Supporting Information for:

Ru-Mg promoted reductive cross-coupling of allyl bromides and alkenes to 1,7-Octadienes with an all-carbon quaternary center

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CONTENTS

1. General experimental details and materials

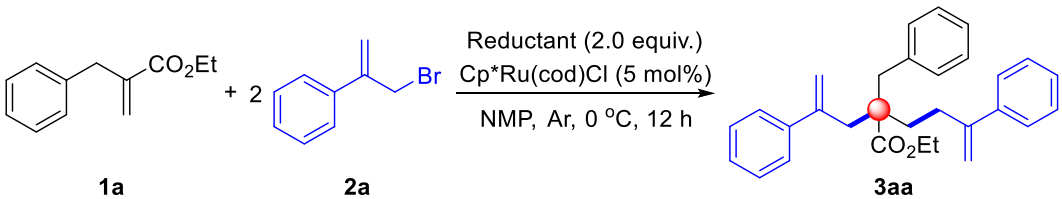
All non-aqueous reactions and manipulations were using standard Schlenk techniques. All solvents before use were dried and degassed by standard methods and stored under argon atmosphere. All reactions were monitored by TLC with silica gel-coated plates. For chromatography, 200-300 mesh silica gel (Qingdao, China) was employed. ^{1}H NMR, ^{13}C NMR spectra were measured in $CDCl_3$ and recorded on Bruker Avance III 500 MHz, Bruker Avance III HD (600 MHz, Bruker BioSpin, Switzerland). Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of $CDCl_3$ (7.26), to the carbon resonance of $CDCl_3$ (77.16). Coupling constants (*J*) were reported in Hertz (Hz) and referred to apparent peak multiplications. The term m, q, t, d, s referred to multiplet, quartet, triplet, doublet, singlet respectively. GC-MS spectra were recorded on a ThermoFisher Scientific ISQ 7000 Series GC-MS system. High resolution mass spectra were recorded on a high-resolution mass spectrometer using electrospray ionization (ESI) techniques.

The electron-deficient olefins **1** and allyl bromides **2** were known compounds and synthesized according to the reported methods.[1-14] Unless extra specified, the used catalysts, reductants, ligands, and solvents etc. were all purchased from Energy.

2. Optimization of the reaction conditions

Ethyl 2-benzylacrylate **1a** (38.0 mg, 0.20 mmol), (3-bromoprop-1-en-2-yl)benzene **2a** (118.2 mg, 0.60 mmol), catalyst (x mol%), reductant (x mol%), ligand (x mol%) and solvent (2.0 mL) were added in a flame-dried Young-type tube. The mixture was degassed by the freeze-thaw method, and then stirred under argon at designed temperature for designed hours. Upon completion of the reaction, the reaction mixture was treated with saturated $NH_4Cl_{(aq.)}$ (25 mL), extracted with ethyl acetate (3 x 10 mL), washed with saturated $NaCl_{(aq.)}$ (3 x 10 mL), and dried over Na_2SO_4. Then, the solvent (ethyl acetate) was removed in vacuo and the residue was purified by flash column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/100) to afford the desired product **3aa**.

Table S1. Screening of the reductant [a]

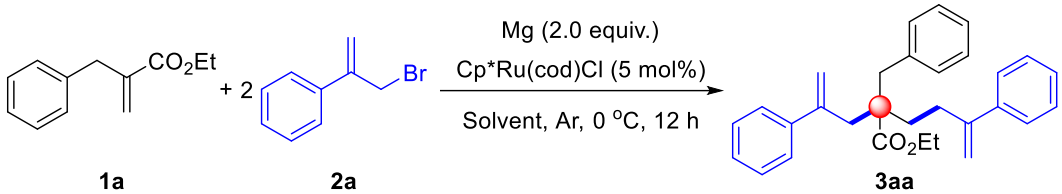


1a **2a** **3aa**

Entry	Reductant (2.0 equiv.)	Yield (%)
1	**Mg**	**61**
2	Fe	NR
3	Cu	NR
4	Mn	NR
5	In	Trace
6	Ni	NR
7	Zn	Trace
8	V	NR
9	Sn	NR
10	B_2Pin_2	NR

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), reductant (0.4 mmol, 2.0 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields, NR = No Reaction.

Table S2. Screening of the solvent [a]

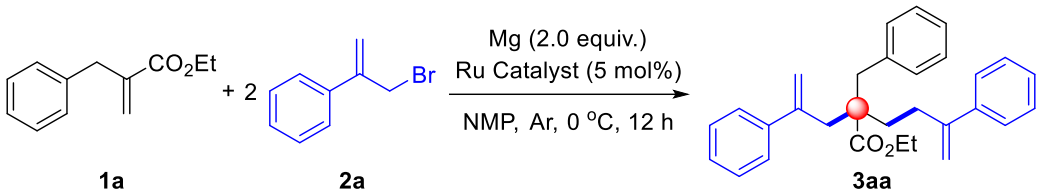


1a **2a** **3aa**

Entry	Solvent (2.0 mL)	Yield (%)
1	**NMP**	**61**
2	DMF	20
3	DMAc	42
4	NEt_3	NR
5	EtOAc	NR
6	Toluene	NR
7	THF	ND
8	DCM	NR
9	DMSO	ND
10	DCE	NR
11	MeCN	NR

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (0.4 mmol, 2.0 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and solvent (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields, NR = No reaction, ND=Not detected.

Table S3. Screening of [Ru] catalyst [a]

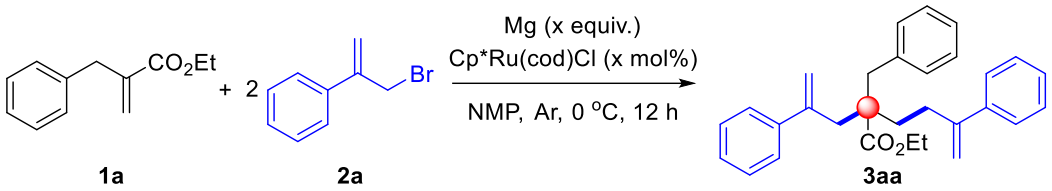


1a **2a** **3aa**

Entry	Ru Catalyst (5 mol%)	Yield (%)
1	**Cp*Ru(cod)Cl**	**61**
2	$[Ru(p\text{-cymene})]_2Cl_2$	32
3	$CpRu(PPh_3)_2Cl$	41
4	$[Ru(bpy)_3]Cl_2 \cdot 6H_2O$	46
5	$Ru(PPh_3)_3Cl_2$	45
6	$RuCl_3 \cdot xH_2O$	41
7	$(PPh_3)_3Ru(CO)(Cl)H$	39
8	$Cp^*Ru(PPh_3)_2Cl$	54

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (0.4 mmol, 2.0 equiv.), [Ru] catalyst (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields.

Table S4. Screening of the loading [a]

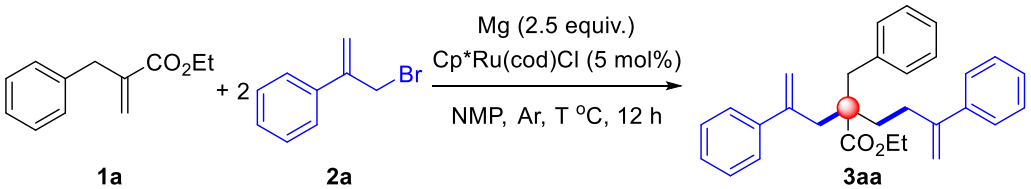


1a **2a** **3aa**

Entry	Cp*Ru(cod)Cl (x mol%)	Mg (x equiv.)	Yield (%)
1	1.0	2.0	50
2	2.5	2.0	58
3	**5.0**	**2.0**	**61**
4	7.5	2.0	60
5	10	2.0	61
6	5.0	1.0	43
7	5.0	1.5	42
8	5.0	2.0	63
9	**5.0**	**2.5**	**69**
10	5.0	3.0	65

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (x mmol, x equiv.), Cp*Ru(cod)Cl (x mmol, x mol%), and NMP (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields.

Table S5. Screening of temperature [a]

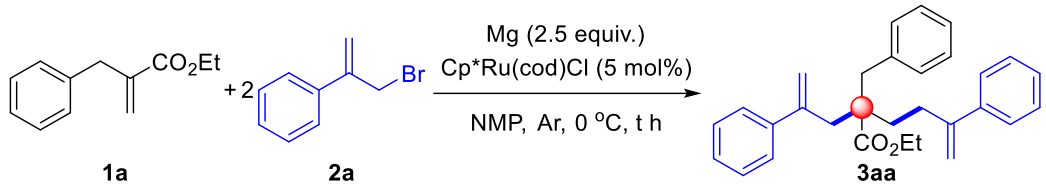


1a **2a** **3aa**

Entry	T (°C)	Yield (%)
1	r.t.	56
2	**0**	**69**
3	-10	60
4	-20	53

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (0.5 mmol, 2.5 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at T °C for 12 h, isolated yields.

Table S6. Screening of time [a]

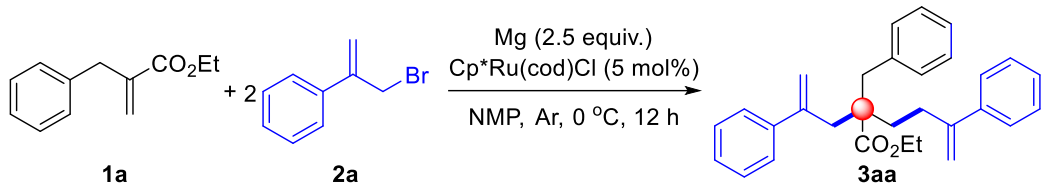


1a **2a** **3aa**

Entry	t (h)	Yield (%)
1	6	57
2	**12**	**69**
3	18	66
4	24	62

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (0.5 mmol, 2.5 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at 0 °C for t h, isolated yields.

Table S7. Screening loading of 2a [a]

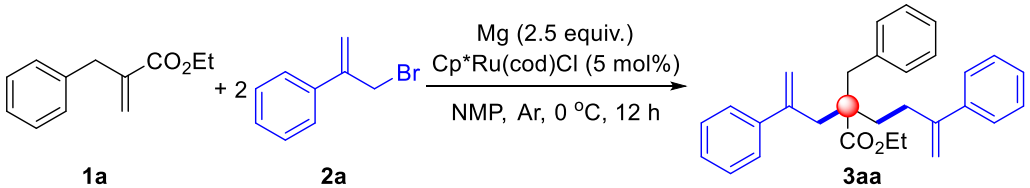


1a **2a** **3aa**

Entry	**2a** (x equiv.)	Yield (%)
1	2.0	48
2	2.5	66
3	**3.0**	**69**
4	3.5	69
5	4.0	64

[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (x mg, x mmol), Mg (0.5 mmol, 2.5 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields.

Table S8. Control experiment [a]



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

Entry	Deviation from "standard" conditions	Yield (%)
1	No Cp*Ru(cod)Cl	41
2	No Mg	NR

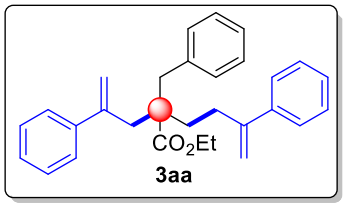
[a] Reaction condition: **1a** (38.0 mg, 0.2 mmol), **2a** (118.2 mg, 0.6 mmol), Mg (0.4 mmol, 2.0 equiv.), Cp*Ru(cod)Cl (0.01 mmol, 5 mol%), and NMP (2.0 mL) under the Ar at 0 °C for 12 h, isolated yields. NR = No Reaction.

3. General procedure

Electron-deficient olefins **1** (0.20 mmol), allyl bromides **2** (0.60 mmol), Cp*Ru(cod)Cl (0.01 mmol, 5.0 mol%), Mg (0.5 mmol, 2.5 equiv.) and NMP (2.0 mL) were added to a flame-dried Young-type tube. The mixture was stirred under Ar at 0 °C for 12 h. After rising to room temperature, the reaction mixture was treated with saturated $NH_4Cl_{(aq.)}$ (25 mL), extracted with ethyl acetate (3 x 10 mL), washed with saturated $NaCl_{(aq.)}$ (3 x 10 mL), and dried over Na_2SO_4. Then, the solvent (ethyl acetate) was removed in vacuo and the residue was purified by flash column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/40-1/200) to afford the desired products **3**.

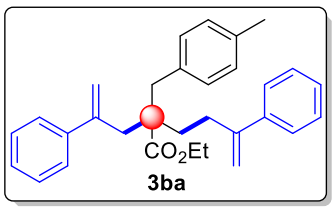
4. Experimental characterization data for products

Ethyl 2-benzyl-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3aa):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (58.6 mg, 69% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.18 (m, 13H), 7.08-7.06 (m, 2H), 5.24 (d, *J* = 1.0 Hz 1H), 5.16 (s, 1H), 5.12 (s, 1H), 4.85 (s, 1H), 3.78 (q, *J* = 7.0 Hz, 2H), 3.12 (d, *J* = 14.0 Hz, 1H), 2.94-2.91 (m, 2H), 2.81 (d, *J* = 15.0 Hz, 1H), 2.46-2.42 (m, 2H), 1.68-1.64 (m, 2H), 1.11 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.67, 148.33, 145.88, 142.87, 141.30, 137.54, 130.15, 128.18, 128.13, 128.09, 127.32, 127.28, 126.79, 126.44, 126.15, 117.26, 112.22, 60.36, 50.42, 41.56, 41.03, 32.20, 29.85, 13.98. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{30}H_{33}O_2^+$ 425.2475; found 425.2470.

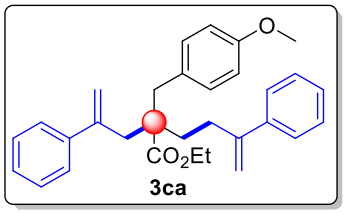
Ethyl 2-(4-methylbenzyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ba):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish solid (52.5 mg, 60% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31-7.23 (m, 11H), 7.01 (d, *J* = 7.8 Hz,

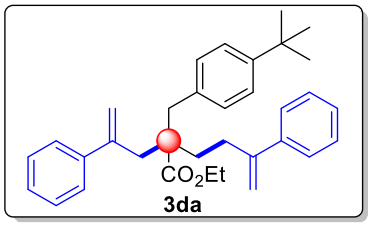
2H), 6.97 (d, *J* = 7.8 Hz, 2H), 5.24 (s, 1H), 5.16 (s, 1H), 5.11 (s, 1H), 4.86 (s, 1H), 3.77 (q, *J* = 7.2 Hz, 2H), 3.08 (d, *J* = 13.8 Hz, 1H), 2.94-2.88 (m, 2H), 2.79 (d, *J* = 14.4 Hz, 1H), 2.49-2.40 (m, 2H), 2.29 (s, 3H), 1.66 (t, *J* = 8.4 Hz, 2H), 1.12 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.73, 148.37, 145.92, 142.91, 141.34, 135.89, 134.34, 130.01, 128.79, 128.15, 128.10, 127.28, 127.25, 126.79, 126.16, 117.17, 112.17, 60.30, 50.41, 41.53, 40.54, 32.14, 29.82, 21.01, 13.98. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{31}H_{35}O_2^+$ 439.2632; found 439.2634.

Ethyl 2-(4-methoxybenzyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ca):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish solid (42.5 mg, 47% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.22 (m, 10H), 6.99-6.97 (m, 2H), 6.74-6.72 (m, 2H), 5.24 (d, *J* = 1.5 Hz, 1H), 5.16 (d, *J* = 1.0 Hz, 1H), 5.11 (d, *J* = 1.0 Hz, 1H), 4.86 (d, *J* = 1.0 Hz, 1H), 3.78-3.74 (m, 5H), 3.05 (d, *J* = 13.5 Hz, 1H), 2.93-2.85 (m, 2H), 2.79 (d, *J* = 14.5 Hz, 1H), 2.49-2.39 (m, 2H), 1.66-1.63 (m, 2H), 1.12 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl3) δ 175.85, 158.34, 148.51, 146.05, 143.01, 141.47, 131.20, 129.59, 128.28, 128.22, 127.41, 127.39, 126.90, 126.29, 117.27, 113.61, 112.31, 60.42, 55.31, 50.60, 41.61, 40.22, 32.20, 29.95, 14.12. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{31}H_{35}O_3^+$ 455.2581; found 455.2591.

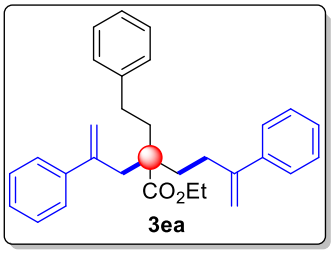
Ethyl 2-(4-(tert-butyl)benzyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3da):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (55.1 mg, 57% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.21 (m, 12H), 7.01 (d, *J* = 8.5 Hz, 2H), 5.24 (d, *J* = 1.0 Hz, 1H), 5.16 (d, *J* = 1.0 Hz, 1H), 5.12 (d, *J* = 0.5 Hz, 1H), 4.85 (d, *J* = 1.0 Hz, 1H), 3.78 (q, *J* = 7.0 Hz, 2H), 3.08 (d, *J* = 14.0 Hz, 1H), 2.95-2.87 (m, 2H), 2.81 (d, *J* = 14.5 Hz, 1H), 2.45-2.42 (m, 2H), 1.69-1.65 (m, 2H), 1.28 (s, 9H), 1.11 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.87,

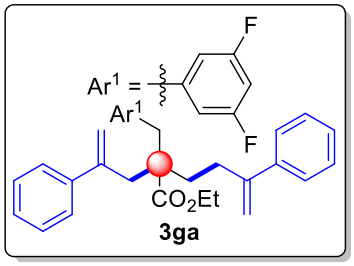
149.32, 148.51, 146.06, 143.06, 141.43, 134.52, 129.91, 128.26, 128.22, 127.39, 127.36, 126.93, 126.28, 125.08, 117.35, 112.29, 60.41, 50.56, 41.62, 40.71, 34.48, 32.36, 31.51, 29.98, 14.08. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{34}H_{41}O_2^+$ 481.3101; found 481.3101.

Ethyl 2-phenethyl-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ea):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (55.3 mg, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33-7.21 (m, 12H), 7.16-7.13 (m, 1H), 6.97 (d, *J* = 7.0 Hz, 2H), 5.21-5.20 (m, 2H), 5.12 (s, 1H), 4.92 (s, 1H), 3.82-3.78 (m, 2H), 2.94 (q, *J* = 14.0 Hz, 2H), 2.39-2.32 (m, 4H), 1.91-1.78 (m, 3H), 1.73-1.67 (m, 1H), 1.17 (t, *J*= 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.96, 148.41, 146.25, 143.01, 142.25, 141.25, 128.45, 128.41, 128.39, 128.30, 127.54, 127.45, 127.04, 126.23, 125.94, 117.90, 112.52, 60.42, 49.79, 40.43, 36.03, 34.00, 30.56, 30.18, 14.28. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{31}H_{35}O_2^+$ 439.2632; found 439.2612.

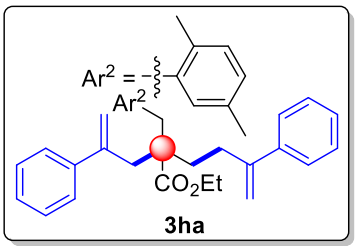
Ethyl 2-(3,5-difluorobenzyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ga):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (45.6 mg, 50% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29-7.24 (m, 10H), 6.64-6.58 (m, 3H), 5.25 (d, *J* = 1.0 Hz, 1H), 5.17 (d, *J* = 1.0 Hz, 1H), 5.11 (d, *J* = 1.0 Hz, 1H), 4.87 (d, *J* = 1.0 Hz, 1H), 3.82-3.77 (m, 2H), 3.06 (d, *J* = 14.5 Hz, 1H), 2.91-2.81 (m, 3H), 2.43-2.39 (m, 2H), 1.67-1.63 (m, 2H), 1.14 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.30, 163.84 (d, *J* = 13.0 Hz), 161.87 (d, *J* = 13.0 Hz), 148.23, 145.75, 142.74, 141.68 (t, *J* = 9.1 Hz), 141.25, 128.38, 128.33, 127.60, 127.55, 126.90, 126.24, 117.80, 113.16 (d, *J* = 5.5 Hz), 113.00 (d, *J* = 5.7 Hz), 112.62, 102.32 (t, *J* = 25.3 Hz), 60.77, 50.57, 41.66, 40.84, 32.68, 30.05, 14.09. HRMS

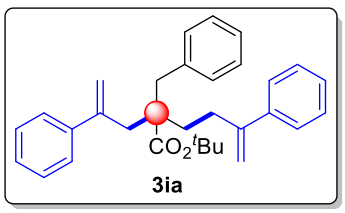
(ESI) m/z: $[M+Na]^+$ calcd for $C_{30}H_{30}NaO_2^+$ 483.2106; found 483.2112.

Ethyl 2-(2,5-dimethylbenzyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ha):



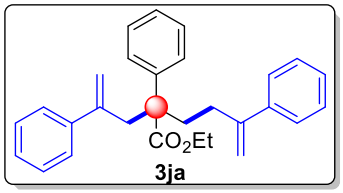
The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish solid (45.2 mg, 50% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.21 (m, 8H), 7.15-7.13 (m, 2H), 6.99 (d, *J* = 7.5 Hz, 1H), 6.90 (d, *J* = 7.5 Hz, 1H), 6.80 (s, 1H), 5.24 (d, *J* = 1.5 Hz, 1H), 5.14 (d, *J* = 1.0 Hz, 1H), 5.06 (d, *J* = 1.0 Hz, 1H), 4.78 (d, *J* = 1.0 Hz, 1H), 3.88-3.80 (m, 2H), 3.07 (d, *J* = 14.0 Hz, 1H), 2.95-2.91 (m, 2H), 2.86 (d, *J* = 15.0 Hz, 1H), 2.36-2.31 (m, 2H), 2.19 (s, 3H), 2.13 (s, 3H), 1.77-1.71 (m, 2H), 1.10 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.35, 148.45, 146.06, 143.15, 141.21, 136.19, 134.91, 133.92, 130.40, 130.10, 128.29, 128.27, 127.40, 127.33, 126.92, 126.82, 126.15, 117.81, 112.14, 60.53, 50.22, 40.96, 38.37, 33.05, 30.04, 21.14, 19.84, 14.02. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{32}H_{37}O_2^+$ 453.2788; found 453.2789.

Tert-butyl 2-benzyl-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3ia):



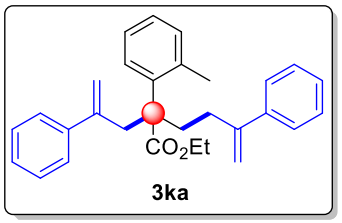
The title compound was prepared according to the general procedure and purified by column chromatography to give a colorless oil (40.5 mg, 45% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32-7.23 (m, 10H), 7.18-7.16 (m, 3H), 7.10-7.08 (m, 2H), 5.30 (s, 1H), 5.16 (d, *J* = 1.0 Hz, 1H), 5.14 (d, *J* = 1.0 Hz, 1H), 4.84 (d, *J* = 1.0 Hz, 1H), 3.01 (s, 2H), 2.79 (q, *J*=15.5 Hz, 2H), 2.51-2.39 (m, 2H), 1.66-1.59 (m, 2H), 1.38 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.27, 148.56, 145.92, 143.72, 141.54, 137.93, 130.46, 128.37, 128.29, 128.09, 127.40, 127.39, 126.65, 126.41, 126.29, 116.54, 112.27, 80.95, 50.53, 40.63, 40.33, 34.10, 30.20, 28.13. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_2^+$ 475.2608; found 475.2607.

Ethyl 2,5-diphenyl-2-(2-phenylallyl)hex-5-enoate (3ja):



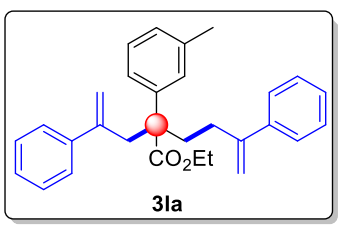
The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (43.1 mg, 53% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25-7.12 (m, 15H), 5.12 (d, J = 4.5 Hz, 2H), 4.84 (s, 1H), 4.69 (s, 1H), 3.98-3.92 (m, 1H), 3.83-3.76 (m, 1H), 3.43 (d, J = 13.5 Hz, 1H), 3.24 (d, J = 13.5 Hz, 1H), 2.22-2.10 (m, 2H), 2.07-2.01 (m, 2H), 1.07 (t, J = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.29, 147.99, 145.50, 142.90, 142.57, 140.77, 128.21, 128.10, 127.33, 127.18, 126.90, 126.70, 126.64, 126.03, 118.37, 112.29, 60.75, 54.49, 40.00, 33.48, 30.10, 13.98. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{29}H_{30}NaO_2^+$ 433.2138; found 433.2137.

Ethyl 5-phenyl-2-(2-phenylallyl)-2-(*o*-tolyl)hex-5-enoate (3ka):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (50.0 mg, 59% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24-7.00 (m, 14H), 5.10 (d, *J* = 8.4 Hz, 2H), 4.75 (s, 1H), 4.68 (s, 1H), 4.03-3.97 (m, 1H), 3.85-3.80 (m, 1H), 3.34-3.29 (m, 2H), 2.18-2.17 (m, 1H), 2.13 (s, 3H), 2.02-1.99 (m, 2H), 1.33-1.28 (m, 1H), 1.13 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 176.15, 147.89, 145.52, 142.92, 140.85, 140.39, 136.04, 131.82, 128.26, 128.06, 127.39, 127.20, 127.08, 126.73, 126.04, 125.49, 118.80, 112.39, 60.91, 53.77, 38.66, 32.77, 30.16, 20.46, 14.17. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{30}H_{32}NaO_2^+$ 447.2295; found 447.2291.

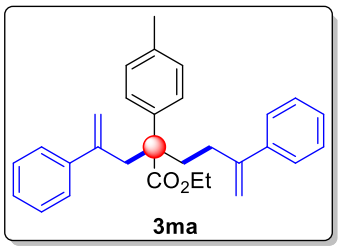
Ethyl 5-phenyl-2-(2-phenylallyl)-2-(*m*-tolyl)hex-5-enoate (3la):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (33.2 mg, 40% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25-7.12 (m, 11H), 6.98-6.93 (m, 3H), 5.13-5.12 (m, 2H), 4.89 (s, 1H), 4.69 (s, 1H), 3.98-3.93 (m, 1H), 3.82-3.77 (m, 1H), 3.41 (d,

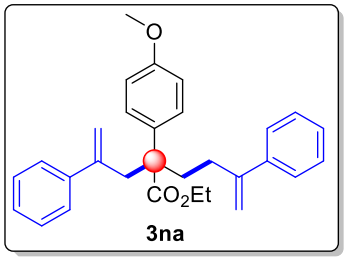
J = 13.2 Hz, 1H), 3.24 (d, *J* = 13.2 Hz, 1H), 2.27 (s, 3H), 2.21-2.10 (m, 2H), 2.03-2.00 (m, 2H), 1.09 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.45, 148.08, 145.71, 142.96, 142.47, 140.80, 137.70, 128.24, 128.10, 128.06, 127.48, 127.39, 127.36, 127.17, 126.91, 126.08, 123.70, 118.30, 112.33, 60.76, 54.36, 39.98, 33.47, 30.12, 21.71, 14.02. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{30}H_{32}NaO_2^+$ 447.2295; found 447.2291.

Ethyl 5-phenyl-2-(2-phenylallyl)-2-(*p*-tolyl)hex-5-enoate (3ma):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (37.3 mg, 45% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25-7.19 (m, 8H), 7.15-7.13 (m, 2H), 7.05 (s, 4H), 5.12-5.11 (m, 2H), 4.84 (s, 1H), 4.67 (s, 1H), 3.97-3.92 (m, 1H), 3.81-3.76 (m, 1H), 3.41 (d, *J* = 13.2 Hz, 1H), 3.21 (d, *J* = 13.8 Hz, 1H), 2.30 (s, 3H), 2.19-2.10 (m, 2H), 2.03-1.99 (m, 2H), 1.09 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.50, 148.10, 145.62, 143.02, 140.89, 139.57, 136.24, 128.94, 128.23, 128.11, 127.34, 127.14, 126.96, 126.54, 126.09, 118.32, 112.25, 60.76, 54.18, 40.12, 33.53, 30.13, 21.08, 14.04. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{30}H_{32}NaO_2^+$ 447.2295; found 447.2291.

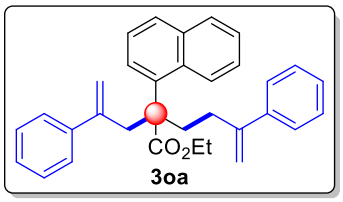
Ethyl 2-(4-methoxyphenyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3na):



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (40.2 mg, 46% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24-7.15 (m, 10H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.78 (d, *J* = 9.0 Hz, 2H), 5.12 (d, *J* = 4.2 Hz, 2H), 4.85 (s, 1H), 4.69 (s, 1H), 3.98-3.92 (m, 1H), 3.82-3.80 (m, 1H), 3.77 (s, 3H), 3.40 (d, *J* = 13.8 Hz, 1H), 3.20 (d, *J* = 13.8 Hz, 1H), 2.20-2.11 (m, 2H), 2.03-1.98 (m, 2H), 1.09 (t, *J* = 7.2 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.64, 158.26, 148.21, 145.73, 143.10, 140.98, 134.77, 128.35, 128.22, 127.84, 127.47, 127.26, 127.04, 126.20,

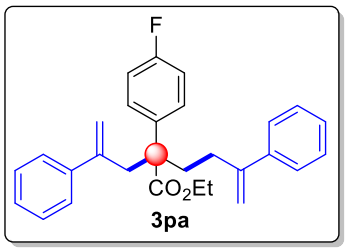
118.42, 113.64, 112.40, 60.87, 55.43, 53.91, 40.32, 33.67, 30.28, 14.16. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{30}H_{33}O_2^+$ 441.2424; found 441.2421.

Ethyl 2-(naphthalen-1-yl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3oa):



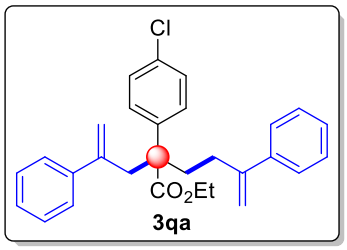
The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (50.2 mg, 55% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.5 Hz, 1H), 7.79-7.78 (m, 1H), 7.64-7.63 (m, 1H), 7.42-7.38 (m, 2H), 7.27 (d, *J* = 5.5 Hz, 2H), 7.16 (d, *J* = 7.0 Hz, 3H), 7.06-7.05 (m, 7H), 5.12 (s, 1H), 5.05 (s, 1H), 4.71 (s, 1H), 4.67 (s, 1H), 3.99-3.92 (m, 1H), 3.84-3.82 (m, 1H), 3.62 (d, *J* = 13.5 Hz, 1H), 3.52 (d, *J* = 13.5 Hz, 1H), 2.29 (d, *J* = 10.5 Hz, 2H), 2.18-2.15 (m, 2H), 0.95 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.86, 147.89, 145.63, 142.59, 140.81, 138.19, 134.37, 131.53, 129.46, 128.24, 128.18, 127.85, 127.35, 126.97, 126.60, 126.03, 125.90, 125.08, 125.07, 124.79, 123.95, 118.65, 112.49, 61.00, 53.63, 39.23, 33.67, 30.25, 14.00. HRMS (ESI) m/z: $[M+H]^+$ cald for $C_{33}H_{33}O_2^+$ 461.2475; found 461.2474.

Ethyl 2-(4-fluorophenyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3pa):



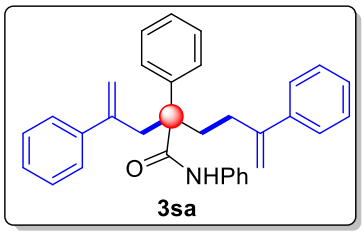
The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (17.4 mg, 20% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26-7.16 (m, 10H), 7.11-7.09 (m, 2H), 6.92-6.89 (m, 2H), 5.13-5.12 (m, 2H), 4.84 (s, 1H), 4.71 (s, 1H), 4.0-3.95 (m, 1H), 3.86-3.81 (m, 1H), 3.39 (d, *J* = 13.8 Hz, 1H), 3.21 (d, *J* = 13.8 Hz, 1H), 2.23-2.13 (m, 2H), 2.06-1.97 (m, 2H), 1.10 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.19, 162.34 (d, *J* = 245.5 Hz), 147.93, 145.41, 142.74, 140.78, 138.30 (d, *J* = 3.2 Hz), 128.36, 128.31, 128.14, 127.47, 127.24, 126.88, 126.08, 118.51, 115.04 (d, *J* = 21.1 Hz), 112.49, 60.95, 53.98, 40.41, 33.54, 30.21, 14.04. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{29}H_{29}FNaO_2^+$ 451.2044; found 451.2047.

Ethyl 2-(4-chlorophenyl)-5-phenyl-2-(2-phenylallyl)hex-5-enoate (3qa):



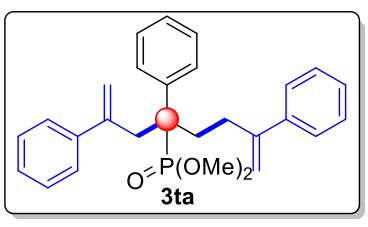
The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (19.1 mg, 21% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26-7.15 (m, 12H), 7.07 (d, *J* = 8.5 Hz, 2H), 5.13-5.12 (m, 2H), 4.85 (s, 1H), 4.72 (s, 1H), 4.0-3.94 (m, 1H), 3.87-3.81 (m, 1H), 3.38 (d, *J* = 13.5 Hz, 1H), 3.21 (d, *J* = 14.0 Hz, 1H), 2.23-2.12 (m, 2H), 2.06-1.96 (m, 2H), 1.10 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.95, 148.00, 145.38, 142.73, 141.12, 140.89, 132.59, 128.33, 128.31, 128.26, 128.17, 127.49, 127.25, 126.91, 126.12, 118.53, 112.51, 61.01, 54.21, 40.50, 33.56, 30.27, 14.04. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{30}ClO_2^+$ 445.1929; found 445.1927.

***N*,2,5-Triphenyl-2-(2-phenylallyl)hex-5-enamide (3sa)**



The title compound was prepared according to the general procedure and purified by column chromatography to give a white solid (16.7 mg, 18% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30-7.27 (m, 4H), 7.25-7.13 (m, 15H), 7.07 (t, *J* = 7.2 Hz, 1H), 6.67 (s, 1H), 5.10 (d, *J* = 20.4 Hz, 2H), 4.67 (d, *J* = 6.0 Hz, 2H), 3.53 (d, *J* = 13.8 Hz, 1H), 3.25 (d, *J* = 13.8 Hz, 1H), 2.40-2.30 (m, 2H), 2.14-2.09 (m, 1H), 1.91-1.86 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.99, 147.82, 145.43, 143.19, 142.57, 140.64, 137.90, 129.00, 128.62, 128.37, 128.19, 127.73, 127.46, 127.43, 127.06, 126.78, 126.12, 124.28, 119.86, 118.54, 112.69, 55.99, 40.38, 33.89, 30.30. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{33}H_{32}NO^+$ 458.2478; found 458.2479.

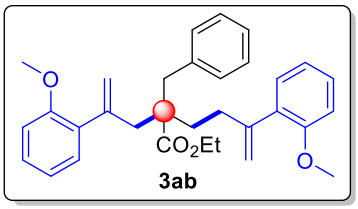
Dimethyl (2,4,7-triphenylocta-1,7-dien-4-yl)phosphonate (3ta)



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (26.9 mg, 30% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.8 Hz, 2H), 7.32-7.28 (m, 5H), 7.25-7.17 (m, 8H), 5.19 (s, 1H), 5.14 (s, 1H), 4.88 (s, 1H),

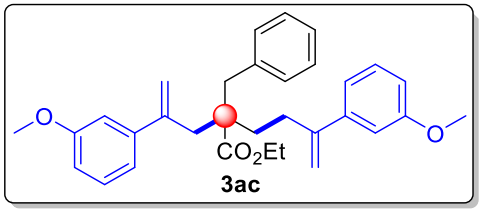
4.73 (s, 1H), 3.60 (d, J = 10.2 Hz, 3H), 3.49 (d, J = 10.2 Hz, 3H), 3.40 (t, J = 15 Hz, 1H), 3.32 (dd, J = 15, 8.4 Hz, 1H), 2.69-2.64 (m, 1H), 2.55-2.49 (m, 1H), 2.33-2.24 (m, 1H), 2.12-2.06 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 148.82, 143.96 (d, J = 14.3 Hz), 143.36, 141.26, 138.45 (d, J = 5.7 Hz), 128.85 (d, J = 5.9 Hz), 128.31 (d, J = 10.9 Hz), 128.06 (d, J = 2.3 Hz), 127.46, 127.22, 126.89 (d, J = 2.7 Hz), 126.58, 126.32, 118.04, 112.20, 53.81 (d, J = 7.675 Hz), 52.94 (d, J = 7.7 Hz), 48.38, 47.50, 38.71, 31.94 (d, J = 3.8 Hz), 30.54 (d, J = 3.5 Hz). ^{31}P NMR (243 MHz, $CDCl_3$) δ 32.94 (s, 1P). HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{32}O_3P^+$ 447.2084; found 447.2082.

Ethyl 2-benzyl-5-(2-methoxyphenyl)-2-(2-(2-methoxyphenyl)allyl)hex-5-enoate



(3ab): The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (48.7 mg, 50% yield). ^{1}H NMR (500 MHz, $CDCl_3$) 7.24-7.17 (m, 7H), 7.09-7.07 (m, 2H), 6.82-6.78 (m, 4H), 5.19 (d, J = 1.5 Hz, 1H), 5.10 (d, J = 1.0 Hz, 1H), 5.04 (s, 1H), 4.81 (d, J = 1.0 Hz, 1H), 3.82 (d, J = 6.0 Hz, 5H), 3.78 (s, 3H), 3.12 (d, J = 14.0 Hz, 1H), 2.94 (dd, J = 18.0, 14.0 Hz, 2H), 2.78 (d, J = 14.5 Hz, 2H), 2.44-2.40 (m, 2H), 1.67 (t, J = 8.5 Hz, 2H), 1.14 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.91, 159.17, 159.13, 147.80, 145.35, 137.78, 135.49, 133.84, 130.31, 128.20, 127.97, 127.33, 126.53, 116.08, 113.66, 113.63, 110.78, 60.49, 55.43, 55.42, 50.54, 41.79, 41.16, 32.52, 30.04, 14.12. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_4^+$ 507.2506; found 507.2505.

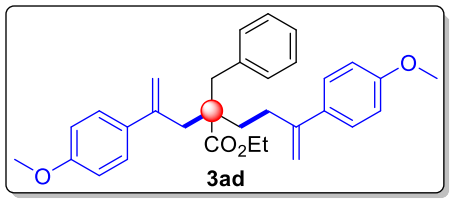
Ethyl 2-benzyl-5-(3-methoxyphenyl)-2-(2-(3-methoxyphenyl)allyl)hex-5-enoate



(3ac): The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (50.3 mg, 52% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20-7.17 (m, 5H), 7.07 (d, J = 7.5 Hz, 2H), 6.90-6.76 (m, 6H), 5.27 (s, 1H), 5.16 (s, 1H), 5.11 (s, 1H), 4.87 (s, 1H), 3.83 (q, J = 7.5 Hz, 2H), 3.79 (s, 3H), 3.77 (s, 3H), 3.09 (d, J = 14.0 Hz, 1H), 2.94-2.89 (m, 2H), 2.79 (d, J = 14.5 Hz, 1H), 2.45-2.42

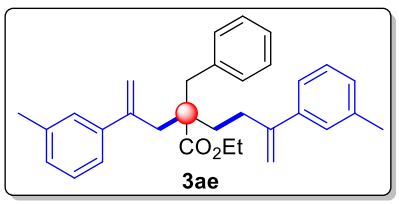
(m, 2H), 1.68-1.66 (m, 2H), 1.13 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.76, 159.64, 159.50, 148.42, 145.88, 144.51, 143.08, 137.61, 130.25, 129.20, 128.16, 126.53, 119.43, 118.82, 117.28, 112.84, 112.71, 112.67, 112.44, 112.34, 60.48, 55.32, 50.54, 41.52, 41.14, 32.39, 30.05, 14.06. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_4^+$ 507.2506; found 507.2505.

Ethyl 2-benzyl-5-(4-methoxyphenyl)-2-(2-(4-methoxyphenyl)allyl)hex-5-enoate (3ad): The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (33.7 mg, 35% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25-7.16 (m, 7H), 7.09-7.08 (m, 2H), 6.82-6.78 (m, 4H), 5.19 (d, J = 1.5 Hz, 1H), 5.10 (d, J = 1.0 Hz, 1H), 5.05 (s, 1H), 4.81 (d, J = 0.5 Hz, 1H), 3.82-3.78 (m, 8H), 3.10 (d, J = 14.0 Hz, 1H), 2.91 (dd, J = 17.5, 14.5 Hz, 2H), 2.77 (d, J = 14.5 Hz, 1H), 2.46-2.37 (m, 2H), 1.66 (t, J = 9.0 Hz, 2H), 1.12 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.89, 159.16, 159.12, 147.78, 145.34, 137.76, 135.48, 133.82, 130.30, 128.19, 127.96, 127.31, 126.52, 116.07, 113.65, 113.62, 110.77, 60.48, 55.42, 55.40, 50.53, 41.78, 41.15, 32.50, 30.03, 14.11. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_4^+$ 507.2506; found 507.2505.



3ad

Ethyl 2-benzyl-5-(*m*-tolyl)-2-(2-(*m*-tolyl)allyl)hex-5-enoate (3ae):

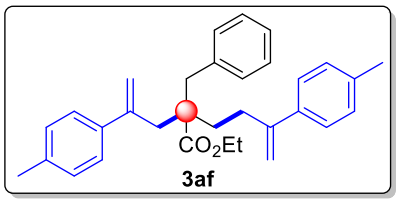


3ae

The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (49.6 mg, 55% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24-7.18 (m, 5H), 7.14-7.06 (m, 8H), 5.27 (d, J = 1.5 Hz, 1H), 5.18 (d, J = 1.0 Hz, 1H), 5.14 (d, J = 1.0 Hz, 1H), 4.88 (d, J = 1.0 Hz, 1H), 3.85 (q, J = 7.0 Hz, 2H), 3.13 (d, J = 14.0 Hz, 1H), 2.98-2.94 (m, 2H), 2.84 (d, J = 14.5 Hz, 1H), 2.49-2.45 (m, 2H), 2.37 (s, 3H), 2.35 (s, 3H), 1.72-1.68 (m, 2H), 1.16 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.83, 148.62, 146.11, 143.00, 141.51, 137.72, 137.70, 137.67, 130.26, 128.18,

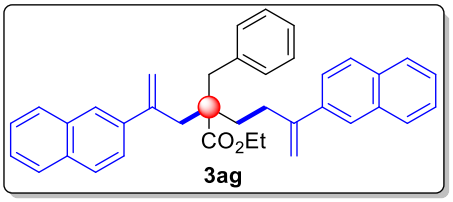
128.16, 128.13, 127.60, 127.02, 126.52, 124.02, 123.42, 116.97, 112.08, 60.42, 50.50, 41.56, 41.06, 32.33, 29.98, 21.62, 21.55, 14.09. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_2^+$ 475.2608; found 475.2602.

Ethyl 2-benzyl-5-(*p*-tolyl)-2-(2-(*p*-tolyl)allyl)hex-5-enoate (3af):

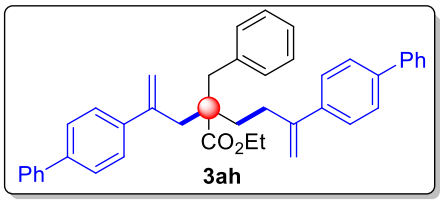


The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (49.5 mg, 55% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26-7.05 (m, 13H), 5.23 (d, *J* = 1.5 Hz, 1H), 5.14 (s, 1H), 5.07 (s, 1H), 4.82 (s, 1H), 3.82 (q, *J* = 7.0 Hz, 2H), 3.10 (d, *J* = 14.0 Hz, 1H), 2.93-2.88 (m, 2H), 2.79 (d, *J* = 14.5 Hz, 1H), 2.45-2.40 (m, 2H), 2.33 (s, 3H), 2.31 (s, 3H), 1.67-1.64 (m, 2H), 1.12 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.86, 148.23, 145.82, 140.14, 138.46, 137.75, 137.07, 130.30, 128.96, 128.89, 128.16, 126.75, 126.50, 126.10, 116.54, 111.48, 60.44, 50.56, 41.60, 41.11, 32.49, 29.96, 21.21, 21.19, 14.08. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{32}H_{36}NaO_2^+$ 475.2608; found 475.2602.

Ethyl 2-benzyl-5-(naphthalen-2-yl)-2-(2-(naphthalen-2-yl)allyl)hex-5-enoate (3ag):

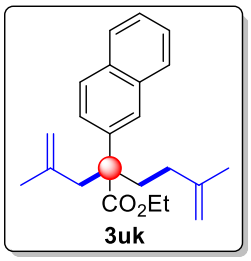


The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (65.8 mg, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81-7.66 (m, 8H), 7.48-7.43 (m, 5H), 7.38-7.36 (m, 1H), 7.19-7.18 (m, 3H), 7.12-7.10 (m, 2H), 5.40 (s, 1H), 5.26 (d, *J* = 4.0 Hz, 2H), 4.94 (s, 1H), 3.73-3.67 (m, 2H), 3.18-3.08 (m, 2H), 3.01-2.93 (m, 2H), 2.62-2.58 (m, 2H), 1.78-1.74 (m, 2H), 1.05 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.86, 148.34, 145.91, 140.21, 138.65, 137.64, 133.45, 133.33, 132.88, 132.82, 130.26, 128.26, 128.23, 128.18, 127.79, 127.64, 126.61, 126.24, 126.16, 125.92, 125.86, 125.48, 125.40, 124.83, 117.89, 112.93, 60.51, 50.62, 41.72, 41.26, 32.44, 30.07, 14.02. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{38}H_{36}NaO_2^+$ 547.2608; found 547.2607.

Ethyl 5-([1,1'-biphenyl]-4-yl)-2-(2-([1,1'-biphenyl]-4-yl)allyl)-2-benzylhex-5-enoate (3ah): 

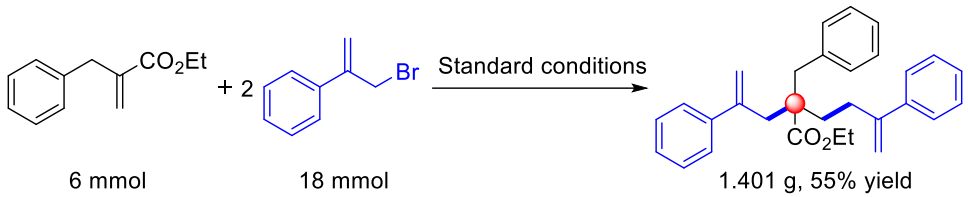
The title compound was prepared according to the general procedure and purified by column chromatography to give a white solid (47.8 mg, 41% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.30 (m, 18H), 7.23-7.22 (m, 3H), 7.14-7.12 (m, 2H), 5.34 (d, *J* = 1.5 Hz, 1H), 5.24 (d, *J* = 1.0 Hz, 1H), 5.28 (s, 1H), 4.91 (d, *J* = 0.5 Hz, 1H), 3.86 (q, *J* = 7.0 Hz, 2H), 3.18 (d, *J* = 14.0 Hz, 1H), 3.02-2.97 (m, 2H), 2.89 (d, *J* = 15.0 Hz, 1H), 2.53-2.49 (m, 2H), 1.74–1.71 (m, 2H), 1.16 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.72, 147.75, 145.41, 141.81, 140.78, 140.75, 140.16, 140.06, 137.52, 130.13, 128.79, 128.77, 128.13, 127.28, 127.24, 127.19, 126.98, 126.96, 126.86, 126.83, 126.47, 117.33, 112.23, 60.44, 50.55, 41.47, 41.17, 32.22, 29.80, 29.71, 14.01. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{42}H_{40}NaO_2^+$ 599.2921; found 599.2931.

Ethyl 5-methyl-2-(2-methylallyl)-2-(naphthalen-2-yl)hex-5-enoate (3uk)



The title compound was prepared according to the general procedure and purified by column chromatography to give a yellowish oil (19.6 mg, 29% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83-7.78 (m, 3H), 7.75 (s, 1H), 7.49-7.45 (m, 2H), 7.41 (dd, *J* = 8.4, 1.8 Hz, 1H), 4.84 (s, 1H), 4.70-4.84 (m, 3H), 4.17-4.08 (m, 2H), 3.06 (d, *J* = 13.8 Hz, 1H), 2.90 (d, *J* = 13.8 Hz, 1H), 2.36-2.32 (m, 1H), 2.29-2.25 (m, 1H), 1.85-1.82 (m, 2H), 1.70 (s, 3H), 1.44 (s, 3H), 1.15 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.81, 145.87, 141.99, 140.30, 133.36, 132.35, 128.30, 128.05, 127.58, 126.19, 126.01, 125.29, 125.13, 115.43, 110.07, 60.98, 53.25, 42.47, 32.60, 32.57, 23.99, 22.76, 14.16. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{29}O_2^+$ 337.2162; found 337.2160.

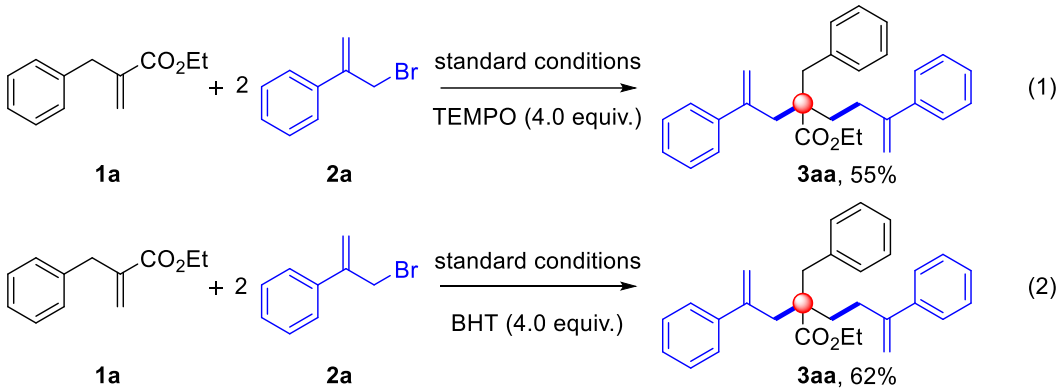
5. Scale-up Experiment



Ethyl 2-benzylacrylate **1a** (1140.0 mg, 6 mmol), 3-bromoprop-1-en-2-ylbenzene **2a** (3546.0 mg, 18 mmol), Cp*Ru(cod)Cl (114.0 mg, 0.3 mmol, 5 mol%), Mg (364.5 mg, 15 mmol), NMP (30.0 mL) were added to a flame-dried Young-type tube. The mixture was stirred under Ar at 0 °C for 12 h. After rising to room temperature, the reaction mixture was treated with saturated $NH_4Cl_{(aq.)}$ (100 mL), extracted with ethyl acetate (3 x 50 mL), washed with saturated $NaCl_{(aq.)}$ (3 x 50 mL), and dried over Na_2SO_4. Then, the solvent (ethyl acetate) was removed in vacuo and the residue was purified by flash column chromatography on silica gel and eluted with ethyl acetate/petroleum ether (1/100) to afford the desired product **3aa** (1.401 g, 55% yield).

6. Radical test

When TEMPO (4 equiv.), or BHT (4 equiv.) was introduced to the standard conditions, the desired product **3aa** was obtained in 55% and 62% yields, respectively. These results indicated that a radical pathway should be ruled out in this reaction.



7. References

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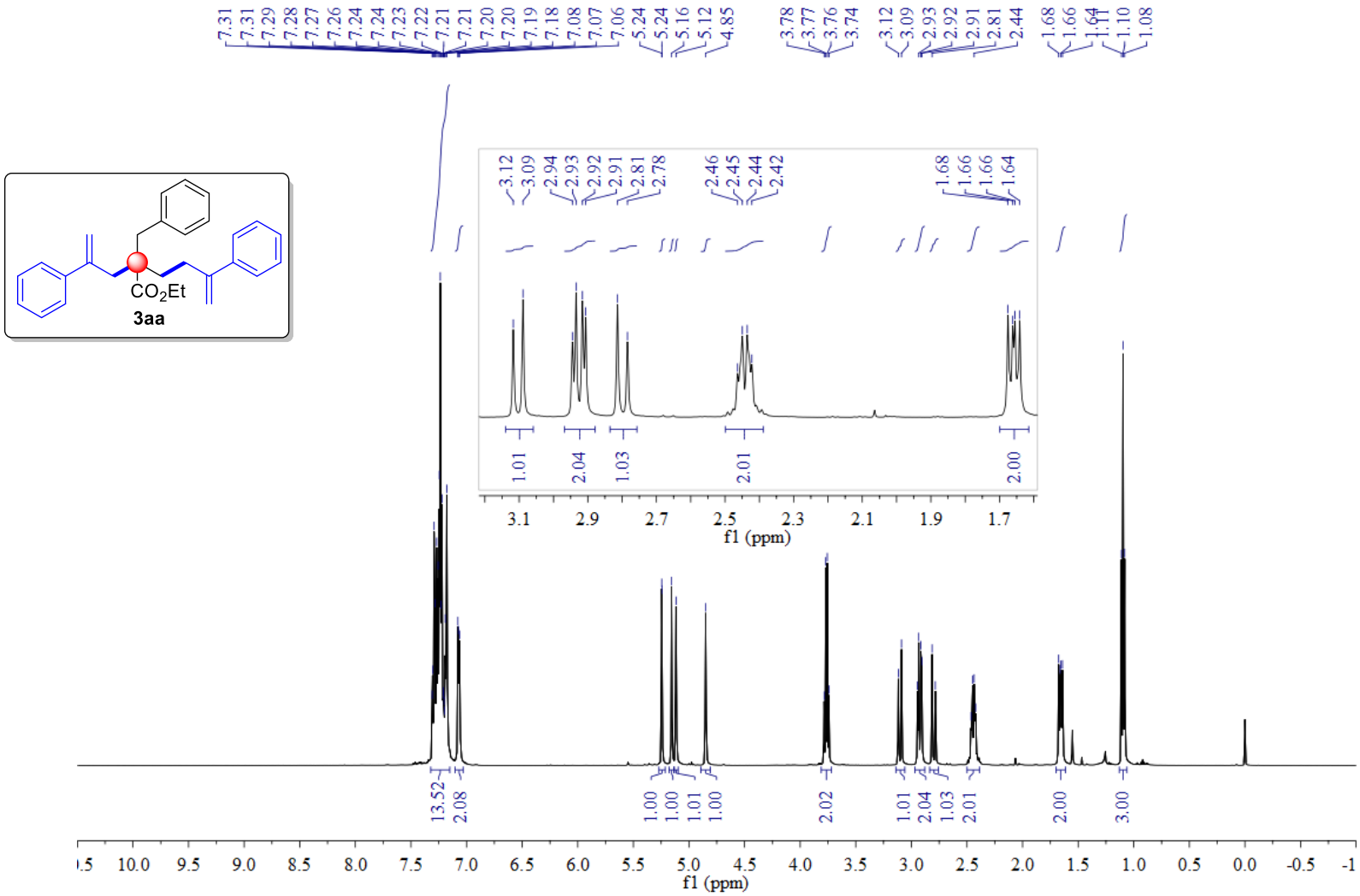
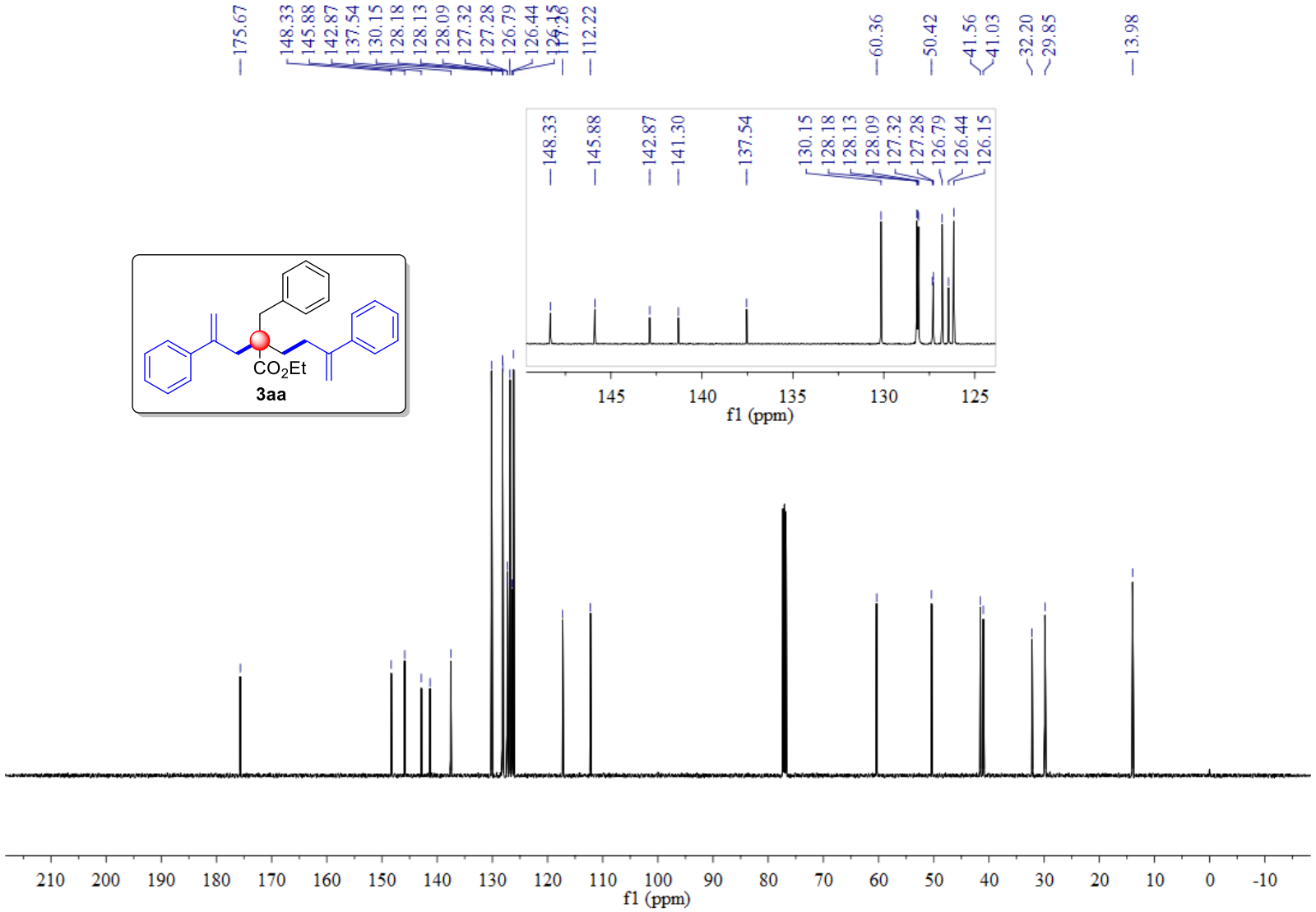
8. Copies for ^{1}H NMR and ^{13}C NMR spectra of the products

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126.44
126.15
112.22
60.36
50.42
41.56
41.03
32.20
29.85
13.98
CO2Et
3aa
148.33
145.88
142.87
141.30
137.54
130.15
128.18
128.13
128.09
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127.28
126.79
126.44
126.15
145
140
135
130
125
f1 (ppm)
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200
190
180
170
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120
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100
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80
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60
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40
30
20
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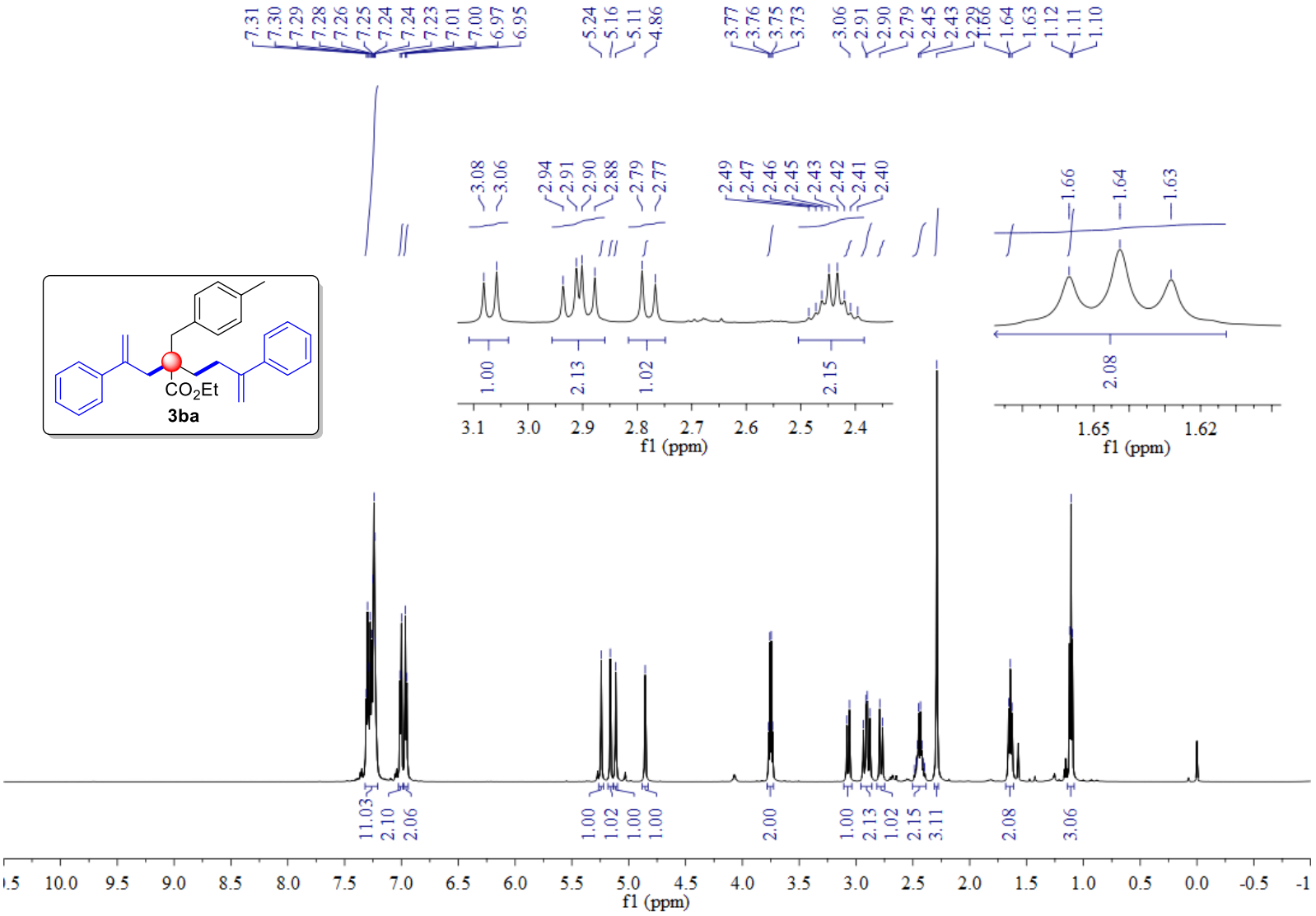
f1 (ppm)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5.24
5.16
5.11
4.86
3.77
3.76
3.75
3.73
3.06
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2.90
2.79
2.45
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2.29
1.66
1.64
1.63
1.12
1.11
1.10
CO2Et
3ba
3.08
3.06
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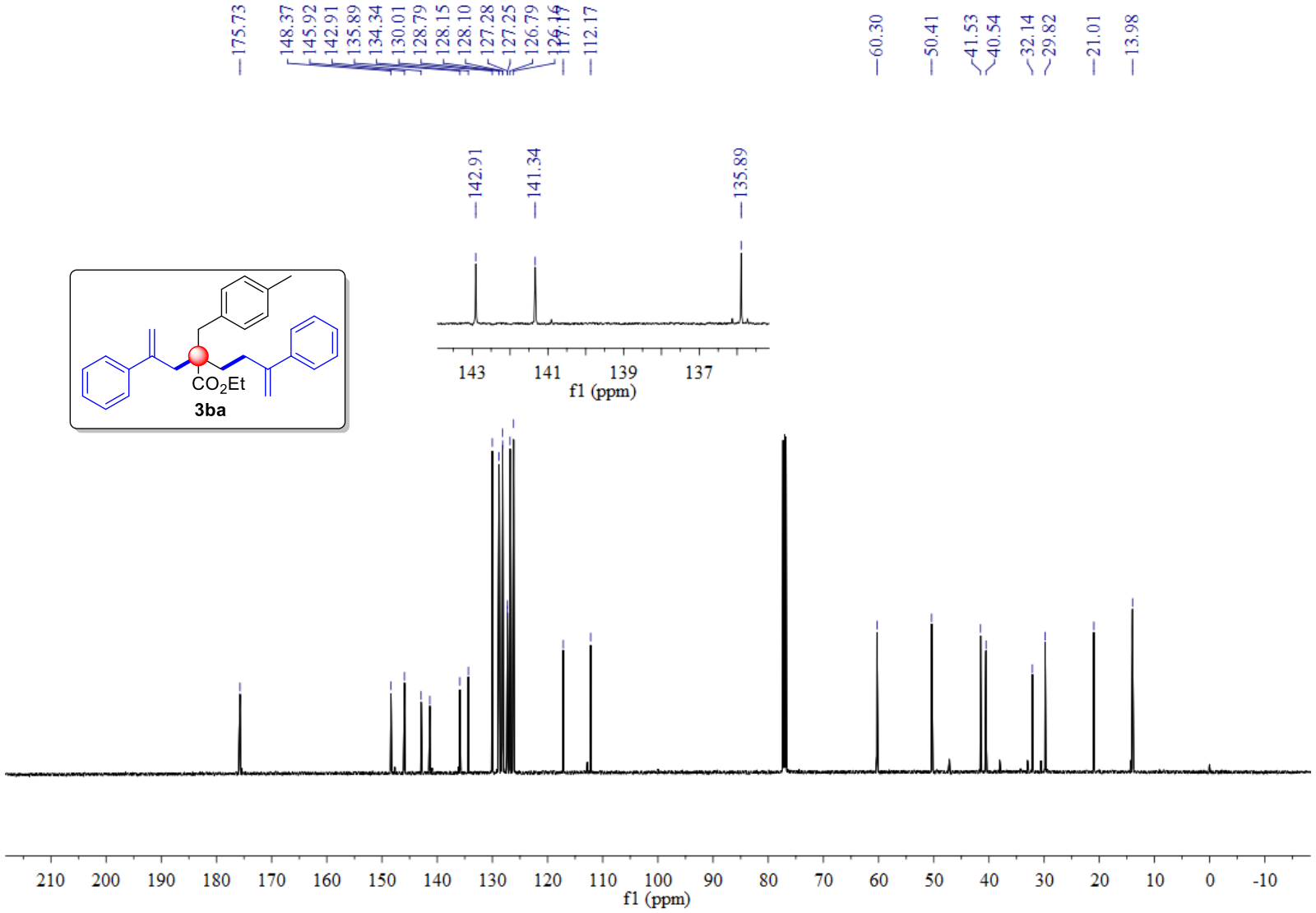
f1 (ppm)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29.82
21.01
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141
139
137
f1 (ppm)
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3ba
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180
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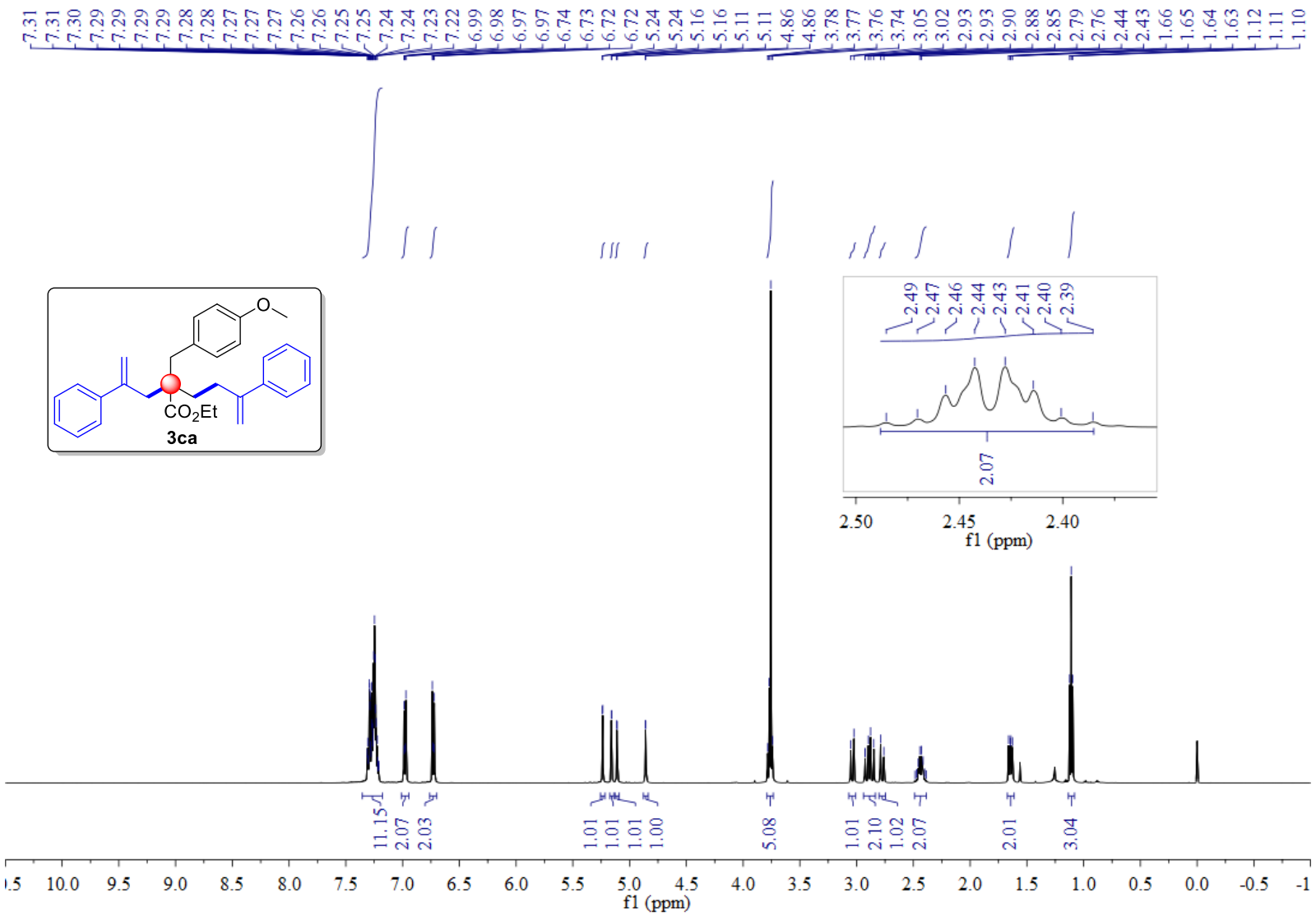
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O
CO2Et
3ca
2.49 2.47 2.46 2.44 2.43 2.41 2.40 2.39
2.07
2.50 2.45 2.40
f1 (ppm)
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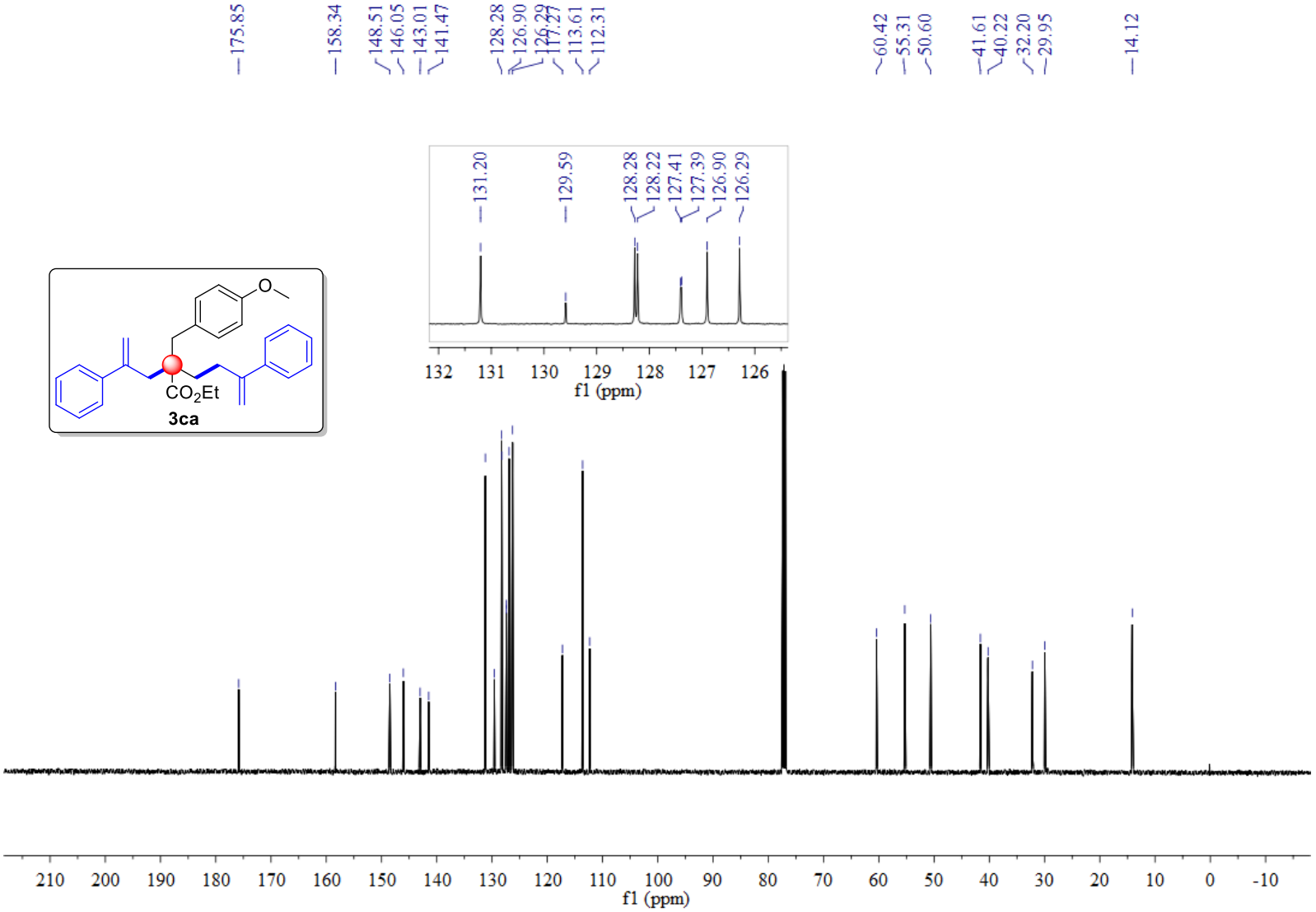
f1 (ppm)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126.29
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CO2Et
3ca
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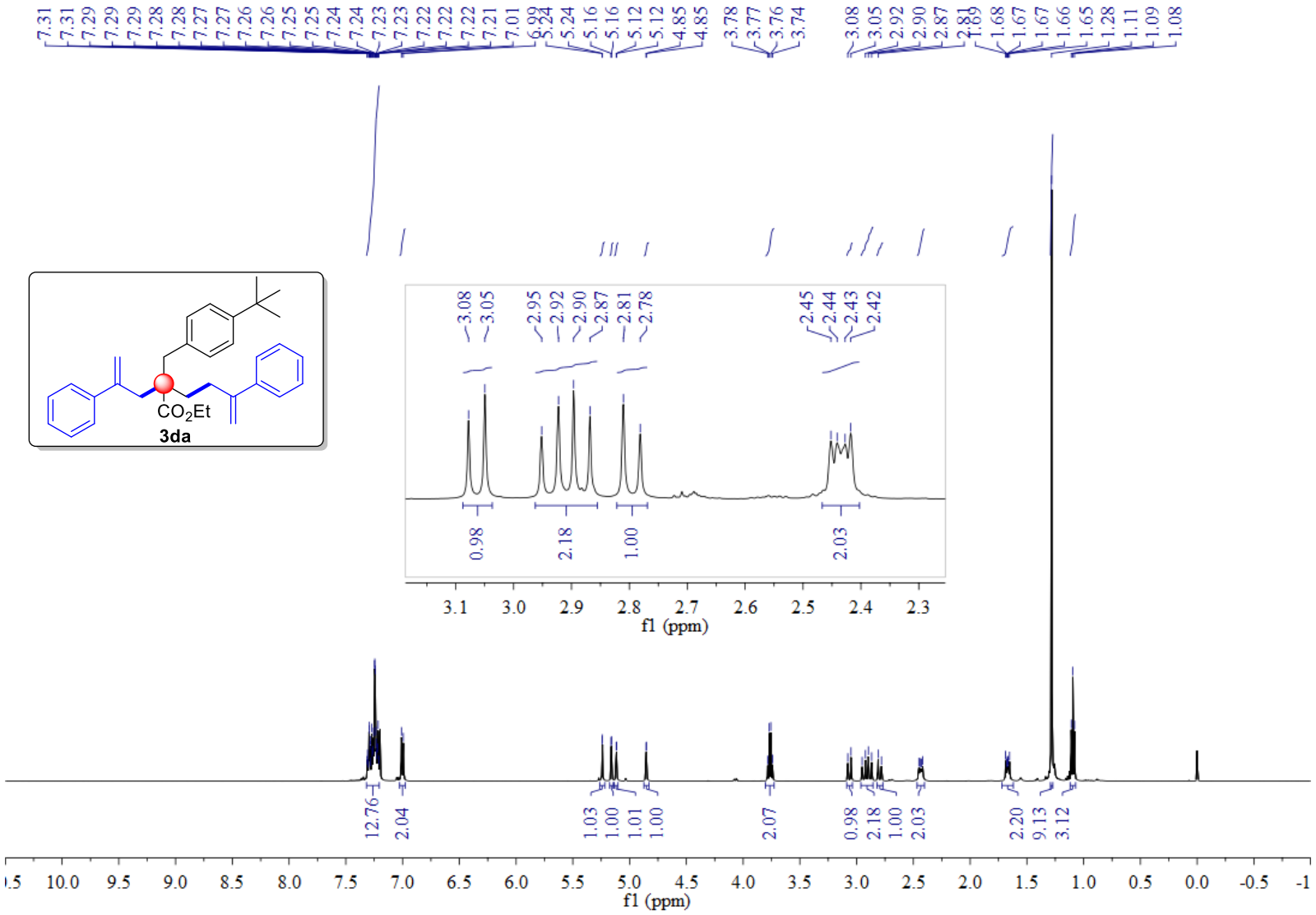
f1 (ppm)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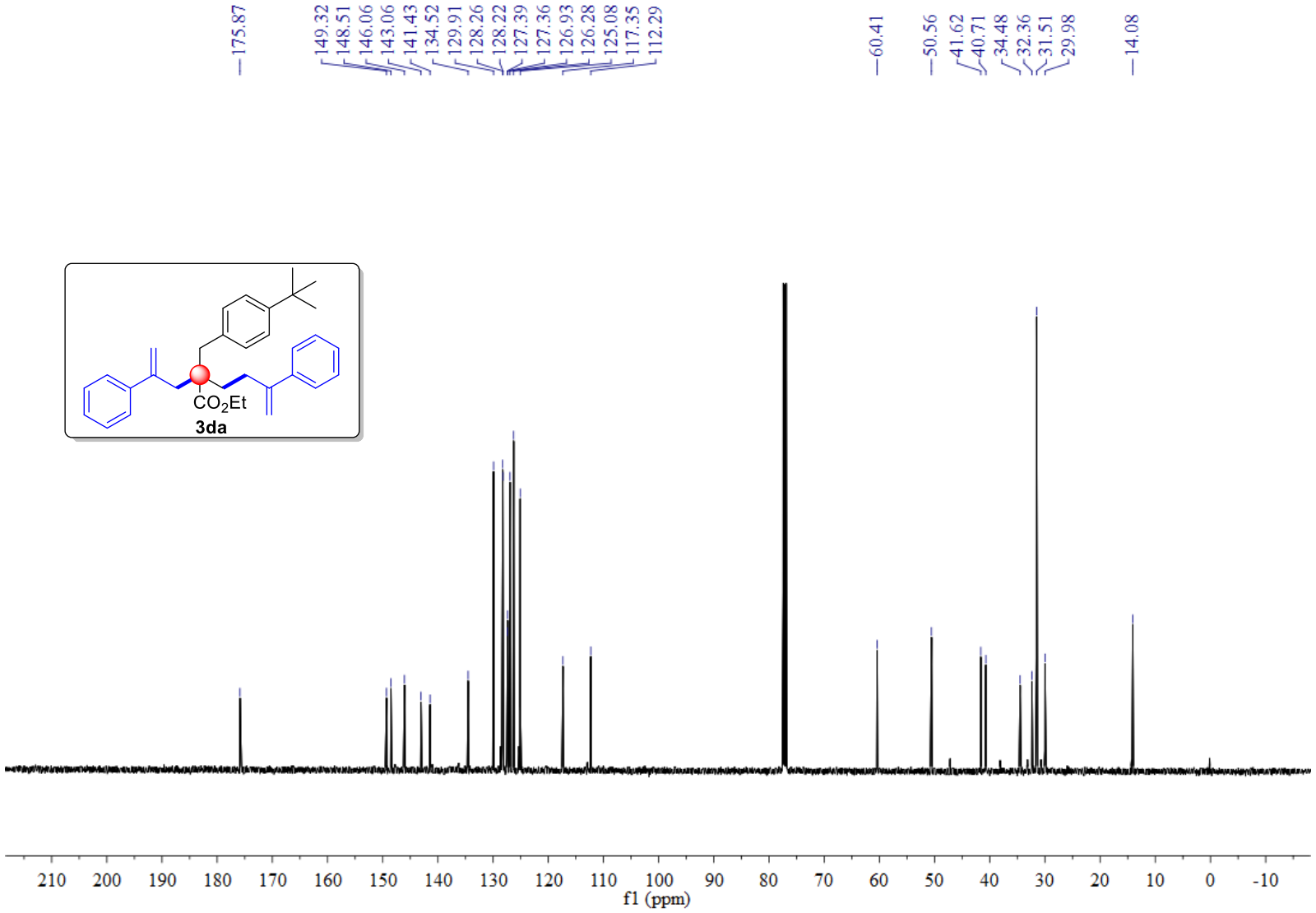
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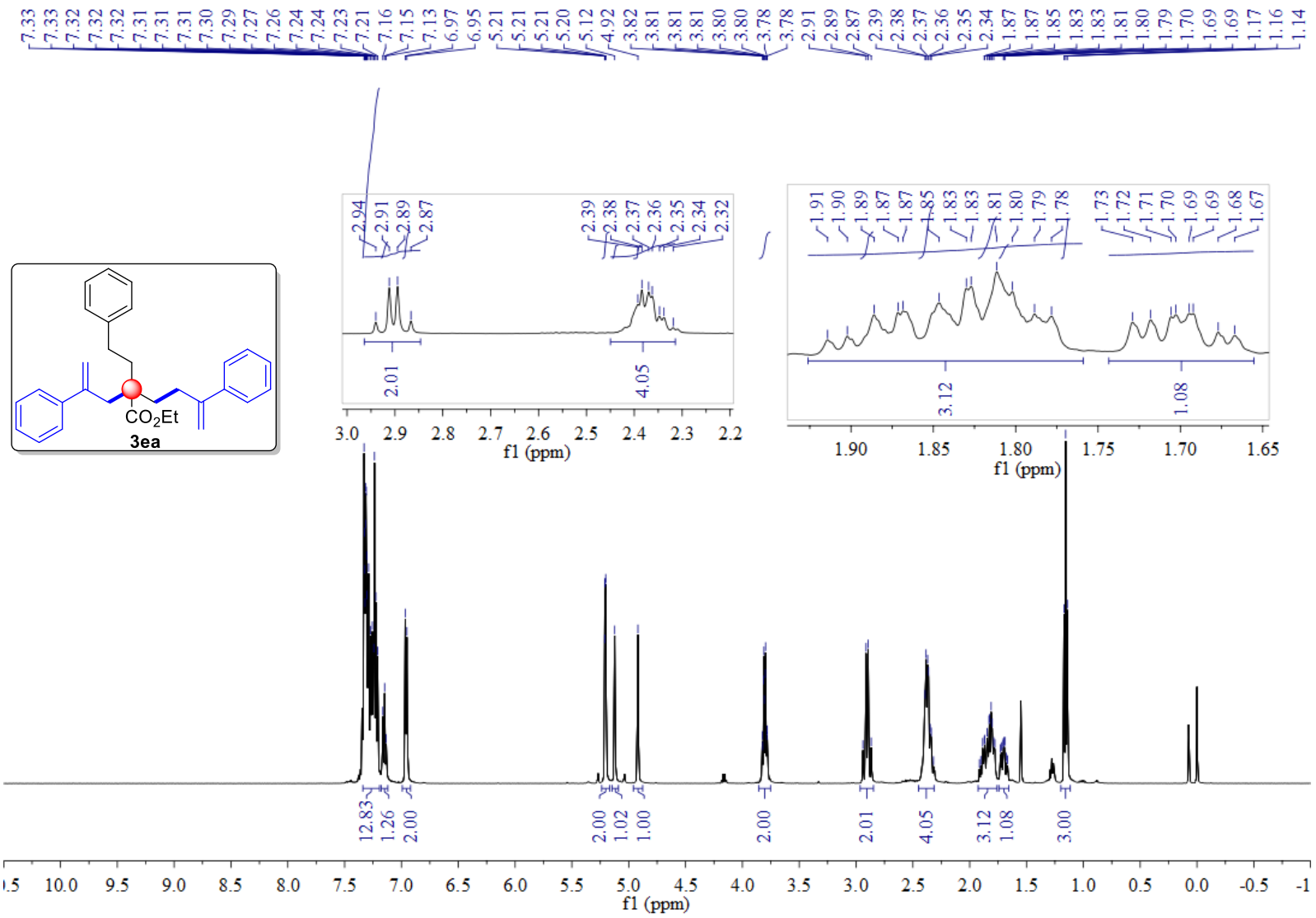
f1 (ppm)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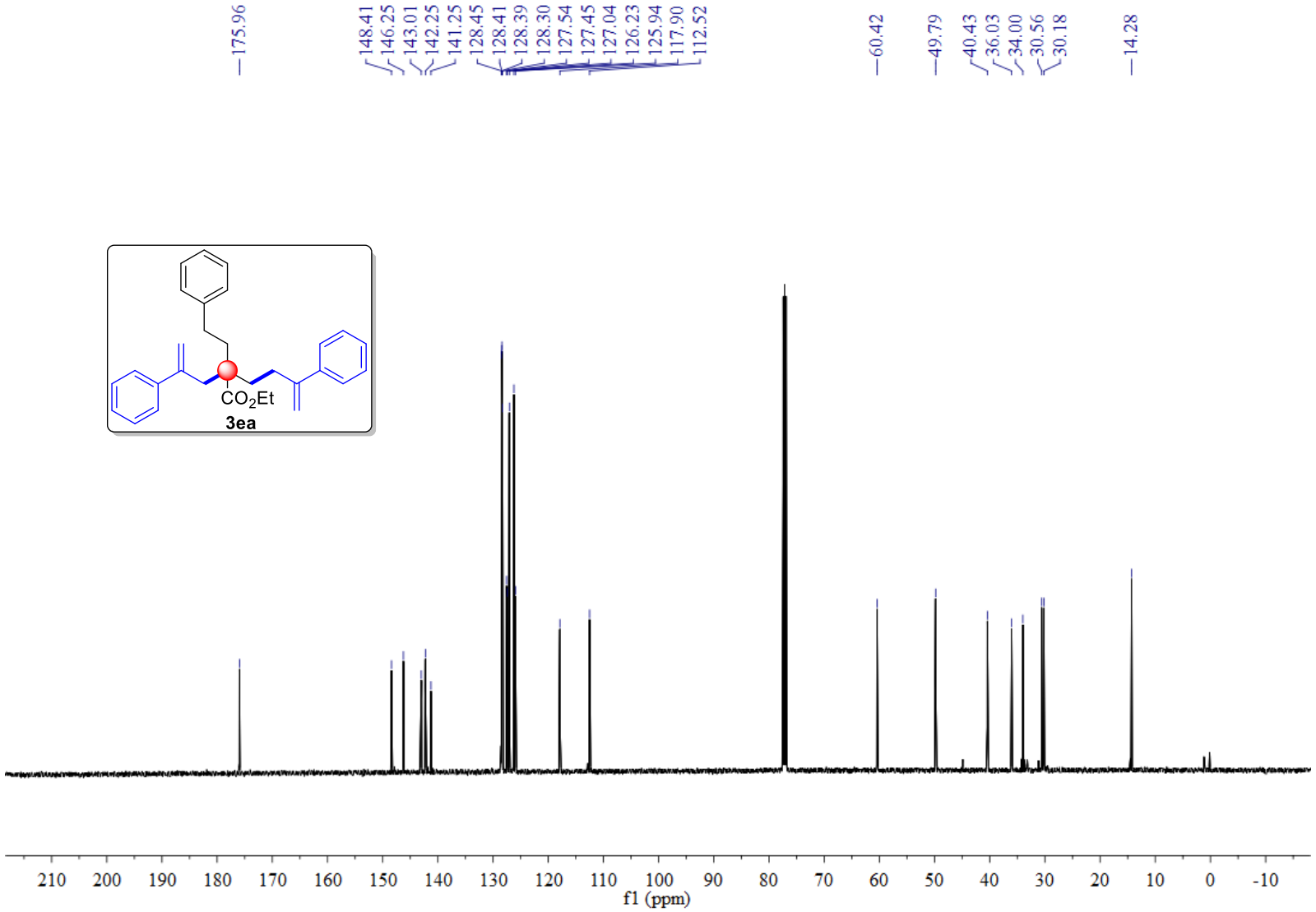
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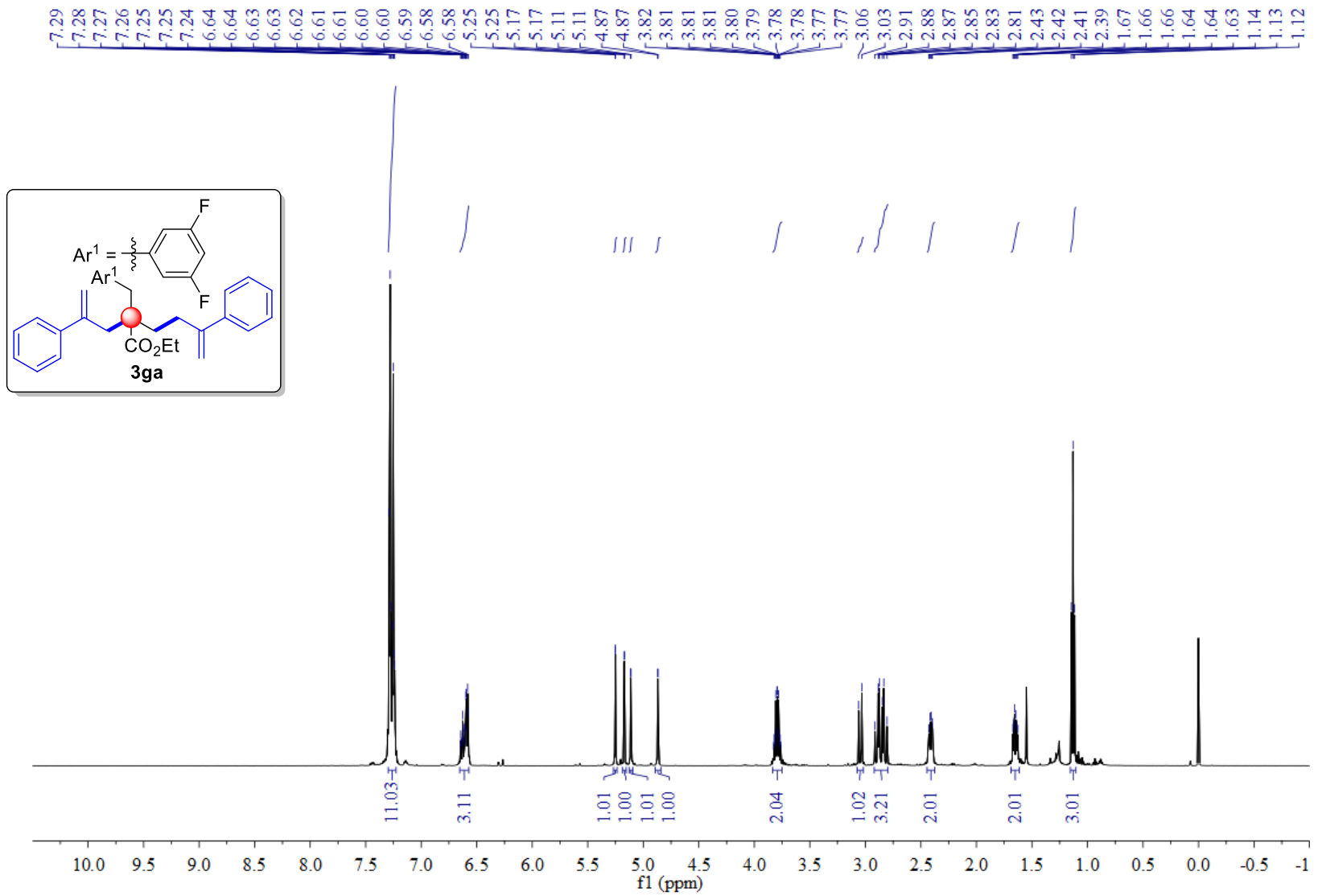
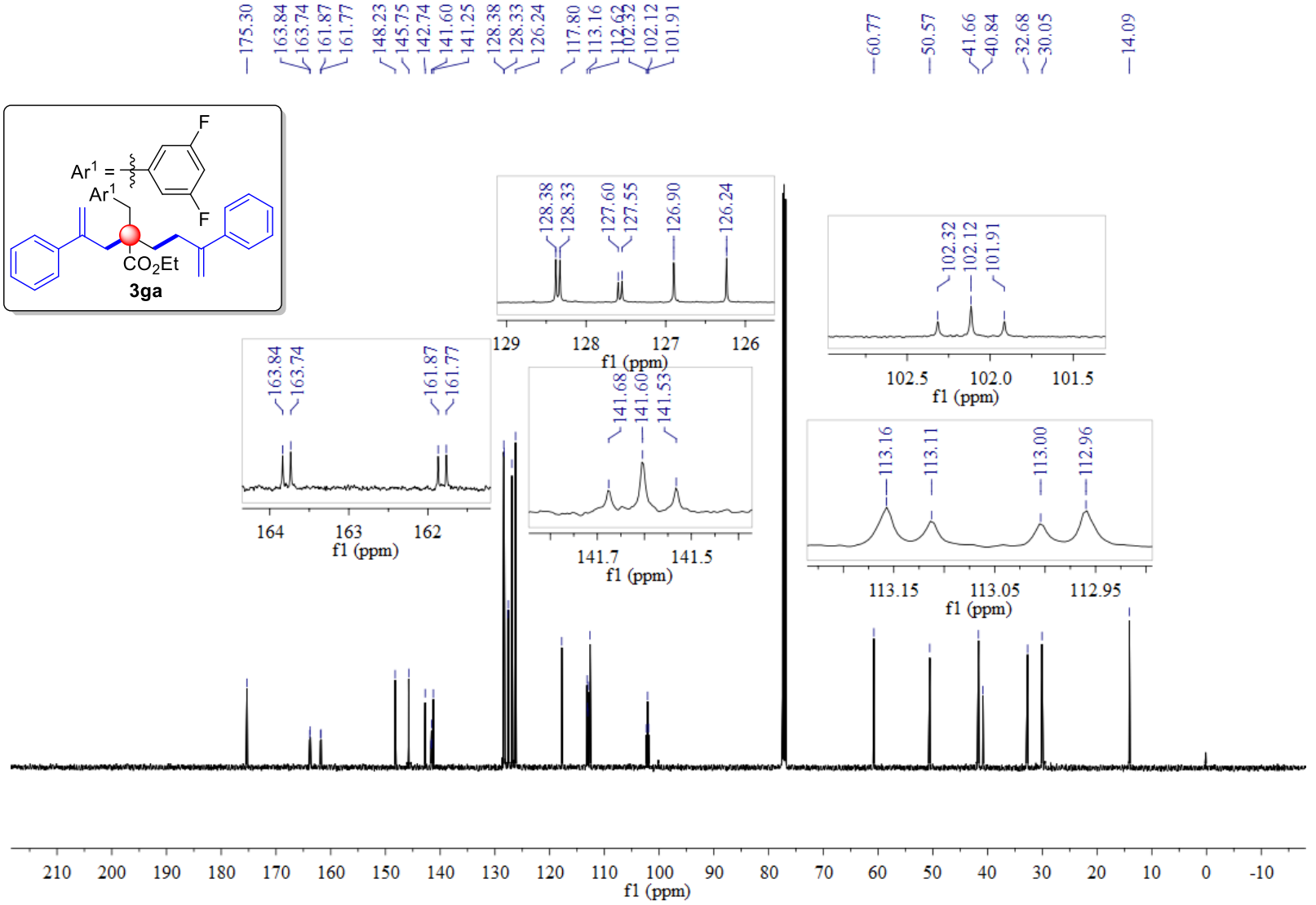
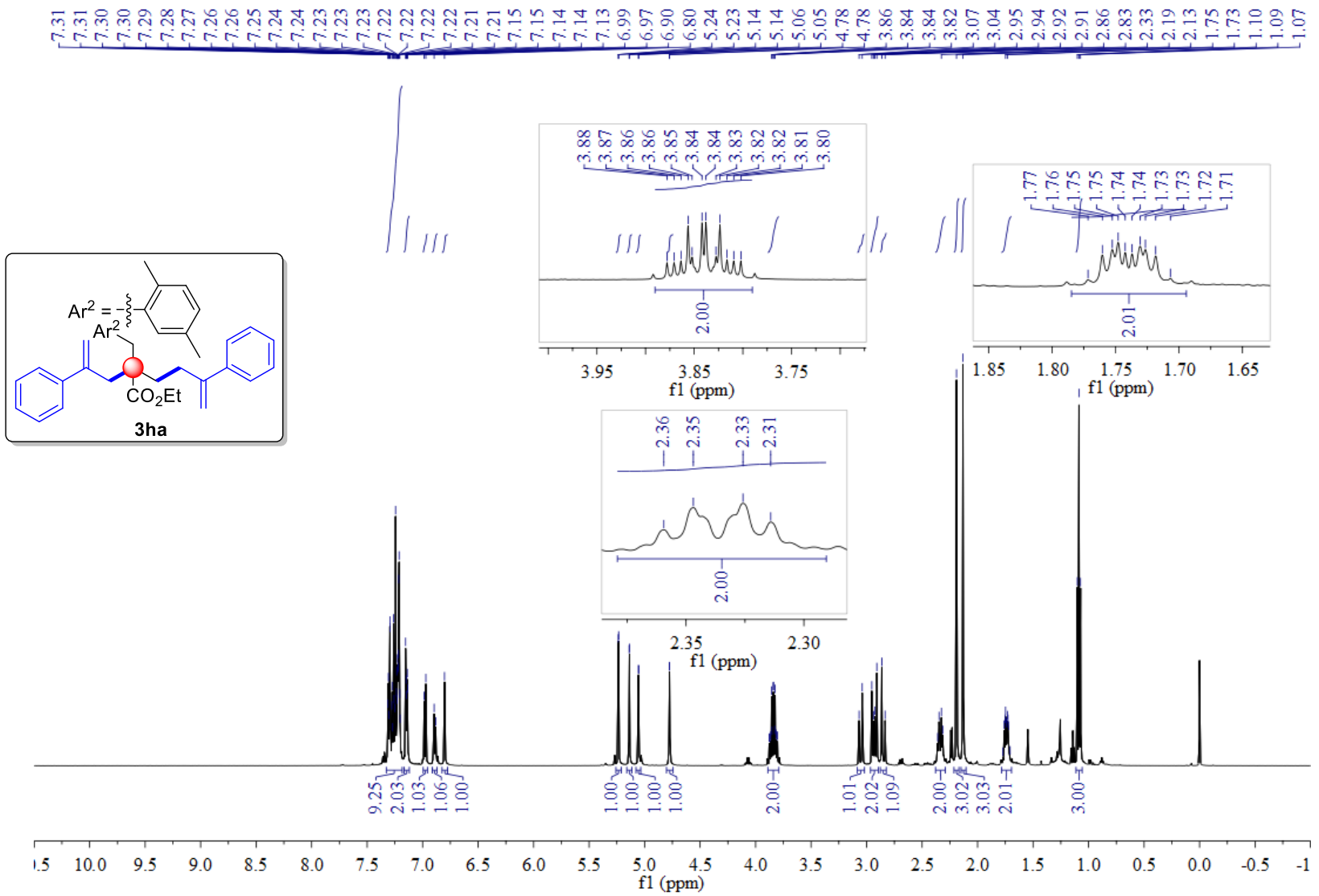
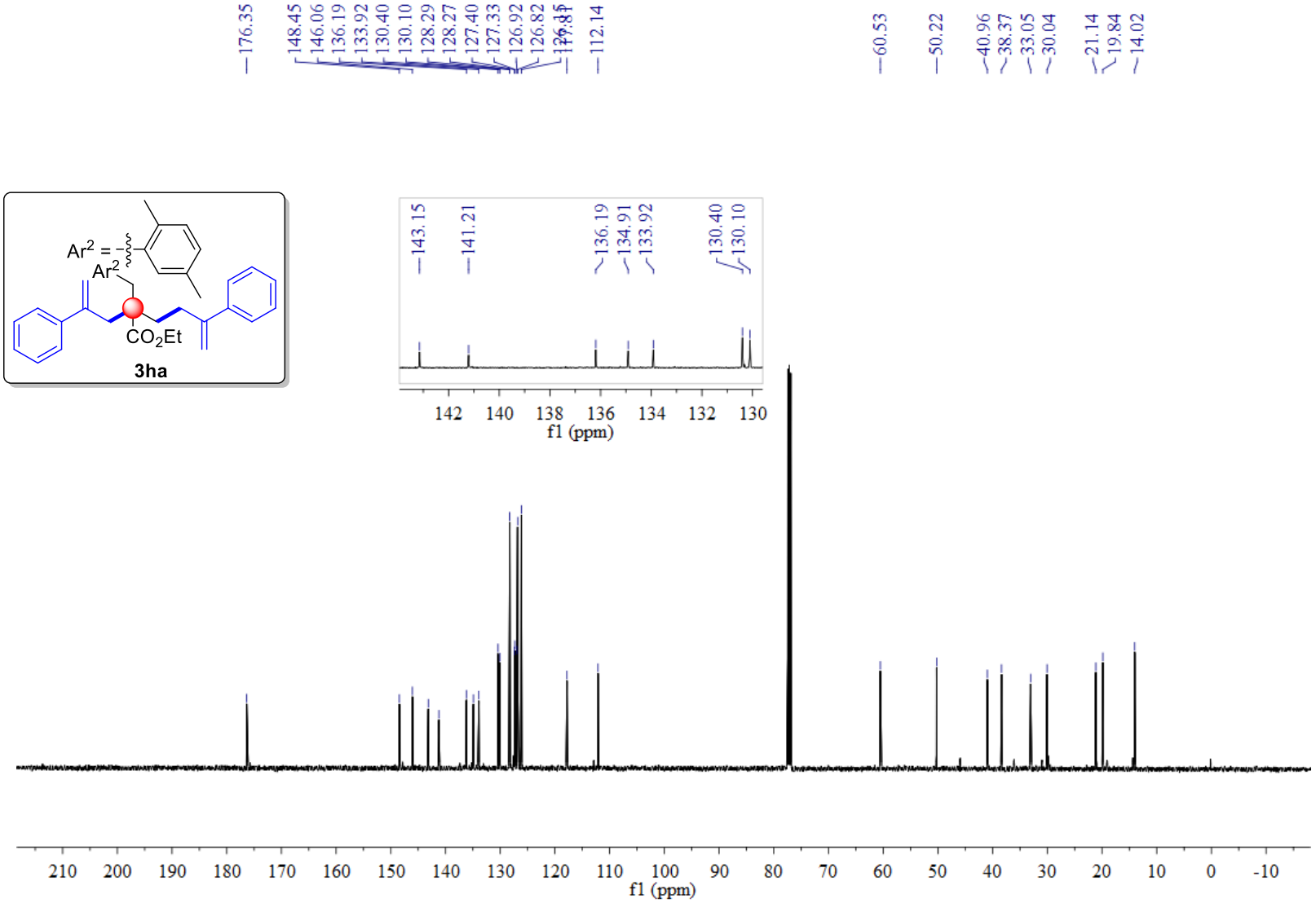
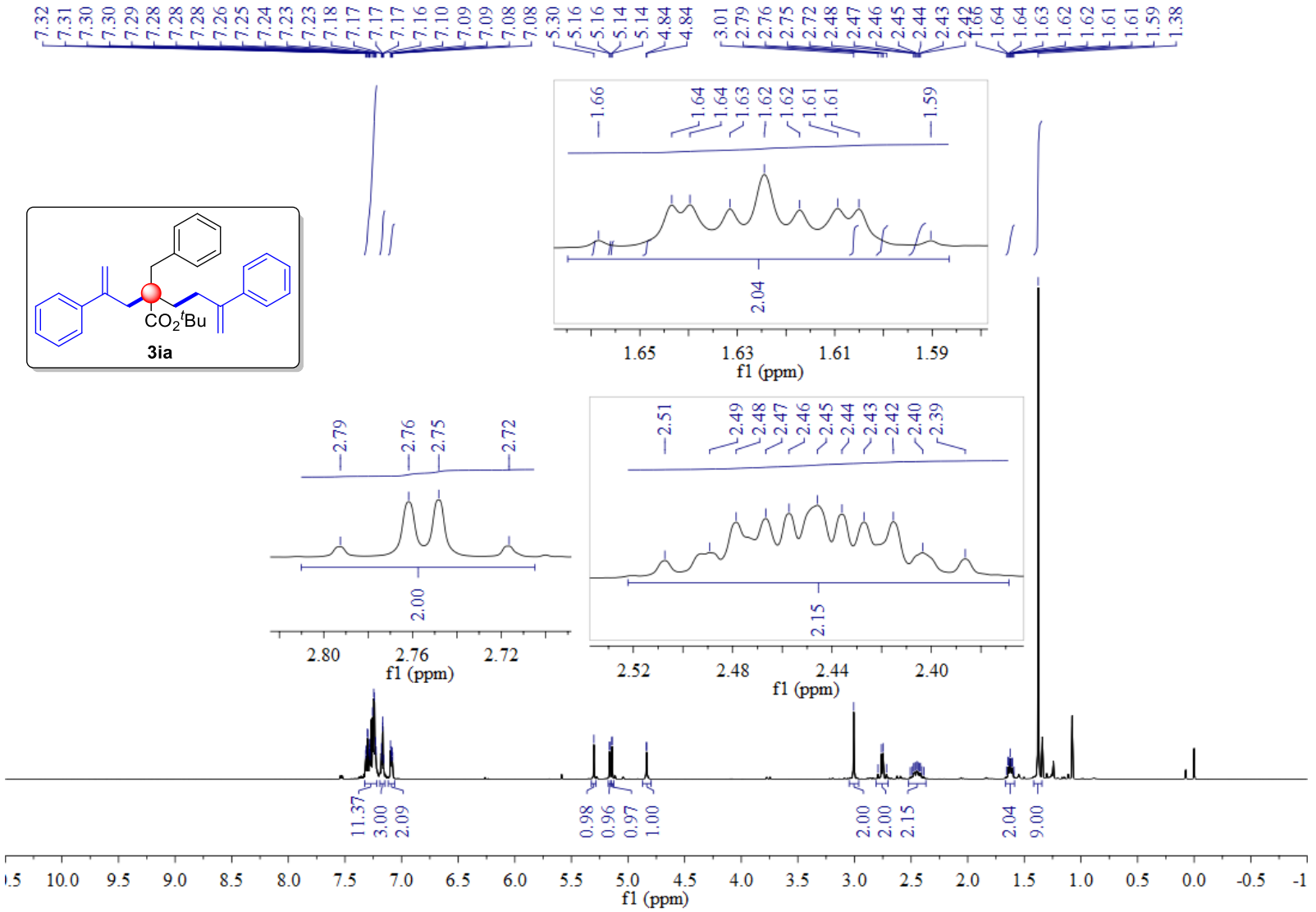
f1 (ppm)

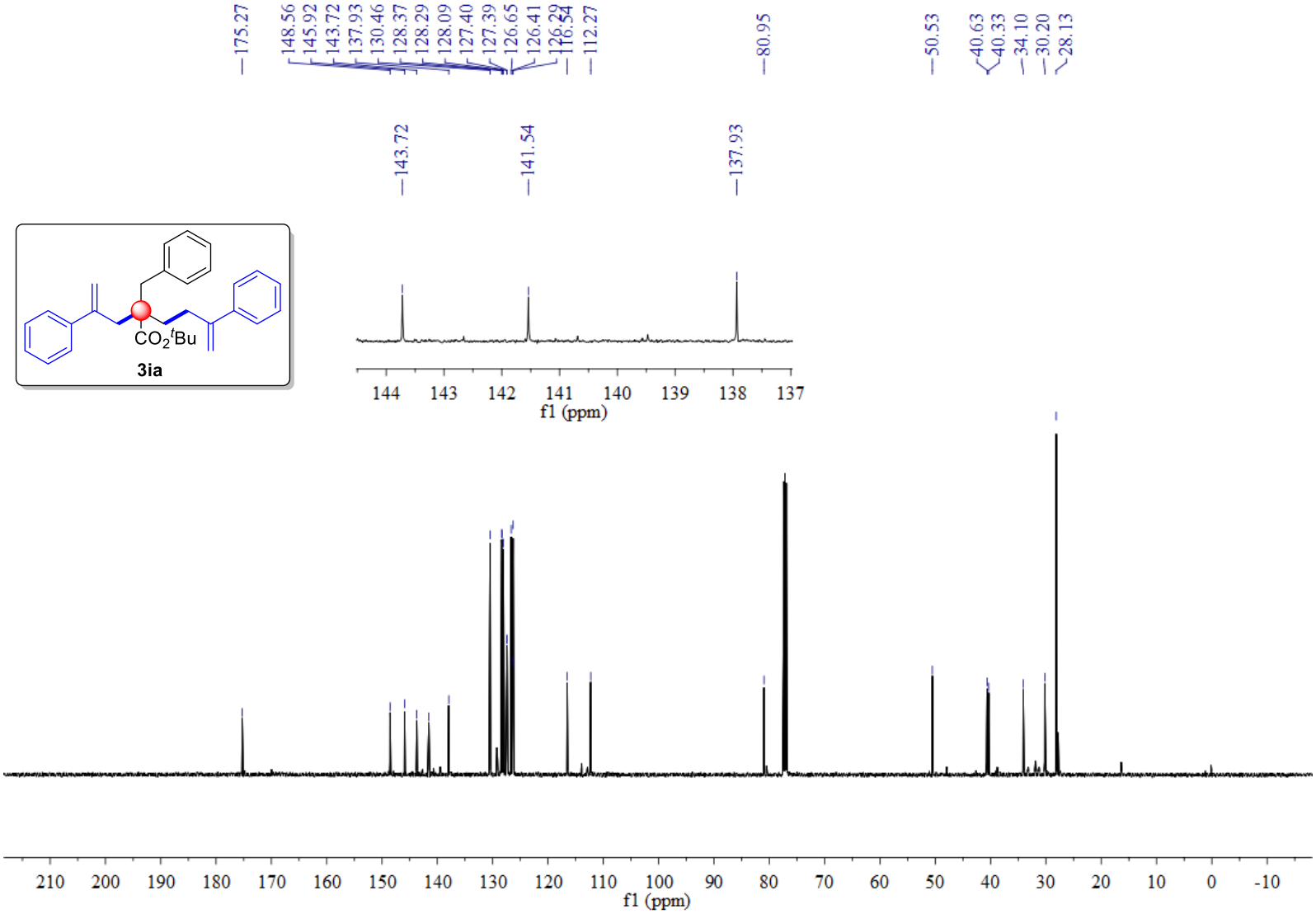

7.29 7.28 7.27 7.26 7.25 7.25 7.24 6.64 6.64 6.63 6.63 6.62 6.61 6.61 6.60 6.60 6.59 6.58 6.58 5.25 5.25 5.17 5.17 5.11 5.11 4.87 4.87 3.82 3.81 3.81 3.81 3.80 3.79 3.78 3.78 3.77 3.77 3.06 3.03 2.91 2.88 2.87 2.85 2.83 2.81 2.43 2.42 2.41 2.39 1.67 1.66 1.66 1.64 1.64 1.63 1.14 1.13 1.12
Ar1 =
Ar1
F
F
CO2Et
3ga
11.03
3.11
1.01
1.00
1.01
1.00
2.04
1.02
3.21
2.01
2.01
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

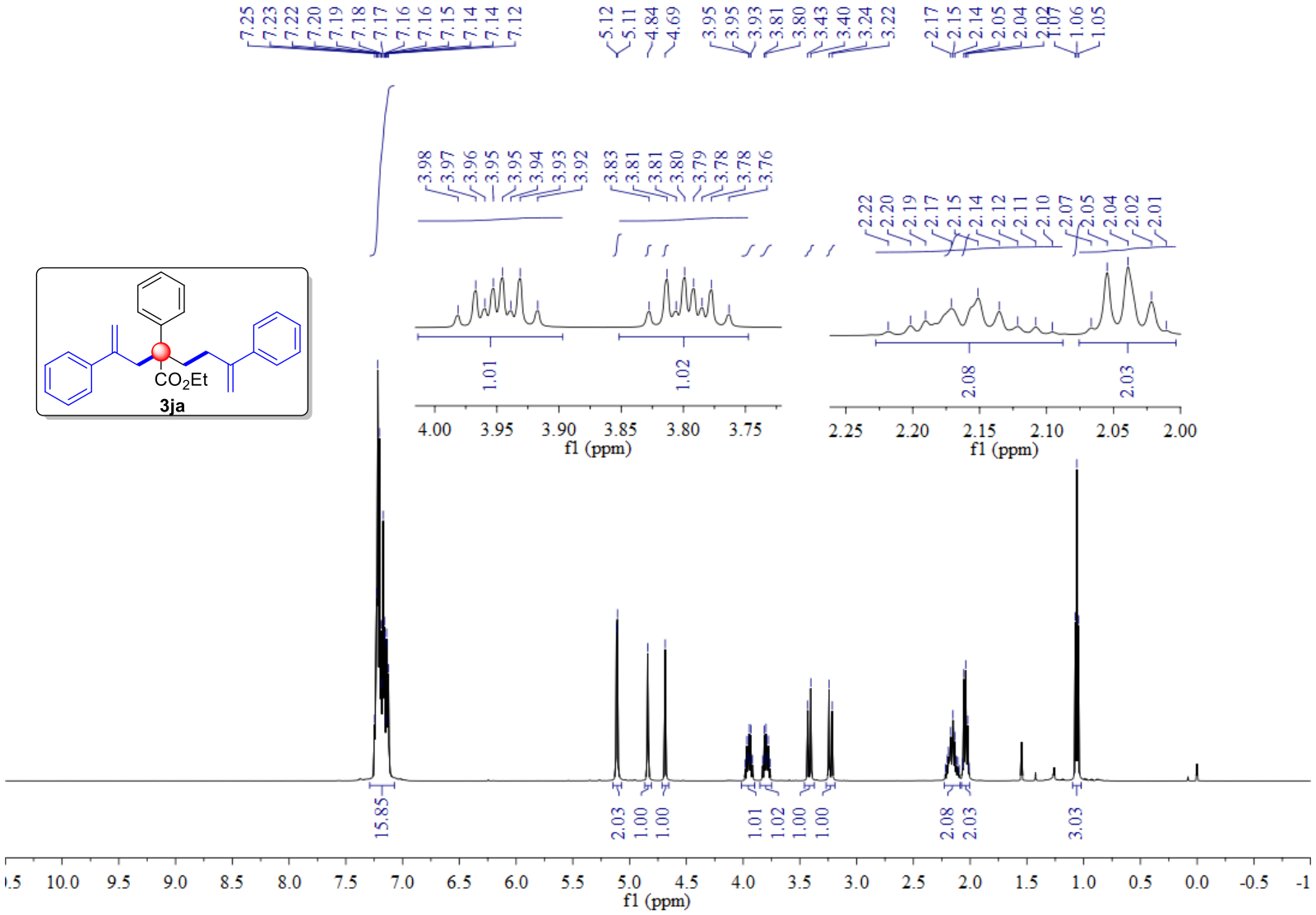

175.30
163.84
163.74
161.87
161.77
148.23
145.75
142.74
141.60
141.25
128.38
128.33
126.24
117.80
113.16
112.62
112.32
102.12
101.91
60.77
50.57
41.66
40.84
32.68
30.05
14.09
Ar1 =
Ar1
F
F
CO2Et
3ga
128.38
128.33
127.60
127.55
126.90
126.24
129
128
127
126
f1 (ppm)
102.32
102.12
101.91
102.5
102.0
101.5
f1 (ppm)
163.84
163.74
161.87
161.77
164
163
162
f1 (ppm)
141.68
141.60
141.53
141.7
141.5
f1 (ppm)
113.16
113.11
113.00
112.96
113.15
113.05
112.95
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

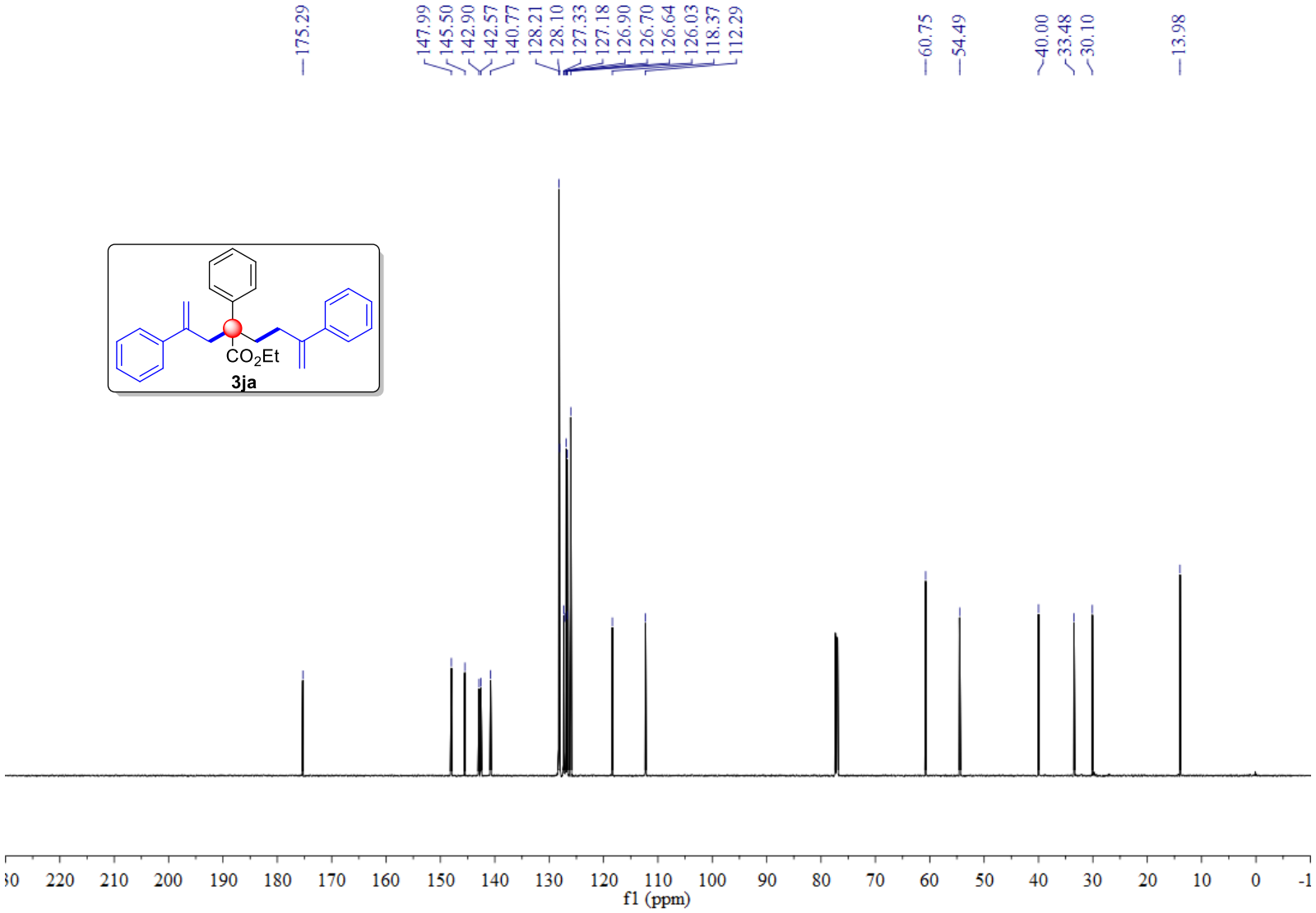

Ar2 = Ar2
CO2Et
3ha
f1 (ppm)

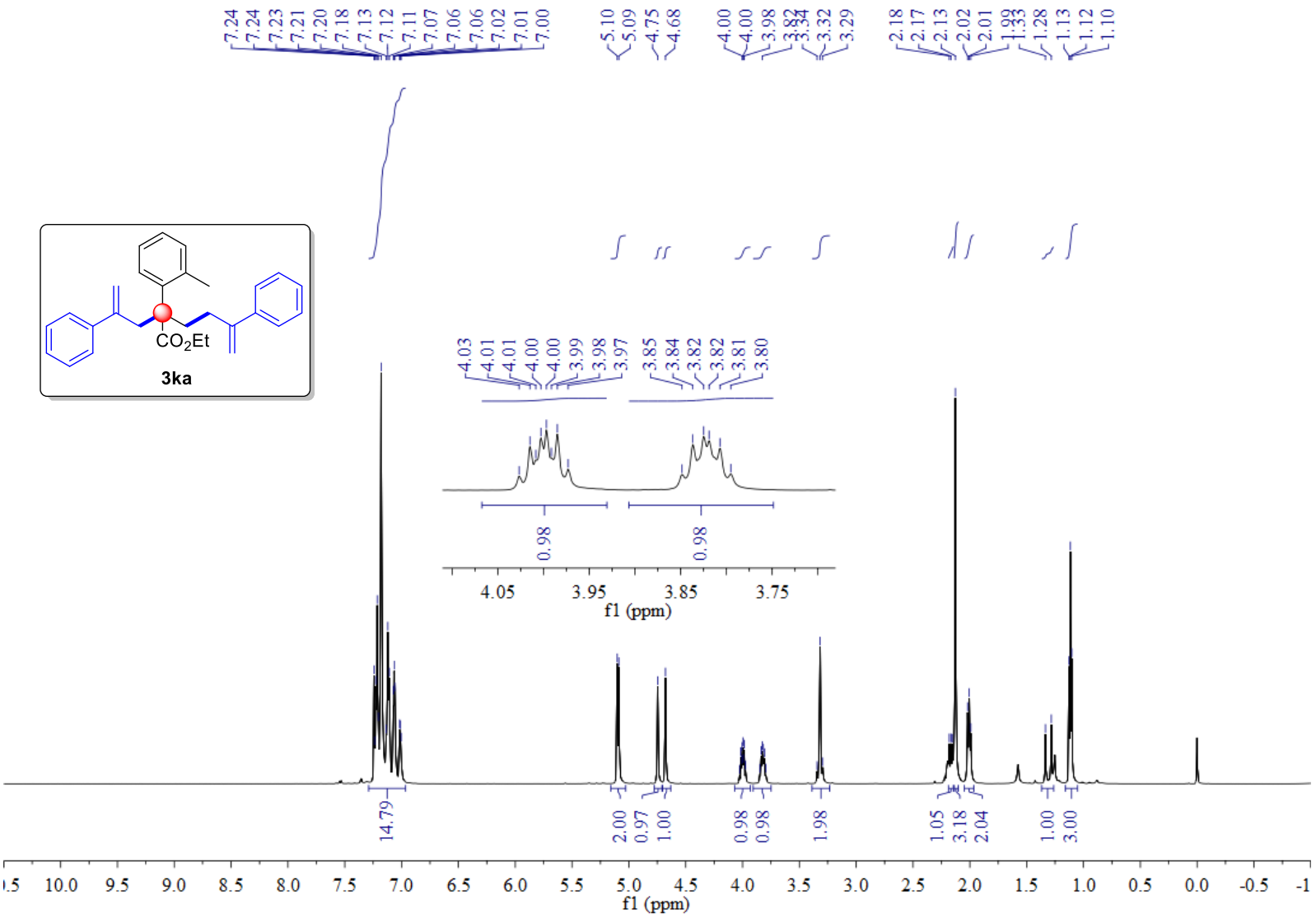

176.35
148.45
146.06
136.19
133.92
130.40
130.10
128.29
128.27
127.40
127.33
126.92
126.82
112.14
60.53
50.22
40.96
38.37
33.05
30.04
21.14
19.84
14.02
Ar2 = -
Ar2
CO2Et
3ha
143.15
141.21
136.19
134.91
133.92
130.40
130.10
142 140 138 136 134 132 130
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

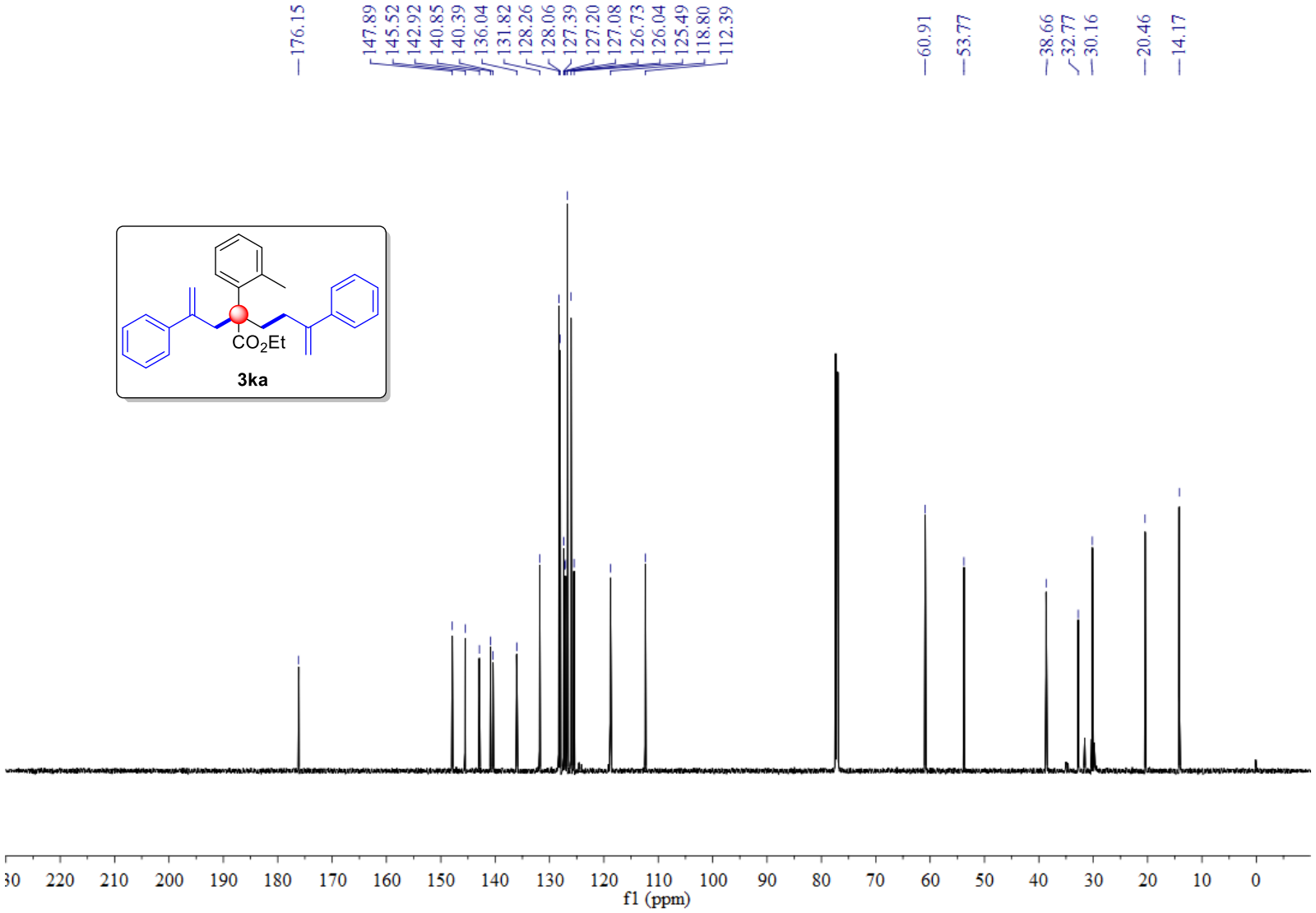

7.32 7.31 7.30 7.30 7.29 7.28 7.28 7.28 7.26 7.25 7.24 7.23 7.23 7.18 7.17 7.17 7.17 7.16 7.10 7.09 7.09 7.08 7.08
5.30 5.16 5.16 5.14 5.14 4.84 4.84
3.01 2.79 2.76 2.75 2.72 2.48 2.47 2.46 2.45 2.44 2.43 2.42 1.66 1.64 1.64 1.63 1.62 1.62 1.61 1.61 1.59 1.38
CO2tBu
3ia
1.66 1.64 1.64 1.63 1.62 1.62 1.61 1.61 1.59
2.04
1.65 1.63 1.61 1.59
f1 (ppm)
2.79 2.76 2.75 2.72
2.00
2.80 2.76 2.72
f1 (ppm)
2.51 2.49 2.48 2.47 2.46 2.45 2.44 2.43 2.42 2.40 2.39
2.15
2.52 2.48 2.44 2.40
f1 (ppm)
11.37 3.00 2.09 0.98 0.96 0.97 1.00 2.00 2.00 2.15 2.04 9.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

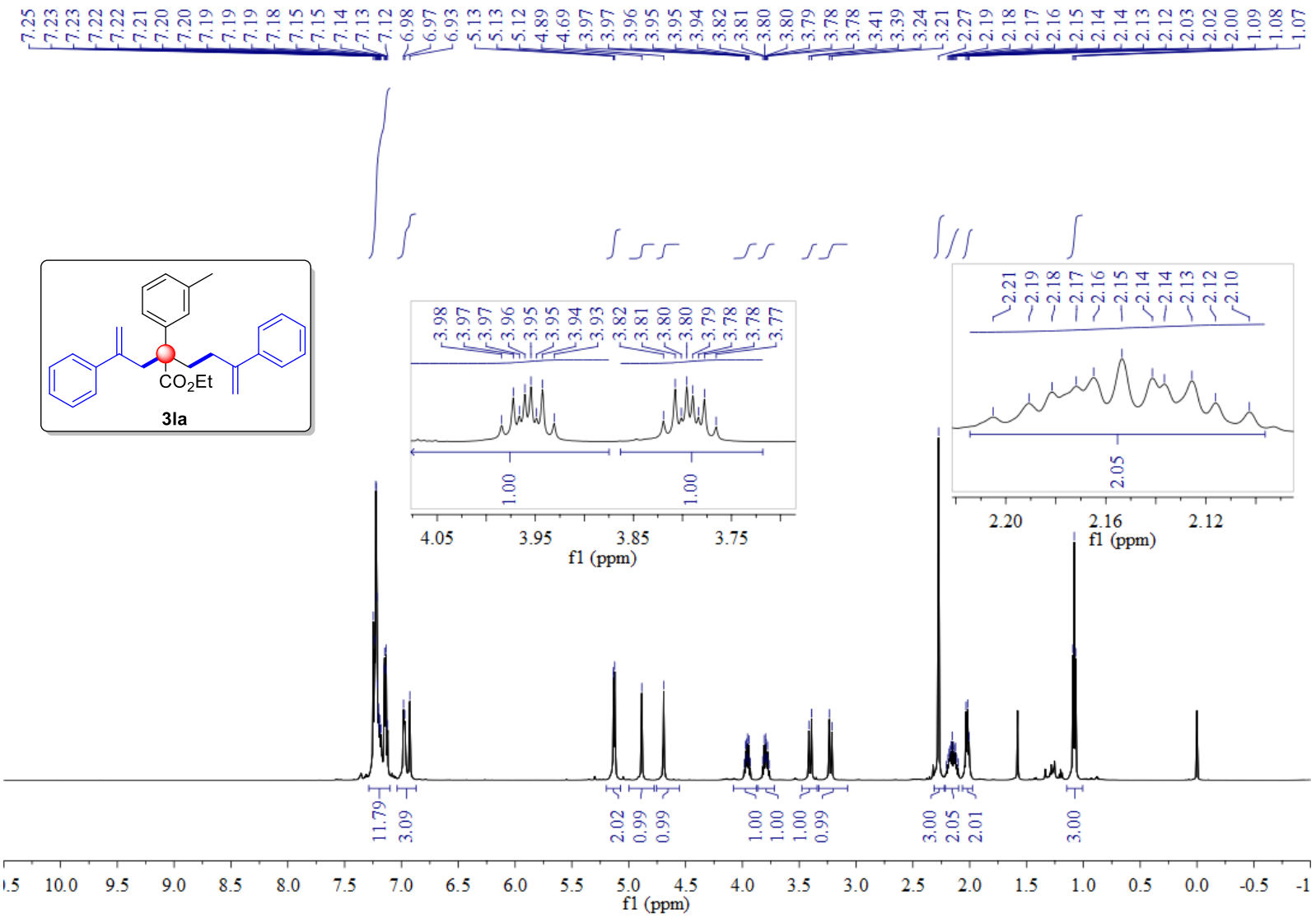

175.27
148.56
145.92
143.72
137.93
130.46
128.37
128.29
128.09
127.40
127.39
126.65
126.41
112.27
80.95
50.53
40.63
40.33
34.10
30.20
28.13
143.72
141.54
137.93
CO2tBu
3ia
144
143
142
141
140
139
138
137
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

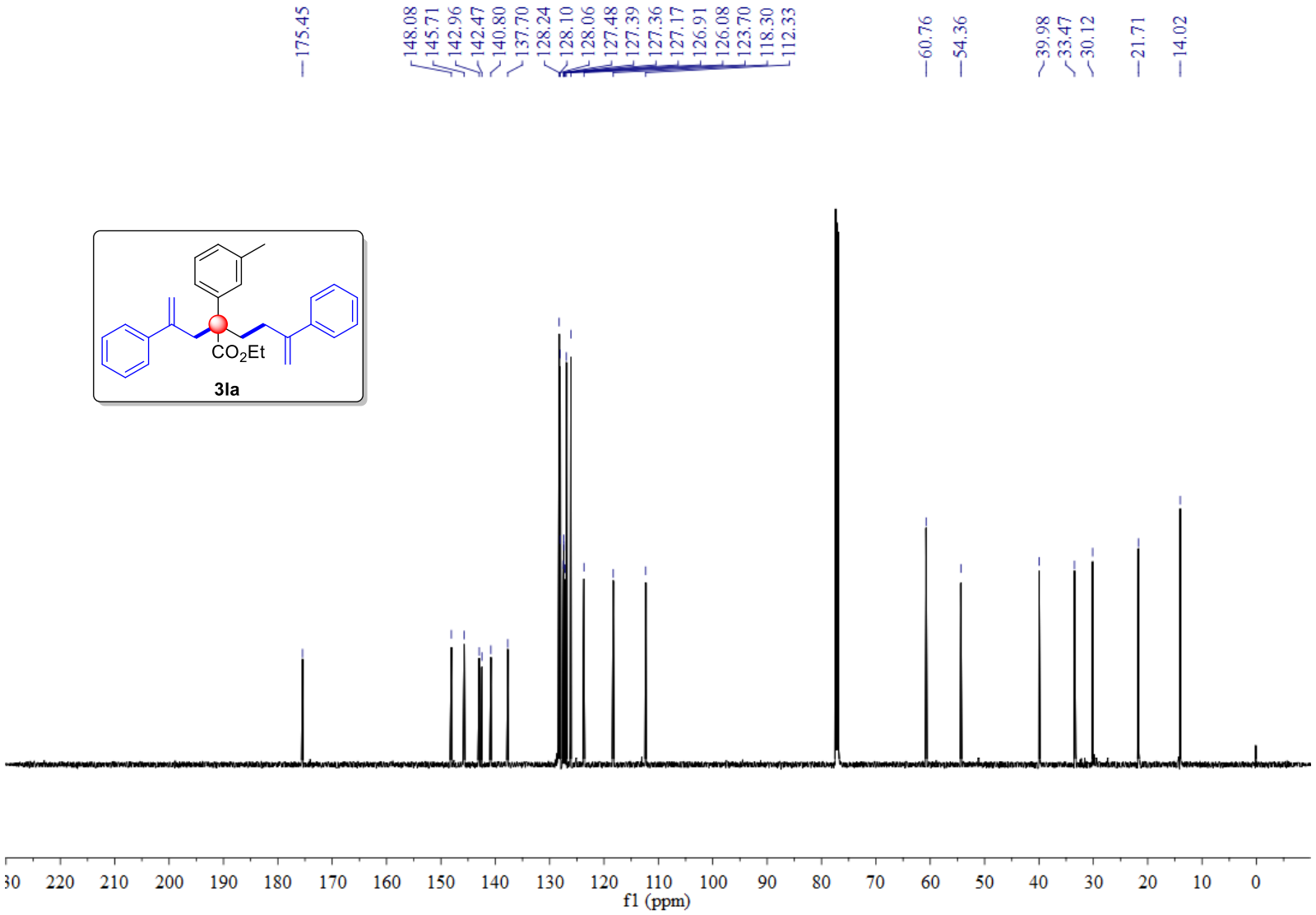

7.25 7.23 7.22 7.20 7.19 7.18 7.17 7.16 7.16 7.15 7.14 7.14 7.12
5.12 5.11 4.84 4.69
3.95 3.95 3.93 3.81 3.80 3.43 3.40 3.24 3.22
2.17 2.15 2.14 2.05 2.04 2.02 1.07 1.06 1.05
3.98 3.97 3.96 3.95 3.95 3.94 3.93 3.92
3.83 3.81 3.81 3.80 3.79 3.78 3.78 3.76
2.22 2.20 2.19 2.17 2.15 2.14 2.12 2.11 2.10 2.07 2.05 2.04 2.02 2.01
CO2Et
3ja
1.01
1.02
2.08
2.03
4.00 3.95 3.90 3.85 3.80 3.75
f1 (ppm)
2.25 2.20 2.15 2.10 2.05 2.00
f1 (ppm)
15.85
2.03 1.00 1.00
1.01 1.02 1.00 1.00
2.08 2.03
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

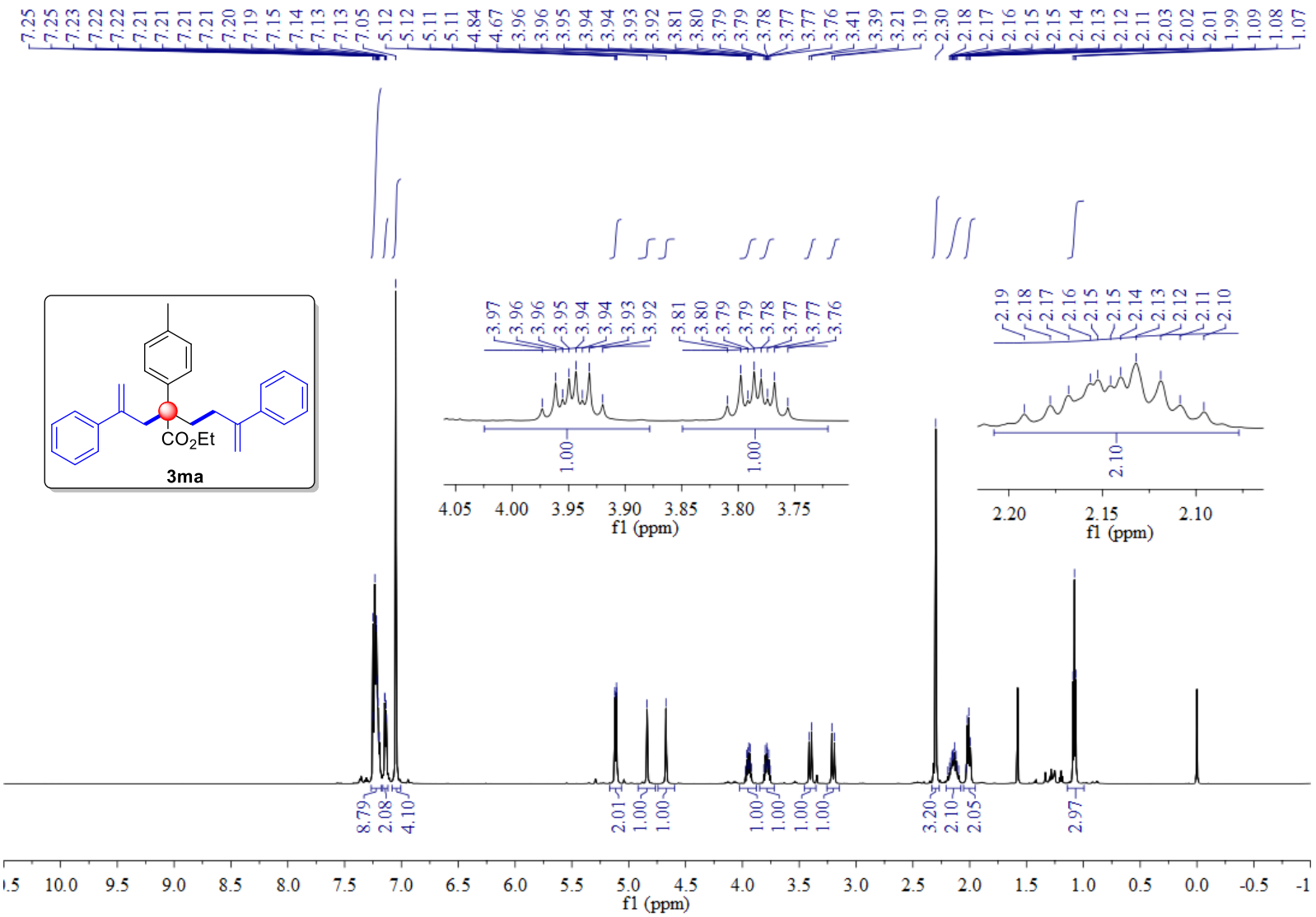

175.29
147.99
145.50
142.90
142.57
140.77
128.21
128.10
127.33
127.18
126.90
126.70
126.64
126.03
118.37
112.29
60.75
54.49
40.00
33.48
30.10
13.98
CO2Et
3ja
f1 (ppm)

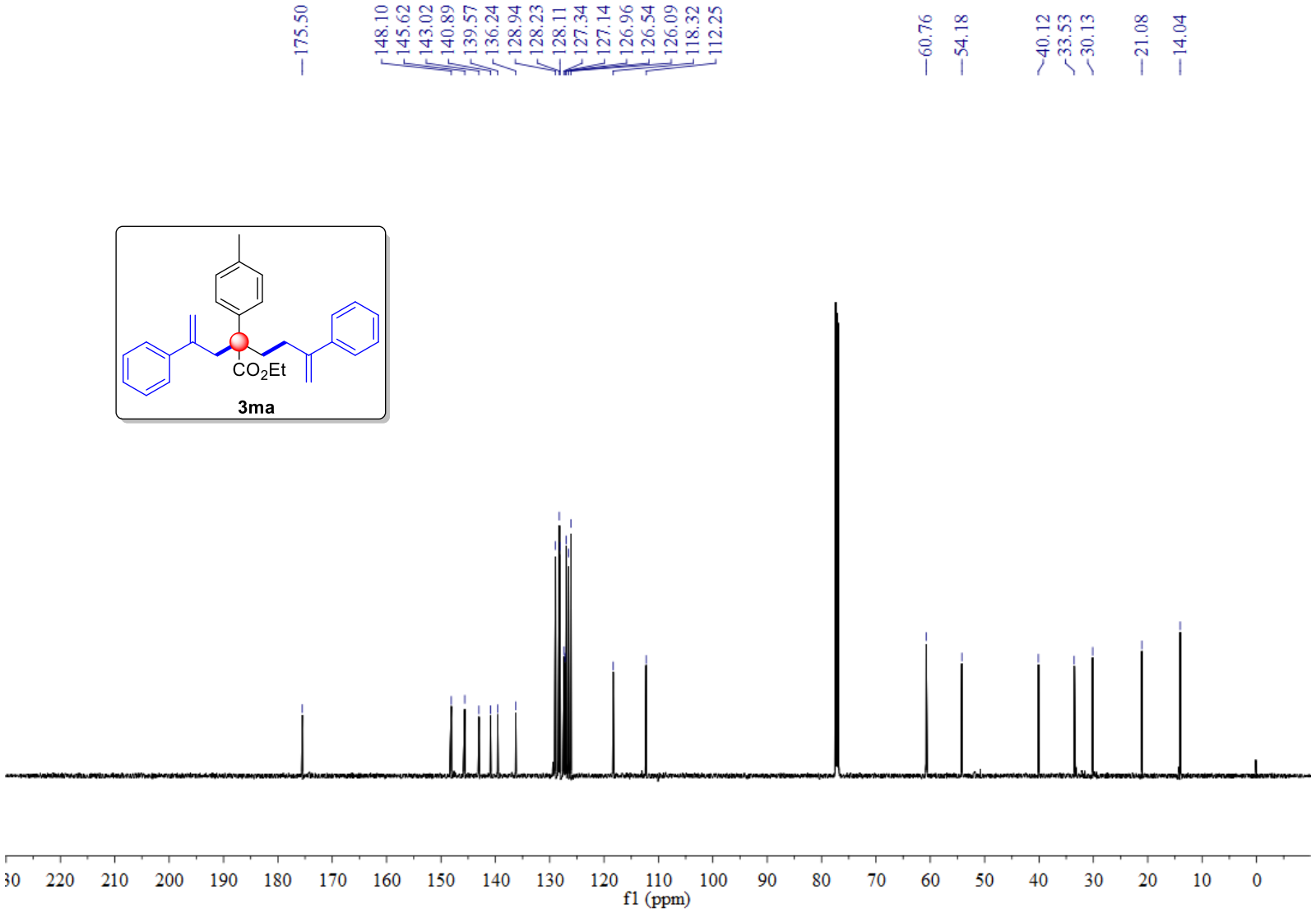

7.24
7.24
7.23
7.21
7.20
7.18
7.13
7.12
7.11
7.07
7.06
7.06
7.02
7.01
7.00
5.10
5.09
4.75
4.68
4.00
4.00
3.98
3.82
3.34
3.32
3.29
2.18
2.17
2.13
2.02
2.01
1.99
1.33
1.28
1.13
1.12
1.10
CO2Et
3ka
4.03
4.01
4.01
4.00
4.00
3.99
3.98
3.97
3.85
3.84
3.82
3.82
3.81
3.80
0.98
0.98
4.05
3.95
3.85
3.75
f1 (ppm)
14.79
2.00
0.97
1.00
0.98
0.98
1.98
1.05
3.18
2.04
1.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

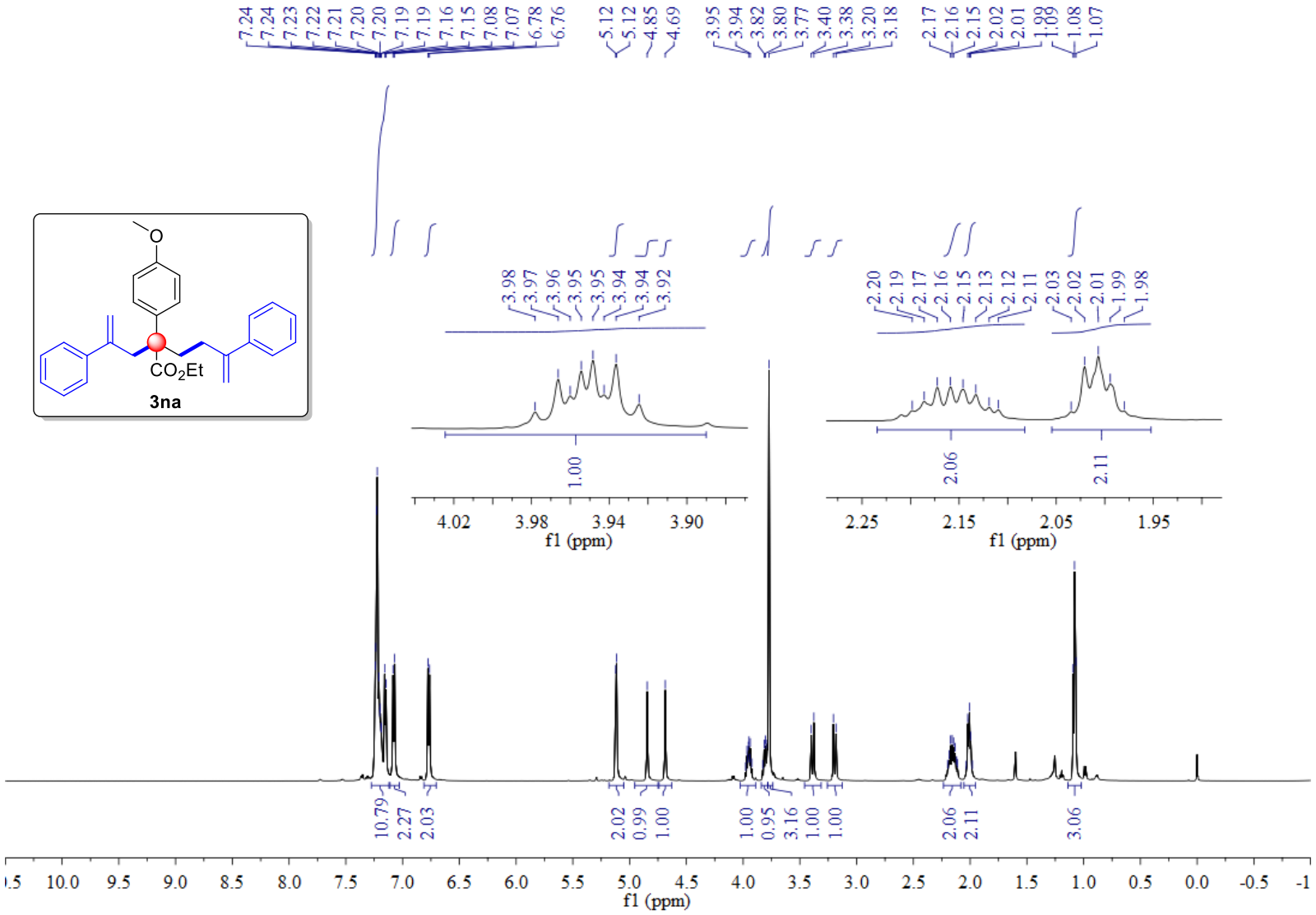

176.15
147.89
145.52
142.92
140.85
140.39
136.04
131.82
128.26
128.06
127.39
127.20
127.08
126.73
126.04
125.49
118.80
112.39
60.91
53.77
38.66
32.77
30.16
20.46
14.17
CO2Et
3ka
f1 (ppm)

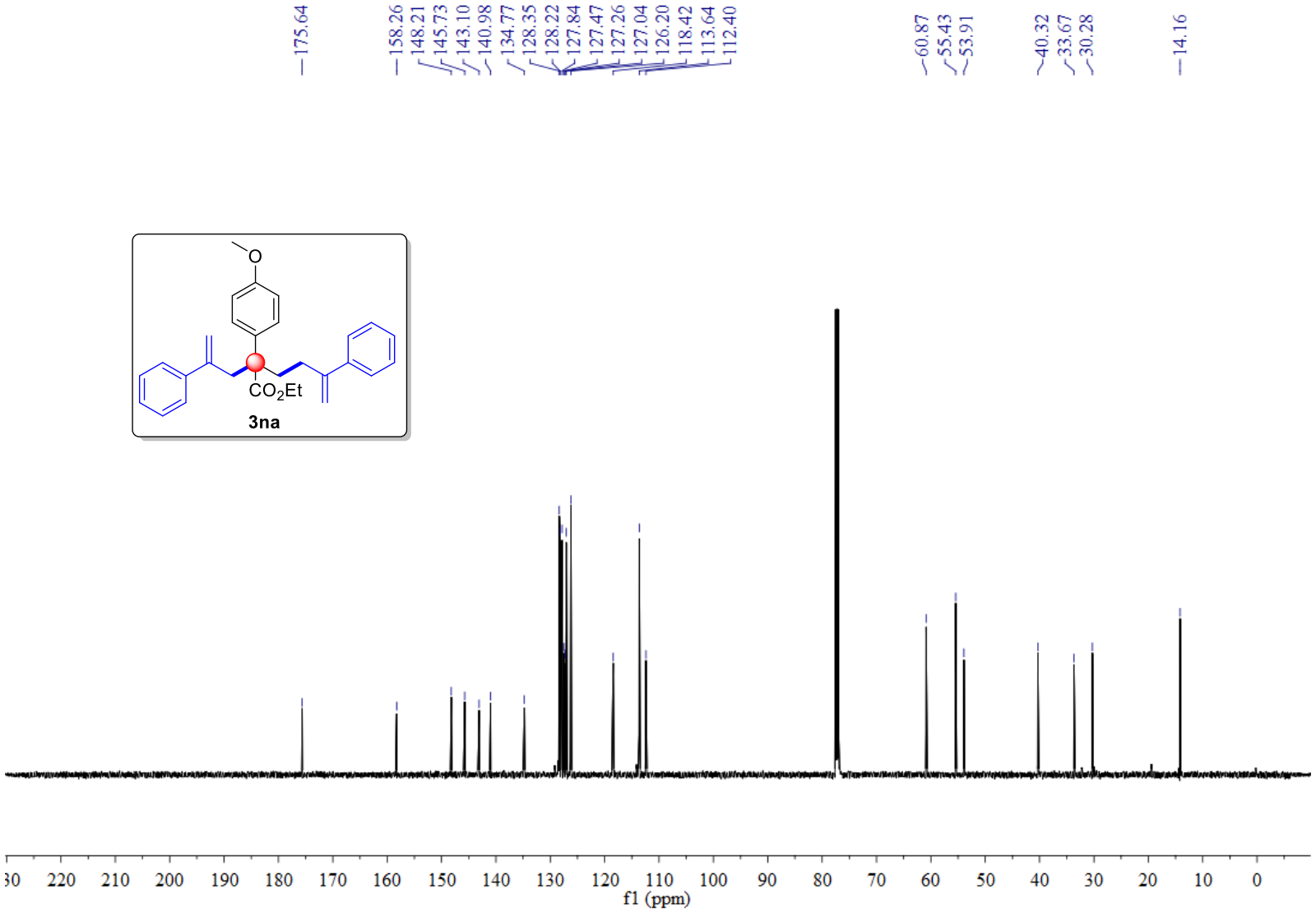

CO2Et
3Ia
f1 (ppm)

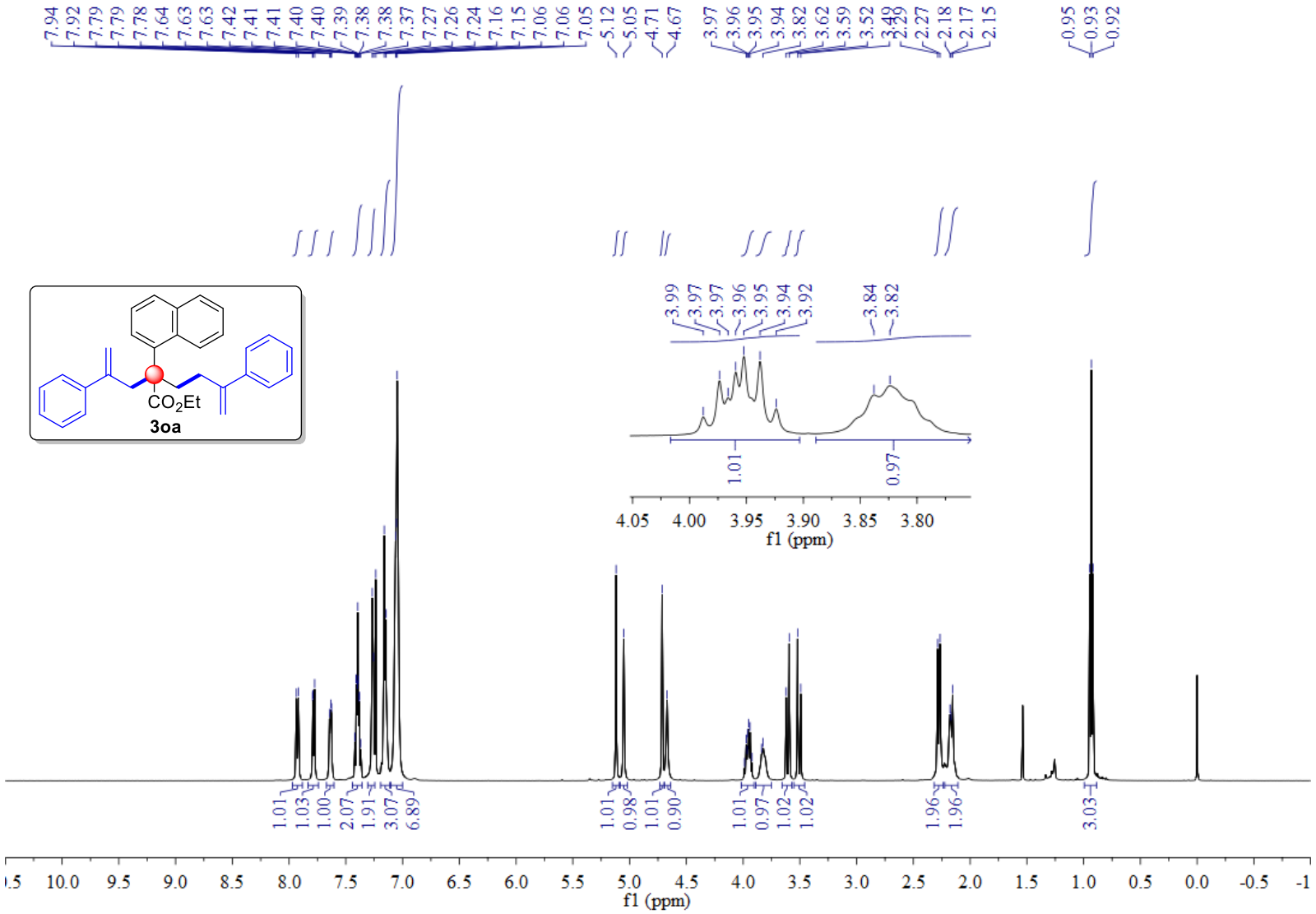

175.45
148.08
145.71
142.96
142.47
140.80
137.70
128.24
128.10
128.06
127.48
127.39
127.36
127.17
126.91
126.08
123.70
118.30
112.33
60.76
54.36
39.98
33.47
30.12
21.71
14.02
CO2Et
3la
f1 (ppm)

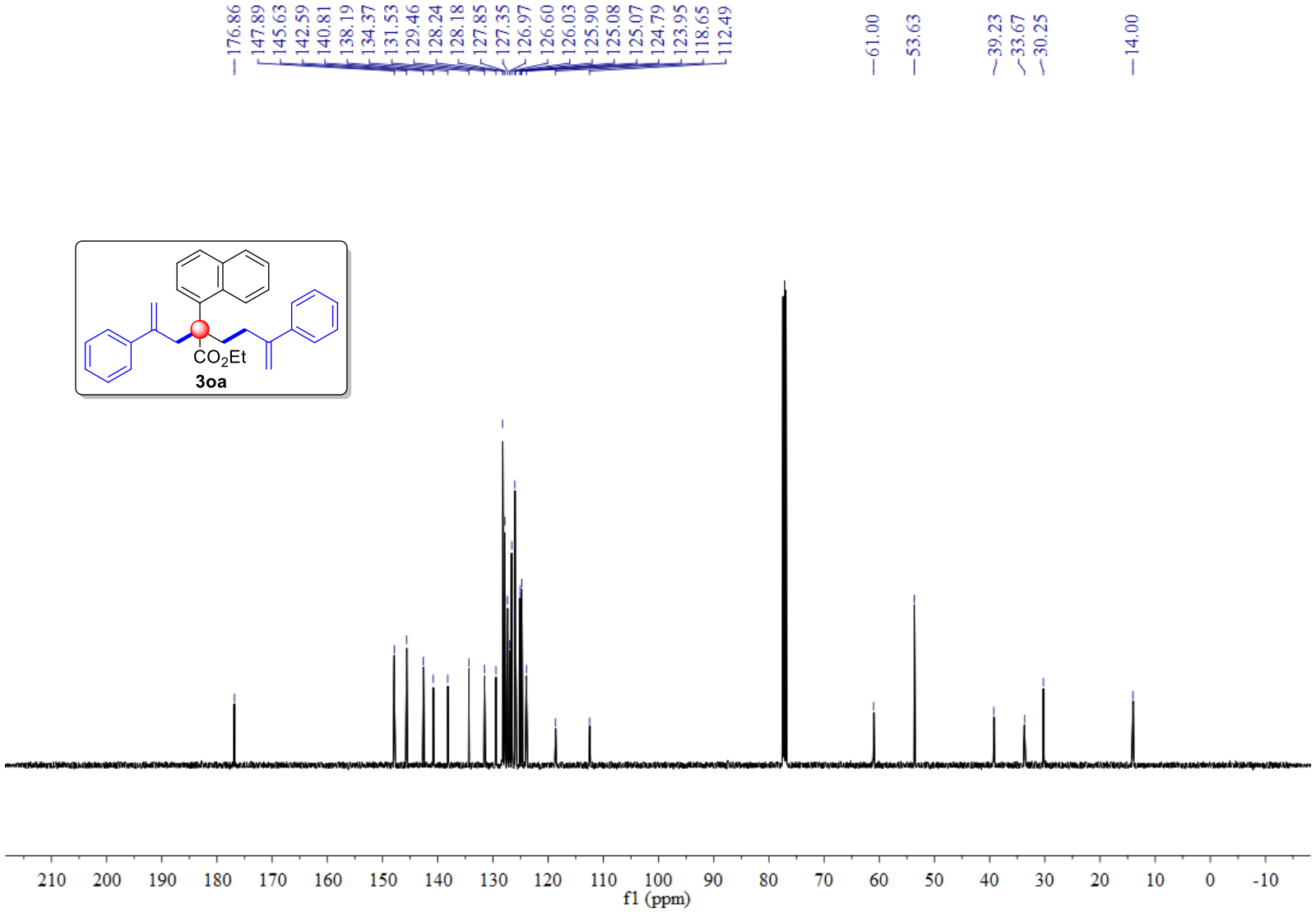

CO2Et
3ma
f1 (ppm)


175.50
148.10
145.62
143.02
140.89
139.57
136.24
128.94
128.23
128.11
127.34
127.14
126.96
126.54
126.09
118.32
112.25
60.76
54.18
40.12
33.53
30.13
21.08
14.04
CO2Et
3ma
f1 (ppm)


7.24 7.24 7.23 7.22 7.21 7.20 7.20 7.19 7.19 7.16 7.15 7.08 7.07 6.78 6.76
5.12 5.12 4.85 4.69
3.95 3.94 3.82 3.80 3.77 3.40 3.38 3.20 3.18
2.17 2.16 2.15 2.02 2.01 1.88 1.89 1.08 1.07
O
CO2Et
3na
3.98 3.97 3.96 3.95 3.95 3.94 3.94 3.92
1.00
4.02 3.98 3.94 3.90
f1 (ppm)
2.20 2.19 2.17 2.16 2.15 2.13 2.12 2.11 2.03 2.02 2.01 1.99 1.98
2.06 2.11
2.25 2.15 2.05 1.95
f1 (ppm)
10.79 2.27 2.03 2.02 0.99 1.00 1.00 0.95 3.16 1.00 1.00 2.06 2.11 3.06
f1 (ppm)


175.64
158.26
148.21
145.73
143.10
140.98
134.77
128.35
128.22
127.84
127.47
127.26
127.04
126.20
118.42
113.64
112.40
60.87
55.43
53.91
40.32
33.67
30.28
14.16
O
CO2Et
3na
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.94 7.92 7.79 7.79 7.78 7.64 7.63 7.63 7.42 7.41 7.41 7.40 7.40 7.39 7.38 7.38 7.37 7.27 7.26 7.24 7.16 7.15 7.06 7.06 7.05 5.12 5.05 4.71 4.67 3.97 3.96 3.95 3.94 3.82 3.62 3.59 3.52 2.29 2.27 2.18 2.17 2.15 0.95 0.93 0.92
CO2Et
3oa
3.99 3.97 3.97 3.96 3.95 3.94 3.92 3.84 3.82
1.01 0.97
4.05 4.00 3.95 3.90 3.85 3.80
f1 (ppm)
1.01 1.03 1.00 2.07 1.91 3.07 6.89 1.01 0.98 1.01 0.90 1.01 0.97 1.02 1.02 1.96 1.96 3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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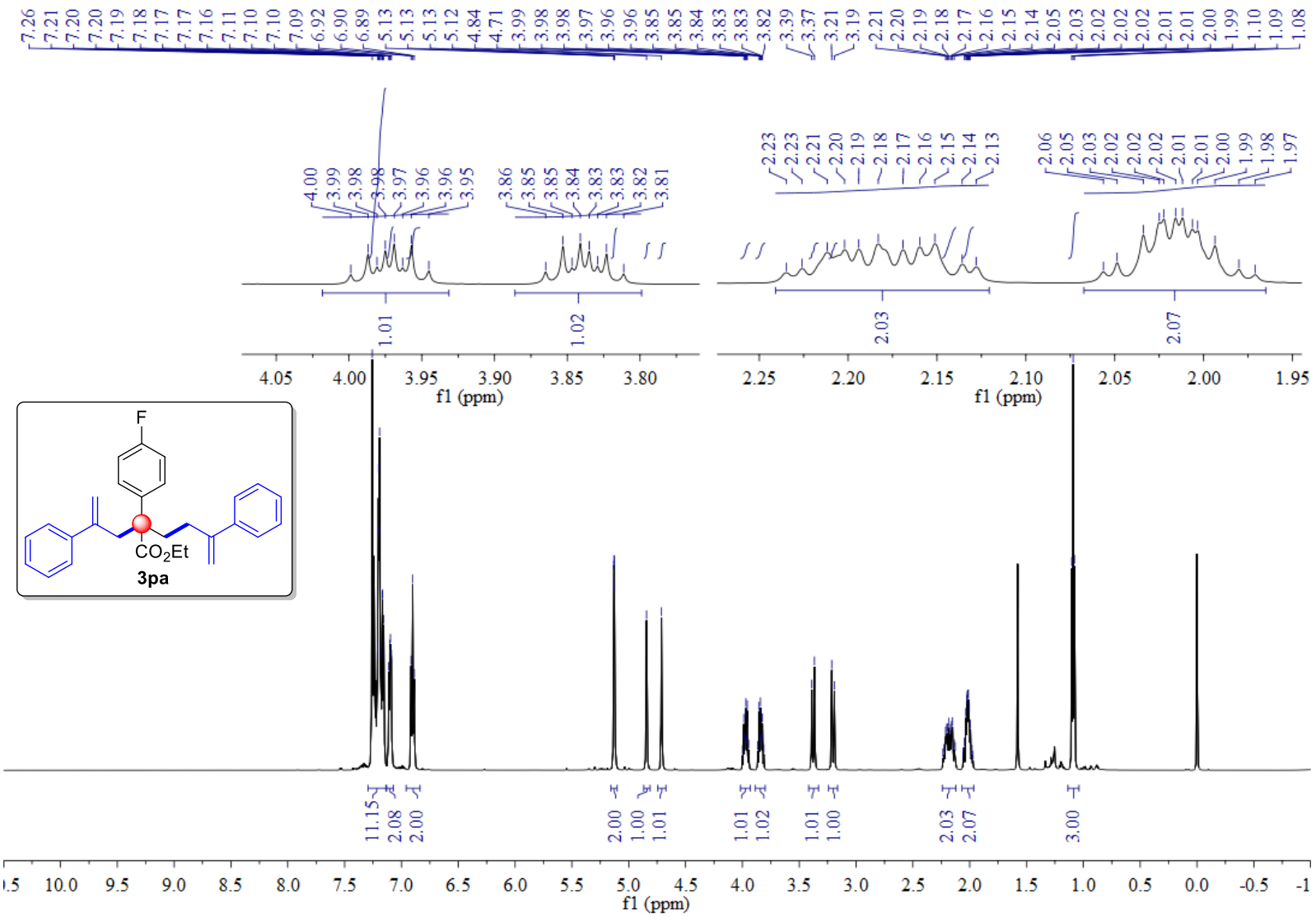
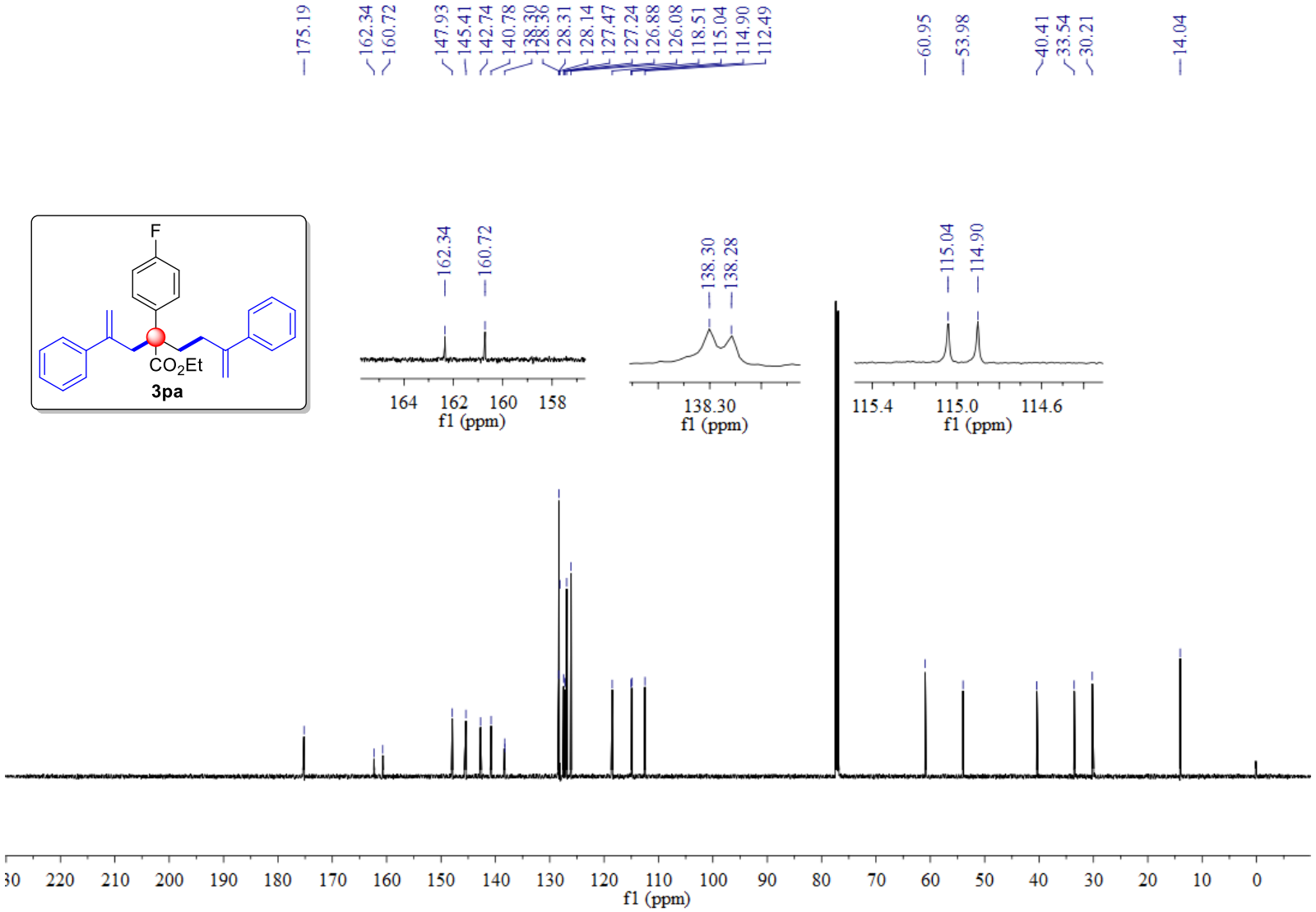
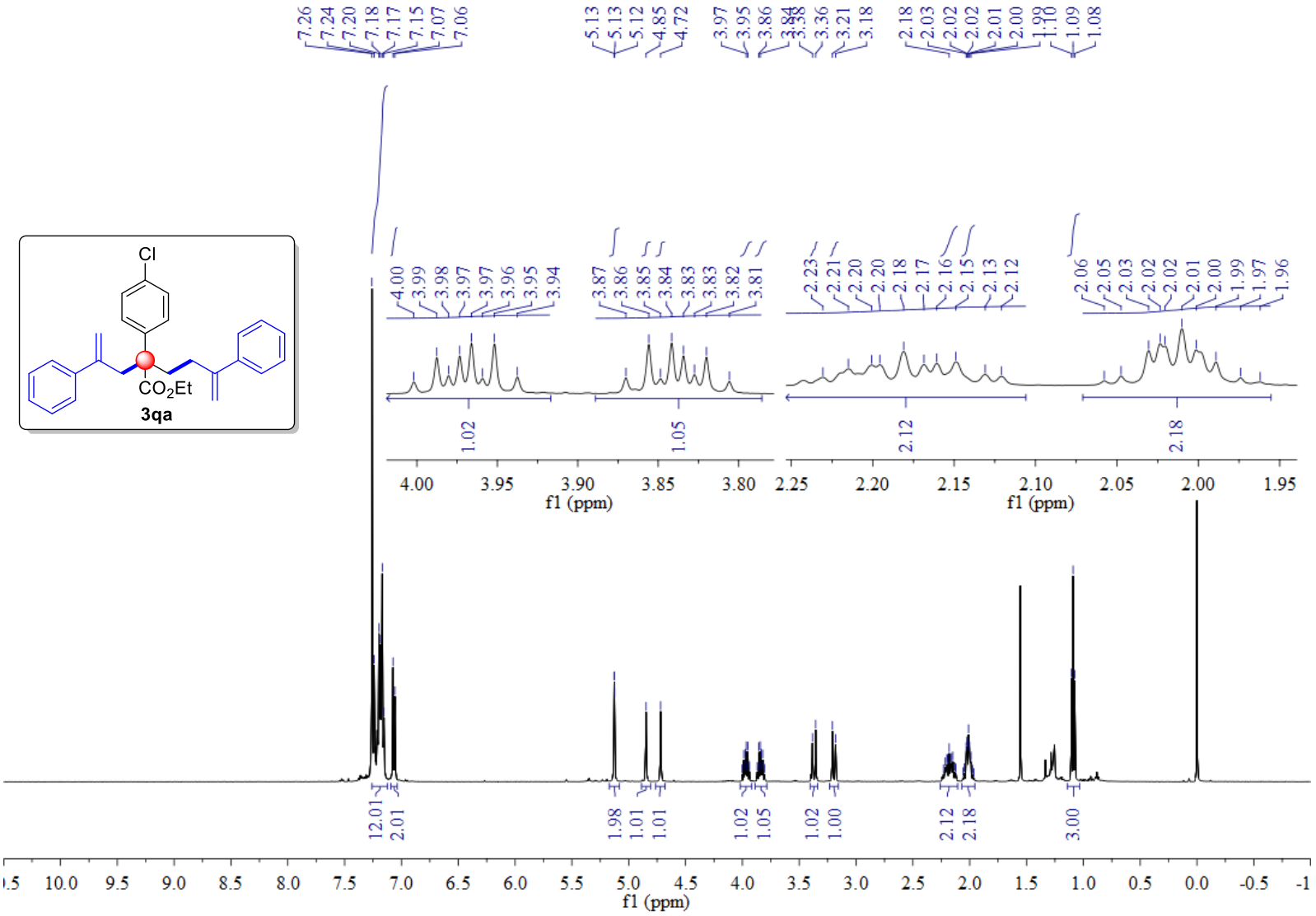
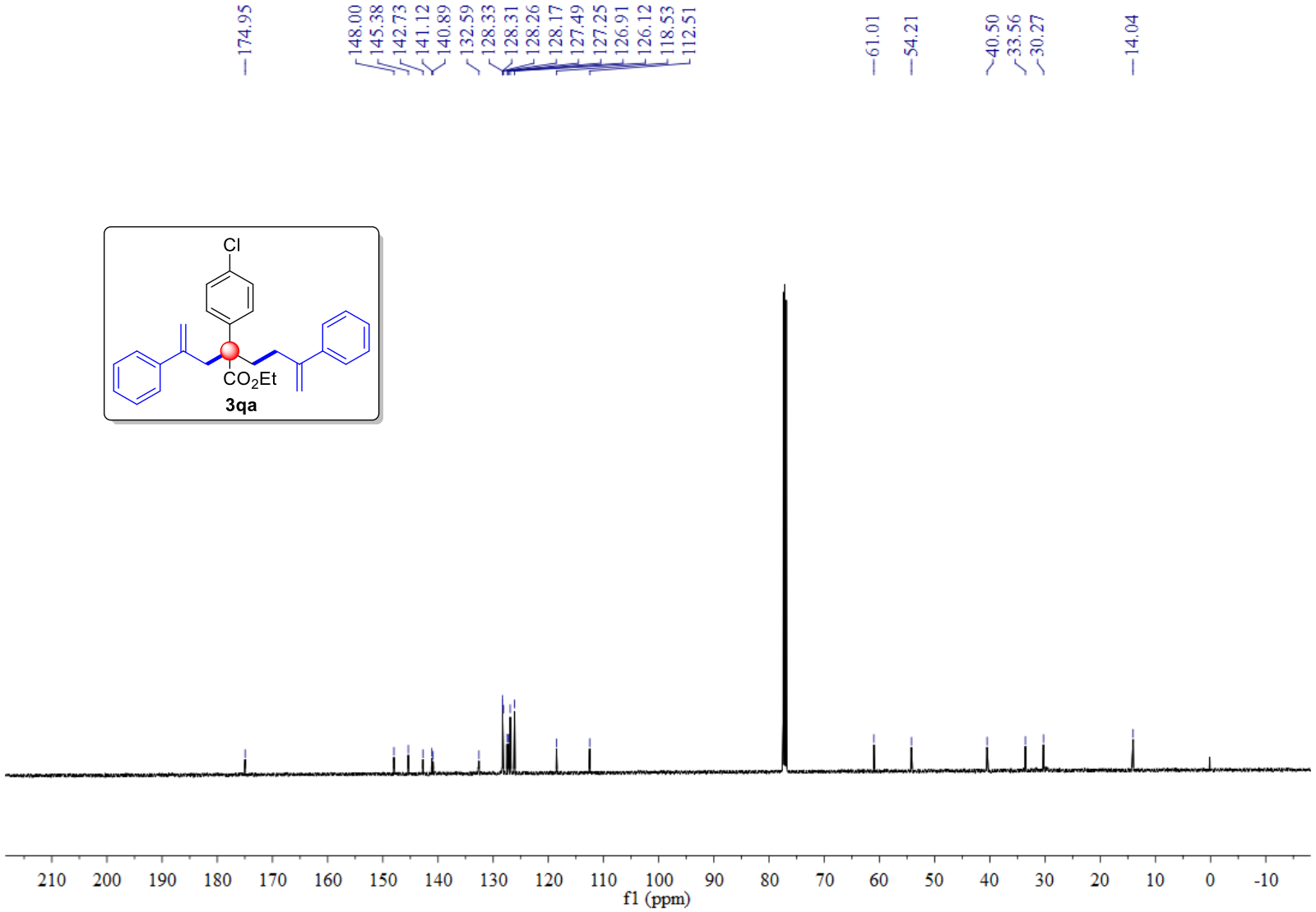
145.63
142.59
140.81
138.19
134.37
131.53
129.46
128.24
128.18
127.85
127.35
126.97
126.60
126.03
125.90
125.08
125.07
124.79
123.95
118.65
112.49
61.00
53.63
39.23
33.67
30.25
14.00
CO2Et
3oa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


F
CO2Et
3pa
f1 (ppm)


175.19
162.34
160.72
147.93
145.41
142.74
140.78
138.30
138.28
128.31
128.14
127.47
127.24
126.88
126.08
118.51
115.04
114.90
112.49
60.95
53.98
40.41
33.54
30.21
14.04
F
CO2Et
3pa
162.34
160.72
164
162
160
158
f1 (ppm)
138.30
138.28
138.30
f1 (ppm)
115.04
114.90
115.4
115.0
114.6
f1 (ppm)
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
CO2Et
3qa
f1 (ppm)


174.95
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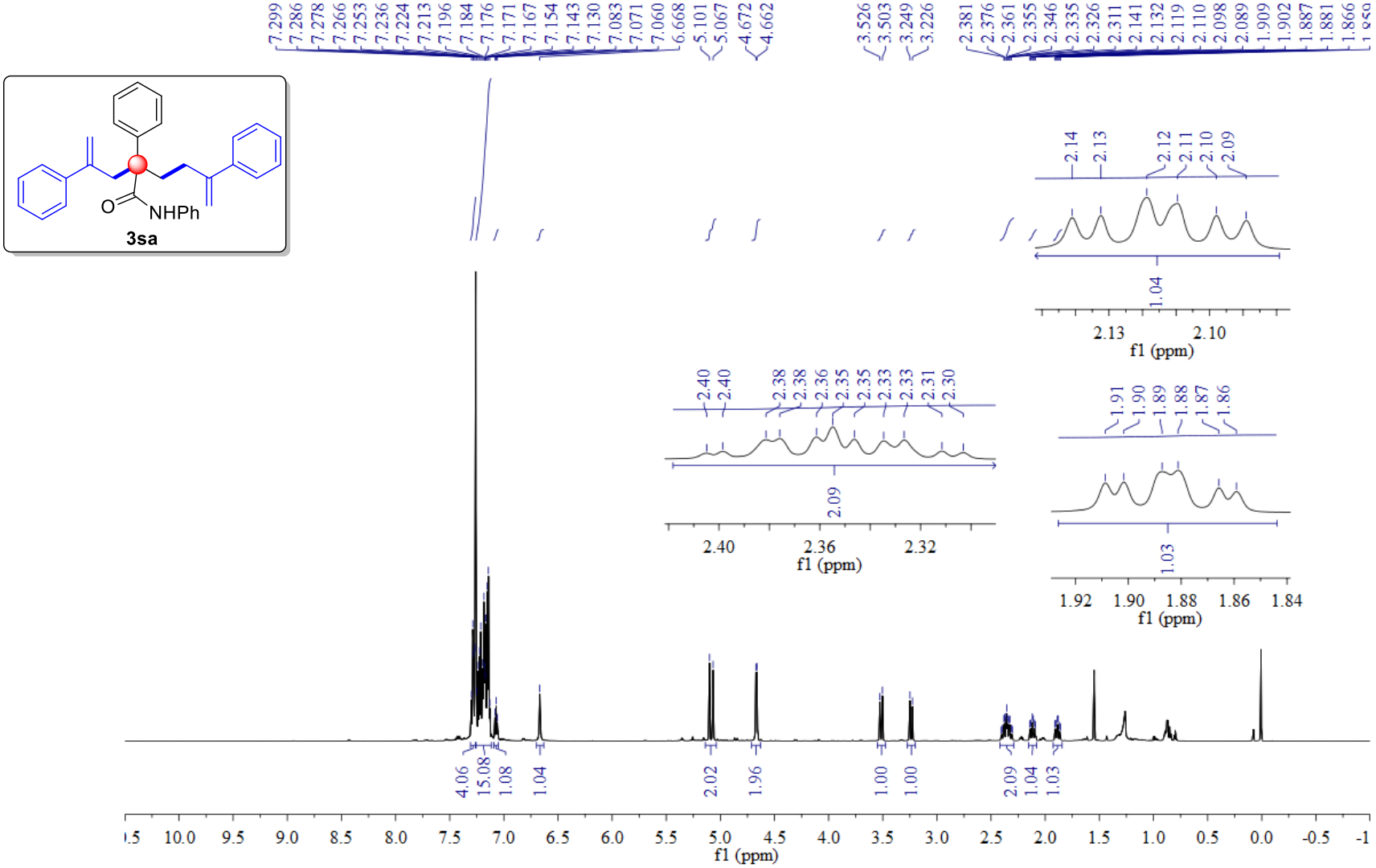
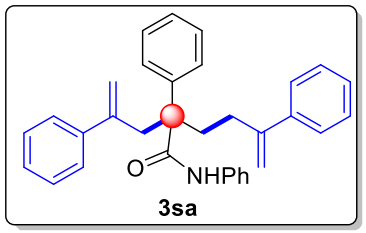
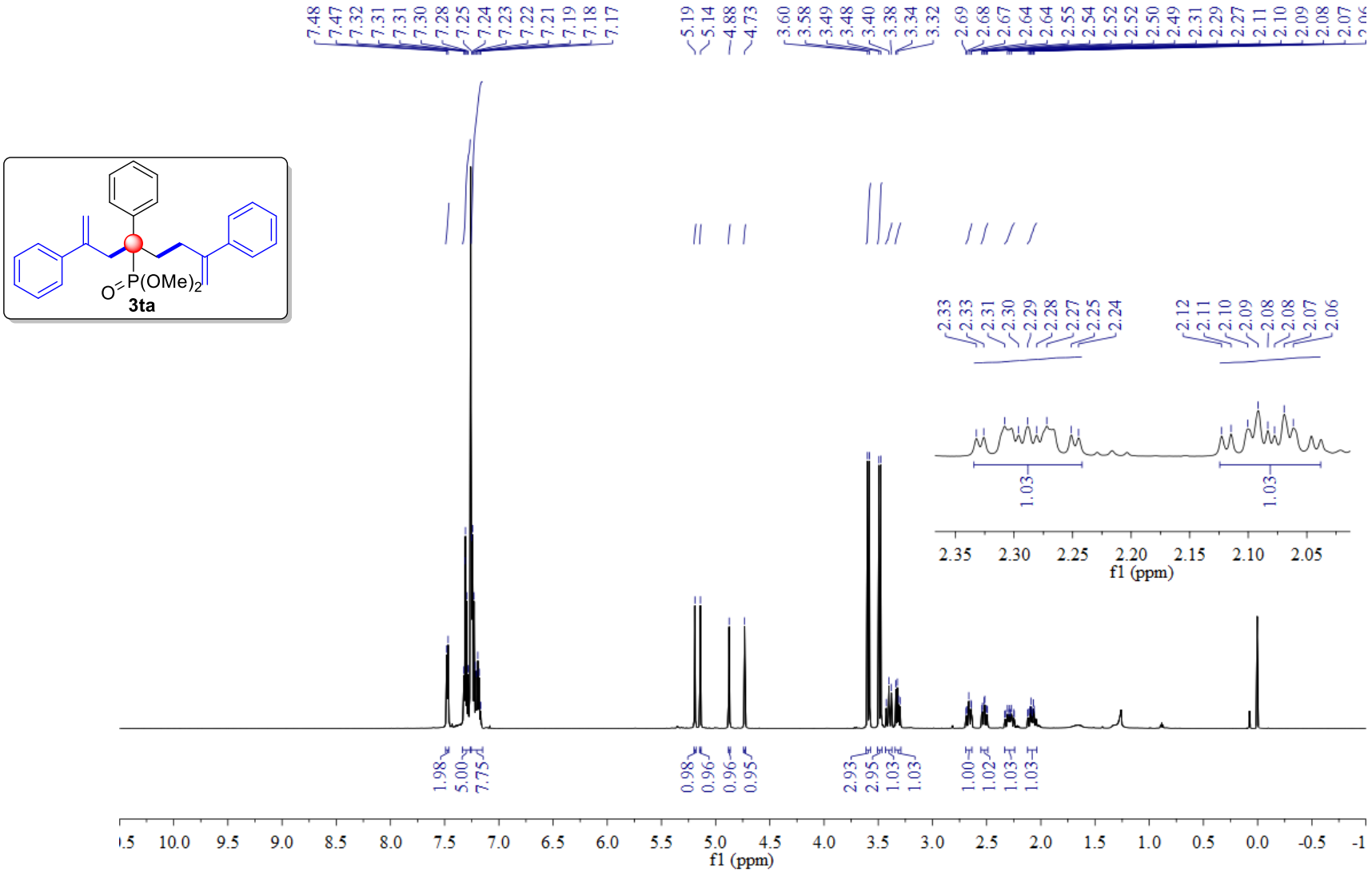
142.73
141.12
140.89
132.59
128.33
128.31
128.26
128.17
127.49
127.25
126.91
126.12
118.53
112.51
61.01
54.21
40.50
33.56
30.27
14.04
Cl
CO2Et
3qa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

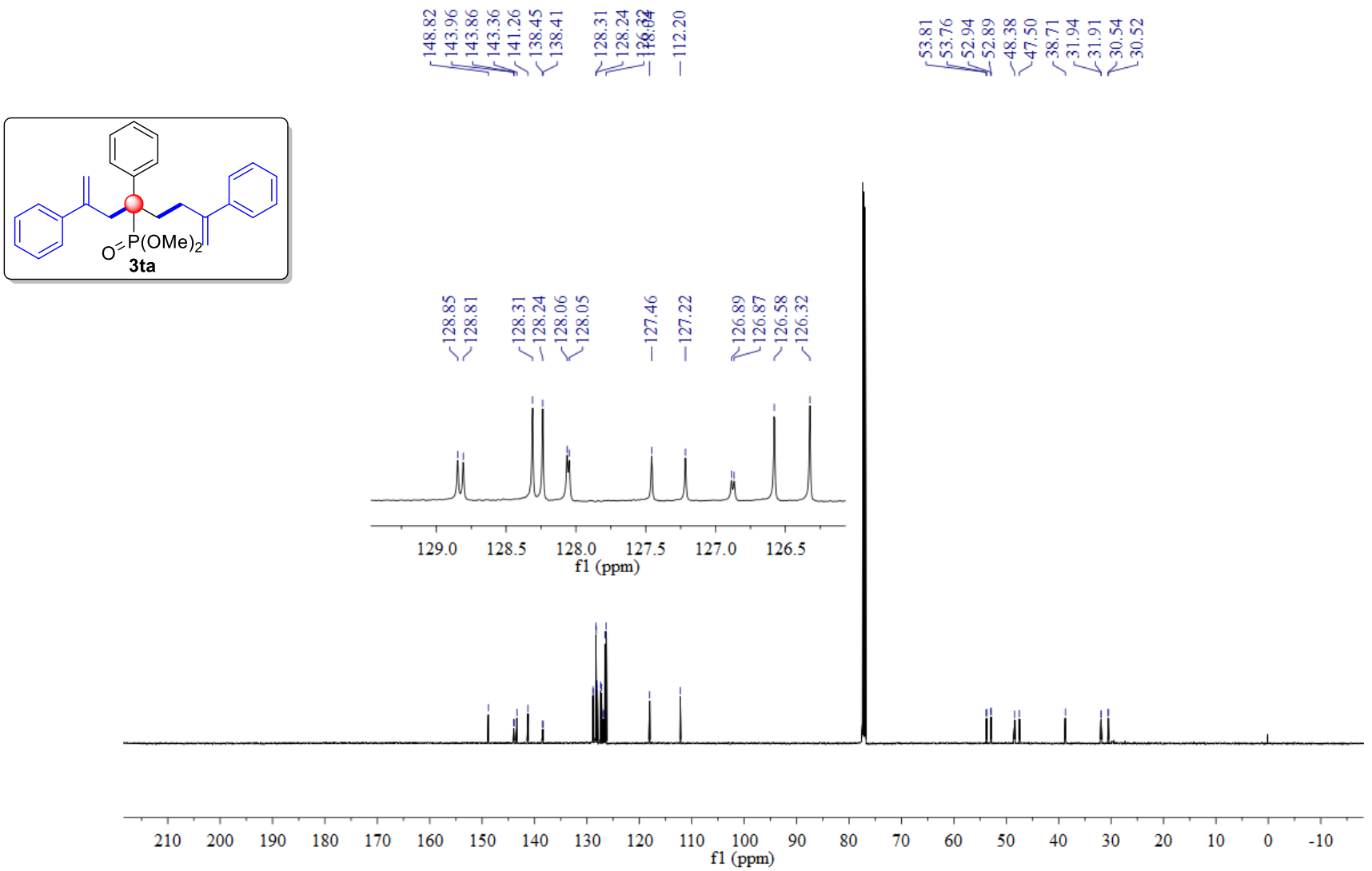

O
NHPh
3sa
f1 (ppm)


O
NHPh

3sa

173.99
147.82
145.43
143.19
142.57
140.64
137.90
129.00
128.62
128.37
128.19
127.73
127.46
127.43
127.06
126.78
126.12
124.28
119.86
118.54
112.69
55.99
40.38
33.89
30.30
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O=P(OMe)2
3ta
f1 (ppm)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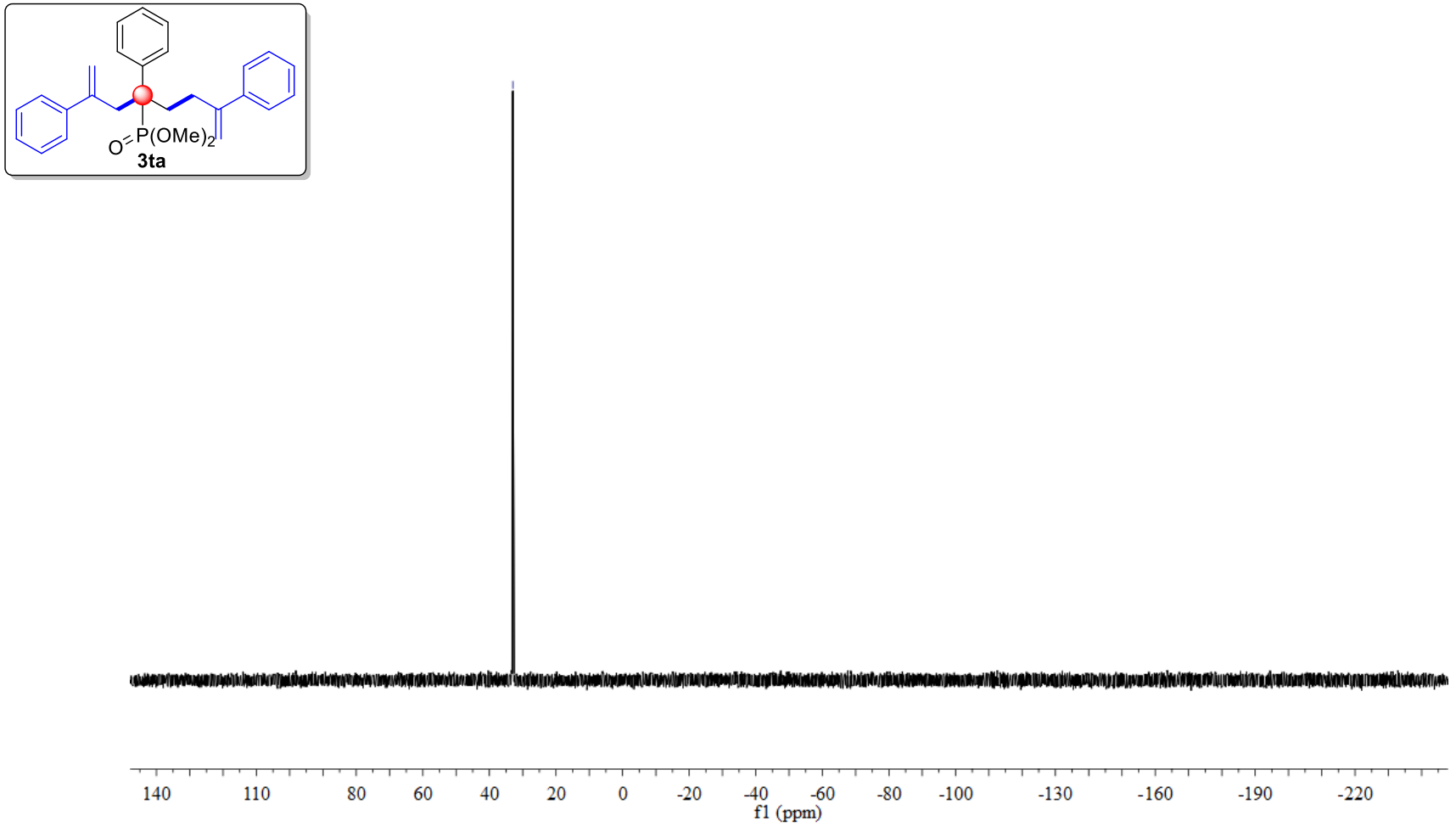
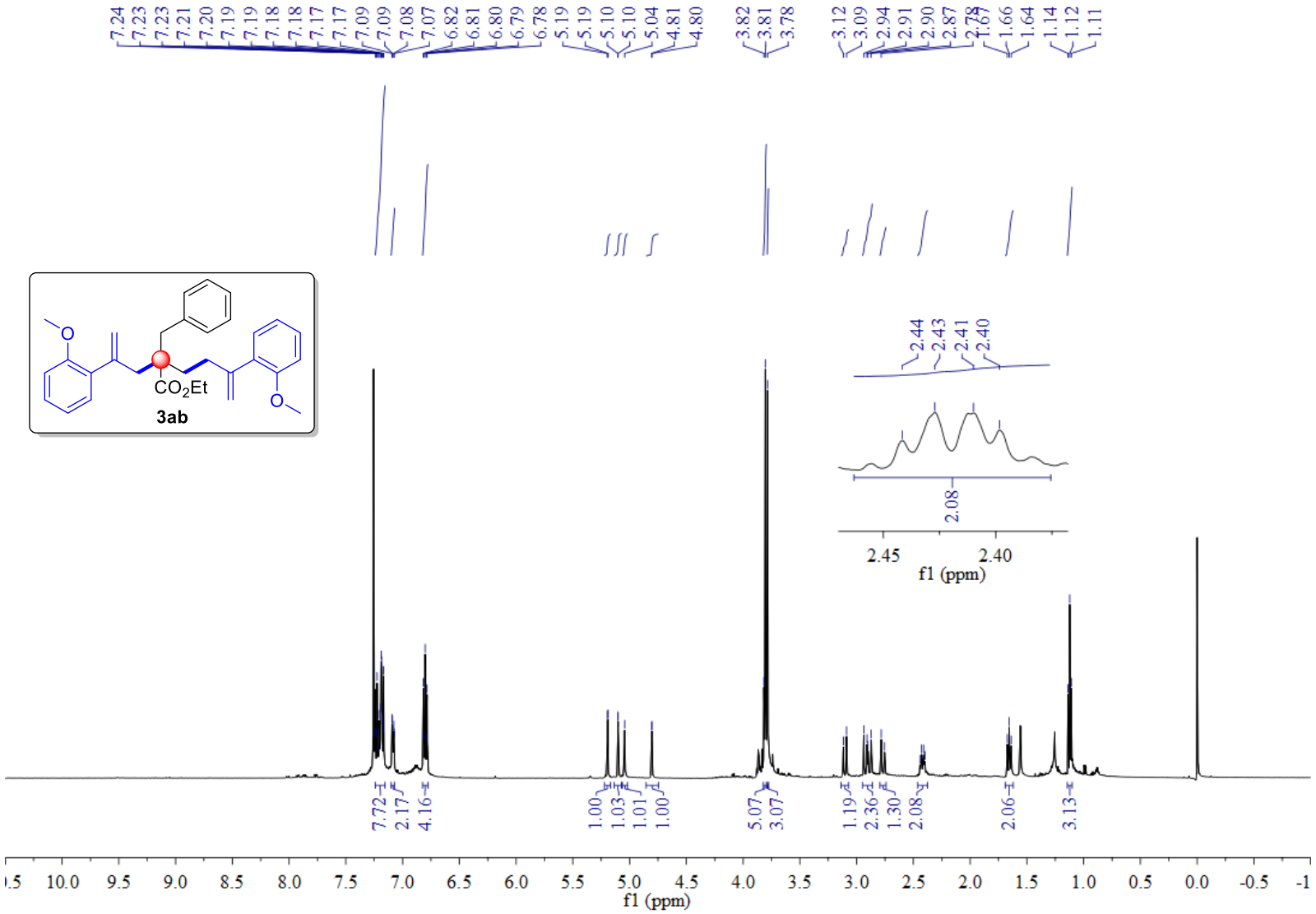
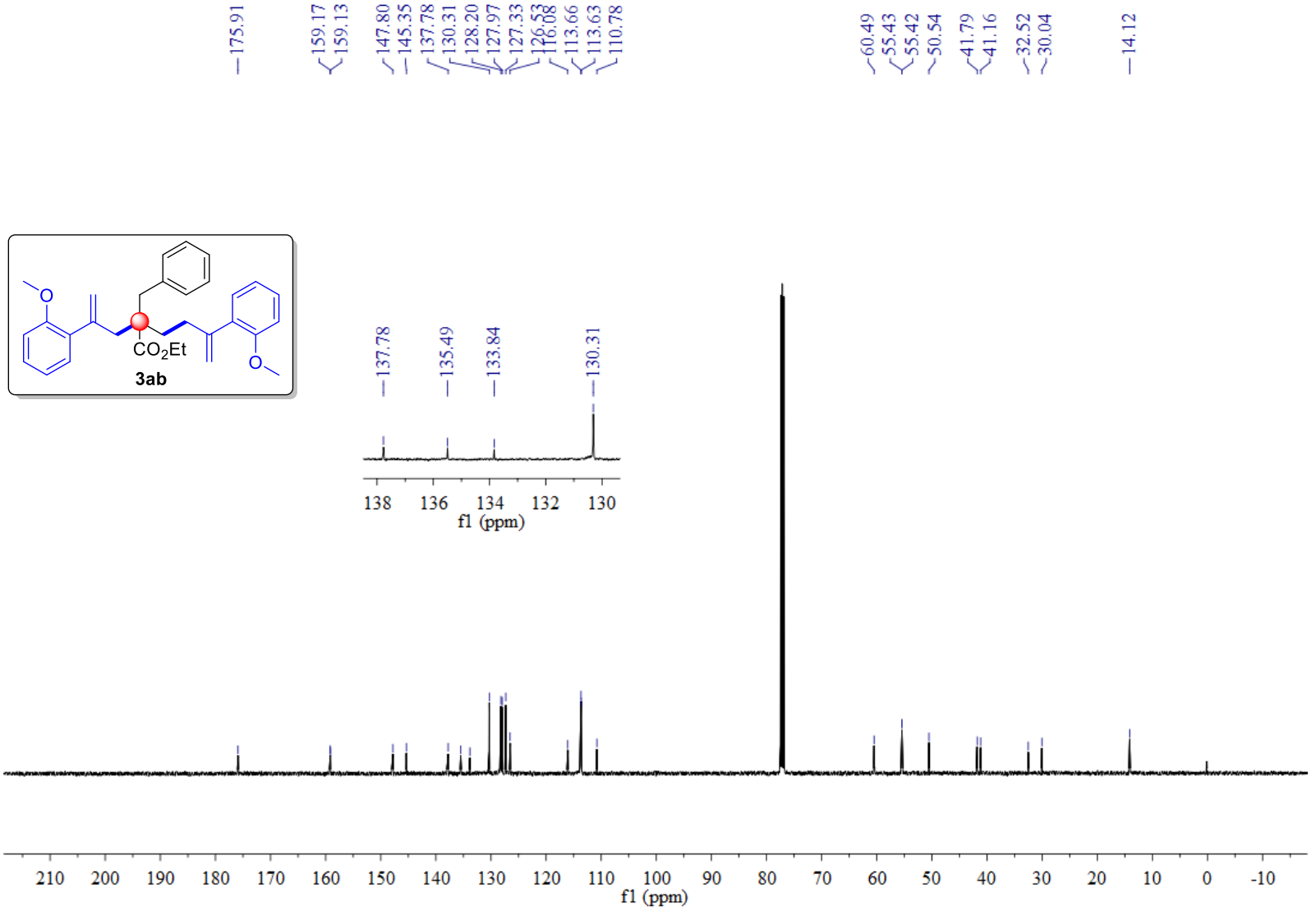
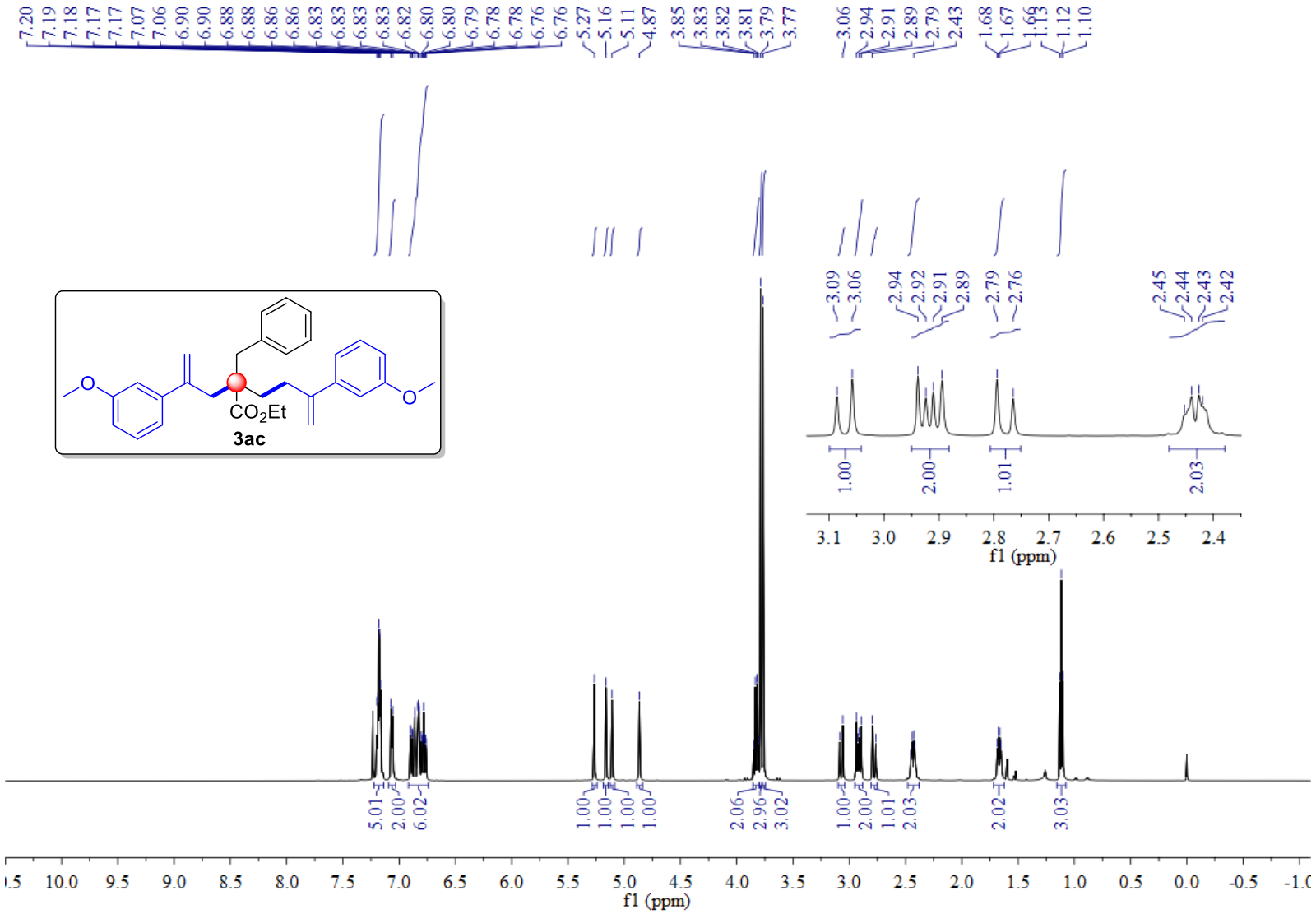
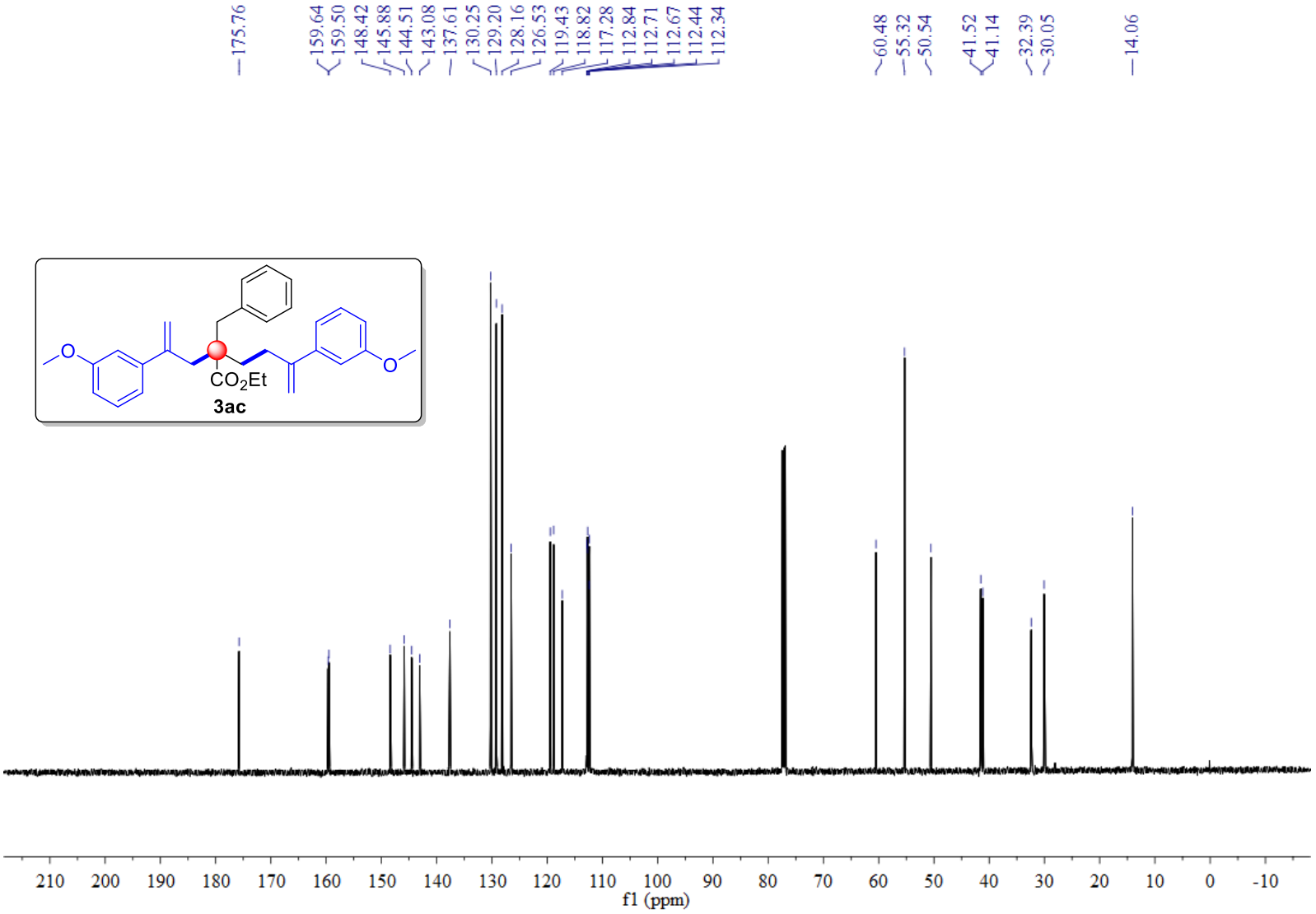
143.96
143.86
143.36
141.26
138.45
138.41
128.31
128.24
112.20
53.81
53.76
52.94
52.89
48.38
47.50
38.71
31.94
31.91
30.54
30.52
O=P(OMe)2
3ta
128.85
128.81
128.31
128.24
128.06
128.05
127.46
127.22
126.89
126.87
126.58
126.32
129.0
128.5
128.0
127.5
127.0
126.5
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


32.94
O
P(OMe)2
3ta
140
110
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)


7.24
7.23
7.23
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.17
7.09
7.09
7.08
7.07
6.82
6.81
6.80
6.79
6.78
5.19
5.19
5.10
5.10
5.04
4.81
4.80
3.82
3.81
3.78
3.12
3.09
2.94
2.91
2.90
2.87
2.78
1.67
1.66
1.64
1.14
1.12
1.11
CO2Et
3ab
2.44
2.43
2.41
2.40
2.08
2.45
2.40
f1 (ppm)
7.72
2.17
4.16
1.00
1.03
1.01
1.00
5.07
3.07
1.19
2.36
1.30
2.08
2.06
3.13
f1 (ppm)


175.91
159.17
159.13
147.80
145.35
137.78
130.31
128.20
127.97
127.33
126.53
116.08
113.66
113.63
110.78
60.49
55.43
55.42
50.54
41.79
41.16
32.52
30.04
14.12
O
CO2Et
O
3ab
137.78
135.49
133.84
130.31
138
136
134
132
130
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.20 7.19 7.18 7.17 7.17 7.07 7.06 6.90 6.90 6.88 6.88 6.86 6.86 6.83 6.83 6.83 6.83 6.82 6.80 6.80 6.79 6.78 6.78 6.76 6.76
5.27 5.16 5.11 4.87
3.85 3.83 3.82 3.81 3.79 3.77
3.06 2.94 2.91 2.89 2.79 2.43
1.68 1.67 1.66 1.13 1.12 1.10
3.09 3.06 2.94 2.92 2.91 2.89 2.79 2.76 2.45 2.44 2.43 2.42
1.00 2.00 1.01 2.03
3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4
f1 (ppm)
O
O
CO2Et
3ac
5.01 2.00 6.02 1.00 1.00 1.00 1.00 2.06 2.96 3.02 1.00 2.00 1.01 2.03 2.02 3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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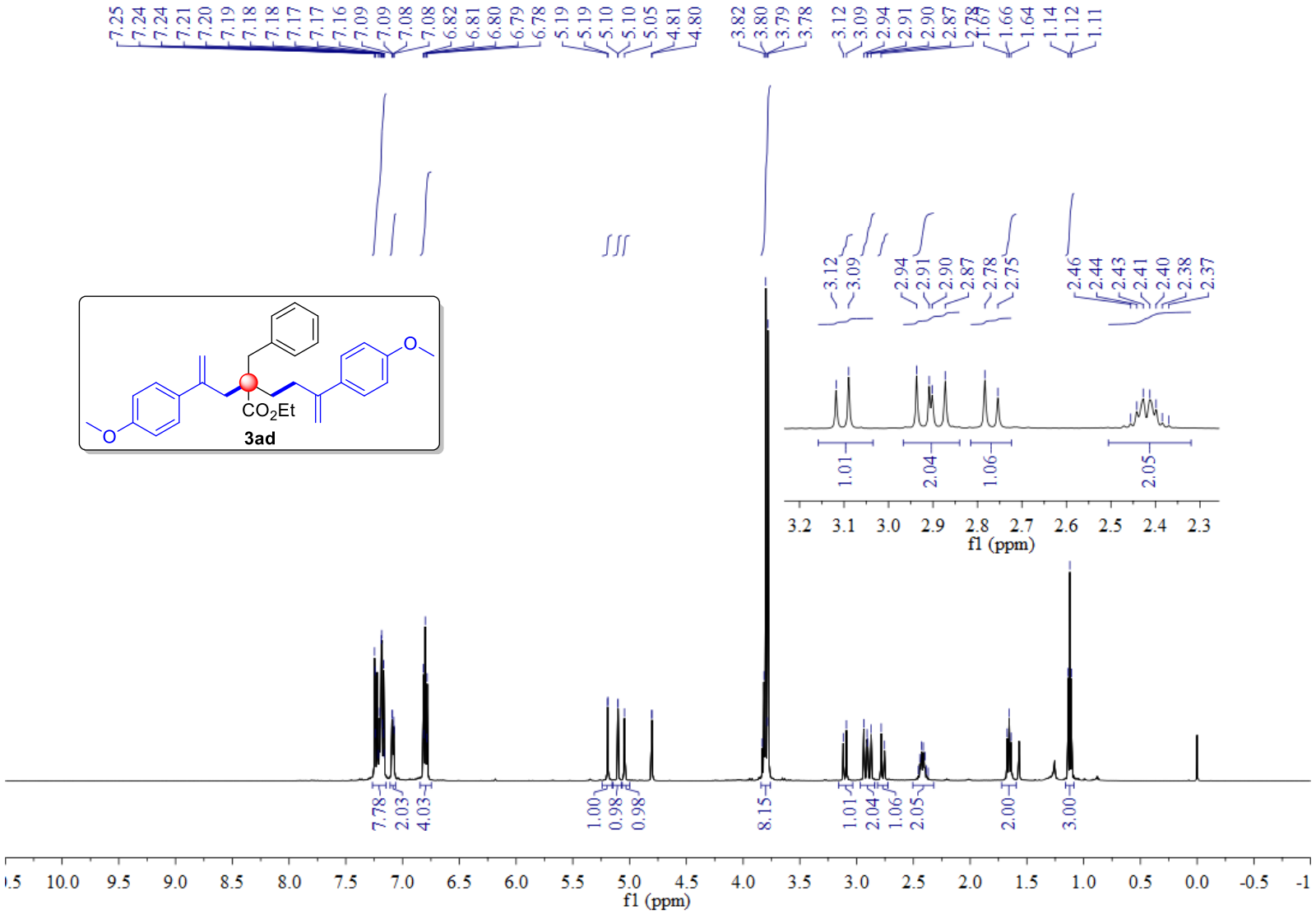
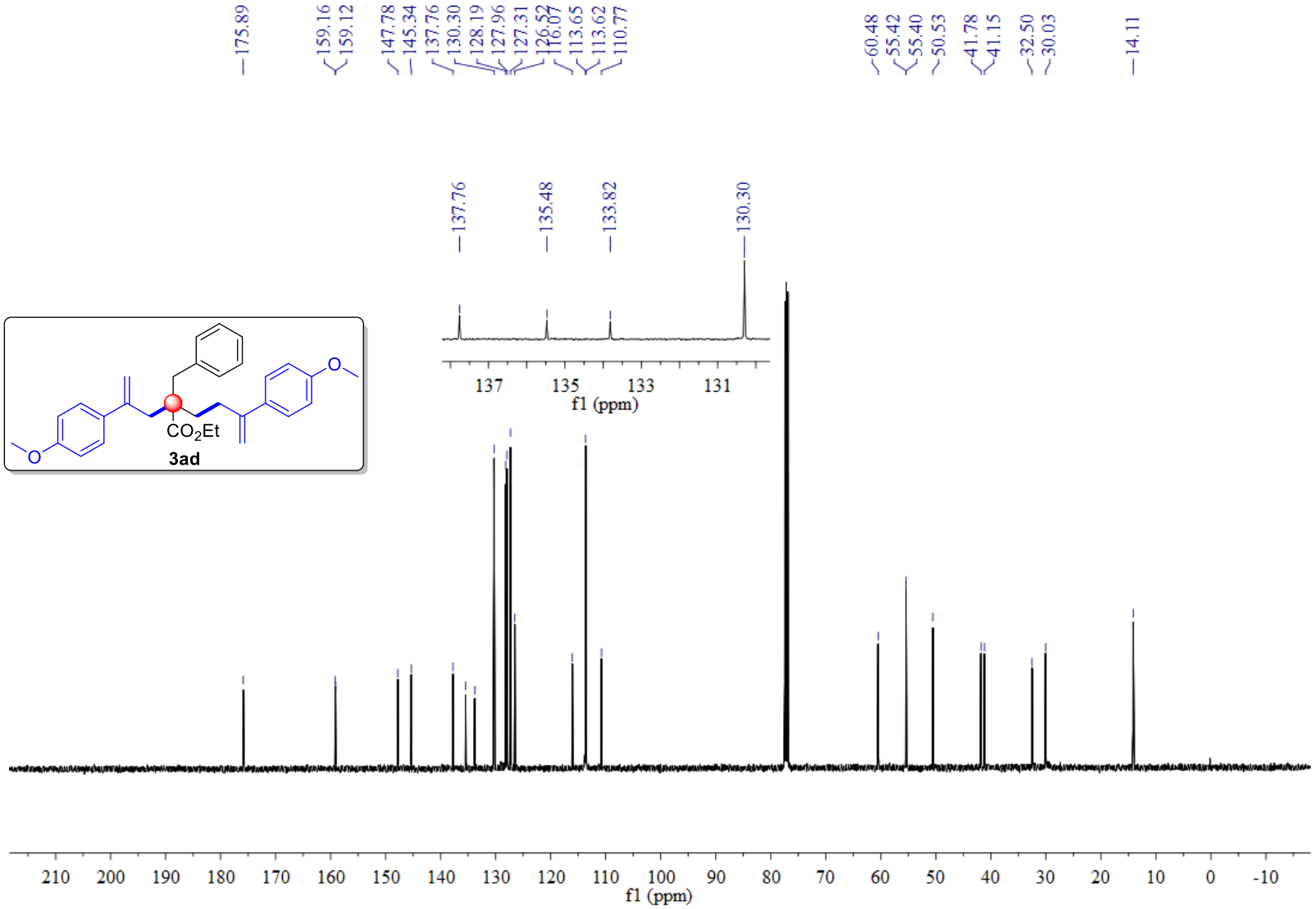
159.64
159.50
148.42
145.88
144.51
143.08
137.61
130.25
129.20
128.16
126.53
119.43
118.82
117.28
112.84
112.71
112.67
112.44
112.34
60.48
55.32
50.54
41.52
41.14
32.39
30.05
14.06
O
O
CO2Et
3ac
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


CO2Et
3ad
f1 (ppm)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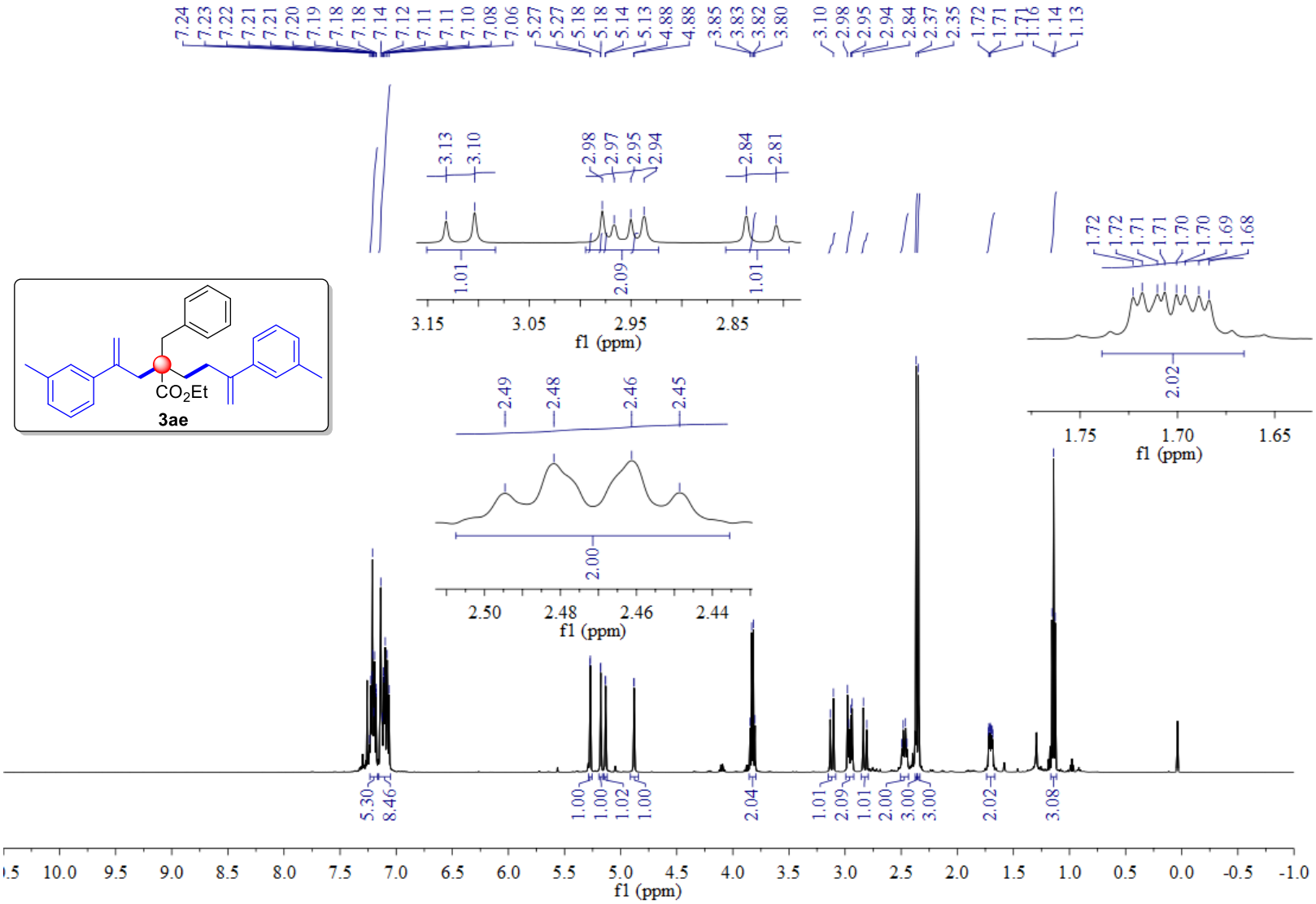
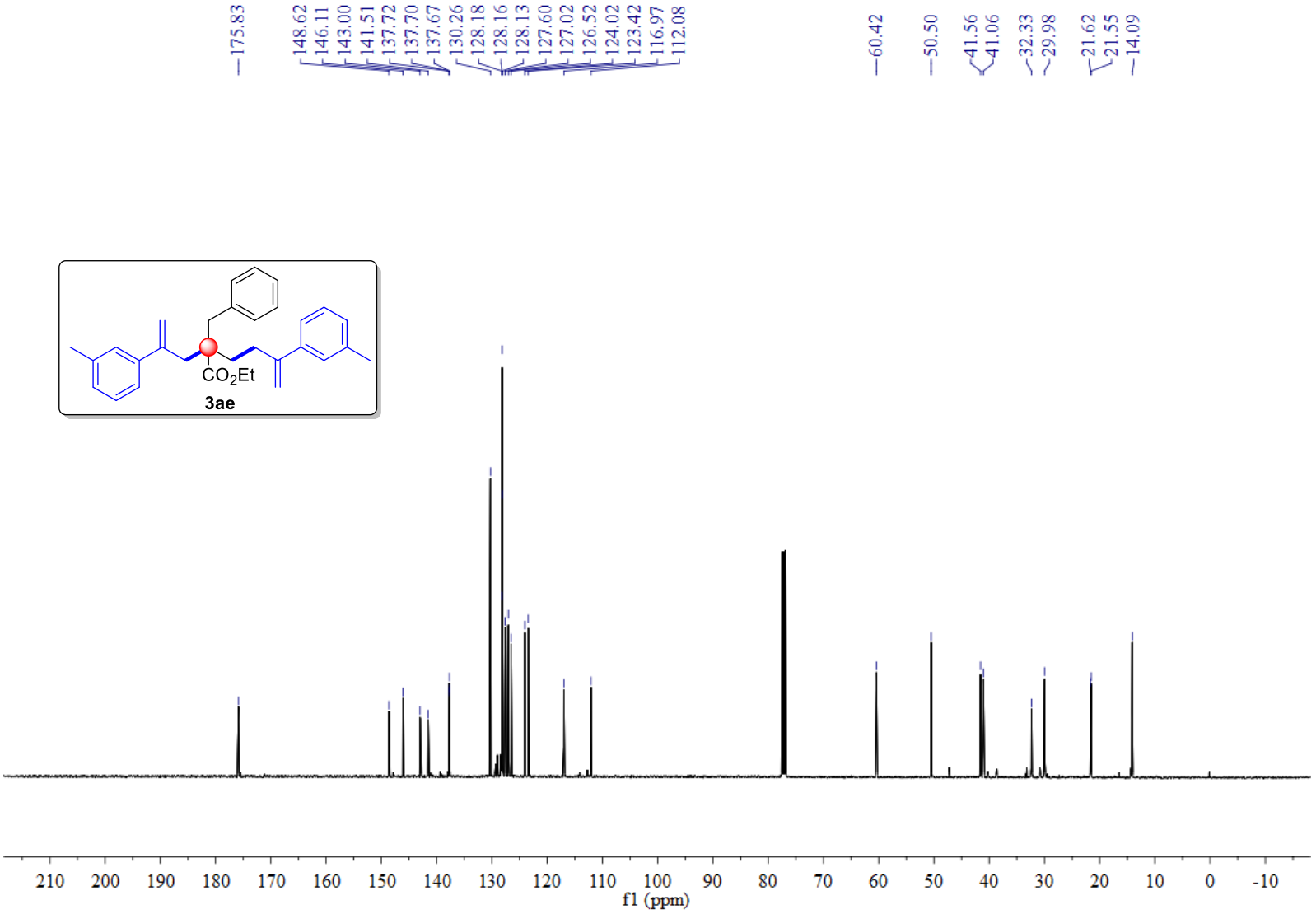
159.16
159.12
147.78
145.34
137.76
130.30
128.19
127.96
127.31
126.52
116.07
113.65
113.62
110.77
60.48
55.42
55.40
50.53
41.78
41.15
32.50
30.03
14.11
137.76
135.48
133.82
130.30
137
135
133
131
f1 (ppm)
CO2Et
3ad
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


CO2Et
3ae
f1 (ppm)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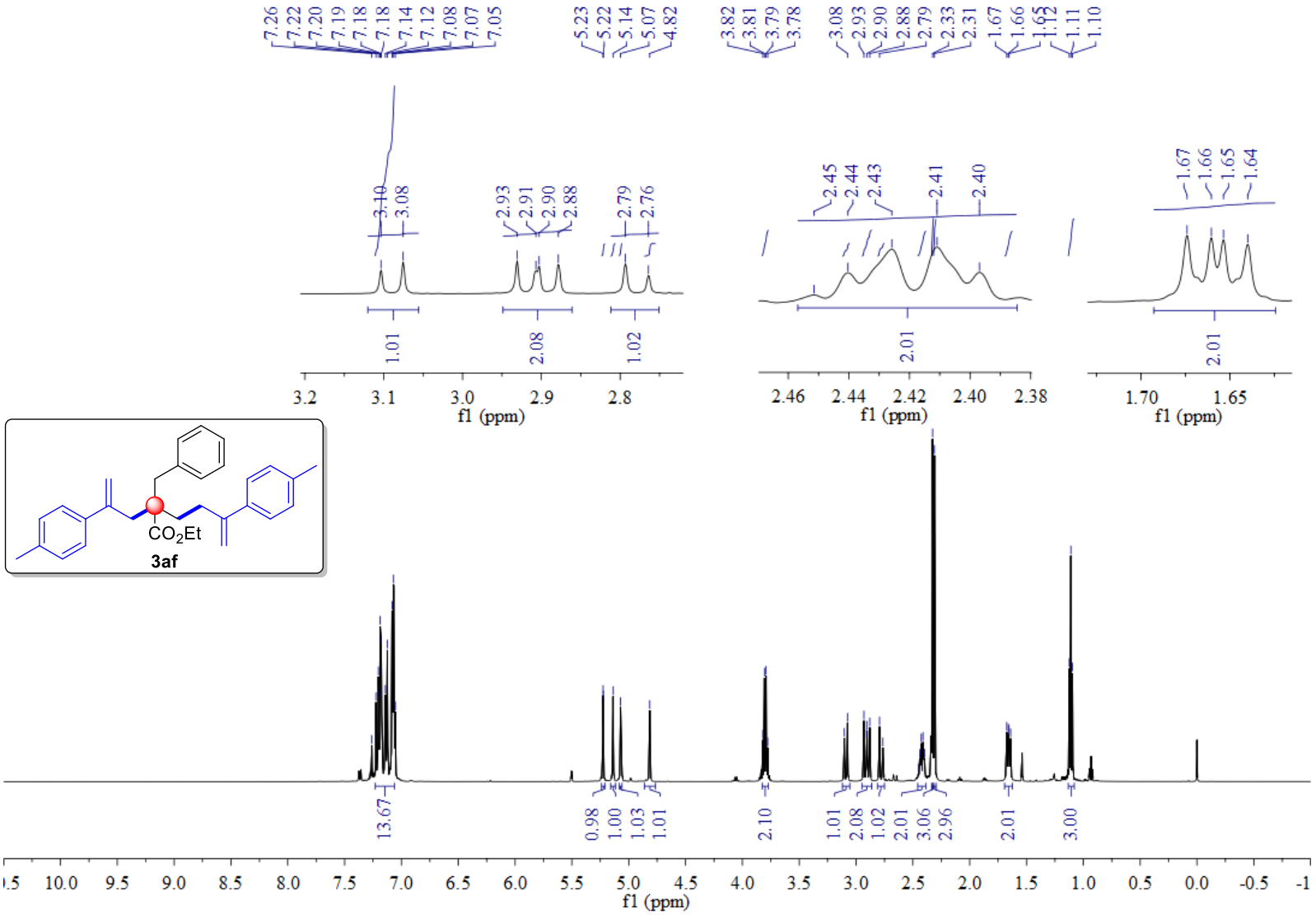
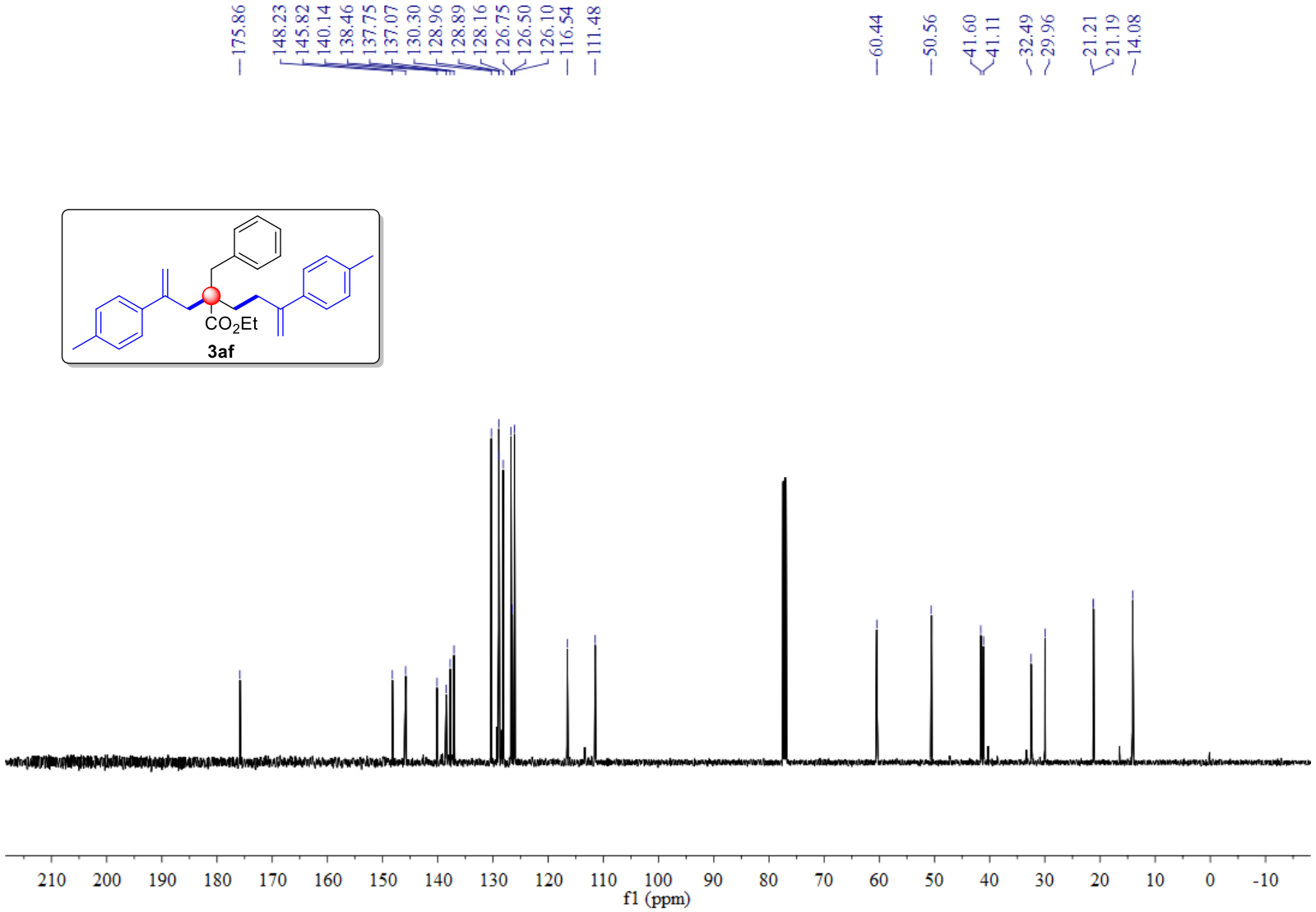
146.11
143.00
141.51
137.72
137.70
137.67
130.26
128.18
128.16
128.13
127.60
127.02
126.52
124.02
123.42
116.97
112.08
60.42
50.50
41.56
41.06
32.33
29.98
21.62
21.55
14.09
CO2Et
3ae
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.26
7.22
7.20
7.19
7.18
7.18
7.14
7.12
7.08
7.07
7.05
5.23
5.22
5.14
5.07
4.82
3.82
3.81
3.79
3.78
3.08
2.93
2.90
2.88
2.79
2.33
2.31
1.67
1.66
1.65
1.12
1.11
1.10
3.10
3.08
2.93
2.91
2.90
2.88
2.79
2.76
2.45
2.44
2.43
2.41
2.40
1.67
1.66
1.65
1.64
1.01
2.08
1.02
2.01
2.01
3.2
3.1
3.0
2.9
2.8
2.46
2.44
2.42
2.40
2.38
1.70
1.65
f1 (ppm)
CO2Et
3af
13.67
0.98
1.00
1.03
1.01
2.10
1.01
2.08
1.02
2.01
3.06
2.96
2.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)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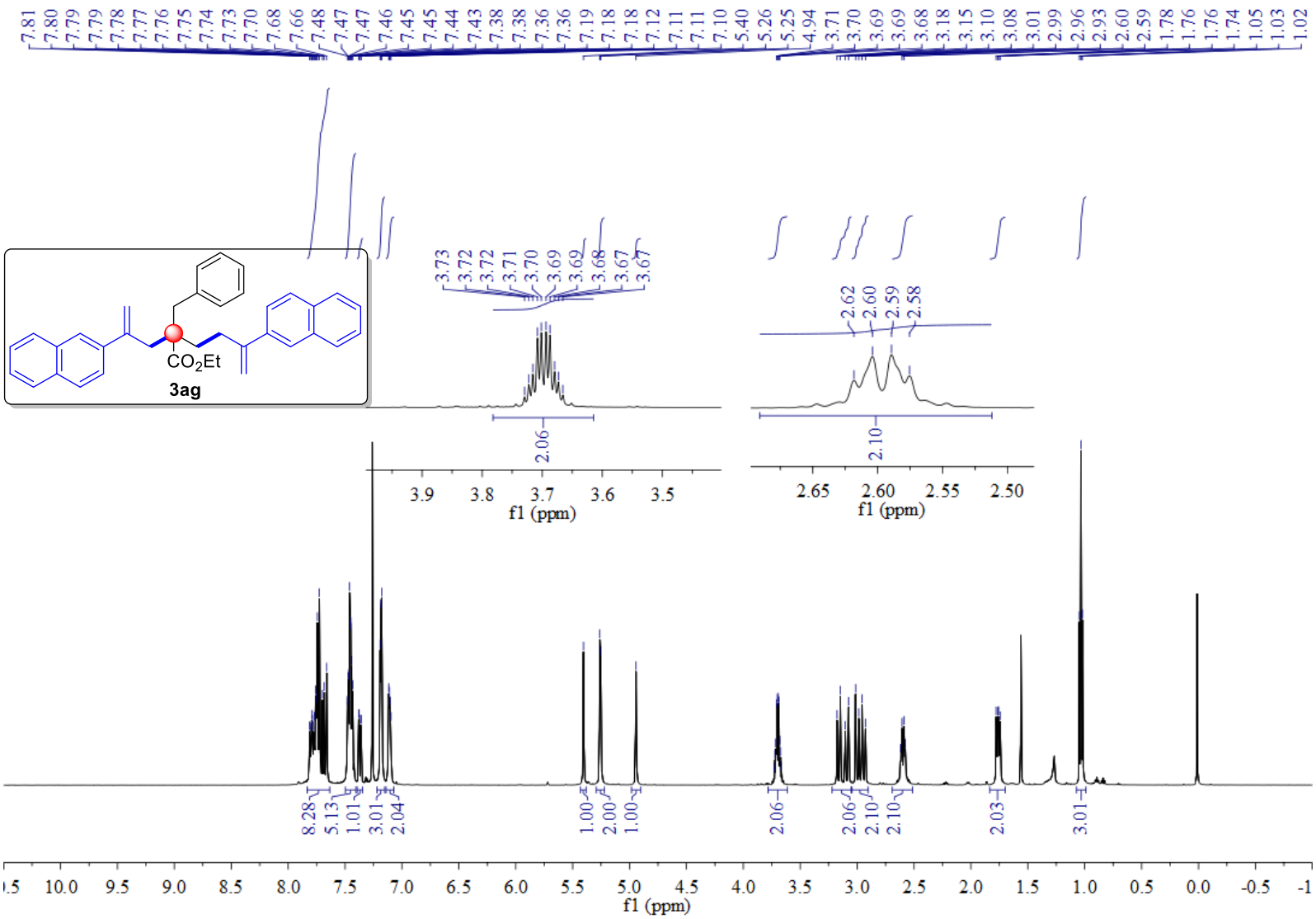
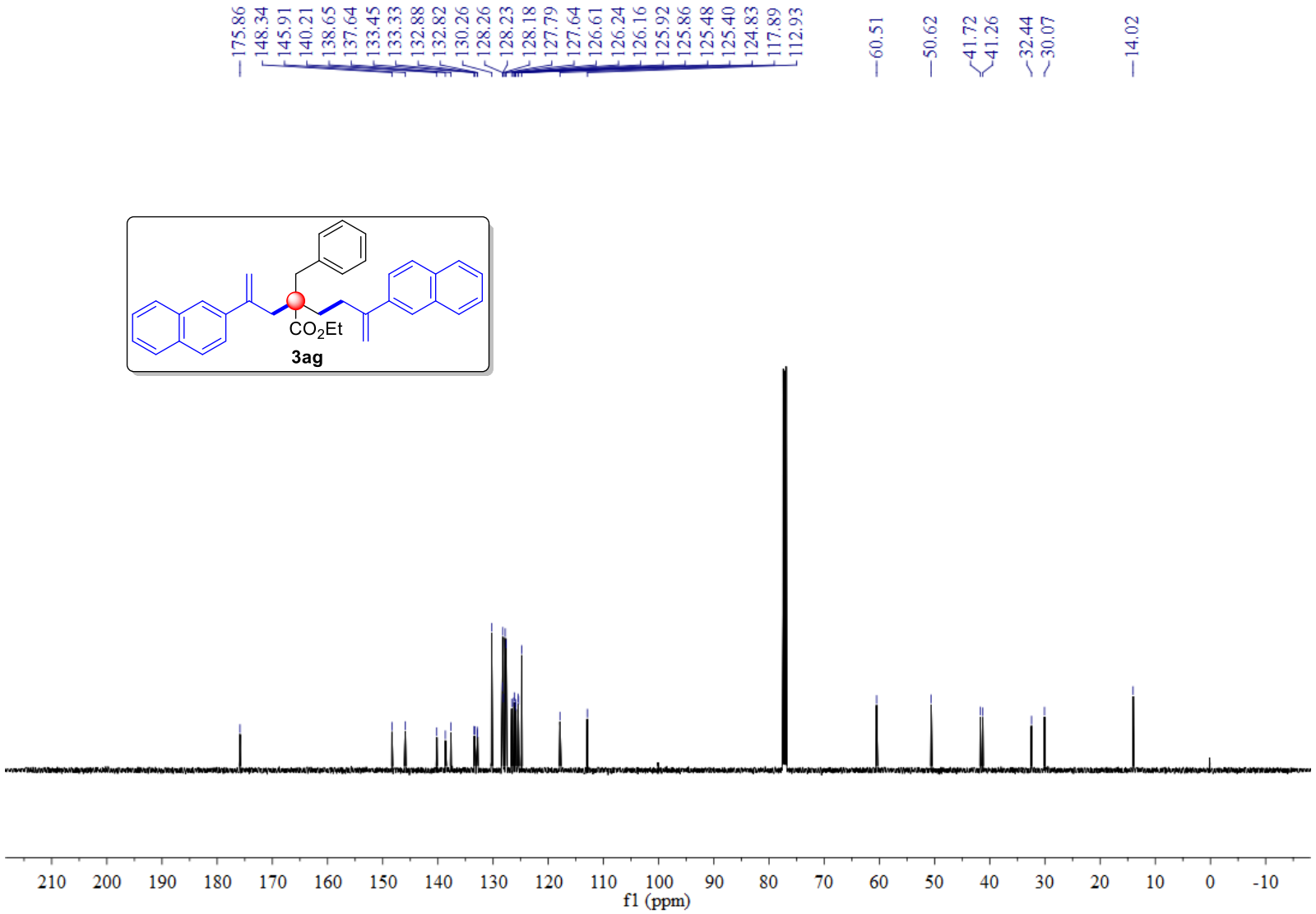
145.82
140.14
138.46
137.75
137.07
130.30
128.96
128.89
128.16
126.75
126.50
126.10
116.54
111.48
60.44
50.56
41.60
41.11
32.49
29.96
21.21
21.19
14.08
CO2Et
3af
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


CO2Et
3ag
f1 (ppm)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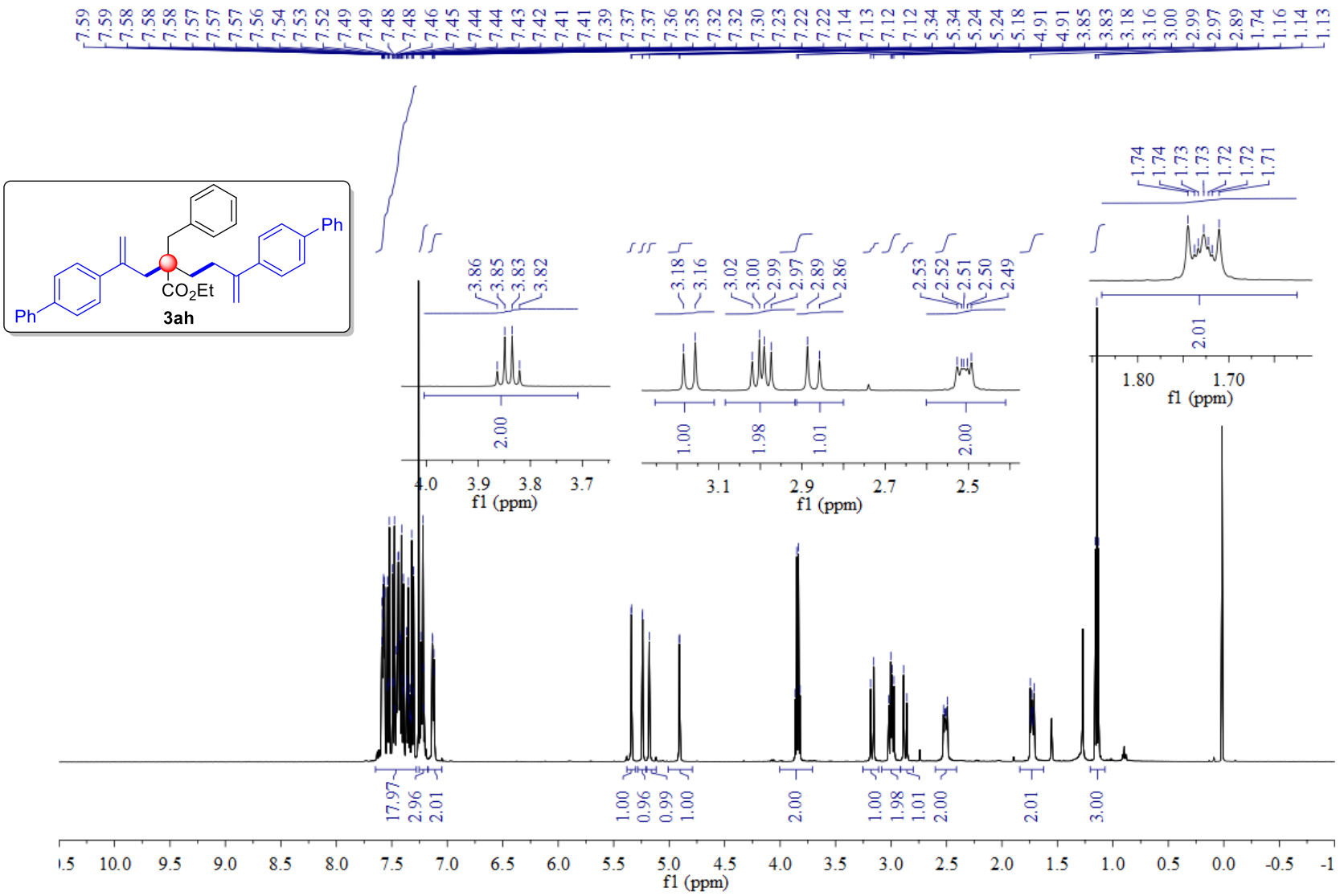
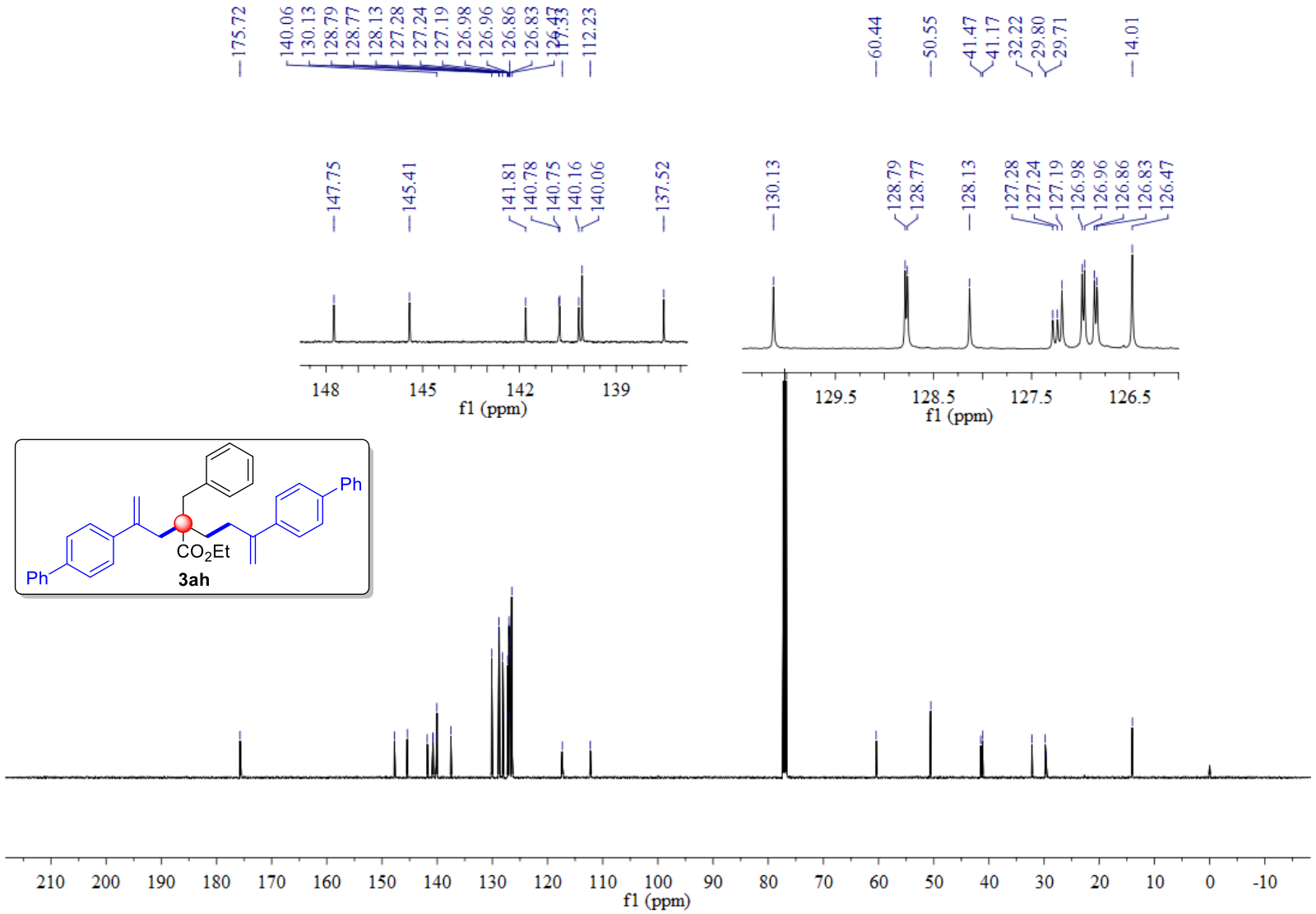
148.34
145.91
140.21
138.65
137.64
133.45
133.33
132.88
132.82
130.26
128.26
128.23
128.18
127.79
127.64
126.61
126.24
126.16
125.92
125.86
125.48
125.40
124.83
117.89
112.93
60.51
50.62
41.72
41.26
32.44
30.07
14.02
CO2Et
3ag
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Ph
CO2Et
Ph
3ah
f1 (ppm)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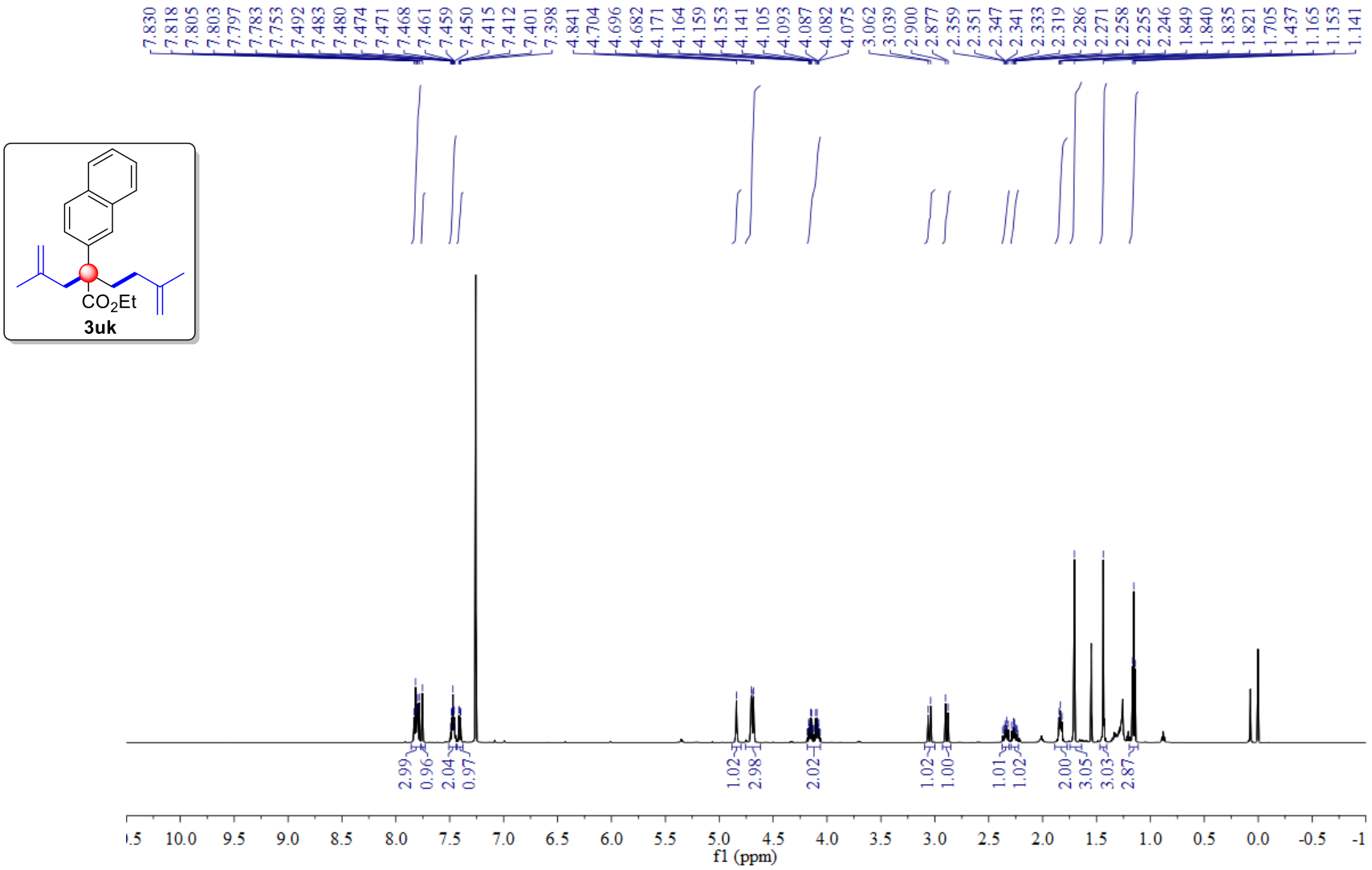
128.13
127.28
127.24
127.19
126.98
126.96
126.86
126.83
126.47
112.23
60.44
50.55
41.47
41.17
32.22
29.80
29.71
14.01
147.75
145.41
141.81
140.78
140.75
140.16
140.06
137.52
130.13
128.79
128.77
128.13
127.28
127.24
127.19
126.98
126.96
126.86
126.83
126.47
148
145
142
139
f1 (ppm)
129.5
128.5
127.5
126.5
f1 (ppm)
Ph
CO2Et
Ph
3ah
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.783
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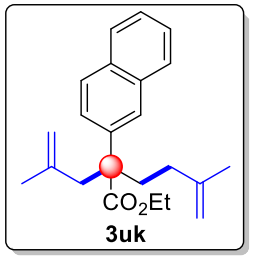
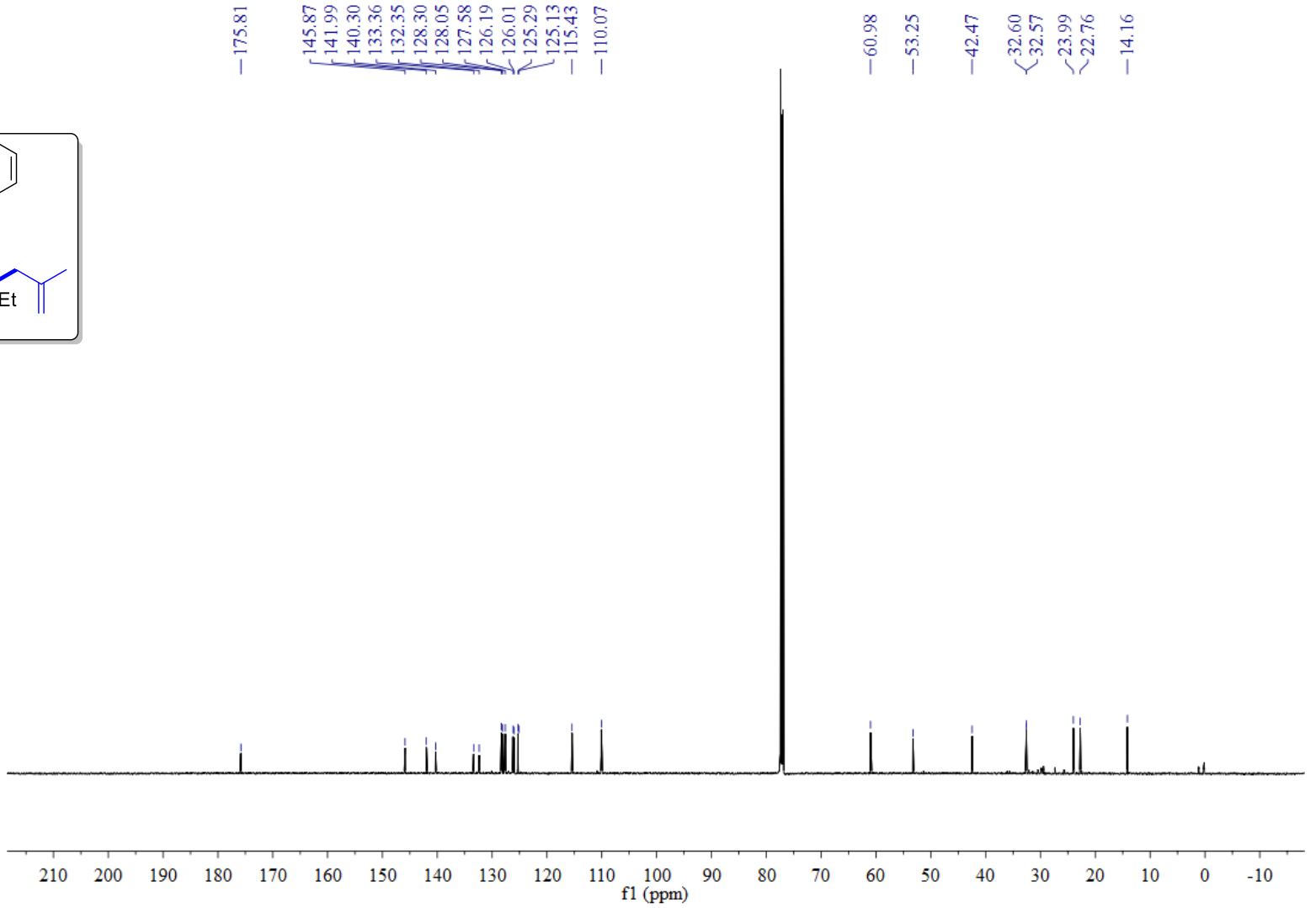
7.492
7.483
7.480
7.474
7.471
7.468
7.461
7.459
7.450
7.415
7.412
7.401
7.398
4.841
4.704
4.696
4.682
4.171
4.164
4.159
4.153
4.141
4.105
4.093
4.087
4.082
4.075
3.062
3.039
2.900
2.877
2.359
2.351
2.347
2.341
2.333
2.319
2.286
2.271
2.258
2.255
2.246
1.849
1.840
1.835
1.821
1.705
1.437
1.165
1.153
1.141
CO2Et
3uk
2.99
0.96
2.04
0.97
1.02
2.98
2.02
1.02
1.00
1.01
1.02
2.00
3.05
3.03
2.87
f1 (ppm)


CO2Et
3uk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140.30
133.36
132.35
128.30
128.05
127.58
126.19
126.01
125.29
125.13
115.43
110.07
60.98
53.25
42.47
32.60
32.57
23.99
22.76
14.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)