Electronic Supplementary Material (ESI) for RSC Advances.

Palladium(II)-Catalyzed Sequential *ortho*-Olefination of *β*-Arylethamines with Assistance of Oxalyl Amide

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Supporting Information

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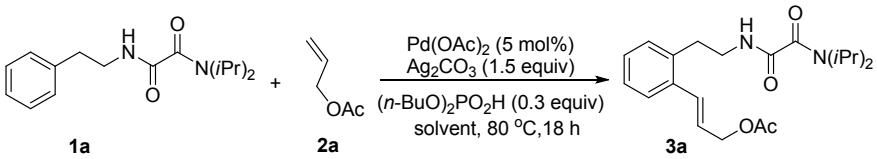
1. Reagents: Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. Column chromatography purifications were performed using 300–400 mesh silica gel.

2. Instruments: NMR spectra were recorded on Varian Inova–400 MHz, Inova–300 MHz, Bruker DRX–400 or Bruker DRX–500 instruments and calibrated using residual solvent peaks as internal reference. Multiplicities are recorded as: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, br = broad singlet, m = multiplet. HRMS analyses were carried out using a Bruker microTOF–Q instrument or a TOF–MS instrument.

3. Screening of Solvents

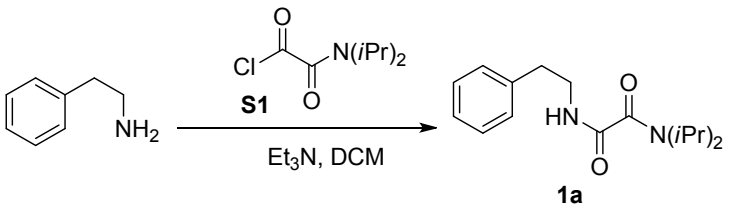
Table 1. Screening of Solvents



Entry	Solvent	Yield(%)[a]
1	Toluene	63
2	PhCl	57
3	DCE	80
4	DMF	44
5	NMP	13
6	Dioxane	46
7	AcOH	<5
8	*t*-AmylOH	60
9	CH_3CN	55
10	HFIP	24

[a] Reactions were carried out on a 0.1 mmol scale; yield was based on LC using acetophenone as the internal standard.

4. Preparation of oxalyl amide substrates



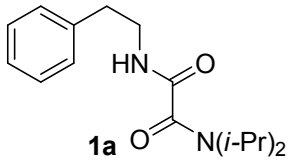
4.1. Preparation of N, N–Diisopropyloxamoyl chloride S1[1]

A solution of Diisopropylamine (7.01 mL, 50 mmol, 1.0 equiv) in CH_2Cl_2 (50 mL) was added dropwise to a solution of oxalyl chloride (6.44 ml, 75 mmol, 1.5 equiv) in CH_2Cl_2 (100 mL) at 0 °C, after stirring for 5 min, triethylamine (7.30 mL, 52.5 mmol, 1.05 equiv) was added dropwise. The solution was warmed to room temperature and stirred for 6 hours. The excess of oxalyl

chloride and the solvent were removed under reduce pressure and then CH_2Cl_2 (30 mL) was added and evaporated. This operation was performed twice to give **S1** as a pale yellow solid. The crude product was used in the next step without any purification.

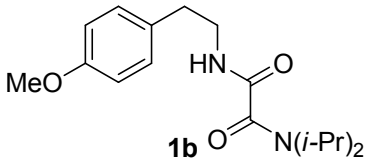
4.2. General procedures for the preparation of oxalyl amide substrates 1a–1i [2-6]

A solution of *β*-arylethamine (20 mmol, 1.0 equiv) and triethylamine (2.92 ml, 21 mmol, 1.05 equiv) in CH_2Cl_2 (40 mL) was added dropwise to a solution of N,N–Diisopropyloxamoyl chloride **S1** (25 mmol, 1.25 equiv) in CH_2Cl_2 (50 mL) at 0 °C, then the mixture was stirred for 6 hours at room temperature before quenched by water (50 mL). The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (20 mL × 2). The combined organic phase was washed with brine (30 mL), and then dried over anhydrous Na_2SO_4. Evaporation and column chromatography on silica gel afforded corresponding amide substrates as white solid or colourless liquid.



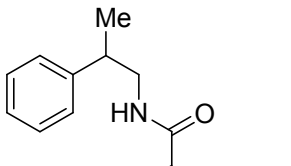
1a

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.32–7.27 (m, 2H), 7.23–7.20 (m, 3H), 7.11 (br, 1H), 4.60–4.53 (m, 1H), 3.58–3.53 (m, 2H), 3.52–3.45 (m, 1H), 2.86 (t, *J* = 7.2 Hz, 2H), 1.40 (d, *J* = 6.8 Hz, 6H), 1.19 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 163.45, 163.41, 138.68, 128.85, 128.70, 126.62, 49.78, 46.52, 40.53, 35.55, 20.92, 20.16. This compound is known.[5]



1b

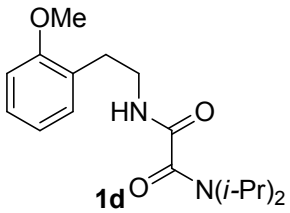
^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.22–7.18 (m, 1H), 7.10 (br, 1H), 6.81–6.75 (m, 3H), 4.55 (d, *J* = 6.4 Hz, 1H), 3.78 (s, 3H), 3.56–3.51 (m, 2H), 3.49–3.45 (m, 1H), 2.82 (t, *J* = 7.2 Hz, 2H), 1.39 (d, *J* = 6.8 Hz, 6H), 1.18 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 163.46, 159.87, 140.22, 129.68, 121.15, 114.36, 112.18, 55.25, 49.80, 46.50, 40.39, 35.57, 20.90, 20.14. This compound is known.[5]



1c

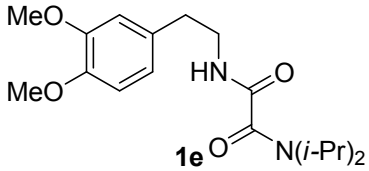
^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.30 (t, *J* = 7.6 Hz, 2H), 7.23–7.19 (m, 3H), 6.94 (br, 1H), 4.42–4.37 (m, 1H), 3.54–3.39 (m, 3H), 3.03–2.94 (m, 1H), 1.38–1.35 (m, 6H), 1.28 (d, *J* = 7.0 Hz, 3H), 1.16–1.12 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 163.54, 143.93, 128.72, 127.28, 126.77, 49.83,

46.40, 45.86, 39.78, 20.88, 20.16, 20.14, 19.45. This compound is known.[6]



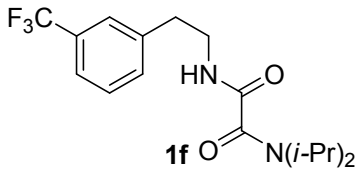
1d

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.21 (t, J = 7.6 Hz, 1H), 7.15 (d, J = 7.2 Hz, 2H), 6.88 (dd, J = 17.8, 7.9 Hz, 2H), 4.63–4.60 (m, 1H), 3.83 (s, 3H), 3.53–3.48 (m, 3H), 2.88 (t, J = 6.7 Hz, 2H), 1.40 (d, J = 6.7 Hz, 6H), 1.19 (d, J = 6.5 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.41, 157.56, 130.64, 128.00, 127.12, 120.69, 110.36, 55.30, 49.64, 46.45, 39.65, 30.13, 20.92, 20.11. This compound is known.[5]



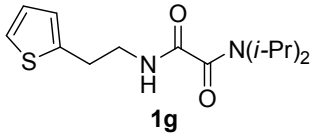
1e

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.03 (br, 1H), 6.81–6.78 (m, 1H), 6.74 (dd, J = 5.9, 1.8 Hz, 2H), 4.61–4.54 (m, 1H), 3.85 (d, J = 9.5 Hz, 6H), 3.54–3.44 (m, 3H), 2.78 (t, J = 7.1 Hz, 2H), 1.38 (d, J = 6.8 Hz, 6H), 1.18 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.41, 163.29, 149.09, 147.80, 131.16, 120.77, 112.02, 111.45, 56.00, 55.94, 49.76, 46.54, 40.60, 35.16, 20.91, 20.14. This compound is known.[6]



1f

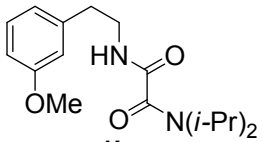
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, J = 7.7 Hz, 2H), 7.42 (d, J = 5.1 Hz, 2H), 7.17 (br, 1H), 4.61–4.58 (m, 1H), 3.59–3.54 (m, 2H), 3.52–3.45 (m, 1H), 2.92 (t, J = 7.2 Hz, 2H), 1.39 (d, J = 6.8 Hz, 6H), 1.19 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.47, 163.16, 139.64, 132.32, 131.19, 130.87, 129.19, 125.63, 125.59, 123.62, 123.58, 122.87, 49.82, 46.65, 40.29, 35.39, 20.91, 20.15. This compound is known.[5]



1g

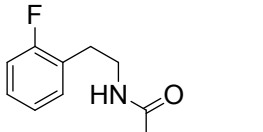
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.29 (br, 1H), 7.13 (d, J = 4.9 Hz, 1H), 6.94–6.90 (m, 1H), 6.85 (s, 1H), 4.59–4.52 (m, 1H), 3.58–3.53 (m, 6.6 Hz, 2H), 3.51–3.44 (m, 1H), 3.06 (t, J = 6.8 Hz, 2H), 1.39 (d, J = 6.8 Hz, 6H), 1.19 (d, J = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.50, 163.42, 140.98, 127.09, 125.47, 123.95, 49.81, 46.47, 40.69, 29.63, 20.88, 20.13; HRMS Calcd for

$C_{14}H_{22}N_2NaO_2S$ [M+Na$^+$]: 305.1300; Found: 305.1308.



1h

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.22–7.18 (m, 1H), 7.10 (br, 1H), 6.80 (d, J = 7.7 Hz, 1H), 6.77–6.75 (m, 2H), 4.58–4.52 (m, 1H), 3.78 (s, 3H), 3.56–3.51 (m, 2H), 3.49–3.44 (m, 1H), 2.82 (t, J = 7.2 Hz, 2H), 1.39 (d, J = 6.8 Hz, 6H), 1.18 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.46, 159.87, 140.22, 129.68, 121.15, 114.36, 112.18, 55.25, 49.80, 46.50, 40.39, 35.57, 20.90, 20.14. This compound is known.[6]

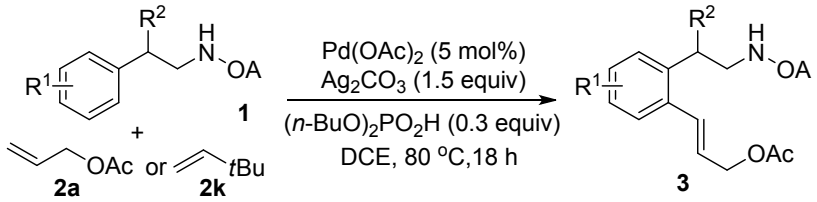


1i

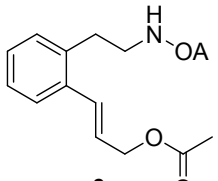
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24–7,19 (m, 2H), 7.10–7.01 (m, 2H), 6.94 (br, 1H), 4.68–4.61 (m, 1H), 3.58–3.53 (m, 2H), 3.51–3.46 (m, 1H), 2.91 (t, J = 7.1 Hz, 2H), 1.41 (d, J = 6.8 Hz, 6H), 1.20 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.49, 163.27, 162.62, 160.18, 131.20, 131.15, 128.51, 128.43, 125.71, 125.55, 124.32, 124.28, 115.59, 115.37, 49.76, 46.55, 39.38, 29.03, 29.01, 20.93, 20.15. This compound is known.[5]

5. Alkenylation of Oxalyl Amide Protected Phenethylamine

5.1 Substrates Scope of Amines

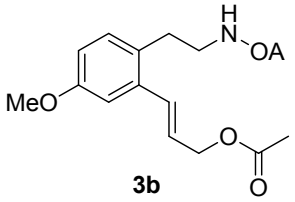


A mixture of oxalyl amide (0.2 mmol, 1 equiv), olefin (2 equiv), $Pd(OAc)_2$ (2.2 mg, 0.05 equiv), Ag_2CO_3 (82.8 mg, 1.5 equiv), (n-BuO)$_2PO_2H$ (12.6 mg, 0.3 equiv) and DCE (0.6 mL) in a 15 mL glass tube (sealed with PTFE cap) was heated at 80 °C for 18 hours. The reaction mixture was cooled to room tempreture, and concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to give the alkenylated product.



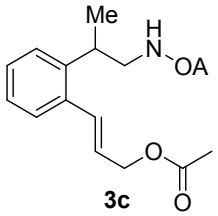
3a

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (dd, J = 6.3, 2.7 Hz, 1H), 7.27–7.17 (m, 3H), 7.03 (s, 1H), 6.96 (d, J = 15.7 Hz, 1H), 6.22-6.15 (m, 1H), 4.77-4.75 (m, 2H), 4.72–4.65 (m, 1H), 3.50-3.45 (m, 3H), 2.96–2.92 (m, 2H), 2.12 (s, 3H), 1.41 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.05, 163.39, 163.09, 136.17, 135.60, 131.38, 130.08, 128.35, 127.20, 126.62, 125.79, 65.28, 49.73, 46.63, 40.09, 32.96, 21.15, 20.98, 20.16; HRMS Calcd for $C_{21}H_{30}N_2O_4$ [M+Na$^+$]: 397.2203; Found: 397.2215.



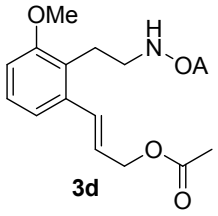
3b

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.09 (d, J = 8.4 Hz, 1H), 7.00–6.99 (m, 2H), 6.91 (d, J = 15.7 Hz, 1H), 6.80 (dd, J = 8.4, 2.7 Hz, 1H), 6.21-6.14 (m, 1H), 4.75 (dd, J = 6.3, 1.3 Hz, 2H), 4.72-4.67 (m, 1H), 3.80 (s, 3H), 3.54-3.47 (m, 1H), 3.46-3.40 (m, 2H), 2.94–2.87 (m, 2H), 2.12 (s, 3H), 1.41 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.04, 163.37, 163.12, 158.63, 136.58, 131.38, 131.19, 128.53, 125.86, 114.12, 111.66, 65.21, 55.43, 49.73, 46.64, 40.34, 32.18, 21.15, 20.99, 20.16; HRMS Calcd for $C_{22}H_{32}N_2O_5$ [M+Na$^+$]: 427.2209; Found: 427.2218.



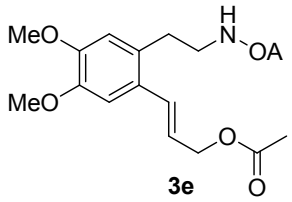
3c

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (d, J = 7.5 Hz, 1H), 7.32–7.20 (m, 3H), 7.03 (d, J = 15.6 Hz, 1H), 6.83 (br, 1H), 6.17-6.10 (m, 1H), 4.77–4.75 (m, 2H), 4.55-4.49 (m, 1H), 3.52–3.45 (m, 3H), 3.43-3.33 (m, 1H), 2.13 (s, 3H), 1.40 (d, J = 6.8 Hz, 6H), 1.30 (d, J = 6.8 Hz, 3H), 1.18 (dd, J = 6.7, 1.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.06, 163.51, 163.29, 141.22, 135.81, 131.70, 128.57, 127.21, 126.86, 126.29, 125.65, 65.28, 49.79, 46.55, 45.25, 34.63, 21.16, 20.95, 20.18, 19.10; HRMS Calcd for $C_{21}H_{31}N_2O_4$ [M+Na$^+$]: 411.2260; Found: 411.2278.



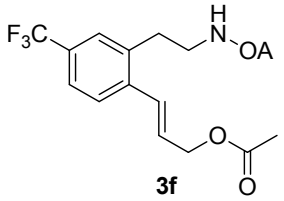
3d

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.19-7.15 (m, 2H), 7.07 (d, J = 7.6 Hz, 1H), 6.94 (d, J = 15.7 Hz, 1H), 6.80 (d, J = 8.1 Hz, 1H), 6.19-6.14 (m, 1H), 4.74 – 4.67 (m, 3H), 3.83 (s, 3H), 3.50 – 3.44 (m, 1H), 3.43-3.38 (m, 1H), 2.98 (t, J = 7.1 Hz, 2H), 2.10 (s, 3H), 1.40 (d, J = 6.8 Hz, 6H), 1.18 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 170.99, 163.34, 163.07, 157.72, 137.23, 131.35, 127.60, 126.35, 125.10, 119.06, 109.84, 65.20, 55.65, 49.52, 46.56, 39.52, 25.21, 21.11, 20.97, 20.12. HRMS Calcd for $C_{23}H_{34}N_2O_6$ [M+Na$^+$]: 427.2209; Found:427.2220.



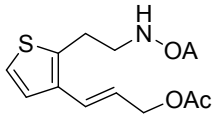
3e

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.03 (br, 1H), 7.00 (s, 1H), 6.90 (d, J = 15.6 Hz, 1H), 6.70 (s, 1H), 6.16-6.09 (m, 1H), 4.77–4.70 (m, 3H), 3.91 (s, 6H), 3.56–3.41 (m, 3H), 2.90 (t, J = 7.3 Hz, 2H), 2.13 (s, 3H), 1.43 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.11, 163.38, 162.97, 149.20, 148.00, 131.38, 129.05, 127.51, 123.50, 113.00, 109.09, 65.54, 56.07, 49.70, 46.68, 40.37, 32.54, 21.21, 20.99, 20.15; HRMS Calcd for $C_{23}H_{34}N_2O_6$ [M+Na$^+$]: 457.2315; Found: 457.2340.



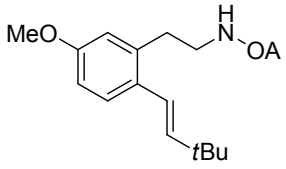
3f

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, J = 8.1 Hz, 1H), δ 7.45 (d, J = 8.2 Hz, 1H), 7.42 (s, 1H), 7.20 (br, 1H), 6.97 (d, J = 15.7 Hz, 1H), 6.28-6.21 (m, 1H), 4.77 (dd, J = 6.1, 1.3 Hz, 2H), 4.72 – 4.62 (m, 1H), 3.54–3.44 (m, 3H), 2.99–2.96 (m, 2H), 2.12 (s, 3H), 1.40 (d, J = 6.8 Hz, 6H), 1.21 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 170.98, 163.46, 162.93, 139.31, 136.90, 130.23, 129.90, 129.85, 128.29, 127.03, 126.84, 126.80, 125.49, 124.00, 123.96, 122.79, 64.85, 49.76, 46.71, 39.86, 32.95, 21.08, 20.94, 20.12; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.54. HRMS Calcd for $C_{22}H_{29}F_3N_2O_4$ [M+Na$^+$]: 465.1977; Found: 465.1987.



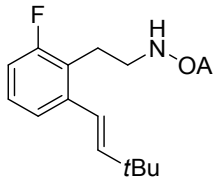
3g

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.14–7.08 (m, 3H), 6.65 (d, J = 15.7 Hz, 1H), 6.13–6.06 (m, 1H), 4.70 (d, J = 6.6 Hz, 2H), 3.53–3.46 (m, 3H), 3.08 (t, J = 7.1 Hz, 2H), 2.08 (s, 3H), 1.40 (d, J = 6.8 Hz, 6H), 1.21 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.98, 162.41, 161.91, 136.50, 134.08, 125.41, 124.57, 122.55, 122.42, 64.39, 48.73, 45.66, 39.75, 26.52, 20.14, 19.98, 19.15; HRMS Calcd for $C_{19}H_{28}N_2O_4S$ [M+Na$^+$]: 403.1667; Found: 403.1672.



3h

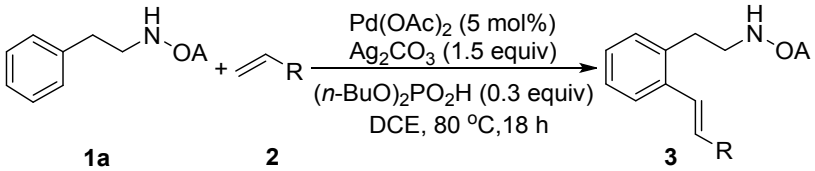
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35 (d, J = 8.5 Hz, 1H), 6.90 (br, 1H), 6.75 (dd, J = 8.5, 2.7 Hz, 1H), 6.69 (d, J = 2.6 Hz, 1H), 6.47 (d, J = 15.9 Hz, 1H), 6.02 (d, J = 15.9 Hz, 1H), 4.76-4.67 (m, 1H), 3.79 (s, 3H), 3.53 – 3.47 (m, 3H), 2.89 (t, J = 7.3 Hz, 2H), 1.41 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H), 1.12 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.30, 163.04, 158.79, 143.05, 136.81, 130.15, 127.59, 121.29, 114.94, 112.89, 55.43, 49.72, 46.69, 39.79, 33.23, 29.86, 21.01, 20.20; HRMS Calcd for $C_{23}H_{37}N_2O_3$ [M+H$^+$]: 389.2804; Found: 389.2825.



3i

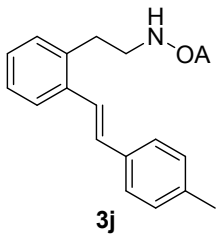
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.21 (d, J = 7.2 Hz, 1H), 7.16-7.11 (m, 1H), 6.94 – 6.87 (m, 2H), 6.54 (d, J = 15.9 Hz, 1H), 6.15 (d, J = 15.9 Hz, 1H), 4.75-4.68 (m, 1H), 3.53-3.44 (m, 3H), 2.99 – 2.95 (m, 2H), 1.41 (d, J = 6.8 Hz, 6H), 1.21 (d, J = 6.7 Hz, 6H), 1.13 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.33, 163.02, 162.81, 160.39, 145.99, 140.19, 127.84, 122.93, 122.06, 121.21, 113.60, 113.37, 49.68, 46.66, 39.12, 33.90, 29.66, 25.10, 20.99, 20.19. ^{19}F NMR (376 MHz, $CDCl_3$) δ: -118.10; HRMS Calcd for $C_{22}H_{33}FN_2O_2$ [M+H$^+$]: 377.2604; Found: 377.2618.

5.2 Substrates Scope of Olefins



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

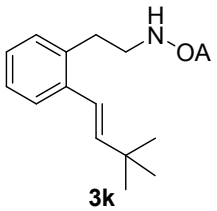
A mixture of **1a** (0.2 mmol, 1 equiv), olefin (2 equiv), $Pd(OAc)_2$ (2.2 mg, 0.05 equiv), Ag_2CO_3 (82.5 mg, 1.5 equiv), (n-BuO)$_2PO_2H$ (12.6 mg, 0.3 equiv) and DCE (0.6 mL) in a 15 mL glass tube (sealed with PTFE cap) was heated at 80 °C for 18 hours. The reaction mixture was cooled to room tempreture, and concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to give the alkenylated product.



3j

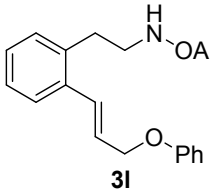
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (d, J = 7.2 Hz, 1H), 7.50 (d, J = 8.1 Hz, 2H), 7.44 (d, J = 16.1

Hz, 1H), 7.30–7.19 (m, 5H), 7.07 (br, 1H), 7.04–6.98 (m, 1H), 4.78-4.71 (m, 1H), 3.57–3.49 (m, 3H), 3.07–3.03 (m, 2H), 2.39 (s, 3H), 1.42 (d, J = 6.8 Hz, 6H), 1.24 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.40, 163.06, 137.76, 136.81, 136.14, 134.80, 131.04, 130.17, 129.52, 127.73, 127.25, 126.78, 126.04, 124.73, 49.74, 46.67, 40.32, 33.14, 21.40, 20.98, 20.16; HRMS Calcd for $C_{25}H_{32}N_2O_2$ [M+Na$^+$]: 415.2361; Found: 415.2378.



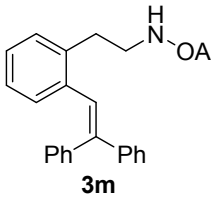
3k

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.46–7.44 (m, 1H), 7.23–7.15 (m, 3H), 6.99 (br, 1H), 6.58 (d, J = 15.9 Hz, 1H), 6.15 (d, J = 15.9 Hz, 1H), 4.73-4.67 (m, 1H), 3.55–3.49 (m, 3H), 2.95 (t, J = 7.4 Hz, 2H), 1.43 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H), 1.15 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.31, 163.18, 144.67, 137.50, 135.49, 129.84, 127.13, 127.07, 126.42, 121.87, 49.74, 46.62, 39.80, 33.79, 33.02, 29.74, 20.98, 20.18; HRMS Calcd for $C_{22}H_{34}N_2O_2$ [M+Na$^+$]: 381.2518; Found: 381.2535.



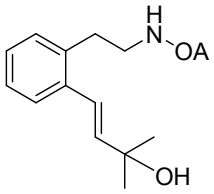
3l

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54–7.50 (m, 1H), 7.34–7.30 (m, 2H), 7.27–7.23(m, 2H), 7.21–7.18 (m, 1H), 7.06–6.97 (m, 4H), 6.92 (br, 1H), 6.37-6.30 (m, 1H), 4.79–4.68 (m, 3H), 3.57–3.42 (m, 3H), 2.95–2.91 (m, 2H), 1.44 (d, J = 6.8 Hz, 6H), 1.24 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.40, 163.09, 158.63, 136.09, 135.88, 130.13, 130.11, 129.62, 128.19, 127.22, 127.13, 126.64, 121.04, 115.03, 68.56, 49.72, 46.66, 40.14, 33.02, 21.00, 20.18. HRMS Calcd for $C_{25}H_{32}N_2O_3$ [M+Na$^+$]: 431.2311; Found: 431.2334.



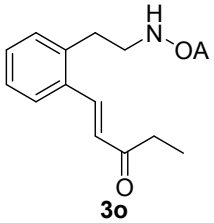
3m

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.36–7.30 (m, 5H), 7.22–7.16 (m, 4H), 7.11–7.04 (m, 4H), 6.95–6.86 (m, 3H), 4.76-4.69 (m, 1H), 3.60–3.49 (m, 3H), 2.93 (t, J = 7.3 Hz, 1H), 1.41 (d, J = 6.8 Hz, 6H), 1.20 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.21, 163.00, 144.27, 143.44, 140.08, 137.27, 137.01, 130.82, 130.65, 129.49, 128.35, 128.29, 128.27, 127.80, 127.39, 127.26, 126.30, 126.25, 49.68, 46.68, 39.76, 33.31, 21.01, 20.19; HRMS Calcd for $C_{30}H_{34}N_2O_2$ [M+Na$^+$]: 477.2518; Found: 477.2518.



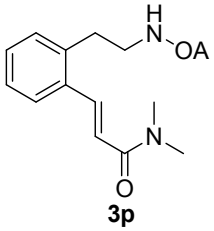
3n

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48–7.46 (m, 1H), 7.24–7.16 (m, 4H), 7.00 (d, J = 15.9 Hz, 1H), 6.28 (d, J = 15.8 Hz, 1H), 4.69–4.62 (m, 1H), 3.53–3.47 (m, 3H), 3.28 (s, 1H), 2.96 (t, J = 7.3 Hz, 2H), 1.44 (s, 6H), 1.42 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H);^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.28, 163.14, 141.07, 136.87, 136.33, 130.05, 127.64, 127.19, 126.48, 123.51, 70.93, 49.92, 46.71, 40.71, 33.27, 29.82, 20.95, 20.19; HRMS Calcd for $C_{23}H_{32}N_2O_3$ [M+Na$^+$]: 383.2311; Found: 383.2331.



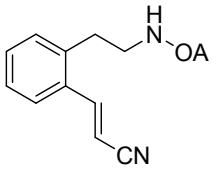
3o

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (d, J = 16.0 Hz, 1H), 7.62 (d, J = 6.6 Hz, 1H), 7.36–7.32 (m, 1H), 7.30–7.24 (m, 2H), 7.11 (br, 1H), 6.68 (d, J = 16.0 Hz, 1H), 4.74–4.68 (m, 1H), 3.54–3.45 (m, 3H), 3.05–3.01 (m, 2H), 281–2.76 (m, 2H), 1.41 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H), 1.17 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 201.38, 163.42, 162.90, 139.21, 138.36, 133.82, 130.63, 130.42, 128.39, 127.47, 126.92, 49.74, 46.70, 40.63, 33.97, 32.99, 20.98, 20.15, 8.30; HRMS Calcd for $C_{25}H_{30}N_2O_3$ [M+Na$^+$]: 381.2154; Found: 381.2167.



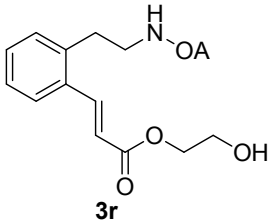
3p

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (d, J = 15.2 Hz, 1H), 7.57–7.55 (m, 1H), 7.35–7.31 (m, 1H), 7.27–7.25(m, 2H), 7.04 (br, 1H), 6.82 (d, J = 15.2 Hz, 1H), 4.68–4.61 (m, 1H), 3.53–3.48 (m, 3H), 3.20 (s, 3H), 3.09 (s, 3H), 3.03 (t, J = 7.3 Hz, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.66, 163.45, 163.13, 139.61, 137.88, 134.65, 130.58, 129.71, 127.25, 126.85, 119.95, 49.73, 46.58, 40.21, 37.57, 36.08, 32.95, 20.99, 20.16; HRMS Calcd for $C_{21}H_{31}N_3O_3$ [M+Na$^+$]: 396.2263; Found: 396.2275.



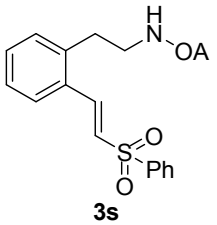
3q

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, J = 16.4 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 7.42-7.39 (m, 1H), 7.33–7.28 (m, 3H), 7.12 (br, 1H), 5.87 (d, J = 16.4 Hz, 1H), 4.81–4.75 (m, 1H), 3.57–3.47 (m, 3H), 2.99 (t, J = 7.3 Hz, 2H), 1.44 (d, J = 6.8 Hz, 6H), 1.25 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.34, 162.68, 147.83, 137.91, 132.80, 131.32, 130.79, 127.64, 126.23, 118.20, 98.47, 49.72, 46.77, 40.46, 32.74, 21.01, 20.16; HRMS Calcd for $C_{19}H_{25}N_3O_2$ [M+Na$^+$]: 350.1844; Found: 350.1857.



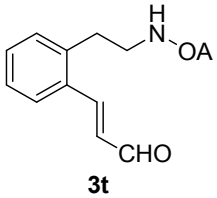
3r

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.19 (d, J = 15.9 Hz, 1H), 7.61 (d, J = 7.1 Hz, 1H), 7.42 (br, 1H), 7.35–7.23 (m, 3H), 6.41 (d, J = 15.8 Hz, 1H), 4.50–4.44 (m, 1H), 4.33–4.31 (m, 2H), 3.90 (s, 2H), 3.77 (s, 1H), 3.55–3.40 (m, 3H), 3.04-3.00 (m, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.99, 164.38, 163.52, 141.83, 138.01, 133.48, 130.66, 130.46, 127.49, 126.73, 119.95, 66.79, 60.88, 50.04, 46.53, 40.88, 33.22, 20.90, 20.11; HRMS Calcd for $C_{21}H_{30}N_2O_5$ [M+Na$^+$]: 413.2052; Found: 413.2070.



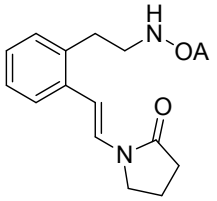
3s

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01–7.97 (m, 3H), 7.65–7.54 (m, 3H), 7.47 (d, J = 7.8 Hz, 1H), 7.38-7.34 (m, 1H), 7.28–7.23 (m, 2H), 7.12 (br, 1H), 6.84 (d, J = 15.2 Hz, 1H), 4.71–4.66 (m, 1H), 3.55–3.46 (m, 3H), 3.04 (t, J = 7.4 Hz, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.23 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.41, 162.99, 140.61, 139.47, 138.67, 133.56, 131.35, 131.26, 130.80, 129.50, 129.41, 127.87, 127.51, 127.42, 49.79, 46.64, 40.25, 32.86, 20.98, 20.15; HRMS Calcd for $C_{24}H_{30}N_2O_4S$ [M+Na$^+$]: 465.1824; Found: 465.1835.



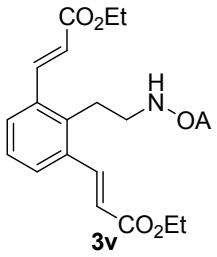
3t

^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.76 (d, J = 7.7 Hz, 1H), 7.97 (d, J = 15.7 Hz, 1H), 7.62 (d, J = 8.0 Hz, 1H), 7.50 (br, 1H), 7.38-7.34 (m, 1H), 7.30-7.26 (m, 2H), 6.67 (dd, J = 15.7, 7.7 Hz, 1H), 4.63-4.56 (m, 1H), 3.53-3.44 (m, 3H), 3.06-3.02 (m, 2H), 1.39 (d, J = 6.8 Hz, 6H), 1.20 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 194.35, 163.68, 163.28, 149.77, 138.57, 133.05, 131.29, 130.76, 130.31, 127.55, 127.14, 49.87, 46.63, 40.69, 32.88, 20.92, 20.10; HRMS Calcd for $C_{19}H_{26}N_2O_3$ [M+Na$^+$]: 353.1841; Found: 353.1860.



3u

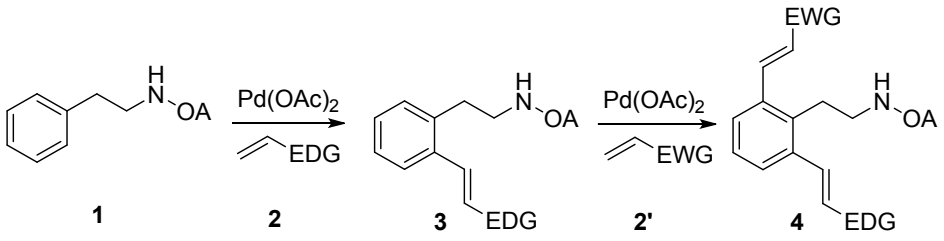
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56–7.50 (m, 2H), 7.22–7.15 (m, 3H), 6.98 (br, 1H), 6.26 (d, J = 14.6 Hz, 1H), 4.70–4.63 (m, 1H), 3.80–3.77 (m, 2H), 3.54–3.44 (m, 3H), 2.94-2.90 (m, 2H), 2.56 (t, J = 8.2 Hz, 2H), 2.22-2.14 (m, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 173.72, 163.57, 163.15, 135.57, 135.42, 130.08, 127.40, 127.09, 125.65, 125.29, 108.98, 49.79, 46.69, 45.59, 40.26, 33.46, 31.51, 20.99, 20.18, 17.63; HRMS Calcd for $C_{22}H_{31}N_3O_3$ [M+Na$^+$]: 408.2263; Found: 408.3386.



3v

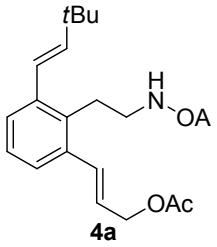
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.08 (d, J = 15.7 Hz, 2H), 7.58 (d, J = 7.8 Hz, 2H), 7.30 (d, J = 7.8 Hz, 1H), 7.17 (br, 1H), 6.36 (d, J = 15.7 Hz, 2H), 4.83–4.77 (m, 1H), 4.31–4.25 (m, 4H), 3.53–3.46 (m, 1H), 3.44–3.38 (m, 2H), 3.18–3.14 (m, 2H), 1.40 (d, J = 6.8 Hz, 6H), 1.35 (t, J = 7.1 Hz, 6H), 1.21 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.66, 163.12, 162.49, 141.67, 136.92, 135.15, 128.71, 127.57, 121.97, 60.82, 49.50, 46.73, 39.91, 28.72, 20.97, 20.08, 14.40; HRMS Calcd for $C_{26}H_{36}N_2O_6$ [M+Na$^+$]: 495.2471; Found: 495.2468.

6. Sequential Alkenylation of Oxalyl Amide Protected Phenylethylamine



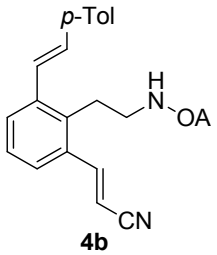
A mixture of oxalyl amide (0.5 mmol, 1 equiv), electron-deficient olefin **2** (2 equiv), $Pd(OAc)_2$ (11 mg, 0.05 equiv), Ag_2CO_3 (206.8 mg, 1.5 equiv), (n-BuO)$_2$PO$_2$H (31.5g, 0.3 equiv) and DCE

(1.0 mL) in a 15 mL glass tube (sealed with PTFE cap) was heated at 80 °C for 18 hours.Then after the mixture cooled down to rt, the electron rich olefin **2'** (2 equiv), Ag_2CO_3 (206.8 mg, 2 equiv), $(n\text{-BuO})_2PO_2H$ (31.5g, 0.3 equiv) were added into the mixture and reacted for another 24 h at 120 °C. The reaction mixture was cooled to room tempreture, and concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to give the sequential alkenylation product.



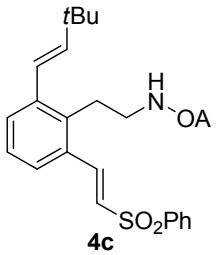
4a

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.34-7.32 (m, 2H), 7.16 (t, J = 7.7 Hz, 1H), 7.03 (d, J = 15.6 Hz, 1H), 6.63 (d, J = 15.8 Hz, 1H), 6.16–6.05 (m, 2H), 4.81–4.75 (m, 3H), 3.55–3.48 (m, 1H), 3.41–3.36 (m, 2H), 3.02-2.98 (m, 2H), 2.12 (s, 3H), 1.42 (d, J = 6.8 Hz, 6H), 1.22 (d, J = 6.7 Hz, 6H), 1.14 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.10, 163.21, 162.92, 145.56, 138.59, 136.40, 133.23, 132.20, 127.06, 126.74, 126.26, 125.65, 122.48, 65.34, 49.63, 46.72, 39.34, 33.86, 29.74, 28.91, 21.19, 21.02, 20.19; HRMS Calcd for $C_{27}H_{40}N_2O_4$ [M+Na$^+$]: 479.2886; Found: 479.2900.



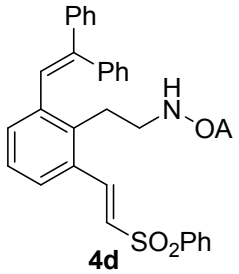
4b

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87 (d, J = 16.4 Hz, 1H), 7.68 (d, J = 7.5 Hz, 1H), 7.51-7.47 (m, 3H), 7.39 (d, J = 7.2 Hz, 1H), 7.29 (t, J = 7.8 Hz, 1H), 7.22 (br, 1H), 7.18 (d, J = 7.9 Hz, 2H), 6.97 (d, J = 16.0 Hz, 1H), 5.83 (d, J = 16.3 Hz, 1H), 4.93-4.86 (m, 1H), 3.54-3.49 (m, 1H), 3.44–3.38 (m, 2H), 3.14-3.10 (m, 2H), 2.37 (s, 3H), 1.41 (d, J = 6.8 Hz, 6H), 1.24 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.33, 162.33, 148.76, 138.70, 138.19, 135.06, 134.42, 133.83, 132.87, 129.58, 128.98, 127.64, 126.96, 125.51, 124.38, 118.16, 99.17, 49.64, 46.88, 39.92, 28.82, 21.44, 21.03, 20.14; HRMS Calcd for $C_{28}H_{33}N_3O_2$ [M+Na$^+$]: 466.2470; Found: 466.2486.



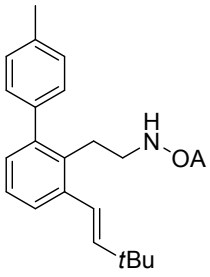
4c

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, J = 15.1 Hz, 1H), 8.00 – 7.98 (m, 2H), 7.63– 7.52 (m, 3H), 7.44 (d, J = 7.5 Hz, 1H), 7.31 (d, J = 7.0 Hz, 1H), 7.18 (t, J = 7.7 Hz, 2H), 6.80 (d, J = 15.1 Hz, 1H), 6.65 (d, J = 15.8 Hz, 1H), 6.07 (d, J = 15.8 Hz, 1H), 4.84 – 4.73 (m, 1H), 3.55-3.47 (m, 1H), 3.40 – 3.32 (m, 2H), 3.09-3.05 (m, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.24 (d, J = 6.7 Hz, 6H), 1.13 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.19, 162.83, 146.49, 140.71, 140.51, 139.59, 135.42, 133.51, 132.01, 129.90, 129.58, 129.48, 127.91, 127.35, 126.08, 122.05, 49.73, 46.72, 39.56, 33.94, 29.63, 29.04, 21.01, 20.18; HRMS Calcd for $C_{30}H_{30}N_2O_4S$ [M+Na$^+$]: 547.2606; Found: 547.2634.



4d

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 – 8.00 (m, 3H), 7.64 – 7.60 (m, 1H), 7.57 – 7.53 (m, 2H), 7.38-7.35 (m, 2H), 7.34 – 7.31 (m, 3H), 7.26 – 7.23 (m, 1H), 7.20-7.17 (m, 3H), 7.14-7.11 (m, 2H), 7.05-7.02 (m, 2H), 6.93-6.88 (m, 2H), 6.80 (d, J = 15.1 Hz, 1H), 4.88-4.78 (m, 1H), 3.55-3.48 (m, 1H), 3.48 – 3.41 (m, 2H), 3.15 – 3.11 (m, 2H), 1.42 (d, J = 6.8 Hz, 6H), 1.21 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.02, 162.55, 145.42, 142.85, 140.77, 140.33, 139.59, 138.86, 137.22, 133.51, 132.01, 130.77, 129.84, 129.49, 128.37, 128.29, 128.01, 127.99, 127.51, 126.71, 126.21, 125.66, 49.63, 46.79, 39.64, 27.55, 21.03, 20.17; HRMS Calcd for $C_{38}H_{40}N_2O_4S$ [M+Na$^+$]: 643.2606; Found: 643.2601.



5k

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.19–7.12 (m, 4H), 6.95 (d, J = 2.7 Hz, 1H), 6.67 – 6.61 (m, 3H), 6.13 (d, J = 15.8 Hz, 1H), 4.69–4.63 (m, 1H), 3.78 (s, 3H), 3.49–3.43 (m, 1H), 3.25–3.19 (m, 2H), 2.79–2.75 (m, 2H), 2.38 (s, 3H), 1.37 (d, J = 6.8 Hz, 6H), 1.13–1.13 (m, 15H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.01, 162.72, 157.66, 145.28, 144.28, 139.64, 139.20, 136.78, 129.02, 125.73, 122.86, 114.36, 111.42, 55.38, 49.51, 46.60, 39.68, 33.86, 29.72, 28.77, 21.30, 20.94, 20.15; HRMS Calcd for $C_{28}H_{42}N_2O_5$ [M+Na$^+$]: 509.2991; Found: 509.3002.

7. References

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[4] Berry, J. M.; Bradshaw, T. D.; Fichtner, I.; Ren, R.; Schwalbe, C. H.; Wells, G.; Stevens, M. G.

and Westwell, A. D. *J. Med. Chem.* **2005**, *48*, 639.
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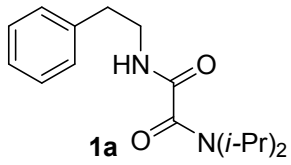
[6]J. Han; P. Liu; C. Wang; Q. Wang; J.-Y. Zhang; Y.-W. Zhao; D.-Q. Shi; Z.-B. Huang; Y.-S. Zhao, *Org. Lett.* **2014**, *16*, 5682-5685.

8. NMR spectra



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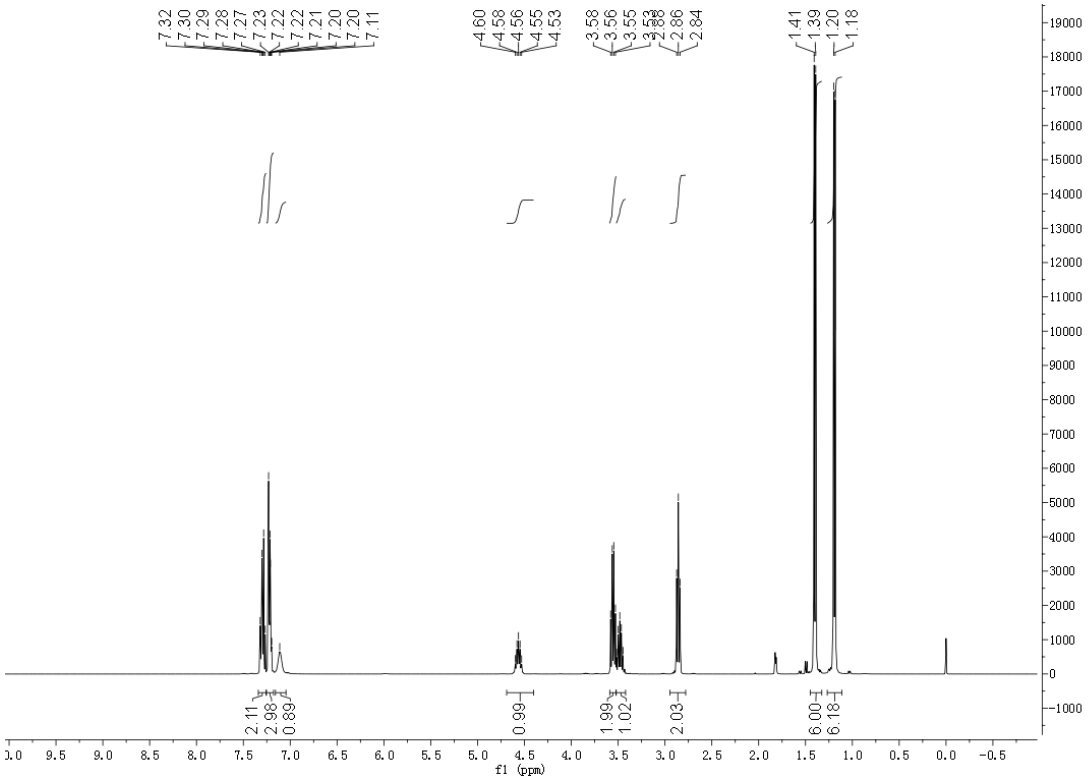

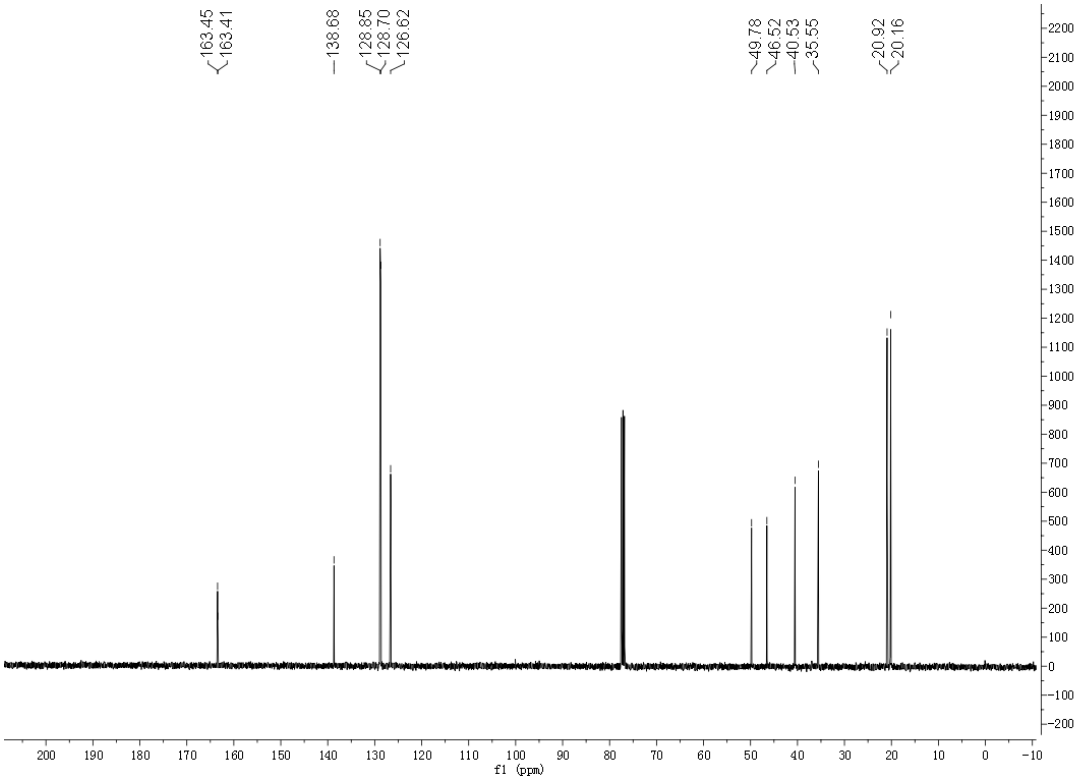

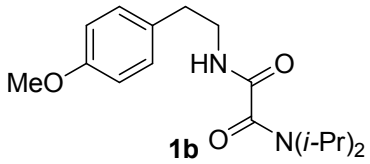
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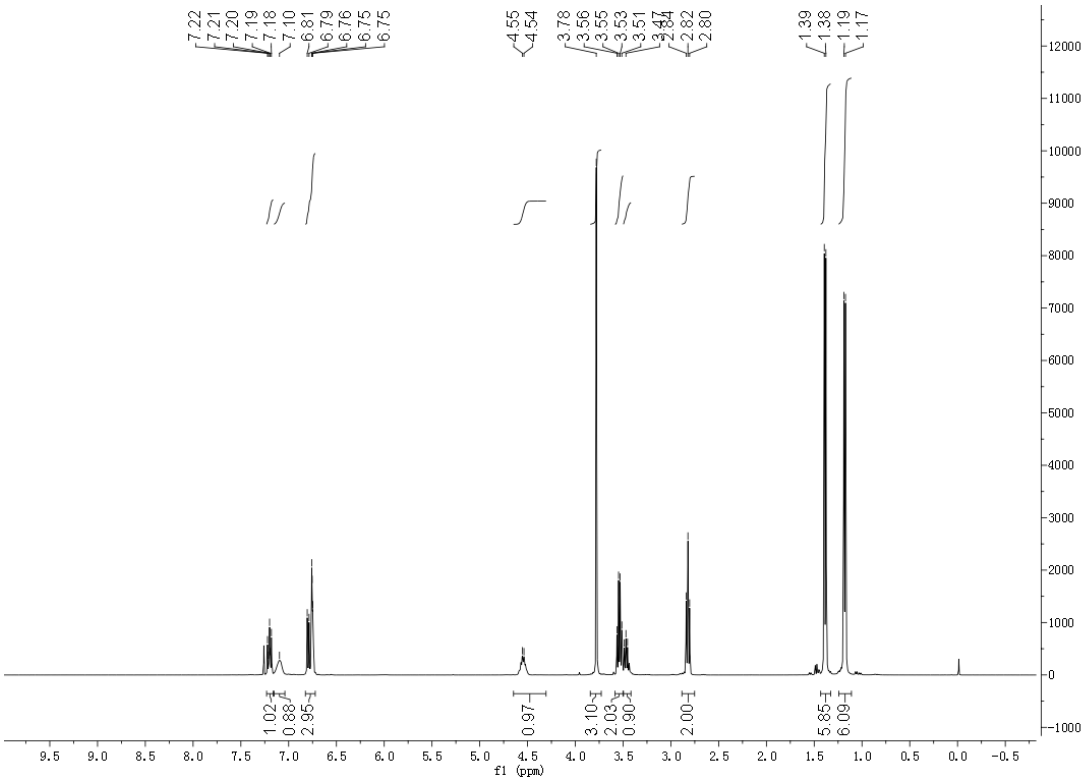
O
N(i-Pr)2

1b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2.82
2.80
1.39
1.38
1.19
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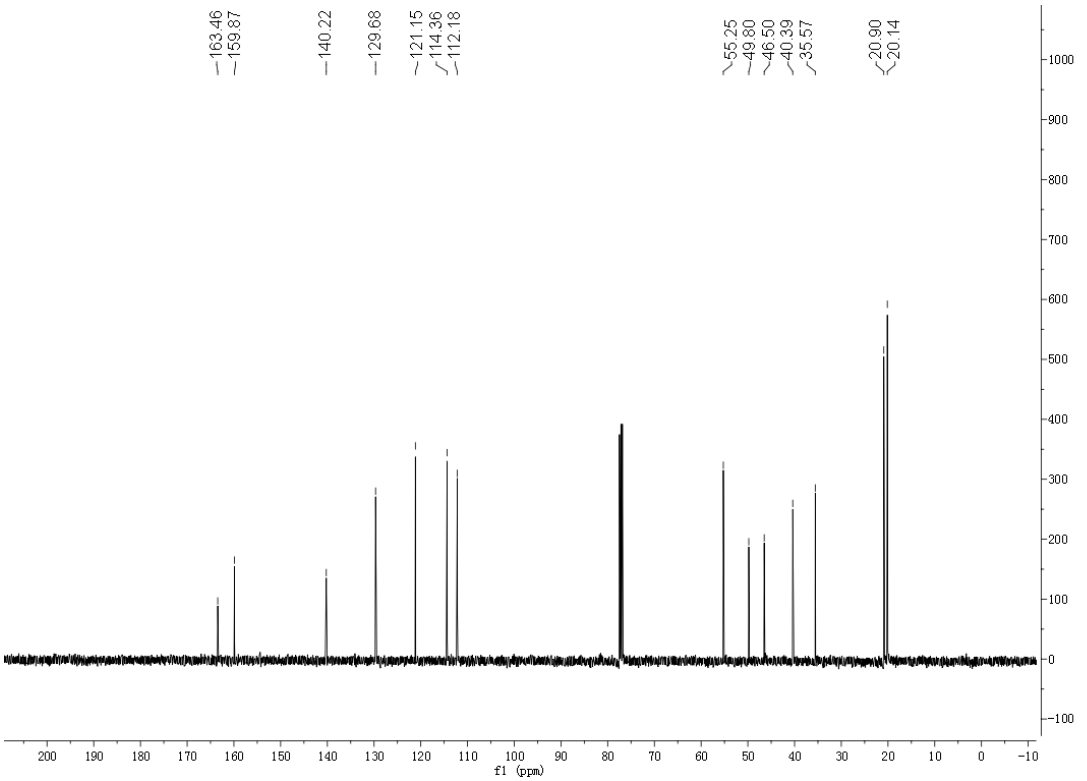
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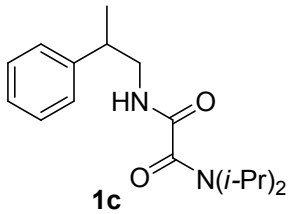
f1 (ppm)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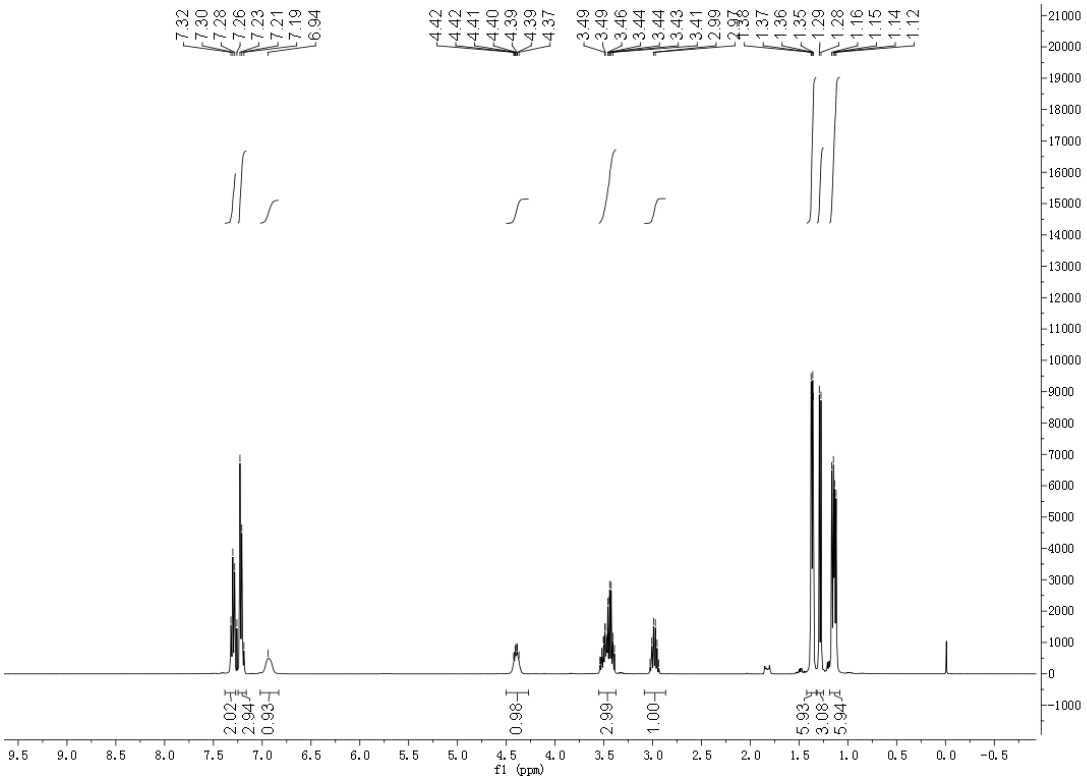
N(i-Pr)2

1c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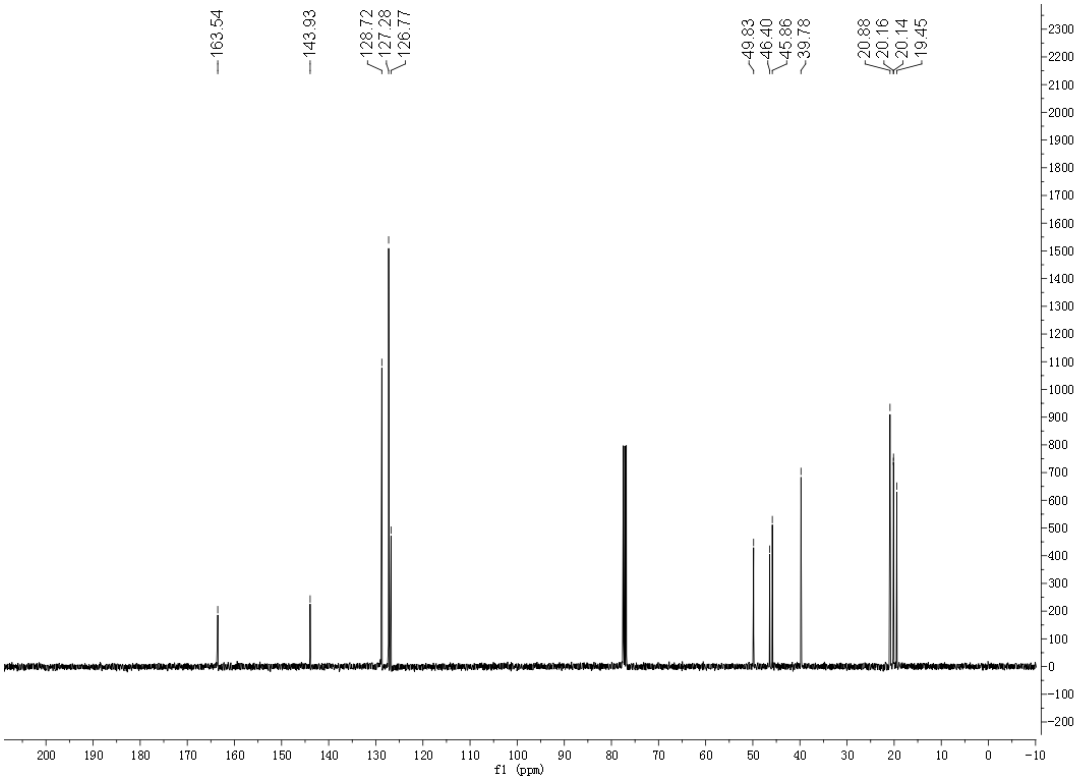
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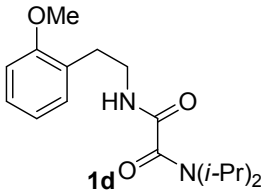
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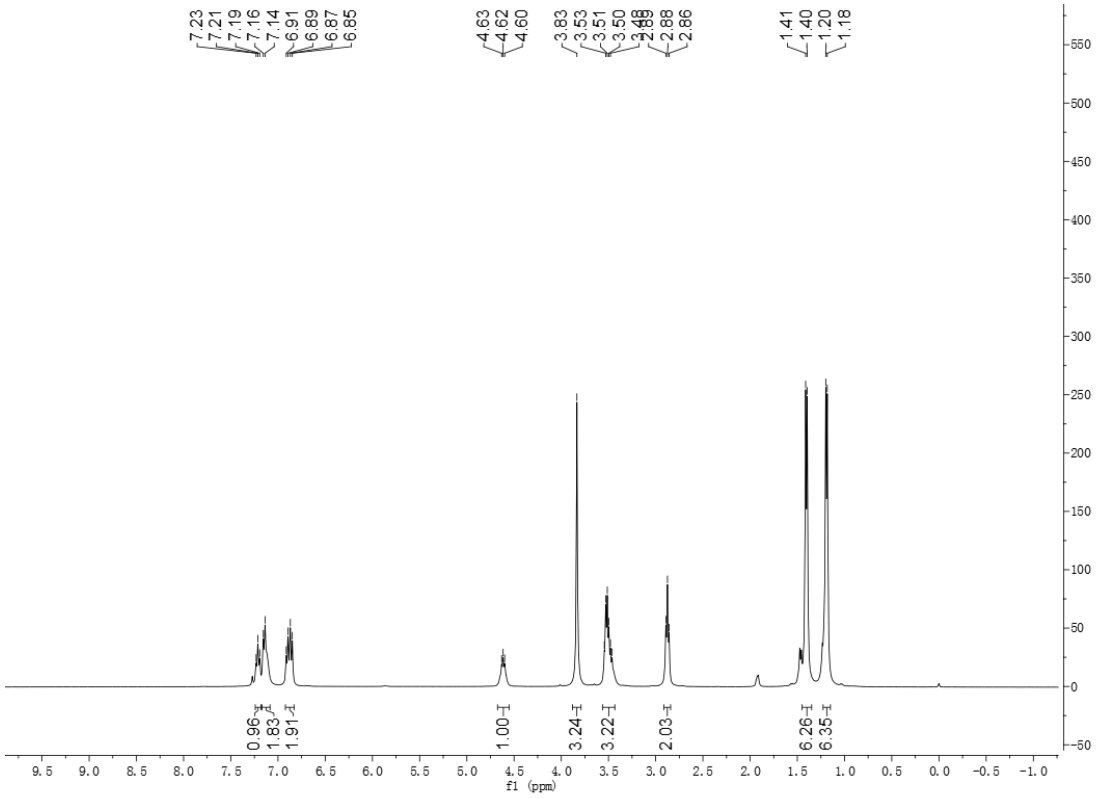
1d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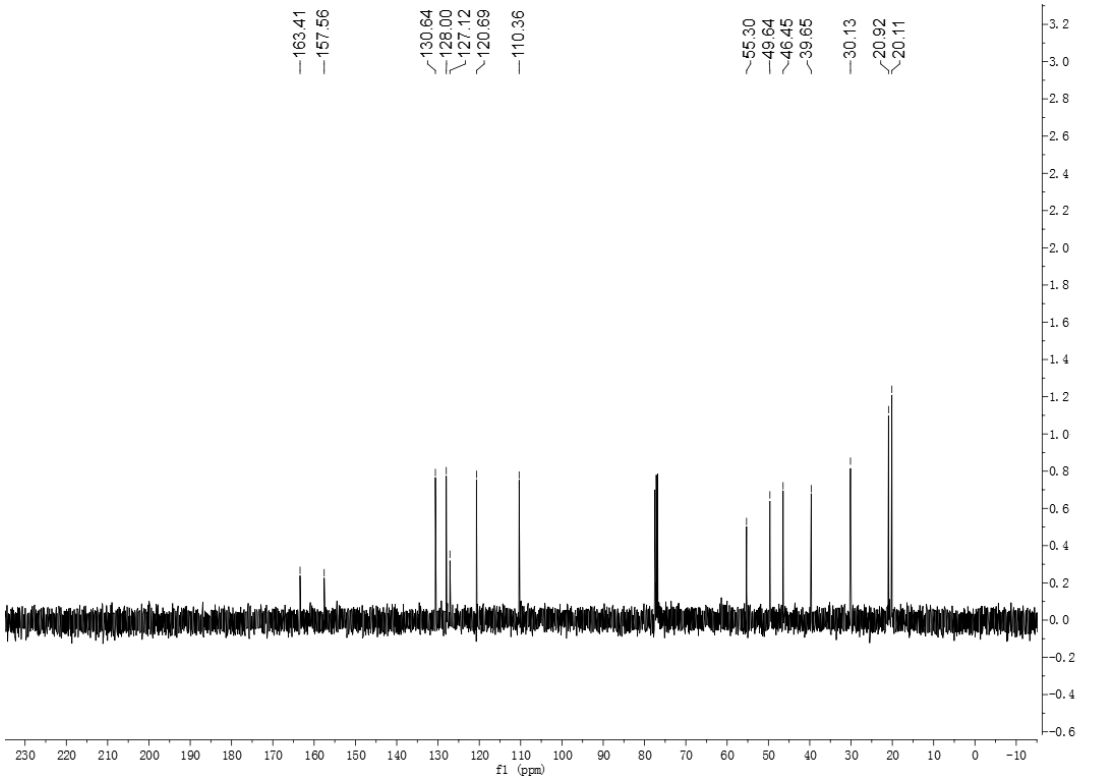
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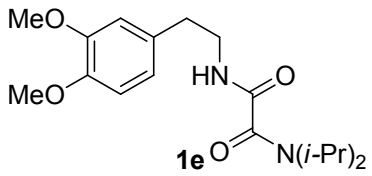
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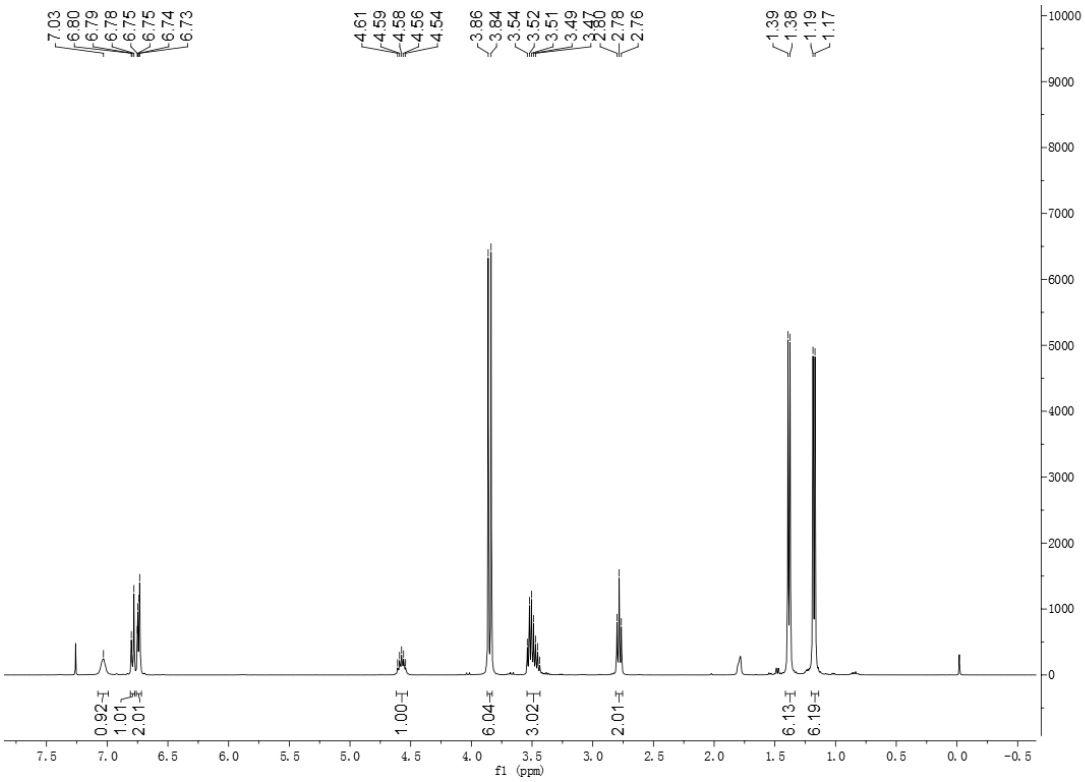
O
O
N(i-Pr)2

1e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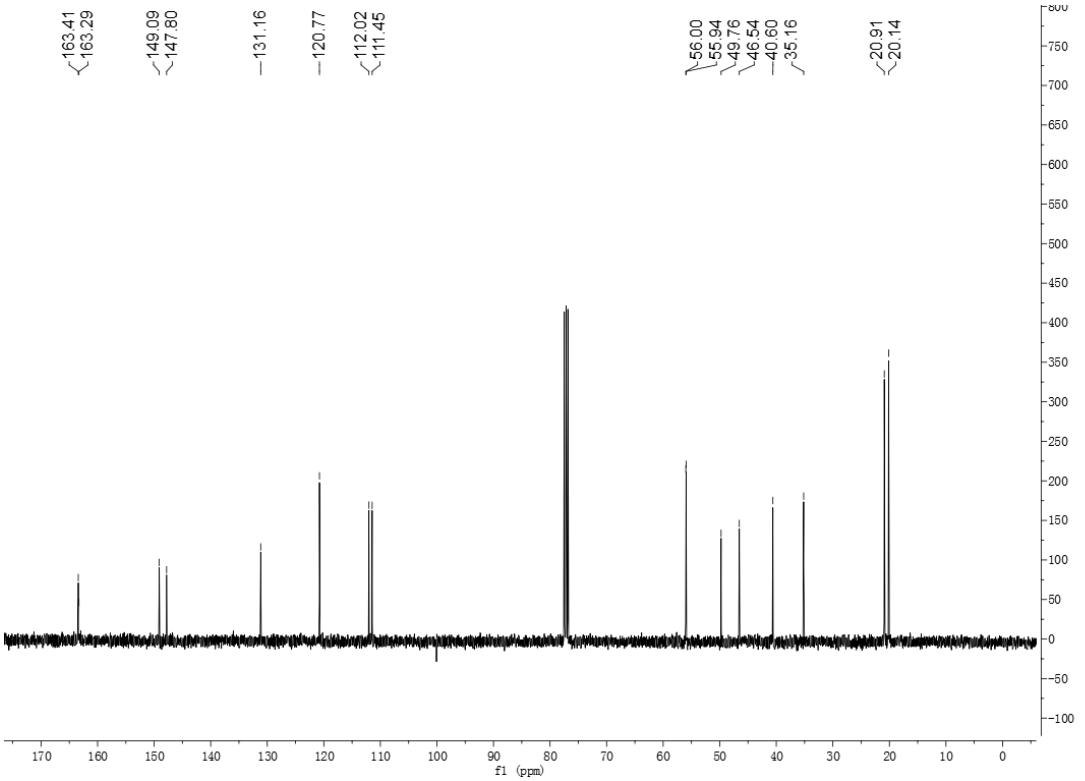
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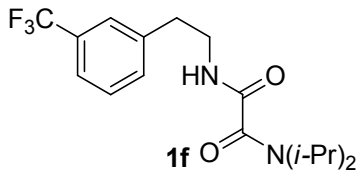
f1 (ppm)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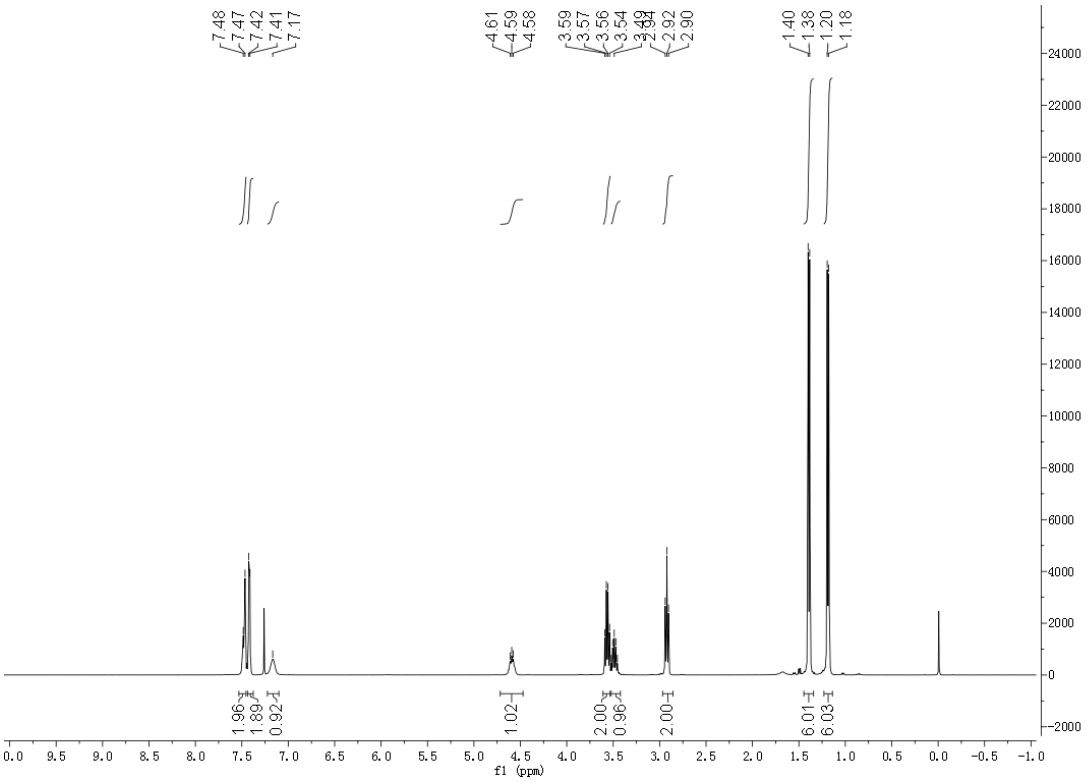
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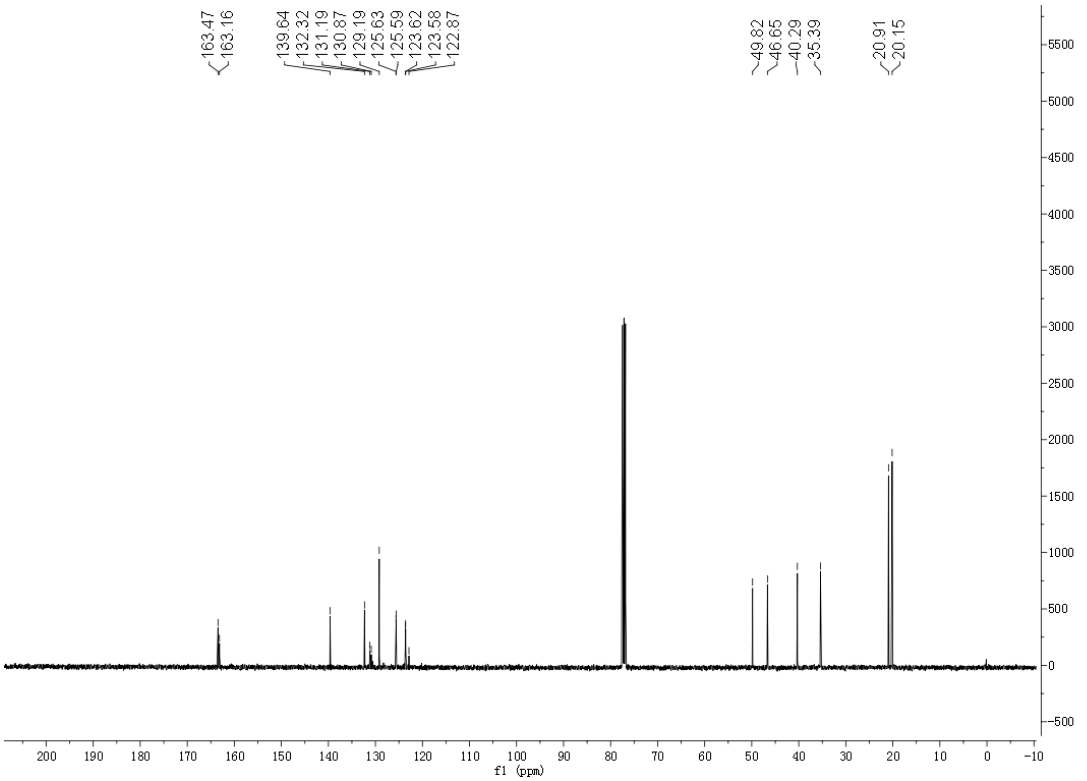
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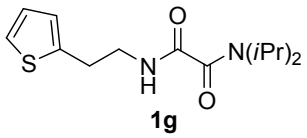
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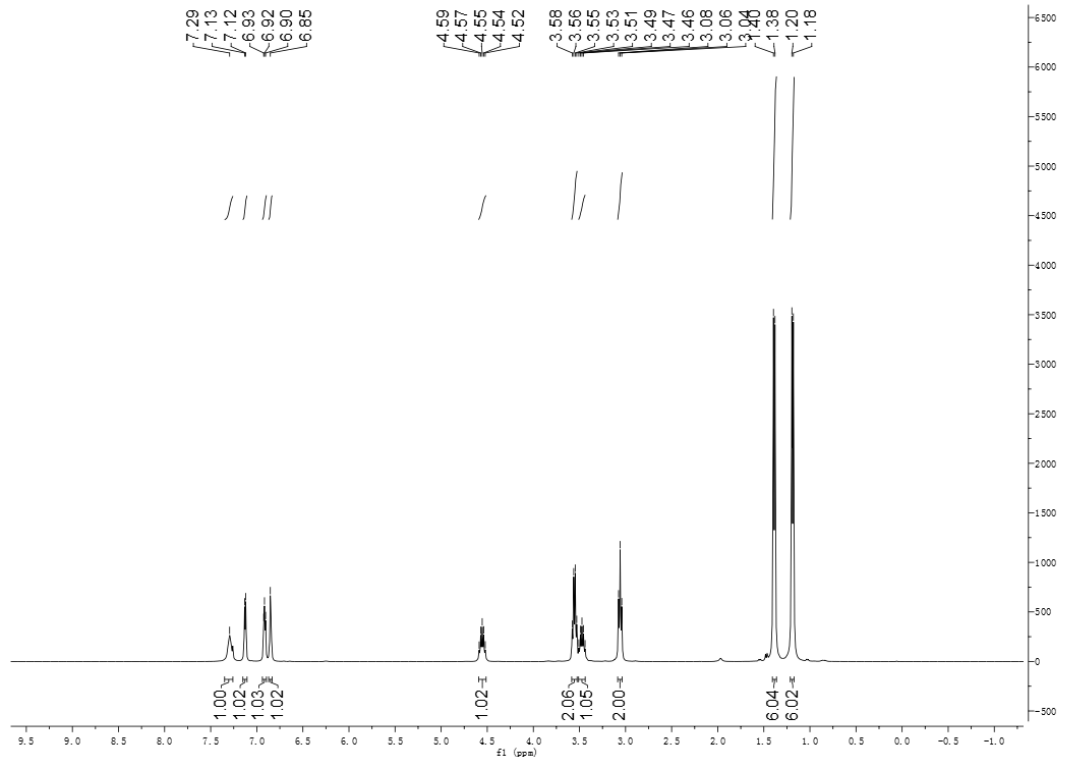
1g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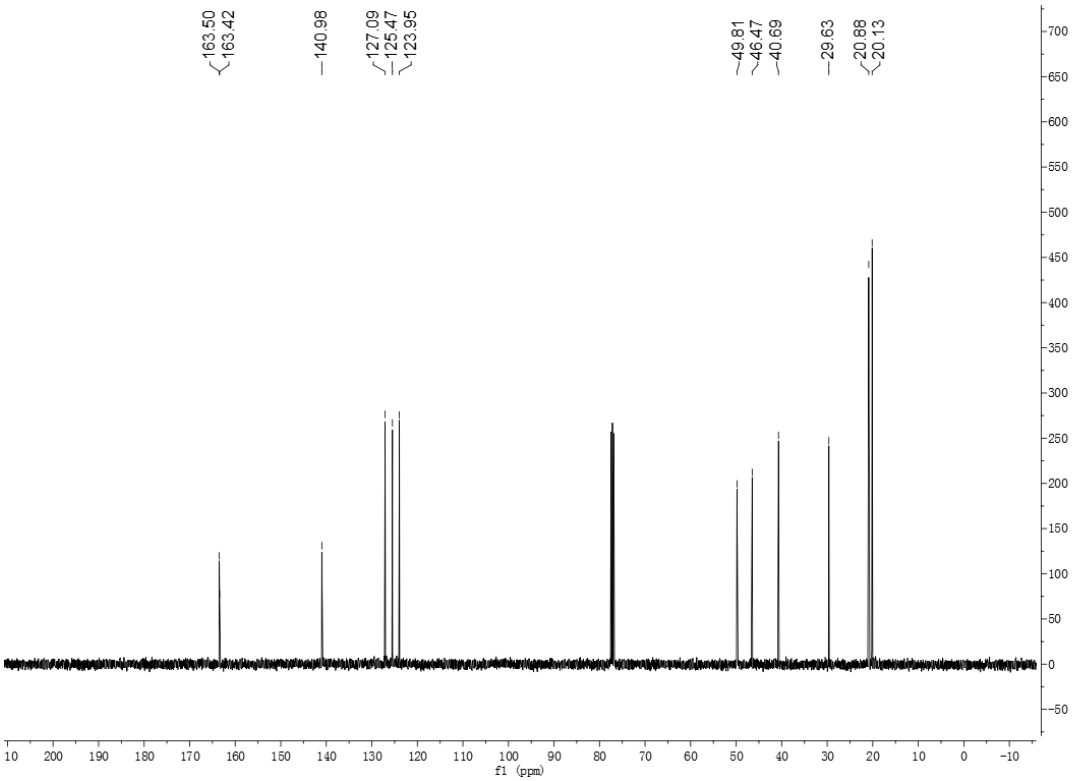
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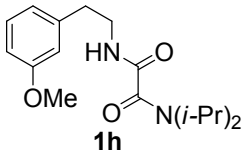
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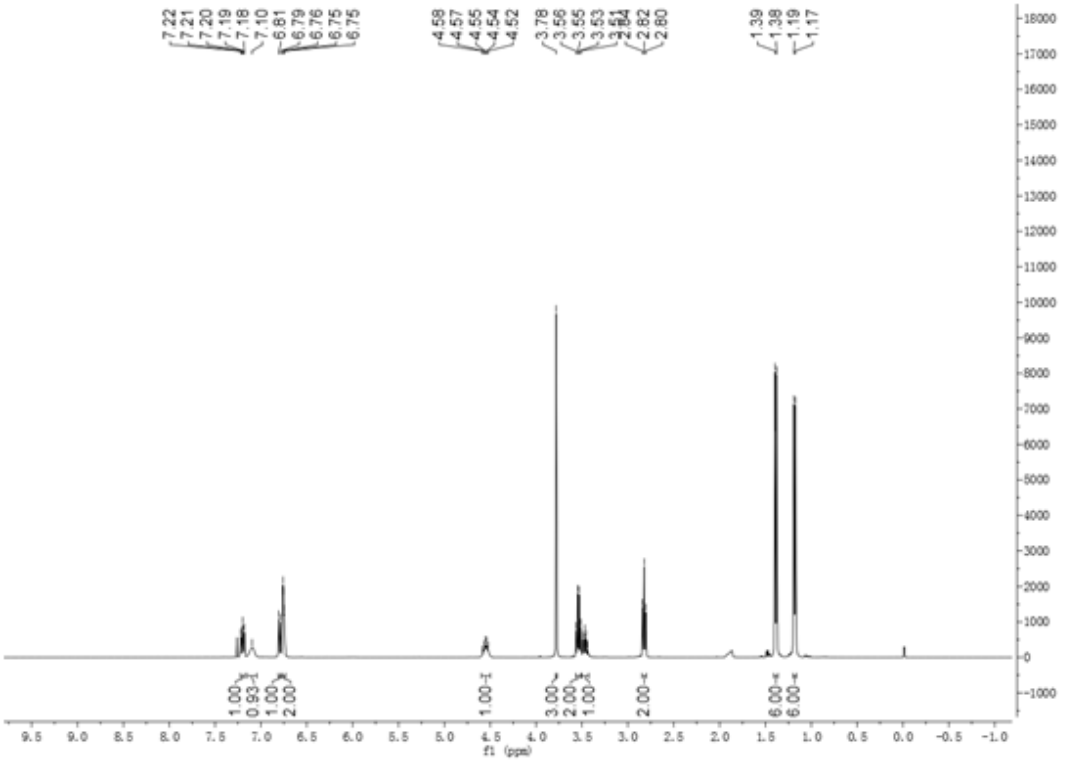
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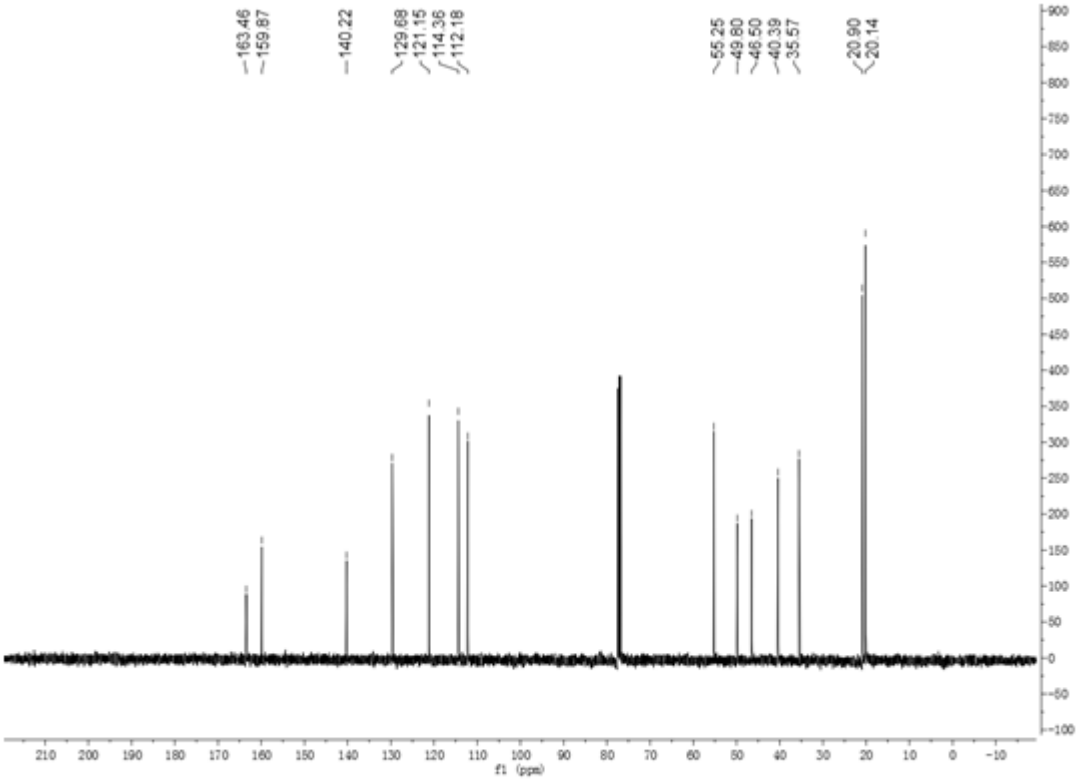
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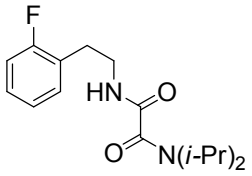
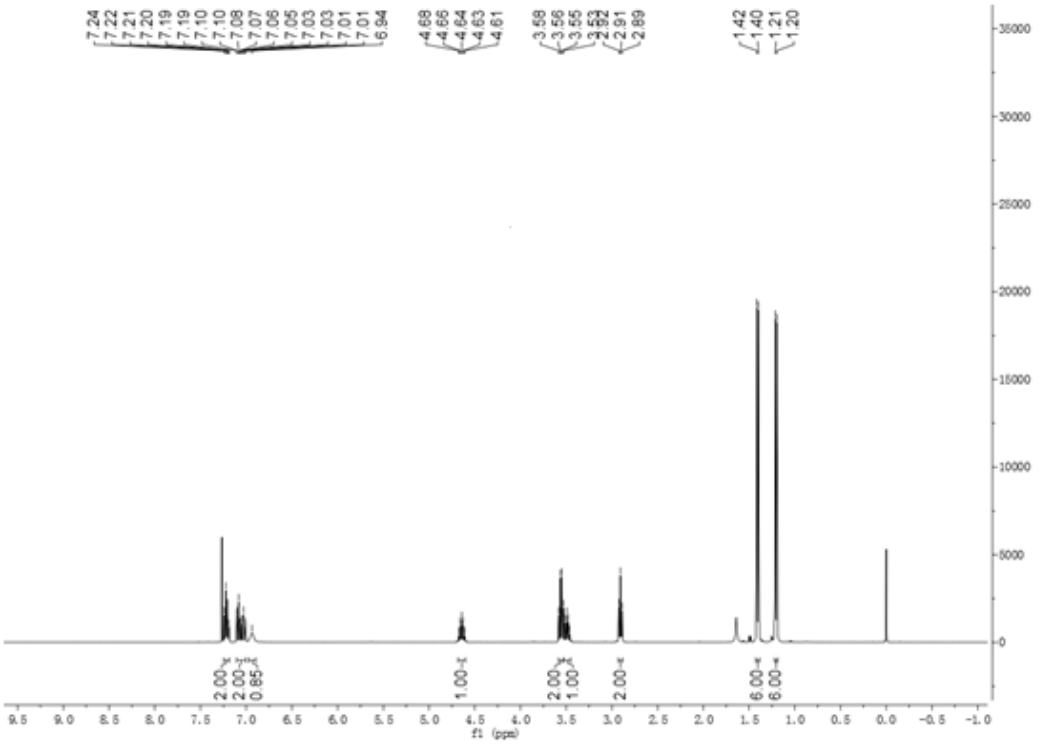
20.90
20.14
f1 (ppm)


F
HN
O
O
N(i-Pr)2

1i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7.03
7.03
7.01
7.01
6.94
4.68
4.66
4.64
4.63
4.61
3.58
3.56
3.55
3.53
2.92
2.91
2.89
1.42
1.40
1.21
1.20
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2.00
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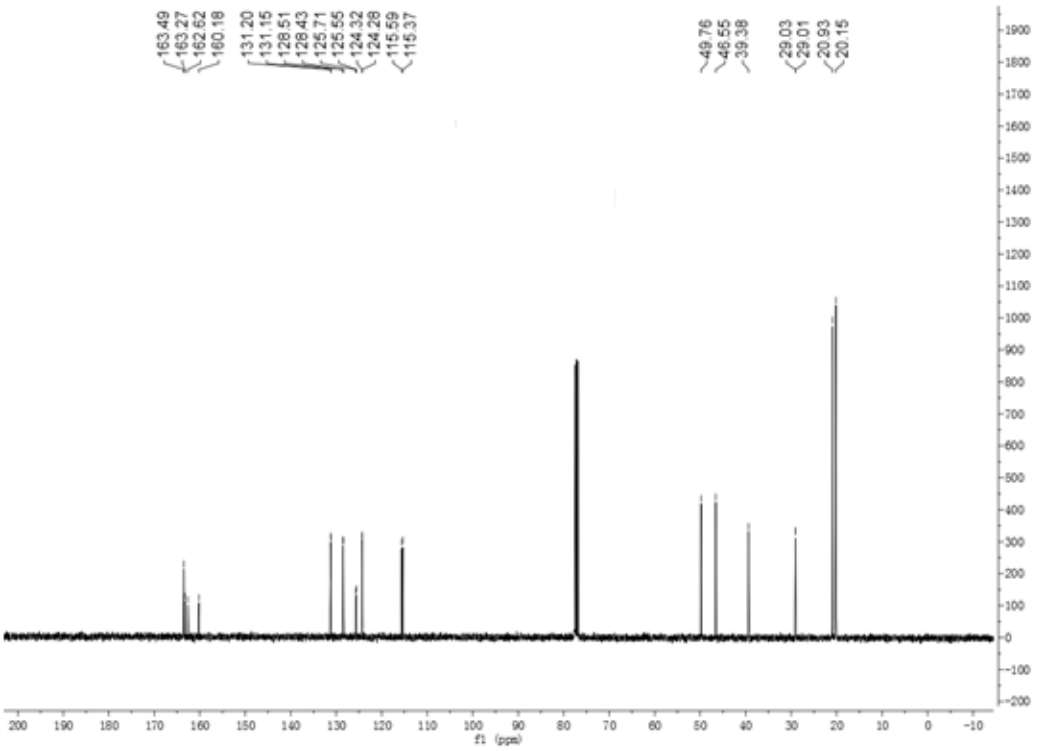
1.00
2.00
1.00
2.00
6.00
6.00
f1 (ppm)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125.55
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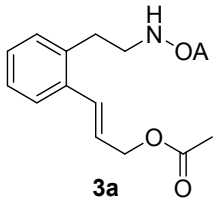
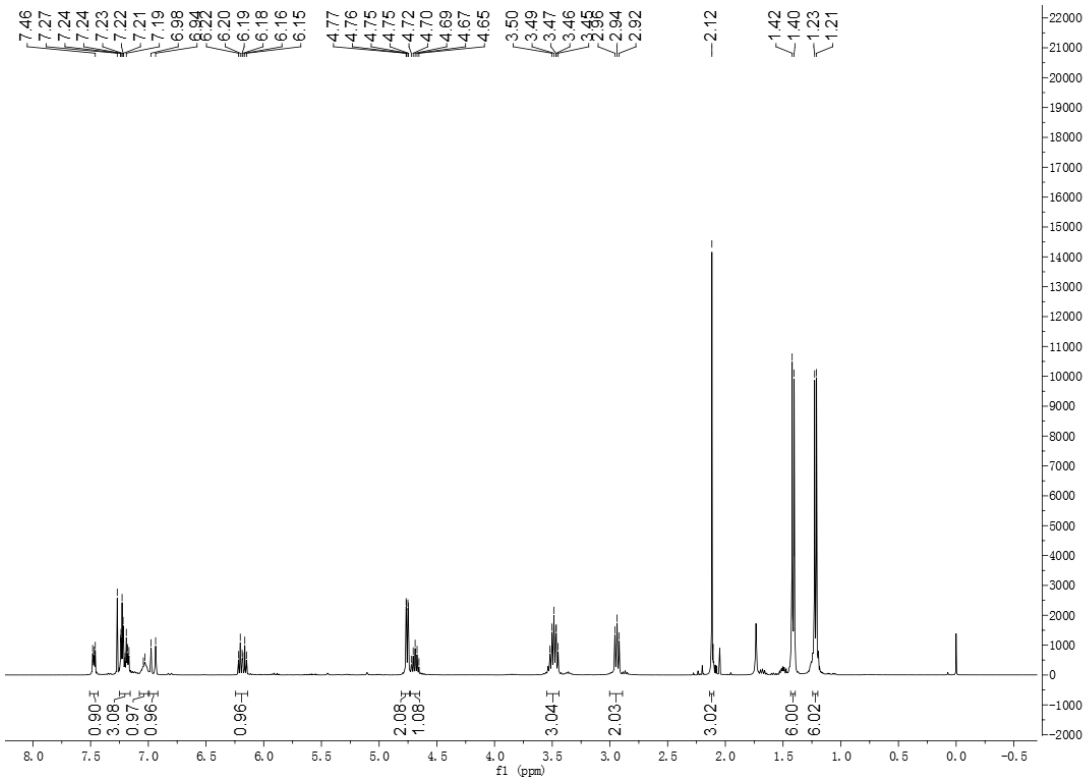
46.55
39.38
29.03
29.01
20.93
20.15
f1 (ppm)


H
N
OA
O
O

3a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7.19
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6.20
6.19
6.18
6.16
6.15
4.77
4.76
4.75
4.75
4.72
4.70
4.69
4.67
4.65
3.50
3.49
3.47
3.46
2.94
2.92
2.12
1.42
1.40
1.23
1.21
0.90
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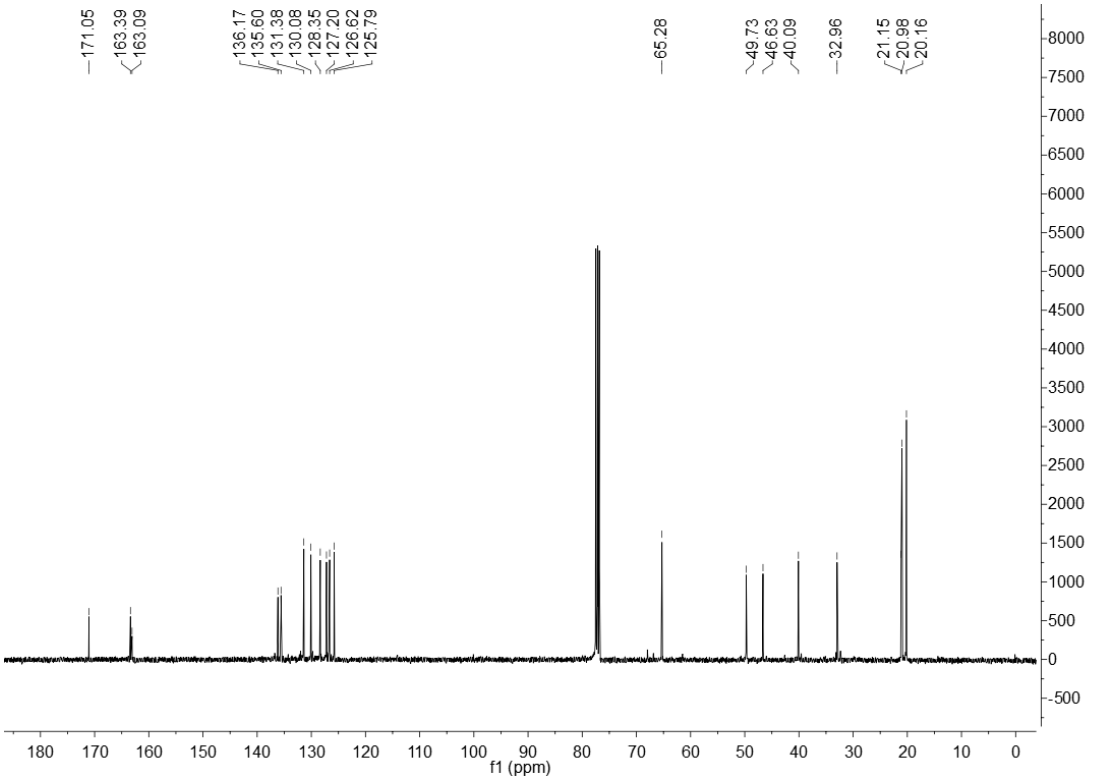
0.96
2.08
1.08
3.04
2.03
3.02
6.00
6.02
f1 (ppm)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131.38
130.08
128.35
127.20
126.62
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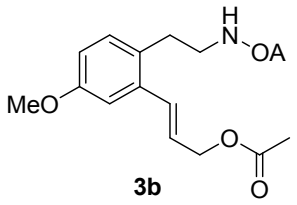
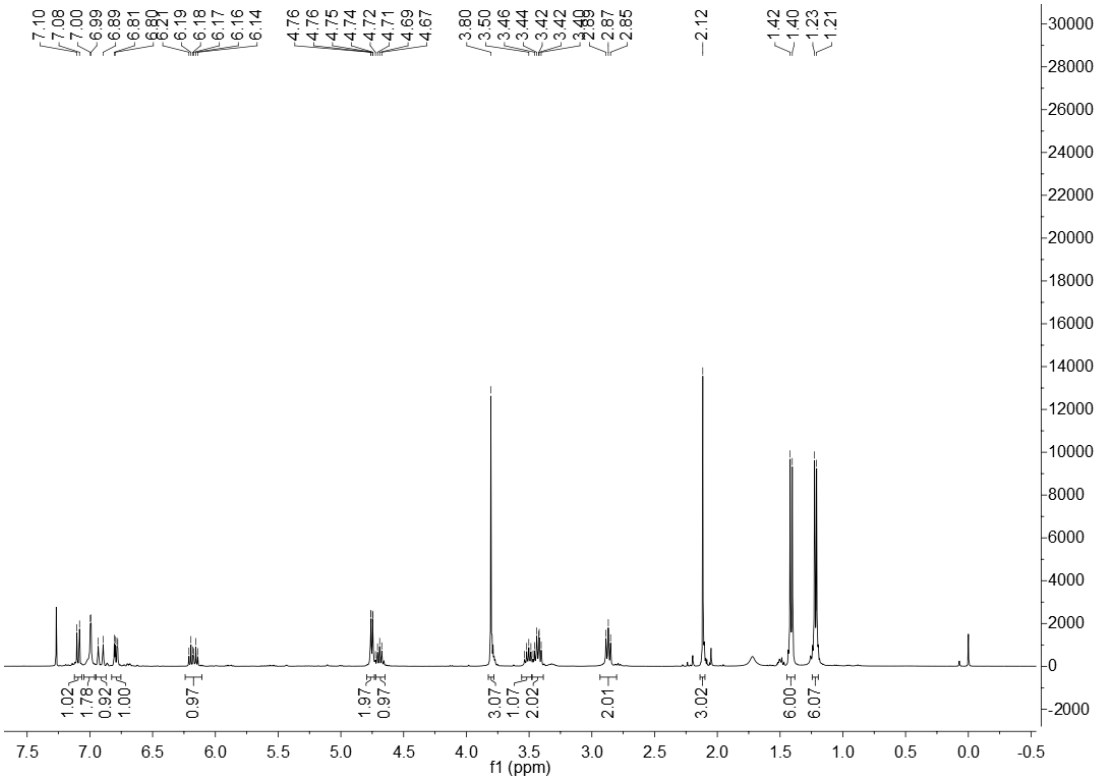
65.28
49.73
46.63
40.09
32.96
21.15
20.98
20.16
f1 (ppm)


H
N
OA
MeO
O
O

3b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7.08
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6.81
6.80
6.21
6.19
6.18
6.17
6.16
6.14
4.76
4.76
4.75
4.74
4.72
4.71
4.69
4.67
3.80
3.50
3.46
3.44
3.42
3.42
2.89
2.87
2.85
2.12
1.42
1.40
1.23
1.21
1.02
1.78
0.92
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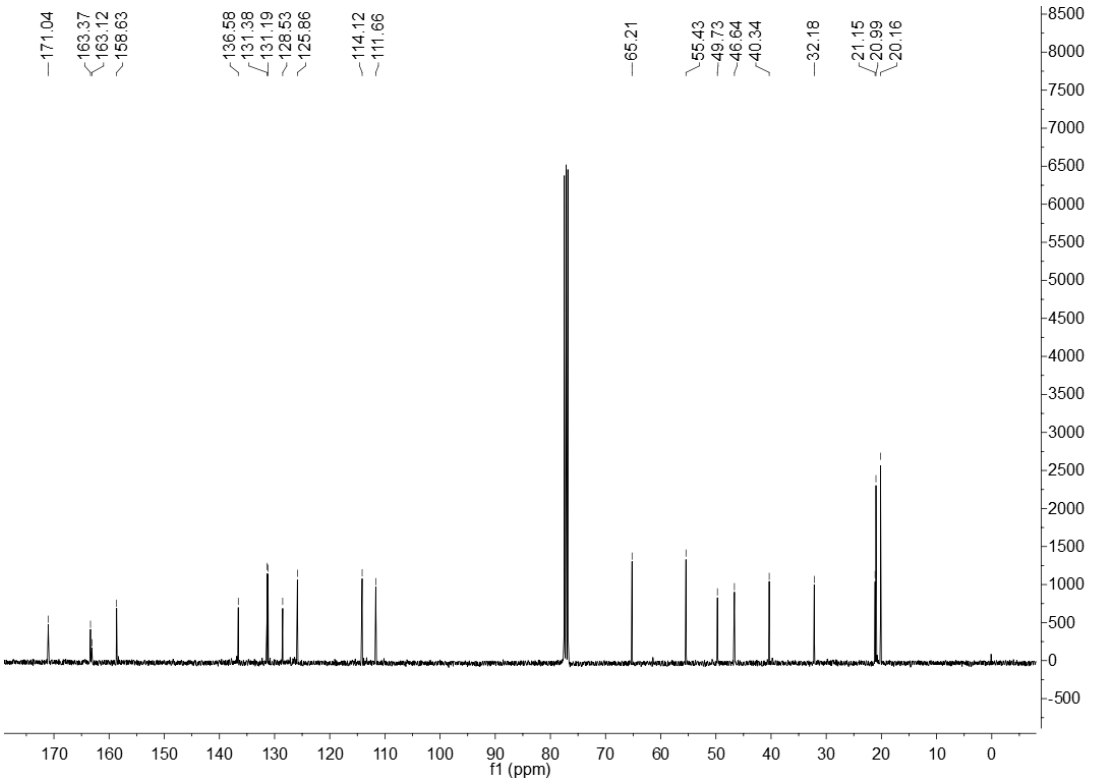
1.97
0.97
3.07
1.07
2.02
2.01
3.02
6.00
6.07
f1 (ppm)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131.19
128.53
125.86
114.12
111.66
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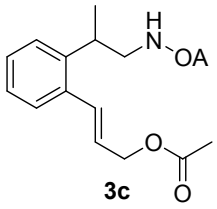
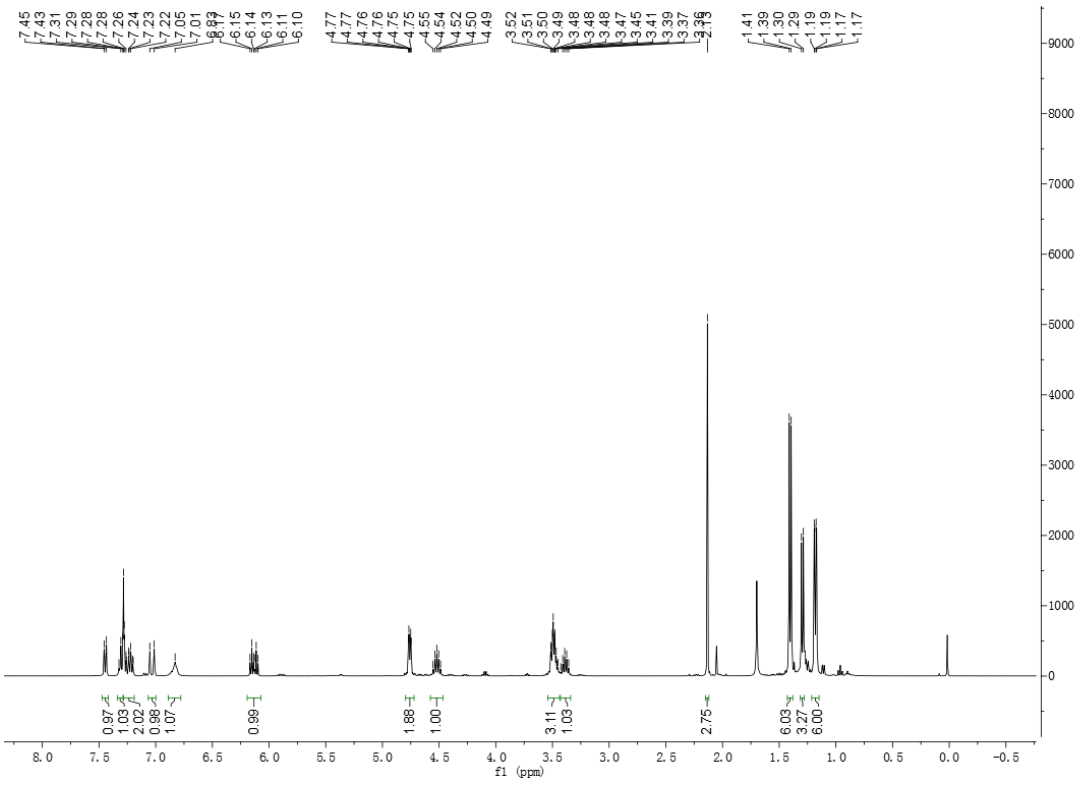
55.43
49.73
46.64
40.34
32.18
21.15
20.99
20.16
f1 (ppm)


H
N
OA
O
O

3c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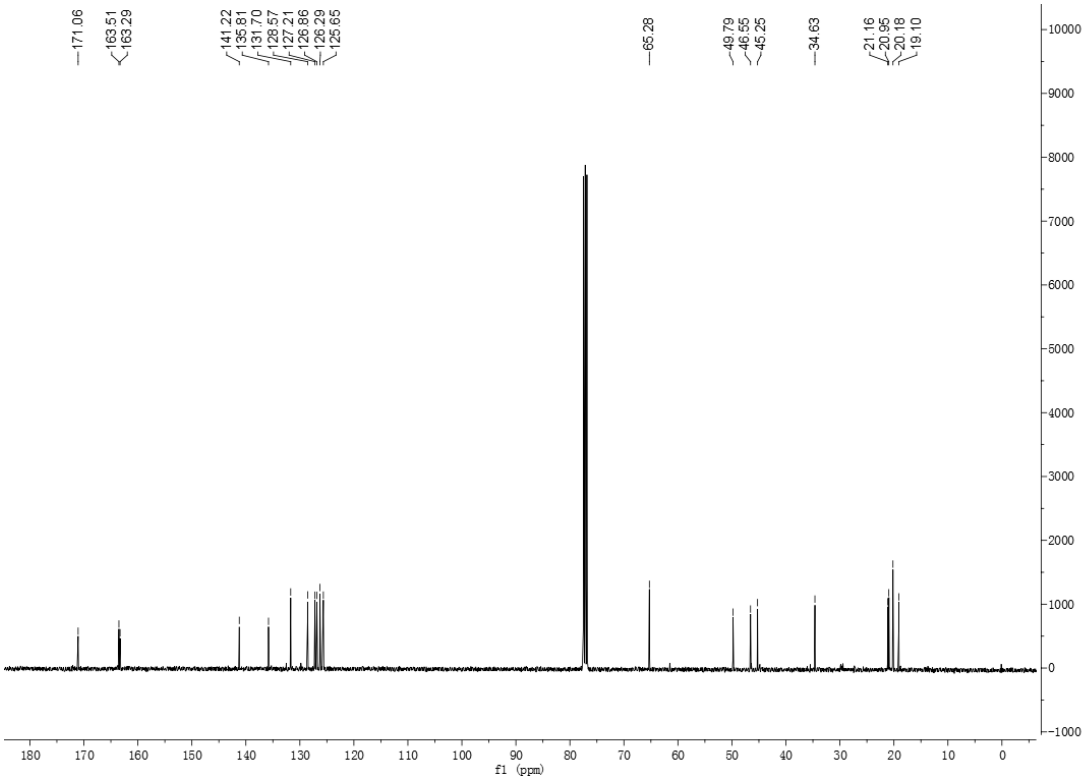
7.28
7.28
7.26
7.24
7.23
7.22
7.05
7.01
6.83
6.15
6.14
6.13
6.11
6.10
4.77
4.77
4.76
4.76
4.75
4.75
4.55
4.54
4.52
4.50
4.49
3.52
3.51
3.50
3.49
3.48
3.48
3.47
3.45
3.41
3.39
3.37
3.36
2.13
1.41
1.39
1.30
1.29
1.19
1.19
1.17
1.17
0.97
1.03
2.02
0.98
1.07
0.99
1.88
1.00
3.11
1.03
2.75
6.03
3.27
6.00
f1 (ppm)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127.21
126.86
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125.65
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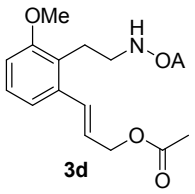
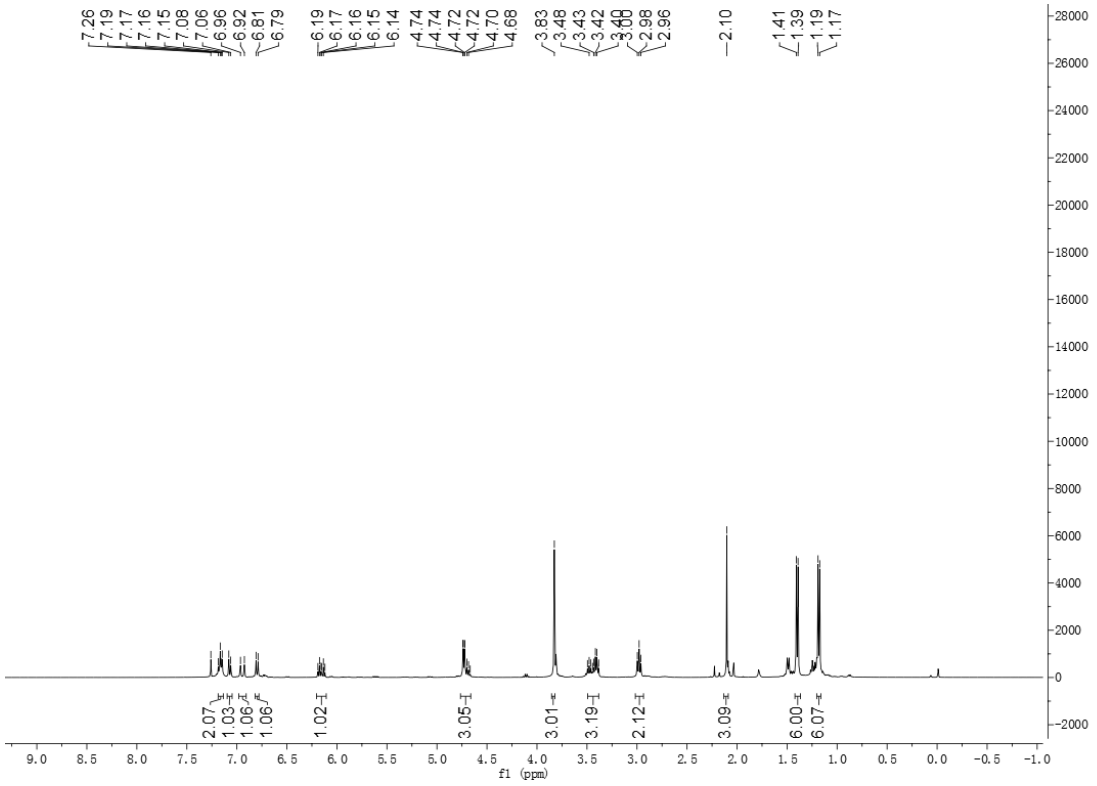
45.25
34.63
21.16
20.95
20.18
19.10
f1 (ppm)


OMe
H
N
OA
O
O
3d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7.16
7.15
7.08
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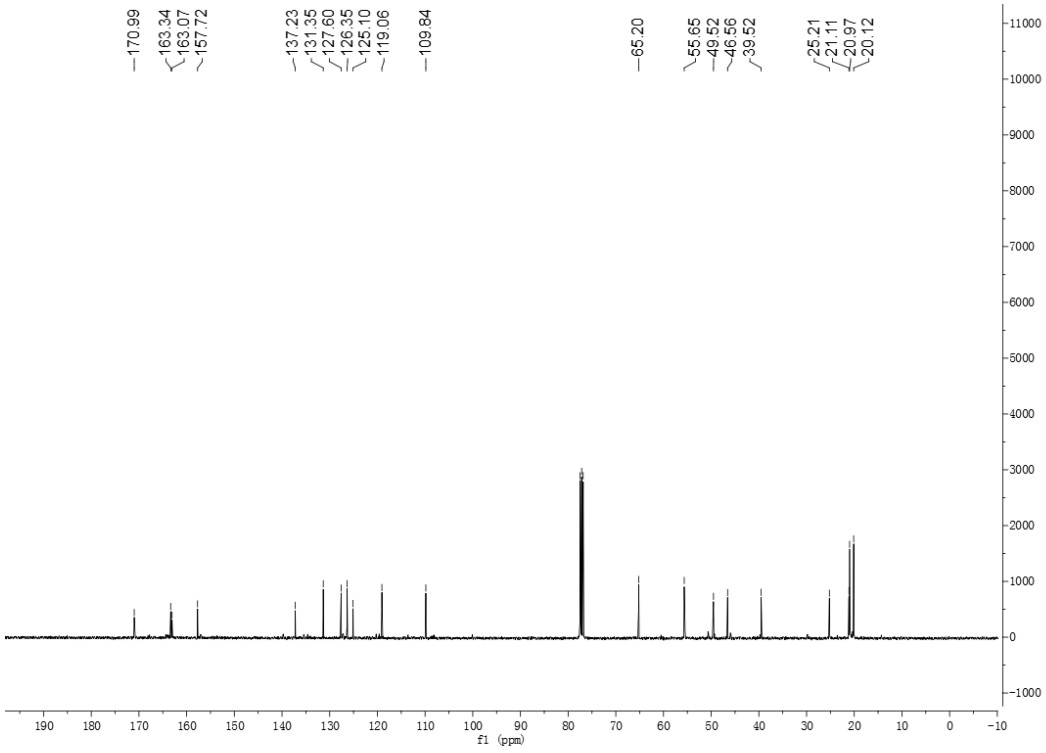
6.92
6.81
6.79
6.19
6.17
6.16
6.15
6.14
4.74
4.74
4.72
4.72
4.70
4.68
3.83
3.48
3.43
3.42
2.98
2.96
2.10
1.41
1.39
1.19
1.17
2.07
1.03
1.06
1.06
1.02
3.05
3.01
3.19
2.12
3.09
6.00
6.07
f1 (ppm)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126.35
125.10
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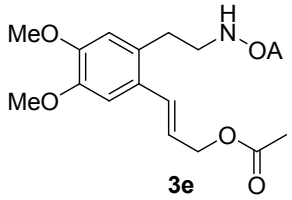
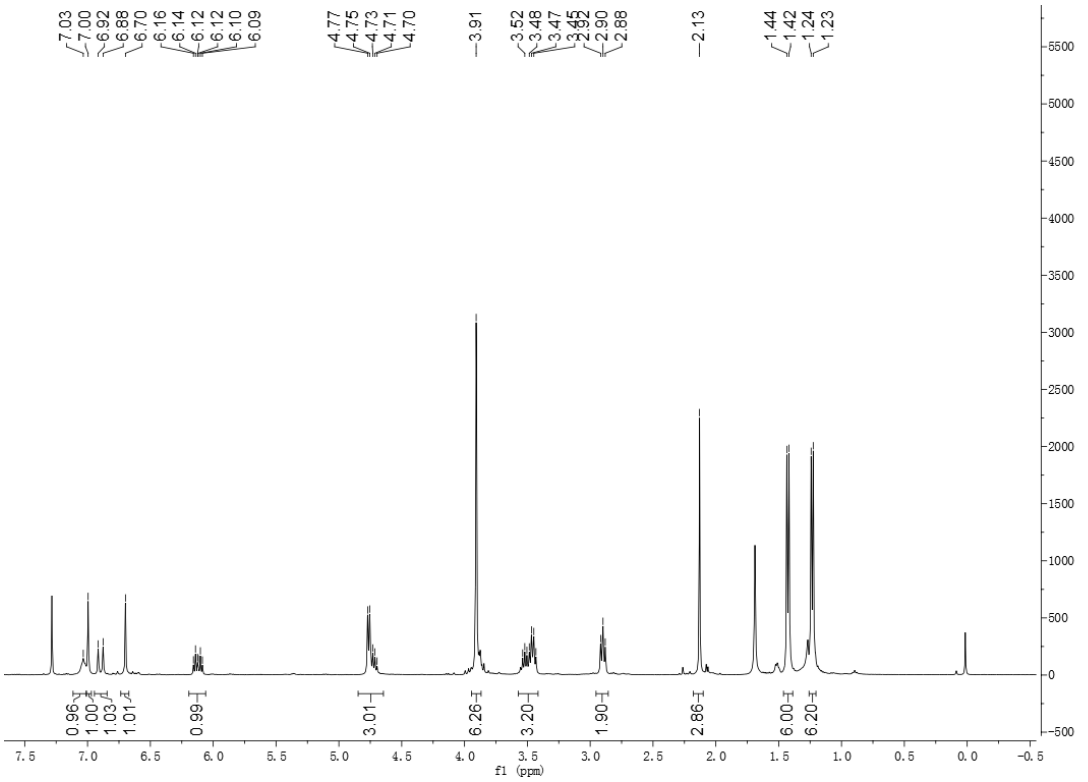
55.65
49.52
46.56
39.52
25.21
21.11
20.97
20.12
f1 (ppm)


MeO
MeO
H
N
OA
O
O

3e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6.16
6.14
6.12
6.12
6.10
6.09
4.77
4.75
4.73
4.71
4.70
3.91
3.52
3.48
3.47
2.90
2.88
2.13
1.44
1.42
1.24
1.23
0.96
1.00
1.03
1.01
0.99
3.01
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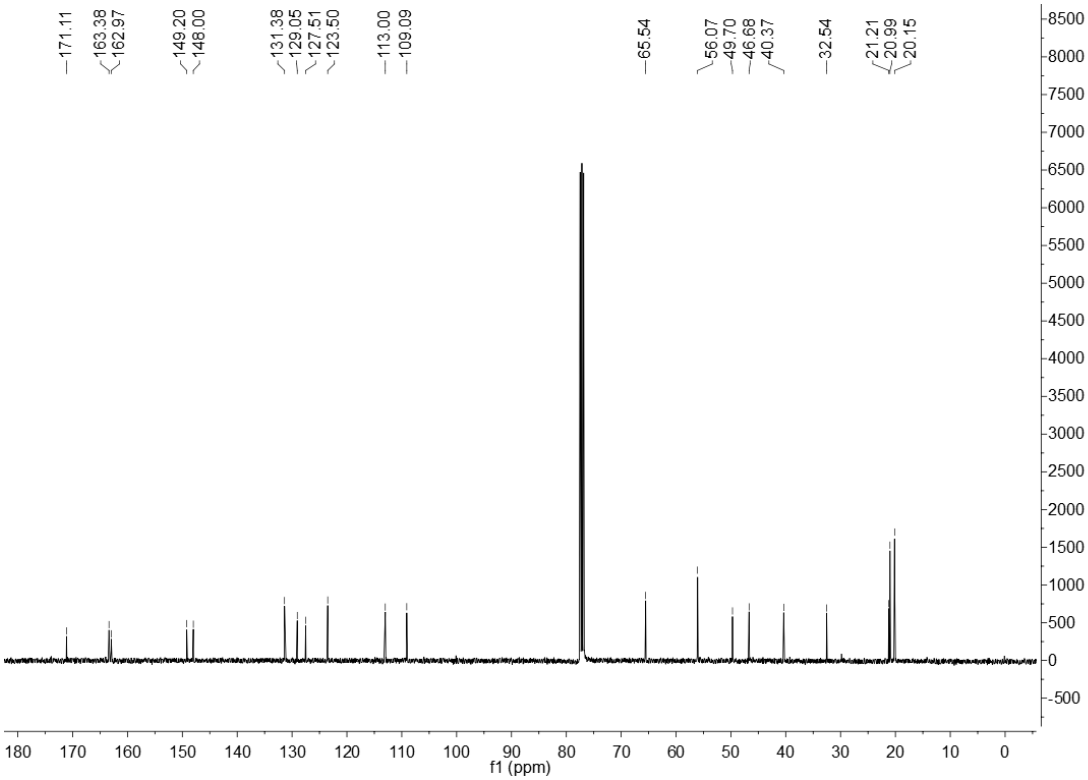
6.00
6.20
f1 (ppm)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129.05
127.51
123.50
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109.09
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56.07
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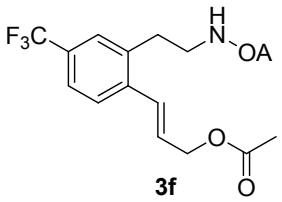
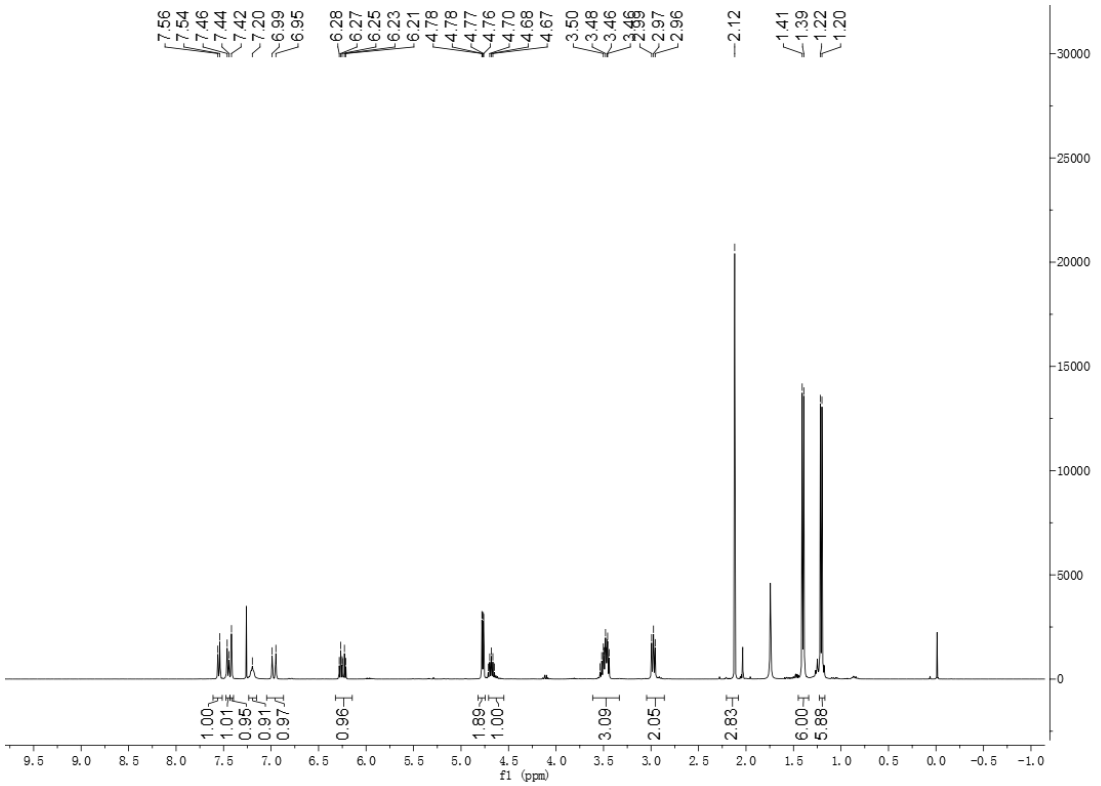
46.68
40.37
32.54
21.21
20.99
20.15
f1 (ppm)



3f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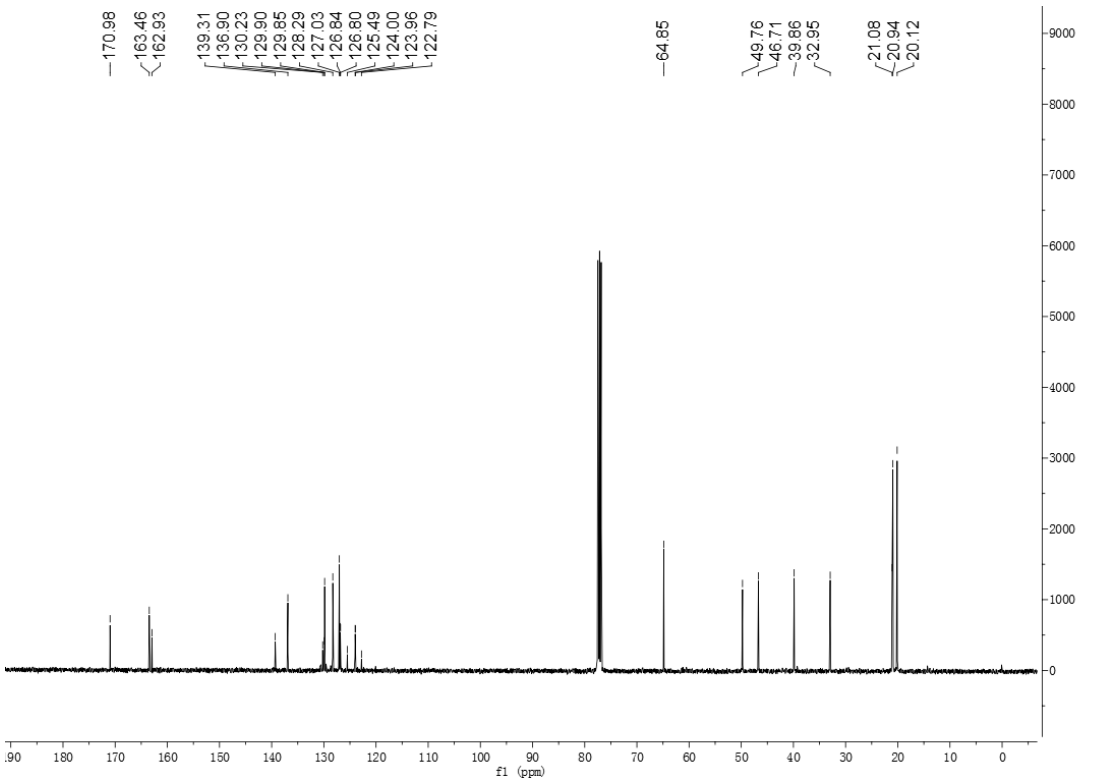
6.27
6.25
6.23
6.21
4.78
4.78
4.77
4.76
4.70
4.68
4.67
3.50
3.48
3.46
2.97
2.96
2.12
1.41
1.39
1.22
1.20
1.00
1.01
0.95
0.91
0.97
0.96
1.89
1.00
3.09
2.05
2.83
6.00
5.88
f1 (ppm)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126.80
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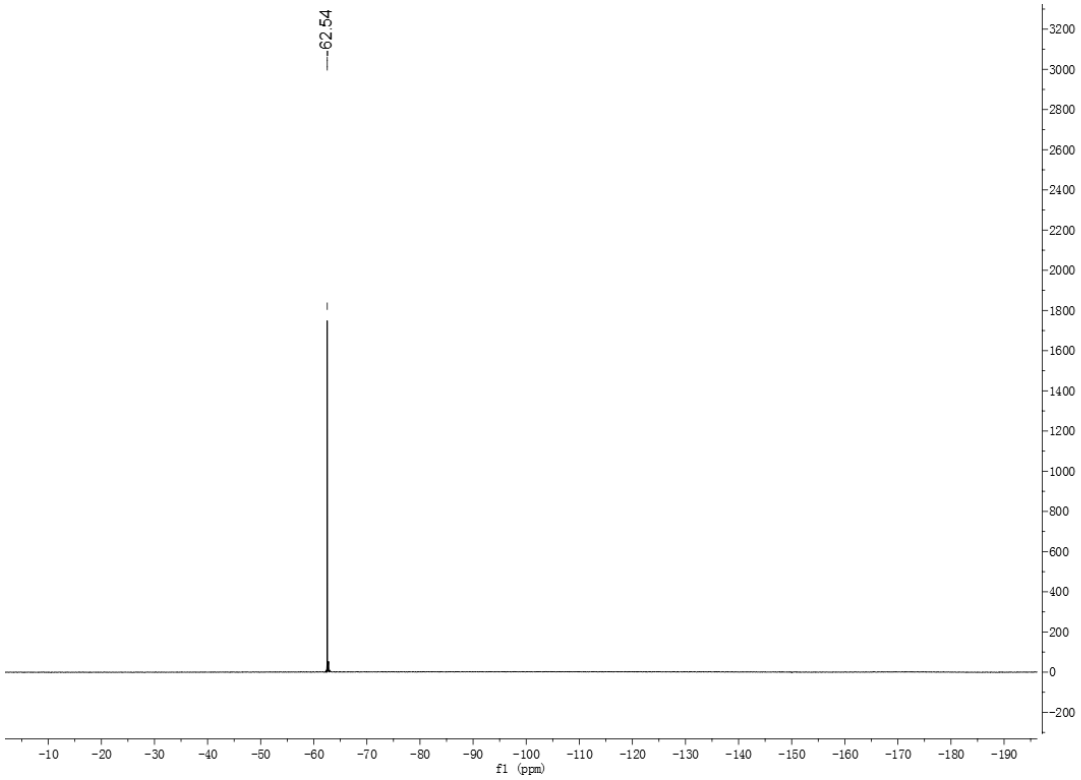
32.95
21.08
20.94
20.12
f1 (ppm)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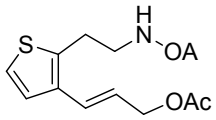
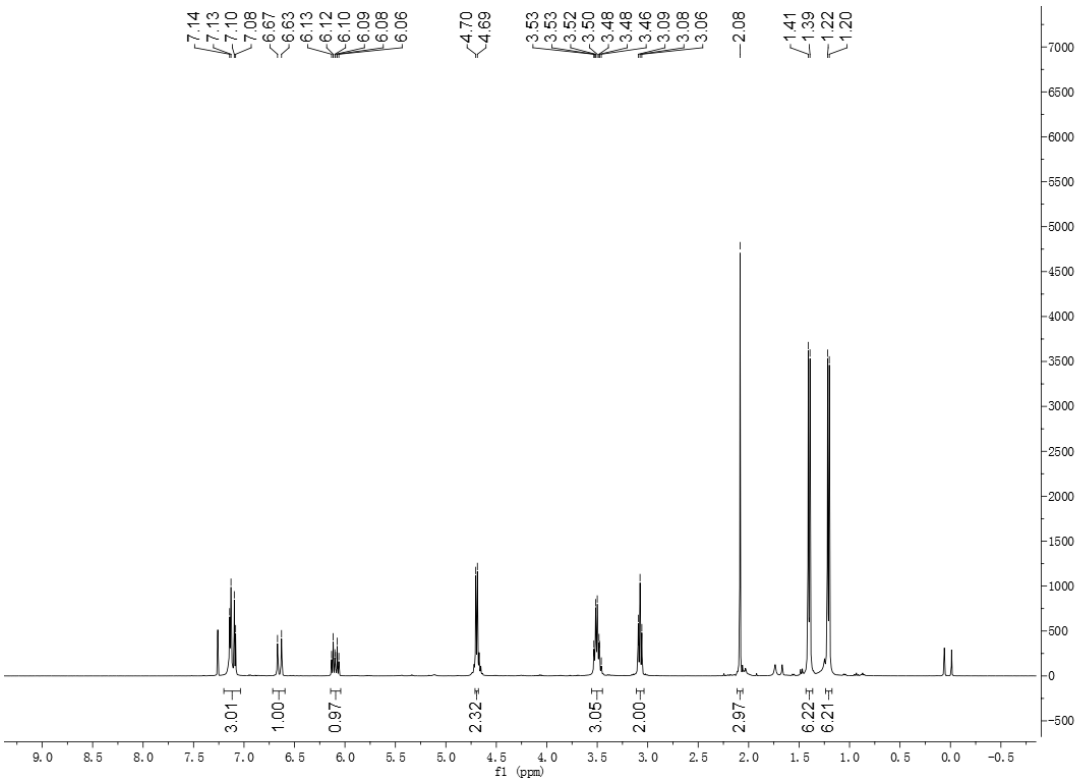

H
N
OA
S
OAc

3g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6.09
6.08
6.06
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4.69
3.53
3.53
3.52
3.50
3.48
3.48
3.46
3.09
3.08
3.06
2.08
1.41
1.39
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1.20
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1.00
0.97
2.32
3.05
2.00
2.97
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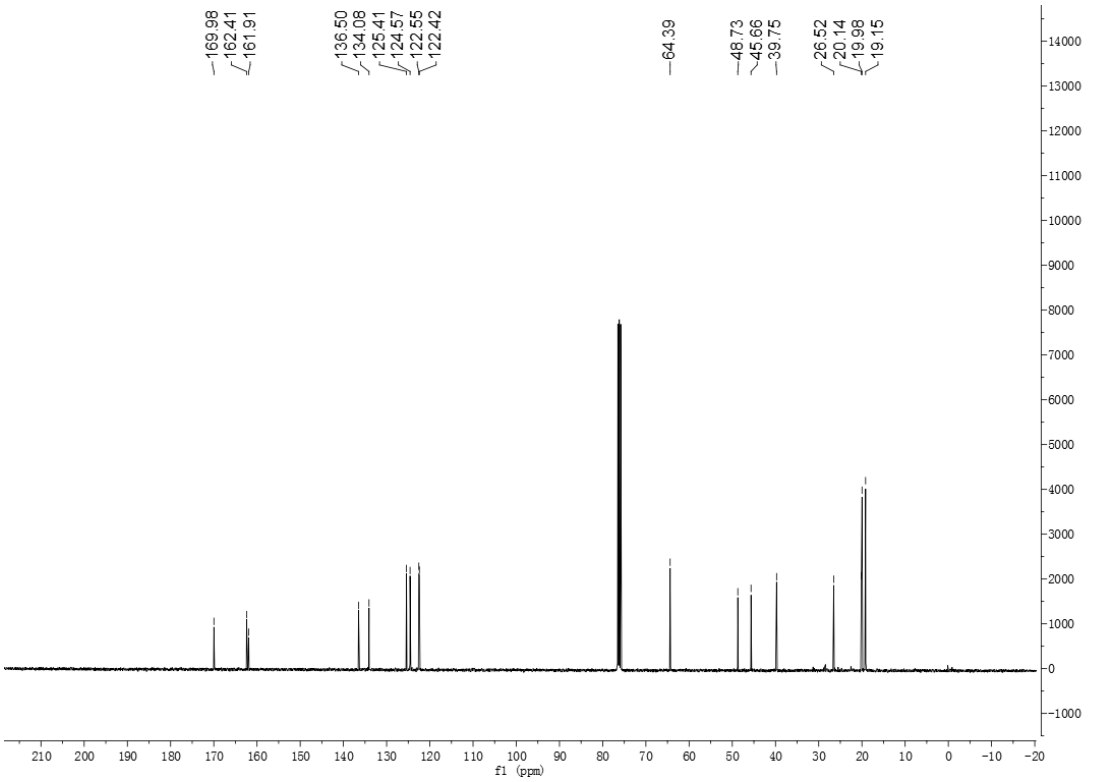
6.21
f1 (ppm)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64.39
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45.66
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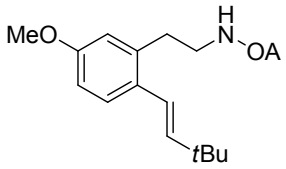
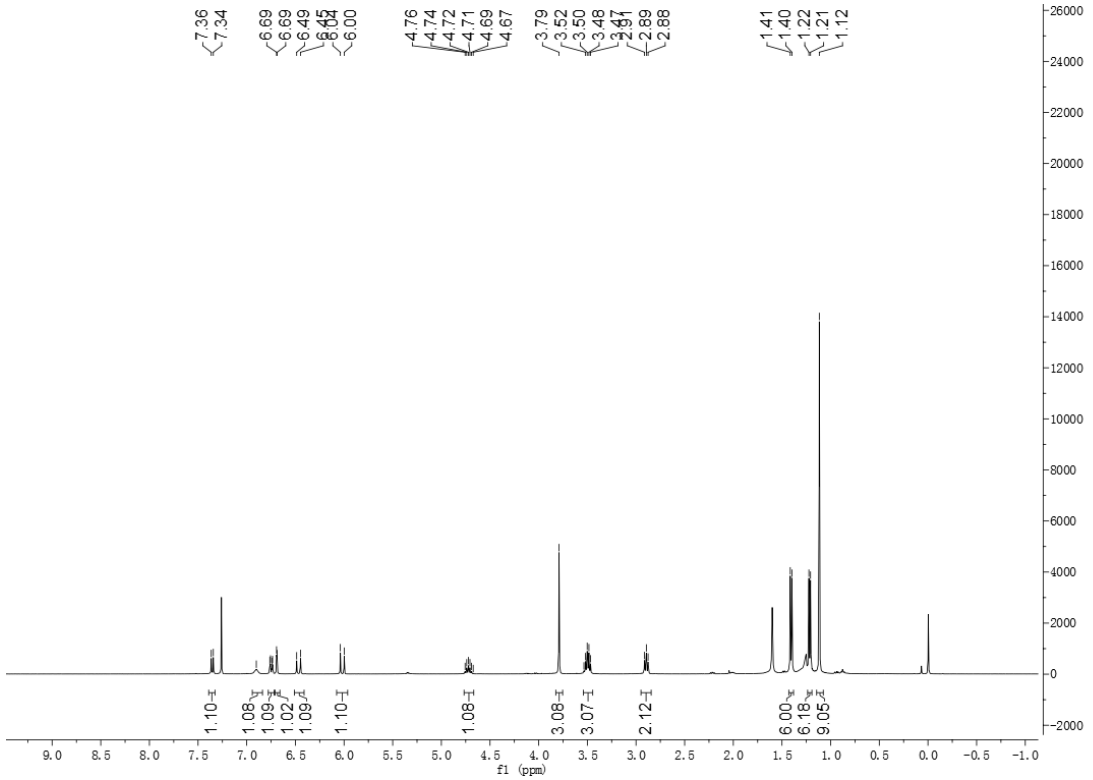
26.52
20.14
19.98
19.15
f1 (ppm)


MeO
H
N
OA
tBu

3h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4.71
4.69
4.67
3.79
3.52
3.50
3.48
2.91
2.89
2.88
1.41
1.40
1.22
1.21
1.12
1.10
1.08
1.09
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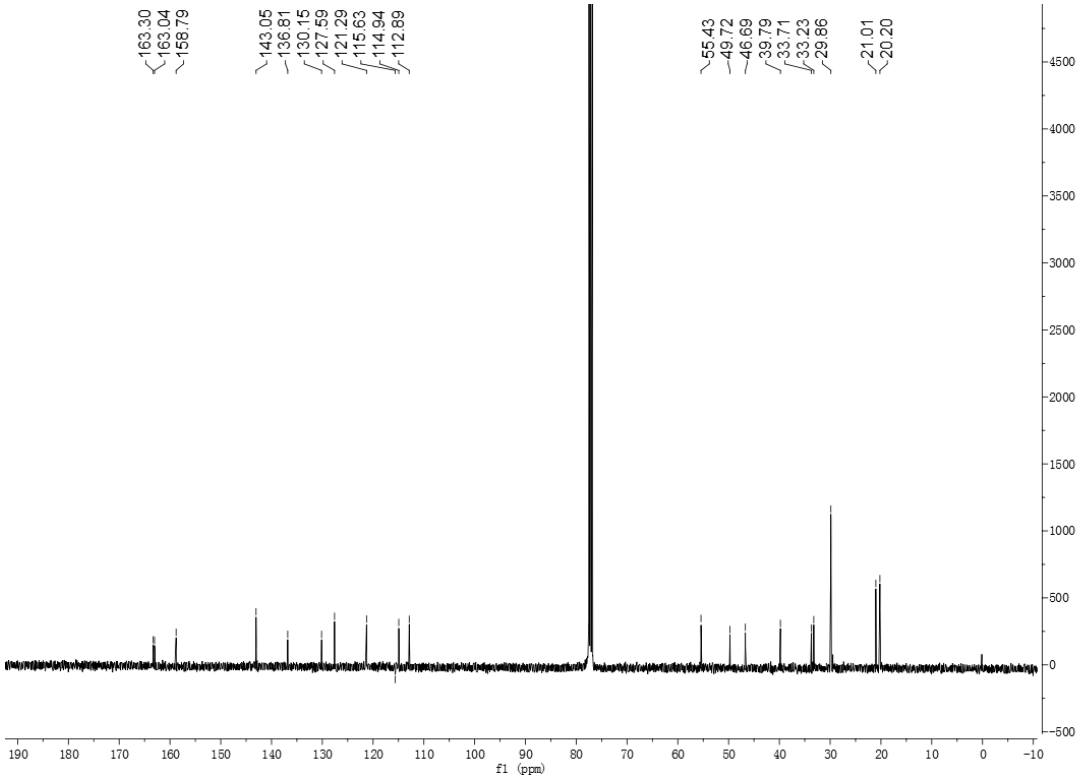
1.10
1.08
3.08
3.07
2.12
6.00
6.18
9.05
f1 (ppm)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121.29
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112.89
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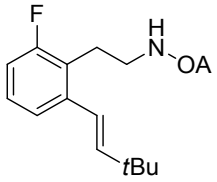
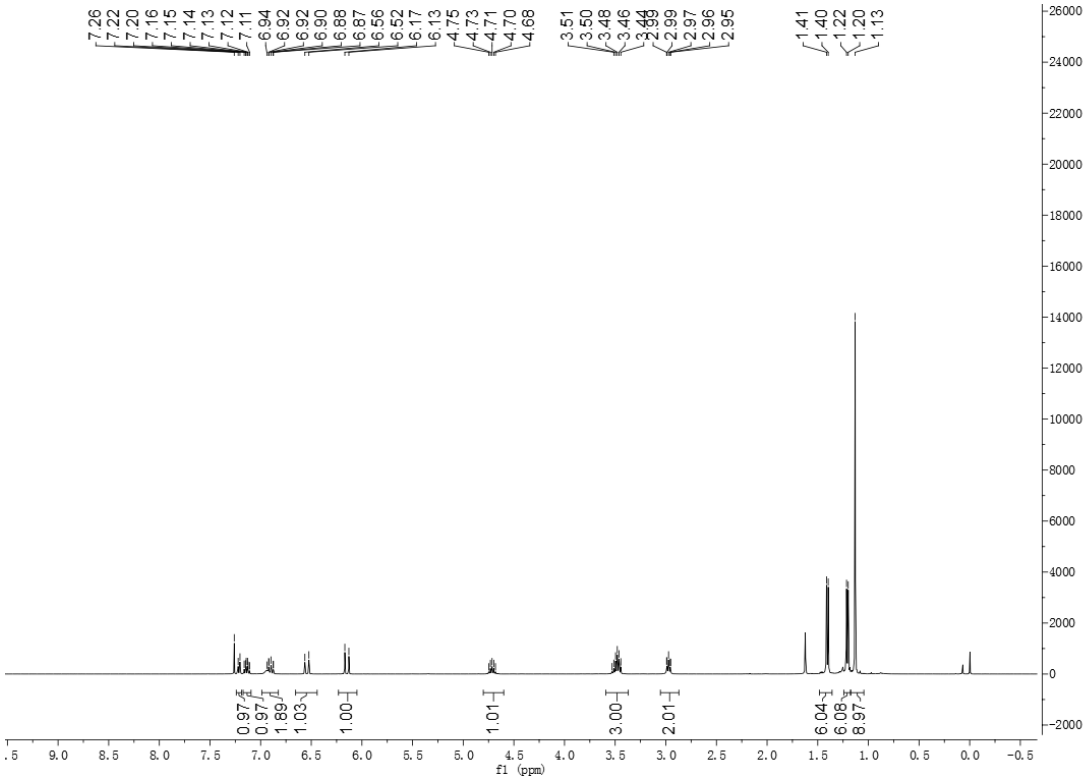
49.72
46.69
39.79
33.71
33.23
29.86
21.01
20.20
f1 (ppm)


F
H
N
OA
tBu

3i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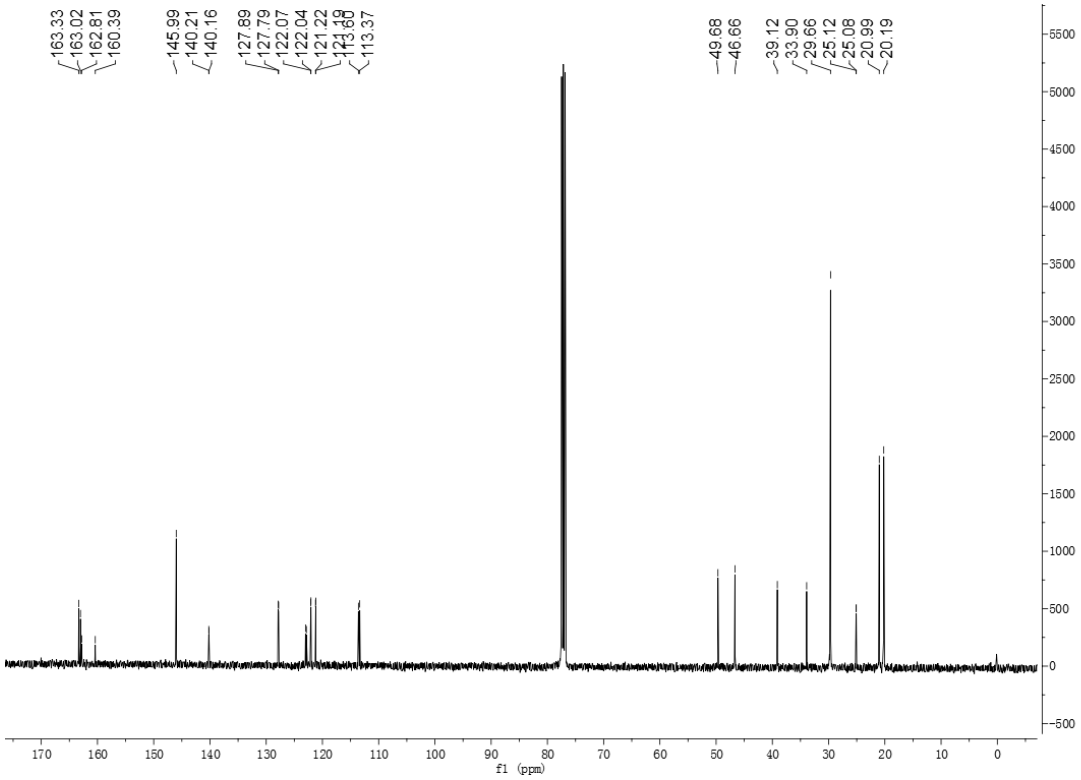
6.92
6.90
6.88
6.87
6.56
6.52
6.17
6.13
4.75
4.73
4.71
4.70
4.68
3.51
3.50
3.48
3.46
2.99
2.97
2.96
2.95
1.41
1.40
1.22
1.20
1.13
0.97
0.97
1.89
1.03
1.00
1.01
3.00
2.01
6.04
6.08
8.97
f1 (ppm)


163.33
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160.39
145.99
140.21
140.16
127.89
127.79
122.07
122.04
121.22
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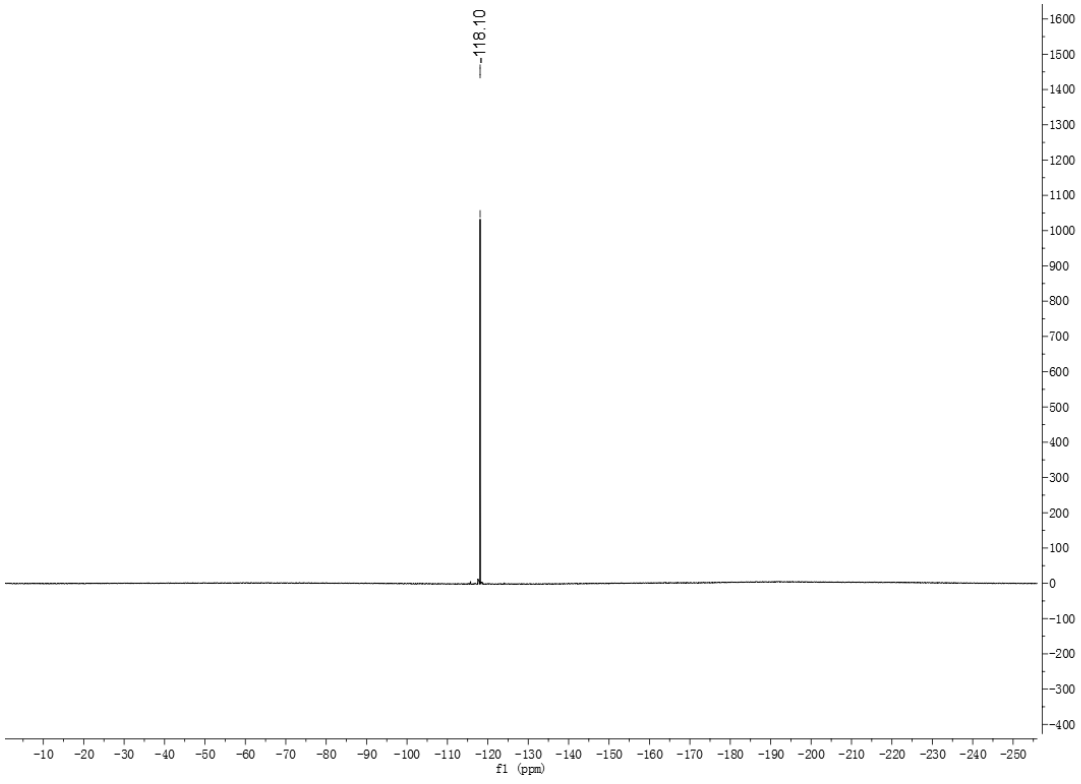
25.12
25.08
20.99
20.19
f1 (ppm)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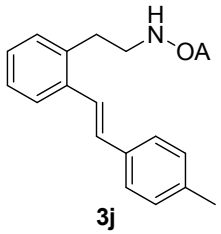
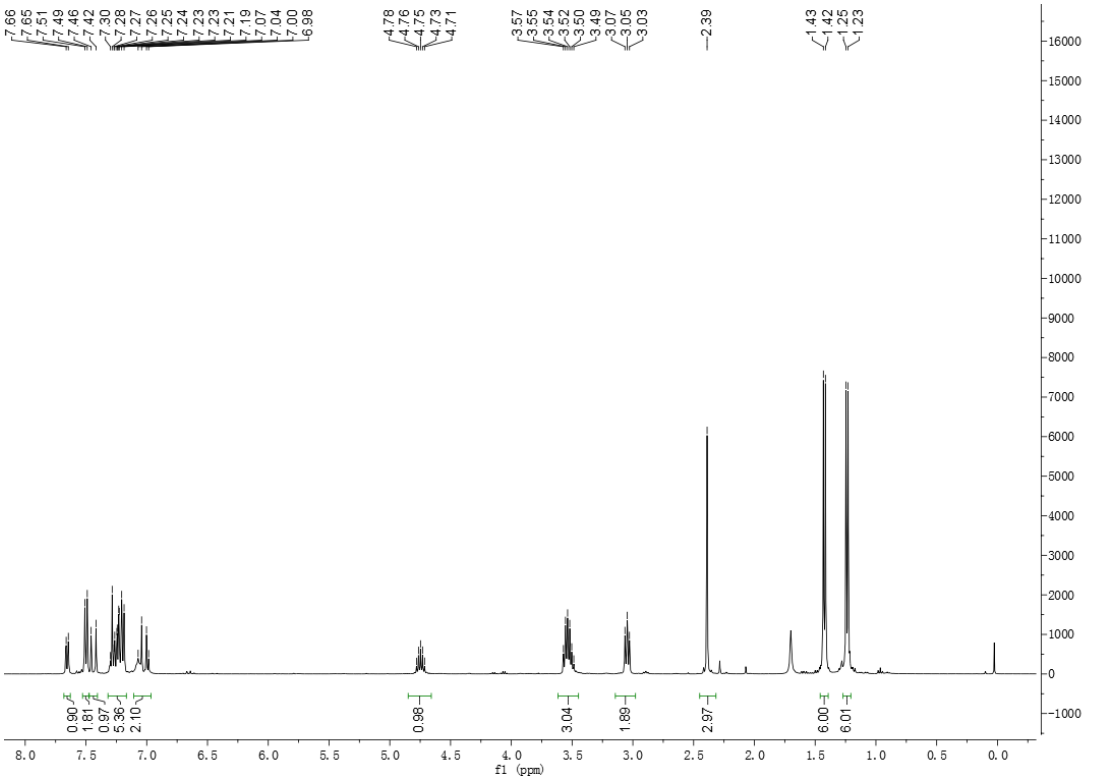

H
N
OA

3j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7.65
7.51
7.49
7.46
7.42
7.30
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7.26
7.25
7.24
7.23
7.23
7.21
7.19
7.07
7.04
7.00
6.98
4.78
4.76
4.75
4.73
4.71
3.57
3.55
3.54
3.52
3.50
3.49
3.07
3.05
3.03
2.39
1.43
1.42
1.25
1.23
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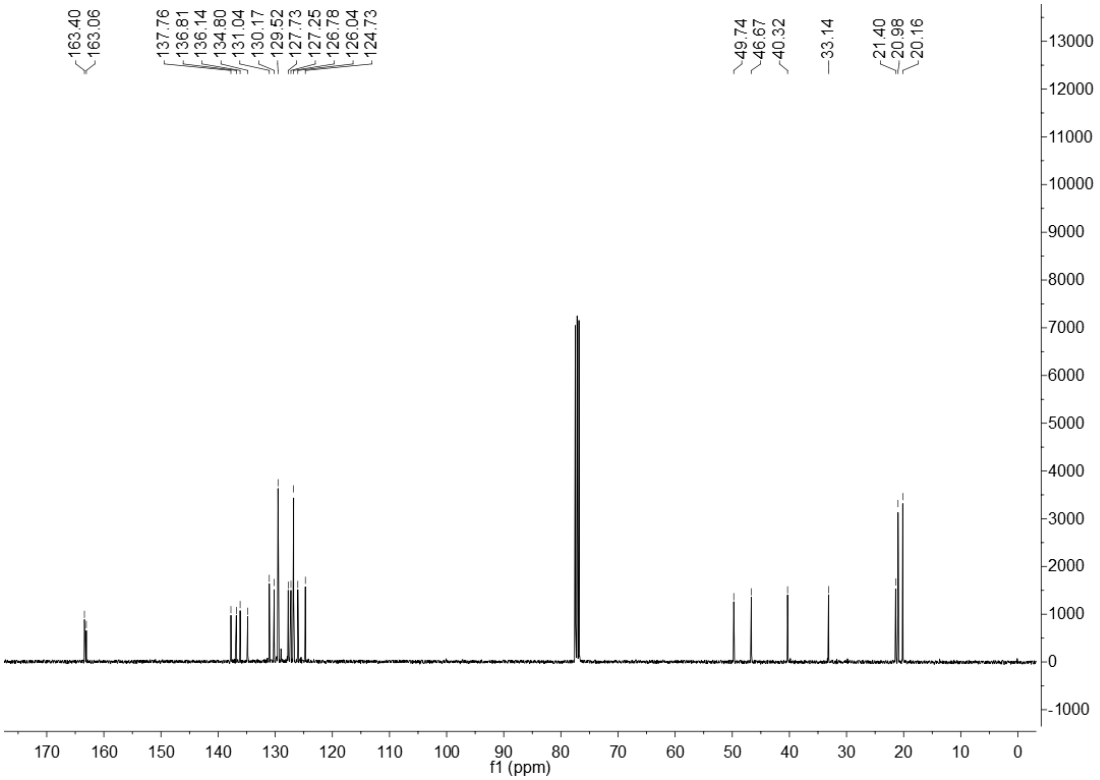
2.97
6.00
6.01
f1 (ppm)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46.67
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33.14
21.40
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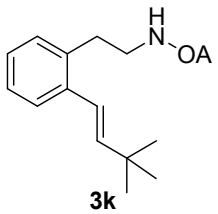
20.16
f1 (ppm)


H
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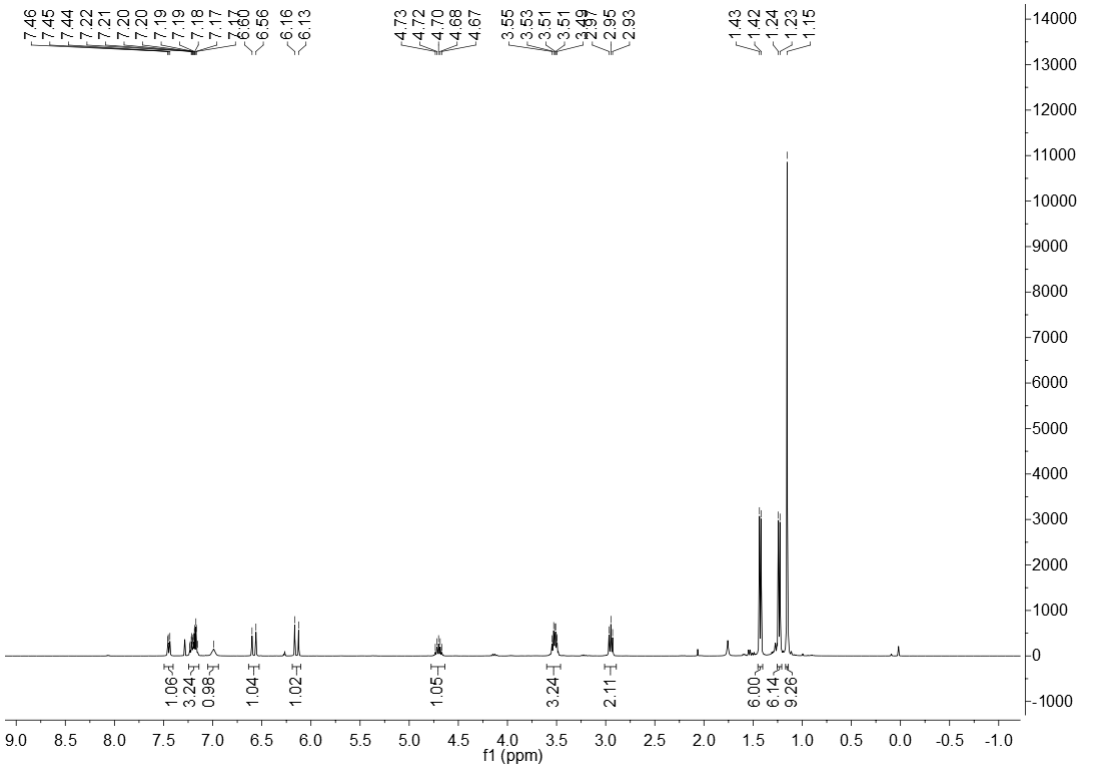
OA

3k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7.19
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6.56
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6.13
4.73
4.72
4.70
4.68
4.67
3.55
3.53
3.51
3.51
2.97
2.95
2.93
1.43
1.42
1.24
1.23
1.15
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1.05
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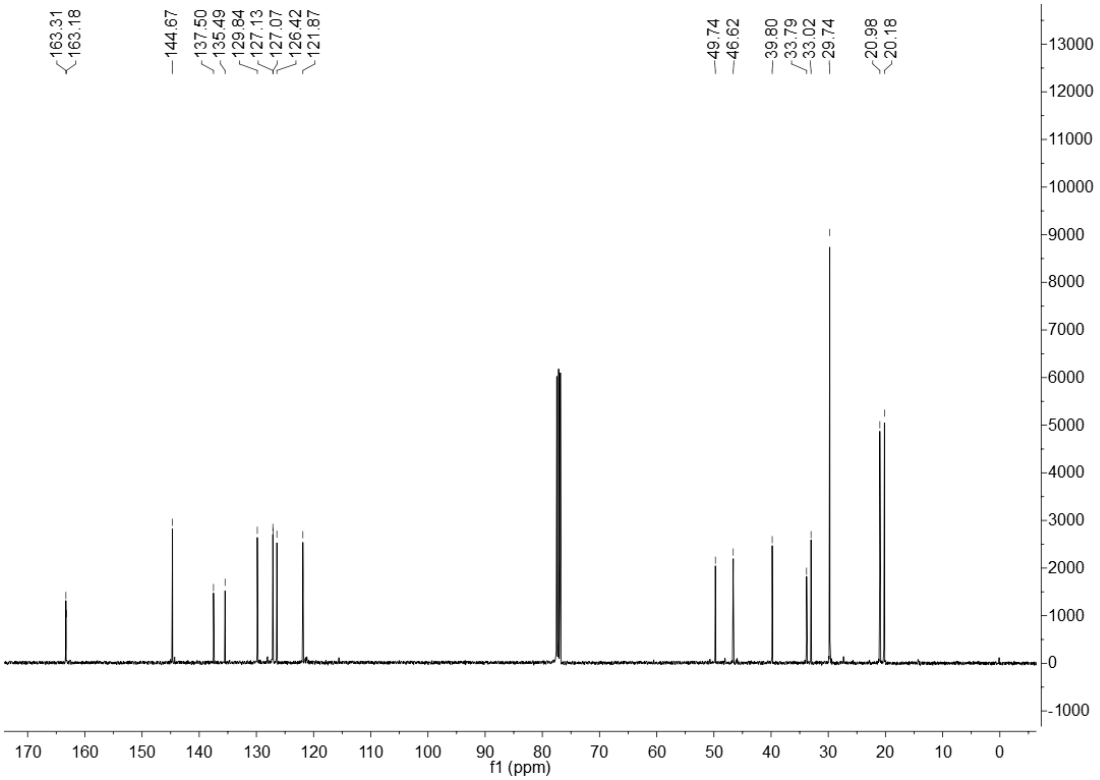
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33.02
29.74
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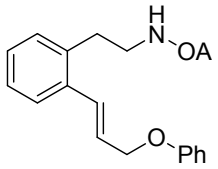
20.18
f1 (ppm)


H
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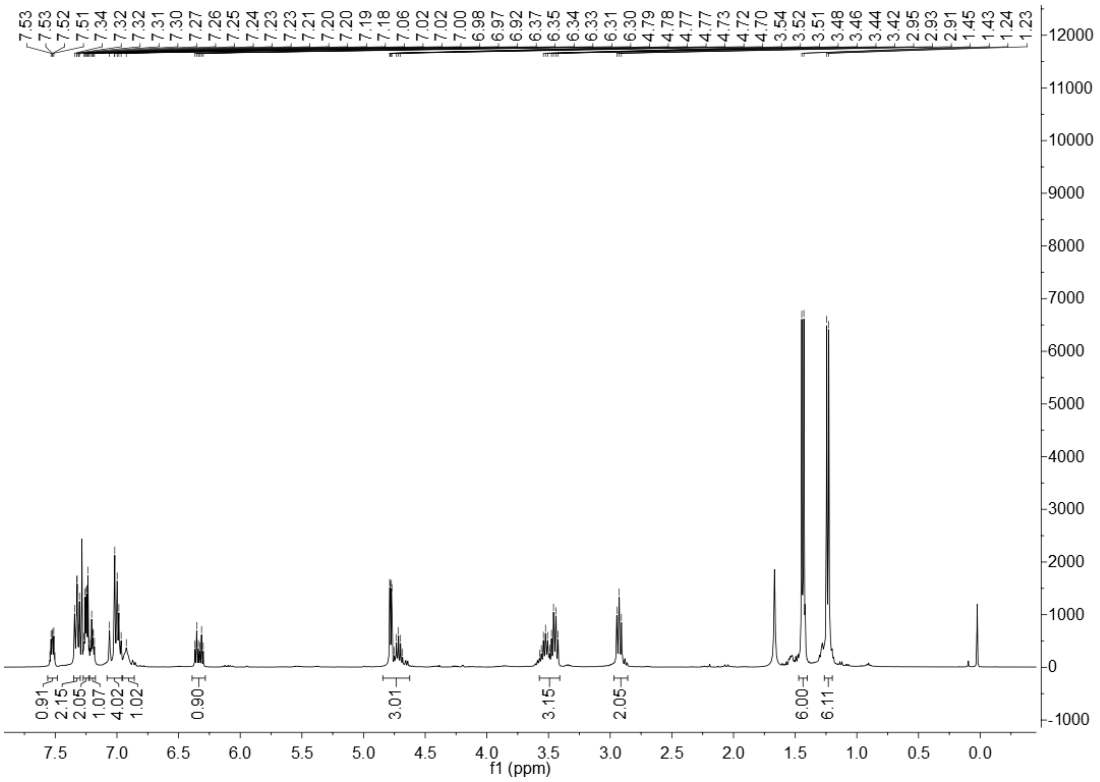
OA
O
Ph

3l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7.24
7.23
7.23
7.21
7.20
7.20
7.19
7.18
7.06
7.02
7.02
7.00
6.98
6.97
6.92
6.37
6.35
6.34
6.33
6.31
6.30
4.79
4.78
4.77
4.77
4.73
4.72
4.70
3.54
3.52
3.51
3.48
3.46
3.44
3.42
2.95
2.93
2.91
1.45
1.43
1.24
1.23
0.91
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2.05
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0.90
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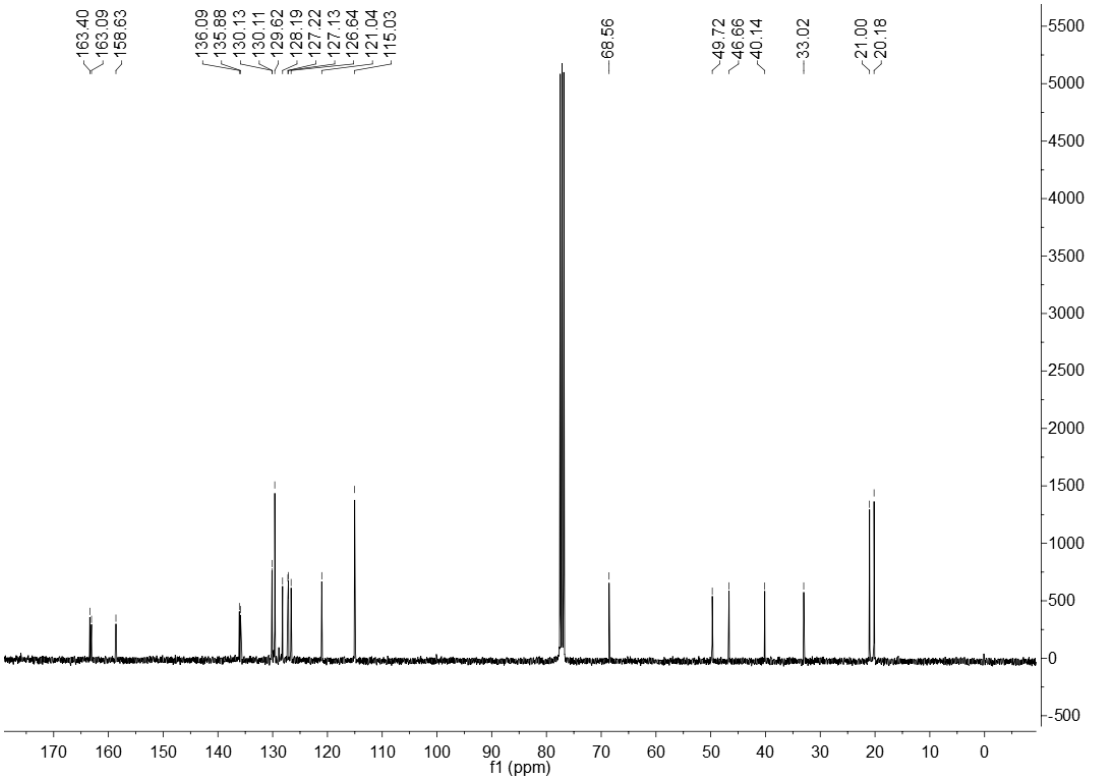
3.15
2.05
6.00
6.11
f1 (ppm)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127.22
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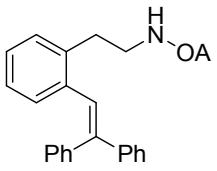
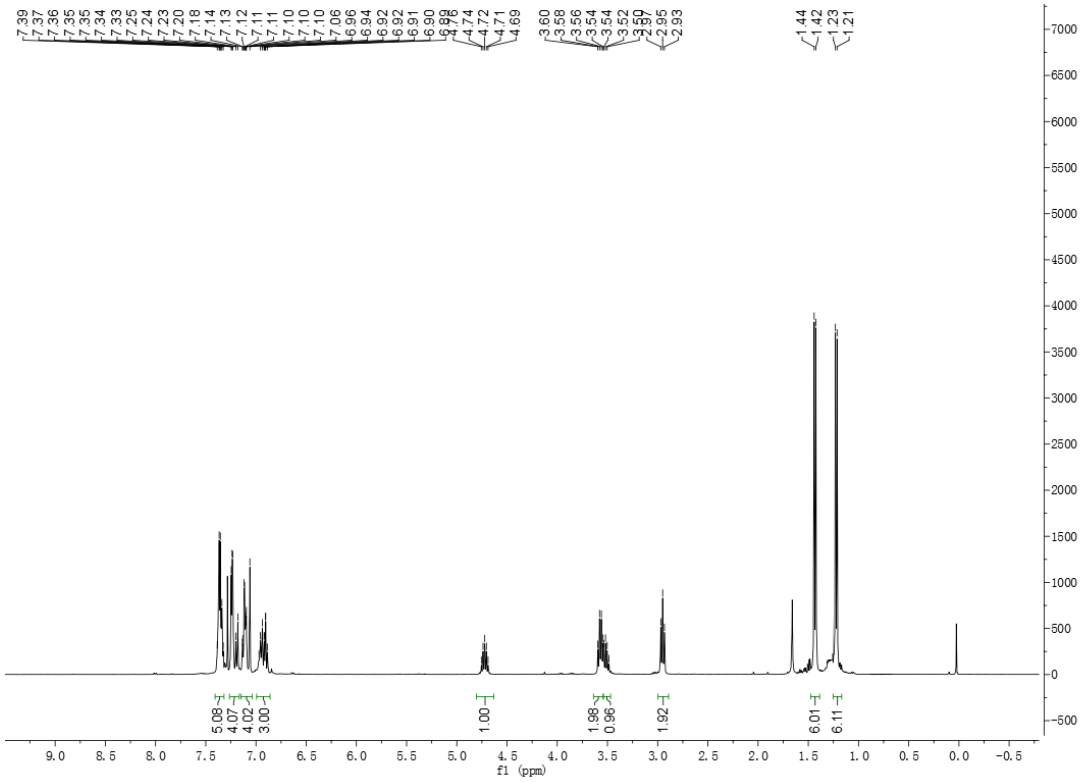
40.14
33.02
21.00
20.18
f1 (ppm)


H
N
OA
Ph
Ph

3m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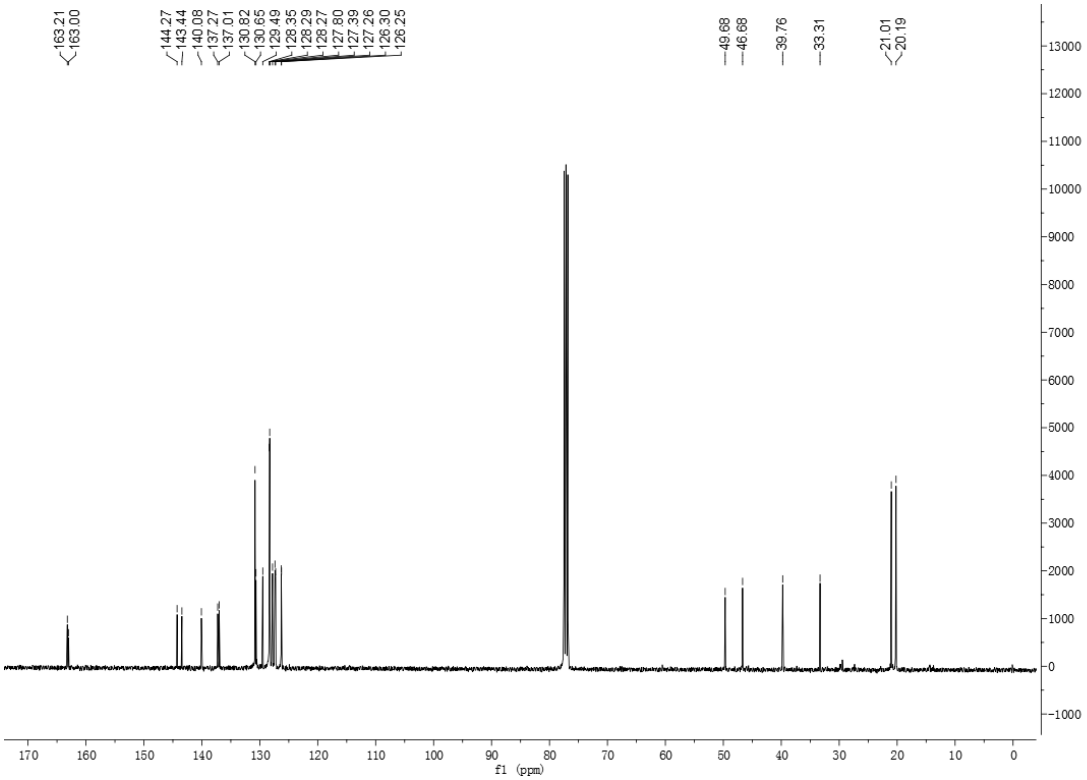
7.25
7.24
7.23
7.20
7.18
7.14
7.13
7.12
7.11
7.11
7.10
7.10
7.10
7.06
6.96
6.94
6.92
6.92
6.91
6.90
6.89
4.76
4.74
4.72
4.71
4.69
3.60
3.58
3.56
3.54
3.54
3.52
3.50
2.97
2.95
2.93
1.44
1.42
1.23
1.21
5.08
4.07
4.02
3.00
1.00
1.98
0.96
1.92
6.01
6.11
f1 (ppm)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137.27
137.01
130.82
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129.49
128.35
128.29
128.27
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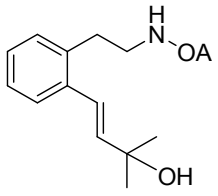
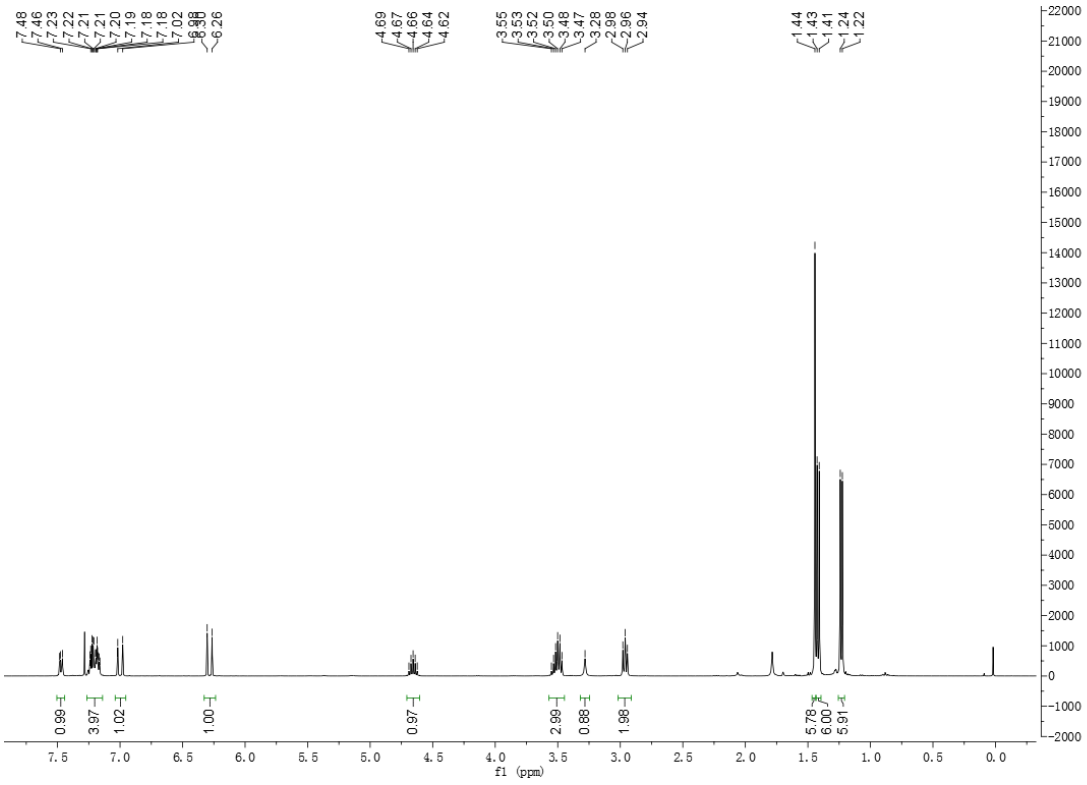
39.76
33.31
21.01
20.19
f1 (ppm)


H
N
OA
OH

3n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7.19
7.18
7.18
7.02
6.30
6.26
4.69
4.67
4.66
4.64
4.62
3.55
3.53
3.52
3.50
3.48
3.47
3.28
2.98
2.96
2.94
1.44
1.43
1.41
1.24
1.22
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3.97
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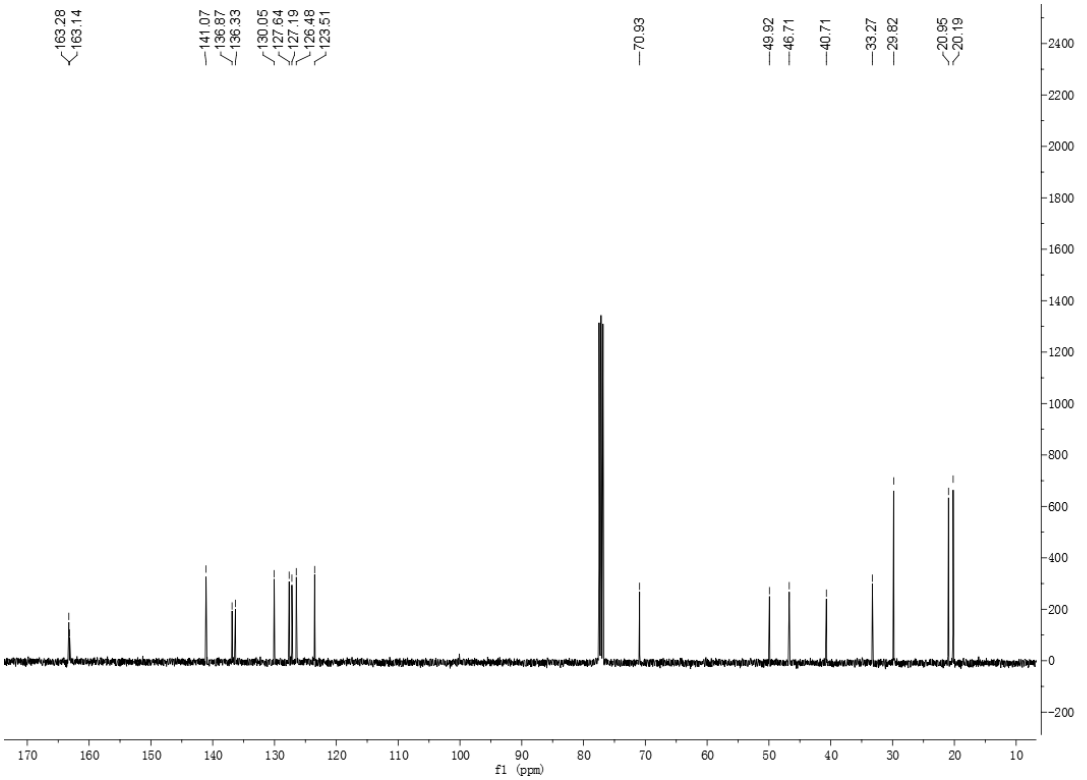
1.00
0.97
2.99
0.88
1.98
5.78
6.00
5.91
f1 (ppm)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127.19
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70.93
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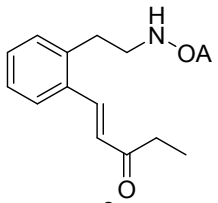
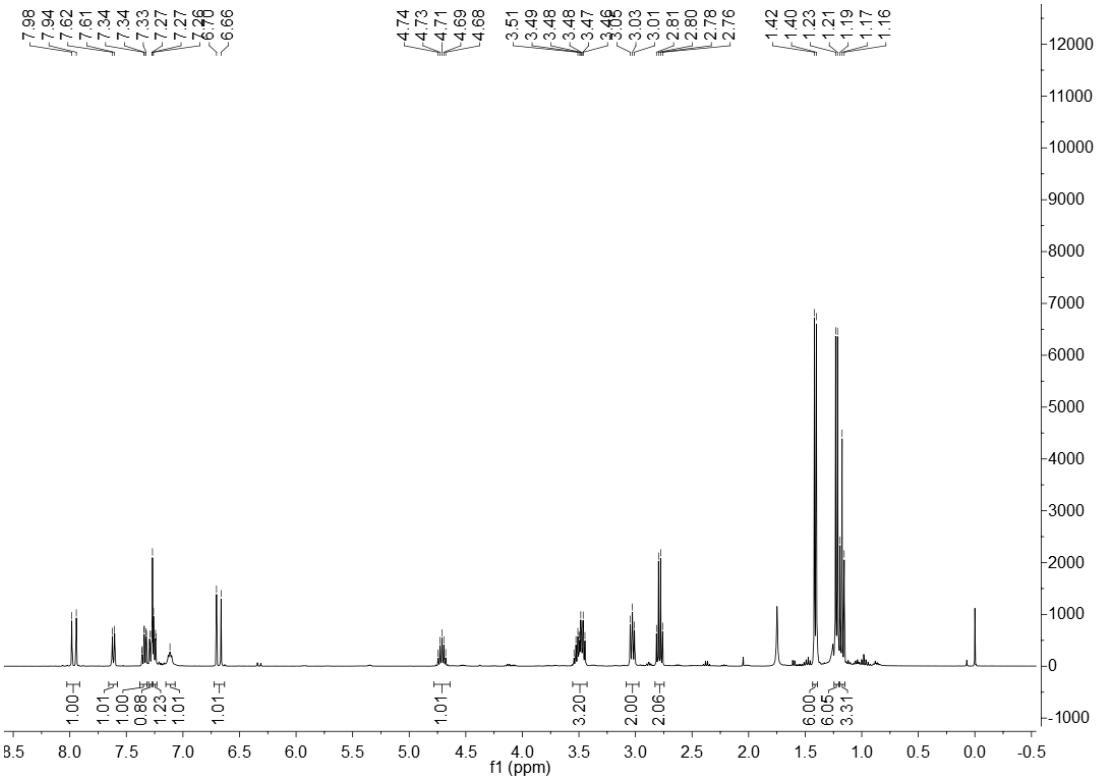
33.27
29.82
20.95
20.19
f1 (ppm)


H
N
OA
O

3o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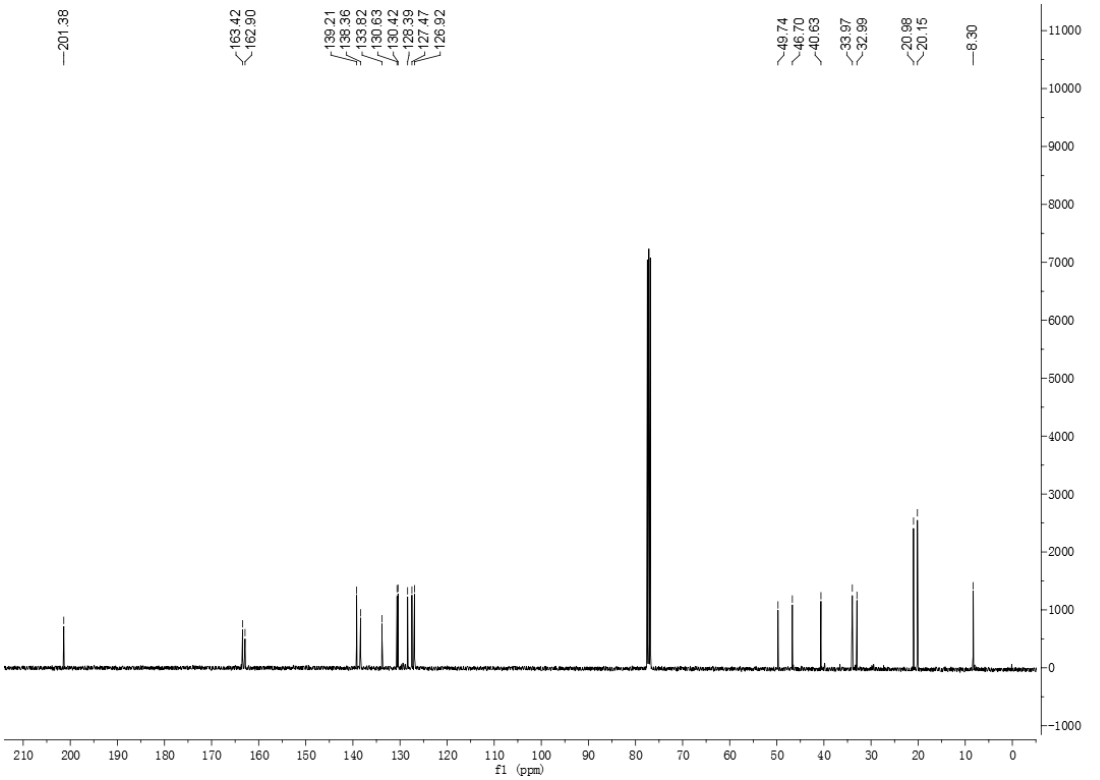
6.78
6.66
4.74
4.73
4.71
4.69
4.68
3.51
3.49
3.48
3.48
3.47
3.03
3.01
2.81
2.80
2.78
2.76
1.42
1.40
1.23
1.21
1.19
1.17
1.16
1.00
1.01
1.00
0.88
1.23
1.01
1.01
1.01
3.20
2.00
2.06
6.00
6.05
3.31
f1 (ppm)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130.42
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127.47
126.92
49.74
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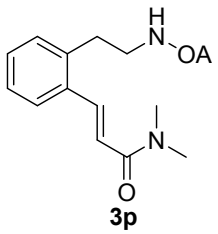
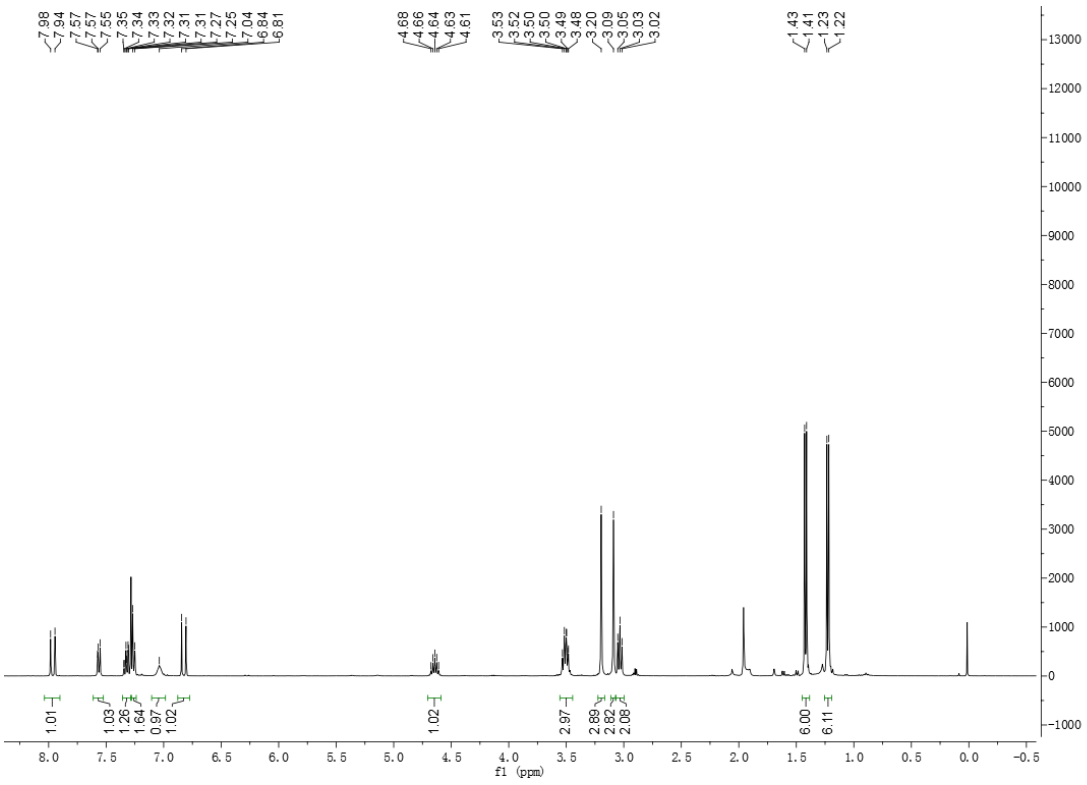
32.99
20.98
20.15
8.30
f1 (ppm)


H
N
OA
N
O

3p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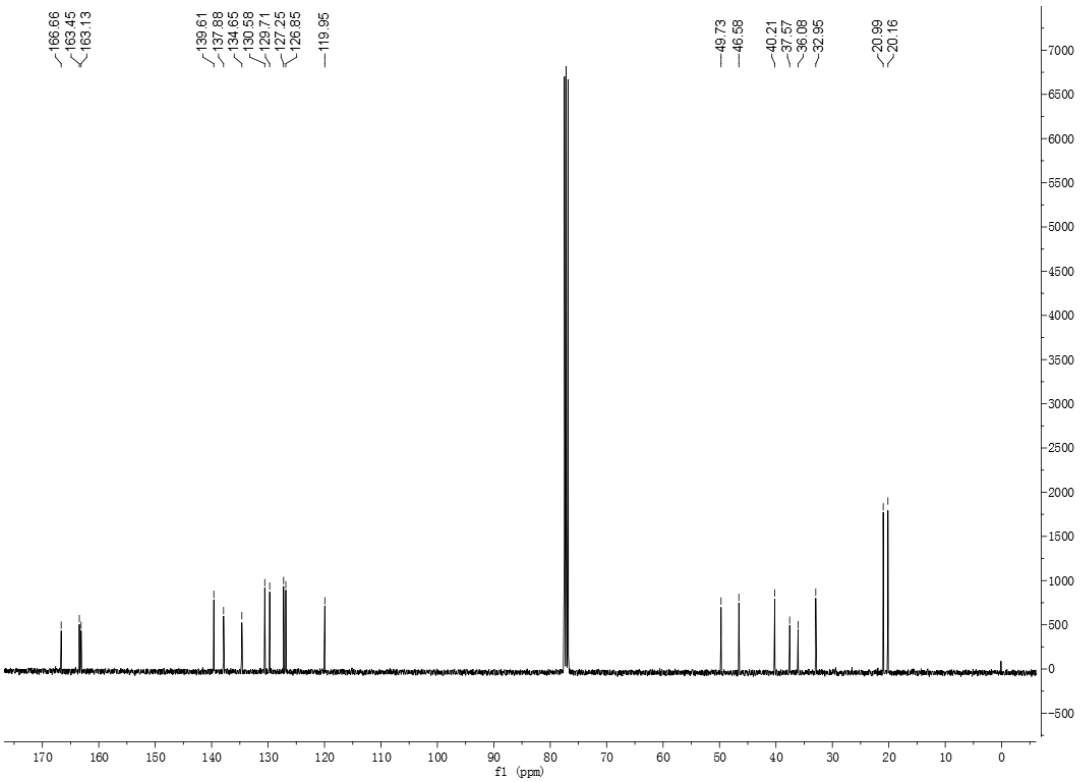
7.35
7.34
7.33
7.32
7.31
7.31
7.27
7.25
7.04
6.84
6.81
4.68
4.66
4.64
4.63
4.61
3.53
3.52
3.50
3.50
3.49
3.48
3.20
3.09
3.05
3.03
3.02
1.43
1.41
1.23
1.22
1.01
1.03
1.26
1.64
0.97
1.02
1.02
2.97
2.89
2.82
2.08
6.00
6.11
f1 (ppm)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134.65
130.58
129.71
127.25
126.65
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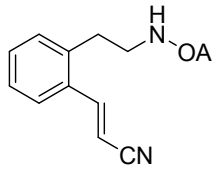
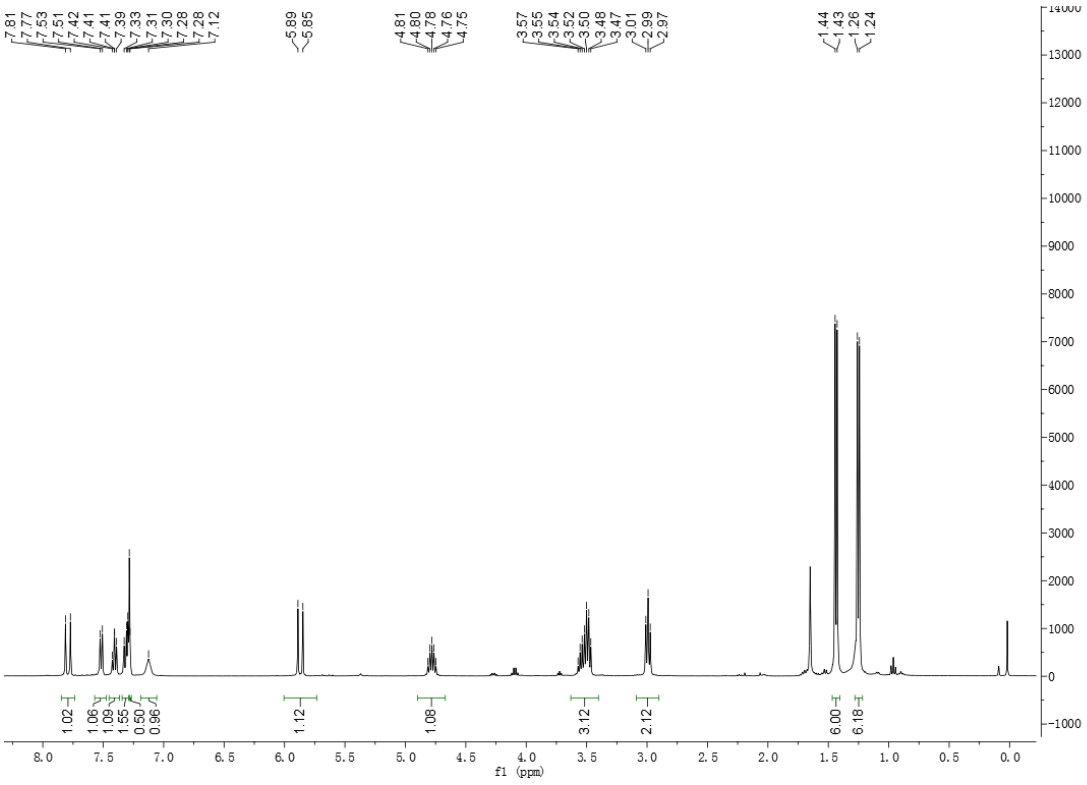
49.73
46.58
40.21
37.57
36.08
32.95
20.99
20.16
f1 (ppm)


H
N
OA
CN

3q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7.41
7.41
7.39
7.33
7.31
7.30
7.28
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7.12
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4.80
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3.57
3.55
3.54
3.52
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3.48
3.47
3.01
2.99
2.97
1.44
1.43
1.26
1.24
1.02
1.06
1.09
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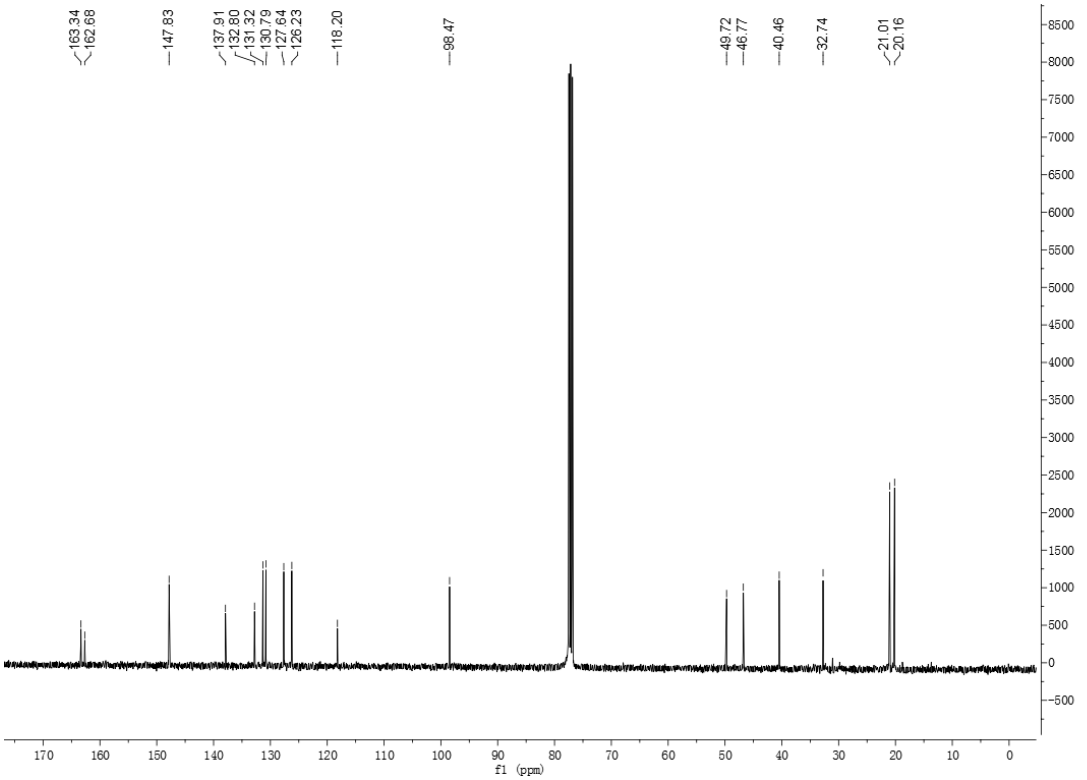
0.50
0.96
1.12
1.08
3.12
2.12
6.00
6.18
f1 (ppm)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127.64
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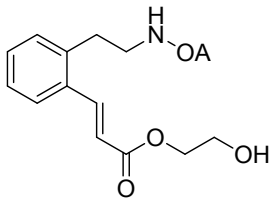
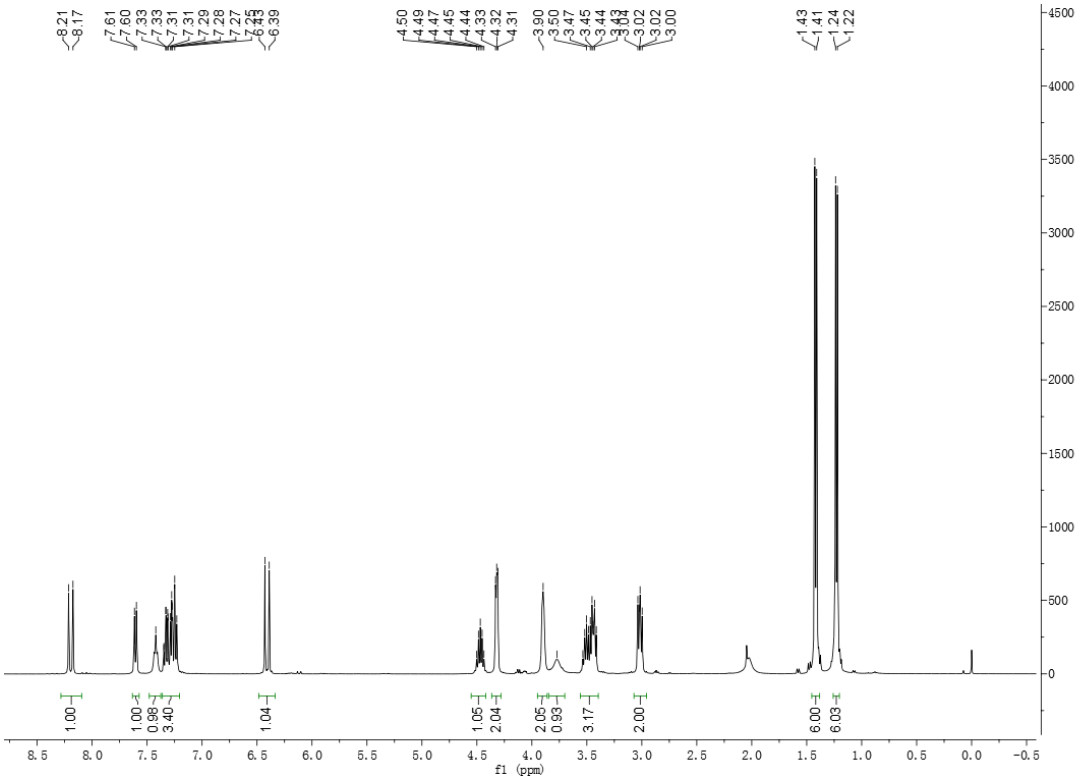
118.20
98.47
49.72
46.77
40.46
32.74
21.01
20.16
f1 (ppm)


H
N
OA
O
OH
O

3r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7.29
7.28
7.27
7.25
6.43
6.39
4.50
4.49
4.47
4.45
4.44
4.33
4.32
4.31
3.90
3.50
3.47
3.45
3.44
3.43
3.04
3.02
3.02
3.00
1.43
1.41
1.24
1.22
1.00
1.00
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3.40
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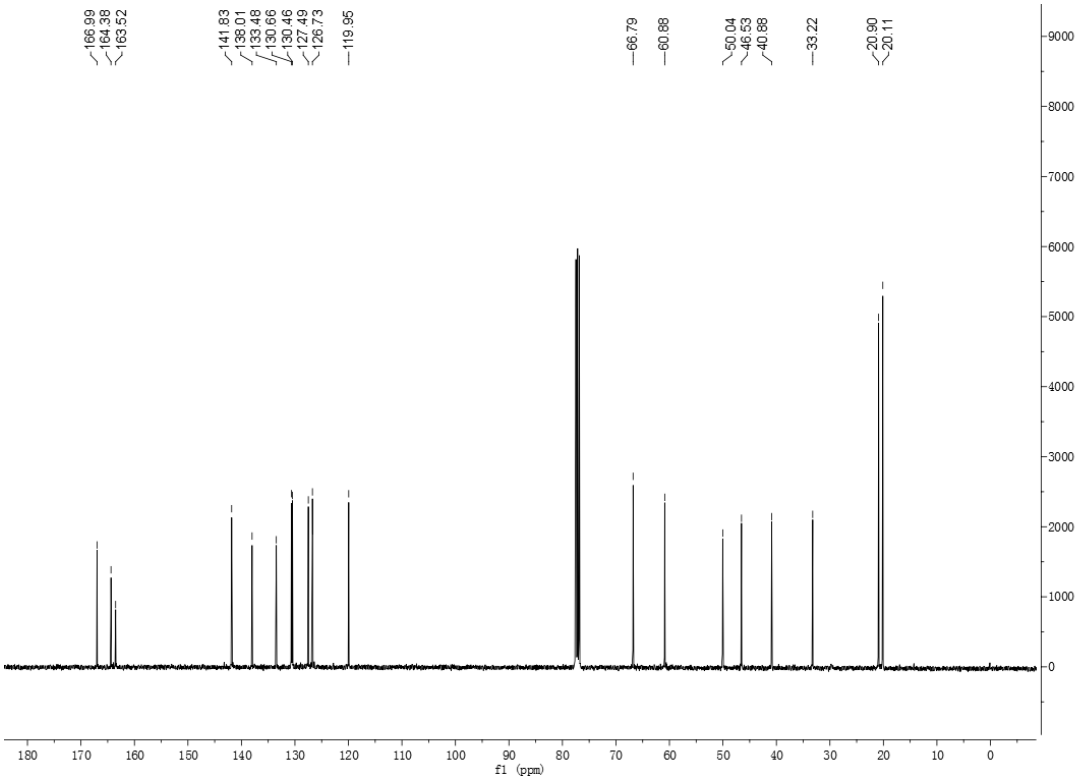
1.05
2.04
2.05
0.93
3.17
2.00
6.00
6.03
f1 (ppm)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126.73
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66.79
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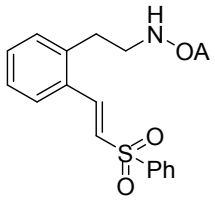
40.88
33.22
20.90
20.11
f1 (ppm)


H
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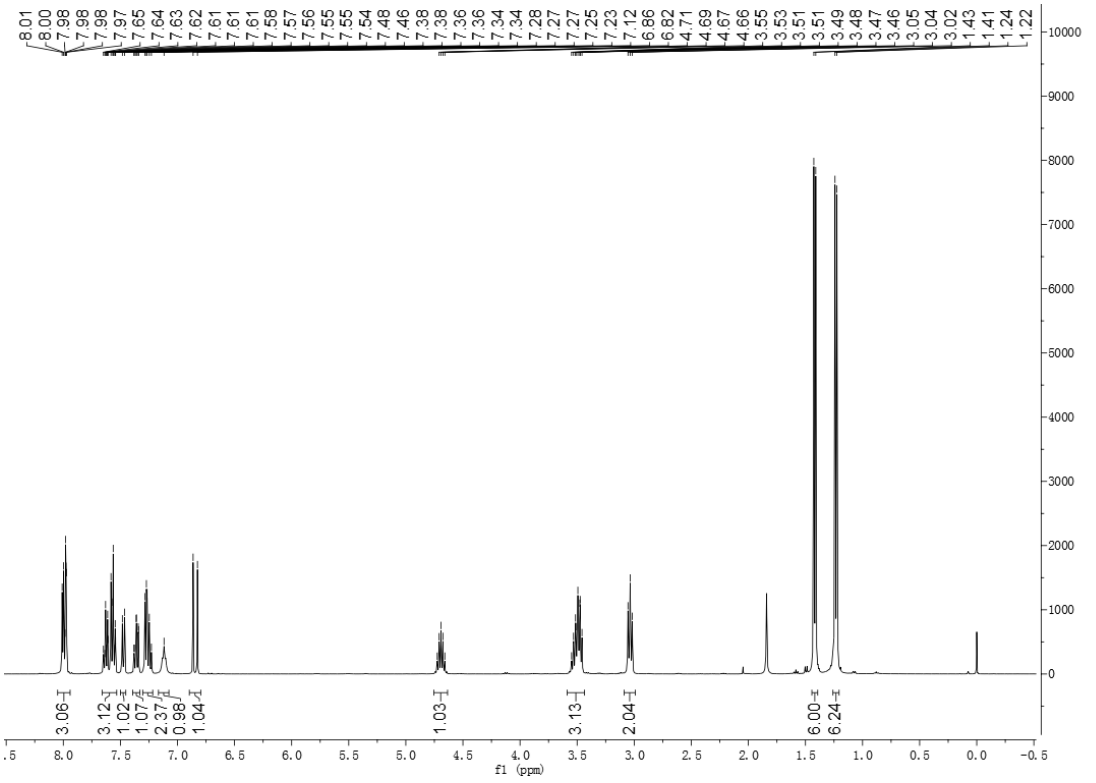
OA
O
S
Ph
O

3s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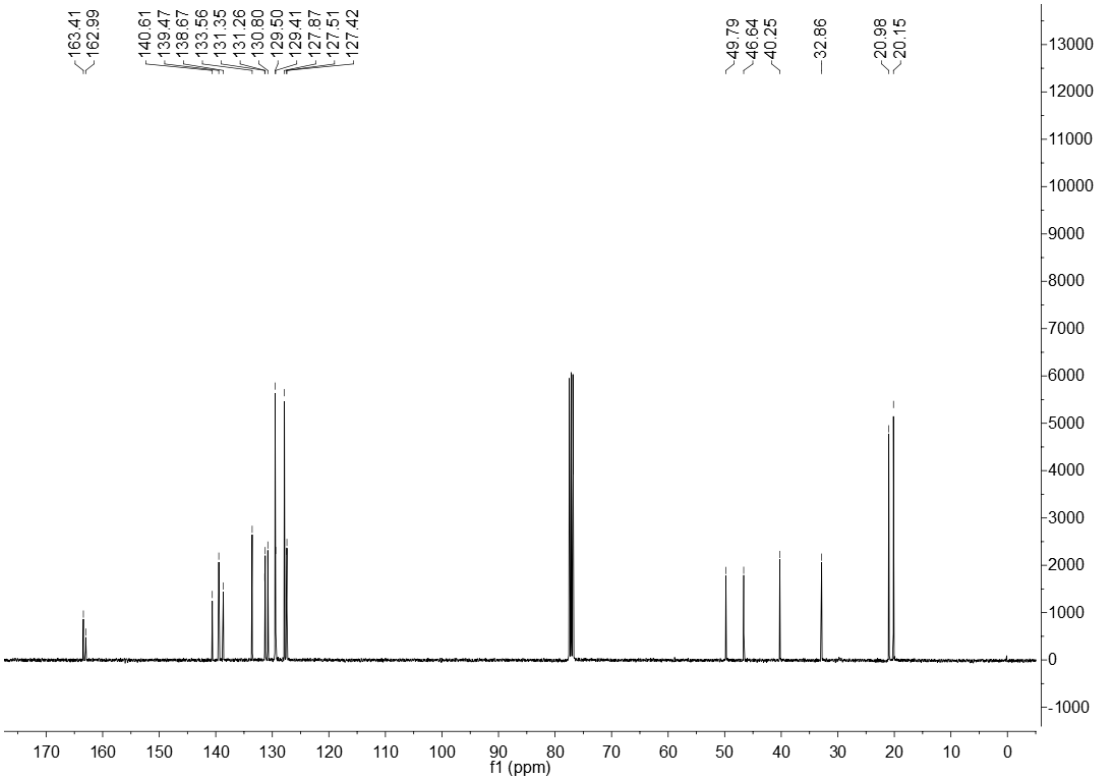
3.06 3.12 1.02 1.07 2.37 0.98 1.04 1.03 3.13 2.04 6.00 6.24
f1 (ppm)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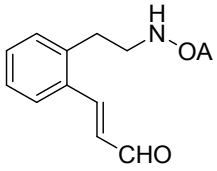
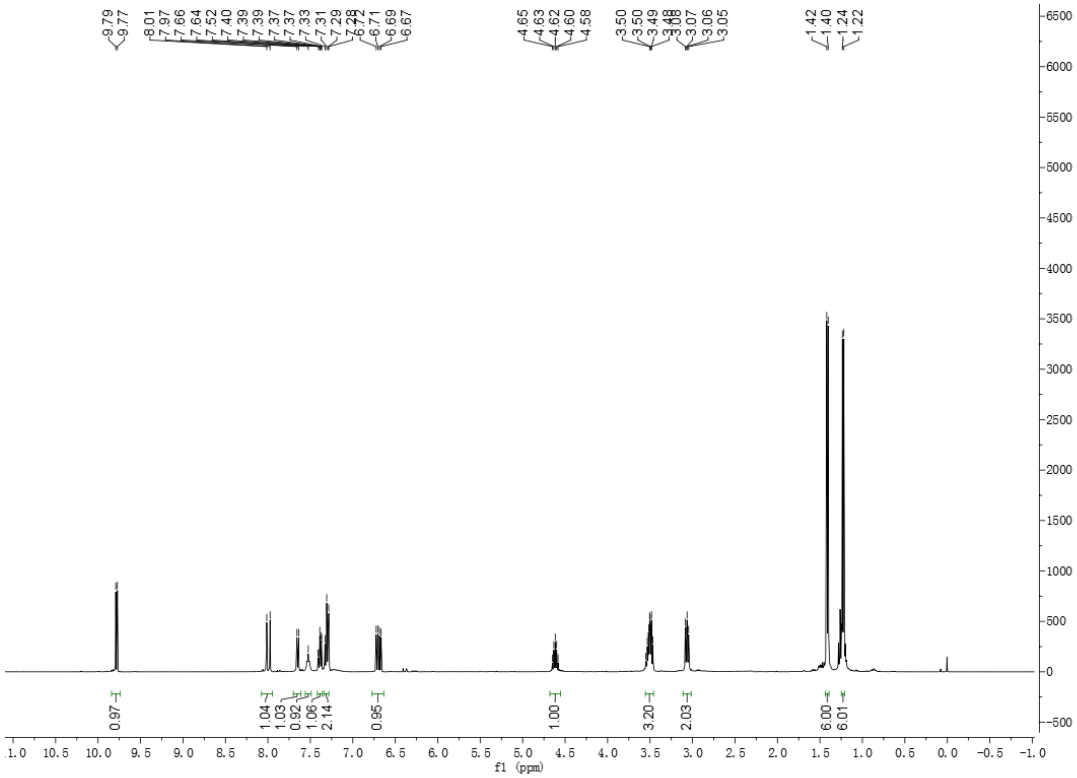
f1 (ppm)


H
N
OA
CHO

3t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7.29
6.71
6.69
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4.63
4.62
4.60
4.58
3.50
3.50
3.49
3.48
3.08
3.07
3.06
3.05
1.42
1.40
1.24
1.22
0.97
1.04
1.03
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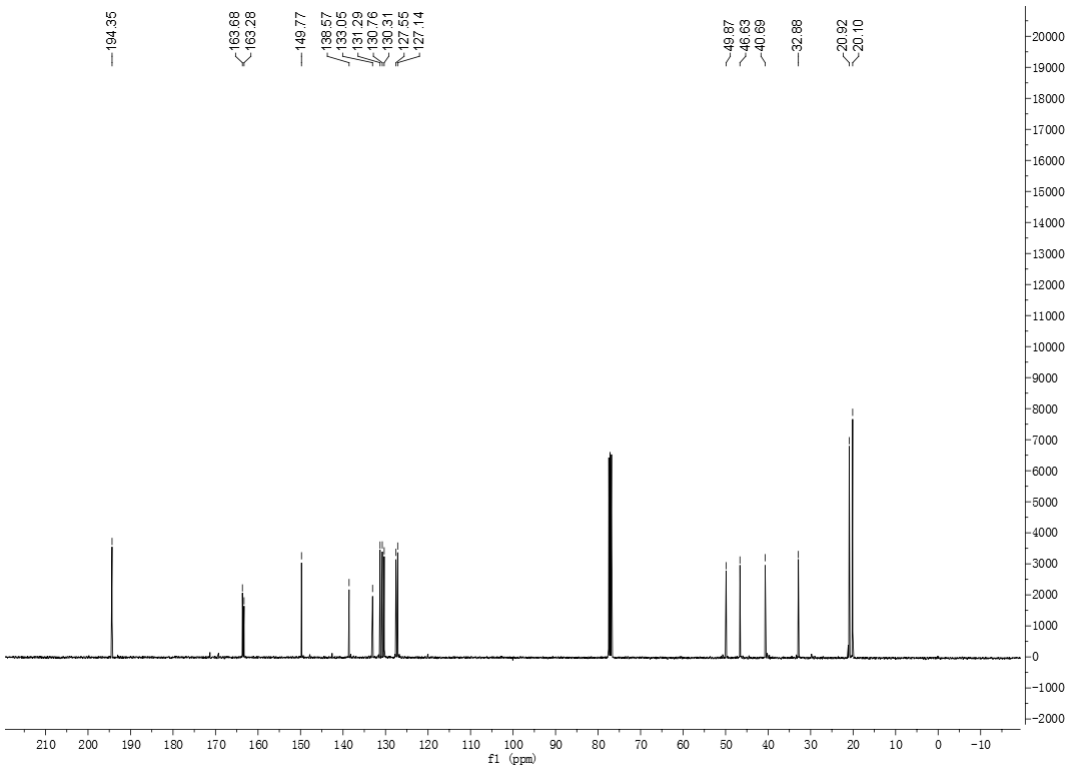
1.06
2.14
0.95
1.00
3.20
2.03
6.00
6.01
f1 (ppm)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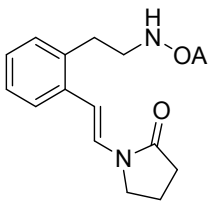
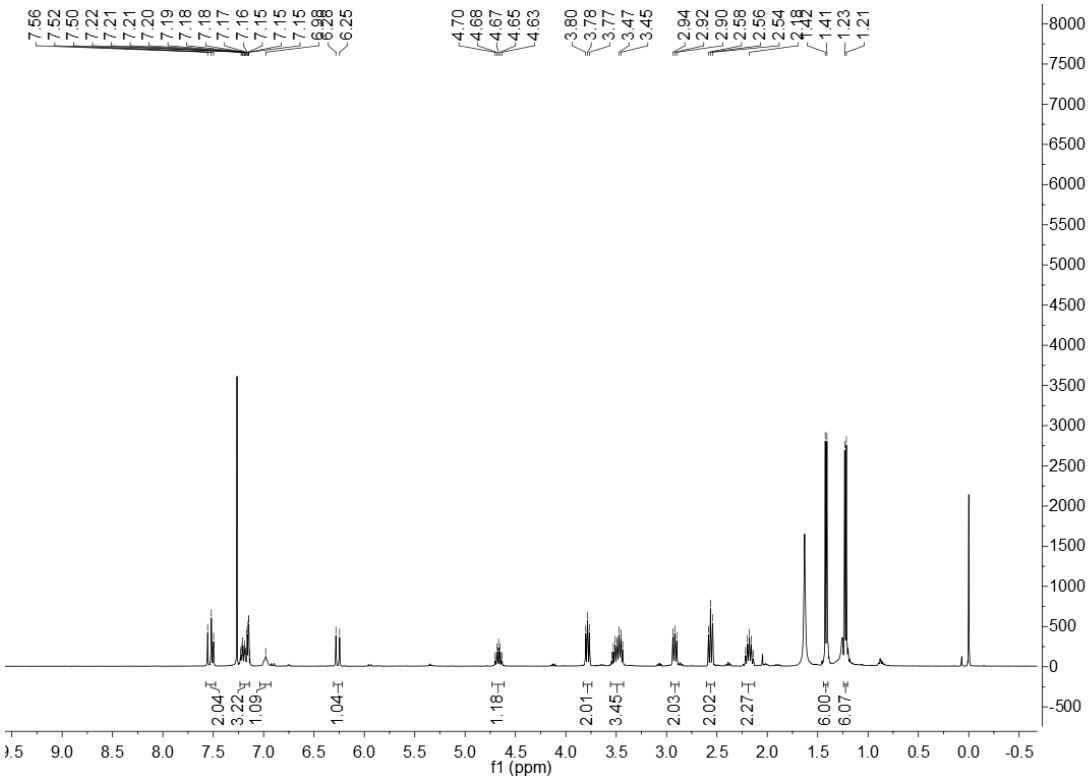
127.55
127.14
49.87
46.63
40.69
32.88
20.92
20.10
f1 (ppm)


H
N
OA
O
N

3u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7.50
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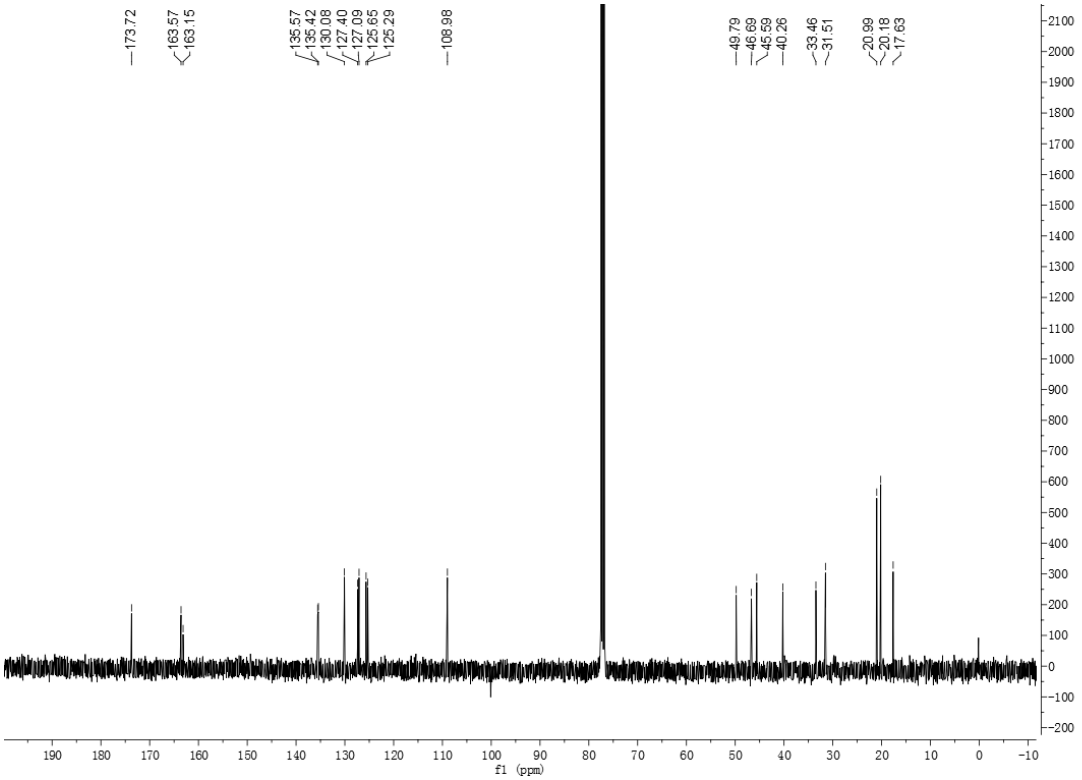
7.20
7.19
7.18
7.18
7.17
7.16
7.15
7.15
7.15
6.98
6.28
6.25
4.70
4.68
4.67
4.65
4.63
3.80
3.78
3.77
3.47
3.45
2.94
2.92
2.90
2.58
2.56
2.54
2.18
1.41
1.23
1.21
2.04
3.22
1.09
1.04
1.18
2.01
3.45
2.03
2.02
2.27
6.00
6.07
f1 (ppm)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163.15
135.57
135.42
130.08
127.40
127.09
125.65
125.29
108.98
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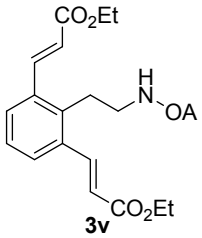
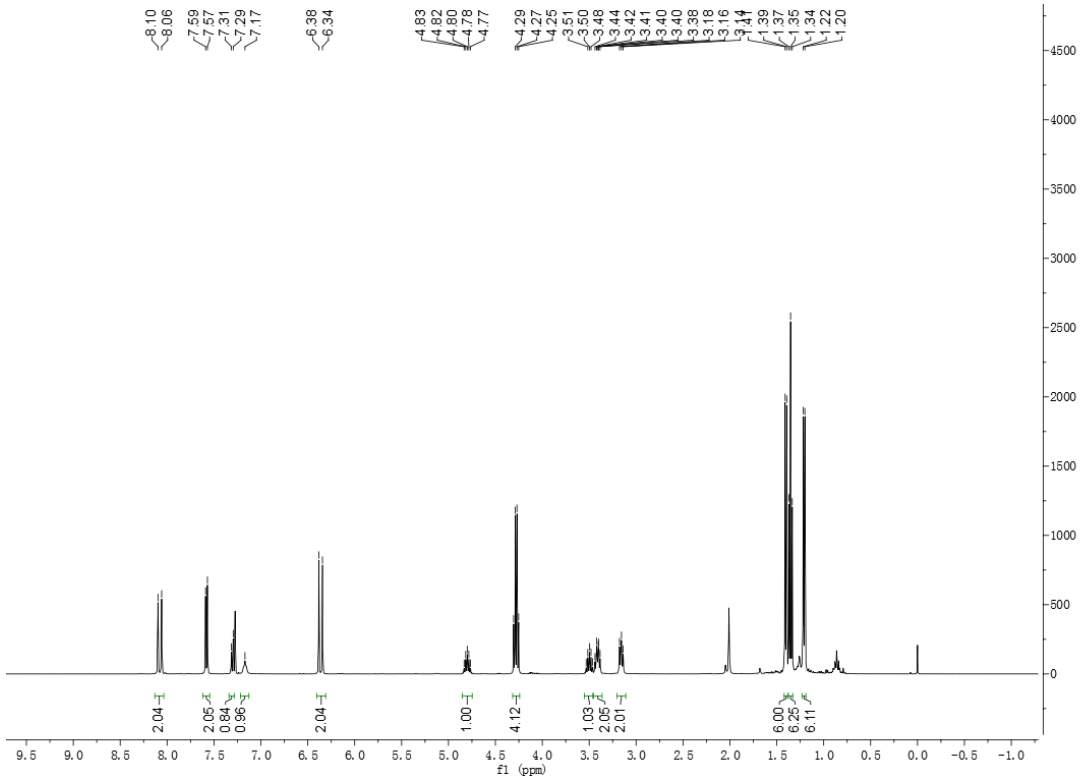
46.69
45.59
40.26
33.46
31.51
20.99
20.18
17.63
f1 (ppm)


CO2Et
H
N
OA
CO2Et

3v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8.06
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7.57
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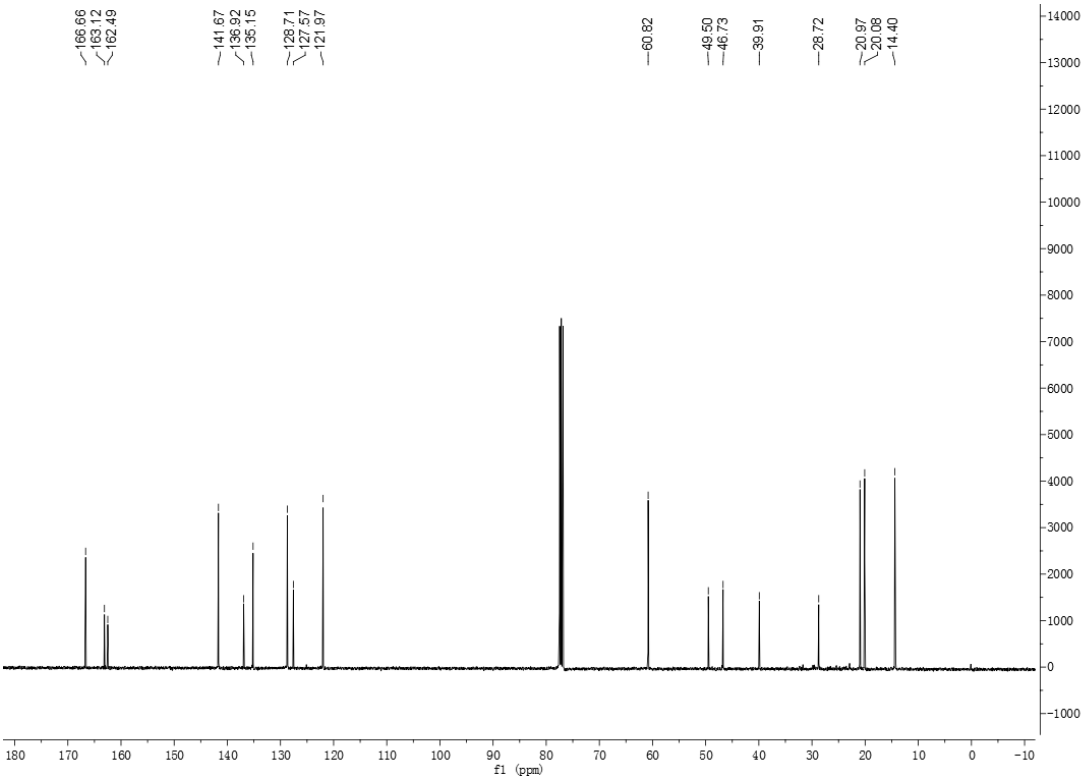
7.29
7.17
6.38
6.34
4.83
4.82
4.80
4.78
4.77
4.29
4.27
4.25
3.51
3.50
3.48
3.44
3.42
3.41
3.40
3.40
3.38
3.18
3.16
3.14
1.41
1.39
1.37
1.35
1.34
1.22
1.20
2.04
2.05
0.84
0.96
2.04
1.00
4.12
1.03
2.05
2.01
6.00
6.25
6.11
f1 (ppm)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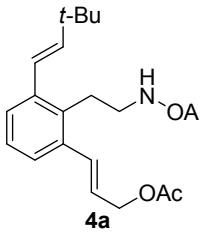
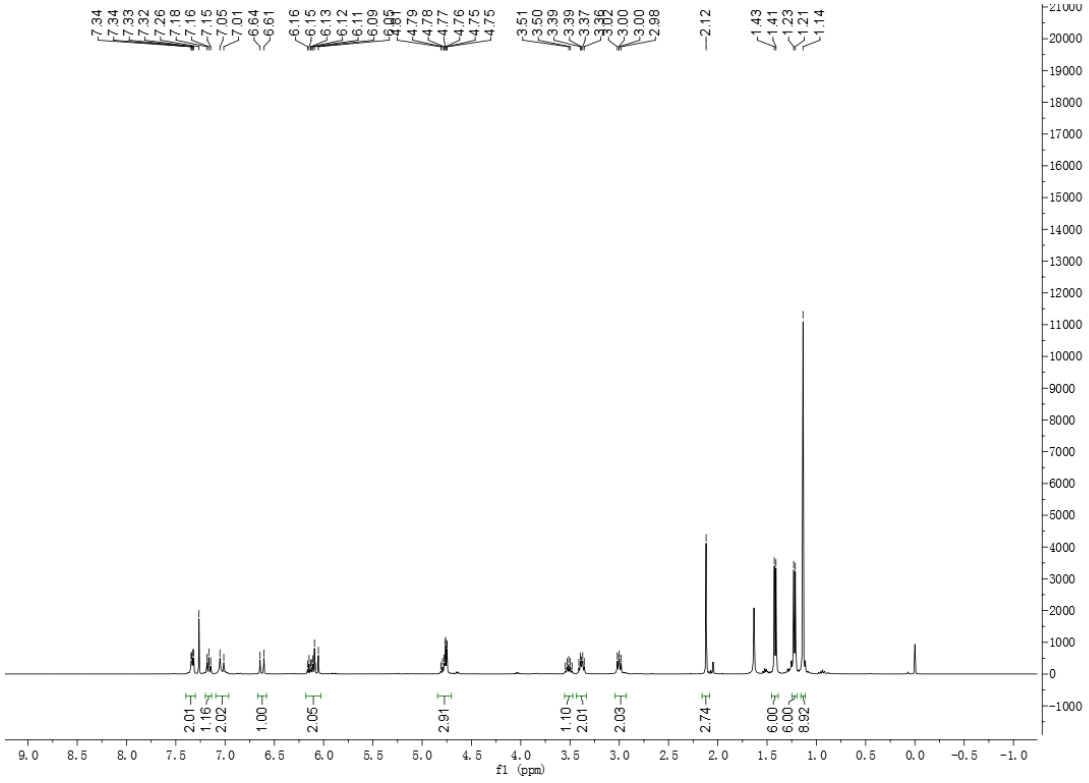
162.49
141.67
136.92
135.15
128.71
127.57
121.97
60.82
49.50
46.73
39.91
28.72
20.97
20.08
14.40
f1 (ppm)


t-Bu
H
N
OA
OAc

4a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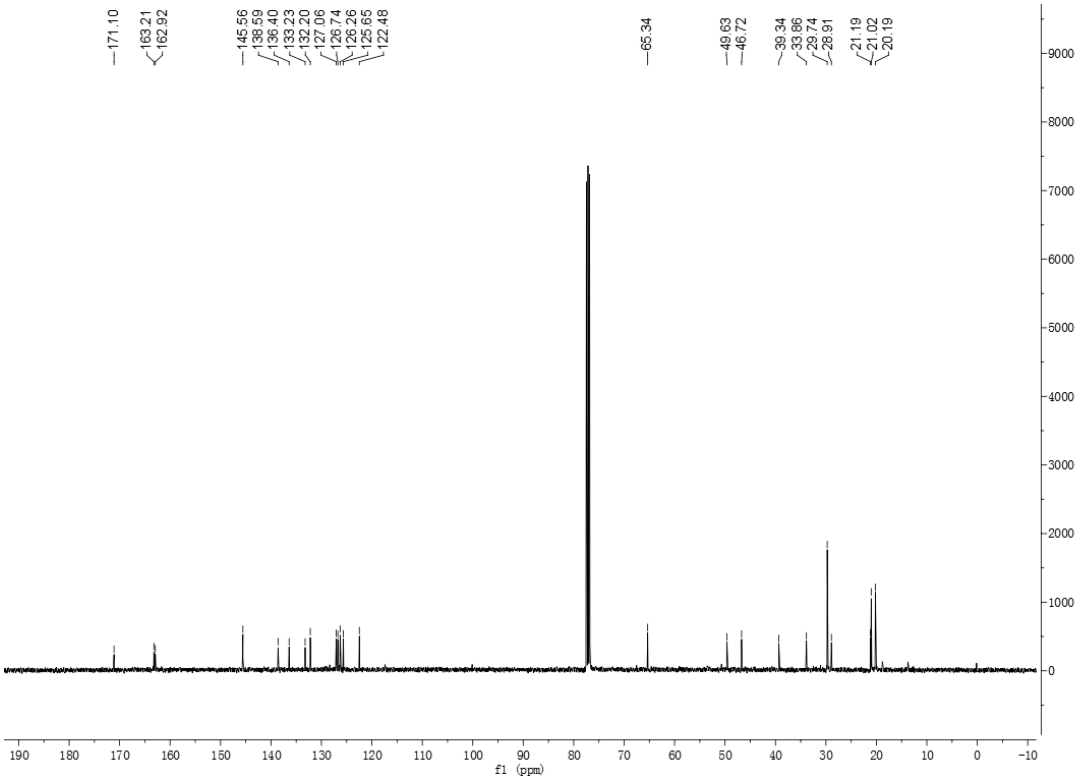
7.32
7.26
7.18
7.16
7.15
7.05
7.01
6.64
6.61
6.16
6.15
6.13
6.12
6.11
6.09
4.81
4.79
4.78
4.77
4.76
4.75
3.51
3.50
3.39
3.39
3.37
3.36
3.02
3.00
3.00
2.98
2.12
1.43
1.41
1.23
1.21
1.14
2.01
1.16
2.02
1.00
2.05
2.91
1.10
2.01
2.03
2.74
6.00
6.00
8.92
f1 (ppm)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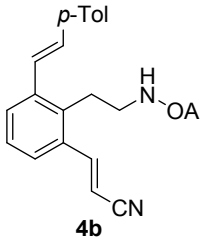
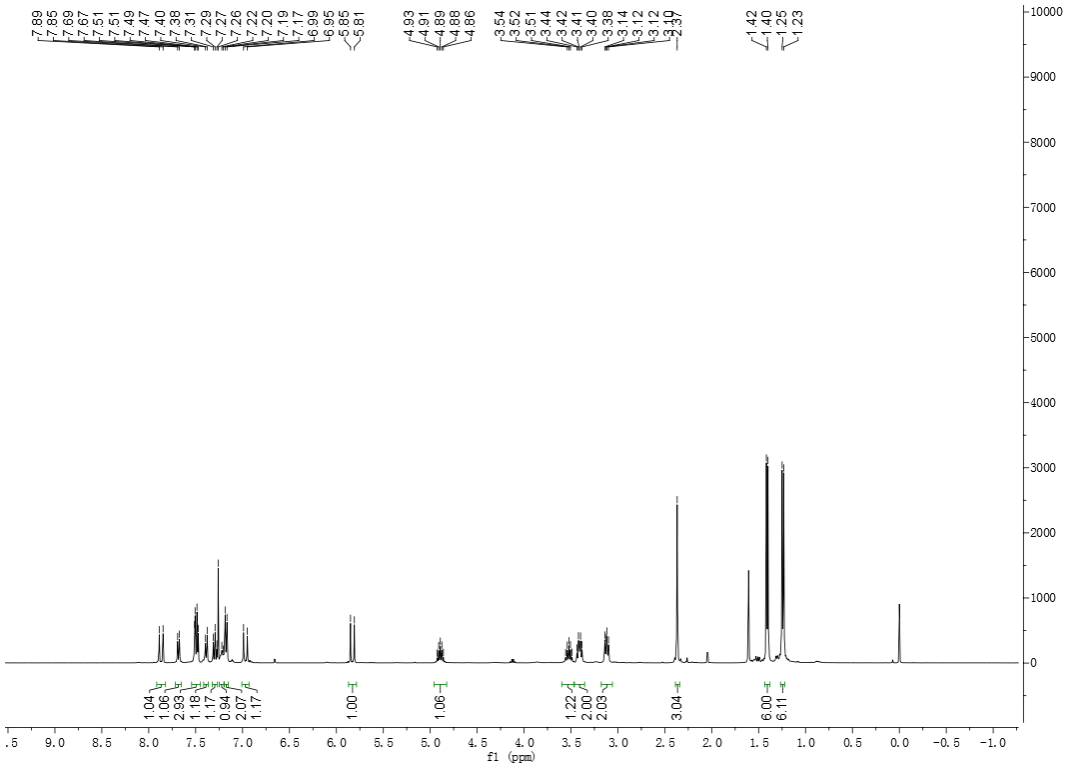
162.92
145.56
138.59
136.40
133.23
132.20
127.06
126.74
126.26
125.65
122.48
65.34
49.63
46.72
39.34
33.86
29.74
28.91
21.19
21.02
20.19
f1 (ppm)


p-Tol
H
N
OA
CN

4b


7.89
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7.67
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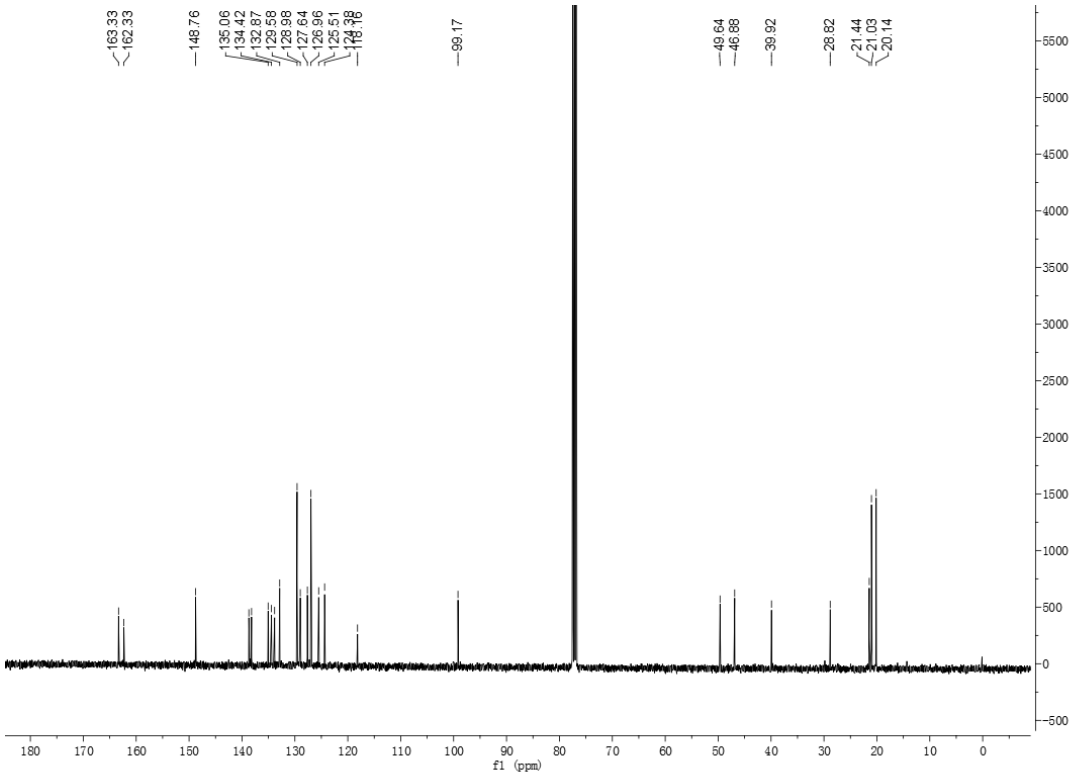
7.49
7.47
7.40
7.38
7.31
7.29
7.27
7.26
7.22
7.20
7.19
7.17
6.99
6.95
5.85
5.81
4.93
4.91
4.89
4.88
4.86
3.54
3.52
3.51
3.44
3.42
3.41
3.40
3.38
3.14
3.12
3.12
3.10
2.37
1.42
1.40
1.25
1.23
1.04
1.06
2.93
1.19
1.17
0.94
2.07
1.17
1.00
1.06
1.22
2.00
2.03
3.04
6.00
6.11
f1 (ppm)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162.33
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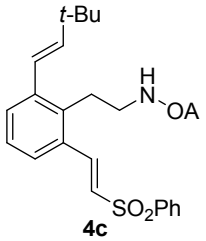
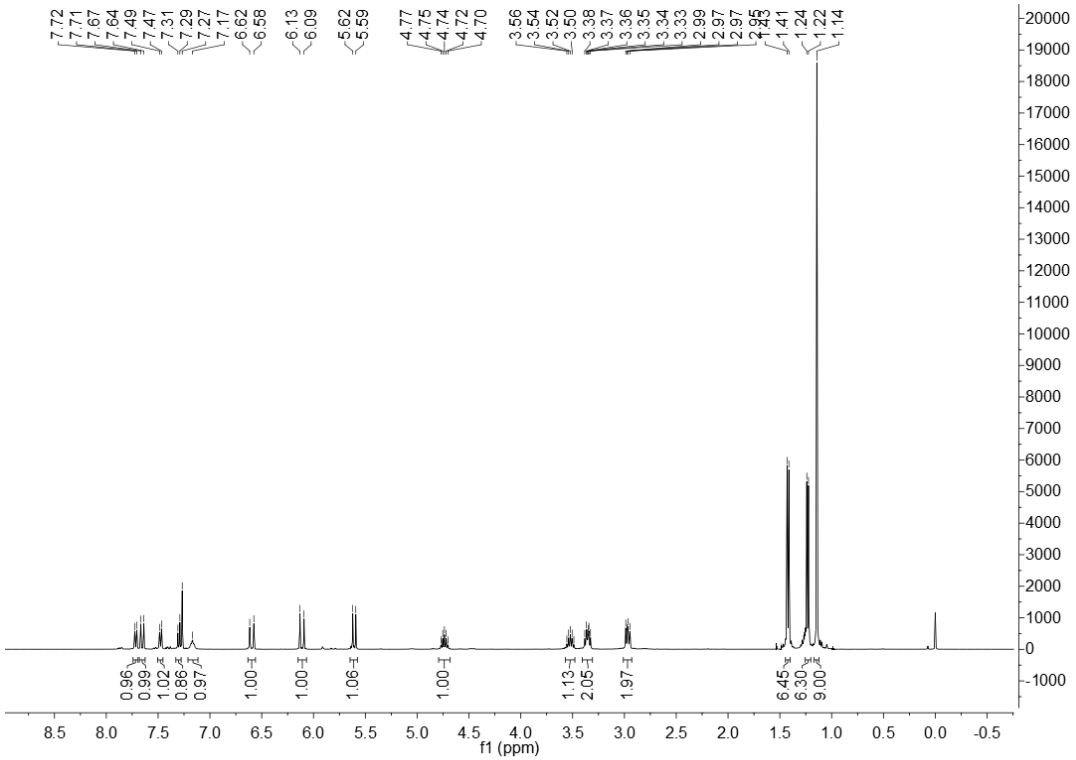
135.06
134.42
132.87
129.58
128.98
127.64
126.96
125.51
99.17
49.64
46.88
39.92
28.82
21.44
21.03
20.14
f1 (ppm)


t-Bu
H
N
OA
SO2Ph

4c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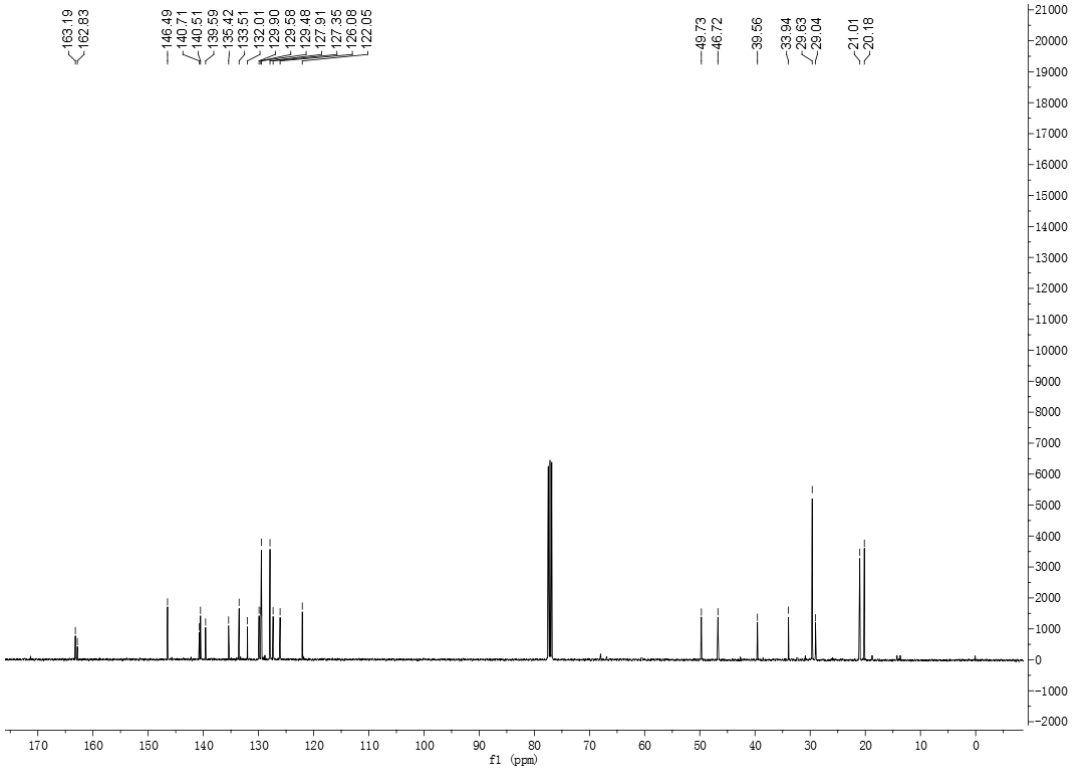
7.47
7.31
7.29
7.27
7.17
6.62
6.58
6.13
6.09
5.62
5.59
4.77
4.75
4.74
4.72
4.70
3.56
3.54
3.52
3.50
3.38
3.37
3.36
3.35
3.34
3.33
2.99
2.97
2.97
2.95
1.41
1.24
1.22
1.14
0.96
0.99
1.02
0.86
0.97
1.00
1.00
1.06
1.00
1.13
2.05
1.97
6.45
6.30
9.00
f1 (ppm)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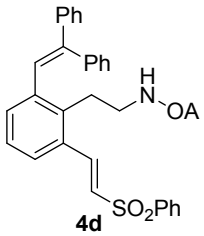
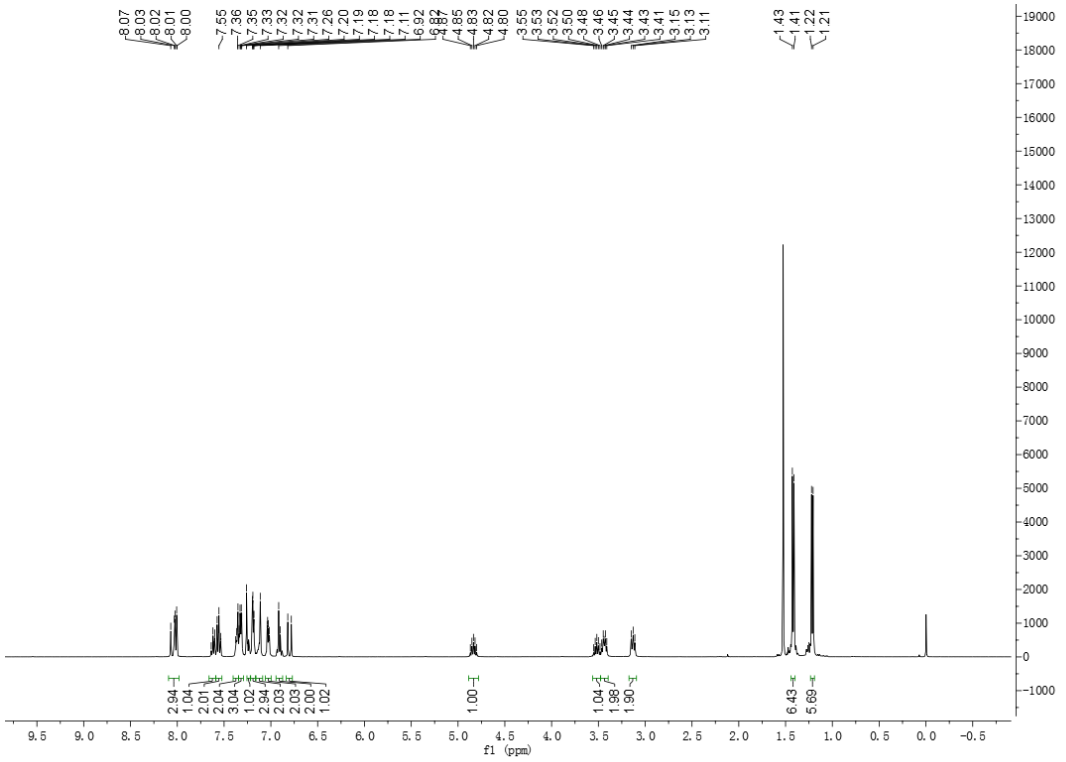
133.51
132.01
129.90
129.58
129.48
127.91
127.35
126.08
122.05
49.73
46.72
39.56
33.94
29.63
29.04
21.01
20.18
f1 (ppm)


Ph
Ph
H
N
OA
SO2Ph

4d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8.03
8.02
8.01
8.00
7.55
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7.35
7.33
7.32
7.32
7.31
7.26
7.20
7.19
7.18
7.18
7.11
6.92
4.85
4.83
4.82
4.80
3.55
3.53
3.52
3.50
3.48
3.46
3.45
3.44
3.43
3.41
3.15
3.13
3.11
1.43
1.41
1.22
1.21
2.94
1.04
2.01
2.04
3.04
1.02
2.94
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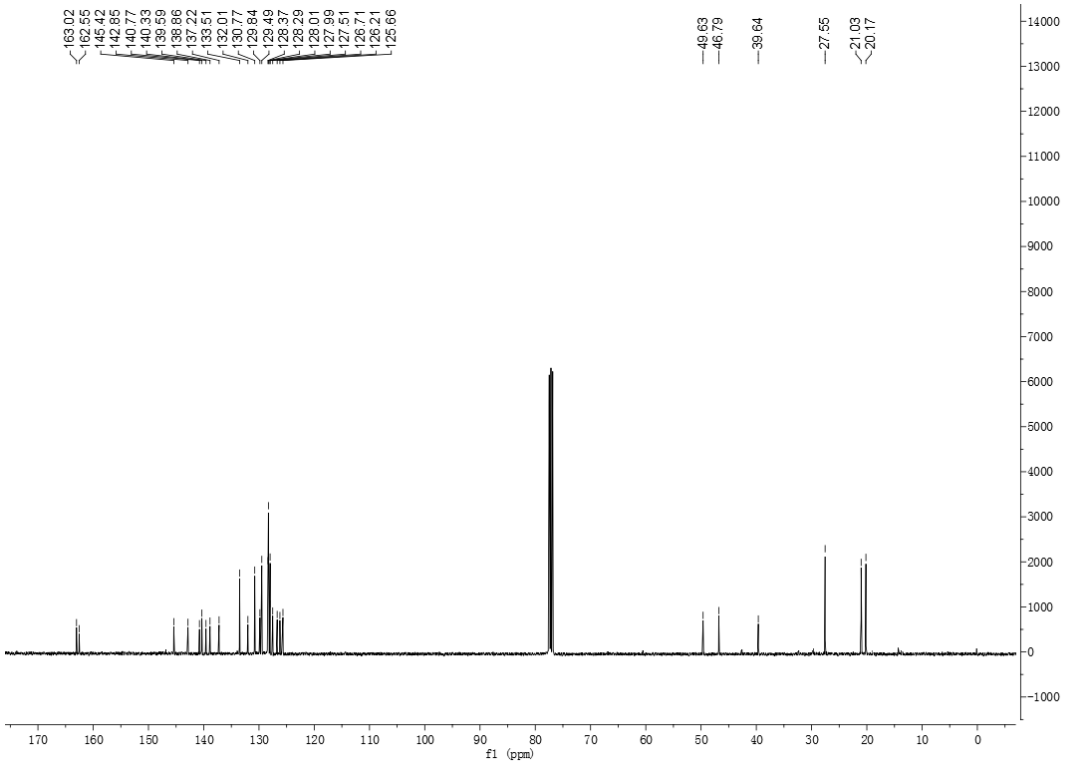
2.00
1.02
1.00
1.04
1.98
1.90
6.43
5.69
f1 (ppm)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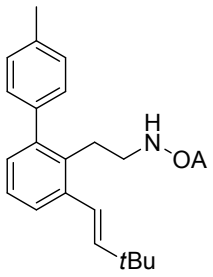
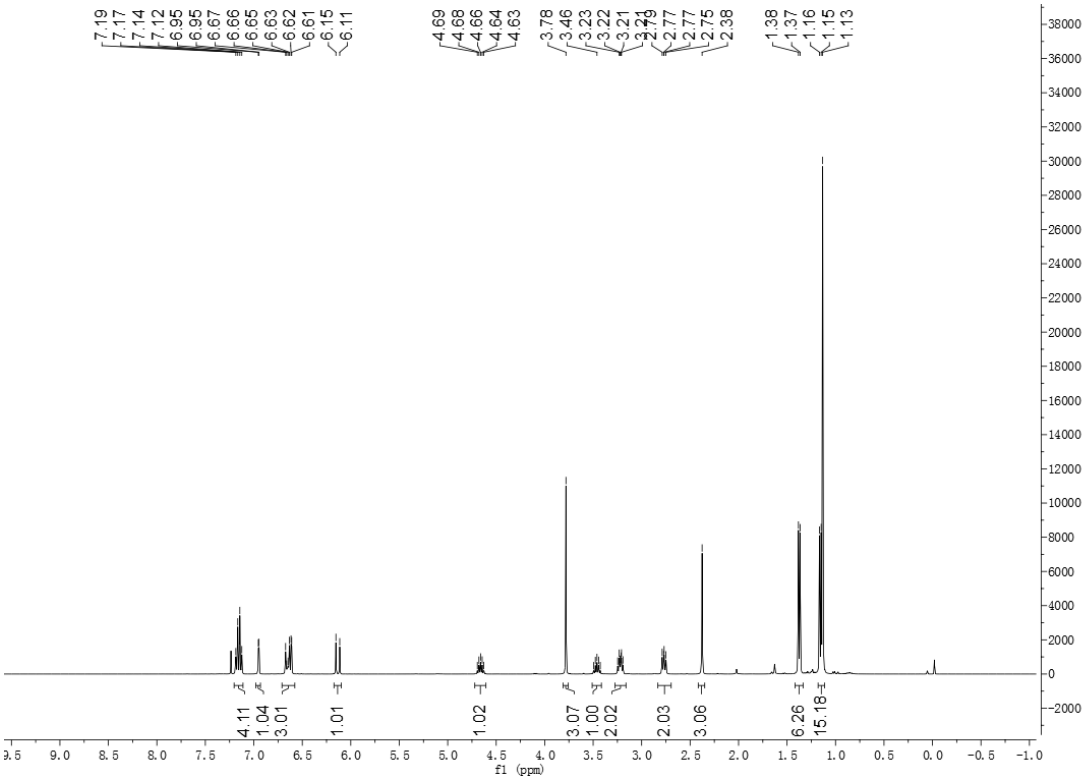
138.86
137.22
133.51
132.01
130.77
129.84
129.49
128.37
128.29
128.01
127.99
127.51
126.71
126.21
125.66
49.63
46.79
39.64
27.55
21.03
20.17
f1 (ppm)


H
N
OA
tBu

5k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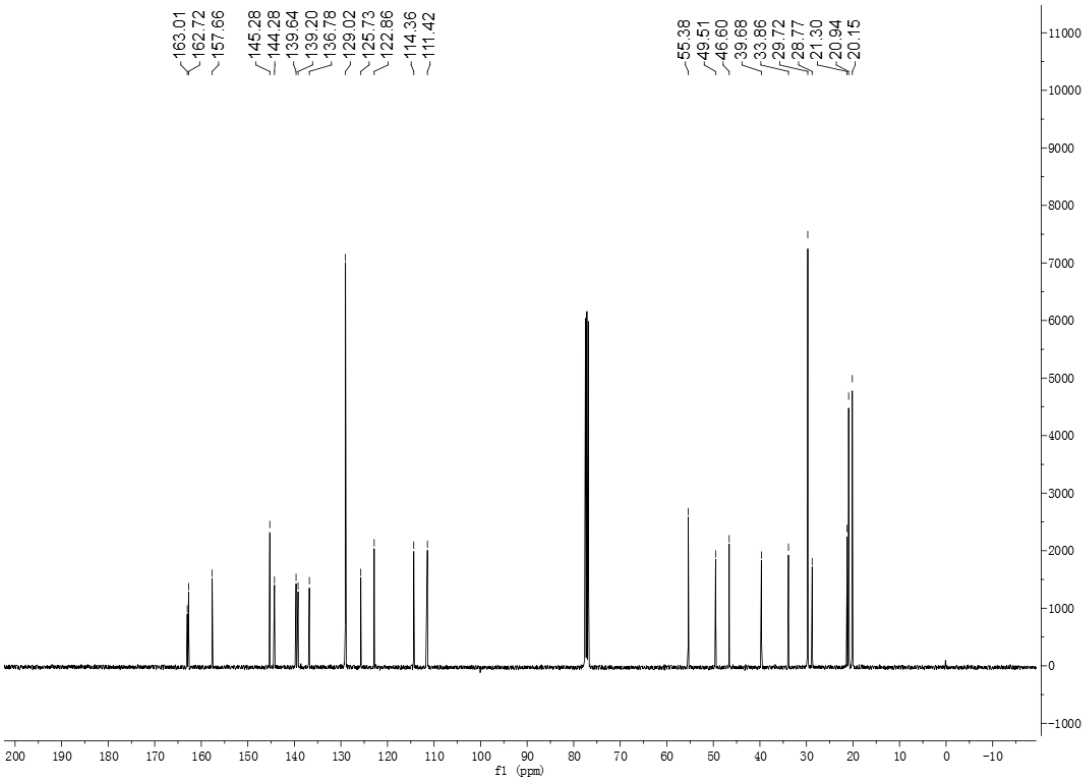
6.63
6.62
6.61
6.15
6.11
4.69
4.68
4.66
4.64
4.63
3.78
3.46
3.23
3.22
3.21
3.21
2.79
2.77
2.77
2.75
2.38
1.38
1.37
1.16
1.15
1.13
4.11
1.04
3.01
1.01
1.02
3.07
1.00
2.02
2.03
3.06
6.26
15.18
f1 (ppm)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139.20
136.78
129.02
125.73
122.86
114.36
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55.38
49.51
46.60
39.68
33.86
29.72
28.77
21.30
20.94
20.15
f1 (ppm)