Supporting Information for

Continuous-Flow Electrochemical Benzylic Dehydrogenation of Arylalkanes to Arylalkenes

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1. General Information

1.1 Analytic methods

All reactions were carried out under a nitrogen atmosphere with dry solvent under anhydrous conditions, unless otherwise noted. All commercial reagents were used without further purification unless otherwise noted. Reactions were monitored by thin-layer chromatography (TLC) analysis. TLC plates were viewed under UV light and stained with potassium permanganate. Yields refer to products isolated after purification by column chromatography unless otherwise stated. Proton nuclear magnetic resonance (^{1}H NMR) spectra, carbon nuclear magnetic resonance (^{13}C NMR) spectra, and fluorine nuclear magnetic resonance (^{19}F NMR) were recorded on Bruker Avance NEO 500 spectrometers. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances. HRMS were obtained on an GC-MS (EI) mass spectrometer with the use of a quadrupole analyzer. Cyclic voltammetry data were measured with a CHI 660E potentiostat (Chinstruments). Electrolysis experiments were performed using MESTEK DC power supply. Electrode clips (PT-3) and platinum plate (99.99%, 15 mm × 15 mm × 0.3 mm) was purchased from Gaoss Union. The carbon cloth (CeTech WOS1002) was cut into 15 mm ×15 mm × 0.3 mm pieces before use, and was clamped between electrode clips.

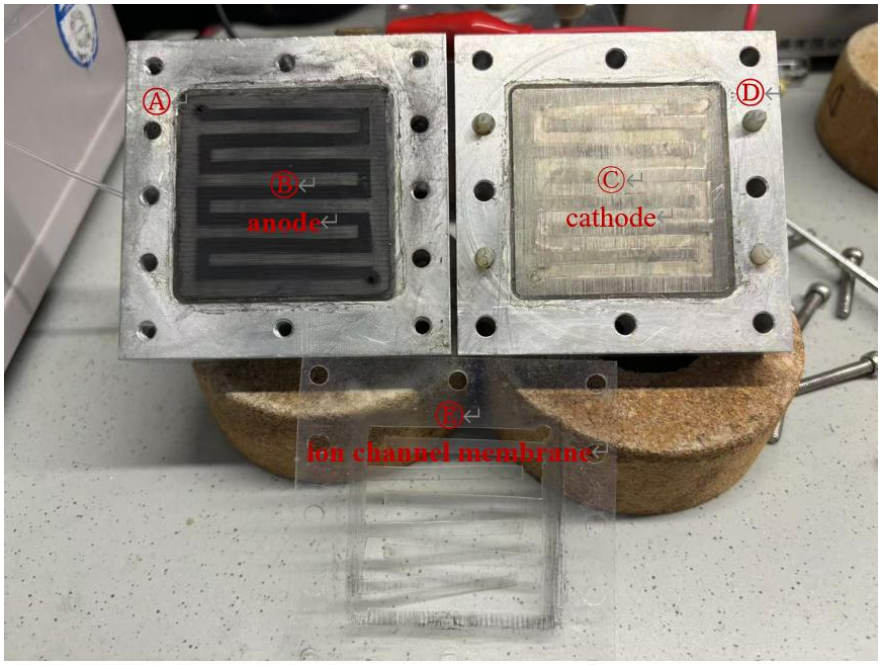
1.2 Reagents

All commercially available compounds were purchased from Energy Chemical, Innochem, TCI, Adamas, *J&K*, Alfa-Aesar. All the solvents and all the other reagents were directly used from purchased without any further purification unless otherwise specified.

2. Design of the Flow Electrolysis Cell [1]

The design is shown in Figure S1. The flow electrolysis cell is assembled using two aluminum bodies (Ⓐand Ⓓ , 75 mm × 75 mm × 15 mm) with a groove (50 mm × 50 mm × 3.0 mm). The anode, which is made of graphite (Ⓑ, 48 mm × 48 mm × 3.0 mm), is insulated from the aluminum body by PTFE or silicone film. The cathode is a Ni plate (Ⓒ, 48 mm × 48 mm × 3.0 mm). The anode and cathode are held apart by a fluorinated ethylene propylene (FEP) foil（Ⓔ） of 200 μm thickness. A rectangular reaction channel (total length: 313 mm, width: 3.2 mm) is cut in the FEP foil to give an overall channel volume of 250 μL. The whole device is held together by steel screws. The inside view of the device is shown in **Figure S1 A**. The reaction setups are shown in **Figure S1 B**.

A



B

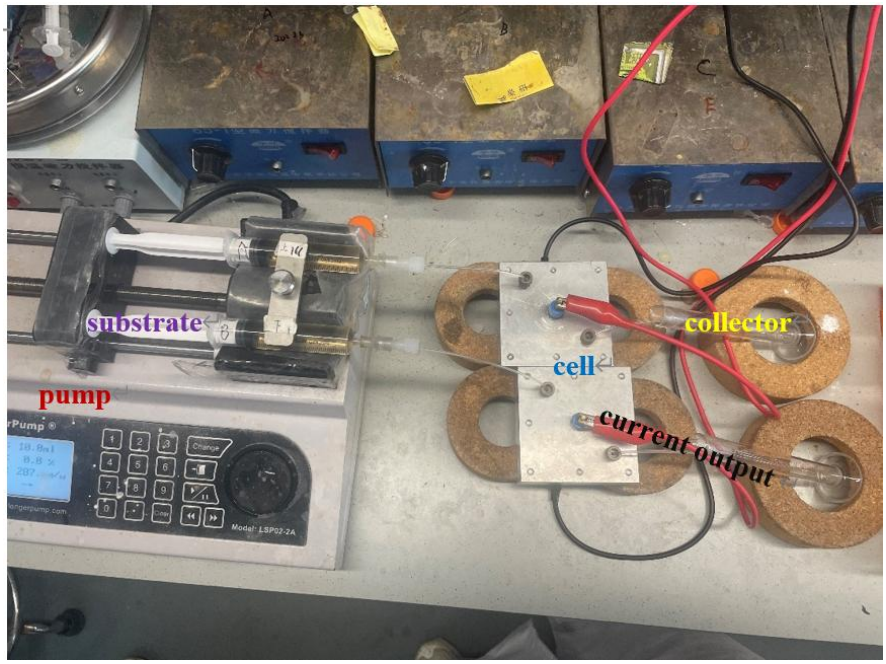


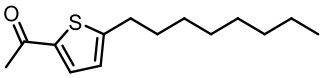
Figure S1 Flow electrolysis setups. Details of the reactor have been reported [1].

3. Experimental Procedures

General procedure for the preparation of newly reported starting materials. Substrates **S18, S19, S34-S38** were prepared according to the reported procedures.

New compounds:

3.1 Synthesis of 1-(5-octylthiophen-2-yl) ethan-1-one (S18)



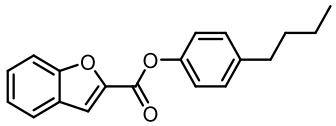
Following a modified procedure [2], a dry 50-mL Schlenk tube containing a magnetic stir bar was charged with 2-octylthiophene (1.0 eq, 980.6 mg, 5.0 mmol), $InCl_3$ (20 mol%, 185.7 mg, 1.0 mmol) and acetic anhydride (5.0 mL). After stirring at room temperature for 3 h, the reaction mixture was concentrated via rotary evaporation. The crude material was purified by flash chromatography using petroleum ether/EtOAc = 20 :1 to afford the product as a light yellow liquid (1.01 g, 85% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, *J* = 3.8 Hz, 1H), 6.80 (d, *J* = 3.8 Hz, 1H), 2.82 (t, *J* = 7.6 Hz, 2H), 2.50 (s, 3H), 1.71–1.65 (m, 2H), 1.38–1.26 (m, 10H), 0.87 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 190.5, 156.0, 141.9, 132.8, 125.5, 31.8, 31.3, 30.6, 29.2, 29.1, 29.0, 26.4, 22.6, 14.0.

HRMS: calc. for $C_{14}H_{22}OS$ (EI), 238.1386, found, 238.1389.

3.2 Synthesis of 4-butylphenyl benzofuran-2-carboxylate (S19)



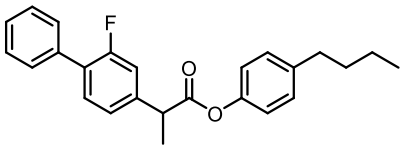
Following a modified procedure from literature [3a], a dry 100-mL round-bottom flask containing a magnetic stir bar was charged with benzofuran-2-carboxylic acid (1.0 eq, 5.0 mmol, 0.81 g), DCC (1.1 eq, 5.5 mmol, 1.13 g), DMAP (0.2 eq, 1.0 mmol, 122 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 5.5 mmol, 826.5 mg) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (1.12 g, 76% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 2H), 2.64 (t, *J* = 7.7 Hz, 2H), 1.65–1.59 (m, 2H), 1.42–1.35 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.1, 156.1, 148.1, 145.0, 141.0, 129.5, 128.1, 127.0, 124.0, 123.0, 121.2, 115.3, 112.5, 35.1, 33.6, 22.4, 14.0.

HRMS: calc. for $C_{19}H_{18}O_3$ (EI), 294.1250, found, 294.1252.

3.3 Synthesis of 4-butylphenyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl) propanoate (S35)



Following a modified procedure from literature [3a], a dry 100 mL round-bottom flask containing a magnetic stir bar was charged with flurbiprofen (1.0 eq, 5.0 mmol, 1.22 g), DCC (1.1 eq, 5.5 mmol, 1.13 g), DMAP (0.2 eq, 1.0 mmol, 122 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 5.5 mmol, 826 mg) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (1.50 g, 80% yield).

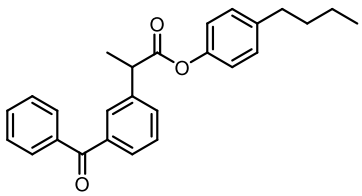
^{1}H NMR (500 MHz $CDCl_3$,): δ 7.57–7.55 (m, 2H), 7.47–7.43 (m, 3H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.26–7.21(m, 2H), 7.15 (d, *J* = 8.5 Hz, 2H), 6.93 (d, *J* = 8.5 Hz, 2H), 3.99 (q, *J* =7.5 Hz, 1H), 2.59 (*J* = 7.7 Hz, 2H), 1.65 (d, *J* =7.2 Hz, 2H), 1.59–1.54 (m, 2H), 1.37–1.31 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 172.7, 159.8 (d, *J* = 244.5 Hz), 142.8, 141.5, 141.4, 140.6, 135.5, 131.0 (d, *J* = 4.1 Hz), 129.3, 129.0 (d, *J* = 2.7 Hz), 128.5, 128.1 (d, *J* = 13.8 Hz), 127.7, 123.6 (d, *J* = 3.4 Hz), 121.0, 115.4 (d, *J* = 23.6 Hz), 45.2, 35.0, 33.6, 22.3, 18.5, 13.9;

^{19}F NMR (471 MHz, $CDCl_3$): -117.4.

HRMS: calc. for $C_{25}H_{25}FO_2$ (EI), 376.1833, found, 376.1836.

3.4 Synthesis of 4-butylphenyl 2-(3-benzoylphenyl) propanoate (S36)



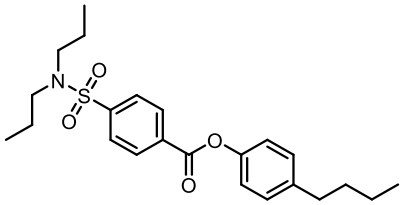
Following a modified procedure[3a], a dry 100-mL round-bottom flask containing a magnetic stir bar was charged with ketoprofen (1.0 eq, 5.0 mmol, 1.27 g), DCC (1.1 eq, 5.5 mmol, 1.13 g), DMAP (0.2 eq, 1.0 mmol, 122 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 5.5 mmol, 826 mg) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (1.39 g, 72% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.86 (s, 1H), 7.82 (d, *J* = 7.5 Hz, 2H), 7.73 (d, *J* = 7.7 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.60 (d, *J* = 7.3 Hz, 1H), 7.51–7.46 (m, 3H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.4 Hz, 2H), 4.04 (q, *J* = 14.3 Hz, 1H), 2.58 (t, *J* = 7.7 Hz, 2H), 1.65 (d, *J* = 7.2 Hz, 3H), 1.60–1.57 (m, 2H), 1.36–1.31(m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 196.5, 172.8, 148.6, 140.6, 140.5, 138.1, 137.5, 132.6, 131.6, 130.1, 129.3, 129.3, 129.2, 128.8, 128.4, 120.9, 45.5, 35.0, 33.6, 22.3, 18.6, 13.9.

HRMS: calc. for $C_{26}H_{26}O_3$ (EI), 386.1882, found, 386.1884.

3.5 Synthesis of 4-butylphenyl 4-(*N*, *N*-dipropylsulfamoyl) benzoate (S37)



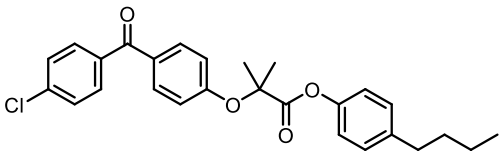
Following a modified procedure from literature [3a], a dry 100-mL round-bottom flask containing a magnetic stir bar was charged with probenecid (1.0 eq, 10.0 mmol, 2.84 g), DCC (1.1 eq, 11.0 mmol, 2.26 g), DMAP (0.2 eq, 2.0 mmol, 244.0 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 11.0 mmol, 1.64 g) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (3.25 g, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.31 (d, *J* = 8.4 Hz, 2H), 7.94 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 3.13 (t, *J* = 7.7 Hz, 4H), 2.64 (t, *J* = 7.7 Hz, 2H), 1.65–1.53 (m, 6H), 1.41–1.34 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H), 0.86 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 164.1, 148.5, 144.8, 141.0, 133.0, 130.8, 129.5, 127.2, 121.1, 50.0, 35.1, 33.6, 22.3, 22.0, 14.0, 11.2.

HRMS: calc. for $C_{23}H_{31}O_4NS$ (EI), 417.1968, found, 417.1973.

3.6 Synthesis of 4-butylphenyl 2-(4-(4-chlorobenzoyl) phenoxy)-2-methylpropanoate (S38)



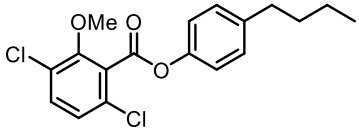
Following a modified procedure from literature [3a], a dry 100-mL round-bottom flask containing a magnetic stir bar was charged with fenofibric acid (1.0 eq, 5.0 mmol, 1.59 g), DCC (1.1 eq, 5.5 mmol, 1.13 g), DMAP (0.2 eq, 1.0 mmol, 122 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 5.5 mmol, 826 mg) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (1.85 g, 82% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.8 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 2H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.82 (s, 6H), 1.59–1.54 (m, 2H), 1.37–1.30 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 194.2, 172.6, 159.6, 148.3, 141.0, 138.5, 136.4, 132.2, 131.2, 130.6, 129.4, 128.6, 120.8, 117.4, 79.5, 35.0, 33.6, 25.5, 22.3, 13.9.

HRMS: calc. for $C_{27}H_{27}O_4Cl$ (EI), 450.1592, found, 450.1597.

3.7 Synthesis of 4-butylphenyl 3, 6-dichloro-2-methoxybenzoate (S39)



Following a modified procedure from literature [3a], a dry 100-mL round-bottom flask containing a magnetic stir bar was charged with dicamba (1.0 eq, 5.0 mmol, 1.11 g), DCC (1.1 eq, 5.5 mmol, 1.13 g), DMAP (0.2 eq, 1.0 mmol, 122 mg), and dry DCM (25.0 mL). After stirring at room temperature for 5 minutes, butylphenol (1.1 eq, 5.5 mmol, 826.0 mg) were added at ice bath. The reaction mixture was slowed to gradually warm to room temperature and stirred for 12 hours. After completion, the mixture was concentrated under reduced pressure, dissolved in EtOAc and filtered through a Celite pad. The filtrate was evaporated and purified by flash column chromatography using petroleum ether/EtOAc = 10:1 to give the product as a colorless oil (1.32 g, 75% yield).

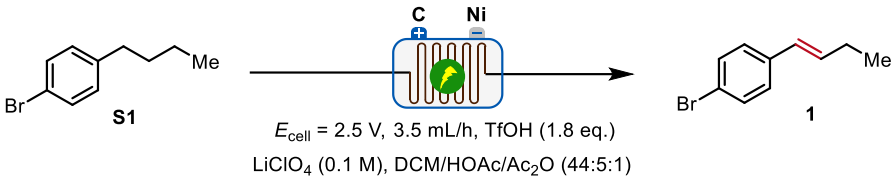
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.7 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 2H), 7.17 (d, *J* = 8.5 Hz, 3H), 4.00 (s, 3H), 2.63 (t, *J* = 7.6 Hz, 2H), 1.64 –1.58 (m, 2H), 1.40–1.34 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 163.3, 154.1, 148.4, 141.2, 132.2, 130.0, 129.9, 129.6, 126.9, 126.0, 121.1, 62.4, 35.1, 33.6, 22.3, 14.0.

HRMS: calc. for $C_{18}H_{18}O_3Cl_2$ (EI), 352.0628, found, 352.0627.

4. Optimization of Reaction Conditions

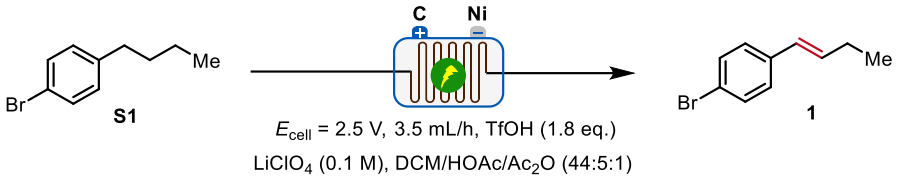
Table S1 Screening of the reaction conditions[a]



Entry	Deviation from standard conditions	Yield (%)[b]
1	**none**	**68**
2	C plate as cathode	48
3	stainless steel as cathode	44
4	I = 5 mA	25
5	I = 10 mA	50
6	E = 2.2 V	44
7	E = 2.8 V	35
8	$BF_3{\cdot}OEt_2$ instead of TfOH	trace
9	TFA instead of TfOH	trace
10	MsOH instead of TfOH	30
11	TMSOTf instead of TfOH	27
12	Without TfOH	0
13	Without Ac_2O	55
14	Without HOAc	46
15	in batch	trace
16	DCE	19
17	CH_3CN	0
18	THF	0
19	EA	9

Electrochemical dehydrogenation in continuous flow. [a] Reaction conditions: graphite plate anode (48.0 mm × 48.0 mm × 3.0 mm) and Ni cathode (48.0 mm × 48.0 mm × 3.0 mm), $LiClO_4$ (0.1 M), substrate: 0.4 mmol / 10 mL of mixed solvents (1.0 mL HOAc, 0.2 mL Ac_2O, 8.8 mL DCM), acid (1.8 eq.), RT. [b] Yields determined by ^1H NMR analysis using tetrachloroethane as the internal standard.

Table S2 Deviation from standard conditions [a]



Entry	Deviation from standard conditions	Yield (%)[b]
1	**none**	**68**
2	Pt plate as anode	28
3	Ni plate as anode	0
4	*n*-Bu_4NClO_4	24
5	Et_4NBF_4	0
6	*n*-Bu_4NBF_4	0
7	*n*-Bu_4NBr	0
8	*n*-Bu_4NPF_6	10
9	$LiClO_4$ (0.05 M)	60
10	$LiClO_4$ (0.2 M)	48
11	DCE	19
12	CH_3CN	11
13	THF	0
14	EA	9
15	DCM/HOAc/Ac_2O (88:11:1)	60
16	DCM/HOAc/Ac_2O (4:1:0)	7
17	DCM/HOAc/Ac_2O (1:1:0)	0
18	DCM/HOAc/Ac_2O (3:22:0)	0

Electrochemical dehydrogenation in continuous flow. [a] Reaction conditions: graphite plate anode (48.0 mm × 48.0 mm × 3.0 mm) and Ni cathode (48.0 mm × 48.0 mm × 3.0 mm), $LiClO_4$ (0.1 M), substrate: 0.4 mmol / 10 mL of mixed solvents (1.0 mL HOAc, 0.2 mL Ac_2O, 8.8 mL DCM), acid (1.8 eq.), RT. [b] Yields determined by ^1H NMR analysis using tetrachloroethane as the internal standard.

Method A: General procedure for the flow electrolysis

A solution (10 mL) of the arylalkanes (c = 0.4 mmol/10 mL or c = 0.2 mmol/10 mL), $LiClO_4$ (0.1M), HOAc (1.0 mL) Ac_2O (0.2 mL), DCM (8.8 mL) and TfOH (33-100 μL, 0.9-2.8 equiv., unless noted, 66 μL, 1.8 equiv. was used) was mixed and pumped through the electrochemical reactor via a syringe pump (3.5 mL/h) for 2.30~3.00 V. After the reaction reached the steady state, the solution is collected in a measuring cylinder (*Yields were based on volume of the collected solution*). The formation of bubbles in the tube can be observed during the reaction (**Figure S2**). The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

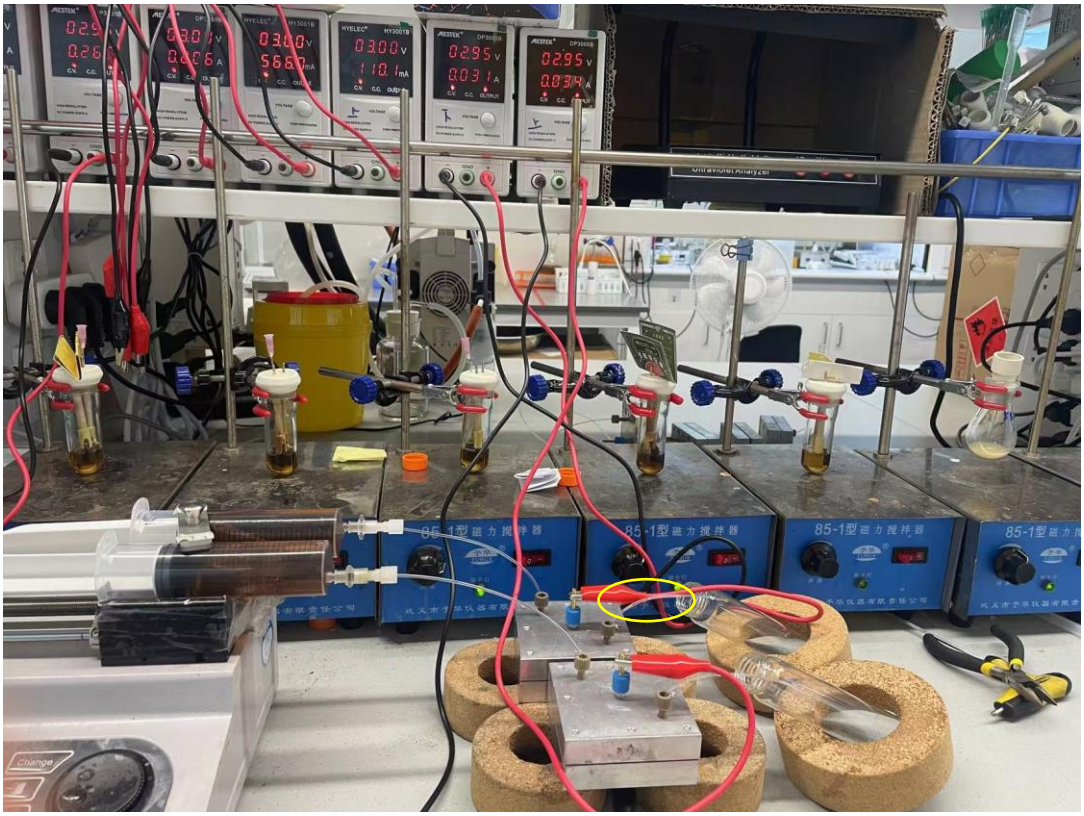


Figure S2 General procedure for the flow electrolysis

Method B: **General procedure in batch, two steps in one-pot**

In an oven-dried undivided glassware (15 mL) equipped with a stirring bar, the arylalkanes (0.3 mmol), $LiClO_4$ (0.1 M), HOAc (0.5 mL), and Ac_2O (0.1 mL) were added. The glassware was equipped with Graphite plate (15 mm × 15 mm × 0.5 mm) as the anode and Nickel plate (15 mm × 15 mm × 0.3 mm) as the cathode. Under the protection of N_2, DCM (4.4 mL) were injected into the glassware via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 6 mA at an ambient temperature for 10 h. Then, TfOH (30 μL, 1.1 equiv.) was injected into the glassware via microsyringes, the reaction mixture was continued to react for 8 h without electricity (**Figure S3**). The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Figure S3 Batch electrolysis setups.

Method C: **General procedure in batch, two steps in one-pot**

In an oven-dried undivided glassware (15 mL) equipped with a stirring bar, the arylalkanes (0.3 mmol), Et_4NBF_4 (0.1 M), HOAc (3.5 mL), Ac_2O (0.5 mL) were added. The glassware was equipped with C cloth (15 mm × 15 mm × 0.1 mm) as the anode and Pt plate (15 mm × 15 mm × 0.3 mm) as the cathode. Under the protection of N_2, DCM (1.0 mL) were injected into the glassware via syringes. The reaction mixture was stirred and electrolyzed at a constant voltage of 3.0 V at an ambient temperature for 17 h. Then, TfOH (30 μL, 1.1 equiv.) was injected into the glassware via microsyringes, the reaction mixture was continued to react for 8 h. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Method D: **General procedure in batch, two steps in one-pot**

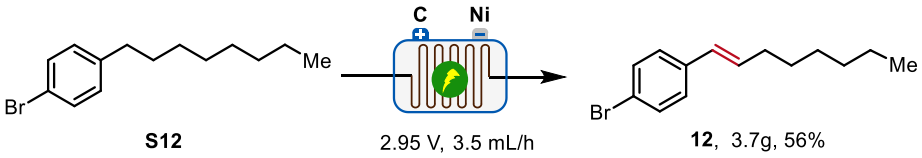
In an oven-dried undivided glassware (15 mL) equipped with a stirring bar, the arylalkanes (0.3 mmol), Et_4NBF_4 (0.1 M), HOAc (3.5 mL), Ac_2O (0.5 mL) were added. The glassware was equipped with C cloth (15 mm × 15 mm × 0.1 mm) as the anode and Pt plate (15 mm × 15 mm × 0.3 mm) as the cathode. Under the protection of N_2, DCM (1.0 mL) were injected into the glassware via syringes. The reaction mixture was stirred and electrolyzed at a constant voltage of 6.0 V at an ambient temperature for 14 h. Then, MsOH (50 μL, 2.6 equiv.) was injected into the glassware via microsyringes, the reaction mixture was continued to react for 8 h. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Method E: **General procedure in batch, two steps in one-pot**

In an oven-dried undivided glassware (15 mL) equipped with a stirring bar, the arylalkanes (0.3 mmol), Et_4NBF_4 (0.1 M), HOAc (0.5 mL), Ac_2O (0.1 mL), TFA (50 μL, 1.6 equiv.) were added.

The glassware was equipped with C cloth (15 mm × 15 mm × 0.1 mm) as the anode and Pt plate (15 mm × 15 mm × 0.3 mm) as the cathode. Under the protection of N_2, DCM (4.4 mL) were injected into the glassware via syringes. The reaction mixture was stirred and electrolyzed at a constant voltage of 4.5 V at an ambient temperature for 10 h. Then, TfOH (30 μL, 1.1 equiv.) was injected into the glassware via microsyringes, the reaction mixture was continued to react for 8 h. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Method F: **Gram-scale flow synthesis of products 12 and 24**



The gram-scale electrolysis was conducted with a constant current of 2.95 V using four flow electrolytic cells, which was equipped with a graphite anode and a Ni cathode and interelectrode distance of 250 μm (**Figure S1**). The substrate (0.8 mmol/10 mL) and $LiClO_4$ (0.1 M) were dissolved in HOAc/Ac_2O/DCM/TfOH (5/1/44/1), the reactant was pushed using a syringe pump to pass through the flow electrolytic cell with a flow rate of 3.5 mL/h. In the beginning the system was stabilized for 2 min. After steady state was reached, the outlet solution was collected and four flow cells were carried out simultaneously. The reaction mixture was concentrated in vacuo, and the crude residue **12** was subjected to flash column chromatography on silica gel to yield the desired product (56%, 3.7 g), (**Figure S4**).

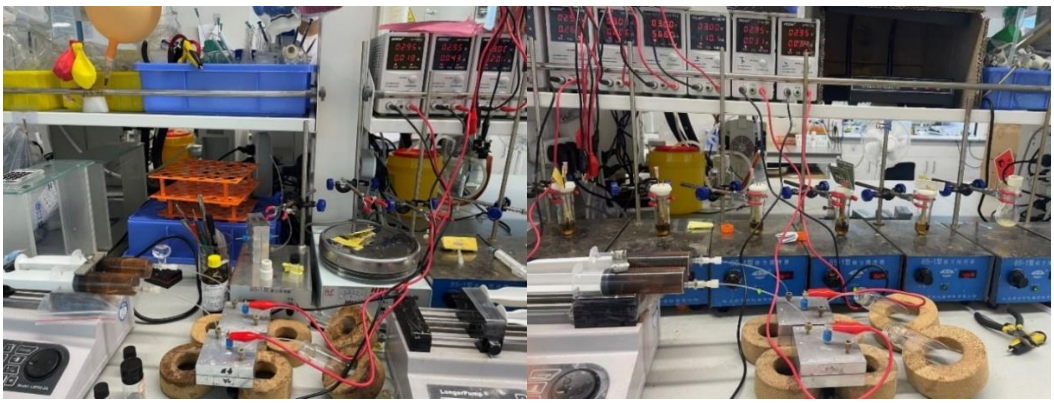
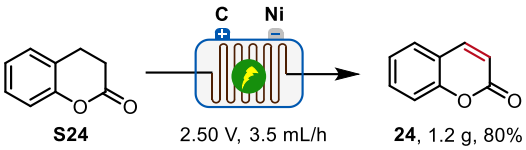


Figure S4 Gram-scale flow synthesis of products **12**.



The gram-scale electrolysis was conducted with a constant current of 2.50 V using four flow electrolytic cells, which was equipped with a graphite anode and a Ni cathode and interelectrode distance of 250 μm (**Figure S1**). The substrate **S24** (0.4 mmol/10 mL) and $LiClO_4$ (0.1 M) were dissolved in HOAc/Ac_2O/DCM/TfOH (1/0.2/8.8/0.066), the reactant was pushed using a syringe pump to pass through the flow electrolytic cell with a flow rate of 3.5 mL/h. In the beginning the system was stabilized for 2 min. After steady state was reached, the outlet solution was collected and four flow cells were carried out simultaneously. The reaction mixture was concentrated in vacuo, and the crude residue **24** was subjected to flash column chromatography on silica gel to yield the desired product (80%, 1.20 g), (**Figure S5**).

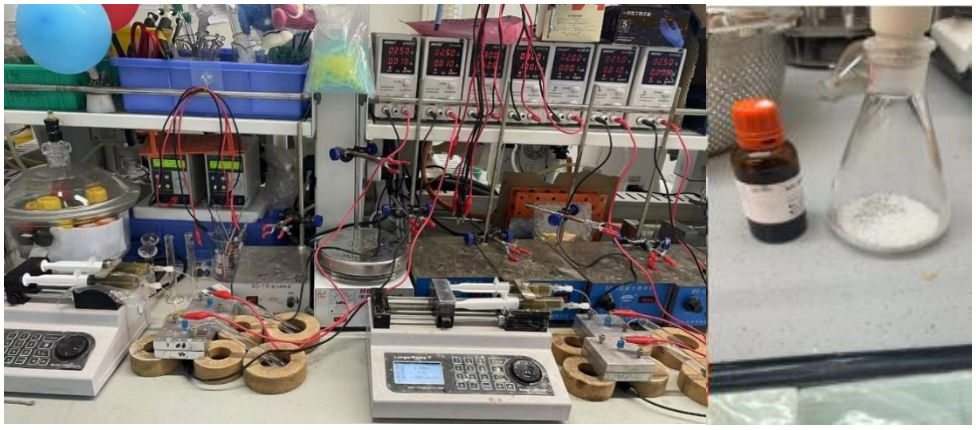
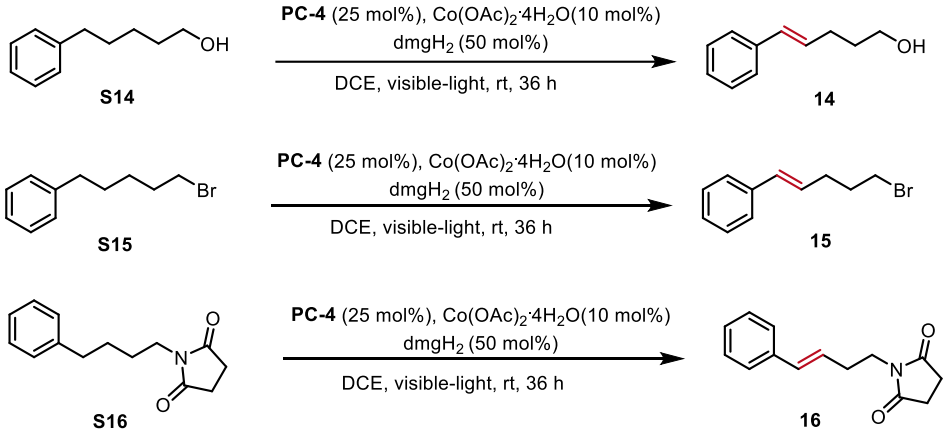


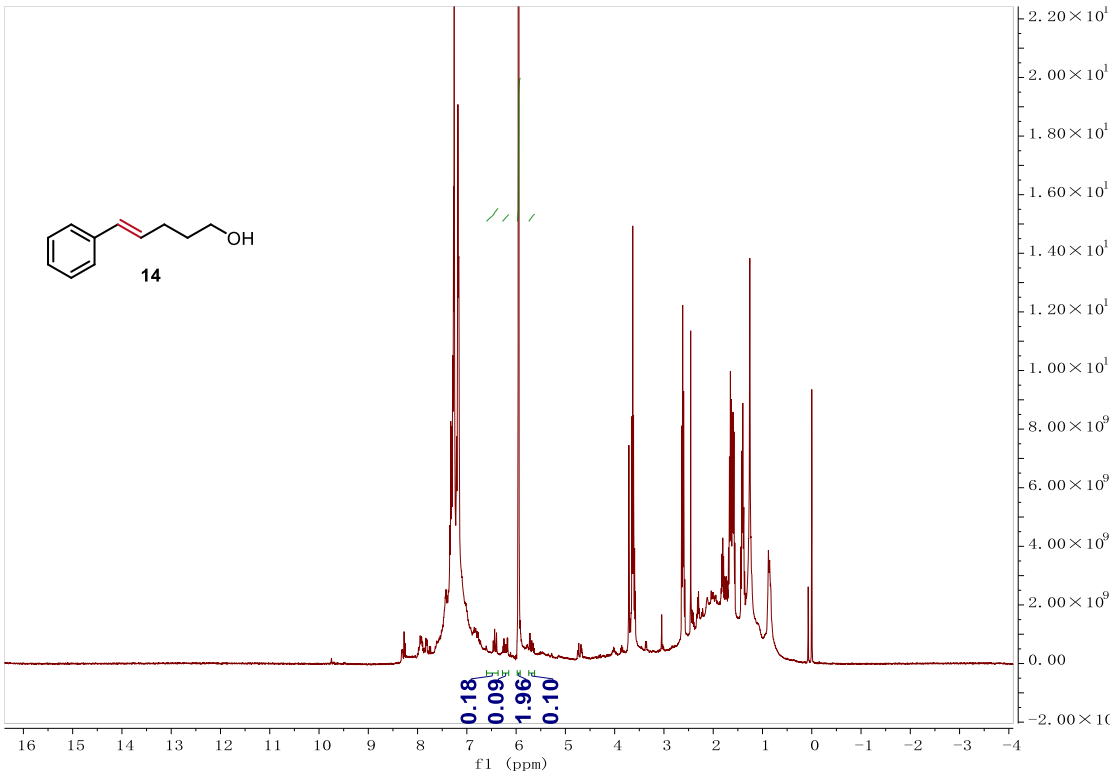
Figure S5 Gram-scale flow synthesis of products **24**

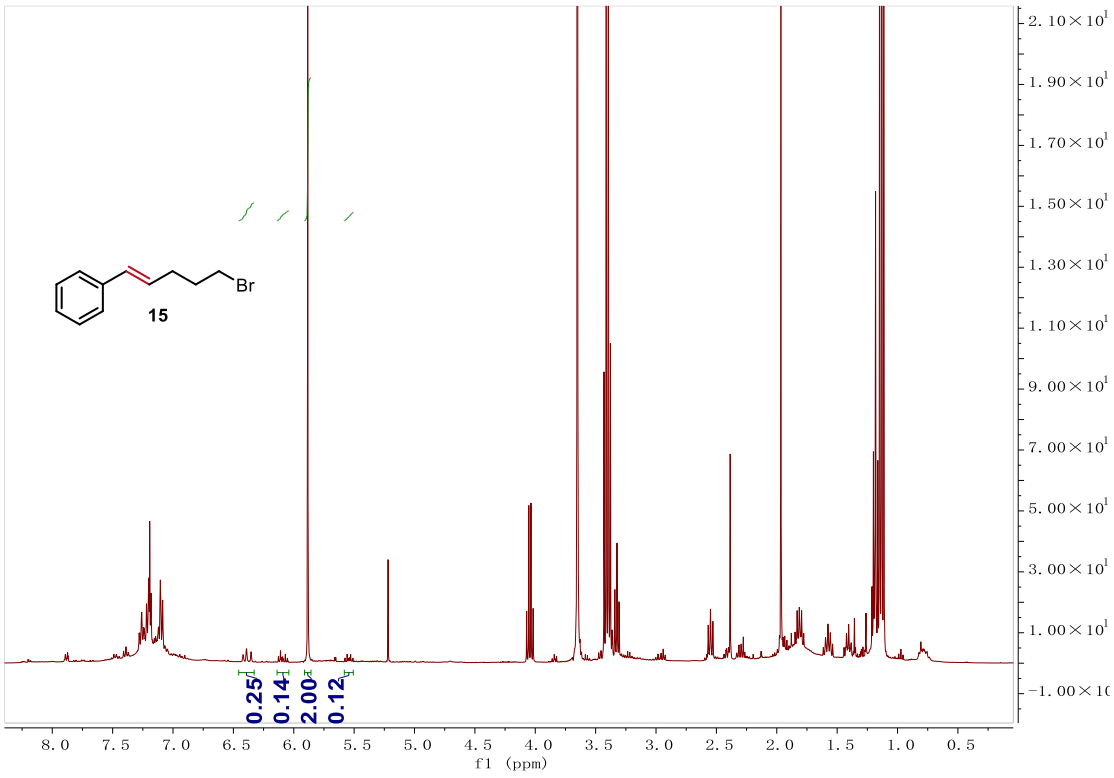
Method G: General procedure for substrates **S14**-**S16** with polar functional groups [3a]

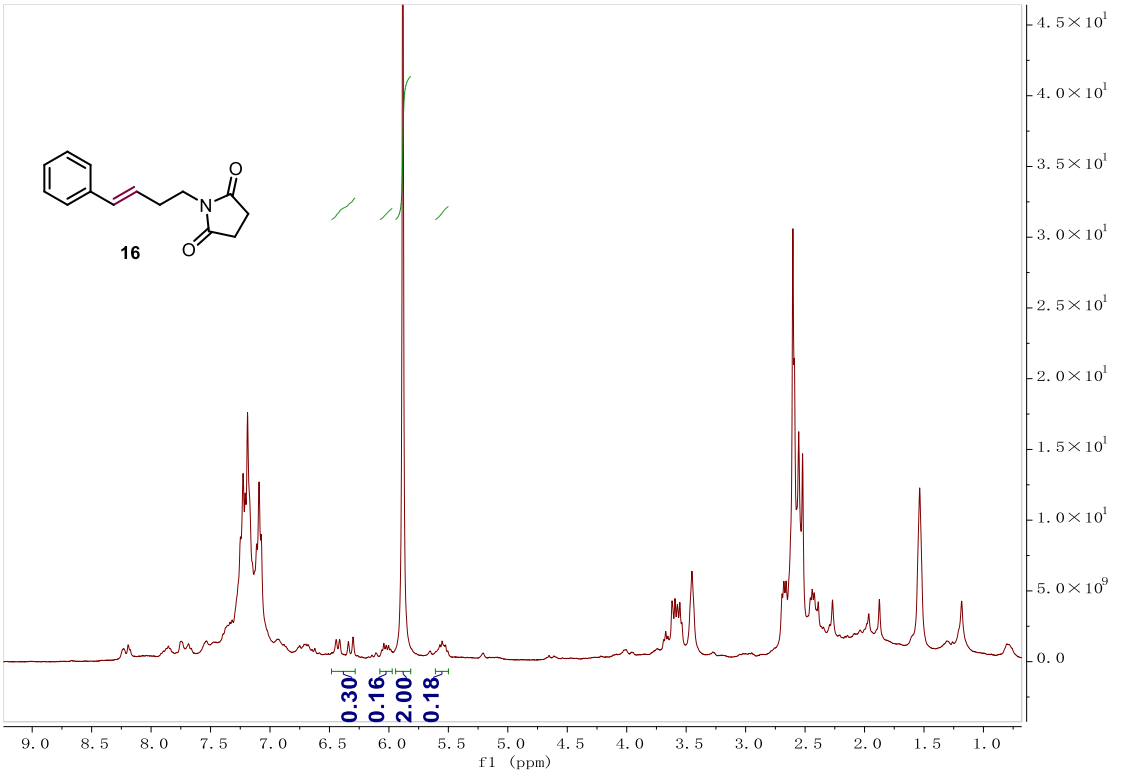


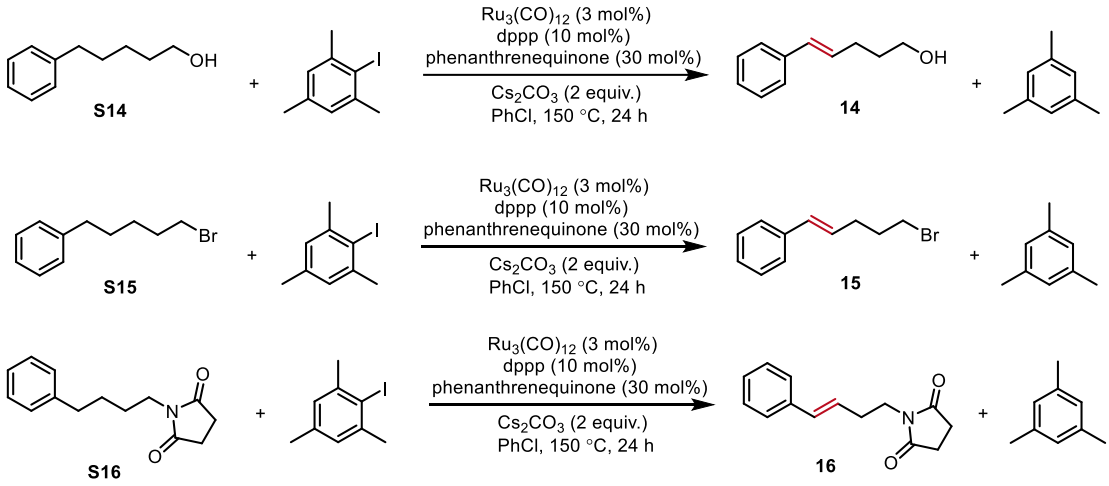
In argon-filled glovebox, to an oven-dried 10 mL Schlenk tube equipped with a stir bar was added 2- chloroanthraquinone (0.025 mmol, 6.1 mg), $Co(OAc)_2{\cdot}4H_2O$ (0.01 mmol, 2.5 mg), $dmgH_2$ (0.05 mmol, 5.8 mg), **S14** (0.1 mmol, 22.7 mg) and DCE (8 mL), The reaction mixture was sealed and stirred in dark for 30 minutes at room temperature. Then, the reaction was irradiated with two 20 W

400-415 nm LEDs for 36 hours (tube 2 cm away from lights, fans for cooling, 20-30 ℃). After that, the solvent of the reaction mixture was removed on a rotary evaporator under reduced pressure. Yields was determined by ^{1}H NMR analysis using tetrachloroethane (0.1 mmol, 10.6 μL) as the internal standard.



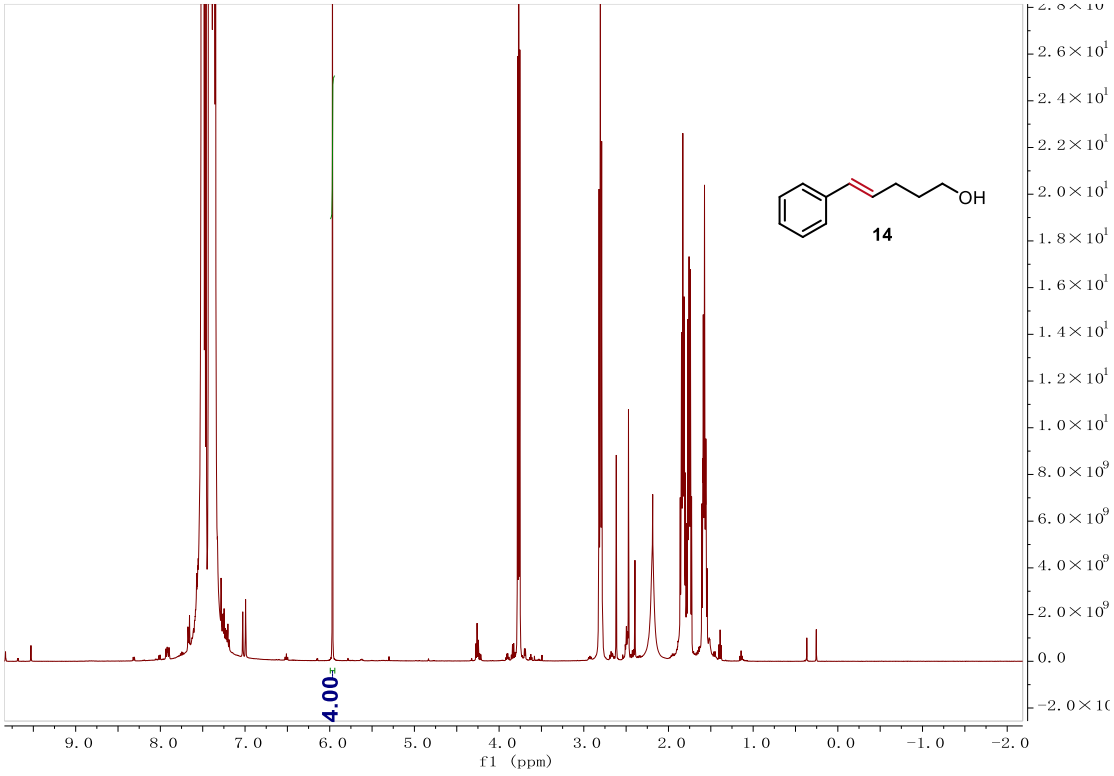


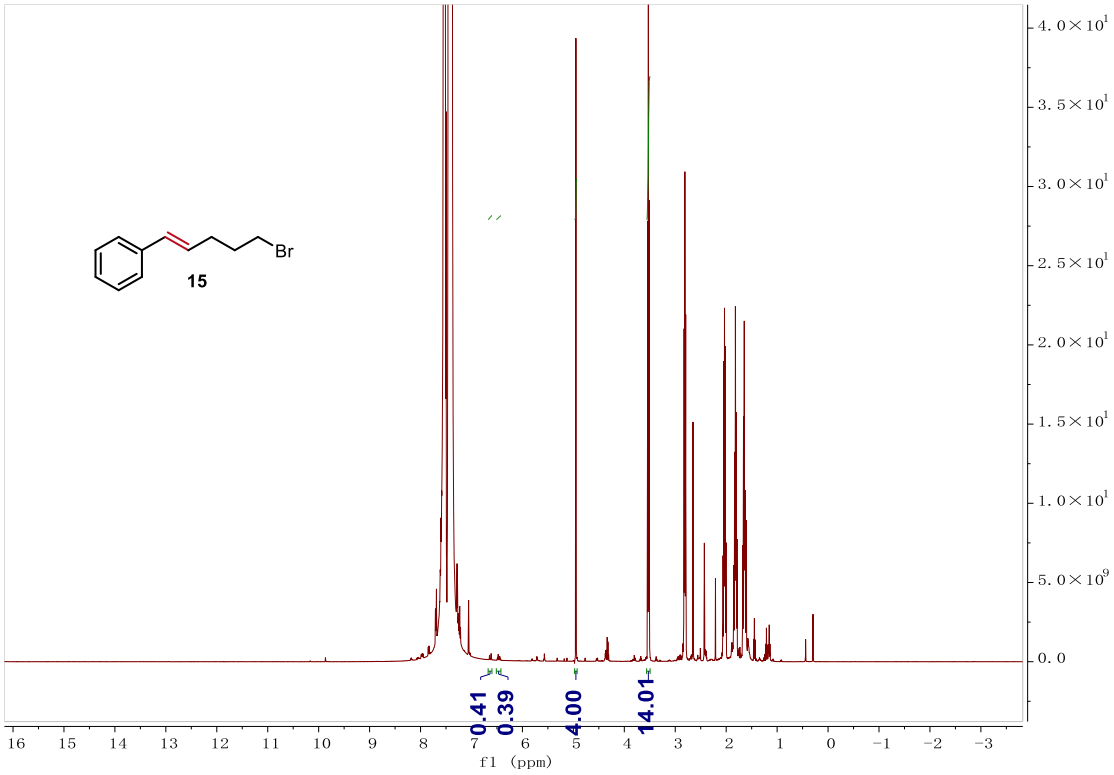


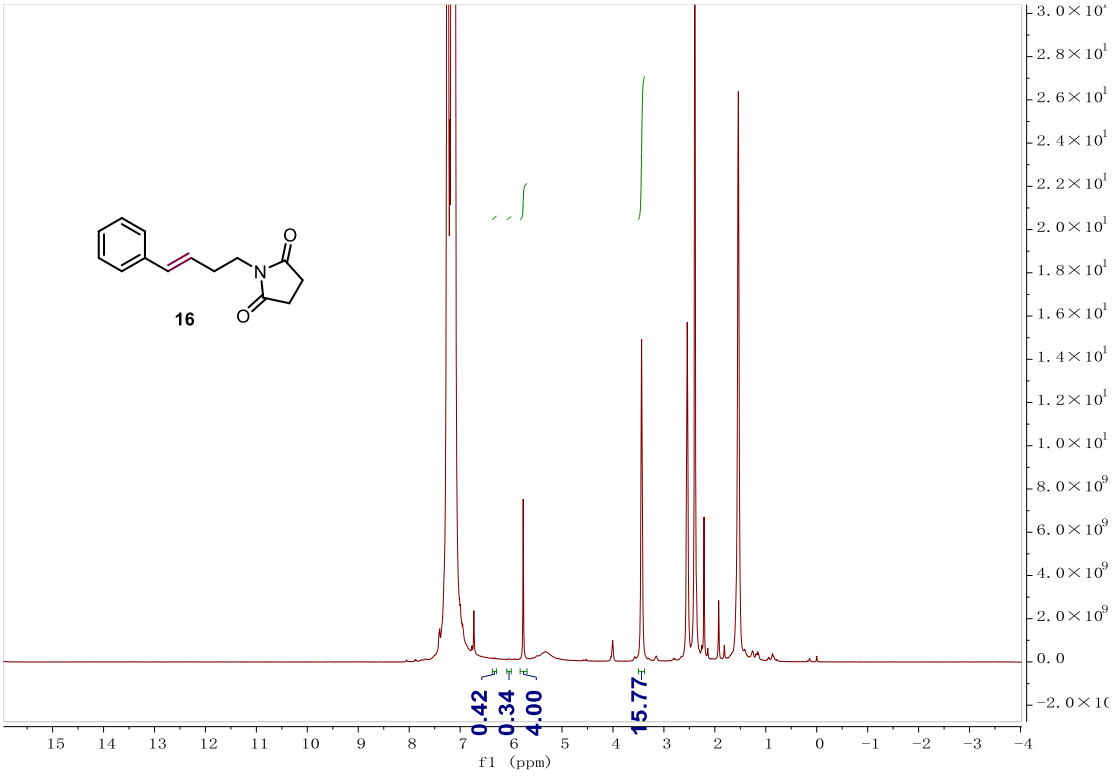


Method H: General procedure for substrates **S14-16** with polar functional groups [3b]

In an argon-filled glovebox, $Ru_3(CO)_{12}$ (3 mol%, 0.001 mmol, 0.8 mg), dppp (10 mol%, 0.004 mmol, 1.6 mg), additive phenanthrenequinone (30 mol%, 0.012 mmol, 2.5 mg), 2-iodo-1,3,5-trimethyl benzene (0.04 mmol, 10 mg), Cs_2CO_3 (0.08 mmol, 26 mg), and **S14** (0.6 mmol, 98.5 mg) were added into an oven-dried 8 mL vial with a magnetic stirring bar, followed by addition of chlorobenzene (1.0 mL). The vial was sealed and removed from the glovebox and was heated at 150 °C. After 24 h, the vial was cooled to room temperature. The reaction was quenched by exposing the solution to air and diluted with chloroform-*d*. Yields was determined by ^{1}H NMR analysis using dibromomethane (0.16 mmol, 11.2 μL) as the internal standard. was added to the solution as an internal standard.

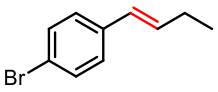

OH
14
4.00
f1 (ppm)


Br
15
0.41
0.39
4.00
14.01
f1 (ppm)



5. Characterization of Products

5.1 Synthesis of (*E*)-1-bromo-4-(but-1-en-1-yl) benzene (1)



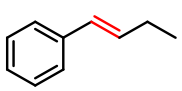
Following **Method A** (E = 2.50 V), the reaction of 1-bromo-4-butylbenzene **S1** in flow electrolytic cell afforded 49.2 mg (58% yield) of **1** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [4].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41–7.39 (m, 2H), 7.21–7.20 (m, 2H), 6.31 (d, *J* = 16.0 Hz, 1H), 6.26 (dt, *J* = 15.8, 5.7 Hz, 1H), 2.25–2.19 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 133.6, 131.5, 127.7, 127.5, 120.4, 26.1, 13.5.

5.2 Synthesis of (*E*)-but-1-en-1-ylbenzene (2)



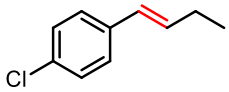
Following **Method A** (E = 2.50 V), the reaction of butylbenzene **S2** in flow electrolytic cell afforded 30.0 mg (56% yield) of **2** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [5].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.34 (m, 2H), 7.31–7.27 (m, 2H), 7.20–7.17 (m, 1H), 6.38 (d, *J* = 15.9 Hz, 1H), 6.27 (dt, *J* = 15.8, 6.4 Hz, 1H), 2.26–2.21 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 138.0, 132.7, 128.8, 128.5, 126.8, 125.9, 26.1, 13.7.

5.3 Synthesis of (*E*)-1-(but-1-en-1-yl)-4-chlorobenzene (3)



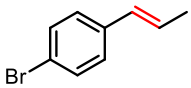
Following **Method A** (E = 2.50 V), the reaction of 1-butyl-4-chlorobenzene **S3** in flow electrolytic cell afforded 28.6 mg (73% yield) of **3** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [6].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26 (s, 4H), 6.33 (d, *J* = 15.9 Hz, 1H), 6.25 (dt, *J* = 15.9, 6.1 Hz, 1H), 2.23 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.5, 133.4, 132.3, 128.6, 127.7, 127.1, 26.0, 13.5.

5.4 Synthesis of (*E*)-1-bromo-4-(prop-1-en-1-yl) benzene (4)



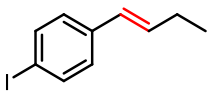
Following **Method A** (E = 2.36 V), the reaction of 1-bromo-4-propylbenzene **S4** in flow electrolytic cell afforded 21.0 mg (55% yield) of **4** a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [7].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.33 (d, *J* = 15.9, 1H), 6.23 (dt, *J* = 15.7, 6.4 Hz, 1H), 1.87 (dd, *J* = 6.5, 1.1 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 131.5, 129.9, 127.4, 126.7, 120.4, 18.5.

5.5 Synthesis of (*E*)-1-(but-1-en-1-yl)-4-iodobenzene (5)



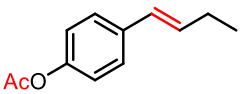
Following **Method A** (E = 2.50 V), the reaction of 1-butyl-4-iodobenzene **S5** in flow electrolytic cell afforded 31.0 mg (52% yield) of **5** as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (d, *J* = 10.5 Hz, 2H), 7.08 (d, *J* = 10.5 Hz, 2H), 6.28 (d, *J* = 10.5 Hz, 2H), 2.25–2.18 (m, 2H), 1.09 (t, *J* = 9.3 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 137.5, 133.7, 127.8,127.8, 91.7, 26.1, 13.5.

HRMS: calc. for $C_{10}H_{11}I$ (EI), 257.9900, found, 257.9899.

5.6 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl acetate (6)



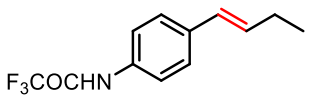
Following **Method A** (E = 2.25 V), the reaction of 4-butylphenol **S6** (c = 0.2 mmol/10 mL) in flow electrolytic cell afforded 20.1 mg (40% yield) of **6** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [7].

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.34 (d, *J* = 8.6 Hz, 2H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.35 (d, *J* = 15.9 Hz, 1H), 6.22 (dt, *J* = 15.8, 6.4 Hz, 1H), 2.29 (s, 3H), 2.25–2.20 (m, 2H), 1.08 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 169.6, 149.4, 135.8, 133.0, 127.8, 126.8, 121.5, 26.1, 21.2, 13.6.

5.7 Synthesis of (*E*)-*N*-(4-(but-1-en-1-yl) phenyl)-2,2,2-trifluoroacetamide (7)



Following **Method A** (E = 2.30 V), the reaction of *N*-(4-butylphenyl)-2, 2, 2-trifluoroacetamide **S7** in flow electrolytic cell afforded 24.1 mg (34 % yield) of **7** as a white solid.

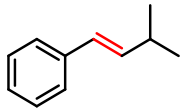
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 (brs, 1H), 7.49 (d, *J* = 8.7 Hz, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.27 (dt, *J* = 15.8, 6.2 Hz, 1H); 2.24 (m, 2H), 1.10 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 154.6 (q, *J* = 36.9 Hz), 136.3, 133.5, 133.5, 127.7, 126.7, 120.5, 115.8 (q, *J* = 286.3 Hz), 26.1, 13.6.

^{19}F NMR (471 MHz, $CDCl_3$): -75.7.

HRMS: calc. for $C_{12}H_{12}ONF_3$ (EI), 243.0866, found, 243.0866.

5.8 Synthesis of (*E*)-(3-methylbut-1-en-1-yl) benzene (8)



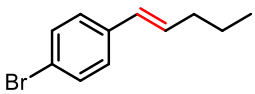
Following **Method A** (E = 2.50 V), the reaction of isopentylbenzene **S8** in flow electrolytic cell afforded 17.9 mg (45 % yield) of **8** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [8].

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.35 (d, *J* = 7.8 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.19 (t, *J* = 7.3 Hz, 1H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.20 (dd, *J* = 15.9, 6.8 Hz, 1H), 2.50-2.44 (m, 1H), 1.10 (d, *J* = 6.8 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 138.0, 138.0, 128.5, 126.8, 126.8, 126.0, 31.5, 22.5.

5.9 Synthesis of (*E*)-1-bromo-4-(pent-1-en-1-yl) benzene (9)



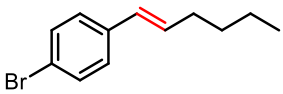
Following **Method A** (E = 2.50 V, TfOH, 100 μL), the reaction of 1-bromo-4-pentylbenzene **S9** in flow electrolytic cell afforded 30.4 mg (52% yield) of **9** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [9].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41–7.39 (m, 2H), 7.21–7.19 (m, 2H), 6.31 (d, *J* = 15.9 Hz, 1H), 6.22 (dt, *J* = 15.8, 6.8 Hz, 1H), 2.17 (q, *J* = 7.1 Hz, 2H), 1.53–1.45 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 131.9, 131.5, 128.8, 127.5, 120.4, 35.1, 22.4, 13.7.

5.10 Synthesis of (*E*)-1-bromo-4-(hex-1-en-1-yl) benzene (10)



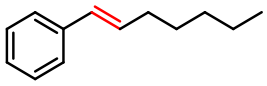
Following **Method A** (E = 2.50 V), the reaction of 1-bromo-4-hexylbenzene **S10** in flow electrolytic cell afforded 35.9 mg (58% yield) of **10** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [10].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41–7.39 (m, 2H), 7.21–7.19 (m, 2H), 6.31 (d, *J* = 15.9 Hz, 1H), 6.26 – 6.19 (m, 1H), 2.20 (q, *J* = 7.1 Hz, 2H), 1.48–1.42 (m, 2H),1.40–1.26 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 132.1, 131.5, 128.6, 127.5, 120.4, 32.7, 31.4, 22.3, 14.0.

5.11 Synthesis of (*E*)-hept-1-en-1-ylbenzene (11)



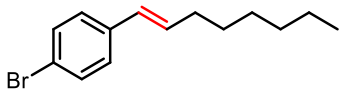
Following **Method A** (E = 2.75 V), the reaction of heptylbenzene **S11** in flow electrolytic cell afforded 22.0 mg (53 % yield) of **11** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [11].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36–7.34 (m, 2H), 7.31–7.28 (m, 2H), 7.21–7.18 (m, 1H), 6.39 (d, *J* = 15.8, 1H), 6.24 (dt, *J* = 15.7, 6.9 Hz, 1H), 2.21 (q, *J* = 7.3, 6.9 Hz, 2H), 1.51–1.45 (m, 2H), 1.35–1.27 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 138.0, 131.3, 129.7, 128.5, 126.8, 125.9, 33.0, 31.5, 29.1, 22.6, 14.1.

5.12 Synthesis of (*E*)-1-bromo-4-(oct-1-en-1-yl) benzene (12)



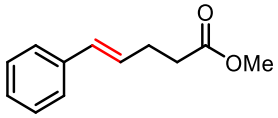
Following **Method A** (E = 2.60 V), the reaction of 1-bromo-4-octylbenzene **S12** in flow electrolytic cell afforded 45.0 mg (68 % yield) of **12** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.5 Hz, 2H), 6.31 (d, *J* = 15.9 Hz, 1H), 6.22 (dt, *J* = 15.7, 6.7 Hz, 1H), 2.19 (q, *J* = 6.9 Hz, 2H), 1.49–1.43 (m, 2H), 1.36–1.29 (m, 6H), 0.90 (t, *J* = 6.6 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 136.9, 132.2, 131.5, 128.6, 127.5, 120.4, 33.1, 31.8, 29.2, 28.9, 22.7, 14.1.

HRMS: calc. for $C_{14}H_{19}Br_2$ (EI), 266.0665, found, 266.0663.

5.13 Synthesis of methyl (*E*)-5-phenylpent-4-enoate (13)



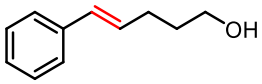
Following **Method A** (E = 2.80 V), the reaction of methyl 5-phenylpentanoate **S13** in flow electrolytic cell afforded 27.0 mg (59 % yield) of **13** as a yellow oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [12].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.33 (m, 2H), 7.31–7.28 (m, 2H), 7.22–7.18 (m, 1H), 6.43 (d, *J* = 15.9, 1H), 6.24 (dt, *J* = 15.9, 6.5 Hz, 1H), 3.69 (s, 3H), 2.57–2.53 (m, 2H), 2.51–2.48 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 173.4, 137.4, 131.0, 128.5, 128.4, 127.2, 126.1, 51.6, 33.8, 28.3.

5.14 Synthesis of (*E*)-5-phenylpent-4-en-1-ol (14)



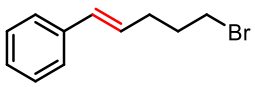
5-Phenylpentyl acetate **S14** was stirred in mixed solution of $LiClO_4$ (0.1 M) in HOAc (1.0 mL), Ac_2O (0.2 mL), DCM (8.8 mL) and TfOH (1.8 equiv.) for 4 h at RT, then continued in flow electrolytic cell following **Method A** (E = 2.55 V) for 170 min. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was stirred solution in MeOH (15 mL), K_2CO_3 (138.5 mg, 1.0 mmol, 3.0 eq) was added, the resulting mixture was stirred about 6 h at room temperature. The mixture was quenched with saturated NH_4Cl solution (20 mL). The solvent was removed, and residual was extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure to afford the title primary alcohol **14**（20.3 mg, 48%, over two steps) as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [13].

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.35 –7.34 (m, 2H), 7.31–7.28 (m, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 6.43 (d, *J* = 15.9, 1H), 6.24 (dt, *J* = 15.7, 6.9 Hz, 1H); 3.72 (t, *J* = 6.5 Hz, 2H), 2.34–2.30 (m, 2H), 1.79–1.73 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 137.6, 130.4, 130.1, 128.5, 127.0, 126.0, 62.5, 32.3, 29.3.

5.15 Synthesis of (*E*)-(5-bromopent-1-en-1-yl) benzene (15)



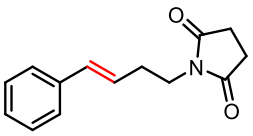
Following **Method A** (E = 2.40 V, TfOH, 100 μL), the reaction of (5-bromopentyl) benzene **S15** (c = 0.2 mmol/10 mL) in flow electrolytic cell afforded 13.7 mg (41% yield) of **15** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [14].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.34 (m, 2H), 7.31–7.28 (m, 2H), 7.22–7.20 (m, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.17 (dt, *J* = 15.9, 7.0 Hz, 1H), 3.46 (t, *J* = 6.7 Hz, 2H), 2.38 (dq, *J* = 1.4, 7.1 Hz, 2H), 2.03 (quint, *J* = 6.8 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 137.4, 131.3, 128.5, 128.4, 127.1, 126.0, 33.2, 32.2, 31.3.

5.16 Synthesis of (*E*)-1-(4-phenylbut-3-en-1-yl) pyrrolidine-2, 5-dione (16)



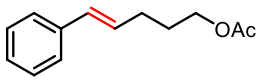
Following **Method B** (I = 6 mA, TfOH, 30 μL), the reaction of 1-(4-phenylbutyl) pyrrolidine-2, 5-dione **S16** in undivided cell afforded 23.4 mg (34% yield) of **16** as a white solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [15].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32− 7.26 (m, 4H), 7.22− 7.15 (m, 1H), 6.39 (d, *J* = 15.8 Hz, 1H), 6.13− 6.07 (m, 1H), 3.67 (t, *J* = 7.1 Hz, 2H), 2.66 (s, 4H), 2.53− 2.48 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 177.2, 137.1, 132.6, 128.6, 127.3, 126.1, 126.0, 38.2, 31.3, 28.1.

5.17 Synthesis of (*E*)-5-phenylpent-4-en-1-yl acetate (17)



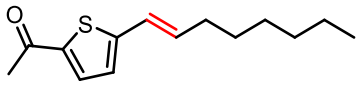
Following **Method A** (E = 2.55 V), the reaction of 5-phenylpentyl acetate **S17** in flow electrolytic cell afforded 29.8 mg (55% yield) of **17** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [16].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 –7.29 (m, 4H), 7.22–7.19 (m, 1H), 6.41 (d, *J* = 15.8 Hz, 1H), 6.41 (dt, *J* = 15.8, 6.9 Hz, 1H); 4.12 (t, *J* = 6.6 Hz, 2H), 2.32–2.28 (m, 2H), 2.06 (s, 3H), 1.85–1.79 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 171.2, 137.6, 130.7, 129.4, 128.5, 127.1, 126.0, 64.0, 29.4, 28.3, 21.0.

5.18 Synthesis of (*E*)-1-(5-(oct-1-en-1-yl) thiophen-2-yl) ethan-1-one (18)



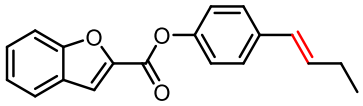
Following **Method B** (I = 6 mA, TfOH, 30μL), the reaction of 1-(5-octylthiophen-2-yl) ethan-1-one **S18** in undivided cell afforded 22.0 mg (41% yield) of **18** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.52 (d, *J* = 3.9 Hz, 1H), 6.86 (d, *J* = 3.9 Hz, 1H), 6.47 (d, *J* = 15.7 Hz, 1H), 6.31–6.25 (m, 1H), 2.51 (s, 3H), 2.19 (q, *J* = 14.5 Hz, 2H), 1.48–1.43 (m, 2H), 1.36–1.29 (m, 6H), 0.89 (t, *J* = 6.3 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 190.5, 151.7, 141.3, 135.7, 133.2, 124.9, 122.7, 33.0, 31.7, 28.9, 28.9, 26.5, 22.6, 14.1.

HRMS: calc. for $C_{14}H_{20}OS$ (EI), 236.1229, found, 236.1230.

5.19 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl benzofuran-2-carboxylate (19)



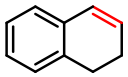
Following **Method A** (E = 2.40 V), the reaction of 4-butylphenyl benzofuran-2-carboxylate **S19** afforded 36% NMR yield of **19** as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.7 Hz, 2H), 7.64 (d, *J* = 8.5 Hz, 1H), 7.51–7.49 (m, 1H), 7.41 (d, *J* = 8.6 Hz, 2H), 7.36–7.33 (m, 1H), 7.20 (d, *J* = 8.6 Hz, 2H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.27 (dt, *J* = 15.8, 6.4 Hz, 1H), 2.28–2.22 (m, 2H), 1.82 (s, 6H), 1.11 (d, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.0, 156.1, 149.0, 144.9, 136.2, 133.2, 128.1, 127.8, 127.0, 124.0, 123.1, 121.5, 115.4, 112.5, 26.1, 13.6.

HRMS: calc. for $C_{19}H_{16}O_3$ (EI) 292.1094, found, 292.1094.

5.20 Synthesis of 1, 2-dihydronaphthalene (20)



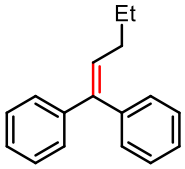
Following **Method C** (E = 3.00 V, TfOH, 30 μL), the reaction of 1, 2, 3, 4-tetrahydronaphthalene **S20** afforded 19.8 mg (50%) of **20** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [17].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.19–7.09 (m, 3H), 7.05–7.03 (m, 1H), 6.48 (dt, *J* = 9.6 Hz, 1.8 Hz, 1H), 6.05 (dt, *J* = 9.6, 4.4 Hz, 1H), 2.82 (t, *J* = 8.2 Hz, 2H), 2.36–2.32 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ δ 135.5, 134.2, 128.7, 127.8, 127.6, 126.9, 126.5, 125.9, 27.5, 23.2.

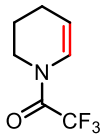
5.21 Synthesis of pent-1-ene-1,1-diyldibenzene (21)



Following **Method A** (E = 3.46 V), the reaction of pentane-1,1-diyldibenzene **S21** afforded 38% NMR yield of **21** as a white solid. [18, 19]

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38-7.16 (m, 10H), 6.09 (t, *J* = 7.5 Hz, 1H), 2.12-2.06 (m, 2H), 1.46 (q, *J* = 7.5 Hz, 2H), 0.90 (t, *J* = 7.5 Hz, 3H).

5.23 Synthesis of 1-(3,4-dihydropyridin-1(2H)-yl)-2,2,2-trifluoroethan-1-one (23)



Following **Method E** (E = 4.50 V, TfOH, 30 μL), the reaction of 2,2,2-trifluoro-1-(piperidin-1-yl)ethan-1-one **S23** afforded 36% NMR yield of **23** as a colorless oil.

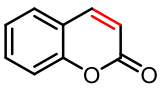
^{1}H NMR (500 MHz, $CDCl_3$, rotamers are present) δ 7.05 (dt, *J* = 8.5, *J* = 2.0 Hz, 0.6H), 6.70–6.67 (m, 1H), 5.40–5.36 (m, 0.6H) , 5.20–5.17 (m, 1H), 3.76 (t, *J* = 5.8, 2H), 3.73 (t, *J* = 5.6, 1.2H), 2.17–2.12 (m, 3.2H), 1.94–1.88 (m, 3.2H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers are present) δ 154.5 (d, *J* = 13.6 Hz), 154.2 (d, *J* = 15.2 Hz), 123.6, 123.1 (q, *J* = 4.5 Hz), 117.5, 115.2, 114.1, 112.4, 43.7 (q, *J* = 3.6 Hz), 42.0, 21.8, 21.7, 21.1.

^{19}F NMR (471 MHz, $CDCl_3$): -68.9, -69.2.

HRMS: calc. for $C_7H_8NOF_3$ (EI) 179.0553, found, 179.0557.

5.24 Synthesis of 2*H*-chromen-2-one (24)



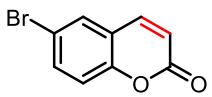
Following **Method A** (E = 2.50 V), the reaction of chroman-2-one **S24** in flow electrolytic cell afforded 30.8 mg (81% yield) of **24** as a white solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [20].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.72 (d, *J* = 9.5 Hz, 1H), 7.54 (t, *J* = 7.8 Hz, 1H), 7.50 (d, *J* = 7.7 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 6.44 (dd, *J* = 9.6, 1.7 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.8, 154.1, 143.5, 131.9, 127.9, 124.5, 118.9, 116.9, 116.7.

5.25 Synthesis of 6-bromo-2*H*-chromen-2-one (25)



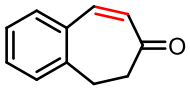
Following **Method A** (E = 2.80 V), the reaction of 6-bromochroman-2-one **S25** in flow electrolytic cell afforded 28.2 mg (60 % yield) of **25** as a white solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [21].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.64–7.61 (m, 3H), 7.23 (d, *J* = 9.7 Hz, 1H), 6.46 (d, *J* = 9.6 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.0, 153.0, 142.1, 134.6, 130.2, 120.4, 118.7, 117.9, 117.0.

5.26 Synthesis of 5, 6-dihydro-7*H*-benzo [7] annulen-7-one (26)



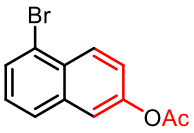
Following **Method B** (I = 6 mA, TfOH, 30μL), the reaction of 5, 6, 8, 9-tetrahydro-7*H*-benzo [7] annulen-7-one **S26** in flow electrolytic cell afforded 15.1 mg (30% yield) of **26** as a colorless oil.

^{1}H NMR and ^{13}C NMR data match previously reported data [22].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40–7.39 (m, 1H), 7.36–7.34 (m, 2H), 7.31–7.30 (m, 1H), 6.22 (d, *J* = 12.6 Hz, 1H), 3.04 (t, *J* = 5.9 Hz, 2H), 2.80 (t, *J* = 5.9 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 201.4, 142.9, 141.8, 134.3, 132.6, 130.2, 129.3, 129.1, 127.2, 42.2, 29.4.

5.28 Synthesis of 5-bromonaphthalen-2-yl acetate (28)



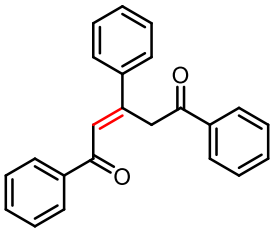
Following **Method A** (E = 2.50 V), the reaction of 5-bromo-3, 4-dihydronaphthalen-2(1*H*)-one **27** in flow electrolytic cell afforded 28.6 mg (49 % yield) of **28** as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (m, 1H), 7.75 (d, *J* = 8.9 Hz, 1H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.56–7.53 (m, 2H), 7.26–7.24 (m, 1H), 2.35 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 169.5, 148.6, 132.5, 132.2, 130.0, 129.8, 129.3, 128.5, 122.3, 119.6, 118.7, 21.2.

HRMS: calc. for $C_{10}H_7OBr$ (EI, M-Ac), 221.9675, found, 221.9669.

5.29 Synthesis of (*E*)-1, 3, 5-triphenylpent-2-ene-1, 5-dione (29)



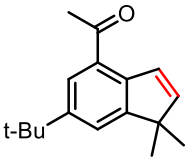
Following **Method A** (E = 2.40 V), the reaction of 4-butylphenol **S29** in flow electrolytic cell afforded 35.2 mg (47% yield) of **29** as a red solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [23].

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.08 (d, *J* = 7.2 Hz, 2H), 7.99 (d, *J* = 7.2 Hz, 2H), 7.60 –7.53 (m, 4H), 7.50 –7.45 (m, 5H), 7.43 –7.39 (m, 3H), 4.87 (s, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 196.2, 191.0, 152.8, 142.1, 139.1, 137.1, 133.2, 132.8, 129.4, 128.8, 128.7, 128.6, 128.3, 126.9, 123.7, 43.0.

5.30 Synthesis of 1-(6-(*tert*-butyl)-1, 1-dimethyl-1H-inden-4-yl) ethan-1-one (30)



Following **Method B** (I = 6 mA, TfOH, 30 μL), the reaction of 1-(6-(tert-butyl)-1,1-dimethyl-2,3-dihydro-1*H*-inden-4-yl) ethan-1-one **S30** in undivided cell afforded 37.3 mg (51 % yield) of **30** as a yellow solid.

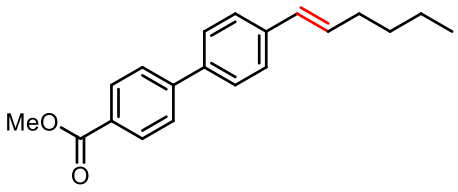
^{1}H NMR and ^{13}C NMR data match previously reported data [3a].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73 (d, *J* = 1.6Hz, 1H), 7.50 (d, *J* = 1.3Hz, 1H), 7.36 (d, *J* = 5.6Hz, 1H), 6.52 (d, *J* = 5.6Hz,1H), 2.64 (s, 3H), 1.39 (s, 9H), 1.32 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 200.1, 155.0, 150.2, 148.2, 140.0, 130.3, 127.7, 124.4, 122.3, 49.0, 34.8, 31.6, 28.4, 24.3.

HRMS: calc. for $C_{17}H_{22}O$ (EI), 242.1665, found, 242.1663.

5.31 Synthesis of methyl (*E*)-4'-(hex-1-en-1-yl)-[1, 1'-biphenyl]-4-carboxylate (31)



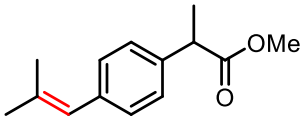
Following **Method A** (E = 2.40 V), the reaction of methyl 4'-hexyl-[1,1'-biphenyl]-4-carboxylate **S31** (c = 0.2 mmol/10 mL) in flow electrolytic cell afforded 37.1 mg (48 % yield) of **31** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 6.42 (d, *J* = 15.9 Hz, 1H), 6.33− 6.28 (m, 1H), 3.94 (s, 3H), 2.27− 2.22 (m, 2H), 1.51− 1.45 (m, 2H), 1.42−1.35 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 167.1, 145.3, 138.1, 138.0, 132.1, 130.1, 129.1, 128.7, 127.4, 126.7, 126.5, 52.1, 32.8, 31.5, 22.3, 14.0.

HRMS: calc. for $C_{20}H_{22}O_2$ (EI), 291.1614, found, 294.1612.

5.32 Synthesis of methyl 2-(4-(2-methylprop-1-en-1-yl) phenyl) propanoate (32)



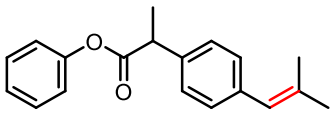
Following **Method A** (E = 2.30 V), the reaction of methyl 2-(4-isobutylphenyl) propanoate **S32** in flow electrolytic cell afforded 22.0 mg (41 % yield) of **32** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.3Hz, 2H), 7.18 (d, *J* = 8.2Hz, 2H), 6.23 (s, 1H), 3.71 (q, *J* = 7.2Hz, 1H), 3.66 (s, 3H), 1.90 (s, 3H), 1.86 (s, 3H), 1.50 (d, *J* = 7.2Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 175.1, 137.9, 137.6, 135.6, 129.0, 127.1, 124.7, 52.0, 45.1, 26.9, 19.4, 18.6.

HRMS: calc. for $C_{14}H_{18}O_2$ (EI), 218.1301, found, 218.1299.

5.33 Synthesis of phenyl 2-(4-(2-methylprop-1-en-1-yl) phenyl) propanoate (33)



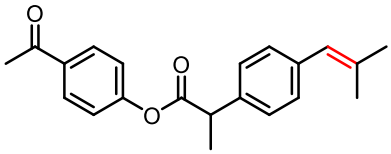
Following **Method A** (E = 2.37 V), the reaction of phenyl 2-(4-isobutylphenyl) propanoate **S33** afforded 25.5 mg (36 % yield) of **33** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.36– 7.32 (m, 4H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 7.5 Hz, 1H), 7.00 (d, *J* = 7.8 Hz, 2H), 6.26 (s, 1H), 3.95 (q, *J* = 7.2 Hz, 1H), 1.90 (d, *J* = 15.6 Hz, 6H), 1.62 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 173.2, 150.9, 137.8, 137.4, 135.8, 129.3, 129.1, 127.2, 125.8, 124.7, 121.4, 45.4, 27.0, 19.5, 18.5.

HRMS: calc. for $C_{19}H_{20}O_2$ (EI) 280.1458, found, 280.1459.

5.34 Synthesis of 4-acetylphenyl 2-(4-(2-methylprop-1-en-1-yl) phenyl) propanoa te (34)



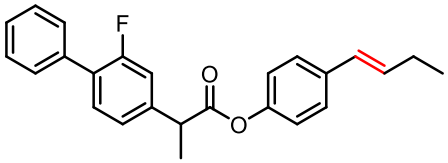
Following **Method A** (E = 2.42 V), the reaction of phenyl 2-(4-isobutylphenyl)-propanoate **S34** afforded 34.8 mg (42 % yield) of **34** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.8 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 6.26 (s, 1H), 3.96 (q, *J* = 7.2 Hz, 1H), 2.57 (s, 3H), 1.90 (d, *J* = 16.6 Hz, 6H), 1.62 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 196.9, 172.6, 1554.6, 138.0, 137.0, 136.0, 134.7, 129.9, 129.2, 127.2, 124.6, 121.7, 45.4, 27.0, 26.6, 19.5, 18.4.

HRMS: calc. for $C_{21}H_{22}O_3$ (EI) 322.1563, found, 322.1564.

5.35 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl 2-(2-fluoro- [1, 1'-biphenyl]-4-yl) propanoate (35)



Following **Method A** (E = 2.40 V), the reaction of 4-butylphenyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl) propanoate **S35** afforded 22.2 mg (21% yield, NMR, 44%) of **35** as a colorless oil.

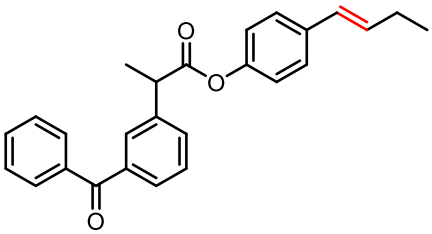
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58 (d, *J* = 1.5 Hz, 1H), 7.56 (s, 1H), 7.46 (td, *J* = 7.3 Hz, *J* = 2.2 Hz, 3H), 7.38 (tt, *J* = 8.6 Hz, *J* = 1.3 Hz, 1H), 7.33 (dt, *J* = 8.6 Hz, *J* = 2.7 Hz, 2H), 7.26 (dd, *J* = 7.8 Hz, *J* = 1.8 Hz, 1H), 7.24 (dd, *J* = 11.4 Hz, *J* = 1.6 Hz, 1H), 6.97 (dt, *J* = 8.7 Hz, *J* = 2.7 Hz, 2H), 6.35 (d, *J* = 15.9 Hz, 1H), 6.25-6.19 (m, 1H), 4.00 (q, *J* = 7.2 Hz, 1H), 2.26-2.20 (m, 2H), 1.66 (d, *J* = 7.2 Hz, 3H), 1.09 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 172.5, 159.8 (d, *J* = 246.9 Hz), 149.5, 141.3 (d, *J* = 7.9 Hz), 135.9, 135.5, 133.0, 131.0 (d, *J* = 3.8 Hz), 129.0, 129.0, 128.5, 128.1 (d, *J* = 13.6 Hz), 127.8, 127.8, 126.8, 123.6 (d, *J* = 3.0 Hz), 121.3, 115.4 (d, *J* = 23.5 Hz), 45.2, 26.0, 18.5, 13.6.

^{19}F NMR (471 MHz, $CDCl_3$): -117.3.

HRMS: calc. for $C_{25}H_{23}O_2F$ (EI) 374.1677, found, 374.1679.

5.36 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl 2-(3-benzoylphenyl) propanoate (36)



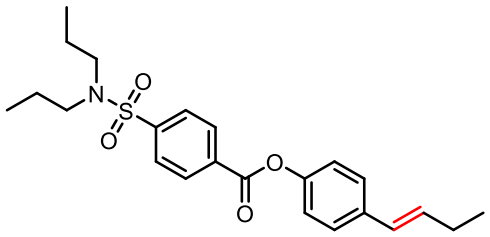
Following **Method A** (E = 2.45 V), the reaction of 4-butylphenyl 2-(3-benzoylphenyl)- propanoate **S36** afforded 47.1 mg (42% yield) of **36** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.86 (t, *J* = 1.5 Hz, 1H), 7.83 (s, 1H), 7.81 (d, *J* = 1.4 Hz, 1H), 7.73 (dt, *J* = 7.7, *J* = 1.3 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.31 (dt, *J* = 8.6, J = 2.6 Hz, 2H), 6.94 (dt, *J* = 8.6, J = 2.7 Hz, 2H), 6.34 (d, *J* = 15.9 Hz, 1H), 6.24-6.18 (m, 1H), 4.04 (q, *J* = 7.2 Hz, 1H), 2.25–2.19 (m, 2H), 1.66 (d, *J* = 7.2 Hz, 3H), 1.08 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 196.5, 172.6, 149.4, 140.4, 138.1, 137.5, 135.9, 133.0, 132.6, 131.5, 130.1, 129.3, 129.2, 128.8, 128.4, 127.8, 126.8, 121.3, 45.5, 26.0, 18.5, 13.6.

HRMS: calc. for $C_{26}H_{24}O_3$ (EI) 384.1720, found, 384.1719.

5.37 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl 4-(*N*, *N*-dipropylsulfamoyl) benzoate (37)



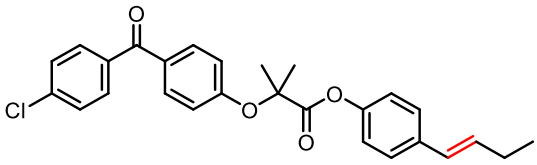
Following **Method A** (E = 2.80 V), the reaction of 4-butylphenyl 4-(*N*, *N*-dipropylsulfamoyl) benzoate **S37** (c = 0.2 mmol/10 mL) in flow electrolytic cell afforded 54.8 mg (55 % yield) of **37** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.31 (d, *J* = 8.4 Hz, 2H), 7.94 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.6 Hz, 2H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.26 (dt, *J* = 15.8, 6.4 Hz, 1H), 3.13 (t, *J* = 7.6 Hz, 4H), 2.25 (quint, *J* = 7.0 Hz, 2H), 1.56 (m, 4H), 1.10 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 163.9, 149.4, 144.9, 136.2, 133.3, 132.9, 130.8, 127.8, 127.2, 127.0, 121.5, 49.9, 26.1, 22.0, 13.6, 11.2.

HRMS: calc. for $C_{23}H_{29}O_4NS$ (EI), 415.1812, found, 415.1813.

5.38 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl 2-(4-(4-chlorobenzoyl) phenoxy)-2-methylpropanoate (38)



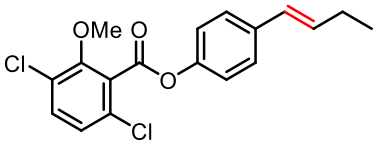
Following **Method A** (E = 2.70 V), the reaction of 4-butylphenyl 2-(4-(4-chlorobenzoyl) phenoxy)-2-methylpropanoate **S38** afforded 75.2 mg (54 % yield) of **38** as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$), δ 7.79 (d, *J* = 8.8 Hz, 2H), 7.72 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.6 Hz, 2H), 6.99 (d, *J* = 8.6 Hz, 2H), 6.91 (d, *J* = 8.6 Hz, 2H), 6.34 (d, *J* = 15.9 Hz, 1H), 6.22 (dt, *J* = 15.8, 6.4 Hz, 1H), 2.25–2.19 (m, 2H), 1.82 (s, 6H), 1.08 (d, *J* = 7.5Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 194.2, 172.5, 159.6, 149.1, 138.5, 136.4, 136.0, 133.3, 132.2, 131.2, 130.7, 128.6, 127.7, 126.9, 121.1, 117.4, 79.5, 26.0, 25.5, 13.6.

HRMS: calc. for $C_{27}H_{25}O_4Cl$ (EI) 448.1436, found, 448.1442.

5.39 Synthesis of (*E*)-4-(but-1-en-1-yl) phenyl 2, 4-dichloro-5-methoxybenzoate (39)



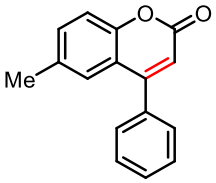
Following **Method A** (E = 2.50 V), the reaction of 4-butylphenyl 2,4-dichloro-5-methoxybenzoate **S39** afforded 68.5 mg (61% yield) of **39** as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43–7.40 (m, 3H), 7.21–7.18 (m, 3H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.26 (dt, *J* = 15.8, 6.4 Hz, 1H), 4.00 (s, 3H), 2.27–2.21 (m, 2H), 1.11 (d, *J* = 7.5Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 163.2, 154.1, 149.2, 136.5, 133.4, 132.3, 129.9, 129.8, 127.8, 127.0, 126.9, 126.0, 121.4, 62.4, 26.1, 13.6.

HRMS: calc. for $C_{18}H_{16}O_3Cl_2$ (EI) 350.0471, found, 350.0474.

5.40 Synthesis of 6-methyl-4-phenyl-2*H*-chromen-2-one (40)



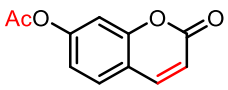
Following **Method A** (E = 2.50 V), the reaction of 6-methyl-4-phenylchroman-2-one **S40** in flow electrolytic cell afforded 16.1 mg (33% yield) of **40** as a white solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [24].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 –7.52(m, 3H), 7.47 –7.43 (m, 2H), 7.36 (dd, *J* = 2.6, *J* = 10.6 Hz, 1H), 7.30 (d, *J* = 10.5 Hz, 1H), 7.23 (d, *J* =2.5 Hz, 1H), 6.35 (s, 1H), 2.33 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 161.0, 155.6, 152.3, 135.4, 133.9, 132.9, 129.6, 128.8, 126.7, 118.7, 117.0, 115.2, 20.9.

5.41 Synthesis of 2-oxo-2*H*-chromen-7-yl acetate (41)



Following **Method A** (E = 2.80 V), the reaction of 7-hydroxychroman-2-one **S41** in flow electrolytic cell afforded 15.3 mg (33% yield) of **41** as a pink solid.

^{1}H NMR and ^{13}C NMR data match previously reported data [25].

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.69 (d, *J* = 9.6 Hz, 1H), 7.49 (d, *J* = 8.5 Hz, 1H), 7.12 (d, *J* = 2.1 Hz, 1H), 7.06 (dd, *J* = 8.4 Hz, 2.2 Hz, 1H), 6.40 (d, *J* = 9.6 Hz, 1H), 2.34 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 168.7, 160.3, 154.7, 153.2, 142.8, 128.6, 118.4, 116.7, 116.1, 110.5, 21.1.

6. Cyclic voltammetry studies

The cyclic voltammogram of some substrates were recorded at room temperature using a glassy carbon disk working electrode (diameter, 1 mm), a Pt wire auxiliary electrode and a SCE reference electrode. The scan rate was 100 mV/s. $E_{p/2}$ was measured. For purple line (**S2**+HOTf), CV was tested in [0.1 M] Et_4NBF_4 in CH_3CN:HOTf (5 mL/33 uL).

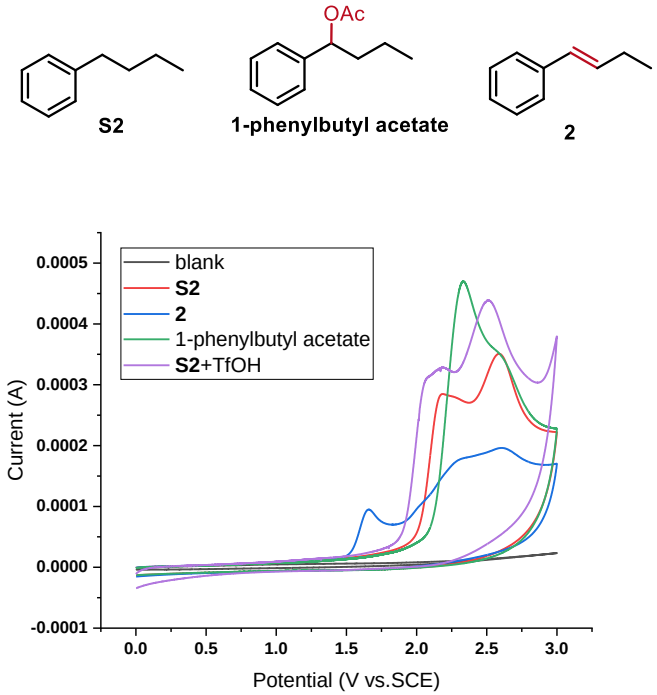
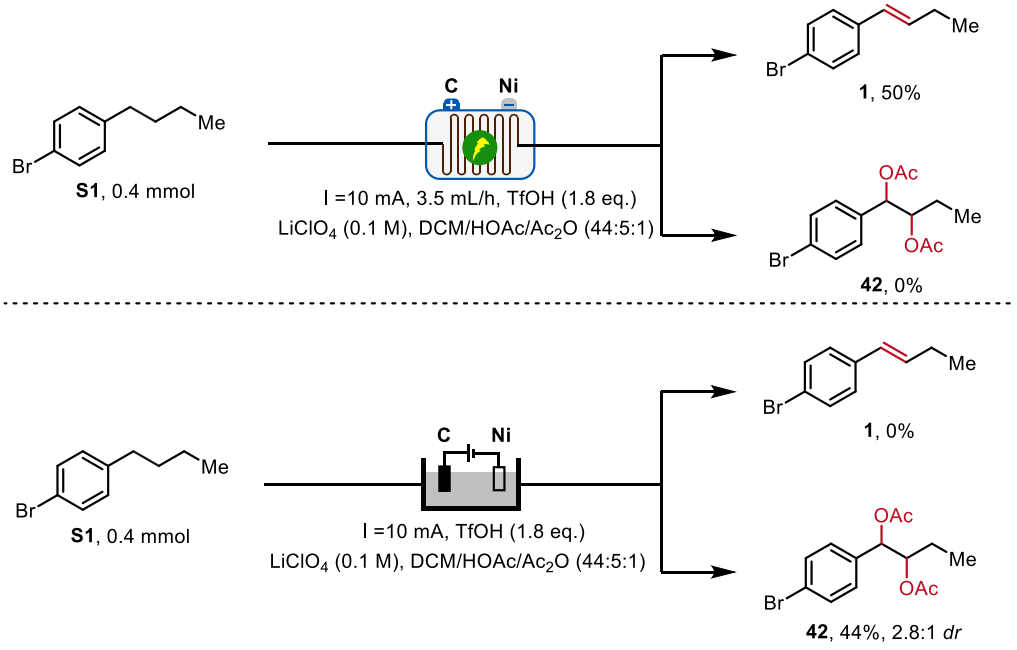


Figure S6 Cyclic voltammogram of substrates [3 mM] in [0.1 M] Et_4NBF_4 in CH_3CN.

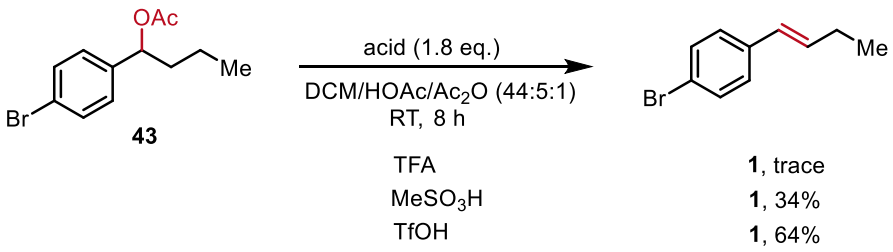
7. Comparison of reactions in batch and flow systems



Reaction in flow: see **Method A**. A solution (10 mL) of **S1** (0.4 mmol), $LiClO_4$ (0.1M), HOAc (1.0 mL) Ac_2O (0.2 mL), DCM (8.8 mL) and TfOH (66 μL, 1.8 equiv.) was mixed and pumped through the electrochemical reactor via a syringe pump (3.5 mL/h). The cell was operated at a constant current of 10 mA for 170 min. After the reaction reached the steady state, the solution is collected in a measuring cylinder. The formation of bubbles in the tube can be observed during the reaction (**Figure S2**). The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product. The reaction resulted in the formation of product **1** in 50% yield (42.2 mg). Dioxygenation product **42** was not observed.

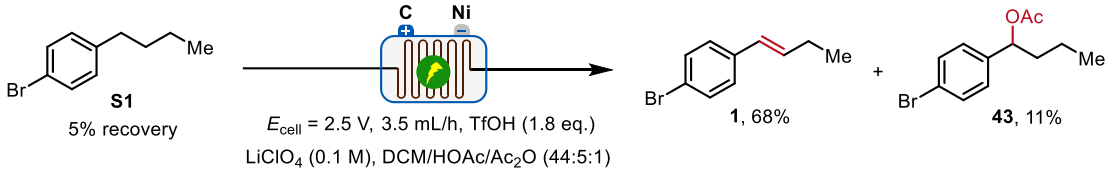
Reaction in batch: see **Method B**, **Figure S3**. An oven-dried undivided cell was equipped with a stir bar. To the cell was added **S1** (0.4 mmol), $LiClO_4$ (106.0 mg, 0.1M), HOAc (1.0 mL) Ac_2O (0.2 mL), DCM (8.8 mL) and TfOH (66 μL, 1.8 equiv.). The cell was sealed using a rubber septum and parafilm and was backfilled with N_2 atmosphere. The solution was then stirred at room temperature at a controlled current of 10 mA for 5 h. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. Following concentration in vacuo, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product. The reaction resulted in the formation of dioxygenation product **42** in 44% yield (42.2 mg, 2.8:1 *dr*)[26]. Product **1** was not observed.

8. Investigation the role of TfOH



Reaction conditions: A solution (10 mL) of **43** (108.0 mg, 0.4 mmol), $LiClO_4$ (0.1M), HOAc (1.0 mL) Ac_2O (0.2 mL), DCM (8.8 mL) and acid (1.8 equiv.) was mixed and stirred at room temperature for 8 h. The reaction mixture was subsequently poured into a saturated sodium carbonate solution (ca. 15 mL). The aqueous layer was separated and extracted with ethyl acetate or diethyl ether (3×5 mL), and the combined organic layers were washed with brine and dried over sodium sulfate. After concentration in vacuo, yields were determined by ^1H NMR analysis using tetrachloroethane as the internal standard.

9. Side reaction analysis of S1



We have studied the dioxygenation reaction of 4-bromobutylbenzene **S1** to determine the mass balance for a representative reaction. The reaction was run under the standard conditions, the reaction mixture was analyzed by GC-MS and crude NMR. The peak time of each compound was referenced with standard samples.

The conversion of substrates is usually above 90%. In cases where yields are moderate, the following factors may contribute to the diminished efficiency:

1) For low-boiling products, minor losses may occur during the post-reaction work-up and purification processes, such as in the rotary evaporation step.

2) The non-dehydrogenated benzyl acetyl ester intermediates products could sometimes be detected (5-10%). In some electron deficient substrates, benzylic acetoxylation products were much more prevalent.

3) In some cases, especially for some very electron-rich substrates, substrate decomposition was competitive.

10. Unsuccessful and challenging substrates

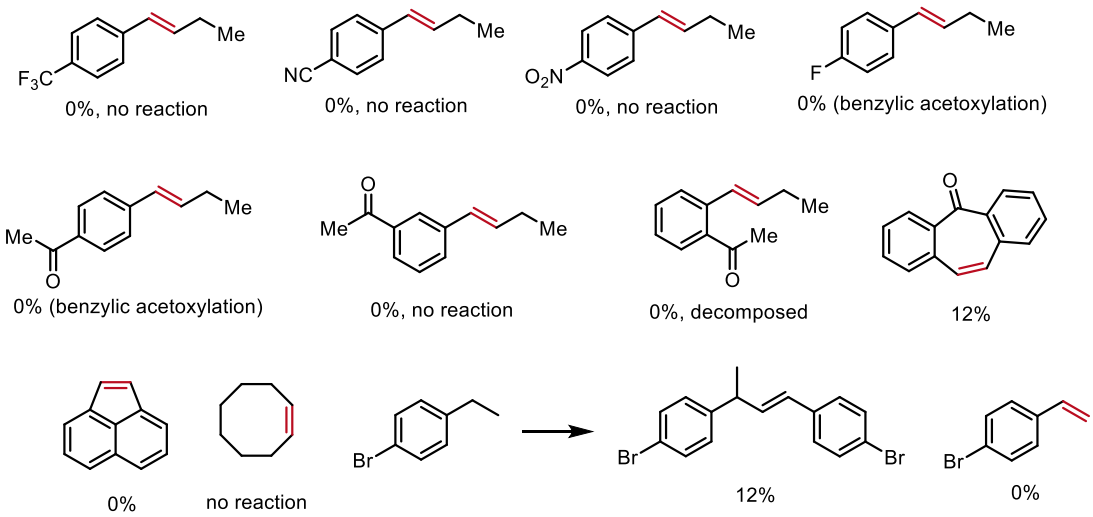


Figure S7. Unsuccessful and Challenging Substrates

11. Gas chromatography analysis of electrolysis headspace

We conducted hydrogen evolution monitoring using gas chromatography (GC) equipped with a thermal conductivity detector (TCD).

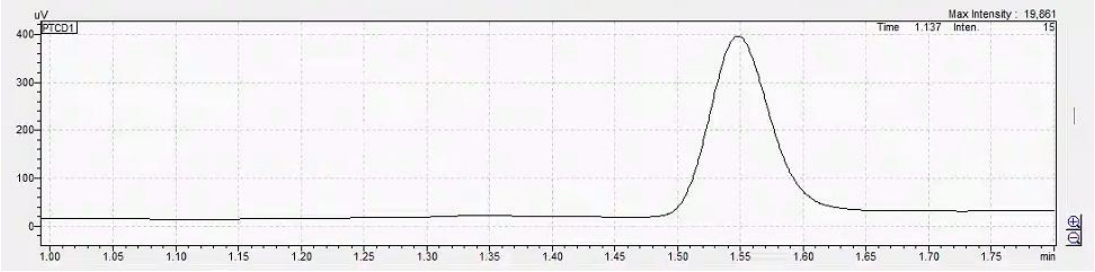
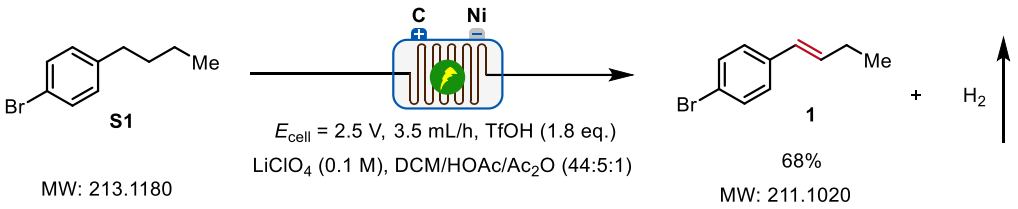


Figure S8. Detection of H_2.

12. Evaluation of green chemistry metrics and comparation with previous work

We tried to evaluate the green chemistry metrics such as atom-economy, atom-efficiency, reaction-mass efficiency, carbon-efficiency, E-factor, and compared with previous work. The results demonstrate that our method exhibits superior green metrics, including higher atom economy, atom efficiency, reaction mass efficiency, and carbon efficiency, as well as a lower E-factor compared to previously reported methods.

For example, our work: (**S1**: 0.4 mmol)



atom-economy: AE = 211/213*100%= 99.1%;

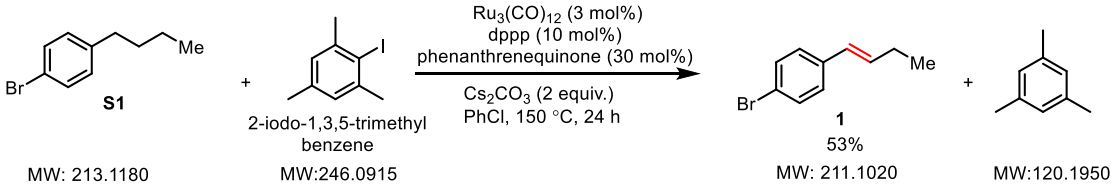
atom-efficiency: AEf = 211/ 213 *100% *68%= 67.4%;

reaction-mass efficiency: RME = 0.4*0.68*211/(0.4*213) *100% = 67.4%;

carbon-efficiency: CE = 0.4*0.68*10/(0.4*10) *100% = 68%;

E-factor: E = 2/211*100% = 0.95%.

Morandi's work: (**S1**: 7.5 mmol, 2-iodo-1,3,5-trimethyl benzene: 0.50 mmol)



atom-economy: AE = 211/(211+246)*100% = 46.2%;

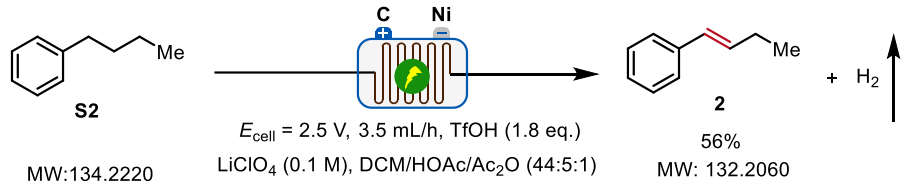
atom-efficiency: AEf = 211/ (211+120) *100% *53%= 24.5%;

reaction-mass efficiency: RME = 0.5*53%*211/(7.5*213+0.5*246) *100% = 3.2 %;

carbon-efficiency: CE = 0.5*0.53*10/(7.5*10+0.5*9) *100% = 3.3 %;

E-factor: E = 0.5*120/0.5*211*100% = 56.9 %。

For example: our work: (**S2**: 0.4 mmol)



atom-economy: AE = 132/134*100% =98.5%;

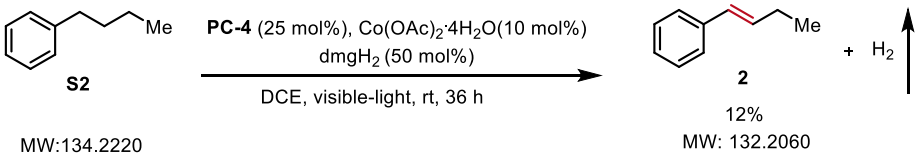
atom-efficiency: AEf = 132/ (132+2) *56% = 55.2%

reaction-mass efficiency: RME = 0.4*0.56*132/(0.4*134) *100% =55.2%

carbon-efficiency: CE = 0.4*0.56*10/(0.4*10) *100% = 56%

E-factor: E = 0.4*2/0.4*132*100% =1.5 %

Huang ’s work: (**S2**: 0.1 mmol):



atom-economy: AE = 132/134*100% = 98.5%;

atom-efficiency: AEf = 132/ (132+2) *12% = 11.8%;

reaction-mass efficiency: RME = 0.1*0.12*132/(0.1*134) *100% = 11.8 %;

carbon-efficiency: CE = 0.1*0.12*10/(0.1*10) *100% = 12 %;

E-factor: E = 0.1*2/0.1*132*100% = 1.5 %。

12. References

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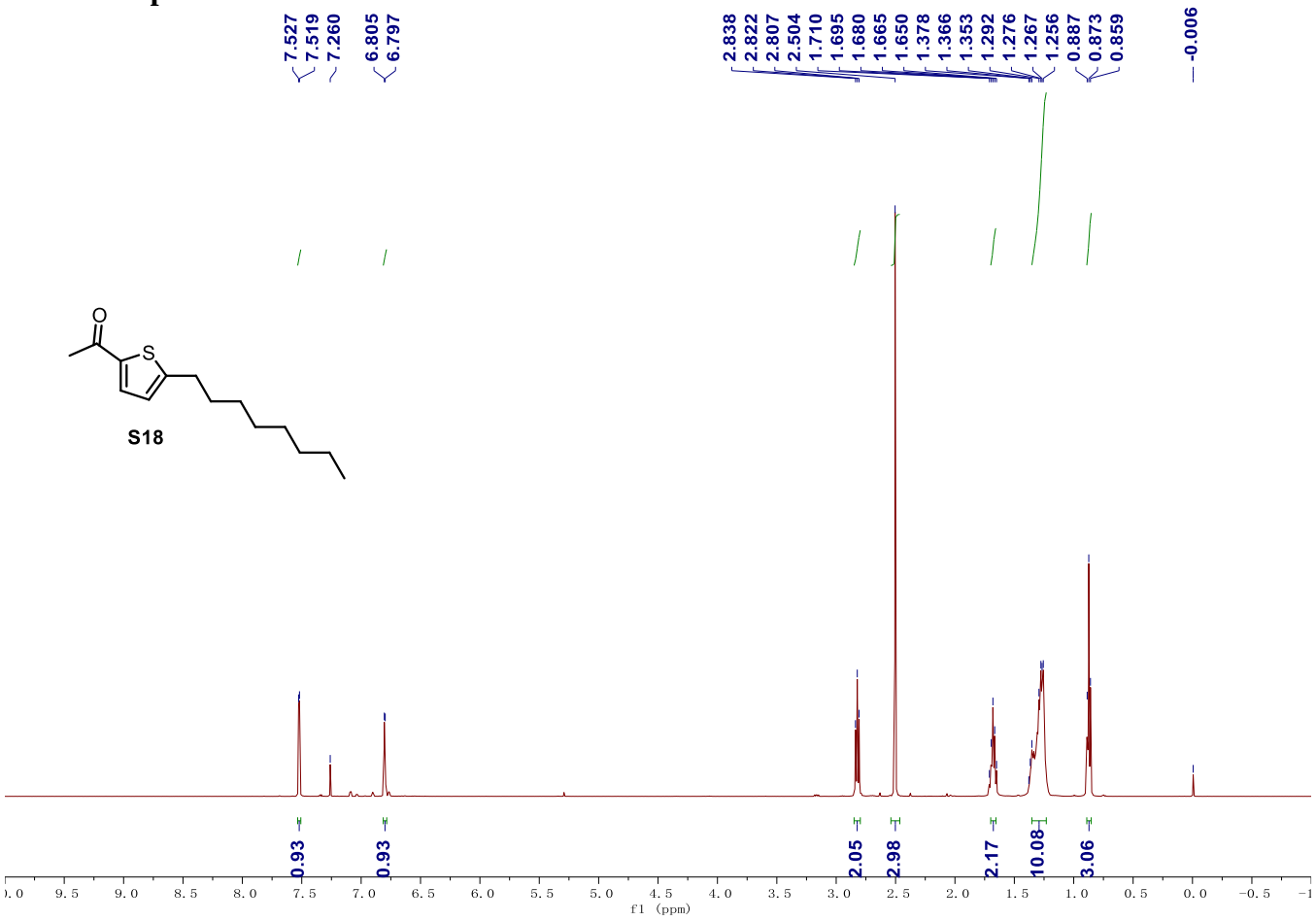
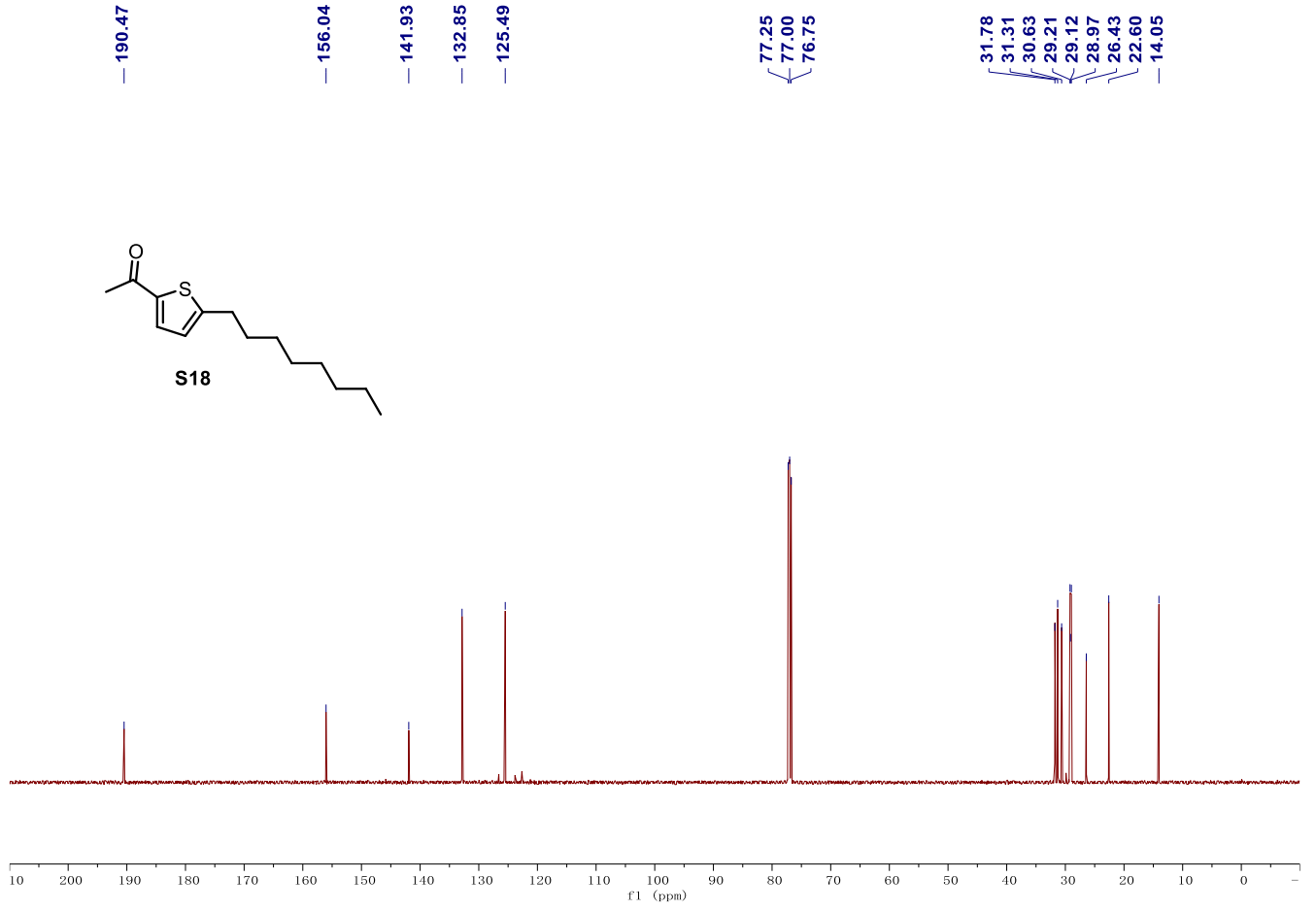
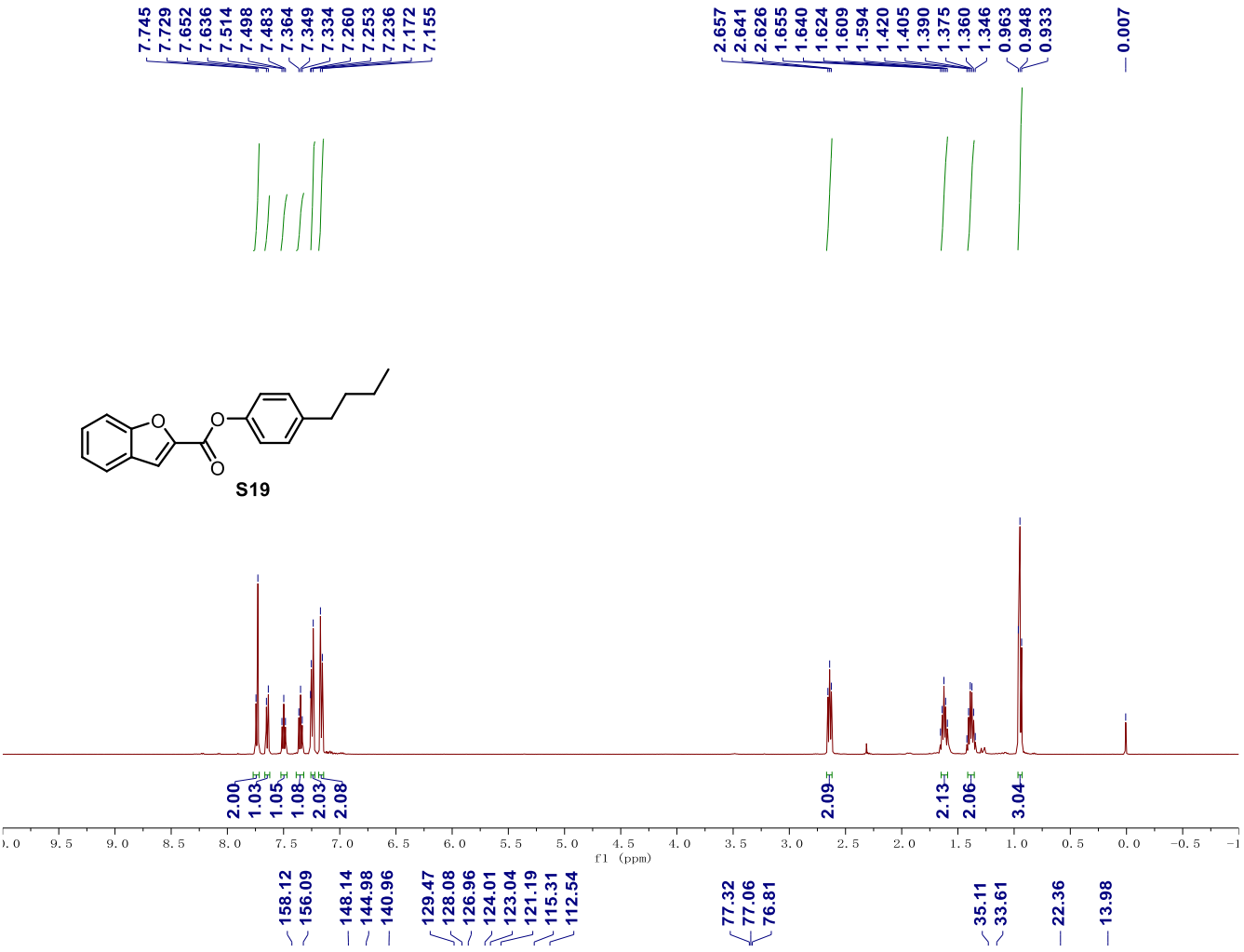
26. T. Shen, Y.-L. Li, K.-Y. Ye, T. H. Lambert, *Nature* **2023**, *614*, 275–280.

13. NMR Spectra



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7.498
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7.364
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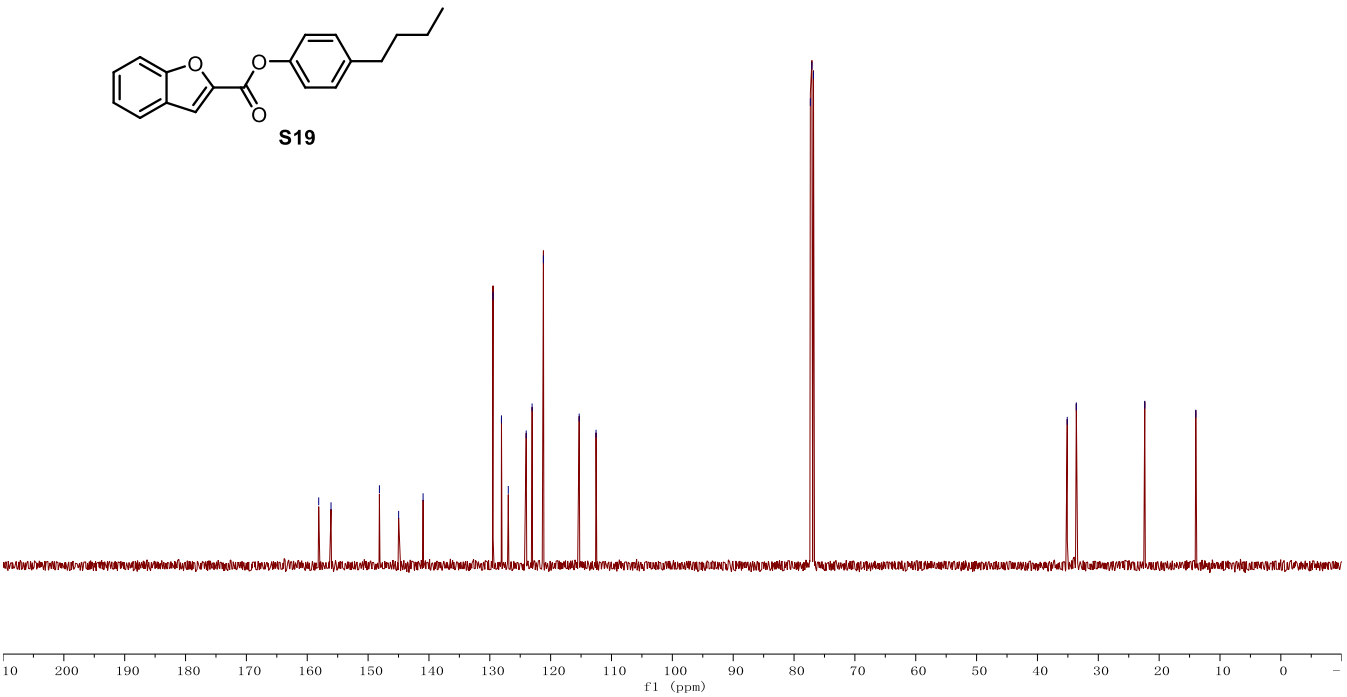
7.334
7.260
7.253
7.236
7.172
7.155
2.657
2.641
2.626
1.655
1.640
1.624
1.609
1.594
1.420
1.405
1.390
1.375
1.360
1.346
0.963
0.948
0.933
0.007
S19
2.00
1.03
1.05
1.08
2.03
2.08
2.09
2.13
2.06
3.04
f1 (ppm)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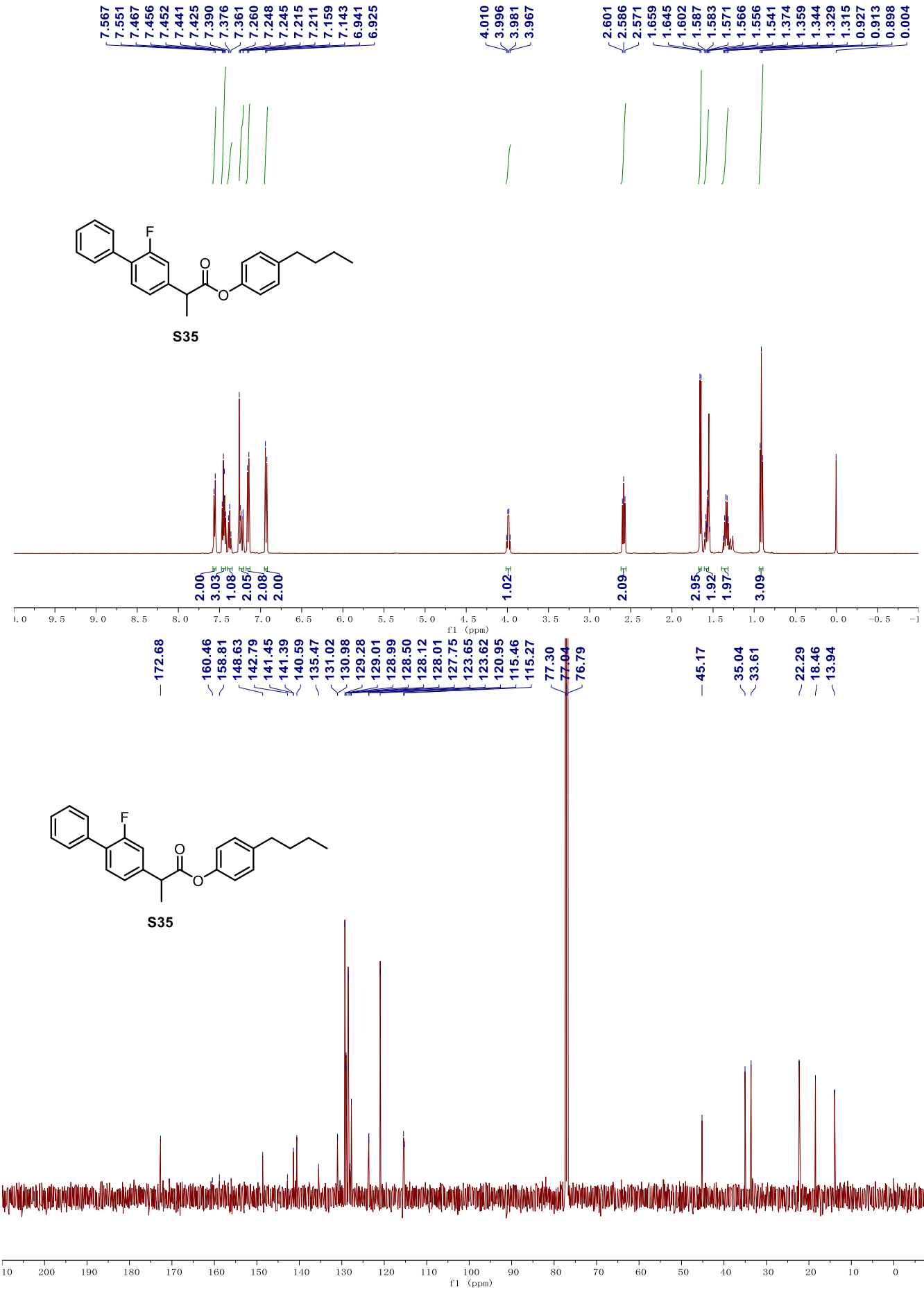
156.09
148.14
144.98
140.96
129.47
128.08
126.96
124.01
123.04
121.19
115.31
112.54
77.32
77.06
76.81
35.11
33.61
22.36
13.98
S19
f1 (ppm)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7.551
7.467
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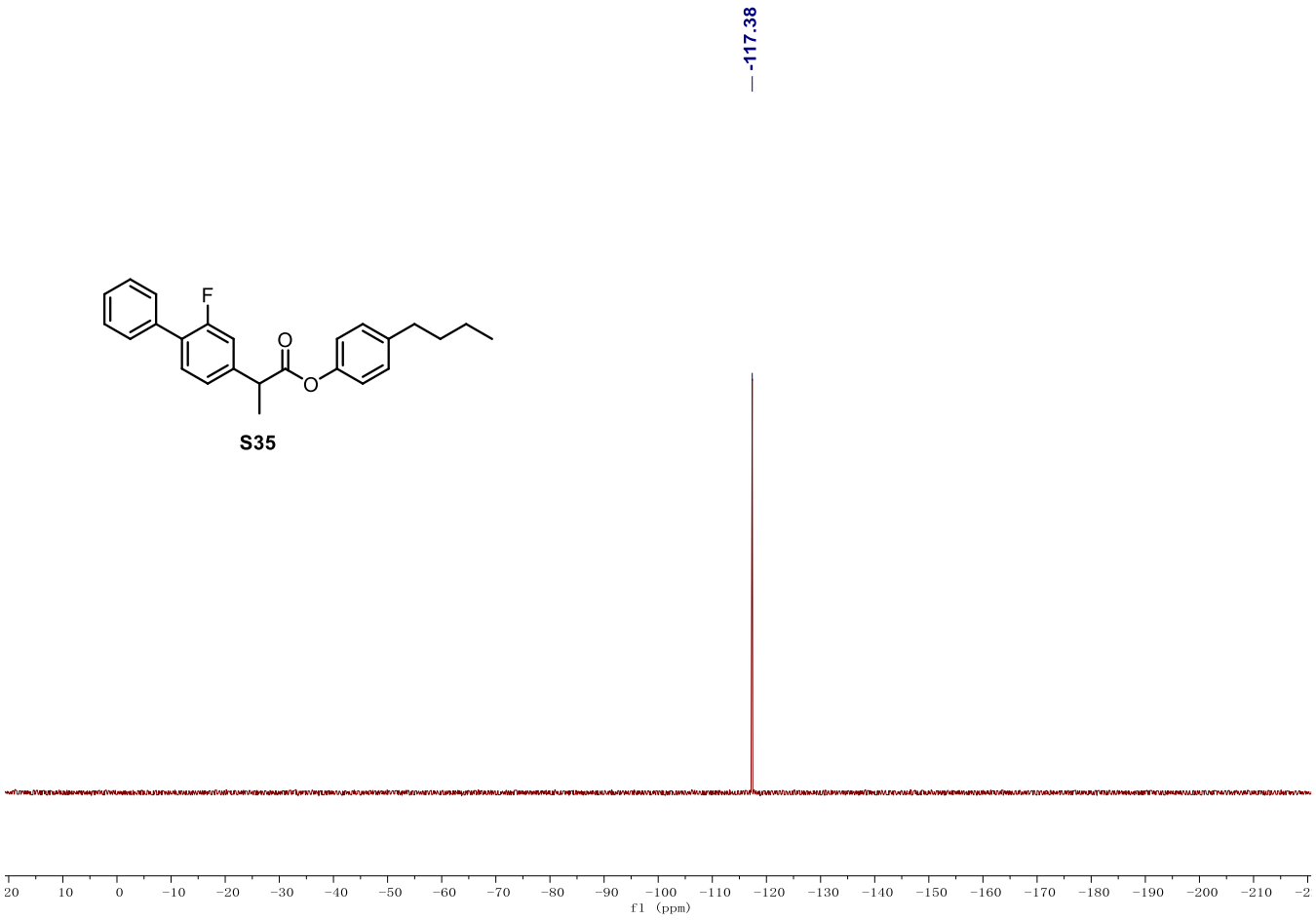
7.260
7.248
7.245
7.215
7.211
7.159
7.143
6.941
6.925
4.010
3.996
3.981
3.967
2.601
2.586
2.571
1.659
1.645
1.602
1.587
1.583
1.571
1.566
1.556
1.541
1.374
1.359
1.344
1.329
1.315
0.927
0.913
0.898
0.004
F
O
O
S35
2.00
3.03
1.08
2.05
2.08
2.00
1.02
2.09
2.95
1.92
1.97
3.09
f1 (ppm)
172.68
160.46
158.81
148.63
142.79
141.45
141.39
140.59
135.47
131.02
130.98
129.28
129.01
128.99
128.50
128.12
128.01
127.75
123.65
123.62
120.95
115.46
115.27
77.30
77.04
76.79
45.17
35.04
33.61
22.29
18.46
13.94
F
O
O
S35
f1 (ppm)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F
O
O
S35
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
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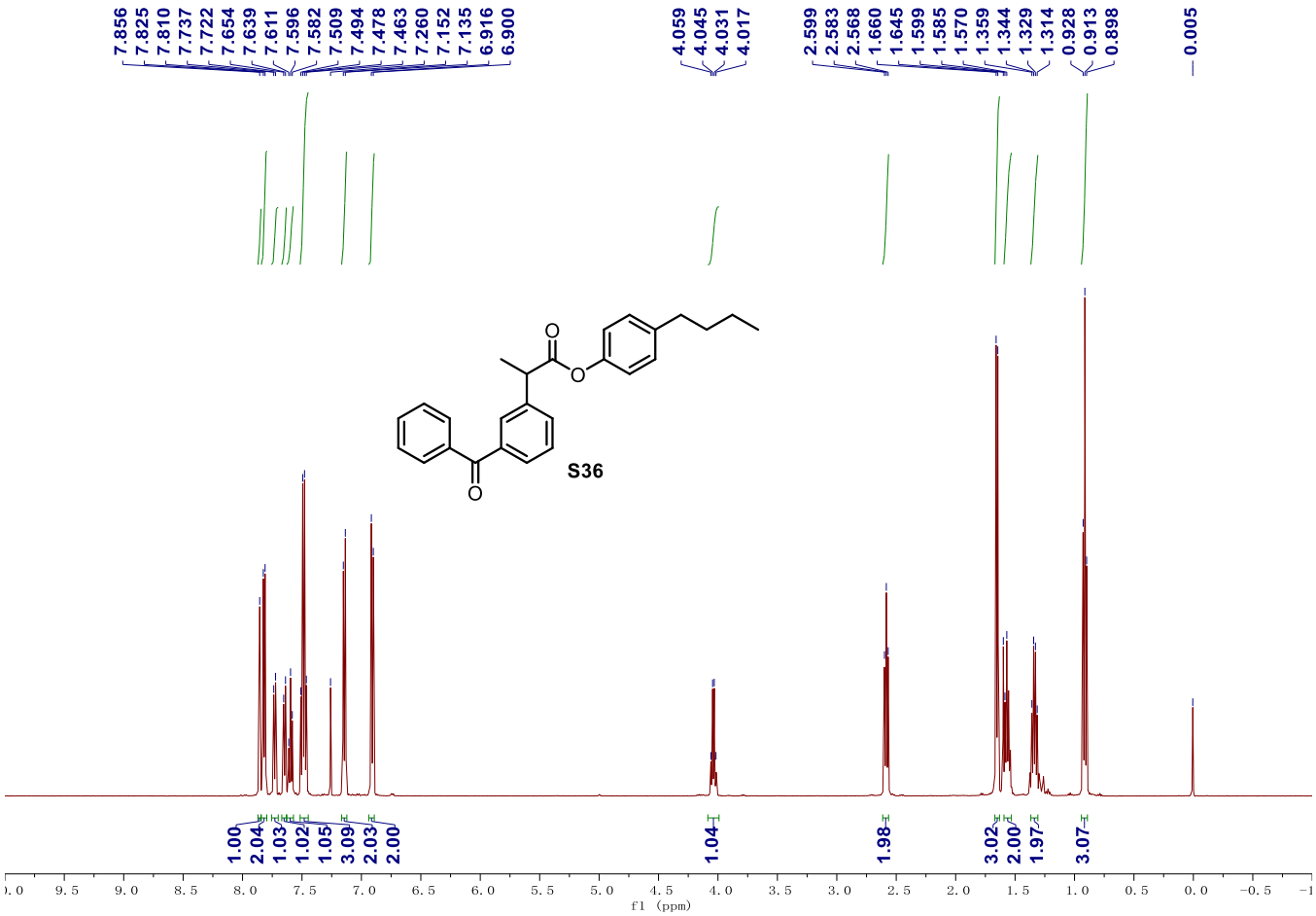
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)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7.654
7.639
7.611
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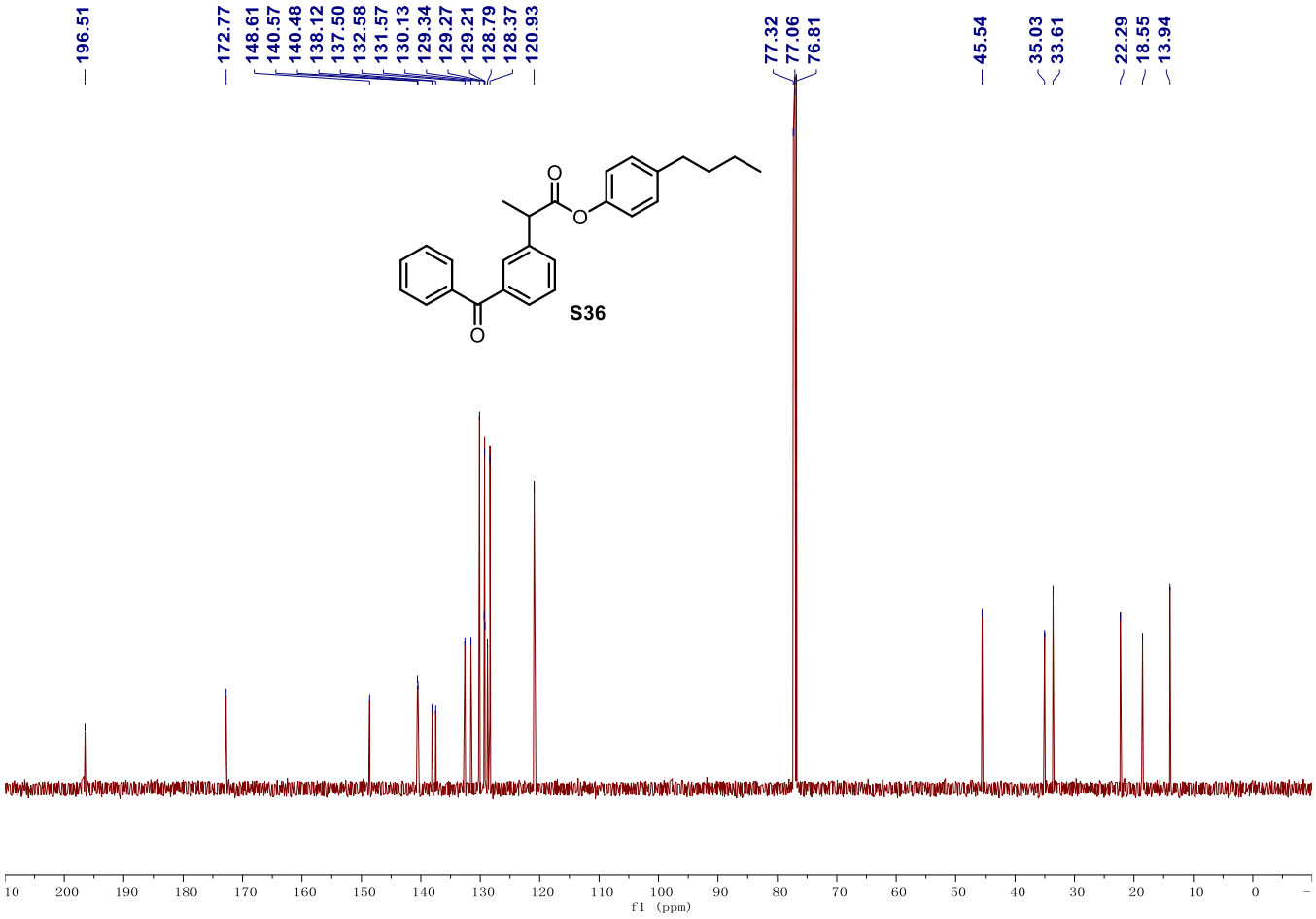
7.478
7.463
7.260
7.152
7.135
6.916
6.900
4.059
4.045
4.031
4.017
2.599
2.583
2.568
1.660
1.645
1.599
1.585
1.570
1.359
1.344
1.329
1.314
0.928
0.913
0.898
0.005
S36
1.00
2.04
1.03
1.02
1.05
3.09
2.03
2.00
1.04
1.98
3.02
2.00
1.97
3.07
f1 (ppm)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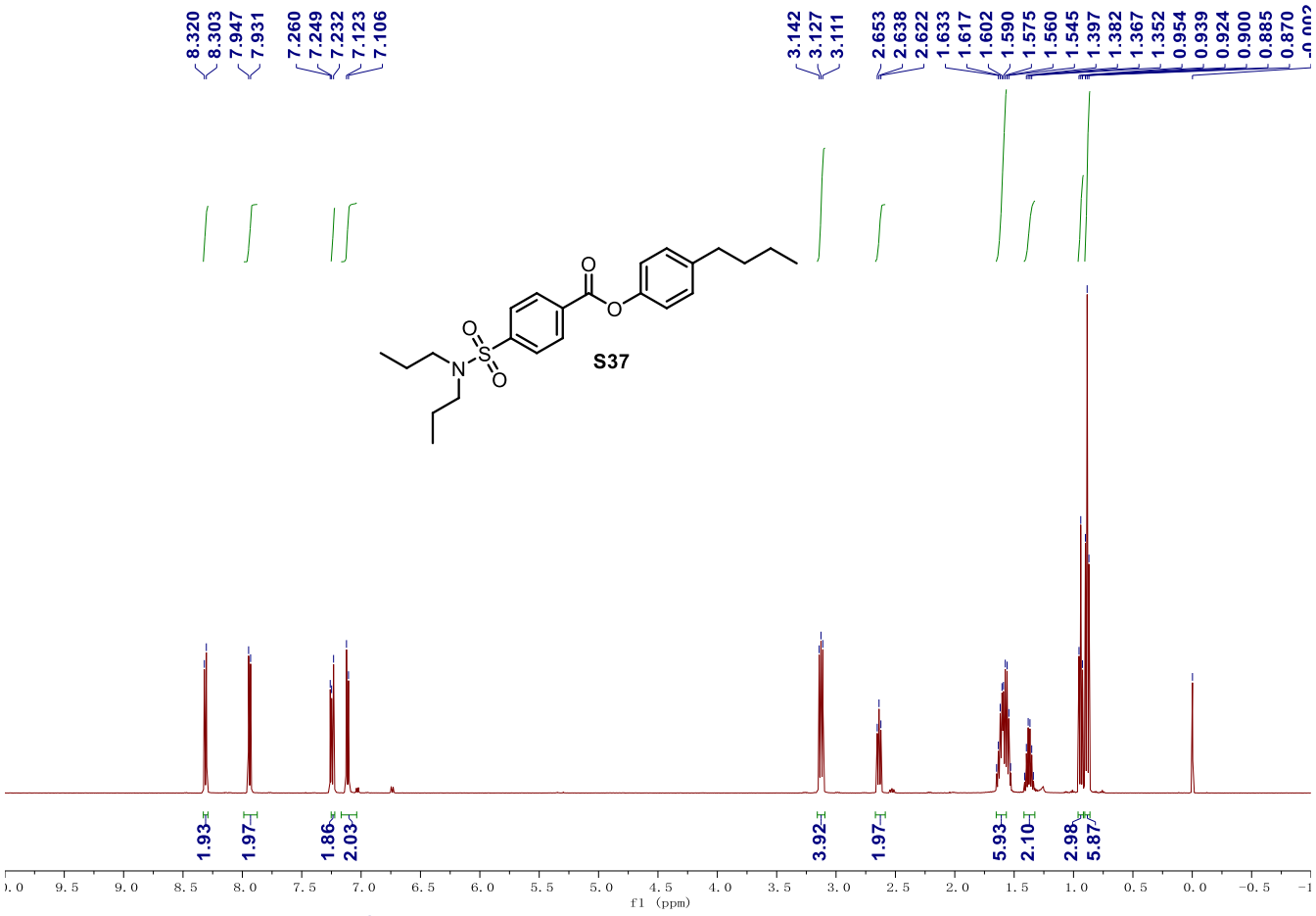
172.77
148.61
140.57
140.48
138.12
137.50
132.58
131.57
130.13
129.34
129.27
129.21
128.79
128.37
120.93
77.32
77.06
76.81
45.54
35.03
33.61
22.29
18.55
13.94
S36
f1 (ppm)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8.303
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7.260
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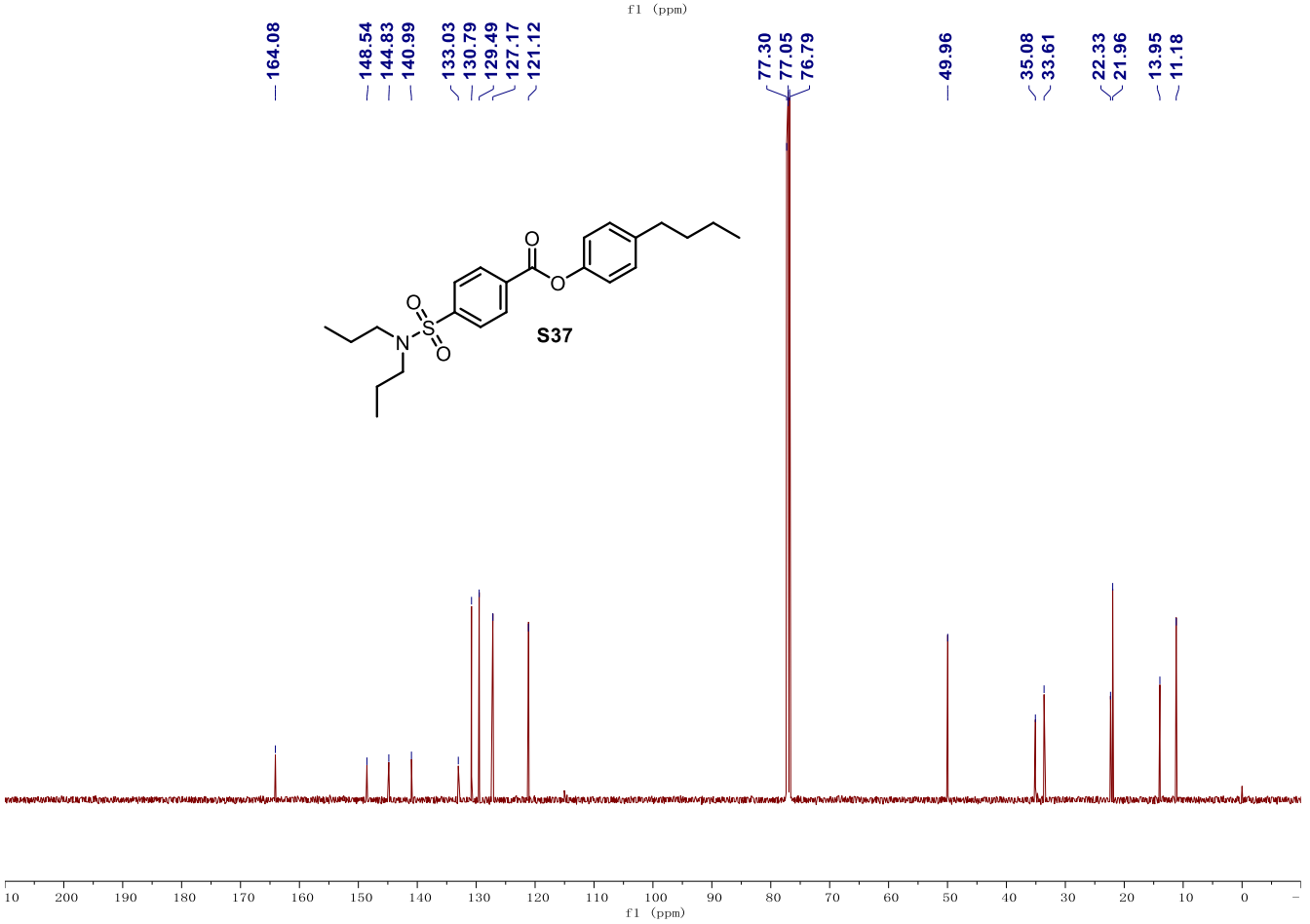
7.232
7.123
7.106
3.142
3.127
3.111
2.653
2.638
2.622
1.633
1.617
1.602
1.590
1.575
1.560
1.545
1.397
1.382
1.367
1.352
0.954
0.939
0.924
0.900
0.885
0.870
1.93
1.97
1.86
2.03
3.92
1.97
5.93
2.10
2.98
5.87
f1 (ppm)
S37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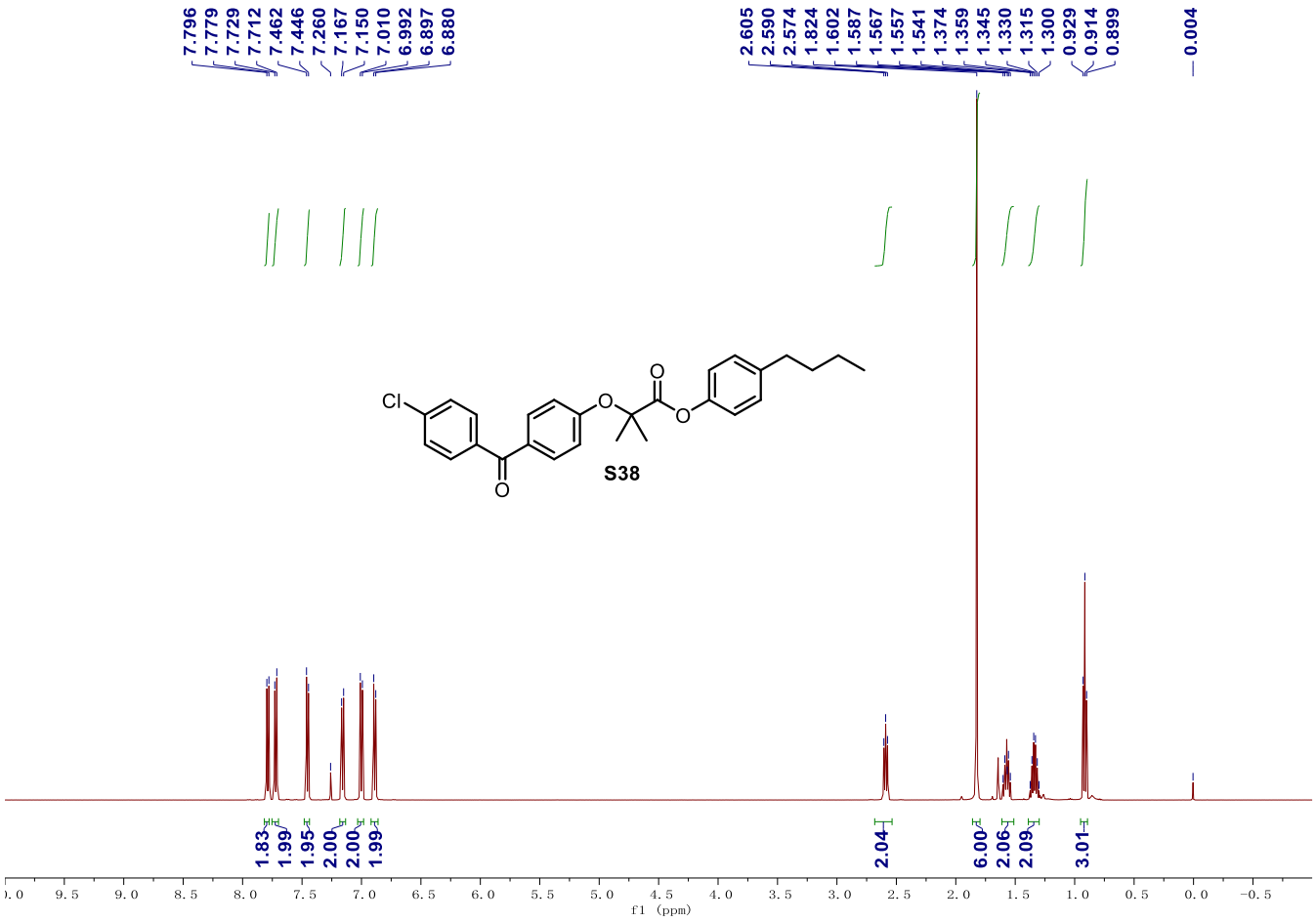
49.96
35.08
33.61
22.33
21.96
13.95
11.18
f1 (ppm)
S37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7.010
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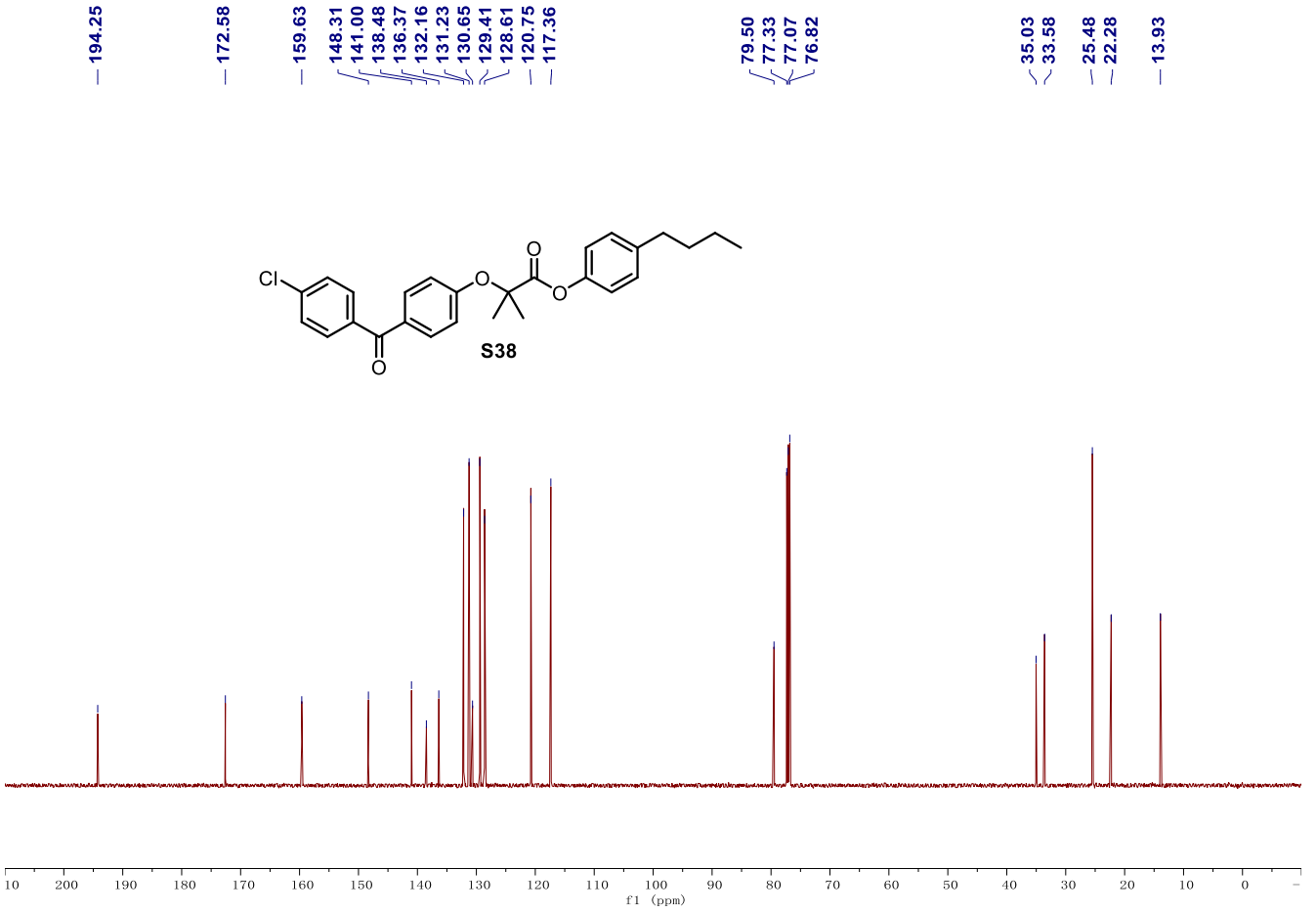
6.897
6.880
2.605
2.590
2.574
1.824
1.602
1.587
1.567
1.557
1.541
1.374
1.359
1.345
1.330
1.315
1.300
0.929
0.914
0.899
0.004
Cl
O
O
O
O
S38
1.83
1.99
1.95
2.00
2.00
1.99
2.04
6.00
2.06
2.09
3.01
f1 (ppm)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132.16
131.23
130.65
129.41
128.61
120.75
117.36
79.50
77.33
77.07
76.82
35.03
33.58
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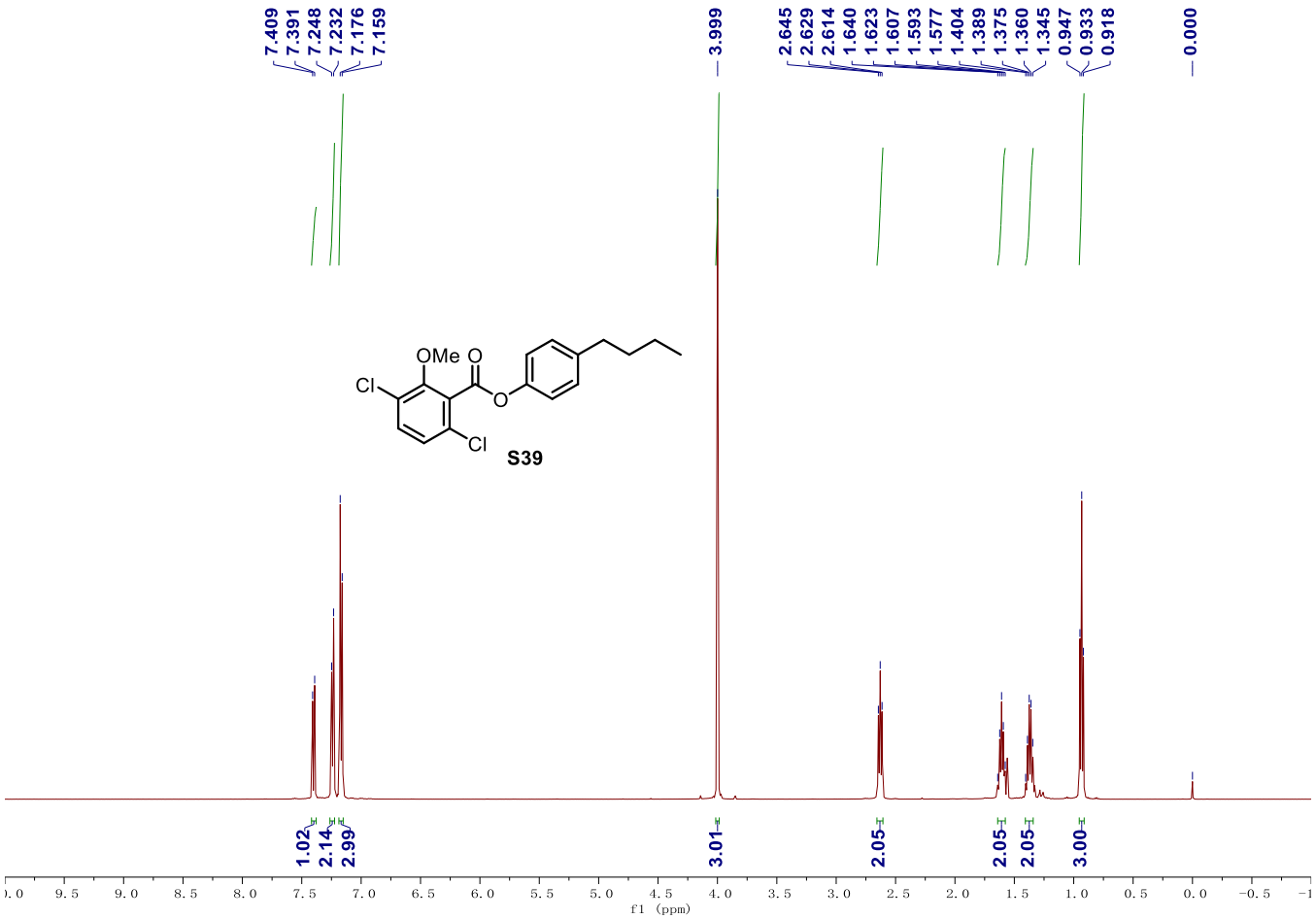
22.28
13.93
Cl
O
O
O
O
S38
f1 (ppm)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1.623
1.607
1.593
1.577
1.404
1.389
1.375
1.360
1.345
0.947
0.933
0.918
0.000
OMe
O
Cl
O
Cl
S39
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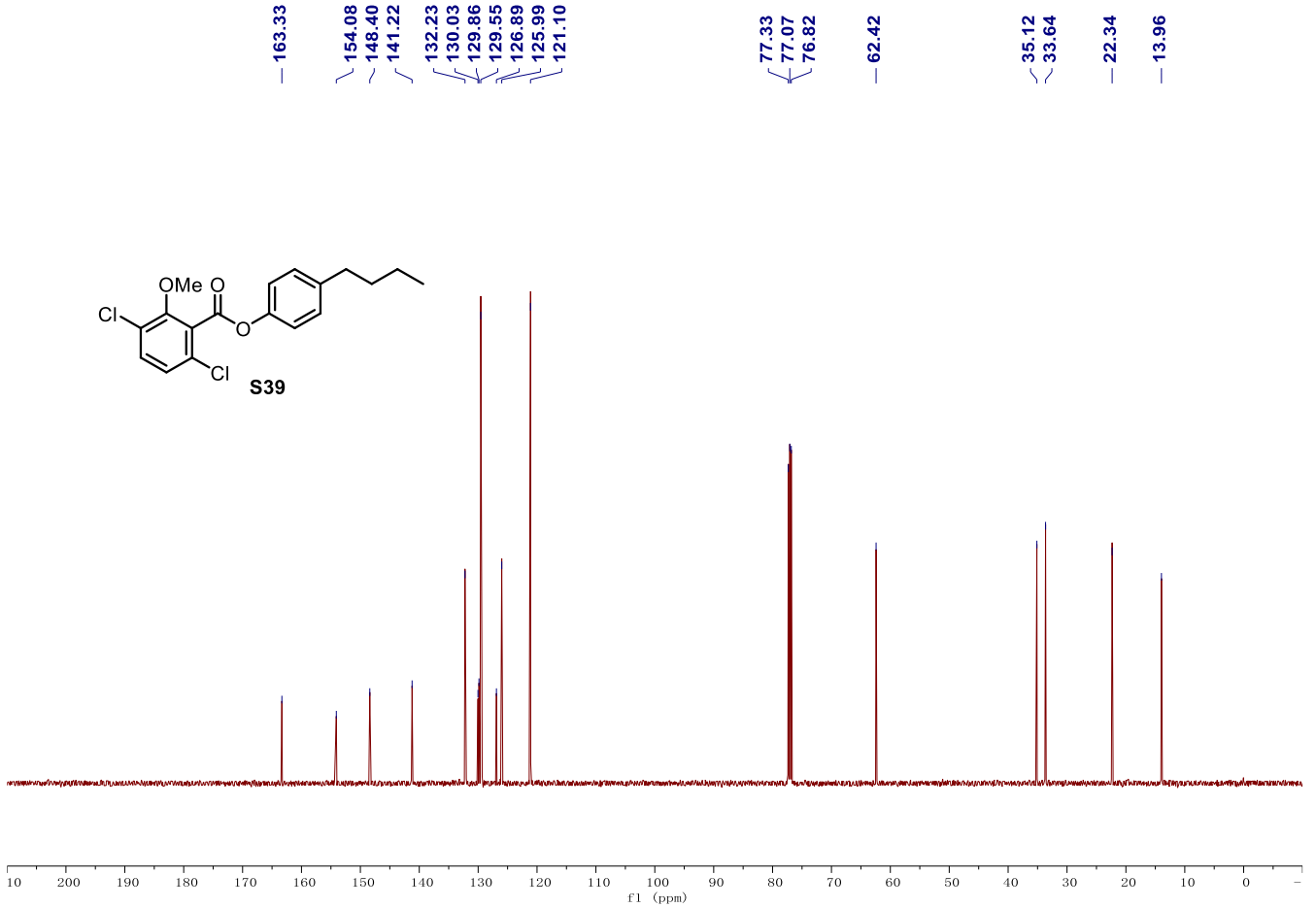
2.14
2.99
3.01
2.05
2.05
2.05
3.00
f1 (ppm)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35.12
33.64
22.34
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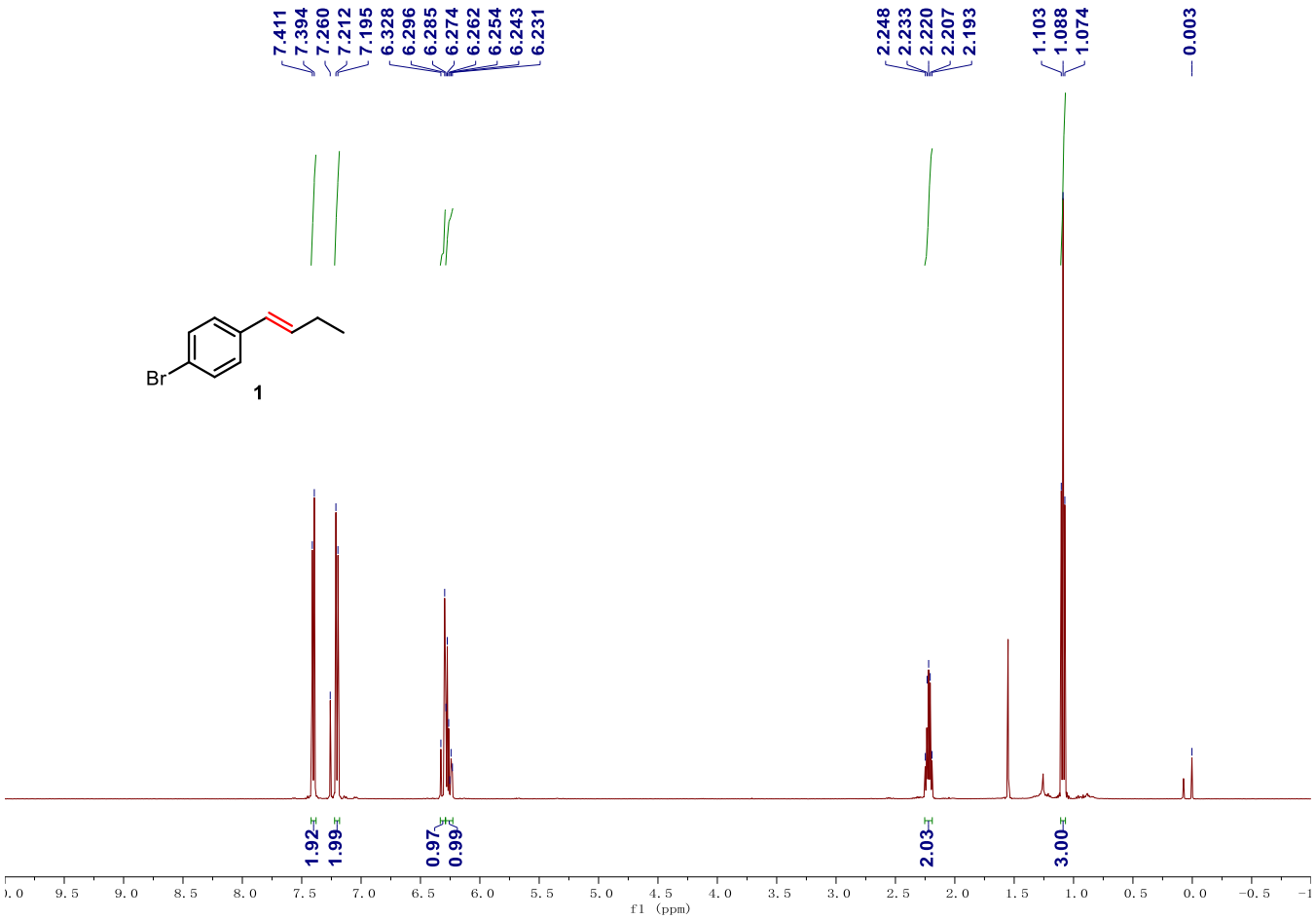
OMe
O
Cl
O
Cl
S39
f1 (ppm)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6.262
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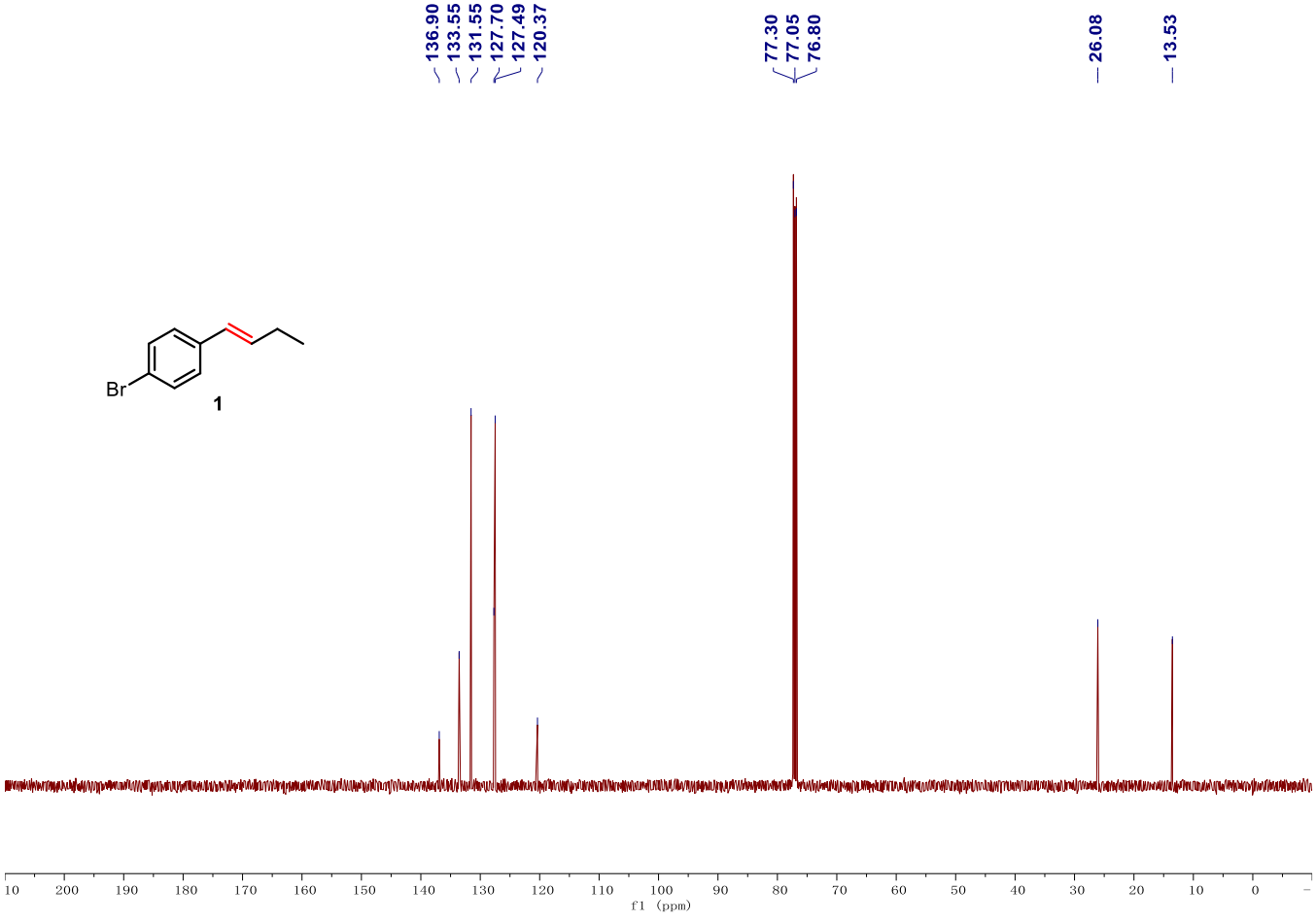
2.233
2.220
2.207
2.193
1.103
1.088
1.074
0.003
Br
1
1.92
1.99
0.97
0.99
2.03
3.00
f1 (ppm)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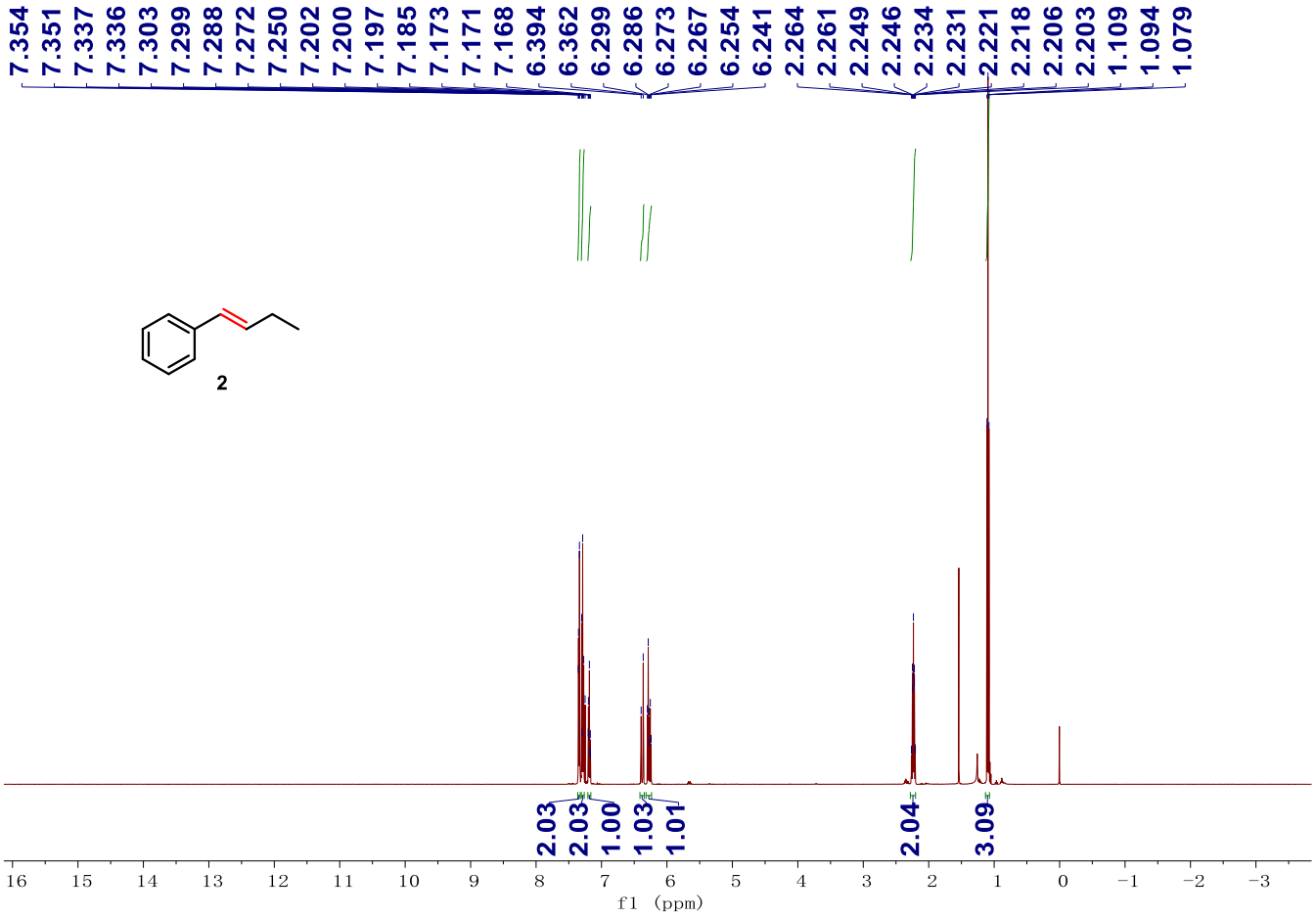
127.49
120.37
77.30
77.05
76.80
26.08
13.53
Br
1
f1 (ppm)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7.299
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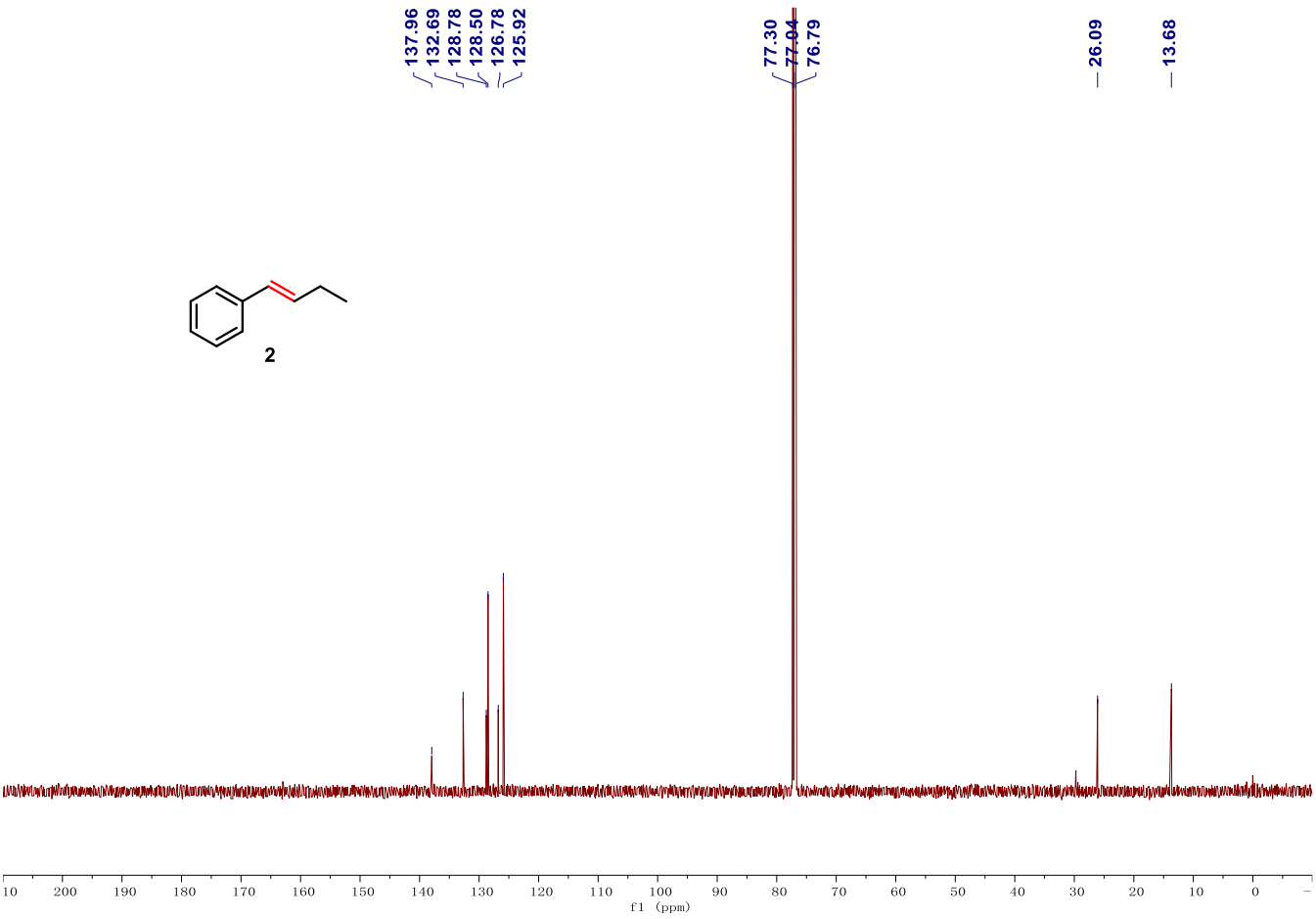
7.272
7.250
7.202
7.200
7.197
7.185
7.173
7.171
7.168
6.394
6.362
6.299
6.286
6.273
6.267
6.254
6.241
2.264
2.261
2.249
2.246
2.234
2.231
2.221
2.218
2.206
2.203
1.109
1.094
1.079
2
2.03
2.03
1.00
1.03
1.01
2.04
3.09
f1 (ppm)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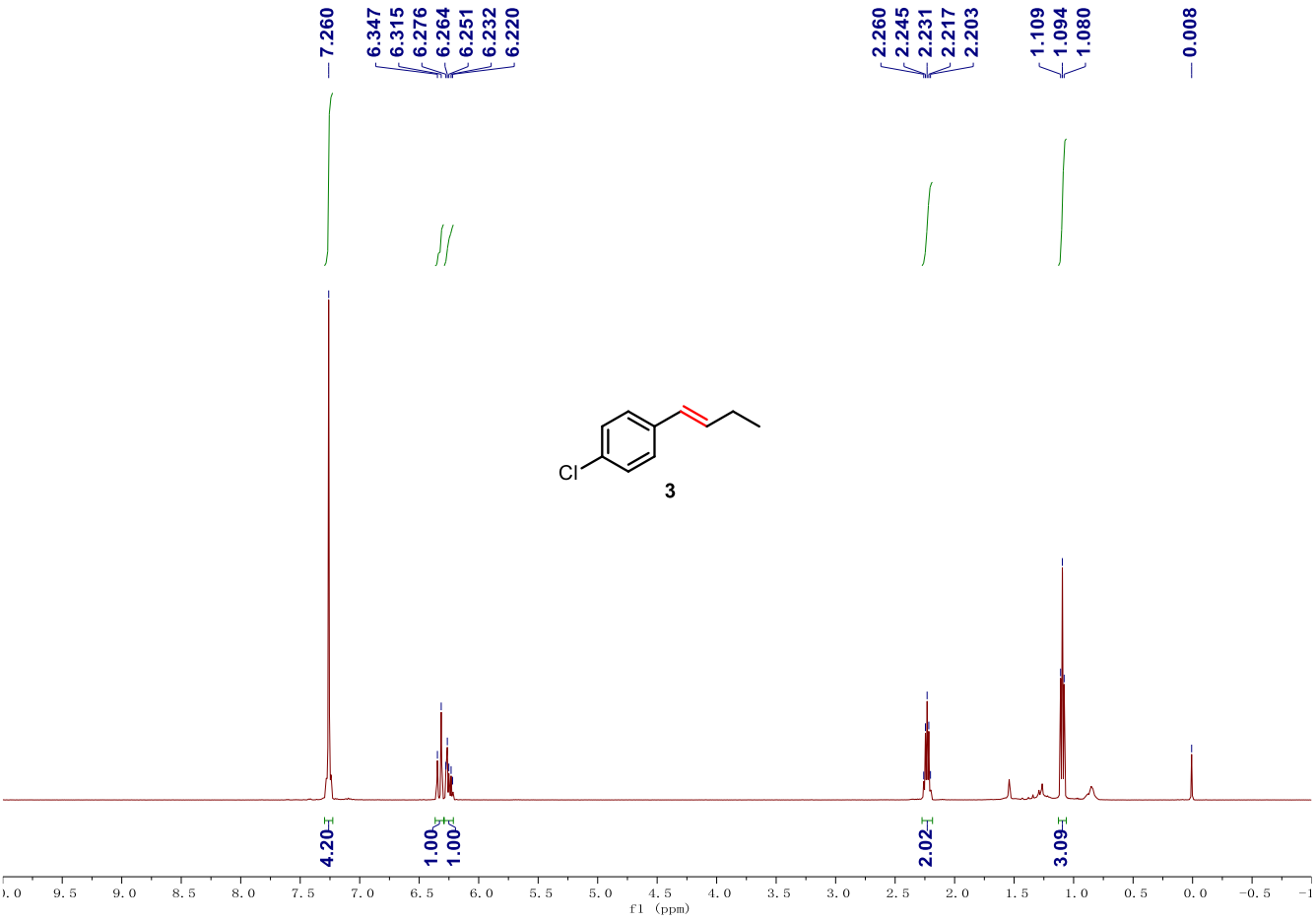
76.79
26.09
13.68
2
f1 (ppm)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2.245
2.231
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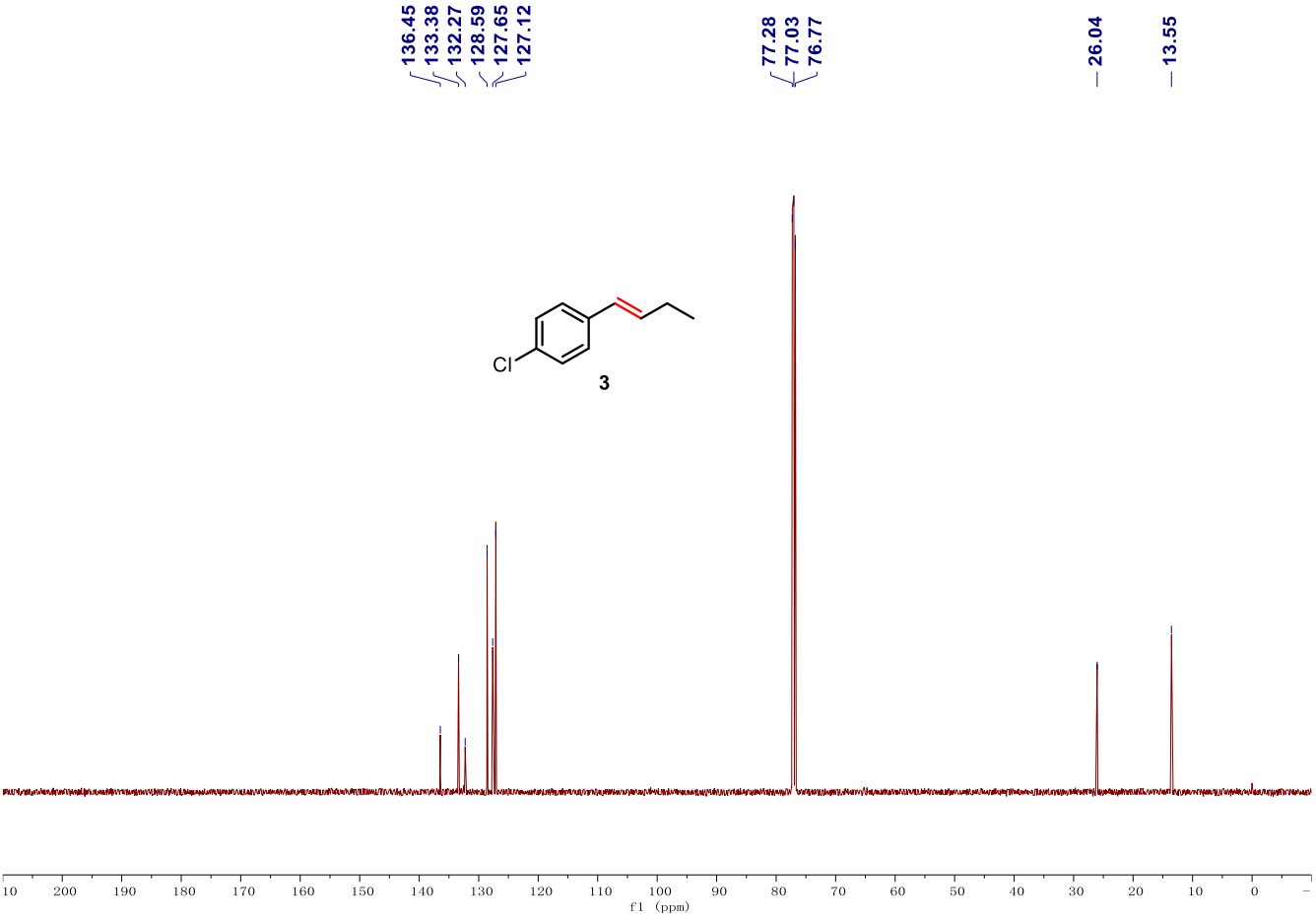
2.203
1.109
1.094
1.080
0.008
Cl
3
4.20
1.00
1.00
2.02
3.09
f1 (ppm)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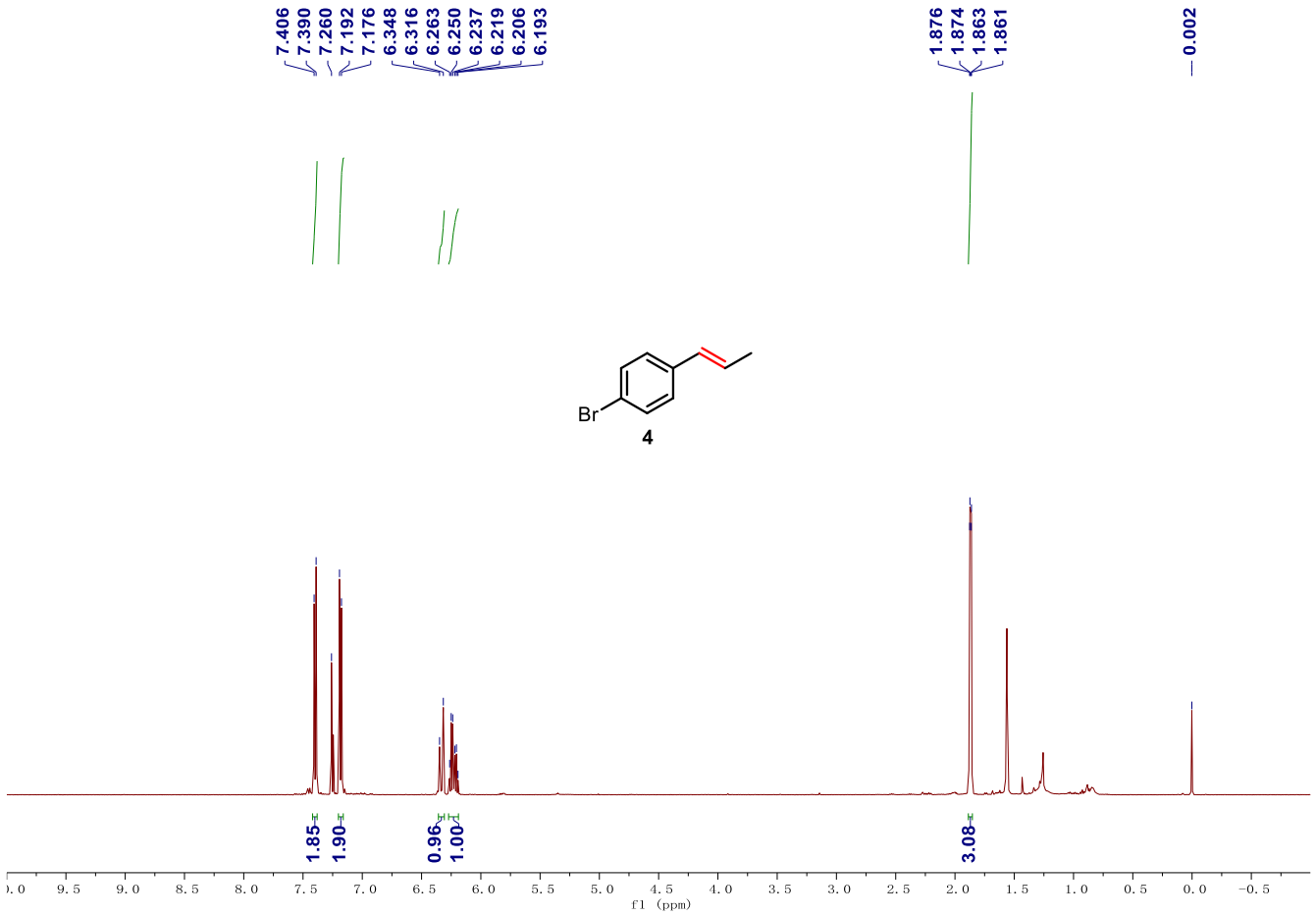
77.03
76.77
26.04
13.55
Cl
3
f1 (ppm)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7.390
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6.316
6.263
6.250
6.237
6.219
6.206
6.193
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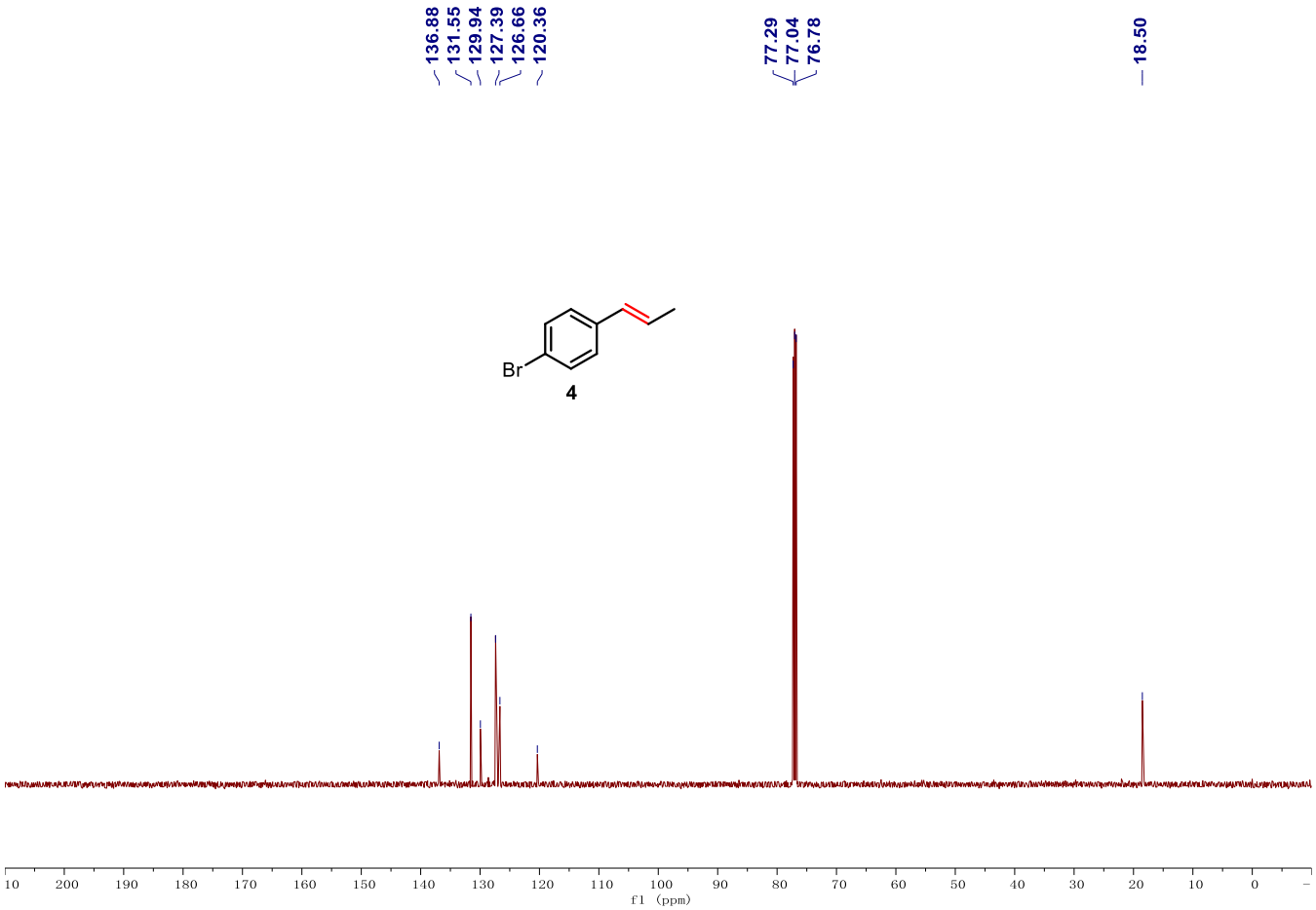
1.861
0.002
Br
4
1.85
1.90
0.96
1.00
3.08
f1 (ppm)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77.04
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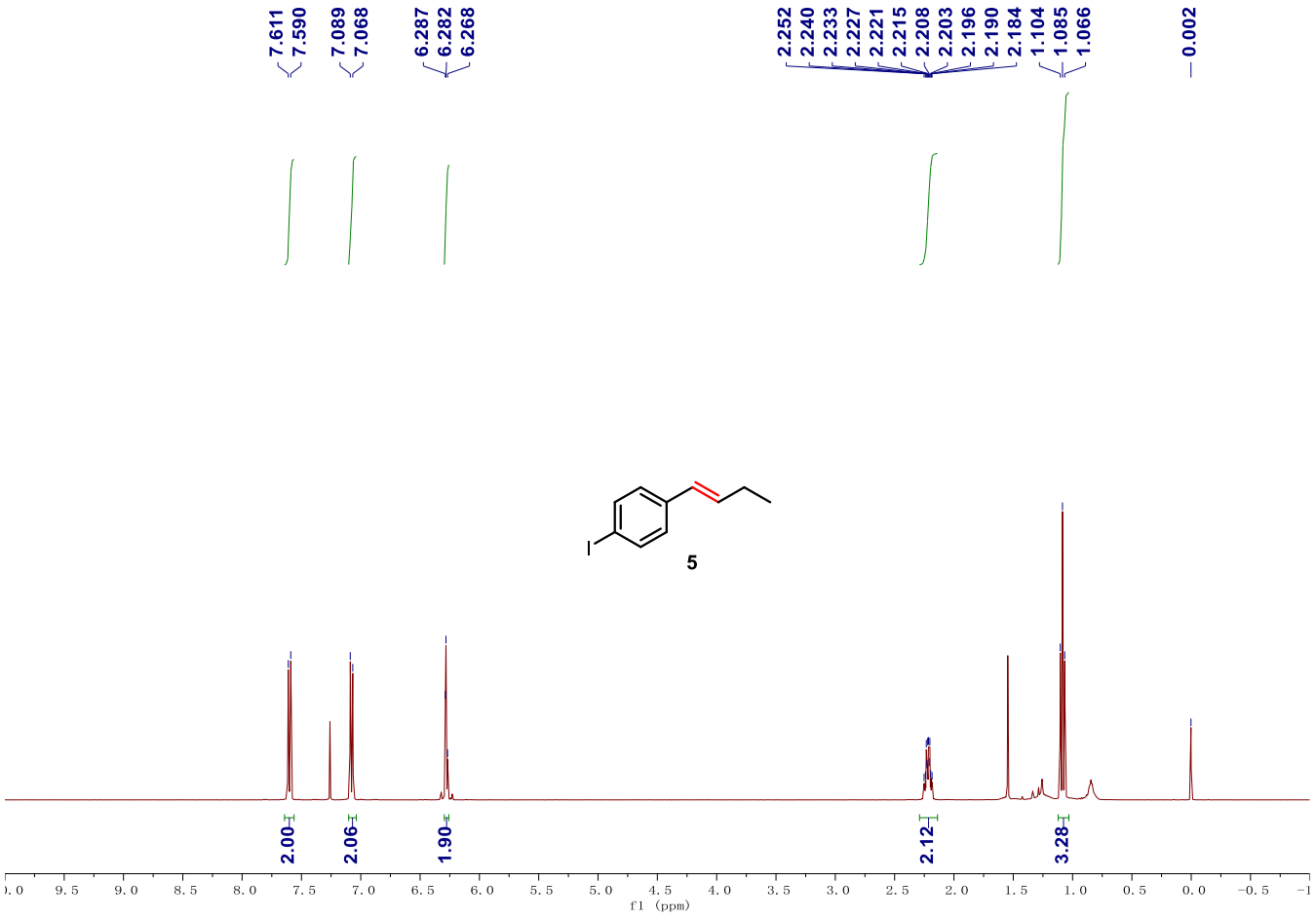
Br
4
f1 (ppm)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6.268
2.252
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2.233
2.227
2.221
2.215
2.208
2.203
2.196
2.190
2.184
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1.085
1.066
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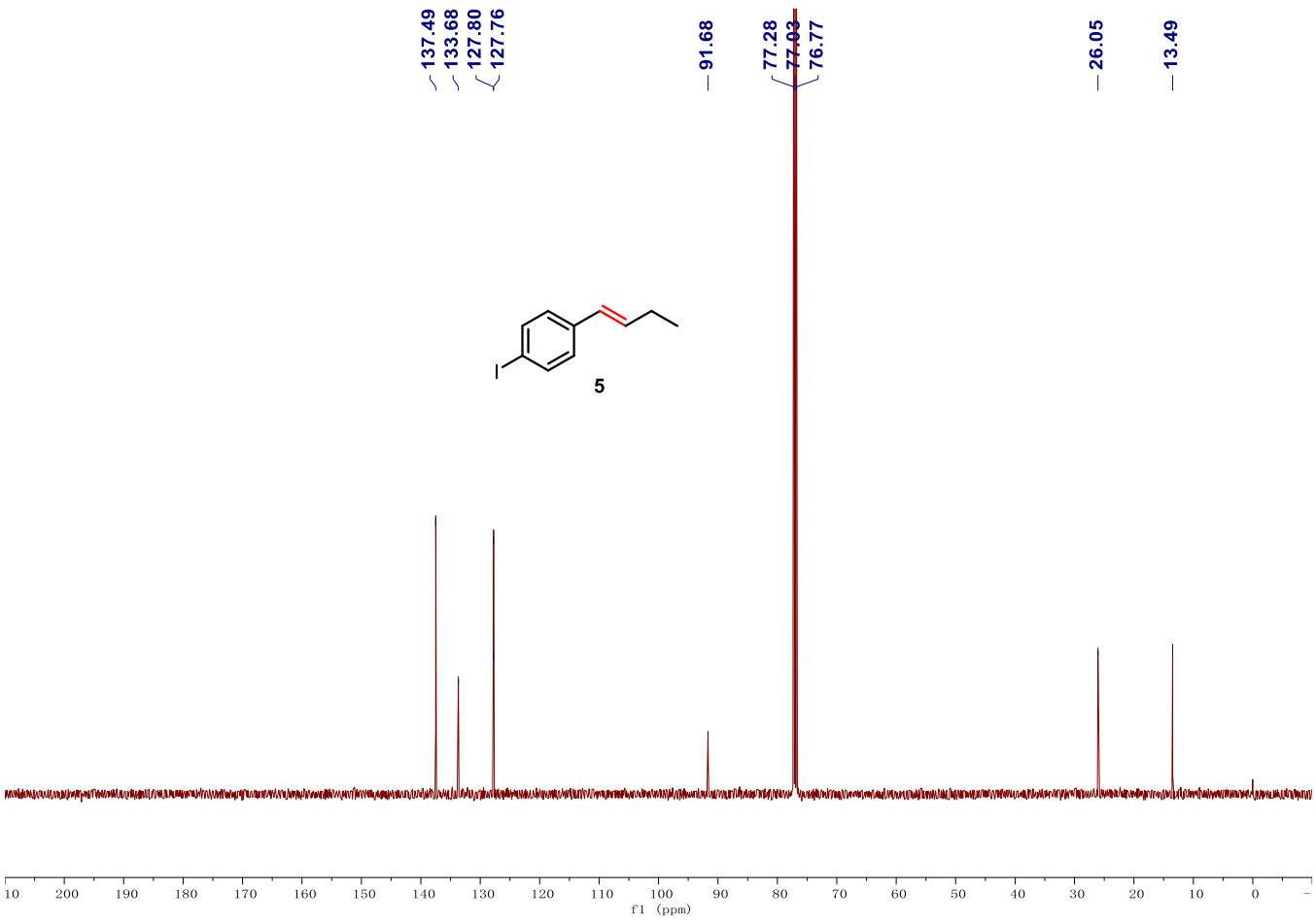
2.00
2.06
1.90
2.12
3.28
f1 (ppm)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77.03
76.77
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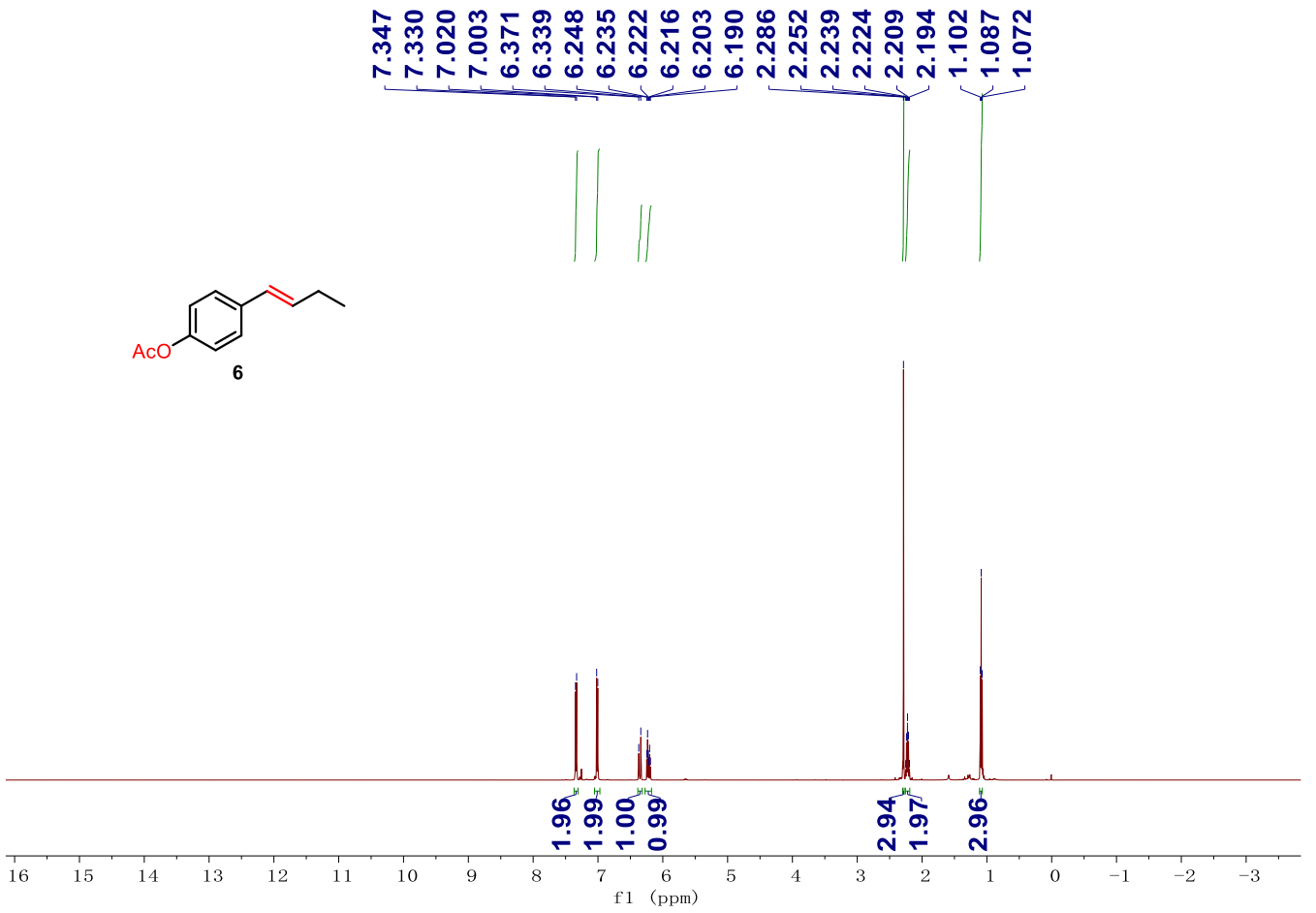
5
f1 (ppm)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6.235
6.222
6.216
6.203
6.190
2.286
2.252
2.239
2.224
2.209
2.194
1.102
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AcO
6
1.96
1.99
1.00
0.99
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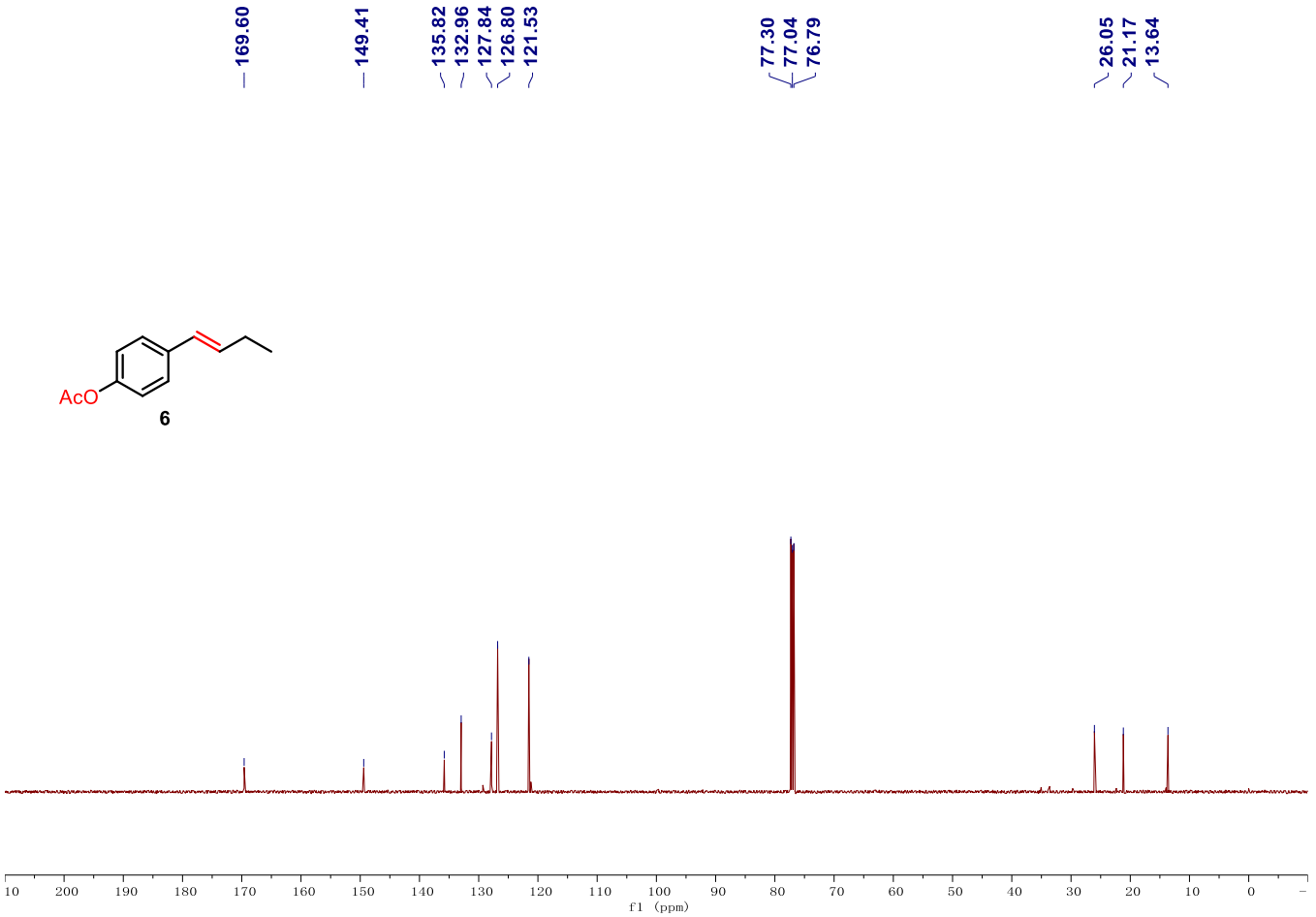
1.97
2.96
f1 (ppm)


169.60
149.41
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121.53
77.30
77.04
76.79
26.05
21.17
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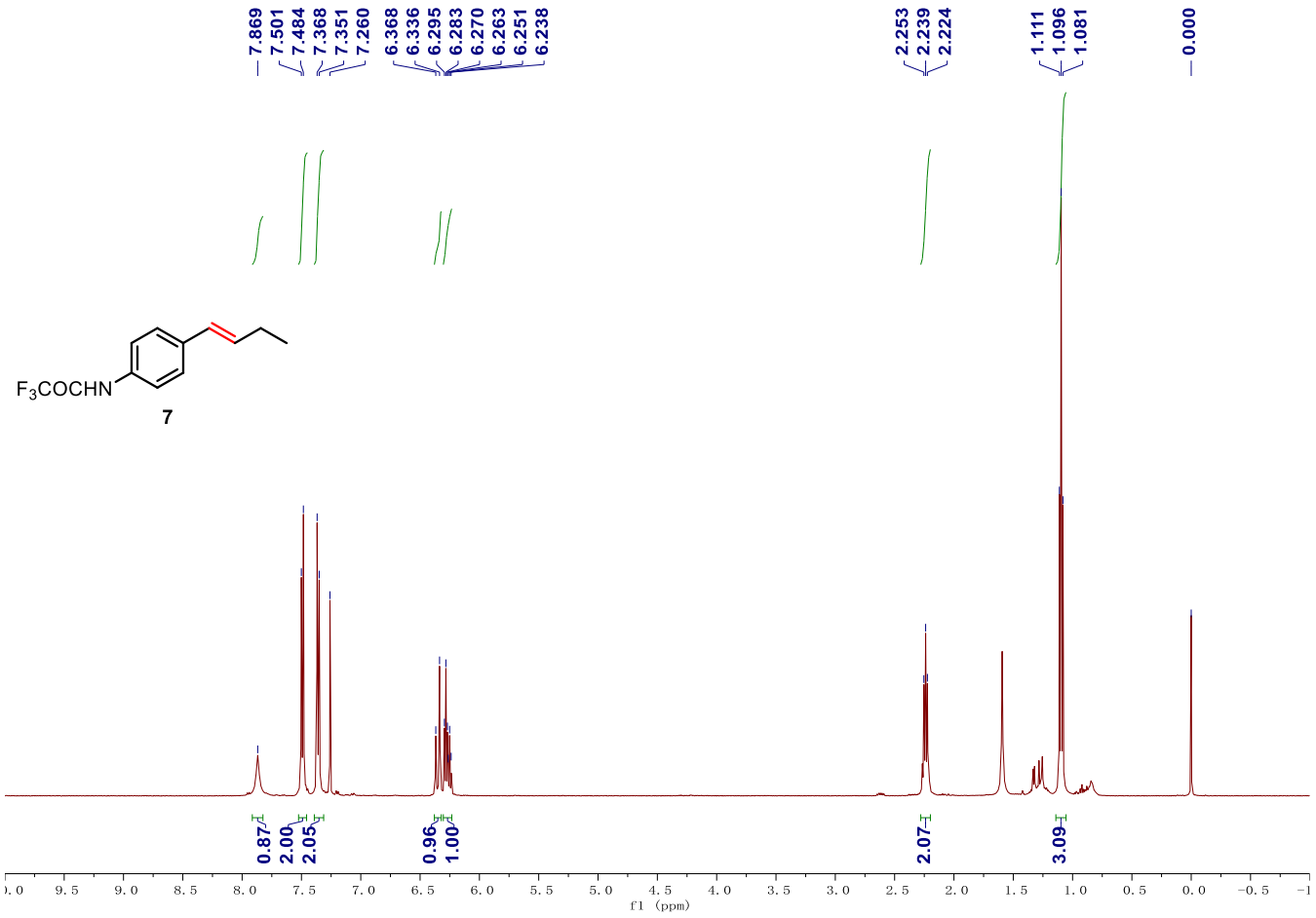
AcO
6
f1 (ppm)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6.336
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6.270
6.263
6.251
6.238
2.253
2.239
2.224
1.111
1.096
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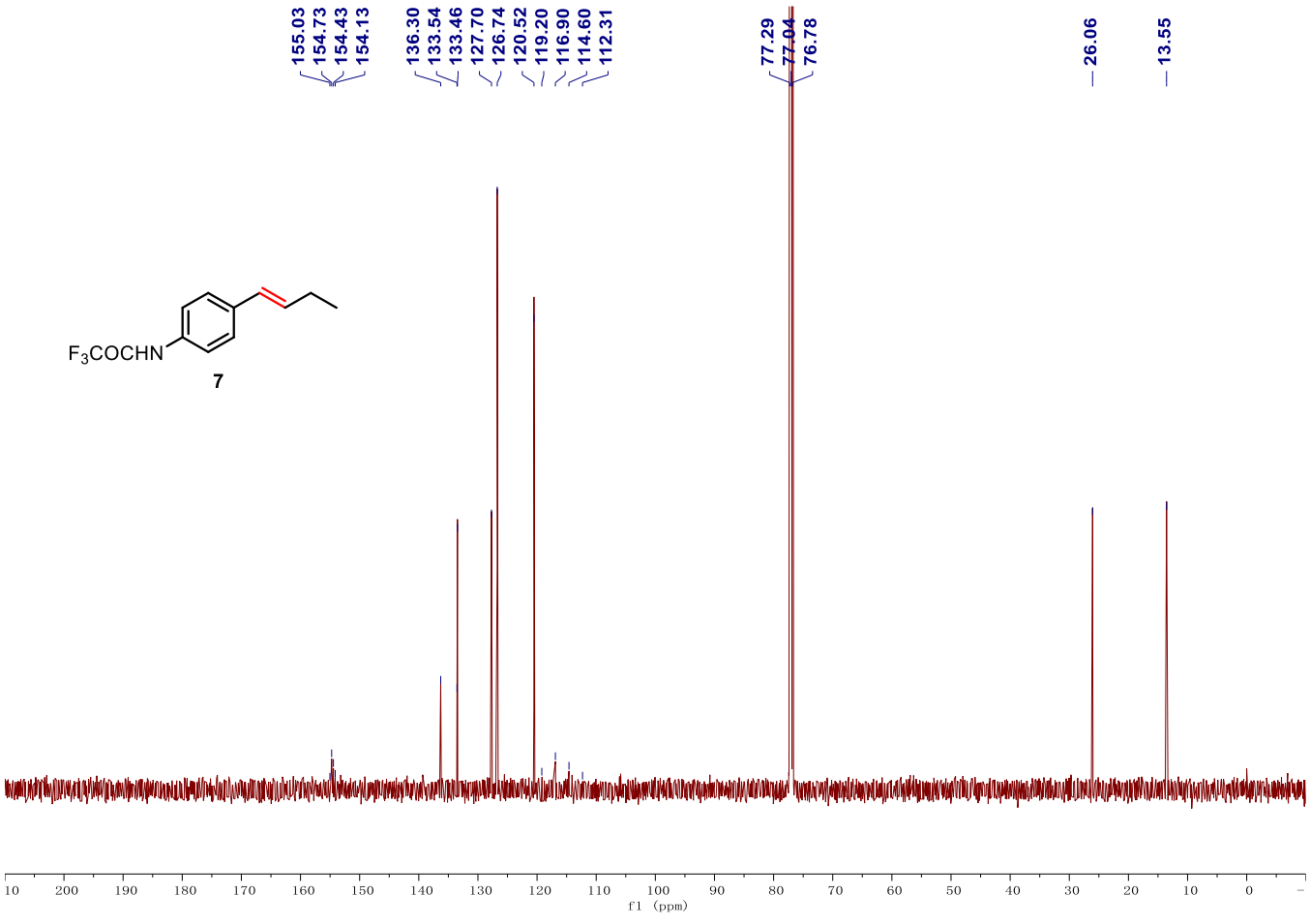
0.000
F3COCHN
7
0.87
2.00
2.05
0.96
1.00
2.07
3.09
f1 (ppm)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154.73
154.43
154.13
136.30
133.54
133.46
127.70
126.74
120.52
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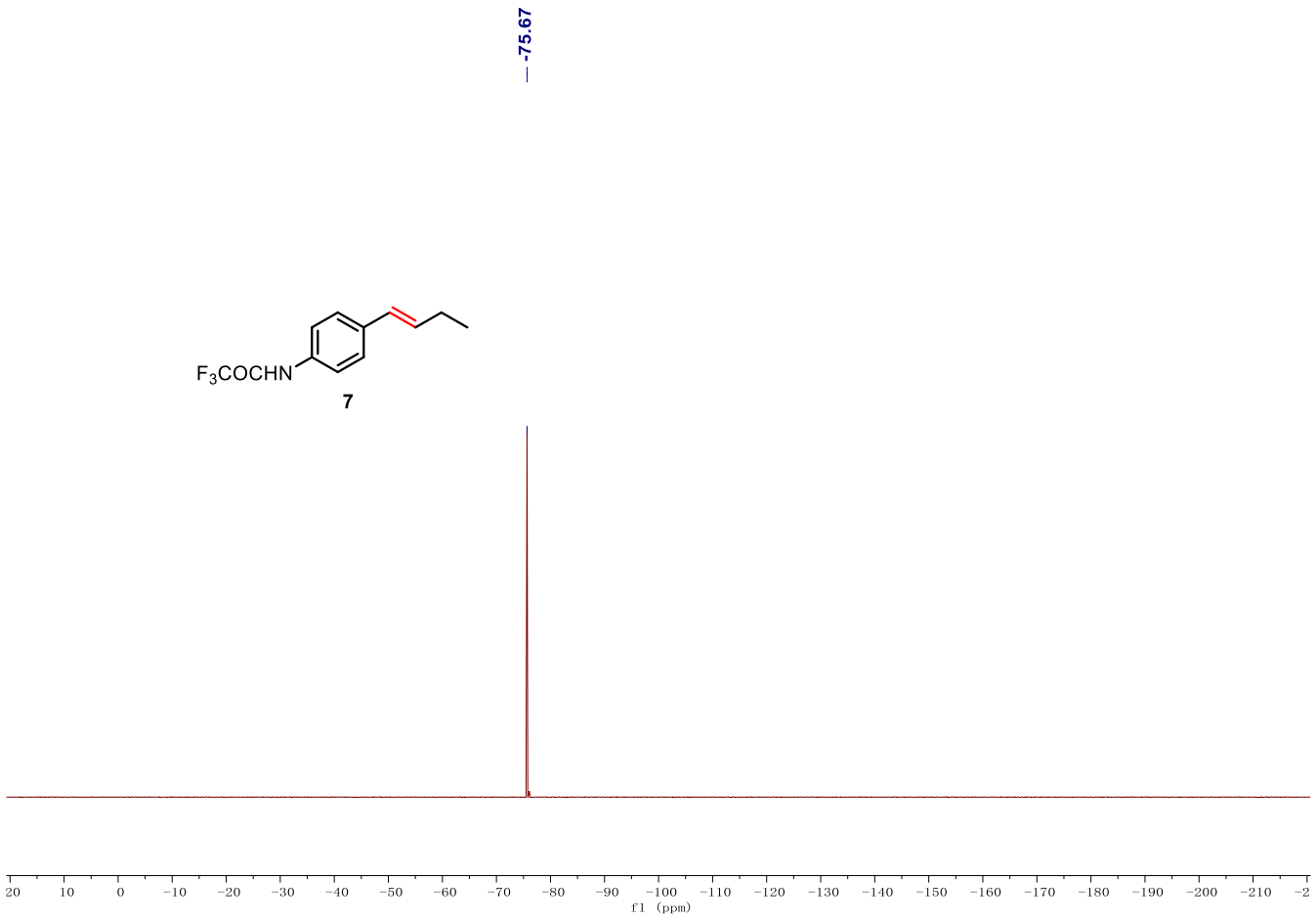
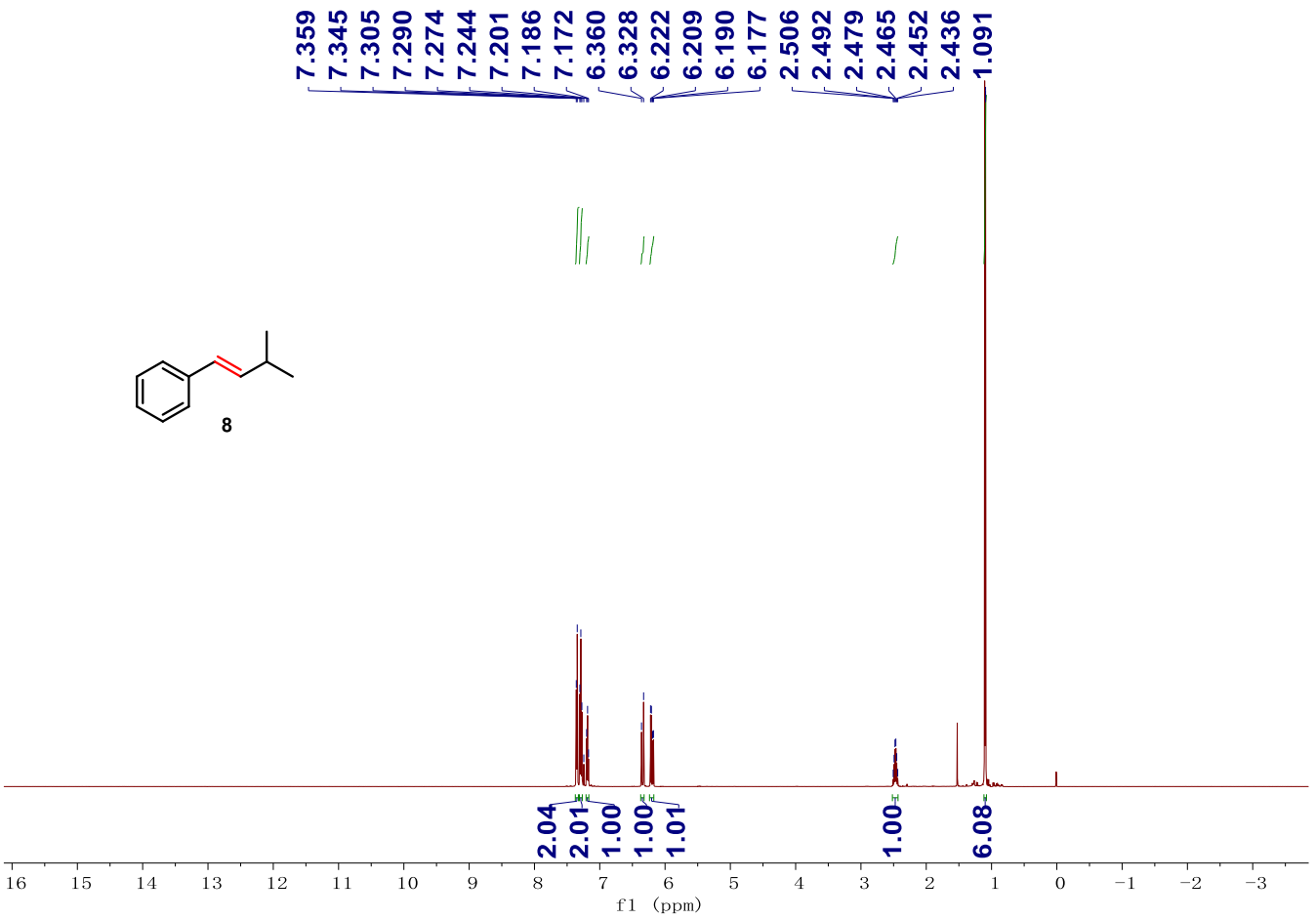
112.31
77.29
77.04
76.78
26.06
13.55
F3COCHN
7
f1 (ppm)


-75.67
F3COCHN
7
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210 −2
f1 (ppm)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7.290
7.274
7.244
7.201
7.186
7.172
6.360
6.328
6.222
6.209
6.190
6.177
2.506
2.492
2.479
2.465
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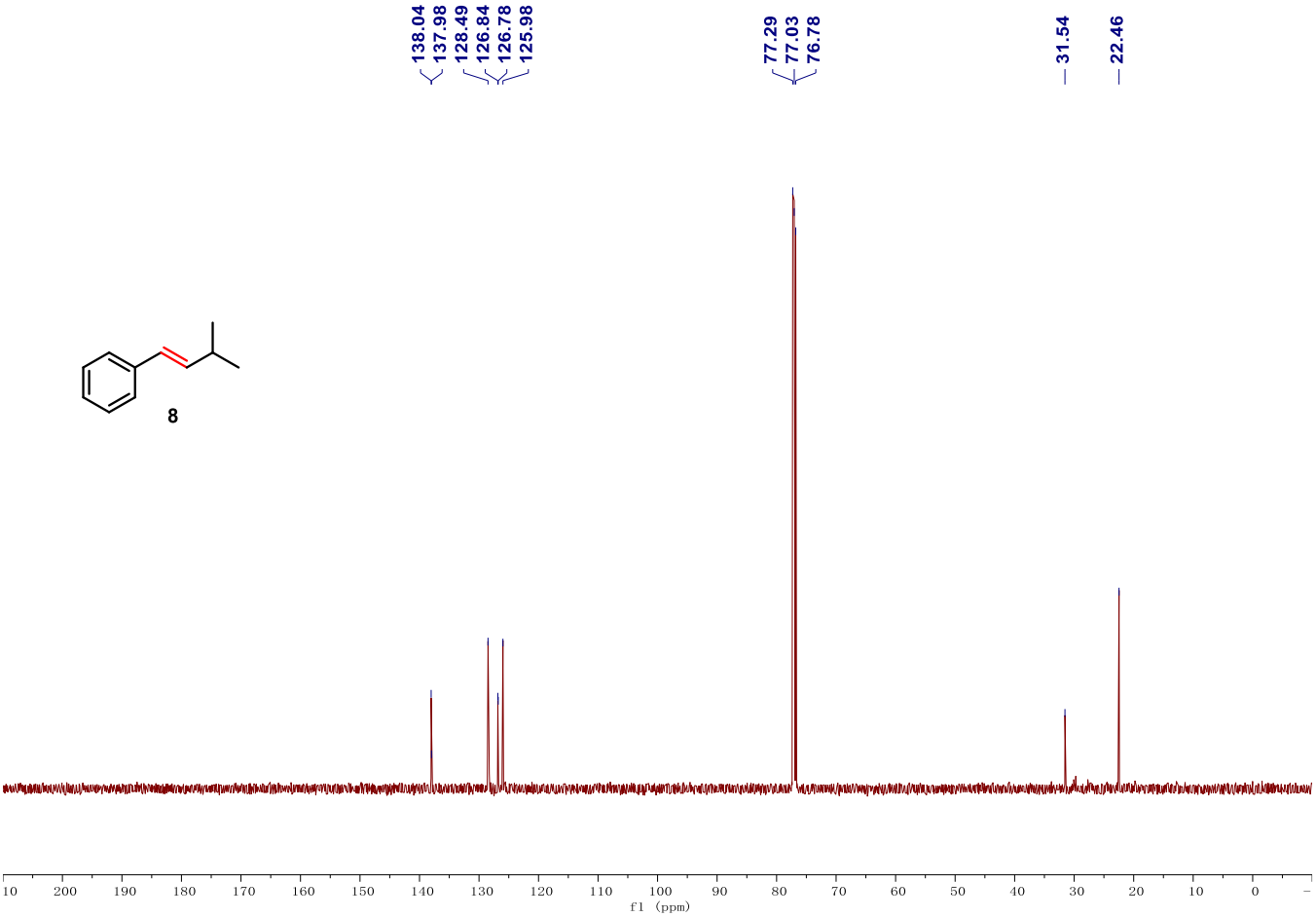
2.436
1.091
8
2.04
2.01
1.00
1.00
1.01
1.00
6.08
f1 (ppm)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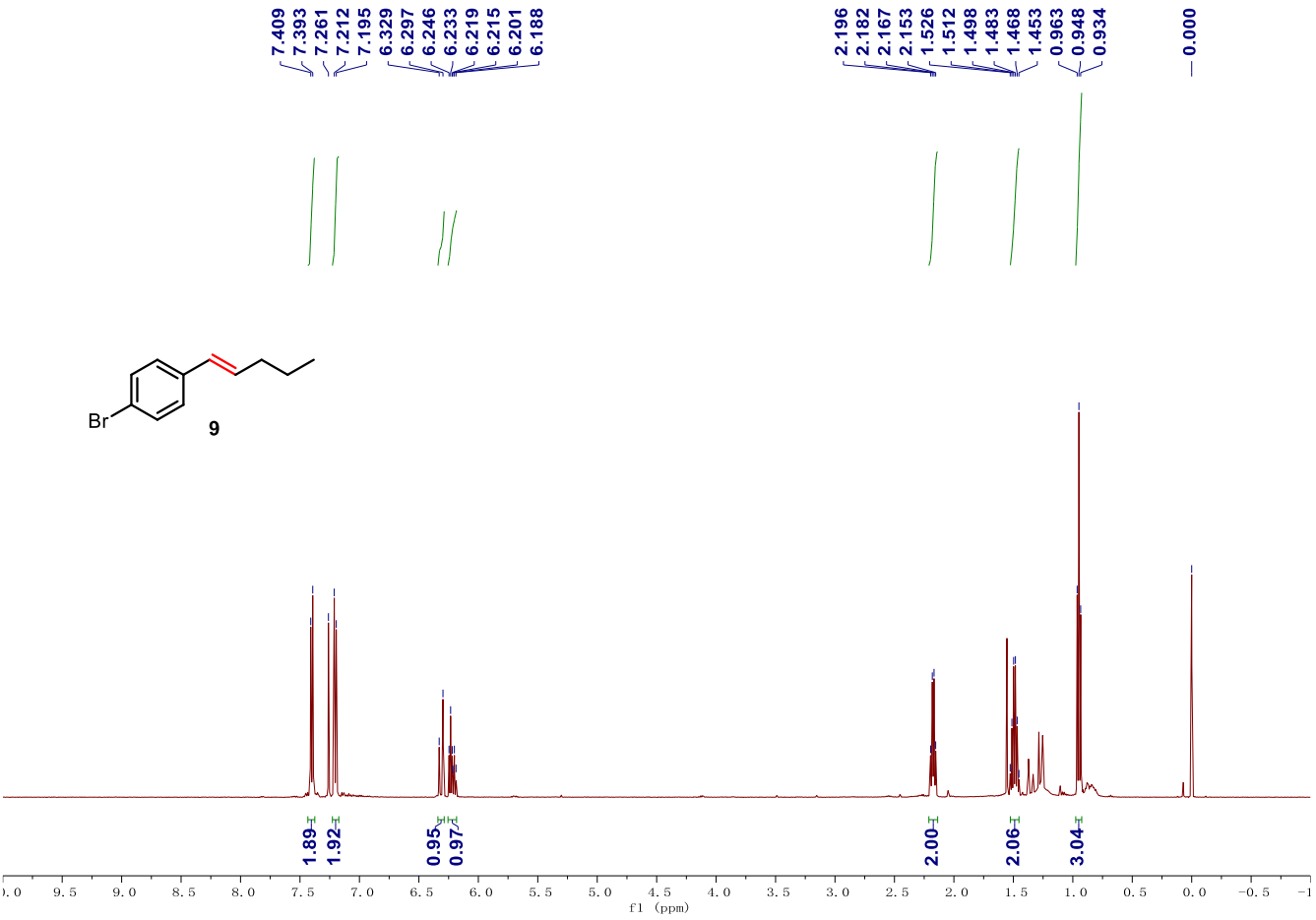
126.84
126.78
125.98
77.29
77.03
76.78
31.54
22.46
8
f1 (ppm)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7.261
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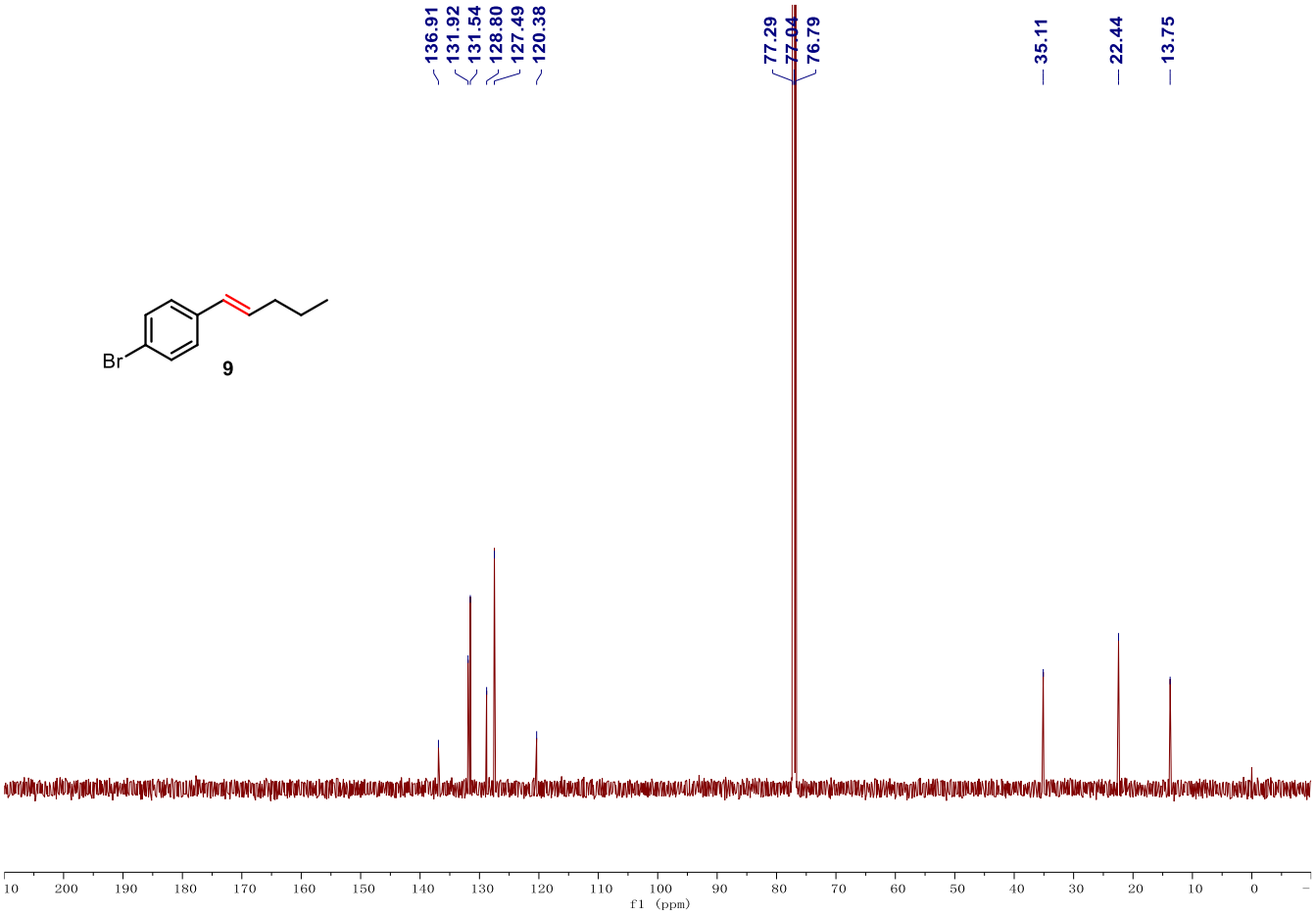
7.195
6.329
6.297
6.246
6.233
6.219
6.215
6.201
6.188
2.196
2.182
2.167
2.153
1.526
1.512
1.498
1.483
1.468
1.453
0.963
0.948
0.934
0.000
Br
9
1.89
1.92
0.95
0.97
2.00
2.06
3.04
f1 (ppm)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131.92
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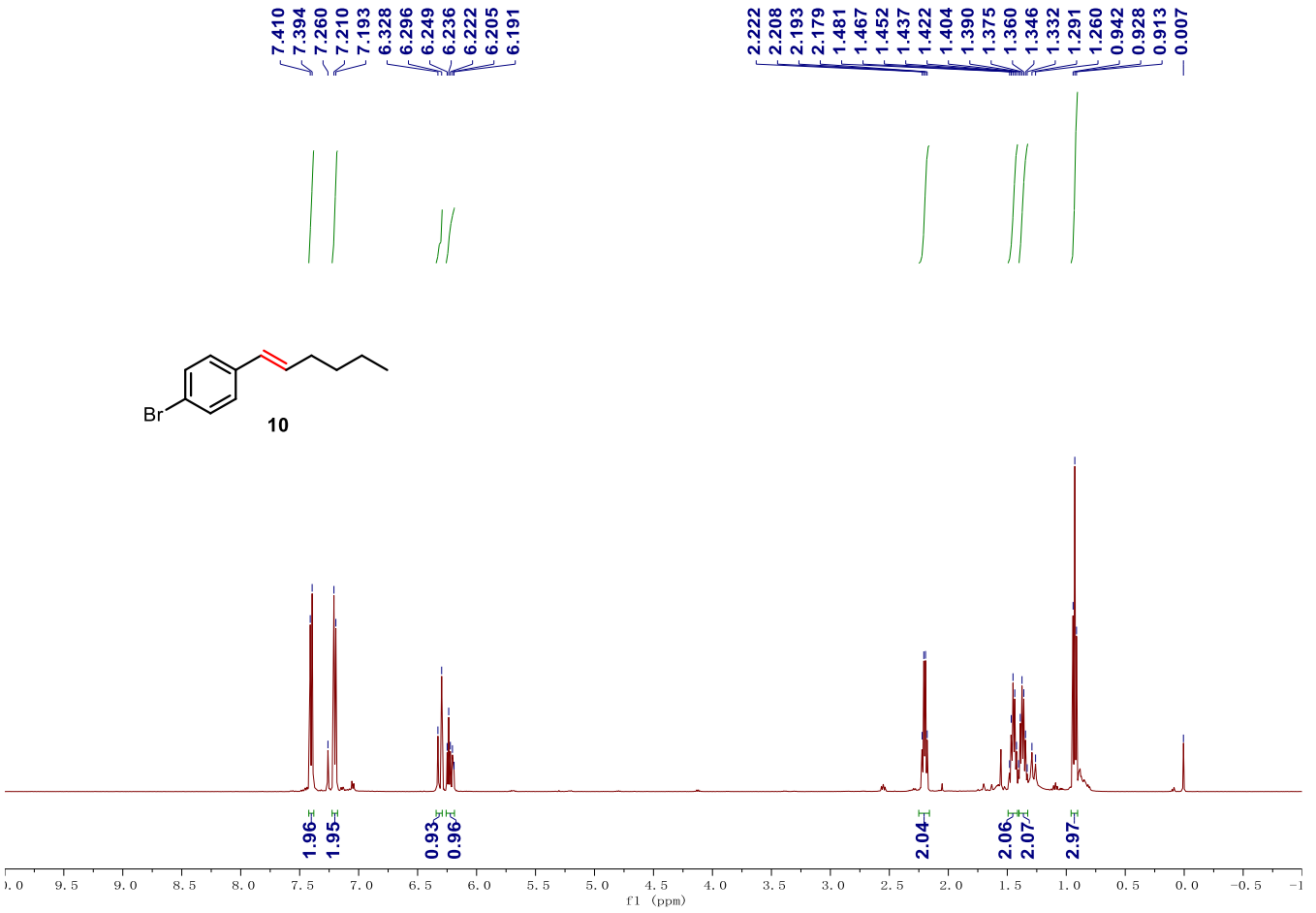
120.38
77.29
77.04
76.79
35.11
22.44
13.75
Br
9
f1 (ppm)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7.193
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6.296
6.249
6.236
6.222
6.205
6.191
2.222
2.208
2.193
2.179
1.481
1.467
1.452
1.437
1.422
1.404
1.390
1.375
1.360
1.346
1.332
1.291
1.260
0.942
0.928
0.913
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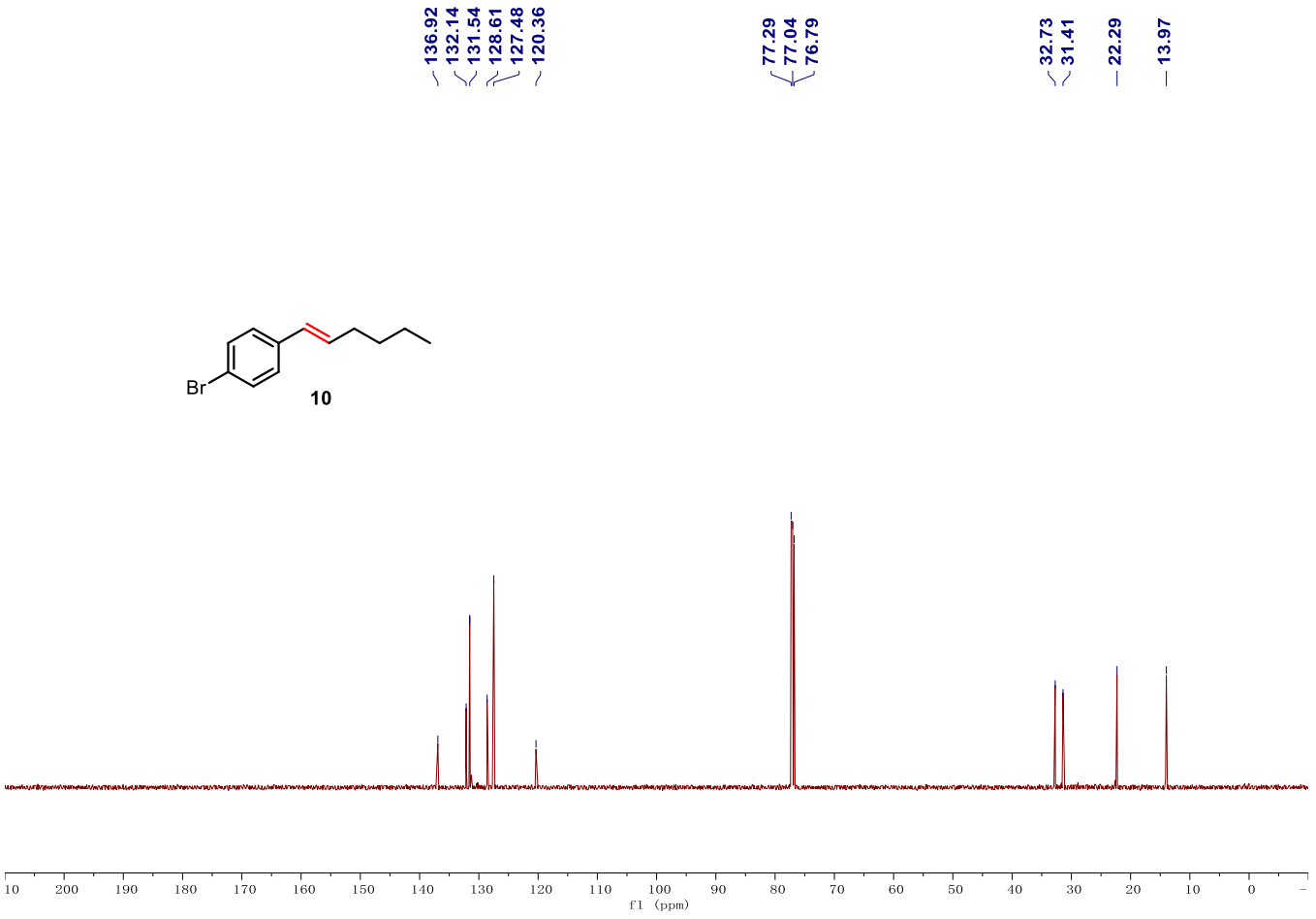
1.96
1.95
0.93
0.96
2.04
2.06
2.07
2.97
f1 (ppm)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77.04
76.79
32.73
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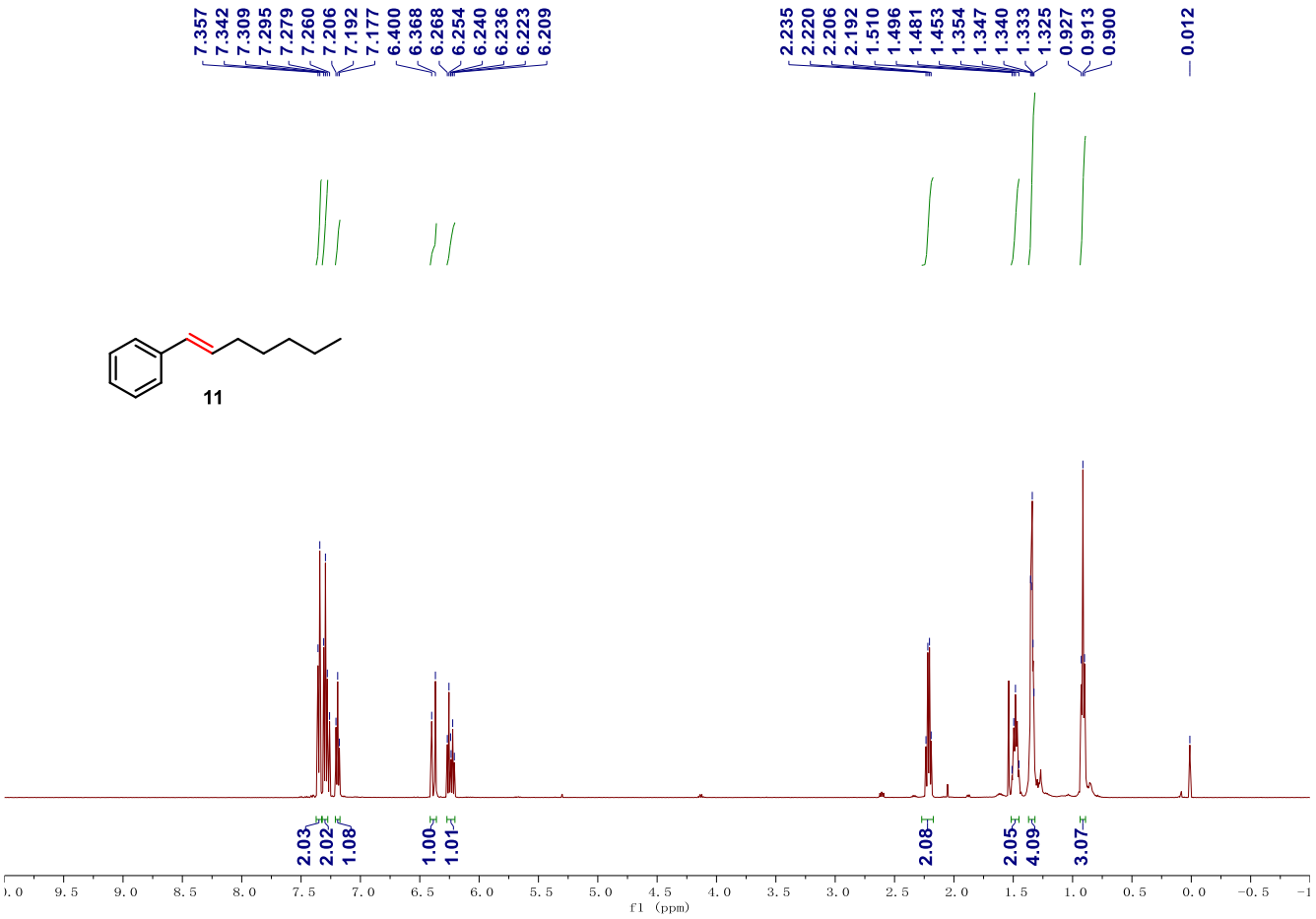
22.29
13.97
Br
10
f1 (ppm)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6.254
6.240
6.236
6.223
6.209
2.235
2.220
2.206
2.192
1.510
1.496
1.481
1.453
1.354
1.347
1.340
1.333
1.325
0.927
0.913
0.900
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2.02
1.08
1.00
1.01
2.08
2.05
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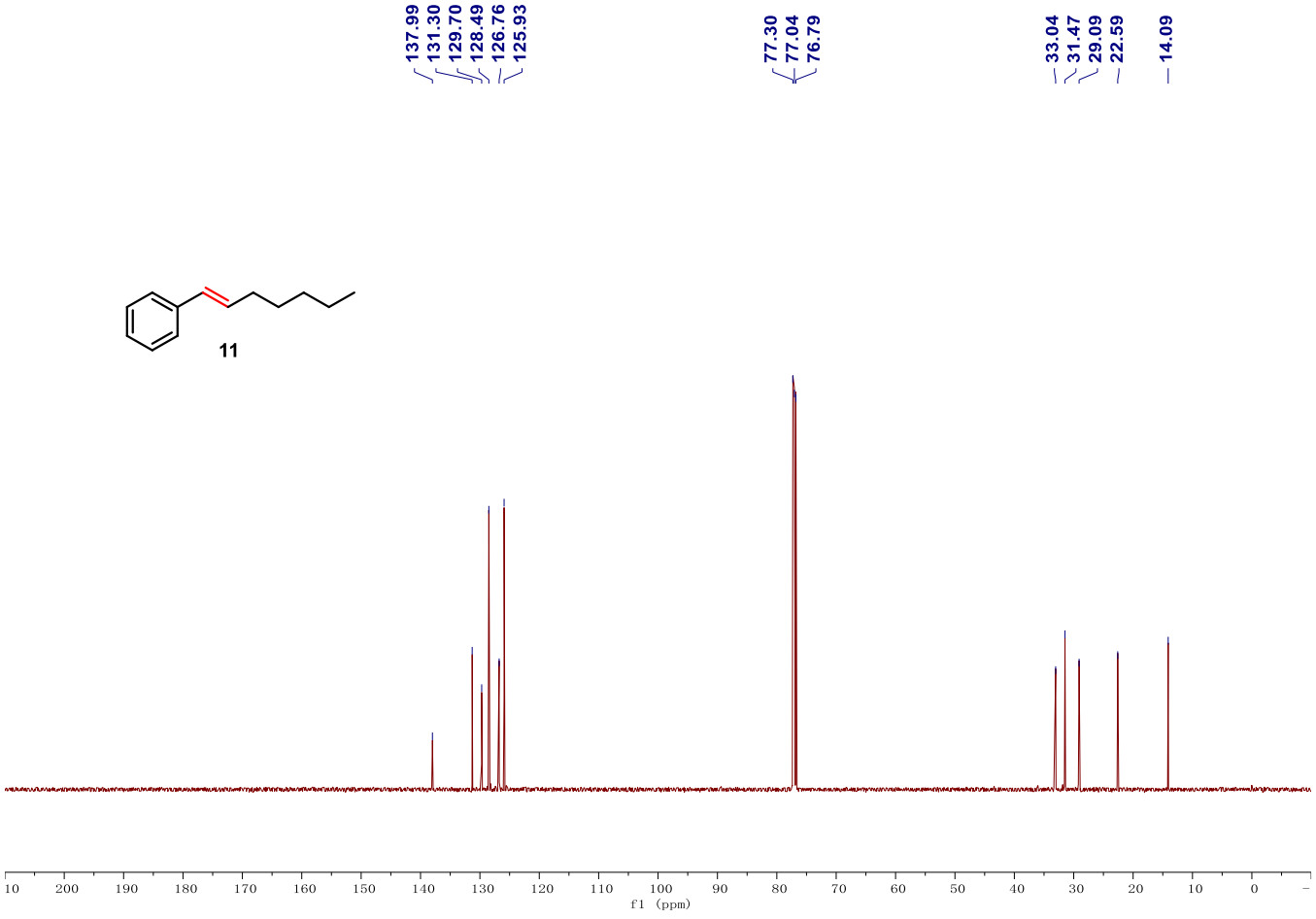
3.07
f1 (ppm)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76.79
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29.09
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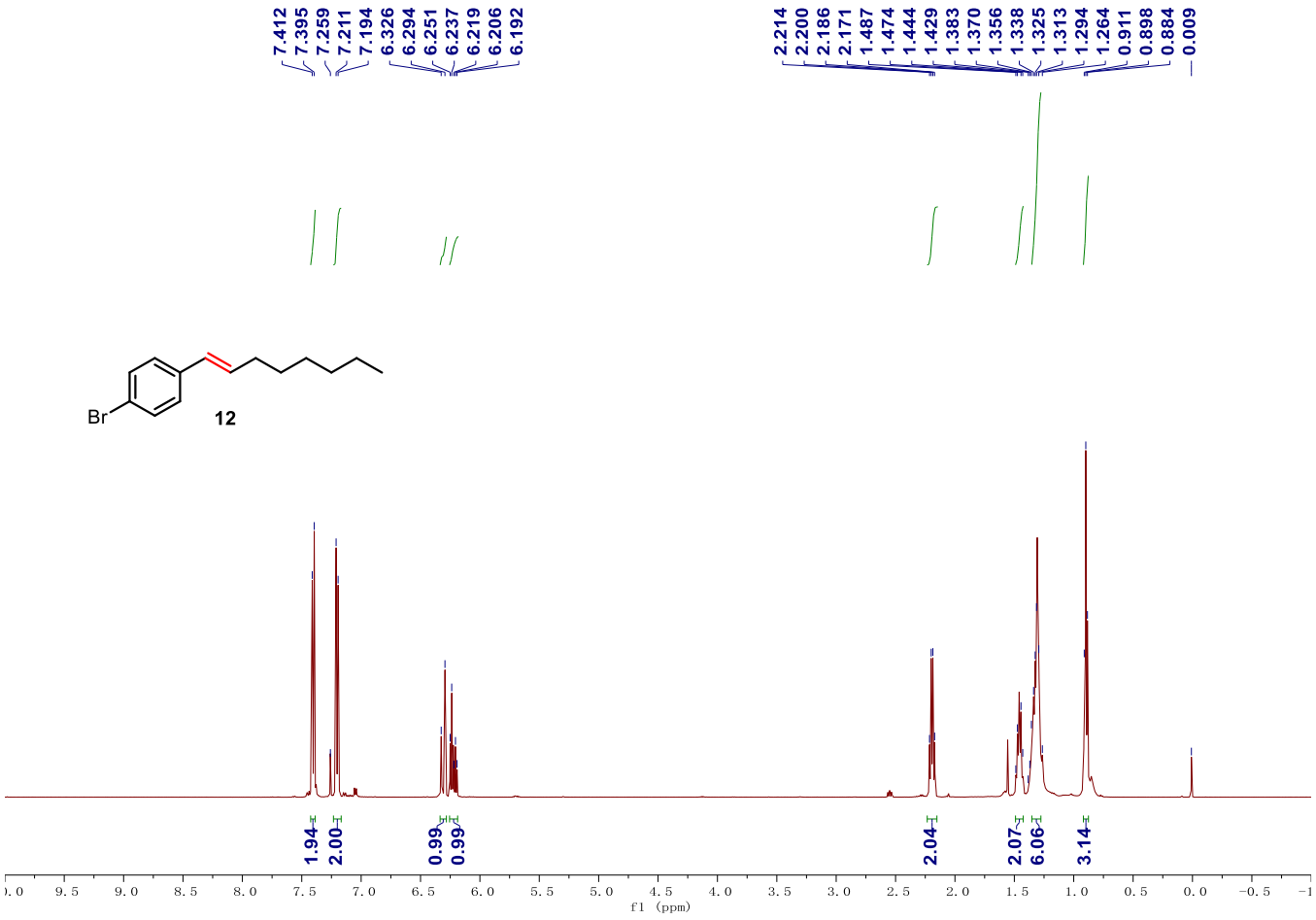
14.09
11
f1 (ppm)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6.192
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2.200
2.186
2.171
1.487
1.474
1.444
1.429
1.383
1.370
1.356
1.338
1.325
1.313
1.294
1.264
0.911
0.898
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2.07
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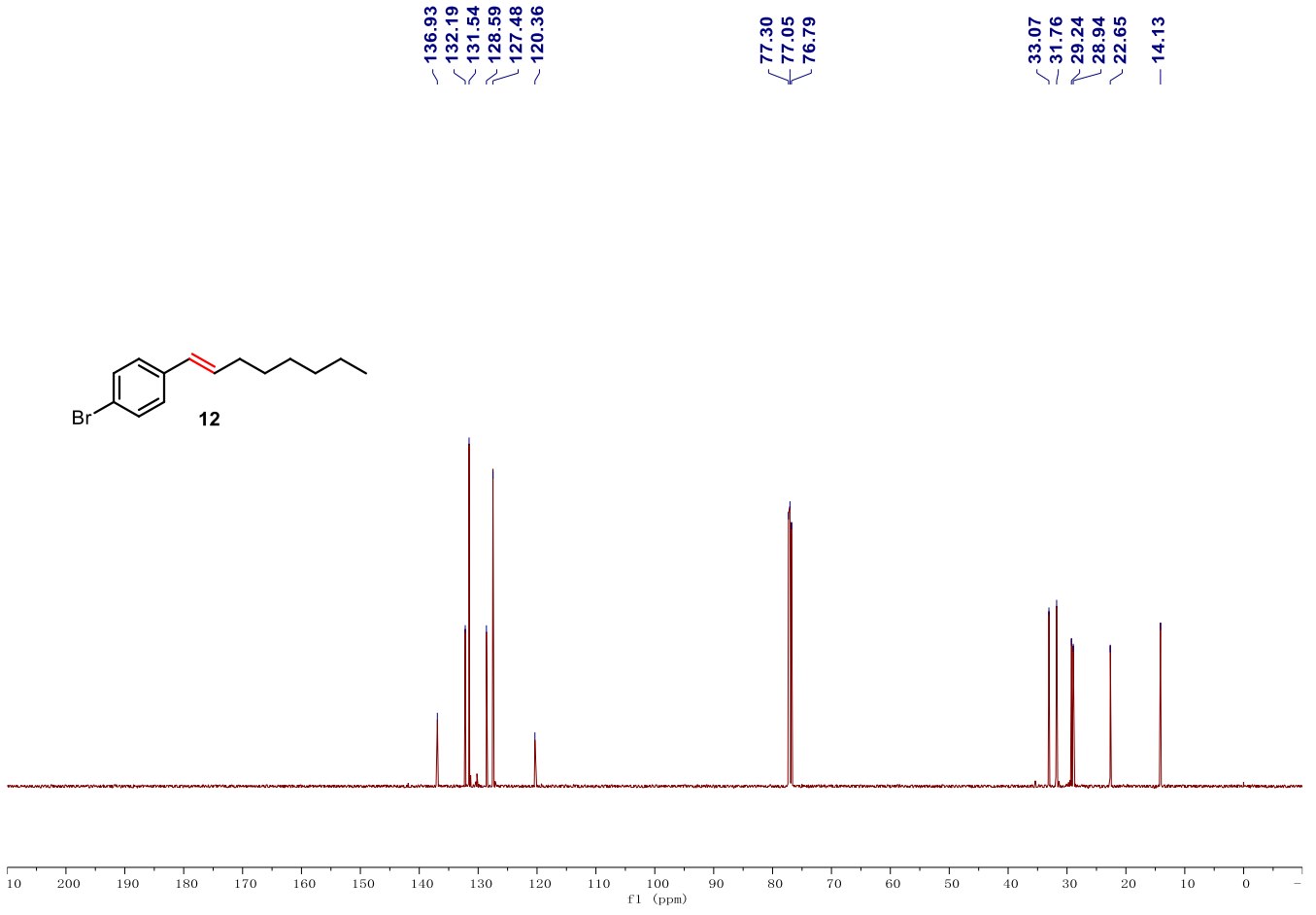
f1 (ppm)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22.65
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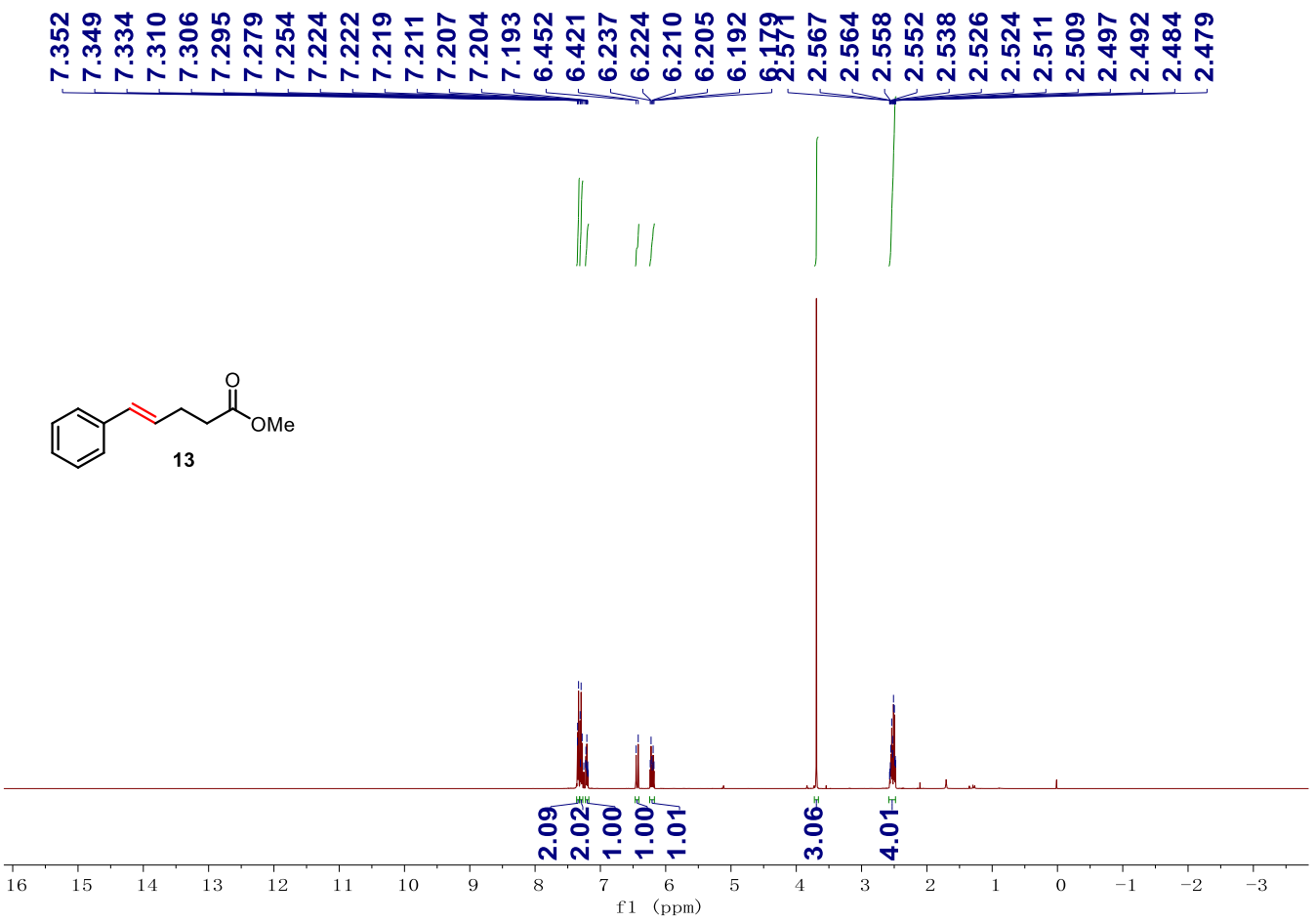
f1 (ppm)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7.207
7.204
7.193
6.452
6.421
6.237
6.224
6.210
6.205
6.192
6.179
6.171
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2.564
2.558
2.552
2.538
2.526
2.524
2.511
2.509
2.497
2.492
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1.00
1.01
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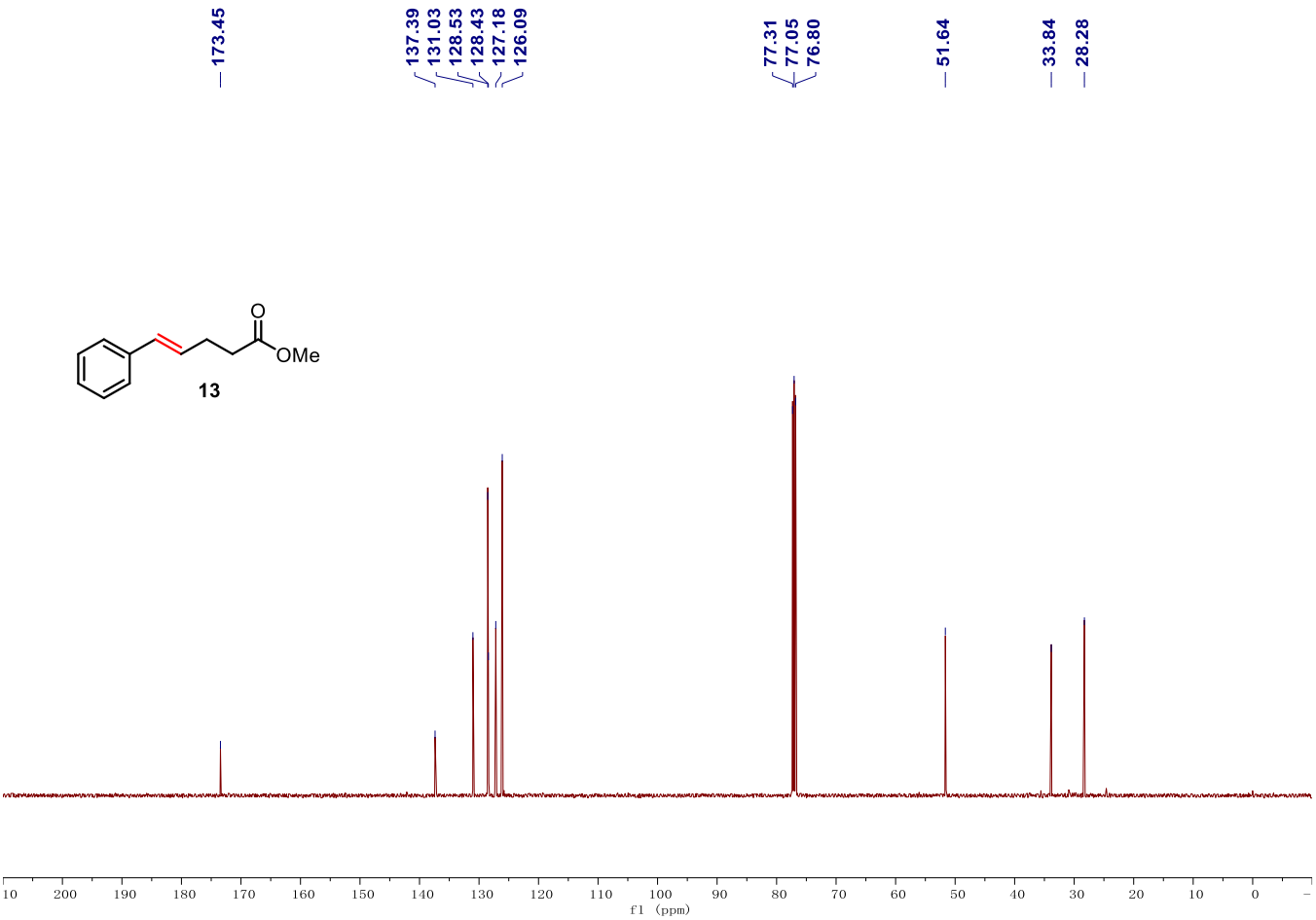
4.01
f1 (ppm)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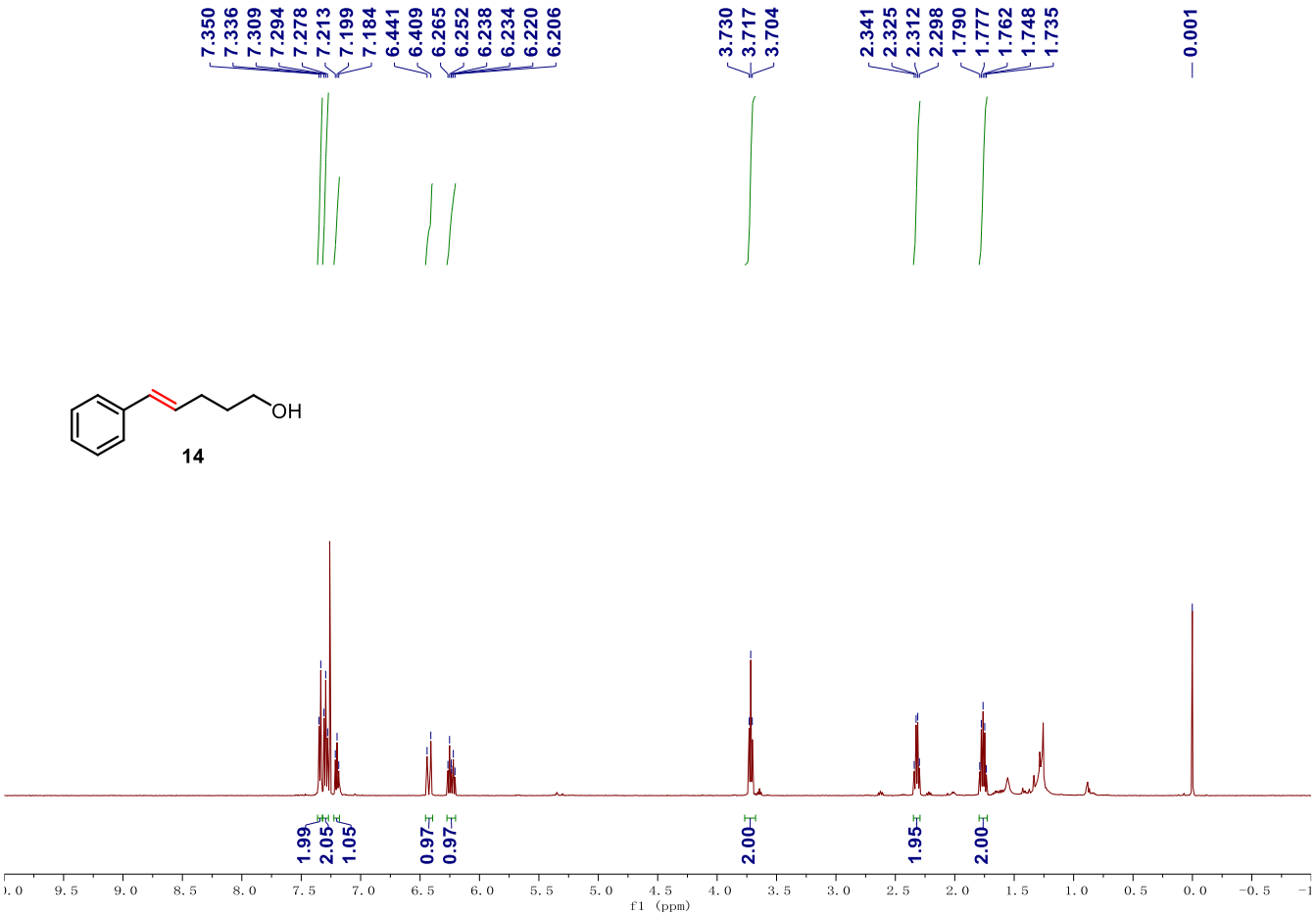
O
OMe
13
f1 (ppm)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6.265
6.252
6.238
6.234
6.220
6.206
3.730
3.717
3.704
2.341
2.325
2.312
2.298
1.790
1.777
1.762
1.748
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2.00
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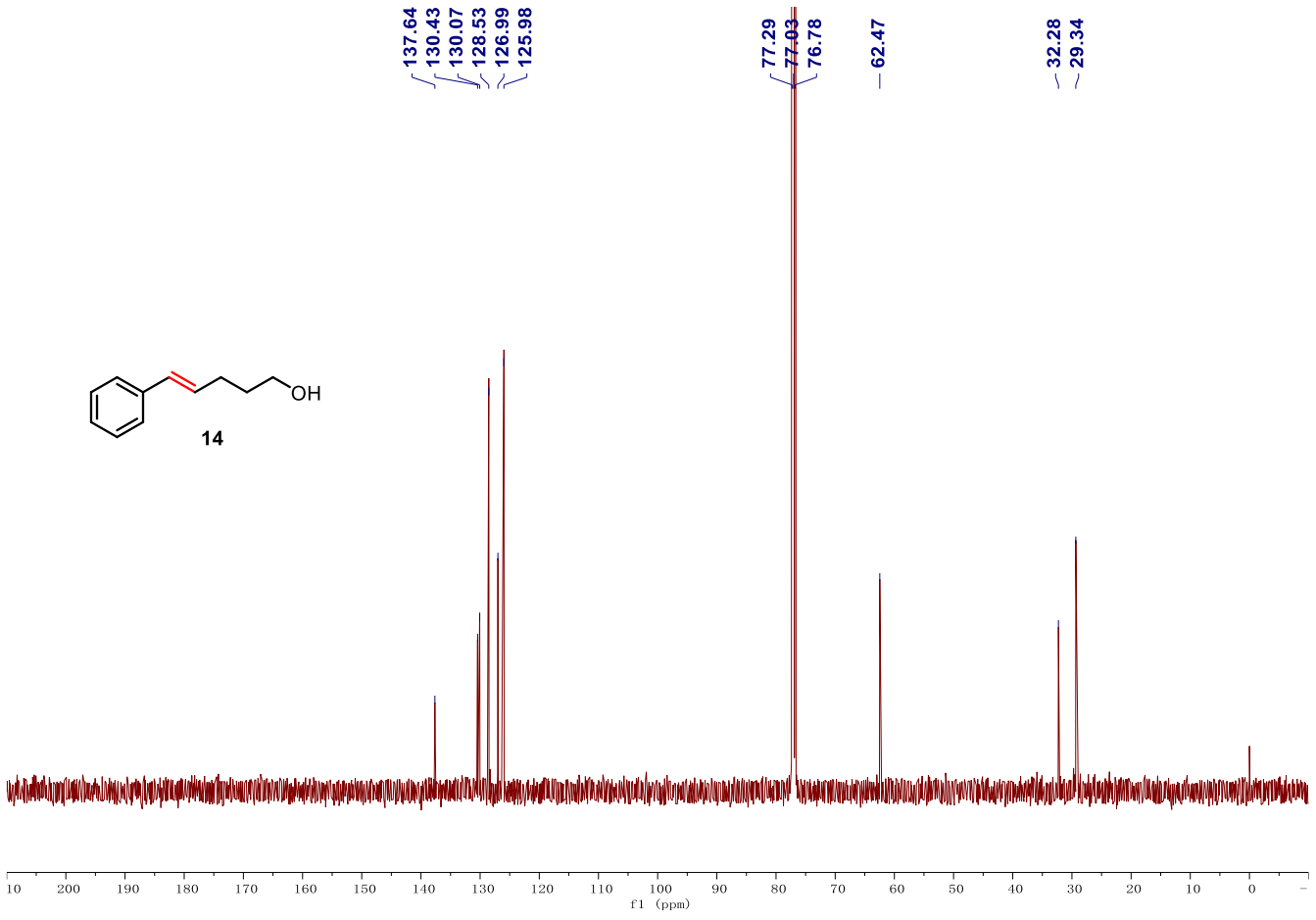
2.00
f1 (ppm)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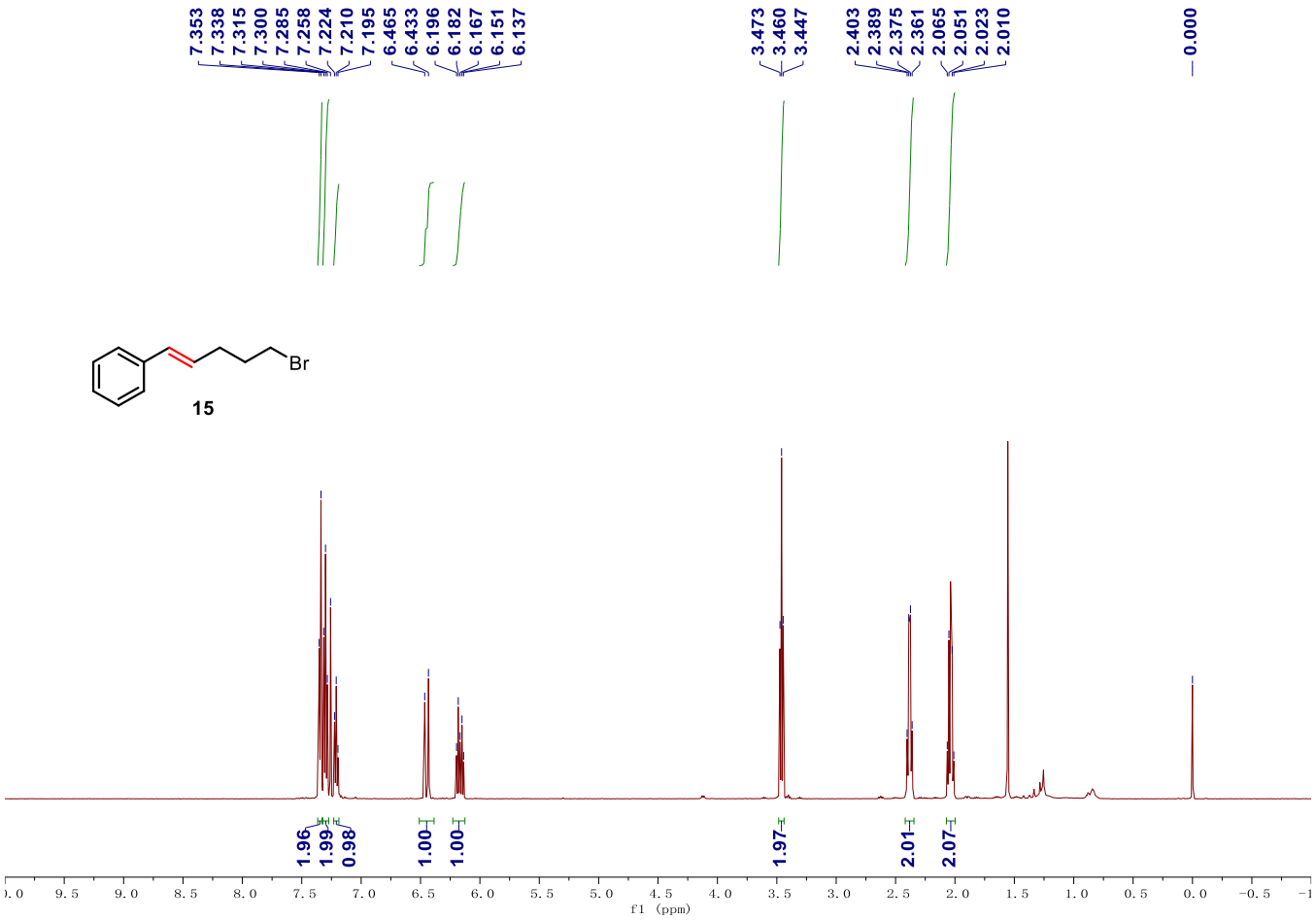
OH
14
f1 (ppm)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6.167
6.151
6.137
3.473
3.460
3.447
2.403
2.389
2.375
2.361
2.065
2.051
2.023
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Br
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0.98
1.00
1.00
1.97
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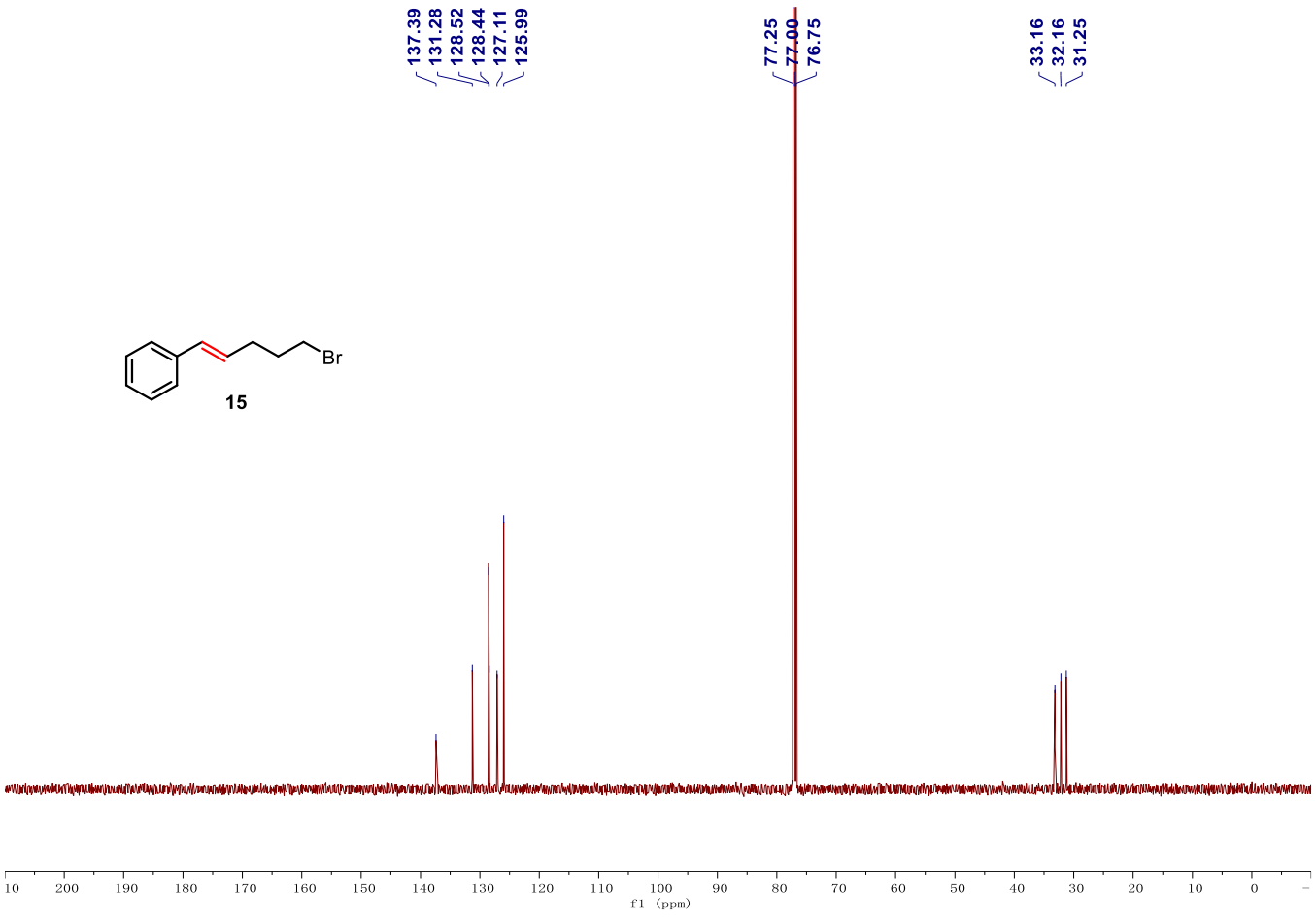
2.07
f1 (ppm)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77.00
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Br
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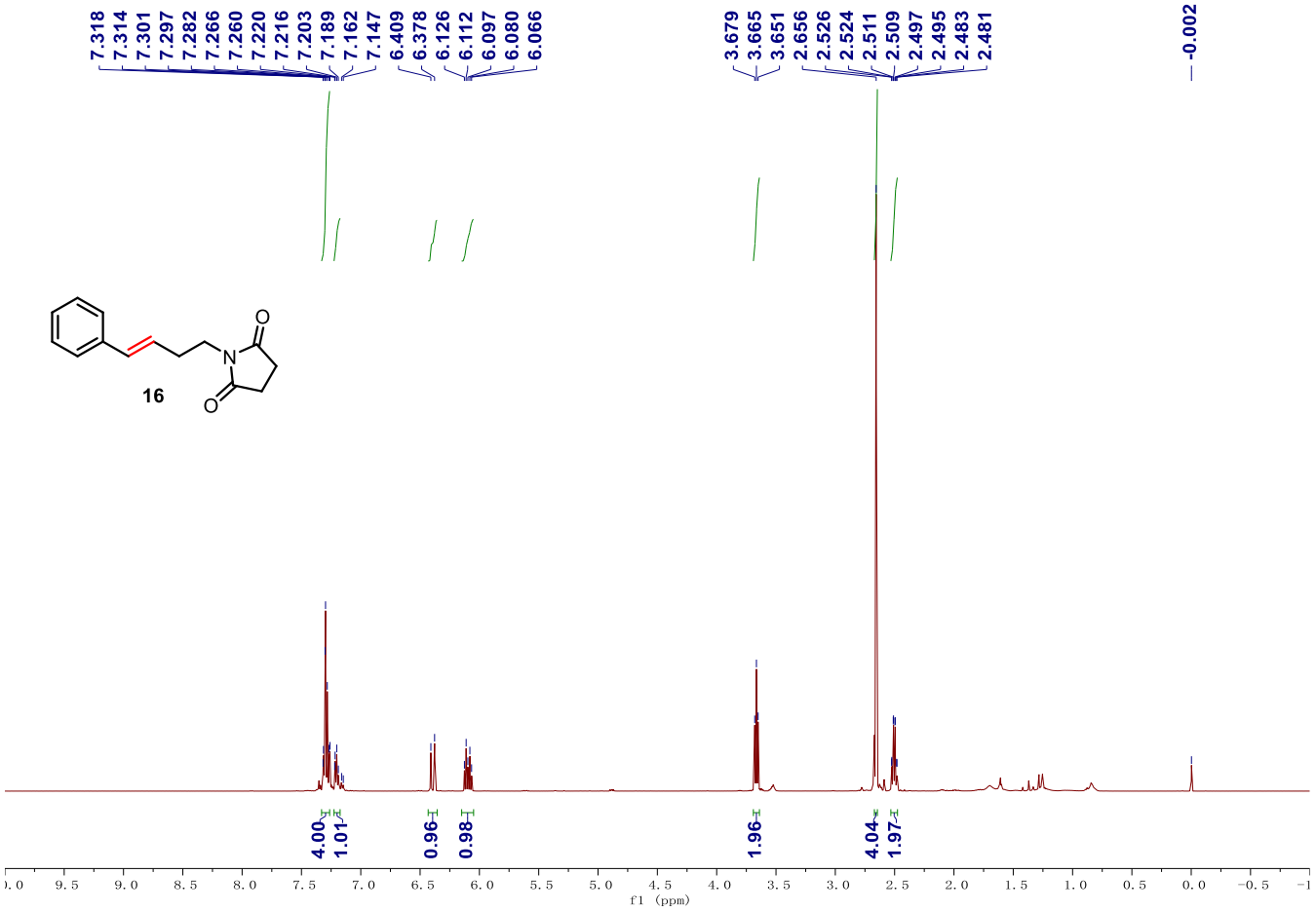
f1 (ppm)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3.665
3.651
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2.526
2.524
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2.509
2.497
2.495
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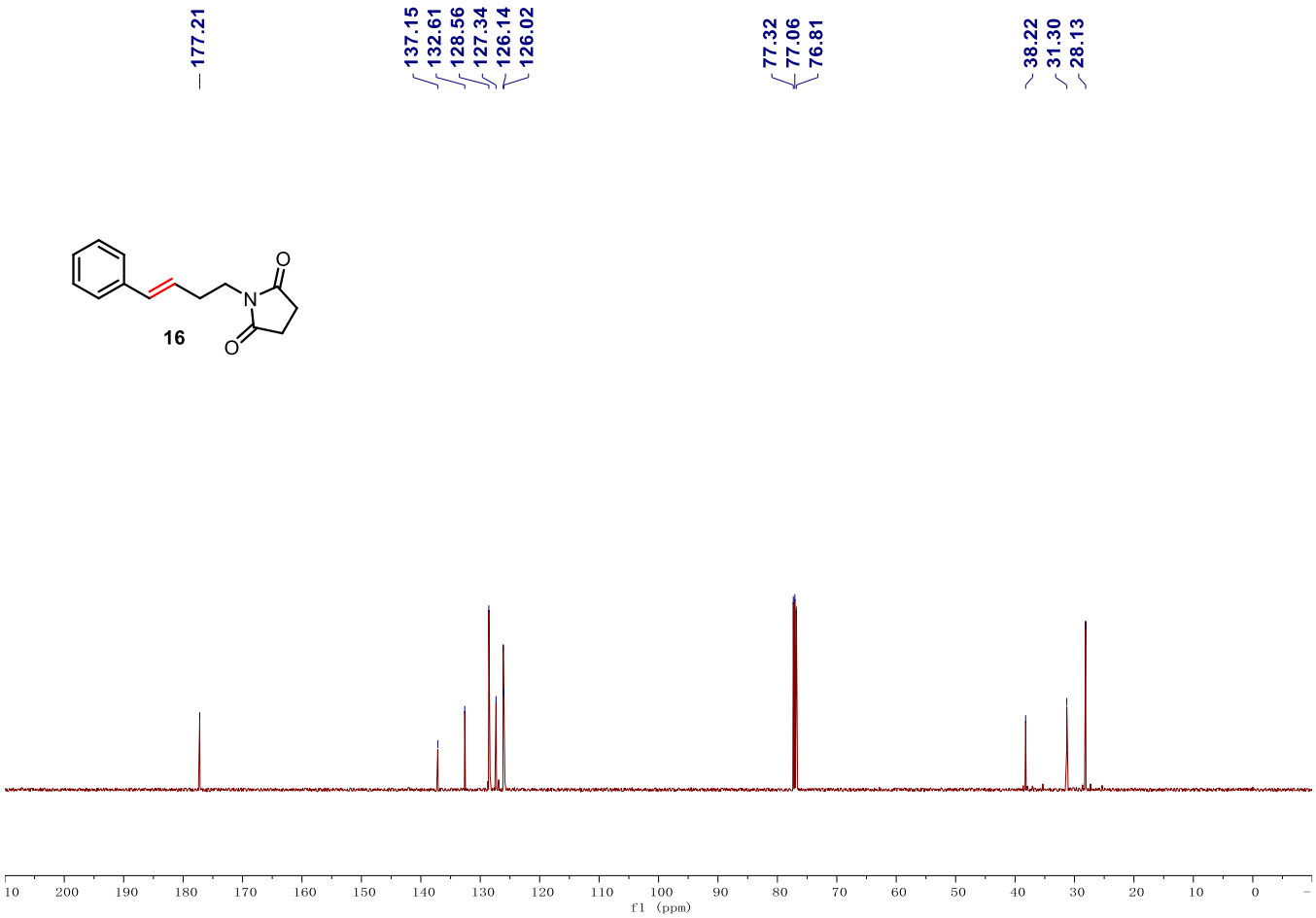
1.96
4.04
1.97
f1 (ppm)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38.22
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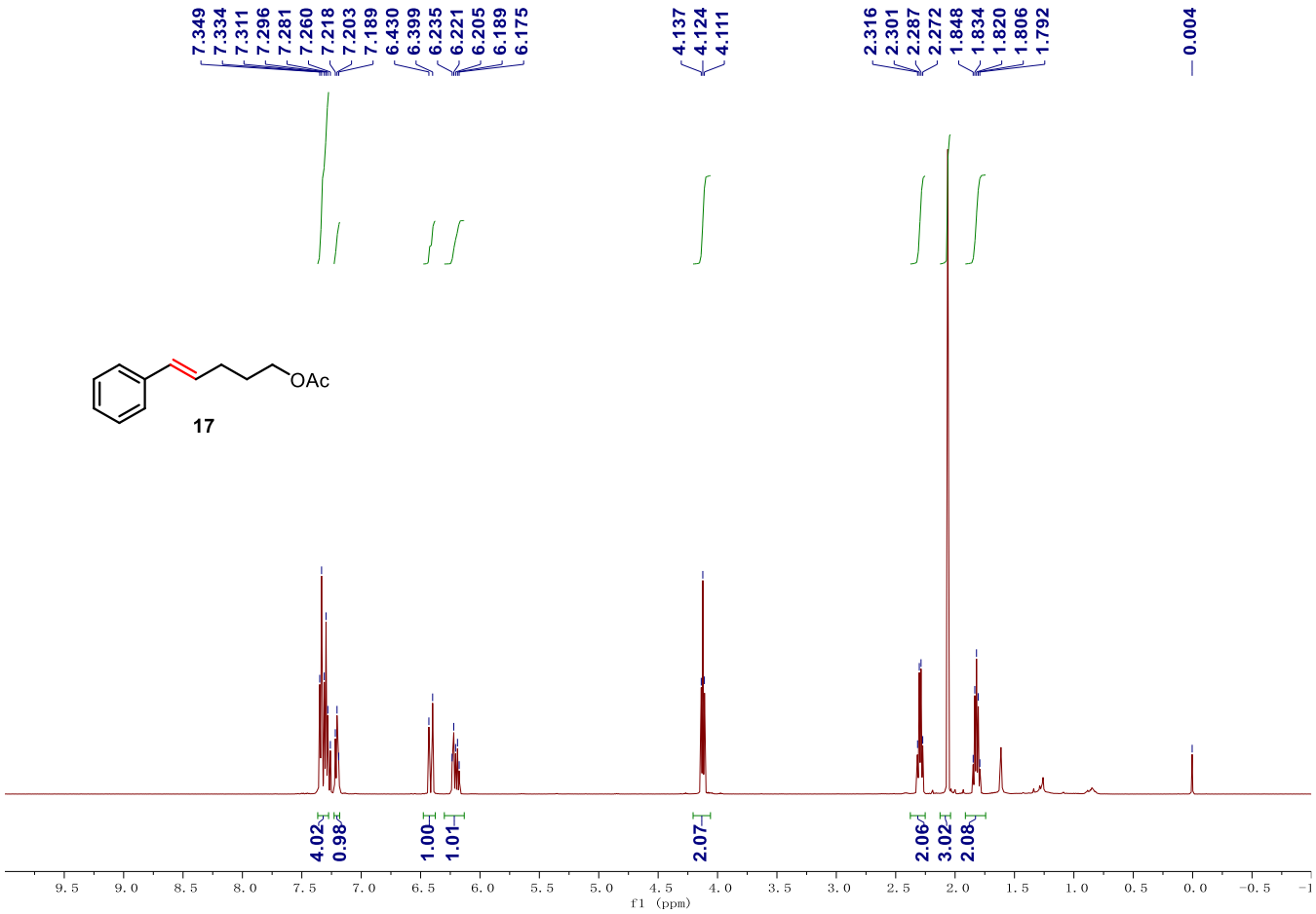
16
f1 (ppm)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6.399
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6.221
6.205
6.189
6.175
4.137
4.124
4.111
2.316
2.301
2.287
2.272
1.848
1.834
1.820
1.806
1.792
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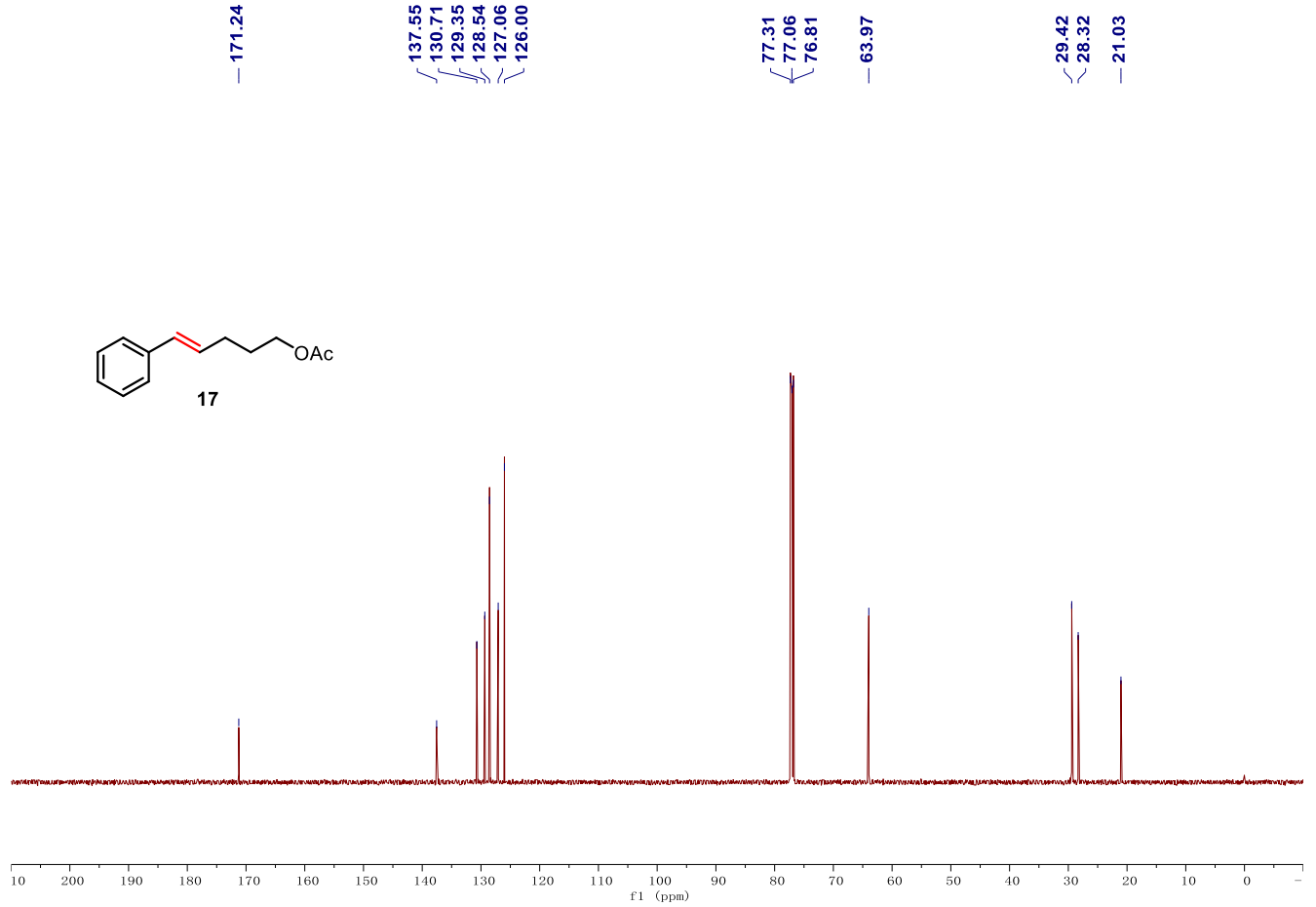
1.01
2.07
2.06
3.02
2.08
f1 (ppm)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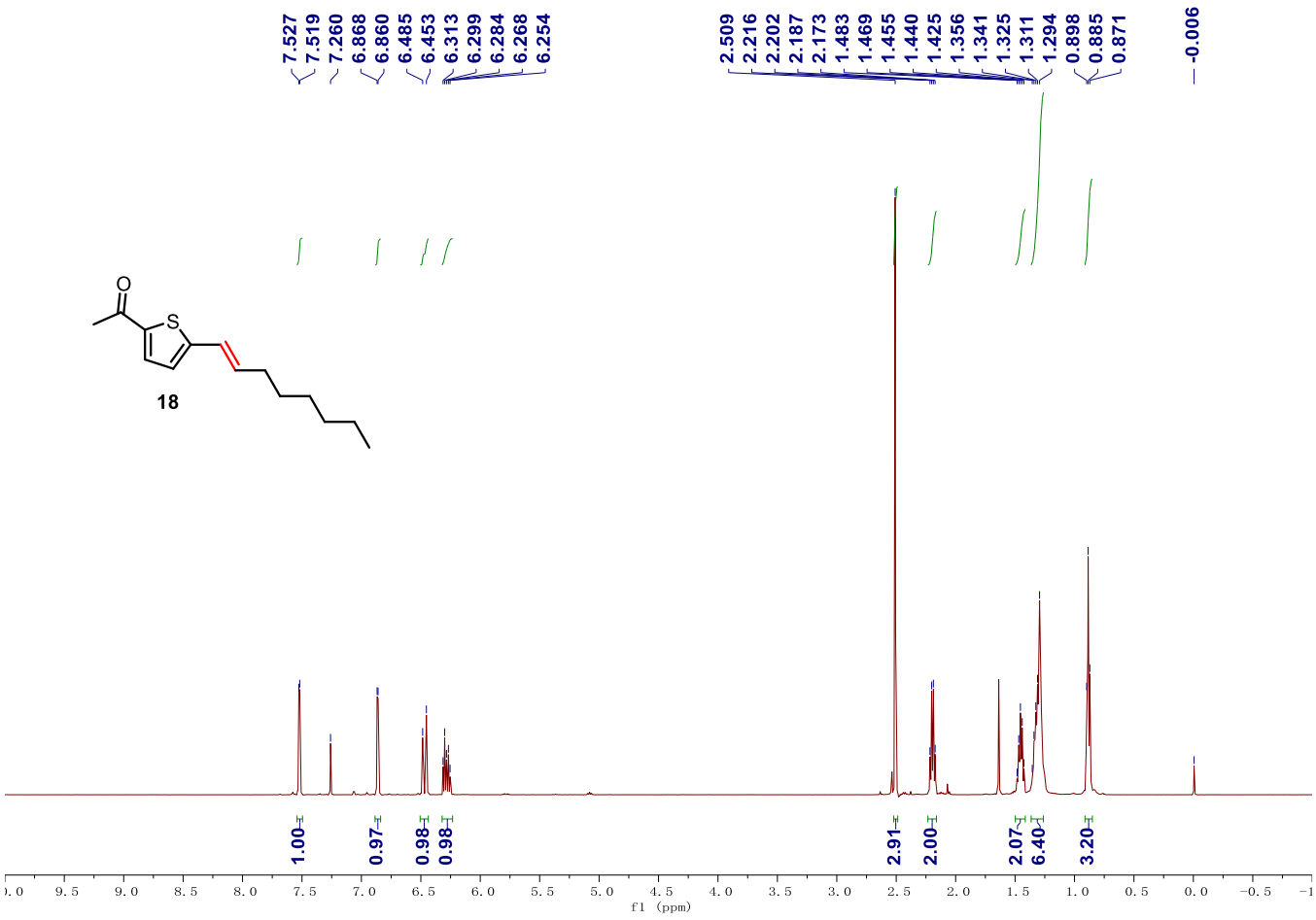
OAc
17
f1 (ppm)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6.254
2.509
2.216
2.202
2.187
2.173
1.483
1.469
1.455
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1.425
1.356
1.341
1.325
1.311
1.294
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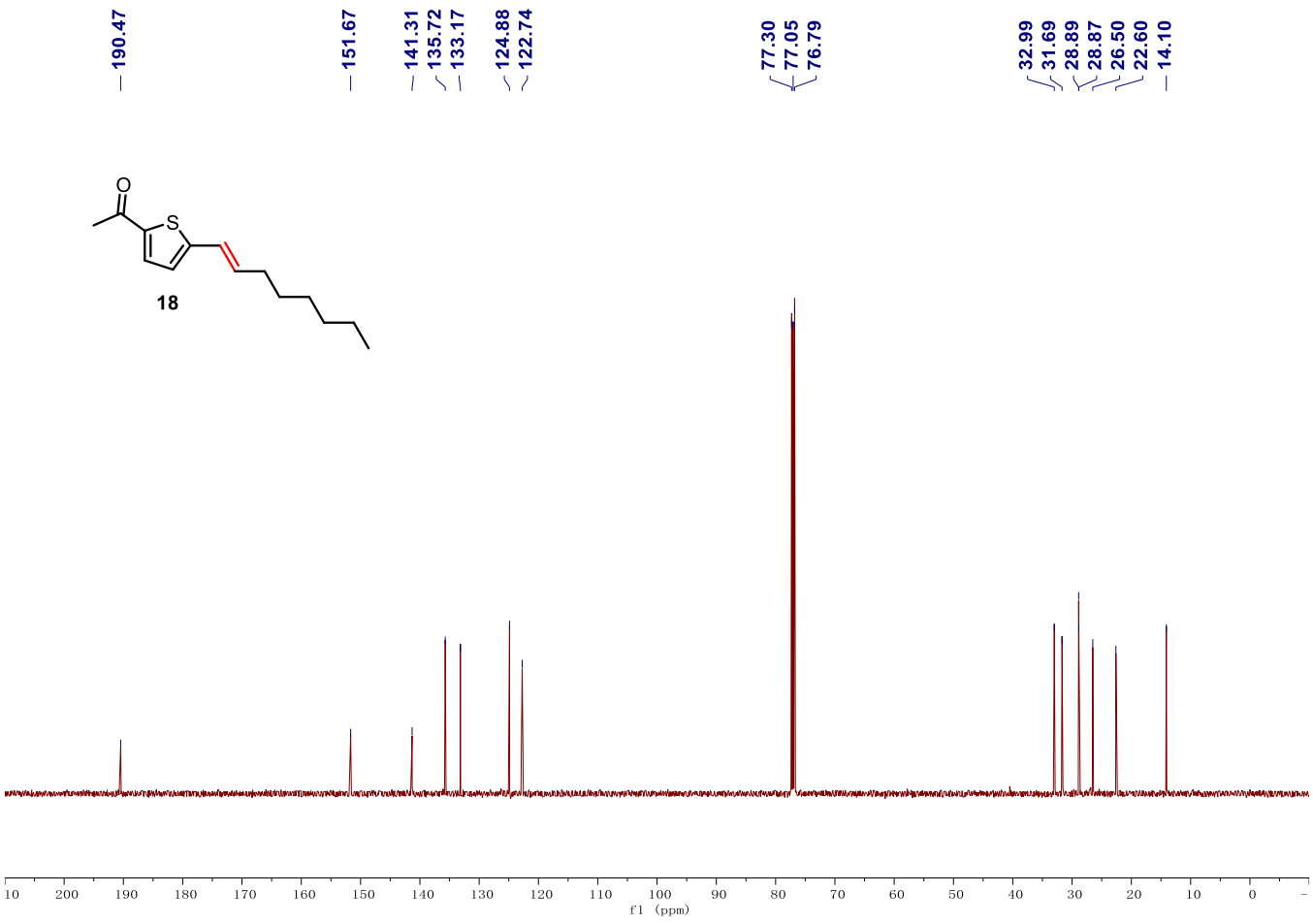
0.885
0.871
-0.006
O
S
18
1.00
0.97
0.98
0.98
2.91
2.00
2.07
6.40
3.20
f1 (ppm)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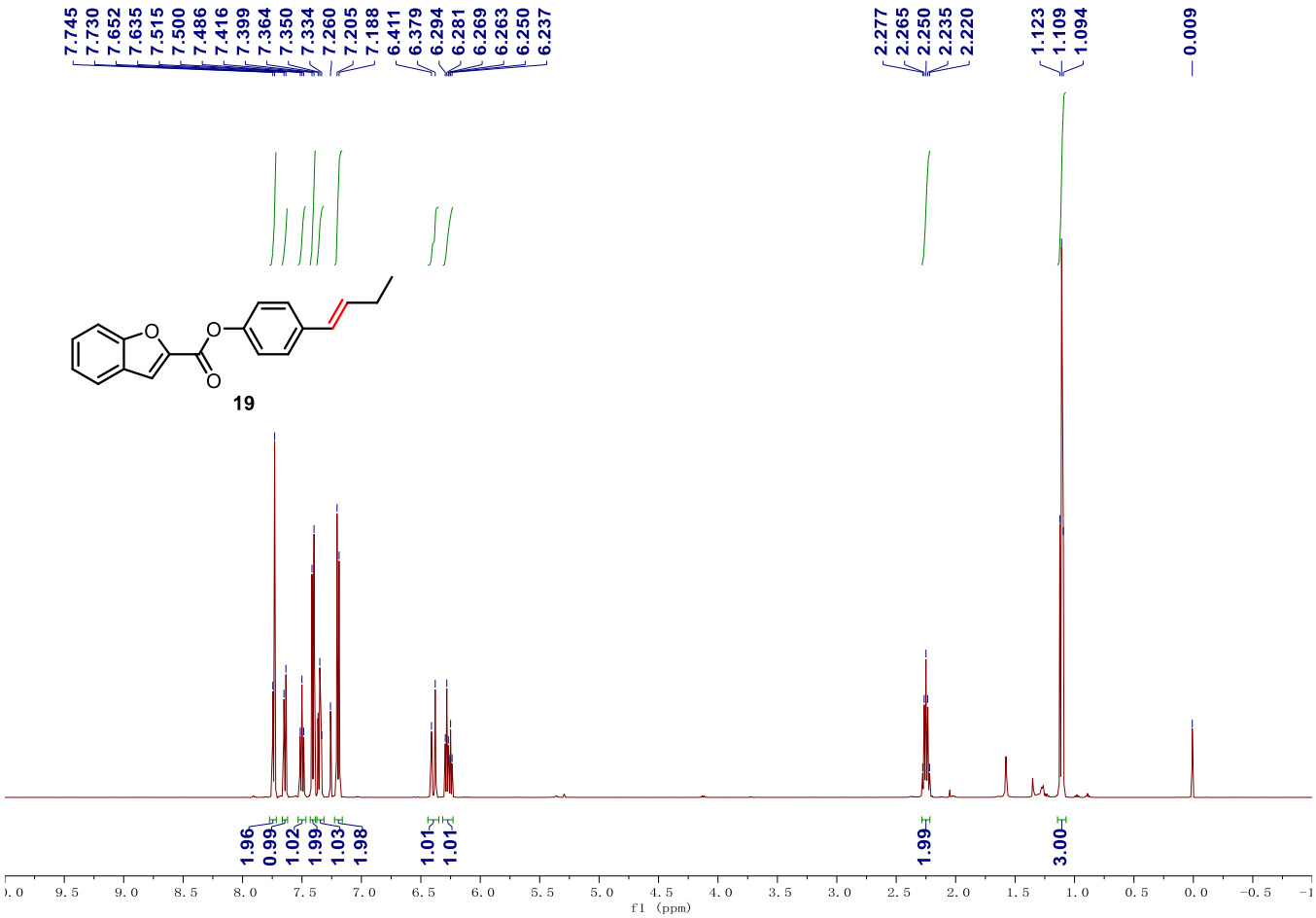
151.67
141.31
135.72
133.17
124.88
122.74
77.30
77.05
76.79
32.99
31.69
28.89
28.87
26.50
22.60
14.10
O
S
18
f1 (ppm)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7.730
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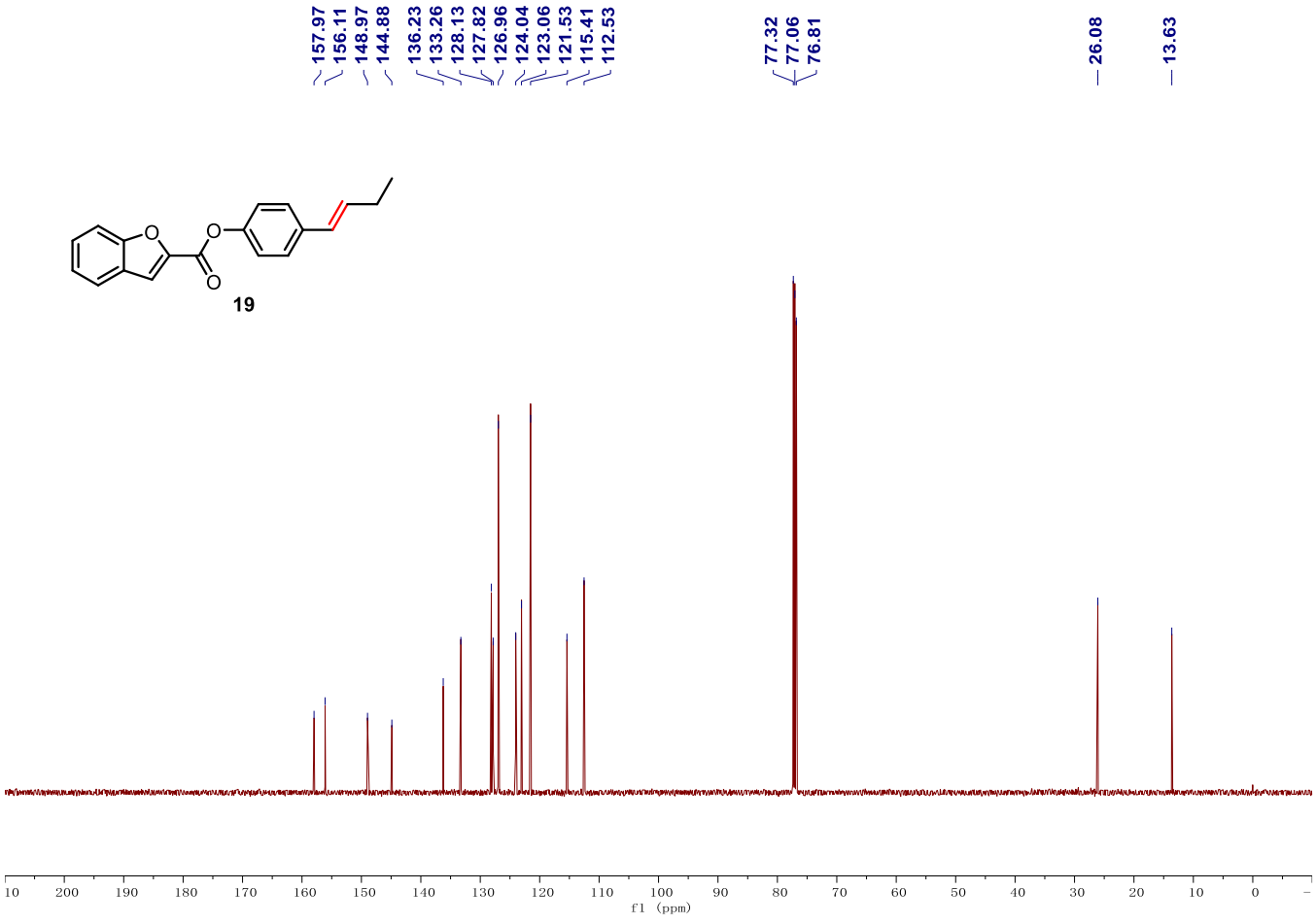
7.635
7.515
7.500
7.486
7.416
7.399
7.364
7.350
7.334
7.260
7.205
7.188
6.411
6.379
6.294
6.281
6.269
6.263
6.250
6.237
2.277
2.265
2.250
2.235
2.220
1.123
1.109
1.094
0.009
19
1.96
0.99
1.02
1.99
1.03
1.98
1.01
1.01
1.99
3.00
f1 (ppm)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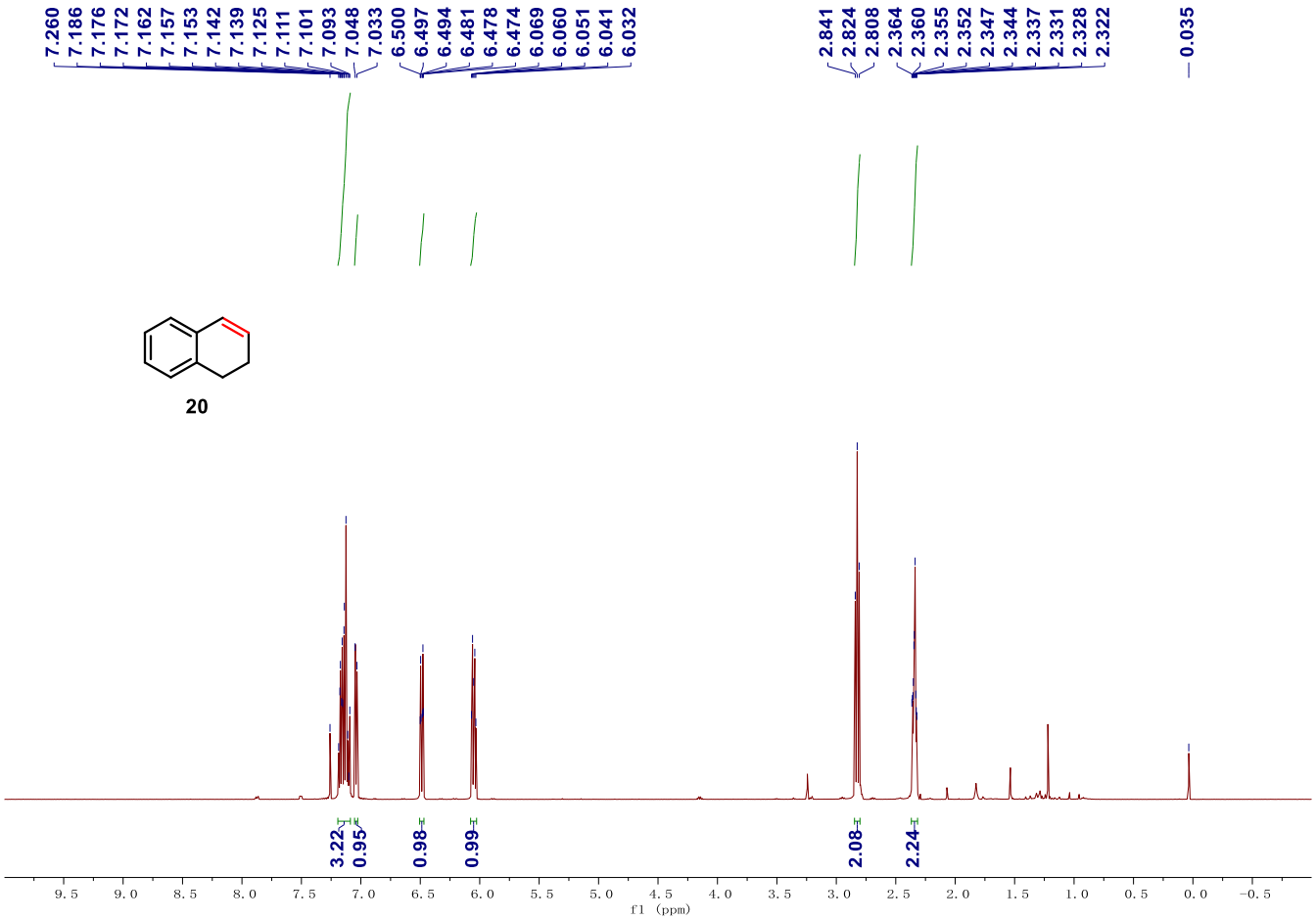
156.11
148.97
144.88
136.23
133.26
128.13
127.82
126.96
124.04
123.06
121.53
115.41
112.53
77.32
77.06
76.81
26.08
13.63
19
f1 (ppm)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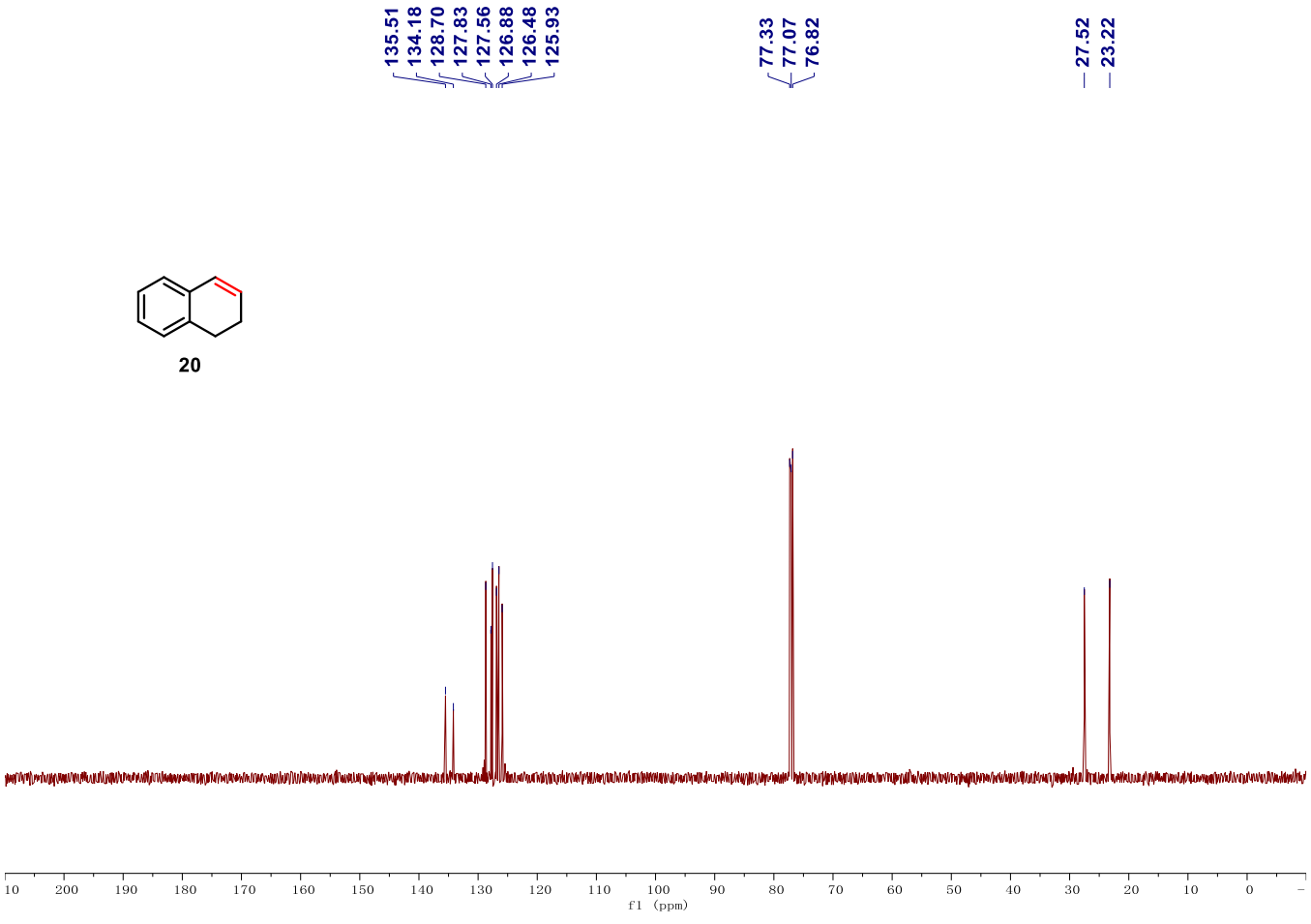
7.157
7.153
7.142
7.139
7.125
7.111
7.101
7.093
7.048
7.033
6.500
6.497
6.494
6.481
6.478
6.474
6.069
6.060
6.051
6.041
6.032
2.841
2.824
2.808
2.364
2.360
2.355
2.352
2.347
2.344
2.337
2.331
2.328
2.322
0.035
20
3.22
0.95
0.98
0.99
2.08
2.24
f1 (ppm)


135.51
134.18
128.70
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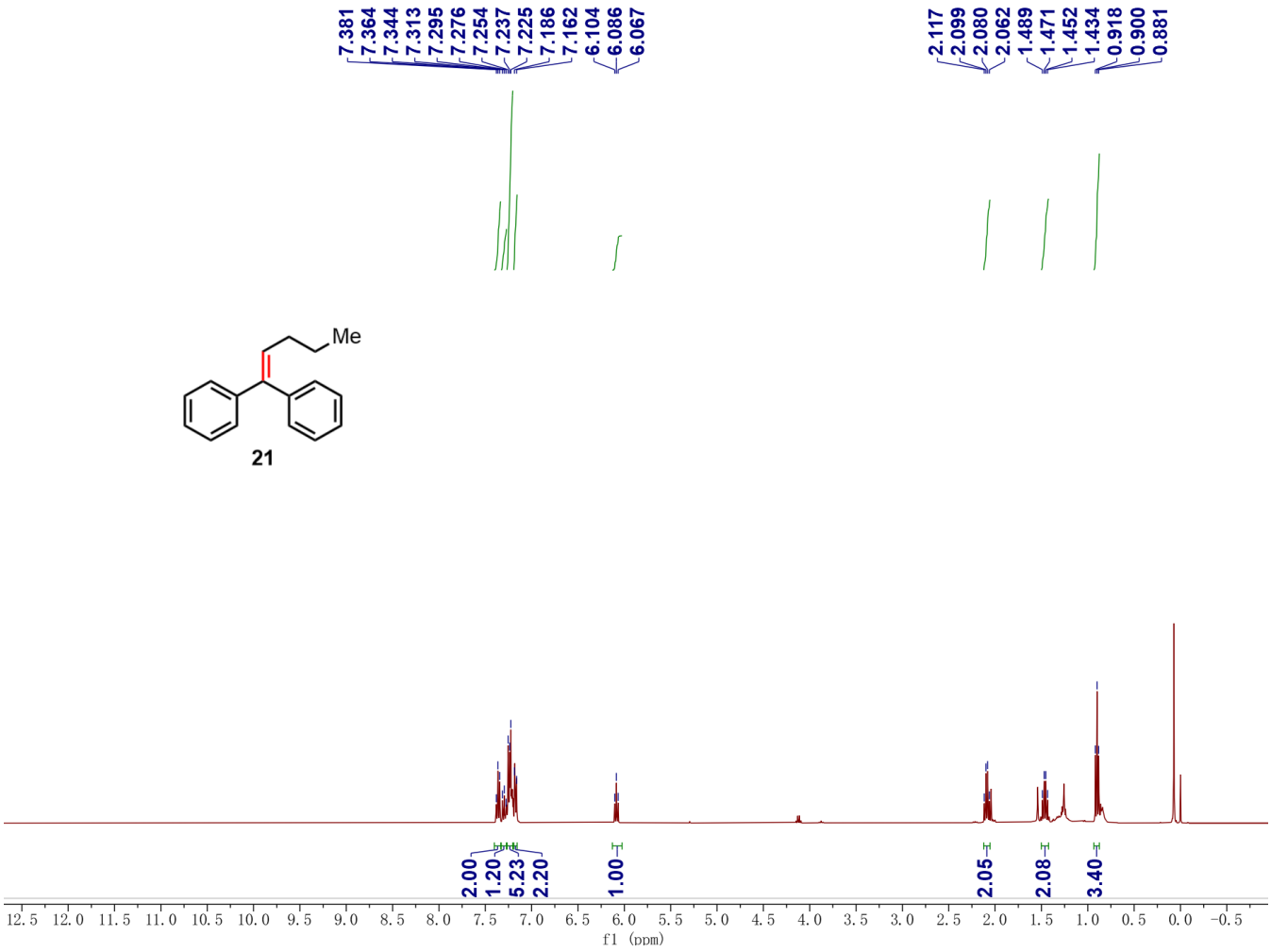
127.56
126.88
126.48
125.93
77.33
77.07
76.82
27.52
23.22
20
f1 (ppm)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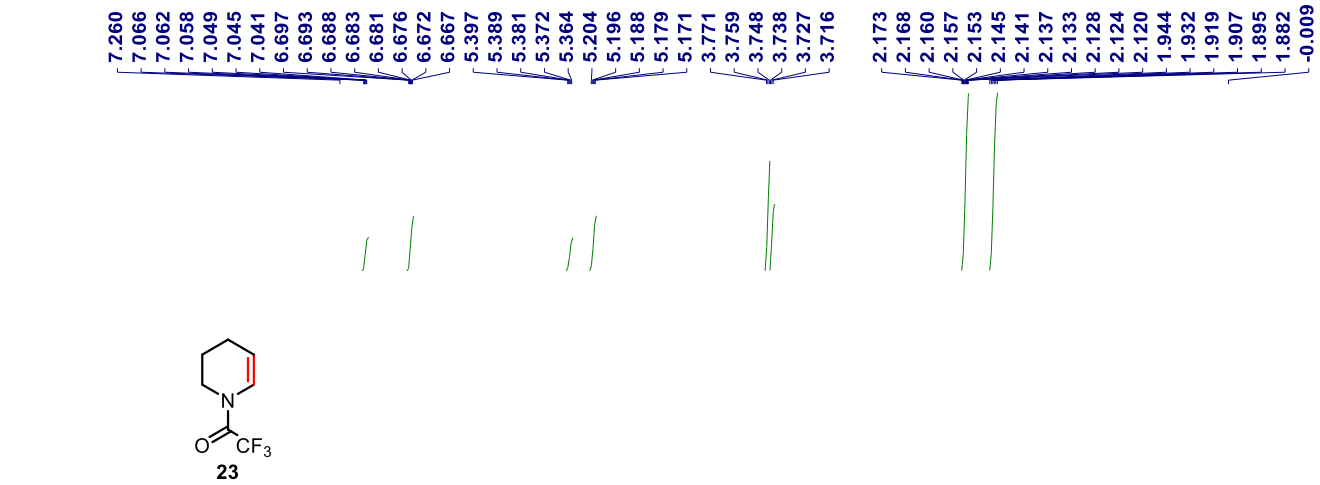
7.364
7.344
7.313
7.295
7.276
7.254
7.237
7.225
7.186
7.162
6.104
6.086
6.067
2.117
2.099
2.080
2.062
1.489
1.471
1.452
1.434
0.918
0.900
0.881
Me
21
2.00
1.20
5.23
2.20
1.00
2.05
2.08
3.40
f1 (ppm)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7.062
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7.049
7.045
7.041
6.697
6.693
6.688
6.683
6.681
6.676
6.672
6.667
5.397
5.389
5.381
5.372
5.364
5.204
5.196
5.188
5.179
5.171
3.771
3.759
3.748
3.738
3.727
3.716
2.173
2.168
2.160
2.157
2.153
2.145
2.141
2.137
2.133
2.128
2.124
2.120
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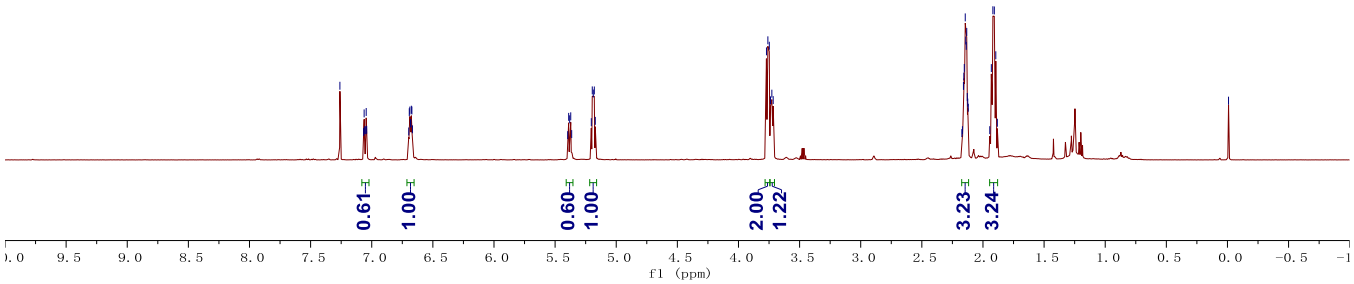
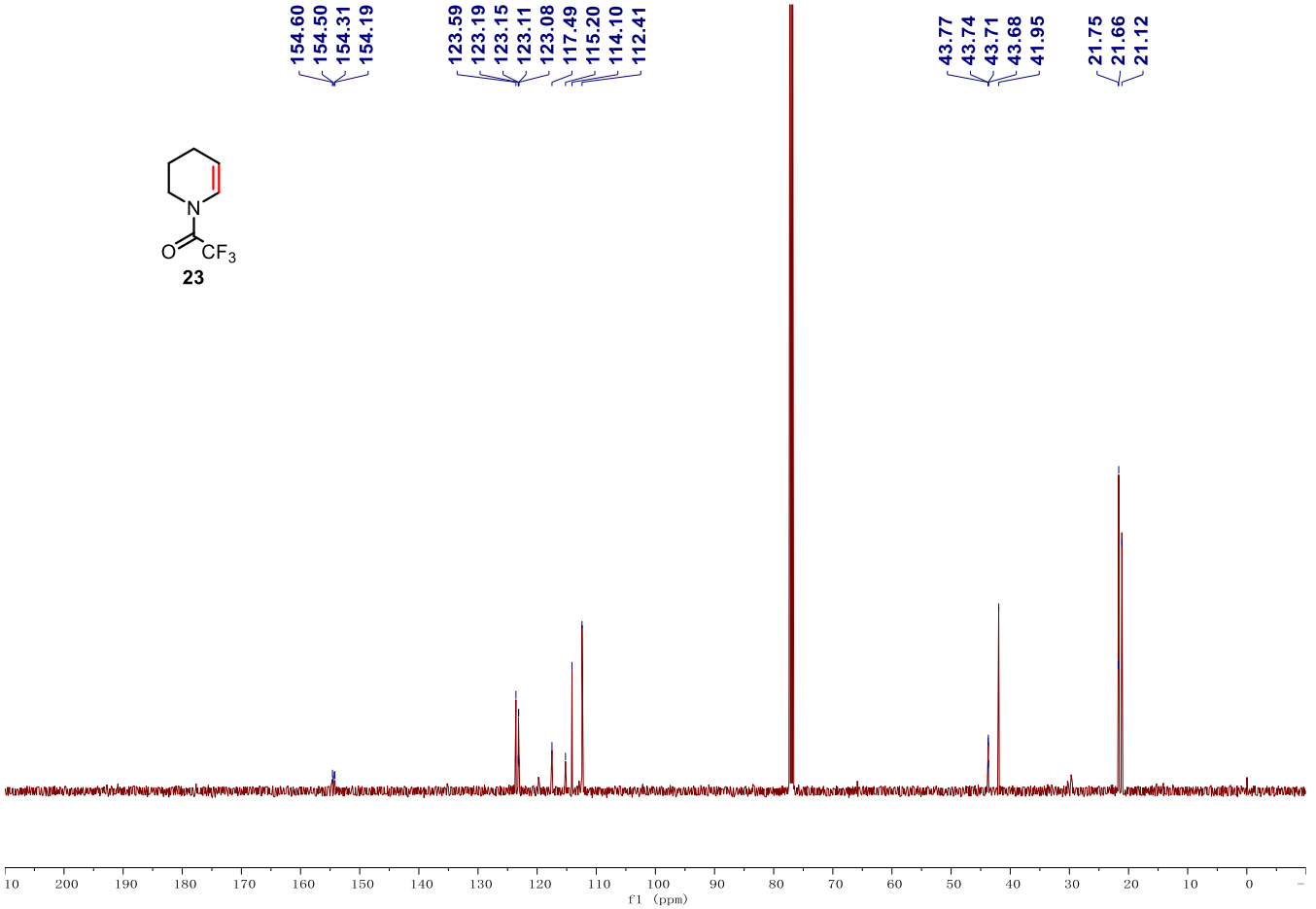
1.932
1.919
1.907
1.895
1.882
-0.009

N
O
CF3

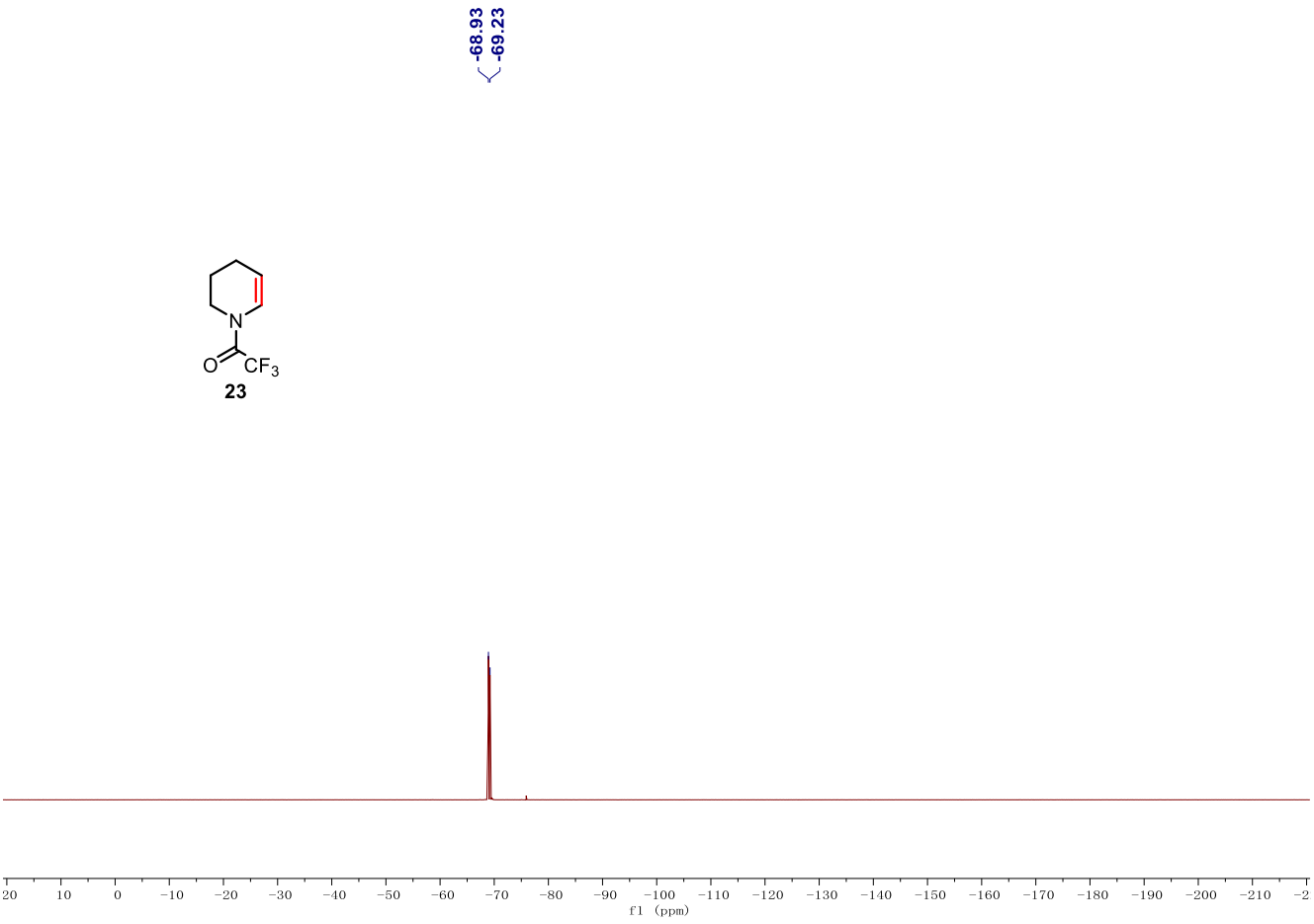
23

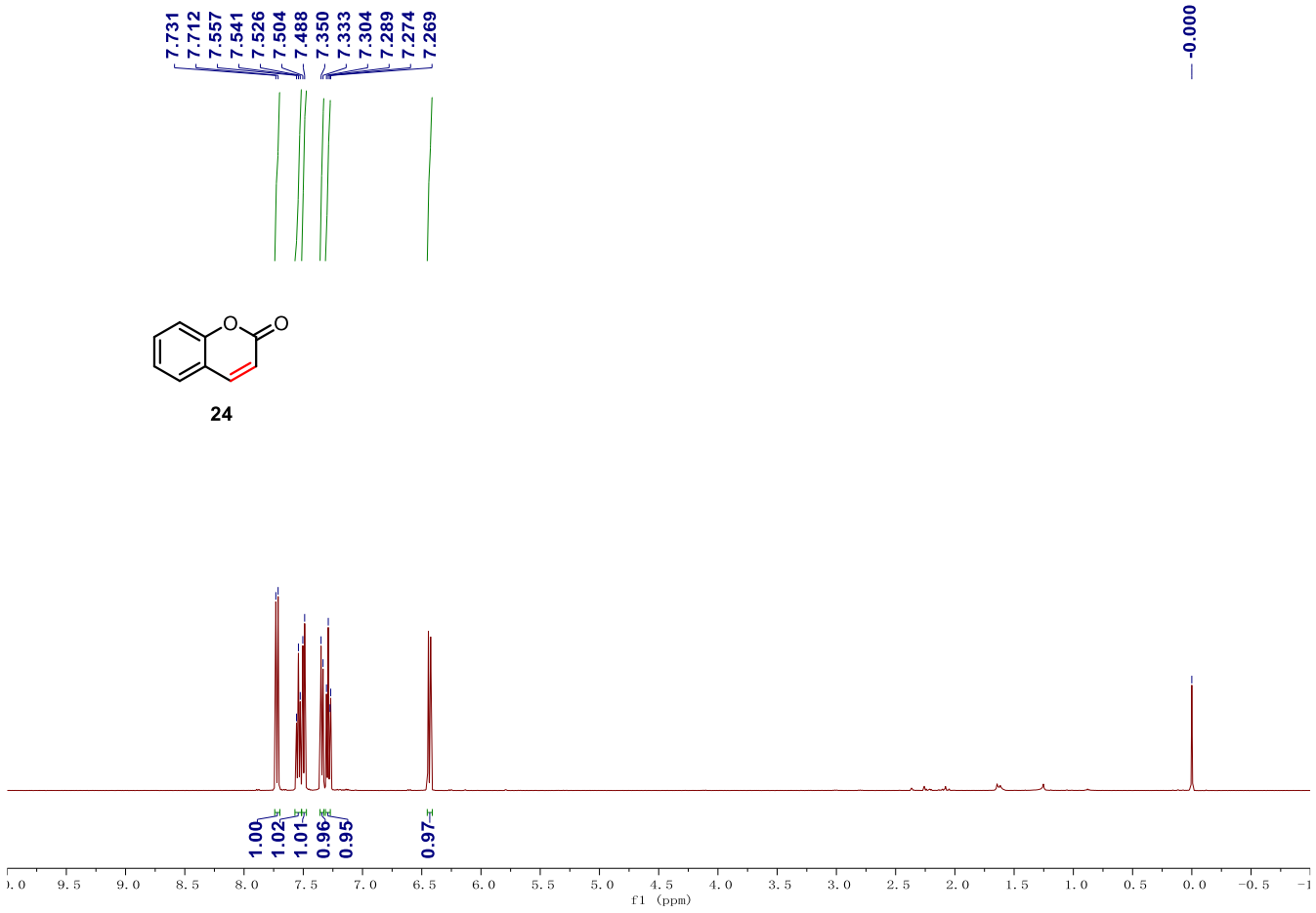

0.61
1.00
0.60
1.00
2.00
1.22
3.23
3.24
f1 (ppm)


154.60
154.50
154.31
154.19
123.59
123.19
123.15
123.11
123.08
117.49
115.20
114.10
112.41
43.77
43.74
43.71
43.68
41.95
21.75
21.66
21.12
f1 (ppm)

N
O
CF3

23


-68.93
-69.23
N
O
CF3
23
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)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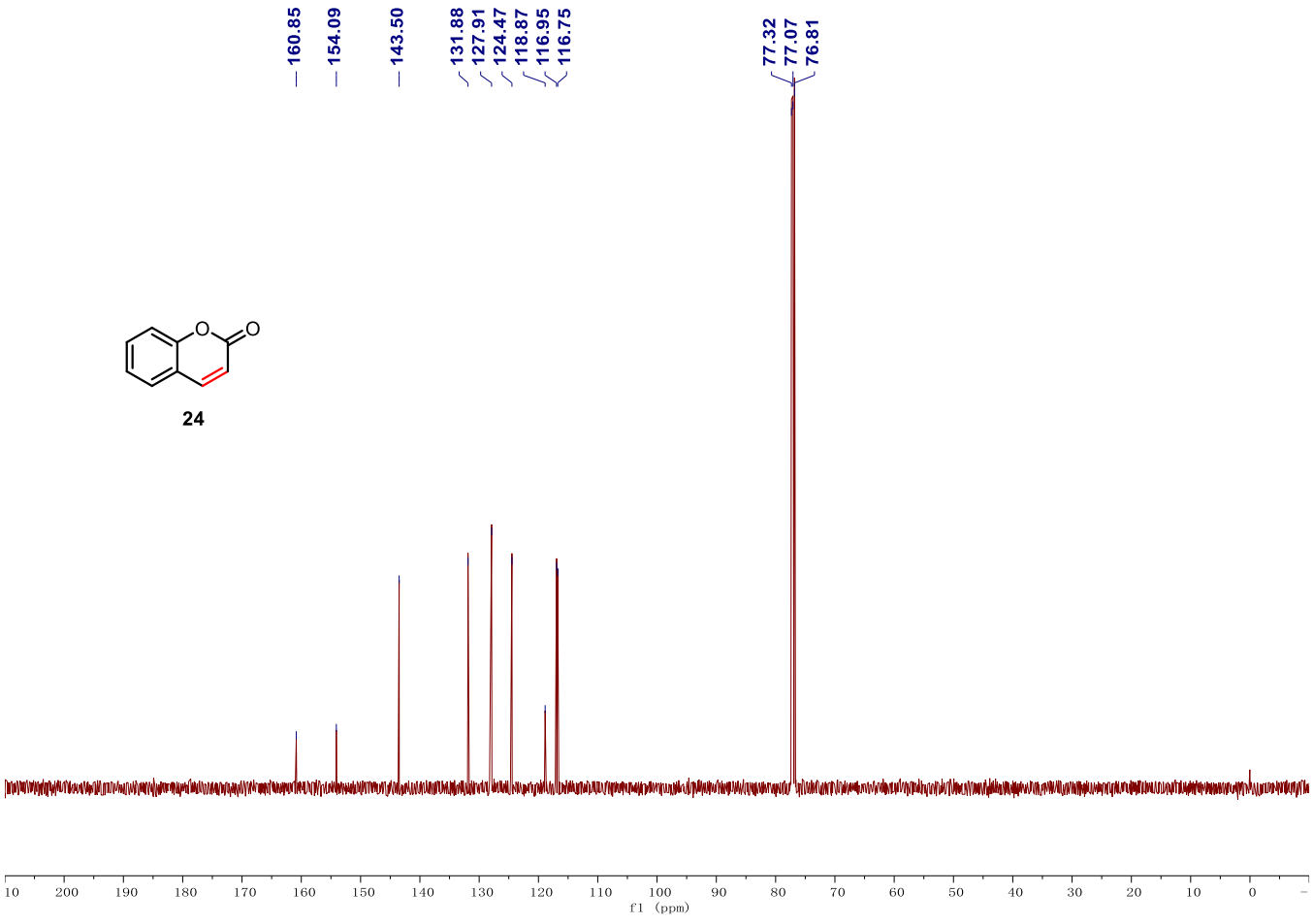
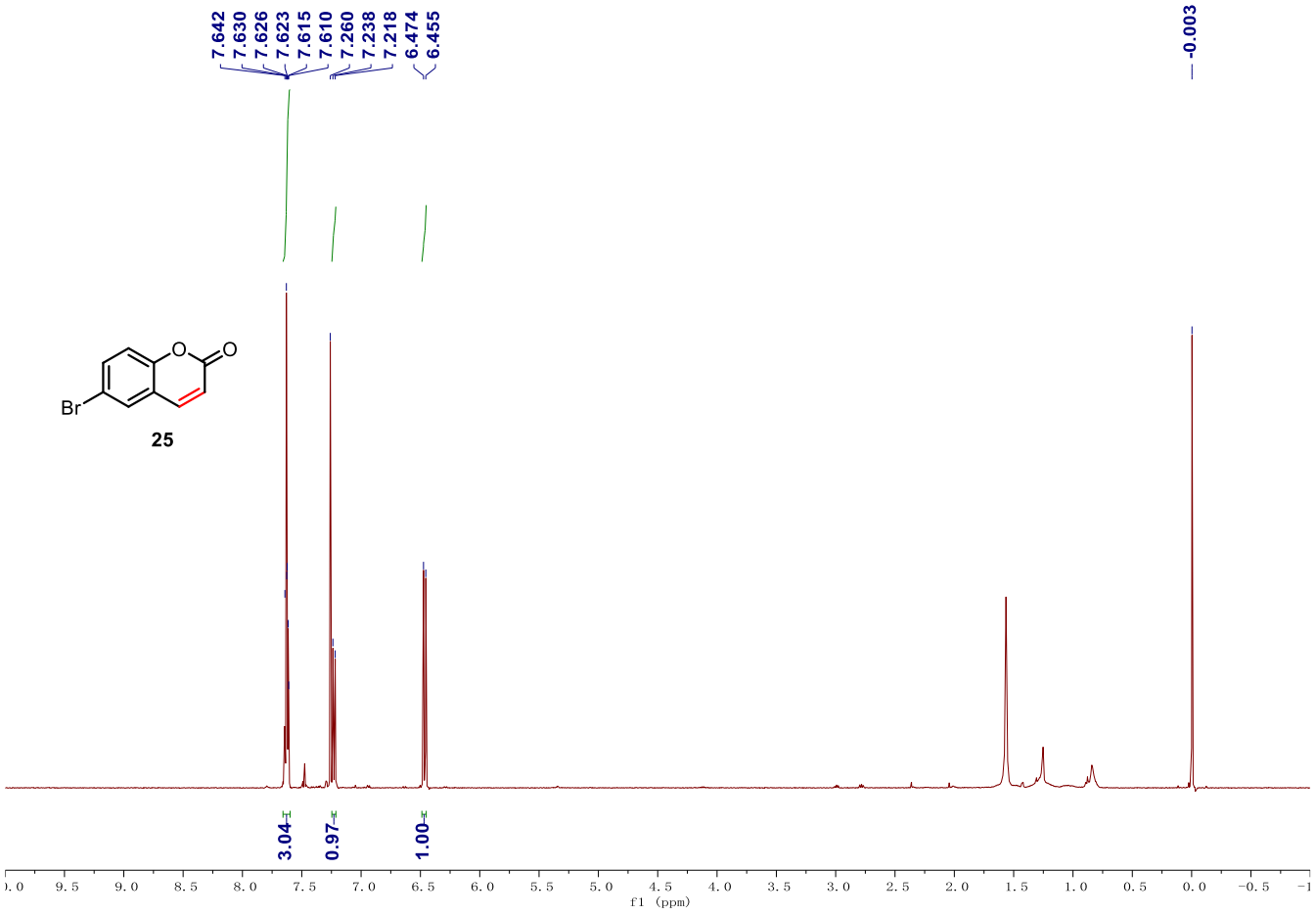
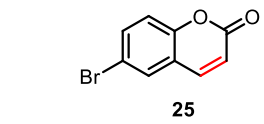
7.712
7.557
7.541
7.526
7.504
7.488
7.350
7.333
7.304
7.289
7.274
7.269
-0.000
24
1.00
1.02
1.01
0.96
0.95
0.97
f1 (ppm)


160.85
154.09
143.50
131.88
127.91
124.47
118.87
116.95
116.75
77.32
77.07
76.81
24
f1 (ppm)


7.642
7.630
7.626
7.623
7.615
7.610
7.260
7.238
7.218
6.474
6.455
-0.003
3.04
0.97
1.00
f1 (ppm)

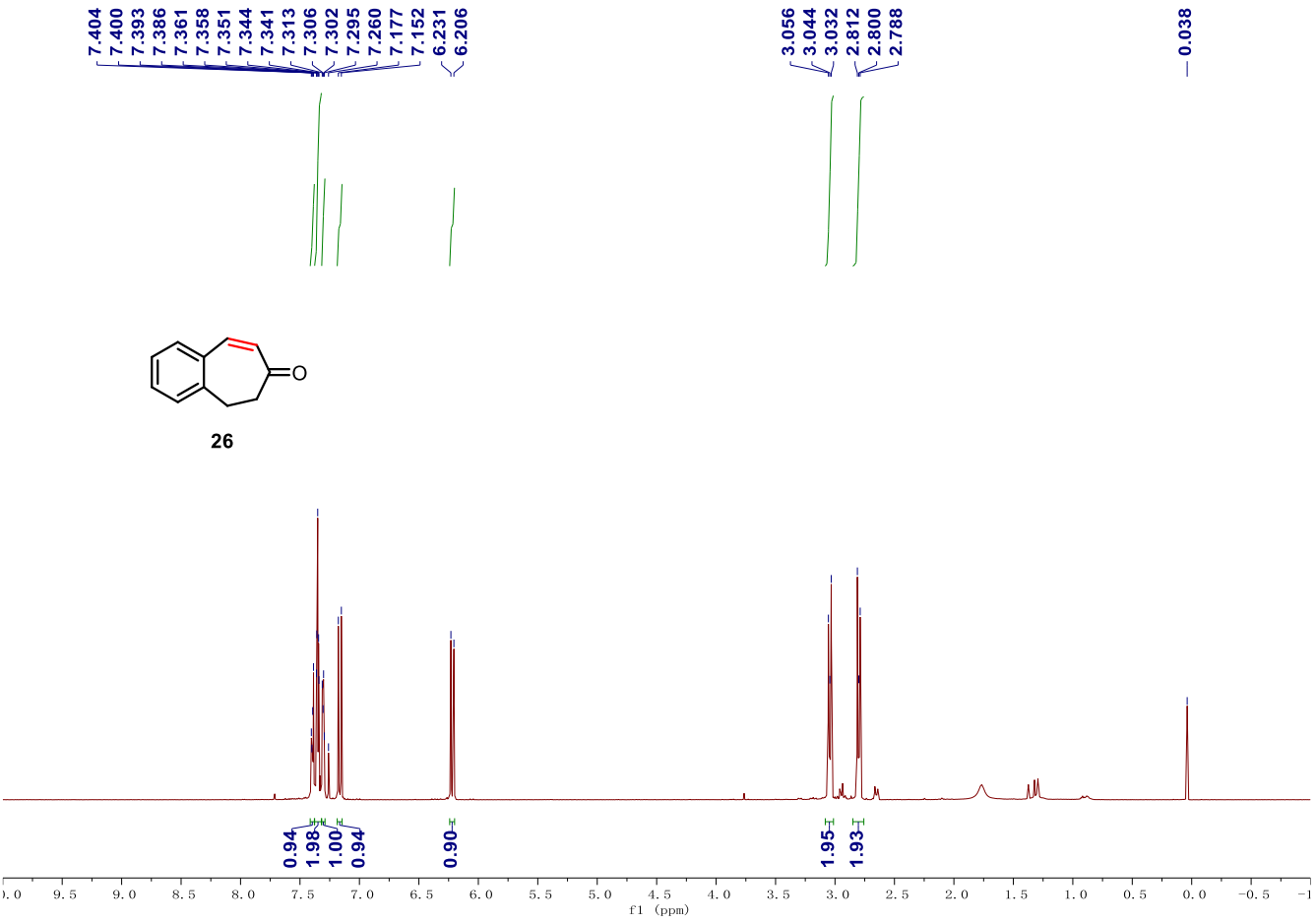

O
O
Br

25

159.98
152.97
142.11
134.63
130.20
120.35
118.69
117.91
117.02
77.29
77.03
76.78
f1 (ppm)

O
O
Br

25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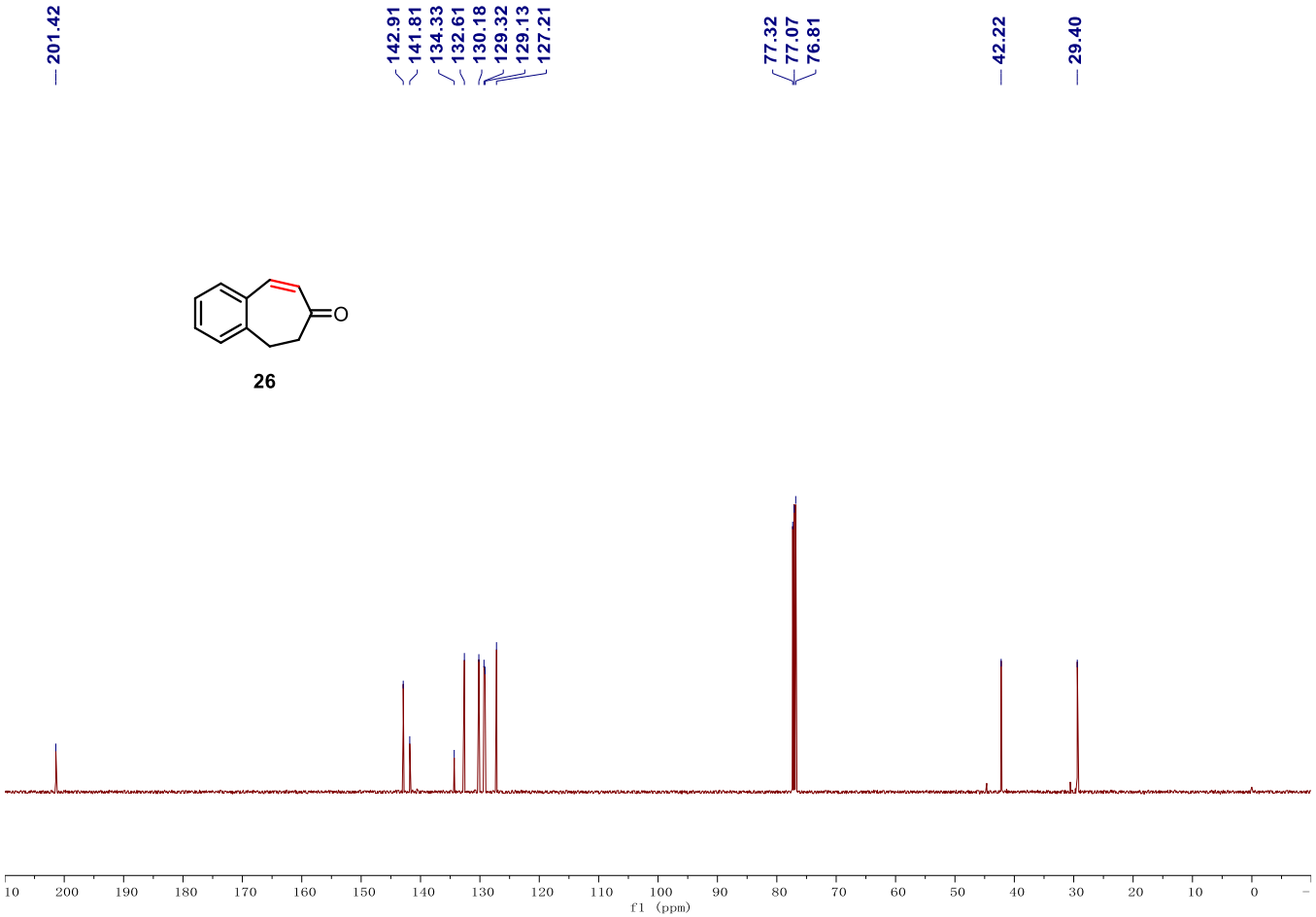
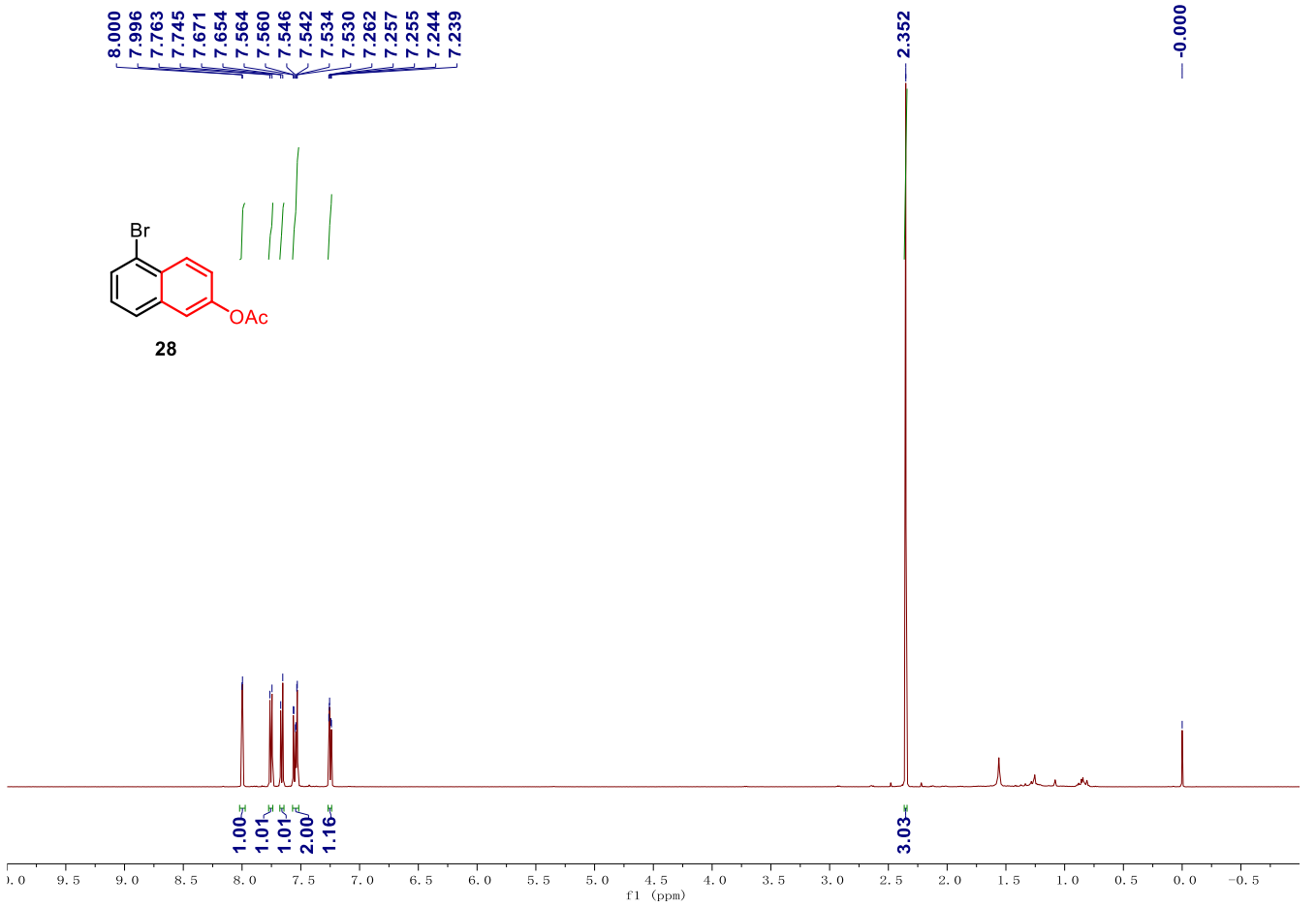
7.400
7.393
7.386
7.361
7.358
7.351
7.344
7.341
7.313
7.306
7.302
7.295
7.260
7.177
7.152
6.231
6.206
3.056
3.044
3.032
2.812
2.800
2.788
0.038
O
26
0.94
1.98
1.00
0.94
0.90
1.95
1.93
f1 (ppm)


201.42
142.91
141.81
134.33
132.61
130.18
129.32
129.13
127.21
77.32
77.07
76.81
42.22
29.40
O
26
f1 (ppm)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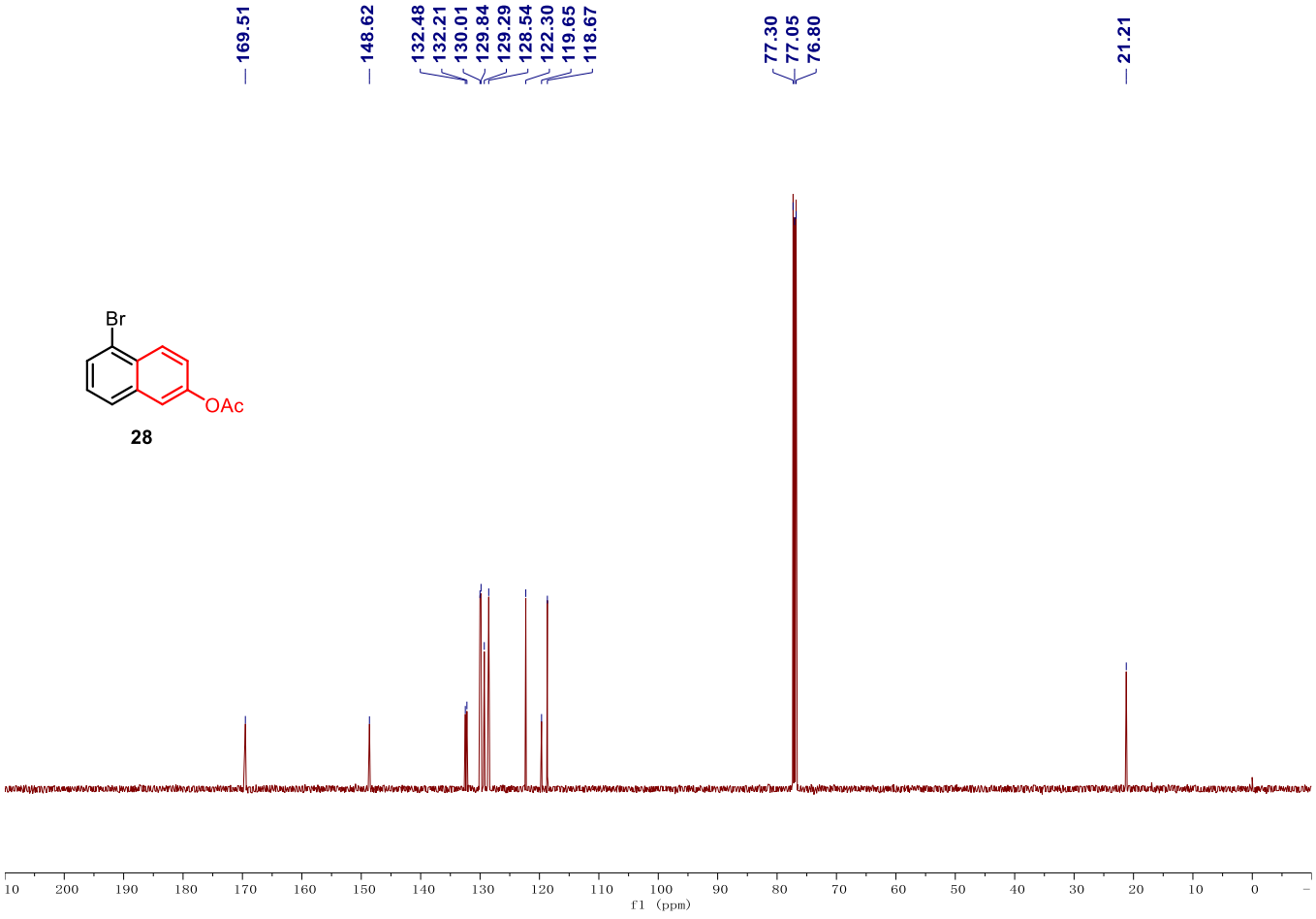
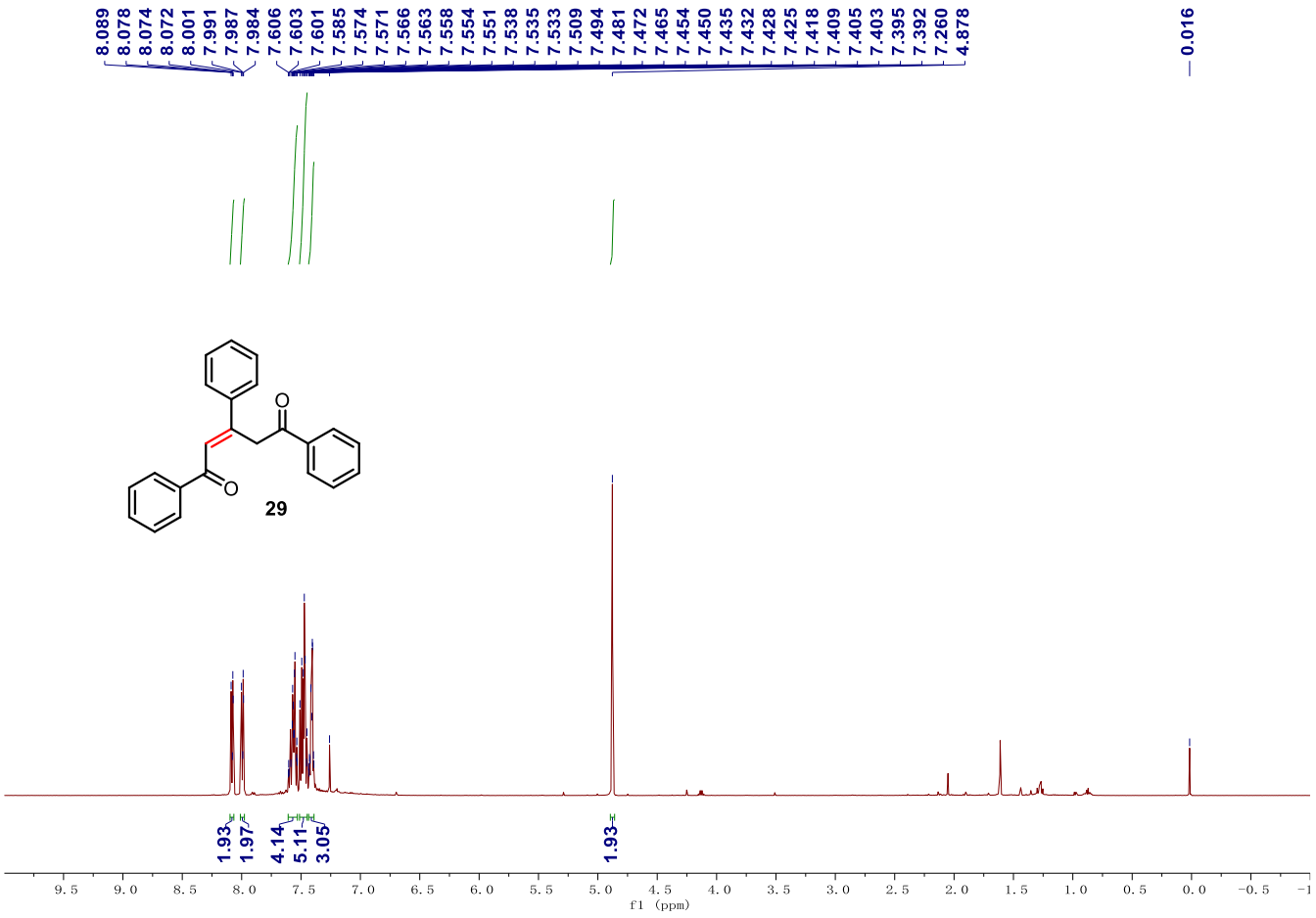
7.763
7.745
7.671
7.654
7.564
7.560
7.546
7.542
7.534
7.530
7.262
7.257
7.255
7.244
7.239
2.352
-0.000
Br
OAc
28
1.00
1.01
1.01
2.00
1.16
3.03
f1 (ppm)


169.51
148.62
132.48
132.21
130.01
129.84
129.29
128.54
122.30
119.65
118.67
77.30
77.05
76.80
21.21
Br
OAc
28
f1 (ppm)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8.074
8.072
8.001
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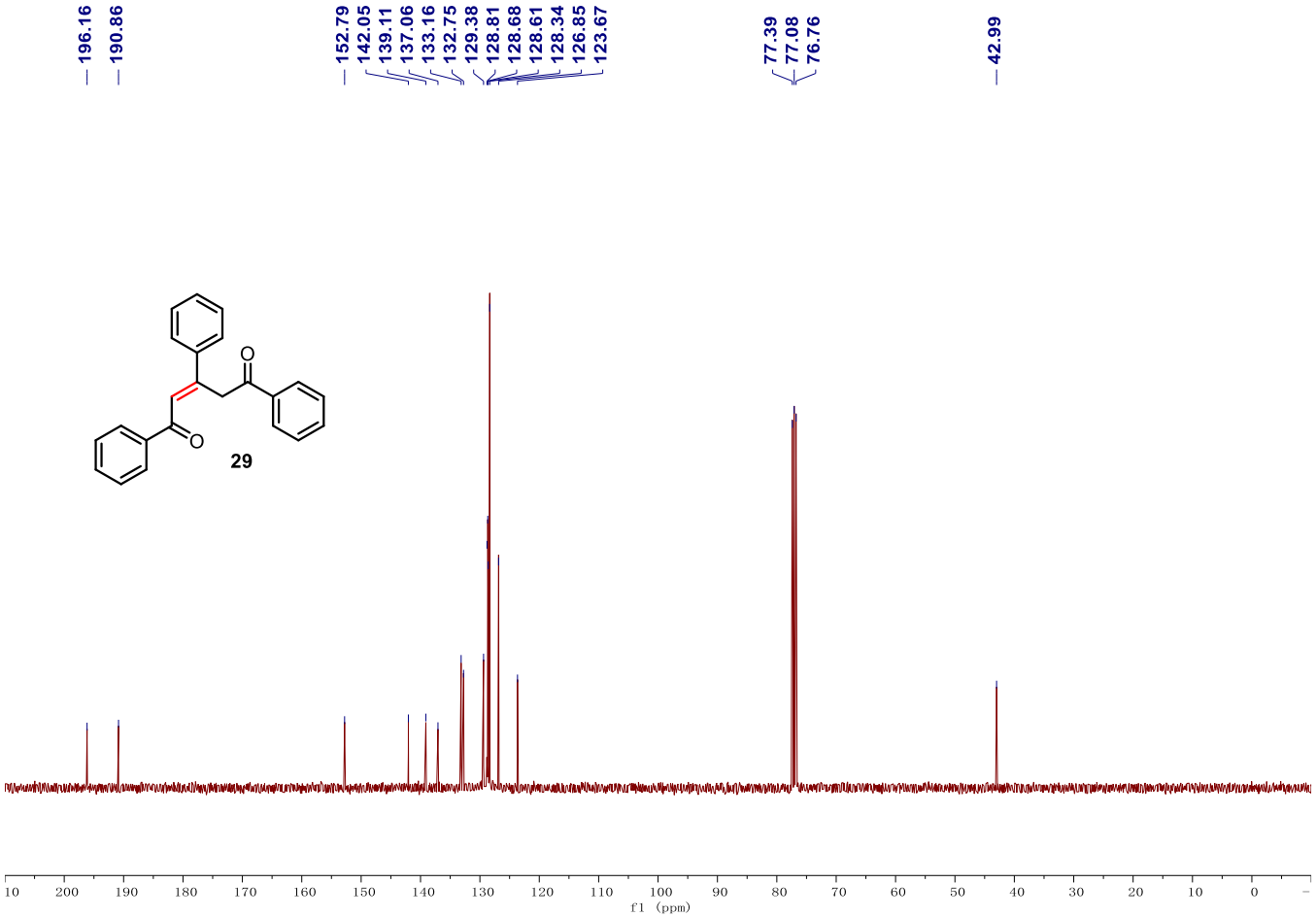
7.987
7.984
7.606
7.603
7.601
7.585
7.574
7.571
7.566
7.563
7.558
7.554
7.551
7.538
7.535
7.533
7.509
7.494
7.481
7.472
7.465
7.454
7.450
7.435
7.432
7.428
7.425
7.418
7.409
7.405
7.403
7.395
7.392
7.260
4.878
0.016
O
O
29
1.93
1.97
4.14
5.11
3.05
1.93
f1 (ppm)


196.16
190.86
152.79
142.05
139.11
137.06
133.16
132.75
129.38
128.81
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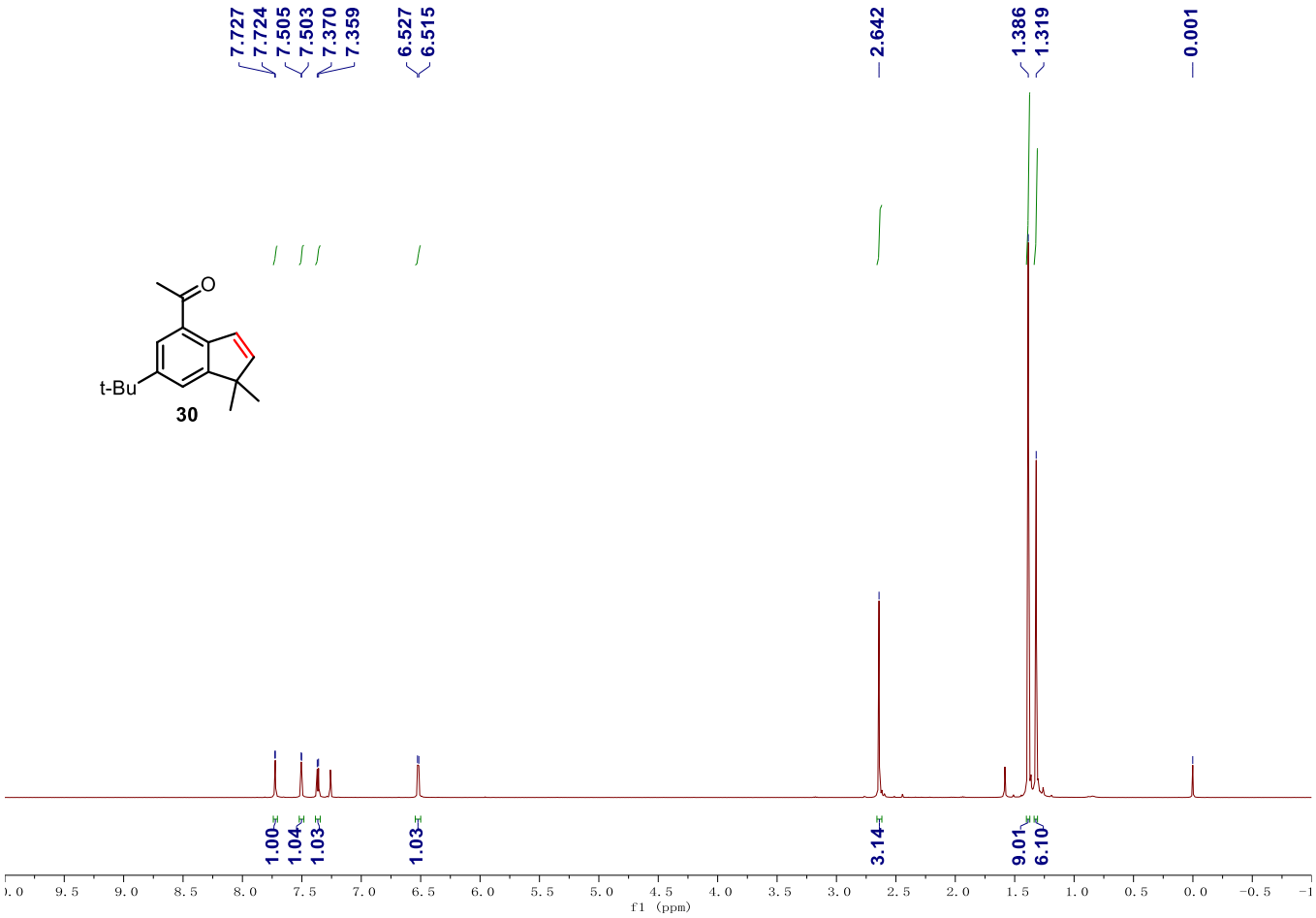
128.34
126.85
123.67
77.39
77.08
76.76
42.99
O
O
29
f1 (ppm)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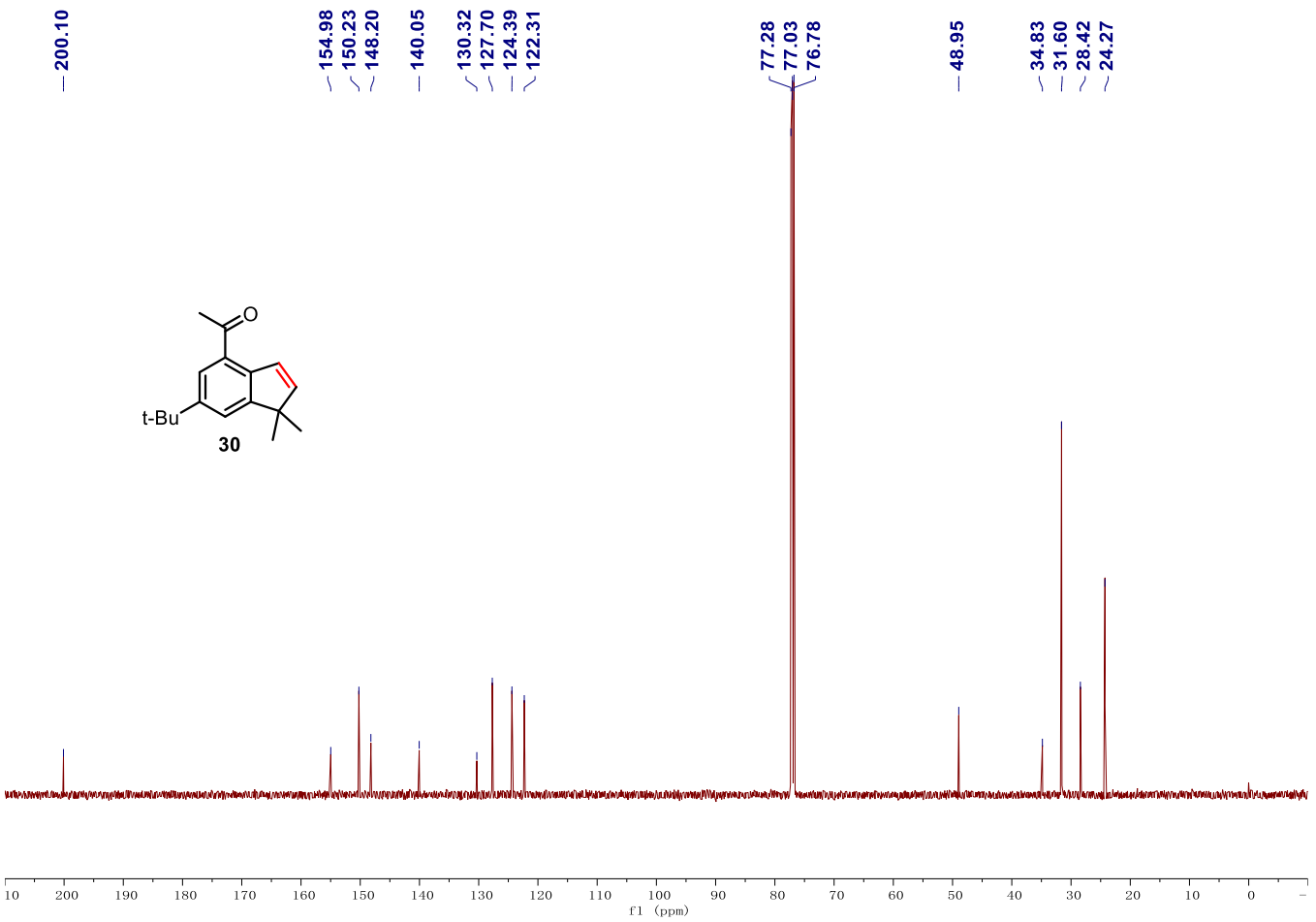
7.724
7.505
7.503
7.370
7.359
6.527
6.515
2.642
1.386
1.319
0.001
t-Bu
30
1.00
1.04
1.03
1.03
3.14
9.01
6.10
f1 (ppm)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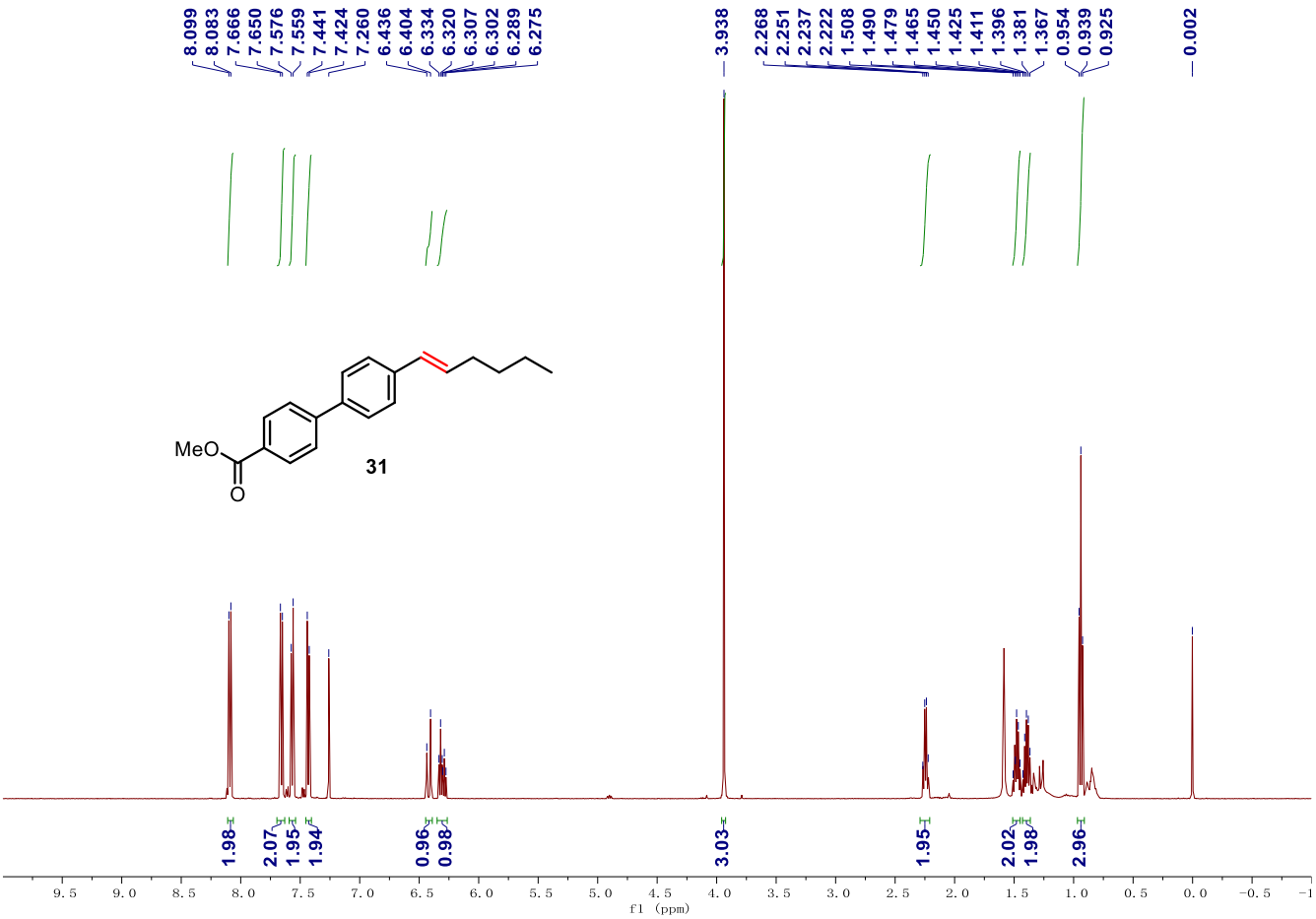
154.98
150.23
148.20
140.05
130.32
127.70
124.39
122.31
77.28
77.03
76.78
48.95
34.83
31.60
28.42
24.27
t-Bu
30
f1 (ppm)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8.083
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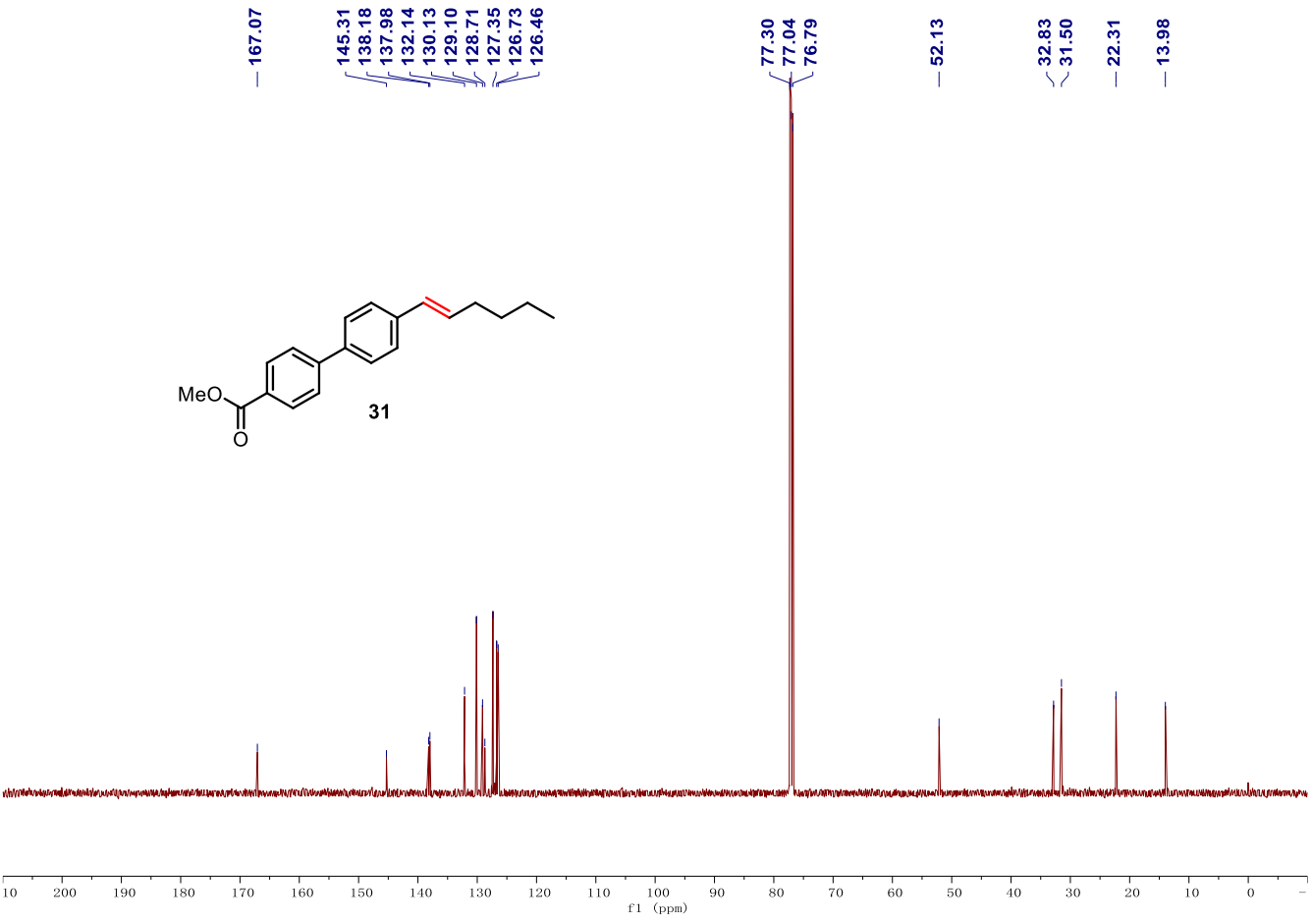
7.559
7.441
7.424
7.260
6.436
6.404
6.334
6.320
6.307
6.302
6.289
6.275
3.938
2.268
2.251
2.237
2.222
1.508
1.490
1.479
1.465
1.450
1.425
1.411
1.396
1.381
1.367
0.954
0.939
0.925
0.002
MeO
O
31
1.98
2.07
1.95
1.94
0.96
0.98
3.03
1.95
2.02
1.98
2.96
f1 (ppm)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138.18
137.98
132.14
130.13
129.10
128.71
127.35
126.73
126.46
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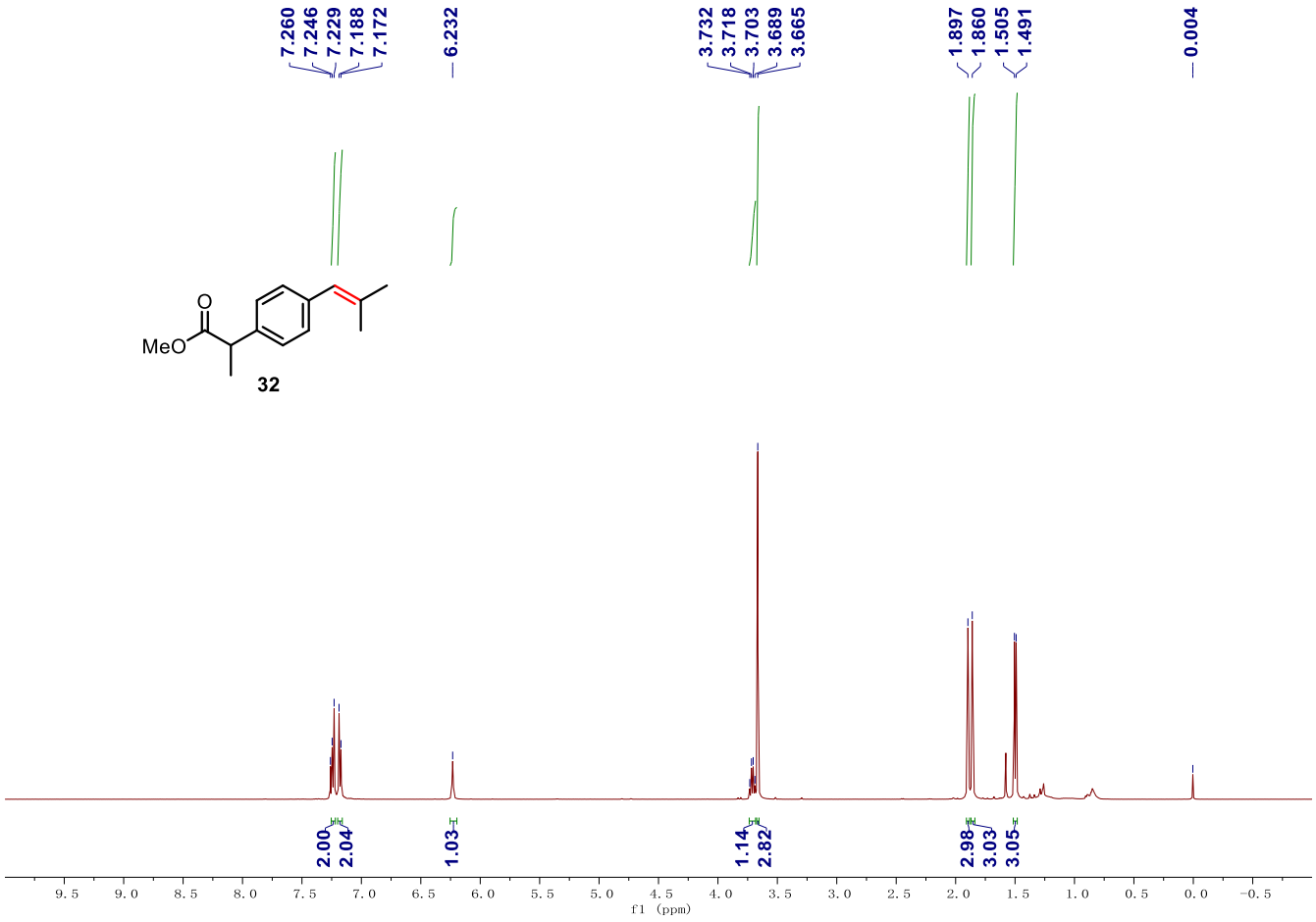
77.04
76.79
52.13
32.83
31.50
22.31
13.98
MeO
O
31
f1 (ppm)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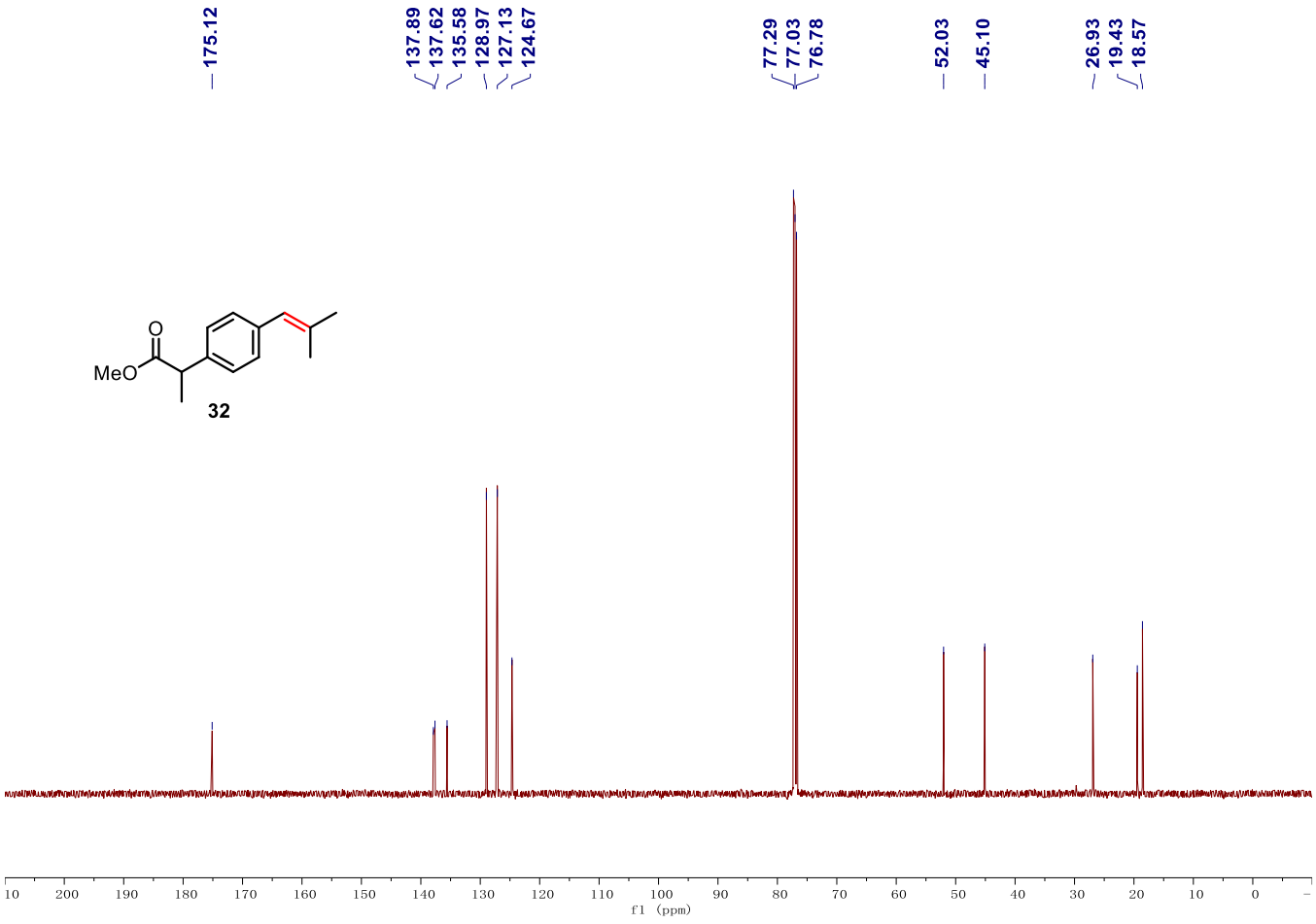
7.246
7.229
7.188
7.172
6.232
3.732
3.718
3.703
3.689
3.665
1.897
1.860
1.505
1.491
0.004
MeO
O
32
2.00
2.04
1.03
1.14
2.82
2.98
3.03
3.05
f1 (ppm)


175.12
137.89
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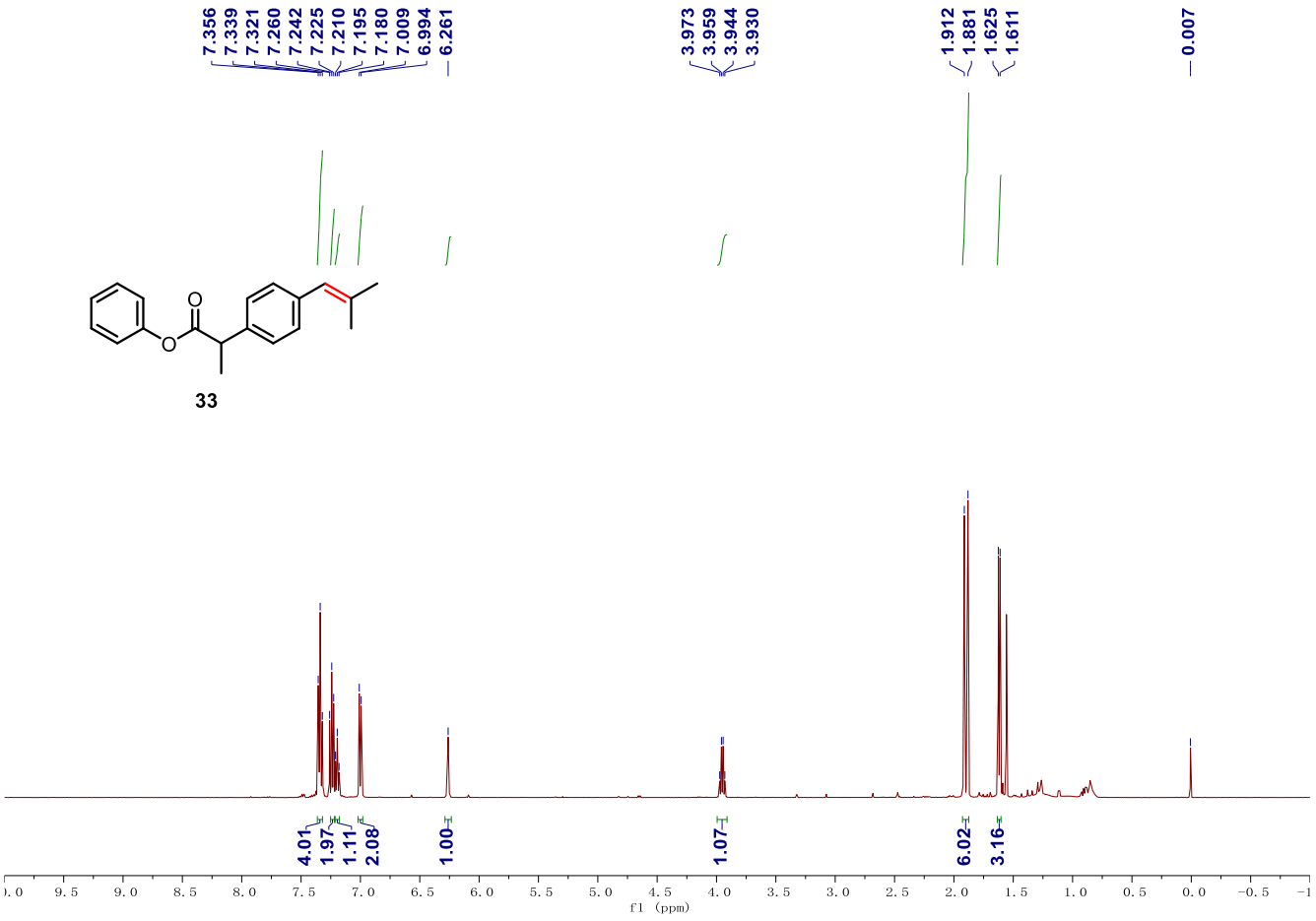
135.58
128.97
127.13
124.67
77.29
77.03
76.78
52.03
45.10
26.93
19.43
18.57
MeO
O
32
f1 (ppm)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6.994
6.261
3.973
3.959
3.944
3.930
1.912
1.881
1.625
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4.01
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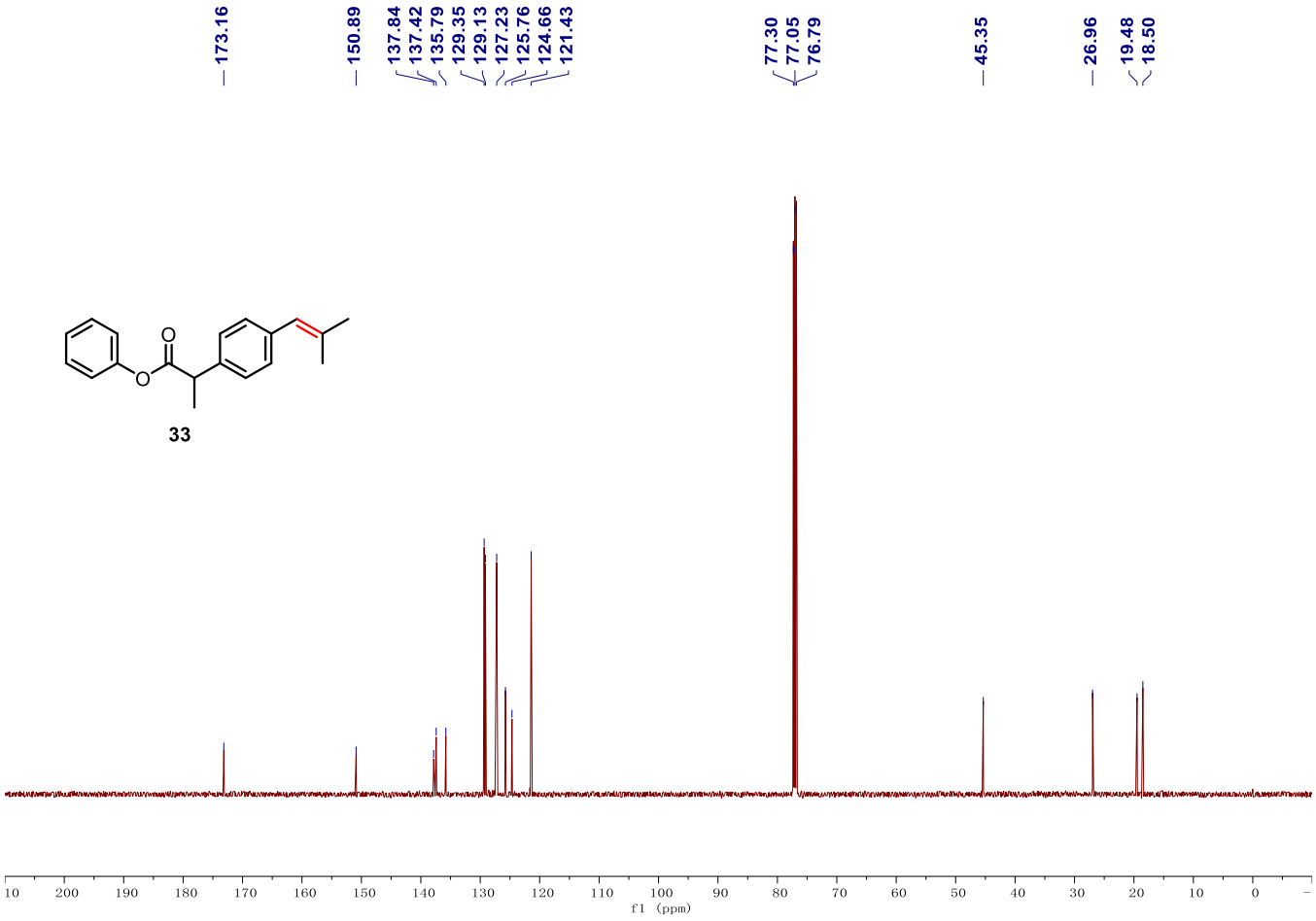
1.11
2.08
1.00
1.07
6.02
3.16
f1 (ppm)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129.35
129.13
127.23
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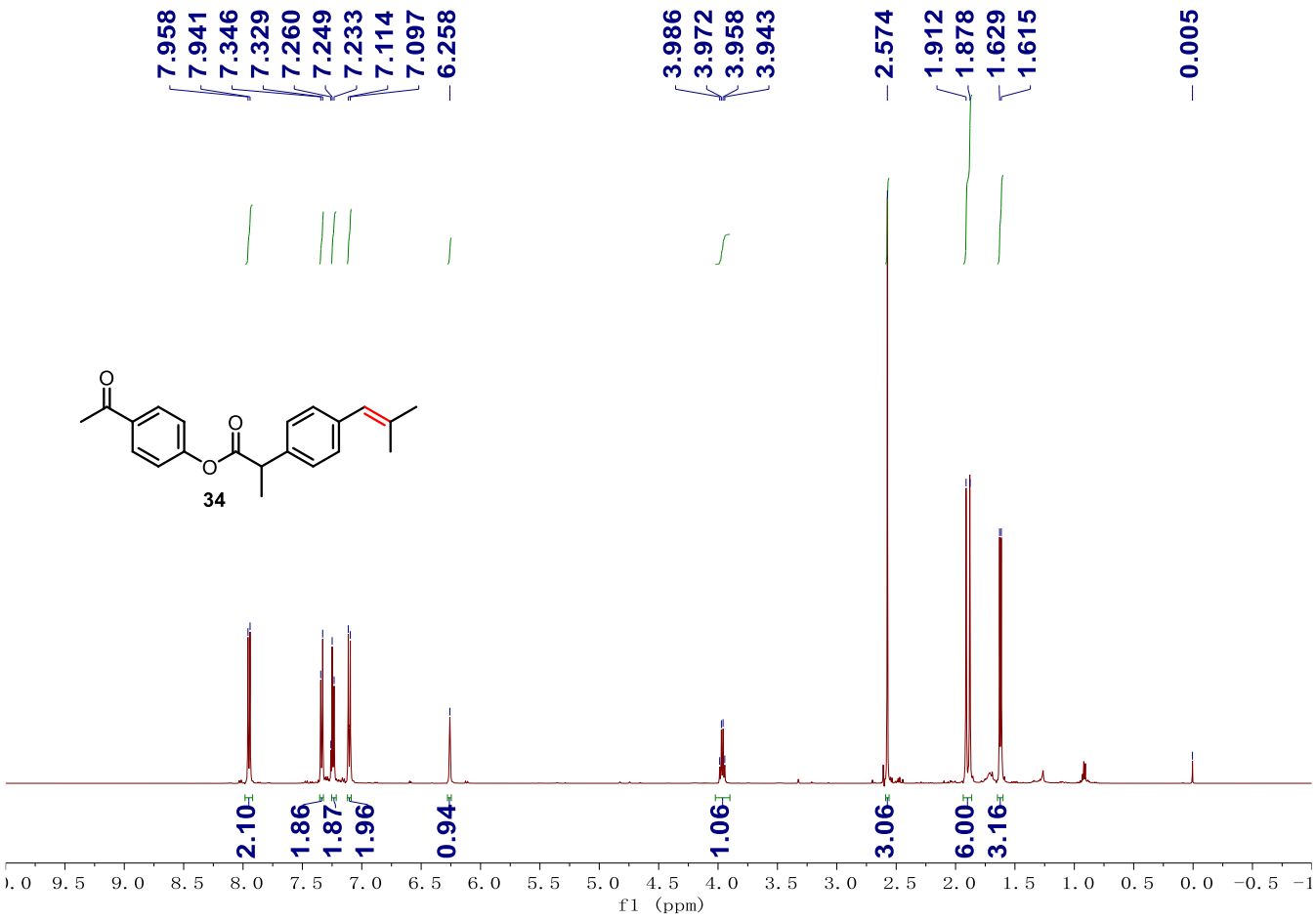
121.43
77.30
77.05
76.79
45.35
26.96
19.48
18.50
33
f1 (ppm)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7.346
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7.249
7.233
7.114
7.097
6.258
3.986
3.972
3.958
3.943
2.574
1.912
1.878
1.629
1.615
0.005
34
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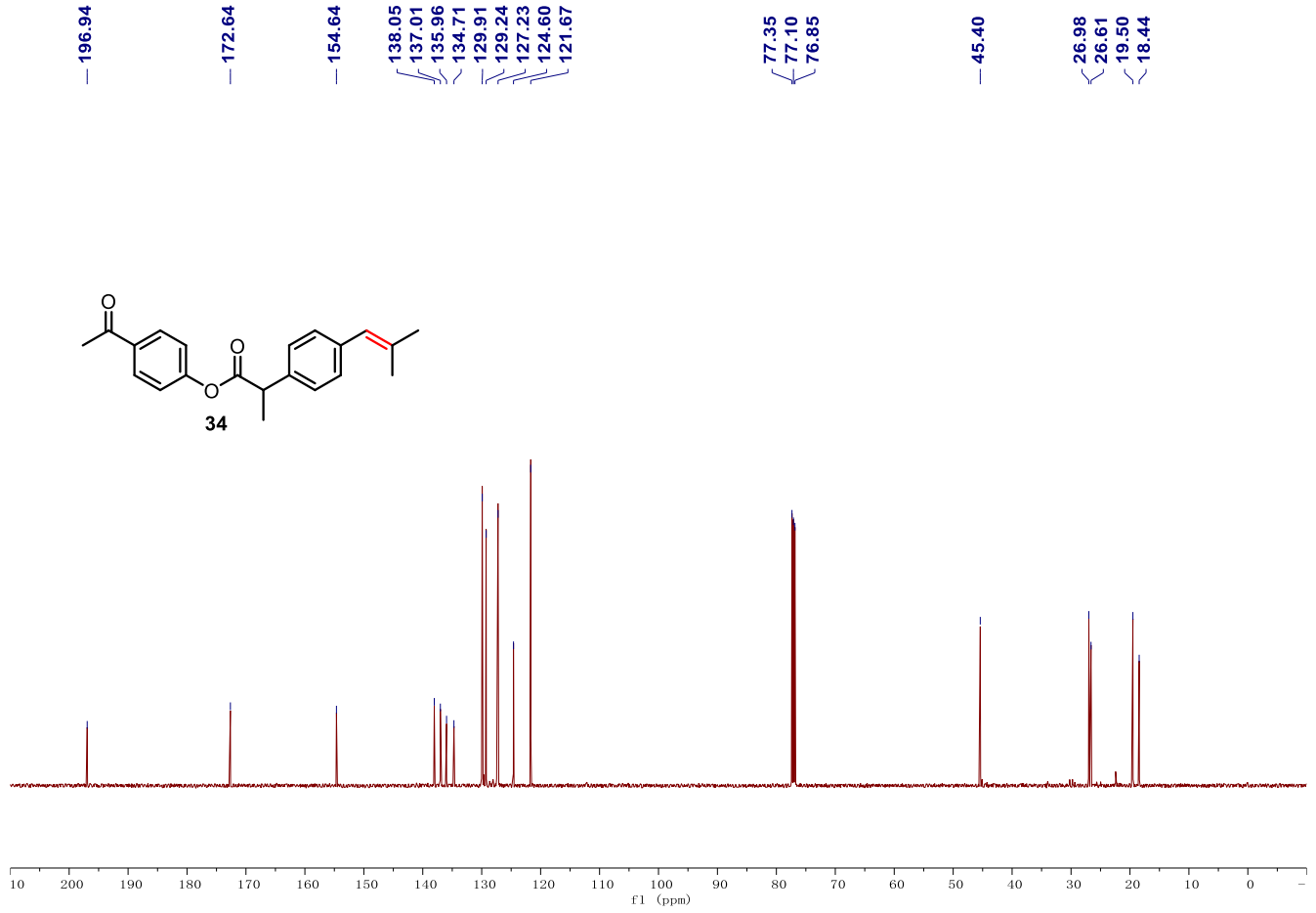
1.86
1.87
1.96
0.94
1.06
3.06
6.00
3.16
f1 (ppm)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154.64
138.05
137.01
135.96
134.71
129.91
129.24
127.23
124.60
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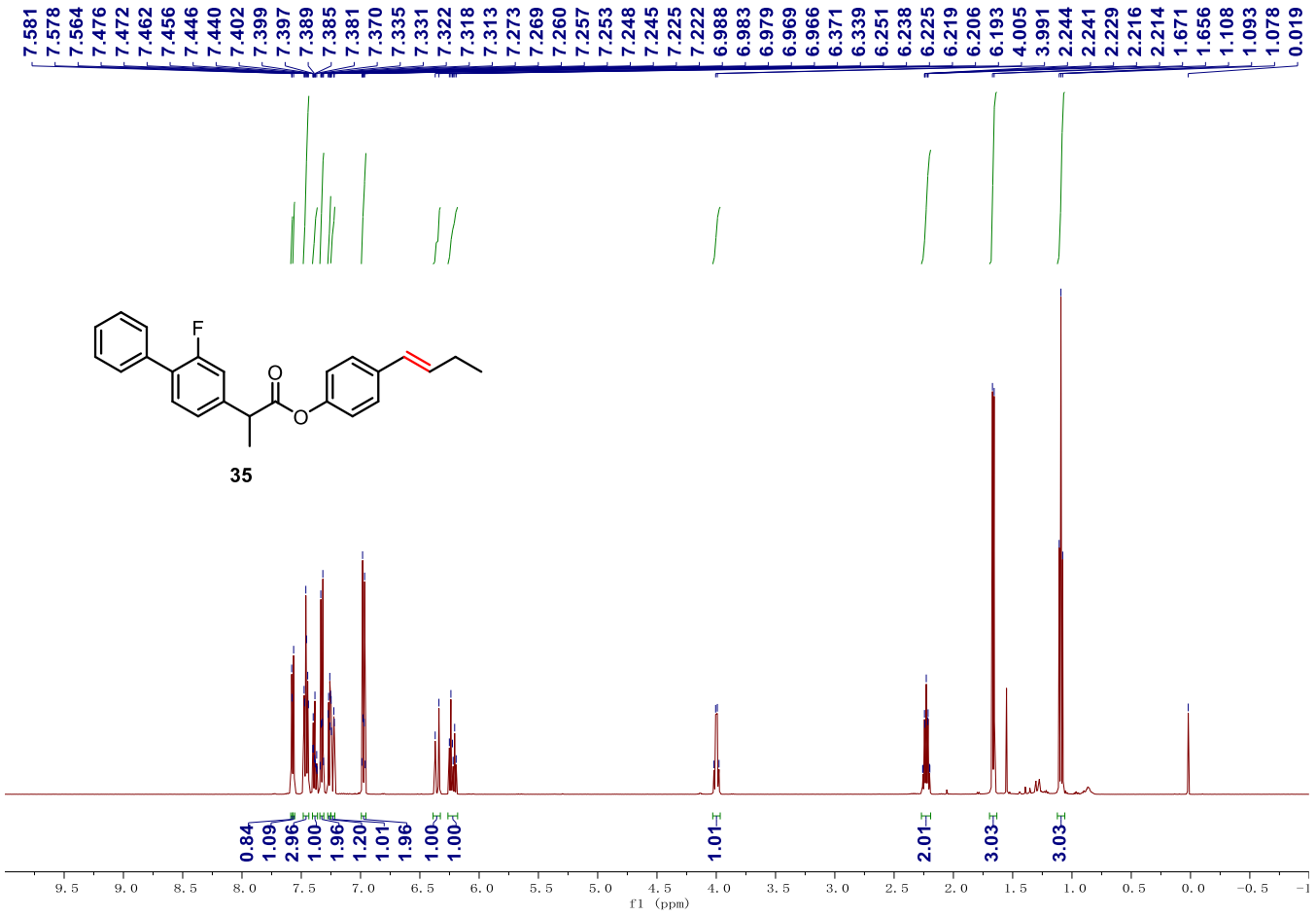
77.35
77.10
76.85
45.40
26.98
26.61
19.50
18.44
34
f1 (ppm)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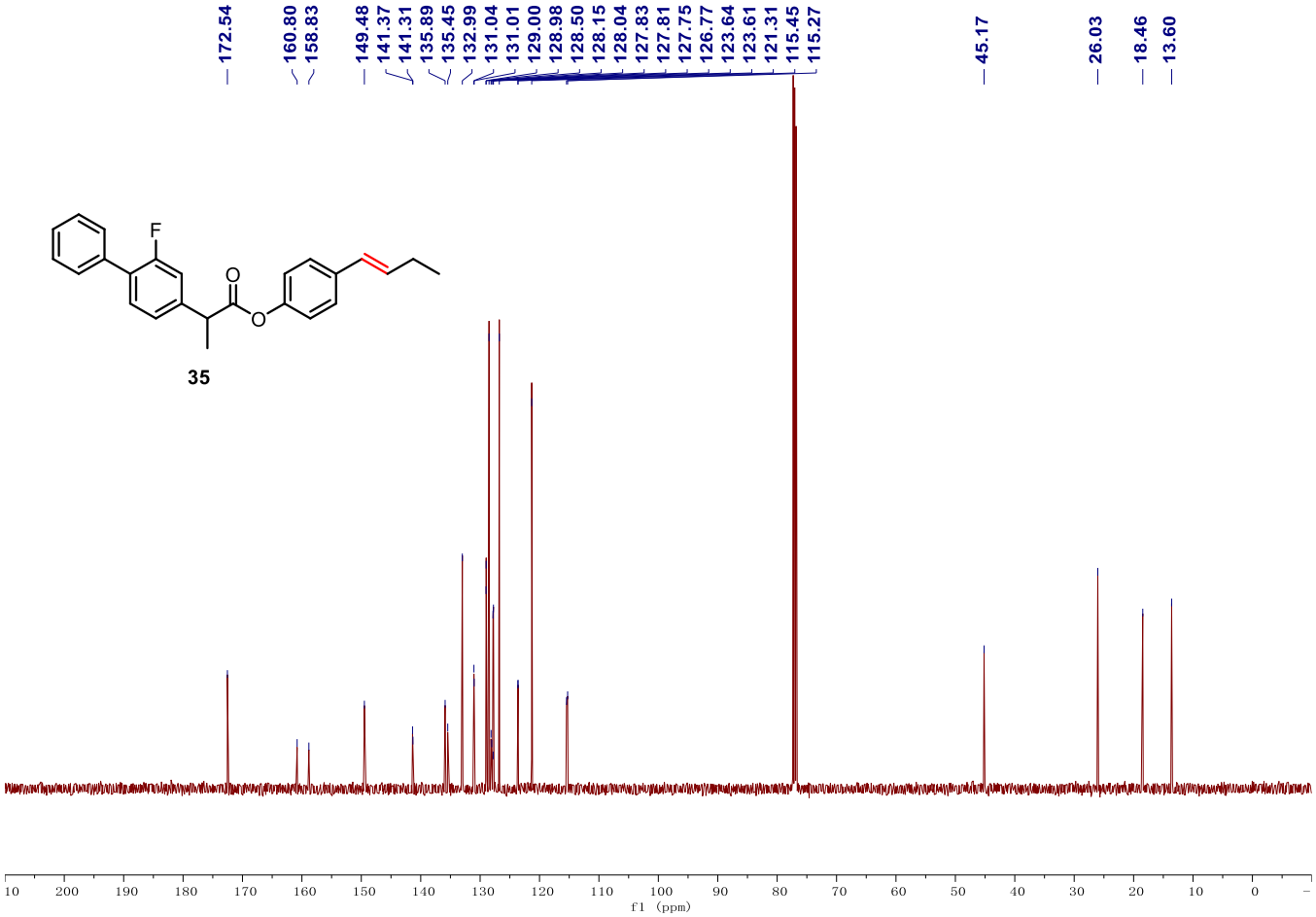
7.402
7.399
7.397
7.389
7.385
7.381
7.370
7.335
7.331
7.322
7.318
7.313
7.273
7.269
7.260
7.257
7.253
7.248
7.245
7.225
7.222
6.988
6.983
6.979
6.969
6.966
6.371
6.339
6.251
6.238
6.225
6.219
6.206
6.193
4.005
3.991
2.244
2.241
2.229
2.216
2.214
1.671
1.656
1.108
1.093
1.078
0.019
F
O
O
35
0.84
1.09
2.96
1.00
1.96
1.20
1.01
1.96
1.00
1.00
1.01
2.01
3.03
3.03
f1 (ppm)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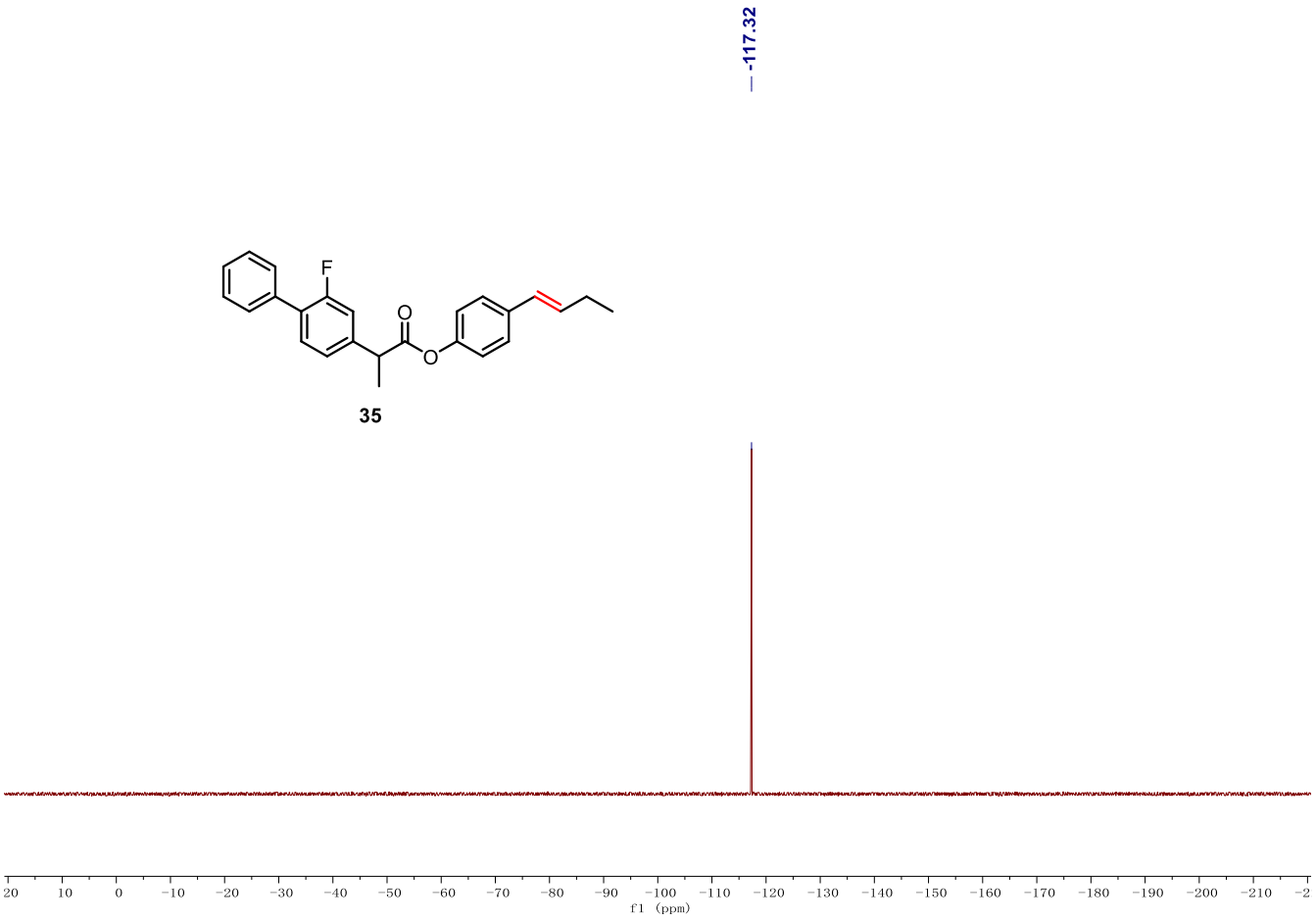
160.80
158.83
149.48
141.37
141.31
135.89
135.45
132.99
131.04
131.01
129.00
128.98
128.50
128.15
128.04
127.83
127.81
127.75
126.77
123.64
123.61
121.31
115.45
115.27
45.17
26.03
18.46
13.60
F
O
O
35
f1 (ppm)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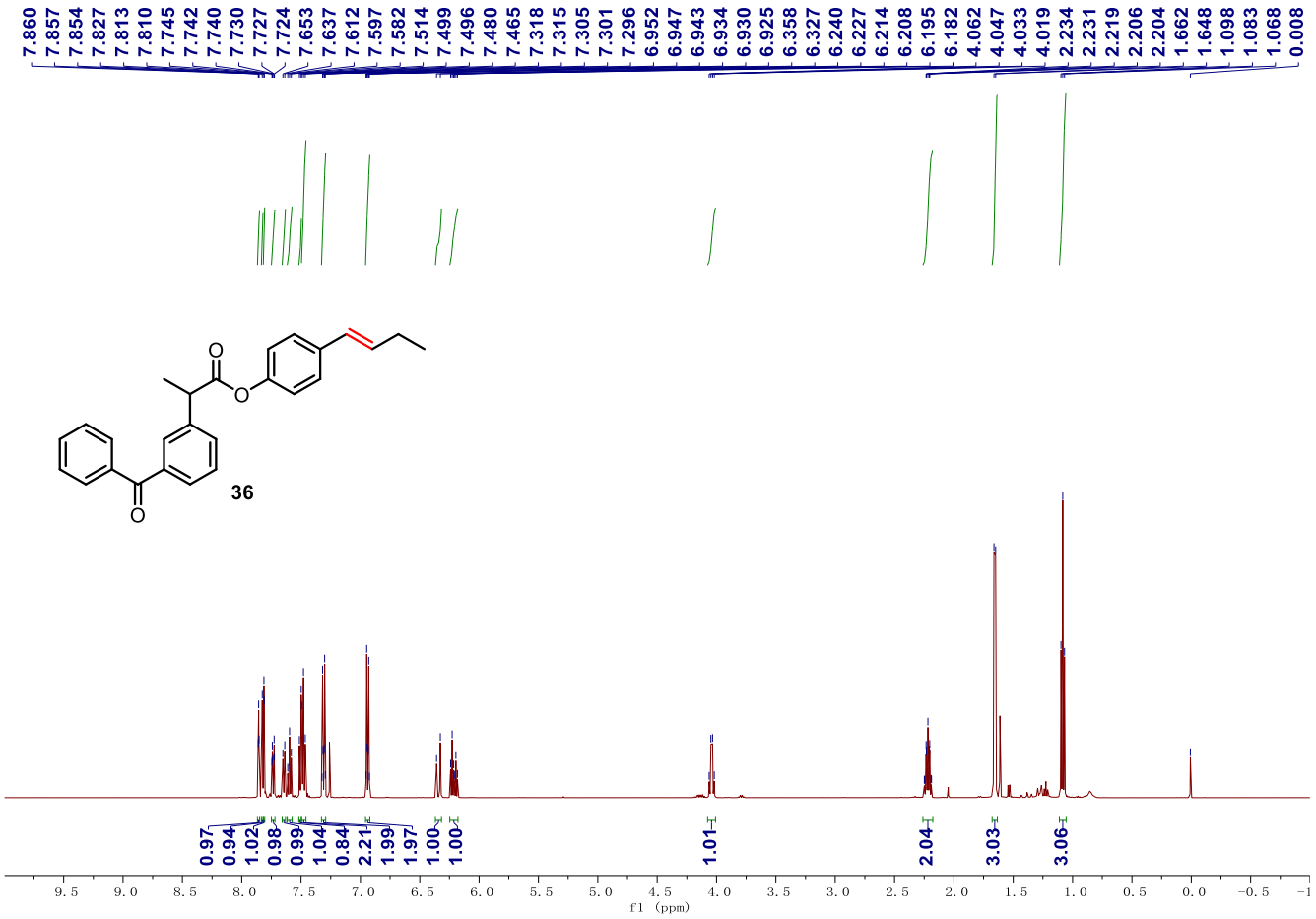
F
O
O
35
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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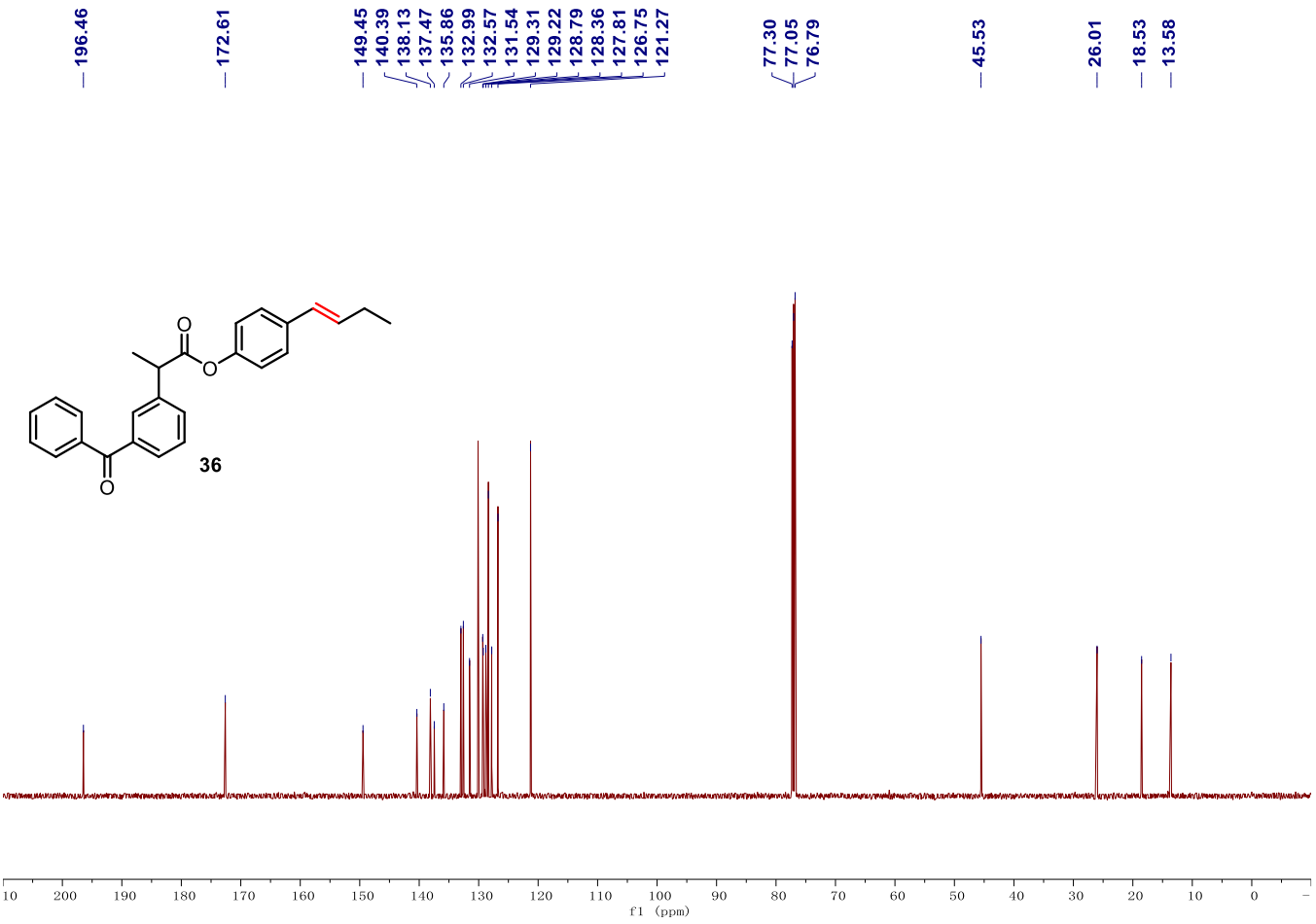
7.857
7.854
7.827
7.813
7.810
7.745
7.742
7.740
7.730
7.727
7.724
7.653
7.637
7.612
7.597
7.582
7.514
7.499
7.496
7.480
7.465
7.318
7.315
7.305
7.301
7.296
6.952
6.947
6.943
6.934
6.930
6.925
6.358
6.327
6.240
6.227
6.214
6.208
6.195
6.182
4.062
4.047
4.033
4.019
2.234
2.231
2.219
2.206
2.204
1.662
1.648
1.098
1.083
1.068
0.008
O
O
O
36
0.97
0.94
1.02
0.98
0.99
1.04
0.84
2.21
1.99
1.97
1.00
1.00
1.01
2.04
3.03
3.06
f1 (ppm)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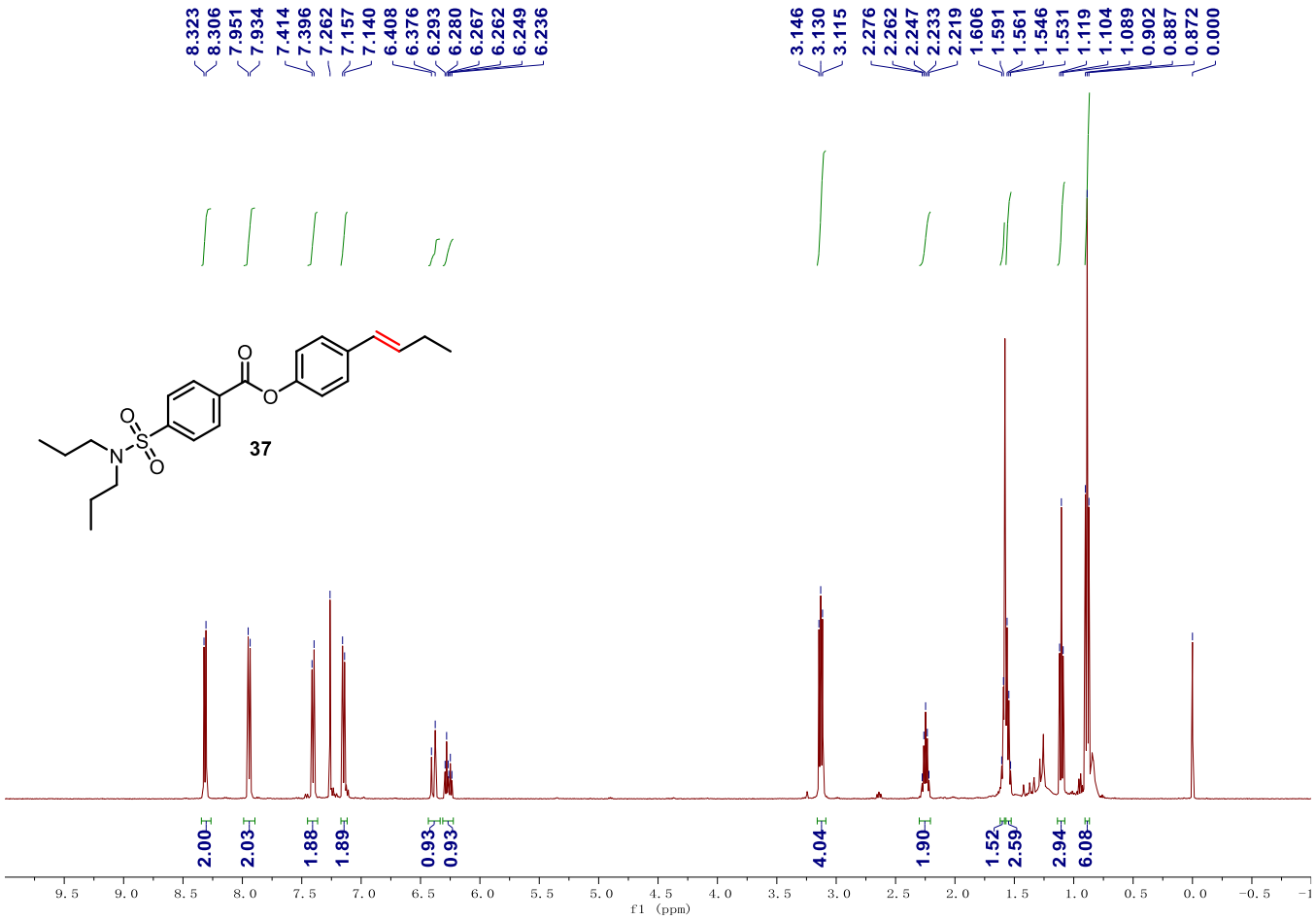
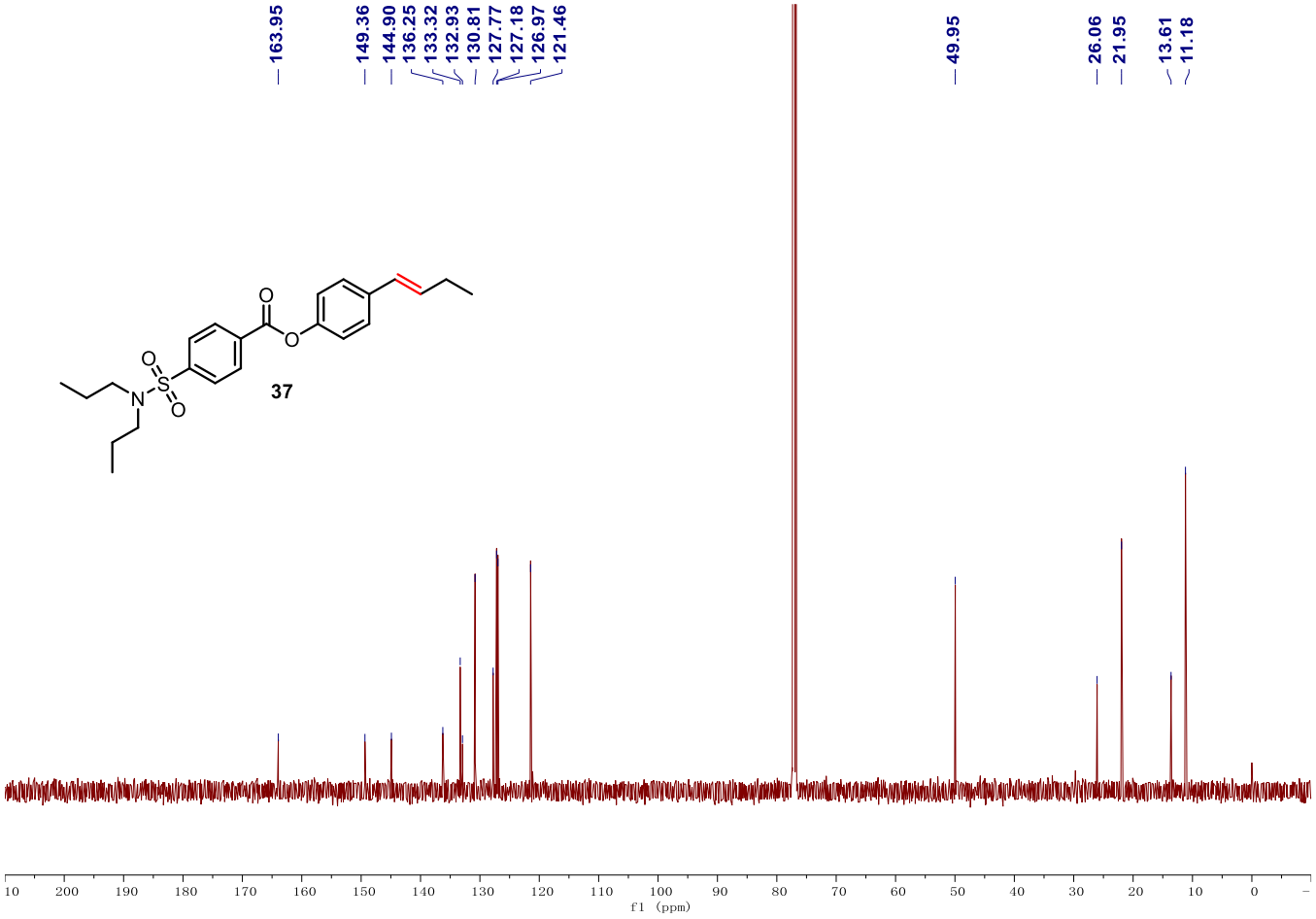
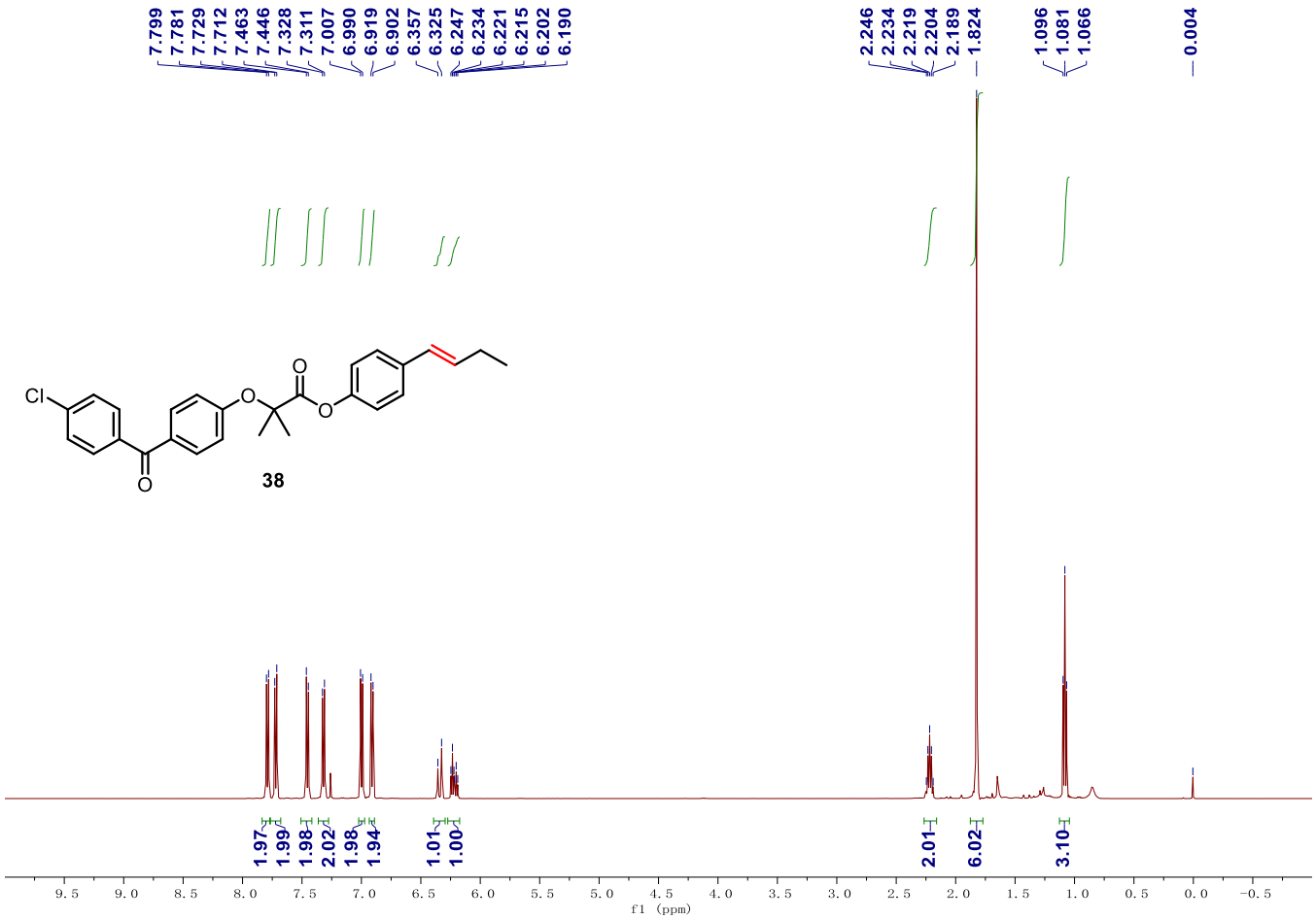
172.61
149.45
140.39
138.13
137.47
135.86
132.99
132.57
131.54
129.31
129.22
128.79
128.36
127.81
126.75
121.27
77.30
77.05
76.79
45.53
26.01
18.53
13.58
O
O
O
36
f1 (ppm)


8.323
8.306
7.951
7.934
7.414
7.396
7.262
7.157
7.140
6.408
6.376
6.293
6.280
6.267
6.262
6.249
6.236
3.146
3.130
3.115
2.276
2.262
2.247
2.233
2.219
1.606
1.591
1.561
1.546
1.531
1.119
1.104
1.089
0.902
0.887
0.872
0.000
37
2.00
2.03
1.88
1.89
0.93
0.93
4.04
1.90
1.52
2.59
2.94
6.08
f1 (ppm)


163.95
149.36
144.90
136.25
133.32
132.93
130.81
127.77
127.18
126.97
121.46
49.95
26.06
21.95
13.61
11.18
37
f1 (ppm)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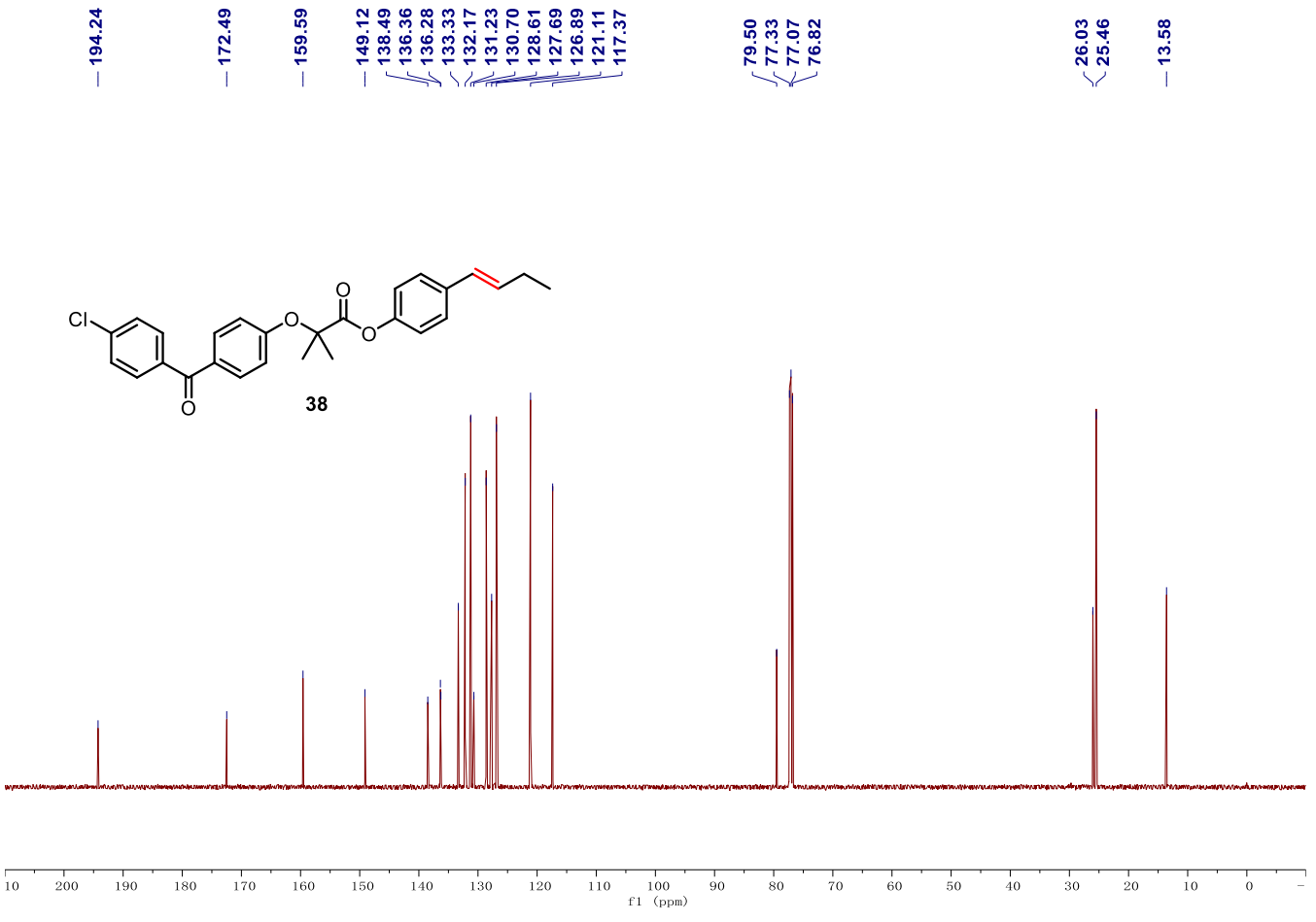
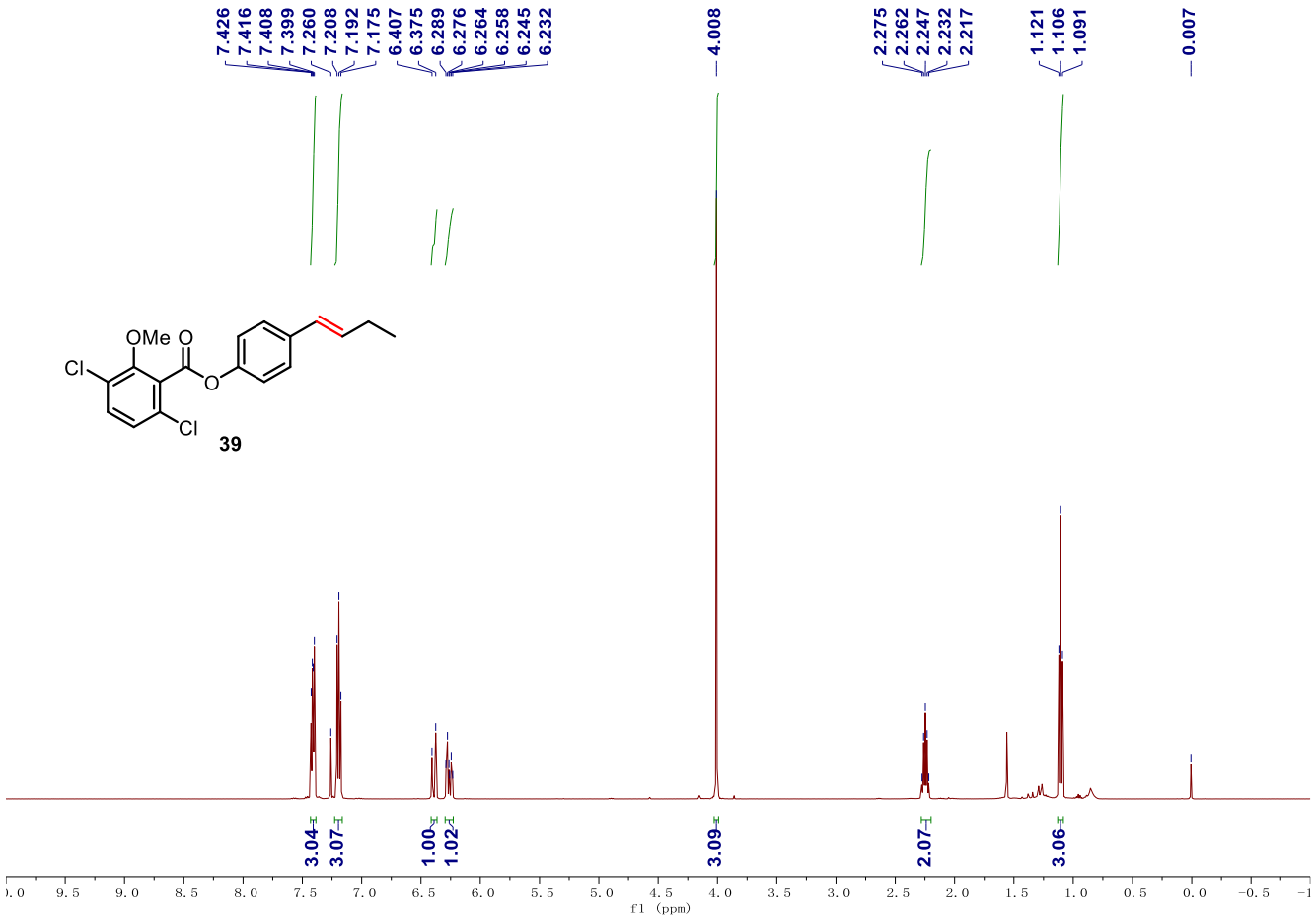
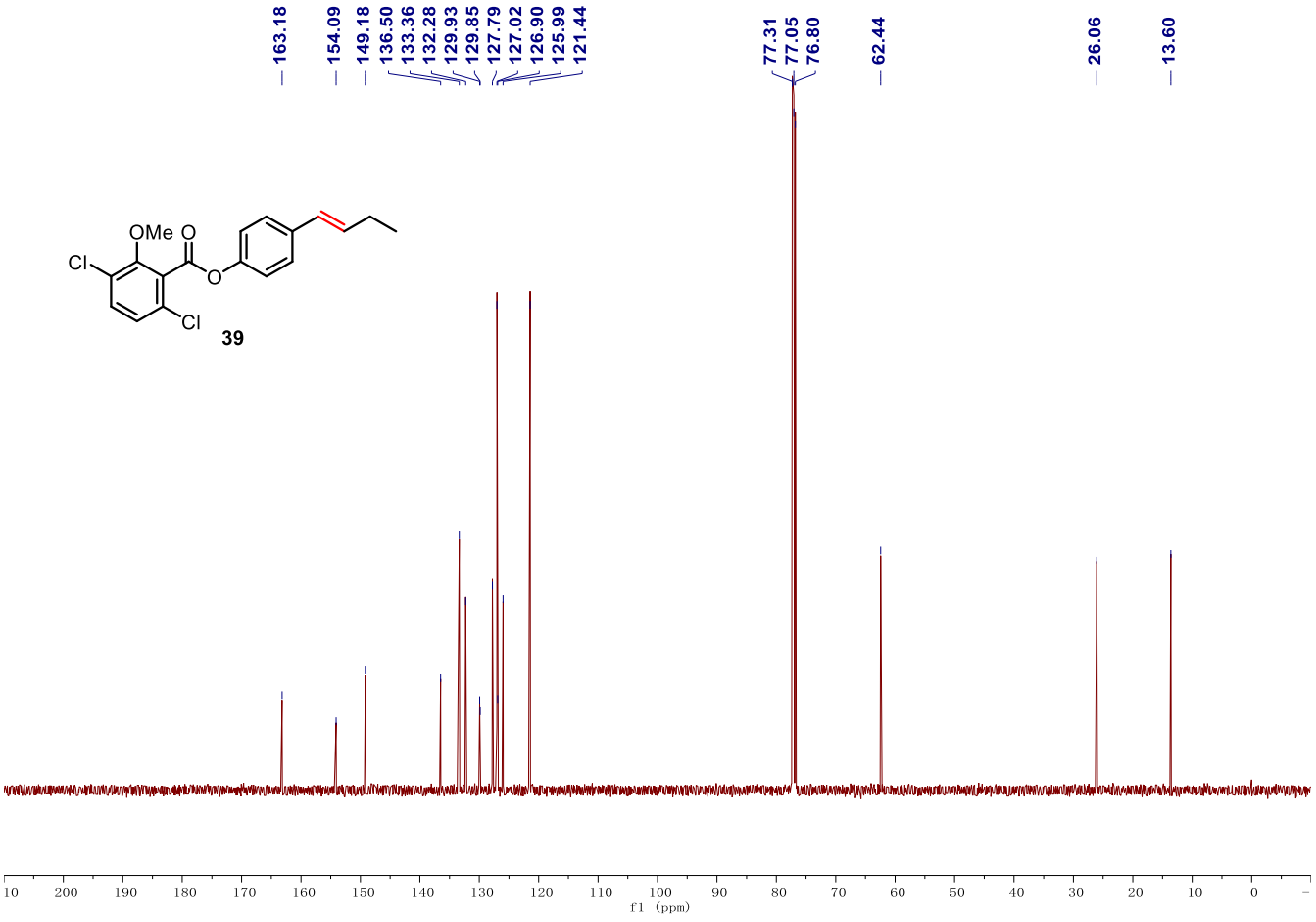
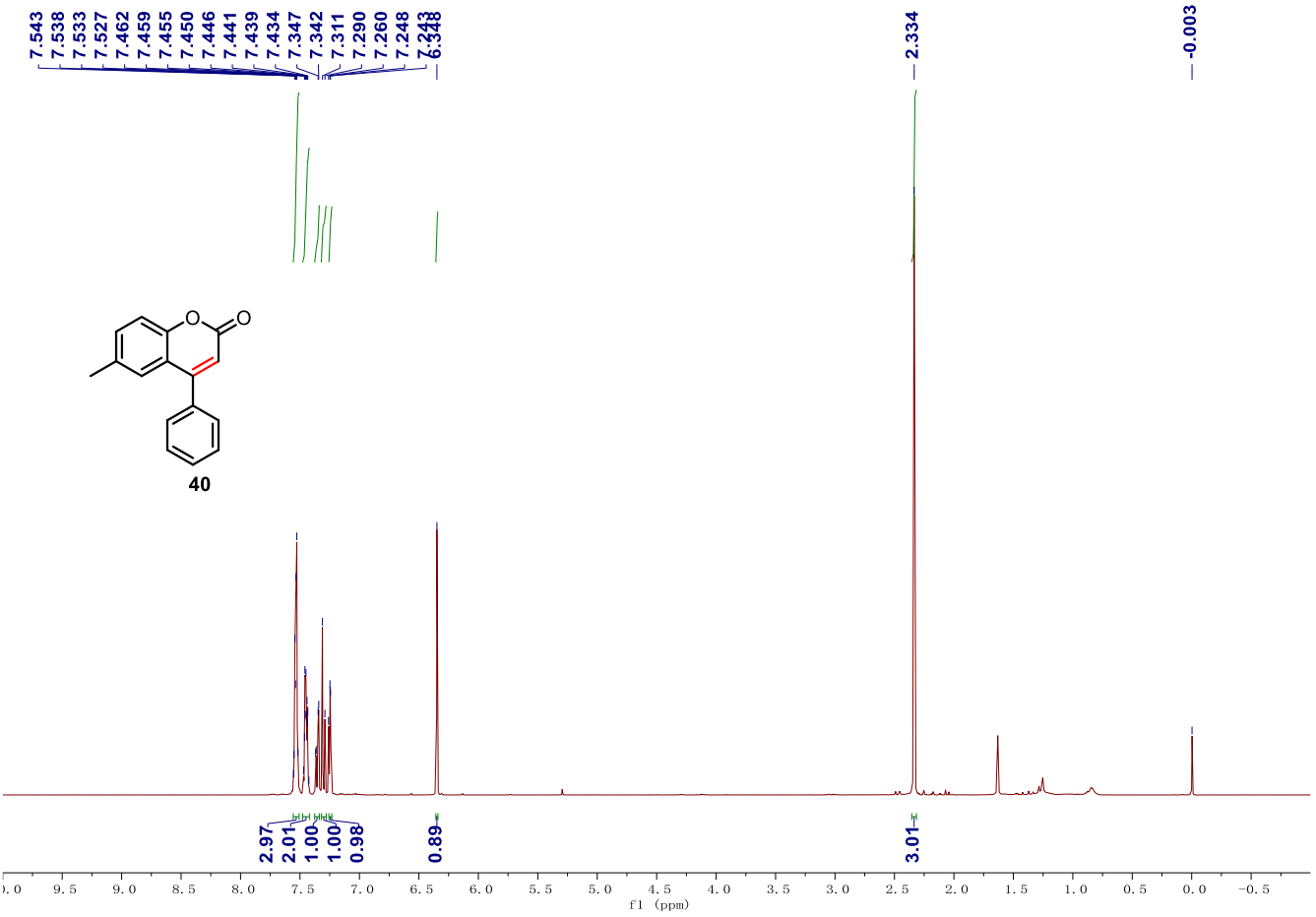
7.781
7.729
7.712
7.463
7.446
7.328
7.311
7.007
6.990
6.919
6.902
6.357
6.325
6.247
6.234
6.221
6.215
6.202
6.190
2.246
2.234
2.219
2.204
2.189
1.824
1.096
1.081
1.066
0.004
Cl
O
O
O
O
38
1.97
1.99
1.98
2.02
1.98
1.94
1.01
1.00
2.01
6.02
3.10
f1 (ppm)


194.24
172.49
159.59
149.12
138.49
136.36
136.28
133.33
132.17
131.23
130.70
128.61
127.69
126.89
121.11
117.37
79.50
77.33
77.07
76.82
26.03
25.46
13.58
Cl
O
O
O
O
38
f1 (ppm)


7.426
7.416
7.408
7.399
7.260
7.208
7.192
7.175
6.407
6.375
6.289
6.276
6.264
6.258
6.245
6.232
4.008
2.275
2.262
2.247
2.232
2.217
1.121
1.106
1.091
0.007
OMe
O
Cl
O
Cl
39
3.04
3.07
1.00
1.02
3.09
2.07
3.06
f1 (ppm)


163.18
154.09
149.18
136.50
133.36
132.28
129.93
129.85
127.79
127.02
126.90
125.99
121.44
77.31
77.05
76.80
62.44
26.06
13.60
OMe
O
Cl
O
Cl
39
f1 (ppm)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7.533
7.527
7.462
7.459
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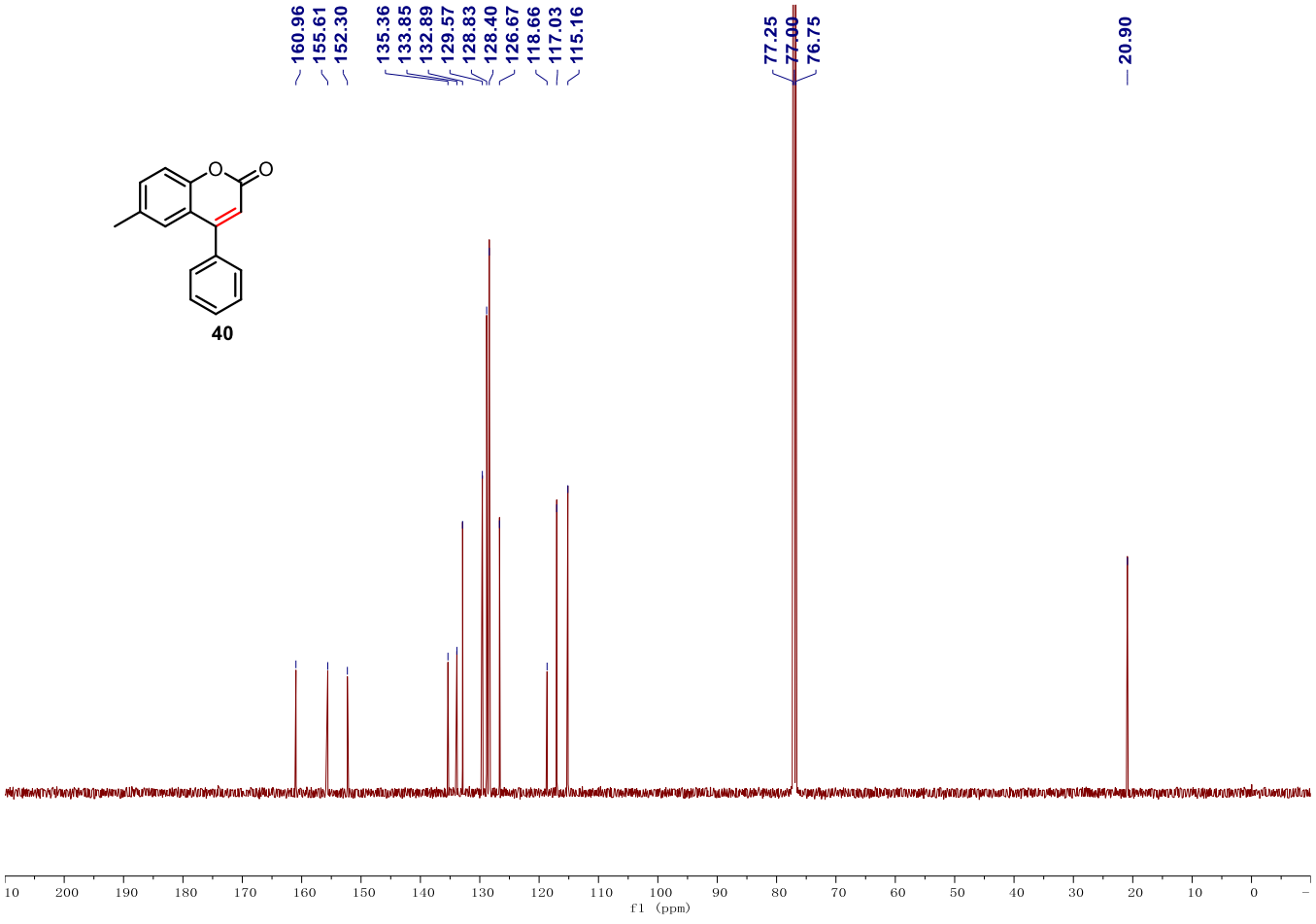
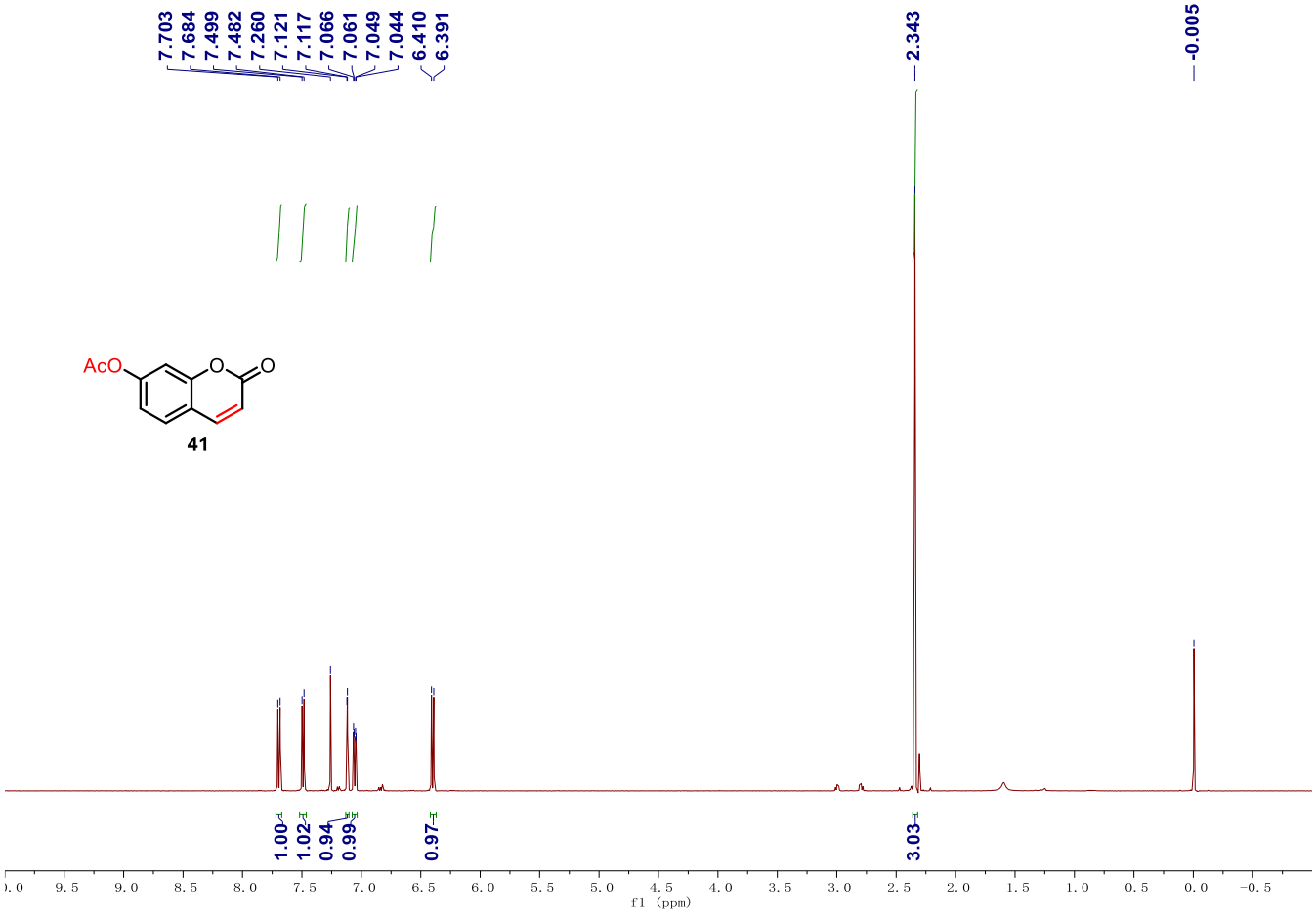
7.450
7.446
7.441
7.439
7.434
7.347
7.342
7.311
7.290
7.260
7.248
7.243
6.348
2.334
-0.003
40
2.97
2.01
1.00
1.00
0.98
0.89
3.01
f1 (ppm)


160.96
155.61
152.30
135.36
133.85
132.89
129.57
128.83
128.40
126.67
118.66
117.03
115.16
77.25
77.00
76.75
20.90
40
f1 (ppm)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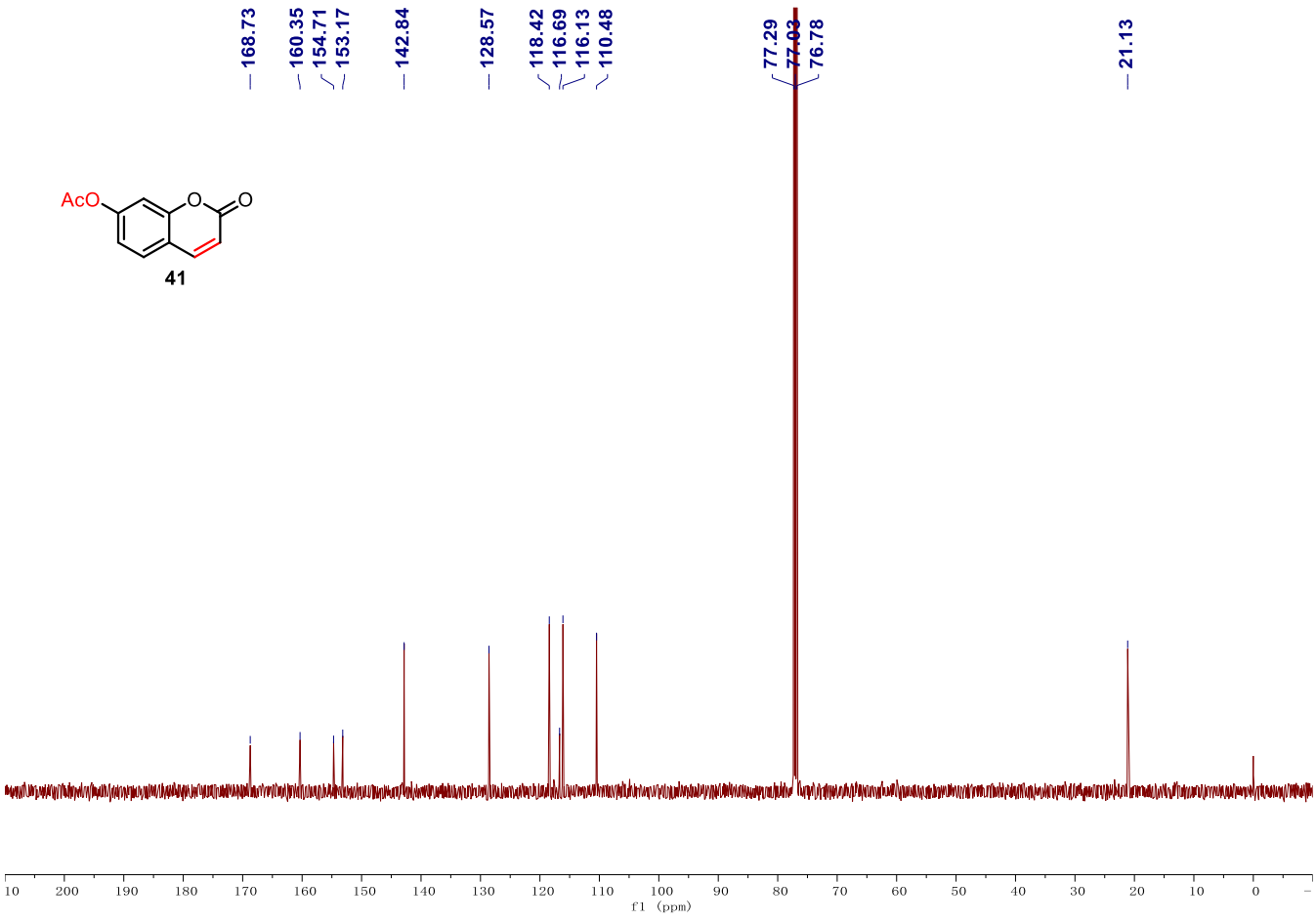
7.684
7.499
7.482
7.260
7.121
7.117
7.066
7.061
7.049
7.044
6.410
6.391
2.343
-0.005
AcO
O
O
41
1.00
1.02
0.94
0.99
0.97
3.03
f1 (ppm)


168.73
160.35
154.71
153.17
142.84
128.57
118.42
116.69
116.13
110.48
77.29
77.03
76.78
21.13
AcO
O
O
41
f1 (ppm)