Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Palladium-catalyzed direct *ortho*-alkynylation of arylalkylacid derivatives at γ and δ positions via an N,O-bidentate directing group

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Supporting Information

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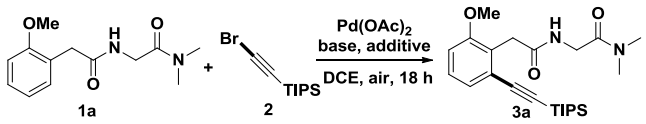
1. Reagents: Unless otherwise noted, all reagents were purchased from Acros, Alfa, Adamas and used without further purification. Column chromatography purifications were performed using 200–300 mesh silica gel.

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, brs = broad single, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet. HRMS analysis were carried out using TOF-MS instrument with EI source.

3. Optimization of reaction conditions

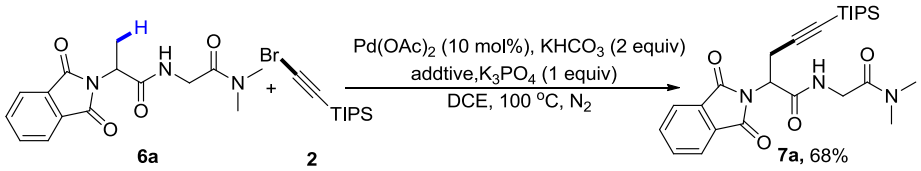
Table 3-1. Optimization of Reaction Conditions.



Entry[a]	$Pd(OAc)_2$(mol%)	Base	Additive(equiv)	Yield(%)
1	5	CsOAc	-	50
2	5	NaOAc	-	51
3	5	Na_2CO_3	-	23
4	5	K_2CO_3	-	42
5	5	Cs_2CO_3	-	46
6	5	Ag_2CO_3	-	59
7	5	$KHCO_3$	-	68
8	5	$KHCO_3$	1-AdOH (0.3)	78
9	5	$KHCO_3$	$HO_2P(OBn)_2$ (0.3)	62
10	5	$KHCO_3$	$MesCO_2H$ (0.3)	64
11[b]	**5**	**$KHCO_3$**	**PivOH(0.3)**	**96(94)**
12	-	$KHCO_3$	PivOH(0.3)	-

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), $Pd(OAc)_2$ (5 mol%), base (2 equiv), Additive (0.3 equiv), DCE (0.5 mL) in a 15 mL sealed tube at 80 ^oC for 18 h. Yields were based on LC using acetophenone as the internal standard. [b] Isolated yield on a 0.2 mmol scale.

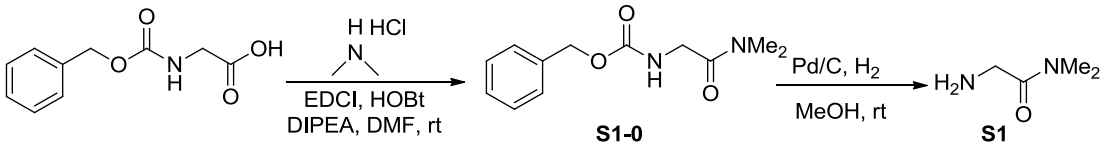
Table 3-2. Optimization of Reaction Conditions.



Entry[a]	$Pd(OAc)_2$(mol%)	Additive(equiv)	Yield(%)
1	5	1-AdOH (0.3)	68
2	5	$HO_2P(OBn)_2$ (0.3)	23
3	5	$MesCO_2H$ (0.3)	39
4	5	PivOH(0.3)	52

[a] Reaction conditions: **6a** (0.1 mmol), **2** (0.15 mmol), $Pd(OAc)_2$ (5 mol%), $KHCO_3$ (2 equiv), K_3PO_4 (1 equiv), Additive (0.3 equiv), DCE (0.5 mL) in a 15 mL sealed tube under N_2 at 100 °C for 18 h. Isolated yield on a 0.2 mmol scale.

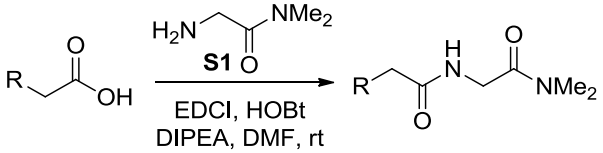
4. Preparation of S1-0 and S1



A mixture of acid (50 mmol, 1.0 equiv), amine (55 mmol, 1.1 equiv), EDCI (55 mmol, 1.1 equiv), HOBt (55 mmol, 1.1 equiv), and DIPEA (150 mmol, 3.0 equiv) in DMF(50 mL) was stirred at room temperature over night. Saturated ammonium chloride was added and the mixture was extraced with DCM. The combined organic layers was washed sodium bicarbonate and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to give **S1-0** as a white solid in 87% yield.

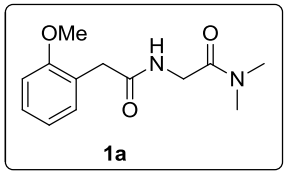
Compound **S1-0** (7.08 g, 30 mmol) was dissloved in methanol (150 mL) and 10% palladium-carbon(0.71 g) was added. The reaction mixture was shaked under hydrogen gas at room temperature over night. After removal of catalyst by filtration with aid of calite, the filtrate was concentrated under vacuum to give the **S1** as pale yellow liquid in 95% yield. The crude product was used in the next step without any purification.

5. General procedures for the preparation of various carboxylic acid substrates



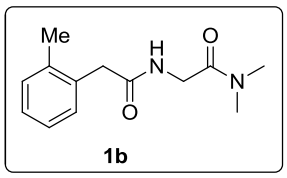
A mixture of carboxylic acid (10 mmol, 1.0 equiv), **S1** (11 mmol, 1.1 equiv), EDCI (11 mmol, 1.1 equiv), HOBt (11 mmol, 1.1 equiv), and DIPEA (13 mmol, 3.0 equiv) in DMF (15 mL) was stirred at room temperature over night. Water was added and the mixture was extracted with DCM. The combined organic layer was washed with saturated ammonium chloride, sodium bicarbonate

and brine, dried over Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to give corresponding carboxylic acid substrates as white solid or colourless liquid with >85% yield.



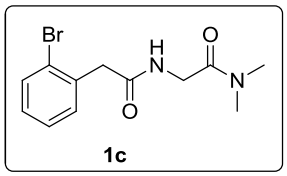
1a

2-(2-(2-methoxyphenyl)acetamido)-*N,N*-dimethylacetamide. White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 2H, ArH), 6.97 (brs, 1H, NH), 6.95–6.86 (m, 2H, ArH), 3.97 (d, *J* = 3.9 Hz, 2H, CH_2), 3.88 (s, 3H, OCH_3), 3.61 (s, 2H, CH_2), 2.93 (d, *J* = 4.3 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.28, 167.91, 157.13, 131.08, 128.76, 123.51, 120.91, 110.64, 55.47, 41.52, 38.50, 35.88, 35.49. HRMS Calcd for $C_{13}H_{18}N_2O_3$ $[M+H]^+$: 251.1396; Found: 251.1394.



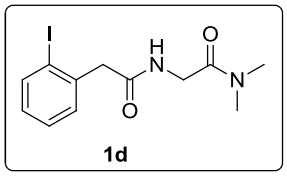
1b

***N, N*-dimethyl-2-(2-(*o*-tolyl)acetamido)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (s, 4H, ArH), 6.46 (brs, 1H, NH), 3.98 (d, *J* = 3.6 Hz, 2H, CH_2),3.61 (s, 2H, CH_2), 3.00 – 2.85 (m, 6H, CH_3), 2.30 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.85, 167.79, 137.10, 133.12, 130.81, 130.45, 127.87, 126.65, 41.47, 35.90, 35.56, 19.60. HRMS Calcd for $C_{13}H_{18}N_2O_2$ $[M+H]^+$: 235.1447; Found: 235.1439.



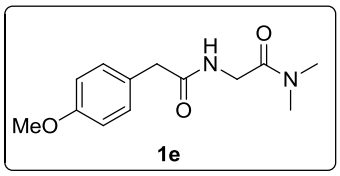
1c

2-(2-(2-bromophenyl)acetamido)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (dd, J_1 = 8.0 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.09 (dd, J_1 = 7.6 Hz, J_2 = 1.8 Hz, 1H, ArH), 6.99–7.07(m, 1H, ArH), 6.91–6.86 (m, ArH), 6.38 (brs, 1H, NH), 3.75 (d, *J* = 4.0 Hz, 2H, CH_2), 3.49 (s, 2H, CH_2), 2.67 (s, 6H, CH_3).^{13}C NMR (101 MHz, $CDCl_3$) δ 169.63, 167.73, 134.67, 133.05, 131.65, 129.16, 127.98, 125.03, 43.73, 41.52, 35.91, 35.57. HRMS Calcd for $C_{12}H_{15}BrN_2O_2$ $[M+H]^+$: 299.0395; Found: 299.0392.



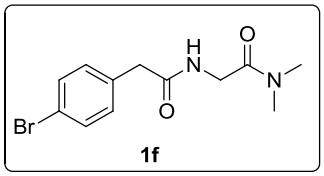
1d

2-(2-(2-iodophenyl)acetamido)-*N,N*-dimethylacetamide,White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93–7.78 (m, 1H, ArH), 7.41–7.28 (m, 2H,ArH), 6.99–6.96 (m, 1H, ArH), 6.61 (brs, 1H, NH), 4.02 (d, *J* = 3.9 Hz, 2H, CH_2), 3.77 (s, 2H, CH_2), 2.95 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.60, 167.77, 139.90, 138.20, 130.87, 129.26, 128.94, 101.25, 48.43, 41.60, 35.97, 35.64. HRMS Calcd for $C_{12}H_{15}IN_2O_2$ $[M+H]^+$: 347.0256; Found: 347.0262.



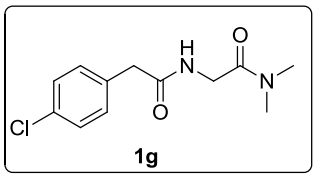
1e

2-(2-(4-methoxyphenyl)acetamido)-*N,N*-dimethylacet-amide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.6 Hz, 2H, ArH), 6.81 (d, *J* = 8.6 Hz, 2H, ArH), 6.66 (brs, 1H, NH), 3.94 (d, *J* = 4.1 Hz, 2H, CH_2), 3.71 (s, 3H, OCH_3), 3.48 (s, 2H, CH_2), 2.88 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.34, 167.80, 158.69, 130.34, 126.65, 114.23, 55.13, 42.49, 41.29, 35.79, 35.42. HRMS Calcd for $C_{13}H_{18}N_2O_3$ $[M+H]^+$: 251.1396; Found: 251.1385.



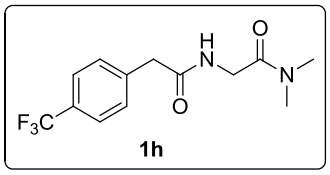
1f

2-(2-(4-bromophenyl)acetamido)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.4 Hz, 2H, ArH), 7.18 (d, *J* = 8.4 Hz, 2H, ArH), 6.63 (brs, 1H, NH), 4.02 (d, *J* = 4.0 Hz, 2H, CH_2), 3.56 (s, 2H, CH_2), 2.97 (d, *J* = 3.0 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.36, 167.80, 133.75, 132.03, 131.12, 121.38, 42.87, 41.50, 35.98, 35.65. HRMS Calcd for $C_{12}H_{15}BrN_2O_2$ $[M+H]^+$: 299.0395; Found: 299.0382.



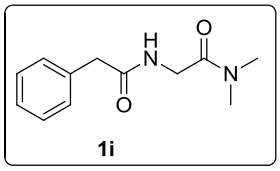
1g

2-(2-(4-chlorophenyl)acetamido)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 7.9 Hz, 2H, ArH), 7.22 (d, *J* = 7.9 Hz, 2H, ArH), 6.65 (brs, 1H, NH), 4.00 (d, *J* = 3.6 Hz, 2H, CH_2), 3.56 (s, 2H, CH_2), 2.95 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.46, 167.83, 133.27, 130.78, 129.12, 42.86, 41.52, 35.98, 35.65. HRMS Calcd for $C_{12}H_{15}ClN_2O_2$ $[M+H]^+$: 255.0900; Found: 255.0894.



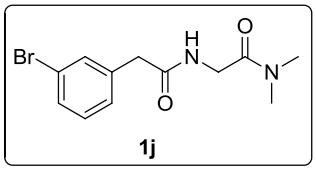
1h

N,N-dimethyl-2-(2-(4-(trifluoromethyl)phenyl)acetamido)acetamide, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 6.75 (s, 1H), 4.02 (d, *J* = 4.0 Hz, 2H), 3.64 (s, 2H), 2.95 (d, *J* = 2.8 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.99, 167.83, 138.98, 129.69, 129.42 (J_{C-F} =32 Hz), 125.69 (J_{C-F} =4 Hz), 124.16 (J_{C-F} =271 Hz), 43.05, 41.47, 35.92, 35.58. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.53. HRMS Calcd for $C_{13}H_{15}F_3N_2O_2$ $[M+H]^+$: 289.1164; Found: 289.1160.



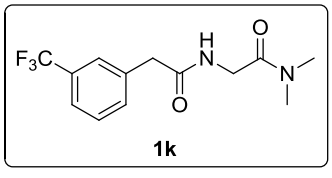
1i

***N, N*-dimethyl-2-(2-phenylacetamido)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 –7.28 (m, 2H, ArH), 7.27–7.22 (m, 3H, ArH), 6.55 (brs, 1H, NH), 3.97 (d, *J* = 4.0 Hz, 2H, CH_2), 3.57 (s, 2H, CH_2), 2.91 (s, 6H, CH_3). ^{13}C NMR (101 MHz, CDCl3) δ 170.47, 167.29, 134.17, 128.88, 128.48, 126.86, 43.11, 40.93, 35.38, 35.04. HRMS Calcd for $C_{12}H_{16}N_2O_2$ $[M+H]^+$: 221.1290; Found: 221.1285.



1j

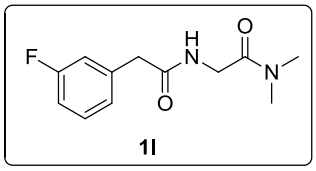
2-(2-(3-bromophenyl)acetamido)-*N, N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.32 (d, *J* = 7.7 Hz, 1H, ArH), 7.19–7.11(m, 2H, ArH), 6.93 (brs, 1H, NH), 3.97 (d, *J* = 4.2 Hz, 2H, CH_2), 3.51 (s, 2H, CH_2), 2.89 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.16, 167.81, 137.15, 132.19, 130.16, 127.89, 122.56, 42.66, 41.36, 35.87, 35.50. HRMS Calcd for $C_{12}H_{15}BrN_2O_2$ $[M+H]^+$: 299.0395; Found: 299.0392.



1k

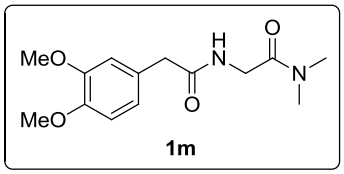
***N, N*-dimethyl-2-(2-(3-(trifluoromethyl)phenyl)acetami-do)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (s, 1H, ArH), 7.52–7.45 (m, 2H, ArH), 7.45–7.37 (m, 1H, ArH), 6.91 (brs, 1H, NH), 4.02 (d, *J* = 4.0 Hz, 2H, CH_2), 3.63 (s, 2H, CH_2), 2.93 (d, *J* = 1.0 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.02, 167.86, 136.17, 132.44, 130.17 (J_{C-F} =32 Hz), 128.61, 125.61

($J_{C\text{-}F}$ =3 Hz), 123.80 ($J_{C\text{-}F}$ =246 Hz), 123.26 ($J_{C\text{-}F}$ =1 Hz), 42.08, 41.02, 35.46, 35.02. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.59. HRMS Calcd for $C_{13}H_{15}F_3N_2O_2$ $[M+H]^+$: 289.1164; Found: 289.1154.



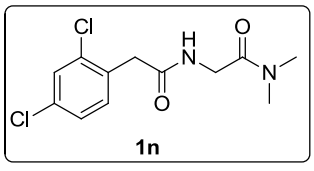
1l

2-(2-(3-fluorophenyl)acetamido)-*N*, *N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.28(m, 1H, ArH), 7.07 (d, *J* = 7.7 Hz, 1H, ArH), 7.04–6.92 (m, 2H, ArH), 6.65 (brs, 1H, NH), 4.01 (d, *J* = 4.0 Hz, 2H, CH_2), 3.59 (s, 2H, CH_2), 2.96 (d, *J* = 1.7 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.29, 167.82, 164.29, 137.15 ($J_{C\text{-}F}$ =8 Hz), 130.49 ($J_{C\text{-}F}$ =9 Hz), 125.13 ($J_{C\text{-}F}$ =3 Hz), 116.46 ($J_{C\text{-}F}$ =22 Hz), 114.41 ($J_{C\text{-}F}$ =21 Hz), 43.26 ($J_{C\text{-}F}$ =2 Hz), 41.56, 35.99, 35.68. ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.61. HRMS Calcd for $C_{12}H_{15}FN_2O_2$ $[M+H]^+$: 239.1196; Found: 239.1188.



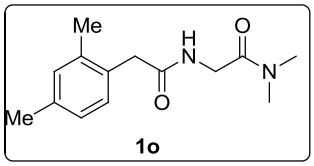
1m

2-(2-(3,4-dimethoxyphenyl)acetamido)-*N*,*N*-dimethyl-acetamide , White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83 (t, *J* = 4.9 Hz, 2H, ArH), 6.80 (s, 1H, ArH), 6.63 (s, 1H, NH), 4.00 (d, *J* = 4.1 Hz, 2H, CH_2), 3.86 (d, *J* = 5.0 Hz, 6H, OCH_3), 3.54 (s, 2H, CH_2), 2.95 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.26, 167.74, 148.99, 148.07, 127.05, 121.37, 112.28, 111.29, 55.74, 42.91, 41.25, 35.77, 35.40. HRMS Calcd for $C_{14}H_{20}N_2O_4$ $[M+H]^+$: 281.1501; Found: 281.1495.



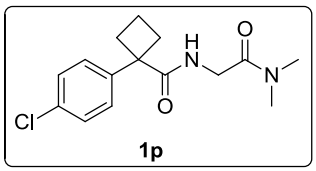
1n

2-(2-(2,4-dichlorophenyl)acetamido)-*N*, *N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 2.1 Hz, 1H, ArH), 7.26-7.28 (m, 1H, ArH), 7.19-7.21 (m, 1H, ArH), 6.80 (brs, 1H, NH), 4.01 (d, *J* = 4.0 Hz, 2H, ArH), 3.68 (s, 2H, CH_2), 2.94 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.07, 167.69, 135.07, 133.82, 132.33, 131.59, 129.43, 127.47, 41.46, 40.37, 35.86, 35.53. HRMS Calcd for $C_{12}H_{14}Cl_2N_2O_2$ $[M+H]^+$: 289.0511; Found: 289.0500.



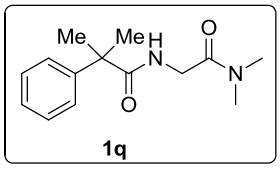
1o

2-(2-(2,4-dimethylphenyl)acetamido)-*N*, *N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *J*= 7.6 Hz, 1H, ArH), 7.09–7.00 (m, 2H, ArH), 6.50 (brs, 1H, NH), 4.05 (d, *J* = 4.1 Hz, 2H, CH_2), 3.64 (s, 2H, CH_2), 2.99 (d, *J* = 4.7 Hz, 6H, CH_3), 2.34 (d, *J* = 12.7 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.32, 131.14, 129.89, 129.40, 126.81, 40.74, 35.39, 35.04, 20.56, 18.97. HRMS Calcd for $C_{14}H_{20}N_2O_2$ $[M+H]^+$: 249.1603; Found: 249.1595.



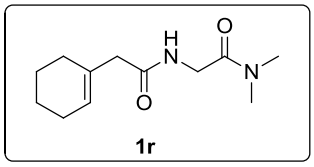
1p

1-(4-chlorophenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)cyclo-butanecarboxamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.32 (m, 2H, ArH), 7.29–7.23 (m, 2H, ArH), 6.41 (brs, 1H, NH), 3.94 (d, *J* = 4.0 Hz, 2H, CH_2), 2.93 (s, 6H, CH_3), 2.87–2.77 (m, 2H, CH_2), 2.52–2.39 (m, 2H, CH_2), 2.15–2.04 (m, 1H, CH_2), 1.95–1.79 (m, 1H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.83, 167.35, 142.41, 132.07, 128.29, 127.15, 51.95, 40.99, 35.27, 34.94, 31.76, 16.01. HRMS Calcd for $C_{15}H_{19}ClN_2O_2$ $[M+H]^+$: 295.1213; Found: 295.1206.



1q

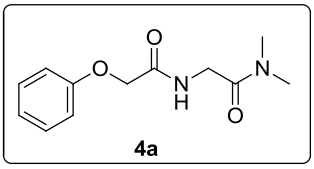
***N*-(2-(dimethylamino)-2-oxoethyl)-2-methyl-2-phenylpropan-amide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.33(m, 4H, ArH), 7.27-7.31 (m, 1H, ArH), 6.45 (brs, 1H, NH), 4.00 (d, *J* = 4.0 Hz, 2H, CH_2), 2.97 (d, *J* = 5.3 Hz, 6H, CH_3), 1.64 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.33, 168.08, 144.90, 128.72, 127.07, 126.34, 46.85, 41.62, 35.91, 35.55, 26.99. HRMS Calcd for $C_{14}H_{20}N_2O_2$ $[M+H]^+$: 249.1603; Found: 249.1596.



1r

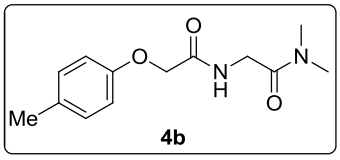
2-(2-(cyclohex-1-en-1-yl)acetamido)-*N*, *N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (s, 1H, NH), 5.65 (brs, 1H, CH), 4.02 (d, *J* = 4.0 Hz, 2H, CH_2), 2.91-2.98 (m, 8H, CH_2), 2.11–1.94 (m, 4H, CH_2), 1.71–1.53 (m, 4H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ

171.44, 168.14, 132.39, 127.29, 46.15, 41.44, 36.03, 35.69, 28.60, 25.48, 22.90, 22.10. HRMS Calcd for $C_{12}H_{20}N_2O_2$ $[M+H]^+$: 225.1603; Found: 225.1598.



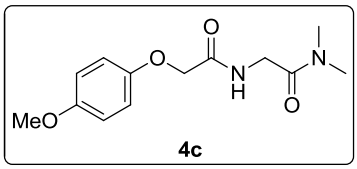
4a

***N, N*-dimethyl-2-(2-phenoxyacetamido)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (brs, 1H, NH), 7.14-7.19 (m, 2H, ArH), 6.92 – 6.75 (m, 3H, ArH), 4.38 (d, *J* = 6.0 Hz, 2H, CH_2), 3.97 (d, *J* = 4.1 Hz, 2H, CH_2), 2.81-2.85 (m, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.70, 166.89, 156.79, 129.12, 121.37, 114.20, 66.66, 40.22, 35.29, 34.95. HRMS Calcd for $C_{12}H_{16}N_2O_3$ $[M+H]^+$: 237.1239; Found: 237.1240.



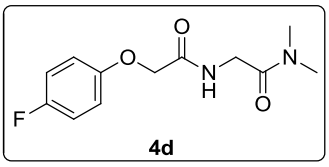
4b

***N,N*-dimethyl-2-(2-(p-tolyloxy)acetamido)acetamide,** White solid. ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.67(brs, 1H,NH), 7.08 (d, *J* = 8.5 Hz, 2H, ArH), 6.84 (d, *J* =8.5 Hz, 2H, ArH), 4.49 (s, 2H, CH_2), 4.11 (d, *J* = 4.2 Hz, 2H, CH_2), 2.99 (d, *J* = 2.2 Hz, 6H, CH_3), 2.27 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.64, 167.53, 155.35, 131.39, 130.21, 114.70, 67.53, 40.90, 36.01, 35.67, 20.57. HRMS Calcd for $C_{13}H_{18}N_2O_3$ $[M+H]^+$: 251.1396; Found: 251.1395.



4c

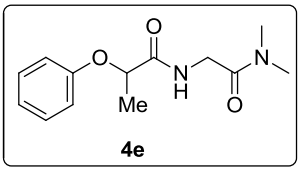
2-(2-(4-methoxyphenoxy)acetamido)-*N,N*-dimethylacet-amide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (brs, 1H, NH), 6.89 (d, *J* = 9.2 Hz, 2H, ArH), 6.83 (d, *J* = 9.1 Hz, 2H, ArH), 4.48 (s, 2H, CH_2), 4.12 (d, *J* = 4.2 Hz, 2H, CH_2), 3.76 (s, 3H, OCH_3), 3.00 (d, *J* = 2.3 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.73, 167.58, 151.65, 115.95, 114.93, 68.22, 55.81, 40.94, 36.05, 35.72. HRMS Calcd for $C_{13}H_{18}N_2O_4$ $[M+H]^+$: 267.1345; Found: 267.1348.



4d

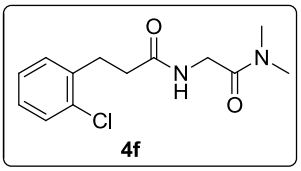
2-(2-(4-fluorophenoxy)acetamido)-*N, N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (brs, 1H), 7.04–6.95 (m, 2H, ArH), 6.94–6.86 (m, 2H, ArH), 4.49 (s,

2H, CH_2), 4.12 (d, *J* = 4.2 Hz, 2H, CH_2), 3.00 (s, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.11, 167.42, 159.09, 156.70, 153.50 (*J* = 3 Hz), 116.24, 116.01, 115.92, 67.96, 40.80, 35.90, 35.58. ^{19}F NMR (376 MHz, $CDCl_3$) δ -122.22 . HRMS Calcd for $C_{12}H_{16}FN_2O_3$ $[M+H]^+$: 255.1145; Found: 255.1148.



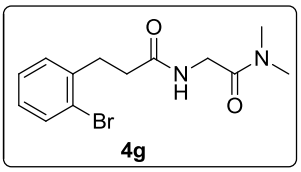
4e

***N*-(2-(dimethylamino)-2-oxoethyl)-2-phenoxypropanamide,** White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.53 (brs, 1H, NH), 7.26-7.30 (m, 2H, ArH), 6.98 (t, *J* = 7.1 Hz, 1H, ArH), 6.94 (d, *J* = 7.9 Hz, 2H, ArH), 4.77–4.69 (m, 1H, CH), 4.10-4.16 (m, 1H, CH_2), 3.94-3.99 (m, 1H, CH_2), 2.97 (d, *J* = 1.9 Hz, 6H, CH_3), 1.59 (d, *J* = 6.7 Hz, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.84, 167.00, 156.57, 129.21, 121.41, 115.10, 74.33, 40.46, 35.39, 35.07, 18.28. HRMS Calcd for $C_{13}H_{18}N_2O_3$ $[M+H]^+$: 251.1396; Found: 251.1398.



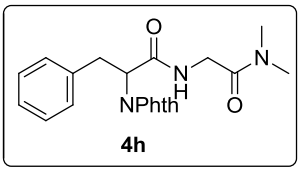
4f

3-(2-chlorophenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)propan-amide, White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.39–7.34 (m, 1H, ArH), 7.31–7.28 (m, 1H, ArH), 7.22–7.16 (m, 2H, ArH), 6.69 (brs, 1H, NH), 4.07 (d, *J*=3.9 Hz, 2H, CH_2), 3.15–3.10 (m, 2H, CH_2), 3.01 (s, 6H, CH_3), 2.64–2.58 (m, 2H, CH_2); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.86, 167.99, 138.44, 133.97, 130.62, 129.61, 127.84, 126.99, 41.40, 36.02, 35.98, 35.63, 29.60; HRMS Calcd for $C_{13}H_{18}ClN_2O_2$ $[M+H]^+$: 269.1057; Found: 269.1065.



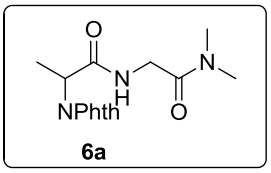
4g

3-(2-bromophenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)prop-Anamide, White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.50–7.46 (m, 1H, ArH), 7.25–7.15 (m, 2H, ArH), 7.04–7.00 (m, 1H, ArH), 6.70 (brs, 1H, NH), 4.00 (d, *J*=4.0 Hz, 2H, CH_2), 3.09–3.03 (m, 2H, CH_2), 2.93 (s, 6H, CH_3), 2.57–2.51 (m, 2H, CH_2); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.72, 167.95, 140.11, 132.85, 130.54, 128.01, 127.59, 124.33, 41.34, 36.07, 35.92, 35.57, 32.03; HRMS Calcd for $C_{13}H_{18}BrN_2O_2$ $[M+H]^+$: 313.0552; Found: 313.0561.



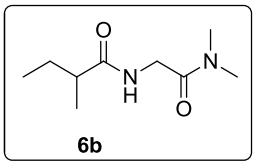
4h

***N*-(2-(dimethylamino)-2-oxoethyl)-2-(1,3-dioxoisoindolin-2-yl)-3-phenylpropanamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76–7.73 (m, 2H, ArH), 7.68–7.62 (m, 2H, ArH), 7.25 (d, *J* = 7.0 Hz, 1H, ArH), 7.14 (t, *J* = 6.7 Hz, 4H, ArH), 7.09 (dd, J_1 = 8.5 Hz, J_2 = 4.0 Hz, 1H, ArH), 5.16 (t, *J* = 8.4 Hz, 1H, CH), 4.06 (d, *J* = 3.8 Hz, 2H, CH_2), 3.57 (d, *J* = 8.4 Hz, 2H, CH_2), 2.95 (d, *J* = 5.3 Hz, 6H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.31, 167.90, 167.57, 136.77, 134.25, 131.49, 128.95, 128.64, 126.93, 123.61, 77.48, 77.16, 76.84, 55.46, 41.76, 35.96, 35.63, 34.73. HRMS Calcd for $C_{21}H_{21}N_3O_4$ $[M+H]^+$: 379.1532; Found:379.1538.



6a

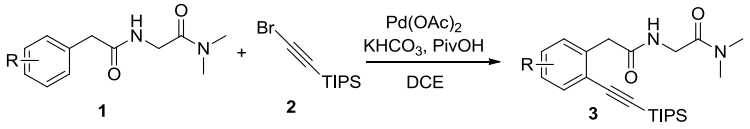
***N*-(2-(dimethylamino)-2-oxoethyl)-2-(1,3-dioxoisoindolin-2-yl)propanamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.84 (m, 2H, ArH), 7.76–7.69 (m, 2H, ArH), 7.06 (brs, 1H, NH), 5.00–4.95 (m, 1H, CH), 4.06 (d, *J*=3.8 Hz, 2H, CH_2), 2.97 (d, *J*=1.4 Hz, 6H, CH_3), 1.73 (d, *J*=7.3 Hz, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.01, 167.87, 167.72, 134.32, 132.04, 123.70, 49.14, 41.79, 35.99, 35.68, 15.40; HRMS Calcd for $C_{15}H_{18}N_3O_4$ $[M+H]^+$: 304.1297; Found: 304.1302.



6b

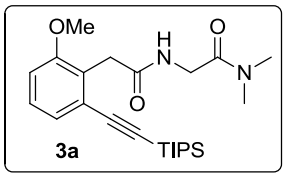
***N*-(2-(dimethylamino)-2-oxoethyl)-2-methylbutanamide,**White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.58 (brs, 1H,NH), 4.03 (d, *J*=3.9 Hz, 2H, CH_2), 2.98 (d, *J*=6.3 Hz, 6H, CH_3), 2.24–2.17 (m, 1H, CH), 1.72–1.61 (m, 1H, CH_2), 1.49–1.38 (m, 1H, CH_2), 1.15–1.11 (m, 3H, CH_3), 0.91–0.84 (m, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.61, 168.29, 43.02, 41.28, 35.99, 35.65, 27.39, 17.48, 12.01; HRMS Calcd for $C_9H_{19}N_2O_2$ $[M+H]^+$:187.1447; Found: 187.1445.

6. General procedures for the alkylation of phenylacetic acid

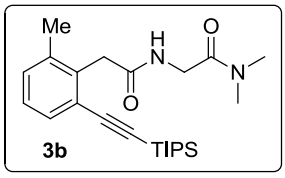


1 **2** **3**

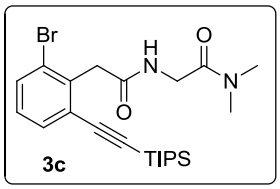
A mixture of **1** (0.2 mmol), **2** (0.3 mmol), $Pd(OAc)_2$ (2.2 mg, 5 mol%), $KHCO_3$ (40 mg, 0.4 mmol), PivOH (6 mg, 0.06 mmol) and DCE (1 mL) in a 15 mL sealed glass vial was heated at 80 ^oC with vigorous stirring for 18 hours. The reaction mixture was cooled to room temperature, and diluted with ethyl acetate and filtered through celite. The filtrate was concentrated in vacuo and purified by column chromatography on silica gel (Ethyl acetate/Petroleum ether = 1:3) to give the corresponding product.



2-(2-(2-methoxy-6-((triisopropylsilyl)ethynyl)phenyl)aceta-mido)-*N*, *N*-dimethylacetamide, White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, *J* = 7.9 Hz, 1H, ArH), 7.14 (d, *J* = 7.7 Hz, 1H, ArH), 6.89 (d, *J* = 8.1 Hz, 1H, ArH), 6.64 (brs, 1H, NH), 3.97 (d, *J* = 3.8 Hz, 2H, CH_2), 3.90 (s, 2H, CH_2), 3.83 (s, 3H, OCH_3), 2.92 (s, 6H, CH_3), 1.14 – 0.94 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.29, 167.89, 157.59, 128.36, 125.59, 125.29, 125.25, 111.21, 104.73, 95.36, 55.79, 41.53, 36.27, 35.89, 35.49, 18.72, 11.32. HRMS Calcd for $C_{24}H_{38}N_2O_3Si$ $[M+H]^+$: 431.2730; Found: 431.2723.

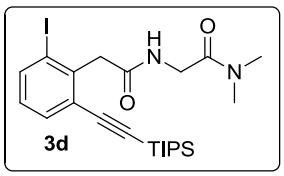


***N*,*N*-dimethyl-2-(2-(2-methyl-6-((triisopropylsilyl)ethynyl)-phenyl)acetamido)acetamide,** White solid. ^1H NMR(400 MHz, $CDCl_3$) δ 7.42–7.31 (m, 1H, ArH), 7.18–7.05 (m, 2H, ArH), 6.42 (brs, 1H, NH), 3.99–3.85 (m, 4H, CH_2), 2.87 (d, *J* = 6.3 Hz, 6H, CH_3), 2.27 (s, 3H, CH_3), 1.13–0.94 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.96, 167.63, 137.79, 135.09, 130.99, 127.42, 124.49, 105.40, 95.22, 41.36, 39.44, 35.89, 35.51, 20.10, 18.70, 11.30. HRMS Calcd for $C_{24}H_{38}N_2O_2Si$ $[M+H]^+$: 415.2781; Found: 415.2772.

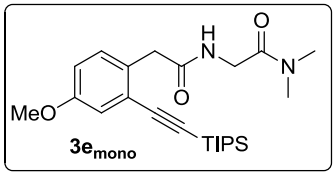


2-(2-(2-bromo-6-((triisopropylsilyl)ethynyl)phenyl)acetamido)-*N*,*N*-dimethylacetamide, White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, J_1 = 8.0 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.47 (d, *J* = 7.7 Hz, 1H, ArH), 7.08 (t, *J* = 7.9 Hz, 1H, ArH), 6.48 (brs, 1H, NH), 4.07 (s, 2H, CH_2), 3.96 (d, *J* = 3.4 Hz, 2H, CH_2), 2.90 (s, 6H, CH_3), 1.12 –0.91 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.51, 167.62, 136.45, 133.31, 132.25, 128.70, 126.26, 125.76, 104.10, 96.95, 42.48,

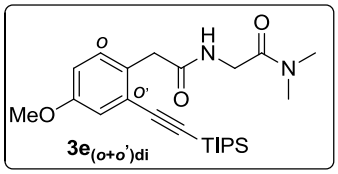
41.52, 35.88, 35.51, 18.65, 11.22. HRMS Calcd for $C_{23}H_{35}BrN_2O_2Si$ $[M+H]^+$: 479.1729; Found: 479.1744.



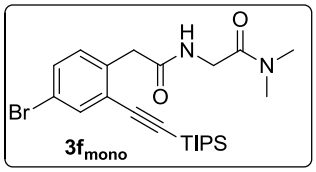
2-(2-(2-iodo-6-((triisopropylsilyl)ethynyl)phenyl)acetamido)-*N*, *N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.72 (m, 1H, ArH), 7.47 (d, *J* = 7.6 Hz, 1H, ArH), 6.88 (t, *J* = 7.8 Hz, 1H, ArH), 6.43 (brs, 1H, NH), 4.07 (s, 2H, CH_2), 3.94 (d, *J* = 3.5 Hz, 2H, CH_2), 2.88 (d, *J* = 1.1 Hz, 6H, CH_3), 1.12– 0.94 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.20, 167.42, 139.88, 139.52, 132.96, 128.73, 125.14, 104.36, 101.54, 96.63, 47.18, 41.38, 35.74, 35.36, 18.51, 11.07. HRMS Calcd for $C_{23}H_{35}IN_2O_2Si$ $[M+H]^+$: 527.1591; Found: 527.1590.



2-(2-(4-methoxy-2-((triisopropylsilyl)ethynyl)phenyl)ace -tamido)-*N*,*N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.5 Hz, 1H, ArH), 7.02 (d, *J* = 2.8 Hz, 1H, ArH), 6.86 (dd, J_1 = 8.5 Hz, J_2 = 2.8 Hz, 1H, ArH), 6.52 (brs, 1H, NH), 3.96 (d, *J* = 4.0 Hz, 2H, CH_2), 3.77 (d, *J* = 7.0 Hz, 5H, CH_2+CH_3), 2.92 (d, *J* = 3.4 Hz, 6H, CH_3), 1.15 – 1.00 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.76, 167.90, 158.68, 131.18, 129.11, 124.93, 117.82, 115.83, 104.89, 95.61, 55.57, 41.56, 36.07, 35.68, 18.87, 11.45. HRMS Calcd for $C_{24}H_{38}N_2O_2Si$ $[M+H]^+$: 431.2730; Found: 431.2719.

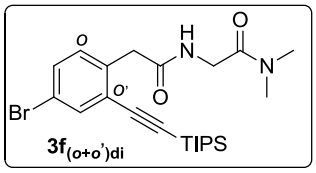


2-(2-(4-methoxy-2,6-bis((triisopropylsilyl)ethynyl)phenyl)acetamido)-*N*,*N*-dimethylacetamid e, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (s, 2H, ArH), 6.40 (s, 1H, NH), 4.01 (s, 2H, CH_2), 3.90 (d, *J* = 3.5 Hz, 2H, CH_2), 3.78 (s, 3H, OCH_3), 2.89 (d, *J* = 4.9 Hz, 6H, CH_3), 1.15–0.92 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.65, 168.04, 158.40, 131.39, 125.99, 119.03, 104.56, 96.27, 55.82, 41.83, 40.45, 36.22, 35.79, 18.98, 11.57. $C_{35}H_{58}N_2O_3Si_2$[M+H]$^+$: 611.4064; Found: 611,4070.



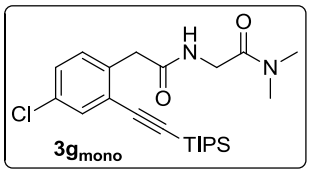
2-(2-(4-bromo-2-((triisopropylsilyl)ethynyl)phenyl)aceta-mido)-*N,N*-dimethylacetamide,

White solid. ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 1.8 Hz, 1H, ArH), 7.41 (dd, J_1 = 8.2 Hz, J_2 = 1.8 Hz, 1H, ArH), 7.17 (d, *J* = 8.3 Hz, 1H, ArH), 6.57 (brs, 1H, NH), 3.97 (d, *J* = 3.9 Hz, 2H, CH_2), 3.77 (s, 2H, CH_2), 2.93 (s, 6H, CH_3), 1.18–0.99 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.56, 167.62, 135.65, 132.07, 131.33, 125.81, 120.91, 103.22, 97.50, 41.51, 35.94, 35.59, 18.71, 11.25. $C_{23}H_{35}BrN_2O_2Si$ $[M+H]^+$: 479.1729; Found: 479.1722.



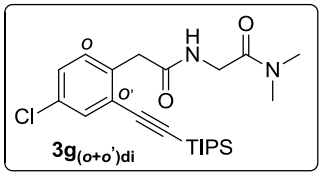
2-(2-(4-bromo-2,6-bis((triisopropylsilyl)ethynyl)phenyl)acetamido)-*N,N*-dimethylacetamide,

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 2.1 Hz, 2H, ArH), 6.50 (brs, 1H, NH), 4.09 (s, 2H, CH_2), 3.97 (d, *J* = 3.5 Hz, 2H, CH_2), 2.96 (s, 6H, CH_3), 1.18–1.06 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.25, 167.62, 137.68, 135.39, 126.48, 120.57, 102.78, 97.94, 41.51, 40.37, 35.92, 35.52, 18.65, 11.20. $C_{34}H_{55}BrN_2O_2Si_2$ $[M+H]^+$: 659.3064; Found: 659.3067.

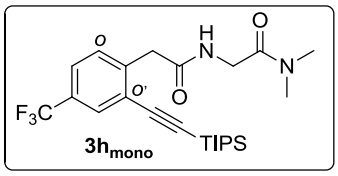


2-(2-(4-chloro-2-((triisopropylsilyl)ethynyl)phenyl)aceta-mido)-*N,N*-dimethylacetamide,

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (s, 1H, ArH), 7.29–7.19 (m, 2H, ArH), 6.58 (brs, 1H, NH), 3.96 (d, *J* = 3.9 Hz, 2H, CH_2), 3.78 (s, 2H, CH_2), 2.92 (s, 6H, CH_3), 1.16–0.98 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.69, 167.63, 135.23, 133.01, 132.66, 131.08, 129.12, 125.44, 103.36, 97.32, 41.41, 35.90, 35.54, 18.67, 11.22. HRMS Calcd for $C_{23}H_{35}ClN_2O_2Si[M+H]^+$: 435.2235; Found: 435.2214.

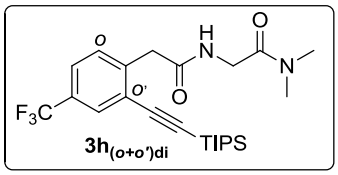


2-(2-(4-chloro-2,6-bis((triisopropylsilyl)ethynyl)phenyl)acetamido)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (s, 2H, ArH), 6.47 (brs, 1H, NH), 4.09 (s, 2H, CH_2), 3.96 (d, *J* = 3.5 Hz, 2H, CH_2), 2.95 (s, 6H, CH_3), 1.18–1.04 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.29, 167.61, 137.23, 132.87, 132.57, 126.26, 102.95, 97.82, 41.50, 40.29, 35.88, 35.48, 18.63, 11.21. $C_{34}H_{55}ClN_2O_2Si_2$ $[M+H]^+$: 615.3569; Found: 615.3575.



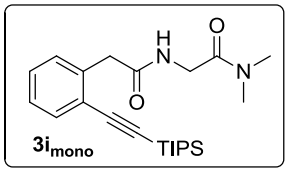
3h$_{mono}$

N,N-dimethyl-2-(2-(4-(trifluoromethyl)-2-((triisopropylsilyl)ethynyl)phenyl)acetamido)aceta mide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (s, 1H, ArH), 7.51 (d, *J* = 8.1 Hz, 1H, ArH), 7.43 (d, *J* = 8.1 Hz, 1H, ArH), 6.66 (brs, 1H, NH), 3.98 (d, *J* = 3.7 Hz, 2H, CH_2), 3.87 (s, 2H, CH_2), 2.92 (s, 6H, CH_3), 1.14 – 0.99 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.16, 167.58, 140.68, 130.30, 129.73 (J_{C-F} = 4 Hz), 129.72 (J_{C-F} =32 Hz), 125.30 (J_{C-F} = 1 Hz), 124.62, 123.68 (J_{C-F} =192 Hz), 103.23, 97.82, 41.74, 41.49, 35.88, 35.53, 18.67, 11.22. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.80. HRMS Calcd for $C_{24}H_{35}F_3N_2O_2Si$ $[M+H]^+$: 469.2498; Found: 469.2495.



3h$_{(o+o')di}$

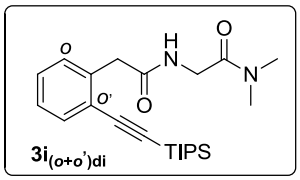
N,N-dimethyl-2-(2-(4-(trifluoromethyl)-2,6-bis((triisopropylsilyl)ethynyl)phenyl)acetamido)a cetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (s, 2H, ArH), 6.46 (brs, 1H, NH), 4.14 (s, 2H, CH_2), 3.93 (d, *J* = 3.3 Hz, 2H, CH_2), 2.92 (s, 6H, CH_3), 1.26 – 0.79 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.77, 167.57, 142.42, 129.97 (J_{C-F} = 33 Hz), 129.15 (J_{C-F} = 4 Hz), 125.76, 123.29 (J_{C-F} =272 Hz), 102.86, 98.35, 41.55, 40.71, 35.92, 35.53, 18.67, 11.23. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.01. HRMS Calcd for $C_{35}H_{55}F_3N_2O_2Si_2$ $[M+H]^+$: 649.3832; Found:. 649.3829.



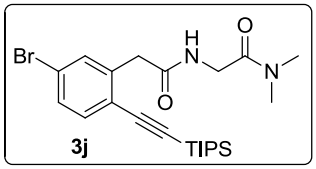
3i$_{mono}$

***N,N*-dimethyl-2-(2-(2-((triisopropylsilyl)ethynyl)phenyl)acetamido)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51(d, *J* = 7.4 Hz, 1H, ArH), 7.29 (d, *J* = 4.4 Hz, 2H, ArH), 7.26–7.18 (m, 1H, ArH), 6.55 (brs, 1H, NH), 3.96 (d, *J* = 3.7 Hz, 2H, CH_2), 3.83 (s, 2H, CH_2),

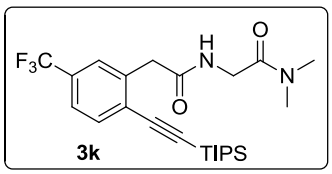
2.98–2.83 (m, 6H, CH_3), 1.18–0.97 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.22, 167.68, 136.71, 133.18, 129.84, 129.00, 127.37, 123.88, 104.75, 95.70, 42.11, 41.46, 35.89, 35.52, 18.69, 11.27. HRMS Calcd for $C_{23}H_{36}N_2O_2Si$ $[M+Na]^+$: 423.2444; Found: 423.2429.



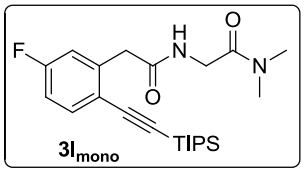
2-(2-(2,6-bis((triisopropylsilyl)ethynyl)phenyl)acetamido-*N*,*N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.7 Hz, 2H, ArH), 7.21 (t, *J* = 7.8 Hz, 1H, ArH), 6.41 (brs, 1H, NH), 4.12 (s, 2H, CH_2), 3.92 (d, *J* = 3.6 Hz, 2H, CH_2), 2.91 (d, *J* = 4.6 Hz, 6H, CH_3), 1.10–1.04 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.90, 167.72, 138.70, 133.08, 127.41, 124.88, 104.26, 96.30, 41.58, 40.94, 35.95, 35.53, 18.72, 11.31. $C_{34}H_{56}N_2O_2Si_2$ $[M+H]^+$: 581.3959; Found: 581.3943.



2-(2-(5-bromo-2-((triisopropylsilyl)ethynyl)phenyl)acet-amido)-*N*,*N*-dimethylacetamide, White solid. ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.48 (s, 1H, ArH), 7.37 (s, 2H, ArH), 6.64 (brs, 1H, NH), 4.01 (d, *J* = 3.9 Hz, 2H, CH_2), 3.81 (s, 2H, CH_2), 2.96 (s, 6H, CH_3), 1.21–0.98 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.35, 167.59, 138.78, 134.32, 132.76, 130.53, 122.87, 103.80, 97.17, 41.65, 35.94, 35.58, 18.72, 11.27. HRMS Calcd for $C_{23}H_{35}BrN_2O_2Si$ $[M+H]^+$: 479.1729; Found: 479.1739.

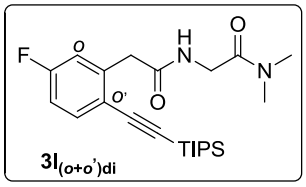


***N*,*N*-dimethyl-2-(2-(5-(trifluoromethyl)-2-((triisoprop-ylsilyl)ethynyl)phenyl)acetamido)aceta mide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68–7.54 (m, 2H, ArH), 7.47 (d, *J* = 8.0 Hz, 1H, ArH), 6.65 (brs, 1H, NH), 4.00 (d, *J* = 3.9 Hz, 2H, CH_2), 3.88 (s, 2H, CH_2), 2.94 (s, 6H, CH_3), 1.11–0.96 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.14, 167.54, 137.71, 133.32, 130.36 ($J_{C\text{-}F}$ = 33 Hz), 127.50, 126.55 ($J_{C\text{-}F}$ = 4 Hz), 123.97 ($J_{C\text{-}F}$ = 3 Hz), 122.40, 103.46, 98.77, 41.65, 41.50, 35.83, 35.47, 18.62, 11.20. HRMS Calcd for $C_{24}H_{35}F_3N_2O_2Si$ $[M+H]^+$: 469.2498; Found: 469.2494.



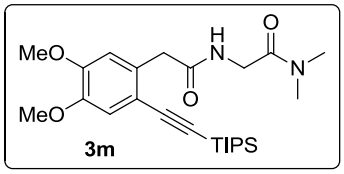
2-(2-(5-fluoro-2-((triisopropylsilyl)ethynyl)phenyl)aceta-mido)-*N,N*-dimethylacetamide,

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.57 (m, 1H, ArH), 7.08-7.11 (m, 1H, ArH), 7.03–6.96 (m, 1H, ArH), 6.66 (brs, 1H, NH), 4.05 (d, J = 3.9 Hz, 2H, CH_2), 3.88 (s, 2H, CH_2), 3.00 (s, 6H, CH_3), 1.18–1.12 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.51, 167.65, 139.37, 134.96 ($J_{C\text{-}F}$ = 8 Hz), 116.99 ($J_{C\text{-}F}$ = 23 Hz), 114.75 ($J_{C\text{-}F}$ = 22 Hz), 103.84, 95.50, 42.04, 41.59, 35.99, 35.64, 18.78, 11.35. ^{19}F NMR (376 MHz, $CDCl_3$) δ -109.71. HRMS Calcd for $C_{23}H_{35}FN_2O_2Si$ $[M+H]^+$: 419.2530; Found: 419.2531.

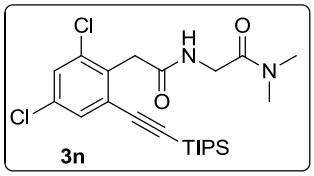


2-(2-(3-fluoro-2,6-bis((triisopropylsilyl)ethynyl)phenyl)ac-etamido)-*N,N*-dimethylacetamide,

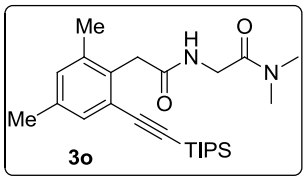
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (m, J_1 = 8.5 Hz, J_2 = 5.5 Hz, 1H, ArH), 6.95 (m, J = 8.5 Hz, 1H, ArH), 6.47 (brs, 1H, NH), 4.08 (s, 2H, CH_2), 3.91 (d, J = 3.5 Hz, 2H, CH_2), 2.90 (s, 6H, CH_3), 1.09–0.99 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.23, 167.68, 161.90, 141.28, 133.99 ($J_{C\text{-}F}$ = 8 Hz), 120.76, 114.89 ($J_{C\text{-}F}$ = 22 Hz), 113.91 ($J_{C\text{-}F}$ = 18 Hz), 103.45, 102.71, 97.16, 95.79, 41.61, 40.89, 35.97, 35.58, 18.73, 18.70, 11.32, 11.27. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.94. $C_{34}H_{55}FN_2O_2Si_2$ $[M+H]^+$: 599.3864; Found: 599.3853.



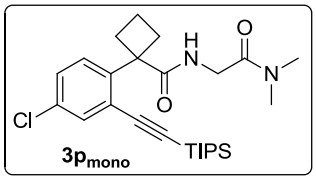
2-(2-(4,5-dimethoxy-2-((triisopropylsilyl)ethynyl)phen-yl)acetamido)-*N,N*-dimethylacetamid e, White solid.^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (s, 1H, ArH), 6.76 (s, 1H, ArH), 6.59 (brs, 1H, NH), 3.94 (d, J = 4.0 Hz, 2H, CH_2), 3.83 (d, J = 1.1 Hz, 6H, OCH_3), 3.75 (s, 2H, CH_2), 2.89 (d, J = 3.7 Hz, 6H, CH_3), 1.13–0.95 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.40, 167.58, 149.71, 147.84, 130.20, 115.71, 114.94, 112.48, 104.91, 93.66, 55.95, 41.80, 41.42, 35.85, 35.47, 29.69, 18.68, 11.27. HRMS Calcd for $C_{25}H_{40}N_2O_2Si$ $[M+H]^+$: 461.2836; Found: 461.2825.



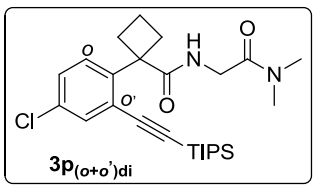
2-(2-(2,4-dichloro-6-((triisopropylsilyl)ethynyl)phenyl)ac-etamido)-*N*,*N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 2.1 Hz, 1H, ArH), 7.38 (d, *J* = 2.1 Hz, 1H, ArH), 6.52 (brs, 1H, NH), 4.08–3.93 (m, 4H, CH_2), 2.95 (d, *J* = 2.9 Hz, 6H, CH_3), 1.19–1.01 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.87, 167.50, 135.91, 133.43, 131.13, 129.56, 127.18, 102.65, 98.30, 41.39, 39.06, 35.75, 35.39, 18.51, 11.07. HRMS Calcd for $C_{23}H_{34}Cl_2N_2O_2Si$ $[M+H]^+$:469.1845; Found: 469.1848.



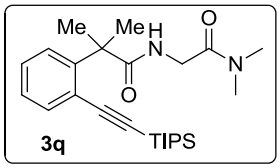
2-(2-(2,4-dimethyl-6-((triisopropylsilyl)ethynyl)phenyl)ac-etamido)-*N*,*N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (s, 1H, ArH), 6.97 (s, 1H, ArH), 6.41 (brs, 1H, NH), 3.94 (d, *J* = 3.6 Hz, 2H, CH_2), 3.87 (s, 2H, CH_2), 2.96–2.81 (m, 6H, CH_3), 2.24 (s, 6H, CH_3), 1.15–0.97 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.65, 167.13, 136.98, 136.44, 131.53, 130.86, 123.74, 105.06, 94.10, 40.79, 38.55, 35.34, 34.94, 20.29, 19.45, 18.14, 10.74. HRMS Calcd for $C_{25}H_{40}N_2O_2Si$ $[M+H]^+$: 429.2937; Found: 429.2929.



1-(4-chloro-2-((triisopropylsilyl)ethynyl)phenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)cyclobutane carboxamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47–7.43 (m, 1H, ArH), 7.33–7.27 (m, 2H, ArH), 6.24 (brs, 1H, NH), 3.93 (d, *J* = 4.0 Hz, 2H, CH_2), 2.91 (d, *J* = 8.6 Hz, 8H, CH_2), 2.57–2.49 (m, 2H, CH_2), 2.29–2.16 (m, 1H, CH_2), 1.86–1.78 (m, 1H, CH2), 1.08–1.01 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.41, 168.06, 144.44, 134.58, 132.62, 128.89, 128.35, 124.05, 103.92, 98.63, 52.81, 41.76, 35.93, 35.53, 32.74, 18.73, 17.09, 11.39. HRMS Calcd for $C_{26}H_{39}ClN_2O_2Si$ $[M+H]^+$: 475.2547; Found: 475.2556.

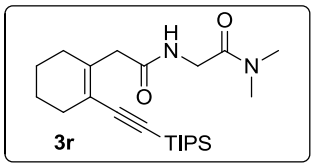


1-(4-chloro-2,6-bis((triisopropylsilyl)ethynyl)phenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)cyclobutanecarboxamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 2H, ArH), 6.49 (brs, 1H, NH), 3.94 (d, *J* = 3.8 Hz, 2H, CH_2), 3.12–2.99 (m, 2H, CH_2), 2.90 (d, *J* = 14.5 Hz, 6H, CH_3), 2.85–2.77 (m, 2H, CH_2), 2.39–2.31 (m, 1H, CH_2), 1.82–1.71 (m, 1H, CH_2), 1.12–1.02 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.76, 167.84, 134.97, 132.00, 124.80, 104.19, 99.78, 53.36, 41.94, 35.97, 35.54, 18.74, 11.38. $C_{37}H_{55}ClN_2O_2Si_2$ $[M+H]^+$: 655.3882; Found: 655.3876.



3q

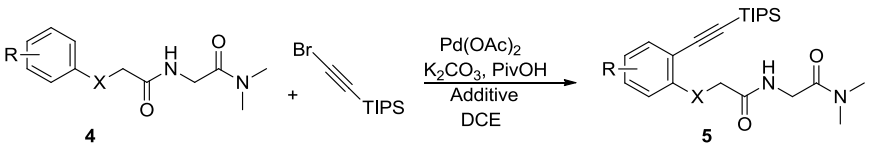
***N*-(2-(dimethylamino)-2-oxoethyl)-2-methyl-2-(2-((trriiso-propylsilyl)ethynyl)phenyl)propanamide,** White solid. ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.52-7.54 (m, 1H, ArH), 7.43 (d, *J* = 7.5 Hz, 1H, ArH), 7.36–7.28 (m, 1H, ArH), 7.25–7.17 (m, 1H, ArH), 6.15 (brs, 1H, NH), 3.96 (d, *J* = 4.0 Hz, 2H, CH_2), 2.90 (d, *J* = 11.5 Hz, 6H, CH_3), 1.67 (s, 6H, CH_3), 1.13–1.04 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.38, 168.23, 145.54, 135.95, 128.88, 127.06, 126.22, 123.26, 105.68, 97.44, 47.65, 41.86, 35.83, 35.44, 26.81, 18.72, 11.52. HRMS Calcd for $C_{25}H_{40}N_2O_2Si$ $[M+H]^+$: 429.2927; Found: 429.2934.



3r

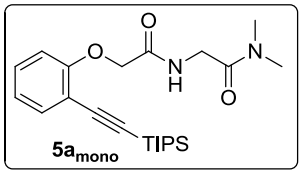
***N,N*-dimethyl-2-(2-(2-((triisopropylsilyl)ethynyl)cyclohex-1-en-1-yl)acetamido)acetamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 (brs, 1H, NH), 4.00 (d, *J* = 4.1 Hz, 2H, CH_2), 3.34 (s, 2H, CH_2), 2.97 (d, *J* = 4.3 Hz, 6H, CH_3), 2.26 (s, 2H, CH_2), 2.13 (s, 2H, CH_2), 1.69–1.60 (m, 4H, CH_2), 1.11–1.03 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.64, 167.33, 138.96, 119.72, 106.18, 93.01, 43.49, 40.81, 35.42, 35.05, 29.52, 29.09, 21.88, 21.66, 18.15, 10.73. HRMS Calcd for $C_{23}H_{40}N_2O_2Si$ $[M+H]^+$: 405.2937; Found: 405.2923.

7. General procedures for the alkylation of phenylpropionic acid

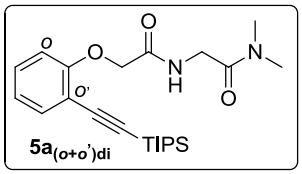


A mixture of **4** (0.2 mmol), **2** (0.3 mmol), $Pd(OAc)_2$ (2.2 mg, 5 mol%), K_2CO_3 (55.2 mg, 0.4 mmol, 2 equiv), PivOH (6 mg, 0.06 mmol) and DCE (1 mL) in a 15 mL sealed glass vial was heated at 100 ^{o}C with vigorous stirring for 18 hours. The reaction mixture was cooled to room

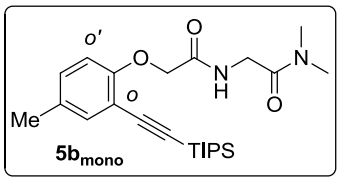
temperature, and diluted with ethyl acetate and filtered through celite. The filtrate was concentrated in vacuo and purified by column chromatography on silica gel (Ethyl acetate/Petroleum ether = 1:3) to give the corresponding product.



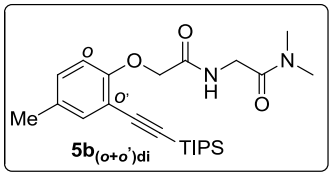
***N,N*-dimethyl-2-(2-(2-((triisopropylsilyl)ethynyl)phenoxy)acetamido)acetamide,** White solid. ^{1}H NMR (400 MHz,$CDCl_3$) δ 7.57 (brs, 1H, NH), 7.45 (dd, J_1 = 7.6 Hz, J_2 = 1.4 Hz, 1H, ArH), 7.30–7.20 (m, 1H, ArH), 6.95 (t, J = 7.3 Hz, 1H, ArH), 6.87 (d, J = 8.3 Hz, 1H, ArH), 4.59 (s, 2H, CH_2), 4.10 (d, J = 4.7 Hz, 2H, CH_2), 2.96 (d, J = 9.3 Hz, 6H, CH_3), 1.17–1.03 (m, 21H, $CH(CH_3)_2$).^{13}C NMR (101 MHz, $CDCl_3$) δ 168.48, 167.22, 158.29, 134.54, 129.88, 122.22, 114.16, 113.95, 102.55, 95.85, 68.69, 40.68, 35.97, 35.56, 18.71, 11.34. HRMS Calcd for $C_{23}H_{36}N_2O_3Si$ $[M+H]^+$: 417.2573; Found: 417.2561.



2-(2-(2,6-bis((triisopropylsilyl)ethynyl)phenoxy)acetamid-o)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400MHz, $CDCl_3$) δ 7.90 (brs, 1H, NH), 7.40 (d, J = 7.7 Hz, 2H, ArH), 7.01 (t, J = 7.7 Hz, 1H, ArH), 4.69 (s, 2H, CH_2), 4.06 (d, J = 4.1 Hz, 2H, CH_2), 2.98 (d, J = 4.8 Hz, 6H, CH_3), 1.12–1.03 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.24, 167.04, 159.14, 134.36, 124.32, 118.08, 101.64, 96.65, 71.13, 40.90, 35.95, 35.48, 18.65, 11.28. HRMS Calcd for $C_{34}H_{56}N_2O_3Si_2$ $[M+H]^+$: 597.3908; Found:597.3901.

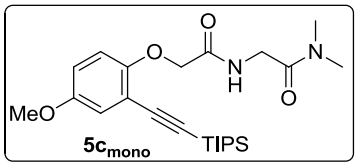


***N,N*-dimethyl-2-(2-(4-methyl-2-((triisopropylsilyl)ethyny)phenoxy)acetamido)acetamide,**
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (brs, 1H, NH), 7.26 (s, 1H, ArH), 7.05 (dd, J_1 = 8.4 Hz, J_2 = 1.9 Hz, 1H, ArH), 6.78 (d, J = 8.4 Hz, 1H, ArH), 4.57 (s, 2H, CH_2), 4.10 (d, J = 4.7 Hz, 2H, CH_2), 2.98 (d, J = 8.8 Hz, 6H, CH_3), 2.25 (s, 3H, CH_3), 1.18–1.00 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.79, 167.34, 156.43, 134.87, 131.85, 130.53, 114.44, 114.01, 102.82, 95.46, 69.15, 40.76, 36.06, 35.65, 20.42, 18.78, 11.42. HRMS Calcd for $C_{24}H_{38}N_2O_3Si$ $[M+H]^+$: 431.2730; Found: 431.2715.



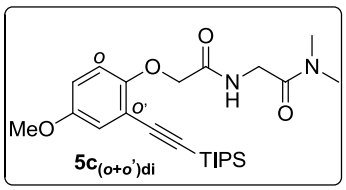
5b$_{(o+o')di}$

***N,N*-dimethyl-2-(2-(4-methyl-2,6-bis((triisopropylsil-yl)ethynyl)phenoxy)acetamido)acetamid e,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (brs, 1H, NH), 7.23–7.17 (m, 2H, ArH), 4.65 (s, 2H, CH_2), 4.06 (d, *J* = 4.1 Hz, 2H, CH_2), 2.99 (d, *J* = 4.6 Hz, 6H, CH_3), 2.25 (s, 3H, CH_3), 1.11–1.02 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.49, 167.17, 157.18, 134.89, 134.05, 117.73, 101.95, 96.15, 71.27, 40.97, 36.07, 35.59, 20.46, 18.71, 11.36. HRMS Calcd for $C_{35}H_{58}N_2O_3Si_2$ $[M+H]^+$: 611.4064; Found:611.4057.



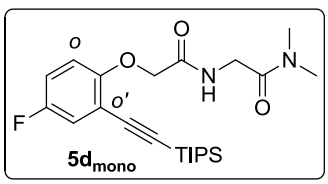
5c$_{mono}$

2-(2-(4-methoxy-2-((triisopropylsilyl)ethynyl)phenoxy)acetamido)-*N,N*-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (brs, 1H, NH), 6.94 (d, *J* = 3.0 Hz, 1H, ArH), 6.85 (d, *J* = 9.0 Hz, 1H, ArH), 6.78 (dd, J_1 = 9.0 Hz, J_2 = 3.0 Hz, 1H, ArH), 4.53 (s, 2H, CH_2), 4.08 (d, *J* = 4.6 Hz, 2H, CH_2), 3.73 (s, 3H, OCH_3), 2.96 (d, *J* = 7.0 Hz, 6H, CH_3), 1.14–1.03 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.78, 167.32, 154.66, 152.86, 118.86, 116.76, 115.77, 115.31, 102.39, 95.94, 70.06, 55.77, 40.70, 35.97, 35.57, 18.69, 11.32. HRMS Calcd for $C_{24}H_{38}N_2O_4Si$ $[M+H]^+$: 447.2679; Found:447.2668.



5c$_{(o+o')di}$

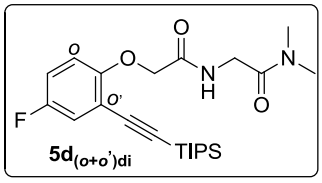
2-(2-(4-methoxy-2,6-bis((triisopropylsilyl)ethynyl)phenoxy)acetamido)-N,N-dimethylacetamide, White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (brs, 1H, NH), 6.91 (s, 2H, ArH), 4.60 (s, 2H, CH_2), 4.05 (d, *J* = 4.0 Hz, 2H, CH_2), 3.76 (s, 3H, OCH_3), 2.98 (d, *J* = 4.1 Hz, 6H, CH_3), 1.12–1.01 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.41, 167.10, 155.40, 153.34, 119.44, 118.69, 101.63, 96.43, 71.39, 55.87, 40.93, 36.02, 35.53, 18.66, 11.31. HRMS Calcd for $C_{35}H_{58}N_2O_4Si_2$ $[M+H]^+$: 627.4013; Found:627.4006.



5d$_{mono}$

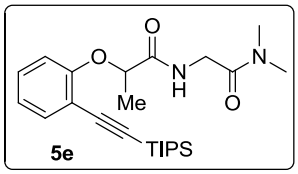
2-(2-(4-fluoro-2-((triisopropylsilyl)ethynyl)phenoxy)acetamido)-*N*,*N*-dimethylacetamide,

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (brs, 1H, NH), 7.13 (dd, J_1 = 8.5 Hz, J_2 = 3.1 Hz, 1H, ArH), 6.99–6.91 (m, 1H, ArH), 6.85 (dd, J_1 = 9.1 Hz, J_2 = 4.5 Hz, 1H, ArH), 4.55 (s, 2H, CH_2), 4.09 (d, J = 4.6 Hz, 2H, CH_2), 2.97 (d, J = 8.0 Hz, 6H, CH_3), 1.16–1.03 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.34, 167.28, 158.73, 156.32, 154.87 ($J_{C\text{-}F}$ = 3 Hz), 120.68 ($J_{C\text{-}F}$ = 24 Hz), 116.55 ($J_{C\text{-}F}$ = 23 Hz),, 116.13 ($J_{C\text{-}F}$ = 9 Hz),, 101.29, 97.46, 69.70, 40.75, 36.00, 35.61, 18.70, 11.32. HRMS Calcd for $C_{23}H_{35}FN_2O_3Si$ $[M+H]^+$: 435.2479; Found: 435.2464.



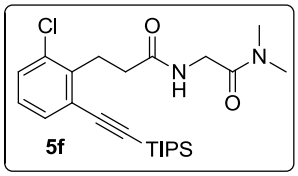
2-(2-(4-fluoro-2,6-bis((triisopropylsilyl)ethynyl)phenoxy)acetamido)-*N*,*N*-dimethylacetamide,

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (brs, 1H, NH), 7.09 (d, J = 8.3 Hz, 2H, ArH), 4.63 (s, 2H, CH_2), 4.05 (d, J = 4.1 Hz, 2H, CH_2), 2.99 (d, J = 3.7 Hz, 6H, CH_3), 1.13–0.99 (m, 42H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.02, 167.08, 158.07 (J = 243 Hz), 155.69(J = 3 Hz), 120.75 (J = 24 Hz), 119.46 (J = 11 Hz), 100.55, 98.12, 71.36, 40.95, 36.02, 35.56, 18.65, 11.29. ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.84. HRMS Calcd for $C_{34}H_{55}FN_2O_3Si_2$ $[M+H]^+$: 615.3813; Found: 615.3817.

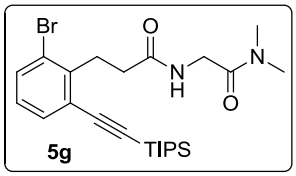


***N*-(2-(dimethylamino)-2-oxoethyl)-2-(2-((triisopropylsilyl)ethynyl)phenoxy)propanamide,**

White solid,. ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.54–7.40 (m, 2H, ArH+NH), 7.26–7.18 (m, 1H, ArH), 6.98–6.89 (m, 1H, ArH), 6.85 (d, J = 8.3 Hz, 1H, ArH), 4.83–4.78 (m, 1H, CH), 4.15–3.94 (m, 2H, CH_2), 2.96 (d, J = 5.1 Hz, 6H, CH_3), 1.60 (d, J = 6.8 Hz, 3H, CH_3), 1.19–1.01 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.49, 167.46, 158.20, 134.44, 129.79, 122.12, 114.89, 102.82, 95.70, 76.49, 40.99, 36.03, 35.66, 18.92, 11.45. HRMS Calcd for $C_{24}H_{38}N_2O_3Si$ $[M+H]^+$: 431.2730; Found: 431.2719.

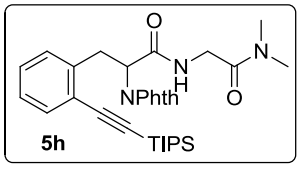


3-(2-chloro-6-((triisopropylsilyl)ethynyl)phenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)propana mide, White solid.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, J_1 = 7.7 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.29 (dd, J_1 = 8.0 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.07 (t, J = 7.9 Hz, 1H, ArH), 6.56 (brs, 1H, NH), 4.04 (d, J = 3.9 Hz, 2H, CH_2), 3.38–3.28 (m, 2H, CH_2), 2.97 (s, 6H, CH_3), 2.59–2.51 (m, 2H, CH_2), 1.13–1.07 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.64, 168.03, 140.73, 134.49, 131.69, 129.87, 127.25, 125.19, 104.29, 95.85, 41.51, 36.02, 35.66, 34.98, 28.23, 18.77, 11.38. HRMS Calcd for $C_{24}H_{37}ClN_2O_2Si$ $[M+H]^+$: 449.2391; Found: 449.2389.



5g

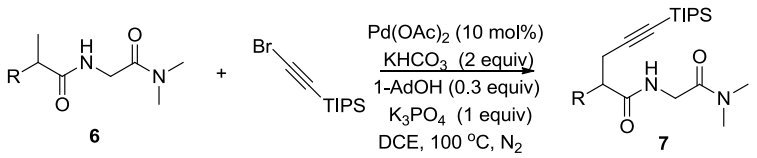
3-(2-bromo-6-((triisopropylsilyl)ethynyl)phenyl)-*N*-(2-(dimethylamino)-2-oxoethyl)propana mide, White solid.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (dd, J_1 = 8.0 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.42 (dd, J_1 = 7.7 Hz, J_2 = 1.1 Hz, 1H, ArH), 7.00 (t, J = 7.9 Hz, 1H, ArH), 6.57 (brs, 1H, NH), 4.05 (d, J = 3.8 Hz, 2H, CH_2), 3.45–3.29 (m, 2H, CH_2), 2.99 (d, J = 1.2 Hz, 6H, CH_3), 2.60–2.50 (m, 2H, CH_2), 1.15–1.03 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.57, 168.05, 142.26, 133.19, 132.35, 127.50, 125.07, 124.75, 104.38, 95.88, 41.49, 36.02, 35.65, 34.94, 30.95, 18.76, 11.36. HRMS Calcd for $C_{24}H_{37}BrN_2O_2Si$ $[M+H]^+$: 493.1886; Found: 493.1891.



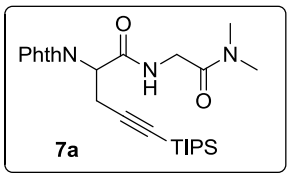
5h

***N*-(2-(dimethylamino)-2-oxoethyl)-2-(1,3-dioxoisoindolin-2-yl)-3-(2-((triisopropylsilyl)et hynyl)phenyl)propanamide,** White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, J_1 = 5.5 Hz, J_2 = 3.0 Hz, 2H, ArH), 7.66 (dd, J_1 = 5.5 Hz, J_2 = 3.1 Hz, 2H, ArH), 7.44 (d, J = 7.4 Hz, 1H, ArH), 7.12–7.02 (m, 2H, ArH), 7.02–6.95 (m, 2H, ArH+NH), 5.47 (dd, J_1 = 11.2 Hz, J_2 = 5.1 Hz, 1H, CH), 4.06 (d, J = 3.8 Hz, 2H, CH_2), 3.90 (dd, J_1 = 13.6 Hz, J_2 = 5.1 Hz, 1H, CH_2), 3.60 (dd, J_1 = 13.6 Hz, J_2 = 11.3 Hz, 1H, CH_2), 2.96 (d, J = 4.9 Hz, 6H, CH_3), 1.21–1.10 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.09, 167.63, 138.88, 134.18, 133.42, 131.64, 129.66, 128.42, 126.95, 123.63, 104.72, 95.96, 53.49, 41.82, 35.98, 35.65, 33.72, 18.82, 11.46. HRMS Calcd for $C_{32}H_{41}N_3O_4Si$[M+H]$^+$: 560.2945; Found:560.2957.

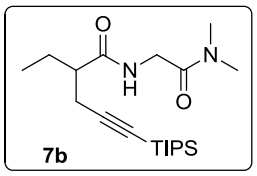
8. General procedures for the alkylation of alkyl acid



A mixture of 6 (0.2 mmol), 2 (0.3 mmol), $Pd(OAc)_2$ (2.2 mg, 5 mol%), K_2CO_3 (55.2 mg, 0.4 mmol, 2 equiv), PivOH (6 mg, 0.06 mmol) and DCE (1 mL) in a 15 mL sealed glass vial was heated at 100 ^oC with vigorous stirring for 18 hours. The reaction mixture was cooled to room temperature, and diluted with ethyl acetate and filtered through celite. The filtrate was concentrated in vacuo and purified by column chromatography on silica gel (Ethyl acetate/Petroleum ether = 1:3) to give the corresponding product.

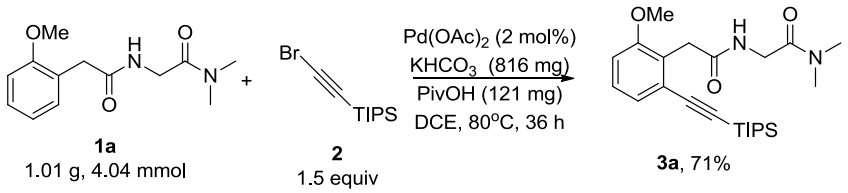


***N*-(2-(dimethylamino)-2-oxoethyl)-2-(1,3-dioxoisoindolin-2-yl)-5-(triisopropylsilyl)pent-4-ynamide,** White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 5.4, 3.0 Hz, 2H, ArH), 7.73 (dd, J_1 = 5.5 Hz, J_2 = 3.0 Hz, 2H, ArH), 7.12 (brs, 1H, NH), 5.08 (dd, J_1 = 11.7 Hz, J_2 = 5.1 Hz, 1H, CH), 4.03 (d, J = 3.8 Hz, 2H, CH_2), 3.41 (dd, J_1 = 17.3 Hz, J_2 = 11.7 Hz, 1H, CH_2), 3.13 (dd, J_1 = 17.3 Hz, J_2 = 5.1 Hz, 1H, CH_2), 2.94 (s, 3H, CH_3), 2.93 (s, 3H, CH_3), 0.84–0.75 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.08, 166.89, 166.83, 133.78, 131.32, 123.21, 102.41, 83.18, 52.10, 41.09, 35.37, 35.04, 20.00, 17.80, 10.49. HRMS Calcd for $C_{26}H_{37}N_3O_4Si$ $[M+H]^+$: 484.2632; Found: 484.2637.



***N*-(2-(dimethylamino)-2-oxoethyl)-2-ethyl-5-(triisopropylsil-yl)pent-4-ynamide,** White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 6.76 (brs, 1H, NH), 4.08–3.99 (m, 2H, CH_2), 3.02–2.92 (m, 6H, CH_3), 2.62–2.47 (m, 1H, CH), 2.47–2.26 (m, 2H, CH_2), 1.66 (dd, J_1 = 14.9 Hz, J_2 = 7.5 Hz, 2H, CH_2), 1.06–0.96 (m, 21H, $CH(CH_3)_2$), 0.90 (t, J = 7.4 Hz, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.61, 167.36, 105.68, 81.28, 47.83, 40.73, 35.37, 35.02, 24.48, 22.49, 18.05, 11.20, 10.68. HRMS Calcd for $C_{20}H_{38}N_2O_2Si$ $[M+H]^+$: 367.2781; Found: 367.2788.

9. Gram scale reaction



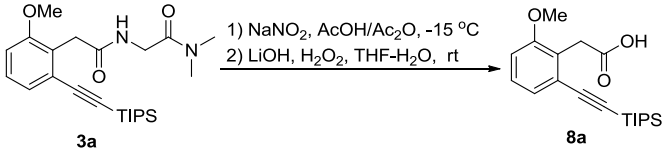
1a
1.01 g, 4.04 mmol

2
1.5 equiv

3a, 71%

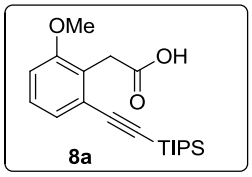
A mixture of 1a (4.04 mmol, 1.01 g), 2 (1.5 equiv, 1.6 g), $Pd(OAc)_2$ (17.8 mg, 2 mol%), $KHCO_3$ (816 mg, 2 equiv), PivOH (121 mg, 0.3 equiv) and DCE (20 mL) in a 50 mL sealed glass vial was heated at 80 ^oC with vigorous stirring for 36 hours. The reaction mixture was cooled to room temperature, and diluted with ethyl acetate and filtered through celite. The filtrate was concentrated in vacuo and purified by column chromatography on silica gel (Ethyl acetate/Petroleum ether = 1:3) to give the corresponding product (71%).

10. The removal directing group



3a **8a**

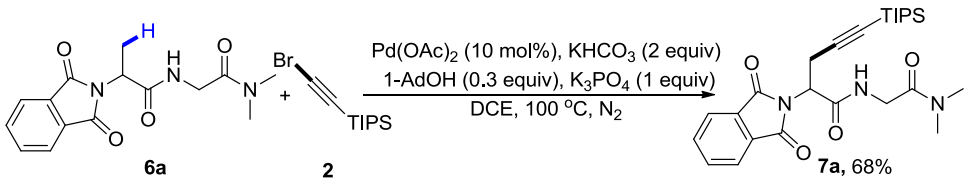
A mixture of the product 3a (0.5 mmol, 1.0 equiv) and $AcOH:Ac_2O$ (4:1, 5 mL) in a 15 mL glass vial (under air atmosphere, sealed with PTFE cap) was stired at -15 ^oC for 5 minutes. Then the $NaNO_2$ (138 mg, 2 qeuiv) was added and the mixture was stired 2 hours. The sodium hydroxide solution and hydrochloric acid was added and the mixture was extracted with DCM. The LiOH (4 equiv), H_2O_2 (1 mL), and $THF:H_2O$ (1:1, 4 mL) were added the organic layer. The mixture was stired at room temperature for 12 hours. Water was added and the mixture was extracted with DCM. The combined organic layer was washed with water and brine, dried over Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to give the product **8a**.

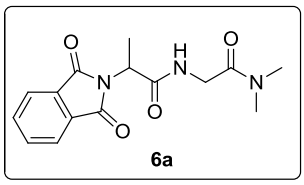


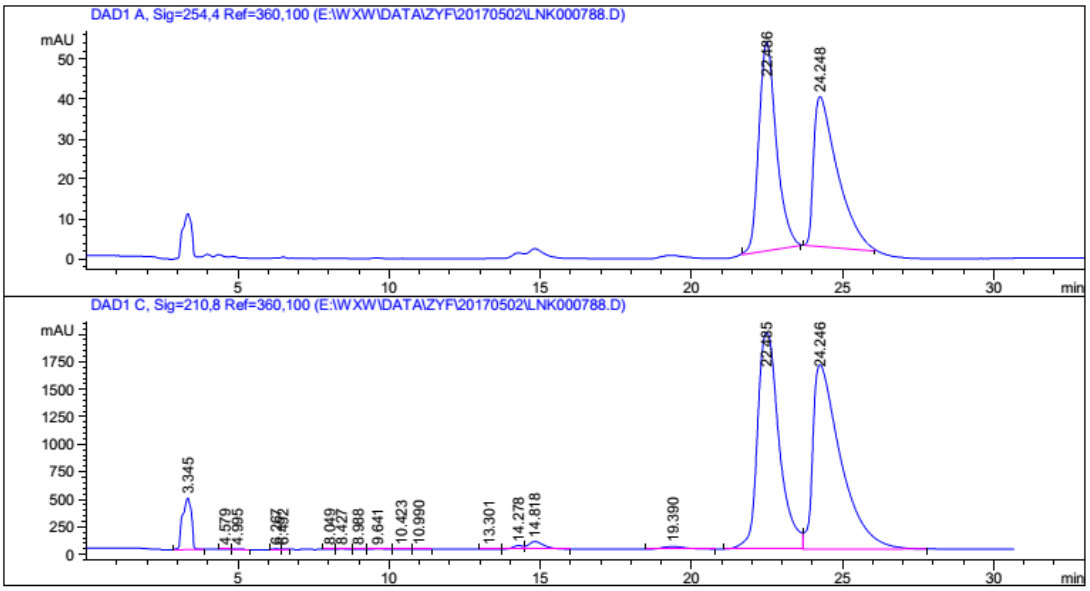
8a

2-(2-methoxy-6-((triisopropylsilyl)ethynyl)phenyl)acetic acid, White solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 8.0 Hz, 1H, ArH), 7.12 (dd, J_1 = 7.7 Hz, J_2 = 1.0 Hz, 1H, ArH), 6.86 (d, J = 7.7 Hz, 1H, ArH), 3.96 (s, 2H, CH_2), 3.82 (s, 3H, OCH_3), 1.18–1.04 (m, 21H, $CH(CH_3)_2$). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.14, 157.06, 127.68, 124.73, 124.34, 110.40, 104.15, 94.79, 55.25, 33.07, 18.15, 10.76. HRMS Calcd for $C_{20}H_{30}O_3Si$ $[M+H]^+$: 347.2042; Found: 347.2039.

11. HPLC spectra of 6a and 7a





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Dilution:                     :      1.0000
Sample Amount:                :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
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   2  24.248 BB    0.7869 2070.56665   37.57566  49.5076

Totals :                  4182.32495   89.80392
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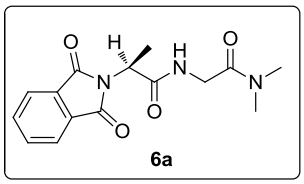
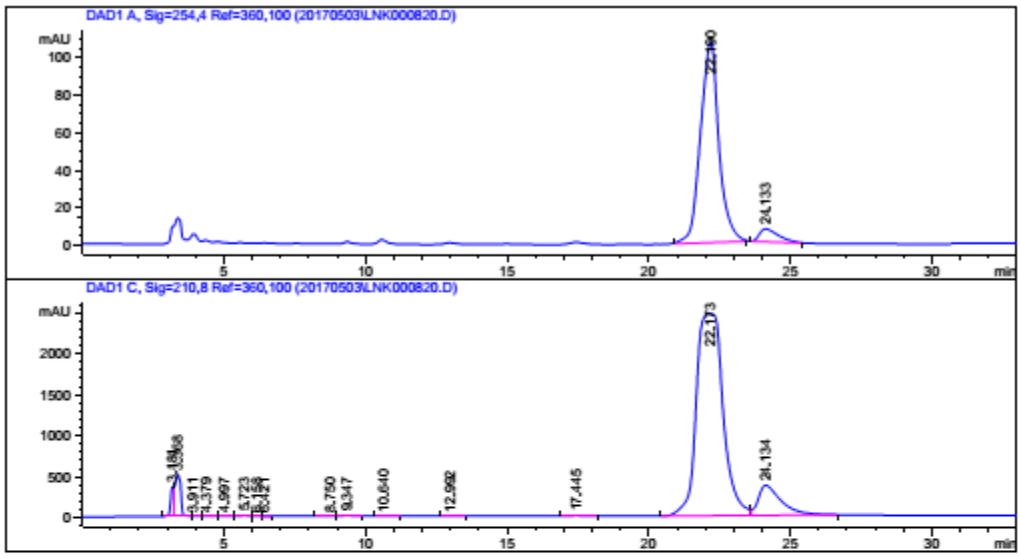


6a

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Dilution:                     :      1.0000
Sample Amount:                :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
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Totals :                  5147.02658  113.97777
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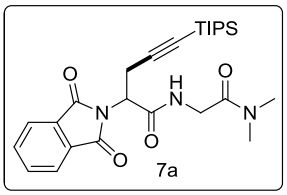

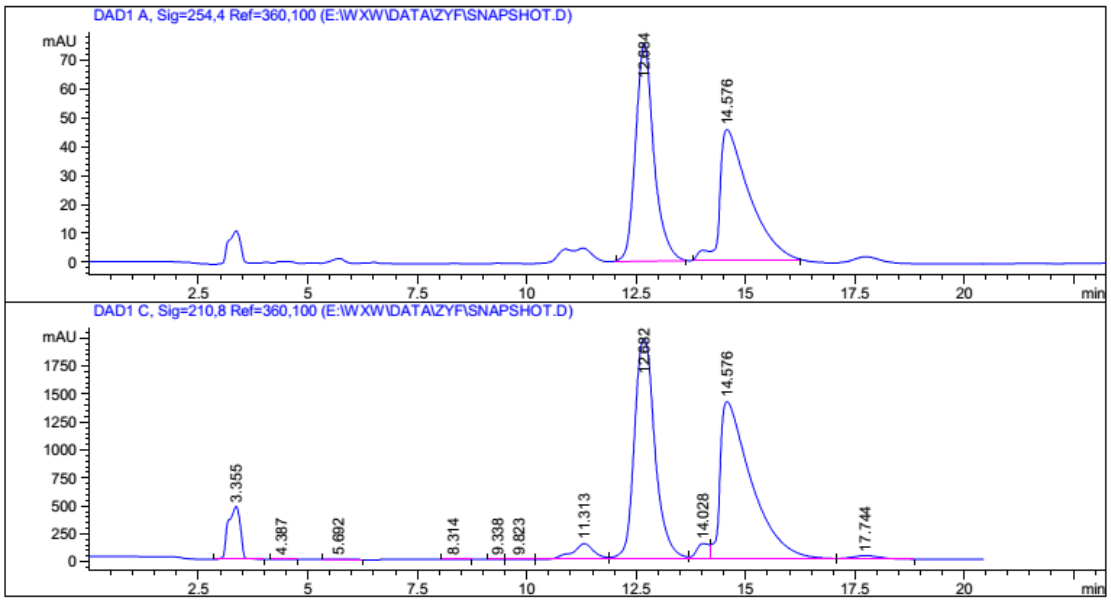


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=====================================================================
                    Area Percent Report
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Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
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   1  12.684 BB    0.4402 2203.13208   75.55520  50.3448
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Totals :                  4376.09058  121.02712
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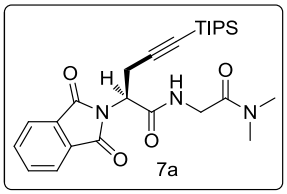
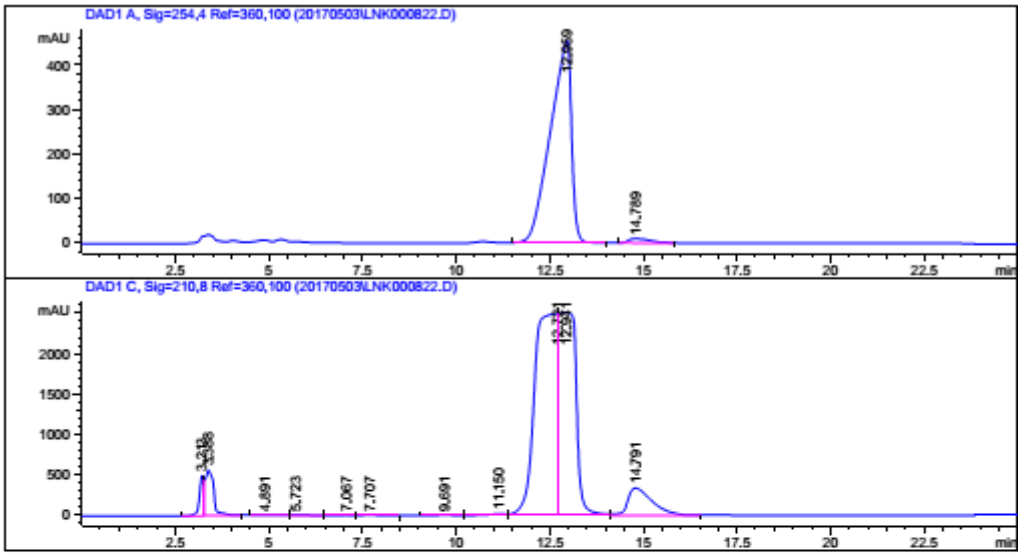


7a

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=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Sample Amount:                :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
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   2  14.789 BB    0.6092  410.73077   10.00046   2.2456

Totals :                  1.82906e4   465.70099
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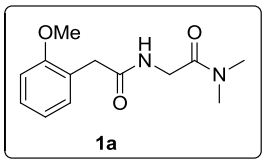
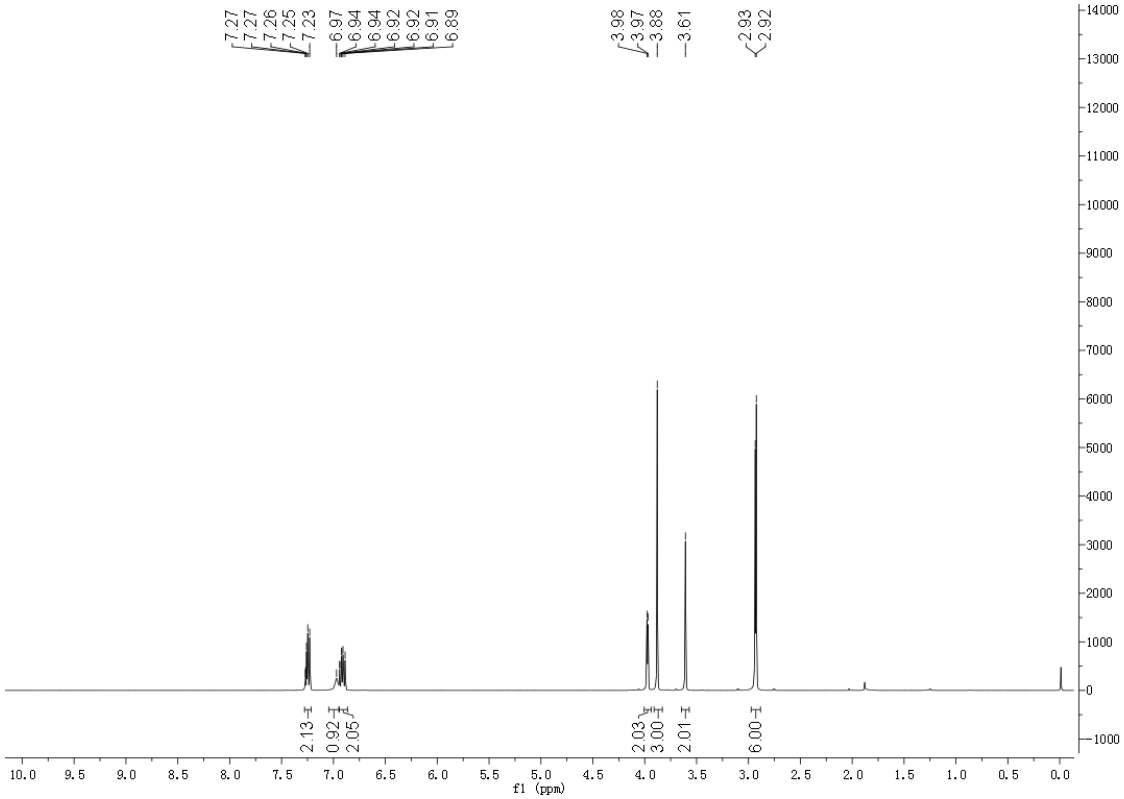
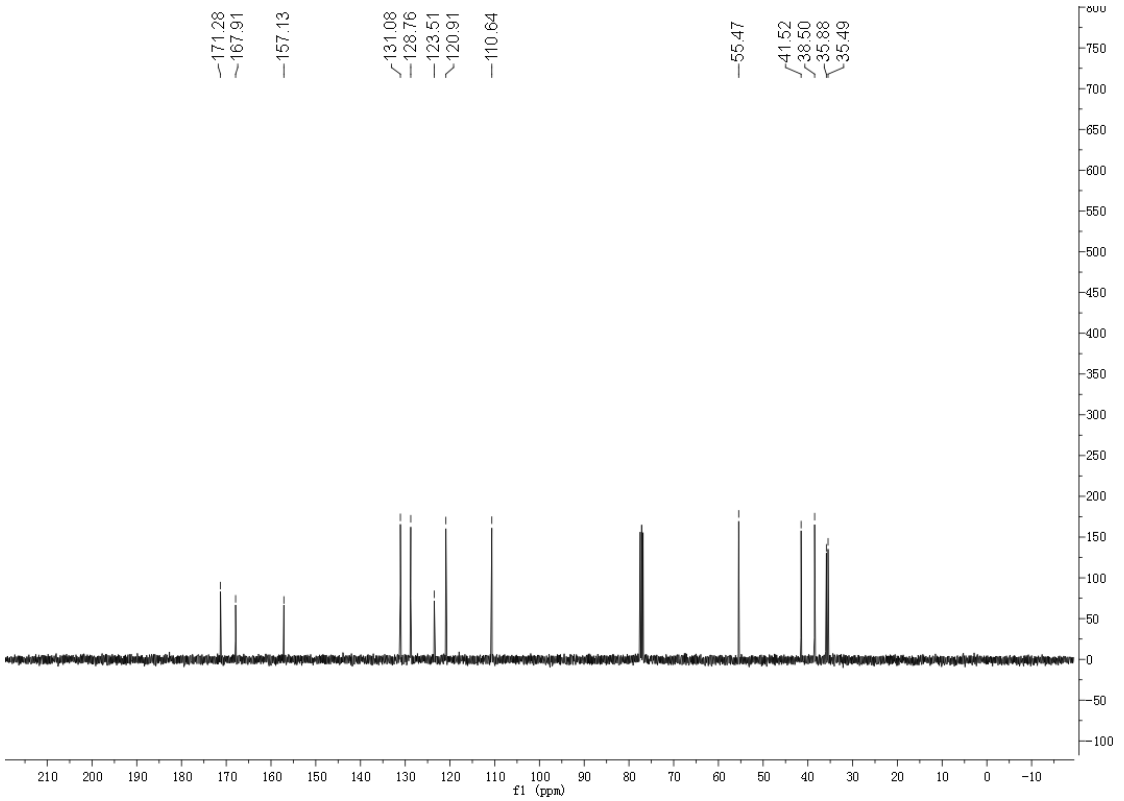
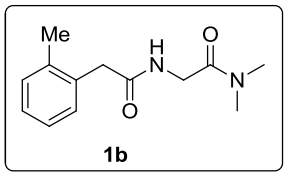
12. Spectroscopic Data of All New Compounds



1a



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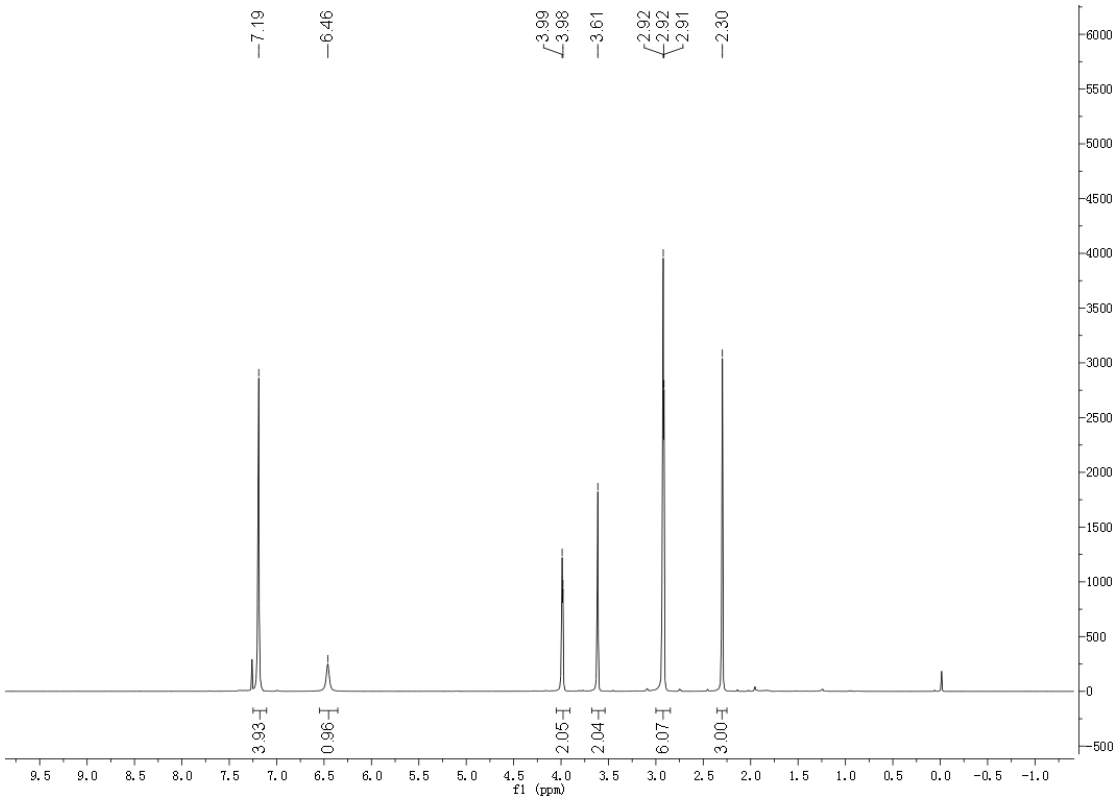
H
N
O
O
N

1b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2.92
2.92
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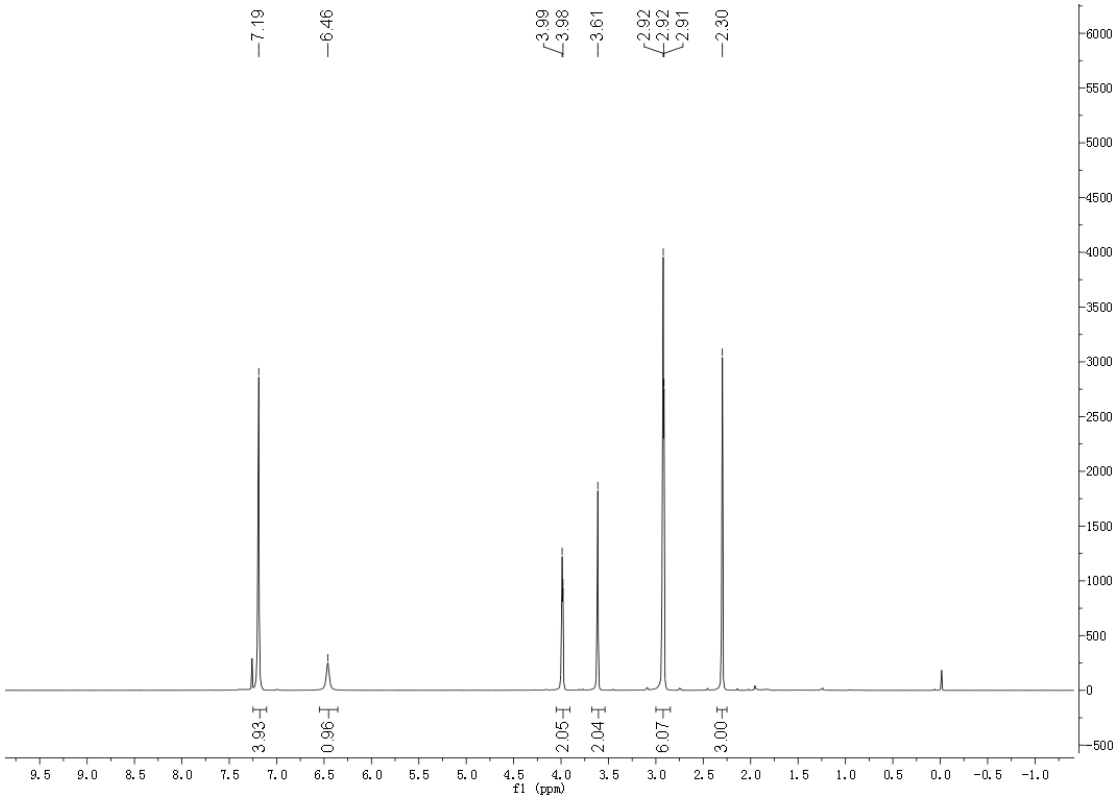
3.93
0.96
2.05
2.04
6.07
3.00
f1 (ppm)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6.46
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3.93
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2.04
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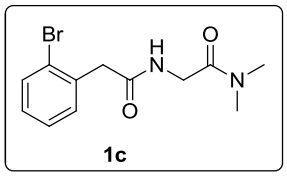
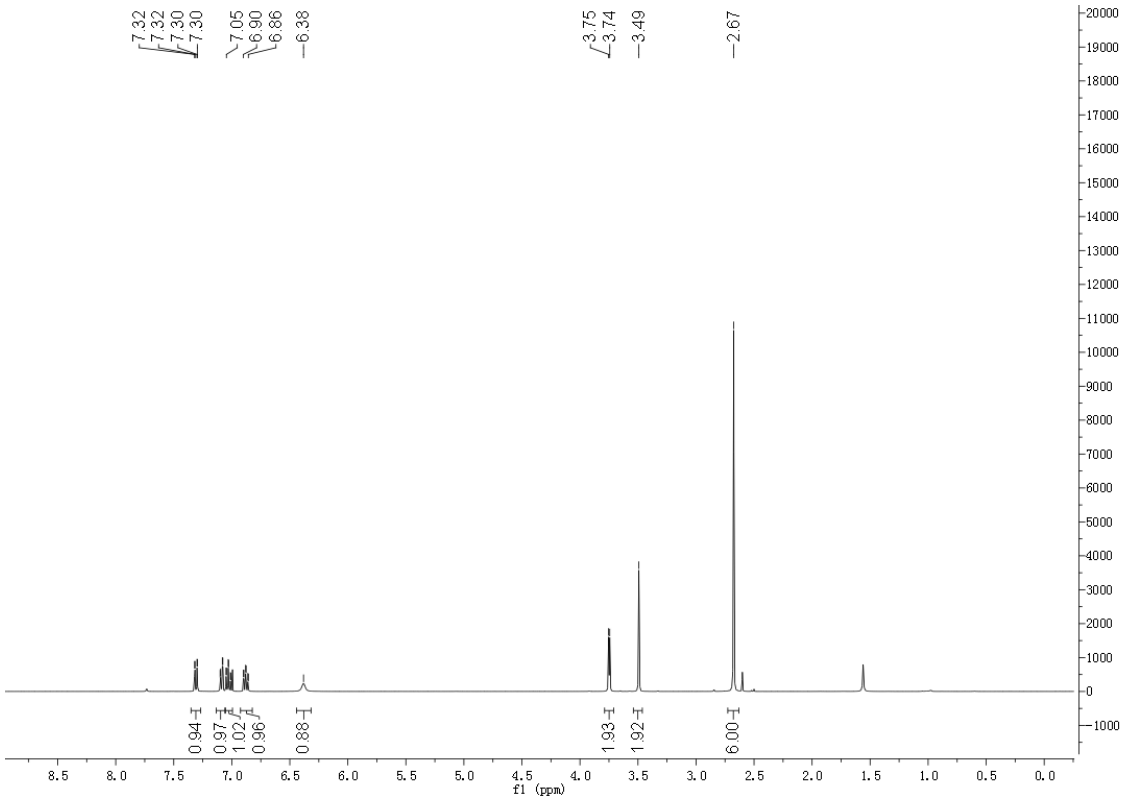
3.00
f1 (ppm)


Br
H
N
O
O
N

1c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1.02
0.96
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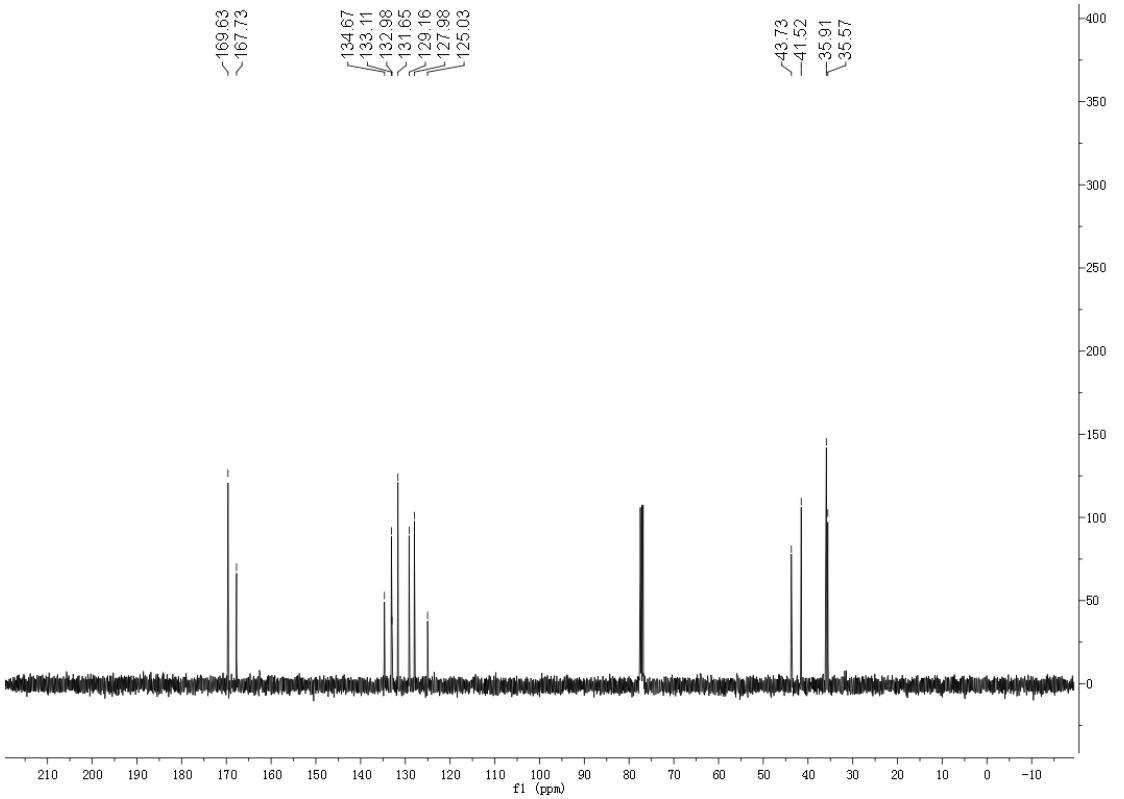
1.92
6.00
f1 (ppm)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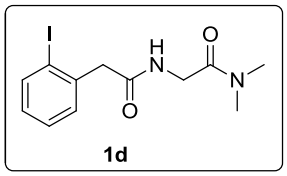
f1 (ppm)


I
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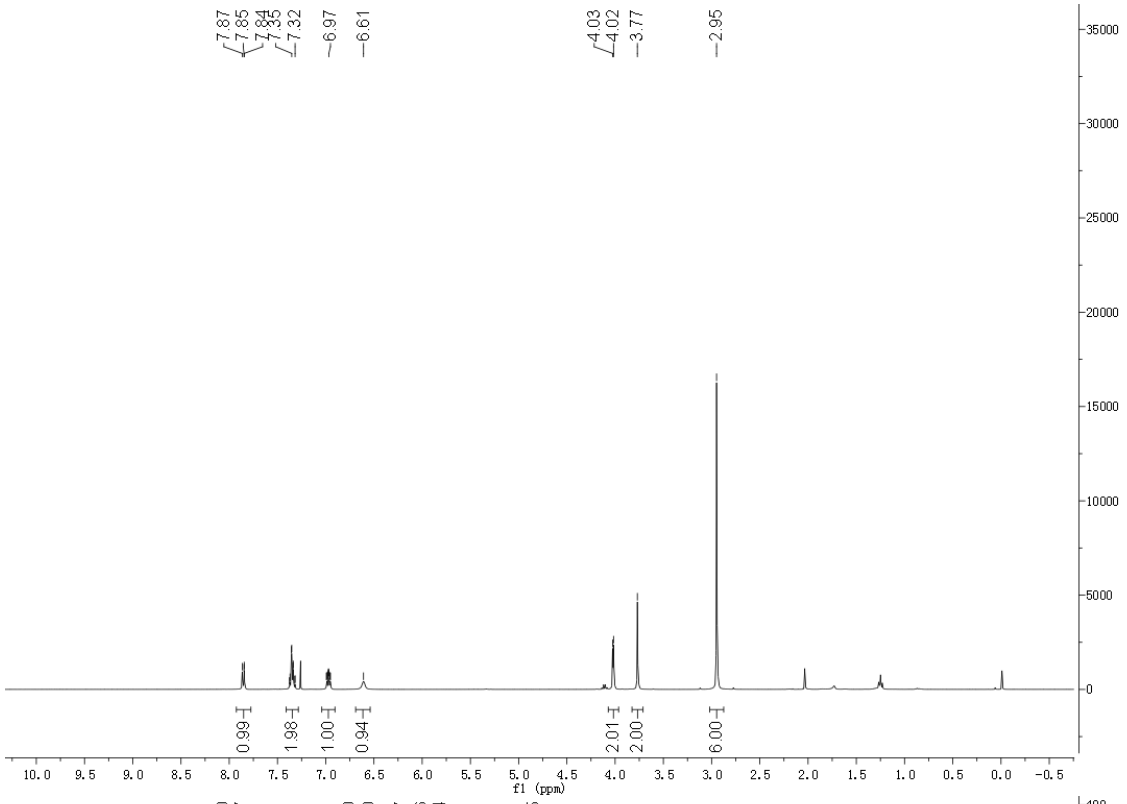
N
O
O
N
1d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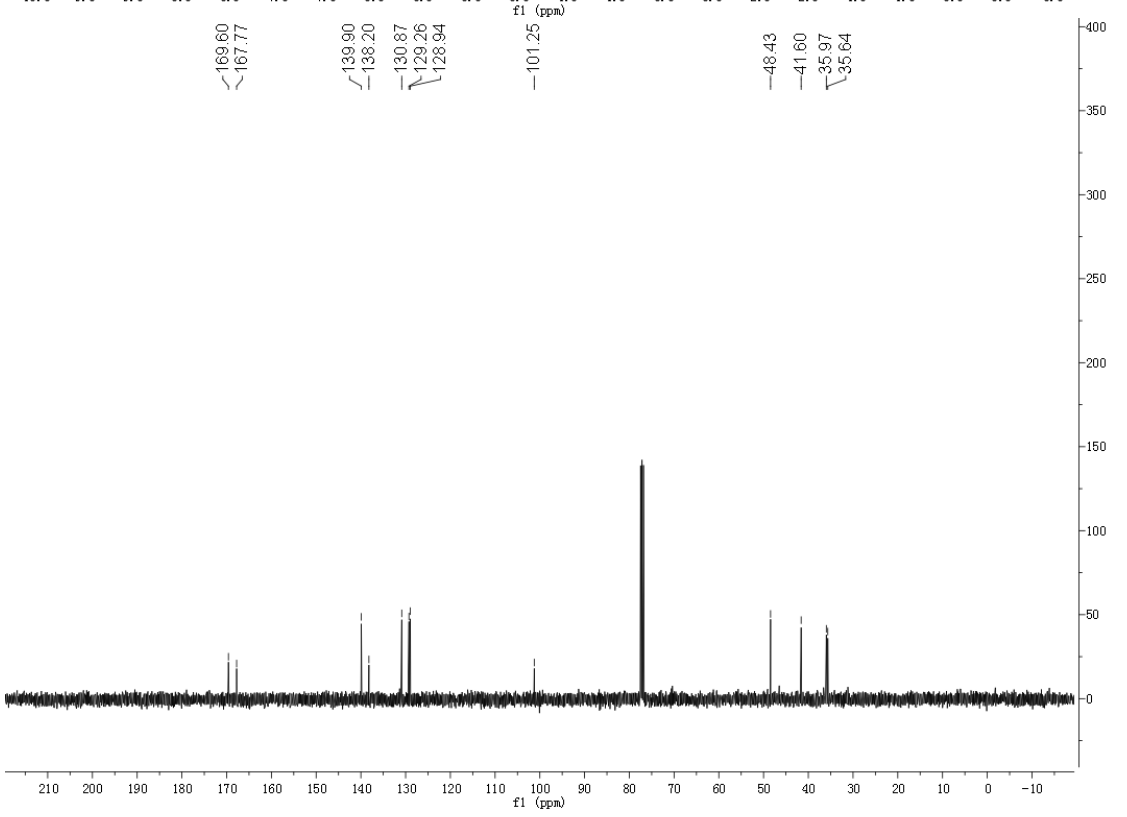
6.00
f1 (ppm)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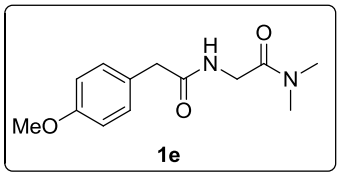
f1 (ppm)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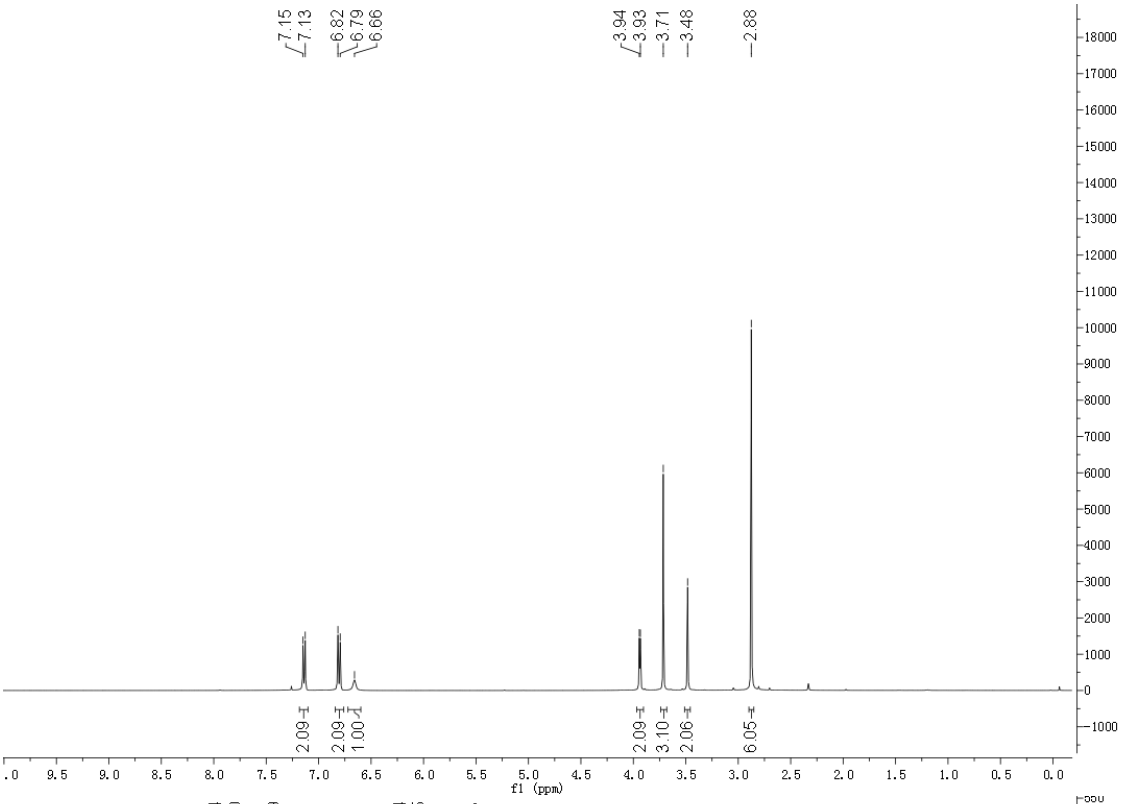
O
N
1e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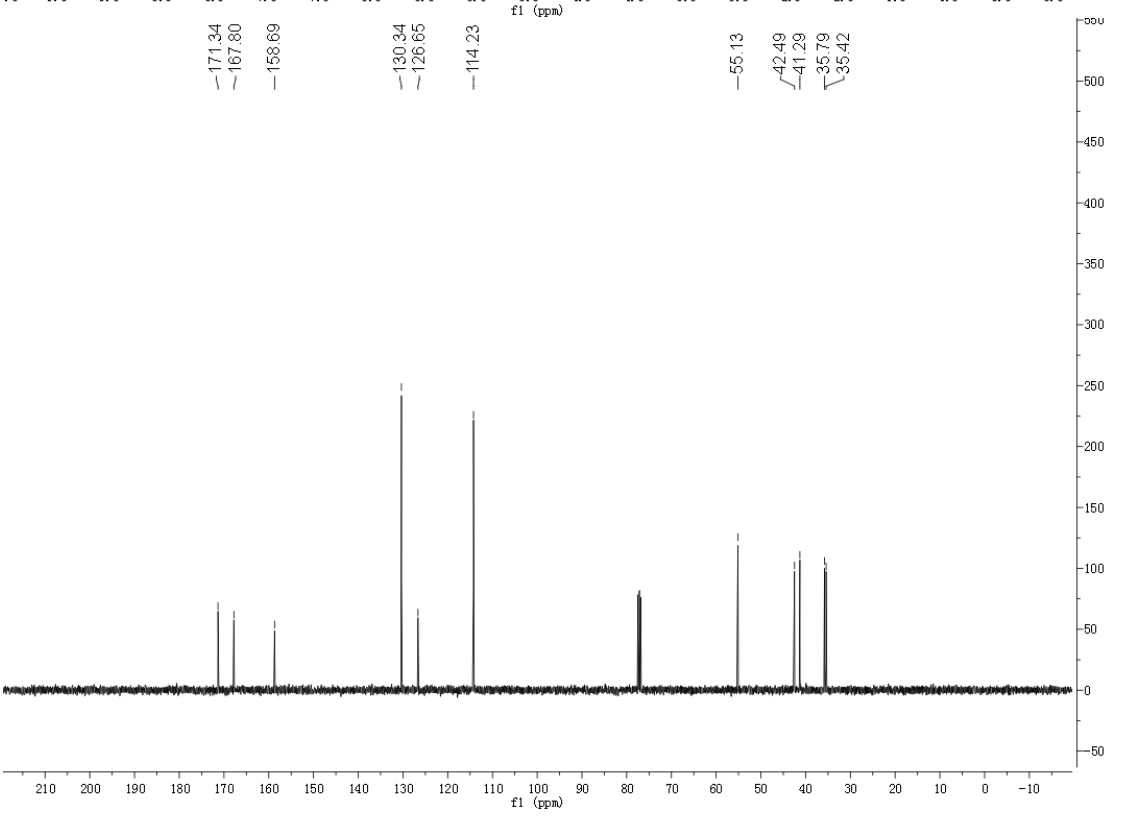
f1 (ppm)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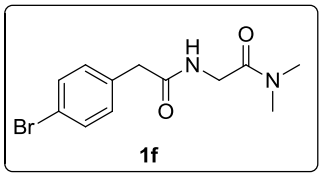
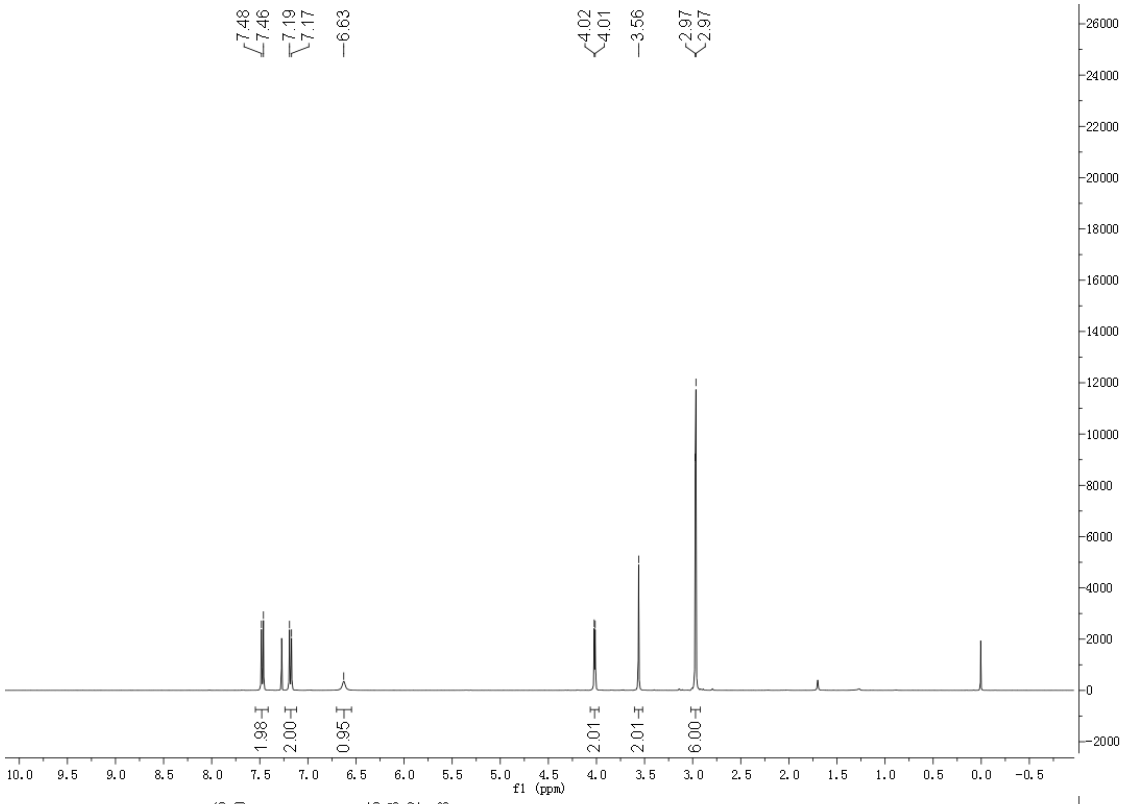
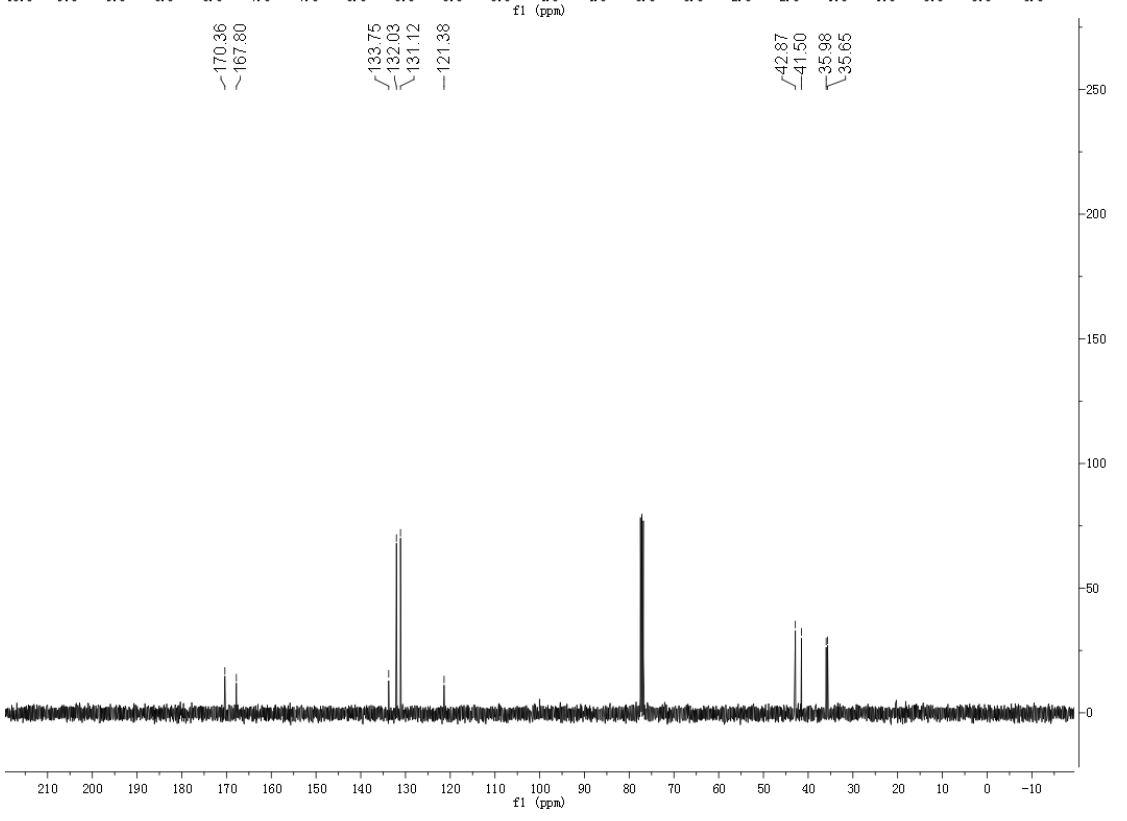
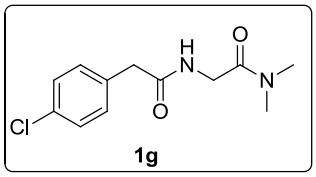

H
N
O
O
N
Br

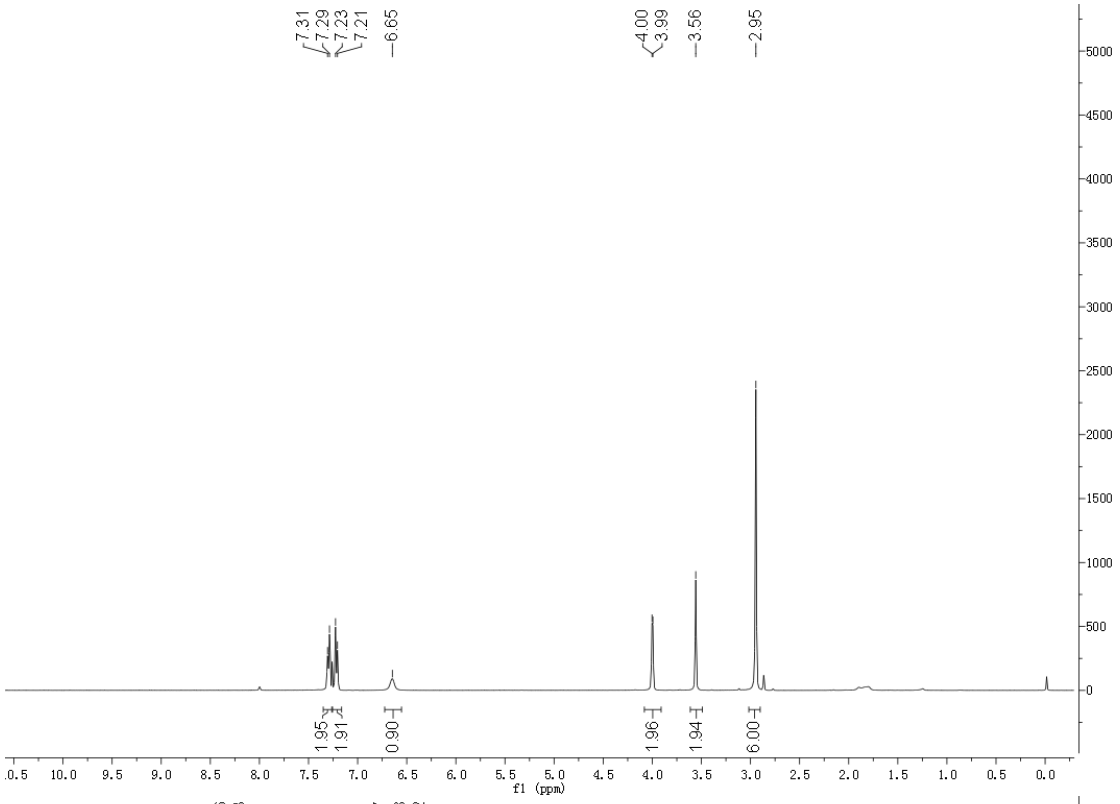
1f

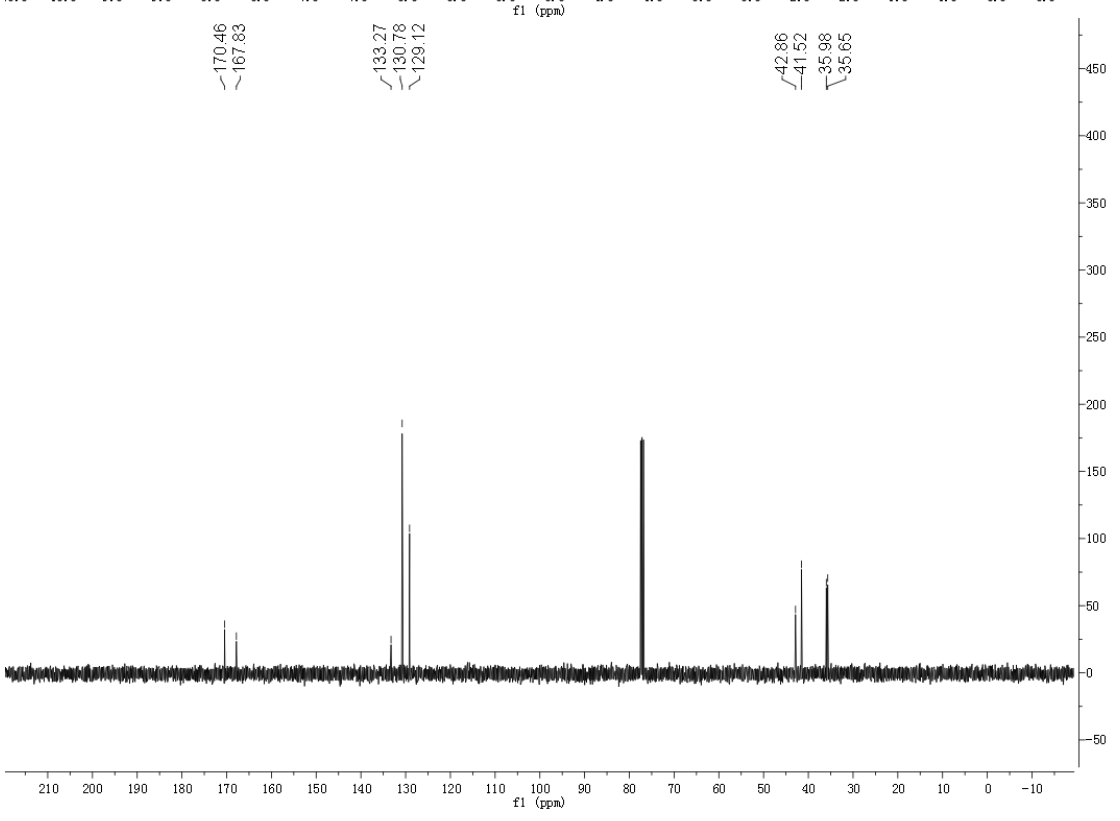

7.48
7.46
7.19
7.17
6.63
4.02
4.01
3.56
2.97
2.97
1.98
2.00
0.95
2.01
2.01
6.00
f1 (ppm)


170.36
167.80
133.75
132.03
131.12
121.38
42.87
41.50
35.98
35.65
f1 (ppm)


H
N
O
O
N
Cl

1g


7.31
7.29
7.23
7.21
6.65
4.00
3.99
3.56
2.95
1.95
1.91
0.90
1.96
1.94
6.00
f1 (ppm)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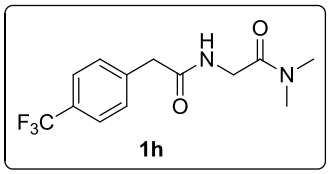
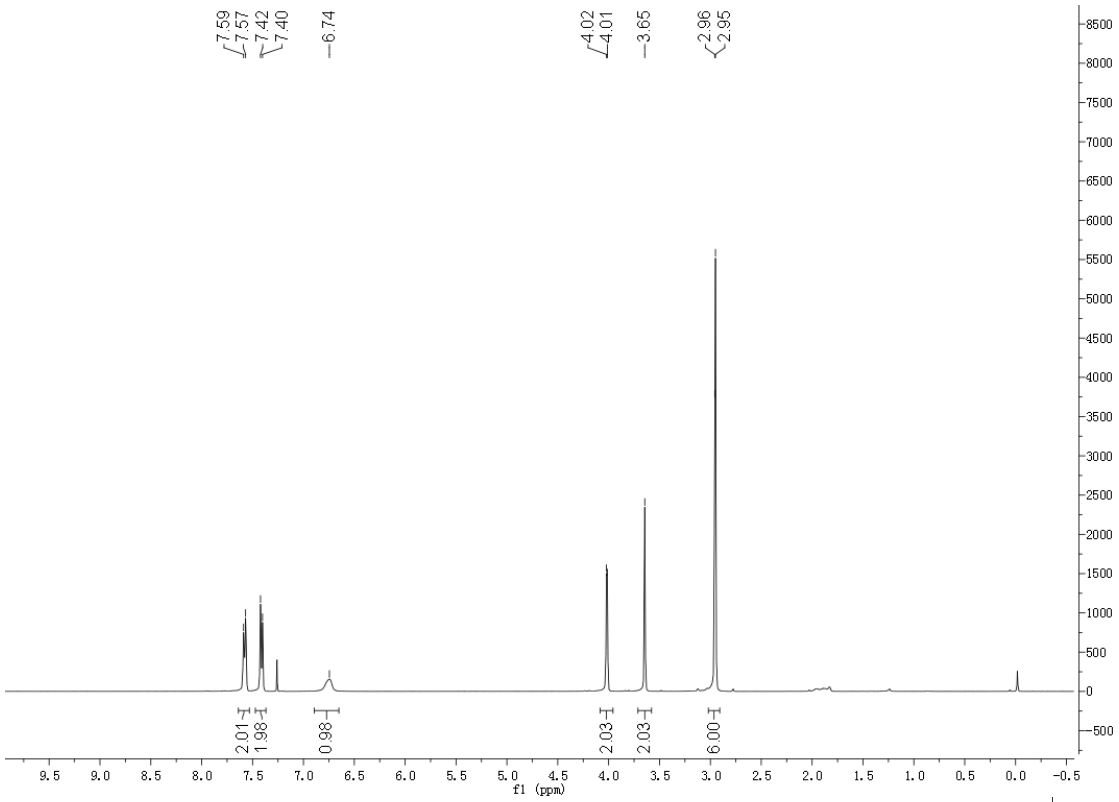
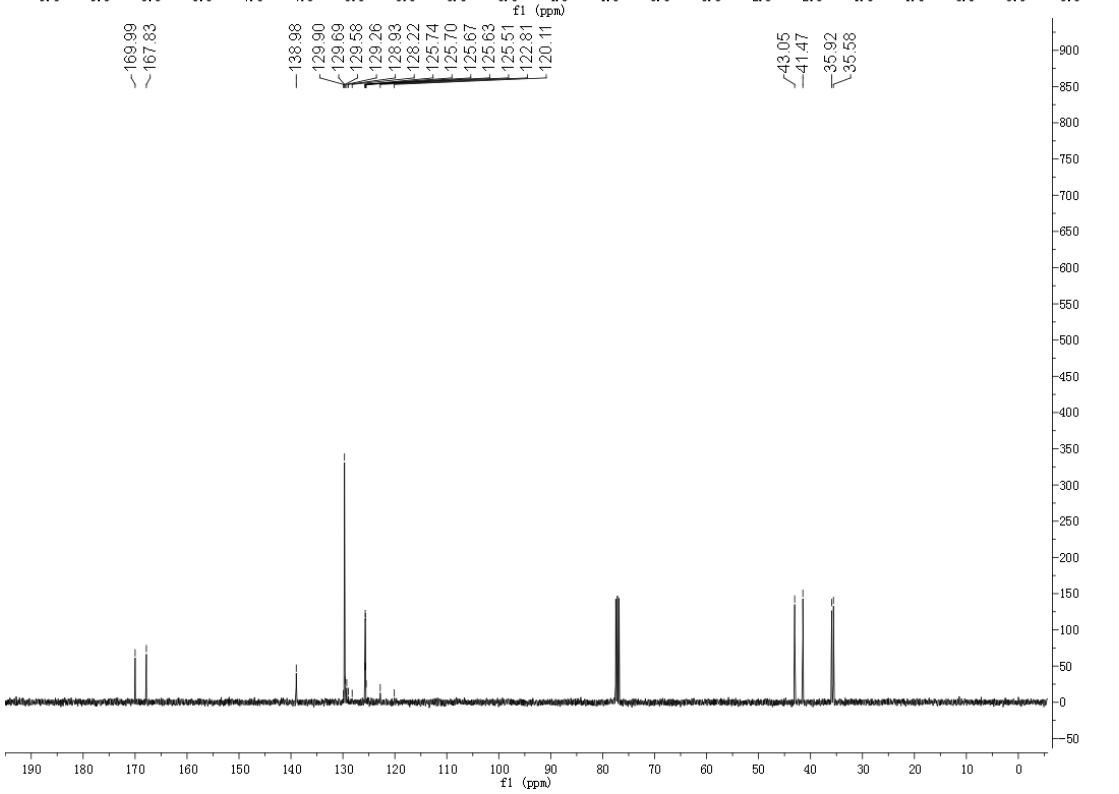
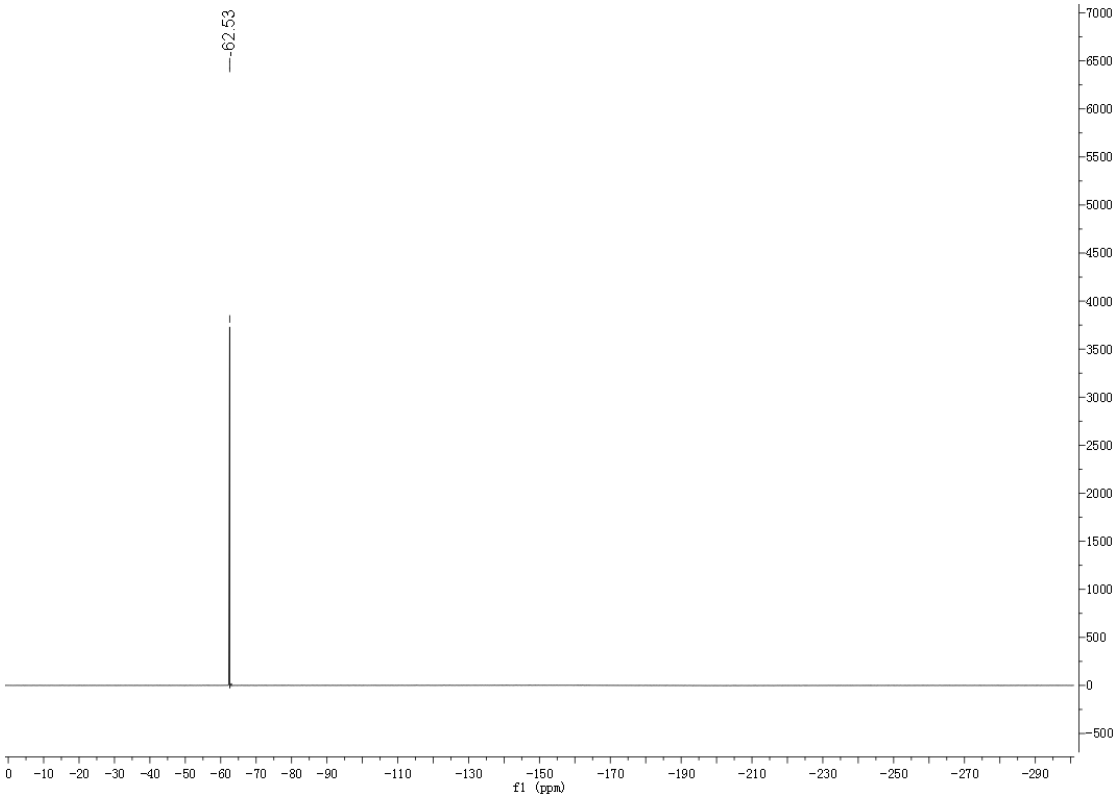
167.83
133.27
130.78
129.12
42.86
41.52
35.98
35.65
f1 (ppm)

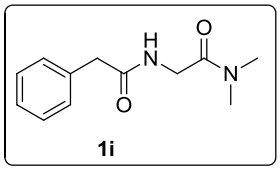

H
N
O
O
N
F3C

1h

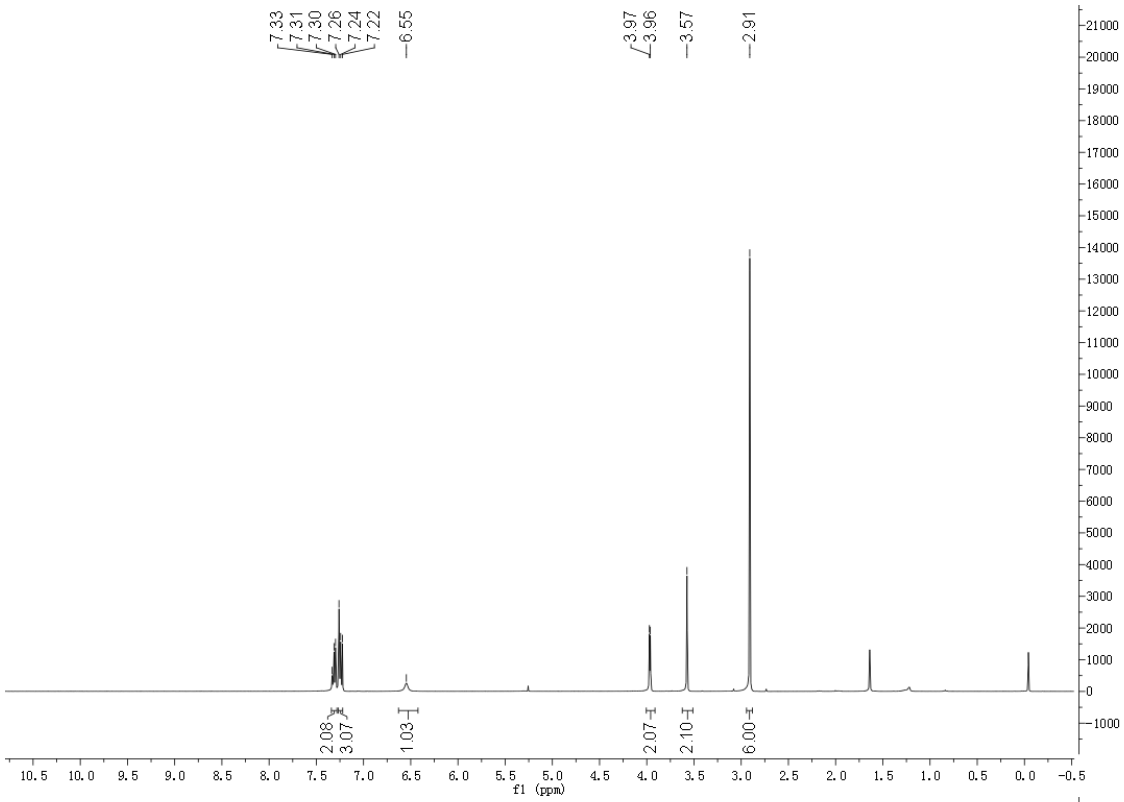

7.59
7.57
7.42
7.40
6.74
4.02
4.01
3.65
2.96
2.95
2.01
1.98
0.98
2.03
2.03
6.00
f1 (ppm)

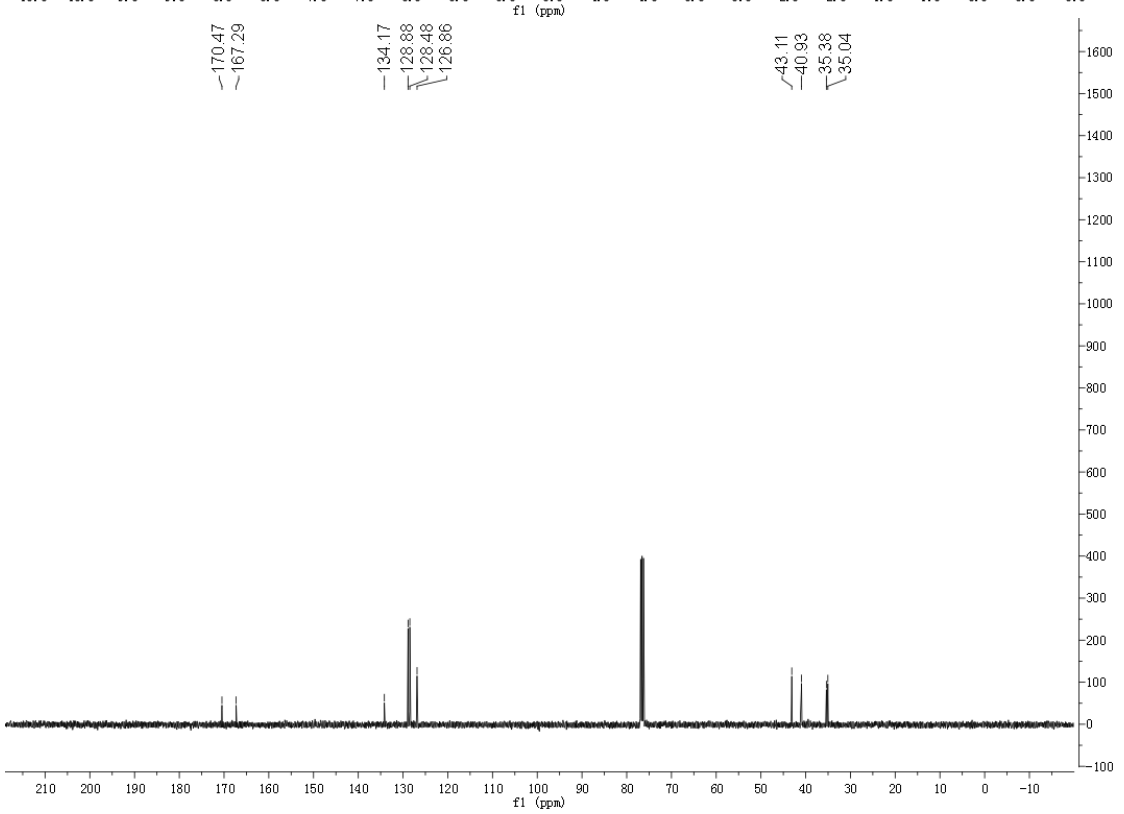

169.99
167.83
138.98
129.90
129.69
129.58
129.26
128.93
128.22
125.74
125.70
125.67
125.63
125.51
122.81
120.11
43.05
41.47
35.92
35.58
f1 (ppm)


-62.53
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)


H
N
O
O
N

1i


7.33
7.31
7.30
7.26
7.24
7.22
6.55
3.97
3.96
3.57
2.91
2.08
3.07
1.03
2.07
2.10
6.00
f1 (ppm)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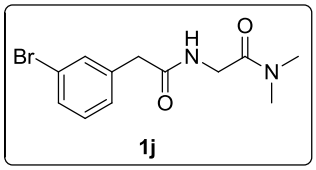
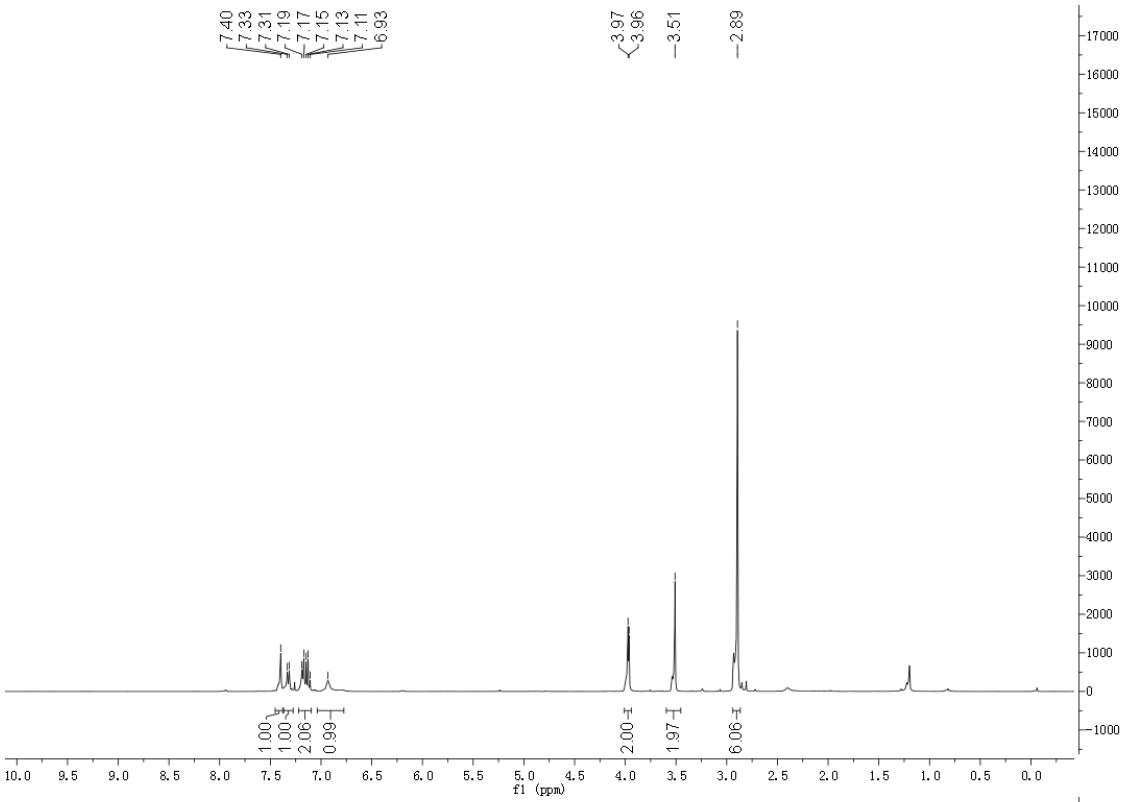
167.29
134.17
128.88
128.48
126.86
43.11
40.93
35.38
35.04
f1 (ppm)


Br
H
N
O
O
N

1j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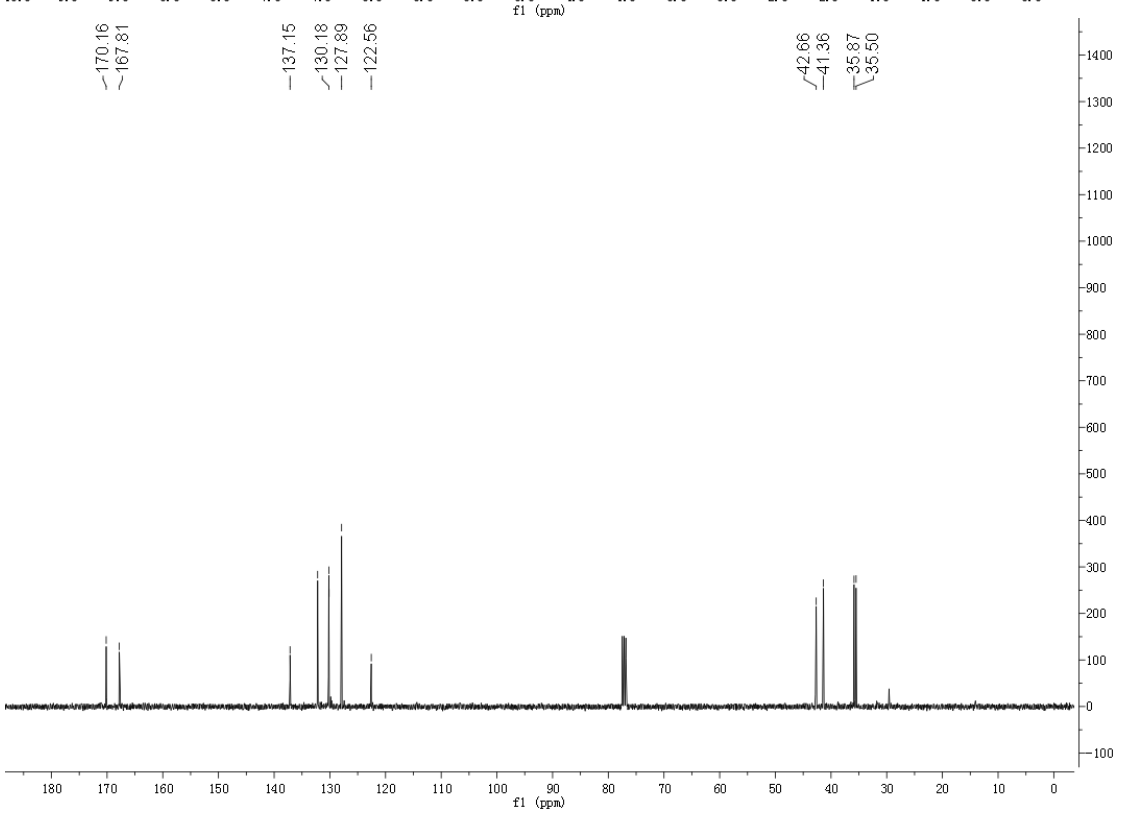
7.33
7.31
7.19
7.17
7.15
7.13
7.11
6.93
3.97
3.96
3.51
2.89
1.00
1.00
2.06
0.99
2.00
1.97
6.06
f1 (ppm)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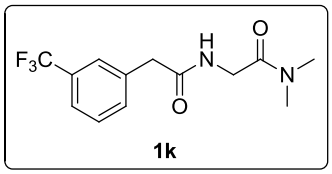
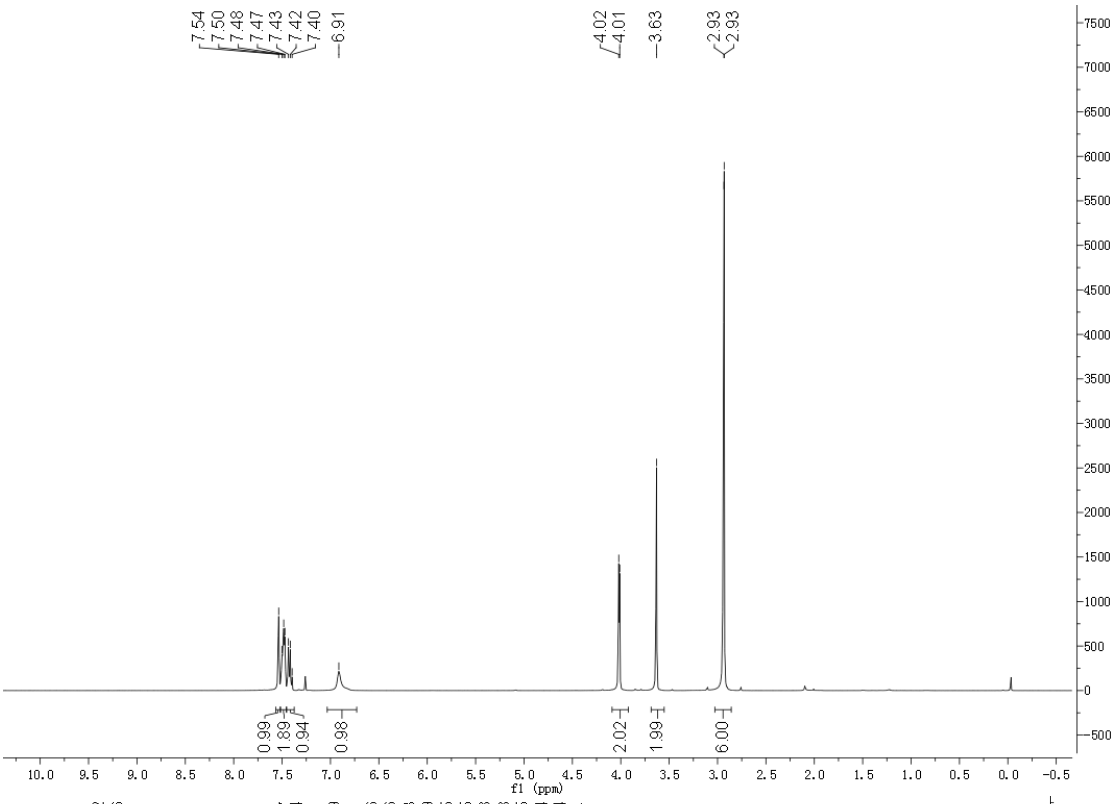
167.81
137.15
130.18
127.89
122.56
42.66
41.36
35.87
35.50
f1 (ppm)


F3C
H
N
O
O
N
1k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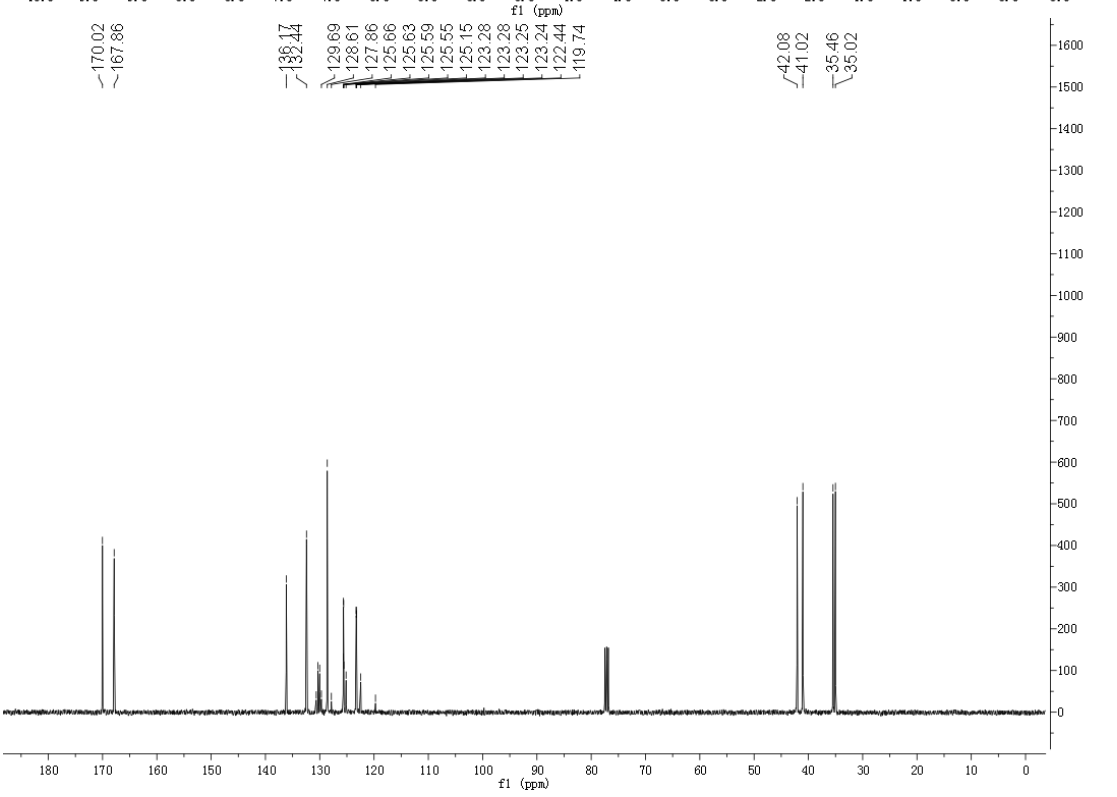
7.50
7.48
7.47
7.43
7.42
7.40
6.91
4.02
4.01
3.63
2.93
2.93
0.99
1.89
0.94
0.98
2.02
1.99
6.00
f1 (ppm)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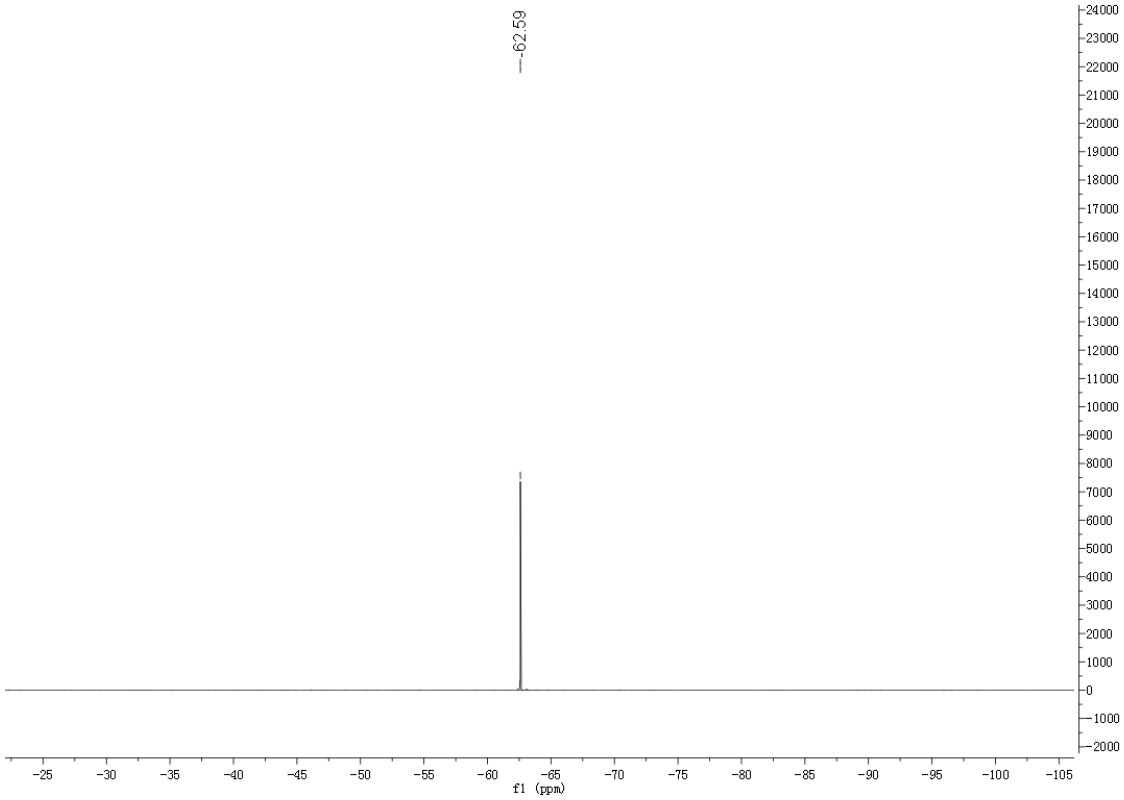
167.86
136.17
132.44
129.69
128.61
127.86
125.66
125.63
125.59
125.55
125.15
123.28
123.28
123.25
123.24
122.44
119.74
42.08
41.02
35.46
35.02
f1 (ppm)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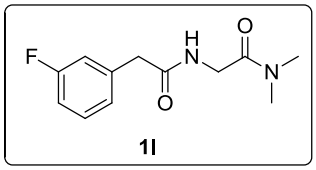
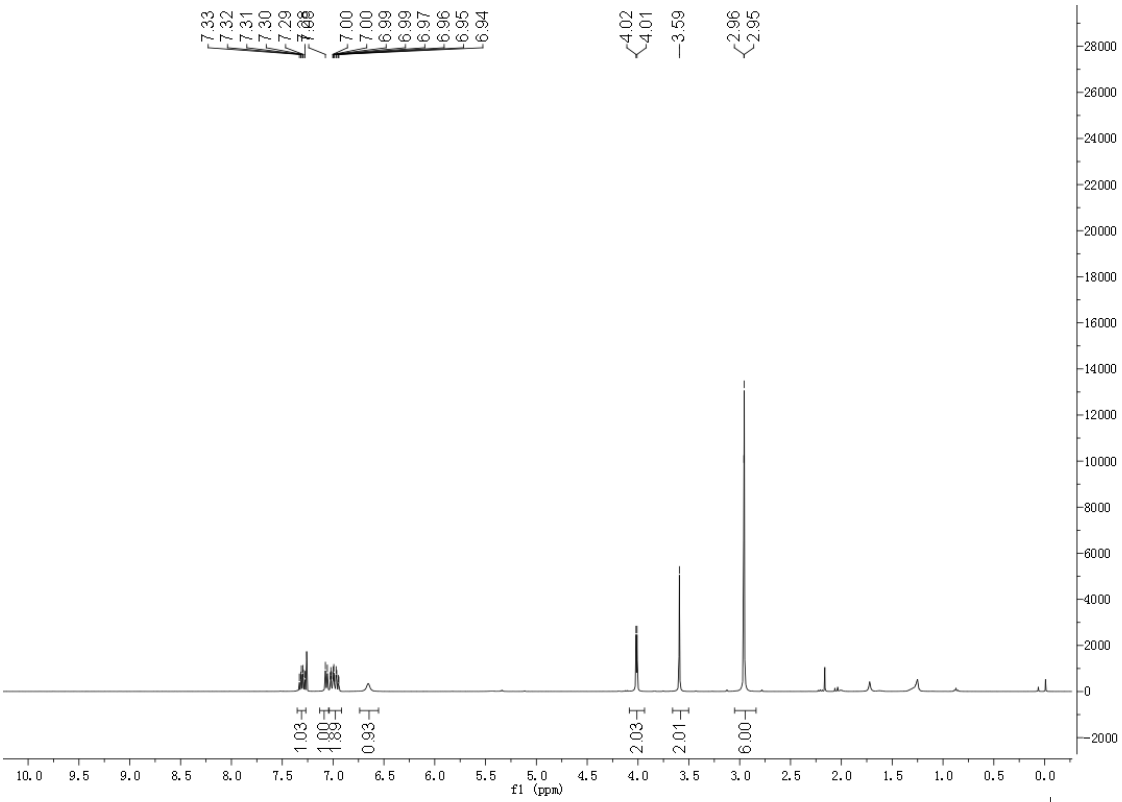
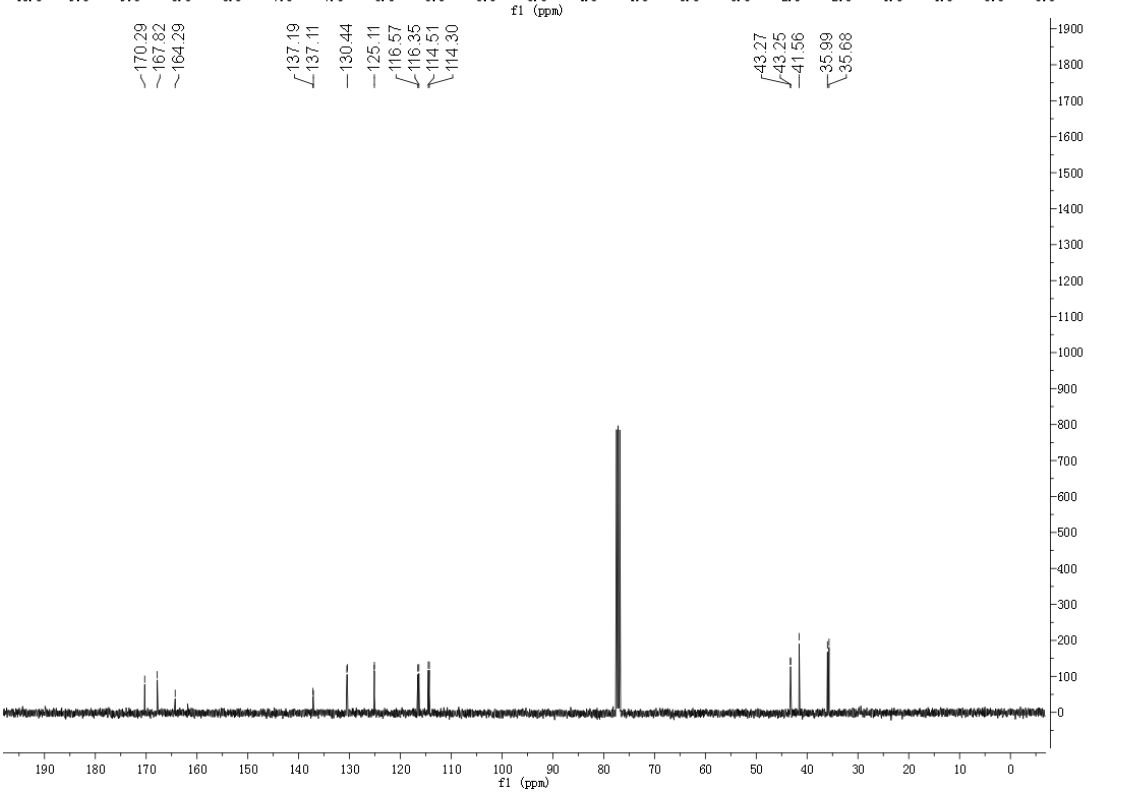
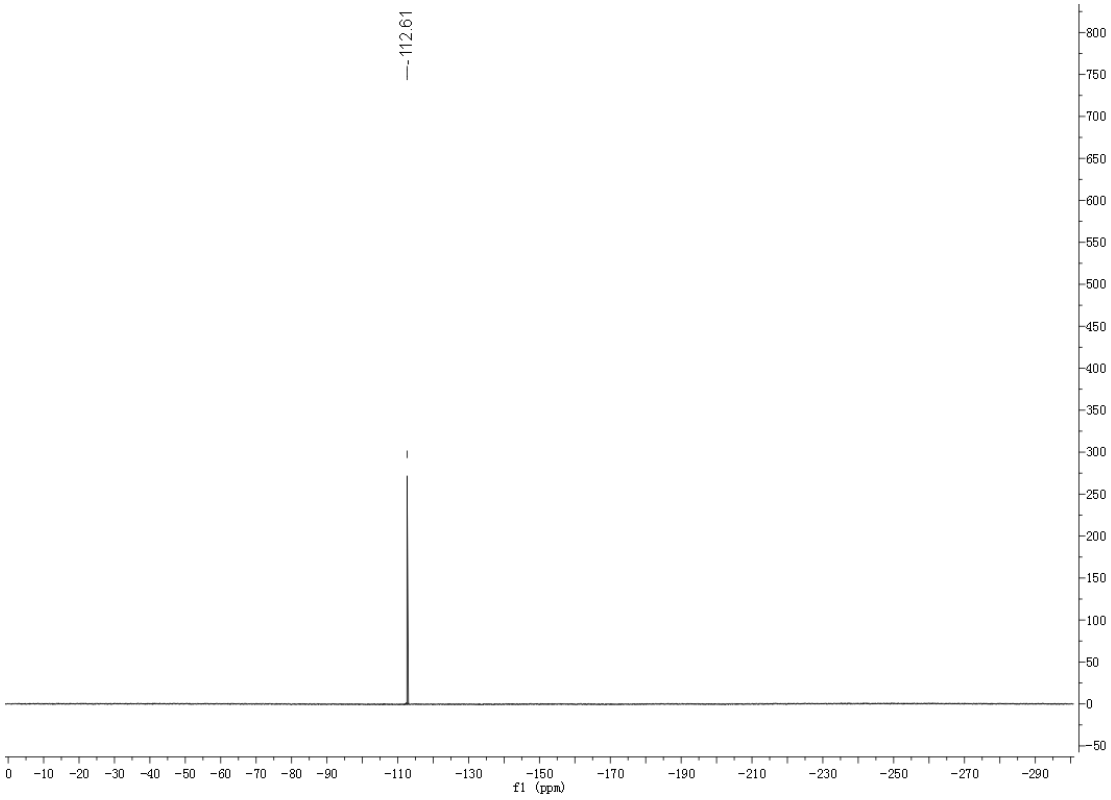
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
f1 (ppm)

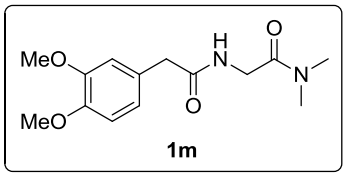

F
H
N
O
O
N

1l

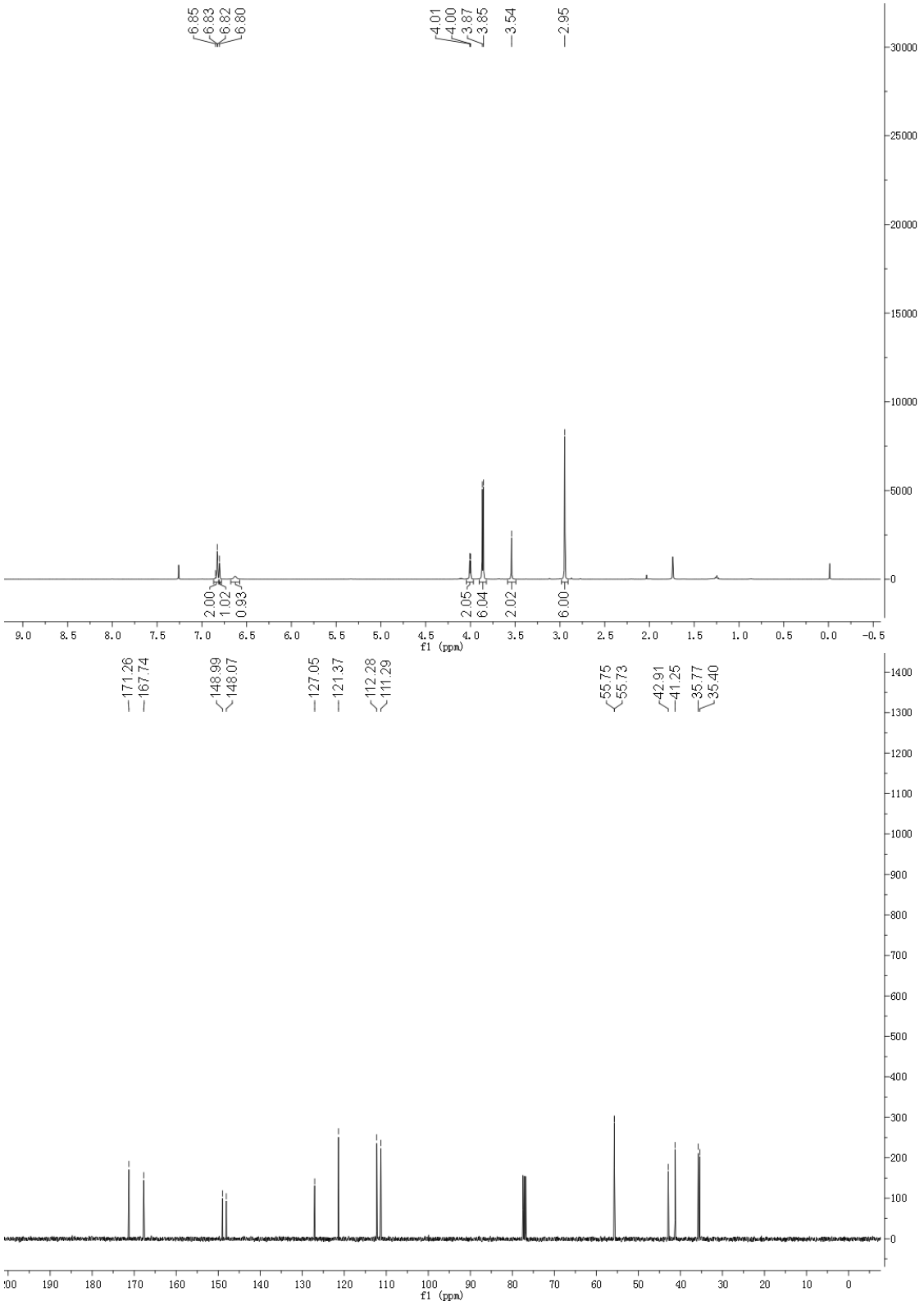

7.33
7.32
7.31
7.30
7.29
7.00
7.00
6.99
6.99
6.97
6.96
6.95
6.94
4.02
4.01
3.59
2.96
2.95
1.03
1.00
1.89
0.93
2.03
2.01
6.00
f1 (ppm)

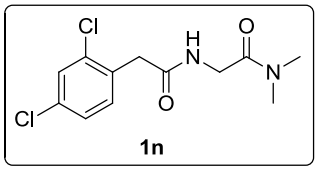

170.29
167.82
164.29
137.19
137.11
130.44
125.11
116.57
116.35
114.51
114.30
43.27
43.25
41.56
35.99
35.68
f1 (ppm)


-112.61
800
750
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

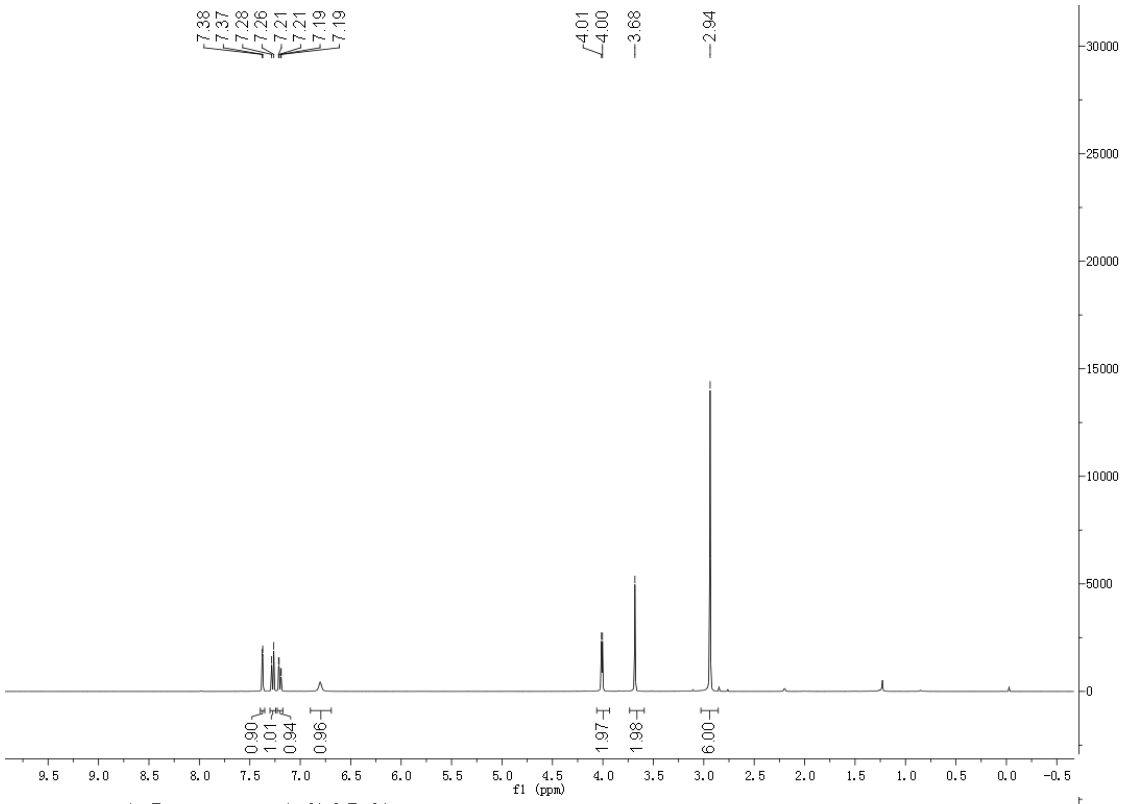

MeO
MeO
H
N
O
O
N

1m


6.85
6.83
6.82
6.80
4.01
4.00
3.87
3.85
3.54
2.95
2.00
1.02
0.93
2.05
6.04
2.02
6.00
f1 (ppm)
171.26
167.74
148.99
148.07
127.05
121.37
112.28
111.29
55.75
55.73
42.91
41.25
35.77
35.40
f1 (ppm)


Cl
H
N
O
O
N
Cl

1n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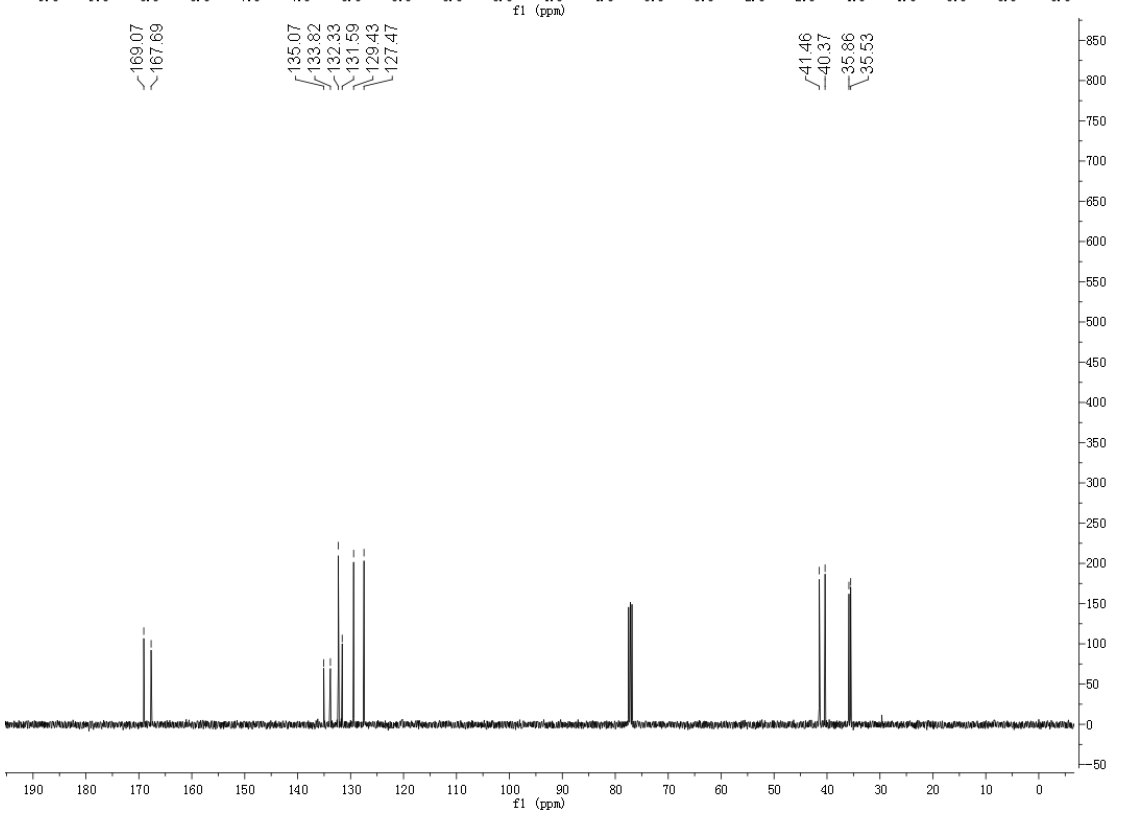
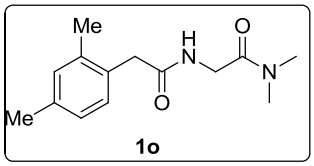
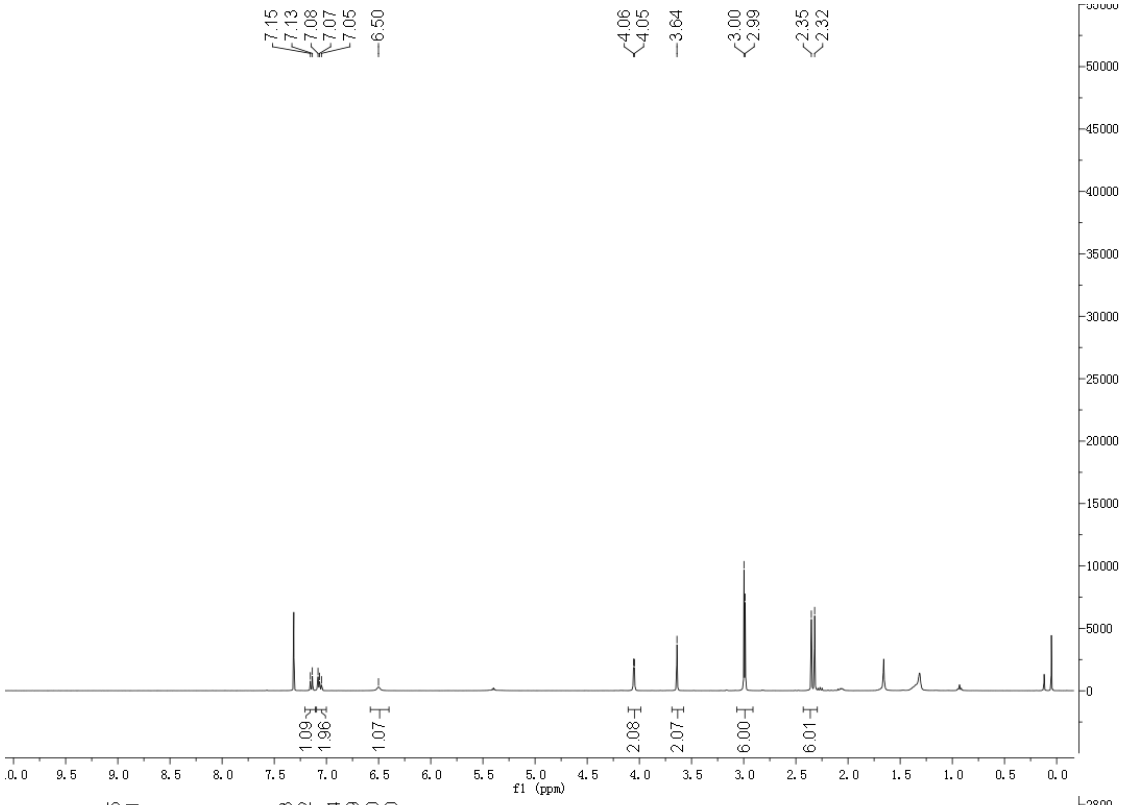
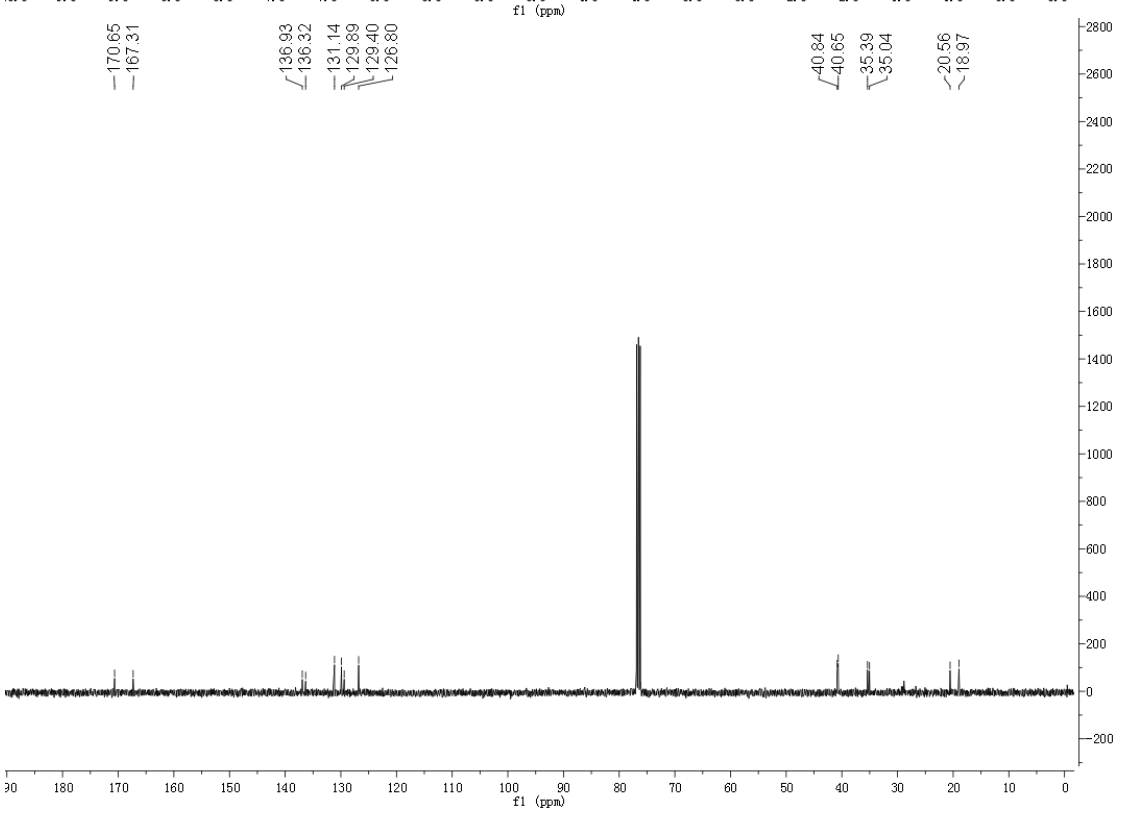
7.37
7.28
7.26
7.21
7.21
7.19
7.19
4.01
4.00
3.68
2.94
0.90
1.01
0.94
0.96
1.97
1.98
6.00
f1 (ppm)

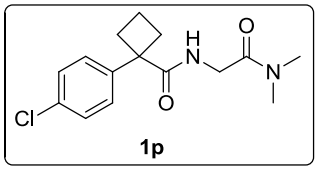

169.07
167.69
135.07
133.82
132.33
131.59
129.43
127.47
41.46
40.37
35.86
35.53
f1 (ppm)


Me
H
N
O
N
O
Me

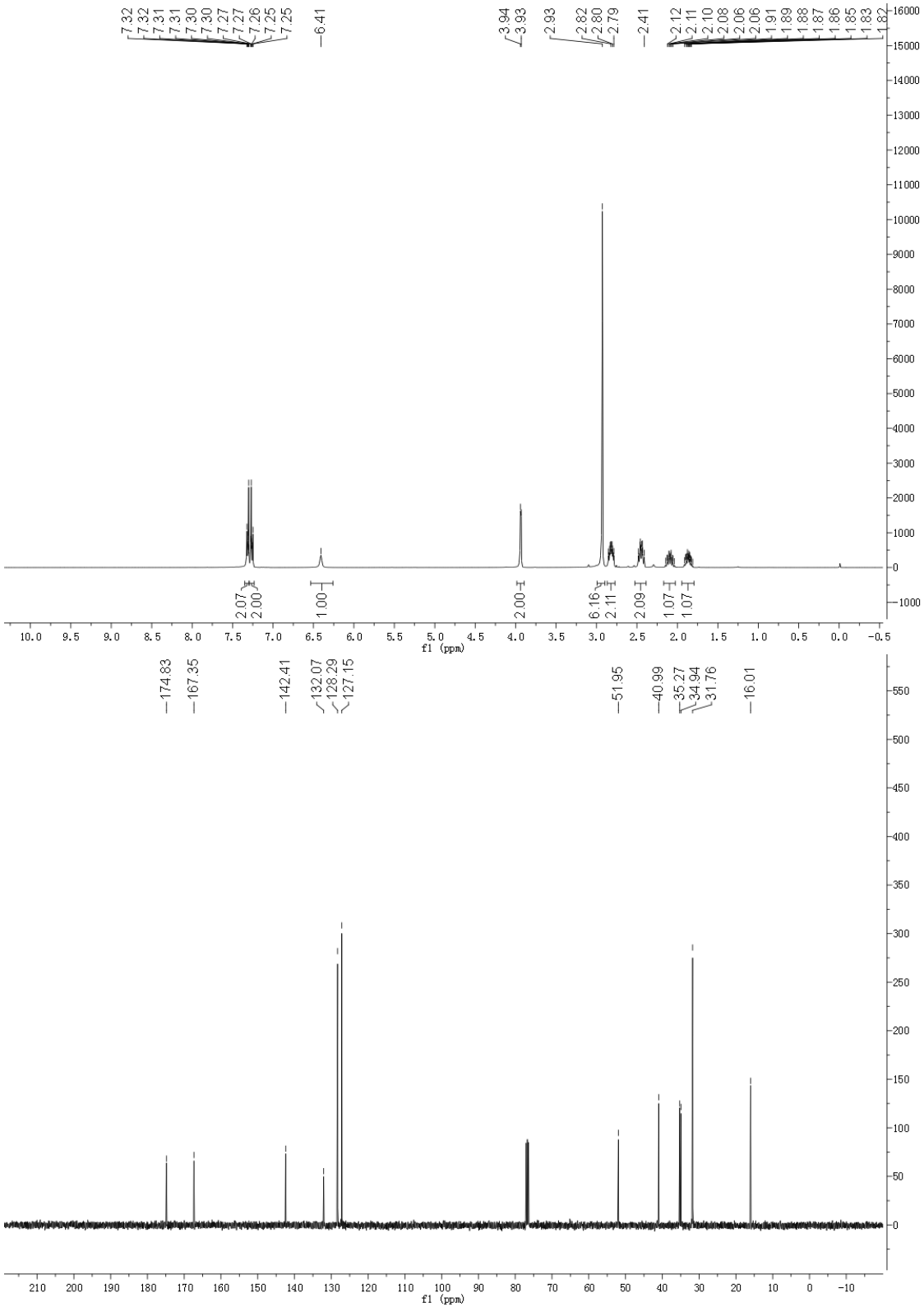
1o


7.15
7.13
7.08
7.07
7.05
6.50
4.06
4.05
3.64
3.00
2.99
2.35
2.32
1.09
1.96
1.07
2.08
2.07
6.00
6.01
f1 (ppm)


170.65
167.31
136.93
136.32
131.14
129.89
129.40
126.80
40.84
40.65
35.39
35.04
20.56
18.97
f1 (ppm)


H
N
O
O
N
Cl

1p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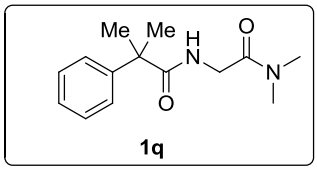
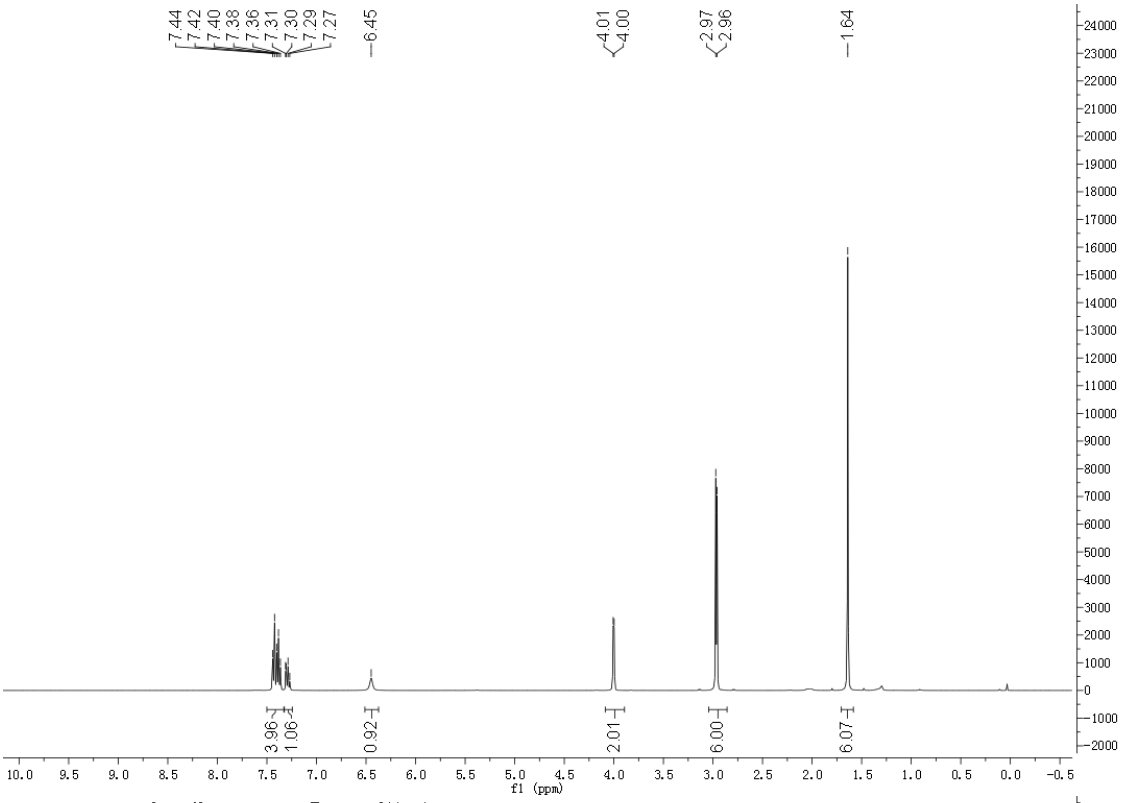
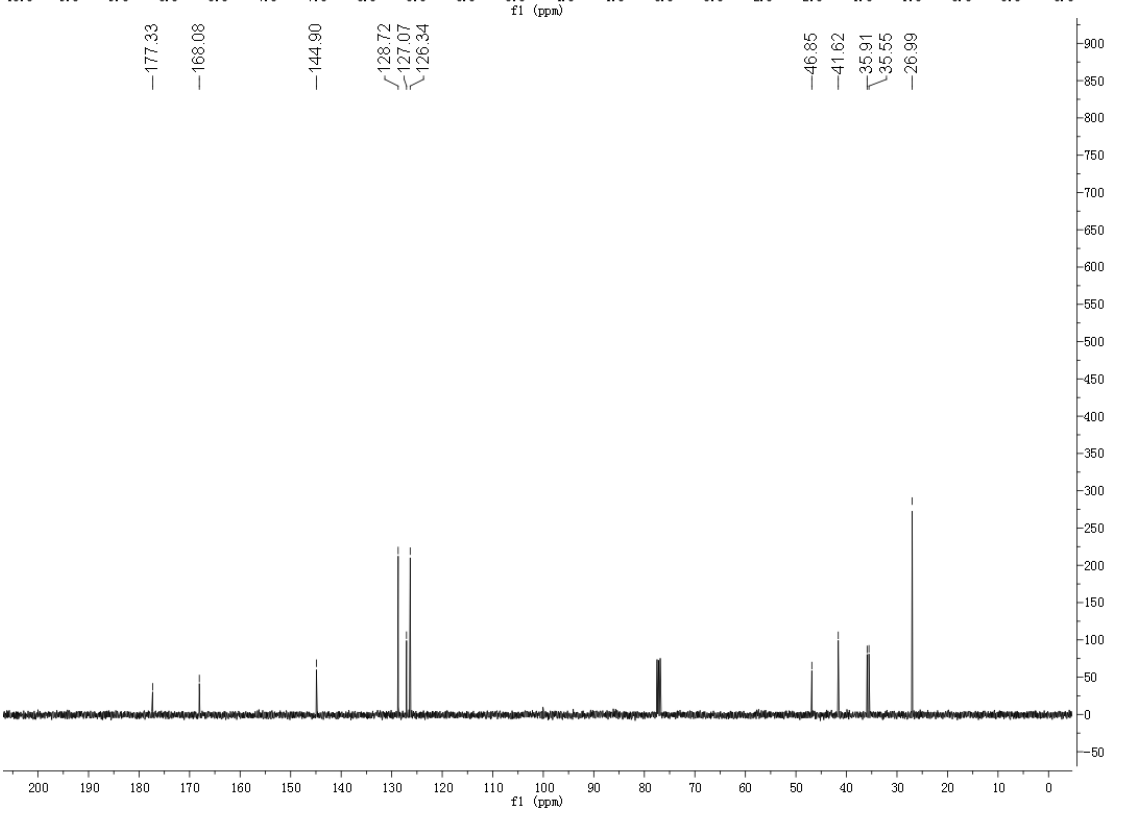
7.32
7.31
7.31
7.30
7.30
7.27
7.27
7.26
7.25
7.25
6.41
3.94
3.93
2.93
2.82
2.80
2.79
2.41
2.12
2.11
2.10
2.08
2.06
2.06
1.91
1.89
1.88
1.87
1.86
1.85
1.83
1.82
2.07
2.00
1.00
2.00
6.16
2.11
2.09
1.07
1.07
f1 (ppm)
174.83
167.35
142.41
132.07
128.29
127.15
51.95
40.99
35.27
34.94
31.76
16.01
f1 (ppm)


Me
Me
H
N
O
O
N

1q


7.44
7.42
7.40
7.38
7.36
7.31
7.30
7.29
7.27
6.45
4.01
4.00
2.97
2.96
1.64
3.96
1.06
0.92
2.01
6.00
6.07
f1 (ppm)


177.33
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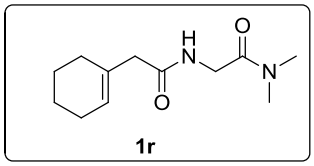
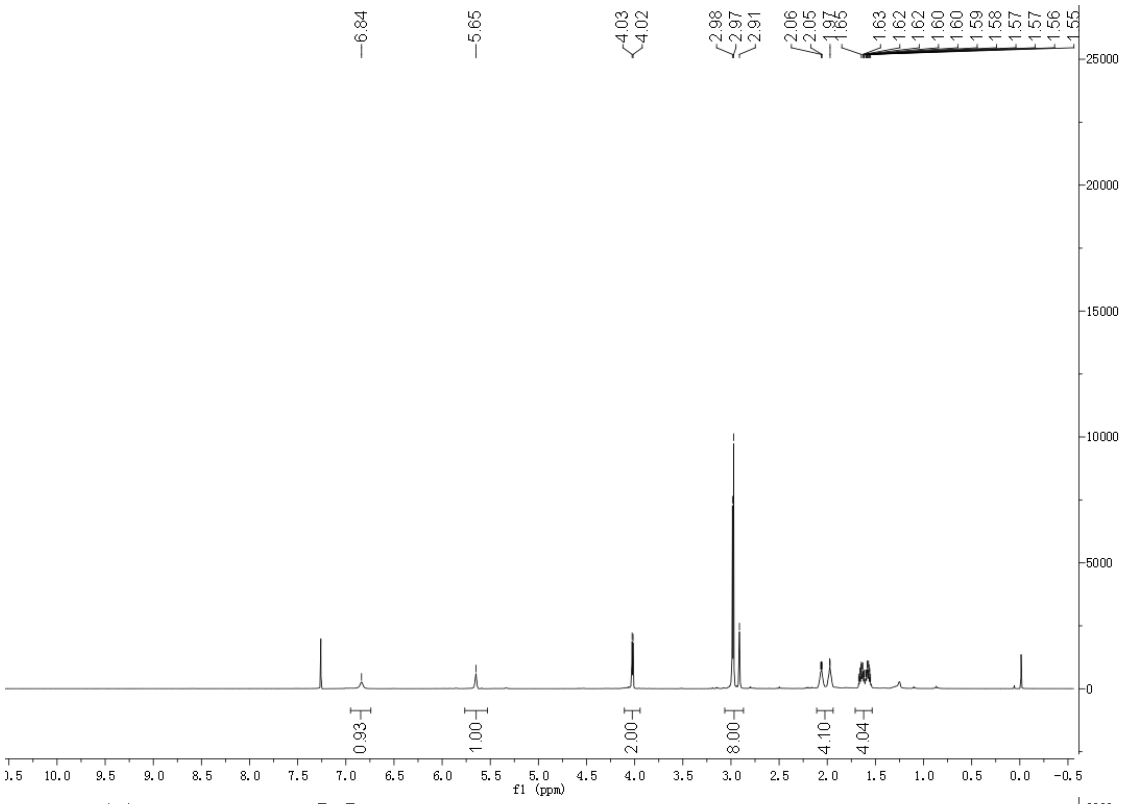
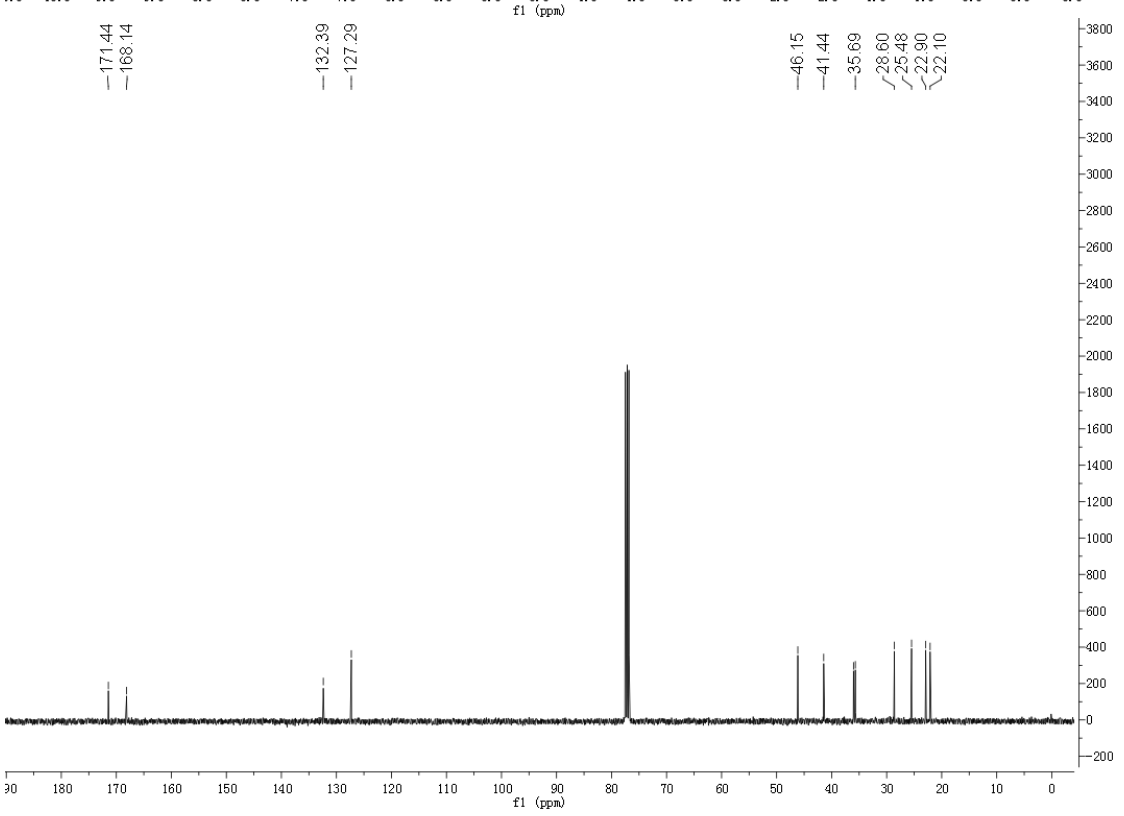
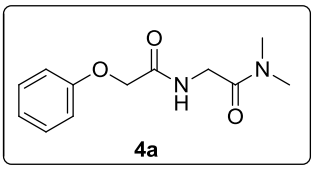
144.90
128.72
127.07
126.34
46.85
41.62
35.91
35.55
26.99
f1 (ppm)

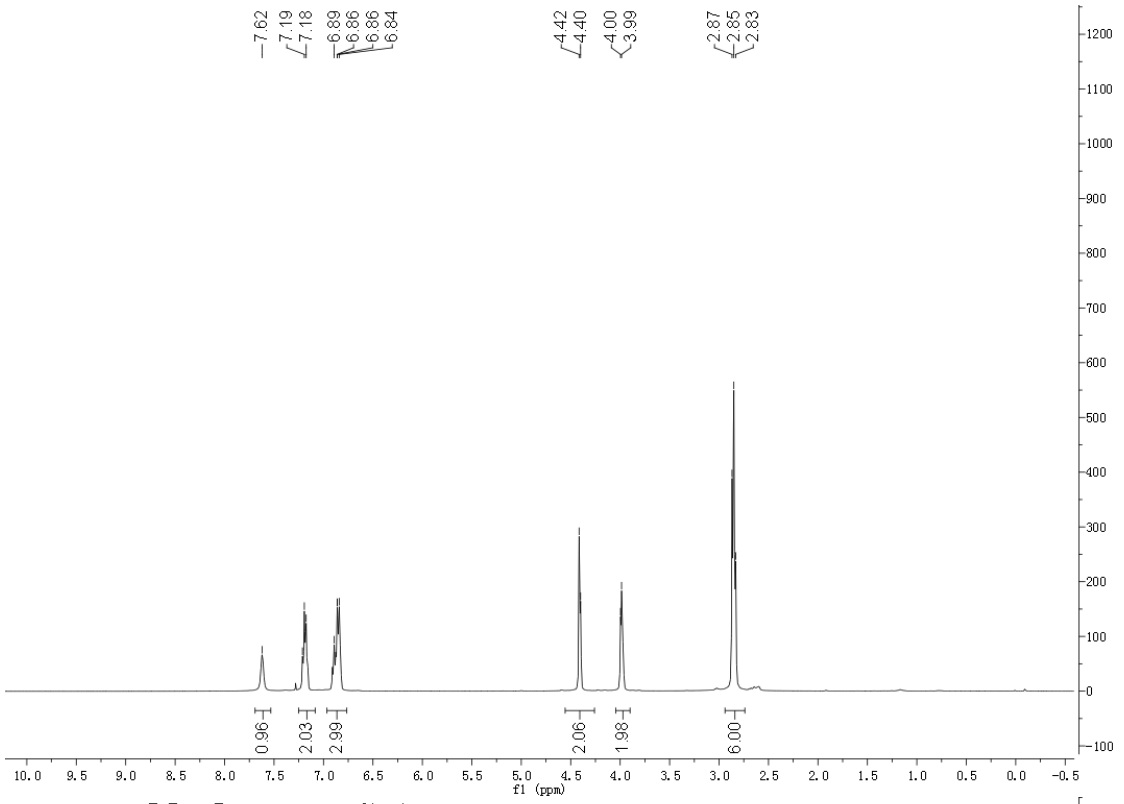

H
N
O
O
N

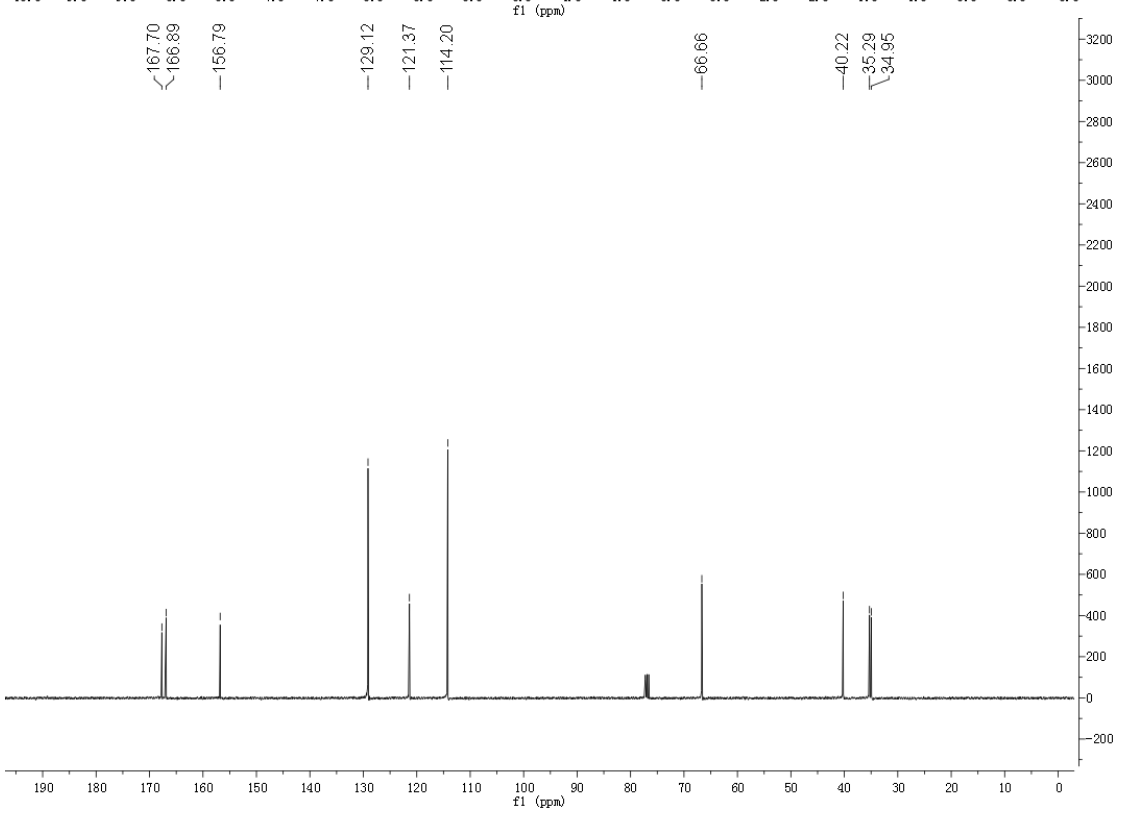
1r

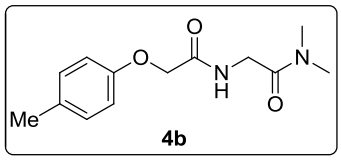

6.84
5.65
4.03
4.02
2.98
2.97
2.91
2.06
2.05
1.97
1.65
1.63
1.62
1.62
1.60
1.60
1.59
1.58
1.57
1.57
1.56
1.55
0.93
1.00
2.00
8.00
4.10
4.04
f1 (ppm)


171.44
168.14
132.39
127.29
46.15
41.44
35.69
28.60
25.48
22.90
22.10
f1 (ppm)

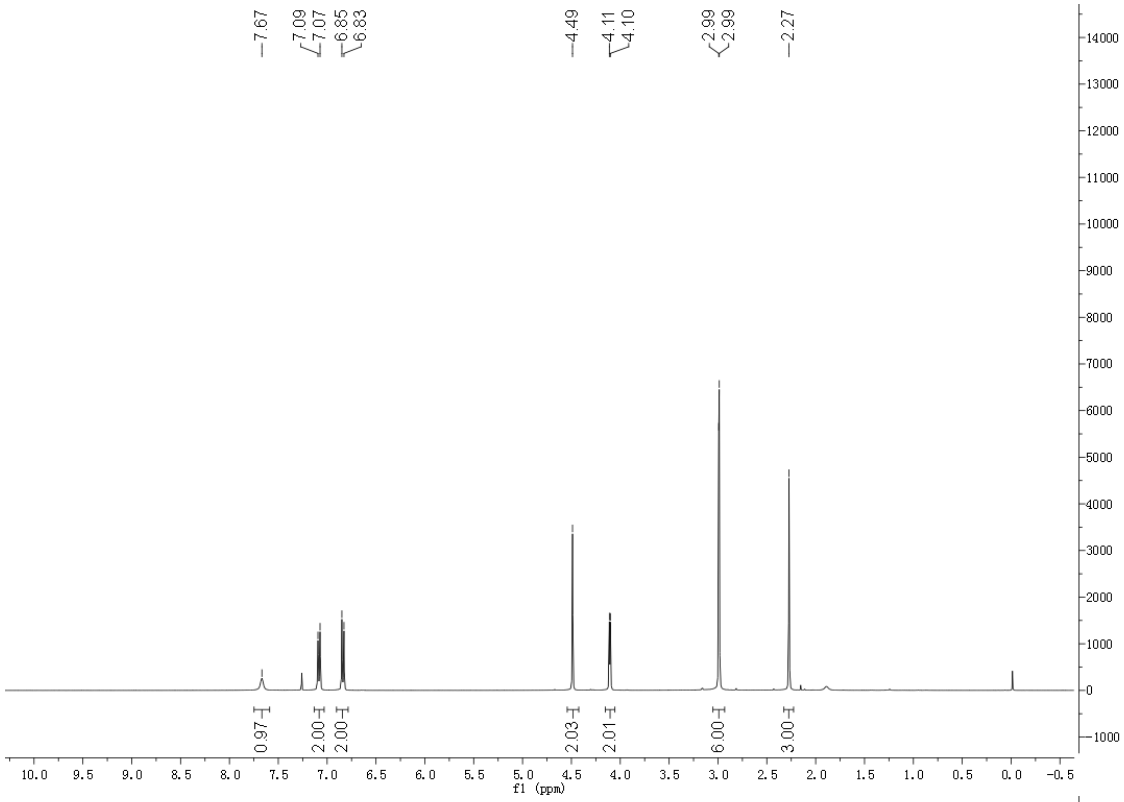

O
O
N
H
O
N
4a

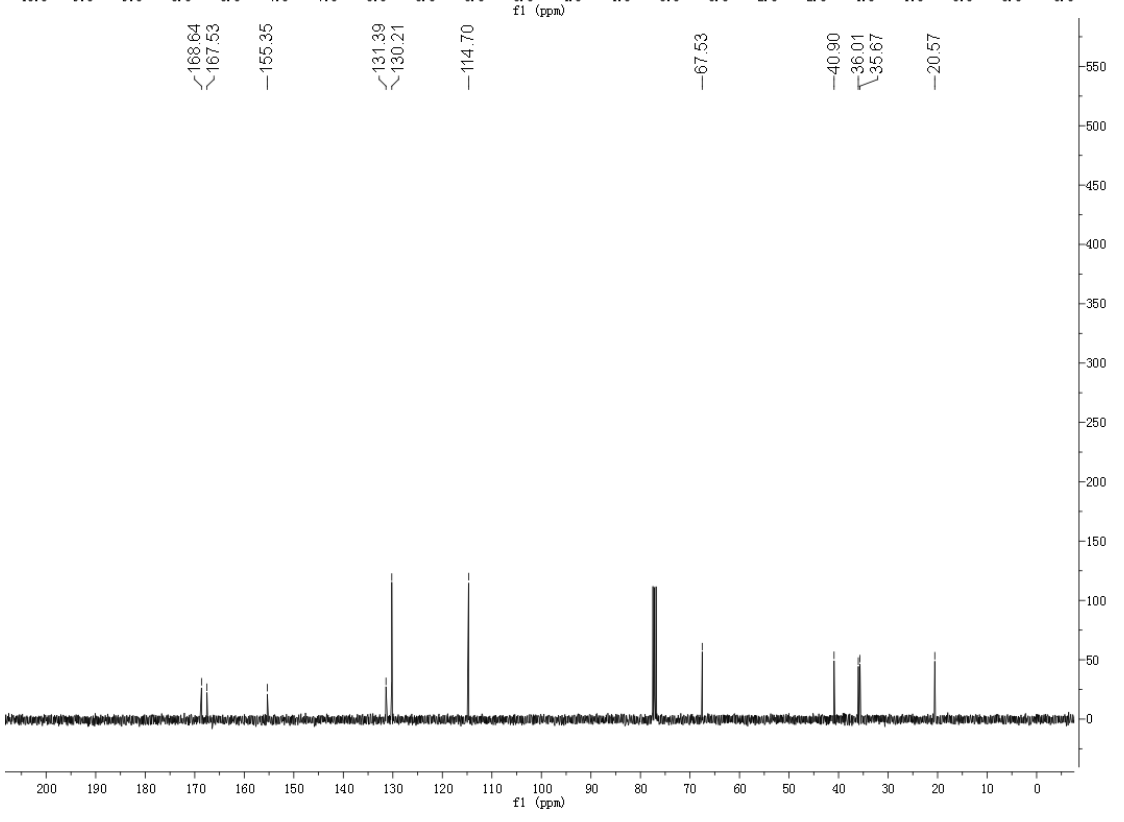

7.62
7.19
7.18
6.89
6.86
6.86
6.84
4.42
4.40
4.00
3.99
2.87
2.85
2.83
0.96
2.03
2.99
2.06
1.98
6.00
f1 (ppm)

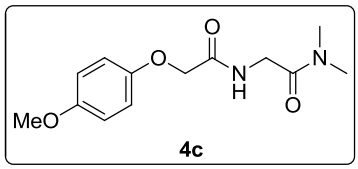

167.70
166.89
156.79
129.12
121.37
114.20
66.66
40.22
35.29
34.95
f1 (ppm)


O
O
N
H
N
O
Me

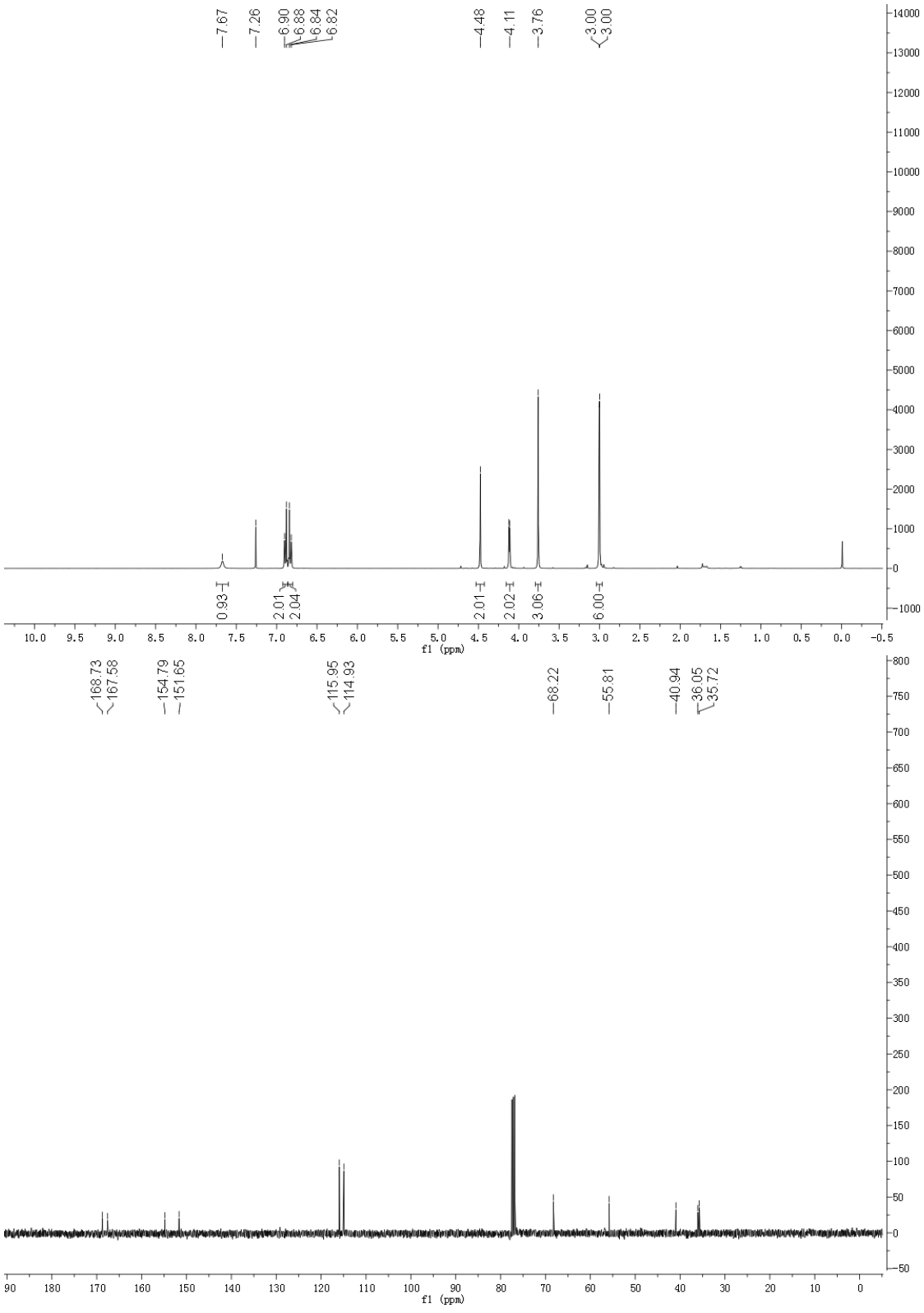
4b

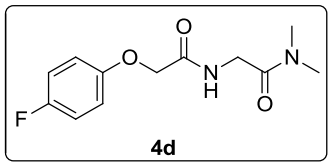

7.67
7.09
7.07
6.85
6.83
4.49
4.11
4.10
2.99
2.99
2.27
0.97
2.00
2.00
2.03
2.01
6.00
3.00
f1 (ppm)


168.64
167.53
155.35
131.39
130.21
114.70
67.53
40.90
36.01
35.67
20.57
f1 (ppm)

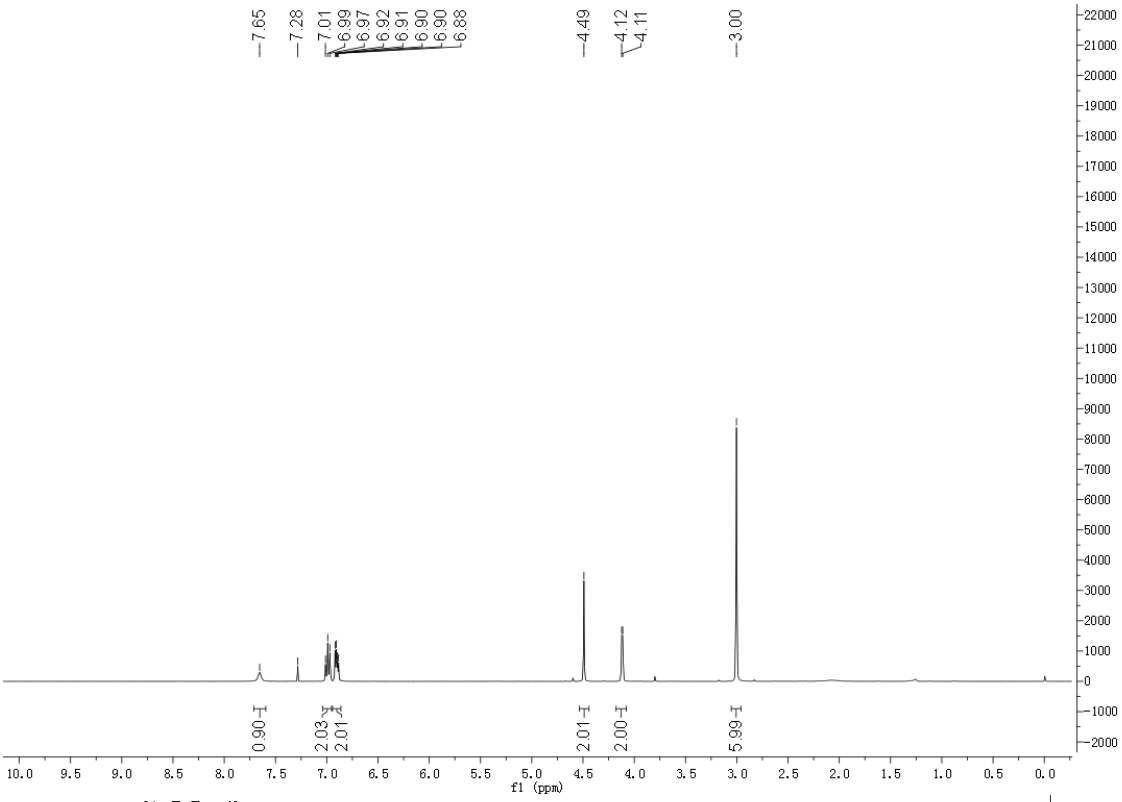

O
O
N
H
N
O
MeO

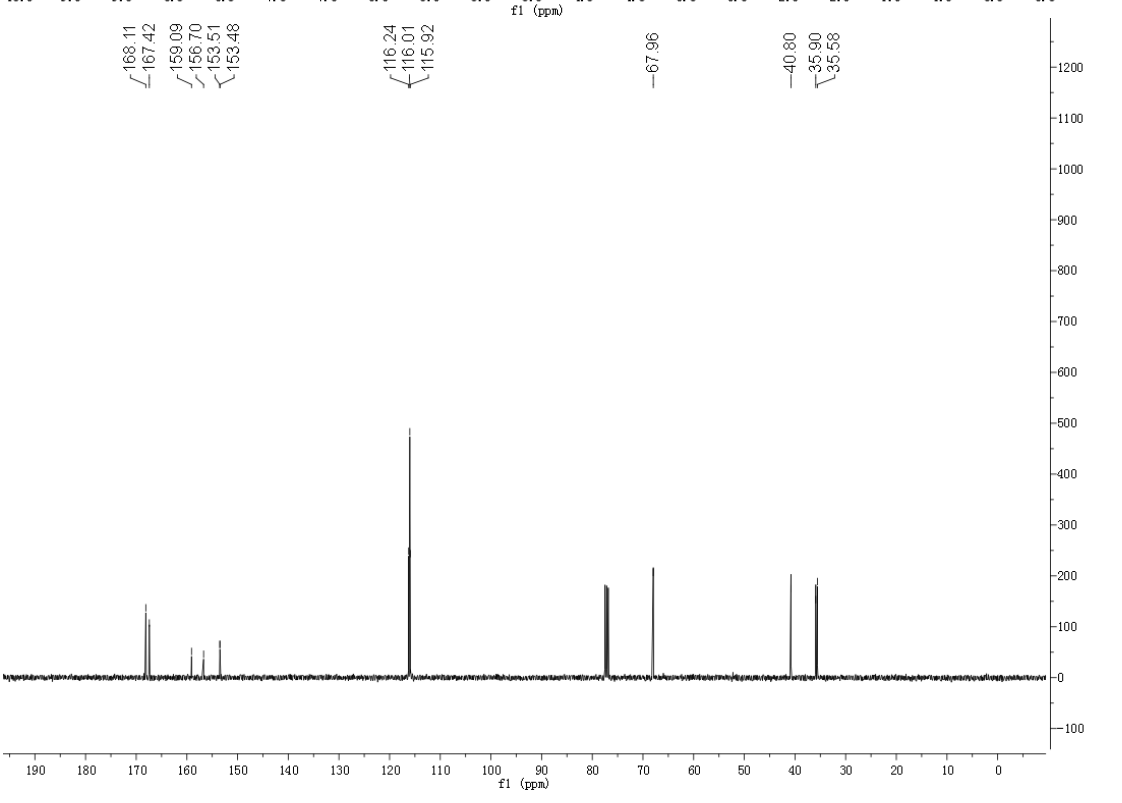
4c

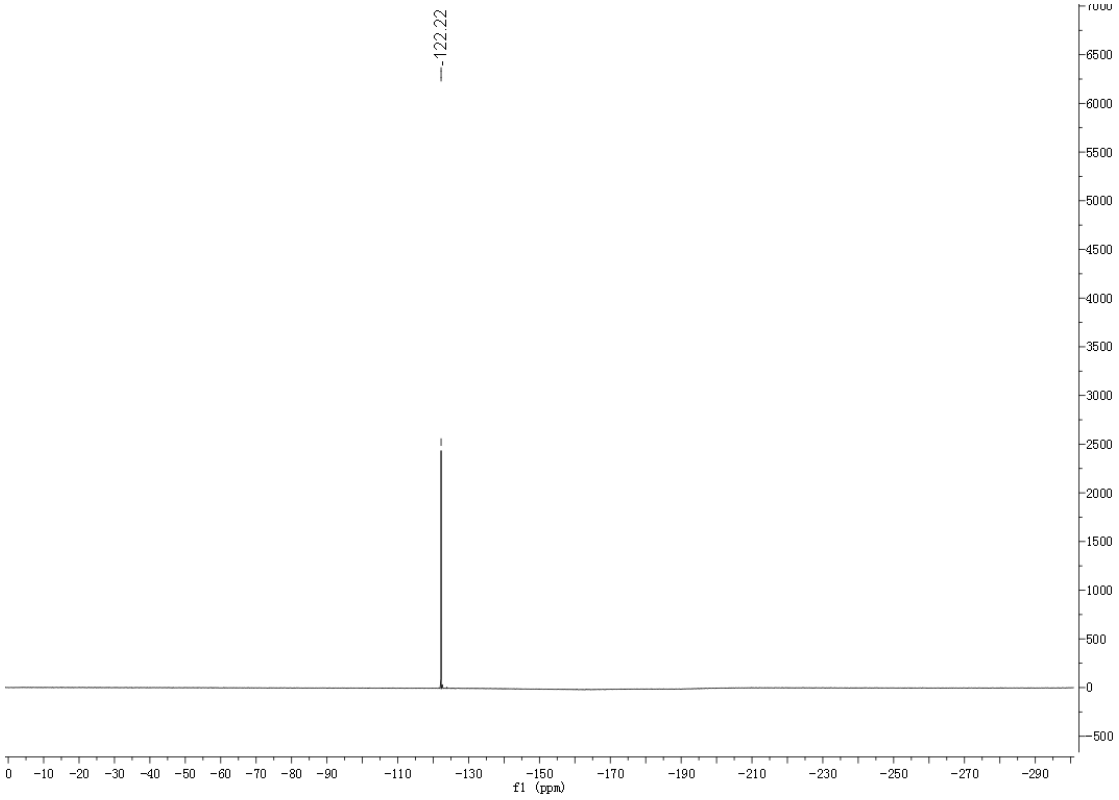

7.67
7.26
6.90
6.88
6.84
6.82
4.48
4.11
3.76
3.00
3.00
0.93
2.01
2.04
2.01
2.02
3.06
6.00
f1 (ppm)
168.73
167.58
154.79
151.65
115.95
114.93
68.22
55.81
40.94
36.05
35.72
f1 (ppm)

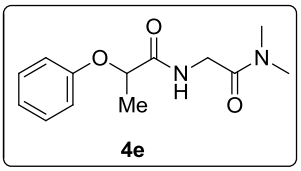

O
O
N
H
N
O
F

4d

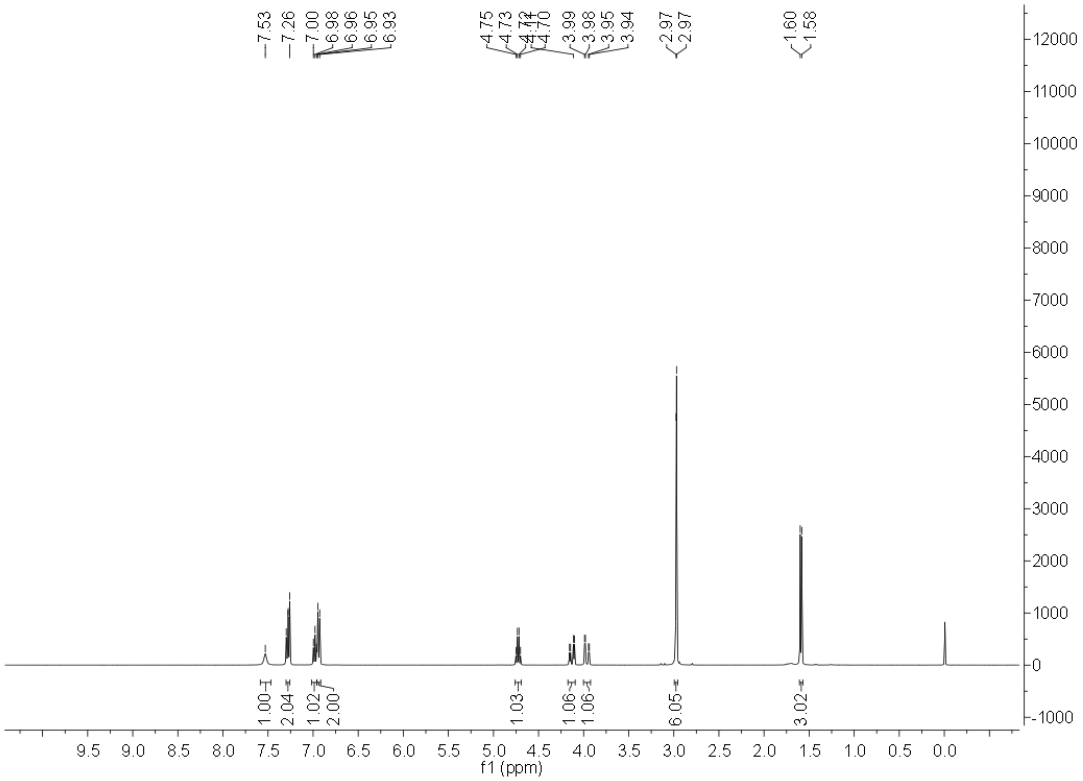

7.65
7.28
7.01
6.99
6.97
6.92
6.91
6.90
6.90
6.88
4.49
4.12
4.11
3.00
0.90
2.03
2.01
2.01
2.00
5.99
f1 (ppm)


168.11
167.42
159.09
156.70
153.51
153.48
116.24
116.01
115.92
67.96
40.80
35.90
35.58
f1 (ppm)


-122.22
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)


O
O
N
H
Me
O
N

4e


7.53
7.26
7.00
6.98
6.96
6.95
6.93
4.75
4.73
4.70
3.99
3.98
3.95
3.94
2.97
2.97
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1.02
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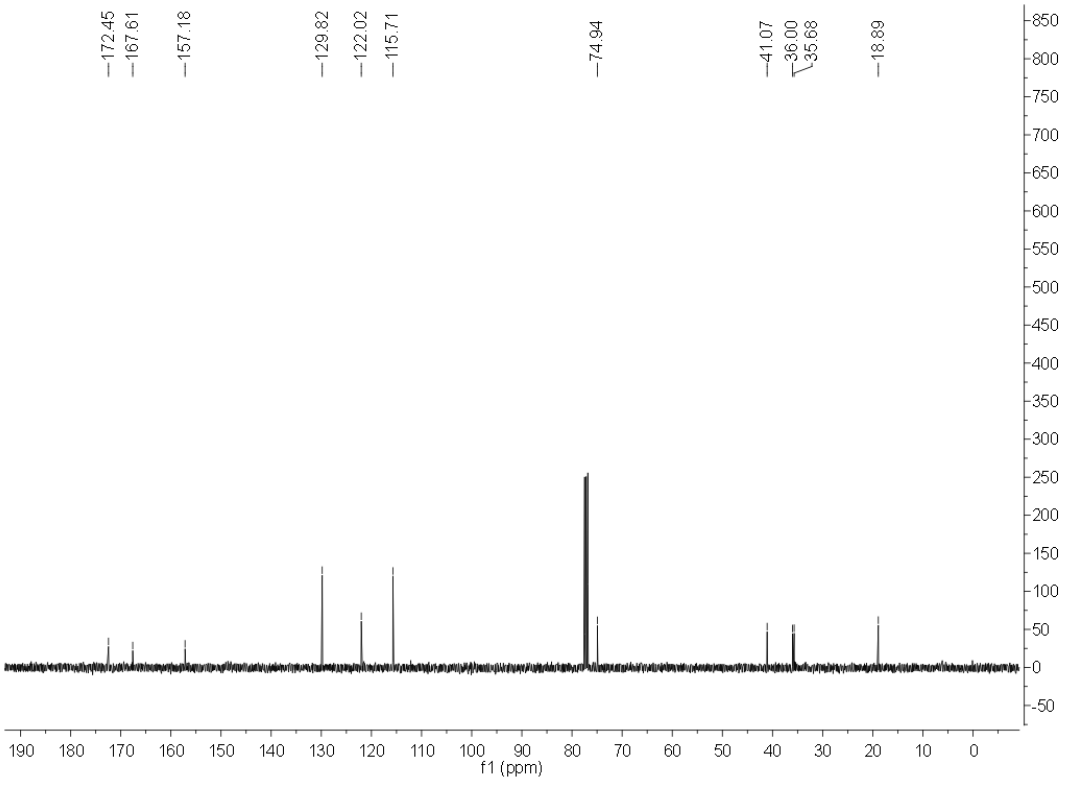
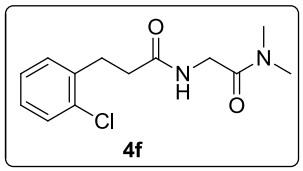
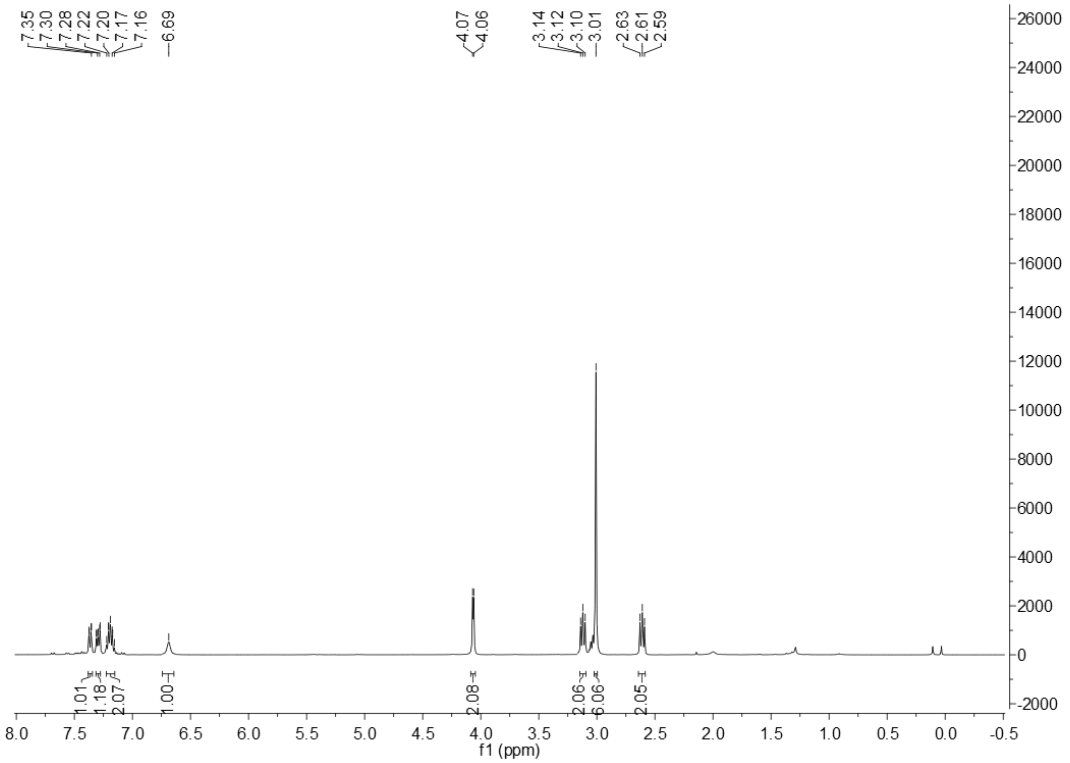
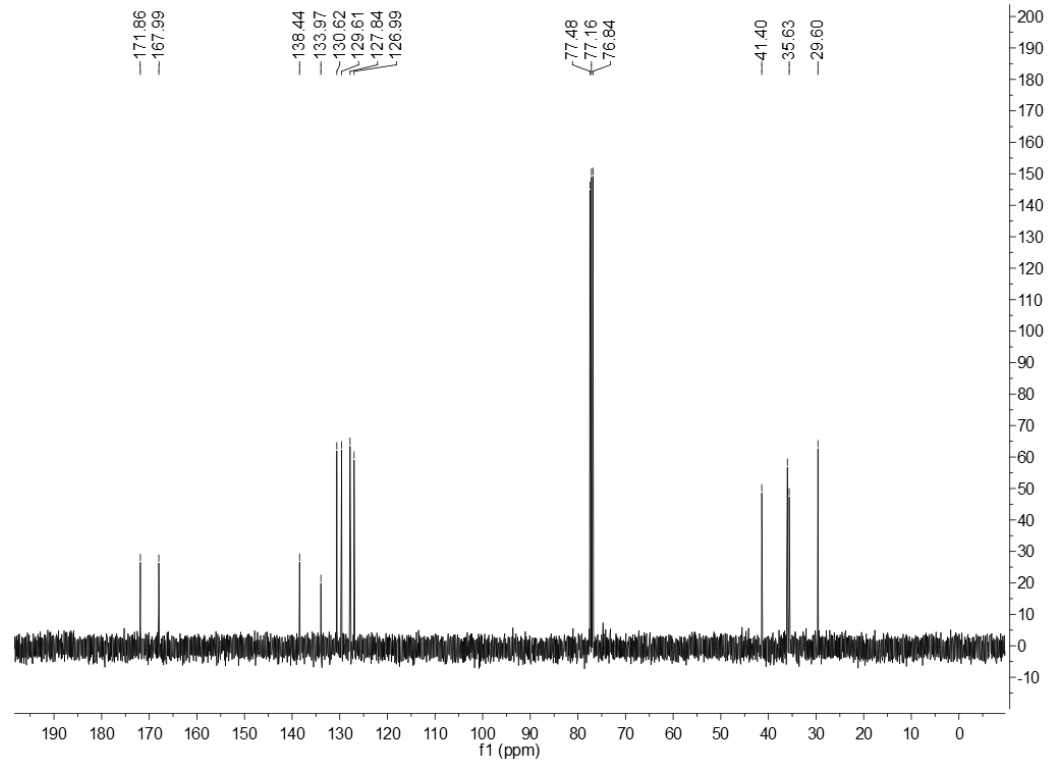
1.03
1.06
1.06
6.05
3.02
f1 (ppm)

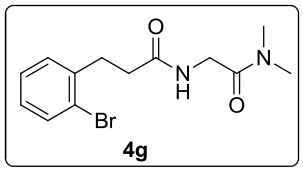

172.45
167.61
157.18
129.82
122.02
115.71
74.94
41.07
36.00
35.68
18.89
f1 (ppm)


O
N
H
O
N
Cl

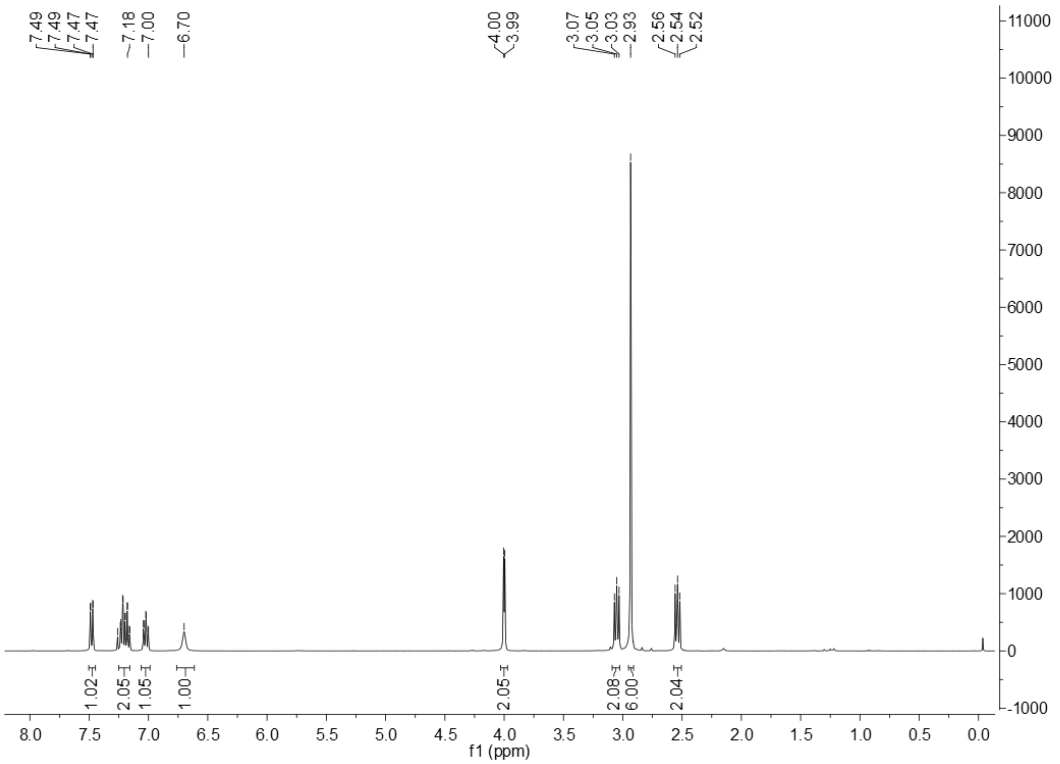
4f


7.35
7.30
7.28
7.22
7.20
7.17
7.16
6.69
4.07
4.06
3.14
3.12
3.10
3.01
2.63
2.61
2.59
1.01
1.18
2.07
1.00
2.08
2.06
6.06
2.05
f1 (ppm)


171.86
167.99
138.44
133.97
130.62
129.61
127.84
126.99
77.48
77.16
76.84
41.40
35.63
29.60
f1 (ppm)


O
N
H
O
N
Br

4g


7.49
7.49
7.47
7.47
7.18
7.00
6.70
4.00
3.99
3.07
3.05
3.03
2.93
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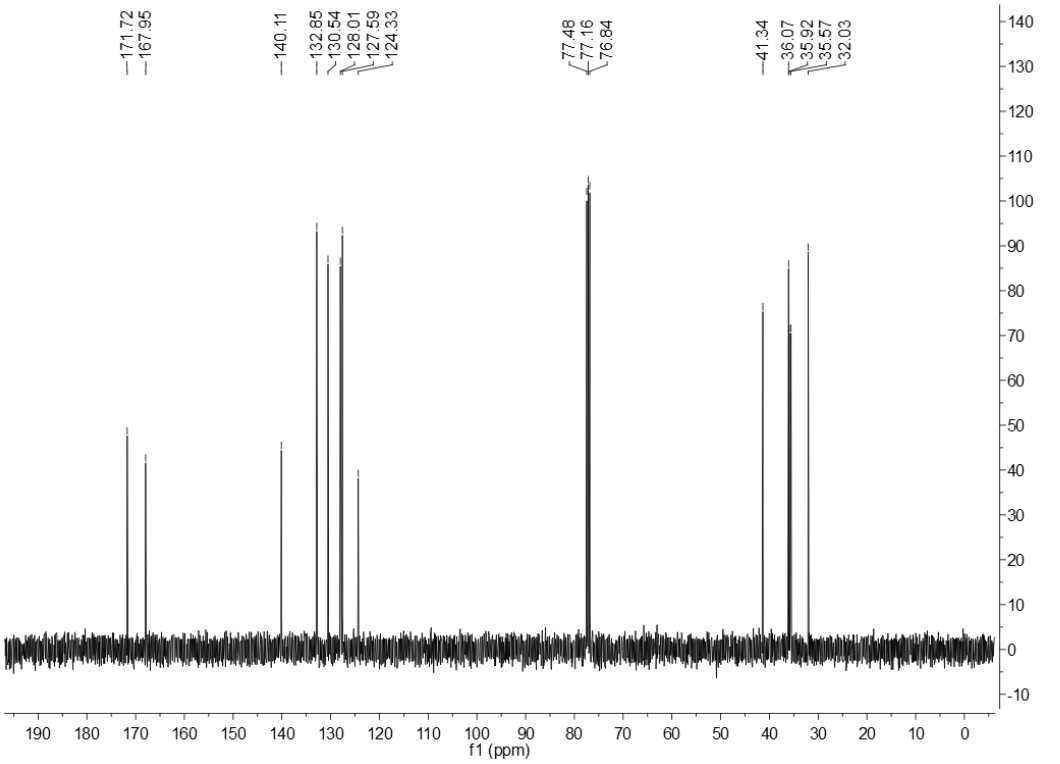
1.02
2.05
1.05
1.00
2.05
2.08
6.00
2.04
f1 (ppm)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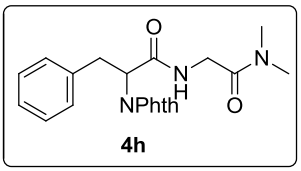
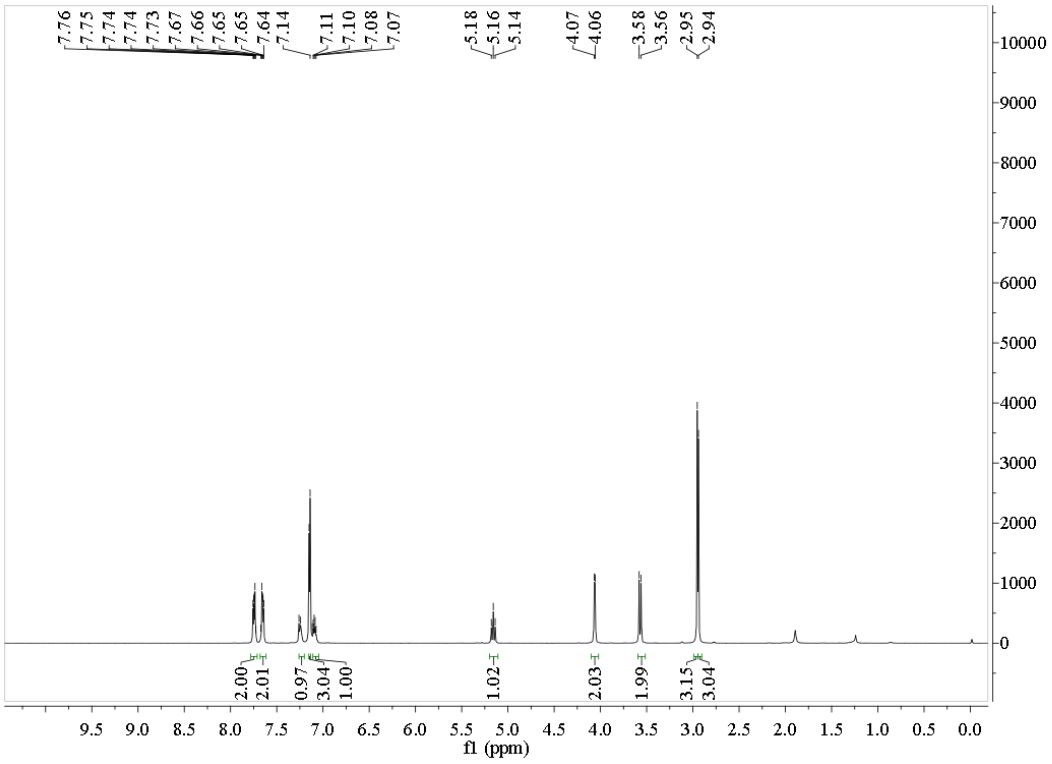
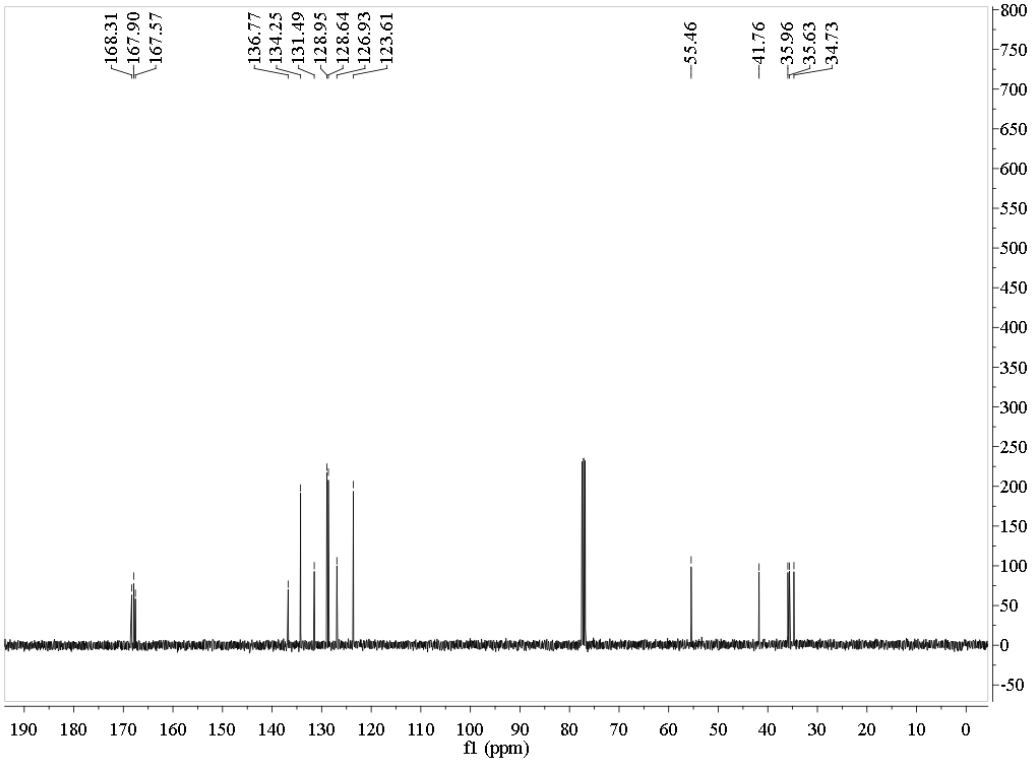
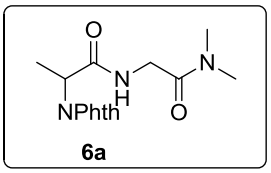
167.95
140.11
132.85
130.54
128.01
127.59
124.33
77.48
77.16
76.84
41.34
36.07
35.92
35.57
32.03
f1 (ppm)


O
N
H
NPhth
O
N

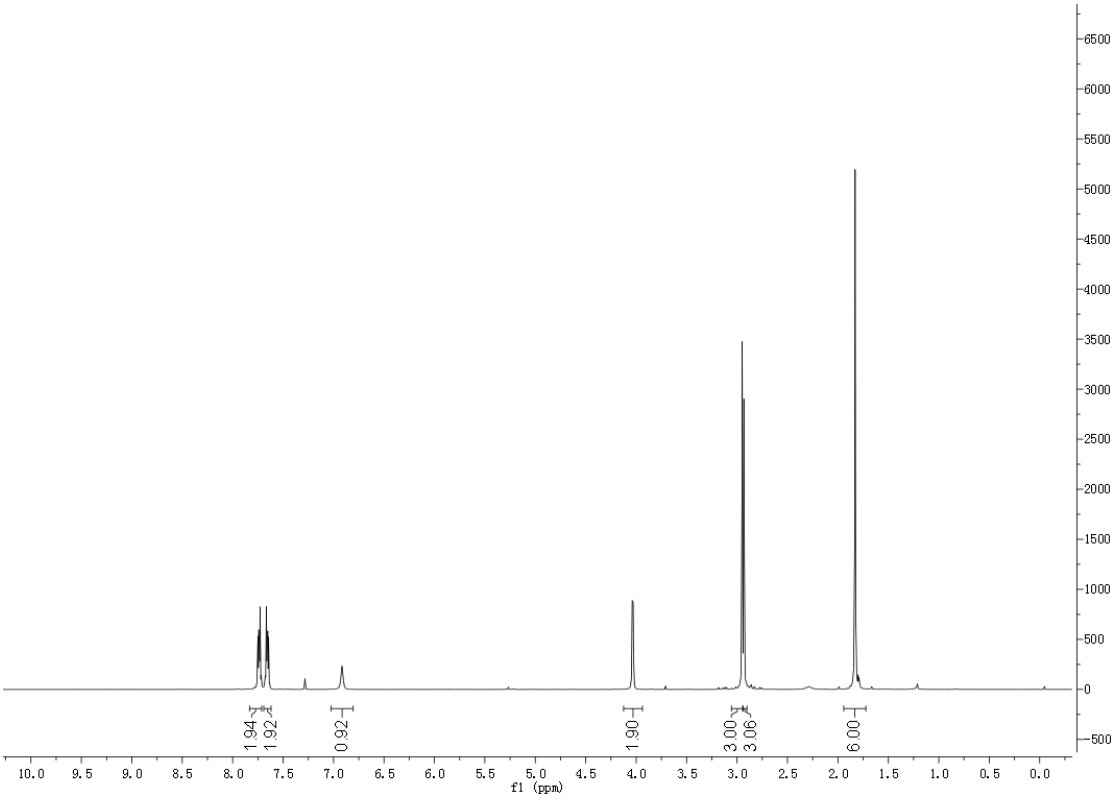
4h

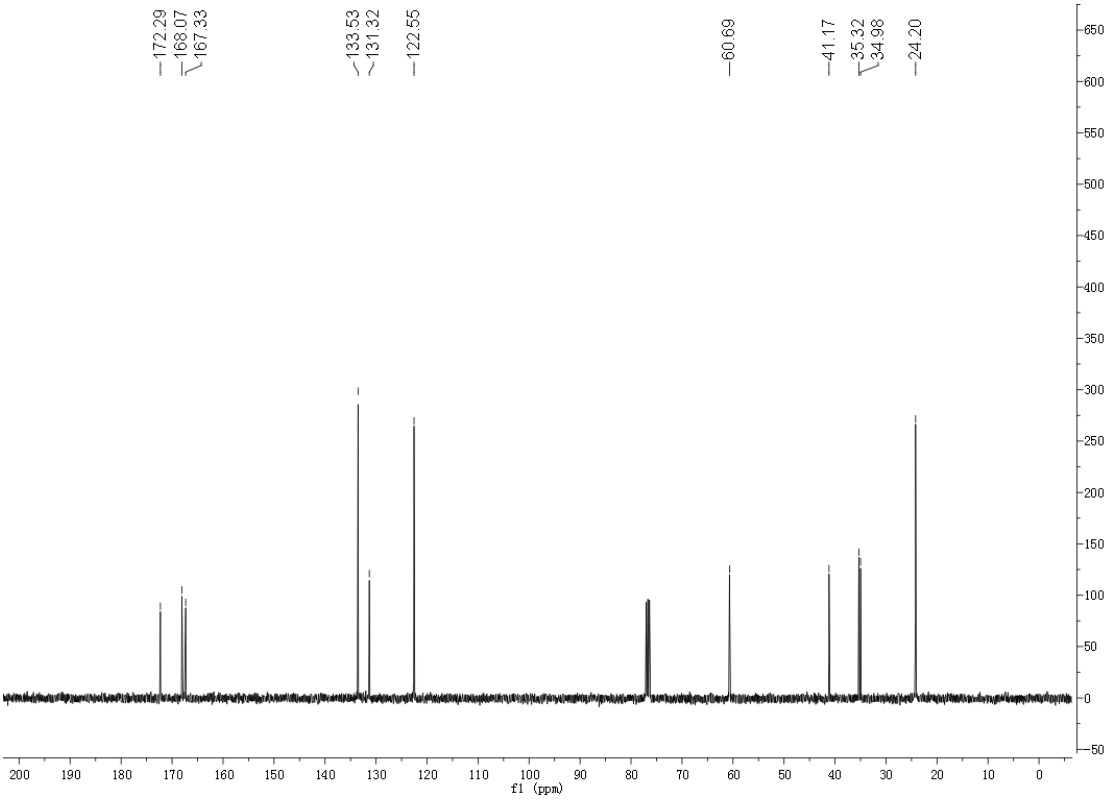

7.76
7.75
7.74
7.74
7.73
7.67
7.66
7.65
7.65
7.64
7.14
7.11
7.10
7.08
7.07
5.18
5.16
5.14
4.07
4.06
3.58
3.56
2.95
2.94
2.00
2.01
0.97
3.04
1.00
1.02
2.03
1.99
3.15
3.04
f1 (ppm)

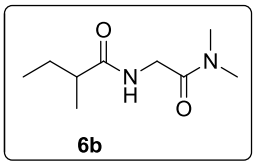

168.31
167.90
167.57
136.77
134.25
131.49
128.95
128.64
126.93
123.61
55.46
41.76
35.96
35.63
34.73
f1 (ppm)

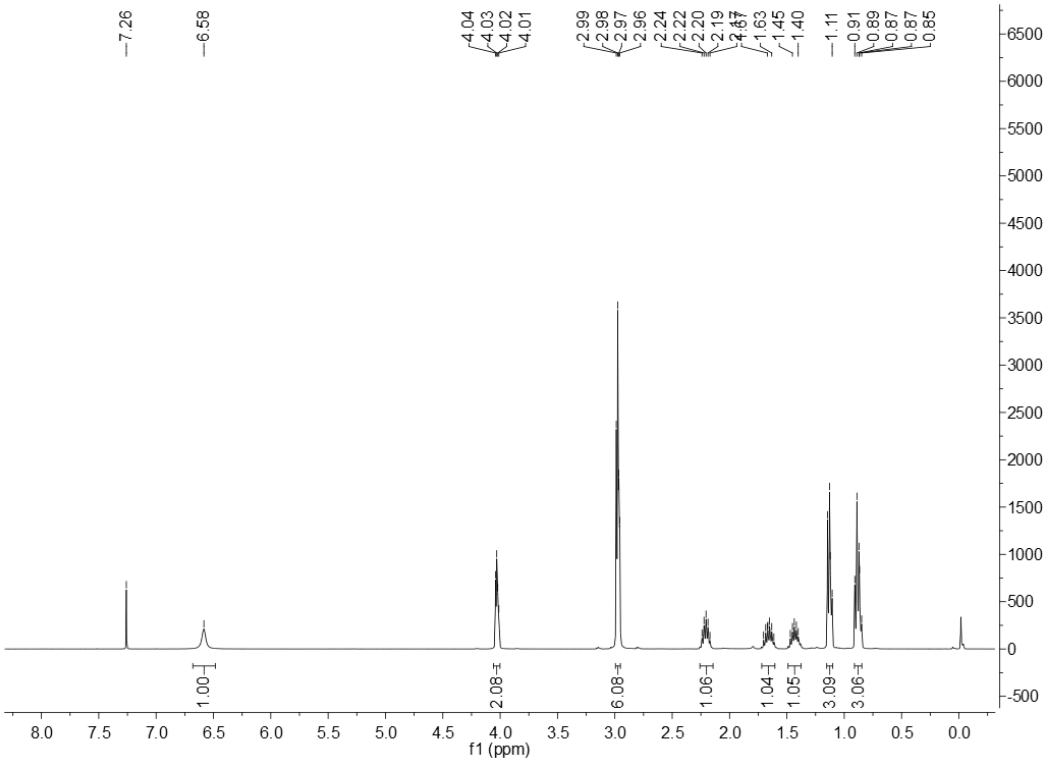

O
N
H
N
O
NPhth

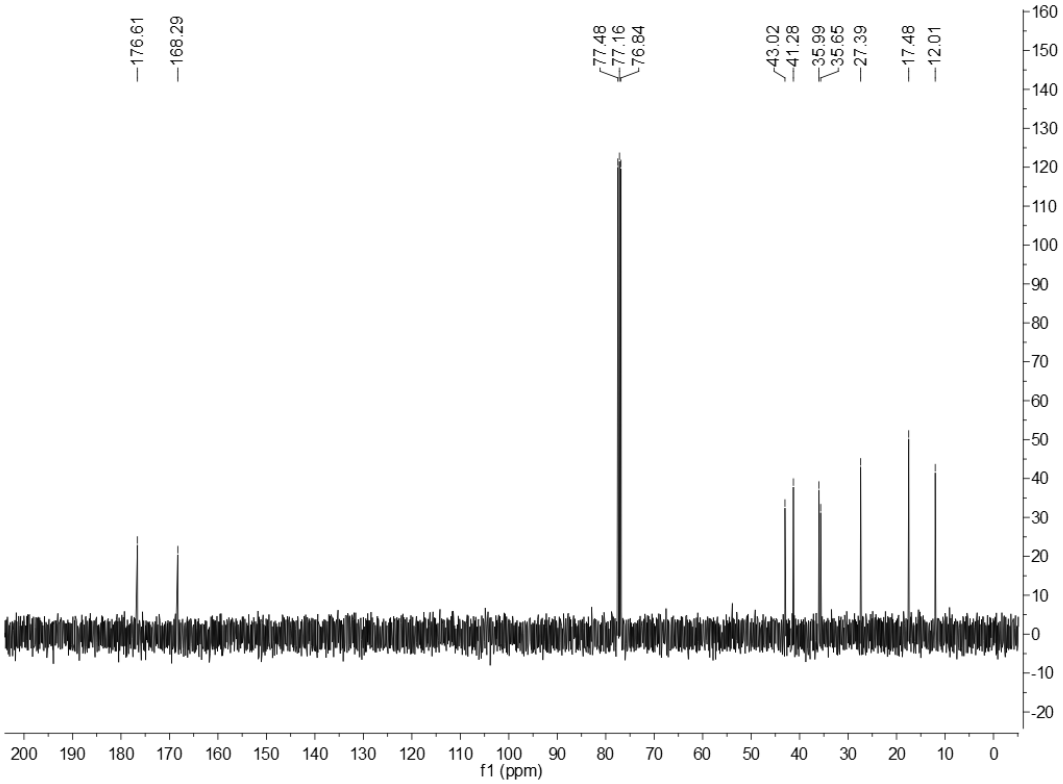
6a

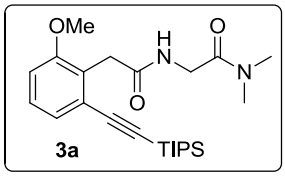

1.94
1.92
0.92
1.90
3.00
3.06
6.00
f1 (ppm)

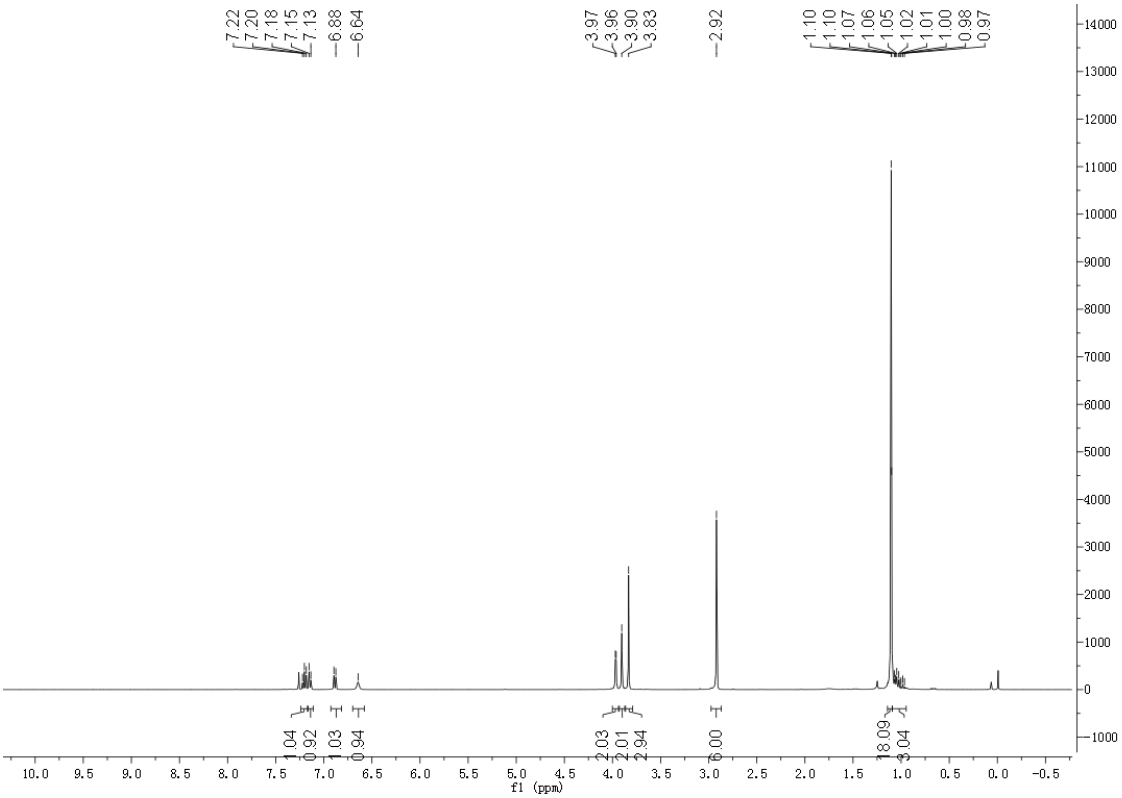

172.29
168.07
167.33
133.53
131.32
122.55
60.69
41.17
35.32
34.98
24.20
f1 (ppm)

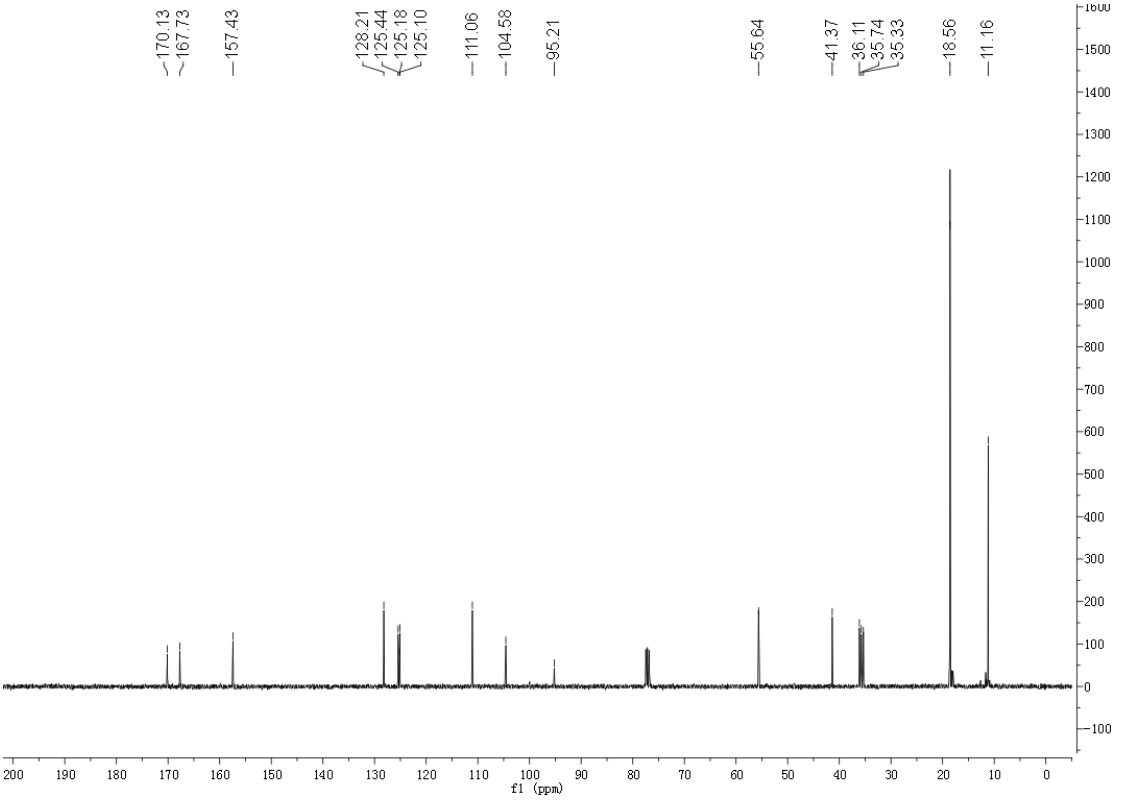

O
N
H
O
N
6b

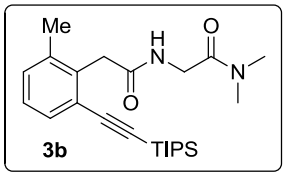

7.26
6.58
4.04
4.03
4.02
4.01
2.99
2.98
2.97
2.96
2.24
2.22
2.20
2.19
1.63
1.45
1.40
1.11
0.91
0.89
0.87
0.87
0.85
1.00
2.08
6.08
1.06
1.04
1.05
3.09
3.06
f1 (ppm)

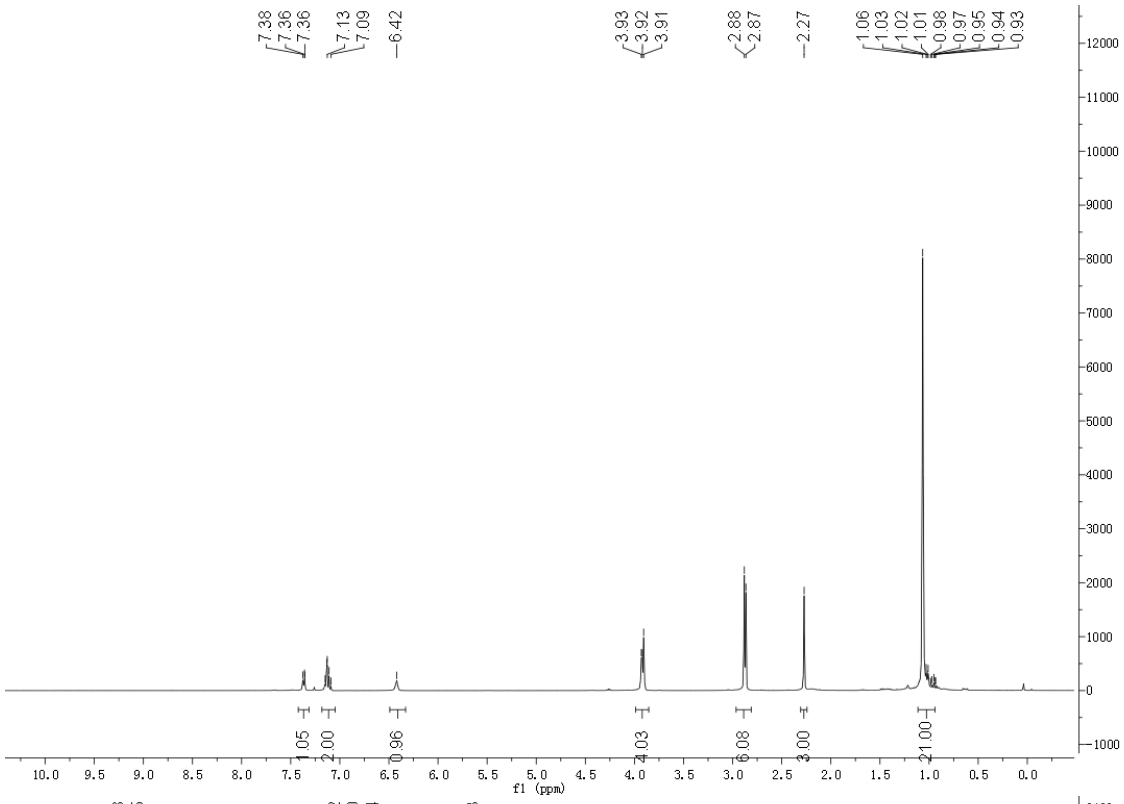

176.61
168.29
77.48
77.16
76.84
43.02
41.28
35.99
35.65
27.39
17.48
12.01
f1 (ppm)

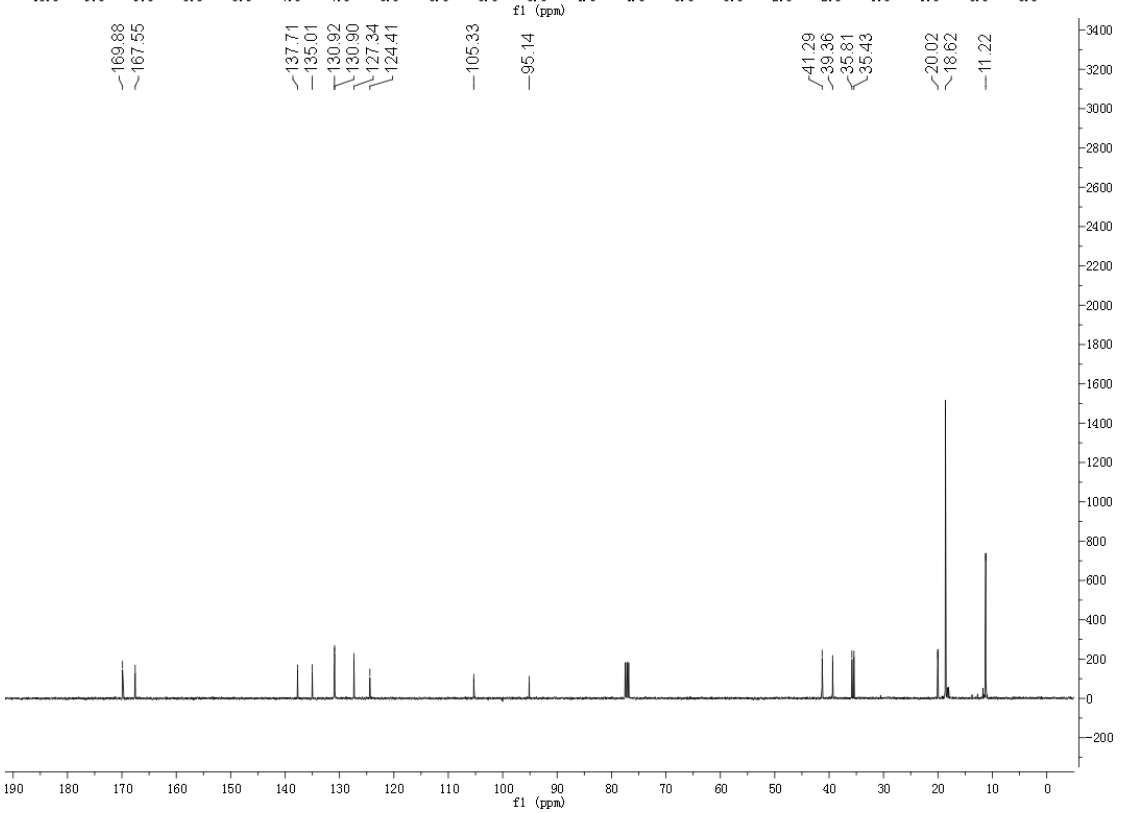

OMe
H
N
O
O
N
3a
TIPS

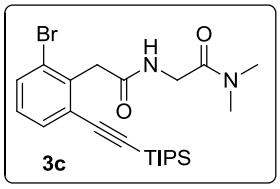

7.22
7.20
7.18
7.15
7.13
6.88
6.64
3.97
3.96
3.90
3.83
2.92
1.10
1.10
1.07
1.06
1.05
1.02
1.01
1.00
0.98
0.97
1.04
0.92
1.03
0.94
2.03
2.01
2.94
6.00
18.09
3.04
f1 (ppm)


170.13
167.73
157.43
128.21
125.44
125.18
125.10
111.06
104.58
95.21
55.64
41.37
36.11
35.74
35.33
18.56
11.16
f1 (ppm)

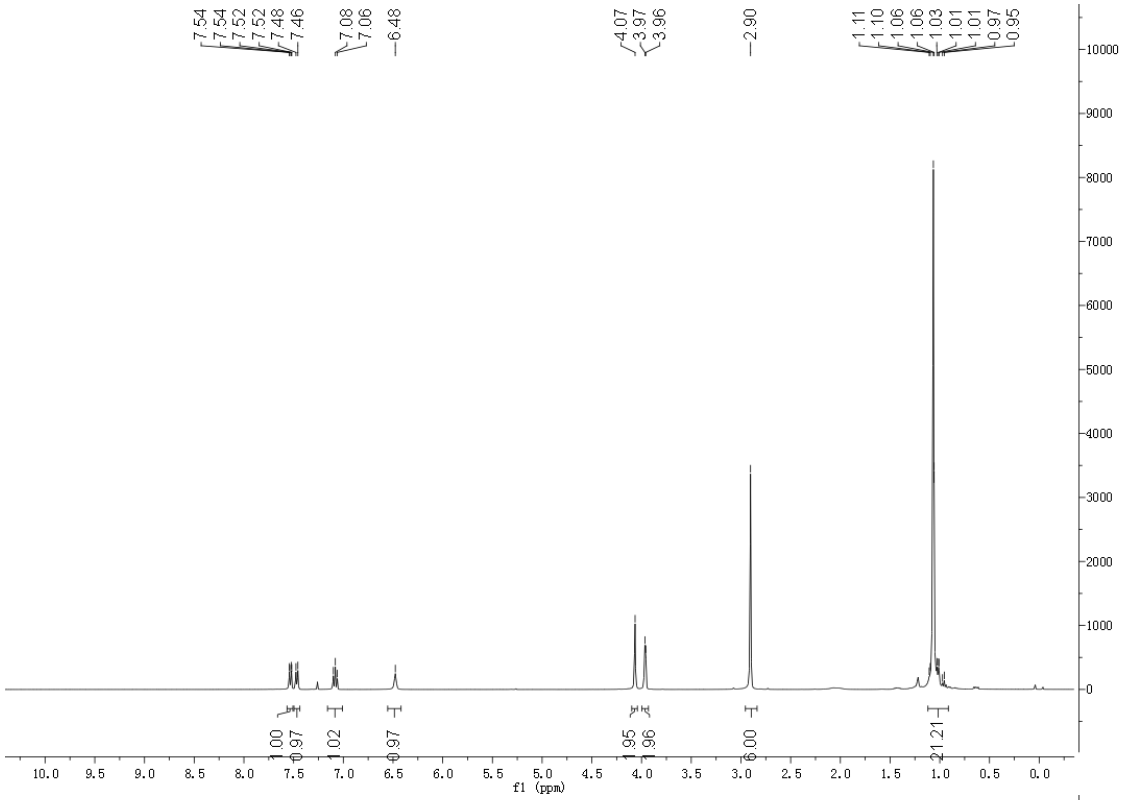

Me
H
N
O
O
N
3b
TIPS

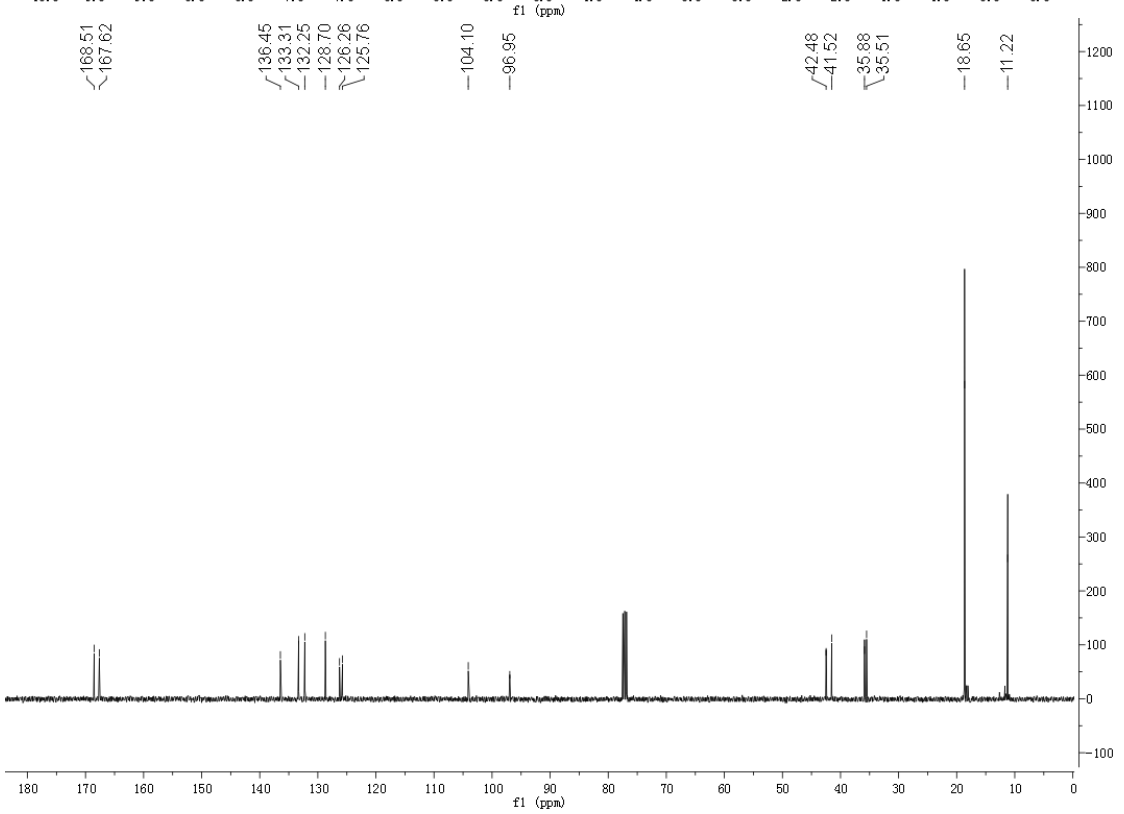

7.38
7.36
7.36
7.13
7.09
6.42
3.93
3.92
3.91
2.88
2.87
2.27
1.06
1.03
1.02
1.01
0.98
0.97
0.95
0.94
0.93
1.05
2.00
0.96
4.03
6.08
3.00
21.00
f1 (ppm)

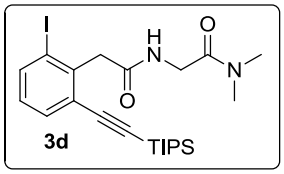

169.88
167.55
137.71
135.01
130.92
130.90
127.34
124.41
105.33
95.14
41.29
39.36
35.81
35.43
20.02
18.62
11.22
f1 (ppm)

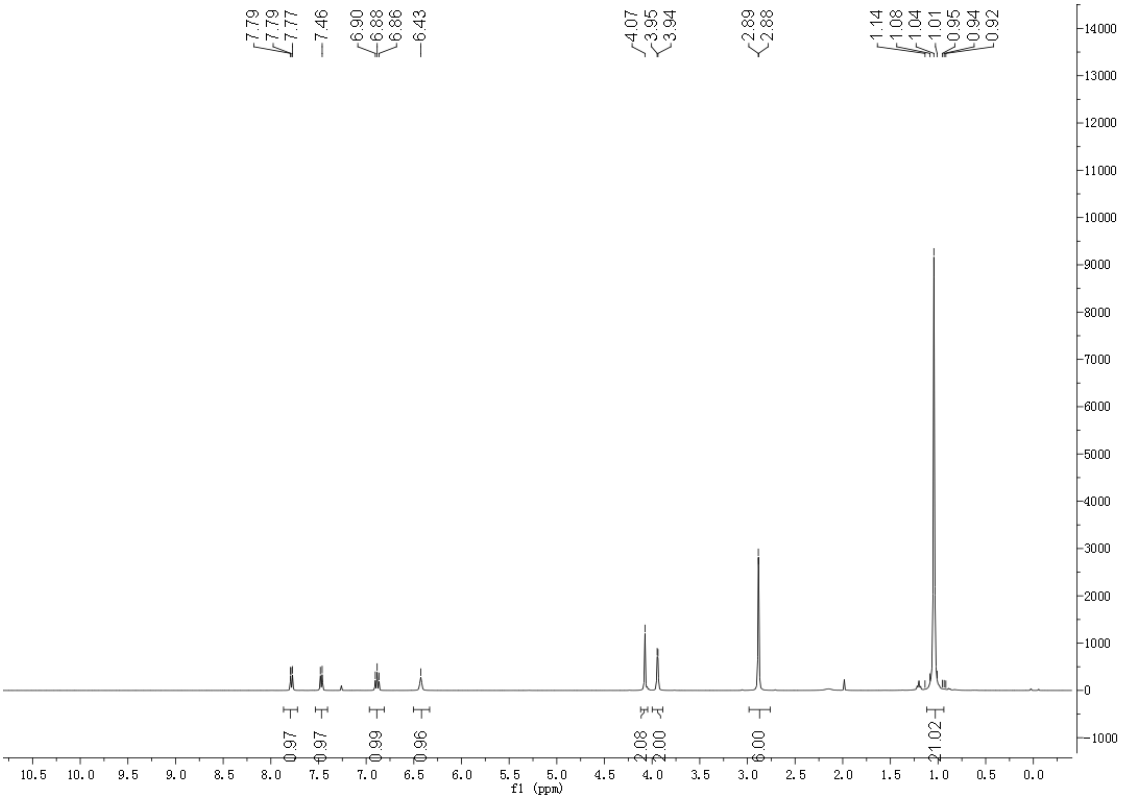

Br
H
N
O
O
N
TIPS

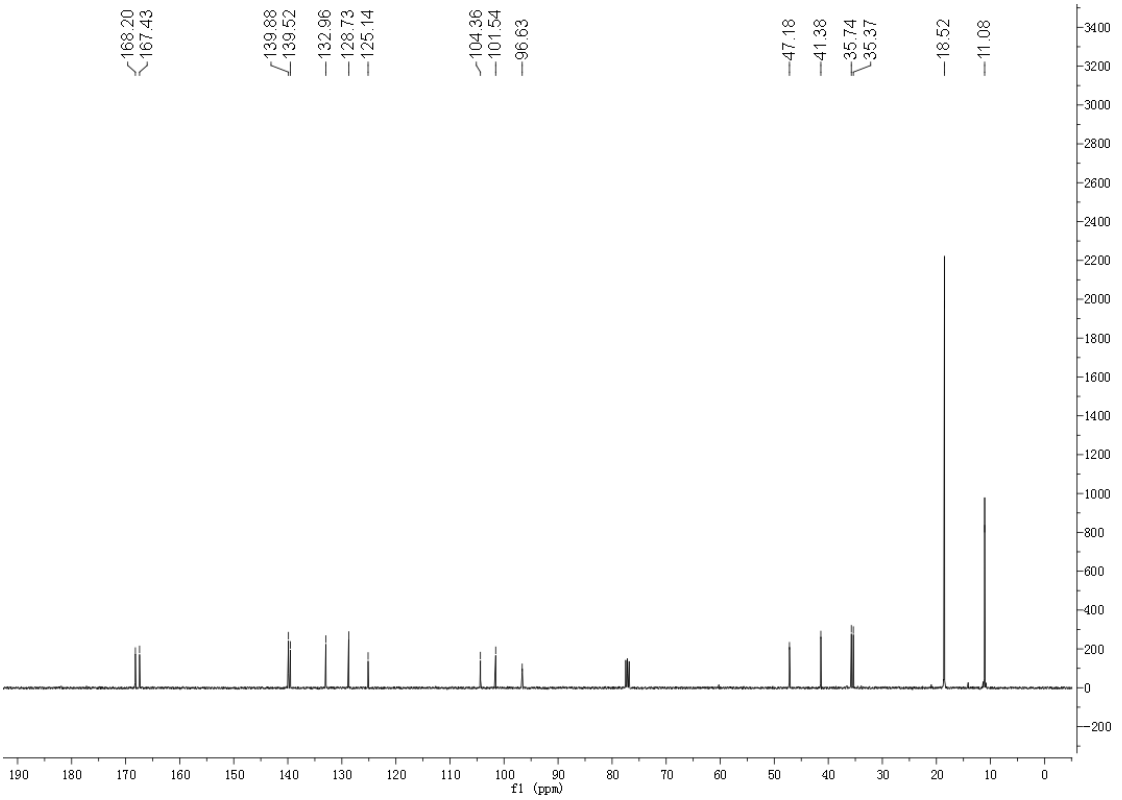
3c

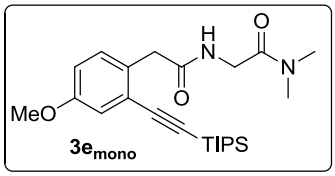

7.54
7.54
7.52
7.52
7.48
7.46
7.08
7.06
6.48
4.07
3.97
3.96
2.90
1.11
1.10
1.06
1.06
1.03
1.01
1.01
0.97
0.95
1.00
0.97
1.02
0.97
1.95
1.96
6.00
21.21
f1 (ppm)

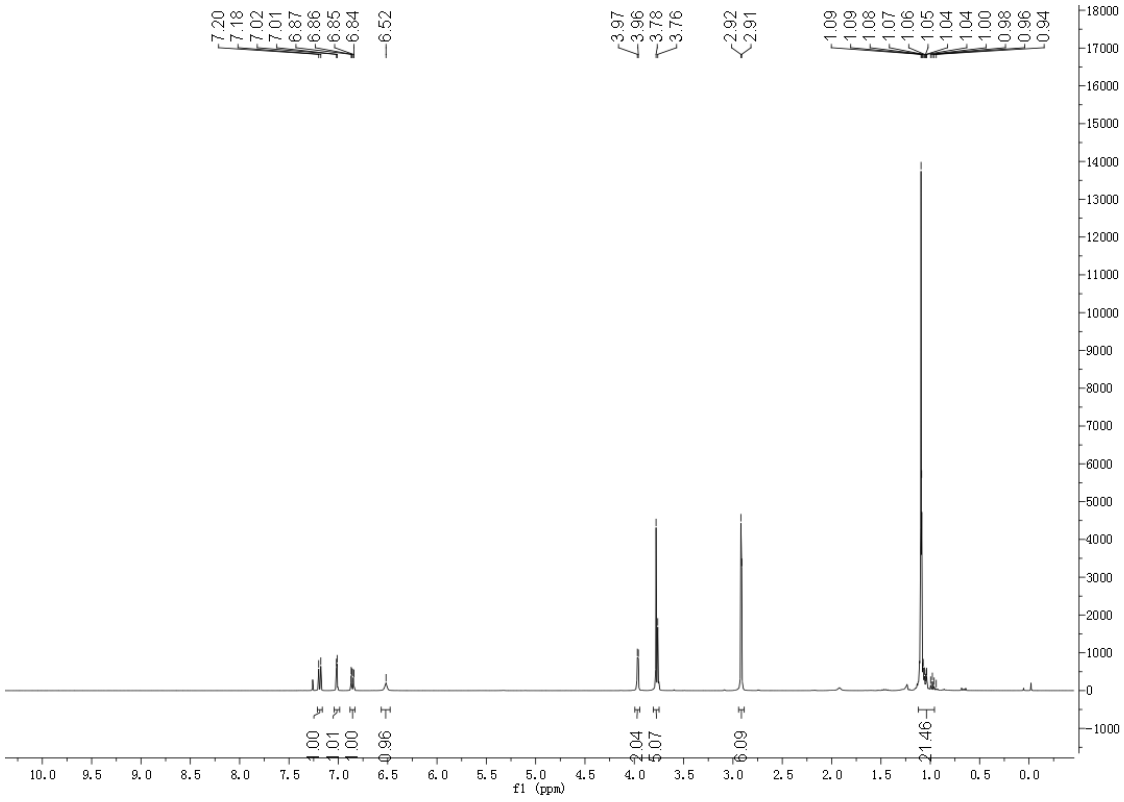

168.51
167.62
136.45
133.31
132.25
128.70
126.26
125.76
104.10
96.95
42.48
41.52
35.88
35.51
18.65
11.22
f1 (ppm)

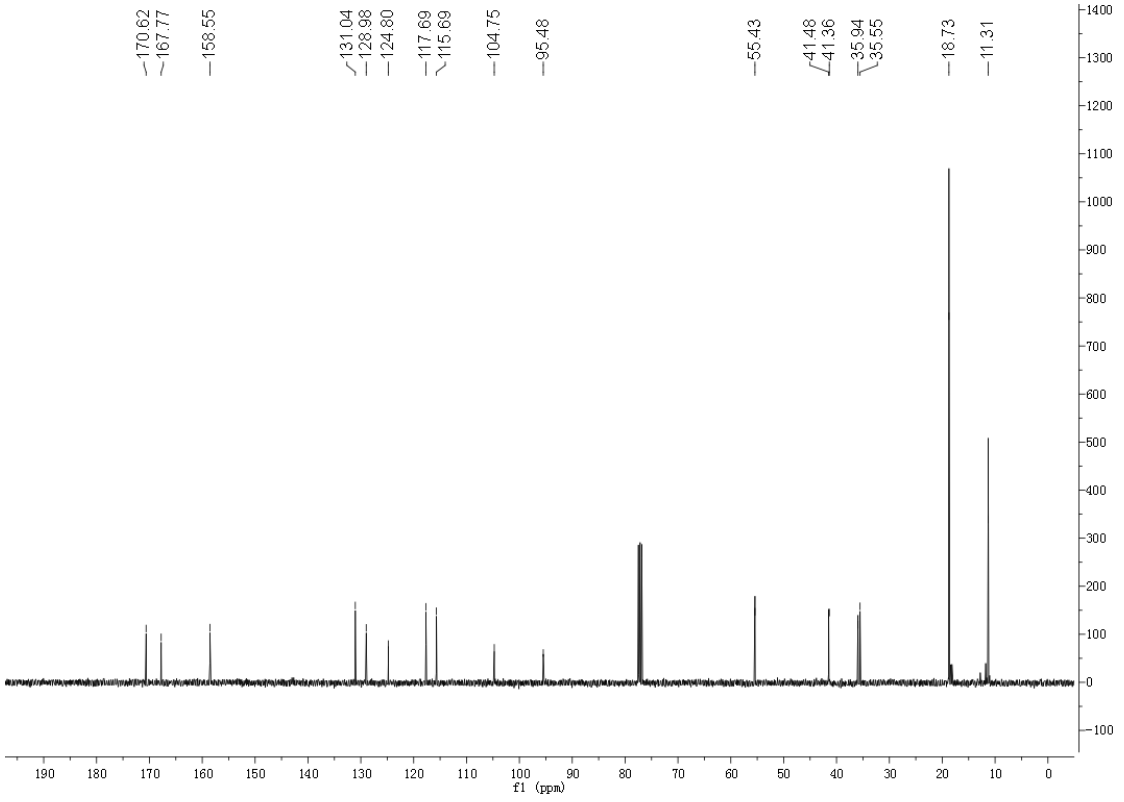

I
H
N
O
O
N
3d
TIPS

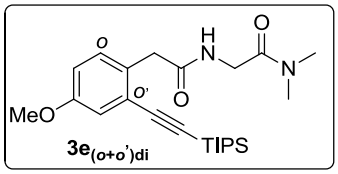

7.79
7.79
7.77
7.46
6.90
6.88
6.86
6.43
4.07
3.95
3.94
2.89
2.88
1.14
1.08
1.04
1.01
0.95
0.94
0.92
0.97
0.97
0.99
0.96
2.08
2.00
6.00
21.02
f1 (ppm)


168.20
167.43
139.88
139.52
132.96
128.73
125.14
104.36
101.54
96.63
47.18
41.38
35.74
35.37
18.52
11.08
f1 (ppm)

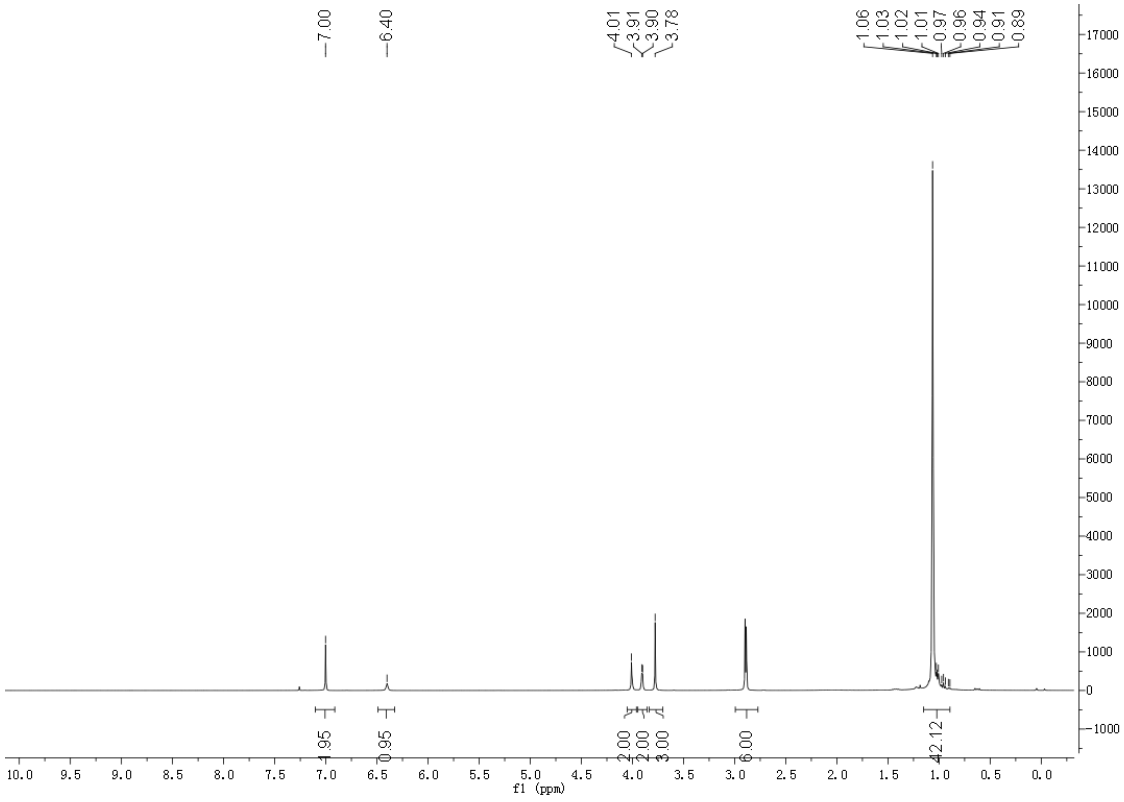

MeO
TIPS
3e mono

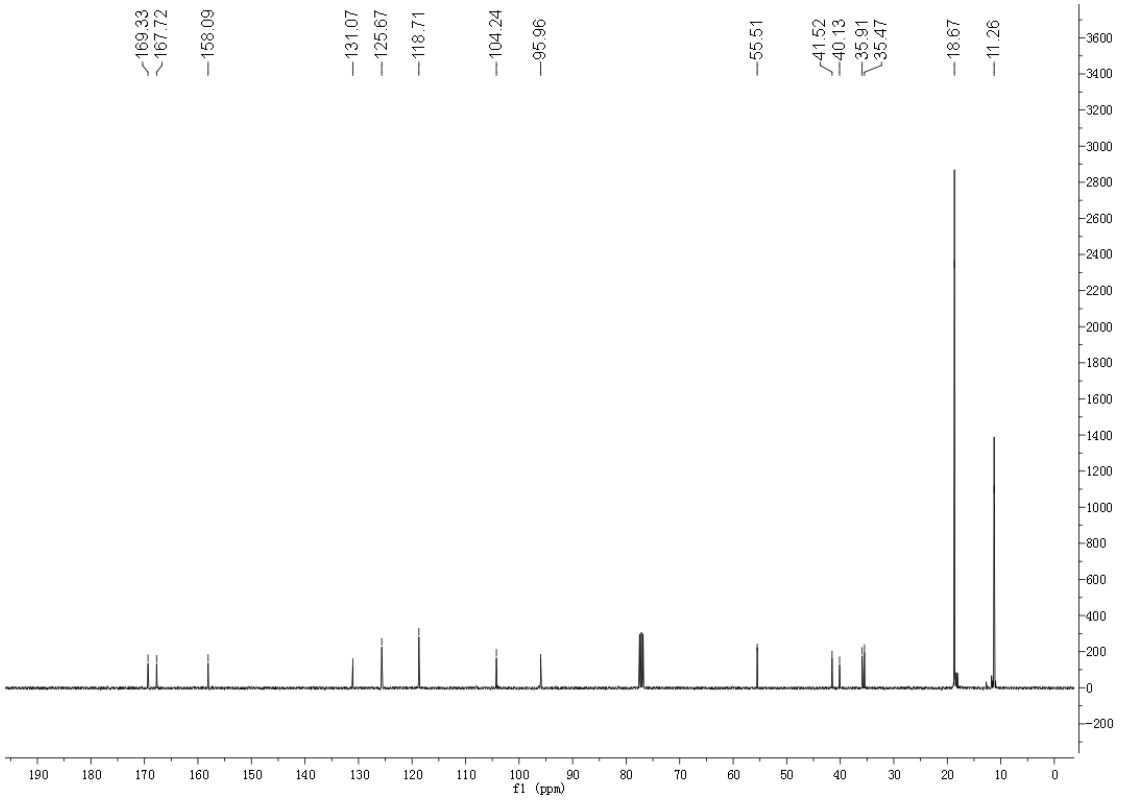

7.20
7.18
7.02
7.01
6.87
6.86
6.85
6.84
6.52
3.97
3.96
3.78
3.76
2.92
2.91
1.09
1.09
1.08
1.07
1.06
1.05
1.04
1.04
1.00
0.98
0.96
0.94
1.00
1.01
1.00
0.96
2.04
5.07
6.09
21.46
f1 (ppm)

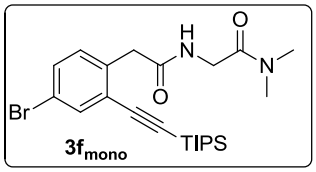

170.62
167.77
158.55
131.04
128.98
124.80
117.69
115.69
104.75
95.48
55.43
41.48
41.36
35.94
35.55
18.73
11.31
f1 (ppm)


O
H
N
O
N
O
O'
MeO
TIPS

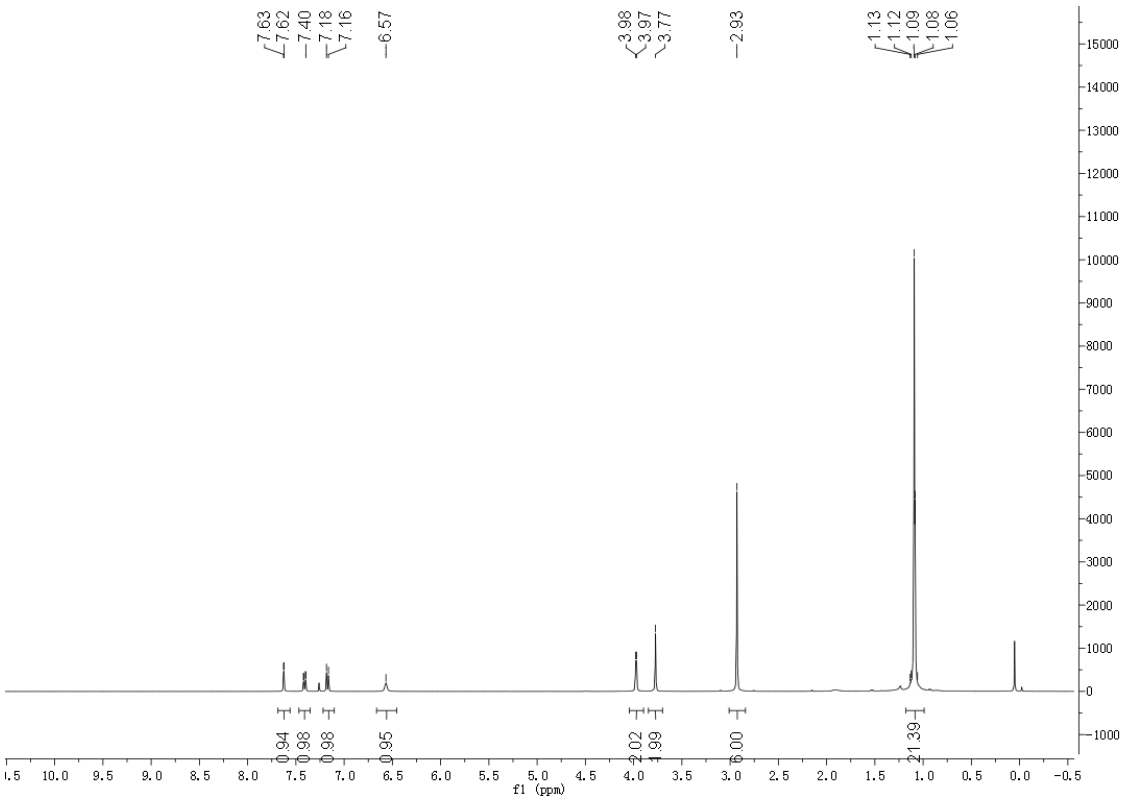
3e$_{(o+o')di}$

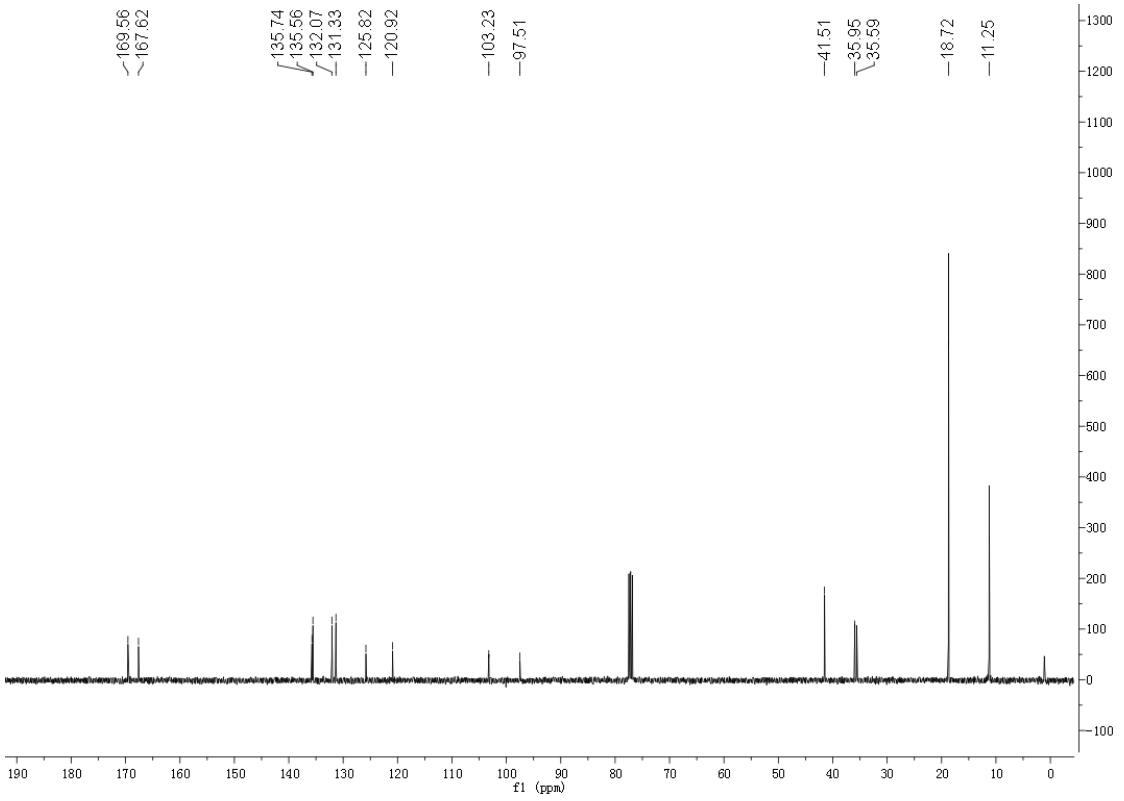

7.00
6.40
4.01
3.91
3.90
3.78
1.06
1.03
1.02
1.01
0.97
0.96
0.94
0.91
0.89
1.95
0.95
2.00
2.00
3.00
6.00
42.12
f1 (ppm)

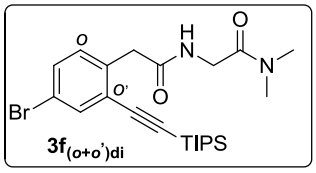

169.33
167.72
158.09
131.07
125.67
118.71
104.24
95.96
55.51
41.52
40.13
35.91
35.47
18.67
11.26
f1 (ppm)


H
N
O
O
N
Br
TIPS

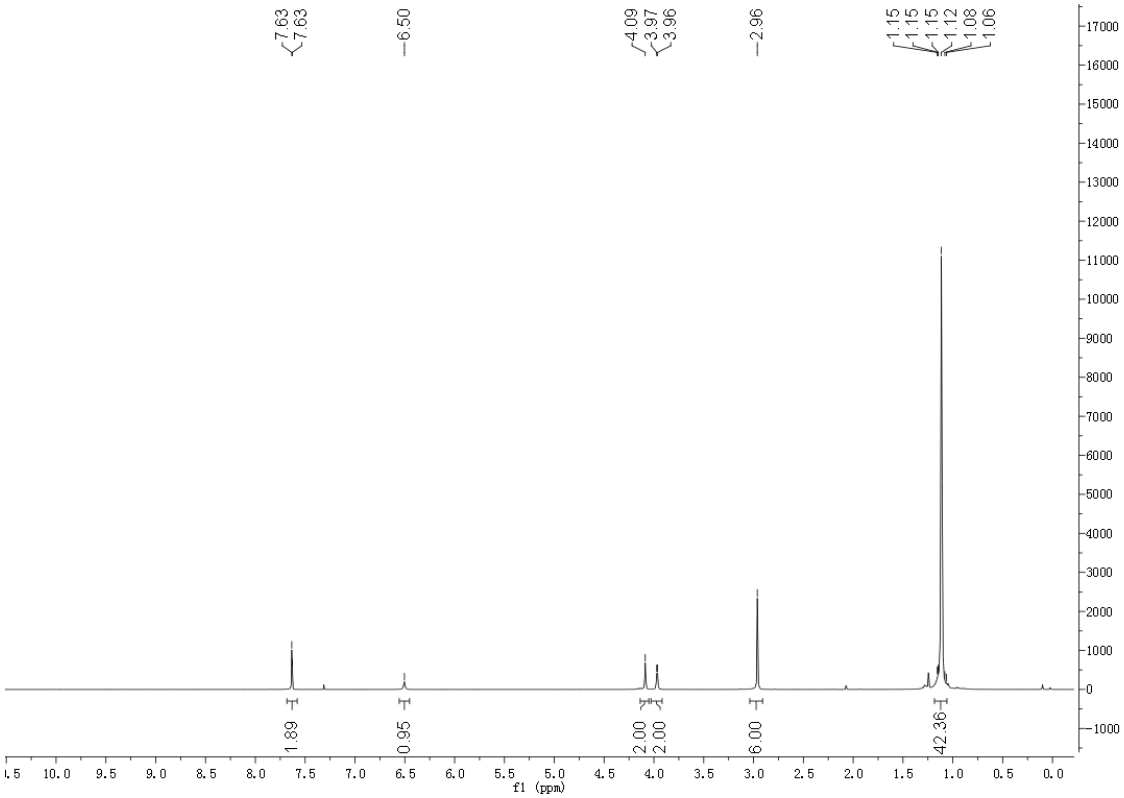
3f$_{mono}$

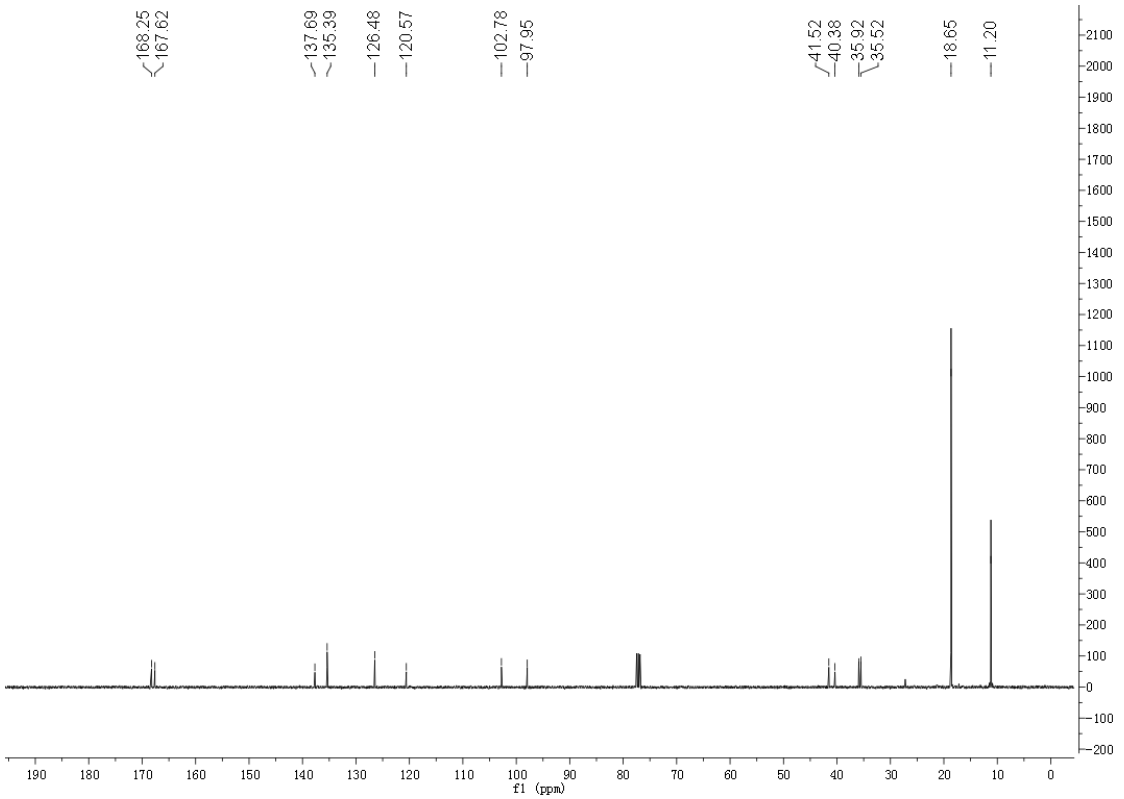

7.63
7.62
7.40
7.18
7.16
6.57
3.98
3.97
3.77
2.93
1.13
1.12
1.09
1.08
1.06
0.94
0.98
0.98
0.95
2.02
1.99
6.00
21.39
f1 (ppm)

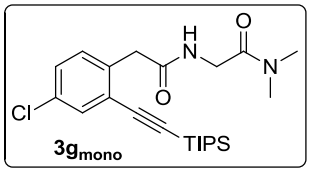

169.56
167.62
135.74
135.56
132.07
131.33
125.82
120.92
103.23
97.51
41.51
35.95
35.59
18.72
11.25
f1 (ppm)


O
H
N
O
N
O
O'
Br
TIPS

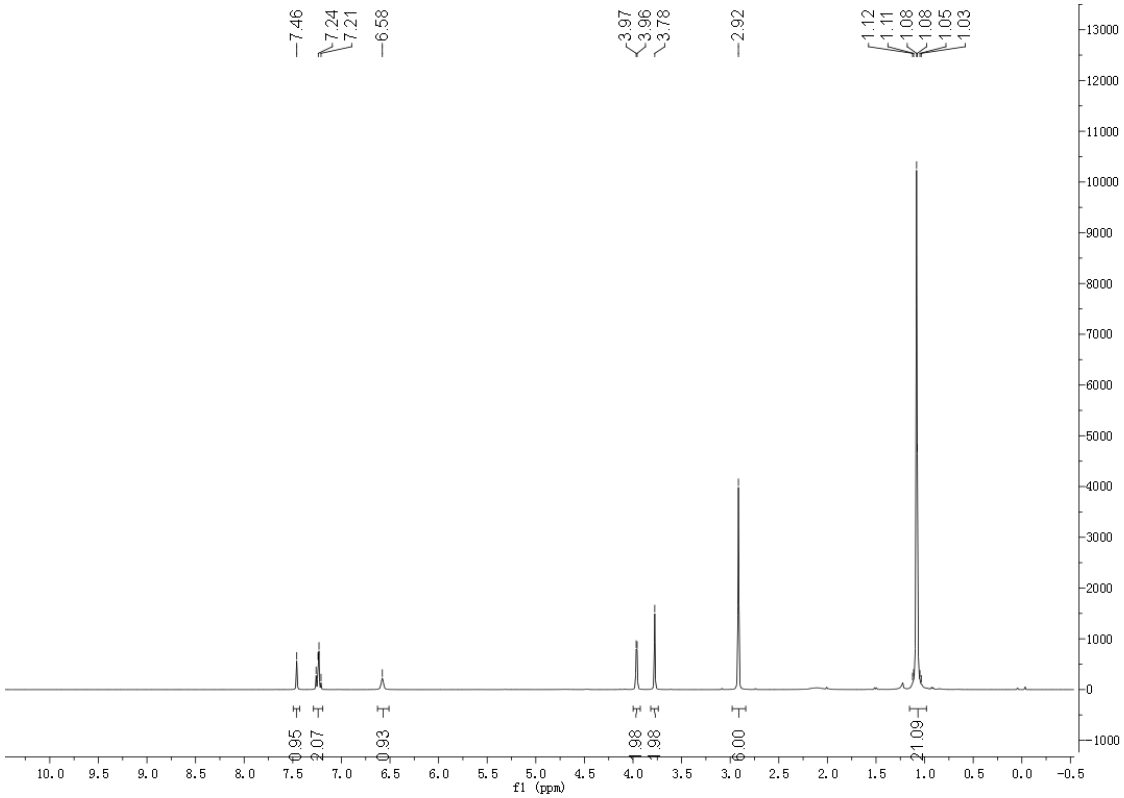
3f$_{(o+o')di}$

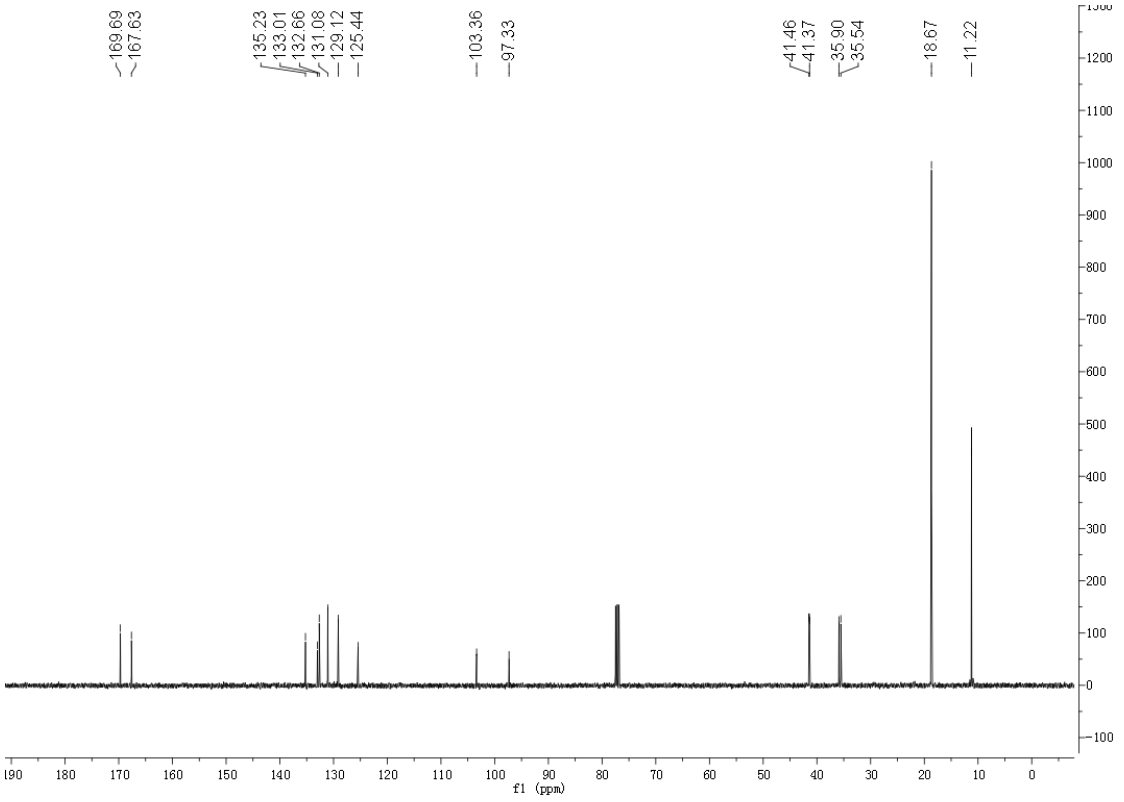

7.63
7.63
6.50
4.09
3.97
3.96
2.96
1.15
1.15
1.15
1.12
1.08
1.06
1.89
0.95
2.00
2.00
6.00
42.36
f1 (ppm)

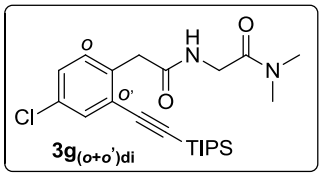

168.25
167.62
137.69
135.39
126.48
120.57
102.78
97.95
41.52
40.38
35.92
35.52
18.65
11.20
f1 (ppm)


H
N
O
O
N
Cl
TIPS

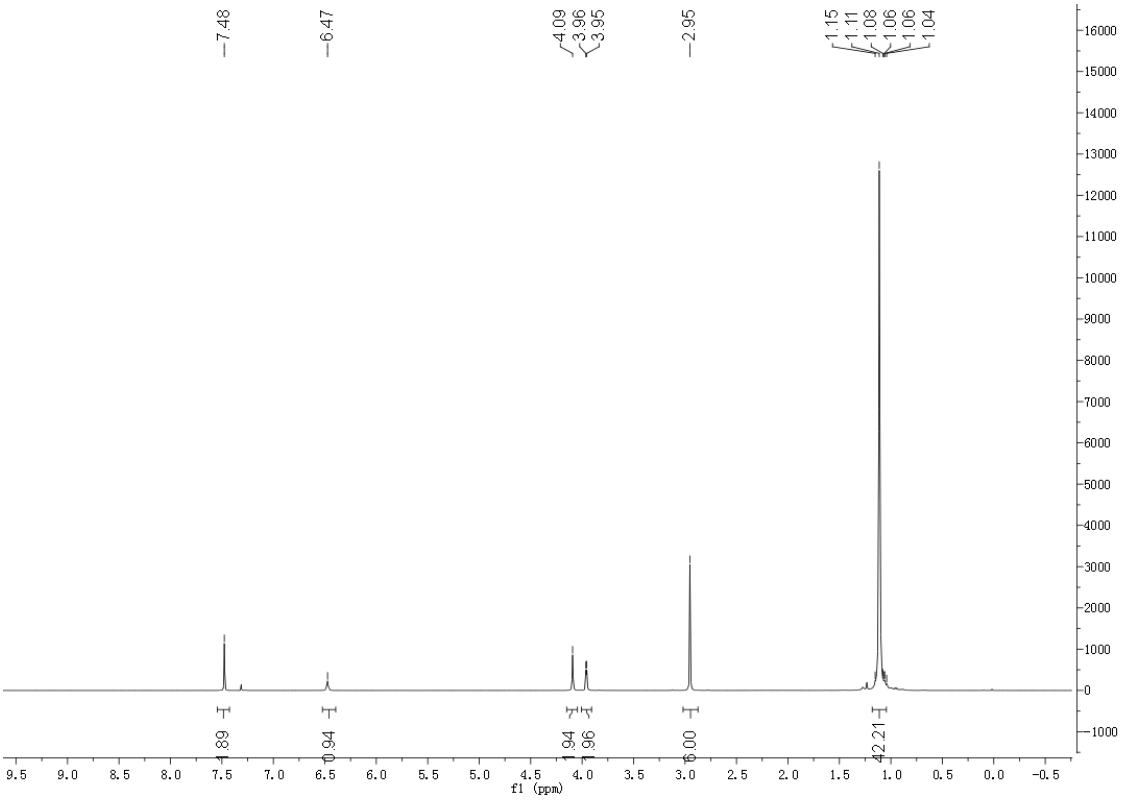
3g$_{mono}$

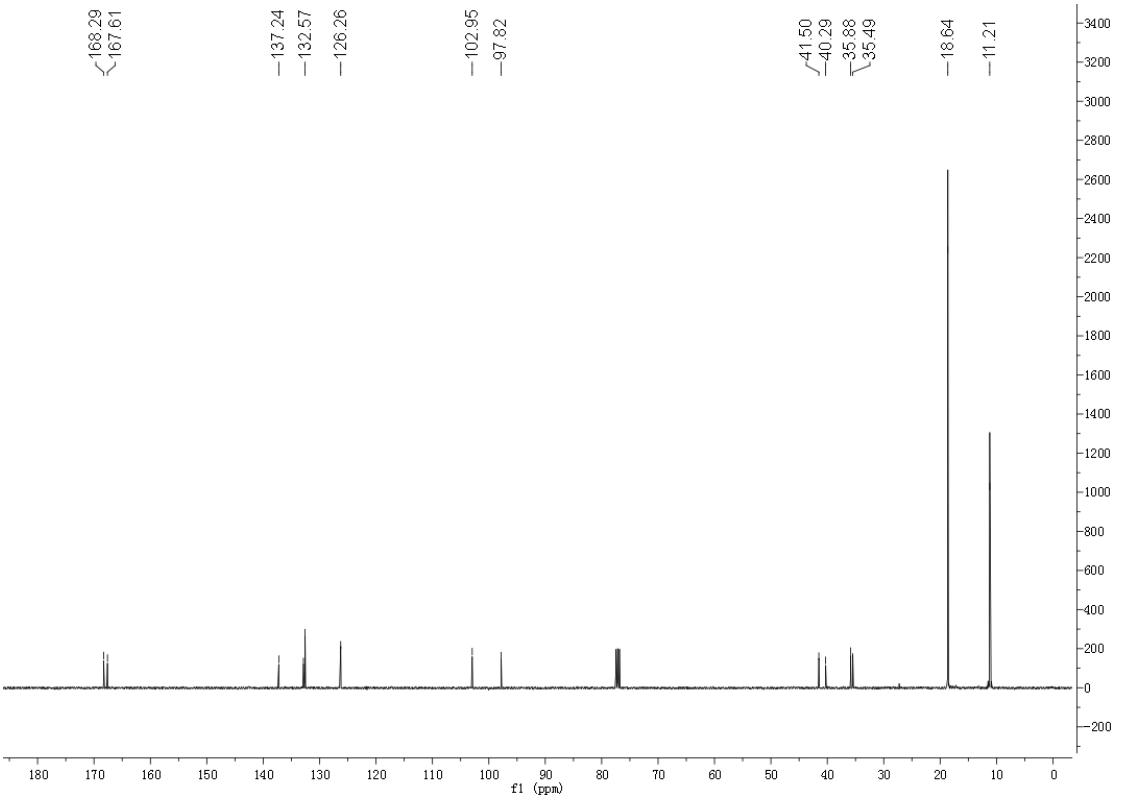

7.46
7.24
7.21
6.58
3.97
3.96
3.78
2.92
1.12
1.11
1.08
1.08
1.05
1.03
0.95
2.07
0.93
1.98
1.98
6.00
21.09
f1 (ppm)

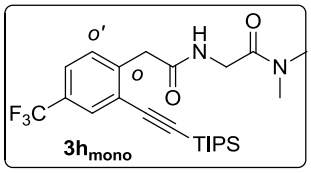

169.69
167.63
135.23
133.01
132.66
131.08
129.12
125.44
103.36
97.33
41.46
41.37
35.90
35.54
18.67
11.22
f1 (ppm)

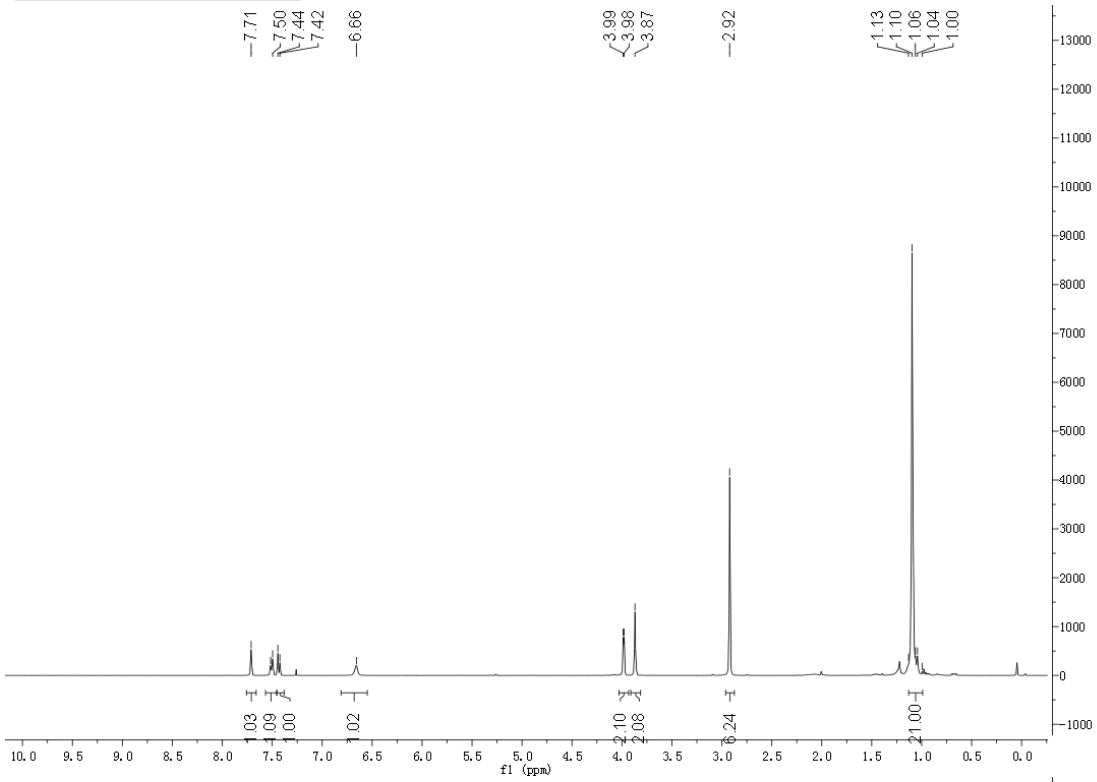

O
H
N
O
N
O
O'
Cl
TIPS

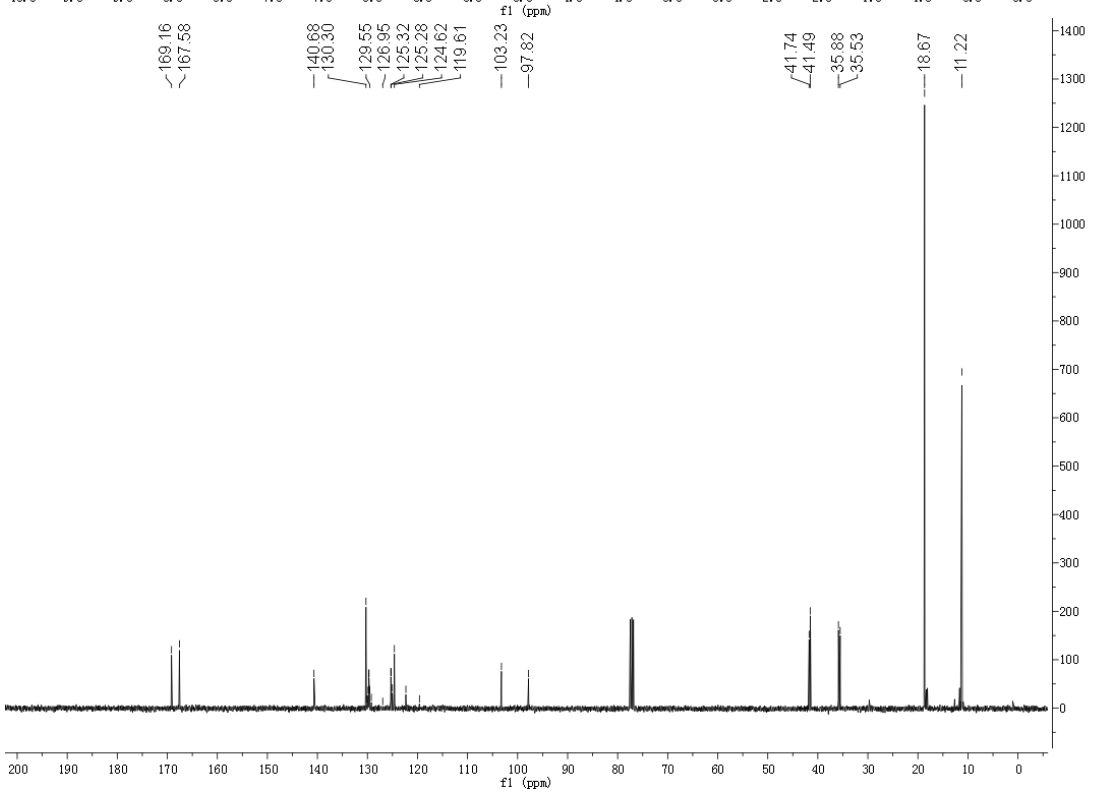
3g$_{(o+o')di}$

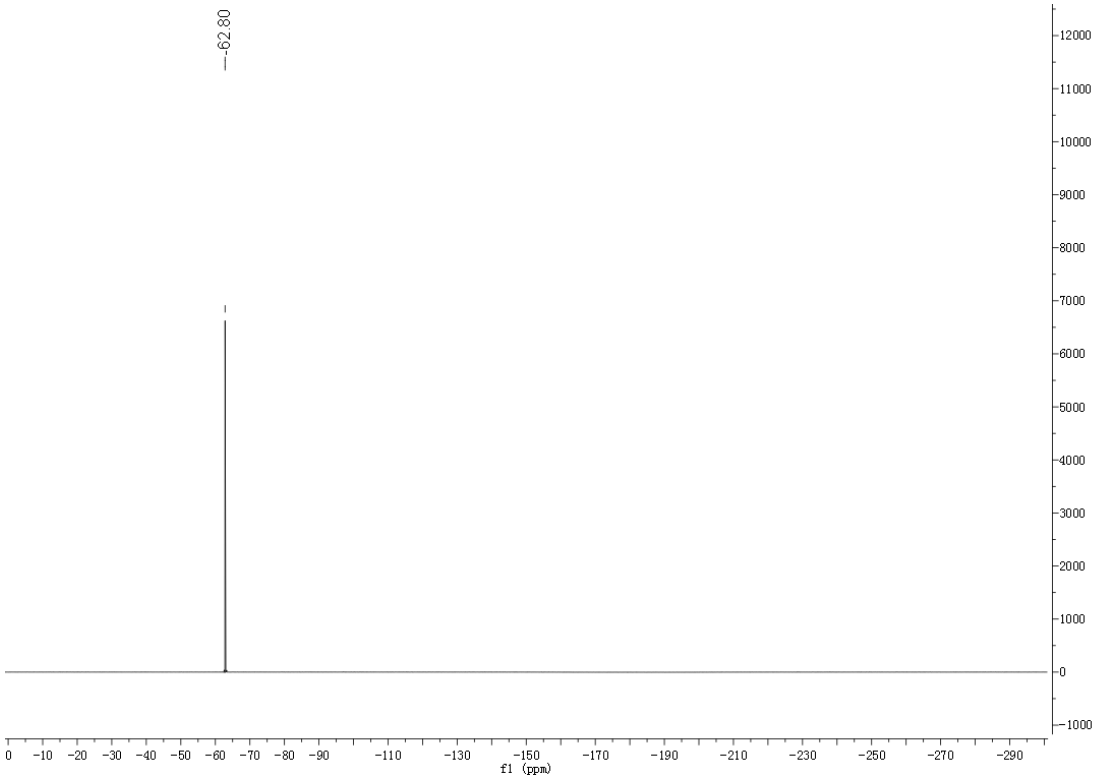

7.48
6.47
4.09
3.96
3.95
2.95
1.15
1.11
1.08
1.06
1.06
1.04
1.89
0.94
1.94
1.96
6.00
42.21
f1 (ppm)

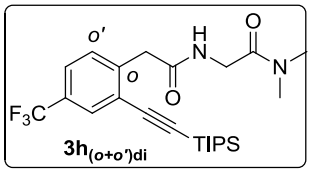

168.29
167.61
137.24
132.57
126.26
102.95
97.82
41.50
40.29
35.88
35.49
18.64
11.21
f1 (ppm)


o'
o
H
N
O
O
N
F3C
TIPS
3hmono

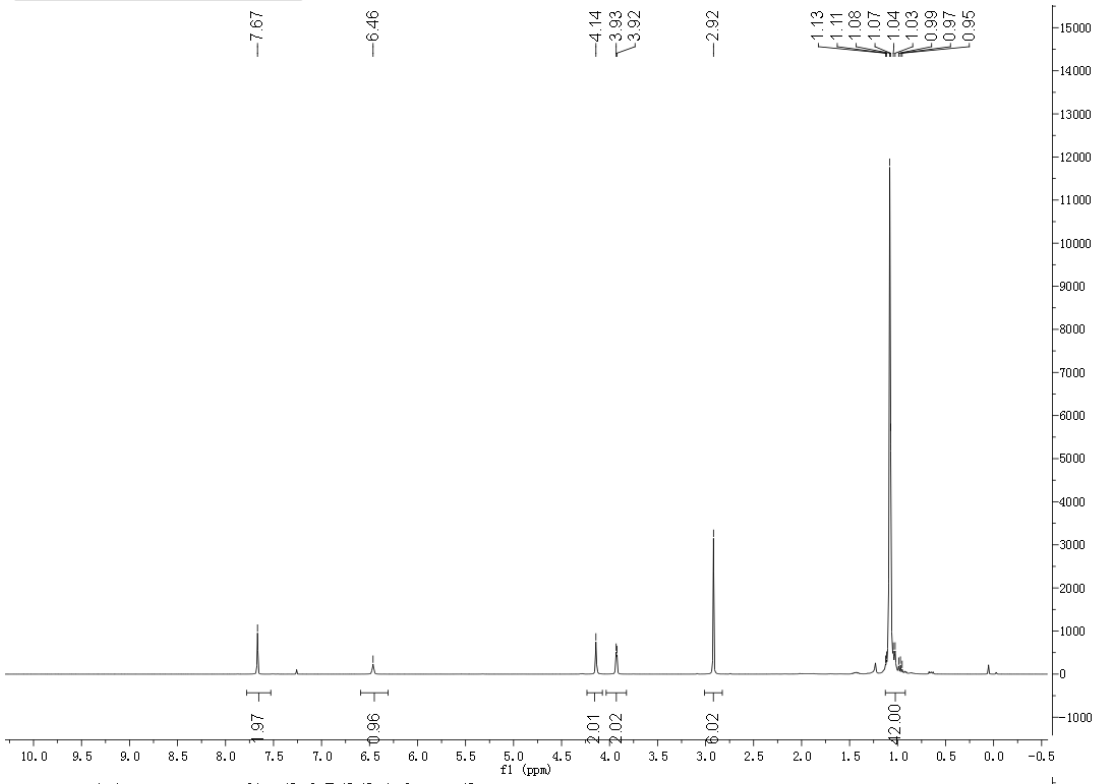

7.71
7.50
7.44
7.42
6.66
3.99
3.98
3.87
2.92
1.13
1.10
1.06
1.04
1.00
1.03
1.09
1.00
1.02
2.10
2.08
6.24
21.00
f1 (ppm)

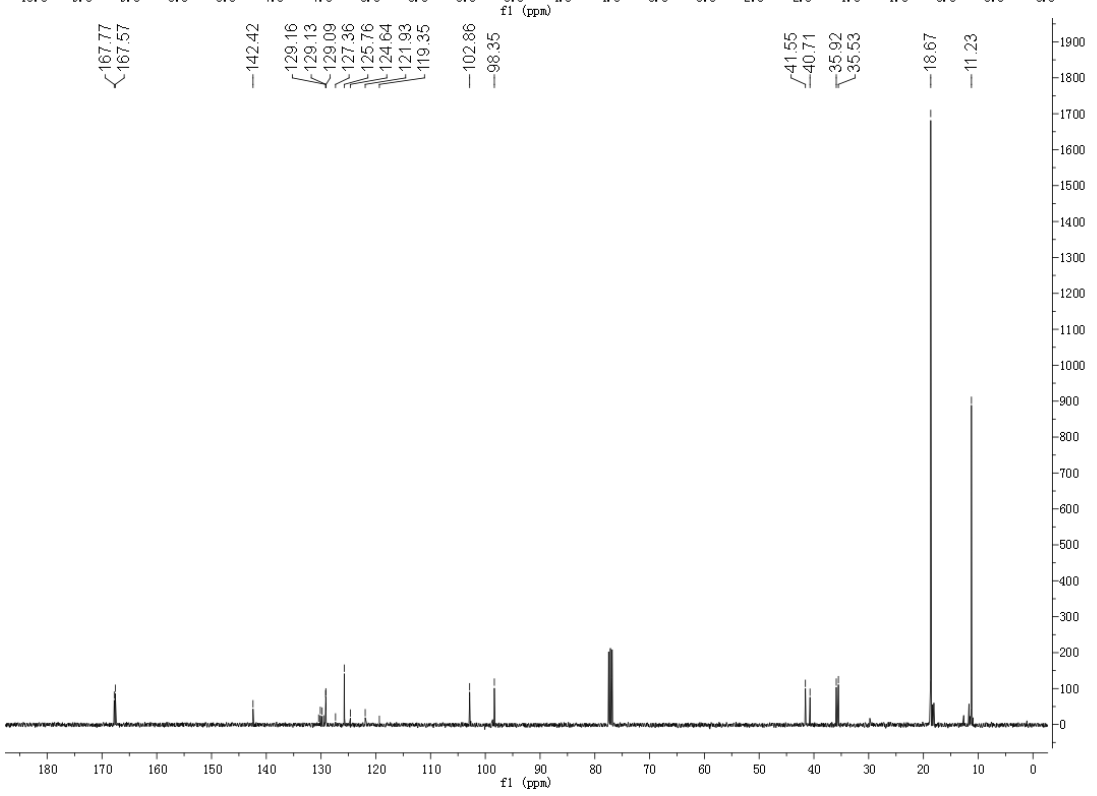

169.16
167.58
140.68
130.30
129.55
126.95
125.32
125.28
124.62
119.61
103.23
97.82
41.74
41.49
35.88
35.53
18.67
11.22
f1 (ppm)

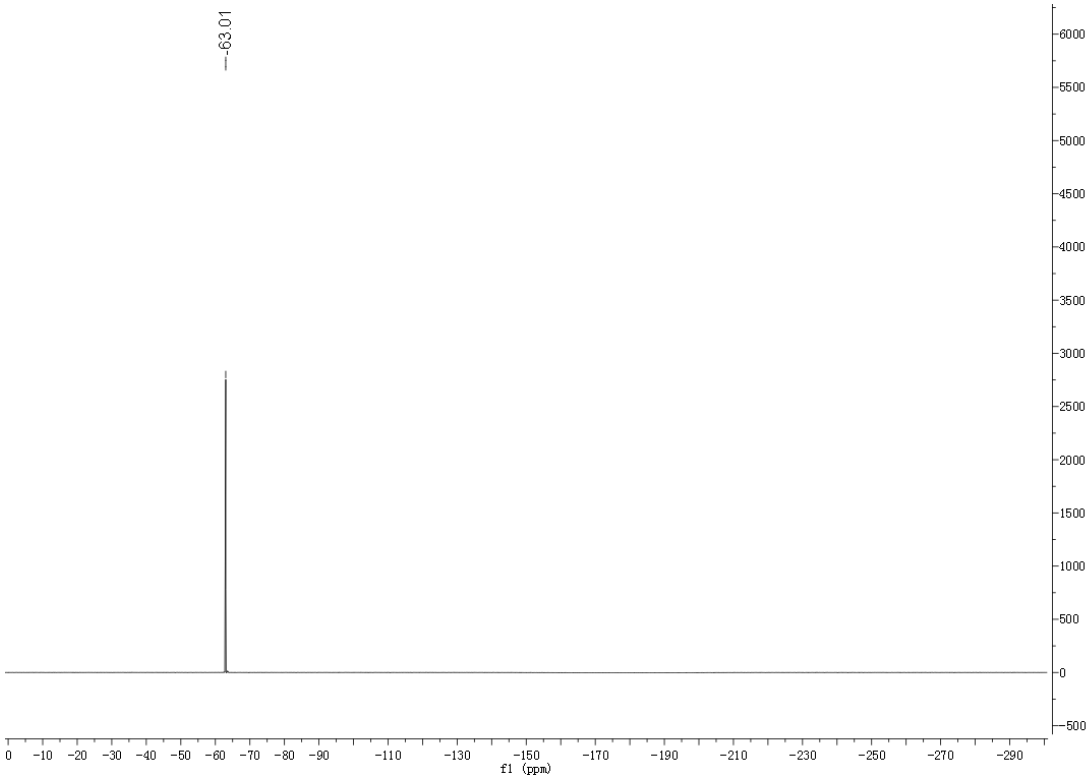

-62.80
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

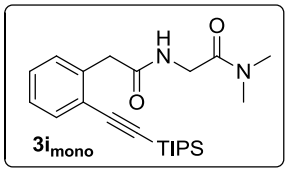

o'
o
H
N
O
O
N
F3C
TIPS

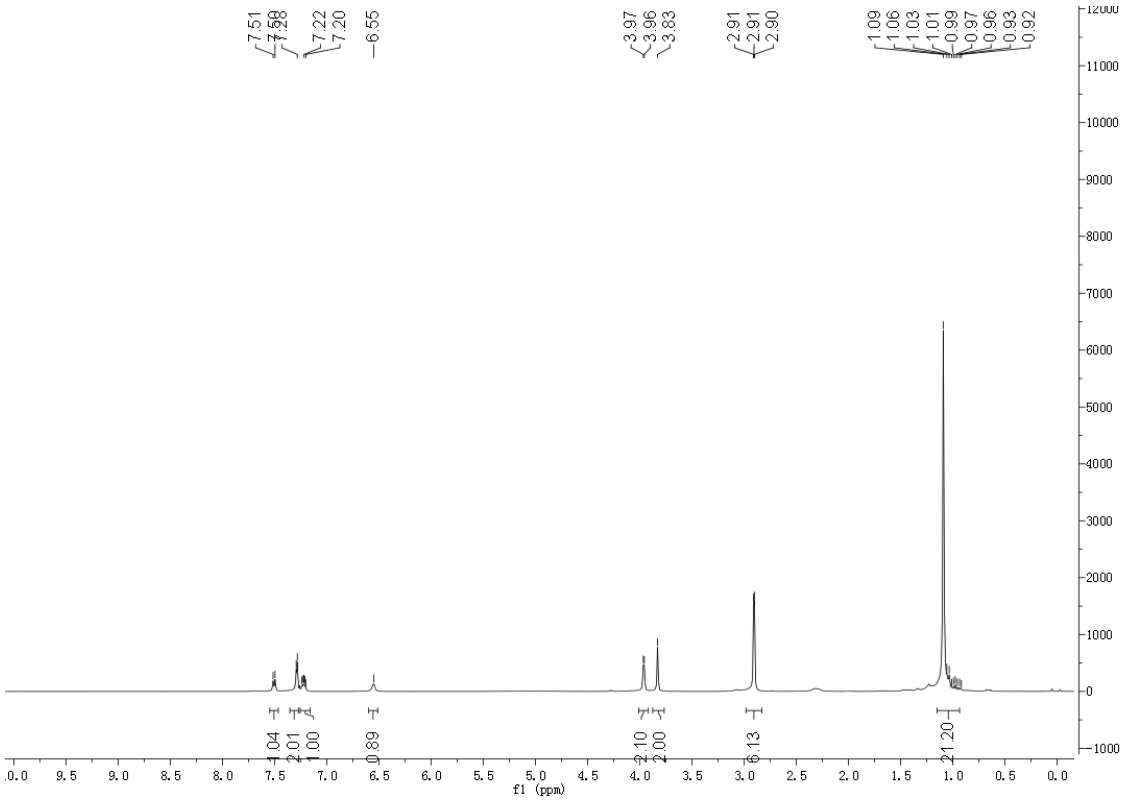
3h$_{(o+o')di}$

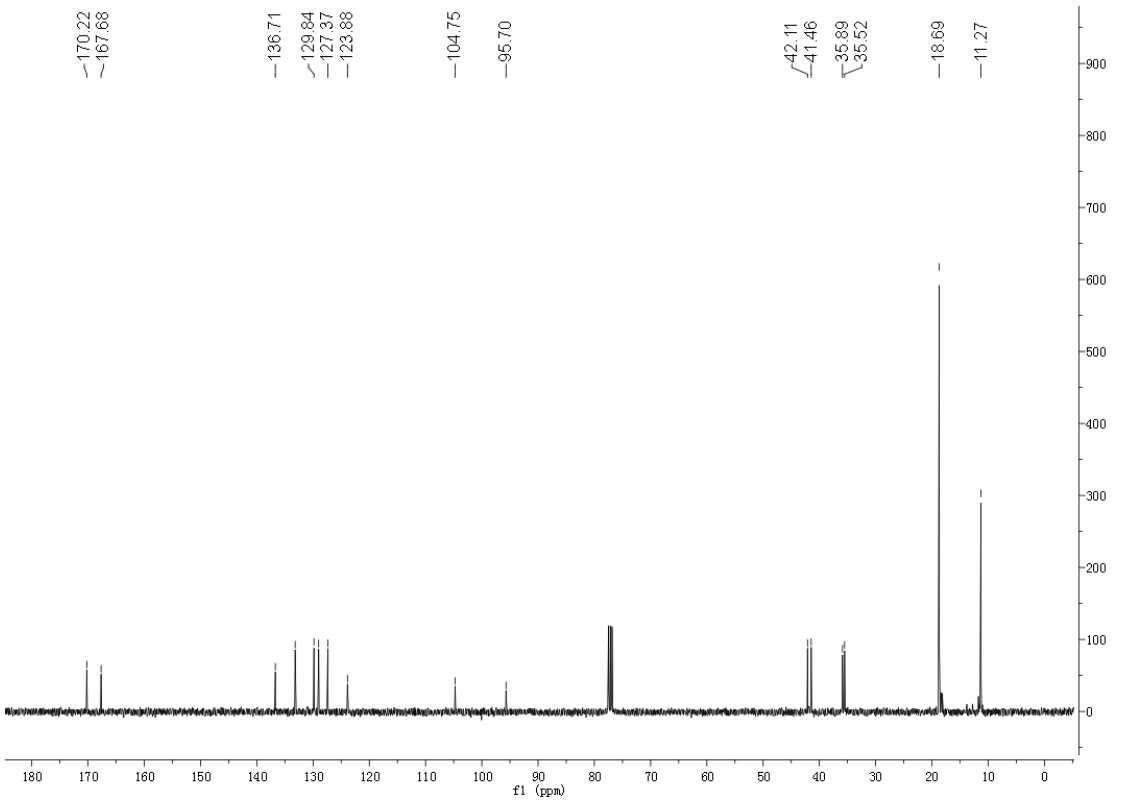

7.67
6.46
4.14
3.93
3.92
2.92
1.13
1.11
1.08
1.07
1.04
1.03
0.99
0.97
0.95
1.97
0.96
2.01
2.02
6.02
42.00
f1 (ppm)

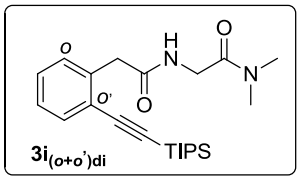

167.77
167.57
142.42
129.16
129.13
129.09
127.36
125.76
124.64
121.93
119.35
102.86
98.35
41.55
40.71
35.92
35.53
18.67
11.23
f1 (ppm)


-63.01
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

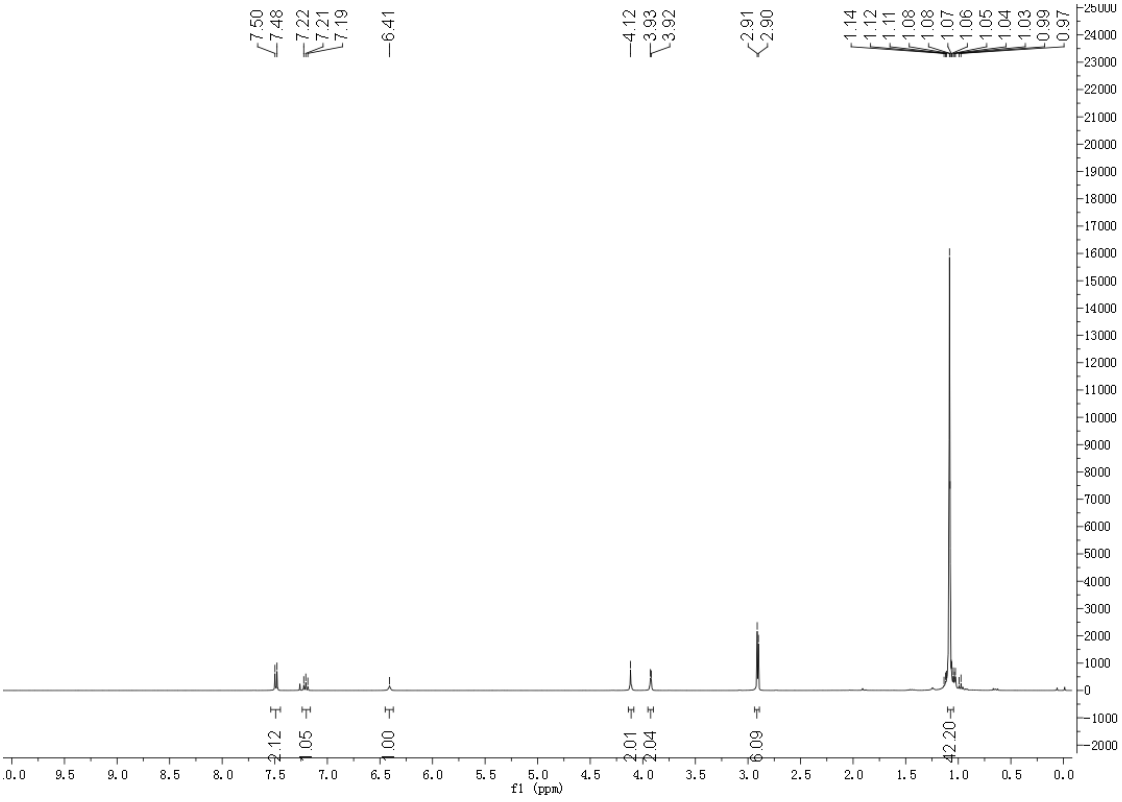

H
N
O
O
N
3i$_{mono}$
TIPS

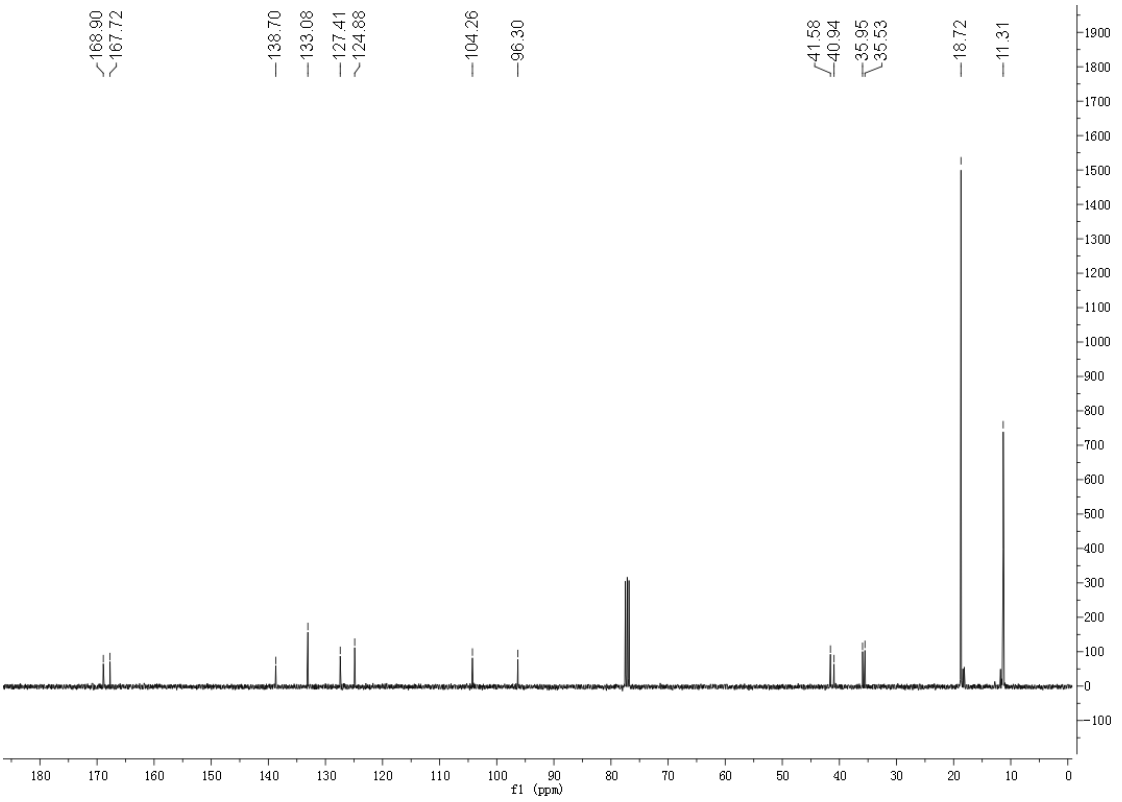

7.51
7.28
7.22
7.20
6.55
3.97
3.96
3.83
2.91
2.91
2.90
1.09
1.06
1.03
1.01
0.99
0.97
0.96
0.93
0.92
1.04
2.01
1.00
0.89
2.10
2.00
6.13
21.20
f1 (ppm)

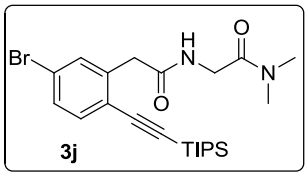

170.22
167.88
136.71
129.84
127.37
123.88
104.75
95.70
42.11
41.46
35.89
35.52
18.69
11.27
f1 (ppm)

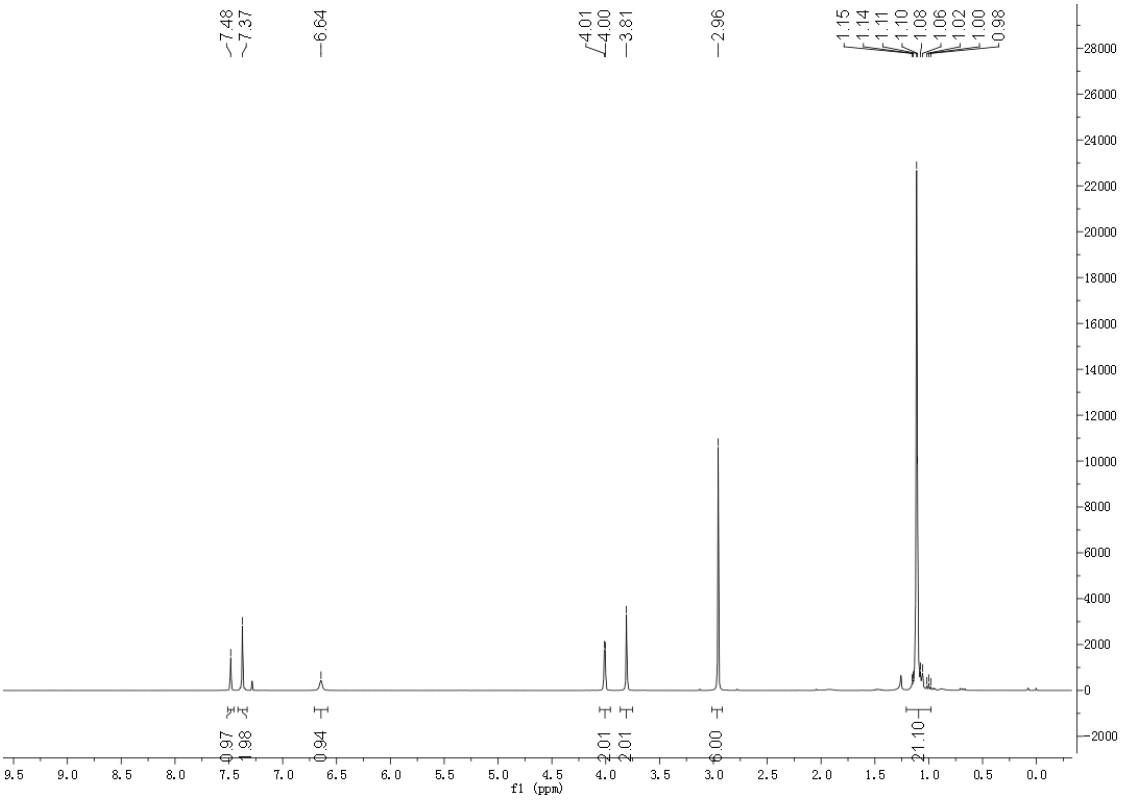

o
o'
H
N
O
O
N
TIPS

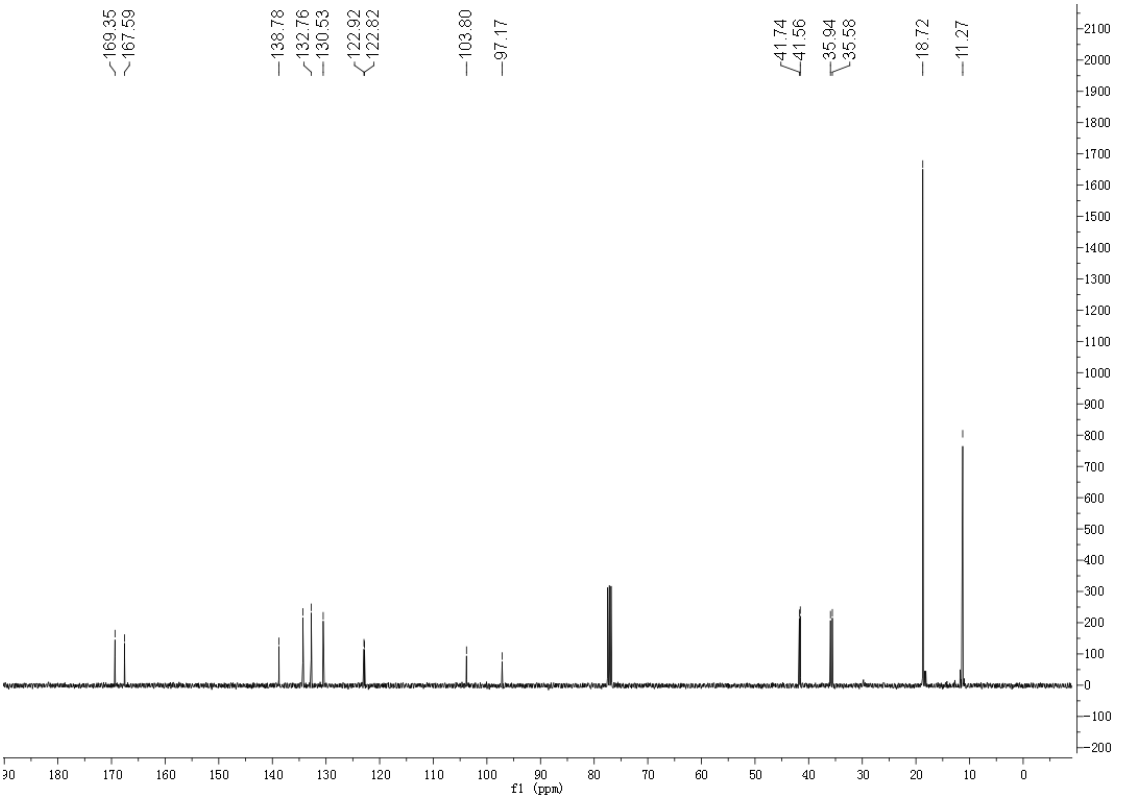
3i$_{(o+o')di}$

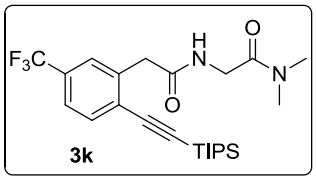

7.50
7.48
7.22
7.21
7.19
6.41
4.12
3.93
3.92
2.91
2.90
1.14
1.12
1.11
1.08
1.08
1.07
1.06
1.05
1.04
1.03
0.99
0.97
2.12
1.05
1.00
2.01
2.04
6.09
42.20
f1 (ppm)

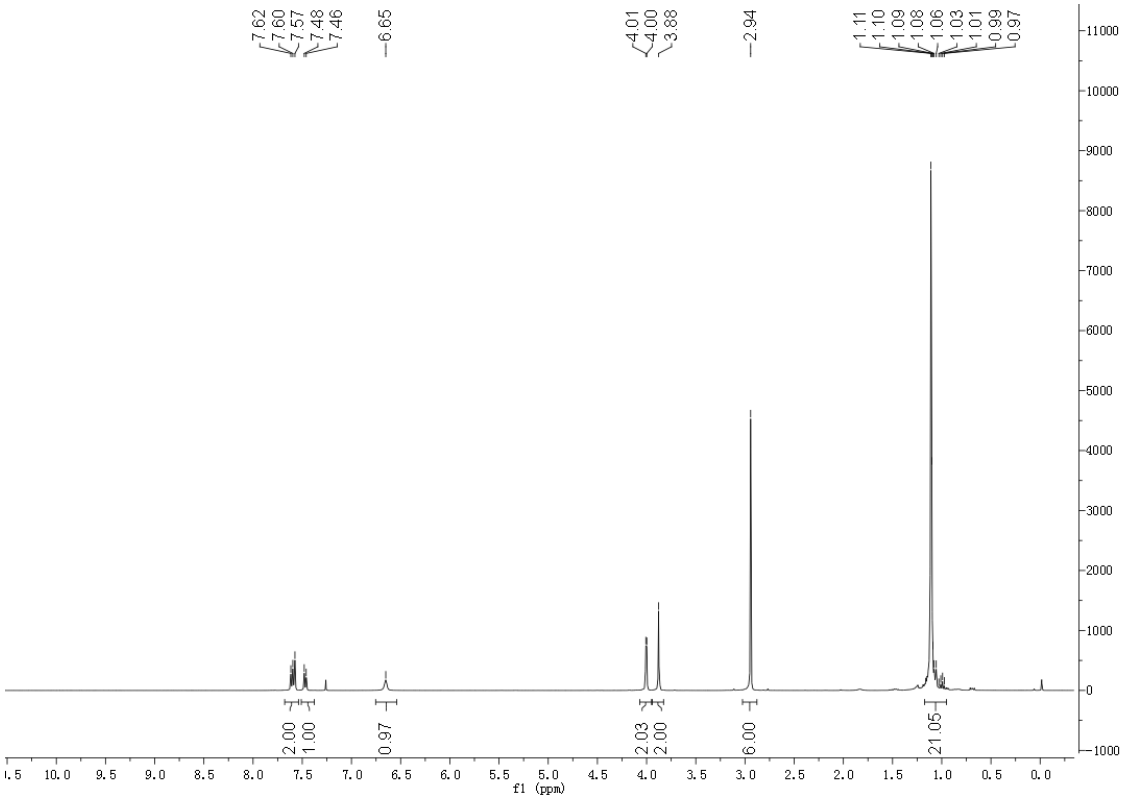

168.90
167.72
138.70
133.08
127.41
124.88
104.26
96.30
41.58
40.94
35.95
35.53
18.72
11.31
f1 (ppm)

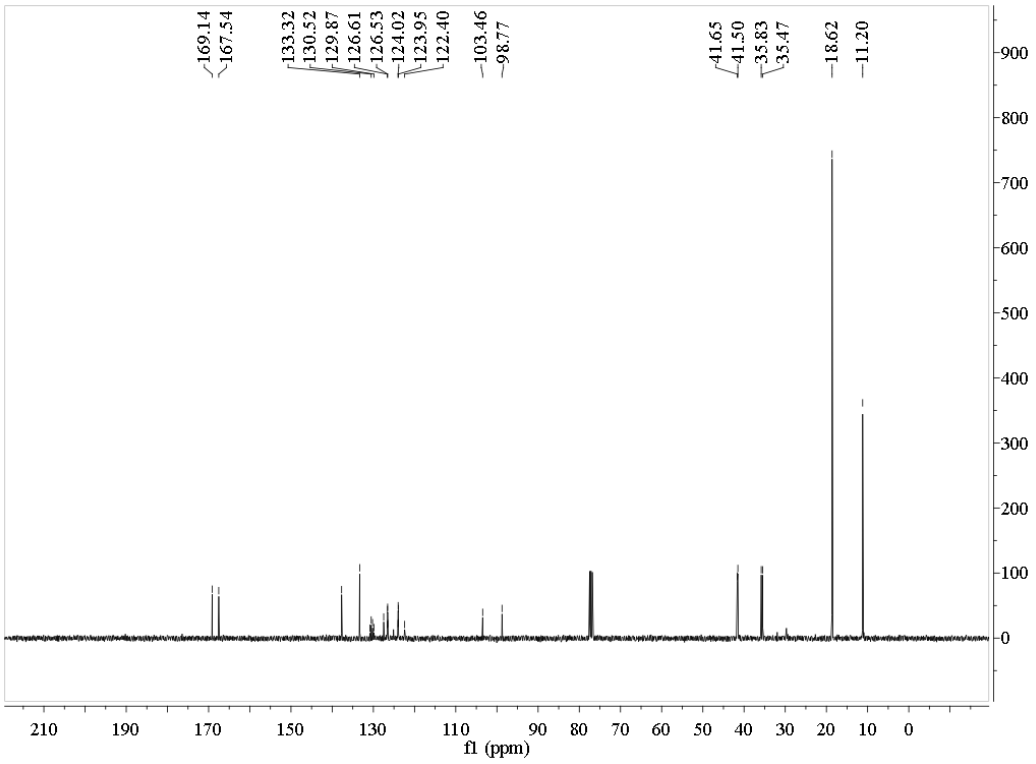

Br
H
N
O
O
N
3j
TIPS

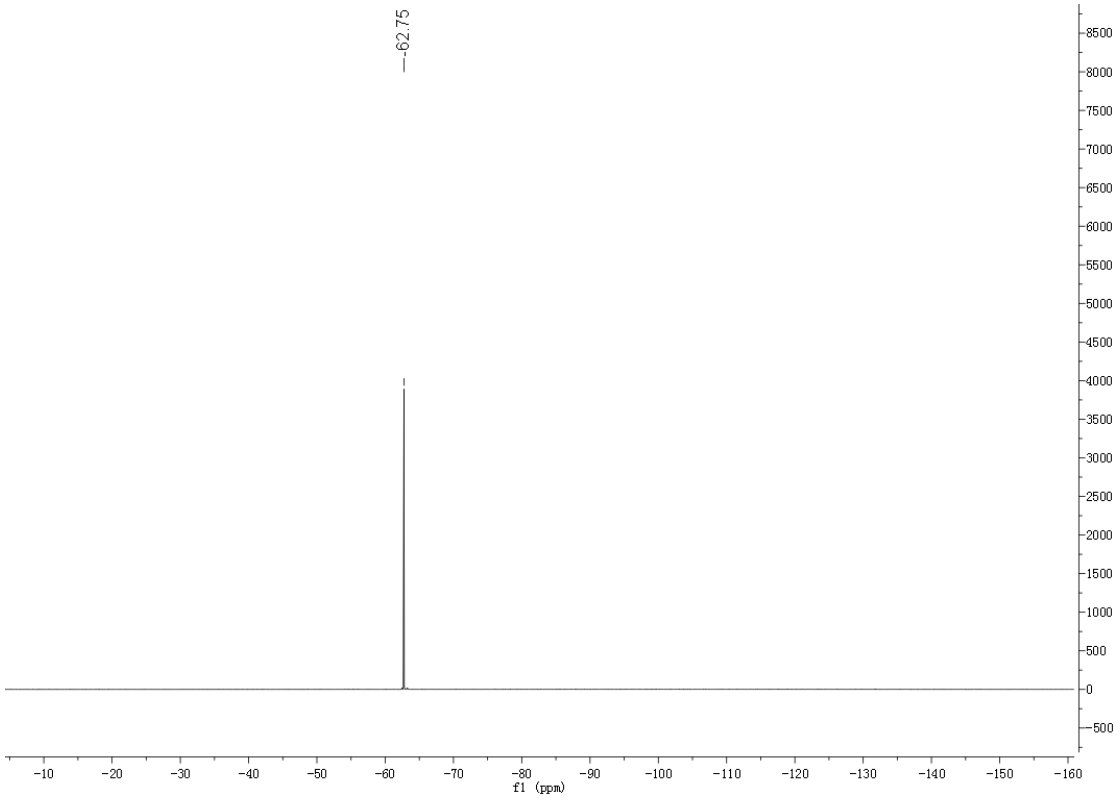

7.48
7.37
6.64
4.01
4.00
3.81
2.96
1.15
1.14
1.11
1.10
1.08
1.06
1.02
1.00
0.98
0.97
1.98
0.94
2.01
2.01
6.00
21.10
f1 (ppm)

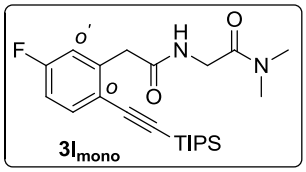

169.35
167.59
138.78
132.76
130.53
122.92
122.82
103.80
97.17
41.74
41.56
35.94
35.58
18.72
11.27
f1 (ppm)


F3C
H
N
O
O
N
3k
TIPS

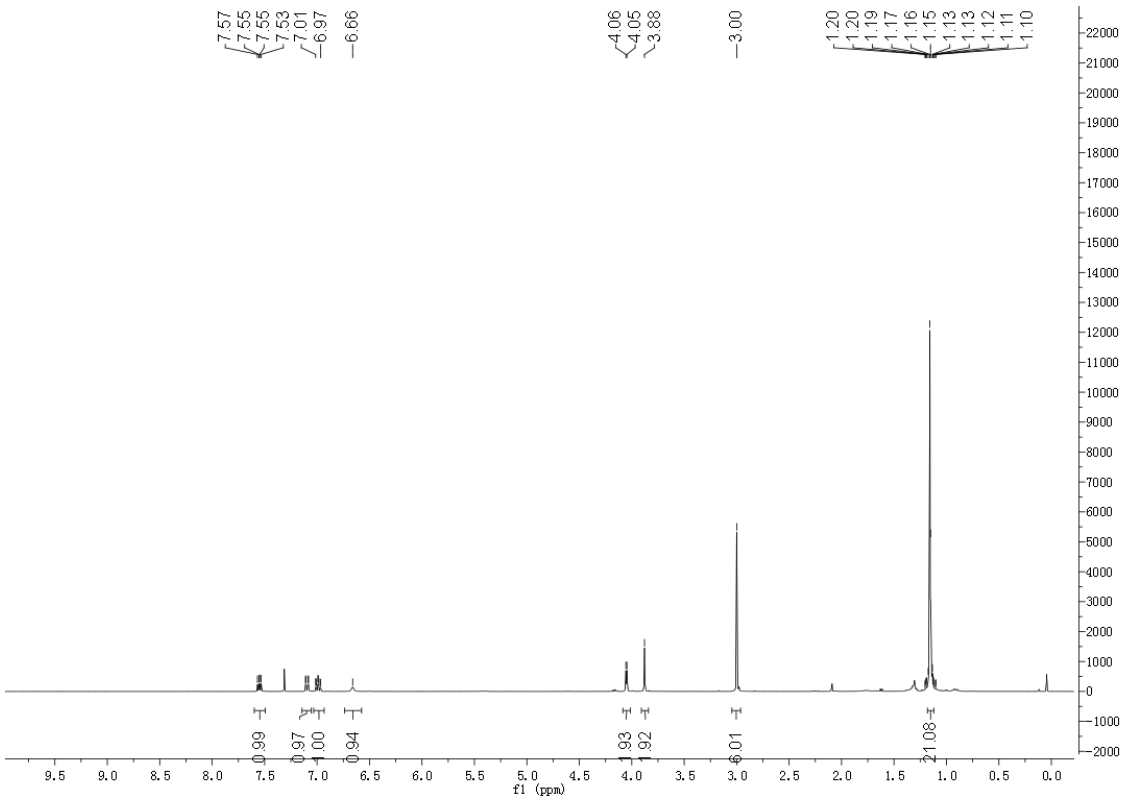

7.62
7.60
7.57
7.48
7.46
6.65
4.01
4.00
3.88
2.94
1.11
1.10
1.09
1.08
1.06
1.03
1.01
0.99
0.97
2.00
1.00
0.97
2.03
2.00
6.00
21.05
f1 (ppm)

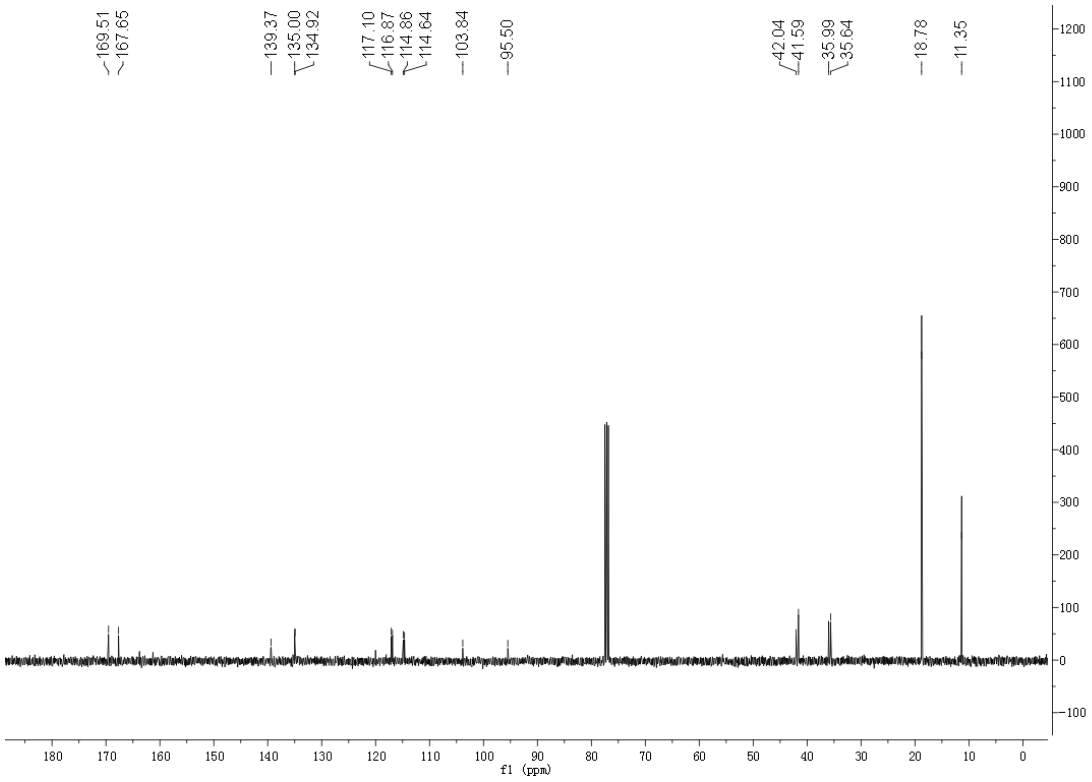

169.14
167.54
133.32
130.52
129.87
126.61
126.53
124.02
123.95
122.40
103.46
98.77
41.65
41.50
35.83
35.47
18.62
11.20
f1 (ppm)

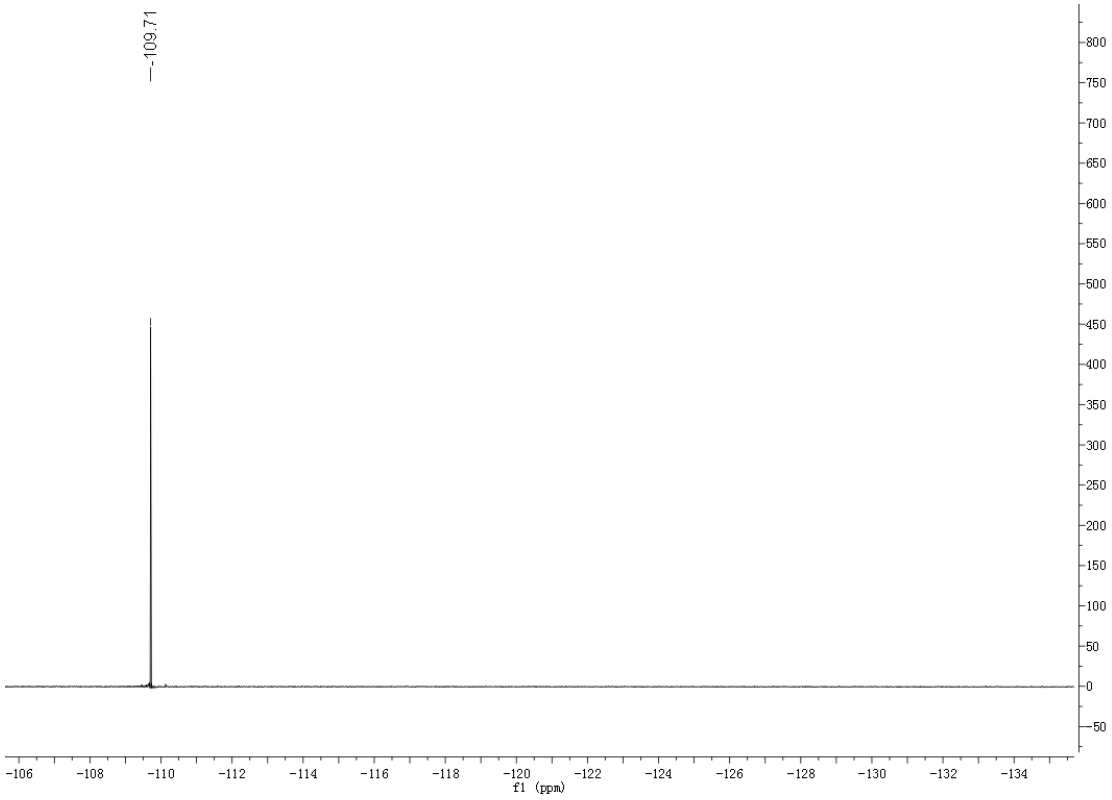

-62.75
8500
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)


F
o'
o
H
N
O
O
N
TIPS

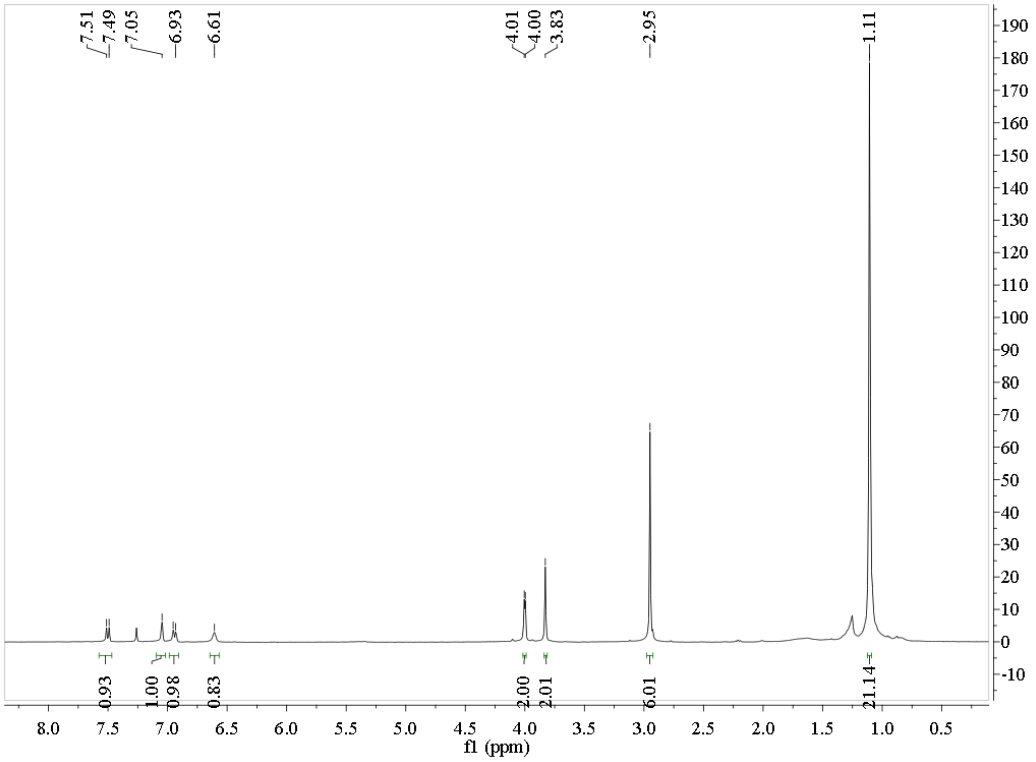
3l$_{mono}$

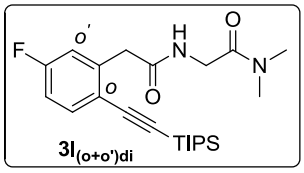

7.57
7.55
7.55
7.53
7.01
6.97
6.66
4.06
4.05
3.88
3.00
1.20
1.20
1.19
1.17
1.16
1.15
1.13
1.13
1.12
1.11
1.10
0.99
0.97
1.00
0.94
1.93
1.92
6.01
21.08
f1 (ppm)


169.51
167.65
139.37
135.00
134.92
117.10
116.87
114.86
114.64
103.84
95.50
42.04
41.59
35.99
35.64
18.78
11.35
f1 (ppm)

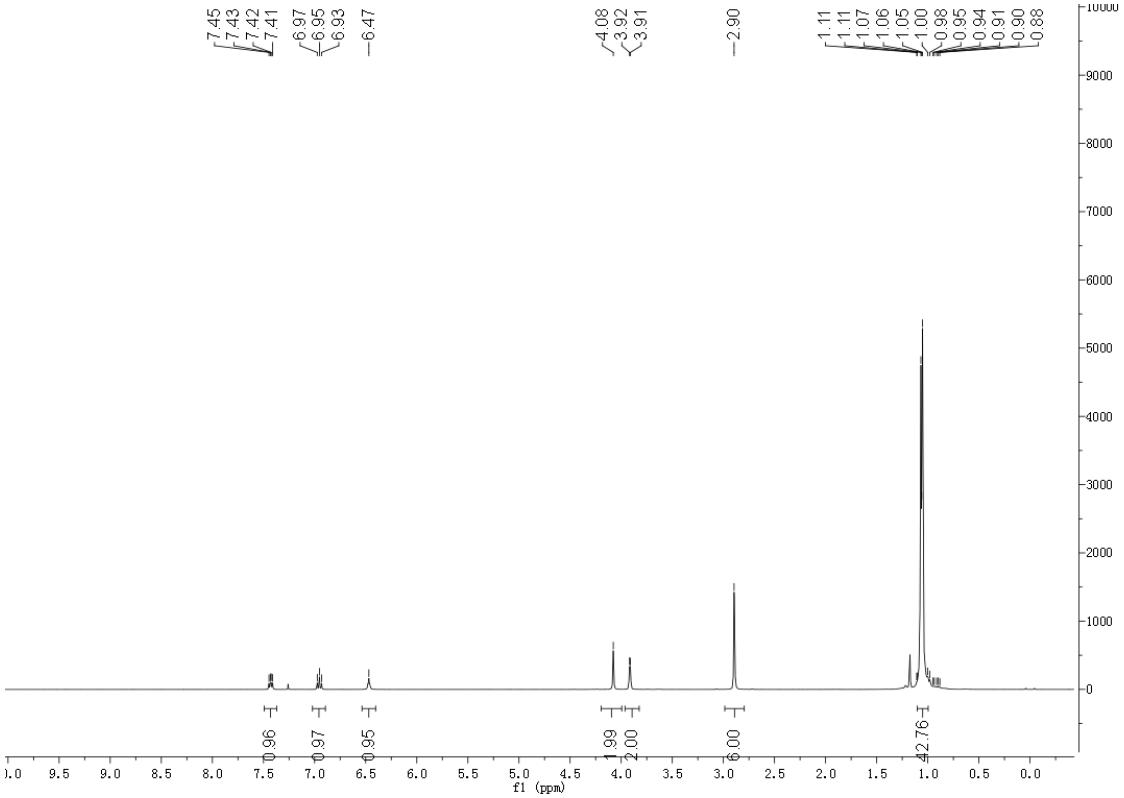


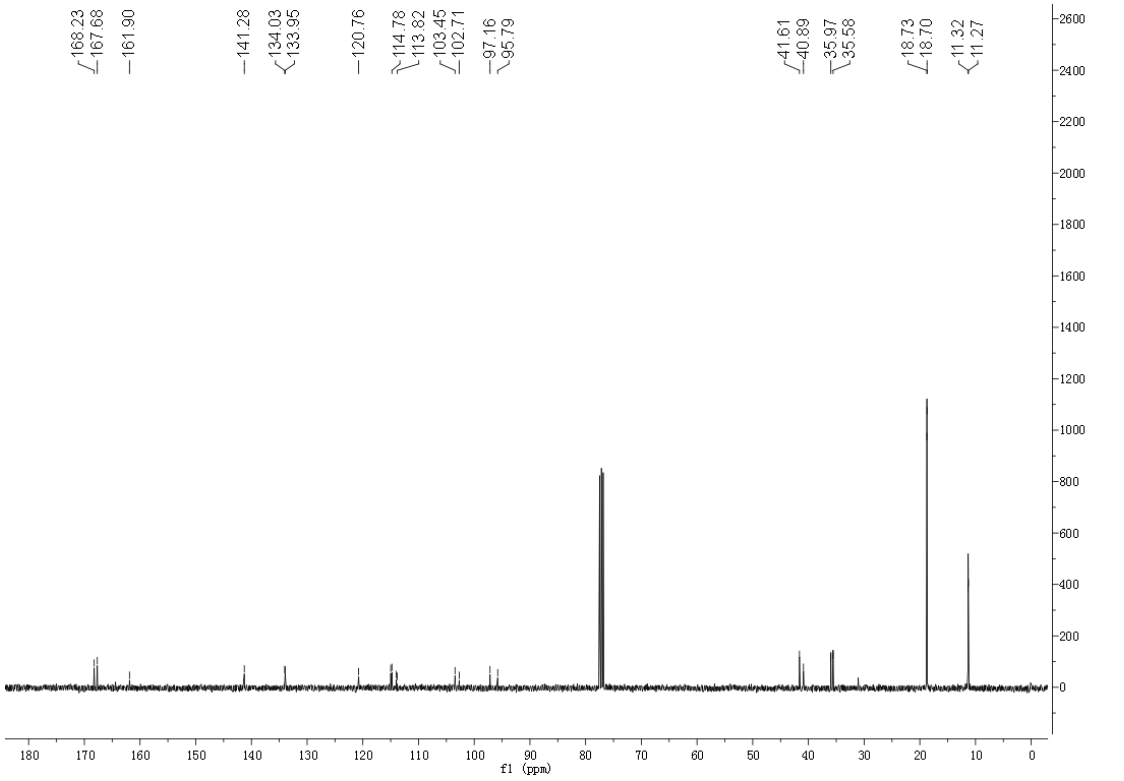
^{1}H NMR spectra of decoupling ^{19}F for 3lmono

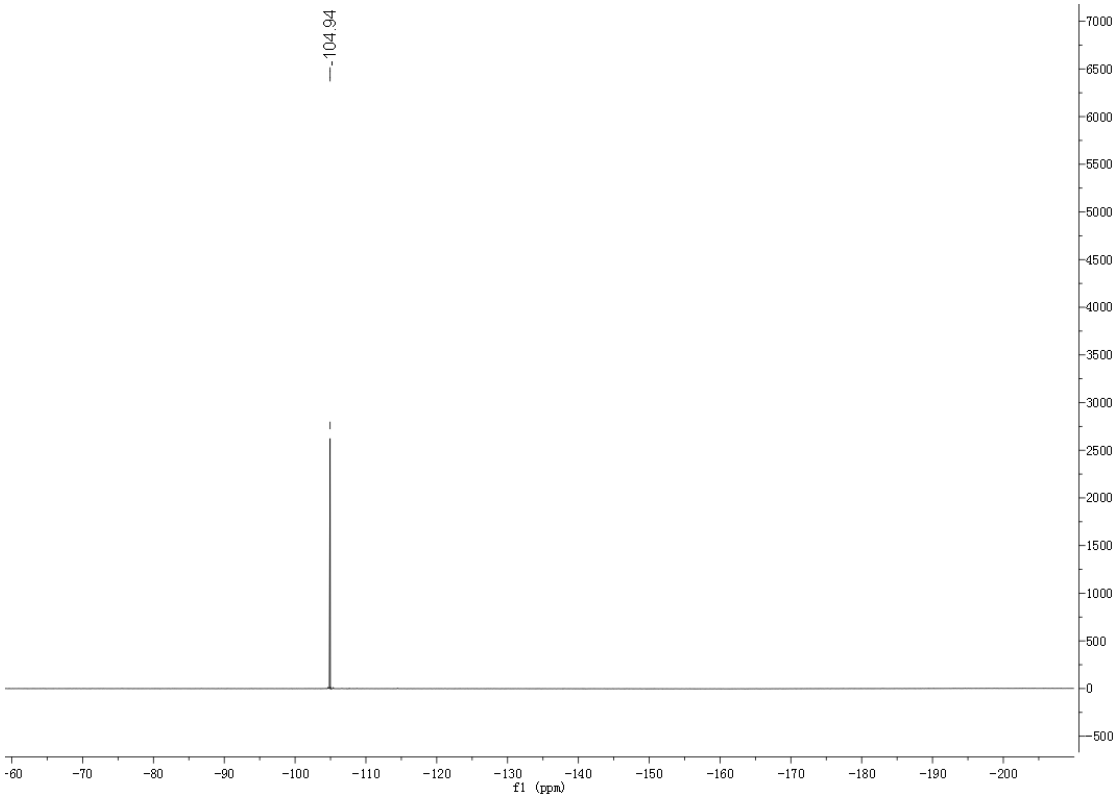


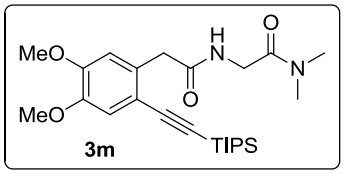

F
o'
o
H
N
O
O
N
TIPS

3l$_{(o+o')di}$

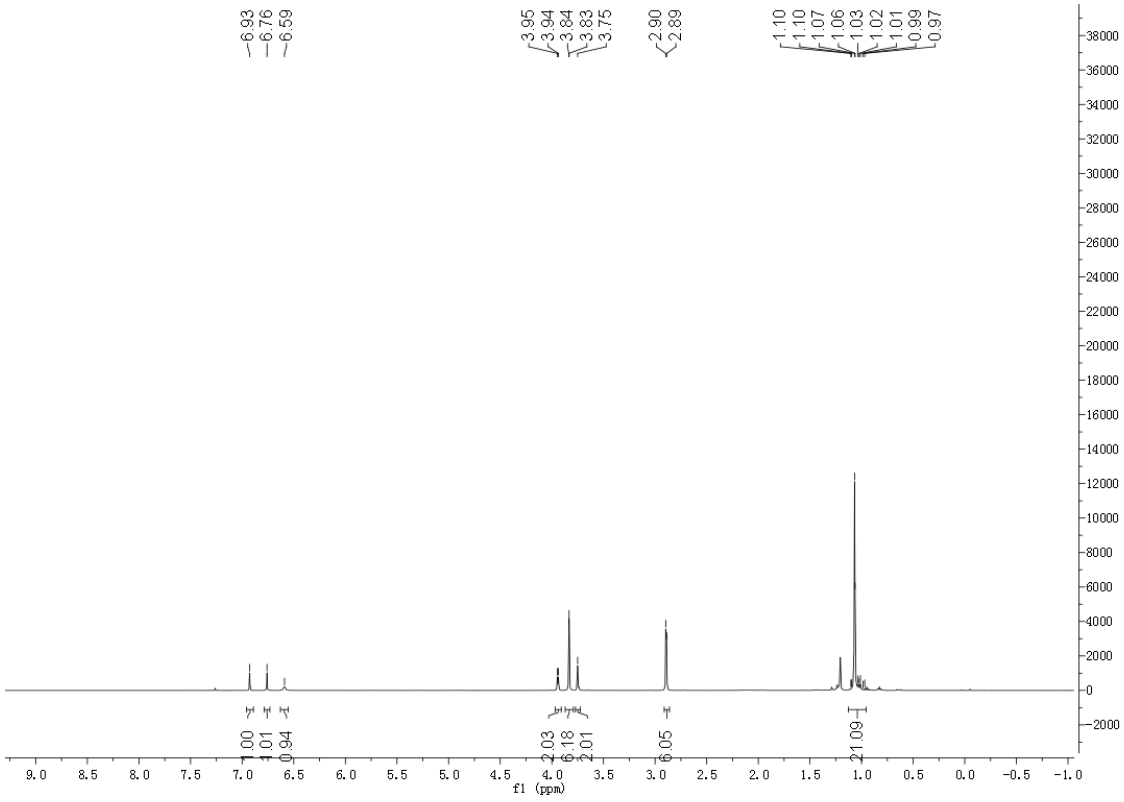

7.45
7.43
7.42
7.41
6.97
6.95
6.93
6.47
4.08
3.92
3.91
2.90
1.11
1.11
1.07
1.06
1.05
1.00
0.98
0.95
0.94
0.91
0.90
0.88
0.96
0.97
0.95
1.99
2.00
6.00
2.76
f1 (ppm)

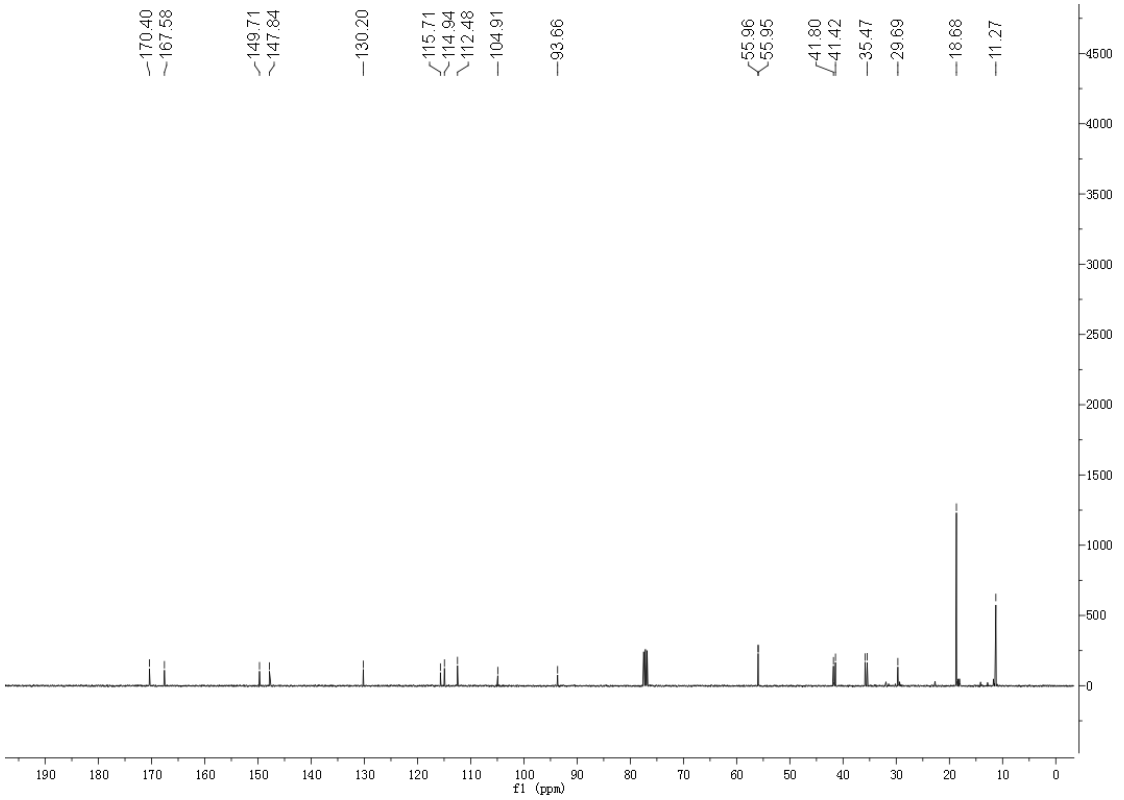

168.23
167.68
161.90
141.28
134.03
133.95
120.76
114.78
113.82
103.45
102.71
97.16
95.79
41.61
40.89
35.97
35.58
18.73
18.70
11.32
11.27
f1 (ppm)

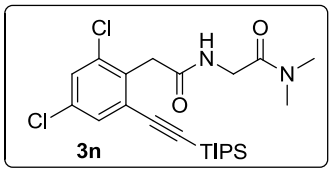

-104.94
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

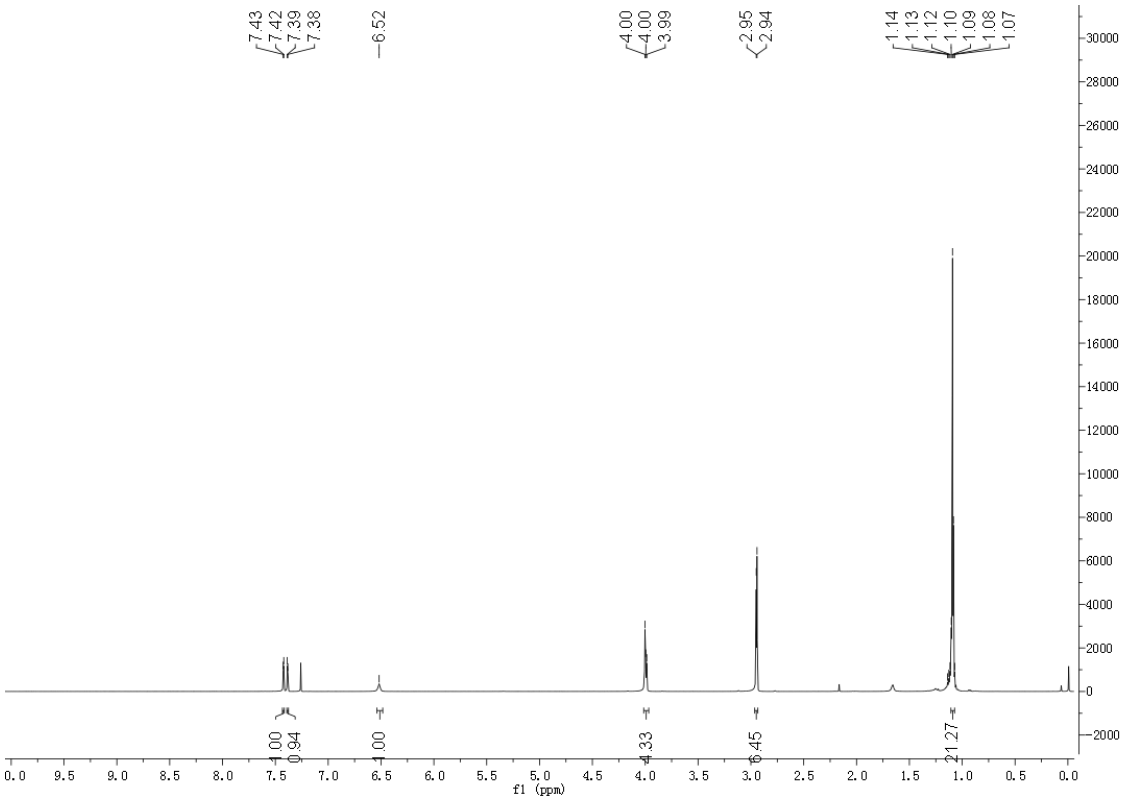

MeO
MeO
H
N
O
O
N
TIPS

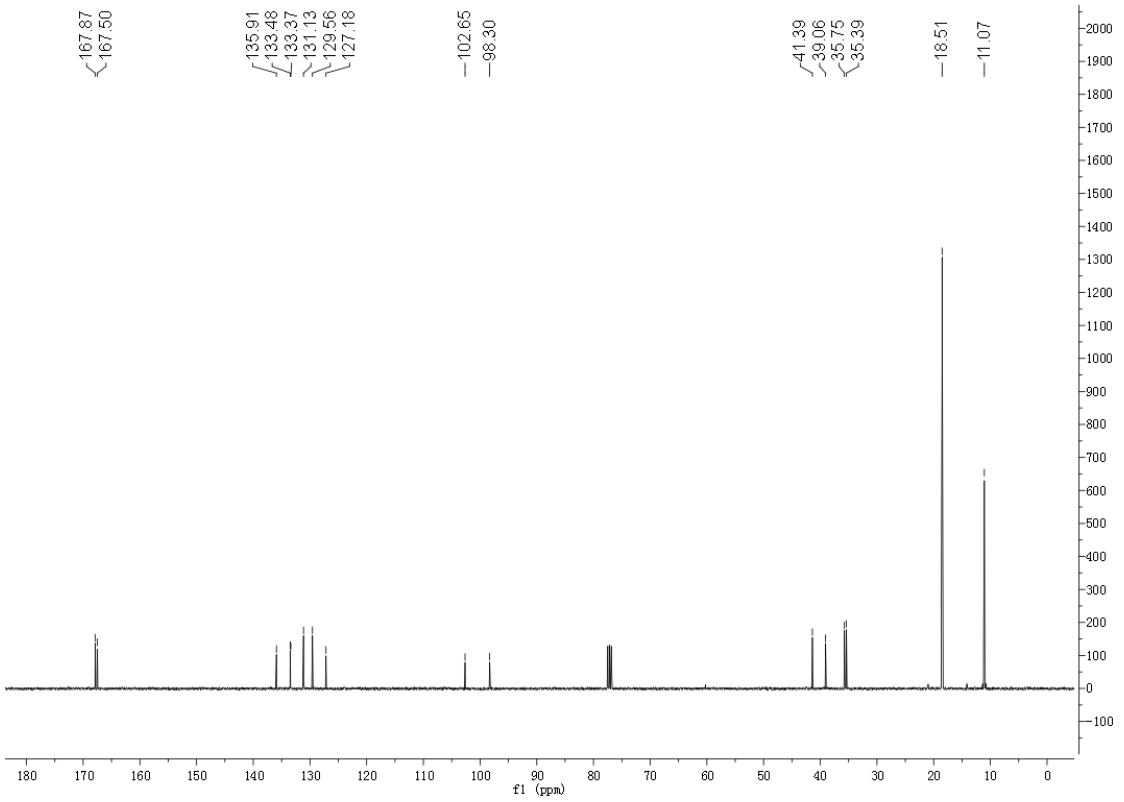
3m

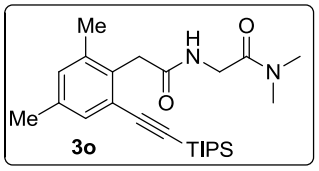

6.93
6.76
6.59
3.95
3.94
3.84
3.83
3.75
2.90
2.89
1.10
1.10
1.07
1.06
1.03
1.02
1.01
0.99
0.97
1.00
1.01
0.94
2.03
6.18
2.01
6.05
21.09
f1 (ppm)


170.40
167.58
149.71
147.84
130.20
115.71
114.94
112.48
104.91
93.66
55.96
55.95
41.80
41.42
35.47
29.69
18.68
11.27
f1 (ppm)

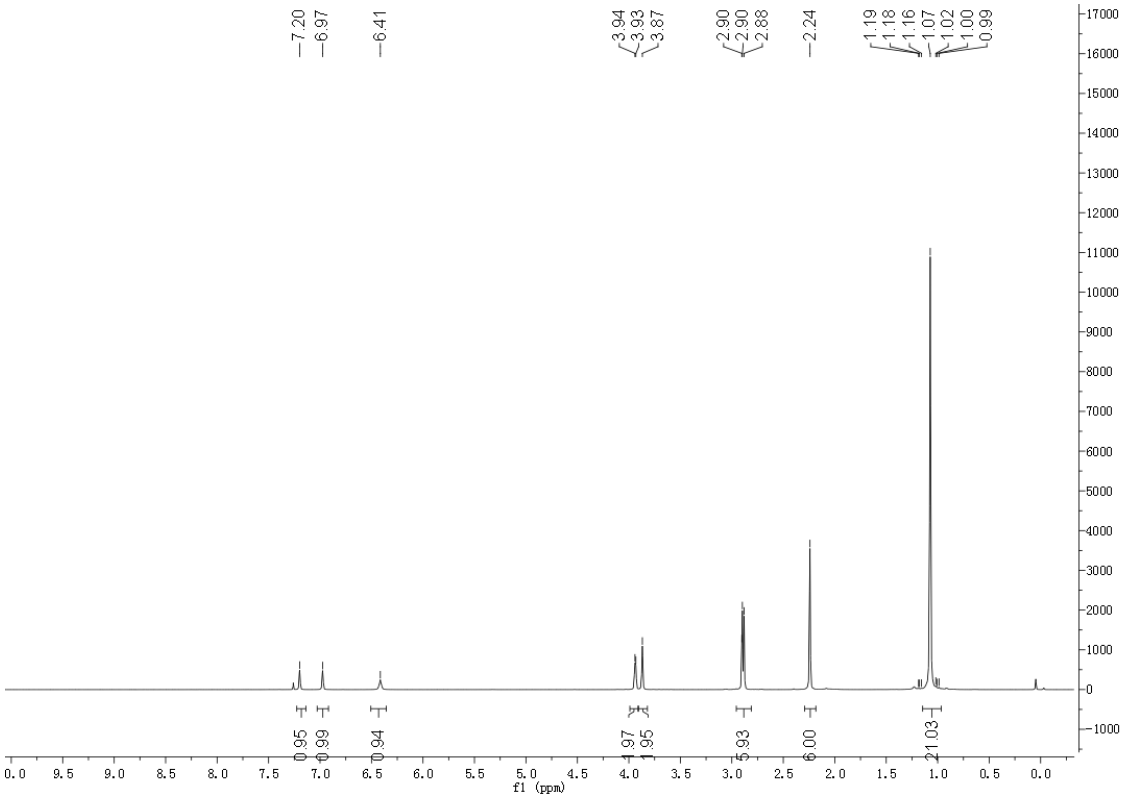

Cl
H
N
O
O
N
Cl
3n
TIPS

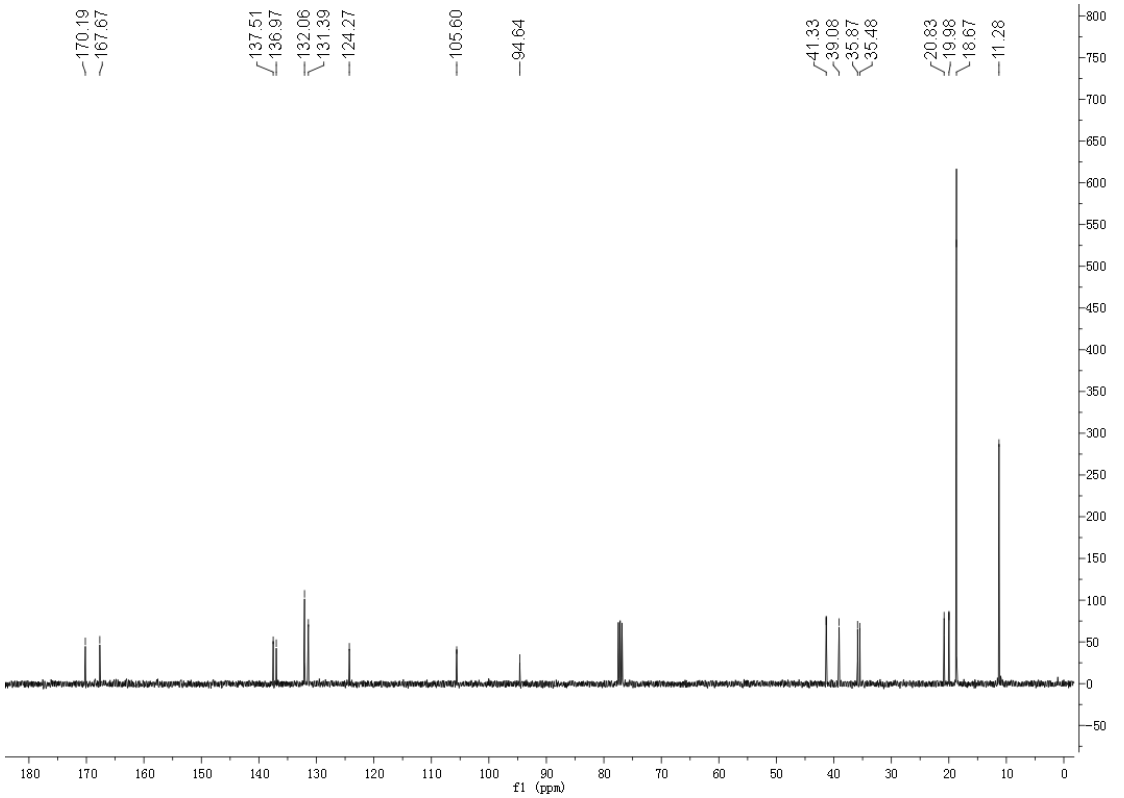

7.43
7.42
7.39
7.38
6.52
4.00
4.00
3.99
2.95
2.94
1.14
1.13
1.12
1.10
1.09
1.08
1.07
1.00
0.94
1.00
4.33
6.45
21.27
f1 (ppm)

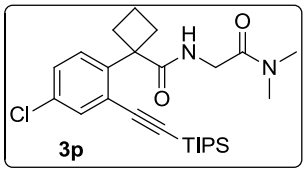

167.87
167.50
135.91
133.48
133.37
131.13
129.56
127.18
102.65
98.30
41.39
39.06
35.75
35.39
18.51
11.07
f1 (ppm)


Me
H
N
O
N
O
Me
TIPS

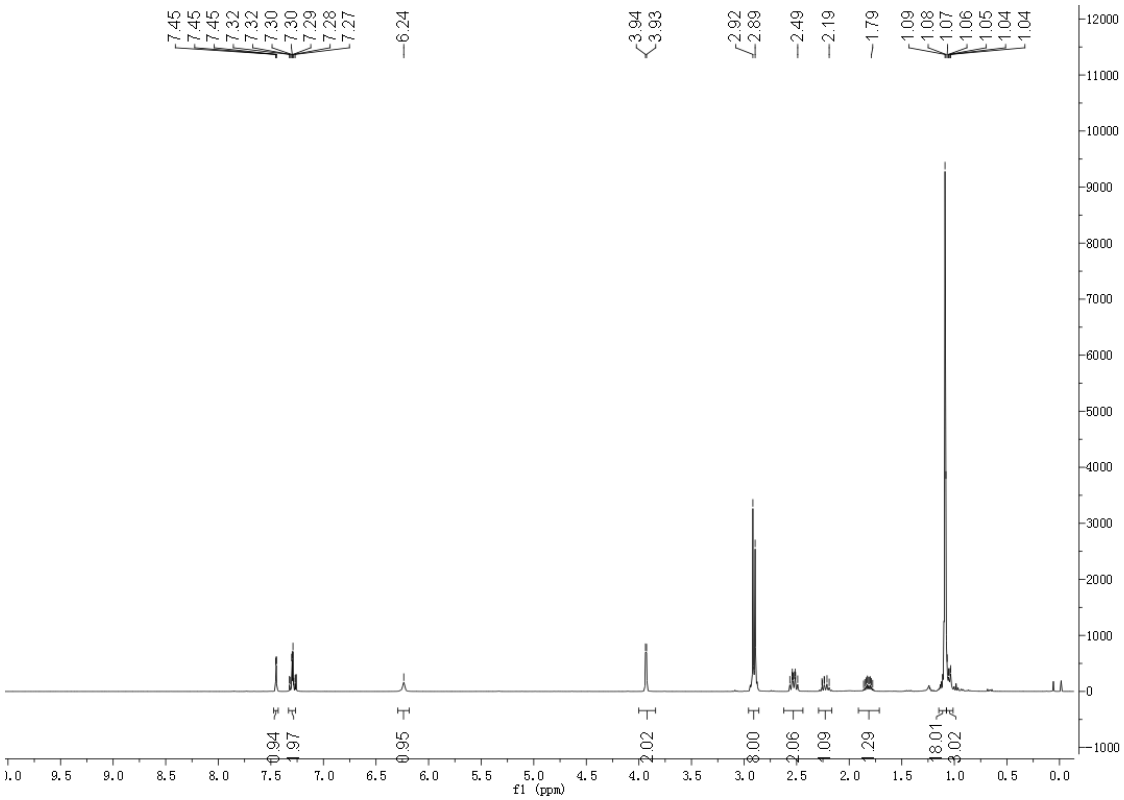
3o

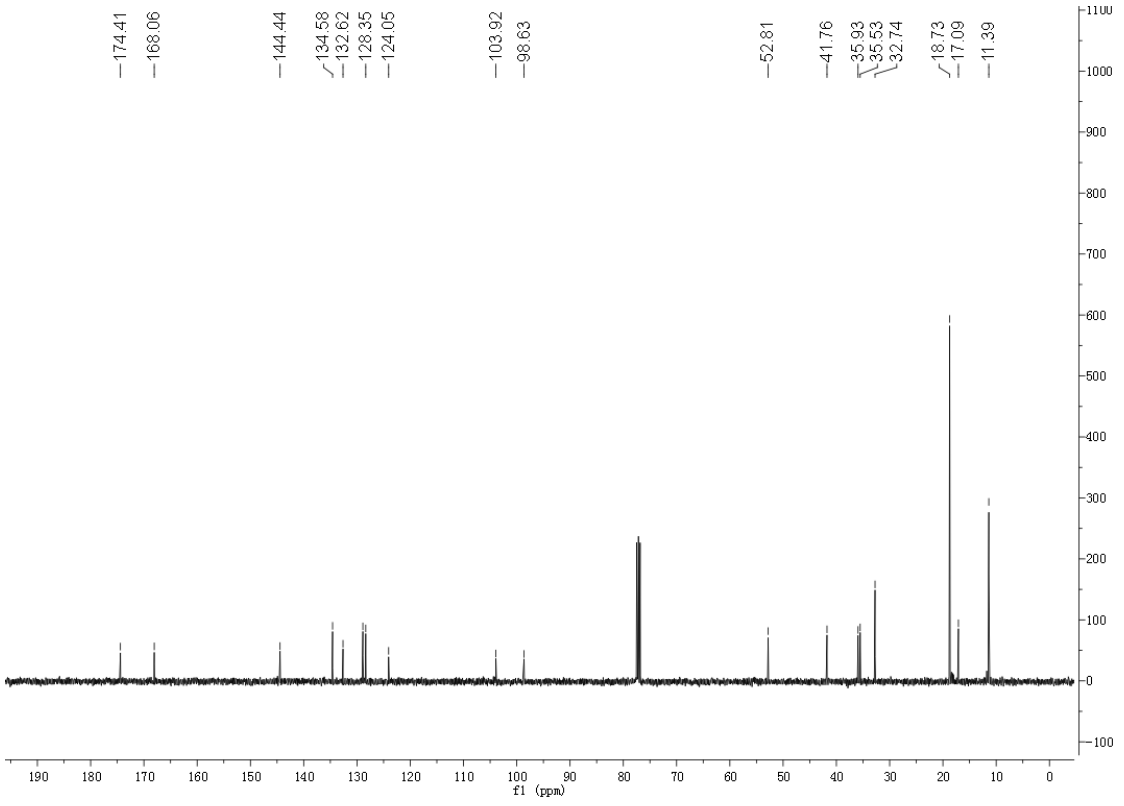

7.20
6.97
6.41
3.94
3.93
3.87
2.90
2.90
2.88
2.24
1.19
1.18
1.16
1.07
1.02
1.00
0.99
0.95
0.99
0.94
1.97
1.95
5.93
6.00
21.03
f1 (ppm)

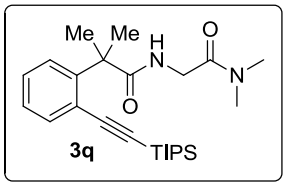

170.19
167.67
137.51
136.97
132.06
131.39
124.27
105.60
94.64
41.33
39.08
35.87
35.48
20.83
19.98
18.67
11.28
f1 (ppm)

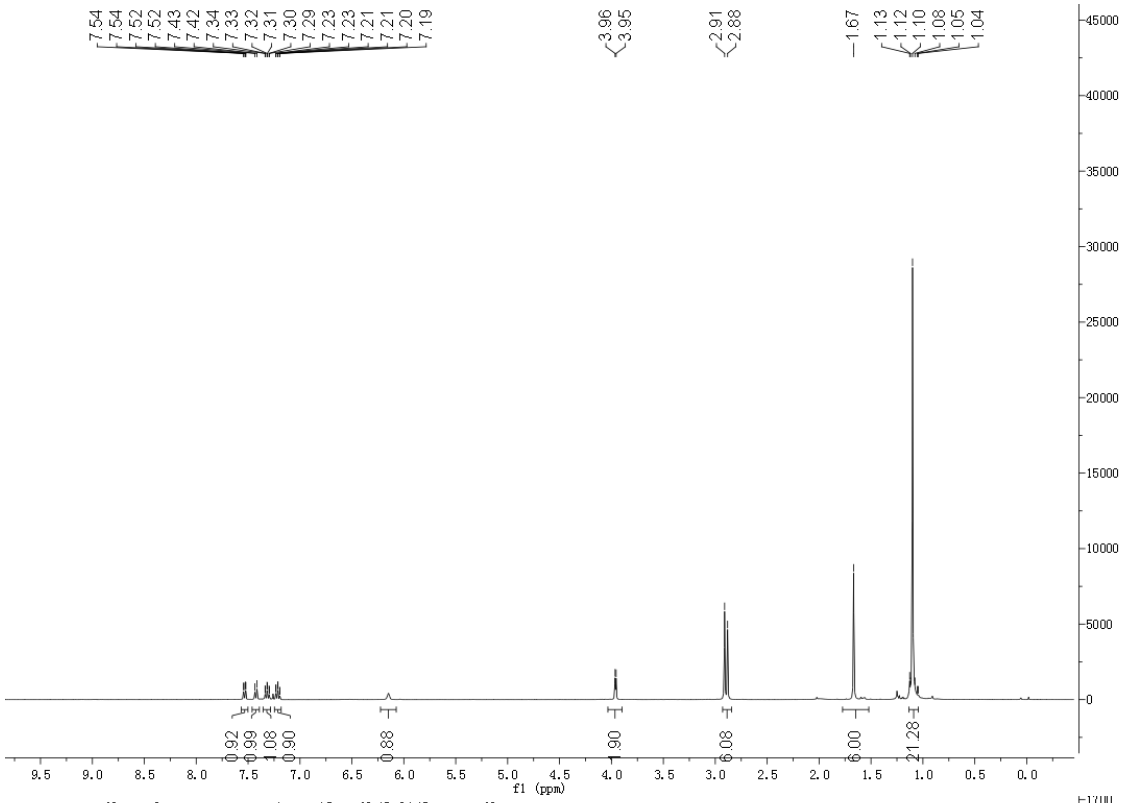

H
N
O
O
N
Cl
TIPS

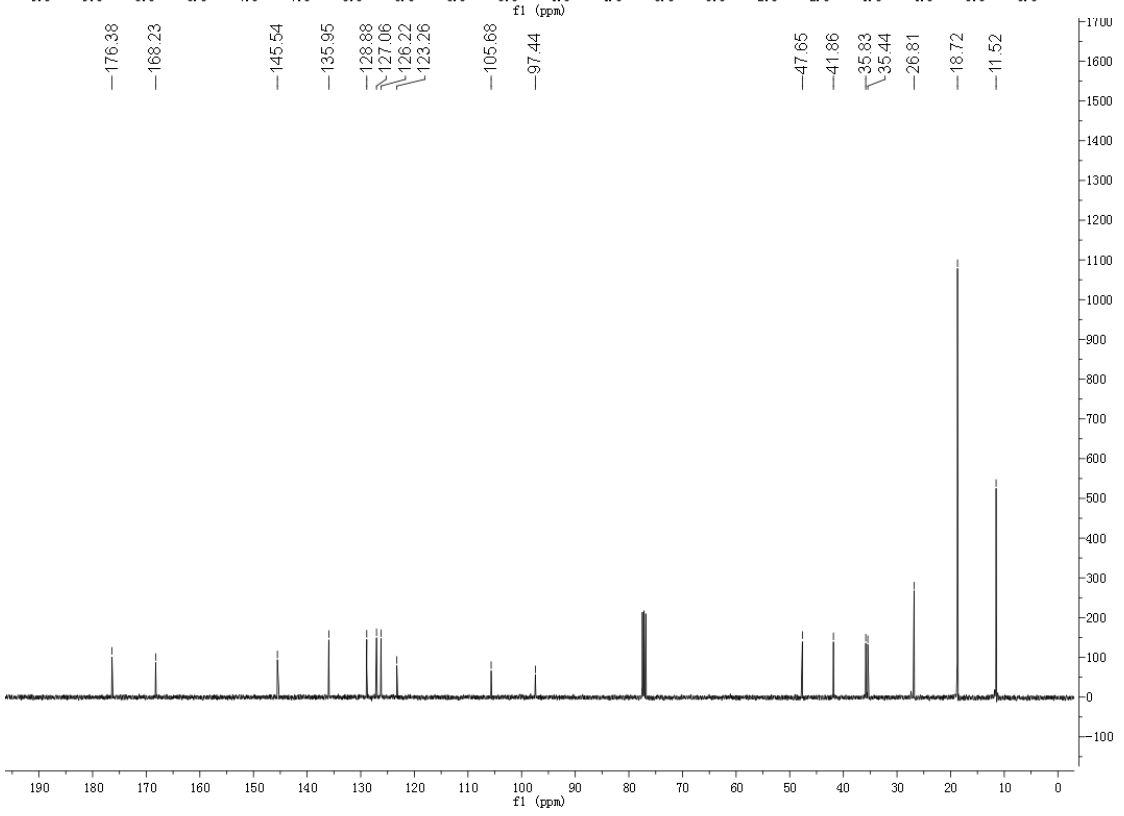
3p

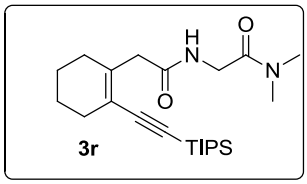

7.45
7.45
7.45
7.32
7.32
7.30
7.30
7.29
7.28
7.27
6.24
3.94
3.93
2.92
2.89
2.49
2.19
1.79
1.09
1.08
1.07
1.06
1.05
1.04
1.04
0.94
1.97
0.95
2.02
3.00
2.06
1.09
1.29
18.01
3.02
f1 (ppm)

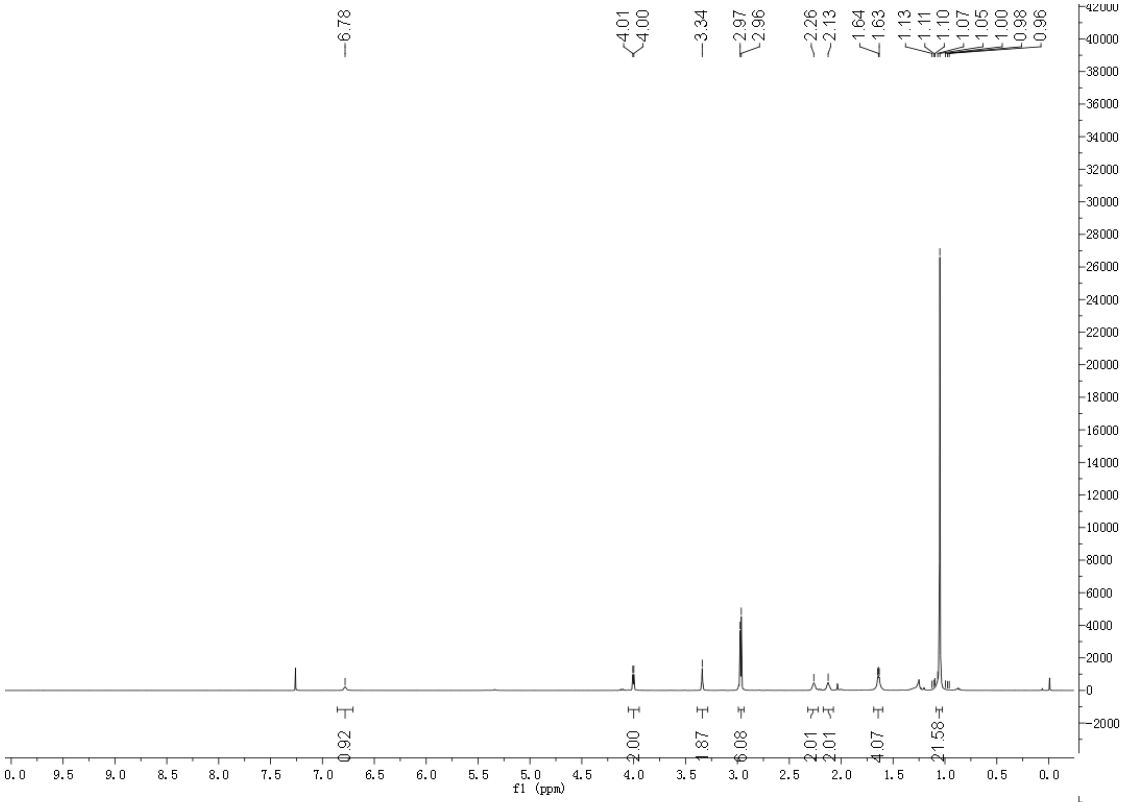

174.41
168.06
144.44
134.58
132.62
128.35
124.05
103.92
98.63
52.81
41.76
35.93
35.53
32.74
18.73
17.09
11.39
f1 (ppm)

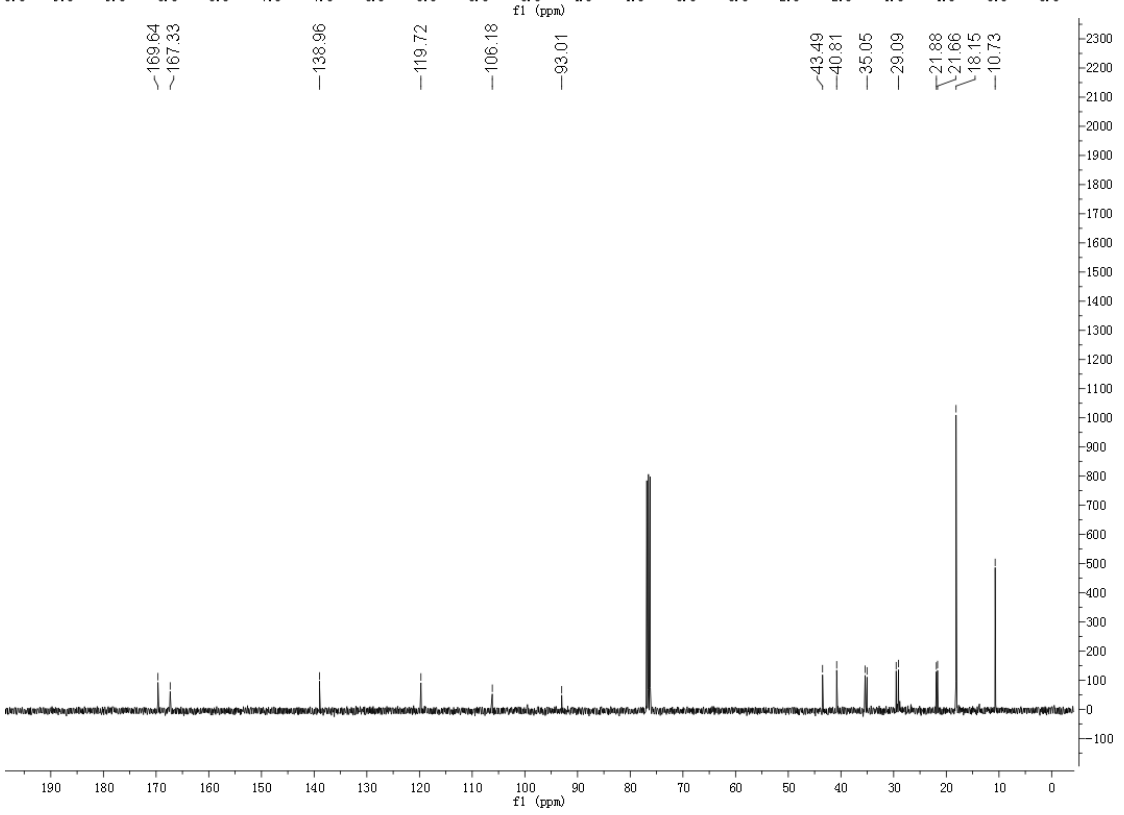

Me
Me
H
N
O
O
N
3q
TIPS

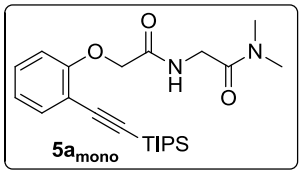

7.54
7.54
7.52
7.52
7.43
7.42
7.34
7.33
7.32
7.31
7.30
7.29
7.23
7.23
7.21
7.21
7.20
7.19
3.96
3.95
2.91
2.88
1.67
1.13
1.12
1.10
1.08
1.05
1.04
0.92
0.99
1.08
0.90
0.88
1.90
6.08
6.00
21.28
f1 (ppm)


176.38
168.23
145.54
135.95
128.88
127.06
126.22
123.26
105.68
97.44
47.65
41.86
35.83
35.44
26.81
18.72
11.52
f1 (ppm)

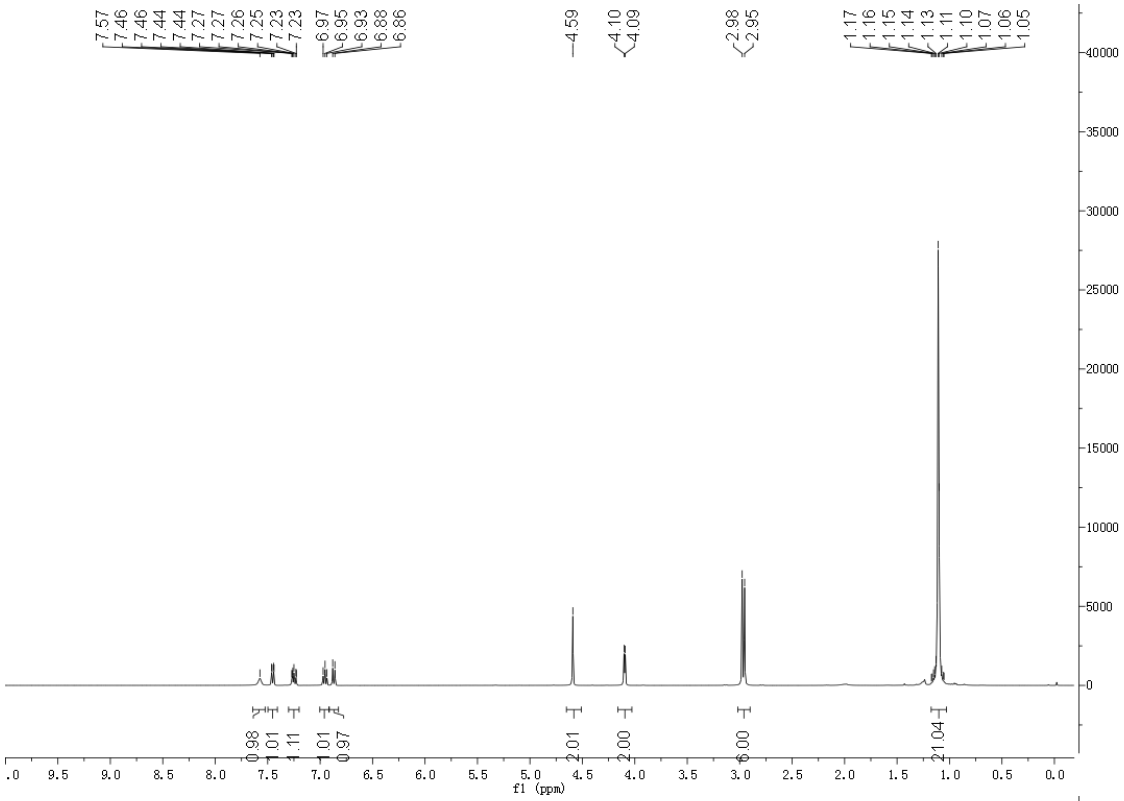

H
N
O
O
N
3r
TIPS

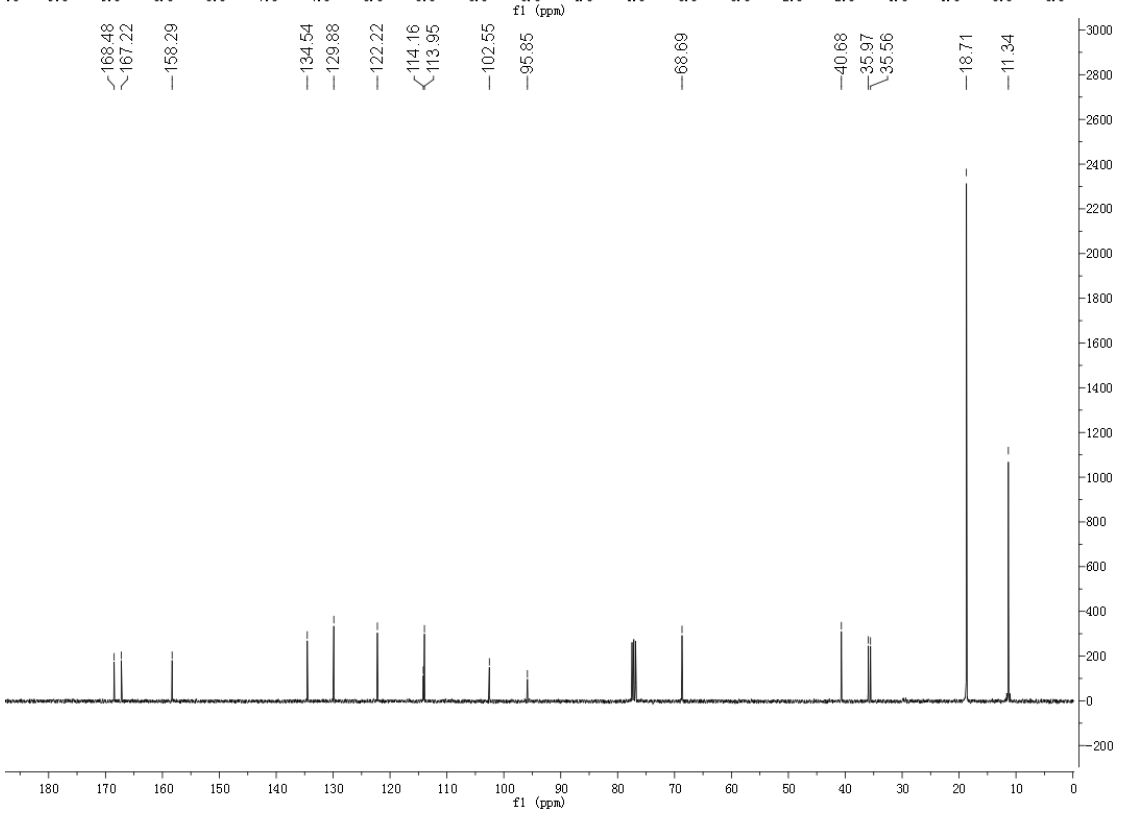

6.78
4.01
4.00
3.34
2.97
2.96
2.26
2.13
1.64
1.63
1.13
1.11
1.10
1.07
1.05
1.00
0.98
0.96
0.92
2.00
1.87
6.08
2.01
2.01
4.07
21.58
f1 (ppm)

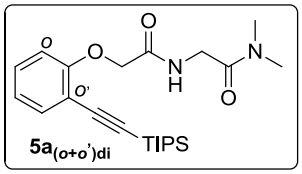

169.64
167.33
138.96
119.72
106.18
93.01
43.49
40.81
35.05
29.09
21.88
21.66
18.15
10.73
f1 (ppm)


O
O
N
H
N
O
TIPS

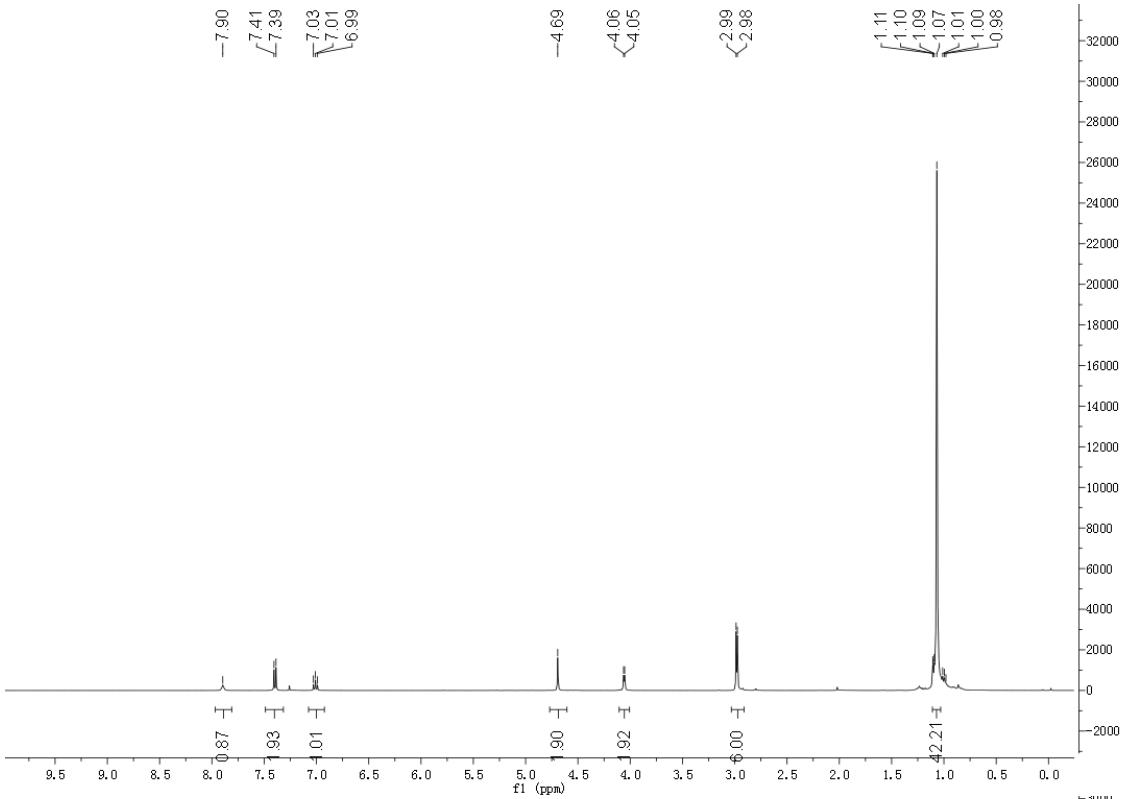
5a$_{mono}$

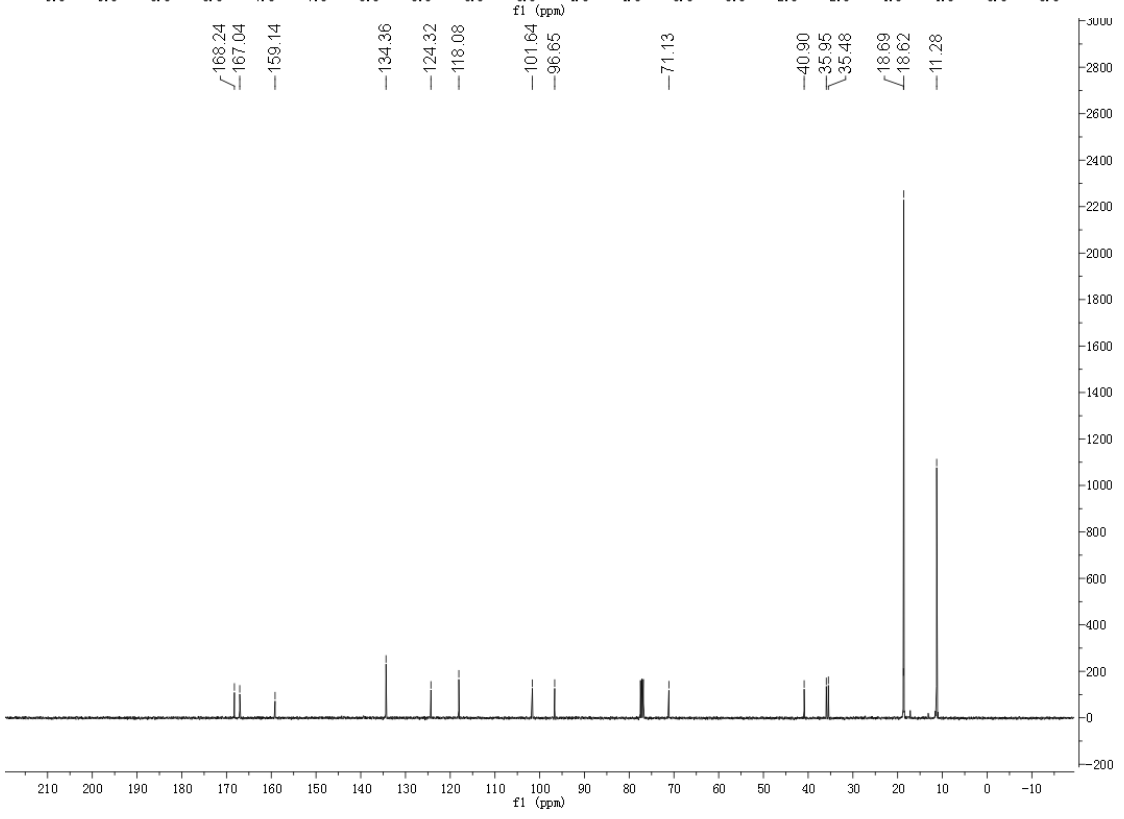

7.57
7.46
7.46
7.44
7.44
7.27
7.27
7.26
7.25
7.23
7.23
6.97
6.95
6.93
6.88
6.86
4.59
4.10
4.09
2.98
2.95
1.17
1.16
1.15
1.14
1.13
1.11
1.10
1.07
1.06
1.05
0.98
1.01
1.11
1.01
0.97
2.01
2.00
6.00
21.04
f1 (ppm)

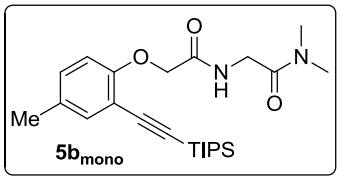

168.48
167.22
158.29
134.54
129.88
122.22
114.16
113.95
102.55
95.85
68.69
40.68
35.97
35.56
18.71
11.34
f1 (ppm)

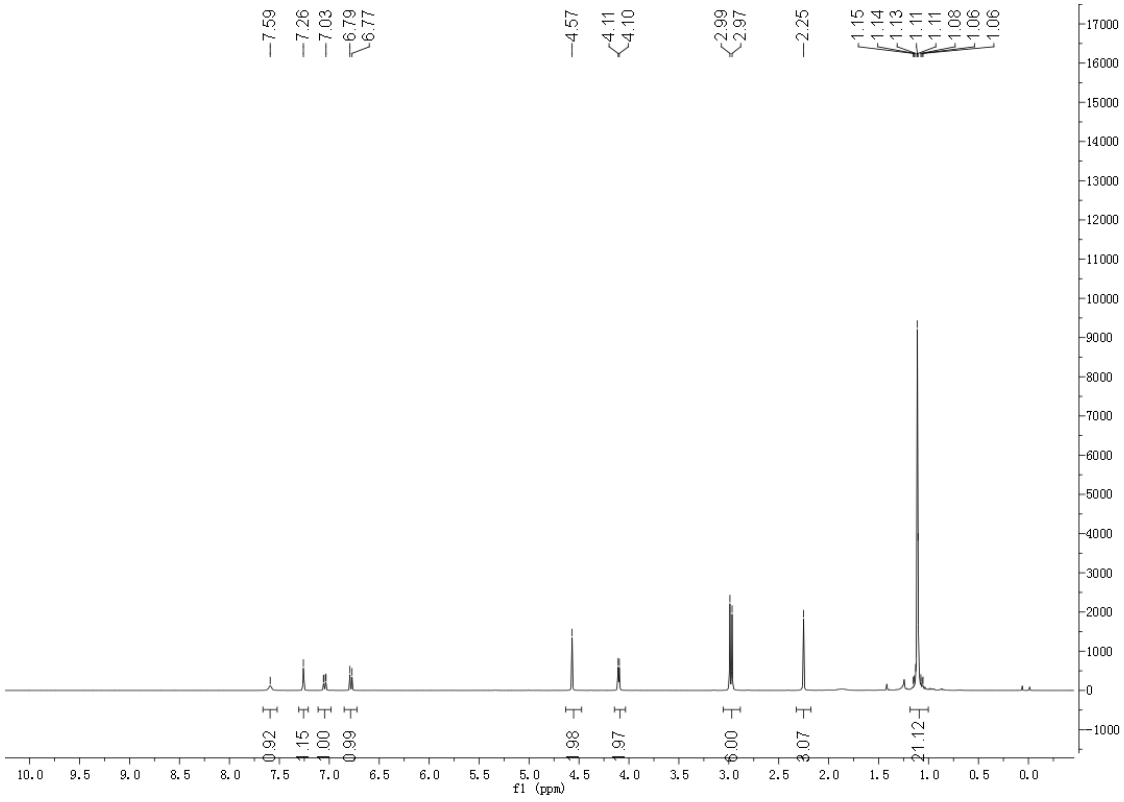

O
O
N
H
N
O
O
O'
TIPS

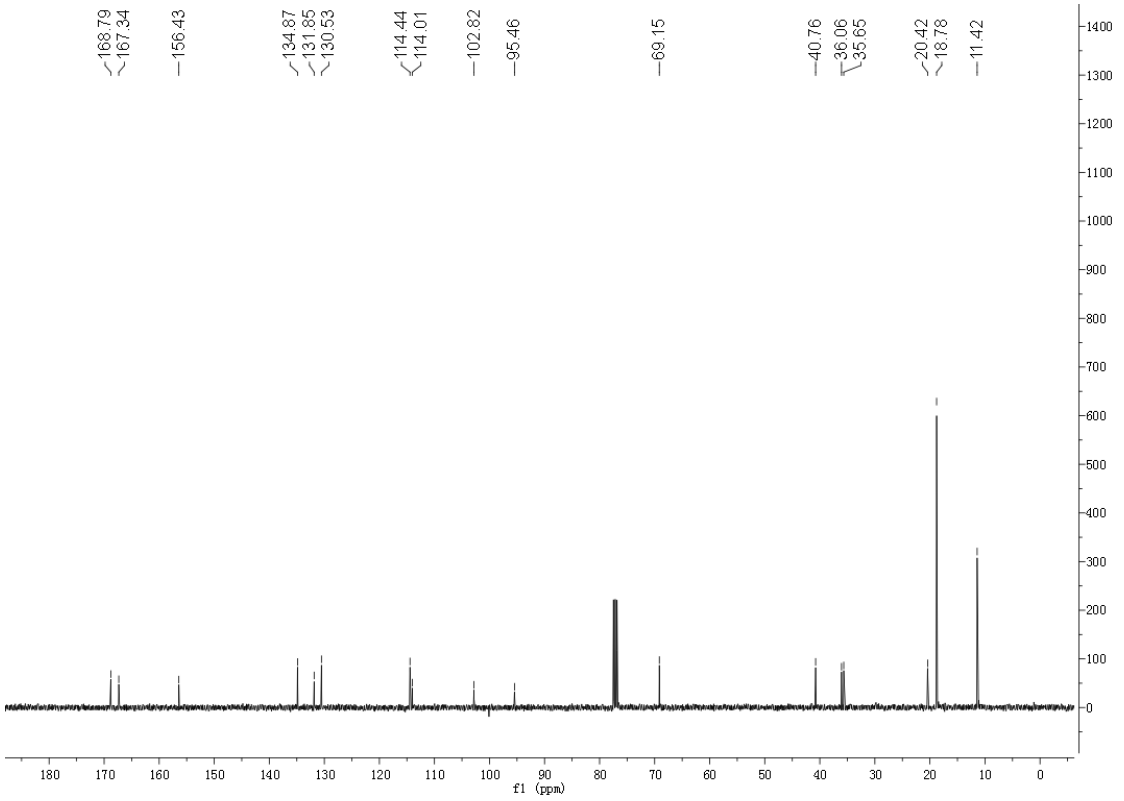
5a$_{(o+o')di}$

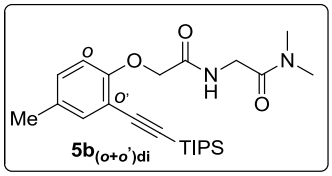

7.90
7.41
7.39
7.03
7.01
6.99
4.69
4.06
4.05
2.99
2.98
1.11
1.10
1.09
1.07
1.01
1.00
0.98
0.87
1.93
1.01
1.90
1.92
6.00
42.21
f1 (ppm)


168.24
167.04
159.14
134.36
124.32
118.08
101.64
96.65
71.13
40.90
35.95
35.48
18.69
18.62
11.28
f1 (ppm)

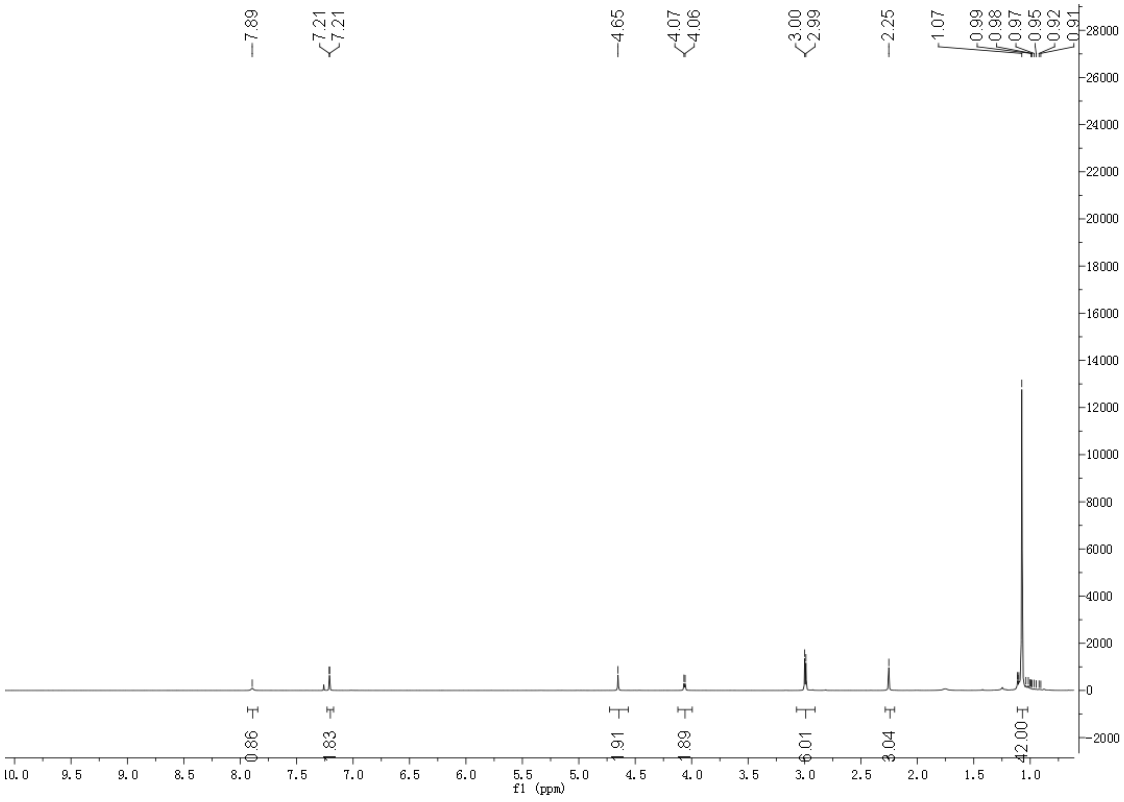

O
O
N
H
N
O
Me
TIPS
5b mono

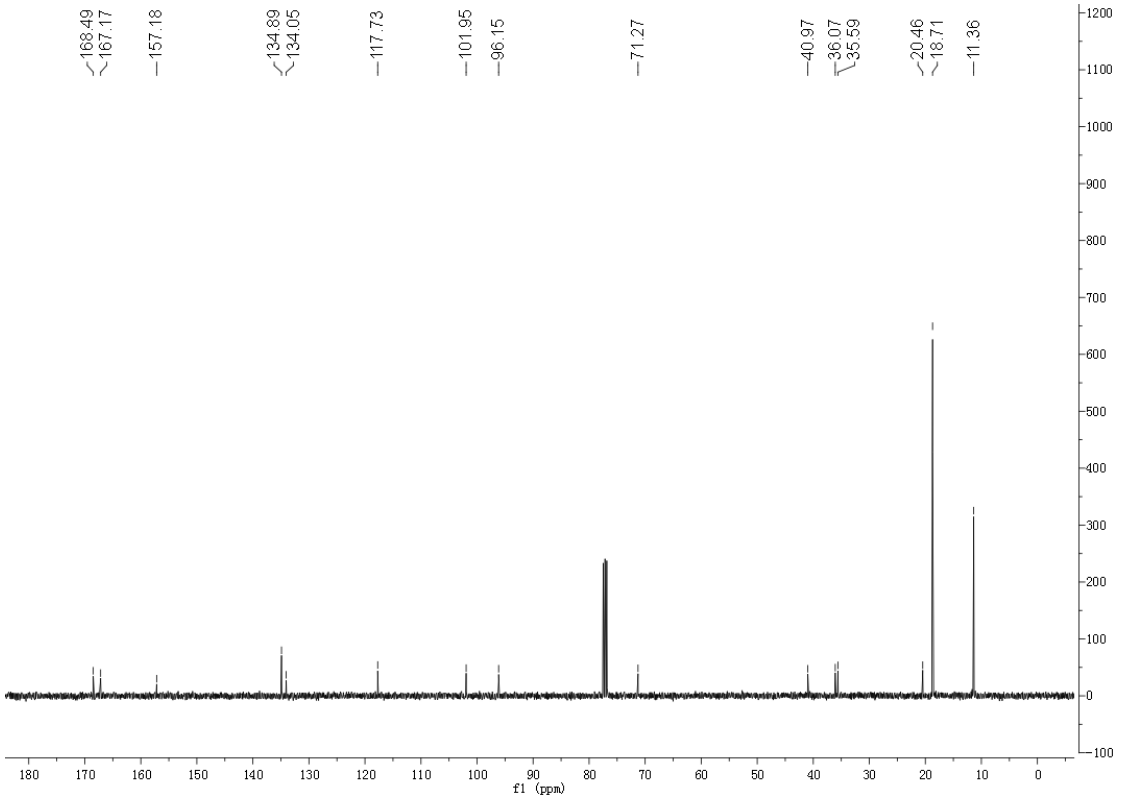

7.59
7.26
7.03
6.79
6.77
4.57
4.11
4.10
2.99
2.97
2.25
1.15
1.14
1.13
1.11
1.11
1.08
1.06
1.06
0.92
1.15
1.00
0.99
1.98
1.97
6.00
3.07
21.12
f1 (ppm)

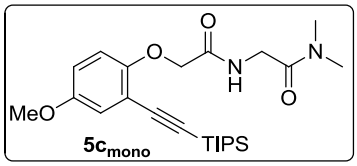

168.79
167.34
156.43
134.87
131.85
130.53
114.44
114.01
102.82
95.46
69.15
40.76
36.06
35.65
20.42
18.78
11.42
f1 (ppm)


O
O
N
H
N
O
o
o'
Me
TIPS

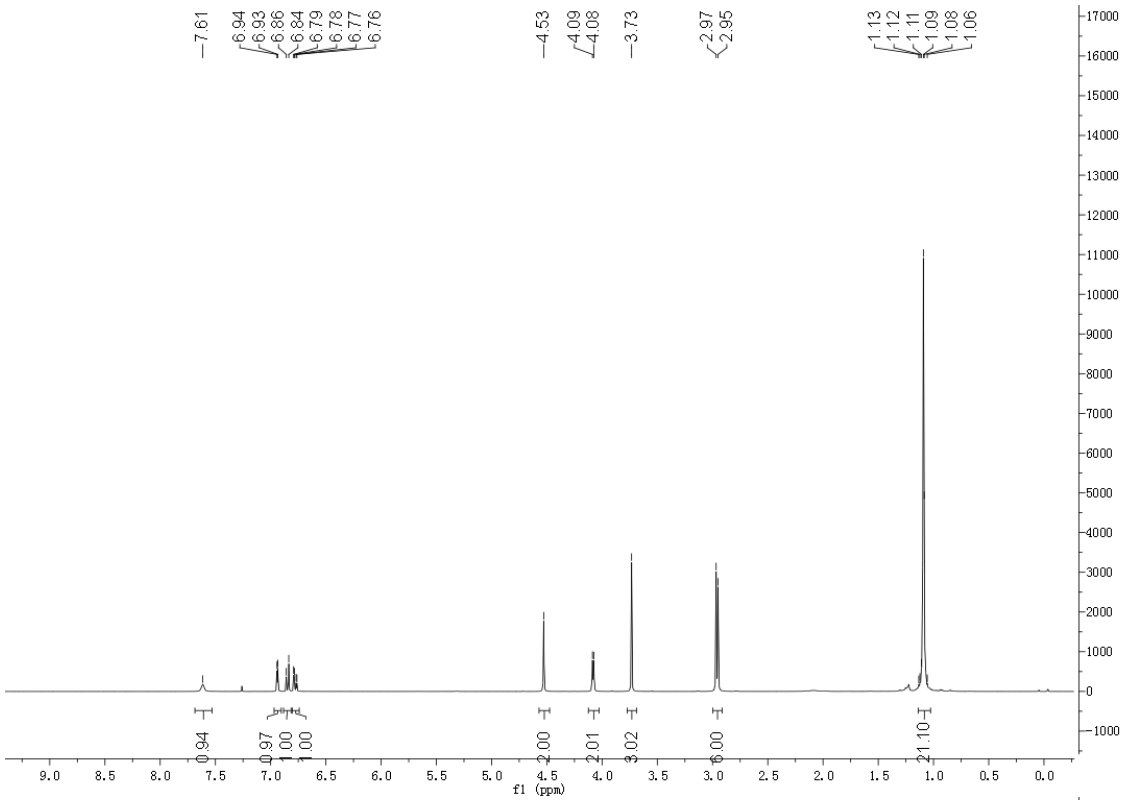
5b$_{(o+o')di}$

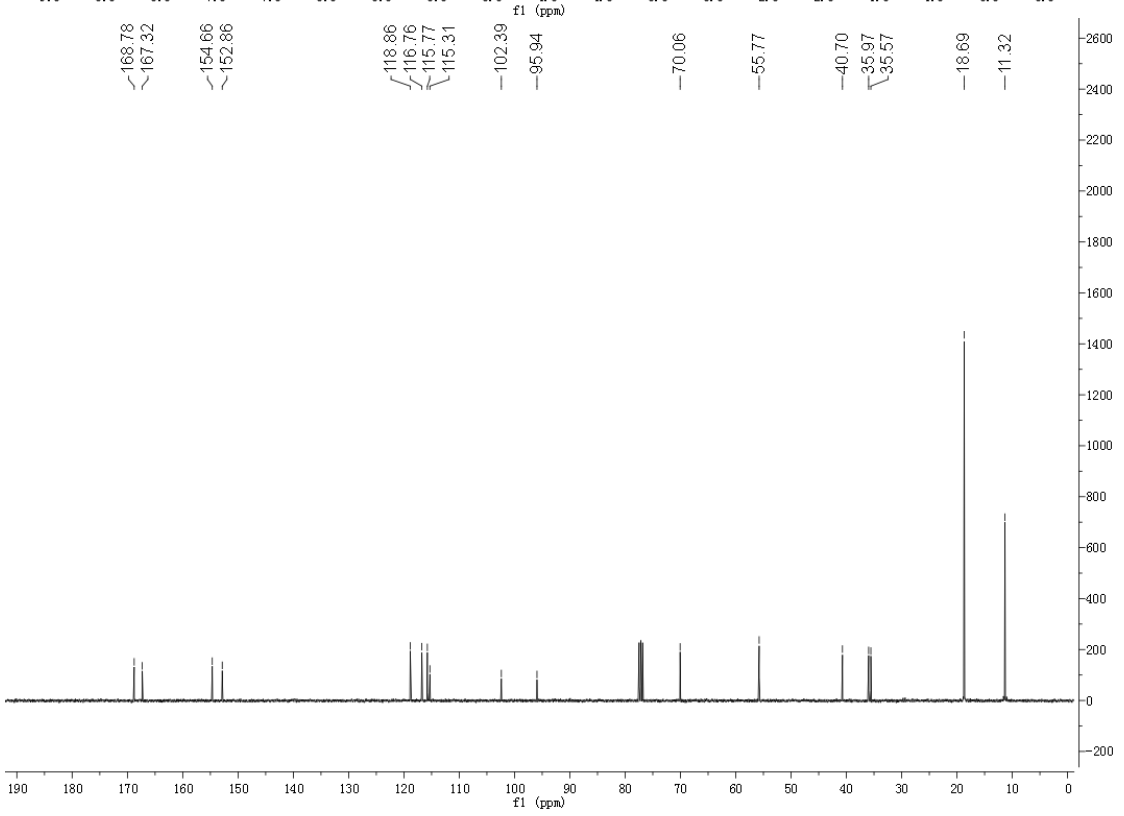

7.89
7.21
7.21
4.65
4.07
4.06
3.00
2.99
2.25
1.07
0.99
0.98
0.97
0.95
0.92
0.91
0.86
1.83
1.91
1.89
6.01
3.04
42.00
f1 (ppm)

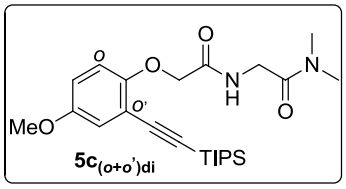

168.49
167.17
157.18
134.89
134.05
117.73
101.95
96.15
71.27
40.97
36.07
35.59
20.46
18.71
11.36
f1 (ppm)


O
O
N
H
N
O
MeO
TIPS

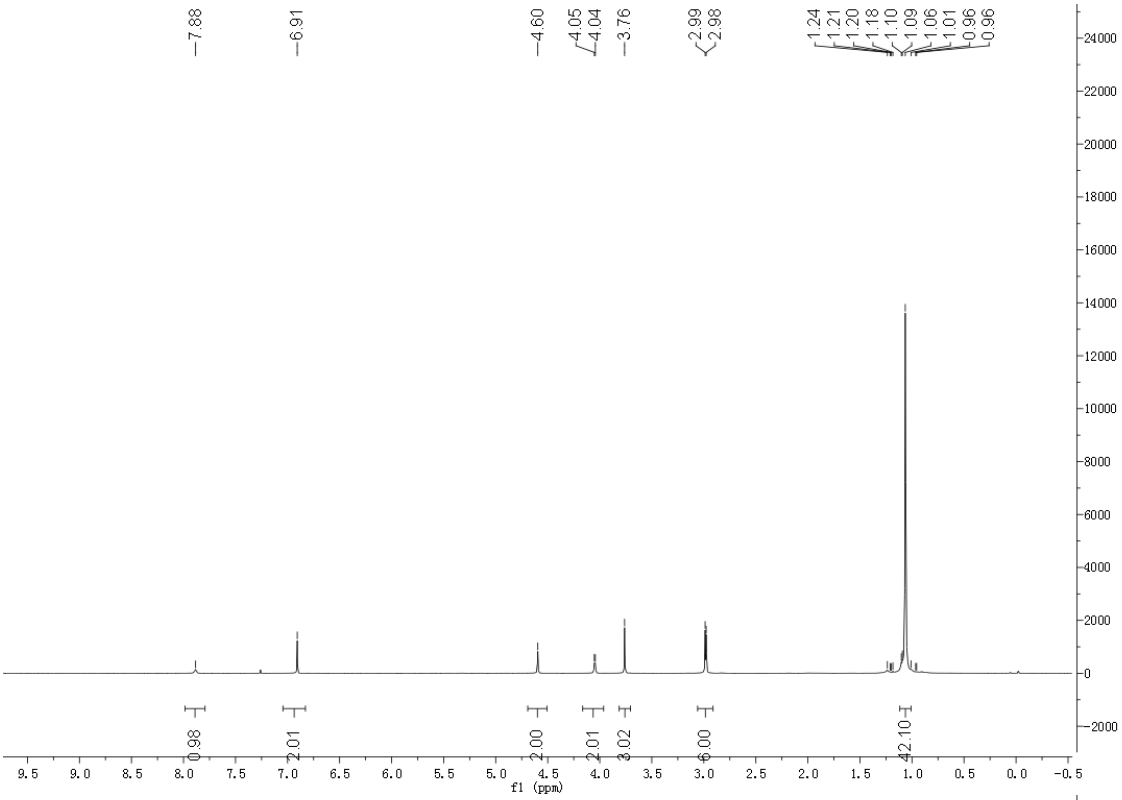
5c$_{mono}$

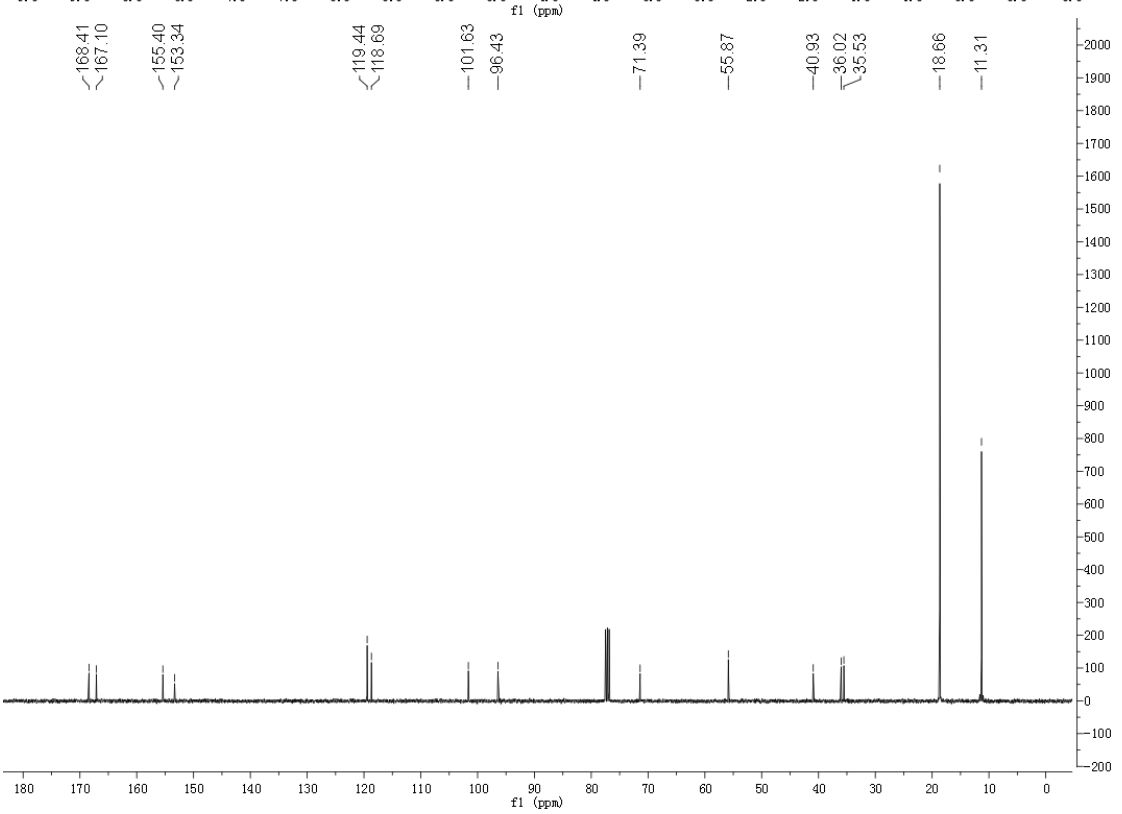

7.61
6.94
6.93
6.86
6.84
6.79
6.78
6.77
6.76
4.53
4.09
4.08
3.73
2.97
2.95
1.13
1.12
1.11
1.09
1.08
1.06
0.94
0.97
1.00
1.00
2.00
2.01
3.02
6.00
21.10
f1 (ppm)

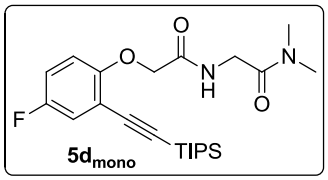

168.78
167.32
154.66
152.86
118.86
116.76
115.77
115.31
102.39
95.94
70.06
55.77
40.70
35.97
35.57
18.69
11.32
f1 (ppm)

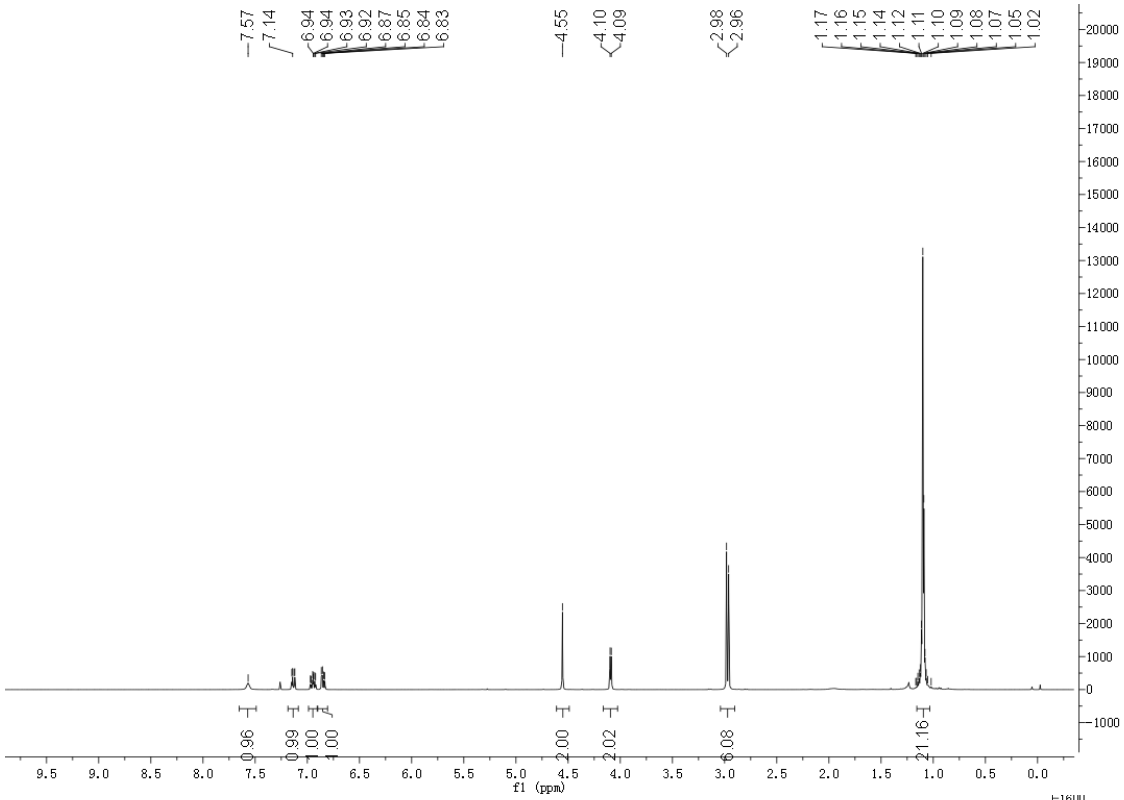

O
O
N
H
N
O
o
o'
MeO
TIPS

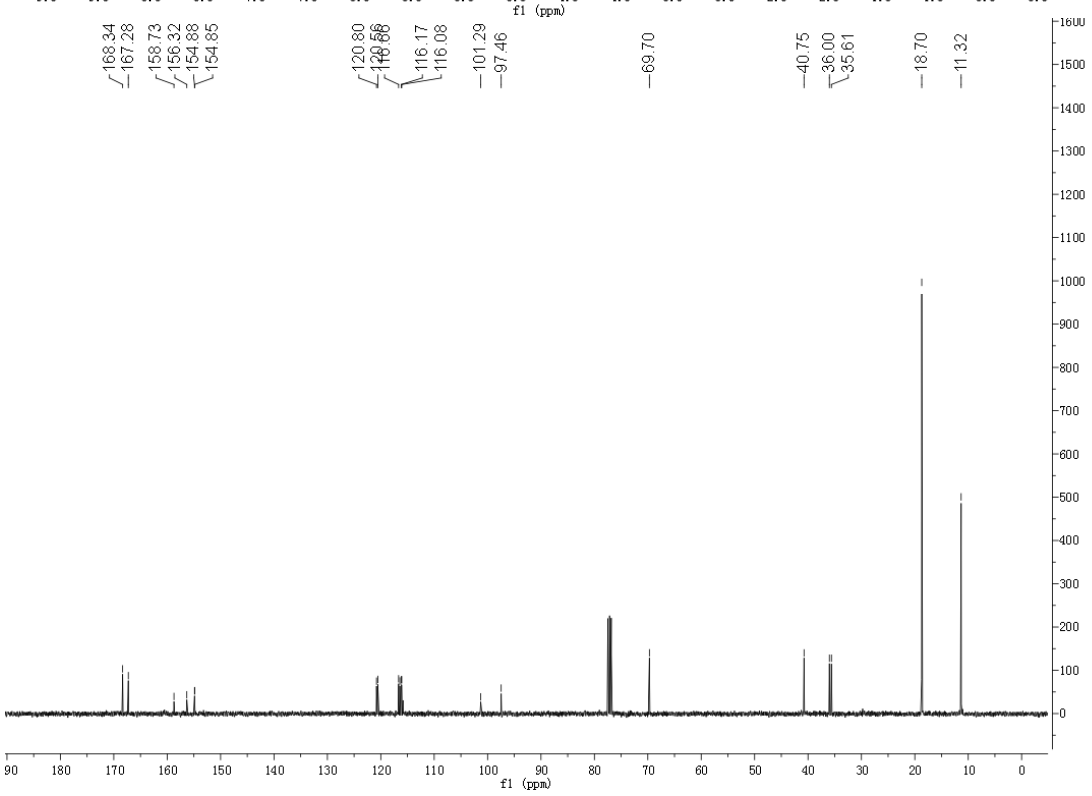
5c$_{(o+o')di}$

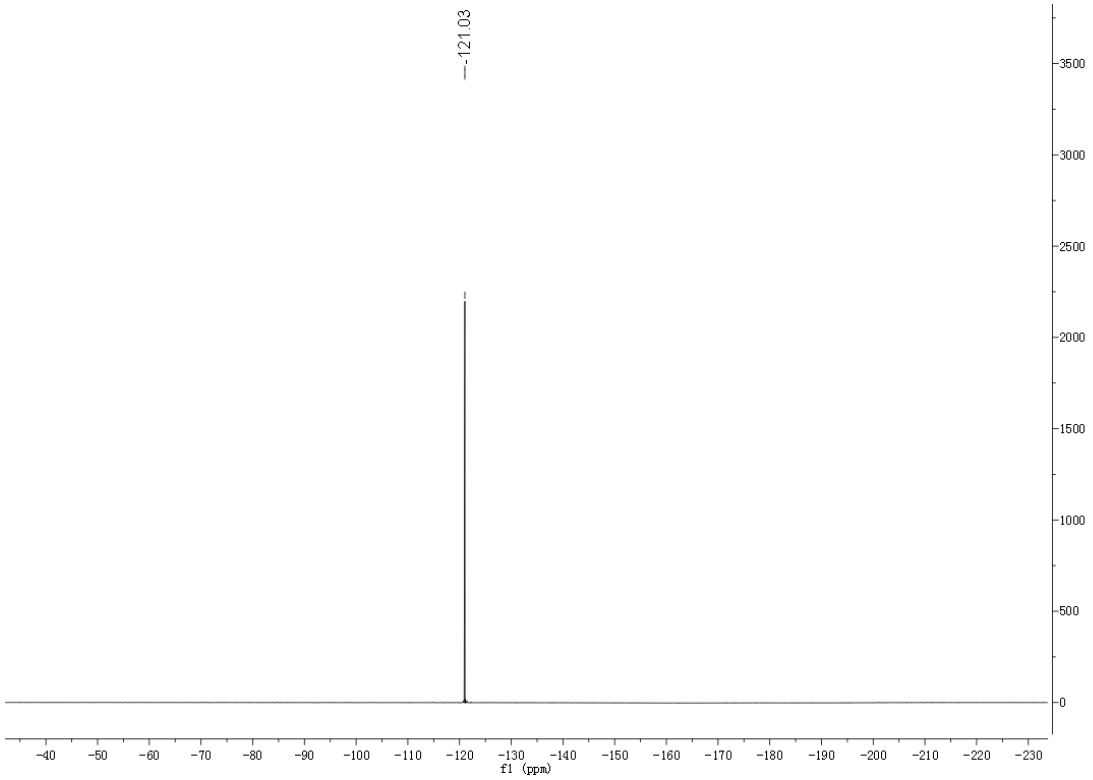

7.88
6.91
4.60
4.05
4.04
3.76
2.99
2.98
1.24
1.21
1.20
1.18
1.10
1.09
1.06
1.01
0.96
0.96
0.98
2.01
2.00
2.01
3.02
6.00
42.10
f1 (ppm)

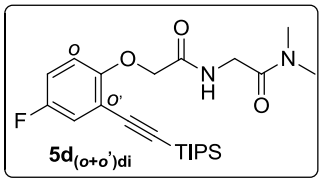

168.41
167.10
155.40
153.34
119.44
118.69
101.63
96.43
71.39
55.87
40.93
36.02
35.53
18.66
11.31
f1 (ppm)


O
O
N
H
N
O
F
TIPS
5d$_{mono}$

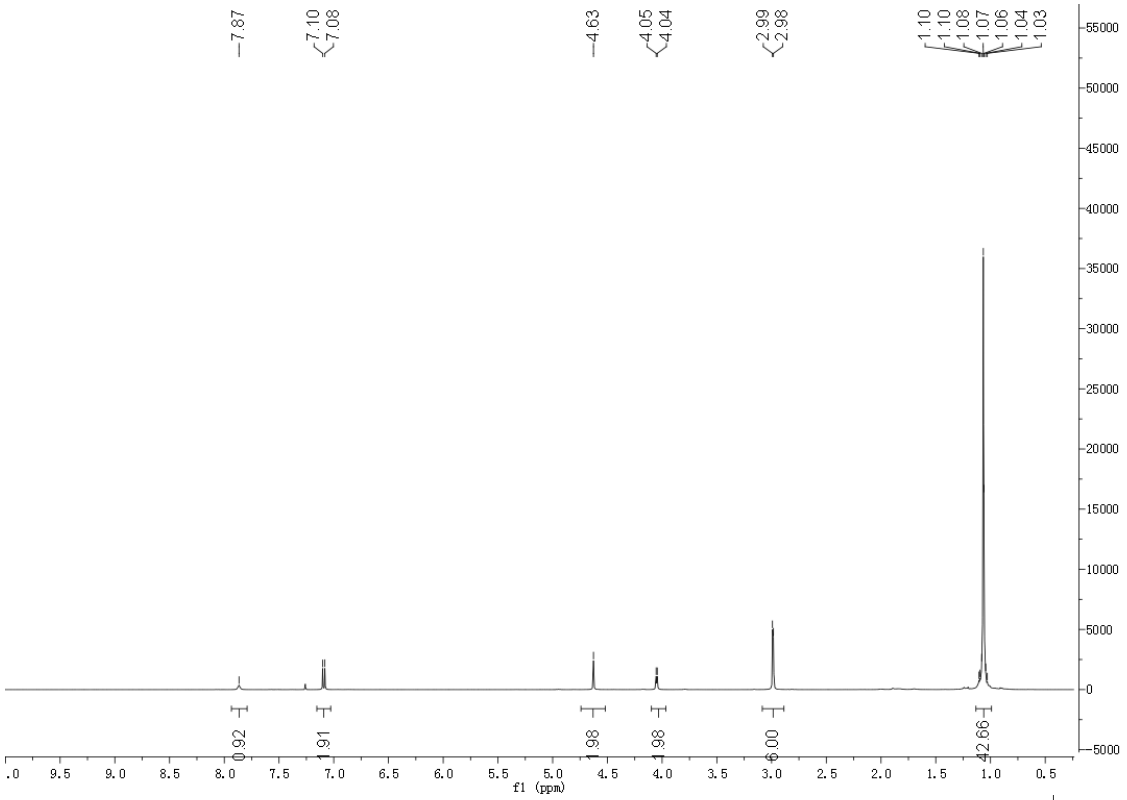

7.57
7.14
6.94
6.94
6.93
6.92
6.87
6.85
6.84
6.83
4.55
4.10
4.09
2.98
2.96
1.17
1.16
1.15
1.14
1.12
1.11
1.10
1.09
1.08
1.07
1.05
1.02
0.96
0.99
1.00
1.00
2.00
2.02
6.08
21.16
f1 (ppm)

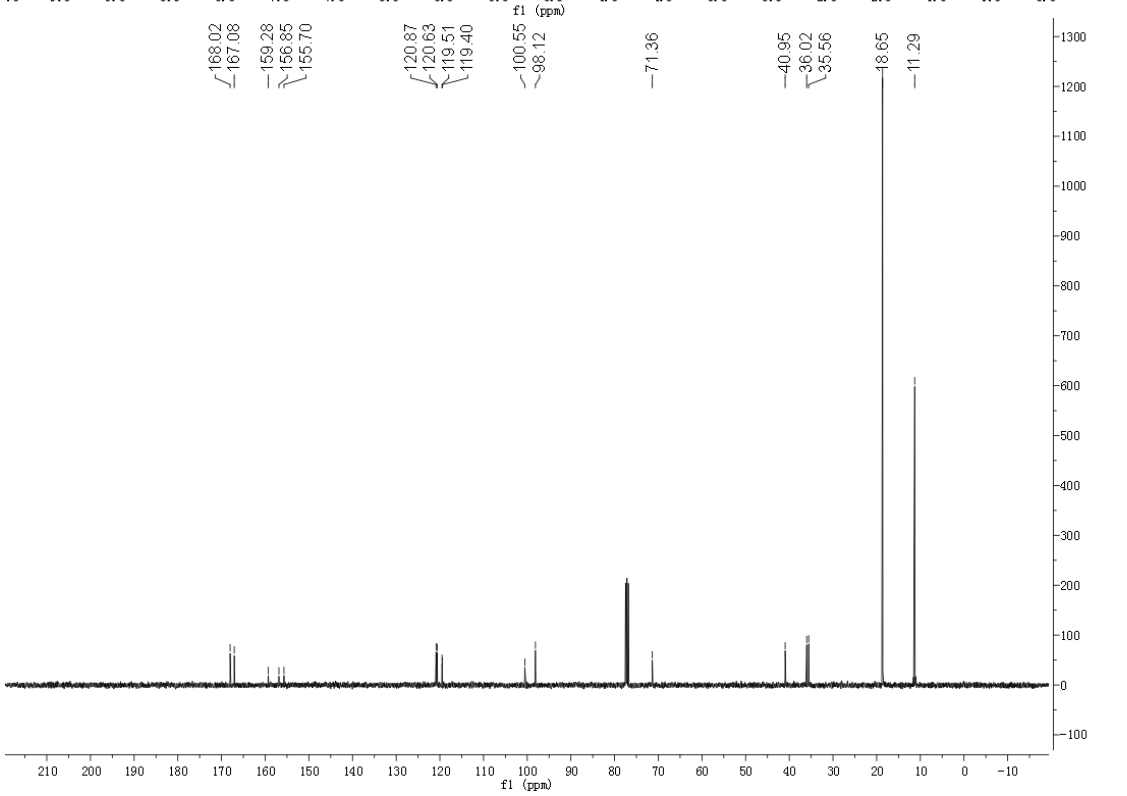

168.34
167.28
158.73
156.32
154.88
154.85
120.80
116.17
116.08
101.29
97.46
69.70
40.75
36.00
35.61
18.70
11.32
f1 (ppm)

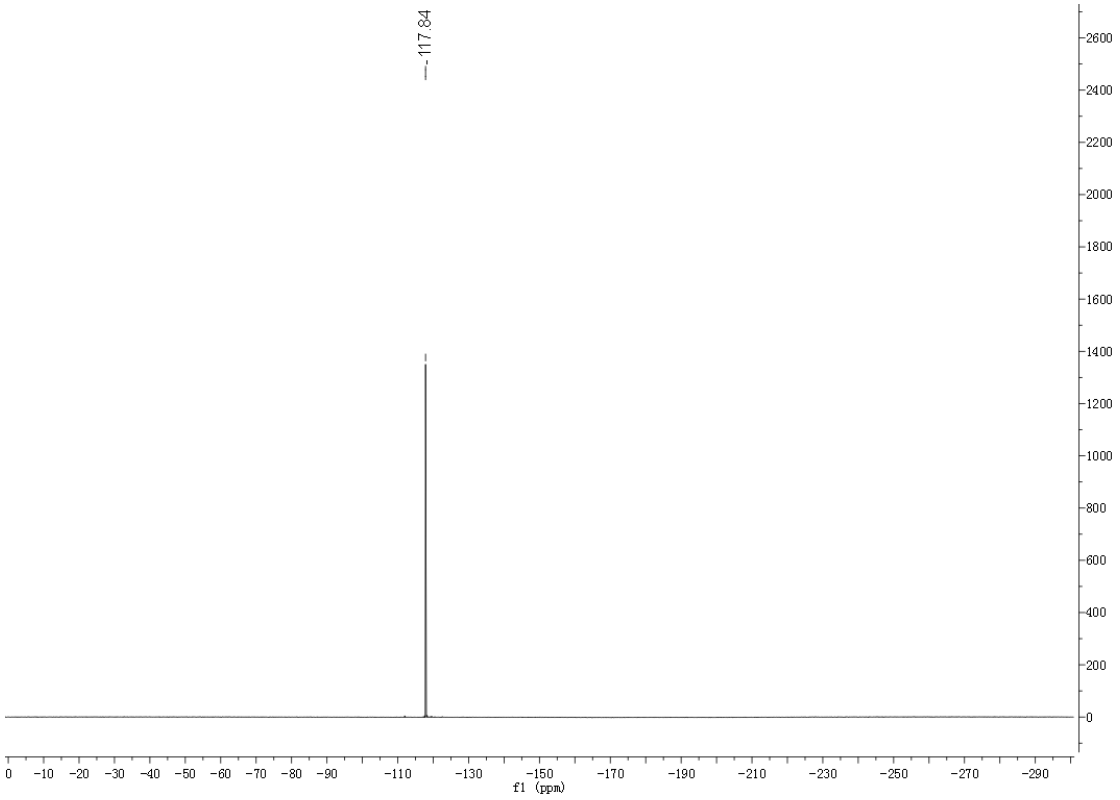

-121.03
3500
3000
2500
2000
1500
1000
500
0
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
f1 (ppm)

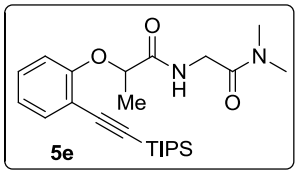

O
O
N
H
N
O
O
O'
F
TIPS

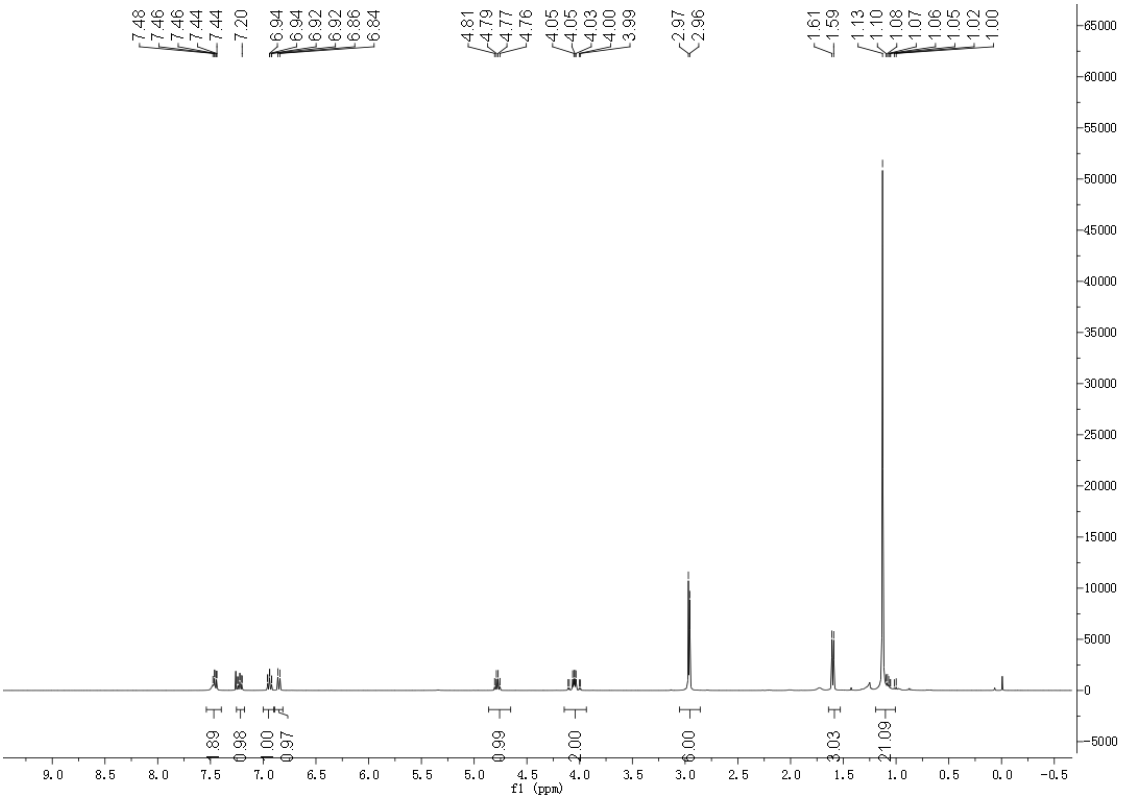
5d$_{(o+o')di}$

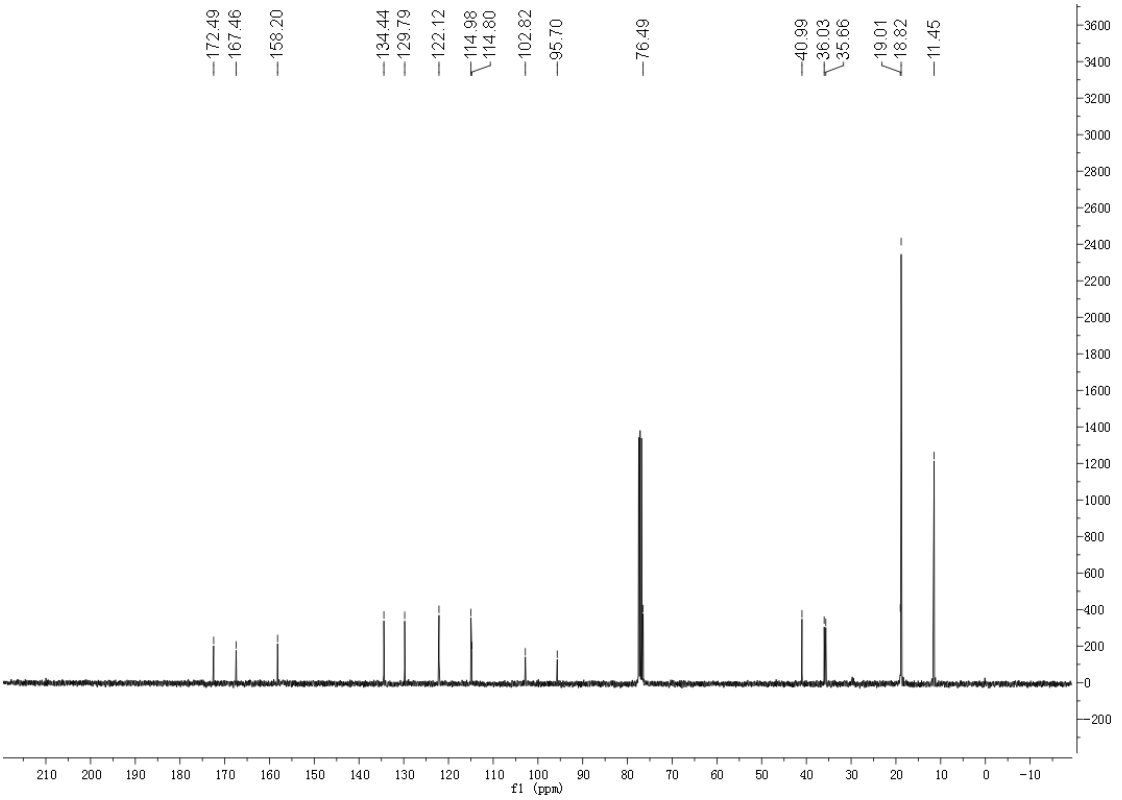

7.87
7.10
7.08
4.63
4.05
4.04
2.99
2.98
1.10
1.10
1.08
1.07
1.06
1.04
1.03
0.92
1.91
1.98
1.98
6.00
12.66
f1 (ppm)

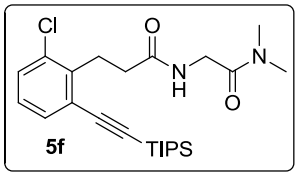

168.02
167.08
159.28
156.85
155.70
120.87
120.63
119.51
119.40
100.55
98.12
71.36
40.95
36.02
35.56
18.65
11.29
f1 (ppm)

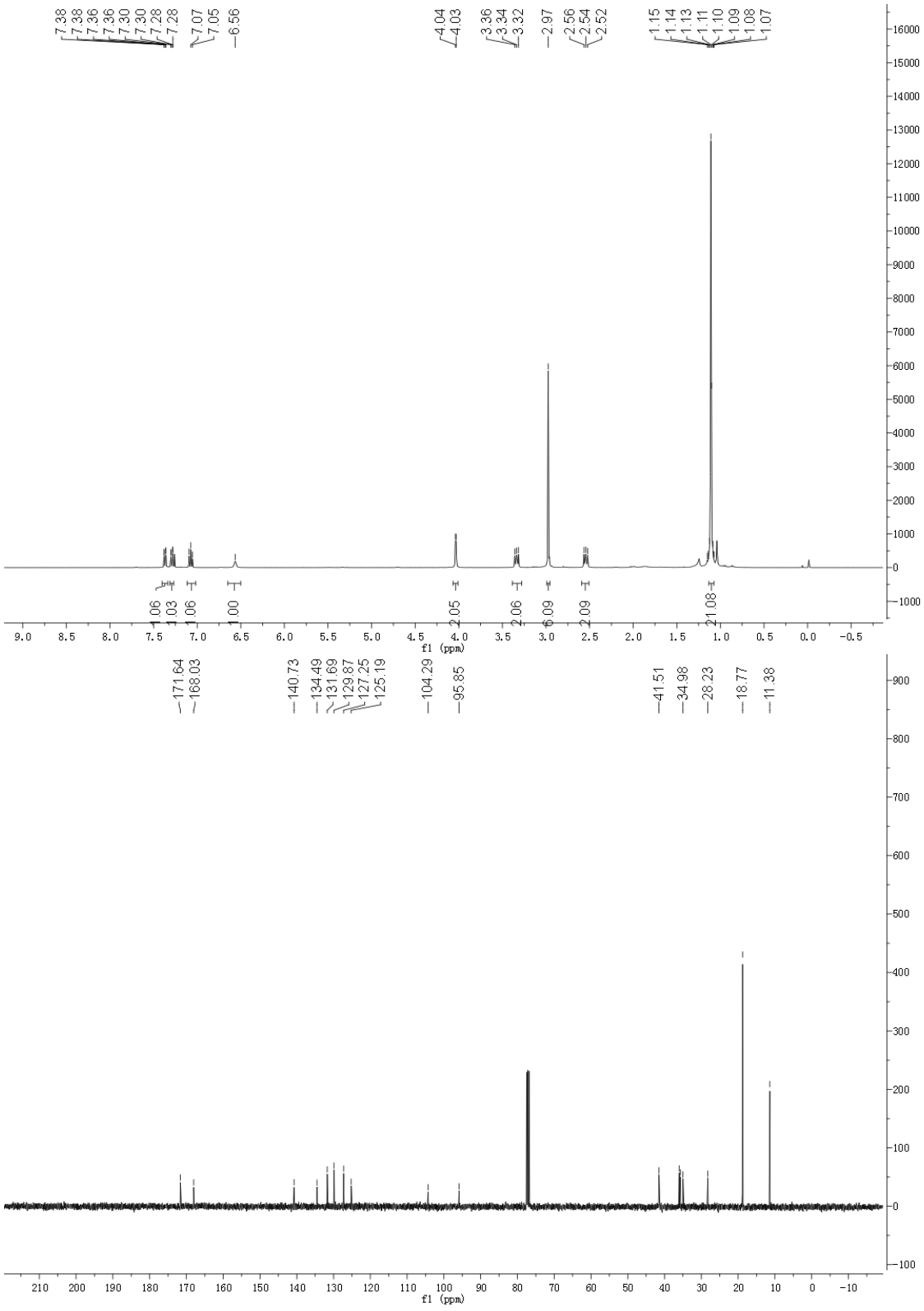

-117.84
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

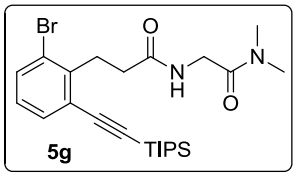

O
O
N
H
Me
O
N
5e
TIPS

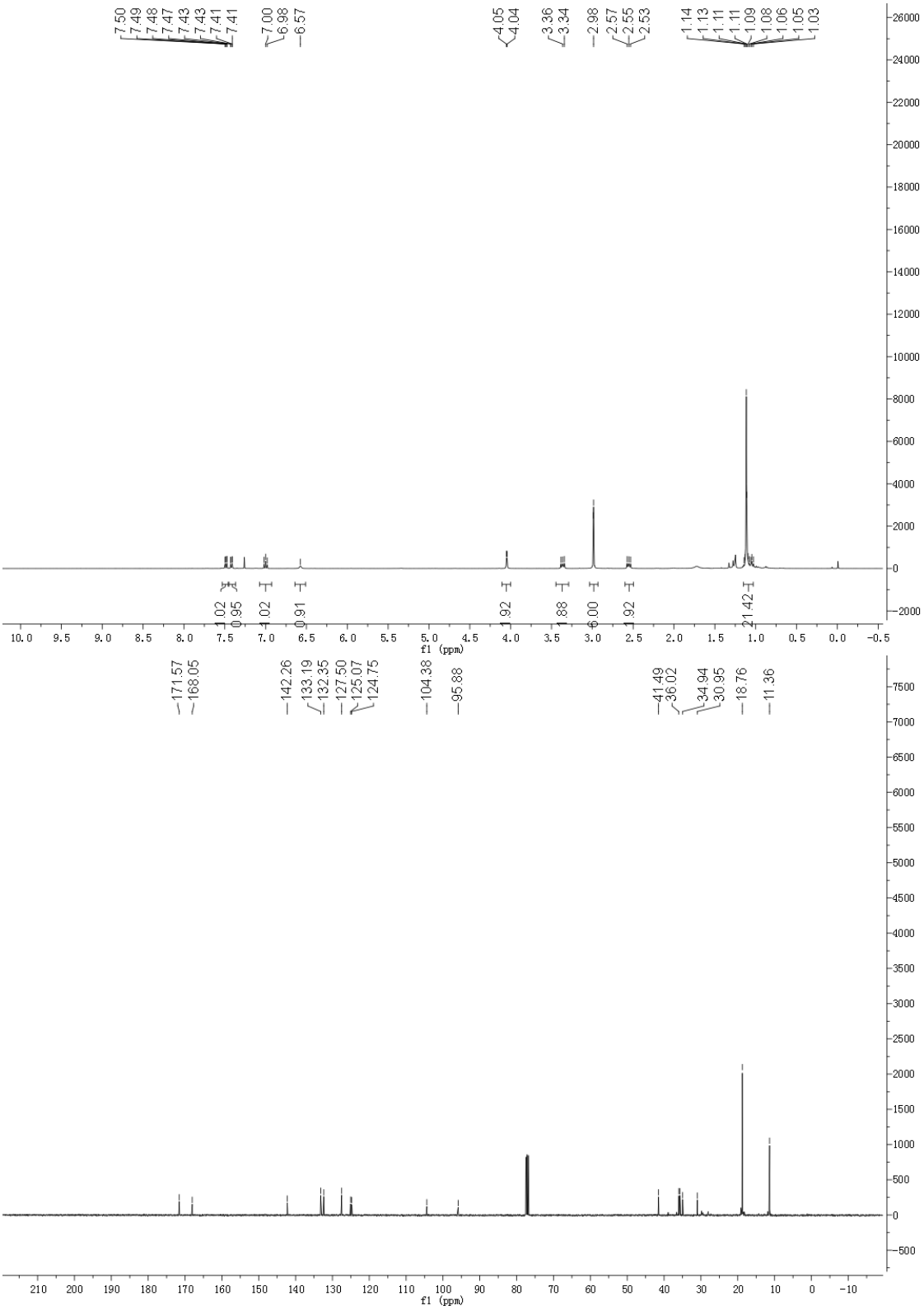

7.48
7.46
7.46
7.44
7.44
7.20
6.94
6.94
6.92
6.92
6.86
6.84
4.81
4.79
4.77
4.76
4.05
4.03
4.00
3.99
2.97
2.96
1.61
1.59
1.13
1.10
1.08
1.07
1.06
1.05
1.02
1.00
1.89
0.98
1.00
0.97
0.99
2.00
6.00
3.03
21.09
f1 (ppm)

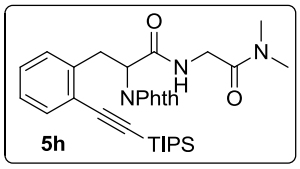

172.49
167.46
158.20
134.44
129.79
122.12
114.98
114.80
102.82
95.70
76.49
40.99
36.03
35.66
19.01
18.82
11.45
f1 (ppm)

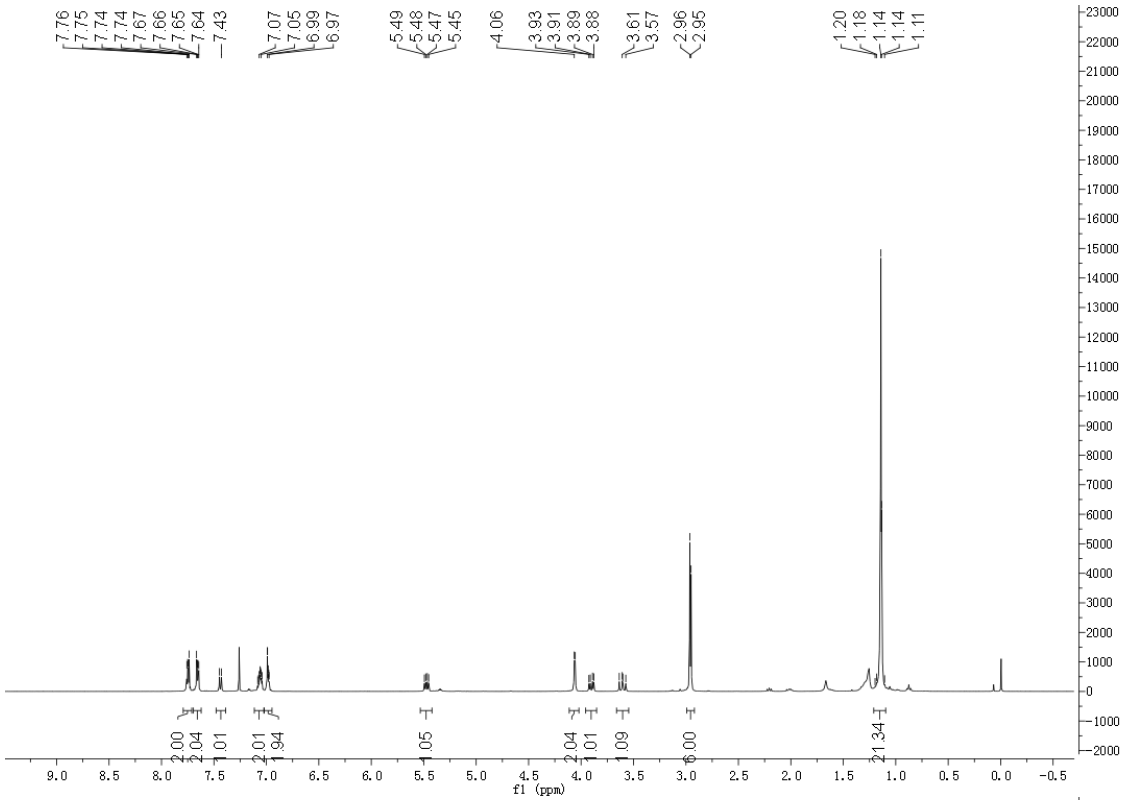

Cl
O
N
H
O
N
5f
TIPS

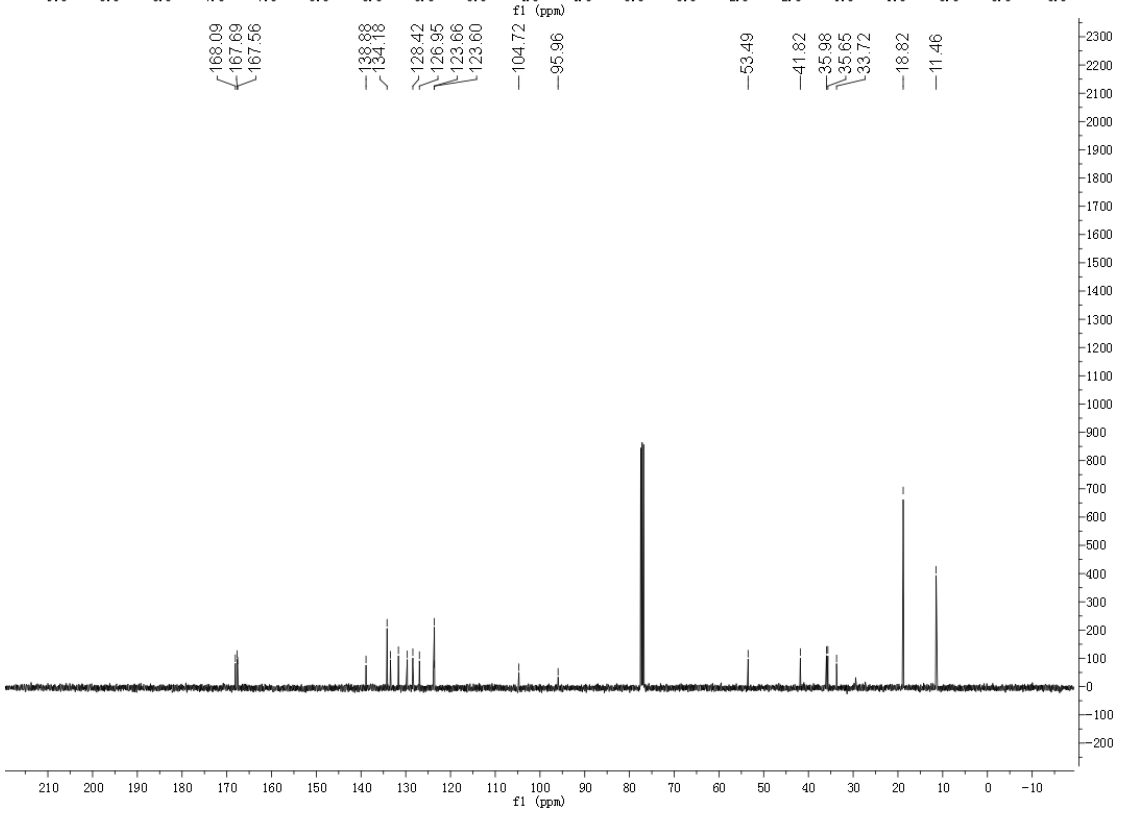

7.38
7.38
7.36
7.36
7.30
7.30
7.28
7.28
7.07
7.05
6.56
4.04
4.03
3.36
3.34
3.32
2.97
2.56
2.54
2.52
1.15
1.14
1.13
1.11
1.10
1.09
1.08
1.07
1.06
1.03
1.06
1.00
2.05
2.06
6.09
2.09
21.08
f1 (ppm)
171.64
168.03
140.73
134.49
131.69
129.87
127.25
125.19
104.29
95.85
41.51
34.98
28.23
18.77
11.38
f1 (ppm)

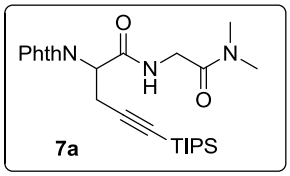

Br
O
N
H
O
N
TIPS
5g

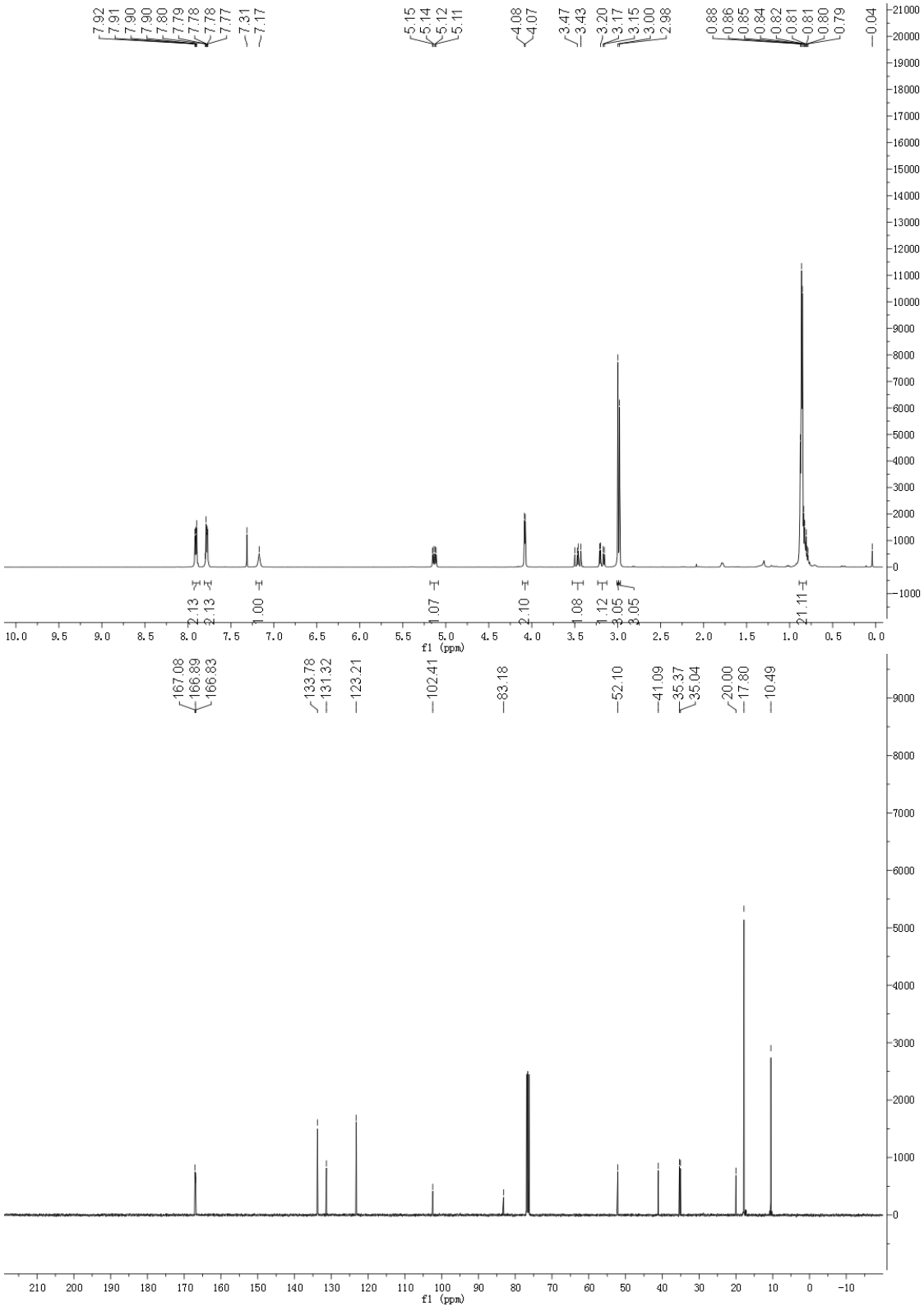

7.50
7.49
7.48
7.47
7.43
7.43
7.41
7.41
7.00
6.98
6.57
4.05
4.04
3.36
3.34
2.98
2.57
2.55
2.53
1.14
1.13
1.11
1.11
1.09
1.08
1.06
1.05
1.03
1.02
0.95
4.02
0.91
1.92
1.88
6.00
1.92
21.42
f1 (ppm)
171.57
168.05
142.26
133.19
132.35
127.50
125.07
124.75
104.38
95.88
41.49
36.02
34.94
30.95
18.76
11.36
f1 (ppm)

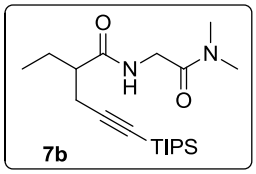

O
N
H
NPhth
O
N
5h
TIPS


7.76
7.75
7.74
7.74
7.67
7.66
7.65
7.64
7.43
7.07
7.05
6.99
6.97
5.49
5.48
5.47
5.45
4.06
3.93
3.91
3.89
3.88
3.61
3.57
2.96
2.95
1.20
1.18
1.14
1.14
1.11
2.00
2.04
1.01
2.01
1.94
1.05
2.04
1.01
1.09
6.00
21.34
f1 (ppm)

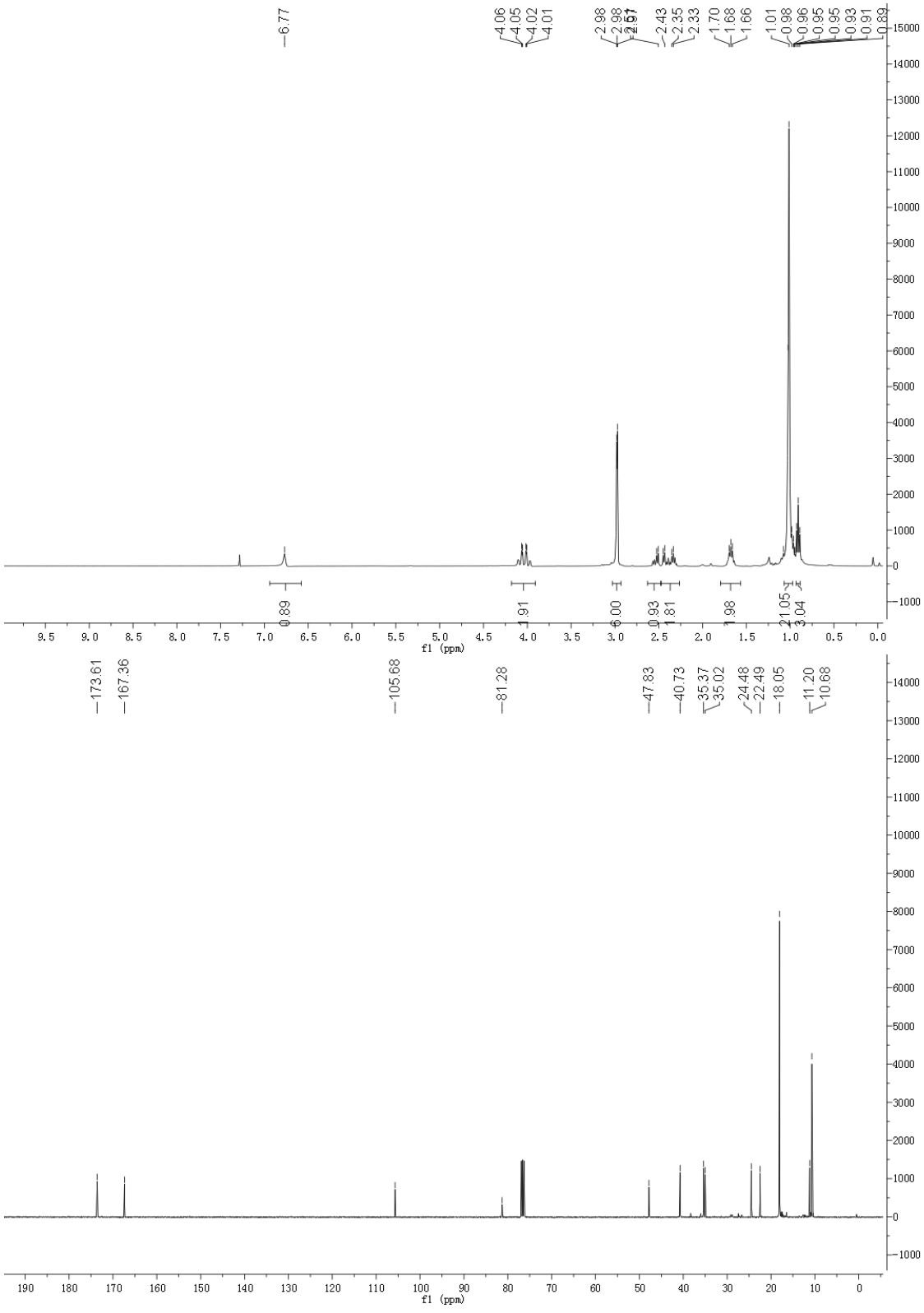

168.09
167.69
167.56
138.88
134.18
128.42
126.95
123.66
123.60
104.72
95.96
53.49
41.82
35.98
35.65
33.72
18.82
11.46
f1 (ppm)

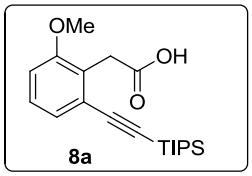

PhthN
O
N
H
N
O
7a
TIPS


7.92
7.91
7.90
7.90
7.80
7.79
7.78
7.78
7.77
7.31
7.17
5.15
5.14
5.12
5.11
4.08
4.07
3.47
3.43
3.20
3.17
3.15
3.00
2.98
0.88
0.86
0.85
0.84
0.82
0.81
0.81
0.80
0.79
0.04
2.13
2.13
1.00
1.07
2.10
1.08
1.12
3.05
3.05
21.11
f1 (ppm)
167.08
166.89
166.83
133.78
131.32
123.21
102.41
83.18
52.10
41.09
35.37
35.04
20.00
17.80
10.49
f1 (ppm)

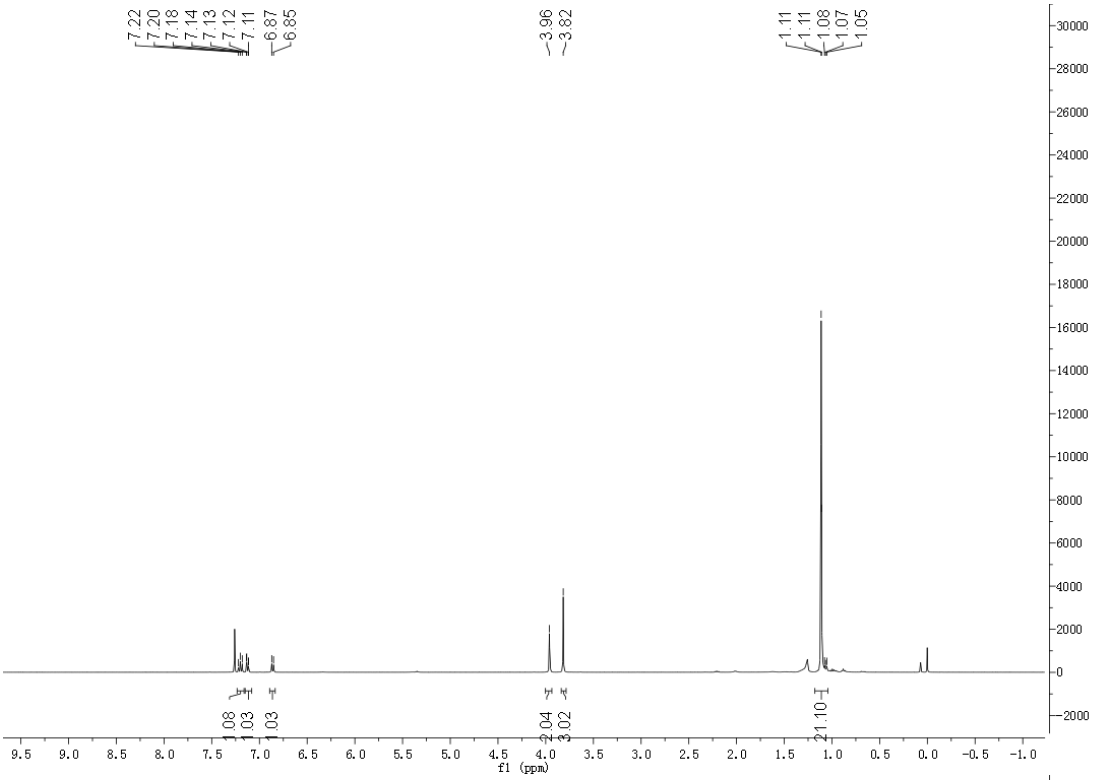

O
N
H
O
N
TIPS

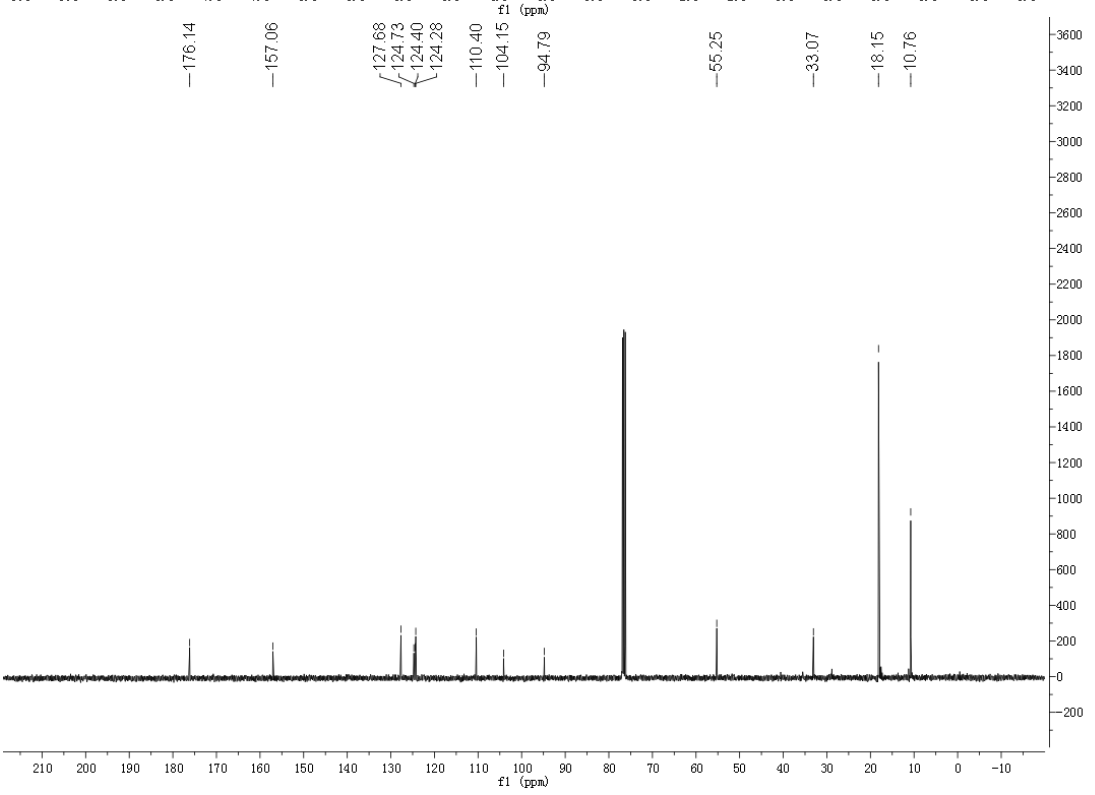
7b


6.77
4.06
4.05
4.02
4.01
2.98
2.98
2.43
2.35
2.33
1.70
1.68
1.66
1.01
0.98
0.96
0.95
0.95
0.93
0.91
0.89
0.89
1.91
6.00
0.93
1.81
1.98
21.05
3.04
f1 (ppm)
173.61
167.36
105.68
81.28
47.83
40.73
35.37
35.02
24.48
22.49
18.05
11.20
10.68
f1 (ppm)


OMe
OH
O
TIPS

8a


7.22
7.20
7.18
7.14
7.13
7.12
7.11
6.87
6.85
3.96
3.82
1.11
1.11
1.08
1.07
1.05
1.08
1.03
1.03
2.04
3.02
21.10
f1 (ppm)


176.14
157.06
127.68
124.73
124.40
124.28
110.40
104.15
94.79
55.25
33.07
18.15
10.76
f1 (ppm)