

Supporting Information

Expanding the Horizon of Intermolecular Trapping of In-Situ Generated α -Oxo Gold Carbenes: Efficient Oxidative Union of Allylic Sulfides and Terminal Alkynes via C-C Bond Formation

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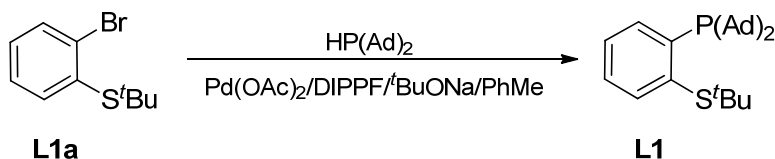
Contents	Page number
General	S2
Procedure for the synthesis	S2
1. Synthesis of catalyst L_1AuCl	S2
2. Syntheses of substrates	S3
3. General Procedure for compound 4a-q	S6
4. Syntheses of compound 5a-l	S14
5. General Procedure for compound 6-7	S21
6. General Procedure for compound 8-9	S22
¹ H, ¹³ C and ³¹ P NMR spectra	S25

GENERAL. Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous 1,2-dichloroethane (HPLC grade) and dichloromethane (HPLC grade) were purified by distillation over calcium hydride. Tetrahydrofuran was distilled over sodium/benzophenone. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using Silicycle precoated silica gel plates. Flash column chromatography was performed over Silicycle silica gel (230-400 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Varian 500 MHz and Varian 600 MHz Unity plus spectrometer using residue solvent peaks as internal standards (CHCl_3 , ^1H : 7.26 ppm; ^{13}C : 77.23 ppm). ^{31}P NMR spectra were recorded on a Varian 400 MHz spectrometer using H_3PO_4 (0.00 ppm) as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Micromass QTOF₂ Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization or Waters GCT Premier time-of-flight mass spectrometer with a field ionization (FI) ion source.

PROCEDURE FOR THE SYNTHESIS

1. Synthesis of Catalyst **L1AuCl**

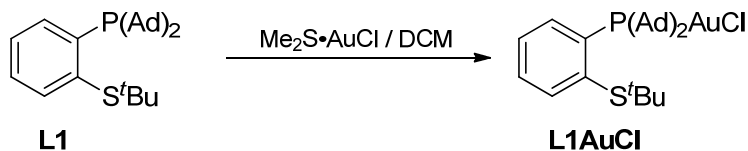
1.1 Ligand of di(Adamantan-1-yl) 2-(*tert*-butylthio)phenyl phosphine



L1a was prepared according to the literature procedure¹. Under nitrogen atmosphere **L1a** (2 mmol, 1 equiv), Pd(OAc)_2 (0.04 mmol, 2 mol%), DiPPF (1,1'-bis(diisopropylphosphino)ferrocene, 0.06 mmol, 3 mol%), *t*-BuONa (2.4 mmol, 1.2 equiv) and 5 mL dry toluene were added to a flame-dried Schlenk flask and the resulting suspension was stirred until apparently homogeneous. Added di(1-adamantyl)phosphine (2.2 mmol, 1.1

equiv), the flask was heated at 110 °C in oil bath for 20 hours, which then was cooled to room temperature, and purified by column chromatography without work-up to yield the final ligand **L1** in 61 % yield. ^1H NMR (500 MHz, CDCl_3) δ 7.84 – 7.79 (m, 1H), 7.70 – 7.64 (m, 1H), 7.30 – 7.22 (m, 2H), 2.00 – 1.87 (m, 18H), 1.66 (s, 12H), 1.39 (s, 9H). ^{13}C NMR (126 MHz, CDCl_3) δ 143.75, 140.51 (d, $J_{\text{PC}} = 23.5$ Hz), 137.17 (d, $J_{\text{PC}} = 2.4$ Hz), 136.77 (d, $J_{\text{PC}} = 3.5$ Hz), 128.12, 125.67, 48.02, 41.97 (d, $J_{\text{PC}} = 12.9$ Hz), 37.89 (d, $J_{\text{PC}} = 26.6$ Hz), 37.21 (d, $J_{\text{PC}} = 1.0$ Hz), 31.98 (d, $J_{\text{PC}} = 1.2$ Hz), 29.12 (d, $J_{\text{PC}} = 8.5$ Hz). ^{31}P NMR (CDCl_3 , 162 MHz) δ 23.33. IR (neat): 2902, 2848, 1451, 1362, 1301, 908, 733 cm^{-1} ; MS (ES^+ , m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{30}\text{H}_{44}\text{PS}$, 467.29; found, 467.24.

1.2 General Procedure for synthesis of Catalyst **L1AuCl**

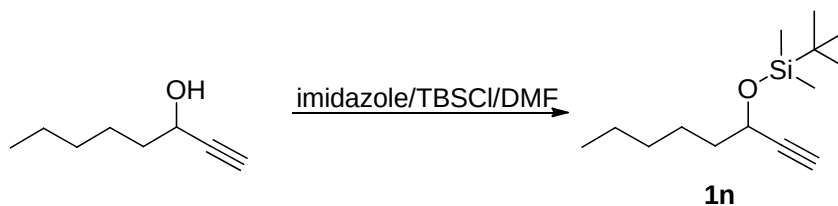


To a solution of 1 mmol ligand **L1** in 5 mL anhydrous DCM was added dimethylsulfide gold (I) chloride (294.5 mg, 1 mmol). The mixture was stirred for 30 min at room temperature and the solvent was evaporated off under reduced pressure to give the desired gold catalyst **L1AuCl** as light beige solid in quantitative yield. ^1H NMR (500 MHz, CDCl_3) δ 7.92 – 7.83 (m, 2H), 7.51 – 7.42 (m, 2H), 2.20 – 2.10 (m, 12H), 1.98 (s, 6H), 1.67 (s, 12H), 1.50 (s, 9H). ^{13}C NMR (126 MHz, CDCl_3) δ 141.54 (d, $J_{\text{PC}} = 10.9$ Hz), 140.29 (d, $J_{\text{PC}} = 4.6$ Hz), 135.61 (d, $J_{\text{PC}} = 2.6$ Hz), 130.40, 130.06 (d, $J_{\text{PC}} = 46.6$ Hz), 127.10 (d, $J_{\text{PC}} = 5.9$ Hz), 52.02, 42.66 (d, $J_{\text{PC}} = 22.0$ Hz), 42.22 (d, $J_{\text{PC}} = 3.0$ Hz), 36.51 (d, $J_{\text{PC}} = 1.7$ Hz), 31.75, 28.81 (d, $J_{\text{PC}} = 9.8$ Hz). ^{31}P NMR (CDCl_3 , 162 MHz) δ 62.45. IR (neat): 2906, 2851, 1447, 1301, 1162, 914, 730 cm^{-1} ; MS (ES^+ , m/z): $[\text{L1Au}]^+$ calcd. for $\text{C}_{30}\text{H}_{43}\text{AuPS}$, 663.25; found, 663.16.

2. Synthesis of substrates

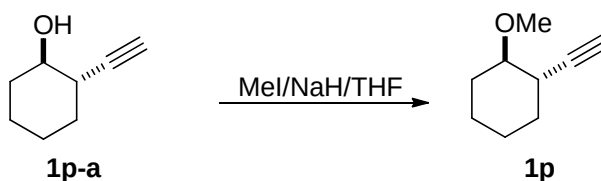
2.1 alkyne substrates

2.1.1 *tert*-butyldimethyl(oct-1-yn-3-yloxy)silane (**1n**)



1n was prepared according to the literature procedure ²

2.1.2 (Trans)-1-ethynyl-2-methoxycyclohexane (**1o**)

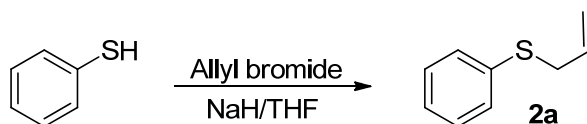


1p-a was prepared according to the literature procedure ³.

To a solution of **1p-a** (891 mg, 7.19 mmol, 1.0 equiv) in anhydrous tetrahydrofuran (30 mL) at 0 °C was slowly added NaH (432 mg, 10.8 mmol, 1.5 equiv, 60% w/w). The reaction mixture was allowed to warm up to ambient temperature. Methyl iodide (0.54 mL, 8.64 mmol, 1.2 equiv) was added. Upon TLC showed complete consumption of the starting material, the reaction was quenched with saturated aqueous ammonium chloride, extracted twice with ethyl acetate, dried over Na₂SO₄, filtered, and concentrated *in vacuo*. Purification by flash column chromatography afforded the desired alkynyl ether **1p** as the colorless oil (718 mg) in 72 % yield. ¹H NMR (500 MHz, CDCl₃) δ 3.41 (s, 3H), 3.15 (td, *J* = 8.0, 3.8 Hz, 1H), 2.45 – 2.37 (m, 1H), 2.08 (d, *J* = 2.4 Hz, 1H), 2.06 – 1.98 (m, 1H), 1.98 – 1.90 (m, 1H), 1.72 – 1.60 (m, 2H), 1.49 – 1.39 (m, 1H), 1.32 – 1.20 (m, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 86.81, 81.30, 69.31, 56.99, 34.77, 30.44, 29.49, 24.12, 23.27; IR (neat): 2925, 2858, 1461, 1100 cm⁻¹; GCMS-EI, *m/z*, 138 (M⁺)

2.2 Sulfide substrates

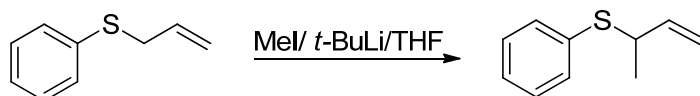
2.2.1 General Procedure for Phenyl allyl sulfide (**2a**)



To a flame-dried flask a suspension of 60% NaH (4 g, 0.1 mol) in THF (100 mL) was added and stirred at 0 °C under water-ice bath. The thiophenol (10.1 mL, 0.1 mol) was then added dropwise to the mixture over 20 min. After completion of the addition, the water-ice bath was removed and the mixture was allowed to warm to room temperature and stirred for 0.5 h. Subsequently, the mixture was again cooled to 0 °C and allyl bromide (0.067 mol) was added dropwise while stirring. After addition, the mixture was warmed to room temperature and then stirred for 2 h or more according to TLC. Finally, 50 mL of saturated NH₄Cl solution was added to quench the reaction. The organic layer was washed with water (60 mL×2), extracted with Et₂O (60 mL×2), then dried over MgSO₄ and concentrated under vacuum. The crude product was purified by silica gel column chromatography (hexanes) to afford **2a** (8.3 g, 82 %) as colorless oil.

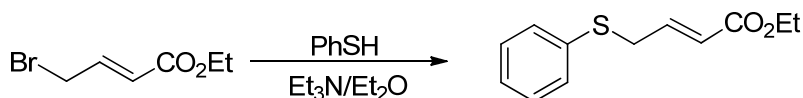
Phenyl 2-methylallyl sulfide (**2d**), phenyl but-2-en-1-yl sulfide(**2e**), phenyl cinnamyl sulfide(**2f**) and benzyl allyl sulfide(**2i**) were prepared using the same procedure for **2a**.

2.2.2 Phenyl but-3-en-2-yl sulfide (**2h**)



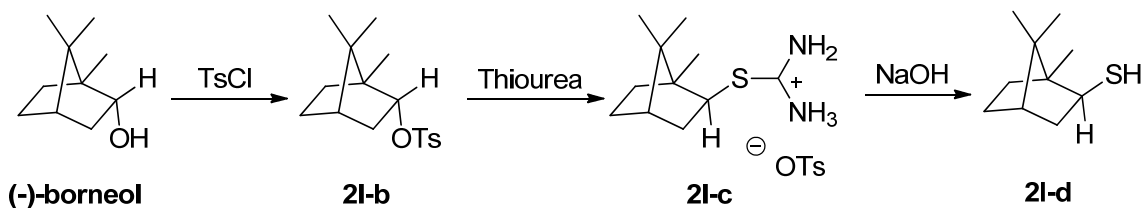
2h was prepared according to the literature procedure ⁴.

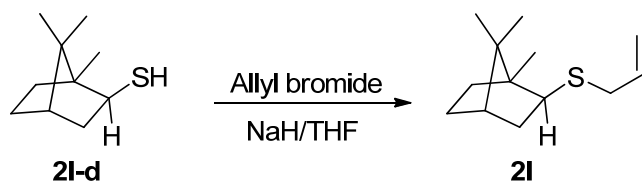
2.2.3 Ethyl (E)-4-(phenylthio)but-2-enoate (**2g**)



2g was prepared according to the literature procedure ⁵.

2.2.4 (1S,2S,4S)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl allyl sulfide

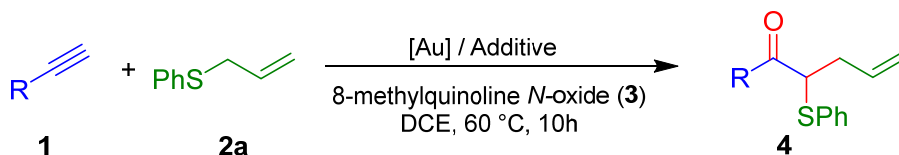




2I-d was synthesized from (-)-borneol via 3 steps according to the literature procedure ⁶.

2I was prepared using the general procedure for sulfide **2a**. Purified by flash column chromatography to afford **2I** as colorless oil in 71 % yield. ¹H NMR (500 MHz, CDCl₃) δ 5.80 (ddt, *J* = 17.1, 10.0, 7.2 Hz, 1H), 5.13 – 5.02 (m, 2H), 3.13 (dt, *J* = 7.2, 1.1 Hz, 2H), 2.62 (dd, *J* = 8.7, 6.3 Hz, 1H), 1.88 – 1.77 (m, 2H), 1.76 – 1.59 (m, 3H), 1.19 – 1.08 (m, 2H), 0.98 (s, 3H), 0.97 (s, 3H), 0.82 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 135.20, 116.84, 52.63, 49.52, 47.56, 46.08, 40.91, 38.63, 37.42, 27.58, 20.65, 20.43, 14.16. IR (neat): 3081, 2985, 2952, 1634, 1454, 1389, 988, 912 cm⁻¹; GCMS-EI, *m/z*, 210 (M⁺)

3. General Procedure for Synthesis of Target Compounds 4 series



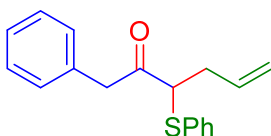
To a 2 dram septum-capped vial were added sequentially **1** (0.20 mmol), **2** (0.30 mmol, 1.5 equiv), **L1**AuCl (2.8 mg, 4 μmol, 2 mol %), NaBAr^F₄ (5.4 mg, 6 μmol, 3 mol %) and 1 mL of dry DCE, then **3** (19.7 mg, 0.26 mmol, 1.3 eq) in 1 mL of dry DCE was introduced into reaction by syringe pump at the rate of 0.1 mL per hour. After stirring at 60 °C for 10 h, the solvent was removed under reduced pressure. Based on the TLC monitoring, if 8-methylquinoline can be clearly separated from the product, there is no need to work up; otherwise, the residue dissolved with 20 mL of ethyl acetate, then washed by HCl solution (1 mol/L, 10 mL×3) and brine (10 mL×2) respectively. After dried over MgSO₄ and removed the solvent, the crude product was purified through flash chromatography on silica gel to afford target compounds **4** series as following.

4-(phenylthio)pentadec-1-en-5-one (4a)



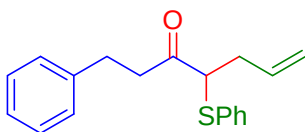
Purified by flash column chromatography (hexanes/ethyl acetate, 50/1) to afford **4a** (55 mg, 83 %) as pale yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ 7.40 – 7.35 (m, 2H), 7.33 – 7.27 (m, 3H), 5.86 – 5.73 (m, 1H), 5.16 – 5.02 (m, 2H), 3.67 (dd, *J* = 8.1, 6.9 Hz, 1H), 2.66 – 2.37 (m, 4H), 1.55 (m, 2H), 1.34 – 1.16 (m, 14H), 0.88 (t, *J* = 7.0 Hz, 3H).; **¹³C NMR** (126 MHz, CDCl₃) δ 206.81, 134.55, 133.20, 132.86, 129.26, 128.31, 118.03, 56.33, 40.27, 34.84, 32.11, 29.77, 29.68, 29.58, 29.52, 29.38, 24.04, 22.90, 14.33. **IR** (neat): 3078, 2925, 2854, 1710, 1467, 1439, 918, 745 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for C₂₁H₃₂NaOS: 355.2; Found: 355.2 [M+Na]⁺.

1-phenyl-3-(phenylthio)hex-5-en-2-one (4b)



Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4b** (45.5 mg, 81 %) as pale yellow oil. **¹H NMR** (600 MHz, CDCl₃) δ 7.40 – 7.35 (m, 2H), 7.34 – 7.28 (m, 5H), 7.28 – 7.24 (m, 1H), 7.16 (d, *J* = 7.2 Hz, 2H), 5.79 – 5.68 (m, *J* = 23.3, 10.7, 6.8 Hz, 1H), 5.10 – 5.02 (m, *J* = 11.8, 6.1 Hz, 2H), 3.93 (d, *J* = 15.5 Hz, 1H), 3.87 (d, *J* = 15.5 Hz, 1H), 3.76 (dd, *J* = 7.9, 7.1 Hz, 1H), 2.65 – 2.50 (m, 1H), 2.50 – 2.39 (m, 1H). **¹³C NMR** (151 MHz, CDCl₃) δ 203.27, 134.36, 134.05, 133.69, 132.28, 129.86, 129.33, 128.80, 128.61, 127.21, 118.07, 55.42, 47.31, 34.59. **IR** (neat): 3062, 3030, 2919, 1711, 1496, 1439, 1025, 919 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for C₁₈H₁₈NaOS: 305.1; Found: 305.1 [M+Na]⁺.

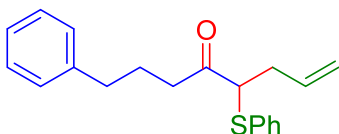
1-phenyl-4-(phenylthio)hept-6-en-3-one (4c)



Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4c** (48 mg, 81 %) as pale yellow oil. **¹H NMR** (600 MHz, CDCl₃) δ 7.32 – 7.26 (m, 7H), 7.22 –

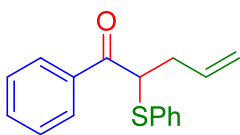
7.16 (m, 3H), 5.82 – 7.70 (m, 1H), 5.13 – 5.05 (m, 2H), 3.63 (dd, $J = 7.9, 7.1$ Hz, 1H), 3.04 – 2.82 (m, 4H), 2.61 – 2.48 (m, 1H), 2.48 – 2.37 (m, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 205.56, 141.10, 134.39, 133.45, 132.42, 129.28, 128.67, 128.65, 128.44, 126.32, 118.09, 56.41, 41.77, 34.62, 30.06. IR (neat): 3062, 3027, 2924, 1709, 1439, 919, 748 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{19}\text{H}_{20}\text{NaOS}$: 319.1; Found: 319.1 $[\text{M}+\text{Na}]^+$.

1-phenyl-5-(phenylthio)oct-7-en-4-one (4d)



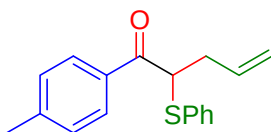
Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4d** (51 mg, 82 %) as pale yellow oil. ^1H NMR (600 MHz, CDCl_3) δ 7.39 – 7.33 (m, 2H), 7.32 – 7.25 (m, 5H), 7.22 – 7.14 (m, 3H), 5.84 – 5.74 (m, 1H), 5.13 – 5.07 (m, 2H), 3.66 (dd, $J = 7.9, 7.1$ Hz, 1H), 2.72 – 2.50 (m, 5H), 2.50 – 2.40 (m, 1H), 1.97 – 1.83 (m, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ 206.38, 141.79, 134.47, 133.25, 132.74, 129.28, 128.66, 128.56, 128.37, 126.12, 118.10, 56.36, 39.50, 35.27, 34.79, 25.50. IR (neat): 3062, 3026, 2928, 2858, 1708, 1439, 747 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{20}\text{H}_{22}\text{NaOS}$: 333.1; Found: 333.2 $[\text{M}+\text{Na}]^+$.

1-phenyl-2-(phenylthio)pent-4-en-1-one (4e)



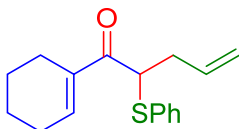
Purified by flash column chromatography (hexanes/ethyl acetate, 20/1) to afford **4e** (46 mg, 86 %) as pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 7.92 (dd, $J = 8.4, 1.2$ Hz, 2H), 7.65 – 7.50 (m, 1H), 7.50 – 7.39 (m, 2H), 7.38 – 7.22 (m, 5H), 5.92 – 5.84 (m, 1H), 5.20 – 5.02 (m, 2H), 4.50 (dd, $J = 7.8, 6.8$ Hz, 1H), 2.81 – 2.73 (m, 1H), 2.63 – 2.56 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 195.37, 136.29, 134.99, 134.98, 133.27, 131.70, 129.16, 128.97, 128.78, 128.76, 117.95, 51.02, 35.27. IR (neat): 3061, 2923, 2853, 1678, 1596, 1447, 1240, 917, 748 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{17}\text{H}_{16}\text{NaOS}$: 291.1; Found: 291.1 $[\text{M}+\text{Na}]^+$. Data was in accordance with that reported in the literature ⁷

2-(phenylthio)-1-(p-tolyl)pent-4-en-1-one (4f)



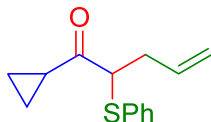
Purified by flash column chromatography (hexanes/ethyl acetate, 20/1) to afford **4f** (45 mg, 80 %) as pale yellow oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.92 – 7.75 (m, 2H), 7.43 – 7.19 (m, 7H), 5.93 – 5.83 (m, 1H), 5.19 – 4.98 (m, 2H), 4.49 (dd, $J = 7.9, 6.7$ Hz, 1H), 2.81 – 2.71 (m, 1H), 2.62 – 2.53 (m, 1H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 195.11, 144.16, 135.07, 134.87, 133.70, 131.91, 129.49, 129.13, 128.90, 128.86, 117.85, 50.95, 35.36, 21.86. **IR** (neat): 3076, 2921, 1675, 1607, 1438, 1247, 1183, 749 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{18}\text{H}_{18}\text{NaOS}$: 305.1; Found: 305.1 $[\text{M}+\text{Na}]^+$.

1-(cyclohex-1-en-1-yl)-2-(phenylthio)pent-4-en-1-one (4g)



Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4g** (42 mg, 78 %) as pale yellow oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.47 – 7.35 (m, 2H), 7.35 – 7.27 (m, 3H), 6.71 – 6.61 (m, 1H), 5.89 – 5.71 (m, 1H), 5.16 – 4.98 (m, 2H), 4.25 (dd, $J = 8.1, 6.5$ Hz, 1H), 2.71 – 2.59 (m, 1H), 2.53 – 2.42 (m, 1H), 2.35 – 2.25 (m, 1H), 2.23 – 2.03 (m, 2H), 1.72 – 1.50 (m, 4H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 197.01, 140.45, 138.64, 135.18, 134.68, 133.00, 129.07, 128.60, 117.64, 49.79, 35.65, 26.32, 23.79, 22.13, 21.62. **IR** (neat): 3075, 2933, 2860, 1660, 1636, 1438, 919, 748 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{17}\text{H}_{20}\text{NaOS}$: 295.1; Found: 295.1 $[\text{M}+\text{Na}]^+$.

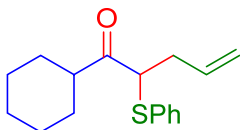
1-cyclopropyl-2-(phenylthio)pent-4-en-1-one (4h)



Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4h** (34.4 mg, 74 %) as pale yellow oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.47 – 7.35 (m, 2H), 7.32 – 7.23 (m, 3H), 5.92 – 5.76 (m, 1H), 5.20 – 5.05 (m, 2H), 3.82 (dd, $J = 7.9, 7.1$ Hz, 1H),

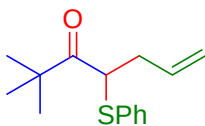
2.70 – 2.58 (m, 1H), 2.56 – 2.44 (m, 1H), 2.23 (tt, $J = 7.8, 4.6$ Hz, 1H), 1.08 – 0.99 (m, 1H), 0.99 – 0.80 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 206.61, 134.53, 133.33, 132.75, 129.18, 128.26, 117.96, 57.54, 34.86, 19.00, 11.83, 11.72. IR (neat): 3077, 3008, 2921, 1694, 1439, 1381, 1086, 918, 744 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{14}\text{H}_{16}\text{NaOS}$: 255.1; Found: 255.1 $[\text{M}+\text{Na}]^+$.

1-cyclohexyl-2-(phenylthio)pent-4-en-1-one (4i)



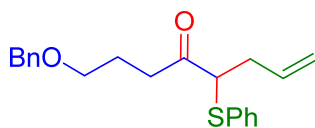
Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4i** (44 mg, 80 %) as pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 7.43 – 7.34 (m, 2H), 7.33 – 7.26 (m, 3H), 5.83 – 5.68 (m, 1H), 5.15 – 5.01 (m, 2H), 3.74 (dd, $J = 8.3, 6.5$ Hz, 1H), 2.67 (tt, $J = 11.2, 2.9$ Hz, 1H), 2.61 – 2.53 (m, 1H), 2.45 – 2.36 (m, 1H), 1.84 – 1.71 (m, 4H), 1.71 – 1.61 (m, 1H), 1.55 – 1.39 (m, 1H), 1.32 – 1.12 (m, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 208.57, 134.84, 133.91, 132.39, 129.19, 128.51, 117.92, 54.65, 48.89, 34.80, 29.66, 28.51, 26.14, 25.97, 25.55. IR (neat): 3077, 2930, 2854, 1705, 1439, 918, 746 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{17}\text{H}_{22}\text{NaOS}$: 297.1; Found: 297.2 $[\text{M}+\text{Na}]^+$.

2,2-dimethyl-4-(phenylthio)hept-6-en-3-one (4j)



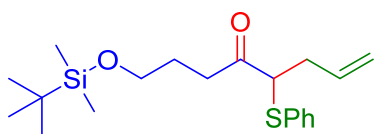
The reaction was run at 35 °C. Purified by flash column chromatography (hexanes/ethyl acetate, 50/1) to afford **4j** (13.6 mg, 28 %) as pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 7.46 – 7.38 (m, 2H), 7.36 – 7.28 (m, 3H), 5.77 – 5.65 (m, 1H), 5.12 – 5.00 (m, 2H), 4.02 (dd, $J = 8.8, 5.9$ Hz, 1H), 2.67 – 2.53 (m, 1H), 2.47 – 2.39 (m, 1H), 1.18 (s, $J = 3.7$ Hz, 9H). ^{13}C NMR (126 MHz, CDCl_3) δ 210.46, 134.95, 134.41, 132.59, 129.12, 128.62, 118.12, 49.82, 44.24, 36.90, 27.06. IR (neat): 3077, 2969, 2932, 2870, 1701, 1476, 1439, 1068, 949, 749 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{15}\text{H}_{20}\text{NaOS}$: 271.1; Found: 271.1 $[\text{M}+\text{Na}]^+$.

1-(benzyloxy)-5-(phenylthio)oct-7-en-4-one (4k)



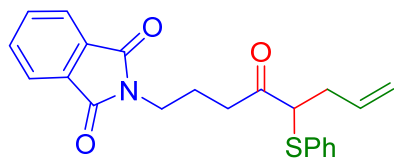
Work-up needed. Purified by flash column chromatography (hexanes/ethyl acetate, 10/1) to afford **4k** (54.2 mg, 80 %) as pale yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ 7.40 – 7.26 (m, 10H), 5.85 – 5.73 (m, 1H), 5.13 – 5.05 (m, 2H), 4.47 (s, 2H), 3.69 (dd, *J* = 8.0, 7.0 Hz, 1H), 3.46 (t, *J* = 6.2 Hz, 2H), 2.81 – 2.62 (m, 2H), 2.60 – 2.41 (m, 2H), 1.94 – 1.84 (m, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 206.36, 138.60, 134.53, 133.22, 132.78, 129.28, 128.56, 128.33, 127.83, 127.76, 118.05, 73.04, 69.45, 56.42, 36.91, 34.74, 24.20. **IR** (neat): 3062, 2925, 2855, 1439, 1099, 745 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for C₂₁H₂₄NaO₂S: 363.2; Found: 363.2 [M+Na]⁺.

1-((*tert*-butyldimethylsilyl)oxy)-5-(phenylthio)oct-7-en-4-one (4l)



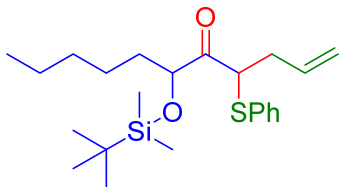
Purified by flash column chromatography (hexanes/ethyl acetate, 20/1) to afford **4l** (60 mg, 82 %) as pale yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ 7.42 – 7.35 (m, 2H), 7.34 – 7.23 (m, 3H), 5.87 – 5.74 (m, 1H), 5.16 – 5.05 (m, 2H), 3.69 (dd, *J* = 8.1, 6.9 Hz, 1H), 3.58 (t, *J* = 6.2 Hz, 2H), 2.80 – 2.68 (m, 1H), 2.68 – 2.52 (m, 2H), 2.51 – 2.40 (m, 1H), 1.80 – 1.72 (m, 2H), 0.88 (s, 9H), 0.03 (s, 6H). **¹³C NMR** (126 MHz, CDCl₃) δ 206.70, 134.54, 133.23, 132.82, 129.28, 128.35, 118.06, 62.27 (t, *J* = 3.6 Hz), 56.46 (d, *J* = 7.0 Hz), 36.54, 34.84, 27.16, 26.15 (d, *J* = 3.8 Hz), 18.50, -5.11 (d, *J* = 6.0 Hz). **IR** (neat): 3077, 2955, 2929, 2885, 2857, 1709, 1472, 1439, 1255, 1097, 836, 745 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for C₂₀H₃₂NaO₂SSi: 387.2; Found: 287.2 [M+Na]⁺.

2-(4-oxo-5-(phenylthio)oct-7-en-1-yl)isoindoline-1,3-dione (4m)



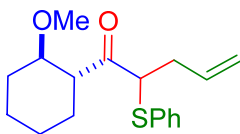
Work-up needed. Purified by flash column chromatography (hexanes/ethyl acetate, 5/1) to afford **4m** (55 mg, 73 %) as white solid. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.89 – 7.79 (m, 2H), 7.76 – 7.69 (m, 2H), 7.38 – 7.32 (m, 2H), 7.28 – 7.22 (m, 3H), 5.84 – 5.74 (m, 1H), 5.14 – 5.05 (m, 2H), 3.71 – 3.63 (m, 3H), 2.71 (ddd, $J = 17.5, 8.6, 6.1$ Hz, 1H), 2.61 – 2.52 (m, 2H), 2.47 – 2.39 (m, 1H), 2.03 – 1.86 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 205.40, 168.55, 134.47, 134.17, 133.39, 132.57, 132.30, 129.30, 128.45, 123.47, 118.15, 56.32, 37.66, 37.55, 34.65, 23.22. **IR** (neat): 3075, 2925, 2851, 1770, 1707, 1394, 750 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{22}\text{H}_{21}\text{NNaO}_3\text{S}$: 418.1; Found: 418.1 $[\text{M}+\text{K}]^+$.

2-(4-oxo-5-(phenylthio)oct-7-en-1-yl)isoindoline-1,3-dione (**4n**)



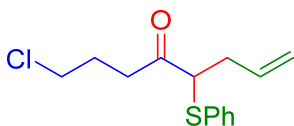
Purified by flash column chromatography (hexanes/ethyl acetate, 30/1) to afford **4n** (56 mg, 69 %, dr = 57/43) as pale yellow oil. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.43 (dd, $J = 6.1, 2.5$ Hz, 1H), 7.37 (d, $J = 8.0$ Hz, 1H), 7.33 – 7.27 (m, 3H), 5.83 – 5.71 (m, 1H), 5.12 – 5.03 (m, 2H), 4.41 – 4.11 (m, 2H), 2.61 – 2.33 (m, 2H), 1.77 – 1.61 (m, 2H), 1.42 – 1.24 (m, 6H), 0.93 (s, 9H, minor diastereoisomer), 0.91 (s, 9H, major diastereoisomer), 0.89 – 0.84 (m, 3H), 0.13 (s, 3H, minor diastereoisomer), 0.10 (s, 3H, minor diastereoisomer), 0.06 (s, 6H, major diastereoisomer). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 207.75, 207.22, 134.93, 134.78, 134.75, 133.62, 133.02, 131.49, 129.18, 129.05, 128.78, 128.36, 118.18, 117.93, 78.08, 76.92, 49.62, 48.88, 35.85, 35.05, 34.58, 32.04, 31.79, 26.11, 26.03, 25.26, 24.09, 22.69, 22.68, 18.36, 14.21, 14.18, -4.22, -4.37, -4.46, -4.56. **IR** (neat): 3078, 2955, 2930, 2858, 1710, 1472, 1257, 1096, 837, 777, 747 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{23}\text{H}_{38}\text{KO}_2\text{SSi}$: 445.2; Found: 445.2 $[\text{M}+\text{K}]^+$.

1-(2-methoxycyclohexyl)-2-(phenylthio)pent-4-en-1-one (**4o**)



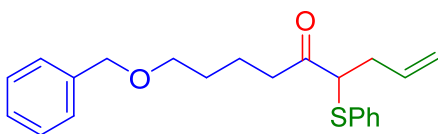
Work-up needed. Purified by flash column chromatography (hexanes/ethyl acetate, 20/1) to afford **4o** (43.2 mg, 71 %, dr = 51/49) as pale yellow oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.43 – 7.35 (m, 2H), 7.32 – 7.22 (m, 3H), 5.86 – 5.71 (m, 1H), 5.14 – 5.01 (m, 2H), 3.81 (t, J = 7.3 Hz, 1H, minor diastereoisomer), 3.72 (t, J = 7.2 Hz, 1H, major diastereoisomer), 3.42 (td, J = 10.2, 4.2 Hz, 1H, minor diastereoisomer), 3.30 (s, 3H, minor diastereoisomer), 3.23 – 3.16 (m, 4H, major diastereoisomer), 2.93 – 2.85 (m, 1H, minor diastereoisomer), 2.80 – 2.72 (m, 1H, major diastereoisomer), 2.64 – 2.55 (m, 1H), 2.44 – 2.30 (m, 1H), 2.25 – 2.13 (m, 1H), 1.92 – 1.51 (m, 4H), 1.19 – 1.02 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 208.70, 207.89, 135.35, 134.79, 133.83, 133.11, 132.85, 132.31, 129.11, 128.97, 128.35, 127.90, 117.82, 117.13, 83.15, 80.00, 57.70, 56.65, 56.45, 55.94, 55.18, 53.62, 34.41, 34.00, 30.71, 30.11, 29.62, 28.98, 25.39, 24.97, 24.35, 24.34. **IR** (neat): 3076, 2933, 2858, 2825, 1707, 1439, 1100, 917, 747 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{18}\text{H}_{24}\text{NaO}_2\text{S}$: 327.2; Found: 327.2 $[\text{M}+\text{Na}]^+$

1-chloro-5-(phenylthio)oct-7-en-4-one (4p)



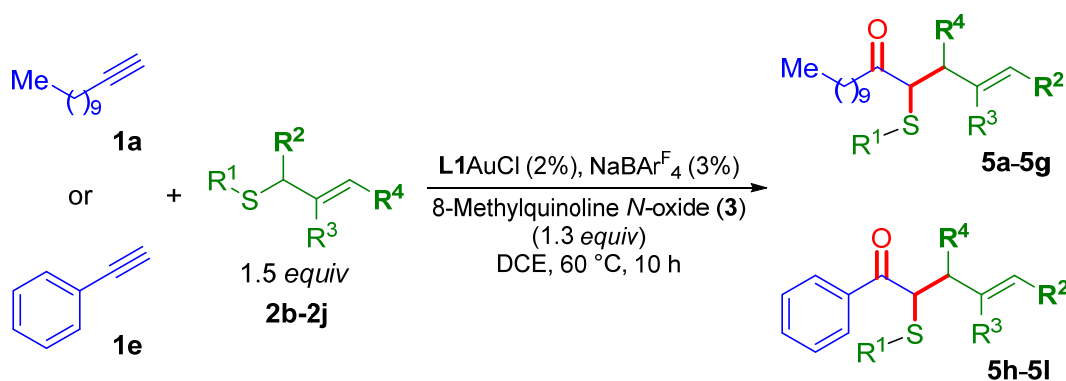
Purified by flash column chromatography (hexanes/ethyl acetate, 20/1) to afford **4p** (43.4 mg, 81 %) as pale yellow oil. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.43 – 7.35 (m, 2H), 7.34 – 7.27 (m, 3H), 5.86 – 5.74 (m, 1H), 5.16 – 5.07 (m, 2H), 3.70 (dd, J = 8.2, 6.9 Hz, 1H), 3.53 (t, J = 6.3 Hz, 2H), 2.85 (dt, J = 17.9, 7.0 Hz, 1H), 2.71 (dt, J = 17.9, 6.9 Hz, 1H), 2.64 – 2.54 (m, 1H), 2.53 – 2.44 (m, 1H), 2.03 (p, J = 6.7 Hz, 2H). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 205.61, 134.27, 133.25, 132.61, 129.35, 128.49, 118.26, 56.47, 44.55, 36.82, 34.81, 26.72. **IR** (neat): 3077, 2923, 1708, 1481, 1439, 920, 748 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{14}\text{H}_{17}\text{ClNaOS}$: 291.1; Found: 291.1 $[\text{M}+\text{Na}]^+$.

9-(benzyloxy)-4-(phenylthio)non-1-en-5-one (4q)



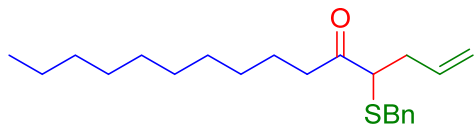
Work-up needed. Purified by flash column chromatography (hexanes/ethyl acetate, 10/1) to afford **4q** (50 mg, 71 %) as a pale yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ 7.42 – 7.36 (m, 2H), 7.36 – 7.31 (m, 4H), 7.31 – 7.24 (m, 4H), 5.79 (ddt, *J* = 17.0, 10.2, 6.8 Hz, 1H), 5.13 – 5.06 (m, 2H), 4.49 (s, 2H), 3.67 (dd, *J* = 8.1, 6.9 Hz, 1H), 3.46 (t, *J* = 6.2 Hz, 2H), 2.71 – 2.62 (m, 1H), 2.61 – 2.52 (m, 2H), 2.49 – 2.41 (m, 1H), 1.70 – 1.56 (m, 4H). **¹³C NMR** (126 MHz, CDCl₃) δ 206.45, 138.73, 134.47, 133.18, 132.79, 129.26, 128.55, 128.32, 127.82, 127.71, 118.06, 77.48, 77.23, 76.98, 73.09, 70.16, 56.27, 39.89, 34.79, 29.33, 20.79. **IR** (neat): 3062, 2925, 2854, 1439, 1360, 1100, 919, 745 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for [C₂₂H₂₇O₂S]: 355.17; Found: 355.17[M+H]⁺.

4. Synthesis of target compounds 5 series



Compound **5** series were prepared using the same procedure of Compound **4** series.

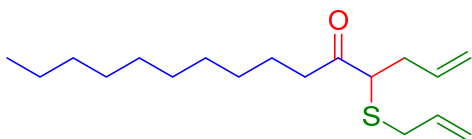
4-(benzylthio)pentadec-1-en-5-one (**5a**)



Purified by flash chromatography (hexanes/ethyl acetate, 30/1) to afford **5a** (41.3 mg, 60 %) as a pale yellow oil. **¹H NMR** (600 MHz, CDCl₃) δ 7.33 – 7.27 (m, 4H), 7.26 – 7.21 (m, 1H), 5.78 – 5.67 (m, 1H), 5.11 – 5.00 (m, 2H), 3.67 (d, *J* = 13.0 Hz, 1H), 3.59 (d, *J* = 13.0 Hz, 1H), 3.25 (dd, *J* = 8.3, 7.0 Hz, 1H), 2.63 – 2.56 (m, 1H), 2.55 – 2.50 (m, 2H), 2.43 – 2.36 (m, 1H), 1.59 – 1.51 (m, 2H), 1.33 – 1.22 (m, 14H), 0.88 (t, *J* = 7.0 Hz, 3H).

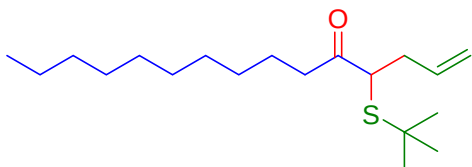
^{13}C NMR (151 MHz, CDCl_3) δ 206.55, 137.54, 134.67, 129.34, 128.76, 127.45, 117.71, 52.53, 39.36, 34.93, 34.45, 32.12, 29.80, 29.73, 29.63, 29.55, 29.42, 24.20, 22.91, 14.34. **IR** (neat): 3063, 3029, 2957, 2925, 2854, 1704, 1495, 1454, 917 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{22}\text{H}_{34}\text{NaOS}$: 369.2; Found: 369.2 $[\text{M}+\text{Na}]^+$.

4-(allylthio)pentadec-1-en-5-one (5b)



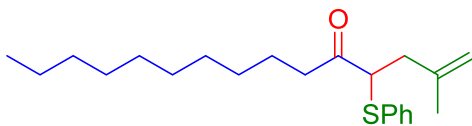
Purified by flash chromatography (hexanes/ethyl acetate, 50/1) to afford **5b** (43.2 mg, 76 %) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 5.82 – 5.67 (m, 2H), 5.23 – 5.00 (m, 4H), 3.35 – 3.20 (m, 1H), 3.16 – 2.98 (m, 2H), 2.65 – 2.48 (m, 3H), 2.46 – 2.33 (m, 1H), 1.66 – 1.48 (m, 2H), 1.34 – 1.19 (m, 14H), 0.87 (t, $J = 7.0$ Hz, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 206.71, 134.69, 133.64, 118.28, 117.71, 52.04, 39.32, 34.64, 33.68, 32.09, 29.77, 29.69, 29.61, 29.51, 29.38, 24.17, 22.88, 14.31. **IR** (neat): 3082, 2955, 2925, 2855, 1705, 1639, 1466, 1439, 989, 918 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{18}\text{H}_{32}\text{NaOS}$: 319.2; Found: 319.2 $[\text{M}+\text{Na}]^+$.

4-(*tert*-butylthio)pentadec-1-en-5-one (5c)



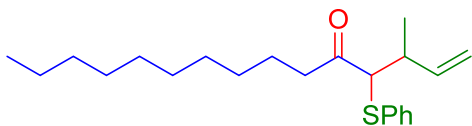
Purified by flash chromatography (hexanes/ethyl acetate, 50/1) to afford **5c** (29.3 mg, 47 %) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 5.80 – 5.67 (m, 1H), 5.12 – 5.01 (m, 2H), 3.33 (dd, $J = 9.4, 6.1$ Hz, 1H), 2.66 (dt, $J = 17.0, 7.4$ Hz, 1H), 2.62 – 2.54 (m, 1H), 2.49 (dt, $J = 17.0, 7.4$ Hz, 1H), 2.44 – 2.36 (m, 1H), 1.61 – 1.56 (m, 2H), 1.33 (s, 9H), 1.29 – 1.24 (m, 14H), 0.88 (t, $J = 7.0$ Hz, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 209.17, 134.88, 117.67, 51.91, 44.78, 38.49, 37.50, 32.12, 31.40, 29.81, 29.72, 29.65, 29.54, 29.44, 24.15, 22.91, 14.34. **IR** (neat): 3081, 2957, 2925, 2855, 1708, 1460, 1366, 1160, 917 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{19}\text{H}_{36}\text{NaOS}$, 335.2; Found 335.2 $[\text{M}+\text{Na}]^+$.

2-methyl-4-(phenylthio)pentadec-1-en-5-one (5d)



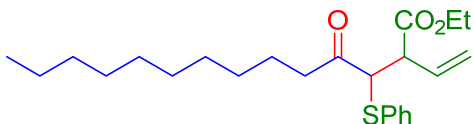
Purified by flash chromatography (hexanes/ethyl acetate, 30/1) to afford **5d** (50.5 mg, 73 %) as a pale yellow oil. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.38 (dd, $J = 7.8, 1.4$ Hz, 2H), 7.32 – 7.27 (m, 3H), 4.83 (s, 1H), 4.73 (s, 1H), 3.84 (dd, $J = 8.8, 6.6$ Hz, 1H), 2.60 – 2.54 (m, 3H), 2.42 (dd, $J = 14.9, 6.5$ Hz, 1H), 1.74 (s, 3H), 1.57 – 1.49 (m, 2H), 1.32 – 1.18 (m, 14H), 0.88 (t, $J = 7.0$ Hz, 3H). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 206.95, 141.84, 133.15, 132.89, 129.24, 128.17, 113.47, 55.02, 39.72, 38.70, 32.11, 29.77, 29.69, 29.59, 29.53, 29.38, 23.99, 22.90, 22.66, 14.33. **IR** (neat): 3077, 2925, 2854, 1709, 1456, 1439, 895, 747 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{22}\text{H}_{34}\text{NaOS}$: 369.2; Found: 369.2 $[\text{M}+\text{Na}]^+$.

3-methyl-4-(phenylthio)pentadec-1-en-5-one (5e)



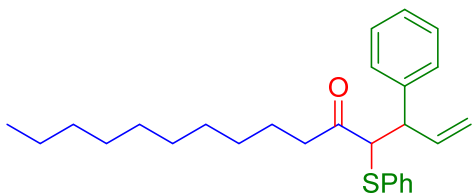
Purified by flash chromatography (hexanes/ethyl acetate, 30/1) to afford **5e** (51.2 mg, 74 %, dr = 65/35) as a pale yellow oil. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.40 – 7.34 (m, 2H), 7.31 – 7.21 (m, 3H), 5.93 – 5.82 (m, 1H, major diastereoisomer), 5.76 – 5.66 (m, 1H, minor diastereoisomer), 5.18 – 5.10 (m, 2H, major diastereoisomer), 5.09 – 4.98 (m, 2H, minor diastereoisomer), 3.53 (d, $J = 9.6$ Hz, 1H, major diastereoisomer), 3.50 (d, $J = 9.9$ Hz, 1H, minor diastereoisomer), 2.71 – 2.62 (m, 1H), 2.59 – 2.50 (m, 1H), 2.49 – 2.37 (m, 1H), 1.53 – 1.45 (m, 2H), 1.28 (d, $J = 6.8$ Hz, 2H, minor diastereoisomer), 1.27 – 1.18 (m, 14H), 1.08 (d, $J = 6.7$ Hz, 2H, major diastereoisomer), 0.88 (t, $J = 7.0$ Hz, 3H). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 207.32, 207.06, 140.43, 140.35, 134.16, 134.04, 132.46, 132.37, 129.32, 129.25, 127.91, 127.90, 116.09, 116.04, 63.80, 63.08, 40.61, 40.51, 38.97, 38.95, 32.15, 29.81, 29.71, 29.62, 29.56, 29.40, 24.02, 23.92, 22.94, 19.19, 18.50, 14.37. **IR** (neat): 3078, 2956, 2925, 2854, 1708, 1466, 917, 744 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{22}\text{H}_{34}\text{NaOS}$: 369.2; Found: 369.2 $[\text{M}+\text{Na}]^+$.

Ethyl 4-oxo-3-(phenylthio)-2-vinyltetradecanoate (5f)



Work-up needed. Purified by flash chromatography (hexanes/ethyl acetate, 10/1) to afford **5f** (59 mg, 73 %, dr = 75/25) as a pale yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ 7.48 – 7.35 (m, 2H), 7.35 – 7.27 (m, 3H), 5.90 (ddd, *J* = 17.1, 10.2, 8.4 Hz, 1H, major diastereoisomer), 5.75 (ddd, *J* = 17.2, 10.2, 8.5 Hz, 1H, minor diastereoisomer), 5.39 – 5.27 (m, 2H, major diastereoisomer), 5.26 – 5.14 (m, 2H, minor diastereoisomer), 4.32 – 4.15 (m, 2H, minor diastereoisomer), 4.07 (q, *J* = 7.1 Hz, 2H, major diastereoisomer), 3.96 (d, *J* = 10.9 Hz, 1H, minor diastereoisomer), 3.90 (d, *J* = 11.2 Hz, 1H, major diastereoisomer), 3.57 (dd, *J* = 10.9, 8.5 Hz, 1H, minor diastereoisomer), 3.42 (dd, *J* = 11.2, 8.4 Hz, 1H, major diastereoisomer), 2.84 – 2.40 (m, 2H), 1.65 – 1.48 (m, 2H), 1.37 – 1.16 (m, 17H), 0.88 (t, *J* = 7.0 Hz, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 205.43, 203.47, 172.35, 171.41, 134.28, 133.99, 133.02, 132.96, 132.64, 132.58, 131.95, 131.01, 129.11, 128.83, 120.17, 120.11, 61.19, 61.14, 57.57, 56.71, 50.82, 49.40, 41.29, 40.90, 31.89, 31.89, 29.56, 29.54, 29.48, 29.45, 29.37, 29.33, 29.31, 29.30, 29.15, 29.08, 23.86, 23.64, 22.68, 14.17, 13.97. **IR** (neat): 3061, 2926, 2855, 1728, 1713, 1466, 1440, 1369, 1353, 1195, 1156, 1025, 924, 748 cm⁻¹; **MS** (ES⁺, *m/z*) Calculated for C₂₄H₃₆NaO₃S: 427.2; Found: 427.2 [M+Na]⁺.

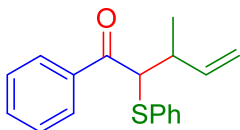
3-phenyl-4-(phenylthio)pentadec-1-en-5-one (**5g**)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **5g** (52.2 mg, 64 %, dr = 51/49) as a pale yellow oil. **¹H NMR** (600 MHz, CDCl₃) δ 7.47 – 7.40 (m, 1H), 7.38 – 7.11 (m, 9H), 6.19 (ddd, *J* = 17.1, 10.1, 8.3 Hz, 1H, major diastereoisomer), 5.95 (ddd, *J* = 16.9, 10.4, 8.2 Hz, 1H, minor diastereoisomer), 5.25 – 5.01 (m, 2H), 4.02 (dd, *J* = 16.0, 11.2 Hz, 1H), 3.75 (dd, *J* = 10.8, 8.6 Hz, 1H), 2.63 – 2.43 (m, 1H, minor diastereoisomer), 2.30 – 2.17 (m, 1H, major diastereoisomer), 1.55 – 1.48 (m, 1H), 1.31 – 1.06 (m, 14H), 1.01 – 0.93 (m, 1H), 0.91 – 0.86 (m, 3H). **¹³C NMR** (151 MHz, CDCl₃)

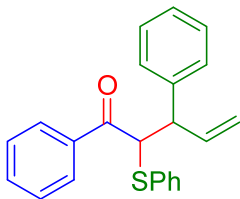
δ 206.47, 205.72, 141.29, 140.34, 138.67, 138.44, 133.44, 133.10, 129.26, 129.10, 128.92, 128.82, 128.62, 128.36, 128.25, 128.14, 127.35, 127.24, 117.63, 117.26, 62.40, 61.78, 51.06, 50.63, 41.23, 40.81, 32.12, 32.10, 29.78, 29.73, 29.69, 29.60, 29.54, 29.51, 29.47, 29.34, 29.04, 23.81, 23.59, 22.90, 22.90, 14.34. **IR** (neat): 3062, 3029, 2925, 2854, 1709, 1454, 1439, 1025, 919, 747 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{27}\text{H}_{36}\text{NaOS}$: 431.1; Found: 431.2 $[\text{M}+\text{Na}]^+$.

3-methyl-1-phenyl-2-(phenylthio)pent-4-en-1-one (5h)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **5h** (46.8 mg, 83 %, dr = 61/39) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 6.89 – 6.82 (m, 2H), 6.57 – 6.50 (m, 1H), 6.45 – 6.38 (m, 2H), 6.37 – 6.32 (m, 2H), 6.28 – 6.21 (m, 3H), 5.16 – 4.96 (m, 1H, major diastereoisomer), 4.86 – 4.69 (m, 1H, minor diastereoisomer), 4.27 – 4.14 (m, 2H, major diastereoisomer), 4.08 – 3.90 (m, 2H, minor diastereoisomer), 3.32 (d, J = 9.7 Hz, 1H), 1.98 – 1.84 (m, 1H), 0.40 (d, J = 6.8 Hz, 3H, minor diastereoisomer), 0.09 (d, J = 6.7 Hz, 3H, major diastereoisomer). **^{13}C NMR** (126 MHz, CDCl_3) δ 195.87, 140.93, 140.54, 137.00, 134.49, 134.24, 133.22, 133.10, 129.17, 129.07, 128.76, 128.73, 128.65, 128.61, 128.58, 116.04, 115.90, 58.25, 57.77, 38.94, 38.47, 19.13, 17.99. **IR** (neat): 3061, 2972, 2928, 1678, 1447, 1270, 1001, 918, 748 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{18}\text{H}_{18}\text{NaOS}$: 305.1; Found: 305.1 $[\text{M}+\text{Na}]^+$.

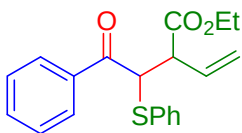
1,3-diphenyl-2-(phenylthio)pent-4-en-1-one (5i)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **5i** (57.8 mg, 84 %, dr = 67/33) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 7.93 – 7.88 (m, 2H, minor diastereoisomer), 7.73 – 7.67 (m, 2H, major diastereoisomer), 7.59 – 6.99 (m,

13H), 6.38 (ddd, $J = 17.0, 10.2, 8.2$ Hz, 1H, major diastereoisomer), 5.96 (ddd, $J = 17.0, 10.5, 7.5$ Hz, 1H, minor diastereo-isomer), 5.32 – 5.12 (m, 2H, major diastereoisomer), 5.00 – 4.93 (m, 2H, minor diastereo -isomer), 4.86 (d, $J = 11.1$ Hz, 1H, major diastereoisomer), 4.81 (d, $J = 11.0$ Hz, 1H, minor diastereoisomer), 4.10 – 3.98 (m, 1H). **^{13}C NMR** (126 MHz, CDCl_3) δ 195.54, 194.71, 141.85, 140.63, 139.20, 138.75, 137.05, 136.89, 135.08, 133.24, 132.95, 129.22, 129.14, 129.00, 128.92, 128.85, 128.78, 128.76, 128.69, 128.60, 128.38, 128.19, 127.27, 126.91, 117.83, 117.19, 56.64, 56.45, 50.90, 50.26. **IR** (neat): 3061, 3028, 2920, 2850, 1678, 1447, 1267, 985, 749 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{23}\text{H}_{20}\text{NaOS}$: 367.1; Found: 367.1 $[\text{M}+\text{Na}]^+$.

Ethyl 2-(2-oxo-2-phenyl-1-(phenylthio)ethyl)but-3-enoate (**5j**)



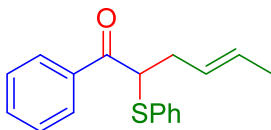
Work-up needed, then purified by flash chromatography (hexanes/ethyl acetate, 10/1) to afford **5j** (51.7 mg, 76 %, dr = 61/39) as a pale yellow oil, two diastereoisomers can be separated by PTLC (hexanes/DCM, 1/1).

Major diastereoisomer: **^1H NMR** (500 MHz, CDCl_3) δ 7.97 – 7.90 (m, 1H), 7.59 – 7.52 (m, 1H), 7.47 – 7.41 (m, 1H), 7.36 – 7.21 (m, 3H), 6.13 – 6.02 (m, 1H), 5.48 – 5.39 (m, 1H), 4.75 (d, $J = 11.1$ Hz, 1H), 4.11 – 3.98 (m, 1H), 3.64 (dd, $J = 11.1, 8.3$ Hz, 1H), 1.13 (t, $J = 7.1$ Hz, 2H). **^{13}C NMR** (126 MHz, CDCl_3) δ 194.97, 172.57, 136.06, 135.72, 133.46, 133.25, 130.30, 129.51, 129.21, 128.89, 128.75, 120.68, 61.38, 52.59, 50.00, 14.15. **IR** (neat): 3061, 2982, 2929, 1725, 1679, 1448, 1261, 1183, 1024, 750 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{20}\text{H}_{20}\text{NaO}_3\text{S}$: 363.1; Found: 363.1 $[\text{M}+\text{Na}]^+$.

Minor diastereoisomer: **^1H NMR** (500 MHz, CDCl_3) δ 7.89 – 7.83 (m, 1H), 7.58 – 7.52 (m, 1H), 7.45 – 7.39 (m, 1H), 7.37 – 7.32 (m, 2H), 7.30 – 7.27 (m, 1H), 5.82 – 5.71 (m, 1H), 5.28 – 5.08 (m, 1H), 4.82 (d, $J = 11.1$ Hz, 1H), 4.41 – 4.23 (m, 1H), 3.83 (dd, $J = 11.1, 8.2$ Hz, 1H), 1.37 (t, $J = 7.1$ Hz, 2H). **^{13}C NMR** (126 MHz, CDCl_3) δ 192.67, 172.06, 136.36, 135.88, 133.39, 132.96, 130.78, 129.54, 129.20, 128.79, 128.76, 120.36, 120.25, 61.44, 52.58, 51.22, 14.49. **IR** (neat): 3060, 2982, 1732, 1679, 1447, 1268, 1154,

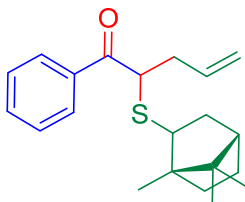
1025, 749 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{20}\text{H}_{20}\text{NaO}_3\text{S}$: 363.1; Found: 363.1 $[\text{M}+\text{Na}]^+$.

1-phenyl-2-(phenylthio)hex-4-en-1-one (5k)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **5k** (35.5 mg, 63 %) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 7.95 – 7.89 (m, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.24 (m, 3H), 5.60 – 5.43 (m, 2H), 4.47 (dd, J = 7.9, 6.6 Hz, 1H), 2.74 – 2.66 (m, 1H), 2.57 – 2.49 (m, 1H), 1.63 (dd, J = 6.0, 1.0 Hz, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 195.71, 136.42, 134.77, 133.20, 132.07, 129.11, 128.80, 128.76, 128.75, 127.36, 51.64, 34.32, 18.20. **IR** (neat): 3059, 3025, 2916, 2854, 1679, 1447, 1234, 967, 748 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{18}\text{H}_{18}\text{NaOS}$: 305.1; Found: 305.1 $[\text{M}+\text{Na}]^+$.

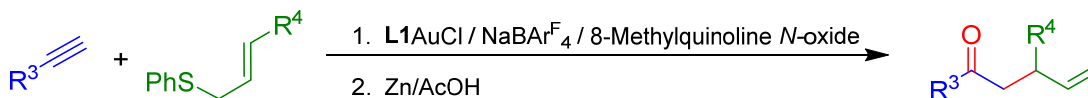
1-phenyl-2-(((1S,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)thio)pent-4-en-1-one (5l)



Purified by flash chromatography (hexanes/ethyl acetate, 30/1) to afford **5l** (51.8 mg, 79 %, dr = 60/40) as a pale yellow oil. **^1H NMR** (500 MHz, CDCl_3) δ 8.01 – 7.96 (m, 2H), 7.57 – 7.50 (m, 1H), 7.48 – 7.41 (m, 2H), 5.93 – 5.77 (m, 1H), 5.17 – 5.00 (m, 2H), 4.23 – 4.11 (m, 1H), 2.89 – 2.78 (m, 1H), 2.73 – 2.64 (m, 1H), 2.63 – 2.51 (m, 1H), 1.93 – 1.86 (m, 1H), 1.80 – 1.53 (m, 4H), 1.28 – 1.06 (m, 3H), 0.90 (s, 3H, minor diastereoisomer), 0.83 (s, 3H, major diastereoisomer), 0.81 (s, 3H, minor diastereoisomer), 0.77 (s, 3H, minor diastereoisomer), 0.74 (s, 3H, major diastereoisomer), 0.62 (s, 3H, major diastereoisomer). **^{13}C NMR** (126 MHz, CDCl_3) δ 196.53, 195.57, 147.83, 136.51, 136.49, 135.46, 135.39, 133.01, 133.01, 128.72, 128.70,

128.67, 128.65, 117.52, 117.50, 51.09, 51.03, 50.67, 50.63, 49.81, 49.79, 48.66, 48.64, 48.62, 48.58, 48.53, 48.53, 48.48, 47.36, 47.27, 46.44, 46.40, 46.26, 43.16, 42.14, 38.59, 38.56, 35.61, 35.53, 27.50, 27.47, 20.87, 20.84, 20.79, 20.75, 20.06, 20.02, 19.99, 19.96, 14.78, 14.75, 14.22, 14.21. **IR** (neat): 3077, 2953, 2878, 1674, 1448, 1236, 916 cm^{-1} ; **MS** (ES^+ , m/z) Calculated for $\text{C}_{21}\text{H}_{28}\text{NaOS}$: 351.2; Found: 351.2 $[\text{M}+\text{Na}]^+$.

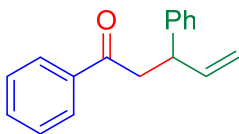
5. General Procedure for Synthesis of compound 6 and 7



To a 2 dram septum-capped vial were added sequentially alkyne (0.20 mmol), substituted allylic sulfide (0.30 mmol, 1.5 *equiv*), **L1AuCl** (2.8 mg, 4 μmol , 0.02 *equiv*), NaBARF_4 (5.4 mg, 6 μmol , 0.03 *equiv*) and 1 mL dry DCE, then 8-methylquinoline N-oxide (19.7 mg, 0.26 mmol, 1.3 *equiv*) in 1 mL dry DCE was introduced into reaction by syringe pump at the rate of 0.1 mL per hour. After stirring at 60°C for 10 h, cooled down to r.t. There is no need to purify for next step.

To above solution activated zinc powder (130 mg, 2 mmol) and acetic acid (0.5 mL) were added, the solution was stirred at 60°C for 12 h. Cooled down to r.t., the precipitate was filtered and washed by chloroform. The filtrate was washed with saturated K_2CO_3 and brine, then dried over anhydrous Na_2SO_4 . After removed the solvent, the residue was purified through flash chromatography on silica gel to afford compound **6** or **7** as following.

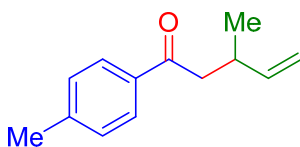
1, 3-Diphenylpent-4-en-1-one (6)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **7** (37 mg, 79 %) as a colorless oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.99 – 7.92 (m, 2H), 7.59 – 7.53 (m, 1H), 7.49 – 7.43 (m, 2H), 7.35 – 7.27 (m, 4H), 7.24 – 7.20 (m, 1H), 6.07 (ddd, $J = 17.1$, 10.3, 6.8 Hz, 1H), 5.12 – 5.01 (m, 2H), 4.21 – 4.12 (m, 1H), 3.46 (dd, $J = 16.6$, 7.7 Hz,

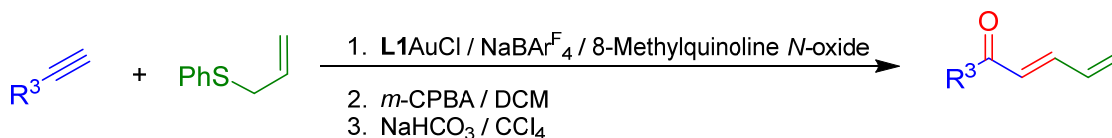
1H), 3.38 (dd, $J = 16.6, 6.5$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 198.43, 143.34, 140.86, 137.31, 133.22, 128.78, 128.77, 128.25, 127.91, 126.74, 114.91, 44.72, 44.21. IR (neat): 3062, 3029, 2979, 2898, 1686, 1598, 1449, 1205, 989, 916, 746 cm^{-1} ; MS (ES^+ , m/z) Calculated for $\text{C}_{17}\text{H}_{16}\text{NaO}$: 259.1; Found: 259.1 $[\text{M}+\text{Na}]^+$. Data was in accordance with that reported in the literature ⁸.

3-Methyl-1-(p-tolyl) pent-4-en-1-one (7)



Purified by flash chromatography (hexanes/ethyl acetate, 20/1) to afford **7** (31 mg, 82 %) as a colorless oil. ^1H NMR (500 MHz, CDCl_3) δ 7.85 (d, $J = 8.2$ Hz, 2H), 7.25 (d, $J = 7.5$ Hz, 2H), 5.85 (ddd, $J = 13.9, 10.4, 6.5$ Hz, 1H), 4.98 (ddt, $J = 32.4, 10.3, 1.3$ Hz, 2H), 3.05 – 2.95 (m, 1H), 2.93 – 2.81 (m, 2H), 2.41 (s, 3H), 1.09 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 199.22, 143.91, 143.37, 135.06, 129.45, 128.45, 113.15, 45.24, 33.91, 21.83, 20.00. IR (neat): 3081, 2963, 2927, 1682, 1606, 1282, 1181, 914, 807 cm^{-1} ; MS (ES^+ , m/z) Calc. for $\text{C}_{13}\text{H}_{16}\text{NaO}$: 211.11; Found: 211.11 $[\text{M}+\text{Na}]^+$.

6. General procedure for synthesis of compound 8 and 9



In a 8 mL septum-capped vial, alkyne (0.20 mmol), substituted allyl sulfide (0.30 mmol, 1.5 equiv), **L1**AuCl (2.8 mg, 4 μmol , 0.02 equiv), NaBARF_4 (5.4 mg, 6 μmol , 0.03 equiv) and 1 mL dry DCE were mixed and sealed, then 8-methylquinoline N-oxide (19.7 mg, 0.26 mmol, 1.3 equiv) in 1 mL DCE was introduced into reaction by syringe pump at the rate of 0.1 mL/hour. After stirring at 60 °C for 10 h, Removing the solvent under reduced pressure, the residue was purified through flash chromatography on silica gel to afford the intermediate (**4q** or **4m**, corresponding yields and spectral data, please see syntheses of compounds **4** series in details)

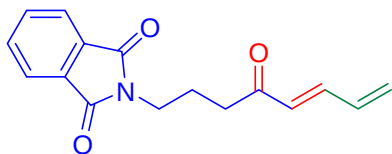
To a cooled (-78°C) solution of allylketo sulfide **4q/4m** (0.15 mmol) in DCM (4 mL), *m*-chloroperoxybenzoic acid (0.15 mmol) in DCM (2 mL) was added. After being stirred for 3 h, the mixture was hydrolysed with 5% sodium bisulfite, then extracted with DCM. The organic layers were washed with saturated NaHCO₃ solution and NaCl solution, dried over anhydrous MgSO₄ and evaporated under reduced pressure. The crude product in CCl₄ (2 mL) was refluxed for 5 h in the presence of NaHCO₃ (25 mg, 0.30 mmol). The reaction mixture was hydrolysed with water and extracted with ether. The organic layer was washed and dried. After concentration under reduced pressure the crude product was purified by flash column chromatography on silica gel to afford compound **8** or **9** as following.

(E)-9-(benzyloxy)nona-1,3-dien-5-one (8)



Synthesized from **4q** and Purified by flash chromatography (hexanes/ethyl acetate, 10/1) to afford **8** (28 mg, 76.5 %) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.31 (m, 4H), 7.31 – 7.26 (m, 1H), 7.12 (dd, *J* = 15.6, 10.8 Hz, 1H), 6.45 (dt, *J* = 16.9, 10.4 Hz, 1H), 6.17 (d, *J* = 15.7 Hz, 1H), 5.64 (d, *J* = 16.9 Hz, 1H), 5.53 (d, *J* = 10.8 Hz, 1H), 4.50 (s, 2H), 3.49 (t, *J* = 6.2 Hz, 2H), 2.60 (t, *J* = 7.3 Hz, 2H), 1.77 – 1.70 (m, 2H), 1.68 – 1.62 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 200.77, 142.57, 138.75, 135.49, 130.57, 128.57, 127.85, 127.73, 126.38, 70.23, 40.47, 29.46, 21.23. IR (neat): 3090, 3063, 3031, 2929, 2859, 1710, 1454, 1363, 1103, 736 cm⁻¹; MS (ES⁺, *m/z*) Calc. for C₁₅H₁₈NaO₂: 267.13; Found: 267.15[M+Na]⁺.

(E)-2-(4-oxoocta-5,7-dien-1-yl)isoindoline-1,3-dione (9)



Synthesized from **4m** and Purified by flash chromatography (hexanes/ethyl acetate, 5/1) to afford **9** (32 mg, 82.5 %) as a white solid. ¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.31

(m, 4H), 7.31 – 7.26 (m, 1H), 7.12 (dd, $J = 15.6, 10.8$ Hz, 1H), 6.45 (dt, $J = 16.9, 10.4$ Hz, 1H), 6.17 (d, $J = 15.7$ Hz, 1H), 5.64 (d, $J = 16.9$ Hz, 1H), 5.53 (d, $J = 10.8$ Hz, 1H), 4.50 (s, 2H), 3.49 (t, $J = 6.2$ Hz, 2H), 2.60 (t, $J = 7.3$ Hz, 2H), 1.77 – 1.70 (m, 2H), 1.68 – 1.62 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 200.77, 142.57, 138.75, 135.49, 130.57, 128.57, 127.85, 127.73, 126.38, 70.23, 40.47, 29.46, 21.23. IR (neat): 3063, 3029, 2927, 2851, 1770, 1709, 1590, 1390, 1361, 1009, 719 cm^{-1} ; MS (ES^+ , m/z) Calc. for $\text{C}_{15}\text{H}_{16}\text{NaNO}_3$: 292.09; Found: 292.09 $[\text{M}+\text{Na}]^+$.

¹ Seth B. Harkins, *et al.* Amido-Bridged Cu_2N_2 Diamond Cores that Minimize Structural Reorganization and Facilitate Reversible Redox Behavior between a $\text{Cu}^{\text{I}}\text{Cu}^{\text{I}}$ and a Class III Delocalized $\text{Cu}^{1.5}\text{Cu}^{1.5}$ Species. *J. Am. Chem. Soc.* **2004** 126 (9), 2885-2893

² Lukas H., *et al.* Aldol Synthesis by anti-Markovnikov hydration of propargyloxy substrates: feasibility, stereospecificity, and reiterative alkynylation-hydration. *Synlett*, **2009** (15), 2412-2416;

³ Fu, J., *et al.* Gold-Catalyzed Rearrangement of Allylic Oxonium Ylides: Efficient Synthesis of Highly Functionalized Dihydrofuran-3-ones. *Angew. Chem. Int. Ed.*, **2013**, 52(15): 4198-4202.

⁴ Furuta, Kyoji *et al.* Selective condensation of [3-(alkylthio)allyl]titanium reagent with carbonyl compounds. *Bull. Chem. Soc. Japan*, **1984**, 57(10), 2781-90

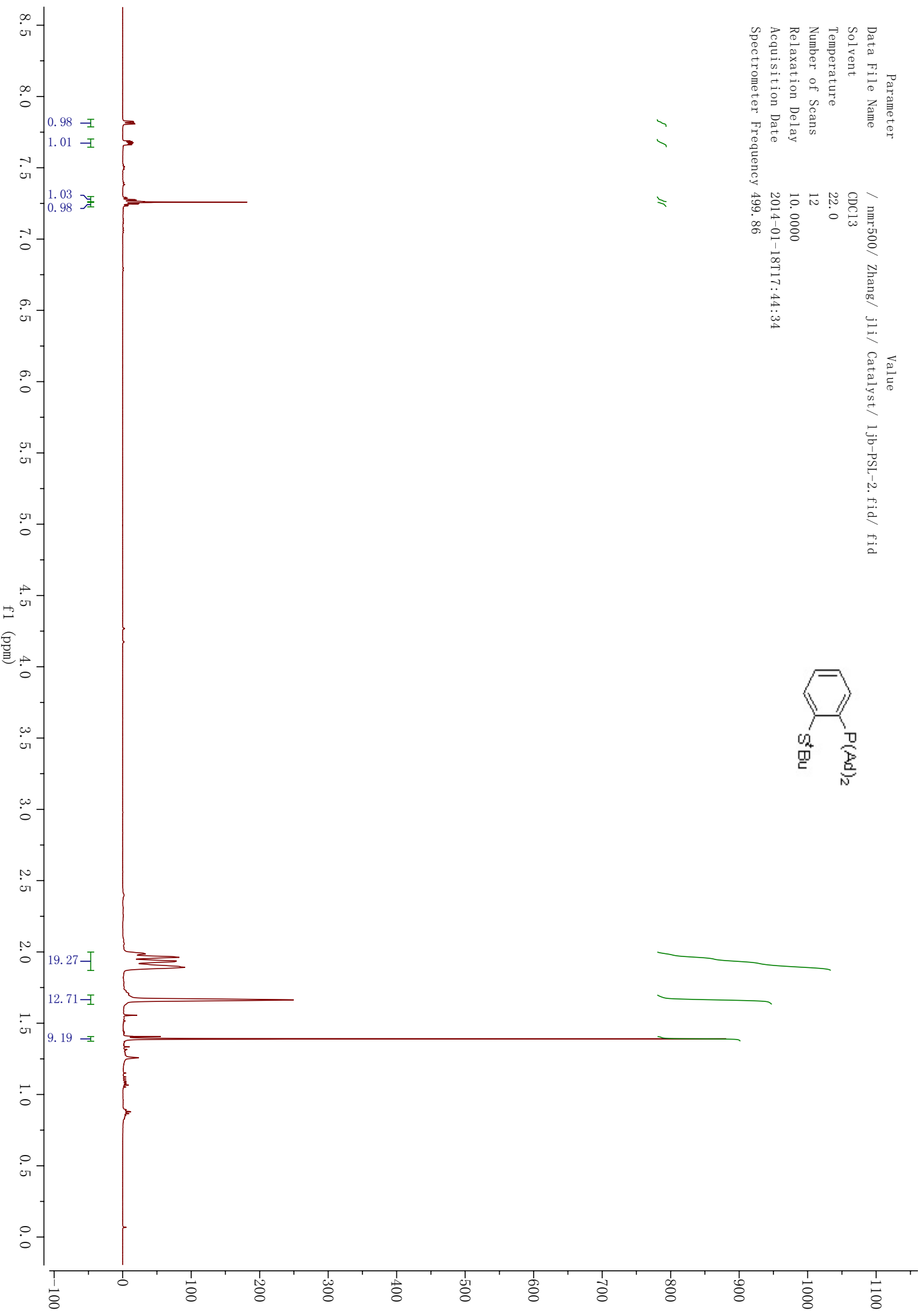
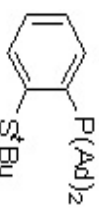
⁵ Jiang, Yubo *et al.* Catalyst-free imidation of allyl sulfides with chloramine-T and subsequent [2,3]-sigmatropic rearrangement. *Chinese J. Chem.*, **2012**, 30(9), 2029-2035;

⁶ José M. Blanco *et al.* Synthesis of Chiral Sulfinic Acids: Sodium(1S-exo)-2-Bornanesulfinate. *Synthesis*, **1990**, (7), 584-586

⁷ Liao Mingyi, *et al.* Highly efficient [2,3]-sigmatropic rearrangement of sulfur ylide derived from Rh(II) carbene and sulfides in water *Green Chemistry*, **2007**, 9(2), 184-188.

⁸ Evans P. Andrew, *et al.* Regioselective and Enantiospecific Rhodium-Catalyzed Allylic Alkylation Reactions Using Copper(I) Enolates: Synthesis of (-)-Sugiresinol Dimethyl Ether. *J. Am. Chem. Soc.*, **2003**, 125(30), 8974-8975.

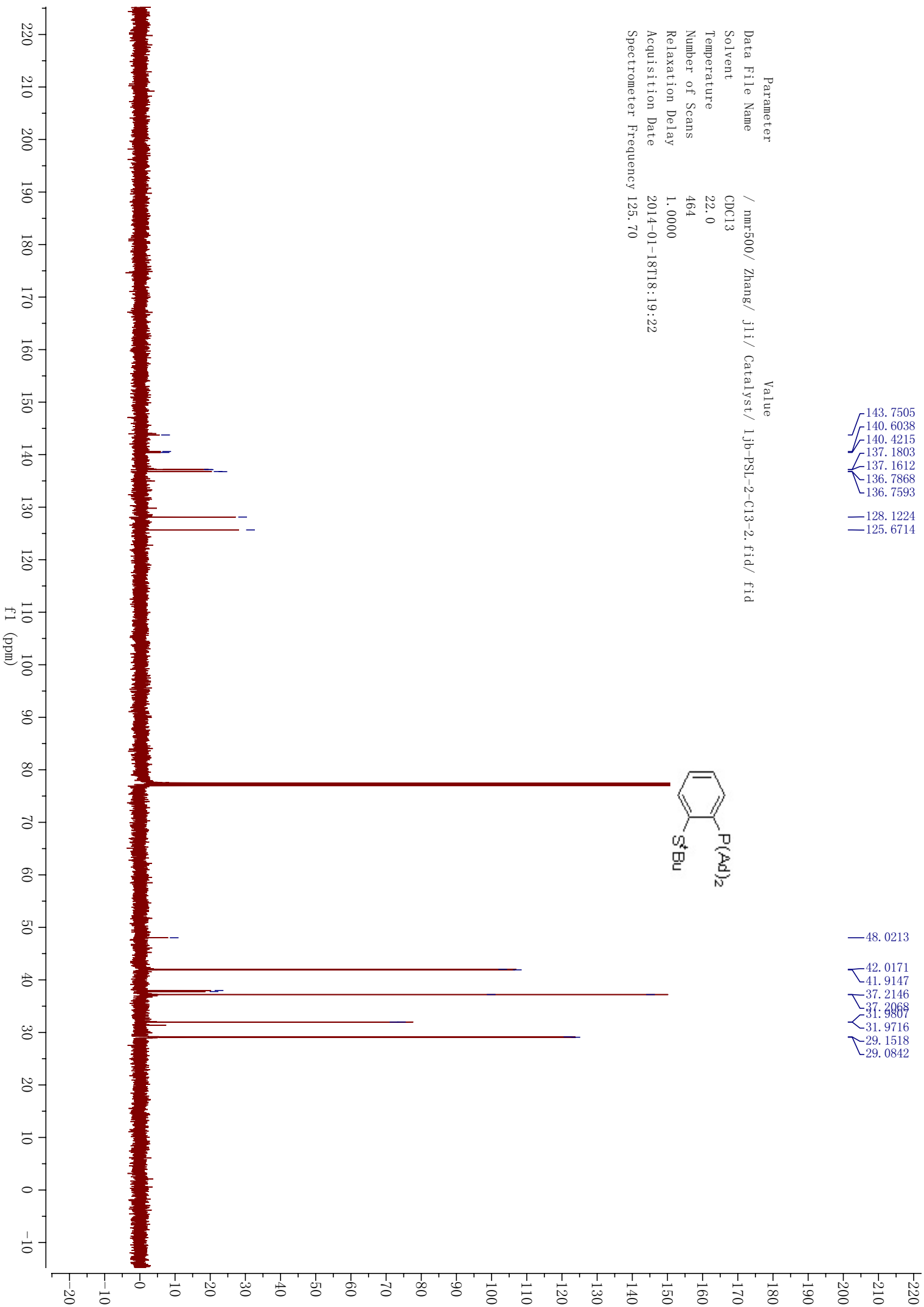
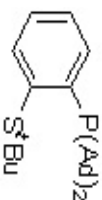
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Spectrometer Frequency	499.86



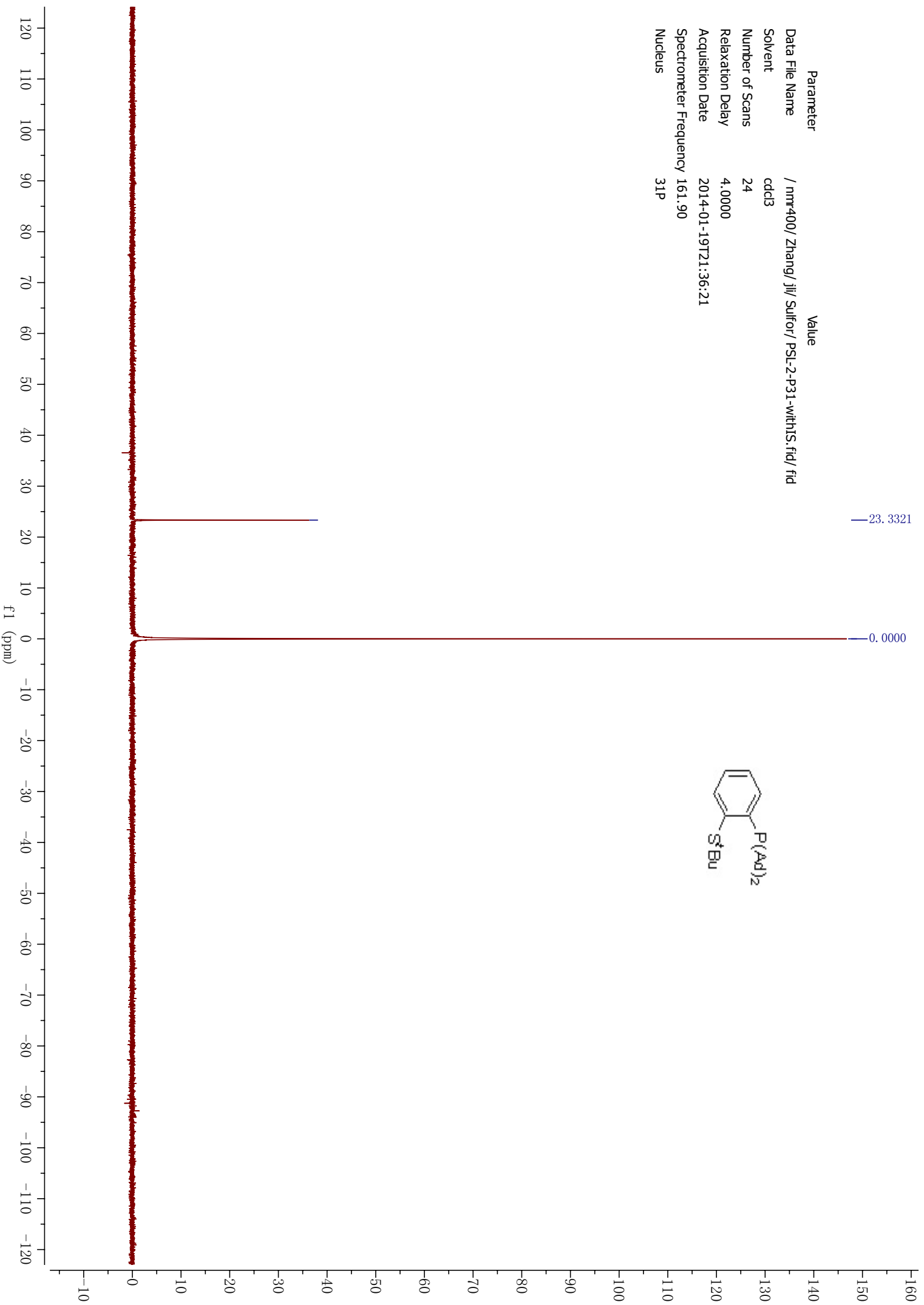
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37.2068
31.9807
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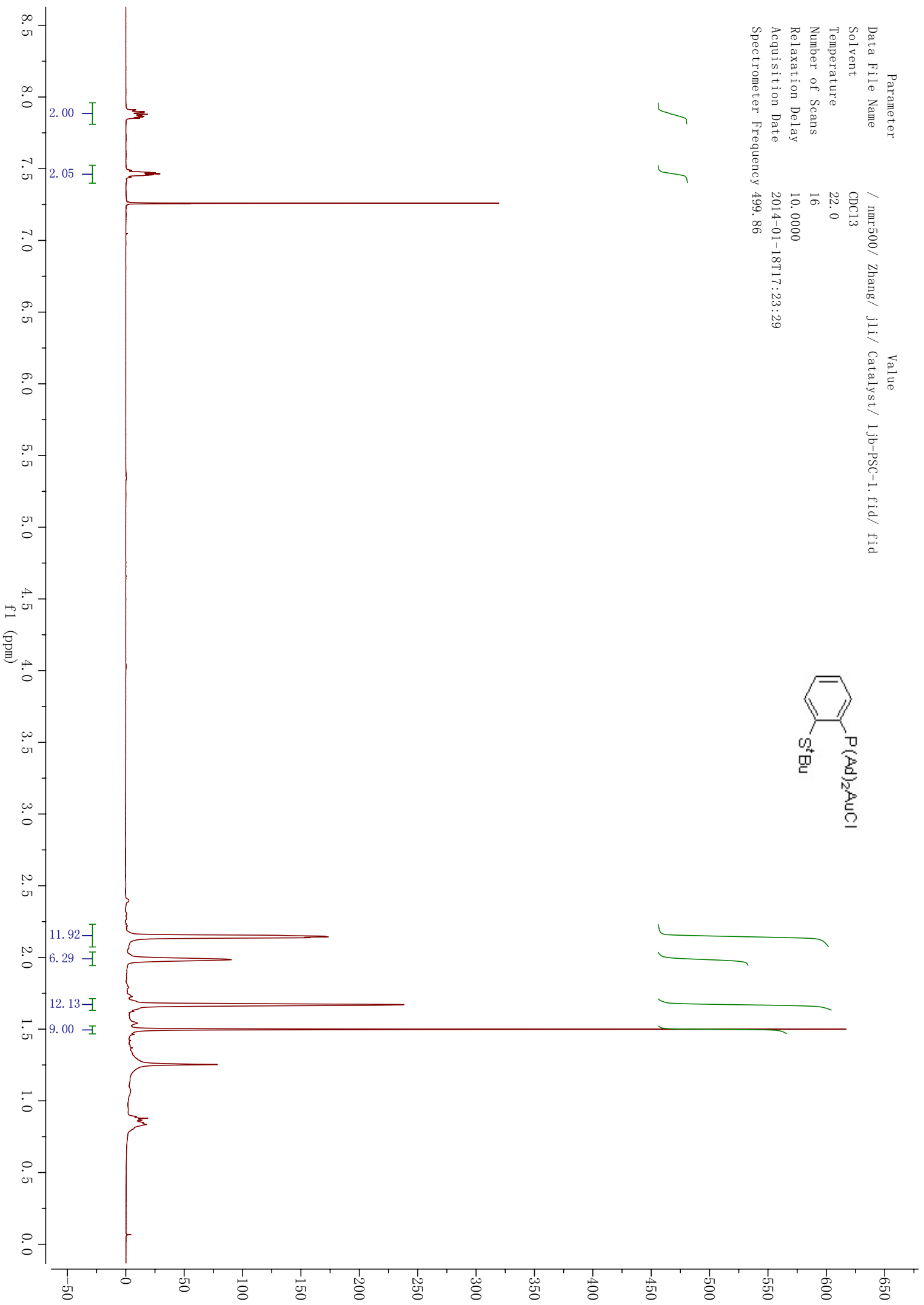
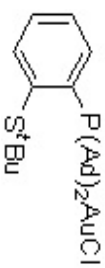
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Spectrometer Frequency	125.70



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Nucleus	31P



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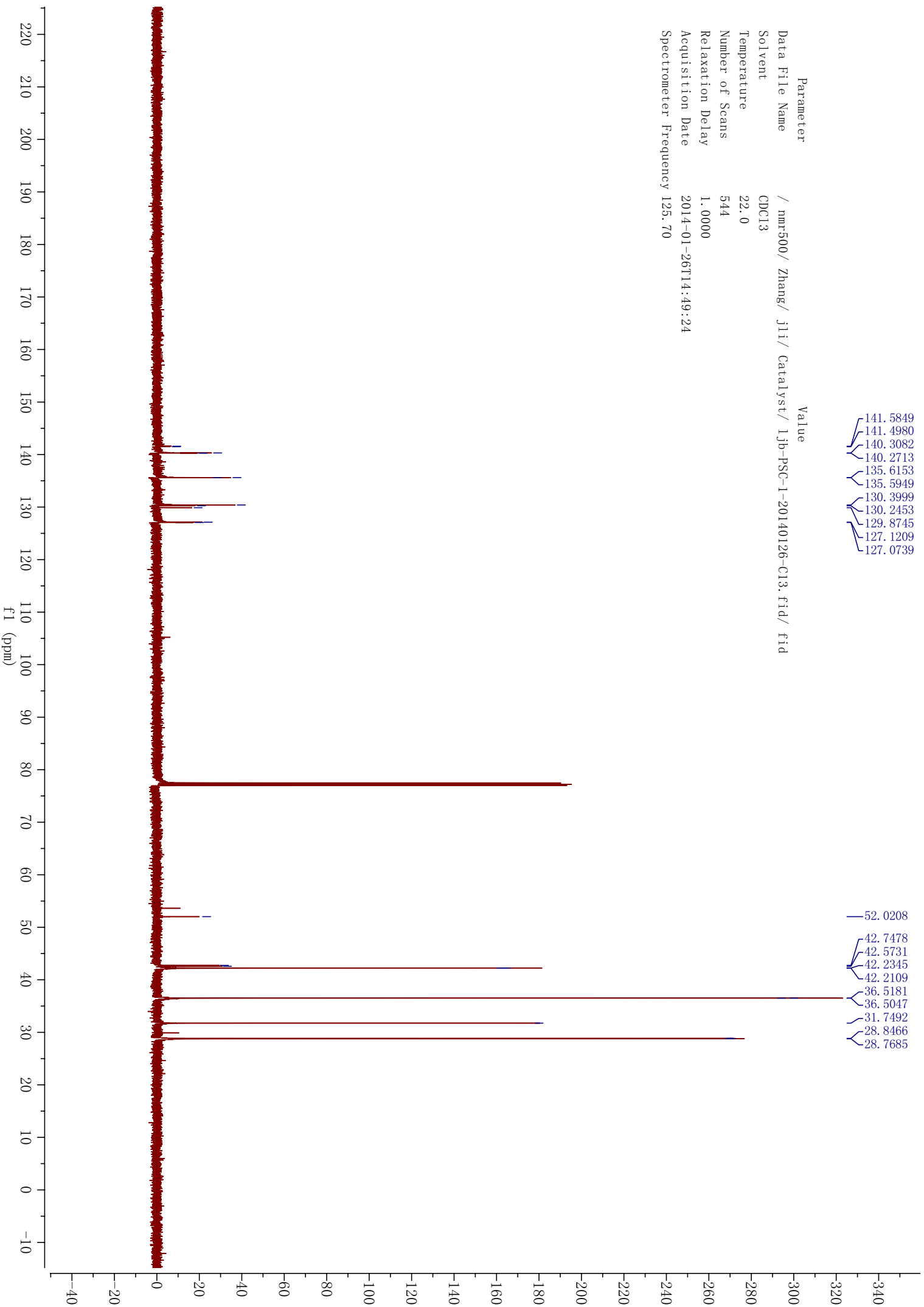
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Spectrometer Frequency	125.70

Value

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127.1209
127.0739

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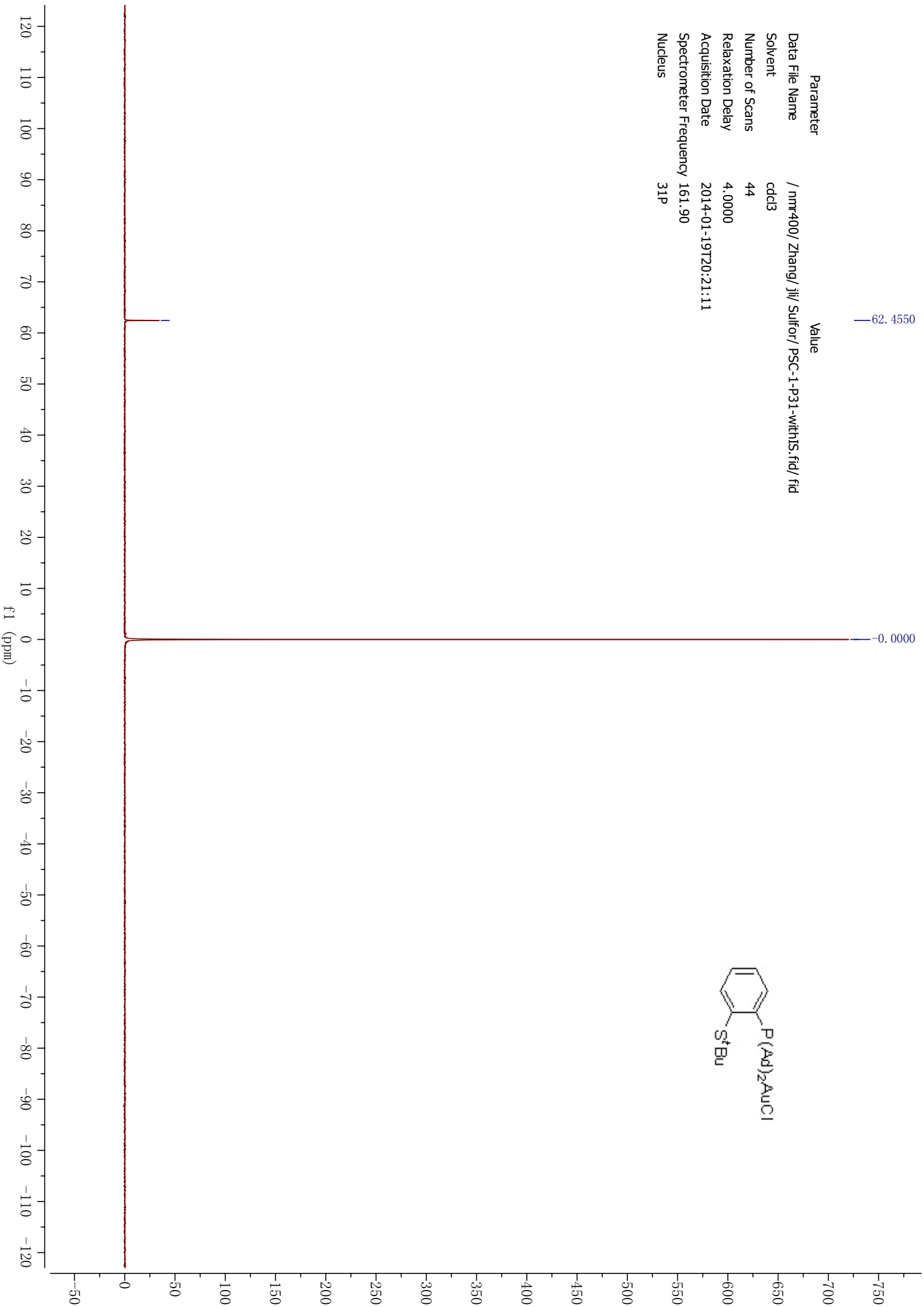
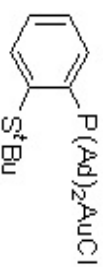
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Nucleus	31P

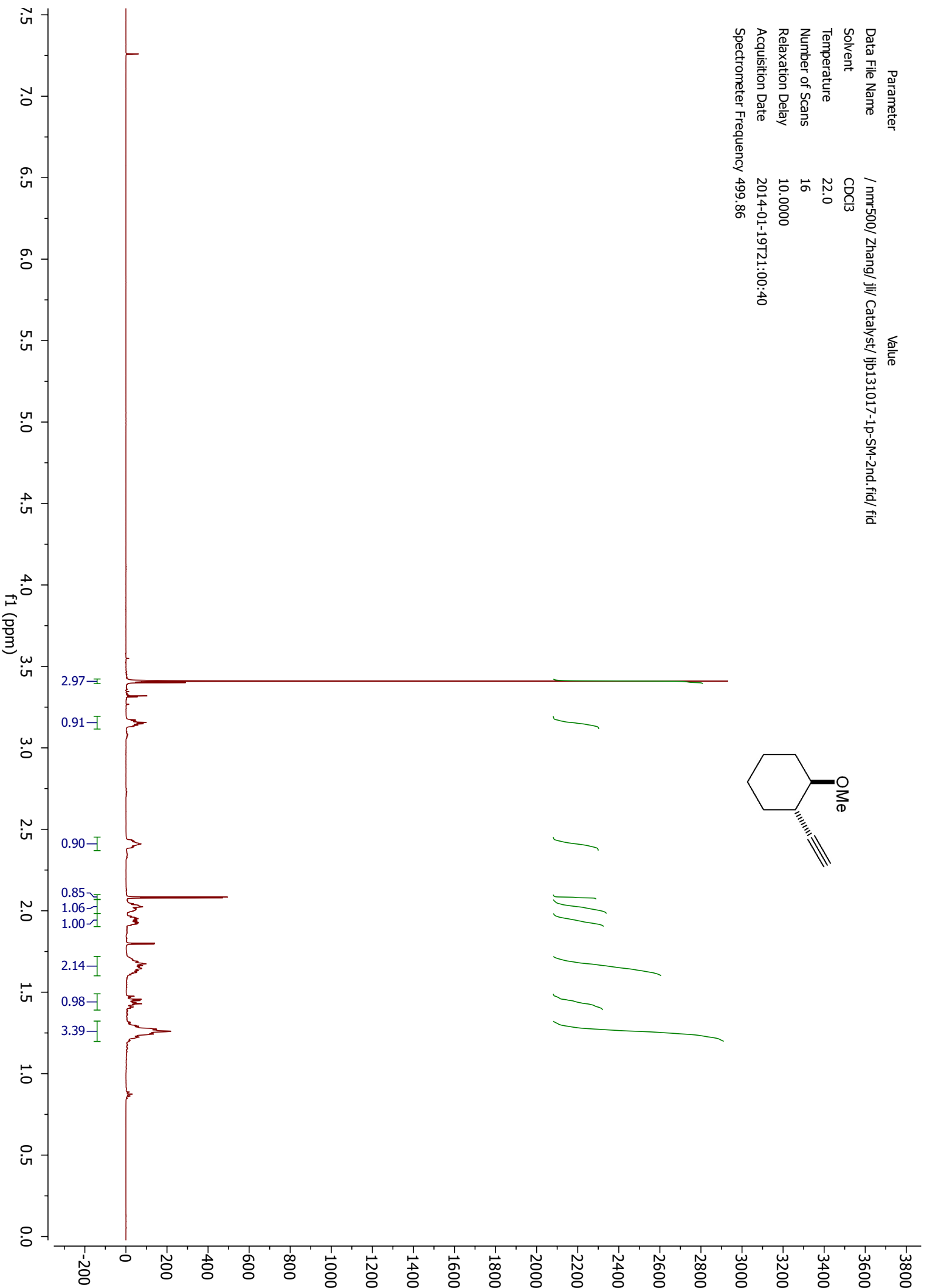
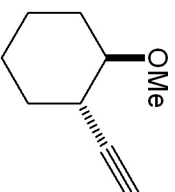
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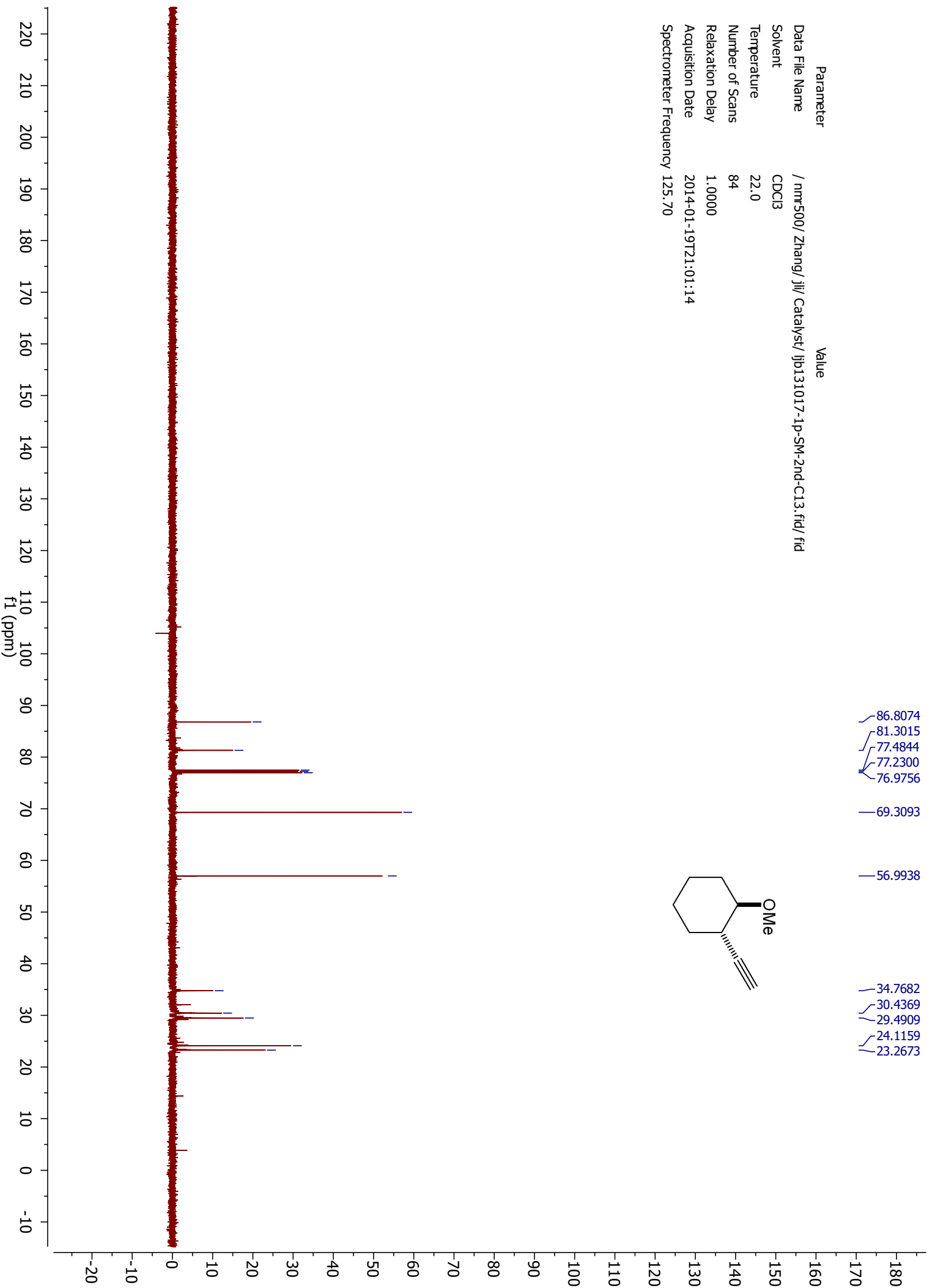
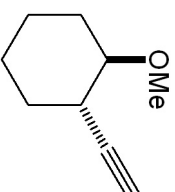


S30

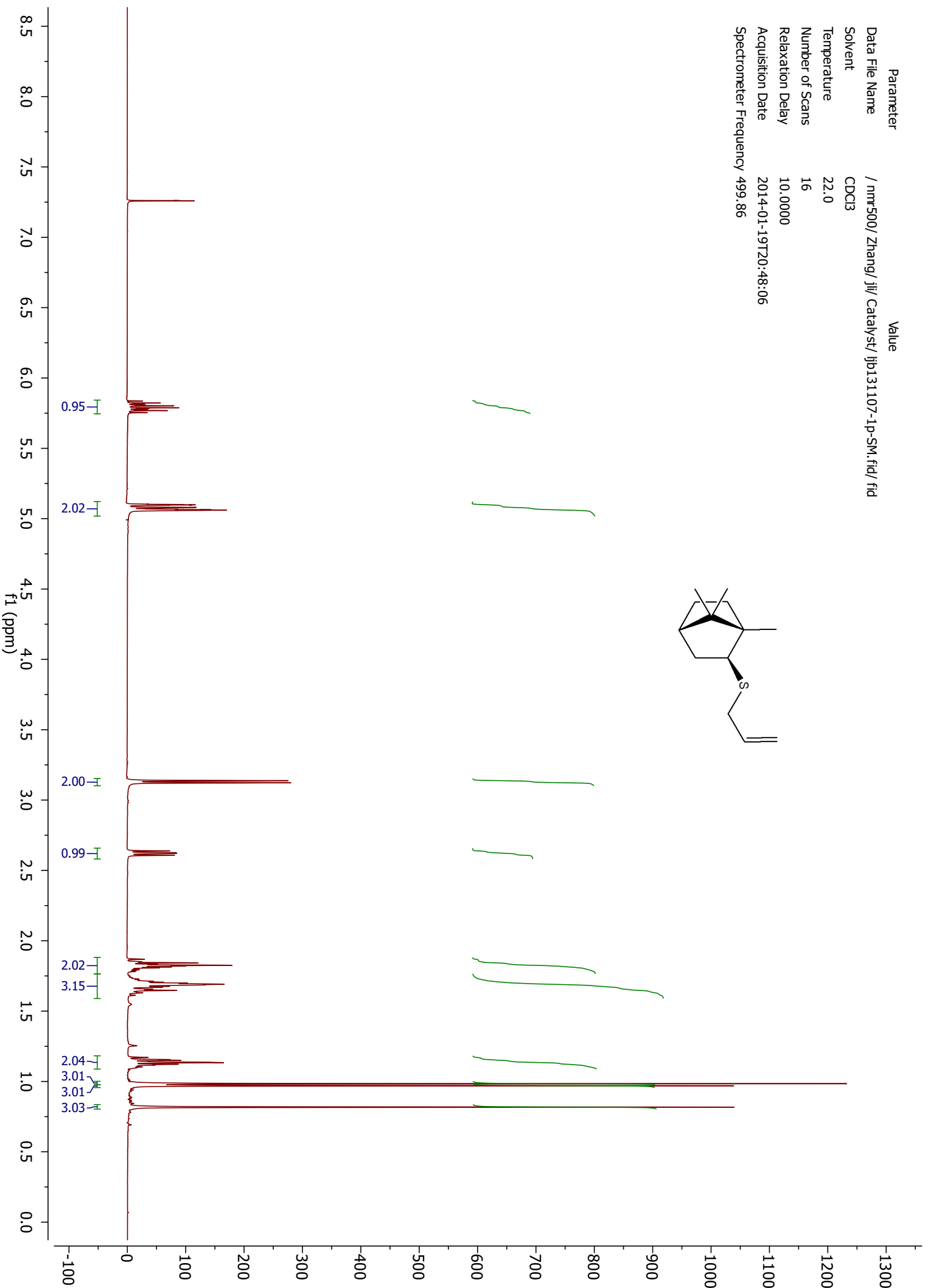
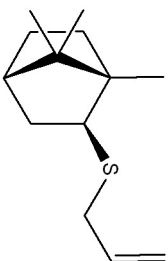
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Spectrometer Frequency	499.86



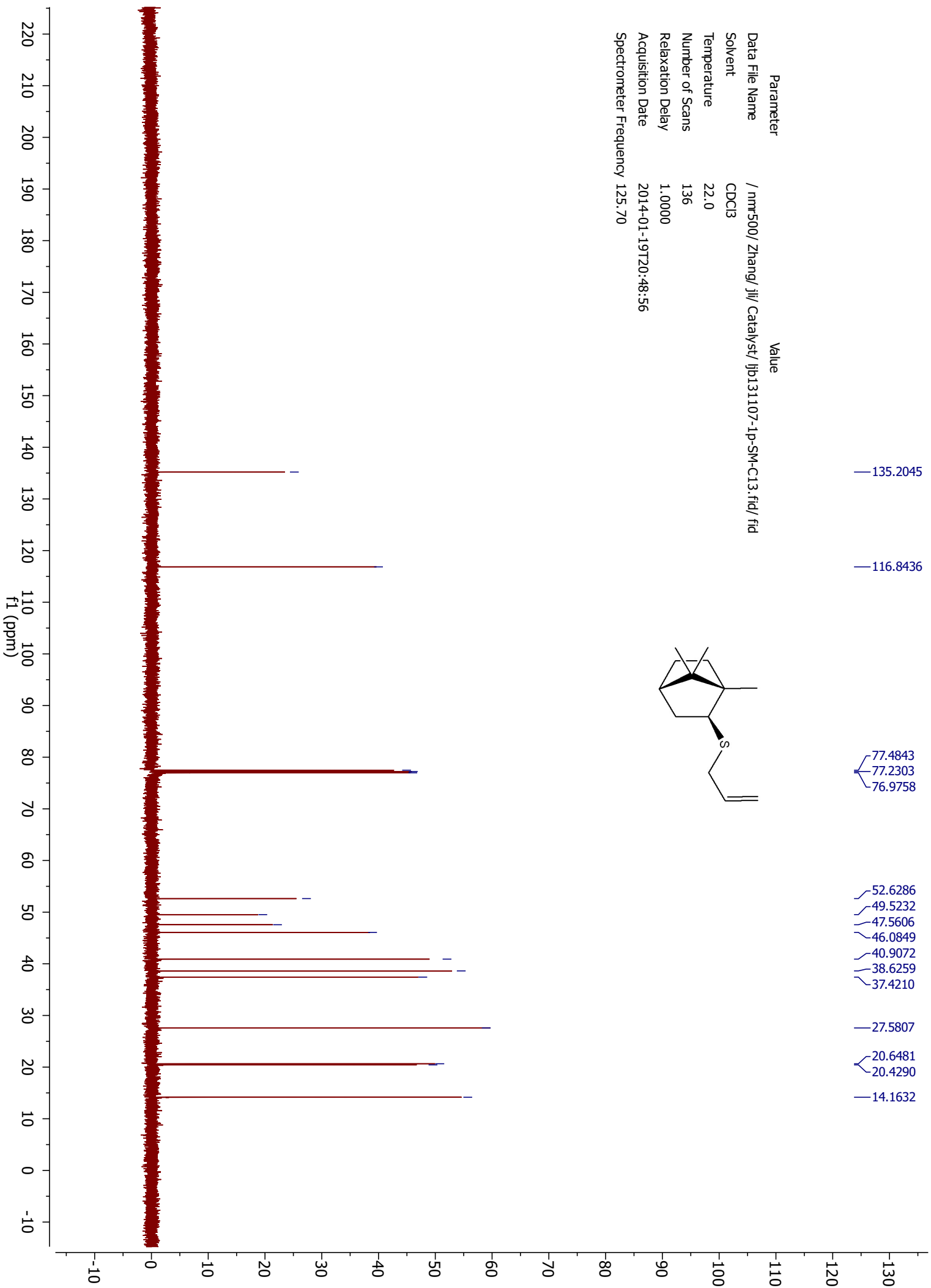
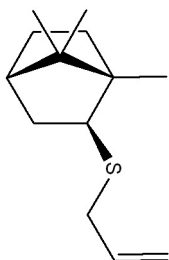
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Spectrometer Frequency	125.70



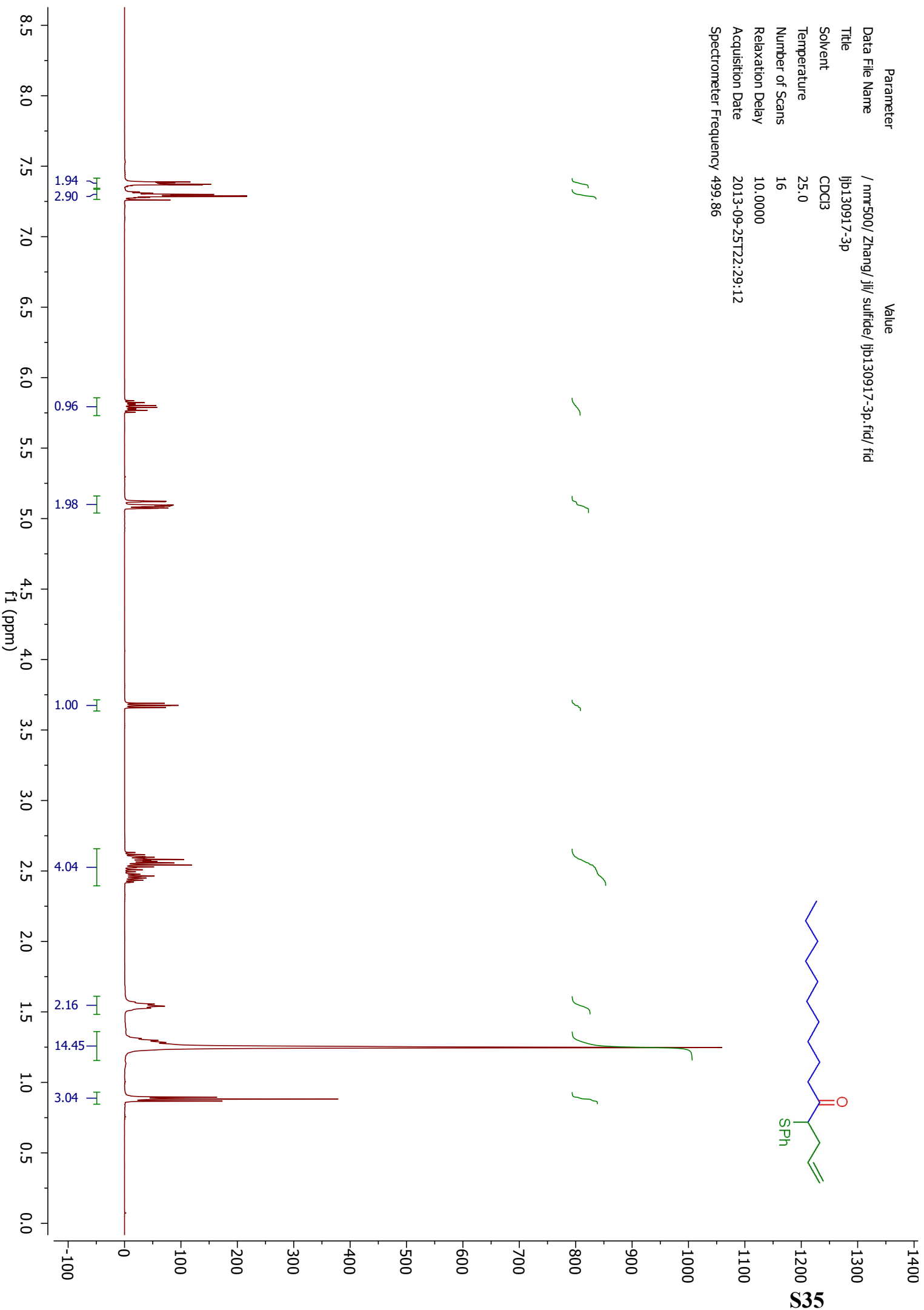
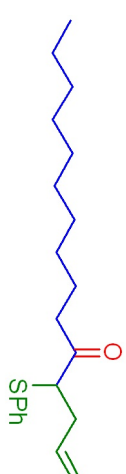
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Spectrometer Frequency	499.86



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Spectrometer Frequency	125.70



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Number of Scans	16
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Spectrometer Frequency	499.86



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128.3099

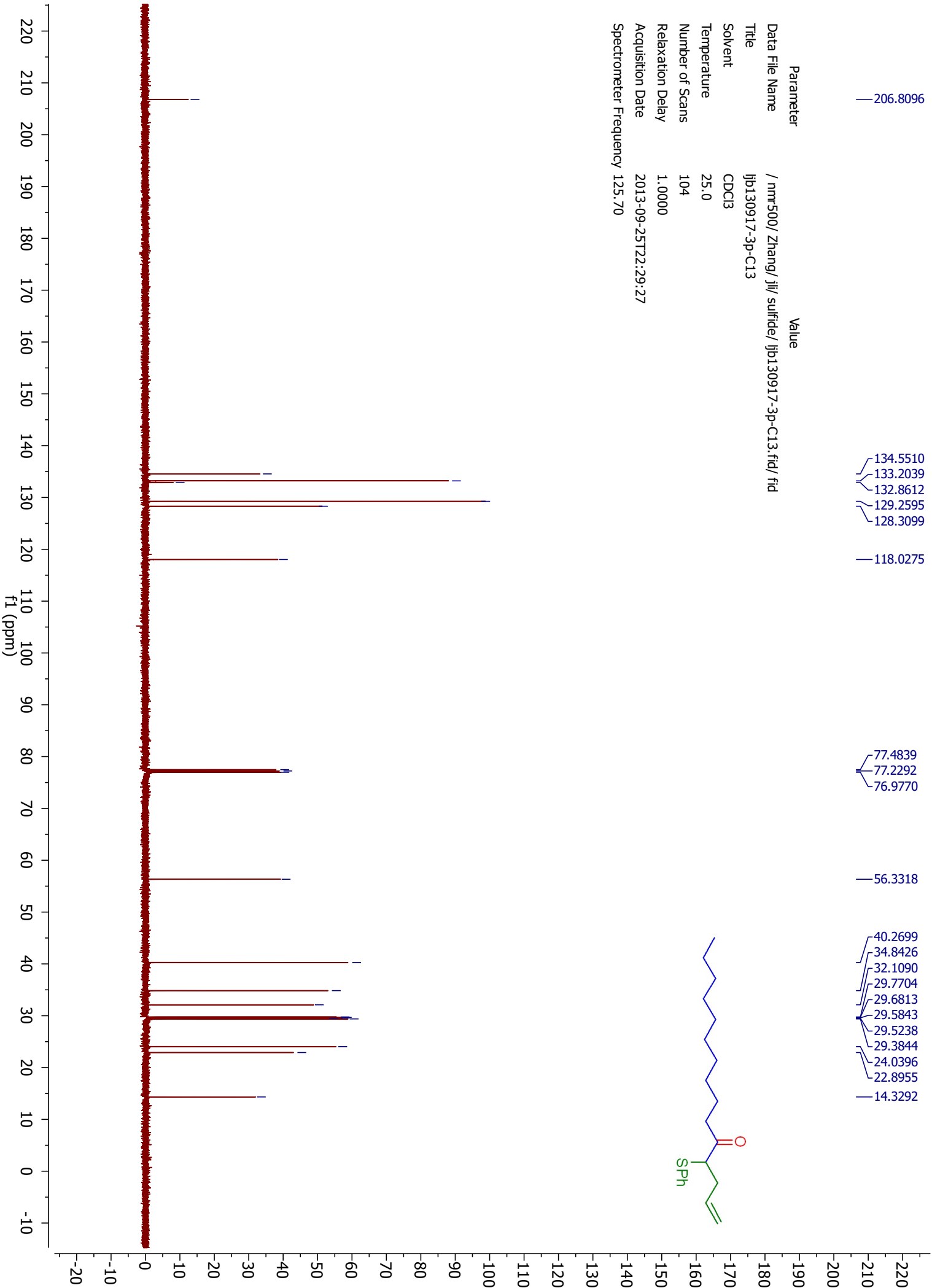
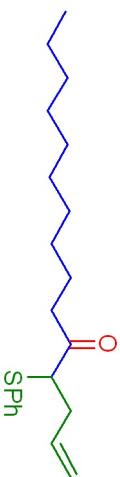
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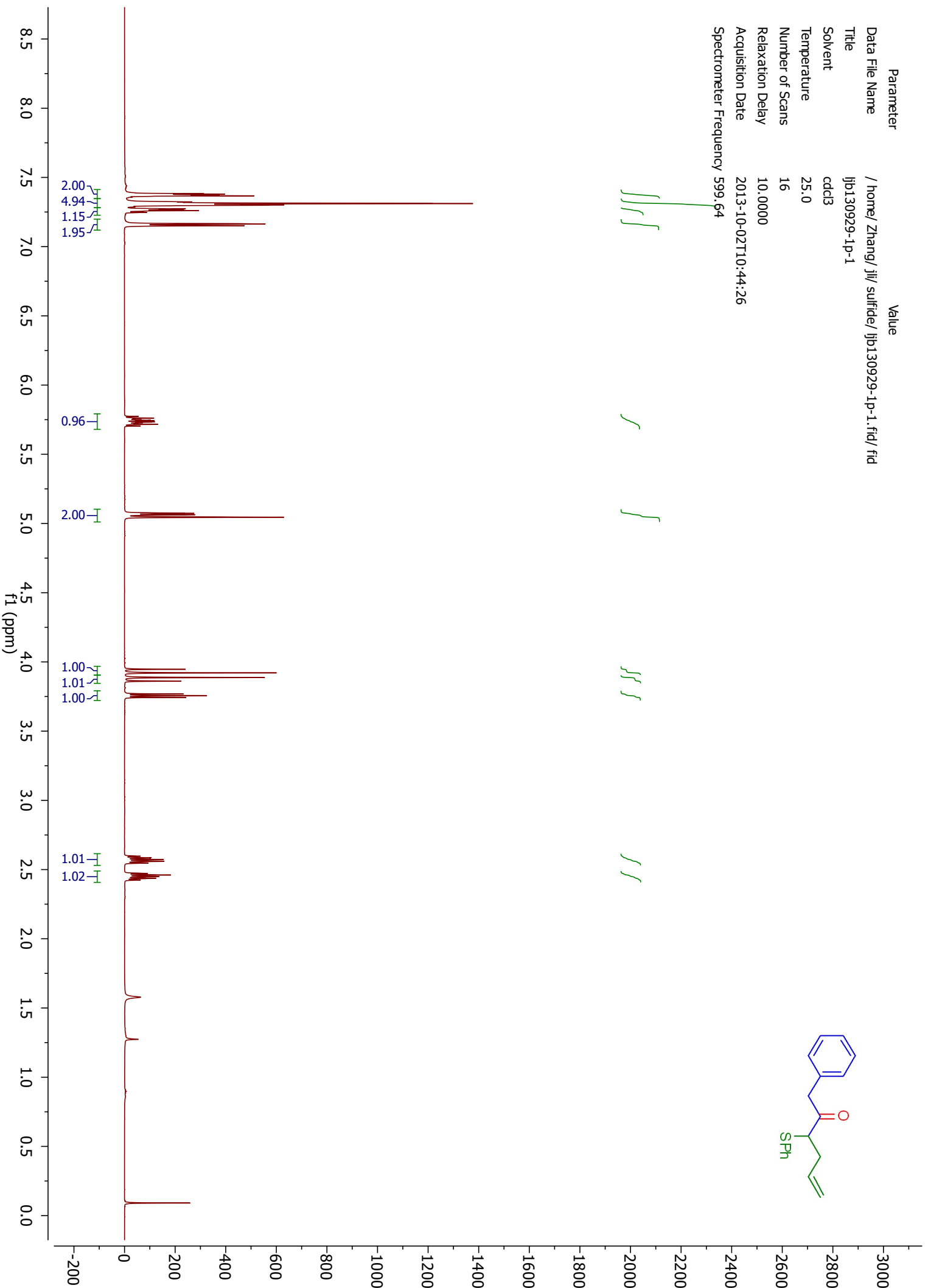
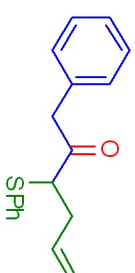
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Spectrometer Frequency	125.70



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Spectrometer Frequency	599.64



203.2717

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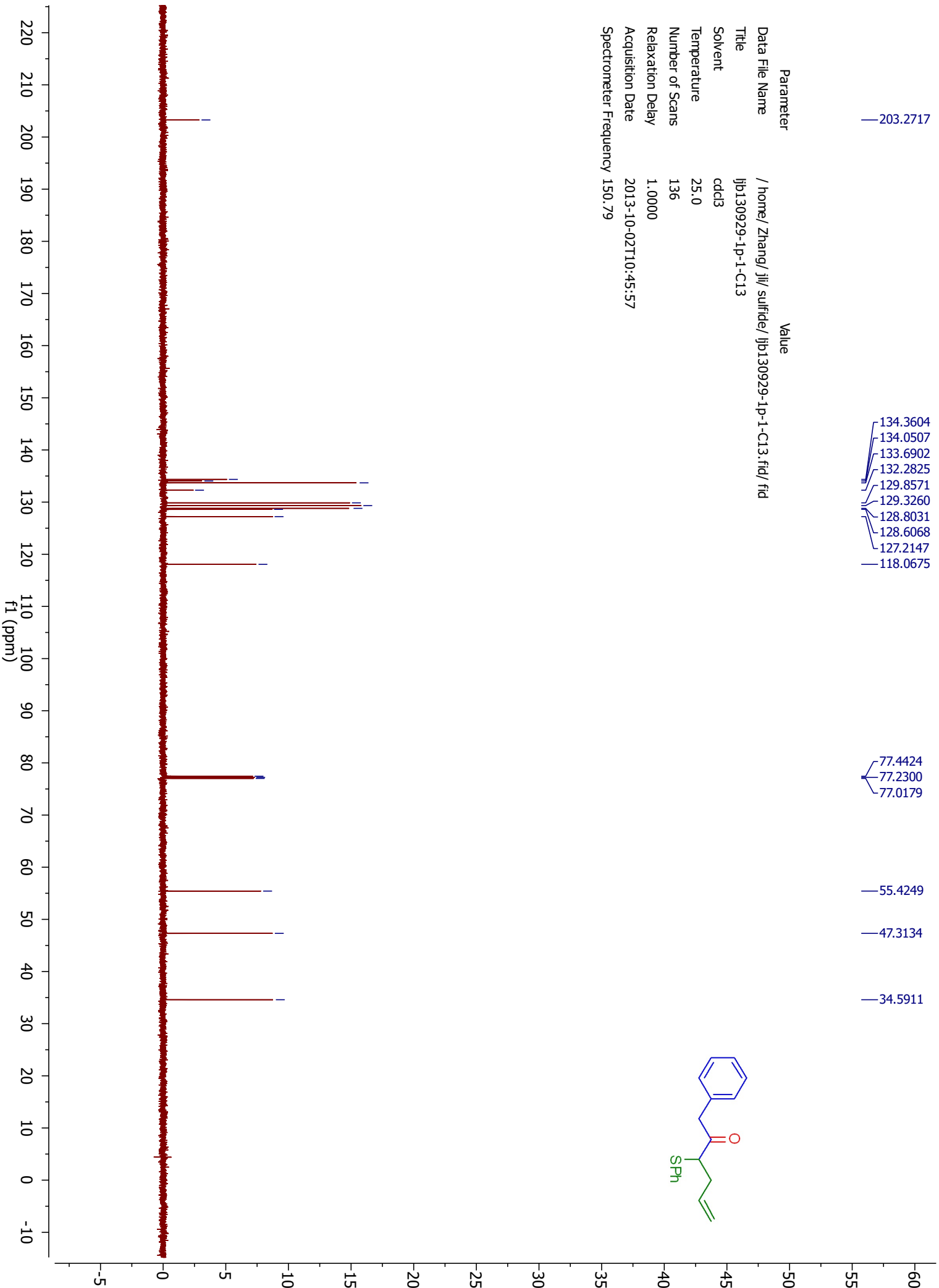
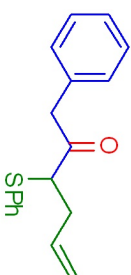
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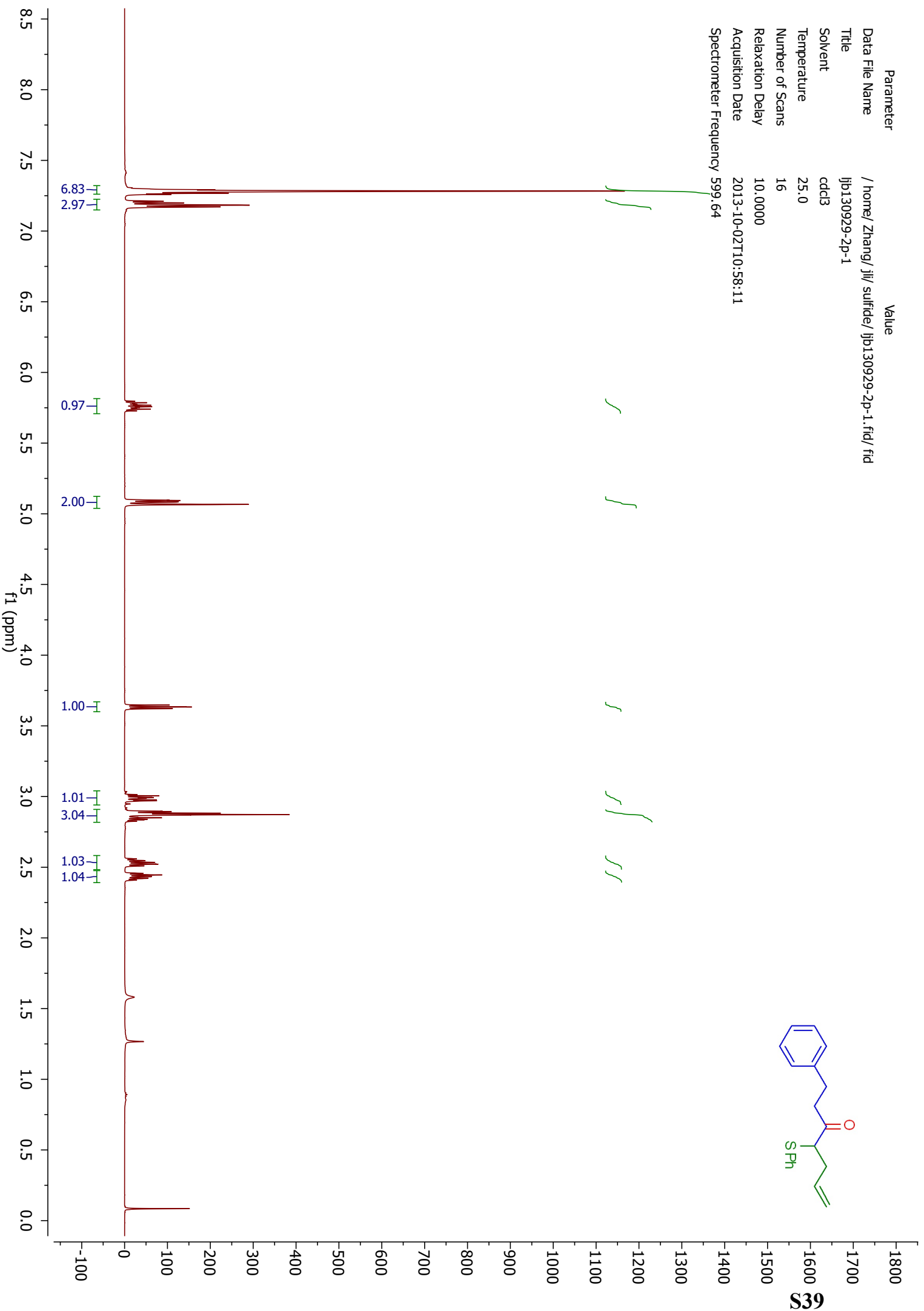
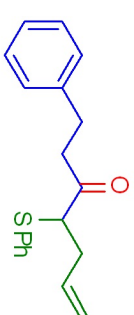
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Relaxation Delay	1.0000
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Spectrometer Frequency	150.79



Parameter	Value
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Acquisition Date	2013-10-02T10:58:11
Spectrometer Frequency	599.64



205.5567

141.1027

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128.6728

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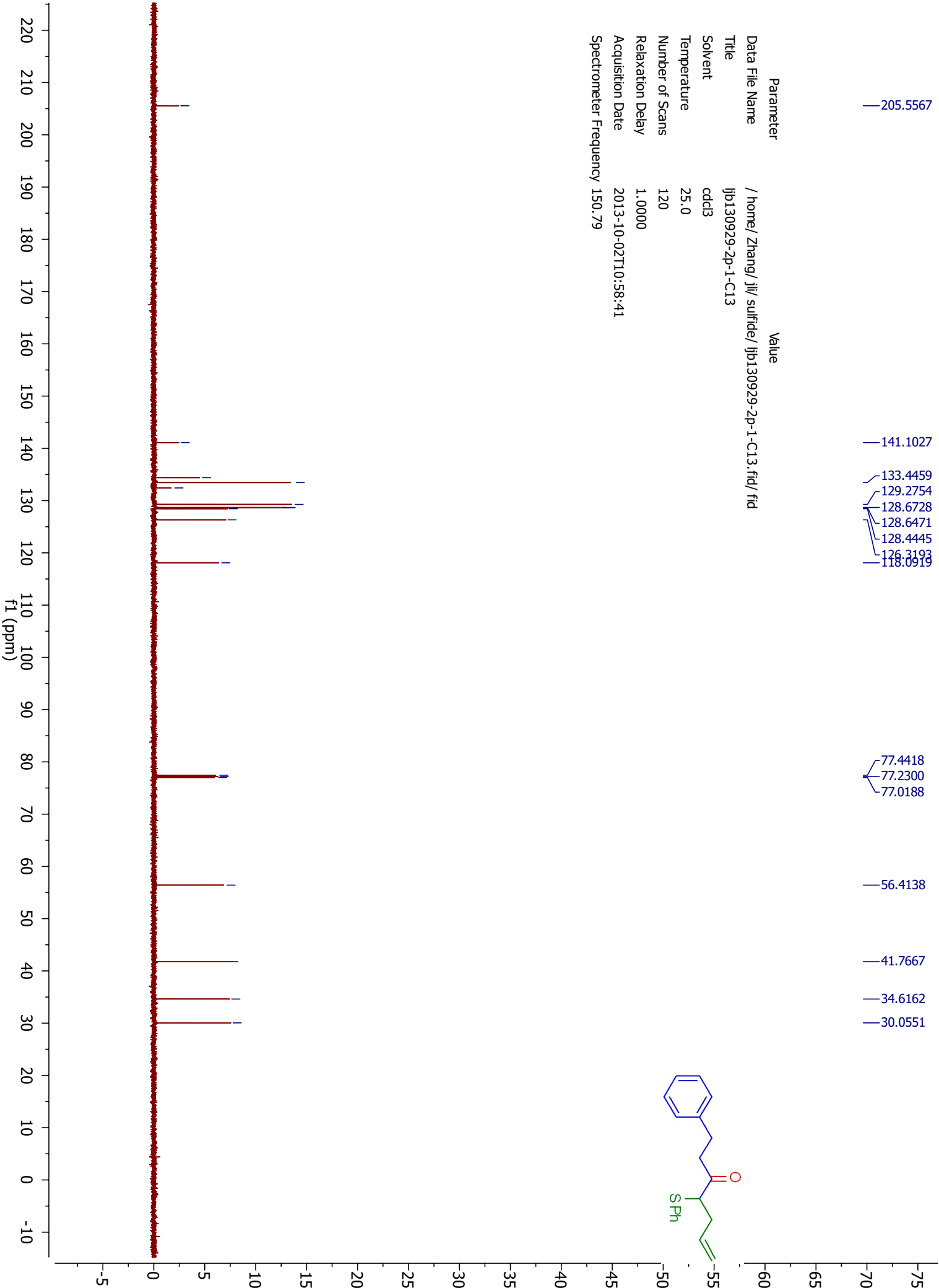
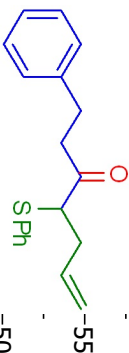
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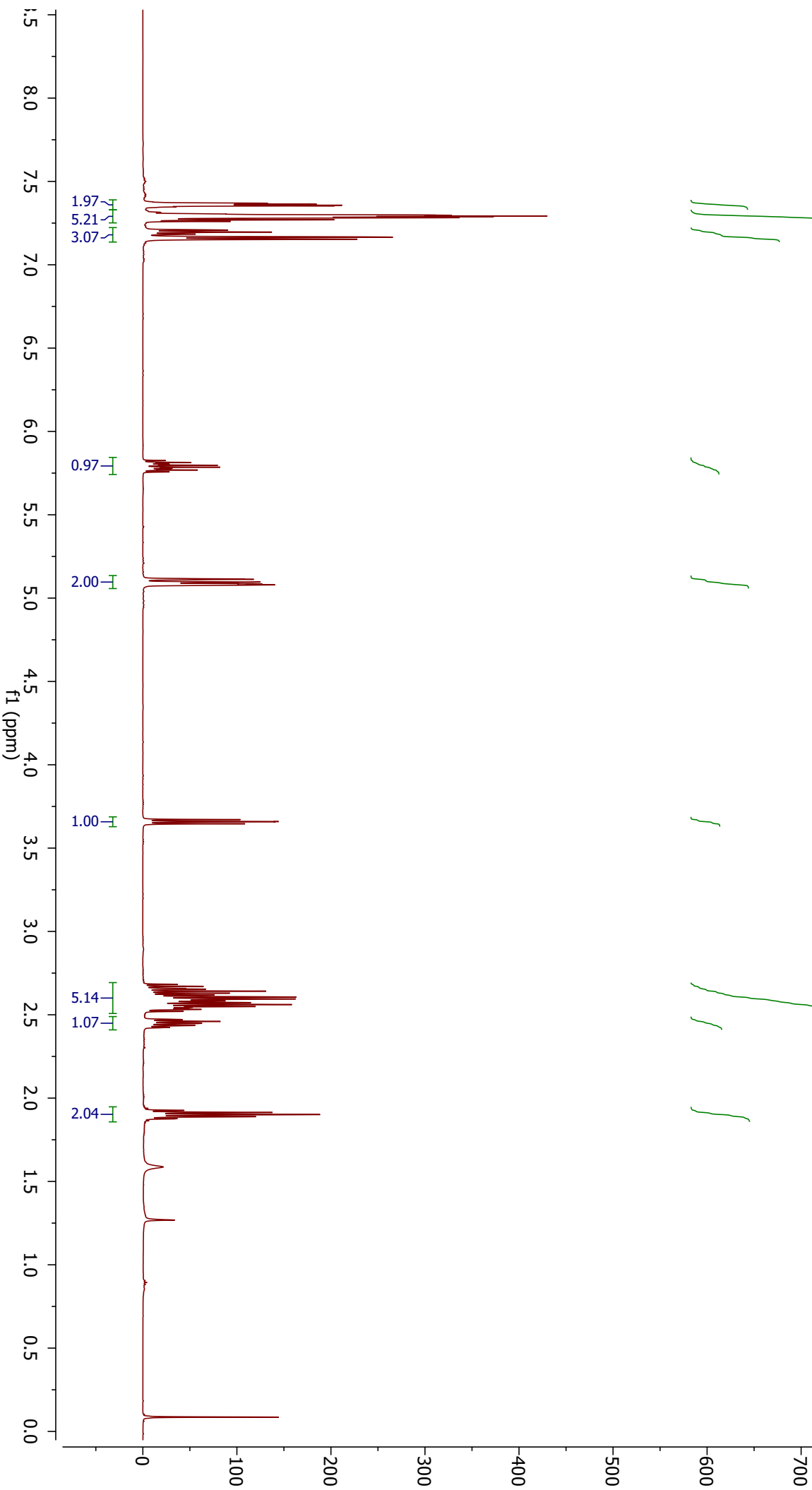
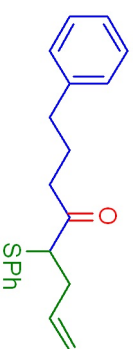
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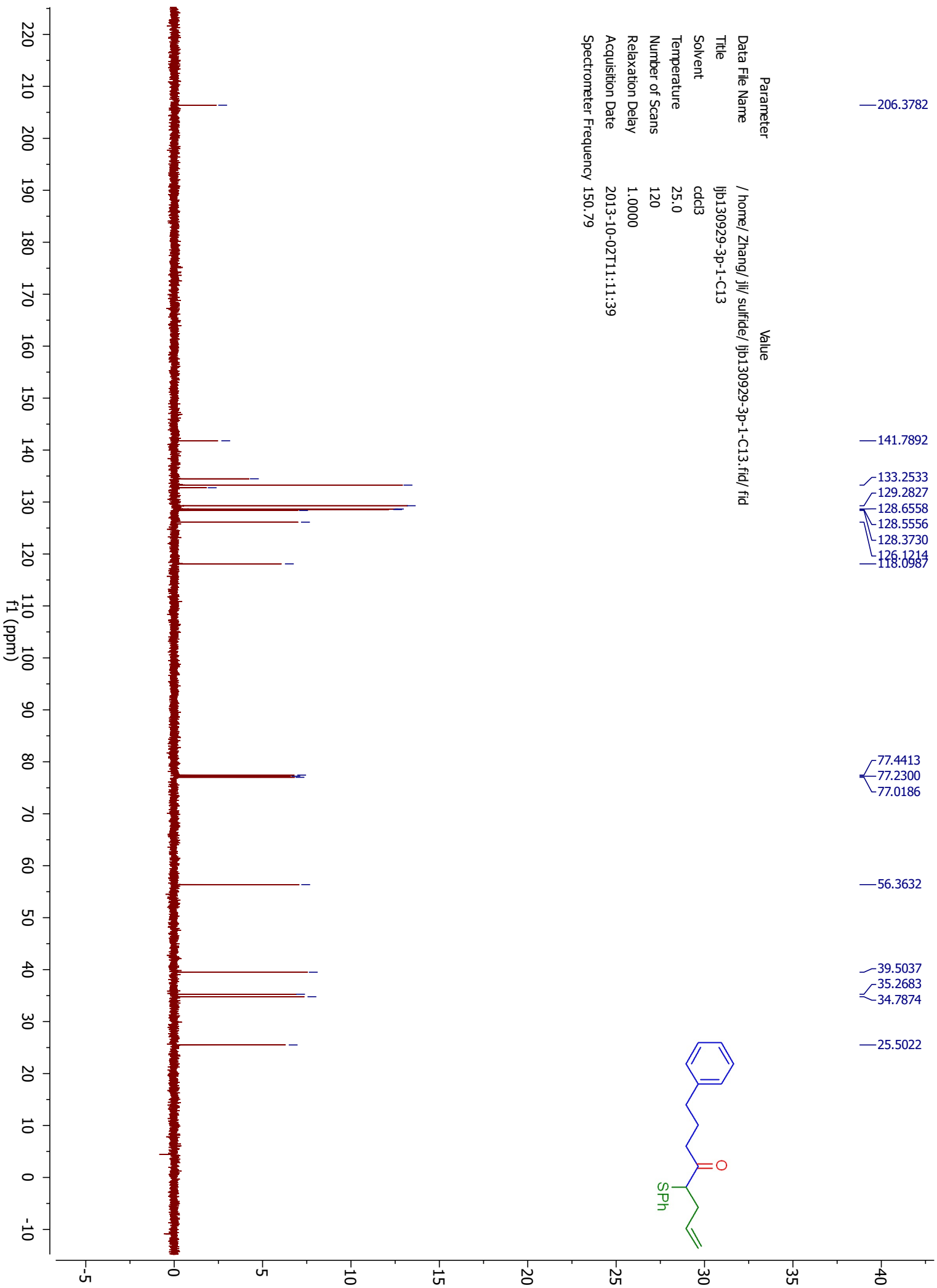
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Spectrometer Frequency	150.79



Parameter	Value
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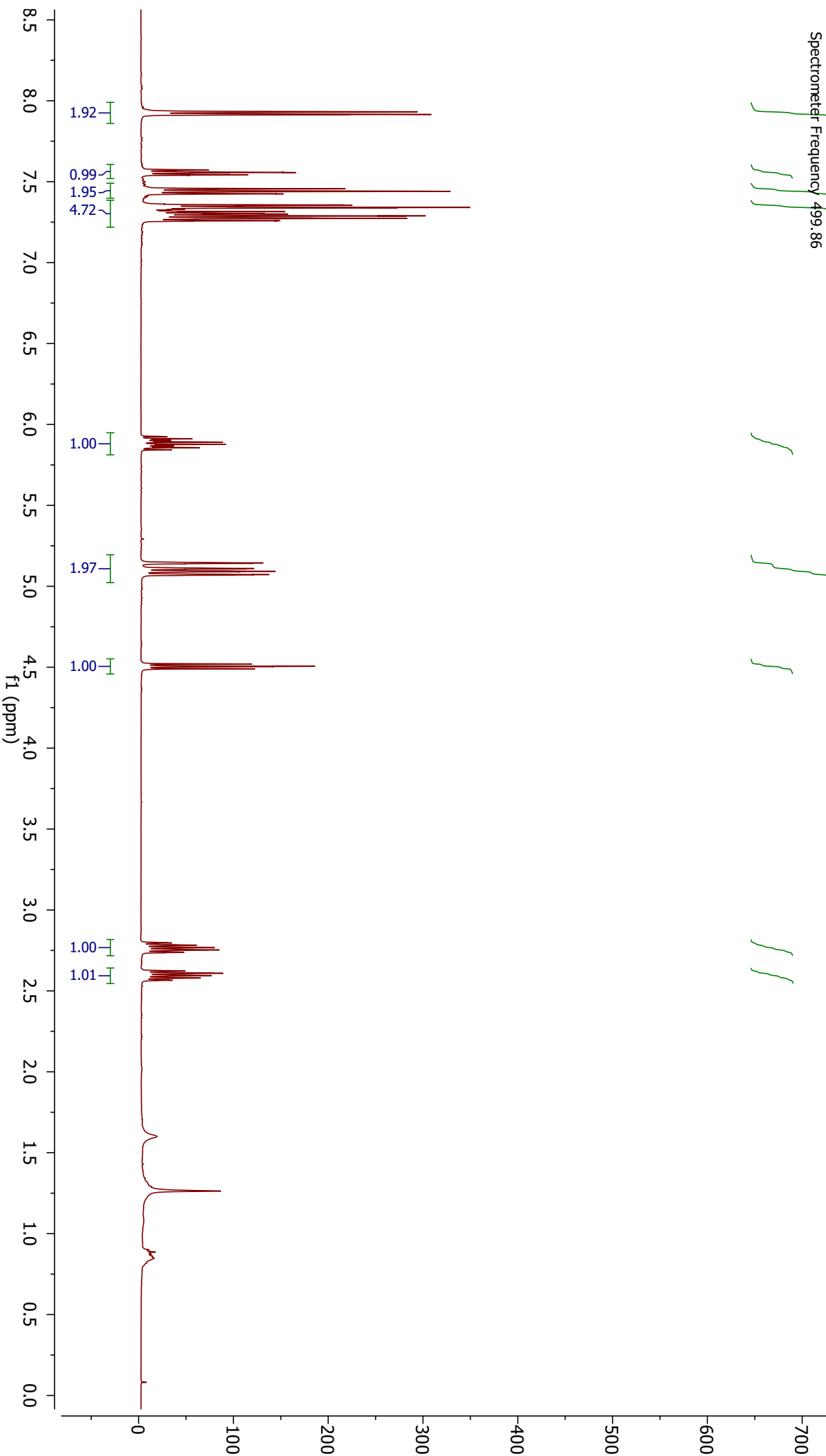
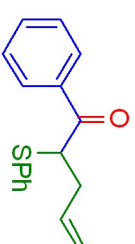
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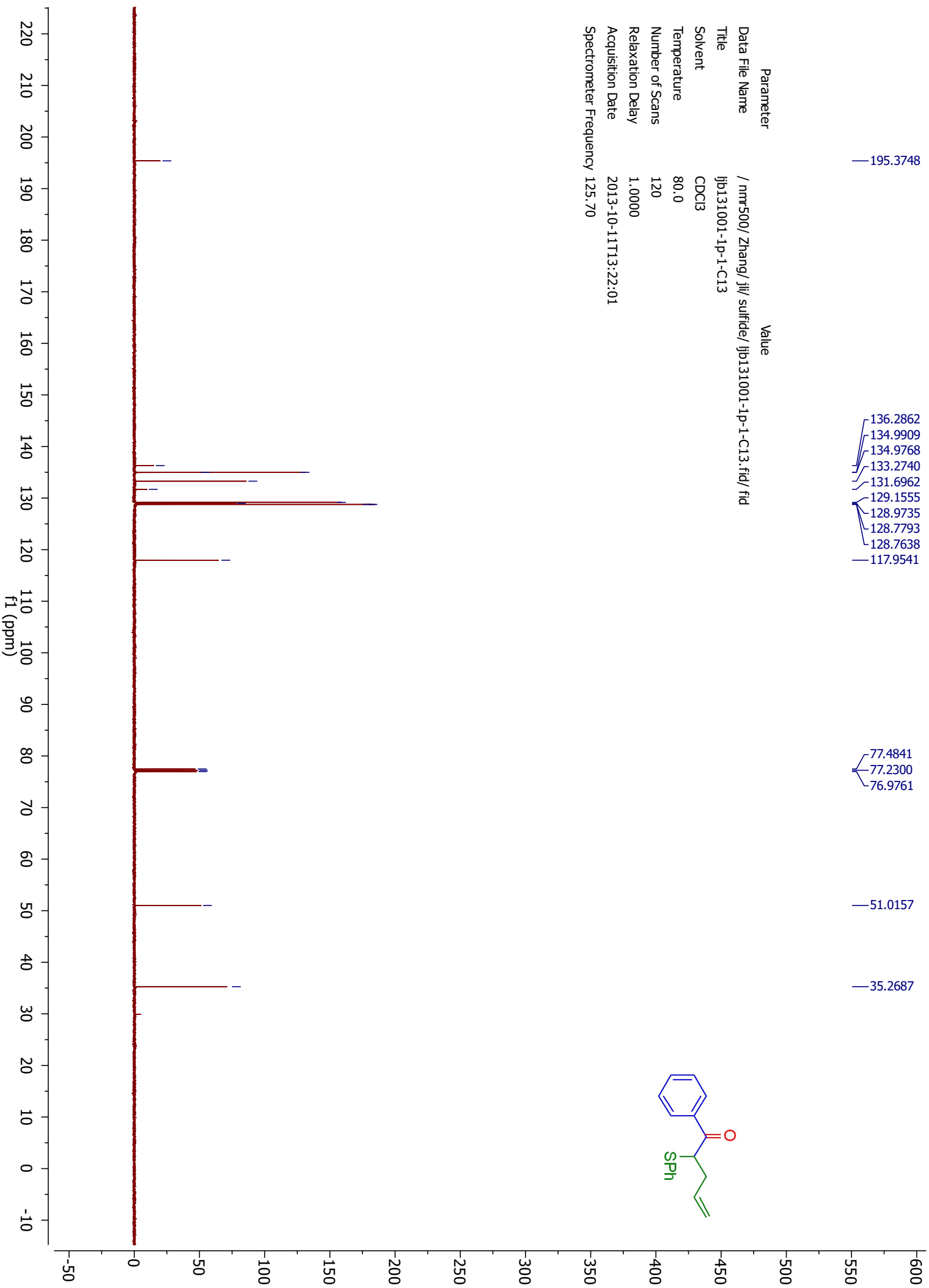
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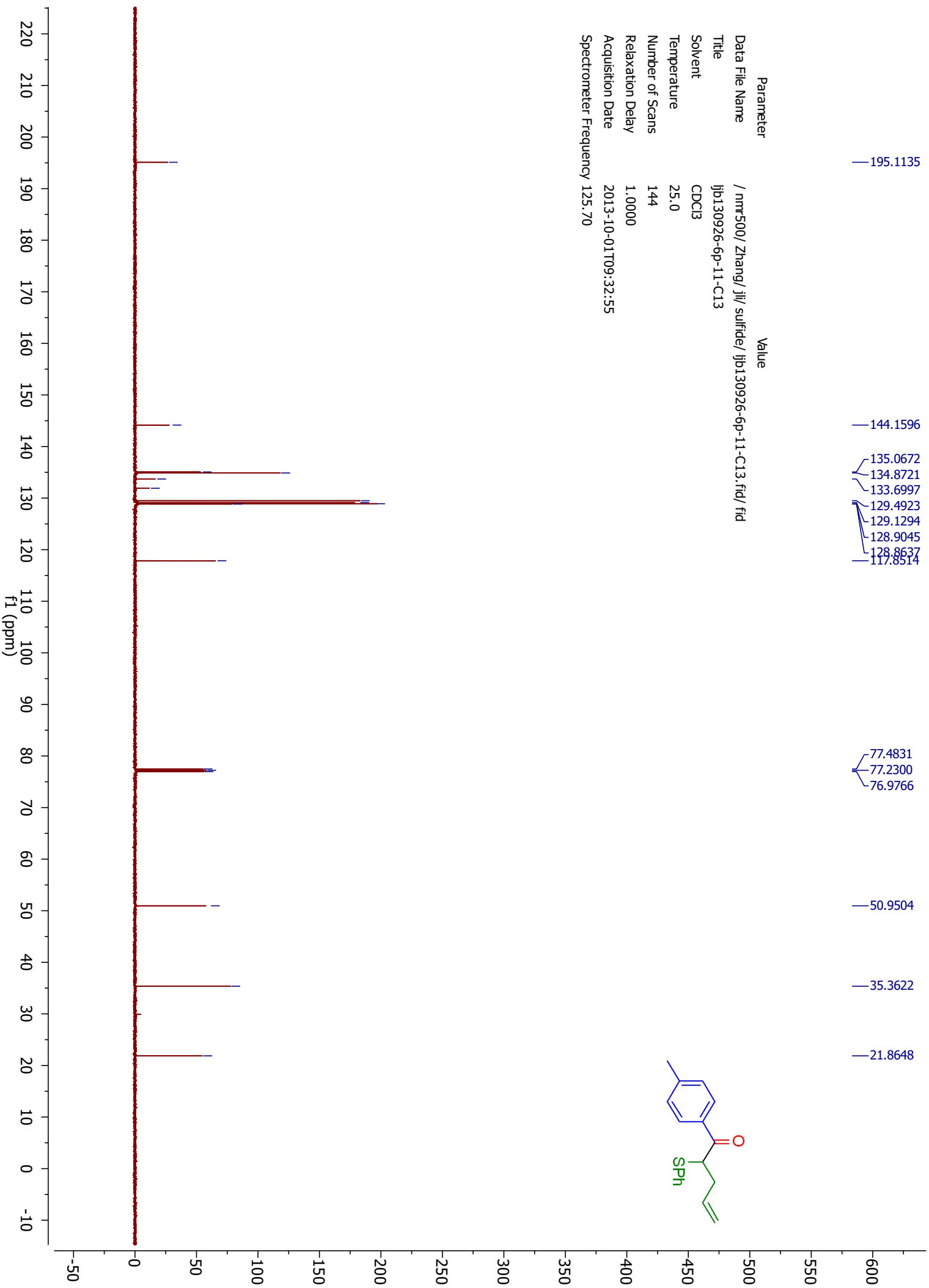
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Spectrometer Frequency	499.86



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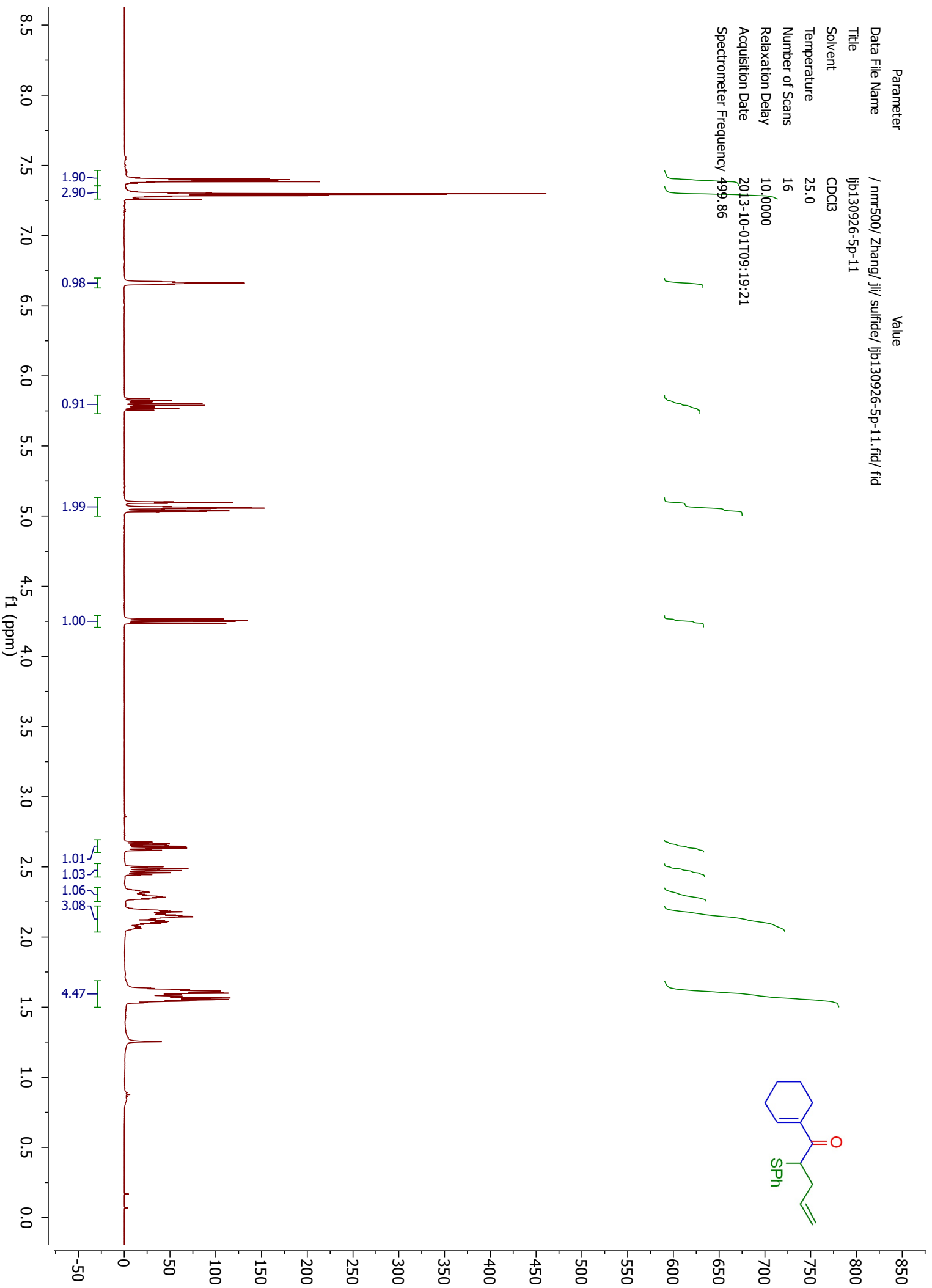
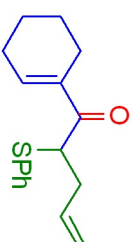
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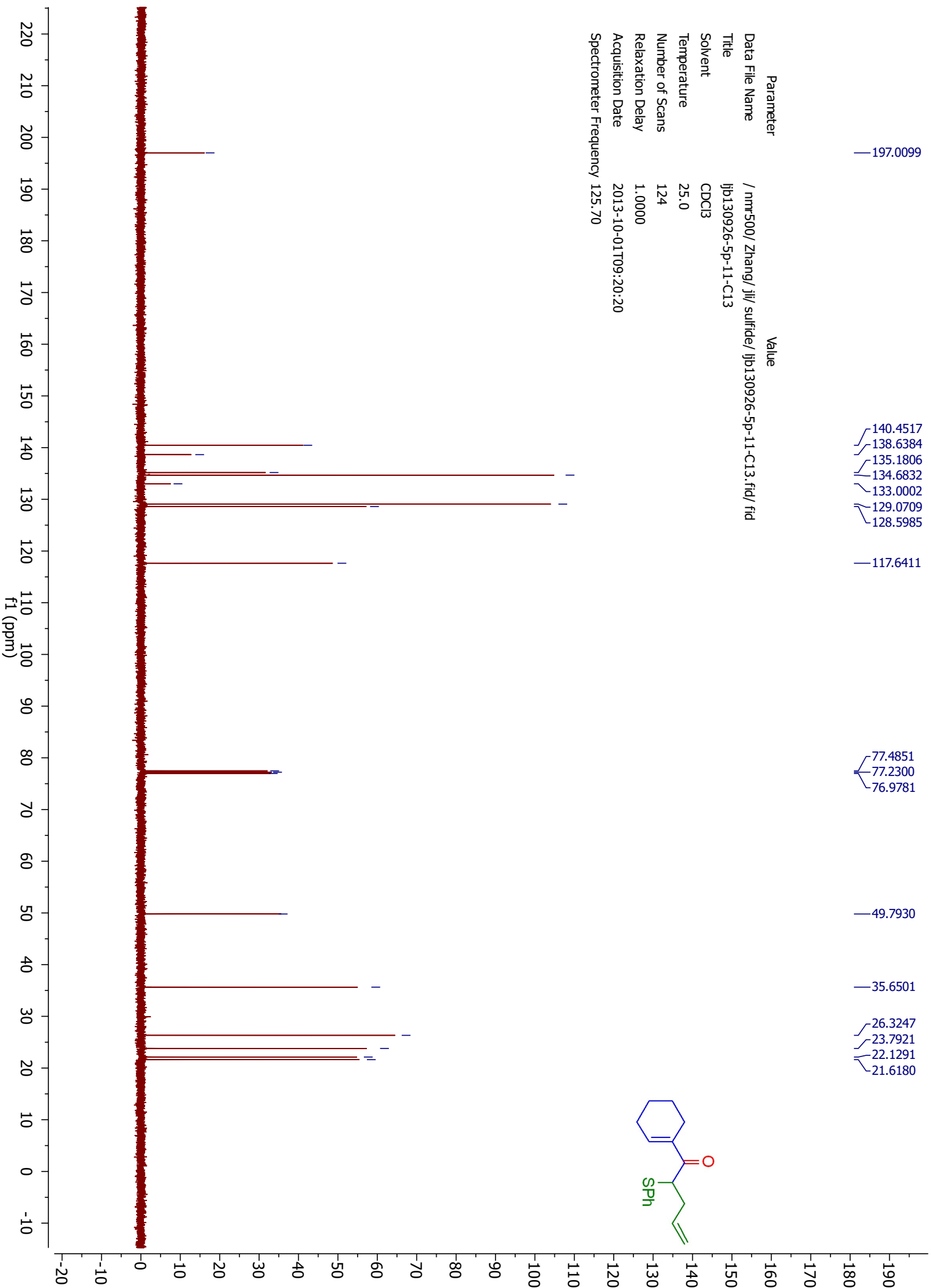
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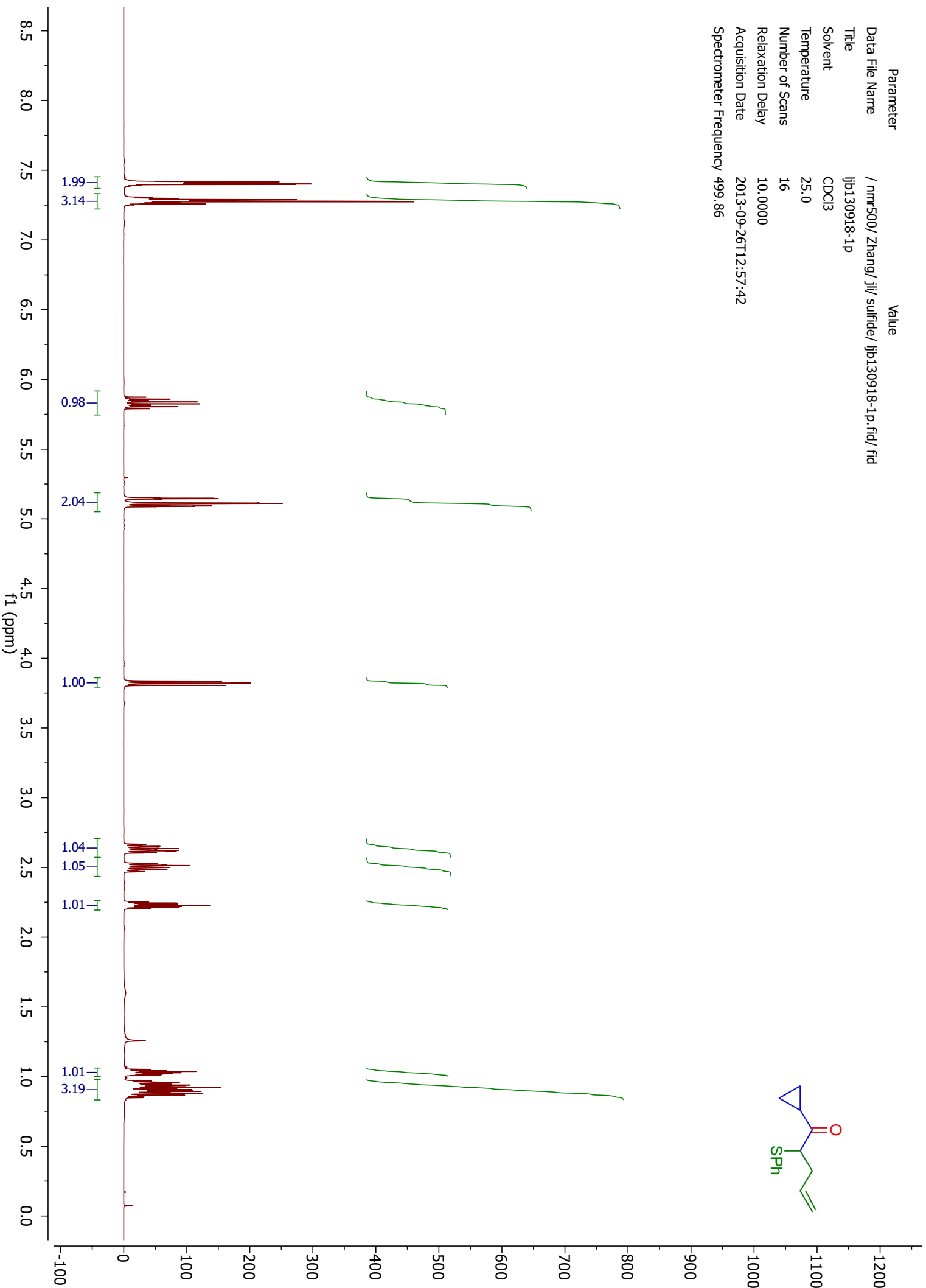
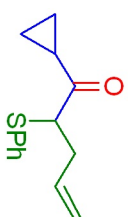
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Spectrometer Frequency	499.86



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117.9644

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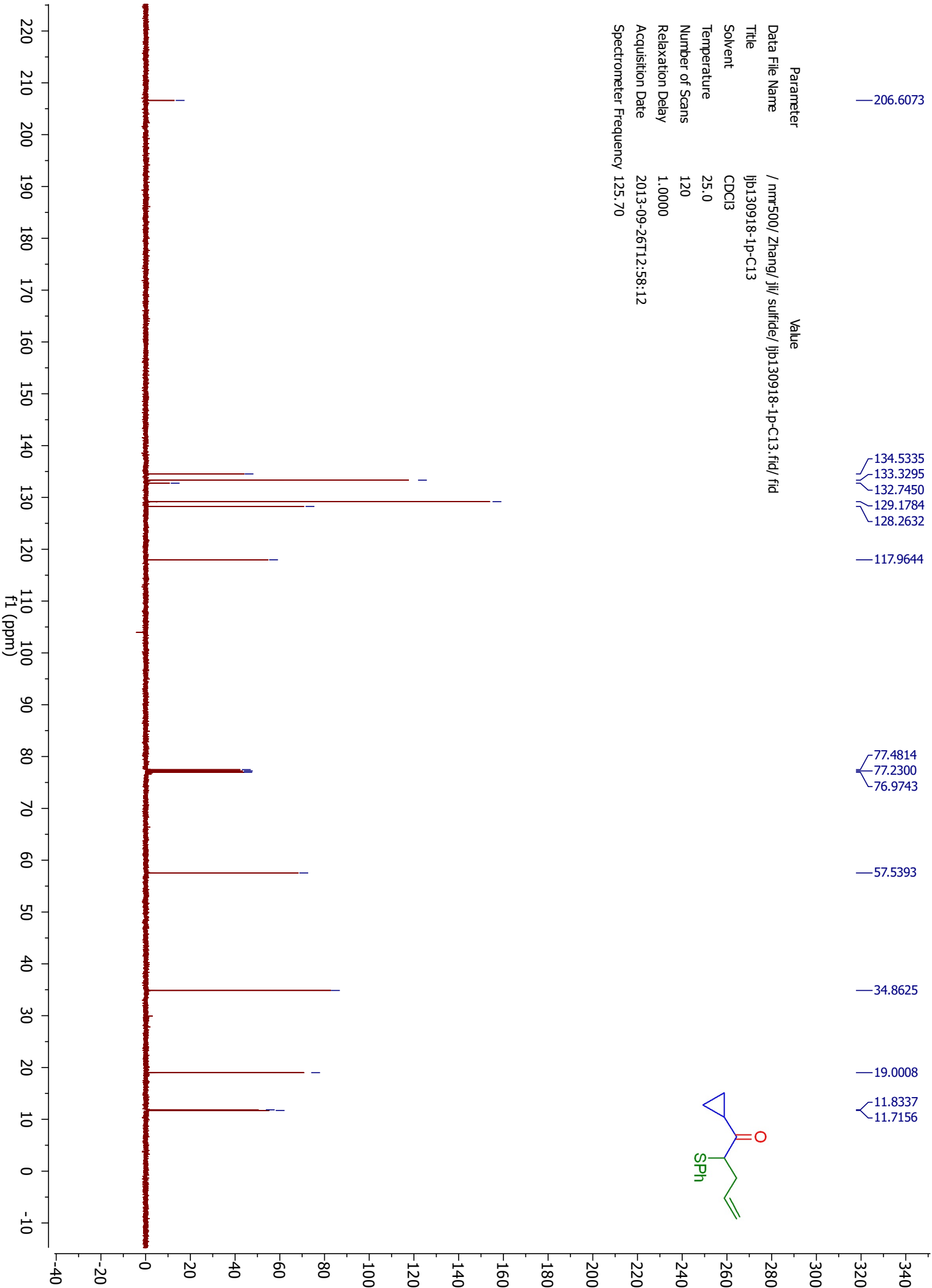
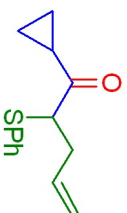
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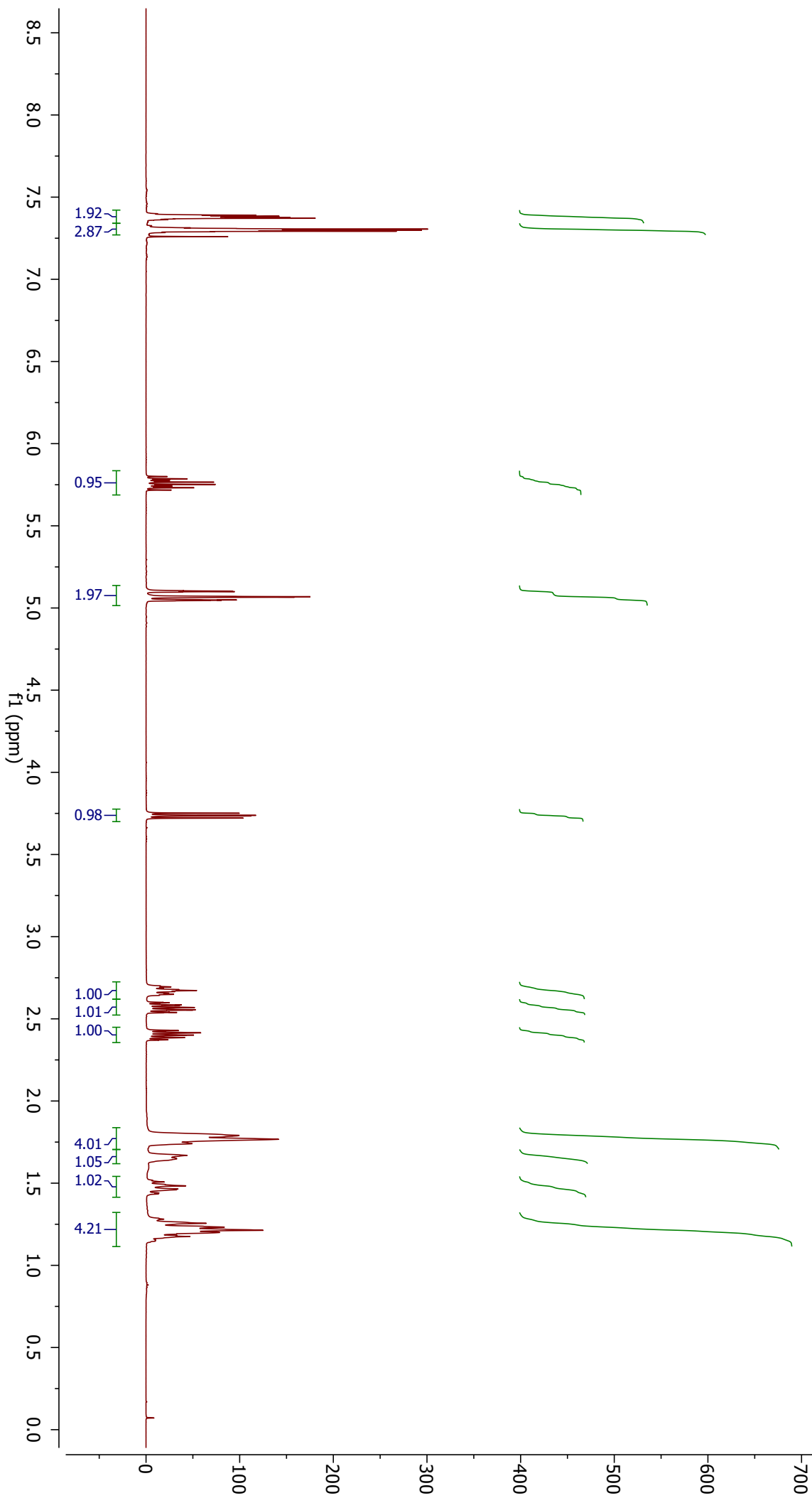
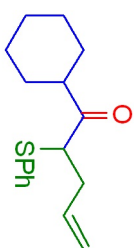
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Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr500/ Zhang/ jll/ sulfide/ jlb130918-2p.fid/ fid
Title	jlb130918-2p
Solvent	CDCl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-09-25T22:18:53
Spectrometer Frequency	499.86



208.5741

134.8397
133.9092
132.3868
129.1879
128.5067

117.9245

77.4836
77.2298
76.9752

54.6456

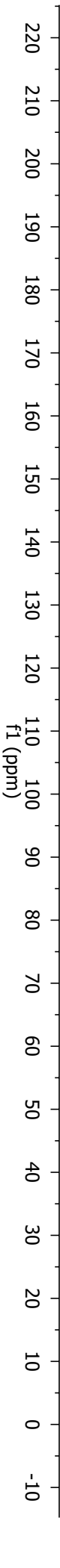
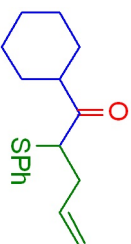
48.8868

34.7981
29.6619
28.5145
26.1361
25.9656
25.5521

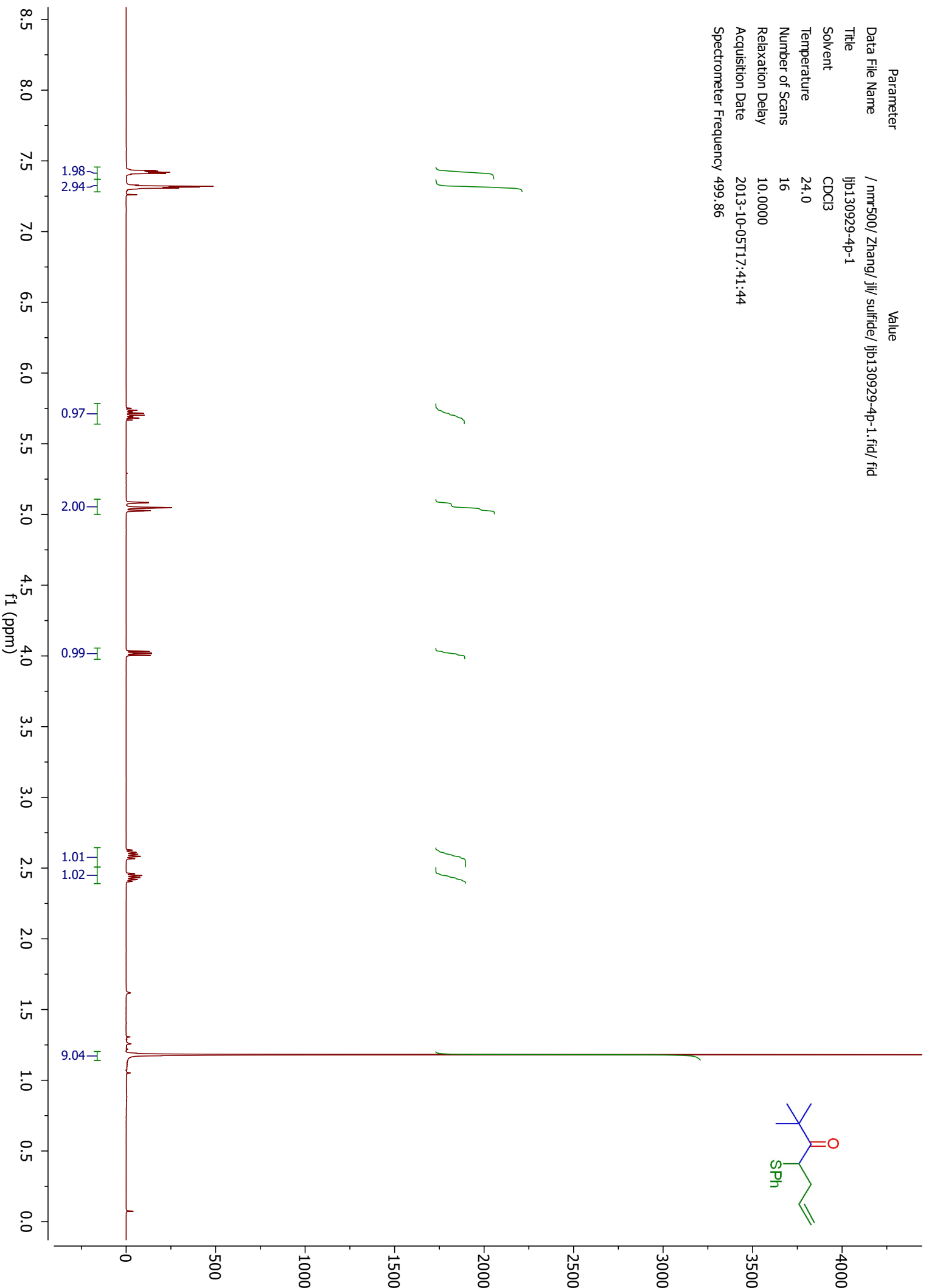
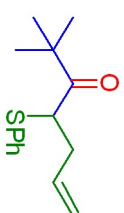
Parameter

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Data File Name	/nmr500/Zhang/jiv/sulfide/jib130918-2p-C13.fid/ fid
Title	jib130918-2p-C13
Solvent	CDCl3
Temperature	25.0
Number of Scans	88
Relaxation Delay	1.0000
Acquisition Date	2013-09-25T22:19:43
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr500/Zhang/jll/sulfide/jlb130929-4p-1.fid/ fid
Title	jlb130929-4p-1
Solvent	CDCl3
Temperature	24.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-05T17:41:44
Spectrometer Frequency	499.86



210.4589

Parameter

Value

Data File Name	/nmr500/Zhang/jl/sulfide/jlb130929-4p-1-C13.fid/fid
Title	jlb130929-4p-1-C13
Solvent	CDCl ₃
Temperature	24.0
Number of Scans	120
Relaxation Delay	1.0000
Acquisition Date	2013-10-05T17:43:44
Spectrometer Frequency	125.70

134.9453
134.4115
132.5945
129.1180
128.6233

118.1161

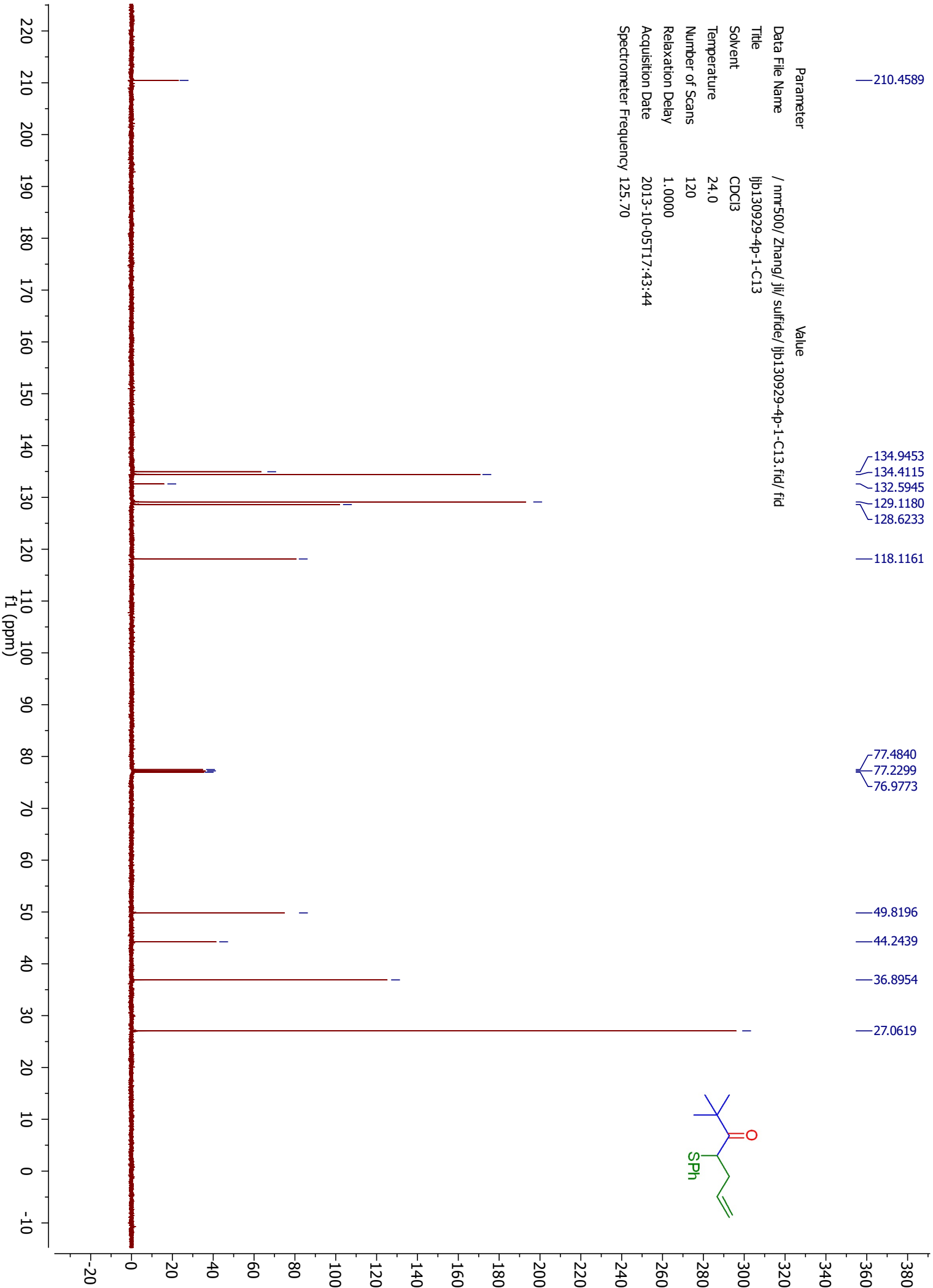
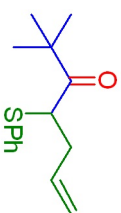
77.4840
77.2299
76.9773

49.8196

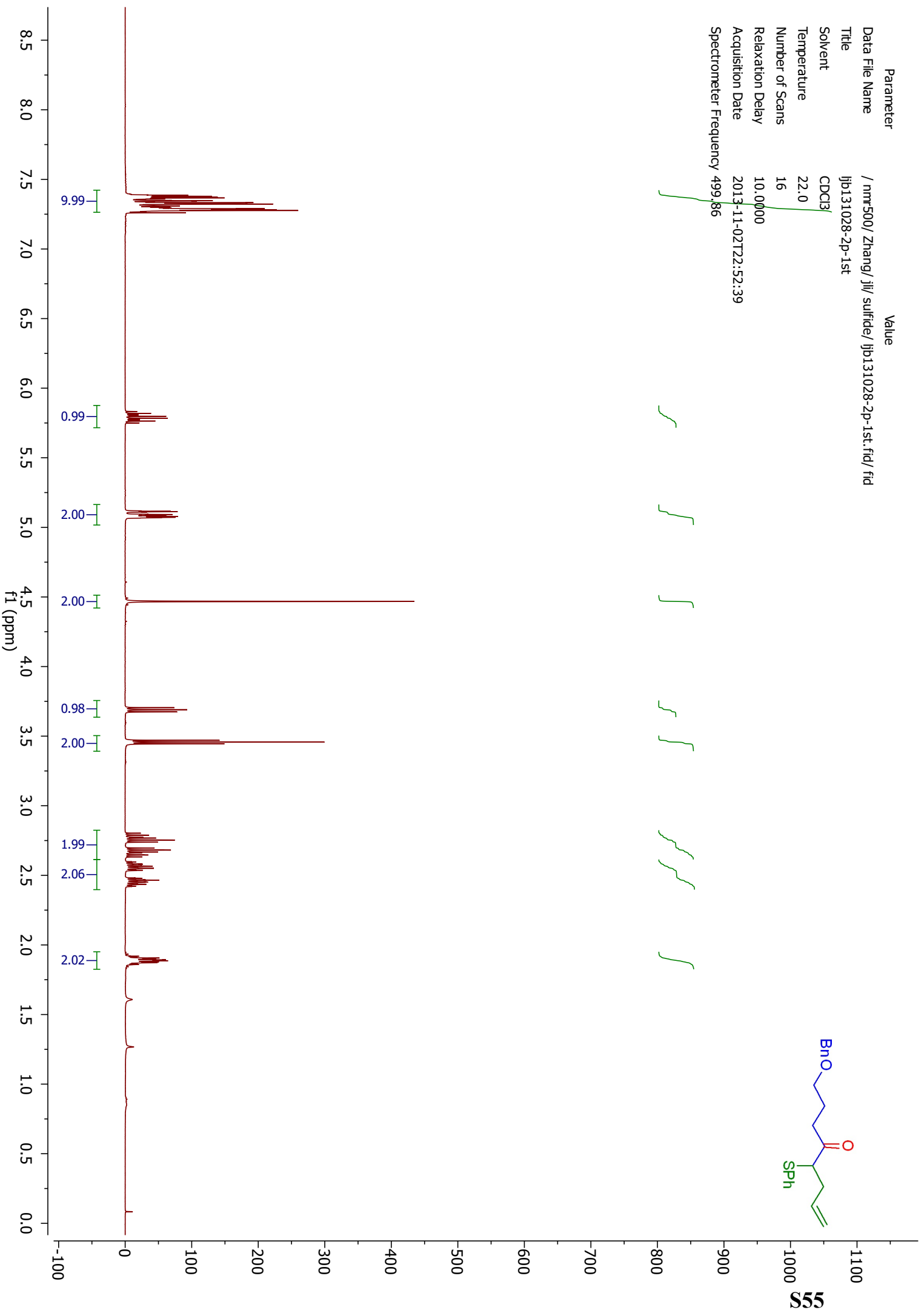
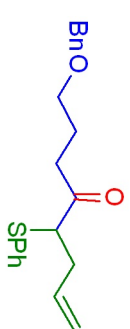
44.2439

36.8954

27.0619



Parameter	Value
Data File Name	/nmr500/Zhang/jli/sulfide/jlb131028-2p-1st.fid/fid
Title	jlb131028-2p-1st
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-02T22:52:39
Spectrometer Frequency	499.86



206.3640

138.6006
134.5316
133.2186
132.7830
129.2752
128.5609
128.3297
127.8253
127.7580
118.0470

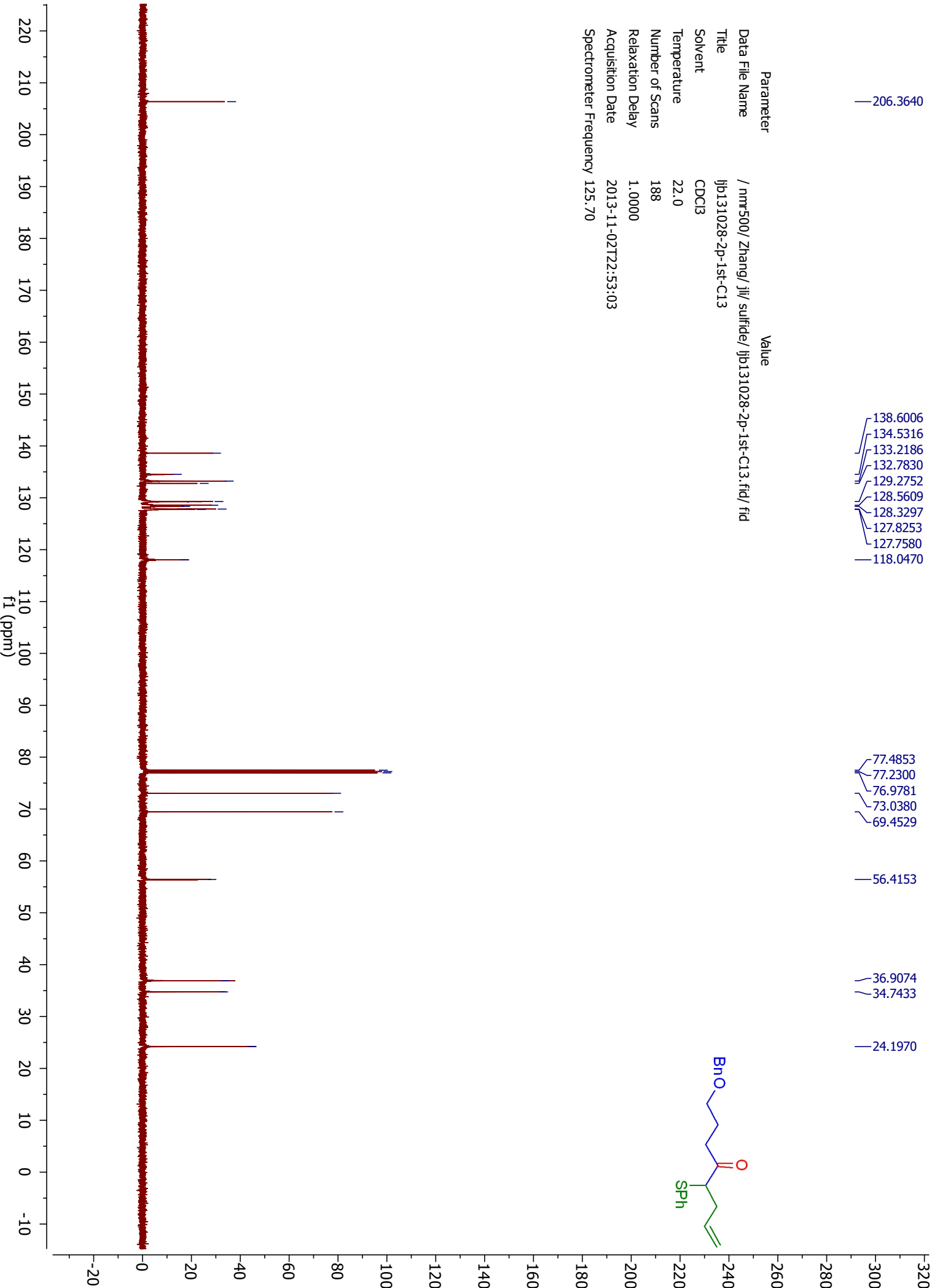
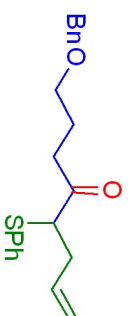
77.4853
77.2300
76.9781
73.0380
69.4529

56.4153

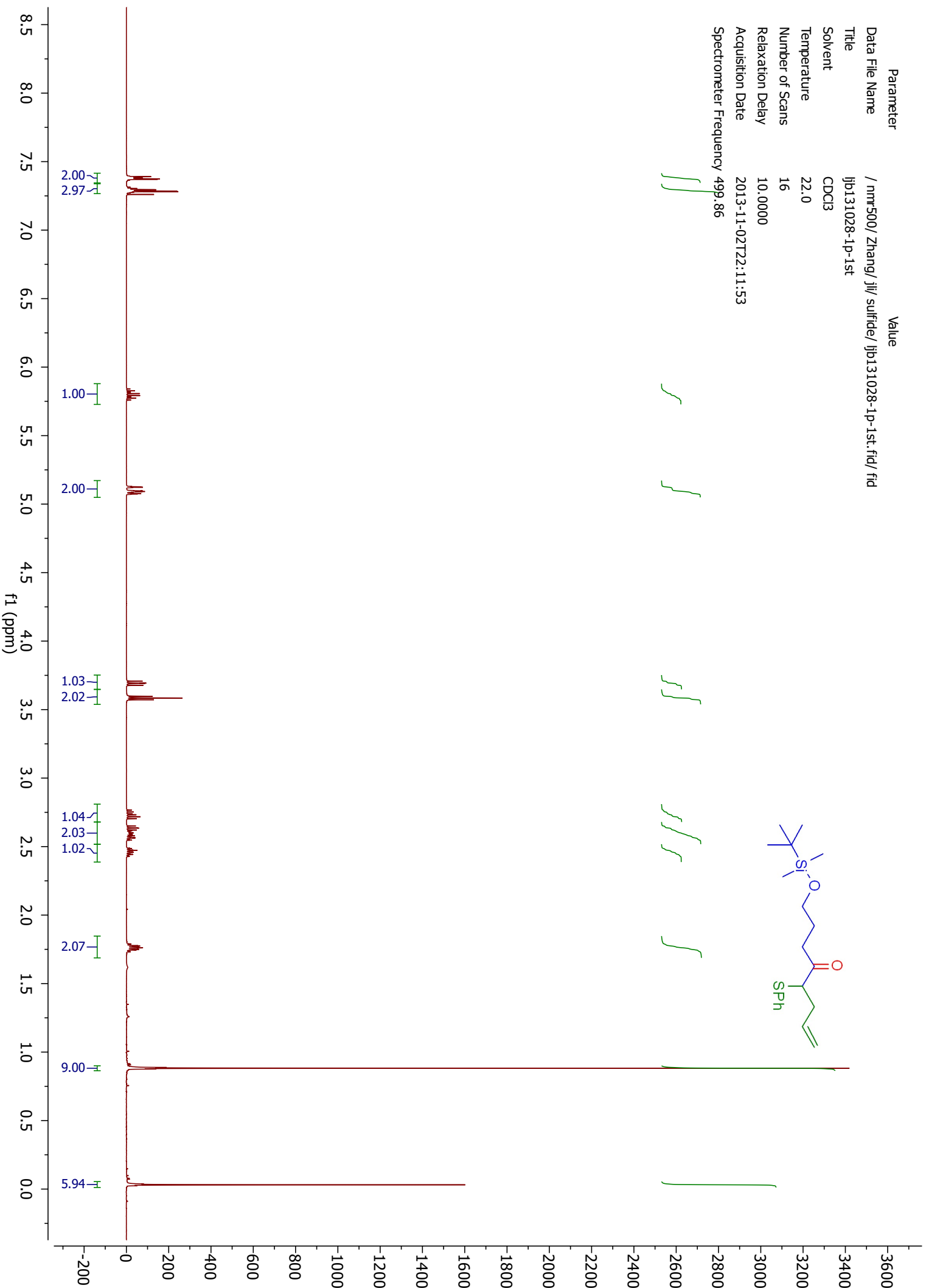
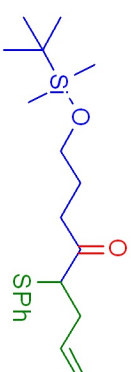
36.9074
34.7433

24.1970

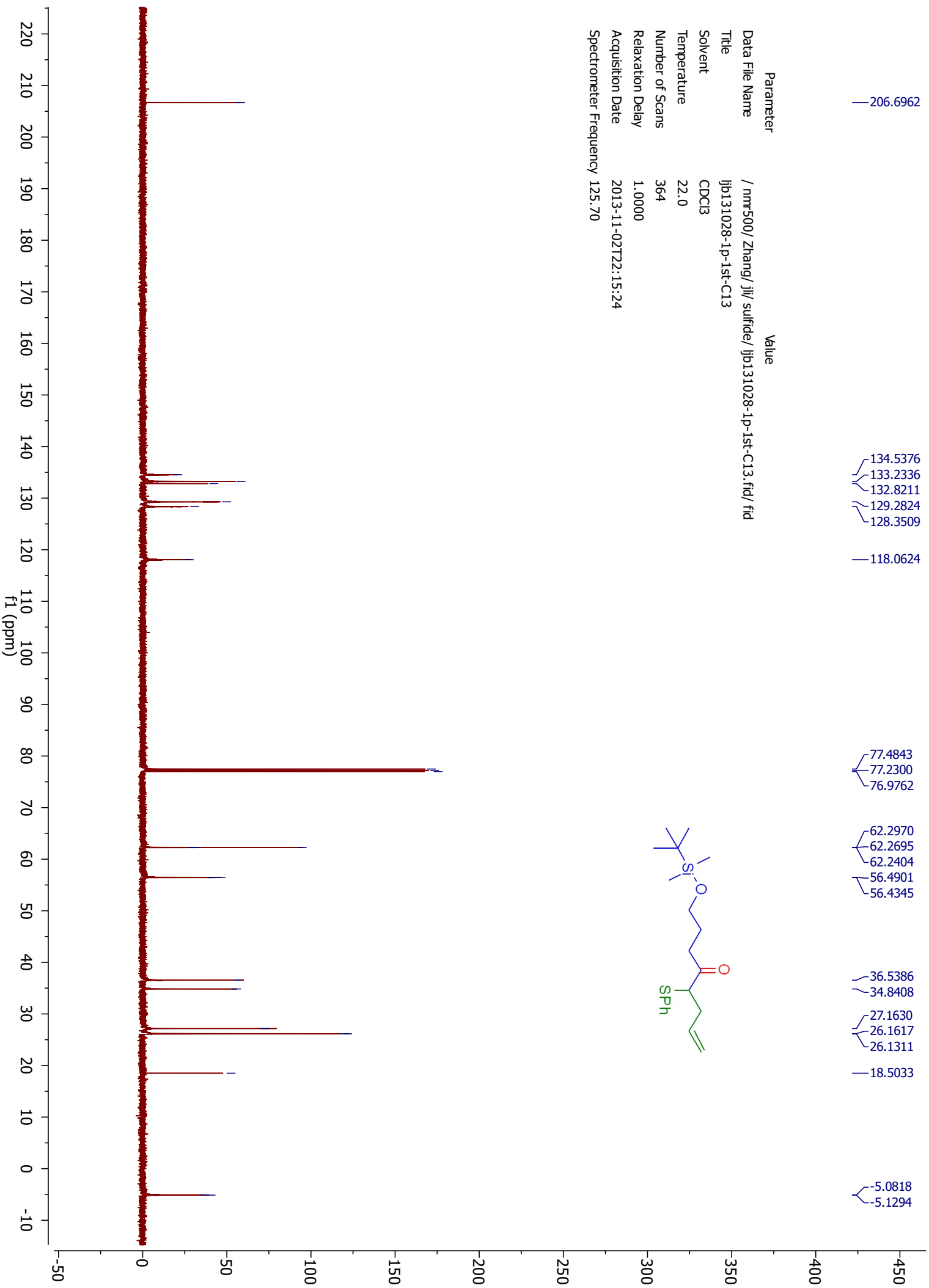
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Data File Name	/nmr500/ Zhang/ jlv/ sulfide/ jlb131028-2p-1st-C13.fid/ fid
Title	jlb131028-2p-1st-C13
Solvent	CDCl3
Temperature	22.0
Number of Scans	188
Relaxation Delay	1.0000
Acquisition Date	2013-11-02T22:53:03
Spectrometer Frequency	125.70



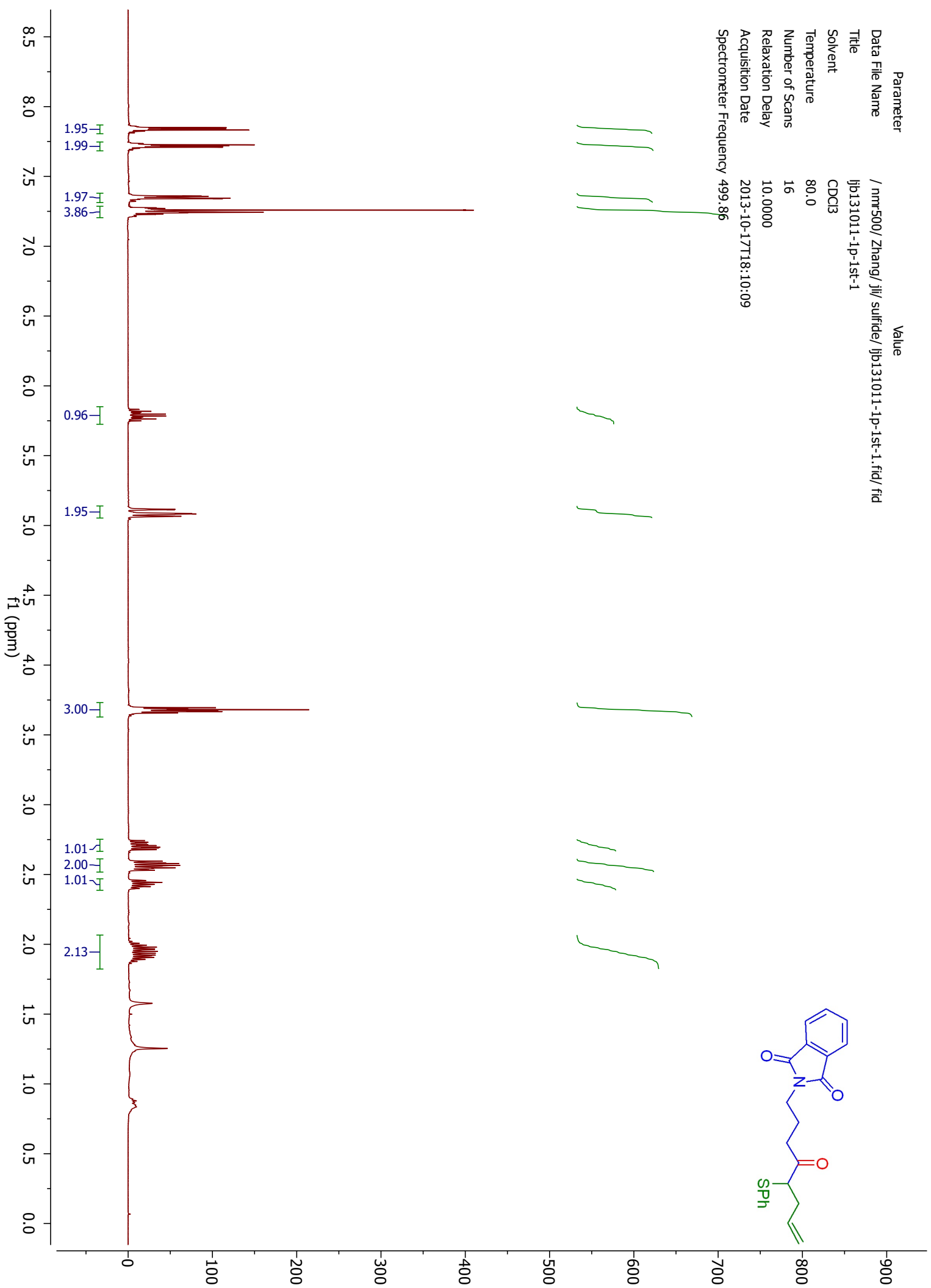
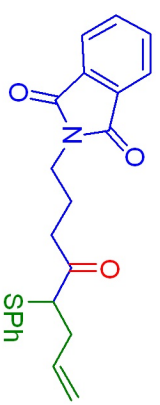
Parameter	Value
Data File Name	/ nmr500/ Zhang/ jll/ sulfide/ jlb131028-1p-1st.fid/ fid
Title	jlb131028-1p-1st
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-02T12:11:53
Spectrometer Frequency	499.86



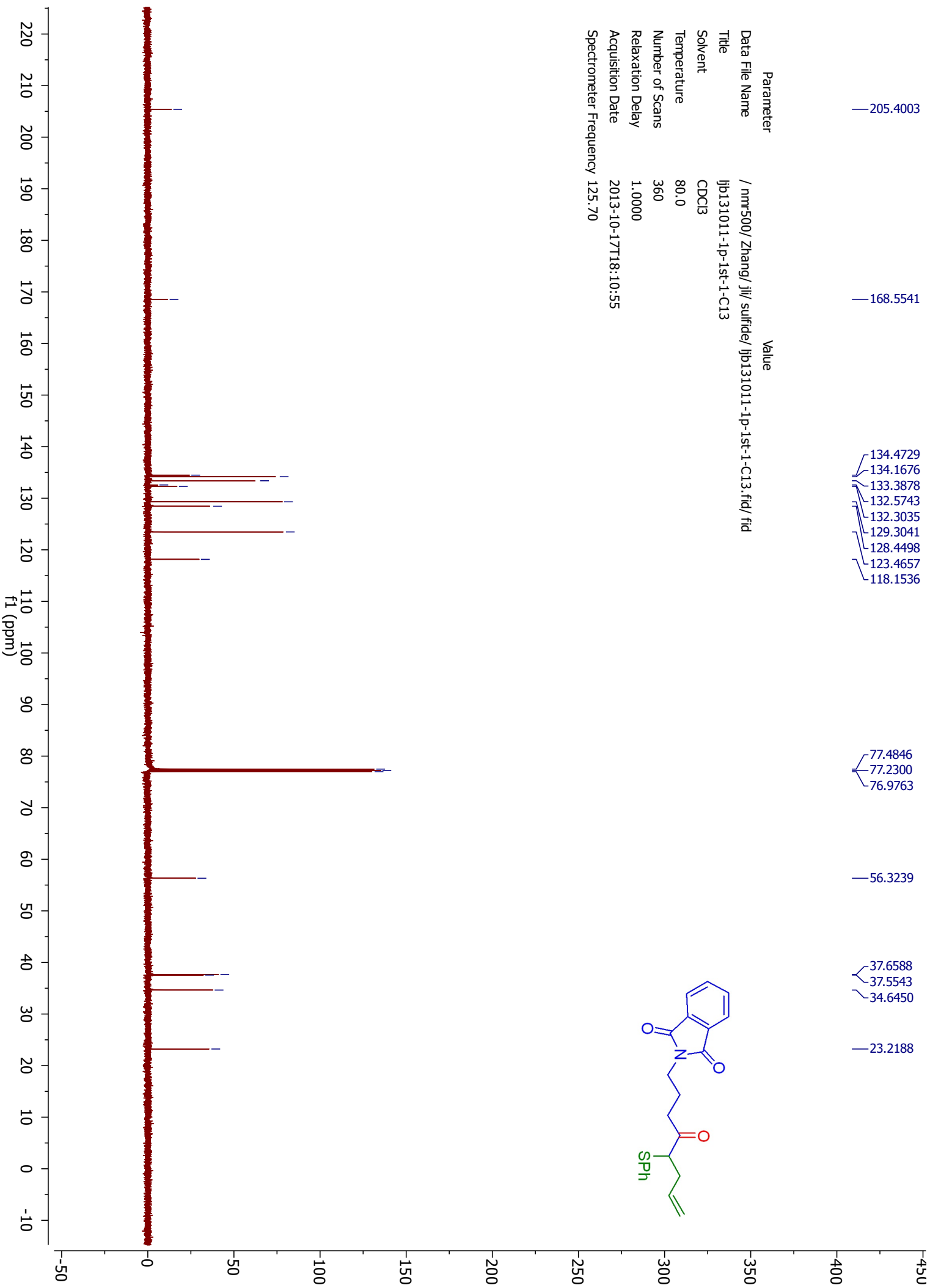
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Data File Name	/nmr500/Zhang/jlj/ sulfide/ jlb131028-1p-1st-C13.fid/ fid
Title	jlb131028-1p-1st-C13
Solvent	CDCl3
Temperature	22.0
Number of Scans	364
Relaxation Delay	1.0000
Acquisition Date	2013-11-02T22:15:24
Spectrometer Frequency	125.70



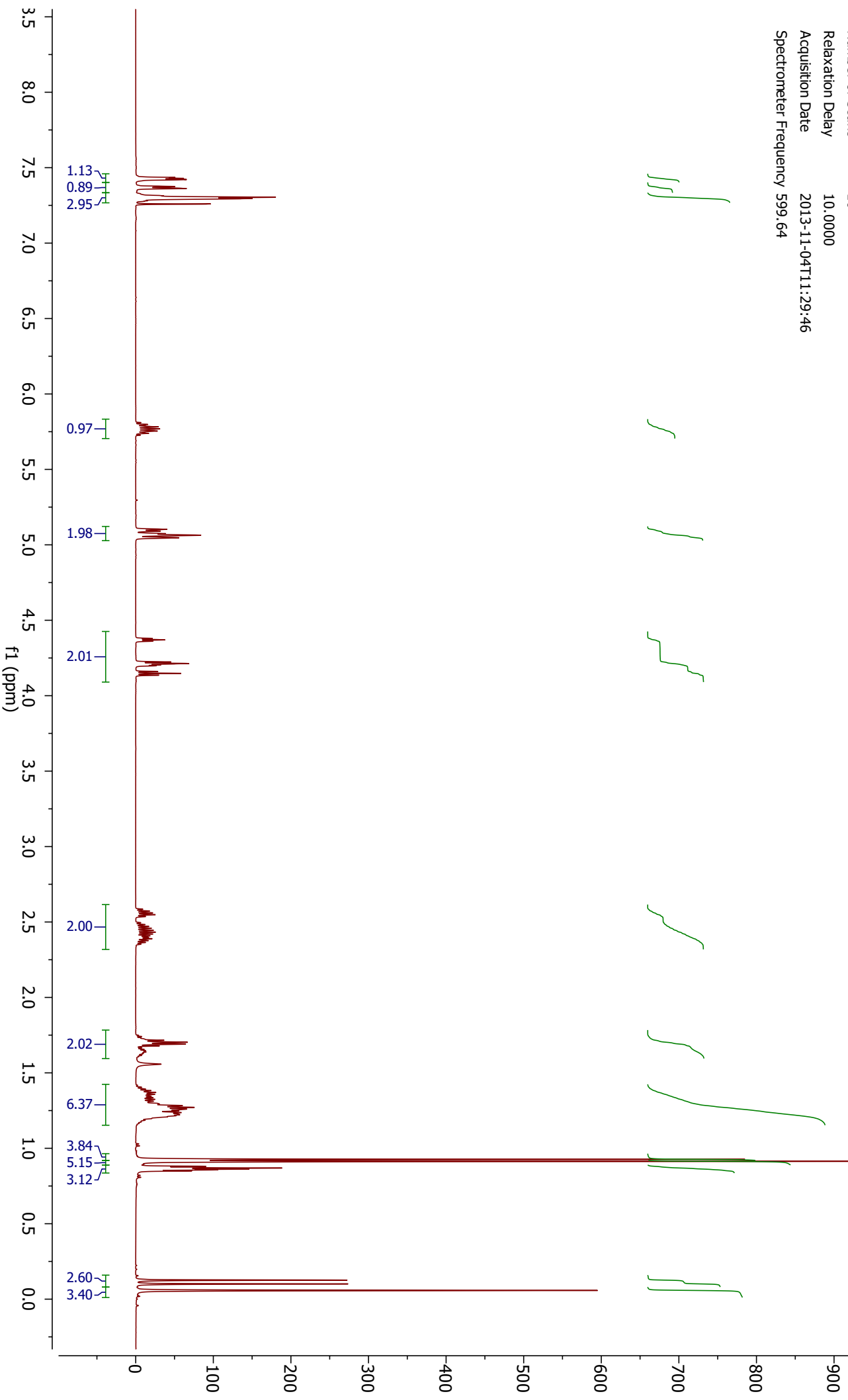
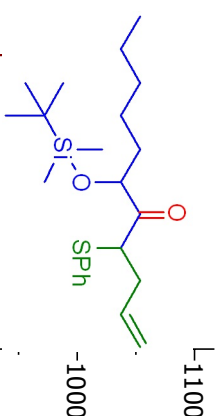
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Data File Name	/nmr500/Zhang/jll/sulfide/jlb131011-1p-1st-1.fid/ fid
Title	jlb131011-1p-1st-1
Solvent	CDCl3
Temperature	80.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-17T18:10:09
Spectrometer Frequency	499.86



Parameter	Value
Data File Name	/nmr500/Zhang/jil/ sulfide/jlb131011-1p-1st-1-C13.fid/ fid
Title	jlb131011-1p-1st-1-C13
Solvent	CDCl3
Temperature	80.0
Number of Scans	360
Relaxation Delay	1.0000
Acquisition Date	2013-10-17T18:10:55
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr600/Zhang/jll/sulfide/jlb131024-2p-1.fid/fid
Title	jlb131024-2p-1
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-04T11:29:46
Spectrometer Frequency	599.64



207.7463
207.2241

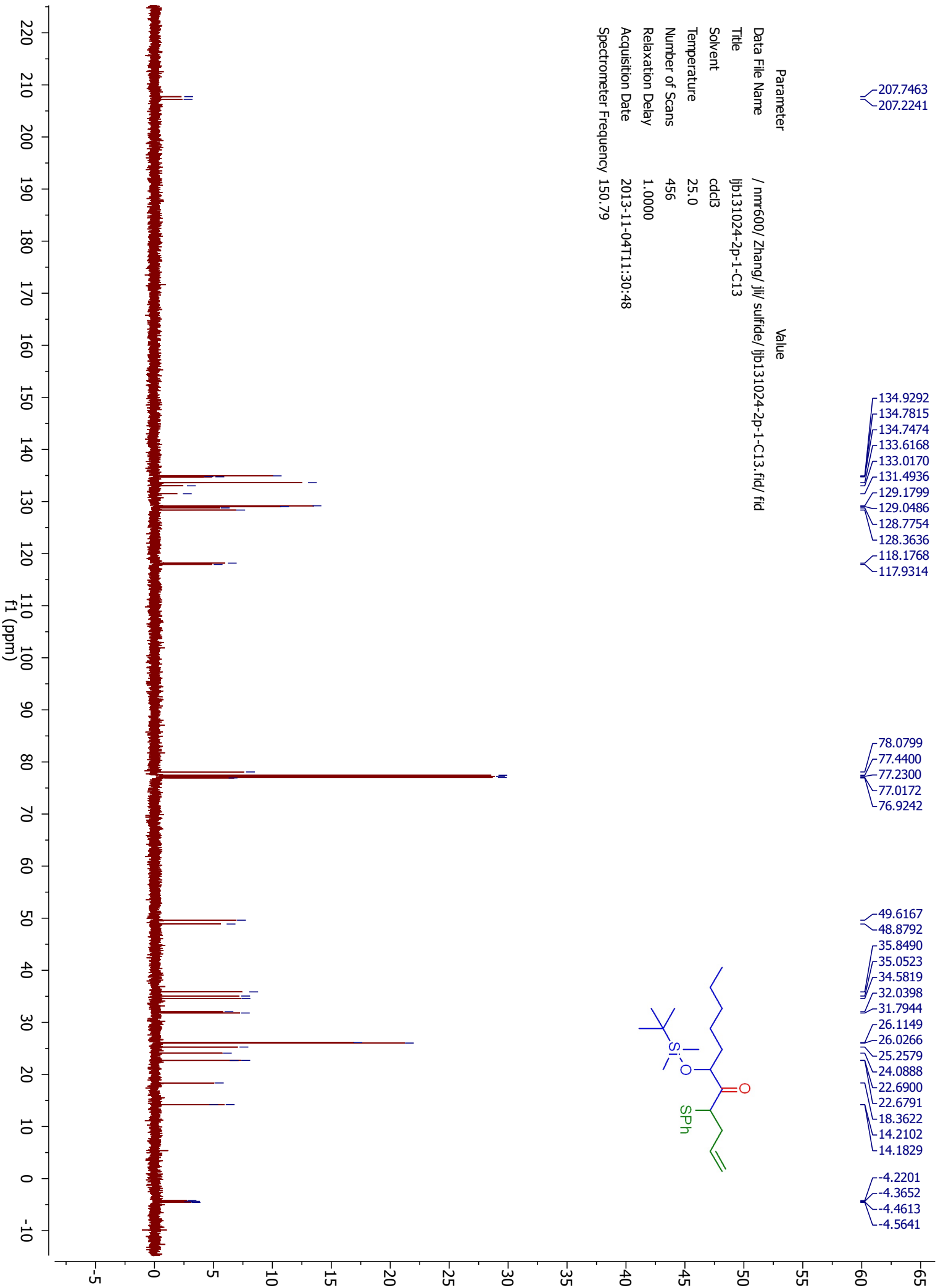
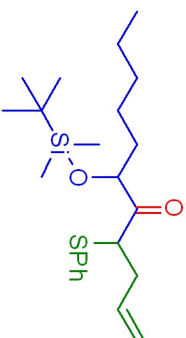
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Title	jpb131024-2p-1-C13
Solvent	cdcl3
Temperature	25.0
Number of Scans	456
Relaxation Delay	1.0000
Acquisition Date	2013-11-04T11:30:48
Spectrometer Frequency	150.79

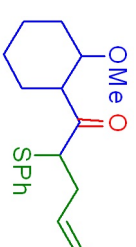
134.9292
134.7815
134.7474
133.6168
133.0170
131.4936
129.1799
129.0486
128.7754
128.3636
118.1768
117.9314

78.0799
77.4400
77.2300
77.0172
76.9242

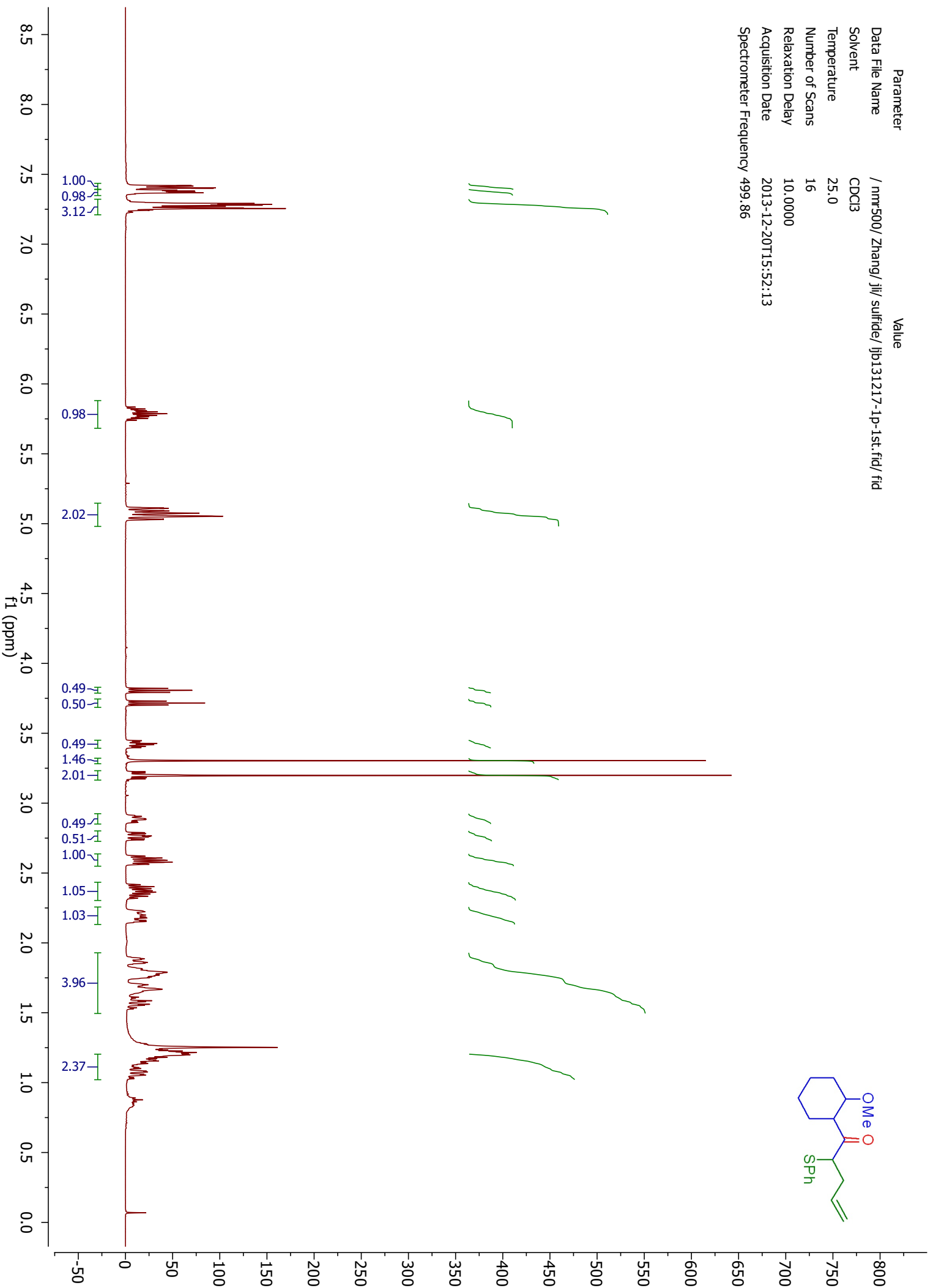
49.6167
48.8792
35.8490
35.0523
34.5819
32.0398
31.7944
26.1149
26.0266
25.2579
24.0888
22.6900
22.6791
18.3622
14.2102
14.1829

-4.2201
-4.3652
-4.4613
-4.5641





Parameter	Value
Data File Name	/nmr500/Zhang/jll/sulfide/jlb131217-1p-1st.fid/fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-12-20T15:52:13
Spectrometer Frequency	499.86



208.6994
207.8920

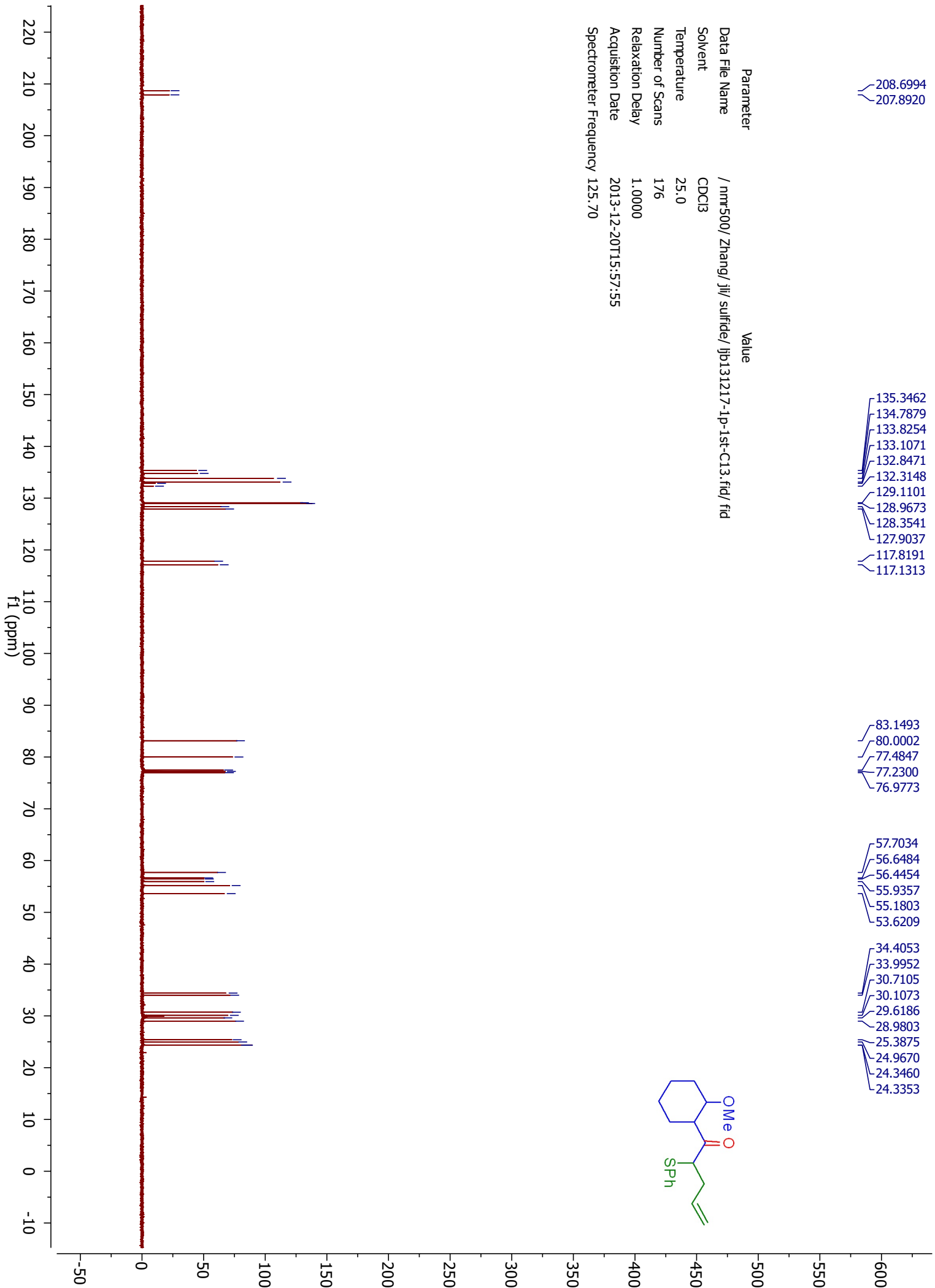
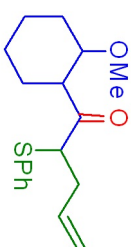
135.3462
134.7879
133.8254
133.1071
132.8471
132.3148
129.1101
128.9673
128.3541
127.9037
117.8191
117.1313

83.1493
80.0002
77.4847
77.2300
76.9773

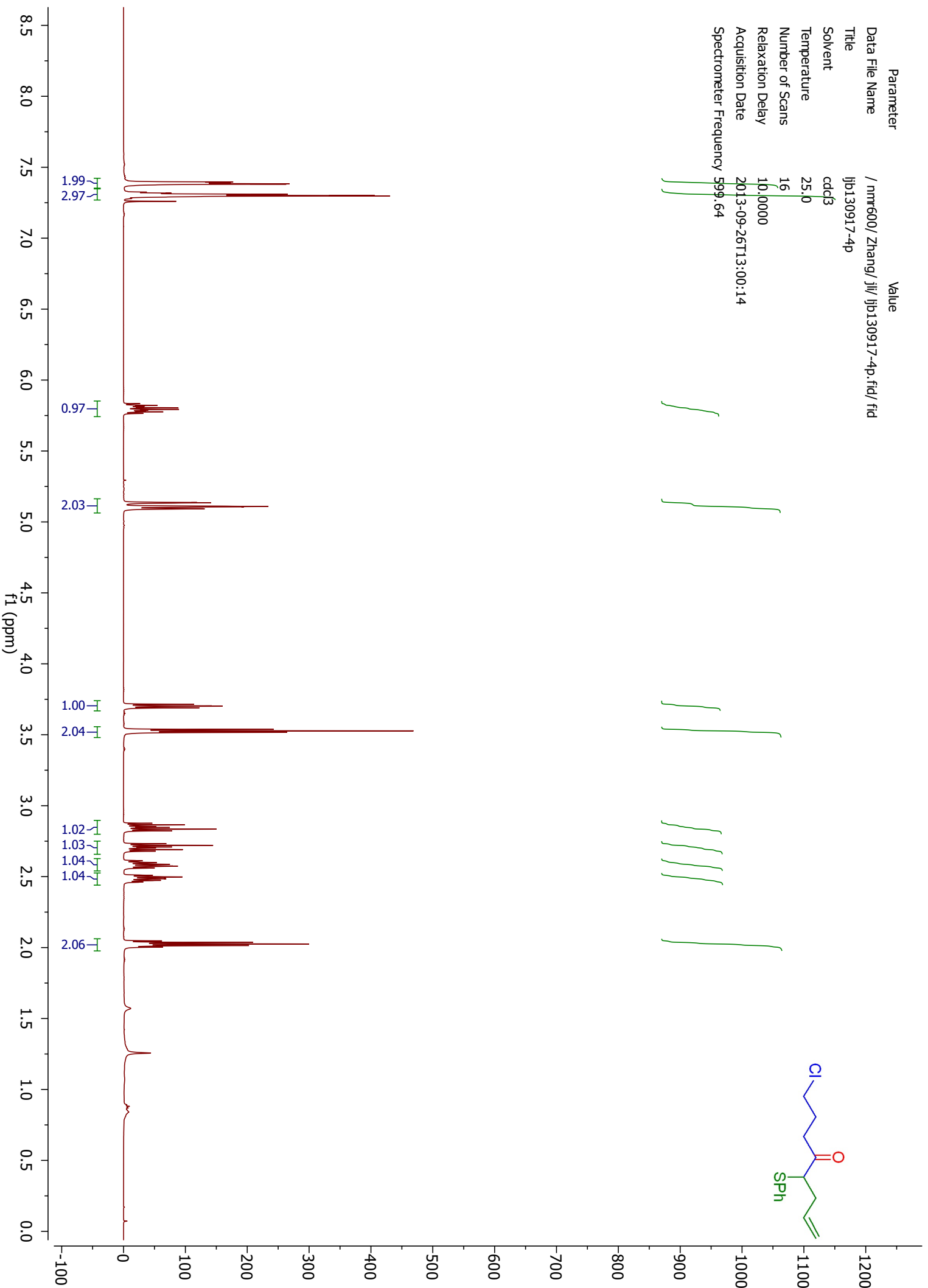
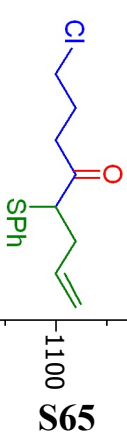
57.7034
56.6484
56.4454
55.9357
55.1803
53.6209

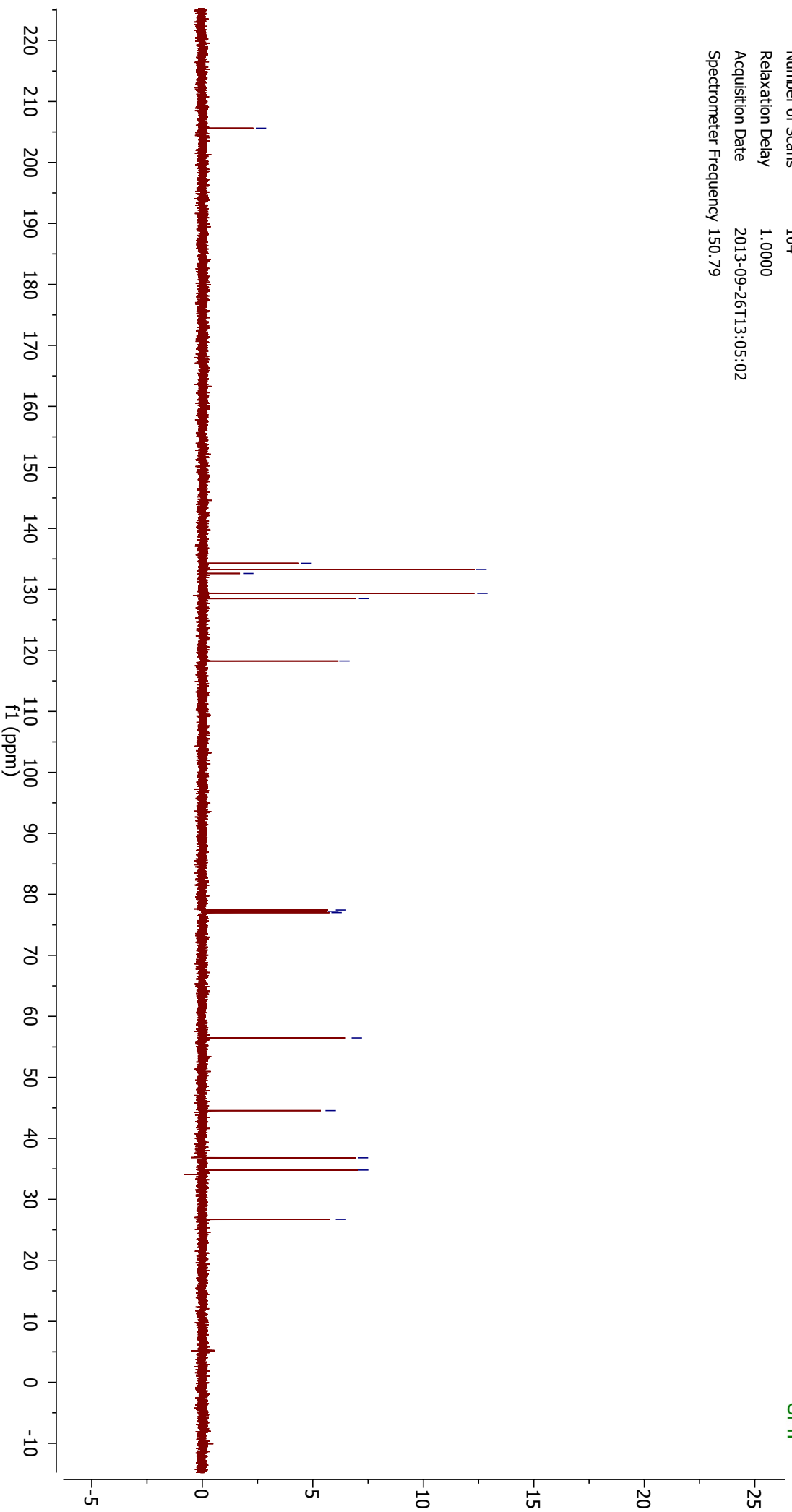
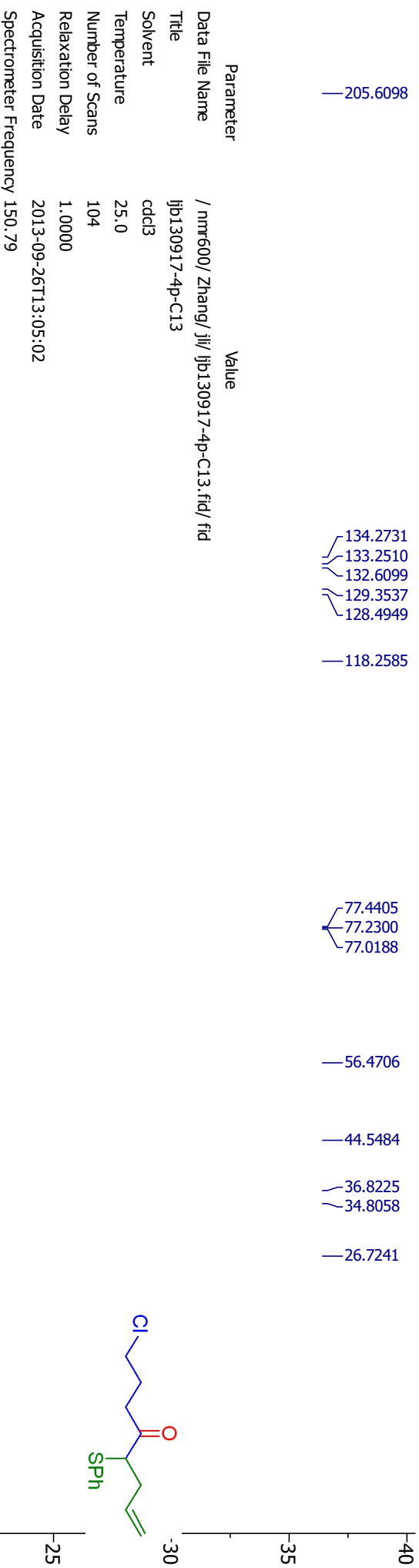
34.4053
33.9952
30.7105
30.1073
29.6186
28.9803
25.3875
24.9670
24.3460
24.3353

Parameter	Value
Data File Name	/nmr500/Zhang/jll/ sulfide/ jlb131217-1p-1st-C13.fid/ fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	176
Relaxation Delay	1.0000
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Spectrometer Frequency	125.70

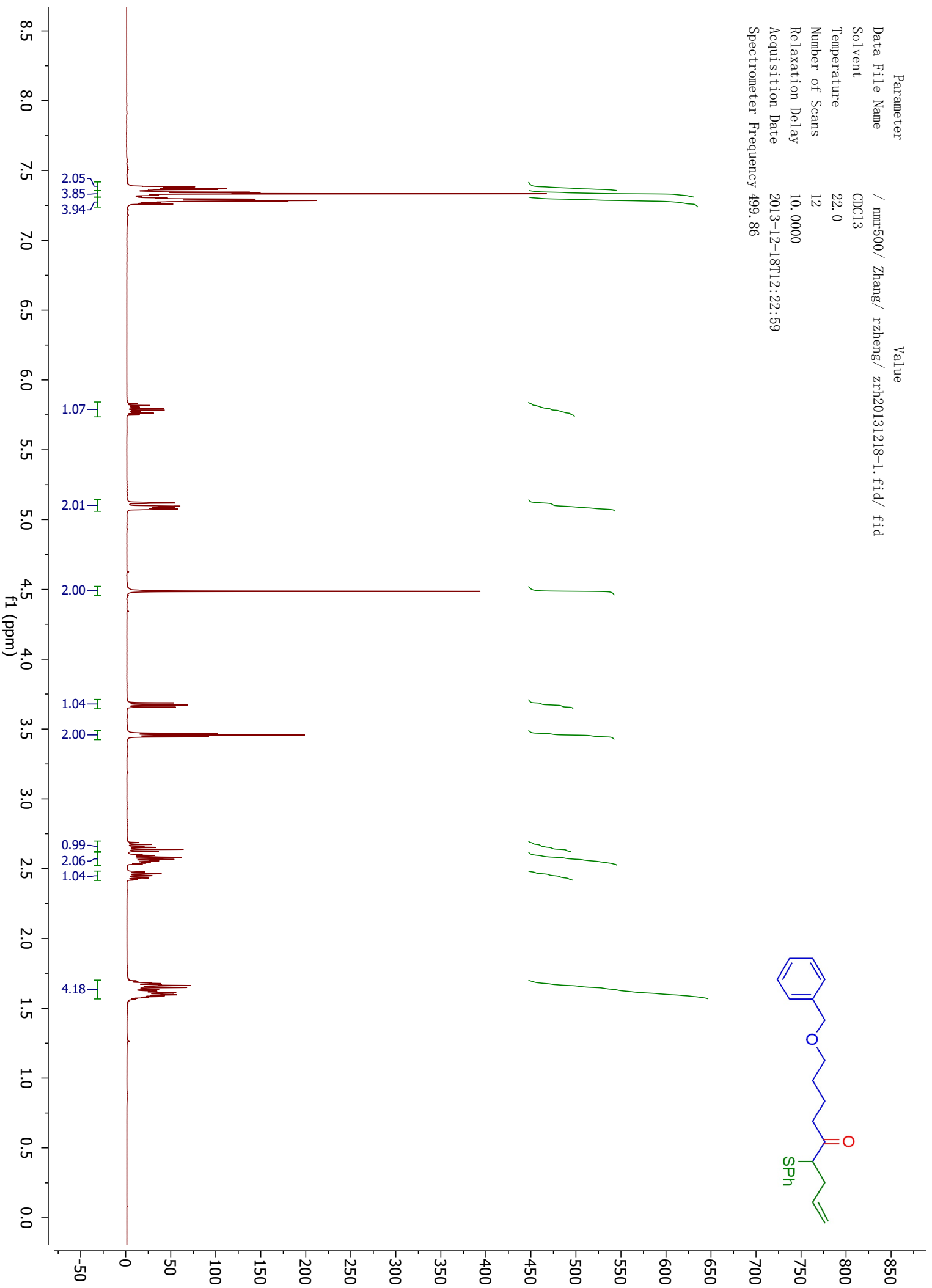
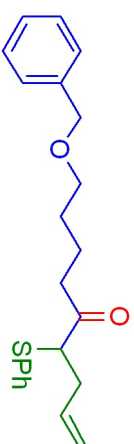


Parameter	Value
Data File Name	/nmr600/Zhang/jli/jlb130917-4p.fid/ fid
Title	jlb130917-4p
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-09-26T13:00:14
Spectrometer Frequency	599.64

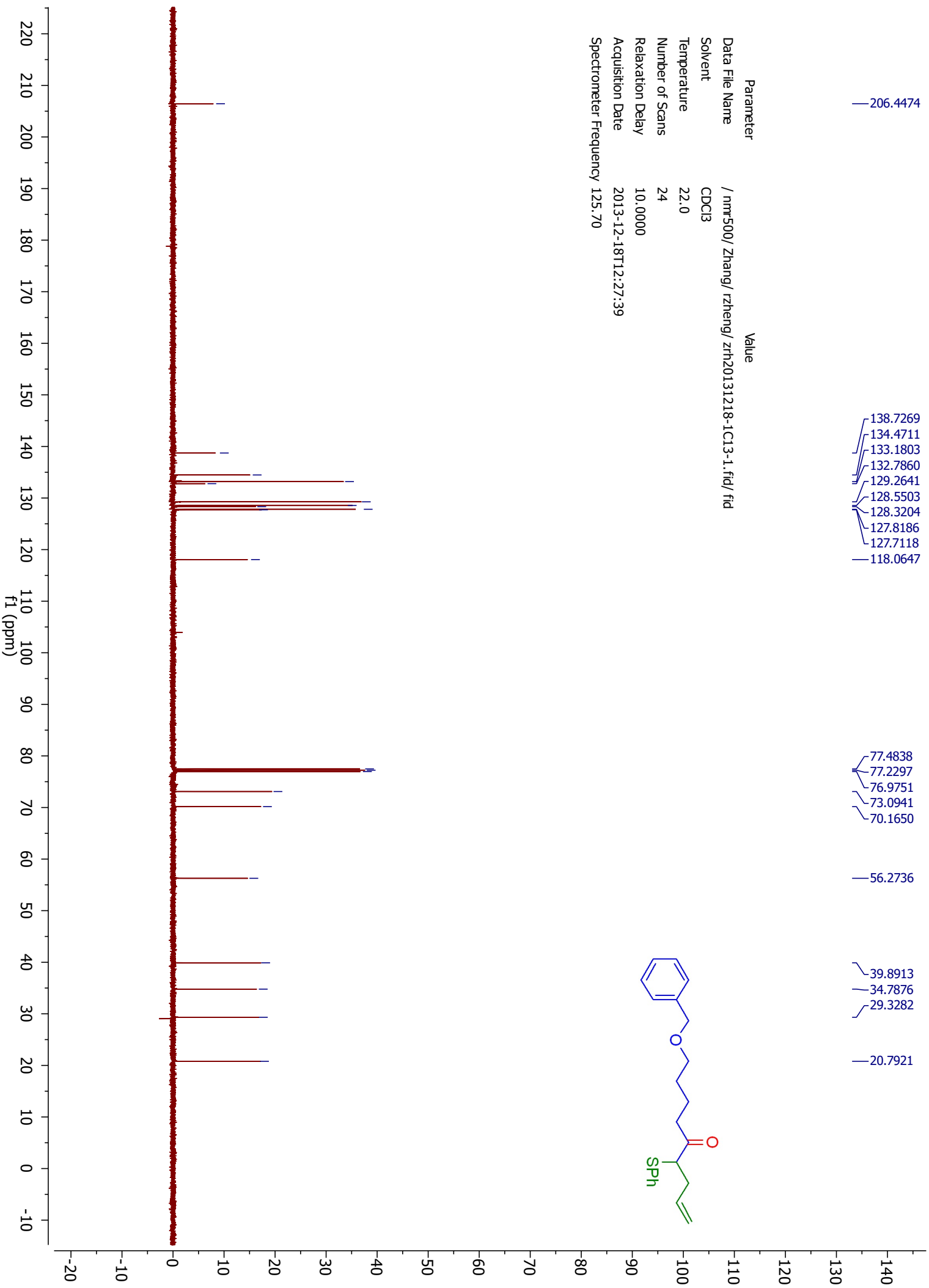




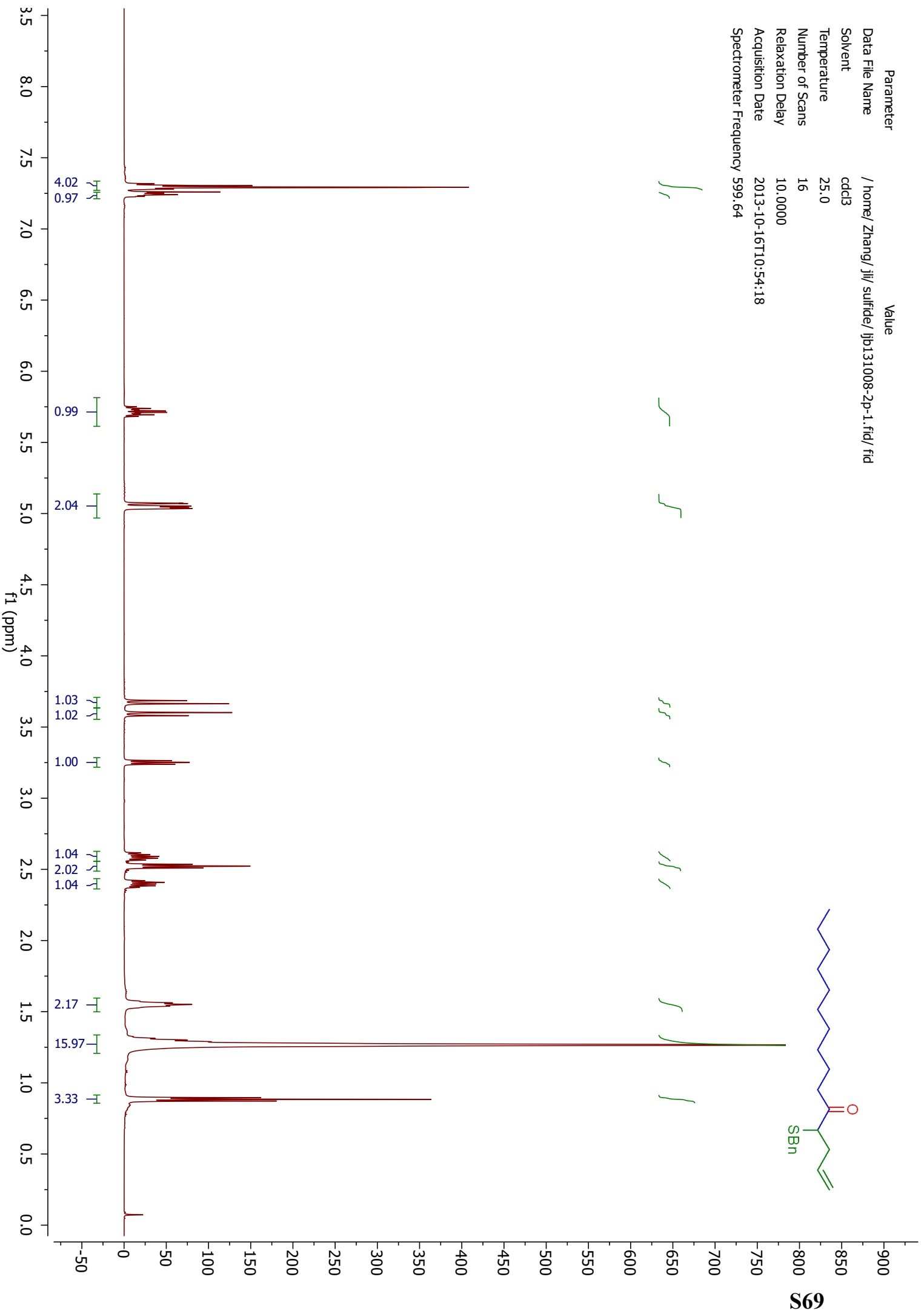
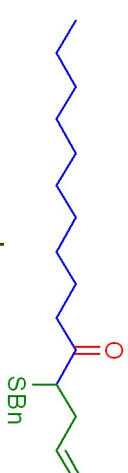
Parameter	Value
Data File Name	/nmr500/ Zhang/ rzheng/ zrth20131218-1.fid/ fid
Solvent	CDCl ₃
Temperature	22.0
Number of Scans	12
Relaxation Delay	10.0000
Acquisition Date	2013-12-18T12:22:59
Spectrometer Frequency	499.86



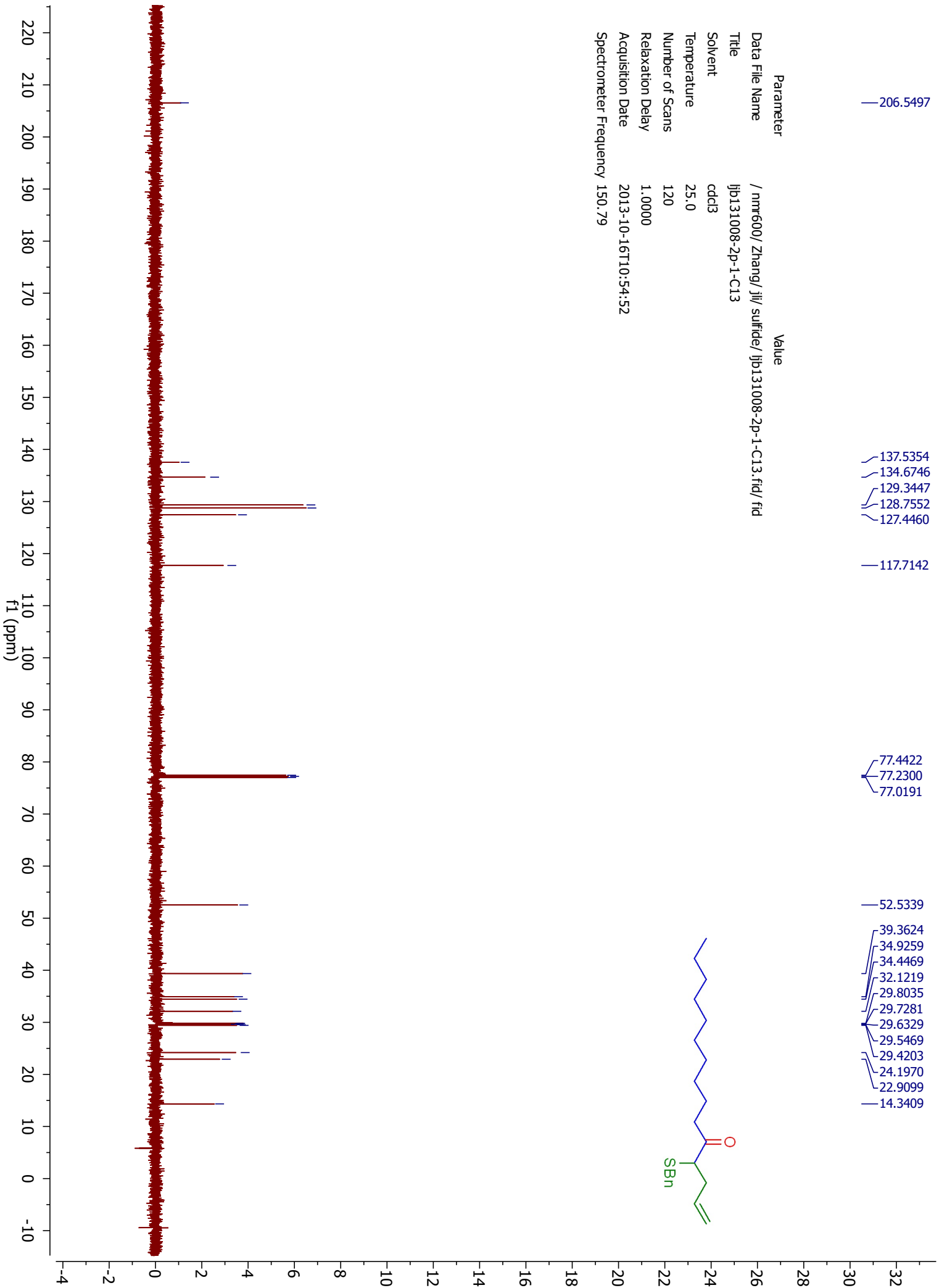
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Data File Name	/nmr500/Zhang/rzheng/zrh20131218-1Cl3-1.fid/fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	24
Relaxation Delay	10.0000
Acquisition Date	2013-12-18T12:27:39
Spectrometer Frequency	125.70



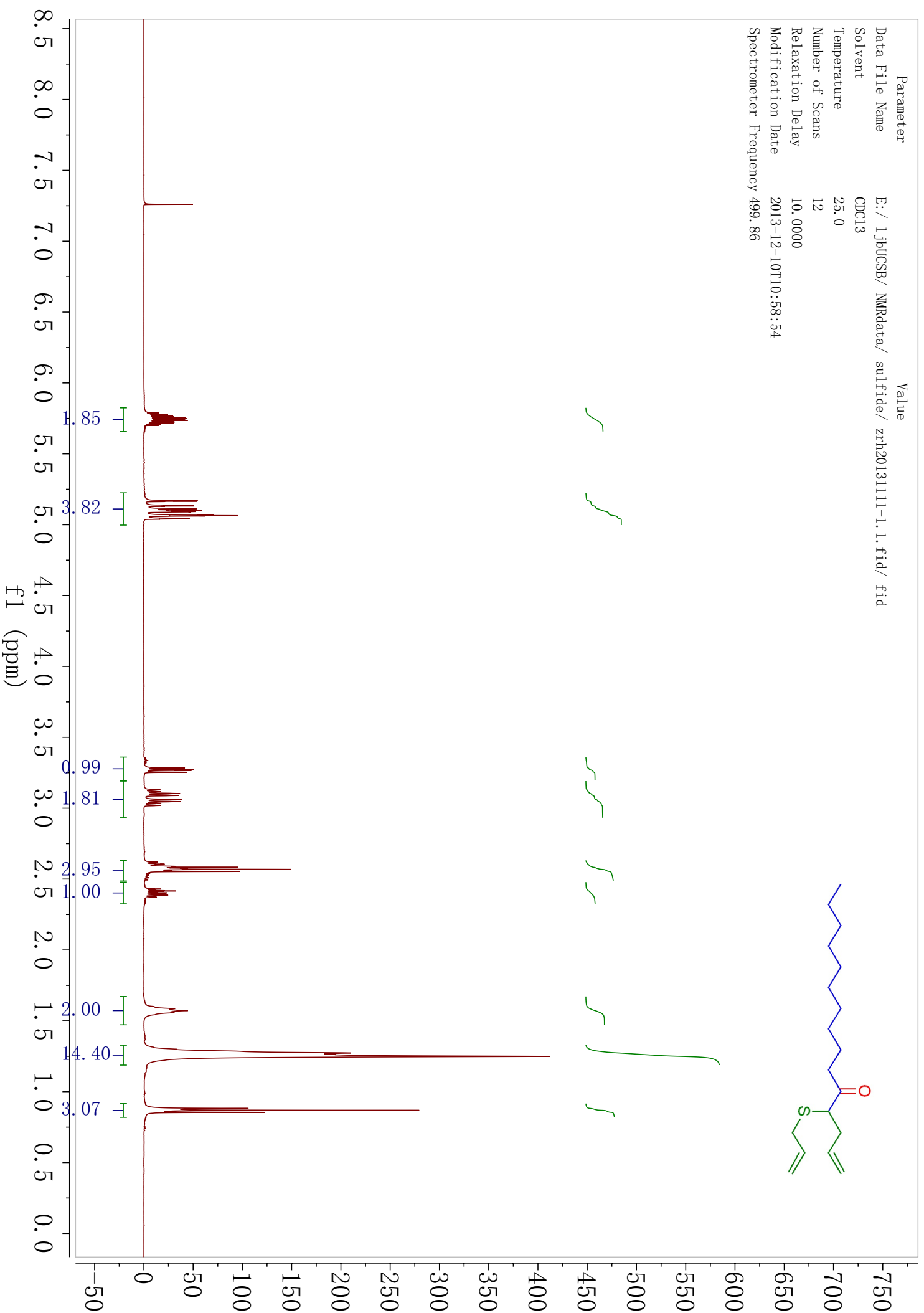
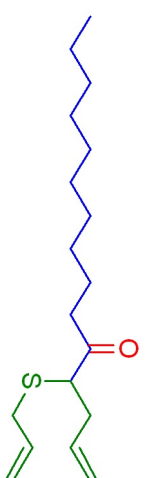
Parameter	Value
Data File Name	/home/Zhang/jll/sulfide/jb131008-2p-1.fid/ fid
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-16T10:54:18
Spectrometer Frequency	599.64



Parameter	Value
Data File Name	/ nmr600/ Zhang/ jil/ sulfide/ jlb131008-2p-1-C13.fid/ fid
Title	jlb131008-2p-1-C13
Solvent	cdcl3
Temperature	25.0
Number of Scans	120
Relaxation Delay	1.0000
Acquisition Date	2013-10-16T10:54:52
Spectrometer Frequency	150.79



Parameter	Value
Data File Name	E:/ 1jubCSB/ NMRdata/ sulfide/ zrh20131111-1.1.fid/ fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	12
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Modification Date	2013-12-10T10:58:54
Spectrometer Frequency	499.86



—206.7066

134.6856
133.6413

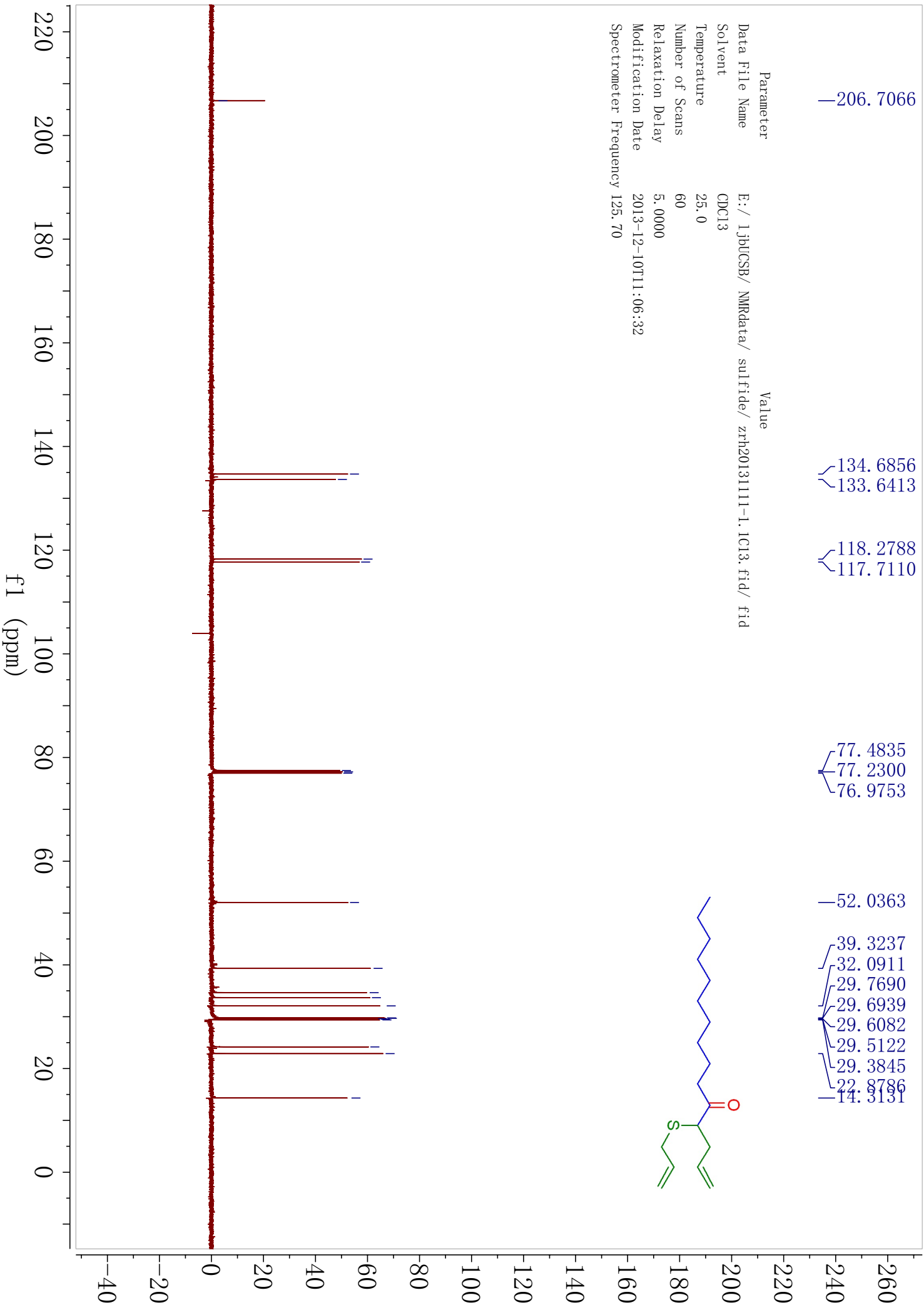
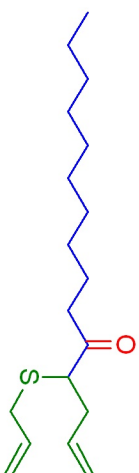
118.2788
117.7110

77.4835
77.2300
76.9753

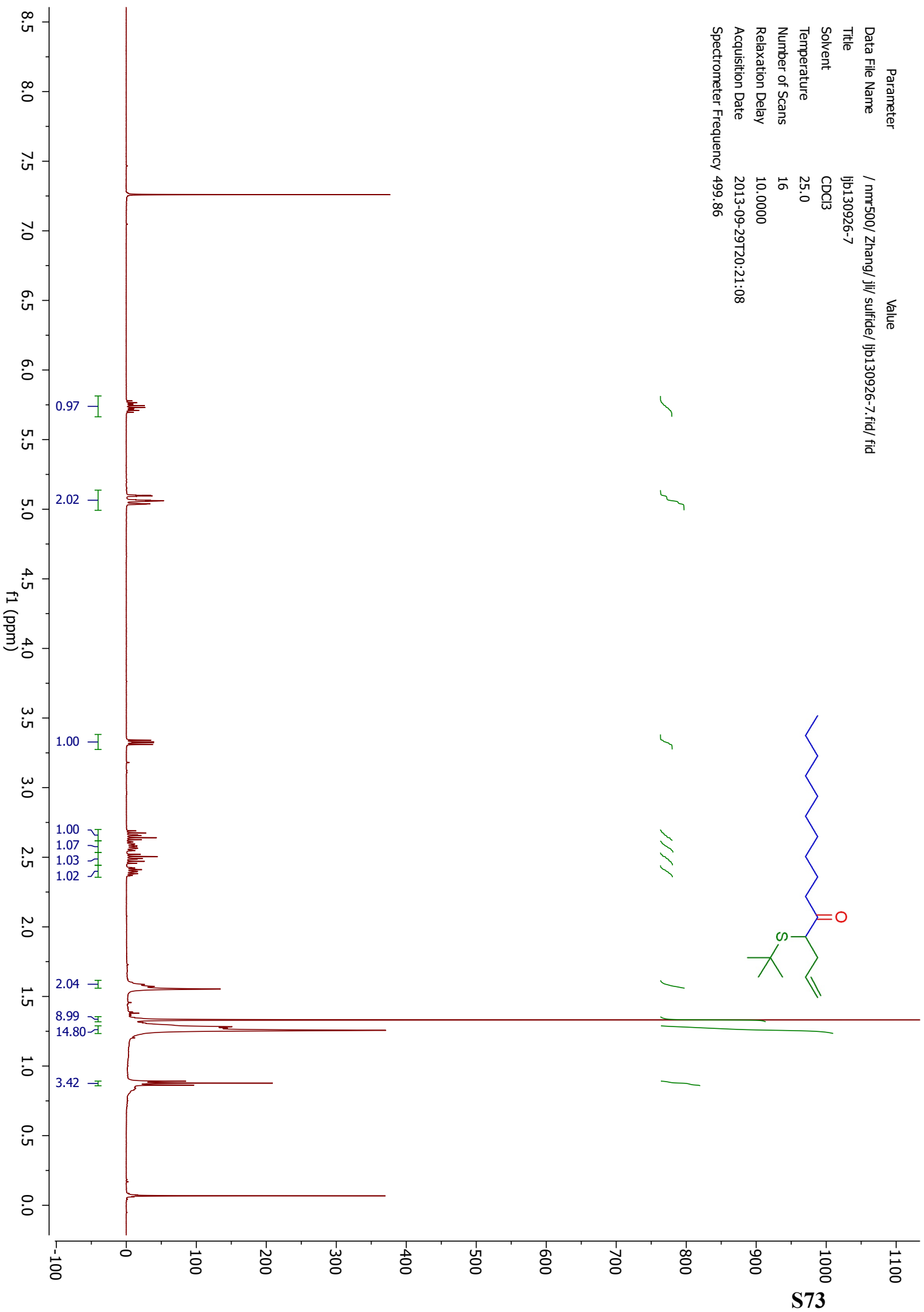
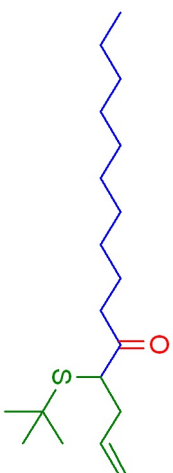
—52.0363

39.3237
32.0911
29.7690
29.6939
29.6082
29.5122
29.3845
22.8786
14.3131

Parameter	Value
Data File Name	E:/ 1jblCSB/ NMRdata/ sulfide/ zrh20131111-1. 1C13. fid/ fid
Solvent	CDCl ₃
Temperature	25.0
Number of Scans	60
Relaxation Delay	5.0000
Modification Date	2013-12-10T11:06:32
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr500/Zhang/jli/sulfide/jlb130926-7.fid/ fid
Title	jlb130926-7
Solvent	CDCl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-09-29T20:21:08
Spectrometer Frequency	499.86



209.1728

134.8786

117.6710

77.4837
77.2300
76.9765

51.9115

44.7783

38.4872

37.4967

31.3982

29.8056

29.7232

29.6459

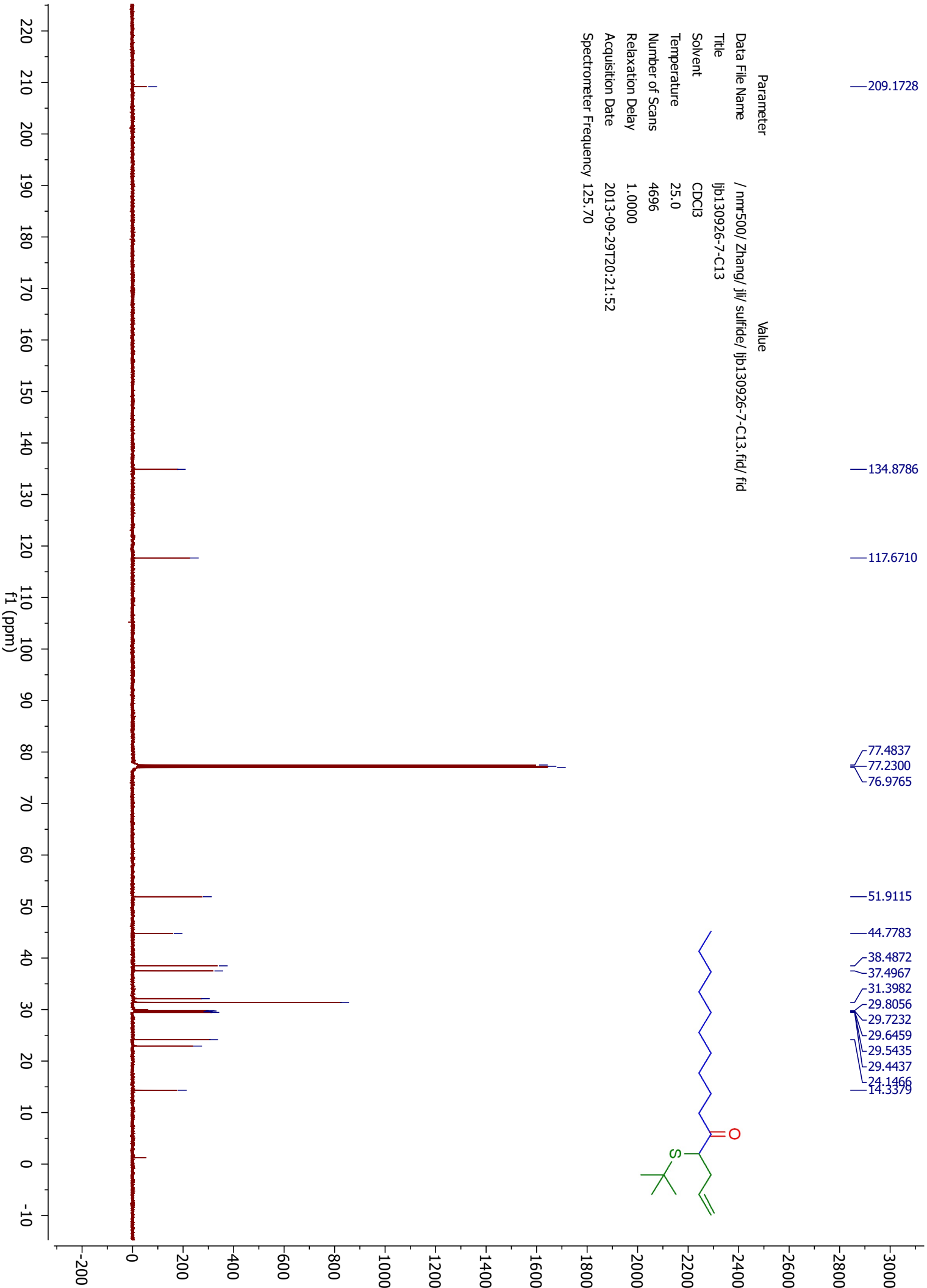
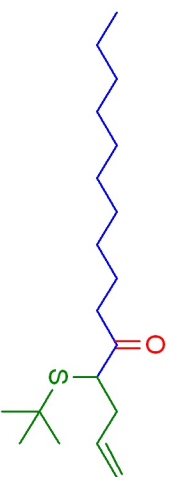
29.5435

29.4437

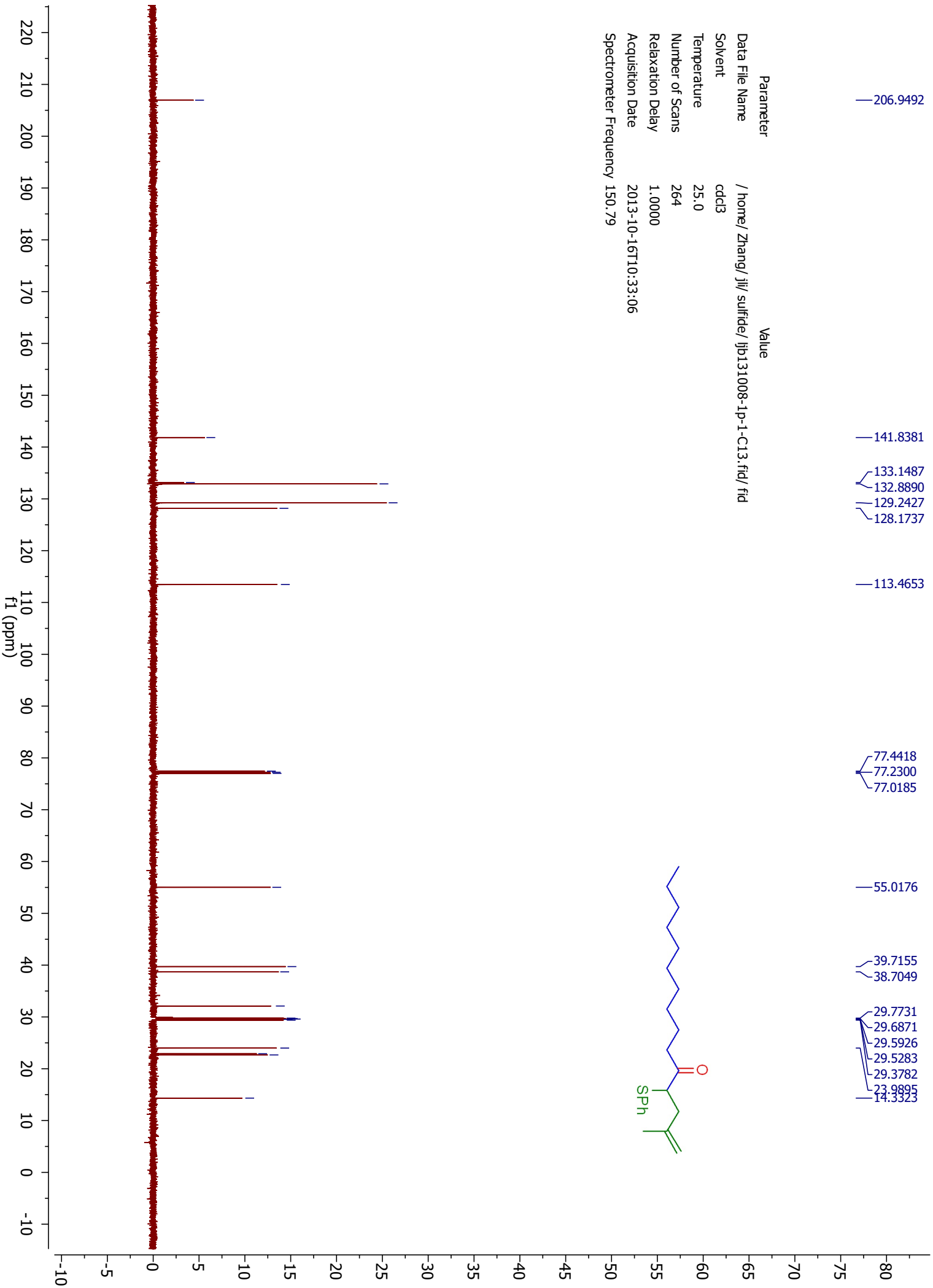
24.1466

14.3379

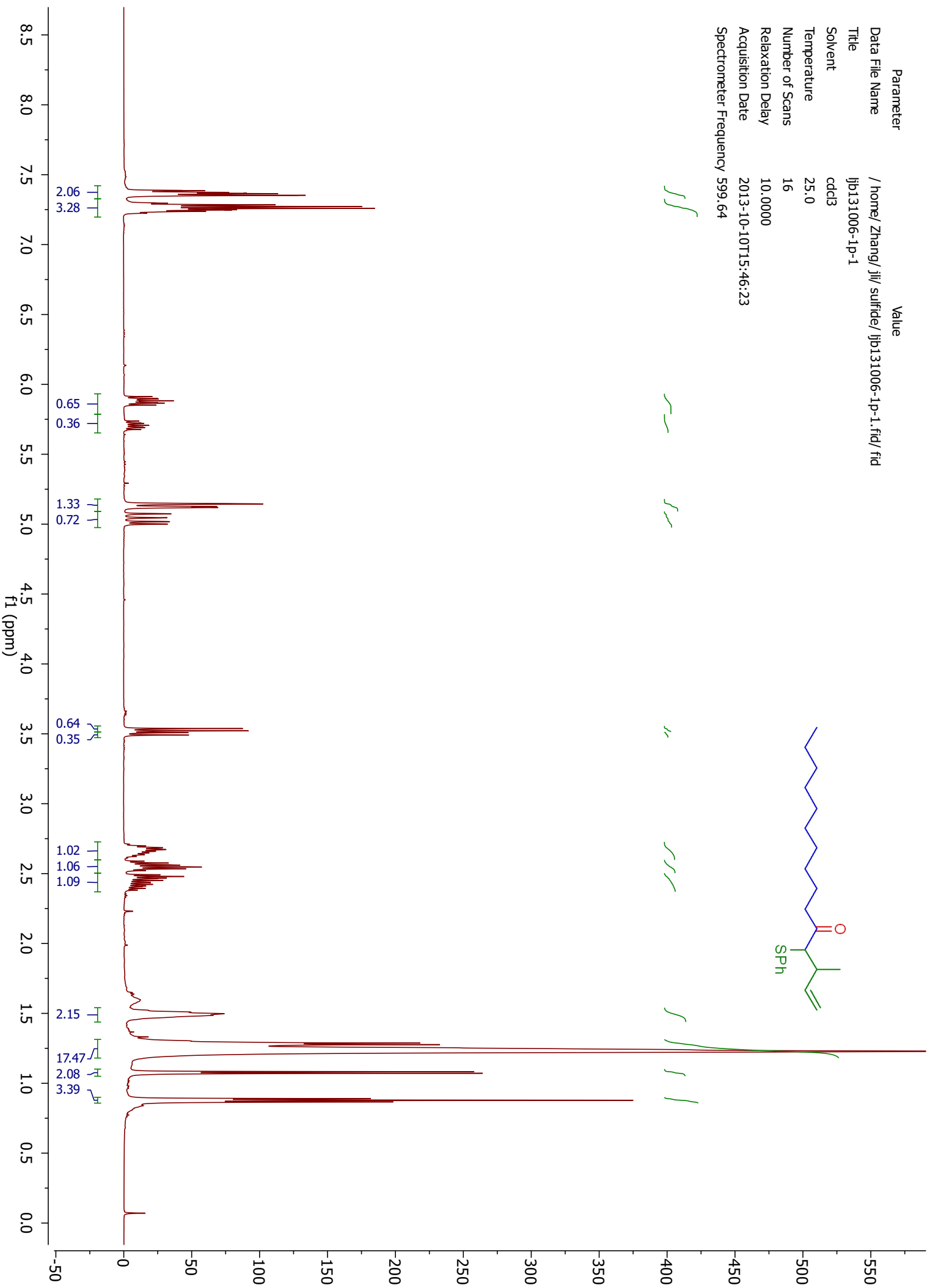
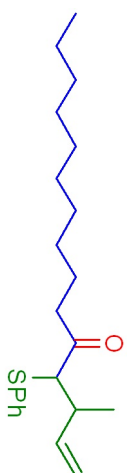
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Title	jlb130926-7-C13
Solvent	CDCl3
Temperature	25.0
Number of Scans	4696
Relaxation Delay	1.0000
Acquisition Date	2013-09-29T20:21:52
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/home/Zhang/jl/ sulfide/ jlb131008-1p-1-C13.fid/ fid
Solvent	cdcl3
Temperature	25.0
Number of Scans	264
Relaxation Delay	1.0000
Acquisition Date	2013-10-16T10:33:06
Spectrometer Frequency	150.79



Parameter	Value
Data File Name	/home/Zhang/jli/sulfide/jb131006-1p-1.fid/ fid
Title	jb131006-1p-1
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-10T15:46:23
Spectrometer Frequency	599.64



207.3232
207.0647

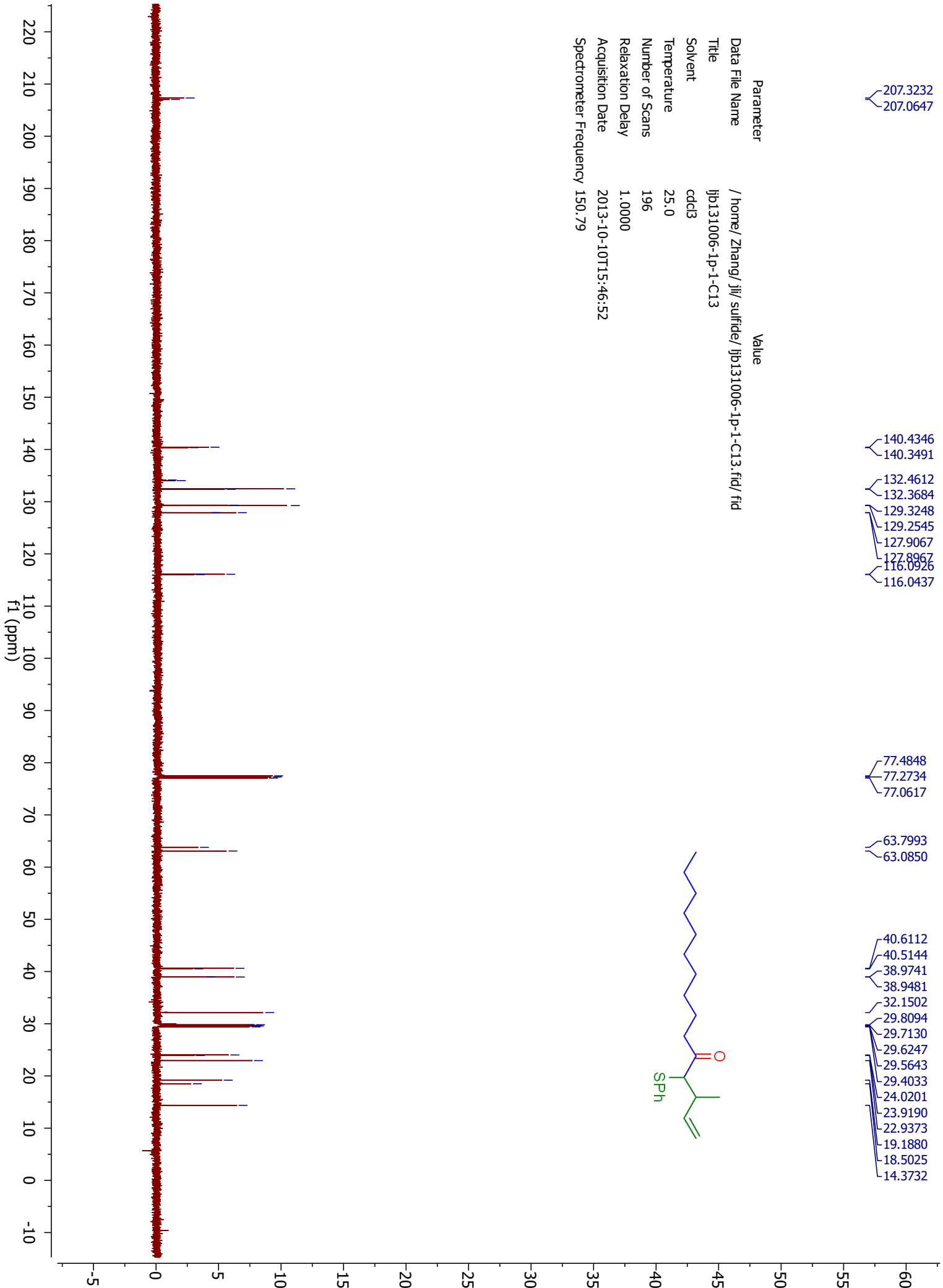
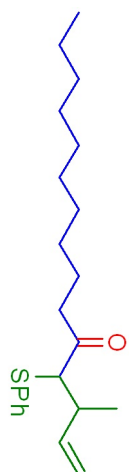
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140.3491
132.4612
132.3684
129.3248
129.2545
127.9067
127.8967
116.0926
116.0437

77.4848
77.2734
77.0617

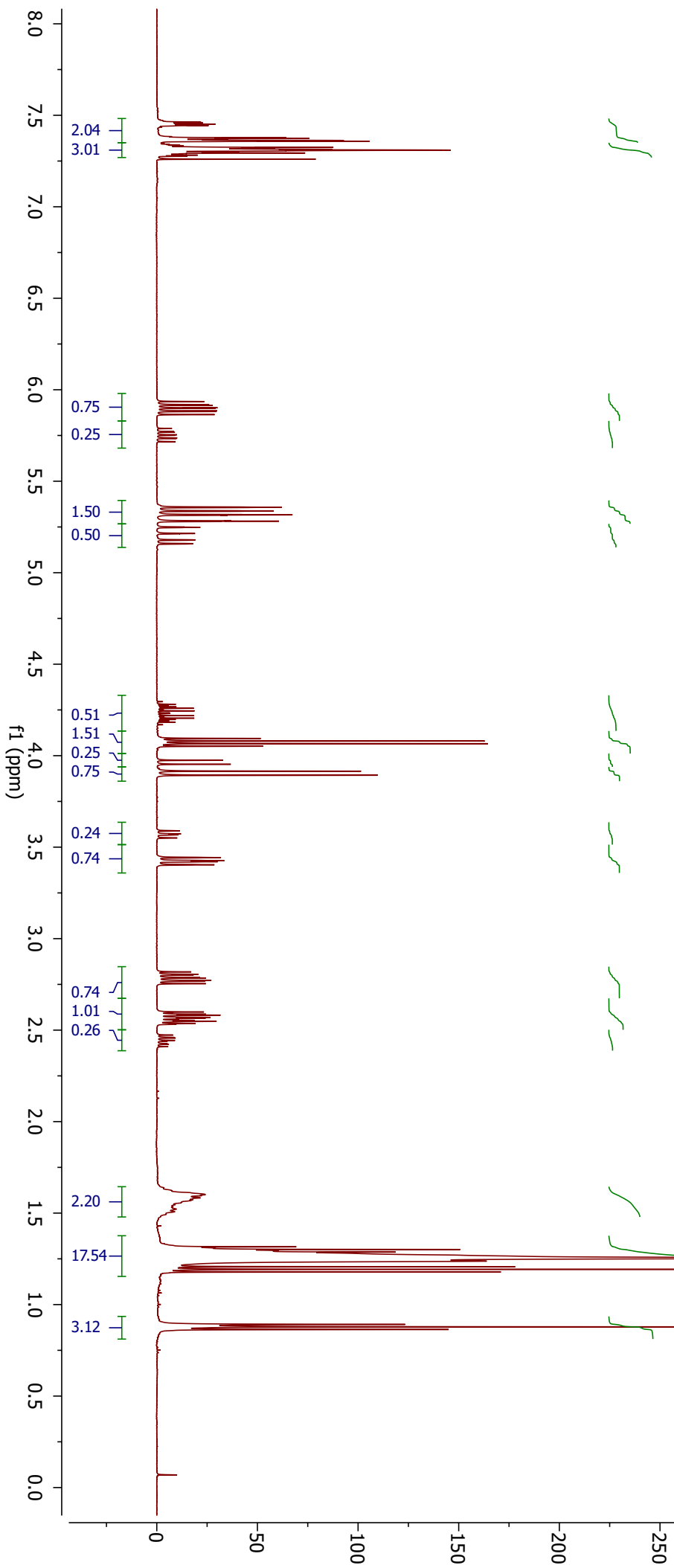
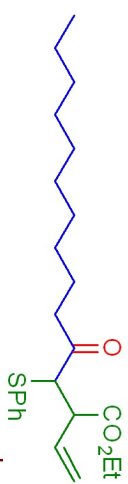
63.7993
63.0850

40.6112
40.5144
38.9741
38.9481
32.1502
29.8094
29.7130
29.6247
29.5643
29.4033
24.0201
23.9190
22.9373
19.1880
18.5025
14.3732

Parameter	Value
Data File Name	/home/Zhang/jl/sulfide/jlb131006-1p-1-C13.fid/ fid
Title	jlb131006-1p-1-C13
Solvent	cdcl3
Temperature	25.0
Number of Scans	196
Relaxation Delay	1.0000
Acquisition Date	2013-10-10T15:46:52
Spectrometer Frequency	150.79



Parameter	Value
Data File Name	/nmr500/Zhang/jli/sulfide/jlb131030-1p-1st.fid/fid
Title	jlb131030-1p-1st
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-02T21:46:44
Spectrometer Frequency	499.86



205.4264
203.4677

172.3502
171.4142

134.2818
133.9852
133.0185
132.9581
132.6395
132.5827
131.9485
131.0051
129.1087
128.8342
120.1725
120.1084

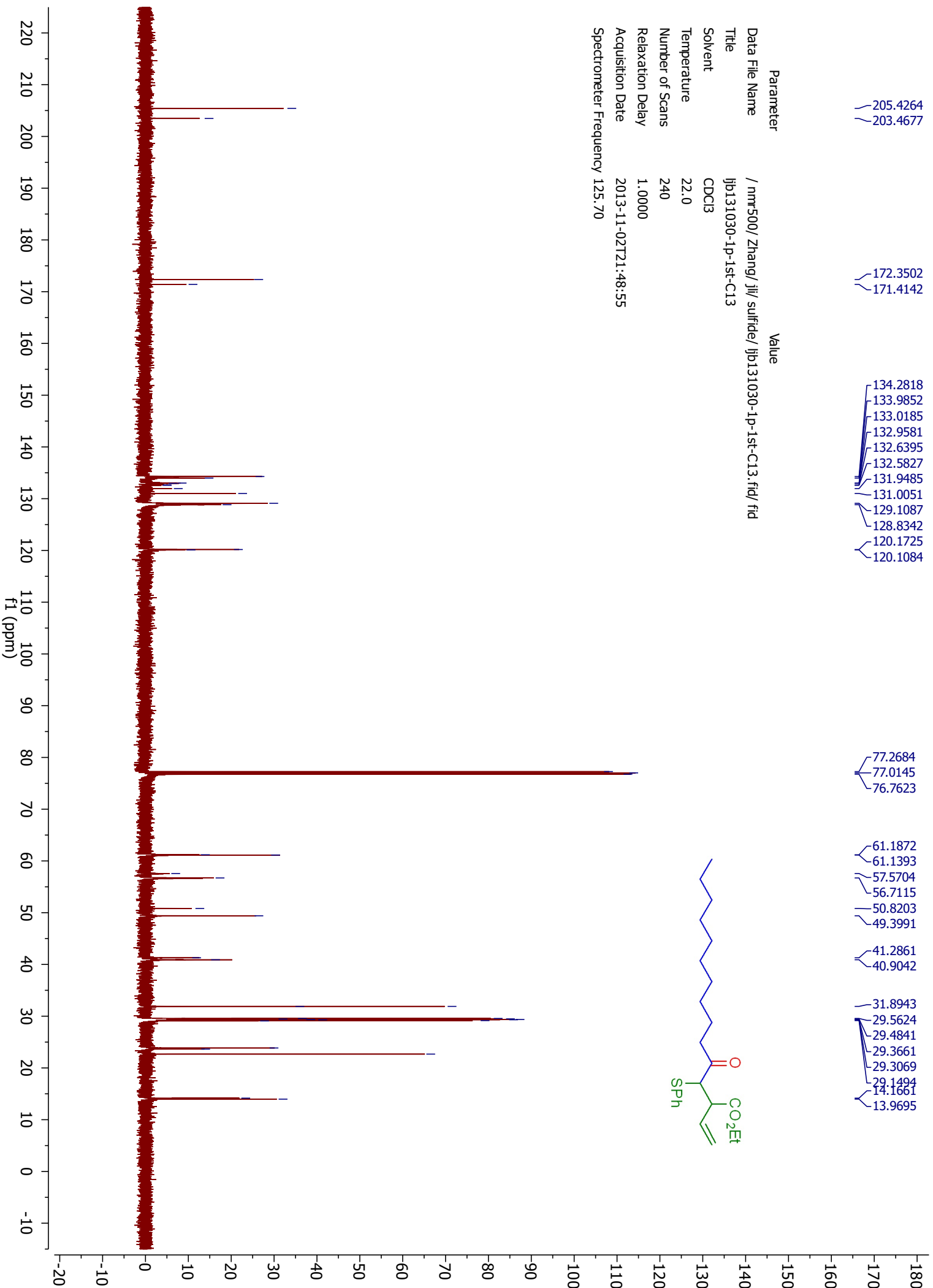
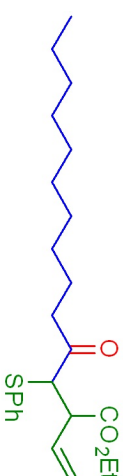
77.2684
77.0145
76.7623

61.1872
61.1393
57.5704
56.7115
50.8203
49.3991

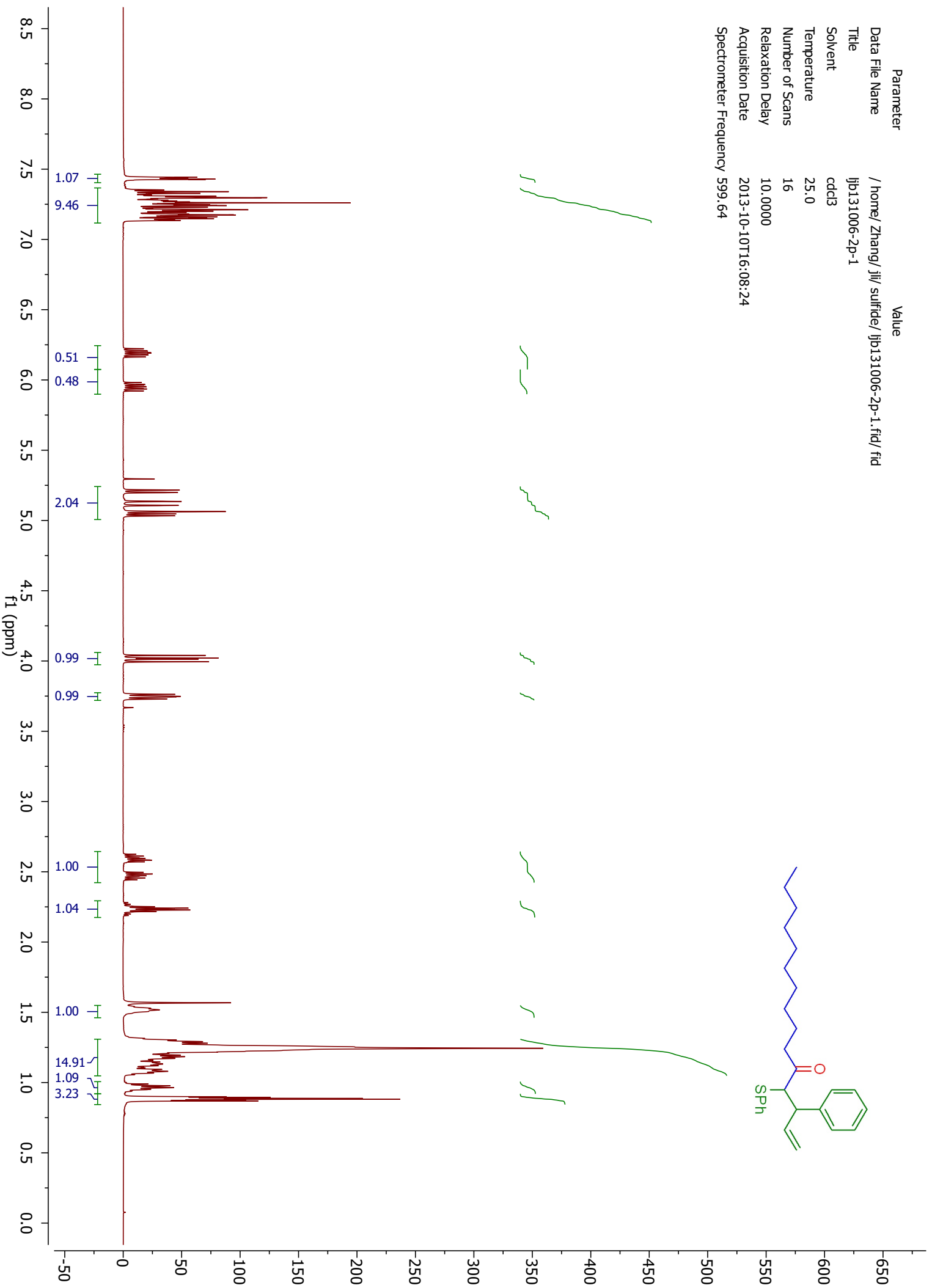
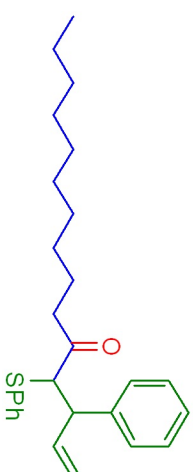
41.2861
40.9042

31.8943
29.5624
29.4841
29.3661
29.3069
29.1494
14.1661
13.9695

Parameter	Value
Data File Name	/nmr500/Zhang/jil/sulfide/jlb131030-1p-1st-C13.fid/ fid
Title	jlb131030-1p-1st-C13
Solvent	CDCl3
Temperature	22.0
Number of Scans	240
Relaxation Delay	1.0000
Acquisition Date	2013-11-02T21:48:55
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/home/Zhang/jli/sulfide/jlb131006-2p-1.fid/ fid
Title	jlb131006-2p-1
Solvent	cdcl3
Temperature	25.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-10T16:08:24
Spectrometer Frequency	599.64



206.4693
205.7170

141.2850
140.3372
138.6737
138.4405
133.4424
133.1010
129.2605
129.0966
128.9180
128.8241
128.6156
128.3646
128.2501
128.1364
127.3492
127.2363
117.6257
117.2642

77.4417
77.2300
77.0178

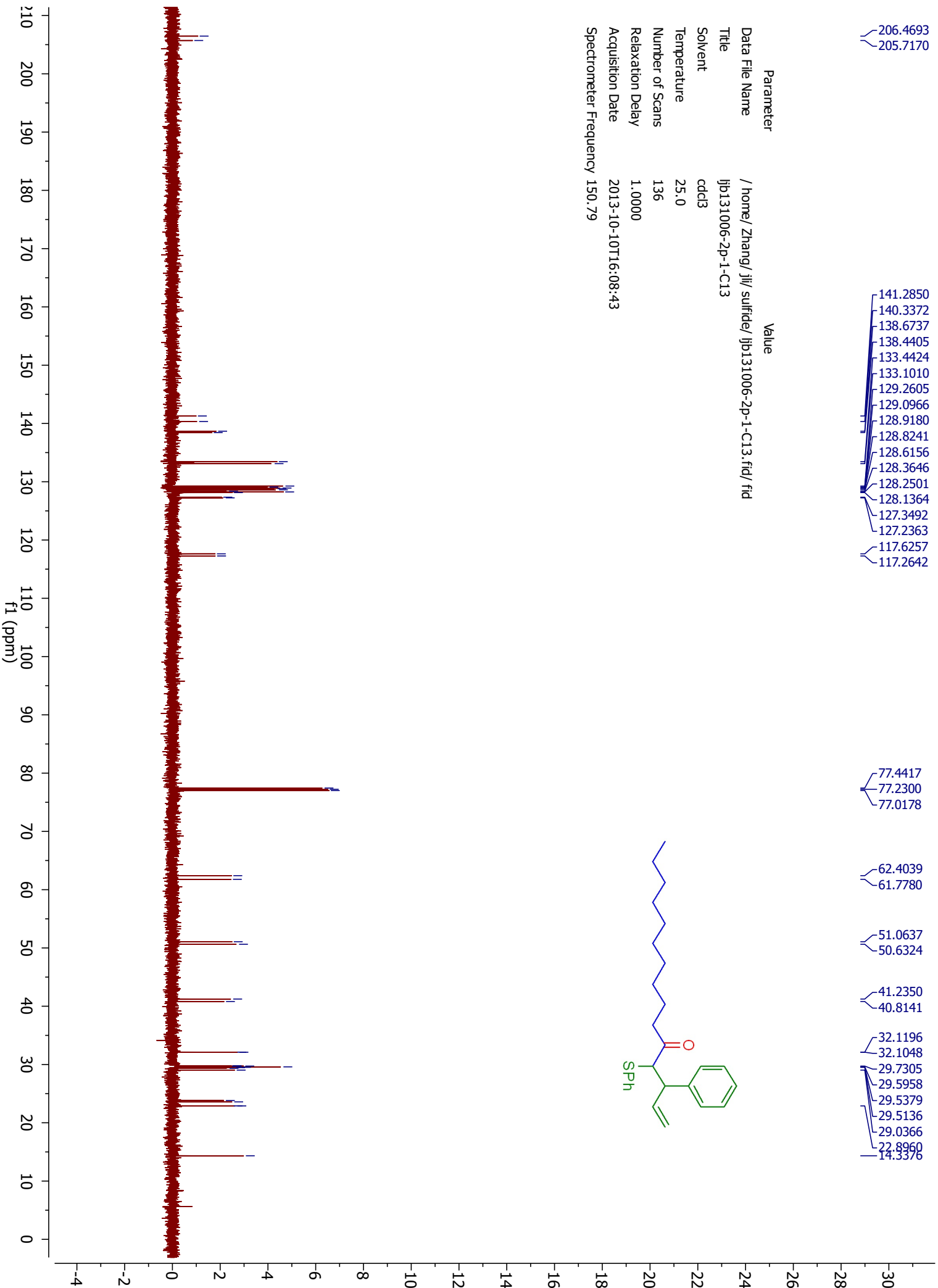
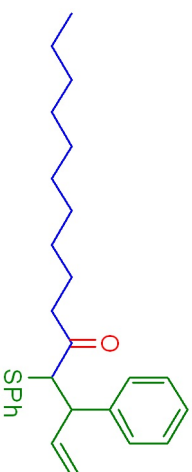
62.4039
61.7780

51.0637
50.6324

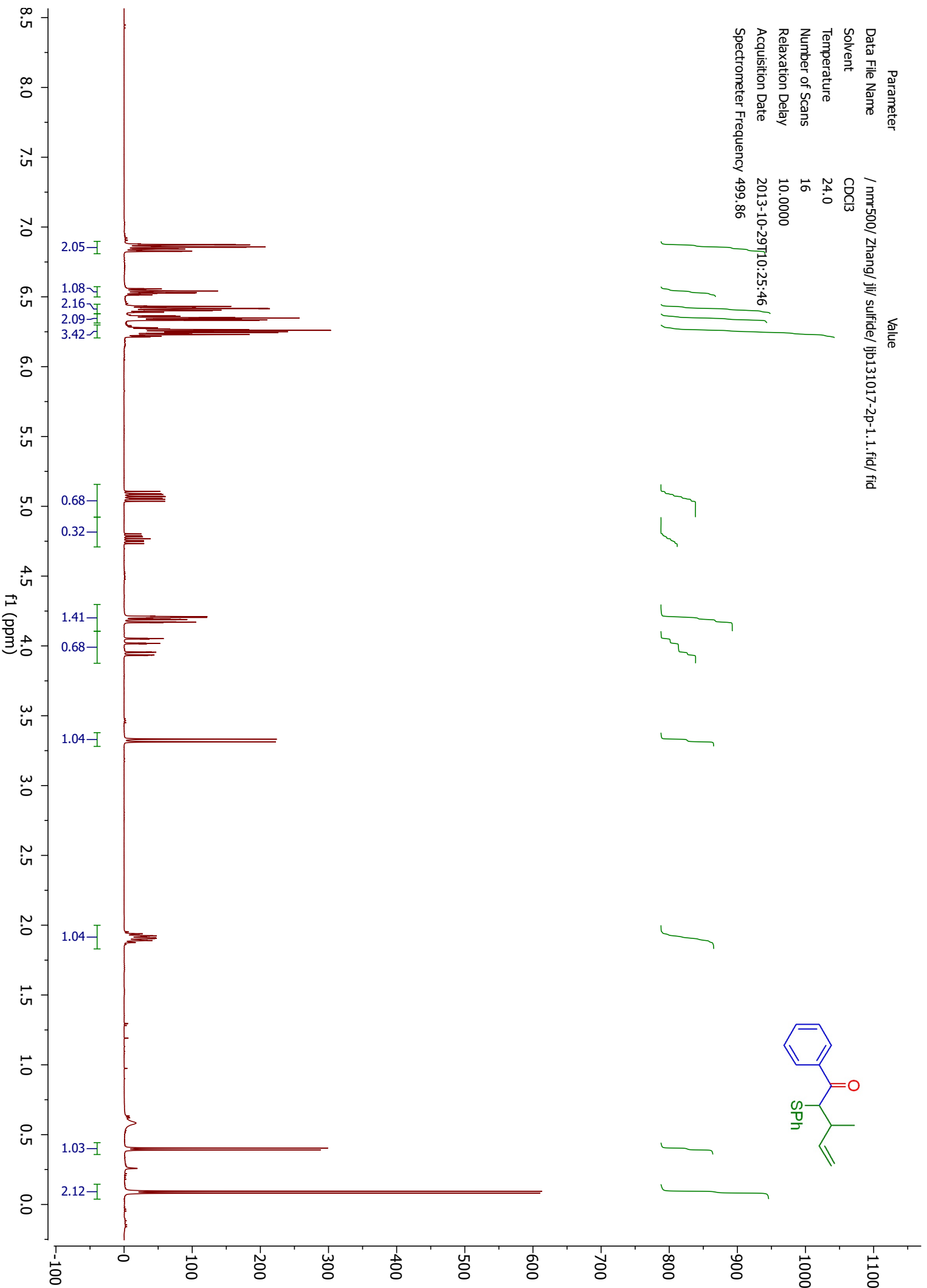
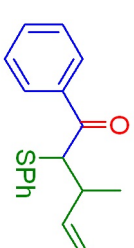
41.2350
40.8141

32.1196
32.1048
29.7305
29.5958
29.5379
29.5136
29.0366
22.8960
14.3376

Parameter	Value
Data File Name	/home/Zhang/jli/sulfide/jlb131006-2p-1-C13.fid/ fid
Title	jlb131006-2p-1-C13
Solvent	cdcl3
Temperature	25.0
Number of Scans	136
Relaxation Delay	1.0000
Acquisition Date	2013-10-10T16:08:43
Spectrometer Frequency	150.79



Parameter	Value
Data File Name	/nmr500/ Zhang/ jll/ sulfide/ jlb131017-2p-1.1.fid/ fid
Solvent	CDCl3
Temperature	24.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-10-29T10:25:46
Spectrometer Frequency	499.86



195.8696

140.9280
140.5412
137.0030
134.4885
134.2433
133.2176
133.1019
129.1740
129.0675
128.7627
128.7288
128.6537
128.6121
128.5807
116.0370
115.9034

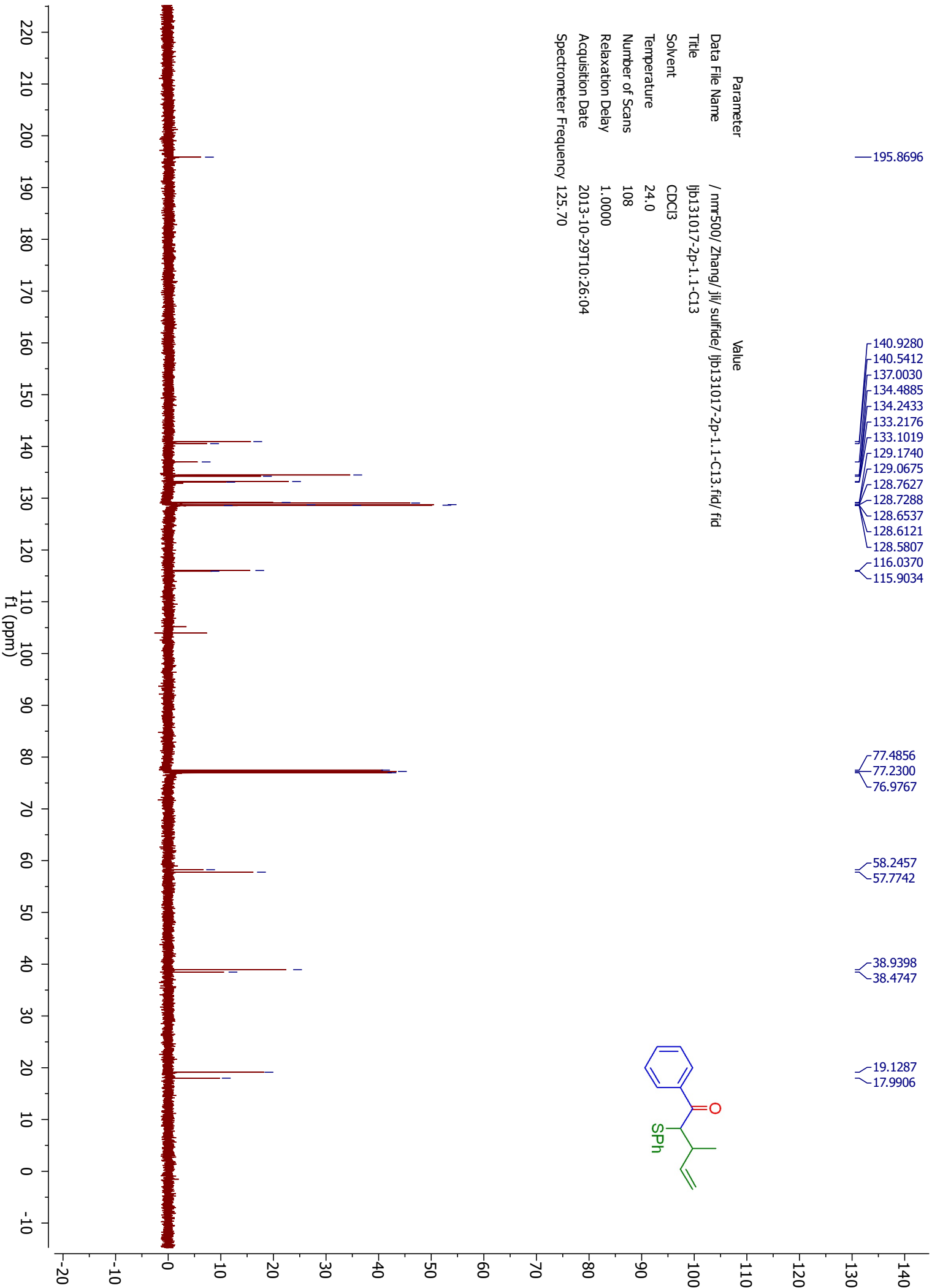
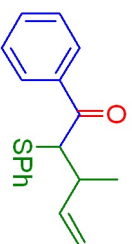
77.4856
77.2300
76.9767

58.2457
57.7742

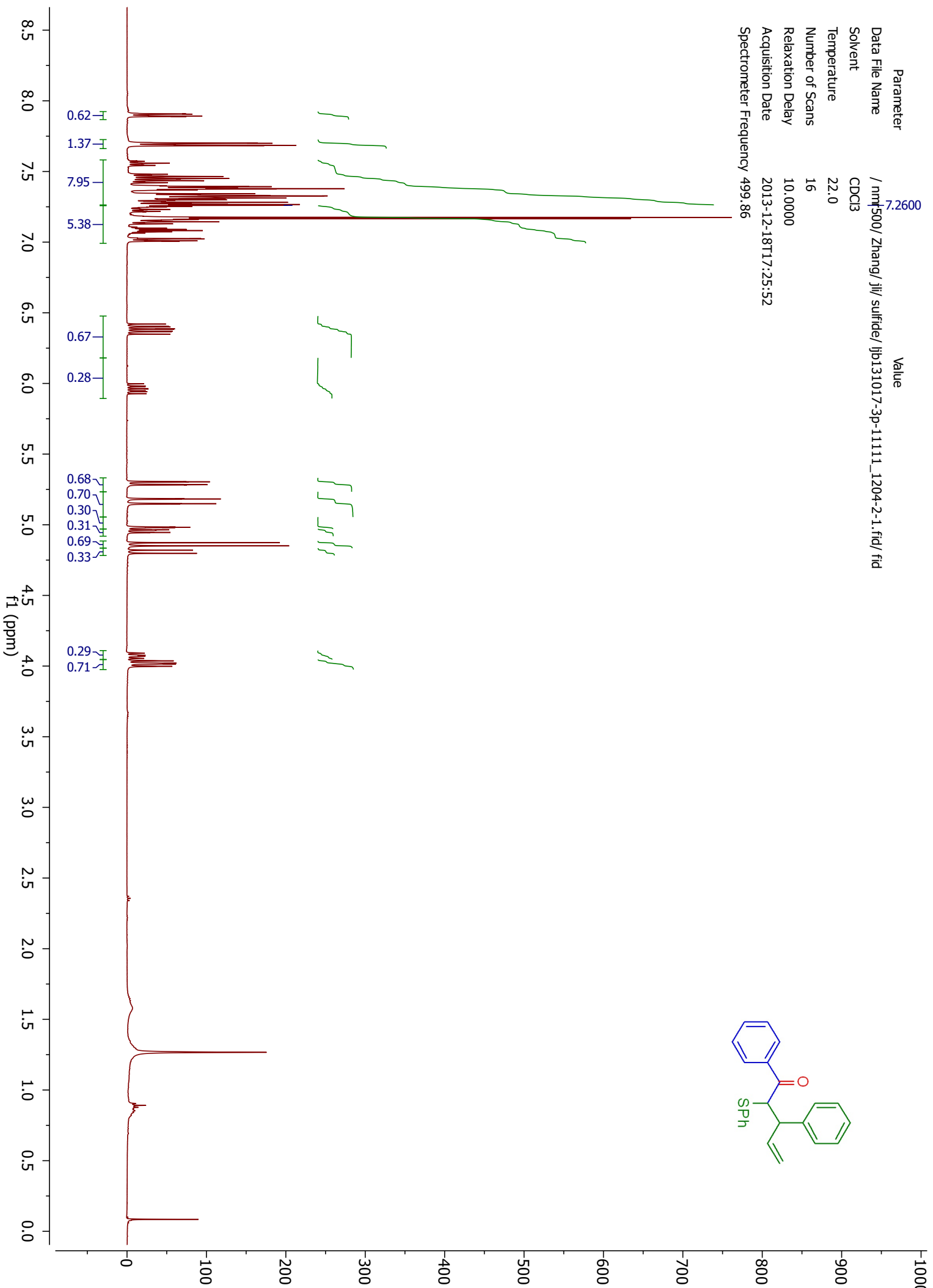
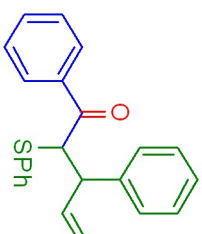
38.9398
38.4747

19.1287
17.9906

Parameter	Value
Data File Name	/nmr500/ Zhang/ jiv/ sulfide/ jpb131017-2p-1.1-C13.fid/ fid
Title	jpb131017-2p-1.1-C13
Solvent	CDCl3
Temperature	24.0
Number of Scans	108
Relaxation Delay	1.0000
Acquisition Date	2013-10-29T10:26:04
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr500/Zhang/jll/sulfide/jlb131017-3p-11111_1204-2-1.fid/ fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-12-18T17:25:52
Spectrometer Frequency	499.86



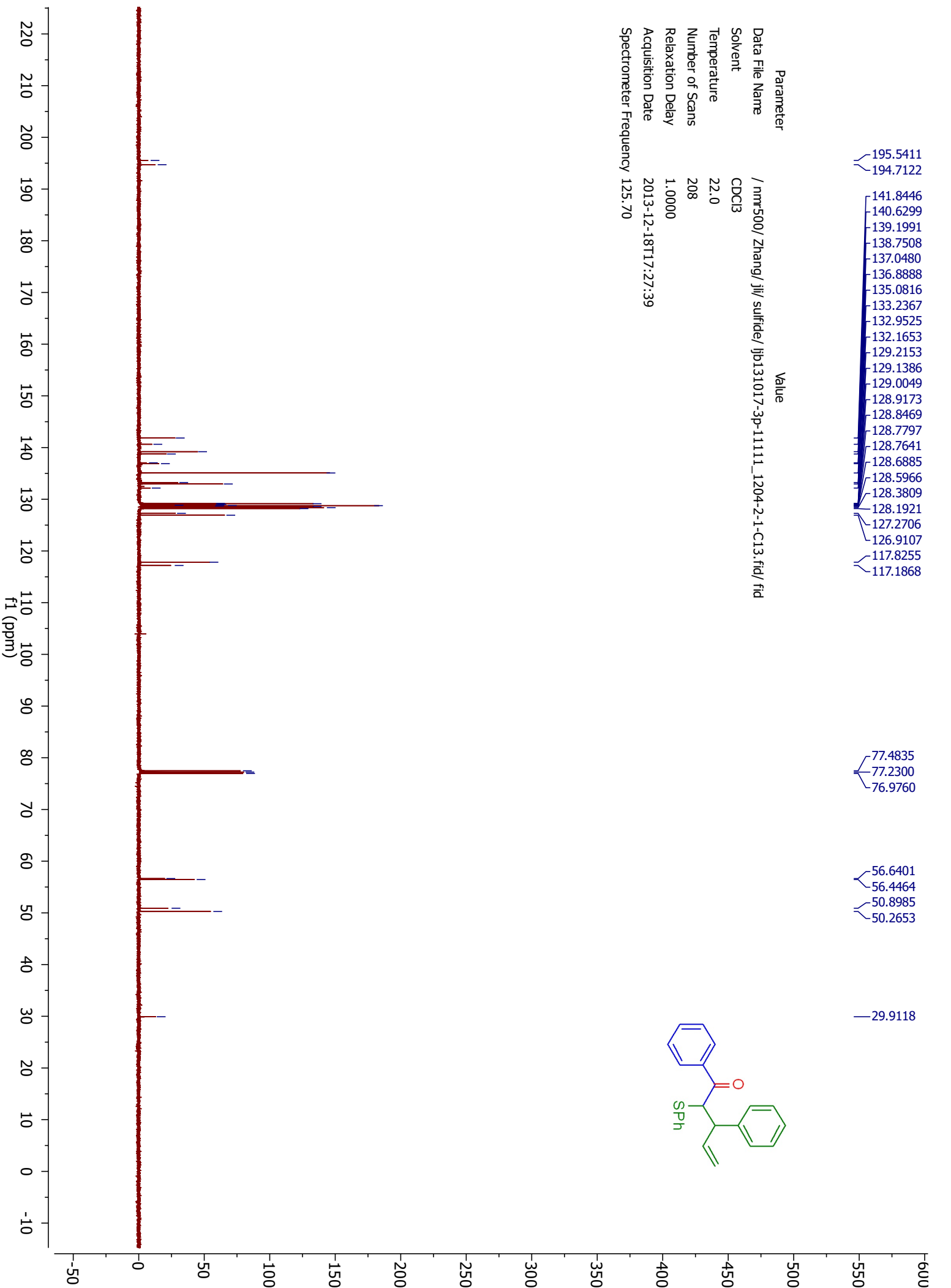
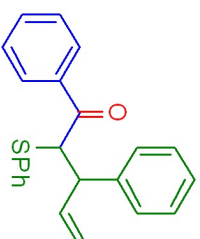
195.5411
194.7122
141.8446
140.6299
139.1991
138.7508
137.0480
136.8888
135.0816
133.2367
132.9525
132.1653
129.2153
129.1386
129.0049
128.9173
128.8469
128.7797
128.7641
128.6885
128.5966
128.3809
128.1921
127.2706
126.9107
117.8255
117.1868

77.4835
77.2300
76.9760

56.6401
56.4464
50.8985
50.2653

29.9118

Parameter	Value
Data File Name	/nmr500/Zhang/jil/sulfide/jlb131017-3p-11111_1204-2-1-C13.fid/fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	208
Relaxation Delay	1.0000
Acquisition Date	2013-12-18T17:27:39
Spectrometer Frequency	125.70

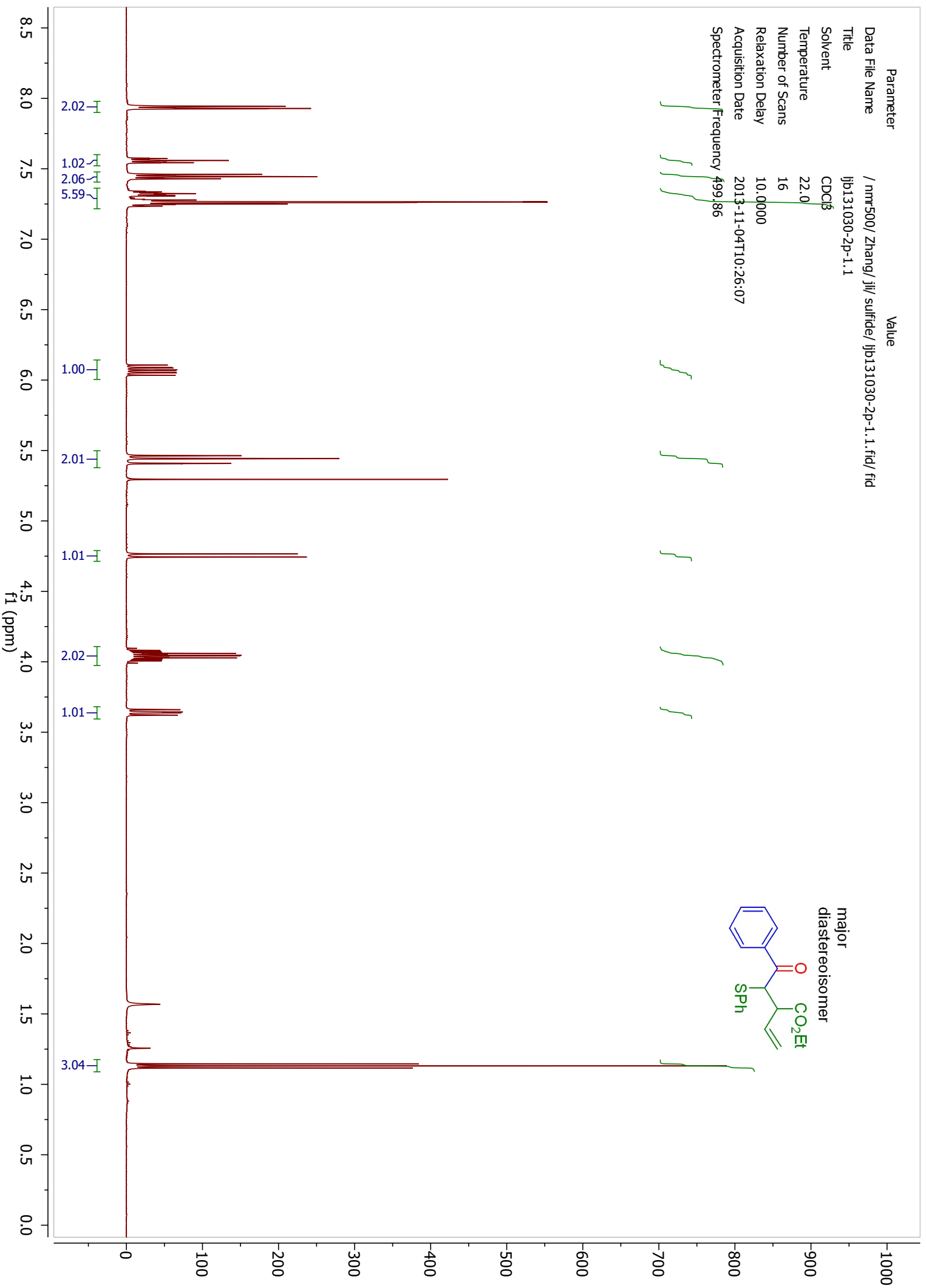
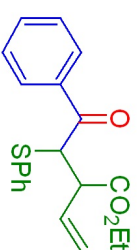


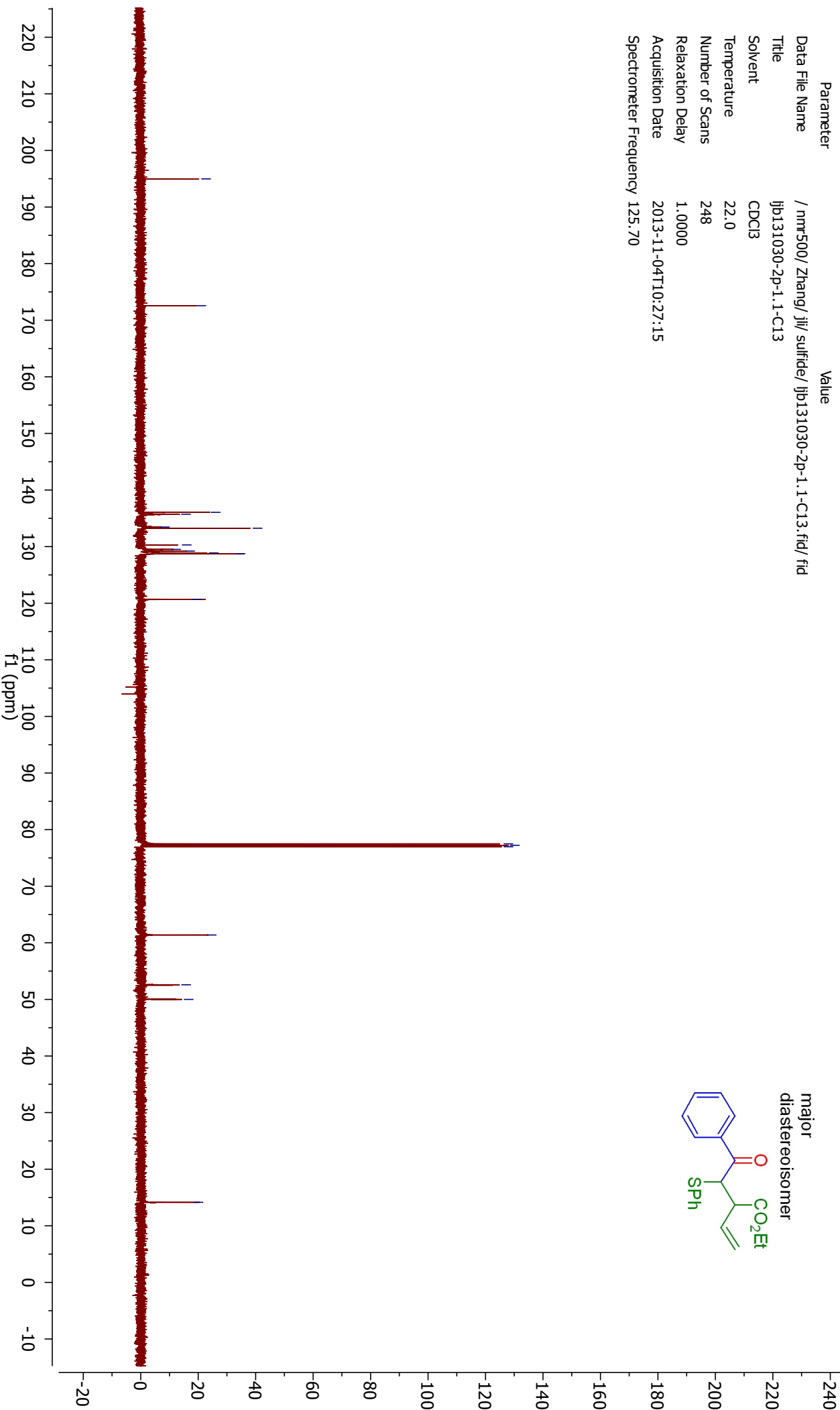
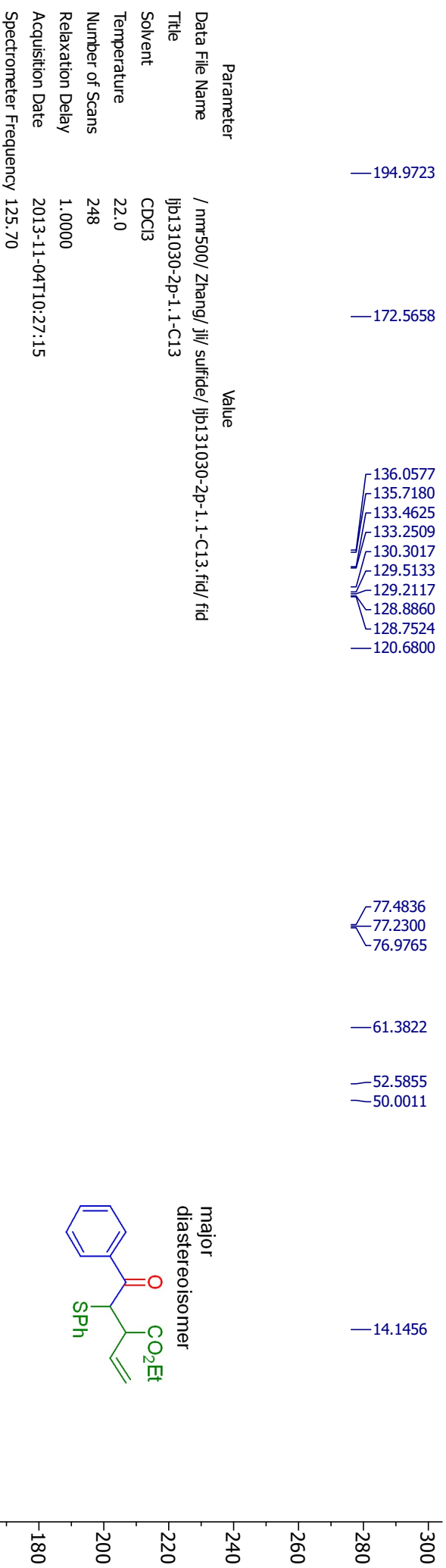
Parameter

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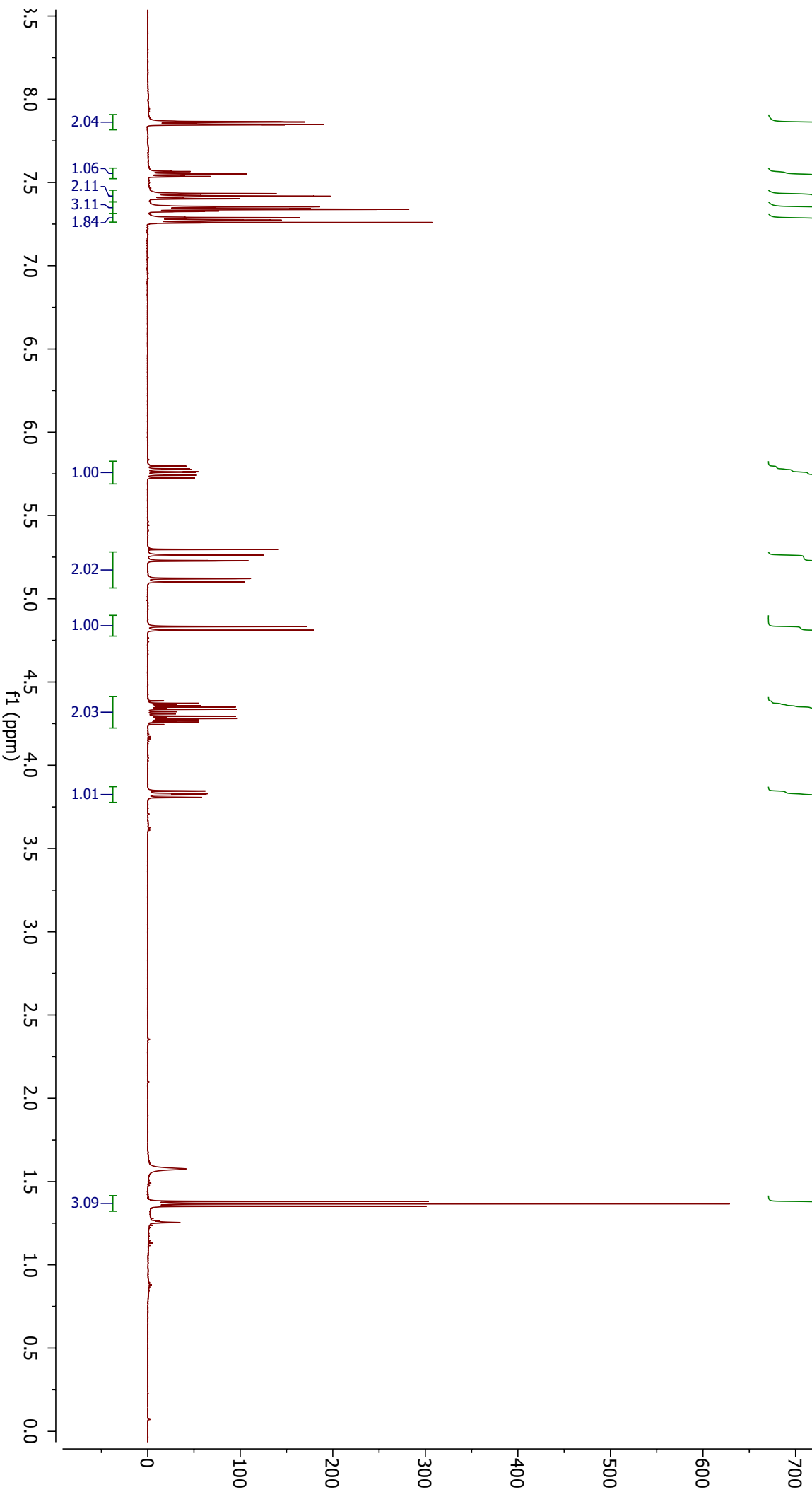
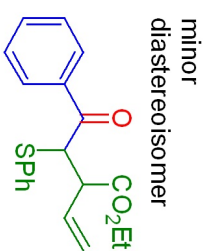
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Title	jlb131030-2p-1.1
Solvent	CDCl ₃
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-04T10:26:07
Spectrometer Frequency	499.86

major
diastereoisomer

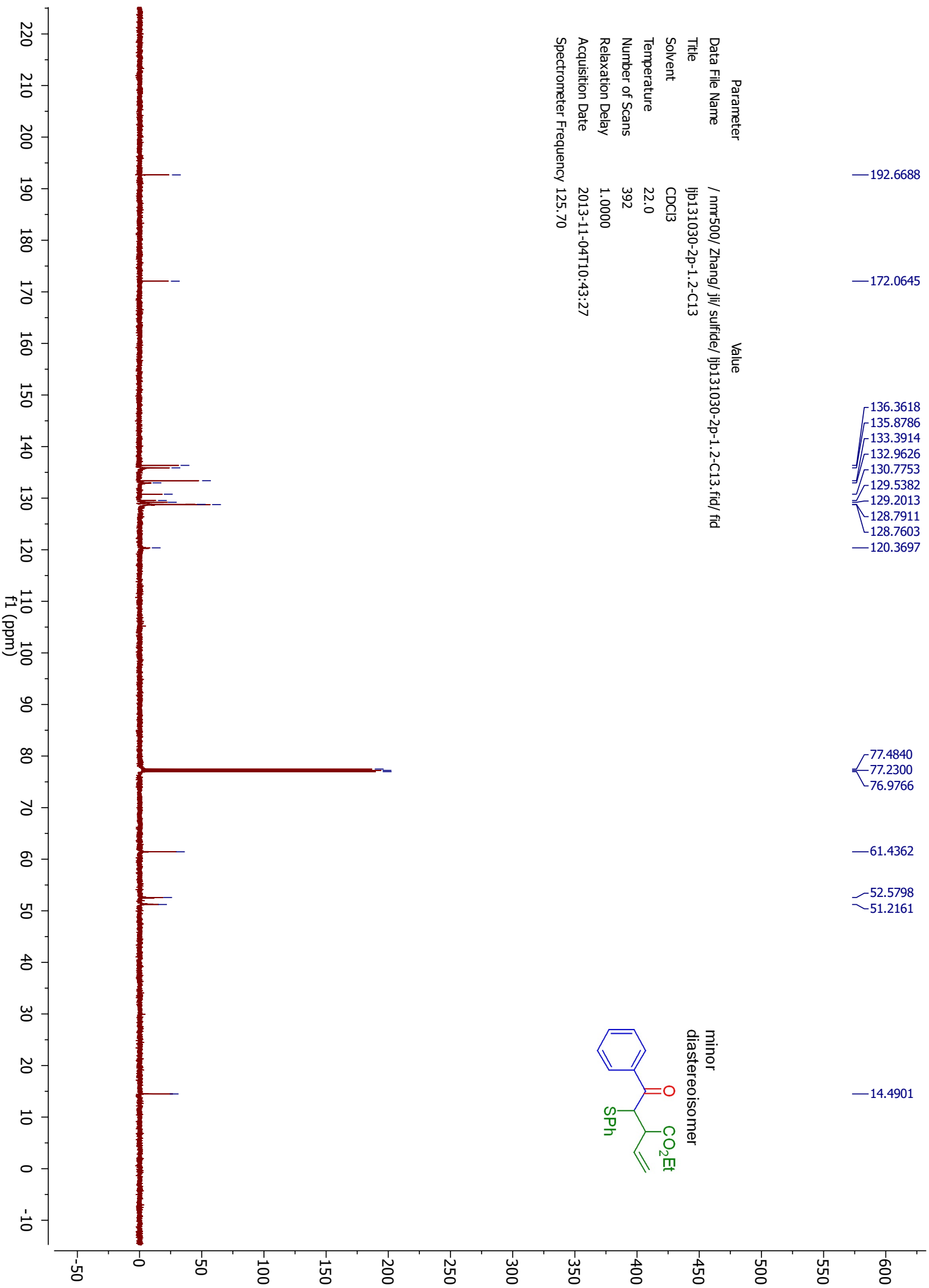




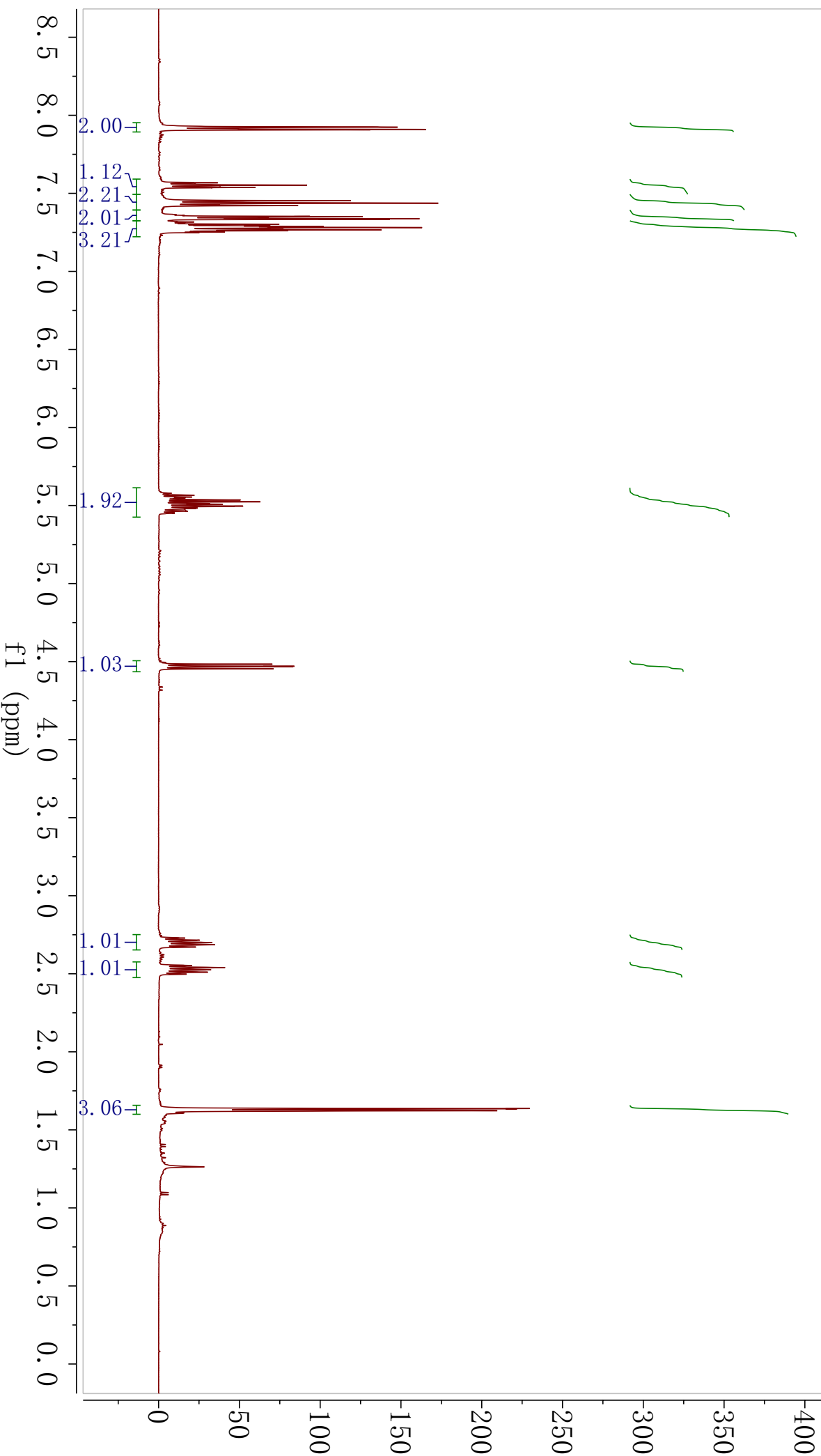
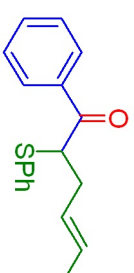
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Title	jlb131030-2p-1.2
Solvent	CDCl ₃
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-04T10:43:05
Spectrometer Frequency	499.86

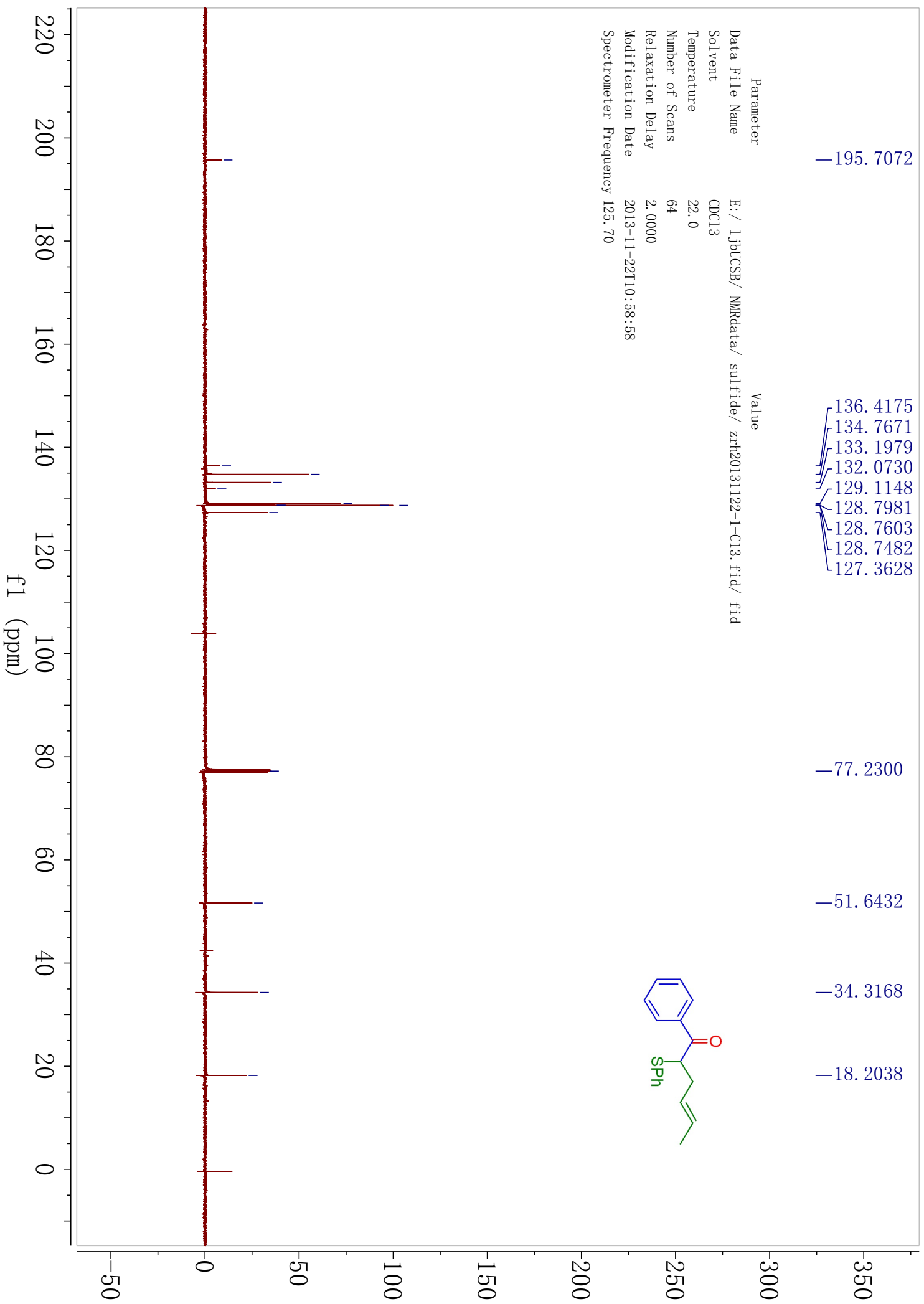


Parameter	Value
Data File Name	/nmr500/Zhang/jil/sulfide/jlb131030-2p-1.2-C13.fid
Title	jlb131030-2p-1.2-C13
Solvent	CDCl ₃
Temperature	22.0
Number of Scans	392
Relaxation Delay	1.0000
Acquisition Date	2013-11-04T10:43:27
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	E:/ 1jubCSB/ NMRdata/ sulfide/ zrh20131122-1.fid/ fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	8
Relaxation Delay	5.0000
Modification Date	2013-11-22T10:54:18
Spectrometer Frequency	499.86

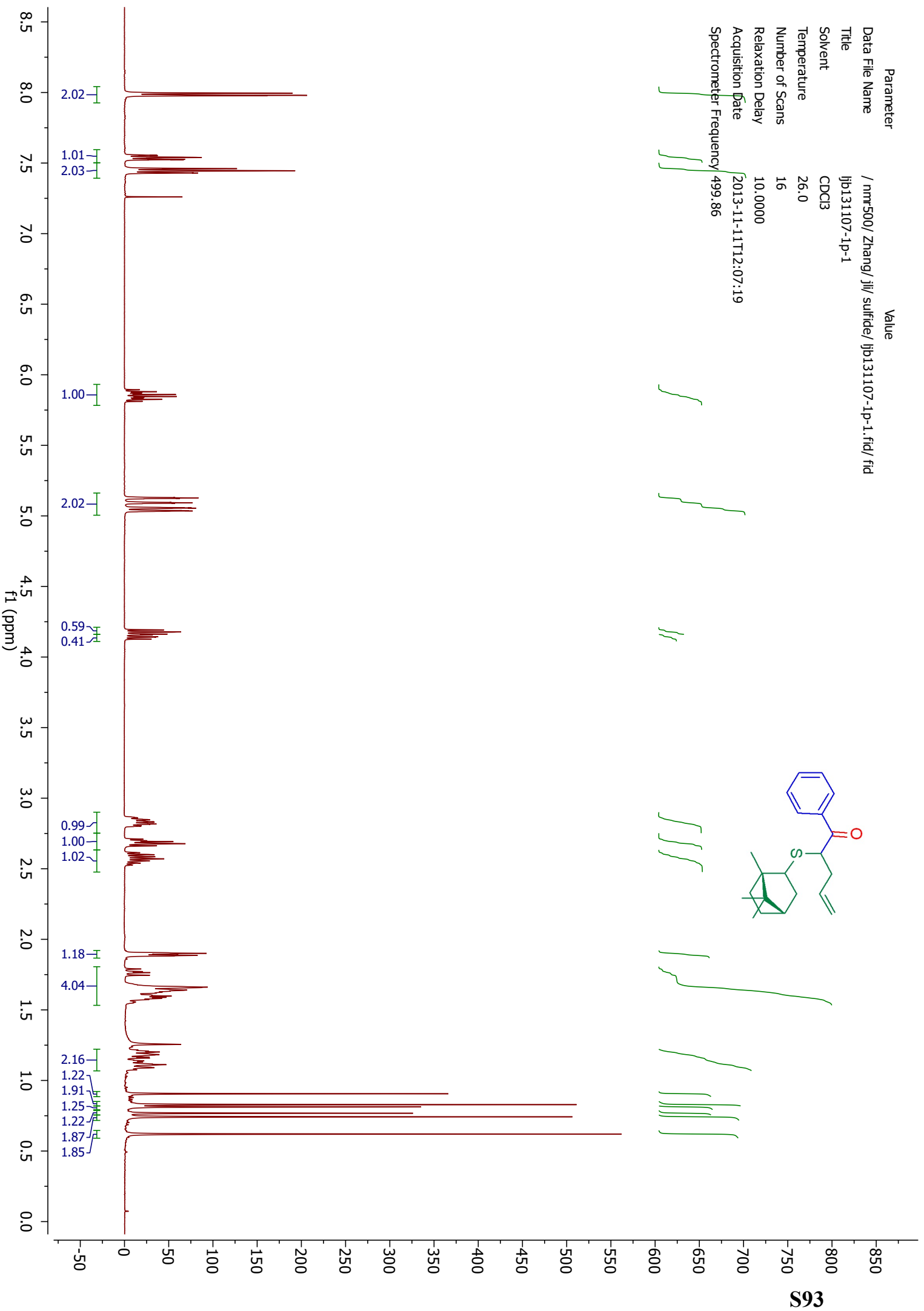
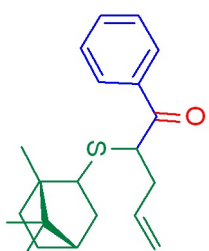




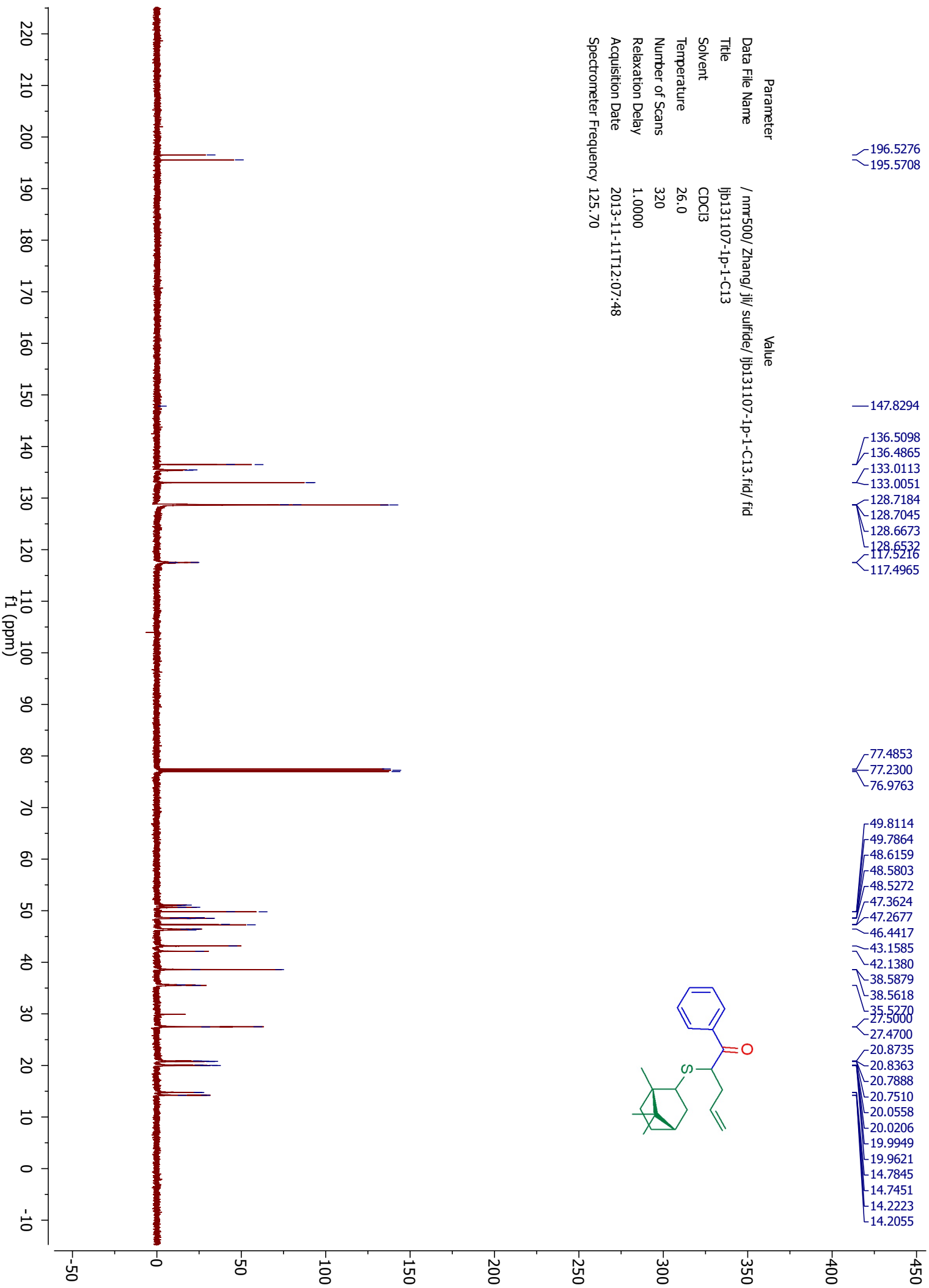
Parameter

Value

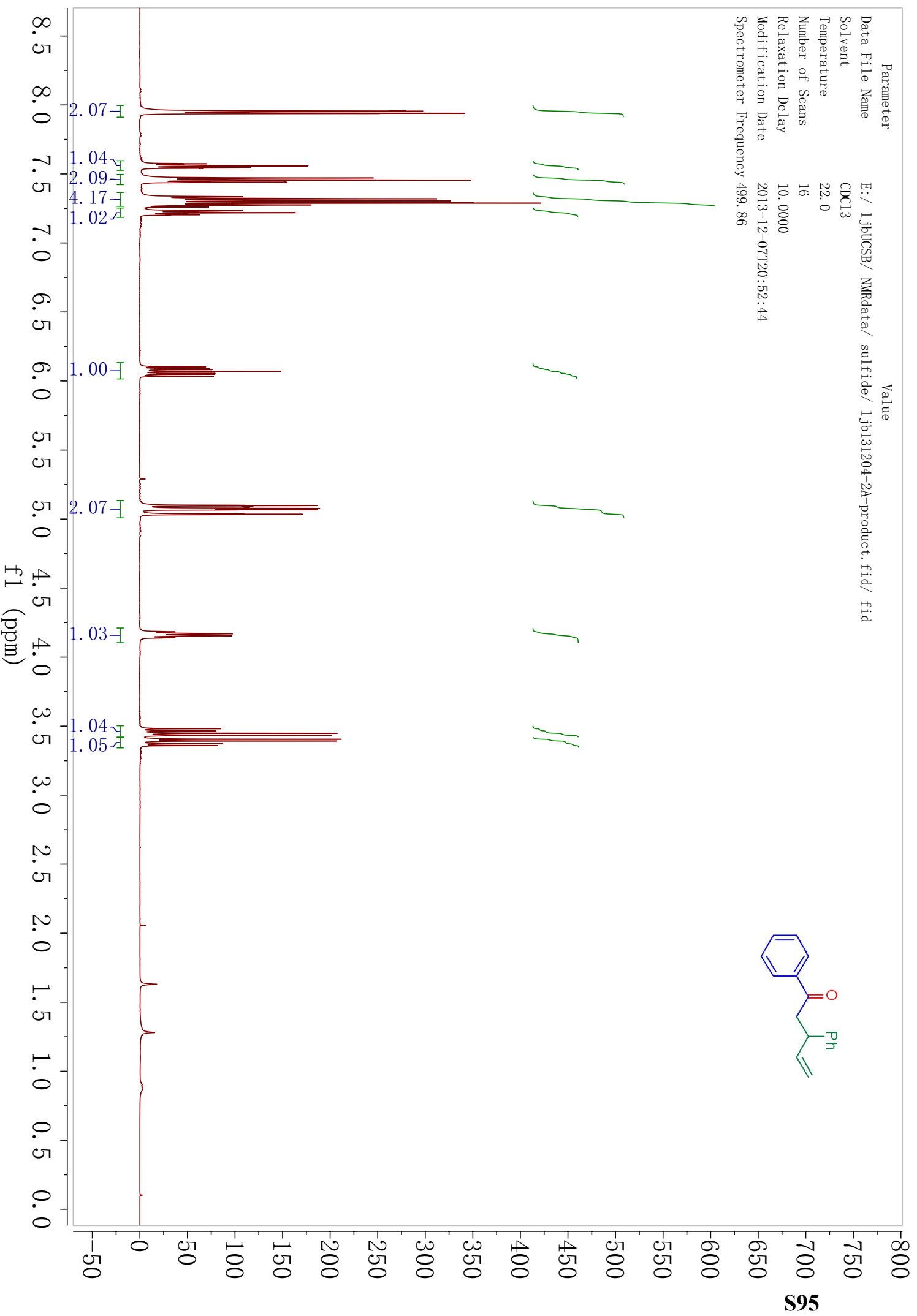
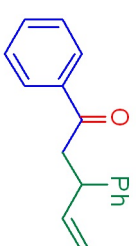
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Title	jlb131107-1p-1
Solvent	CDCl3
Temperature	26.0
Number of Scans	16
Relaxation Delay	10.0000
Acquisition Date	2013-11-11T12:07:19
Spectrometer Frequency	499.86



Parameter	Value
Data File Name	/nmr500/Zhang/jil/sulfide/jlb131107-1p-1-C13.fid/ fid
Title	jlb131107-1p-1-C13
Solvent	CDCl3
Temperature	26.0
Number of Scans	320
Relaxation Delay	1.0000
Acquisition Date	2013-11-11T12:07:48
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	E:/ 1jblCSB/ NMRdata/ sulfide/ 1jbl31204-2A-product.fid/ fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	16
Relaxation Delay	10.0000
Modification Date	2013-12-07T20:52:44
Spectrometer Frequency	499.86



— 198.4285

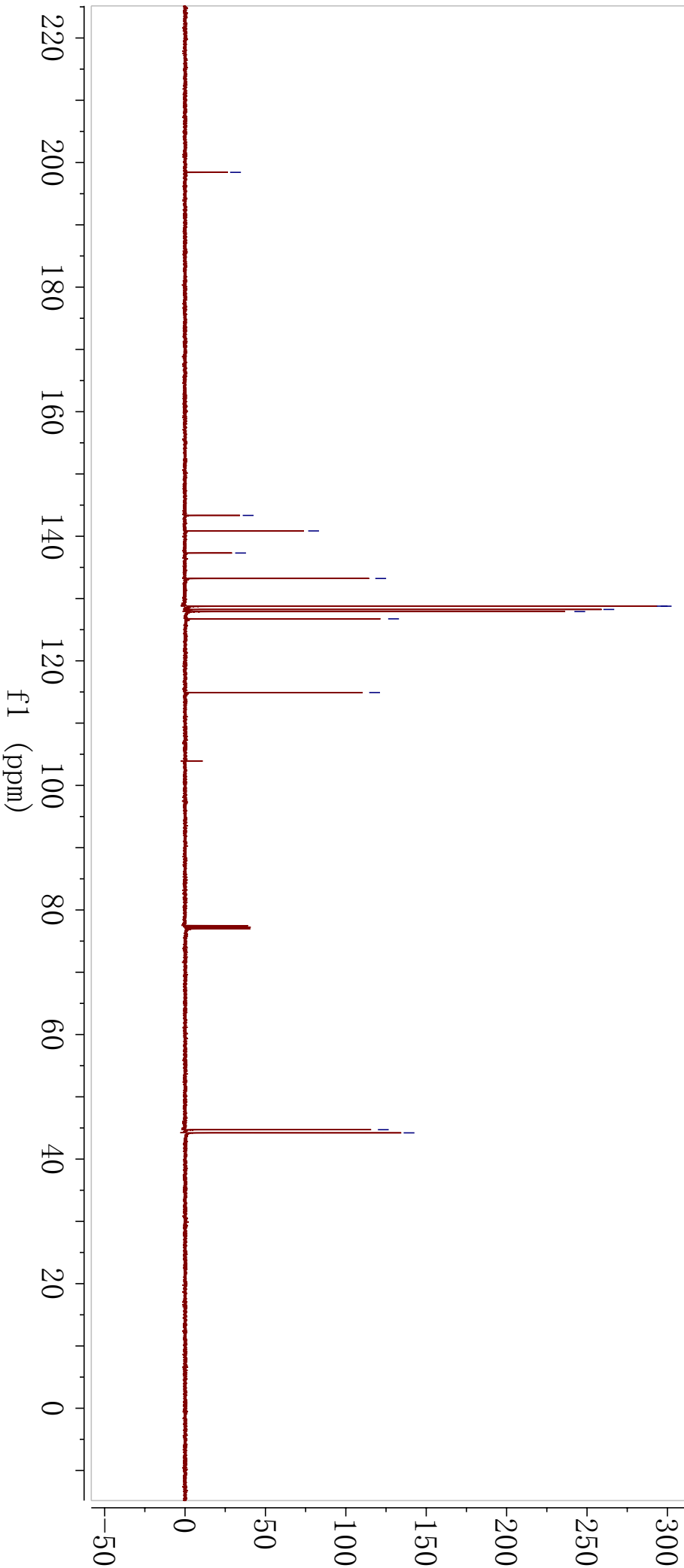
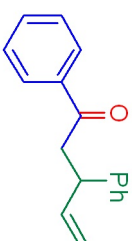
143.3393
140.8567
137.3086
133.2202
128.7772
128.7698
128.2483
127.9072
126.7405
— 114.9074

44.7193
44.2116

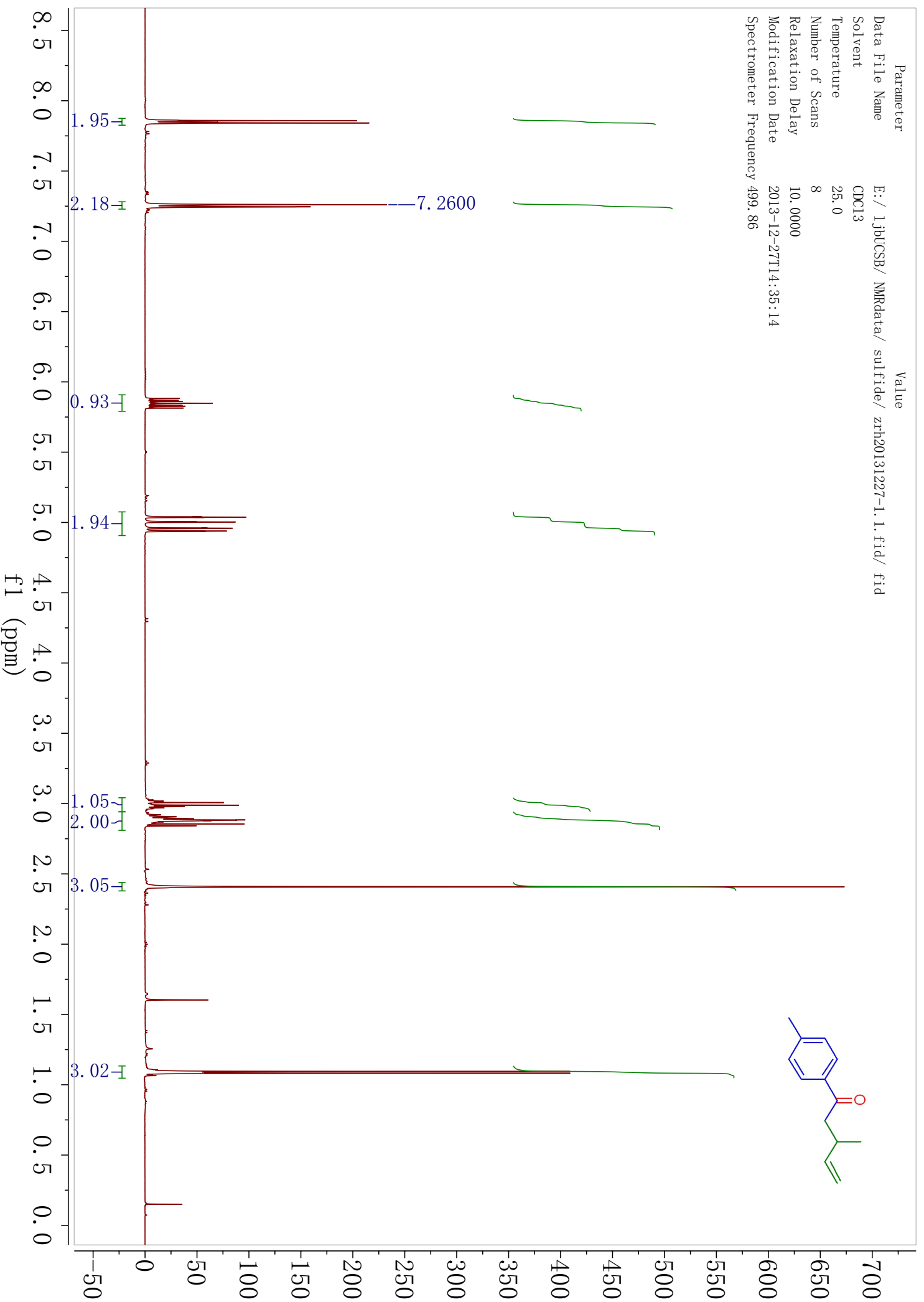
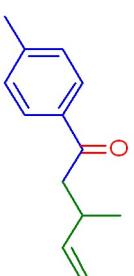
Parameter

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Data File Name E:/ 1jblCSB/ NMRdata/ sulfide/ 1jbl31204-2A-product-C13.fid/ fid
Solvent CDCl₃
Temperature 22.0
Number of Scans 120
Relaxation Delay 1.0000
Modification Date 2013-12-07T20:57:40
Spectrometer Frequency 125.70



Parameter	Value
Data File Name	E:/ 1jubCSB/ NMRdata/ sulfide/ zrh20131227-1.1.fid/ fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	8
Relaxation Delay	10.0000
Modification Date	2013-12-27T14:35:14
Spectrometer Frequency	499.86



— 199.2231

143.9124
143.3698
135.0573
129.4514
128.4511

— 113.1480

77.4839
77.2300
76.9757

— 45.2432

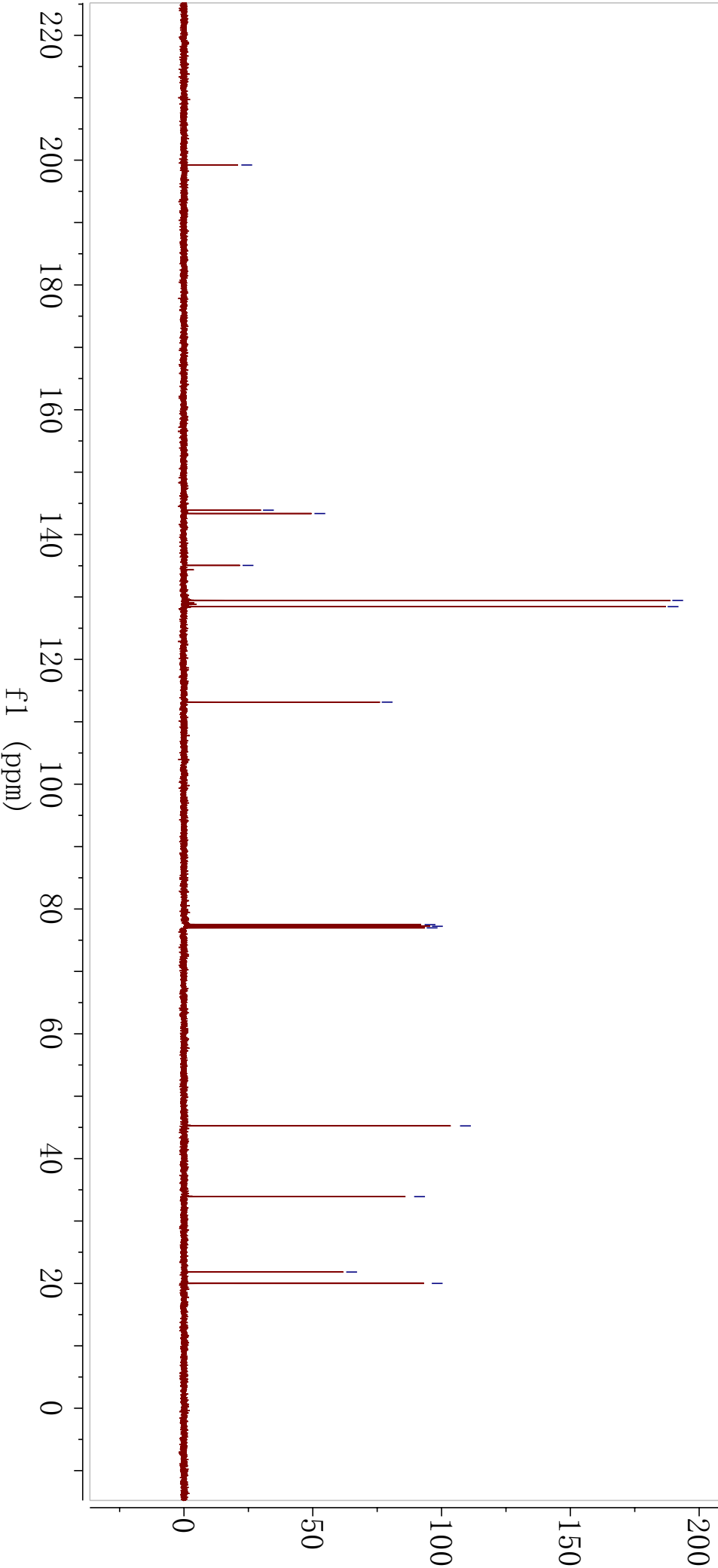
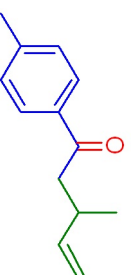
— 33.9099

21.8266
20.0026

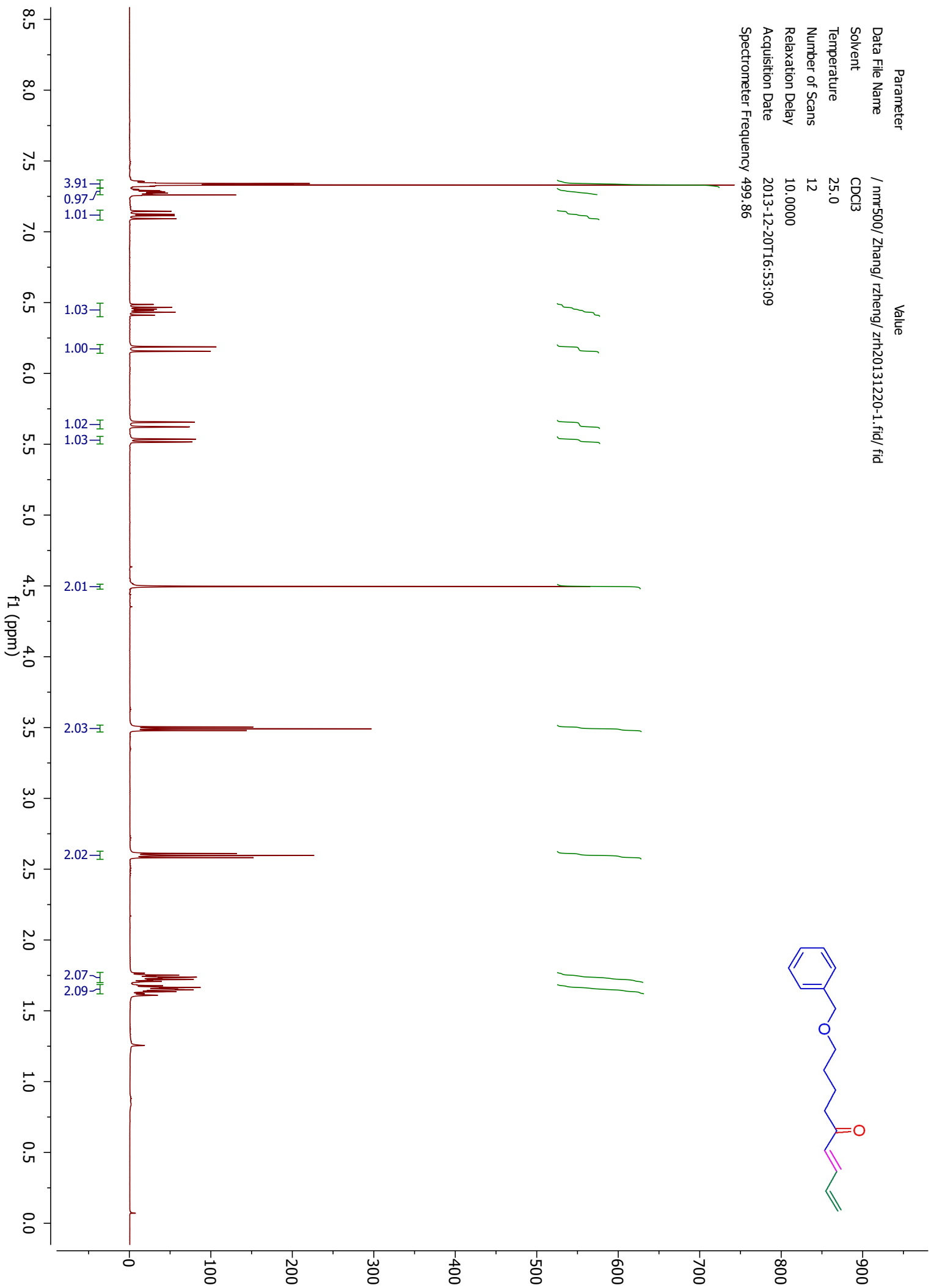
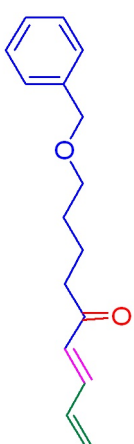
Parameter

Value

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Solvent CDCl3
Temperature 25.0
Number of Scans 172
Relaxation Delay 2.0000
Modification Date 2013-12-27T14:45:26
Spectrometer Frequency 125.70



Parameter	Value
Data File Name	/nmr500/Zhang/rzheng/zrh20131220-1.fid/ fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	12
Relaxation Delay	10.0000
Acquisition Date	2013-12-20T16:53:09
Spectrometer Frequency	499.86



200.7681

142.5712
138.7463
135.4893
130.5736
128.5659
127.8476
127.7258
126.3848

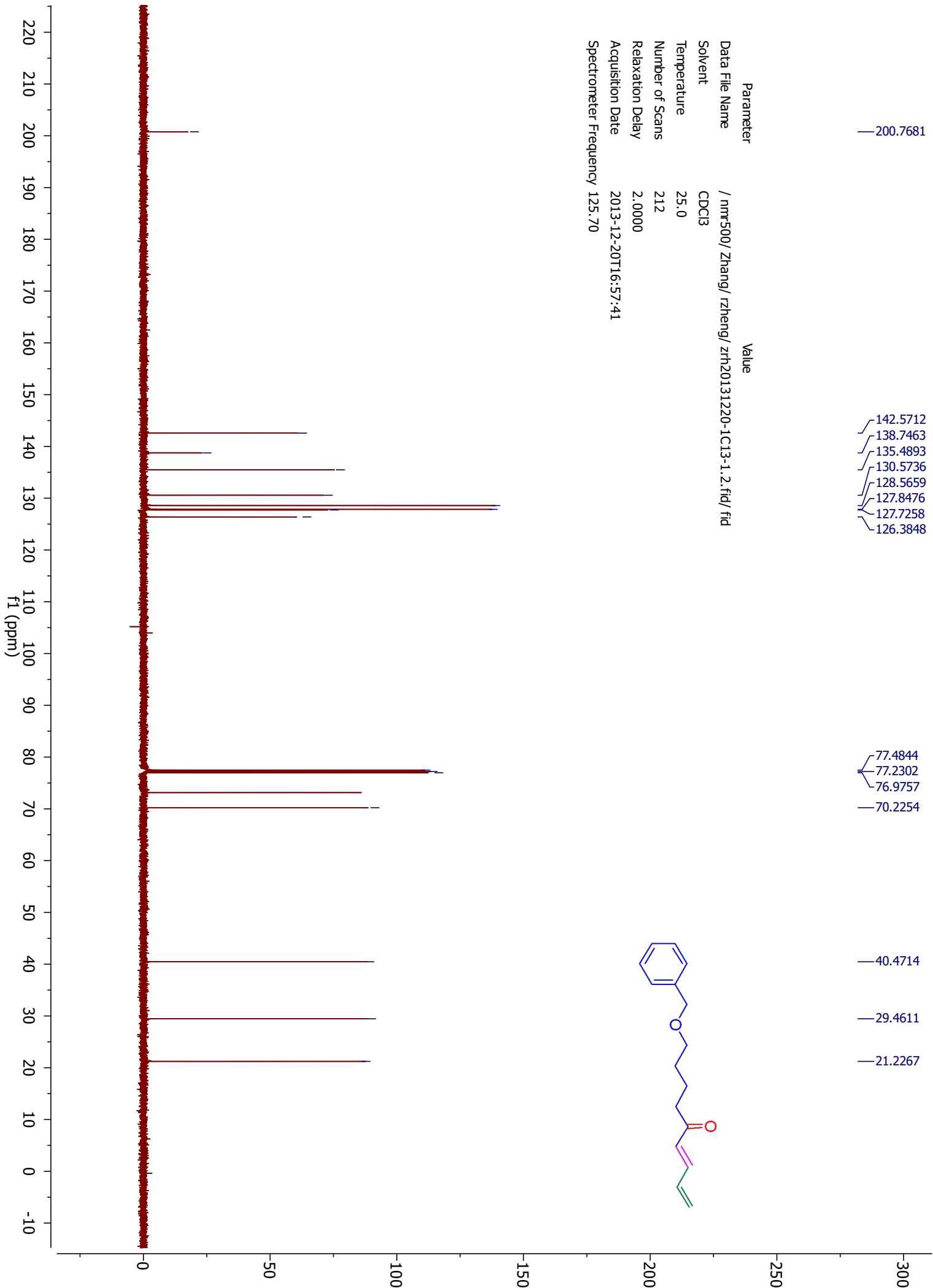
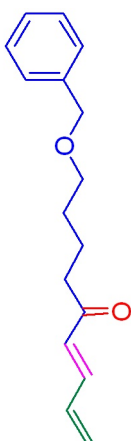
77.4844
77.2302
76.9757
70.2254

40.4714

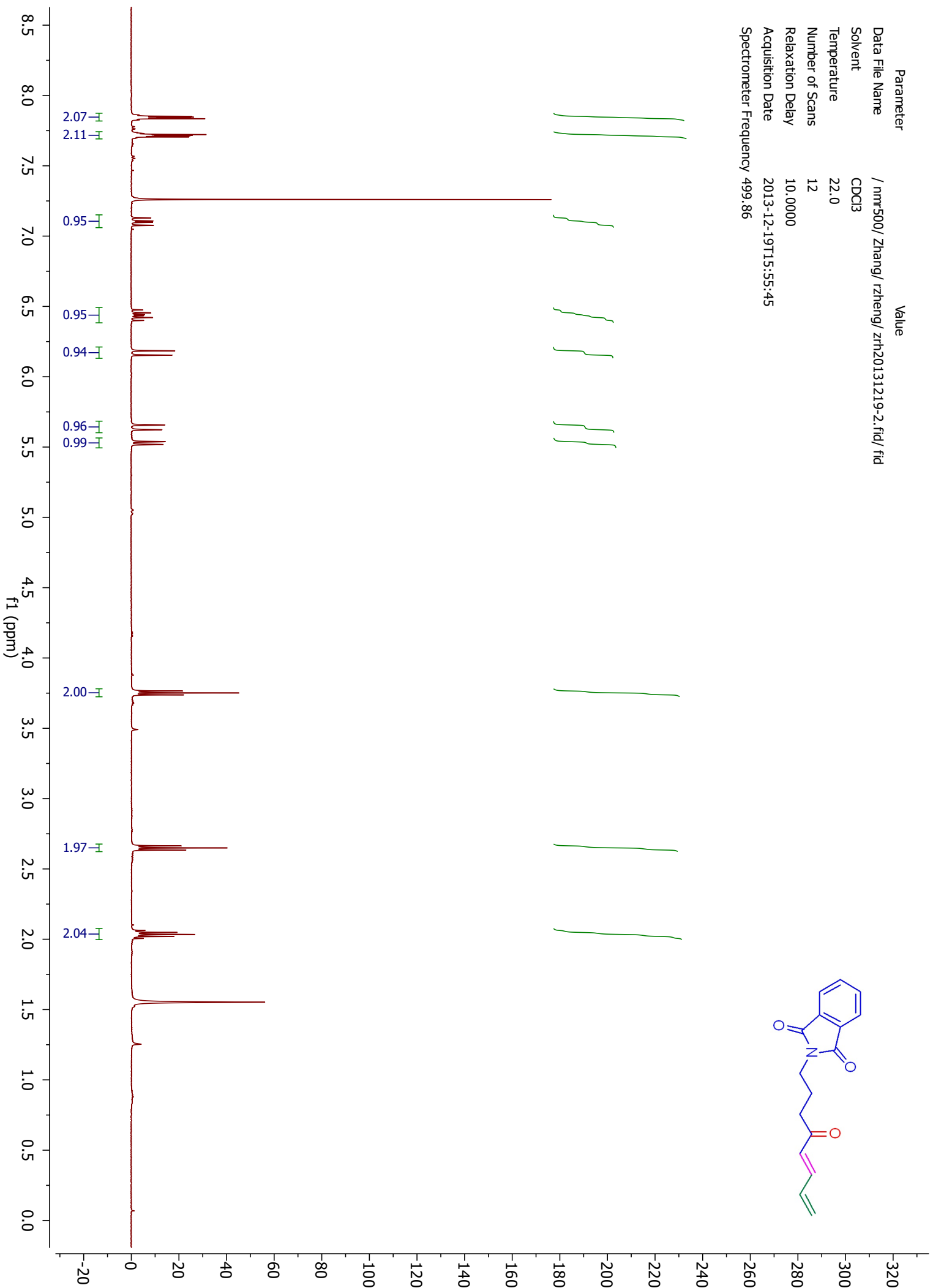
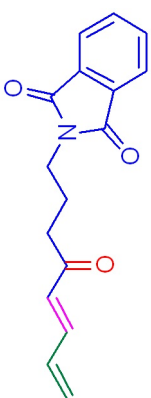
29.4611

21.2267

Parameter	Value
Data File Name	/nmr500/Zhang/rzheng/zrh20131220-1C13-1.2.fid/fid
Solvent	CDCl3
Temperature	25.0
Number of Scans	212
Relaxation Delay	2.0000
Acquisition Date	2013-12-20T16:57:41
Spectrometer Frequency	125.70



Parameter	Value
Data File Name	/nmr500/Zhang/rzheng/zrh20131219-2.fid/fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	12
Relaxation Delay	10.0000
Acquisition Date	2013-12-19T15:55:45
Spectrometer Frequency	499.86



199.2395

168.6251

142.7831

135.4096

134.1563

132.3007

130.2481

126.6408

123.4642

77.4833

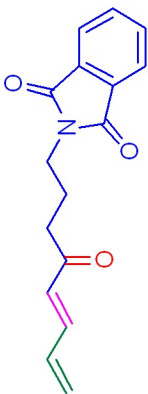
77.2300

76.9758

37.9738

37.6347

23.2591



Parameter	Value
Data File Name	/nmr500/Zhang/rzheng/zrh20131219-2-1C13-1.fid/ fid
Solvent	CDCl3
Temperature	22.0
Number of Scans	188
Relaxation Delay	2.0000
Acquisition Date	2013-12-19T17:19:20
Spectrometer Frequency	125.70

