Supplementary Information

Highly Conducting π-Conjugated Molecular Junctions Covalently Bonded to Gold Electrodes

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Contents:

Synthesis Information:

Chemicals: Solvents, inorganic salts, and organic reagents were purchased from commercial sources and used without further purification unless otherwise mentioned.

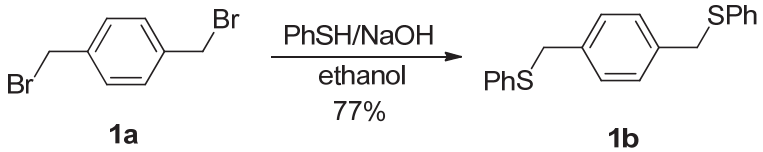
Chromatography: Merck pre-coated 0.25 mm silica plates containing a 254 nm fluorescence indicator were used for analytical thin-layer chromatography. Flash chromatography was performed on 230-400 mesh silica (SiliaFlash® P60) from Silicycle.

Preparative HPLC was run on a Waters 600 liquid chromatography system equipped with a WatersTM 600 pumping system and a Waters 2489 UV-Vis detector. Samples were collected manually. A Waters XBridgeTM C18 reverse phase preparative column (particle size 5 μm, 19x150 mm) was used as stationary phase.

Spectroscopy: NMR spectra were obtained on a Bruker DPX 300 or 400 MHz spectrometer. Spectra were analyzed with the MestreNova Software (Version 6.1).

CI-MS spectra were taken on a Nermag R-10-10 instrument.

Synthesis of $SnMe_3$ terminated compounds: The $SnMe_3$-terminated molecules used in this study were prepared by coupling the corresponding bisbromomethyl reagents (**1d, 1e, 1f, 2, 2a, 3, 4**) with trimethylstannyllithium,[1] which was generated by reacting hexamethyldistannane with n-BuLi. 1,4-bis(trimethylstannylmethyl)benzene (**P1**) was also prepared by converting 1,4-bis(bromomethyl)benzene (p-xylylene dibromide) to the dilithio[2] compound and reacting it with trimethylstannyl choride. The bis-bromomethyl reagents **2a**[3], **3**[4], **1d**[5], **1e**[6], and **1f**[7] were prepared according to known procedures in the literature.

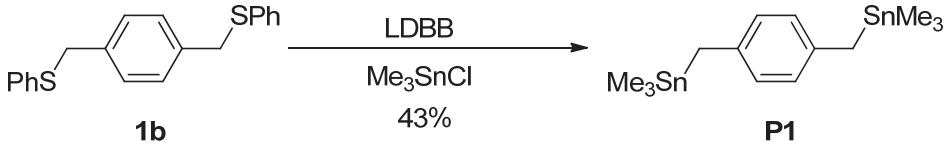


1a **1b**

1,4-bis((phenylthio)methyl)benzene (1b)

Thiophenol (2.2 mL, 21.5 mmol) was added to a solution of sodium hydroxide (1.2 g, 30 mmol) in 15 ml ethanol at 0 ºC and the reaction mixture was stirred for 1 h. Then α,α'-dibromo-p-xylene (**1a**, 2.64 g, 10 mmol) was added. The resulting mixture was warmed to room temperature slowly. After adding water, the reaction mixture was extracted with dichloromethane. The organic layer was then washed with 2 N aqueous sodium hydroxide solution and brine, dried over sodium sulfate and evaporated under reduced pressure. The crude product was further purified by recrystallization from hexane/dichloromethane to yield compound **1b** in 77% yield

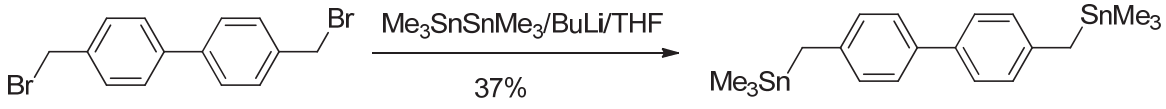
(2.5 g) as a colorless solid. ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.30-7.16 (m, 14H), 4.09 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.4, 136.2, 129.9, 128.9, 128.8, 126.4, 38.7 ; HRMS (FAB+) calcd for $C_{20}H_{18}S_2$: 322.0850; found 322.0861.



1b **P1**

1,4-bis((trimethylstannyl)methyl)benzene (P1)

Lithium ribbon (49 mg, 7 mmol, washed by dry hexane, MeOH and THF) was cut into small pieces and added to a solution of 4,4'-di-tert-butyl-1,1'-biphenyl (DBB, 1.86 g, 7 mmol) in 7 mL anhydrous THF at room temperature under argon atmosphere. As soon as the surface of the lithium turned green, the mixture was cooled to 0 ^{o}C and stirred for 5 h. Then, 7 ml of the aforementioned lithium 4,4′-di-*tert*-butylbiphenylide (LDBB) solution were slowly added to the solution of 1,4-bis((phenylthio)methyl)benzene (**1b**, 322 mg, 1.0 mmol) in 5 ml THF at -78 ^{o}C under Argon. The resulting mixture was stirred for 1 h at -78 ^{o}C and quenched with 6 ml Me_3SnCl (1.0 M in THF). The reaction mixture was then slowly warmed to room temperature and stirred overnight. Finally, the reaction mixture was quenched with water and extracted with dichloromethane. The organic layer was washed with brine, dried over sodium sulfate and evaporated under reduced pressure. Flash column chromatography (hexanes) over silica gel yielded 1,4-bis((trimethylstannyl)methyl)benzene **(P1)** as a colorless solid in 43% yield (189 mg). ^{1}H NMR (400 MHz, CD_2Cl_2) 6.81 (s, 4H), 2.24 (m, 4H), 0.01 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 137.8, 127.1, 19.7, -10.2; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 2.06; HRMS (FAB+) calcd for $C_{14}H_{26}{}^{118}Sn_2$: 430.0077; found 430.0058.

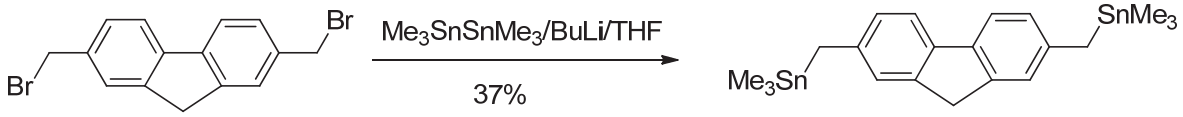


2 **P2**

4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (P2)

A solution of hexamethyldistannane (518 mg, 1.58 mmol) in 20 ml anhydrous THF in a flame dried round bottom flask under Argon atmosphere was cooled to ca. -10º C. Then, a 1.6 M solution of ^{n}BuLi (0.9 ml, 1.43 mmol) in hexanes was added to the reaction mixture dropwise and the reaction mixture was stirred for an additional 45 minutes at -10 ºC. Then, 4,4'-bis(bromomethyl)-1,1'-biphenyl (**2**, 162 mg, 0.47 mmol) was added at ca. -10 ºC, the reaction

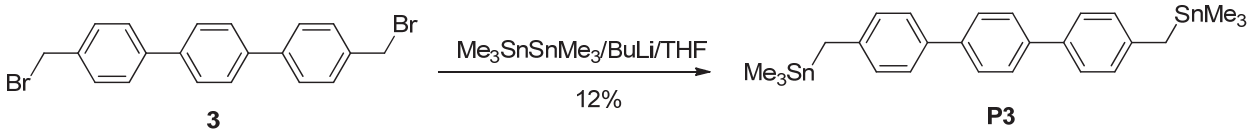
mixture was allowed to warm to room temperature slowly and stirred overnight. Finally, the reaction mixture was quenched with water and extracted with dichloromethane. The organic layer was dried over Na_2SO_4, filtered, evaporated and dried *in vacuo*. Flash column chromatography (hexanes) over silica gel yielded 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**) as a colorless solid in 37% yield (90 mg). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.41 (d, J = 8.0 Hz, 4H), 7.03 (d, J = 8.0 Hz, 4H), 2.34 (m, 4H), 0.06 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 142.4, 136.1, 127.6, 126.7, 20.1, -10.0; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 4.44; HRMS (M+1,FAB+) calcd for $C_{20}H_{31}{}^{116}Sn_2$: 503.0471; found 503.0467.



2a **P2a**

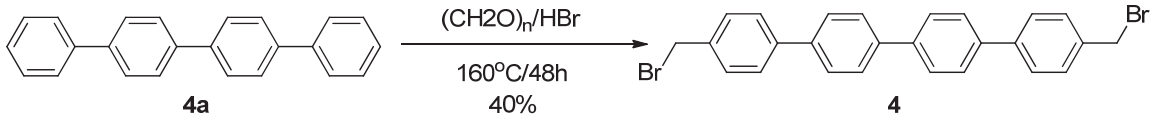
2,7-bis((trimethylstannyl)methyl)-9*H*-fluorene (P2a)

A solution of hexamethyldistannane (0.176 ml, 0.852 mmol) in 20 ml anhydrous THF in a flame dried round bottom flask under Argon atmosphere was cooled to ca. -10º C. Then, a 1.6 M solution of ^{n}BuLi (0.48 ml, 0.77 mmol) in hexanes was added to the reaction mixture dropwise and the reaction mixture was stirred for an additional 45 minutes at -10 ºC. Finally, 2,7-bis(bromomethyl)-9*H*-fluorene (**2a**, 0.100 g, 0.284 mmol) was added at ca. -10 ºC, the reaction mixture was allowed to warm to room temperature and was stirred for 14 h at room temperature. The reaction mixture was then diluted with ca. 100 ml dichloromethane and carefully quenched and washed with H_2O. The organic layer was dried over $MgSO_4$, filtered, evaporated and dried *in vacuo*. The crude product was purified with flash column chromatography (hexanes) to give 2,7-bis((trimethylstannyl)methyl)-9*H*-fluorene **(P2a)** as a colorless solid in 33% yield. An analytical sample was obtained by purification with preparative HPLC (eluent: 10% THF in CH_3CN, flowrate: 5 ml/min, retention time: 13.2 min) followed by flash column chromatography over silica gel (hexanes). ^{1}H NMR (300 MHz, CD_2Cl_2) δ 7.50 (d, J = 7.2 Hz, 2H), 7.13 (s, 2H), 6.96 (d, J = 7.2 Hz, 2H), 3.75 (s, 2H), 2.39 (m, 4H), 0.05 (m, 18H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 143.8, 141.6, 137.8, 125.7, 123.7, 119.2, 37.0, 20.7, -10.1; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 3.57; LRMS (FAB+): 520 $[M]^+$; HRMS (M+1, FAB+) calcd for $C_{21}H_{31}{}^{116}Sn_2$: 515.0471; found 515.0464; HRMS (M+1, FAB+) calcd for $C_{21}H_{31}{}^{116}Sn^{118}Sn$: 517.0466; found 517.0453.



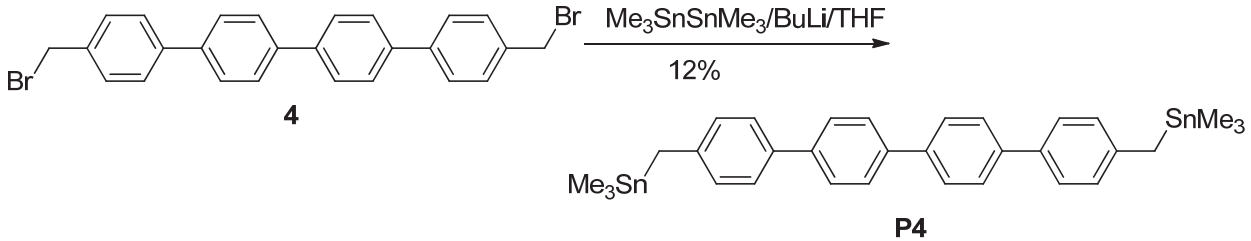
4,4''-bis((trimethylstannyl)methyl)-1,1':4',1''-terphenyl (P3)

4,4''-bis(bromomethyl)-1,1':4',1''-terphenyl (**3**) was stannylated following a similar procedure as described for the preparation of 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**). The crude product was purified by flash column chromatography (hexanes). An analytical sample (**P3,** 12% yield) was obtained by purification with preparative HPLC (eluent: 10% THF in CH_3CN, flow rate: 5 ml/min, retention time: 13.48 min) followed by flash column chromatography over silica gel (hexanes). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.64 (s, 4H), 7.49 (d, J = 8.4 Hz, 4H), 7.07 (d, J = 8.0 Hz, 4H), 2.37 (m, 4H), 0.08 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 143.2, 139.7, 135.6, 127.6, 127.1, 127.0, 20.2, -10.0; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 5.13; HRMS (FAB+) calcd for $C_{26}H_{34}{}^{116}Sn_2$: 578.0707; found 578.0724;



4,4'''-bis(bromomethyl)-1,1':4',1'':4'',1'''-quaterphenyl (4)

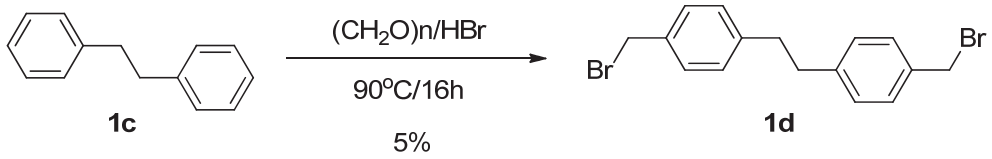
P-quaterphenyl (**4a**, 1.53 g, 5 mmol) and paraformaldehyde (1.5 g, 50 mmol) were placed in a 100 mL pressure vessel, and 33% HBr in HOAc (15 mL) were added. Then, the mixture was heated to 160 ^{o}C for 48 h. After, the reaction mixture was cooled to room temperature, the acetic acid was decanted and ethyl acetate was added. Finally, the mixture was sonicated for 30 min, the solids were separated by filtration and washed with ethyl acetate to yield compound **4** as a colorless solid in 40% yield (1.0 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.68 (m, 8H), 7.63 (d, J = 8.4 Hz, 4H), 7.49 (d, J = 8.0 Hz, 4H), 4.57 (s, 4H).



4,4'''-bis((trimethylstannyl)methyl)-1,1':4',1'':4'',1'''-quaterphenyl (P4)

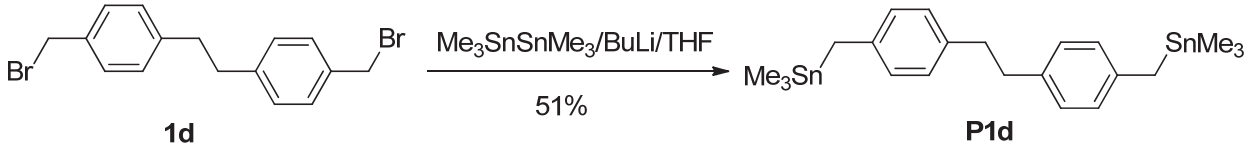
Compound **4** was stannylated following a similar procedure as described for the preparation of 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**). The crude product was purified by flash

column chromatography over silica gel (hexanes). An analytical sample (**P4**, 12% yield) was obtained by purification with preparative HPLC (eluent: 10% THF in CH_3CN, flow rate: 5 ml/min, retention time: 16.5 min) followed by flash column chromatography over silica gel (hexanes). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.73-7.67 (m, 8H), 7.51 (d, *J* = 8.4 Hz, 4H), 7.09 (d, *J* = 8.0 Hz, 4H), 2.38 (m, 4H), 0.09 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 143.4, 140.3, 139.2, 135.5, 127.7, 127.5, 127.2, 127.0, 20.2, -10.0; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 5.30; HRMS (FAB+) calcd for $C_{32}H_{38}{}^{116}Sn_2$: 654.1020; found 654.1044.



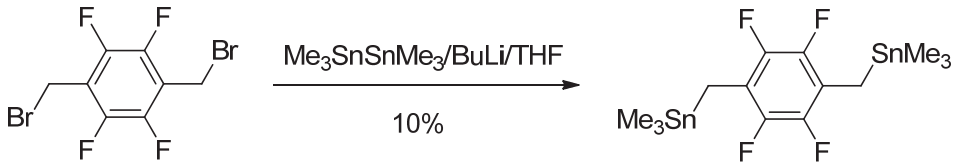
1,2-bis(4-(bromomethyl)phenyl)ethane (1d)

Bibenzyl (**1c**, 3.64 g, 20 mmol) and paraformaldehyde (1.26 g, 42 mmol) were placed in a 50 mL round bottom flask, and 33% HBr in HOAc (20 mL) were added. The mixture was heated to 90 ^{o}C for 16 h and then cooled to room temperature. The solids were filtered, washed with hexanes and recrystallized twice from hexanes to yield pure 1,2-bis(4-(bromomethyl)phenyl)ethane (**1d**, 400 mg) in 5% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.0 Hz, 4H), 7.15 (d, *J* = 8.0 Hz, 4H), 4.49 (s, 4H), 2.90 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.9, 135.5, 129.1, 128.9, 37.3, 33.6; HRMS (FAB+) calcd for $C_{16}H_{16}{}^{79}Br_2$: 365.9619; found 365.9641.



1,2-bis(4-((trimethylstannyl)methyl)phenyl)ethane (P1d)

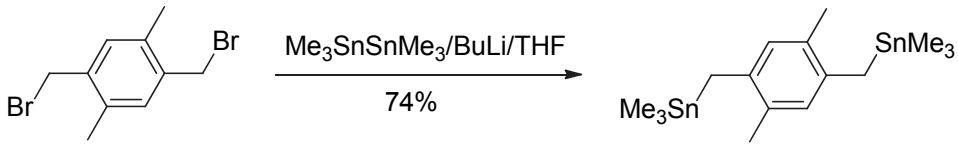
Compound **1d** was stannylated following a similar procedure as described for the preparation of 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**). The crude product was purified by flash column chromatography (hexanes) over silica gel to yield pure 1,2-bis(4-((trimethylstannyl)methyl)phenyl)ethane (**P1d)** in 51% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (d, *J* = 8.0 Hz, 4H), 6.87 (d, *J* = 8.0 Hz, 4H), 2.79 (s, 4H), 2.27 (m, 4H), 0.04 (m, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.3, 136.6, 128.5, 126.6, 37.6, 19.6, -10.1 ; ^{119}Sn NMR (112 MHz $CDCl_3$) δ 2.58; HRMS (M-15,FAB+) calcd for $C_{21}H_{31}{}^{116}Sn_2$: 515.0471; found 515.0489.



1e **4FP1**

((perfluoro-1,4-phenylene)bis(methylene))bis(trimethylstannane) (4FP1)

Compound (**1e**) was stannylated following a similar procedure as used for the preparation of 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**). The crude product was purified by flash column chromatography (hexanes) over silica gel. An analytical sample of **4FP1** was obtained in 10% yield by further purification with preparative HPLC (eluent: 10% THF in CH_3CN, flow rate: 5 ml/min, retention time: 11.3 min). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 2.21 (m, 4H), 0.10 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 143.7(dm, J = 237.1 Hz), 116.5(m), 5.7, -9.5; ^{119}F NMR (376 MHz, CD_2Cl_2) δ -146.6; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 15.13; HRMS (M-15, FAB+) calcd for $C_{13}H_{19}F_4{}^{116}Sn2$: 482.9468; found 482.9473.



1f **2MeP1**

((2,5-dimethyl-1,4-phenylene)bis(methylene))bis(trimethylstannane) (2MeP1)

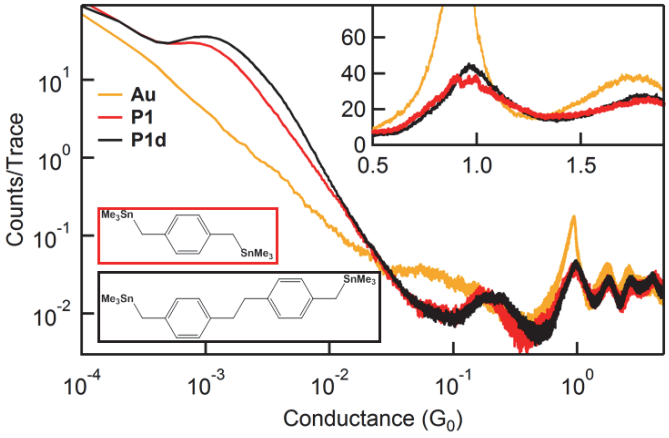
Compound (**1f**) was stannylated following a similar procedure as used for the preparation of 4,4'-bis((trimethylstannyl)methyl)-1,1'-biphenyl (**P2**). The crude product was purified by flash column chromatography (hexanes) over silica gel to yield pure **2MeP1** in 74% yield. ^{1}H NMR (400 MHz, CD_2Cl_2) δ 6.67 (s, 2H), 2.21 (m, 4H), 2.07 (s, 6H), 0.00 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 136.3, 131.1, 129.2, 19.8, 17.6, -9.7; ^{119}Sn NMR (112 MHz, CD_2Cl_2) δ 1.53; HRMS (FAB+) calcd for $C_{16}H_{30}{}^{116}Sn_2$: 454.0392; found 454.0395.

Measurement and Data Analysis:

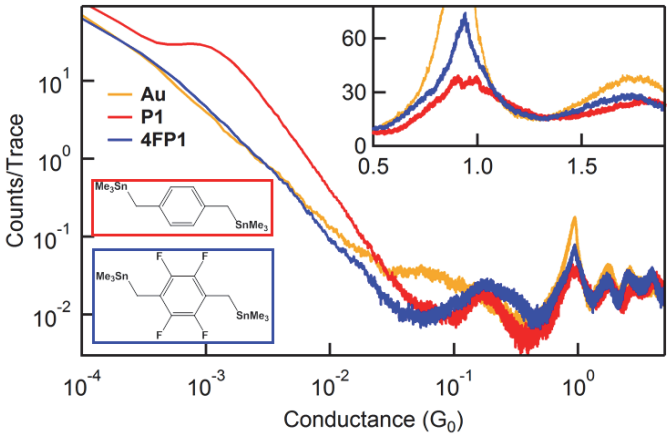
The conductance of each molecule was measured using the STM-based break-junction technique, where an Au tip (Alfa Aesar, 99.999%) cut to be sharp is perpendicularly brought in and out of contact with a substrate of ~100 nm of gold (Alfa Aesar, 99.999%) evaporated onto cleaved mica disks. The substrate is mounted on a piezoelectric positioner (Mad City Labs), so that sub-angstrom resolution in position is achieved. During the entire break junction procedure, a small, constant bias (25-250 mV) is applied between the tip and the substrate while the current is measured (Keithley 428-Prog). Piezo control and data collection was performed using a National Instruments PXI Chassis System (with PXI-4461, PXI-6289) at 40 kHz and driven and managed with a custom-program using Igor Pro (Wavemetrics, Inc.).

The experimental set-up is kept under ambient conditions. For each experiment, the substrate is cleaned under UV/Ozone for 15 minutes prior to use. For every conductance trace measurement, the STM tip is first brought into hard contact with the substrate to achieve a conductance greater than ~ 10 G_0. At this point, the junction electrodes are pulled apart at a speed of 15 nm/s for 0.25 seconds. Conductance is measured as a function of tip-sample displacement to generate conductance traces. For each tip/substrate pair, at least one set of 1,000 traces of clean gold breaks is collected to ensure the system is clean. Then, the target molecule, dissolved in 1,2,4-trichlorobenzene (~10 mM) is deposited and over 10,000 conductance traces are collected for each of the molecules reported here. To determine the conductance of a molecule, every trace is binned linearly into conductance bins, without any data selection, and compiled into a single conductance histogram. The resultant peak in the histogram gives the most frequently measured value of molecular conductance. Every molecule was measured using multiple tip/substrate pairs, on different days to check for consistency and reproducibility.

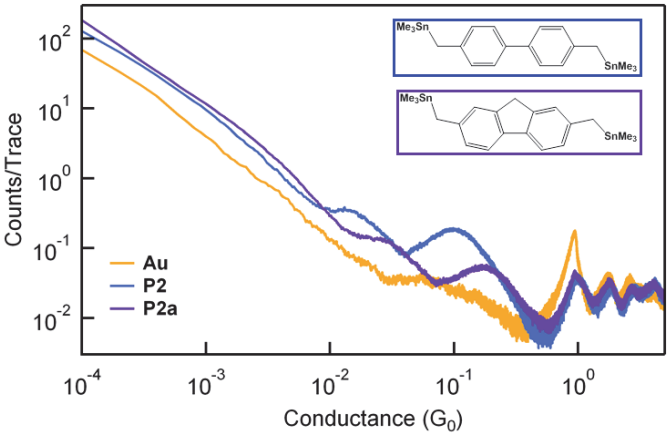
Additional Data: Conductance histograms from additional measurements – all histograms are generated form over 10000 measurements without any data selection**:**



SI Figure 1: Conductance histograms from measurements of **P1** and **P1d** at 25 mV bias.



SI Figure 2: Conductance histograms from measurements of **P1** and **4FP1** at 25 mV bias.

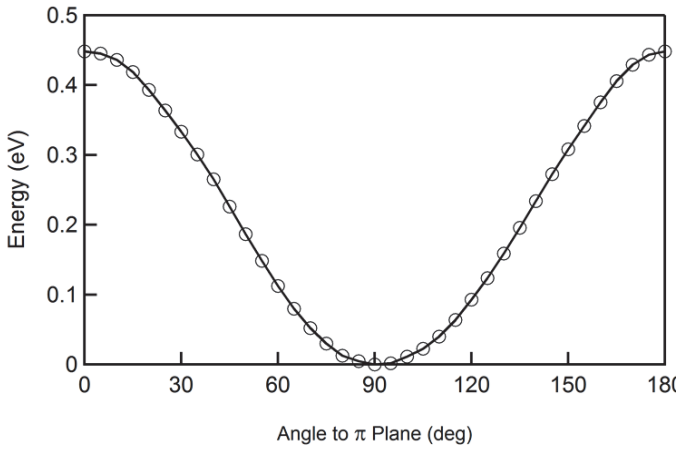


SI Figure 3: Conductance histograms from measurements of **P2** and **P2a** at 250 mV bias.

Procedures for Theoretical Calculations

Calculations of the rotational energy barrier at the Au-C bond:

The energy barrier for rotations of the Au-C bond around the Phenyl ring was calculated for xylelene with a single Au atom to represent the electrodes. Density-Functional Theory calculations were carried out using the Jaguar code[8] to calculate the total energy of this digold-xylylene complex as a function of the angle between the Au-C bonds and the Phenyl plane. The generalized-gradient approximation (PBE implementation) was used for exchange-correlation[9]. Calculations were done using a lacvp** basis set. The angle between the Phenyl ring and the Au-C bonds was fixed and all other coordinates were relaxed. The total energy as a function of Au-C to Phenyl angle is shown in SI Figure 4.

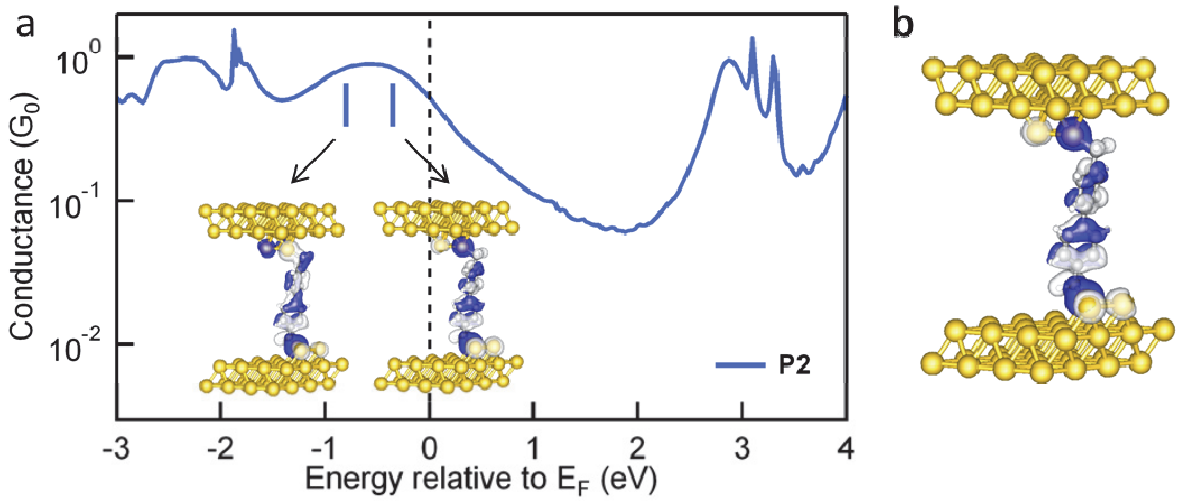


SI Figure 4: Total energy as a function of the angle between the Au-C bond and the phenyl ring for **P1** bonded to two gold atoms.

Transmission calculations:

Structural relaxation calculations of the molecular junctions are carried out using SIESTA[10] with initial structures containing the molecule and Au tips in a 4x4 Au(111) unit cell. The GGA (PBE) approximation is used for exchange-correlation[9]. Au atomic orbitals are described using single-zeta polarized orbitals (with high cutoff radii for tip and surface atoms) and molecular atoms are described by double-zeta polarized orbitals. Initially, the vertical distance is optimized by varying the electrode-electrode separation. The structure containing 6 Au layers is then optimized until the forces on all molecule and tip atoms are smaller than 0.02 eV/Å.

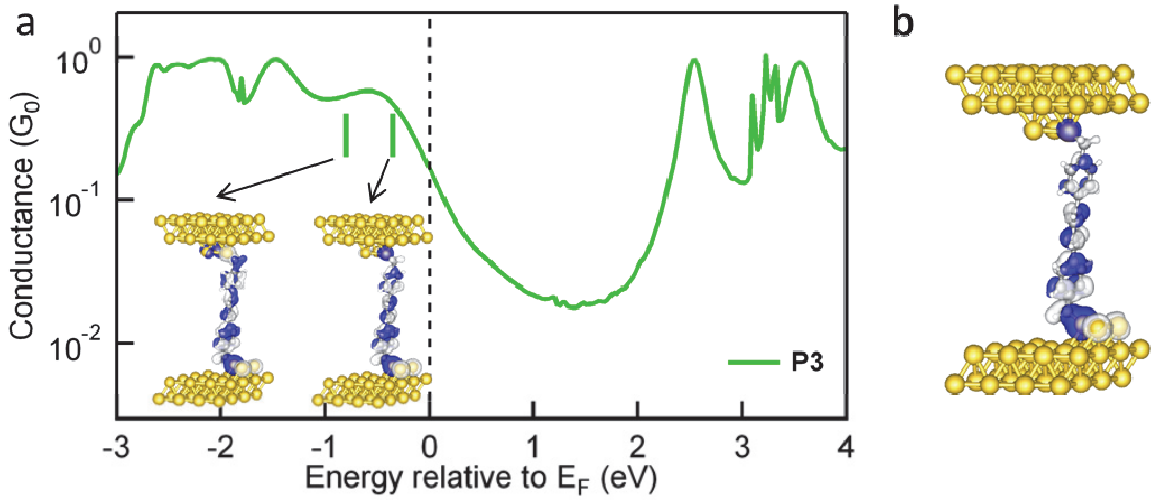
Subsequent transmission calculations are carried out using TranSIESTA[11] for relaxed geometries built from these optimized structures by adding 3 extra Au layers on each side of the supercell. The transport unit cell contains a total of Au 12 layers. The electronic structure is calculated using a 5x5 Monkhorst-Pack grid and a 250 Ry real-space cutoff. Transmission spectra are calculated with a 15x15 sampling of the transverse Brillouin zone. The transmitted scattering states, the real part of which are presented in isosurface plots, are generated at the Gamma point using the method of Paulsson and Brandbyge[12]. The effect of the electrode tip structure was investigated by checking these results against optimized structures bound to each Au electrode through a single adatom. The conductance at the Fermi level differed by ~5%, ~17%, ~25% and 44% for **P1**, **P2**, **P3** and **P4** respectively, within the width of the experimental histograms.



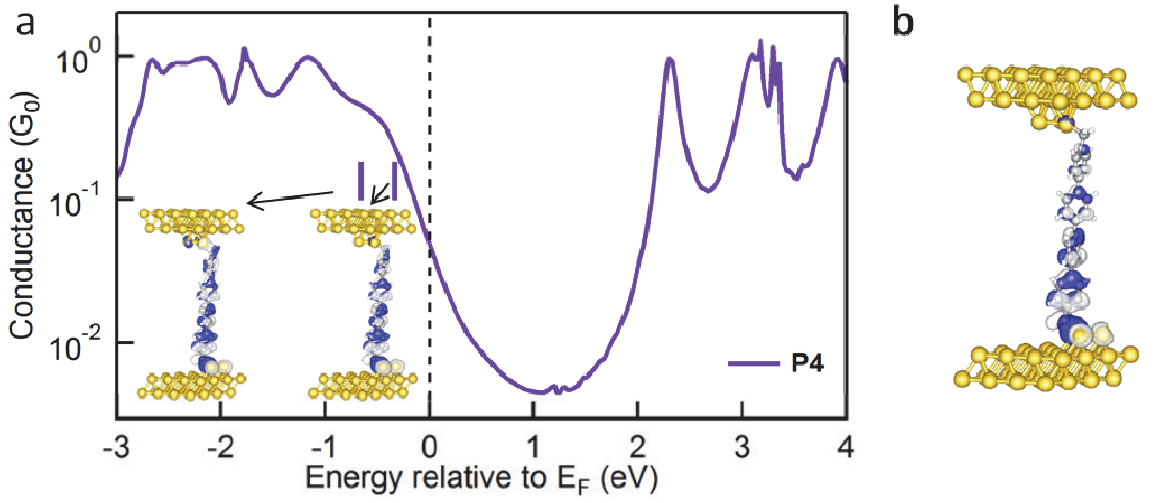
SI Figure 5 (a) Calculated transmission spectrum for **P2** bonded to Au electrodes. Inset: Isosurface plots of the real part of the transmitted scattering states for energies at the vertical bars (-0.8 eV and -0.35 eV) showing the even and odd combinations of the Au-C bonds coupled through the π backbone. (b) Isosurface plot showing the scattering state at E_F.

The identification of the two highest occupied resonances in the transmission spectrum for **P1** is straightforward. For **P2** – **P4**, the coupling to the electrodes is so strong that the two Au-C MO derived resonances are subsumed under a single peak in the transmission spectrum. To arrive at the physical picture presented in Figure 4b in the text, an isolated fragment consisting of the oligomer bonded to a single Au atom on each side is analyzed separately to get a tunnel splitting between the even and odd combinations of the Au-C MOs. Since the strong interaction with the electrodes also shifts the resonance positions relative to the isolated

fragment, we place the energies of the split resonances symmetrically relative to the transmission peak in Figure 4b. We then calculate the transmitted scattering state at those energies to verify the odd /even character of the state, as illustrated below if SI Figures 5-7. The transmitted scattering state at the Fermi energy for **P1** – **P4** has the same physical character as the highest occupied resonance, but shows progressively more attenuation across the series as the tunnel coupling through the back bone state systematically drops with longer oligomers.



SI Figure 6 (a) Calculated transmission spectrum for **P3** bonded to Au electrodes. Inset: Isosurface plots of the real part of the transmitted scattering states for energies at the vertical bars (-0.6 eV and -0.35 eV) showing the even and odd combinations of the Au-C bonds coupled through the π backbone. (b) Isosurface plot showing the scattering state at E_F.



SI Figure 7 (a) Calculated transmission spectrum for **P4** bonded to Au electrodes. Inset: Isosurface plots of the real part of the transmitted scattering states for energies at the vertical bars (-0.5 eV and -0.4 eV) showing the even and odd combinations of the Au-C bonds coupled through the π backbone. (b) Isosurface plot showing the scattering state at E_F.

References:

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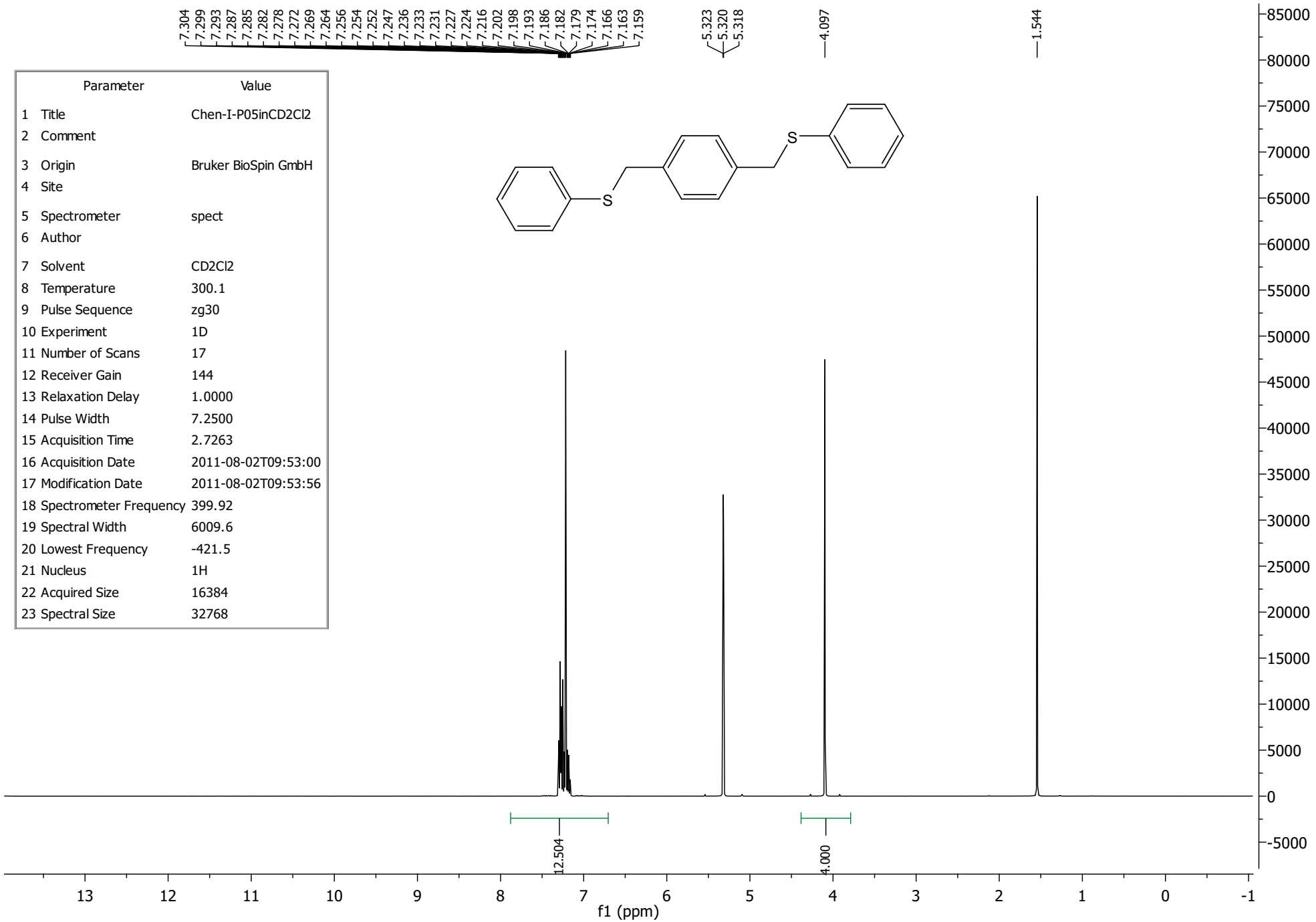

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4 Site	
5 Spectrometer	spect
6 Author	
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9 Pulse Sequence	zg30
10 Experiment	1D
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13 Relaxation Delay	1.0000
14 Pulse Width	7.2500
15 Acquisition Time	2.7263
16 Acquisition Date	2011-08-02T09:53:00
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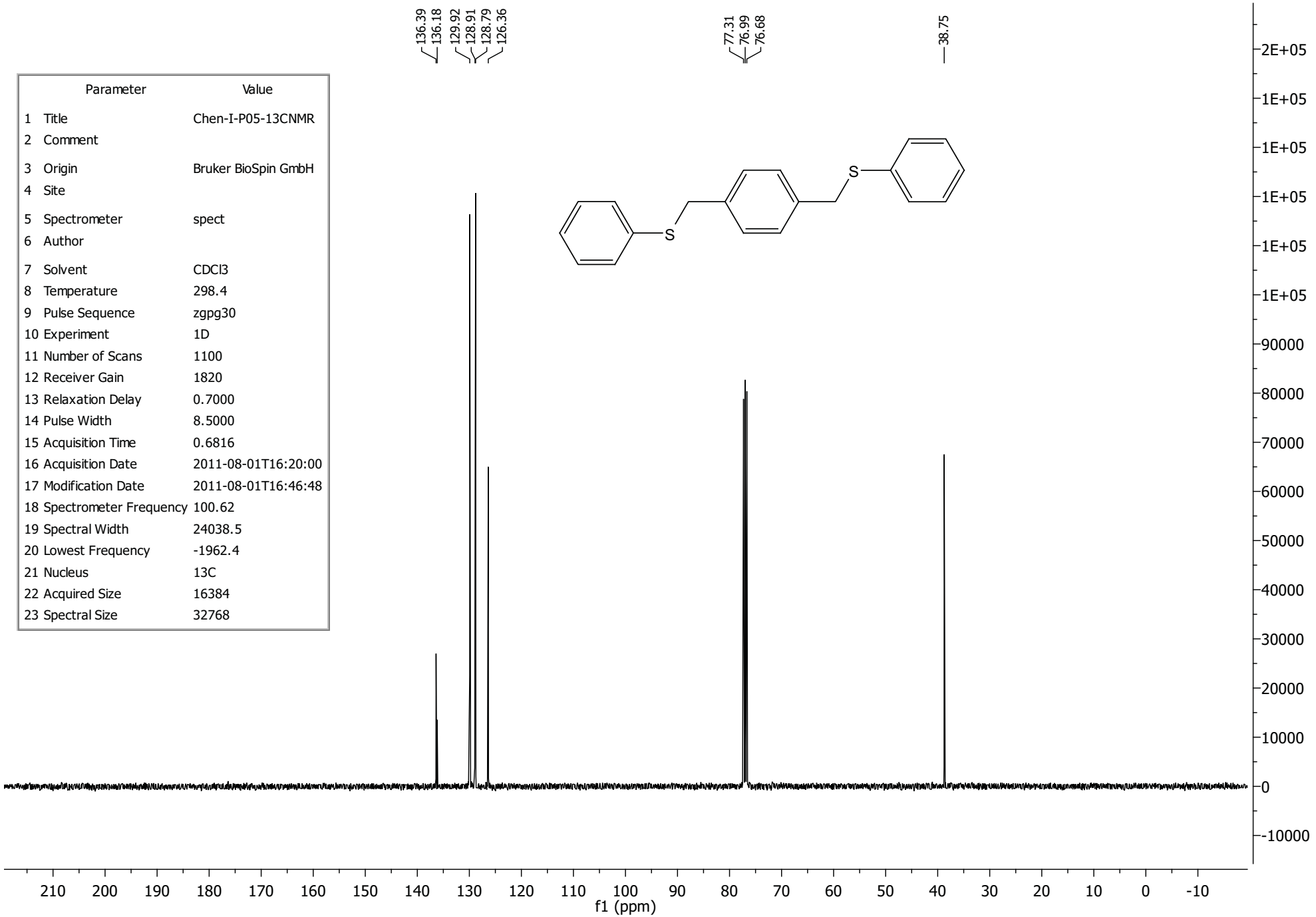
19 Spectral Width	6009.6
20 Lowest Frequency	-421.5
21 Nucleus	1H
22 Acquired Size	16384
23 Spectral Size	32768



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21 Nucleus	13C
22 Acquired Size	16384
23 Spectral Size	32768

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4	Site	
5	Spectrometer	spect
6	Author	
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22	Acquired Size	16384
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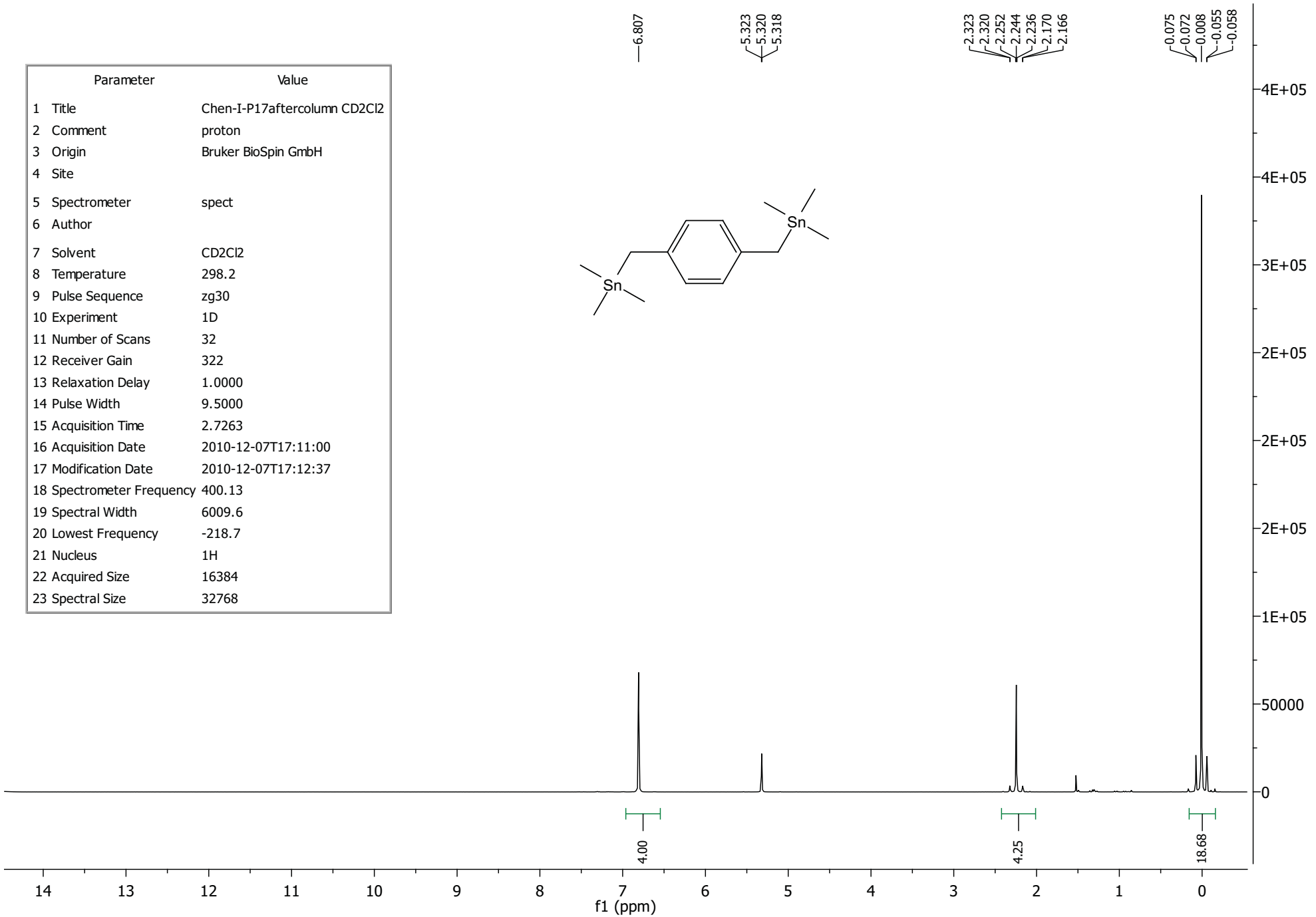

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4	Site	
5	Spectrometer	spect
6	Author	
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21	Nucleus	13C
22	Acquired Size	16384
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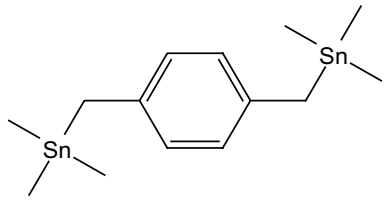

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3 Origin	Bruker BioSpin GmbH
4 Site	
5 Spectrometer	spect
6 Author	
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8 Temperature	298.2
9 Pulse Sequence	zg30
10 Experiment	1D
11 Number of Scans	23
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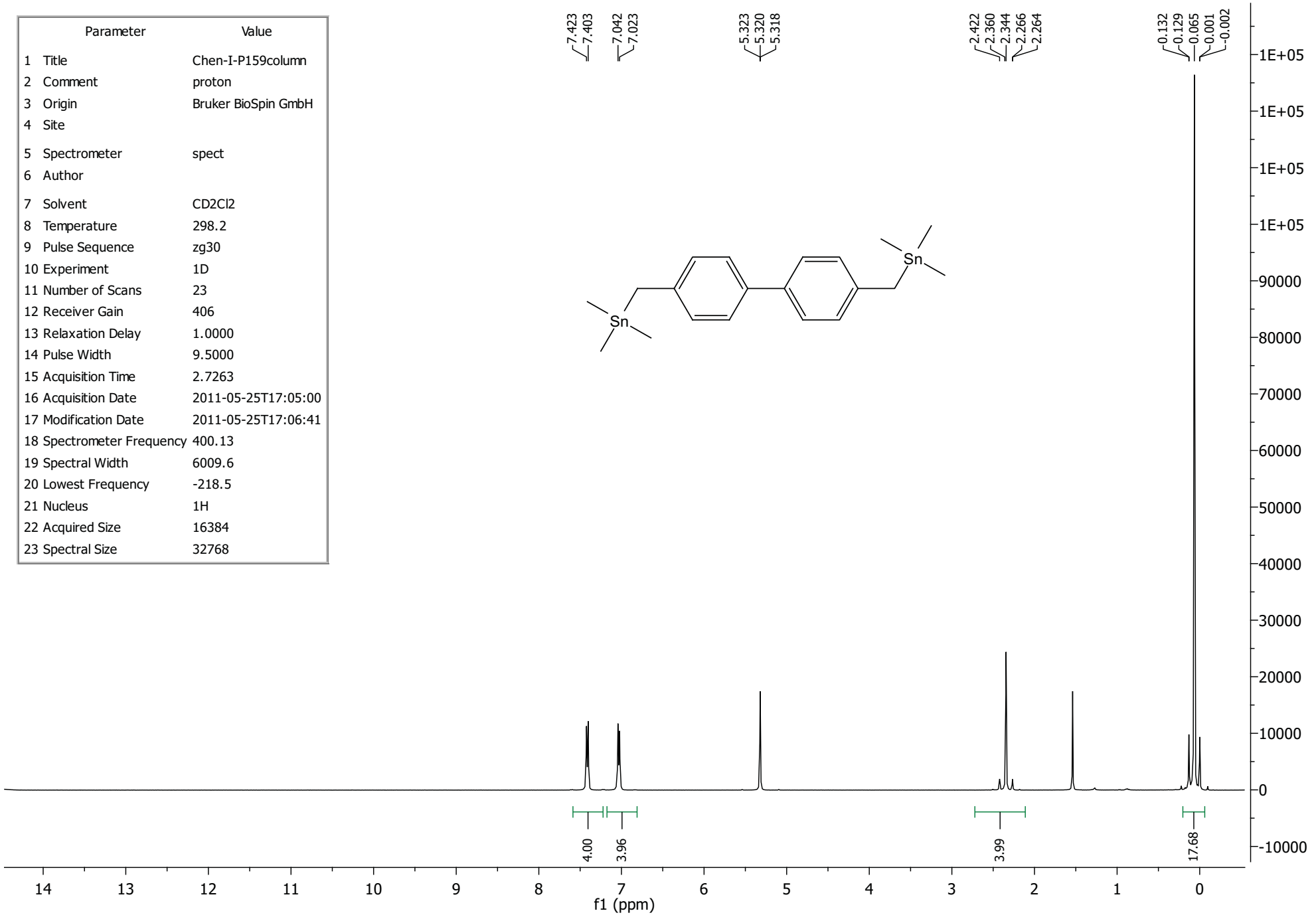
23 Spectral Size	32768



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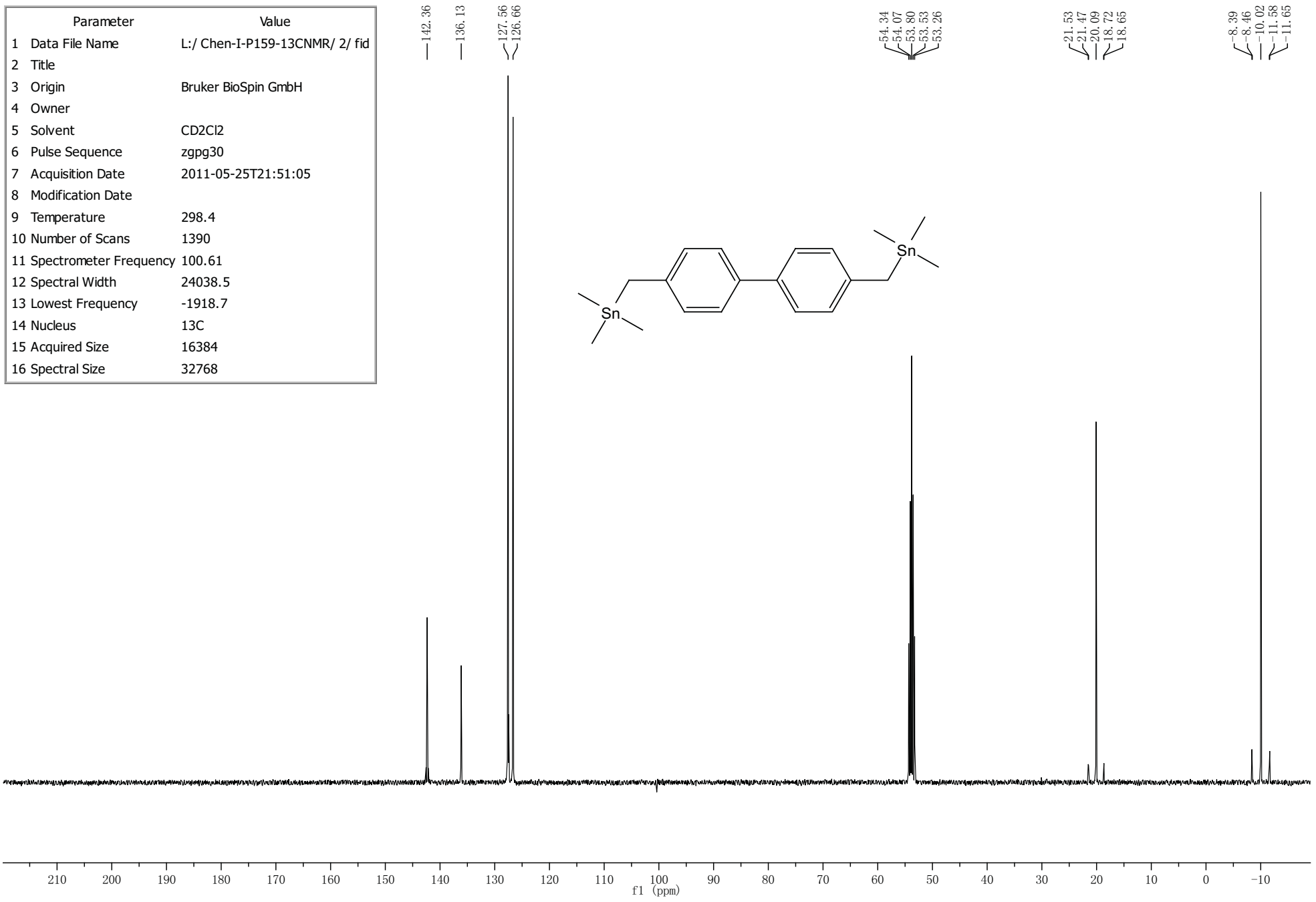

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5 Spectrometer	spect
6 Author	
7 Solvent	CD2Cl2
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9 Pulse Sequence	zg
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13 Relaxation Delay	0.1000
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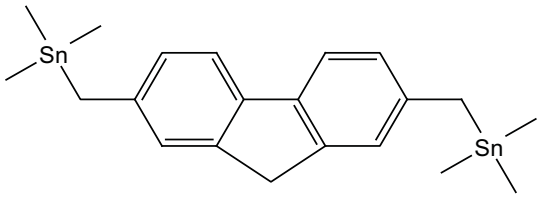

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6	Author	
7	Solvent	CDCl3
8	Temperature	300.0
9	Pulse Sequence	zgpg30
10	Experiment	1D
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12	Receiver Gain	16384
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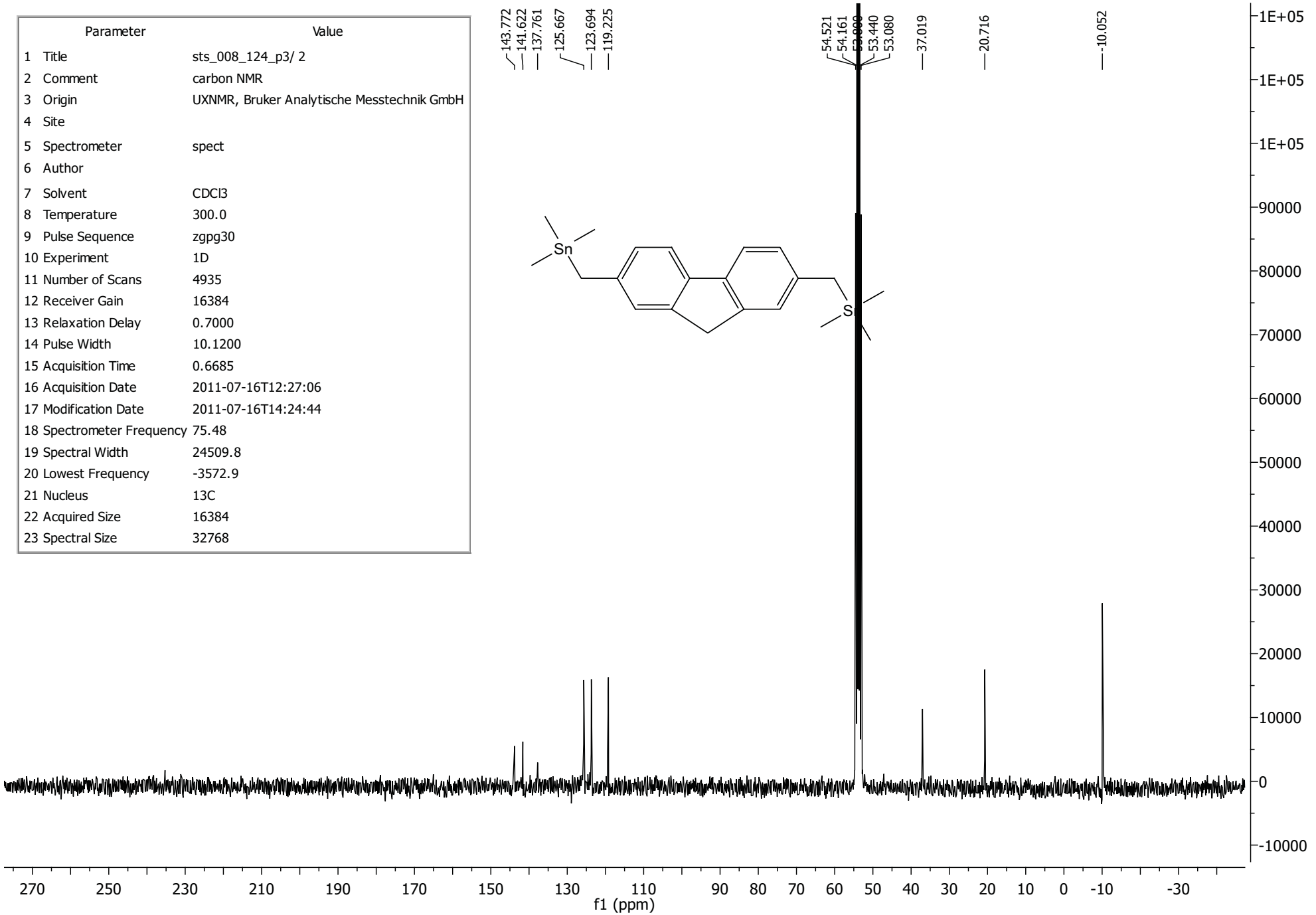

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9	Pulse Sequence	zg30
10	Experiment	1D
11	Number of Scans	19
12	Receiver Gain	575
13	Relaxation Delay	1.0000
14	Pulse Width	9.5000
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22	Acquired Size	16384
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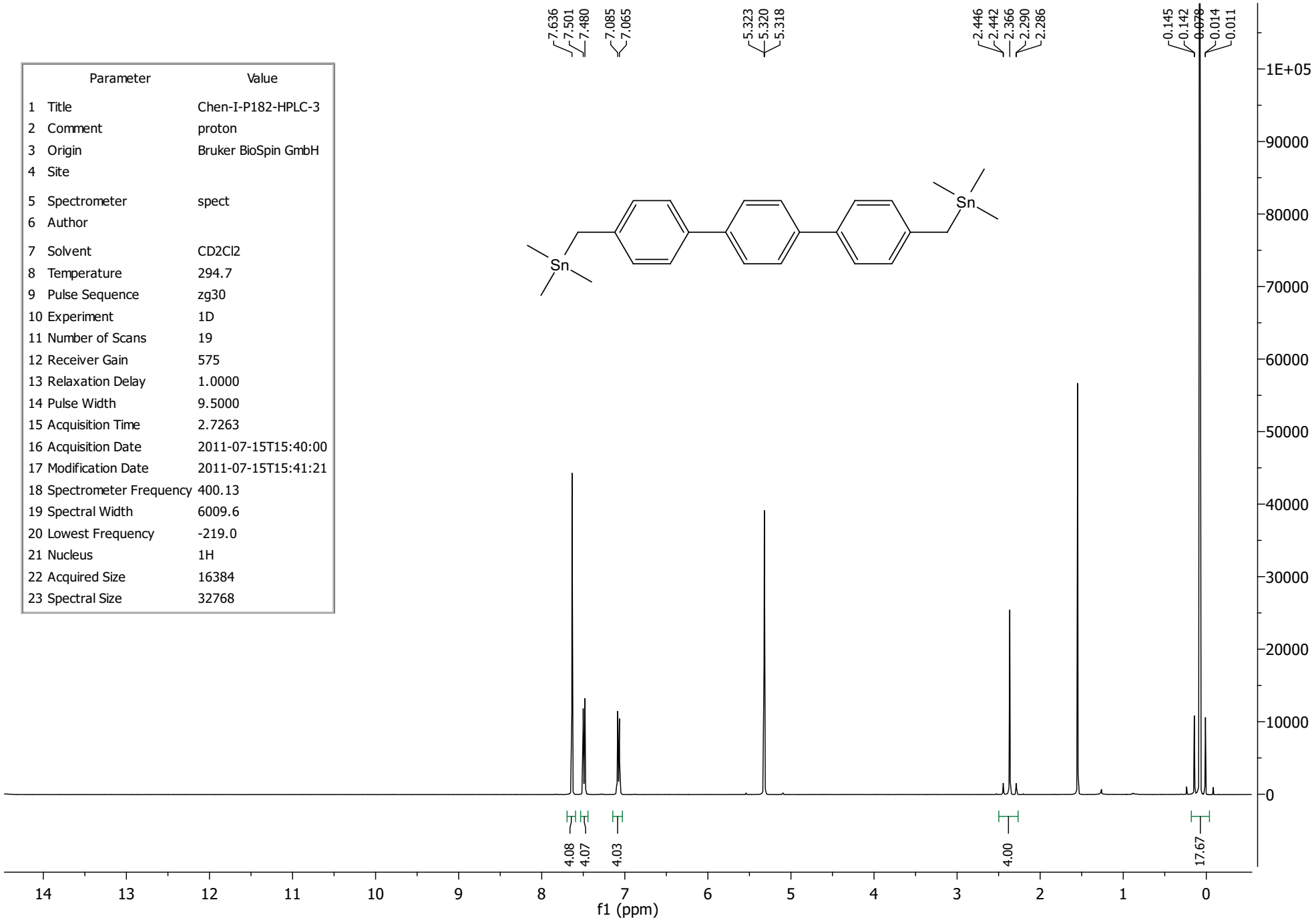

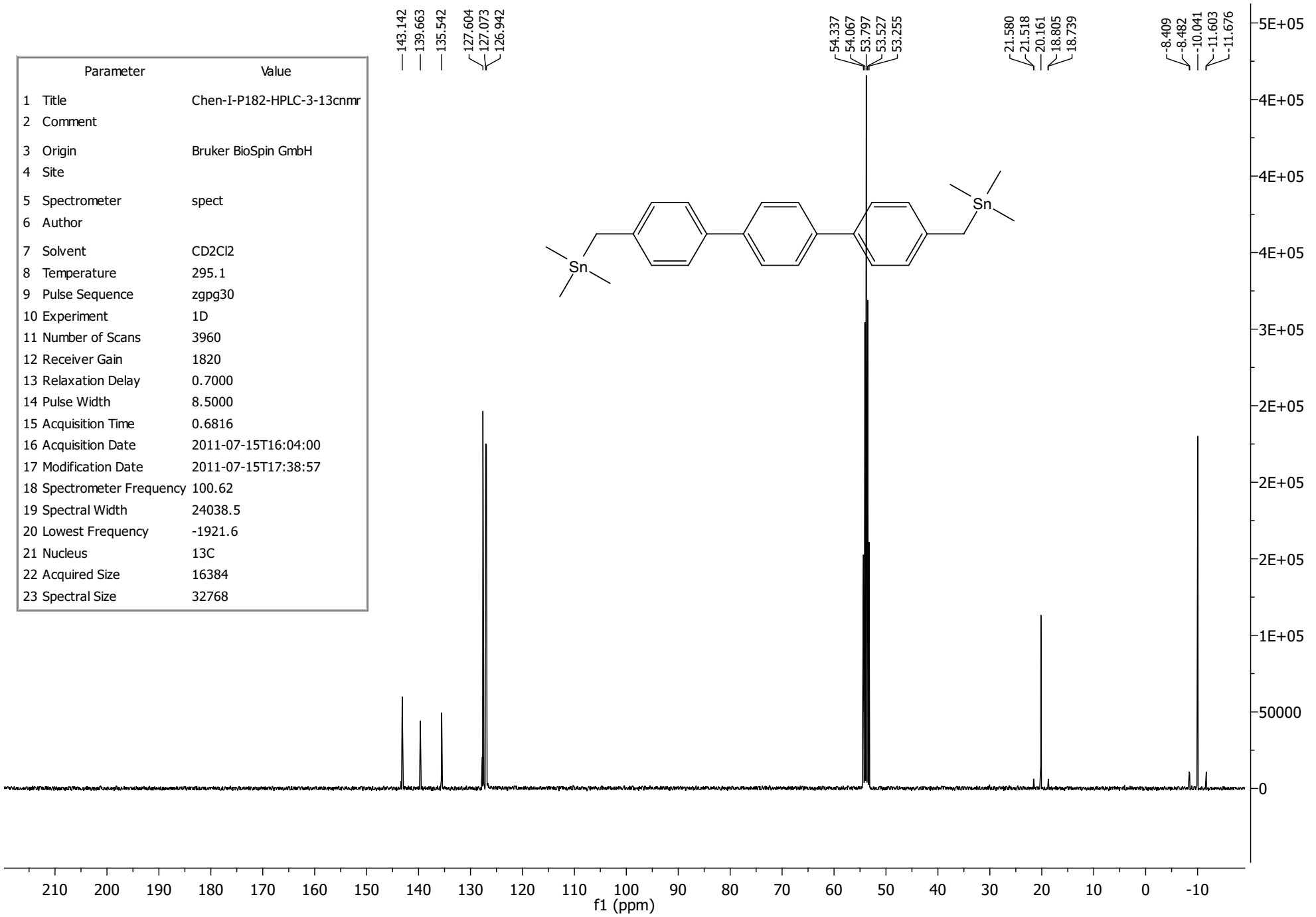

Parameter	Value
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6 Author	
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9 Pulse Sequence	zgpg30
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12 Receiver Gain	1820
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14 Pulse Width	8.5000
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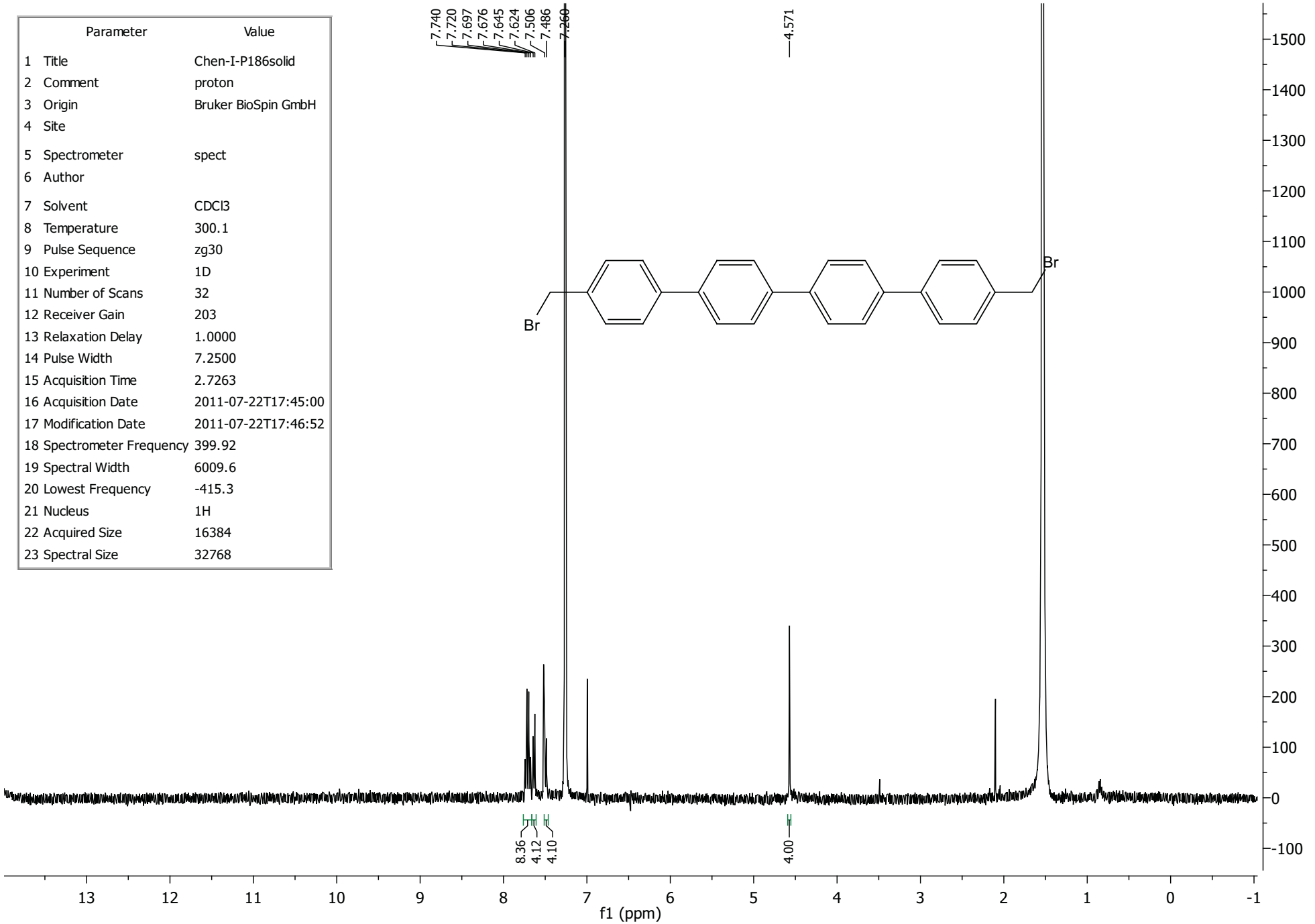
21 Nucleus	13C
22 Acquired Size	16384
23 Spectral Size	32768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6 Author
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17 Modification Date 2011-07-22T17:46:52
18 Spectrometer Frequency 399.92
19 Spectral Width 6009.6
20 Lowest Frequency -415.3
21 Nucleus 1H
22 Acquired Size 16384
23 Spectral Size 32768
7.740
7.720
7.697
7.676
7.645
7.624
7.506
7.486
7.260
4.571
Br
Br
8.36
4.12
4.10
4.00
f1 (ppm)

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5 Spectrometer	spect
6 Author	
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8 Temperature	298.2
9 Pulse Sequence	zg30
10 Experiment	1D
11 Number of Scans	18
12 Receiver Gain	406
13 Relaxation Delay	1.0000
14 Pulse Width	9.5000
15 Acquisition Time	2.7263
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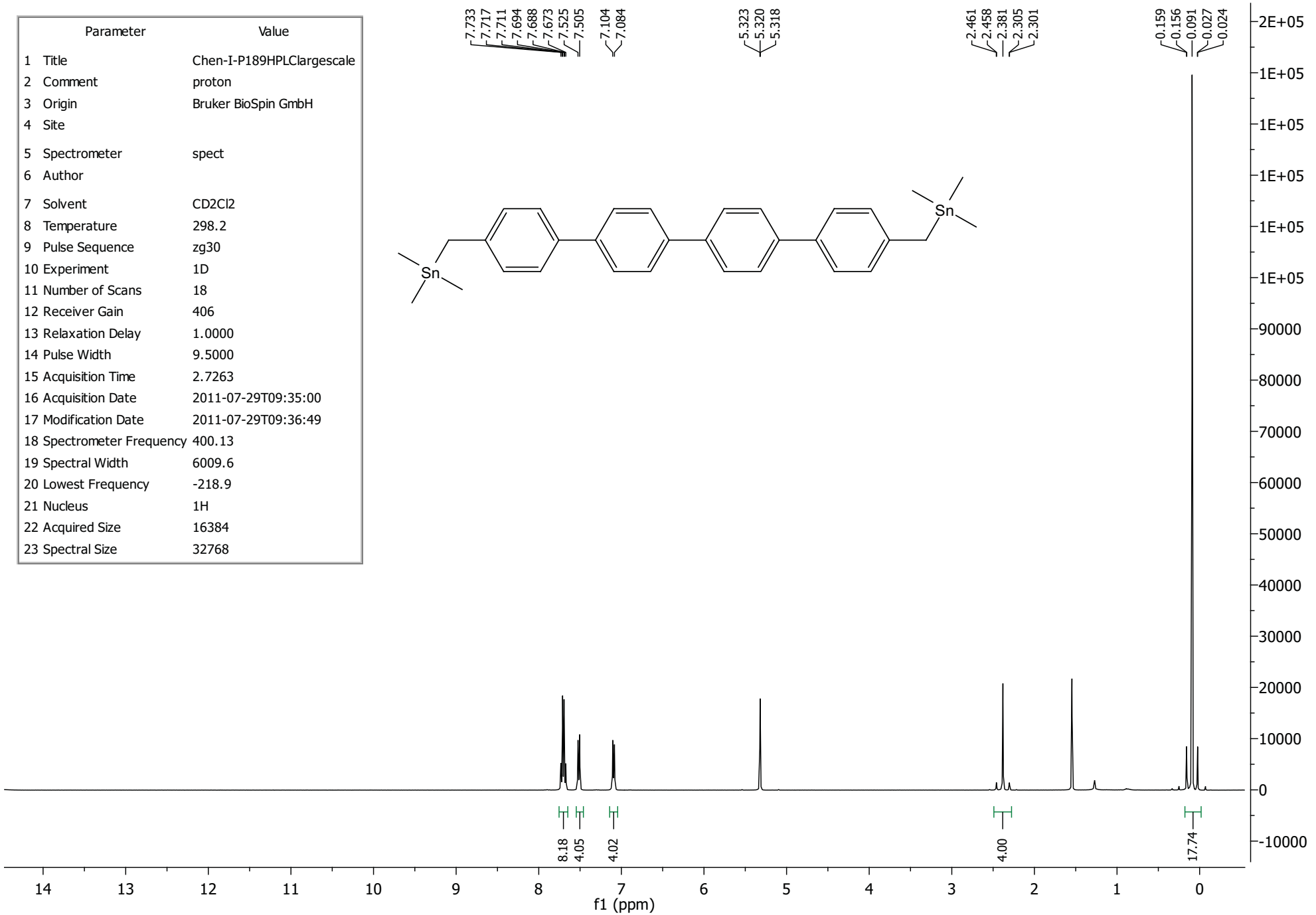
21 Nucleus	1H
22 Acquired Size	16384
23 Spectral Size	32768



Chen-I-P189HPLClargescale

	Parameter	Value
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2	Comment	
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4	Site	
5	Spectrometer	spect
6	Author	
7	Solvent	CD2Cl2
8	Temperature	298.4
9	Pulse Sequence	zgpg30
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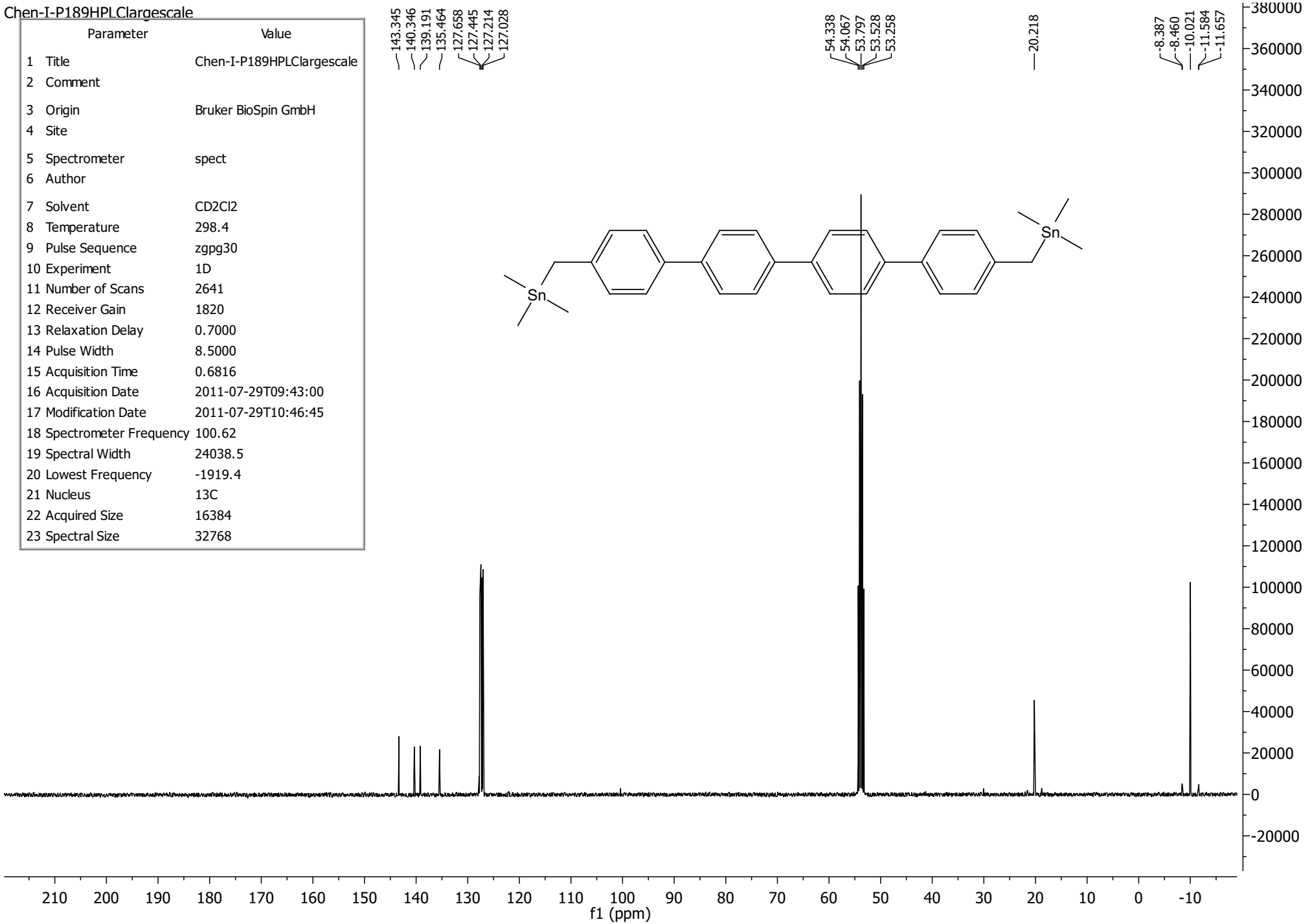
22	Acquired Size	16384
23	Spectral Size	32768



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6	Author	
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9	Pulse Sequence	zg30
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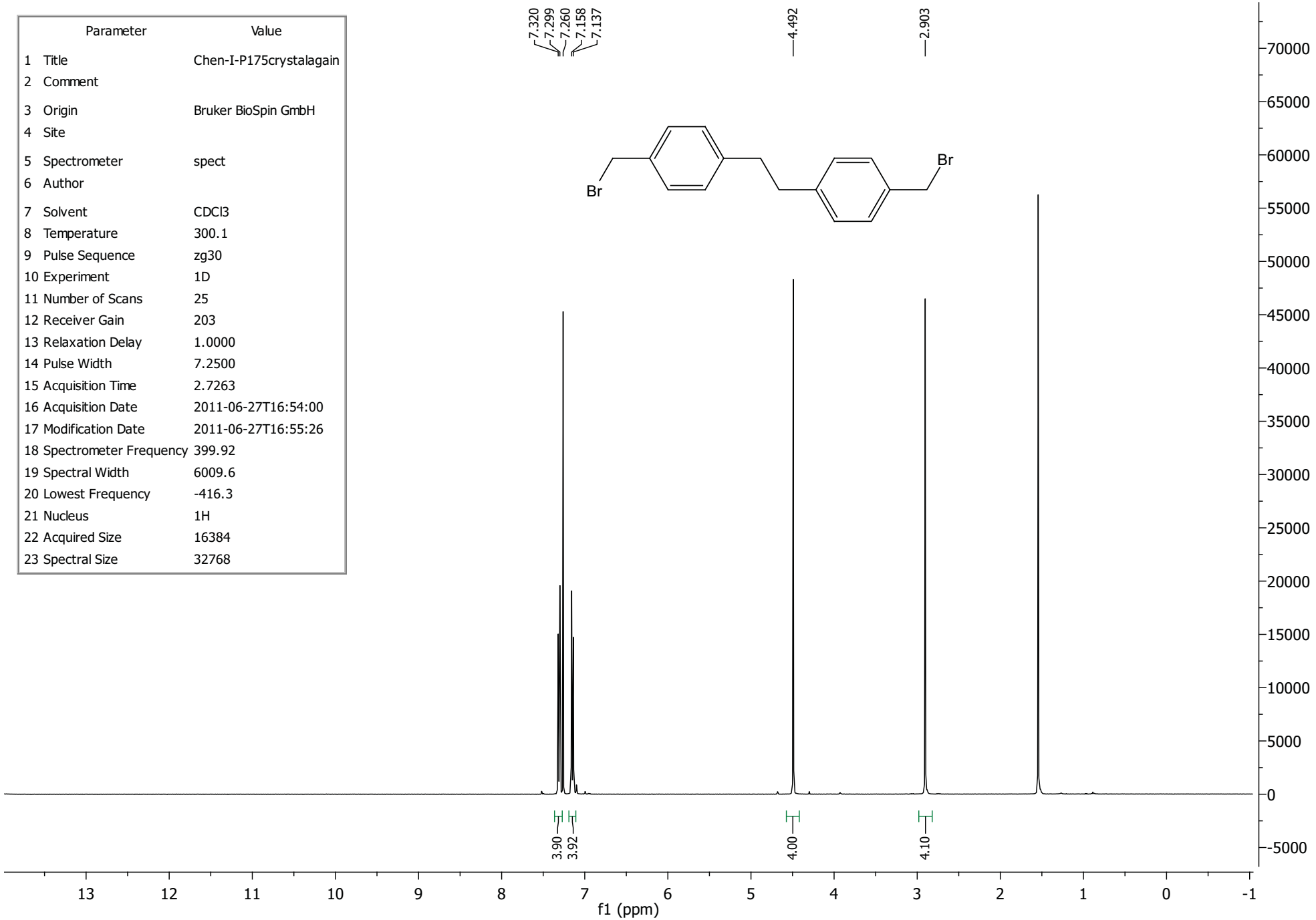

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11	Number of Scans	1978
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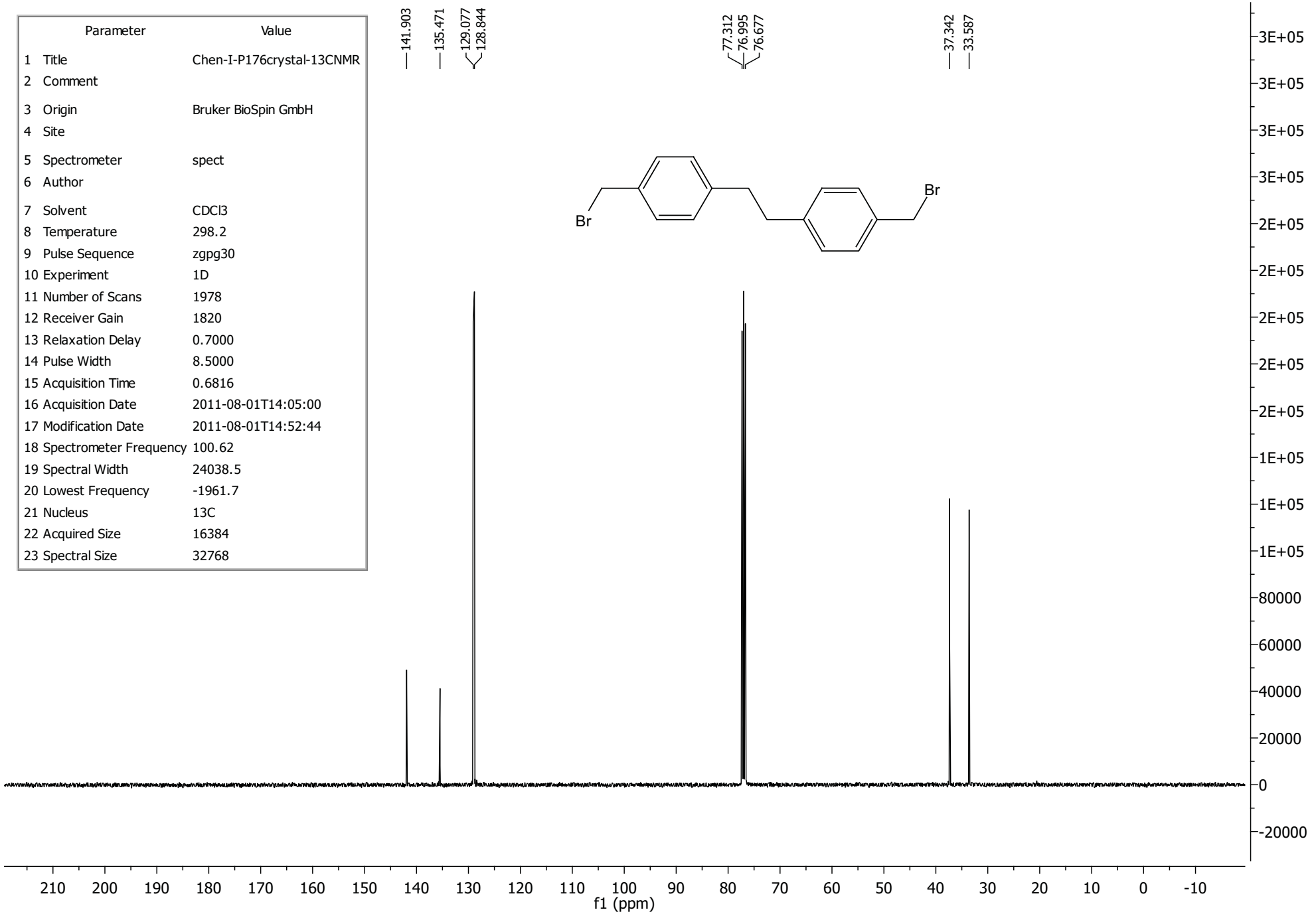

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5	Spectrometer	spect
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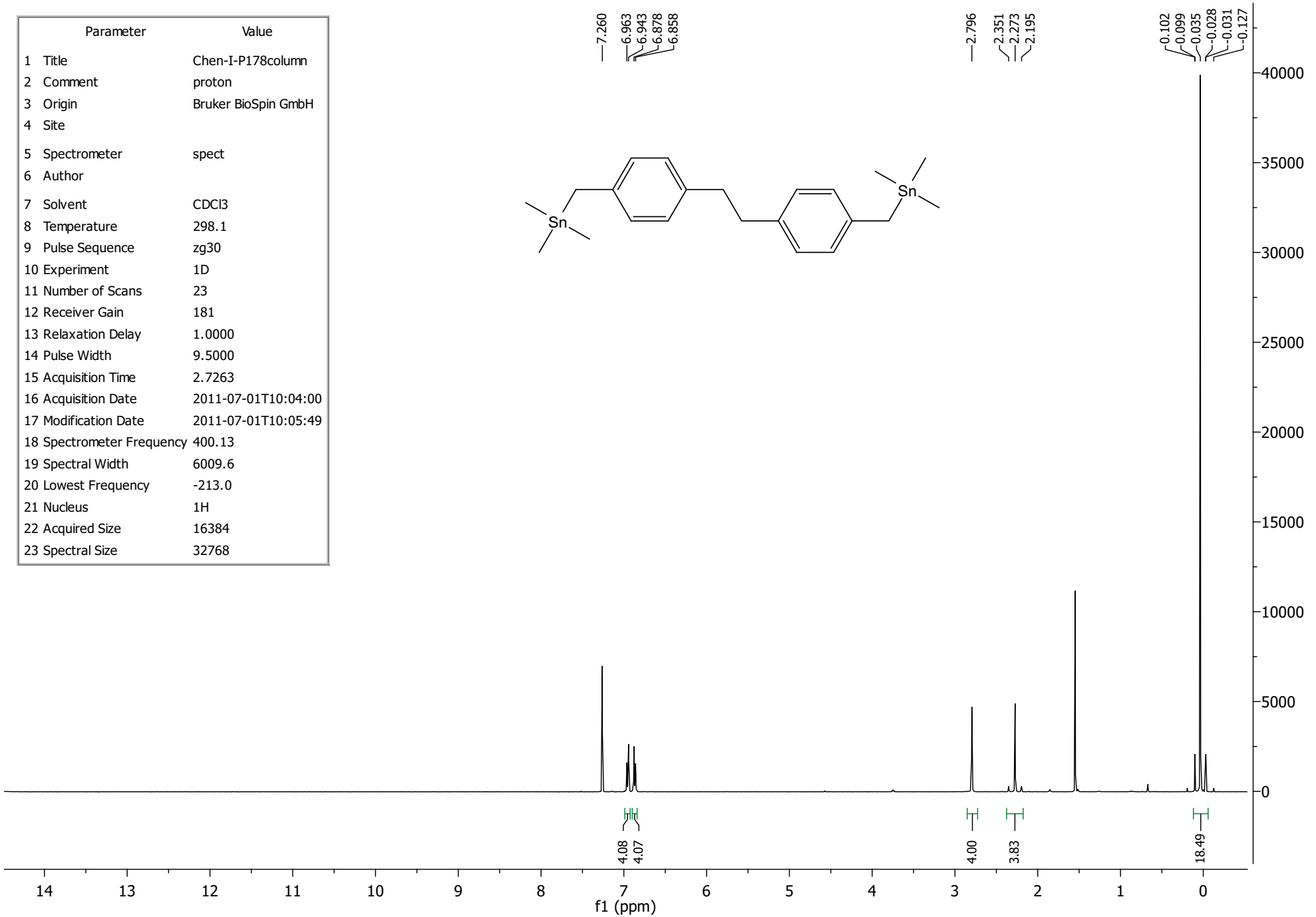
20	Lowest Frequency	-213.0
21	Nucleus	1H
22	Acquired Size	16384
23	Spectral Size	32768



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6	Author	
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10	Experiment	1D
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12	Receiver Gain	1820
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22	Acquired Size	16384
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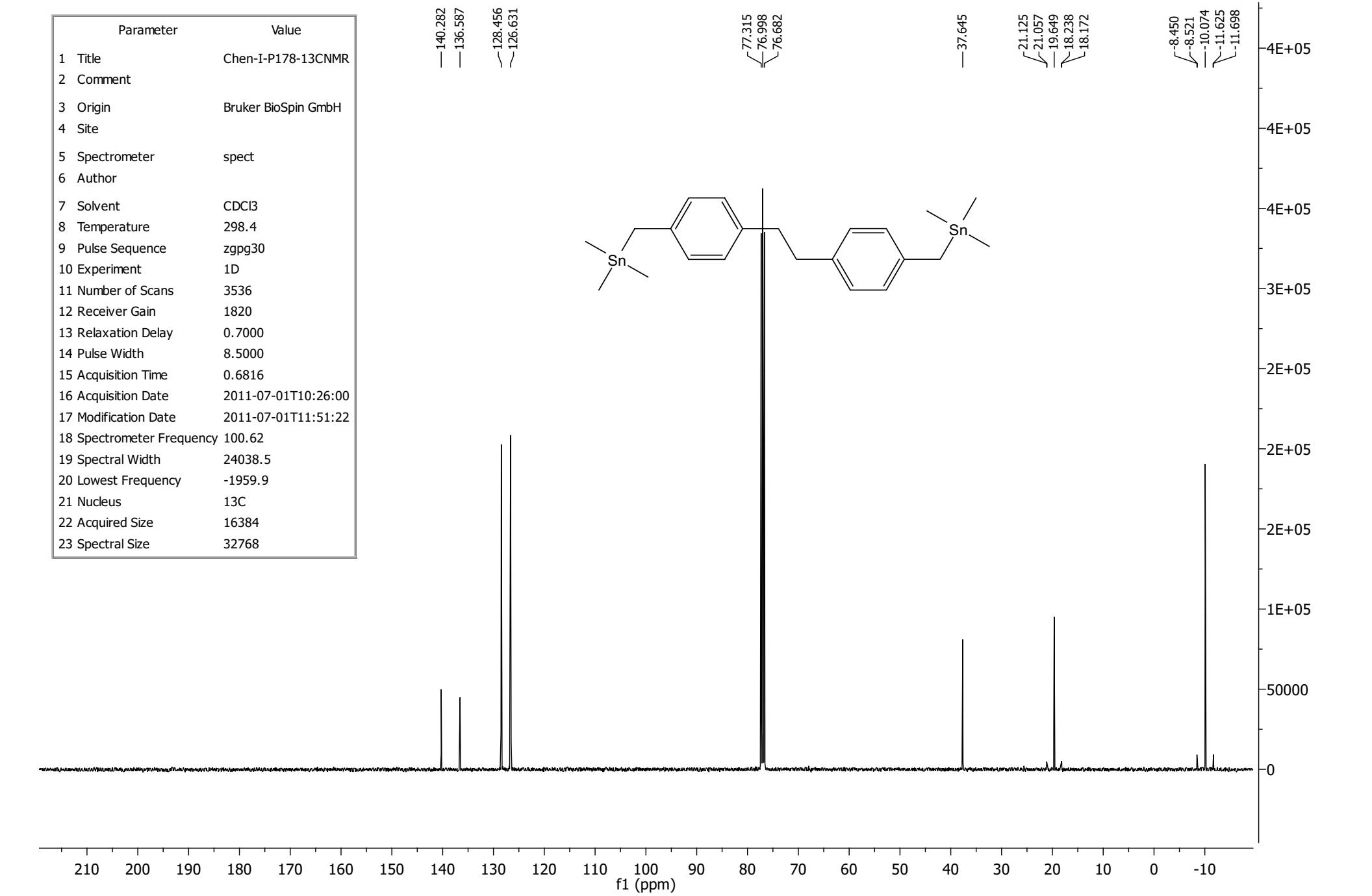

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9	Pulse Sequence	zg30
10	Experiment	1D
11	Number of Scans	30
12	Receiver Gain	114
13	Relaxation Delay	1.0000
14	Pulse Width	7.2500
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17	Modification Date	2011-07-23T16:43:23
18	Spectrometer Frequency	399.92
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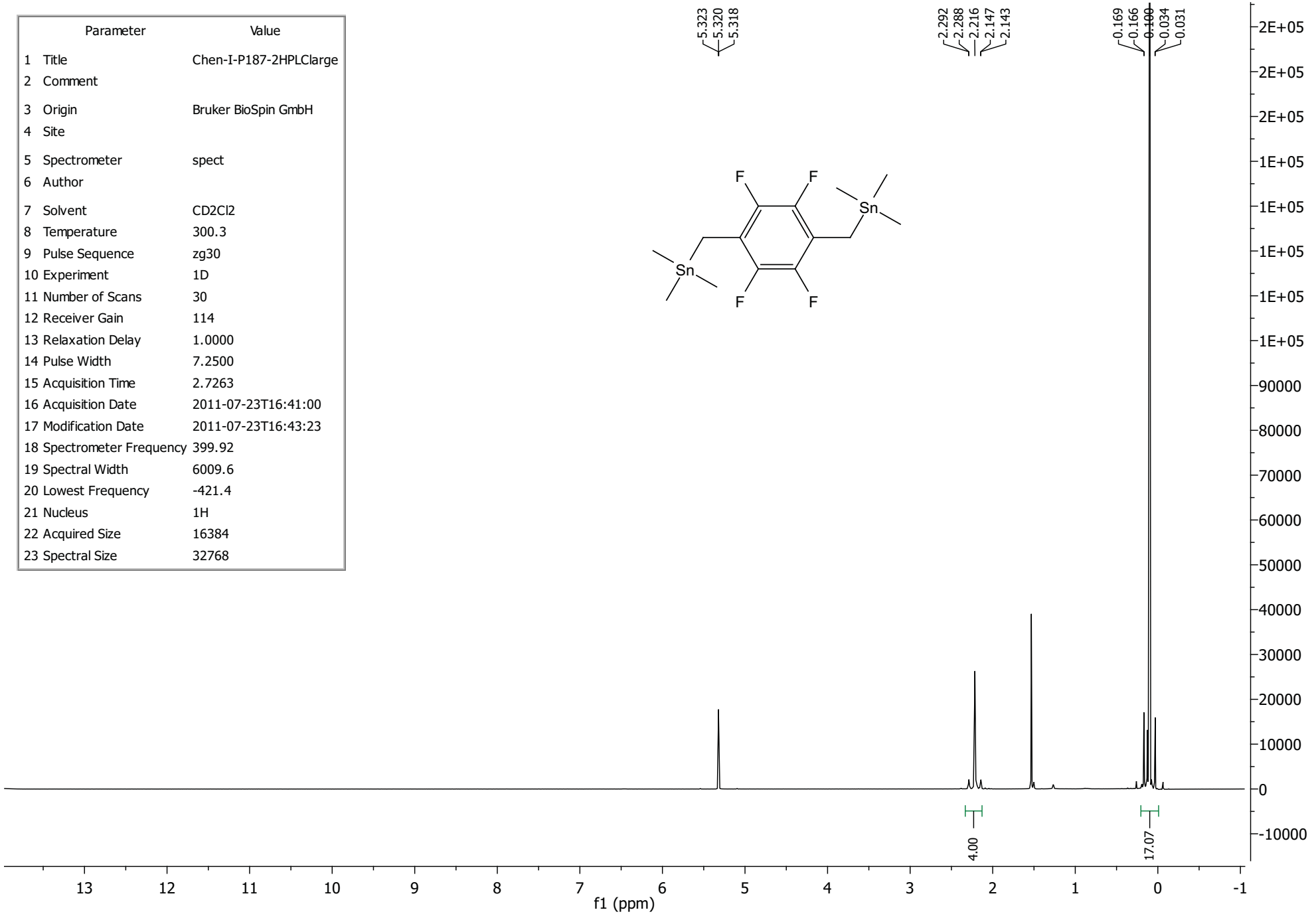
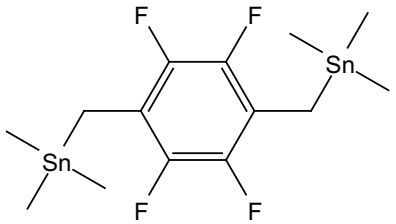
22	Acquired Size	16384
23	Spectral Size	32768

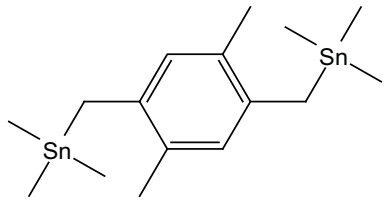


Chen-I-P187-2HPLClarge-13C

	Paramecer	.alt e
1	Vwde	Chen-I-P187-2HPLClarge-13C
2	CTmmenc	
3	i rugun	ort Oer oTBpun k mSH
4	Bwe	
5	BpeGcrTmecer	bpeGc
6	At chTr	
7	BTlvenc	CD2Cl2
8	Vemperact re	298s4
9	Pt lbe Beqt enGe	zgpg30
10	Experumenc	1D
11	Nt mSer Tf BGanb	8528
12	ReGeuver k aun	1820
13	RelaxacoTn Delay	0s7000
14	Pt lbe Wudch	8s5000
15	AGqt uboaTn Vume	0s6816
16	AGqt uboaTn Dace	2011-07-23V17:25:00
17	MTdufuGacoTn Dace	2011-07-23V20:50:56
18	BpeGcrTmecer Freqt enGy	100s62
19	BpeGcral Wudch	24038s5
20	LTwebc Freqt enGy	-1917s8
21	Nt Gebt b	13C
22	AGqt ured Bwe	16384
23	BpeGcral Bwe	32768



	Parameter	Value
1	Title	Chen-I-P194solid
2	Comment	
3	Origin	Bruker BioSpin GmbH
4	Site	
5	Spectrometer	spect
6	Author	
7	Solvent	CD2Cl2
8	Temperature	298.2
9	Pulse Sequence	zg30
10	Experiment	1D
11	Number of Scans	18
12	Receiver Gain	144
13	Relaxation Delay	1.0000
14	Pulse Width	7.2500
15	Acquisition Time	2.7263
16	Acquisition Date	2011-08-04T09:54:00
17	Modification Date	2011-08-04T09:55:22
18	Spectrometer Frequency	399.92
19	Spectral Width	6009.6
20	Lowest Frequency	-421.2
21	Nucleus	1H
22	Acquired Size	16384
23	Spectral Size	32768



	Parameter	Value
1	Title	Chen-I-P194solid-13CNMR
2	Comment	
3	Origin	Bruker BioSpin GmbH
4	Site	
5	Spectrometer	spect
6	Author	
7	Solvent	CD2Cl2
8	Temperature	298.3
9	Pulse Sequence	zgpg30
10	Experiment	1D
11	Number of Scans	3794
12	Receiver Gain	1820
13	Relaxation Delay	0.7000
14	Pulse Width	8.5000
15	Acquisition Time	0.6816
16	Acquisition Date	2011-08-04T16:03:00
17	Modification Date	2011-08-04T17:34:12
18	Spectrometer Frequency	100.62
19	Spectral Width	24038.5
20	Lowest Frequency	-1918.2
21	Nucleus	13C
22	Acquired Size	16384
23	Spectral Size	32768