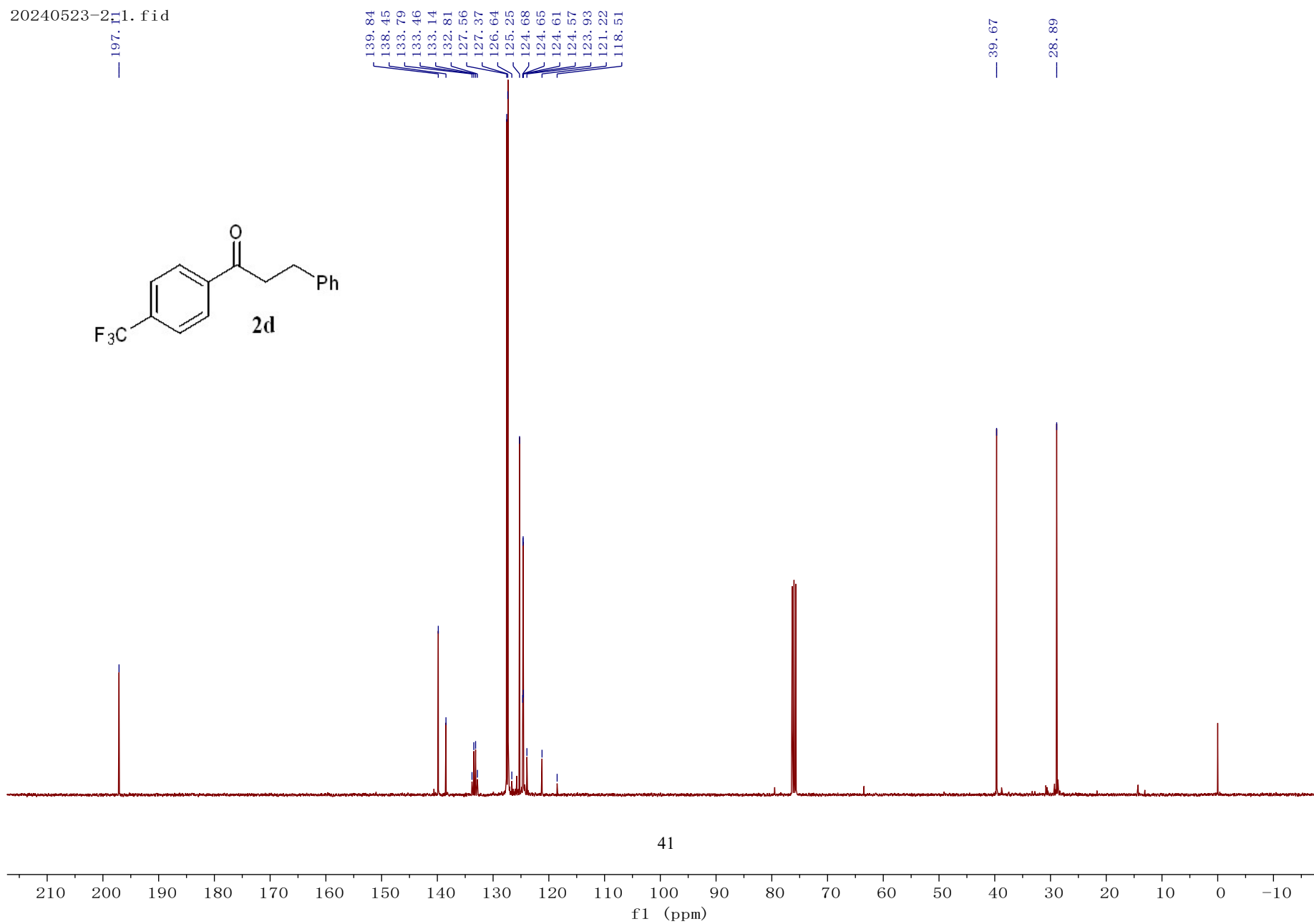
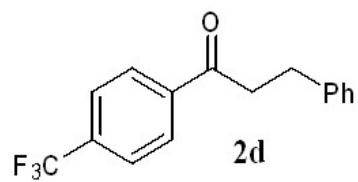


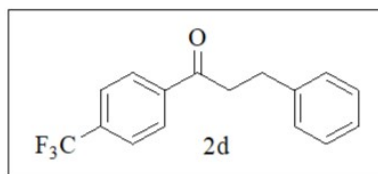
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)





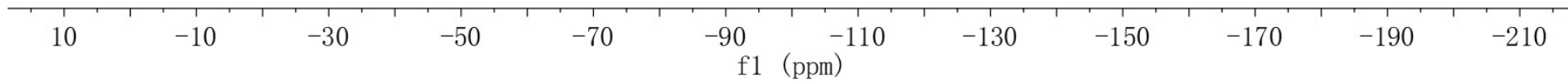
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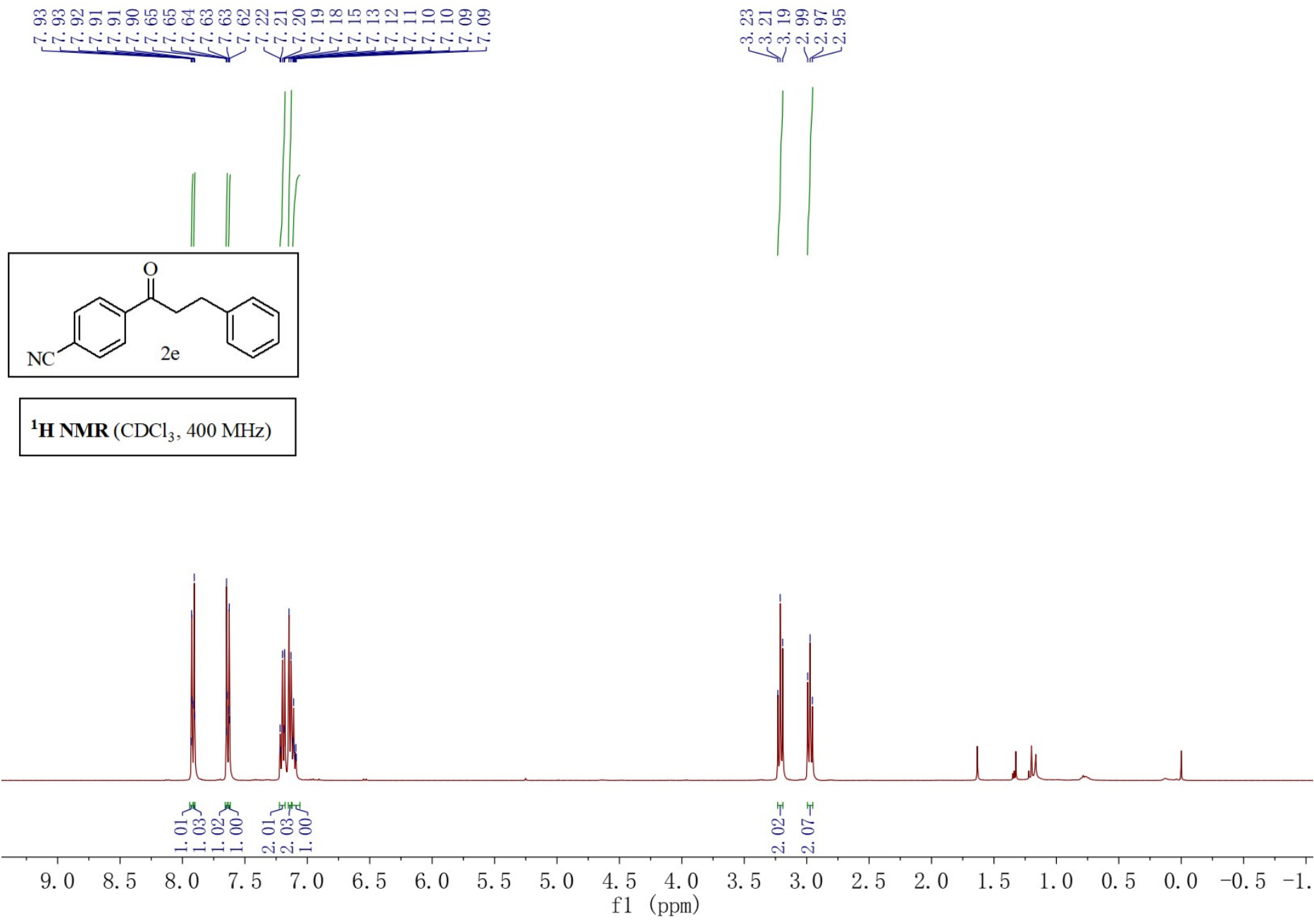


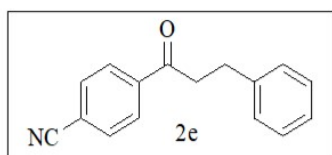


¹⁹F NMR (CDCl₃, 282 MHz)

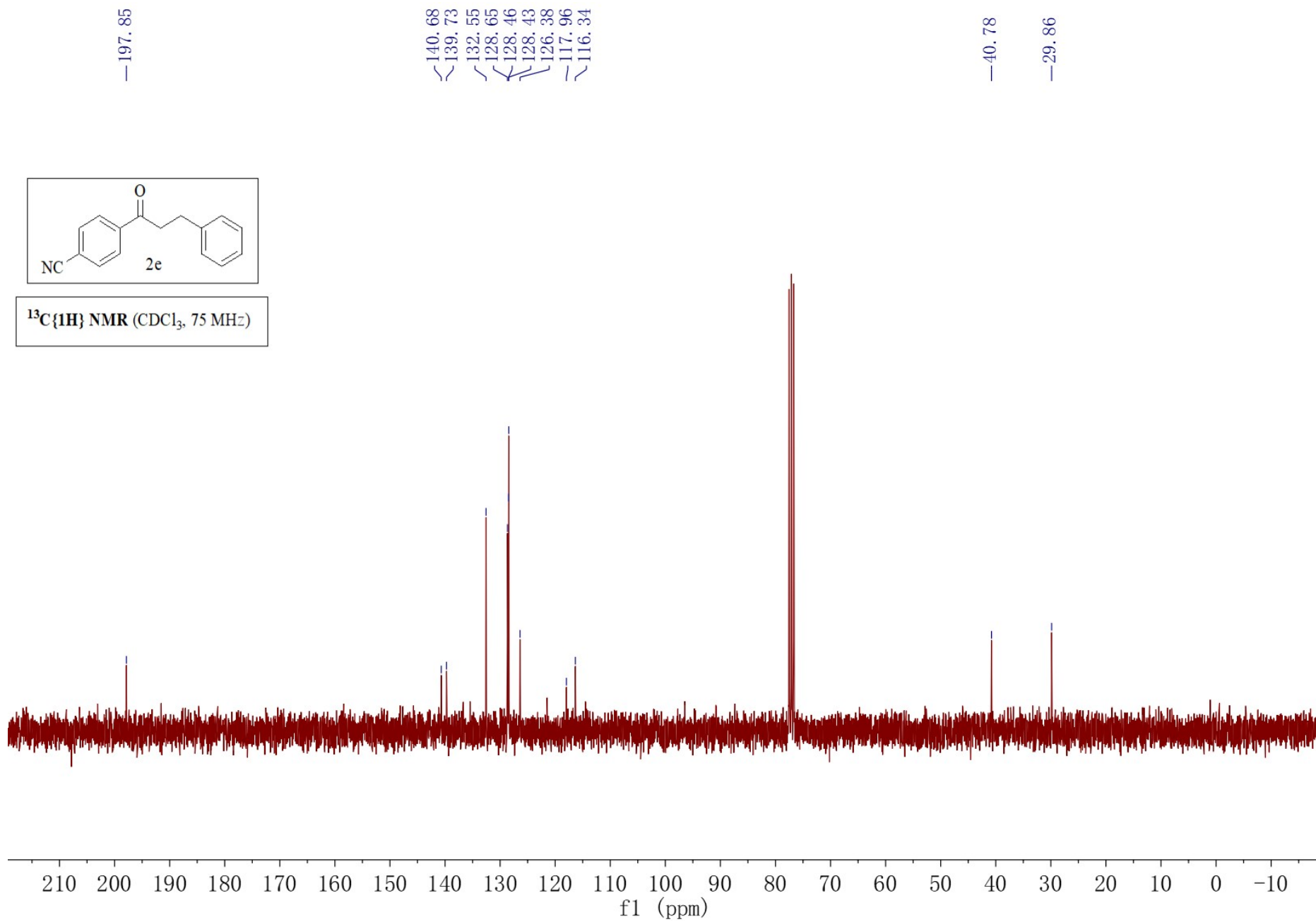
— -63.06

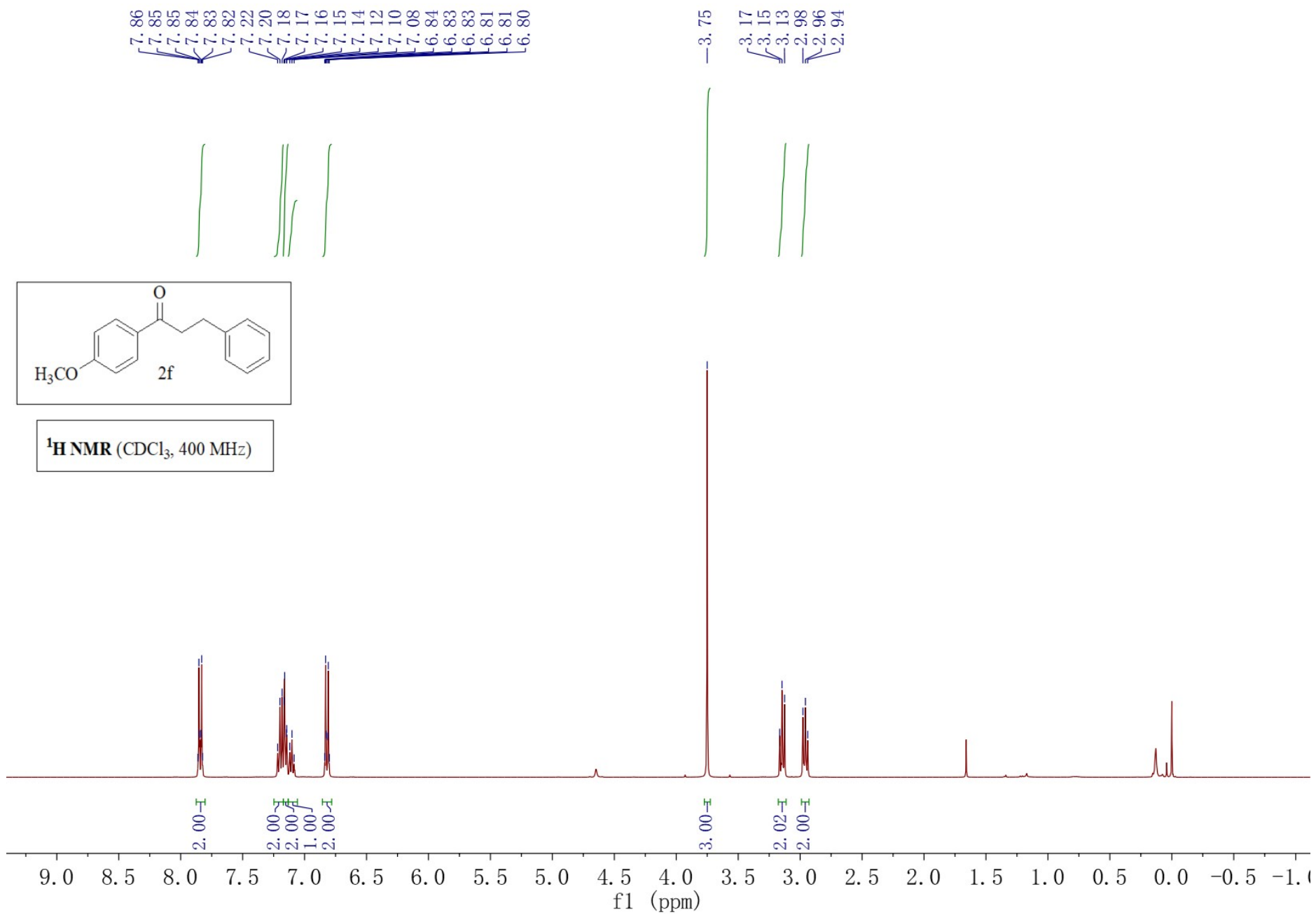


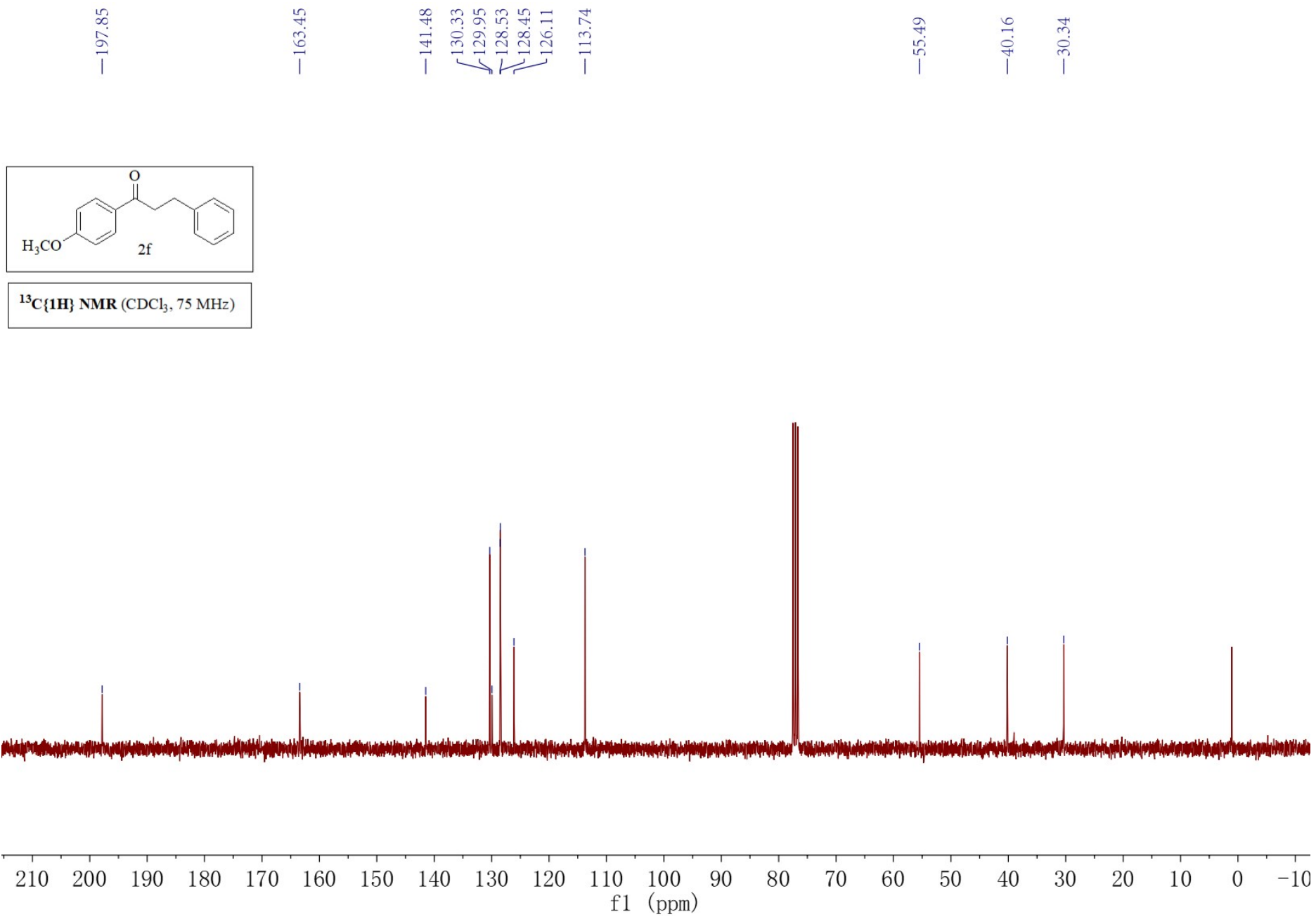


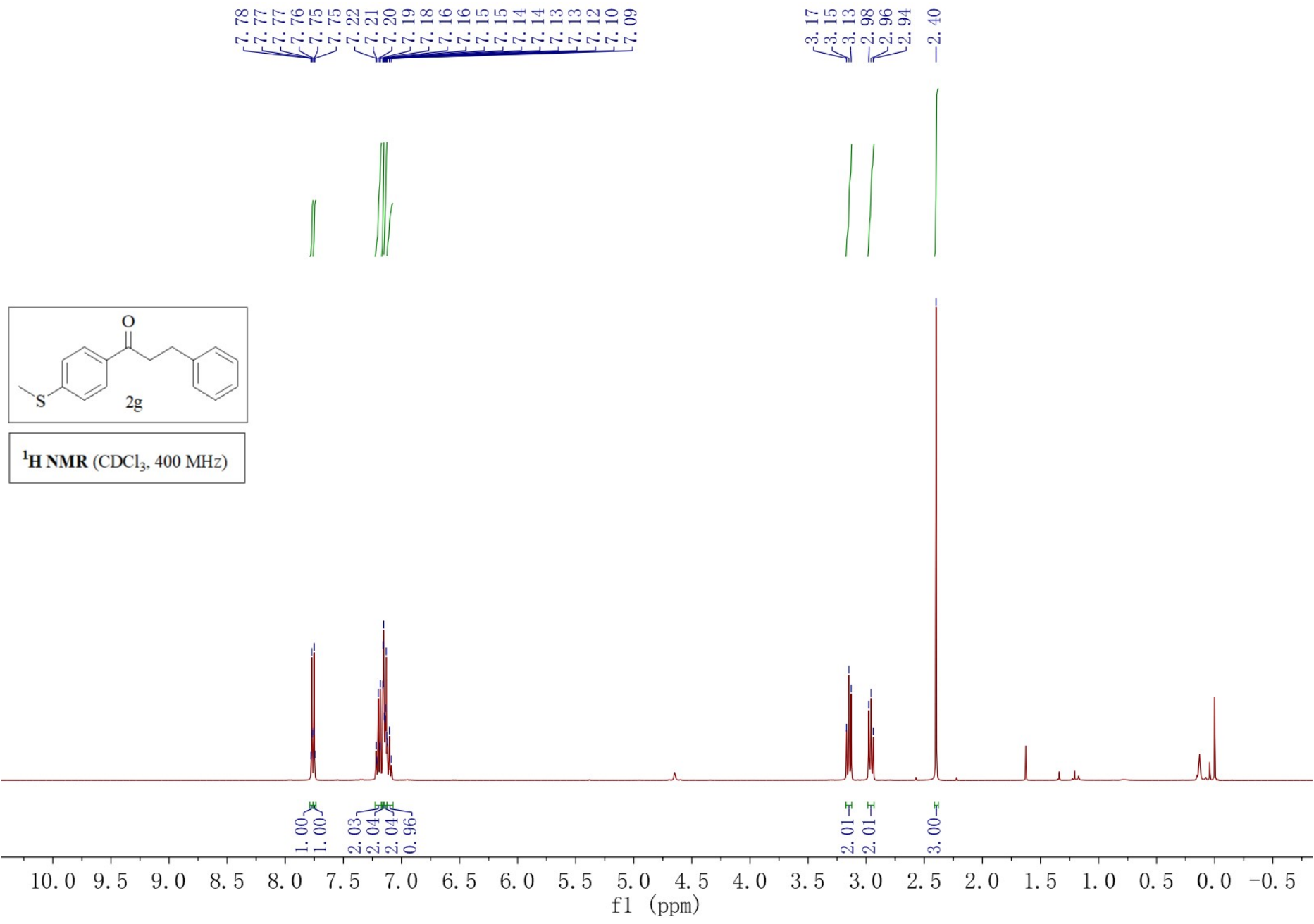


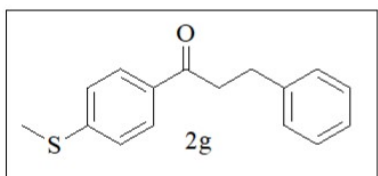
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



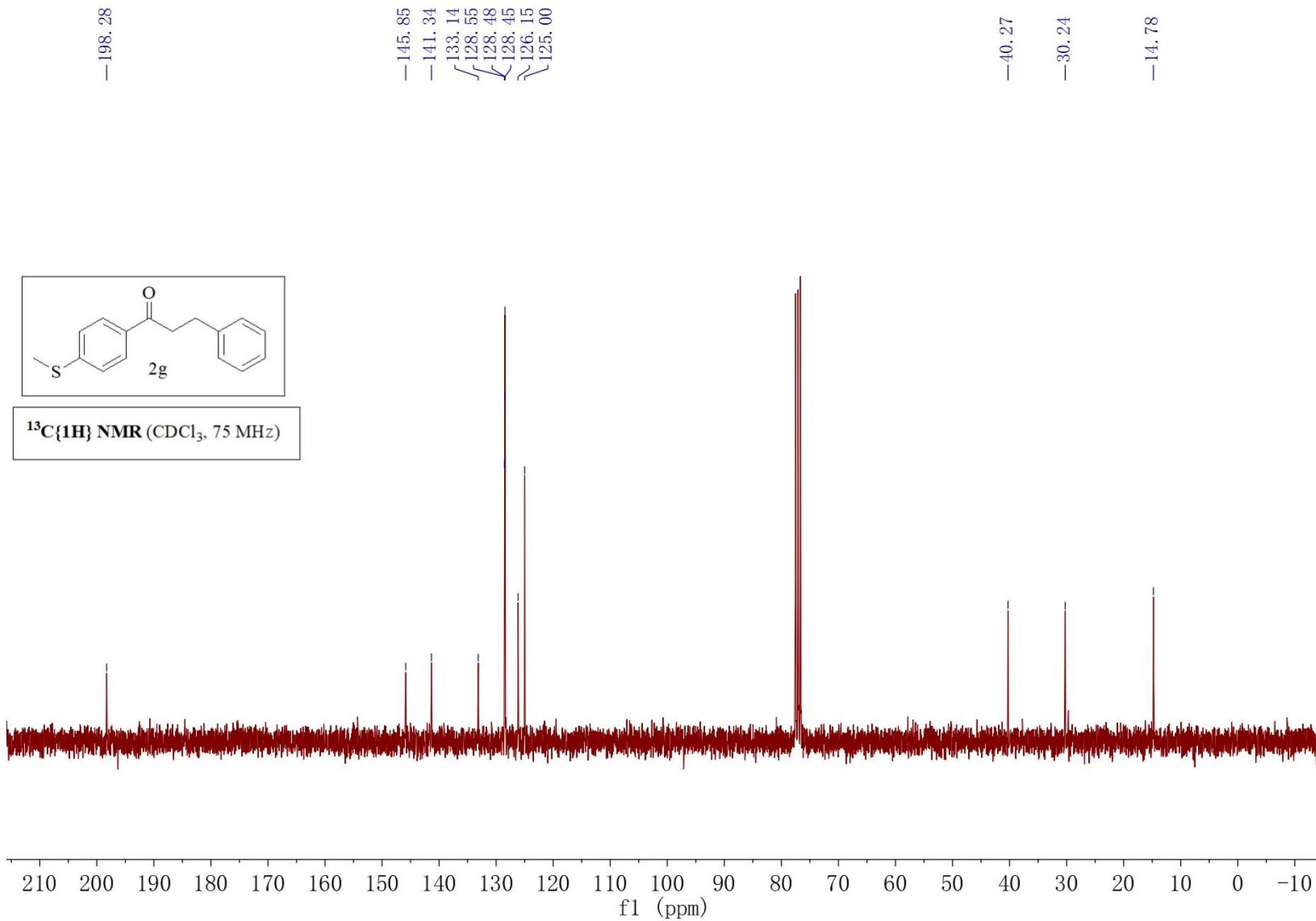


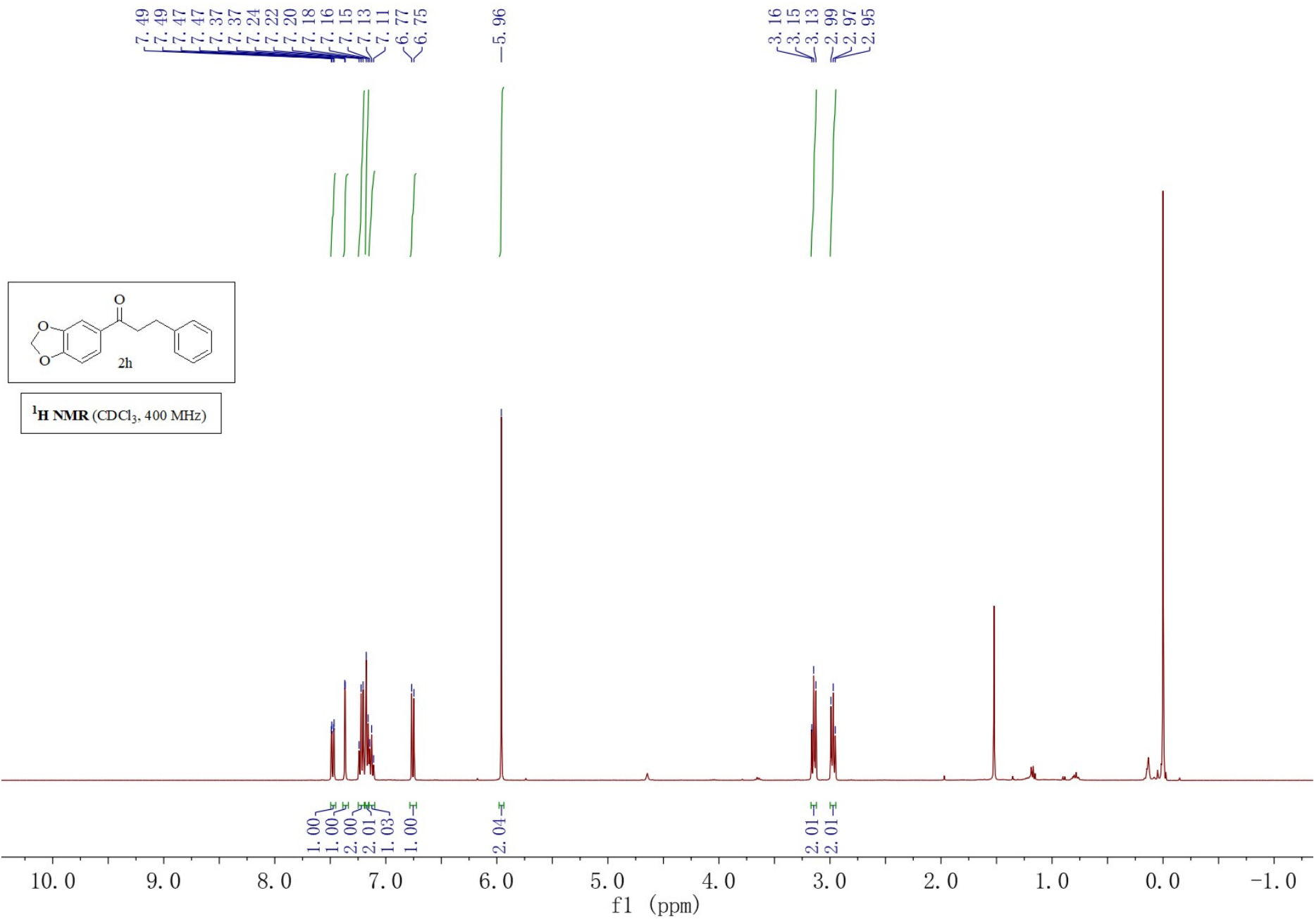


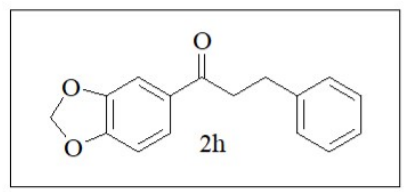




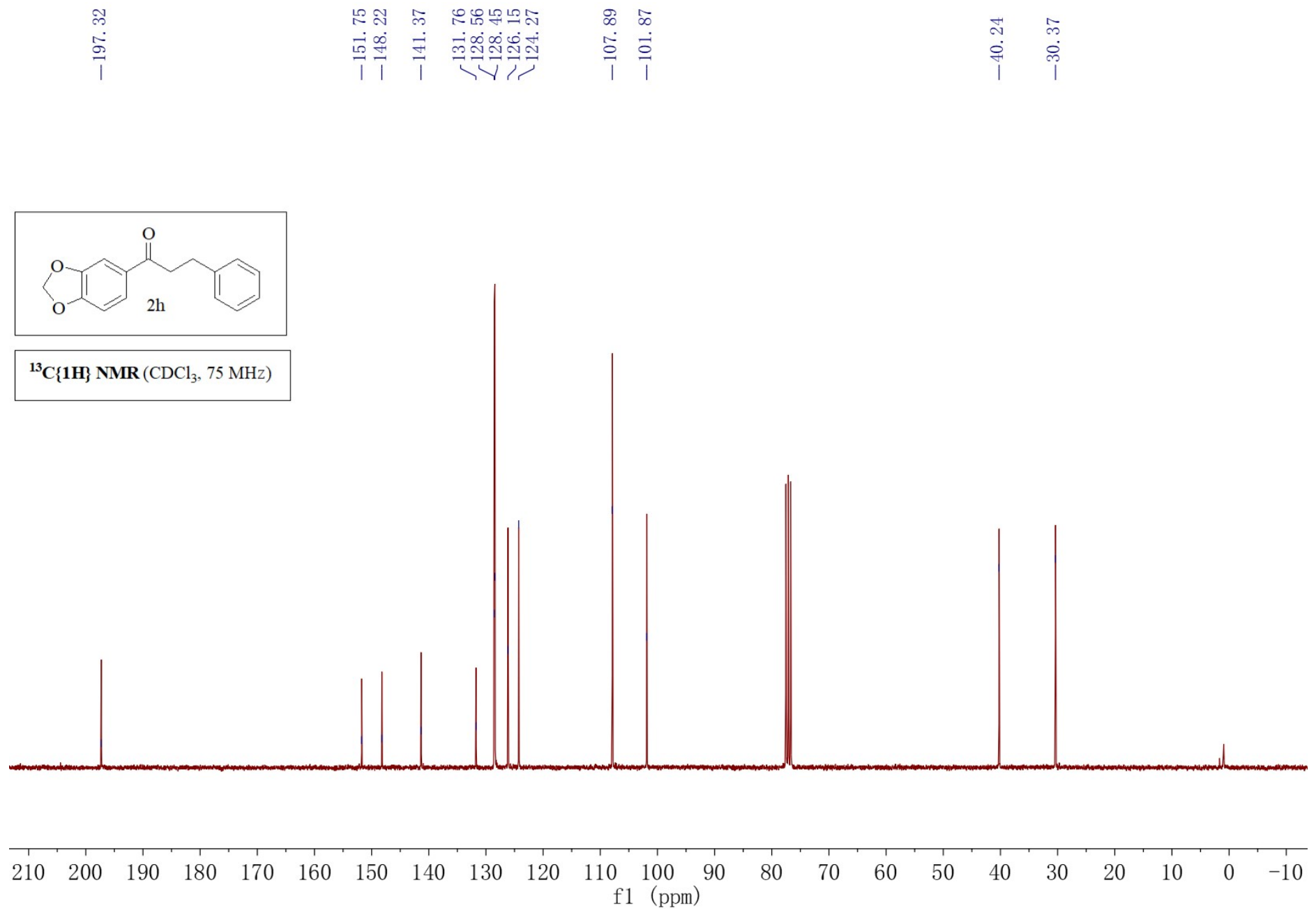
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

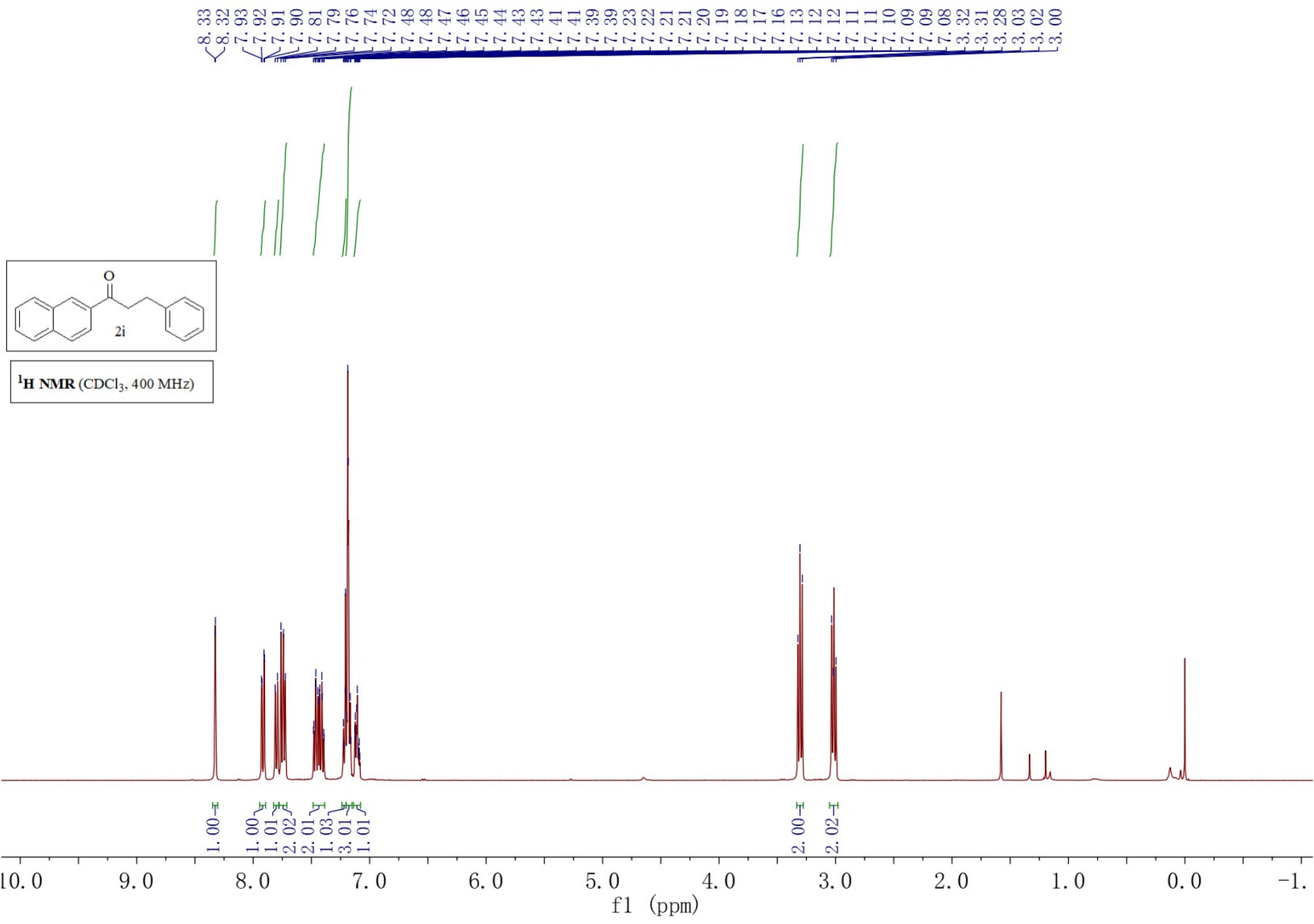


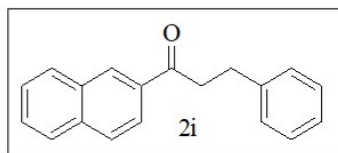




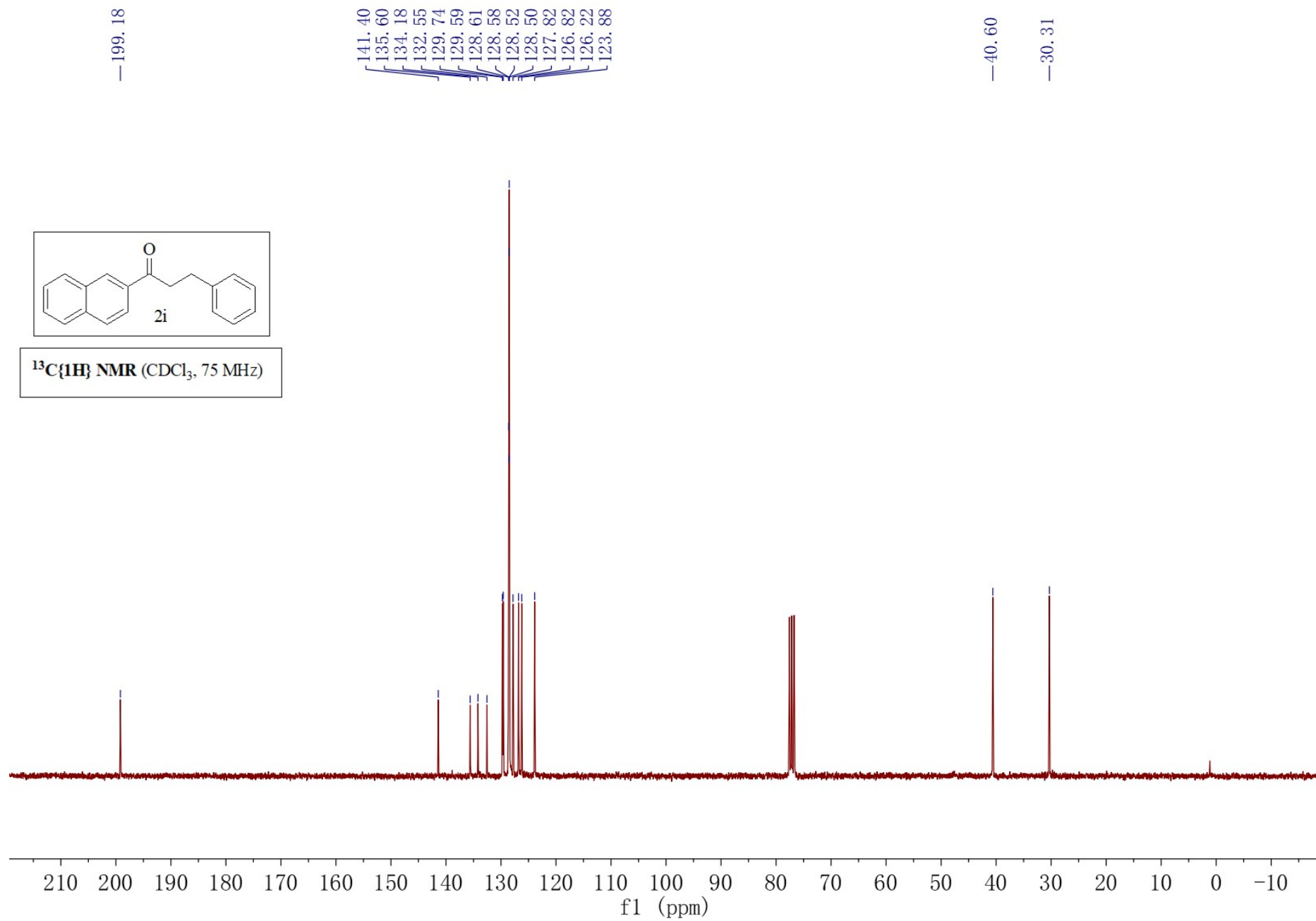
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

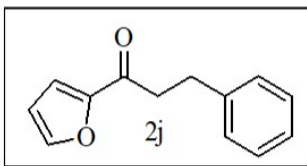




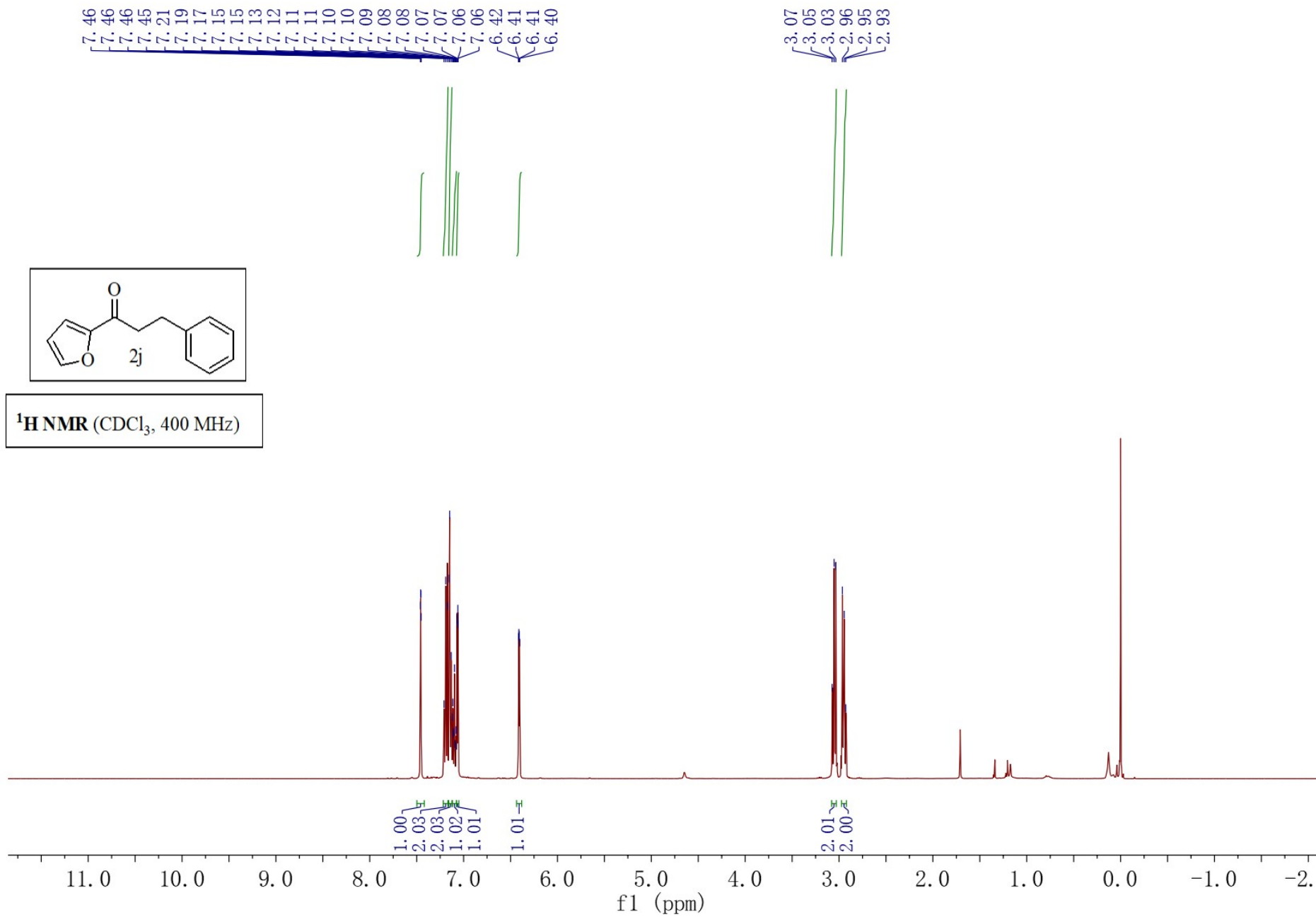


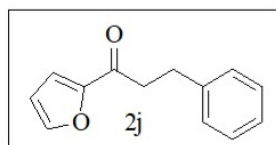
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



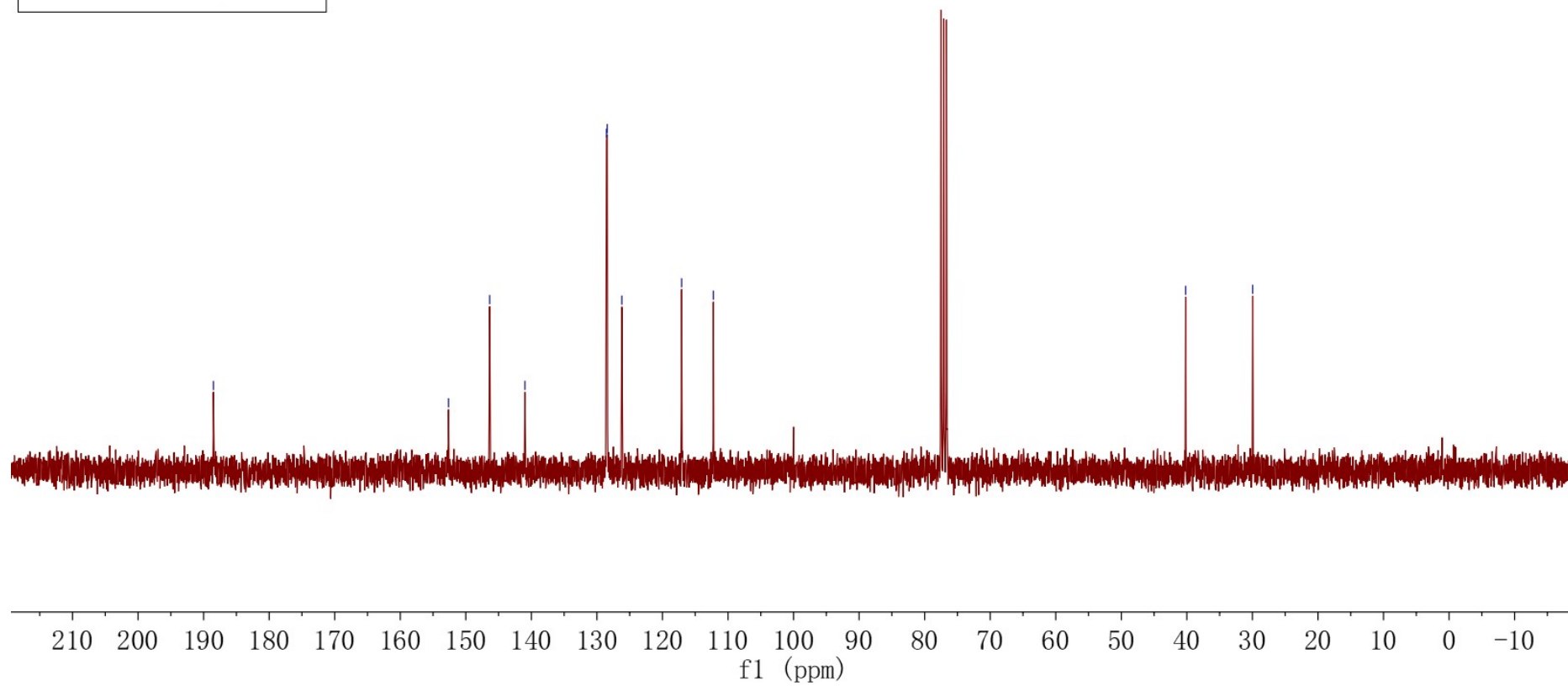


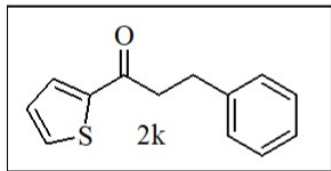
¹H NMR (CDCl₃, 400 MHz)



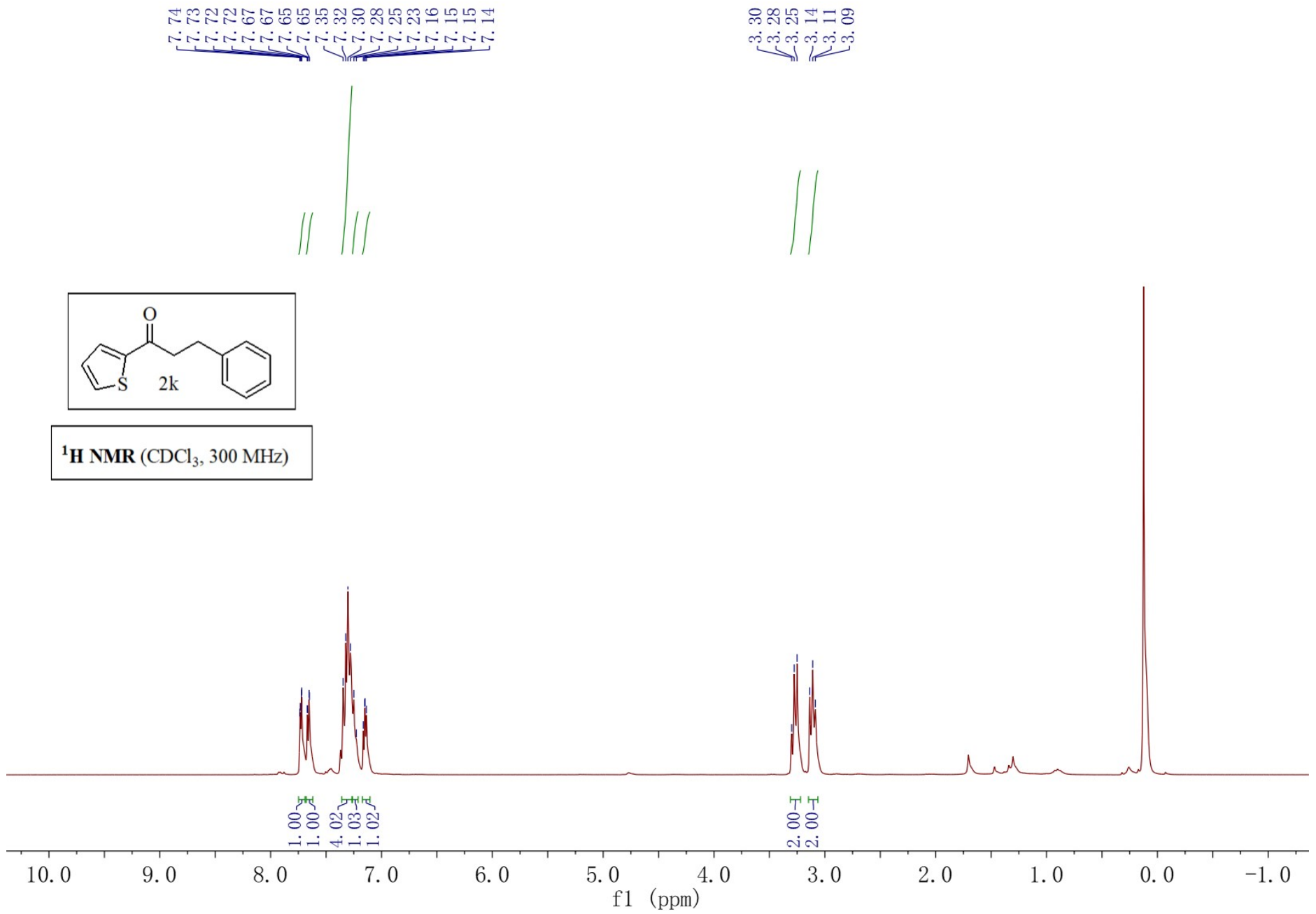


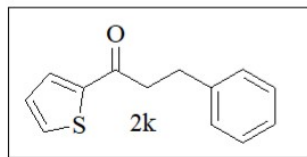
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



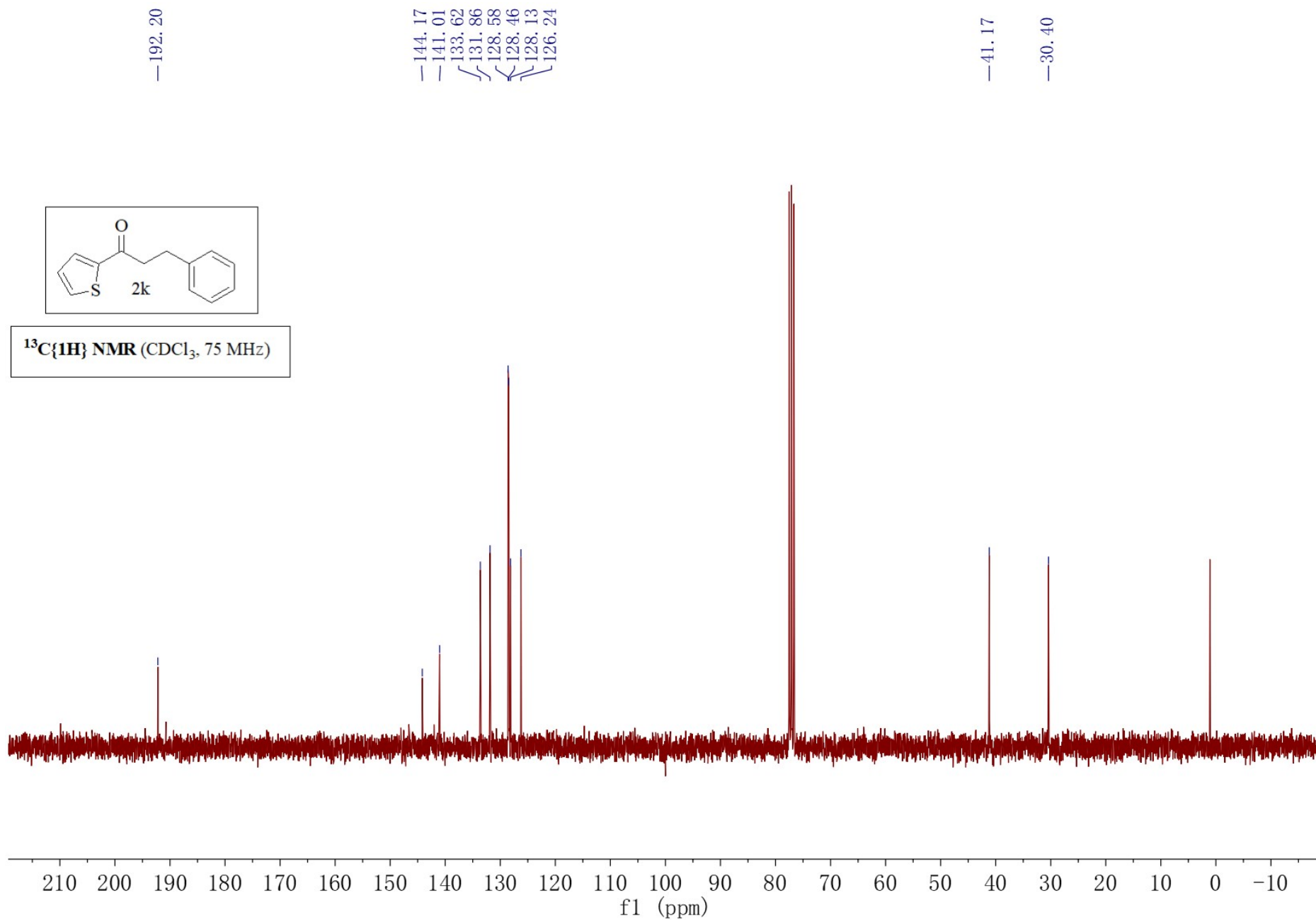


¹H NMR (CDCl₃, 300 MHz)



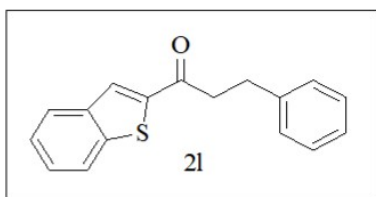


$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

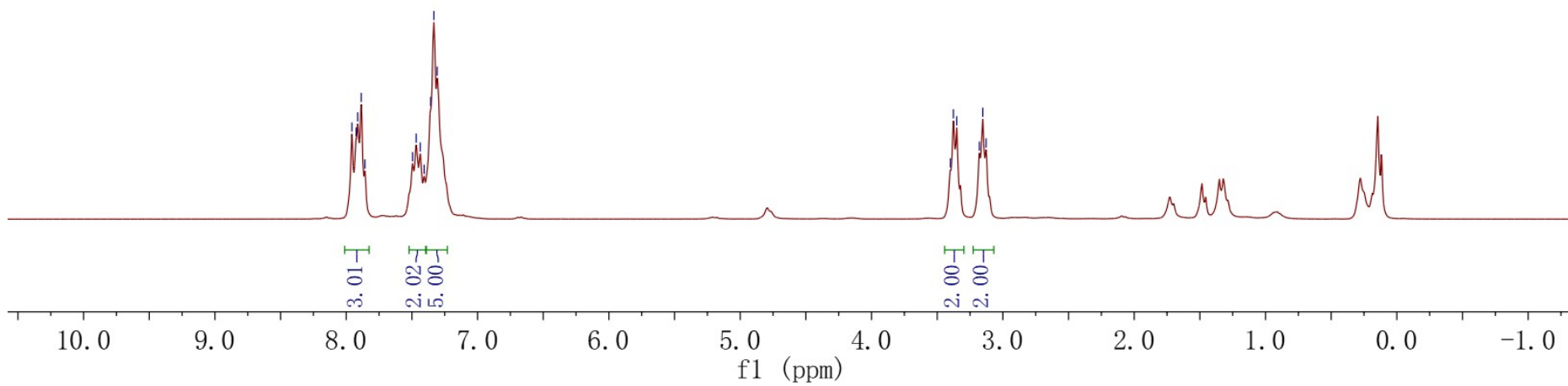


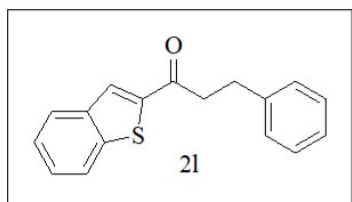
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7.33
7.31

3.40
3.38
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3.18
3.15
3.13

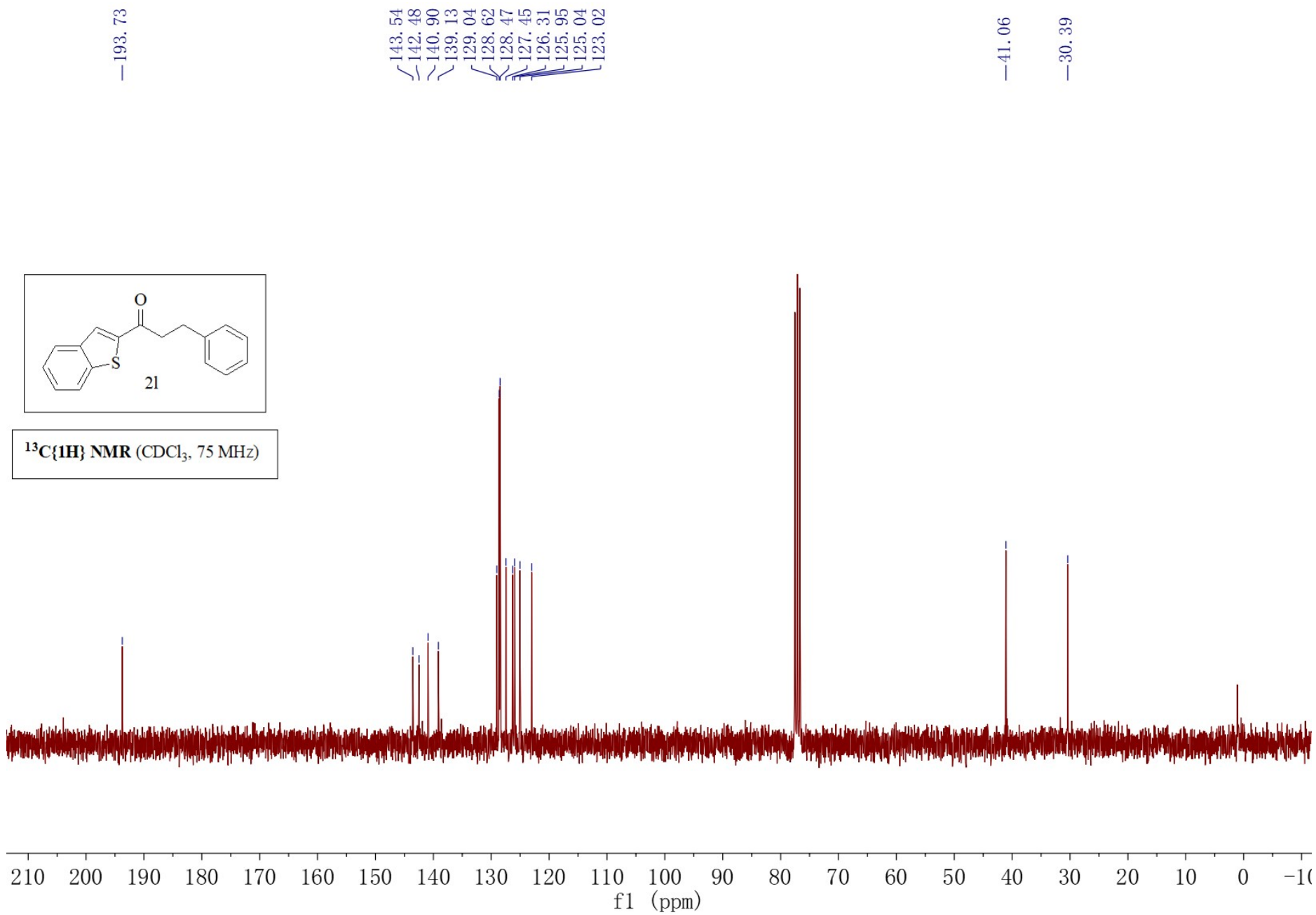


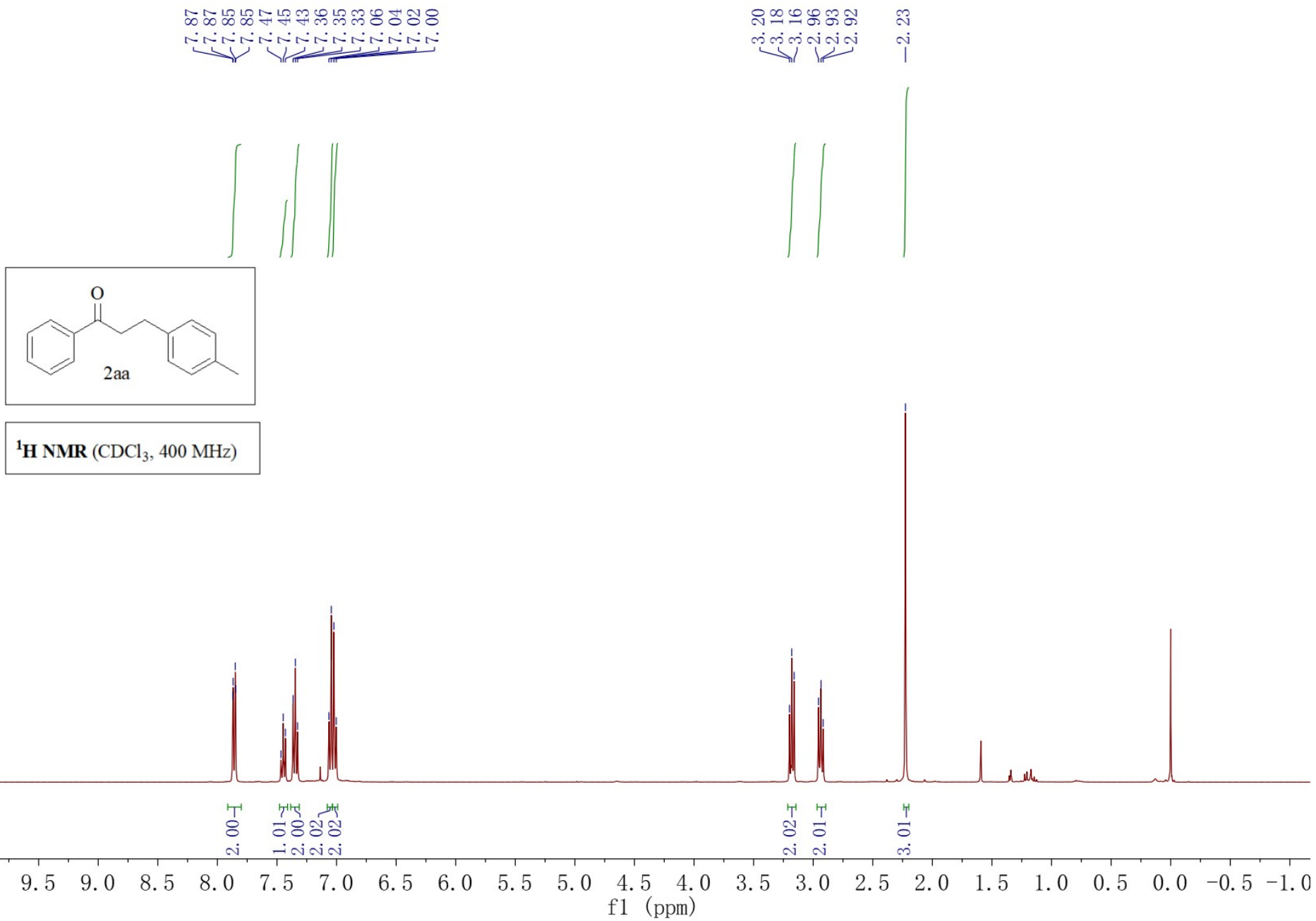
¹H NMR (CDCl₃, 300 MHz)

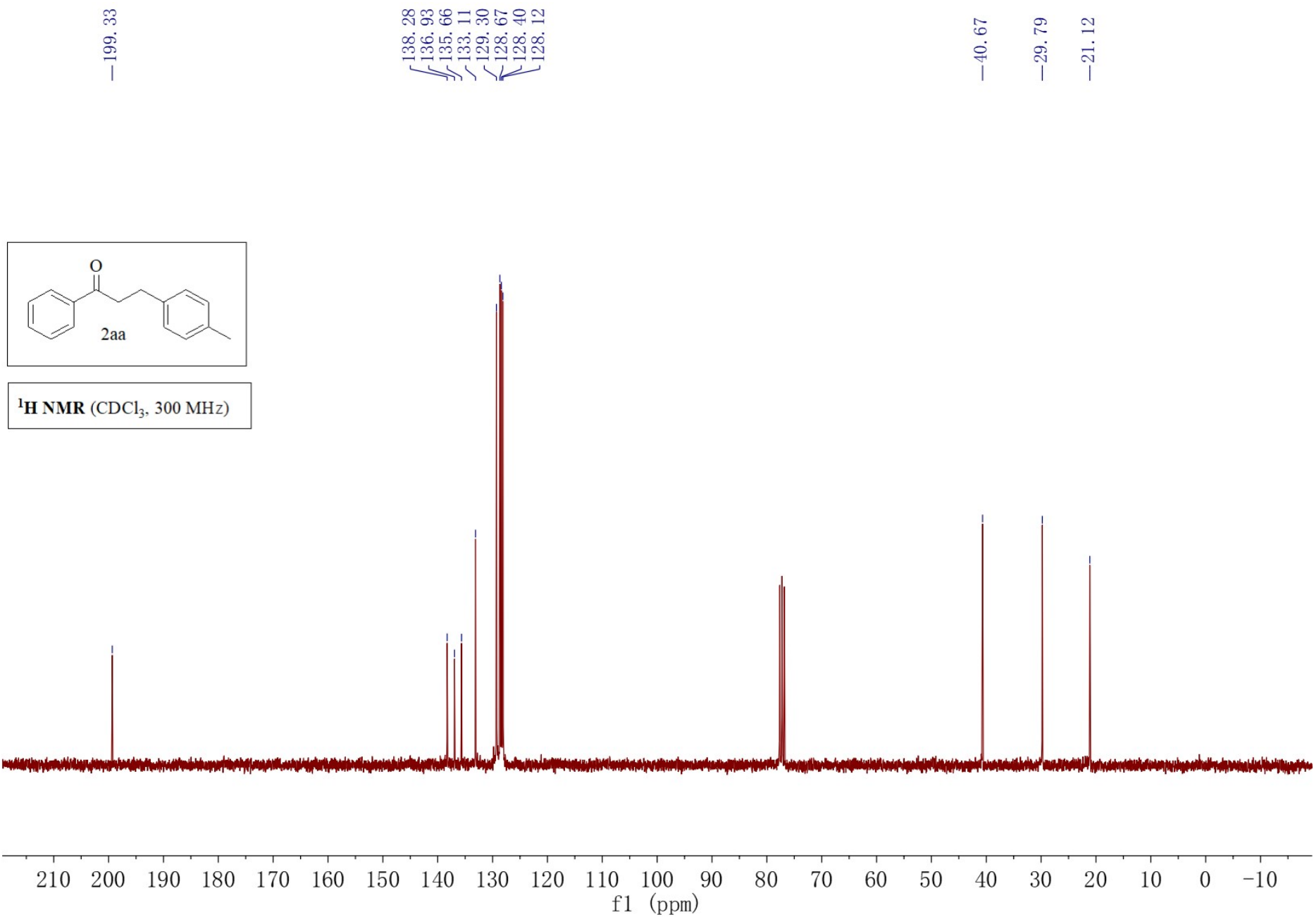


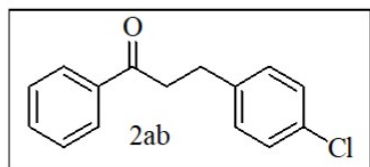


$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

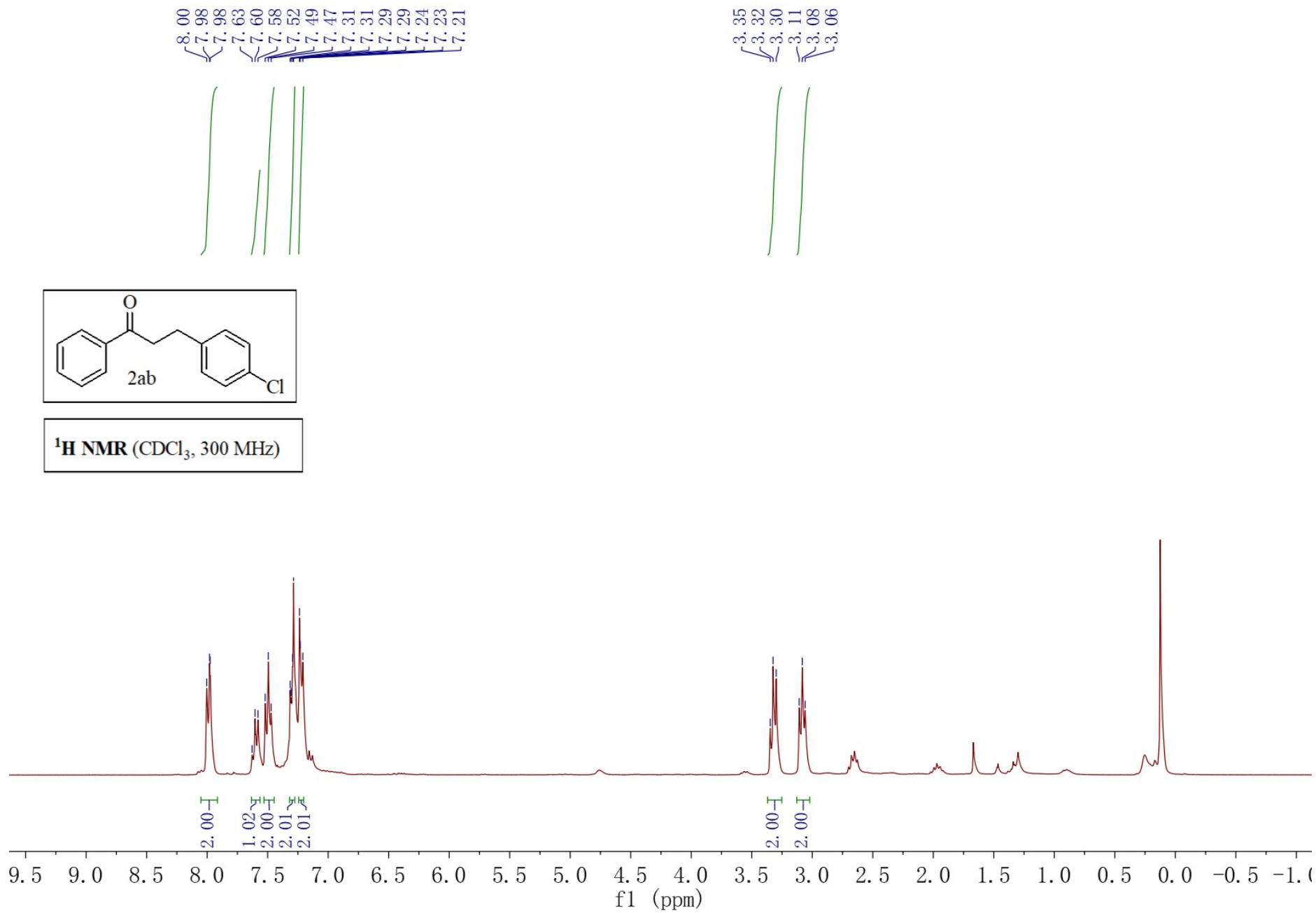


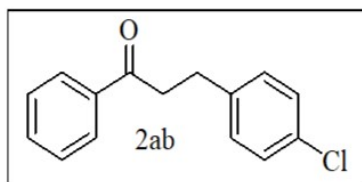




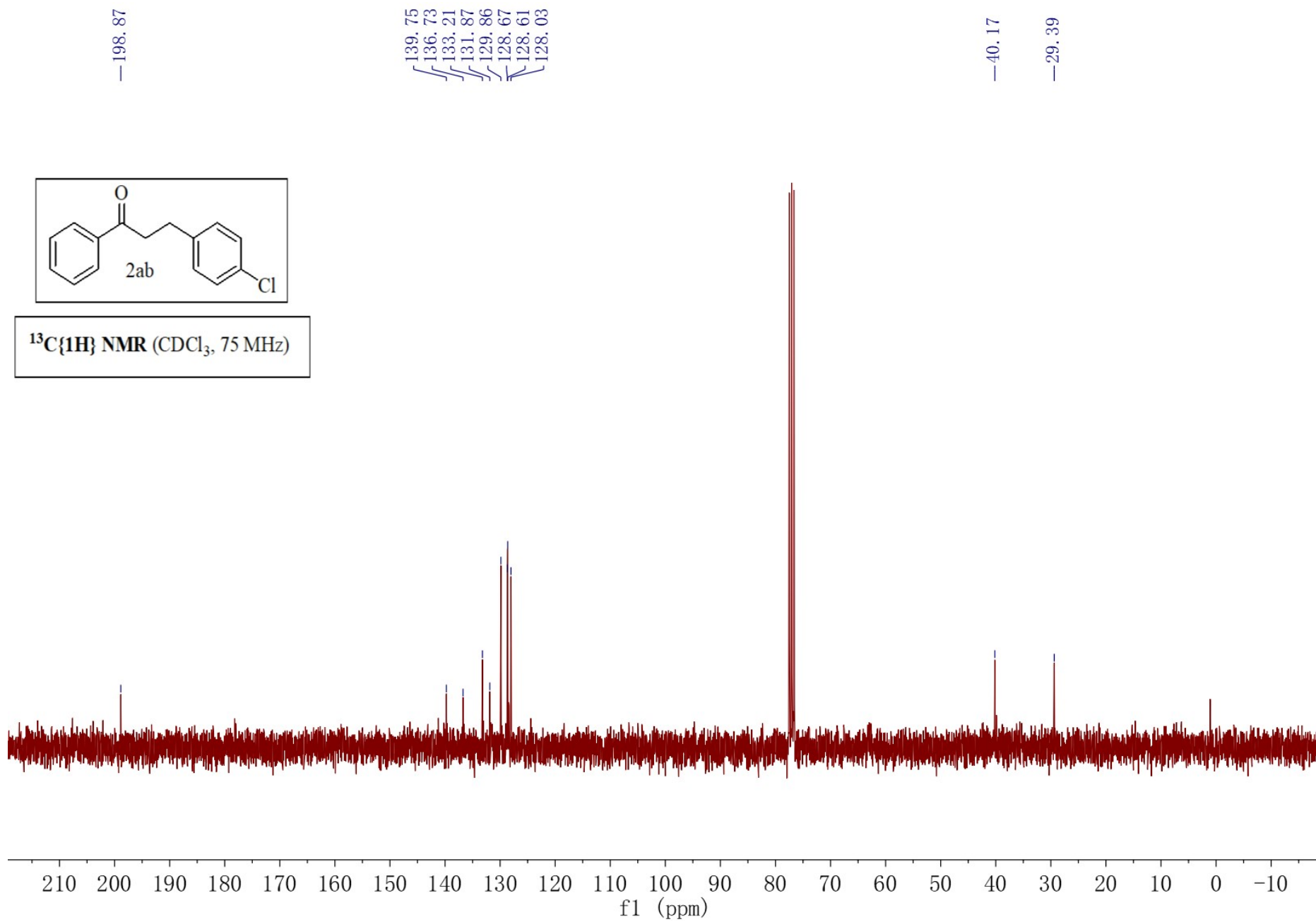


¹H NMR (CDCl₃, 300 MHz)



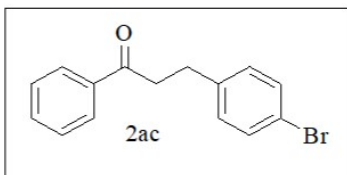


$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

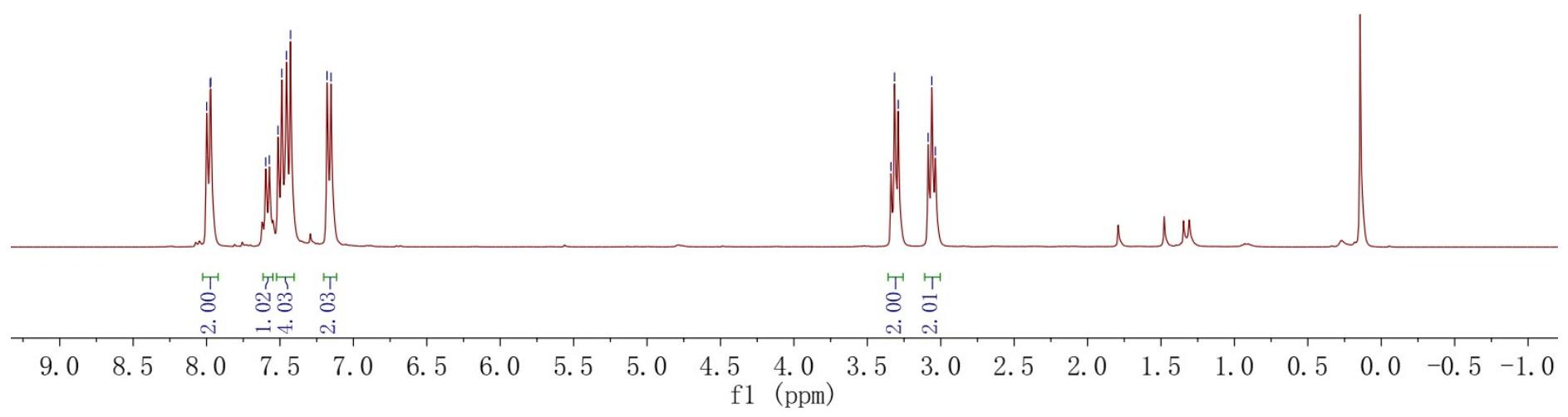


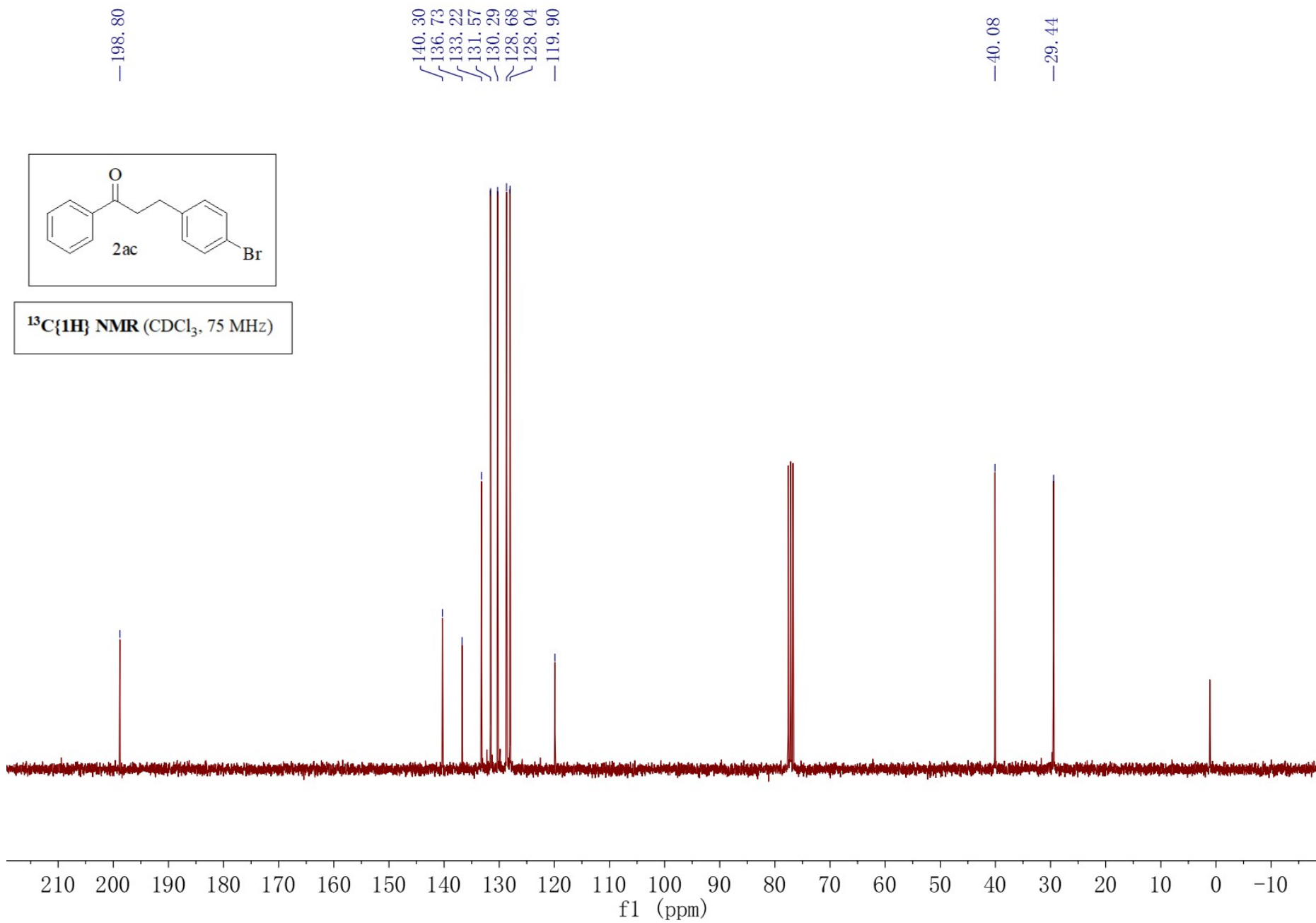
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7.51
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7.18
7.15

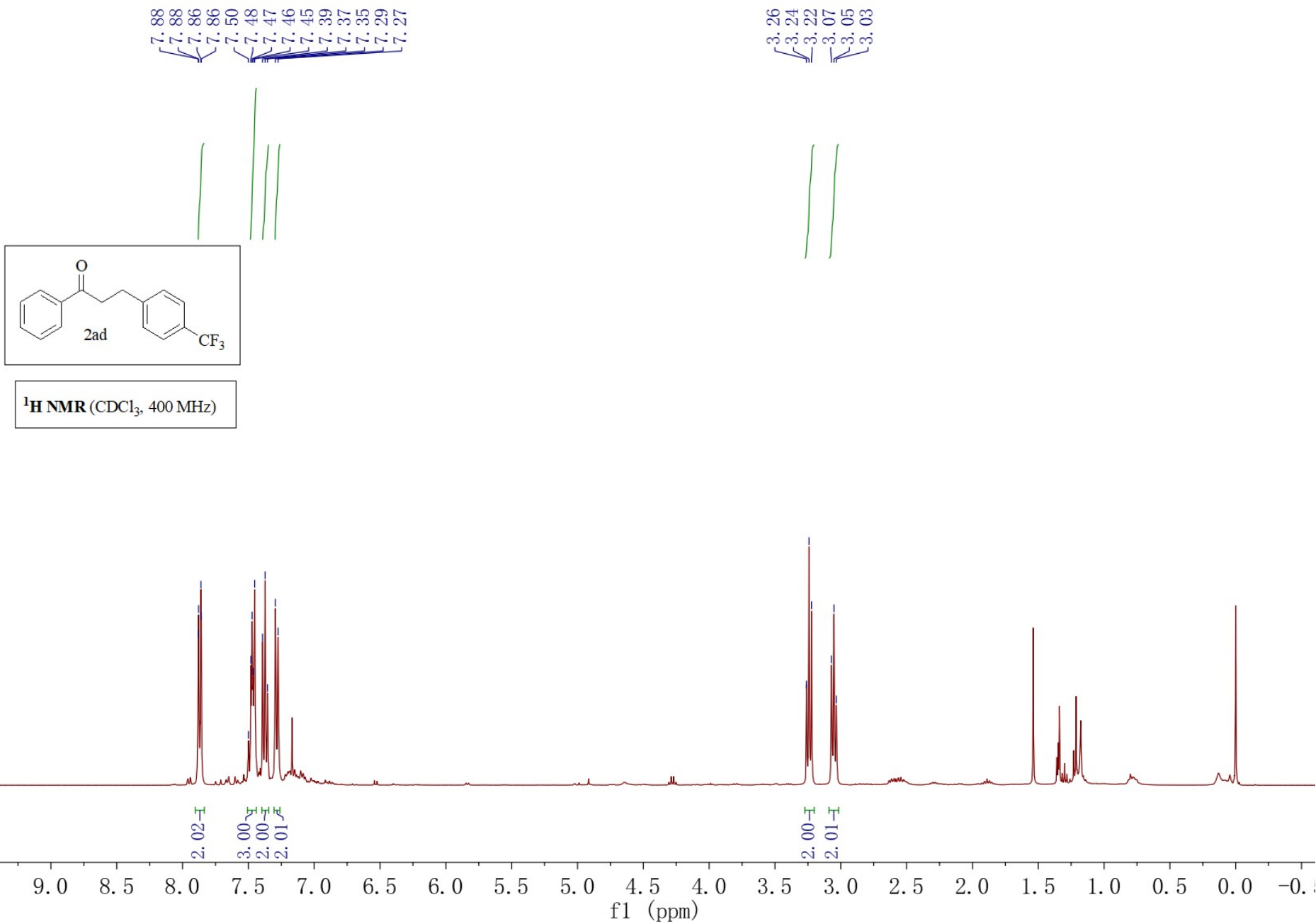
3.34
3.31
3.29
3.09
3.06
3.04



¹H NMR (CDCl₃, 300 MHz)







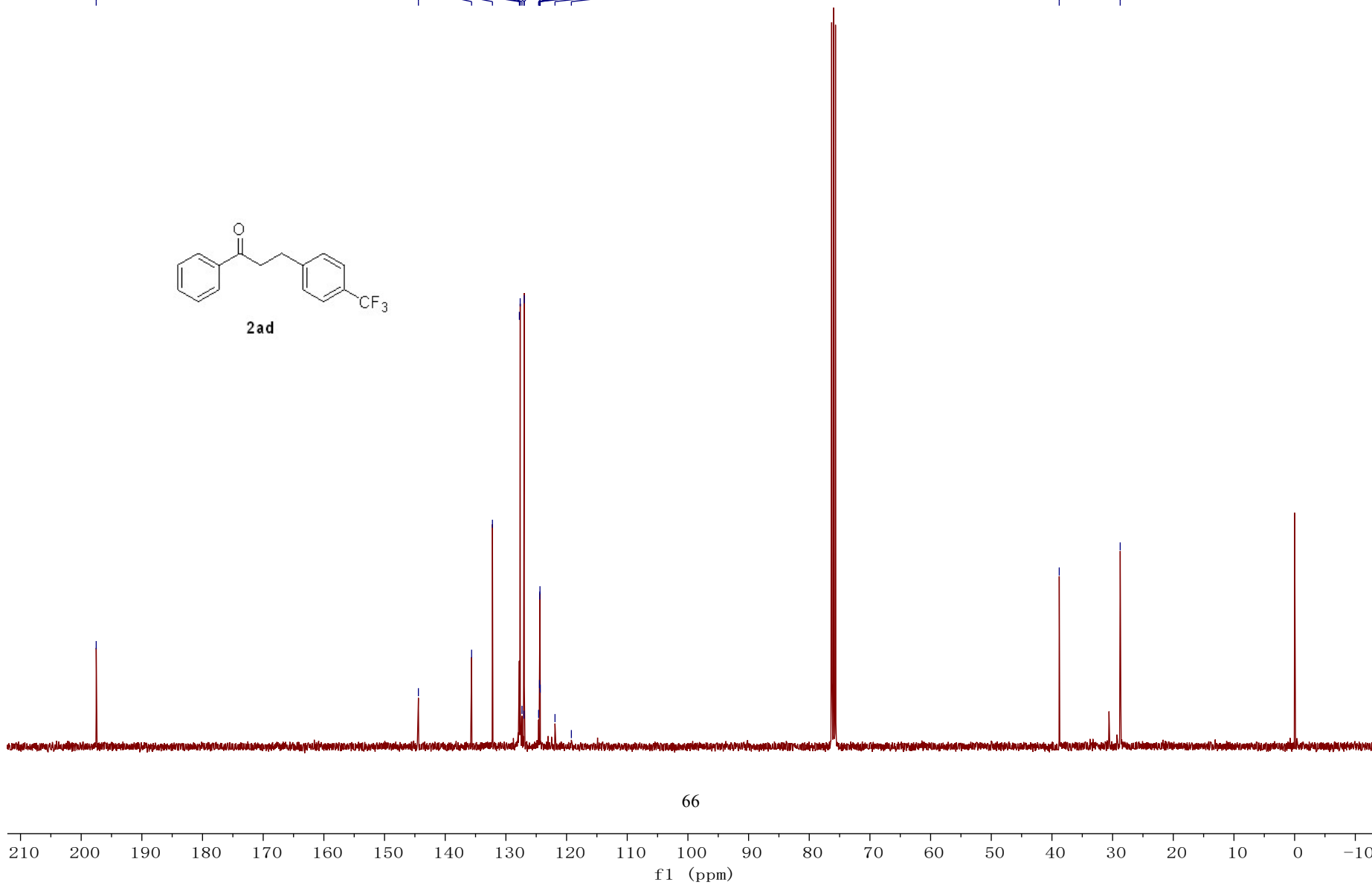
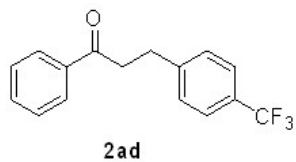
20240523 3.1.fid

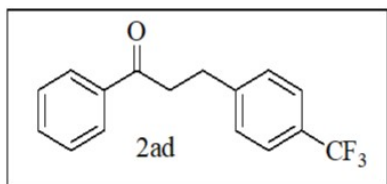
197.64

144.42
135.66
132.21
127.78
127.64
127.34
127.02
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124.61
124.46
124.42
124.38
124.34
121.90
119.21

38.80

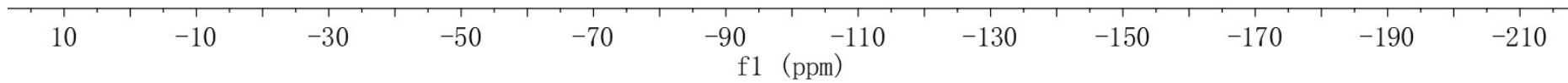
28.75

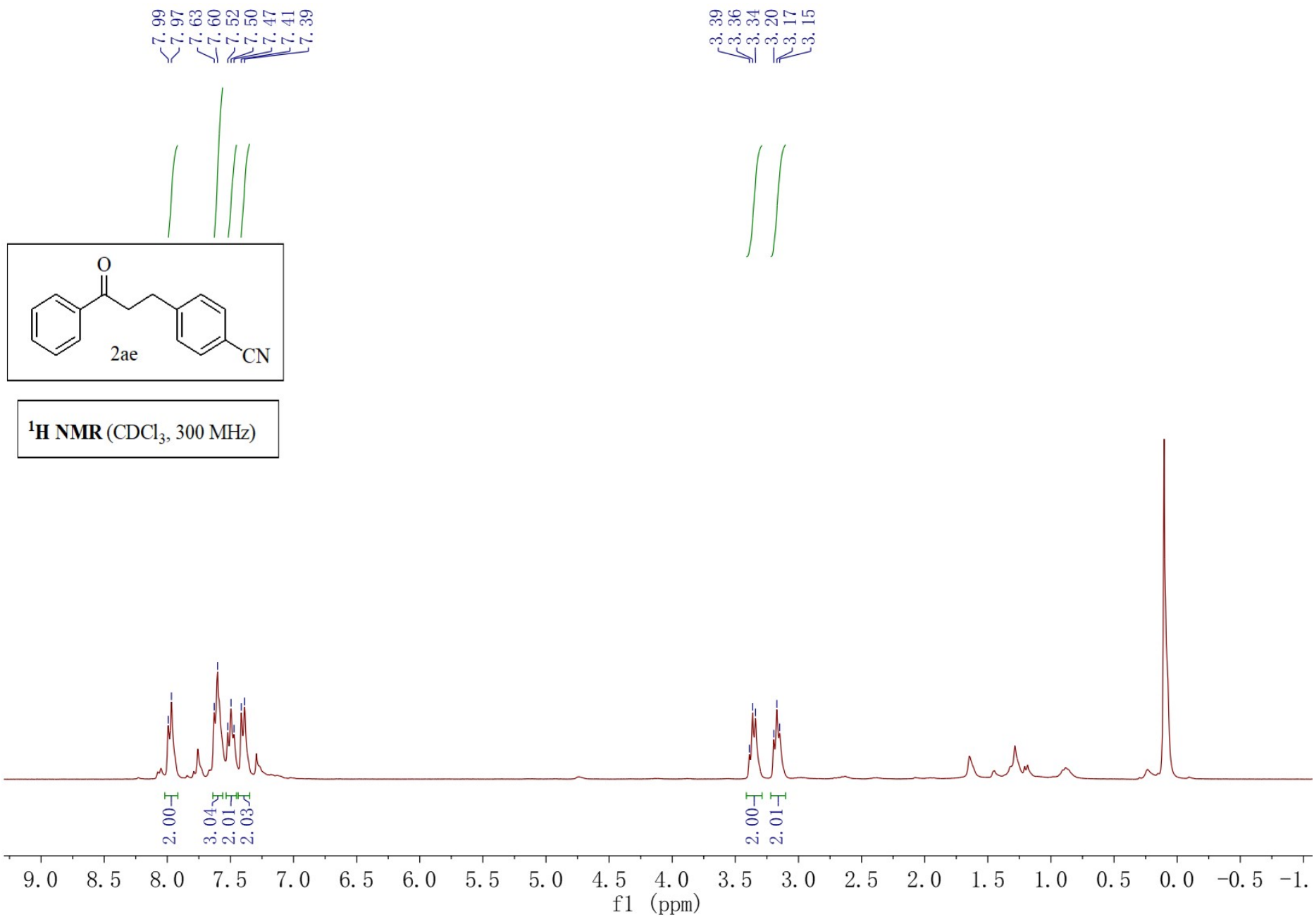


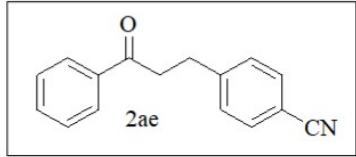


¹⁹F NMR (CDCl₃, 282 MHz)

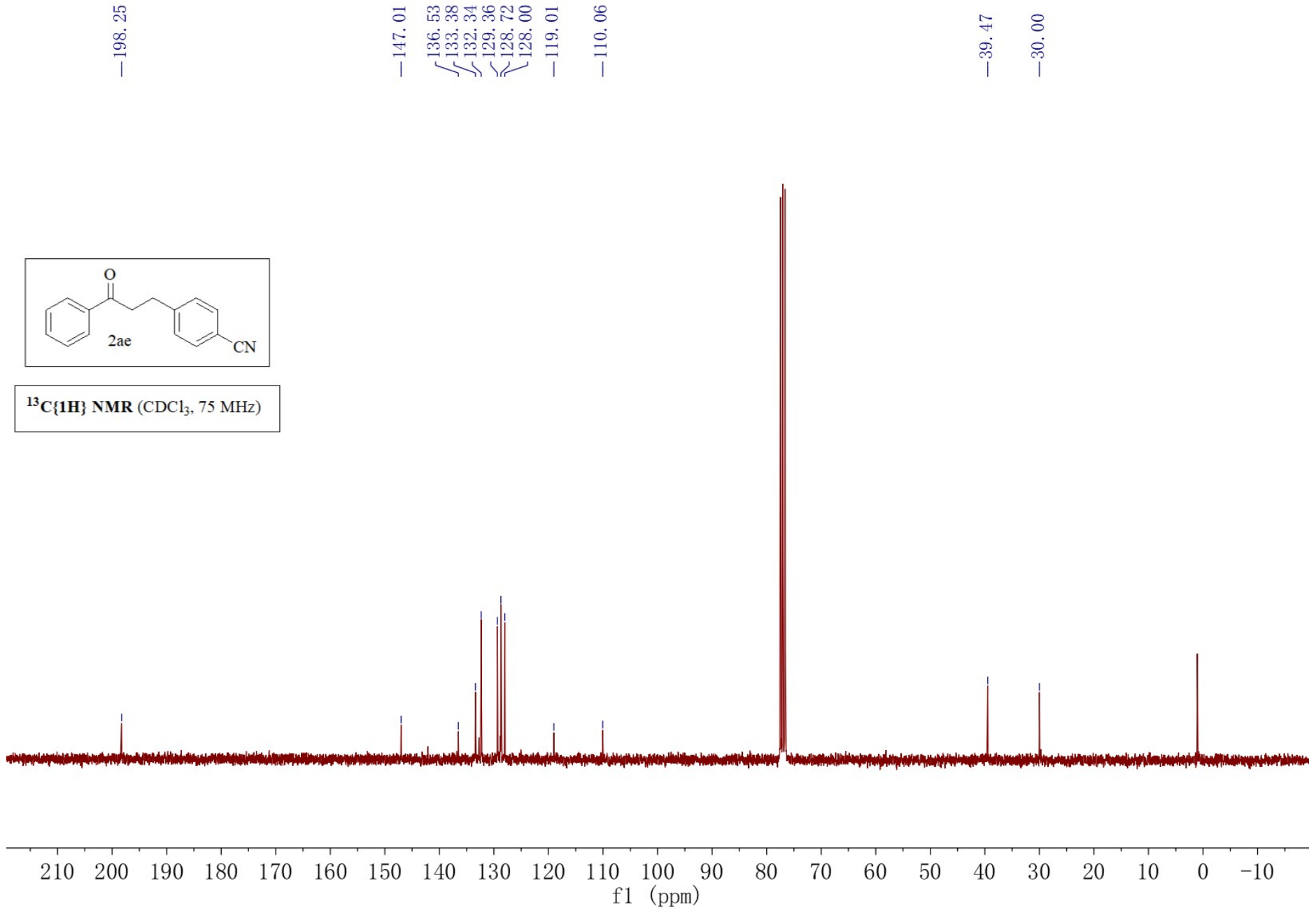
-62.33

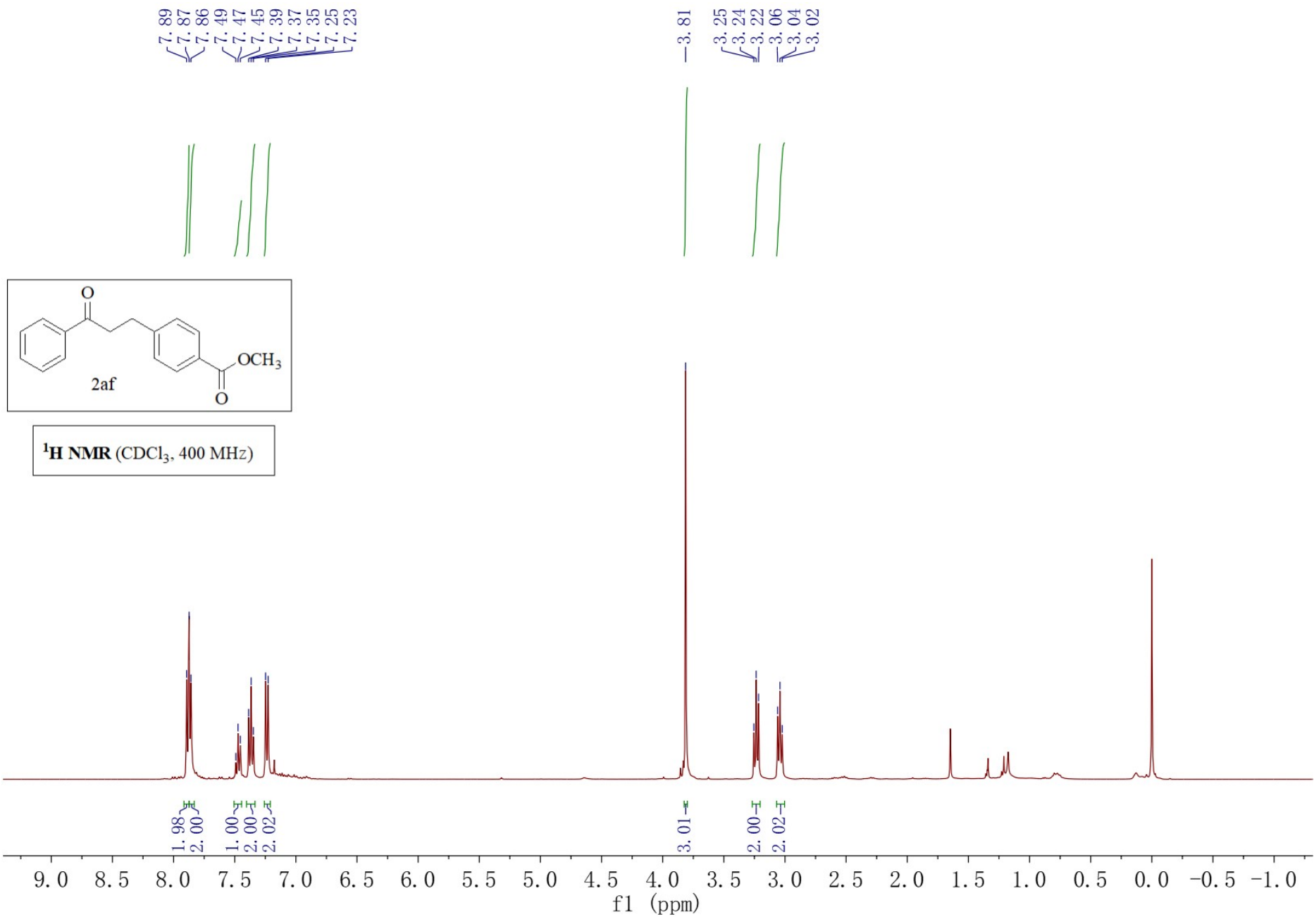


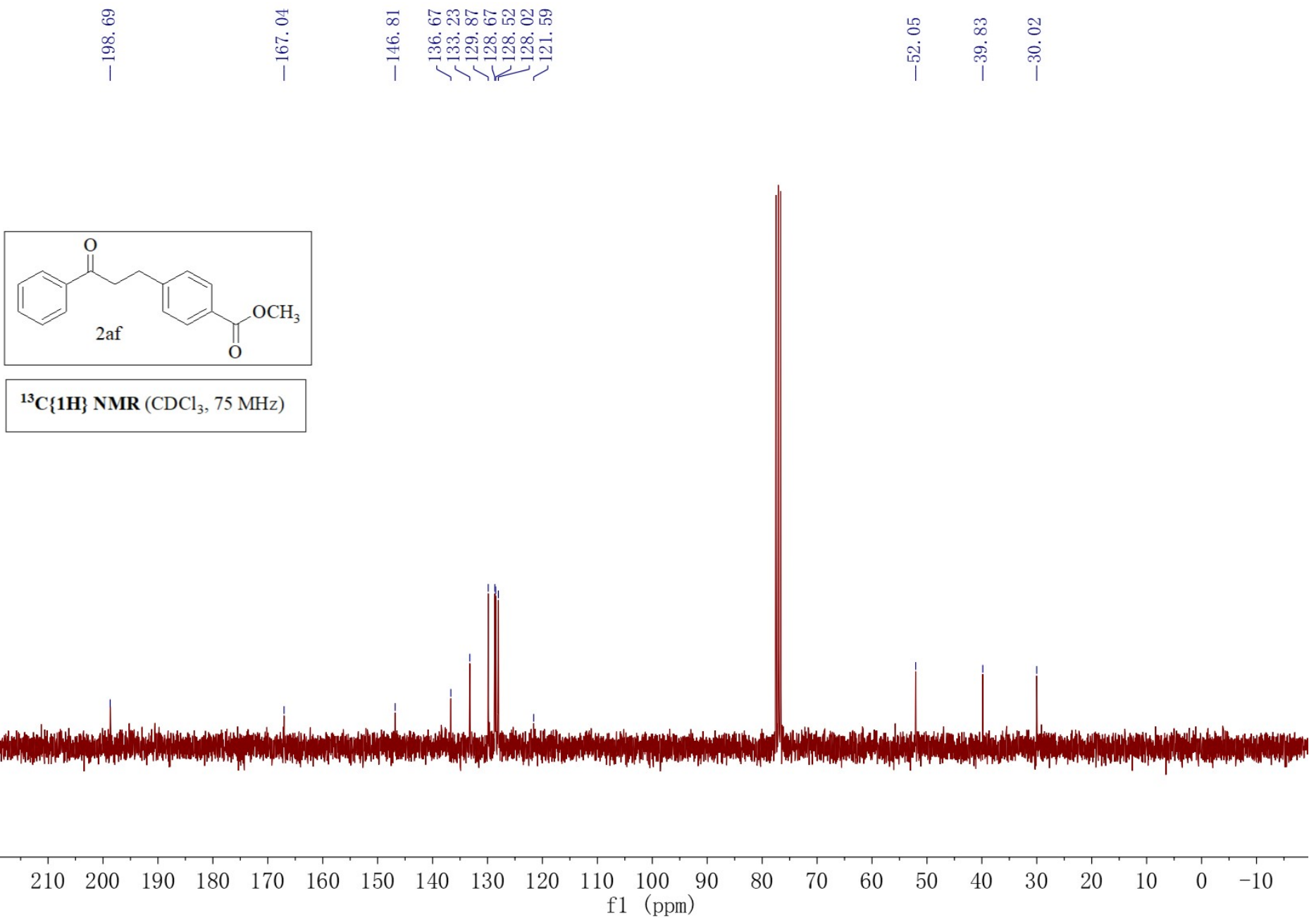


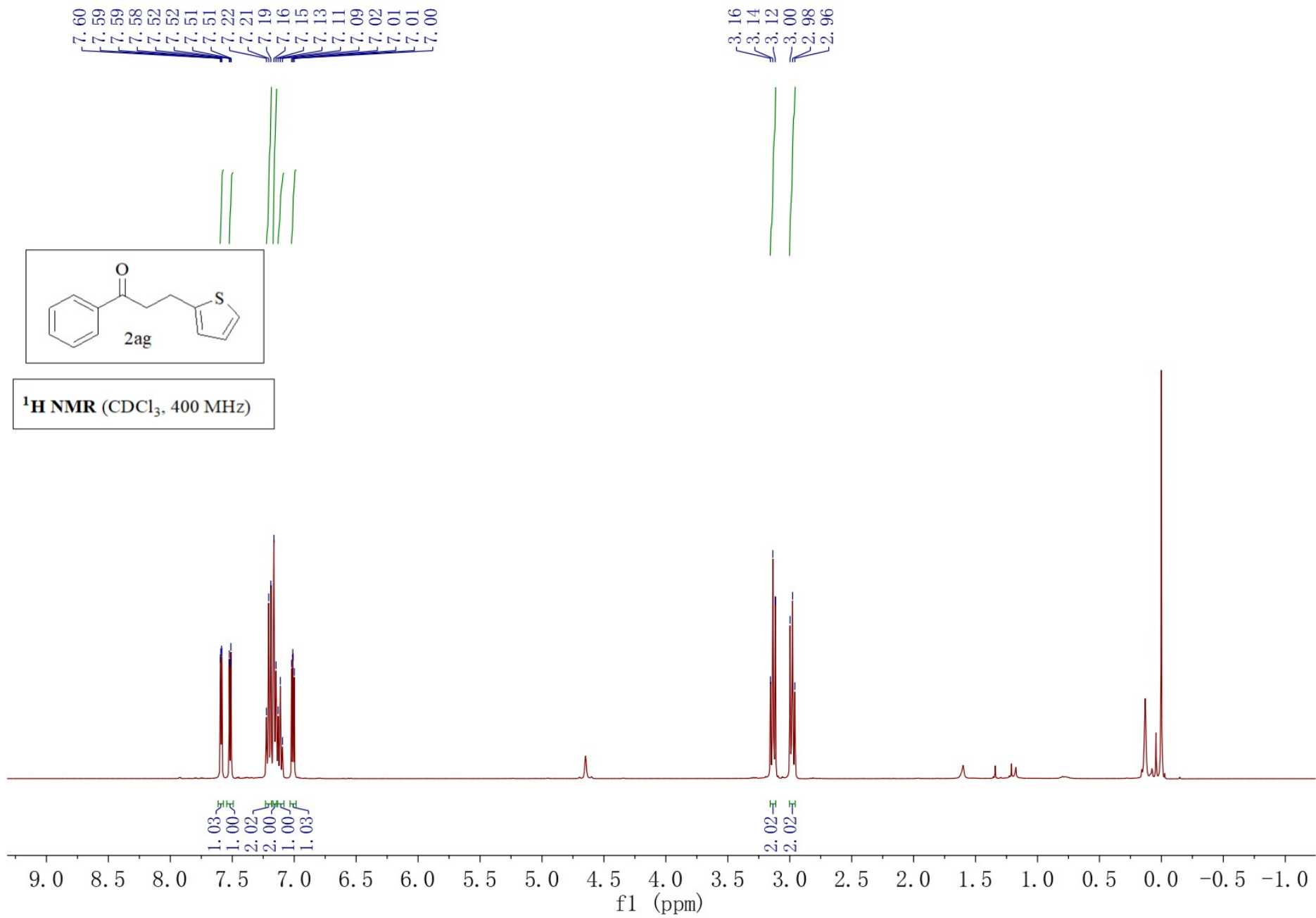


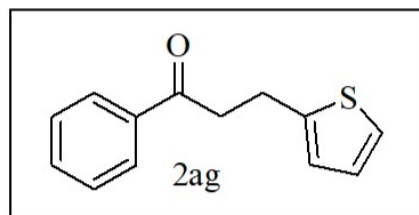
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



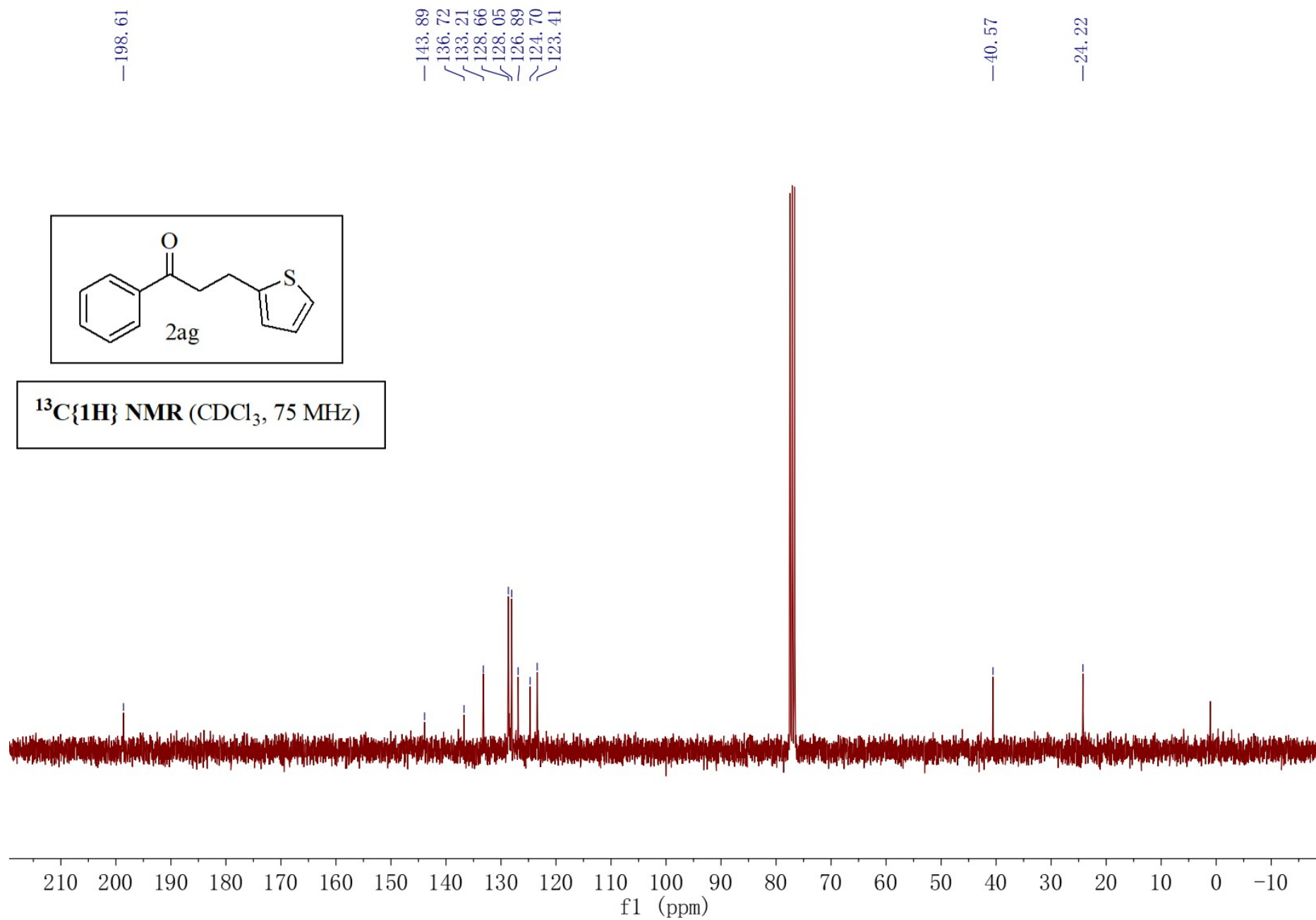


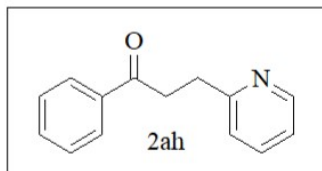




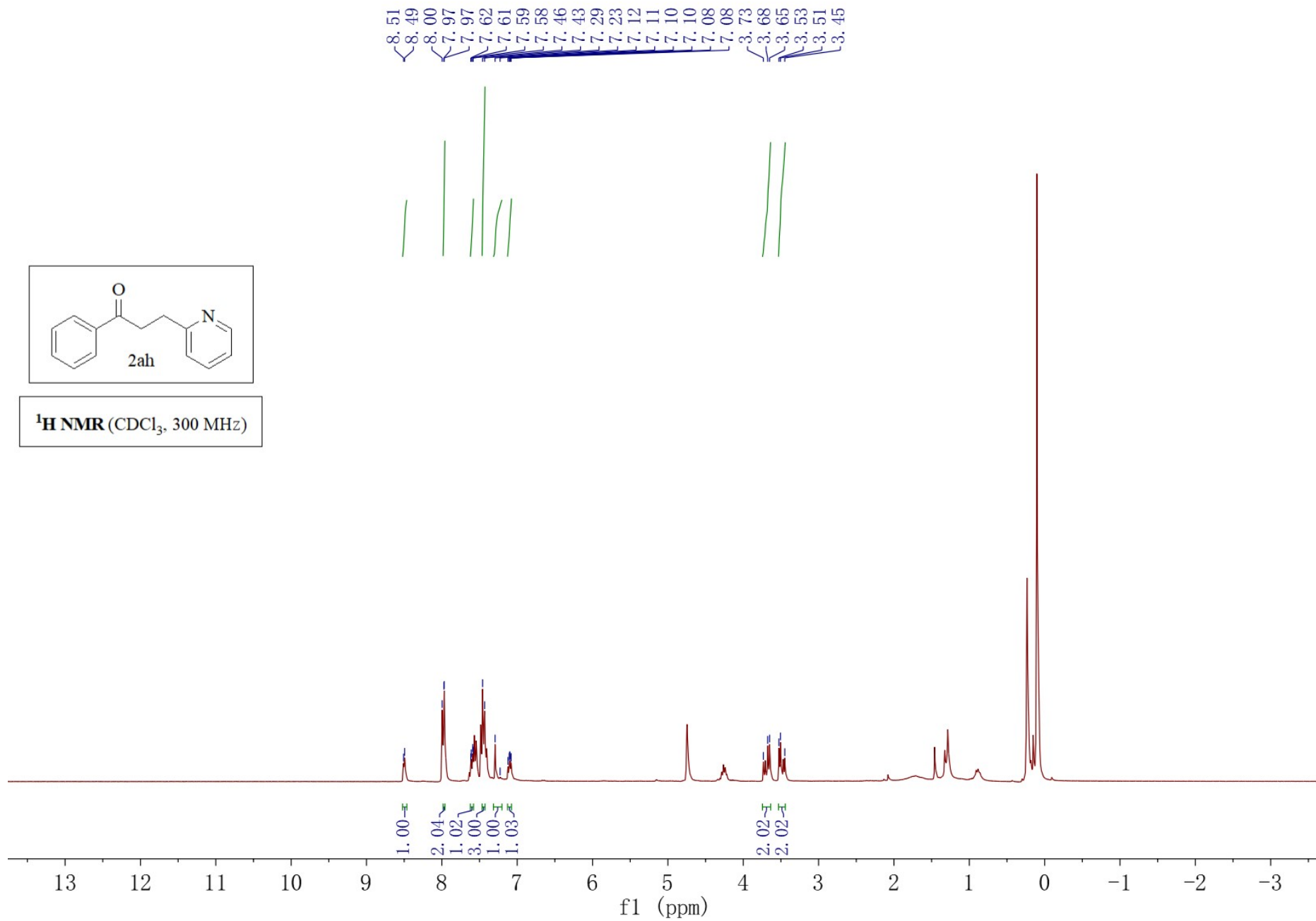


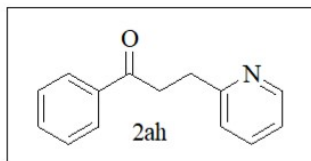
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



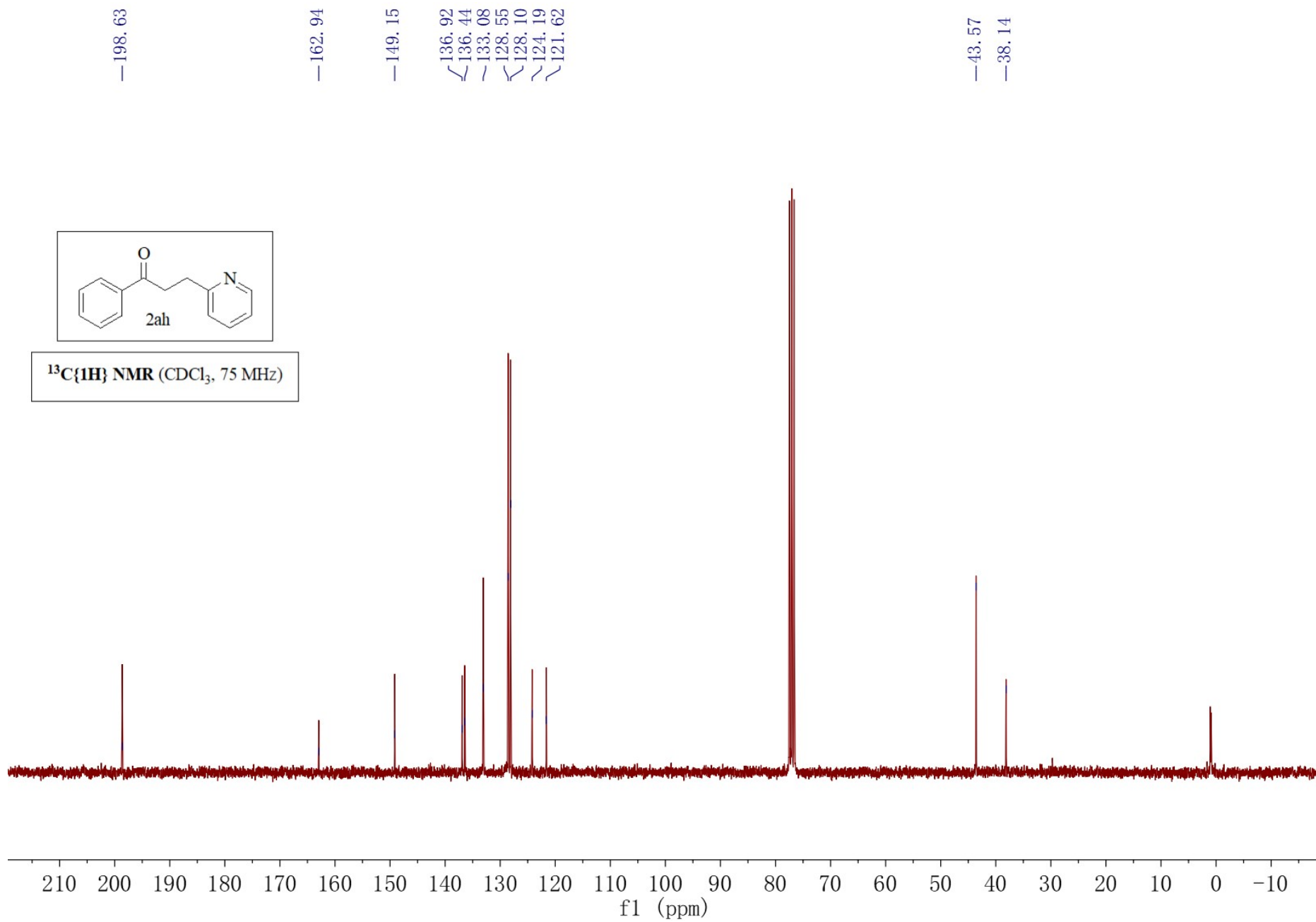


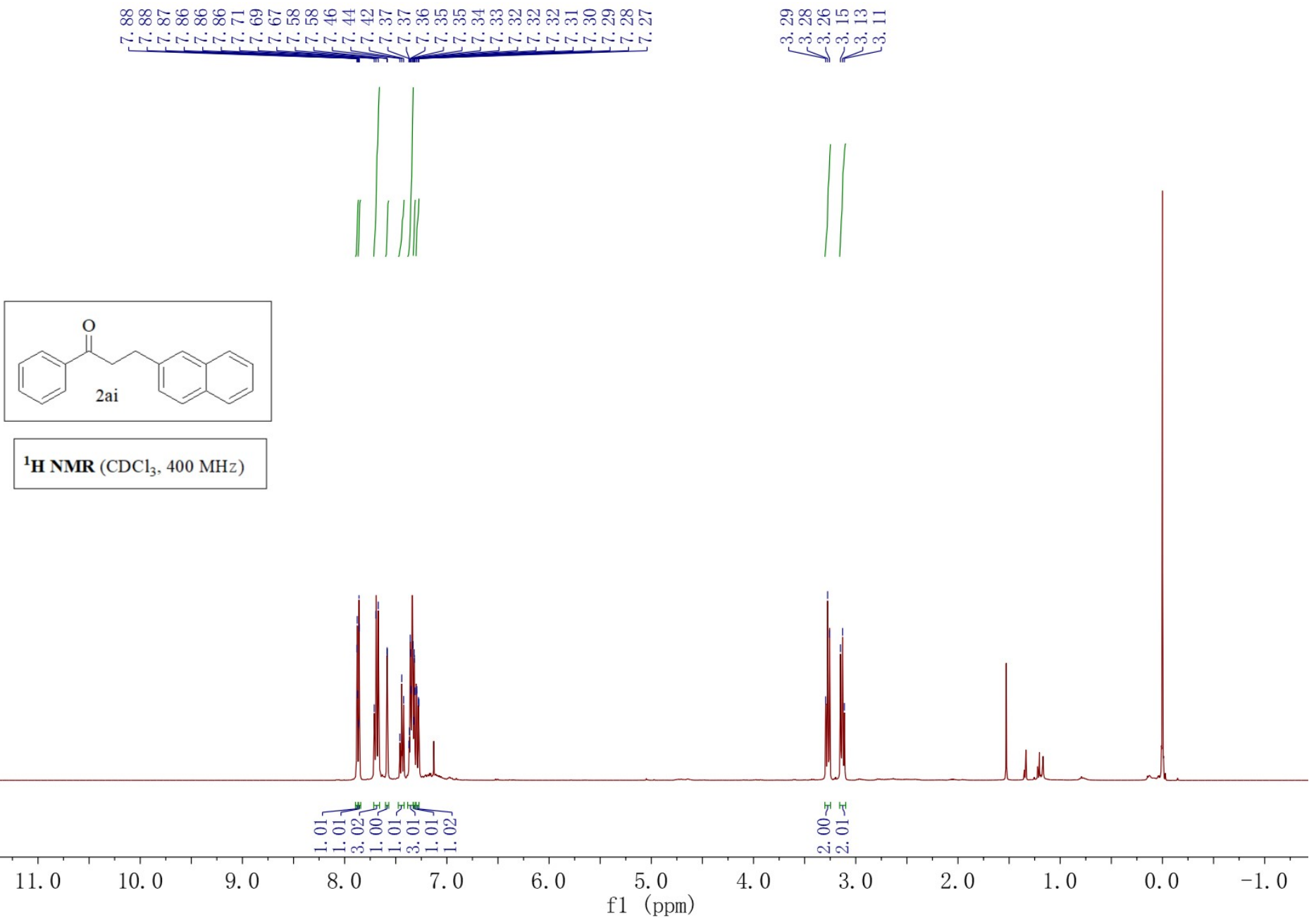
¹H NMR (CDCl₃, 300 MHz)

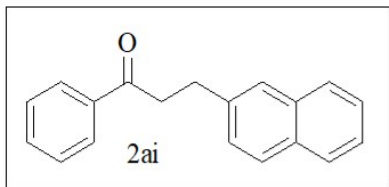




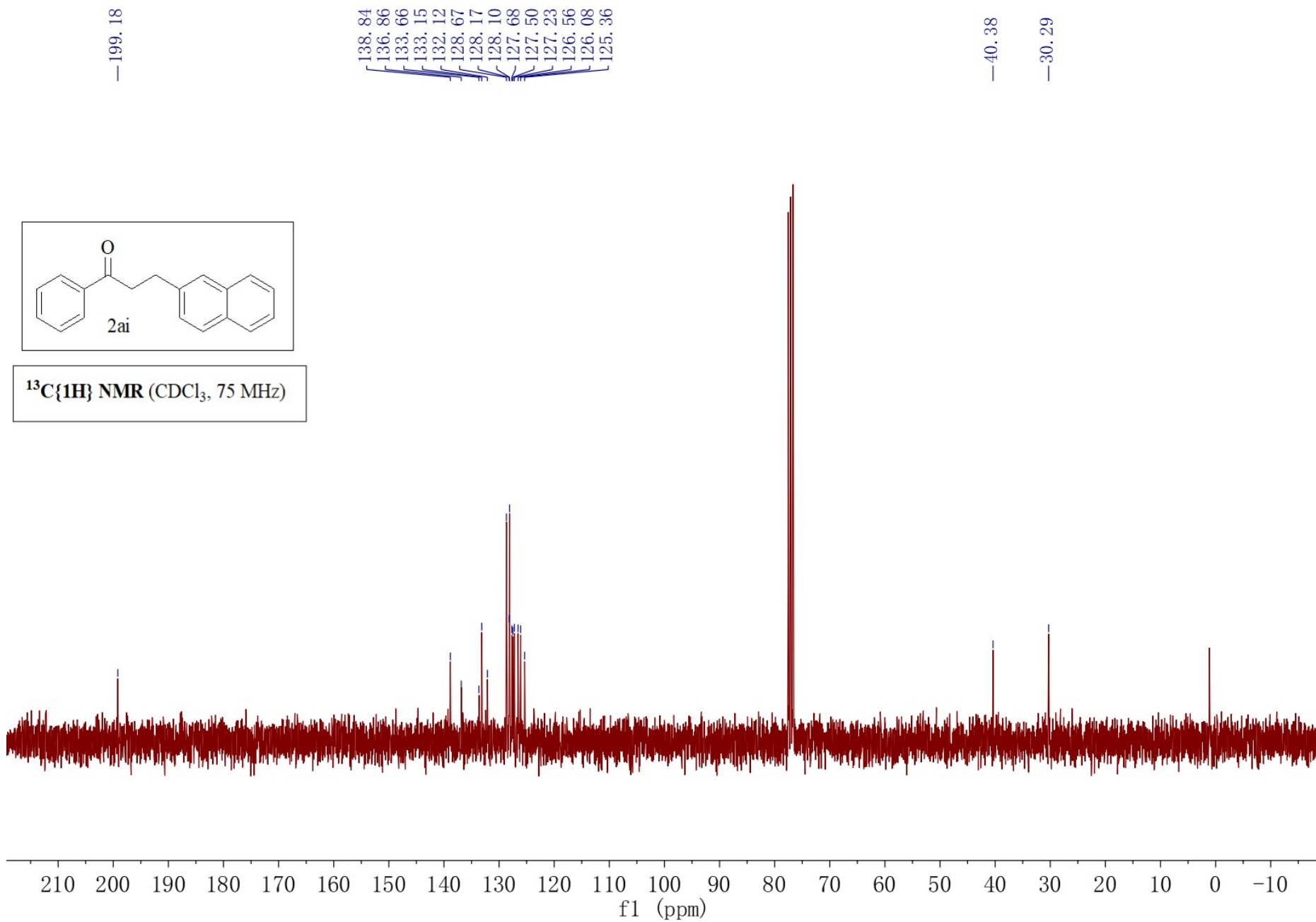
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)





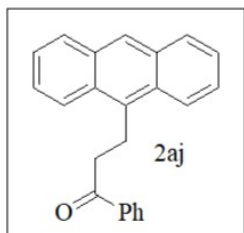


$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

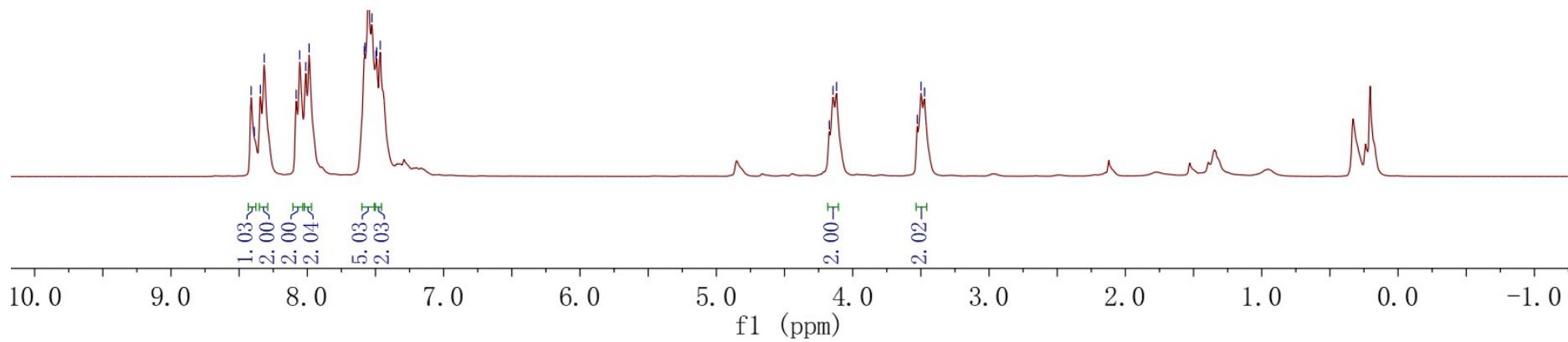


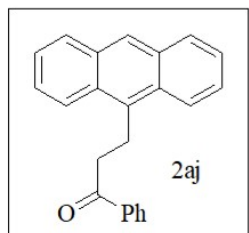
8.41
8.39
8.34
8.32
8.08
8.06
8.01
7.99
7.58
7.58
7.55
7.53
7.50
7.49
7.46

4.17
4.14
4.12
3.53
3.50
3.47

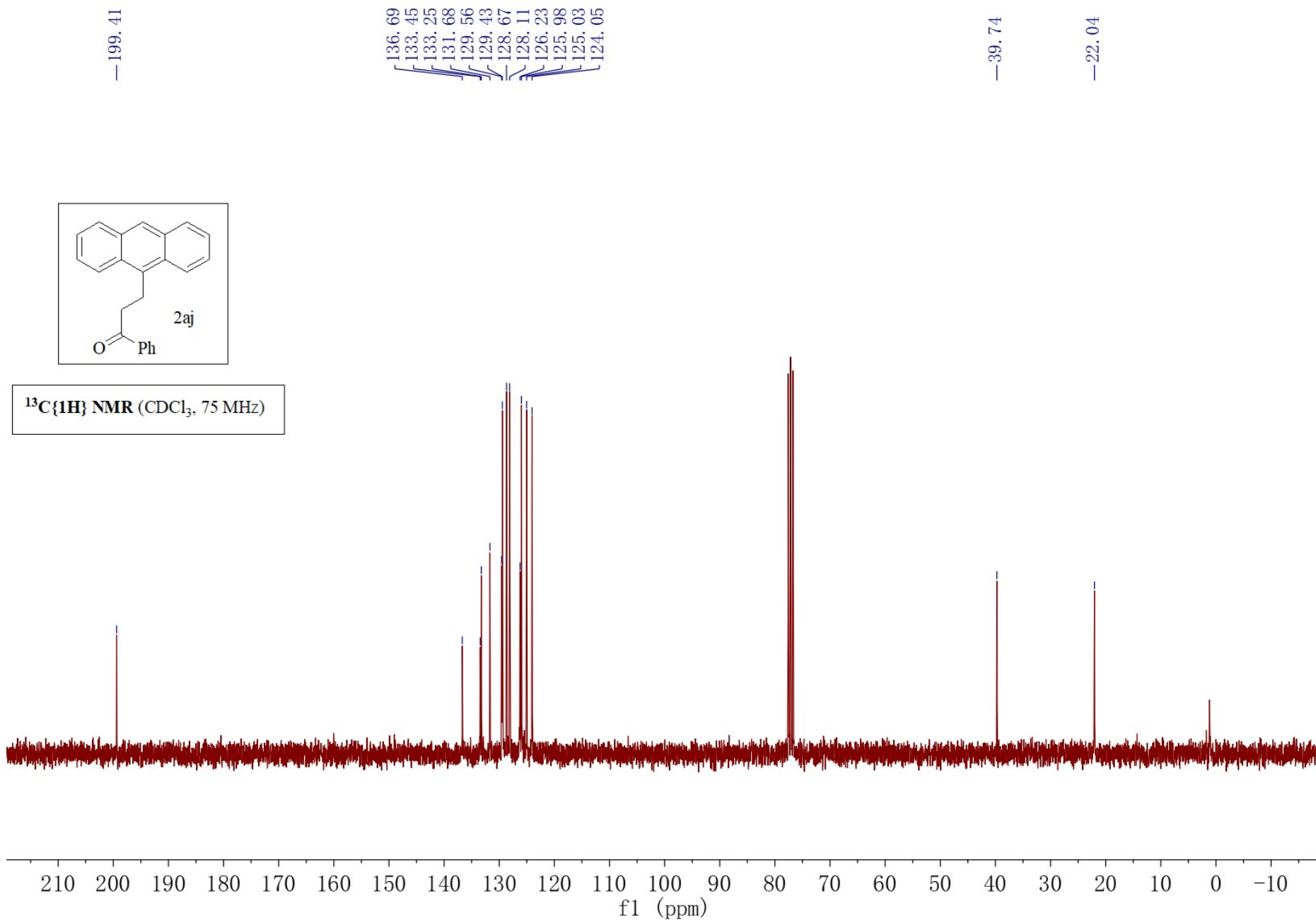


^1H NMR (CDCl_3 , 300 MHz)



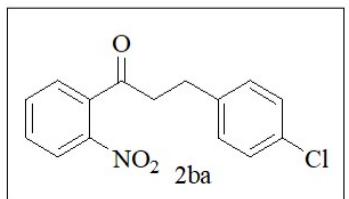


$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

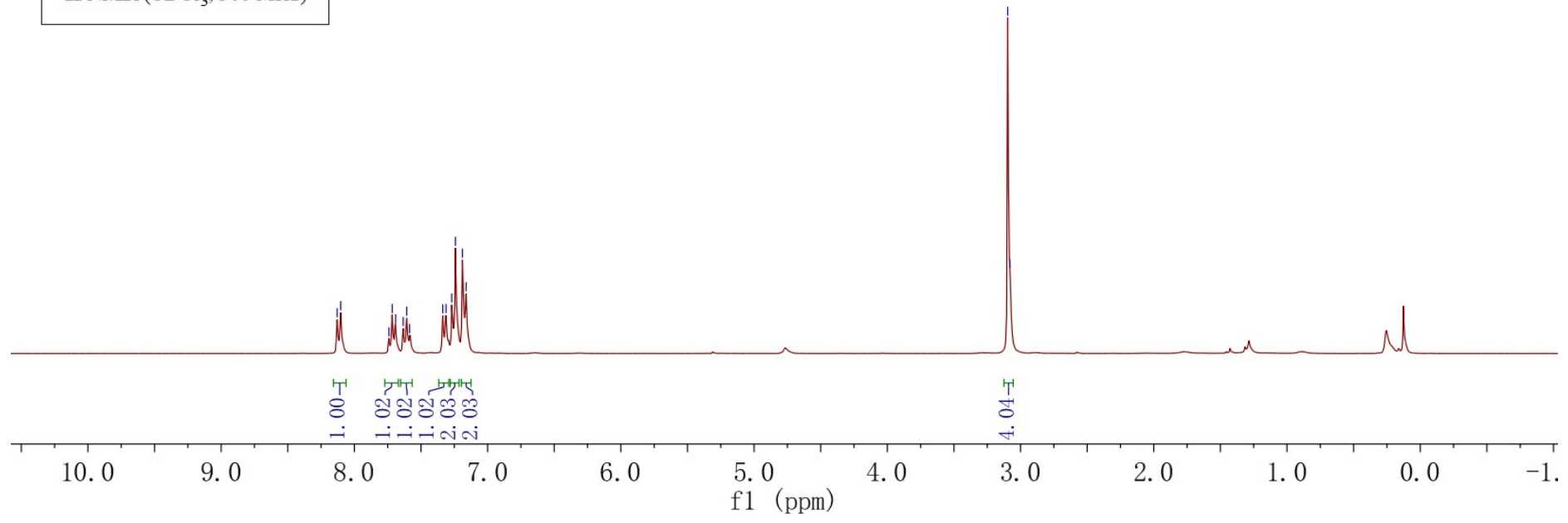


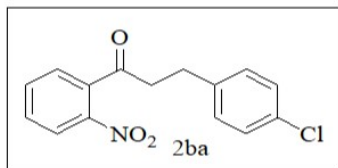
8.13
8.10
7.74
7.72
7.69
7.63
7.61
7.59
7.34
7.31
7.27
7.24
7.19
7.16

3.10
3.08

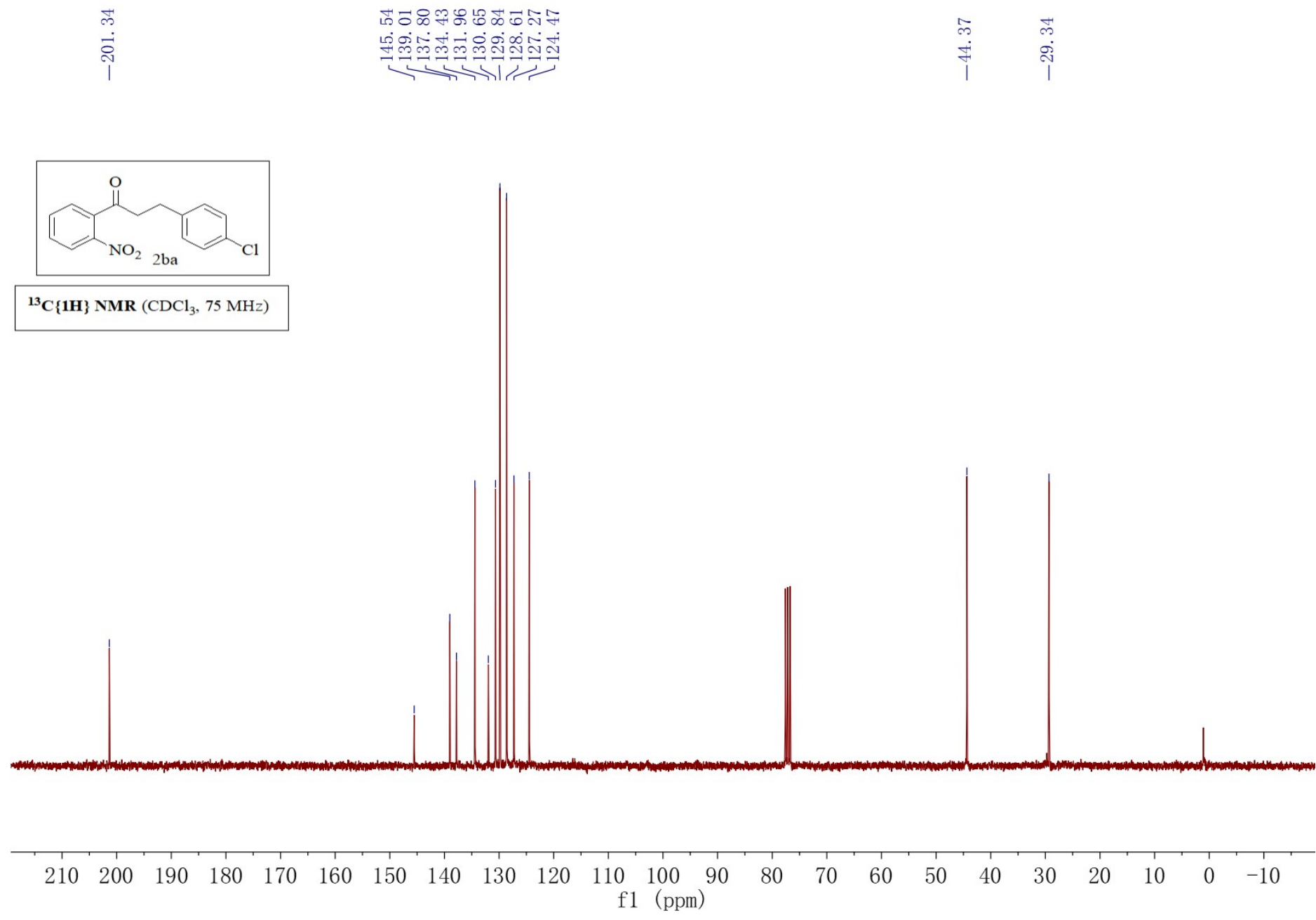


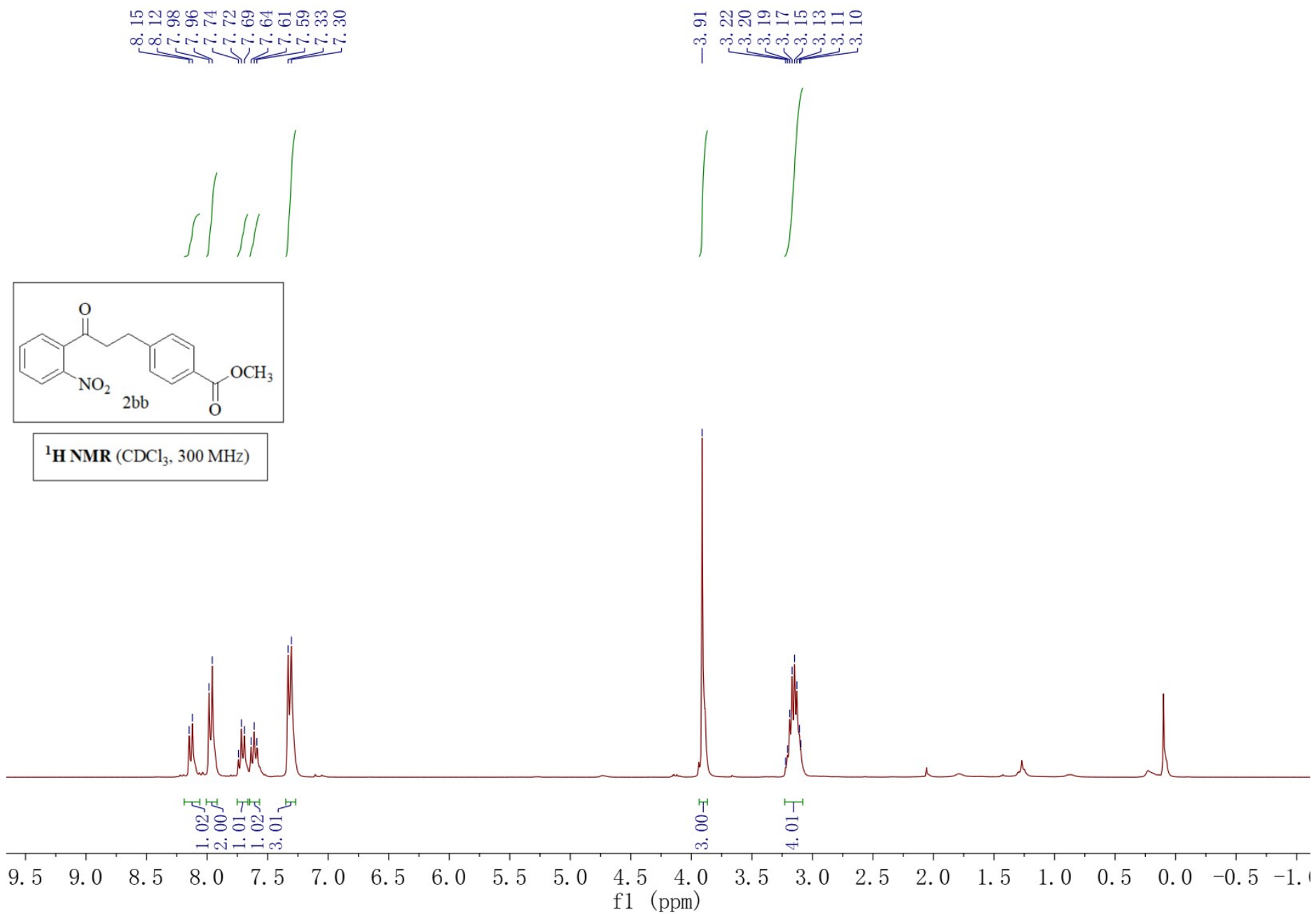
¹H NMR (CDCl₃, 300 MHz)

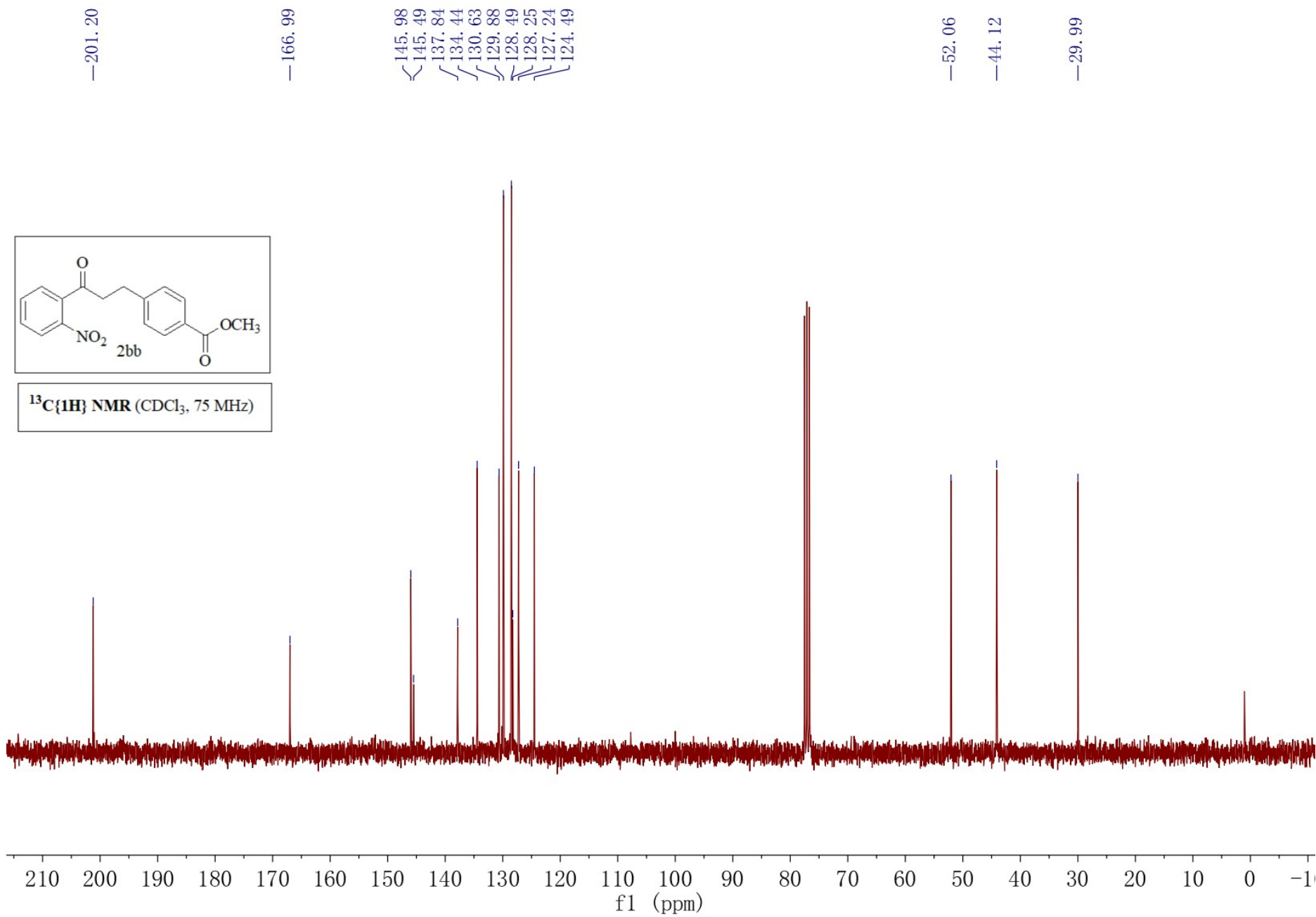


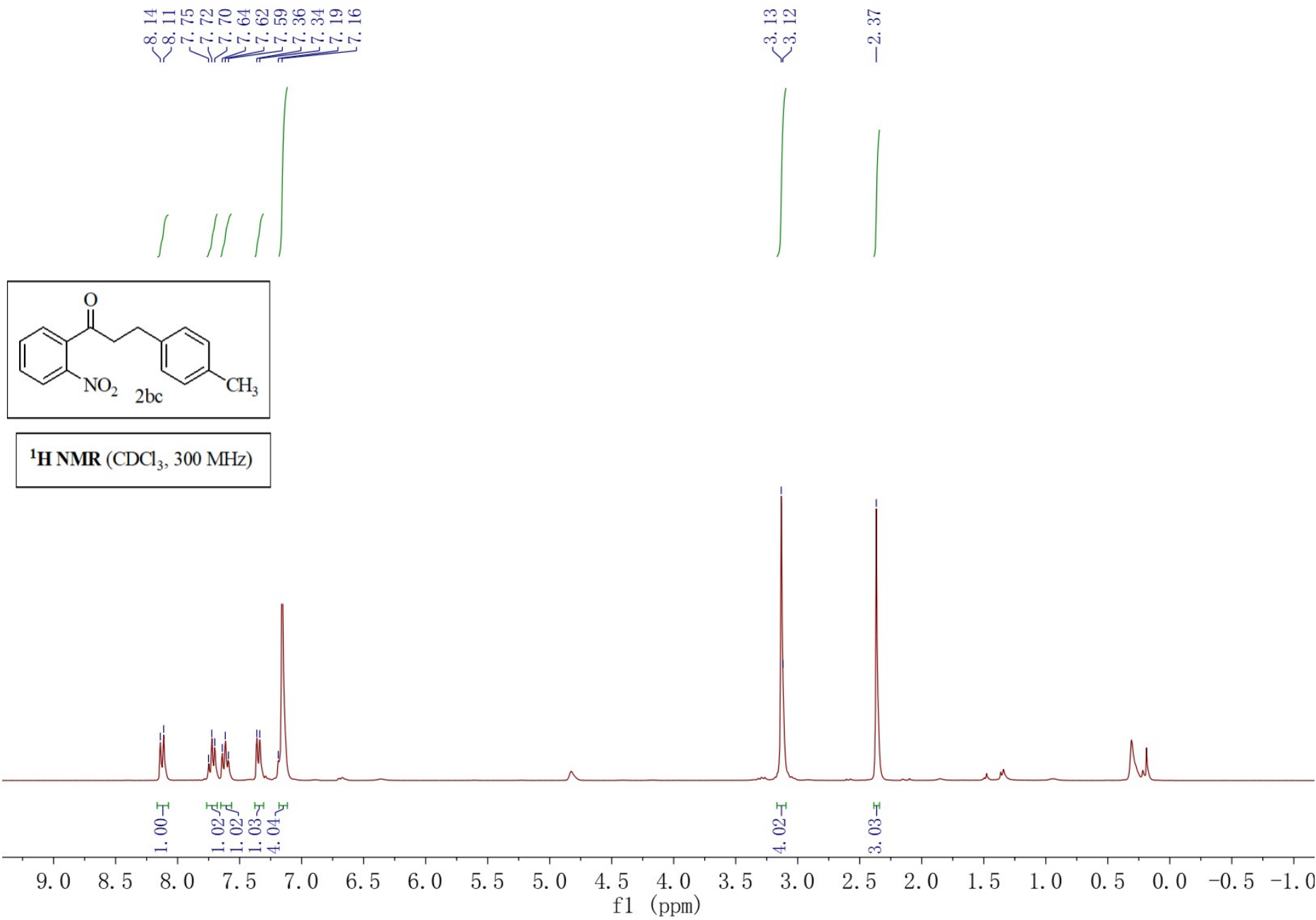


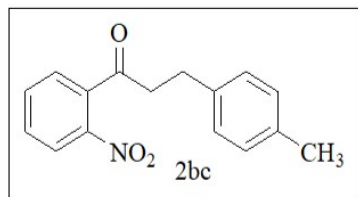
¹³C{¹H} NMR (CDCl₃, 75 MHz)



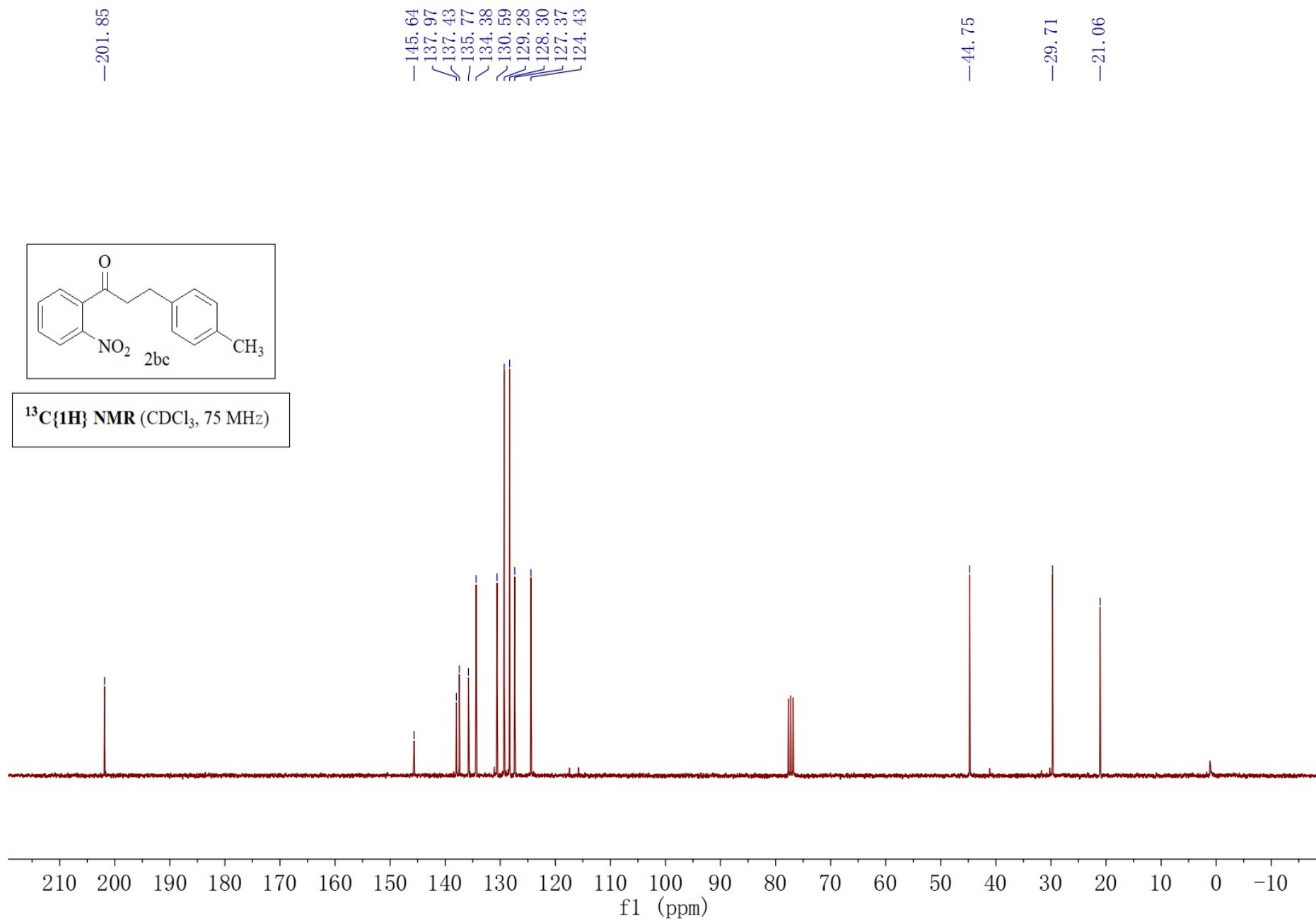


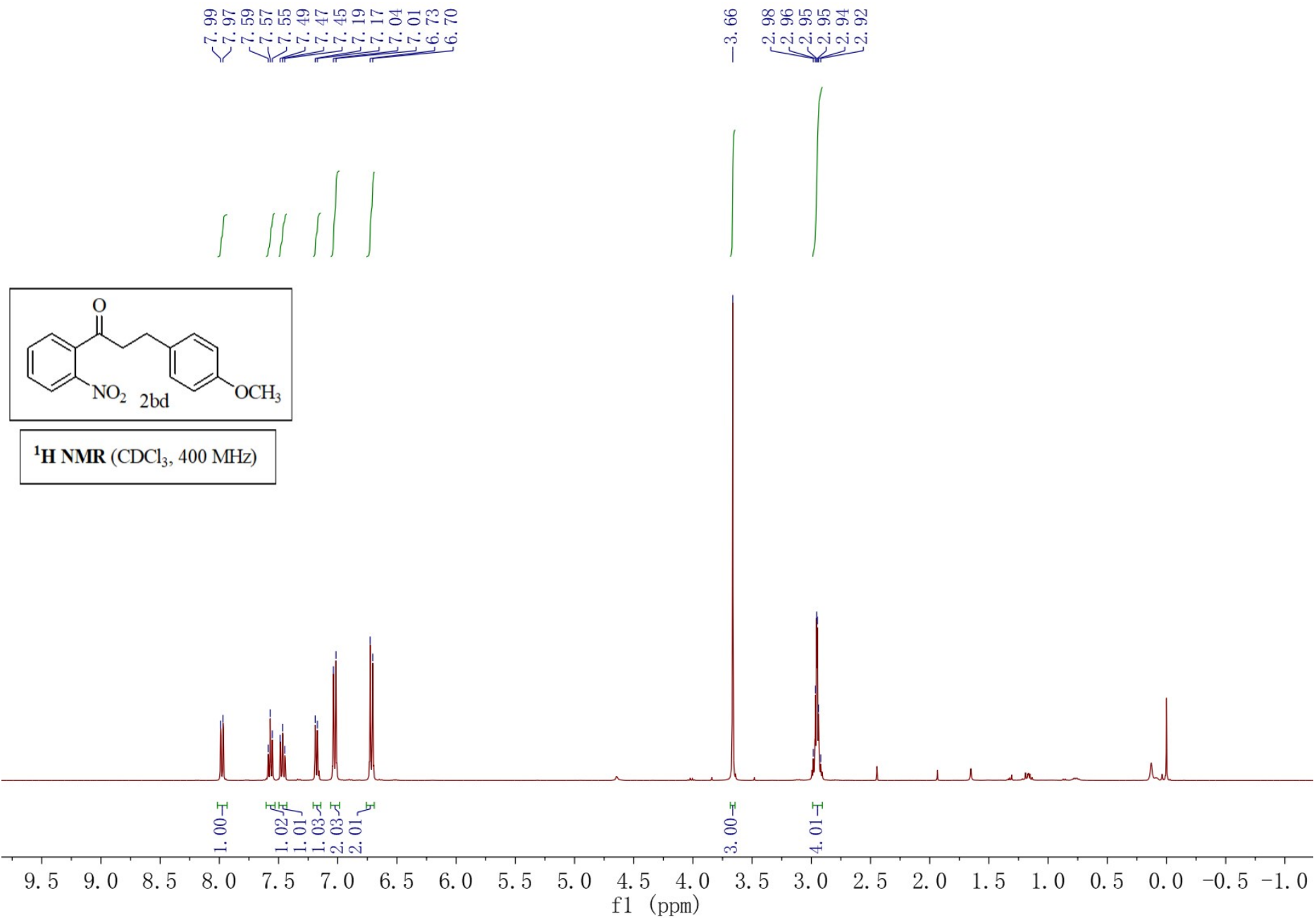


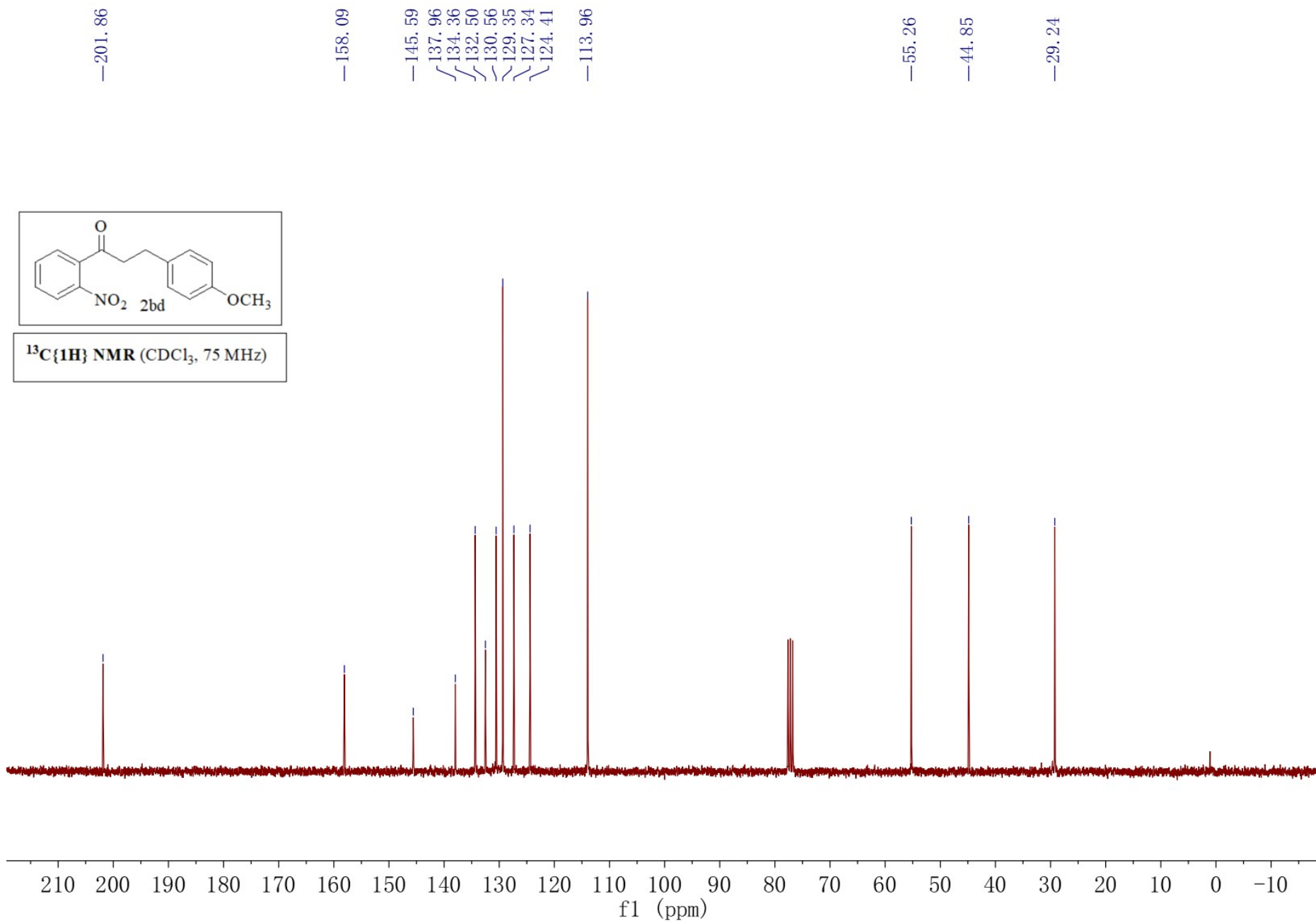


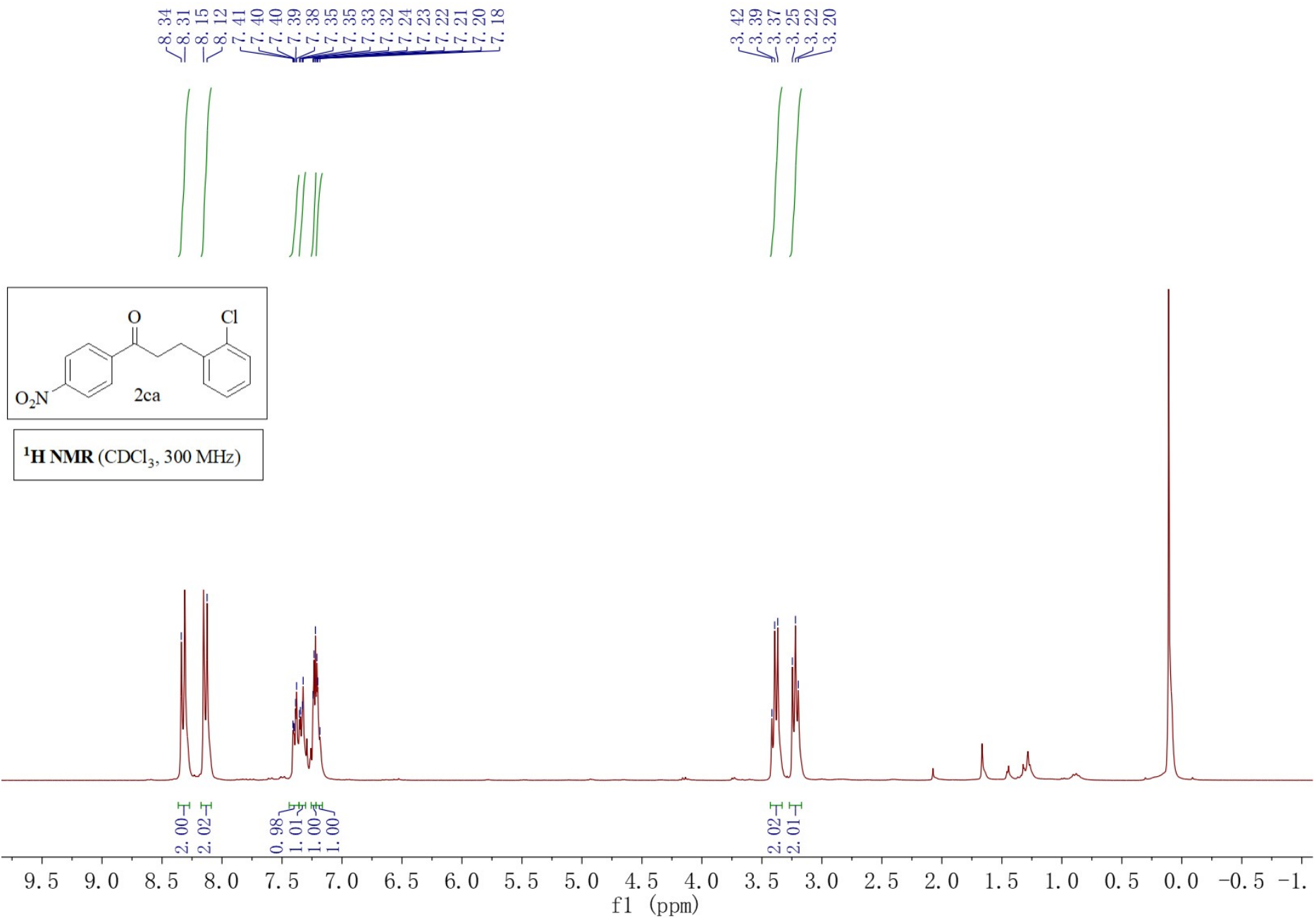


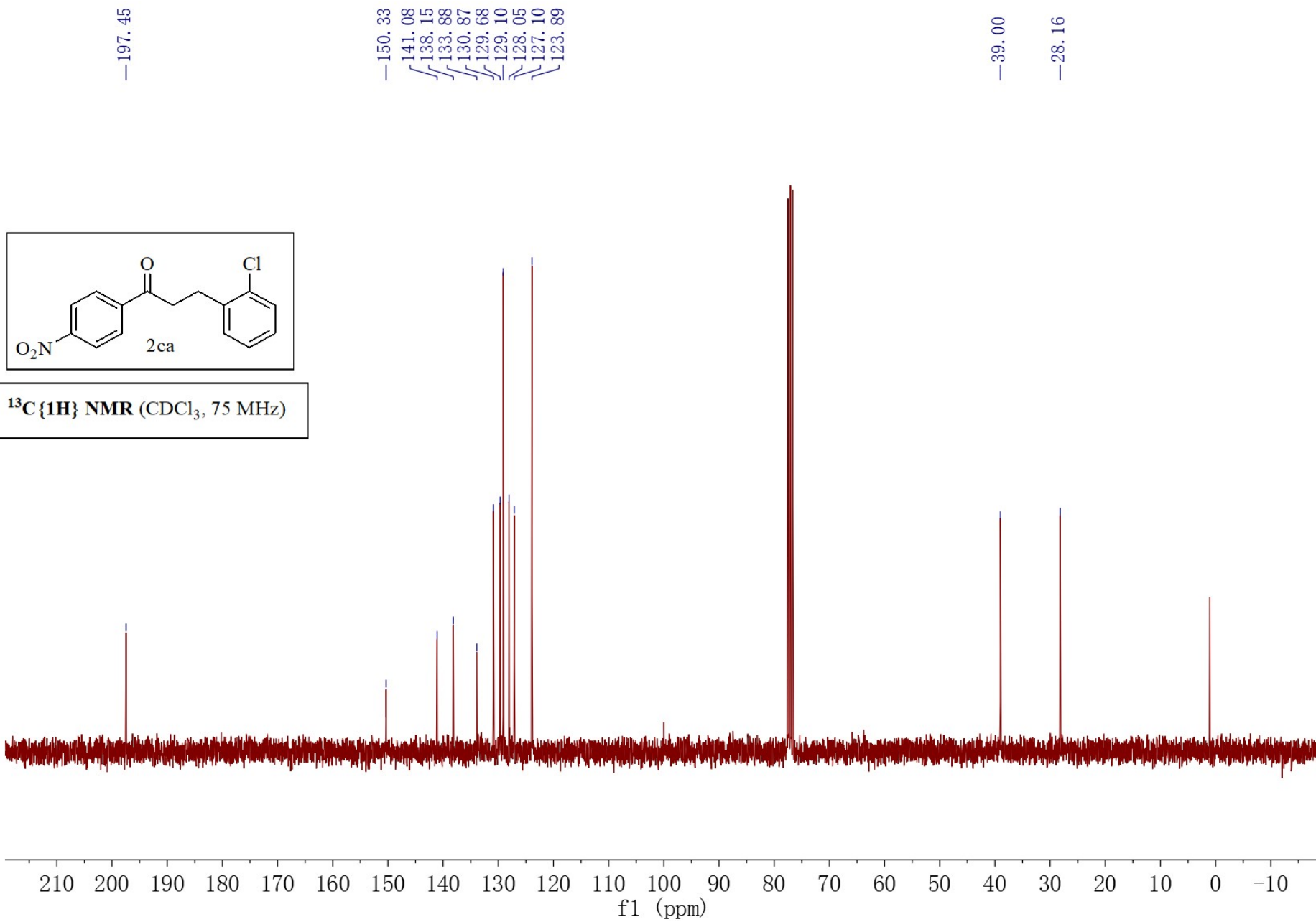
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

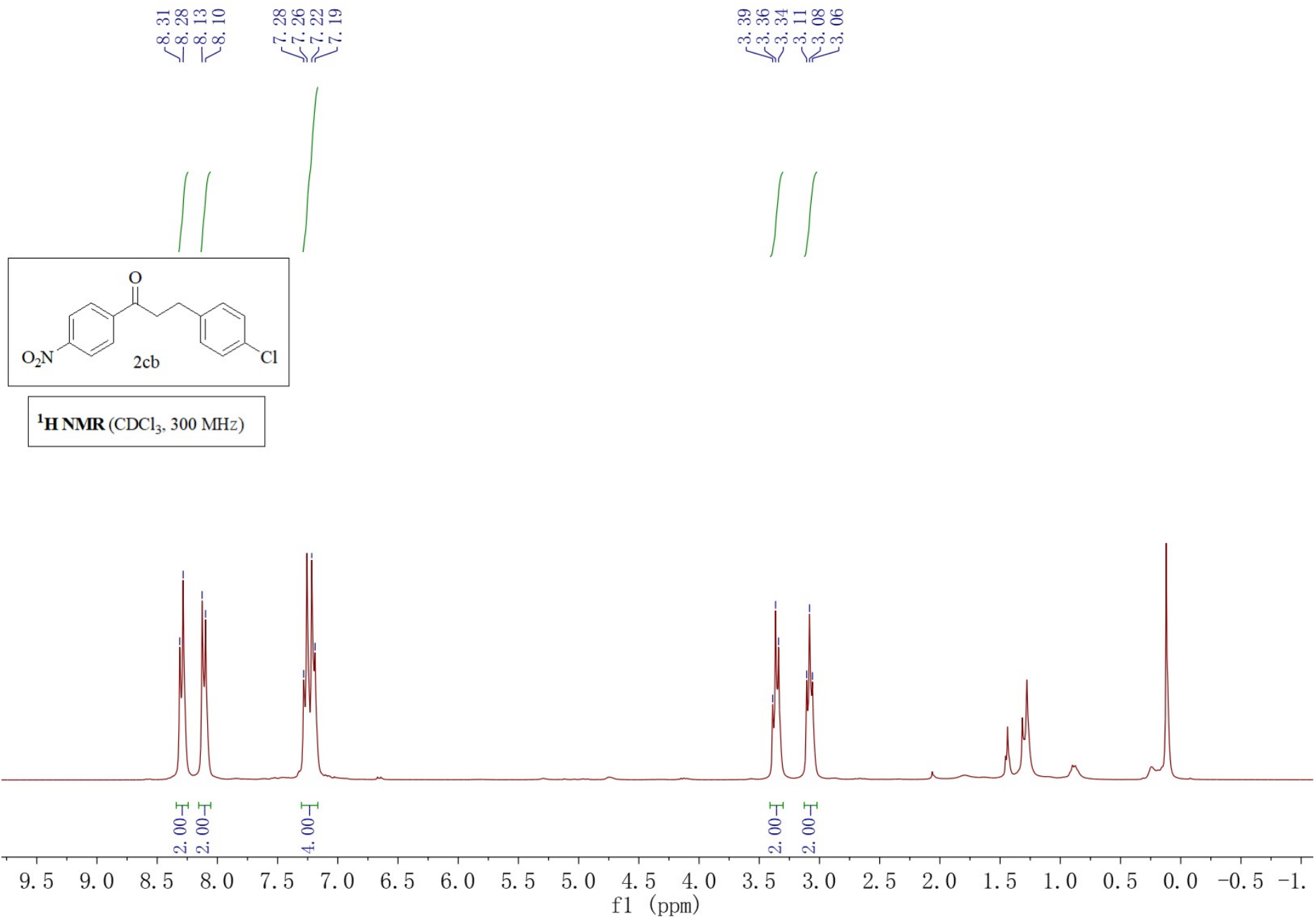


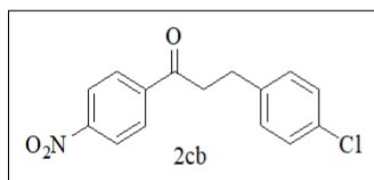




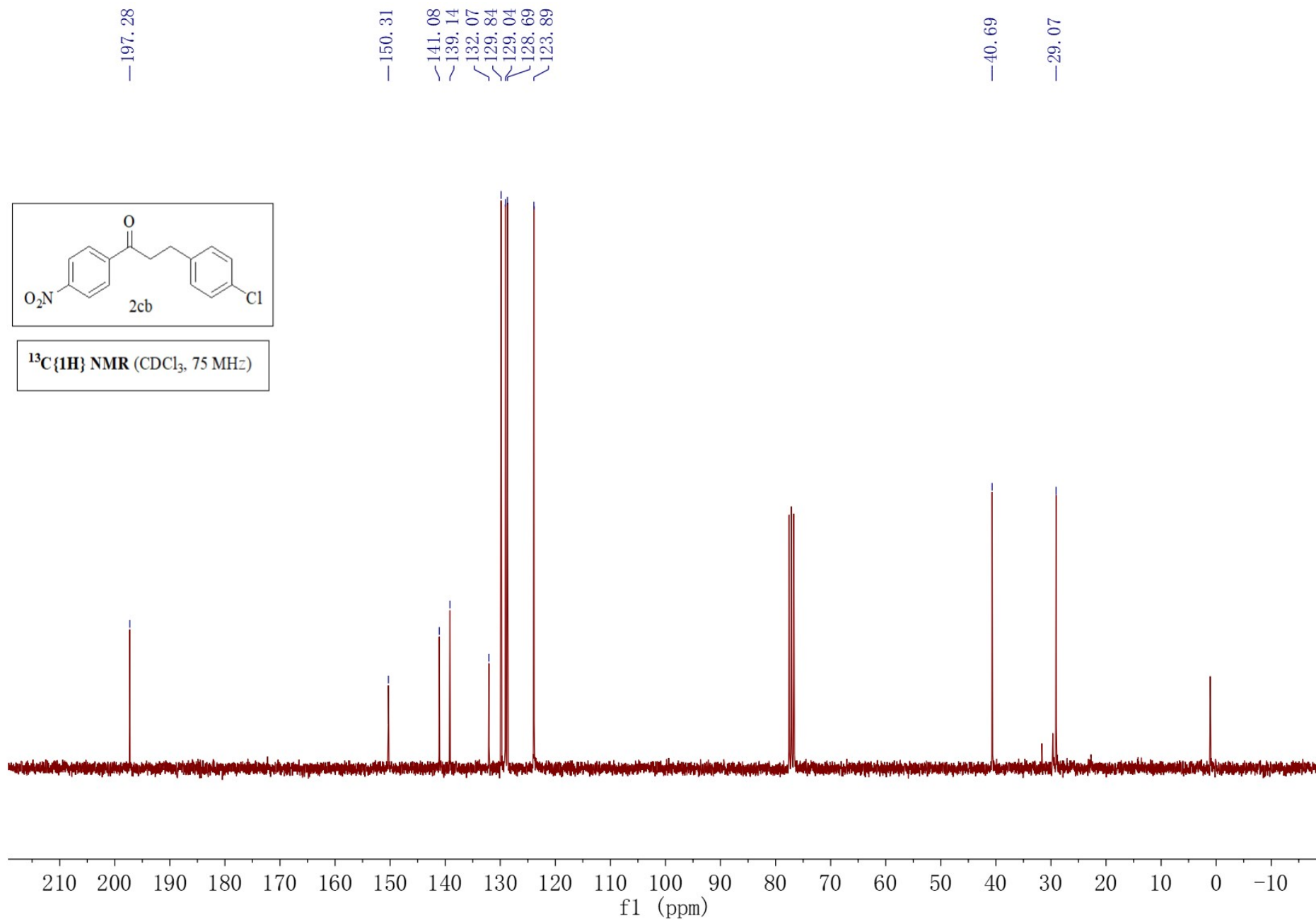


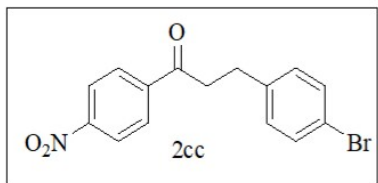




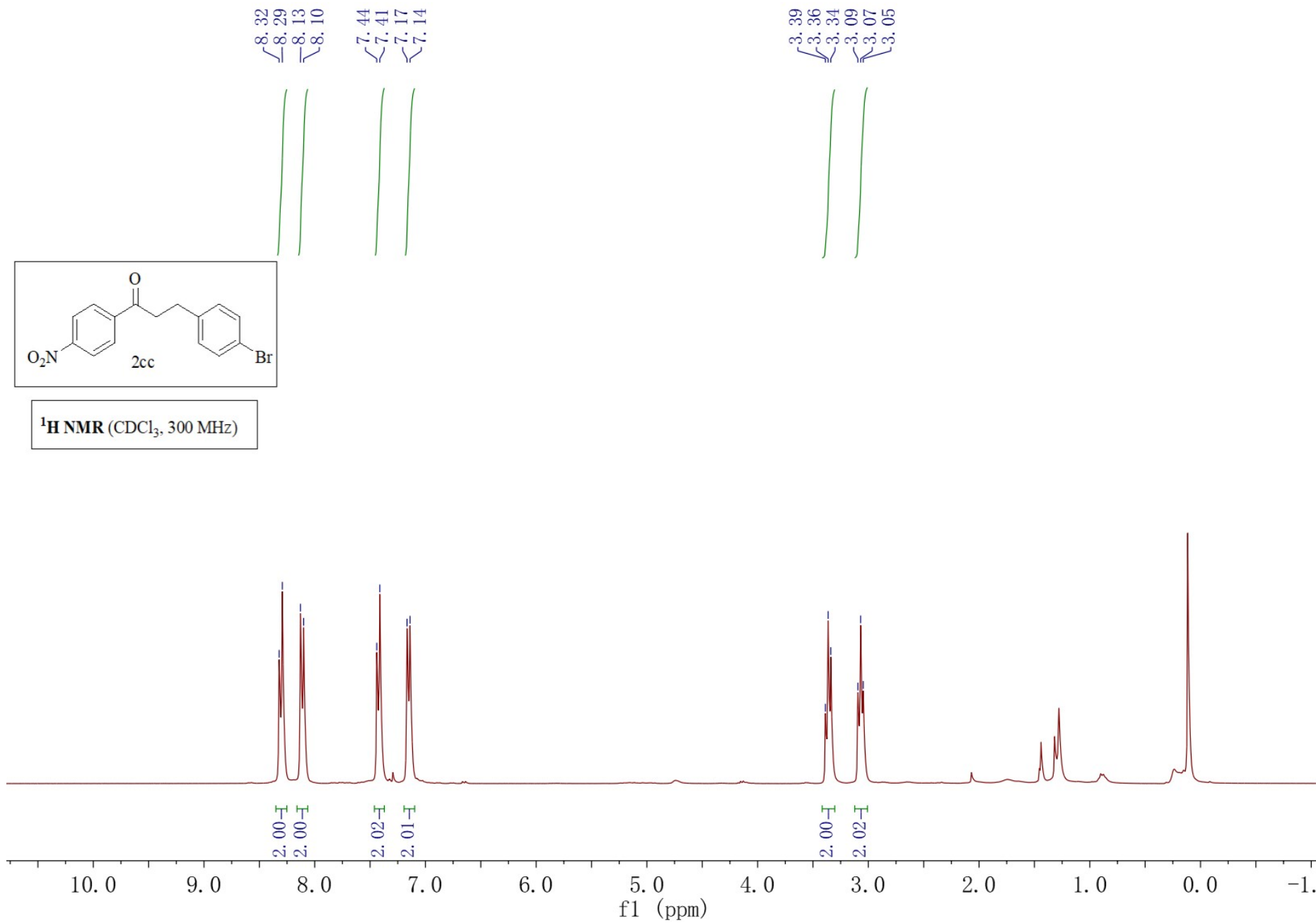


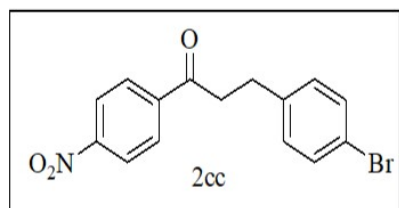
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



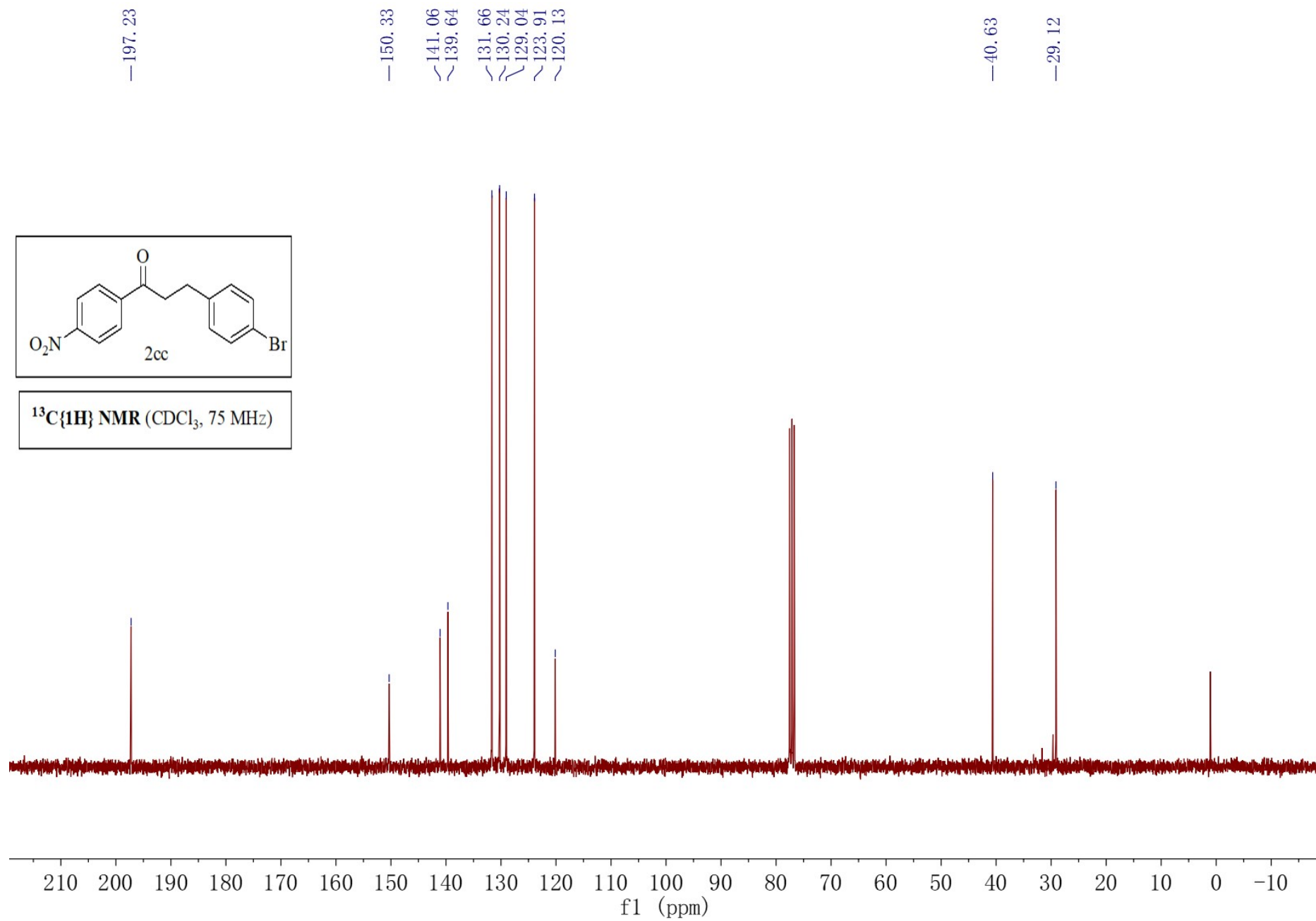


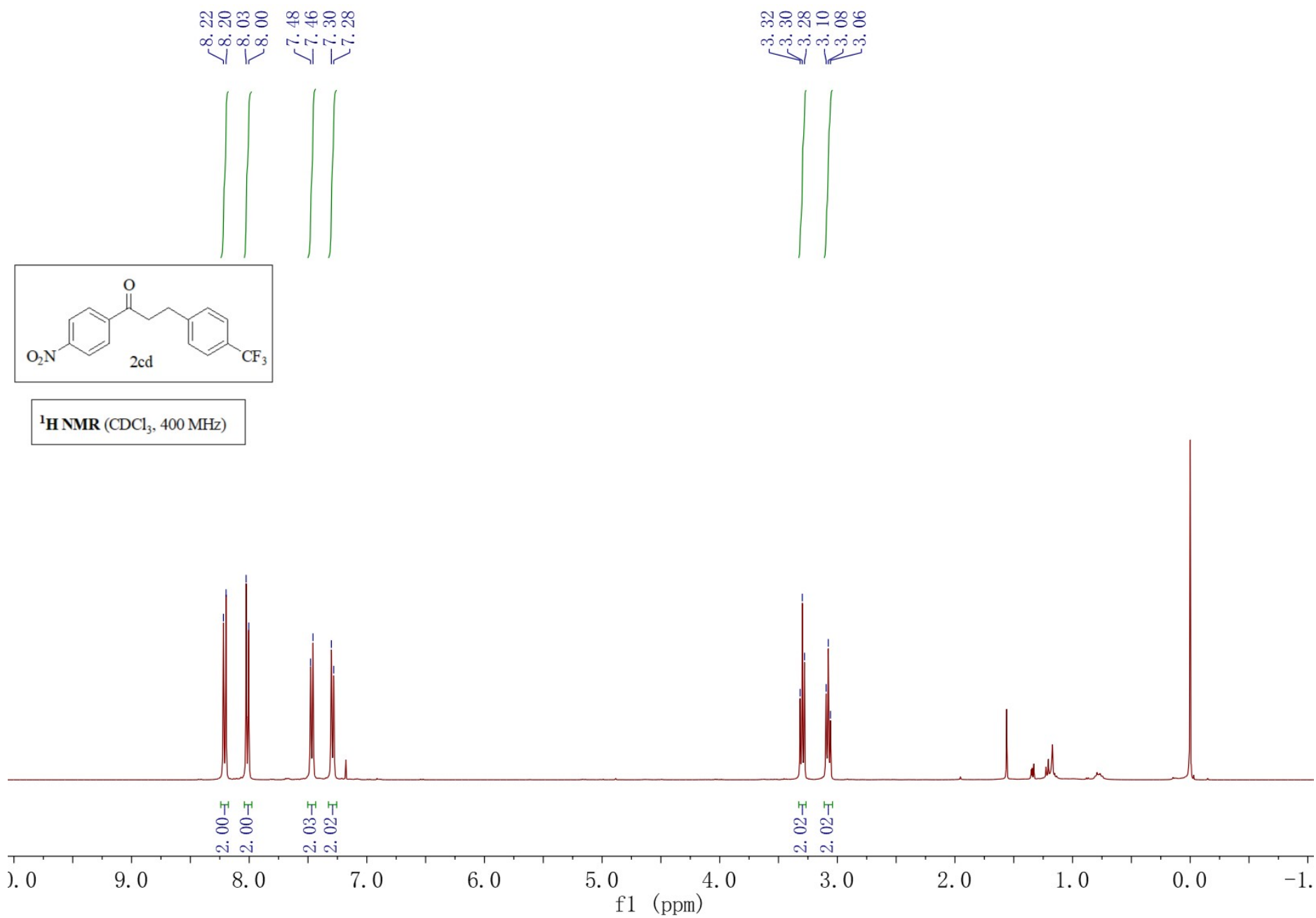
¹H NMR (CDCl₃, 300 MHz)

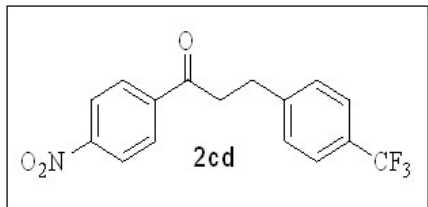




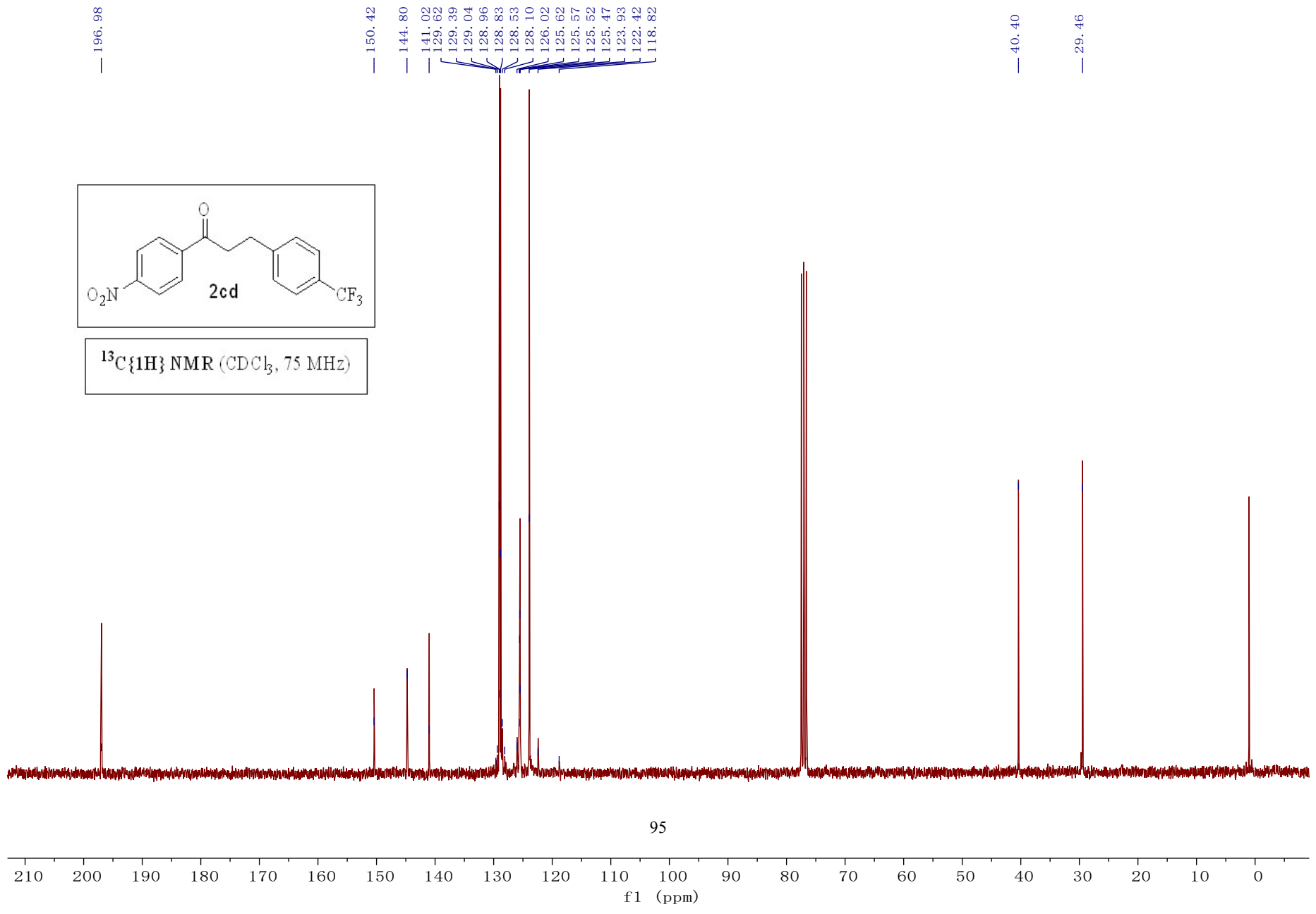
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

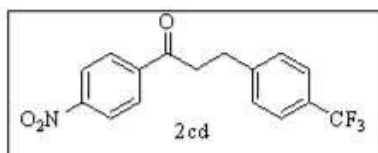






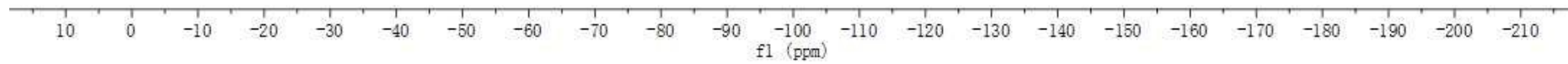
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

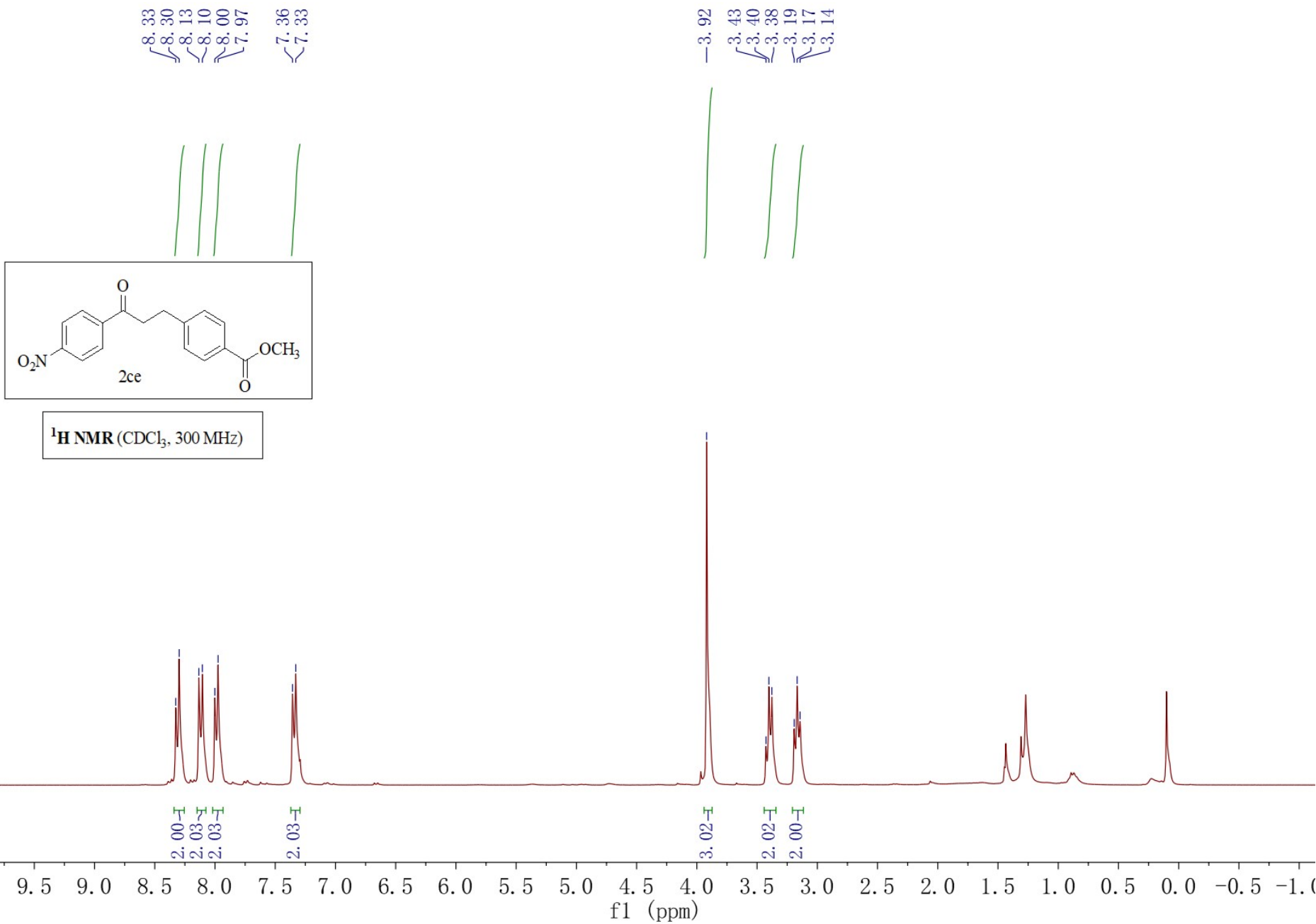


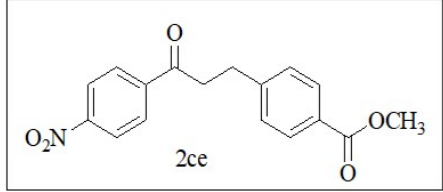


¹⁹F NMR (CDCl₃, 282 MHz)

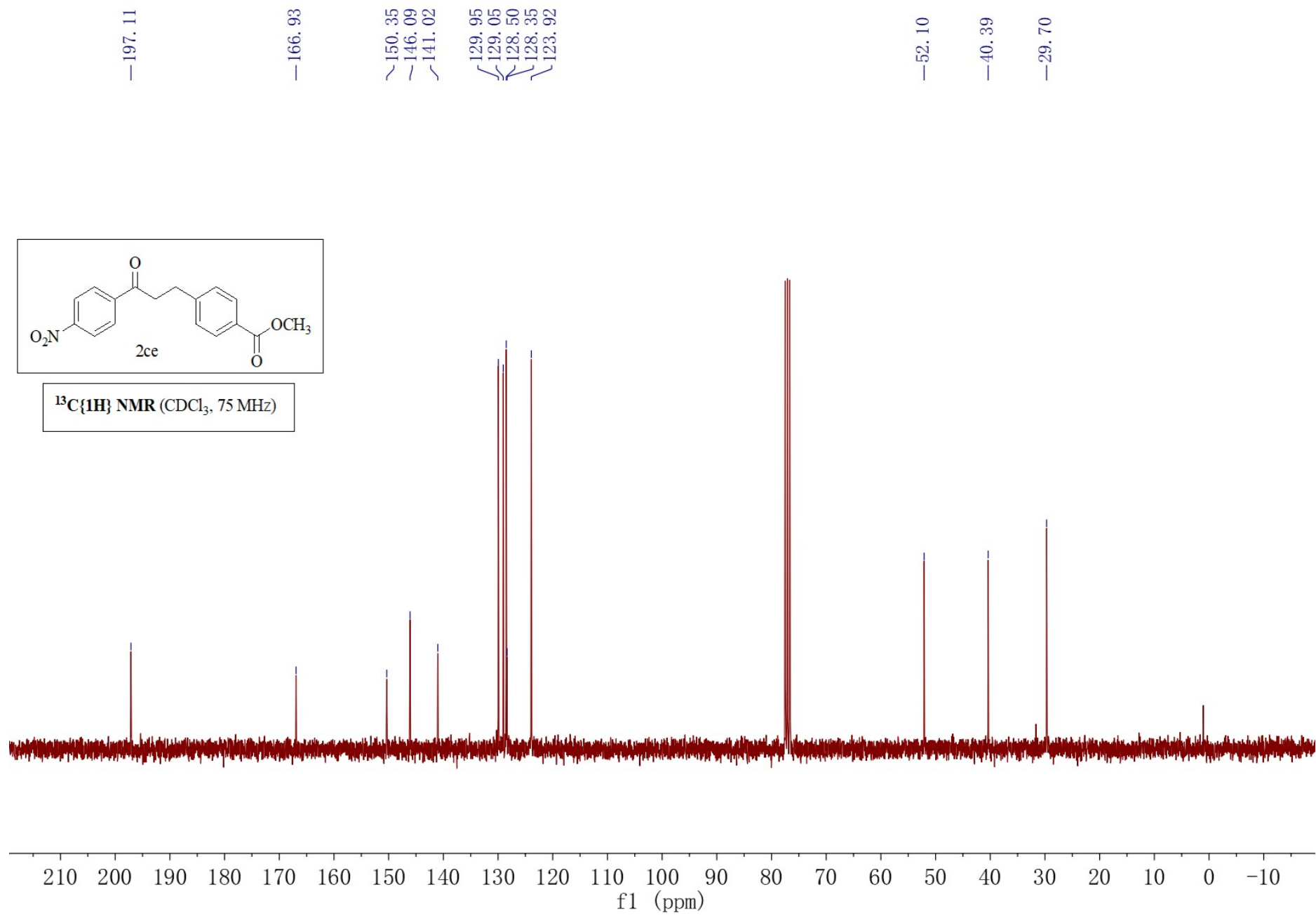
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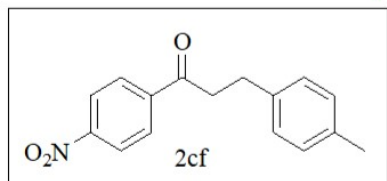




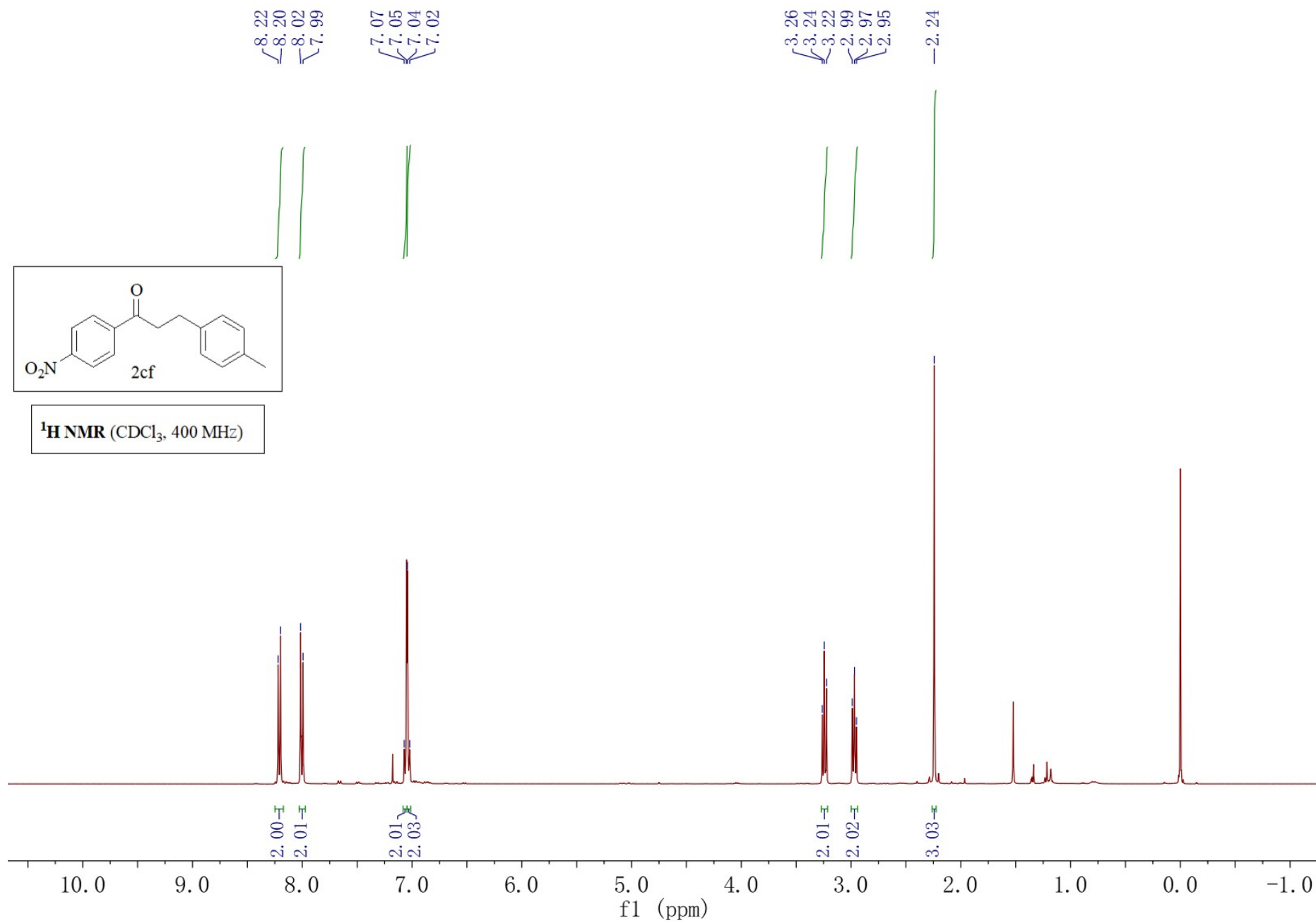


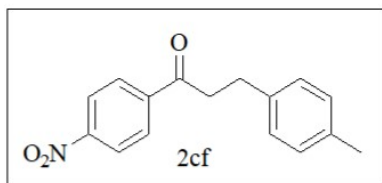
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



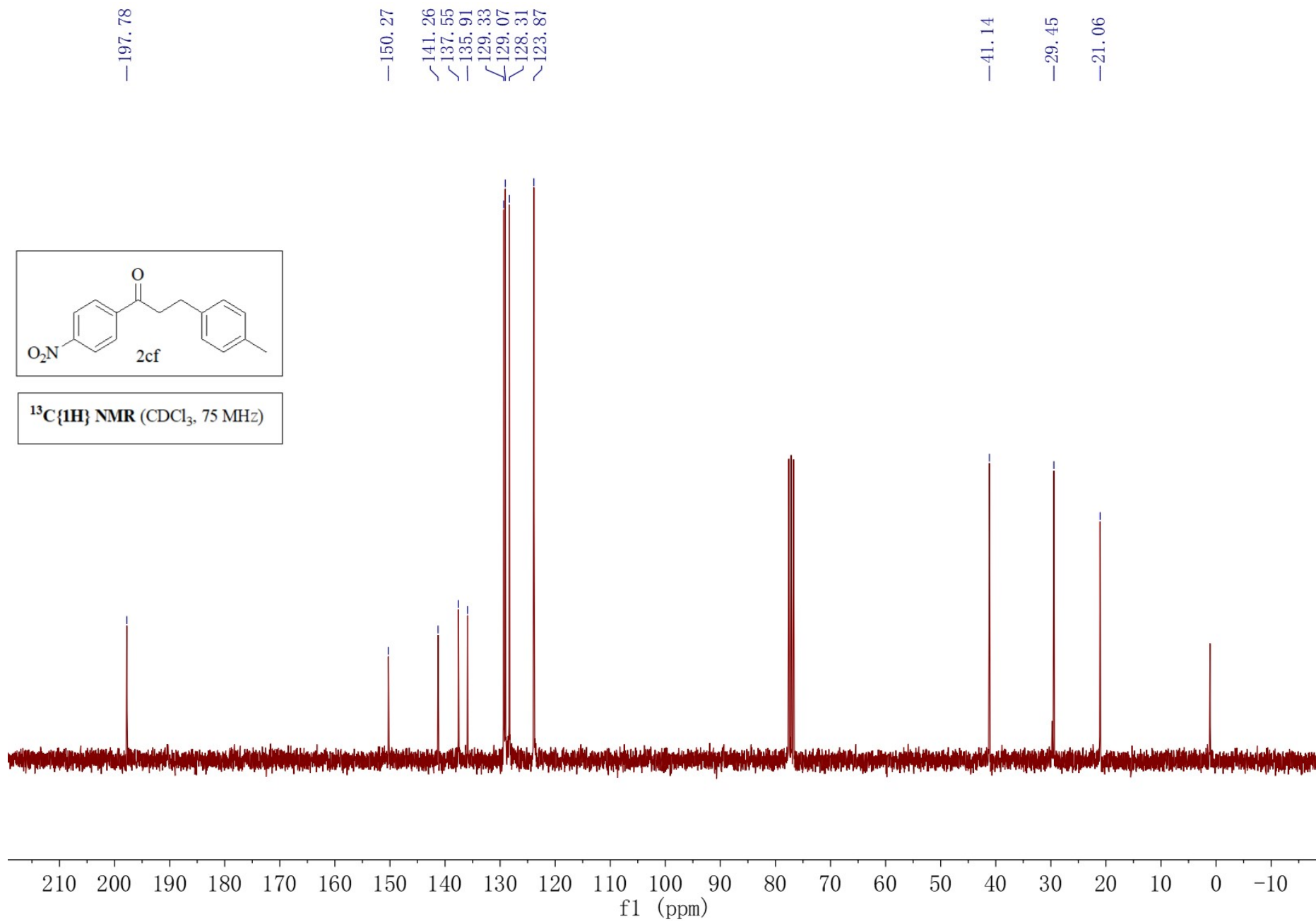


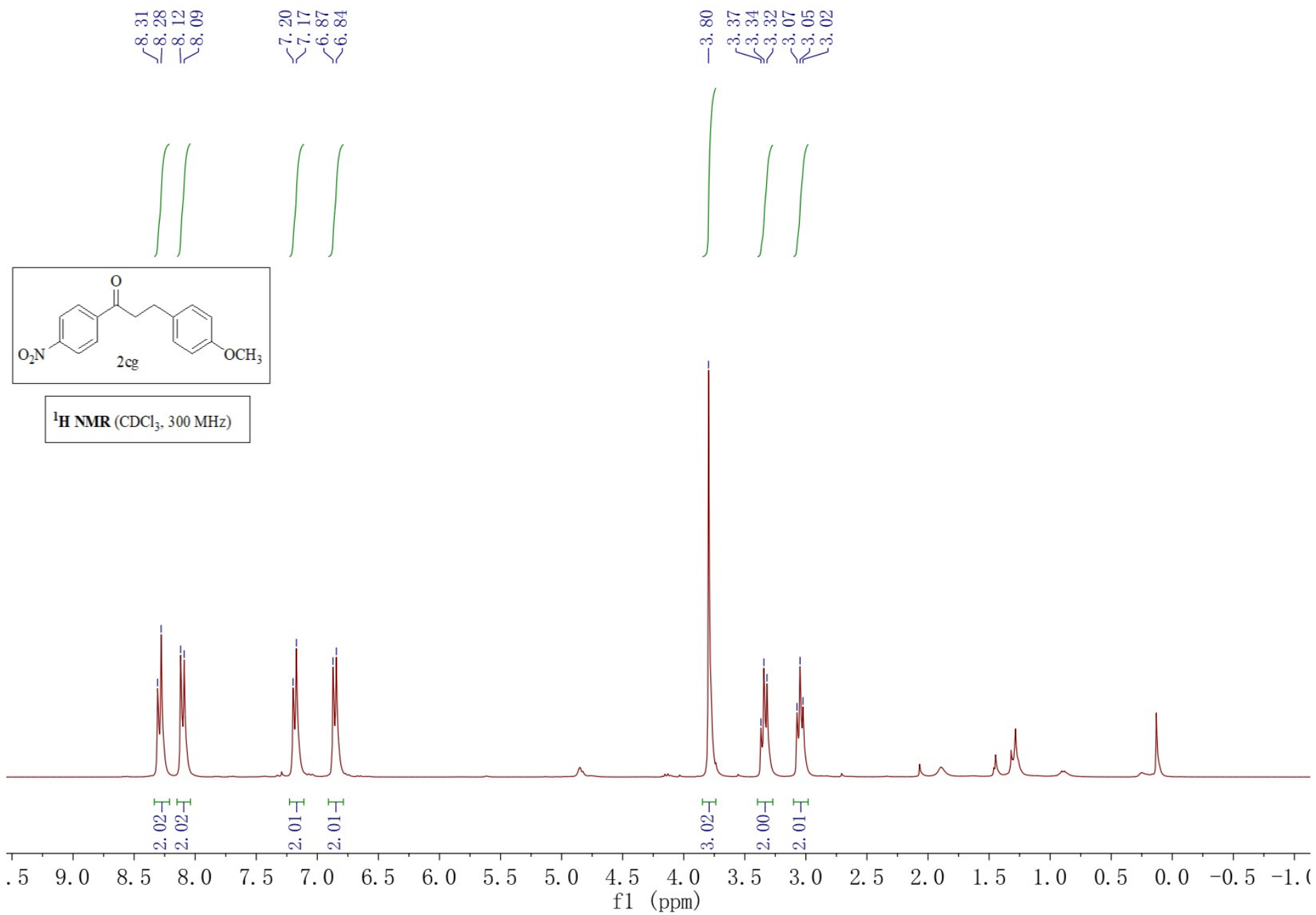
$^1\text{H NMR}$ (CDCl_3 , 400 MHz)

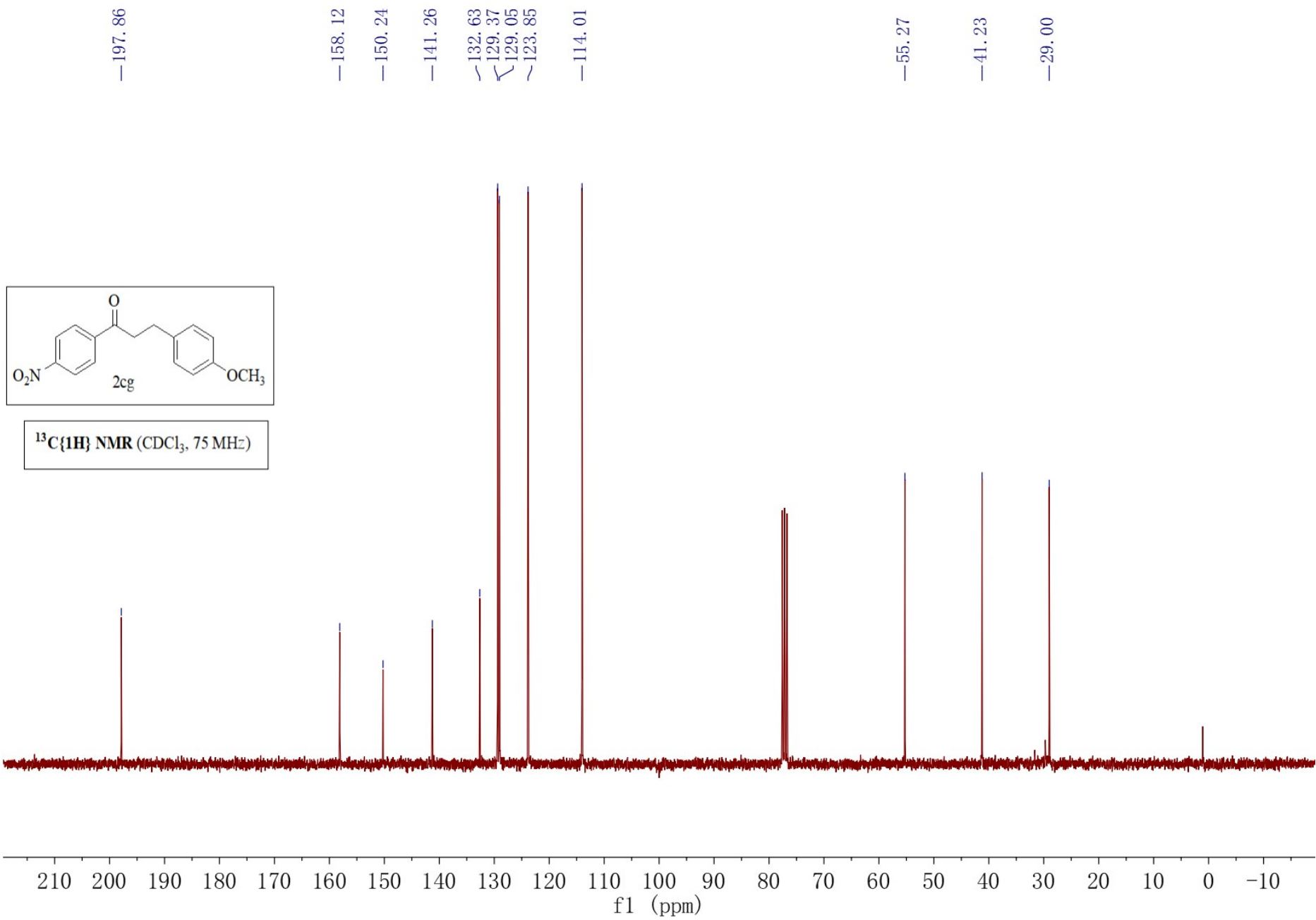


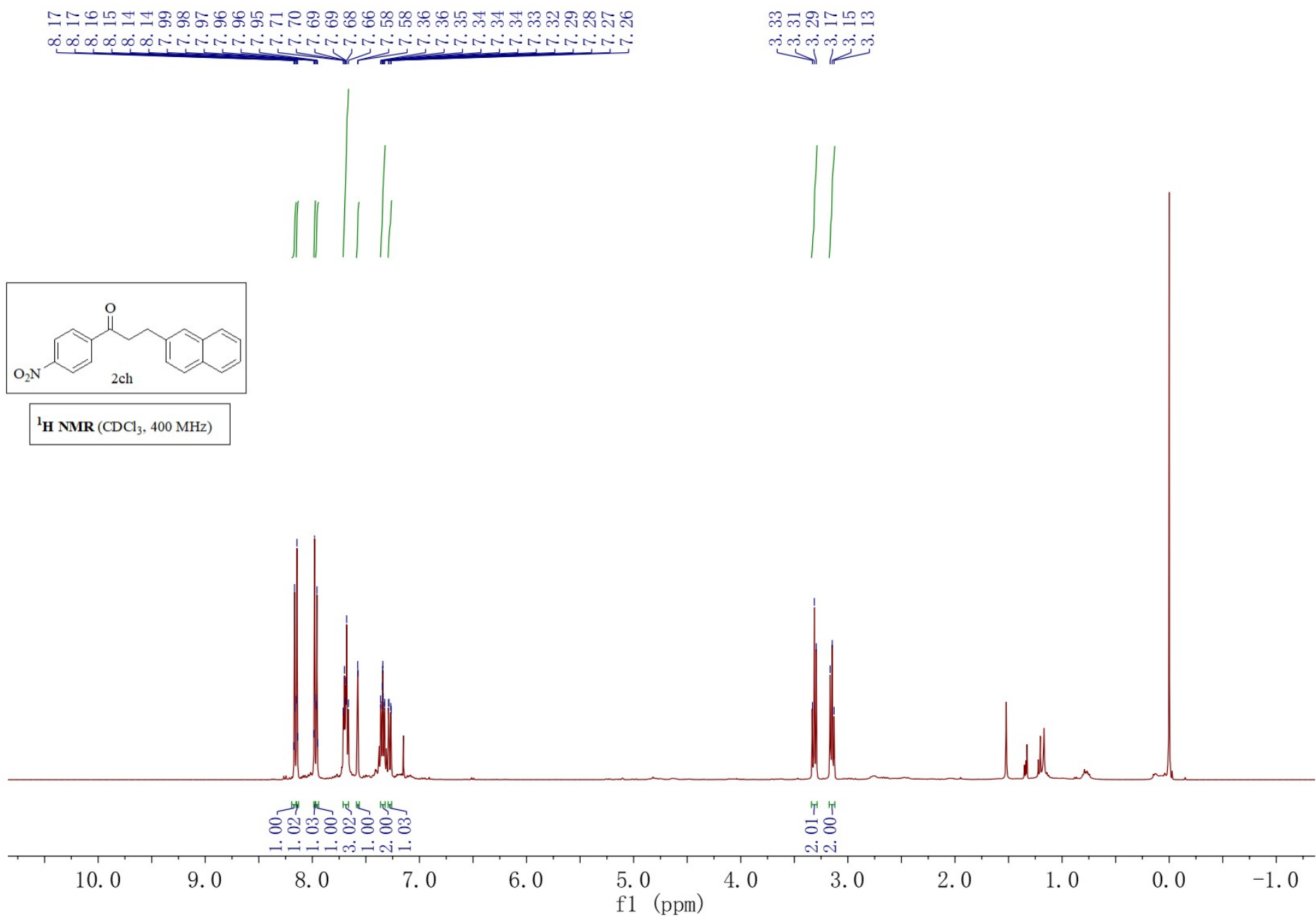


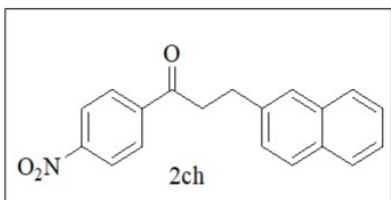
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



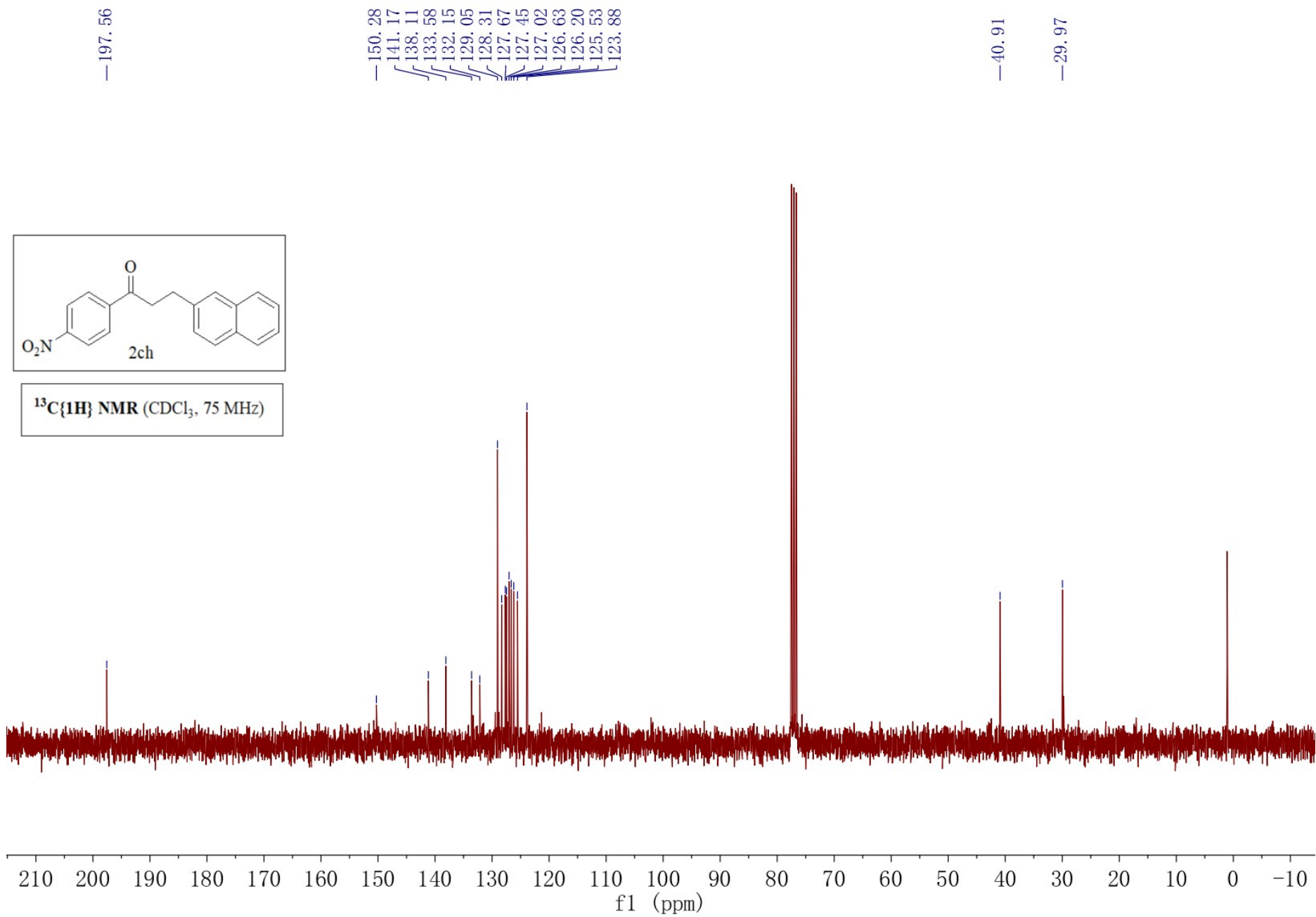


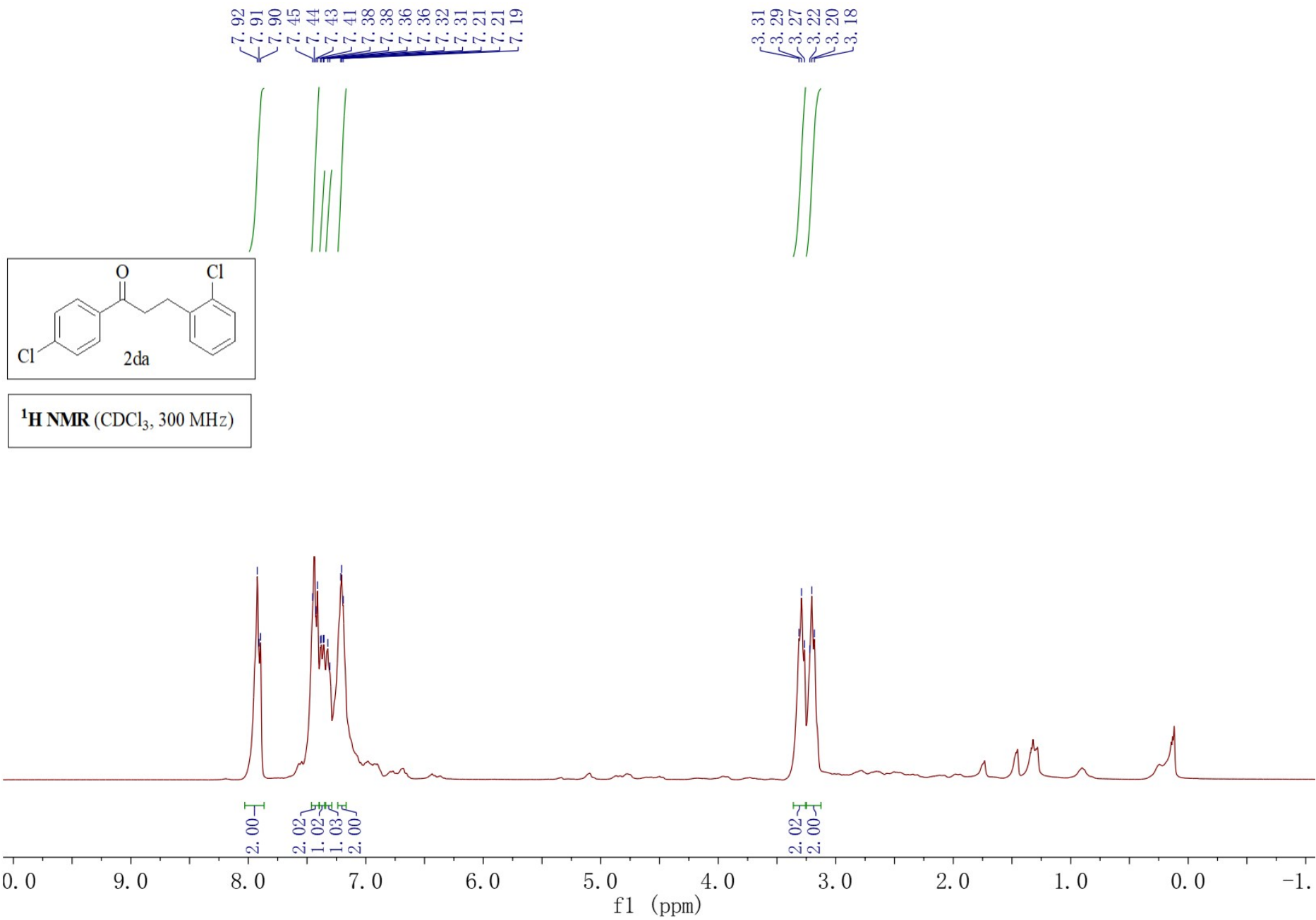


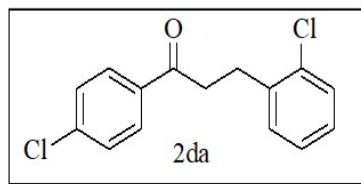




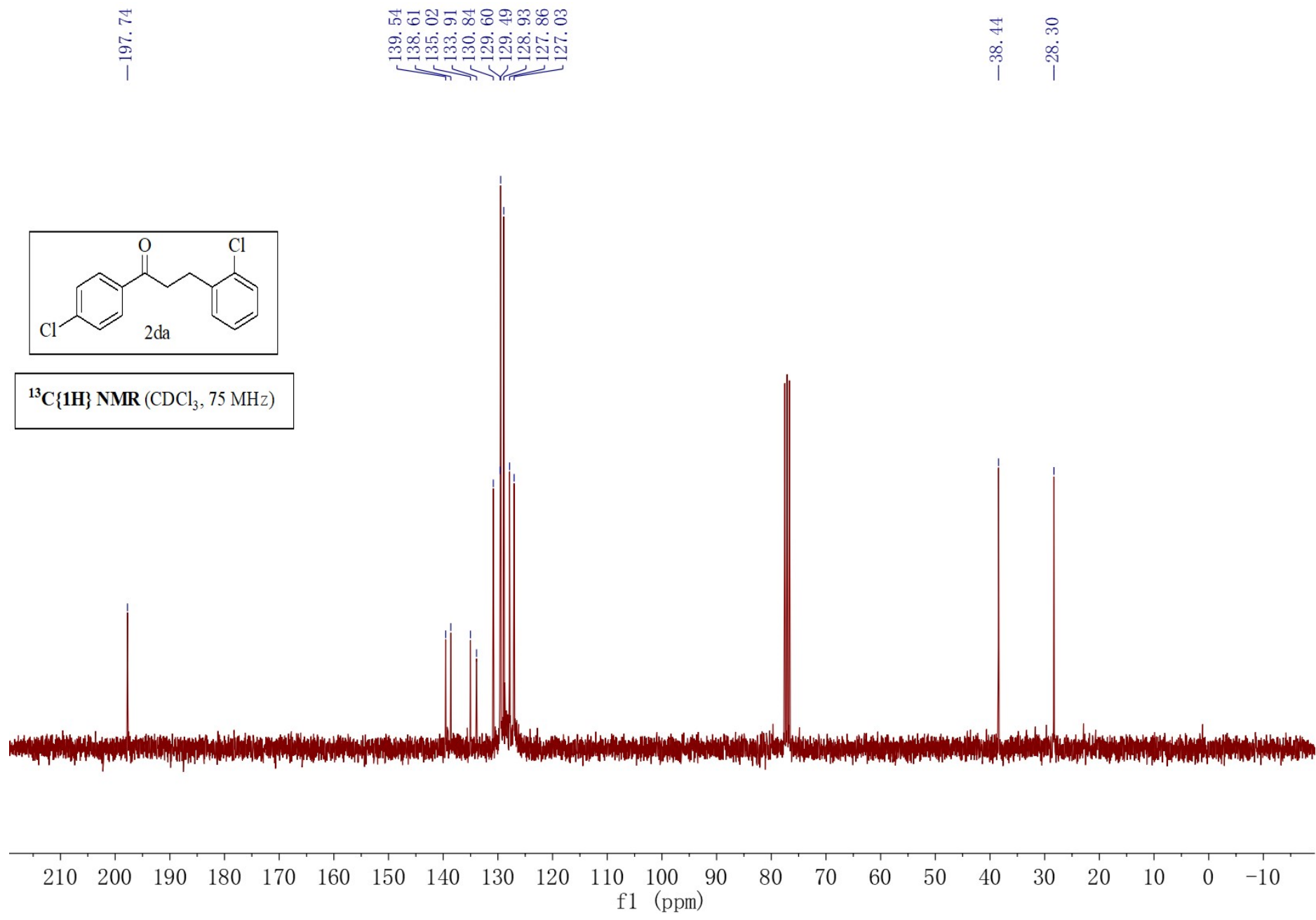
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

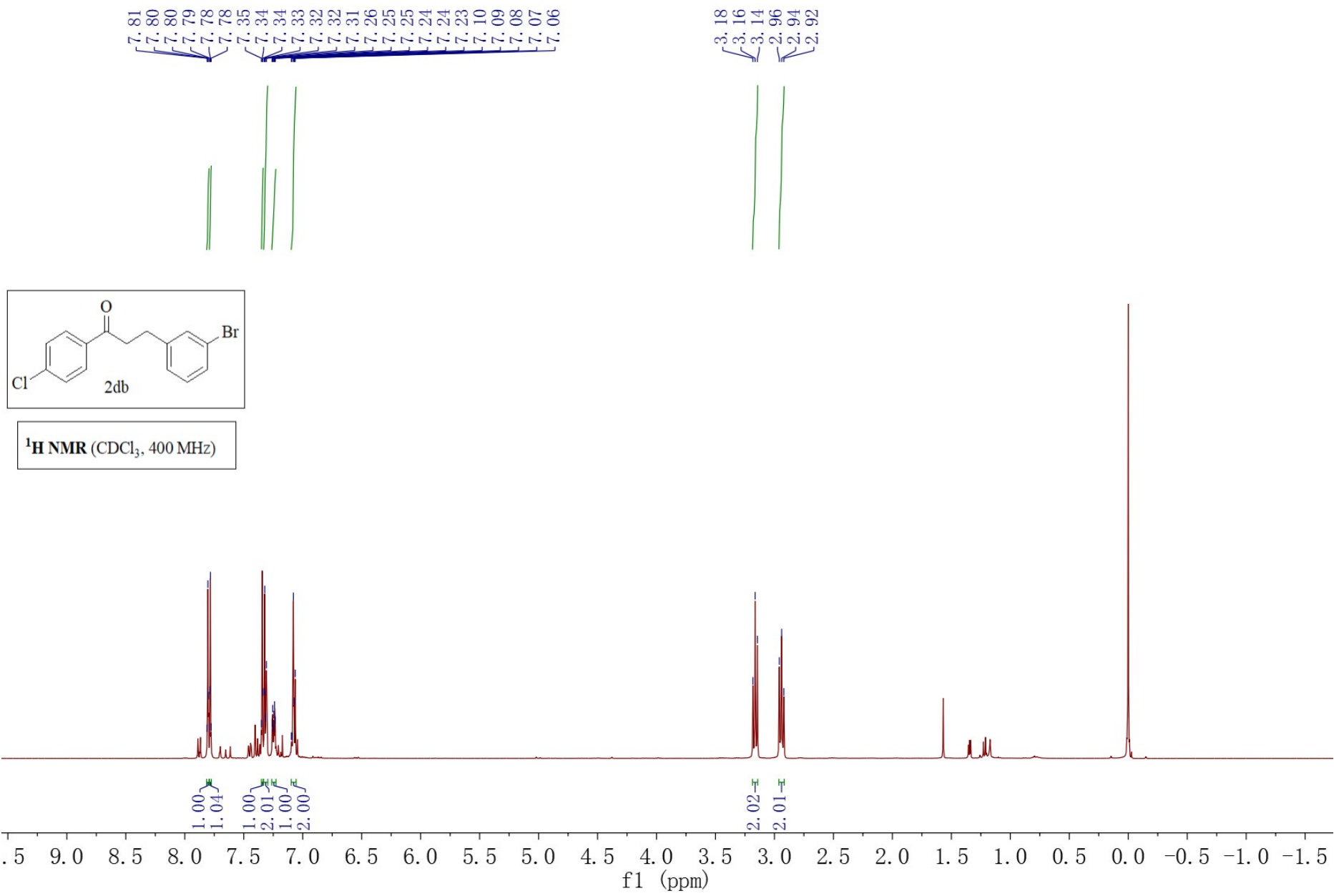


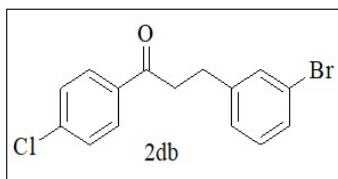




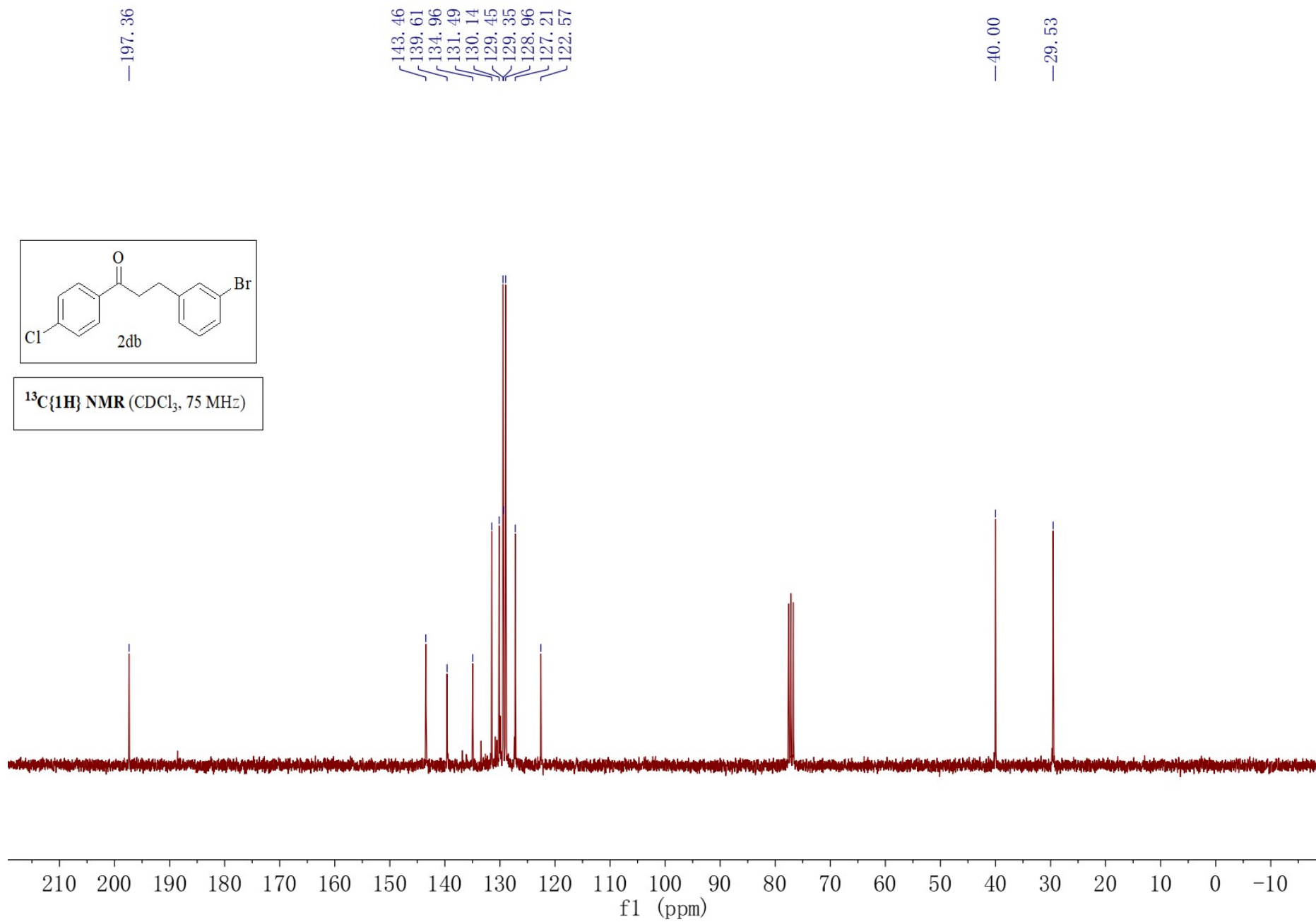
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

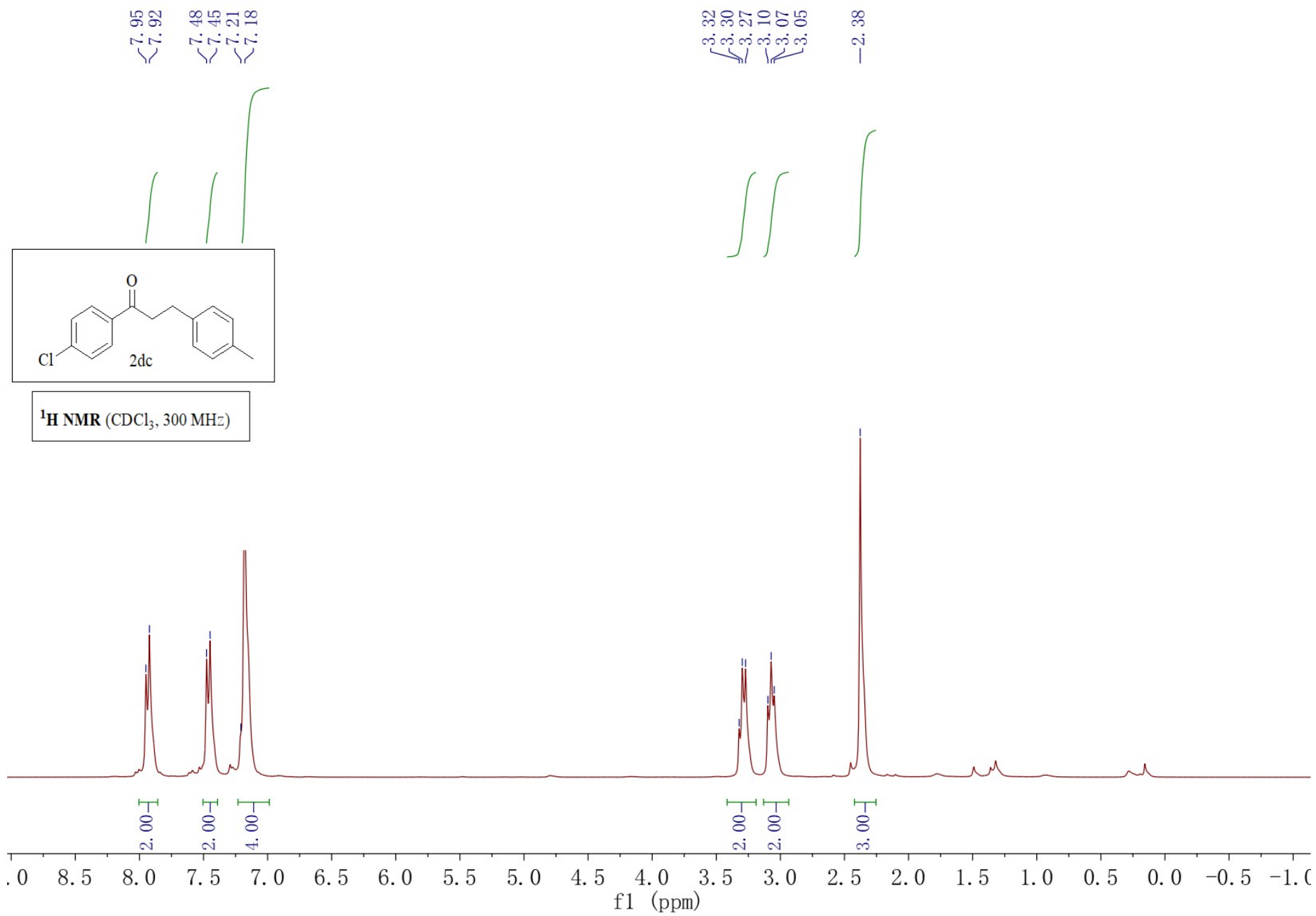


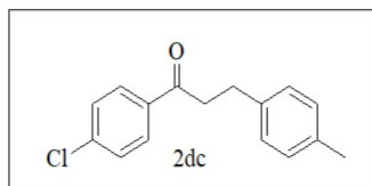




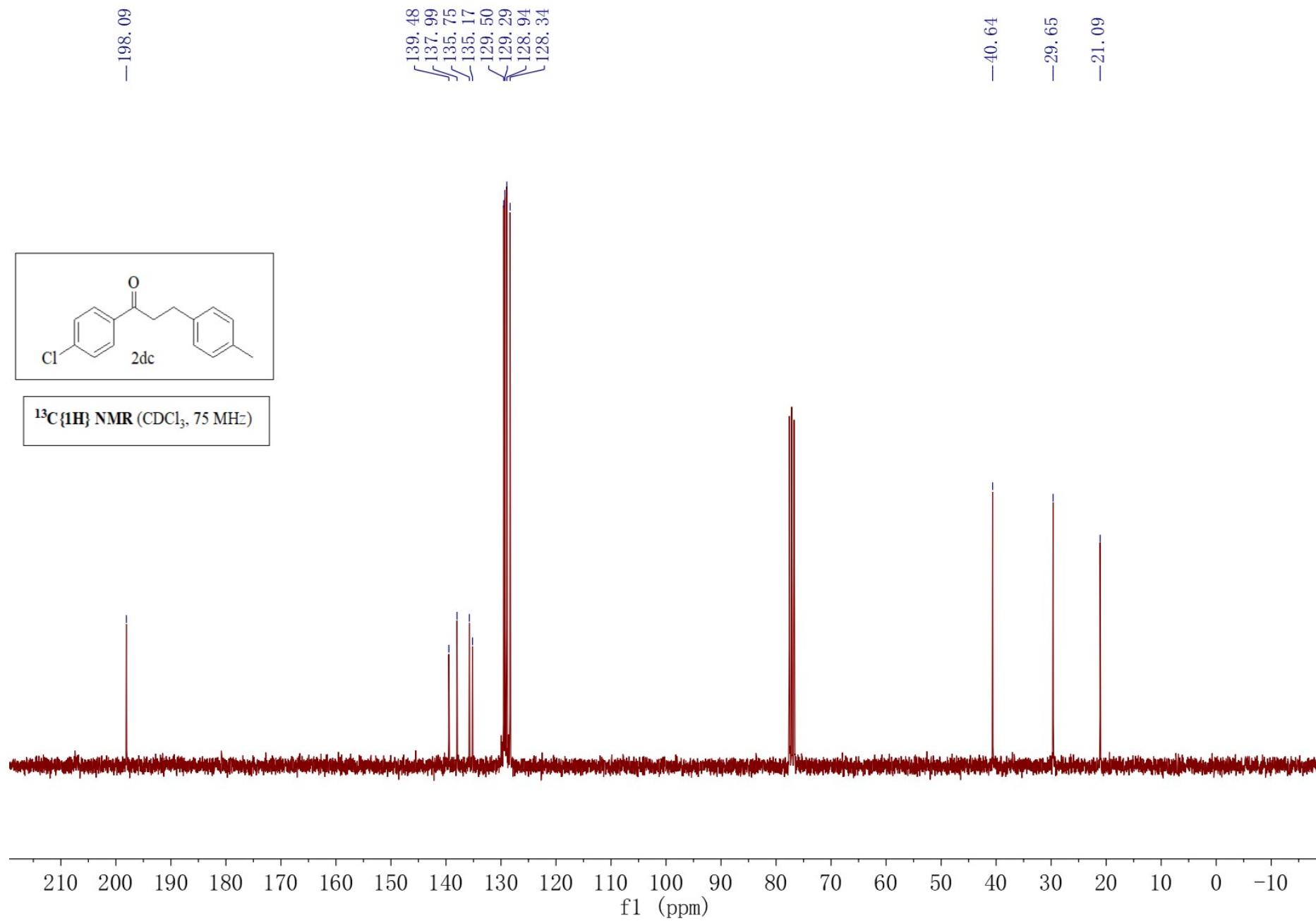
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

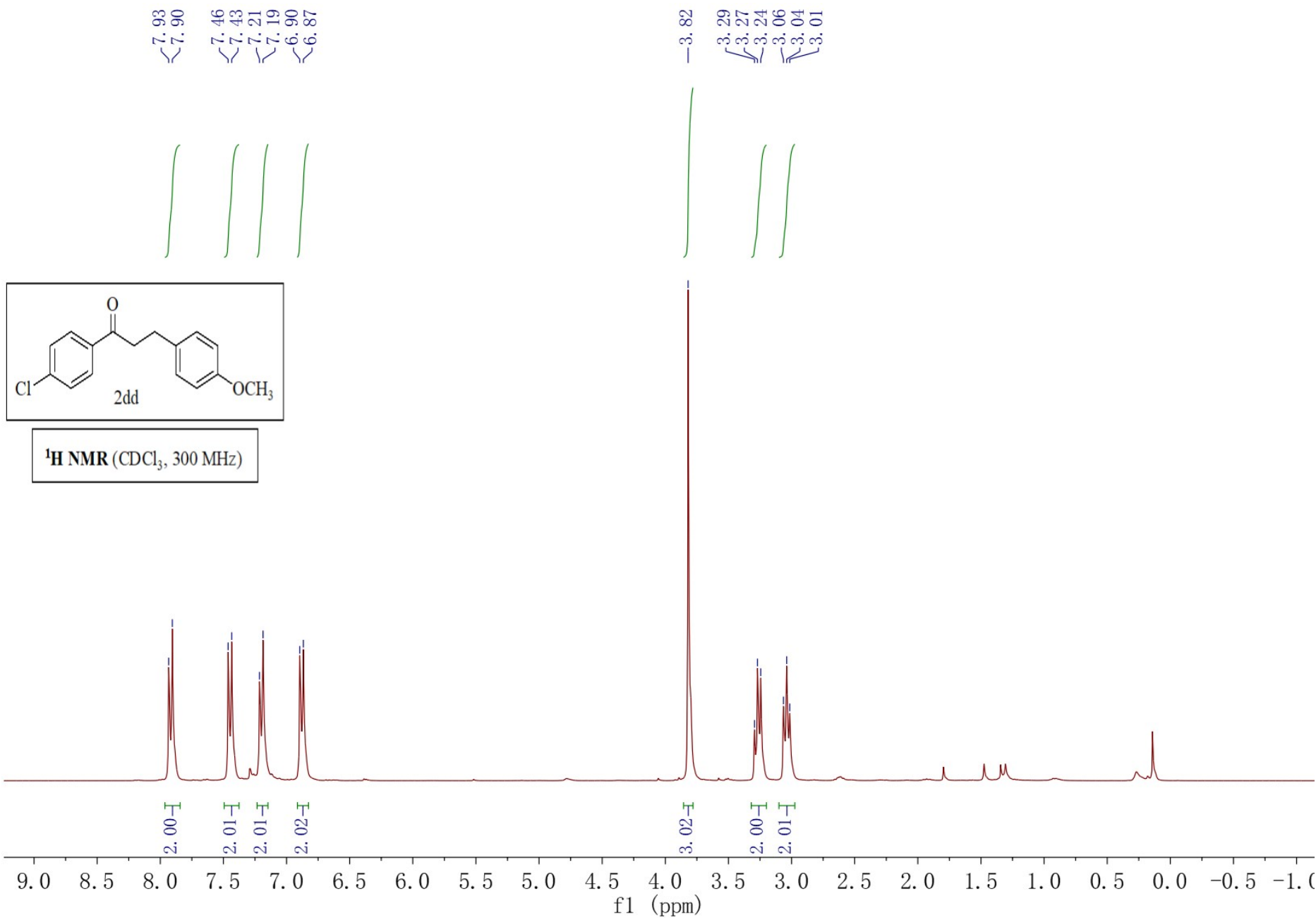


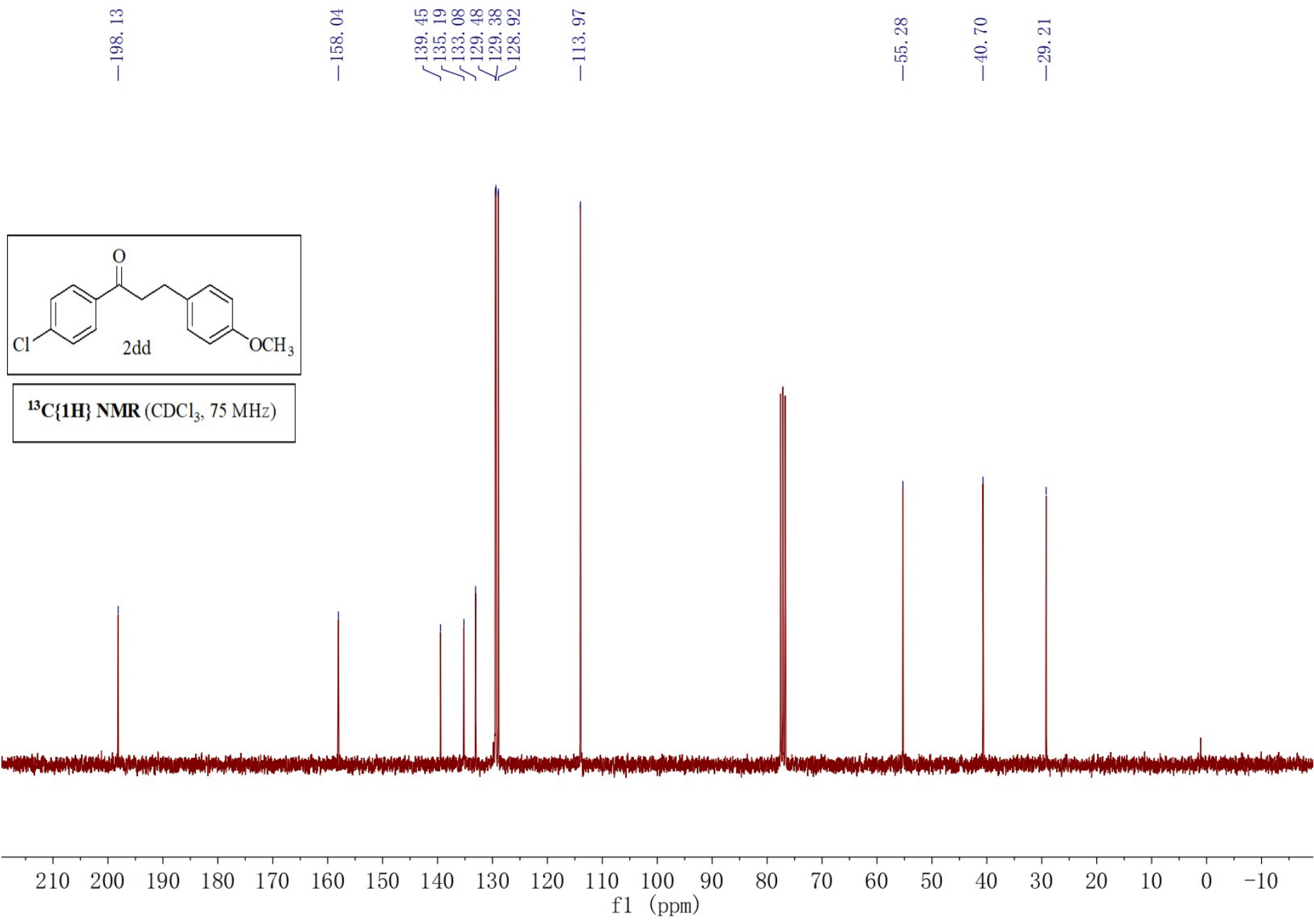


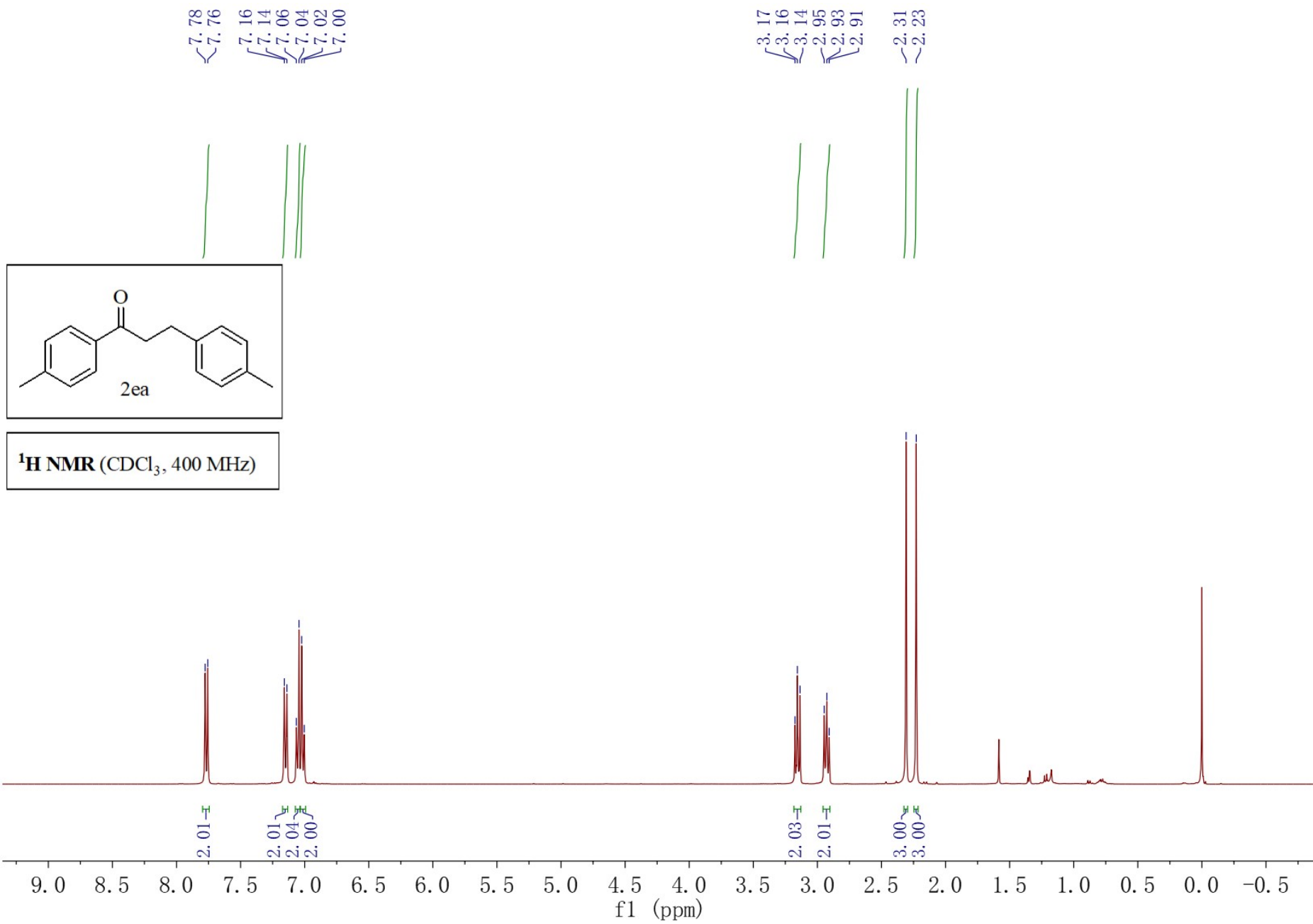


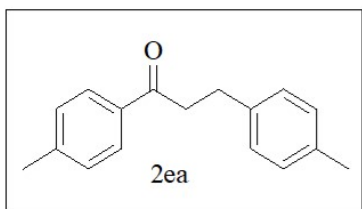
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



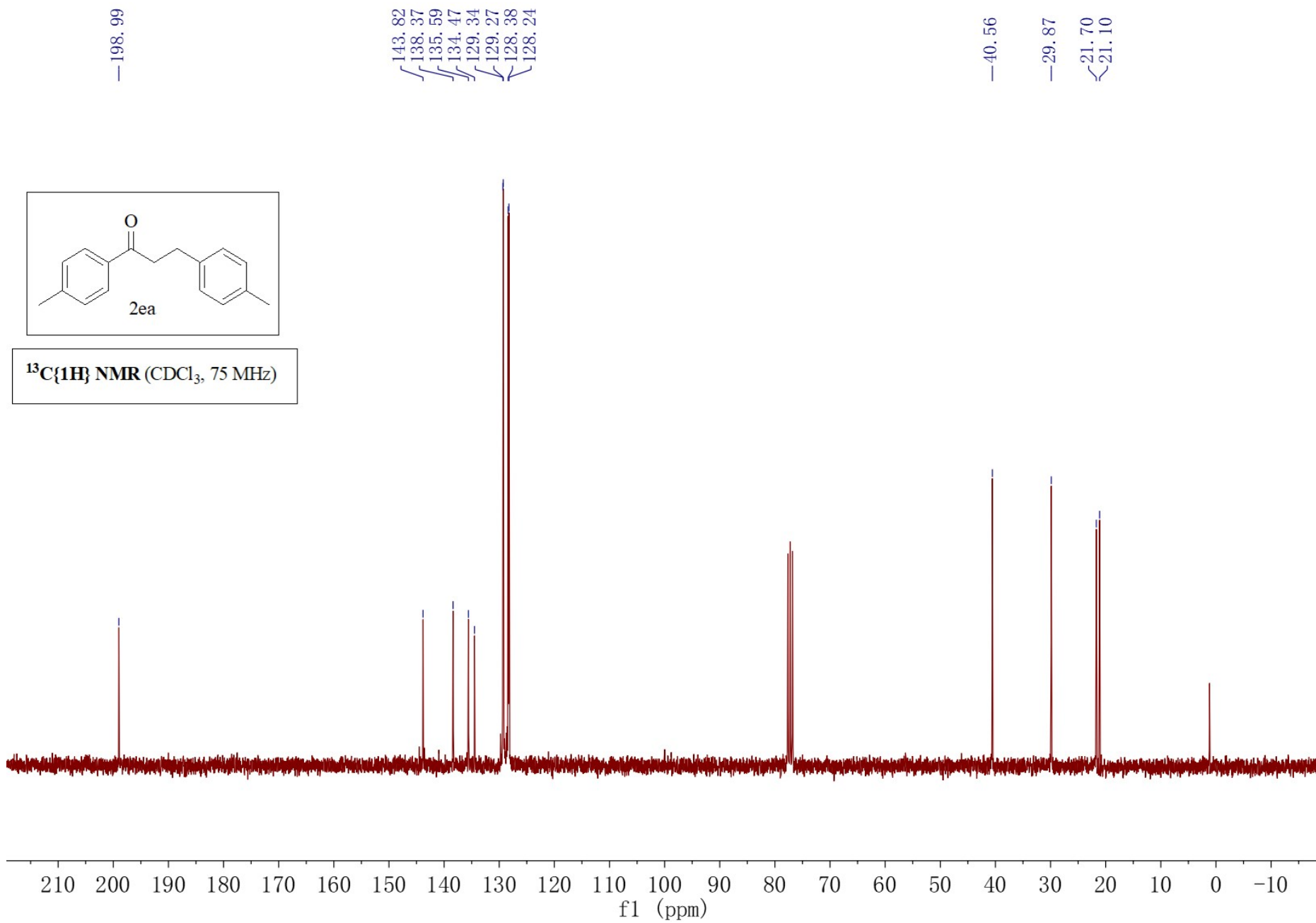


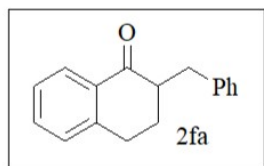




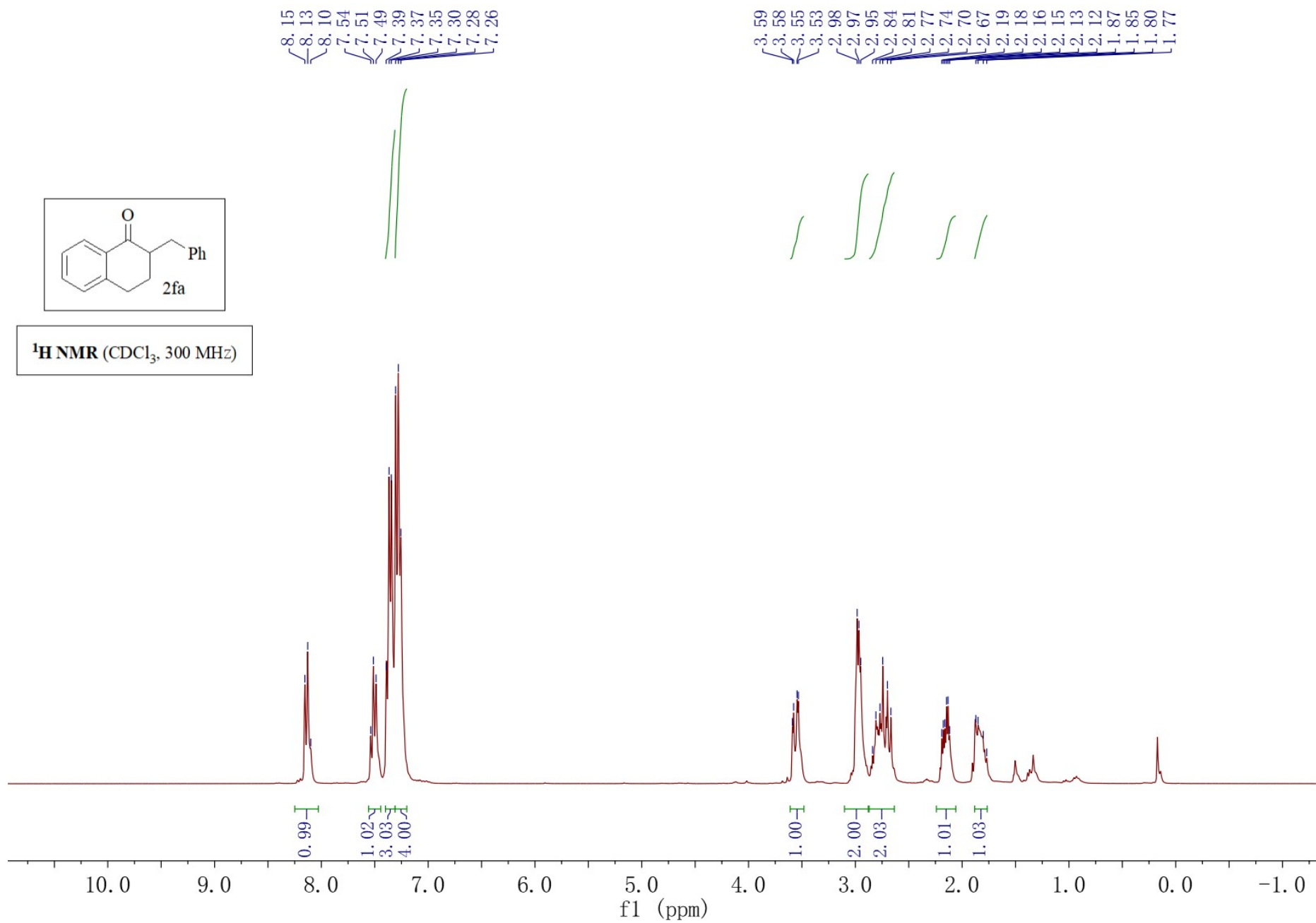


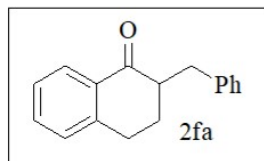
$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)



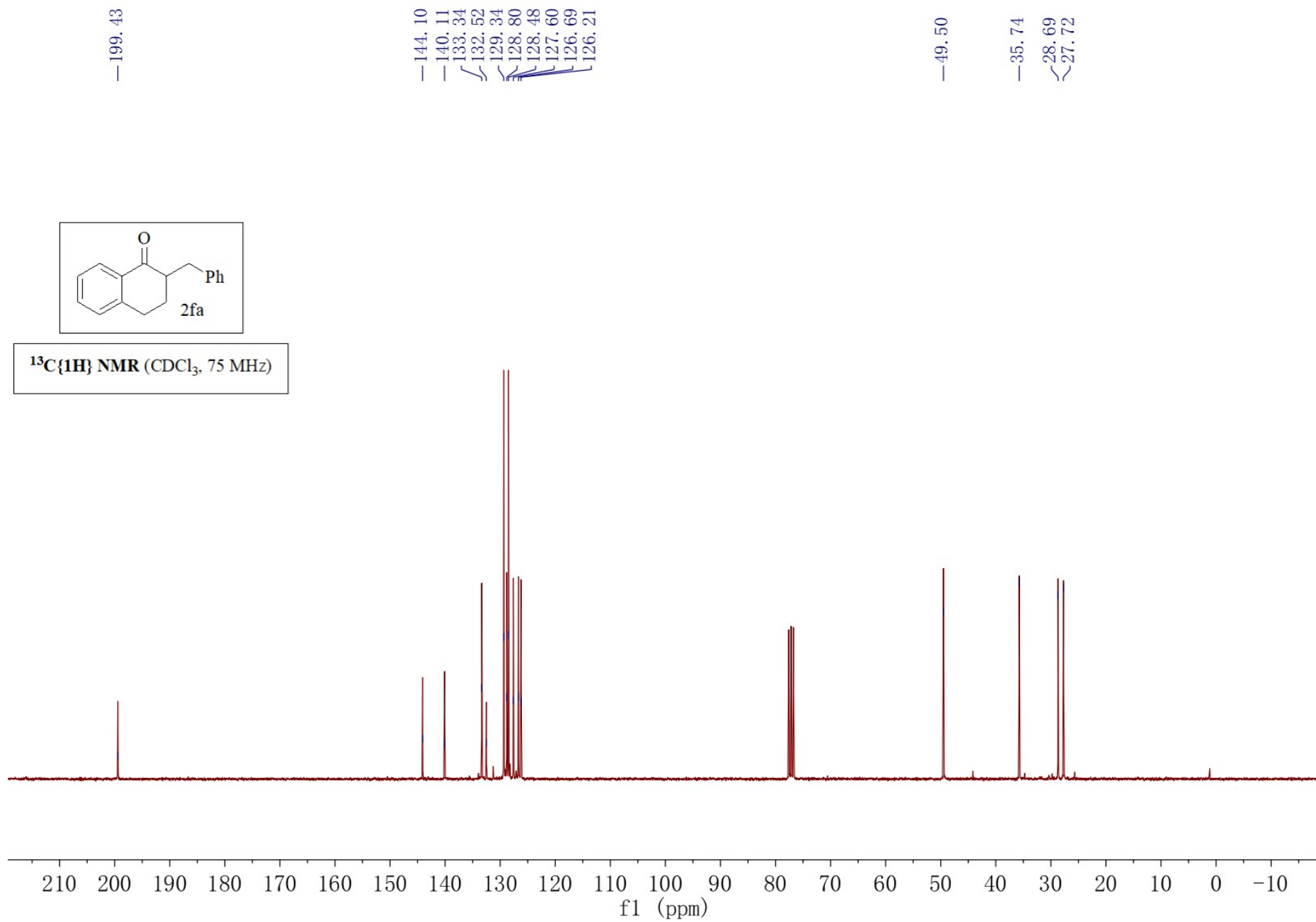


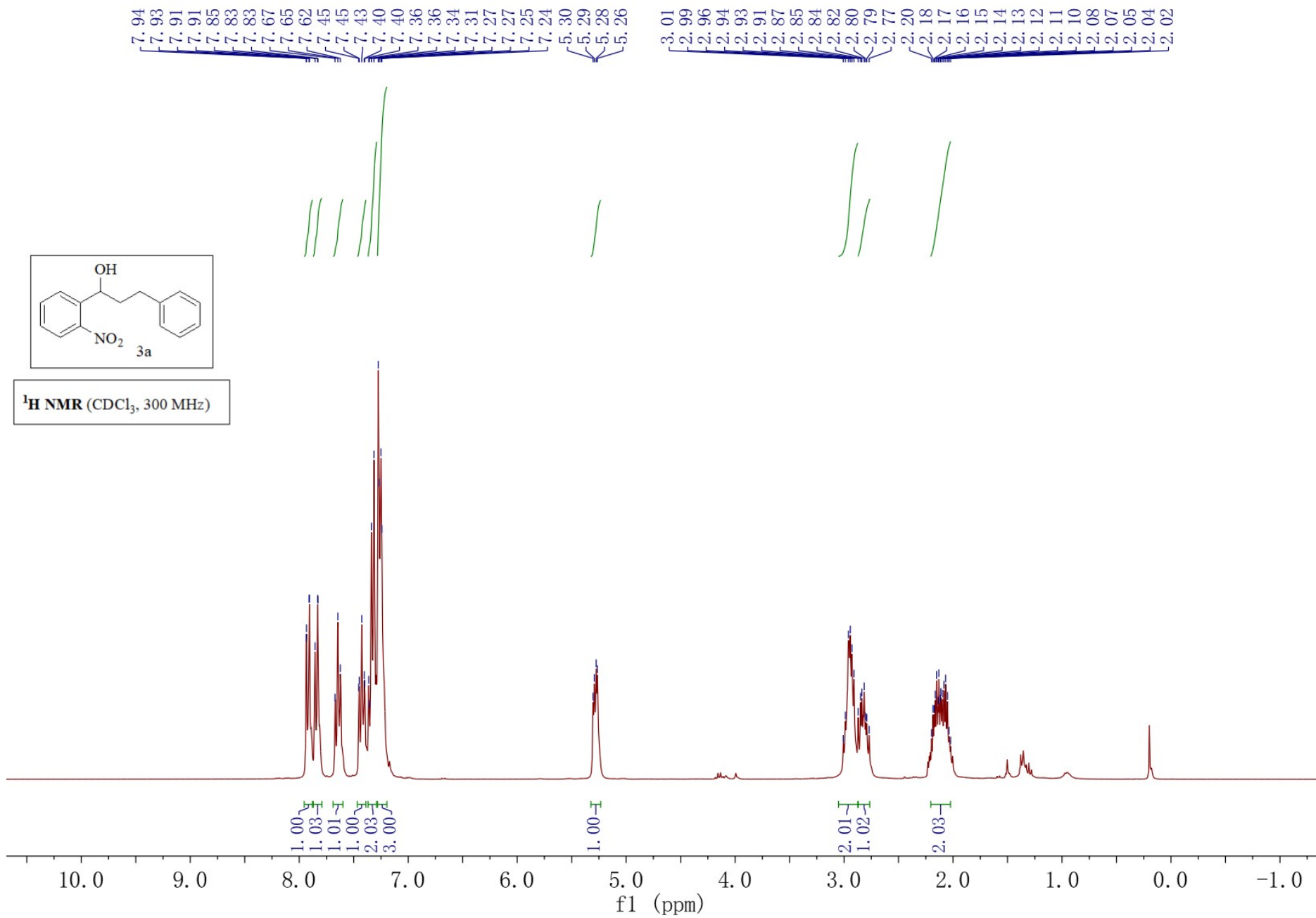
¹H NMR (CDCl₃, 300 MHz)

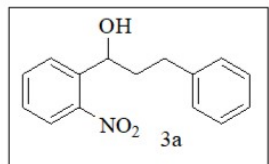




$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)







$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 75 MHz)

