Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Construction of alkynyl and acyl disulfides directly through thiol-modification with N-alkynylthio phthalimides under acid catalysis

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1. General information

NMR

^{1}H and ^{13}C spectra were collected on 300 M, 400 M or 500 M Hz NMR spectrometers (Bruker AVANCE). Chemical shifts for protons are reported in parts per million (ppm) downfield and are referenced to residual protium in the NMR solvent. ($CHCl_3$ = 7.26 ppm, DMSO = 2.50 ppm). Chemical for carbon are reported in parts per million downfield and are referenced to the carbon resonances of solvent ($CHCl_3$ = 77.0 ppm, DMSO = 39.52 ppm). Date are represented as follows: chemical shift, multiplicity (br = broad, s = singlet, d = double, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration.

MS

High-resolution mass spectra (HRMS) were performed on a micrOTOF-Q II instrument with an ESI or EI source.

Chromatography

All solvents were obtained from commercial sources and were purified according to standard procedures. Petroleum ether (PE), where used, has the boiling point range 60-90 ºC. Column chromatography was performed with silica gel (200-300 mesh).

Analytical and preparative HPLC information

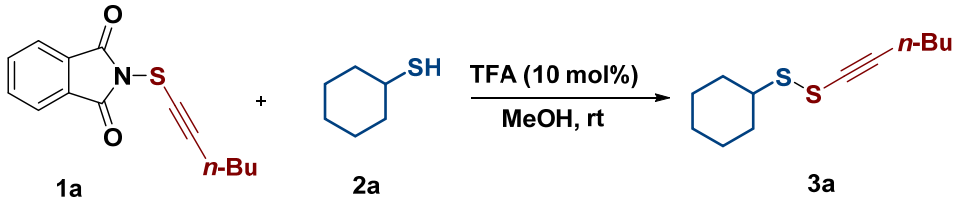
Analytical and preparative HPLC measurements were performed on Shimadzu Essentia LC-16 with DGU-20A detector using C18 column (250 x 4.6 mm, 5 μm). Water (solvent A) and acetonitrile containing 0.1%TFA (solvent B) were used as the mobile phase, at a flow rate of 0.7 mL/min.

Analytic method: 10% B to 90%B (0-20 min), 90%B (20-30 min), 90%B-10%B (30-35 min), 10%B (35-40 min);

Preparation method: 10% B to 90%B (0-10 min), 90%B (10-15 min), 90%B-10%B (15-20 min), 10%B (20-25 min).

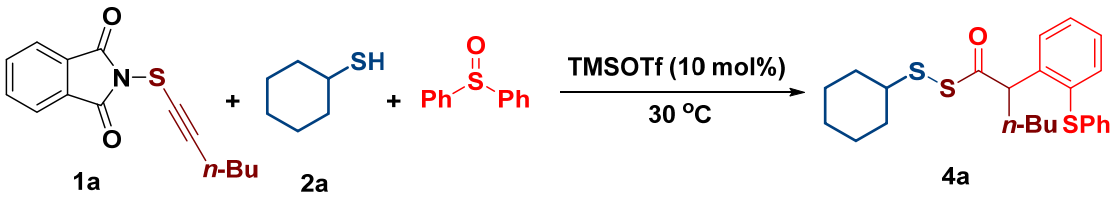
2. General procedure

2.1 General procedure for the synthesis of alkynyl disulfides



In a 10 mL flask, *N*-*n*-butylethynylthio phthalimide **1a** (0.18 mmol, 1.2 equiv.) and cyclohexanethiol **2a** (0.15 mmol, 1.0 equiv.), trifluoroacetic acid (2 μL, 10 mol%) were dissolved in 1 mL of dry MeOH. The reaction mixture was stirred at room temperature for 10 min after cyclohexanethiol was completely consumed. The solvent was removed in vacuum and the crude product was purified by flash column chromatography to give the product **3a** as colorless oil (84% yield).

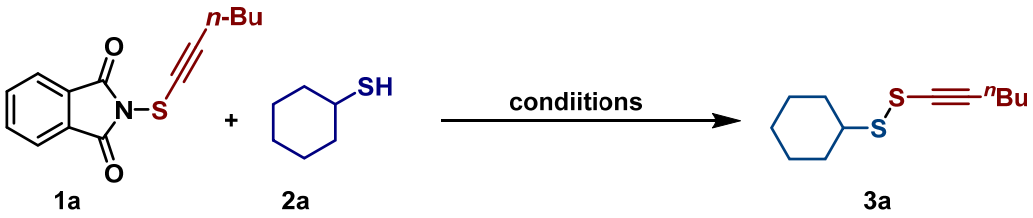
2.1 General procedure for disulfuration and oxyarylation



N-n-butylethynylthio phthalimide **1a** (0.15 mmol), cyclohexanethiol **2a** (0.18 mmol, 1.2 equiv.), phenyl sulfoxide (0.3 mmol, 2.5 equiv.) and TMSOTf (2.7 μL, 10 mol%), were added one portion into a 10 mL flask. The reaction mixture was stirred at 30°C for 20 h. The solvent was removed in vacuum and the crude product was purified by flash column chromatography to give the desired product **4a** as colorless oil (82% yield).

3. Condition optimization

3.1 Table S1. Condition optimization for disulfuration[a]

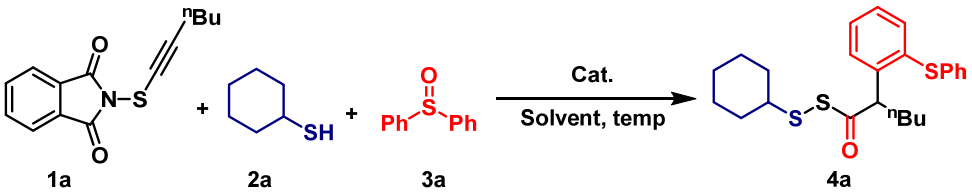


1a 2a 3a

Solvent	Catalyst (10 mol%)	Time	Yield of 3a (%)[b]
CH_2Cl_2 (1 M)	--	24 h	51
MeOH (1 M)	--	2 h	69
MeOH (1 M)	TMSOTf	10 min	77
MeOH (1 M)	TfOH	1 min	80
MeOH (1 M)	TFA	1 min	84

[a]. Conditions: **1a** (0.18 mmol), **2a** (0.15 mmol) and catalyst (10 mol%) in solvent (1.5 mL) were stirred at room temperature until **2a** was consumed completely. [b] Isolated yield.

Table S2 Condition optimization for disulfuration and oxyarylation[a]



1a 2a 3a 4a

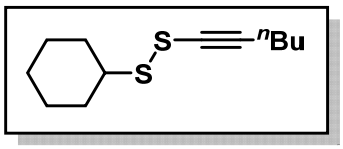
Entry	Solvent	Cat. (mol%)	sulfoxide (equiv.)	Temp. (℃)	Yield (%)[b]
1	--[c]	TfOH (10)	2.5	16	23%
2	--[c]	TfOH (10)	2.5	30	70%
3	--[c]	TfOH (10)	2.5	50	67%
4	--[c]	TfOH (10)	2.5	80	60%
5	--[c]	TfOH (10)	1.5	30	65%
6	--[c]	TfOH (10)	4.0	30	26%
7	--[c]	Tf_2NH (10)	2.5	30	47%
8	--[c]	Tf_2O (10）	2.5	30	78%
9	--[c]	TFA(10)	2.5	30	＜10%

10	**--[c]**	**TMSOTf (10)**	**2.5**	**30**	**82%**
11	--[c]	TMSOTf (10)	1.2	30	69%
12	--[c]	TMSOTf (10)	1.5	30	77%
13	TFE	TfOH(10)	2.5	30	47%
14	CH_2Cl_2	TfOH (10)	2.5	50	19%

[a] Conditions: **1a** (0.15 mmol), **2a** (1.2 equiv), **3a** (m equiv) and **catalyst** (n mol%) were stirred at indicated temperature for 20 h. [b] Isolated yield. [c] No solvent was added.

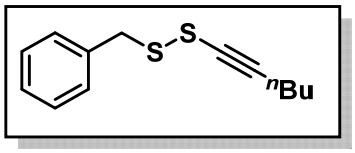
4. Characterization of products

1-Eyclohexyl-2-(hex-1-yn-1-yl)disulfane (3a)



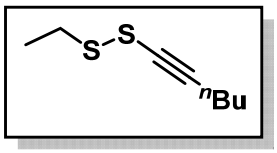
Yield: 28.7 mg (84%); time: 30 s; colorless liquid; TLC, R_f =0.40 (PE:EtOAc = 99:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.00 (tt, J = 10.8, 3.6 Hz, 1H), 2.33 (t, J = 6.8 Hz, 2H), 2.18-2.03 (m, 2H), 1.83-1.79 (m, 2H), 1.66-1.61 (m, 1H), 1.54-1.27 (m, 9H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 95.5, 70.7, 49.9, 32.5, 30.5, 26.0, 25.6, 21.9, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_{12}H_{21}S_2$ $[M+H]^+$: 229.1079, found: 229.1083.

1-Benzyl-2-(hex-1-yn-1-yl)disulfane (3b)



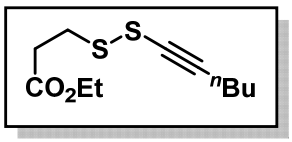
Yield: 21.2 mg (60%); time: 30 s; yellow liquid; TLC, R_f = 0.20 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.34 (m, 3H), 7.32-7.28 (m, 2H), 4,12 (s, 2H), 2.32 (t, J = 6.8 Hz, 2H), 1.53-1.46 (m, 2H), 1.43-1.38 (m, 2H), 0.92 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.3, 129.5, 128.5, 127.6, 97.3, 68.9, 42.6, 30.5, 21.9, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_{13}H_{17}S_2$ $[M+H]^+$: 237.0766, found: 237.0762.

1-Ethyl-2-(hex-1-yn-1-yl)disulfane (3c)



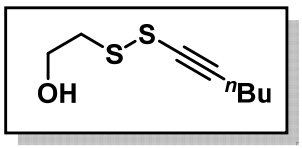
Yield: 15.4 mg (59%); time: 30 s; yellow liquid; TLC, R_f = 0.40 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.92 (q, J = 7.2 Hz, 2H), 2.34 (t, J = 6.8 Hz, 2H), 1.53-1.47 (m, 2H), 1.45-1.38 (m, 5H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 96.5, 69.7, 32.5, 30.5, 21.9, 19.9, 14.2, 13.6. HRMS (ESI) m/z calcd. for $C_8H_{15}S_2$ $[M+H]^+$: 175.0610, found: 175.0603.

6-Ethyl 3-(hex-1-yn-1-yldisulfanyl)propanoate (3d)



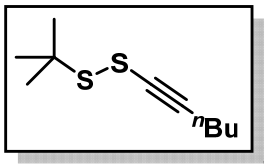
Yield: 29.5 mg (80%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (PE:EtOAc = 19:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.18-4.11 (m, 3H), 3.13 (t, J =7.2 Hz, 2H), 2.83 (t, J = 7.2 Hz, 2H), 2.32 (t, J = 6.8 Hz, 2H), 1.52-1.47 (m, 2H), 1.42-1.38 (m, 2H), 1.27-1.25 (m, 2H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.5, 97.2, 69.2, 60.8, 33.9, 33.2, 30.4, 21.9, 19.9, 14.2, 13.5. HRMS (ESI) m/z calcd. for $C_{11}H_{19}O_2S_2$ $[M+H]^+$: 247.0821, found: 247.0816.

2-(Hex-1-yn-1-yldisulfanyl)ethan-1-ol (3e)



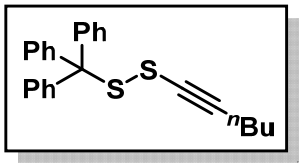
Yield: 26.0 mg (91%); time: 30 s; yellow liquid; TLC, R_f = 0.25 (PE:EtOAc = 17:3); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.99 (t, J = 5.6 Hz, 2H), 3.07 (t, J = 5.6 Hz, 2H), 2.35 (t, J = 7.2 Hz, 2H), 2.13 (s, 1H), 1.55-1.48 (m, 2H), 1.44-1.35 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 97.8, 69.5, 59.7, 41.4, 30.4, 21.9, 19.8, 13.5. HRMS (ESI) m/z calcd. for $C_8H_{15}OS_2$ $[M+H]^+$: 191.0559, found: 191.0559.

1-(Tert-butyl)-2-(hex-1-yn-1-yl)disulfane (3f)



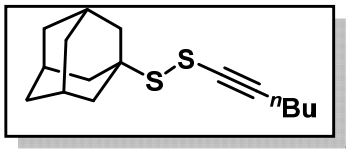
Yield: 26.7 mg (88%); time: 30 s; yellow liquid; TLC, R_f = 0.20 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.30 (t, J = 6.8 Hz, 2H), 1.52-1.45 (m, 2H), 1.42 (s, 9H), 1.40-1.35 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 94.4, 71.4, 49.2, 30.5, 29.7, 21.9, 19.8, 13.5. HRMS (ESI) m/z calcd. for $C_{10}H_{19}S_2$ $[M+H]^+$: 203.0923, found: 203.0910.

1-(Hex-1-yn-1-yl)-2-trityldisulfane (3g)



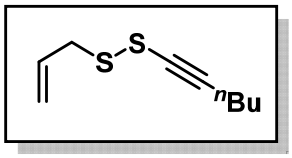
Yield: 46.0 mg (79%); time: 30 s; yellow liquid; TLC, R_f =0.35 (PE:EtOAc =49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.27 (m, 14H), 7.25-7.23 (m, 1H), 2.24 (t, J = 6.8 Hz, 2H), 1.48-1.41 (m, 2H), 1.40-1.31 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 143.4, 130.3, 127.8, 127.2, 97.0, 72.7, 68.9, 30.5, 21.9, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_{25}H_{24}S_2Na$ $[M+Na]^+$: 411.1212, found: 411.1211.

1-((3s,5s,7s)-Adamantan-1-yl)-2-(hex-1-yn-1-yl)disulfane (3h)



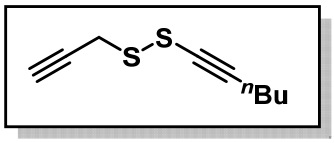
Yield: 36.1 mg (86%); time: 30 s; yellow liquid; TLC, R_f =0.4 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.30 (t, J = 6.8 Hz, 2H), 2.10 (s, 3H), 1.94 (d, J = 3.6, 6H), 1.7 (t, J = 2.4 Hz, 6H), 1.53-1.44 (m, 2H), 1.42-1.35 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 94.0, 71.9, 50.7, 42.3, 36.0, 30.5, 29.7, 21.9, 19.8, 13.5. HRMS (ESI) m/z calcd. for $C_{16}H_{25}S_2$ $[M+H]^+$: 281.1392, found: 281.1399.

1-Allyl-2-(hex-1-yn-1-yl)disulfane (3i)



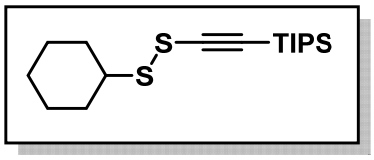
Yield: 24.3 mg (87%); time: 30 s; yellow liquid; TLC, R_f = 0.20 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.94-5.84 (m, 1H), 5.27 (dq, J = 16.8, 3.0 Hz, 1H), 5.21 (dt, J = 10.0, 0.4 Hz, 1H), 3.53 (dt, J = 7.2, 0.8 Hz, 2H), 2.35 (t, J = 7.2 Hz, 2H), 1.53-1.50 (m, 2H), 1.44-1.40 (m, 2H), 0.92 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.3, 119.3, 97.1, 69.2, 41.1, 30.5, 21.9, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_9H_{15}S_2$ $[M+H]^+$: 187.0610, found: 187.0609.

1-(Hex-1-yn-1-yl)-2-(prop-2-yn-1-yl)disulfane (3j)



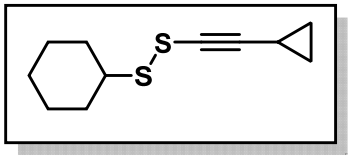
Yield: 20.1 mg (73%); time: 30 s; yellow liquid; TLC, R_f = 0.20 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66 (d, J = 2.8 Hz, 2H), 2.37-2.33 (m, 3H), 1.54-1.48 (m, 2H), 1.43-1.38 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 98.3, 78.5, 72.7, 68.3, 30.4, 26.4, 21.09, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_9H_{13}S_2$ $[M+H]^+$: 185.0453, found: 185.0441.

1-Eyclohexyl-2-(prop-1-yn-1-yl)disulfane compound with triisopropyl-λ^3-silane (1:1) (3k)



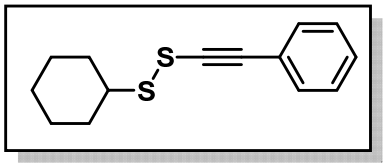
Yield: 42.7 mg (83%); time: 30 s; yellow liquid; TLC, R_f =0.40 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.04 (tt, J = 10.8, 3.6 Hz, 1H), 2.12-2.08 (m, 2H), 1.83-1.79 (m, 2H), 1.66-1.61 (m, 1H), 1.55-1.48 (m, 2H), 1.40-1.30 (m, 3H), 1.07 (s, 21H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 98.6, 97.7, 50.4, 32.6, 26.0, 25.5, 18.5, 11.3. HRMS (ESI) m/z calcd. for $C_{18}H_{36}S_2Si$ $[M+H]^+$: 344.2022, found: 344.2024.

1-1-Eyclohexyl-2-(cyclopropylethynyl)disulfane (3l)



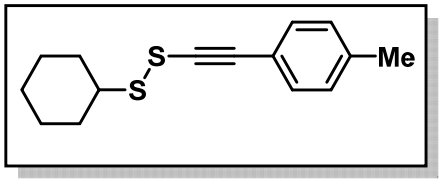
Yield: 27.3 mg (86%); time: 30 s; colorless liquid; TLC, R_f = 0.40 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.00 (tt, J = 10.4, 3.6 Hz, 1H), 2.10-2.06 (m, 2H), 1.83-1.79 (m, 2H), 1.66-1.61 (m, 1H), 1.50-1.41 (m, 2H), 1.40-1.27 (m, 4H), 0.85-0.80 (m, 2H), 0.78-0.74 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 99.6, 66.7, 49.8, 32.5, 25.9, 25.6, 9.2, 0.9. HRMS (ESI) m/z calcd. for $C_{11}H_{16}S_2Na$ $[M+Na]^+$: 235.0586, found: 235.0586.

1-Eyclohexyl-2-(phenylethynyl)disulfane (3m)



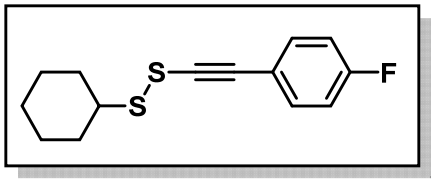
Yield: 28.3 mg (76%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45-7.42 (m, 2H), 7.32-7.29 (m, 3H), 3.09 (tt, J = 10.8, 3.6 Hz, 1H), 2.16-2.12 (m, 2H), 1.87-1.81 (m, 2H), 1.67-1.63 (m, 1H), 1.55-1.48 (m, 2H), 1.43-1.31 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 131.8, 128.6, 128.3, 122.8, 93.2, 81.4, 50.1, 32.5, 26.0, 25.5. HRMS (ESI) m/z calcd. for $C_{14}H_{17}S_2$ $[M+H]^+$: 249.0766, found: 249.0775.

Eyclohexyl-2-(p-tolylethynyl)disulfane (3n)



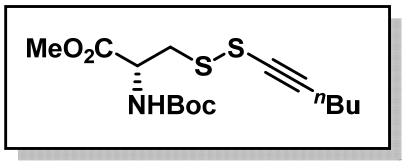
Yield: 19.7 mg (50%); time: 30 s; yellow liquid; TLC, R_f = 0.20 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.33 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 3.09 (tt, J = 10.8, 3.6 Hz, 1H), 2.35 (s, 1H), 2.16-2.12 (m, 2H), 1.85-1.81 (m, 2H), 1.67-1.61 (m, 1H), 1.54-1.47 (m, 2H), 1.42-1.31 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 139.0, 131.9, 129.1, 119.7, 93.5, 80.5, 50.1, 32.5, 26.0, 25.6, 21.5. HRMS (ESI) m/z calcd. for $C_{15}H_{19}S_2$ $[M+H]^+$: 263.0923, found: 263.0938.

1-Eyclohexyl-2-((4-fluorophenyl)ethynyl)disulfane (3o)



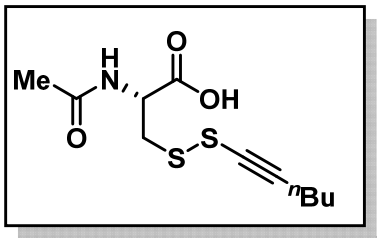
Yield: 19.2 mg (48%); time: 30 s; light yellow liquid; TLC, R_f = 0.30 (PE); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45-7.40 (m, 2H), 7.01 (tt, J = 8.4, 2 Hz, 2H), 3.08 (tt, J = 10.8, 3.6 Hz, 1H), 2.16-2.11 (m, 2H), 1.86-1.81 (m, 2H), 1.68-1.63 (m, 1H), 1.50 (td, J = 10.4, 2.4 Hz, 2H), 1.43-1.31 (m, 3H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.7 (J = 249.0 Hz), 134.0 (J = 9.0 Hz), 118.9 (J = 4.0 Hz), 115.67 (J = 22.0 Hz), 92.1, 81.2, 50.2, 32.5, 26.0, 25.6; ^{19}F NMR ($CDCl_3$, 376.3 MHz): δ -109.84. HRMS (ESI) m/z calcd. for $C_{14}H_{16}FS_2$ $[M+H]^+$: 267.0672, found: 267.0669.

Methyl *N*-(tert-butoxycarbonyl)-*S*-(hex-1-yn-1-ylthio)-*L*-cysteinate (3p)



Yield: 38.0 mg (73%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (PE:EtOAc =10:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.45 (d, J = 8.0 Hz, 1H), 4.72-4.67 (m, 1H), 3.78 (s, 3H), 3.37 (d, J = 4.8 Hz, 2H), 2.37 (t, J = 6.8 Hz, 2H), 1.57-1.49 (m, 2H), 1.45 (s, 9H), 1.42-1.38 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.9, 155.1, 98.7, 80.2, 68.7, 52.6, 40.6, 30.4, 28.3, 28.1, 21.9, 19.9, 13.6. HRMS (ESI) m/z calcd. for $C_{15}H_{25}NO_4S_2Na$ $[M+Na]^+$: 370.1117, found: 370.1129.

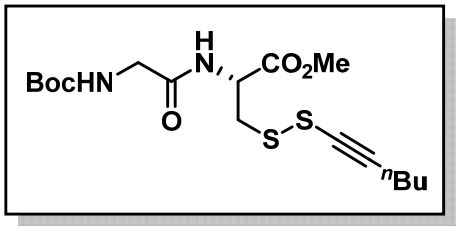
***N*-Acetyl-*S*-(hex-1-yn-1-ylthio)-*L*-cysteine (3q)**



Yield: 22.7 mg (55%); time: 30 s; yellow liquid; TLC, R_f =0.30 (MeOH:DCM = 1.5:10); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.93 (s, 1H), 6.82 (d, J = 7.2 Hz, 1H), 4.95 (d, J = 4.0 Hz, 1H), 3.42 (qd, J = 14.8, 4.0 Hz, 2H), 2.37 (t, J = 6.8 Hz, 2H), 2.1 (s, 3H), 1.56-1.49 (m, 2H), 1.45-1.36 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ

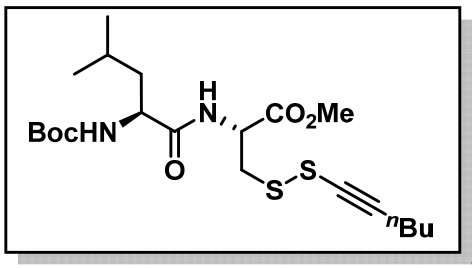
171.6, 167.7, 98.9, 69.0, 52.4, 39.7, 30.4, 22.9, 22.0, 20.0, 13.6. HRMS (ESI) m/*z* calcd. for $C_{11}H_{17}NO_3S_2Na$ $[M+Na]^+$: 298.0542, found: 298.0558.

Methyl *N*-((tert-butoxycarbonyl)glycyl)-*S*-(hex-1-yn-1-ylthio)-*L*-cysteinate (3r)



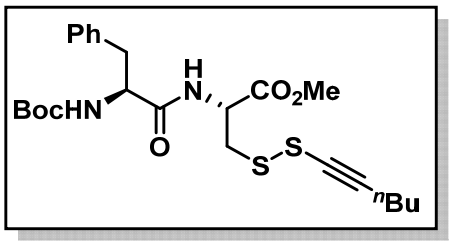
Yield: 44.8 mg (74%); time: 30 s; yellow liquid; TLC, R_f = 0.3 (PE:EtOAc = 7:3); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.00 (d, J = 7.2 Hz, 1H), 5.19 (s, 1H), 4.98 (dt, J = 7.6, 2.8 Hz, 1H), 3.86 (d, J = 5.2 Hz, 2H), 3.78 (s, 3H), 3.39 (ddd, J = 24.0, 14.8, 4.8 Hz, 2H), 2.37 (t, J = 6.8 Hz, 2H), 1.54-1.48 (m, 2H), 1.45 (s, 9H), 1.43-1.37 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.3, 169.2, 155.7, 98.9, 80.3, 68.6, 52.8, 51.7, 44.1, 39.6, 30.4, 28.3, 21.9, 19.9, 13.5. HRMS (ESI) m/*z* calcd. for $C_{17}H_{28}N_2O_5S_2Na$ $[M+Na]^+$: 427.1332, found: 427.1326.

Methyl *N*-((*tert*-butoxycarbonyl)-*L*-leucyl)-*S*-(hex-1-yn-1-ylthio)-*L*-cysteinate (3s)



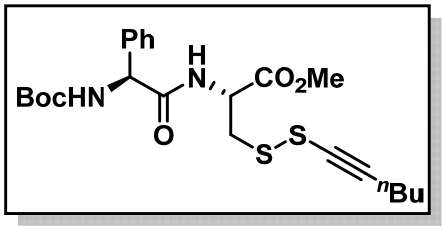
Yield: 65.6 mg (95%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.02 (d, J = 7.2 Hz, 1H), 4.94-4.87 (m, 2H), 4.16 (s, 1H), 3.77 (s, 3H), 3.48 (ddd, J = 42.8, 14.8, 4.8 Hz), 2.37 (t, J = 6.8 Hz, 2H), 1.72-1.65 (m, 2H), 1.55-1.37 (m, 14H), 0.94-0.89 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.4, 170.3, 155.5, 98.6, 80.2, 68.7, 53.0, 52.7, 51.7, 41.0, 39.4, 30.4, 28.3, 24.7, 22.9, 21.9, 19.9, 13.5. HRMS (ESI) m/*z* calcd. for $C_{21}H_{36}N_2O_5S_2Na$ $[M+Na]^+$: 483.1958, found: 483.1959.

Methyl *N*-((*tert*-butoxycarbonyl)-*L*-phenylalanyl)-*S*-(hex-1-yn-1-ylthio)-*L*-cysteinate (3t)



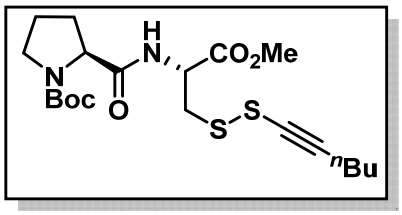
Yield: 65 mg (88%); time: 30 s; white solid; TLC, R_f = 0.2 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.26 (m, 2H), 7.25-7.18 (m, 3H), 6.82 (d, 1H, J = 8.0 Hz), 4.96 (brs, 1H), 4.90-4.80 (m, 1H), 4.41 (brs, 1H), 3.76 (s, 3H), 3.40 (dd, 1H, J = 12.0, 4.0 Hz), 3.30 (dd, 1H, J = 12.0, 4.0 Hz), 3.15-3.01 (m, 2H), 2.36 (t, 2H, J = 8.0 Hz), 1.56-1.48 (m, 2H), 1.41 (m, 11H), 0.91 (t, 3H, J = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.1, 170.1, 155.3, 136.4, 129.3, 128.7, 127.0, 98.8, 80.4, 68.7, 55.6, 52.7, 51.8, 39.5, 38.1, 30.43, 28.2, 21.9, 19.9, 13.5. HRMS (ESI) m/z calcd. for $C_{24}H_{35}N_2O_5S_2$ $[M+H]^+$: 495.1982, found: 495.1982.

Methyl *N*-((*S*)-2-((*tert*-butoxycarbonyl)amino)-2-phenylacetyl)-*S*-(hex-1-yn-1-ylthio)-*L*-cysteinate (3u)



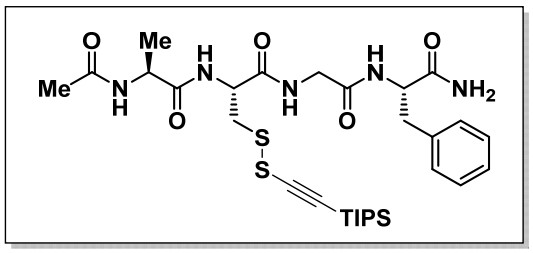
Yield: 64.1 mg (89%); time: 30 s; white solid; TLC, R_f = 0.2 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.30 (m, 5H), 6.75 (d, J = 11.2 Hz, 1H), 5.69 (s, 1H), 5.21 (s, 1H), 4.94-4.90 (m, 1H), 3.71 (s, 3H), 3.39 (ddd, J = 42.6, 14.4, 4.4 Hz, 2H), 2.34 (t, J = 6.8 Hz, 2H), 1.55-1.47 (m, 2H), 1.42 (s, 9H), 1.39-1.35 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.0, 154.9, 137.7, 128.9, 128.4, 127.3, 123.4, 98.8, 80.1, 68.6, 58.7, 52.7, 52.0, 39.4, 30.4, 28.2, 21.9, 19.9, 13.5; HRMS (ESI) m/z calcd. for $C_{23}H_{32}N_2O_5S_2Na$ $[M+Na]^+$: 503.1645, found: 503.1659.

***tert*-Butyl (*S*)-2-(((*R*)-3-(hex-1-yn-1-yldisulfanyl)-1-methoxy-1-oxopropan-2-yl) carbamoyl)pyrrolidine-1-carboxylate (3v)**

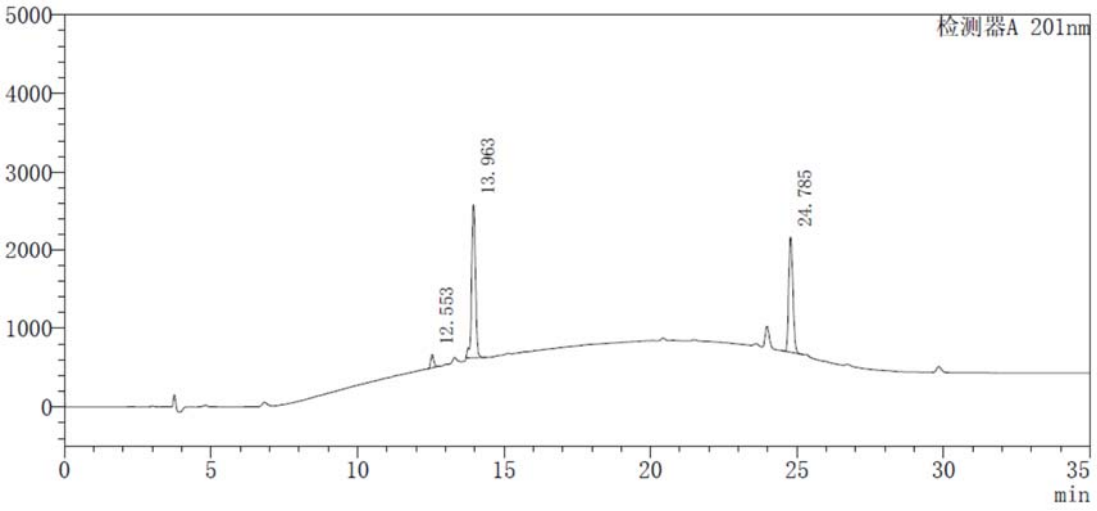


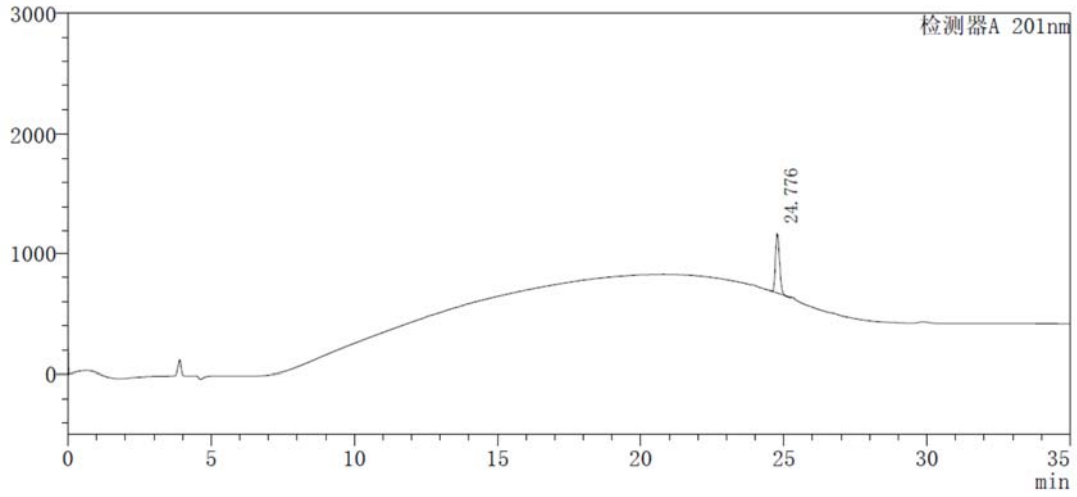
Yield: 50.0 mg (75%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (PE:EtOAc = 7:3); ^{1}H NMR (isomers, $CDCl_3$, 400 MHz): δ 7.64 (s, 0.5H), 6.93 (s, 0.5H), 4.91 (d, J = 27.6 Hz, 1H), 4.28 (d, J = 36 Hz, 1H), 3.75 (s, 3H), 3.45-3.32 (m, 4H), 2.34 (t, J = 6.8 Hz, 2H), 2.13 (s, 1H), 1.90-1.83 (m, 2H), 1.53-1.47 (m, 2H), 1.44 (s, 9H), 1.40-1.33 (m, 3H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR (isomers, $CDCl_3$, 100 MHz): δ 172.6 (171.9), 170.4 (170.1), 155.7 (154.6), 98.8 (98.3), 80.7 (80.4), 68.4, 61.1(59.8), 52.6, 51.5, 47.0, 39.6, 31.0, 30.3, 28.2, 24.5(23.7), 21.9, 19.9, 13.5; HRMS (ESI) m/z calcd. for $C_{20}H_{32}N_2O_5S_2Na$ $[M+Na]^+$: 467.1645, found: 467.1641.

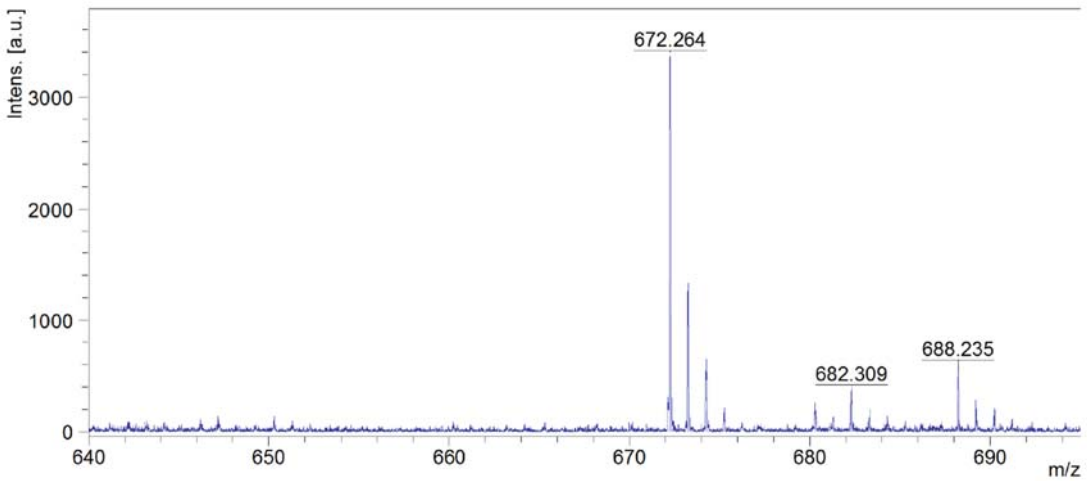
Ac-Ala-Cys-Gly-Phe-NH_2-SS-alkynyl-TIPS (3w)

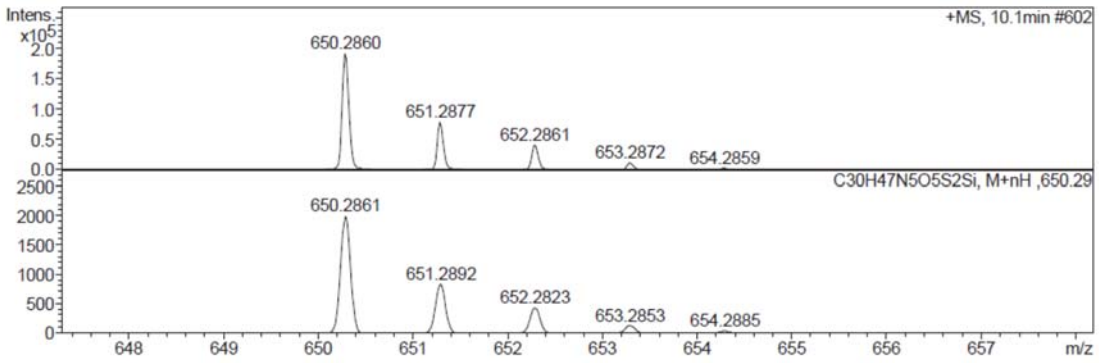


The product was purified by preparative HPLC. Yield: 42 mg (66%); time: 2 h; white solid; LRMS (MALDI/QTOF) m/z: $[M+Na]^+$ $C_{30}H_{47}N_5O_5S_2SiNa$, Calcd. 672.264, found 672.268; $[M+K]^+$ $C_{30}H_{47}N_5O_5S_2SiK$, Calcd. 688.242, found 688.235; HRMS (ESI/micrOTOF) m/z: $[M+H]^+$ $C_{30}H_{48}N_5O_5S_2Si$ Calcd. 650.2861， found: 650.2860.

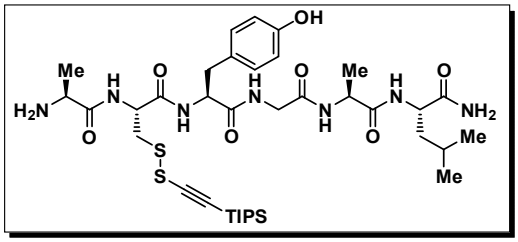

检测器A 201nm
5000
4000
3000
2000
1000
0
12.553
13.963
24.785
0
5
10
15
20
25
30
35
min


检测器A 201nm
3000
2000
1000
0
24.776
0
5
10
15
20
25
30
35
min

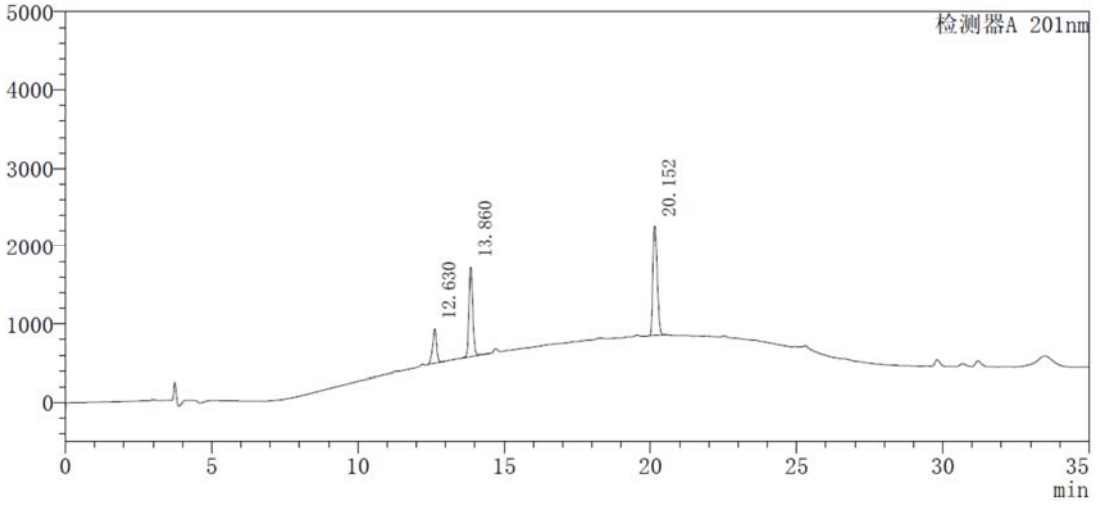

Intens. [a.u.]
3000
2000
1000
0
672.264
682.309
688.235
640
650
660
670
680
690
m/z

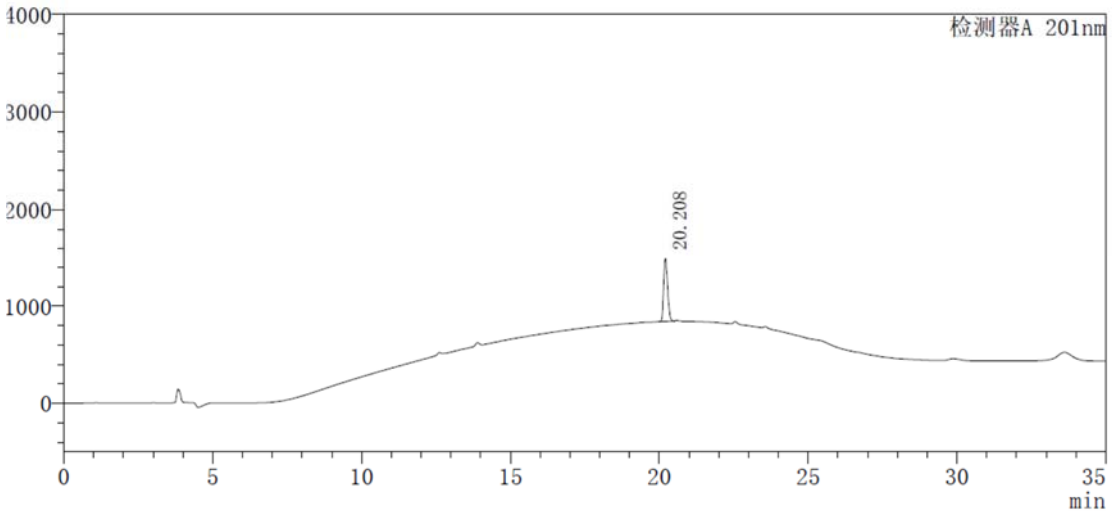


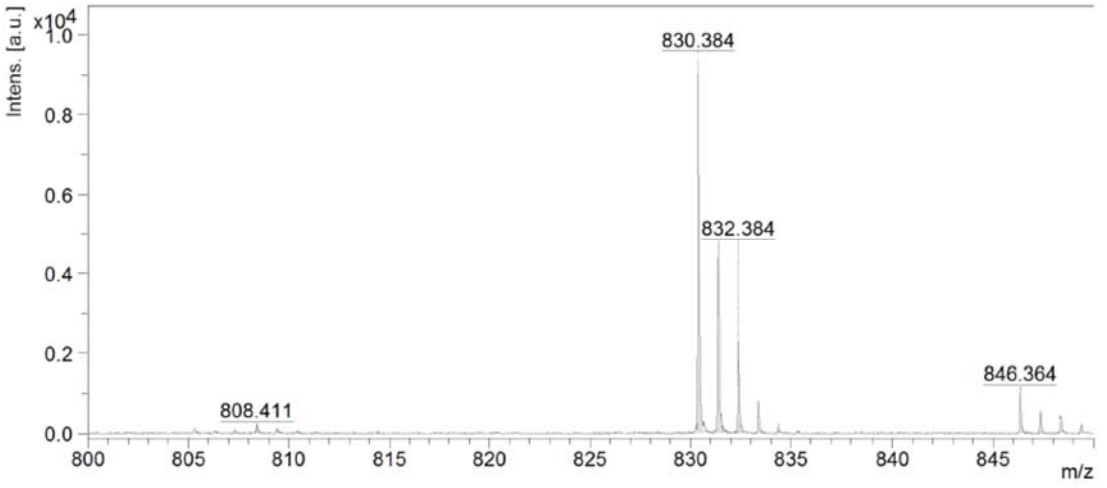
Ala-Cys-Tyr-Gly-Ala-Leu-NH2-SS- alkynyl-TIPS (3x)

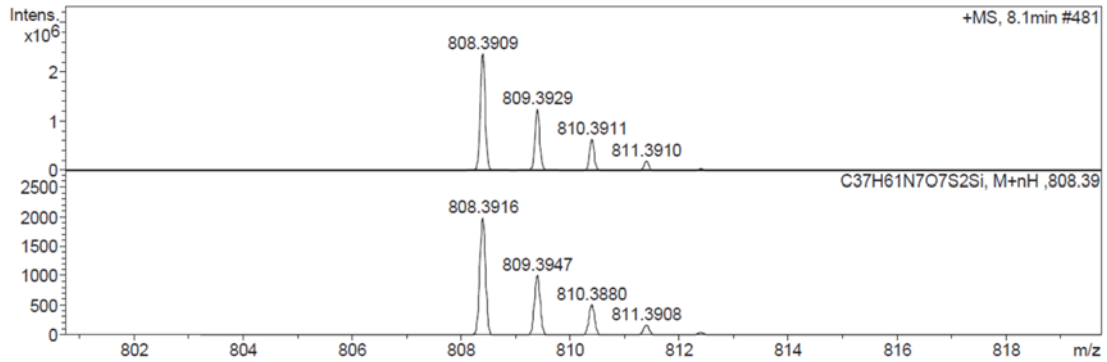


The product was purified by preparative HPLC. Yield: 48 mg (60%); time: 2 h; white solid; LRMS (MALDI/QTOF) m/z: $[M+Na]^+$ $C_{37}H_{61}N_7O_7S_2SiNa$, Calcd. 830.374, found 830.384; $[M+K]^+$ $C_{37}H_{61}N_7O_7S_2SiK$, Calcd. 846.346, found 846.364.HRMS (ESI/micrOTOF) m/z: $[M+H]^+$ $C_{37}H_{62}N_7O_7S_2Si$ Calcd. 808.3916, found: 808.3909.

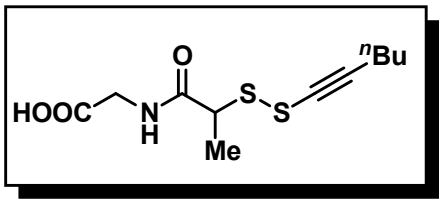








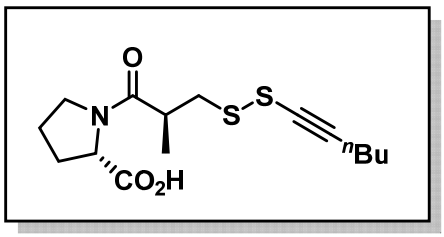
(2-(Hex-1-yn-1-yldisulfanyl)propanoyl)glycine (3y)



Yield: 38.4 mg (93%); time: 30 s; light yellow solid; TLC, R_f = 0.30 (MeOH:DCM =1:4); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.82 (s, 1H), 6.83 (t, J = 4.4 Hz, 1H), 4.13 (ddd, J = 28.4, 28.8 5.2 Hz, 2H), 3.79 (q, J = 7.2 Hz, 1H), 2.33 (t, J = 7.2 Hz, 2H), 1.60 (d, J

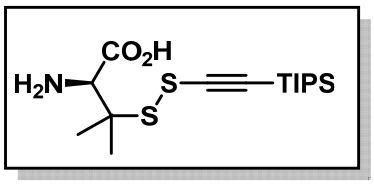
= 7.2 Hz, 3H), 1.53-1.46 (m, 2H), 1.43-1.34 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.3, 171.6, 98.2, 68.5, 49.1, 41.7, 30.4, 21.9, 19.9, 17.0, 13.5; HRMS (ESI) m/*z* calcd. for $C_{11}H_{17}NO_3S_2Na$ $[M+Na]^+$: 298.0542, found: 298.0533.

((*S*)-3-(hex-1-yn-1-yldisulfanyl)-2-methylpropanoyl)-*L*-proline (3z)



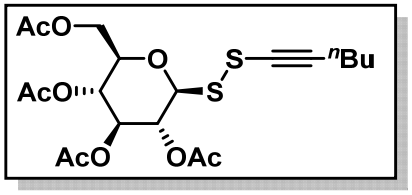
Yield: 46.4 mg (94%); time: 30 s; yellow liquid; TLC, R_f = 0.30 (MeOH:DCM = 1:9); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.80 (s, 1H), 4.60-4.58 (m, 1H), 3.71-3.65 (m, 2H), 3.29-3.16 (m, 2H), 2.88 (dd, *J* = 12.8, 4.8 Hz, 1H), 2.33 (t, *J* = 6.8 Hz, 3H), 2.16-2.01 (m, 3H), 1.53-1.46 (m, 2H), 1.43-1.34 (m, 2H), 1.28 (d, *J* = 6.8 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 175.5, 174.0, 97.5, 68.9, 59.5, 47.4, 41.2, 37.5, 30.5, 27.9, 24.8, 21.9, 19.9, 16.6, 13.5. HRMS (ESI) m/*z* calcd. for $C_{15}H_{23}NO_3S_2Na$ $[M+Na]^+$: 352.1012, found: 352.1012.

(*S*)-2-amino-3-methyl-3-(((triisopropylsilyl)ethynyl)disulfanyl)butanoic acid(3aa')



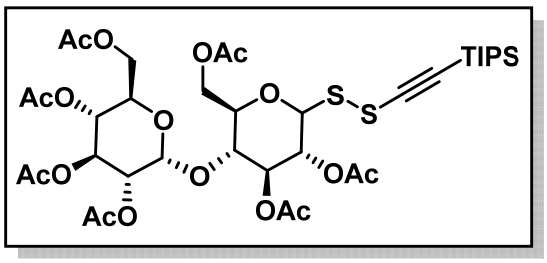
The product was purified by preparative HPLC. Yield: 40.0 mg (74%); time: 4 h; light yellow oil; TLC, R_f = 0.1 (MeOH:DCM = 1:10); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.49 (s, 2H), 4.12 (s, 1H), 1.76 (s, 3H), 1.45 (s, 3H), 1.06 (s, 21H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.3, 100.7, 95.2, 59.8, 52.8, 26.9, 21.8, 18.5, 11.3. HRMS (ESI) m/*z* calcd. for $C_{16}H_{31}NO_2S_2SiNa$ $[M+Na]^+$: 384.1458, found: 384.1464.

(2*R*,3*R*,4*S*,5*R*,6*S*)-2-(Acetoxymethyl)-6-(hex-1-yn-1-yldisulfanyl)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate (3ab')



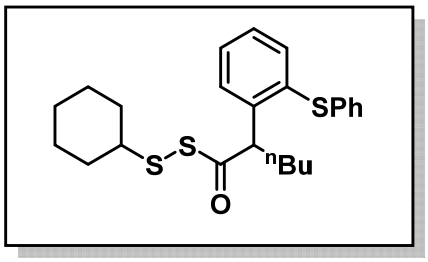
Yield: 47.8 mg (67%); time: 30 s; white solid; TLC, R_f =0.30 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.25 (t, J = 9.2 Hz, 1H), 5.19-5.11 (m, 2H), 4.72 (d, J = 9.6 Hz, 1H), 4.25 (dd, J = 12.4, 4.4 Hz, 1H), 4.15 (dd, J = 12.4, 2.4 Hz, 1H), 3.77 (dq, J = 10.0, 2.4 Hz, 1H), 2.31 (t, J = 6.8 Hz, 2H), 2.07 (s, 3H), 2.02 (d, J = 2.0 Hz, 5H), 2.00 (s, 3H), 1.51-1.44 (m, 2H), 1.42-1.33 (m, 2H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.6, 170.2, 169.3, 169.1, 97.7, 87.0, 76.2, 73.7, 69.5, 68.7, 67.9, 61.9, 30.3, 21.9, 20.7, 20.60, 20.57, 20.5, 19.8, 13.5. HRMS (ESI) m/z calcd. for $C_{20}H_{28}O_9S_2Na$ $[M+Na]^+$: 499.1067, found: 499.1074.

(2*R*,3*R*,4*S*,5*R*,6*R*)-2-(acetoxymethyl)-6-(((2*R*,3*R*,4*S*,5*R*)-4,5-diacetoxy-2-(acetoxymethyl)-6-(((triisopropylsilyl)ethynyl)disulfanyl)tetrahydro-2*H*-pyran-3-yl)oxy)tetrahydro-2H-pyran-3,4,5-triyl triacetate(3ac')



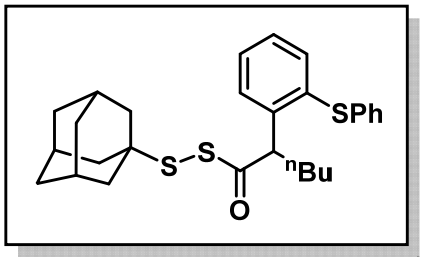
Yield: 58.3 mg (45%); time: 30 s; colorless liquid; TLC, R_f = 0.25 (PE:EtOAc = 7:3); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.40-5.31 (m, 3H), 5.05 (t, J = 9.6 Hz, 1H), 4.95 (t, J = 9.2 Hz, 1H), 4.87-4.82 (m, 2H), 4.41 (dd, J = 12.0, 2.4 Hz, 1H), 4.23 (dd, J = 12.4, 4.4 Hz, 2H), 4.05-3.93 (m, 3H), 3.79-3.75 (m, 1H), 2.14 (s, 3H), 2.09 (s, 3H), 2.04 (s, 3H), 2.02 (s, 3H) 1.99 (s, 6H), 1.06 (s, 21H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.6, 170.54, 170.49, 170.1, 169.9, 169.6, 169.4, 101.3, 95.7, 94.9, 86.5, 76.5, 76.1, 72.8, 70.6, 70.0, 69.3, 68.6, 67.9, 63.2, 61.5, 35.4, 29.7, 20.9, 20.8, 20.7, 20.62, 20.60, 18.6, 11.2. HRMS (ESI) m/z calcd. for $C_{37}H_{60}O_{17}S_2SiN$ $[M+NH_4]^+$: 882.3066, found: 882.3065.

Cyclohexyl 2-(2-(phenylthio)phenyl)hexane (dithioperoxoate) (4a)



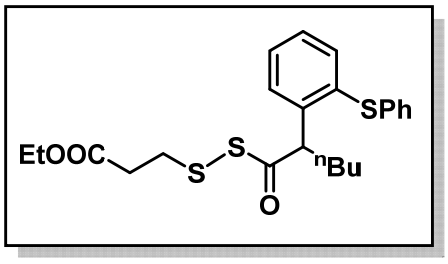
Yield: 52.0 mg (82%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.41 (t, J = 8.0 Hz, 2H), 7.35-7.27 (m, 2H), 7.26-7.22 (m, 3H), 7.21-7.17 (m, 2H), 4.75 (t, J = 8.0 Hz, 1H), 2.69-2.63 (m, 1H), 2.16-2.07 (m, 1H), 1.85-1.89 (m, 1H), 1.73-1.68 (m, 1H), 1.56-1.53 (m, 1H), 1.28-1.17 (m, 9H), 0.80 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.6, 140.1, 136.7, 134.9, 134.4, 131.0, 129.6, 129.1, 128.8, 128.5, 128.3, 127.0, 126.5, 55.3, 49.6, 33.6, 32.5, 29.5, 25.9, 25.4, 22.4, 13.8; HRMS (ESI) m/z calcd. for $C_{24}H_{30}NaOS_3$ $[M+Na]^+$: 453.1351, found: 453.1351.

(Adamantan-1-yl) 2-(2-(phenylthio)phenyl)hexane(dithioperoxoate) (4b)



Yield: 51.1 mg (71%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.425 (dd, J = 12.0, 8.0 Hz, 2H), 7.33-7.28 (m, 2H), 7.27-7.18 (m, 5H), 4.80 (t, J = 8.0 Hz, 1H), 2.16-2.10 (m, 1H), 2.00 (s, 3H), 1.72 (s, 7H), 1.66-1.54 (m, 7H), 1.29-1.12 (m, 4H), 0.81 (t, J = 8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 199.6, 140.2, 136.7, 134.8, 134.5, 129.6, 129.1, 128.7, 128.4, 128.2, 126.5, 55.3, 50.6, 42.2, 35.9, 33.5, 29.8, 29.5, 22.4, 13.8; HRMS (ESI) m/z calcd. for $C_{28}H_{34}NaOS_3$ $[M+Na]^+$: 505.1664, found: 505.1657.

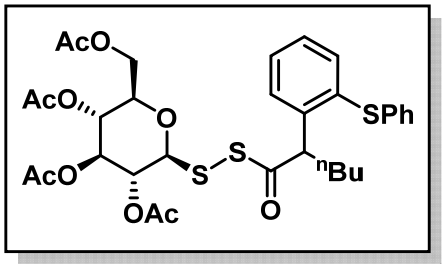
Ethyl 3-((2-(2-(phenylthio)phenyl)hexanoyl)disulfaneyl)propanoate (4c)



Yield: 17.3 mg (24%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44 (d, J = 8.0 Hz, 1H), 7.40 (d, J = 8.0 Hz, 1H), 7.33 (t, J = 8.0 Hz, 1H), 7.27-7.24 (m, 3H), 7.23-7.19 (m, 3H), 4.72 (t, J = 8.0 Hz, 1H), 4.16-

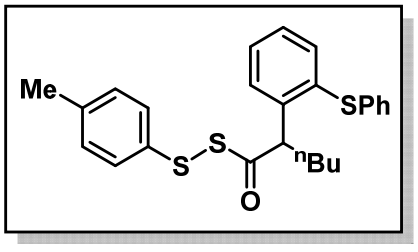
4.11 (m, 2H), 2.86-2.83 (m, 2H), 2.55-2.52 (m, 2H), 2.17-2.08 (m, 1H), 1.77-1.68 (m, 1H), 1.25 (t, J = 8.0 Hz, 7H), 0.82-0.79 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.6, 171.3, 139.8, 136.6, 135.0, 134.4, 129.5, 129.2, 128.9, 128.6, 128.4, 126.6, 60.8, 55.5, 33.9, 33.4, 29.7, 29.5, 22.4, 14.2, 13.8; HRMS (ESI) m/z calcd. for $C_{23}H_{28}NaO_3S_3$ $[M+Na]^+$: 471.1093, found: 471.1094.

2-(Acetoxymethyl)-6-((2-(2-(phenylthio)phenyl)hexanoyl)disulfaneyl)tetrahydro-2H-pyran-3,4,5-triyl triacetate (4d)



Yield: 41.5 mg (41%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37 (d, J = 8.0 Hz, 1H), 7.33-7.27 (m, 2H), 7.24-7.15 (m, 6H), 5.13-5.06 (m, 1H), 4.97-4.88 (m, 2H), 4.66-4.62 (m, 1H), 4.28 (dd, J = 28.0, 12.0 Hz, 1H), 4.17-4.04 (m, 1H), 3.97-3.93 (m, 1H), 3.58-3.47 (m, 1H), 2.05 (d, J = 4.0 Hz, 3H), 2.00 (d, J = 4.0 Hz, 3H), 1.94-1.93 (d, J = 4.0 Hz, 6H), 1.73-1.59 (m, 2H), 1.21-1.16 (m, 4H), 0.74 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.4, 170.6, 170.1, 169.5, 169.3, 139.5, 136.5, 134.9, 134.8, 129.7, 129.2, 128.8, 128.7, 128.6, 126.7, 87.2, 87.0, 76.1, 73.7, 69.6, 67.8, 61.9, 55.6, 33.2, 29.3, 26.9, 22.4, 20.7, 20.5, 13.8; HRMS (ESI) m/z calcd. for $C_{32}H_{38}NaO_{10}S_3$ $[M+Na]^+$:701.1519, found: 701.1518.

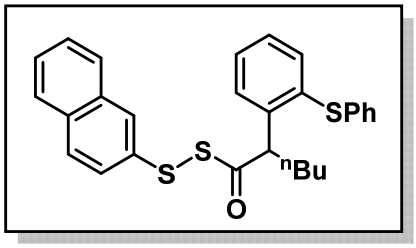
(p-Tolyl) 2-(2-(phenylthio)phenyl)hexane(dithioperoxoate) (4e)



Yield: 39.3 mg (60%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32 (m, 2H), 7.25-7.20 (m, 3H), 7.18-7.10 (m, 6H), 6.96 (d, J = 8.0 Hz, 2H), 4.65 (t, J = 4.0 Hz, 1H), 2.22 (s, 3H), 2.10-2.01 (m, 1H), 1.70-

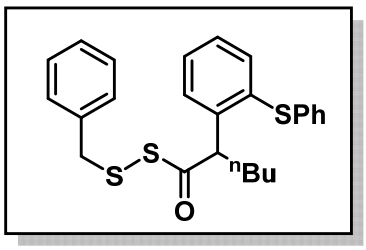
1.60 (m, 1H), 1.22-1.00 (m, 4H), 0.72 (t, J = 4.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.1, 139.7, 138.3, 136.5, 134.8, 134.7, 132.5, 130.7, 129.7, 129.7, 129.2, 128.7, 128.6, 128.4, 126.6, 55.3, 33.5, 29.5, 22.4, 21.1, 13.8; HRMS (ESI) m/z calcd. for $C_{25}H_{26}OS_3$ $[M+Na]^+$: 461.1038 , found: 461.1032 .

(Naphthalen-2-yl) 2-(2-(phenylthio)phenyl)hexane(dithioperoxoate) (4f)



Yield: 47.5 mg (67%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.88 (d, J = 2.0 Hz, 1H), 7.82 (t, J = 4.0 Hz, 3H), 7.76-7.71 (m, 2H), 7.52-7.44 (m, 6H), 7.36 (t, J = 8.0 Hz, 1H), 7.32-7.29 (m, 1H), 7.27-7.26 (m, 3H), 7.23-7.20 (m, 1H), 4.84 (t, J = 8.0 Hz, 1H), 2.25-2.16 (m, 1H), 1.84-1.74 (m, 1H), 1.34-1.23 (m, 4H), 0.84 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.7, 139.5, 136.4, 134.8, 134.7, 133.3, 133.0, 132.6, 129.7, 129.2, 128.8, 128.8, 128.7, 128.6, 128.5, 127.7, 127.5, 127.0, 126.6, 126.5, 55.4, 33.4, 29.5, 22.4, 13.8; HRMS (ESI) m/z calcd. for $C_{28}H_{26}OS_3$ $[M+H]^+$: 475.1219 , found: 475.1216.

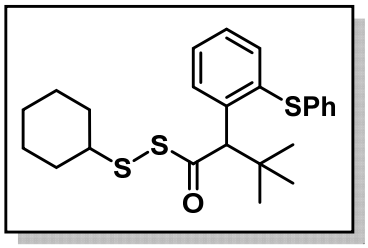
Benzyl 2-(2-(phenylthio)phenyl)hexane(dithioperoxoate) (4g)



Yield: 39.9 mg (61%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36 (d, J = 8.0 Hz, 1H), 7.31-7.23 (m, 2H), 7.19-7.14 (m, 6H), 7.12-7.07 (m, 5H), 4.63 (t, J = 8.0 Hz, 1H), 3.71 (dd, J = 17.2, 12.0 Hz, 2H), 2.06-1.92 (m, 1H), 1.66-1.57 (m, 1H), 1.21-1.16 (m, 4H), 0.74 (t, J = 4.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.4, 139.9, 136.6, 136.1, 134.9, 134.5, 129.6, 129.4,

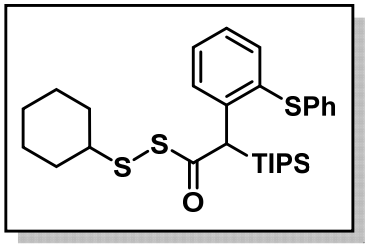
129.2, 128.8, 128.5, 128.4, 128.3, 127.6, 126.5, 55.3, 42.4, 33.5, 29.4, 22.4, 13.8; HRMS (ESI) m/z calcd. for $C_{25}H_{26}OS_3$ $[M+H]^+$: 439.1219 , found: 439.1226.

Cyclohexyl 3,3-dimethyl-2-(2-(phenylthio)phenyl)butane(dithioperoxoate) (4h)



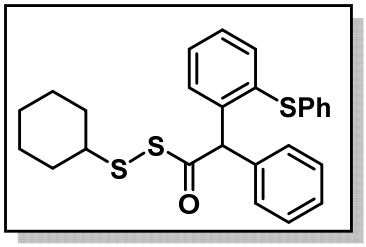
Yield: 45.6 mg (71%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.61 (d, *J* = 8.0 Hz, 1H), 7.40 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.27-7.26 (m, 5H), 7.24-7.22 (m, 1H), 7.21-7.20 (m, 1H), 4.95 (s, 1H), 2.71-2.64 (m, 2H), 1.90-1.84 (m, 2H), 1.29-1.17 (m, 6H), 1.08 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.4, 136.9, 136.7, 136.5, 134.7, 130.1, 130.1, 129.1, 128.2, 127.4, 126.6, 63.3, 49.5, 36.9, 32.4, 28.1, 25.9, 25.4; HRMS (ESI) m/z calcd. for $C_{24}H_{30}OS_3$ $[M+Na]^+$: 453.1351 , found: 453.1345.

Cyclohexyl 2-(2-(phenylthio)phenyl)-2-(triisopropylsilyl)ethane(dithioperoxoate) (4i)



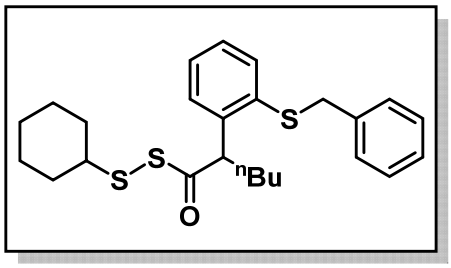
Yield: 31.6 mg (40%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.41 (d, *J* =8.0 Hz, 1H), 7.32-7.26 (m, 3H), 7.24-7.21 (m, 2H), 7.18-7.15 (m, 2H), 5.12 (s, 1H), 2.45 (m, 1H), 1.90-1.72 (m, 6H), 1.29-1.17 (m, 5H), 1.08 (m, 21 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.78, 138.24, 136.79, 135.07, 133.35, 131.68, 131.52, 130.99, 130.40, 129.60, 129.34, 129.15, 129.11, 128.27, 127.20, 126.40, 49.14, 48.65, 32.56, 32.50, 25.93, 25.46, 18.80, 18.70, 12.11.; HRMS (ESI) m/z calcd. for $C_{29}H_{42}OS_3Si$ $[M+Na]^+$: 553.2059 , found: 553.2047.

Cyclohexyl 2-phenyl-2-(2-(phenylthio)phenyl)ethane(dithioperoxoate) (4j)



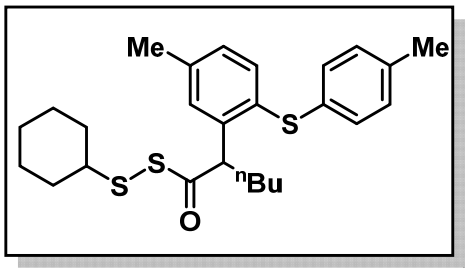
Yield: 45.1 mg (67%); time: 20 h; yellow oil; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.43 (dd, J = 7.6, 1.6 Hz, 1H), 7.34-7.27 (m, 6 H), 7.24-7.21 (m, 4H), 7.20-7.15 (m, 3H), 6.11 (s, 1H), 2.71-2.64 (m, 1H), 1.93-1.89 (m, 2H), 1.74-1.70 (m, 2H), 1.29-1.22 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.3, 163.3, 140.0, 135.0, 130.1, 129.4 129.2, 128.8, 128.7, 128.6, 127.6, 126.6, 119.6, 113.1, 60.6, 49.7, 32.6(32.5), 29.7, 25.9 (25.5); HRMS (ESI) m/z calcd. for $C_{26}H_{26}NaOS_3$ $[M+Na]^+$: 473.1038, found: 473.1030.

Cyclohexyl 2-(2-(benzylthio)phenyl)hexane(dithioperoxoate) (4k)



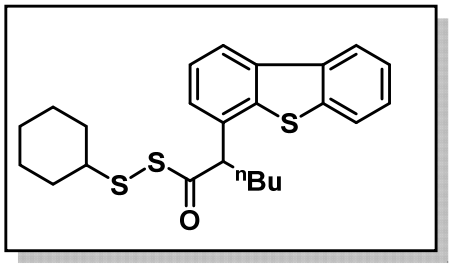
Yield: 51.3 mg (77%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39 (dd, J = 7.6, 1.6 Hz, 1H), 7.31-7.23 (m, 6 H), 7.22-7.18 (m, 2H), 4.76 (t, J = 7.2 Hz, 3H), 4.11 (s, 2H), 2.71-2.65 (m, 1H), 2.15-2.06 (m, 1H), 1.92-1.86 (m, 2H), 1.74-1.70 (m, 2H), 1.63-1.54 (m, 2H), 1.32-1.12 (m, 9H), 0.85 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.5, 139.3, 137.2, 135.6, 132.6, 128.9, 128.5, 128.0, 127.8, 127.6, 127.3, 55.2, 49.5, 40.5, 33.5, 32.4, 29.5, 25.9, 25.4, 22.5, 13.9; HRMS (ESI) m/z calcd. for $C_{25}H_{32}OS_3$ $[M+Na]^+$: 467.1507 , found: 467.1500.

Cyclohexyl 2-(5-methyl-2-(p-tolylthio)phenyl)hexane(dithioperoxoate) (4l)



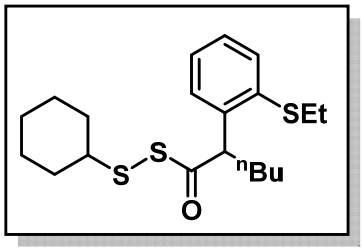
Yield: 44.5 mg (65%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.31 (dd, J = 8.0 Hz, 1H), 7.17 (s, 1 H), 7.14-7.11 (m, 2H), 7.07-7.05 (m, 2H), 7.04-7.01 (m, 1H), 4.74 (t, J = 4.0 Hz, 1H), 2.71-2.63 (m, 1H), 2.31 (d, J = 8.0 Hz, 6H), 2.14-2.05 (m, 1H), 1.92-1.86 (m, 1H), 1.74-1.56 (m, 4H), 1.26-1.23 (m, 9H), 0.81 (t, J = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.7, 139.9, 138.8, 136.4, 134.8, 133.5, 131.4, 129.9, 129.7, 129.1, 128.9, 55.2, 49.5, 33.7, 32.5, 29.6, 26.0, 25.4, 22.5, 21.2, 21.0, 13.8; HRMS (ESI) m/z calcd. for $C_{26}H_{34}OS_3$ $[M+Na]^+$: 481.1664 , found: 481.1655.

Cyclohexyl 2-(dibenzo[b,d]thiophen-4-yl)hexane(dithioperoxoate) (4m)



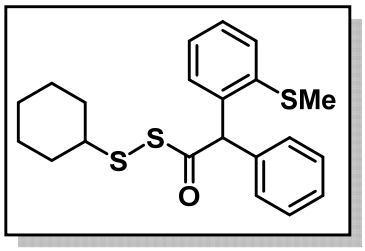
Yield: 48.7 mg (76%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.17-8.15 (m, 1H), 8.10 (dd, J = 7.6, 1.6 Hz, 1H), 7.90-7.87 (m, 1H), 7.50-7.45 (m, 4H), 4.24 (t, J = 4.0 Hz, 1H), 2.68-2.61 (m, 1H), 2.33-2.23 (m, 1H), 2.10-2.01 (m, 1H), 1.89-1.80 (m, 2H), 1.72-1.64 (m, 2H), 1.57-1.49 (m, 3H), 1.43-1.30 (m, 3H), 1.28-1.08 (m, 4H), 0.86 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.9, 139.9, 138.8, 136.1, 136.0, 132.3, 127.0, 125.5, 125.2, 124.6, 122.8, 121.8, 120.9, 57.9, 49.6, 32.6, 32.4, 29.5, 25.8, 25.4, 22.5, 13.8; HRMS (ESI) m/z calcd. for $C_{24}H_{28}OS_3$ $[M+Na]^+$: 451.1194 , found: 451.1193.

Cyclohexyl 2-(2-(ethylthio)phenyl)hexane(dithioperoxoate) (4n)



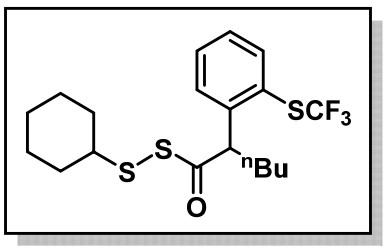
Yield: 26 mg (44%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.43-7.41 (m, 1H), 7.32-7.29 (m, 1H), 7.25-7.17 (m, 2H), 4.74 (t, J = 8.0 Hz, 1H), 2.96 (q, J = 8.0 Hz, 2H), 2.79-2.65 (m, 2H), 2.20-2.10 (m, 1H), 2.00-1.96 (m, 1H), 1.92-1.87 (m, 2H), 1.82-1.66 (m, 5H), 1.34-1.30 (m, 3H), 1.27-1.19 (m, 6H), 0.86 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.6, 138.4, 136.3, 130.7, 128.0, 127.9, 126.8, 55.0, 49.5, 33.4, 32.4, 29.5, 29.0, 25.9, 25.5, 22.5, 14.4, 13.9; HRMS (ESI) m/z calcd. for $C_{20}H_{30}OS_3$ $[M+Na]^+$: 383.1532 , found: 383.1526.

Cyclohexyl 2-(2-(methylthio)phenyl)-2-phenylethane(dithioperoxoate) (4o)



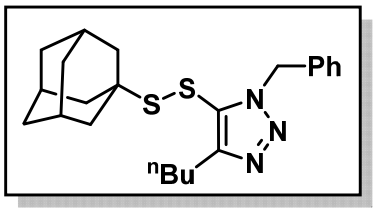
Yield: 31.4 mg (54%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37-7.27 (m, 7H), 7.24-7.21 (m, 1H), 7.17-7.13 (m, 1H), 5.99 (s, 1H), 2.74-2.67 (m, 1H), 2.45 (s, 3H), 1.97-1.92 (m, 2H), 1.77-1.72 (m, 2H), 1.35-1.14 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.5, 138.0, 136.8, 136.5, 129.3, 129.1, 128.7, 128.3, 127.8, 127.7, 125.7, 60.1, 49.6, 32.5, 25.9, 25.5, 17.1; HRMS (ESI) m/z calcd. for $C_{21}H_{24}OS_3$ $[M+Na]^+$: 411.0881 , found: 411.0877

Cyclohexyl 2-(2-((trifluoromethyl)thio)phenyl)hexane(dithioperoxoate) (4p)



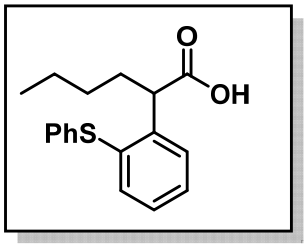
Yield: 25.2 mg (40%); time: 20 h; yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 49:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.75 (d, 1H), 7.53-7.47 (m, 2H), 7.36-7.31 (m, 1H), 4.86 (t, J = 8.0 Hz, 1H), 2.72-2.65 (m, 1H), 2.25-2.16 (m, 1H), 2.02-1.83 (m, 4H), 1.79-1.70 (m, 2H), 1.25-1.20 (m, 9H), 0.87 (t, J = 4.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.2, 143.3, 138.2, 131.9, 129.3(q, J = 309 Hz), 128.6 (d, J = 58 Hz), 124.28, 55.5, 49.6, 42.4, 33.9, 32.5, 31.1, 29.7, 25.9, 25.2, 22.5, 13.8; ^{19}F (376 MHz, $CDCl_3$) δ -42.1. HRMS (ESI) m/z calcd. for $C_{19}H_{26}F_3OS_3$ $[M+H]^+$: 423.1092 , found: 423.1094.

5-(((3*s*,5*s*,7*s*)-Adamantan-1-yl)disulfanyl)-1-benzyl-4-butyl-1*H*-1,2,3-triazole (6)



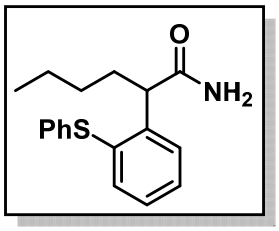
Yield: 25.4 mg (70%); time: 12 h; light yellow solid; TLC, R_f = 0.30 (PE:EtOAc = 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.26 (m, 5H), 5.67 (s, 2H), 2.78 (t, J = 7.6 Hz, 2H), 2.05 (s, 3H), 1.76-1.59 (m, 14H), 1.40 (q, J = 7.6 Hz, 2H), 0.94 (t, J = 7,2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 150.6, 135.0, 128.7, 128.1, 128.0, 127.9, 52.4, 50.8, 42.4, 35.9, 31.1, 29.7, 25.6, 22.6, 13.9. HRMS (ESI) m/z calcd. for $C_{23}H_{32}N_3S_2$ $[M+H]^+$: 414.2032, found: 414.2049.

2-(2-(Phenylthio)phenyl)hexanoic acid (7)



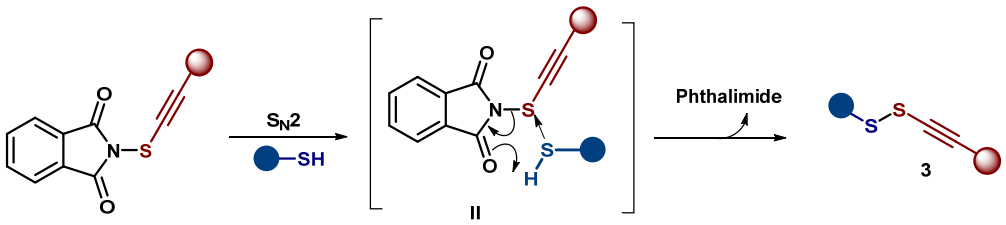
Yield: 34.5 mg (77%); time: 2 h; light yellow liquid; TLC, R_f =0.20 (PE:EtOAc = 3:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45-7.41 (m, 2H), 7.32 (t, J = 8.0 Hz, 1H), 7.26-7.15 (m, 6H), 4.41 (t, J = 8.0 Hz, 1H), 2.08-2.00 (m, 1H), 1.74-1.64 (m, 1H), 1.31-1.08 (m, 5H), 0.81 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 179.9, 141.2, 136.8, 134.8, 134.1, 129.4, 129.0, 128.7, 128.1, 128.0, 126.4, 47.7, 32.9, 29.6, 22.4, 13.8; HRMS (ESI) m/z calcd. for $C_{18}H_{19}O_2S$ $[M-H]^+$: 299.1111 , found: 299.1113.

2-(2-(Phenylthio)phenyl)hexanamide (8)



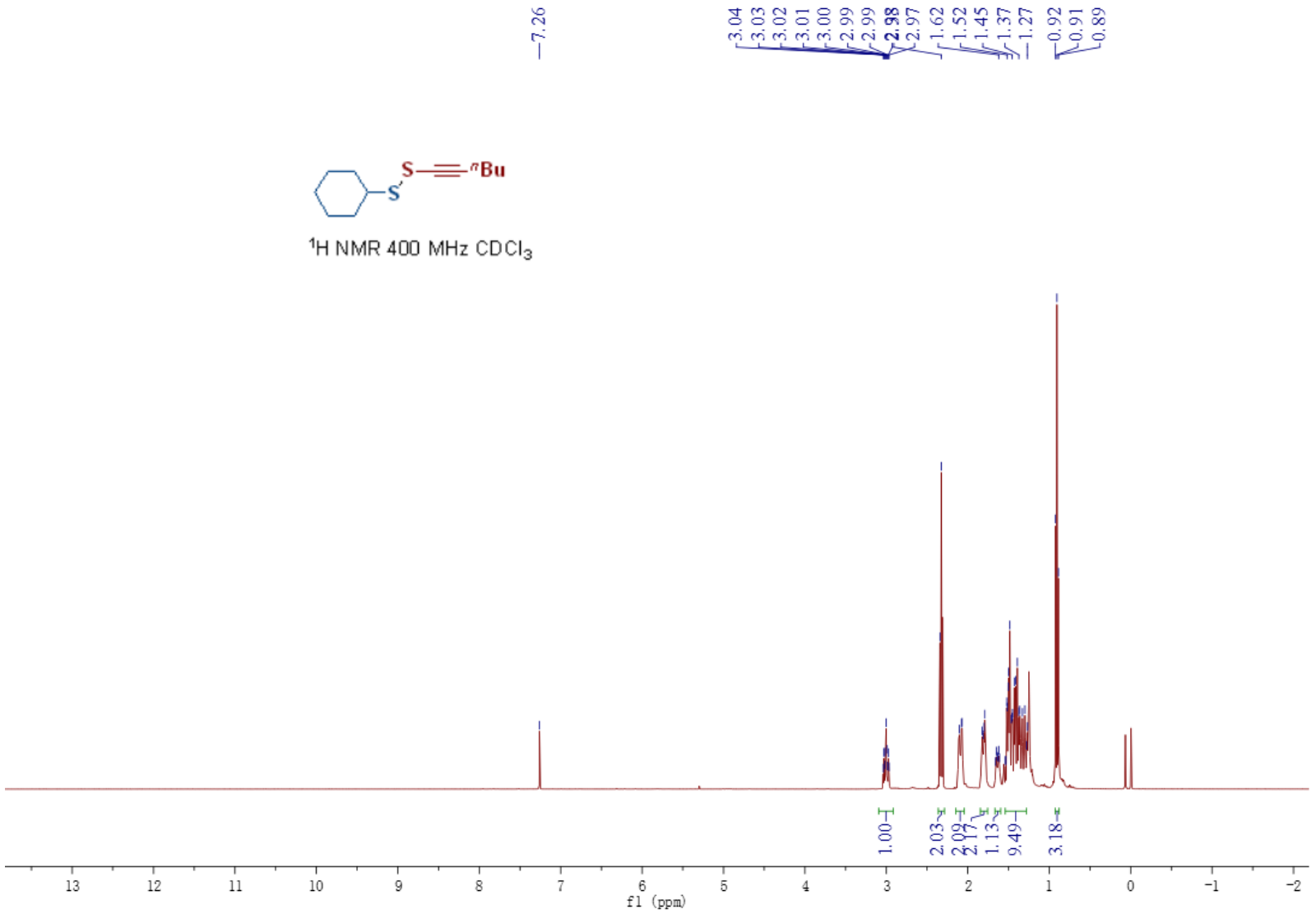
Yield: 33.1 mg (74%); time: 2 h; light yellow solid; TLC, R_f =0.20 (PE:EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.49 (d, J = 8.0 Hz, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.29 (t, J = 8.0 Hz, 1H), 7.22-7.09 (m, 6H), 5.29 (d, J = 76.0 Hz, 2H), 4.06 (t, J = 8.0 Hz, 1H), 2.07-1.98 (m, 1H), 1.62-1.53 (m, 1H), 1.20-1.12 (m, 3H), 1.06-0.94 (m, 1H), 0.74 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 175.5, 142.7, 137.0, 135.1, 132.7, 129.4, 129.3, 129.0, 128.3, 127.9, 126.5, 48.1, 32.8, 29.7, 22.5, 13.9; HRMS (ESI) m/z calcd. for $C_{18}H_{21}NOS$ $[M+Na]^+$: 322.1236 , found: 322.1234.

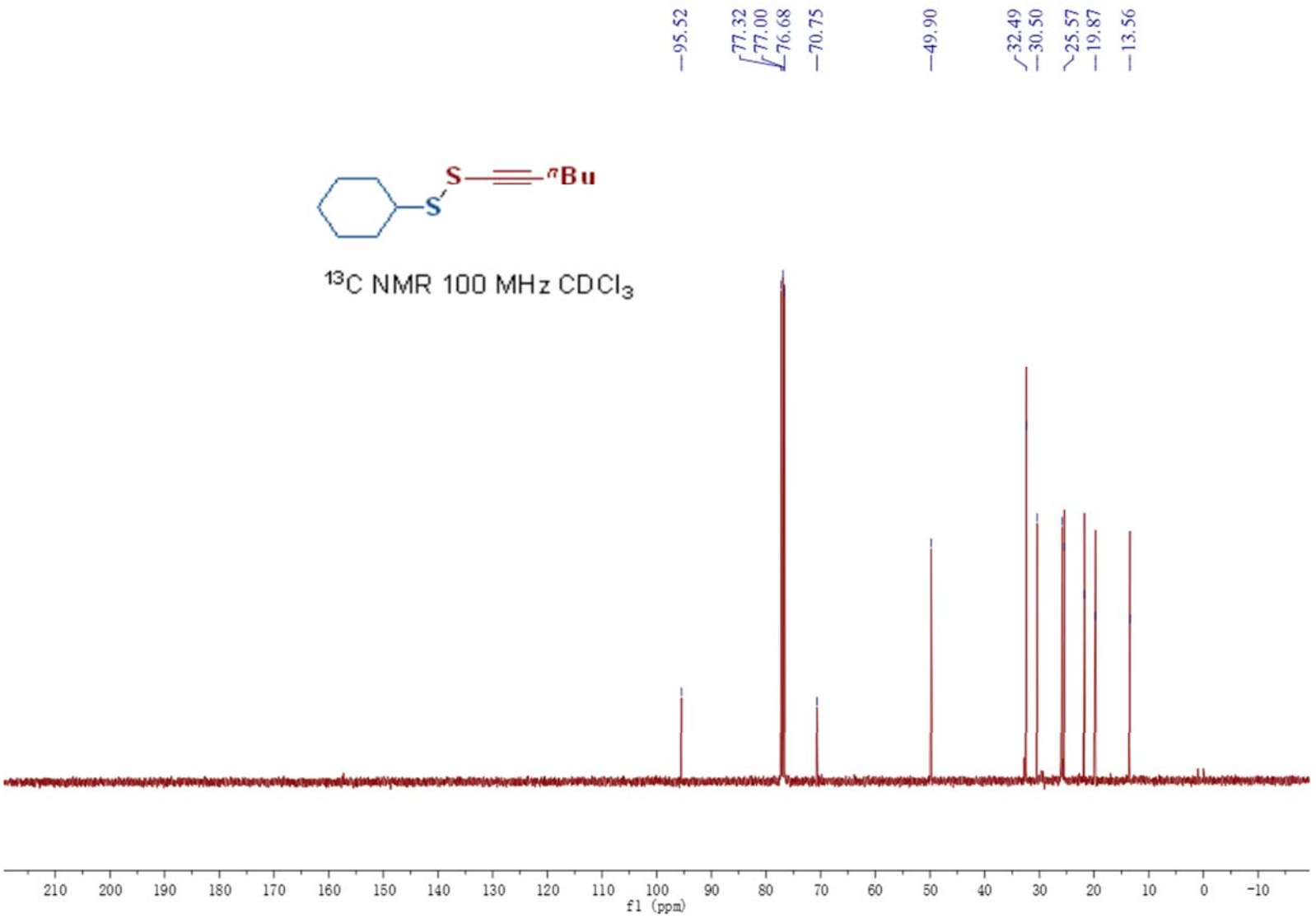
5. Possible mechanism for S-S bond formation



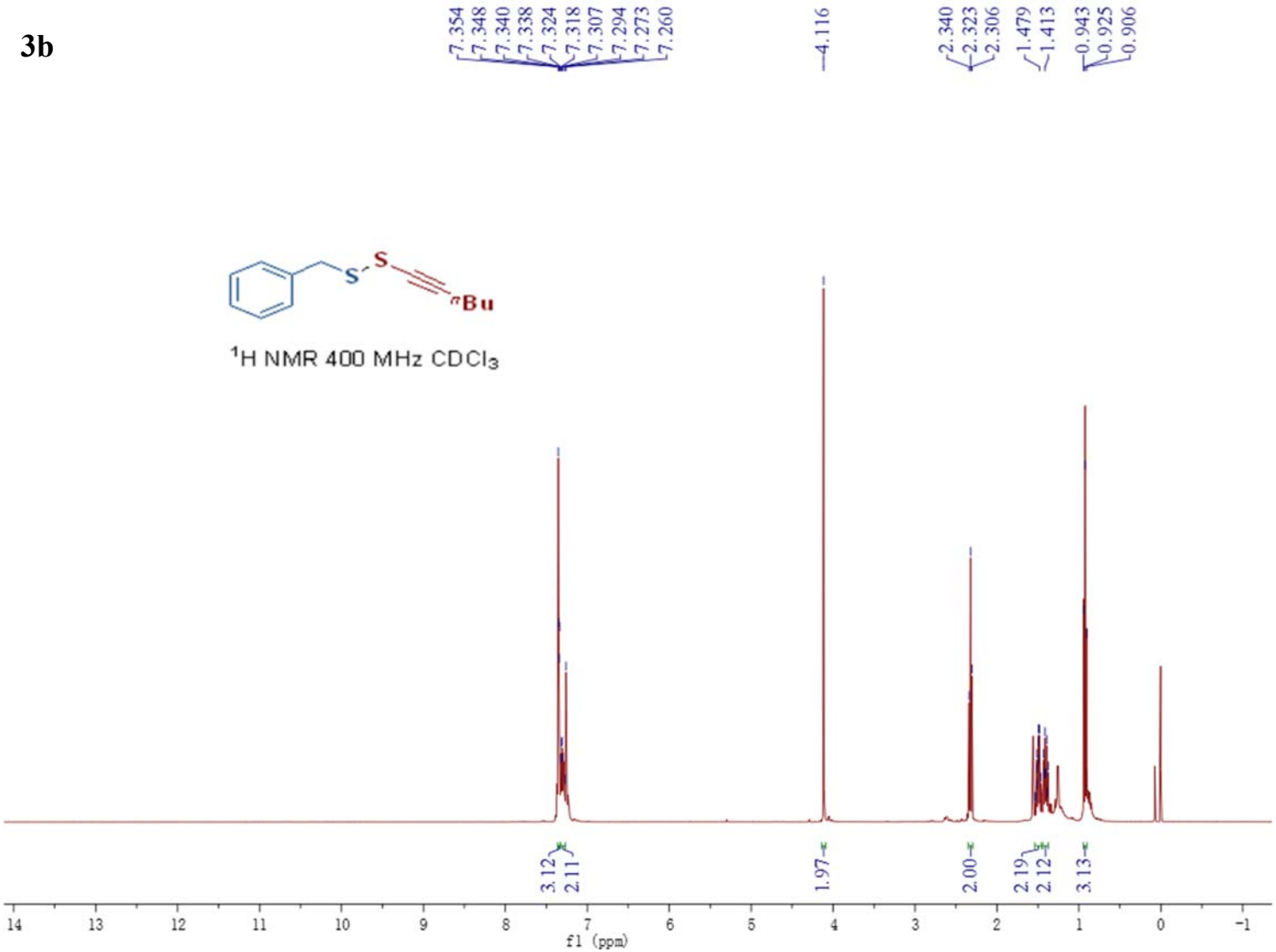
6. Copies of NMR spectra

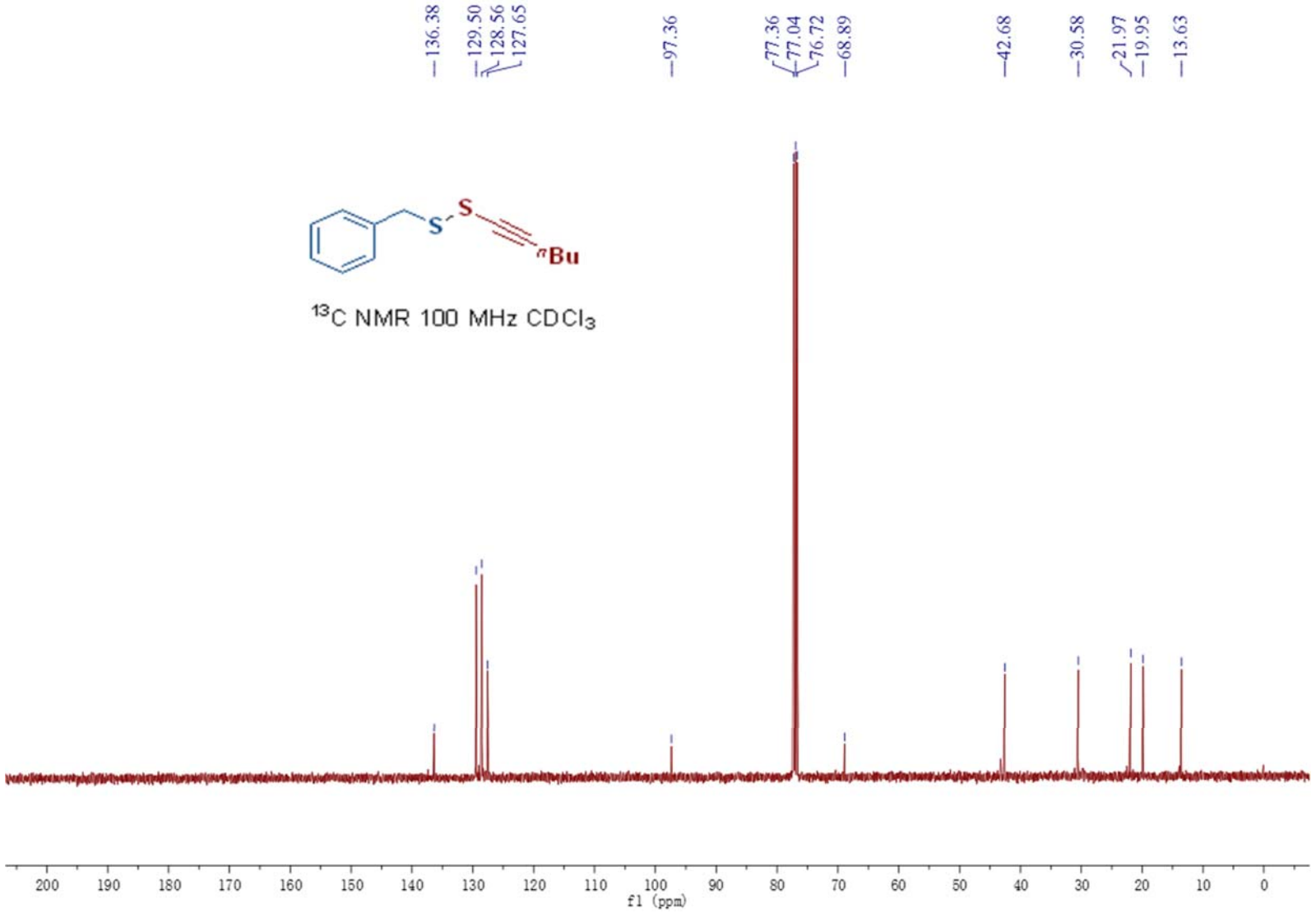
3a


7.26
3.04
3.03
3.02
3.01
3.00
2.99
2.99
2.98
2.97
1.62
1.52
1.45
1.37
1.27
0.92
0.91
0.89
S
nBu
S
1H NMR 400 MHz CDCl3
1.00
2.03
2.02
2.17
1.13
9.49
3.18
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

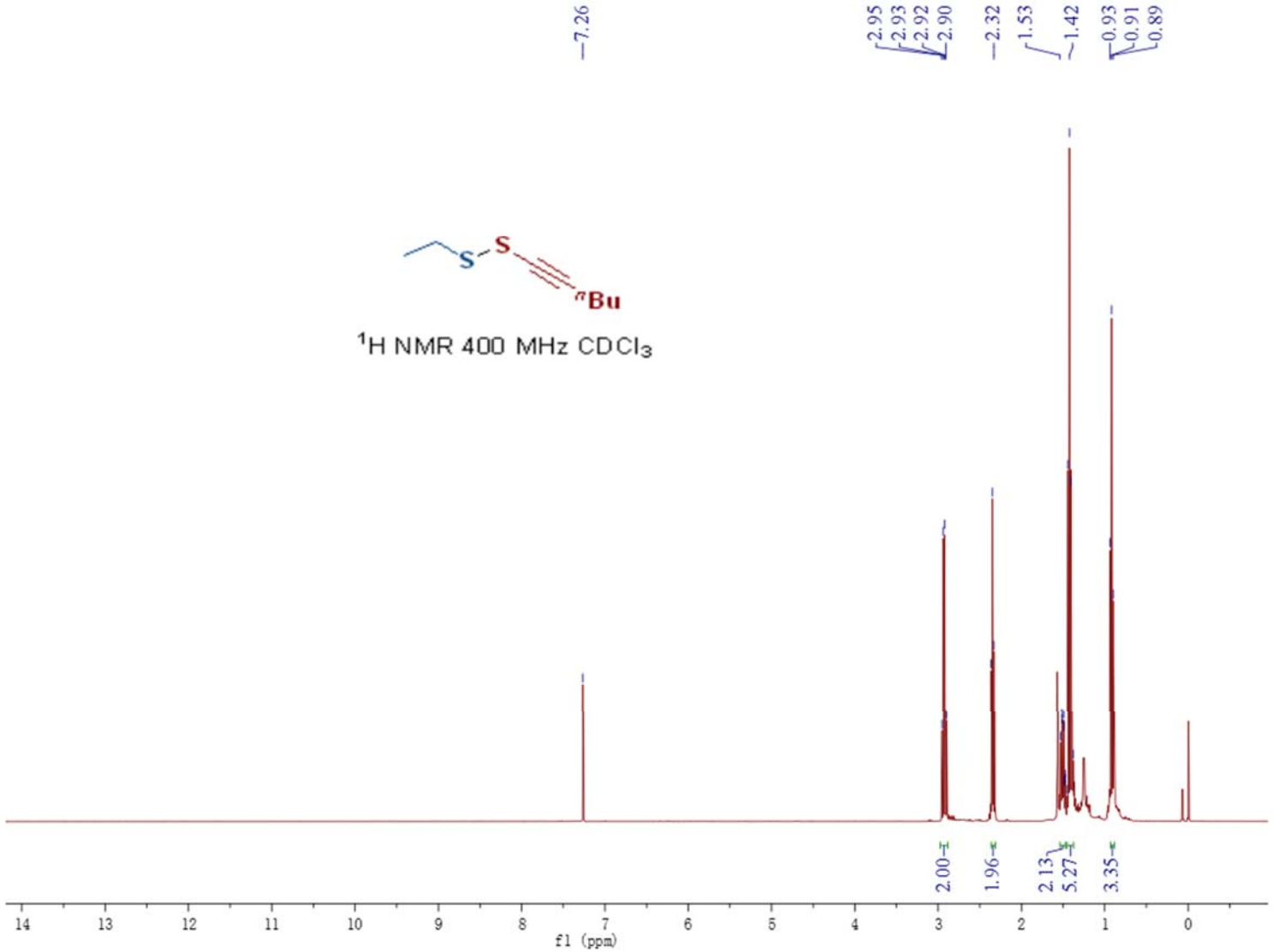

95.52
77.32
77.00
76.68
70.75
49.90
32.49
30.50
25.57
19.87
13.56
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

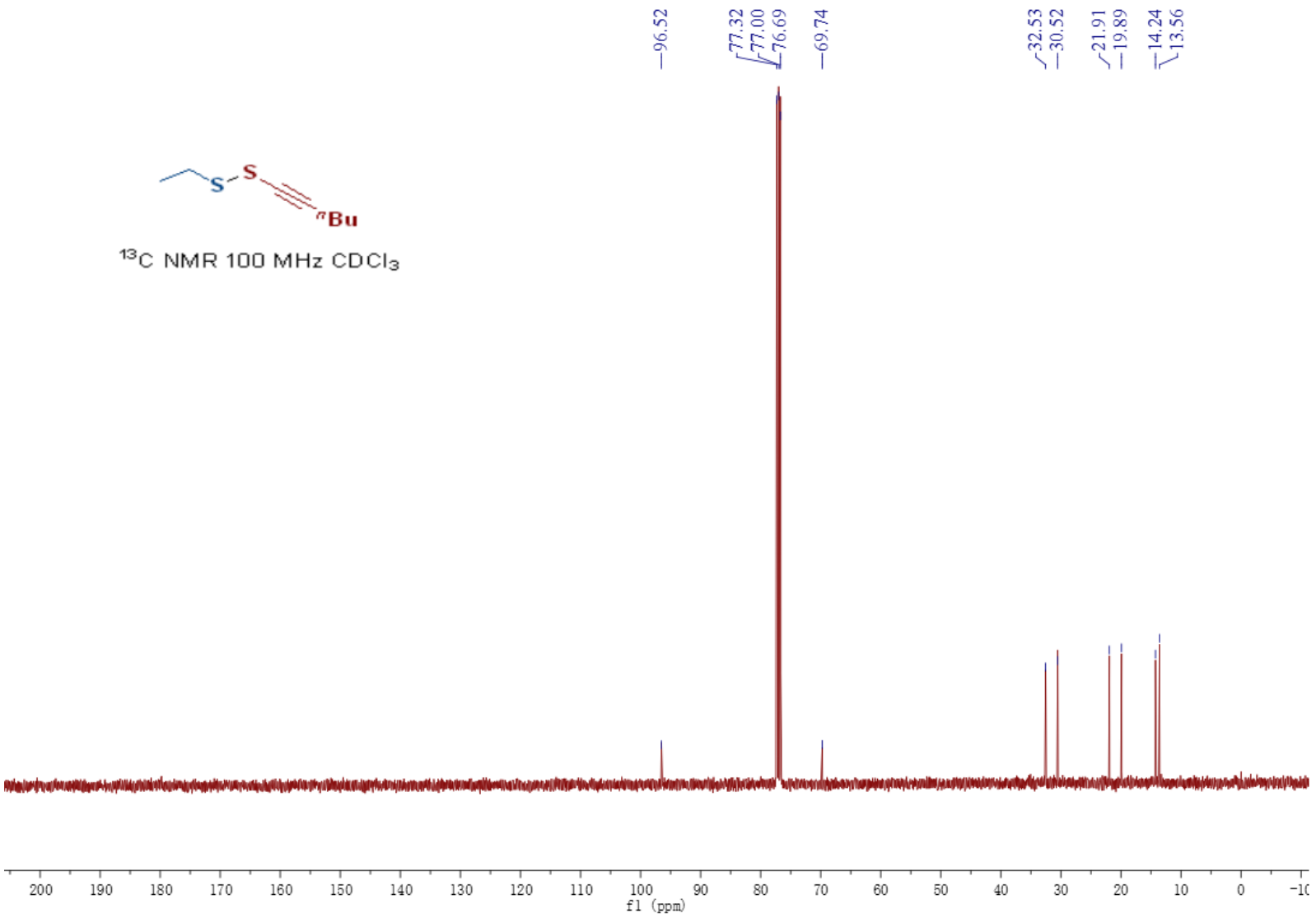
3b


7.354
7.348
7.340
7.338
7.324
7.318
7.307
7.294
7.273
7.260
4.116
2.340
2.323
2.306
1.479
1.413
0.943
0.925
0.906
S
S
nBu
1H NMR 400 MHz CDCl3
3.12
2.11
1.97
2.00
2.19
2.12
3.13
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

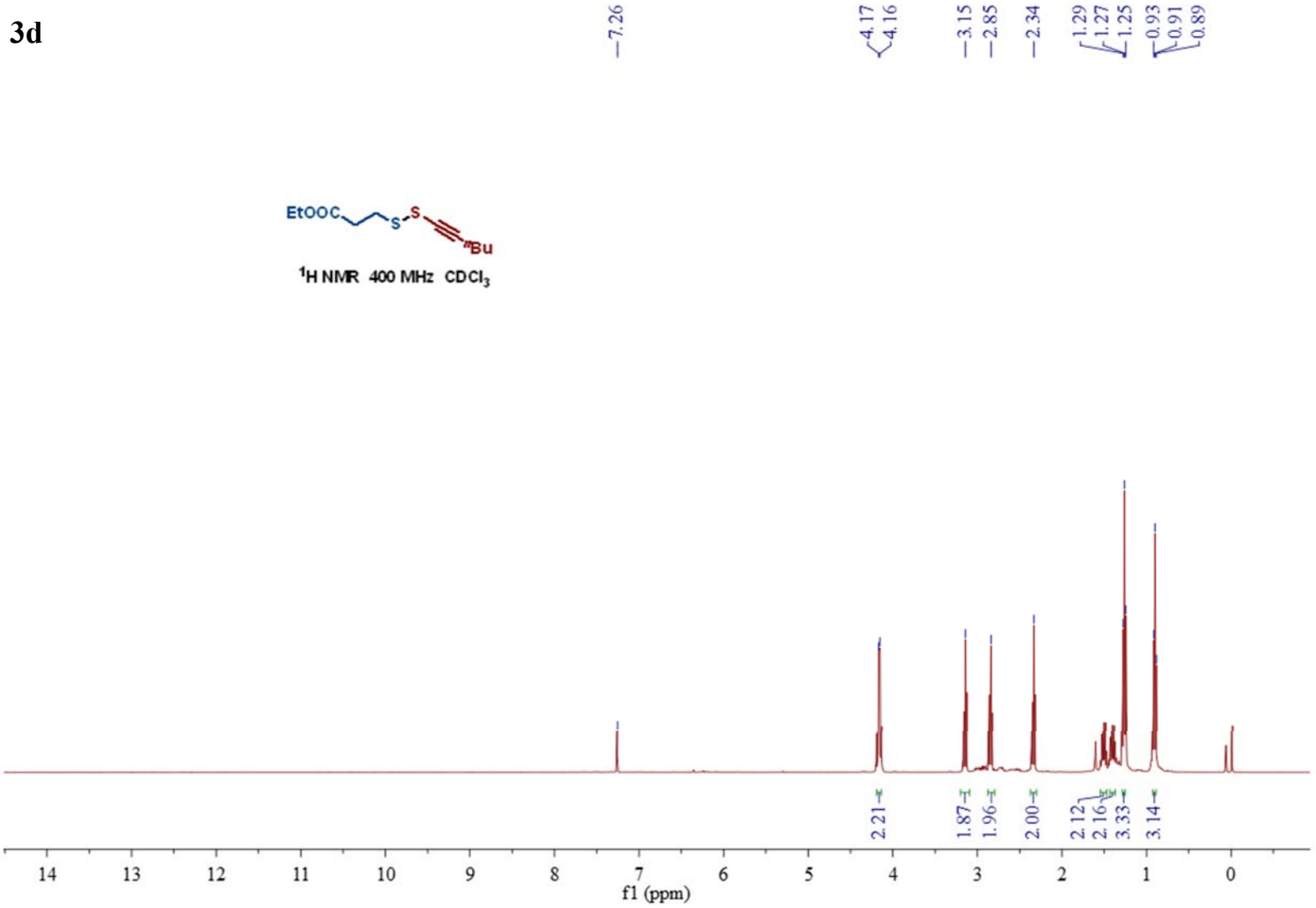

136.38
129.50
128.56
127.65
97.36
77.36
77.04
76.72
68.89
42.68
30.58
21.97
19.95
13.63
S
S
nBu
13C NMR 100 MHz CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

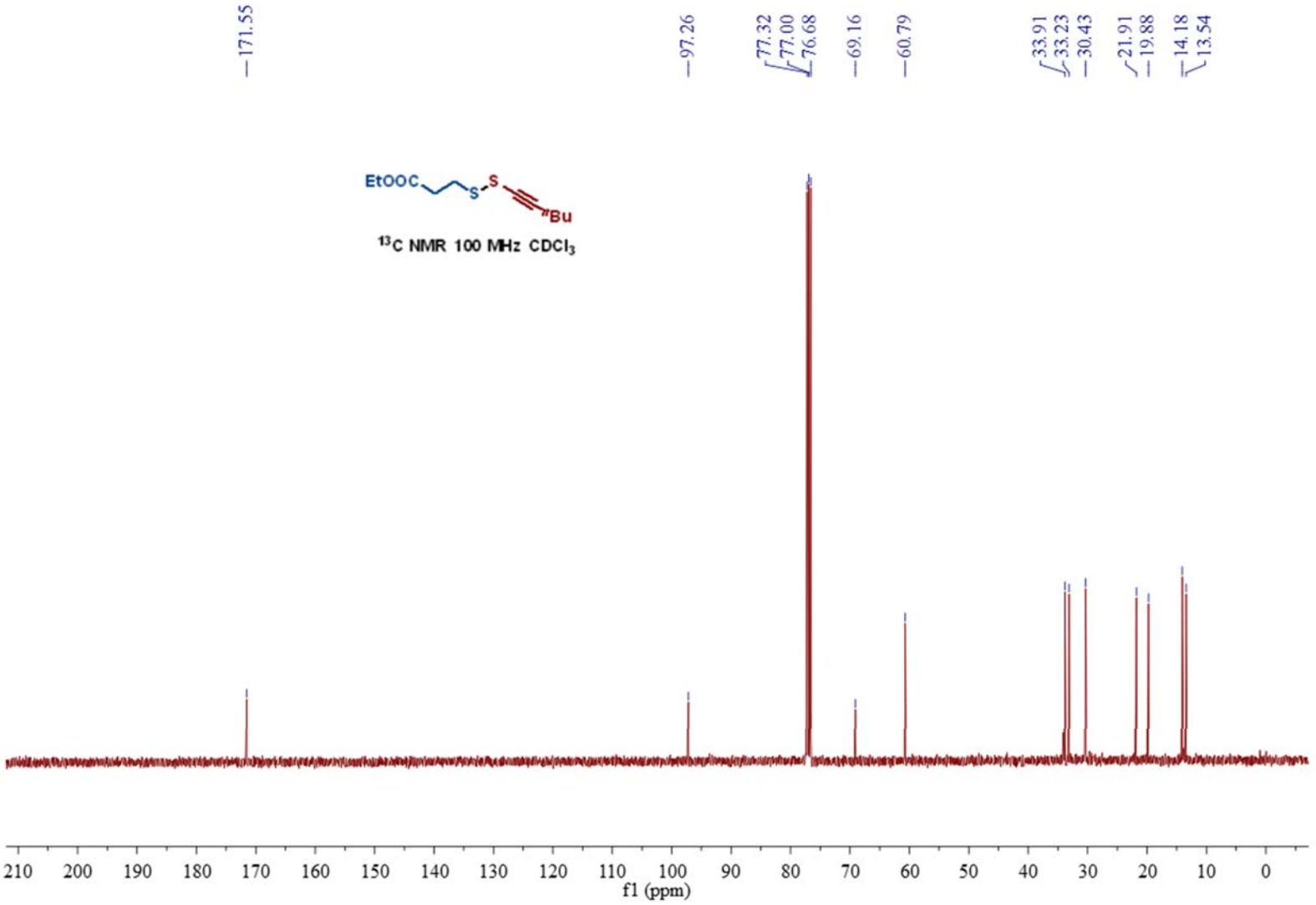
3c


7.26
2.95
2.93
2.92
2.90
2.32
1.53
1.42
0.93
0.91
0.89
S
S
nBu
1H NMR 400 MHz CDCl3
2.00
1.96
2.13
5.27
3.35
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

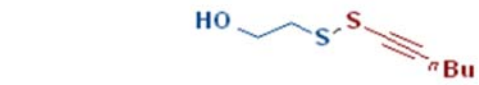

96.52
77.32
77.00
76.69
69.74
32.53
30.52
21.91
19.89
14.24
13.56
S
S
nBu
13C NMR 100 MHz CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3d

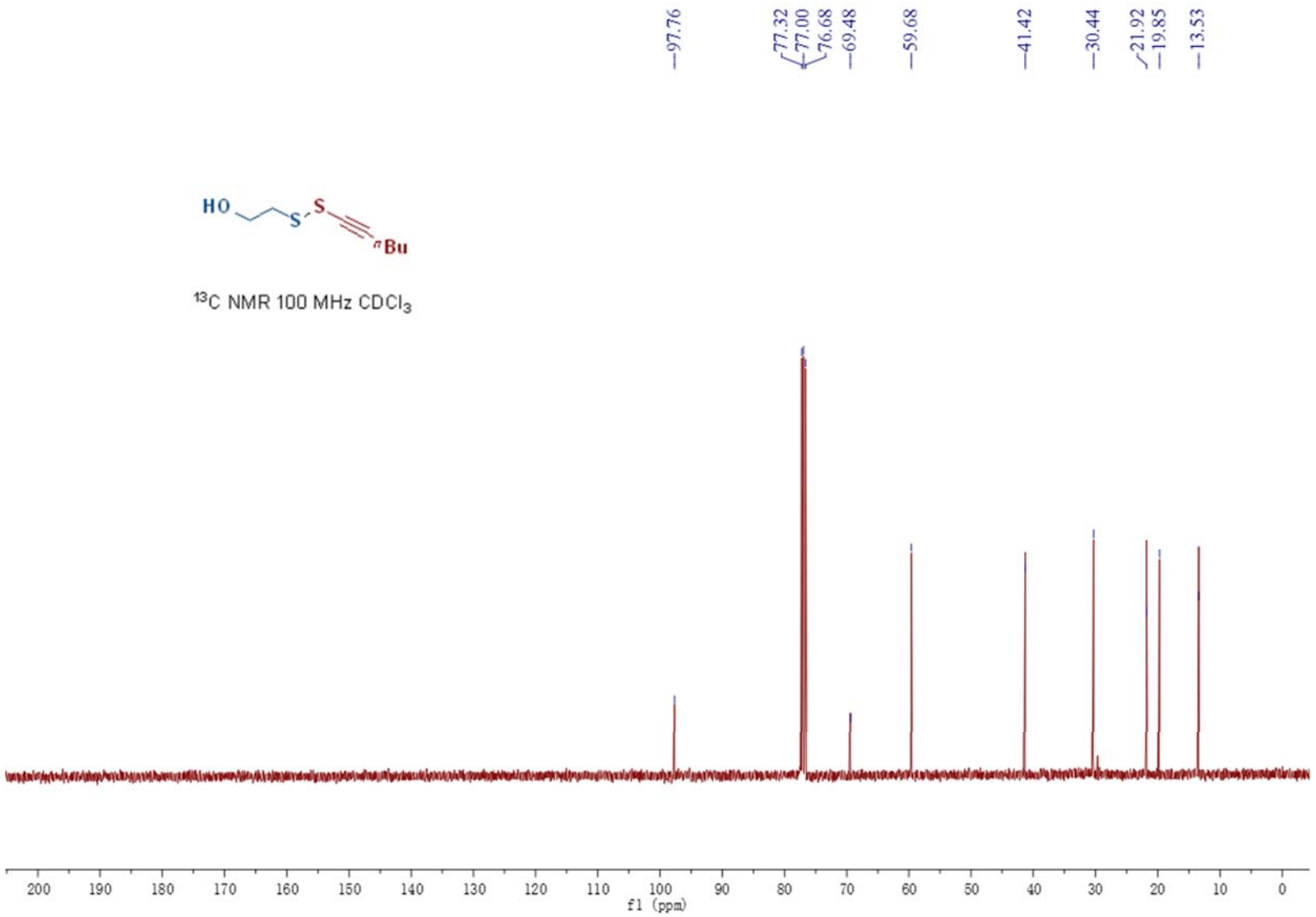

7.26
4.17
4.16
3.15
2.85
2.34
1.29
1.27
1.25
0.93
0.91
0.89
EtOOC
S
S
nBu
1H NMR 400 MHz CDCl3
2.21
1.87
1.96
2.00
2.12
2.16
3.33
3.14
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


171.55
97.26
77.32
77.00
76.68
69.16
60.79
33.91
33.23
30.43
21.91
19.88
14.18
13.54
EtOOC
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

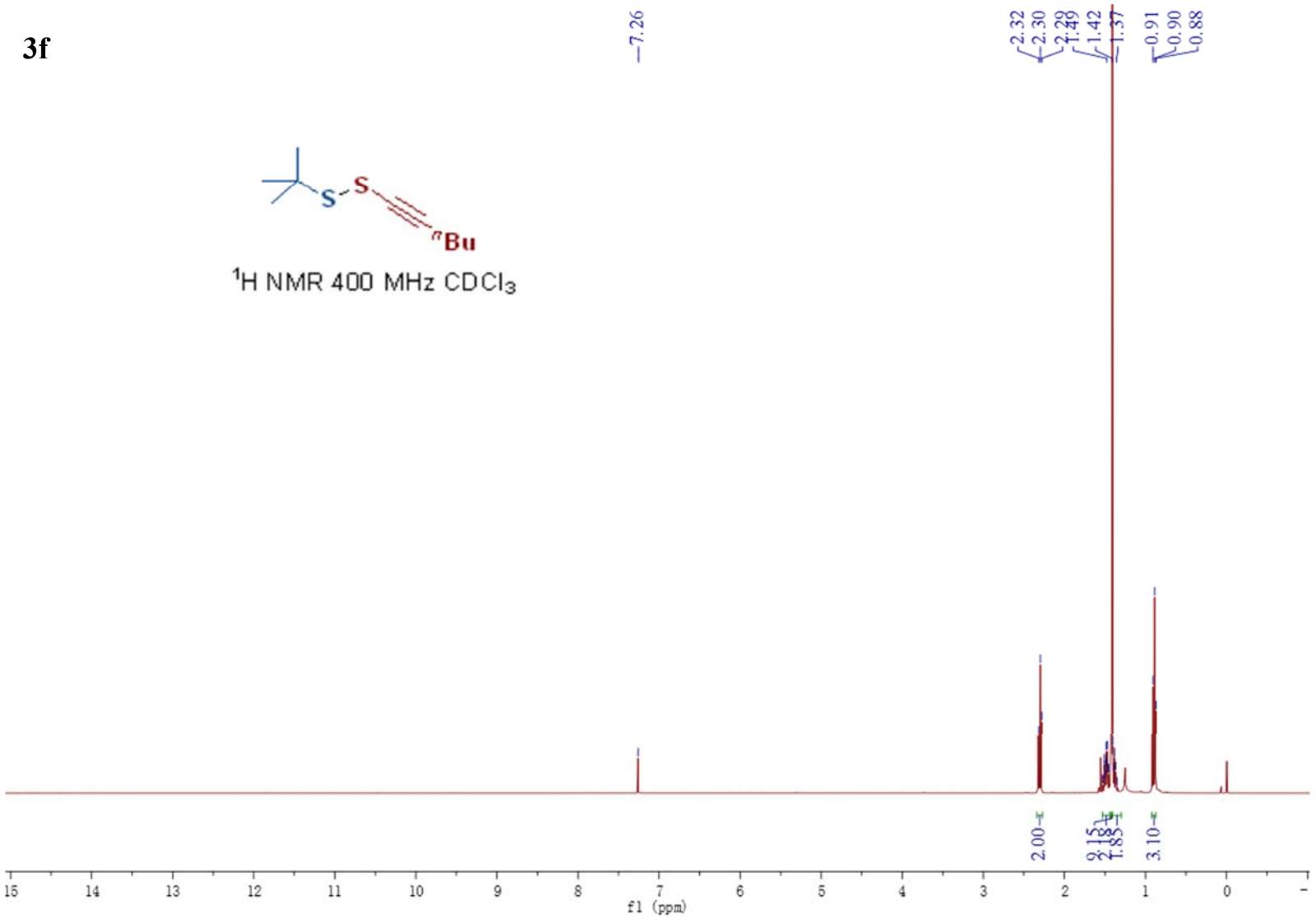
3e

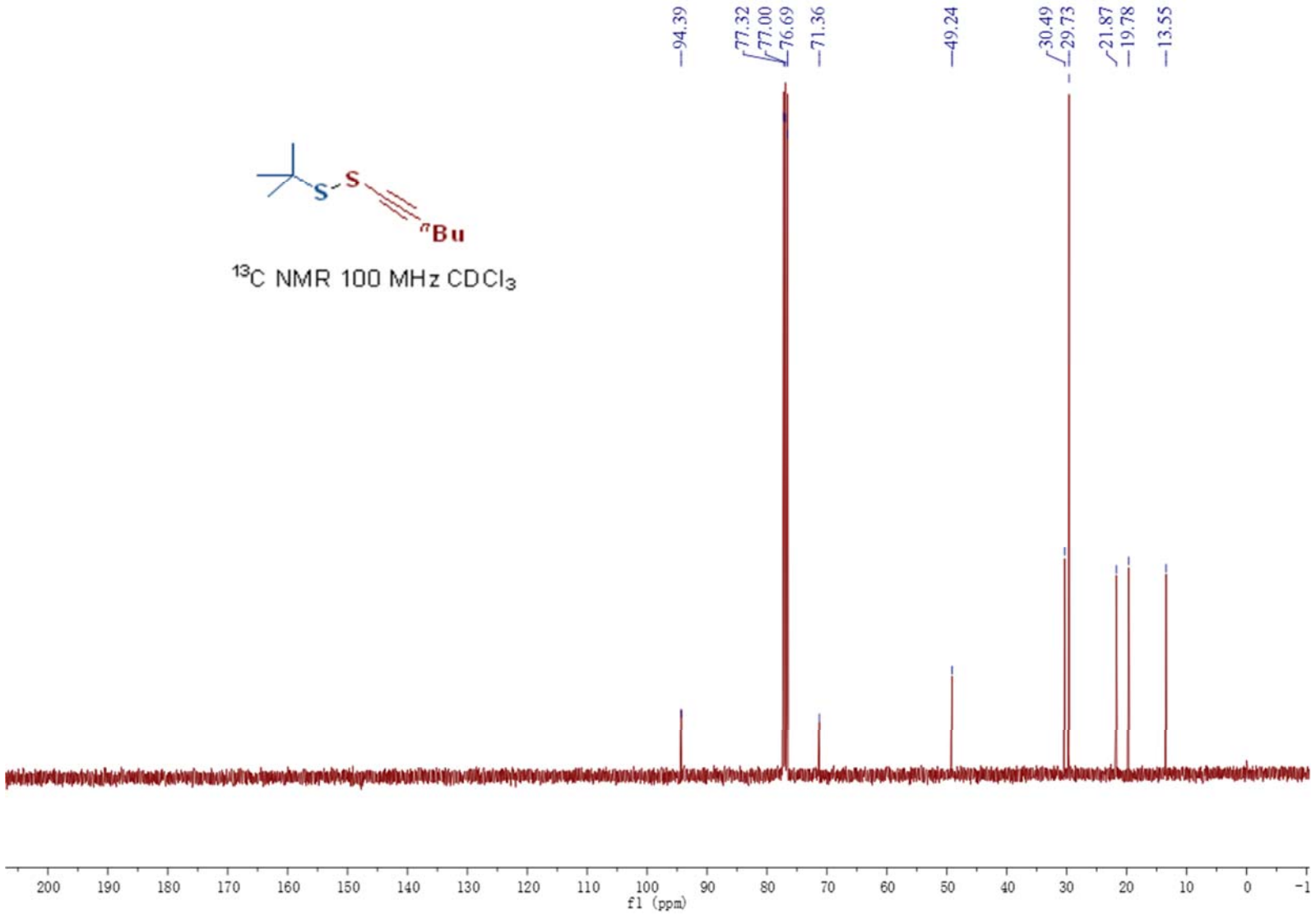


^{1}H NMR 400 MHz $CDCl_3$

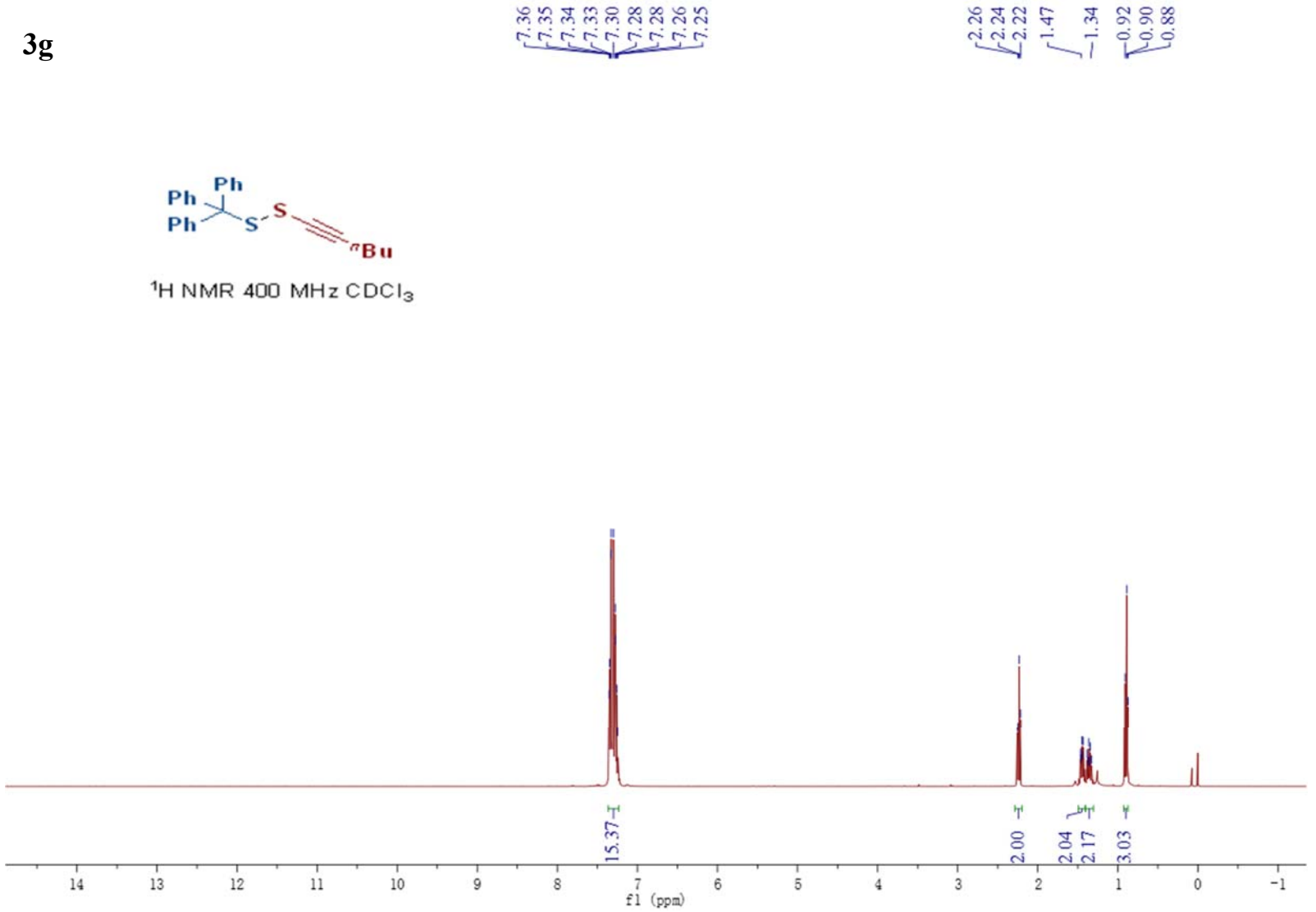

97.76
77.32
77.00
76.68
69.48
59.68
41.42
30.44
21.92
19.85
13.53
HO
S
S
nBu
13C NMR 100 MHz CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

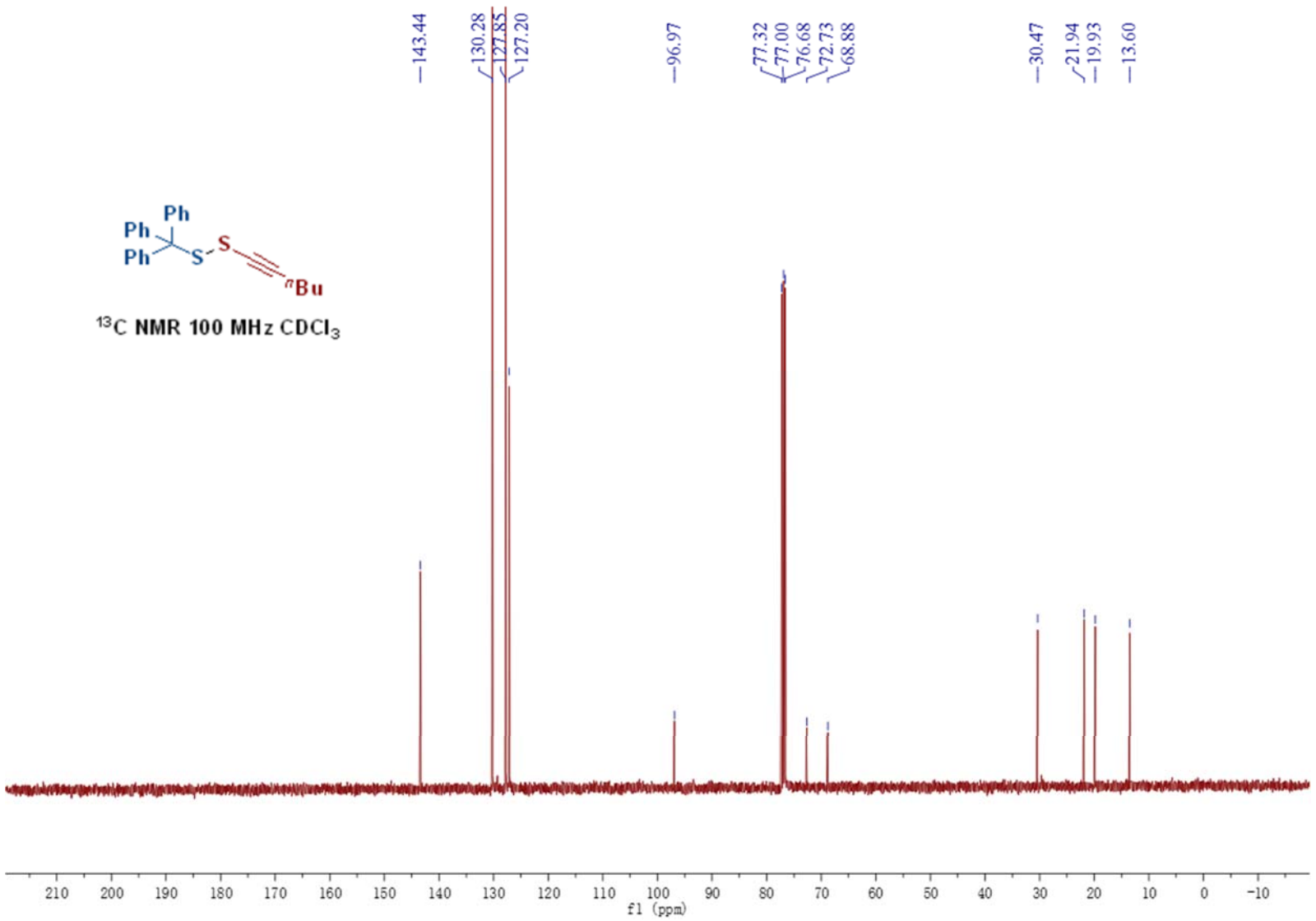
3f


7.26
2.32
2.30
1.42
1.37
0.91
0.90
0.88
S
S
nBu
1H NMR 400 MHz CDCl3
2.00
3.10
f1 (ppm)


94.39
77.32
77.00
76.69
71.36
49.24
30.49
29.73
21.87
19.78
13.55
S
S
nBu
13C NMR 100 MHz CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

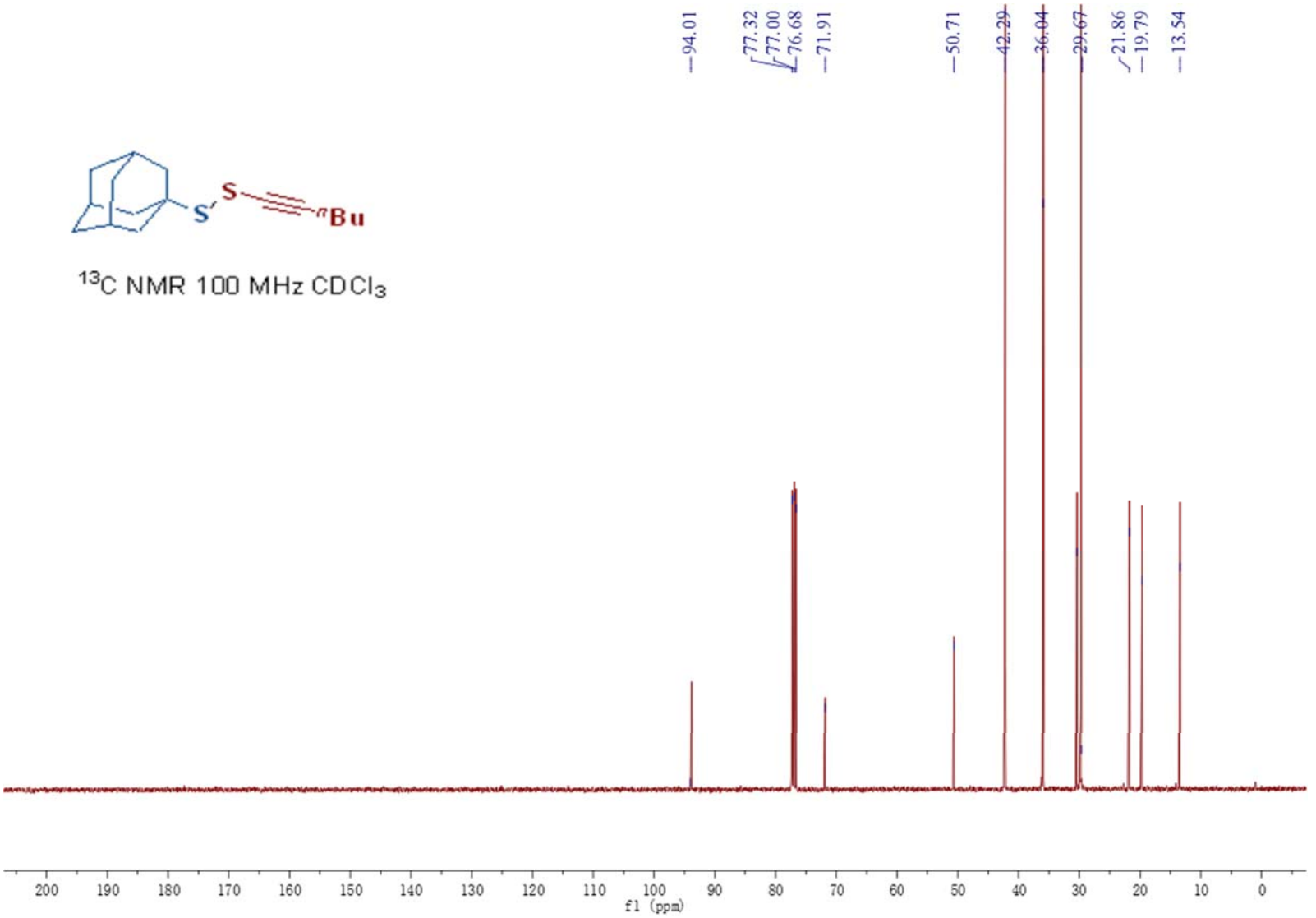
3g


7.36
7.35
7.34
7.33
7.30
7.28
7.28
7.26
7.25
2.26
2.24
2.22
1.47
1.34
0.92
0.90
0.88
Ph
Ph
Ph
S
S
nBu
1H NMR 400 MHz CDCl3
15.37
2.00
2.04
2.17
3.03
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

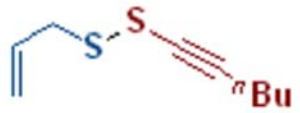

143.44
130.28
127.85
127.20
96.97
77.32
77.00
76.68
72.73
68.88
30.47
21.94
19.93
13.60
Ph
Ph
Ph
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3h

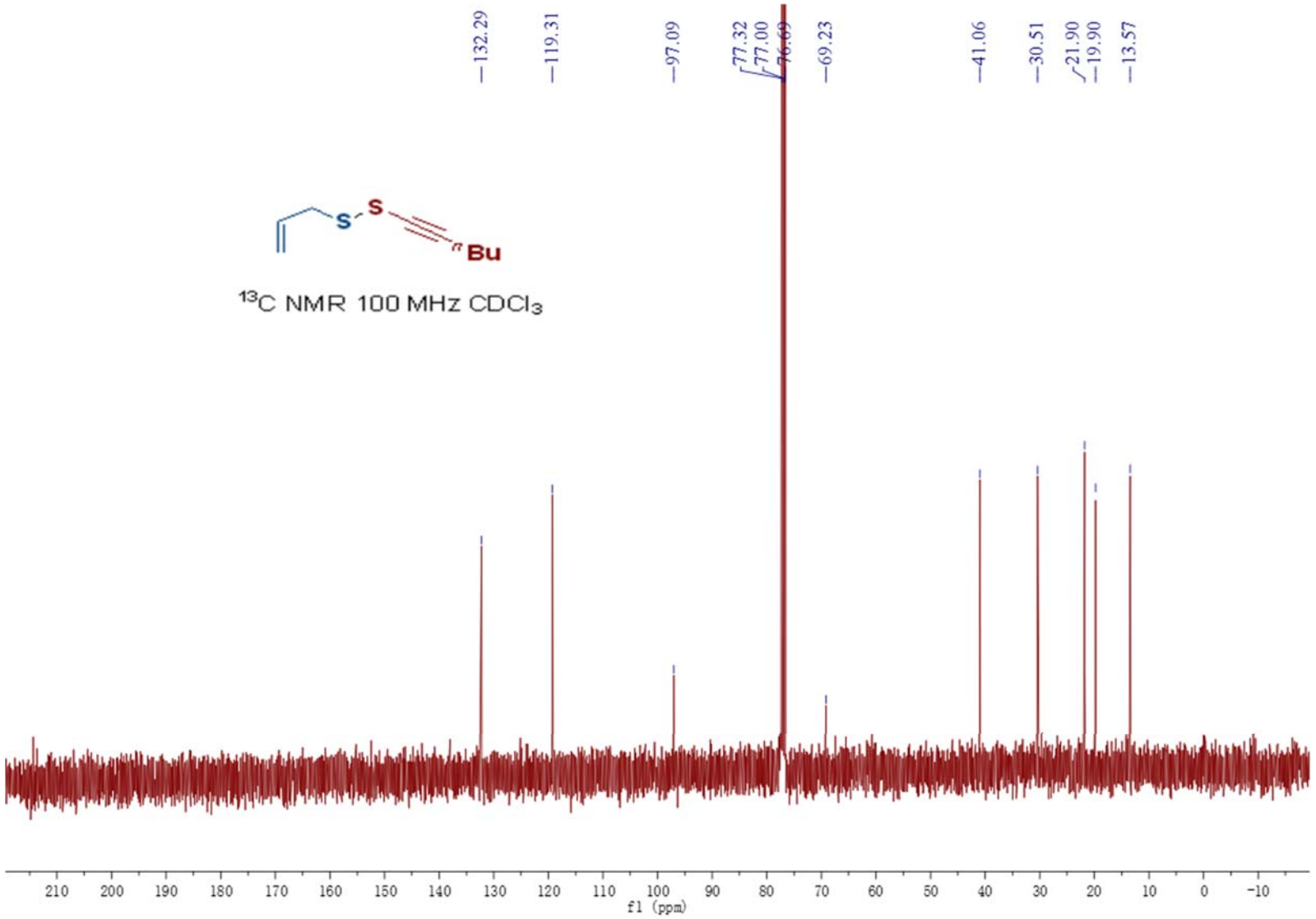
^{1}H NMR 400 MHz $CDCl_3$


S
S
nBu
13C NMR 100 MHz CDCl3
94.01
77.32
77.00
76.68
71.91
50.71
42.29
36.04
29.67
21.86
19.79
13.54
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

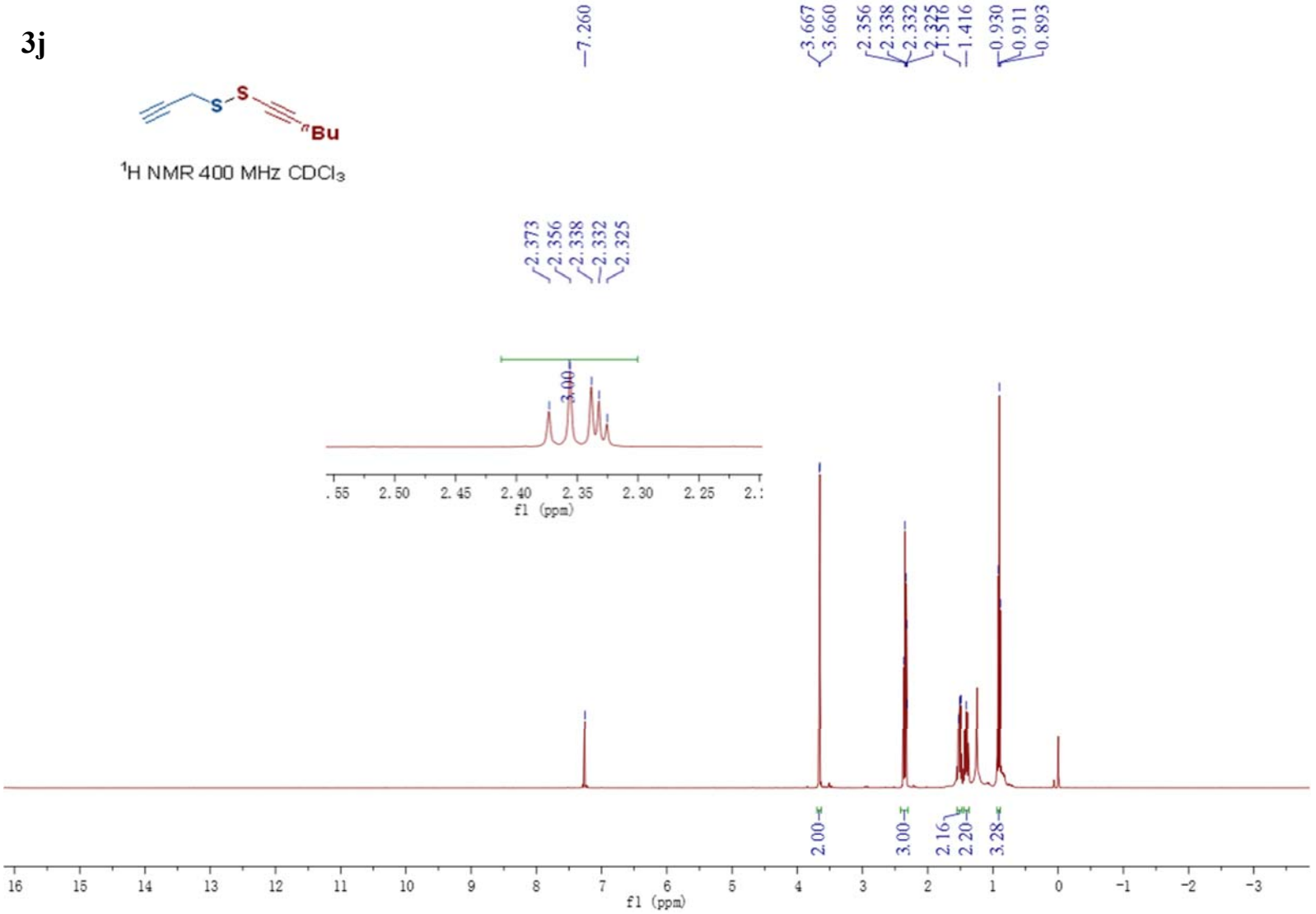
3i



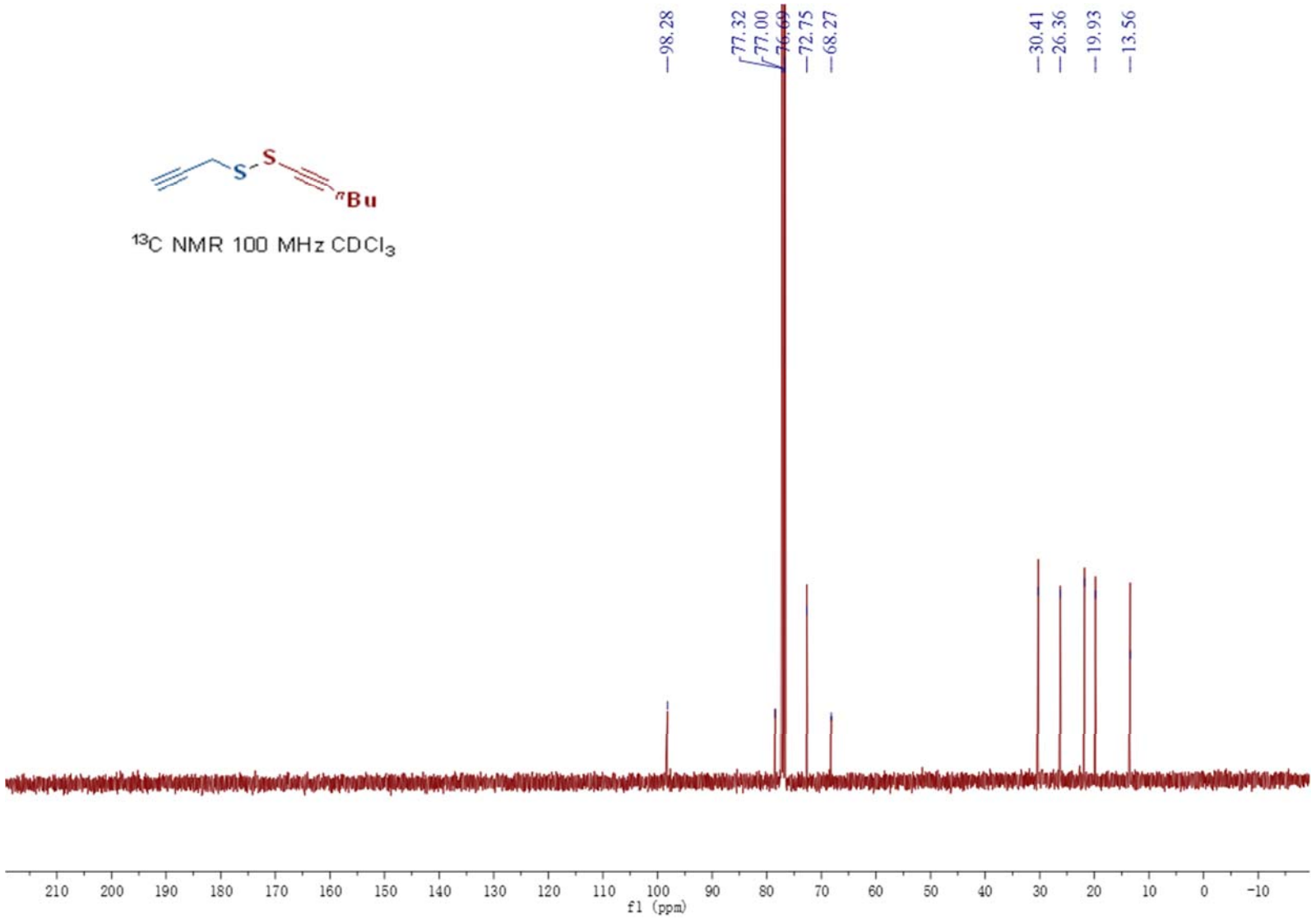
^{1}H NMR 400 MHz $CDCl_3$


132.29
119.31
97.09
77.32
77.00
76.69
69.23
41.06
30.51
21.90
19.90
13.57
S
S
Bu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

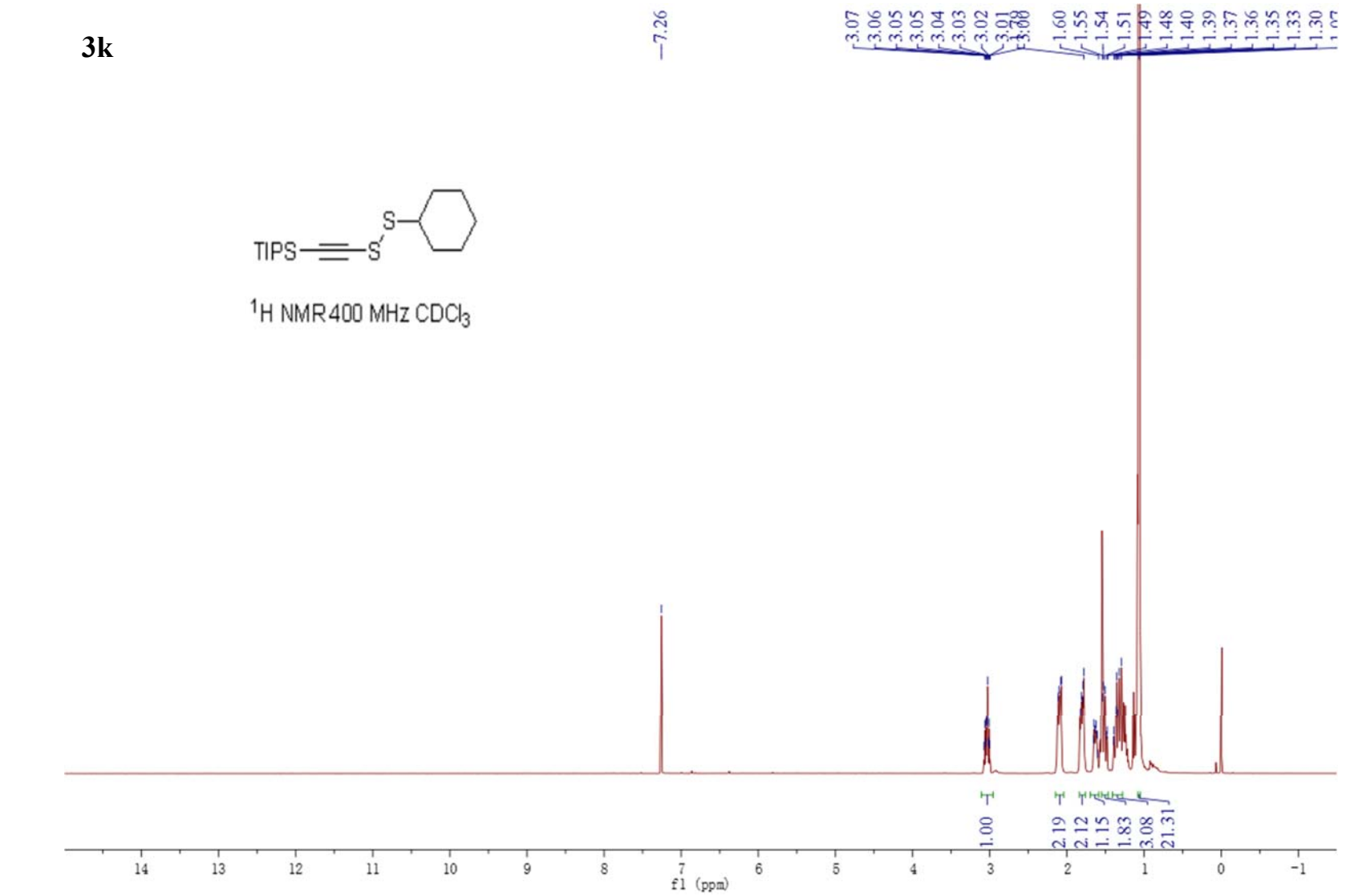
3j

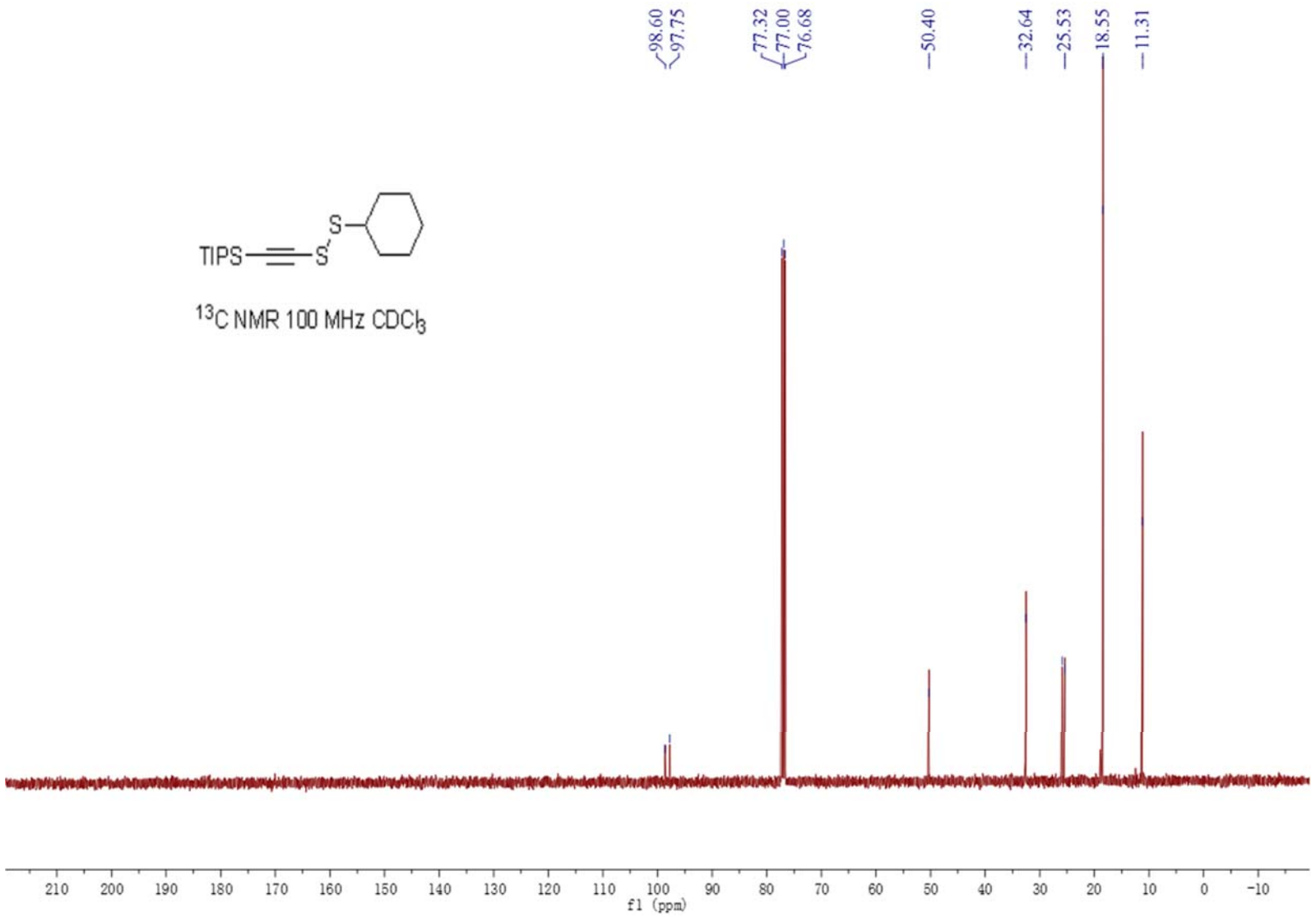


^{1}H NMR 400 MHz $CDCl_3$

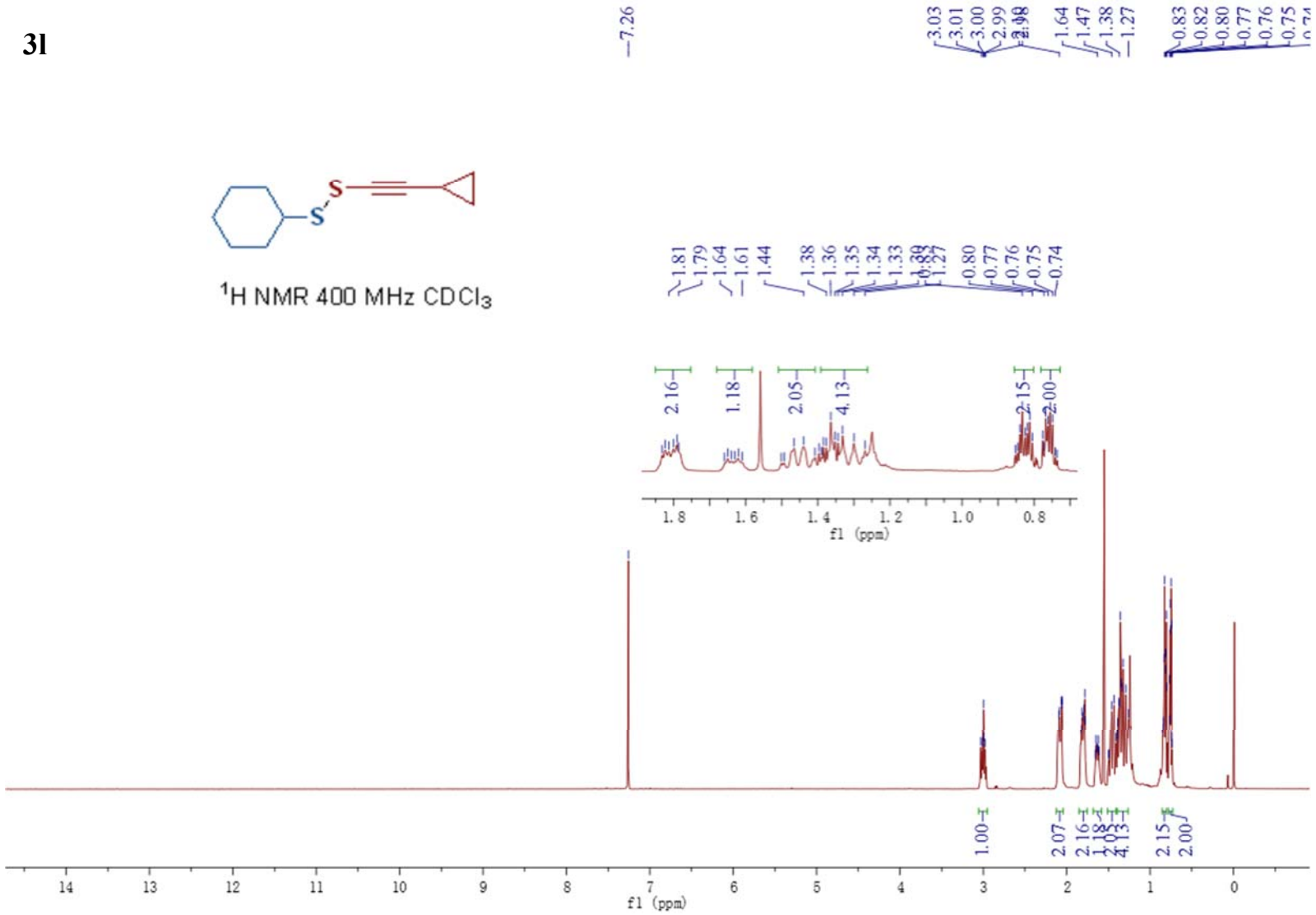

98.28
77.32
77.00
76.69
72.75
68.27
30.41
26.36
19.93
13.56
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3k

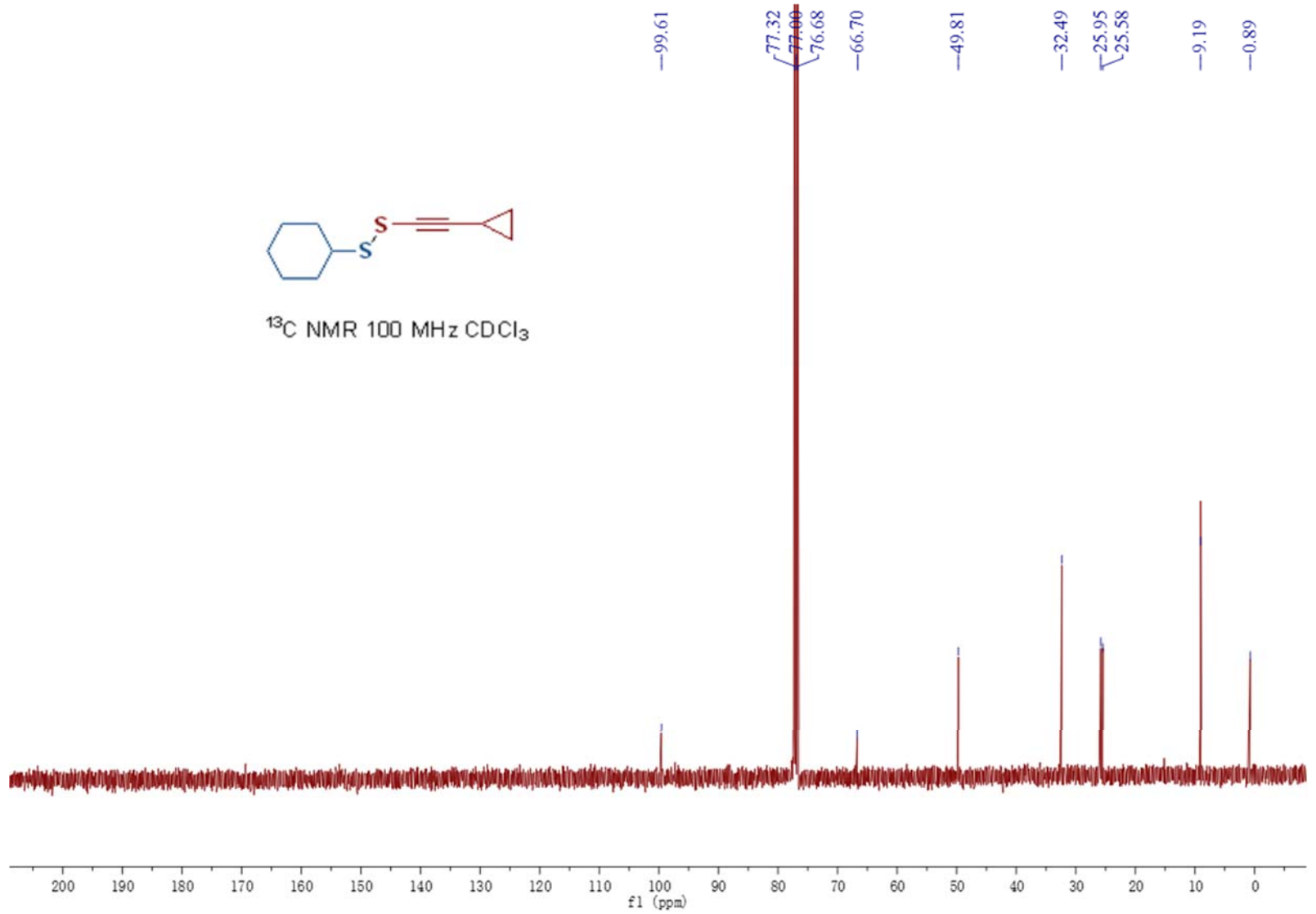

TIPS
S
S
^{1}H NMR 400 MHz $CDCl_3$
7.26
3.07
3.06
3.05
3.05
3.04
3.03
3.02
3.01
3.00
1.60
1.55
1.54
1.51
1.49
1.48
1.40
1.39
1.37
1.36
1.35
1.33
1.30
1.00
2.19
2.12
1.15
1.83
3.08
21.31
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


98.60
97.75
77.32
77.00
76.68
50.40
32.64
25.53
18.55
11.31
TIPS
S
S
^{13}C NMR 100 MHz $CDCl_3$
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

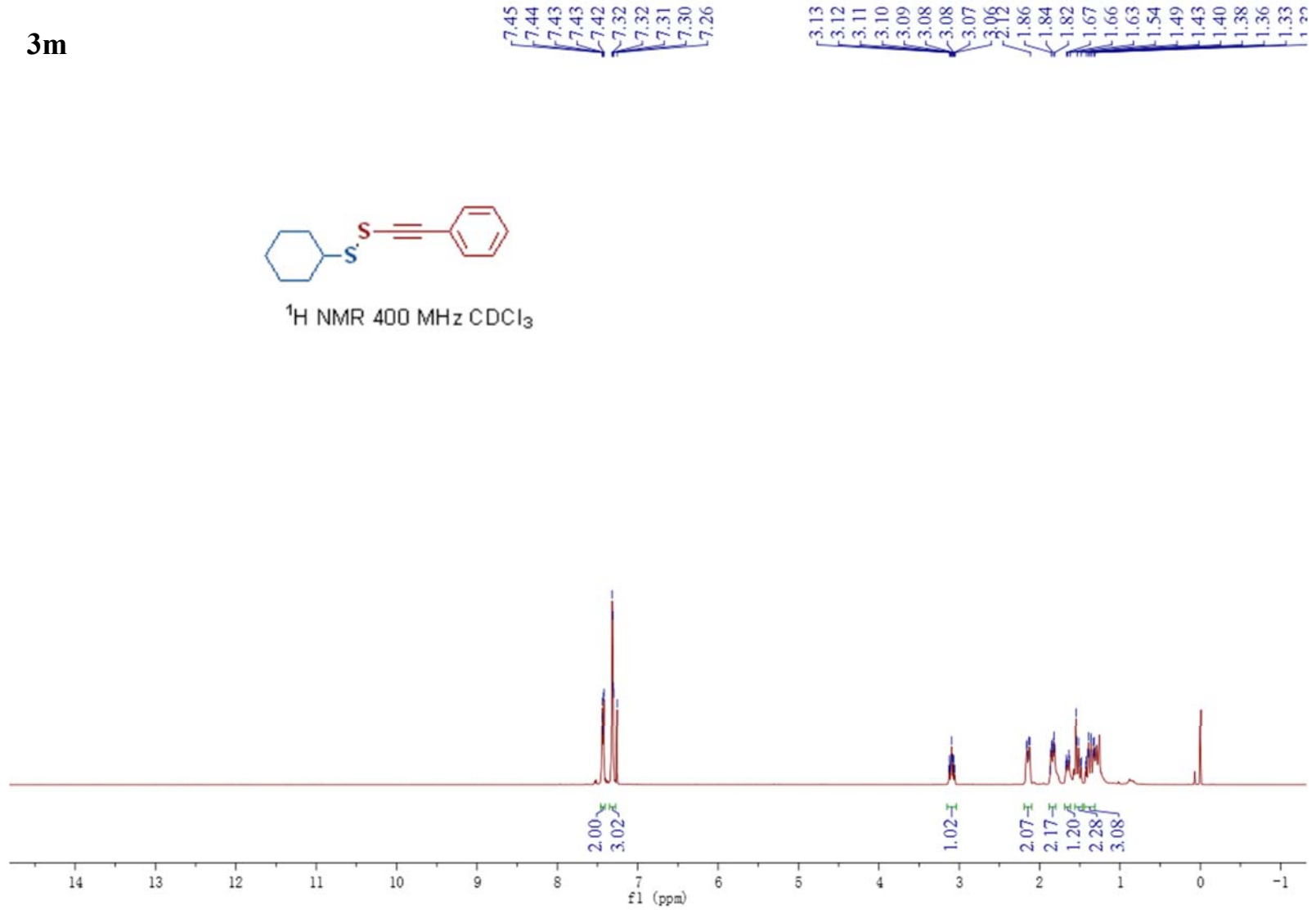
3l

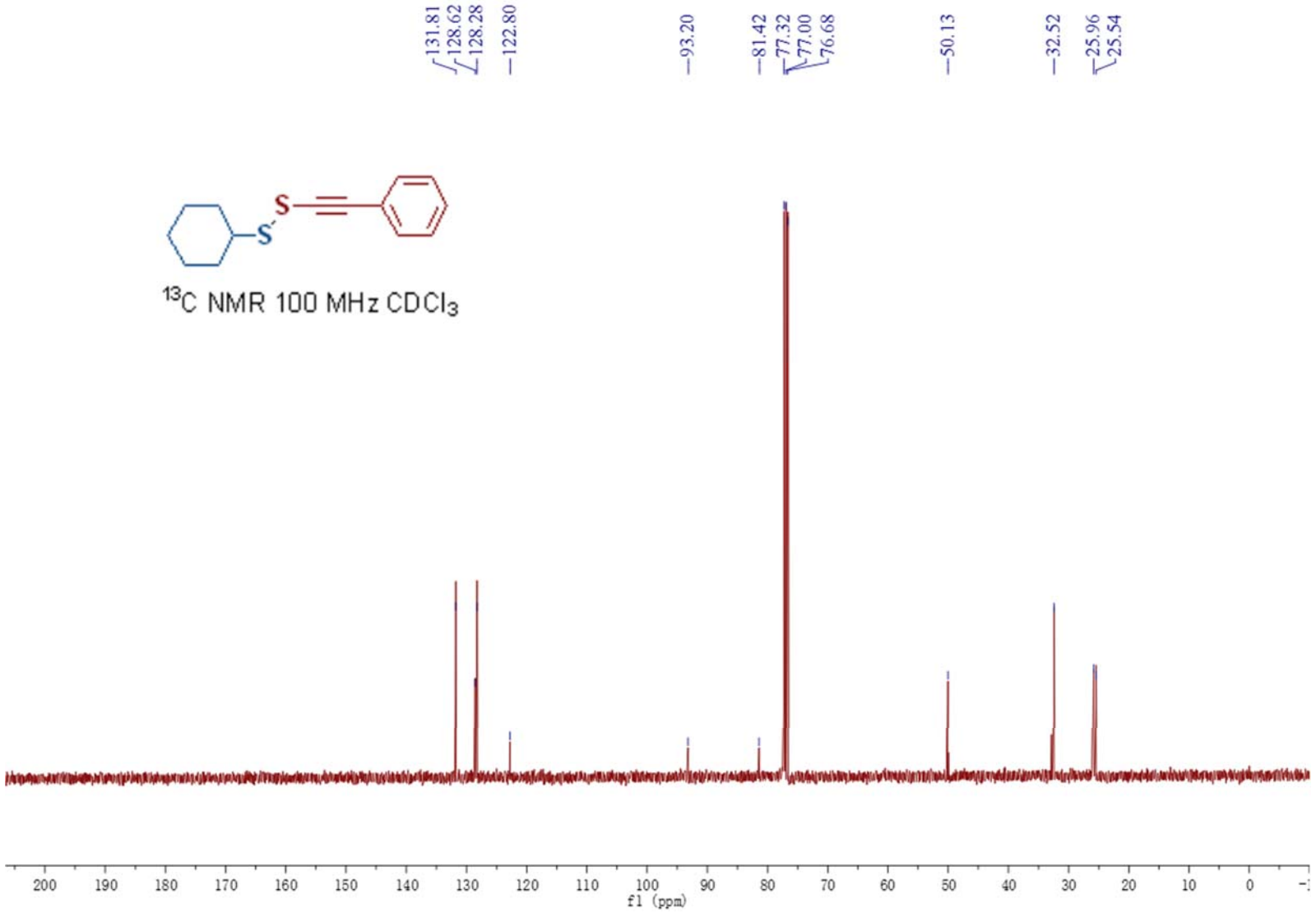


^{1}H NMR 400 MHz $CDCl_3$

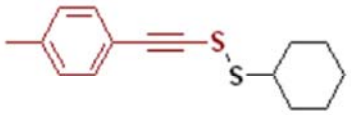

99.61
77.32
77.00
76.68
66.70
49.81
32.49
25.95
25.58
9.19
0.89
S
S
^{13}C NMR 100 MHz CDCl$_3$
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3m

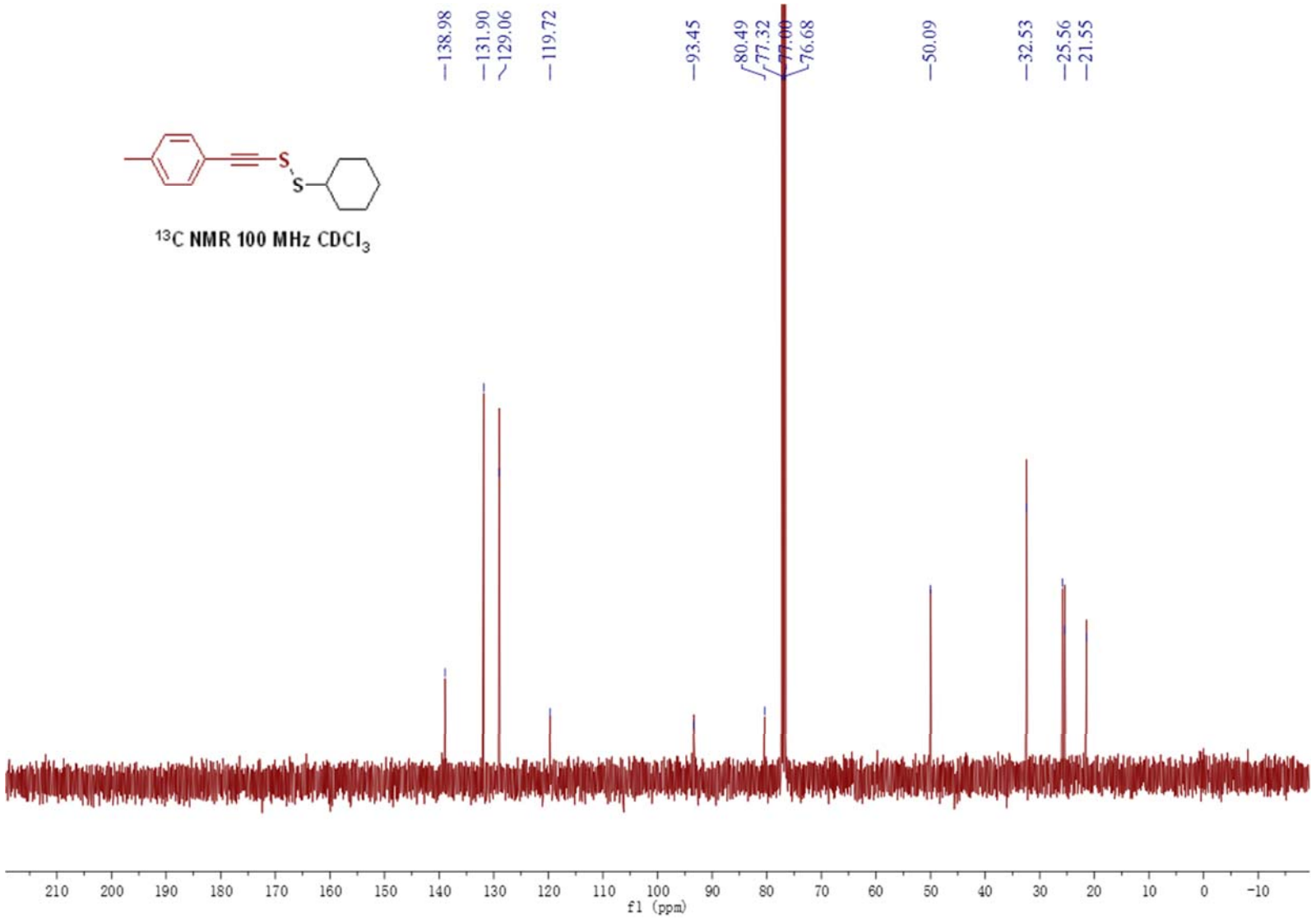

7.45
7.44
7.43
7.43
7.42
7.32
7.32
7.31
7.30
7.26
3.13
3.12
3.11
3.10
3.09
3.08
3.08
3.07
3.06
2.12
1.86
1.84
1.82
1.67
1.66
1.63
1.54
1.49
1.43
1.40
1.38
1.36
1.33
S
S
1H NMR 400 MHz CDCl3
2.00
3.02
1.02
2.07
2.17
1.20
2.28
3.08
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


131.81
128.62
128.28
122.80
93.20
81.42
77.32
77.00
76.68
50.13
32.52
25.96
25.54
S
S
13C NMR 100 MHz CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

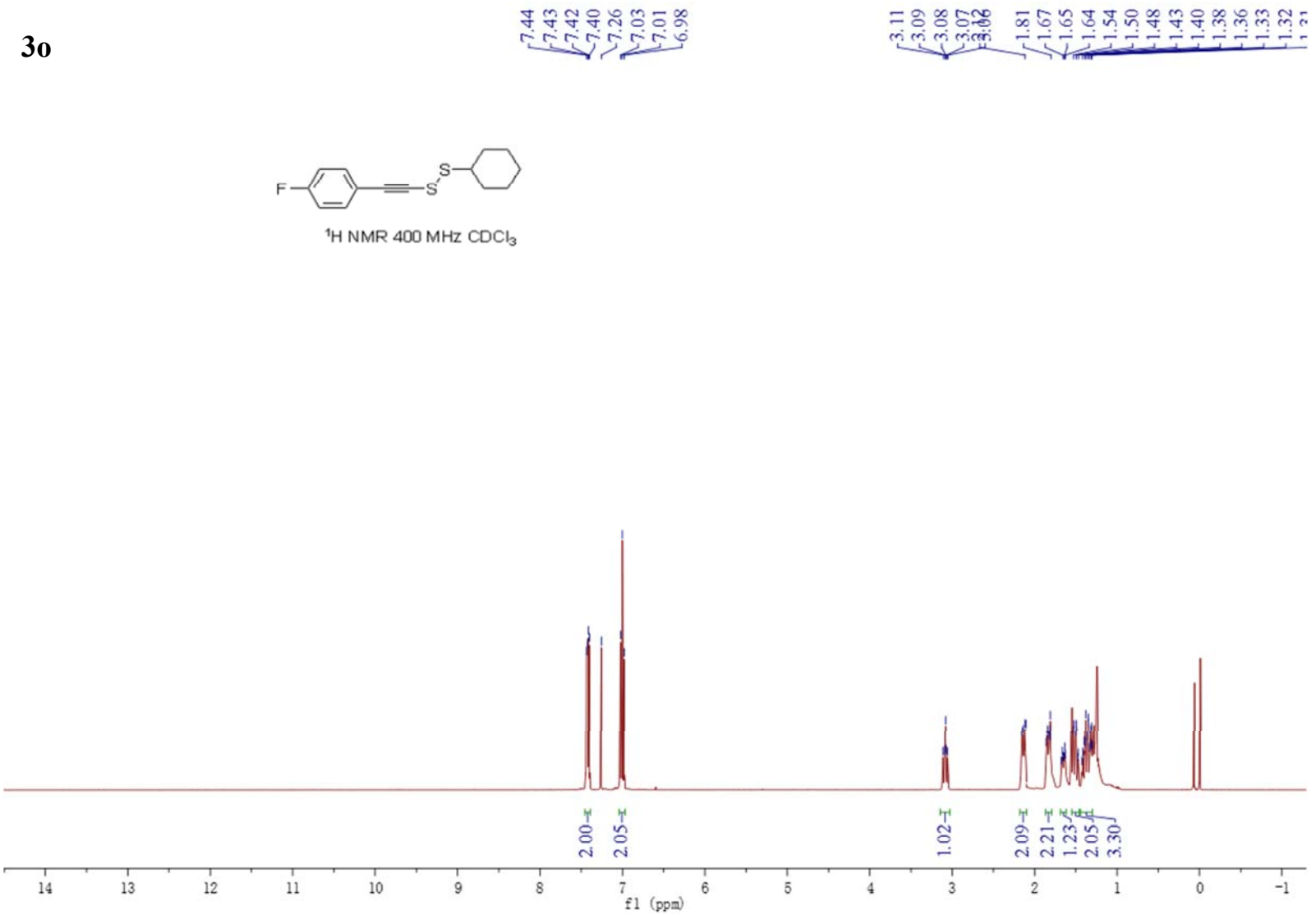
3n

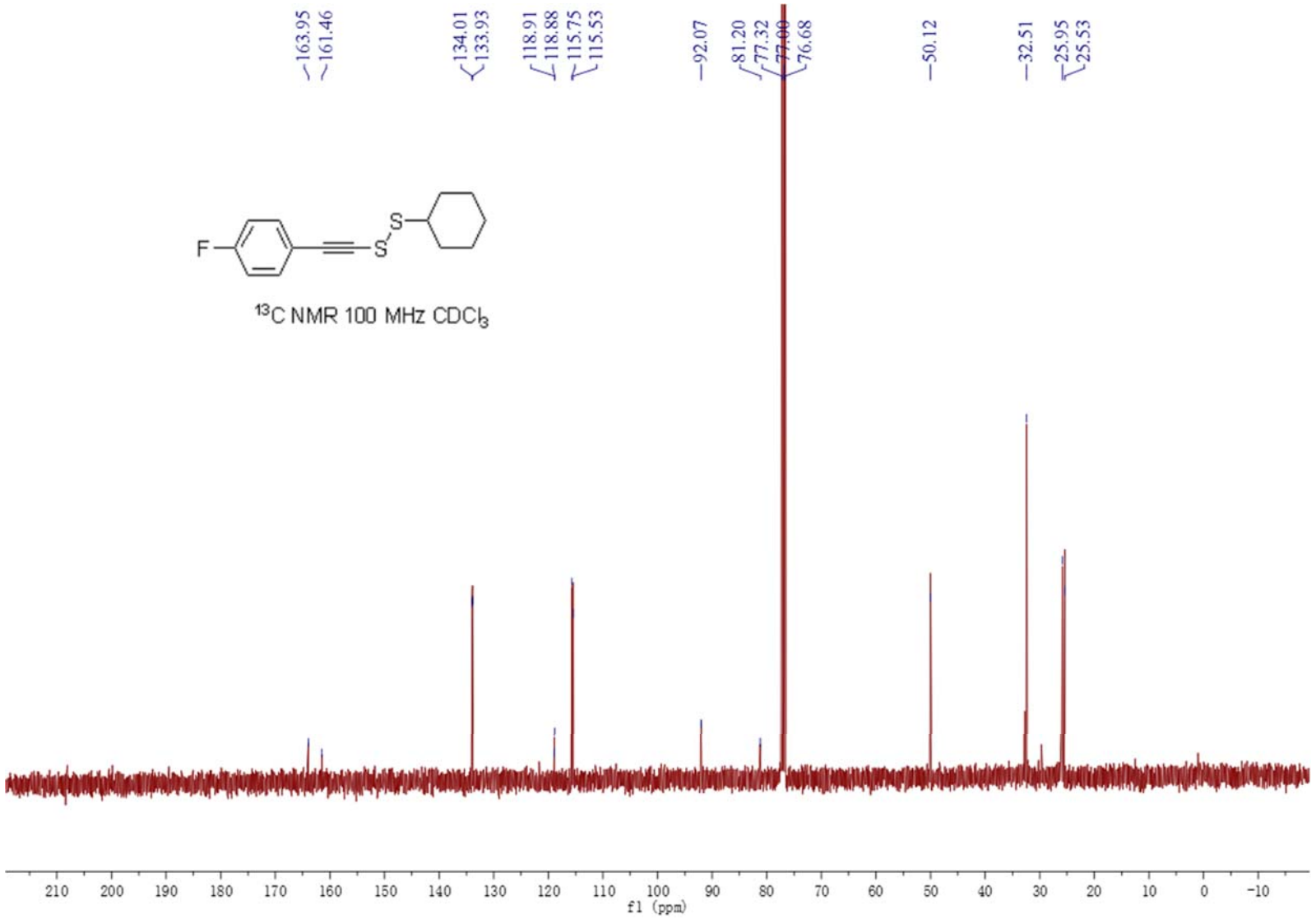


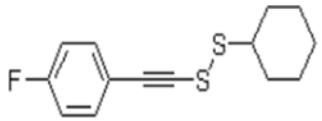
^{1}H NMR 400 MHz $CDCl_3$


138.98
131.90
129.06
119.72
93.45
80.49
77.32
77.00
76.68
50.09
32.53
25.56
21.55
13C NMR 100 MHz CDCl3
f1 (ppm)

3o

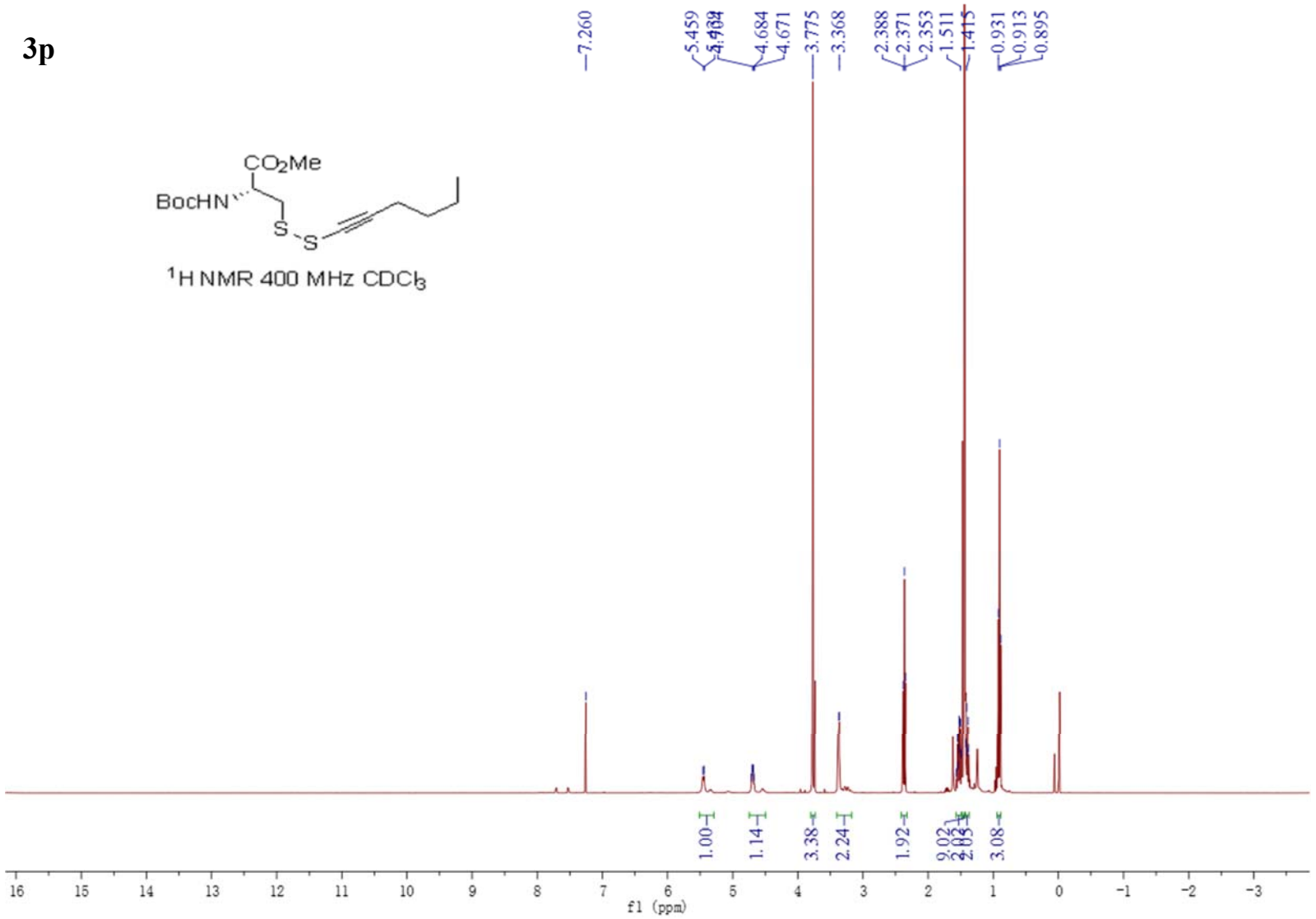

7.44
7.43
7.42
7.40
7.26
7.03
7.01
6.98
3.11
3.09
3.08
3.07
1.81
1.67
1.65
1.64
1.54
1.50
1.48
1.43
1.40
1.38
1.36
1.33
1.32
F
S
S
^{1}H NMR 400 MHz CDCl$_3$
2.00
2.05
1.02
2.09
2.21
1.23
2.05
3.30
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

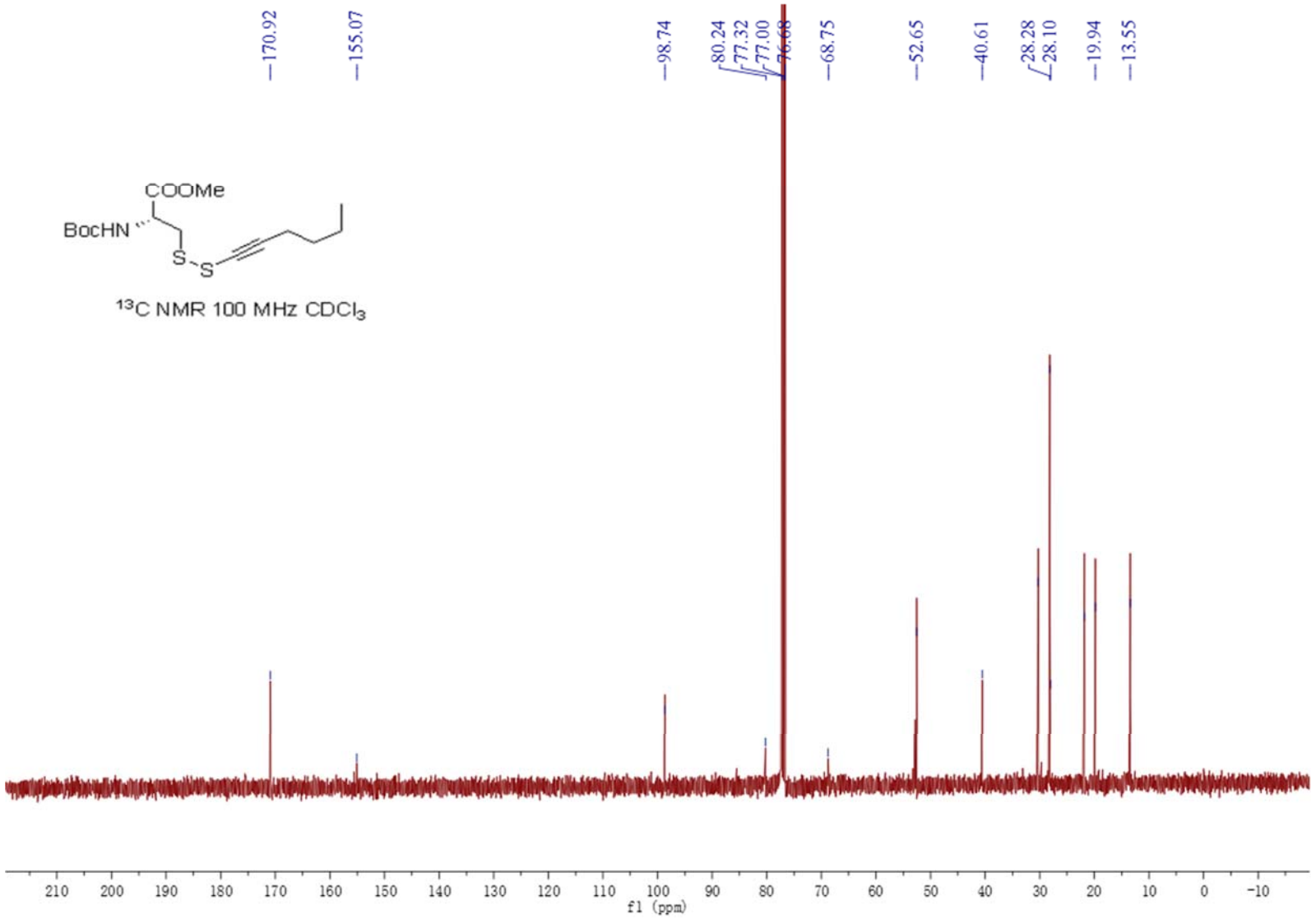

163.95
161.46
134.01
133.93
118.91
118.88
115.75
115.53
92.07
81.20
77.32
77.00
76.68
50.12
32.51
25.95
25.53
F
S
S
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



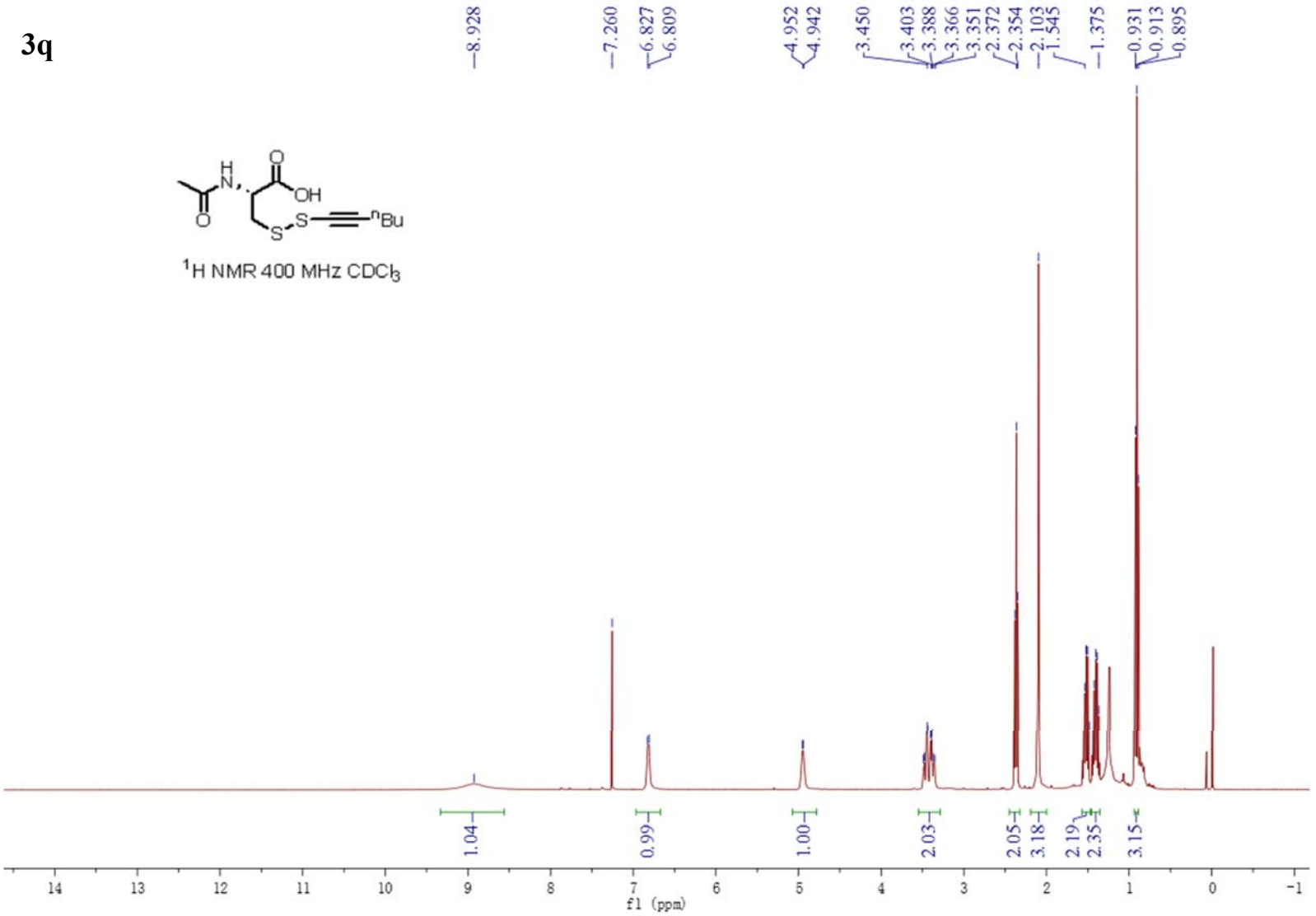
^{19}F NMR 376.3 MHz $CDCl_3$

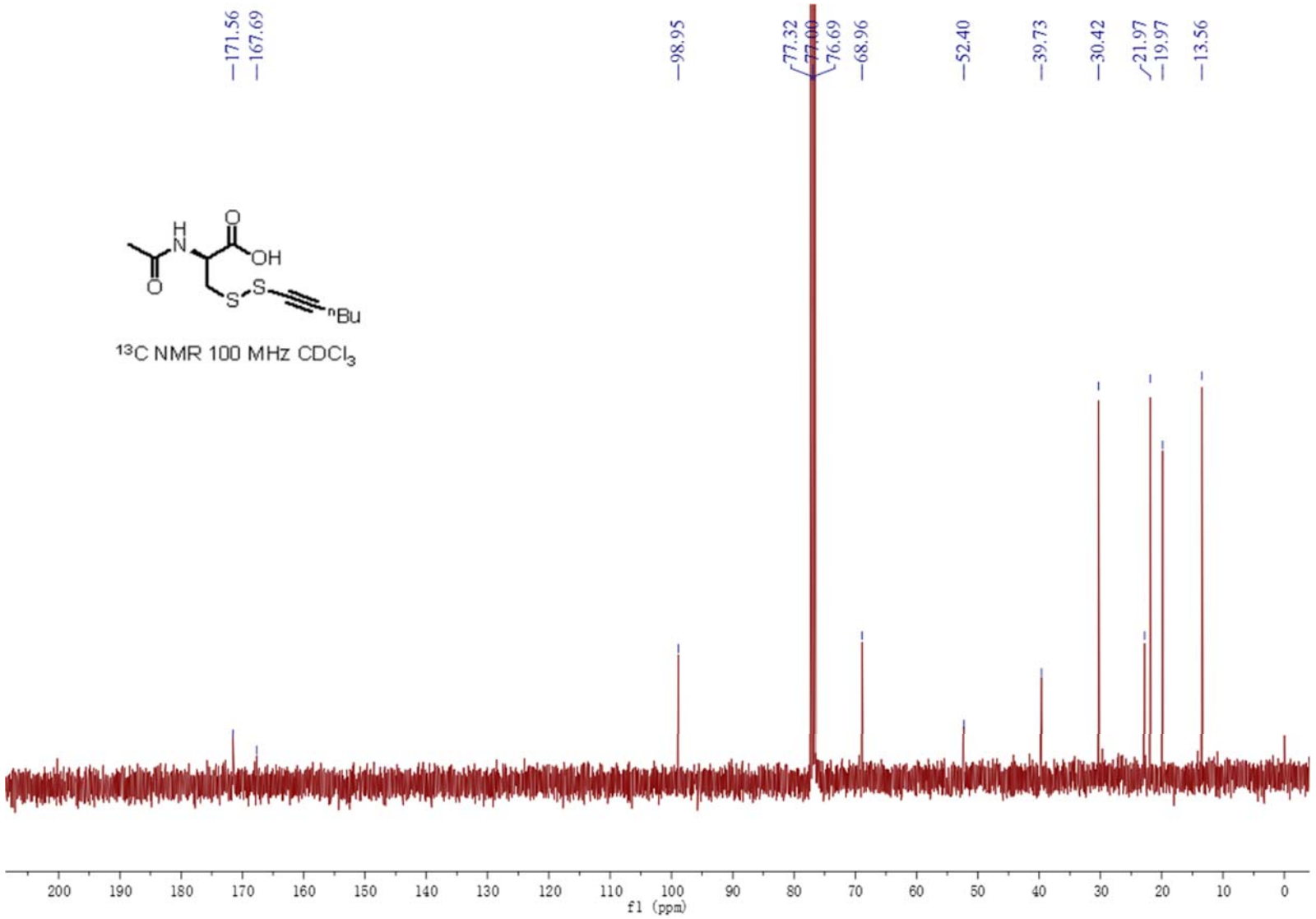
3p


CO2Me
BocHN
S
S
1H NMR 400 MHz CDCl3
7.260
5.459
4.684
4.671
3.775
3.368
2.388
2.371
2.353
1.511
1.415
0.931
0.913
0.895
1.00
1.14
3.38
2.24
1.92
9.02
2.05
3.08
f1 (ppm)

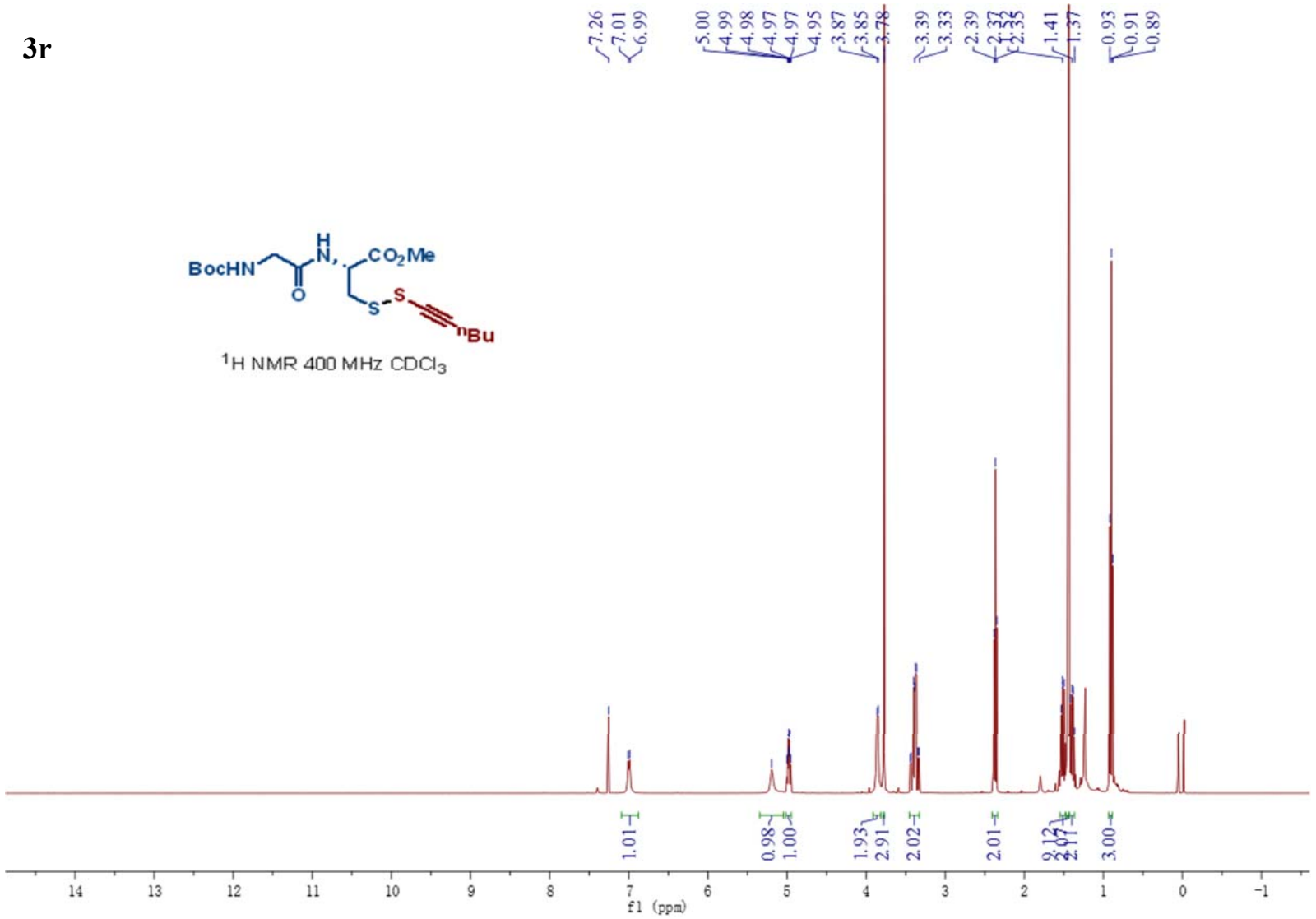

170.92
155.07
98.74
80.24
77.32
77.00
76.68
68.75
52.65
40.61
28.28
28.10
19.94
13.55
COOMe
BocHN
S
S
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

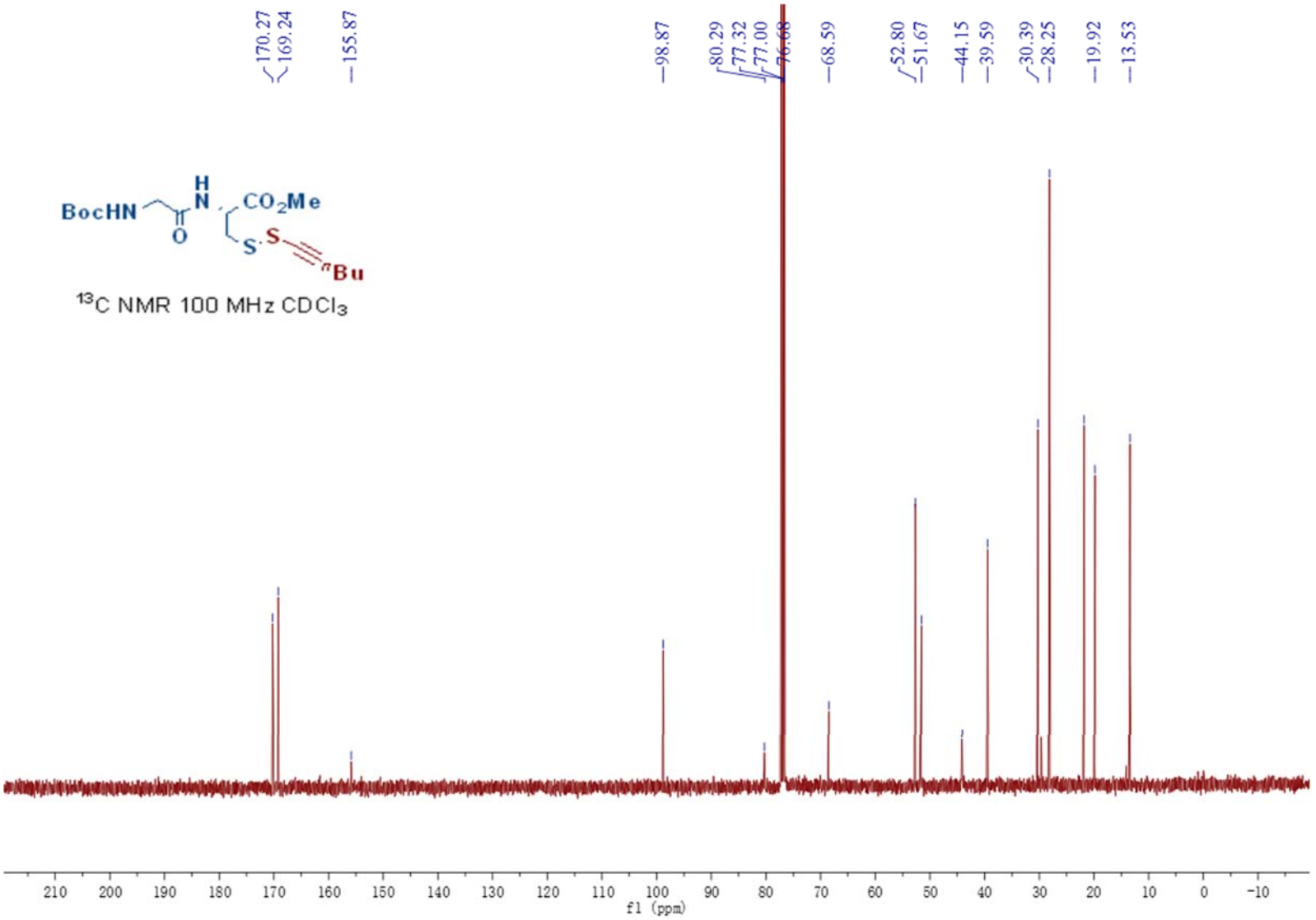
3q


8.928
7.260
6.827
6.809
4.952
4.942
3.450
3.403
3.388
3.366
3.351
2.372
2.354
2.103
1.545
1.375
0.931
0.913
0.895
OH
S
S
nBu
1H NMR 400 MHz CDCl3
1.04
0.99
1.00
2.03
2.05
3.18
2.19
2.35
3.15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

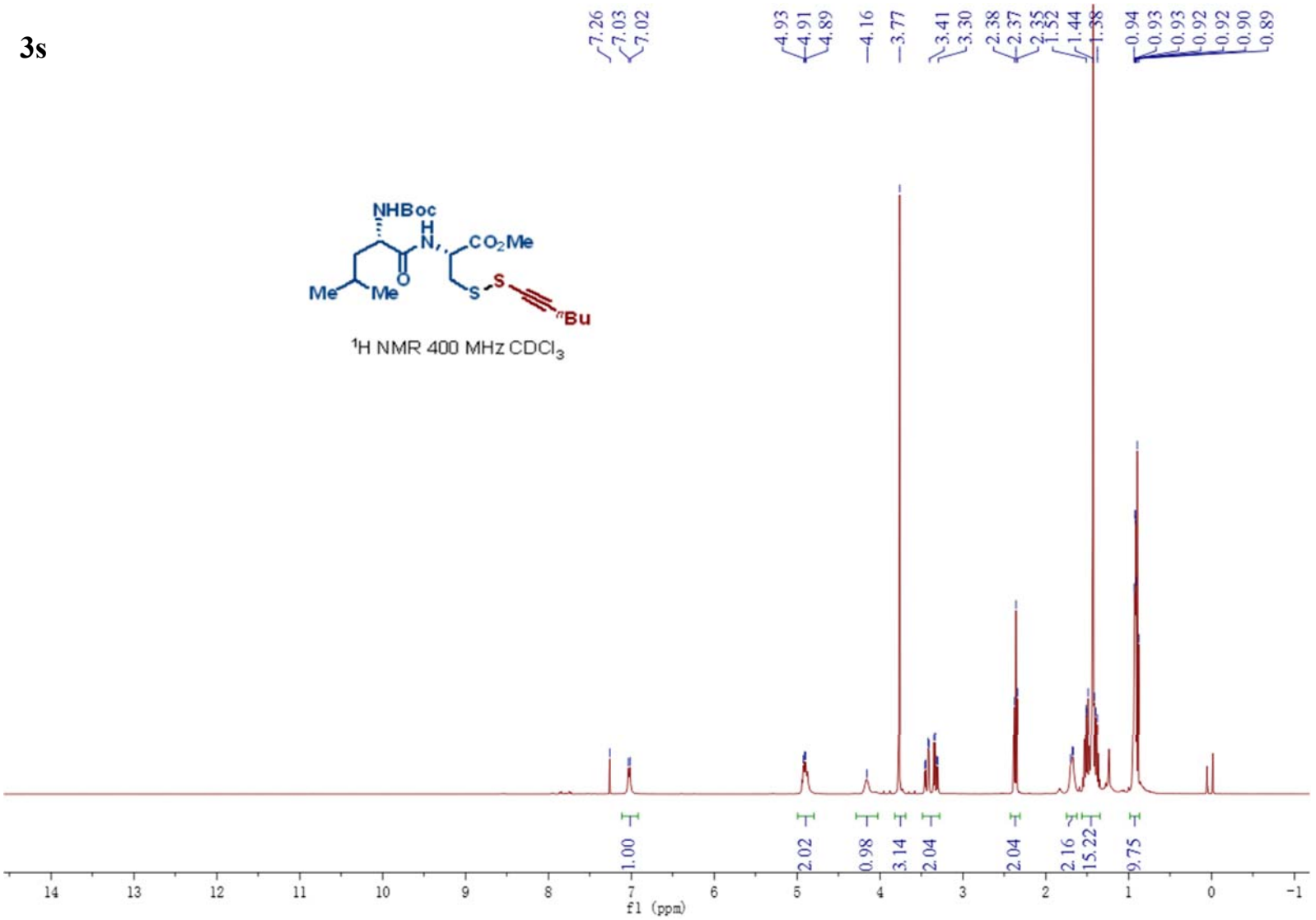

171.56
167.69
98.95
77.32
77.00
76.69
68.96
52.40
39.73
30.42
21.97
19.97
13.56
OH
nBu
13C NMR 100 MHz CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

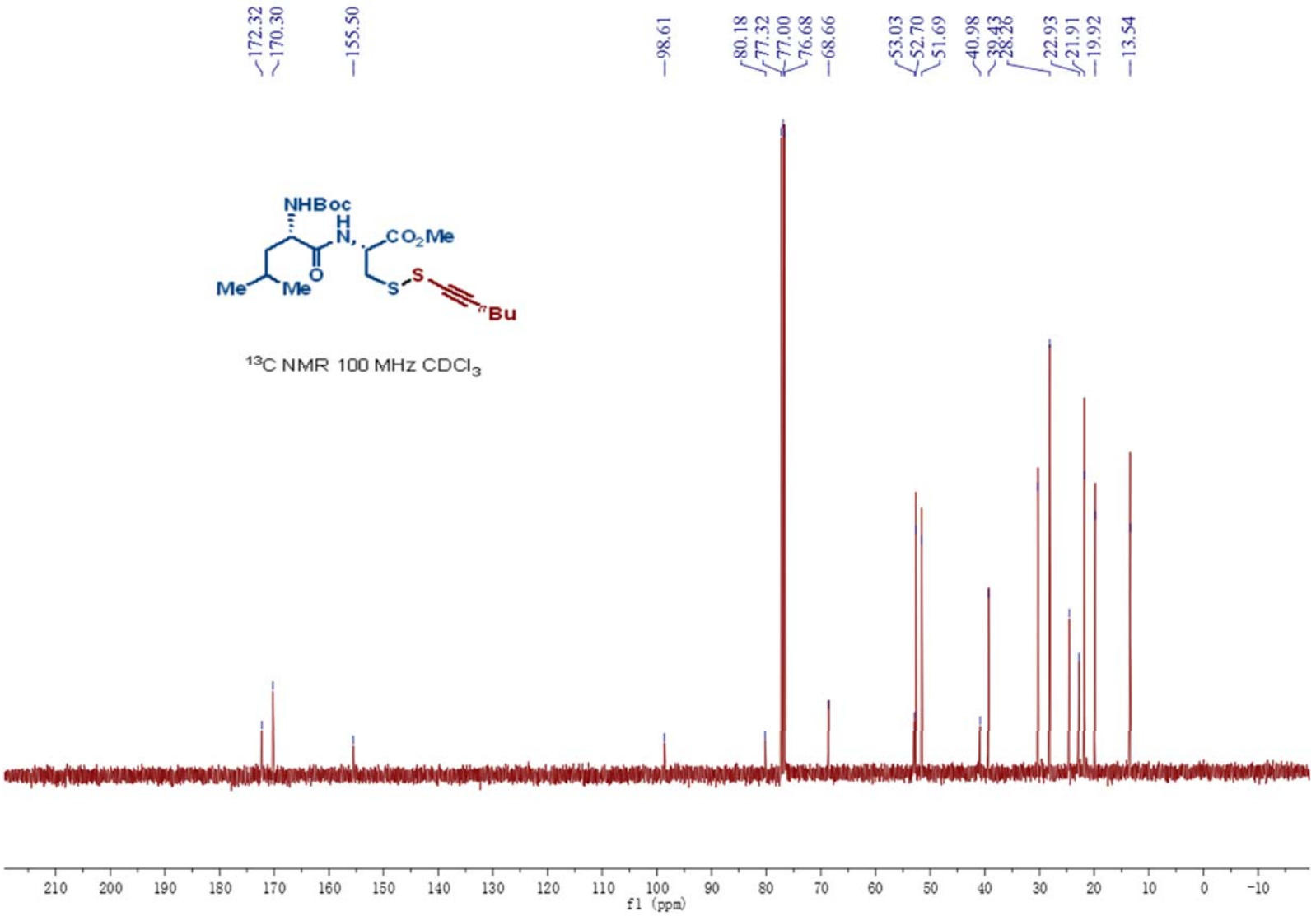
3r


BocHN
H
N
CO2Me
S
S
nBu
1H NMR 400 MHz CDCl3
7.26
7.01
6.99
5.00
4.99
4.98
4.97
4.97
4.95
3.87
3.85
3.78
3.39
3.33
2.39
2.37
2.35
1.41
1.37
0.93
0.91
0.89
1.01
0.98
1.00
1.93
2.91
2.02
2.01
3.00
f1 (ppm)

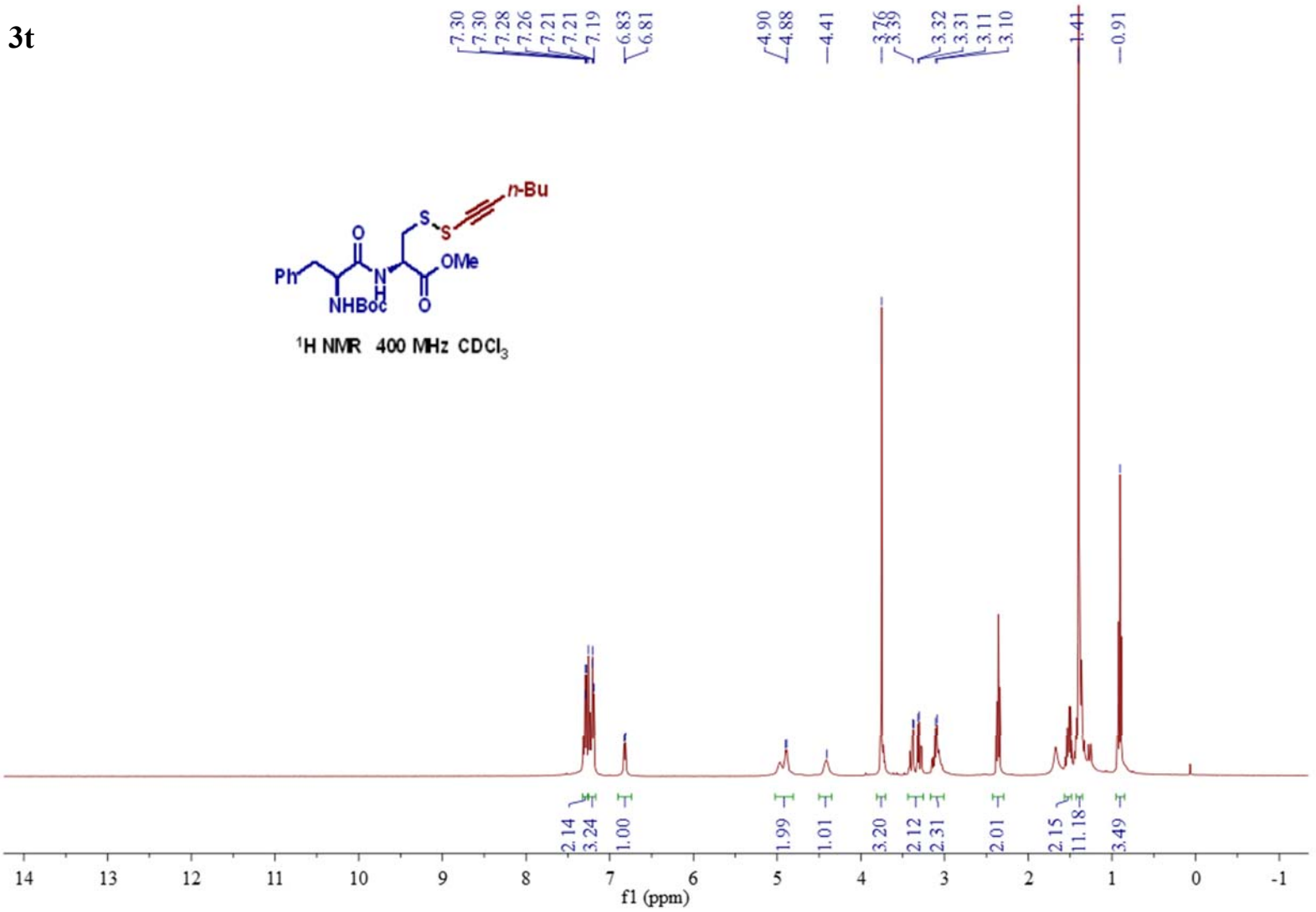

170.27
169.24
155.87
98.87
80.29
77.32
77.00
76.68
68.59
52.80
51.67
44.15
39.59
30.39
28.25
19.92
13.53
BocHN
H
N
O
CO2Me
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

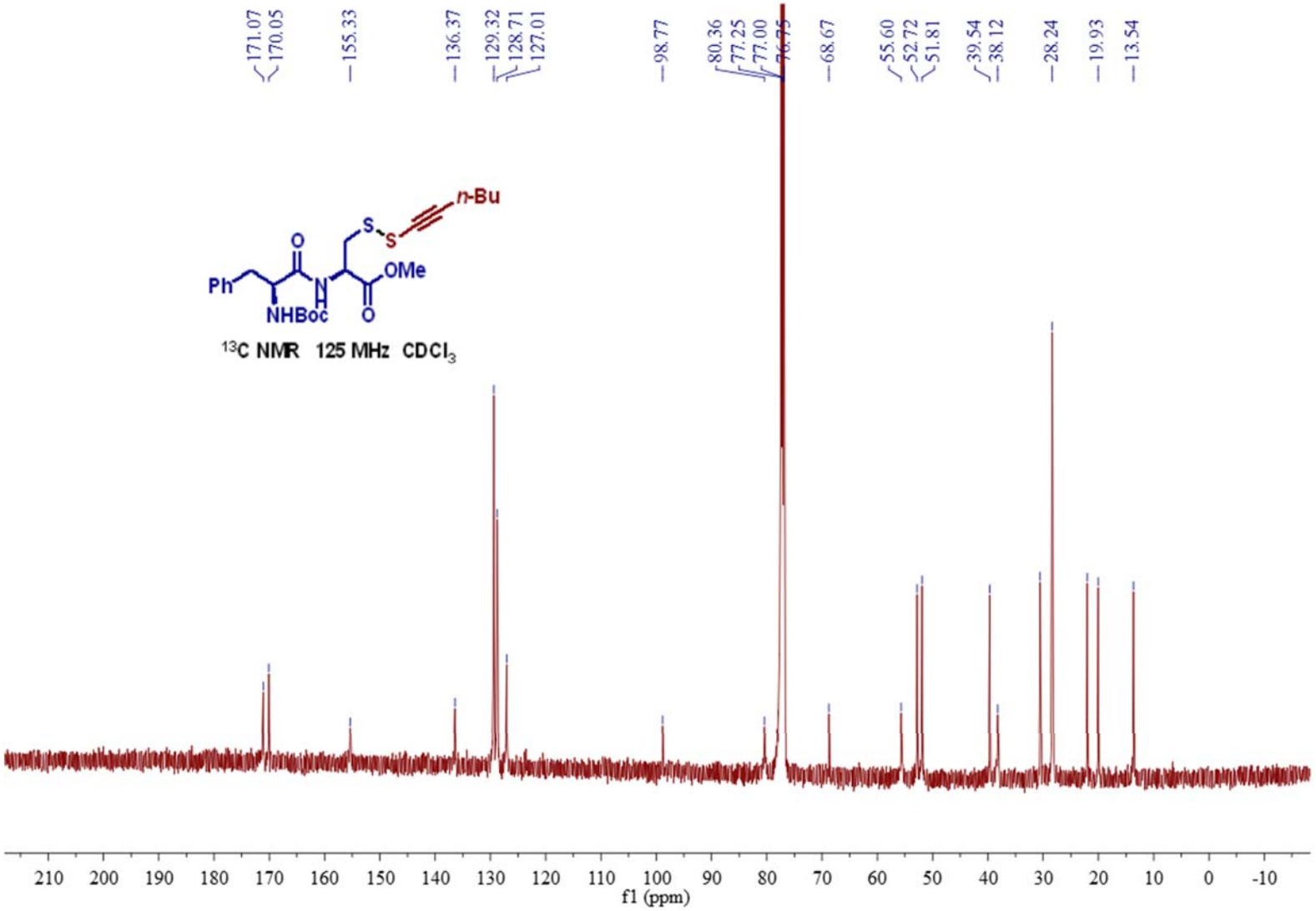
3s


7.26
7.03
7.02
4.93
4.91
4.89
4.16
3.77
3.41
3.30
2.38
2.37
2.35
1.52
1.44
1.38
0.94
0.93
0.93
0.92
0.92
0.90
0.89
NHBoc
H
N
CO2Me
O
Me
Me
S
S
nBu
1H NMR 400 MHz CDCl3
1.00
2.02
0.98
3.14
2.04
2.04
2.16
15.22
9.75
f1 (ppm)

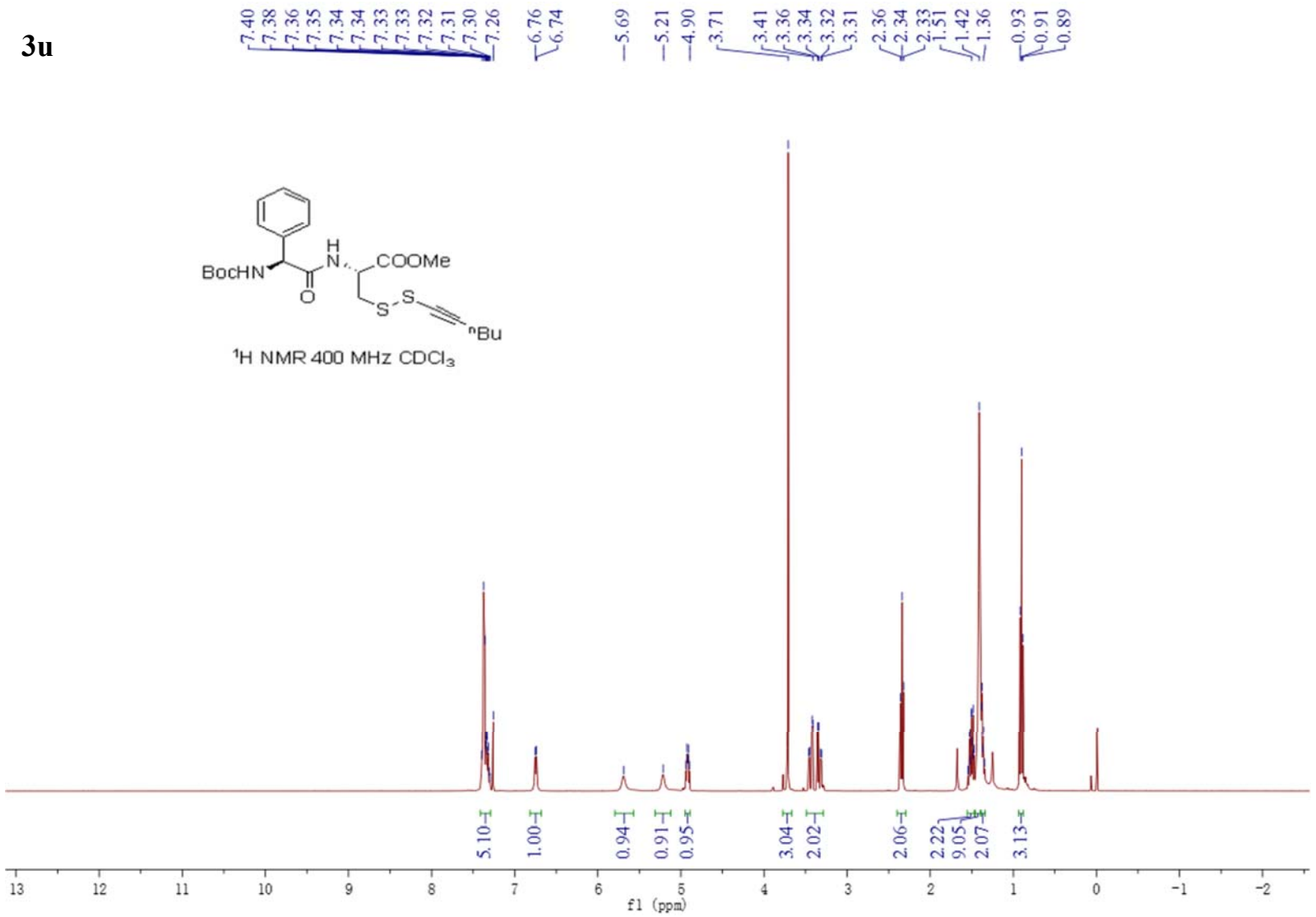

172.32
170.30
155.50
98.61
80.18
77.32
77.00
76.68
68.66
53.03
52.70
51.69
40.98
39.43
28.26
22.93
21.91
19.92
13.54
NHBoc
H
N
CO2Me
Me
Me
O
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

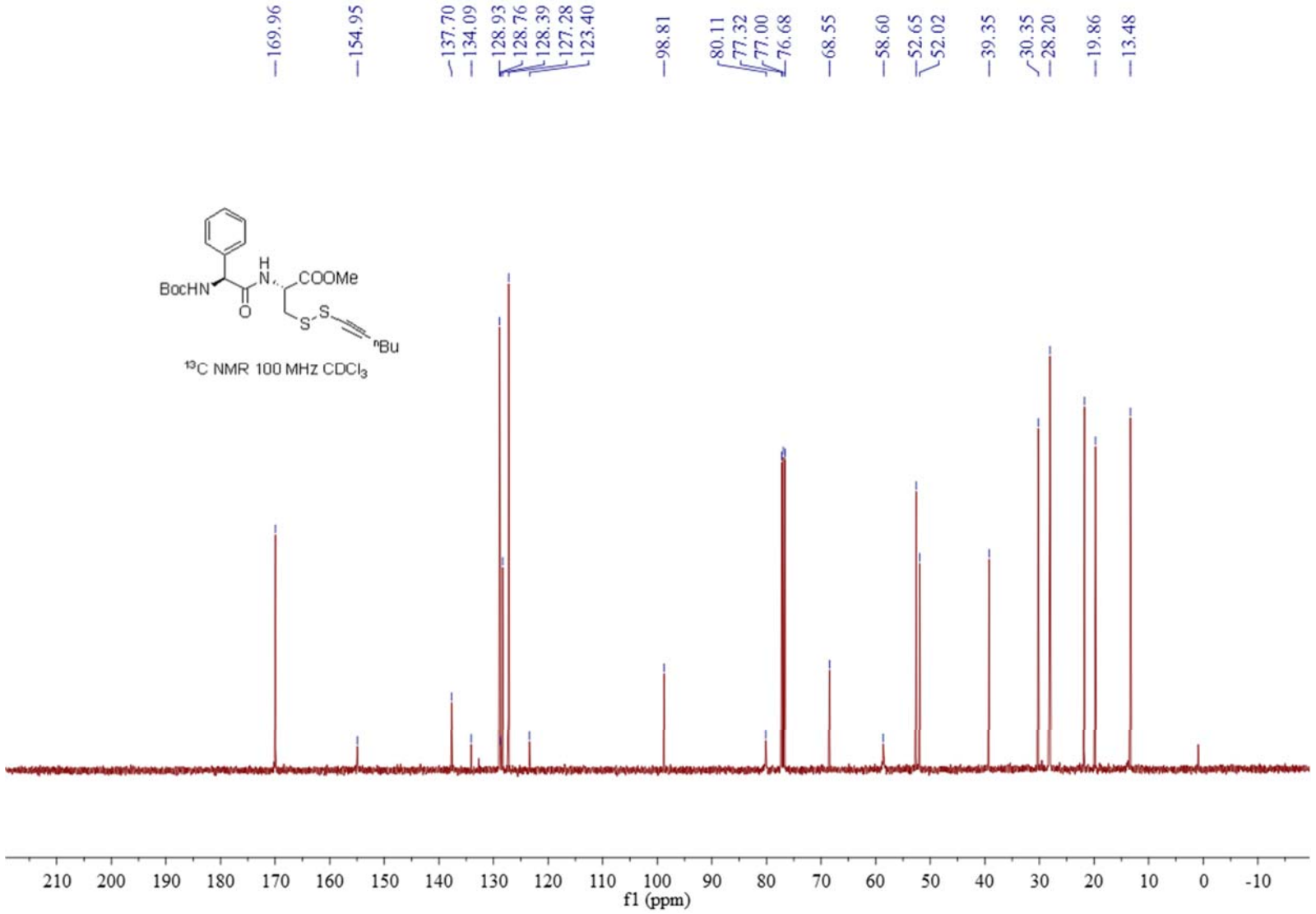
3t


7.30
7.30
7.28
7.26
7.21
7.21
7.19
6.83
6.81
4.90
4.88
4.41
3.76
3.39
3.32
3.31
3.11
3.10
1.41
0.91
n-Bu
S
S
O
Ph
N
H
OMe
NHBoc
O
¹H NMR 400 MHz CDCl₃
2.14
3.24
1.00
1.99
1.01
3.20
2.12
2.31
2.01
2.15
11.18
3.49
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

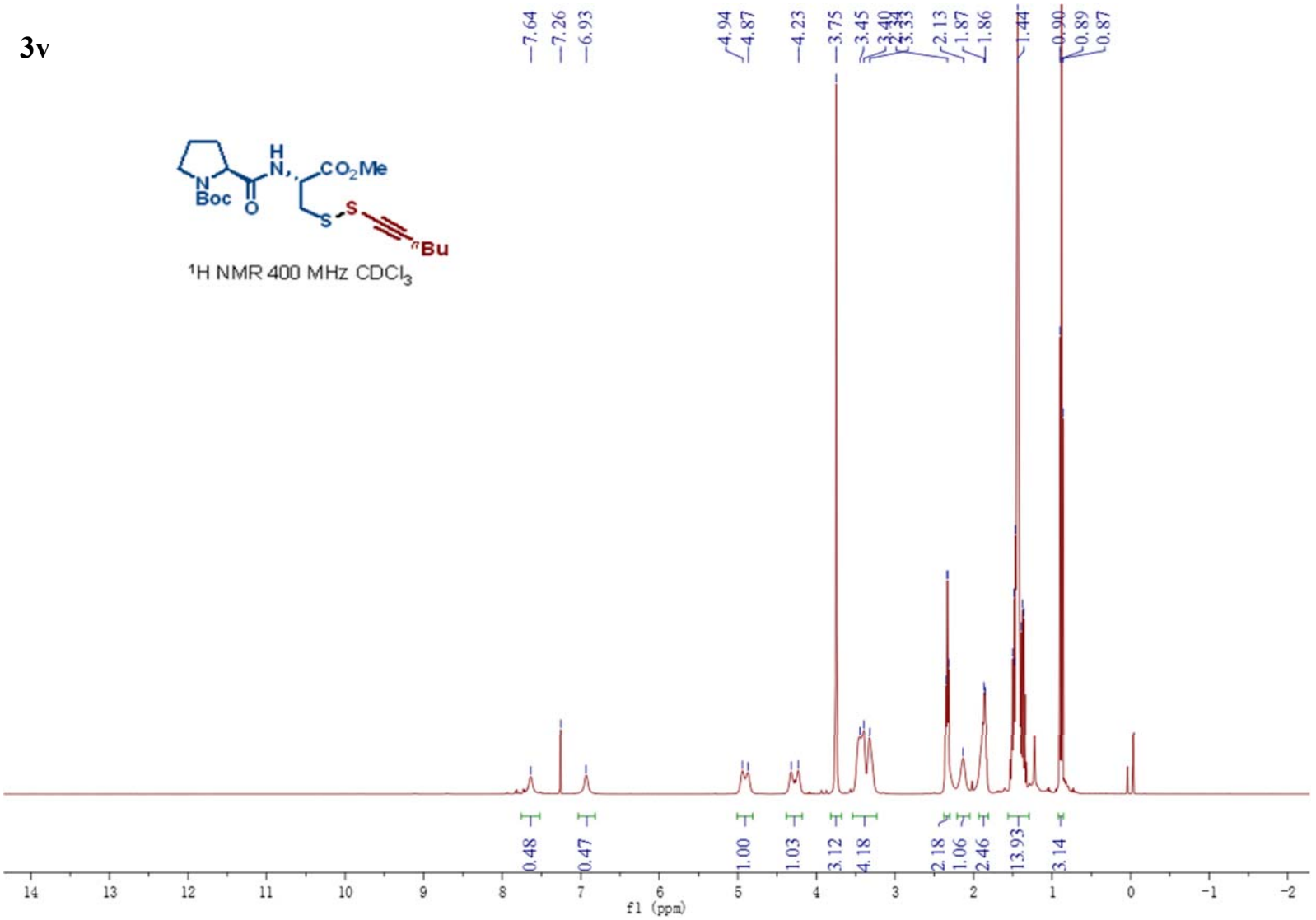

171.07
170.05
155.33
136.37
129.32
128.71
127.01
98.77
80.36
77.25
77.00
76.75
68.67
55.60
52.72
51.81
39.54
38.12
28.24
19.93
13.54
n-Bu
S
S
O
Ph
N
H
OMe
NHBoc
O
13C NMR 125 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

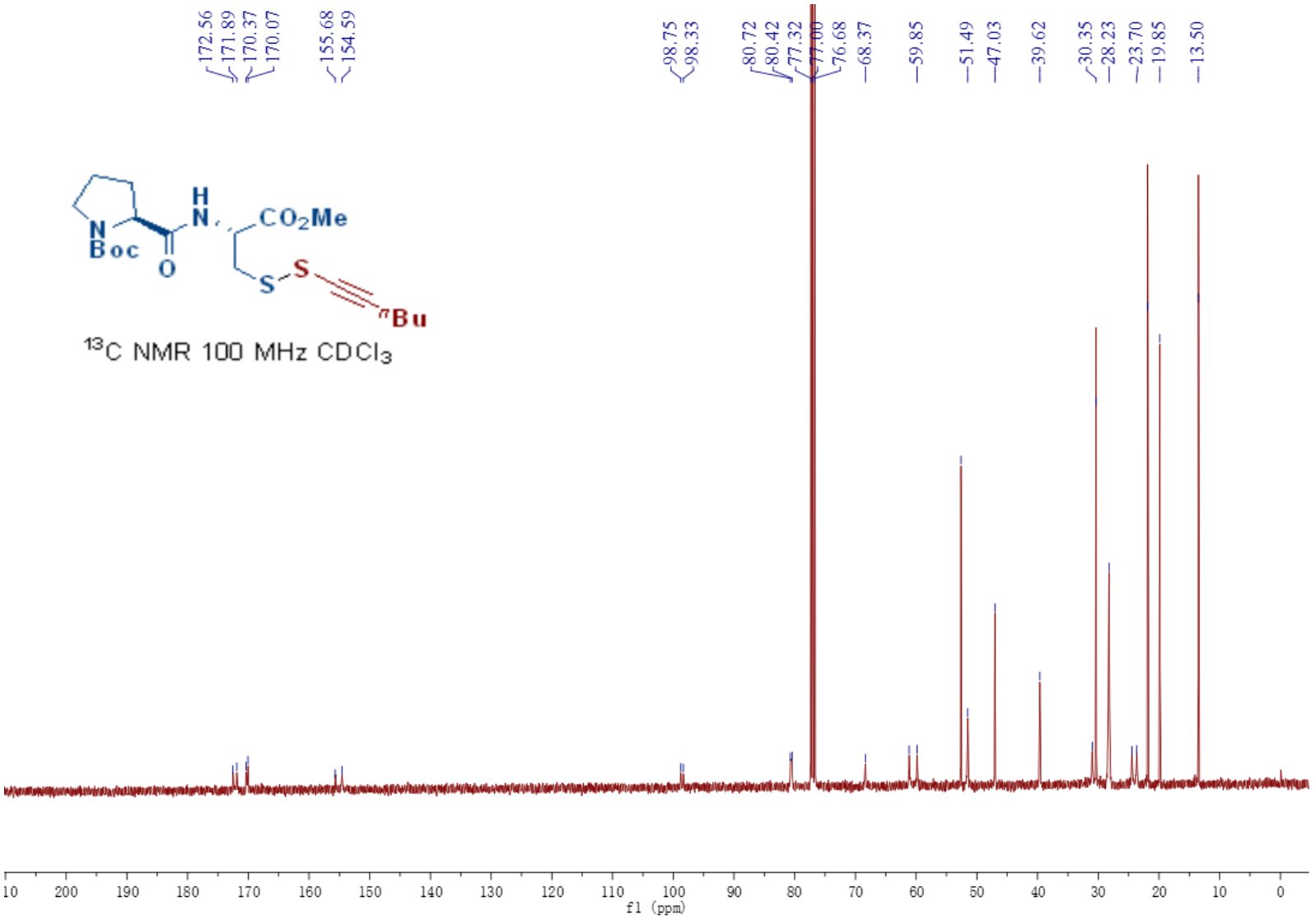
3u


7.40
7.38
7.36
7.35
7.34
7.34
7.33
7.33
7.32
7.31
7.30
7.26
6.76
6.74
5.69
5.21
4.90
3.71
3.41
3.36
3.34
3.32
3.31
2.36
2.34
2.33
1.51
1.42
1.36
0.93
0.91
0.89
BocHN
H
N
O
COOMe
S
S
nBu
1H NMR 400 MHz CDCl3
5.10
1.00
0.94
0.91
0.95
3.04
2.02
2.06
2.22
9.05
2.07
3.13
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

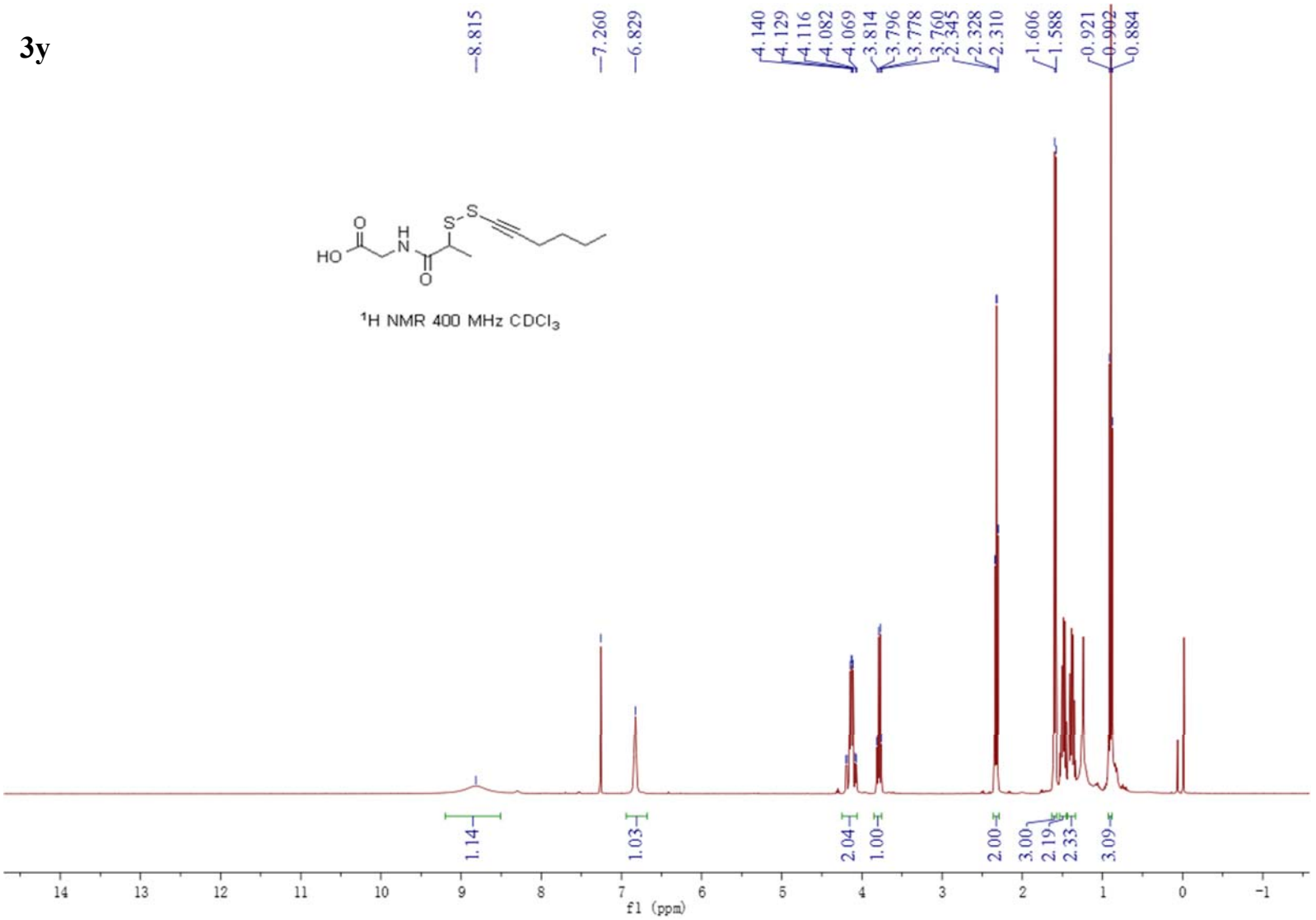

169.96
154.95
137.70
134.09
128.93
128.76
128.39
127.28
123.40
98.81
80.11
77.32
77.00
76.68
68.55
58.60
52.65
52.02
39.35
30.35
28.20
19.86
13.48
BocHN
H
N
O
COOMe
S
S
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

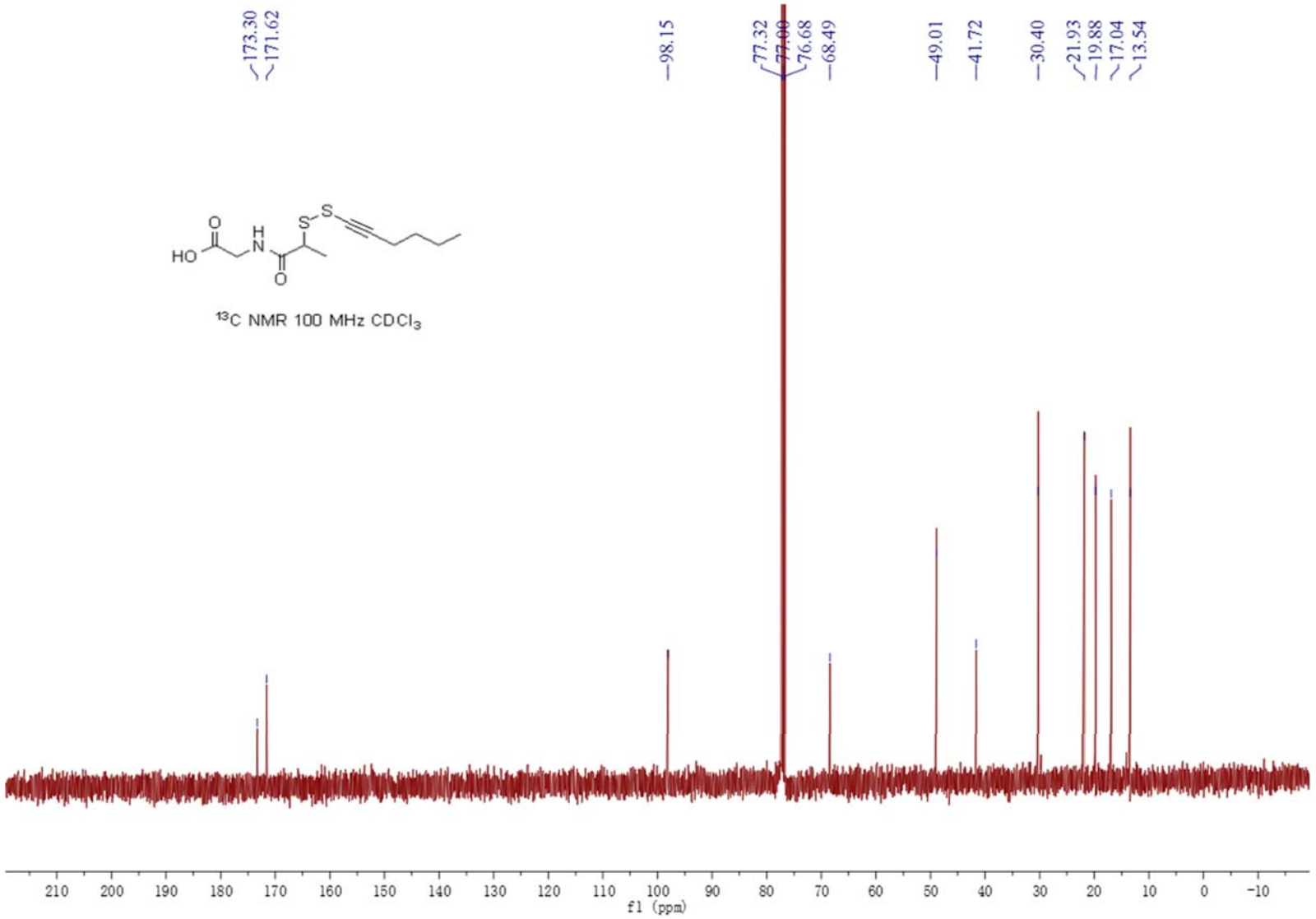
3v


7.64
7.26
6.93
4.94
4.87
4.23
3.75
3.45
3.40
3.34
3.33
2.13
1.87
1.86
1.44
0.90
0.89
0.87
Boc
CO2Me
nBu
1H NMR 400 MHz CDCl3
0.48
0.47
1.00
1.03
3.12
4.18
2.18
1.06
2.46
13.93
3.14
f1 (ppm)

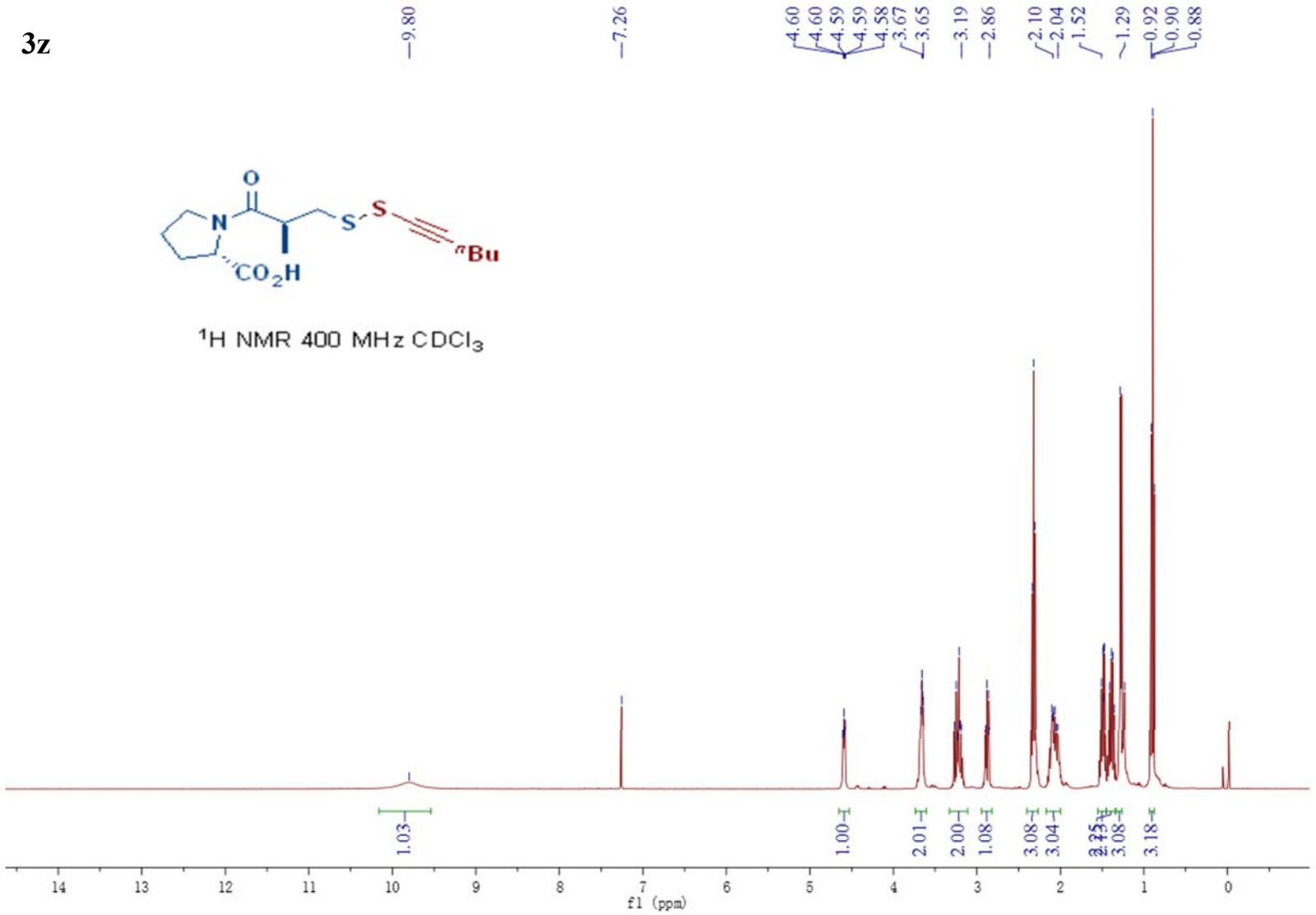

172.56
171.89
170.37
170.07
155.68
154.59
98.75
98.33
80.72
80.42
77.32
77.00
76.68
68.37
59.85
51.49
47.03
39.62
30.35
28.23
23.70
19.85
13.50
Boc
CO2Me
nBu
13C NMR 100 MHz CDCl3
f1 (ppm)

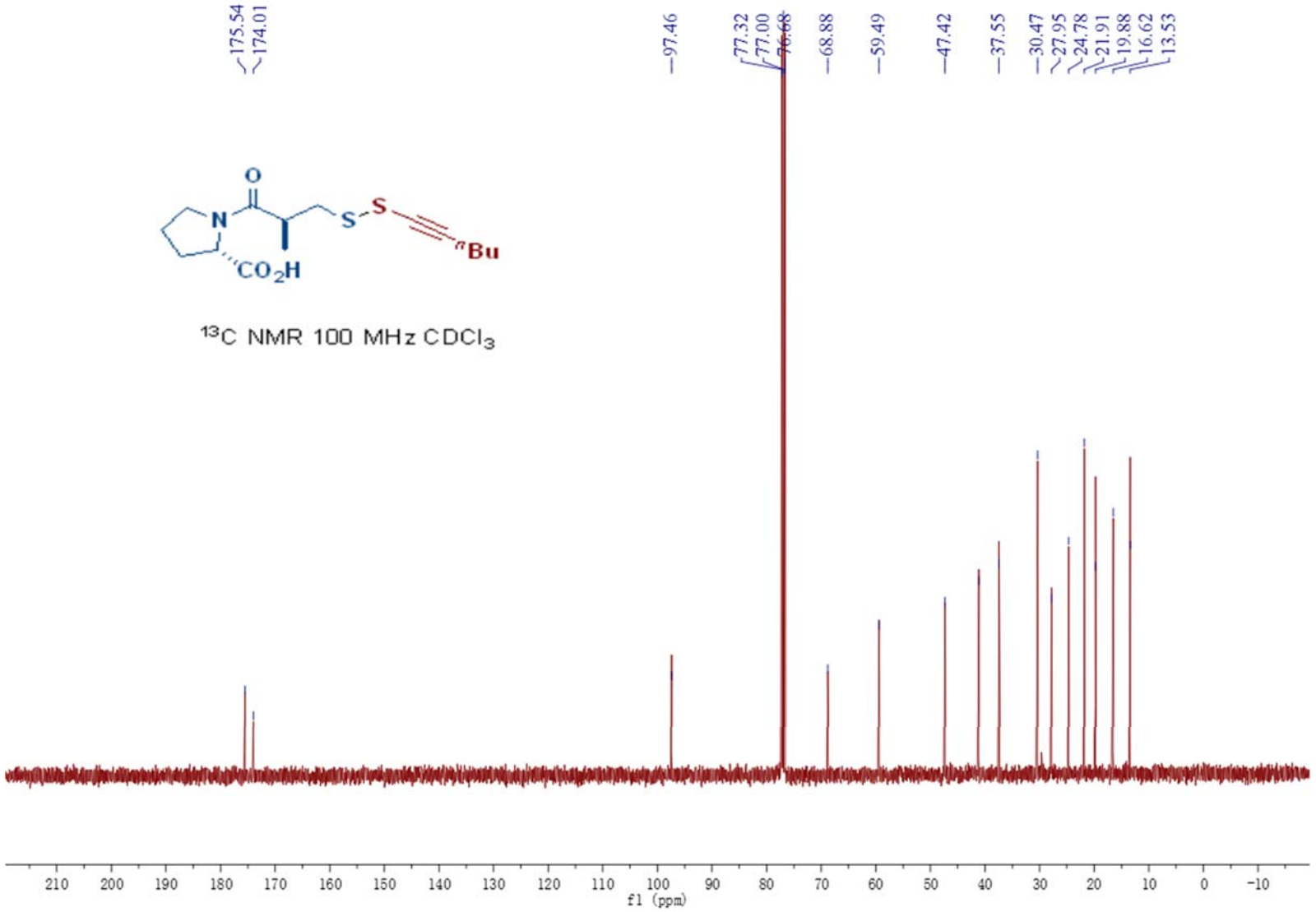
3y


8.815
7.260
6.829
4.140
4.129
4.116
4.082
4.069
3.814
3.796
3.778
3.760
2.345
2.328
2.310
1.606
1.588
0.921
0.902
0.884
HO
O
H
N
O
S
S
1H NMR 400 MHz CDCl3
1.14
1.03
2.04
1.00
2.00
3.00
2.19
2.33
3.09
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

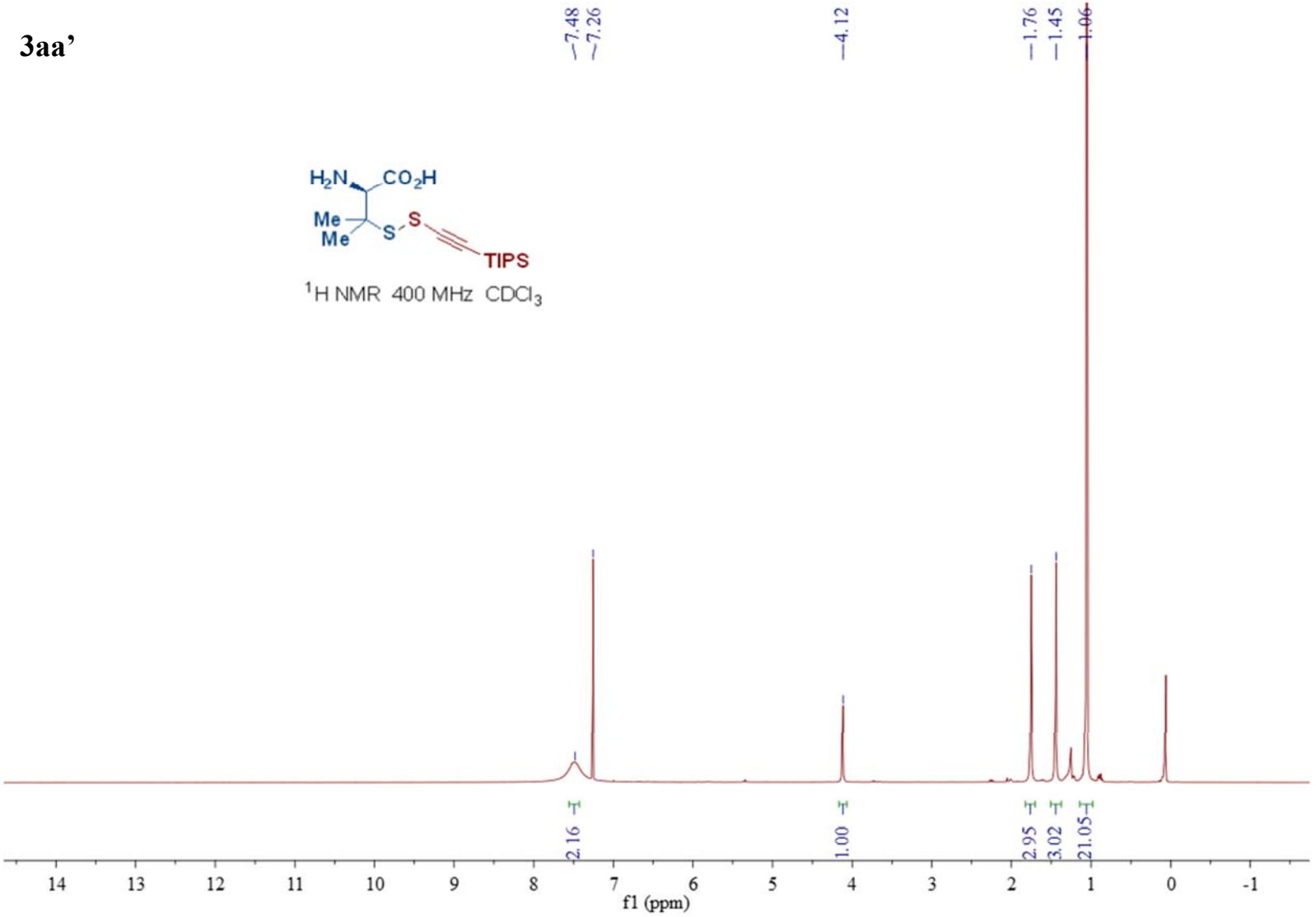

173.30
171.62
98.15
77.32
77.00
76.68
68.49
49.01
41.72
30.40
21.93
19.88
17.04
13.54
HO
O
H
N
S
S
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

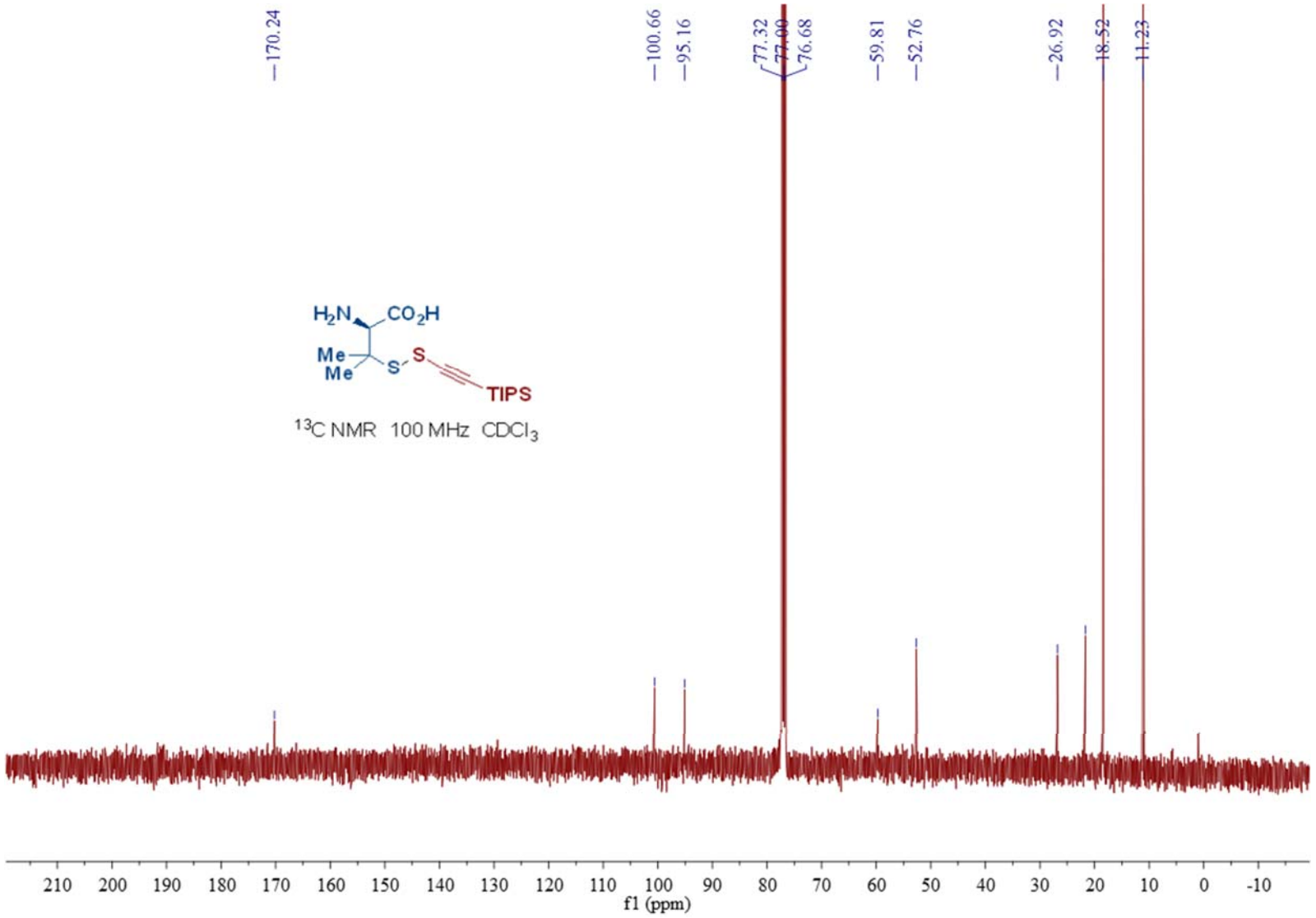
3z


9.80
7.26
4.60
4.60
4.59
4.59
4.58
3.67
3.65
3.19
2.86
2.10
2.04
1.52
1.29
0.92
0.90
0.88
O
N
S
S
nBu
CO2H
1H NMR 400 MHz CDCl3
1.03
1.00
2.01
2.00
1.08
3.08
3.04
3.08
3.18
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

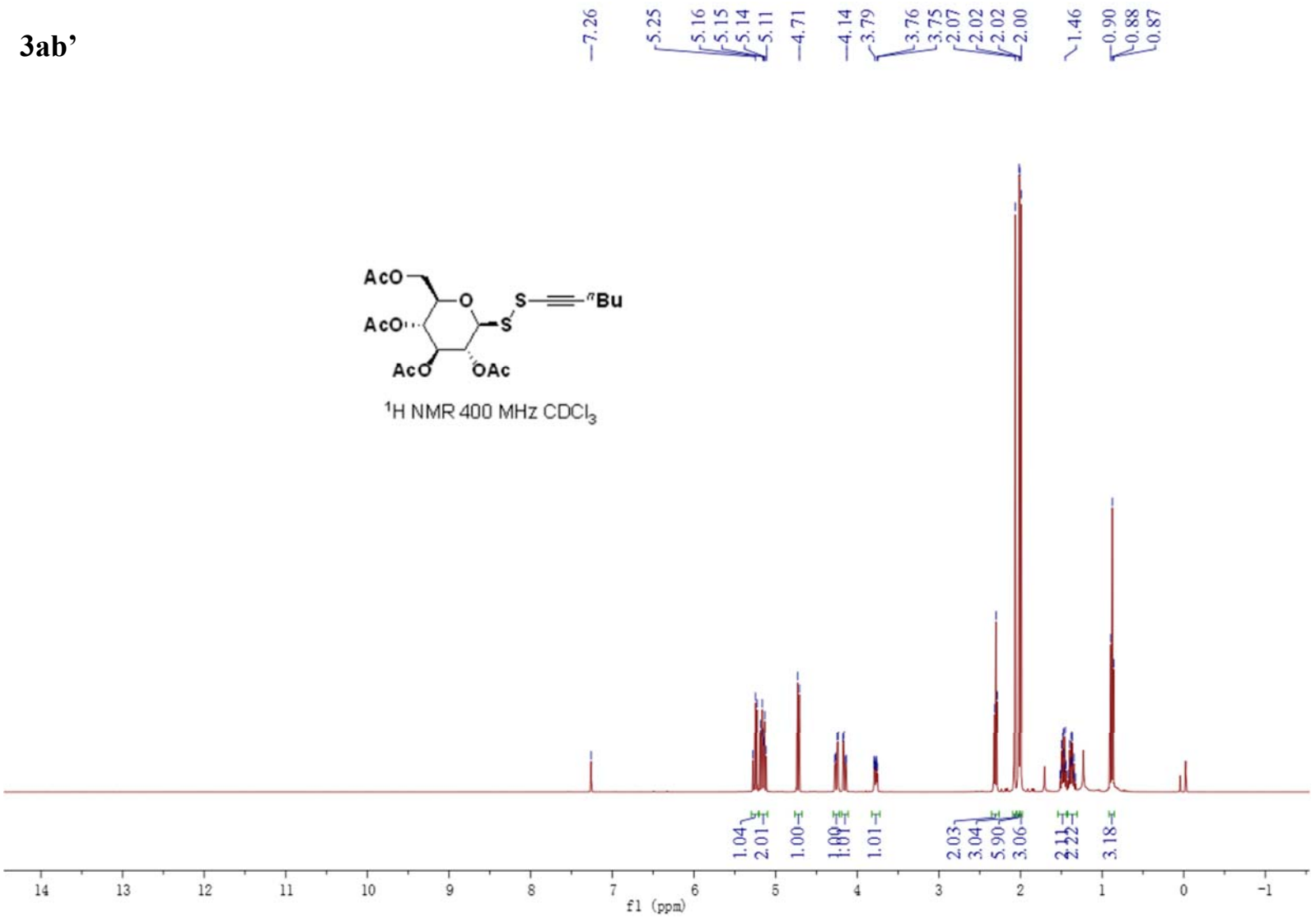

175.54
174.01
97.46
77.32
77.00
76.68
68.88
59.49
47.42
37.55
30.47
27.95
24.78
21.91
19.88
16.62
13.53
O
N
S
S
nBu
CO2H
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3aa'


7.48
7.26
4.12
1.76
1.45
1.06
H2N
CO2H
Me
Me
S
S
TIPS
1H NMR 400 MHz CDCl3
2.16
1.00
2.95
3.02
21.05
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


170.24
100.66
95.16
77.32
77.00
76.68
59.81
52.76
26.92
18.52
11.23
H2N
CO2H
Me
Me
S
S
TIPS
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3ab'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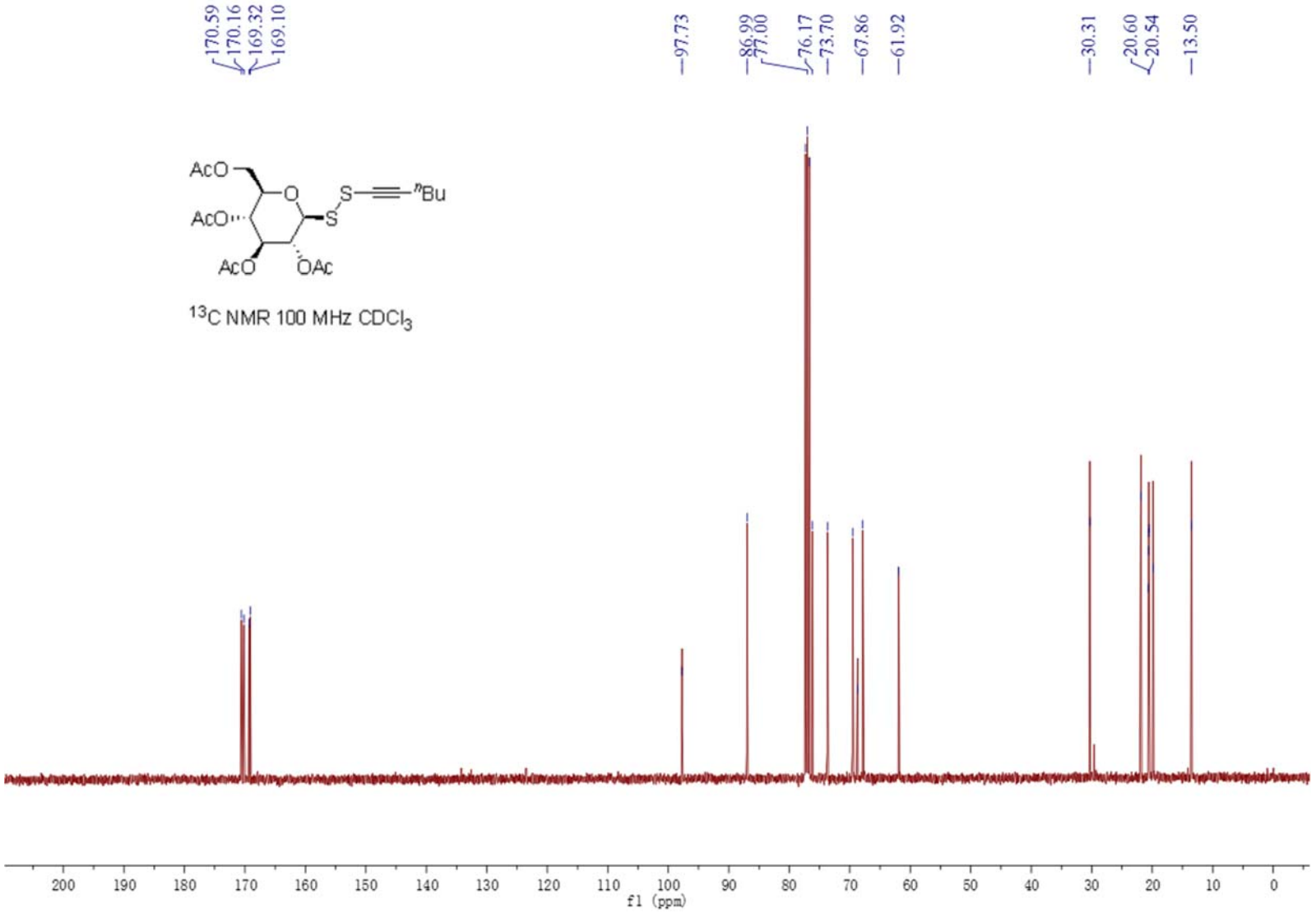
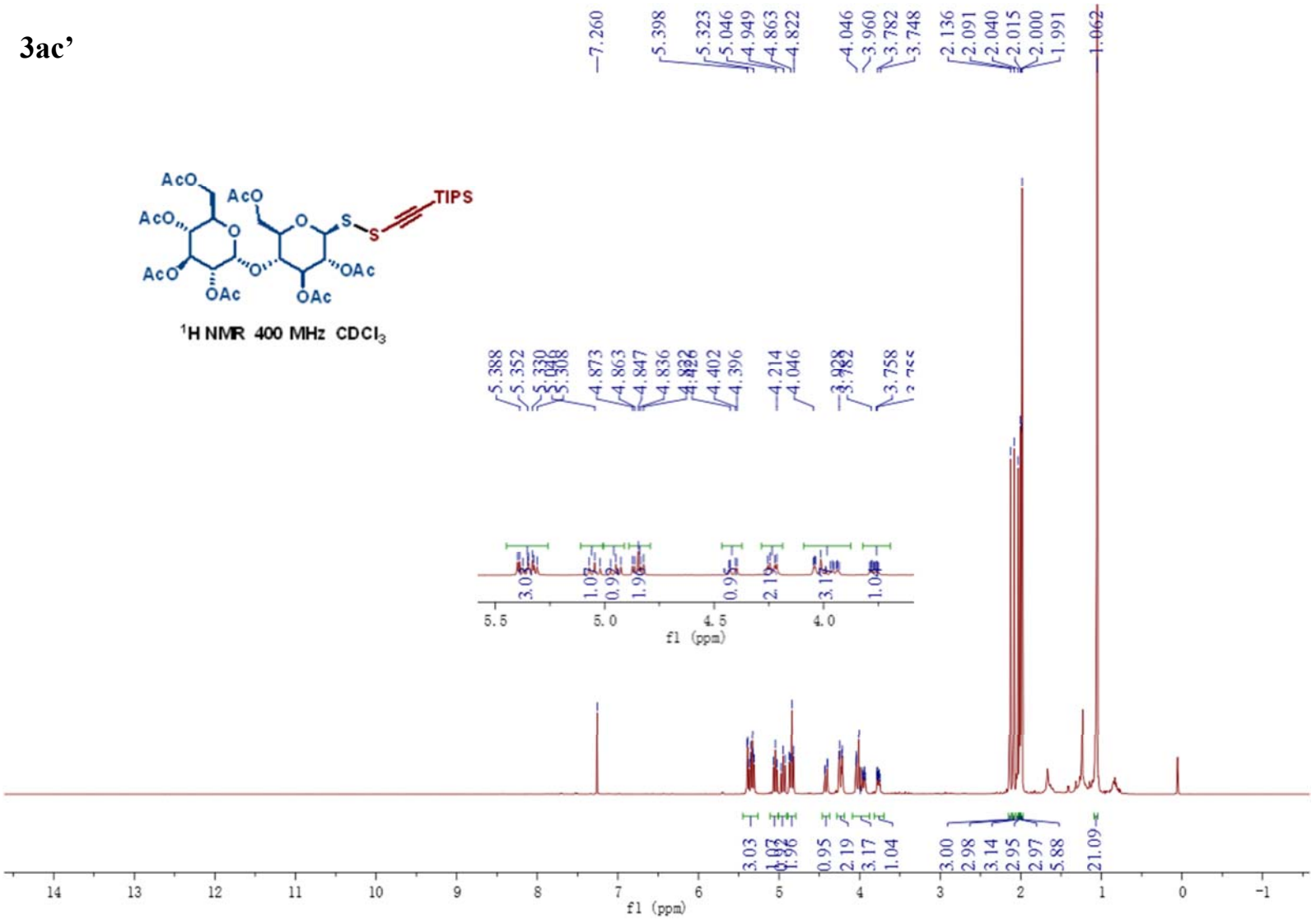
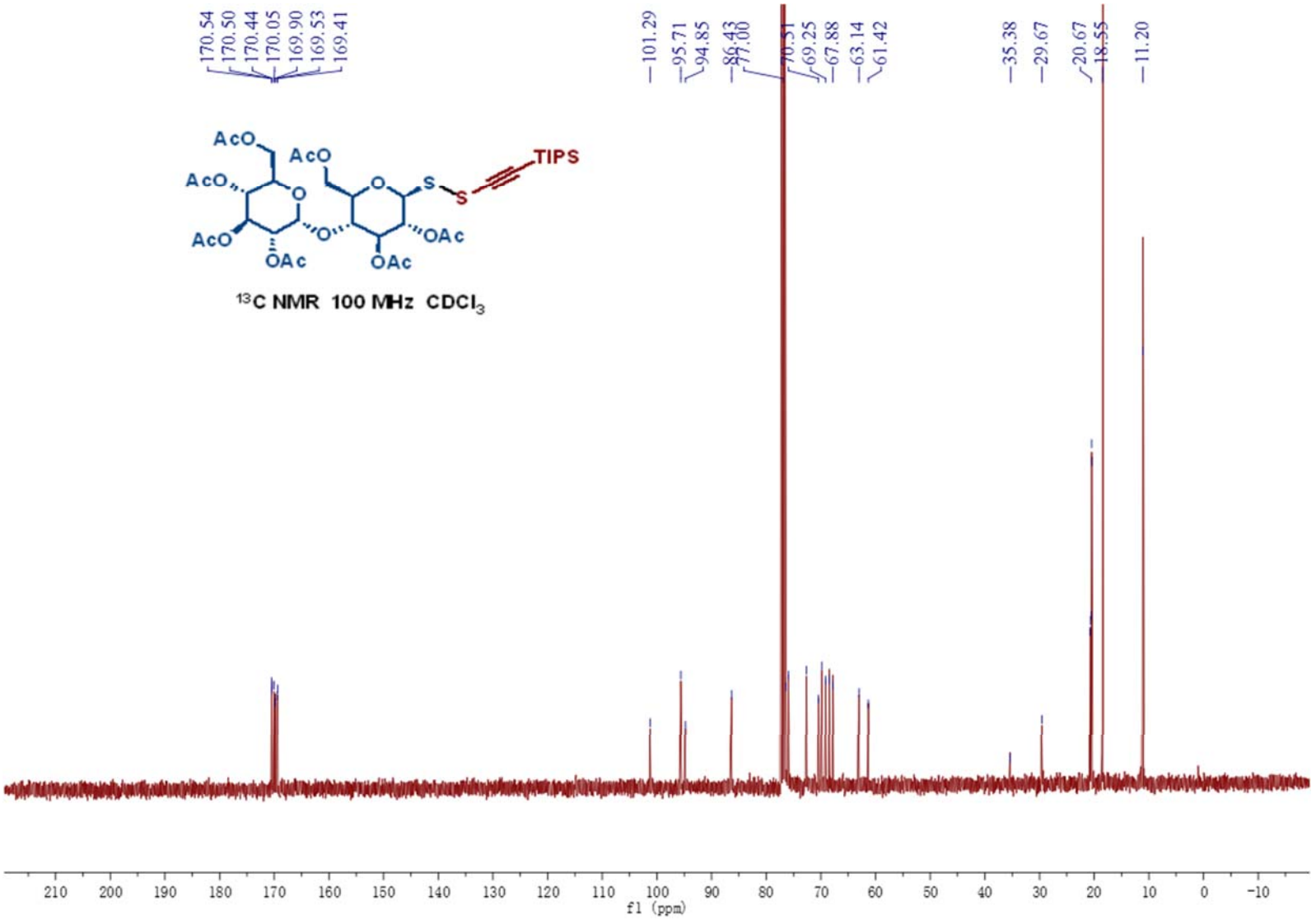
5.25
5.16
5.15
5.14
5.11
4.71
4.14
3.79
3.76
3.75
2.07
2.02
2.02
2.00
1.46
0.90
0.88
0.87
AcO
AcO
AcO
OAc
O
S
S
nBu
1H NMR 400 MHz CDCl3
1.04
2.01
1.00
1.01
2.03
3.04
5.90
3.06
3.18
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

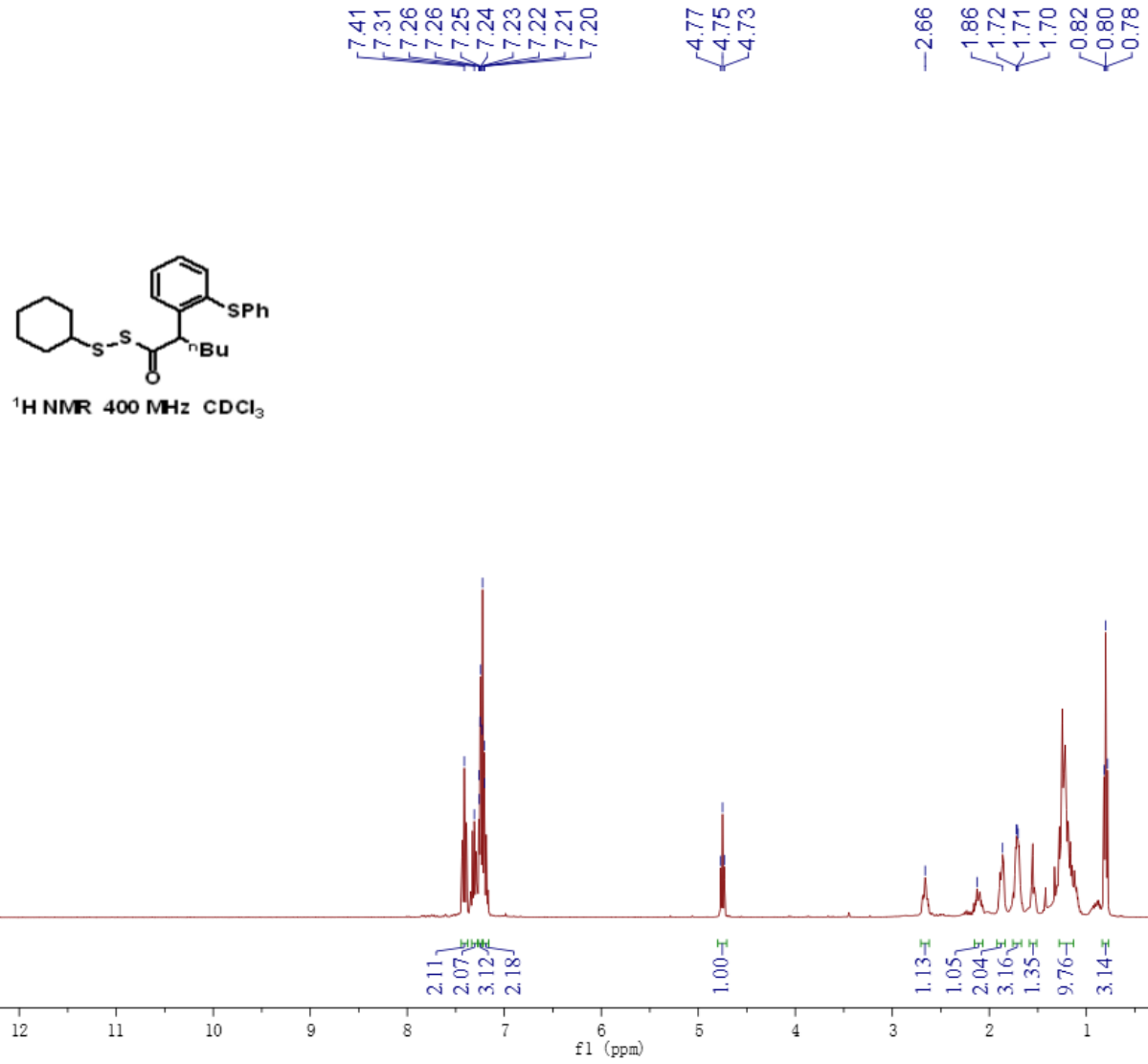

170.59
170.16
169.32
169.10
97.73
86.99
77.00
76.17
73.70
67.86
61.92
30.31
20.60
20.54
13.50
AcO
AcO
AcO
OAc
S
S
nBu
13C NMR 100 MHz CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

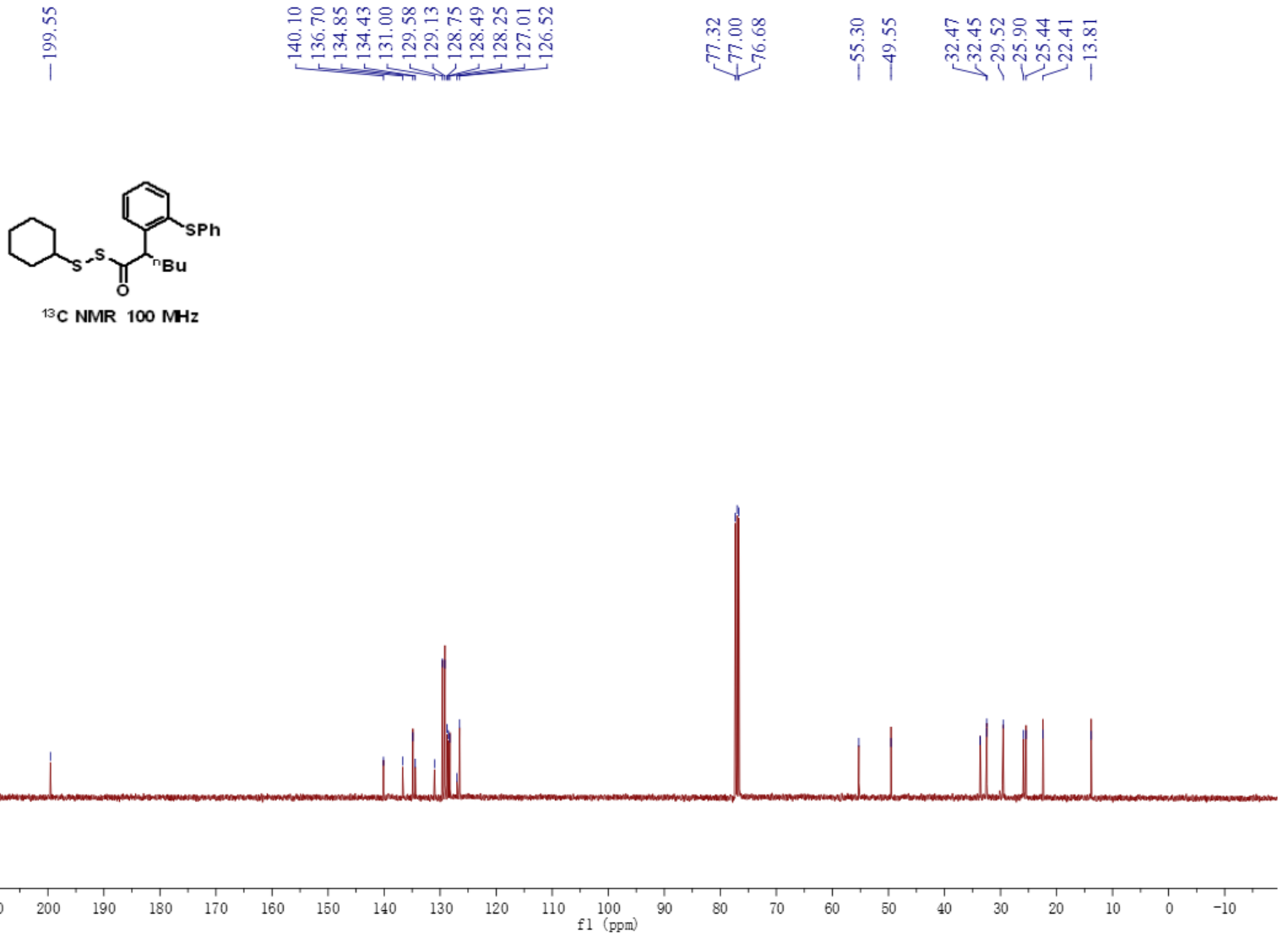
3ac'


AcO
AcO
AcO
AcO
AcO
S
S
TIPS
OAc
OAc
OAc
^{1}H NMR 400 MHz CDCl$_3$
7.260
5.398
5.323
5.046
4.949
4.863
4.822
4.046
3.960
3.782
3.748
2.136
2.091
2.040
2.015
2.000
1.991
1.062
f1 (ppm)

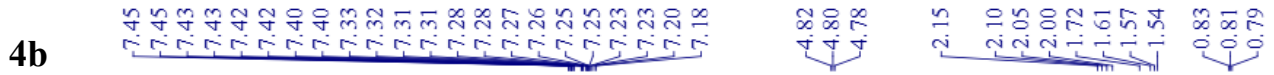

170.54
170.50
170.44
170.05
169.90
169.53
169.41
101.29
95.71
94.85
86.43
77.00
70.51
69.25
67.88
63.14
61.42
35.38
29.67
20.67
18.55
11.20
AcO
AcO
AcO
AcO
O
O
O
S
S
TIPS
OAc
OAc
OAc
13C NMR 100 MHz CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

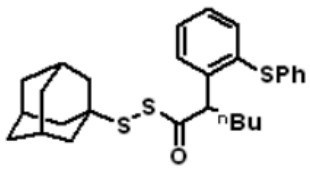
4a


7.41
7.31
7.26
7.26
7.25
7.24
7.23
7.22
7.21
7.20
4.77
4.75
4.73
2.66
1.86
1.72
1.71
1.70
0.82
0.80
0.78
SPh
S
S
nBu
1H NMR 400 MHz CDCl3
2.11
2.07
3.12
2.18
1.00
1.13
1.05
2.04
3.16
1.35
9.76
3.14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

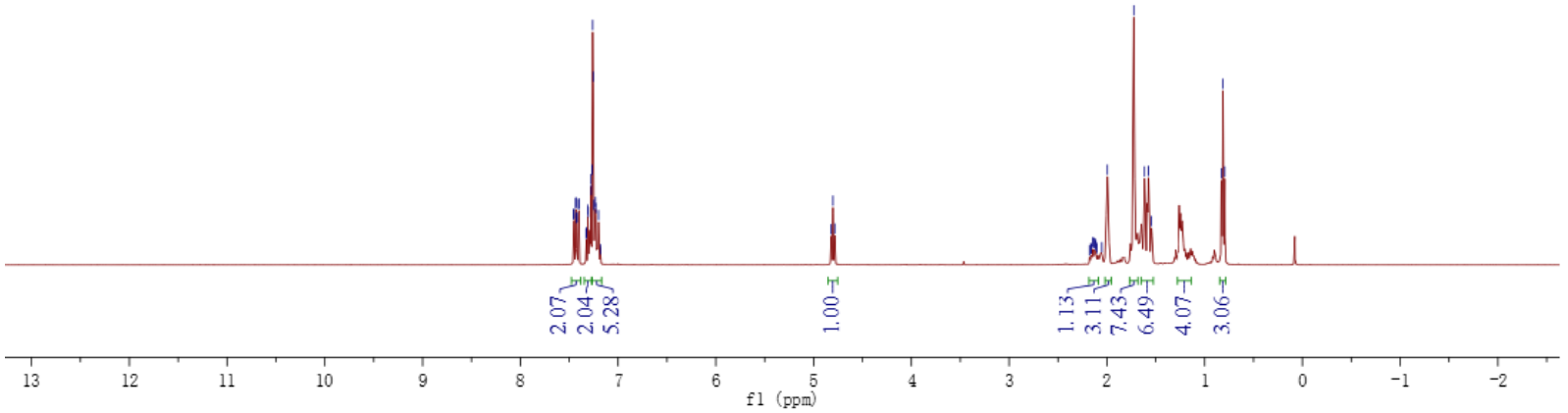

199.55
140.10
136.70
134.85
134.43
131.00
129.58
129.13
128.75
128.49
128.25
127.01
126.52
77.32
77.00
76.68
55.30
49.55
32.47
32.45
29.52
25.90
25.44
22.41
13.81
SPh
S
S
O
nBu
13C NMR 100 MHz
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

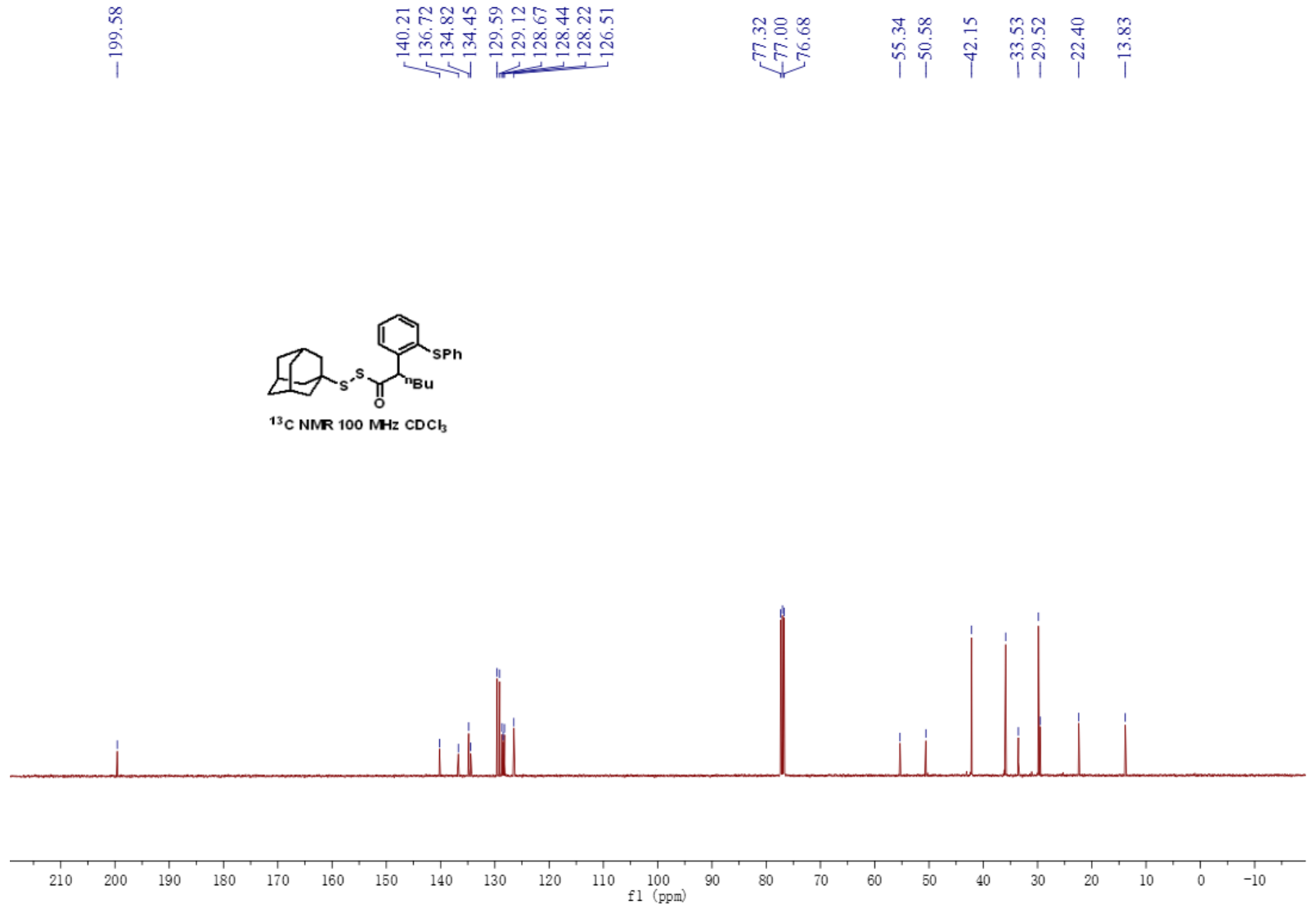
4b

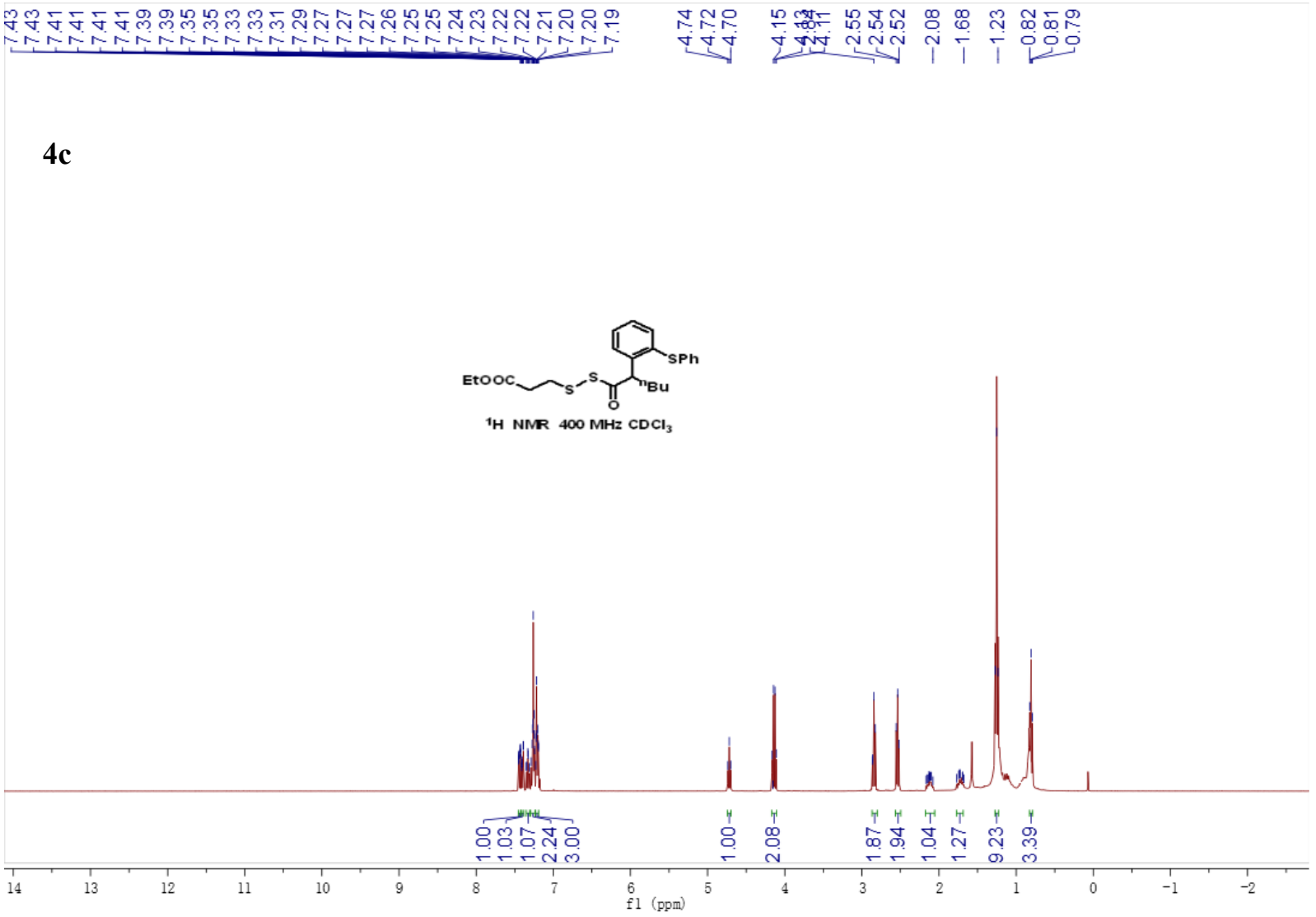


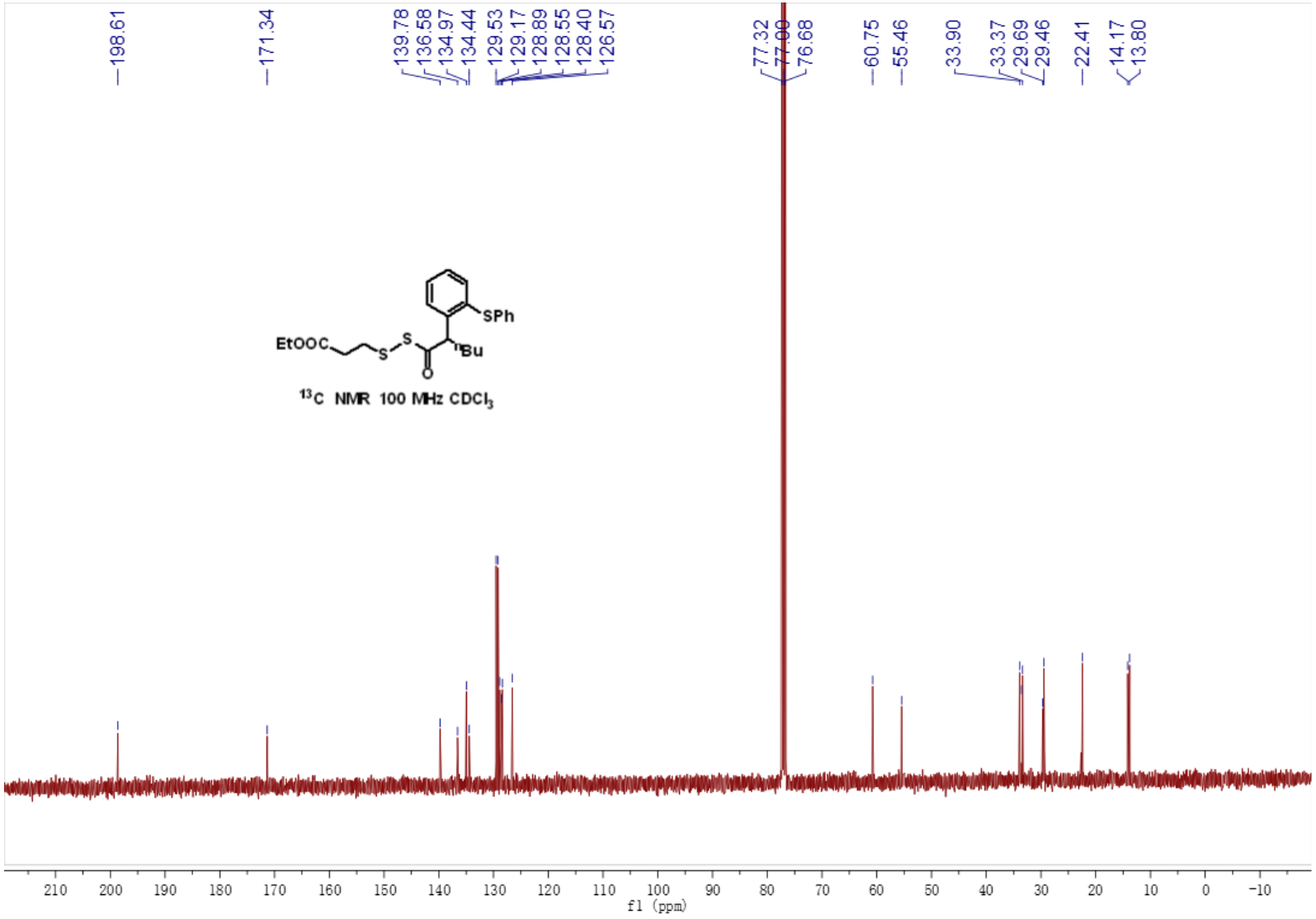


^{1}H NMR 400 MHz $CDCl_3$



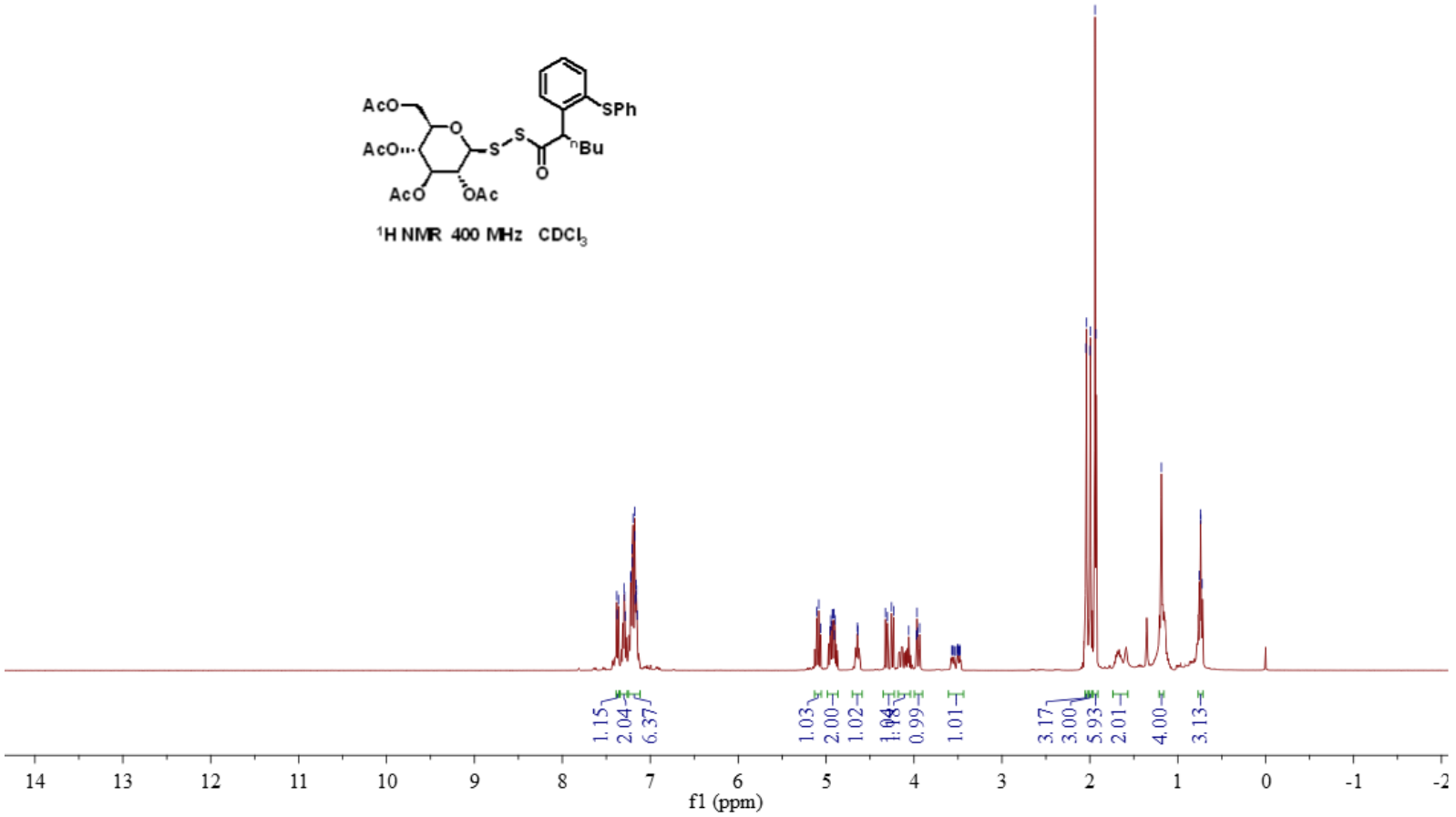

199.58
140.21
136.72
134.82
134.45
129.59
129.12
128.67
128.44
128.22
126.51
77.32
77.00
76.68
55.34
50.58
42.15
33.53
29.52
22.40
13.83
SPh
nBu
O
13C NMR 100 MHz CDCl3
f1 (ppm)

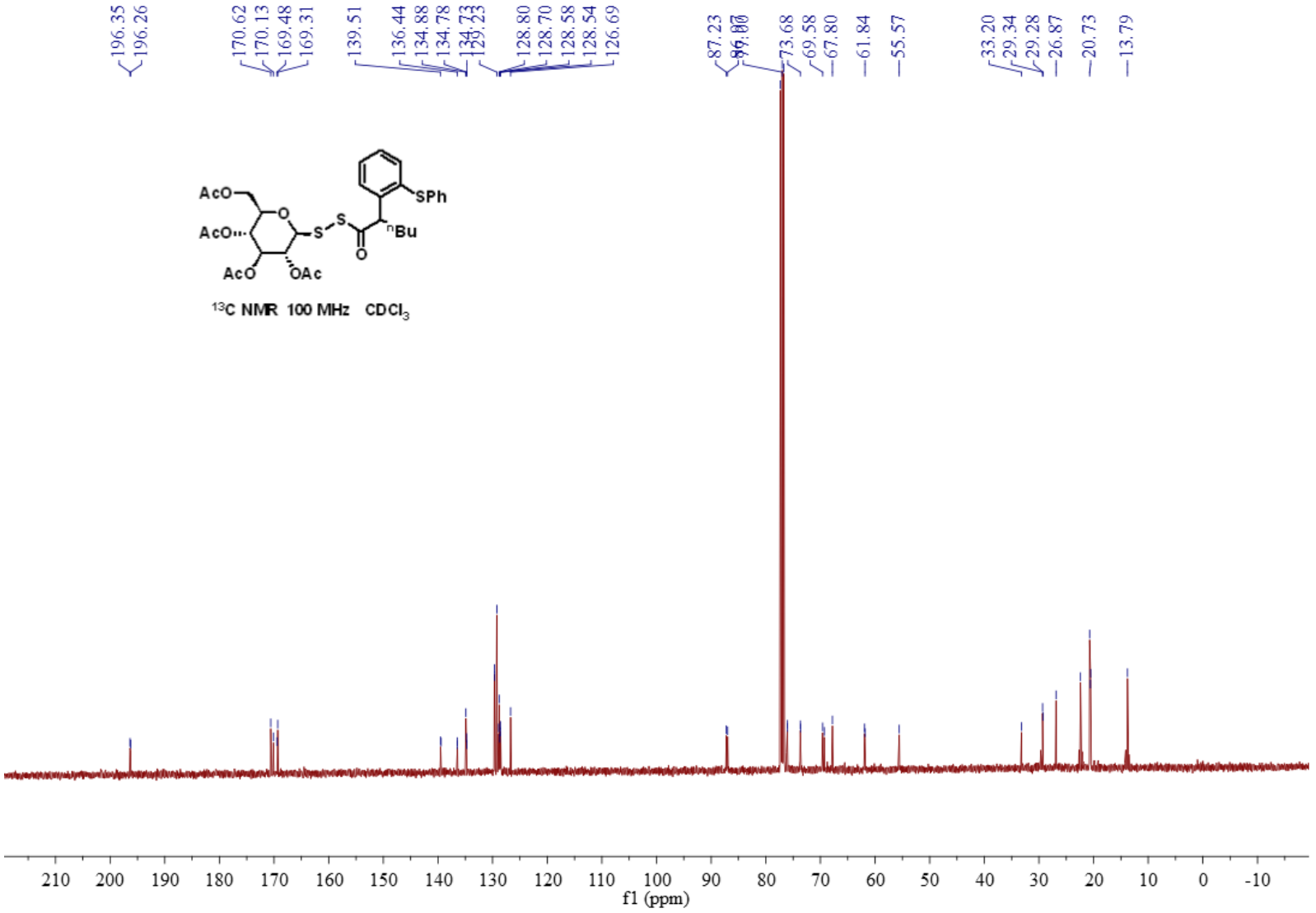

7.43
7.43
7.41
7.41
7.41
7.41
7.39
7.39
7.35
7.35
7.33
7.33
7.31
7.29
7.27
7.27
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.22
7.21
7.20
7.20
7.19
4.74
4.72
4.70
4.15
4.11
2.55
2.54
2.52
2.08
1.68
1.23
0.82
0.81
0.79
4c
EtOOC
SPh
nBu
1H NMR 400 MHz CDCl3
1.00
1.03
1.07
2.24
3.00
1.00
2.08
1.87
1.94
1.04
1.27
9.23
3.39
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
f1 (ppm)


198.61
171.34
139.78
136.58
134.97
134.44
129.53
129.17
128.89
128.55
128.40
126.57
77.32
77.00
76.68
60.75
55.46
33.90
33.37
29.69
29.46
22.41
14.17
13.80
SPh
EtOOC
S
S
tBu
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

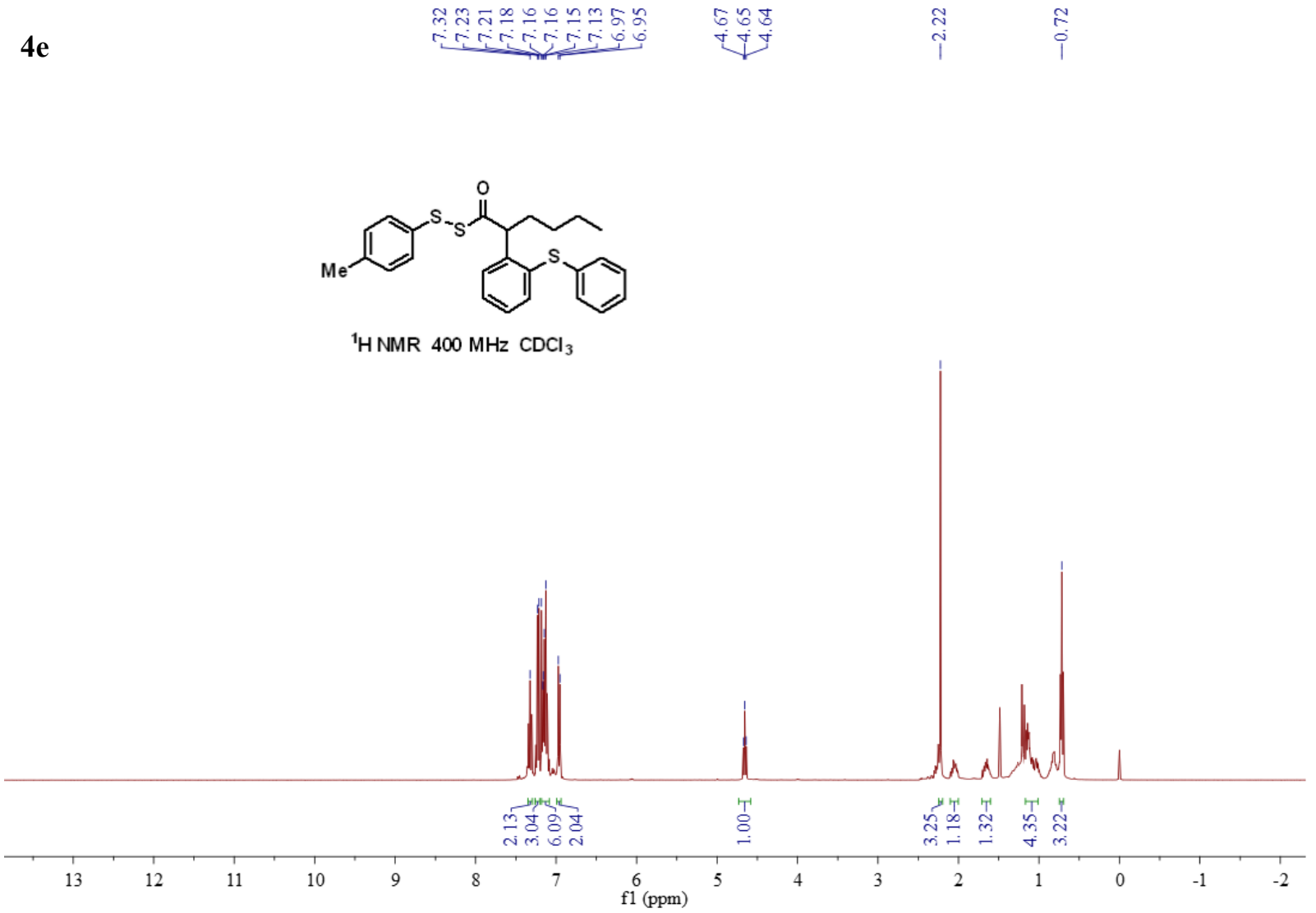

7.38
7.38
7.36
7.30
7.29
7.29
7.22
7.22
7.21
7.20
7.20
7.18
7.17
7.17
7.16
7.16
7.16
7.15
5.10
4.95
4.93
4.91
4.90
4.64
4.33
3.94
3.57
3.50
2.00
1.99
1.94
1.93
1.19
0.75
0.74
0.74
0.72

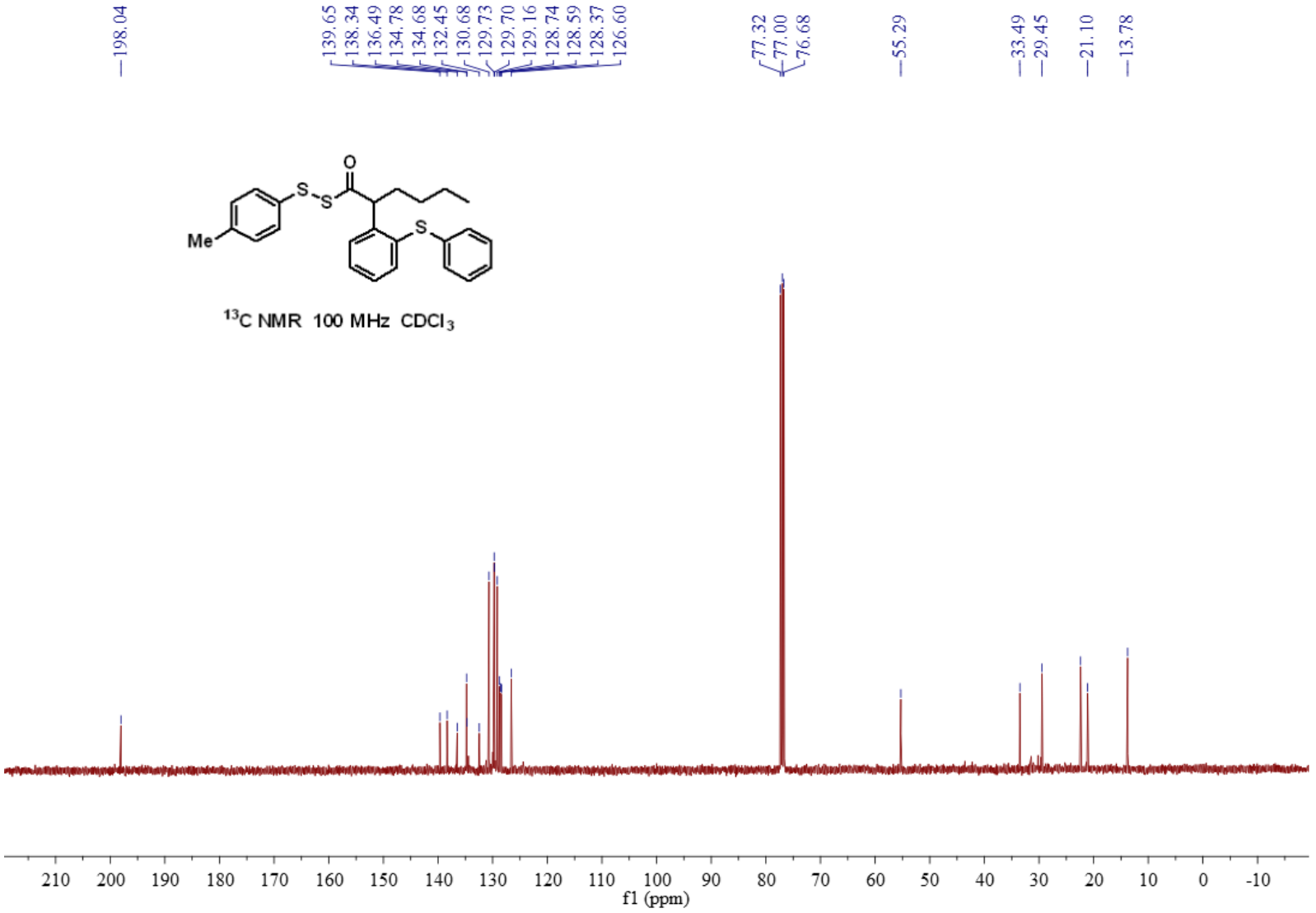
4d

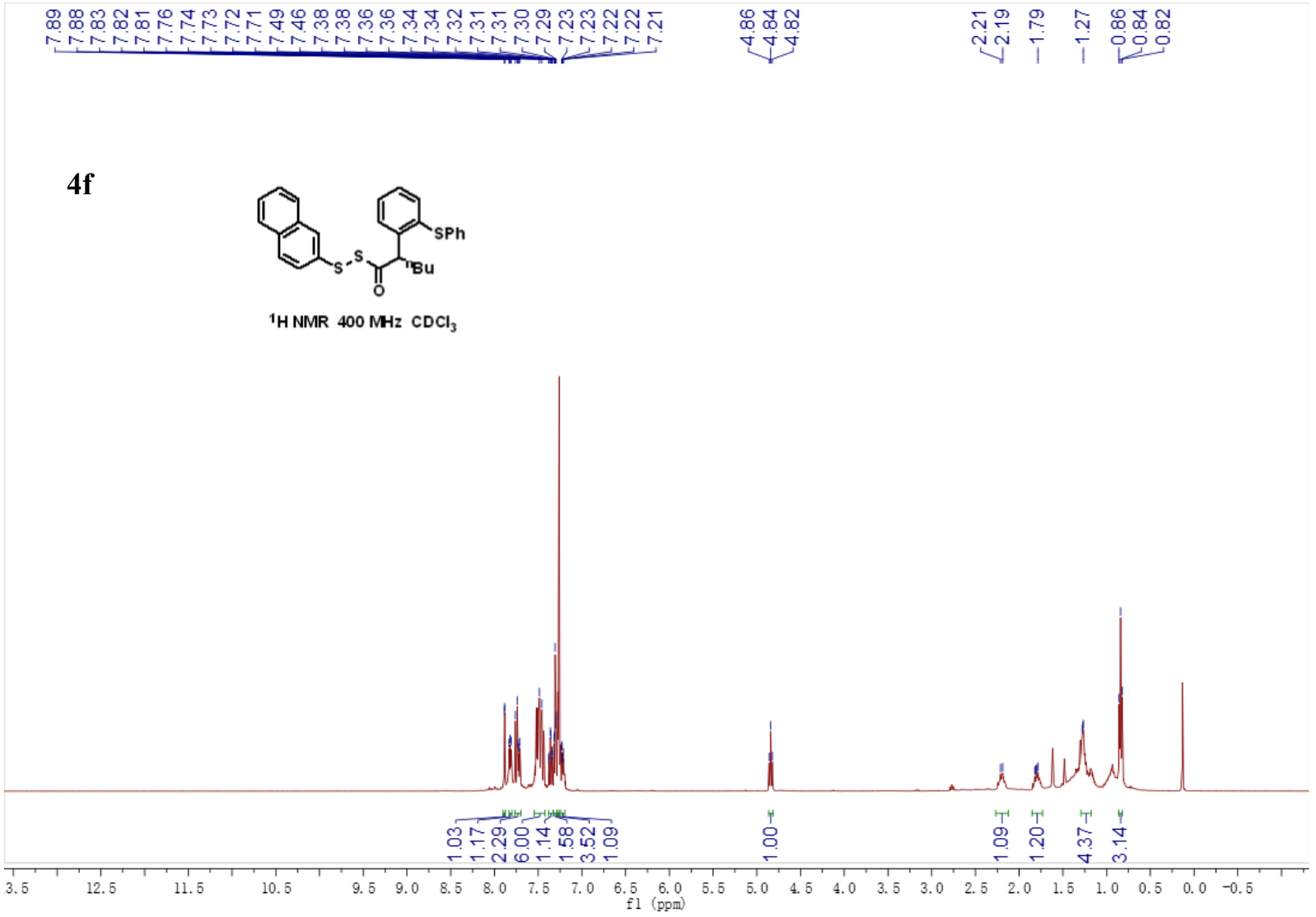

AcO
AcO
AcO
OAc
O
S
S
O
SPh
nBu
1H NMR 400 MHz CDCl3
1.15
2.04
6.37
1.03
2.00
1.02
0.99
1.01
3.17
3.00
5.93
2.01
4.00
3.13
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

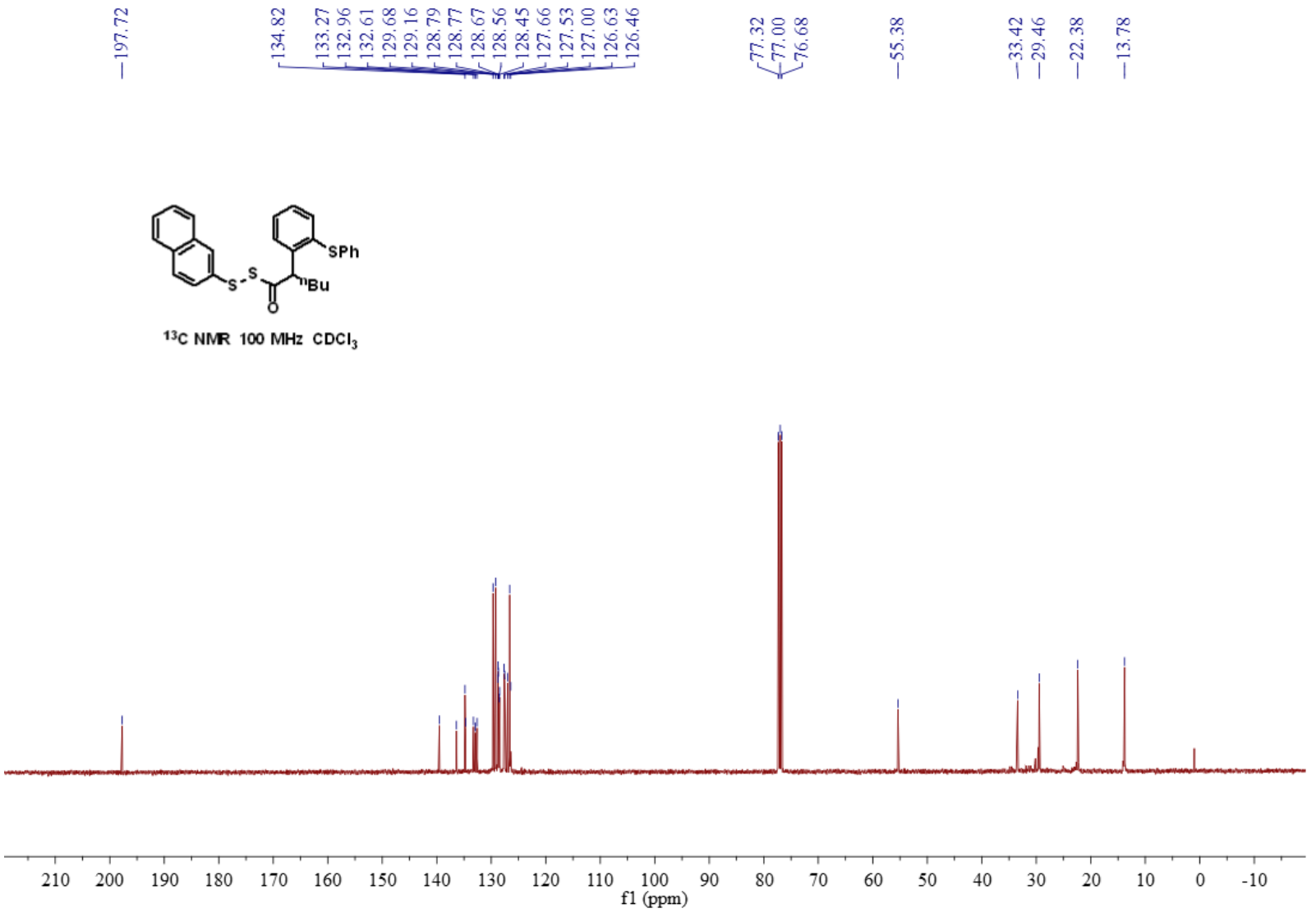

196.35
196.26
170.62
170.13
169.48
169.31
139.51
136.44
134.88
134.78
134.73
129.23
128.80
128.70
128.58
128.54
126.69
87.23
86.80
73.68
69.58
67.80
61.84
55.57
33.20
29.34
29.28
26.87
20.73
13.79
AcO
AcO
AcO
OAc
O
S
S
O
SPh
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4e


7.32
7.23
7.21
7.18
7.16
7.16
7.15
7.13
6.97
6.95
4.67
4.65
4.64
2.22
0.72
Me
S
S
O
S
^{1}H NMR 400 MHz CDCl$_3$
2.13
3.04
6.09
2.04
1.00
3.25
1.18
1.32
4.35
3.22
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


198.04
139.65
138.34
136.49
134.78
134.68
132.45
130.68
129.73
129.70
129.16
128.74
128.59
128.37
126.60
77.32
77.00
76.68
55.29
33.49
29.45
21.10
13.78
Me
S
S
O
S
^{13}C NMR 100 MHz CDCl$_3$
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.89
7.88
7.83
7.82
7.81
7.76
7.74
7.73
7.72
7.71
7.49
7.46
7.38
7.38
7.36
7.36
7.34
7.34
7.32
7.31
7.31
7.30
7.29
7.23
7.23
7.22
7.22
7.21
4.86
4.84
4.82
2.21
2.19
1.79
1.27
0.86
0.84
0.82
4f
SPh
S
S
tBu
O
1H NMR 400 MHz CDCl3
1.03
1.17
2.29
6.00
1.14
1.58
3.52
1.09
1.00
1.09
1.20
4.37
3.14
f1 (ppm)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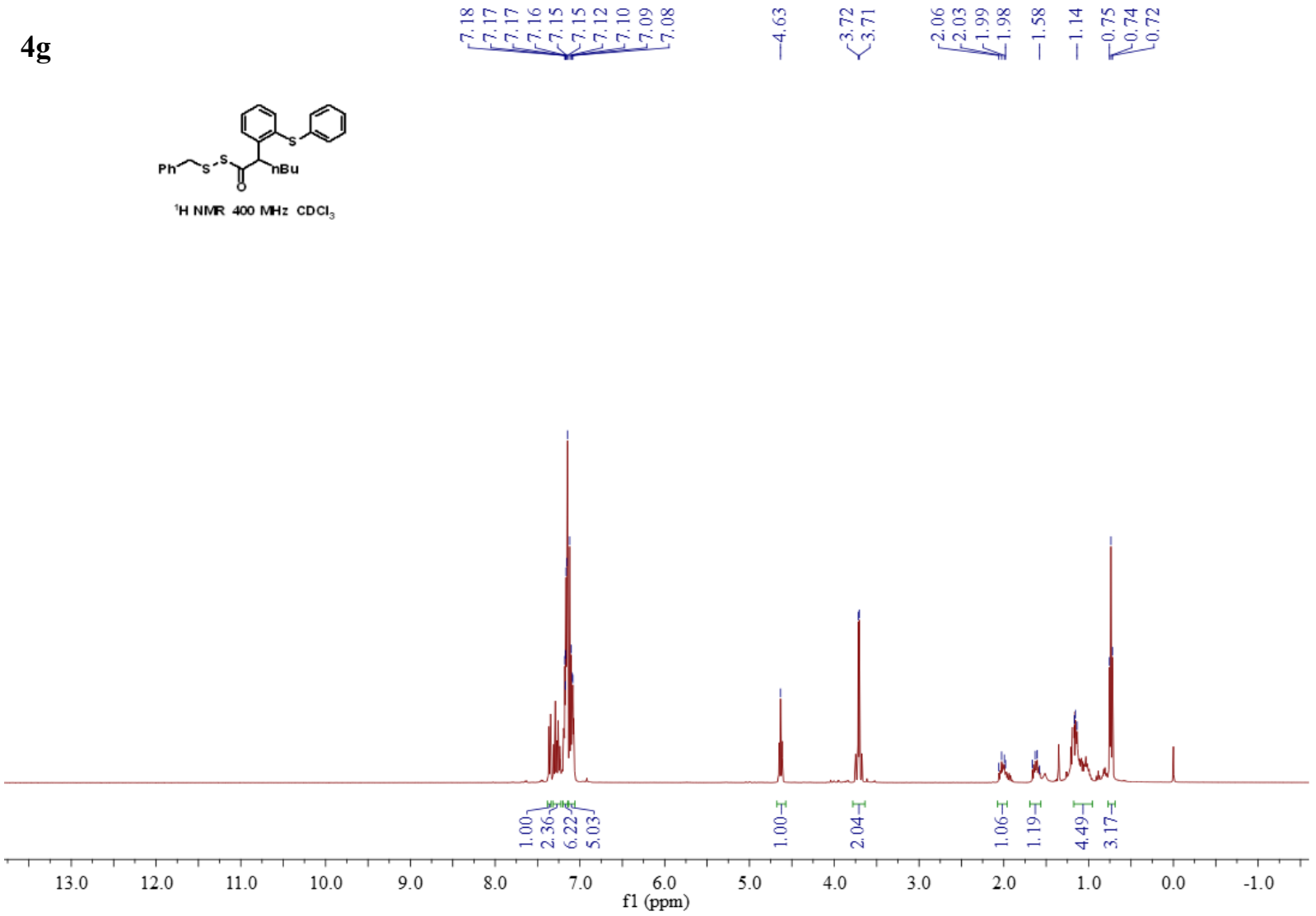
134.82
133.27
132.96
132.61
129.68
129.16
128.79
128.77
128.67
128.56
128.45
127.66
127.53
127.00
126.63
126.46
77.32
77.00
76.68
55.38
33.42
29.46
22.38
13.78
SPh
S
S
nBu
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4g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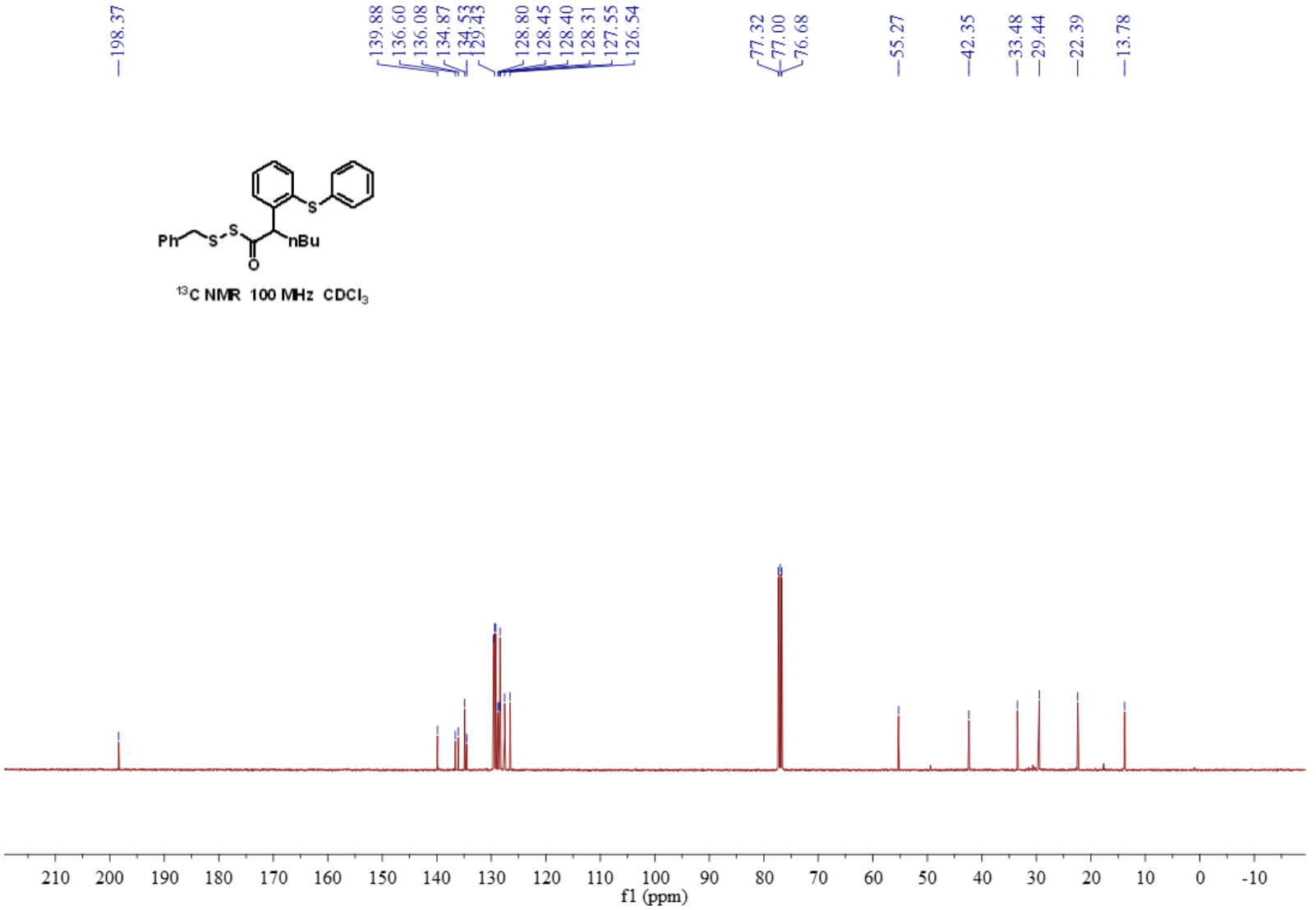
7.17
7.17
7.16
7.15
7.15
7.12
7.10
7.09
7.08
4.63
3.72
3.71
2.06
2.03
1.99
1.98
1.58
1.14
0.75
0.74
0.72
Ph
S
S
S
O
nBu
^{1}H NMR 400 MHz CDCl$_3$
1.00
2.36
6.22
5.03
1.00
2.04
1.06
1.19
4.49
3.17
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)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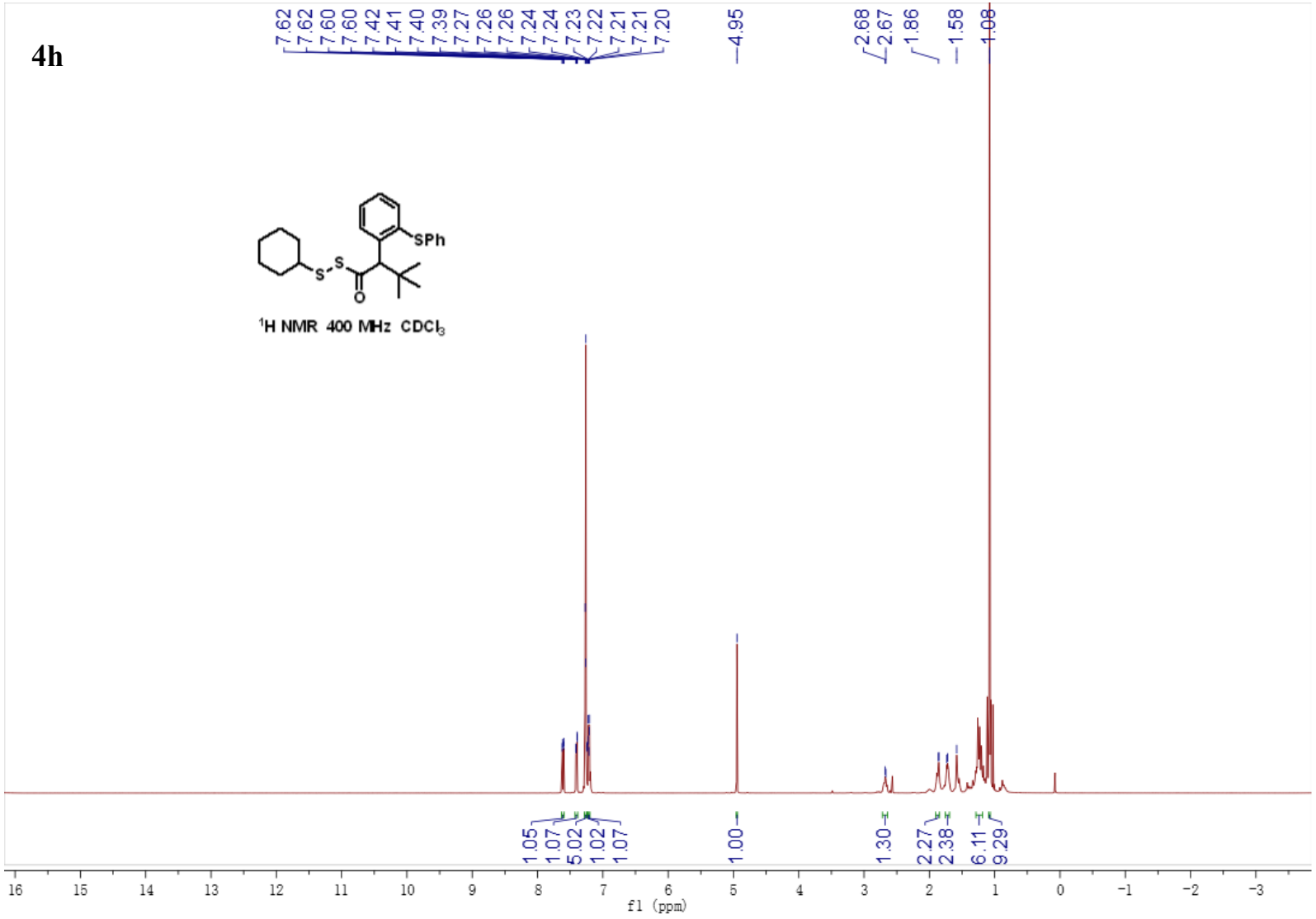
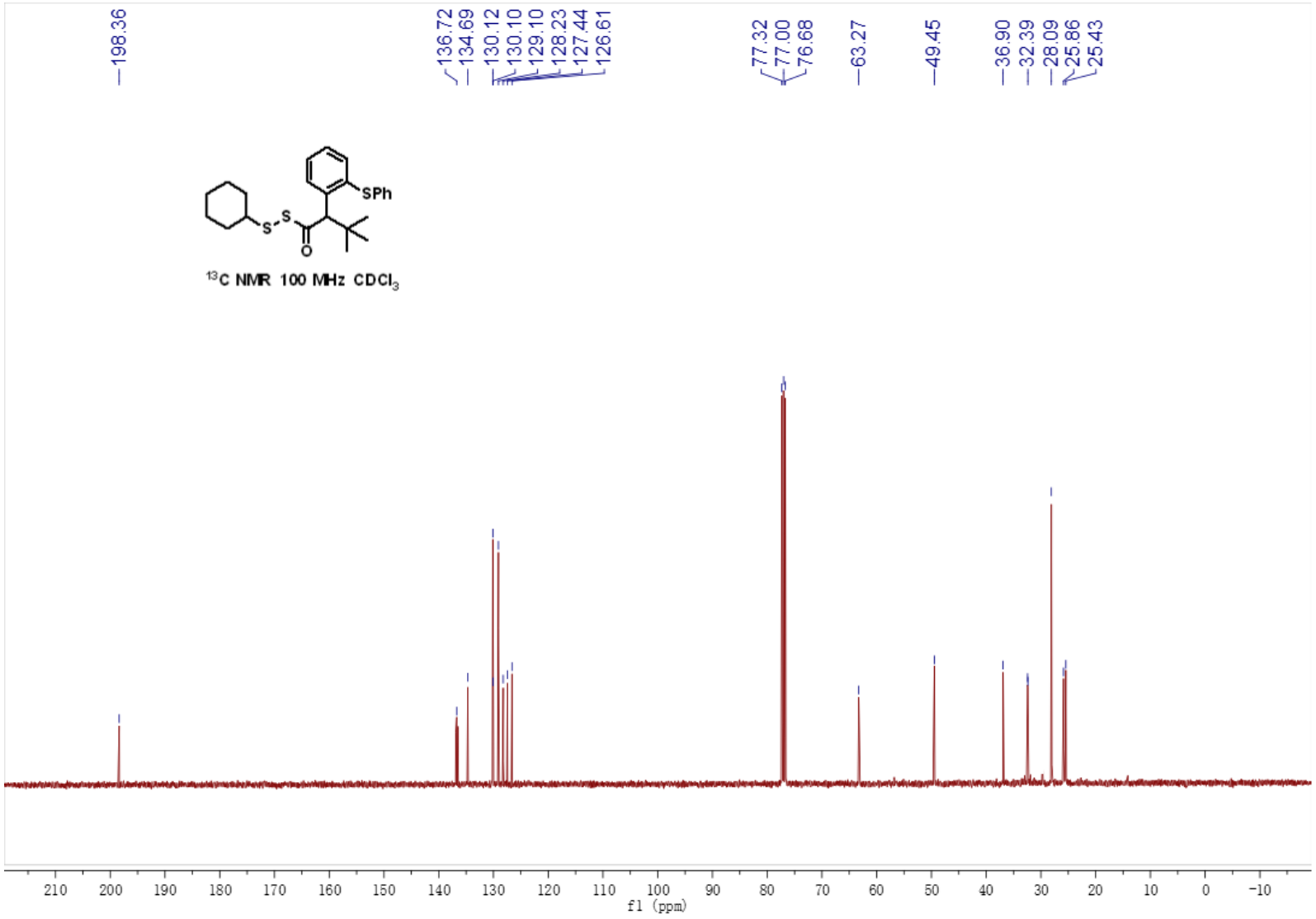
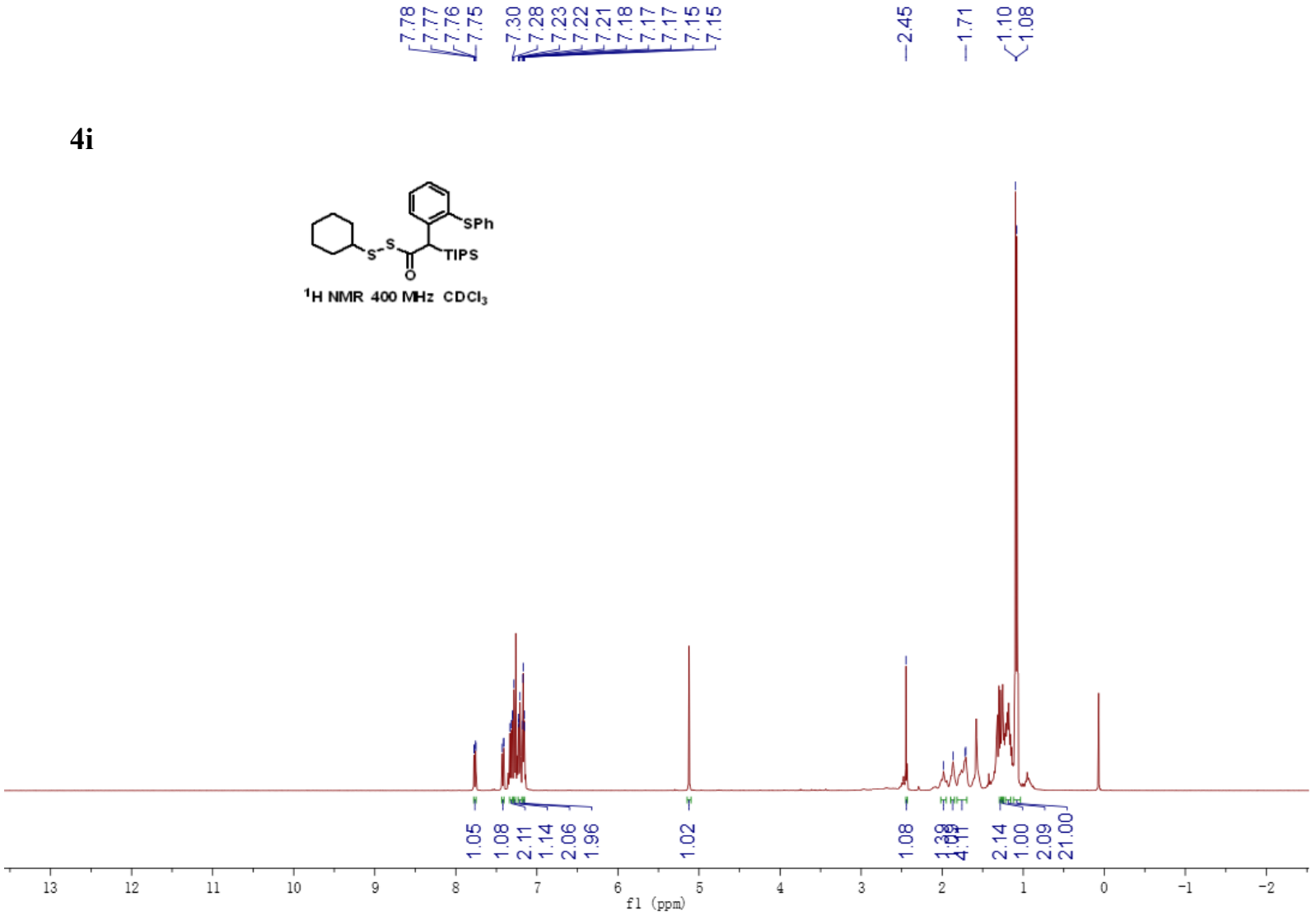
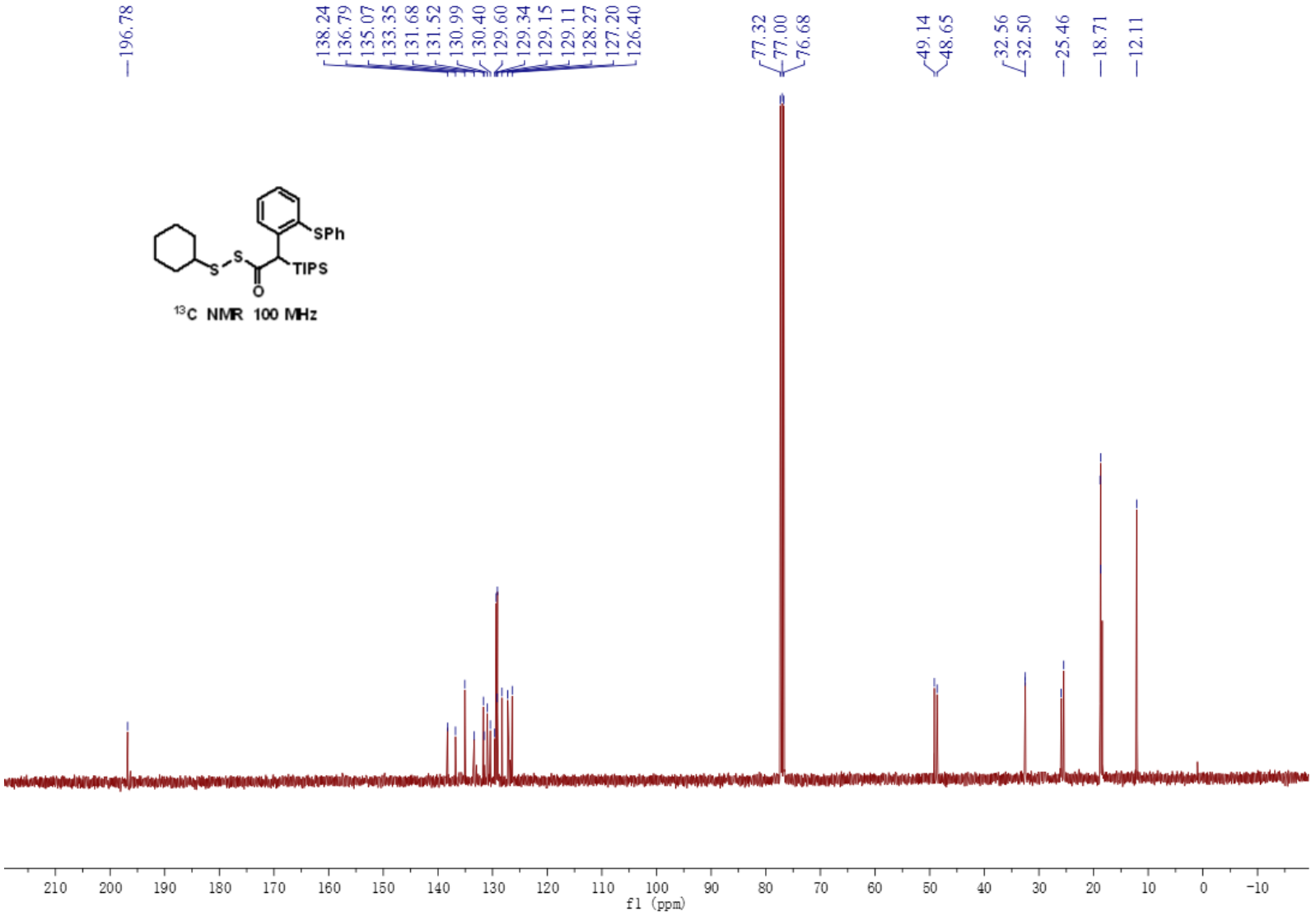
139.88
136.60
136.08
134.87
134.53
129.43
128.80
128.45
128.40
128.31
127.55
126.54
77.32
77.00
76.68
55.27
42.35
33.48
29.44
22.39
13.78
Ph
S
S
S
nBu
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


4h
7.62
7.62
7.60
7.60
7.42
7.41
7.40
7.39
7.27
7.26
7.26
7.24
7.24
7.23
7.22
7.21
7.21
7.20
4.95
2.68
2.67
1.86
1.58
1.08
SPh
S
S
O
^{1}H NMR 400 MHz CDCl$_3$
1.05
1.07
5.02
1.02
1.07
1.00
1.30
2.27
2.38
6.11
9.29
f1 (ppm)

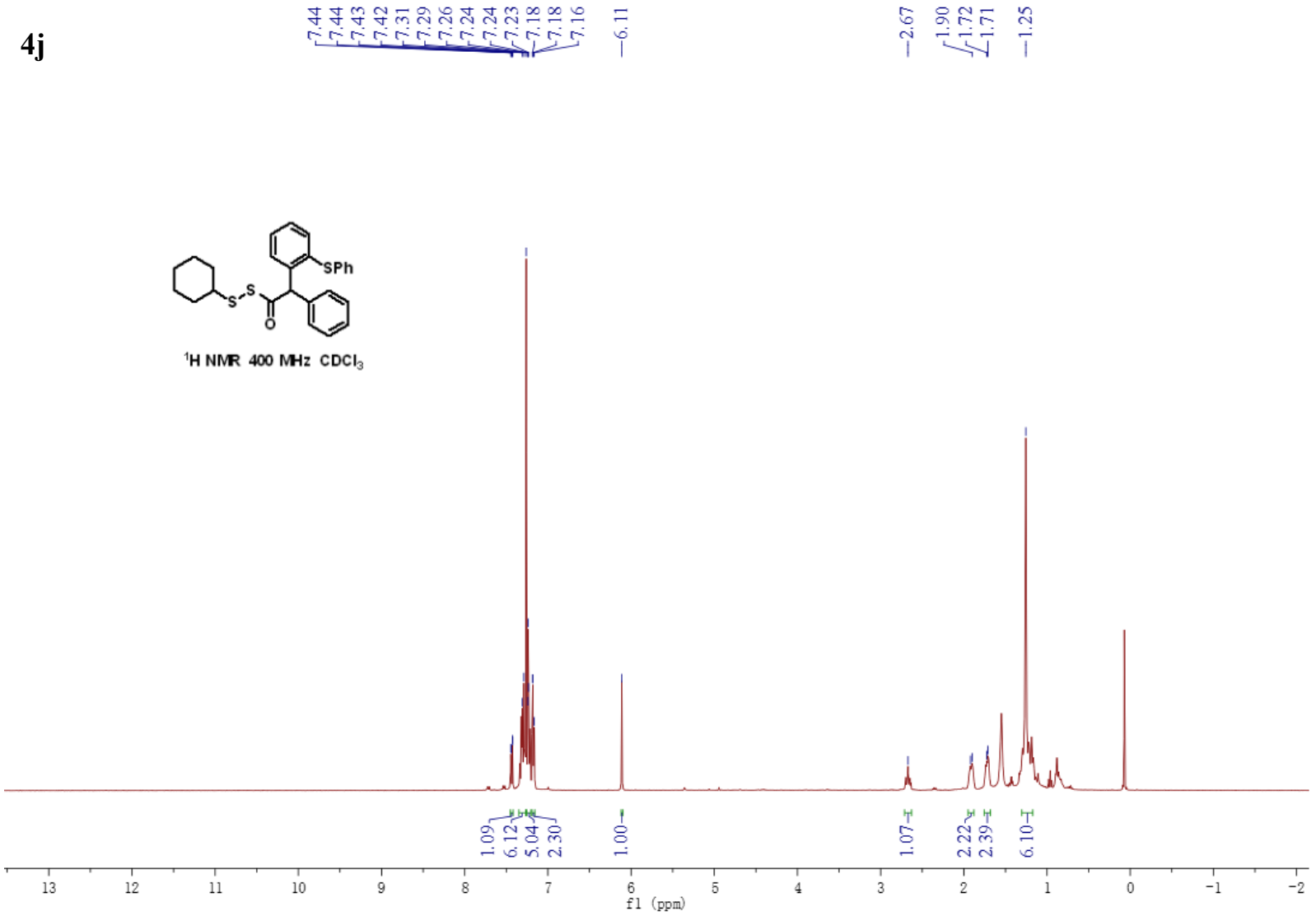

198.36
136.72
134.69
130.12
130.10
129.10
128.23
127.44
126.61
77.32
77.00
76.68
63.27
49.45
36.90
32.39
28.09
25.86
25.43
SPh
S
S
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4i


7.78
7.77
7.76
7.75
7.30
7.28
7.23
7.22
7.21
7.18
7.17
7.17
7.15
7.15
2.45
1.71
1.10
1.08
SPh
S
S
TIPS
O
^{1}H NMR 400 MHz CDCl$_3$
1.05
1.08
2.11
1.14
2.06
1.96
1.02
1.08
2.14
1.00
2.09
21.00
f1 (ppm)


196.78
138.24
136.79
135.07
133.35
131.68
131.52
130.99
130.40
129.60
129.34
129.15
129.11
128.27
127.20
126.40
77.32
77.00
76.68
49.14
48.65
32.56
32.50
25.46
18.71
12.11
SPh
TIPS
S
S
O
13C NMR 100 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4j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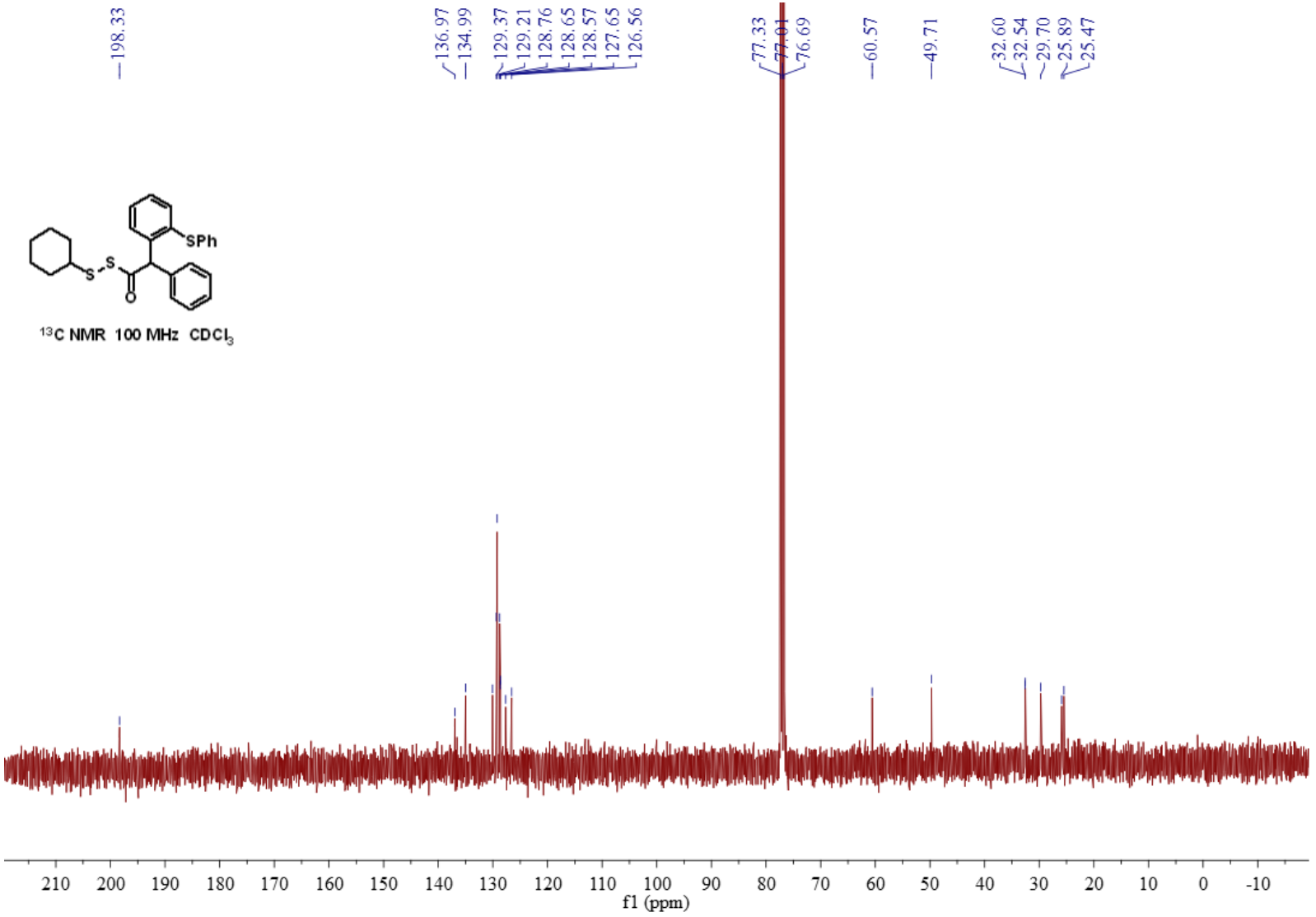
7.18
7.16
6.11
2.67
1.90
1.72
1.71
1.25
SPh
S
S
O
^{1}H NMR 400 MHz CDCl$_3$
1.09
6.12
5.04
2.30
1.00
1.07
2.22
2.39
6.10
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)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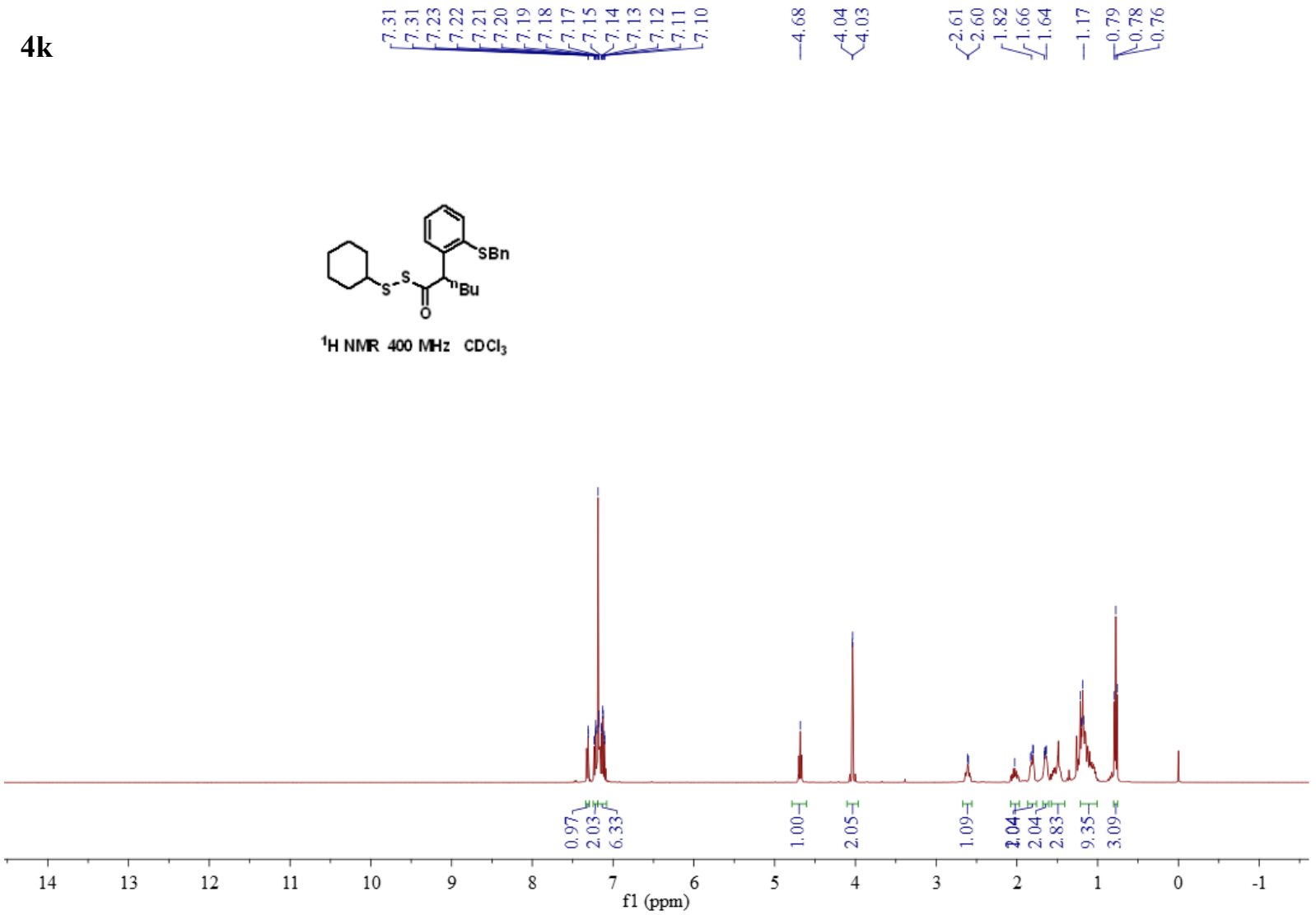
77.01
76.69
60.57
49.71
32.60
32.54
29.70
25.89
25.47
SPh
S
S
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4k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7.23
7.22
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7.19
7.18
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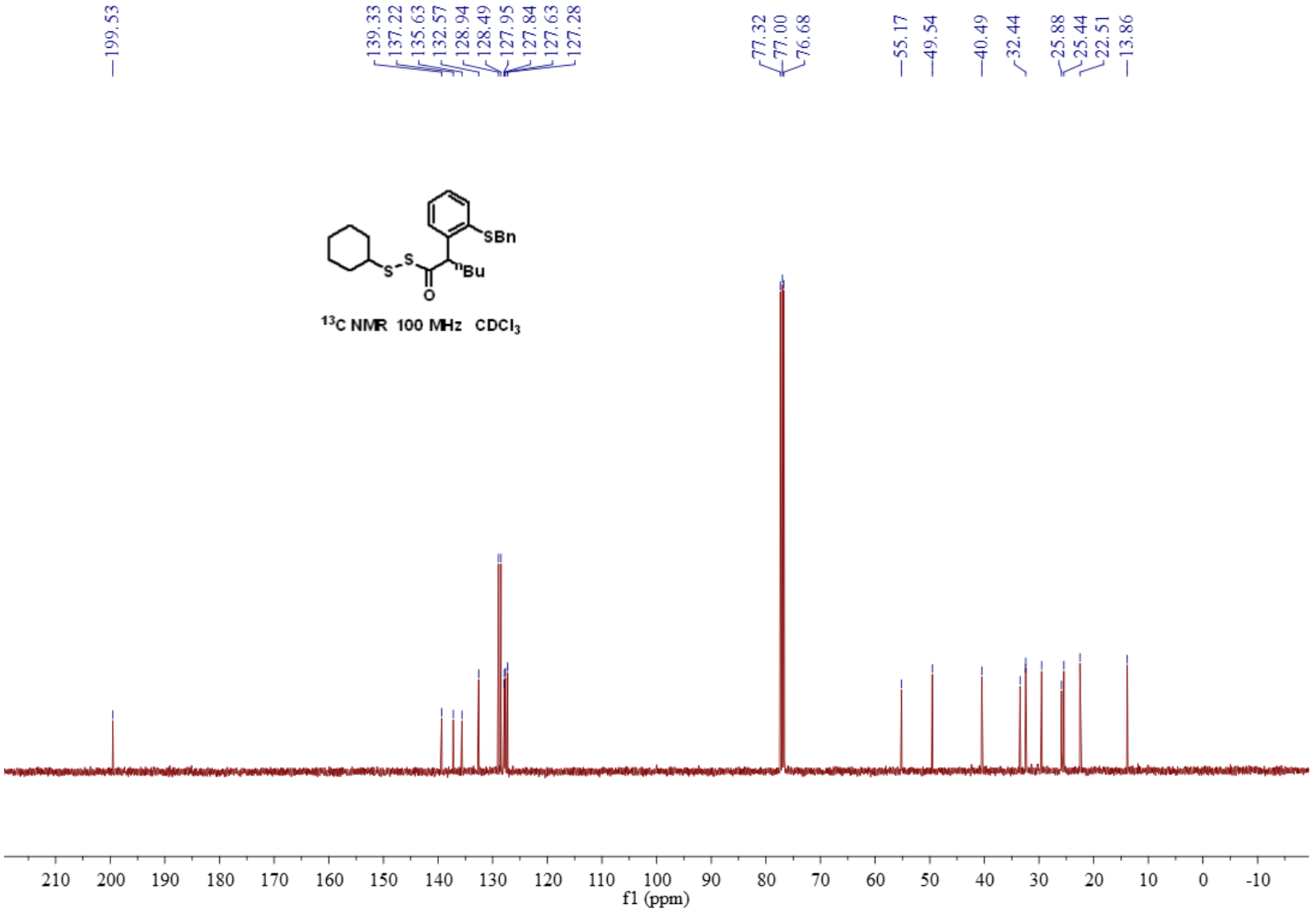
7.14
7.13
7.12
7.11
7.10
4.68
4.04
4.03
2.61
2.60
1.82
1.66
1.64
1.17
0.79
0.78
0.76
SBn
S
S
O
nBu
1H NMR 400 MHz CDCl3
0.97
2.03
6.33
1.00
2.05
1.09
2.04
2.04
2.83
9.35
3.09
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)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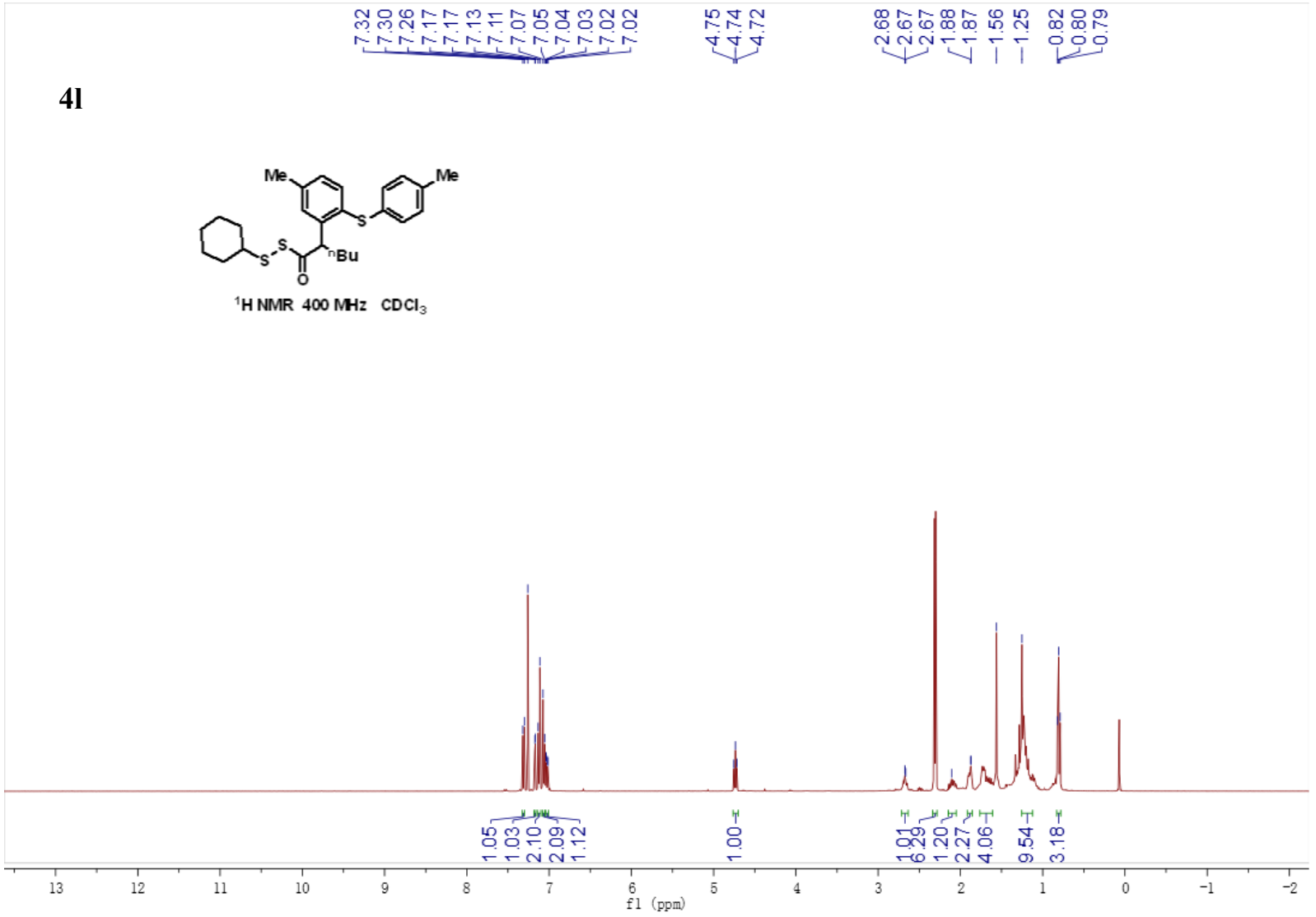
77.32
77.00
76.68
55.17
49.54
40.49
32.44
25.88
25.44
22.51
13.86
SBn
S
S
O
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4l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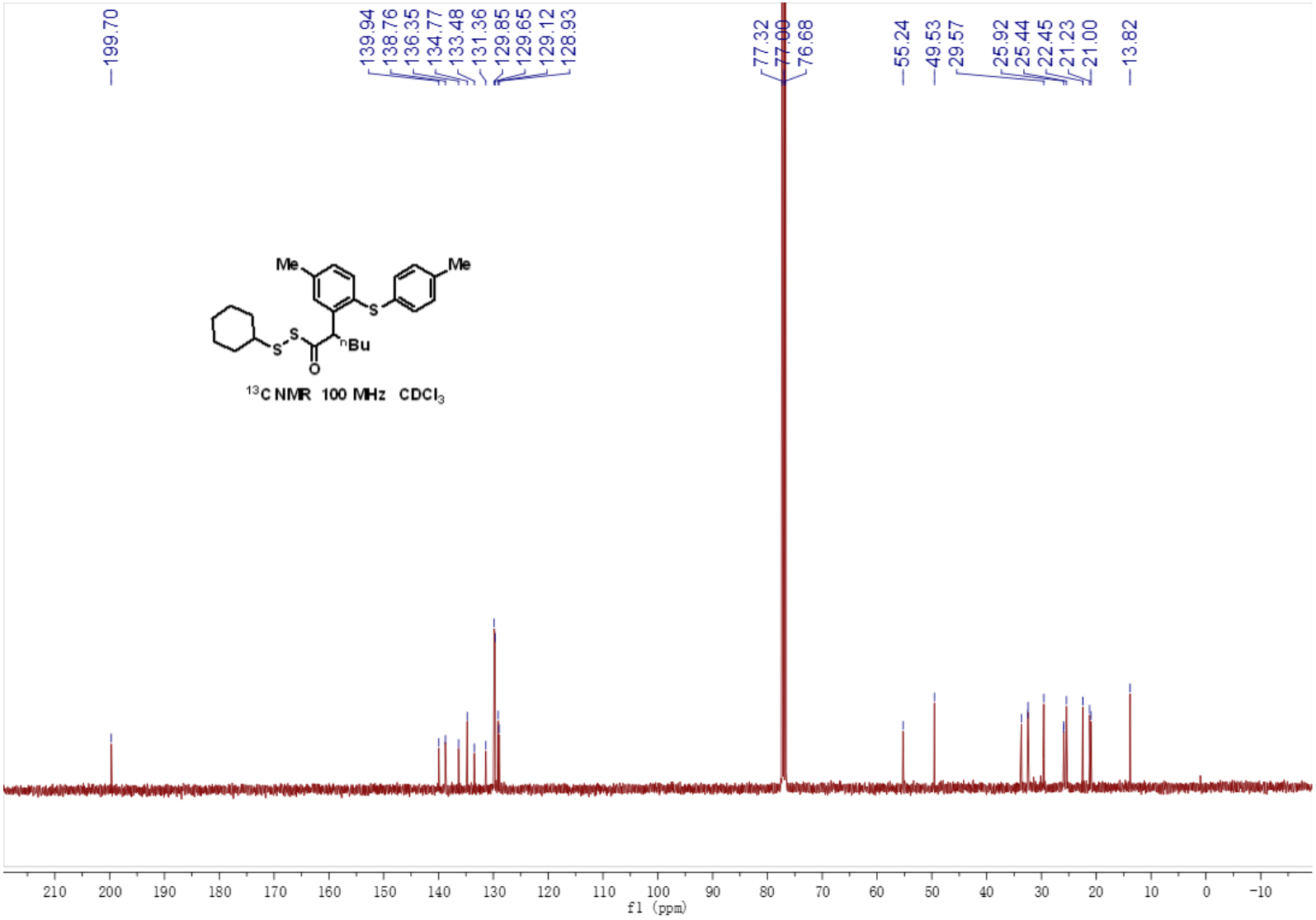
7.30
7.26
7.17
7.17
7.13
7.11
7.07
7.05
7.04
7.03
7.02
7.02
4.75
4.74
4.72
2.68
2.67
2.67
1.88
1.87
1.56
1.25
0.82
0.80
0.79
Me
Me
S
S
S
nBu
O
1H NMR 400 MHz CDCl3
1.05
1.03
2.10
2.09
1.12
1.00
1.01
6.29
1.20
2.27
4.06
9.54
3.18
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
f1 (ppm)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139.94
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133.48
131.36
129.85
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129.12
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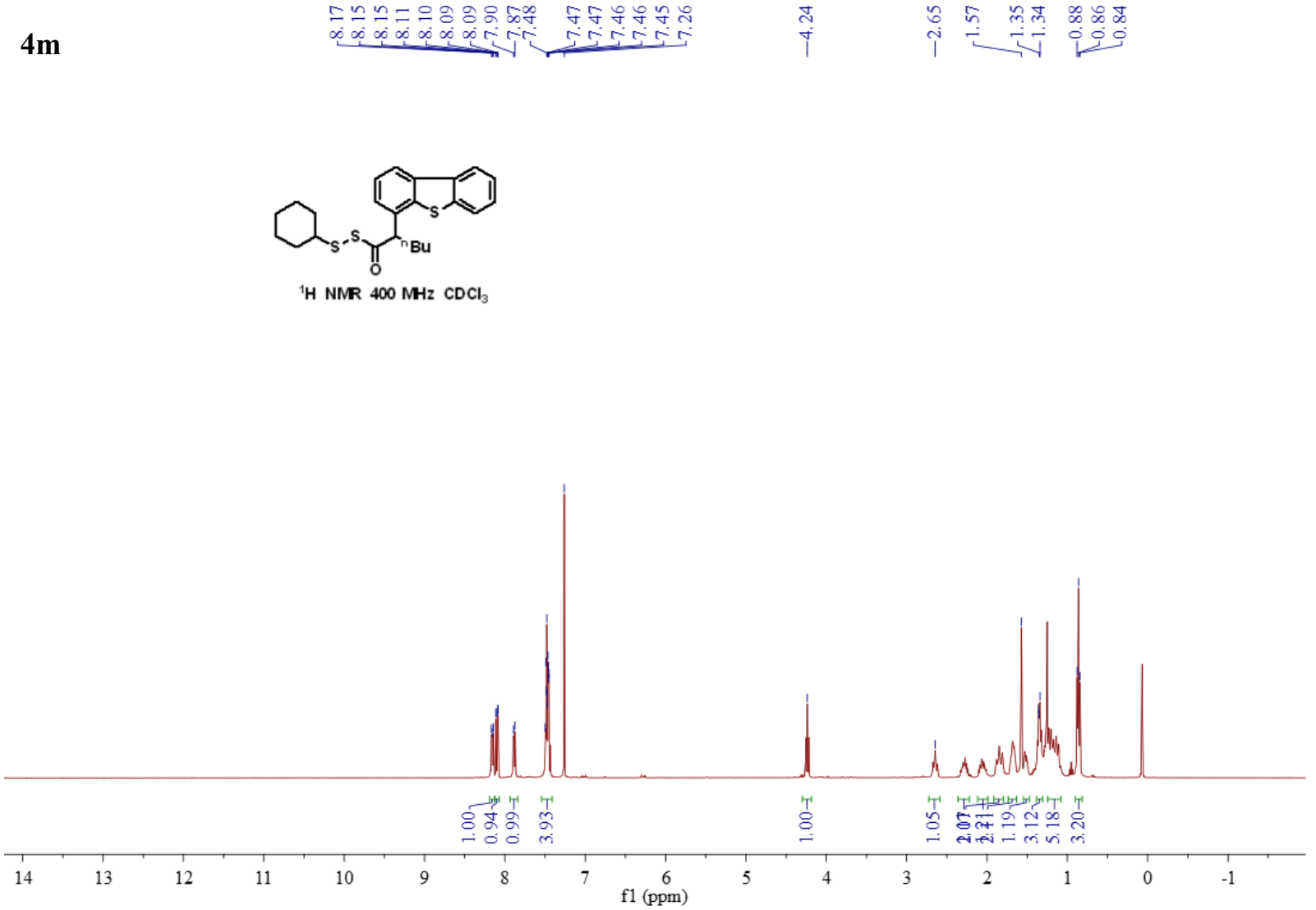
77.32
77.00
76.68
55.24
49.53
29.57
25.92
25.44
22.45
21.23
21.00
13.82
Me
Me
S
S
S
nBu
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4m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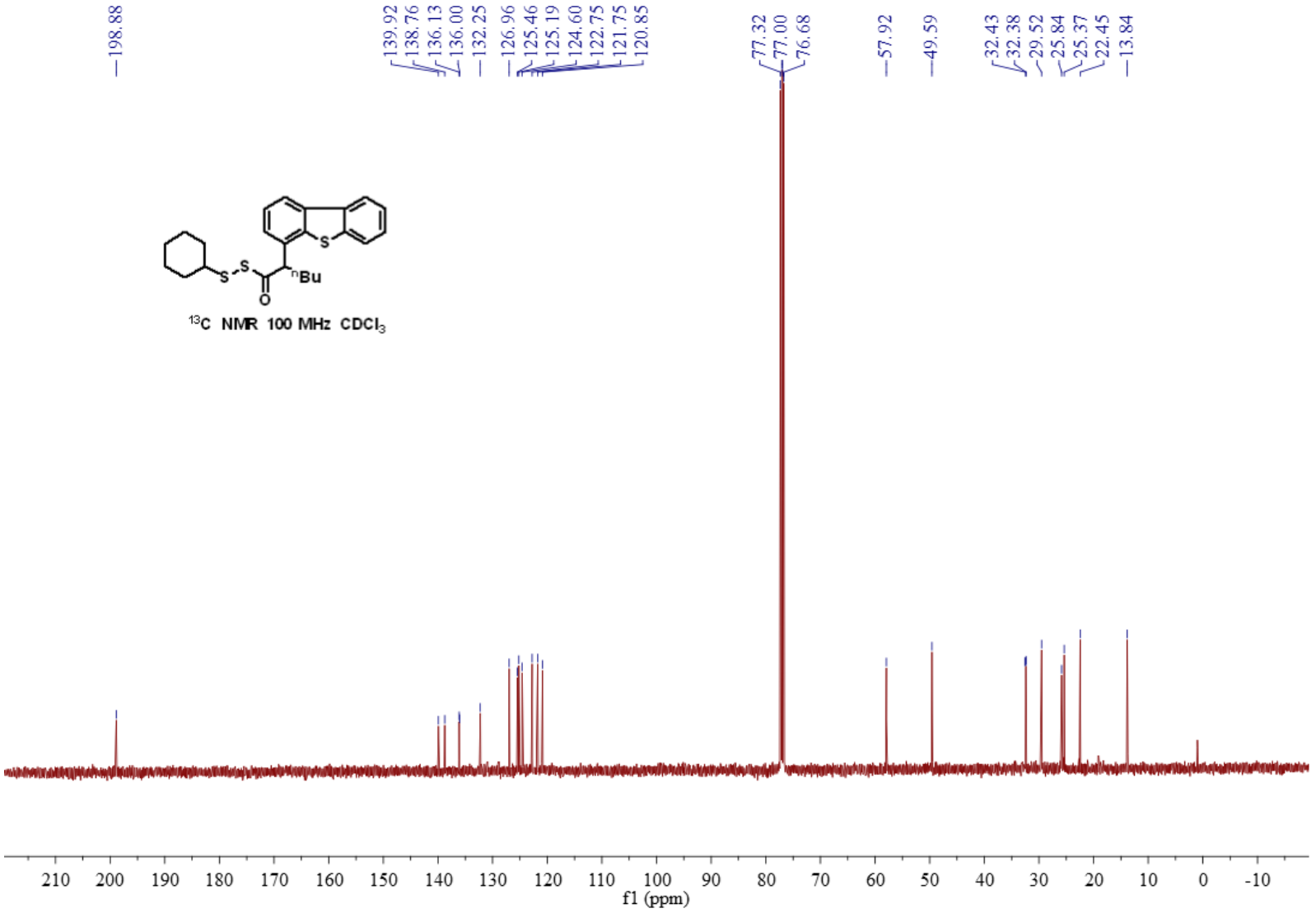
8.15
8.15
8.11
8.10
8.09
8.09
7.90
7.87
7.48
7.47
7.47
7.46
7.46
7.45
7.26
4.24
2.65
1.57
1.35
1.34
0.88
0.86
0.84
S
S
S
O
nBu
1H NMR 400 MHz CDCl3
1.00
0.94
0.99
3.93
1.00
1.05
3.12
5.18
3.20
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)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139.92
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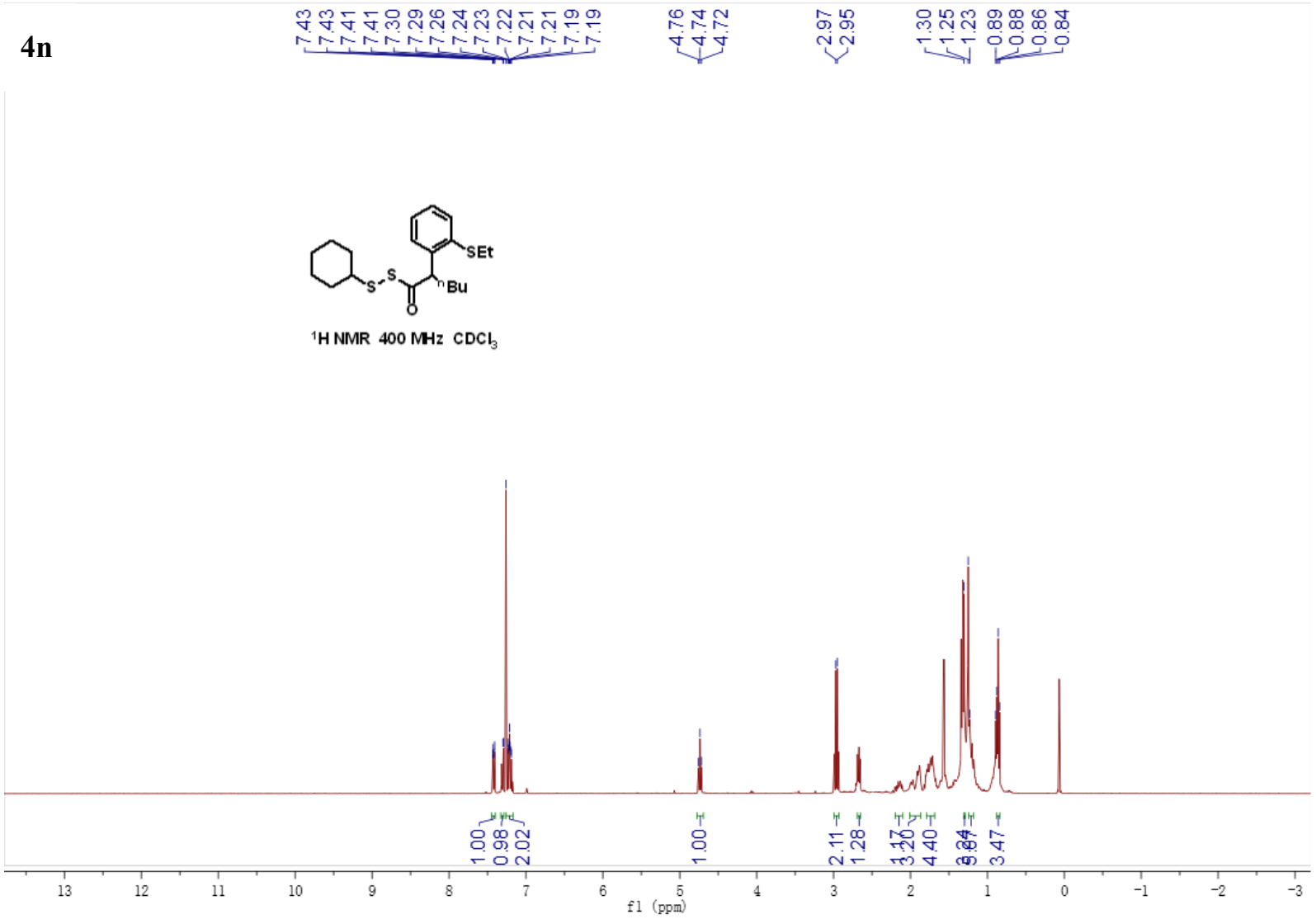
136.13
136.00
132.25
126.96
125.46
125.19
124.60
122.75
121.75
120.85
77.32
77.00
76.68
57.92
49.59
32.43
32.38
29.52
25.84
25.37
22.45
13.84
nBu
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4n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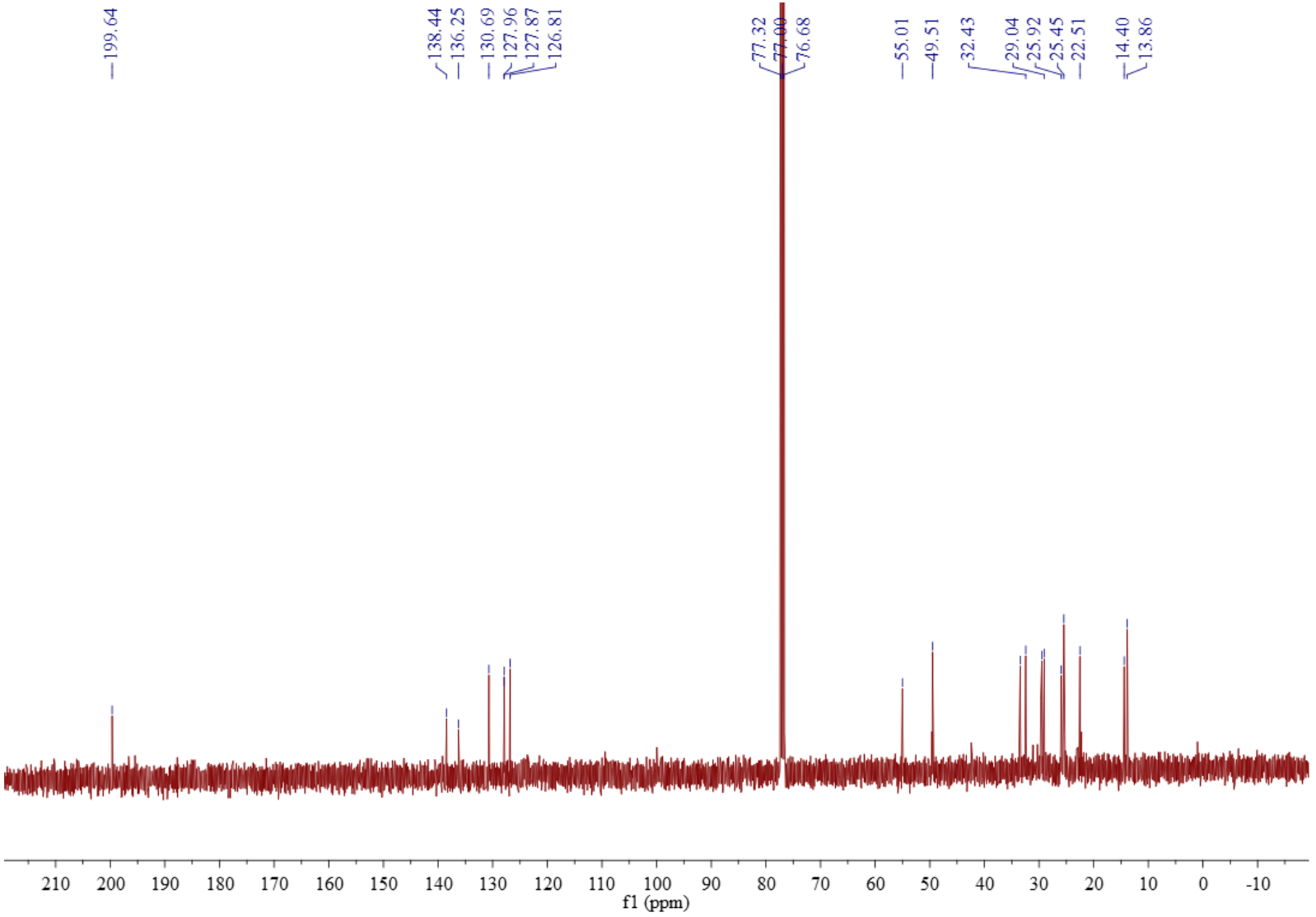
7.43
7.41
7.41
7.30
7.29
7.26
7.24
7.23
7.22
7.21
7.21
7.19
7.19
4.76
4.74
4.72
2.97
2.95
1.30
1.25
1.23
0.89
0.88
0.86
0.84
SEt
S
S
nBu
O
1H NMR 400 MHz CDCl3
1.00
0.98
2.02
1.00
2.11
1.28
3.47
f1 (ppm)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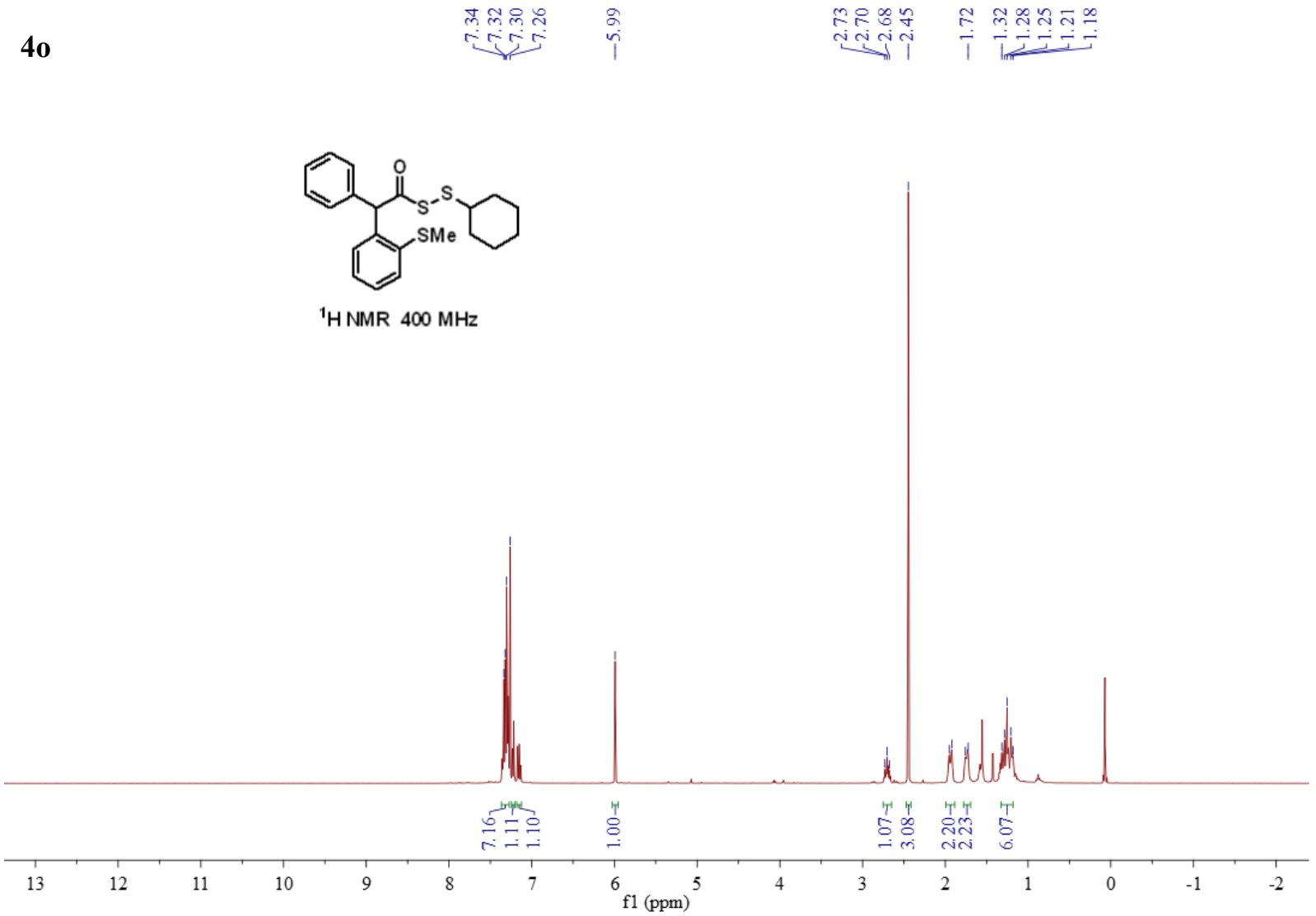
136.25
130.69
127.96
127.87
126.81
77.32
77.00
76.68
55.01
49.51
32.43
29.04
25.92
25.45
22.51
14.40
13.86
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4o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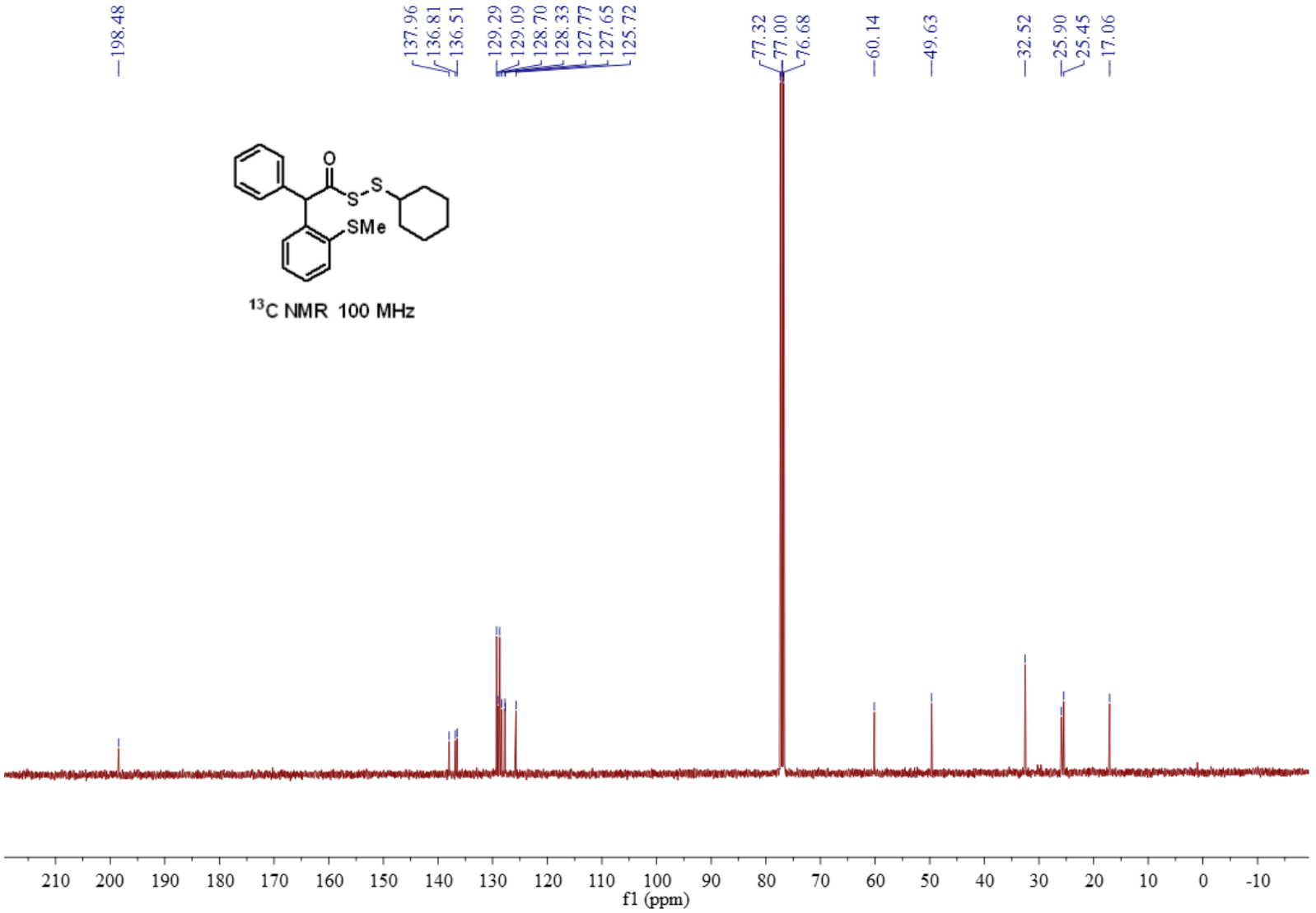
7.32
7.30
7.26
5.99
2.73
2.70
2.68
2.45
1.72
1.32
1.28
1.25
1.21
1.18
O
S
S
SMe
^{1}H NMR 400 MHz
7.16
1.11
1.10
1.00
1.07
3.08
2.20
2.23
6.07
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)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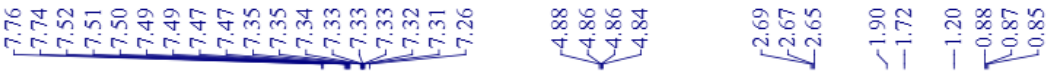
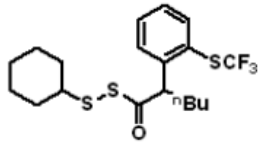
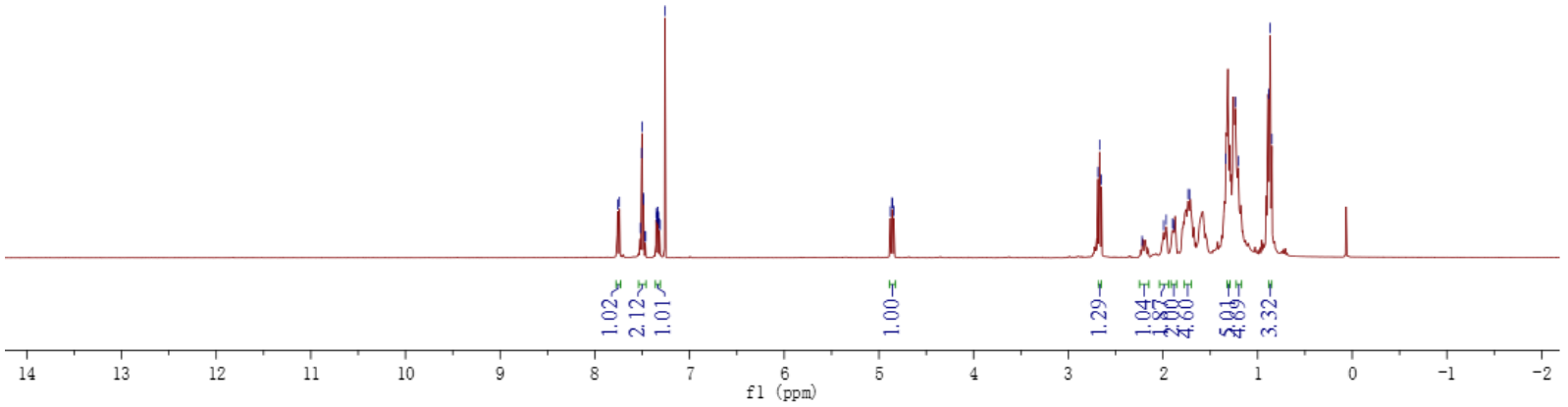
136.81
136.51
129.29
129.09
128.70
128.33
127.77
127.65
125.72
77.32
77.00
76.68
60.14
49.63
32.52
25.90
25.45
17.06
O
S
S
SMe
13C NMR 100 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

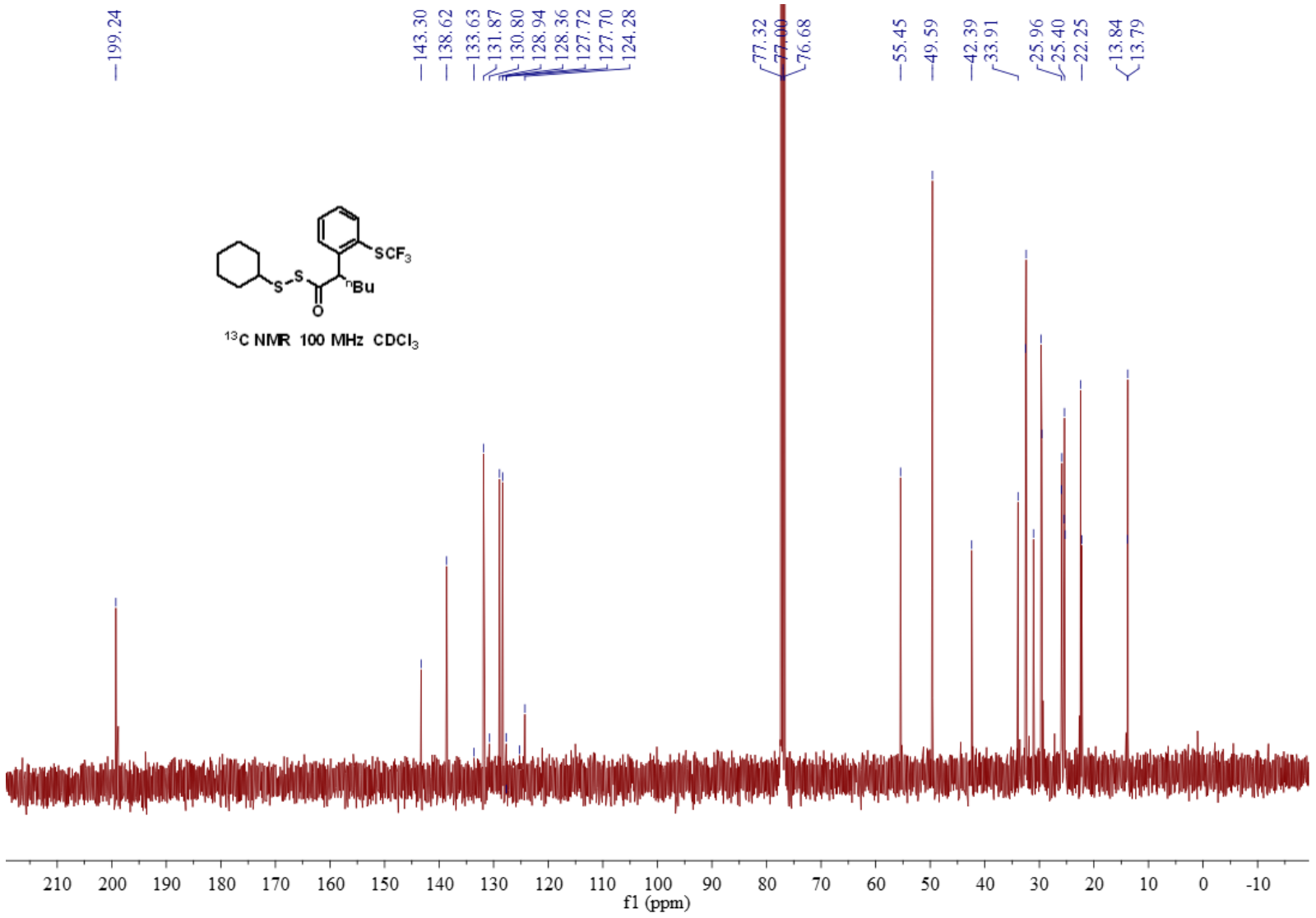
4p





^{1}H NMR 400 MHz $CDCl_3$

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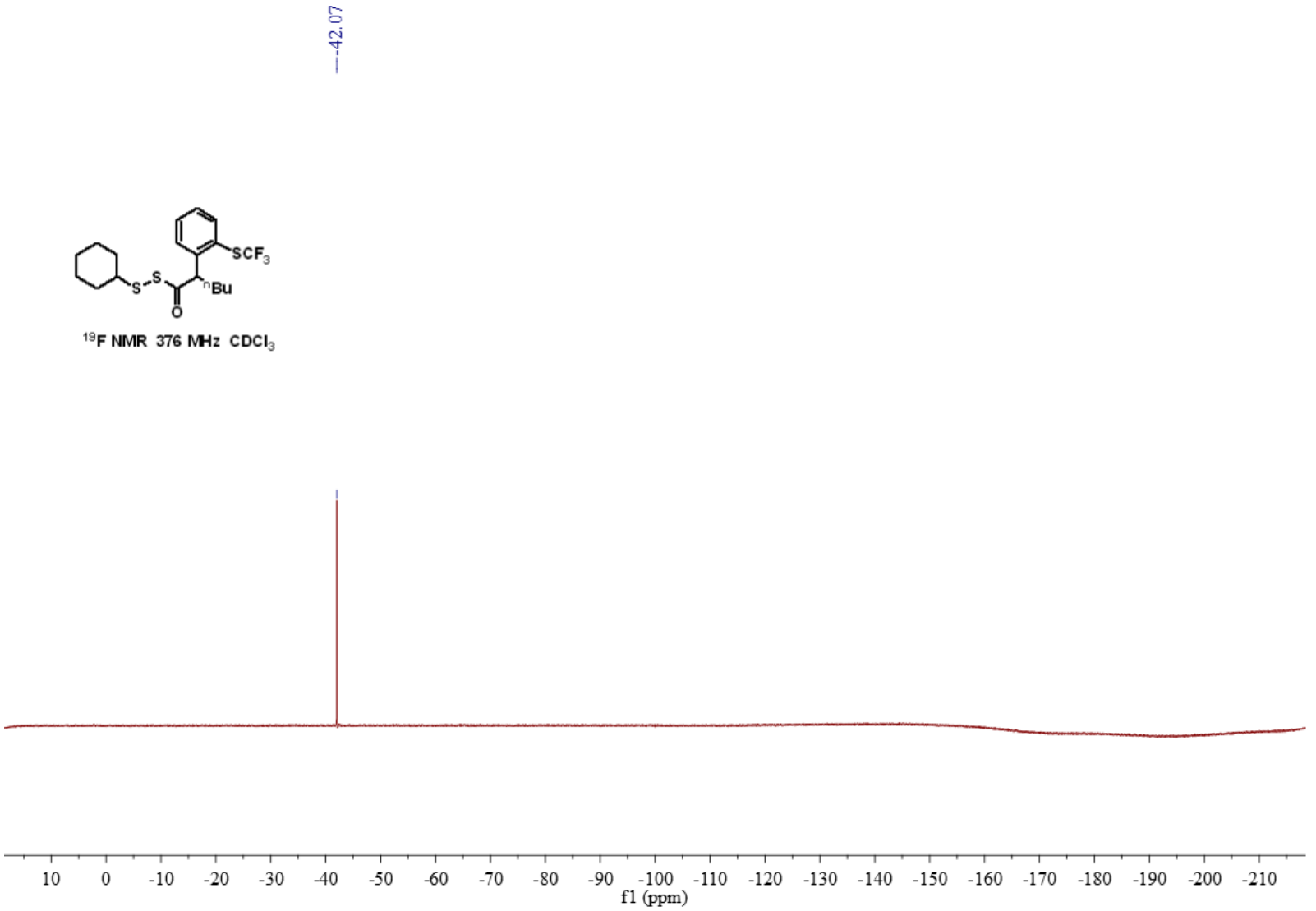
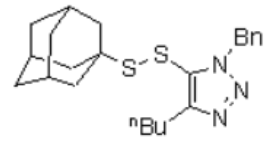
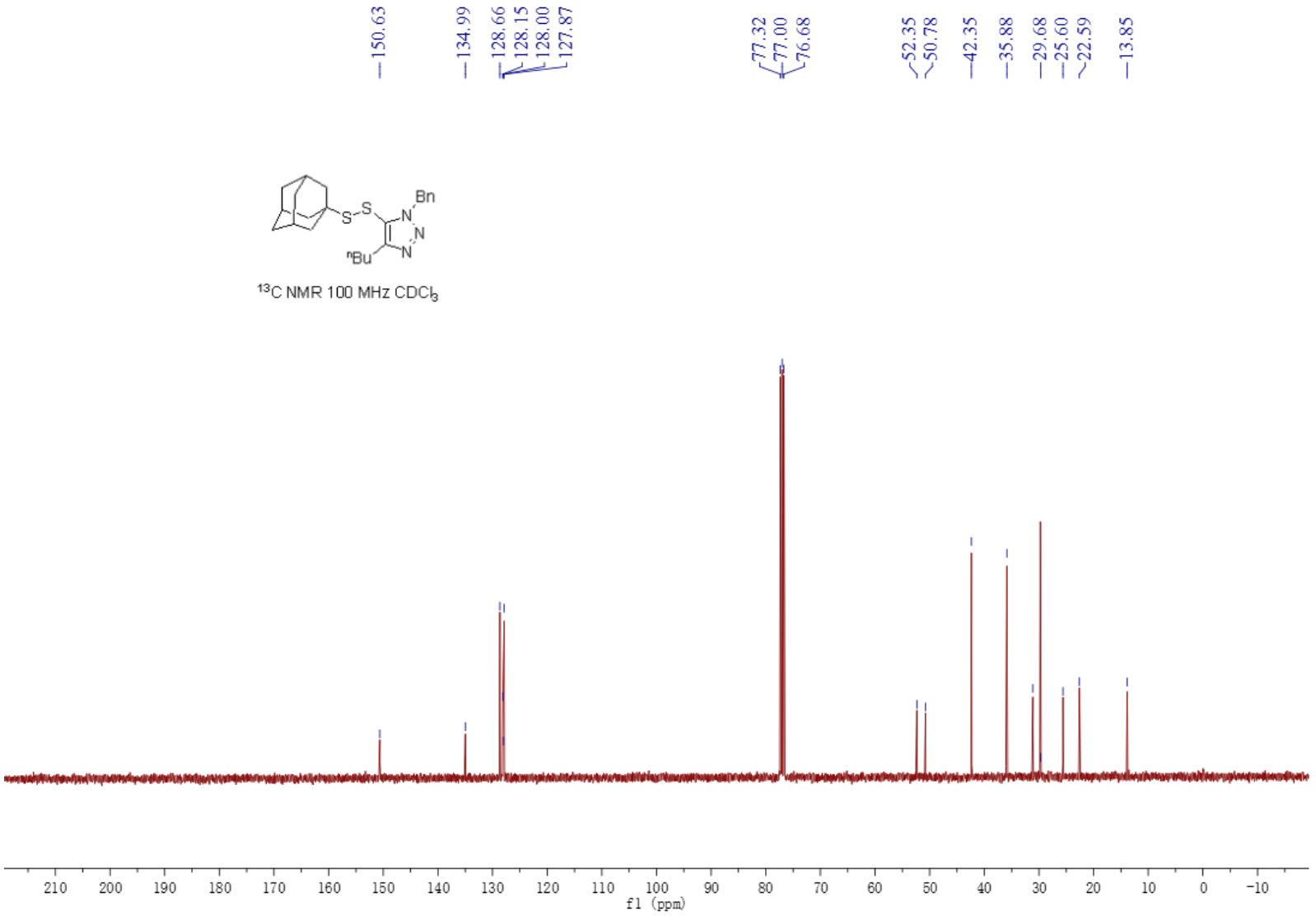
143.30
138.62
133.63
131.87
130.80
128.94
128.36
127.72
127.70
124.28
77.32
77.00
76.68
55.45
49.59
42.39
33.91
25.96
25.40
22.25
13.84
13.79
SCF3
S
S
nBu
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-42.07
SCF3
S
S
nBu
O
19F NMR 376 MHz CDCl3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

6



^{1}H NMR 400 MHz $CDCl_3$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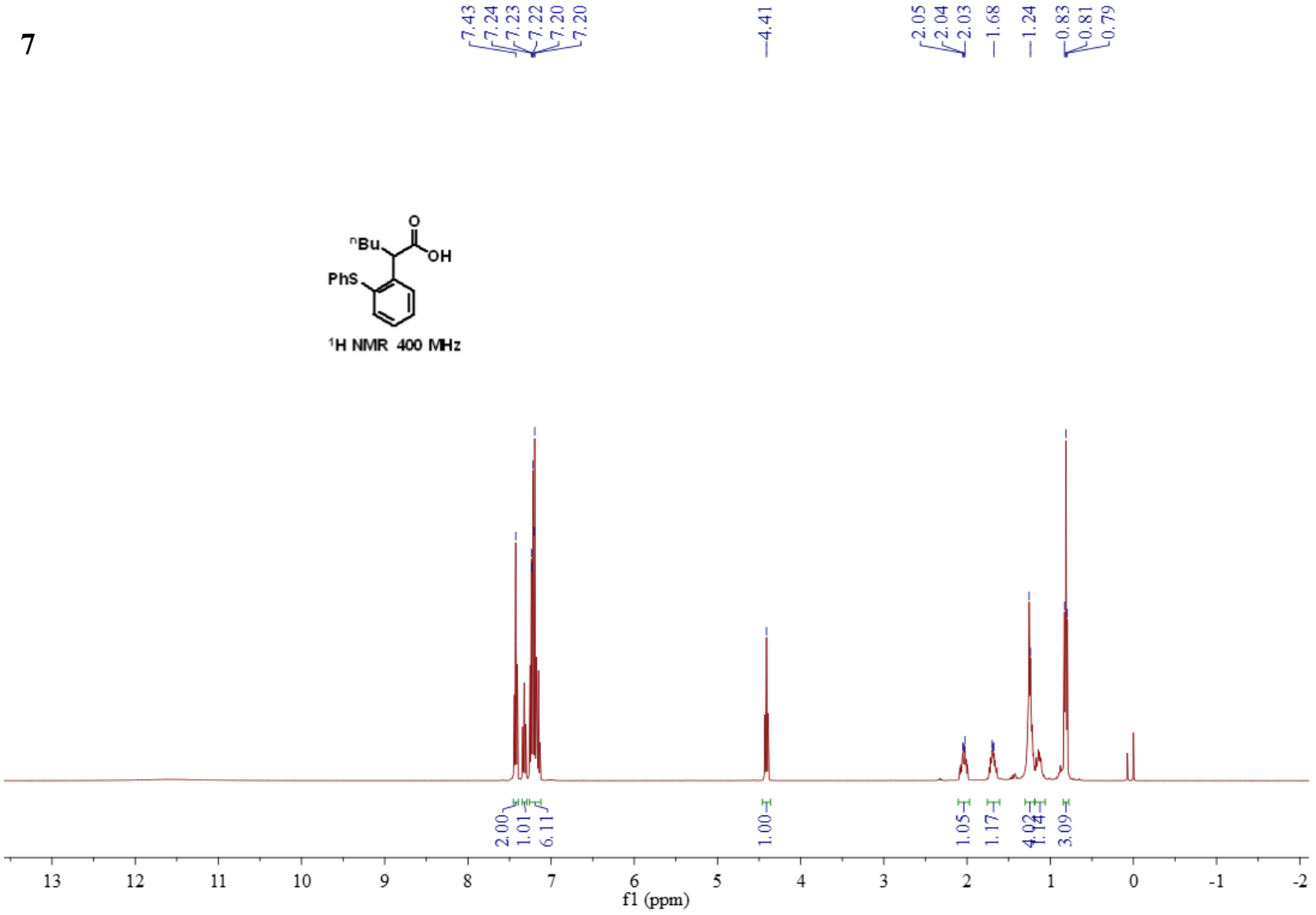
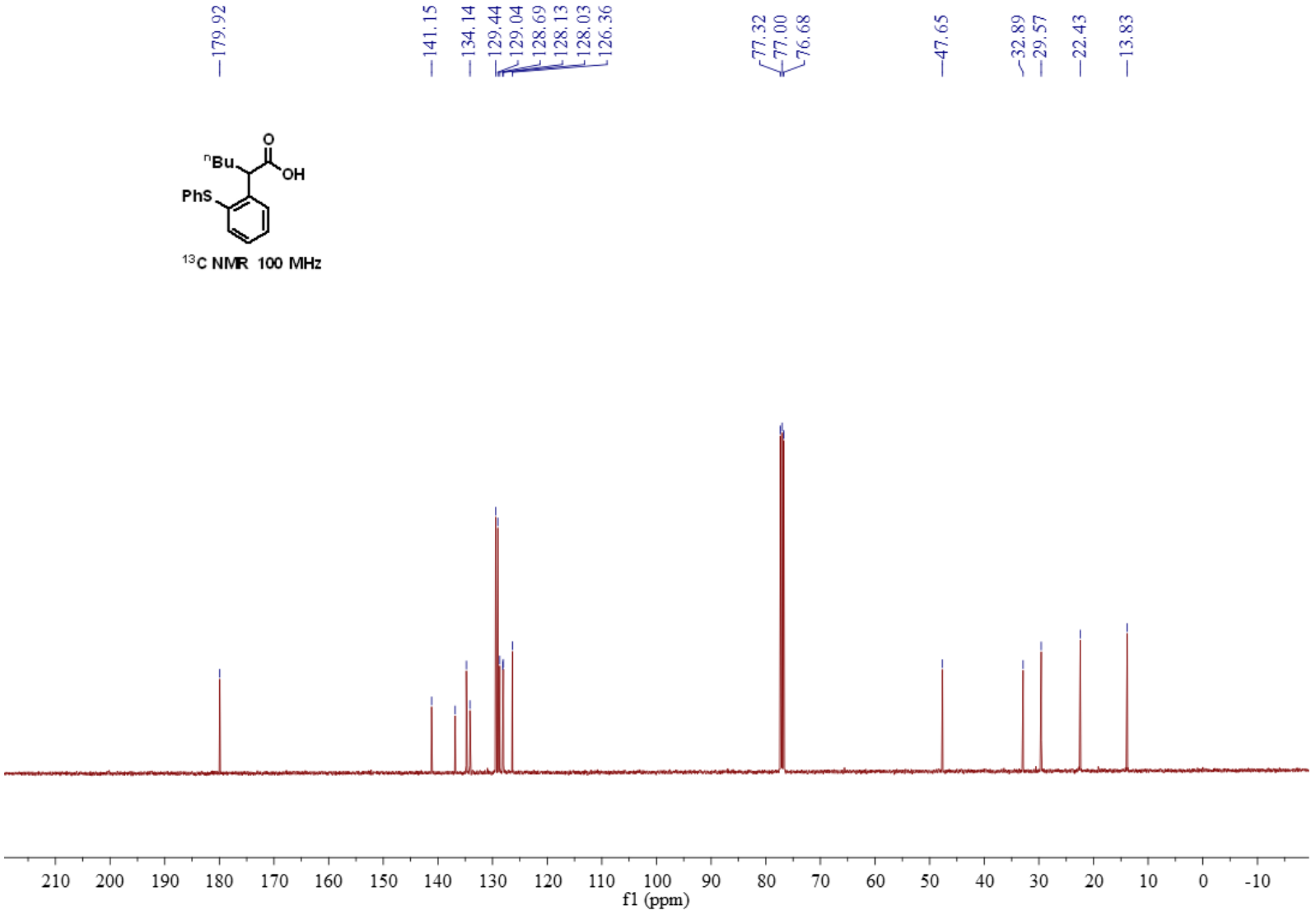
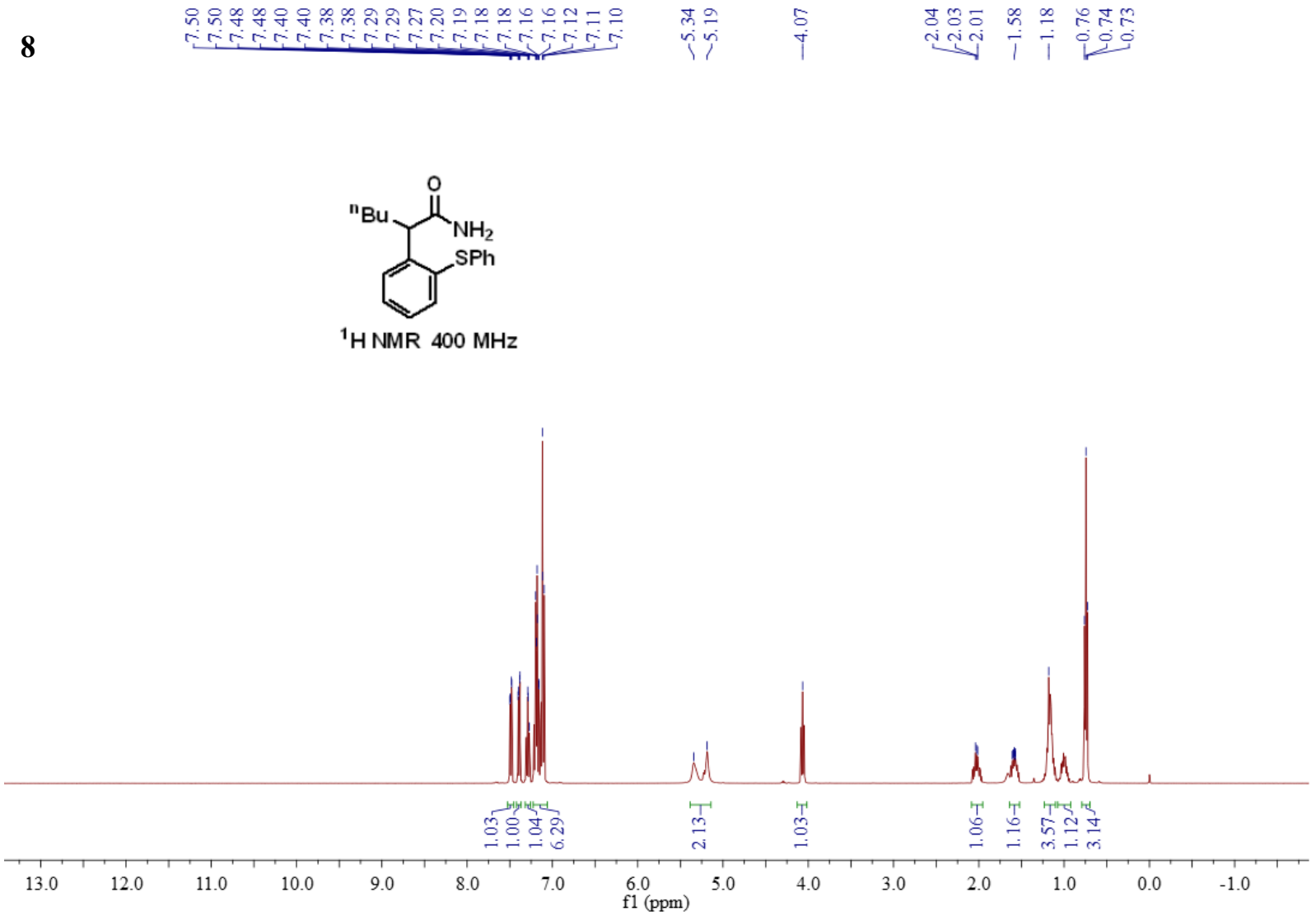
128.66
128.15
128.00
127.87
77.32
77.00
76.68
52.35
50.78
42.35
35.88
29.68
25.60
22.59
13.85
Bn
nBu
13C NMR 100 MHz CDCl3
f1 (ppm)

7


7.43
7.24
7.23
7.22
7.20
7.20
4.41
2.05
2.04
2.03
1.68
1.24
0.83
0.81
0.79
nBu
OH
O
PhS
1H NMR 400 MHz
2.00
1.01
6.11
1.00
1.05
1.17
4.02
4.14
3.09
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


179.92
141.15
134.14
129.44
129.04
128.69
128.13
128.03
126.36
77.32
77.00
76.68
47.65
32.89
29.57
22.43
13.83
O
nBu
OH
PhS
13C NMR 100 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

8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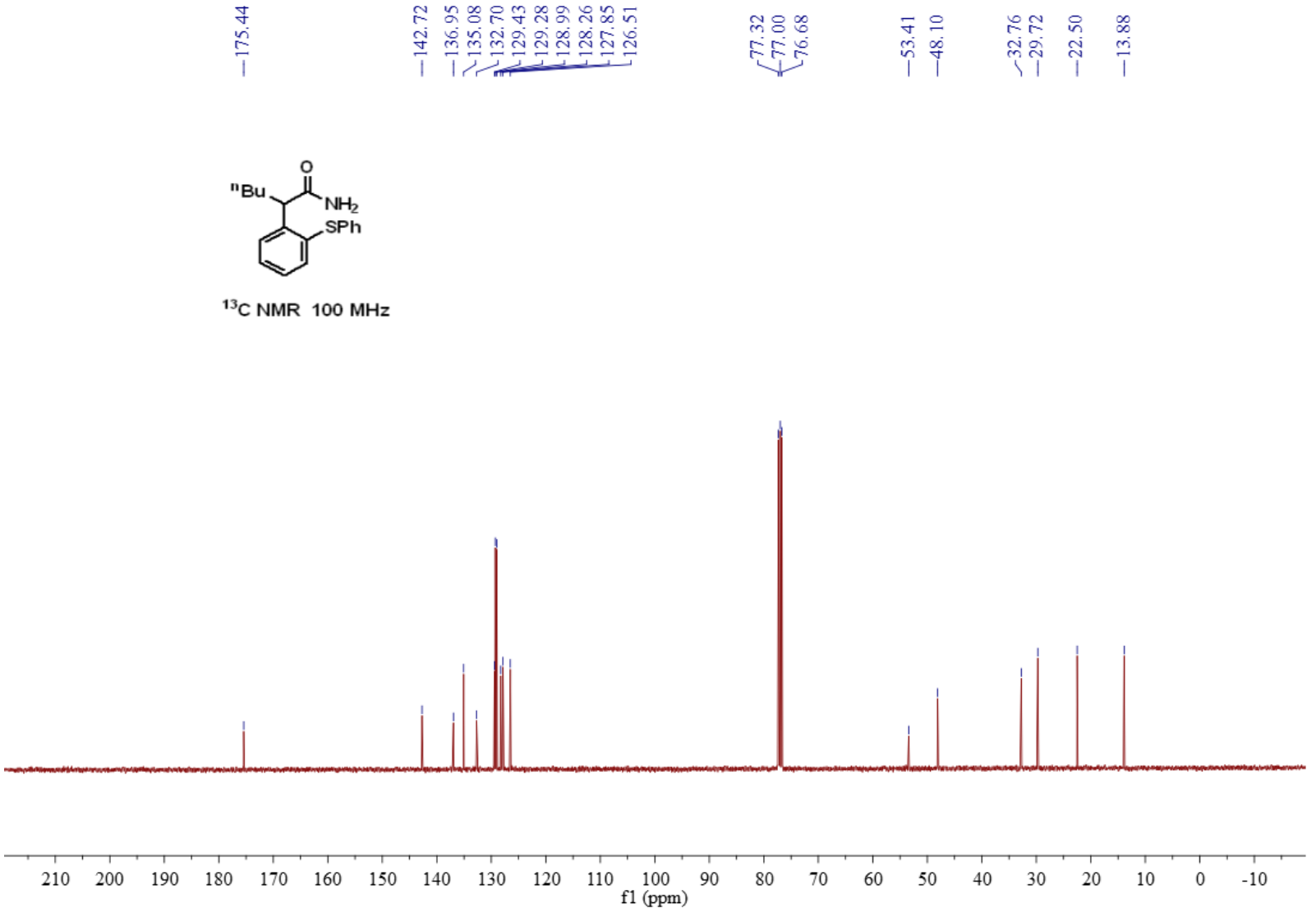
7.50
7.48
7.48
7.40
7.40
7.38
7.38
7.29
7.29
7.27
7.20
7.19
7.18
7.18
7.16
7.16
7.12
7.11
7.10
5.34
5.19
4.07
2.04
2.03
2.01
1.58
1.18
0.76
0.74
0.73
O
nBu
NH2
SPh
1H NMR 400 MHz
1.03
1.00
1.04
6.29
2.13
1.03
1.06
1.16
3.57
1.12
3.14
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)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142.72
136.95
135.08
132.70
129.43
129.28
128.99
128.26
127.85
126.51
77.32
77.00
76.68
53.41
48.10
32.76
29.72
22.50
13.88
O
"Bu
NH2
SPh
13C NMR 100 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)