Ligand-Enabled Palladium-Catalyzed Hydroesterification of Vinyl Arenes with High Linear Selectivity to Access 3-Arylpropanoate Esters

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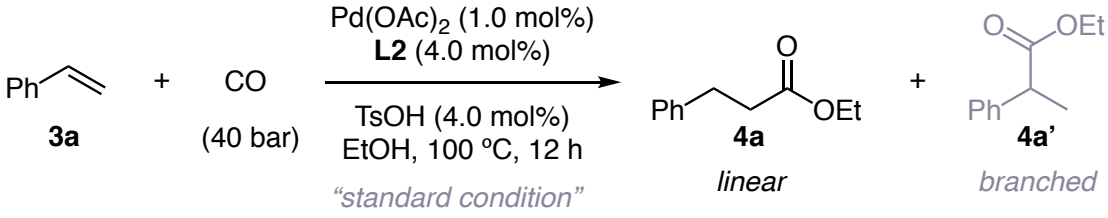
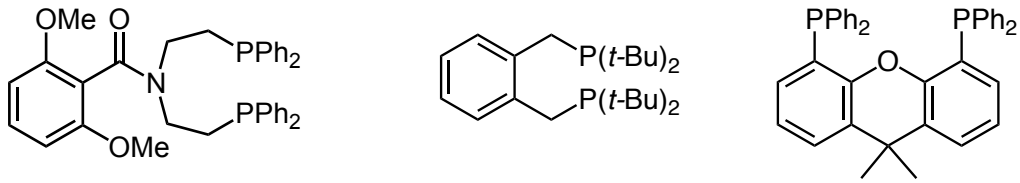
1. Supporting Tables and Schemes.

Table S1. Hydroesterification with different ligands under optimized conditions.



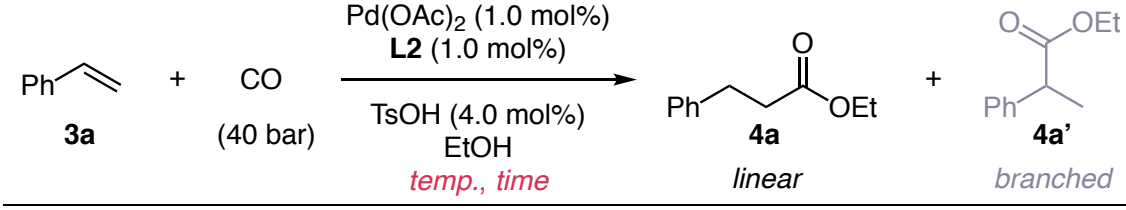


L2 dtbpx (**L15**) xantphos (**L16**)

Entry	Variation from standard conditions	Conv. (%)	l/b ratio
1	none	100	90/10
2	**L2** (1.0 mol%)	100	86/14
3	S/C = 300 (0.33 mol% [Pd]), 20 h	98	90/10
4	**L2** stored on benchtops for 3 months	100	88/12
5	**L15** (dtbpx)	100	82/18
6	**L16** (xantphos)	100	74/26

a. Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 ml). Conversions and *l/b* ratios were determined with GC-MS analysis

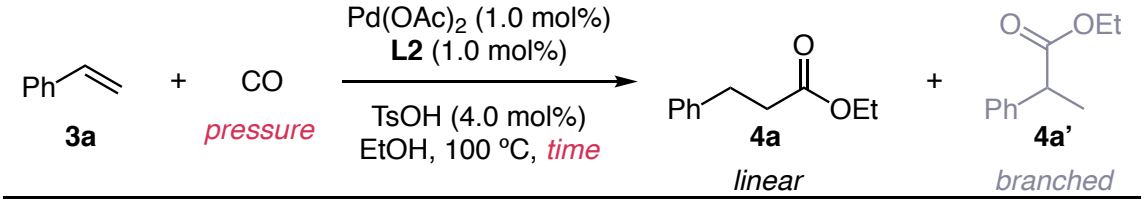
Table S2. Effect of the reaction temperature. [a]



entry	*temp* (°C)	*time* (h)	conv. (%)	*l/b* ratio
1	60	40	64	83/17
2	80	24	95	86/14
3	100	20	98	88/12
4	120	20	97	88/12

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l/b* ratios were determined with and GC-MS.

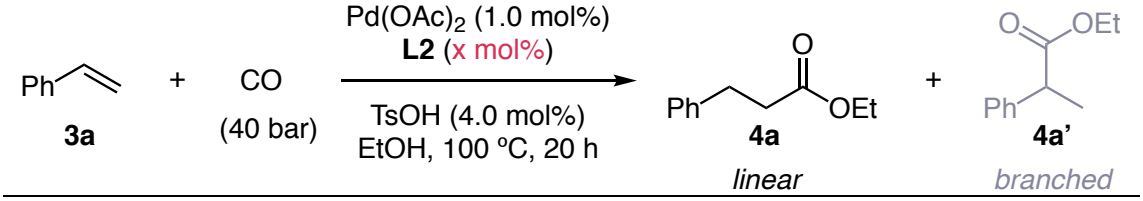
Table S3. Effect of the reaction pressure. [a]



entry	*pressure* (bar)	*time* (h)	Conv. (%)	*l*/*b* ratio
1	10	48	100	87/13
2	20	48	100	88/12
3	40	20	98	88/12
4	50	20	95	88/12

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l*/*b* ratios were determined with and GC-MS.

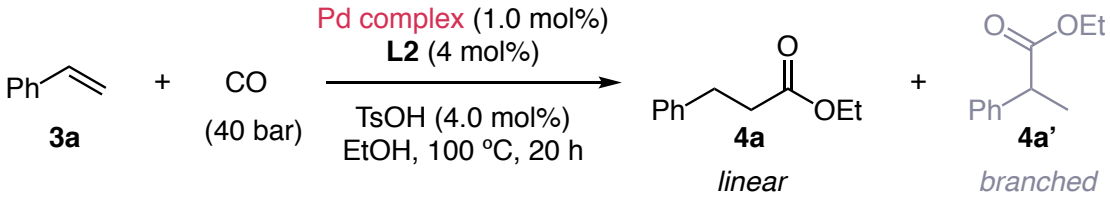
Table S4. Effect of the [Pd]/L ratio. [a]



entry	[Pd]/L	Conv. (%)	*l*/*b* ratio
1	1:1 (x = 1)	98	88/12
2	1:2 (x = 2)	100	87/13
3	1:4 (x = 4)	100	90/10
4	1:6 (x = 6)	100	89/11

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l*/*b* ratios were determined with and GC-MS.

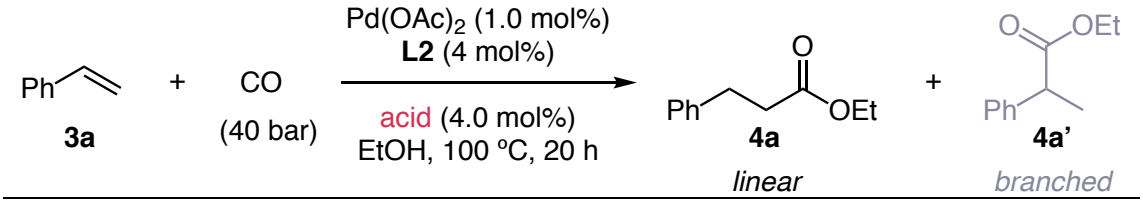
Table S5. Effect of the palladium precursor. [a]



Entry	Pd catalyst	conv.	*l*/*b* ratio
1	$Pd(OAc)_2$	100%	90/10
2	$PdCl_2$	83%	89/11
3	$Pd(PhCN)_2Cl_2$	50%	86/14
4	$Pd(PPh_3)_4$	100%	84/16
5	$Pd(acac)_2$	100%	87/13
6	$Pd(dppf)Cl_2$	100%	84/16
7	$Pd(dppf)Cl_2{\cdot}CH_2Cl_2$	100%	82/18
8	$[PdCl(C_3H_5)]_2$	50%	78/22
9	$Pd(PPh_3)Cl_2$	52%	89/11

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l*/*b* ratios were determined with and GC-MS.

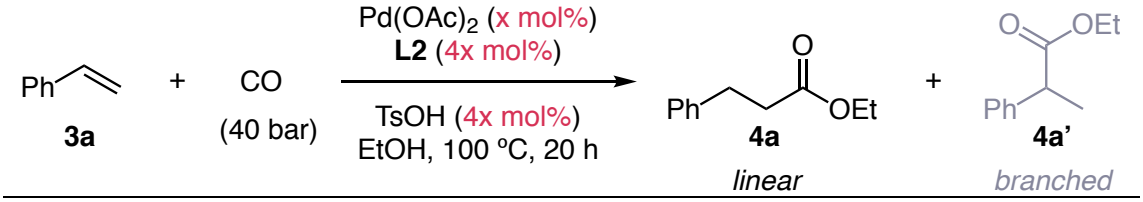
Table S6. Effect of the acid.[a]



entry	acid	Conv. (%)	*l*/*b* ratio
1	-	-	-
2	TsOH	100	90/10
3	MsOH	100	85/15
4	TfOH	100	90/10

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l*/*b* ratios were determined with and GC-MS.

Table S7. Effect of catalyst loading.[a]

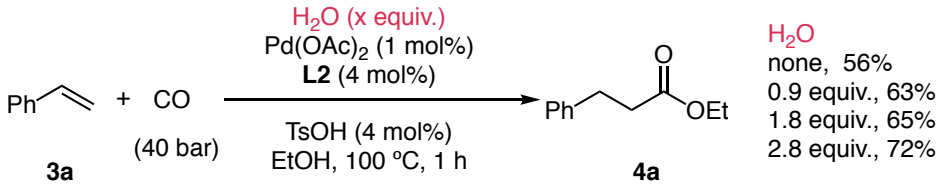


entry	S/C	Conv. (%)	*l*/*b* ratio
1	100 (x = 1)	100	90/10
2	300 (x = 0.33)	98	90/10
3	500 (x = 0.2)	10	88/12
4	1000 (x = 0.1)	6	88/12

[a]Reactions were performed with **3a** (0.6 mmol) in EtOH (2.0 mL). The convertions and *l*/*b* ratios were determined with and GC-MS.

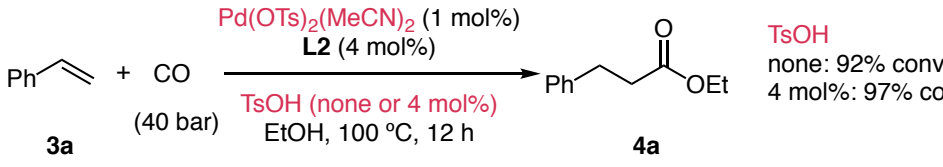
Scheme S1. Control experiments.

A. Effect of water



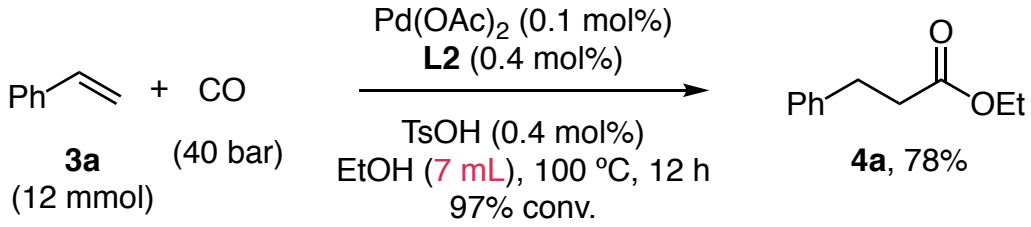
H_2O
none, 56%
0.9 equiv., 63%
1.8 equiv., 65%
2.8 equiv., 72%

B. Reaction with $Pd(OTs)_2(MeCN)_2$

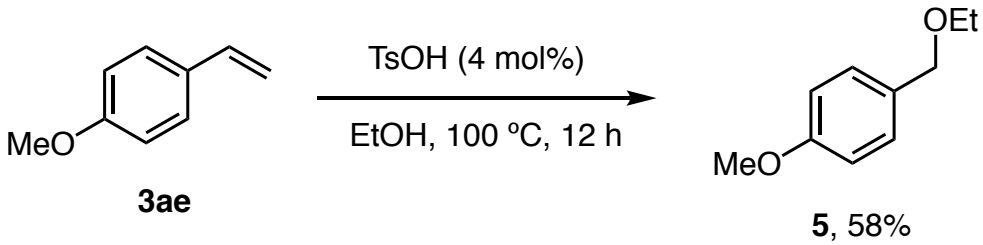


TsOH
none: 92% conv., *l*/*b* = 86/14
4 mol%: 97% conv., *l*/*b* = 87/13

Scheme S2. Gram-scale conversion with high S/C ratio.[a]



Scheme S3. Generation of Ether 5.[a]



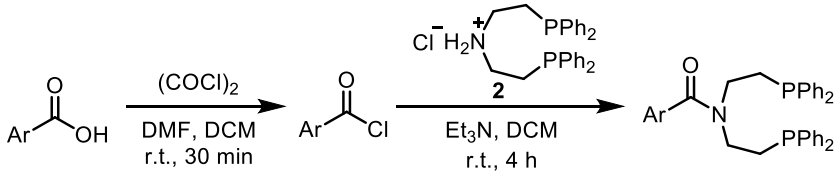
a. Yields of **5** was determined by GC-MS analysis.

2. General information: Materials and methods

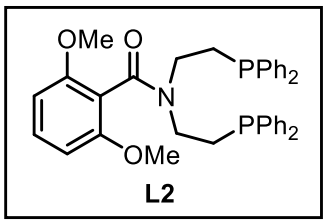
All commercial reagents and solvents were ordered from Bidepharm, TCI and Macklin. Reagents and solvents were used as received unless otherwise noted. Where necessary, solvents were purified by passing through columns of alumina using a solvent purification system. Air- and moisture sensitive synthesis were performed under nitrogen atmosphere with oven-dried glassware. Column chromatography was performed on silica gel (200-300 mesh). Thin-layer chromatography (TLC) was performed on EM reagents 0.25 mm silica 60-F plates. NMR spectra were recorded with a Bruker Avance III (400 MHz) spectrometer. ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz. Chemical shifts are reported in ppm downfield from $CDCl_3$ (δ = 7.26 ppm) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.16 ppm) for ^{13}C NMR spectroscopy. Data are reported as follows: chemical shift [multiplicity (br = broad, s = singlet, d = doublet, t = triplet, m = multiplet), coupling constant(s) in Hertz, integration]. GC-MS analysis was carried out on Agilent 7820A GC system and Angilent 5977B MSD. LC-MS analysis was carried out on Agilent 1260 Infinity II and Agilent 6545 LC/Q-TOF. High-resolution mass spectra (HRMS) were recorded on a Bruker microTOF Q III spectrometer with electronspray ionization (ESI). Melting points (m.p.) were recorded on an SRS–optic melting point apparatus.

3. Synthesis ligands.

3.1 General produce for the synthesis of L1-L4, L6-L14



***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2,6-dimethoxybenzamide (L2)**



A 25 mL flask was charged with 2,6-dimethoxybenzoic acid (546.5 mg, 3.0 mmol) in dichloromethane (6.0 mL), oxalyl chloride (761.6 mg, 0.5 mL, 6.0 mmol) was slowly added via syringe under nitrogen. DMF (1-2 drops) was added and the solution was stirred for 30 min at room temperature. The mixture was concentrated *in vacuo* to afford corresponding acyl chloride as a colorless oil. The product was used in the next step without further purification.

To a solution of triethylamine (1.21 g, 1.7 mL, 12.0 mmol) and 2,6-dimethoxybenzoyl chloride in dichloromethane (10.0 mL) was added **2** (1.15 g, 2.4 mmol) under nitrogen at 0 °C. After stirring at 0 ºC for 10 min, the reaction mixture was allowed to warm to room temperature and stirred for 4 h. After completion of the reaction (confirmed by TLC analysis), saturated aqueous NH_4Cl was added, and the aqueous layer was extracted with dichloromethane. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography (petroleum ether/ethyl acetate = 4:1) to afford **L2** as a white solid (843.1 mg, 58% yield).

M.p. 44.8-46.0 ºC.

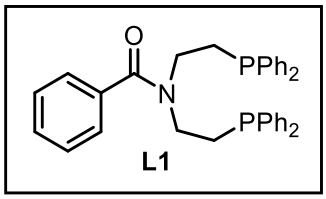
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 (td, *J* = 7.6, 1.6 Hz, 4H), 7.37-7.27 (m, 8H), 7.26-7.20 (m, 5H), 7.13 (td, *J* = 7.6, 1.6 Hz, 4H), 6.50 (d, *J* = 8.4 Hz, 2H), 3.71 (s, 6H), 3.64-3.55 (m, 2H), 3.25-3.14 (m, 2H), 2.50-2.39 (m, 2H), 2.16-2.06 (m, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.8, 156.5, 138.0 (d, *J* = 12.3 Hz), 137.5 (d, *J* = 12.1 Hz), 132.8 (d, *J* = 18.6 Hz), 132.3 (d, *J* = 18.6 Hz), 130.4, 128.8, 128.73, 128.68, 128.6, 115.0, 104.1, 55.9, 46.3 (d, *J* = 26.4 Hz), 42.9 (d, *J* = 24.6 Hz), 27.4 (d, *J* = 14.5 Hz), 26.3 (d, *J* = 13.9 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.12, -21.06.

HRMS(ESI): Calcd. for $C_{37}H_{38}NO_3P_2$: $[M+H]^+$ 606.2321; found: 606.2319.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)benzamide (L1)**



L1

L1 was prepared from benzoic acid (122.1 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Sticky colorless oil (257.5 mg, 59% yield).

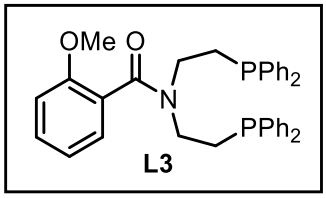
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63-7.45 (m, 5H), 7.44-7.27 (m, 14H), 7.25-7.12 (m, 6H), 3.72-3.52 (m, 2H), 3.47-3.24 (m, 2H), 2.56-2.39 (m, 2H), 2.25-2.08 (m, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.7, 137.9 (d, *J* = 10.7 Hz), 137.1 (d, *J* = 10.8 Hz), 136.6, 132.7 (d, *J* = 18.3 Hz), 132.4 (d, *J* = 18.8 Hz), 129.4, 129.0, 128.9, 128.72, 128.66 (d, *J* = 6.7 Hz), 128.6 (d, *J* = 8.7 Hz), 126.5, 47.0 (d, *J* = 21.4 Hz), 43.0 (d, *J* = 25.2 Hz), 27.8 (d, *J* = 17.1 Hz), 26.6 (d, *J* = 13.9 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.68, -21.52.

HRMS(ESI): Calcd. for $C_{35}H_{34}NOP_2$: $[M+H]^+$ 546.2110; found: 546.2112.

***N*,*N*-bis(2-(diphenylphosphaneyl)ethyl)-2-methoxybenzamide (L3)**



L3

L3 was prepared from 2-methoxybenzoic acid (304.3 mg, 2.0 mmol) and **2** (763.4 mg, 1.6 mmol) following the general procedure.

Pale yellow solid (315.0 mg, 34% yield).

M.p. 36.1-37.4 °C.

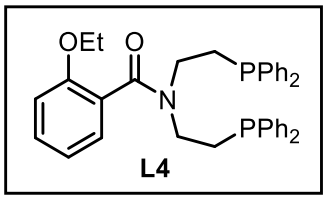
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (t, *J* = 7.2 Hz, 4H), 7.38-7.26 (m, 8H), 7.26-7.19 (m, 5H), 7.16-7.07 (m, 5H), 6.94 (td, *J* = 7.6, 0.8 Hz, 1H), 6.81 (dd, *J* = 8.4, 0.8 Hz, 1H), 3.70 (s, 3H), 3.65-3.55 (m, 2H), 3.27-3.16 (m, 2H), 2.52-2.38 (m, 2H), 2.17-2.05 (m, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 169.3, 155.1, 138.0 (d, *J* = 12.5 Hz), 137.0 (d, *J* = 10.3 Hz), 132.8 (d, *J* = 18.6 Hz), 132.4, 132.3 (d, *J* = 19.2 Hz), 130.3, 128.8, 128.7, 128.6, 127.7, 126.3, 121.0, 111.2, 55.5, 46.4 (d, *J* = 26.4 Hz), 42.8 (d, *J* = 24.4 Hz), 27.6 (d, *J* = 14.7 Hz), 26.5 (d, *J* = 14.0 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.49, -21.15.

HRMS(ESI): Calcd. for $C_{36}H_{36}NO_2P_2$: $[M+H]^+$ 576.2216; found: 576.2225.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2-ethoxybenzamide (L4)**



L4

L4 was prepared from 2-ethoxybenzoic acid (166.2 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Colorless oil (292.5 mg, 62% yield).

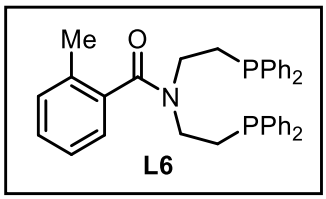
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (q, *J* = 8.0, 4H), 7.38-7.26 (m, 8H), 7.26-7.18 (m, 5H), 7.18-7.06 (m, 5H), 6.93 (td, *J* = 7.6, 0.8 Hz, 1H), 6.78 (d, *J* = 8.0 Hz, 1H), 4.01-3.83 (m, 2H), 3.80-3.69 (m, 1H), 3.50-3.38 (m, 1H), 3.28-3.18 (m, 2H), 2.52 (td, *J* = 12.0, 5.2 Hz, 1H), 2.36 (td, *J* = 12.0, 4.8 Hz, 1H), 2.22-2.00 (m, 2H), 1.25 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 169.3, 154.4, 138.1 (d, *J* = 11.9 Hz), 137.8 (d, *J* = 12.4 Hz), 137.6 (d, *J* = 11.8 Hz), 136.9 (d, *J* = 11.8 Hz), 132.8 (d, *J* = 18.6 Hz), 132.7 (d, *J* = 18.5 Hz), 132.4 (d, *J* = 18.9 Hz), 132.2 (d, *J* = 18.5 Hz), 130.2, 128.83, 128.78, 128.75 (d, *J* = 7.7 Hz), 128.74 (d, *J* = 7.7 Hz), 128.73 (d, *J* = 7.5 Hz), 128.72 (d, *J* = 7.5 Hz), 128.69, 128.6, 127.7, 126.5, 120.9, 112.0, 63.8, 46.2 (d, *J* = 26.1 Hz), 42.6 (d, *J* = 24.5 Hz), 27.6 (d, *J* = 14.8 Hz), 26.3 (d, *J* = 14.2 Hz), 14.8.

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.82, -21.29.

HRMS(ESI): Calcd. for $C_{37}H_{38}NO_2P_2$: $[M+H]^+$ 590.2372; found: 590.2388.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2-methoxybenzamide (L6)**



L6

L6 was prepared from 2-methylbenzoic acid (544.6 mg, 4.0 mmol) and **2** (1.53 g, 3.2 mmol) following the general procedure.

White solid (1.47 g, 82% yield).

M.p. 30.9-32.3 °C.

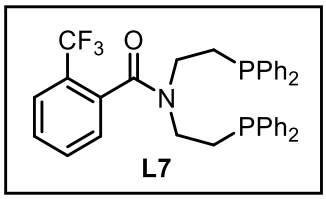
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (td, *J* = 7.6, 2.0 Hz, 4H), 7.39-7.26 (m, 9H), 7.26-7.20 (m, 4H), 7.18-7.07 (m, 7H), 3.85-3.42 (m, 2H), 3.28-3.14 (m, 2H), 2.45 (brs, 2H), 2.24 (s, 3H), 2.08 (t, *J* = 8.4, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.3, 137.7 (d, *J* = 13.7 Hz), 136.9 (d, *J* = 13.0 Hz), 136.5, 133.9, 132.7 (d, *J* = 18.6 Hz), 132.3 (d, *J* = 18.7 Hz), 130.5, 128.91, 128.89, 128.88, 128.68 (d, *J* = 6.7 Hz), 128.66 (d, *J* = 6.7 Hz), 126.0, 125.7, 46.2 (d, *J* = 26.5 Hz), 42.4 (d, *J* = 24.4 Hz), 27.5 (d, *J* = 15.3 Hz), 26.6 (d, *J* = 14.2 Hz), 19.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.95, -21.45.

HRMS(ESI): Calcd. for $C_{36}H_{36}NOP_2$: $[M+H]^+$ 560.2267; found: 560.2267.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2-(trifluoromethyl)benzamide (L7)**



L7

L7 was prepared from 2-(trifluoromethyl)benzoic acid (190.1 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Colorless oil (313.0 mg, 64% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63-7.58 (m, 1H), 7.54-7.42 (m, 6H), 7.39-7.32 (m, 6H), 7.30-7.26 (m, 2H), 7.25-7.09 (m, 9H), 3.84-3.73 (m, 1H), 3.49-3.36 (m, 1H), 3.24-3.02 (m, 2H), 2.57 (td, *J* = 12.0, 5.2 Hz, 1H), 2.30 (td, *J* = 12.4, 4.8 Hz, 1H), 2.13 (td, *J* = 12.4, 4.4 Hz, 1H), 2.01 (td, *J* = 12.4, 5.6 Hz, 1H).

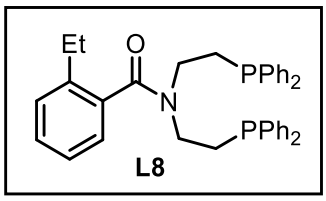
^{13}C NMR (100 MHz, Chloroform-*d*) δ 168.5, 137.9 (d, *J* = 12.3 Hz), 137.5 (d, *J* = 12.1 Hz), 136.9 (d, *J* = 11.8 Hz), 136.7 (d, *J* = 12.0 Hz), 135.0 (q, *J* = 2.1 Hz), 132.8 (d, *J* = 18.7 Hz), 132.7 (d, *J* = 18.7 Hz), 132.4 (d, *J* = 19.0 Hz), 132.3 (d, *J* = 18.6 Hz), 132.09, 132.08, 129.1, 129.01, 128.95, 128.92, 128.70 (d, *J* = 6.7 Hz), 128.66 (d, *J* = 6.9 Hz), 127.2, 126.71 (q, *J* = 4.6 Hz), 126.6 (q, *J* = 31.6 Hz), 123.1 (q, *J* = 272.4 Hz), 46.2 (d, *J* = 27.4 Hz), 42.23 (d, *J* = 25.2 Hz), 27.0 (d, *J* = 15.2 Hz), 25.6 (d, *J* = 14.2 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.80, -21.62.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -59.61.

HRMS(ESI): Calcd. for $C_{36}H_{33}F_3NOP_2$: $[M+H]^+$ 614.1984; found: 614.1976.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2-ethylbenzamide (L8)**



L8

L8 was prepared from 2-ethylbenzoic acid (150.2 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Colorless oil (349.0 mg, 76% yield).

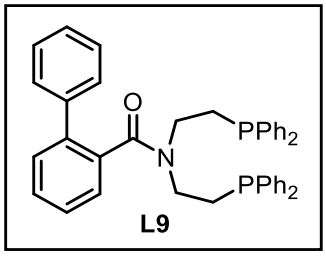
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.43 (m, 4H), 7.39-7.26 (m, 9H), 7.22 (t, *J* = 7.2 Hz, 5H), 7.18-7.05 (m, 6H), 3.82-3.44 (m, 2H), 3.28-3.10 (m, 2H), 2.70-2.29 (m, 4H), 2.07 (t, *J* = 8.4 Hz, 2H), 1.17 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.3, 140.1, 137.8 (d, *J* = 12.8 Hz), 137.2 (d, *J* = 14.4 Hz), 136.0, 132.8 (d, *J* = 18.8 Hz), 132.3 (d, *J* = 18.6 Hz), 129.1, 128.91, 128.87, 128.8, 128.69 (d, *J* = 6.5 Hz), 128.66 (d, *J* = 6.8 Hz), 126.0, 125.75, 46.44 (d, *J* = 26.9 Hz), 42.4 (d, *J* = 24.0 Hz), 27.5 (d, *J* = 15.4 Hz), 26.6 (d, *J* = 14.3 Hz), 26.0, 15.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.92, -21.32.

HRMS(ESI): Calcd. for $C_{37}H_{38}NOP_2$: $[M+H]^+$ 574.2423; found: 574.2424.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-[1,1'-biphenyl]-2-carboxamide (L9)**



L9

L9 was prepared from [1,1'-biphenyl]-2-carboxylic acid (597.7 mg, 3.0 mmol) and **2** (1.15 g, 2.4 mmol) following the general procedure.

White solid (1.14 g, 77% yield).

M.p. 43.7-45.0 °C.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46-7.26 (m, 18H), 7.26-7.18 (m, 7H), 7.10-6.98 (m, 4H), 3.88-3.76 (m, 1H), 3.00-2.81 (m, 2H), 2.73-2.61 (m, 1H), 2.20 (td, *J* = 12.4, 4.8 Hz, 1H), 1.83-1.63 (m, 3H).

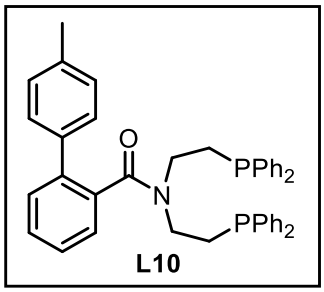
^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.8, 139.8, 138.4, 138.2 (d, *J* = 13.4 Hz), 137.8 (d, *J* = 12.2 Hz), 137.7 (d, *J* = 11.7 Hz), 136.4 (d, *J* = 11.6 Hz), 135.8, 132.73 (d, *J* = 19.4 Hz), 132.69 (d, *J* = 18.5 Hz), 132.66 (d, *J* = 18.7 Hz), 131.9 (d, *J* = 17.9 Hz), 129.6, 129.3, 129.1, 129.0, 128.84, 128.79 (d, *J* = 7.9 Hz), 128.72, 128.69, 128.65 (d, *J* = 6.7 Hz), 128.60 (d, *J* = 6.1 Hz), 128.54 (d, *J* = 6.0 Hz), 128.48, 127.8, 127.7, 127.3, 45.7 (d, *J* = 26.7 Hz), 42.1 (d, *J* = 25.4 Hz), 26.8 (d, *J* = 14.9 Hz), 25.4 (d, *J* = 14.0 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -21.06, -21.19.

HRMS(ESI): Calcd. for $C_{41}H_{38}NOP_2$: $[M+H]^+$ 622.2423; found: 622.2426.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-4'-methyl-[1,1'-biphenyl]-2-carboxamide (L10)**

L10 was prepared from 4'-methyl-[1,1'-biphenyl]-2-carboxylic acid (212.2 mg, 1.0 mmol) and **2** (381.7 g, 0.8 mmol) following the general procedure.



L10

White solid (151.0 mg, 30% yield).

M.p. 32.0-33.2 °C.

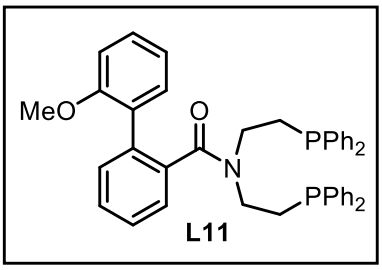
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55-7.27 (m, 16H), 7.26-7.15 (m, 6H), 7.11-6.99 (m, 6H), 3.94-3.77 (m, 1H), 3.02-2.80 (m, 2H), 2.76-2.63 (m, 1H), 2.24 (s, 3H), 1.86-1.67 (m, 3H), 1.64-1.53 (m, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.90, 138.33 (d, *J* = 12.4 Hz), 138.30, 137.9 (d, *J* = 11.6 Hz), 137.8 (d, *J* = 12.1 Hz), 137.5, 136.9, 136.5 (d, *J* = 12.5 Hz), 135.7, 132.7 (d, *J* = 19.5 Hz), 132.65 (d, *J* = 18.5 Hz), 132.61 (d, *J* = 18.6 Hz), 131.9 (d, *J* = 18.1 Hz), 129.5, 129.3, 129.2, 129.0, 128.9, 128.84, 128.80, 128.69, 128.68 (d, *J* = 7.2 Hz), 128.6 (d, *J* = 8.7 Hz), 128.54 (d, *J* = 8.4 Hz), 128.51 (d, *J* = 8.5 Hz), 127.5, 127.3, 45.6 (d, *J* = 26.5 Hz), 42.1 (d, *J* = 25.9 Hz), 26.8 (d, *J* = 15.0 Hz), 25.5 (d, *J* = 14.0 Hz), 21.2.

^{31}P NMR (162 MHz, Chloroform-*d*) δ -21.10.

HRMS(ESI): Calcd. for $C_{42}H_{40}NOP_2$: $[M+H]^+$ 636.2580; found: 636.2576.

_N,N_-bis(2-(diphenylphosphaneyl)ethyl)-2'-methoxy-[1,1'-biphenyl]-2-carboxamide (L11)



L11

L11 was prepared from 2'-methoxy-[1,1'-biphenyl]-2-carboxylic acid (228.3 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

White solid (324.0 mg, 62% yield).

M.p. 51.0-52.3 °C.

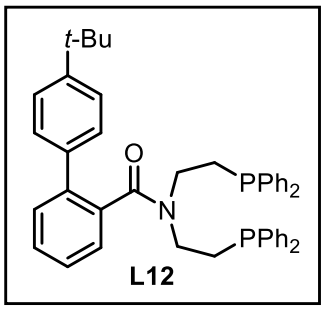
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46-7.27 (m, 16H), 7.25-7.08 (m, 8H), 7.00 (t, *J* = 7.2 Hz, 2H), 6.85 (td, *J* = 7.6, 1.2 Hz, 1H), 6.78 (d, *J* = 8.0 Hz, 1H), 3.94-3.78 (m, 1H), 3.40 (s, 3H), 3.34-3.21 (m, 1H), 2.83-2.63 (m, 2H), 2.06-1.75 (m, 3H), 1.55-1.43 (m, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.6, 156.4, 138.4 (d, *J* = 12.8 Hz), 138.0 (d, *J* = 12.8 Hz), 137.9 (d, *J* = 14.5 Hz), 137.7 (d, *J* = 12.0 Hz), 136.6, 135.3, 132.7 (d, *J* = 18.9 Hz), 132.6 (d, *J* = 18.7 Hz), 132.3 (d, *J* = 17.8 Hz), 132.2 (d, *J* = 18.4 Hz), 131.8, 131.3, 129.2, 128.71 (d, *J* = 7.9 Hz), 128.70 (d, *J* = 7.8 Hz), 128.69 (d, *J* = 7.6 Hz), 128.67 (d, *J* = 6.6 Hz), 128.63, 128.61, 128.59, 128.58, 128.34, 128.29, 127.6, 126.7, 120.4, 110.5, 55.0, 45.4 (d, *J* = 27.0 Hz), 41.7 (d, *J* = 25.8 Hz), 27.2 (d, *J* = 14.9 Hz), 25.3 (d, *J* = 13.8 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -21.14, -21.21.

HRMS(ESI): Calcd. for $C_{42}H_{40}NO_2P_2$: $[M+H]^+$ 652.2529; found: 652.2529.

4'-(*tert*-butyl)-*N*,*N*-bis(2-(diphenylphosphaneyl)ethyl)-[1,1'-biphenyl]-2-carboxamide (L12)



L12

L12 was prepared from 4'-(*tert*-butyl)-[1,1'-biphenyl]-2-carboxylic acid (254.3 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

White solid (352.0 mg , 65% yield).

M.p. 45.9-47.0 °C.

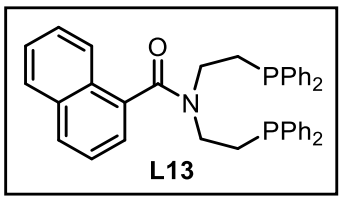
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51-7.26 (m, 18H), 7.26-7.18 (m, 6H), 7.09-6.98 (m, 4H), 3.91 (tt, *J* = 12.8, 4.8 Hz, 1H), 2.95-2.78 (m, 2H), 2.77-2.65 (m, 1H), 2.37 (td, *J* = 12.8, 4.8 Hz, 1H), 1.80-1.68 (m, 2H), 1.57-1.46 (m, 1H), 1.19 (s, 9H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.0, 150.8, 138.2, 138.1 (d, *J* = 12.3 Hz), 138.0 (d, *J* = 12.0 Hz), 137.6 (d, *J* = 12.1 Hz), 136.7, 136.3 (d, *J* = 12.1 Hz), 135.7, 132.8 (d, *J* = 19.6 Hz), 132.7 (d, *J* = 18.9 Hz), 132.6 (d, *J* = 18.1 Hz), 131.9 (d, *J* = 17.8 Hz), 129.6, 129.3, 129.1, 128.8 (d, *J* = 8.6 Hz), 128.73, 128.71 (d, *J* = 7.2 Hz), 128.70, 128.66 (d, *J* = 6.7 Hz), 128.59 (d, *J* = 6.9 Hz), 128.57, 128.5, 127.6, 127.4, 125.3, 45.7 (d, *J* = 26.9 Hz), 42.3 (d, *J* = 26.8 Hz), 34.5, 31.4, 26.5 (d, *J* = 14.9 Hz), 25.5 (d, *J* = 14.1 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.76, -21.13.

HRMS(ESI): Calcd. for $C_{45}H_{46}NOP_2$: $[M+H]^+$ 678.3049; found: 678.3059.

***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-1-naphthamide (L13)**



L13

L13 was prepared from 1-naphthoic acid (172.2 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Pale yellow solid (299.0 mg, 63% yield).

M.p. 46.7-47.9 ℃.

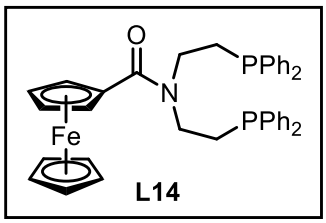
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91-7.78 (m, 3H), 7.59-7.46 (m, 6H), 7.42-7.32 (m, 7H), 7.29 (dd, *J* = 6.8, 1.2 Hz, 1H), 7.24-7.18 (m, 2H), 7.12 (q, *J* = 8.0 Hz, 4H), 7.01-6.88 (m, 4H), 3.89-3.62 (m, 2H), 3.31-3.06 (m, 2H), 2.71-2.43 (m, 2H), 2.06 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.8, 137.8 (d, *J* = 13.2 Hz), 136.9 (d, *J* = 13.3 Hz), 134.5, 133.6, 132.8 (d, *J* = 18.6 Hz), 132.1 (d, *J* = 18.9 Hz), 129.6, 129.1, 129.0, 128.8, 128.7, 128.6, 128.5, 127.1, 126.6, 125.2, 124.9, 123.6, 46.7 (d, *J* = 26.5 Hz), 42.8 (d, *J* = 24.4 Hz), 27.9 (d, *J* = 15.0 Hz), 26.8 (d, *J* = 14.3 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.85, -21.59.

HRMS(ESI): Calcd. for $C_{39}H_{36}NOP_2$: $[M+H]^+$ 596.2267; found: 596.2264.

[[bis[2-(diphenylphosphino)ethyl]amino]carbonyl]ferrocene[1] (L14)



L14 was prepared from ferrocenecarboxylic acid (230.0 mg, 1.0 mmol) and **2** (381.7 mg, 0.8 mmol) following the general procedure.

Yellow solid (381.0 mg, 73% yield).

M.p. 116.9-117.8 ℃.

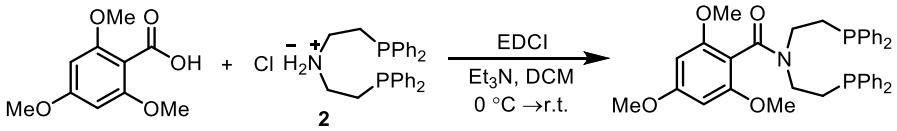
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56-7.29 (m, 20H), 4.43-4.32 (m, 2H), 4.22-4.17 (m, 2H), 4.15 (s, 5H), 3.71-3.40 (m, 4H), 2.53-2.16 (m, 4H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.3, 137.9, 137.5, 132.9, 132.8, 128.98, 128.95, 128.8, 128.7, 77.9, 70.4, 69.8, 69.7, 46.3 (d, *J* = 25.0 Hz), 44.4 (d, *J* = 23.0 Hz), 28.3 (d, *J* = 13.0 Hz), 26.4 (d, *J* = 14.1 Hz).

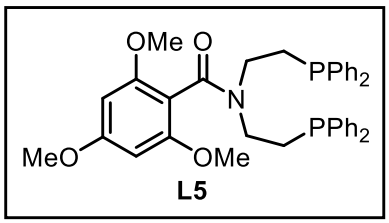
^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.16, -21.55.

HRMS(ESI): Calcd. for $C_{39}H_{38}FeNOP_2$: $[M+H]^+$ 654.1773; found: 654.1791.

3.2 Synthesis of L5



***N,N*-bis(2-(diphenylphosphaneyl)ethyl)-2,4,6-trimethoxybenzamide (L5)**



To a solution of **2** (190.9 mg, 0.4 mmol) and 2,4,6-trimethoxybenzoic acid (127.3 mg, 0.6 mmol) in dichloromethane (3.0 mL) was added EDCI (153.4 mg, 0.8 mmol) and triethylamine (121.4 mg, 167 μL, 1.2 mmol) at 0 ℃. The solution was stirred for 30 min prior to warming to room temperature and stirring for 18 h. The solution was

diluted with dichloromethane (5.0 mL) and washed with water, 1N HCl, saturated aqueous $NaHCO_3$ and brine. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was further purified by flash chromatography (petroleum ether/ethyl acetate = 3:1) to afford **L5** as a white solid. (212.0 mg, 83% yield).

M.p. 42.6-43.8 °C.

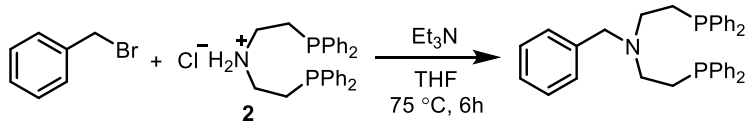
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (td, *J* = 7.6, 1.6 Hz, 4H), 7.38-7.26 (m, 8H), 7.26-7.20 (m, 4H), 7.18-7.12 (m, 4H), 6.03 (s, 2H), 3.84 (s, 3H), 3.68 (s, 6H), 3.62-3.54 (m, 2H), 3.25-3.15 (m, 2H), 2.48-2.37 (m, 2H), 2.15-2.05 (m, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.8, 161.9, 157.3, 138.0 (d, *J* = 12.3 Hz), 137.5 (d, *J* = 12.0 Hz), 132.8 (d, *J* = 18.6 Hz), 132.3 (d, *J* = 18.6 Hz), 128.73, 128.72, 128.60 (d, *J* = 6.7 Hz), 128.56 (d, *J* = 6.8 Hz), 108.0, 90.7, 55.8, 55.5, 46.4 (d, *J* = 26.6 Hz), 43.0 (d, *J* = 24.6 Hz), 27.5 (d, *J* = 14.4 Hz), 26.3 (d, *J* = 13.8 Hz).

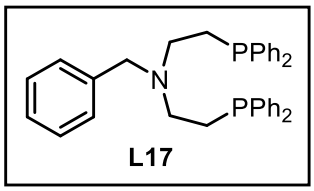
^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.09, -20.99.

HRMS(ESI): Calcd. for $C_{38}H_{40}NO_4P_2$: $[M+H]^+$ 636.2427; found: 636.2420.

3.3 Synthesis of L17



***N*-benzyl-2-(diphenylphosphaneyl)-*N*-(2-(diphenylphosphaneyl)ethyl)ethan-1-amine[2] (L17)**



Triethylamine (404.8 mg, 0.6 mL, 4.0 mmol) was added to a solution of **2** (381.7 mg, 0.8 mmol) in anhydrous tetrahydrofuran (4.0 mL) under nitrogen at 0 °C. After stirring for 10 min, (bromomethyl)benzene (171.0 mg, 1.0 mmol) was added, and the mixture was stirred at 75 °C for 6 h. Saturated aqueous $NaHCO_3$ was added to adjust pH to 8-9, and the aqueous layer was extracted with ethyl acetate. The combined

organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was further purified by flash chromatography (petroleum ether/ethyl acetate = 20:1) to afford **L17** as a white solid. (120.0 mg, 28% yield).

M.p. 87.8-88.9 ℃.

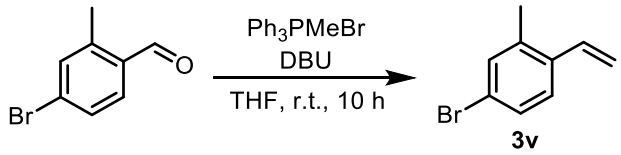
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40-7.26 (m, 23H), 7.26-7.20 (m, 2H), 3.59 (s, 2H), 2.69-2.55 (m, 4H), 2.24-2.07 (m, 4H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 139.2, 138.5 (d, *J* = 13.0 Hz), 132.70 (d, *J* = 18.5 Hz), 129.0, 128.6, 128.5 (d, *J* = 6.8 Hz), 128.3, 126.97, 58.1, 49.4 (d, *J* = 22.3 Hz), 25.5 (d, *J* = 12.6 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ -20.21.

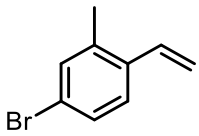
HRMS(ESI): Calcd. for $C_{35}H_{36}NP_2$: $[M+H]^+$ 532.2317; found: 532.2316.

4. Synthesis of substrates



3v

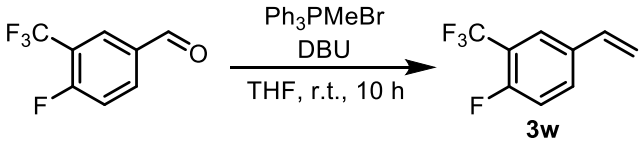
4-bromo-2-methyl-1-vinylbenzene[3] (**3v**)



To a solution of triphenylmethylphosphonium bromide (3.57 g, 10.0 mmol) in tetrahydrofuran (40.0 mL) was added DBU (1.67 g, 1.6 mL, 11.0 mmol) at 0 ℃. The mixture was stirring at 0 ℃ for 4 h and 4-bromo-2-methylbenzaldehyde (995.2 mg, 5.0 mmol) was added. The mixture was stirred for 10 h at room temperature. The solution was filtrated and concentrated. The residue was purified by flash column chromatography (petroleum ether) to get the product as a colorless oil (234.0 mg, 30% yield).

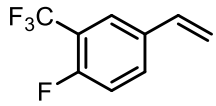
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36-7.28 (m, 3H), 6.85 (dd, *J* = 17.6, 11.2 Hz, 1H), 5.64 (dd, *J* = 17.6, 1.3 Hz, 1H), 5.32 (dd, *J* = 11.0, 1.2 Hz, 1H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 137.6, 135.9, 133.9, 133.0, 129.3, 127.0, 121.4, 116.0, 19.6.



3w

1-fluoro-2-(trifluoromethyl)-4-vinylbenzene[4] **(3w)**



To a solution of triphenylmethylphosphonium bromide (3.57 g, 10.0 mmol) in tetrahydrofuran (40.0 mL) was added DBU (1.67 g, 1.6 mL, 11.0 mmol) at 0 ºC. The mixture was stirring at 0 ºC for 4 h and 4-fluoro-3-(trifluoromethyl)benzaldehyde (960.6 mg, 5.0 mmol) was added. The mixture was stirred for 10 h at room temperature. The solution was filtrated and concentrated. The residue was purified by flash column chromatography (petroleum ether) to get the product as a colorless oil (290.0 mg, 38% yield).

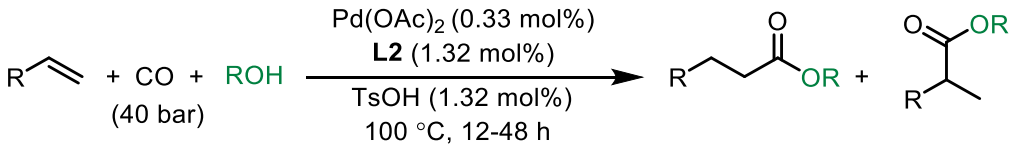
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64-7.59 (m, 1H), 7.59-7.53 (m, 1H), 7.15 (t, *J* = 9.2 Hz, 1H), 6.69 (dd, *J* = 17.2, 10.8 Hz, 1H), 5.74 (d, *J* = 17.6 Hz, 1H), 5.34 (d, *J* = 10.8 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 159.2 (dq, *J* = 255.1 Hz, *J* = 2.2 Hz), 134.6, 134.1 (d, *J* = 4.0 Hz), 131.3 (d, *J* = 8.3 Hz), 124.9 (qd, *J* = 4.6 Hz, *J* = 1.5 Hz), 122.7 (q, *J* = 270.9 Hz), 118.6 (qd, *J* = 32.6 Hz, *J* = 12.9 Hz), 117.1 (d, *J* = 20.8 Hz), 115.7 (d, *J* = 2.1 Hz).

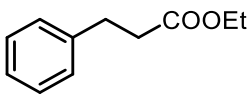
^{19}F NMR (376 MHz, Chloroform-*d*) δ -61.56 (d, *J* = 12.4 Hz), -116.09 (q, *J* = 12.4 Hz).

5. Palladium catalyzed hydroesterification of aromatic alkenes

General proceduce



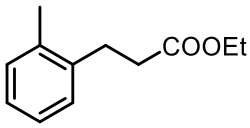
A 10 mL reaction tube was charged with $Pd(OAc)_2$ (0.5 mg, 0.33 mol%), TsOH (1.4 mg, 1.32 mol%) and equipped with a stirring bar. The alcohol (2.0 mL) solution of ligand (4.8 mg, 1.32 mol%) was added, followed by the addition of substrate alkenes (0.6 mmol). The tube was placed in a WP-MSAR-250A autoclave. At room temperature, the autoclave was purged with nitrogen three times and carbon monoxide three times, then pressurized to 40 *bar* of carbon monoxide. The reaction mixture was heated at 100 ℃ for specified time (12-48 h). After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. A sample of the mixture was analyzed by GC-MS and ^{1}H NMR. The desired product was obtained by flash column chromatography on silica gel (general eluent: petroleum ether /ethyl acetate = 60:1 to 20:1).



ethyl 3-phenylpropanoate[5] (**4a**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33-7.27 (m, 2H), 7.25-7.18 (m, 3H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.63 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

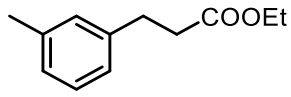
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.0, 140.7, 128.6, 128.4, 126.3, 60.5, 36.1, 31.1, 14.3.



ethyl 3-(*o*-tolyl)propanoate[5] (**4b**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.22-7.09 (m, 4H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.60 (t, *J* = 8.0 Hz, 2H), 2.36 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 138.8, 136.0, 130.3, 128.6, 126.5, 126.2, 60.5, 34.7, 28.4, 19.3, 14.3.

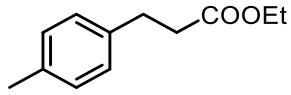


ethyl 3-(*m*-tolyl)propanoate[6] (**4c**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23-7.12 (m, 1H), 7.06-6.98 (m, 3H), 4.15 (q, *J* = 7.2 Hz, 2H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.63 (t, *J* = 8.0 Hz, 2H), 2.35 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.0, 140.6, 138.1, 129.2, 128.5, 127.0, 125.4, 60.4, 36.1, 31.0, 21.5, 14.3.

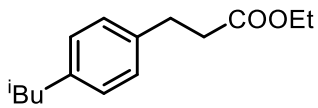
HRMS(ESI): Calcd. for $C_{12}H_{16}NaO_2$: $[M+Na]^+$ 215.1043; found: 215.1043.



ethyl 3-(*p*-tolyl)propanoate[5] (**4d**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.13-7.09 (m, 4H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.61 (t, *J* = 8.0 Hz, 2H), 2.33 (s, 3H), 1.25 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 137.6, 135.8, 129.3, 128.3, 60.5, 36.2, 30.7, 21.1, 14.3.

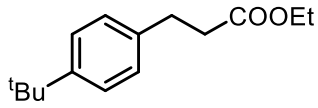


ethyl 3-(4-isobutylphenyl)propanoate (**4e**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17-7.05 (m, 4H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.62 (t, *J* = 8.0 Hz, 2H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.92-1.80 (m, 1H), 1.25 (t, *J* = 7.2 Hz, 3H), 0.91 (d, *J* = 6.8 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 139.6, 137.8, 129.3, 128.1, 60.4, 45.1, 36.1, 30.7, 30.3, 22.5, 14.3.

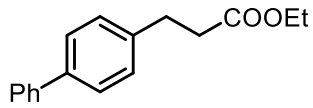
HRMS(ESI): Calcd. for $C_{15}H_{22}NaO_2$: $[M+Na]^+$ 257.1512; found: 257.1512.



ethyl 3-(4-(*tert*-butyl)phenyl)propanoate[7] (**4f**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36-7.30 (m, 2H), 7.18-7.13 (m, 2H), 4.15 (q, *J* = 7.2 Hz, 2H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.63 (t, *J* = 8.0 Hz, 2H), 1.32 (s, 9H), 1.25 (t, *J* = 7.2 Hz, 3H).

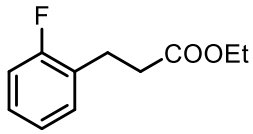
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.2, 149.1, 137.6, 128.1, 125.5, 60.5, 36.1, 34.5, 31.5, 30.6, 14.3.



ethyl 3-([1,1'-biphenyl]-4-yl)propanoate[8] (**4g**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65-7.61 (m, 2H), 7.61-7.56 (m, 2H), 7.50-7.45 (m, 2H), 7.41-7.35 (m, 1H), 7.35-7.31 (m, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 3.05 (t, *J* = 8.0 Hz, 2H), 2.71 (t, *J* = 8.0 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.9, 141.0, 139.7, 139.2, 128.79, 128.78, 127.24, 127.15, 127.0, 60.5, 35.9, 30.6, 14.3.

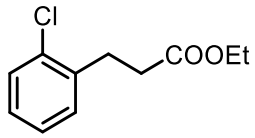


ethyl 3-(2-fluorophenyl)propanoate[9] (**4h**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24-7.15 (m, 2H), 7.08-6.97 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.98 (t, *J* = 8.0 Hz, 2H), 2.62 (t, *J* = 8.0 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.8, 161.3 (d, *J* = 243.8 Hz), 130.7 (d, *J* = 5.0 Hz), 128.2 (d, *J* = 8.1 Hz), 127.5 (d, *J* = 15.4 Hz), 124.1 (d, *J* = 3.6 Hz), 115.4 (d, *J* = 21.9 Hz), 60.6, 34.6 (d, *J* = 1.4 Hz), 24.7 (d, *J* = 2.8 Hz), 14.3.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -118.56.

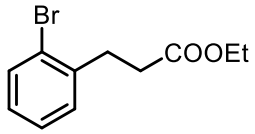


ethyl 3-(2-chlorophenyl)propanoate (**4i**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36-7.32 (m, 1H), 7.26-7.23 (m, 1H), 7.21-7.13 (m, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.8, 138.2, 134.1, 130.6, 129.7, 127.9, 127.0, 60.6, 34.1, 29.1, 14.3.

HRMS(ESI): Calcd. for $C_{11}H_{13}ClNaO_2$: $[M+Na]^+$ 235.0496; found: 235.0497.

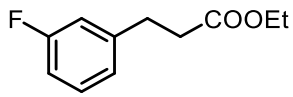


ethyl 3-(2-bromophenyl)propanoate[6] (**4j**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54-7.81 (m, 1H), 7.27-7.20 (m, 2H), 7.10-7.04 (m, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.06 (t, *J* = 8.0 Hz, 2H), 2.64 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.7, 139.9, 133.0, 130.6, 128.2, 127.6, 124.5, 60.6, 34.2, 31.5, 14.3.

HRMS(ESI): Calcd. for $C_{11}H_{14}BrO_2$: $[M+H]^+$ 257.0172; found: 257.0172.



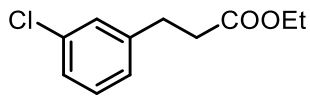
ethyl 3-(3-fluorophenyl)propanoate[6] (**4k**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27-7.21 (m, 1H), 7.00-6.95 (m, 1H), 6.93-6.85 (m, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.7, 163.0 (d, *J* = 244.0 Hz), 143.2 (d, *J* = 7.2 Hz), 130.0 (d, *J* = 8.4 Hz), 124.1 (d, *J* = 2.9 Hz), 115.3 (d, *J* = 20.9 Hz), 113.3 (d, *J* = 20.8 Hz), 60.6, 35.7, 30.8 (d, *J* = 1.8 Hz), 14.3.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.54.

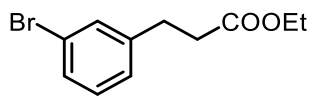
HRMS(ESI): Calcd. for $C_{11}H_{13}FNaO_2$: $[M+Na]^+$ 219.0792; found: 219.0788.



ethyl 3-(3-chlorophenyl)propanoate[10] (**4l**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24-7.15 (m, 3H), 7.11-7.06 (m, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.92 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

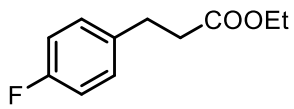
^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.6, 142.7, 134.3, 129.8, 128.6, 126.7, 126.6, 60.7, 35.7, 30.7, 14.3.



ethyl 3-(3-bromophenyl)propanoate[10] (**4m**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37-7.31 (m, 2H), 7.17-7.10 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.6, 143.0, 131.5, 130.1, 129.5, 127.1, 122.6, 60.6, 35.7, 30.6, 14.3.



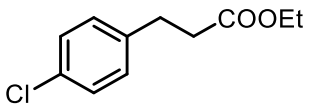
ethyl 3-(4-fluorophenyl)propanoate[6] (**4n**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.18-7.12 (m, 2H), 7.00-6.93 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.92 (t, *J* = 7.6 Hz, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.8, 161.6 (d, *J* = 242.5 Hz), 136.3 (d, *J* = 3.3 Hz), 129.8 (d, *J* = 7.7 Hz), 115.3 (d, *J* = 21.0 Hz), 60.6, 36.1, 30.3, 14.3.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.14.

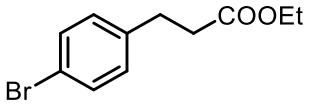
HRMS(ESI): Calcd. for $C_{11}H_{13}FNaO_2$: $[M+Na]^+$ 219.0792; found: 219.0796.



ethyl 3-(4-chlorophenyl)propanoate[5] (**4o**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.26-7.23 (m, 2H), 7.16-7.11 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

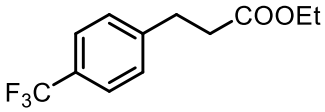
^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.7, 139.2, 132.1, 129.8, 128.7, 60.6, 35.9, 30.4, 14.3.



ethyl 3-(4-bromophenyl)propanoate[11] (**4p**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43-7.35 (m, 2H), 7.11-7.04 (m, 2H), 4.11 (q, *J* = 7.2 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.7, 139.6, 131.6, 130.2, 120.1, 60.6, 35.8, 30.4, 14.3.



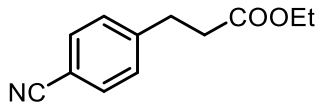
ethyl 3-(4-(trifluoromethyl)phenyl)propanoate[11] (**4q**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.00 (t, *J* = 7.6 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.6, 144.8 (d, *J* = 1.5 Hz), 128.8, 128.7 (q, *J*

= 32.1 Hz), 125.5 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 270.2 Hz), 60.7, 35.5, 30.8, 14.3.

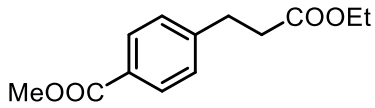
^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.44.



ethyl 3-(4-cyanophenyl)propanoate[9] (**4r**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.09 (q, *J* = 7.2 Hz, 2H), 2.98 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

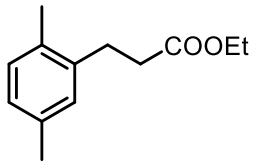
^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.2, 146.2, 132.3, 129.2, 119.0, 110.2, 60.7 35.1, 30.9, 14.2.



ethyl 4-(3-ethoxy-3-oxopropyl)benzoate[12] (**4s**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.90 (s, 3H), 3.00 (t, *J* = 7.6 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.7, 167.2, 146.1, 130.0, 128.5, 128.4, 60.7, 52.2, 35.5, 31.0, 14.3.

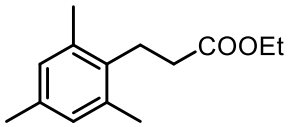


ethyl 3-(2,5-dimethylphenyl)propanoate (**4t**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.07-7.03 (m, 1H), 7.01-6.92 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.58 (t, *J* = 8.0 Hz, 2H), 2.31 (s, 3H), 2.30 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.2, 138.6, 135.6, 132.9, 130.3, 129.5, 127.1, 60.5, 34.9, 28.5, 21.1, 18.9, 14.3.

HRMS(ESI): Calcd. for $C_{13}H_{18}NaO_2$: $[M+Na]^+$ 229.1199; found: 229.1197.

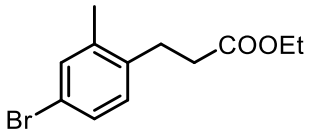


ethyl 3-mesitylpropanoate (**4u**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.86 (s, 2H), 4.19 (q, *J* = 7.2 Hz, 2H), 2.99-2.92 (m, 2H), 2.47-2.42 (m, 2H), 2.32 (s, 6H), 2.27 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 136.0, 135.4, 134.1, 129.0, 60.4, 33.6, 24.7, 20.8, 19.6, 14.2.

HRMS(ESI): Calcd. for $C_{14}H_{20}NaO_2$: $[M+Na]^+$ 243.1356; found: 243.1353.

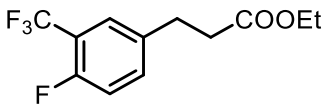


ethyl 3-(4-bromo-2-methylphenyl)propanoate (**4v**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30-7.27 (m, 1H), 7.26-7.22 (m, 1H), 7.03-6.98 (m, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.88 (t, *J* = 8.0 Hz, 2H), 2.54 (t, *J* = 8.0 Hz, 2H), 2.29 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.8, 138.4, 137.8, 133.1, 130.3, 129.1, 120.0, 60.6, 34.5, 27.9, 19.2, 14.3.

HRMS(ESI): Calcd. for $C_{12}H_{16}BrO_2$: $[M+H]^+$ 271.0328; found: 271.0329.



ethyl 3-(4-fluoro-3-(trifluoromethyl)phenyl)propanoate (**4w**, pale yellow oil)

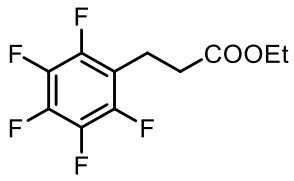
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45-7.41 (m, 1H), 7.40-7.34 (m, 1H), 7.14-7.07 (m, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.96 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.4, 157.3 (dd, *J* = 52.9, 2.2 Hz), 136.8 (d, *J*

= 3.9 Hz), 133.8 (d, *J* = 8.1 Hz), 127.0 (qd, *J* = 4.4, 1.3 Hz), 121.4 (qd, *J* = 270.6, 1.1 Hz), 118.2 (qd, *J* = 32.4, 12.5 Hz), 116.9 (d, *J* = 20.4 Hz), 60.8, 35.7, 30.1, 14.3.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -61.43 (d, *J* = 12.8 Hz), -118.40 (q, *J* = 12.8 Hz,).

HRMS(ESI): Calcd. for $C_{12}H_{13}F_4O_2$: $[M+H]^+$ 265.0846; found: 265.0840.

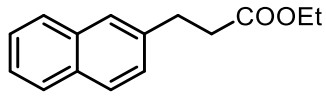


ethyl 3-(perfluorophenyl)propanoate[13] (**4x**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.13 (q, *J* = 7.2 Hz, 2H), 3.02 (t, *J* = 7.6 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.7, 145.3 (dm, *J* = 244.7 Hz), 140.2 (dm, *J* = 246.7 Hz), 137.4 (dm, *J* = 247.6 Hz), 113.6 (m), 60.9, 33.2, 18.0, 14.1.

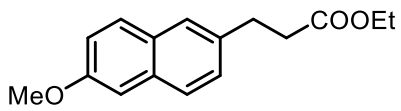
^{19}F NMR (376 MHz, Chloroform-*d*) δ -143.56-143.67 (m, 2F), -156.99 (t, *J* = 20.8 Hz, 1F), -162.53-162.72 (m, 2F).



ethyl 3-(naphthalen-2-yl)propanoate[11] (**4y**, pale yellow oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84-7.76 (m, 3H), 7.67-7.64 (m, 1H), 7.49-7.41 (m, 2H), 7.38-7.33 (m, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.13 (t, *J* = 7.6 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 138.2, 133.7, 132.2, 128.2, 127.7, 127.6, 127.1, 126.6, 126.1, 125.5, 60.6, 36.0, 31.2, 14.3.

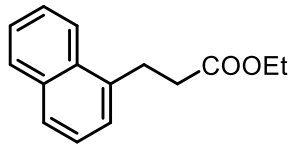


ethyl 3-(6-methoxynaphthalen-2-yl)propanoate[14] (**4z**, yellow solid)

M.p. 67.9-69.0 ℃.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70-7.65 (m, 2H), 7.61-7.55 (m, 1H), 7.34-7.29 (m, 1H), 7.16-7.10 (m, 2H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 3.09 (t, *J* = 8.0 Hz, 2H), 2.70 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

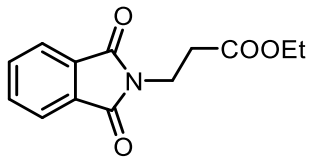
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 157.4, 135.8, 133.3, 129.13, 129.08, 127.6, 127.1, 126.4, 118.9, 105.7, 60.5, 55.4, 36.1, 31.1, 14.3.



ethyl 3-(naphthalen-1-yl)propanoate[5] (**4aa**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.08-8.01 (m, 1H), 7.90-7.86 (m, 1H), 7.77-7.72 (m, 1H), 7.57-7.47 (m, 2H), 7.44-7.34 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.44 (t, *J* = 8.0 Hz, 2H), 2.77 (t, *J* = 8.0 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.9, 136.5, 133.8, 131.6, 128.8, 127.1, 126.0, 125.9, 125.5, 125.5, 123.4, 60.4, 35.2, 28.1, 14.2.

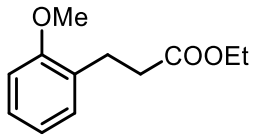


ethyl 3-(1,3-dioxoisoindolin-2-yl)propanoate[15] (**4ab**, white soild)

M.p. 50.0-50.9 ℃.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89-7.79 (m, 2H), 7.75-7.66 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.98 (t, *J* = 7.2 Hz, 2H), 2.71 (t, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H).

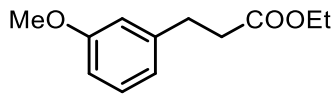
^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.9, 168.1, 134.2, 132.1, 123.4, 60.9, 33.9, 33.1, 14.2.



ethyl 3-(2-methoxyphenyl)propanoate[5] (**4ac**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23-7.13 (m, 2H), 6.91-6.82 (m, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.82 (s, 3H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.61 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

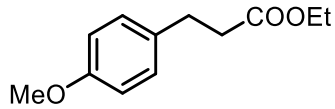
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.5, 157.6, 130.0, 129.0, 127.7, 120.5, 110.3, 60.4, 55.3, 34.3, 26.2, 14.3.



ethyl 3-(3-methoxyphenyl)propanoate[5] (**4ad**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24-7.16 (m, 1H), 6.83-6.72 (m, 3H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.79 (s, 3H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.62 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

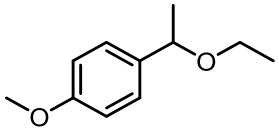
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.0, 159.8, 142.3, 129.6, 120.7, 114.1, 111.7, 60.5, 55.2, 36.0, 31.1, 14.3.



ethyl 3-(4-methoxyphenyl)propanoate[5] (**4ae**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15-7.09 (m, 2H), 6.86-6.80 (m, 2H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 2.89 (t, *J* = 8.0 Hz, 2H), 2.58 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

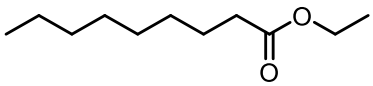
^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.1, 158.2, 132.8, 129.4, 114.0, 60.5, 55.3, 36.4, 30.2, 14.3.



1-(1-ethoxyethyl)-4-methoxybenzene[16] (**5**, colorless oil).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.36 (q, *J* = 6.4 Hz, 1H), 3.81 (s, 3H), 3.33 (q, *J* = 7.2 Hz, 2H), 1.42 (d, *J* = 6.4 Hz, 3H), 1.17 (t, *J* = 7.2 Hz, 3H).

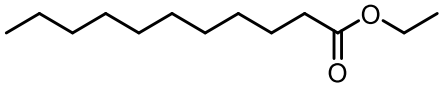
^{13}C NMR (100 MHz, Chloroform-*d*) δ 159.0, 136.4, 127.5, 113.9, 63.8, 55.4, 24.3, 15.6.



ethyl nonanoate[17] (**4af**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.11 (q, *J* = 7.2 Hz, 2H), 2.27 (t, *J* = 7.6 Hz, 2H), 1.65 – 1.55 (m, 2H), 1.35 – 1.19 (m, 13H), 0.86 (t, *J* = 6.8 Hz, 3H).

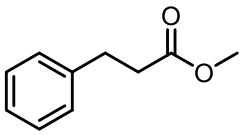
^{13}C NMR (100 MHz, Chloroform-*d*) δ 174.1, 60.3, 34.5, 32.0, 29.4, 29.28, 29.25, 25.1, 22.8, 14.4, 14.2.



ethyl undecanoate[18] (**4ag**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.12 (q, *J* = 7.2 Hz, 2H), 2.28 (t, *J* = 7.6 Hz, 2H), 1.65 – 1.56 (m, 2H), 1.33 – 1.20 (m, 17H), 0.87 (t, *J* = 6.0 Hz, 3H).

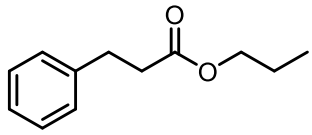
^{13}C NMR (100 MHz, Chloroform-*d*) δ 174.1, 60.3, 34.5, 32.0, 29.7, 29.6, 29.44, 29.40, 29.3, 25.1, 22.8, 14.4, 14.2.



methyl 3-phenylpropanoate[10] (**6**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32-7.27 (m, 2H), 7.25-7.17 (m, 3H), 3.68 (s, 3H), 2.96 (t, *J* = 7.8 Hz, 2H), 2.64 (t, *J* = 7.8 Hz, 2H).

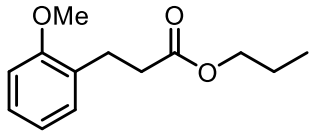
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.5, 140.6, 128.6, 128.4, 126.4, 51.7, 35.8, 31.1.



propyl 3-phenylpropanoate[19] (**7a**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34-7.27 (m, 2H), 7.25-7.17 (m, 3H), 4.04 (t, *J* = 6.8 Hz, 2H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.64 (t, *J* = 8.0 Hz, 2H), 1.68-1.59 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 140.7, 128.6, 128.4, 126.3, 66.2, 36.0, 31.1, 22.1, 10.5.

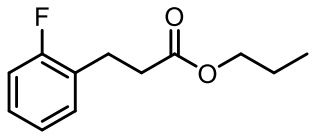


propyl 3-(2-methoxyphenyl)propanoate (**7b**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.13 (m, 2H), 6.90 – 6.82 (m, 2H), 4.03 (t, *J* = 6.8 Hz, 2H), 3.83 (s, 3H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.61 (t, *J* = 8.0 Hz, 2H), 1.68 – 1.58 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.7, 157.6, 130.0, 129.0, 127.7, 120.5, 110.3, 66.1, 55.3, 34.3, 26.3, 22.1, 10.5.

HRMS(ESI): Calcd. for $C_{13}H_{18}NaO_3$: $[M+Na]^+$ 245.1148; found: 245.1148.

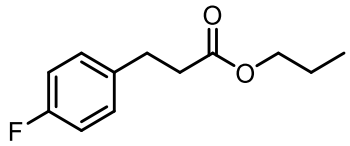


propyl 3-(2-fluorophenyl)propanoate[20] (**7c**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.15 (m, 2H), 7.08 – 6.98 (m, 2H), 4.03 (t, *J* = 6.8 Hz, 2H), 2.98 (t, *J* = 7.8 Hz, 2H), 2.64 (t, *J* = 7.8 Hz, 2H), 1.67 – 1.57 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.0, 161.3 (d, *J* = 243.7 Hz), 130.7 (d, *J* = 4.9 Hz), 128.2 (d, *J* = 8.2 Hz), 127.5 (d, *J* = 15.4 Hz), 124.2 (d, *J* = 3.6 Hz), 115.4 (d, *J* = 21.6 Hz), 66.3, 34.6 (d, *J* = 1.1 Hz), 24.8 (d, *J* = 2.6 Hz), 22.1, 10.5.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -118.54.



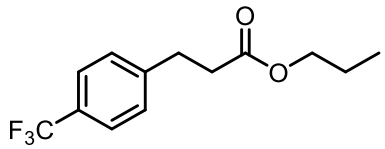
propyl 3-(4-fluorophenyl)propanoate (**7d**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.19 – 7.12 (m, 2H), 7.00 – 6.92 (m, 2H), 4.02 (t, *J* = 6.4 Hz, 2H), 2.92 (t, *J* = 7.8 Hz, 2H), 2.60 (t, *J* = 7.8 Hz, 2H), 1.68 – 1.56 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.0, 161.6 (d, *J* = 242.3 Hz), 136.3 (d, *J* = 3.3 Hz), 129.8 (d, *J* = 7.8 Hz), 115.3 (d, *J* = 21.1 Hz), 66.2, 36.1, 30.3, 22.1, 10.5.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.13.

HRMS(ESI): Calcd. for $C_{12}H_{15}FNaO_2$: $[M+Na]^+$ 233.0948; found: 233.0946.



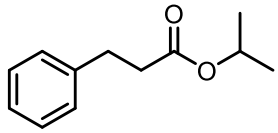
propyl 3-(4-(trifluoromethyl)phenyl)propanoate (**7e**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.02 (t, *J* = 6.8 Hz, 2H), 3.01 (t, *J* = 7.6 Hz, 2H), 2.65 (t, *J* = 7.6 Hz, 2H), 1.67 – 1.56 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.6, 144.7 (q, *J* = 1.3 Hz), 128.7, 128.6 (q, *J* = 32.2 Hz), 125.4 (q, *J* = 3.8 Hz), 124.3 (d, *J* = 270.1 Hz), 66.24, 35.41, 30.73, 21.93, 10.32.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.40.

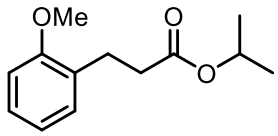
HRMS(ESI): Calcd. for $C_{13}H_{15}F_3NaO_2$: $[M+Na]^+$ 283.0916; found: 283.0916.



isopropyl 3-phenylpropanoate[19] (**8a**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34-7.27 (m, 2H), 7.25-7.18 (m, 3H), 5.09-4.96 (m, 1H), 2.96 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 8.0 Hz, 2H), 1.22 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.5, 140.7, 128.5, 128.4, 126.2, 67.7, 36.3, 31.1, 21.9.

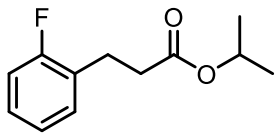


isopropyl 3-(2-methoxyphenyl)propanoate (**8b**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.12 (m, 2H), 6.90 – 6.81 (m, 2H), 5.05 – 4.95 (m, 1H), 3.83 (s, 3H), 2.93 (t, *J* = 8.0 Hz, 2H), 2.57 (t, *J* = 8.0 Hz, 2H), 1.21 (d, *J* = 6.0 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.1, 157.6, 130.1, 129.0, 127.6, 120.5, 110.3, 67.6, 55.3, 34.7, 26.3, 22.0.

HRMS(ESI): Calcd. for $C_{13}H_{18}NaO_3$: $[M+Na]^+$ 245.1148; found: 245.1147.



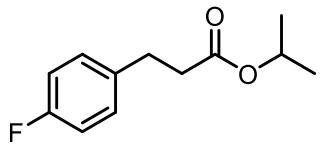
isopropyl 3-(2-fluorophenyl)propanoate (**8c**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.15 (m, 2H), 7.08 – 6.97 (m, 2H), 5.05 – 4.94 (m, 1H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.60 (t, *J* = 8.0 Hz, 2H), 1.20 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.4, 161.3 (d, *J* = 243.8 Hz), 130.8 (d, *J* = 5.0 Hz), 128.1 (d, *J* = 8.0 Hz), 127.5 (d, *J* = 15.6 Hz), 124.1 (d, *J* = 3.6 Hz), 115.4 (d, *J* = 21.8 Hz), 67.9, 34.9 (d, *J* = 1.5 Hz), 24.8 (d, *J* = 2.8 Hz), 21.9.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -118.48.

HRMS(ESI): Calcd. for $C_{12}H_{15}FNaO_2$: $[M+Na]^+$ 233.0948; found: 233.0945.



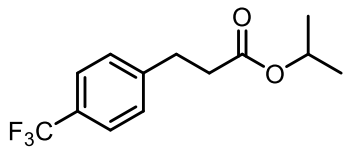
isopropyl 3-(4-fluorophenyl)propanoate (**8d**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.19 – 7.12 (m, 2H), 7.00 – 6.93 (m, 2H), 5.05 – 4.93 (m, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.19 (d, *J* = 6.0 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.3, 161.5 (d, *J* = 242.4 Hz), 136.2 (d, *J* = 3.2 Hz), 129.8 (d, *J* = 7.7 Hz), 115.2 (d, *J* = 21.1 Hz), 67.8, 36.3, 30.2, 21.8.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.19.

HRMS(ESI): Calcd. for $C_{12}H_{15}FNaO_2$: $[M+Na]^+$ 233.0948; found: 233.0946.



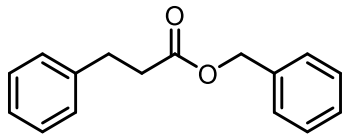
isopropyl 3-(4-(trifluoromethyl)phenyl)propanoate (**8e**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 7.6 Hz, 2H), 5.05 – 4.94 (m, 1H), 2.99 (t, *J* = 7.6 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.19 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.1, 144.8 (q, *J* = 1.3 Hz), 128.8, 128.7 (q, *J* = 32.1 Hz), 125.5 (q, *J* = 1.3 Hz), 124.4 (q, *J* = 270.2 Hz), 68.1, 35.8, 30.9, 21.9.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.39.

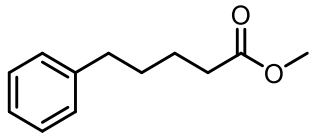
HRMS(ESI): Calcd. for $C_{13}H_{15}F_3NaO_2$: $[M+Na]^+$ 283.0916; found: 283.0927.



benzyl 3-phenylpropanoate[9] (**9**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43-7.30 (m, 8H), 7.25-7.22 (m, 2H), 5.16 (s, 2H), 3.02 (t, *J* = 7.8 Hz, 2H), 2.73 (t, *J* = 7.8 Hz, 2H).

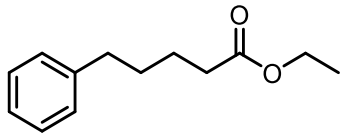
^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.8, 140.5, 136.0, 128.62, 128.58, 128.4, 128.29, 128.28, 126.3, 66.3, 36.0, 31.0.



methyl 5-phenylpentanoate[19] (**10**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34-7.27 (m, 2H), 7.23-7.17 (m, 3H), 3.68 (s, 3H), 2.65 (t, *J* = 7.2 Hz, 2H), 2.36 (t, *J* = 7.2 Hz, 2H), 1.74-1.64 (m, 4H).

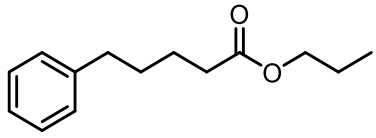
^{13}C NMR (100 MHz, Chloroform-*d*) δ 174.1, 142.2, 128.5, 128.4, 125.8, 51.5, 35.6, 34.0, 31.0, 24.7.



ethyl 5-phenylpentanoate[5] (**11**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32-7.27 (m, 2H), 7.22-7.17 (m, 3H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.65 (t, *J* = 7.2 Hz, 2H), 2.34 (t, *J* = 7.2 Hz, 2H), 1.73-1.65 (m, 4H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.7, 142.2, 128.4, 128.3, 125.8, 60.2, 35.6, 34.2, 30.9, 24.6, 14.3.

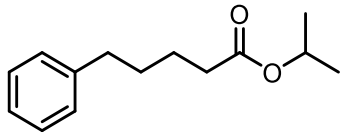


propyl 5-phenylpentanoate (**12**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32-7.26 (m, 2H), 7.21-7.15 (m, 3H), 4.03 (t, *J* = 6.8 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 2.34 (t, *J* = 7.2 Hz, 2H), 1.74-1.61 (m, 6H), 0.94 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.9, 142.3, 128.5, 128.4, 125.9, 66.0, 35.7, 34.3, 31.0, 24.8, 22.1, 10.5.

HRMS(ESI): Calcd. for $C_{14}H_{20}NaO_2$: $[M+Na]^+$ 243.1356; found: 243.1355.

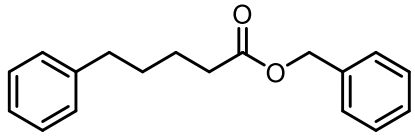


isopropyl 5-phenylpentanoate (**13**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33-7.26 (m, 2H), 7.23-7.15 (m, 3H), 5.08-4.97 (m, 1H), 2.64 (t, *J* = 6.8 Hz, 2H), 2.31 (t, *J* = 6.8 Hz, 2H), 1.72-1.64 (m, 4H), 1.24 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.3, 142.3, 128.5, 128.4, 125.9, 67.5, 35.7, 34.6, 31.0, 24.8, 22.0.

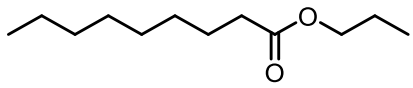
HRMS(ESI): Calcd. for $C_{14}H_{20}NaO_2$: $[M+Na]^+$ 243.1356; found: 243.1356.



benzyl 5-phenylpentanoate[21] (**14**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39-7.33 (m, 5H), 7.31-7.26 (m, 2H), 7.21-7.15 (m, 3H), 5.12 (s, 2H), 2.63 (t, *J* = 7.2 Hz, 2H), 2.40 (t, *J* = 7.2 Hz, 2H), 1.75-1.64 (m, 4H).

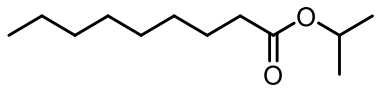
^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.6, 142.2, 136.2, 128.7, 128.5, 128.4, 128.3, 128.3, 125.9, 66.3, 35.7, 34.3, 31.0, 24.7.



propyl nonanoate[22] (**15**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.02 (t, *J* = 6.8 Hz, 2H), 2.29 (t, *J* = 7.6 Hz, 2H), 1.69 – 1.56 (m, 4H), 1.35 – 1.17 (m, 10H), 0.93 (t, *J* = 7.2 Hz, 3H), 0.87 (t, *J* = 6.0 Hz, 3H).

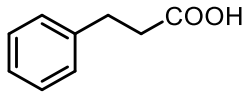
^{13}C NMR (100 MHz, Chloroform-*d*) δ 174.2, 66.0, 34.5, 31.9, 29.4, 29.29, 29.27, 25.2, 22.8, 22.1, 14.2, 10.5.



isopropyl nonanoate[23] (**16**, colorless oil)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.05 – 4.94 (m, 1H), 2.25 (t, *J* = 7.6 Hz, 2H), 1.66 – 1.53 (m, 2H), 1.36 – 1.24 (m, 10H), 1.22 (dd, *J* = 6.4 Hz, 6H), 0.87 (t, *J* = 6.0 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 173.6, 67.4, 34.9, 32.0, 29.4, 29.27, 29.26, 25.2, 22.8, 22.0, 14.3.



3-phenylpropanoic acid[24] (**17**, white solid)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 11.49 (brs, 1H), 7.34 – 7.27 (m, 2H), 7.25 – 7.19 (m, 3H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.70 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 179.3, 140.3, 128.7, 128.4, 126.5, 35.7, 30.7.

6. Gram-scale reaction

A 25 mL reaction tube was charged with $Pd(OAc)_2$ (9.0 mg, 0.33 mol%), TsOH (27.6 mg, 1.32 mol%) and equipped with a stirring bar. The EtOH (20.0 mL) solution of

ligand (96.9 mg, 1.32 mol%) was added, followed by the addition of styrene (1.25 g, 12.0 mmol). The tube was placed in a YZPR-50 autoclave. At room temperature, the autoclave was purged with nitrogen three times and carbon monoxide three times, then pressurized to 40 *bar* of carbon monoxide. The reaction mixture was heated at 100 °C for 24 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. The desired product was obtained by flash column chromatography on silica gel (general eluent: petroleum ether /ethyl acetate = 50:1) as a colorless oil (1.75 g, 82% yield).

7. References

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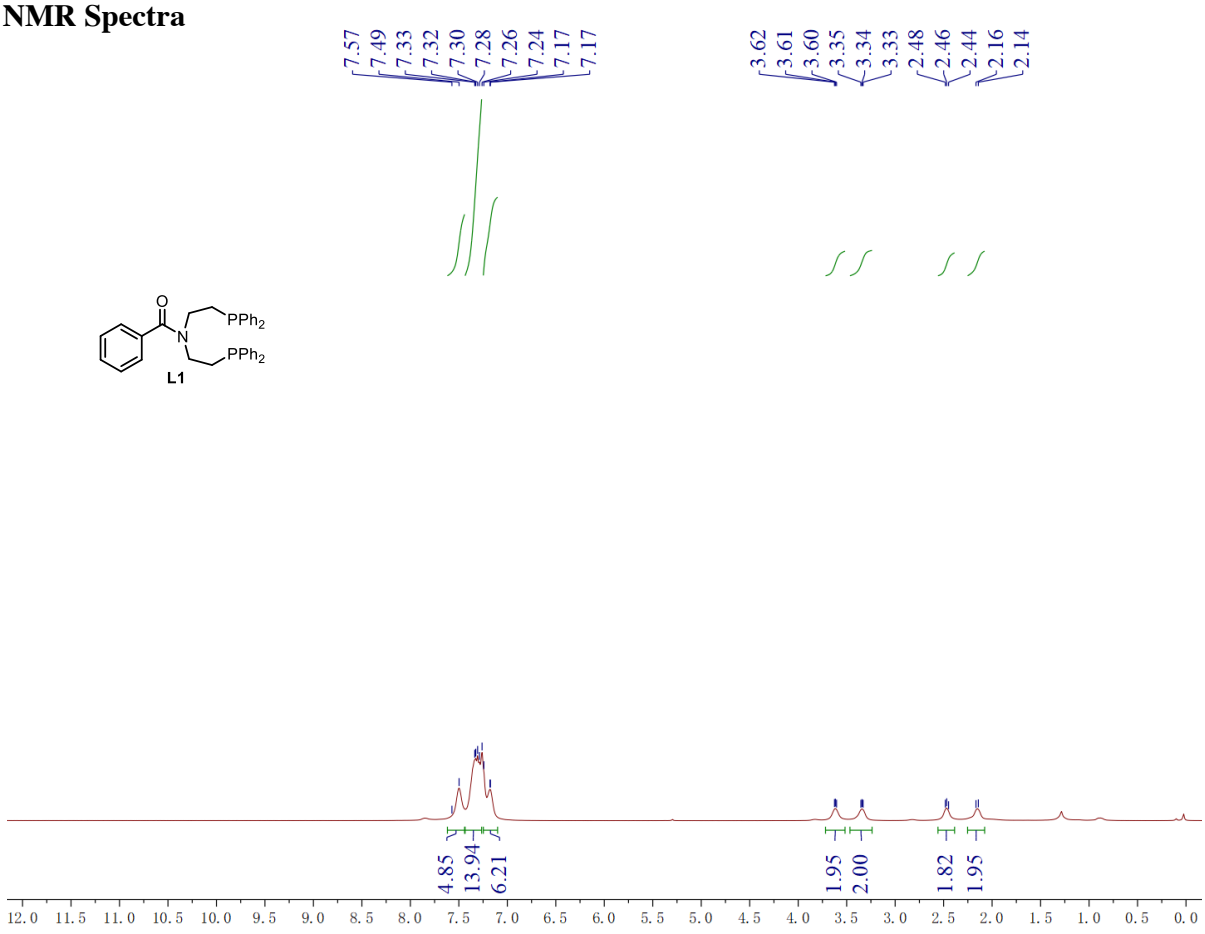
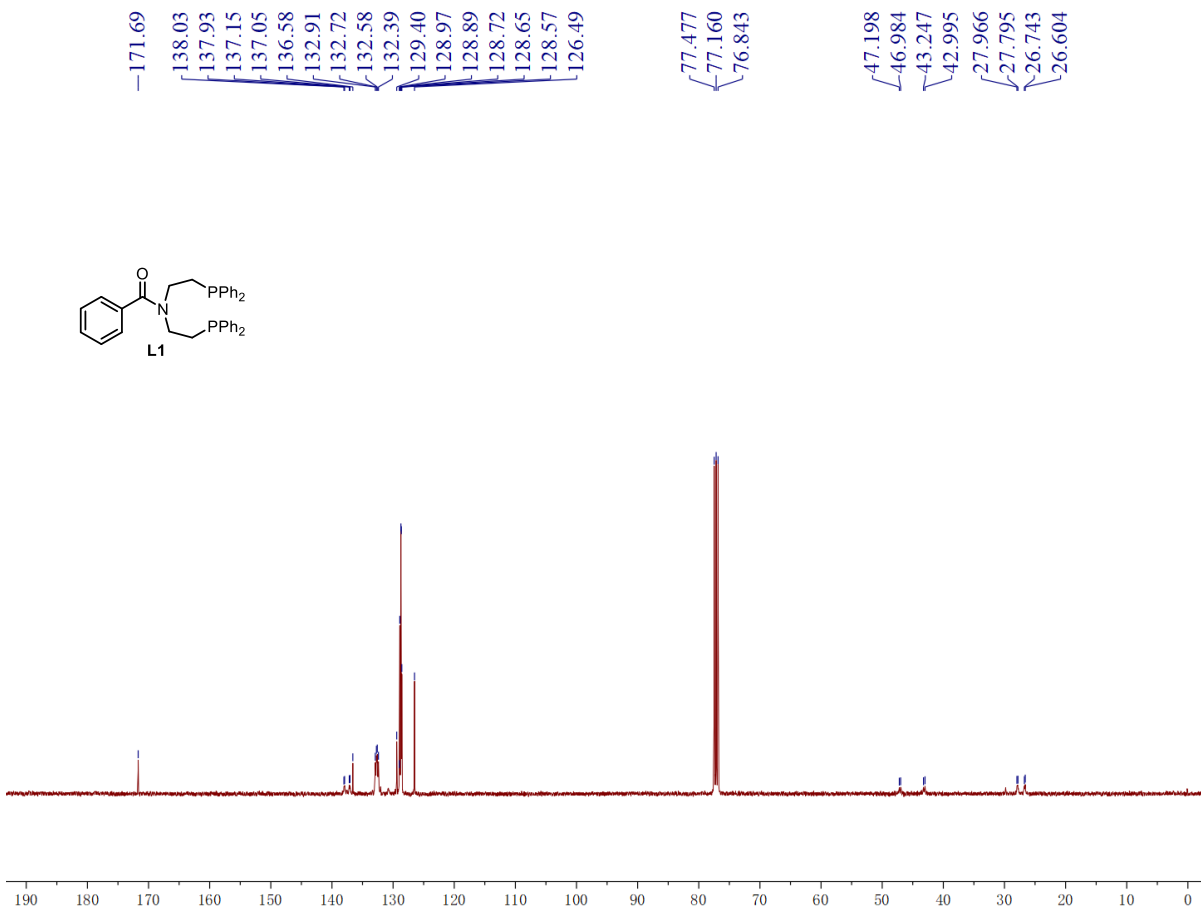
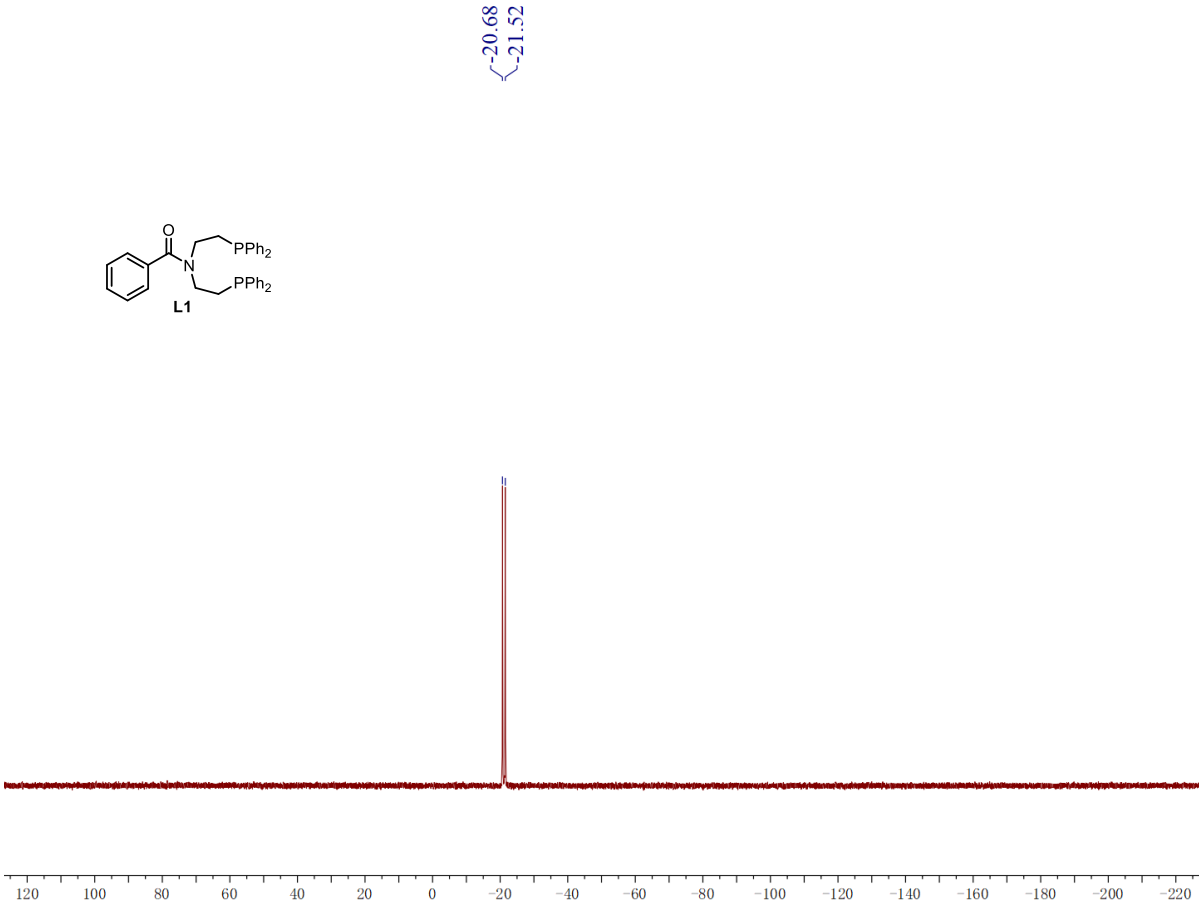
8. NMR Spectra



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-21.52
O
N
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PPh2
L1
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-80
-100
-120
-140
-160
-180
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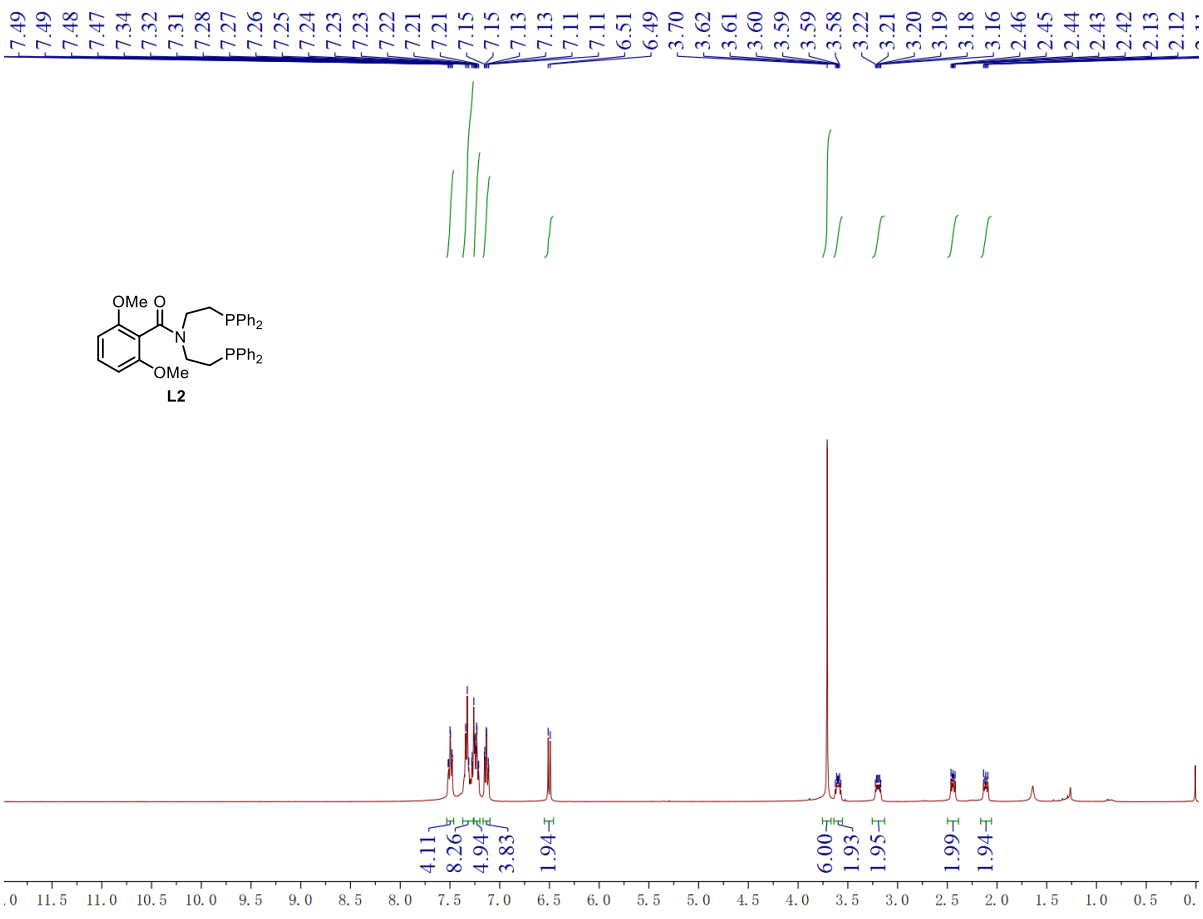
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OMe
O
N
PPh2
PPh2
OMe
L2
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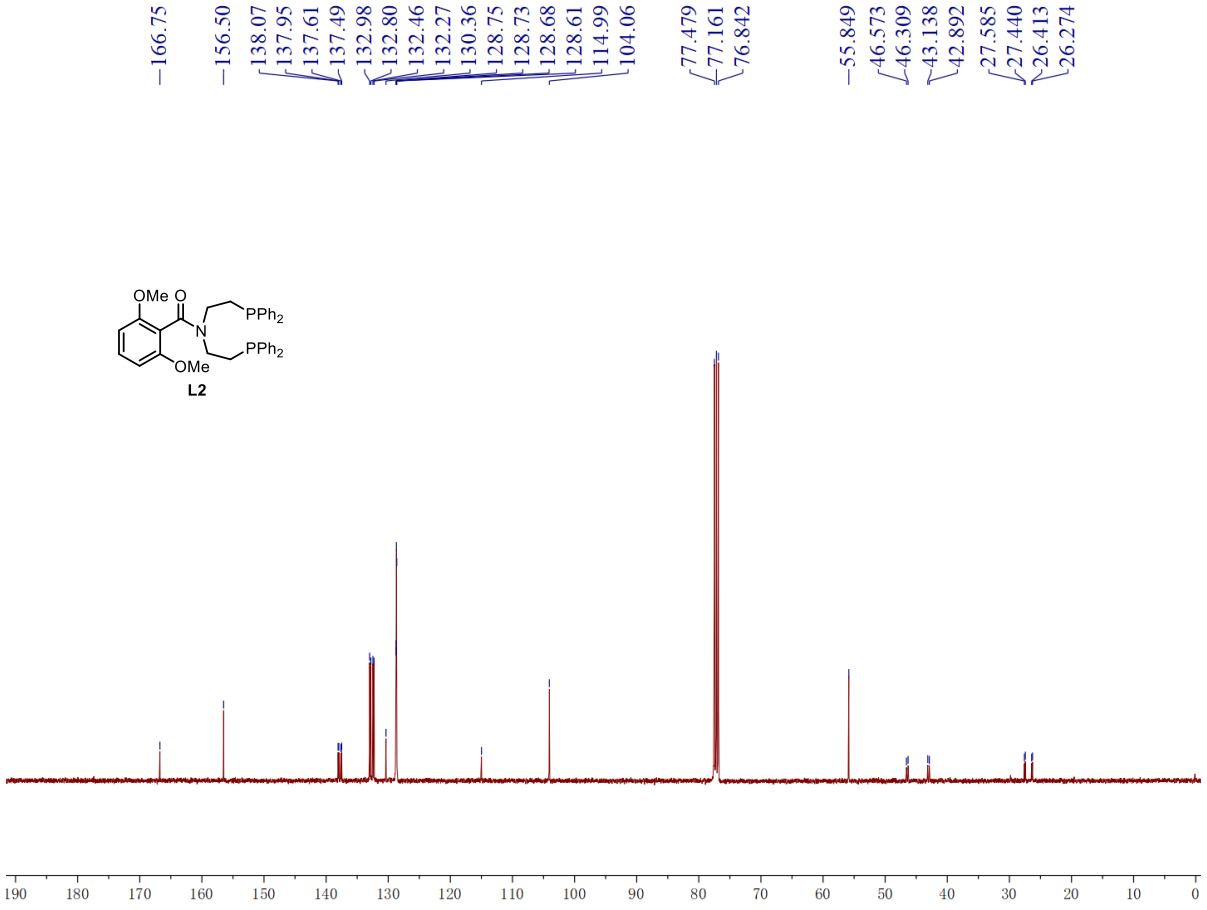
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L2
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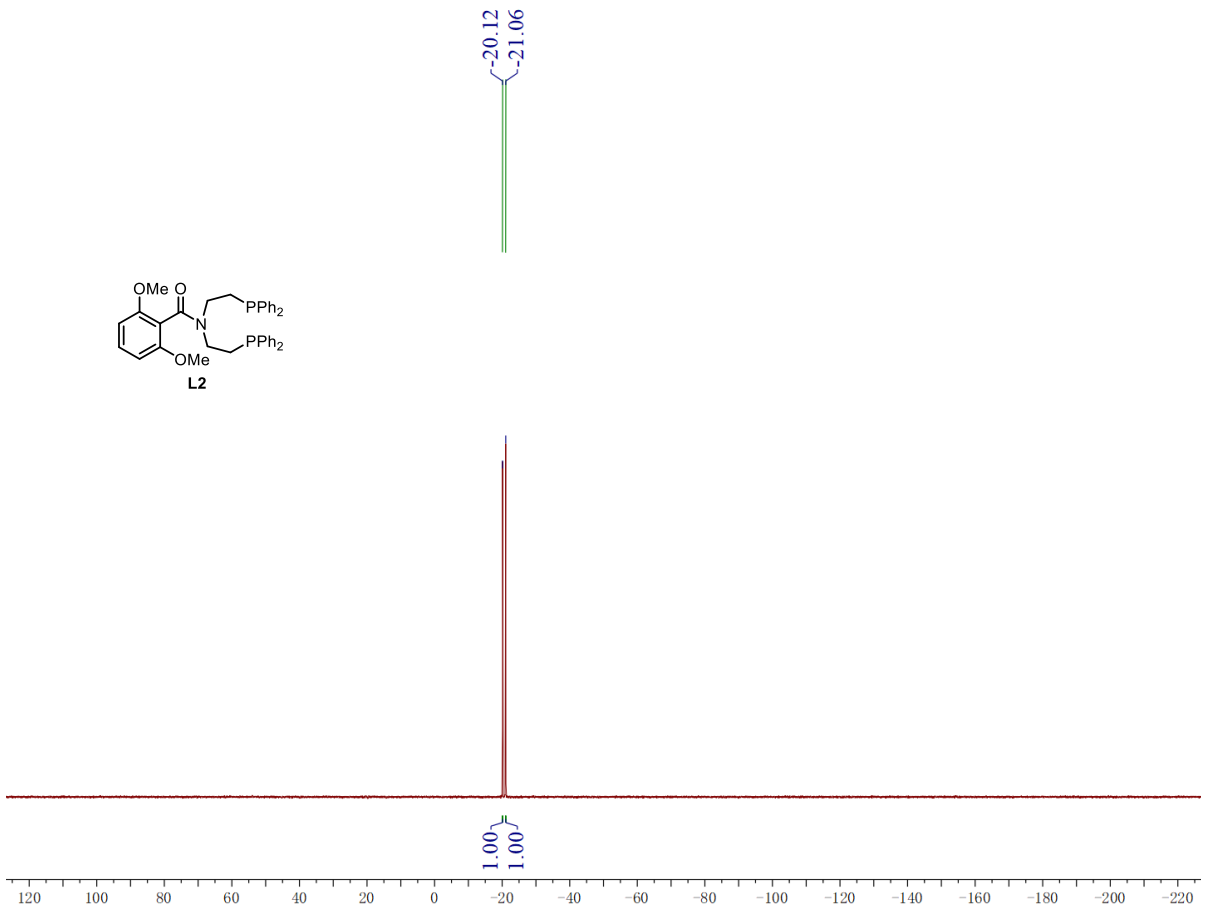
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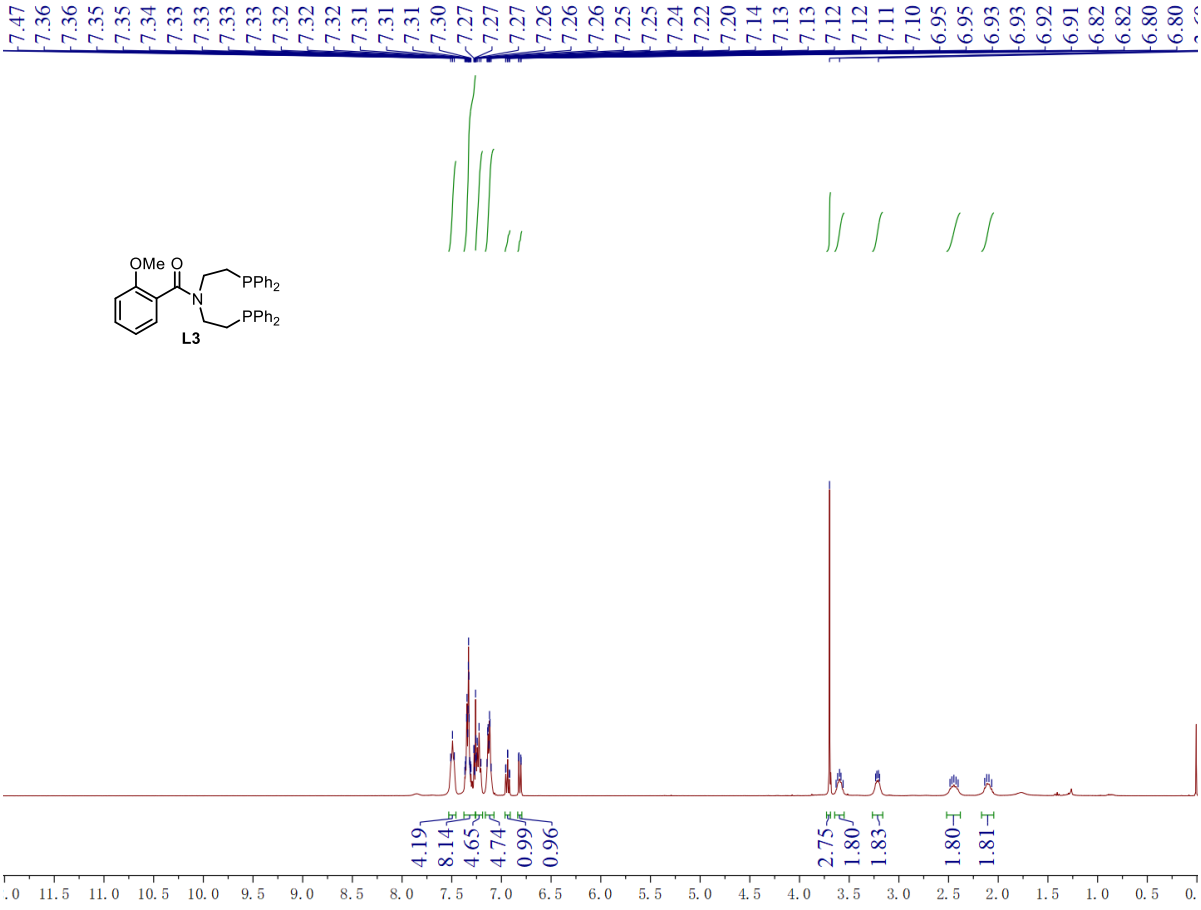
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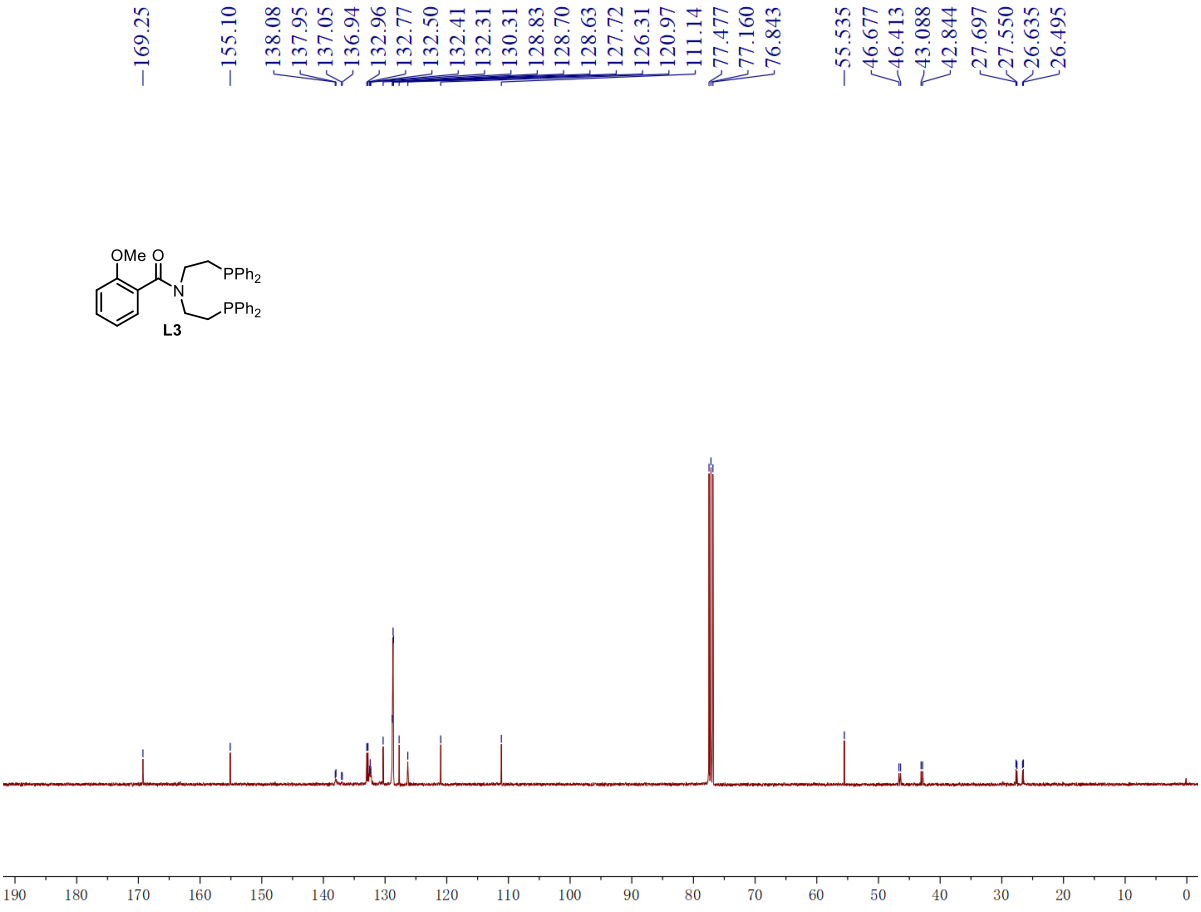
OMe O
PPh2
PPh2
L3
4.19 8.14 4.65 4.74 0.99 0.96 2.75 1.80 1.83 1.80 1.81
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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PPh2
L3
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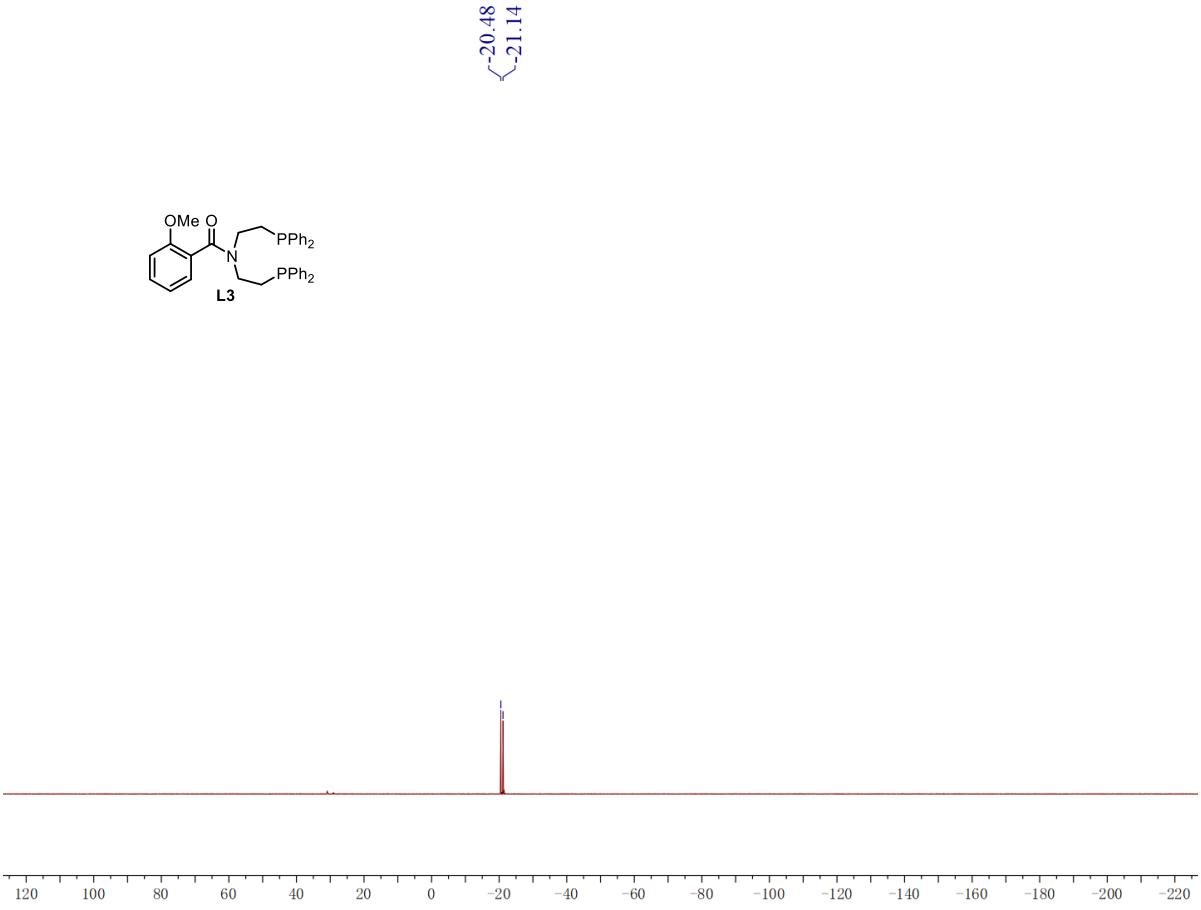
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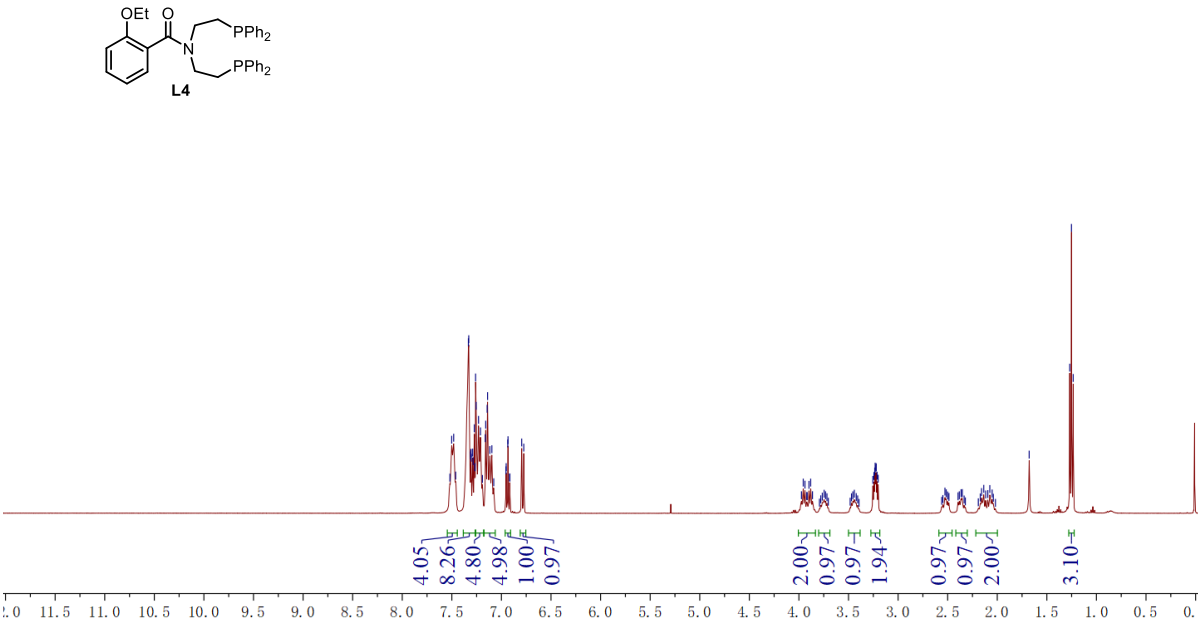
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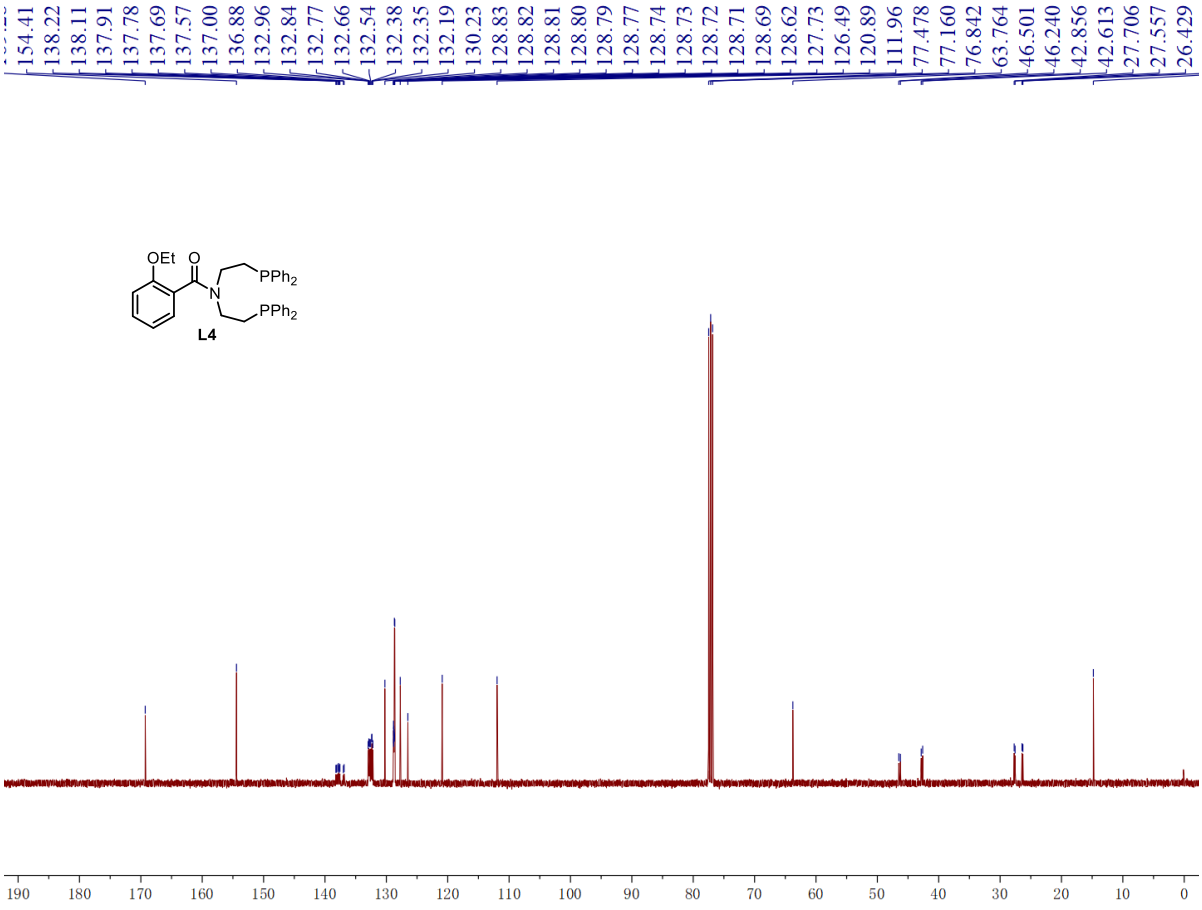
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PPh2
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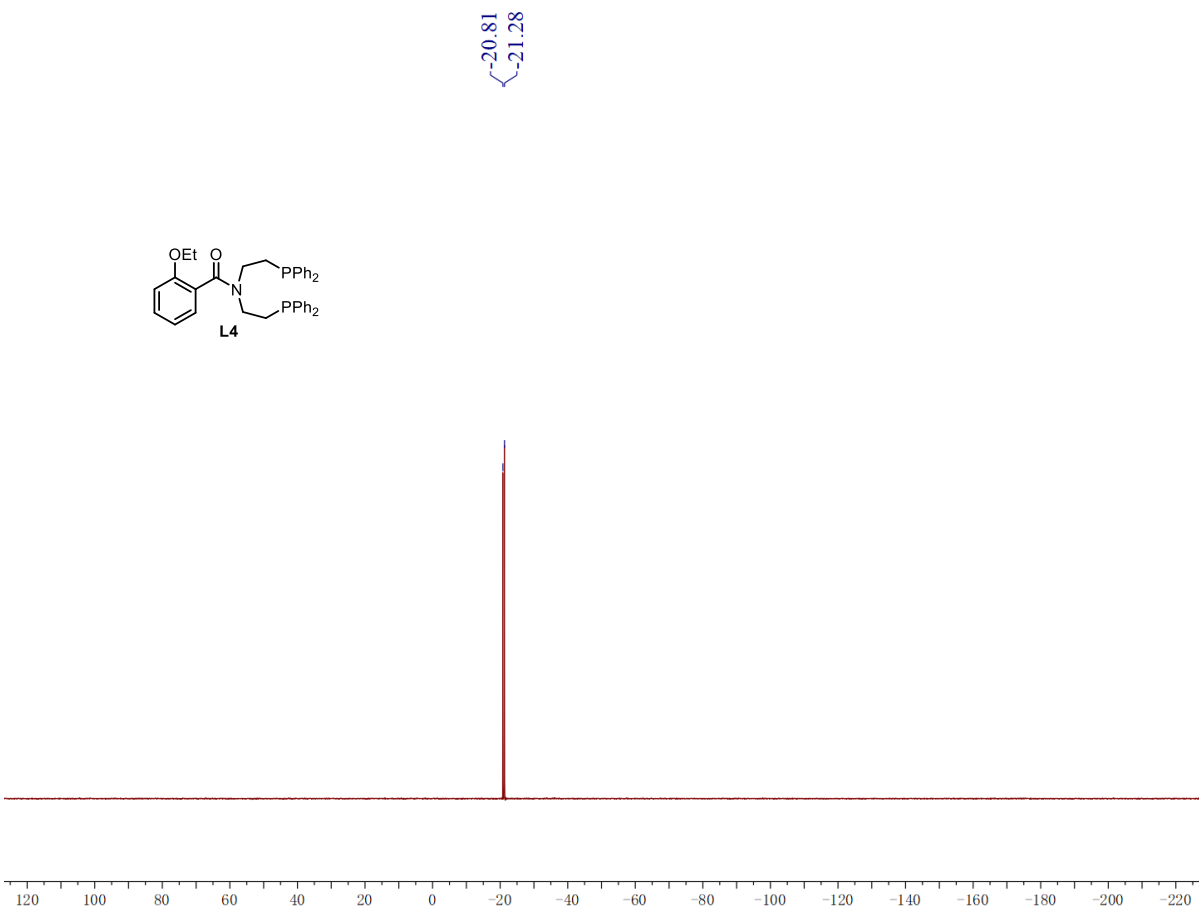
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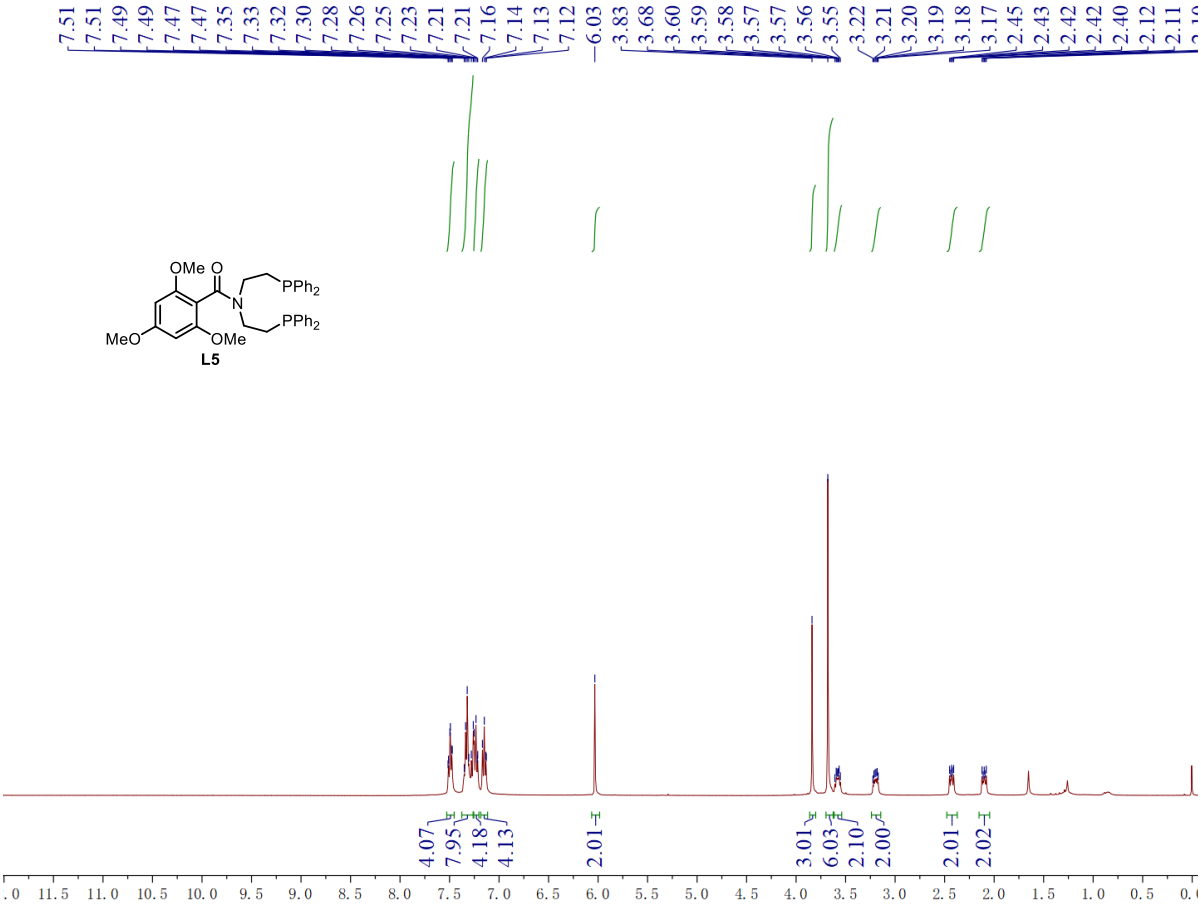
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2.12
2.11
OMe O
PPh2
PPh2
MeO
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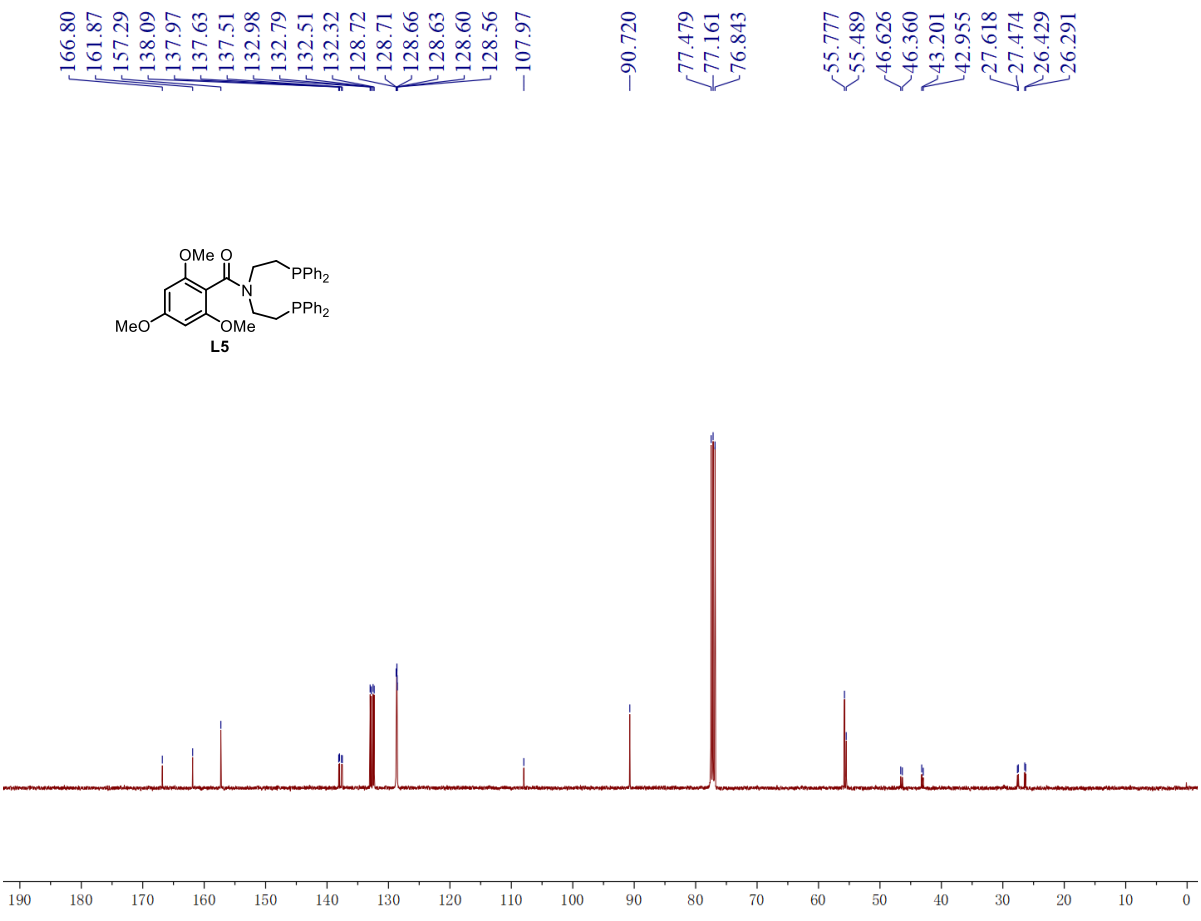
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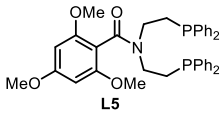
PPh2
PPh2
MeO
OMe
L5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PPh2
PPh2
MeO
OMe

L5

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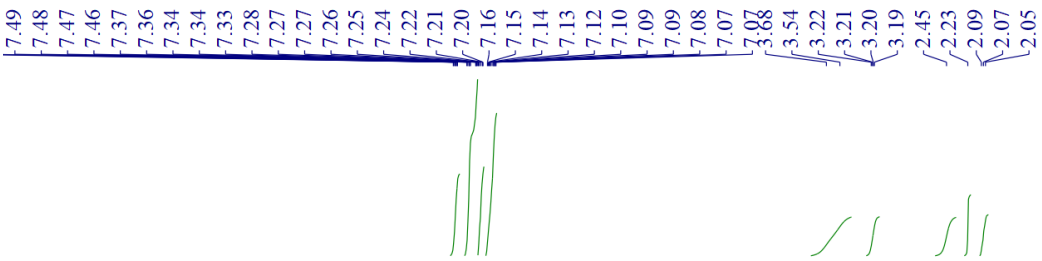
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7.08
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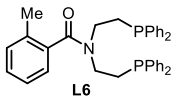
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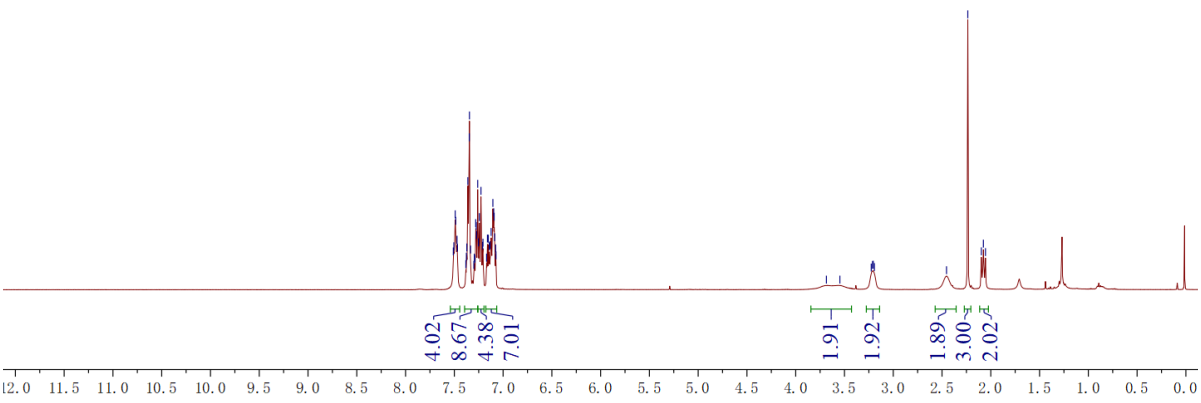
PPh2
PPh2

L6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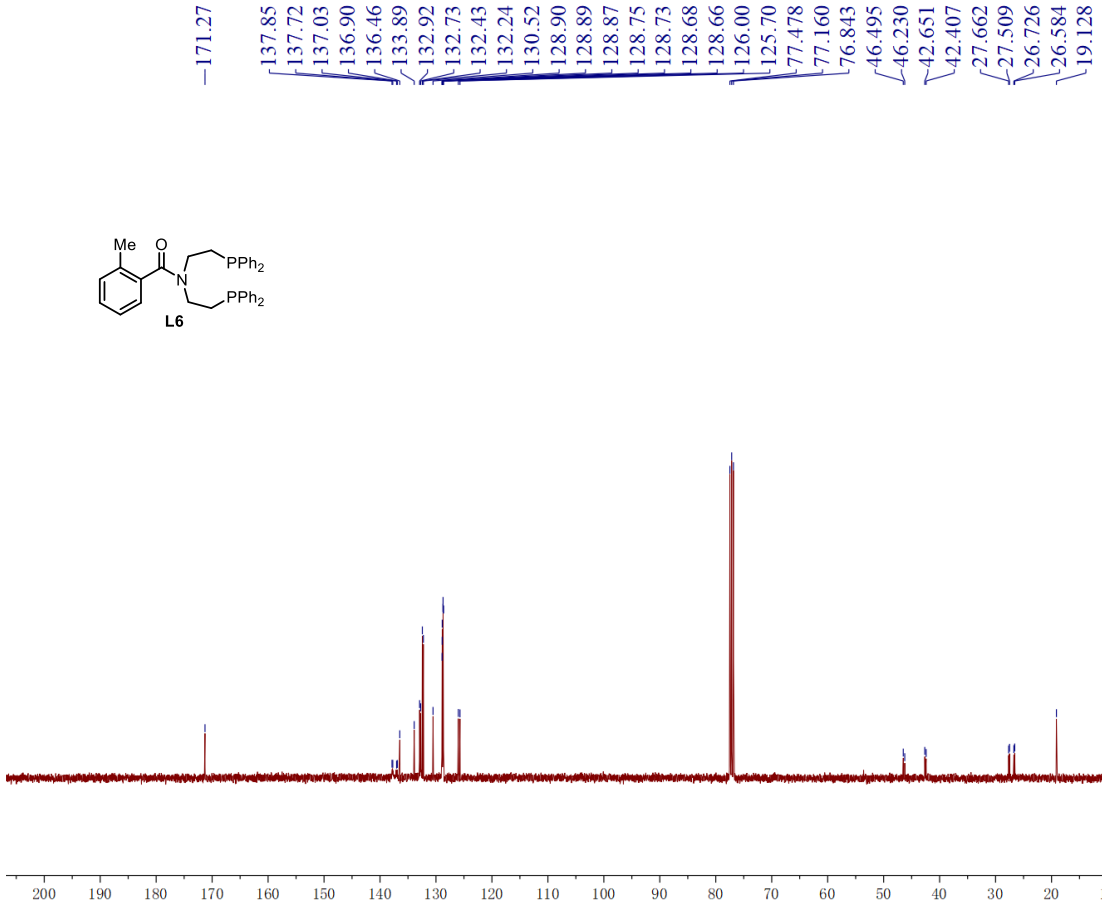
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PPh2
PPh2
L6
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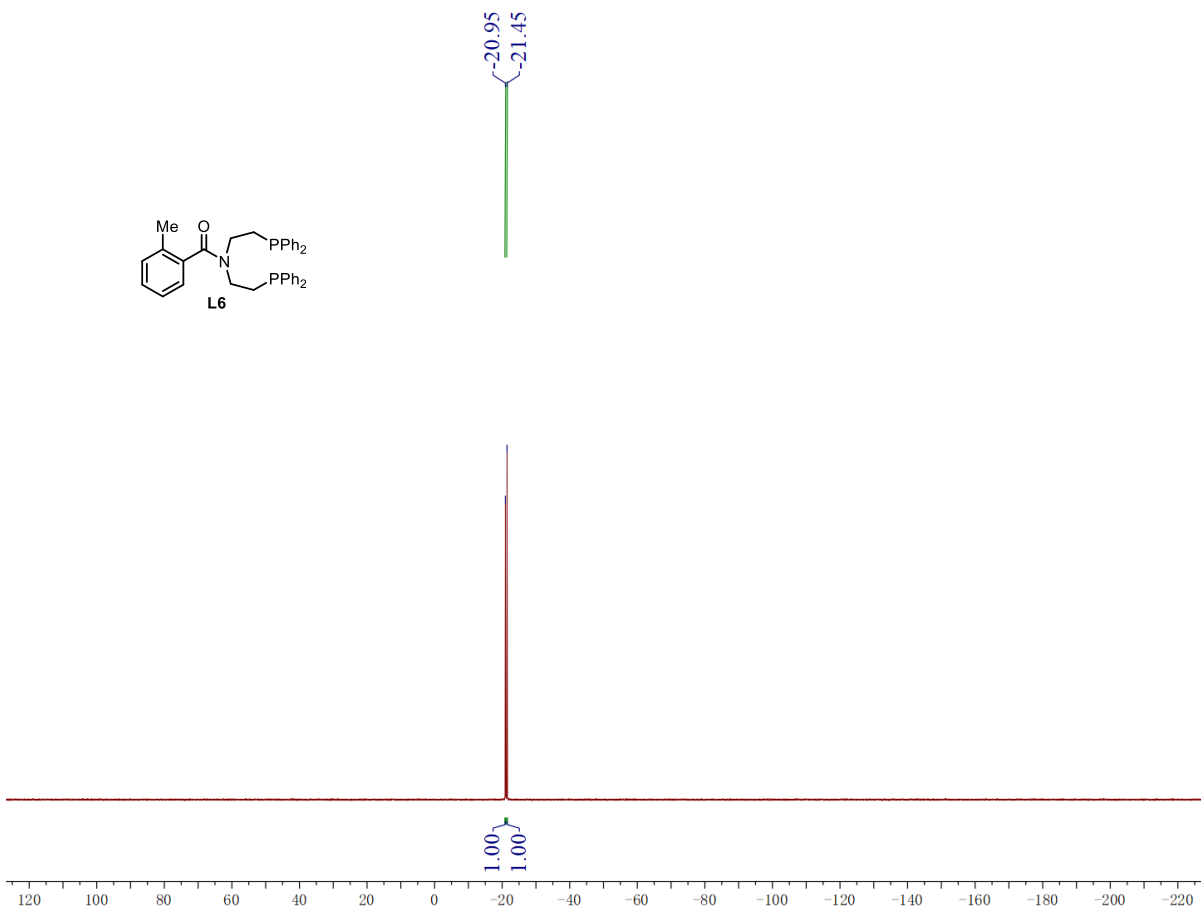
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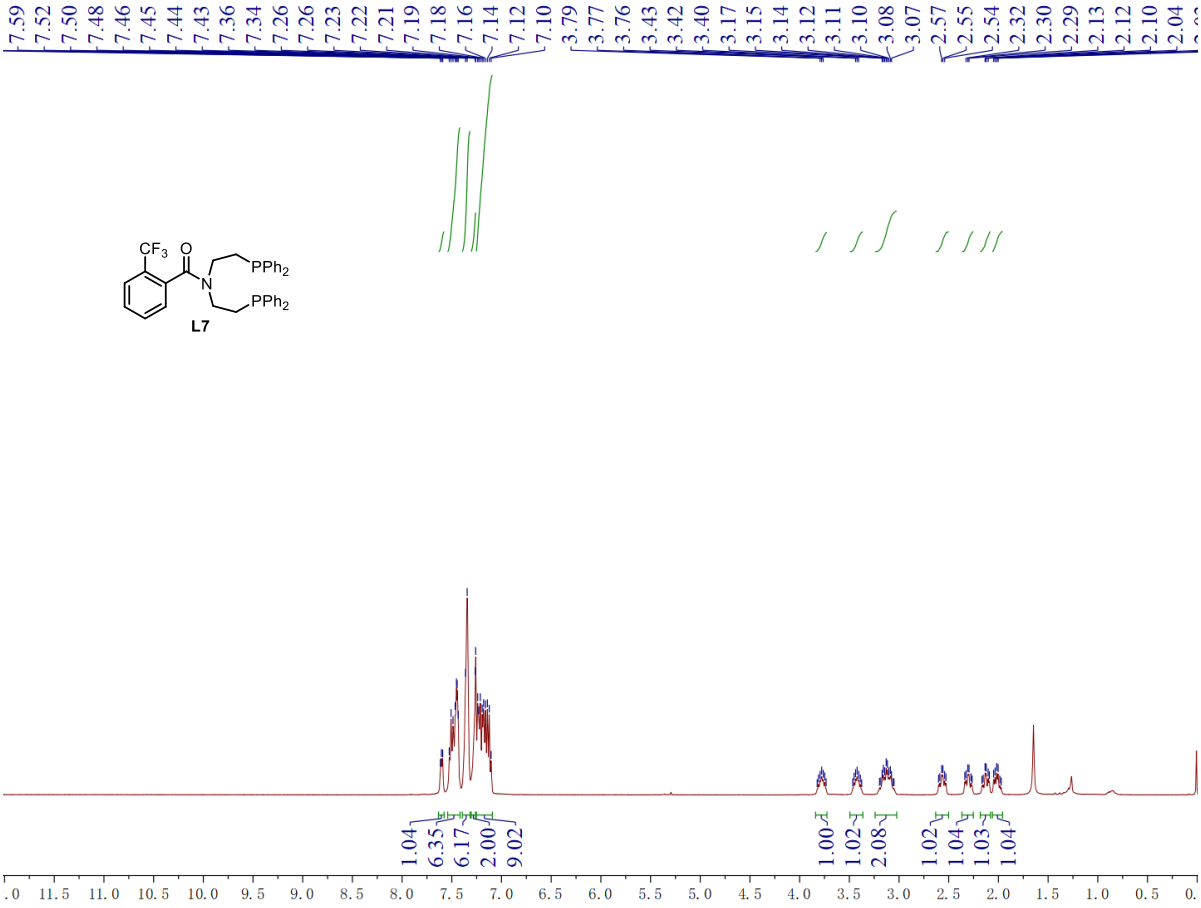
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3.79 3.77 3.76 3.43 3.42 3.40 3.17 3.15 3.14 3.12 3.11 3.10 3.08 3.07
2.57 2.55 2.54 2.32 2.30 2.29 2.13 2.12 2.10 2.04
CF3 O PPh2 N PPh2
L7
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1.00 1.02 2.08 1.02 1.04 1.03 1.04
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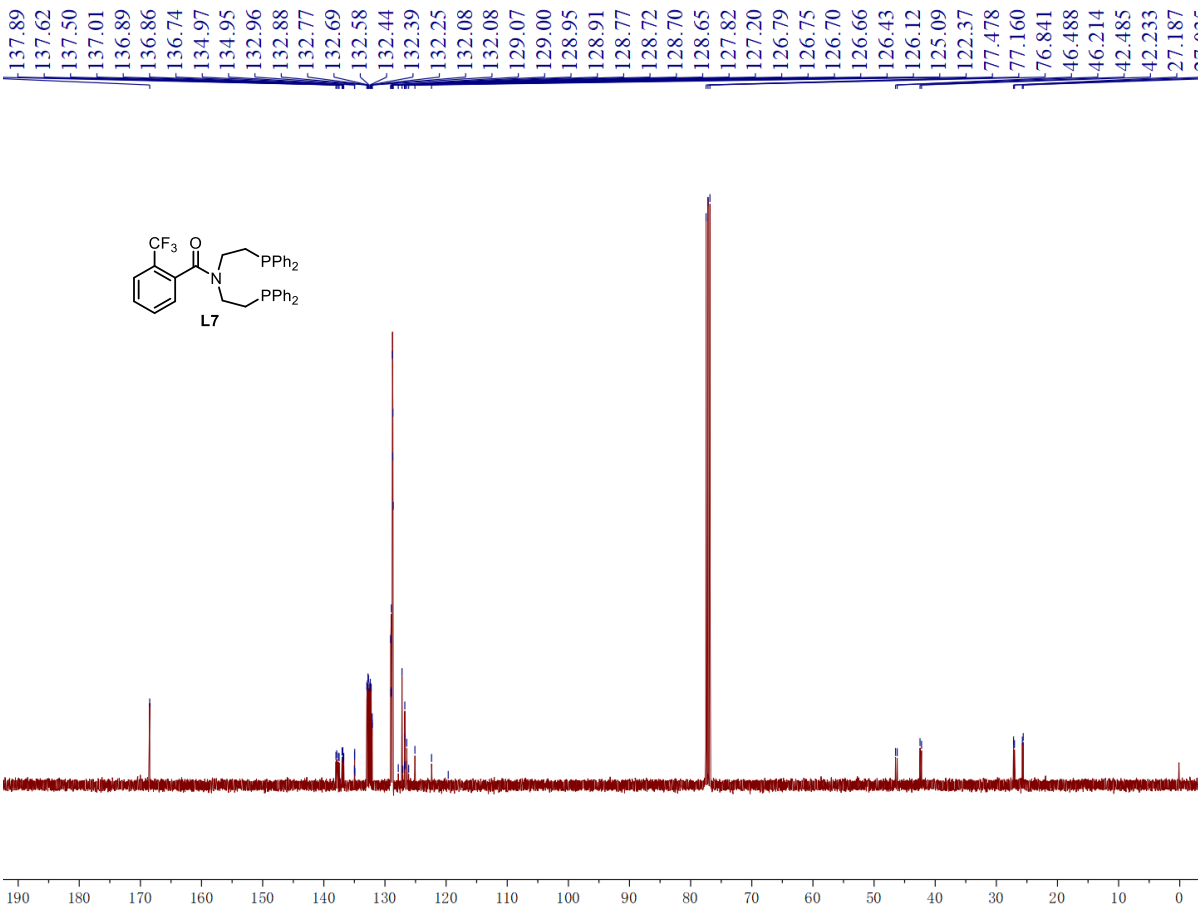
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L7
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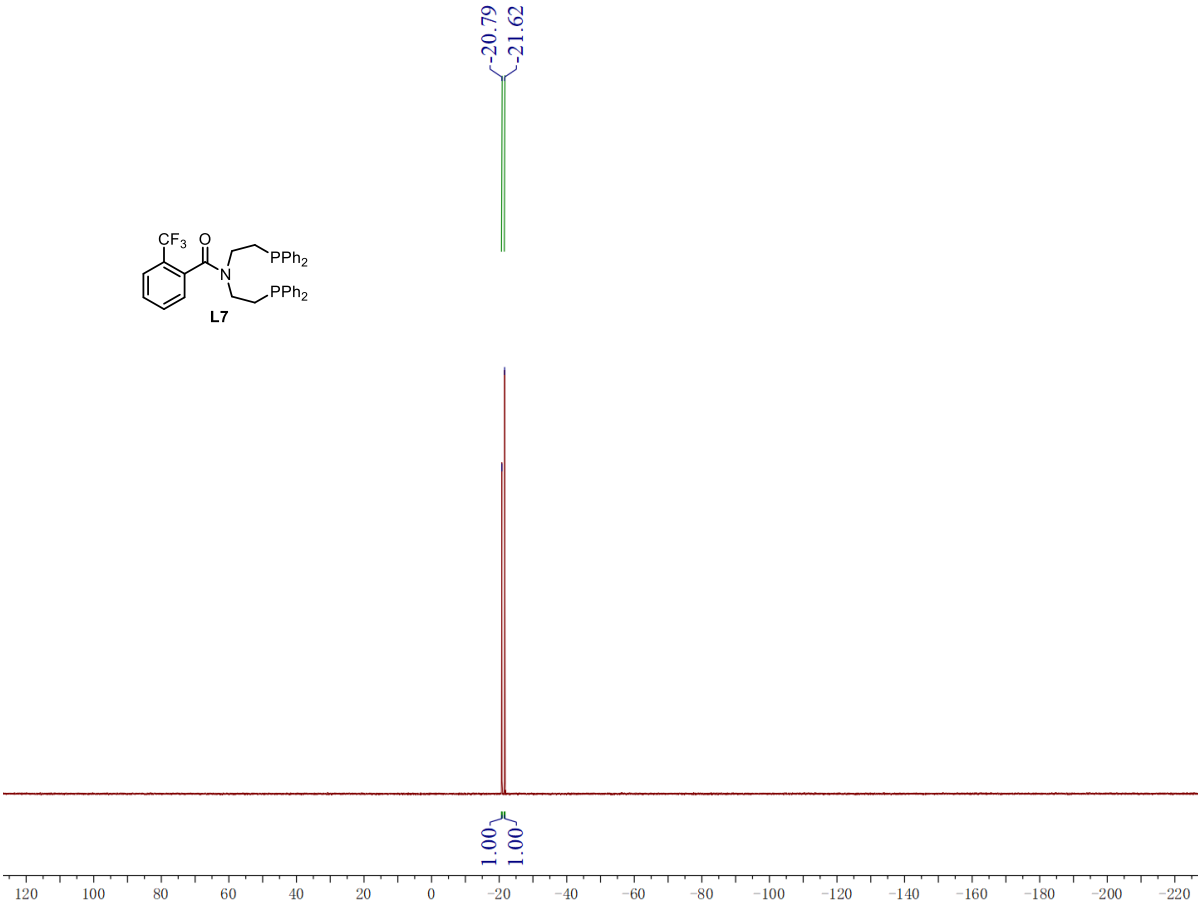
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PPh2
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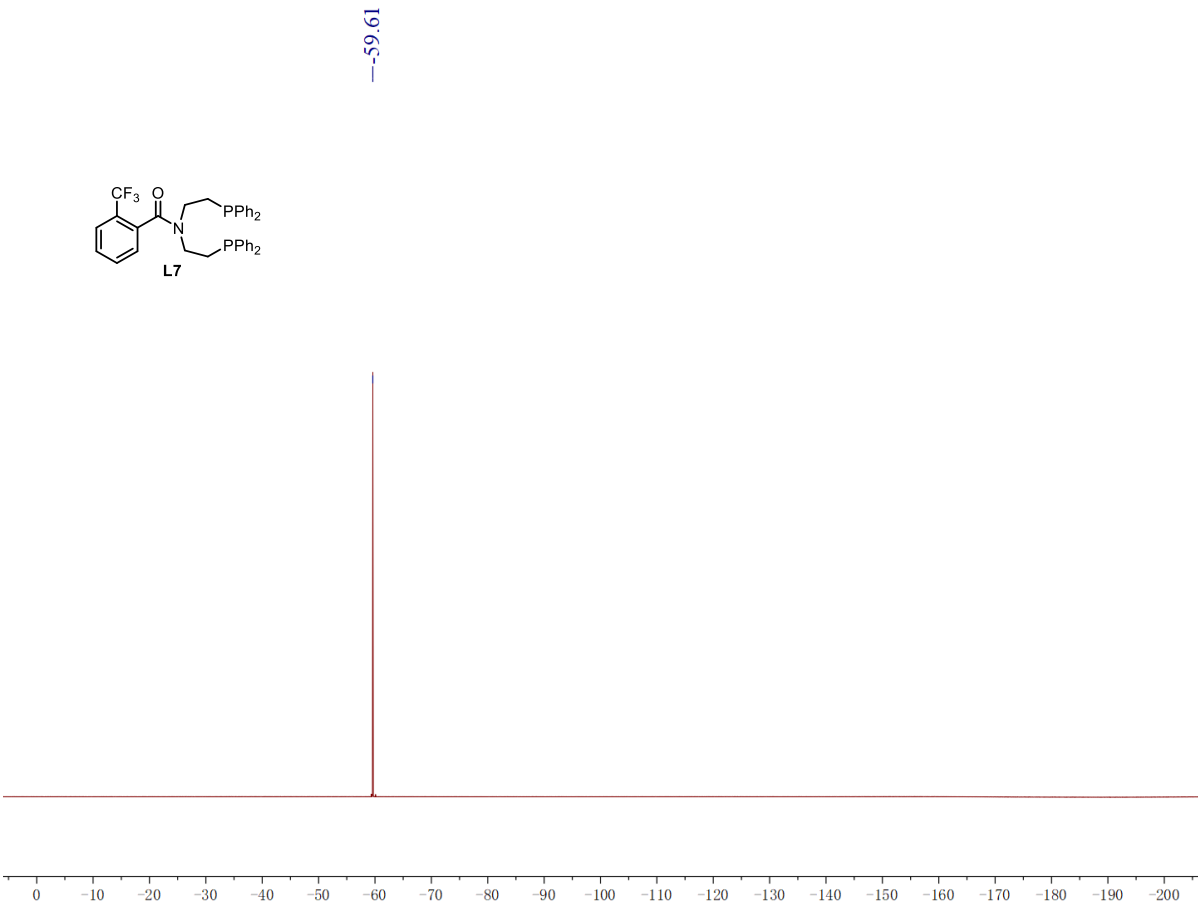
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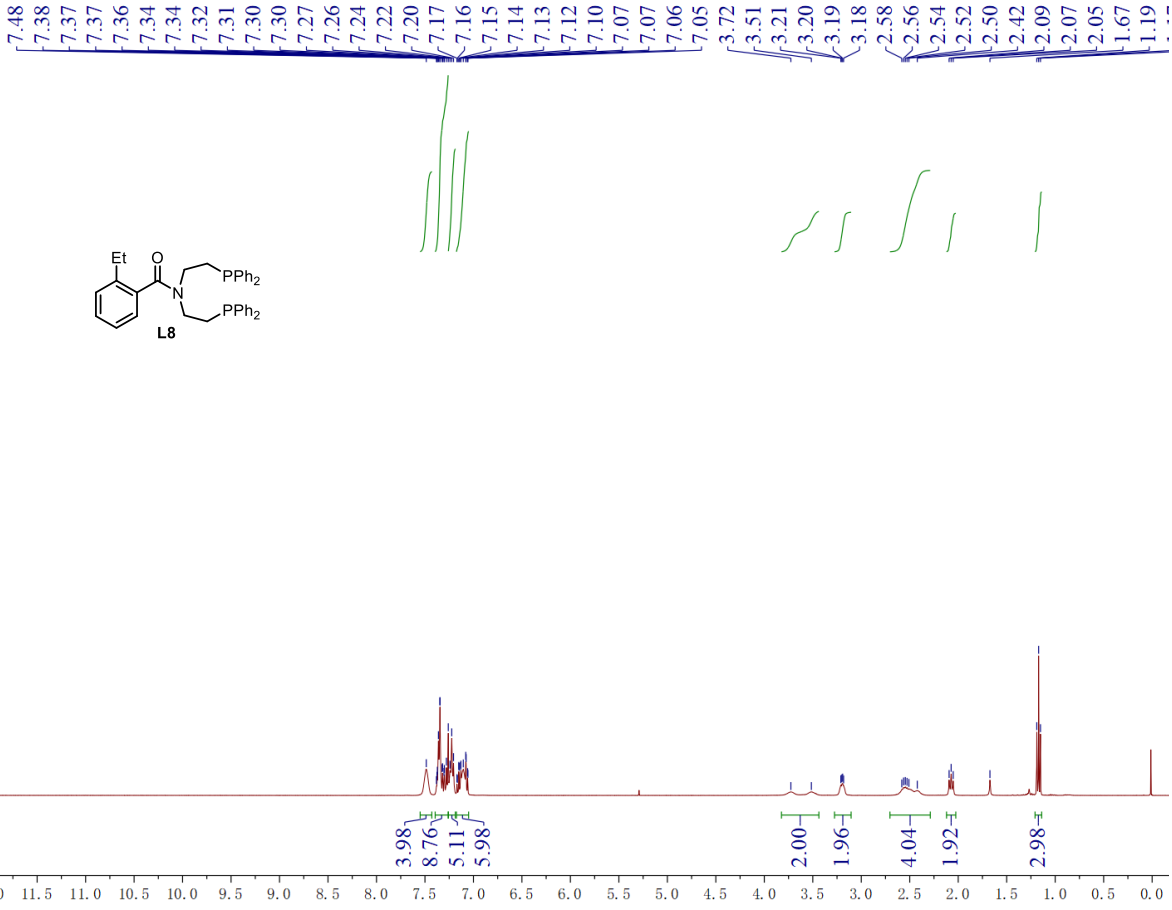
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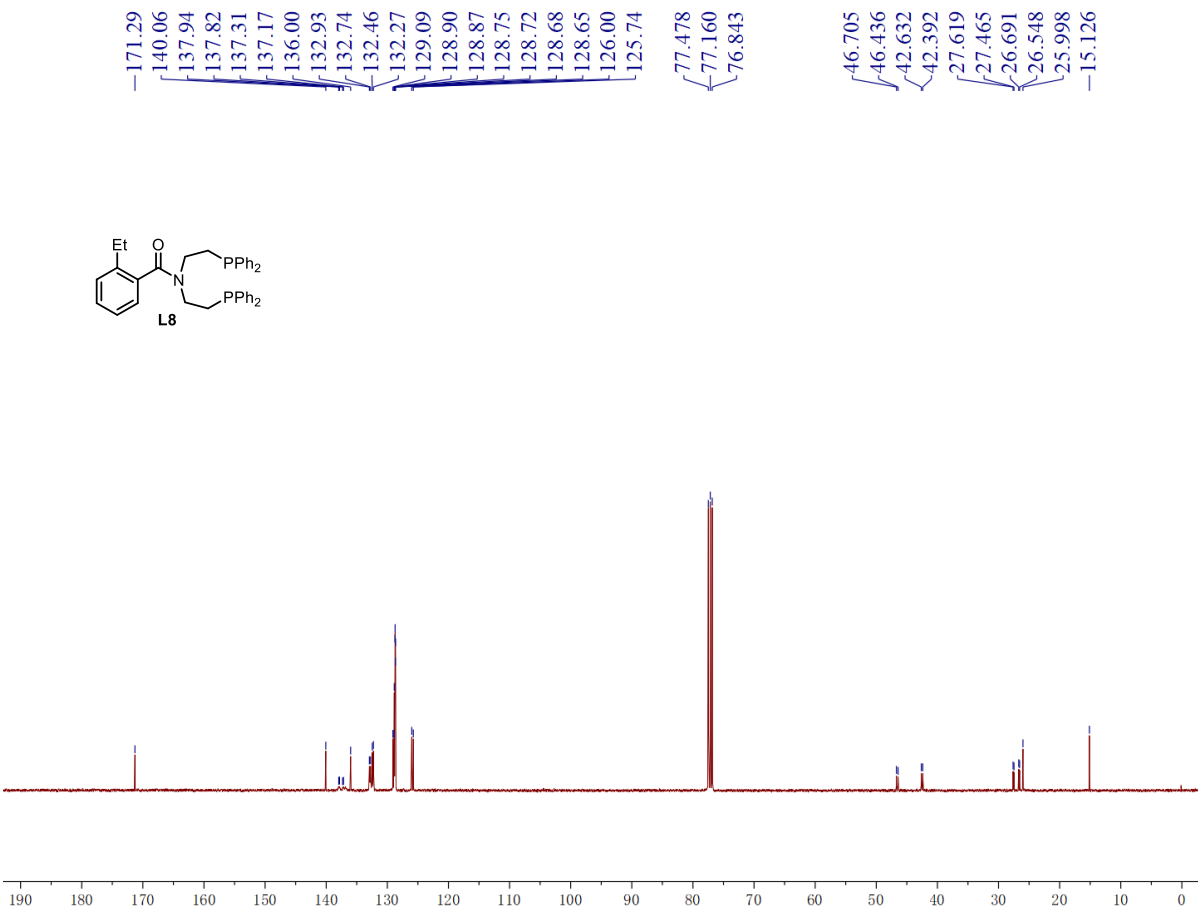
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PPh2
L8
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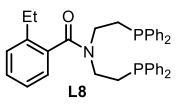
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PPh2
PPh2

L8

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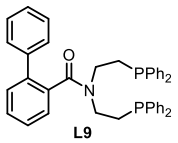
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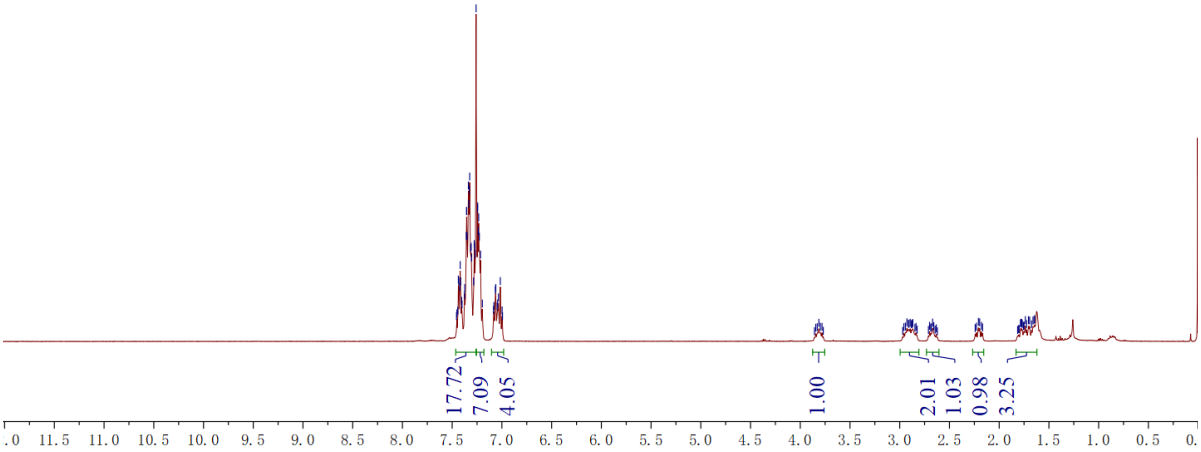
PPh2

L9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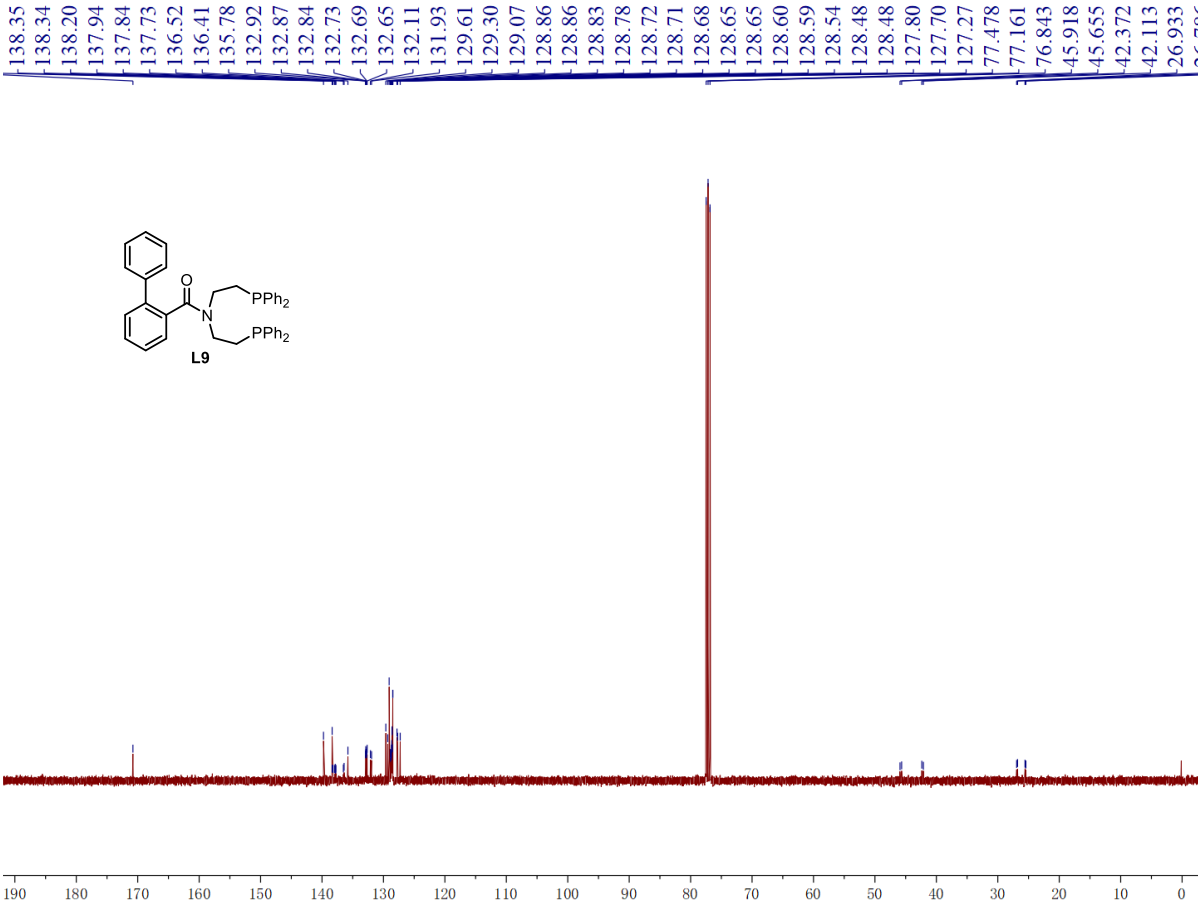
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45.655
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42.113
26.933
O
N
PPh2
PPh2
L9
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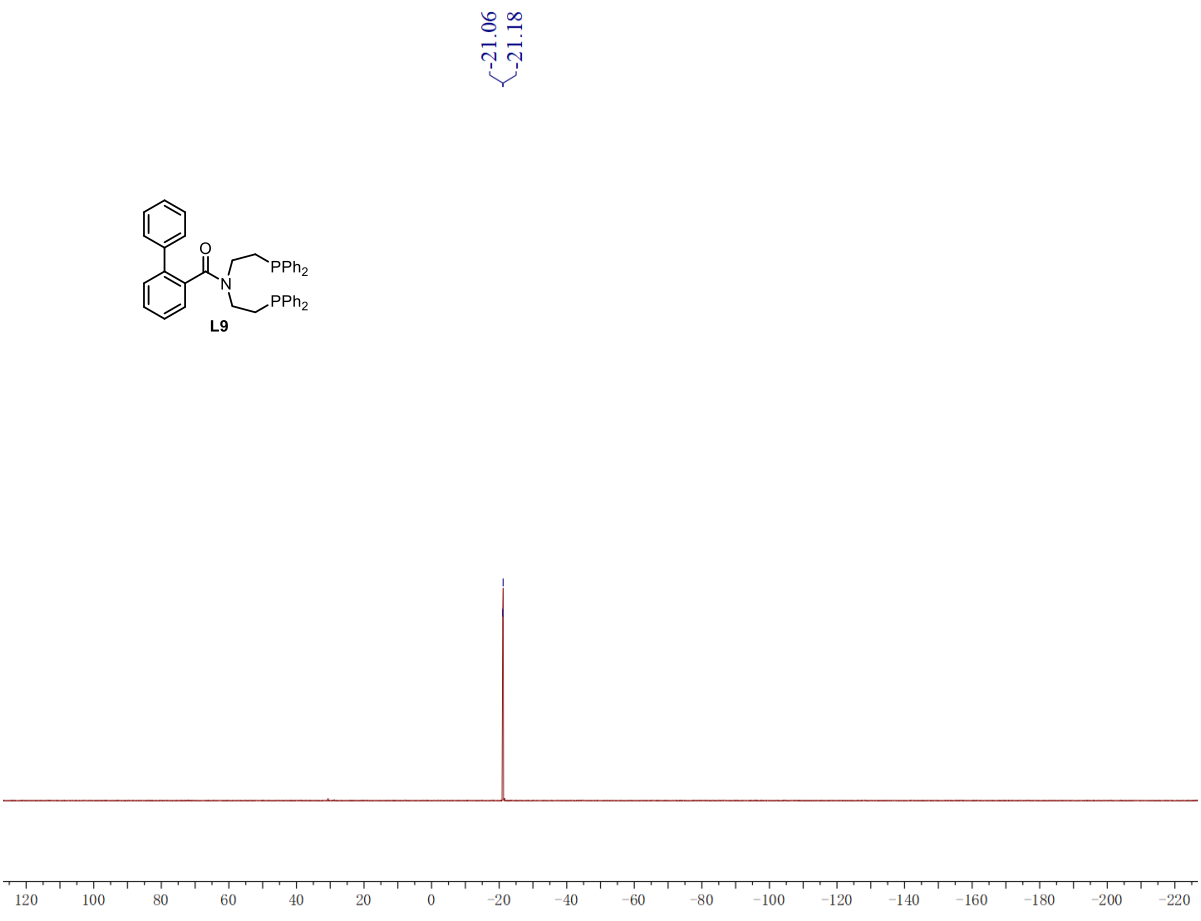
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L9
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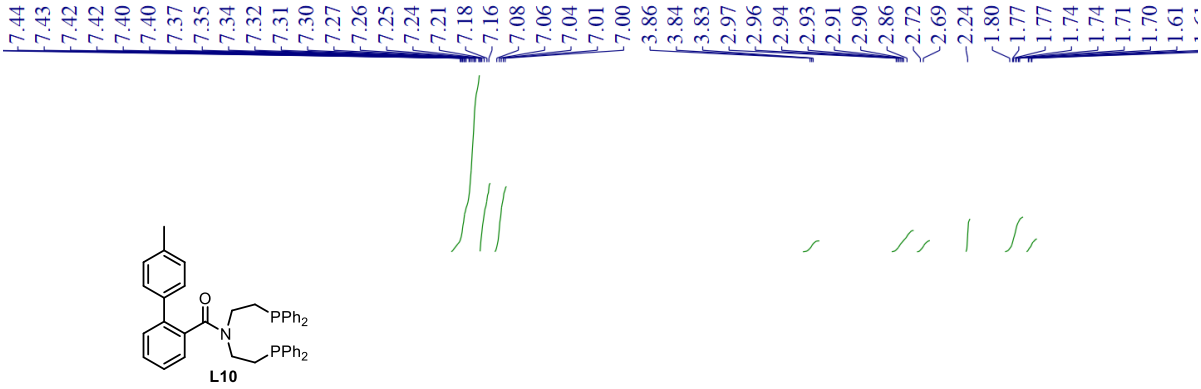
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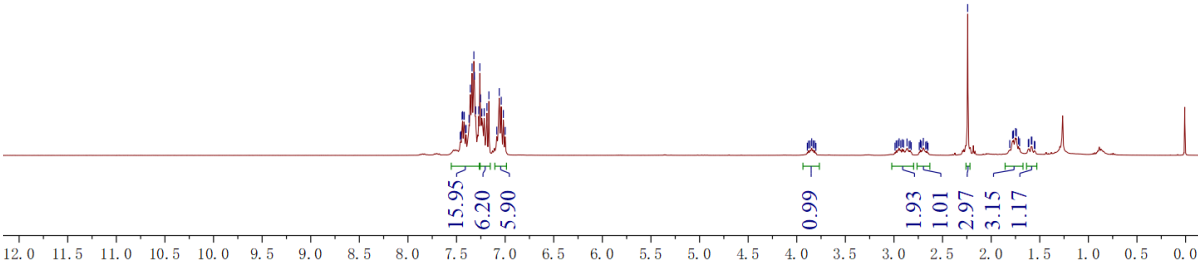
PPh2
PPh2

L10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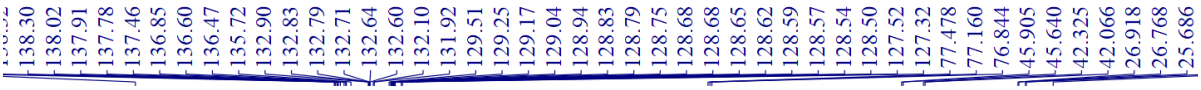
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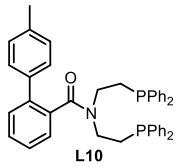
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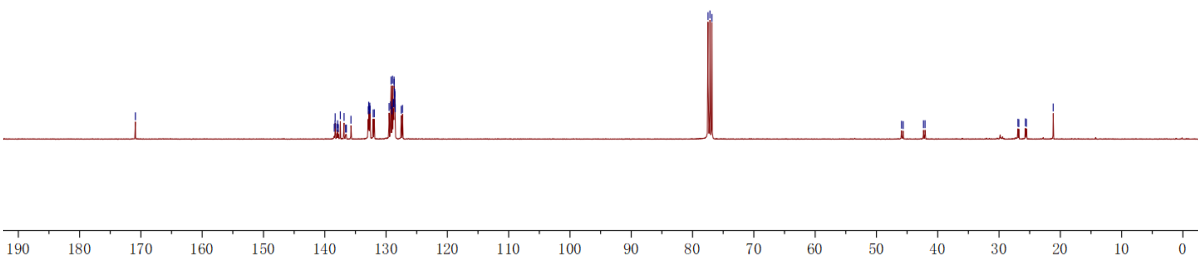
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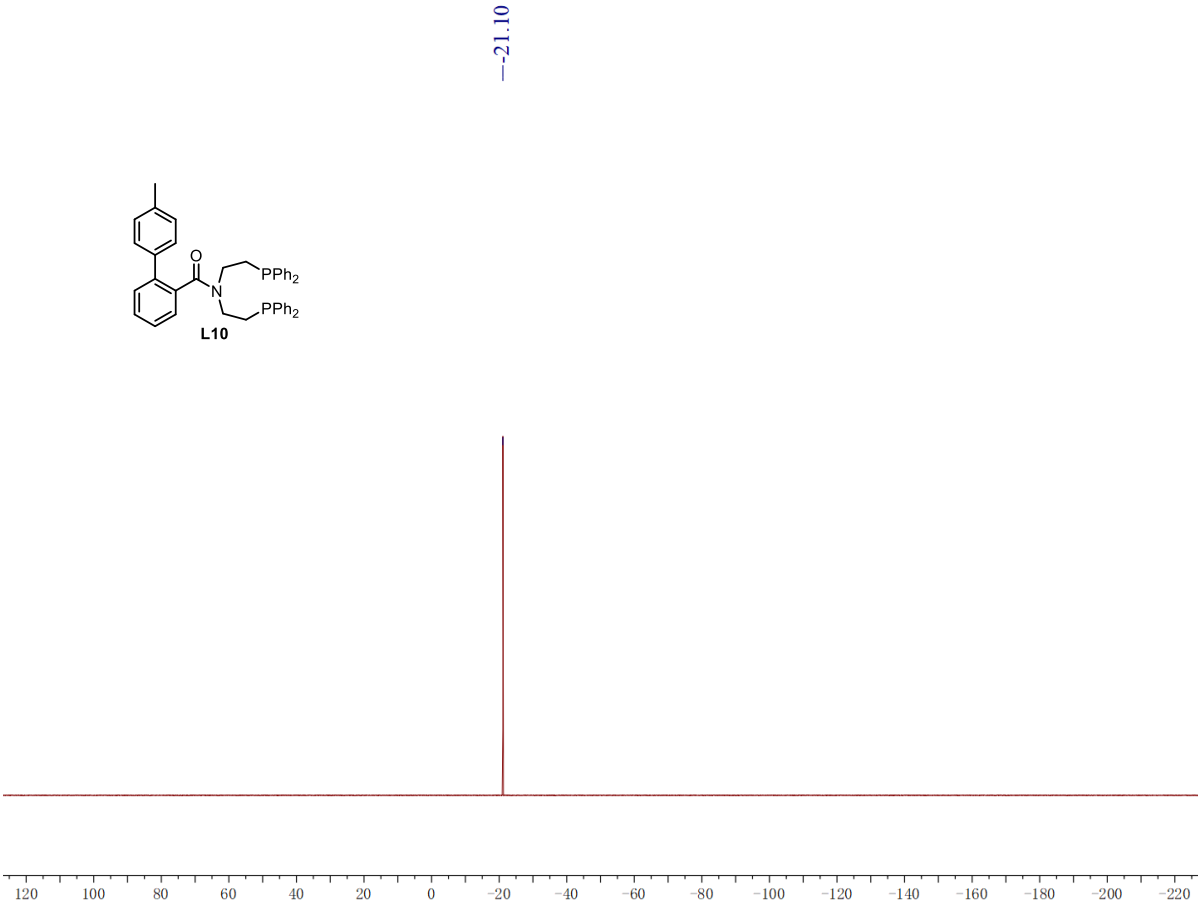
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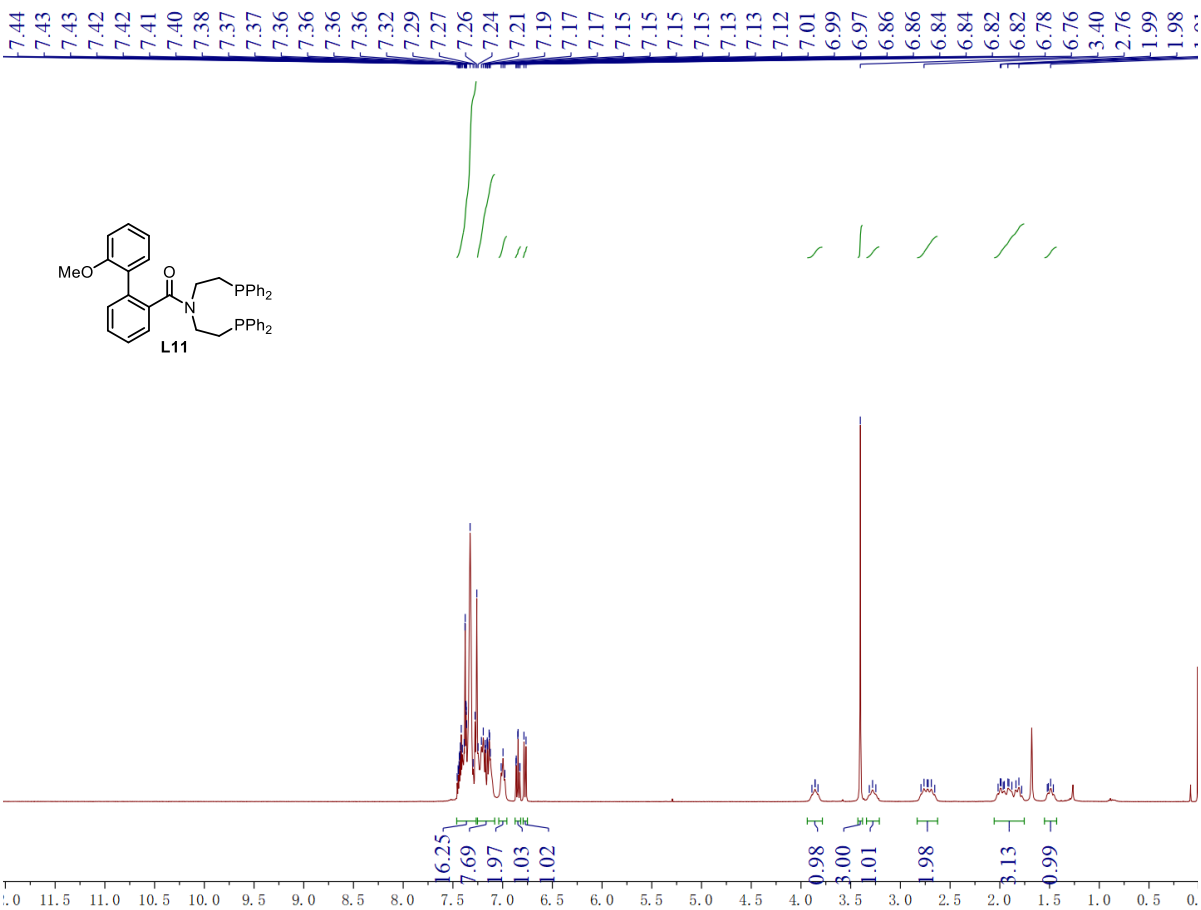
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PPh2
PPh2
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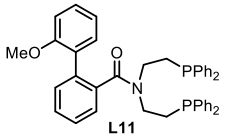

L11

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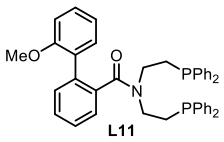
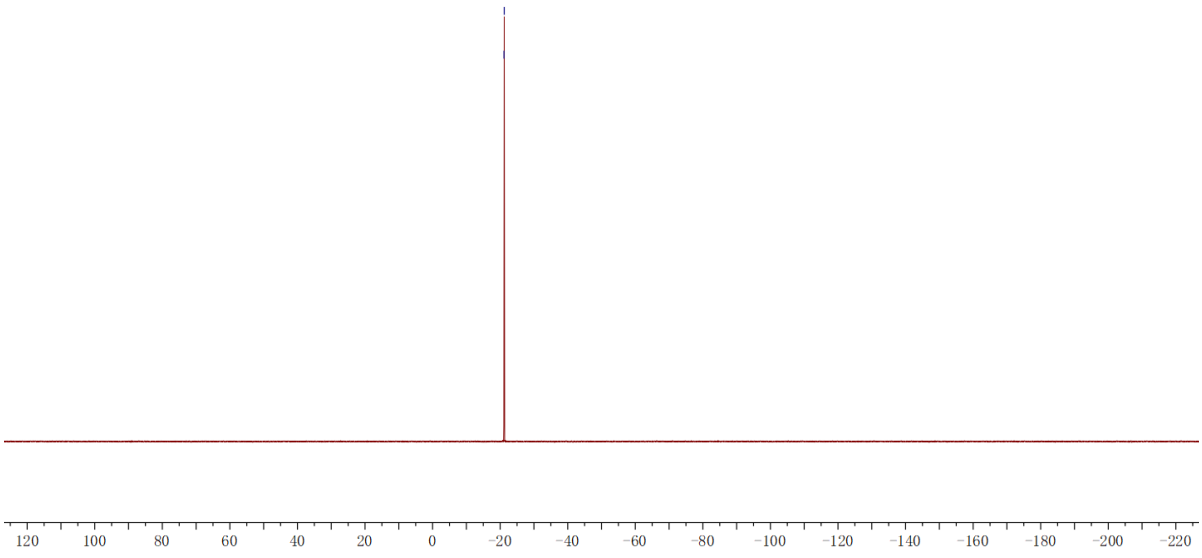


L11



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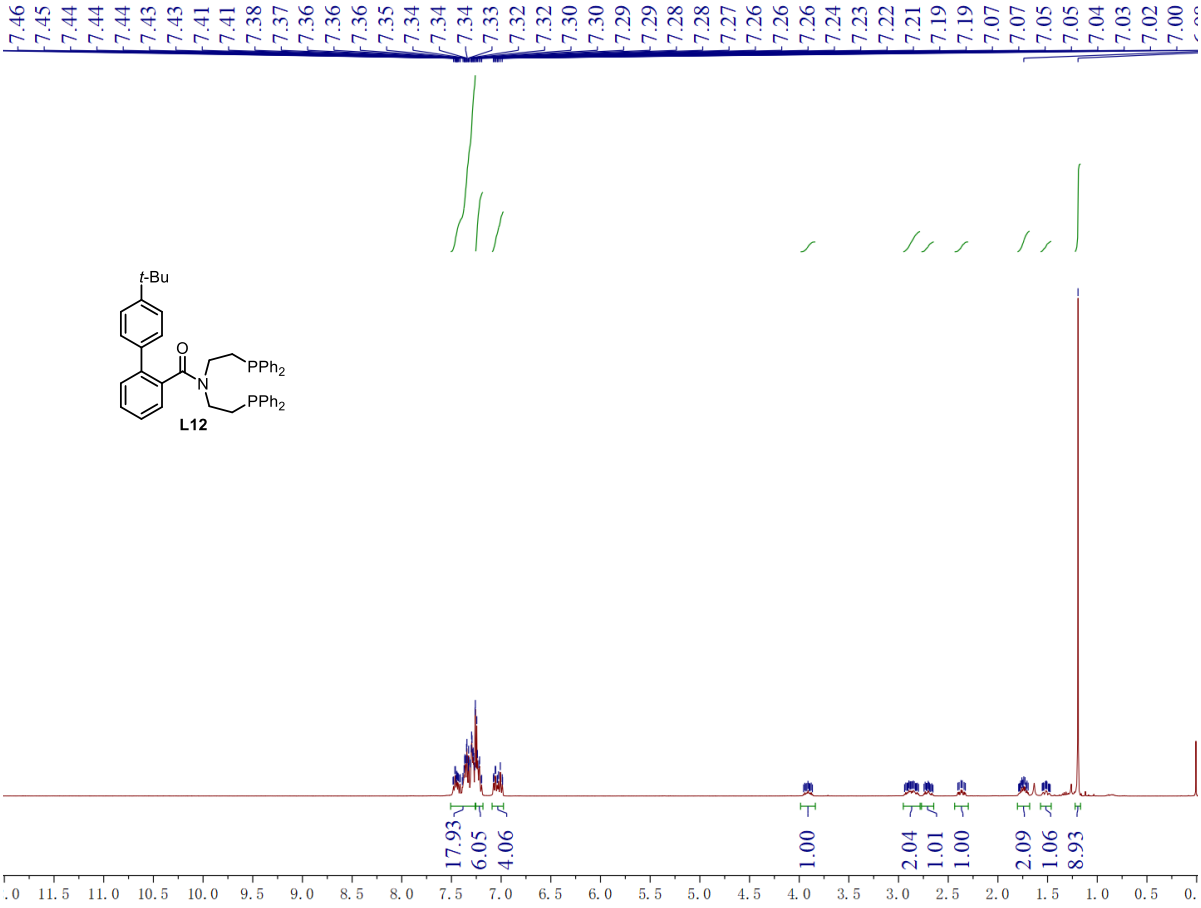
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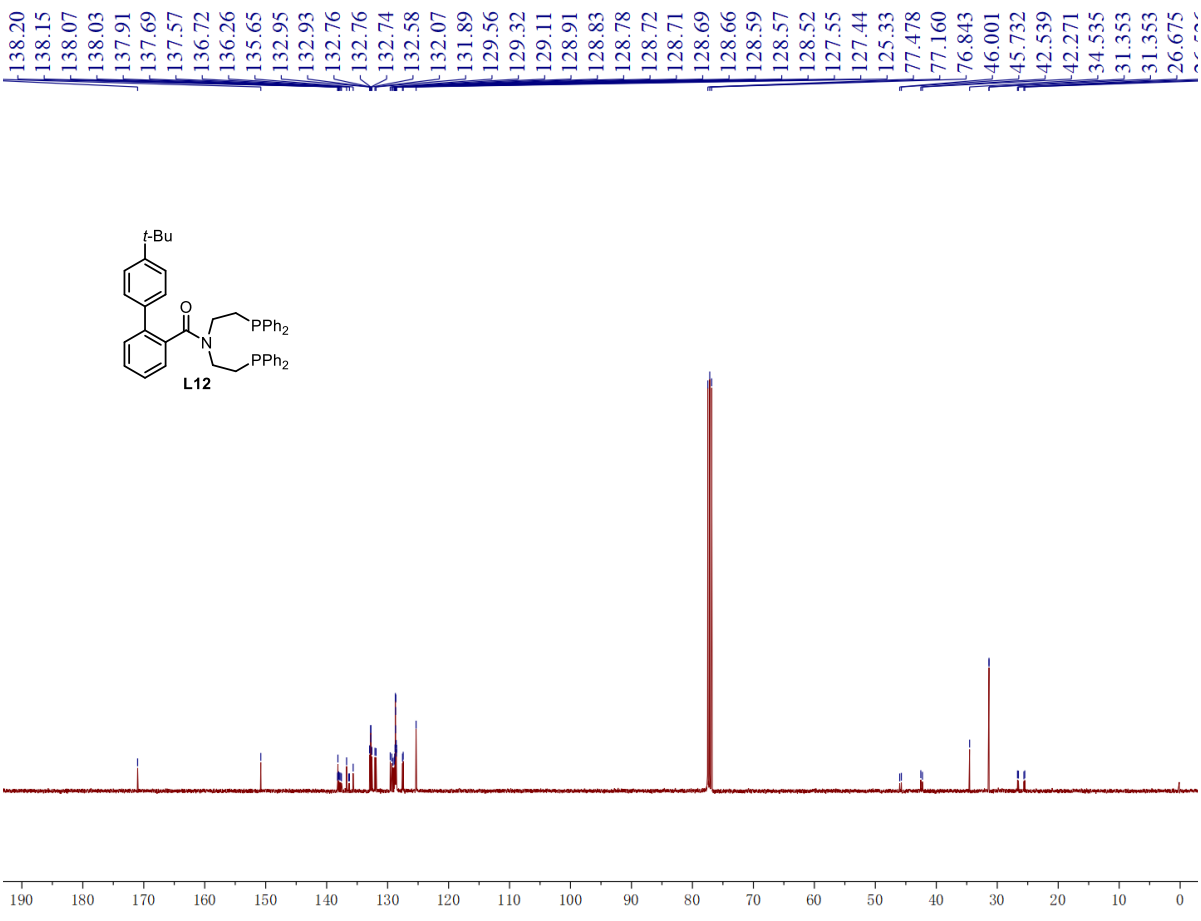
t-Bu
O
N
PPh2
PPh2
L12
17.93 6.05 4.06 1.00 2.04 1.01 1.00 2.09 1.06 8.93
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.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PPh2
PPh2
L12
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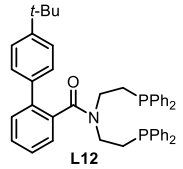
-21.12



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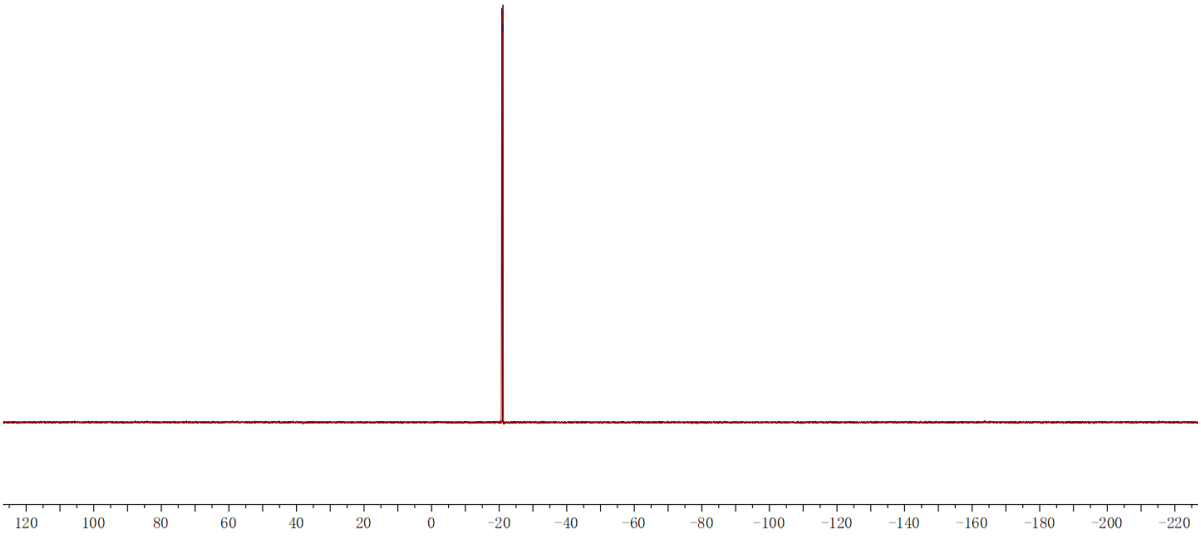

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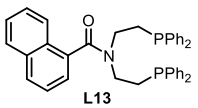

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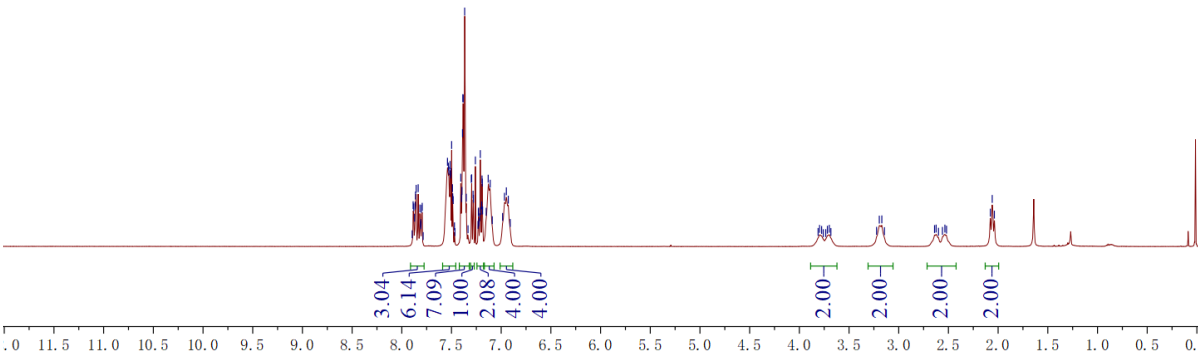

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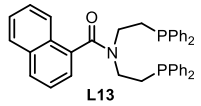

L13

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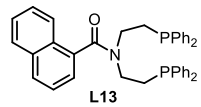

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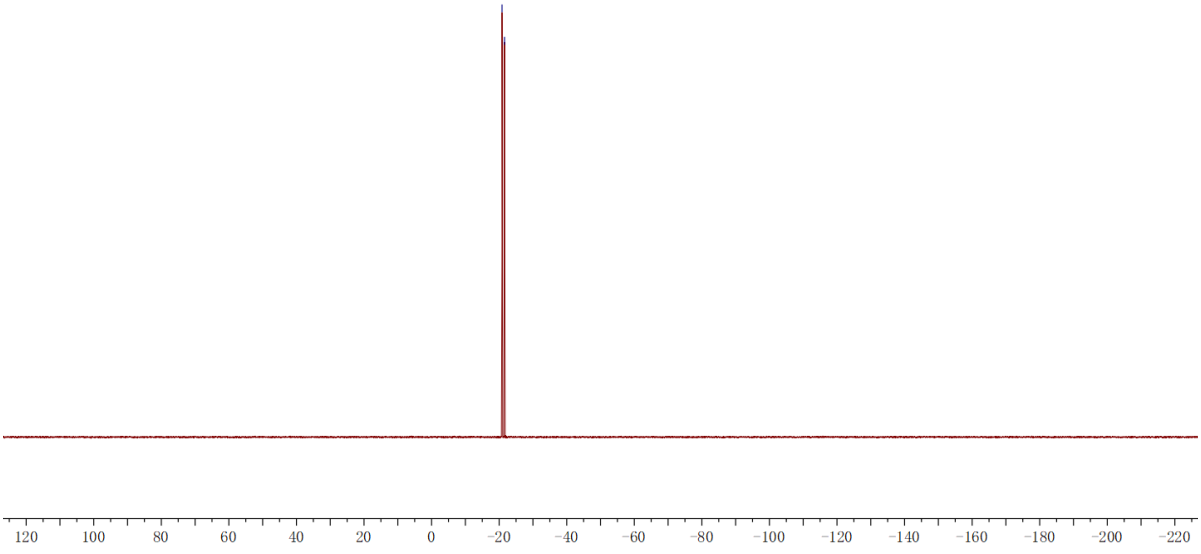

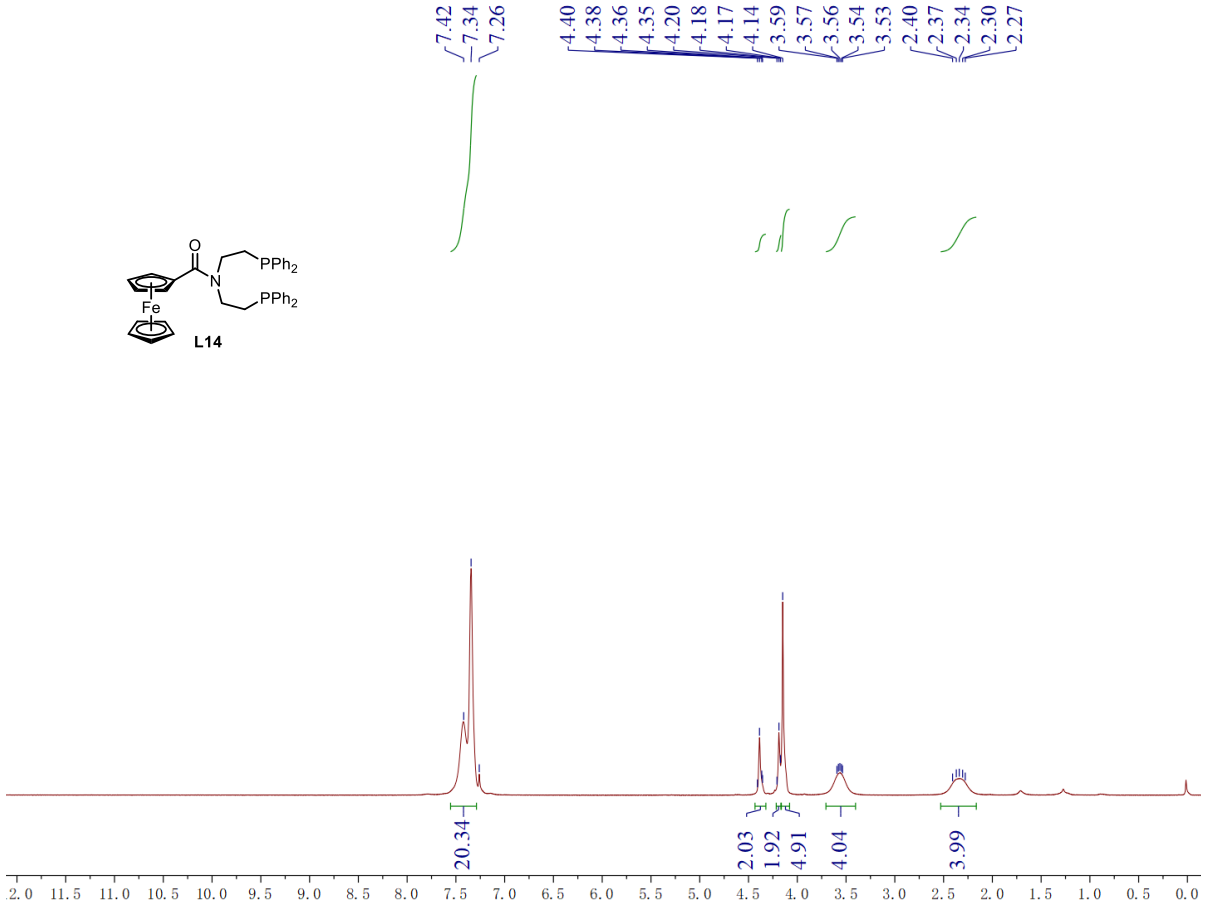
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PPh2
PPh2
Fe
L14
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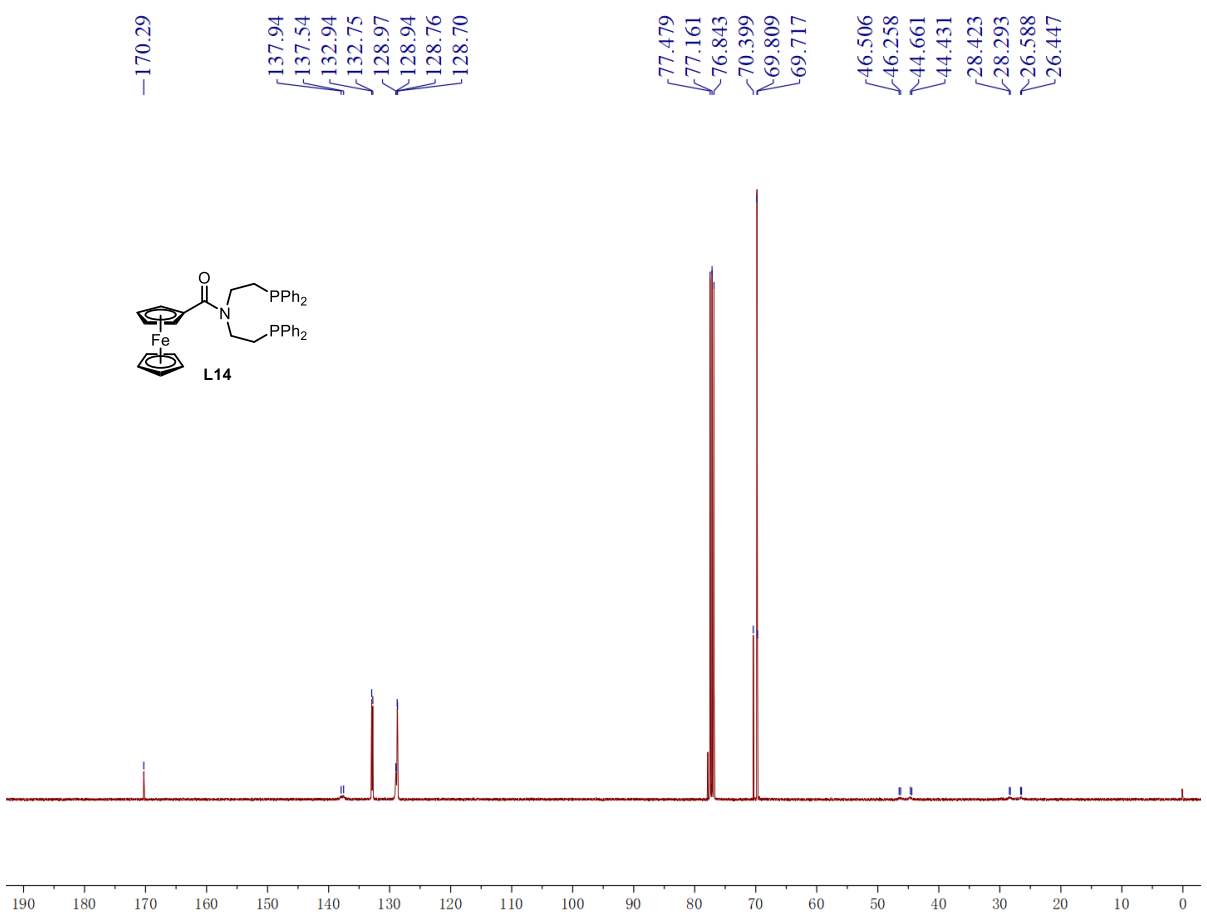
4.04
3.99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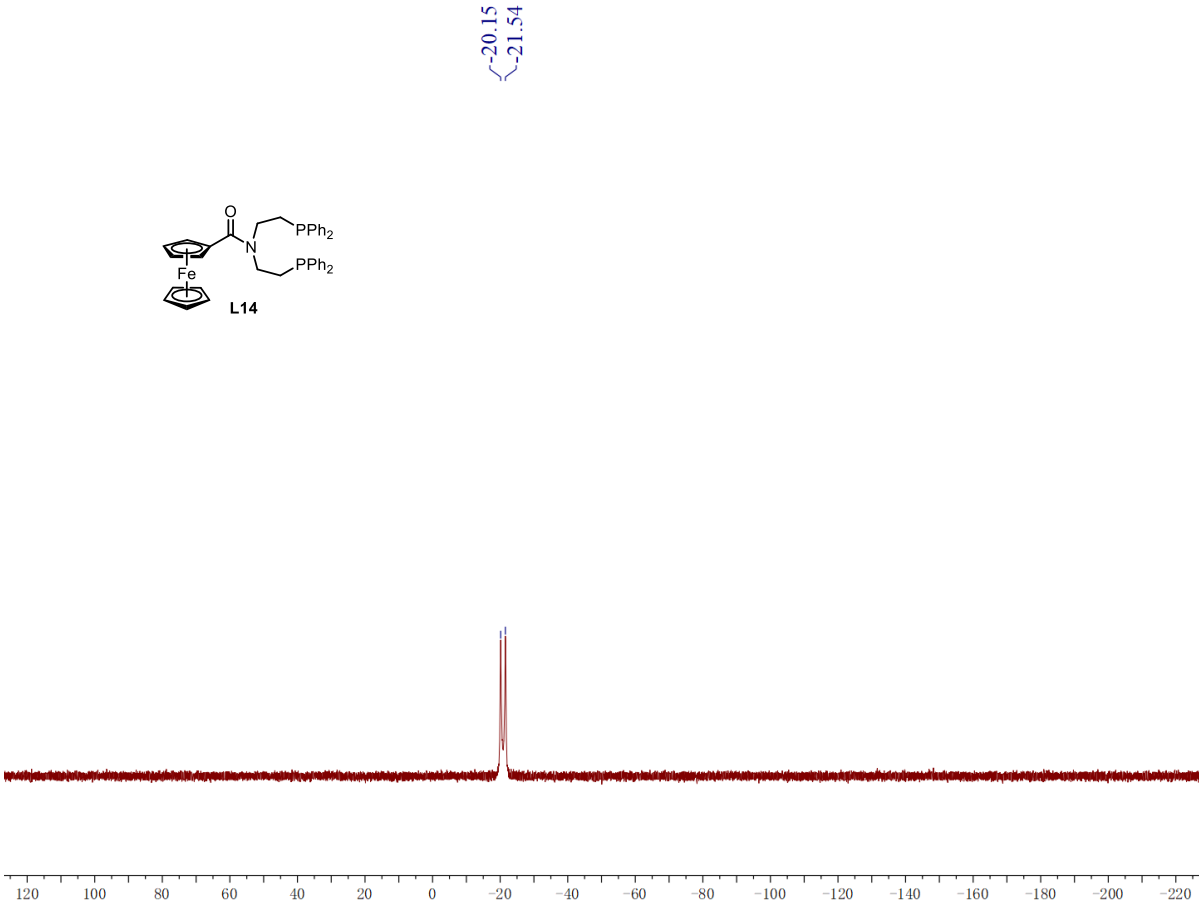
26.447
PPh2
PPh2
Fe
L14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PPh2
Fe
L14
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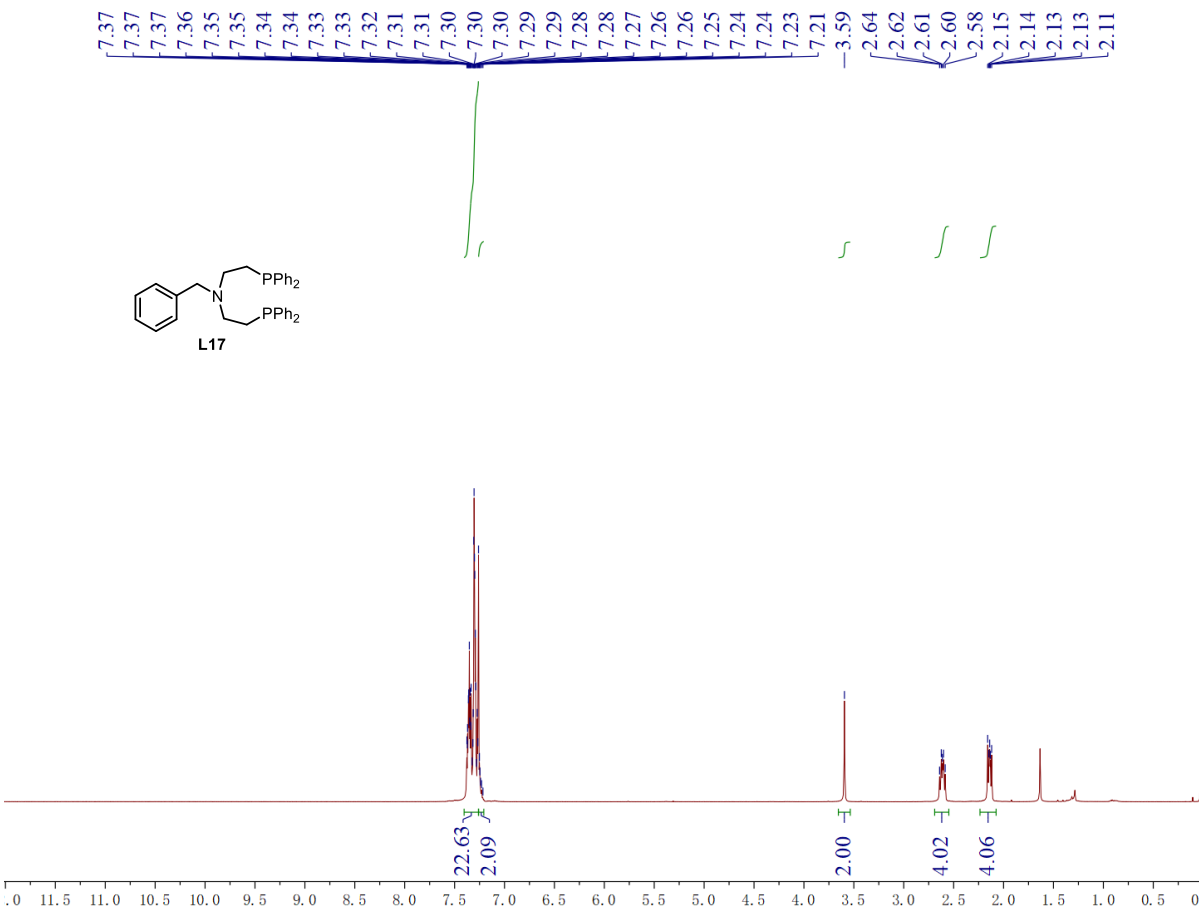
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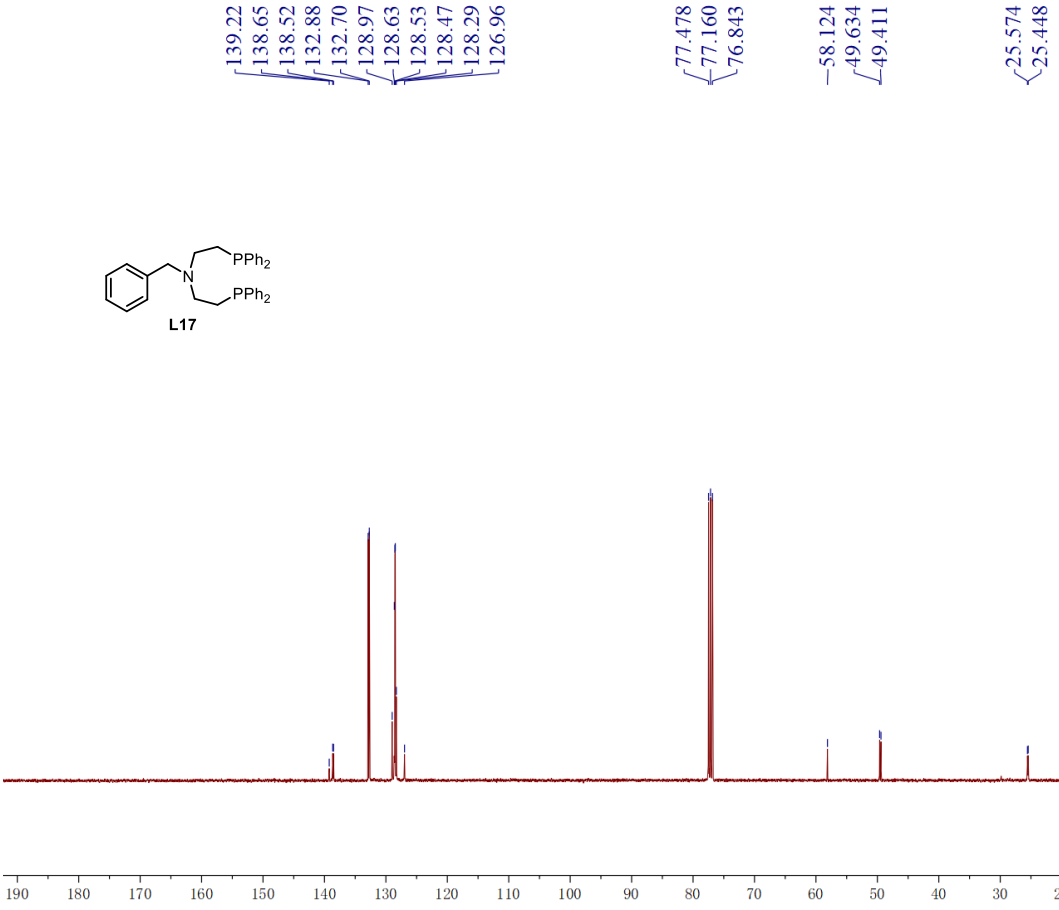
N
PPh2
PPh2
L17
22.63 2.09 2.00 4.02 4.06
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0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L17
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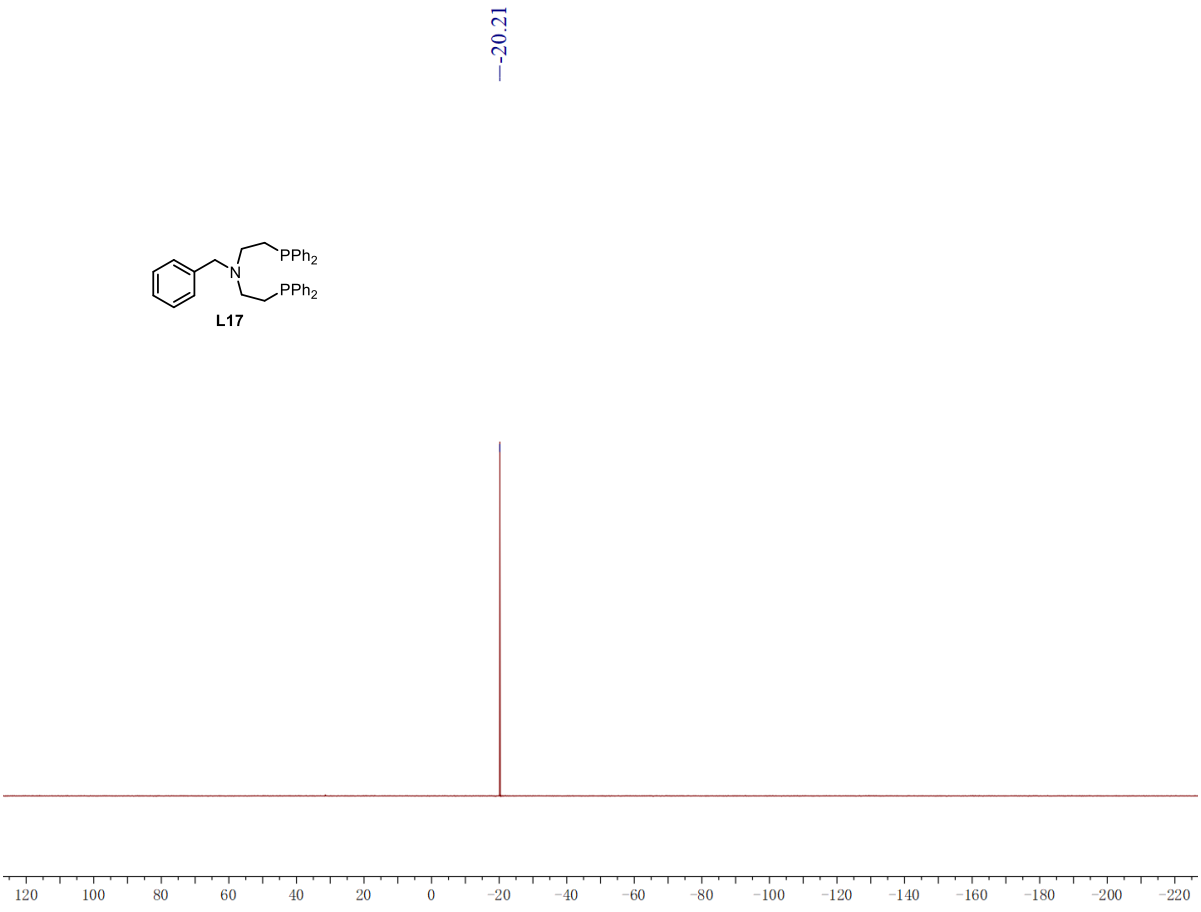
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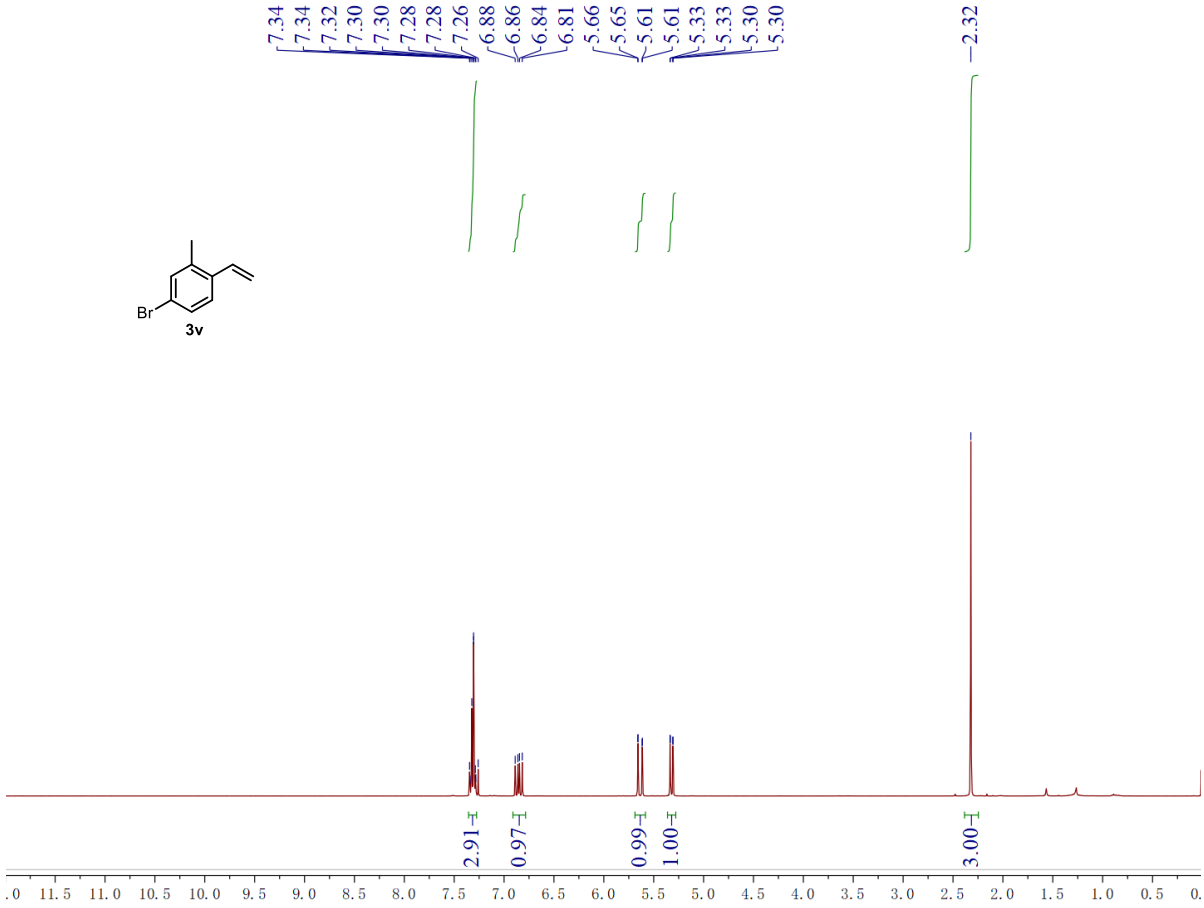
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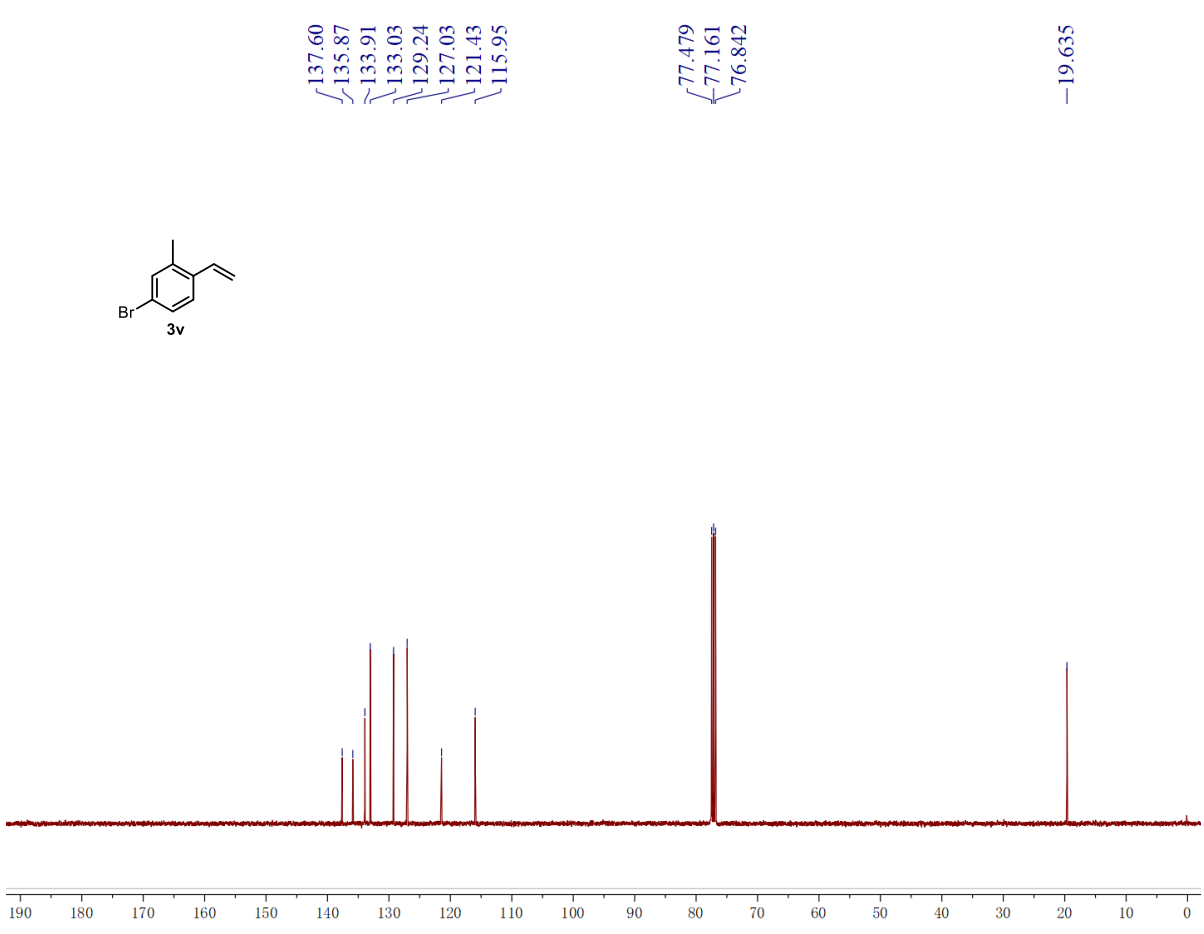
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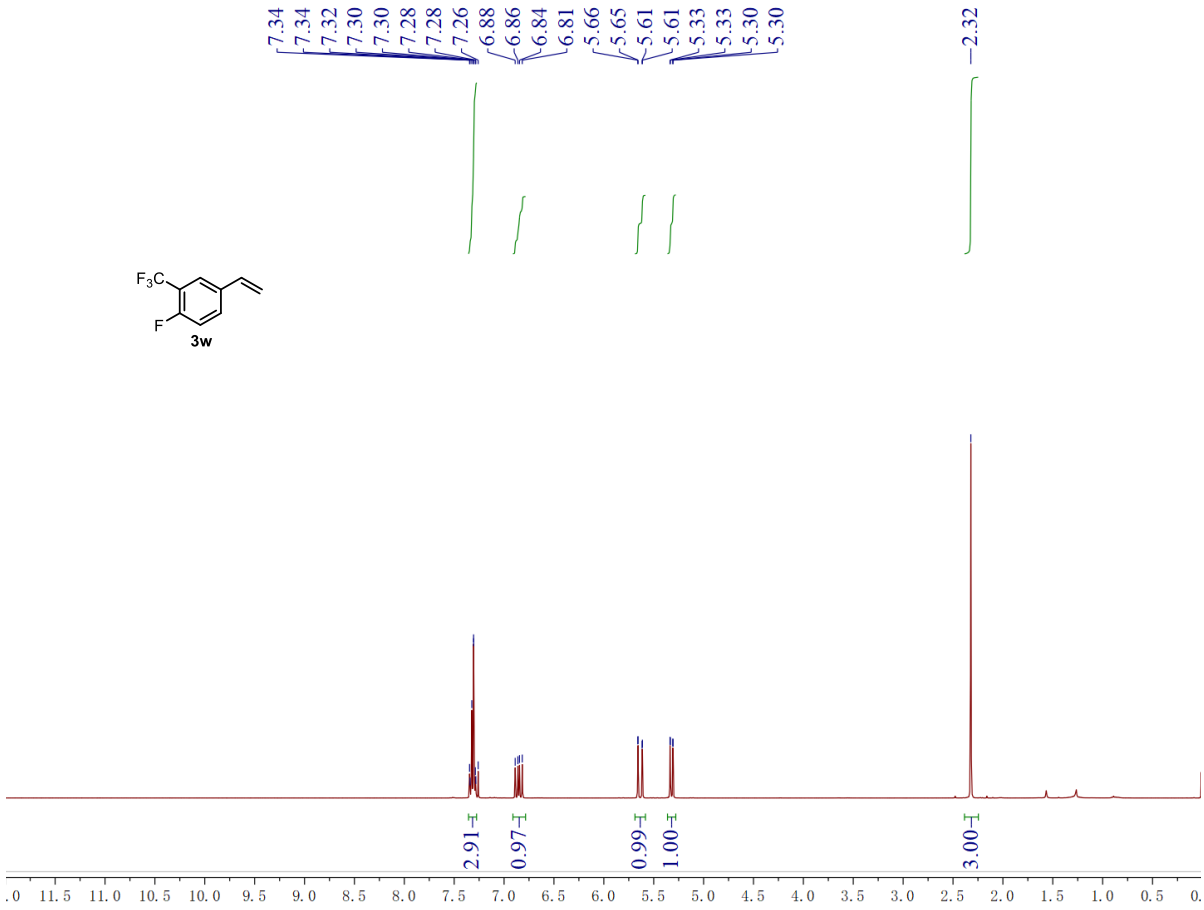
Br
3v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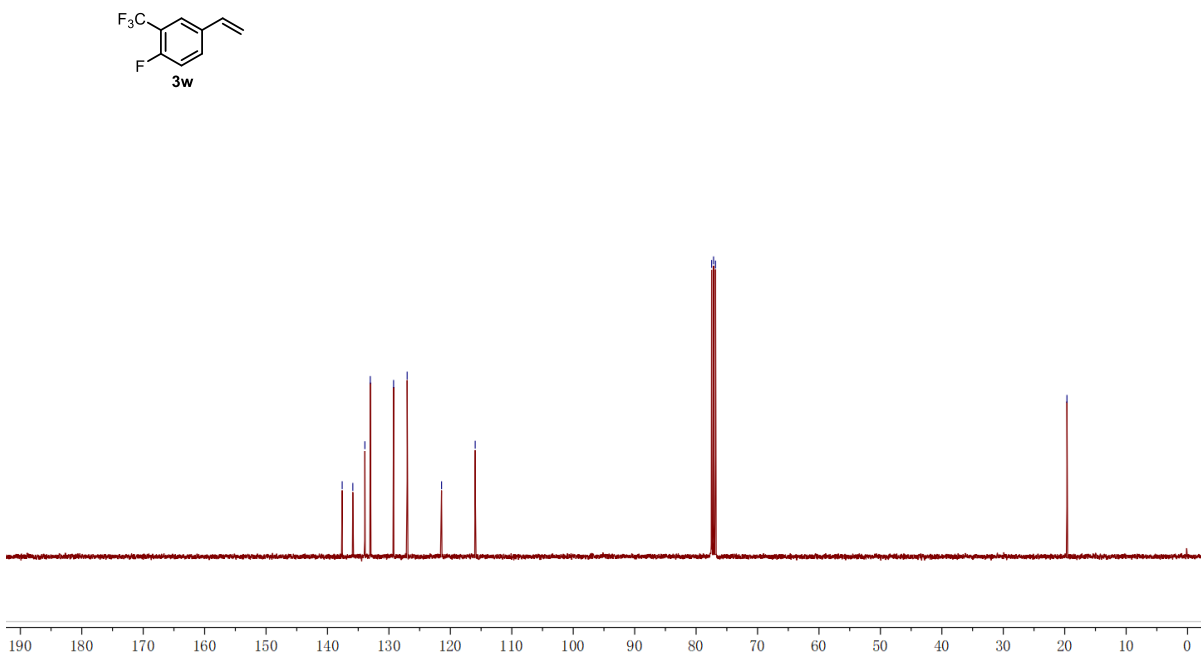
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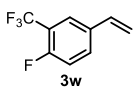
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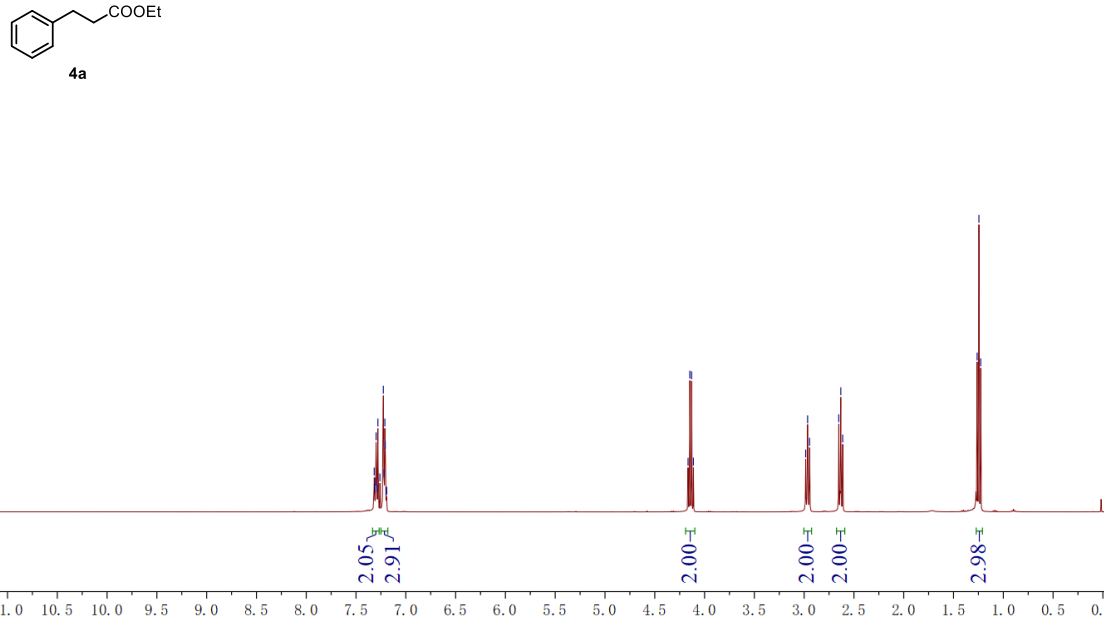
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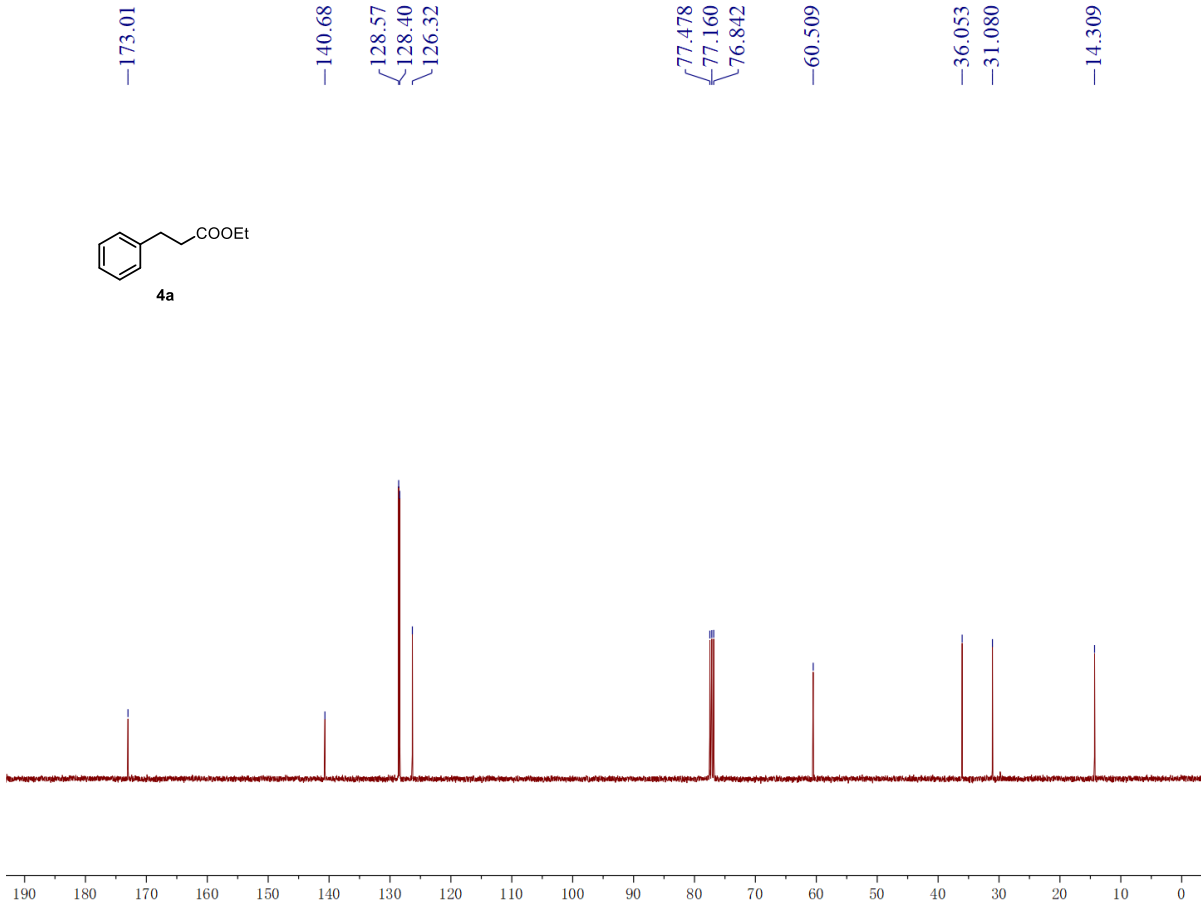
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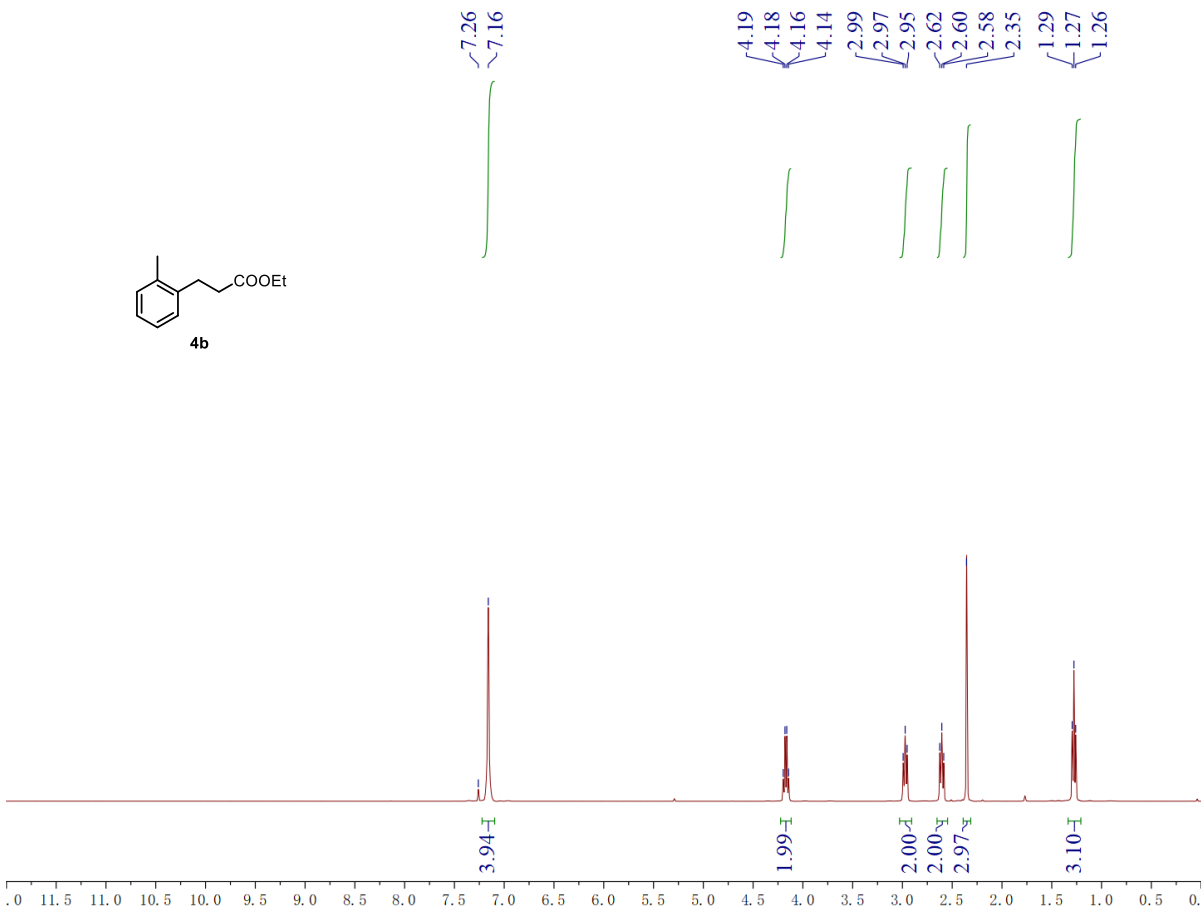
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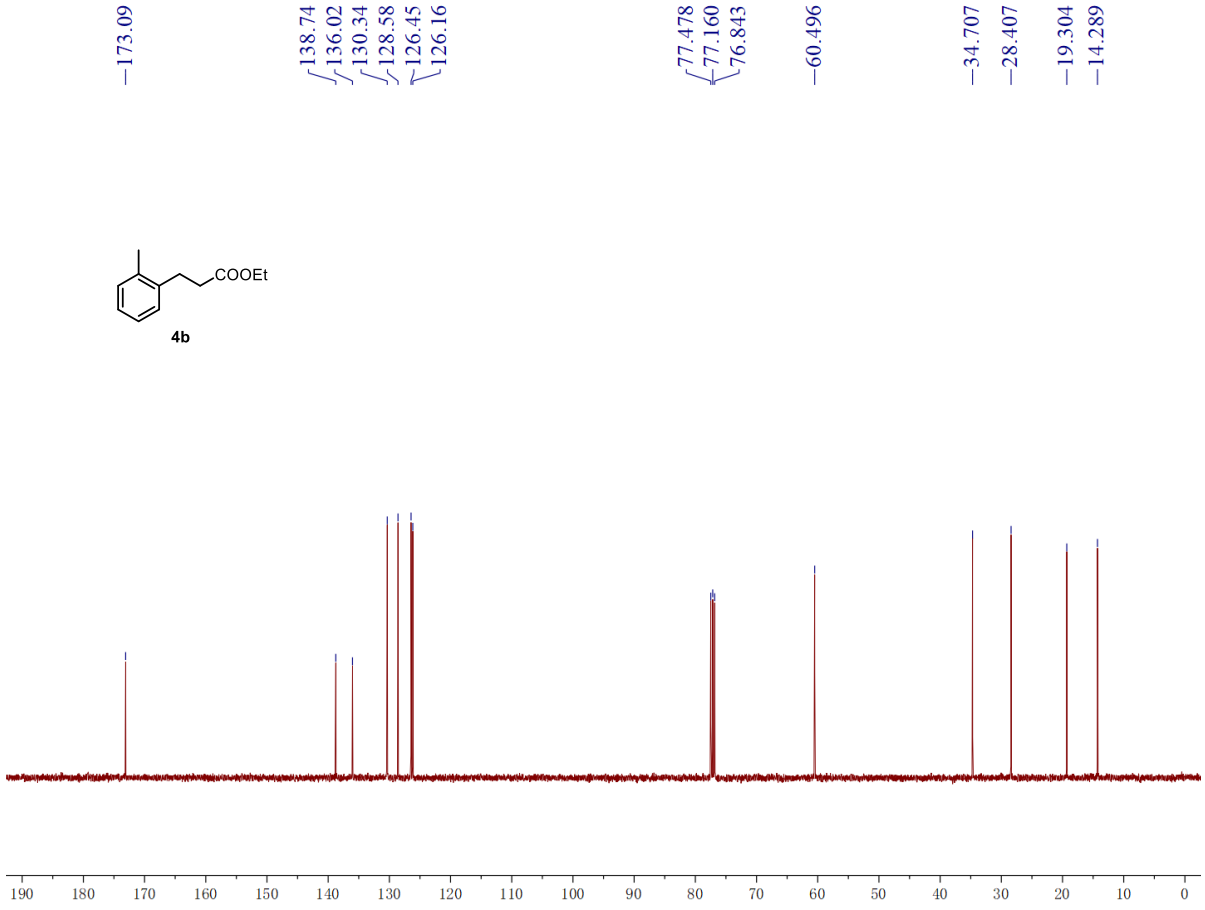
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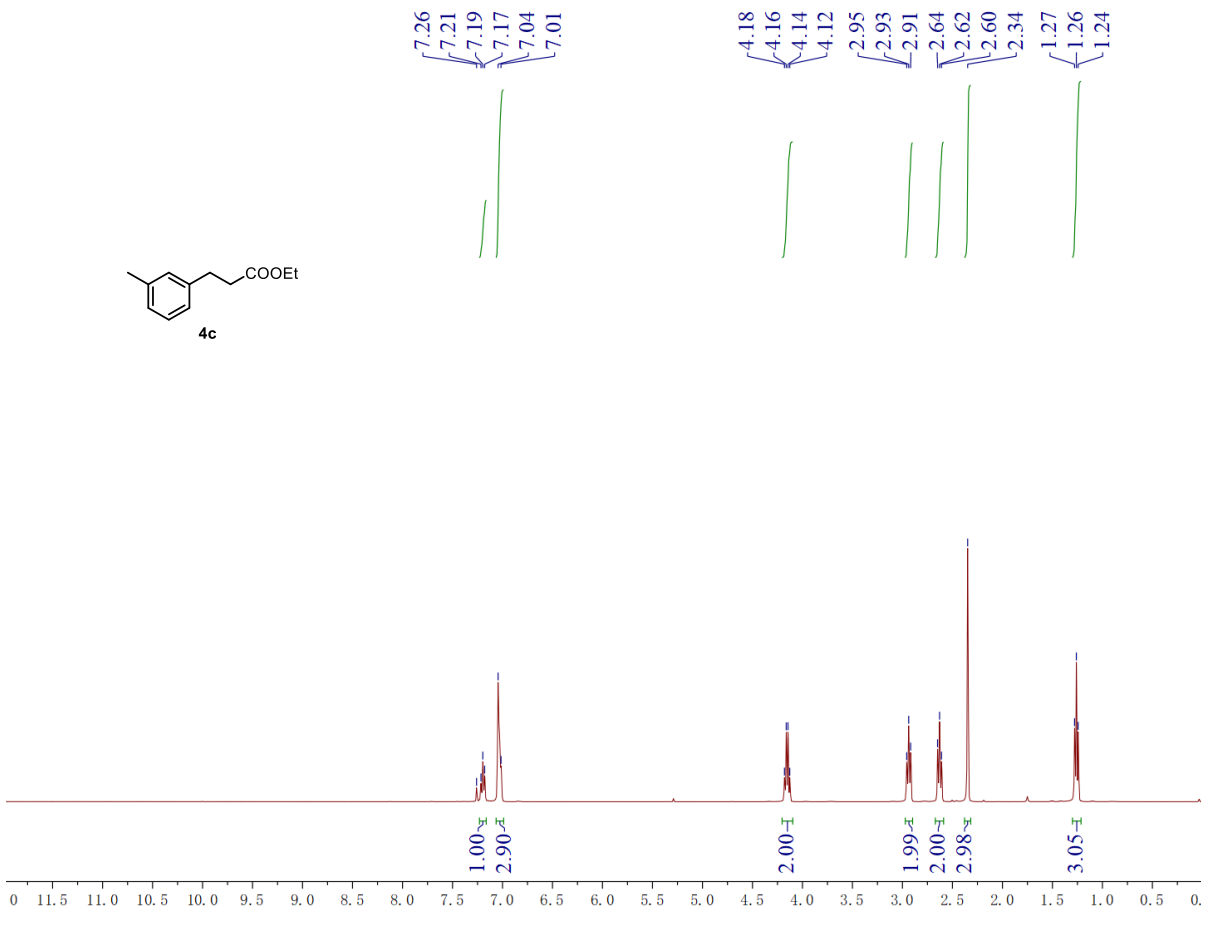
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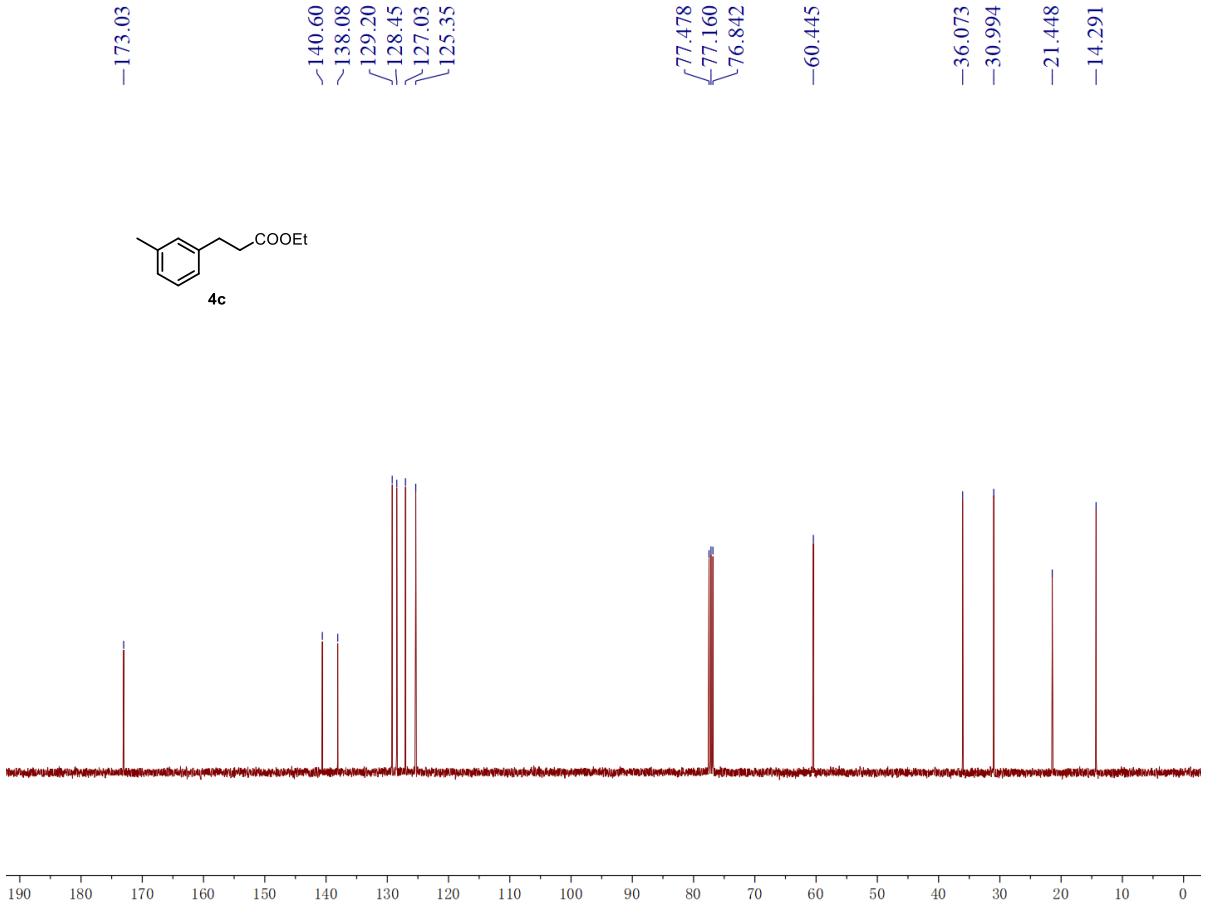
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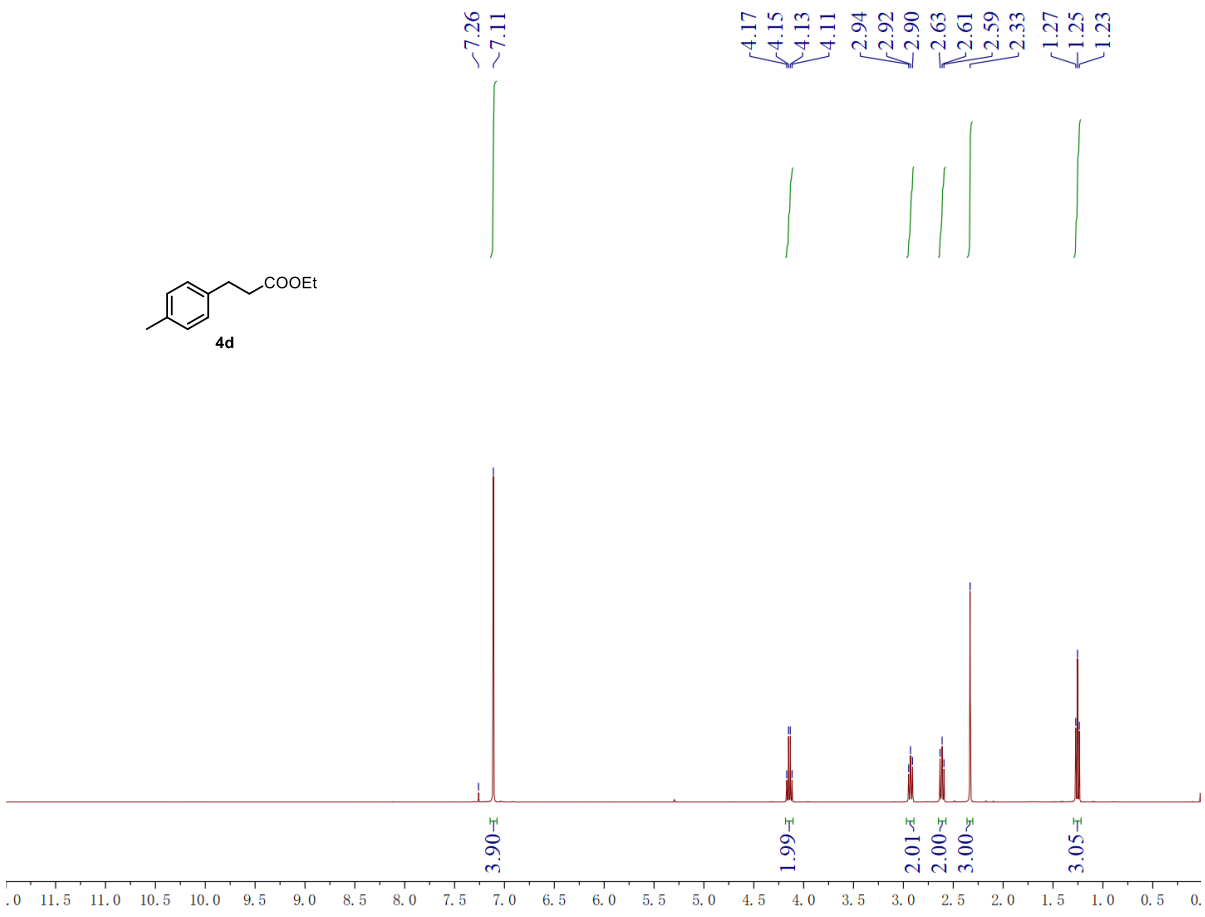
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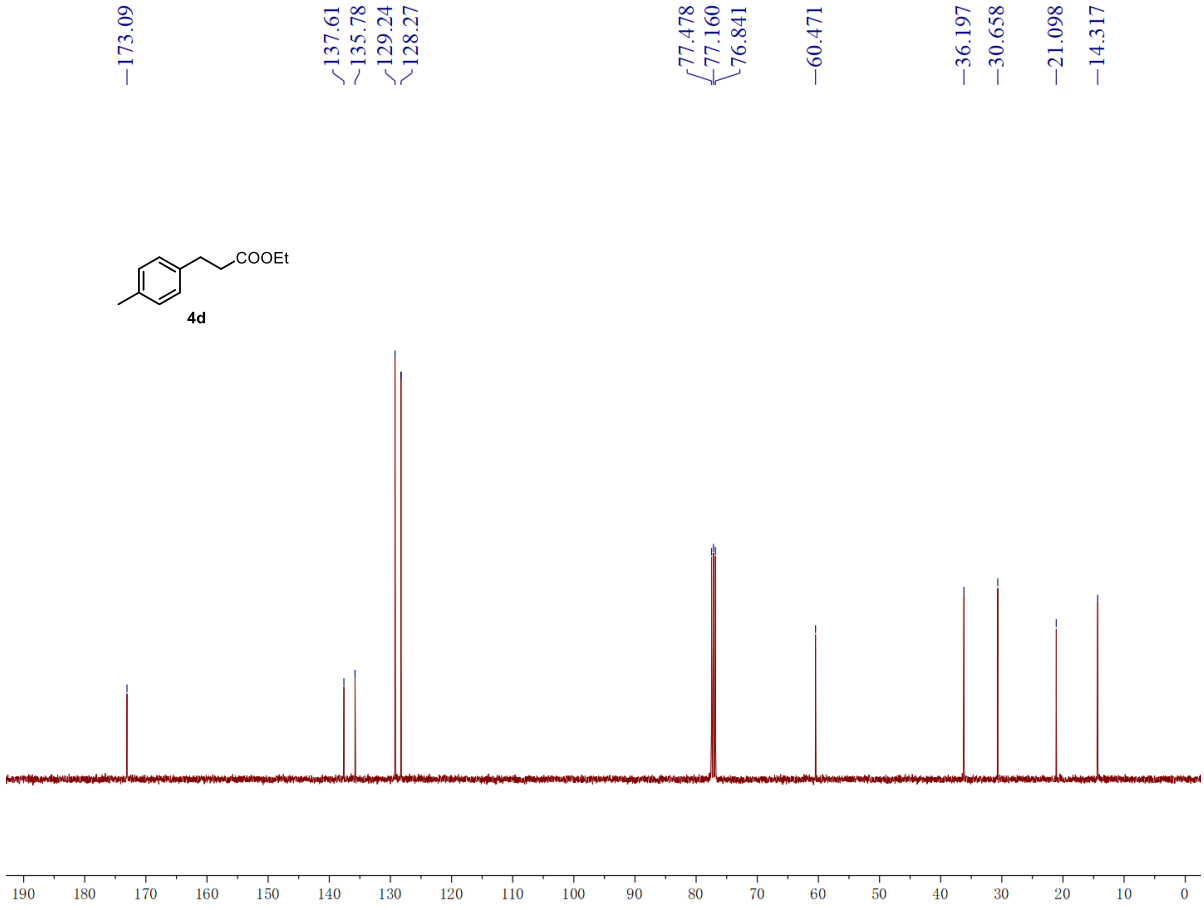
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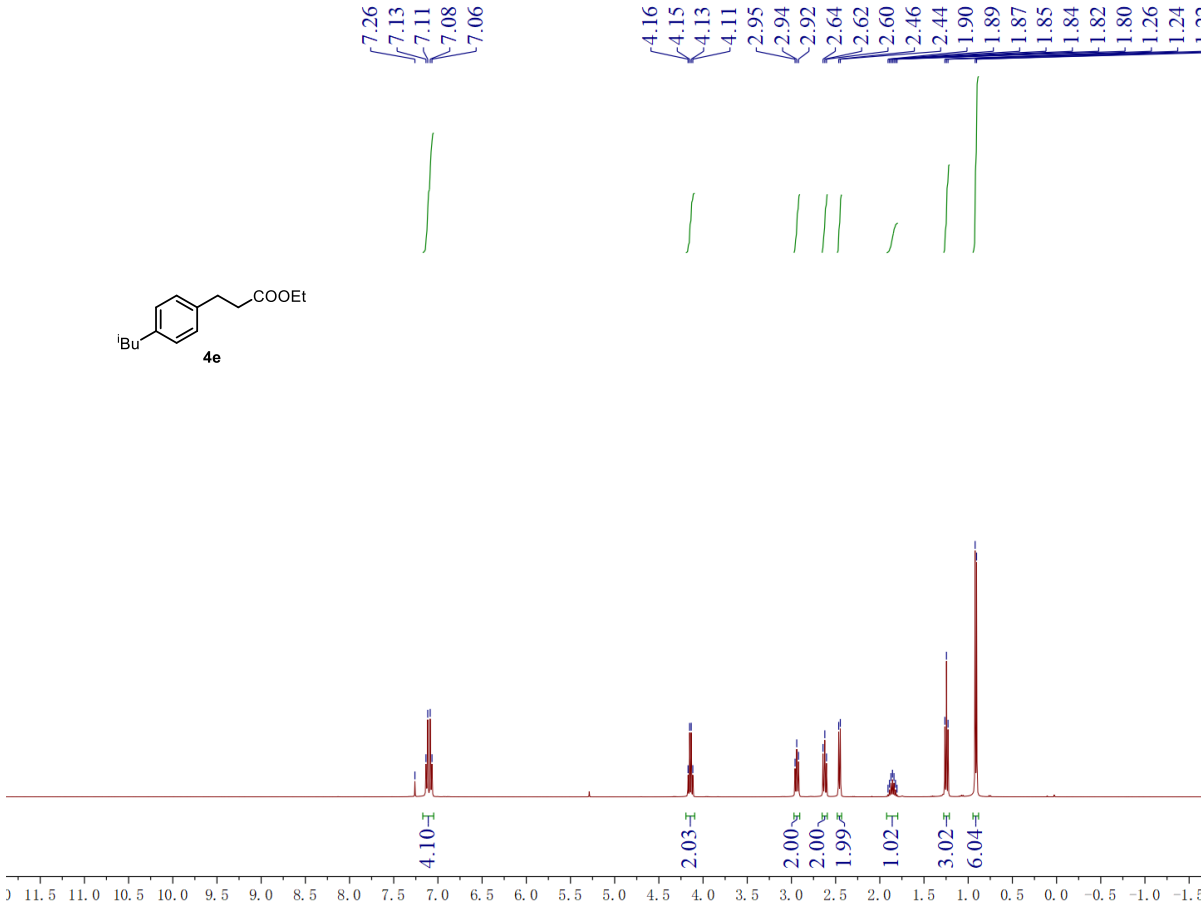
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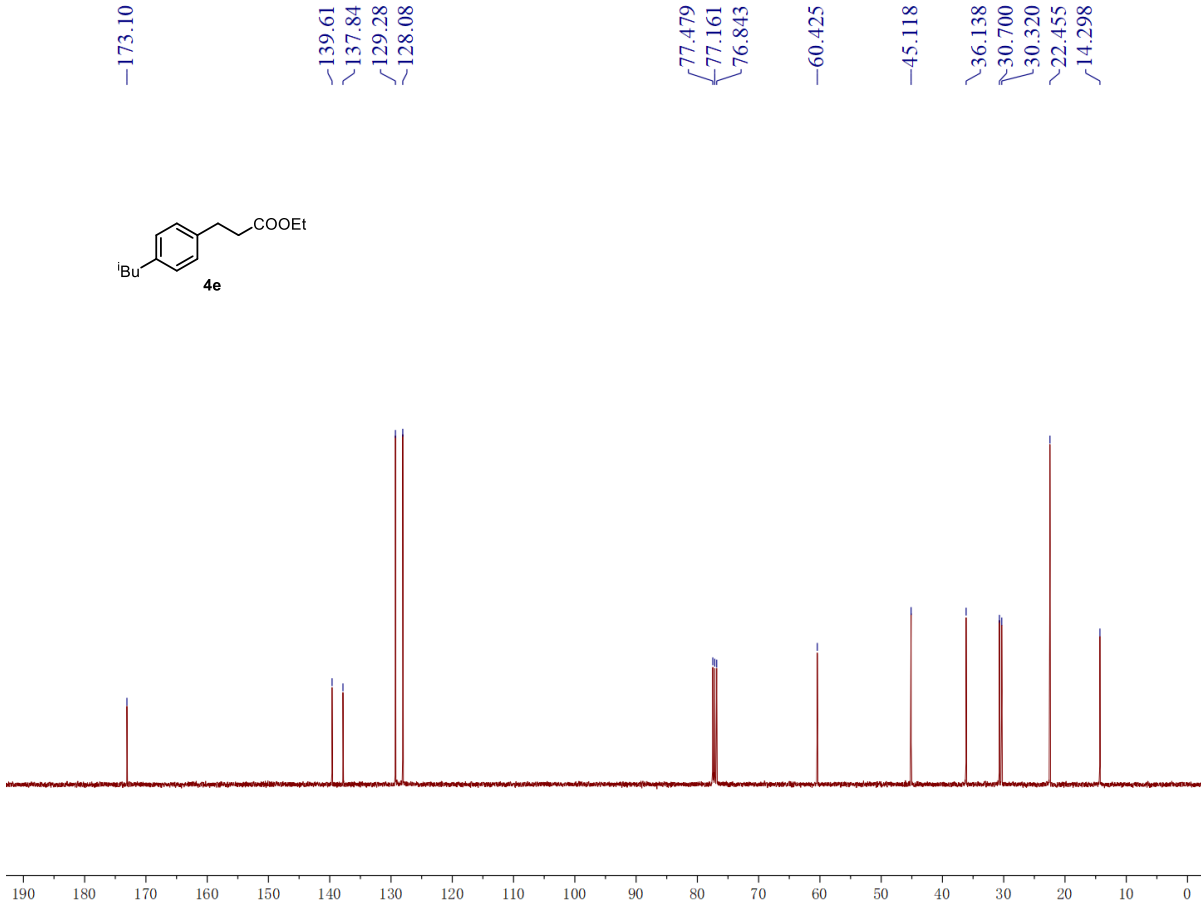
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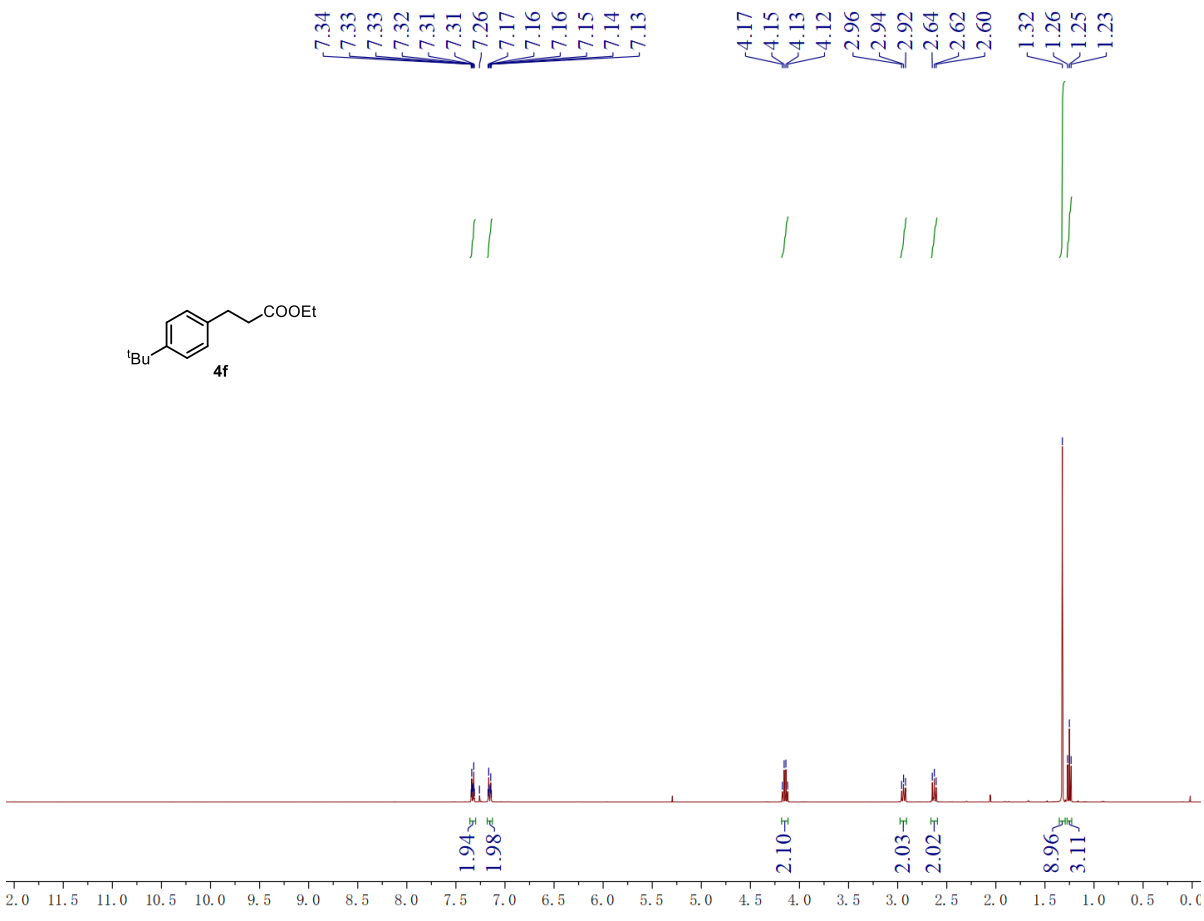
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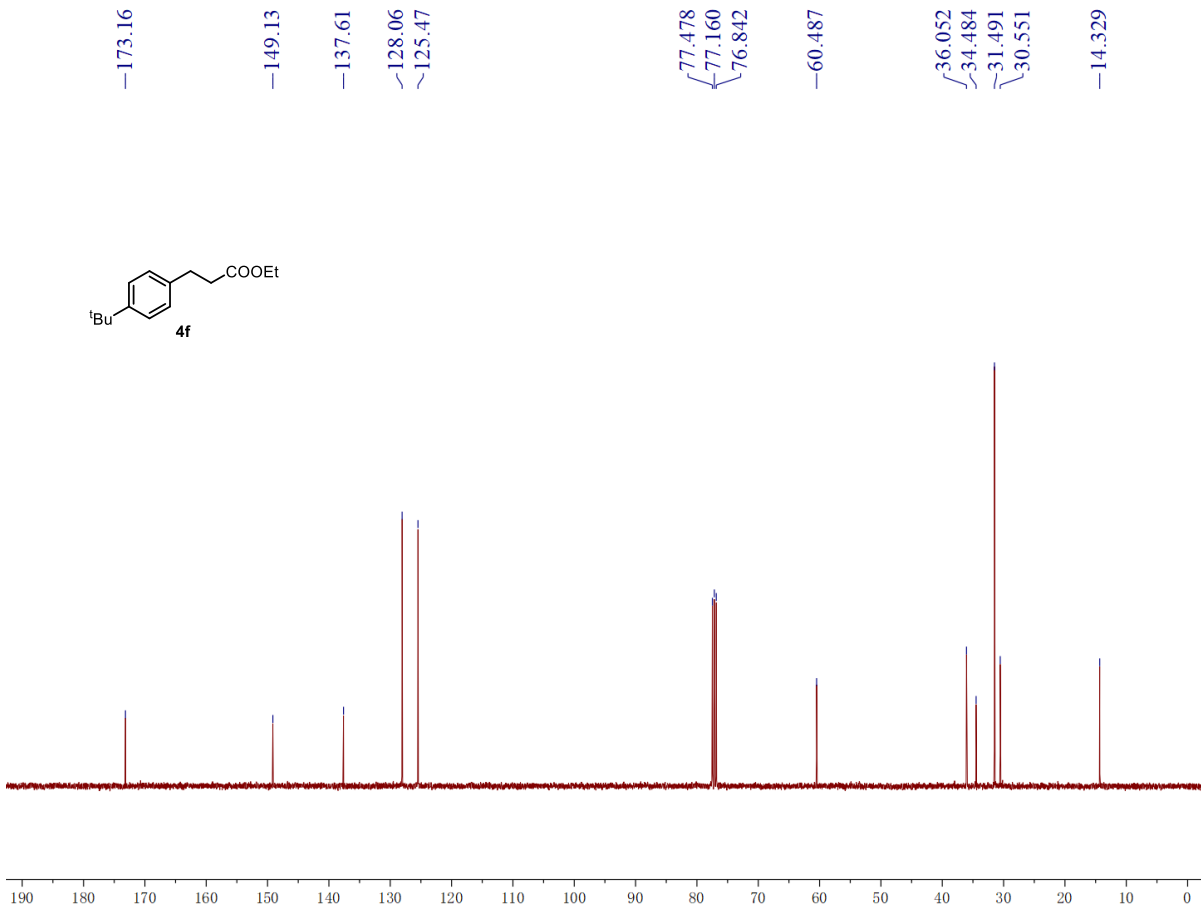
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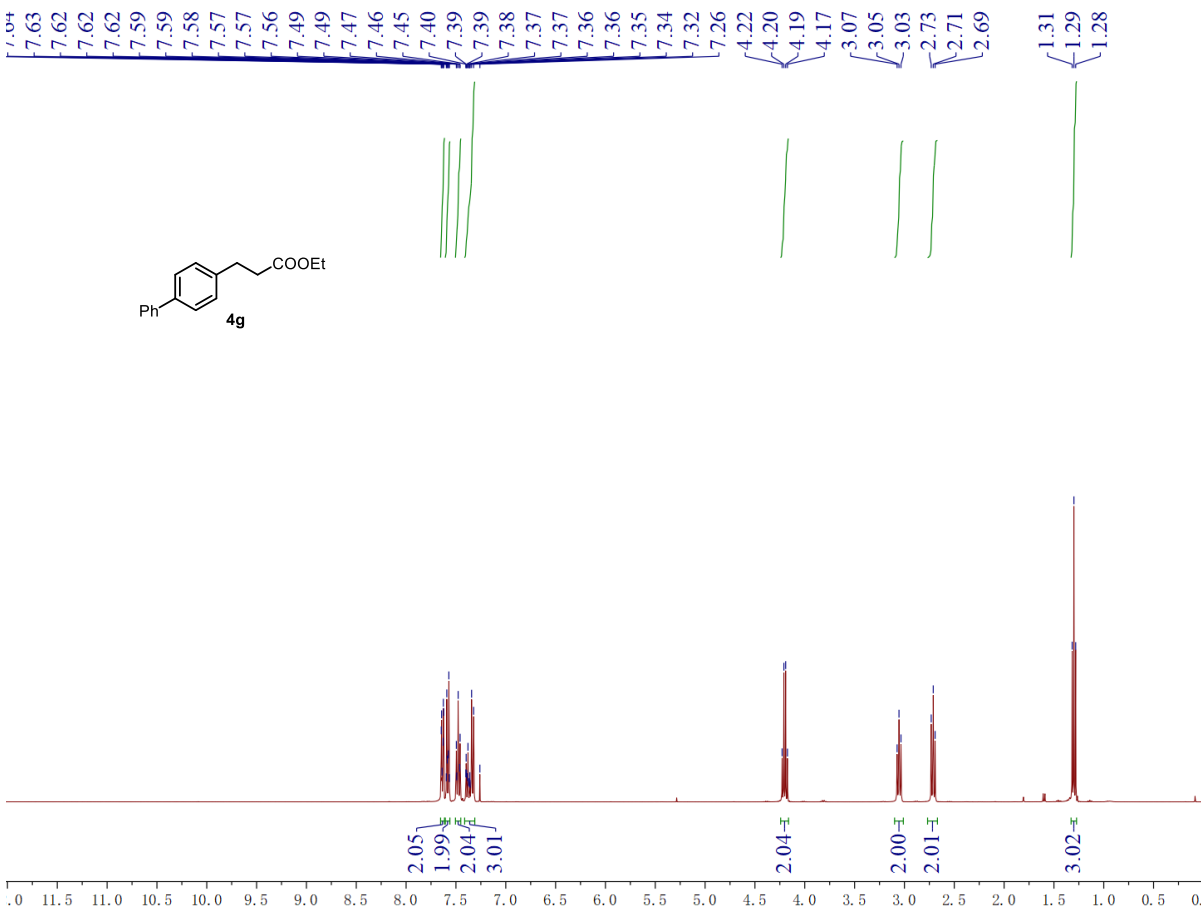
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3.02
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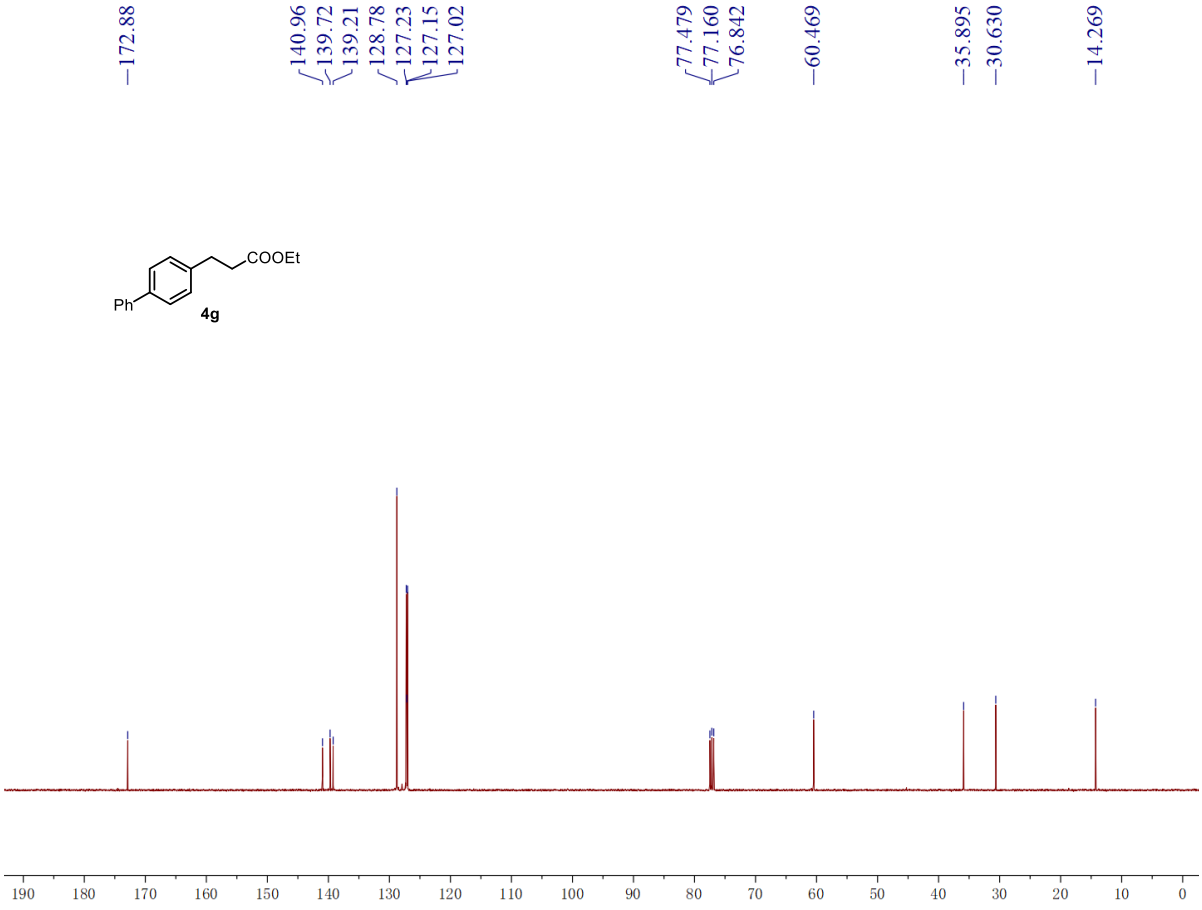
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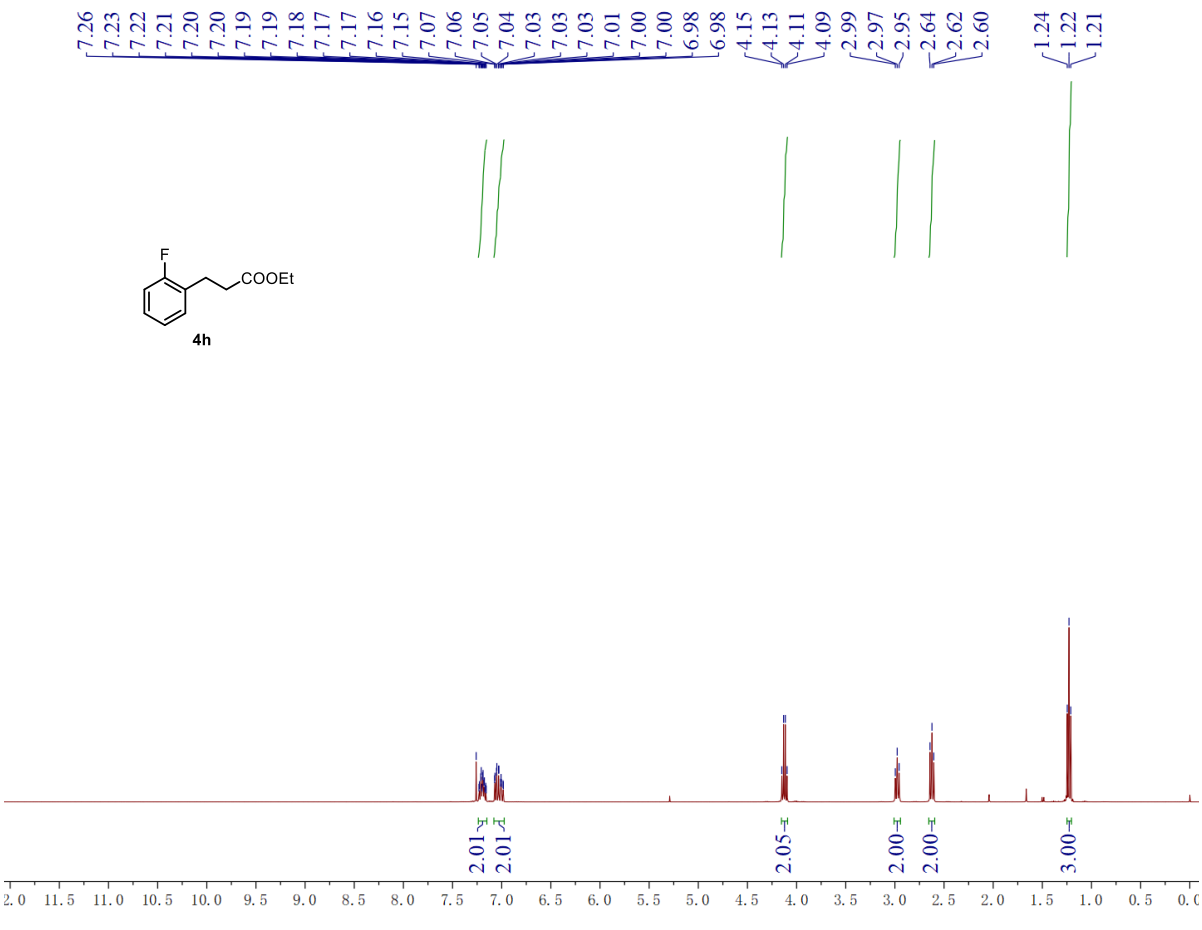
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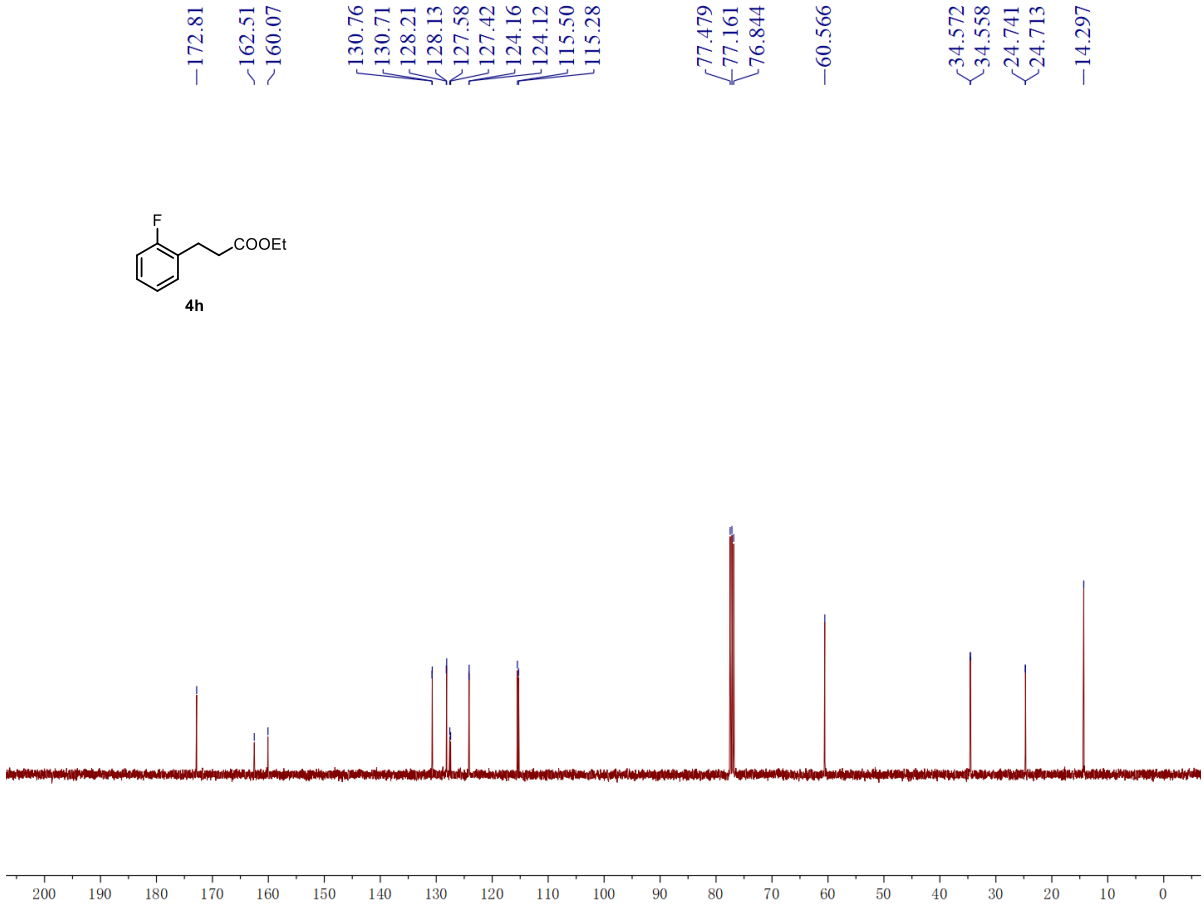
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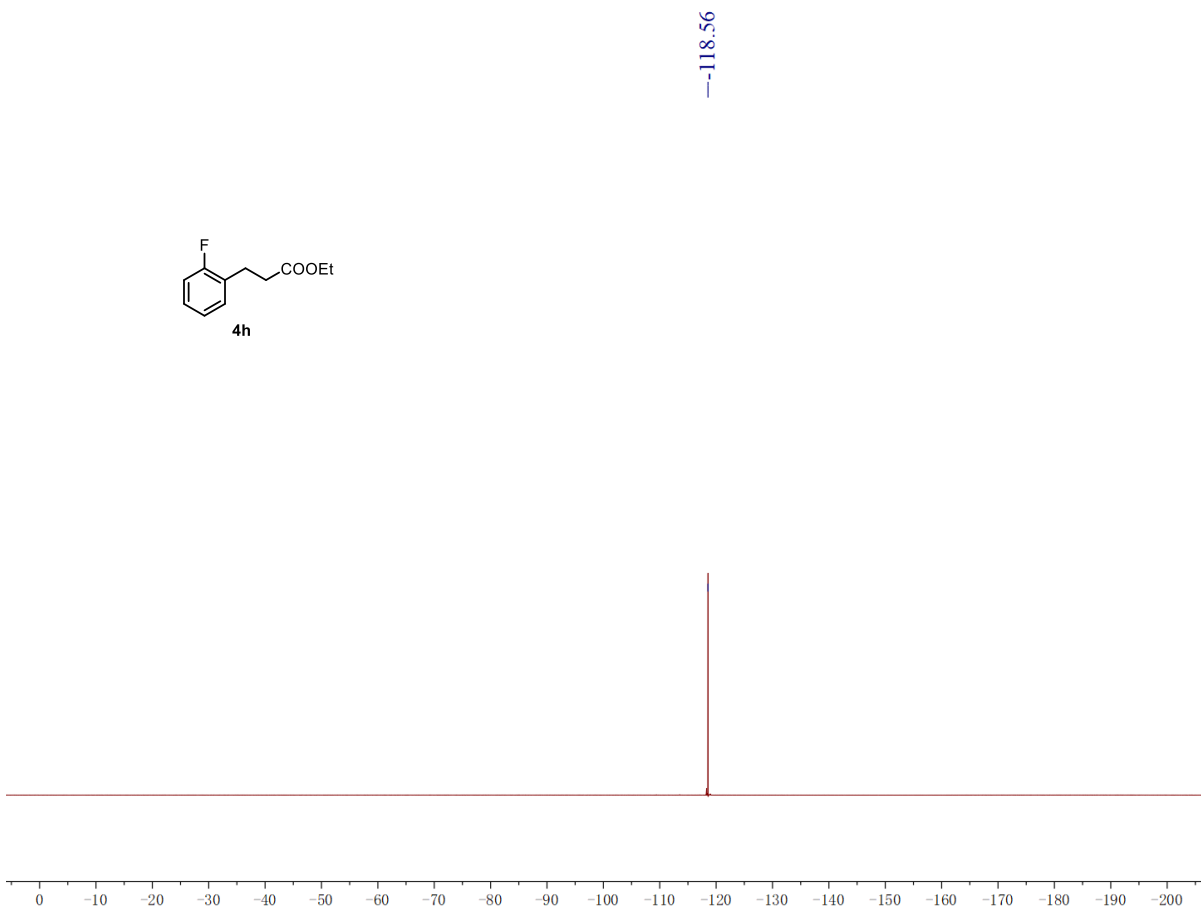
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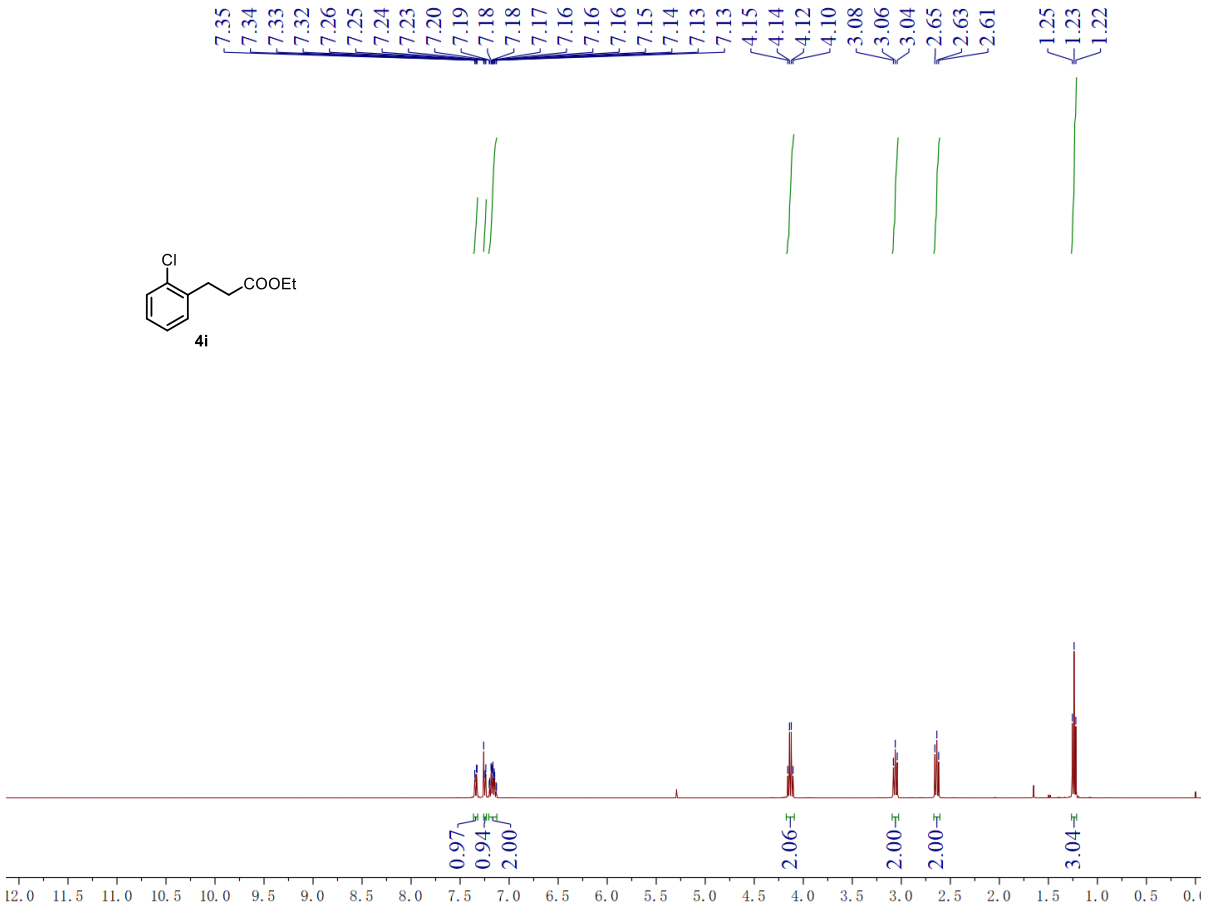
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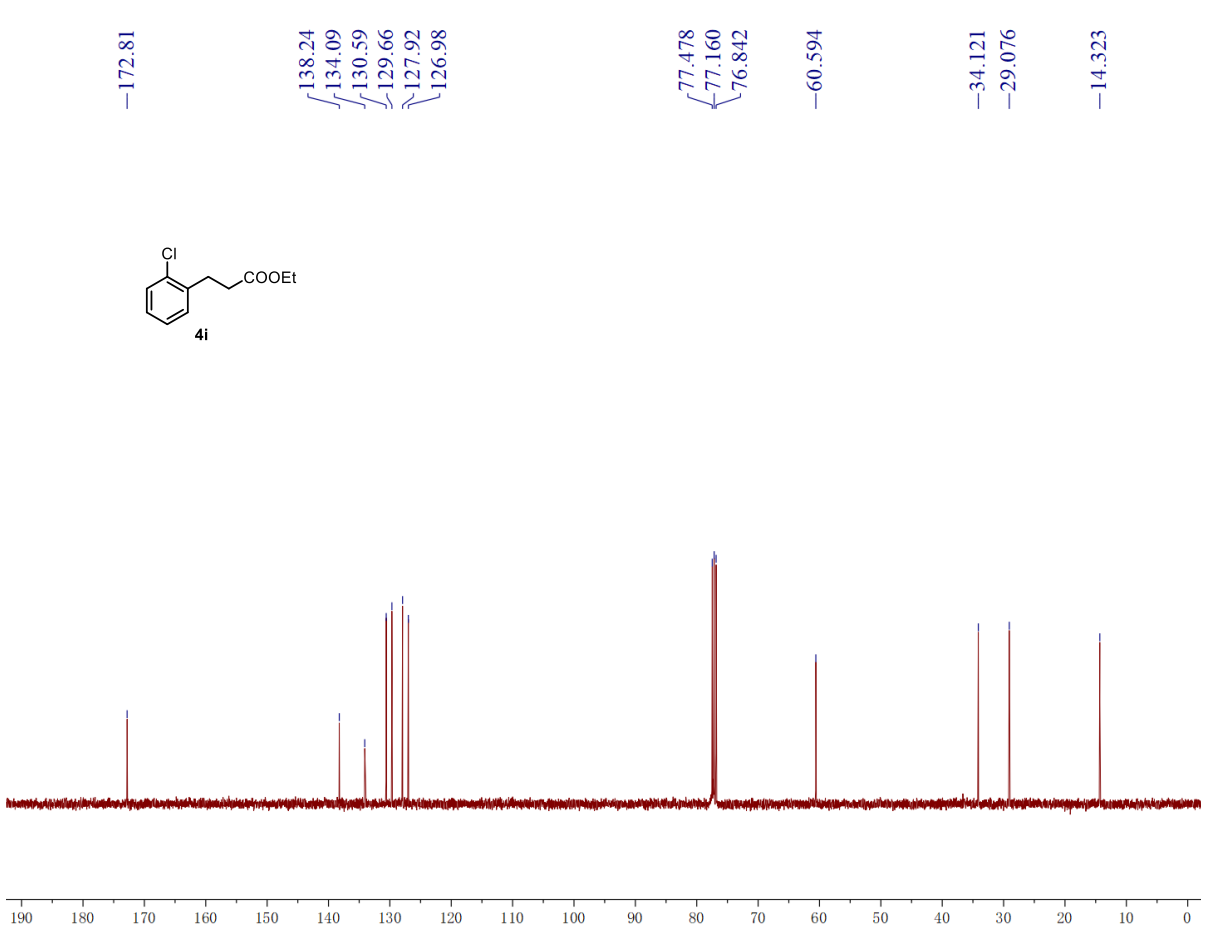
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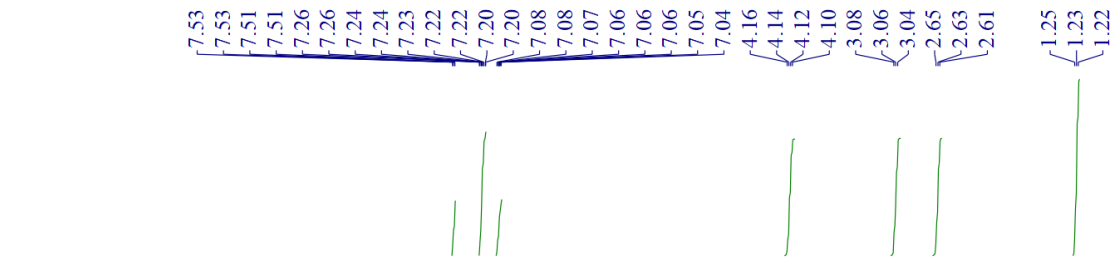
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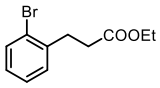
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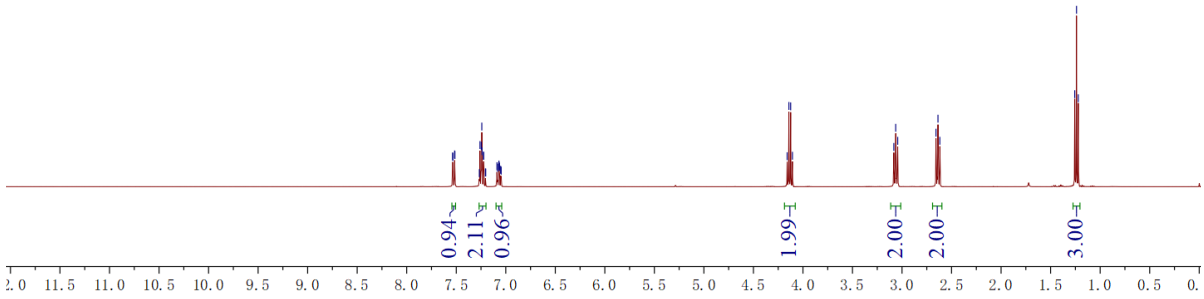
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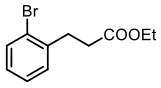
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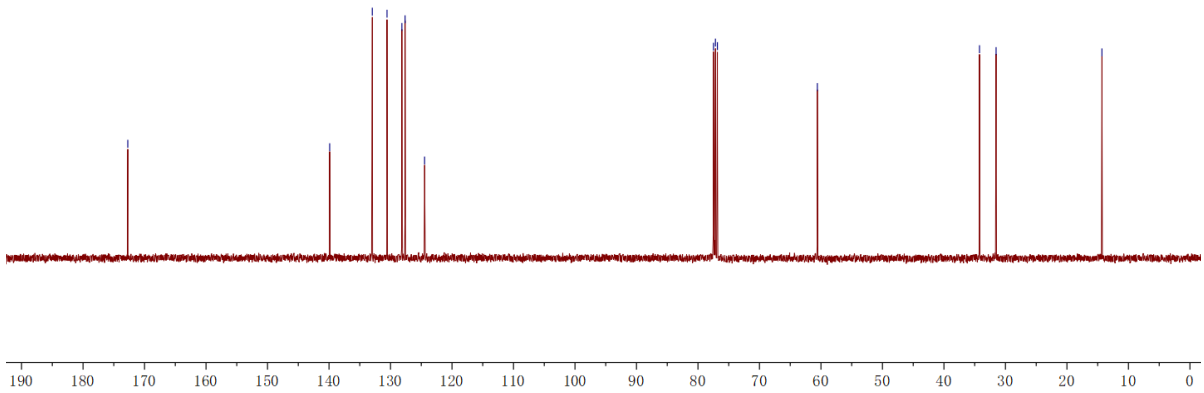
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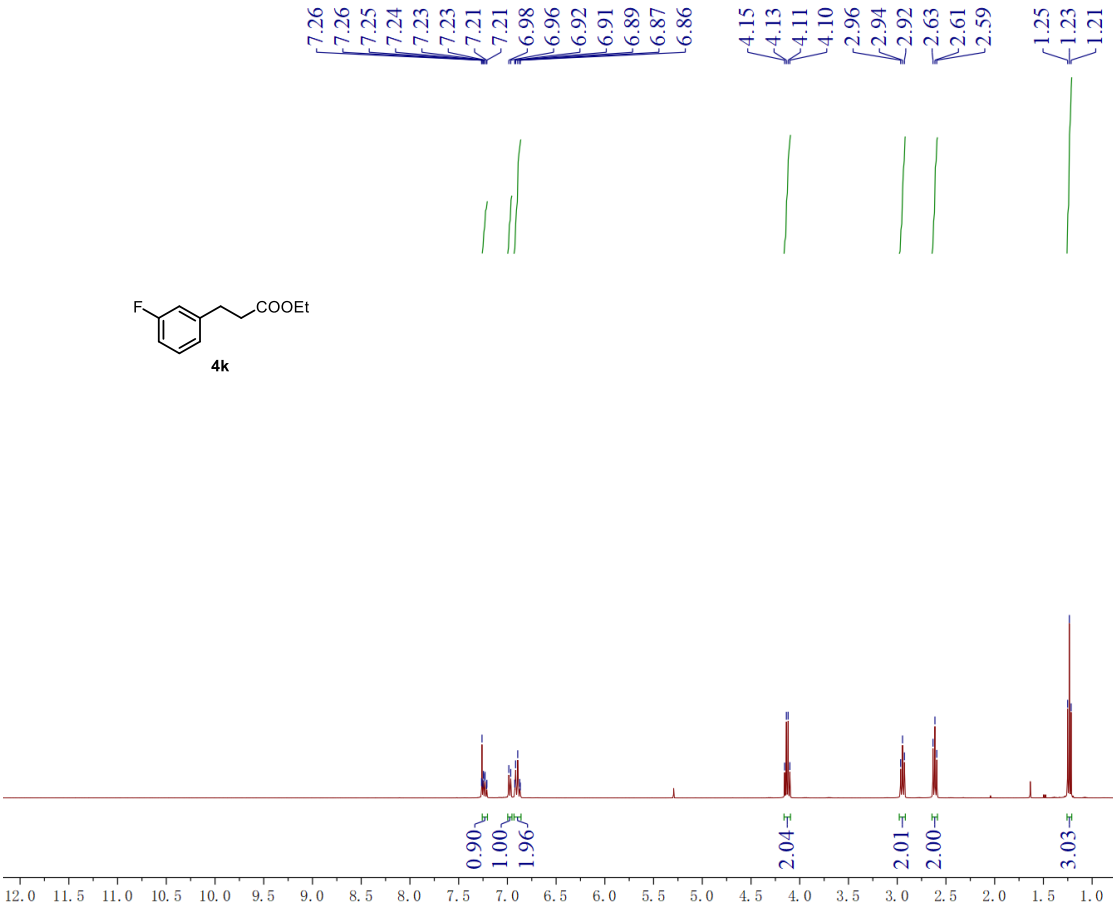
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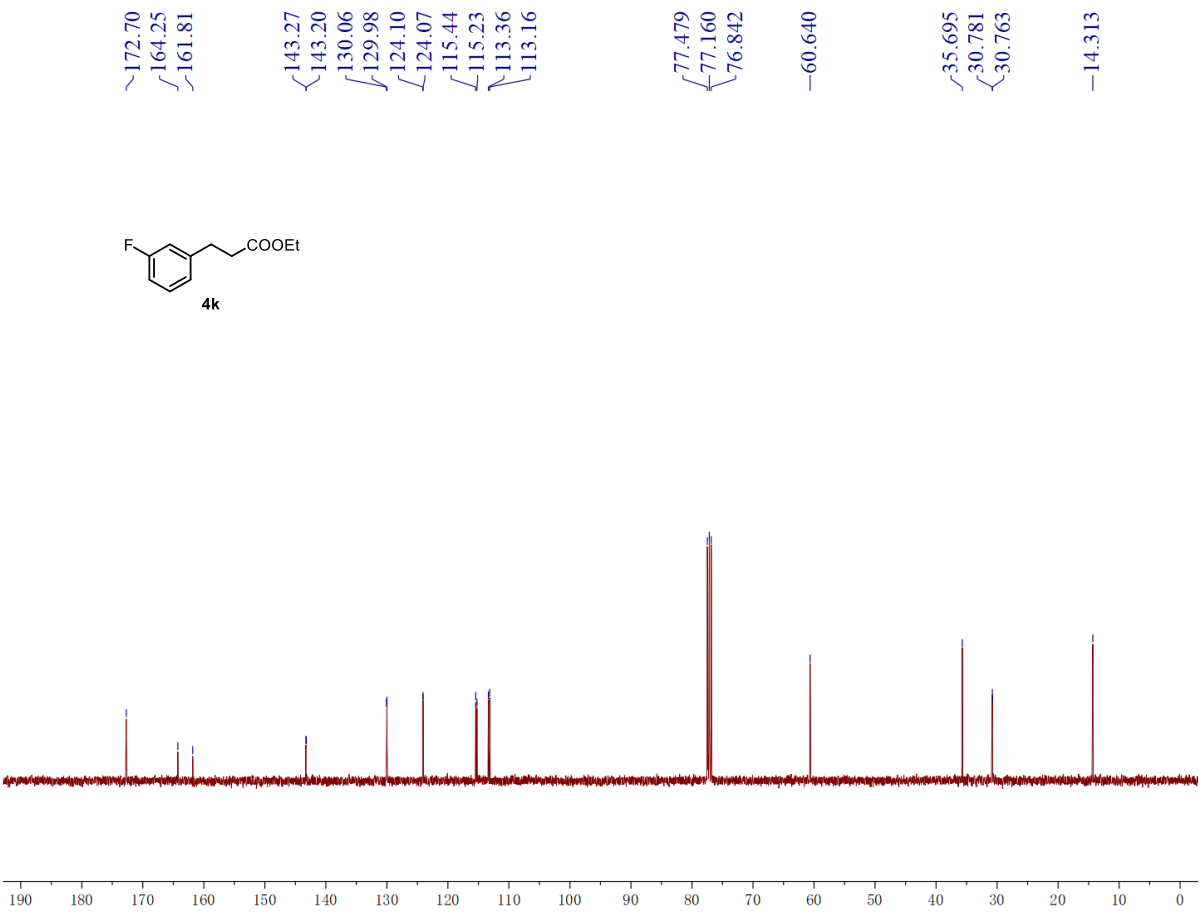
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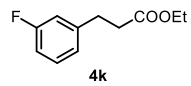
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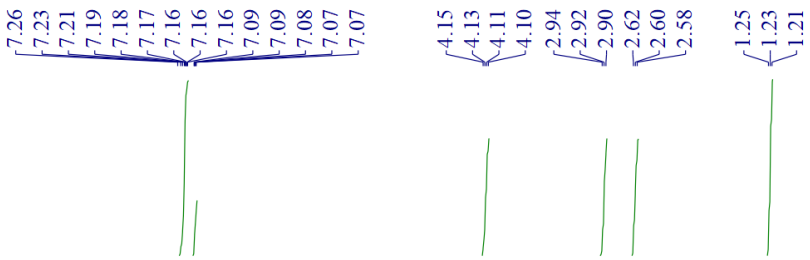
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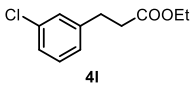
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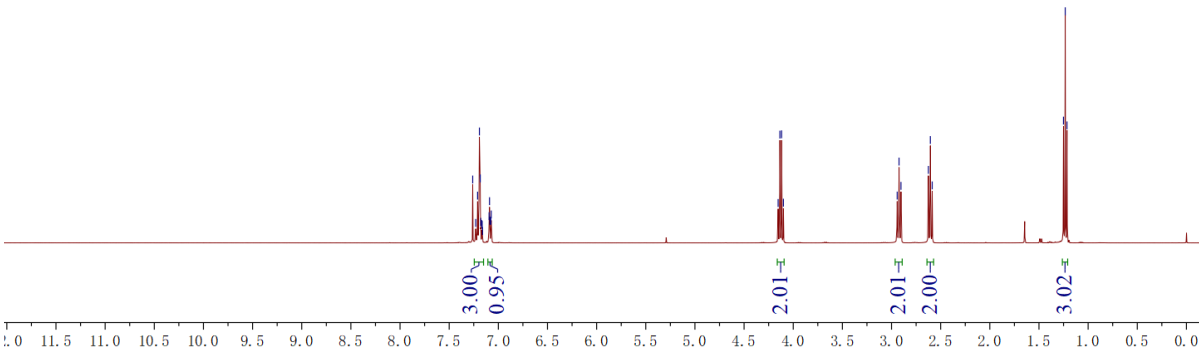
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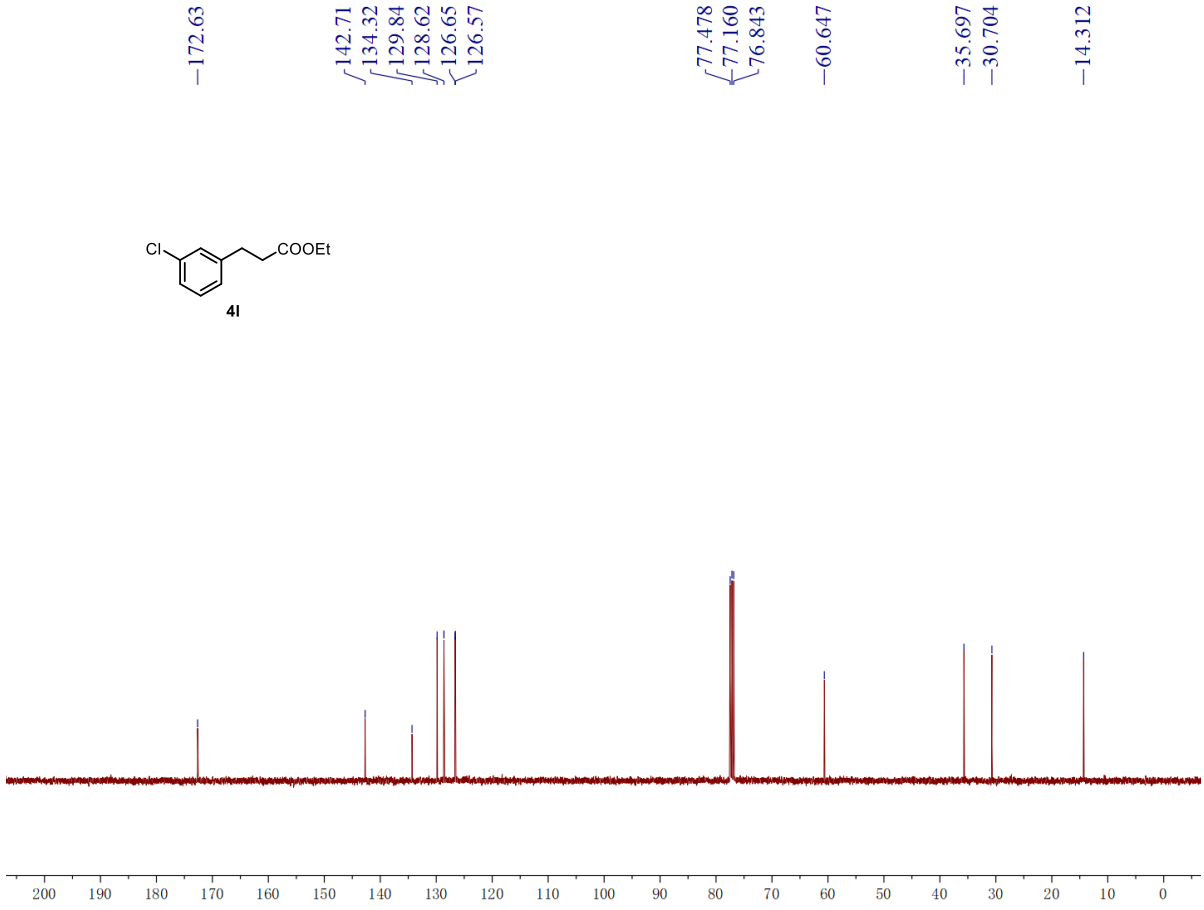
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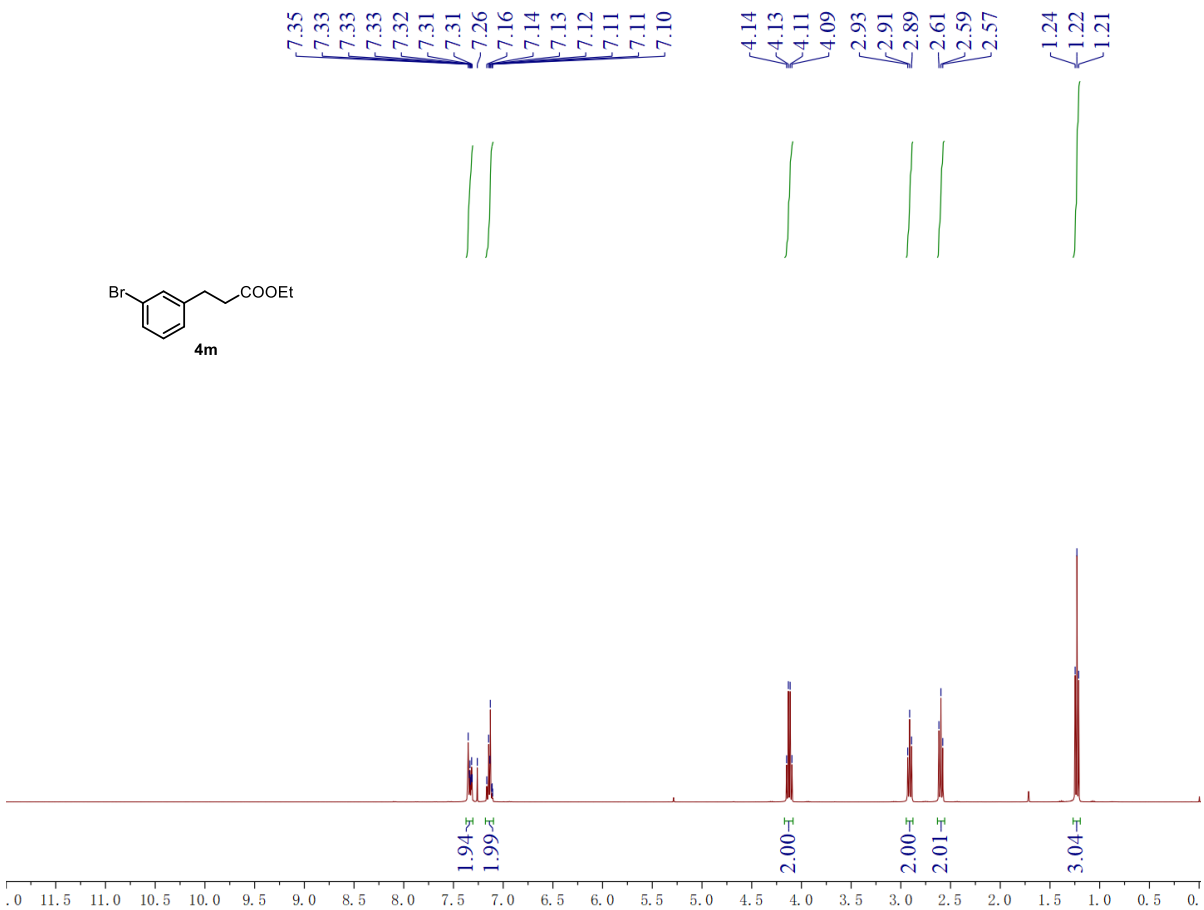
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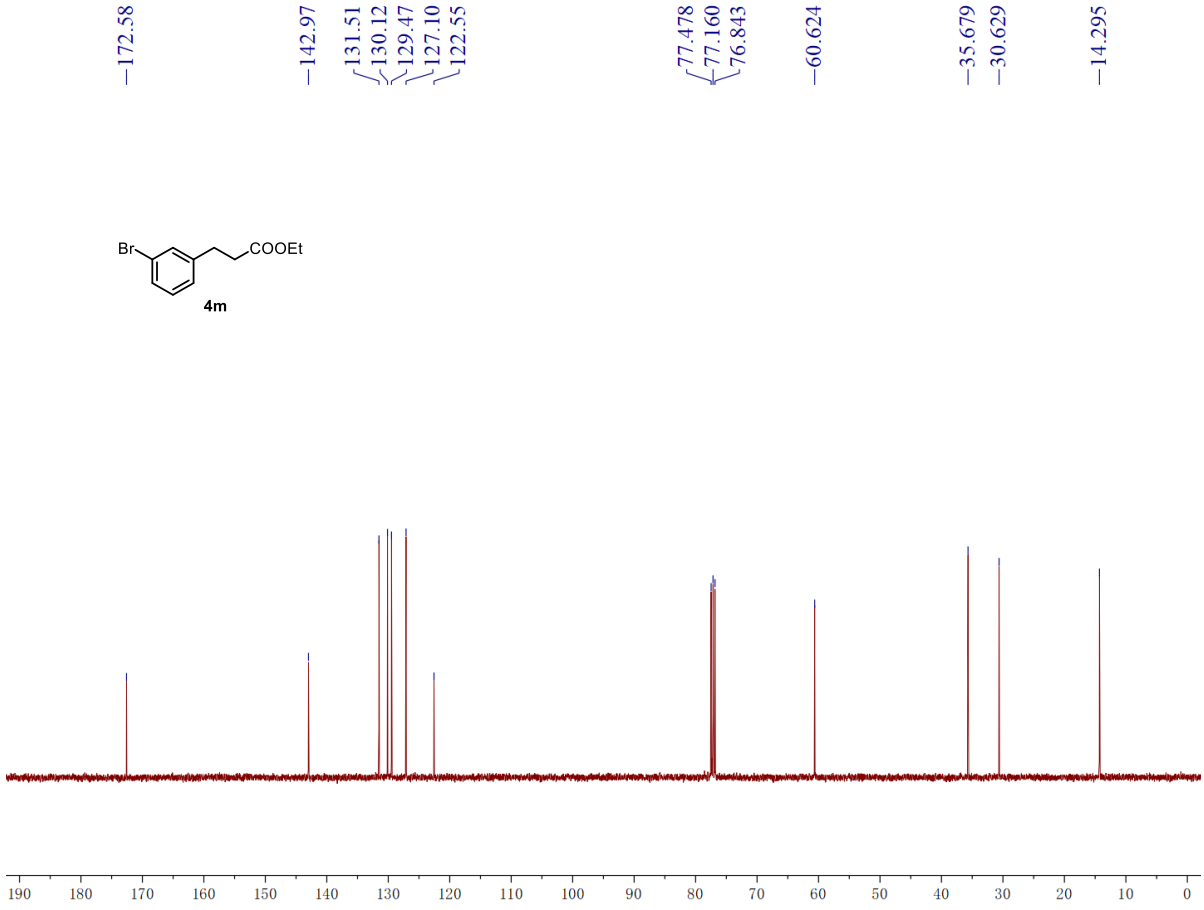
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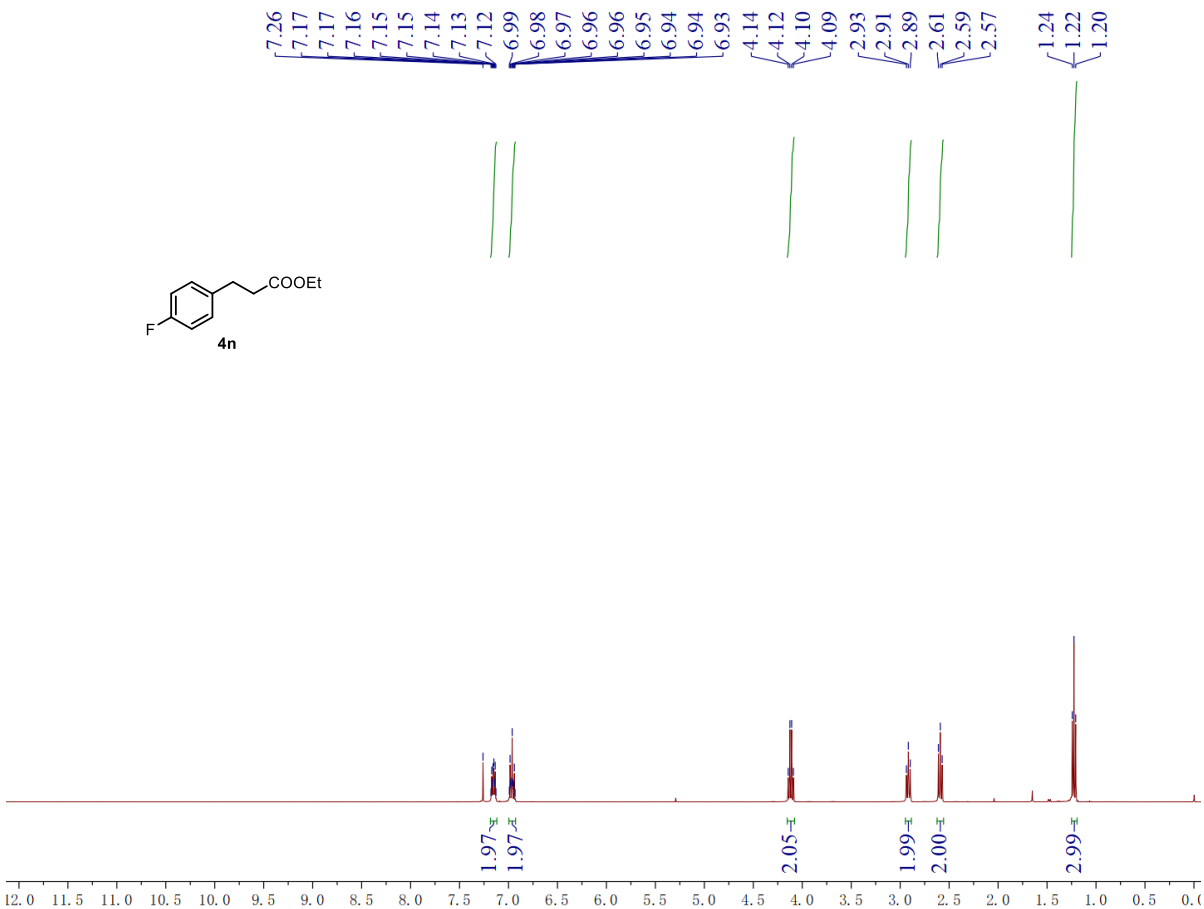
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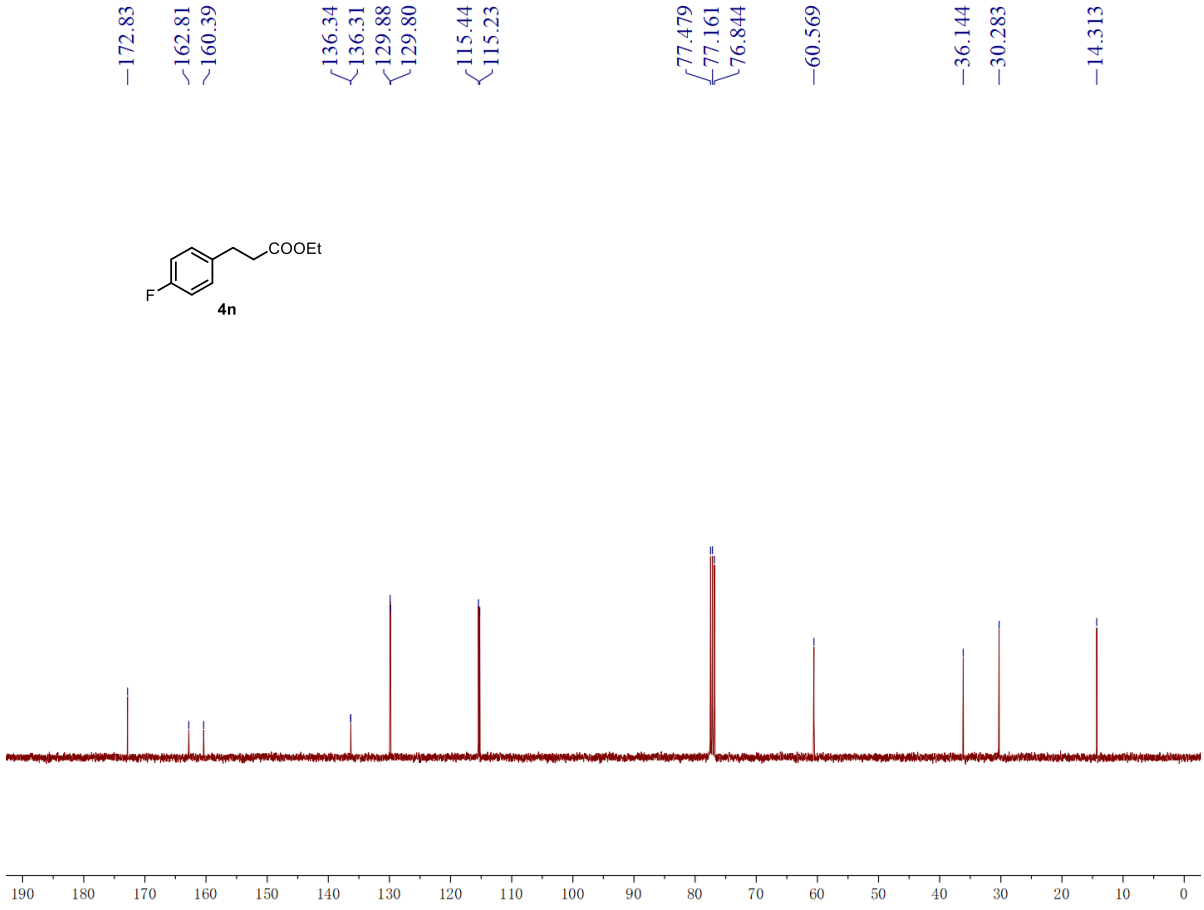
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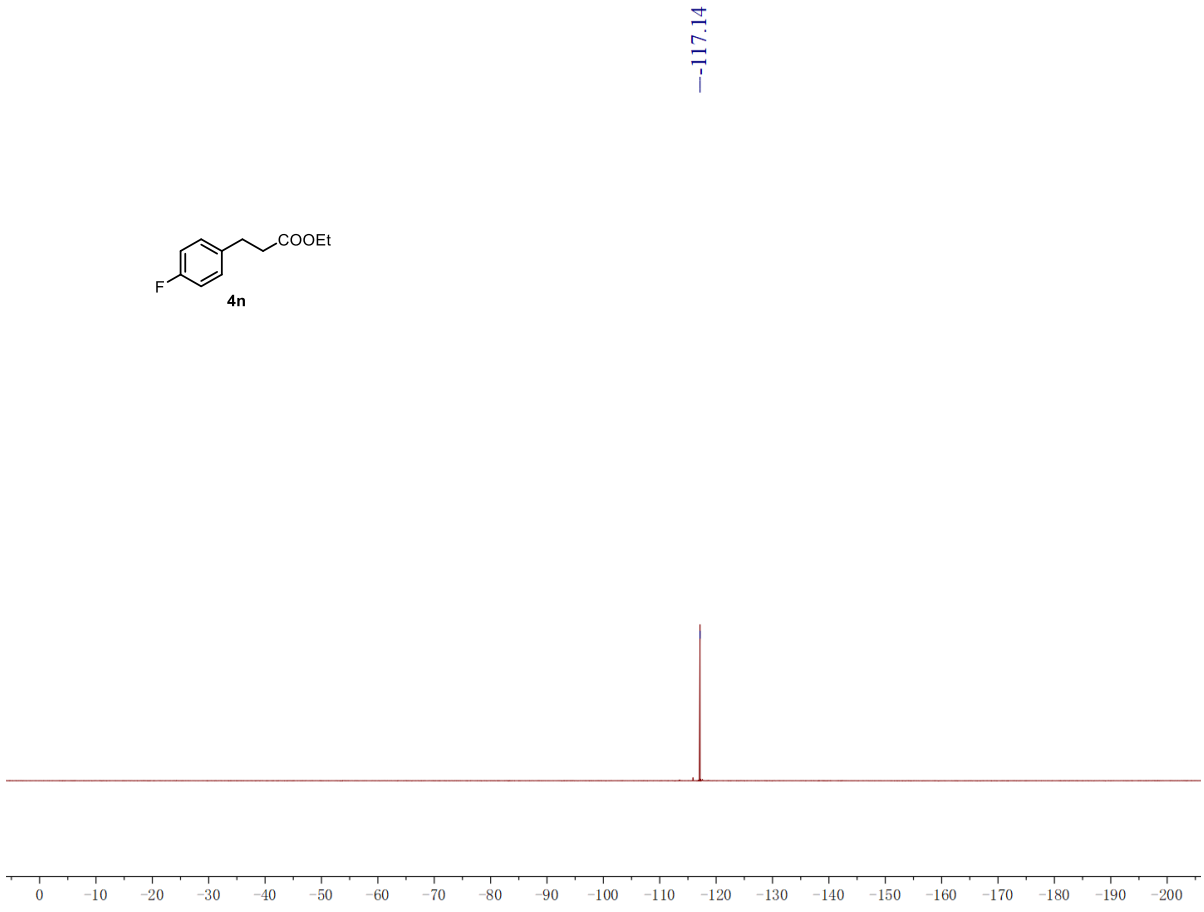
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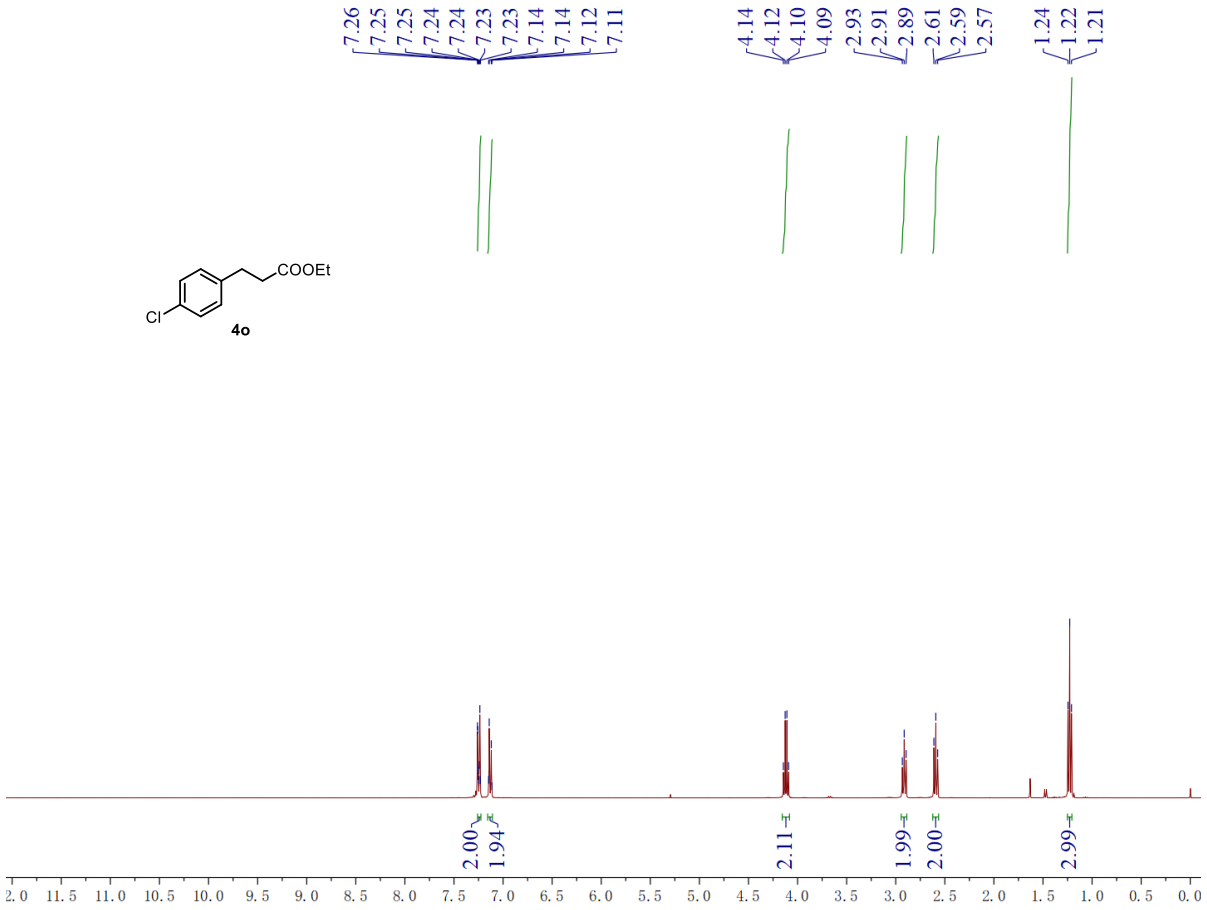
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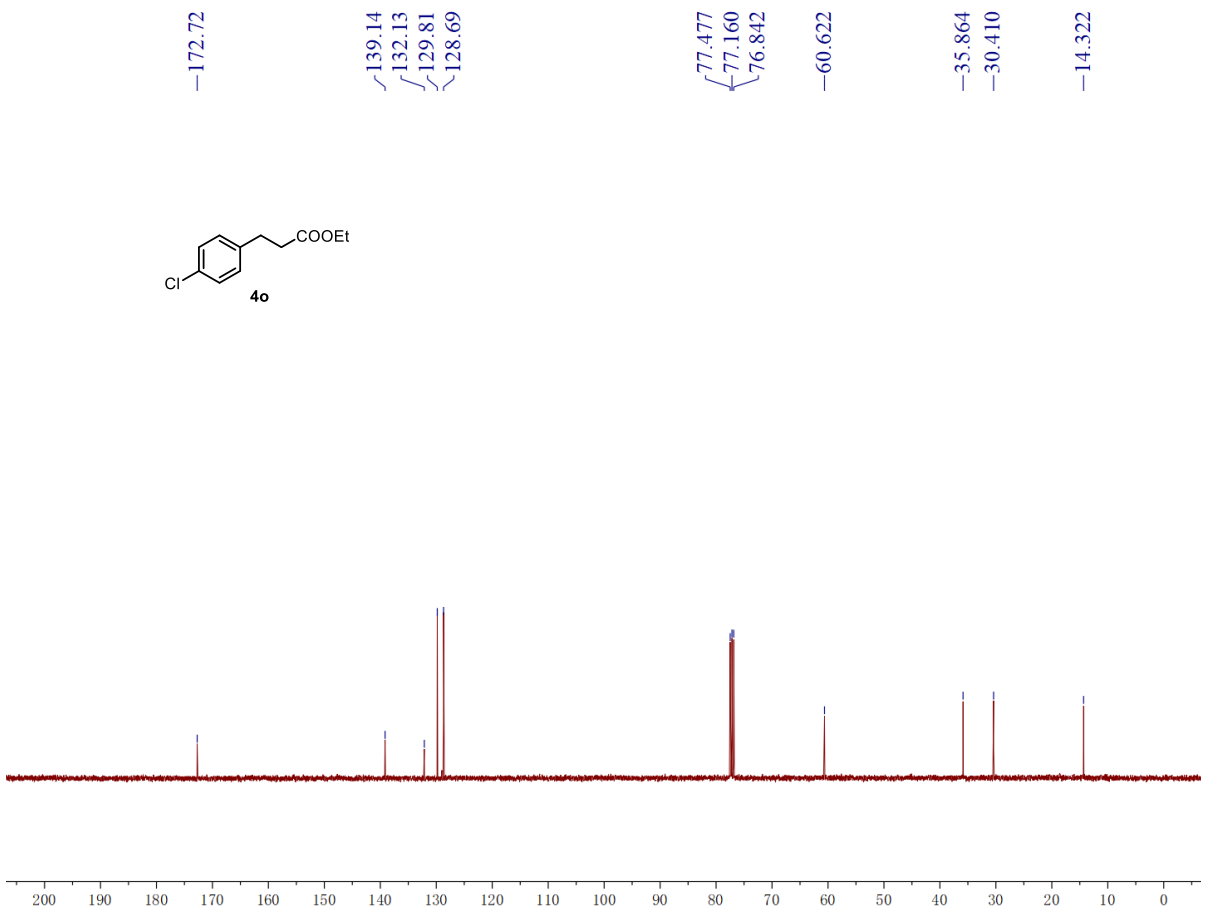
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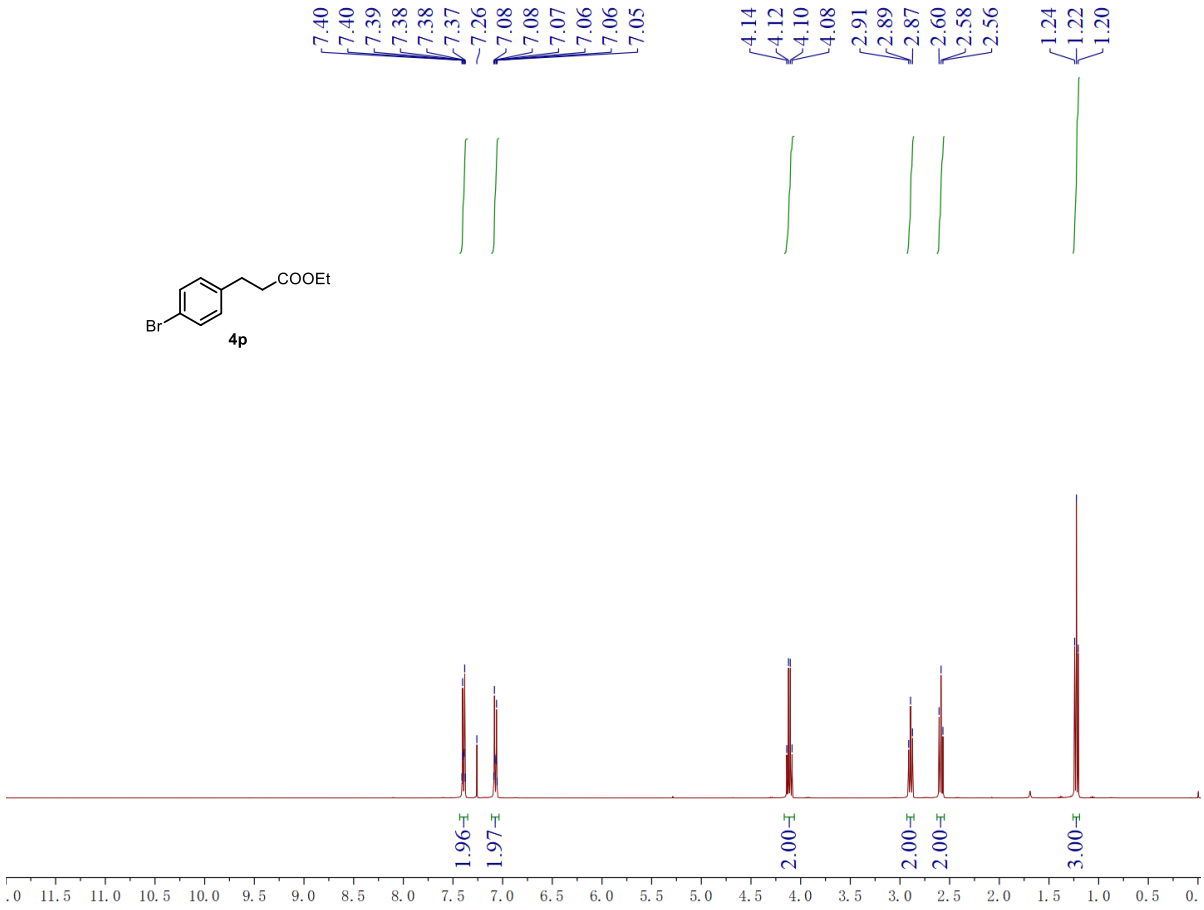
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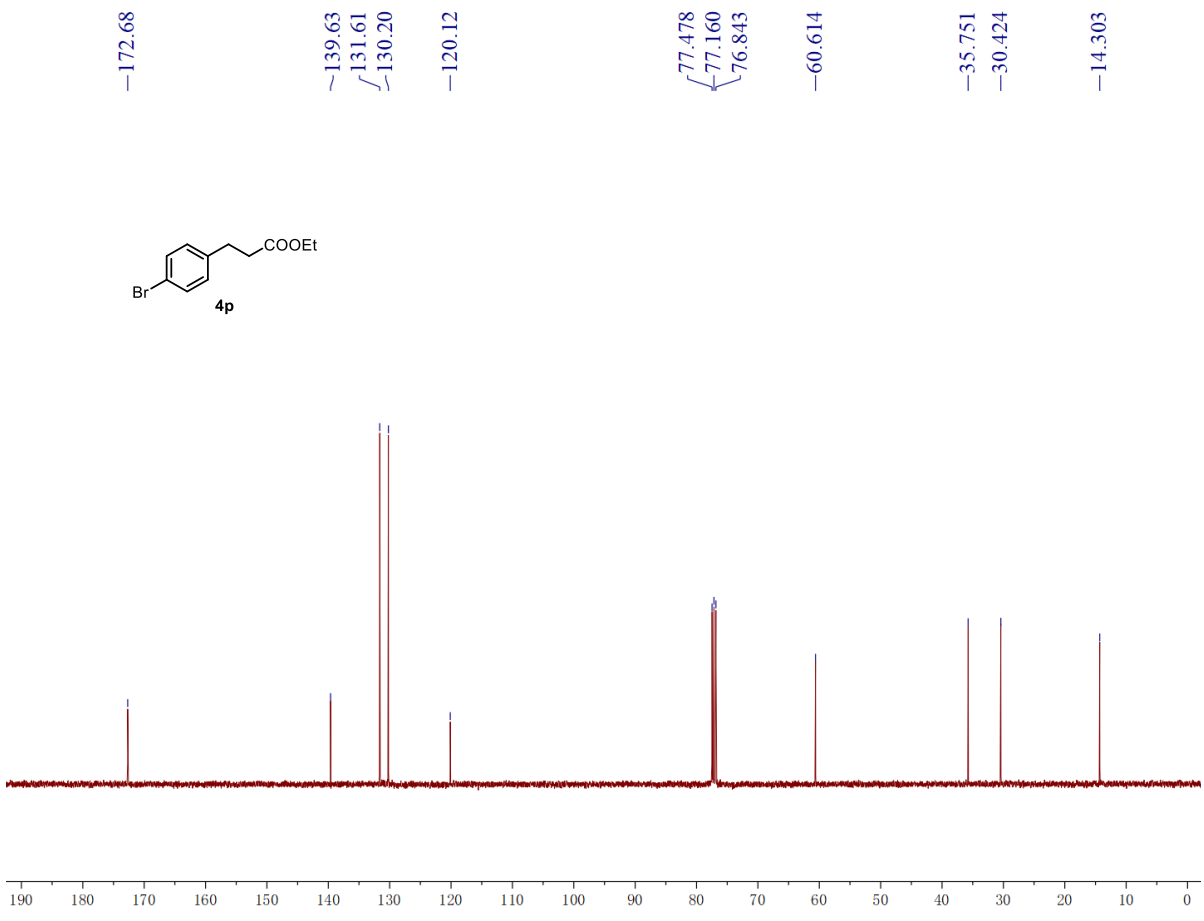
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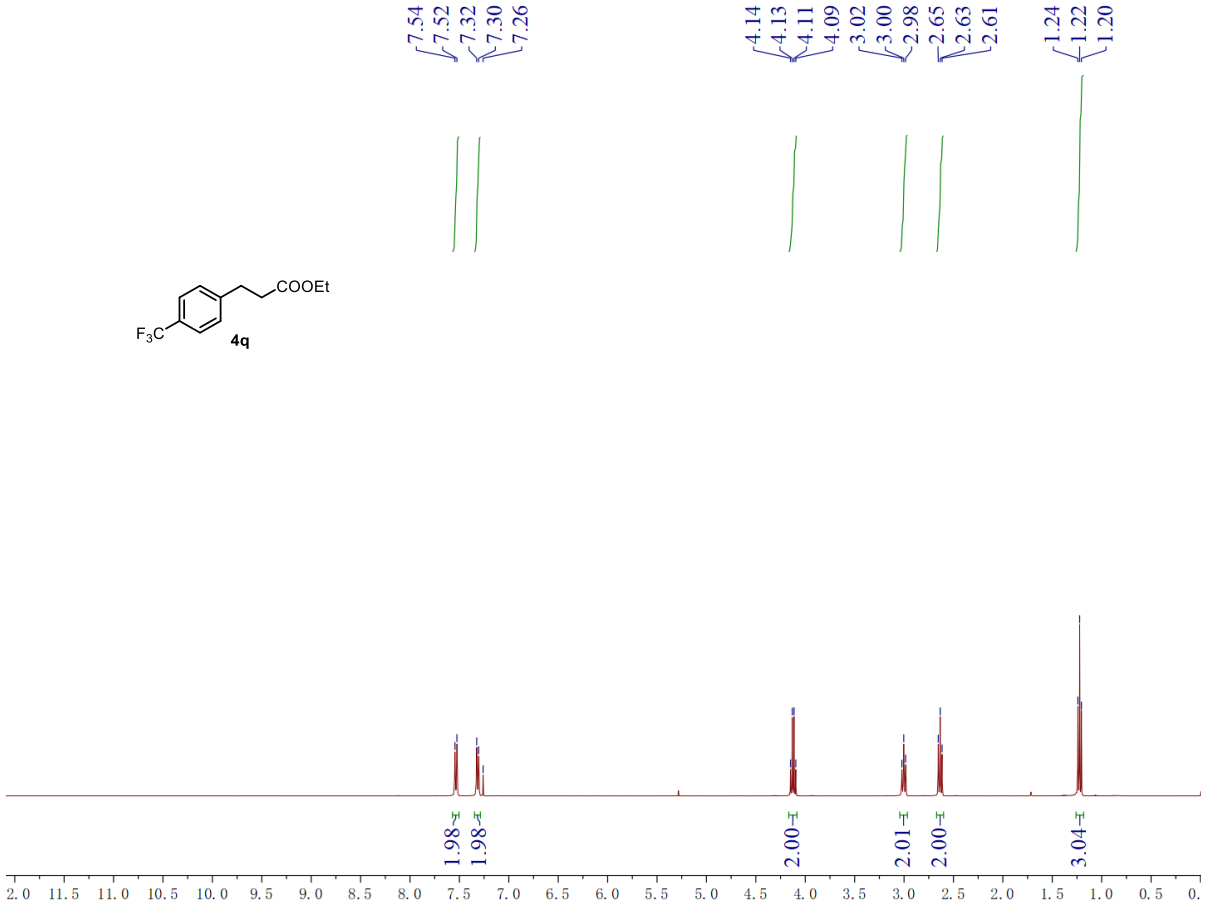
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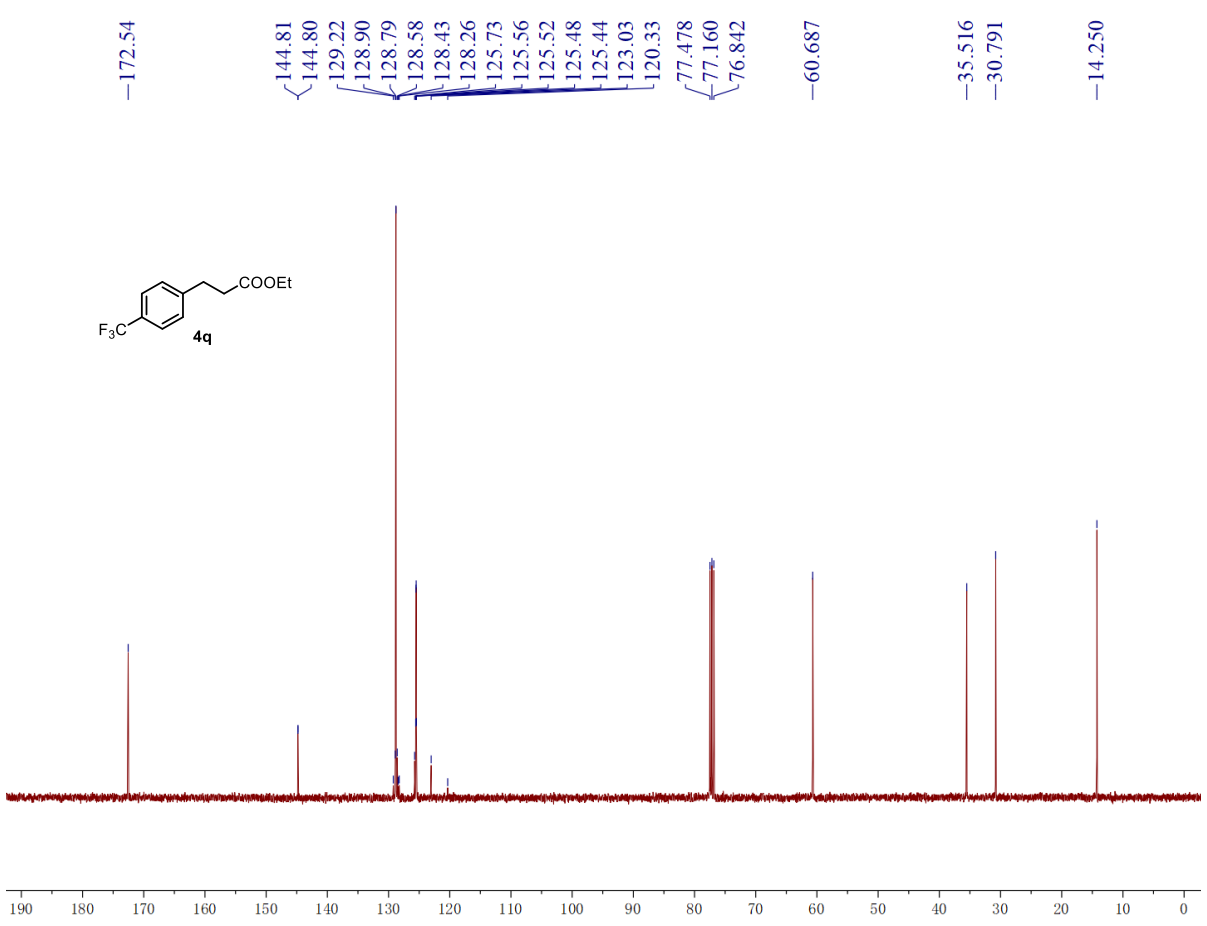
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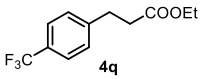
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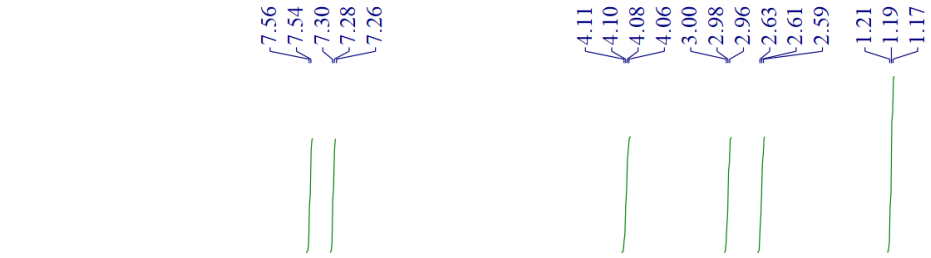
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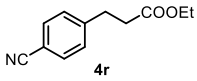
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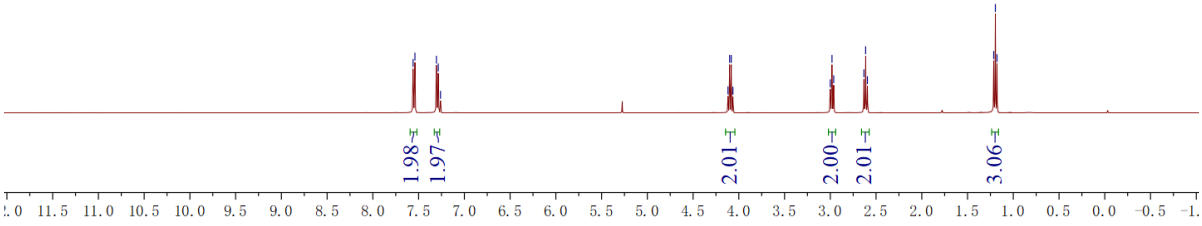
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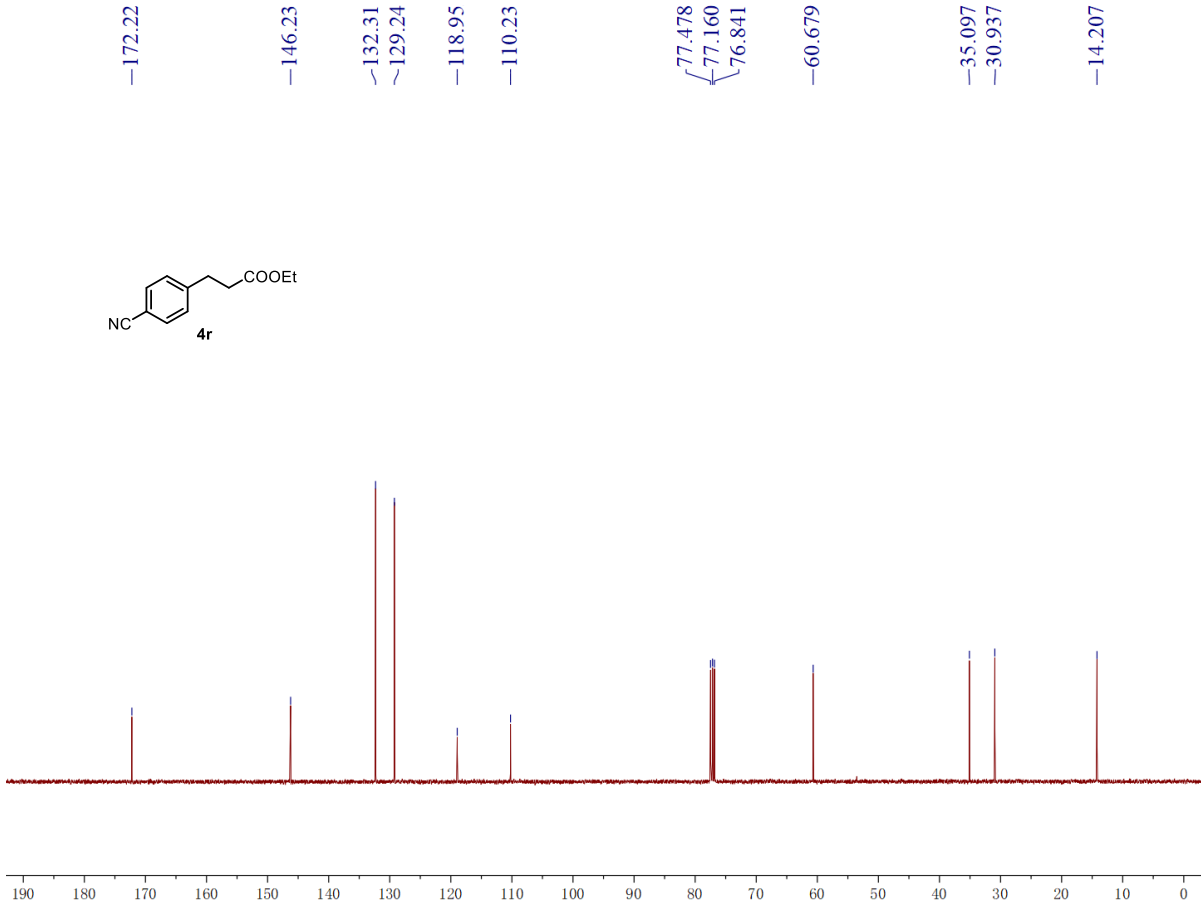
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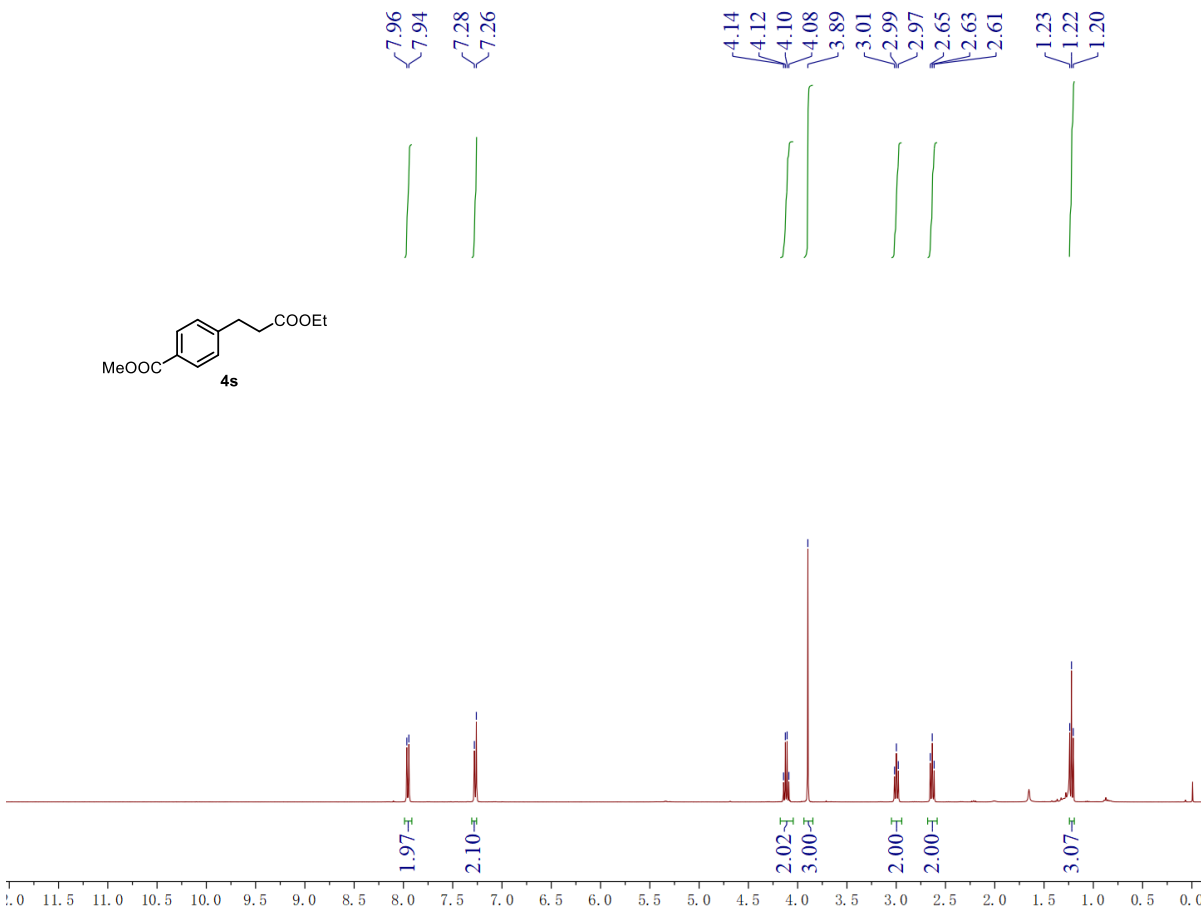
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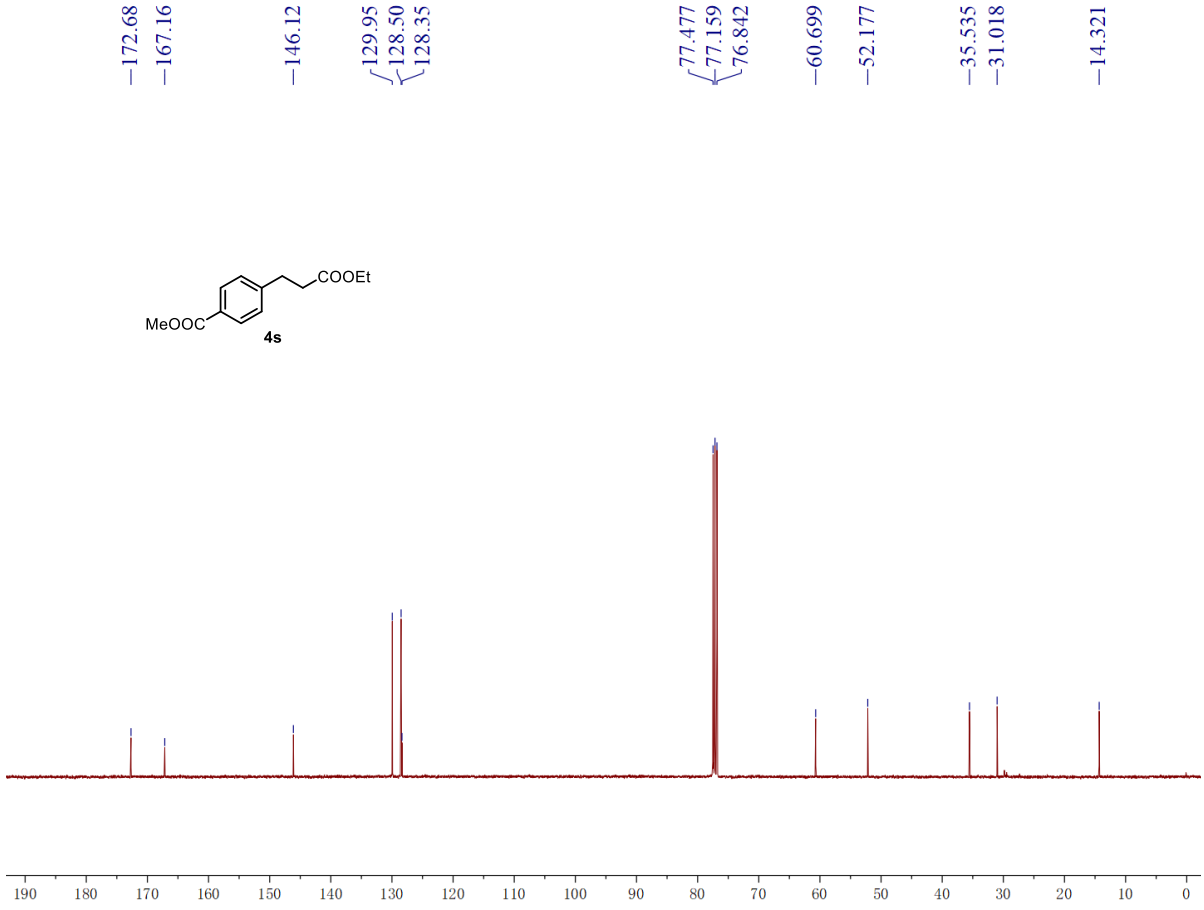
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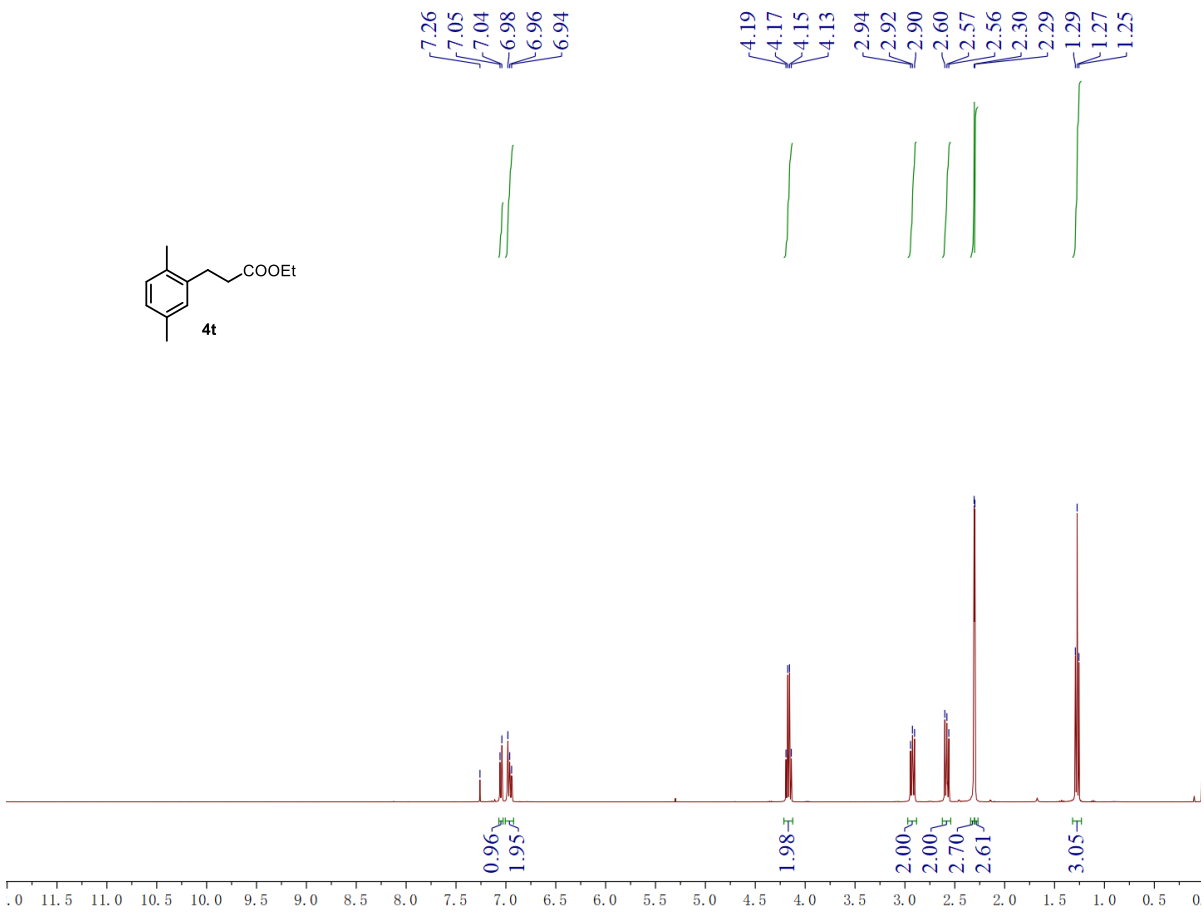
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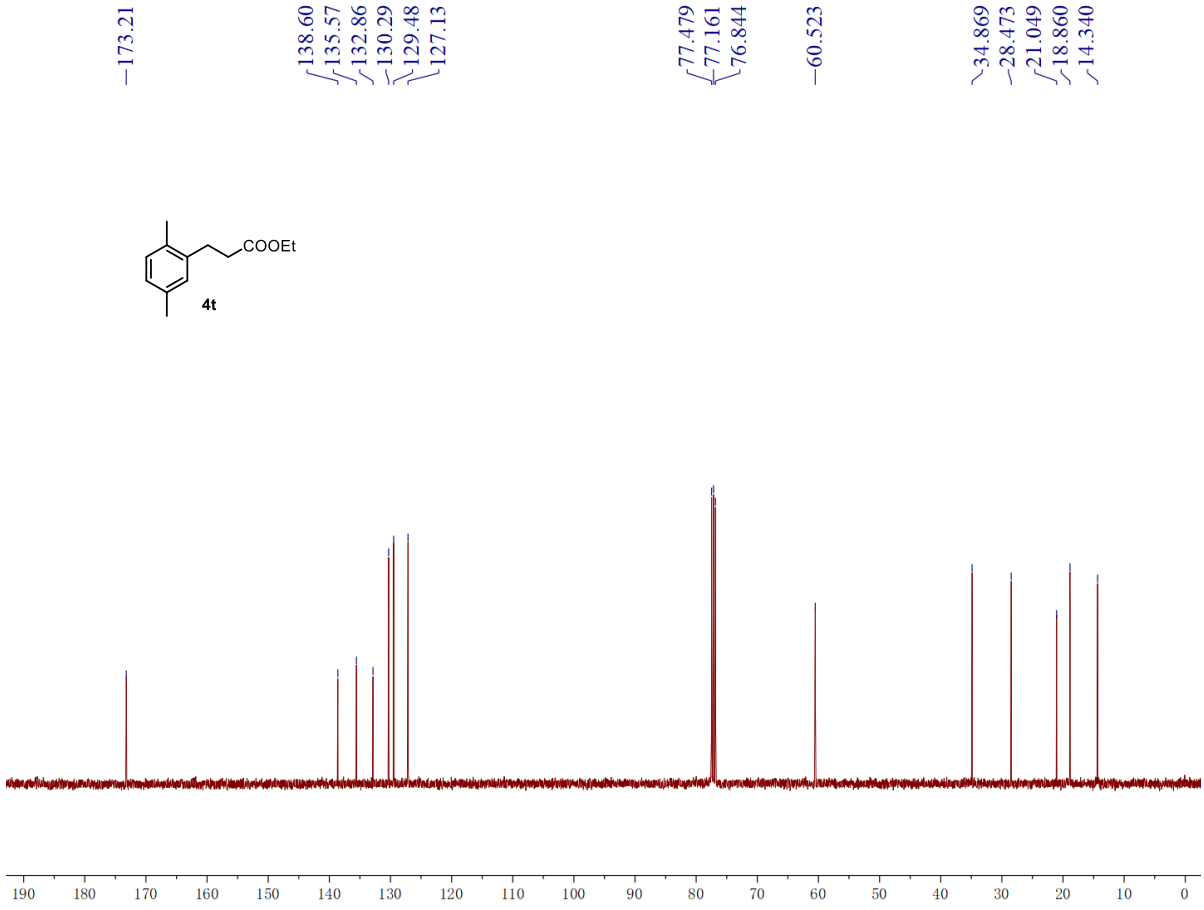
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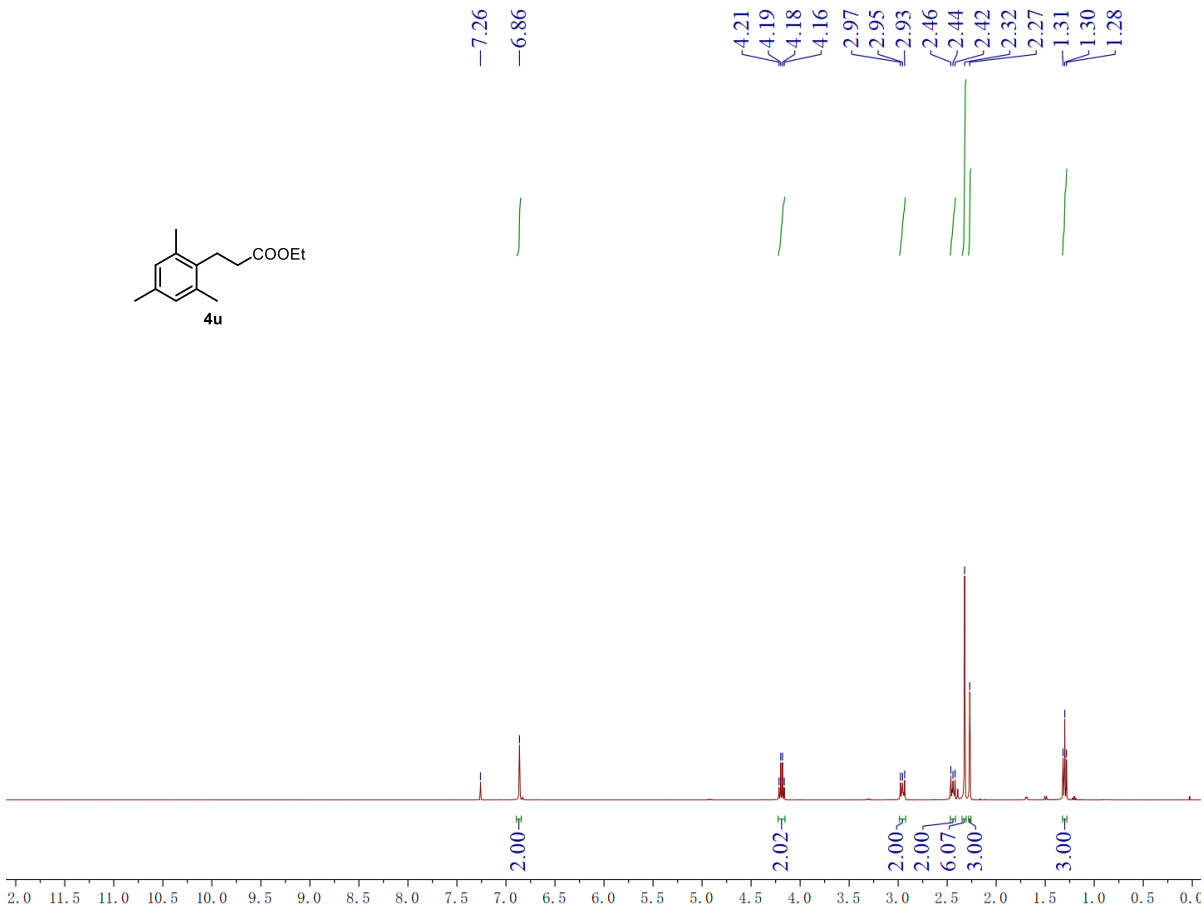
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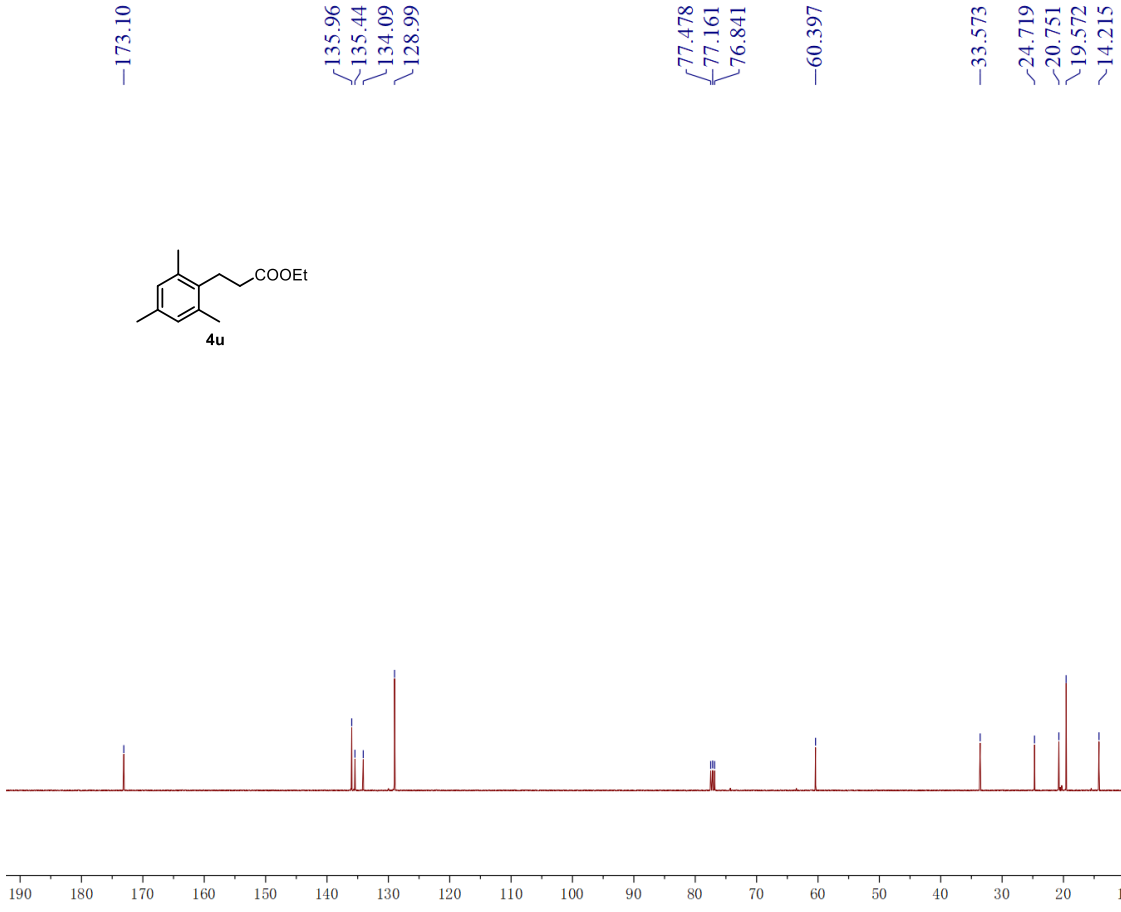
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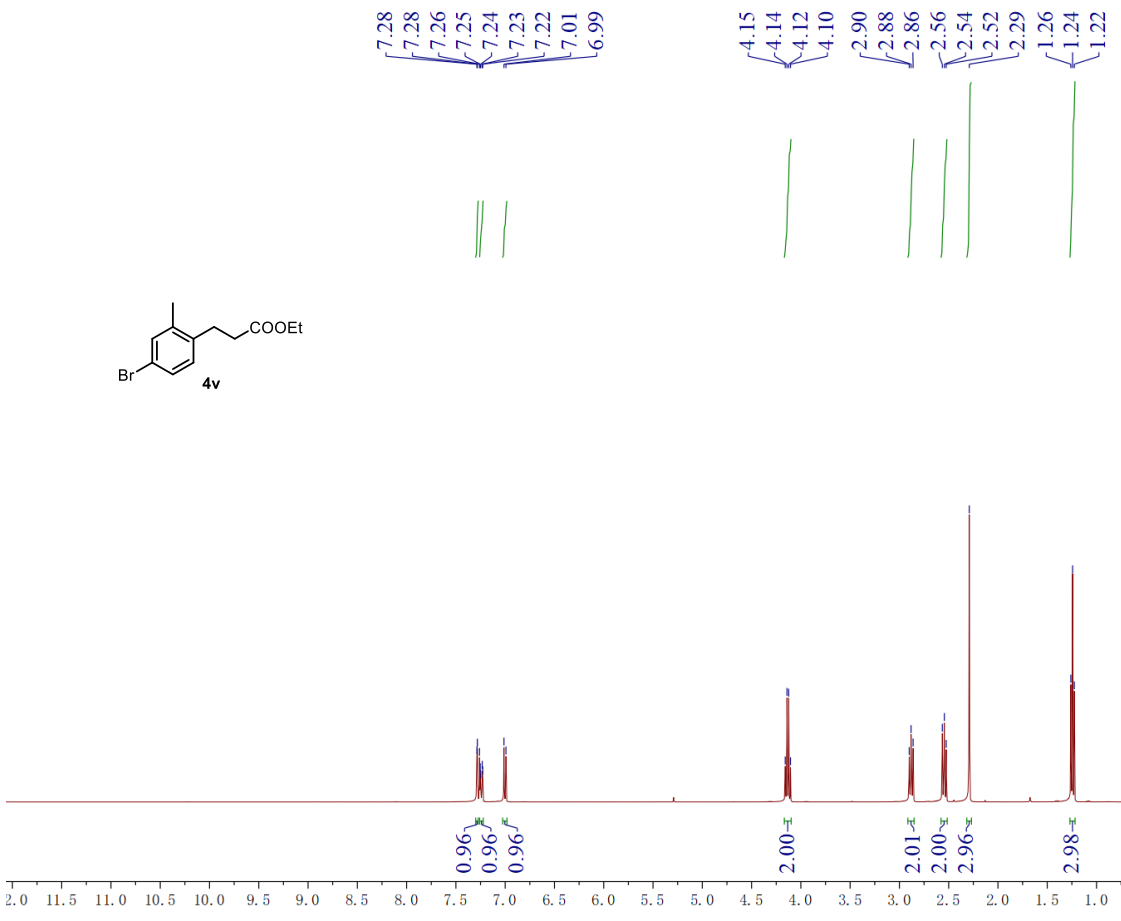
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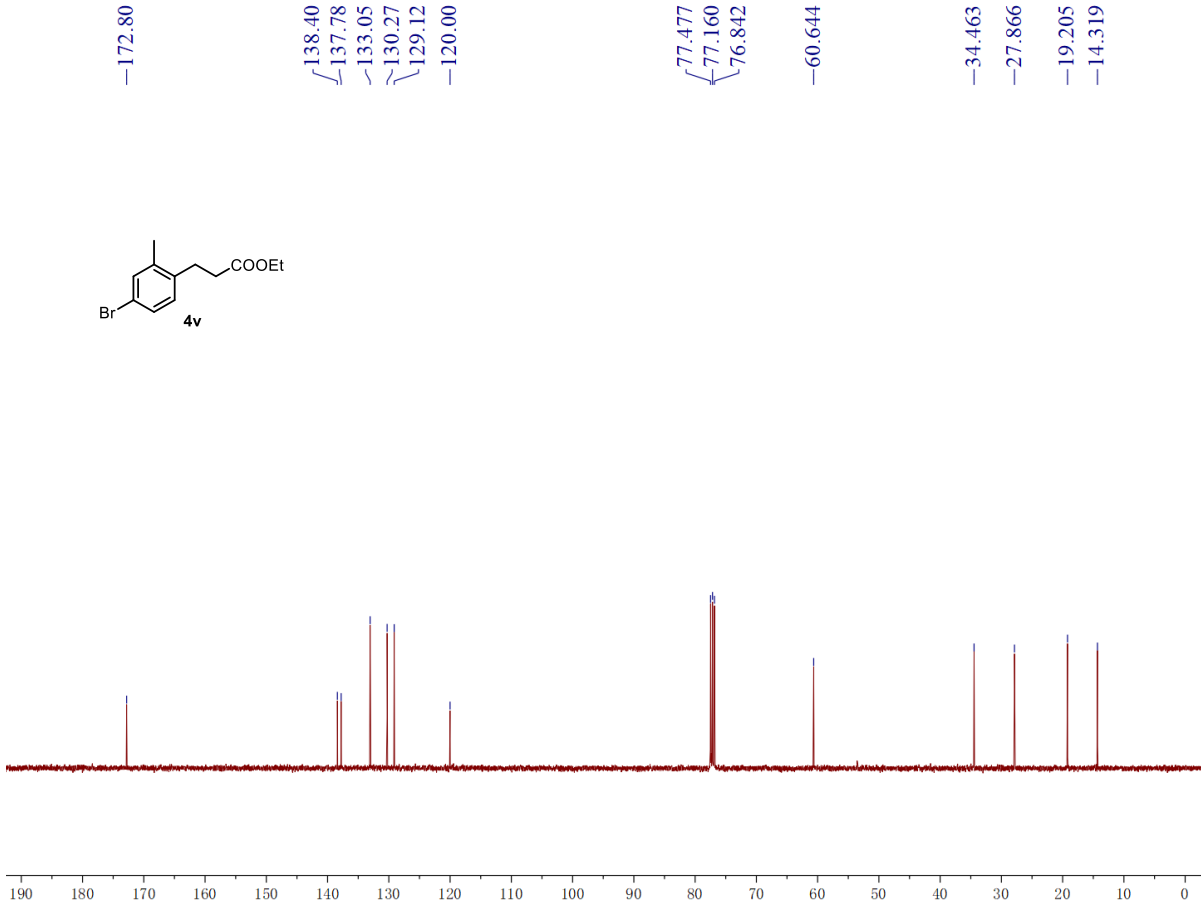
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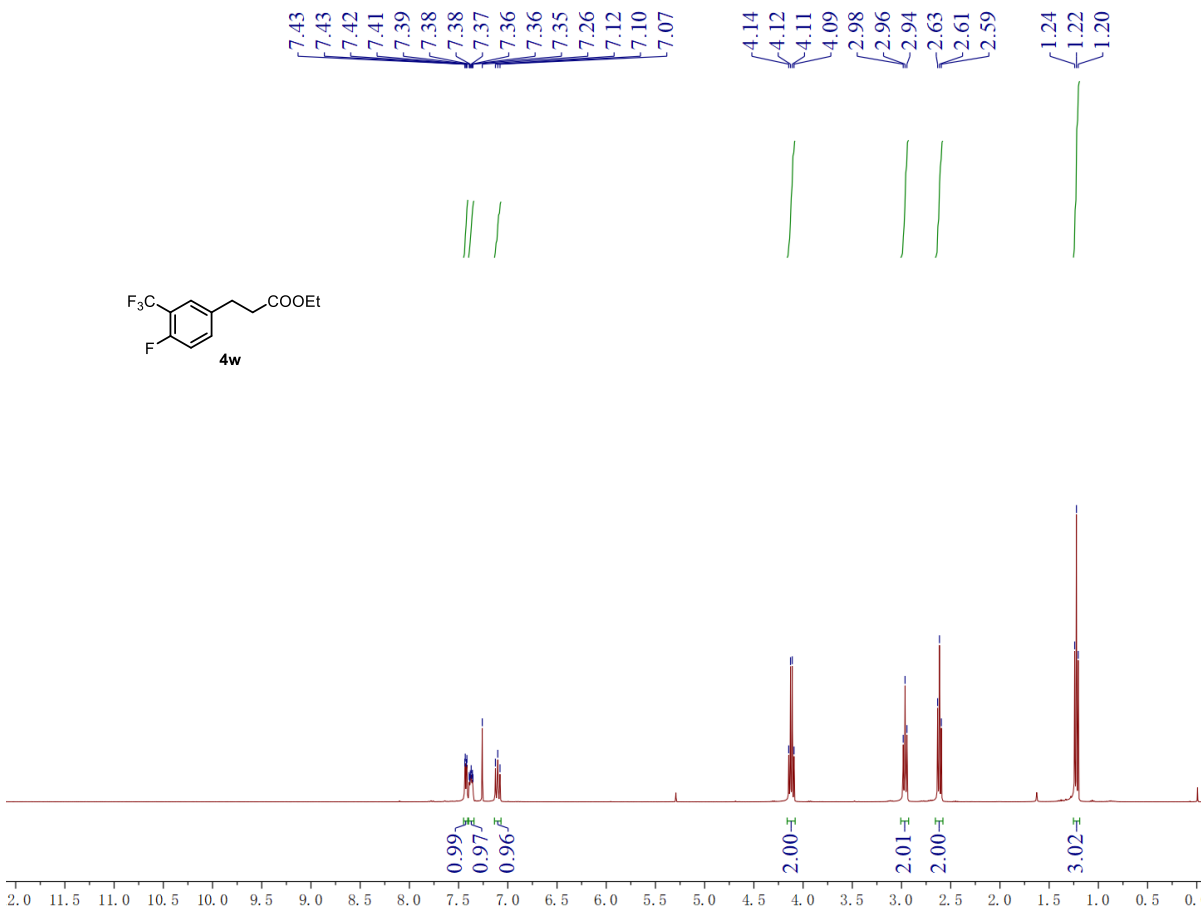
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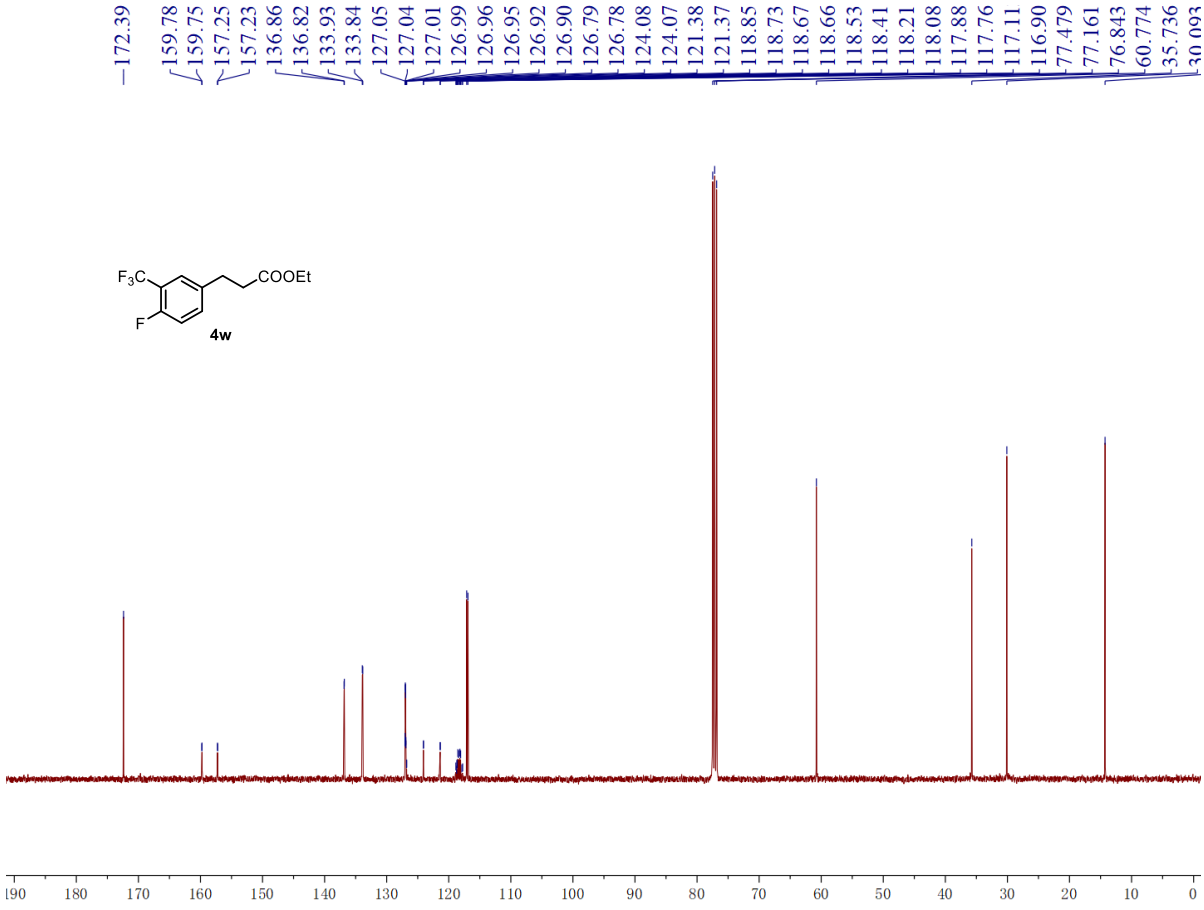
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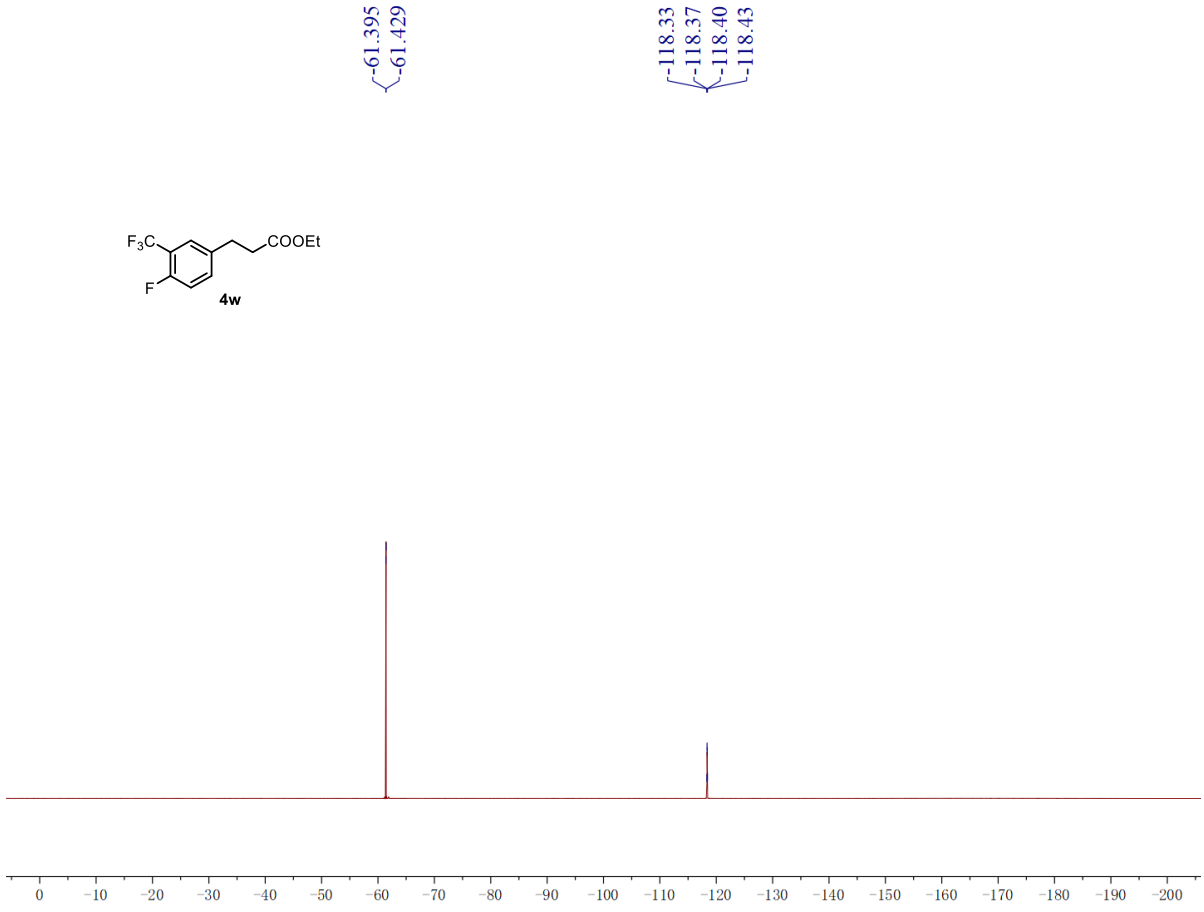
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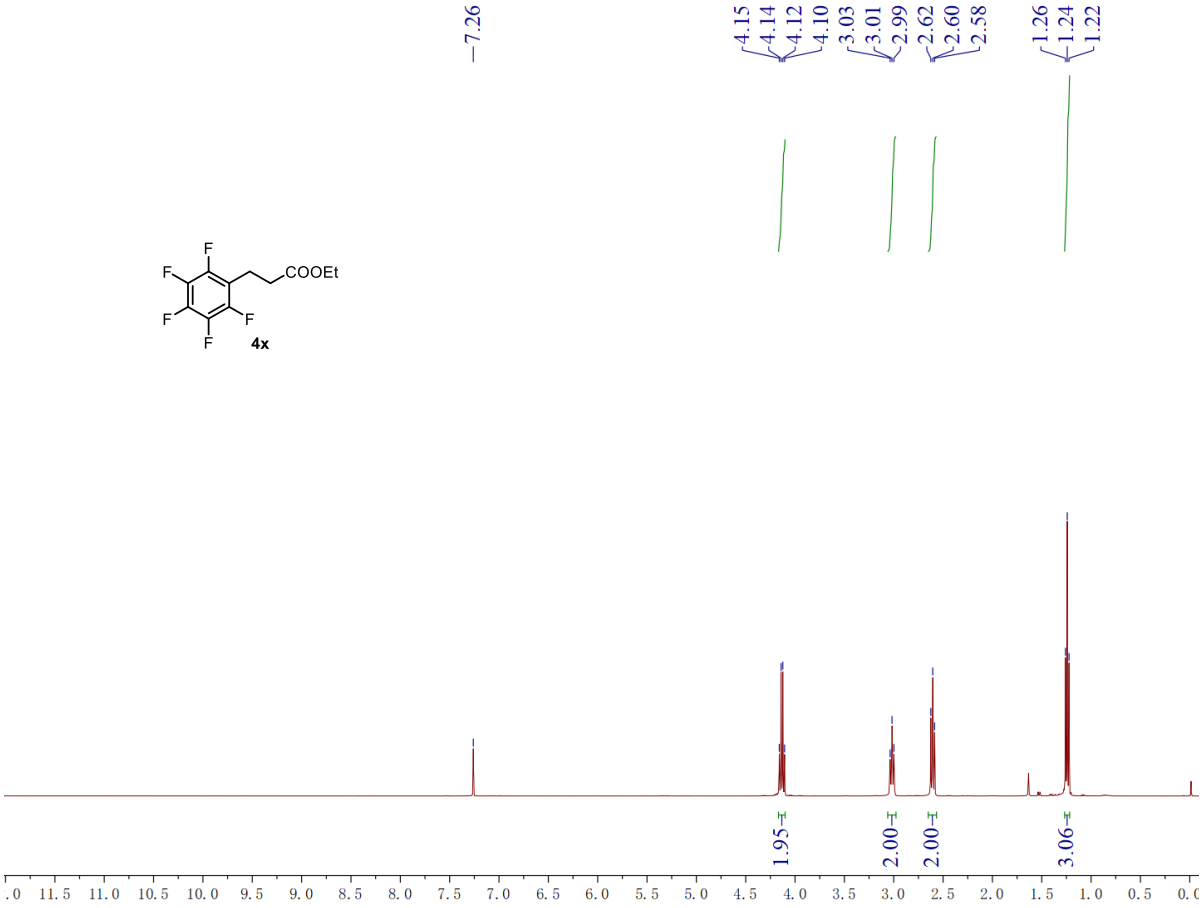
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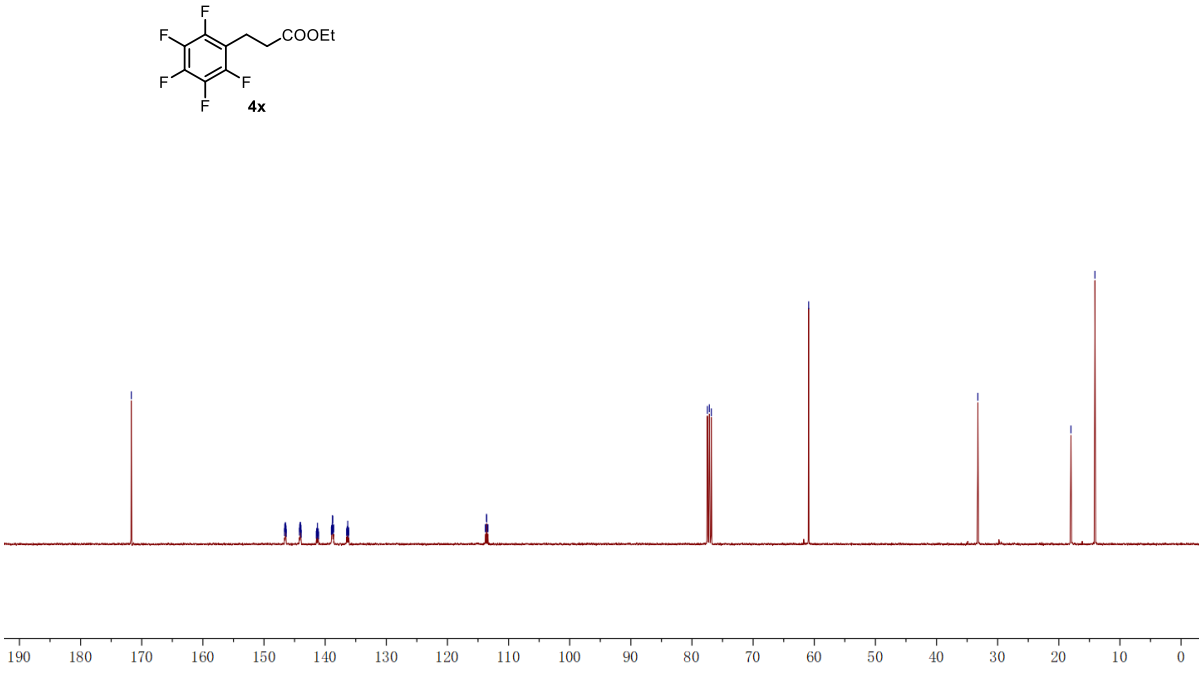
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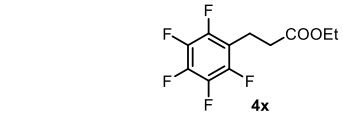
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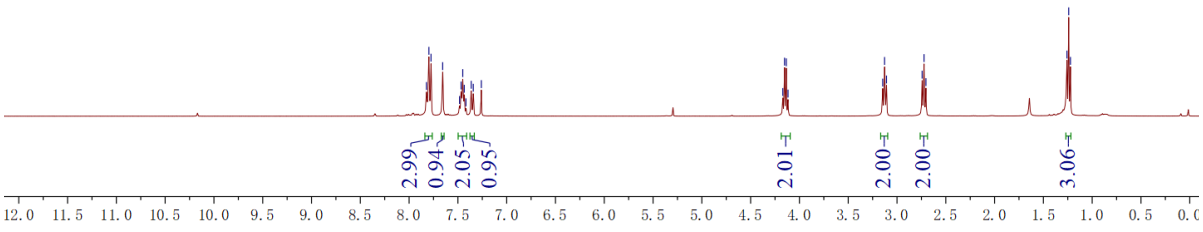
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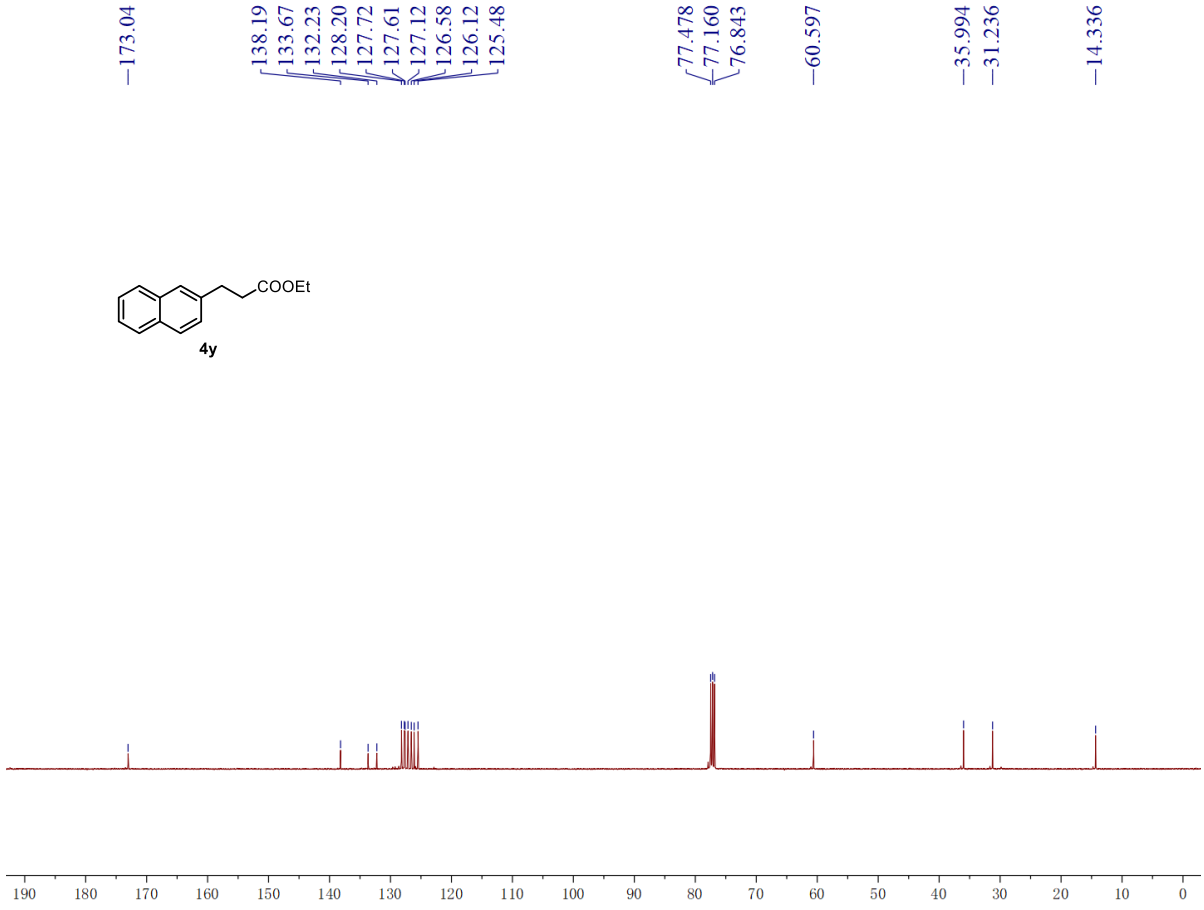
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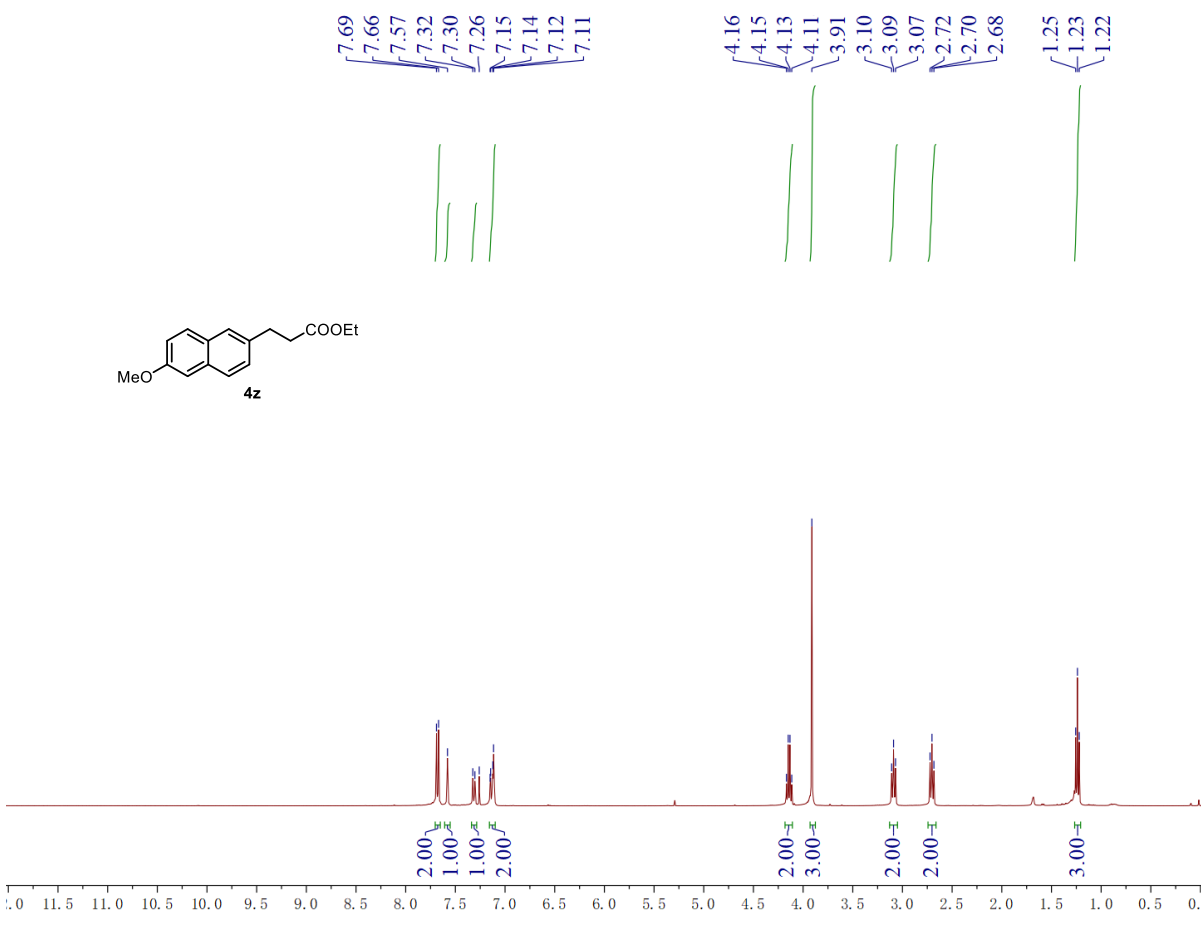
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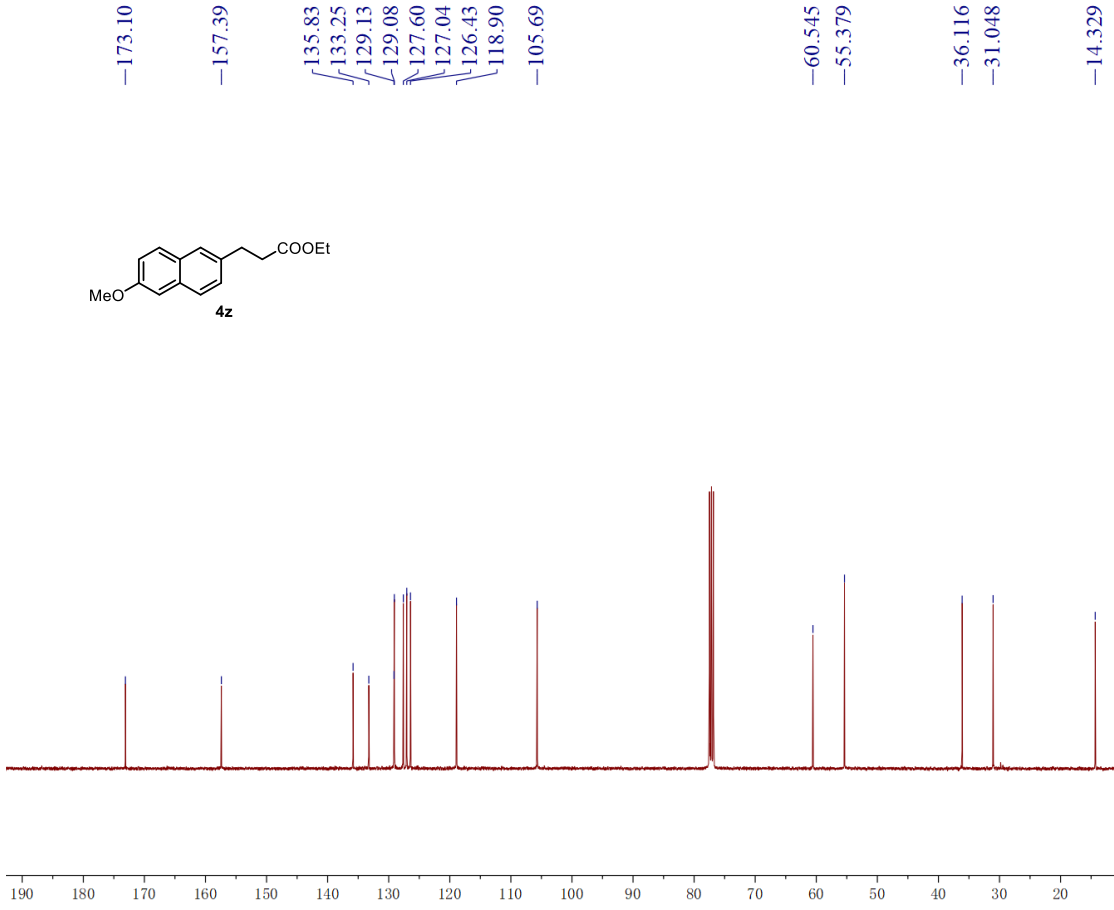
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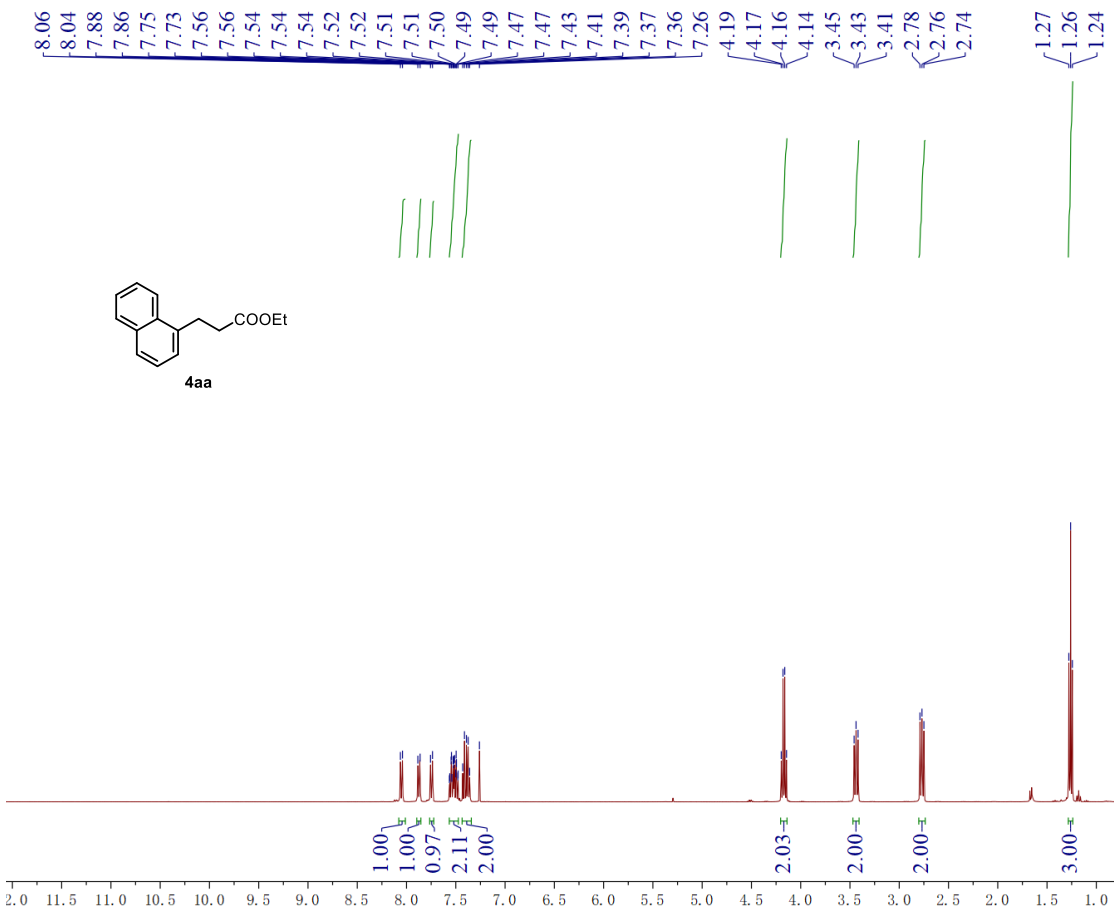
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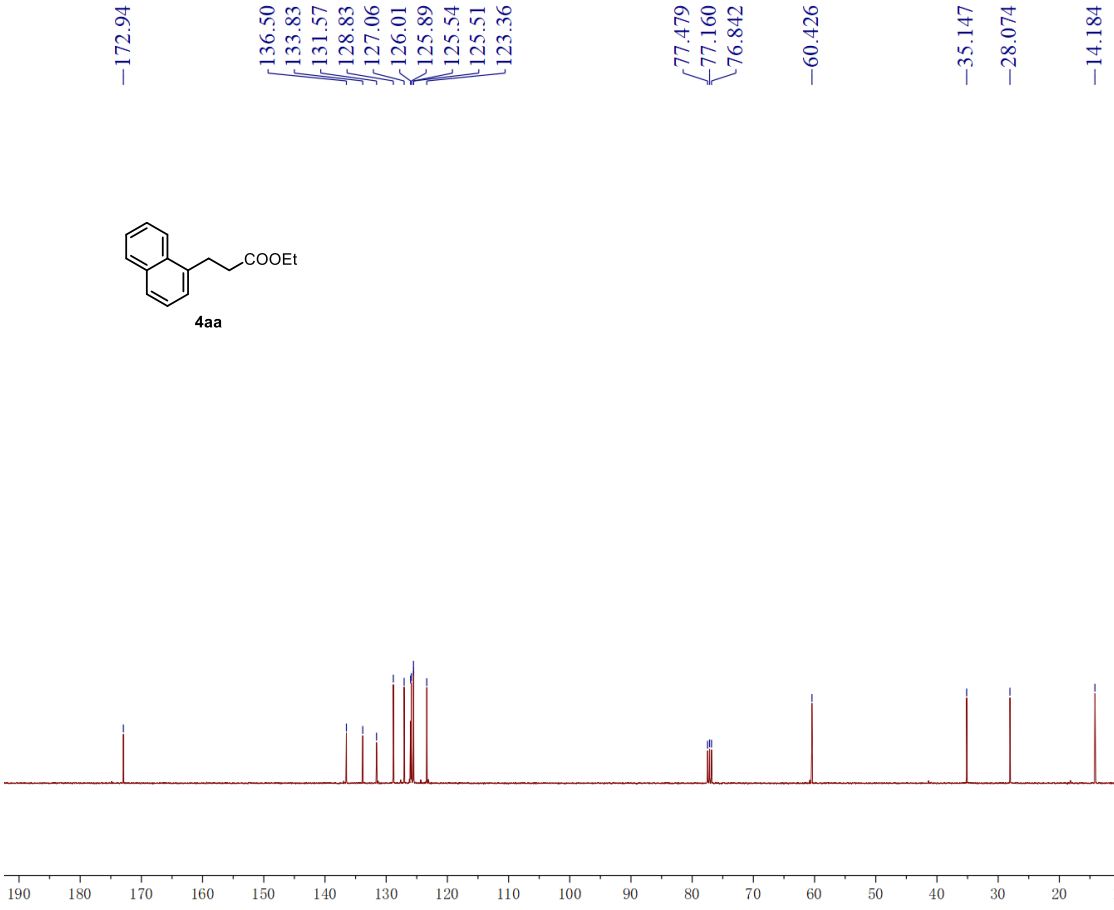
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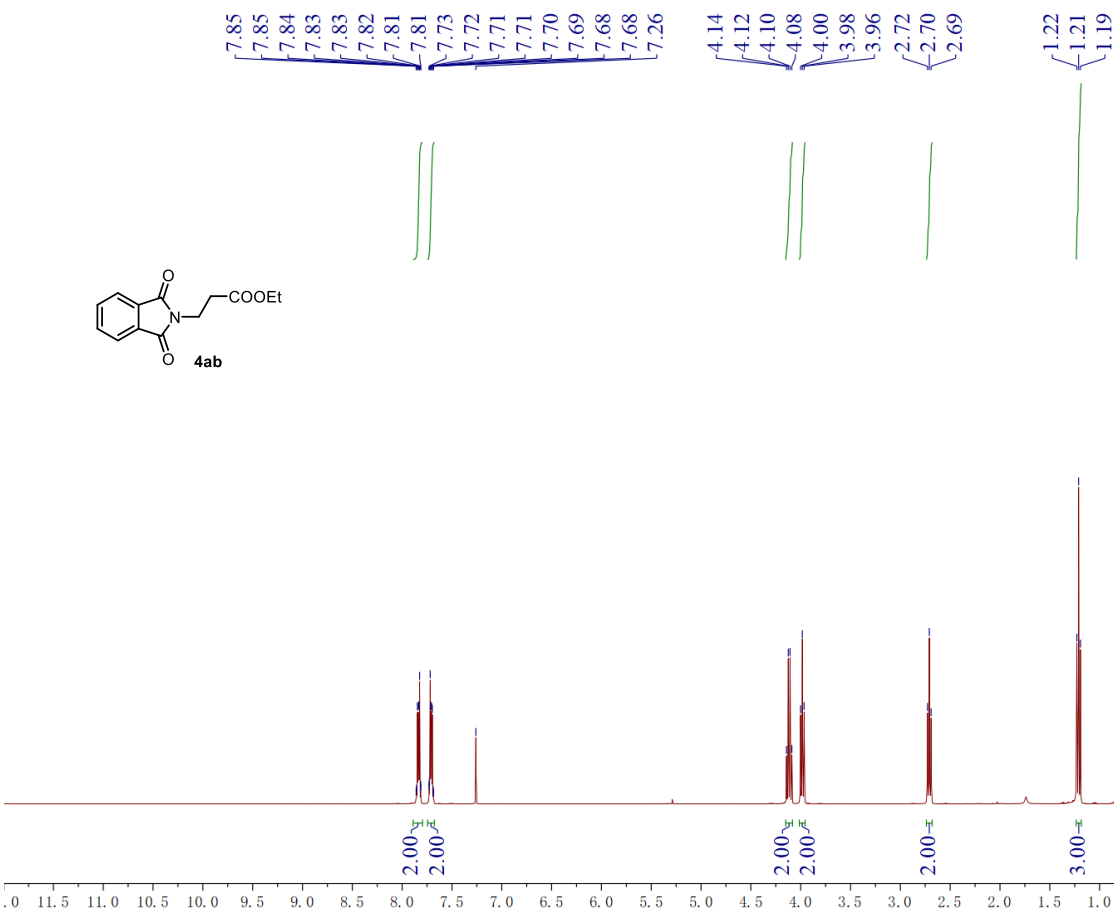
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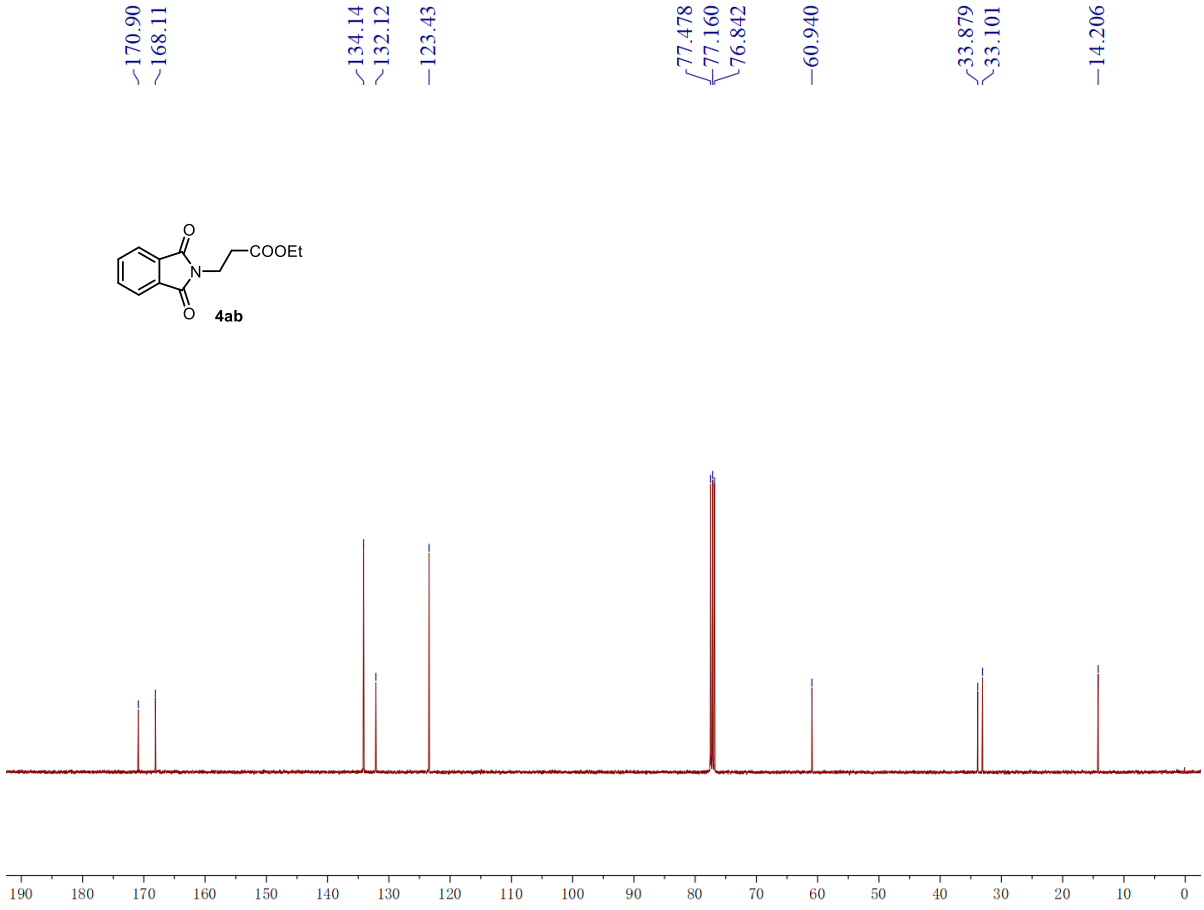
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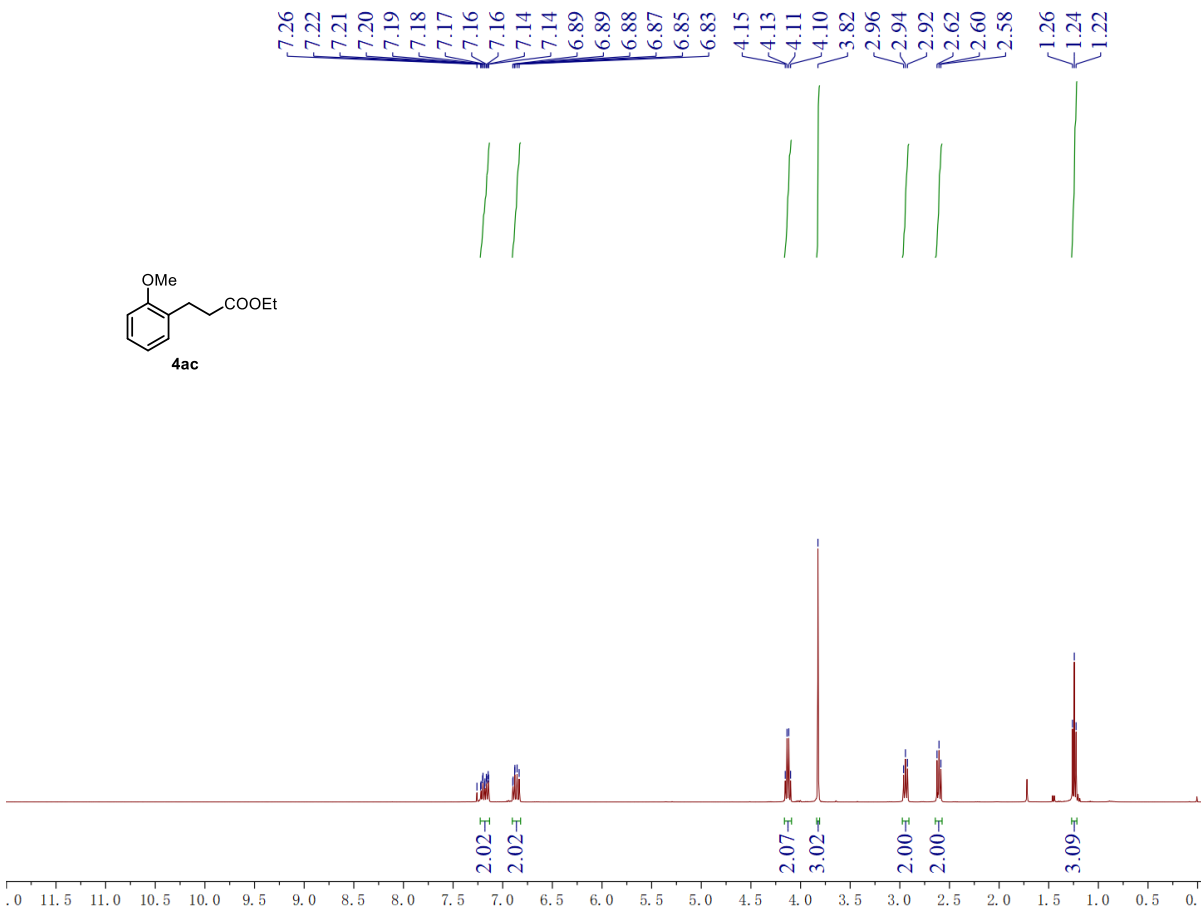
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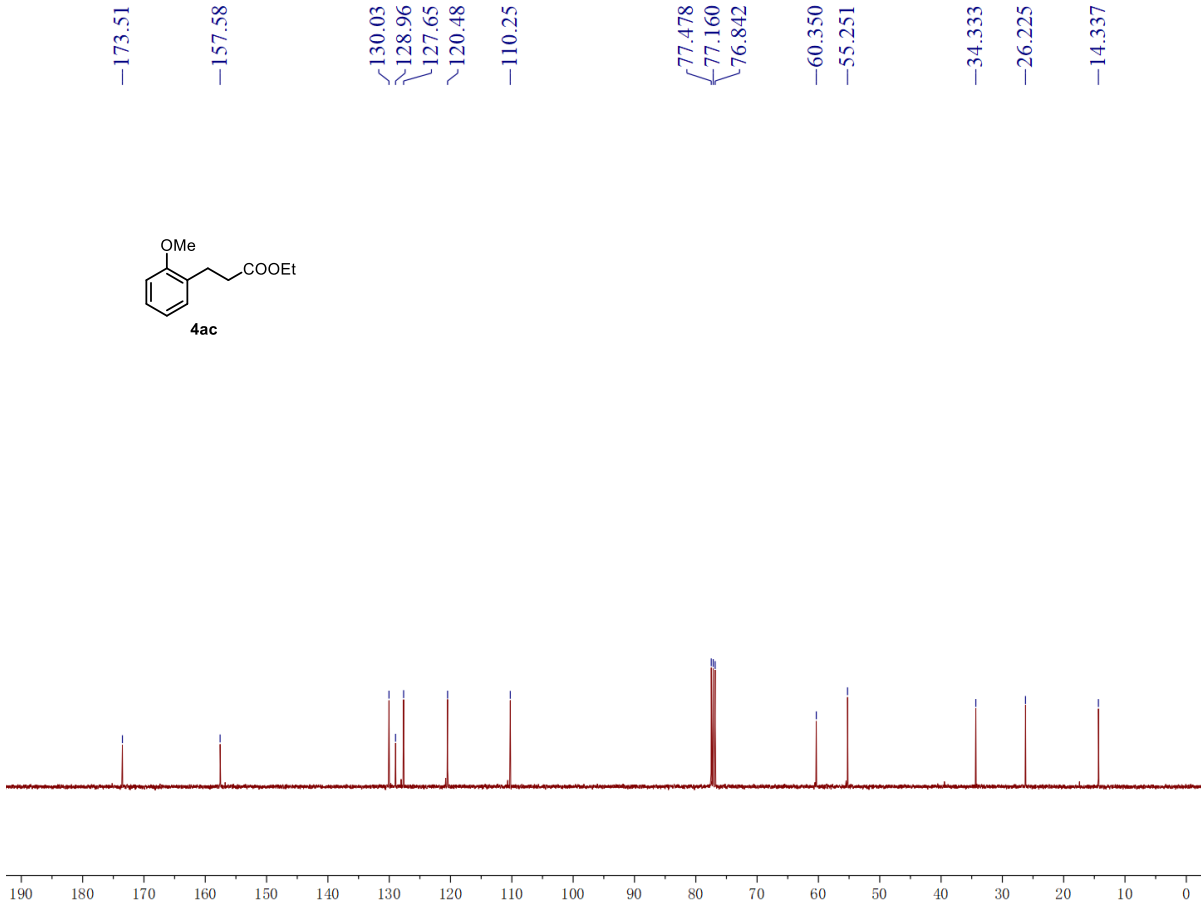
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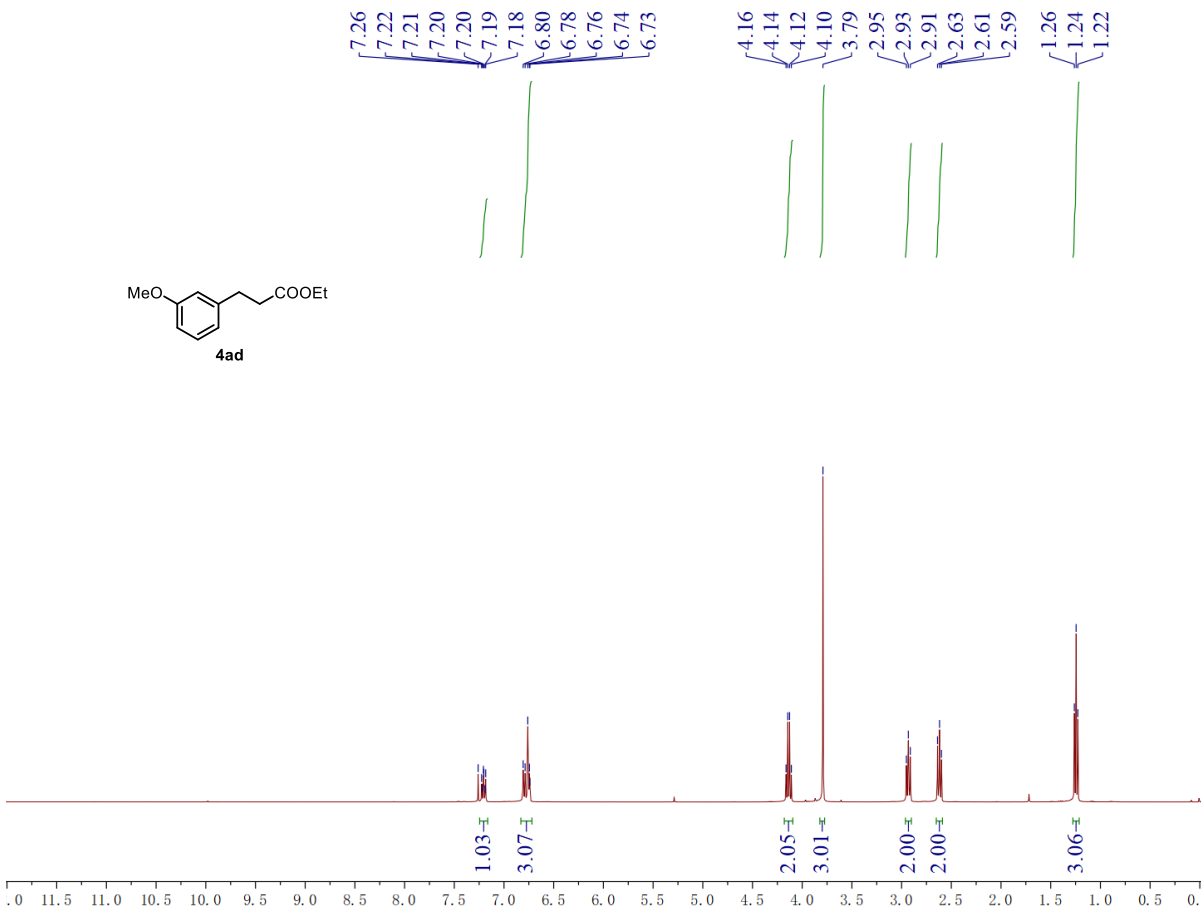
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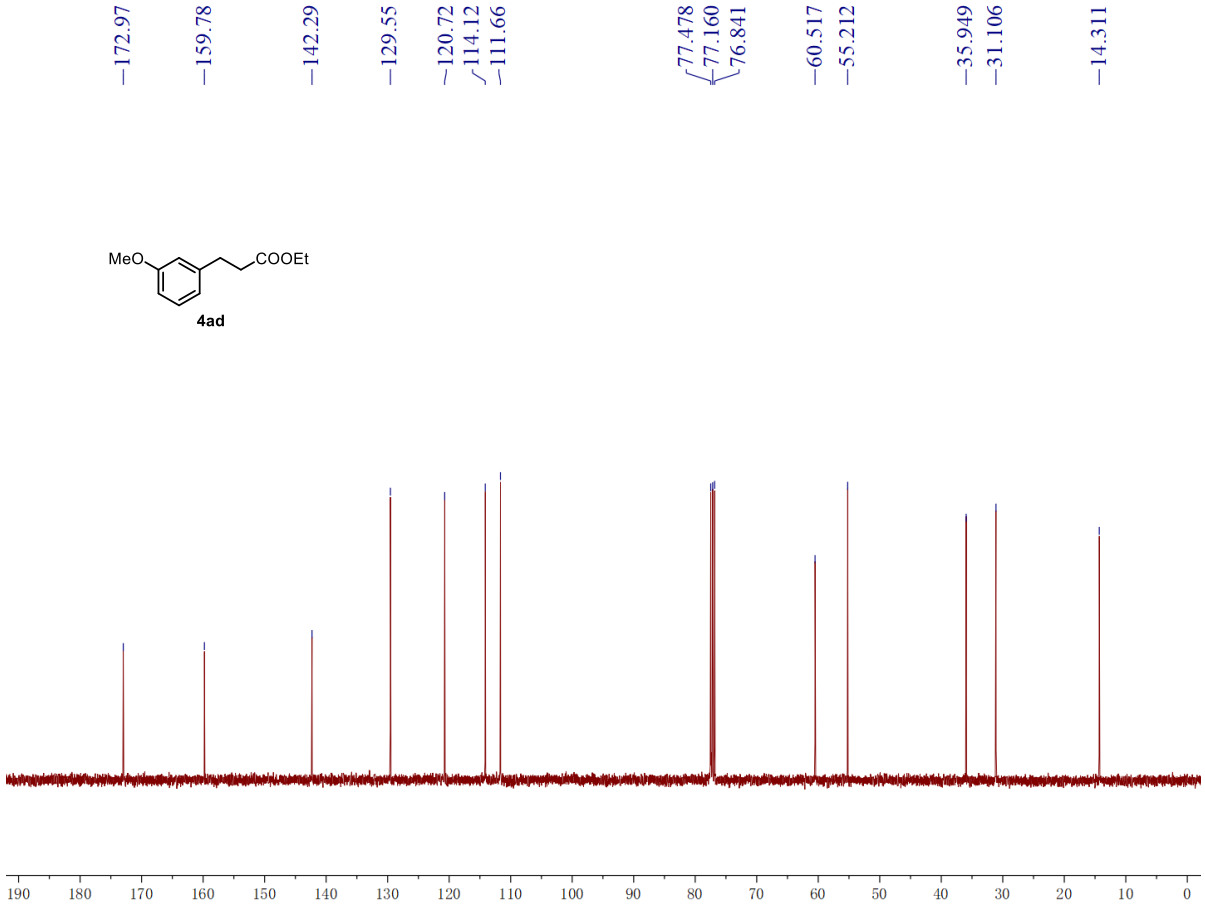
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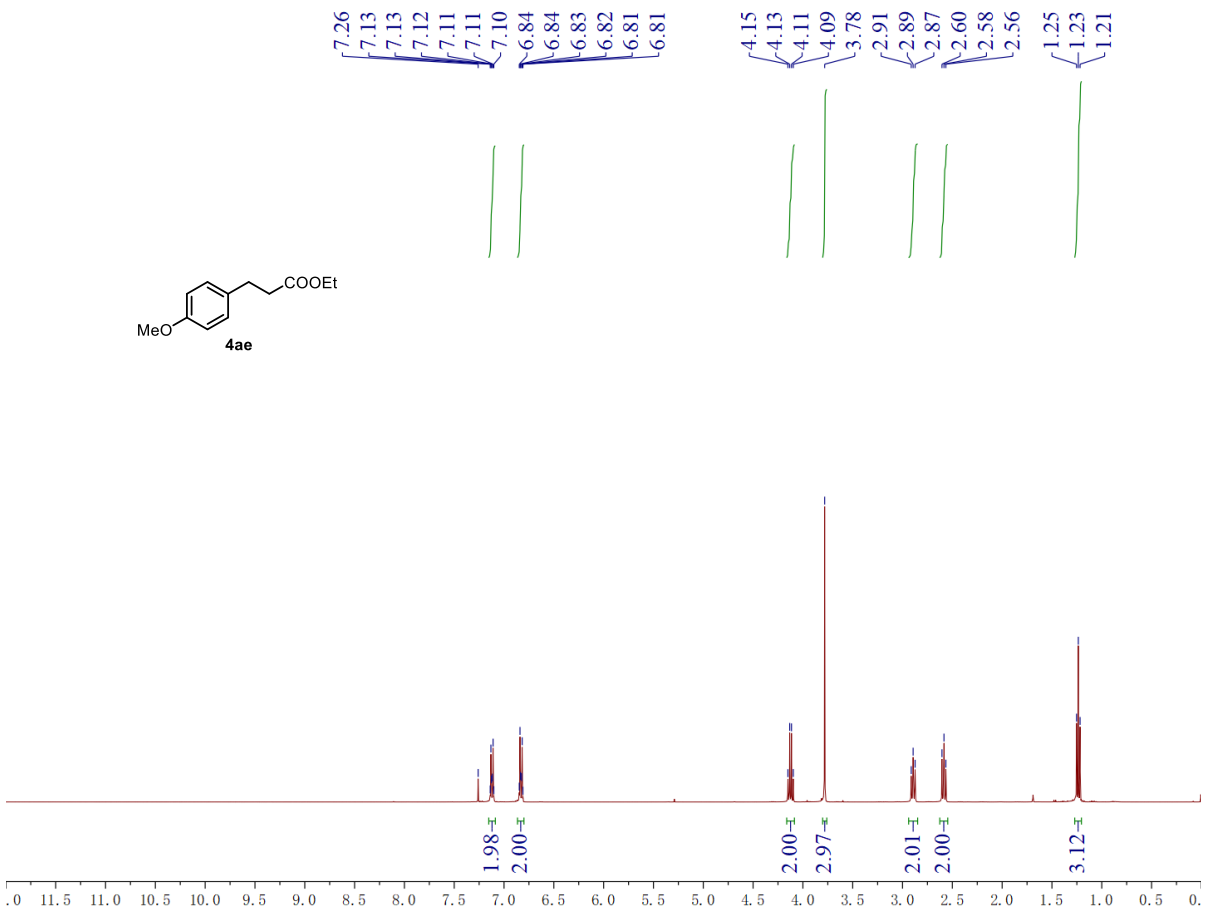
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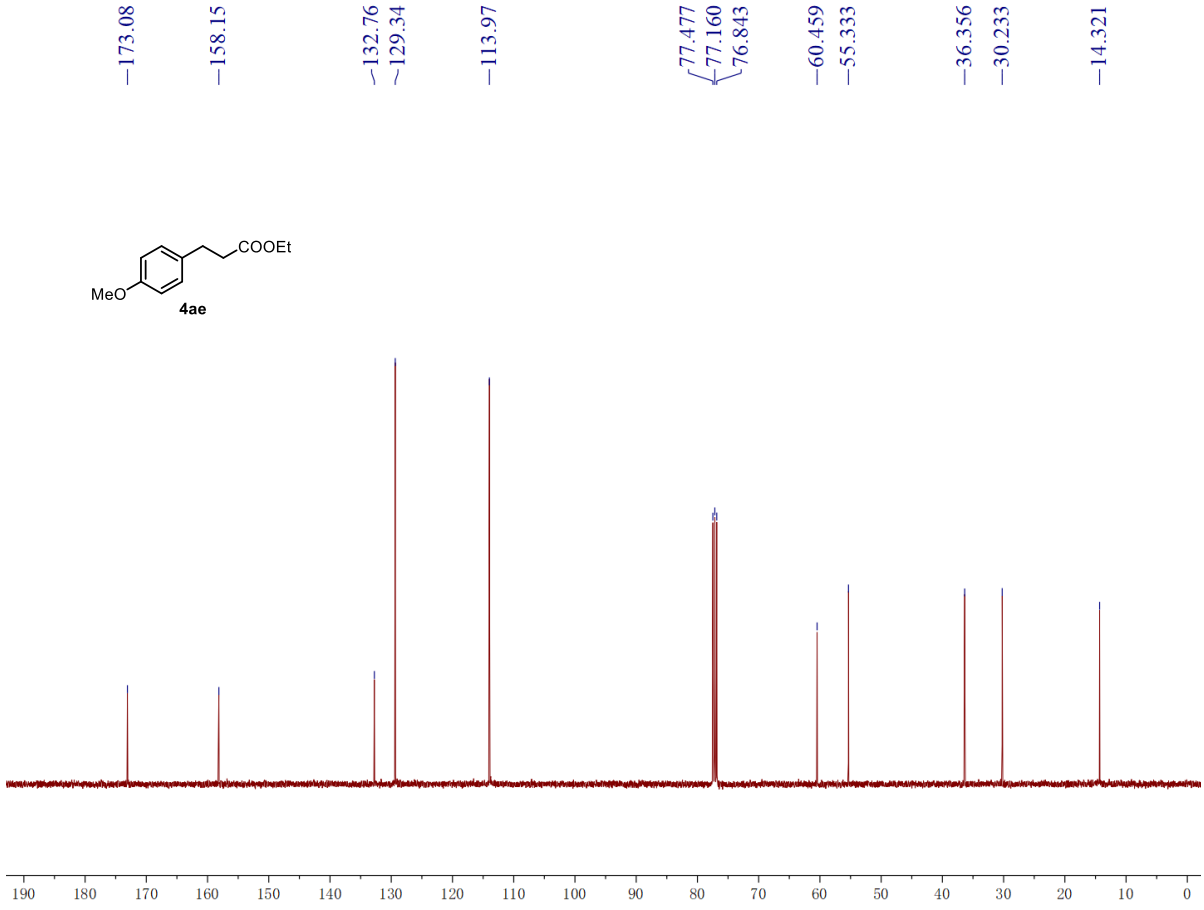
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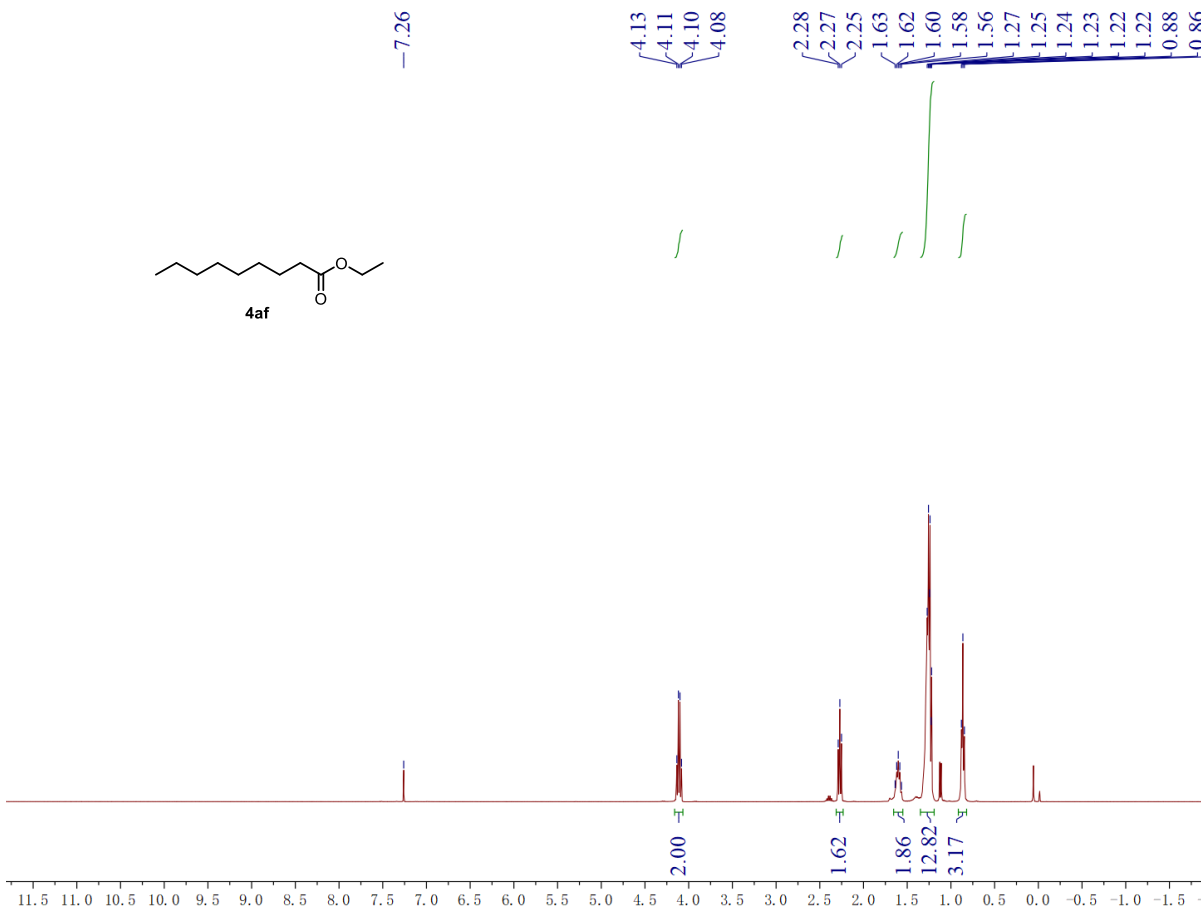
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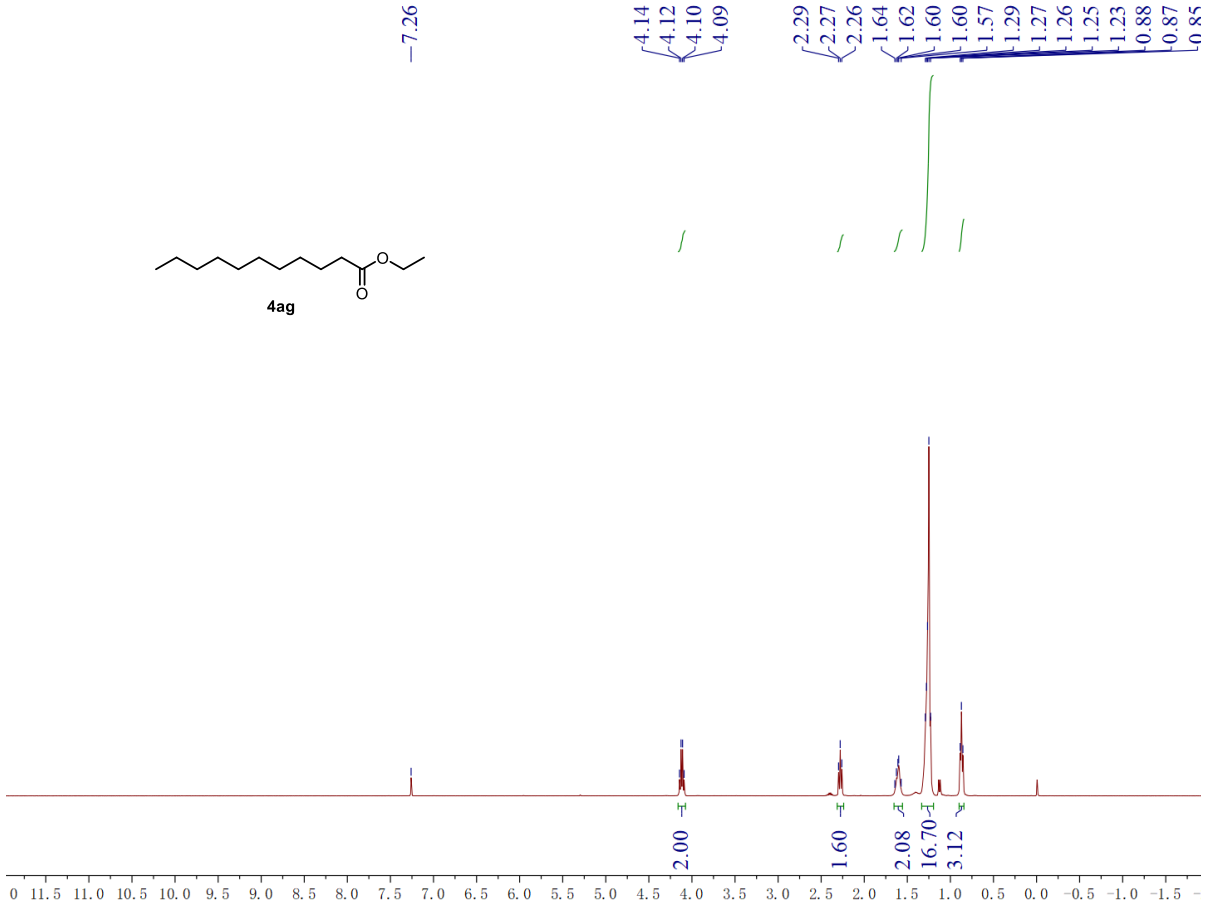
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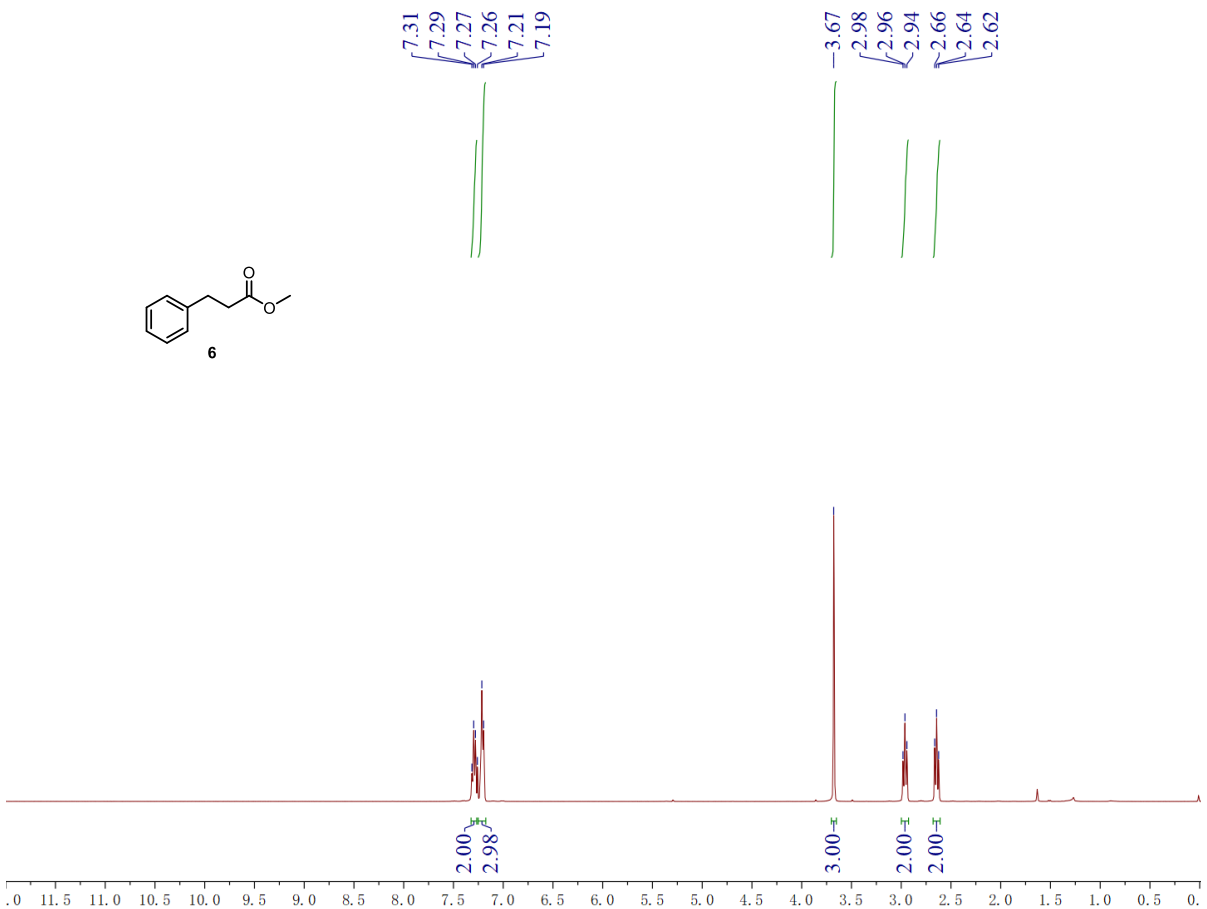
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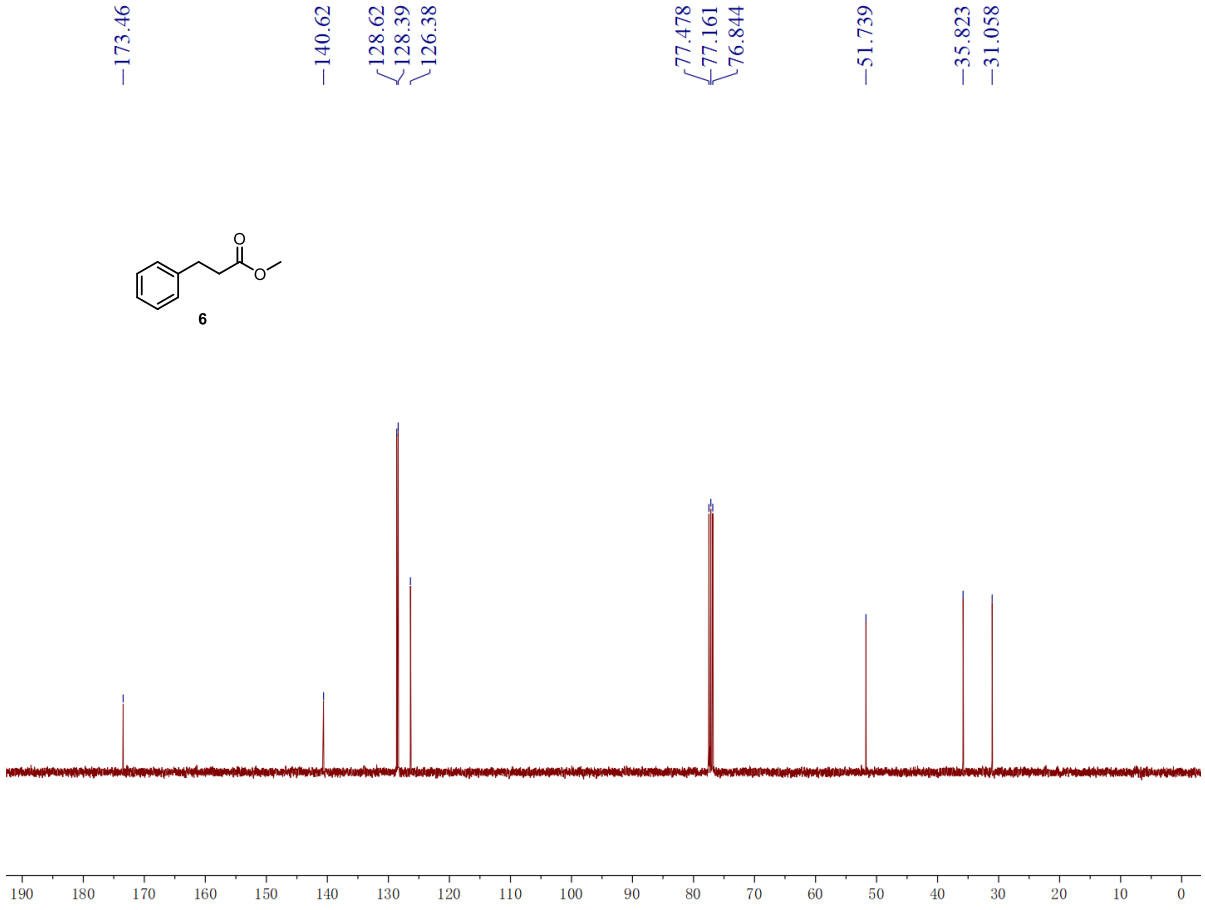
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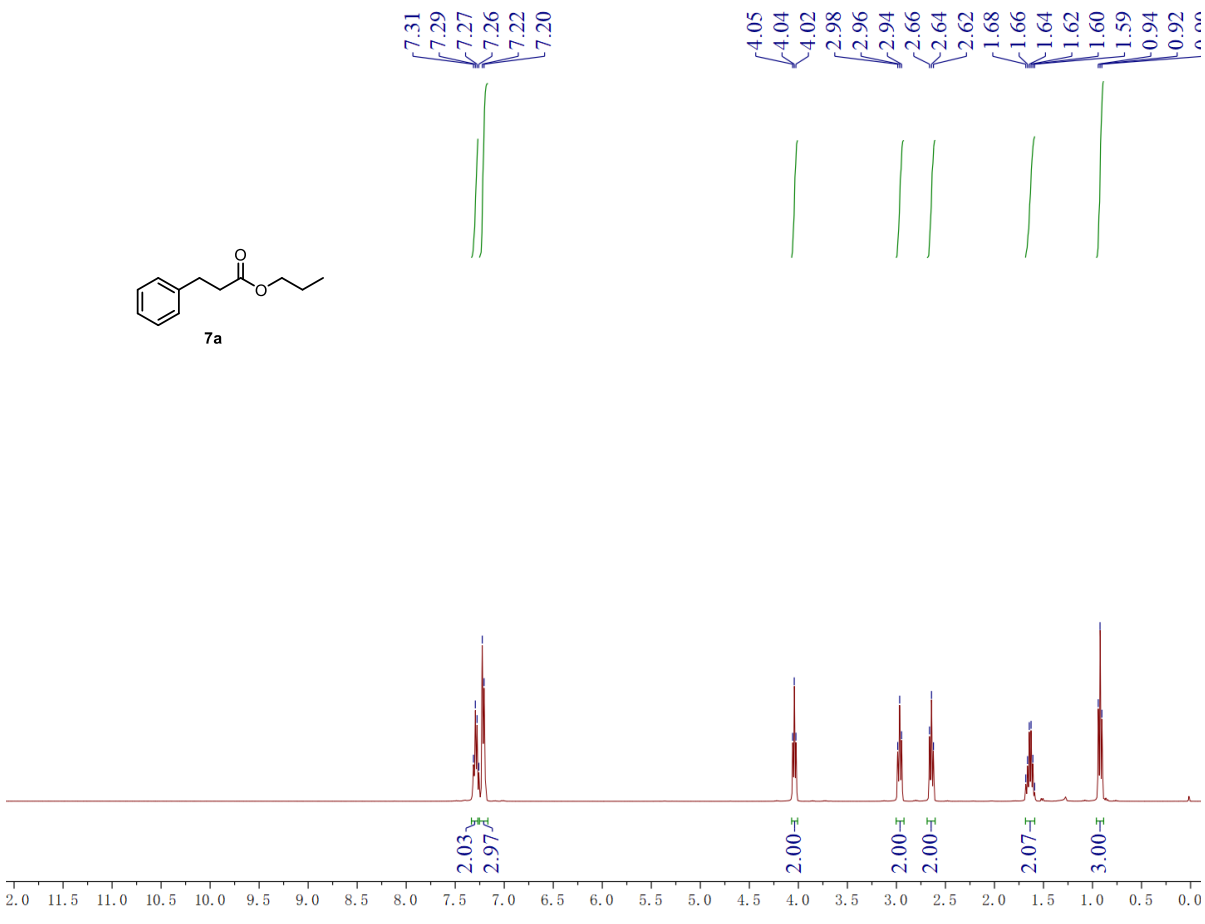
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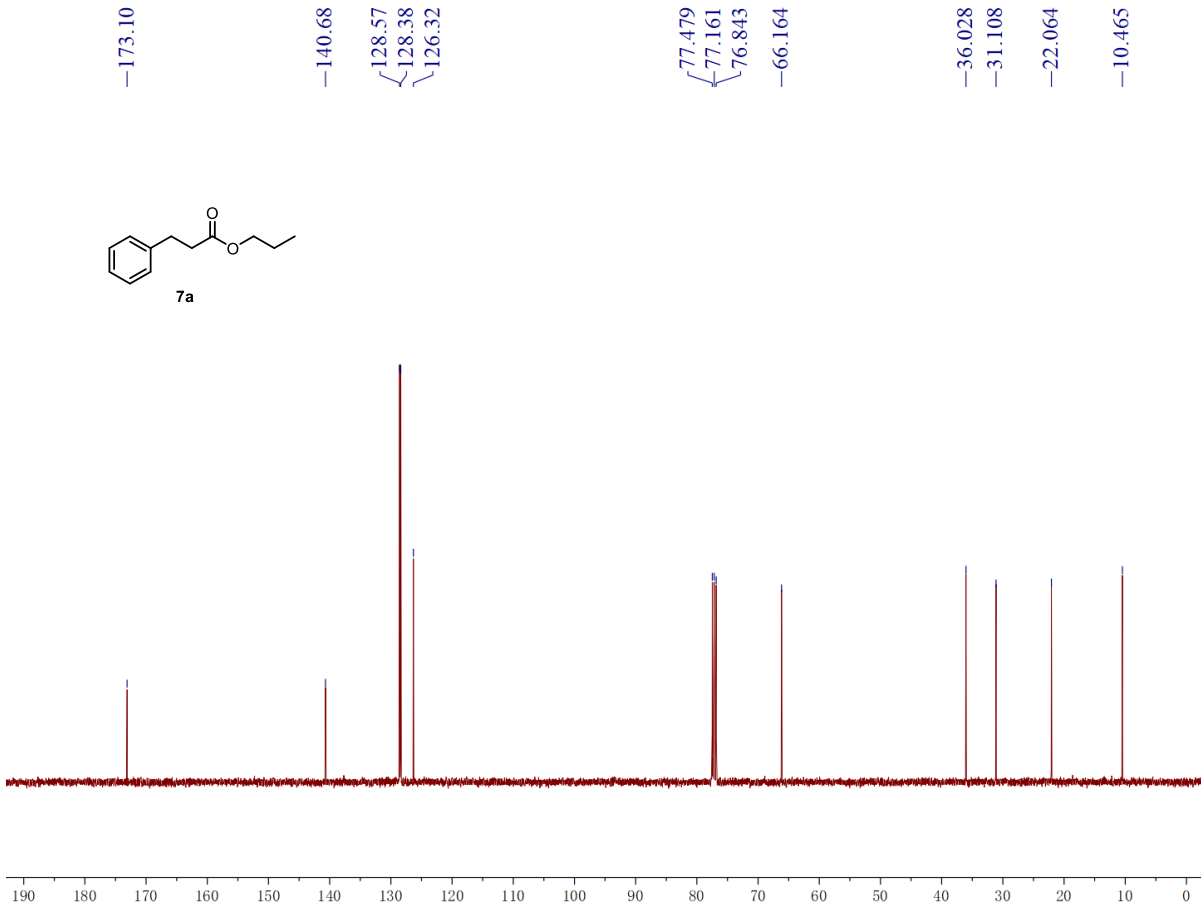
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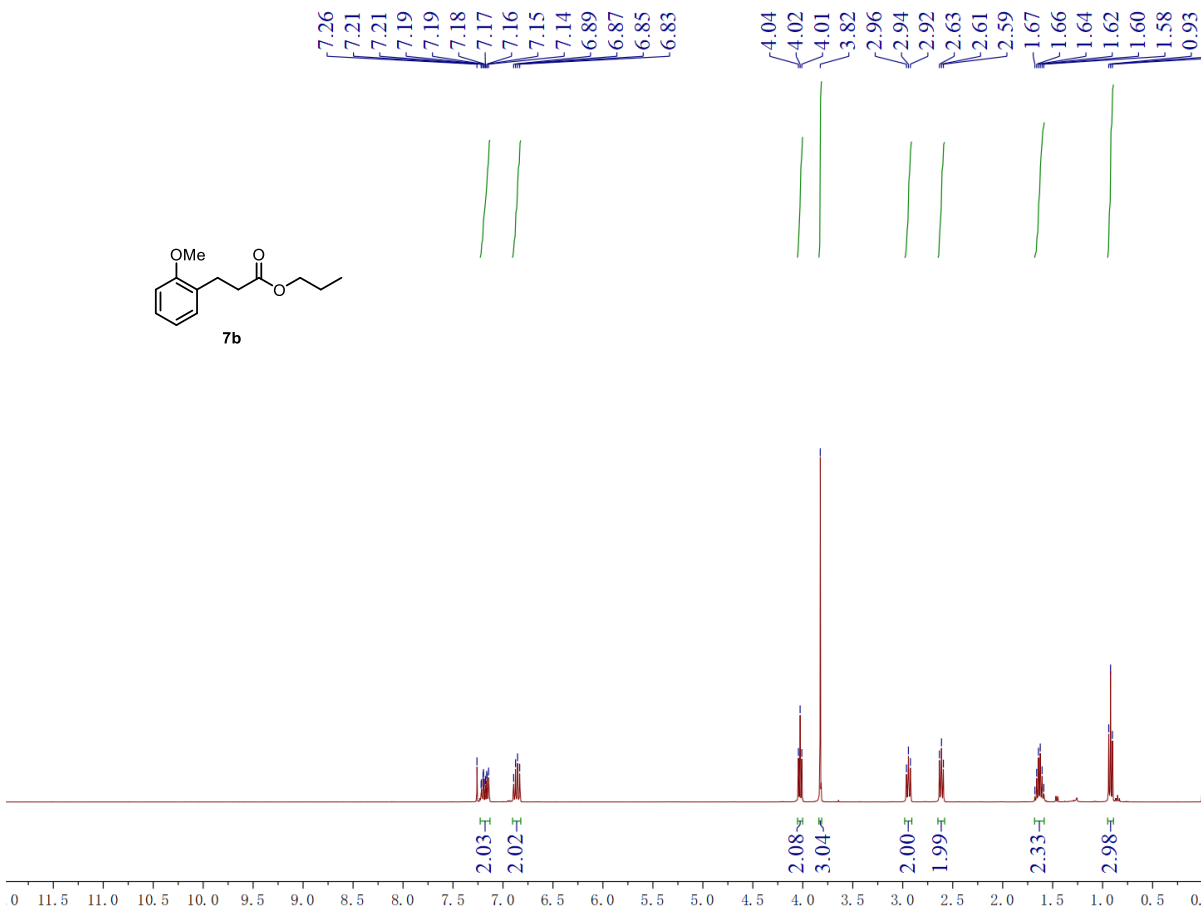
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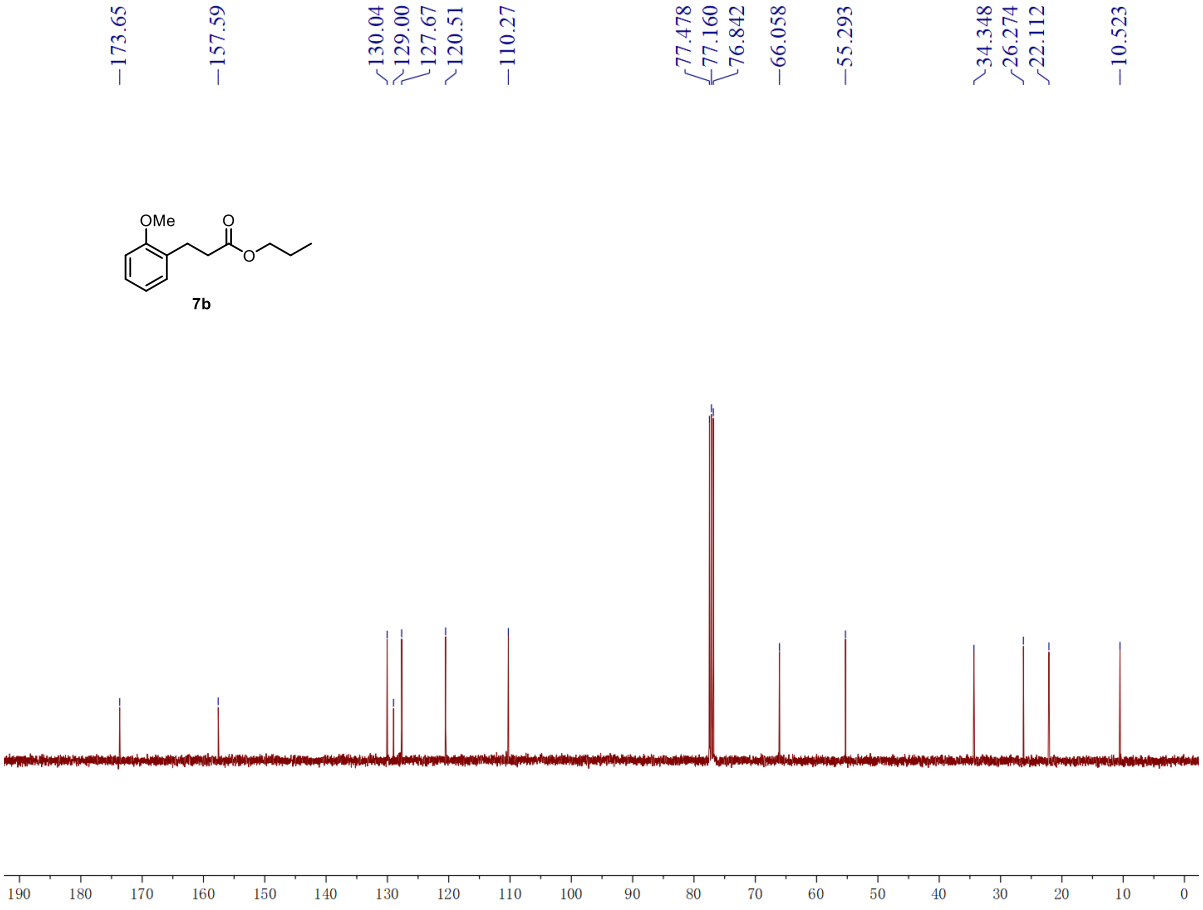
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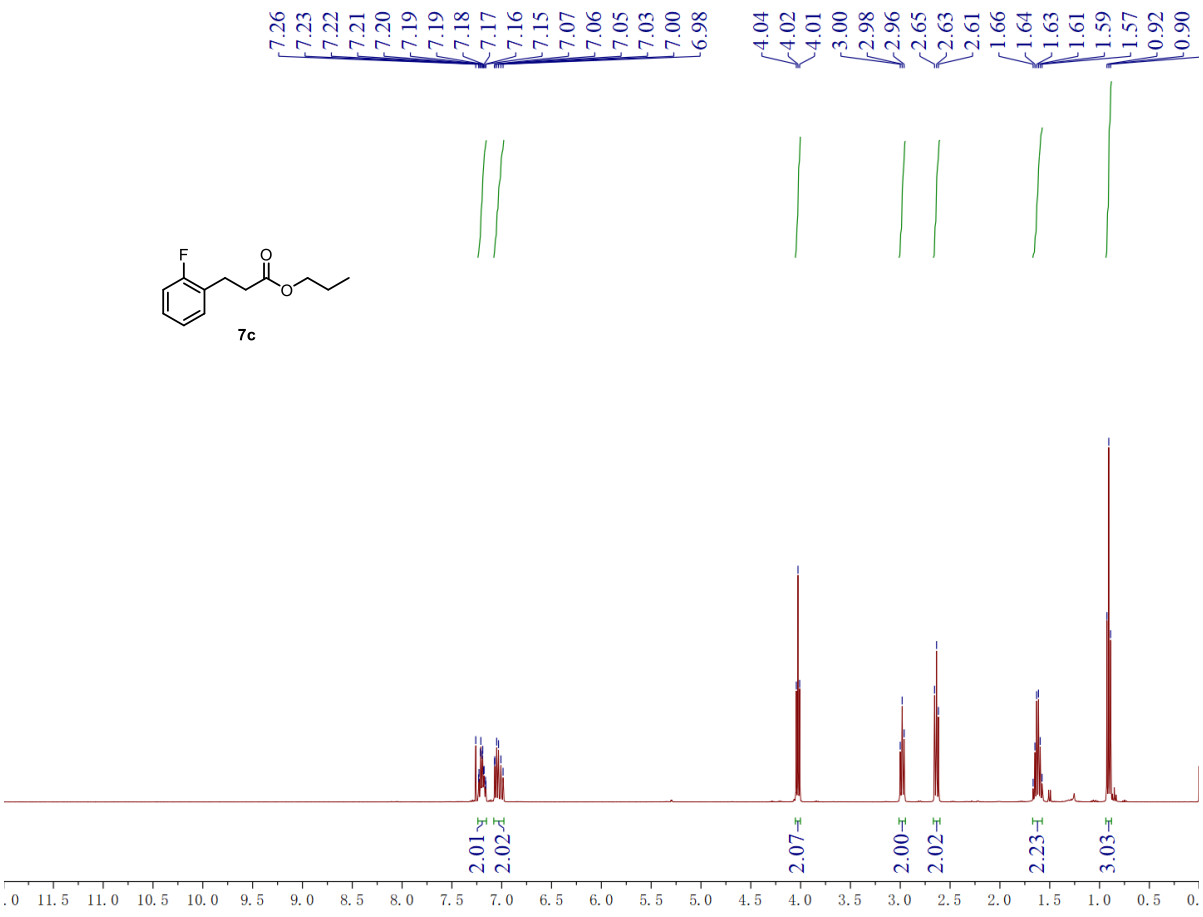
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7c
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2.07
2.00
2.02
2.23
3.03
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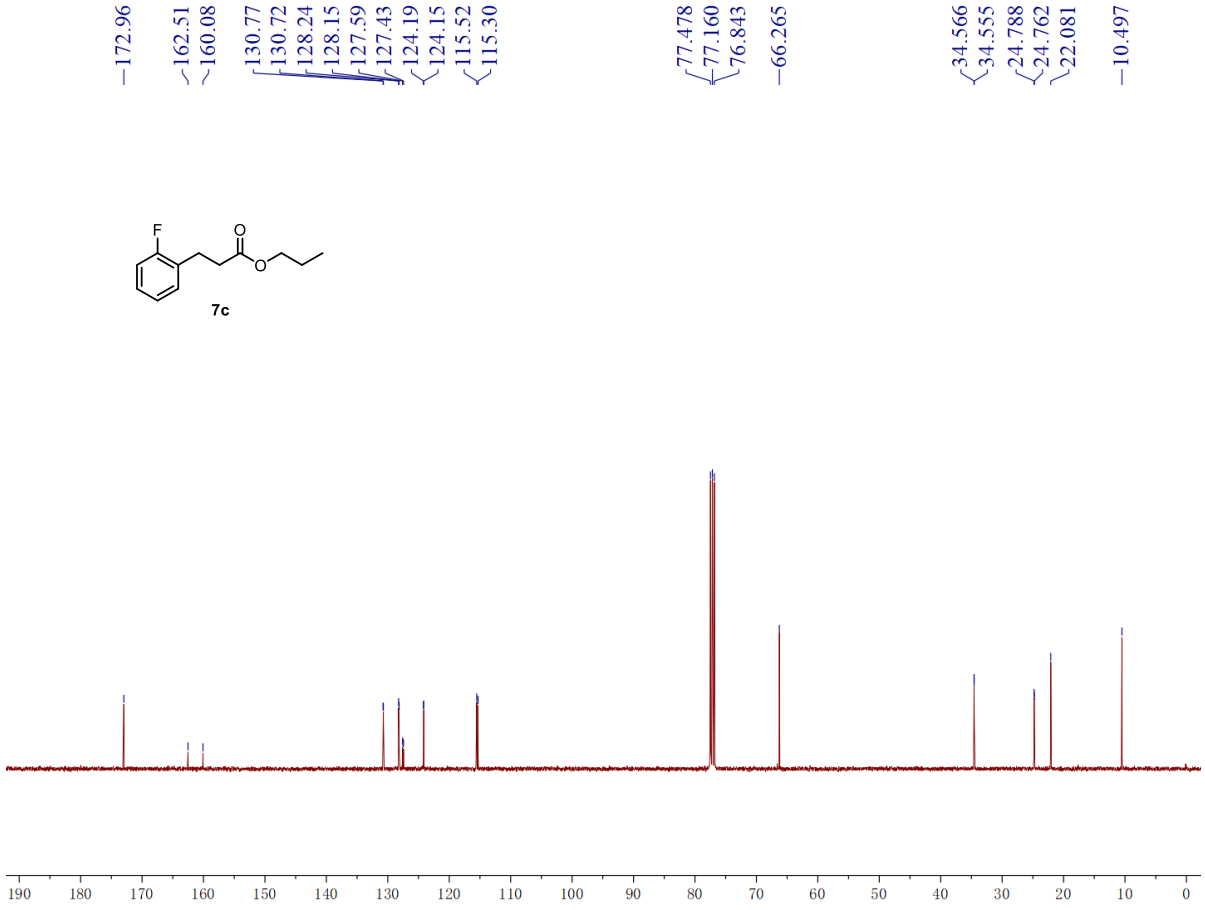
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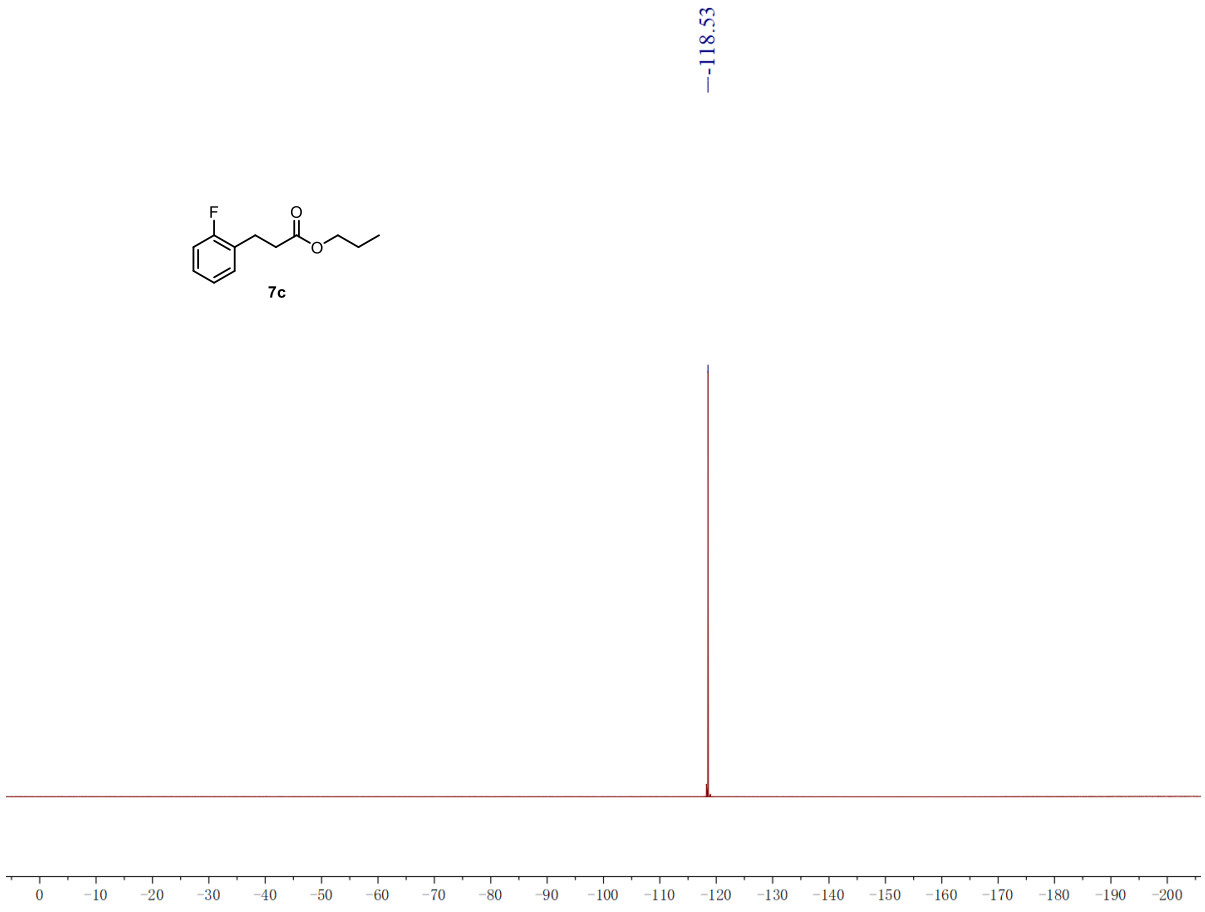
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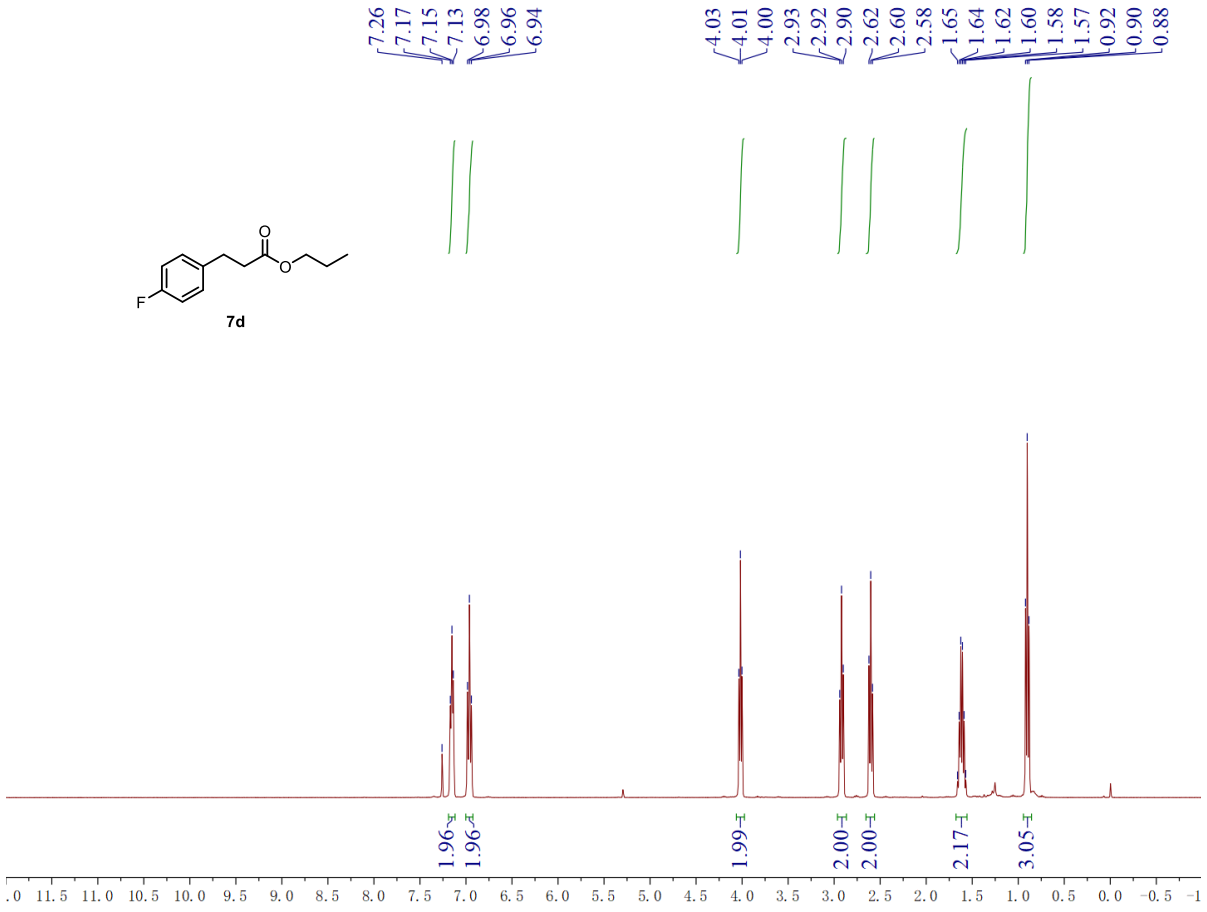
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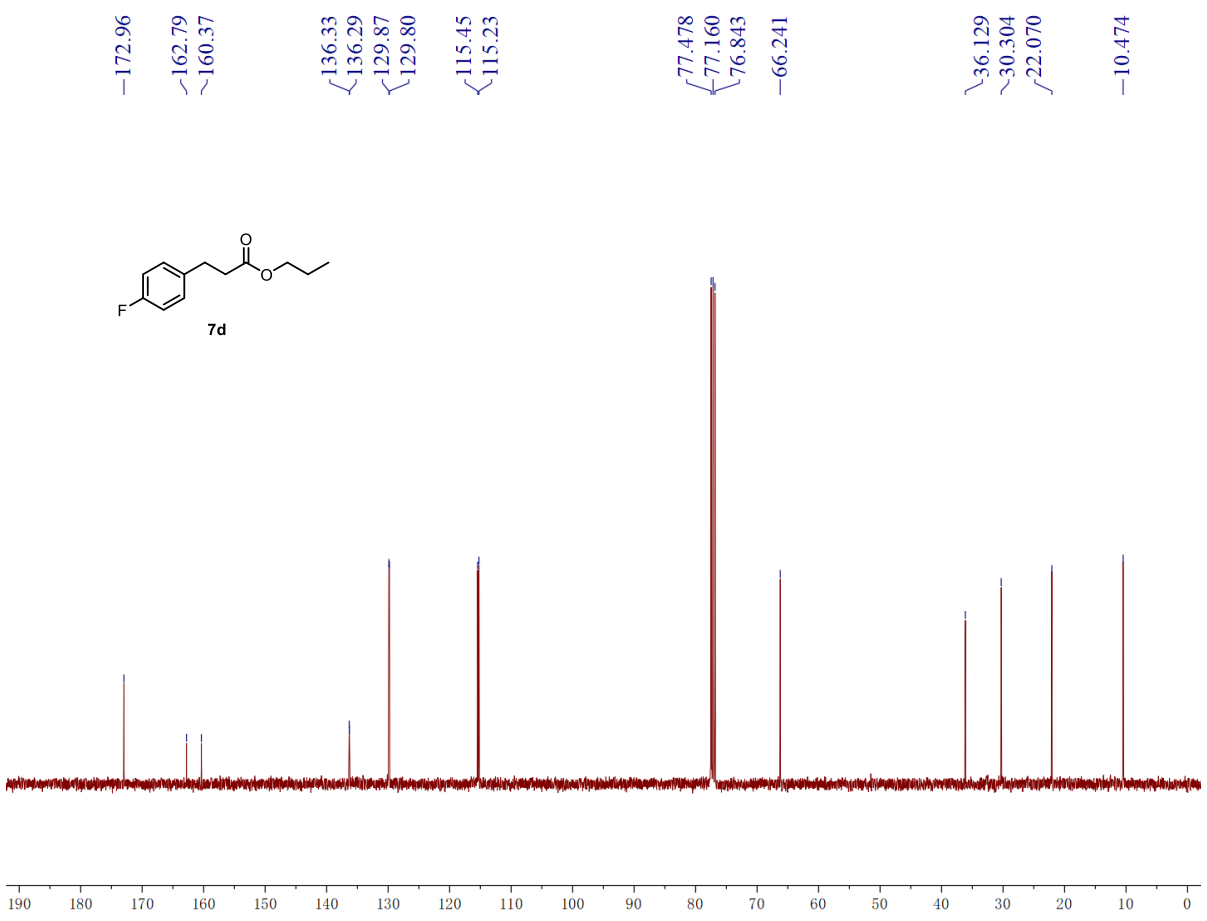
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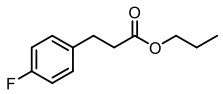

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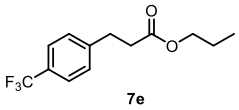

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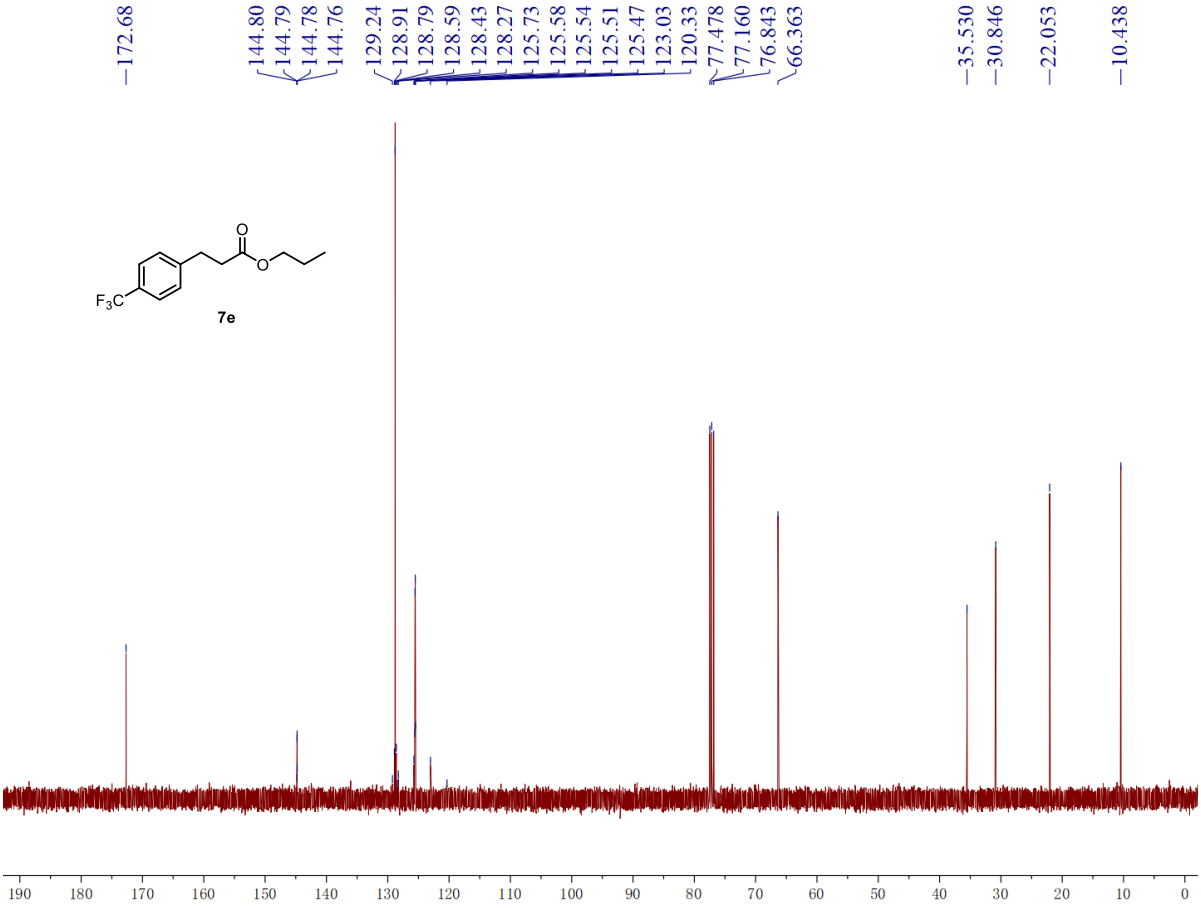
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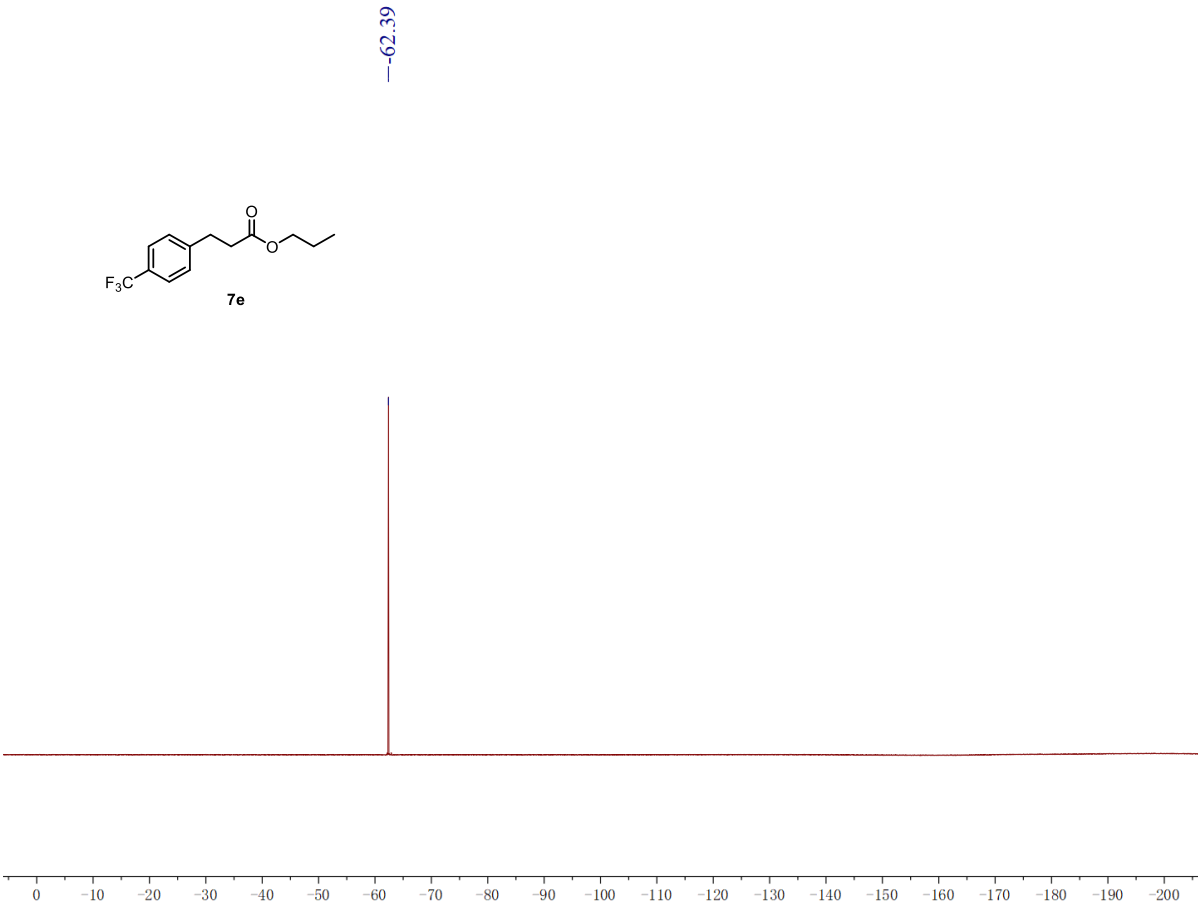
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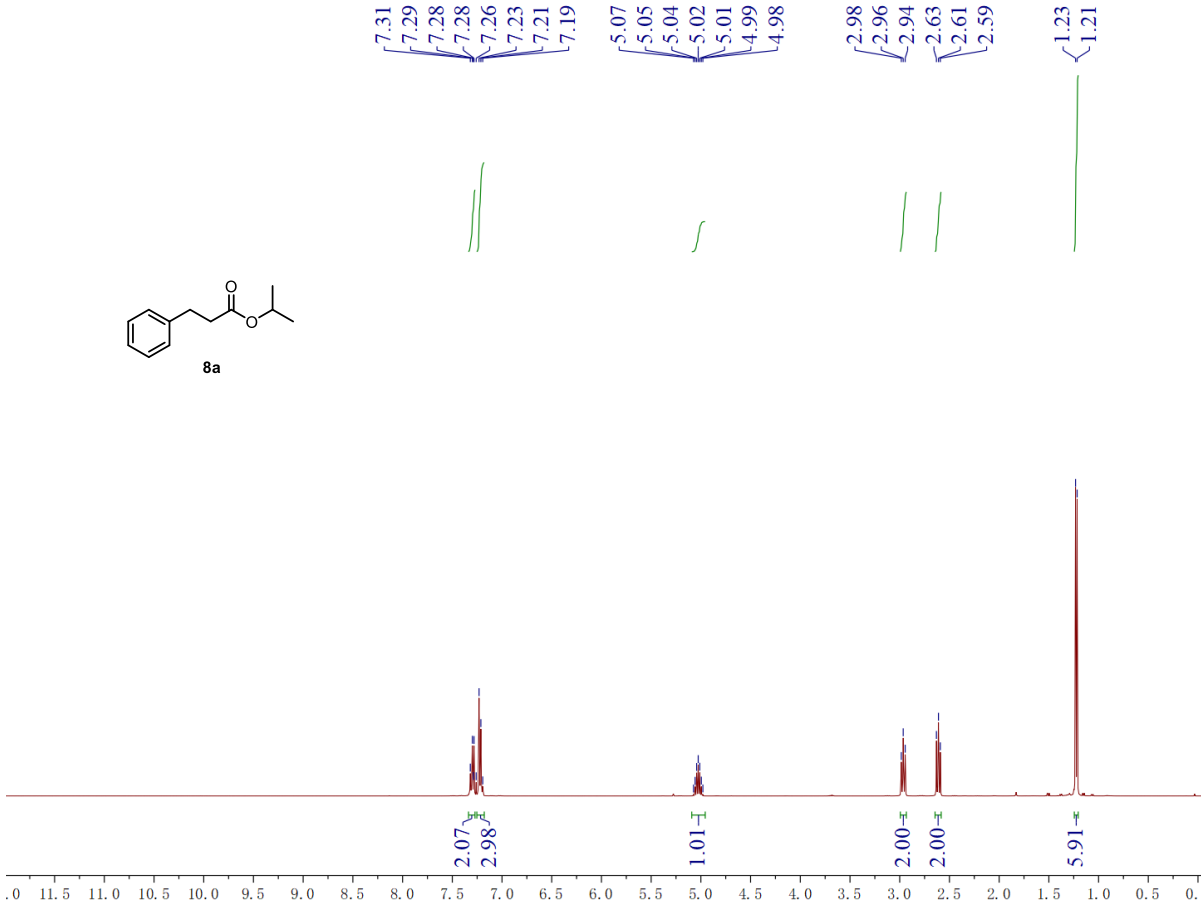
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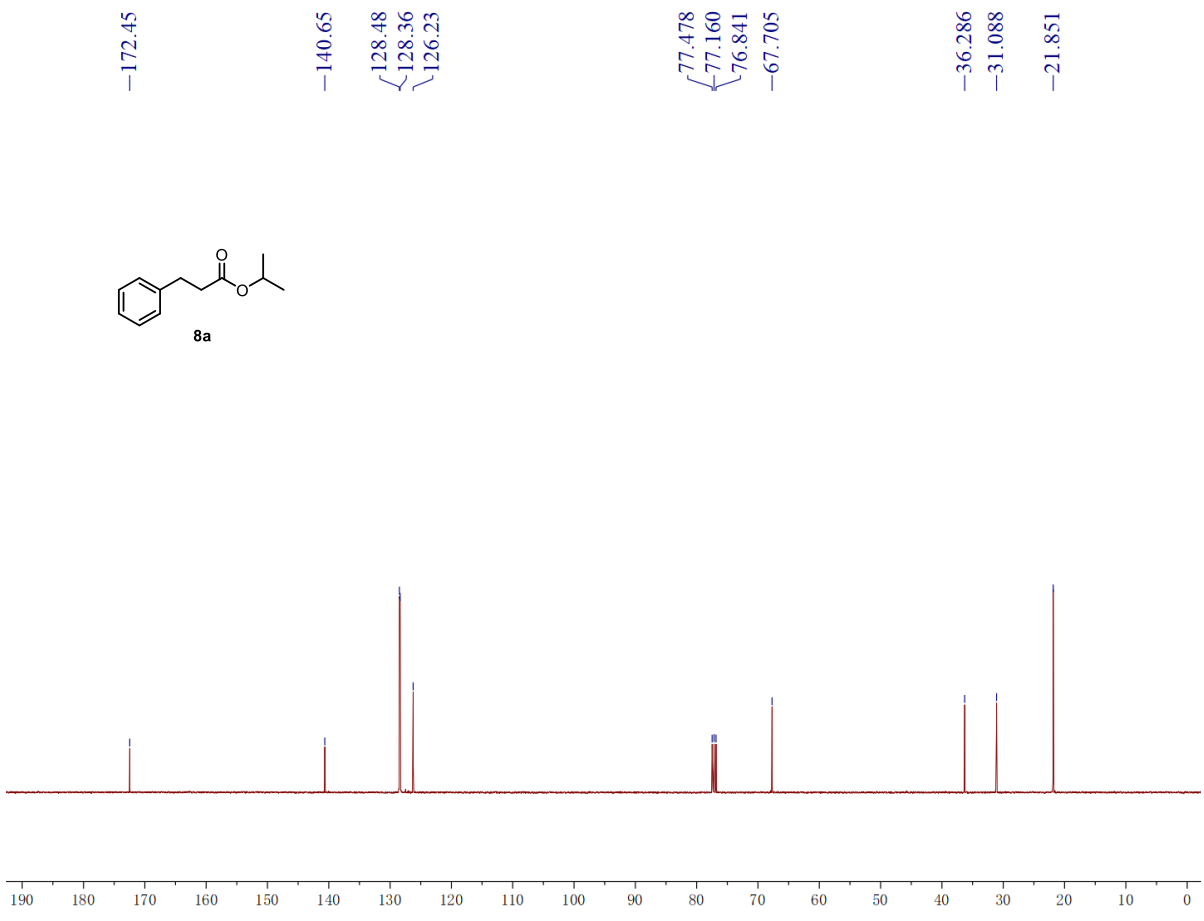
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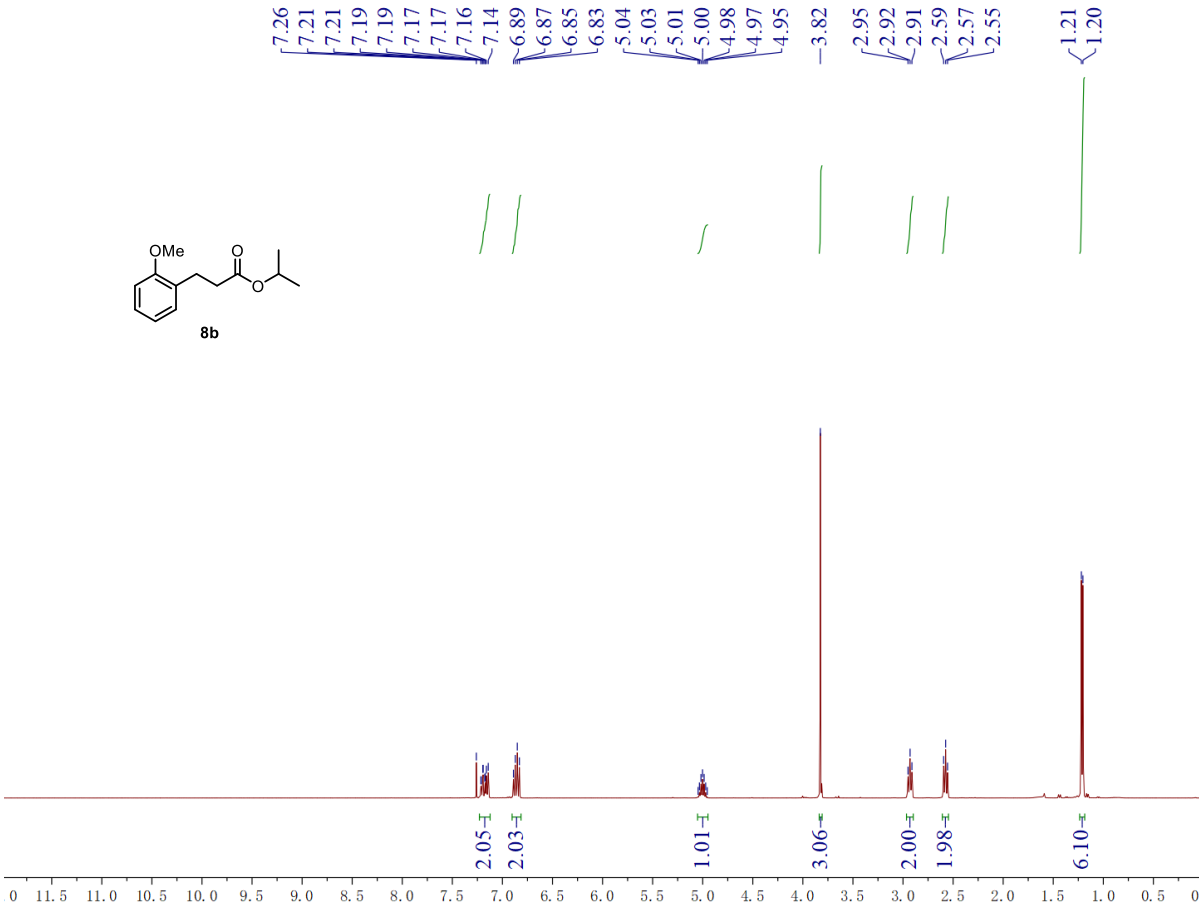
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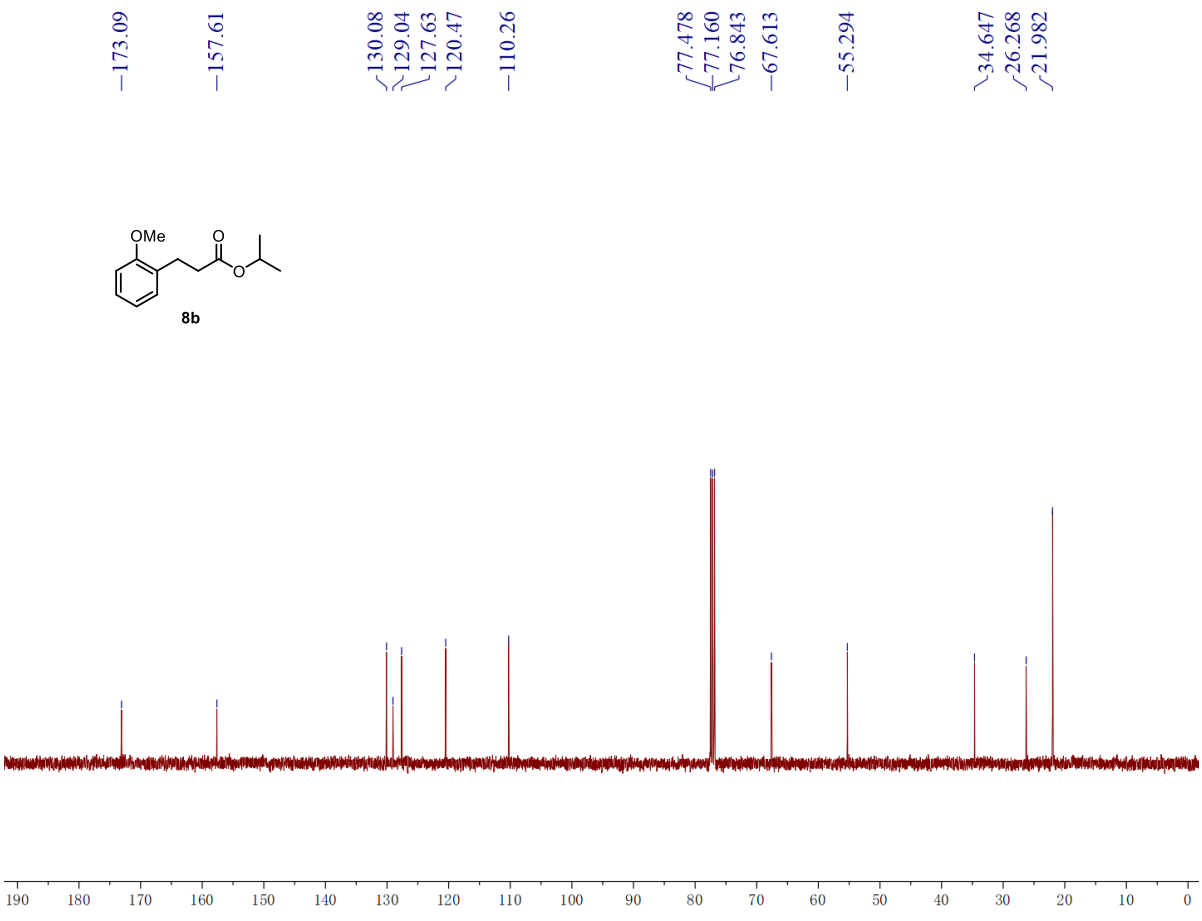
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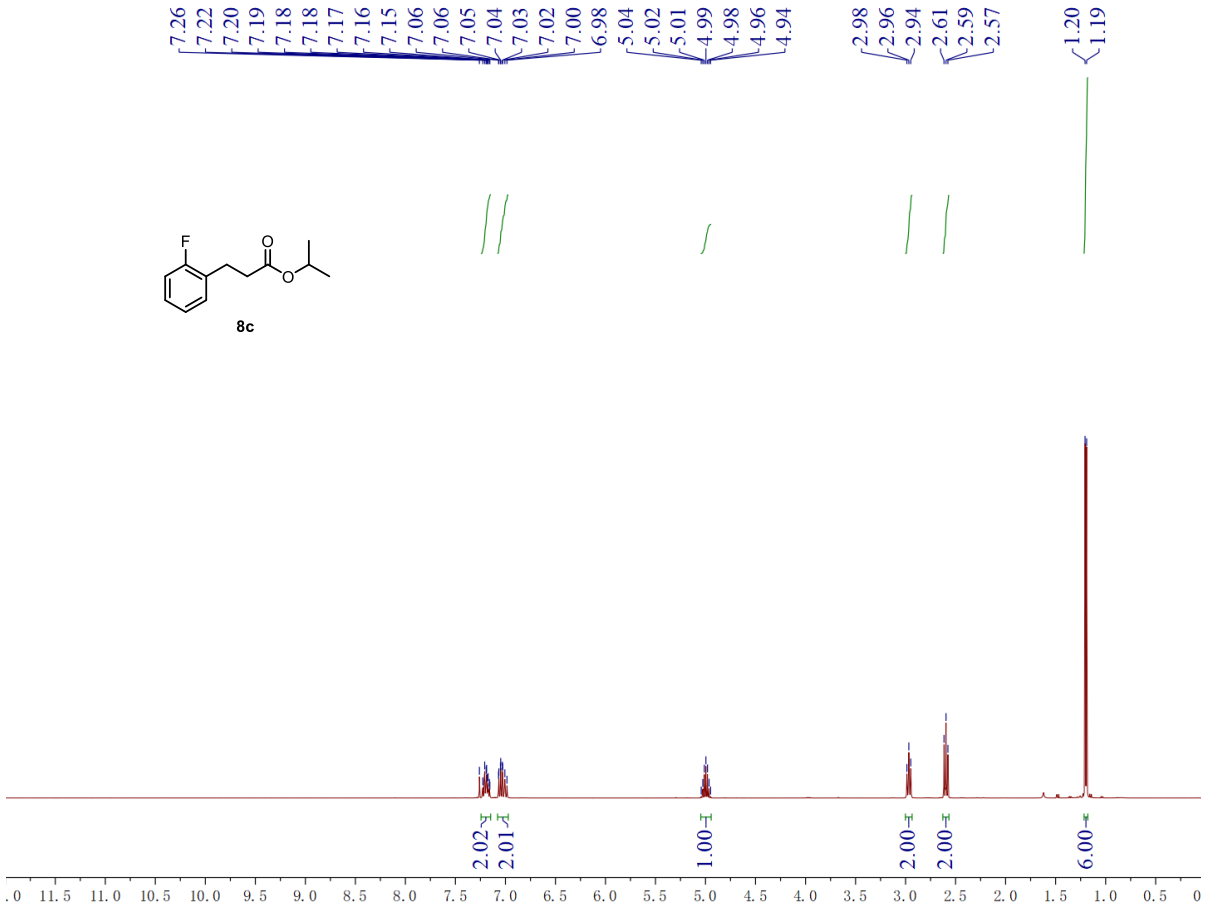
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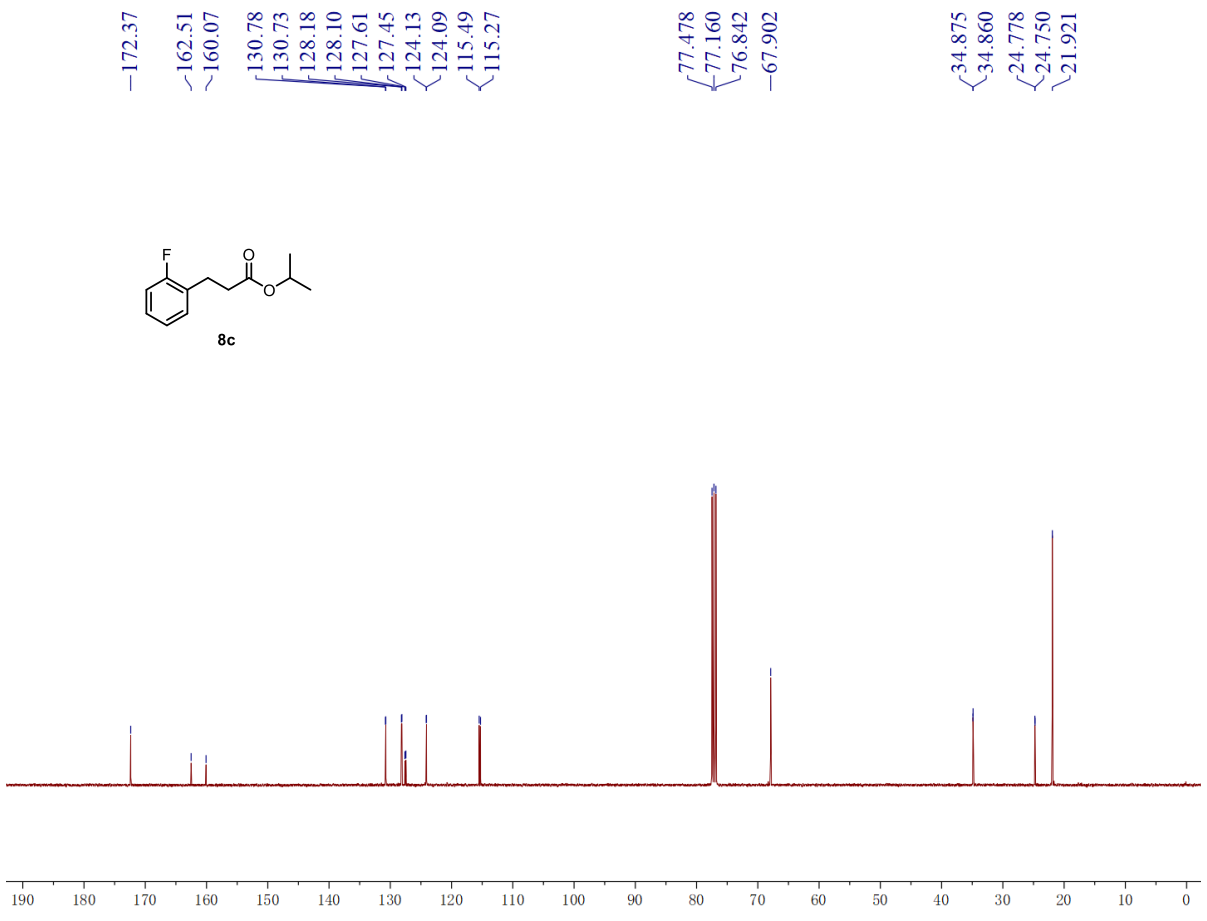
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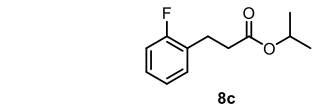

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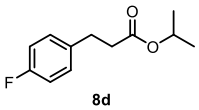

8d

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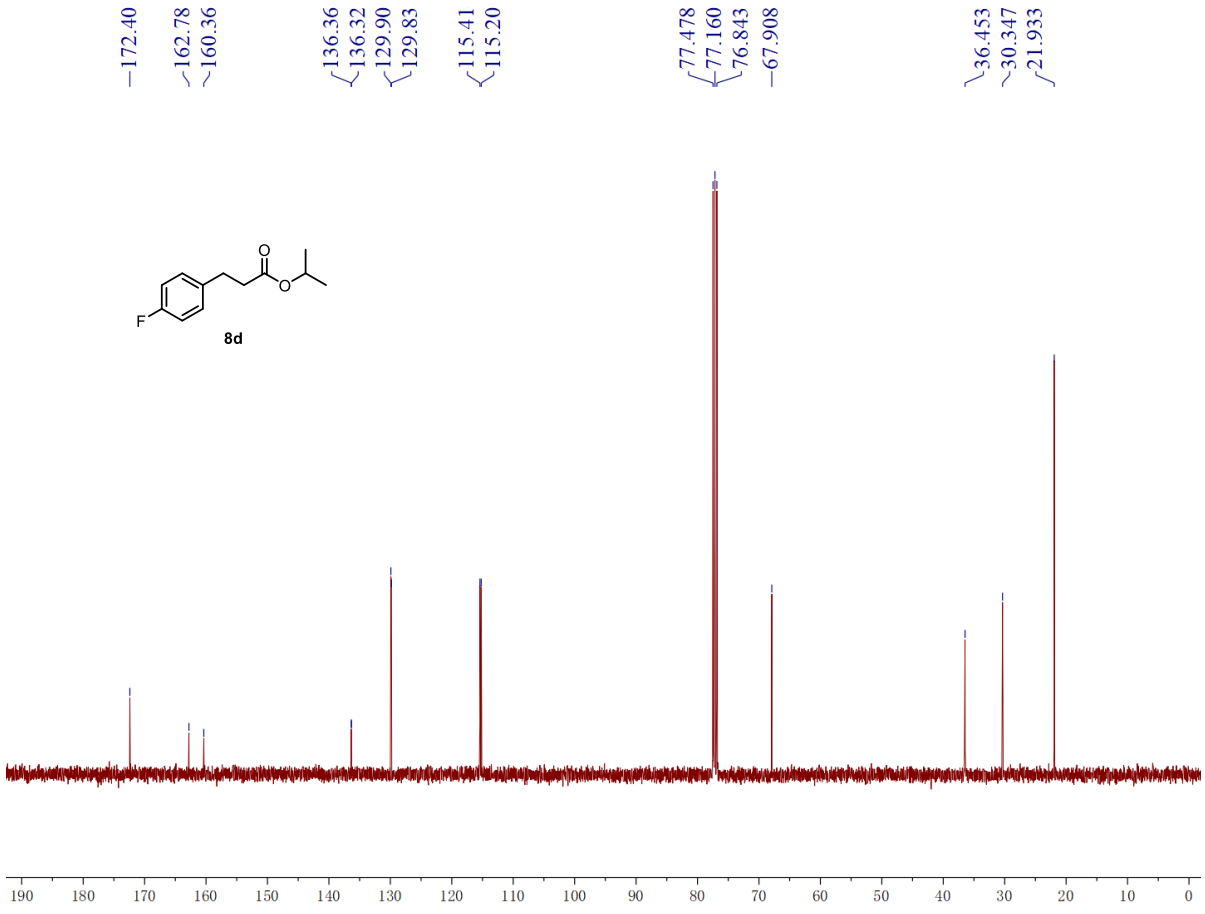
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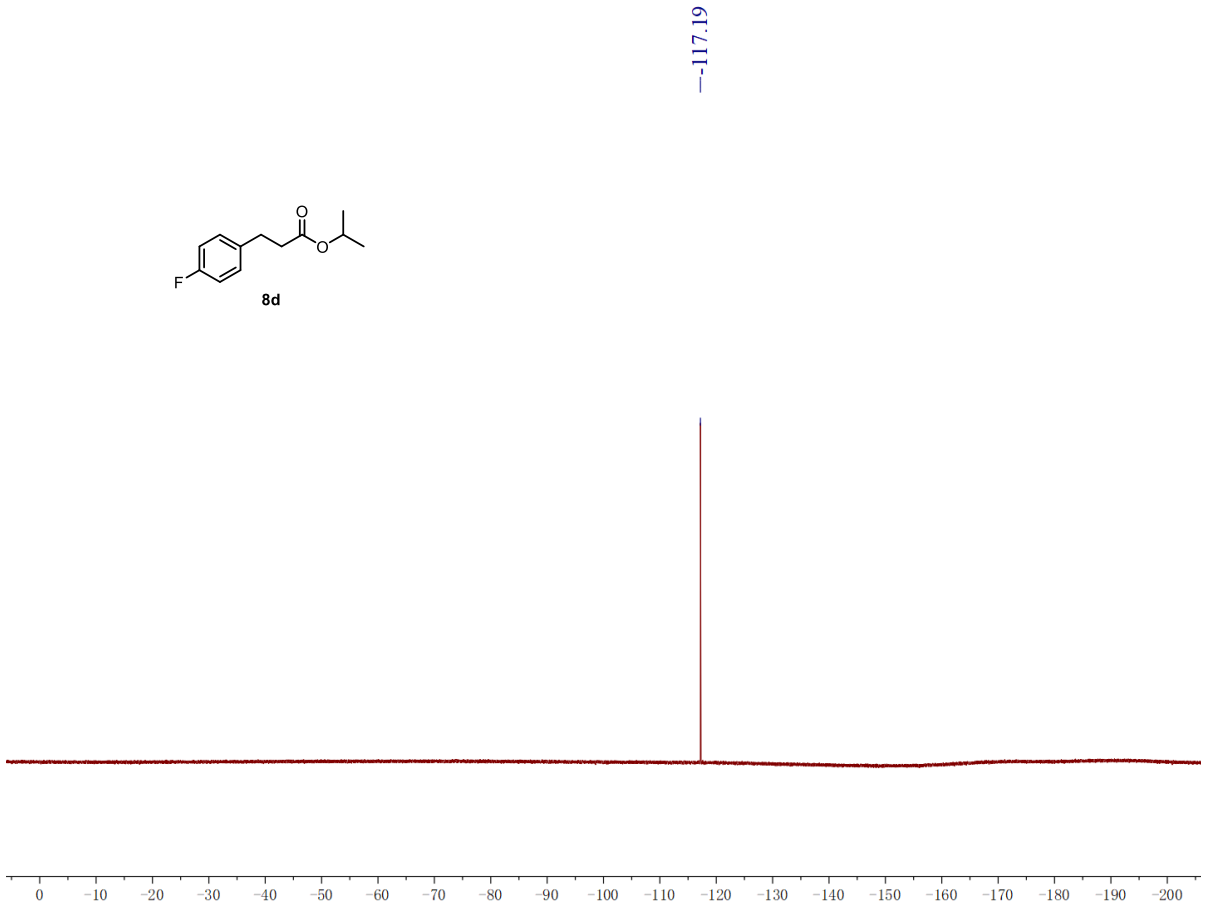
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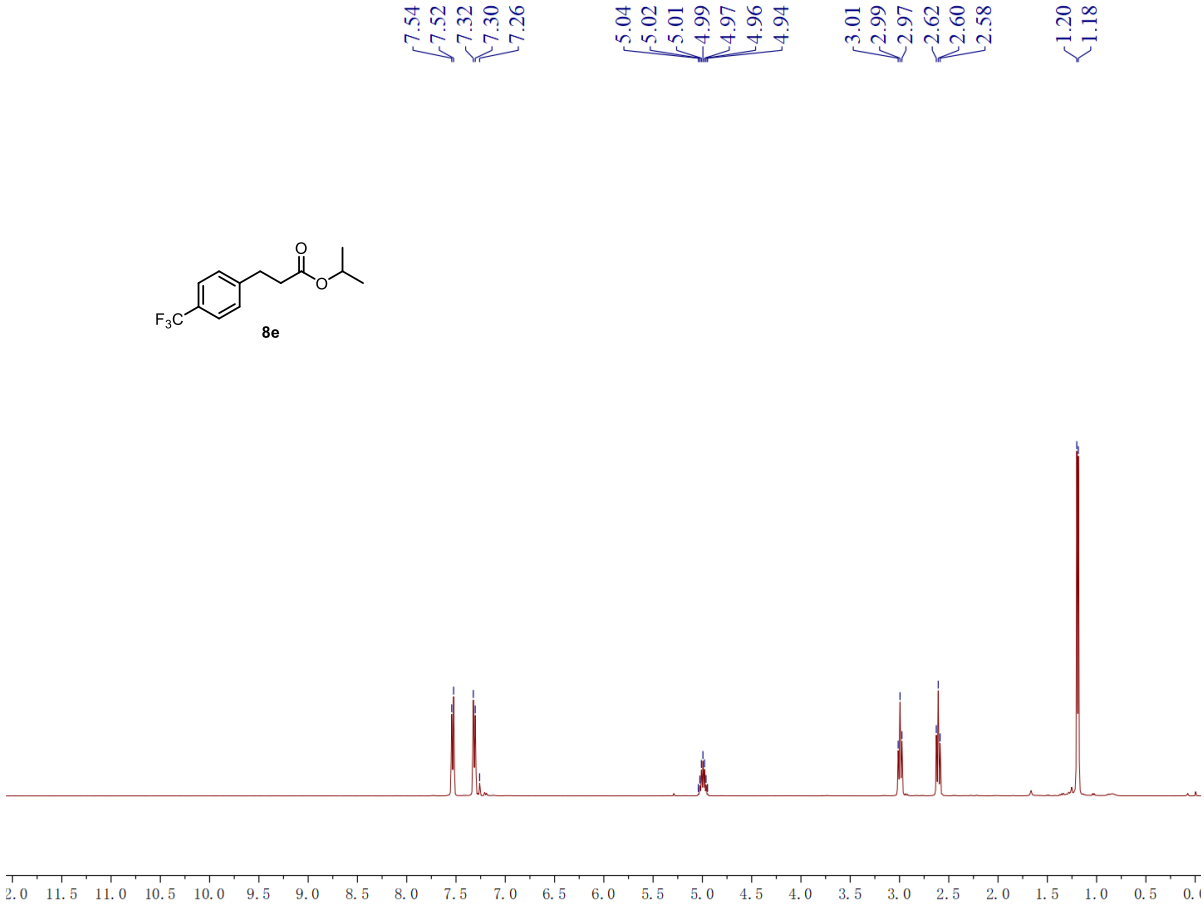
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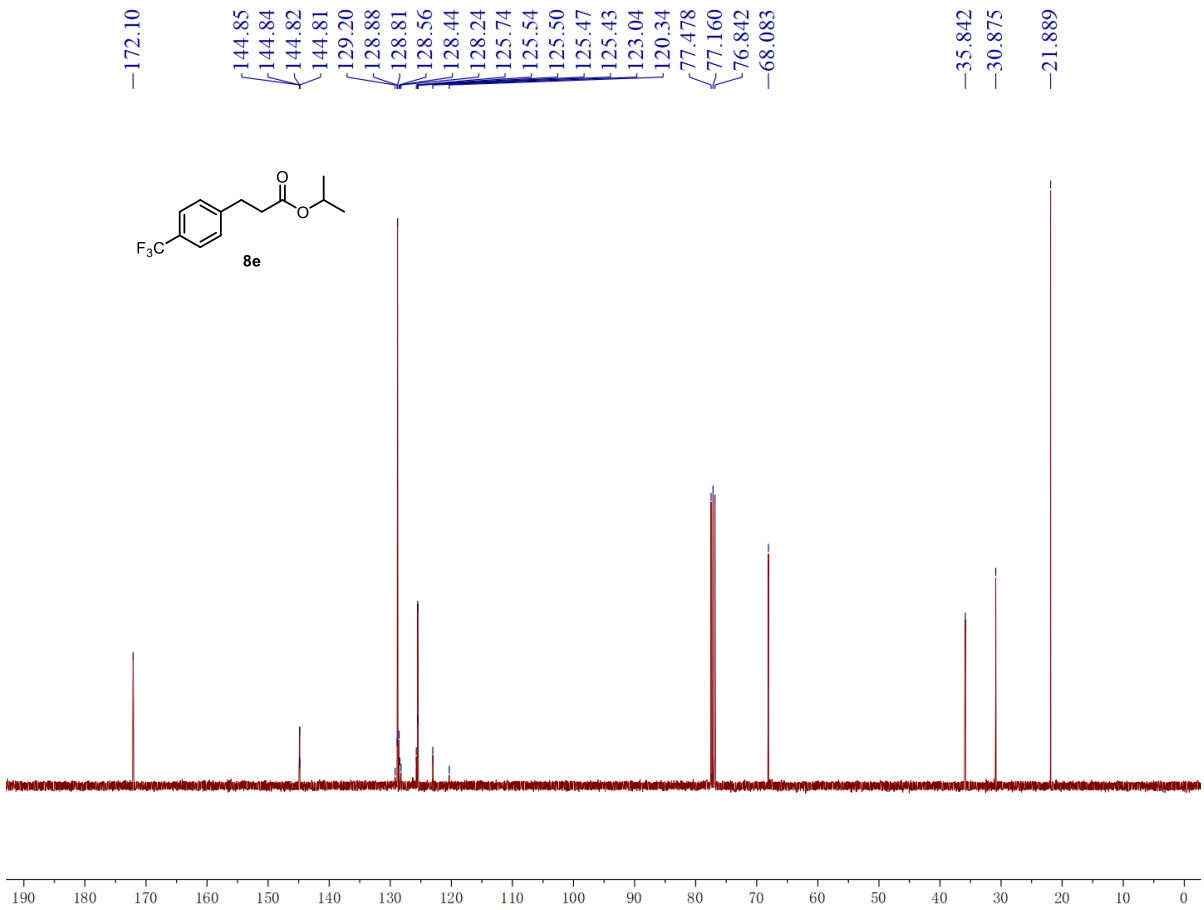
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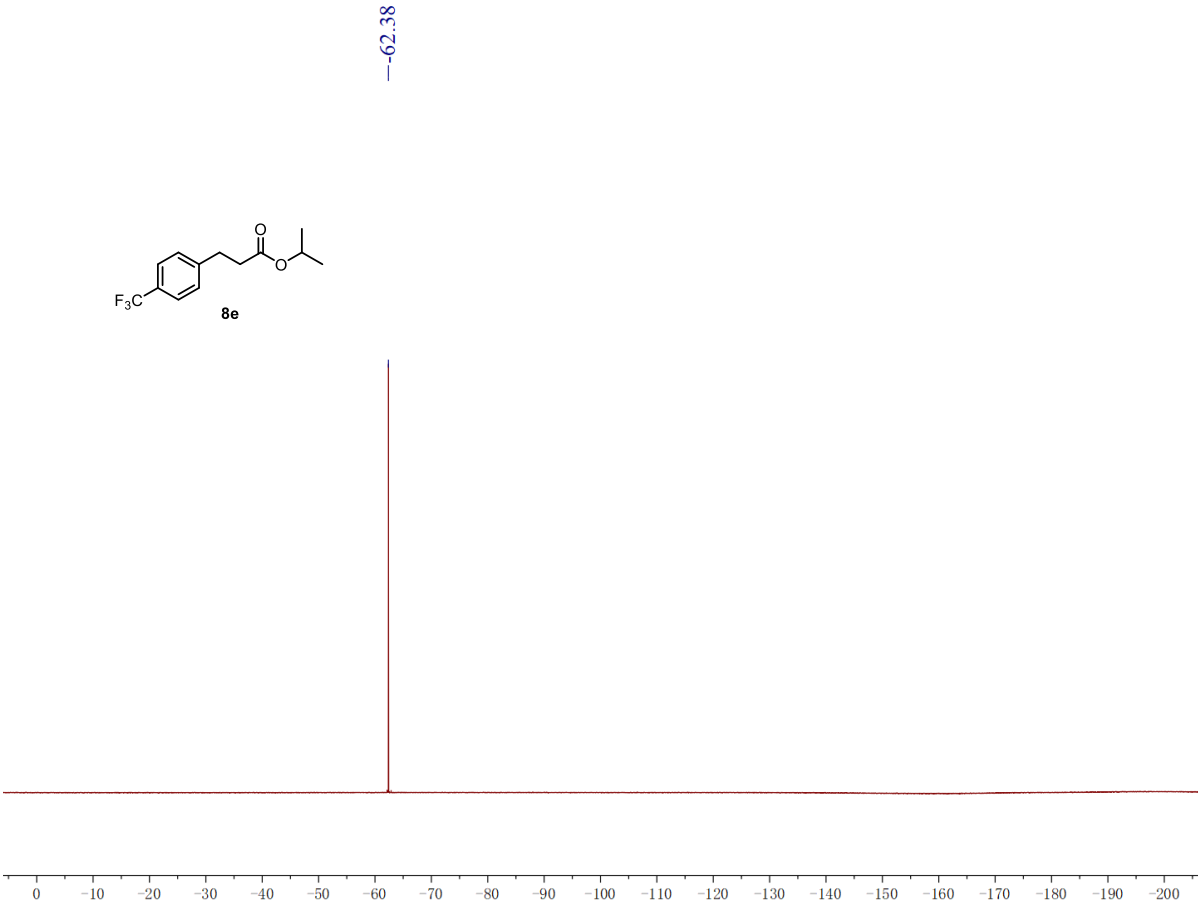
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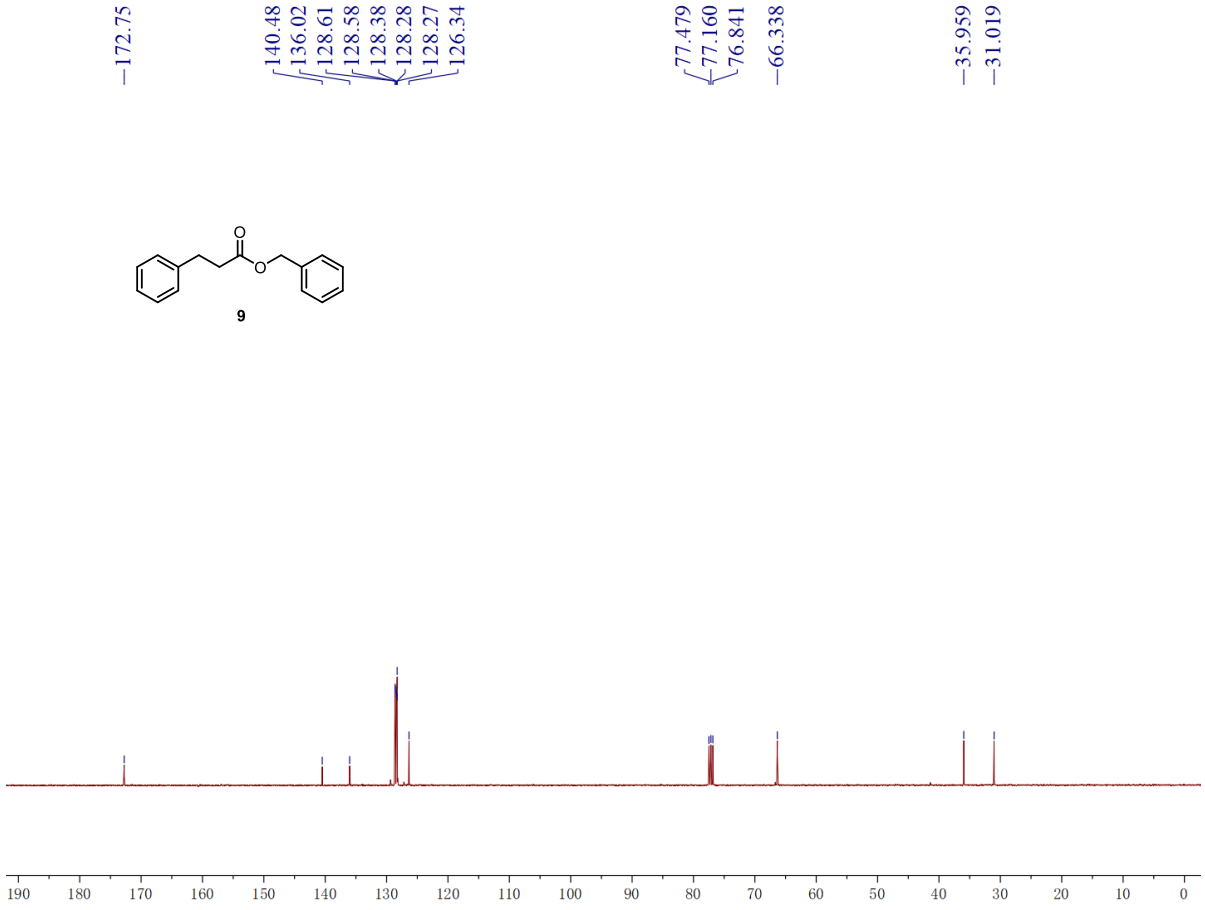
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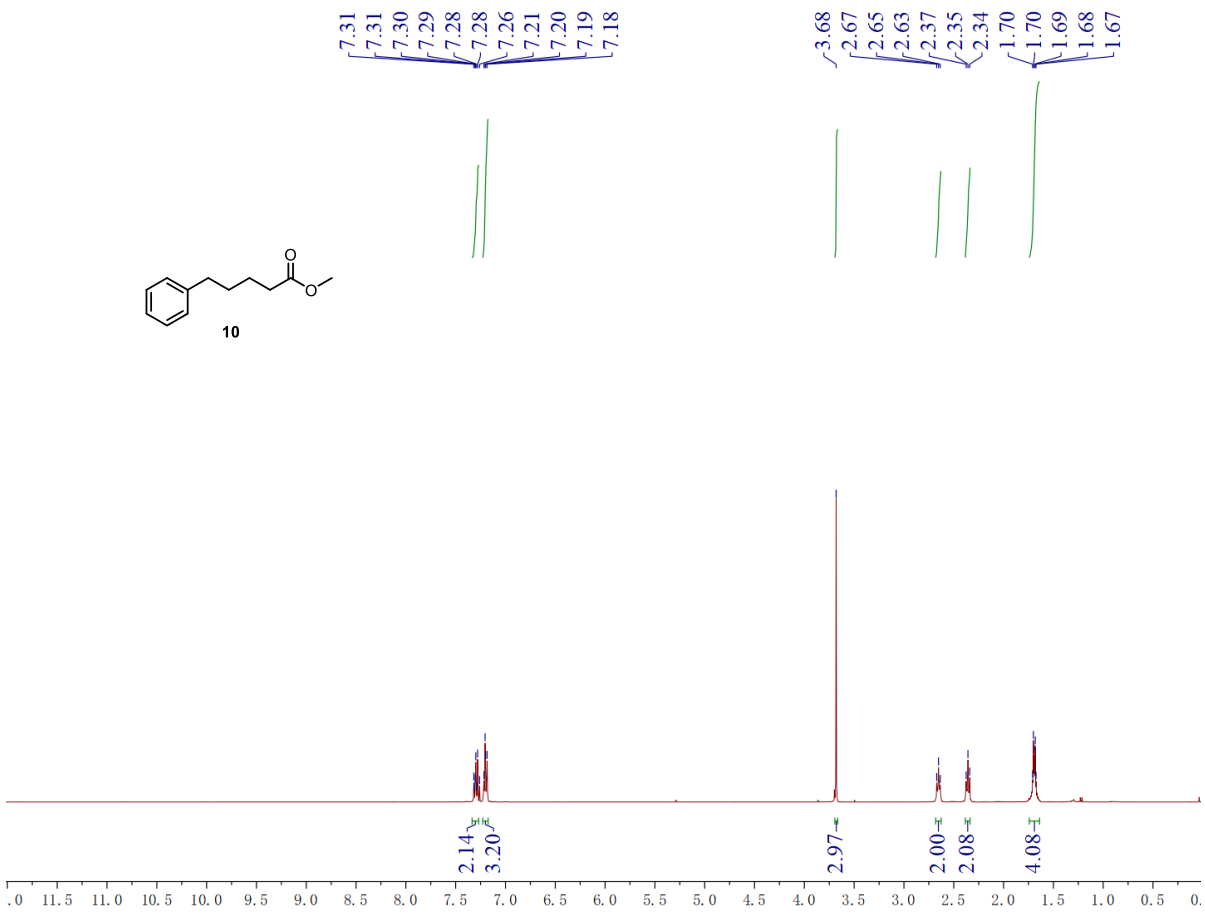
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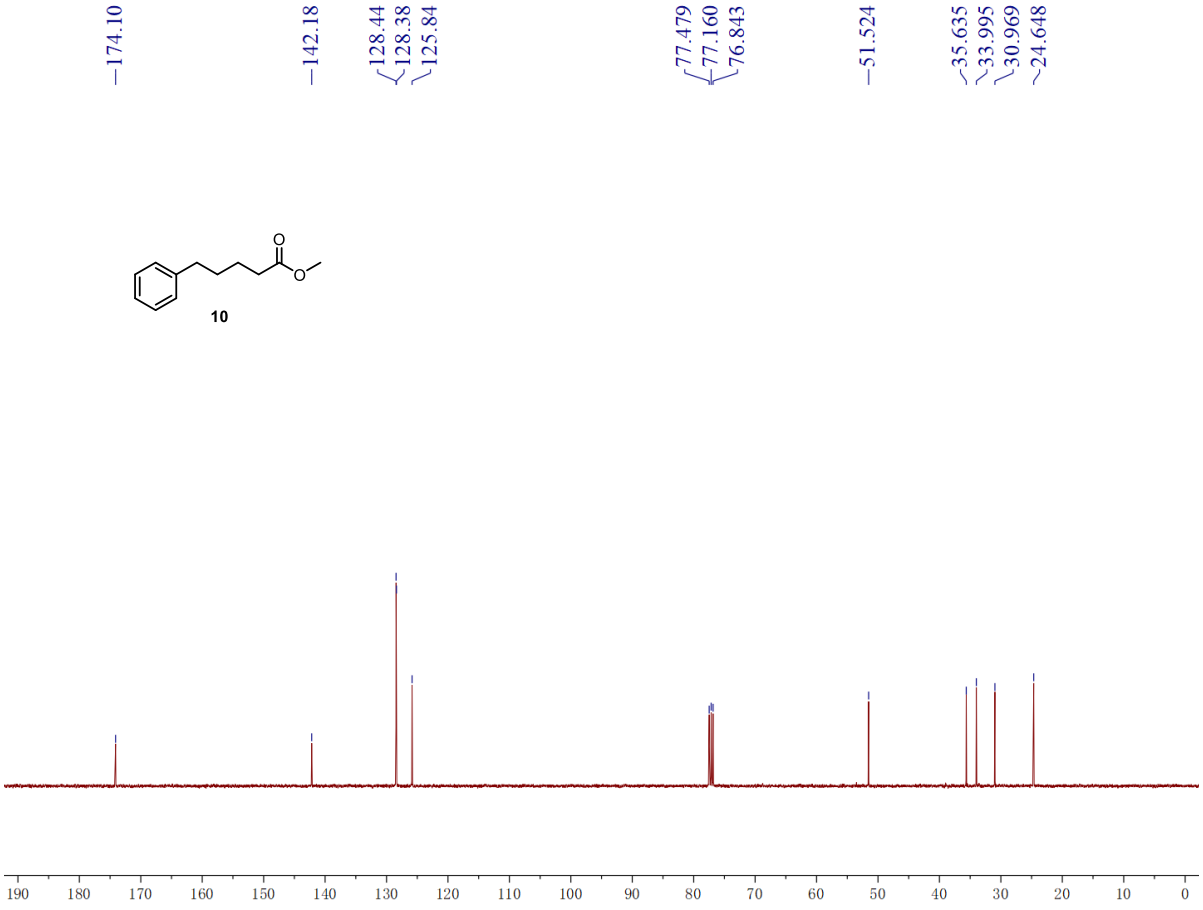
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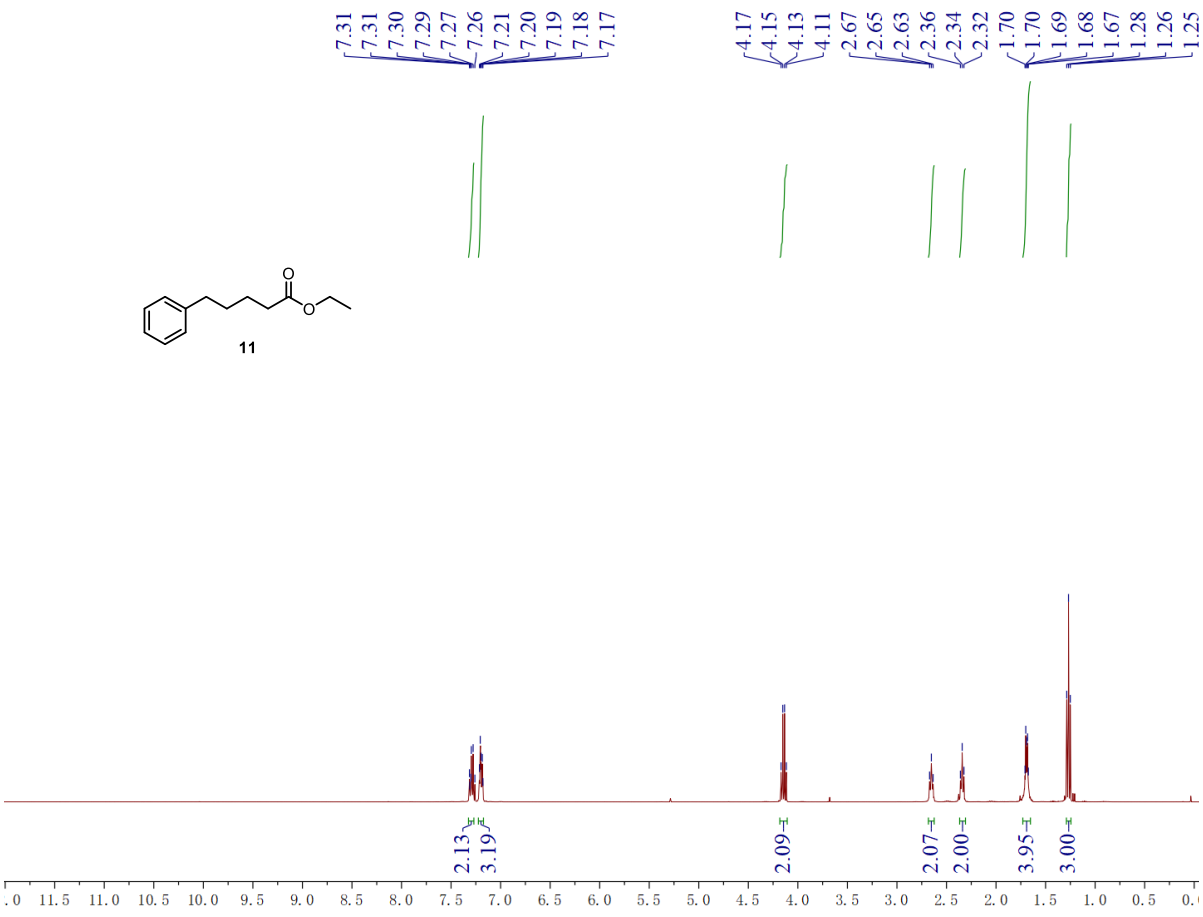
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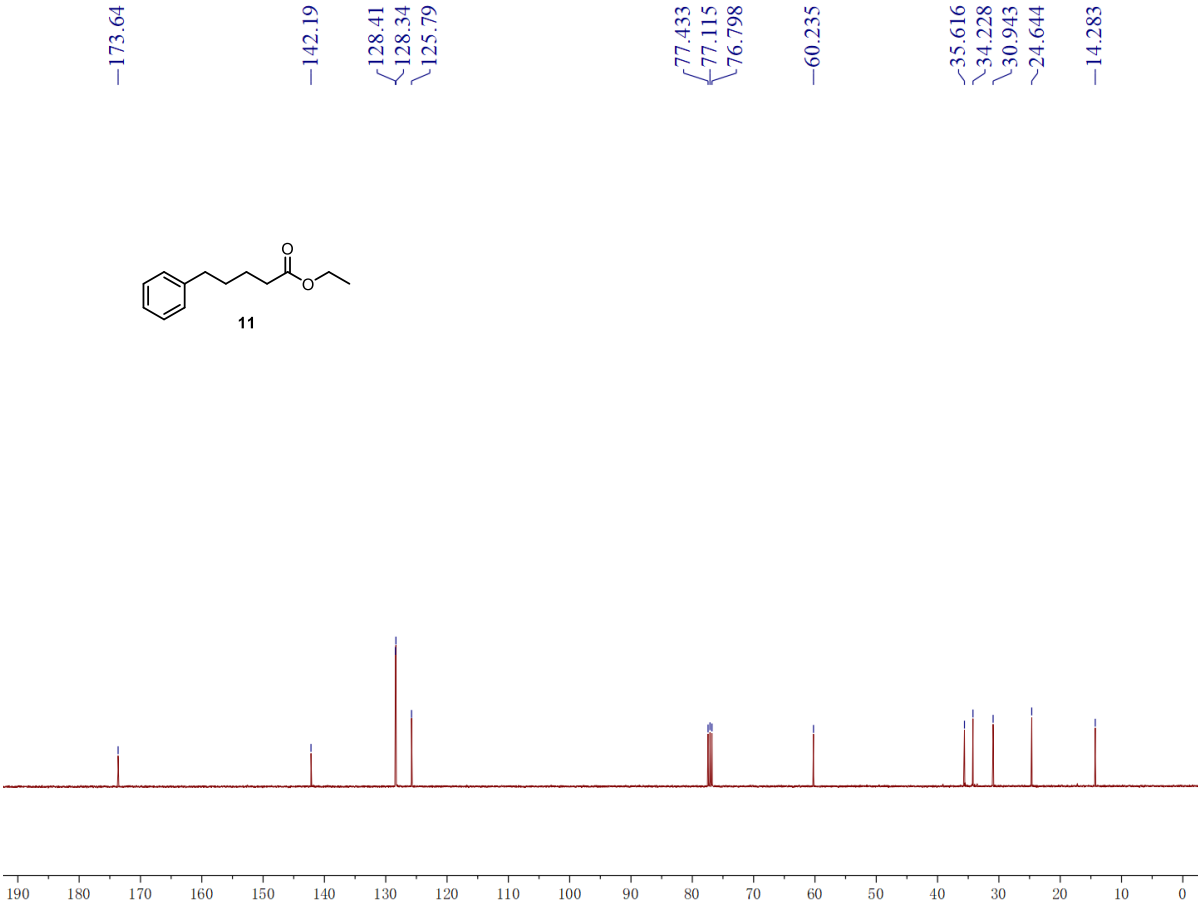
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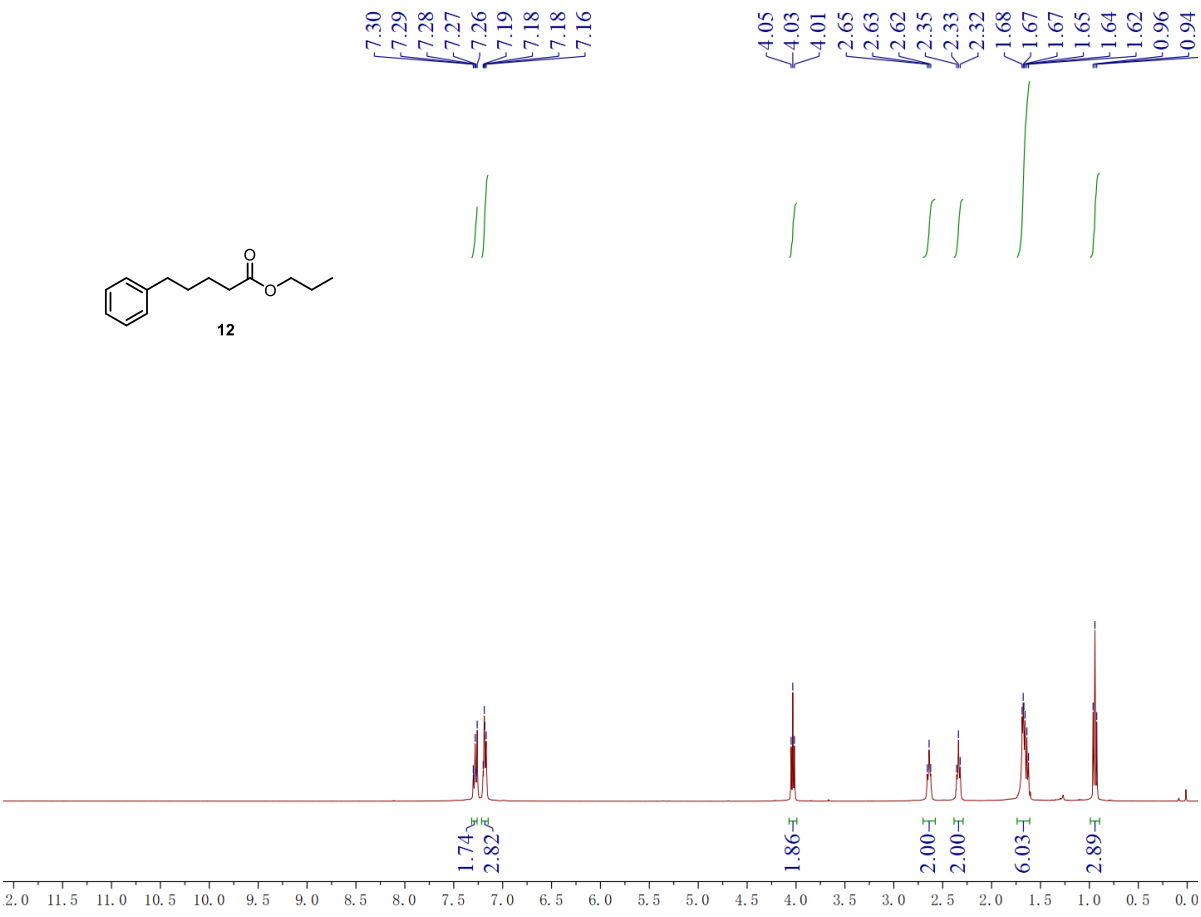
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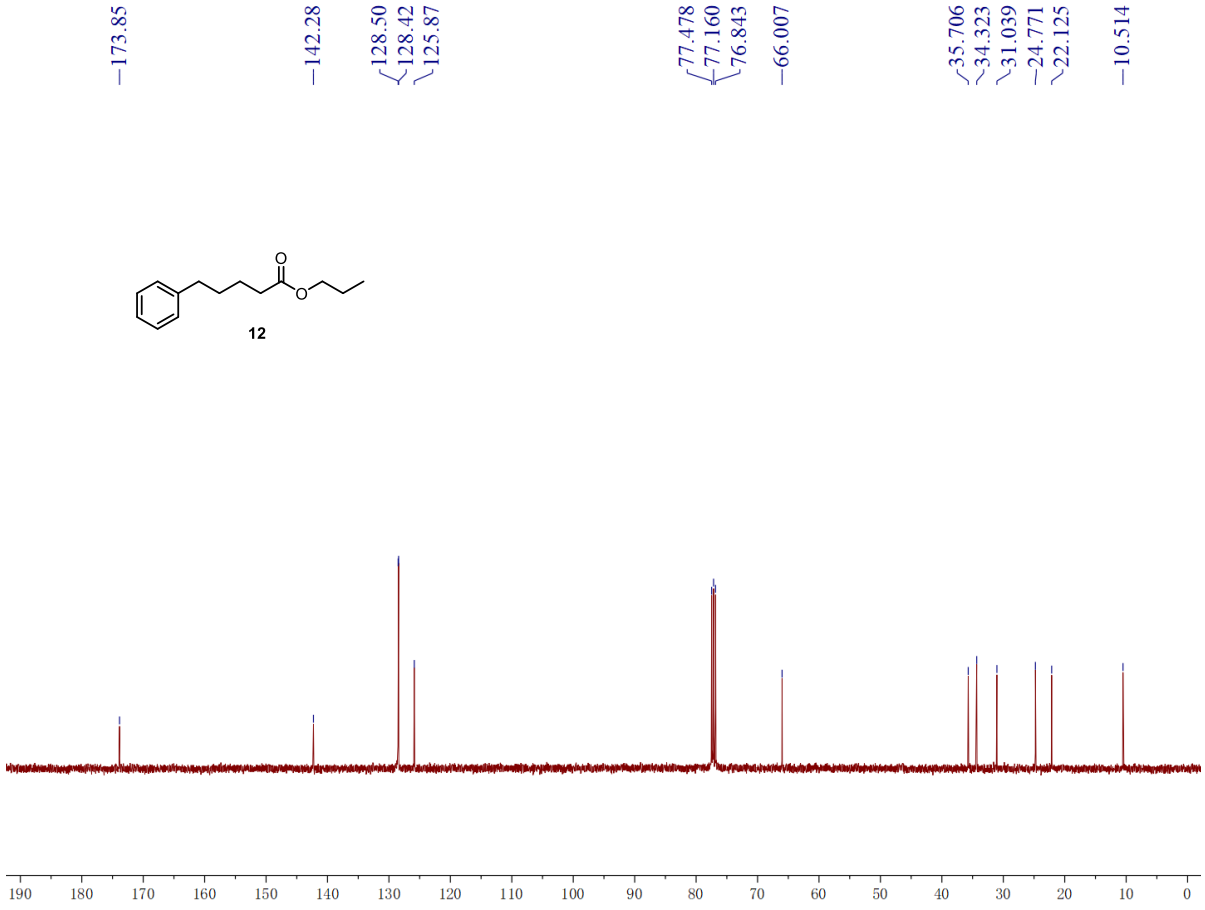
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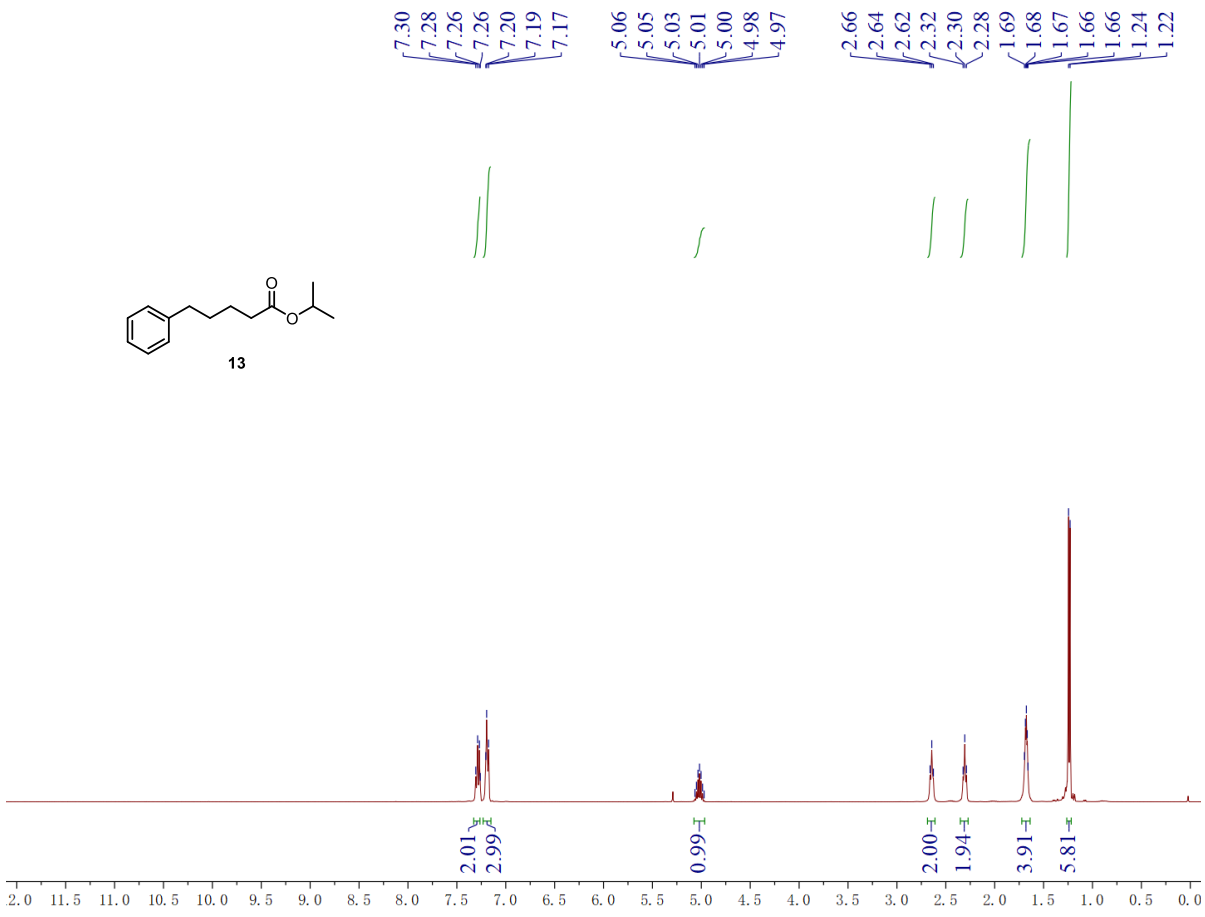
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1.22
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0.99
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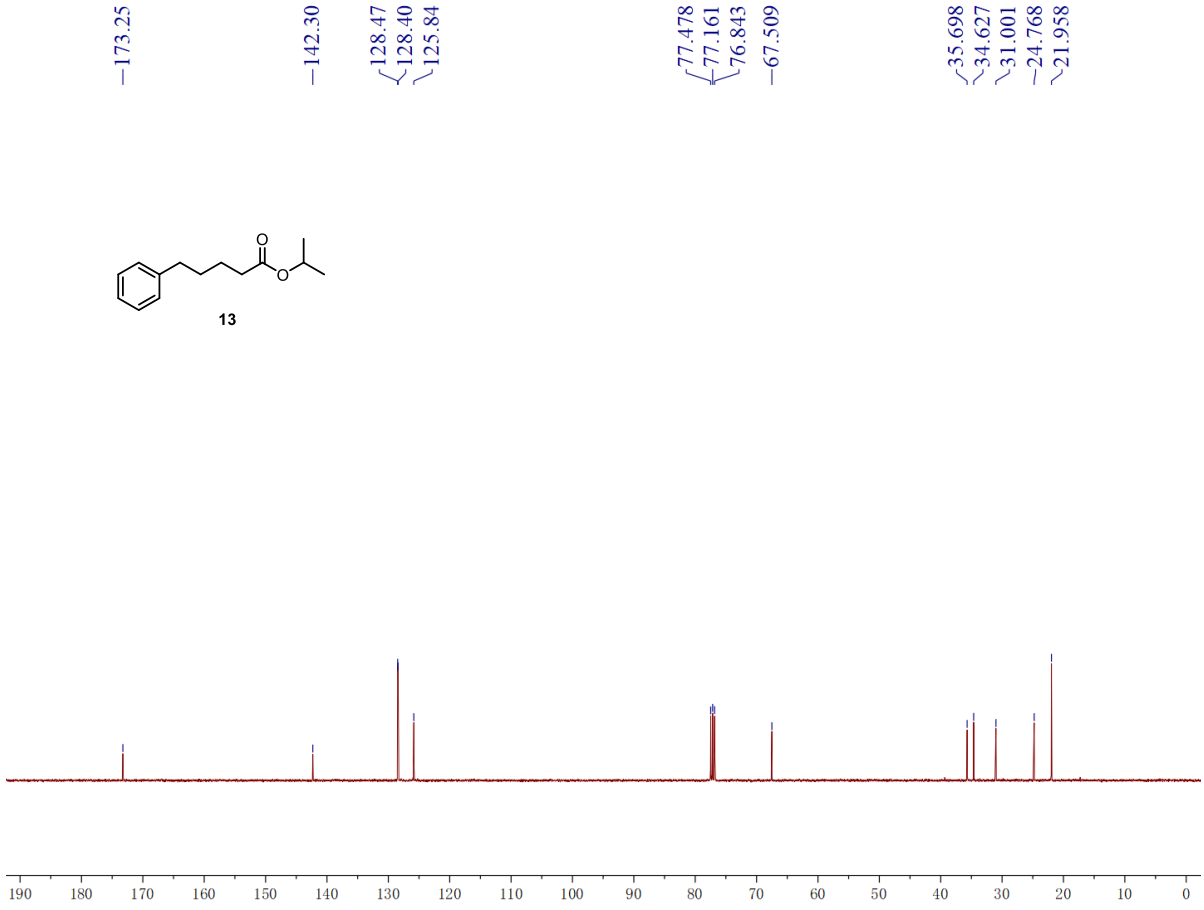
3.91
5.81
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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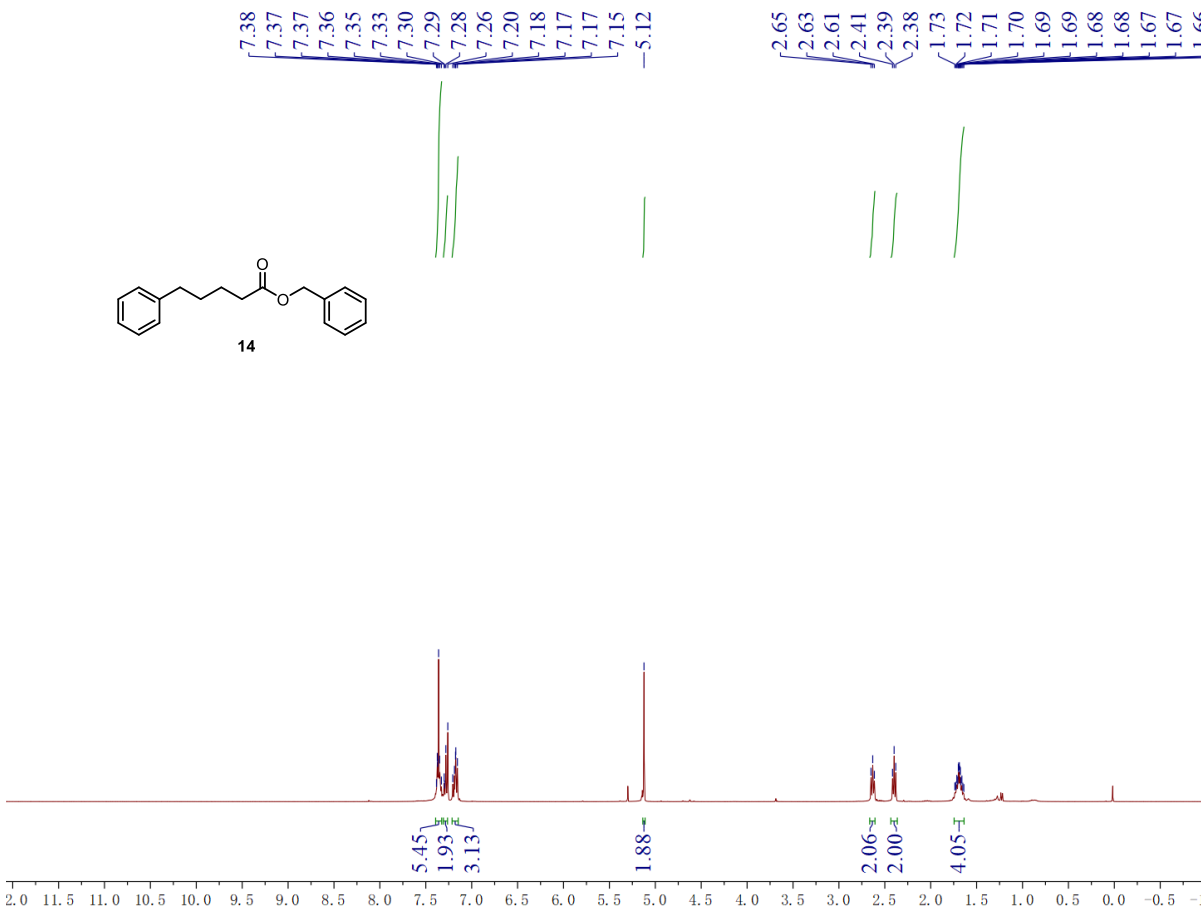
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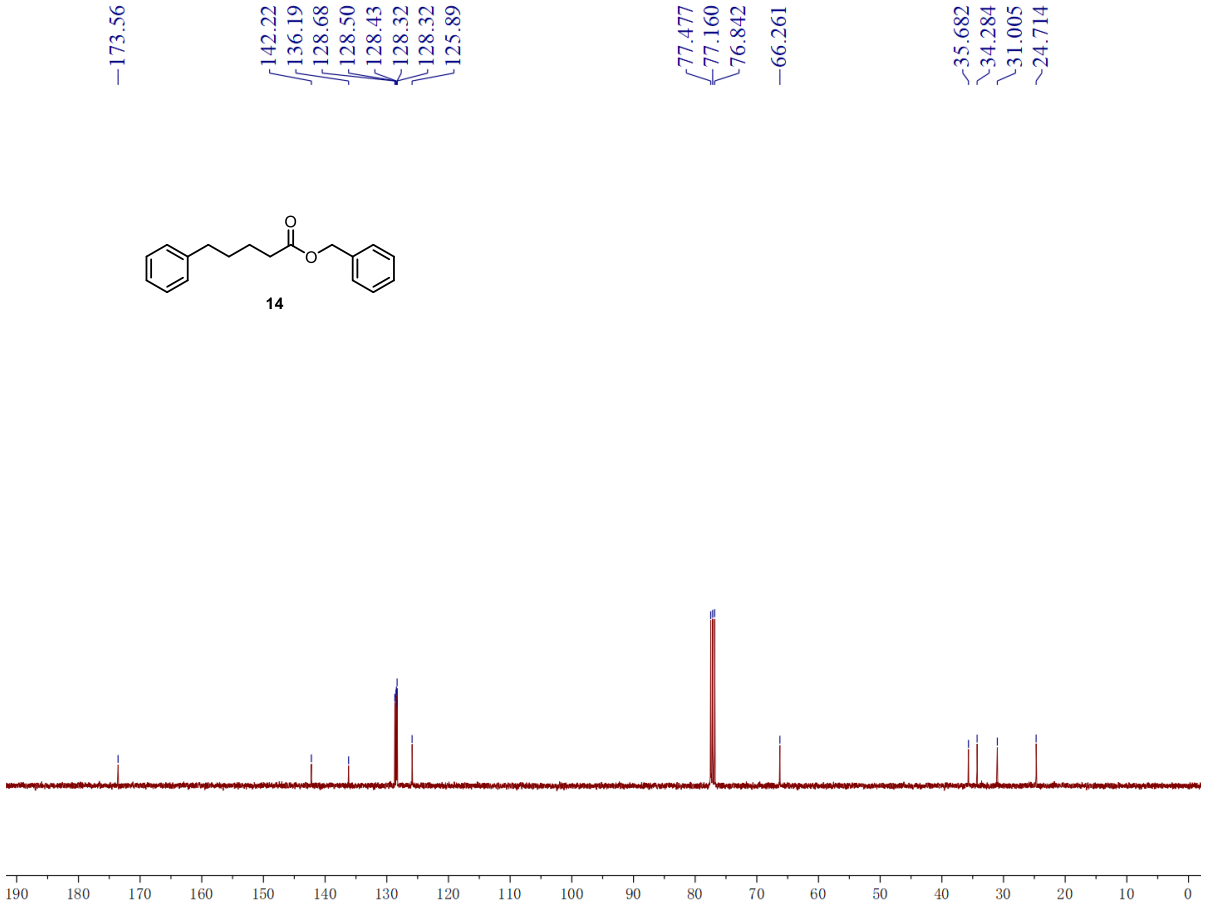
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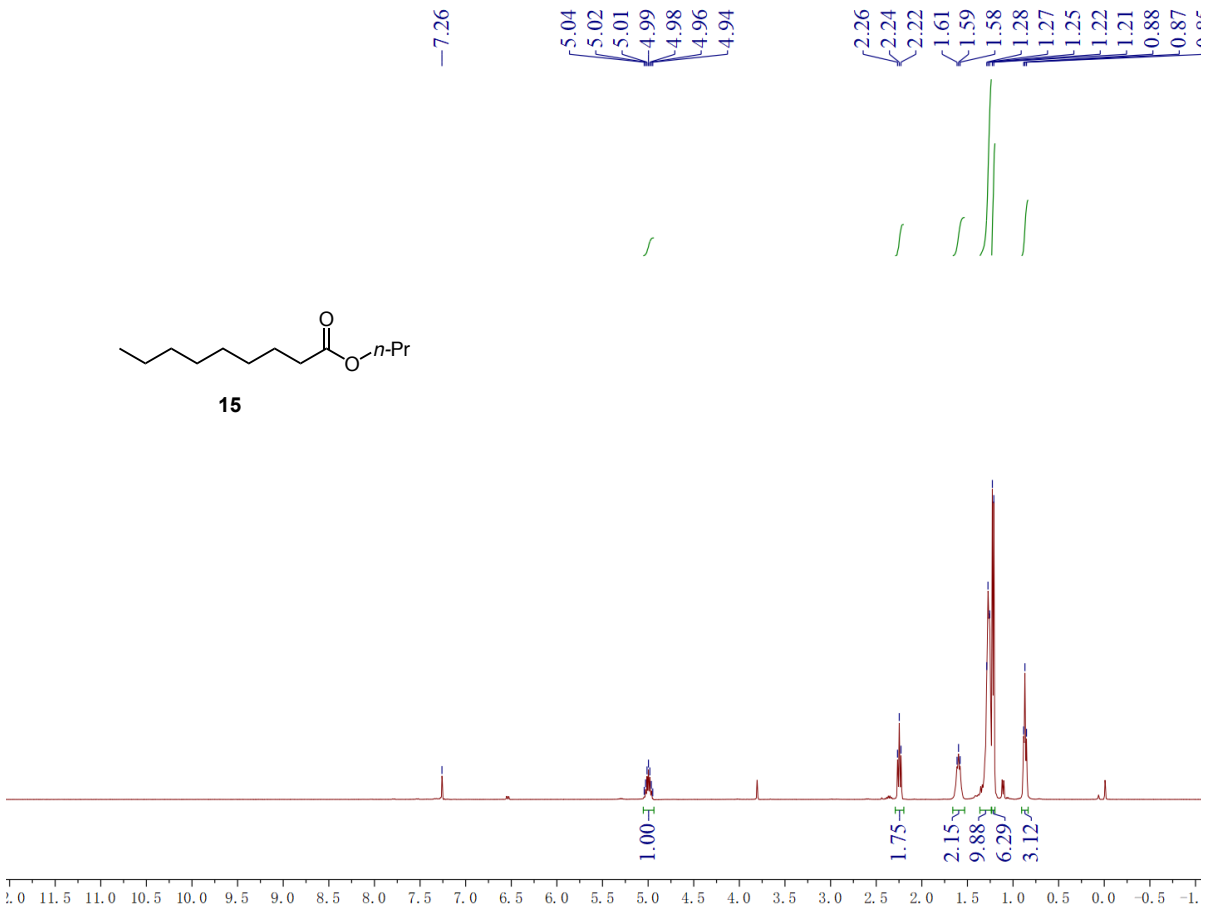
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n-Pr
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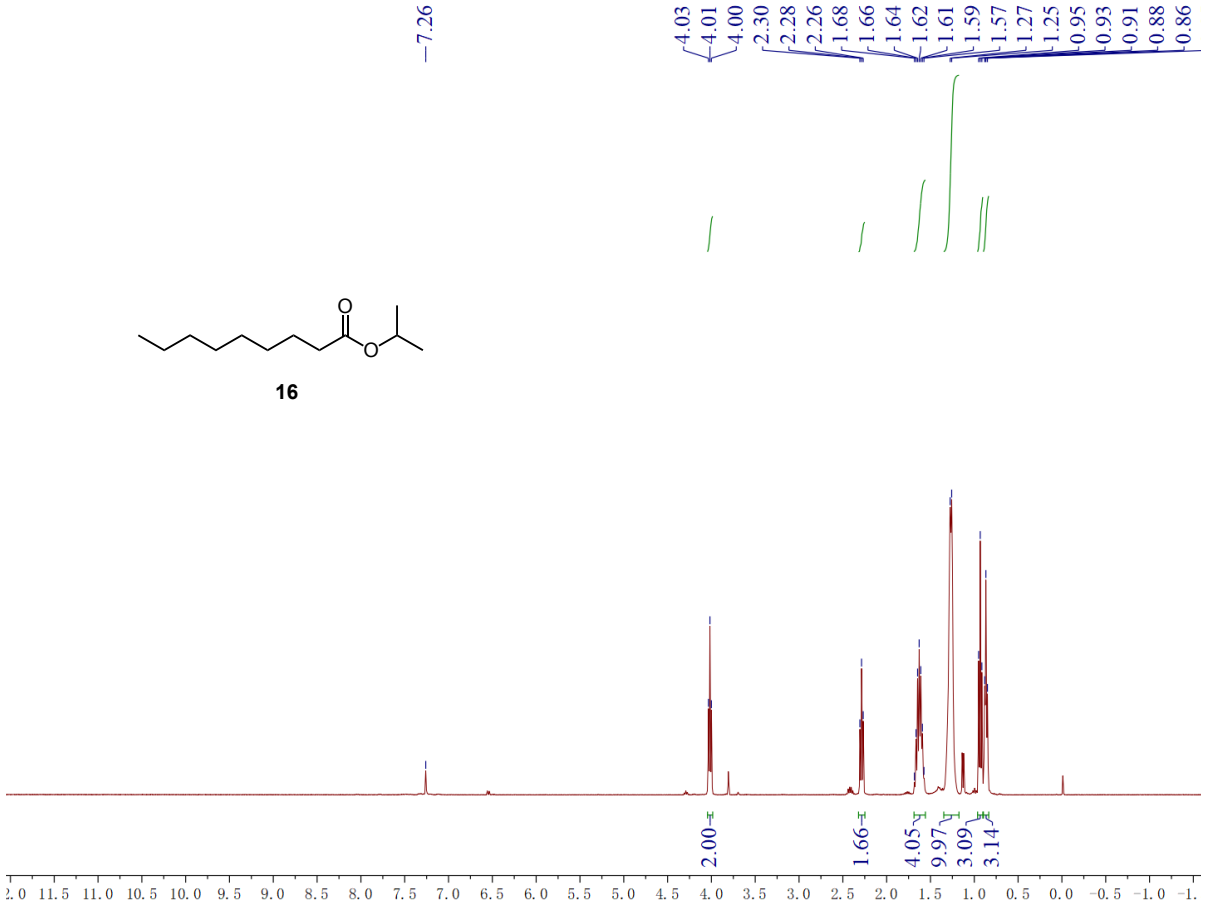
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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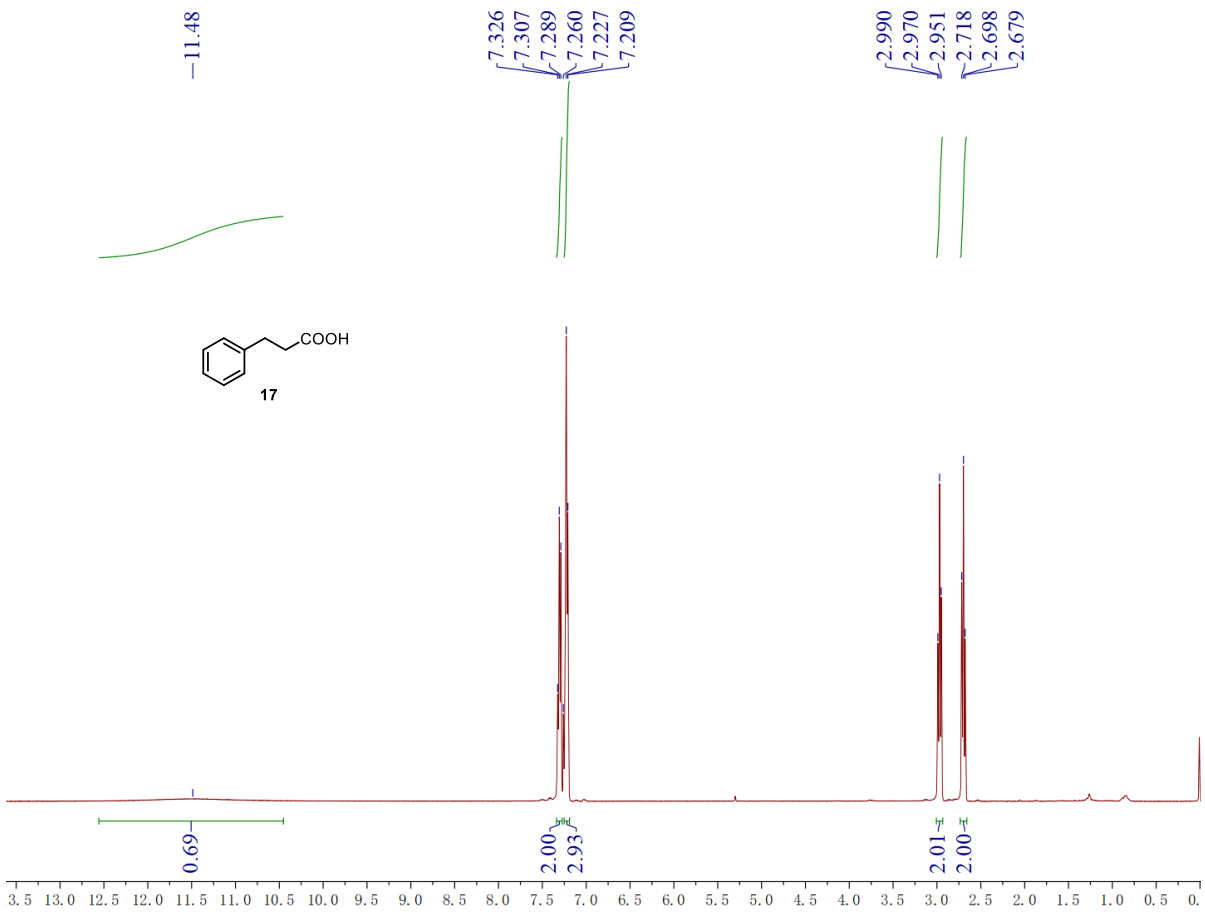
16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17