Supporting information

Highly Site Selectivity Sequential Alkenylation of Oxalyl Amide Protected Phenylpropylamine Derivatives via a Seven-Membered Palladacycle

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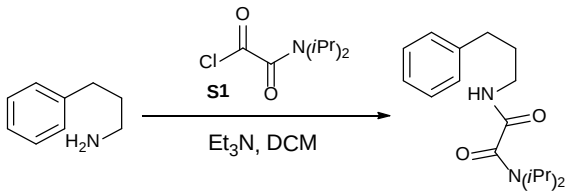
1. Reagents: Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. Column chromatography purifications were performed using 300–400 mesh silica gel.

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. Preparation of oxalamide substrates



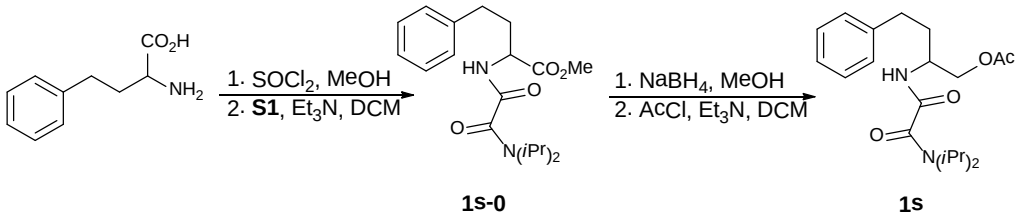
3.1. Preparation of N, N–Diisopropyloxamoyl chloride S1[1]

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 15 min, triethylamine (7.30 mL, 52.5 mmol, 1.05 equiv) was added dropwised. The solution was warmed to room temperature and stirred for 6 hrs. The excess of oxalyl chloride and the solvent were removed under reduce pressure and 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.

3.2. General procedures for the preparation of oxalamide substrates 1n–1y (except 1s) [2]

A solution of amine (20 mmol, 1.0 eq) 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, after stirring for 5 min, triethylamine (2.92 ml, 21 mmol, 1.05 equiv) was added dropwise and 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 with >90% yield.

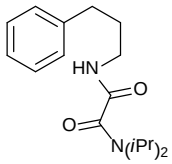
3.3. Preparation of 1s[3]



To a solution of 2–amino–4–phenylbutanoic acid (3.58 g, 20 mmol, 1.0 eq) in MeOH (30 mL), at

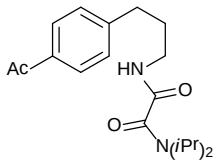
0 °C, was added $SOCl_2$ (4.35 mL, 60 mmol, 3.0 eq) dropwise. The resulting mixture was allowed to stir from 0 °C to room temperature overnight. The solvent was removed under reduced pressure afford a white solid, which was used directly for next step. The second step followed the general oxalamide coupling procedure, to give compound **1s–0** 3.90 g, 56%.

To a solution of **1s–0**(3.48 g, 10 mmol, 1.0 eq) in MeOH (10 ml) at room temperature, was added $NaBH_4$ (0.76 g, 20 mmol, 2.0 eq) in portions. Water was added after the reaction was determined by TLC, and the mixture was extracted with DCM. The combined organic layers was washed with water and brine, dried over anhydrous Na_2SO_4, the resulting solution was used directly for next step. The solution was treated with AcCl (0.78 mL, 11 mmol, 1.1 eq) and Et_3N (2.78 mL, 20 mmol, 2.0 eq) at room temperature overnight. Water was added and the mixture was extracted with DCM. The combined organic layers was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to give the product **1s** 3.15 g, 87%.



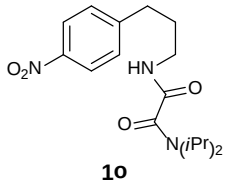
1a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.28 (m, 2H), 7.22–7.19 (m, 3H), 7.12 (br, 1H), 4.79–4.73 (m, 1H), 3.55–3.50 (m, 1H), 3.36–3.31 (m, 2H), 2.71–2.67 (m, 2H), 1.94–1.87 (m, 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 163.36, 163.26, 141.32, 128.57, 128.49, 126.13, 49.77, 46.67, 39.03, 33.28, 30.92, 20.98, 20.19; HRMS Calcd for $C_{17}H_{25}N_2O_2$ [M–H$^+$]: 289.1916; Found: 289.1913.



1n

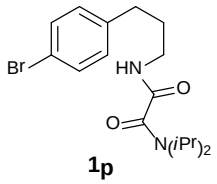
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.2 Hz, 2H), 7.40 (br, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 4.63 (m, 1H), 3.48 (m, 1H), 3.29 (m, 2H), 2.72–2.67 (m, 2H), 2.54 (s, 3H), 1.91–1.84 (m, 2H), 1.38 (d, *J* = 6.8 Hz, 6H), 1.20 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.87, 163.57, 163.54, 147.17, 135.26, 128.67, 128.64, 49.83, 46.49, 38.80, 33.18, 30.48, 26.60, 20.88, 20.12; HRMS Calcd for $C_{19}H_{29}N_2O_3$ [M+H+]: 333.2178; Found: 333.2178.



1o

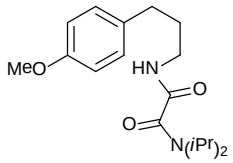
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.6 Hz, 2H), 7.41 (br, 1H), 7.35 (d, *J* = 8.5 Hz, 2H), 4.72–4.63 (m, 1H), 3.52 (m, 1H), 3.33 (q, *J* = 6.7 Hz, 2H), 2.80–2.76 (m, 2H), 1.96–1.89 (m, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.57, 149.26,

146.58, 129.29, 123.83, 49.88, 46.63, 38.74, 33.10, 30.47, 20.92, 20.15; HRMS Calcd for C17H26N3O4 [M+H+]: 336.1923; Found: 336.1917.



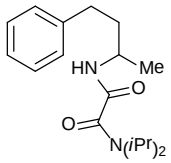
1p

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (br, 1H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.03 (d, *J* = 8.1 Hz, 2H), 4.65–4.62 (m, 1H), 3.50–3.47 (m, 1H), 3.29–3.24 (m, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.87–1.81 (m, 2H), 1.39 (d, *J* = 6.7 Hz, 6H), 1.21 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.51, 140.30, 131.53, 130.20, 119.78, 49.83, 46.53, 38.80, 32.61, 30.70, 20.92, 20.16; HRMS Calcd for $C_{17}H_{24}BrN_2O_2$ [M–H$^+$]: 367.1021; Found: 367.1028.



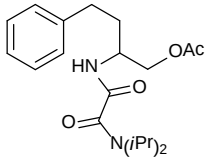
1q

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (d, *J* = 8.6 Hz, 2H), 6.95 (br, 1H), 6.82 (d, *J* = 8.6 Hz, 2H), 4.81–4.74 (m, 1H), 3.78 (s, 3H), 3.53–3.48 (m, 1H), 3.32–3.27 (m, 2H), 2.63 – 2.59 (m, 2H), 1.88 – 1.81 (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 163.17, 162.96, 157.90, 133.22, 129.28, 113.88, 55.27, 49.60, 46.59, 38.88, 32.24, 31.04, 20.88, 20.08; HRMS Calcd for $C_{18}H_{27}N_2O_3$ [M–H$^+$]: 319.2022; Found: 319.2045.



1r

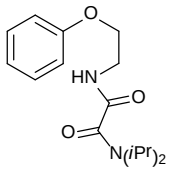
^{1}H NMR (400 MHz, CDCl3) δ 7.29 (s, 1H), 7.25 (s, 1H), 7.22 –7.14 (m, 4H), 7.04 (s, 1H), 4.73 (dt, J = 13.3, 6.6 Hz, 1H), 4.01 (dt, J = 14.5, 7.1 Hz, 1H), 3.51 (dt, J = 13.6, 6.8 Hz, 1H), 2.66 (m, 2H), 1.91–1.70 (m, 2H), 1.43 (dd, J = 6.8, 1.5 Hz, 6H), 1.23 (t, J = 7.0 Hz, 9H). ^{13}C NMR (101 MHz, CDCl3) δ 163.49, 162.77, 141.67, 128.50, 128.43, 126.00, 49.77, 46.59, 45.30, 38.48, 32.57, 21.00, 20.93, 20.74, 20.24, 20.18.



1s

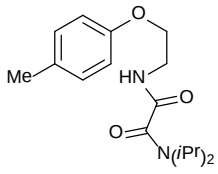
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 7.5 Hz, 2H), 7.15 (t, *J* = 8.6 Hz, 3H), 4.69–4.62 (m, 1H), 4.19–4.15 (m, 1H), 4.12–4.08 (m, 2H), 3.53–3.47 (m, 1H), 2.73–2.58 (m, 2H), 2.03 (s, 3H), 1.88–1.82 (m, 2H), 1.42–1.40 (m, 6H), 1.23–1.20 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.88,

163.27, 141.02, 128.51, 128.38, 126.12, 65.69, 49.86, 48.06, 46.52, 33.08, 32.20, 20.87, 20.19, 20.11; HRMS Calcd for $C_{20}H_{29}N_2O_4$ [M–H$^+$]: 361.2127; Found: 361.2142.



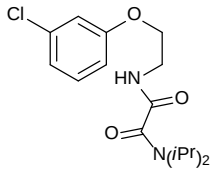
1t

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (br, 1H), 7.31–7.26 (m, 2H), 6.96 (t, *J* = 7.4 Hz, 1H), 6.91–6.88 (m, 2H), 4.76–4.69 (m, 1H), 4.07 (t, *J* = 5.2 Hz, 2H), 3.72–3.68 (m, 2H), 3.55–3.48 (m, 1H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.45, 162.84, 158.45, 129.64, 121.28, 114.55, 66.19, 49.76, 46.69, 38.93, 20.96, 20.16; HRMS Calcd for $C_{17}H_{25}N_2O_2$ [M–H$^+$]: 291.1709; Found: 291.1731.



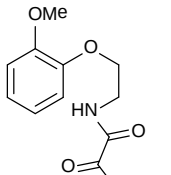
1u

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (br, 1H), 7.06 (d, *J* = 8.5 Hz, 2H), 6.78 (d, *J* = 8.5 Hz, 2H), 4.71–4.67 (m, 1H), 4.06–4.02 (m, 2H), 3.71–3.65 (m, 2H), 3.53–3.47 (m, 1H), 2.27 (s, 3H), 1.41 (d, *J* = 6.8 Hz, 6H), 1.21 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.46, 162.93, 156.32, 130.47, 130.03, 114.39, 66.32, 49.77, 46.63, 38.93, 20.94, 20.57, 20.15; HRMS Calcd for $C_{17}H_{25}N_2O_3$ [M–H$^+$]: 305.1865; Found: 305.1882.



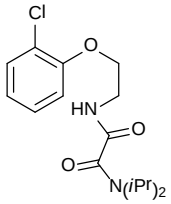
1v

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (br, 1H), 7.17 (t, *J* = 8.1 Hz, 1H), 6.92 (d, *J* = 7.9 Hz, 1H), 6.86 (s, 1H), 6.76 (d, *J* = 8.1 Hz, 1H), 4.69–4.62 (m, 1H), 4.03 (t, *J* = 5.2 Hz, 2H), 3.69–3.65 (m, 2H), 3.52–3.45 (m, 1H), 1.39 (d, *J* = 6.8 Hz, 6H), 1.20 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.51, 162.94, 159.17, 134.94, 130.36, 121.37, 115.01, 112.95, 66.47, 49.81, 46.62, 38.72, 20.90, 20.11; HRMS Calcd for $C_{16}H_{22}ClN_2O_3$ [M–H$^+$]: 325.1319; Found: 325.1345.



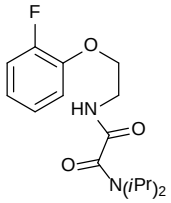
1w

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (br, 1H), 6.96–6.88 (m, 4H), 4.66–4.61 (m, 1H), 4.13–4.10 (m, 2H), 3.86 (s, 3H), 3.69–3.68 (m, 2H), 3.51–3.46 (m, 1H), 1.42 (d, *J* = 6.6 Hz, 6H), 1.21 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.62, 163.01, 150.07, 147.80, 122.50, 121.03, 115.48, 111.99, 68.63, 55.86, 49.77, 46.55, 38.94, 20.94, 20.13; HRMS Calcd for $C_{17}H_{27}N_2O_4$ [M+H$^+$]: 323.1971; Found: 323.1972.



1x

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.30 (br, 1H), 7.22–7.18 (m, 1H), 6.93–6.89 (m, 2H), 4.69–4.66 (m, 1H), 4.13 (t, *J* = 5.2 Hz, 2H), 3.77–3.73 (m, 2H), 3.52–3.49 (m, 1H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.63, 162.87, 154.07, 130.51, 127.92, 123.35, 122.22, 114.04, 67.80, 49.81, 46.69, 38.81, 20.98, 20.17; HRMS Calcd for $C_{16}H_{22}ClN_2O_3$ [M–H$^+$]: 325.1319; Found: 325.1346.

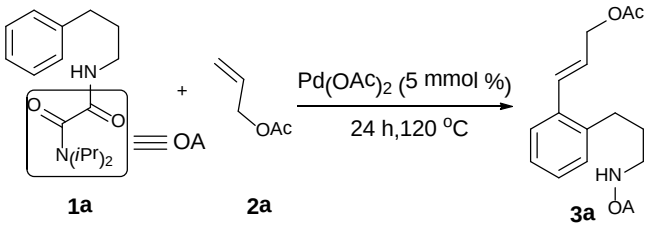


1y

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (br, 1H), 7.11 – 7.01 (m, 2H), 7.09–7.02 (m, 2H), 4.68–4.64 (m, 1H), 4.14 (t, *J* = 5.2 Hz, 2H), 3.74–3.70 (m, 2H), 3.54–3.47 (m, 1H), 1.41 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.66, 162.91, 154.14, 151.70, 146.60, 146.50, 124.52, 124.48, 122.01, 121.94, 116.58, 116.40, 115.56, 68.04, 49.85, 46.67, 38.89, 20.95, 20.16; HRMS Calcd for $C_{16}H_{22}FN_2O_3$ [M–H$^+$]: 309.1614; Found: 309.1639.

4. Screening of Solvents

Table S1. Screening of Solvents



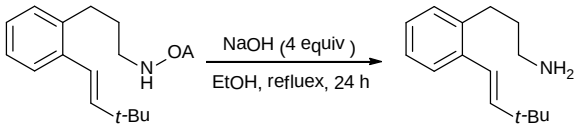
1a **2a** **3a**

Entry	solvent	Yield[a]
1	dioxane	54
2	HFIP	38
3	PhCl	67
4	CH_3CN	63

5	Toluene	66
6	H_2O	47
7	THF	78
8	*t*–Amyl-OH	77
9	DMF	55
10	DCE	84

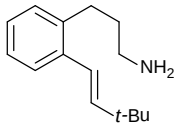
[a] Reactions were carried out on a 0.2 mmol scale; yield was based on LC using acetophenone as the internal standard.

5. Removal of the Directing Group[3]



3c **3c-2** (87%)

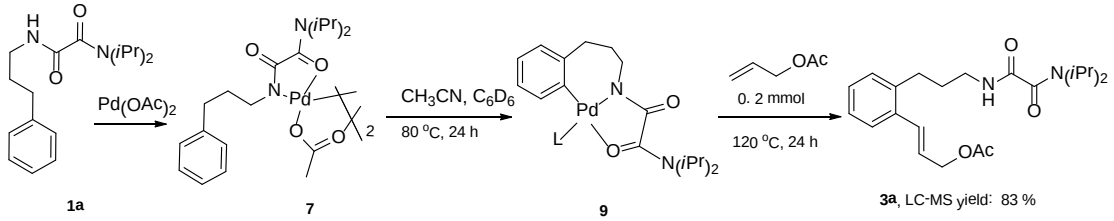
Compound **3c** (74.4 mg, 0.2 mmol) was dissolved in a mixture of EtOH (1 mL), NaOH (32 mg, 0.8 mmol, 4 eq) was then added. The mixture was heated to 80 ^{o}C and stirred for 12 hours. Water was added and the mixture was extracted with DCM. The combined organic layers was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the desired product **3c-2** with 37.8 mg, 87 % yield.



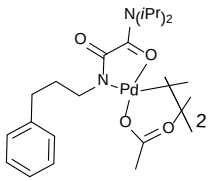
3c-2 (87%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 6.9 Hz, 1H), 7.16 – 7.12 (m, 3H), 6.50 (d, *J* = 16.0 Hz, 1H), 6.10 (d, *J* = 15.9 Hz, 1H), 3.99 (br, 2H), 2.84 (t, *J* = 7.3 Hz, 2H), 2.79 – 2.68 (m, 2H), 1.98 – 1.77 (m, 2H), 1.12 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.22, 137.99, 137.04, 129.45, 127.07, 126.63, 126.29, 122.03, 40.73, 33.76, 31.50, 30.38, 29.78.

6. Synthesis of Palladacycle 7 and 9

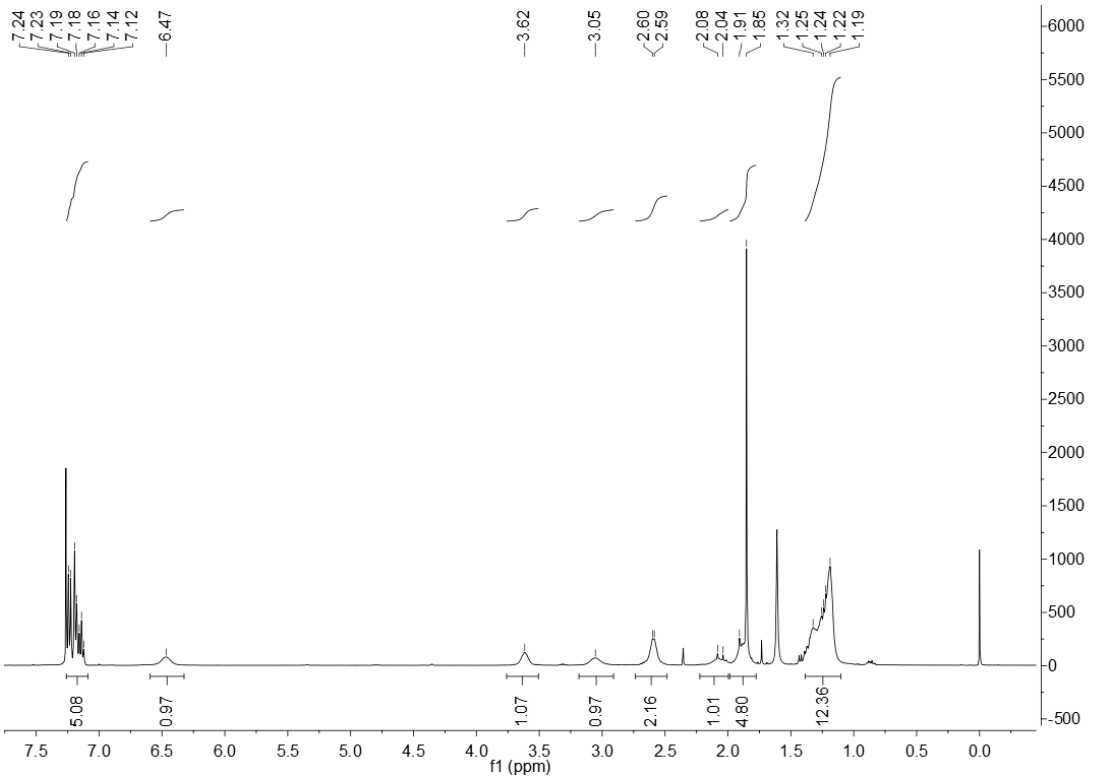


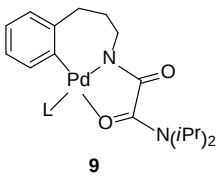
1a **7** **9** **3a**, LC-MS yield: 83 %



7

$Pd(OAc)_2$ (0.112 g, 0.5 mmol) and **1a** (0.145 g, 0.5 mmol) were added to toluene (2 mL). The reaction was stirred in a sealed tube at 40 °C for 12 h, and then the solvent was removed in vacuum. To the crude product, toluene (3 mL) was added, and then the purified product was collected by filtration, washed with hexane (0.5 mL). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.11 (m, 5H), 6.47 (s, 1H), 3.62 (s, 1H), 3.05 (s, 1H), 2.59 (d, *J* = 5.6 Hz, 2H), 2.08 – 2.04(m, 1H), 1.91 – 1.85 (m, 5H), 1.32 – 1.19 (m, 12H).

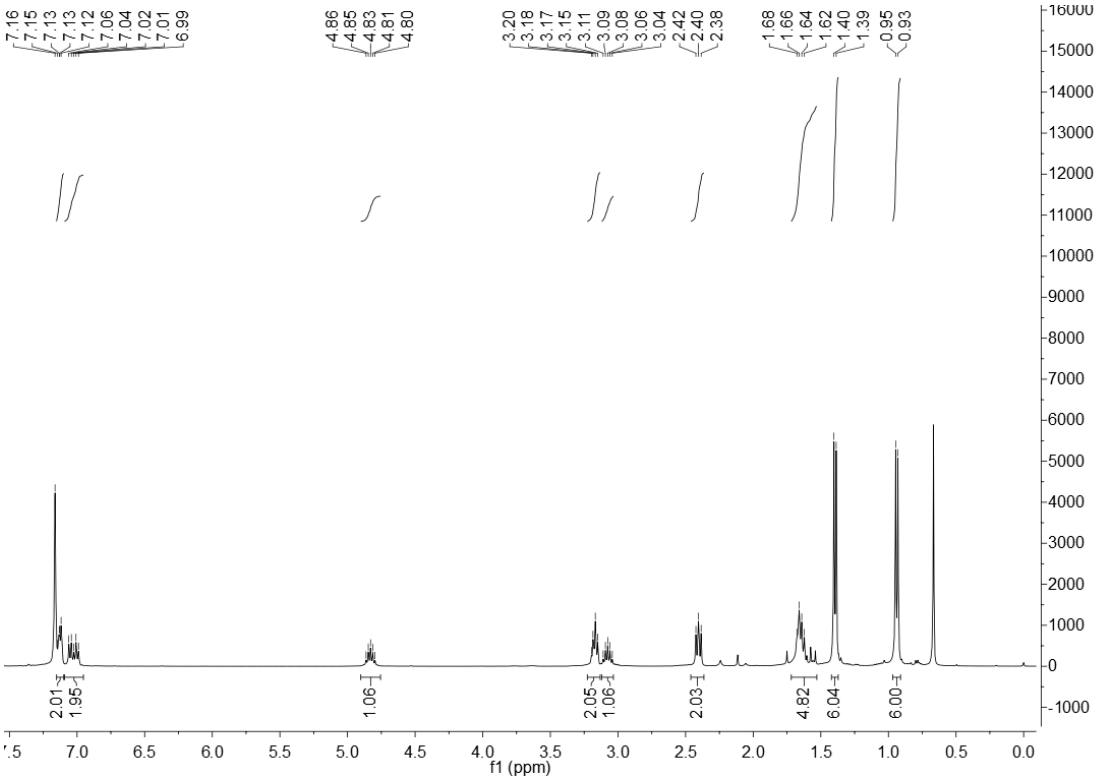




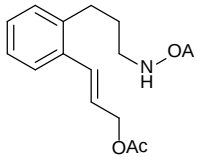
9

A mixture of **7** (22.7 mg, 0.05 mmol), acetonitrile(4.1 mg, 0.1 mmol, 2 equiv) and Benzene-d_6 (0.5 mL) in NMR tube was heated at 80 ^{o}C for 24 hrs respectively. The reaction mixture was cooled to rt and ^{1}H NMR was tested. From the ^{1}H NMR spectra of reaction, we can conclude that C–H activation can take place. The result showed the palladium intermediate **9** might be formed during the catalytical cycle. ^{1}H NMR (400 MHz, C_6D_6) δ 7.16 – 7.12 (m, 2H), 7.06 – 6.99 (m, 1H), 4.83 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.17 (dd, *J* = 12.7, 5.8 Hz, 2H), 3.08 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.48 – 2.36 (m, 1H), 1.65 (dd, *J* = 14.9, 7.2 Hz, 2H), 1.39 (d, *J* = 6.8 Hz, 6H), 0.94 (d, *J* = 6.7 Hz,

6H).

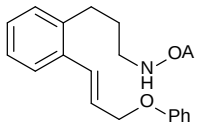


2a (0.2 mmol, 2 equiv) was added in the NMR tube contained the mixture mentioned above, the mixture was heated at 120 ^{o}C for 12 hrs respectively. **3a** was obtained in 83 % yield based on LC-MS using acetophenone as the internal standard.



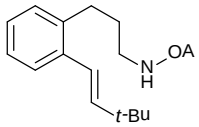
3a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 6.5 Hz, 1H), 7.28-7.13 (m, 4H), 6.91 (d, *J* = 15.6 Hz, 1H), 6.15 (dt, *J* = 15.4, 6.4 Hz, 1H), 4.75 (d, *J* = 6.3 Hz, 2H), 4.70-4.60 (m, 1H), 3.51 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.32 (dd, *J* = 13.1, 6.6 Hz, 2H), 2.77-2.70 (m, 2H), 2.10(s, 3H), 1.86-1.76 (m, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.07, 163.55, 163.22, 139.05, 135.12, 131.98, 129.66, 128.30, 126.71, 126.51, 125.28, 65.47, 49.76, 46.65, 39.02, 30.66, 30.58, 21.18, 21.00, 20.20. HRMS Calcd for $C_{22}H_{32}N_2O_4$ [M+Na^+]: 411.2260; Found: 411.2262.



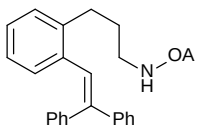
3b

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56–7.46 (m, 1H), 7.36–7.30 (m, 2H), 7.24 – 7.19 (m, 3H), 7.01-6.95 (m, 4H), 6.87 (br, 4H), 6.33 (dt, *J* = 15.8, 5.6 Hz, 1H), 4.85–4.72 (m, 3H), 3.53 (dt, *J* = 13.7, 6.8 Hz, 1H), 3.28 (dd, *J* = 13.4, 7.0 Hz, 2H), 2.79–2.67 (m, 2H), 1.78 (dt, *J* = 14.8, 7.4 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 163.38, 163.08, 158.66, 138.89, 135.35, 130.34, 129.68, 129.63, 129.54, 128.11, 126.72, 126.57, 126.47, 121.03, 115.04, 68.64, 49.73, 46.71, 39.05, 30.71, 30.56, 21.01, 20.20. HRMS Calcd for $C_{26}H_{34}N_2O_3$ [M+Na^+]: 445.2467; Found: 411.2472.



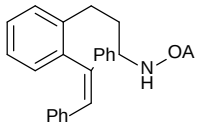
3c

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.36 (m, 1H), 7.21–7.07 (m, 3H), 6.93 (br, 1H), 6.51 (d, *J* = 15.9 Hz, 1H), 6.11 (d, *J* = 15.9 Hz, 1H), 4.81 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.31 (dd, *J* = 13.4, 6.8 Hz, 2H), 2.80–2.67 (m, 2H), 1.90–1.78 (m, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H), 1.13 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.13, 163.26, 162.98, 144.14, 138.29, 137.05, 129.46, 127.05, 126.59, 126.26, 122.06, 49.68, 46.73, 39.18, 33.75, 31.07, 30.25, 29.78, 21.02, 20.20. HRMS Calcd for $C_{23}H_{36}N_2O_2$ [M+Na^+]: 395.2674; Found: 411.2684.



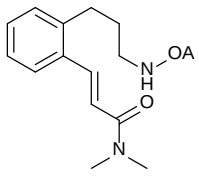
3d

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 5H), 7.22 – 7.05 (m, 7H), 7.00 (s, 1H), 6.96 (br, 1H), 6.86 (m, 2H), 4.76 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.55 – 3.45 (m, 1H), 3.34 – 3.29 (m, 2H), 2.75 – 2.71 (m, 2H), 1.92 – 1.85 (m, 1H), 1.42 (t, *J* = 6.5 Hz, 6H), 1.21 (t, *J* = 4.6 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.28, 163.08, 143.82, 143.58, 141.31, 140.18, 140.00, 136.62, 130.83, 130.53, 129.04, 128.60, 128.50, 128.36, 128.28, 128.24, 127.73, 127.32, 127.16, 126.63, 126.16, 125.78, 49.70, 46.69, 39.28, 33.29, 31.12, 30.00, 20.99, 20.20, 14.33. HRMS Calcd for $C_{31}H_{36}N_2O_2$ [M+Na^+]: 491.2674; Found: 411.2677.



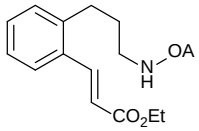
3e

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 6H), 7.25 – 7.09 (m, 6H), 7.07 (s, 1H), 6.94 (dd, *J* = 7.4, 2.0 Hz, 1.6H), 6.55 (br, 1H), 4.65 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.49 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.10 – 2.97 (m, 2H), 2.51 – 2.34 (m, 2H), 1.58 – 1.48 (m, 2H), 1.41 (t, *J* = 7.1 Hz, 6H), 1.19 (t, *J* = 7.5 Hz, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.18, 163.07, 142.84, 141.38, 139.64, 139.43, 137.21, 130.92, 130.02, 129.91, 129.57, 129.37, 128.83, 128.59, 128.43, 128.27, 128.18, 128.16, 127.69, 127.18, 126.92, 126.81, 49.66, 46.64, 39.26, 30.61, 29.60, 20.97, 20.20. HRMS Calcd for $C_{31}H_{36}N_2O_2$ [M+Na^+]: 491.2674; Found: 411.2677.



3f

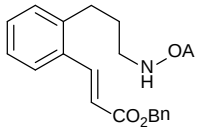
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 15.2 Hz, 1H), 7.60 – 7.46 (m, 1H), 7.29 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.25 – 7.18 (m, 2H), 7.08 (br, 1H), 6.79 (d, *J* = 15.2 Hz, 1H), 4.68 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.51 (dt, *J* = 13.6, 6.7 Hz, 1H), 3.30 (dd, *J* = 13.3, 6.9 Hz, 2H), 3.18 (s, 3H), 3.07 (s, 3H), 2.82 (dd, *J* = 13.7, 5.8 Hz, 2H), 1.87 (d, *J* = 7.5 Hz, 1H), 1.82 (d, *J* = 7.3 Hz, 1H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.83, 163.70, 163.36, 140.77, 140.02, 134.24, 130.15, 129.65, 126.75, 126.61, 119.44, 49.81, 46.57, 38.95, 37.56, 36.05, 30.75, 30.72, 20.99, 20.21. HRMS Calcd for $C_{22}H_{33}N_3O_3$ [M+Na^+]: 410.2420; Found: 410.2424.



3g

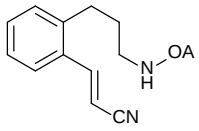
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 15.8 Hz, 1H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.33 – 7.29(m, 1H), 7.22 (dd, *J* = 11.2, 7.5 Hz, 2H), 7.02 (br, 1H), 6.36 (d, *J* = 15.8 Hz, 1H), 4.76 (dt, *J* = 13.3, 6.6 Hz, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.32 (dd, *J* = 13.5, 6.9 Hz, 2H), 2.94 – 2.71 (m, 2H), 1.84 (dt, *J* = 14.9, 7.5 Hz, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.35 (t, *J* = 7.1 Hz, 3H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.06, 163.42, 163.09, 141.84,

140.85, 133.12, 130.26, 130.13, 126.91, 126.87, 120.00, 60.68, 49.74, 46.67, 38.97, 31.05, 30.83, 30.66, 20.98, 20.18, 14.45. HRMS Calcd for $C_{22}H_{32}N_2O_4$ [M+Na^+]: 411.2274; Found: 411.2267.



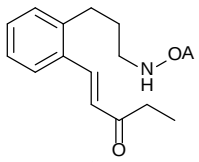
3h

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 15.8 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.43 – 7.29 (m, 6H), 7.24 – 7.20 (m, 2H), 6.99 (br, 1H), 6.41 (dd, *J* = 15.7, 11.3 Hz, 1H), 5.26 (s, 2H), 4.76 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.64 – 3.42 (m, 1H), 3.31 (dd, *J* = 13.5, 6.8 Hz, 2H), 2.84 – 2.80 (m, 2H), 1.84 (dt, *J* = 14.8, 7.4 Hz, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.88, 163.39, 163.02, 142.47, 140.95, 136.19, 132.99, 130.42, 130.18, 128.74, 128.41, 126.96, 126.90, 119.54, 66.53, 49.73, 46.71, 38.98, 31.06, 30.68, 21.00, 20.19. HRMS Calcd for $C_{27}H_{34}N_2O_4$ [M+Na^+]: 473.2416; Found: 473.2419.



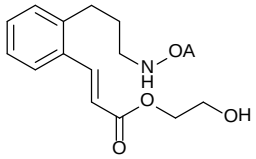
3i

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 16.5 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.38 (dd, *J* = 7.4, 6.6 Hz, 1H), 7.29 – 7.18 (m, 2H), 7.09 (br, 1H), 5.84 (d, *J* = 16.5 Hz, 1H), 4.80 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.55 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.35 (dd, *J* = 13.5, 6.8 Hz, 2H), 2.89 – 2.69 (m, 2H), 1.86 (dd, *J* = 15.0, 7.5 Hz, 2H), 1.45 (d, *J* = 6.8 Hz, 6H), 1.26 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.43, 162.95, 148.02, 140.56, 132.24, 131.29, 130.36, 127.17, 126.10, 118.32, 98.00, 49.77, 46.76, 38.91, 30.93, 30.53, 21.01, 20.19. HRMS Calcd for $C_{20}H_{27}N_3O_2$ [M+Na^+]: 324.2001; Found: 324.2007.



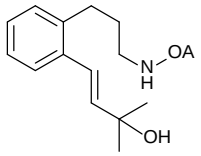
3j

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 15.9 Hz, 1H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.32 (m, 1H), 7.26 – 7.19 (m, 2H), 7.01 (br, 1H), 6.68 (d, *J* = 15.9 Hz, 1H), 4.84 – 4.67 (m, 1H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.32 (dd, *J* = 13.4, 6.9 Hz, 2H), 2.88 – 2.78 (m, 2H), 2.71 (q, *J* = 7.3 Hz, 2H), 1.84 (dt, *J* = 14.8, 7.5 Hz, 2H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H), 1.18 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.02, 163.45, 163.04, 141.22, 139.29, 133.24, 130.40, 130.20, 127.59, 126.96, 126.82, 49.77, 46.72, 39.00, 34.59, 30.98, 30.72, 21.00, 20.19, 8.31. HRMS Calcd for $C_{22}H_{32}N_2O_3$ [M+Na^+]: 395.2311; Found: 395.2314.



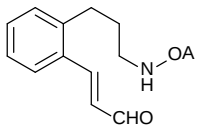
3k

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 15.8 Hz, 1H), 7.73 – 7.46 (m, 1H), 7.31 (m, 1H), 7.22 (m, 3H), 6.39 (d, *J* = 15.8 Hz, 1H), 4.64 (dt, *J* = 13.3, 6.7 Hz, 1H), 4.43 – 4.29 (m, 2H), 4.01 – 3.84 (m, 2H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.34 (dd, *J* = 13.4, 6.9 Hz, 2H), 2.97 (s, 1H), 2.85 – 2.74 (m, 2H), 1.83 (dt, *J* = 14.9, 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 167.21, 163.71, 163.41, 142.49, 141.09, 132.90, 130.45, 130.18, 126.96, 126.85, 119.40, 66.42, 61.22, 49.95, 46.63, 39.08, 31.09, 30.82, 20.94, 20.16. HRMS Calcd for $C_{22}H_{32}N_2O_5$ [M+Na^+]: 427.2209; Found: 427.2209.



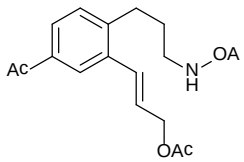
3l

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.35 (m, 1H), 7.26 – 7.09 (m, 4H), 6.93 (d, *J* = 15.8 Hz, 1H), 6.24 (d, *J* = 15.8 Hz, 1H), 4.70 (m, 1H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.29 (q, *J* = 6.5 Hz, 2H), 3.21 (br, 1H), 2.81 – 2.70 (m, 2H), 1.90 – 1.78 (m, 2H), 1.48 – 1.39 (m, 12H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.57, 163.37, 140.57, 138.91, 136.28, 129.52, 127.48, 126.53, 126.45, 123.56, 71.02, 49.92, 46.71, 38.69, 30.71, 30.14, 30.12, 20.93, 20.20. HRMS Calcd for $C_{22}H_{34}N_2O_3$ [M+Na^+]: 397.2467; Found: 397.2477.



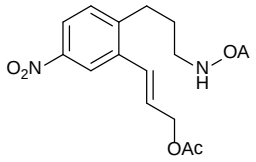
3m

^{1}H NMR (400 MHz, CDCl3) δ 9.76 (d, *J* = 7.7 Hz, 1H), 7.81 (d, *J* = 15.7 Hz, 1H), 7.61 (d, J = 7.8 Hz, 1H), 7.39 – 7.35 (m, 1H), 7.30 – 7.25 (m, 2H), 7.13 (br, 1H), 6.68 (dd, *J* = 15.7, 7.7 Hz, 1H), 4.75 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.61 – 3.46 (m, 1H), 3.35 (dd, *J* = 13.3, 6.8 Hz, 2H), 2.94 – 2.80 (m, 2H), 1.95 – 1.80 (m, 2H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.14, 163.50, 163.02, 149.69, 141.28, 132.50, 131.30, 130.34, 130.24, 127.21, 127.15, 49.80, 46.76, 38.95, 31.21, 30.52, 21.00, 20.18. HRMS Calcd for $C_{20}H_{28}N_2O_3$ [M+Na^+]: 344.2100; Found: 344.2109.



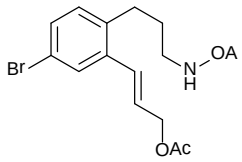
3n

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 1.6 Hz, 1H), 7.79 (m, 1H), 7.27 (dd, *J* = 16.7, 8.1 Hz, 1H), 7.08 (br, 1H), 6.92 (d, *J* = 15.7 Hz, 1H), 6.25 (dt, *J* = 15.7, 6.4 Hz, 1H), 4.87 – 4.57 (m, 3H), 3.53 (m, 1H), 3.38 – 3.26 (m, 2H), 2.83 – 2.73 (m, 2H), 2.59 (s, 3H), 2.12 (s, 2H), 1.88 – 1.77 (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 197.88, 171.05, 163.60, 163.12, 144.55, 135.80, 135.70, 131.64, 130.97, 129.91, 128.11, 126.93, 126.57, 65.18, 49.79, 46.69, 38.93, 30.76, 30.24, 29.68, 21.17, 21.00, 20.19. HRMS Calcd for $C_{24}H_{34}N_2O_5$ [M+Na^+]: 453.2365; Found: 453.2373.



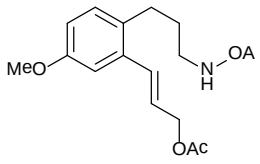
3o

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 2.4 Hz, 1H), 8.08 – 7.99 (m, 1H), 7.38 – 7.31(m, 2H), 7.11 (br, 1H), 6.91 (d, *J* = 15.7 Hz, 1H), 6.30 (dt, *J* = 15.7, 6.2 Hz, 1H), 4.81 – 4.73(m, 3H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.35 (dd, *J* = 13.2, 6.8 Hz, 2H), 2.81 (dd, *J* = 8.9, 6.9 Hz, 2H), 2.13 (s, 3H), 1.88 – 1.78 (m, 2H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.02, 163.68, 163.03, 146.97, 146.46, 136.76, 130.55, 129.67, 128.57, 122.76, 121.58, 100.12, 64.85, 49.82, 46.73, 38.82, 30.73, 30.21, 29.85, 21.16, 21.01, 20.19. HRMS Calcd for $C_{22}H_{31}N_3O_6$ [M+Na^+]: 456.2111; Found: 456.2121.



3p

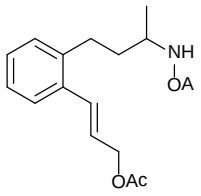
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 2.0 Hz, 1H), 7.35 (m, 2H), 7.06 (m, 2H), 6.85 (d, *J* = 15.7 Hz, 1H), 6.17 (dt, *J* = 15.6, 6.4 Hz, 1H), 4.81 – 4.73 (m, 3H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.41 – 3.24 (m, 2H), 2.79 – 2.65 (m, 2H), 2.13 (s, 3H), 1.80 (dt, *J* = 14.6, 7.1 Hz, 2H), 1.44 (d, *J* = 6.8 Hz, 6H), 1.25 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.87, 163.44, 163.02, 137.85, 137.10, 131.15, 130.91, 130.39, 129.23, 126.62, 120.28, 64.97, 49.65, 46.55, 38.75, 30.94, 30.28, 30.03, 21.01, 20.87, 20.06. HRMS Calcd for $C_{22}H_{31}BrN_2O_4$ [M+Na^+]: 489.1365; Found: 489.1373.



3q

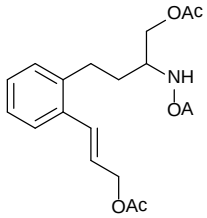
^{1}H NMR (400 MHz, CDCl3) δ 7.07 – 6.97(m, 3H), 6.87 (d, *J* = 15.7 Hz, 1H), 6.77 (dd, *J* = 8.4, 2.7 Hz, 1H), 6.15 (dt, *J* = 15.6, 6.5 Hz, 1H), 4.76 – 4.72 (m, 3H), 3.80 (s, 3H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.30 (dd, *J* = 13.3, 6.9 Hz, 2H), 2.69 – 2.65 (m, 2H), 2.11 (s, 3H), 1.78 (dt, *J* = 14.7, 7.3 Hz, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13C} NMR (101 MHz, CDCl3) δ

171.06, 163.52, 163.23, 158.29, 136.05, 131.95, 131.44, 130.72, 126.80, 125.37, 114.07, 111.56, 65.38, 61.49, 55.44, 49.76, 46.66, 38.98, 30.83, 29.85, 21.18, 21.01, 20.20. HRMS Calcd for $C_{23}H_{34}N_2O_5$ [M+Na^+]: 441.2365; Found: 441.2368.



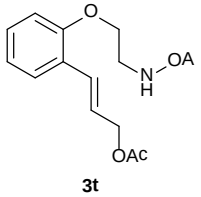
3r

^{1}H NMR (400 MHz, CDCl3) δ 7.44 – 7.41 (m, 1H), 7.21 – 7.13 (m, 3H), 6.96 – 6.92 (m, 2H), 6.17 – 6.10 (m, 1H), 4.86 – 4.80(m, 1H), 4.75 – 4.66 (m, 2H), 4.08 – 4.01 (m, 1H), 3.55 – 3.48(m, 1H), 2.81 – 2.64(m, 2H), 2.09 (s, 3H), 1.76 – 1.68 (m, 2H), 1.44 (dd, *J* = 6.8, 1.9 Hz, 6H), 1.27 – 1.21 (m, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.04, 163.46, 163.09, 139.50, 135.03, 132.26, 129.61, 128.29, 126.61, 126.43, 125.14, 65.56, 49.81, 46.60, 45.43, 38.35, 30.19, 21.21, 21.03, 20.99, 20.91, 20.24. HRMS Calcd for $C_{23}H_{34}N_2O_4$ [M+Na^+]: 425.2416; Found: 425.2420.



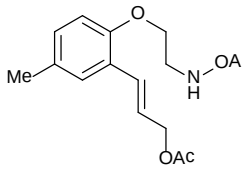
3s

^{1}H NMR (400 MHz, CDCl3) δ 7.43 – 7.41 (m, 1H), 7.21 – 7.13 (m, 4H), 6.95 (d, *J* = 15.6 Hz, 1H), 6.15 – 6.08(m, 1H), 4.90 – 4.85(m, 1H), 4.70 – 4.59 (m, 2H), 4.27 – 4.20(m, 1H), 4.15 – 4.12(m, 2H), 3.58 – 3.49(m, 1H), 2.88 – 2.65(m, 2H), 2.09 (s, 3H), 2.06 (s, 3H), 1.82 – 1.76 (m, 1H), 1.70 – 1.67(m, 1H), 1.44 (dd, *J* = 6.8, 2.9 Hz, 6H), 1.25 (dd, *J* = 9.4, 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.13, 170.91, 163.79, 163.33, 139.03, 135.08, 132.40, 129.66, 128.35, 126.81, 126.52, 125.32, 65.85, 65.69, 49.92, 48.29, 46.54, 33.09, 29.95, 21.22, 20.98, 20.95, 20.90, 20.26, 20.16. HRMS Calcd for $C_{25}H_{36}N_2O_6$ [M+Na^+]: 483.2471; Found: 483.2476.



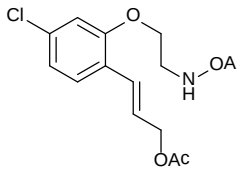
3t

^{1}H NMR (400 MHz, CDCl3) δ 7.45 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.35 (br, 1H), 7.24 – 7.20 (m, 1H), 7.00 – 6.93 (m, 1H), 6.85 (d, *J* = 8.2 Hz, 1H), 6.31 (dt, *J* = 16.0, 6.6 Hz, 1H), 4.78 – 4.71 (m, 1H), 4.13 (t, *J* = 5.3 Hz, 2H), 3.74 (dd, *J* = 11.1, 5.5 Hz, 2H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.10 (s, 3H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.09, 163.53, 162.70, 155.75, 129.31, 129.17, 127.31, 125.68, 124.17, 121.45, 112.28, 67.00, 65.76, 49.75, 46.73, 39.01, 21.18, 20.97, 20.17. HRMS Calcd for $C_{21}H_{30}N_2O_5$ [M+Na^+]: 413.2052; Found: 413.2052.



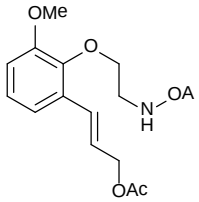
3u

^{1}H NMR (400 MHz, CDCl3) δ 7.32 (br, 1H), 7.36 – 7.25(m, 1H), 7.02 (dd, *J* = 8.3, 1.8 Hz, 1H), 6.95 (d, *J* = 16.0 Hz, 1H), 6.75 (d, *J* = 8.3 Hz, 1H), 6.29 (dt, *J* = 16.0, 6.6 Hz, 1H), 4.78 – 4.72 (m, 3H), 4.08 (t, *J* = 5.2 Hz, 2H), 3.72 (dd, *J* = 11.1, 5.5 Hz, 2H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.28 (s, 3H), 2.10 (s, 3H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.22 (d, *J* = 6.7 Hz, 6H).^{13}C NMR (101 MHz, CDCl3) δ 171.11, 163.51, 162.72, 153.75, 130.70, 129.76, 129.25, 127.82, 125.40, 123.92, 112.50, 67.25, 65.81, 49.75, 46.73, 39.06, 21.18, 20.98, 20.65, 20.17. HRMS Calcd for $C_{22}H_{32}N_2O_5$ [M+Na$^+$]: 427.2209; Found: 427.2209.



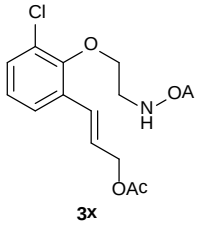
3v

^{1}H NMR (400 MHz, CDCl3) δ 7.42 (s, 1H), 7.35 (d, *J* = 8.3 Hz, 1H), 6.91 – 6.83 (m, 3H), 6.28 (dt, *J* = 16.0, 6.6 Hz, 1H), 4.78 – 4.72 (m, 3H), 4.09 (t, *J* = 5.3 Hz, 2H), 3.74 (dd, *J* = 11.1, 5.5 Hz, 2H), 3.52 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.10 (s, 3H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.05, 163.53, 162.67, 156.16, 134.46, 128.11, 128.04, 124.65, 124.26, 121.52, 112.82, 67.25, 65.56, 49.77, 46.74, 38.84, 21.13, 20.95, 20.14. HRMS Calcd for $C_{21}H_{29}ClN_2O_5$ [M+Na$^+$]: 447.1663; Found: 447.1662.



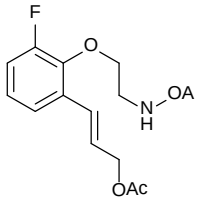
3w

^{1}H NMR (400 MHz, CDCl3) δ 7.79 (s, 1H), 7.10 – 6.95 (m, 3H), 6.85 (dd, *J* = 7.9, 1.6 Hz, 1H), 6.30 (dt, *J* = 16.0, 6.5 Hz, 1H), 4.76 (dd, *J* = 6.5, 1.2 Hz, 2H), 4.74 – 4.65 (m, 1H), 4.08 – 4.03 (m, 2H), 3.90 (s, 3H), 3.62 (dd, *J* = 10.1, 5.2 Hz, 2H), 3.56 – 3.49 (m, 1H), 2.10 (s, 3H), 1.45 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 170.99, 163.76, 163.25, 152.71, 145.31, 130.89, 128.49, 125.33, 124.65, 118.58, 111.76, 72.04, 65.45, 55.92, 49.76, 46.55, 39.90, 21.15, 21.02, 20.19. HRMS Calcd for $C_{22}H_{32}N_2O_6$ [M+Na$^+$]: 443.2158; Found: 443.2158.



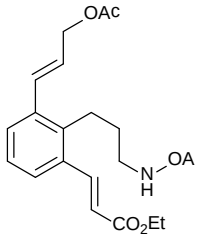
3x

^{1}H NMR (400 MHz, CDCl3) δ 7.49 (s, 1H), 7.37 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.32 – 7.28 (m, 1H), 7.06 – 7.02(m, 1H), 6.94 (d, *J* = 16.0 Hz, 1H), 6.30 (dt, *J* = 16.0, 6.4 Hz, 1H), 4.77 (dd, *J* = 6.4, 1.2 Hz, 2H), 4.71 (dt, *J* = 13.4, 6.7 Hz, 1H), 4.04 (dd, *J* = 9.3, 4.2 Hz, 2H), 3.71 (dd, *J* = 11.0, 5.5 Hz, 2H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.10 (s, 3H), 1.44 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 170.98, 163.66, 162.93, 152.04, 132.12, 129.97, 128.34, 128.02, 126.53, 125.57, 125.31, 72.04, 65.18, 49.77, 46.65, 39.53, 32.38, 32.31, 21.00, 20.16, 18.76. HRMS Calcd for $C_{21}H_{29}ClN_2O_5$ [M+Na$^+$]: 447.1663; Found: 447.1662.



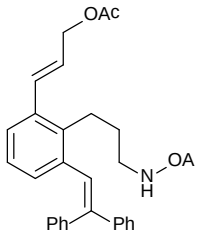
3y

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.28 – 7.20 (m, 1H), 7.00 (ddd, *J* = 24.4, 12.2, 9.9 Hz, 3H), 6.32 (dt, *J* = 16.0, 6.4 Hz, 1H), 4.78 (dd, *J* = 6.4, 1.2 Hz, 2H), 4.76 – 4.67 (m, 1H), 4.15 (t, *J* = 5.1 Hz, 2H), 3.68 (dd, *J* = 10.8, 5.5 Hz, 2H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.11 (s, 3H), 1.45 (d, *J* = 6.8 Hz, 7H), 1.25 (d, *J* = 6.7 Hz, 7H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.99, 163.63, 162.87, 157.04, 154.59, 143.81, 143.69, 131.76, 131.74, 127.73, 127.70, 126.21, 124.27, 124.19, 122.08, 122.04, 116.31, 116.12, 72.48, 72.43, 65.29, 49.76, 46.65, 39.55, 21.13, 20.96, 20.16. HRMS Calcd for $C_{21}H_{29}N_2O_5$ [M+Na$^+$]: 431.1958; Found: 431.1960.



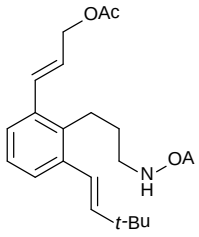
4a

^{1}H NMR (400 MHz, CDCl3) δ 8.00 (d, *J* = 15.7 Hz, 1H), 7.58 – 7.34 (m, 2H), 7.22 – 7.11 (m, 2H), 6.98 (dd, *J* = 15.6, 10.5 Hz, 1H), 6.39 – 6.28 (m, 1H), 6.14 – 6.06 (m, 1H), 4.77 (dt, *J* = 6.6, 3.4 Hz, 2H), 4.69 – 4.62 (m, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.35 (dd, *J* = 13.2, 6.7 Hz, 2H), 2.89 – 2.77 (m, 2H), 2.10 (s, 3H), 1.82 – 1.66 (m, 2H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.35 (t, *J* = 7.1 Hz, 3H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.32, 171.11, 166.96, 163.86, 163.36, 142.38, 138.65, 136.68, 133.96, 132.20, 128.71, 126.93, 126.88, 120.99, 65.39, 60.76, 49.83, 46.58, 38.97, 30.51, 29.46, 21.20, 20.99, 20.19, 14.47. HRMS Calcd for $C_{27}H_{38}N_2O_6$ [M+Na$^+$]: 509.2628; Found: 509.2630.



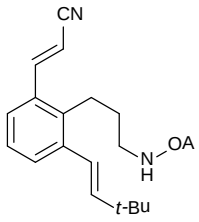
4b

^{1}H NMR (400 MHz, CDCl3) δ 7.39 – 7.32 (m, 5H), 7.26 – 7.19 (m, 4H), 7.13 – 6.96 (m, 4H), 6.86 (t, *J* = 7.6 Hz, 1H), 6.79 (d, *J* = 7.0 Hz, 1H), 6.11 (dt, *J* = 15.5, 6.5 Hz, 1H), 4.79 (dd, *J* = 6.5, 0.9 Hz, 2H), 4.69 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.51 (dt, *J* = 10.4, 6.8 Hz, 1H), 3.37 (q, *J* = 6.8 Hz, 2H), 2.91 – 2.82 (m, 2H), 2.12 (s, 3H), 1.85 – 1.77 (m, 2H)1.43 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 171.16, 163.74, 163.36, 144.25, 143.45, 140.02, 138.20, 137.35, 135.61, 132.87, 130.88, 130.65, 128.38, 128.32, 128.18, 127.79, 127.29, 126.86, 125.87, 125.60, 67.99, 66.92, 65.63, 49.77, 46.56, 42.68, 39.28, 32.33, 31.07, 29.89, 27.13, 21.22, 20.99, 20.18, 18.78, 13.70. HRMS Calcd for $C_{36}H_{42}N_2O_4$ [M+Na$^+$]: 589.3042; Found: 589.3042.



4c

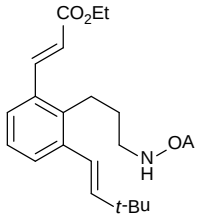
^{1}H NMR (400 MHz, CDCl3) δ 7.30 (t, *J* = 6.9 Hz, 2H), 7.12 (dd, *J* = 13.4, 5.7 Hz, 2H), 6.97 (d, *J* = 15.6 Hz, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 6.14 – 6.00 (m, 2H), 4.86 – 4.69 (m, 3H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.33 (dd, *J* = 13.1, 6.7 Hz, 2H), 2.85 – 2.75 (m, 2H), 2.10 (s, 3H), 1.72 (dt, *J* = 12.7, 8.7 Hz, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H), 1.12 (s, 9H). ^{13}C NMR (101 MHz, CDCl3) δ 171.11, 163.59, 163.18, 145.08, 137.97, 136.42, 135.76, 132.91, 128.59, 128.49, 128.35, 126.70, 126.52, 125.82, 125.62, 122.55, 65.54, 49.73, 46.62, 39.19, 33.78, 29.85, 29.75, 26.39, 21.19, 21.00, 20.18. HRMS Calcd for $C_{28}H_{42}N_2O_6$ [M+Na$^+$]: 493.3042; Found: 493.3041.



4d

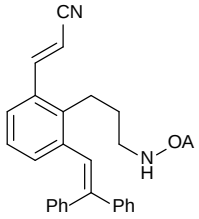
^{1}H NMR (400 MHz, CDCl3) δ 7.77 (d, *J* = 16.4 Hz, 1H), 7.46 (d, *J* = 7.3 Hz, 1H), 7.35 (d, *J* = 7.7 Hz, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 7.07 (br, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 6.09 (d, *J* = 15.8 Hz, 1H), 5.81 (d, *J* = 16.4 Hz, 1H), 4.87 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.36 (dd, *J* = 13.4, 6.9 Hz, 2H), 2.82 (dd, *J* = 9.2, 6.9 Hz, 2H), 1.75 (dt, *J* = 15.1, 7.4 Hz, 2H), 1.45 (d, *J* = 6.8 Hz, 6H), 1.26 (d, *J* = 6.7 Hz, 6H), 1.15 (s, 9H). ^{13}C NMR (101 MHz, CDCl3) δ 163.28, 162.67, 148.90, 146.24, 138.93, 137.70, 132.81, 129.67, 126.98, 125.00, 122.02, 118.28, 98.56,

49.65, 46.83, 39.25, 33.89, 30.38, 29.68, 26.59, 21.04, 20.20. HRMS Calcd for $C_{26}H_{37}N_3O_2$ [M+Na$^+$]: 423.2886; Found: 423.2891.



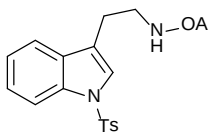
4e

^{1}H NMR (400 MHz, CDCl3) δ 8.02 (d, *J* = 15.7 Hz, 1H), 7.41 (t, *J* = 6.6 Hz, 2H), 7.18 (t, *J* = 7.7 Hz, 1H), 7.07 (br, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 6.33 (d, *J* = 15.7 Hz, 1H), 6.06 (d, *J* = 15.8 Hz, 1H), 4.77 (dt, *J* = 13.3, 6.7 Hz, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 3.51 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.33 (dd, *J* = 13.3, 6.8 Hz, 2H), 3.01 – 2.73 (m, 2H), 1.79 – 1.72 (m, 2H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.34 (t, *J* = 7.1 Hz, 3H), 1.22 (d, *J* = 6.7 Hz, 6H), 1.13 (s, 9H). ^{13}C NMR (101 MHz, CDCl3) δ 167.06, 163.34, 162.93, 145.64, 142.73, 138.53, 138.04, 133.60, 128.66, 126.70, 125.71, 122.29, 120.50, 60.67, 49.67, 46.66, 39.20, 33.82, 30.24, 29.68, 26.45, 20.96, 20.15, 14.44. HRMS Calcd for $C_{28}H_{42}N_2O_4$ [M+Na$^+$]: 493.3042; Found: 493.3036.



4f

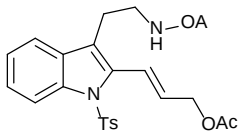
^{1}H NMR (400 MHz, CDCl3) δ 7.76 (d, *J* = 16.4 Hz, 1H), 7.36 (s, 5H), 7.28 – 7.19 (m, 4H), 7.06 – 6.93(m, 6H), 5.80 (d, *J* = 16.4 Hz, 1H), 4.80 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.37 (dd, *J* = 13.7, 7.0 Hz, 2H), 2.97 – 2.80 (m, 2H), 1.90 – 1.74 (m, 2H), 1.43 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 163.37, 162.78, 148.63, 145.30, 143.02, 139.66, 139.39, 138.25, 133.60, 132.77, 130.79, 128.61, 128.47, 128.31, 128.08, 127.56, 126.34, 125.71, 125.08, 118.27, 98.39, 67.99, 67.93, 66.92, 60.54, 49.70, 46.78, 42.69, 39.38, 32.41, 32.34, 30.39, 29.84, 27.25, 21.02, 20.18, 18.79, 14.34, 13.71. HRMS Calcd for $C_{34}H_{37}N_3O_2$ [M+Na$^+$]: 542.2782; Found: 542.2789.



5

^{1}H NMR (400 MHz, CDCl3) δ 7.99 (d, *J* = 8.3 Hz, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.44 (s, 1H), 7.35 – 7.32 (m, 1H), 7.25 – 7.23(m, 3H), 7.06 (br, 1H), 4.75 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.61 (dd, *J* = 13.5, 7.0 Hz, 2H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.95 (t, *J* = 7.1 Hz, 2H), 2.35 (s, 3H), 1.45 (d, *J* = 6.8 Hz, 6H), 1.24 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3)

δ 163.37, 162.87, 144.96, 135.41, 135.33, 130.69, 130.04, 127.01, 124.99, 123.63, 123.31, 119.50, 119.45, 113.91, 49.78, 46.74, 38.71, 25.08, 21.71, 20.99, 20.20. HRMS Calcd for $C_{25}H_{41}N_3O_4$ [M+Na^+]: 492.1933; Found: 492.1942.



6

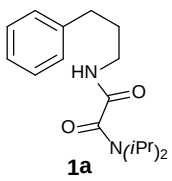
^{1}H NMR (400 MHz, CDCl3) δ 8.17 (d, *J* = 7.9 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.49 – 7.48 (m, 1H), 7.31 – 7.20 (m, 5H), 7.07 (d, *J* = 6.3 Hz, 1H), 7.00 (br, 1H), 5.21 – 5.16 (m, 1H), 4.78 (dd, *J* = 13.4, 6.7 Hz, 1H), 3.93 (dd, *J* = 7.0, 1.5 Hz, 2H), 3.54 (dd, *J* = 13.6, 6.8 Hz, 1H), 3.45 (dd, *J* = 14.3, 6.7 Hz, 2H), 2.97 – 2.93 (m, 1H), 2.35 (s, 3H), 2.21 (s, 3H), 1.44 (d, *J* = 6.8 Hz, 6H), 1.23 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 167.95, 163.28, 162.65, 144.84, 136.69, 136.02, 135.99, 134.53, 130.11, 129.97, 126.85, 126.50, 124.71, 123.78, 118.64, 118.27, 115.29, 111.15, 49.62, 46.75, 38.97, 29.84, 29.46, 24.27, 22.20, 21.70, 21.00, 20.88, 20.16. HRMS Calcd for $C_{30}H_{46}N_3O_6$ [M+Na^+]: 567.2403; Found: 567.2413.

7. References

[1] Mei, T.–S.; Wang, X.; Yu, J.–Q. *J. Am. Chem. Soc.* **2009**, *131*, 10806.

[2] He, G.; Lu, C.; Zhao, Y.; Nack, W. A.; Chen, G. *Org. Lett.* **2012**, *14*, 2944.

[3] He, G.; Zhao, Y.; Zhang, S.; Lu, C.; Chen, G. *J. Am. Chem. Soc.* **2012**, *134*, 3.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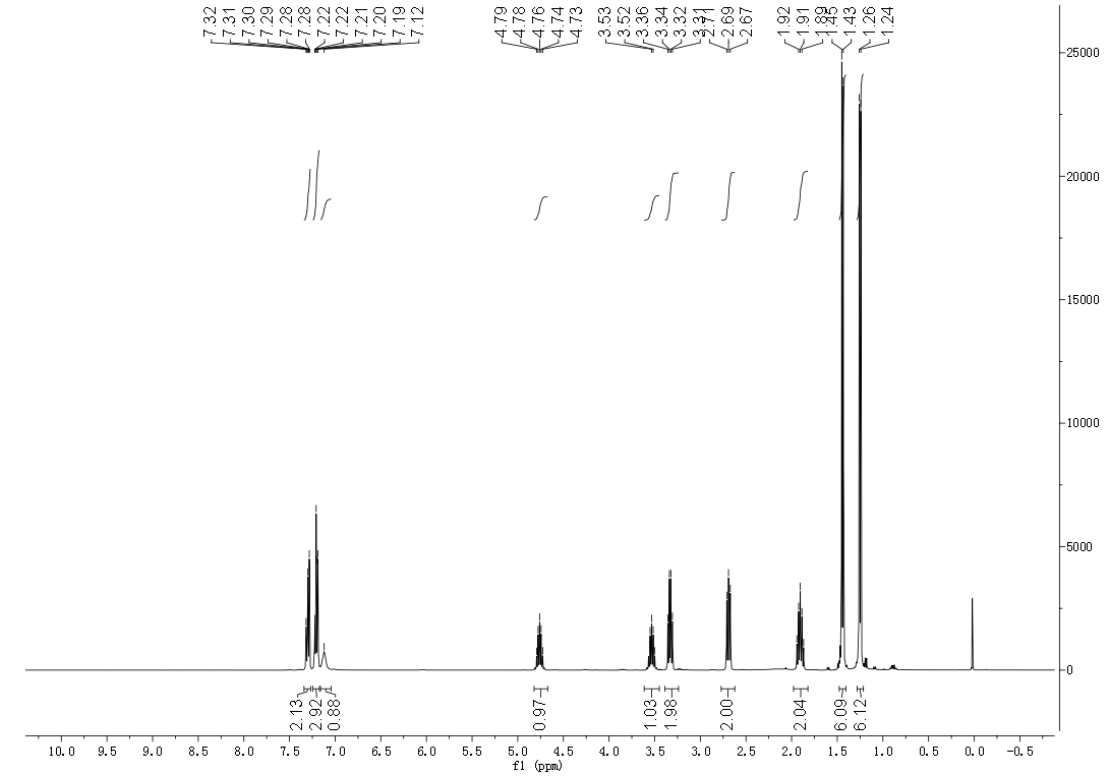
O
O
N(iPr)2

1a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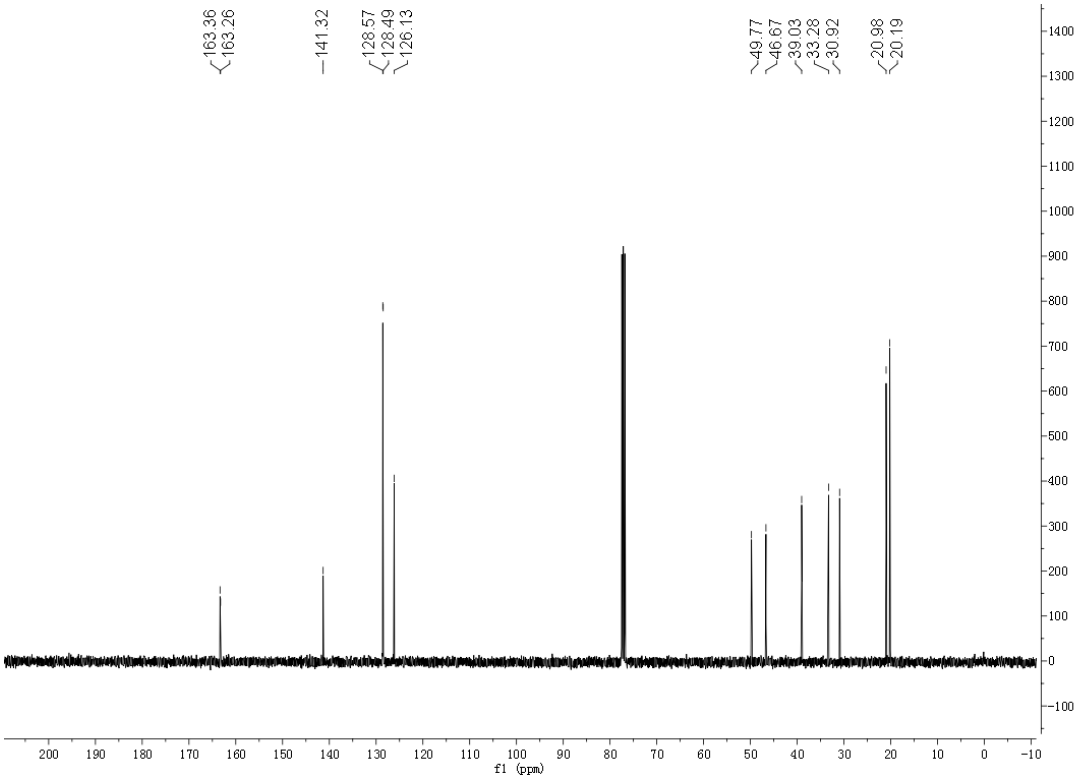
7.31
7.30
7.29
7.28
7.28
7.22
7.22
7.21
7.20
7.19
7.12
4.79
4.78
4.76
4.74
4.73
3.53
3.52
3.36
3.34
3.32
3.31
2.71
2.69
2.67
1.92
1.91
1.89
1.45
1.43
1.26
1.24
2.13
2.92
0.88
0.97
1.03
1.98
2.00
2.04
6.09
6.12
f1 (ppm)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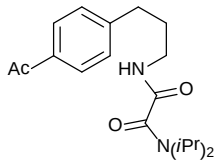
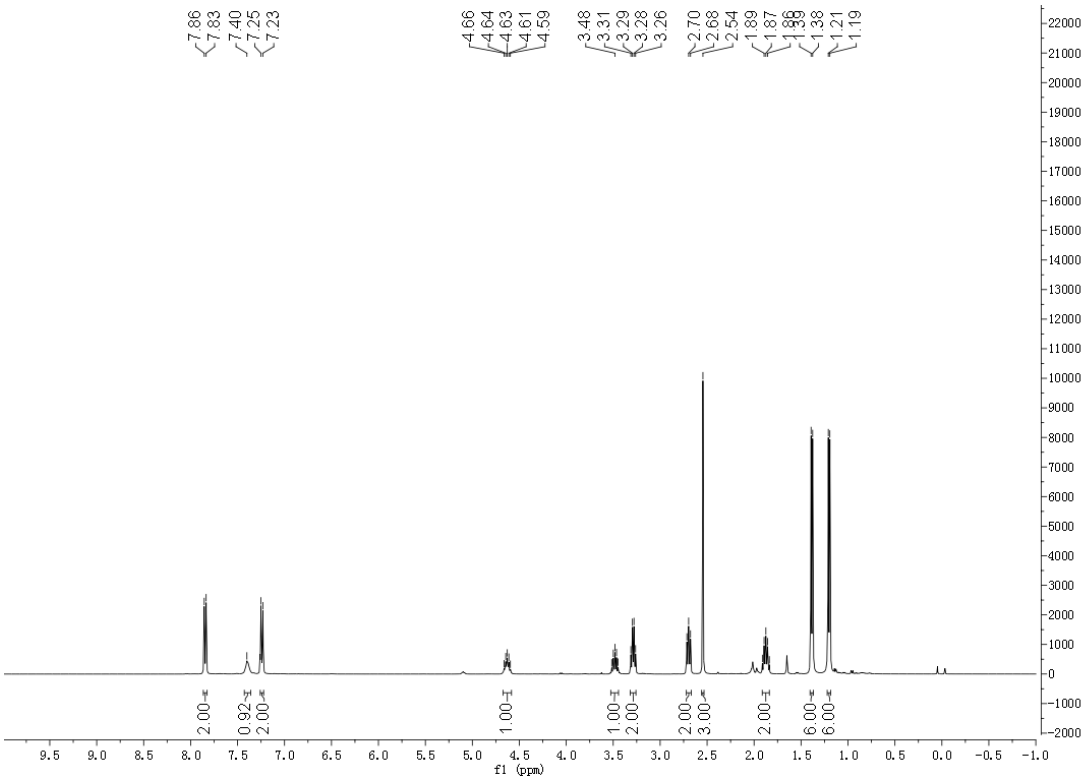
163.26
141.32
128.57
128.49
126.13
49.77
46.67
39.03
33.28
30.92
20.98
20.19
f1 (ppm)


Ac
HN
O
O
N(iPr)2

1n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4.63
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4.59
3.48
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3.29
3.28
3.26
2.70
2.68
2.54
1.89
1.87
1.38
1.21
1.19
2.00
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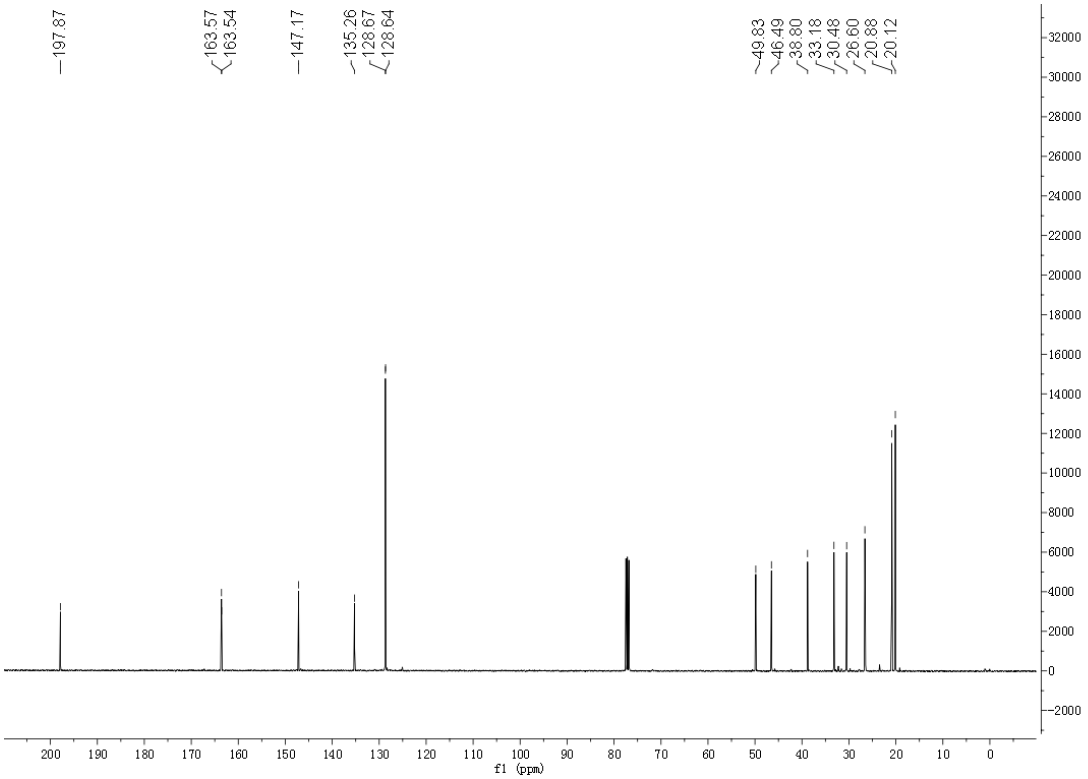
1.00
1.00
2.00
2.00
3.00
2.00
6.00
6.00
f1 (ppm)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163.57
163.54
147.17
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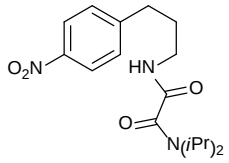
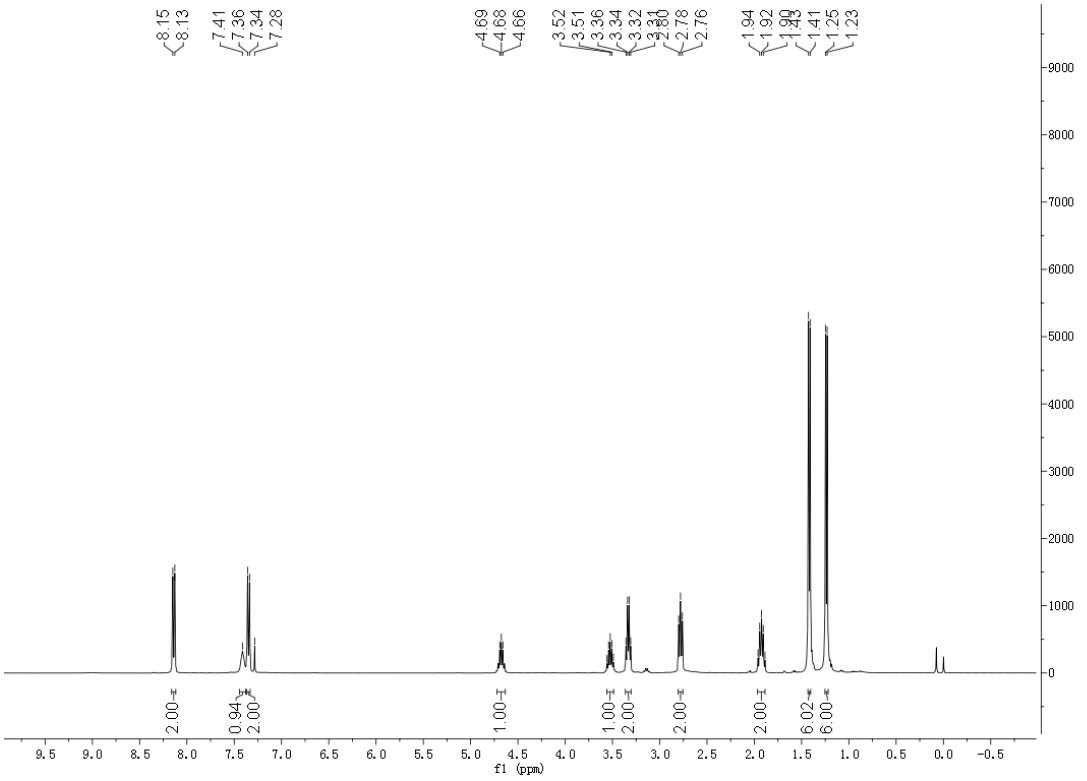
38.80
33.18
30.48
26.60
20.88
20.12
f1 (ppm)


O2N
HN
O
O
N(iPr)2

1o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7.28
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4.68
4.66
3.52
3.51
3.36
3.34
3.32
2.78
2.76
1.94
1.92
1.41
1.25
1.23
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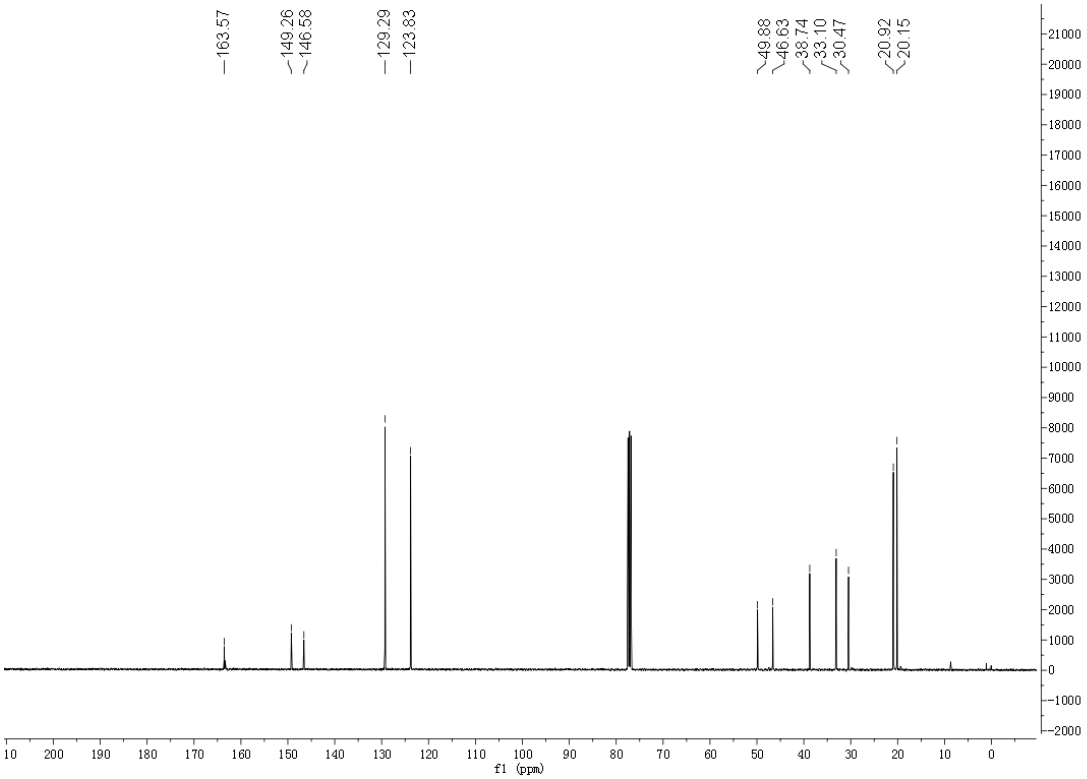
1.00
1.00
2.00
2.00
2.00
6.02
6.00
f1 (ppm)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146.58
129.29
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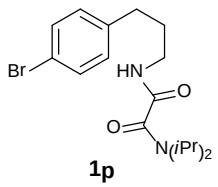
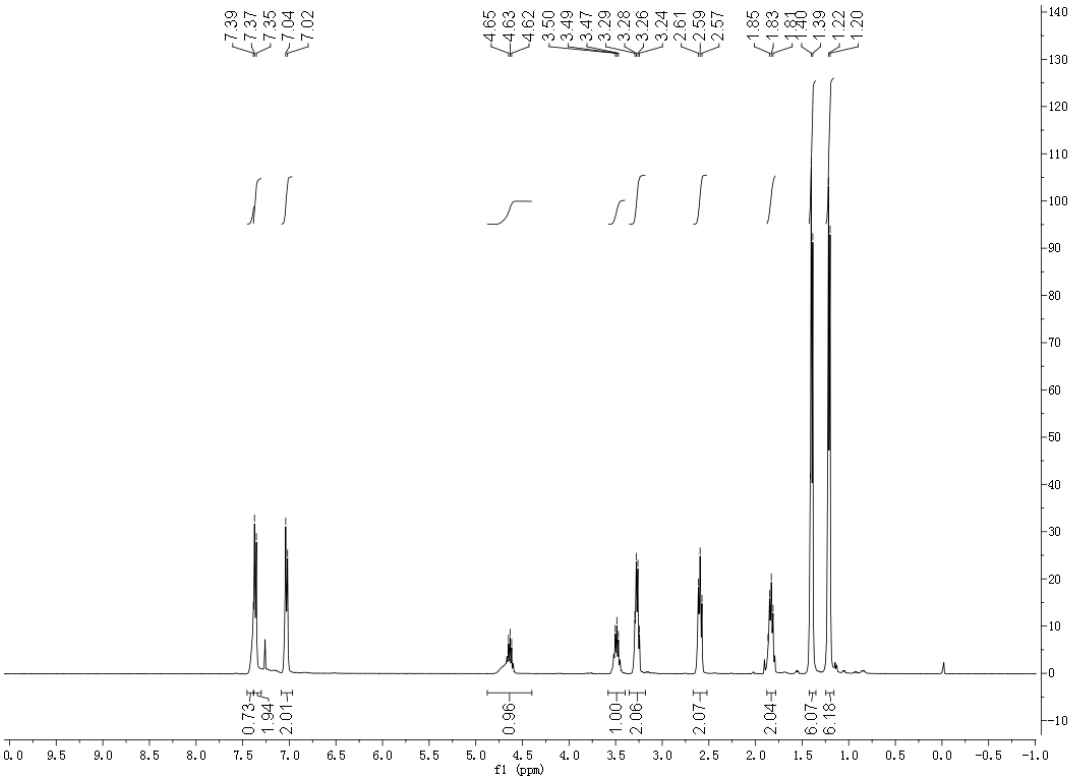
46.63
38.74
33.10
30.47
20.92
20.15
f1 (ppm)


Br
HN
O
O
N(iPr)2

1p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4.62
3.50
3.49
3.47
3.29
3.28
3.26
3.24
2.61
2.59
2.57
1.85
1.83
1.81
1.40
1.39
1.22
1.20
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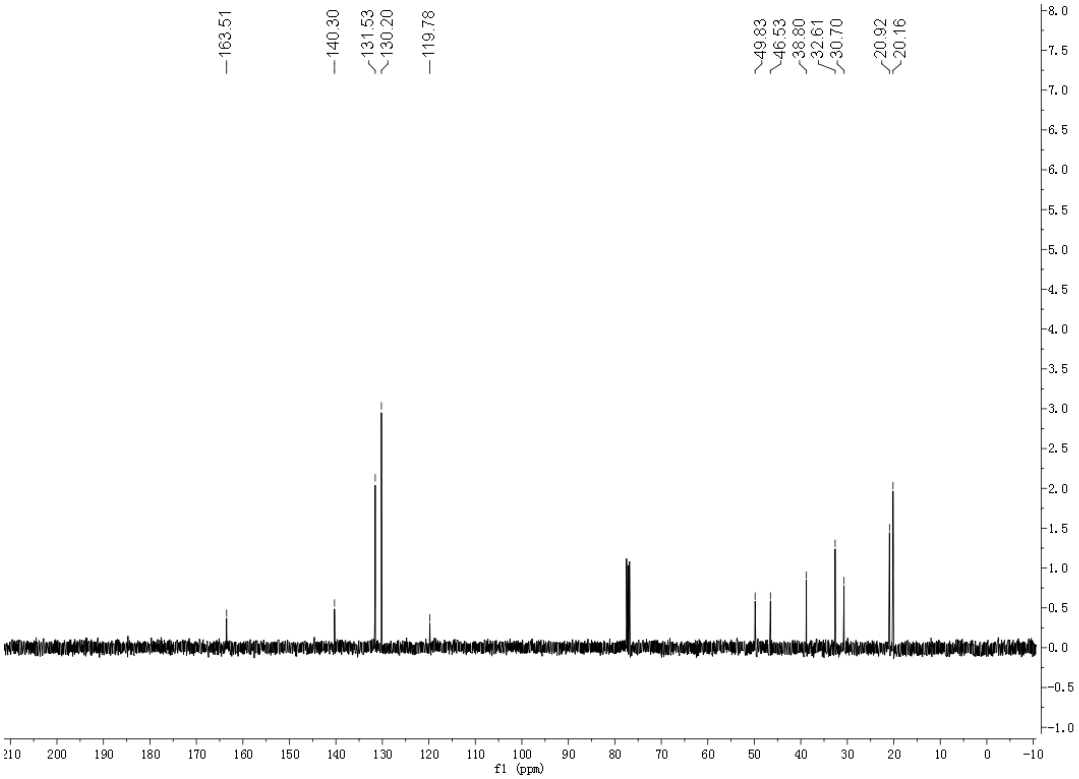
2.01
0.96
1.00
2.06
2.07
2.04
6.07
6.18
f1 (ppm)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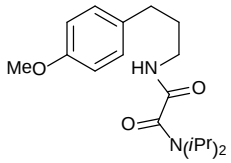
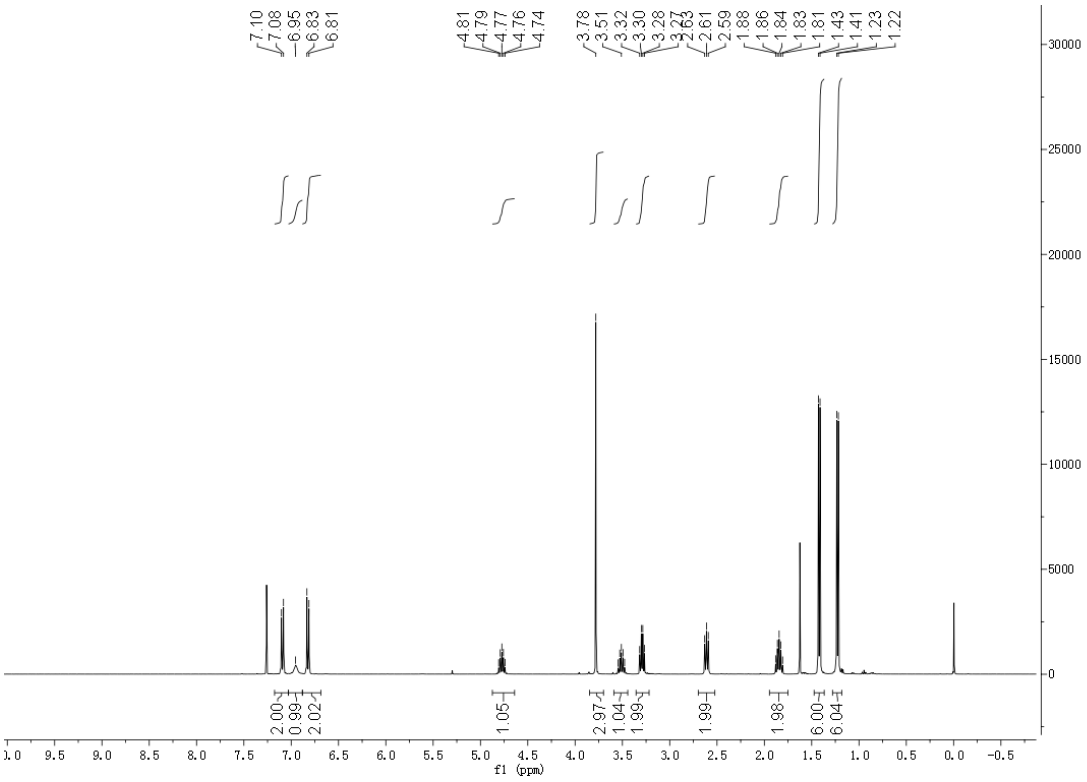
20.16
f1 (ppm)


MeO
HN
O
O
N(iPr)2

1q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6.81
4.81
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4.77
4.76
4.74
3.78
3.51
3.32
3.30
3.28
2.61
2.59
1.88
1.86
1.84
1.83
1.81
1.43
1.41
1.23
1.22
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1.05
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1.04
1.99
1.99
1.98
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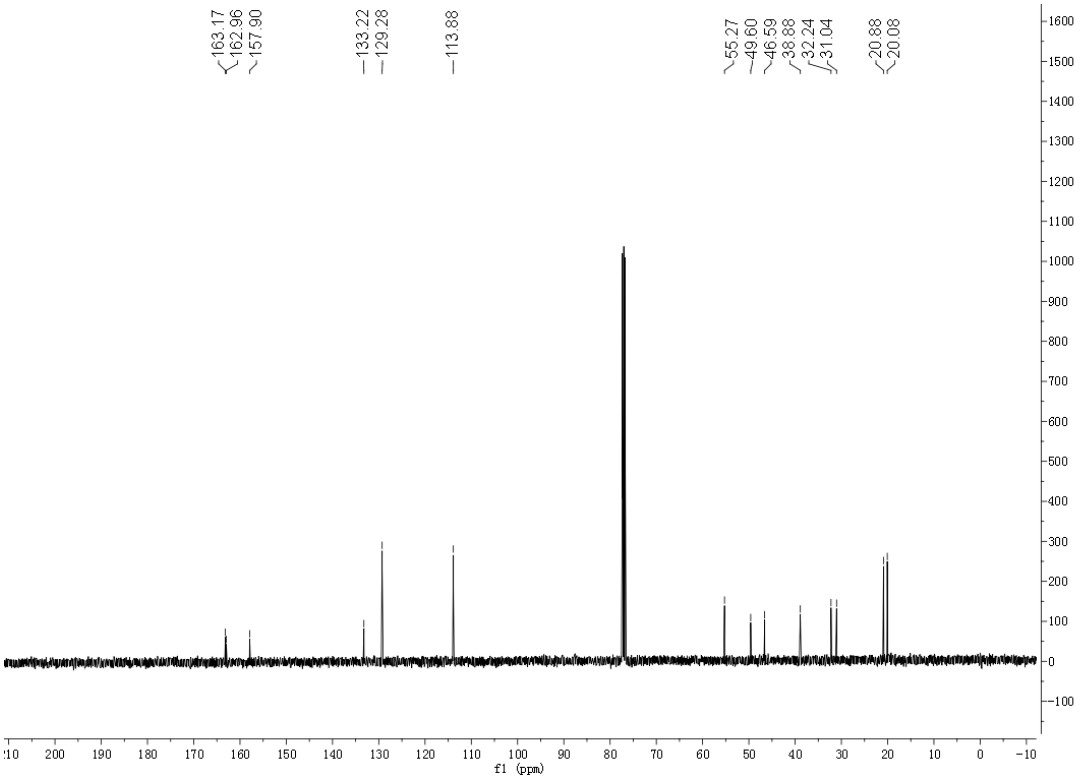
6.04
f1 (ppm)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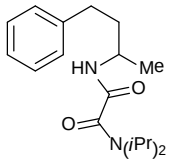
20.08
f1 (ppm)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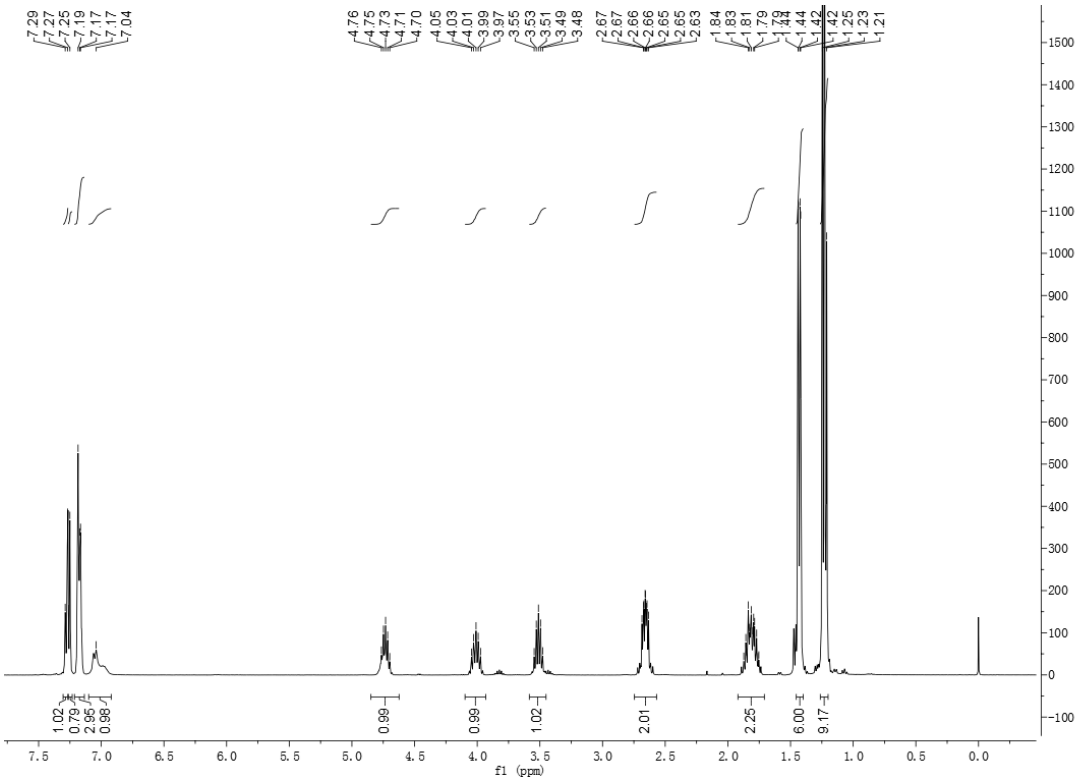
Me
O
O
N(iPr)2

1r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4.03
4.01
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3.53
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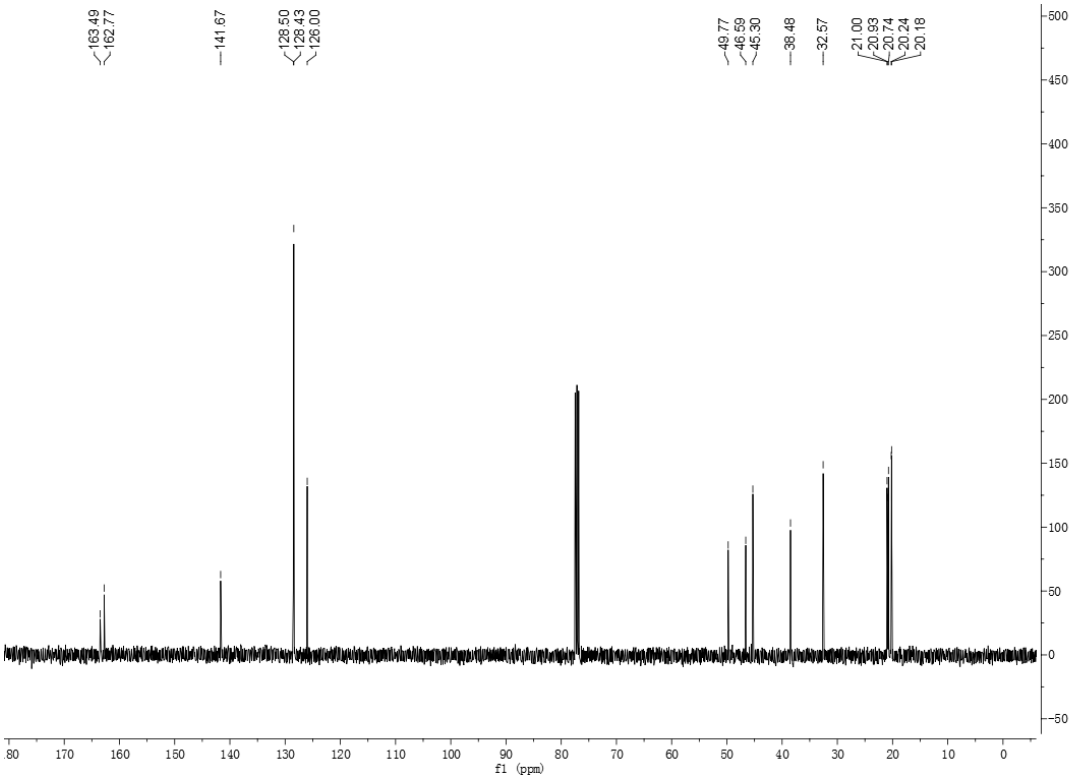
f1 (ppm)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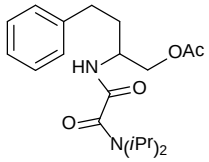
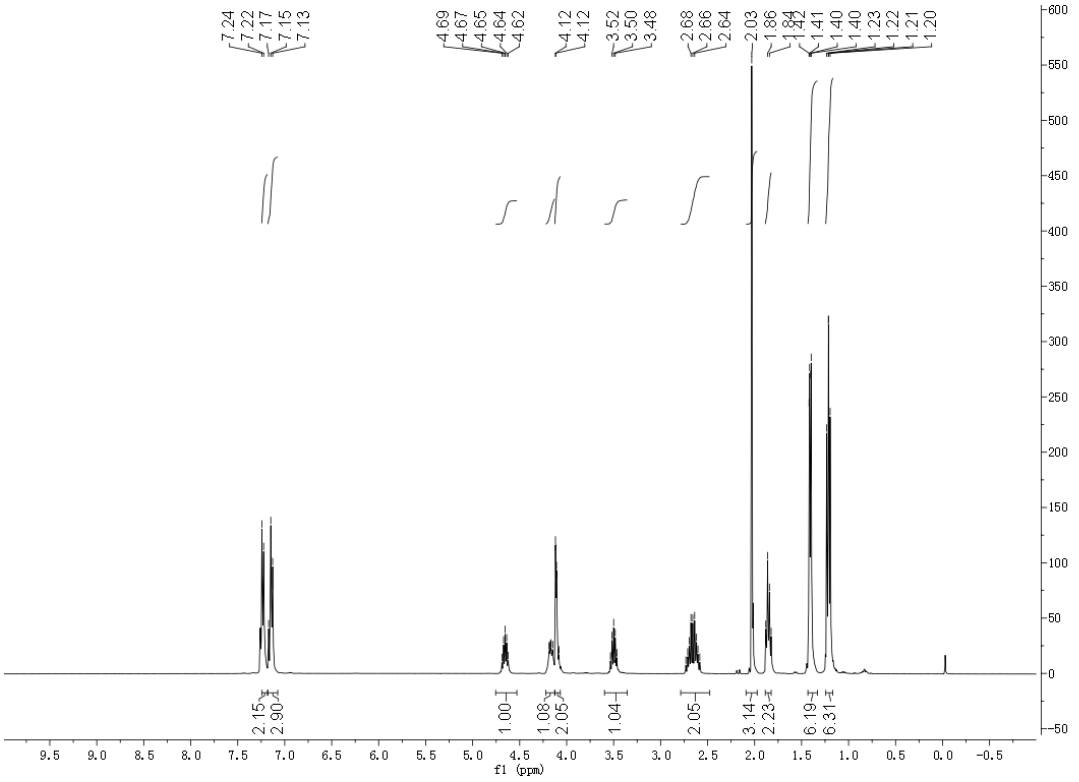
f1 (ppm)


OAc
HN
O
O
N(iPr)2

1s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2.64
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1.41
1.40
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1.22
1.21
1.20
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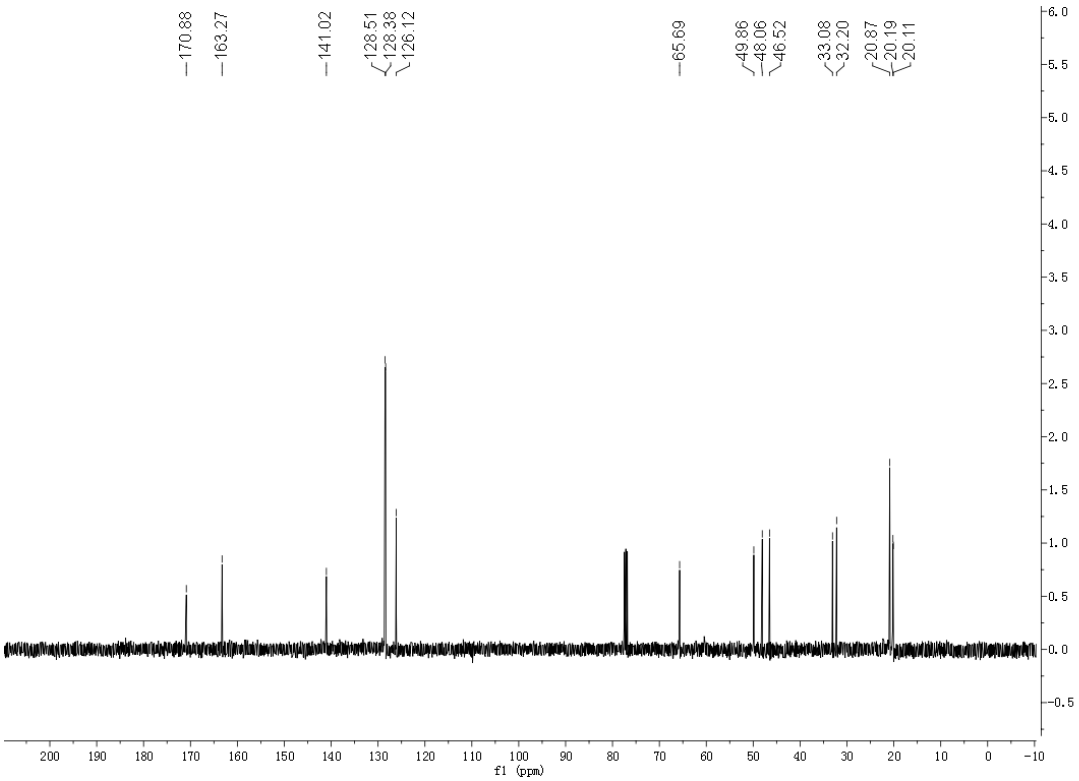
f1 (ppm)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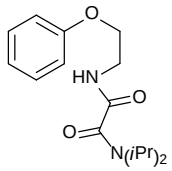
f1 (ppm)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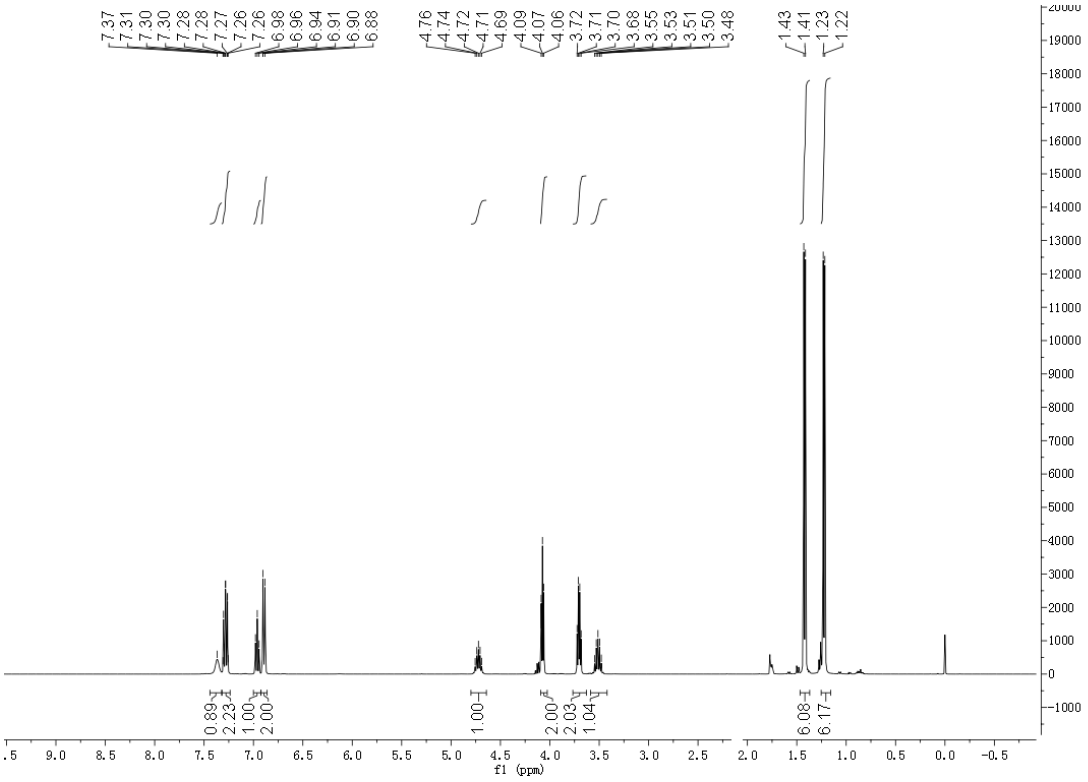
O
O
N(iPr)2

1t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3.70
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3.55
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3.51
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3.48
1.43
1.41
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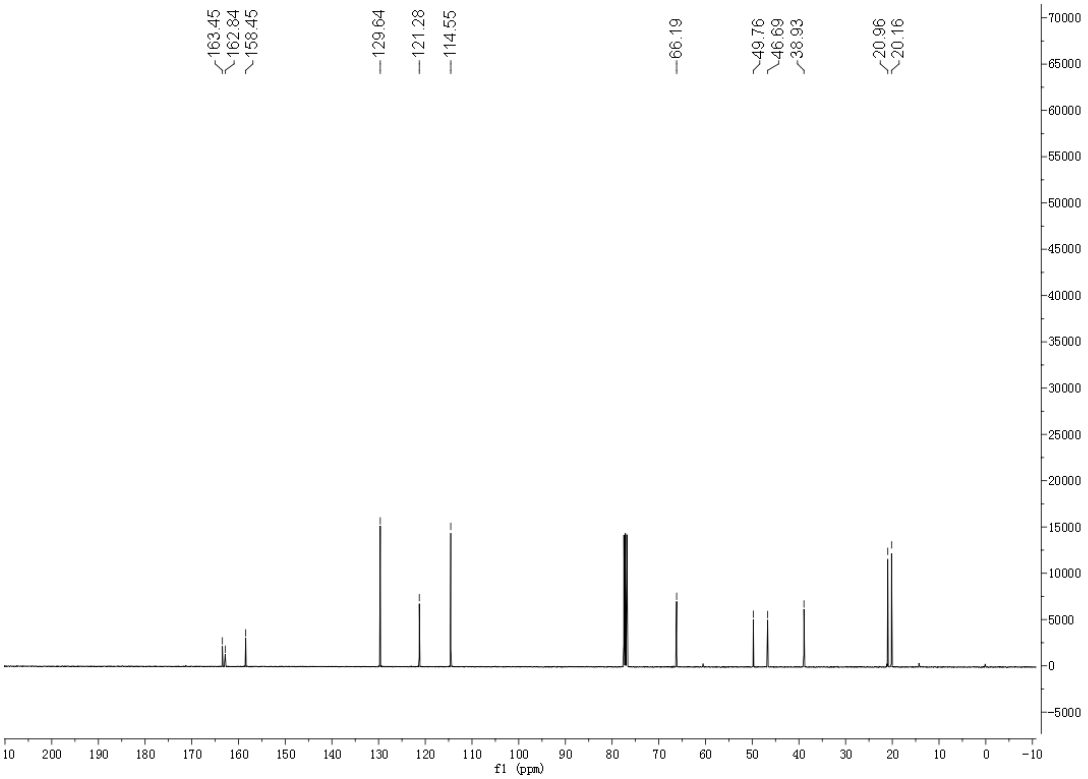
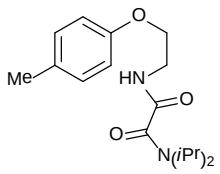
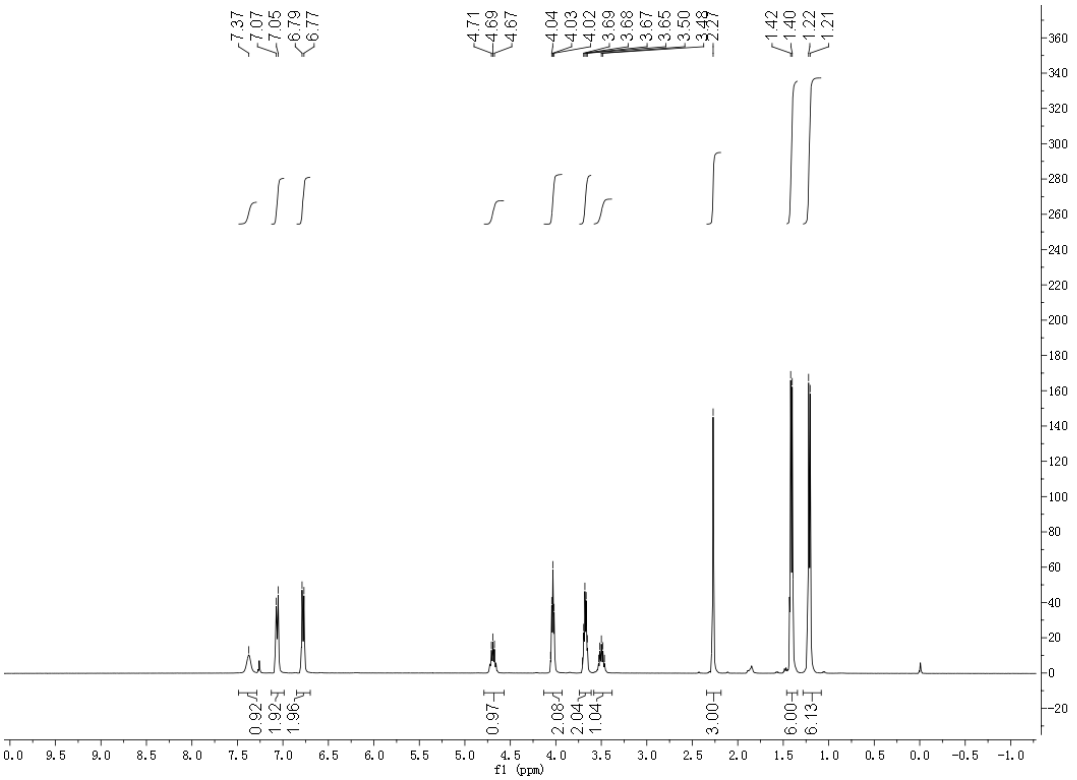
2.00
1.00
2.00
2.03
1.04
6.08
6.17
f1 (ppm)


163.45
162.84
158.45
129.64
121.28
114.55
66.19
49.76
46.69
38.93
20.96
20.16
f1 (ppm)


Me
HN
O
O
O
N(iPr)2

1u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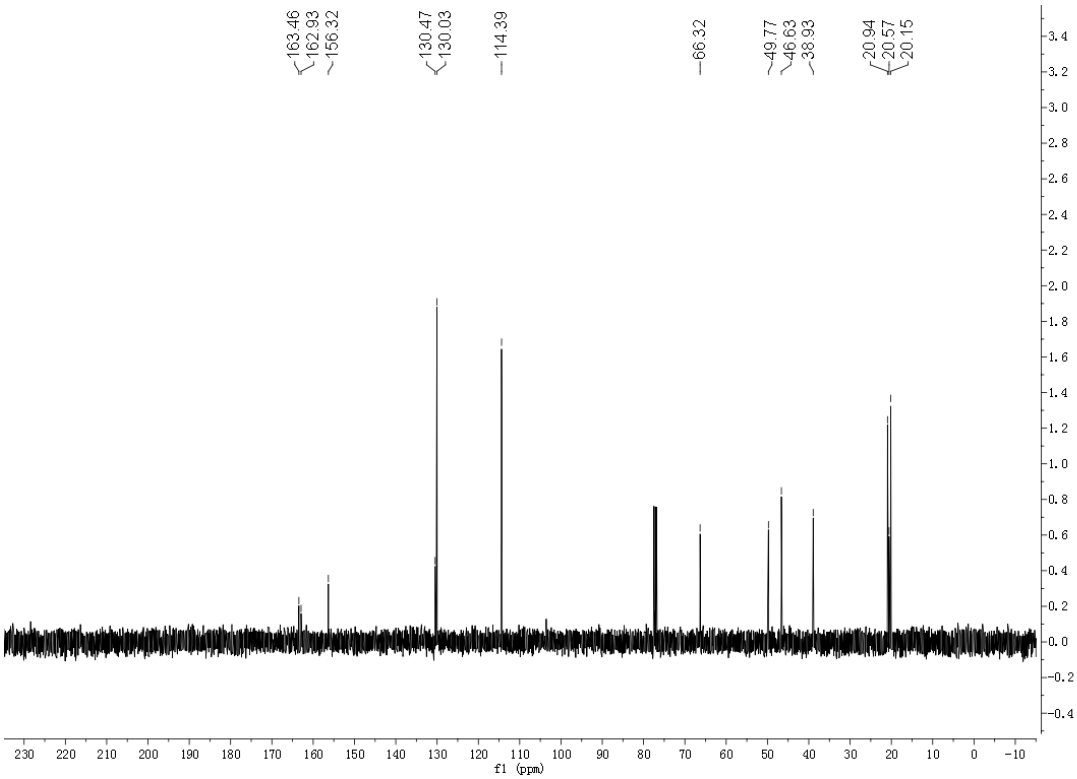
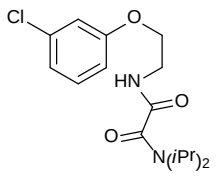
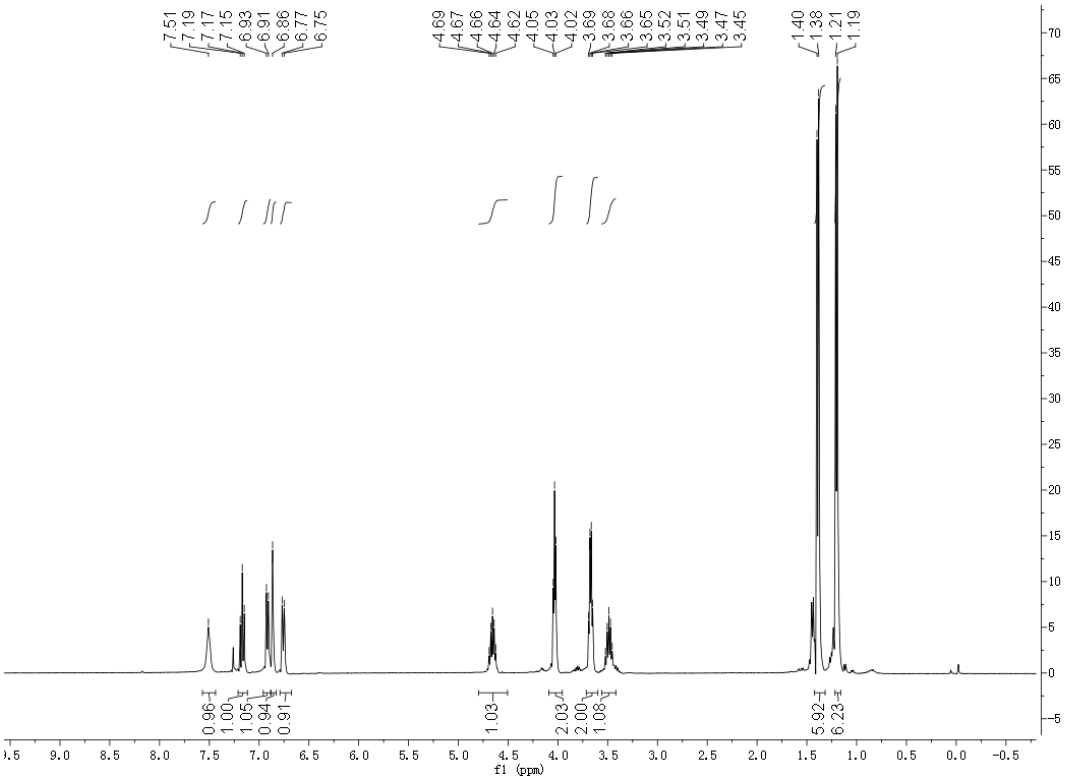
7.07
7.05
6.79
6.77
4.71
4.69
4.67
4.04
4.03
4.02
3.69
3.68
3.67
3.65
3.50
3.48
3.47
2.27
1.42
1.40
1.22
1.21
0.92
1.92
1.96
0.97
2.08
2.04
1.04
3.00
6.00
6.13
f1 (ppm)


163.46
162.93
156.32
130.47
130.03
114.39
66.32
49.77
46.63
38.93
20.94
20.57
20.15
f1 (ppm)


Cl
O
HN
O
O
N(iPr)2

1v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7.19
7.17
7.15
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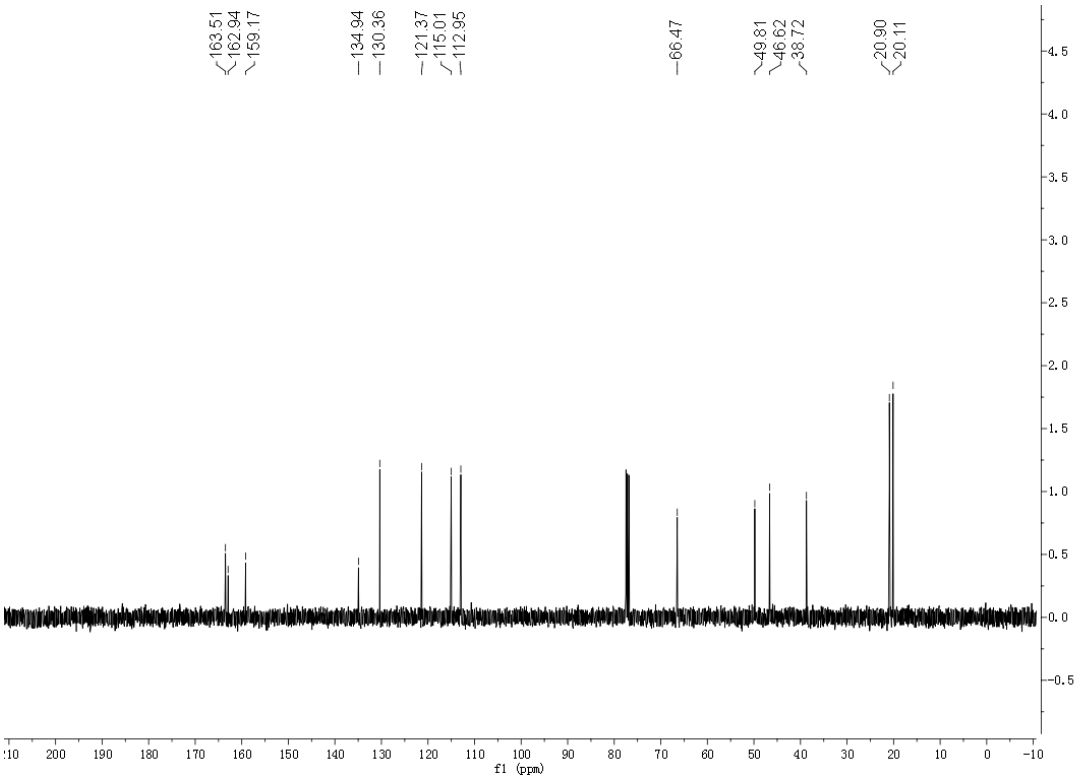
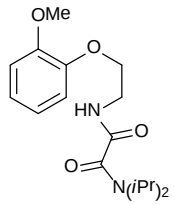
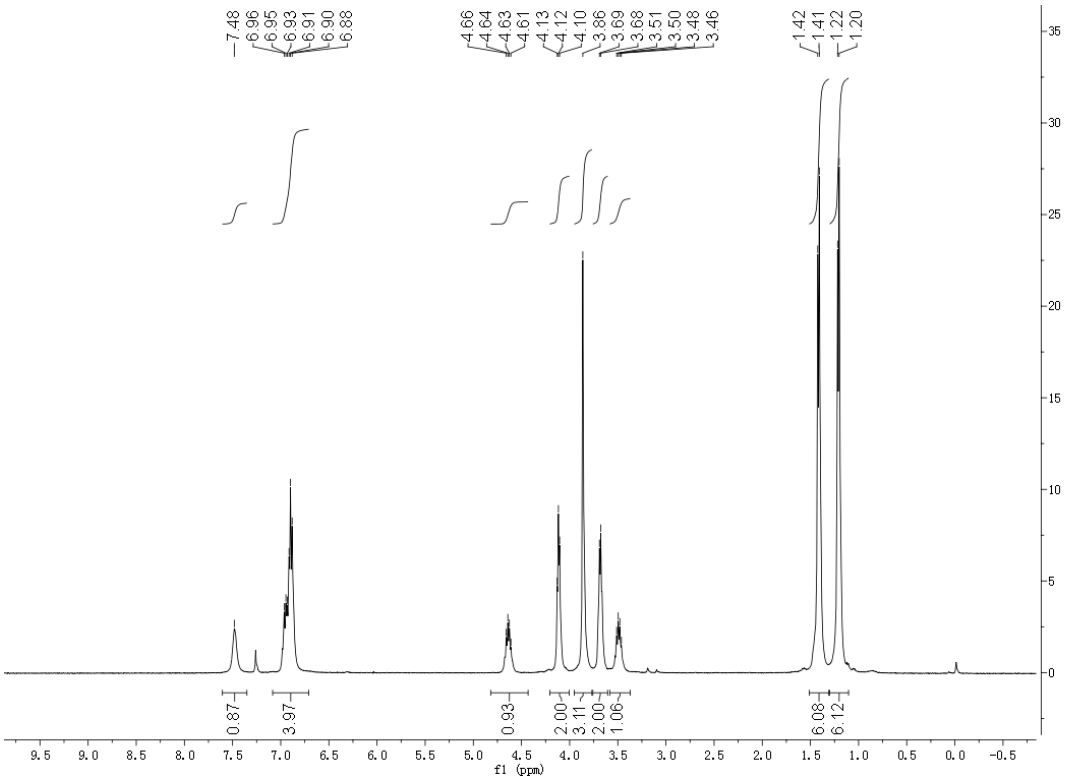
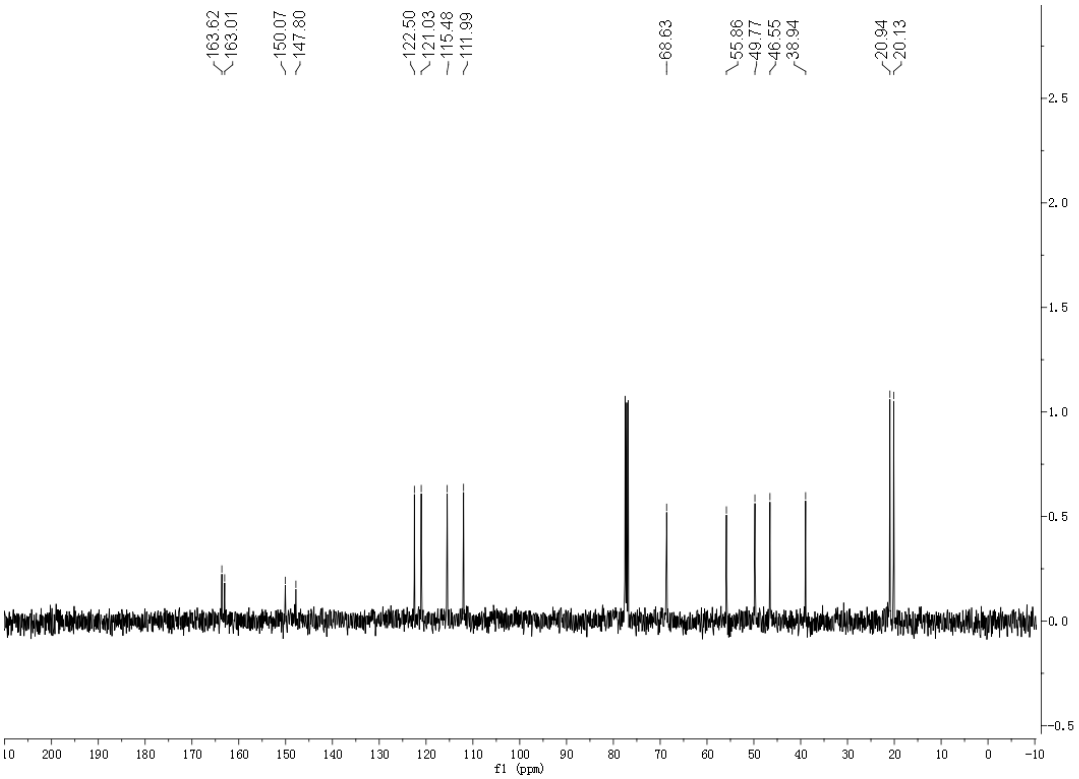
6.91
6.86
6.77
6.75
4.69
4.67
4.66
4.64
4.62
4.05
4.03
4.02
3.69
3.68
3.66
3.65
3.52
3.51
3.49
3.47
3.45
1.40
1.38
1.21
1.19
0.96
1.00
1.05
0.94
0.91
1.03
2.03
2.00
1.08
5.92
6.23
f1 (ppm)

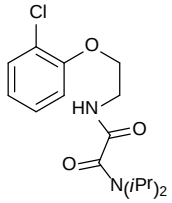

163.51
162.94
159.17
134.94
130.36
121.37
115.01
112.95
66.47
49.81
46.62
38.72
20.90
20.11
f1 (ppm)


OMe
O
HN
O
O
N(iPr)2

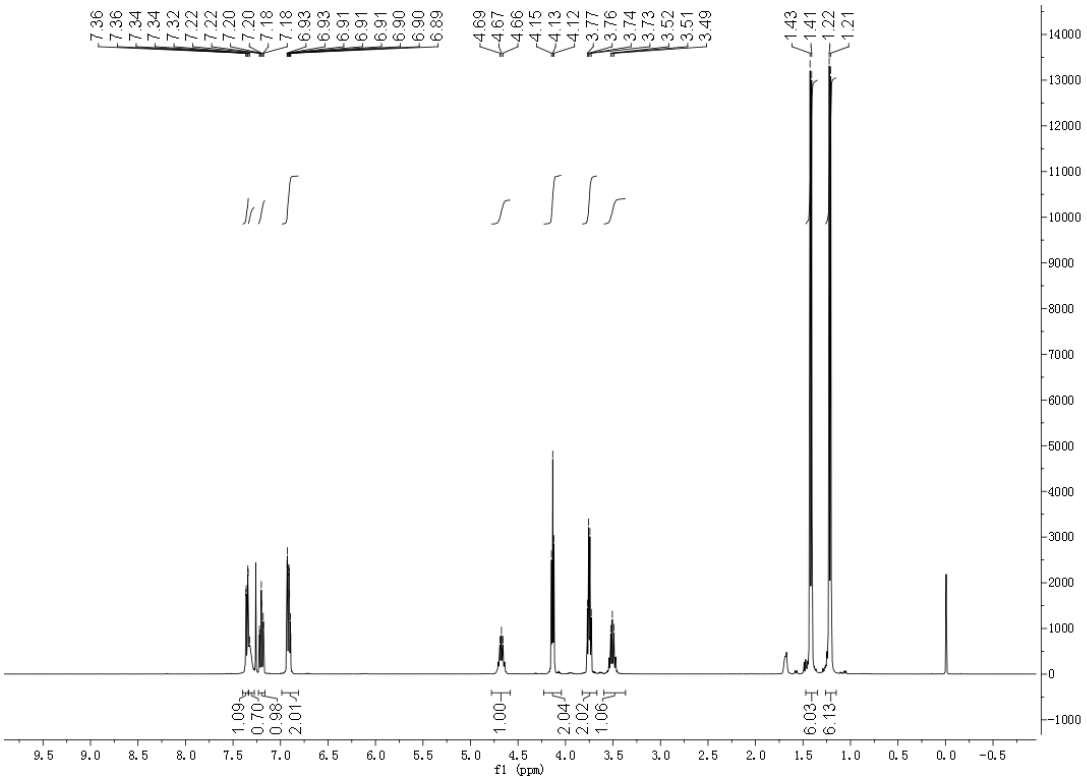
1w

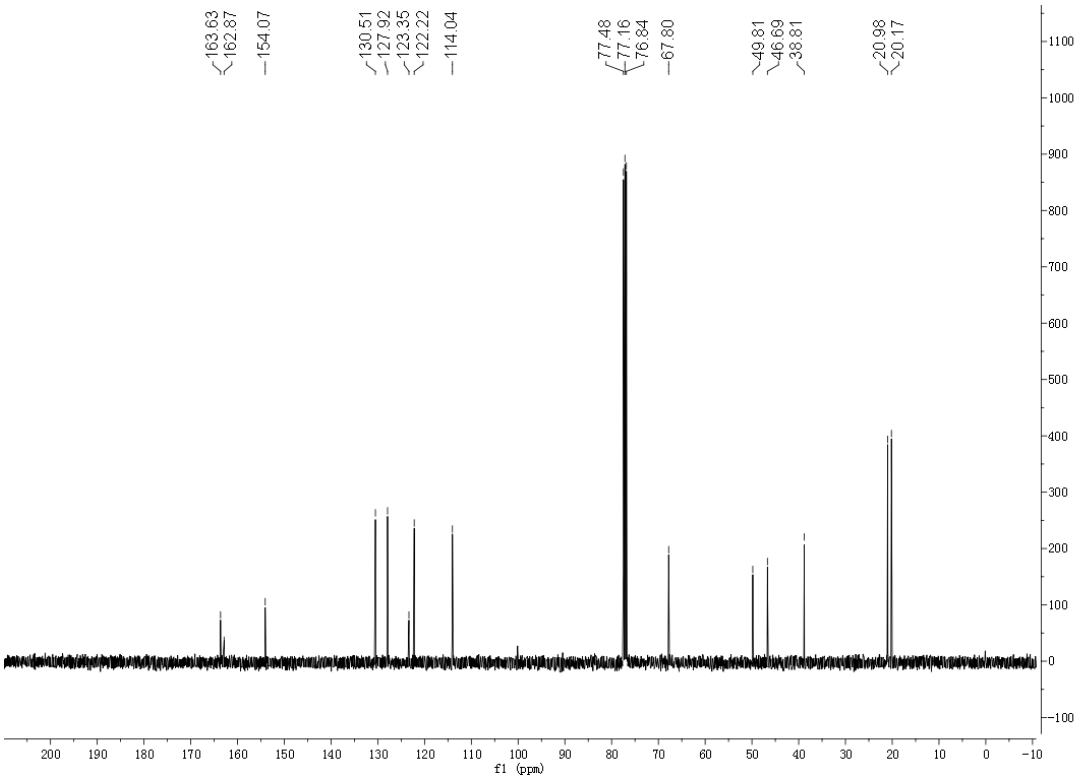

7.48
6.96
6.95
6.93
6.91
6.90
6.88
4.66
4.64
4.63
4.61
4.13
4.12
4.10
3.86
3.69
3.68
3.51
3.50
3.48
3.46
1.42
1.41
1.22
1.20
0.87
3.97
0.93
2.00
3.11
2.00
1.06
6.08
6.12
f1 (ppm)

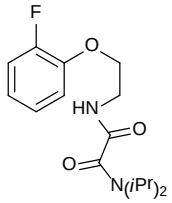

163.62
163.01
150.07
147.80
122.50
121.03
115.48
111.99
68.63
55.86
49.77
46.55
38.94
20.94
20.13
f1 (ppm)


Cl
O
HN
O
O
N(iPr)2

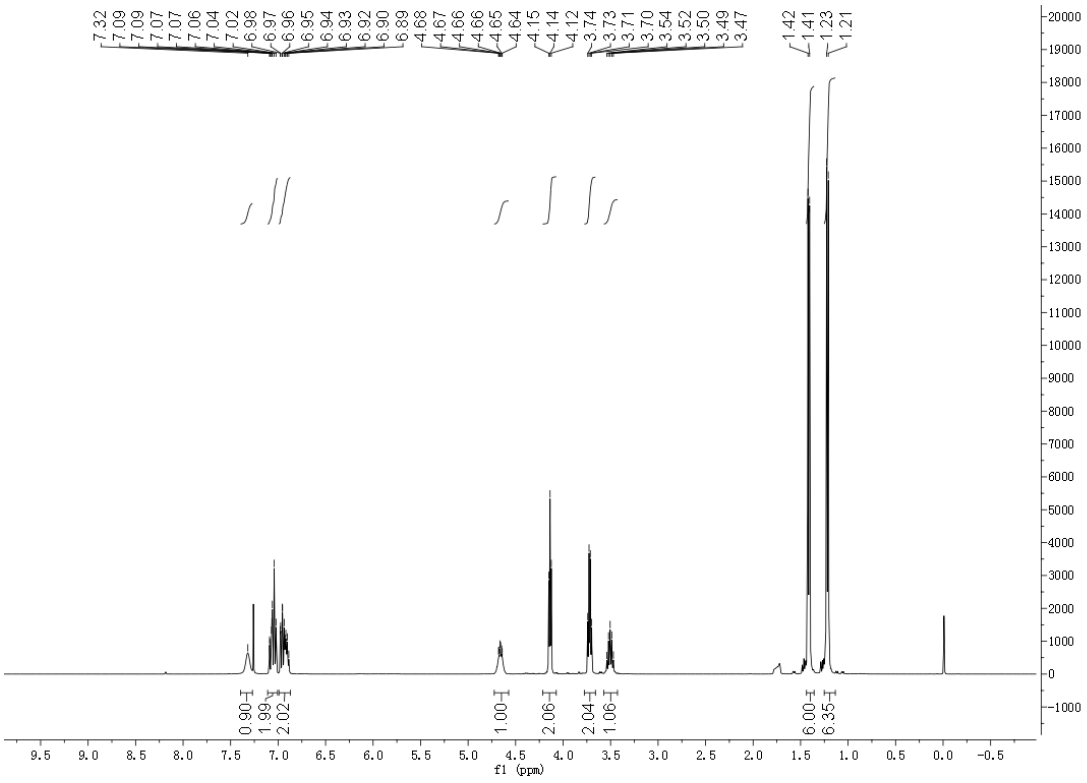
1x


7.36
7.36
7.34
7.34
7.32
7.22
7.22
7.20
7.20
7.18
7.18
6.93
6.93
6.91
6.91
6.91
6.90
6.90
6.89
4.69
4.67
4.66
4.15
4.13
4.12
3.77
3.76
3.74
3.73
3.52
3.51
3.49
1.43
1.41
1.22
1.21
1.09
0.70
0.98
2.01
1.00
2.04
2.02
1.06
6.03
6.13
f1 (ppm)


163.63
162.87
154.07
130.51
127.92
123.35
122.22
114.04
77.48
77.16
76.84
67.80
49.81
46.69
38.81
20.98
20.17
f1 (ppm)


F
O
HN
O
O
N(iPr)2

1y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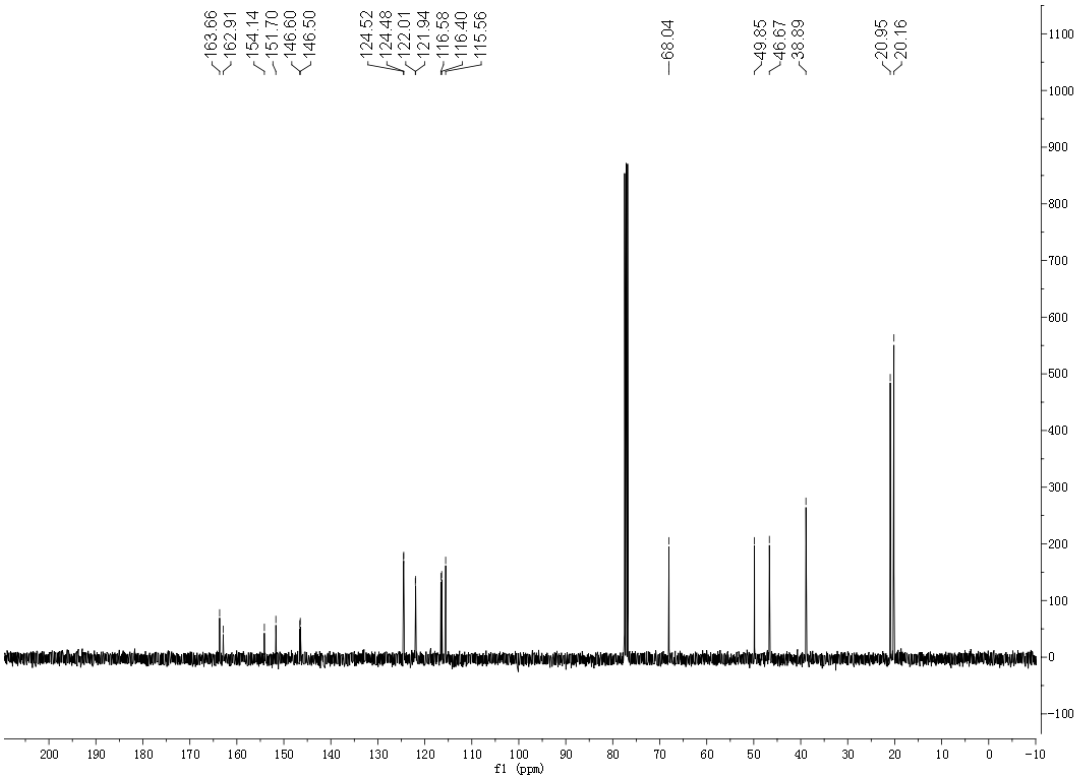
7.09
7.09
7.07
7.07
7.06
7.04
7.02
6.98
6.97
6.96
6.95
6.94
6.93
6.92
6.90
6.89
4.68
4.67
4.66
4.66
4.65
4.64
4.15
4.14
4.12
3.74
3.73
3.71
3.70
3.54
3.52
3.50
3.49
3.47
1.42
1.41
1.23
1.21
0.90
1.99
2.02
1.00
2.06
2.04
1.06
6.00
6.35
f1 (ppm)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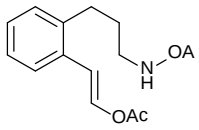
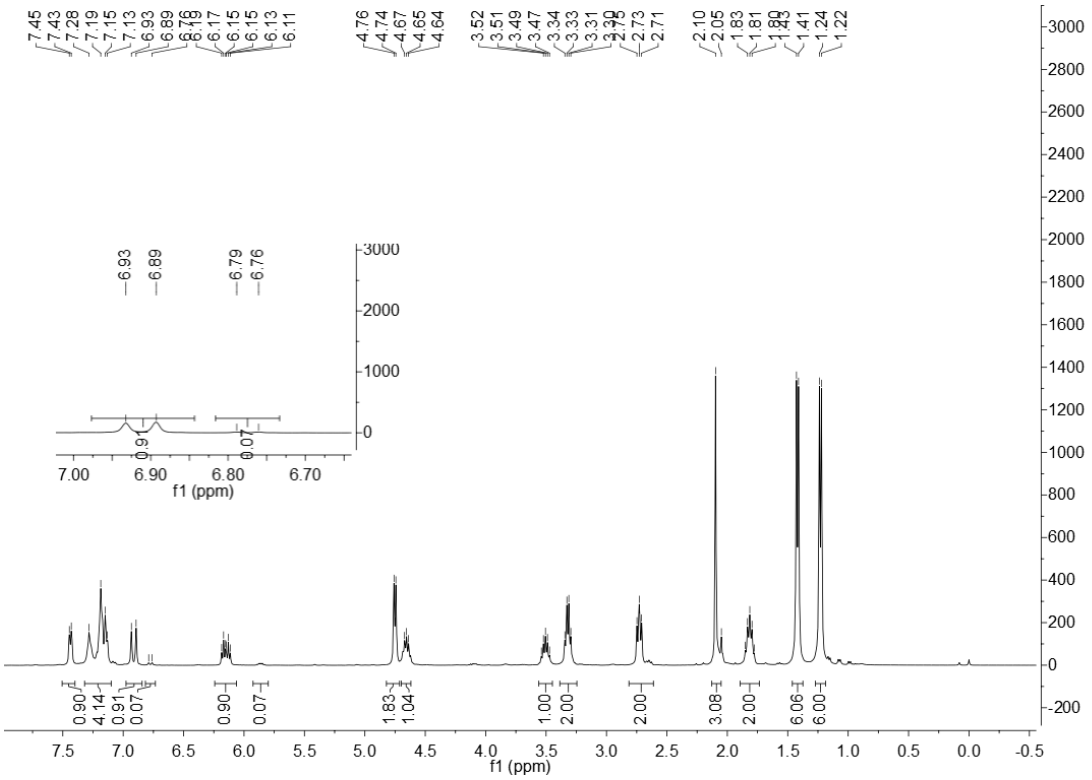
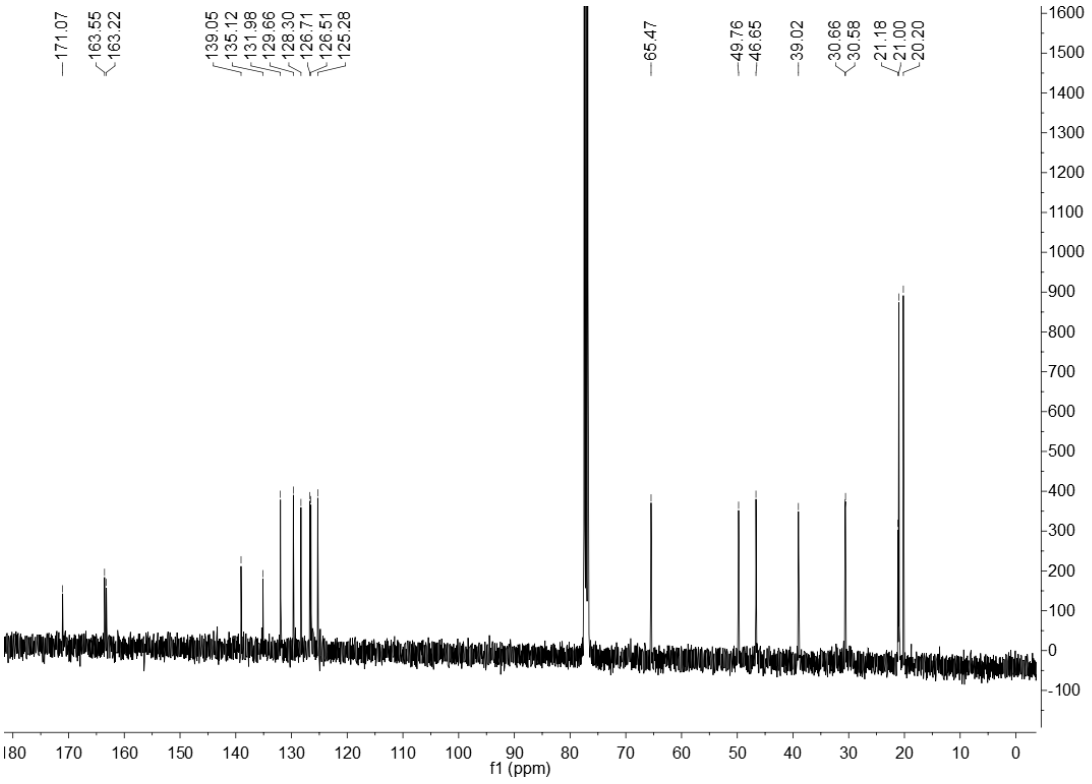
162.91
154.14
151.70
146.60
146.50
124.52
124.48
122.01
121.94
116.58
116.40
115.56
68.04
49.85
46.67
38.89
20.95
20.16
f1 (ppm)


OA
N
H
OAc

3a


7.45
7.43
7.28
7.19
7.15
7.13
6.93
6.89
6.17
6.15
6.13
6.11
4.76
4.74
4.67
4.65
4.64
3.52
3.51
3.49
3.47
3.34
3.33
3.31
2.73
2.71
2.10
2.05
1.83
1.81
1.41
1.24
1.22
6.93
6.89
6.79
6.76
0.90
0.07
7.00
6.90
6.80
6.70
f1 (ppm)
0.90
4.14
0.91
0.07
0.90
0.07
1.83
1.04
1.00
2.00
2.00
3.08
2.00
6.06
6.00
f1 (ppm)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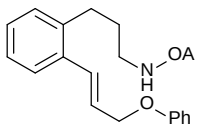
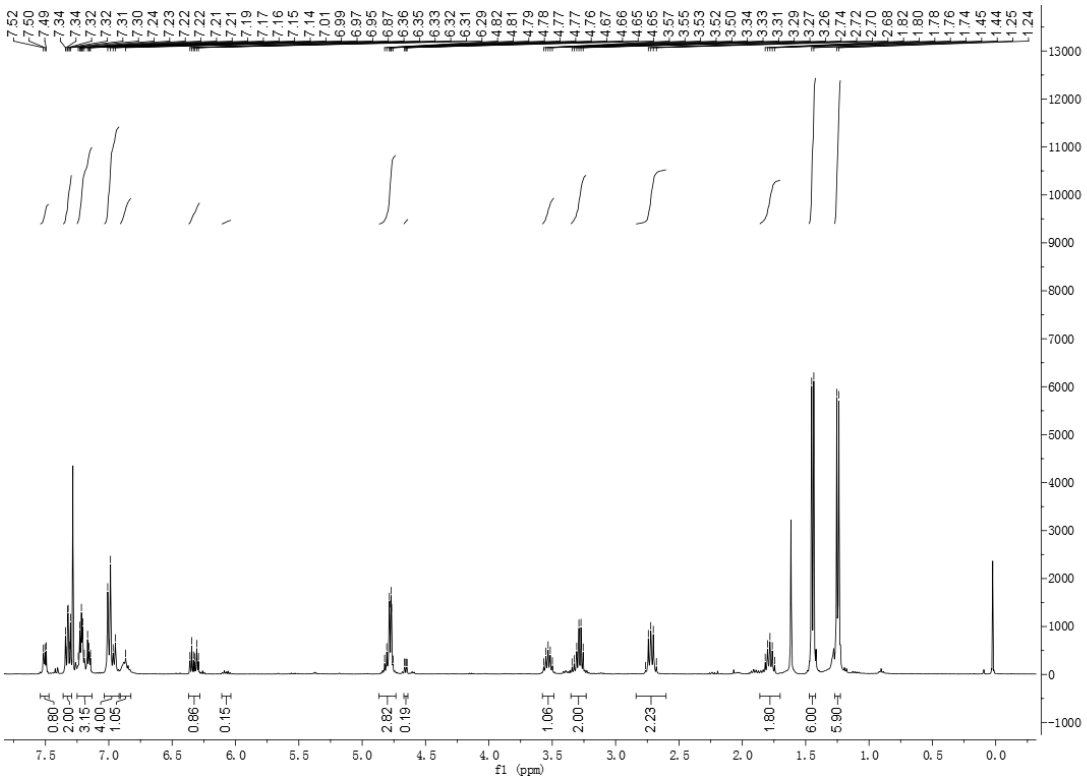
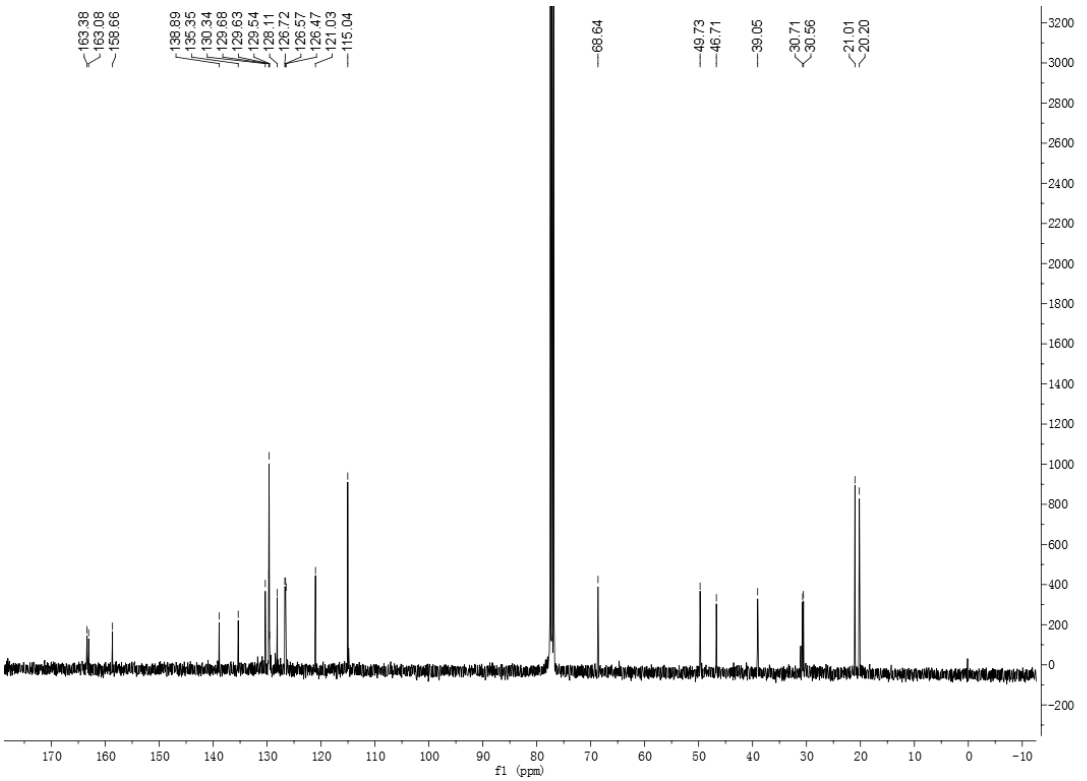
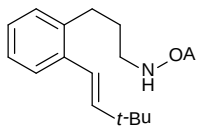
163.55
163.22
139.05
135.12
131.98
129.66
128.30
126.71
126.51
125.28
65.47
49.76
46.65
39.02
30.66
30.58
21.18
21.00
20.20
f1 (ppm)



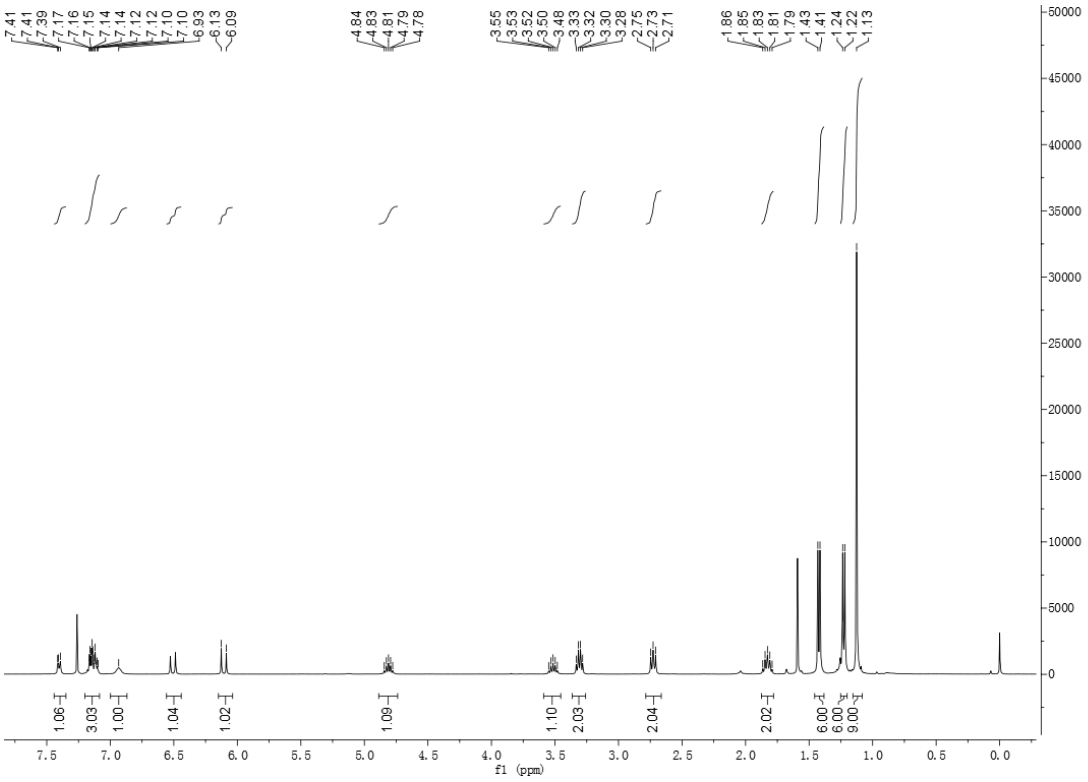
3b

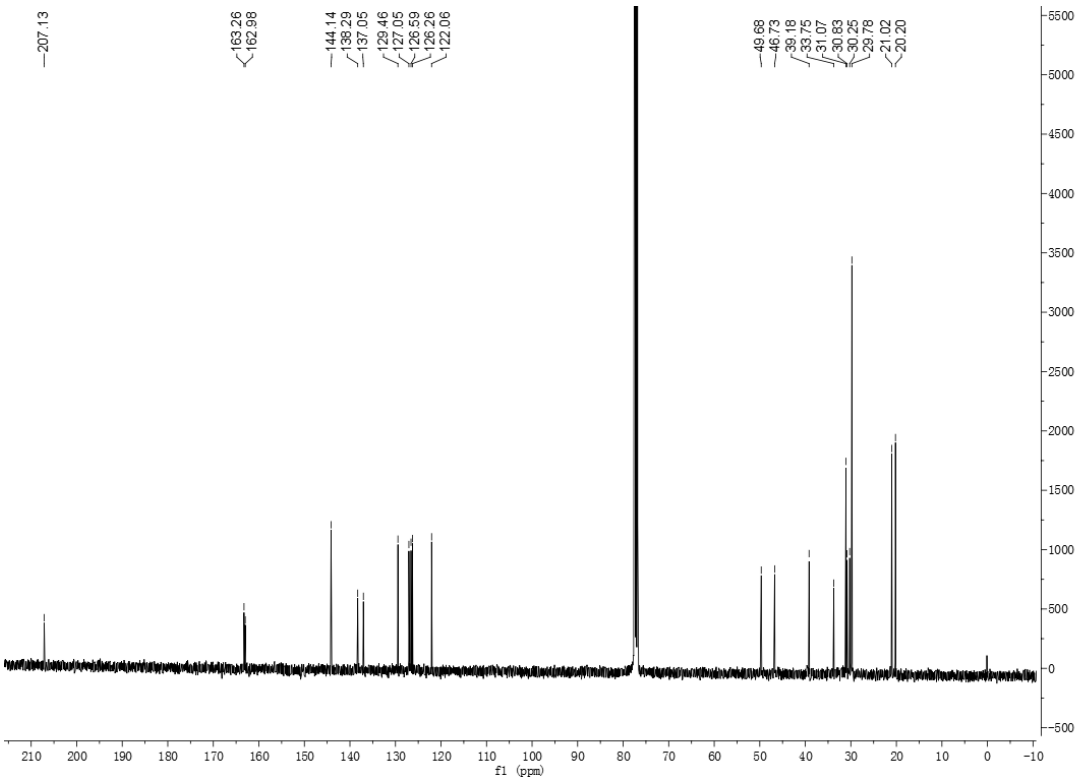





OA
N
H
t-Bu

3c


7.41
7.41
7.39
7.17
7.16
7.15
7.14
7.14
7.12
7.12
7.10
7.10
6.93
6.13
6.09
4.84
4.83
4.81
4.79
4.78
3.55
3.53
3.52
3.50
3.48
3.33
3.32
3.30
3.28
2.75
2.73
2.71
1.86
1.85
1.83
1.81
1.79
1.43
1.41
1.24
1.22
1.13
1.06
3.03
1.00
1.04
1.02
1.09
1.10
2.03
2.04
2.02
6.00
6.00
9.00
f1 (ppm)


207.13
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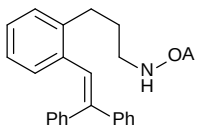
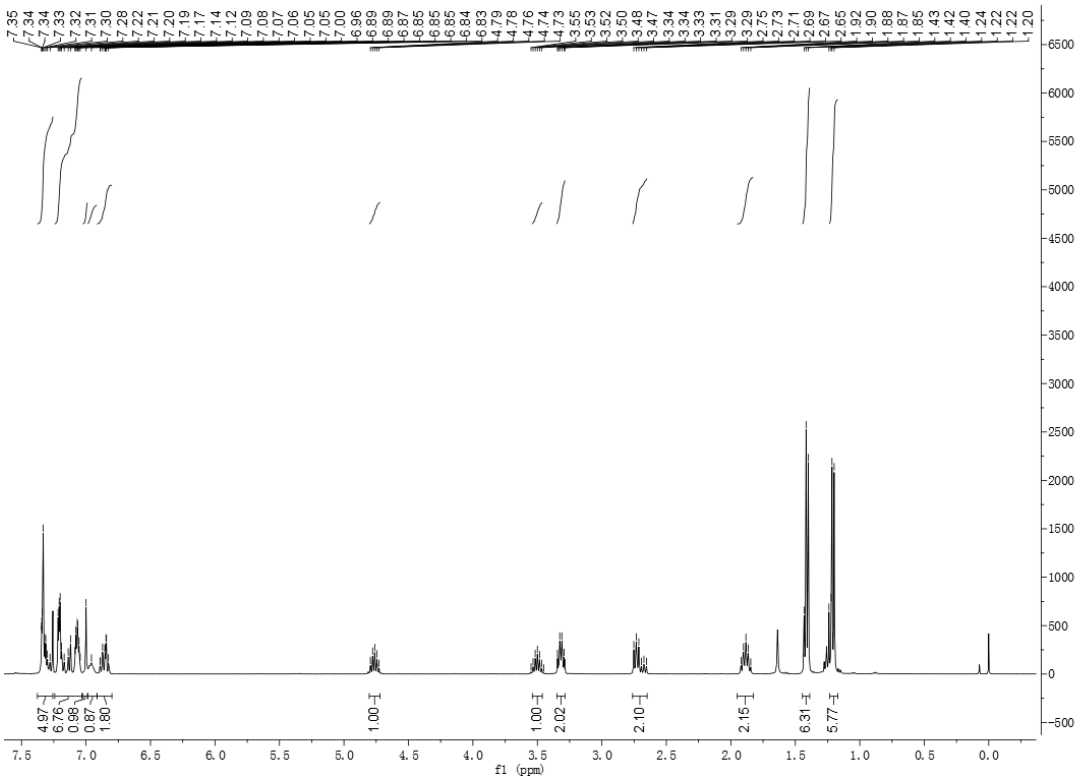
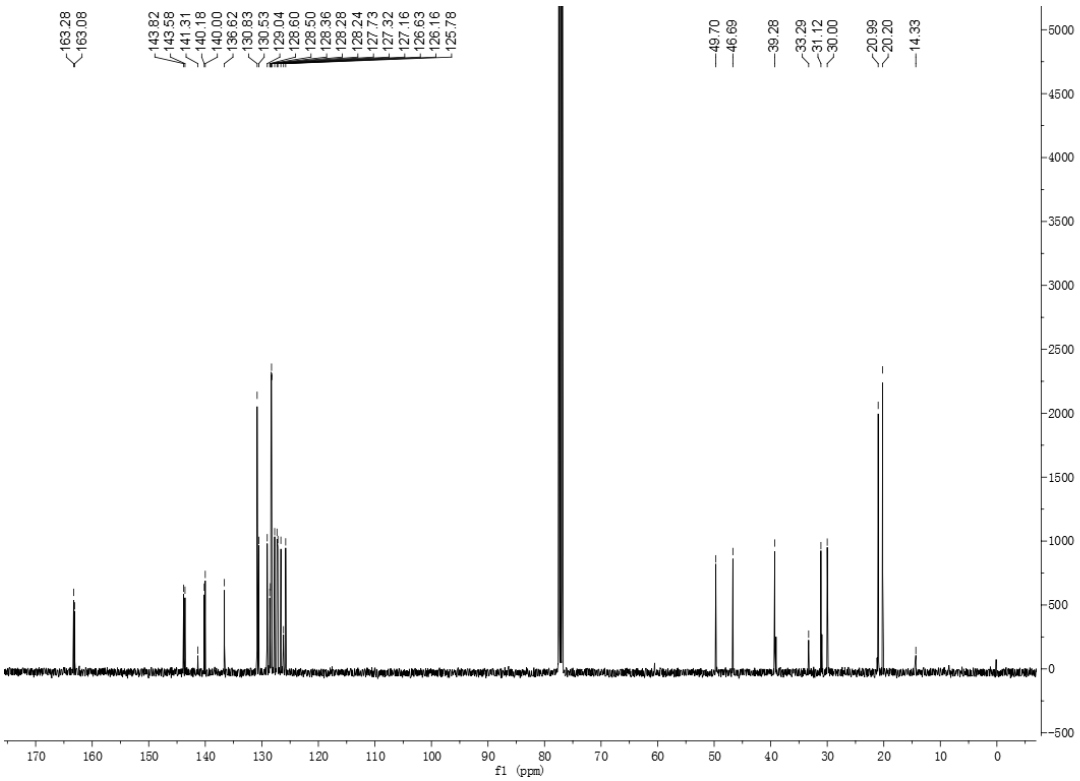
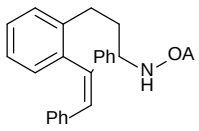
162.98
144.14
138.29
137.05
129.46
127.05
126.59
126.26
122.06
49.68
46.73
39.18
33.75
31.07
30.83
30.25
29.78
21.02
20.20
f1 (ppm)


OA
N
H
Ph
Ph

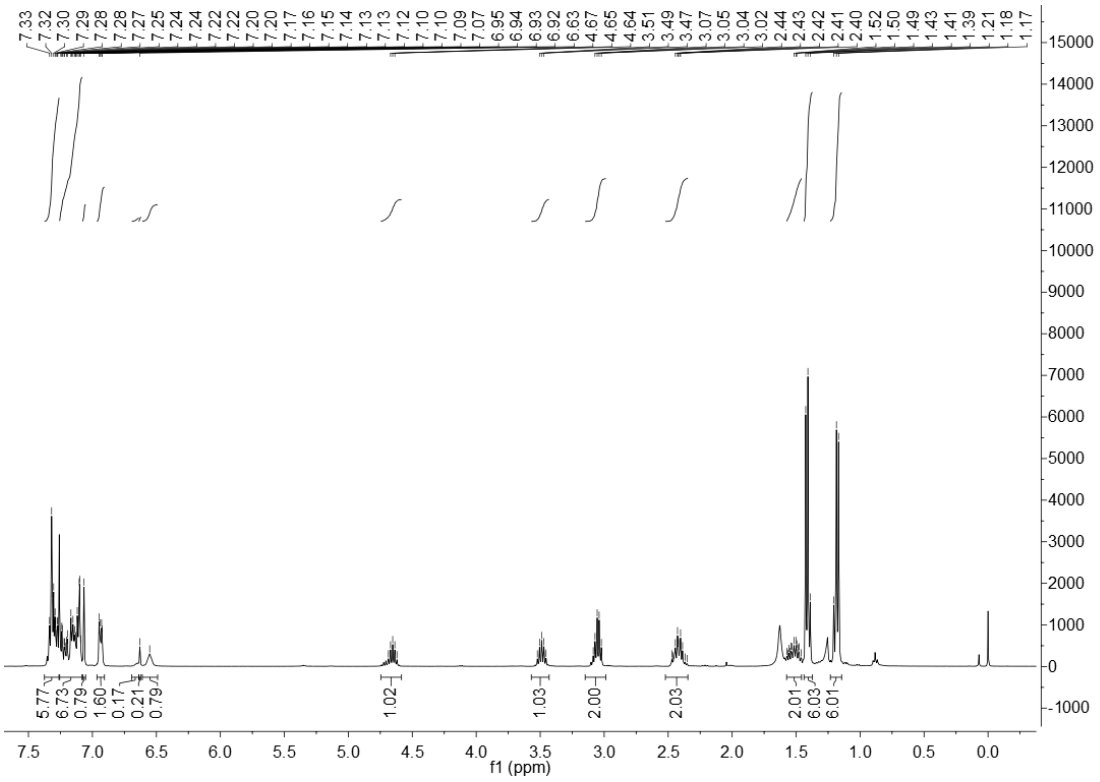
3d

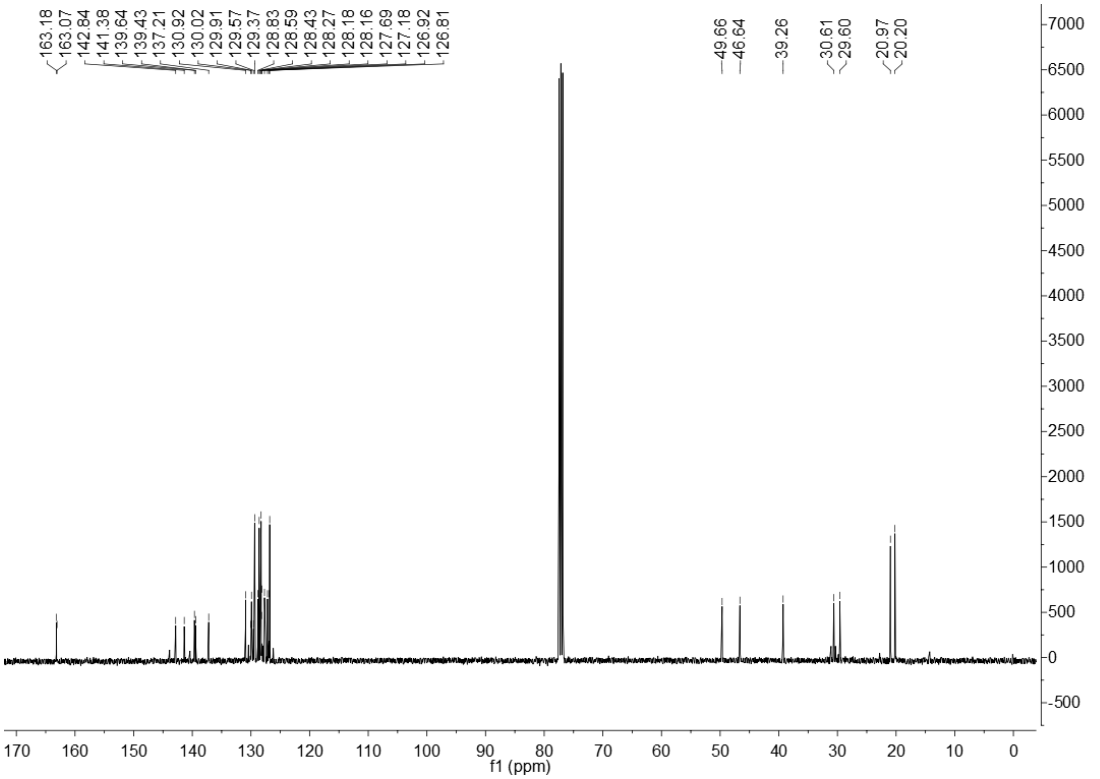

7.35 7.34 7.34 7.33 7.32 7.31 7.30 7.28 7.22 7.21 7.20 7.19 7.17 7.14 7.12 7.09 7.08 7.07 7.06 7.05 7.05 7.00 6.96 6.89 6.89 6.87 6.85 6.85 6.85 6.84 6.83 4.79 4.78 4.76 4.74 4.73 3.55 3.53 3.52 3.50 3.48 3.47 3.34 3.34 3.33 3.31 3.29 3.29 2.75 2.73 2.71 2.69 2.67 2.65 1.92 1.90 1.88 1.87 1.85 1.43 1.42 1.40 1.24 1.22 1.22 1.20
4.97 6.76 0.98 0.87 1.80 1.00 1.00 2.02 2.10 2.15 6.31 5.77
f1 (ppm)

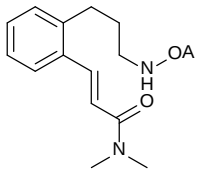

163.28 163.08 143.82 143.58 141.31 140.18 140.00 136.62 130.83 130.53 129.04 128.60 128.50 128.36 128.28 128.24 127.73 127.32 127.16 126.63 126.16 125.78 49.70 46.69 39.28 33.29 31.12 30.00 20.99 20.20 14.33
f1 (ppm)


Ph
N
H
OA
Ph

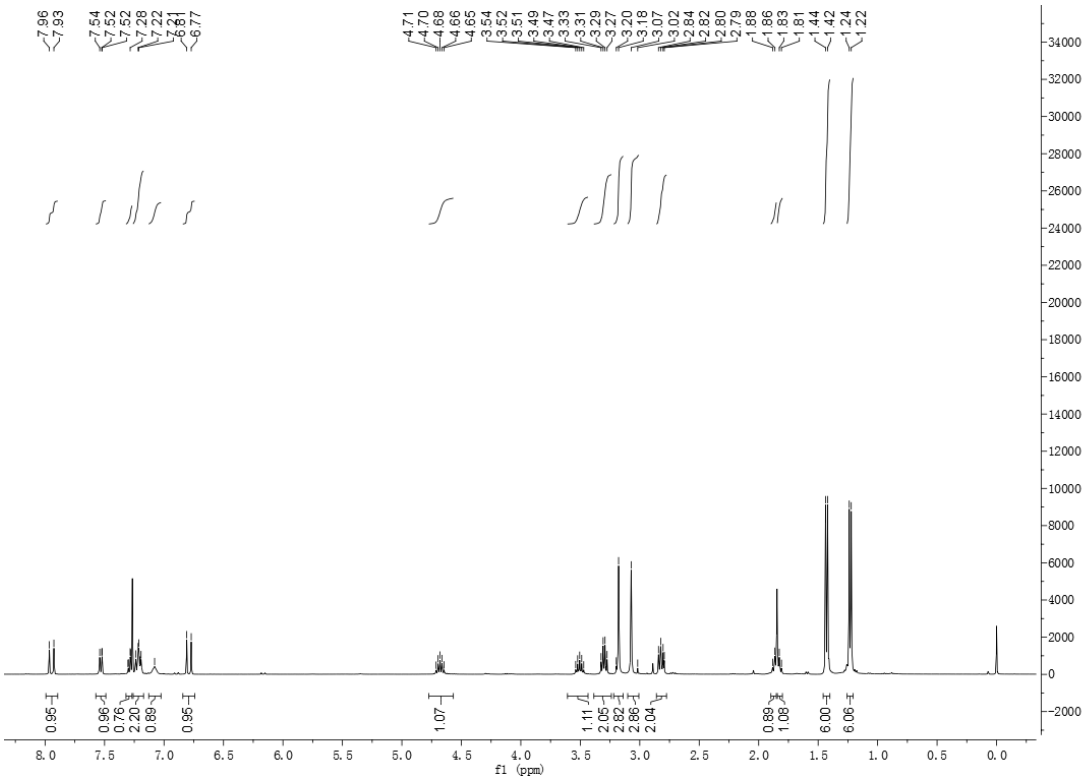
3e

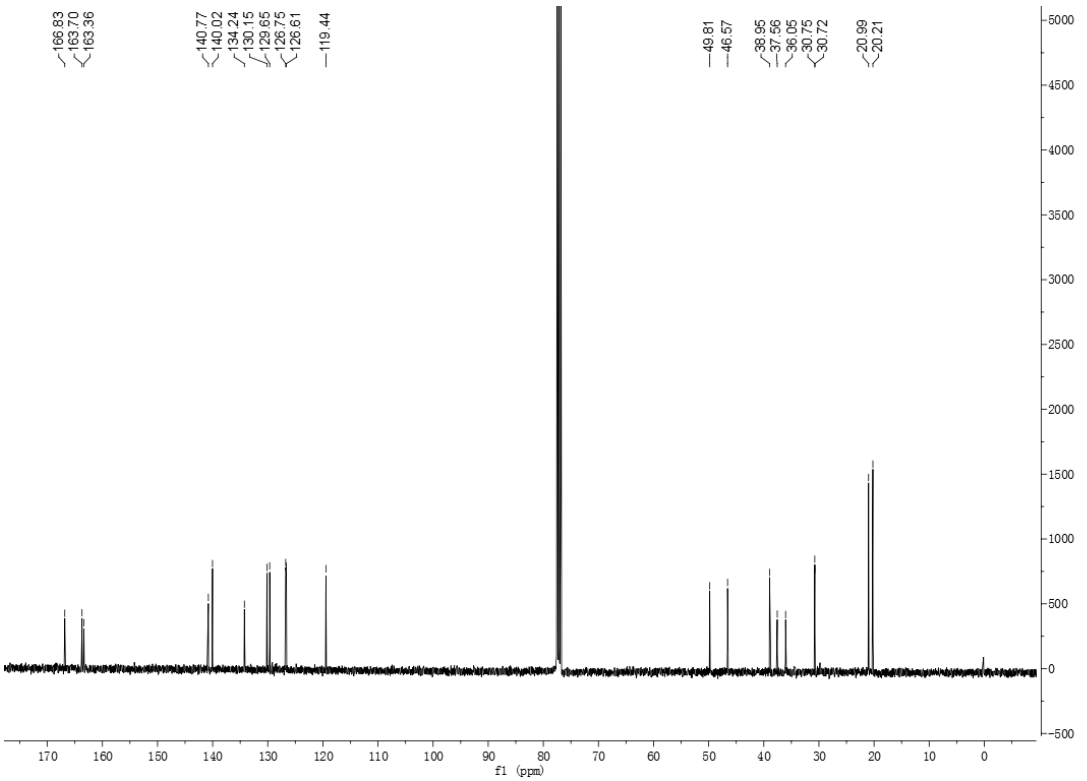

7.33 7.32 7.30 7.29 7.28 7.28 7.27 7.25 7.24 7.24 7.22 7.22 7.20 7.20 7.17 7.16 7.15 7.14 7.13 7.13 7.12 7.10 7.10 7.09 7.07 6.95 6.94 6.93 6.92 6.63 4.67 4.65 4.64 3.51 3.49 3.47 3.07 3.05 3.04 3.02 2.44 2.43 2.42 2.41 2.40 1.52 1.50 1.49 1.43 1.41 1.39 1.21 1.18 1.17
5.77 6.73 0.79 1.60 0.17 0.21 0.79 1.02 1.03 2.00 2.03 2.01 6.03 6.01
f1 (ppm)

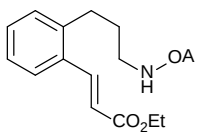

163.18 163.07 142.84 141.38 139.64 139.43 137.21 130.92 130.02 129.91 129.57 129.37 128.83 128.59 128.43 128.27 128.18 128.16 127.69 127.18 126.92 126.81
49.66 46.64 39.26 30.61 29.60 20.97 20.20
f1 (ppm)


OA
N
H
O
N

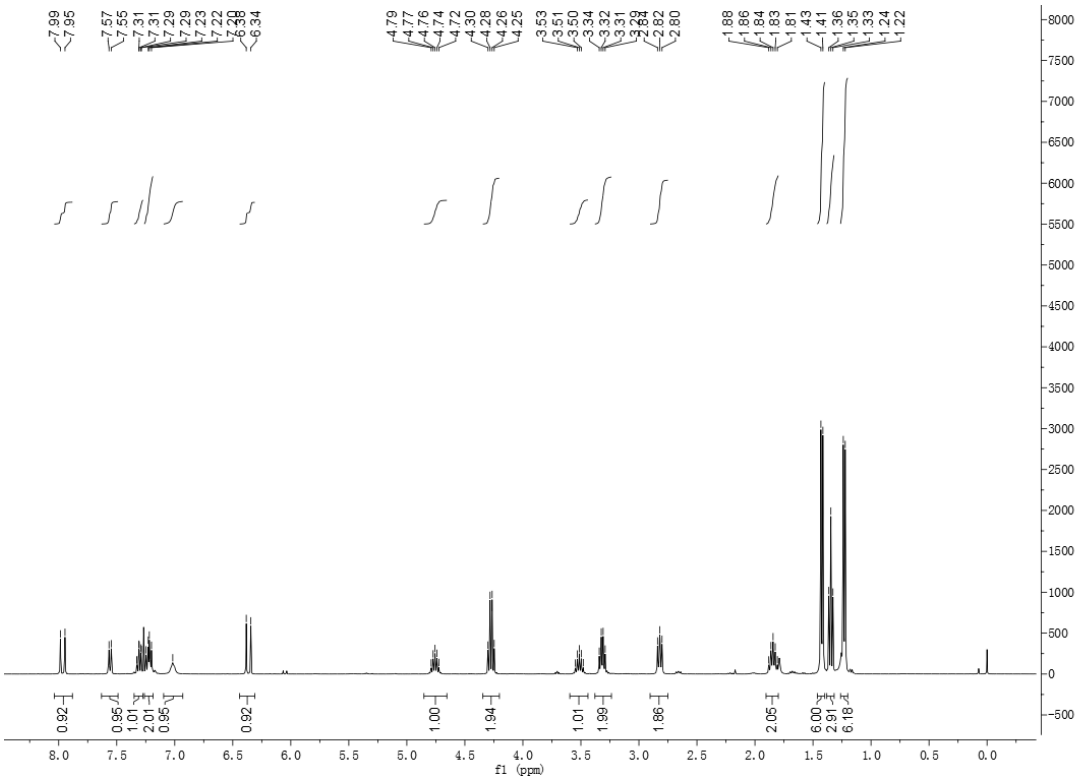
3f


7.96
7.93
7.54
7.52
7.52
7.52
7.28
7.22
6.81
6.77
4.71
4.70
4.68
4.66
4.65
3.54
3.52
3.51
3.49
3.47
3.33
3.31
3.29
3.27
3.20
3.18
3.07
3.02
2.84
2.82
2.80
2.79
1.88
1.86
1.83
1.81
1.44
1.42
1.24
1.22
0.95
0.96
0.76
2.20
0.89
0.95
1.07
1.11
2.05
2.82
2.86
2.04
0.89
1.08
6.00
6.06
f1 (ppm)


166.83
163.70
163.36
140.77
140.02
134.24
130.15
129.65
126.75
126.61
119.44
49.81
46.57
38.95
37.56
36.05
30.75
30.72
20.99
20.21
f1 (ppm)


OA
N
H
CO2Et

3g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7.22
6.38
6.34
4.79
4.77
4.76
4.74
4.72
4.30
4.28
4.26
4.25
3.53
3.51
3.50
3.34
3.32
3.31
2.82
2.80
1.88
1.86
1.84
1.83
1.81
1.43
1.41
1.36
1.35
1.33
1.24
1.22
0.92
0.95
1.01
2.01
0.95
0.92
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1.94
1.01
1.99
1.86
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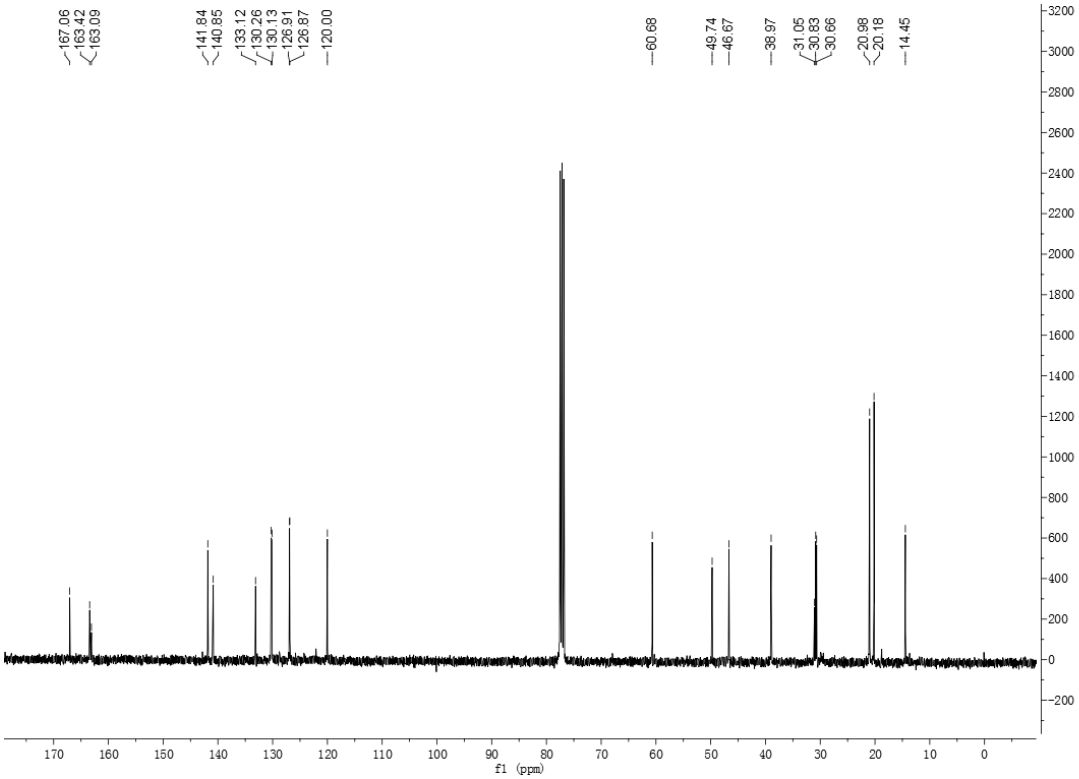
6.00
2.91
6.18
f1 (ppm)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130.26
130.13
126.91
126.87
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60.68
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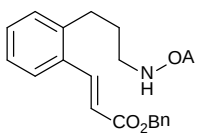
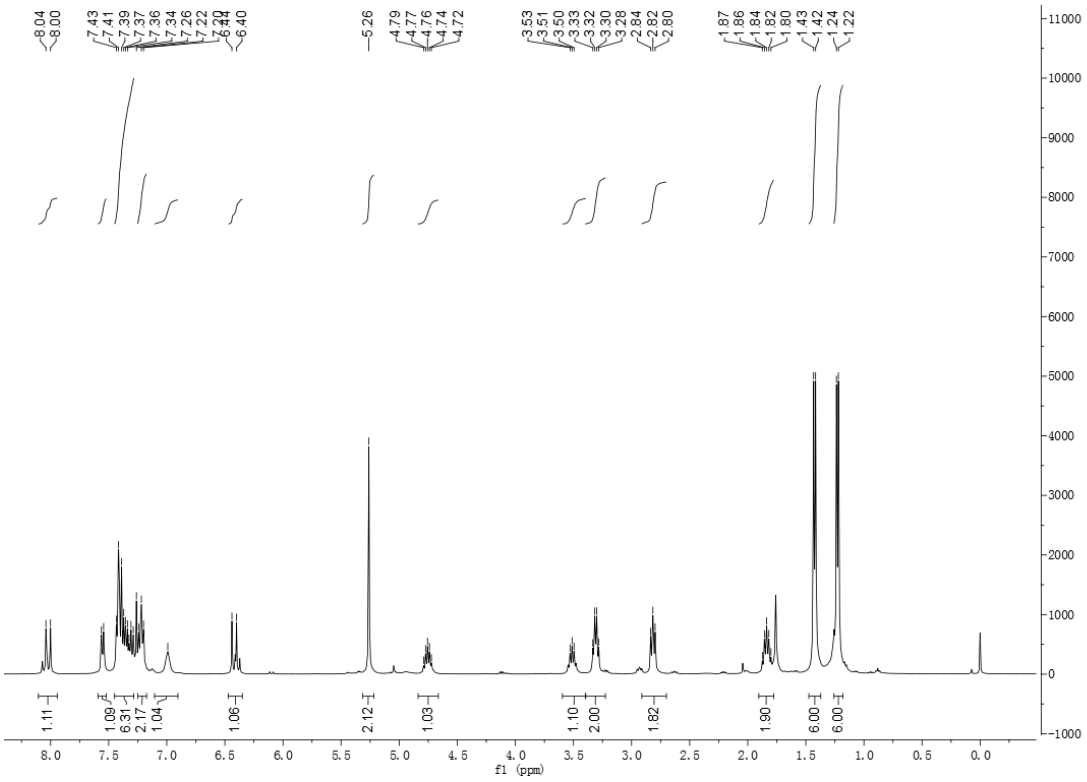
46.67
38.97
31.05
30.83
30.66
20.98
20.18
14.45
f1 (ppm)


OA
N
H
CO2Bn

3h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7.22
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5.26
4.79
4.77
4.76
4.74
4.72
3.53
3.51
3.50
3.33
3.32
3.30
3.28
2.84
2.82
2.80
1.87
1.86
1.84
1.82
1.80
1.43
1.42
1.24
1.22
1.11
1.09
6.31
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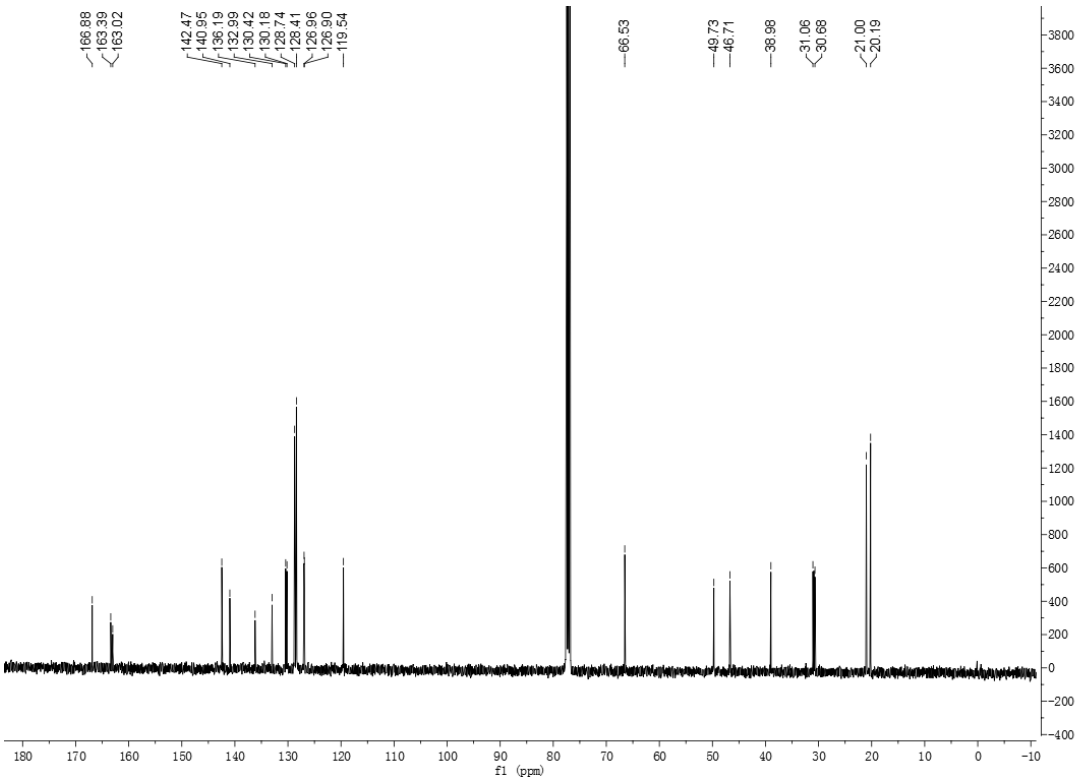
2.12
1.03
1.10
2.00
1.82
1.90
6.00
6.00
f1 (ppm)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130.42
130.18
128.74
128.41
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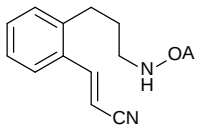
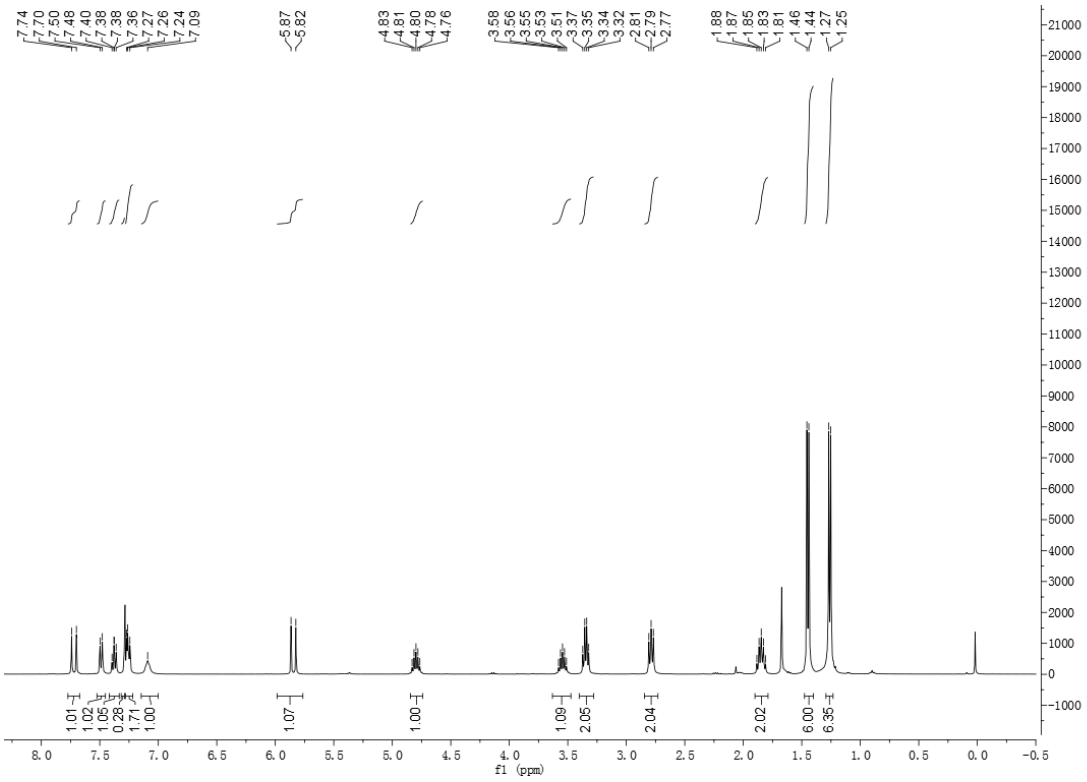
31.06
30.68
21.00
20.19
f1 (ppm)


OA
N
H
CN

3i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7.36
7.27
7.26
7.24
7.09
5.87
5.82
4.83
4.81
4.80
4.78
4.76
3.58
3.56
3.55
3.53
3.51
3.37
3.35
3.34
3.32
2.81
2.79
2.77
1.88
1.87
1.85
1.83
1.81
1.46
1.44
1.27
1.25
1.01
1.02
1.05
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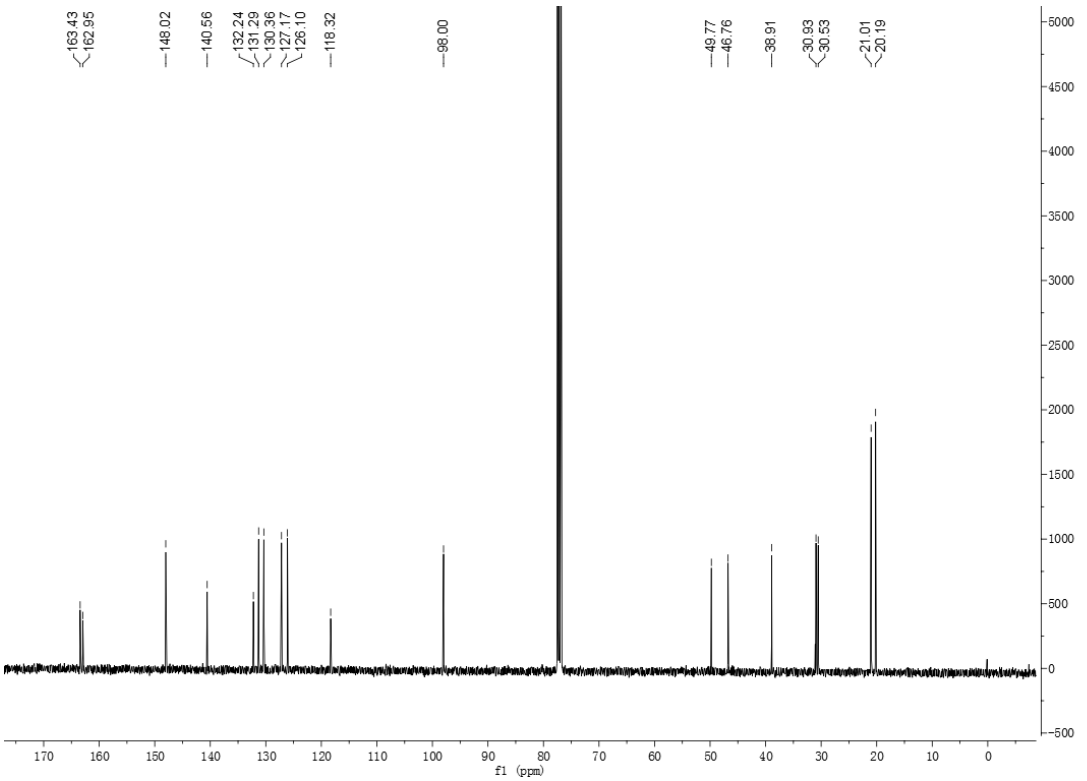
1.07
1.00
1.09
2.05
2.04
2.02
6.00
6.35
f1 (ppm)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130.36
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126.10
118.32
98.00
49.77
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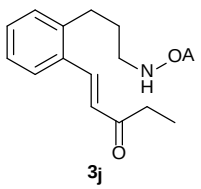
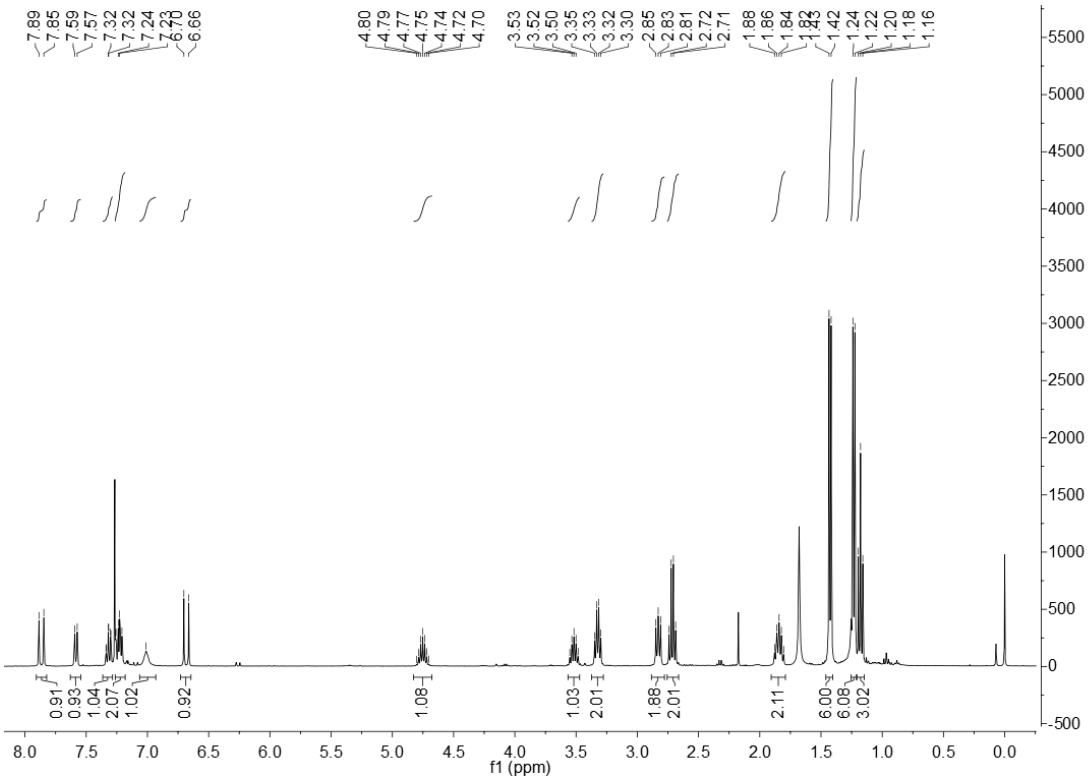
30.93
30.53
21.01
20.19
f1 (ppm)


OA
N
H
O

3j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7.23
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6.66
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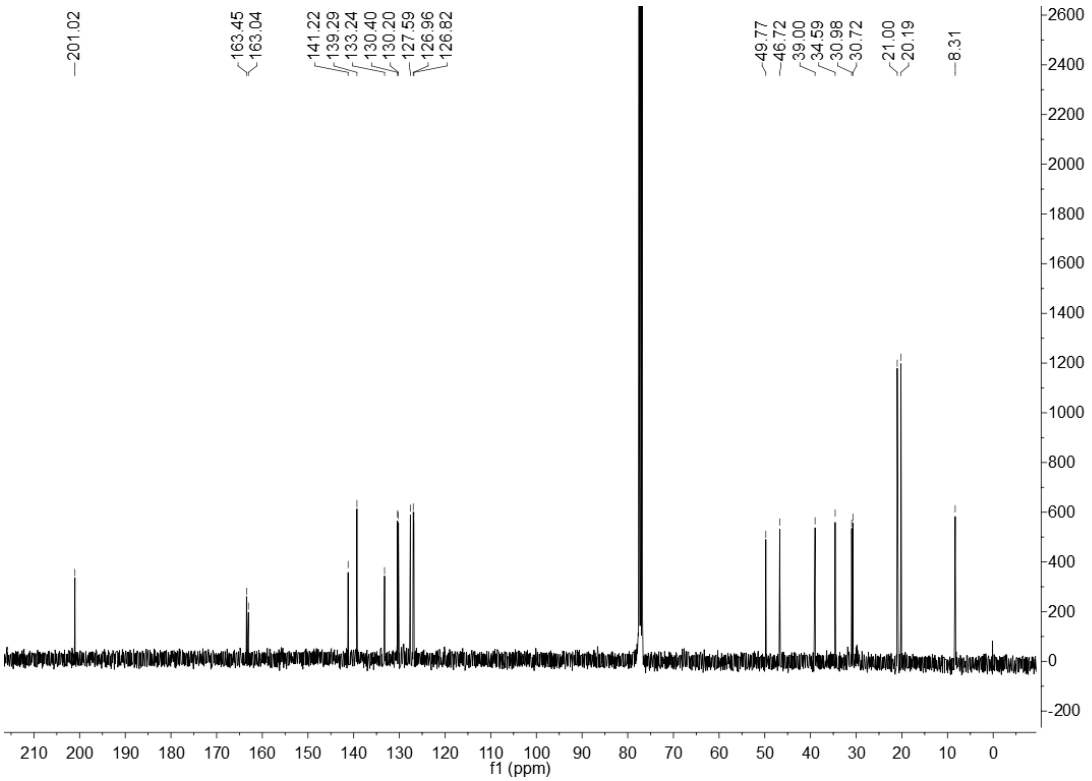
4.79
4.77
4.75
4.74
4.72
4.70
3.53
3.52
3.50
3.35
3.33
3.32
3.30
2.85
2.83
2.81
2.72
2.71
1.88
1.86
1.84
1.42
1.24
1.22
1.20
1.18
1.16
0.91
0.93
1.04
2.07
1.02
0.92
1.08
1.03
2.01
1.88
2.01
2.11
6.00
6.08
3.02
f1 (ppm)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130.40
130.20
127.59
126.96
126.82
49.77
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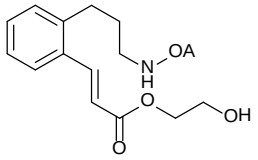
30.72
21.00
20.19
8.31
f1 (ppm)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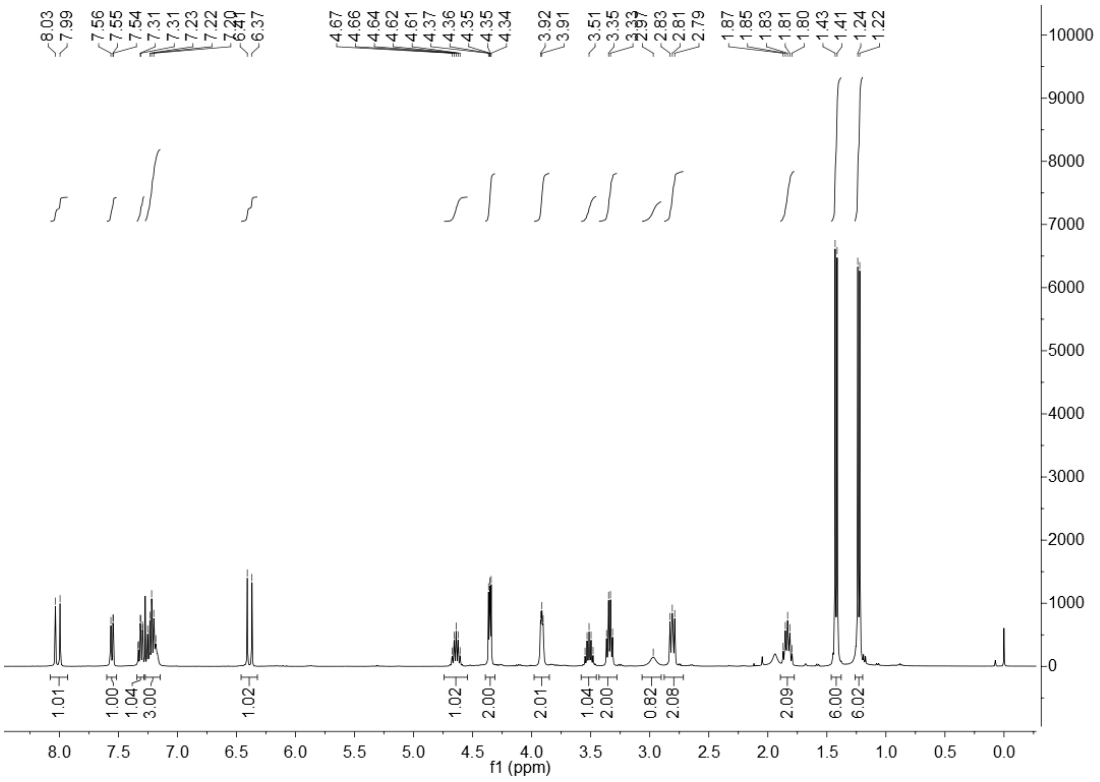
N
H
O
OH
O

3k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7.22
6.41
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4.66
4.64
4.62
4.61
4.37
4.36
4.35
4.35
4.34
3.92
3.91
3.51
3.35
3.33
2.83
2.81
2.79
1.87
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1.83
1.81
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1.24
1.22
1.01
1.00
1.04
3.00
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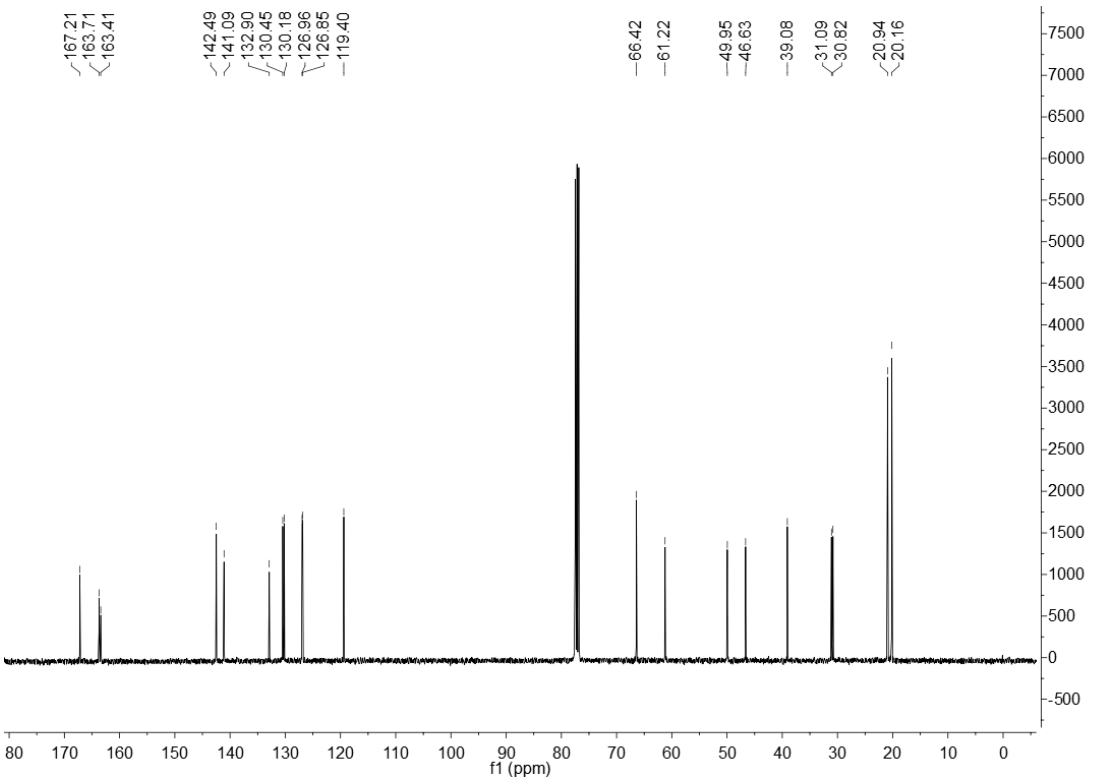
1.02
2.00
2.01
1.04
2.00
0.82
2.08
2.09
6.00
6.02
f1 (ppm)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130.18
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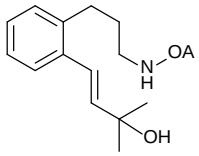
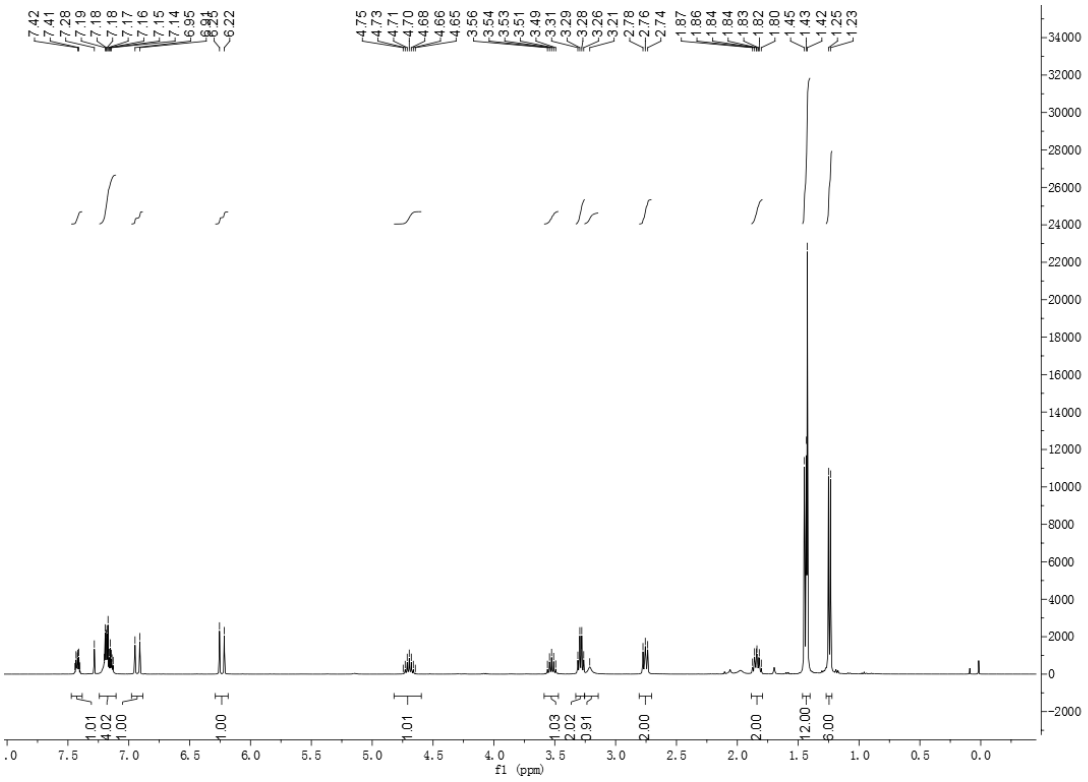
31.09
30.82
20.94
20.16
f1 (ppm)


N
H
OA
OH

3l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7.17
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7.15
7.14
6.95
6.94
6.25
6.22
4.75
4.73
4.71
4.70
4.68
4.66
4.65
3.56
3.54
3.53
3.51
3.49
3.31
3.29
3.28
3.26
3.21
2.78
2.76
2.74
1.87
1.86
1.84
1.84
1.83
1.82
1.80
1.45
1.43
1.42
1.25
1.23
1.01
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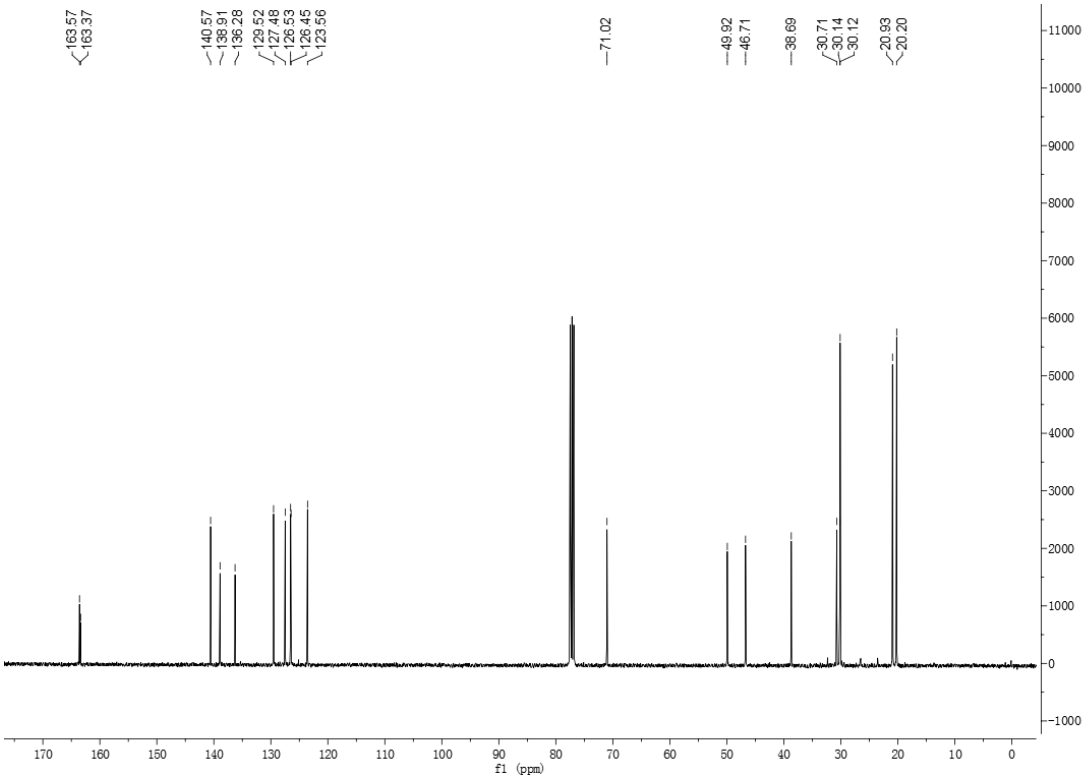
1.01
1.03
2.02
0.91
2.00
2.00
12.00
6.00
f1 (ppm)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136.28
129.52
127.48
126.53
126.45
123.56
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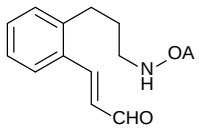
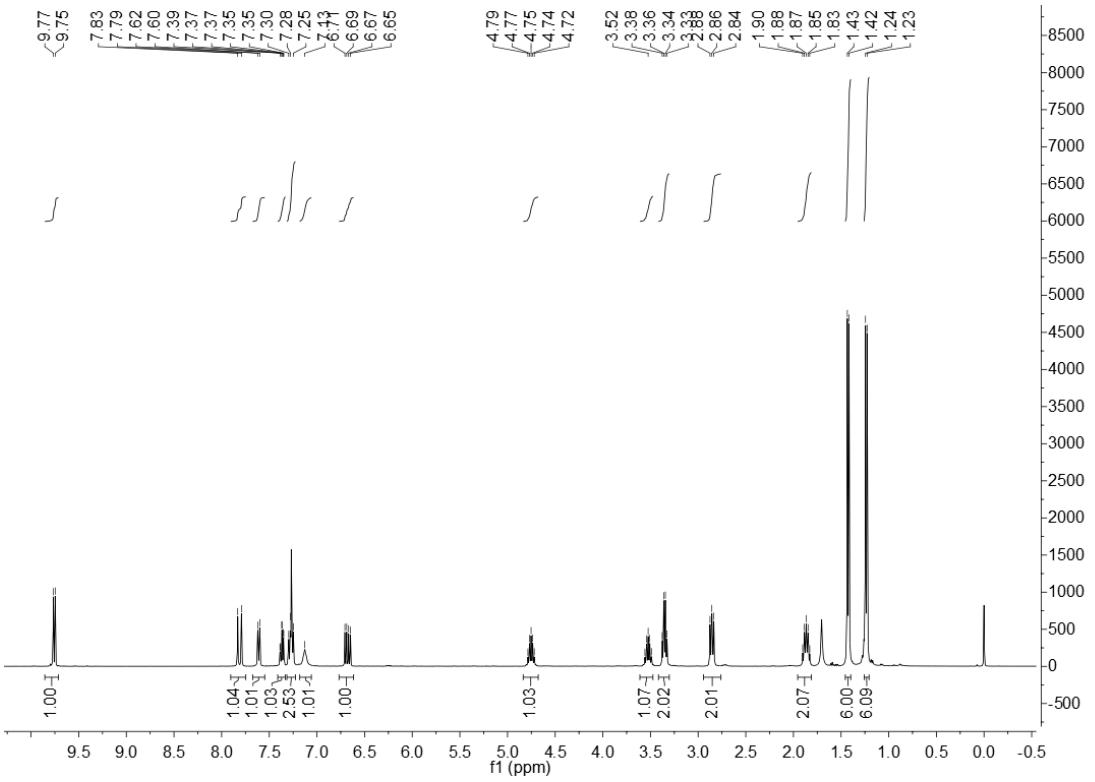
49.92
46.71
38.69
30.71
30.14
30.12
20.93
20.20
f1 (ppm)


OA
N
H
CHO

3m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7.37
7.37
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7.35
7.30
7.28
7.25
7.13
6.71
6.69
6.67
6.65
4.79
4.77
4.75
4.74
4.72
3.52
3.38
3.36
3.34
3.33
2.88
2.86
2.84
1.90
1.88
1.87
1.85
1.83
1.43
1.42
1.24
1.23
1.00
1.04
1.01
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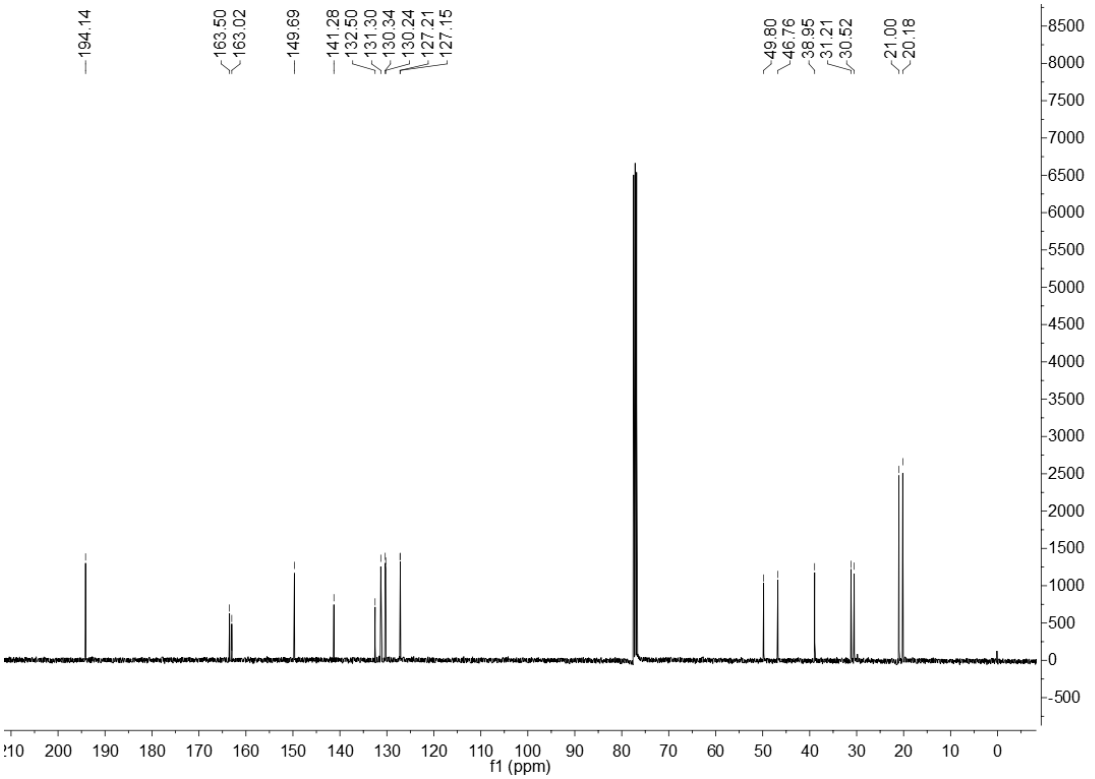
1.00
1.03
1.07
2.02
2.01
2.07
6.00
6.09
f1 (ppm)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132.50
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130.34
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127.21
127.15
49.80
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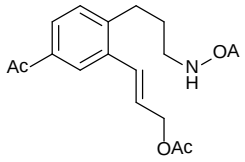
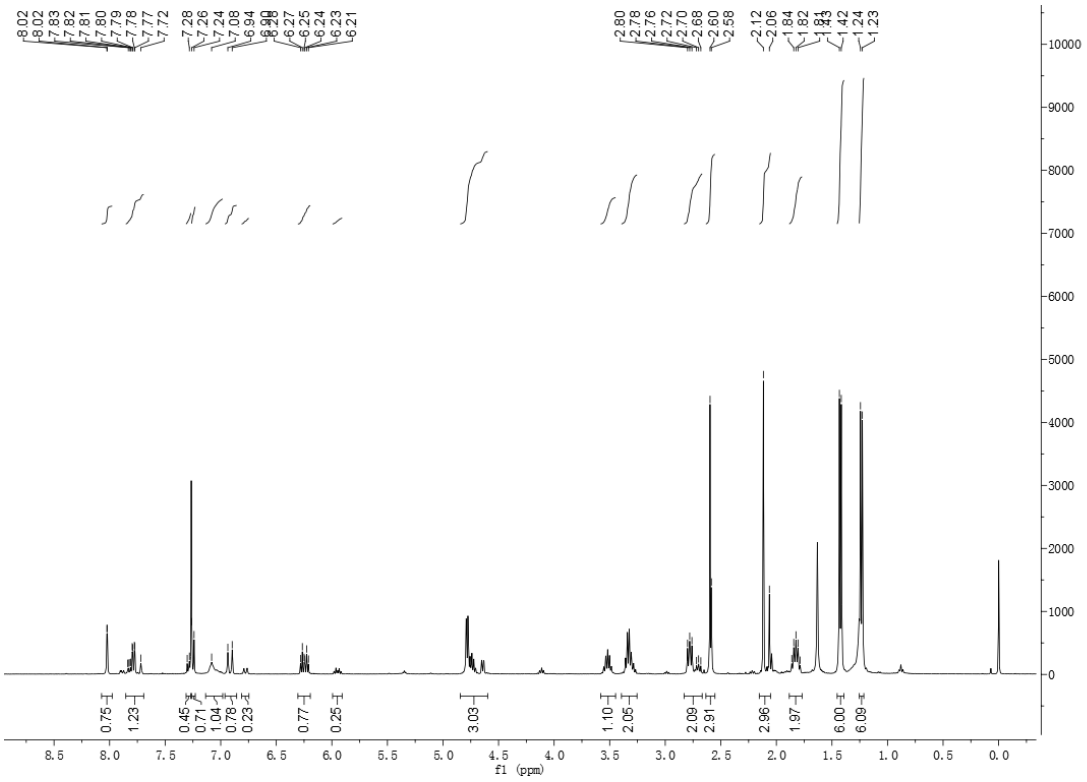
31.21
30.52
21.00
20.18
f1 (ppm)


Ac
N
H
OA
OAc

3n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7.78
7.77
7.72
7.28
7.26
7.24
7.08
6.94
6.27
6.25
6.24
6.23
6.21
2.80
2.78
2.76
2.72
2.70
2.68
2.60
2.58
2.12
2.06
1.84
1.82
1.42
1.24
1.23
0.75
1.23
0.45
0.71
1.04
0.78
0.23
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2.09
2.91
2.96
1.97
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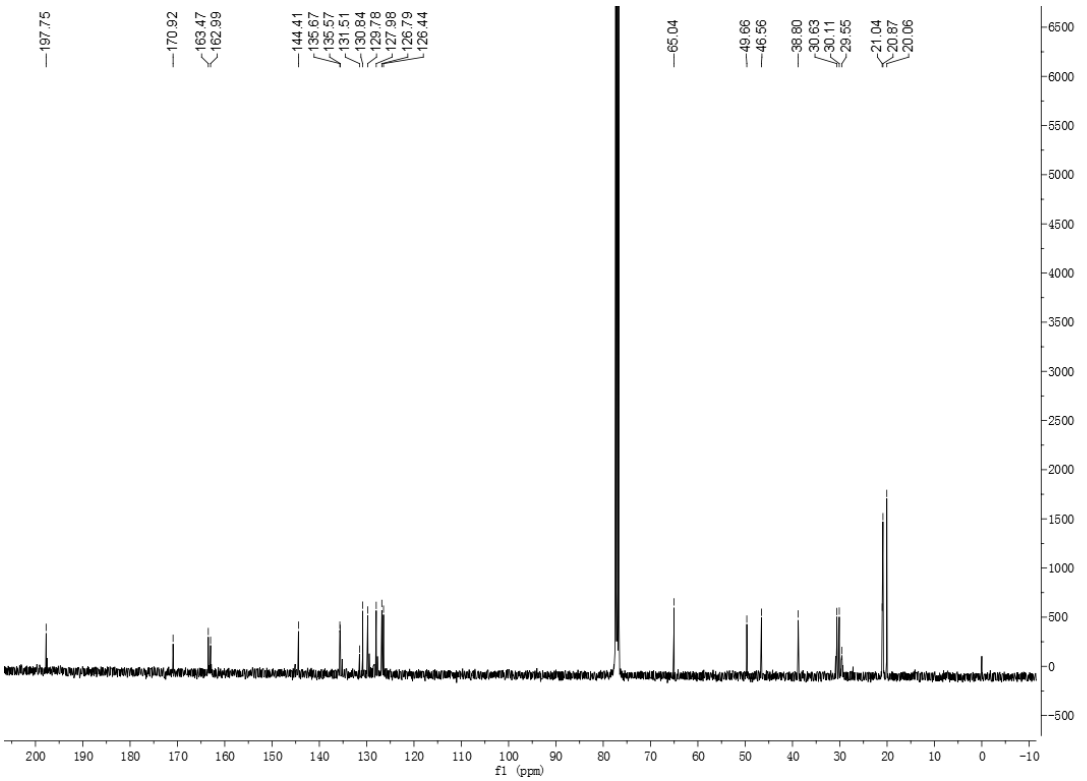
6.09
f1 (ppm)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131.51
130.84
129.78
127.98
126.79
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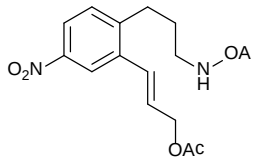
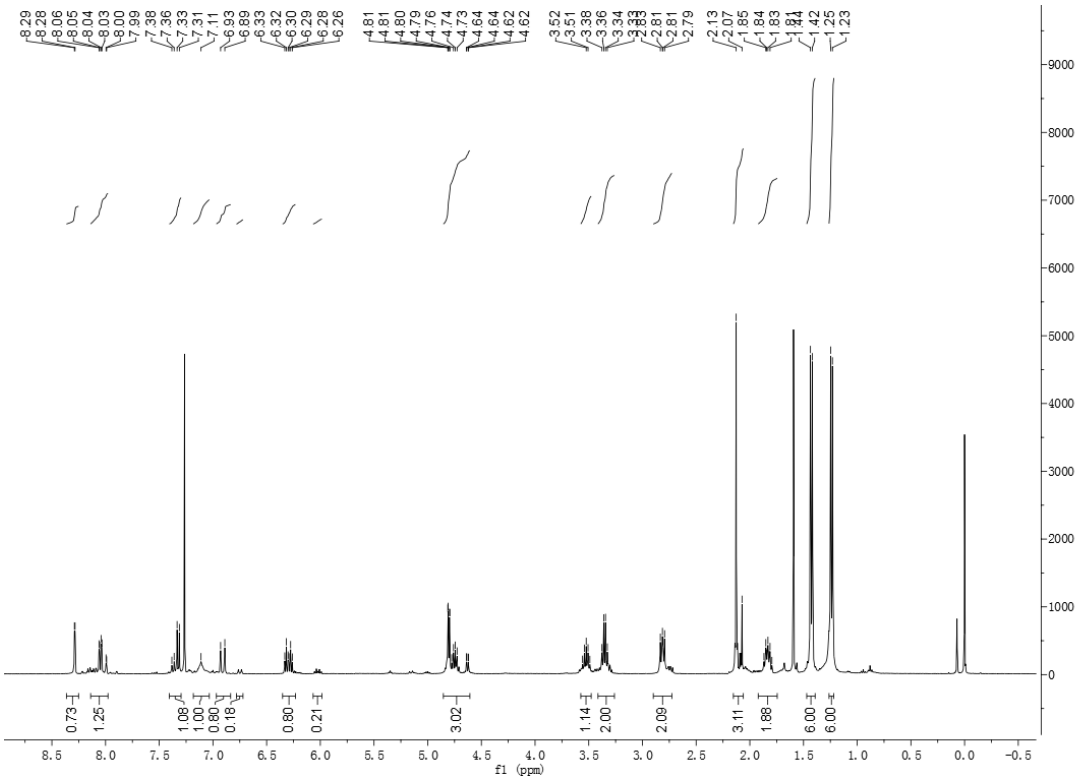
29.55
21.04
20.87
20.06
f1 (ppm)


O2N
N
H
OA
OAc

3o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7.99
7.38
7.36
7.33
7.31
7.11
6.93
6.89
6.33
6.32
6.30
6.29
6.28
6.26
4.81
4.81
4.80
4.79
4.76
4.74
4.73
4.64
4.64
4.62
4.62
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2.07
1.85
1.84
1.83
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1.23
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1.08
1.00
0.80
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0.80
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1.14
2.00
2.09
3.11
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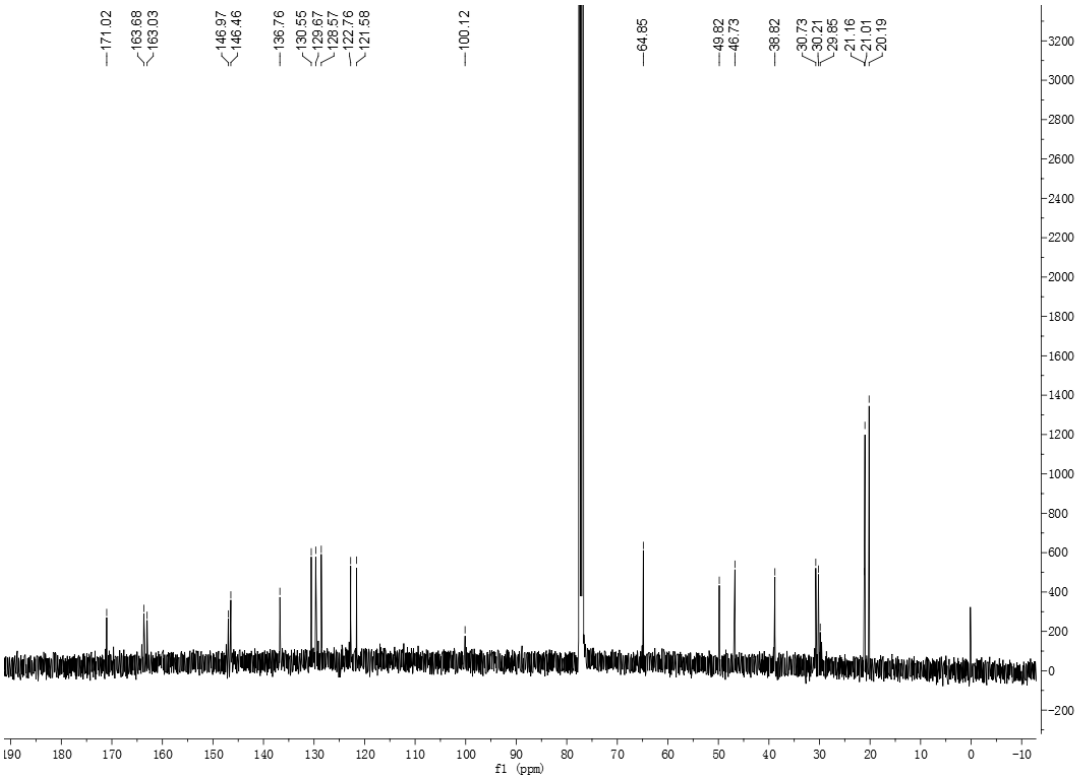
6.00
f1 (ppm)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130.55
129.67
128.57
122.76
121.58
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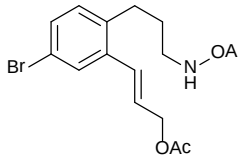
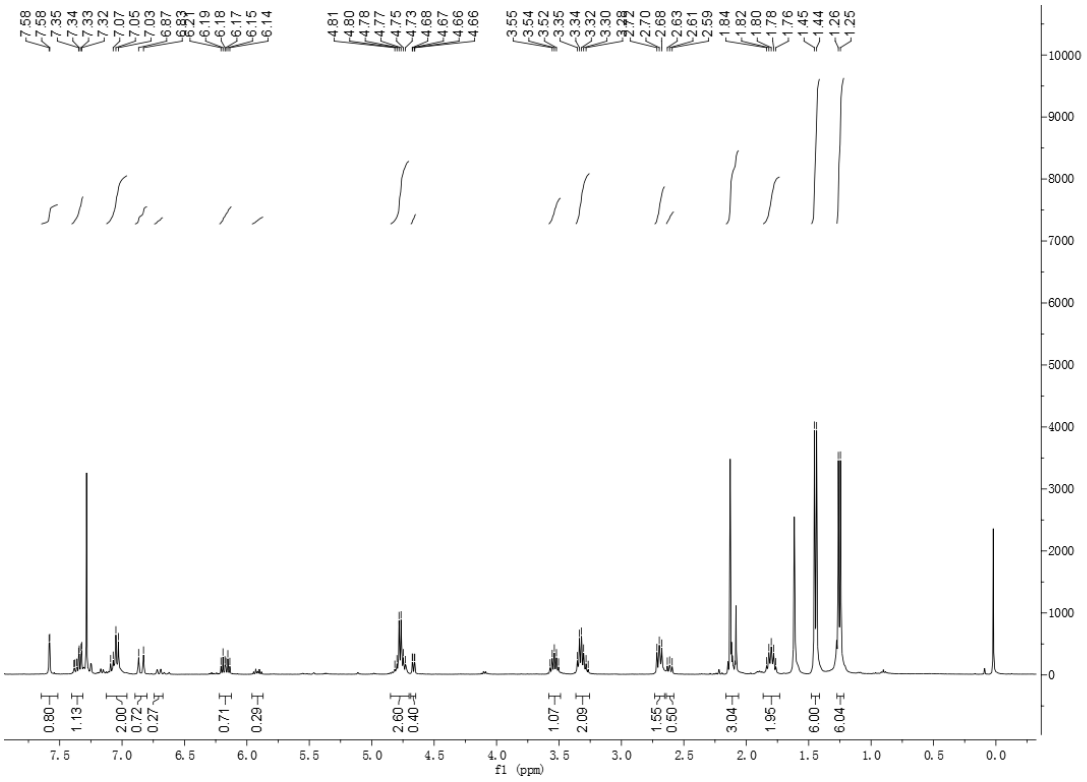
29.85
21.16
21.01
20.19
f1 (ppm)


Br
N
H
OA
OAc

3p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7.33
7.32
7.07
7.05
7.03
6.87
6.83
6.21
6.19
6.18
6.17
6.15
6.14
4.81
4.80
4.78
4.77
4.75
4.73
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4.67
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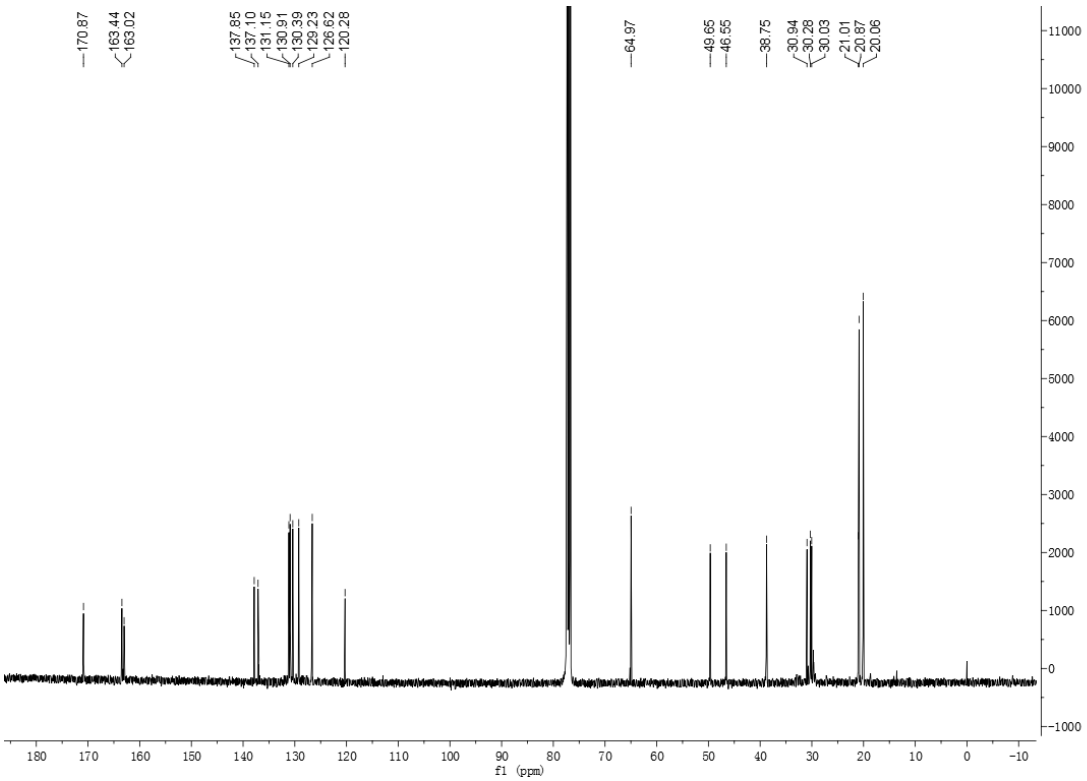
3.54
3.52
3.35
3.34
3.32
3.30
2.70
2.68
2.63
2.61
2.59
1.84
1.82
1.80
1.78
1.76
1.45
1.44
1.26
1.25
0.80
1.13
2.00
0.72
0.27
0.71
0.29
2.60
0.40
1.07
2.09
1.55
0.50
3.04
1.95
6.00
6.04
f1 (ppm)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130.39
129.23
126.62
120.28
64.97
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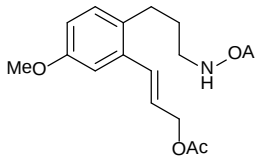
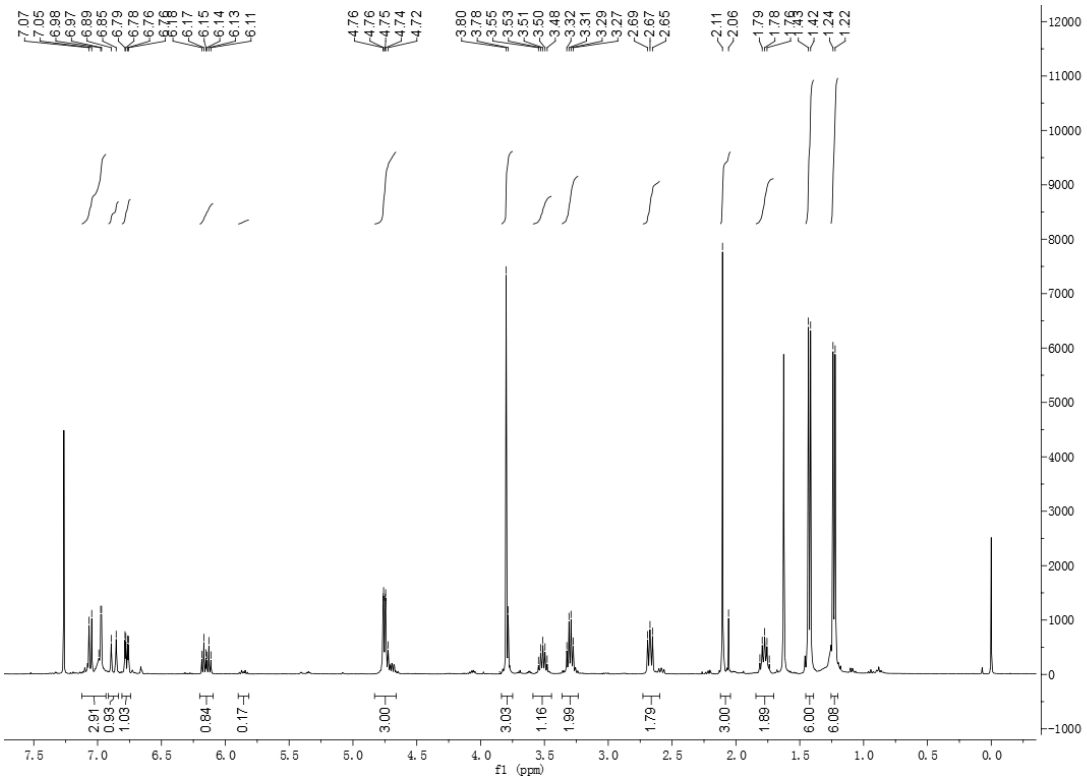
46.55
38.75
30.94
30.28
30.03
21.01
20.87
20.06
f1 (ppm)


MeO
N
H
OA
OAc

3q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7.05
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6.97
6.89
6.85
6.79
6.78
6.76
6.17
6.15
6.14
6.13
6.11
4.76
4.75
4.74
4.72
3.80
3.78
3.55
3.53
3.51
3.50
3.48
3.32
3.31
3.29
3.27
2.69
2.67
2.65
2.11
2.06
1.79
1.78
1.42
1.24
1.22
2.91
0.93
1.03
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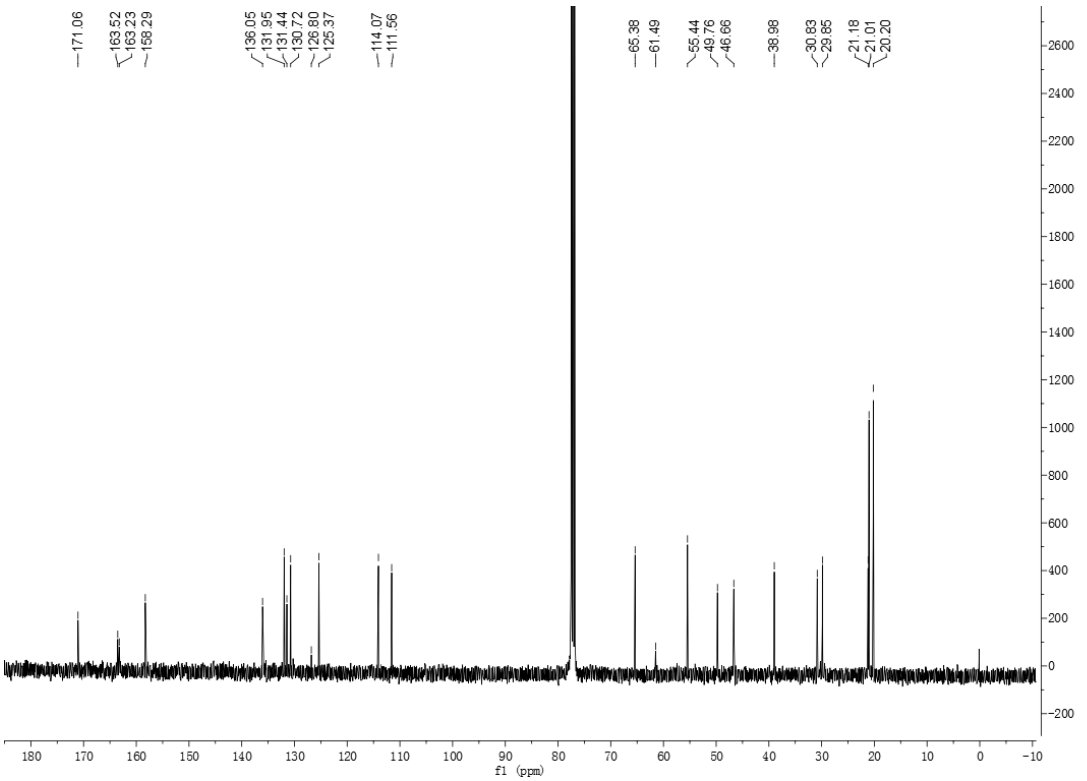
1.99
1.79
3.00
1.89
6.00
6.08
f1 (ppm)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130.72
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114.07
111.56
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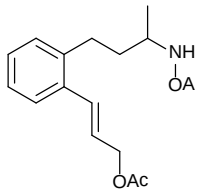
49.76
46.66
38.98
30.83
29.85
21.18
21.01
20.20
f1 (ppm)



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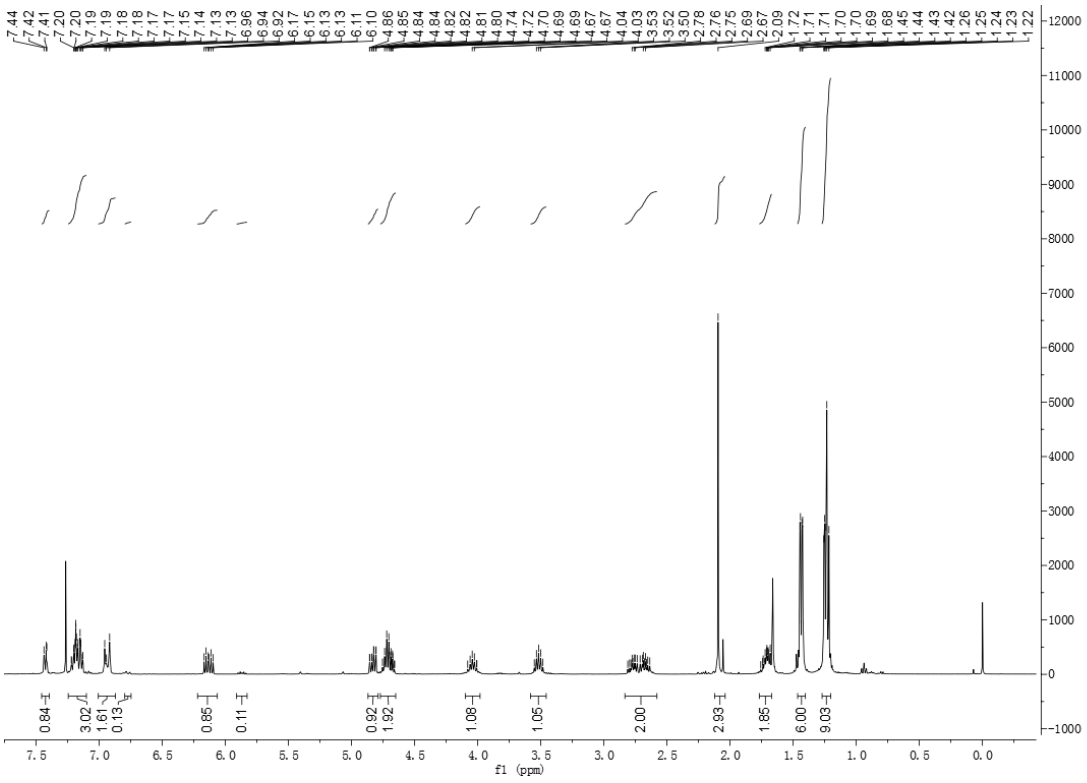

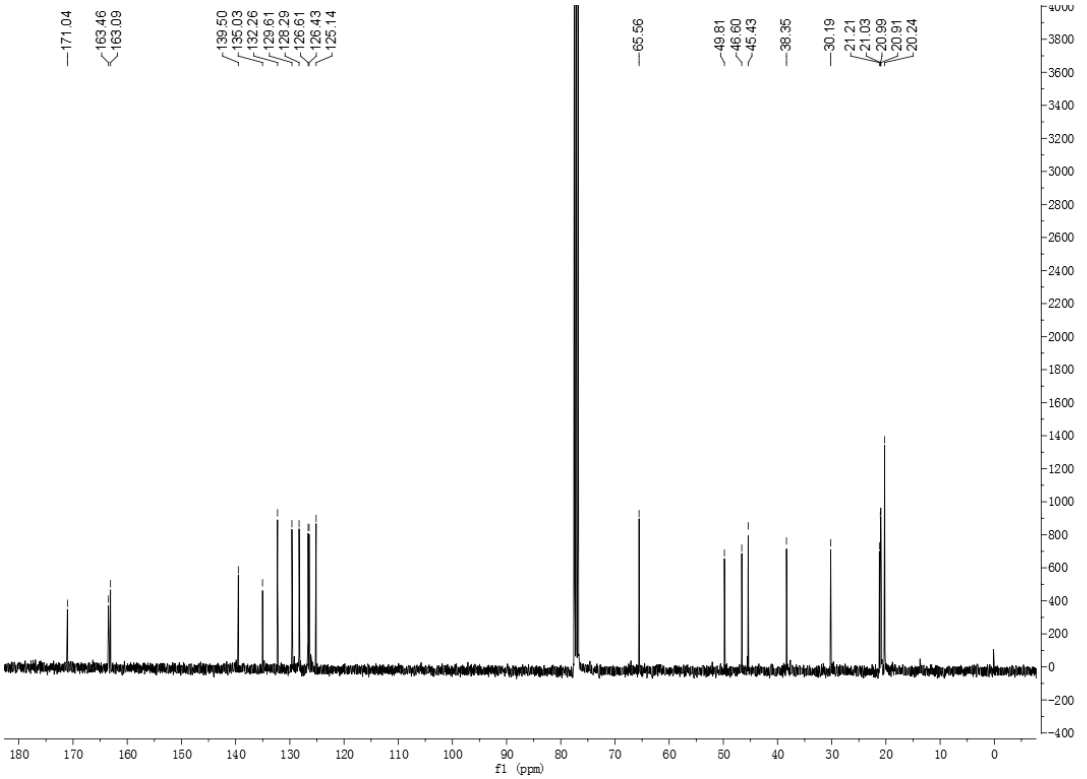

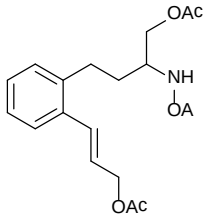

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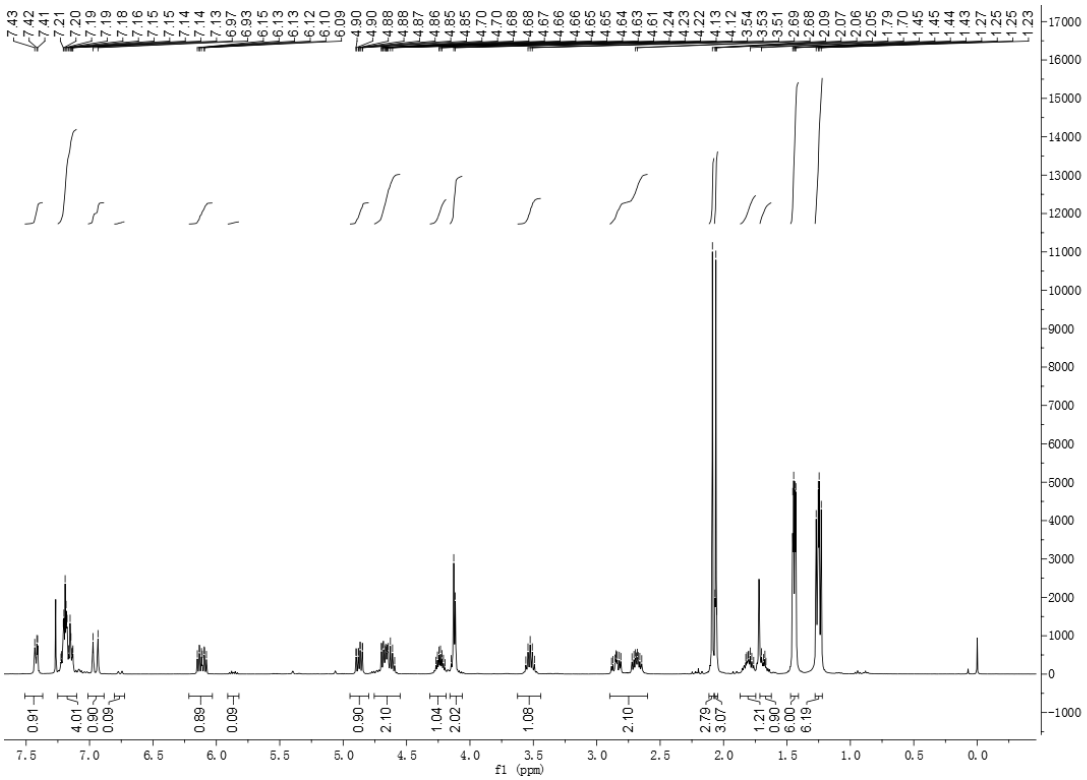
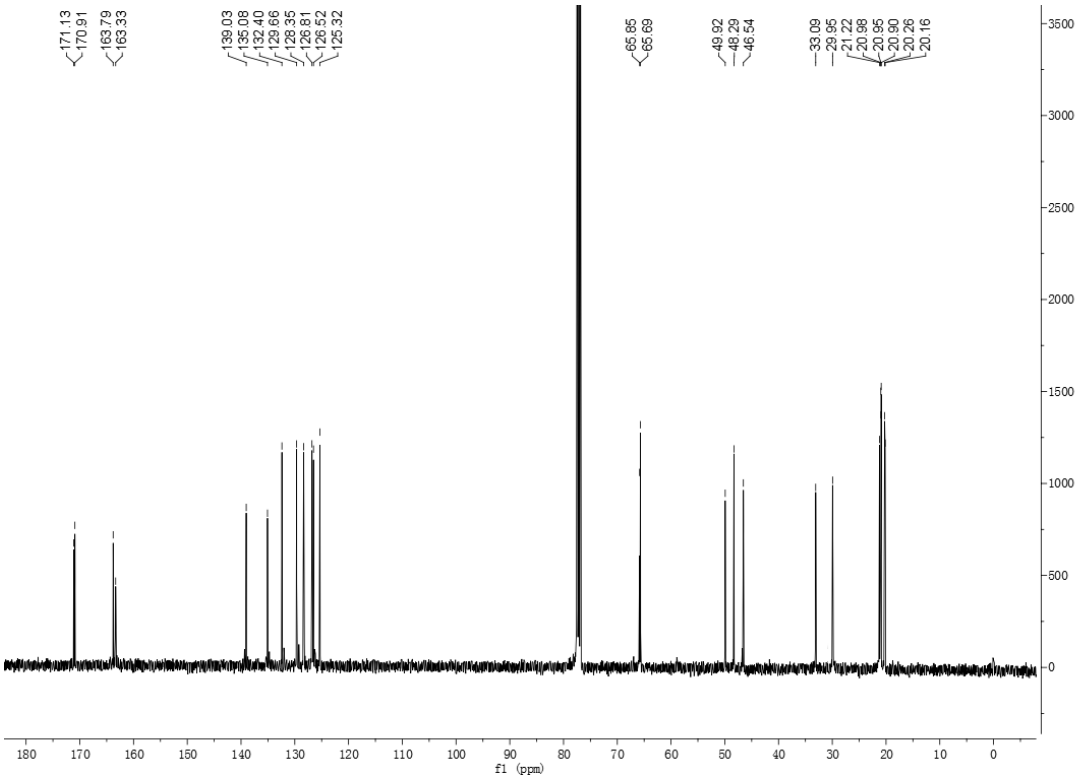
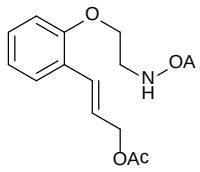
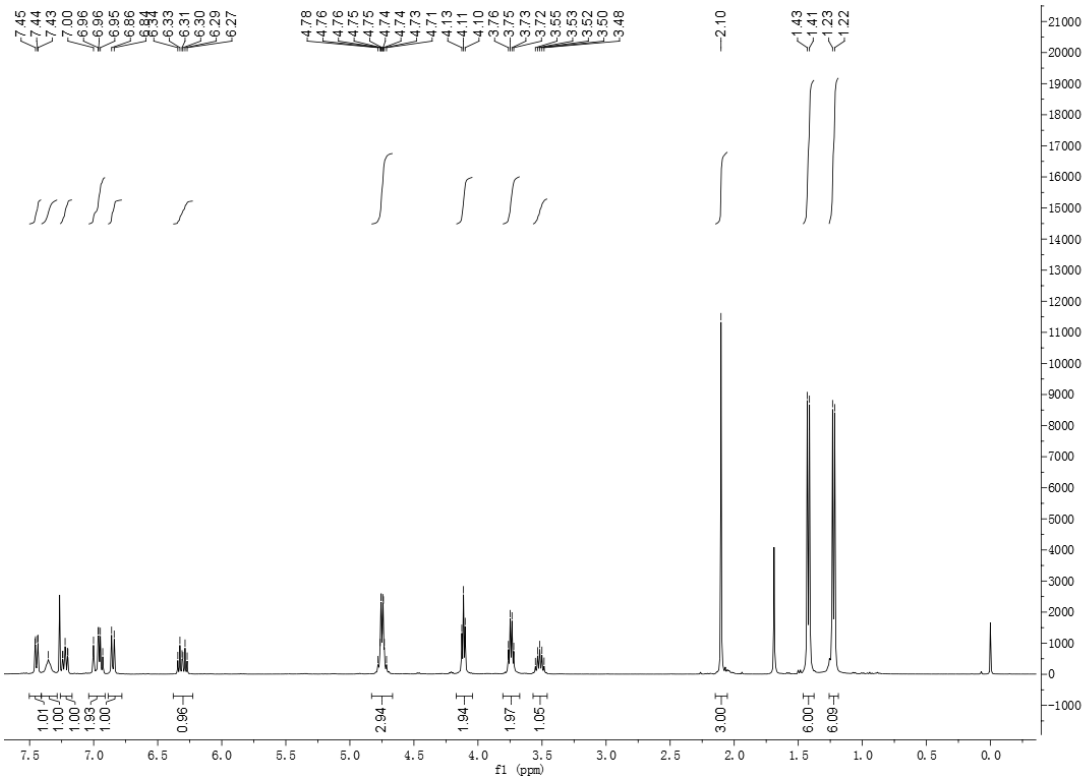





O
N
H
OA
OAc

3t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6.95
6.86
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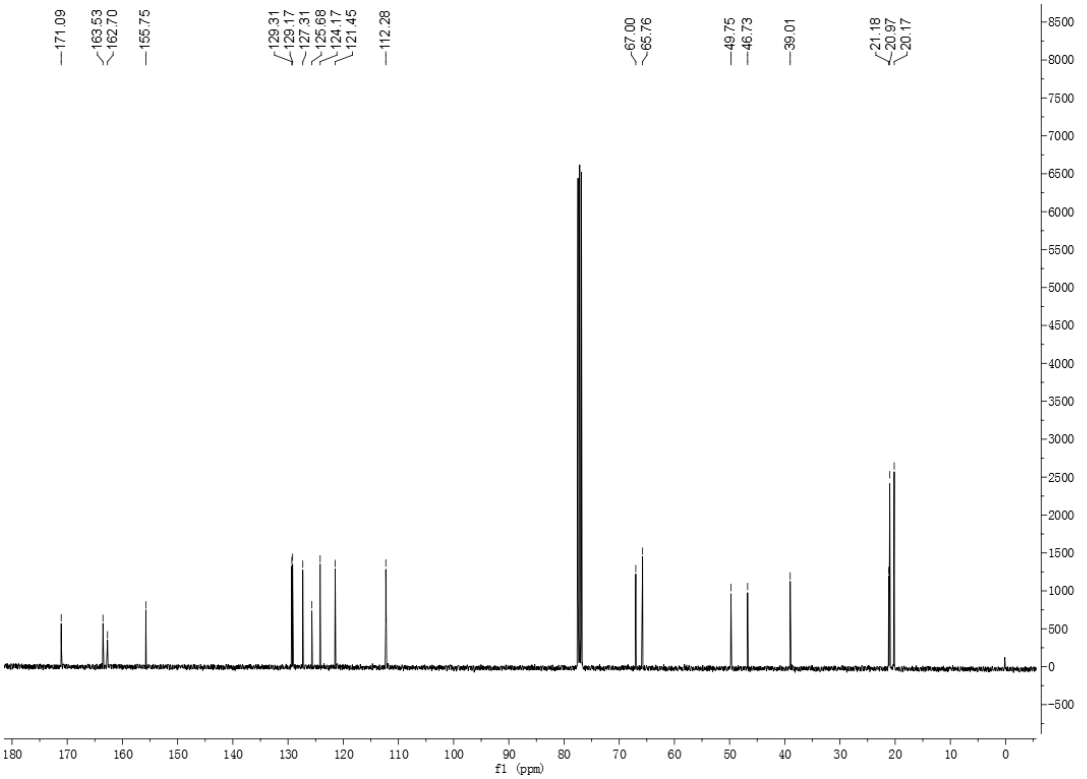
6.31
6.30
6.29
6.27
4.78
4.76
4.76
4.75
4.75
4.74
4.74
4.73
4.71
4.13
4.11
4.10
3.76
3.75
3.73
3.72
3.55
3.53
3.52
3.50
3.48
2.10
1.43
1.41
1.23
1.22
1.01
1.00
1.00
1.93
1.00
0.96
2.94
1.94
1.97
1.05
3.00
6.00
6.09
f1 (ppm)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125.68
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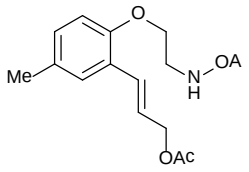
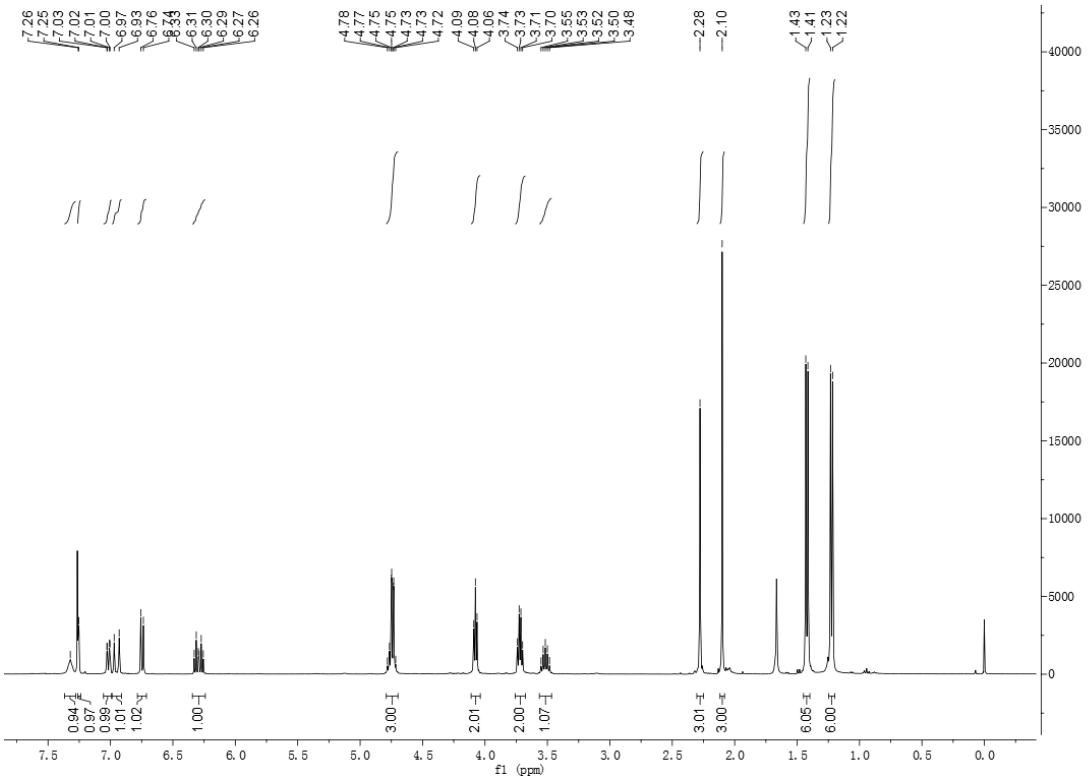
49.75
46.73
39.01
21.18
20.97
20.17
f1 (ppm)


O
Me
N
H
OA
OAc

3u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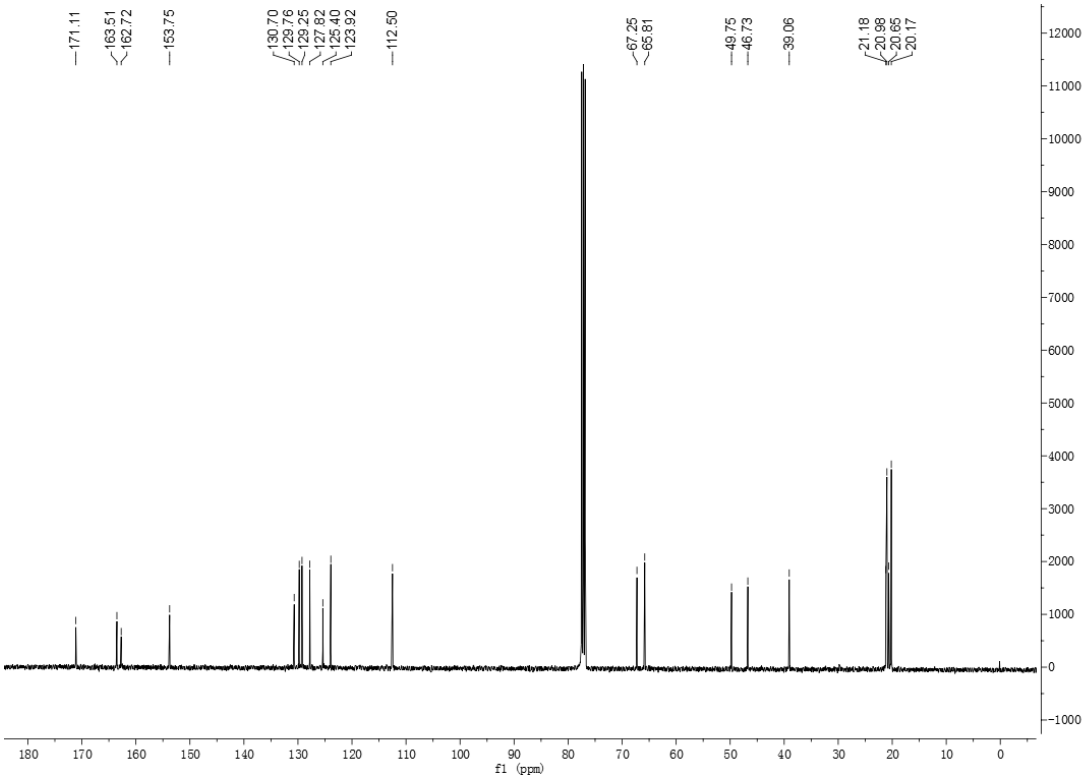
7.02
7.01
7.00
6.97
6.93
6.76
6.74
6.33
6.31
6.30
6.29
6.27
6.26
4.78
4.77
4.75
4.75
4.73
4.73
4.72
4.09
4.08
4.06
3.74
3.73
3.71
3.70
3.55
3.53
3.52
3.50
3.48
2.28
2.10
1.43
1.41
1.23
1.22
0.94
0.97
0.99
1.01
1.02
1.00
3.00
2.01
2.00
1.07
3.01
3.00
6.05
6.00
f1 (ppm)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129.25
127.82
125.40
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112.50
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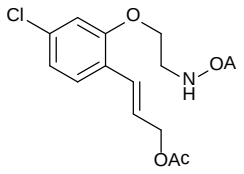
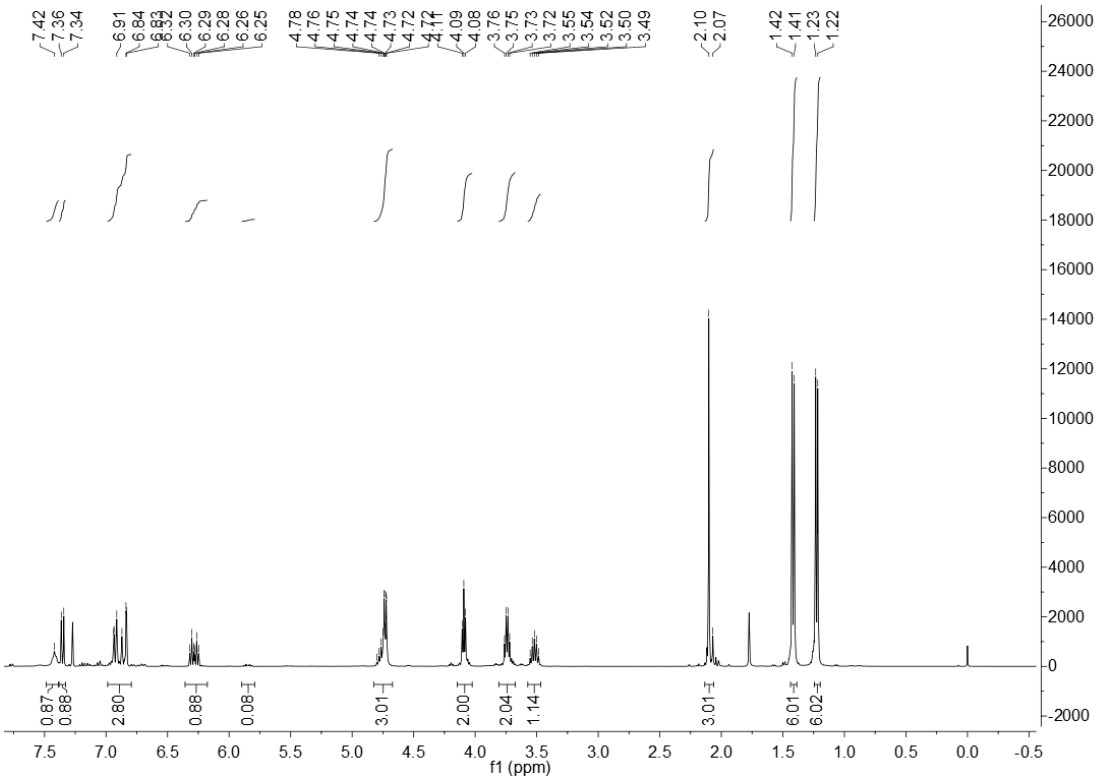
65.81
49.75
46.73
39.06
21.18
20.98
20.65
20.17
f1 (ppm)


Cl
O
N
H
OA
OAc

3v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6.28
6.26
6.25
4.78
4.76
4.75
4.74
4.74
4.73
4.72
4.09
4.08
3.76
3.75
3.73
3.72
3.55
3.54
3.52
3.50
3.49
2.10
2.07
1.42
1.41
1.23
1.22
0.87
0.88
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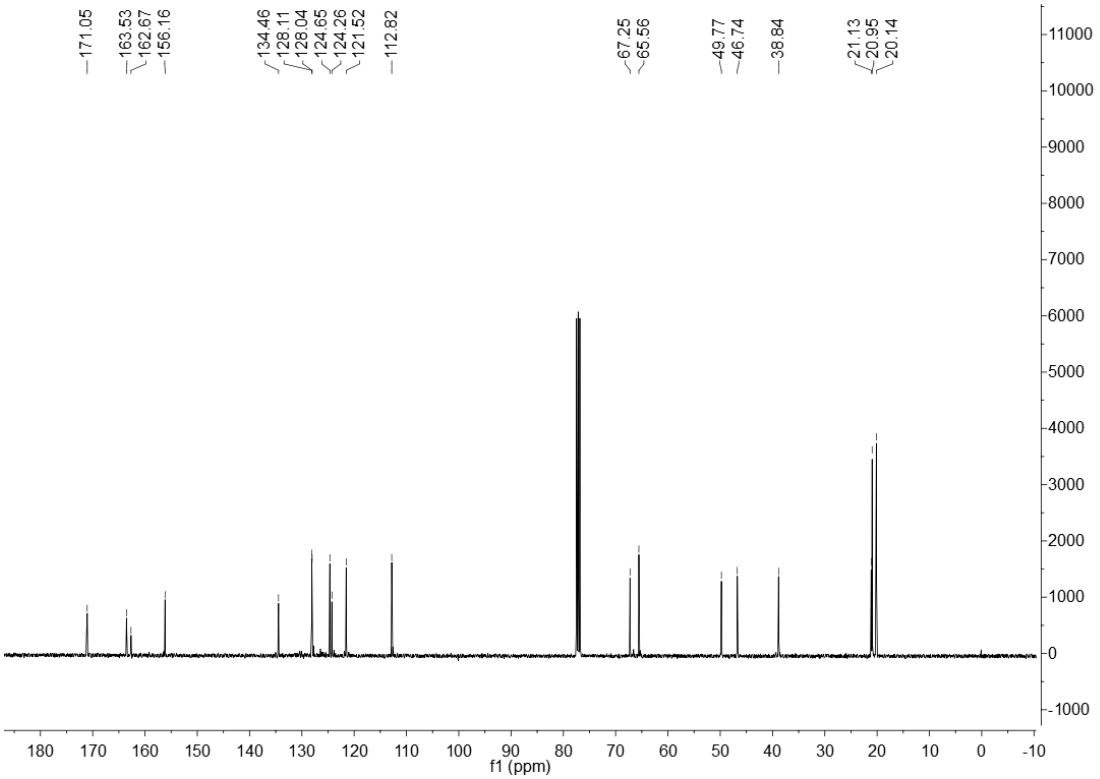
0.88
0.08
3.01
2.00
2.04
1.14
3.01
6.01
6.02
f1 (ppm)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67.25
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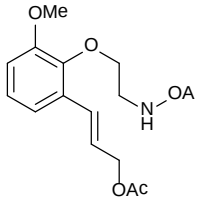
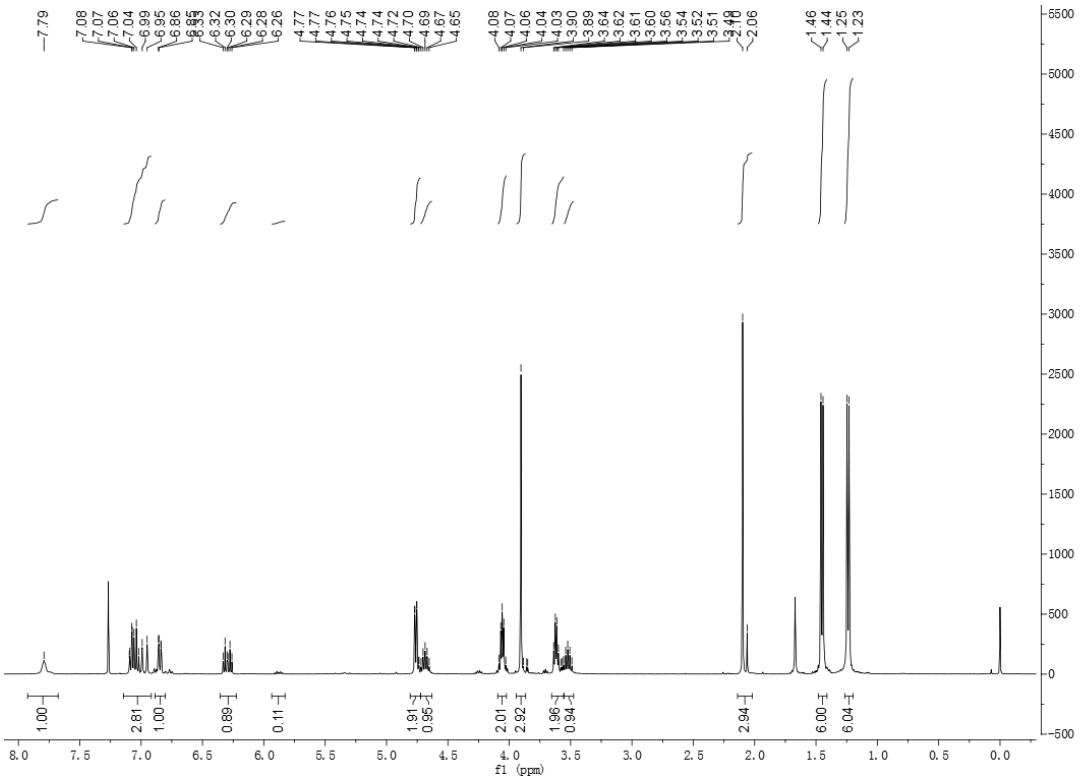
38.84
21.13
20.95
20.14
f1 (ppm)


OMe
O
N
H
OA
OAc

3w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6.28
6.26
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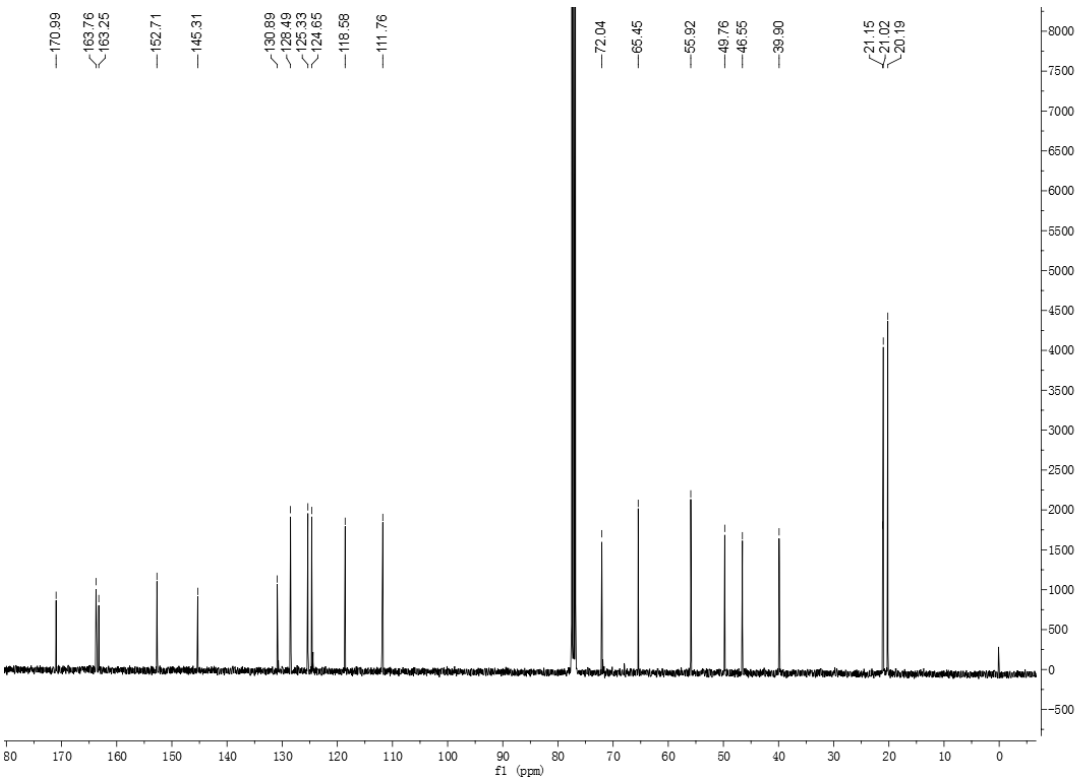
4.76
4.75
4.74
4.72
4.70
4.69
4.67
4.65
4.08
4.07
4.06
4.04
4.03
3.90
3.89
3.64
3.62
3.61
3.60
3.56
3.54
3.52
3.51
2.06
1.46
1.44
1.25
1.23
1.00
2.81
1.00
0.89
0.11
1.91
0.95
2.01
2.92
1.96
0.94
2.94
6.00
6.04
f1 (ppm)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124.65
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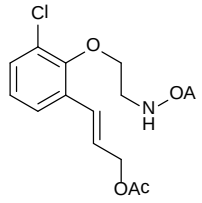
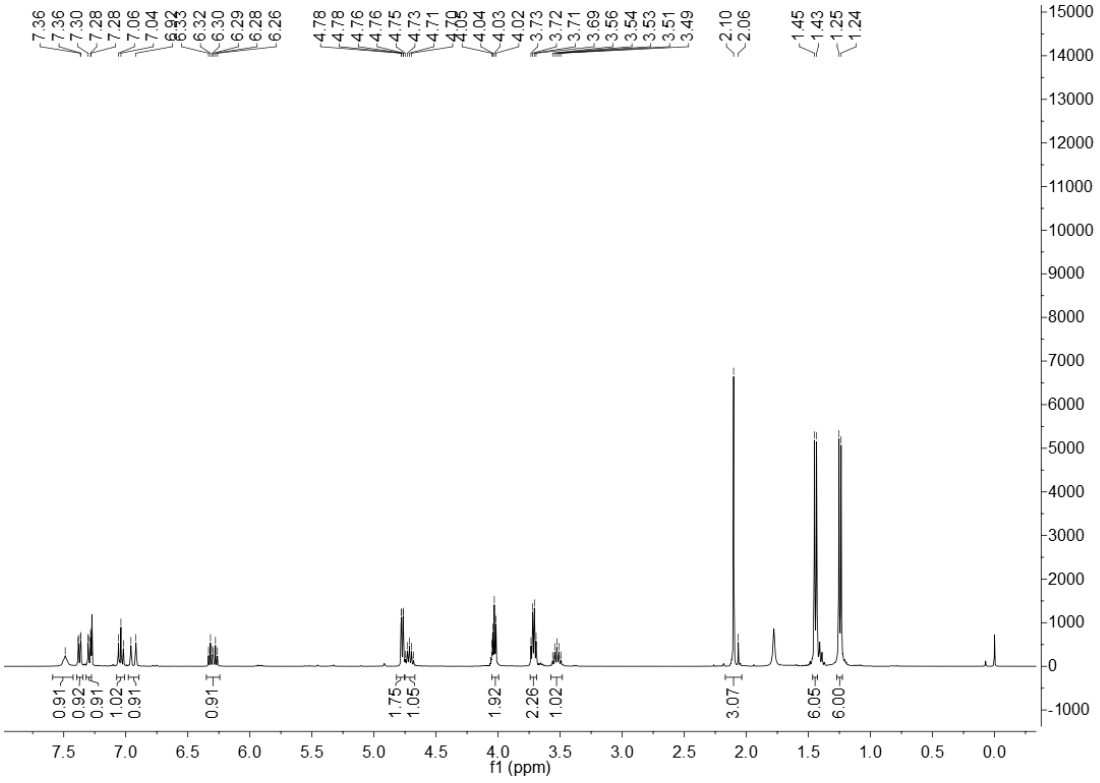
55.92
49.76
46.55
39.90
21.15
21.02
20.19
f1 (ppm)


Cl
O
N
H
OA
OAc

3x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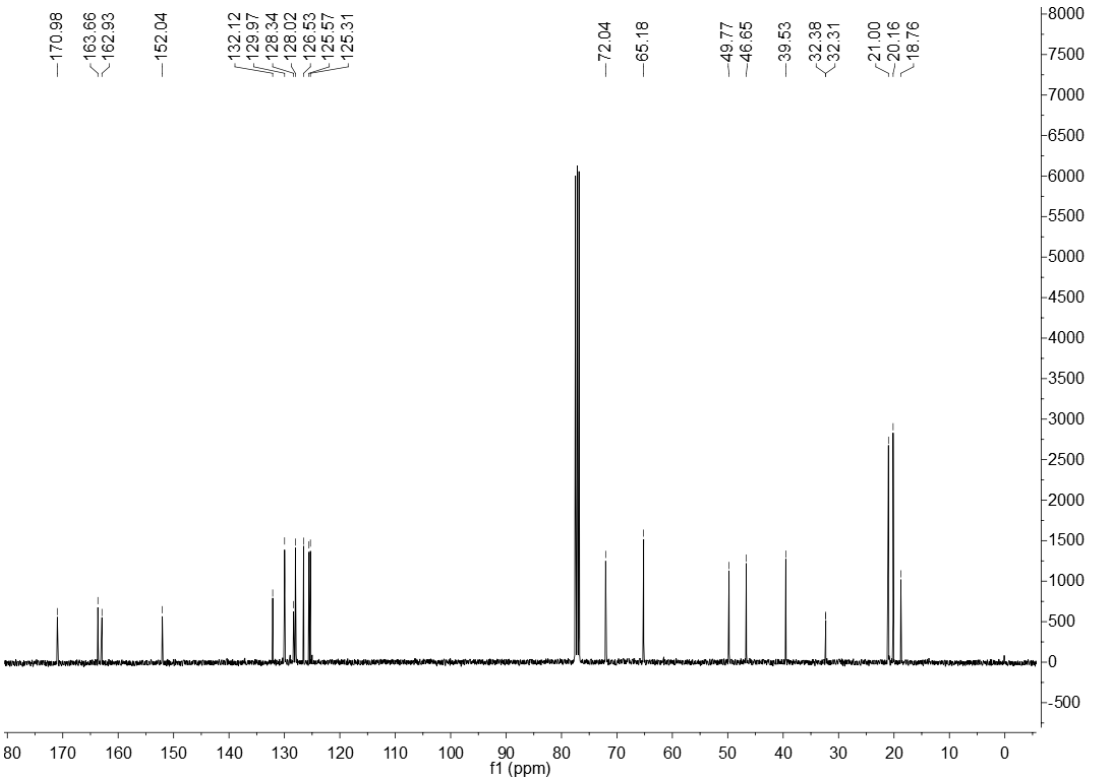
6.29
6.28
6.26
4.78
4.78
4.76
4.76
4.75
4.73
4.71
4.04
4.03
4.02
3.73
3.72
3.71
3.69
3.56
3.54
3.53
3.51
3.49
2.10
2.06
1.45
1.43
1.25
1.24
0.91
0.92
0.91
1.02
0.91
0.91
1.75
1.05
1.92
2.26
1.02
3.07
6.05
6.00
f1 (ppm)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128.34
128.02
126.53
125.57
125.31
72.04
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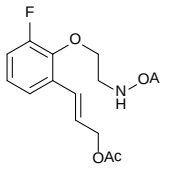
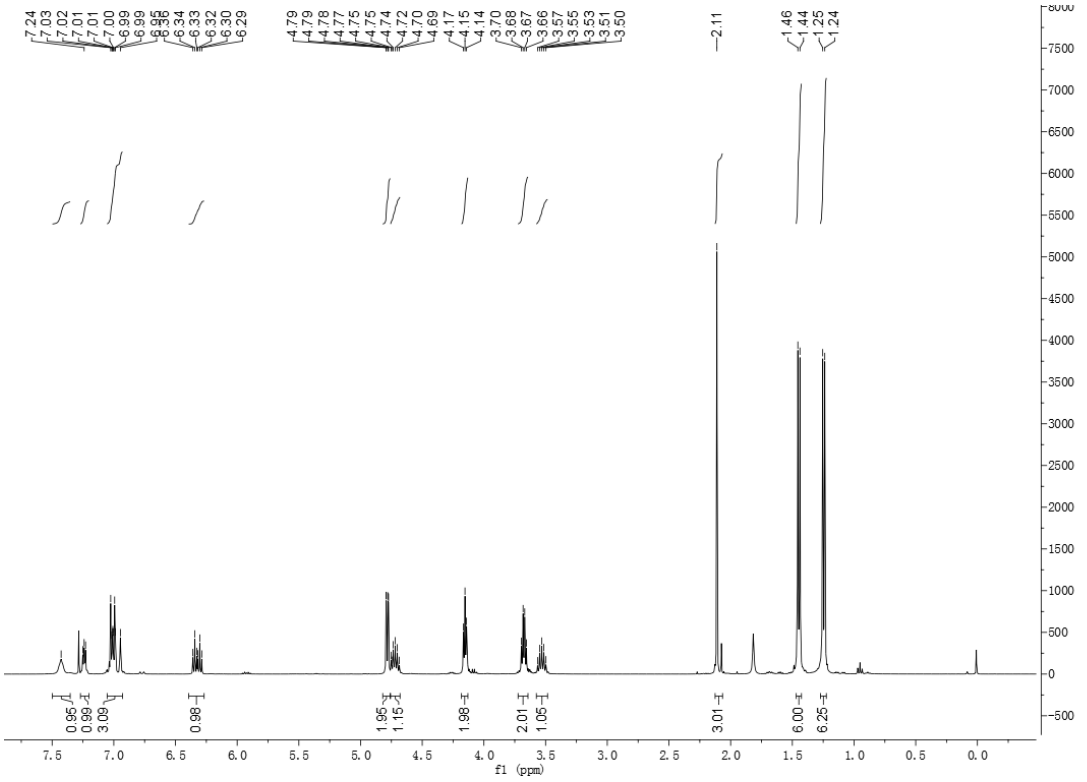
32.31
21.00
20.16
18.76
f1 (ppm)


F
O
N
H
OA
OAc

3y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7.00
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6.99
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6.32
6.30
6.29
4.79
4.79
4.78
4.77
4.75
4.75
4.74
4.72
4.70
4.69
4.17
4.15
4.14
3.70
3.68
3.67
3.66
3.57
3.55
3.53
3.51
3.50
2.11
1.46
1.44
1.25
1.24
0.95
0.99
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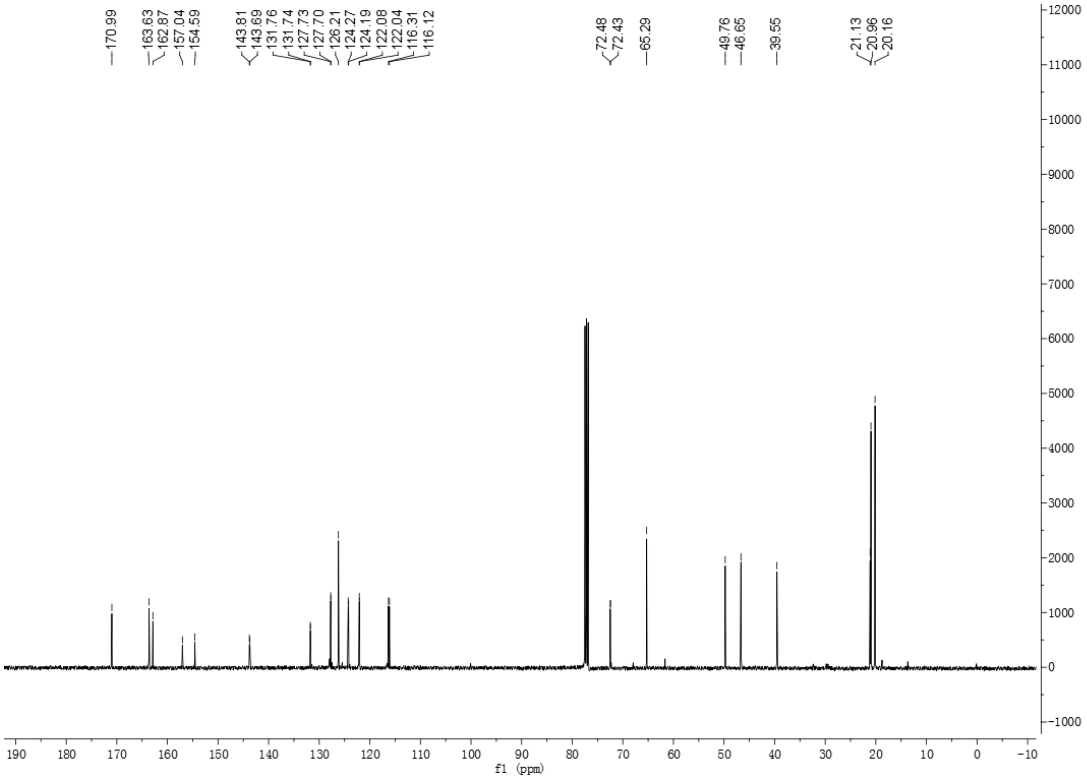
1.95
1.15
1.98
2.01
1.05
3.01
6.00
6.25
f1 (ppm)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143.69
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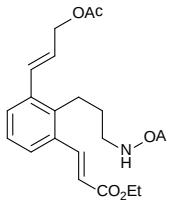
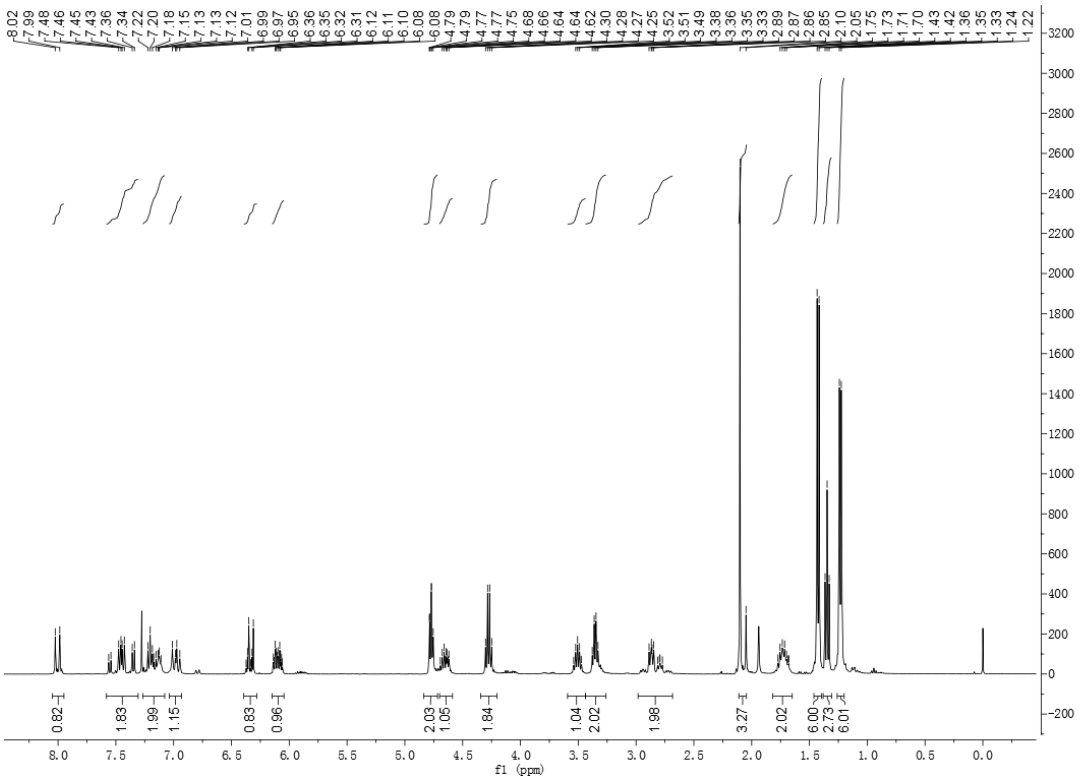
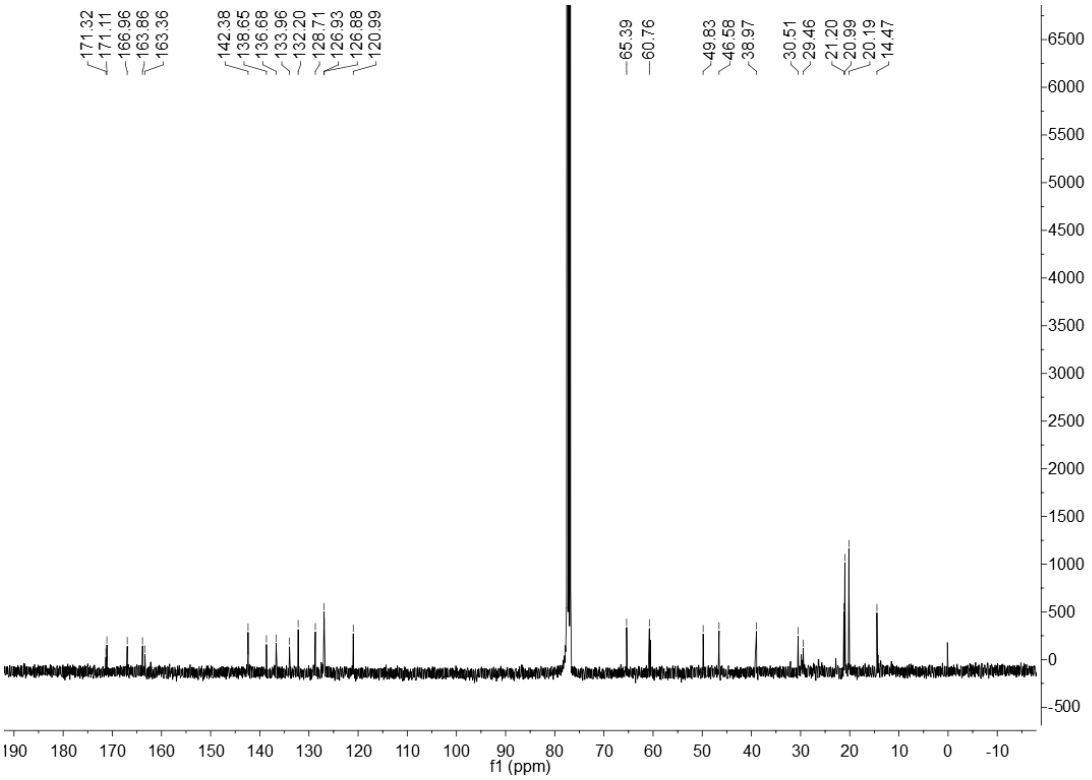
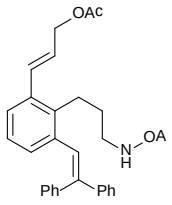
131.74
127.73
127.70
126.21
124.27
124.19
122.08
122.04
116.31
116.12
72.48
72.43
65.29
49.76
46.65
39.55
21.13
20.96
20.16
f1 (ppm)



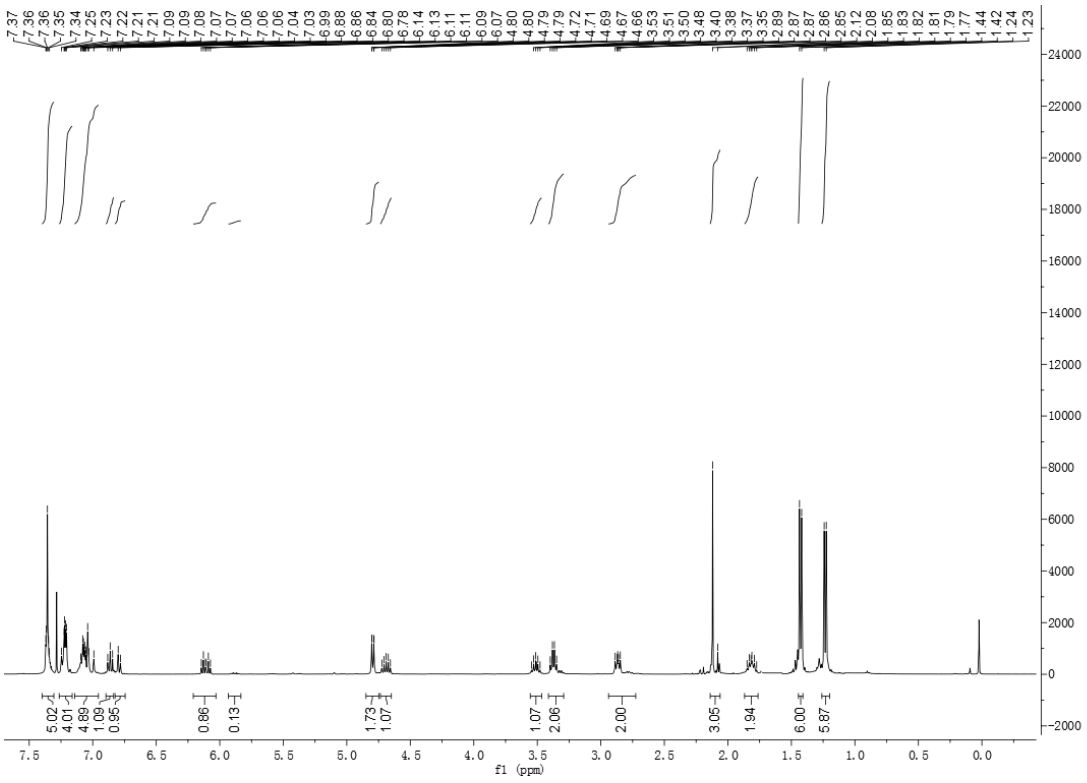
4a






OAc
N
H
OA
Ph
Ph

4b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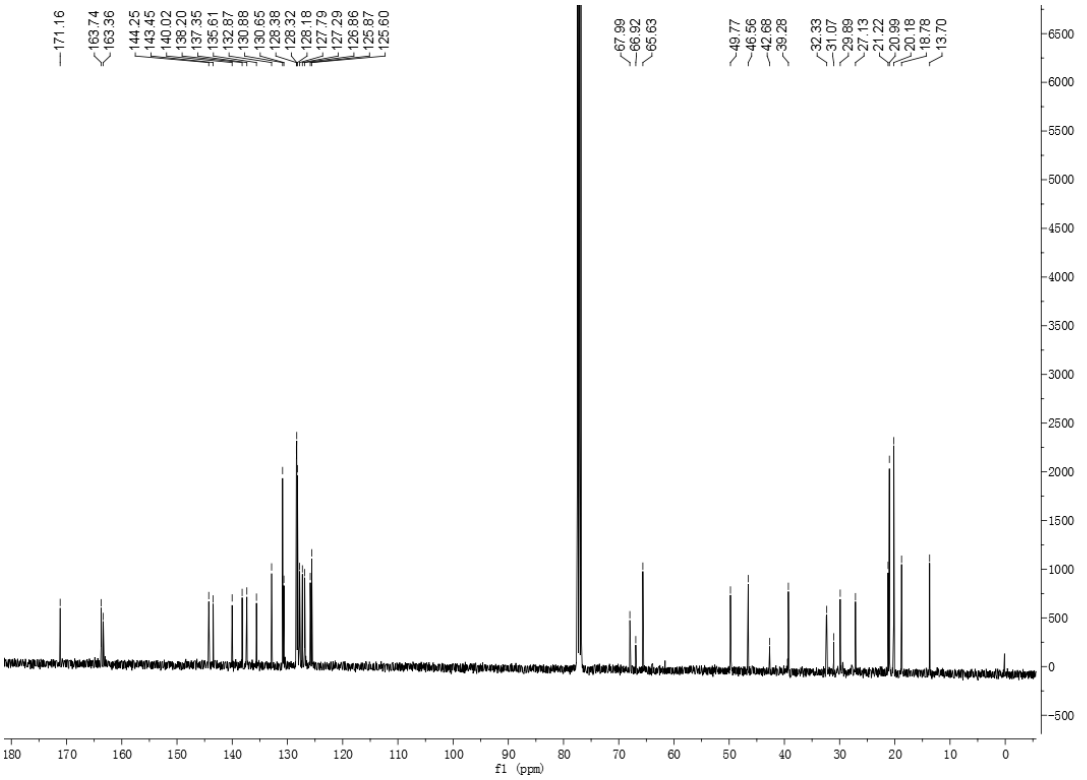
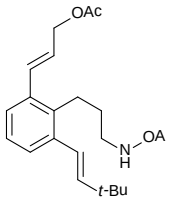
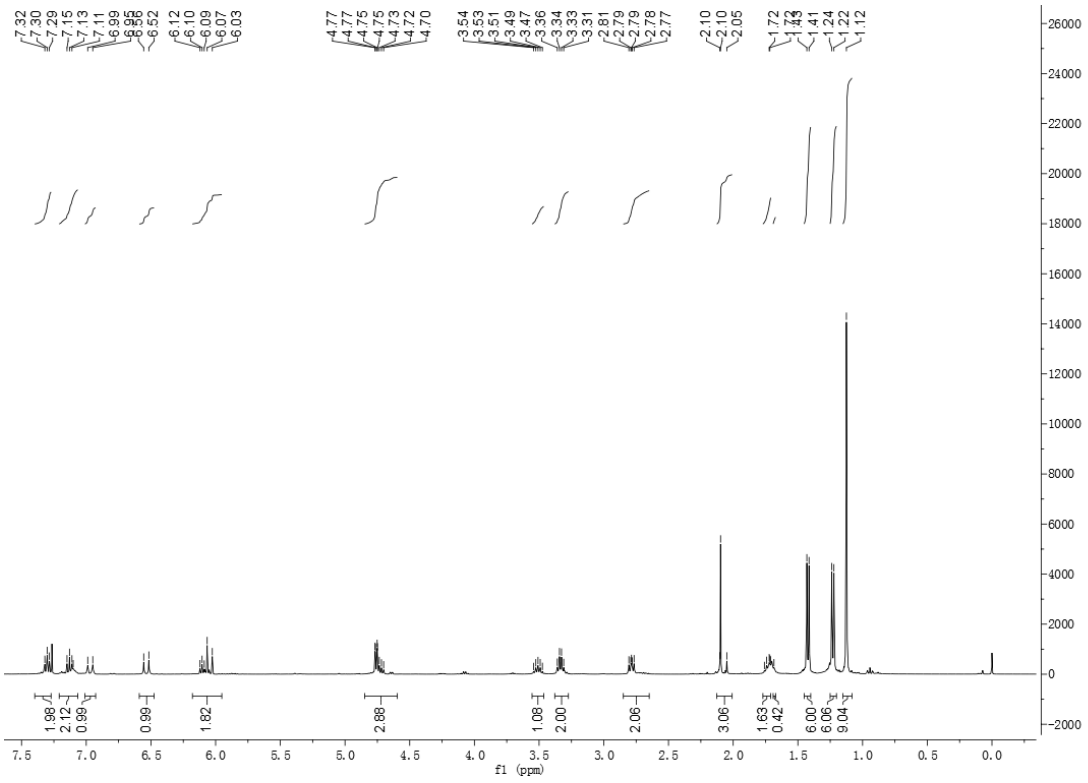
5.02 4.01 4.89 1.09 0.95 0.86 0.13 1.73 1.07 1.07 2.06 2.00 3.05 1.94 6.00 5.87
f1 (ppm)


171.16 163.74 163.36 144.25 143.45 140.02 138.20 137.35 135.61 132.87 130.88 130.65 128.38 128.32 128.18 127.79 127.29 126.86 125.87 125.60 67.99 66.92 65.63 49.77 46.56 42.68 39.28 32.33 31.07 29.89 27.13 21.22 20.99 20.18 18.78 13.70
f1 (ppm)


OAc
N
H
OA
t-Bu

4c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7.11
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6.52
6.12
6.10
6.09
6.07
6.03
4.77
4.75
4.73
4.72
4.70
3.54
3.53
3.51
3.49
3.47
3.36
3.34
3.33
3.31
2.81
2.79
2.78
2.77
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2.05
1.72
1.41
1.24
1.22
1.12
1.98
2.12
0.99
0.99
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2.00
2.06
3.06
1.63
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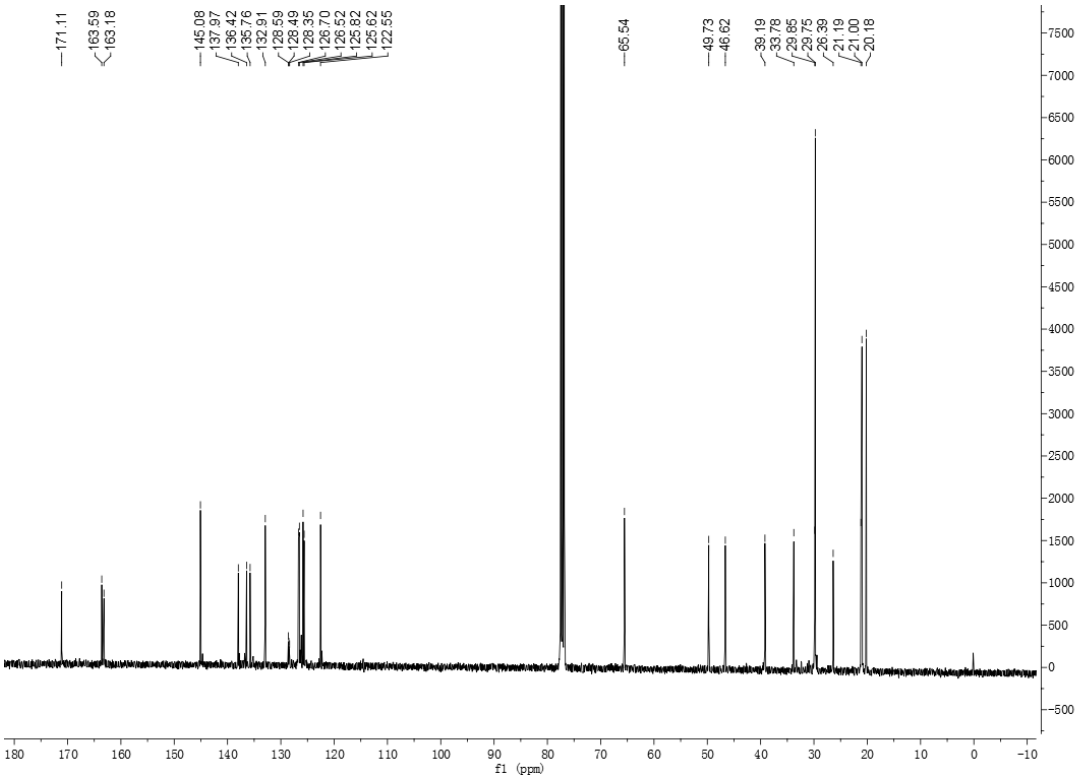
6.00
6.06
9.04
f1 (ppm)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136.42
135.76
132.91
128.59
128.49
128.35
126.70
126.52
125.82
125.62
122.55
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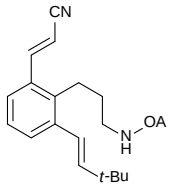
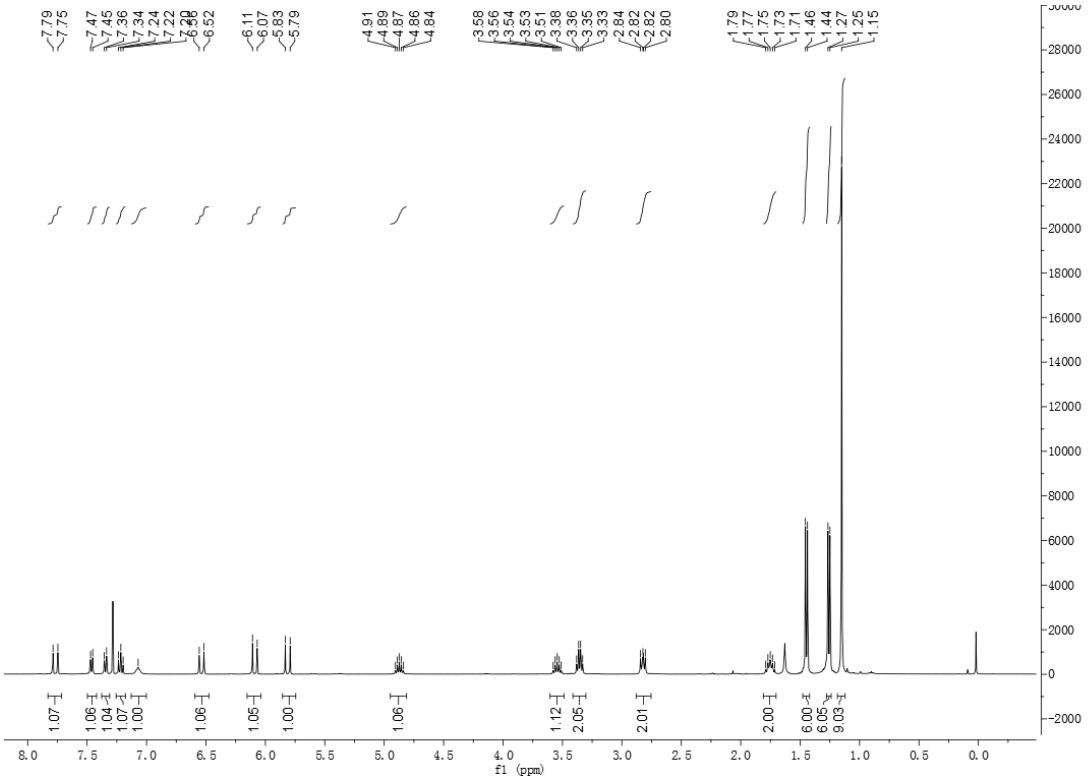
26.39
21.19
21.00
20.18
f1 (ppm)


CN
OA
N
H
t-Bu

4d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7.22
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6.11
6.07
5.83
5.79
4.91
4.89
4.87
4.86
4.84
3.58
3.56
3.54
3.53
3.51
3.38
3.36
3.35
3.33
2.84
2.82
2.80
1.79
1.77
1.75
1.73
1.71
1.46
1.44
1.27
1.25
1.15
1.07
1.06
1.04
1.07
1.00
1.06
1.05
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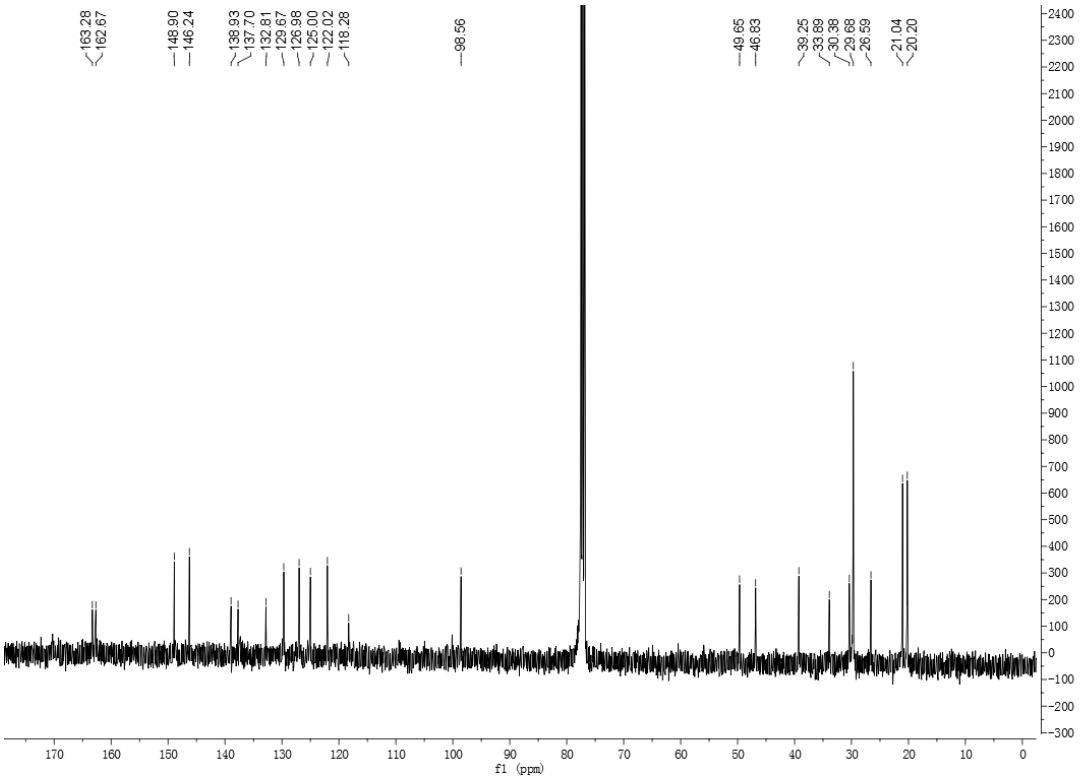
1.06
1.12
2.05
2.01
2.00
6.00
6.05
9.03
f1 (ppm)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132.81
129.67
128.98
125.00
122.02
118.28
98.56
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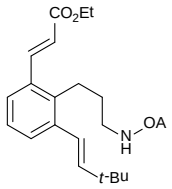
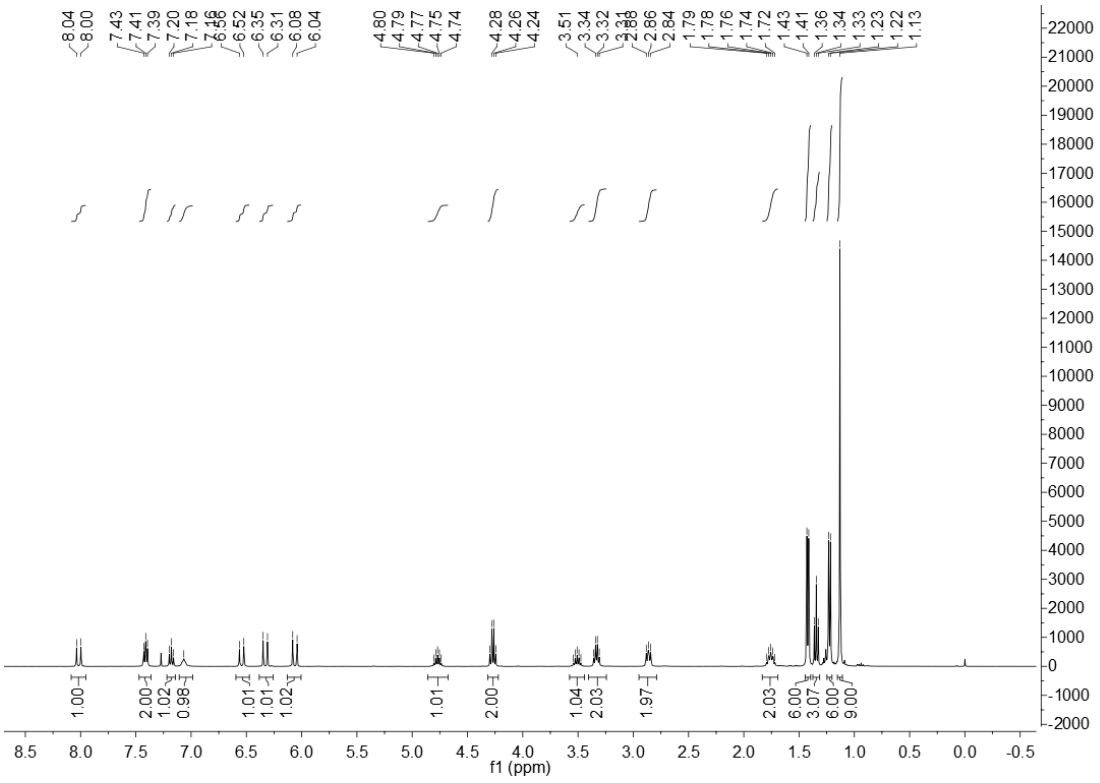
46.83
39.25
33.89
30.38
29.68
26.59
21.04
20.20
f1 (ppm)


CO2Et
N
H
OA
t-Bu

4e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7.16
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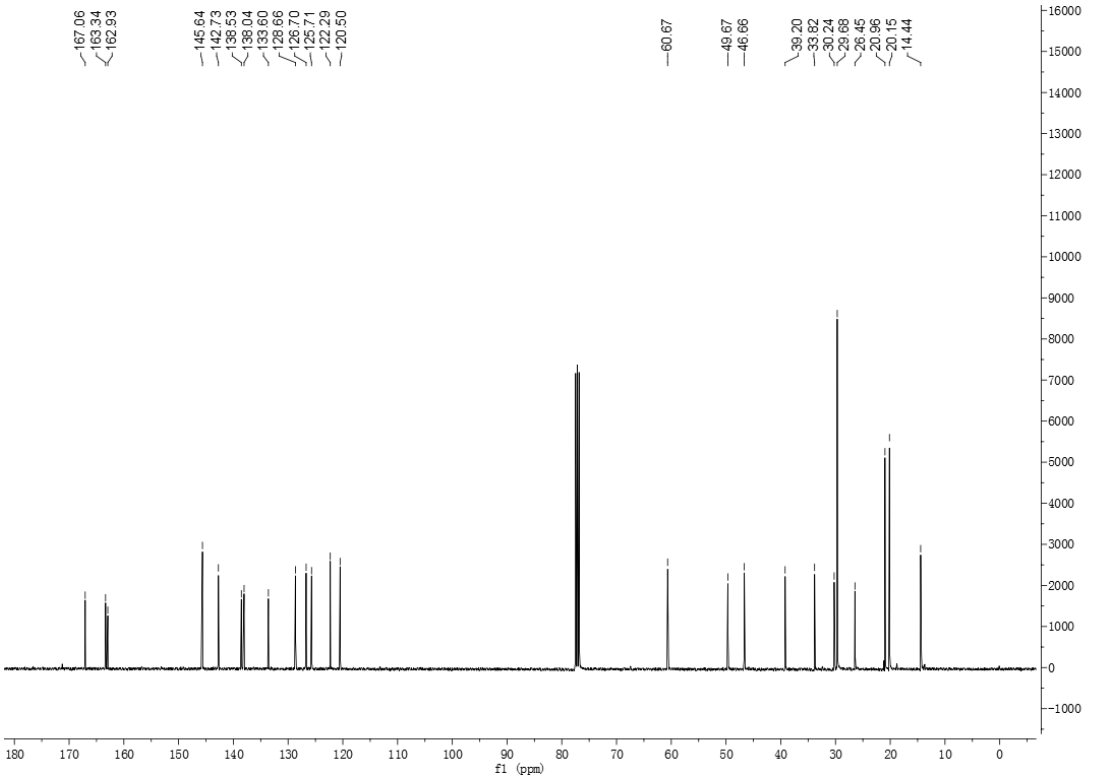
6.52
6.35
6.31
6.08
6.04
4.80
4.79
4.77
4.75
4.74
4.28
4.26
4.24
3.51
3.34
3.32
2.88
2.86
2.84
1.79
1.78
1.76
1.74
1.72
1.43
1.41
1.36
1.34
1.33
1.23
1.22
1.13
1.00
2.00
1.02
0.98
1.01
1.01
1.02
1.01
2.00
1.04
2.03
1.97
2.03
6.00
3.07
6.00
9.00
f1 (ppm)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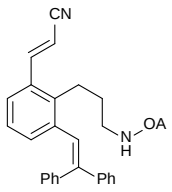
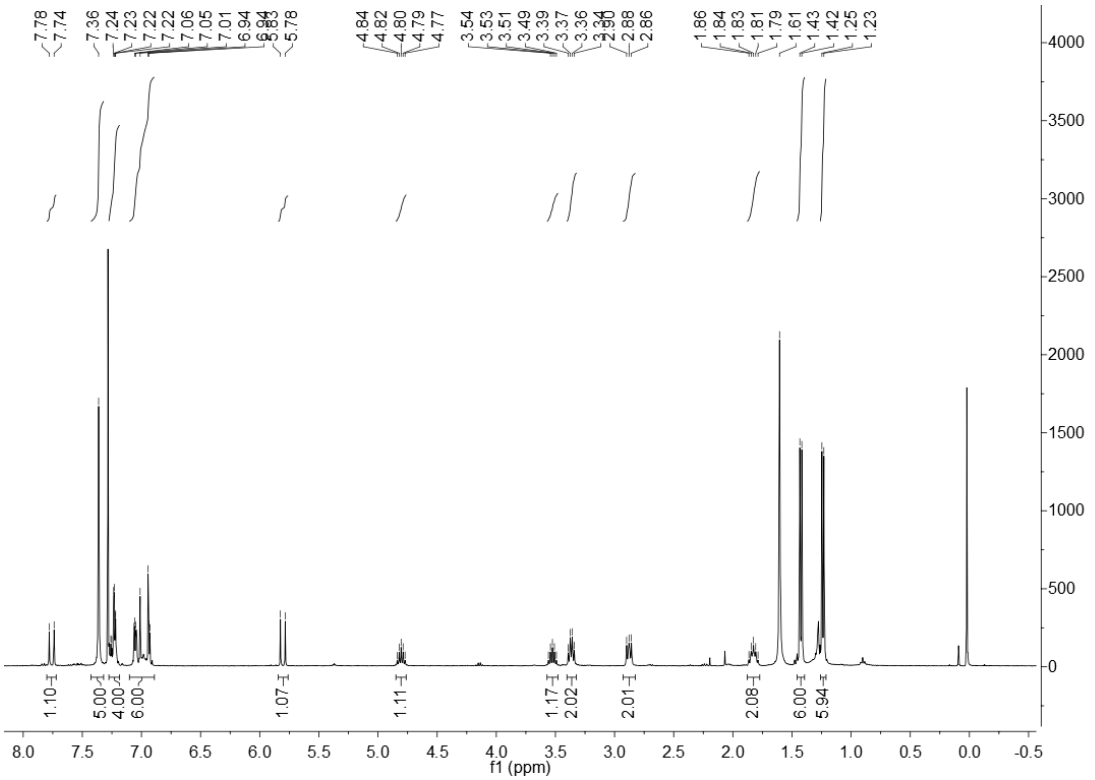
142.73
138.53
138.04
133.60
128.66
126.70
125.71
122.29
120.50
60.67
49.67
46.66
39.20
33.82
30.24
29.68
26.45
20.96
20.15
14.44
f1 (ppm)


CN
N
H
OA
Ph
Ph

4f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7.22
7.06
7.05
7.01
6.94
5.83
5.78
4.84
4.82
4.80
4.79
4.77
3.54
3.53
3.51
3.49
3.39
3.37
3.36
2.88
2.86
1.86
1.84
1.83
1.81
1.79
1.61
1.43
1.42
1.25
1.23
1.10
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4.00
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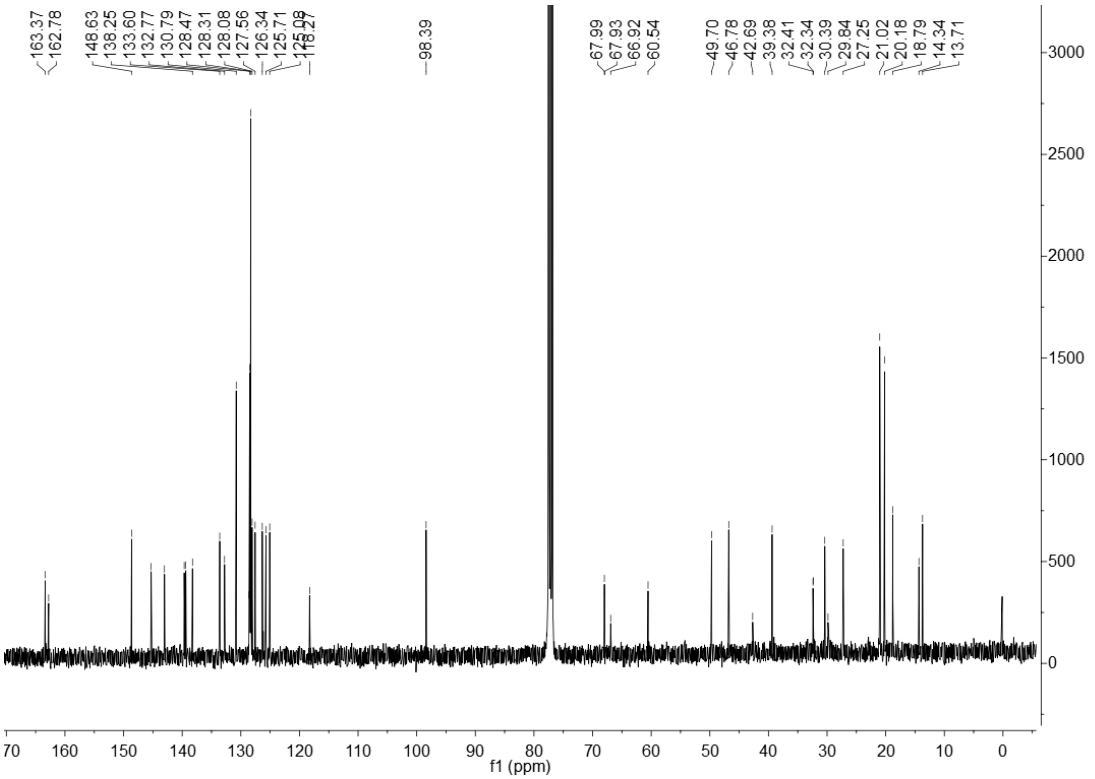
1.07
1.11
1.17
2.02
2.01
2.08
6.00
5.94
f1 (ppm)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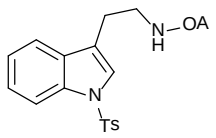
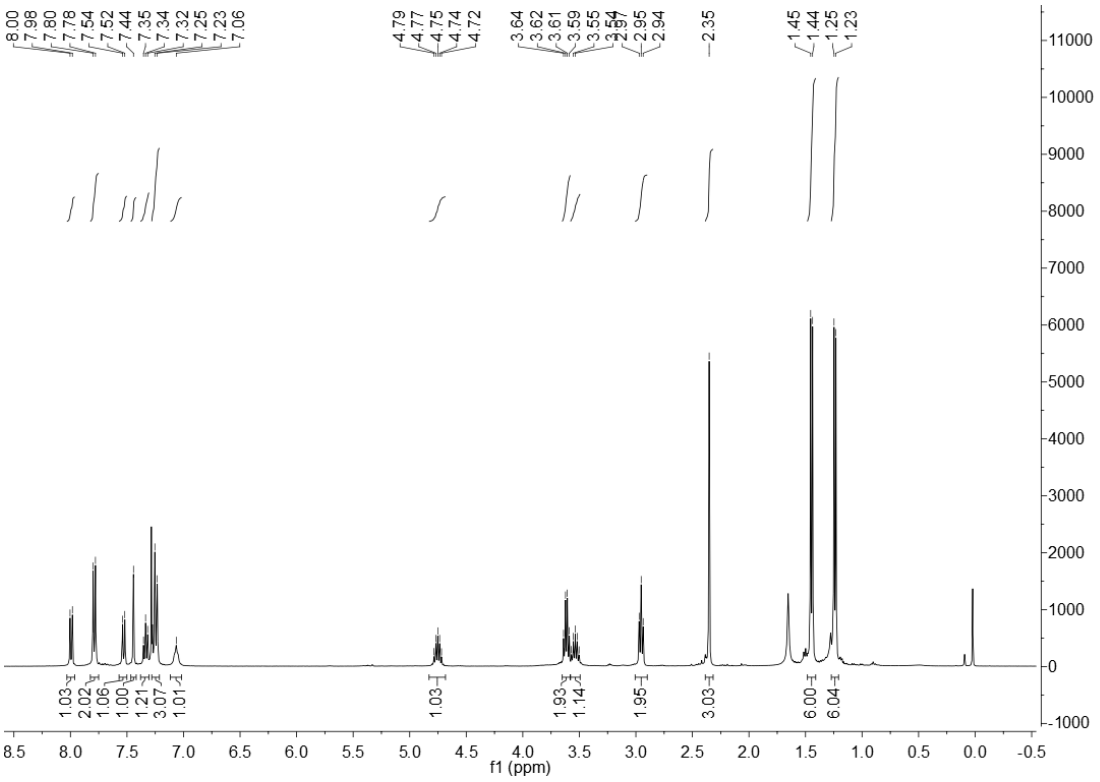
132.77
130.79
128.47
128.31
128.08
127.56
126.34
125.71
98.39
67.99
67.93
66.92
60.54
49.70
46.78
42.69
39.38
32.41
32.34
30.39
29.84
27.25
21.02
20.18
18.79
14.34
13.71
f1 (ppm)


N
OA
H
N
Ts

5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7.80
7.78
7.54
7.52
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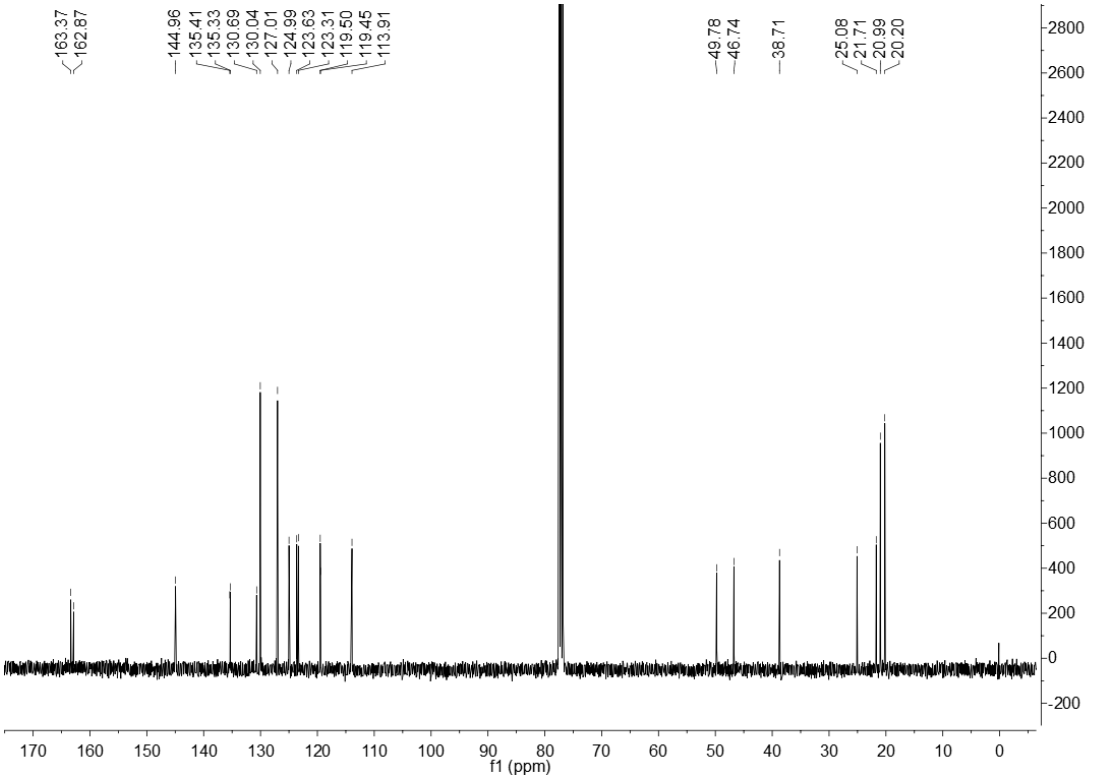
7.35
7.34
7.32
7.25
7.23
7.06
4.79
4.77
4.75
4.74
4.72
3.64
3.62
3.61
3.59
3.55
2.97
2.95
2.94
2.35
1.45
1.44
1.25
1.23
1.03
2.02
1.06
1.00
1.21
3.07
1.01
1.03
1.93
1.14
1.95
3.03
6.00
6.04
f1 (ppm)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130.69
130.04
127.01
124.99
123.63
123.31
119.50
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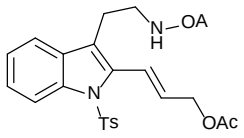
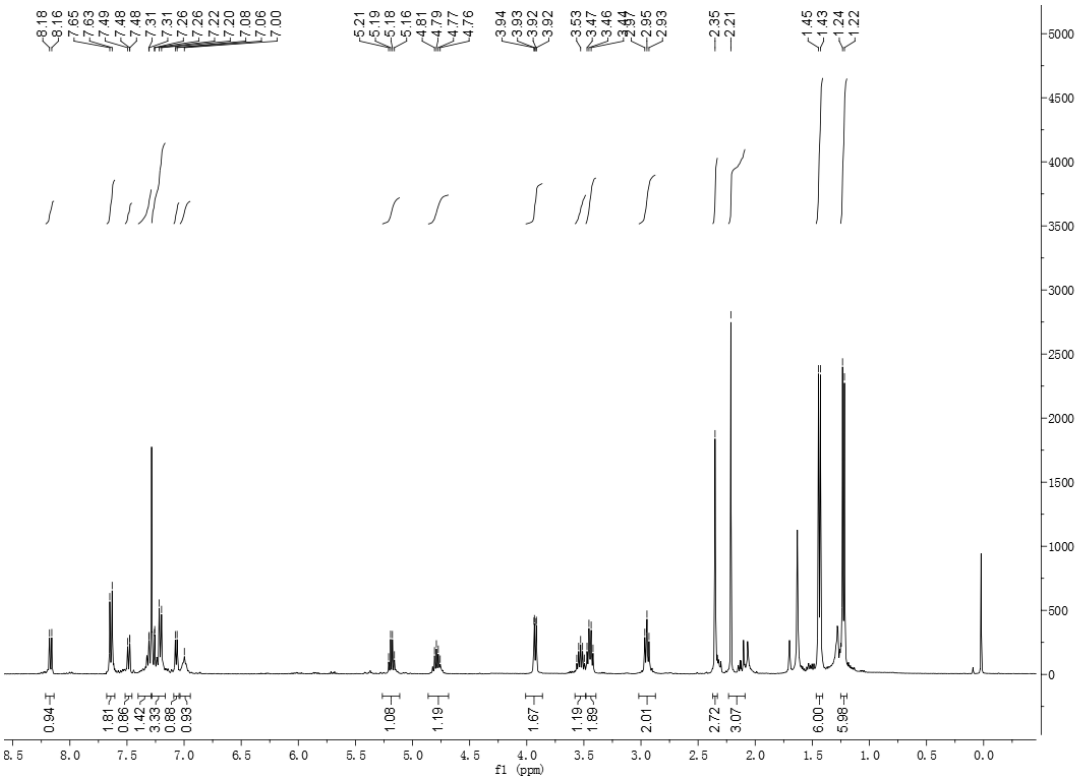
38.71
25.08
21.71
20.99
20.20
f1 (ppm)


N
H
OA
N
Ts
OAc

6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8.16
7.65
7.63
7.49
7.48
7.48
7.31
7.31
7.26
7.26
7.22
7.20
7.08
7.06
7.00
5.21
5.19
5.18
5.16
4.81
4.79
4.77
4.76
3.94
3.93
3.92
3.92
3.53
3.47
3.46
2.95
2.93
2.35
2.21
1.45
1.43
1.24
1.22
0.94
1.81
0.86
1.42
3.33
0.88
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1.08
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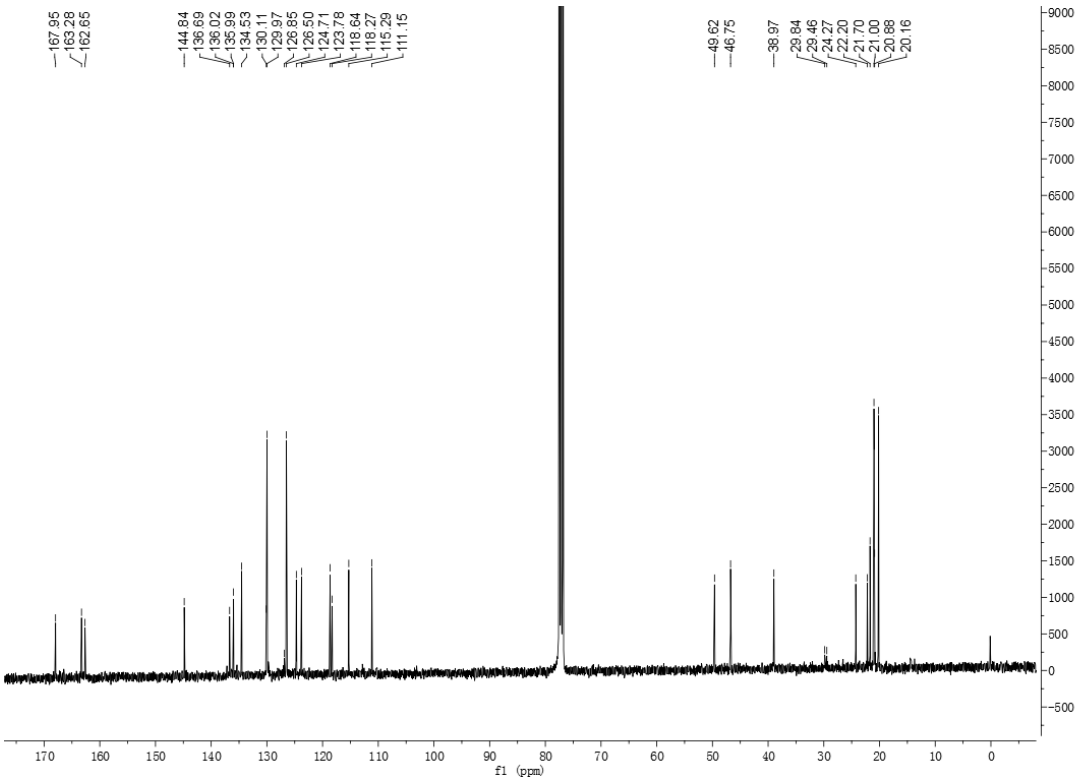
1.67
1.19
1.89
2.01
2.72
3.07
6.00
5.98
f1 (ppm)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126.50
124.71
123.78
118.64
118.27
115.29
111.15
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46.75
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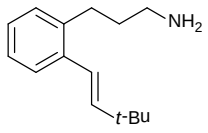
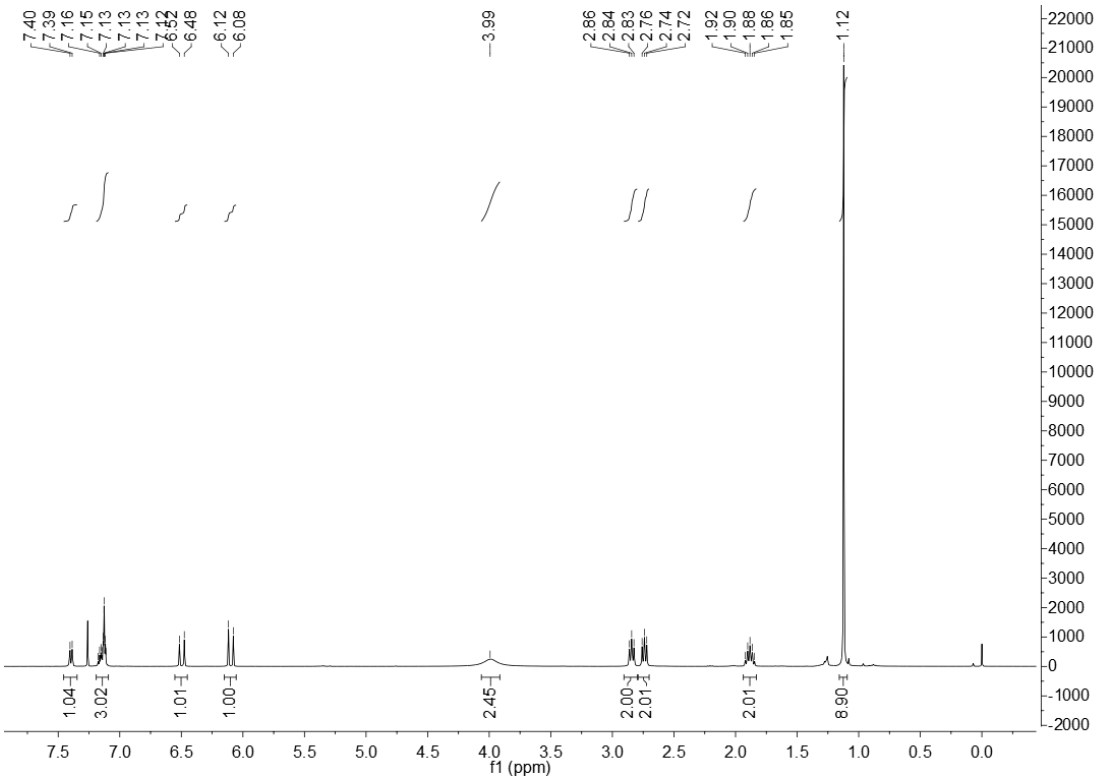
29.84
29.46
24.27
22.20
21.70
21.00
20.88
20.16
f1 (ppm)


NH2
t-Bu

3c-2


7.40
7.39
7.16
7.15
7.13
7.13
6.52
6.48
6.12
6.08
3.99
2.86
2.84
2.83
2.76
2.74
2.72
1.92
1.90
1.88
1.86
1.85
1.12
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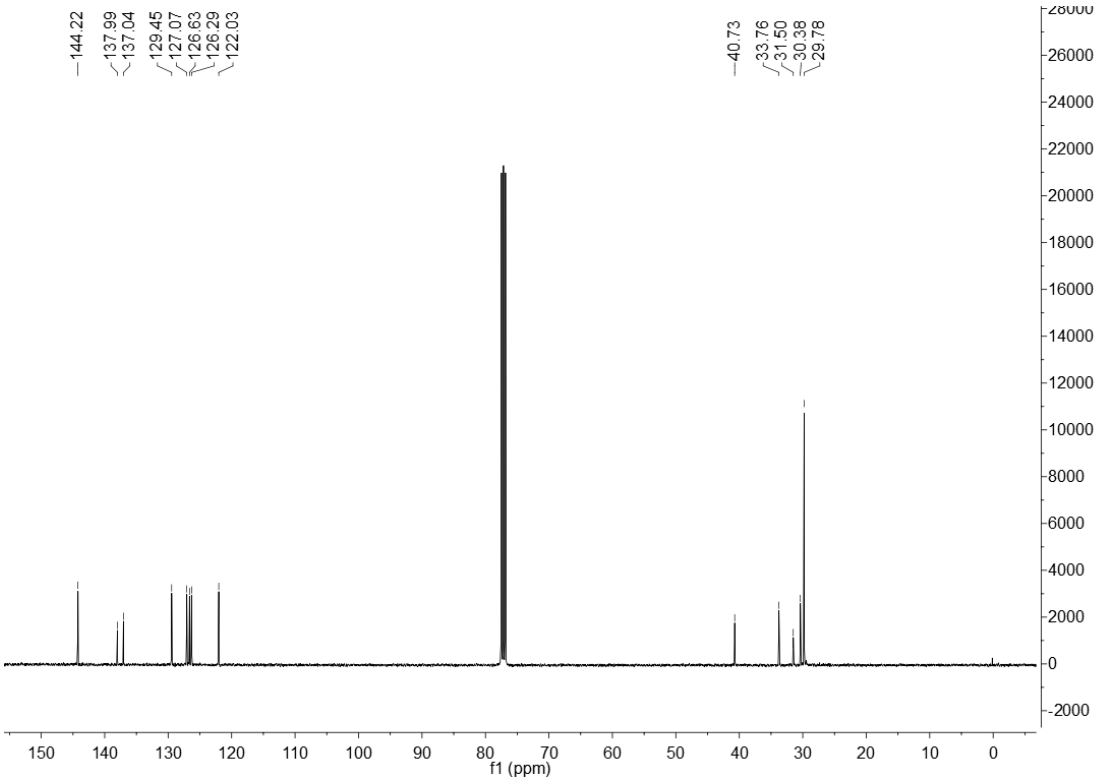
2.01
2.01
8.90
f1 (ppm)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137.99
137.04
129.45
127.07
126.63
126.29
122.03
40.73
33.76
31.50
30.38
29.78
f1 (ppm)