Stereoselective Synthesis of *Cis*- and *Trans*-Tetrasubstituted Vinyl Silanes Using a Silyl-Heck Strategy and Hiyama Conditions for Their Cross-Coupling

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Supporting Information

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1. General Experimental Details:

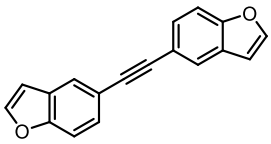
1,4-Dioxane, tetrahydrofuran (THF), and triethylamine (Et_3N) were dried on alumina according to published procedures[1]. Trimethylsilyl iodide was purchased from Gelest, distilled into a Straus flask containing copper beads, and stored under nitrogen. Dimethylbenzylsilyl iodide,[2] dimethylphenylsilyl iodide,[2] and diphenylmethylsilyl iodide[3] were prepared according to published procedures, distilled into a Straus flask containing copper beads, and stored under nitrogen. Bis(3,5-di-*tert*butylphenyl)(*tert*-butyl)phosphine (JessePhos),[4] $(JessePhos)_2PdCl_2$,[5] tris(3,5-di-*tert*butylphenyl)phosphine (DrewPhos),[3] $(DrewPhos)_2PdI_2$,[3] $(COD)Pd(CH_2TMS)_2$,[6] $[(C_6F_5)_3P]_2PdCl_2$,[7] (*E*)-6-iodo-2-hexene,[8] 2-(3-iodopropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane,[9] iodomethylbenzene,[10] bis(4-methoxyphenyl)acetylene,[11] bis(3-methylphenyl)acetylene,[12] bis(4-fluorophenyl)acetylene,[13] bis(4-chlorophenyl)acetylene,[13] bis(4-bromophenyl)acetylene,[13] bis(4-(trifluoromethyl)phenyl)acetylene,[13] bis(3-chlorophenyl)acetylene,[13] and bis(4-methylphenyl)acetylene[13] were prepared according to the published procedures. All other reagents were purchased in the highest purity from commercial suppliers and used as received.

Vials used in the glovebox were dried in a gravity oven at 140 °C for a minimum of 12 h, transferred into the glovebox hot, and then stored at rt in the glovebox prior to use. All hot glassware was oven dried for a minimum of four hours or flame-dried under vacuum prior to use. “Double manifold” refers to a standard Schlenk-line gas manifold equipped with nitrogen and vacuum (ca. 0.100 mm Hg). All optimization reactions (0.25 mmol) were charged in a nitrogen-filled glovebox and alkyl zinc halide was added on the bench via syringe then stirred on a magnetic stir plate. All yields in optimization reactions were determined using ^1H NMR with 1,3,5-trimethoxybenzene an internal standard and *syn*:*anti* ratios were determined using GC of unpurified products. All other reactions were set up using standard Schlenk technique and heated with stirring in temperature-controlled oil baths. **Note:** Any product yields listed in the main text that do not match those listed in the supporting information are the average of multiple isolated yields. The procedures listed below reflect yields from specific experimental runs. In this Supporting Information and in the main text, "dioxane" refers to 1,4-dioxane.

2. Instrumentation and Chromatography:

400 MHz ^1H, 101 MHz ^{13}C and 376 MHz ^{19}F NMR spectra were obtained on a 400 MHz FT-NMR spectrometer equipped with a Bruker CryoPlatform. 600 MHz ^1H, 151 MHz ^{13}C, 119 MHz ^{29}Si, 565 MHz ^{19}F, 193 MHz ^{11}B, and 243 MHz ^{31}P spectra were obtained on a 600 MHz FT-NMR spectrometer equipped with a Bruker SMART probe. All samples were analyzed in the indicated deutero-solvent and were recorded at ambient temperatures. All chemical shifts are reported in ppm. ^1H NMR spectra were calibrated using the residual protio-signal in deutero-solvents as a standard. ^{13}C NMR spectra were calibrated using the deutero-solvent as a standard. IR spectra were recorded on a Nicolet Magma-IR 560 FT-IR spectrometer as thin films on KBr plates. High resolution MS data was obtained on a Waters GCT Premier spectrometer using chemical ionization (CI), electron ionization (EI), or liquid injection field desorption ionization (LIFDI) or a Thermo Q-Exactive Orbitrap using electrospray ionization (ESI). Vacuum controller refers to J-Kem Digital Vacuum Regulator Model 200. Unless otherwise noted, column chromatography was performed either by hand or by use of Isolera 1 Biotage unit with 40-63 μm silica gel for normal phase and an Ultra C18 column for reverse phase. The eluent is reported in parentheses. Analytical thin-layer chromatography (TLC) was performed on silica gel (60 F254 Merck) pre-coated glass plates and visualized by UV or by staining with iodine, $KMnO_4$, or cerium ammonium molybdate stain.

3. Synthesis of Alkynes



S1
Chemical Formula: $C_{18}H_{10}O_2$
Exact Mass: 258.0681
Molecular Weight: 258.2760

(**S1**) **S1** was prepared through a modified version of the procedure described by Grieco[13]. An oven-dried 250 mL round bottom flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The septum was removed and the flask was charged with $(Ph_3P)_2PdCl_2$ (840 mg, 1.2 mmol, 6 mol %) and CuI (380 mg, 2.0 mmol, 10 mol %). The septum was then replaced and the flask was evacuated and refilled with nitrogen 3 times. Nitrogen-sparged benzene (100 mL) and nitrogen-sparged DBU (18 mL, 120 mmol, 6 equiv) were added to the flask via cannula addition. 5-bromobenzofuran (2.5 mL, 20 mmol, 1 equiv) was added to the flask via syringe. In rapid succession, trimethylsilylacetylene (1.38 mL, 10 mmol, 0.5 equiv), and nitrogen-sparged water (144 µL, 8.0 mmol, 0.4 equiv) were added to the flask via syringe. The flask allowed to stir at 80 °C in an oil bath for 48h; at the end of which, the flask was removed from the heat and cooled to rt. The reaction mixture was opened to air and transferred to a separatory funnel containing diethyl ether (50 mL) and water (25 mL). The partitioned solution was washed with 2 M aqueous hydrogen chloride solution (3 x 50 mL), water (25 mL), saturated aqueous sodium chloride solution (30 mL), dried over anhydrous magnesium sulfate, filtered, and concentrated *in vacuo.* The crude material was purified via flash column chromatography (1:99 to 5:95 ethyl acetate : hexanes) to provide **S1** as a pale yellow crystalline solid (1.4 g, 54%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (s, 2H), 7.66 (d, J = 2.2 Hz, 2H), 7.55 – 7.45 (m, 4H), 6.78 (d, J = 2.2 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 154.6, 145.9, 128.1, 127.7, 124.7, 118.1, 111.7, 106.6, 88.4.

FTIR (cm^{-1}): 3774, 1469, 1105, 1026.

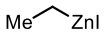
mp = 171 – 172 °C.

HRMS (LIFDI) m/z, calcd for $[C_{18}H_{10}O_2]^+$: 258.0681; found: 258.0687.

4. Synthesis of Alkylzinc Halides

General Procedure A:

An oven dried Schlenk flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the septum removed, and zinc dust (2 equiv) was added. The septum was replaced and the flask was attached to a double manifold and evacuated. Under vacuum, the zinc was heated for 5 minutes with a heat gun then allowed to cool to room temperature under vacuum. The flask was then backfilled with N_2 and dioxane [2 M] and 1,2-dibromoethane (0.03 equiv) were added. The flask was heated until reflux with a heat gun then allowed to cool. The process was repeated twice and after the flask was cooled to room temperature. Trimethylsilyl chloride (0.03 equiv) was added and the flask was stirred for 15 mins before the alkyl iodide (1 equiv) was added. The flask was then stirred in an oil bath at 100 °C for the indicated time. Conversion of starting halide was monitored via GC by quenching reaction aliquots with saturated NH_4Cl solution and extracting with Et_2O. Once all starting halide was consumed, the excess zinc was allowed to settle while the flask cooled. The mixture was cannula filtered to a Schlenk tube. If insoluble particles persisted, filtration through a 0.2 µm PTFE syringe filter was employed. Solutions were then titrated with I_2 according to the literature procedure by Knochel.[14]

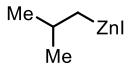


S2
Chemical Formula: C_2H_5IZn
Exact Mass: 219.8727
Molecular Weight: 221.3465

(**S2**) According to general procedure A, 100 mL Schlenk flask was charged with zinc dust (13 g, 200 mmol), dioxane (40 mL), trimethylsilyl chloride (380 µL), and 1-iodoethane (8.0 mL, 100 mmol). No 1,2-dibromoethane was added. The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [1.9 M] solution of ethylzinc iodide in dioxane.

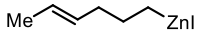
S3
Chemical Formula: C_4H_9IZn
Exact Mass: 247.9040
Molecular Weight: 249.4005

(**S3**) According to general procedure A, 100 mL Schlenk flask was charged with zinc dust (16 g, 250 mmol), dioxane (40 mL), trimethylsilyl chloride (475 µL), and 1-iodobutane (15 mL, 125 mmol). No 1,2-dibromoethane was added. The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [2.0 M] solution of *n*-butylzinc iodide in dioxane.



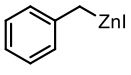
S4
Chemical Formula: C_4H_9IZn
Exact Mass: 247.9040
Molecular Weight: 249.4005

(**S4**) According to general procedure A, 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), trimethylsilyl chloride (76 µL), and 1-iodo-2-methylpropane (1.9 mL, 20 mmol). No 1,2-dibromoethane was added. The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [1.0 M] solution of isobutylzinc iodide in dioxane.



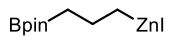
S5
Chemical Formula: $C_6H_{11}IZn$
Exact Mass: 273.9197
Molecular Weight: 275.4385

(**S5**) According to general procedure A, 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), 1,2-dibromoethane (75 µL), trimethylsilyl chloride (75 µL), and (*E*)-6-iodo-2-hexene (4.2 g, 20 mmol). The flask was heated to 100 °C for 20 h. Filtration and titration resulted in a [0.93 M] solution of (*E*)-2-hexenezinc iodide in dioxane



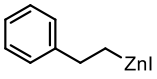
S6
Chemical Formula: C_7H_7IZn
Exact Mass: 281.8884
Molecular Weight: 283.4175

(**S6**) According to general procedure A, 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), trimethylsilyl chloride (75 µL), and iodomethylbenzene (4.3 g, 20 mmol). No 1,2-dibromoethane was added. The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [0.70 M] solution of benzylzinc iodide in dioxane.



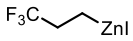
S7
Chemical Formula: $C_9H_{18}BIO_2Zn$
Exact Mass: 359.9736
Molecular Weight: 361.3355

(**S7**) According to general procedure A, 50 mL Schlenk flask was charged with zinc dust (2.1 g, 32 mmol), dioxane (15 mL), 1,2-dibromoethane (100 µL), trimethylsilyl chloride (100 µL), and 2-(3-iodopropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4.9 g, 16 mmol). The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [1.0 M] solution of 3-(4,4,5,5,-tetramethyl-1,3,2-dioxaborolan-2-yl)zinc iodide in dioxane.



S8
Chemical Formula: C_8H_9IZn
Exact Mass: 295.9040
Molecular Weight: 297.4445

(**S8**) According to general procedure A, 25 mL Schlenk flask was charged with zinc dust (3.9 g, 60 mmol), dioxane (15 mL), 1,2-dibromoethane (100 µL), trimethylsilyl chloride (100 µL), and (2-iodoethyl)-benzene (4.3 mL, 30 mmol). The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [1.2 M] solution of phenethylzinc iodide in dioxane.



S9
Chemical Formula: $C_3H_4F_3IZn$
Exact Mass: 287.8601
Molecular Weight: 289.3447

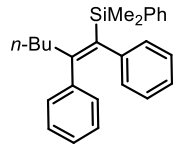
(S9) According to general procedure A, 50 mL Schlenk flask was charged with zinc dust (3.9 g, 60 mmol), dioxane (20 mL), 1,2-dibromoethane (155 µL), trimethylsilyl chloride (113 µL), and 1-iodo-3,3,3-trifluoropropane (3.5 mL, 30 mmol). The flask was heated to 100 °C for 24 h. Filtration and titration resulted in a [1.24 M] solution of (3,3,3-trifluoropropyl)zinc iodide in dioxane.

5. Synthesis of Tetrasubstituted Vinylsilanes via Multicomponent Carbosilylation Reactions

General Procedure B: Aryl Alkynes

Note: THF was used to quench certain reactions to avoid disiloxane formation upon aqueous workup. This quench generates the more easily separated (4-iodobutoxy)silane through silyl-iodide induced ring opening of THF.

A 25 mL Schlenk flask equipped with a magnetic stirbar and rubber septum was flame-dried, allowed to cool to room temperature under vacuum, and refilled with N_2. The flask was briefly opened, charged with the given alkyne (1 equiv) and palladium precatalyst (0.02 equiv), and the septum was replaced. The flask was evacuated and refilled with nitrogen 3 times. Dioxane, triethylamine (3 equiv), and silyl-iodide (3 equiv) were added sequentially via syringe at room temperature with stirring. A solution of alkylzinc iodide in dioxane (1.5 equiv) was then added dropwise over 4 h via syringe pump. The reaction was allowed to stir at room temperature for 0.25 h after the addition was completed. The reaction was quenched as indicated and opened to air. The resultant mixture was transferred to a separatory funnel and partitioned between 1 M aqueous hydrogen chloride solution (3 mL) to solubilize the zinc salts, water (10 mL) and diethyl ether (20 mL). The aqueous layer was extracted with diethyl ether (3 x 15 mL). The combined organic layers were washed with saturated aqueous sodium chloride solution (30 mL), dried over anhydrous magnesium sulfate, filtered, and concentrated *in vacuo*. A small aliquot of the crude reaction mixture (~20 µL) was analyzed by gas chromatography or NMR spectroscopy to determine the *syn*:*anti* ratio. The crude material was purified via silica column chromatography in the given solvent combination, followed by reverse phase column chromatography on a Biotage instrument in the given solvent combination.



2
Chemical Formula: $C_{26}H_{30}Si$
Exact Mass: 370.2117
Molecular Weight: 370.6110

(**2**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), diphenyl acetylene (357 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.5 M], 2.0 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **2** as a clear colorless oil (702 mg, 95%).

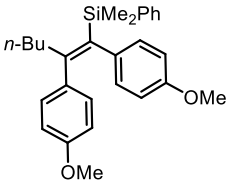
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 – 7.64 (m, 2H), 7.44 – 7.36 (m, 3H), 7.04 (q, *J* = 7.8 Hz, 4H), 6.98 (t, *J* = 7.3 Hz, 1H), 6.92 (dd, *J* = 11.7, 7.3 Hz, 3H), 6.81 (d, *J* = 7.1 Hz, 2H), 2.50 – 2.37 (m, 2H), 1.16 – 0.91 (m, 4H), 0.65 (t, *J* = 7.1 Hz, 3H), 0.28 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 156.2, 144.6, 143.2, 140.3, 138.2, 133.9, 129.4, 128.9, 128.8, 128.0, 127.4, 127.3, 125.8, 124.6, 38.7, 30.7, 22.8, 14.0, 0.0.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.6.

FTIR (cm^{-1}): 3067, 3019, 2956, 2859, 1587, 1486, 1427, 1249, 1112, 834, 809, 699.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{30}Si]^+$: 370.2117; found: 370.2108.



3
Chemical Formula: $C_{28}H_{34}O_2Si$
Exact Mass: 430.2328
Molecular Weight: 430.6630

(**3**) According to the general procedure B $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-methoxyphenyl)acetylene (476 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.9 M], 1.6 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 91:9 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (1:1 to 0 : 100 water : acetonitrile) to afford **3** as a clear colorless oil (692 mg, 81%).

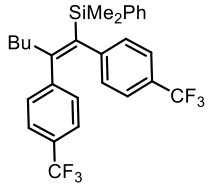
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 – 7.26 (m, 2H), 7.22 (q, *J* = 8.6, 8.1 Hz, 3H), 6.97 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 6.72 (d, *J* = 8.6 Hz, 2H), 3.81 (s, 3H), 3.80 (s, 3H), 2.22 – 2.00 (m, 2H), 1.13 – 0.96 (m, 4H), 0.64 (t, *J* = 7.1 Hz, 3H), -0.14 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 158.6, 157.4, 156.1, 140.6, 139.3, 136.8, 136.5, 134.0, 130.1, 129.7, 128.3, 127.4, 113.4, 113.1, 55.4, 55.3, 37.1, 30.4, 22.6, 14.0, -0.8.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -12.7.

FTIR (cm^{-1}): 2954, 2831, 2361, 1607, 1508, 1242, 1173, 1108, 1037, 834, 811, 701.

HRMS (LIFDI) m/z, calcd for $[C_{28}H_{34}O_2Si]^+$: 430.2328; found: 430.2325.



4
Chemical Formula: $C_{28}H_{28}F_6Si$
Exact Mass: 506.1864
Molecular Weight: 506.6074

(**4**) According to the general procedure B, $[(C_6F_5)P]_2PdCl_2$ (51 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-(trifluoromethyl)phenyl)acetylene (628 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([2.0 M], 1.6 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **4** as a white solid (866 mg, 85%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 – 7.68 (m, 2H), 7.56 – 7.45 (m, 3H), 7.40 (dd, *J* = 10.2, 8.4 Hz, 4H), 7.07 (d, *J* = 8.1 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 2.52 (t, *J* = 7.5 Hz, 2H), 1.18 – 1.00 (m, 4H), 0.74 (t, *J* = 6.8 Hz, 3H), 0.38 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) 155.5, 148.0 (q, *J* = 1.0 Hz), 146.3 (q, *J* = 1.0 Hz), 139.1, 139.0, 133.8, 129.3, 129.3, 128.9, 128.2, 127.2 (q, *J* = 32.3 Hz), 127.2 (q, *J* = 32.3 Hz), 124.7 (q, *J* = 3.0 Hz), 124.6 (q, *J* = 3.4 Hz), 124.4 (q, *J* = 271.8 Hz), 124.2 (q, *J* = 271.7 Hz), 38.4, 30.5, 22.8, 13.9, -0.3.

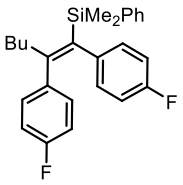
^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -62.3, -62.5.

FTIR (cm^{-1}): 3070, 2959, 2931, 2861, 1614, 1326, 1165, 1124, 1068, 1018, 845, 815, 701.

mp = 46 – 48 °C.

HRMS (LIFDI) m/z, calcd for $[C_{28}H_{28}F_6Si]^+$: 506.1864; found: 506.1866.



5
Chemical Formula: $C_{26}H_{28}F_2Si$
Exact Mass: 406.1928
Molecular Weight: 406.5918

(**5**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-fluorophenyl)acetylene (428 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.10 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.5 M], 2.0 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **5** as a white solid (571 mg, 70%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 – 7.59 (m, 2H), 7.49 – 7.33 (m, 3H), 6.84 (dd, *J* = 8.0, 5.9 Hz, 2H), 6.82 – 6.64 (m, 6H), 2.50 – 2.22 (m, 2H), 1.11 – 0.85 (m, 4H), 0.65 (t, *J* = 6.7 Hz, 3H), 0.28 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 161.6 (d, *J* = 76.9 Hz), 160.0 (d, *J* = 75.5 Hz), 155.9, 140.3 (d, *J* = 3.2 Hz), 139.8, 138.8 (d, *J* = 3.2 Hz), 138.0, 133.8, 130.6 (d, *J* = 7.7 Hz), 130.2 (d, *J* = 7.8 Hz), 129.1, 128.0, 114.5 (d, *J* = 6.4 Hz), 114.4 (d, *J* = 6.4 Hz), 38.6, 30.7, 22.8, 14.0, -0.1.

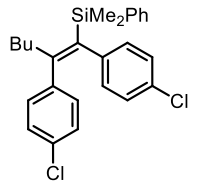
^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -116.7, -118.7.

FTIR (cm^{-1}): 3068, 2957, 2929, 2860, 1504, 1221, 1157, 834, 816, 701.

mp = 55 – 56 °C.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{28}F_2Si]^+$: 406.1928; found: 406.1932.



6
Chemical Formula: $C_{26}H_{28}Cl_2Si$
Exact Mass: 438.1337
Molecular Weight: 439.4950

(**6**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-chlorophenyl)acetylene (552 mg, 2 mmol, 1 equiv), dioxane (20 mL, 0.1 [M]), triethylamine (836 µL, 6 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.9 M], 1.57 mL, 3 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **6** as a white crystalline solid (542 mg, 62%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.59 (m, 2H), 7.42 – 7.35 (m, 3H), 7.04 (d, *J* = 8.3 Hz, 4H), 6.81 (d, *J* = 8.3 Hz, 2H), 6.71 (d, *J* = 8.3 Hz, 2H), 2.38 (t, *J* = 7.5 Hz, 2H), 0.99 (dt, *J* = 7.5, 3.7 Hz, 4H), 0.68 – 0.60 (m, 3H), 0.27 (s, 6H).

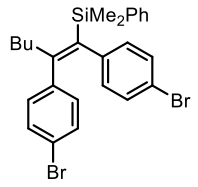
^{13}C NMR (101 MHz, $CDCl_3$) δ 155.6, 142.8, 141.2, 139.6, 138.1, 133.8, 131.8, 130.6, 130.5, 130.0, 129.1, 128.1, 127.9, 127.8, 38.5, 30.6, 22.8, 14.0, -0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.5.

FTIR (cm^{-1}): 2956, 2928, 1486, 1250, 1111, 1090, 1014, 836, 811, 774, 700, 492.

mp = 60 – 61 °C.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{28}SiCl_2]^+$: 438.1337; found: 438.1315.



7
Chemical Formula: $C_{26}H_{28}Br_2Si$
Exact Mass: 526.0327
Molecular Weight: 528.4030

(**7**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (14 mg, 0.02 mmol, 2 mol %), 1,2-bis(4-bromophenyl)acetylene (314 mg, 1 mmol, 1 equiv), dioxane (10 mL, 0.1 [M]), triethylamine (418 µL, 3 mmol, 3 equiv), and dimethylphenylsilyl iodide (560 µL, 3 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.9 M], 790 µL, 1.5 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **7** as a clear pale yellow oil (335 mg, 63%).

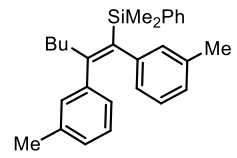
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 6.5, 2.9 Hz, 2H), 7.41 – 7.39 (m, 3H), 7.21 (dd, *J* = 8.3, 6.4 Hz, 4H), 6.78 (d, *J* = 8.4 Hz, 2H), 6.68 (d, *J* = 8.3 Hz, 2H), 2.40 (t, *J* = 7.5 Hz, 2H), 1.02 – 0.98 (m, 4H), 0.65 (t, *J* = 6.9 Hz, 3H), 0.29 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 155.5, 143.3, 141.7, 139.5, 138.2, 133.8, 130.9, 130.8, 130.8, 130.4, 129.1, 128.1, 120.1, 118.9, 38.4, 30.6, 22.7, 13.9, -0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.5.

FTIR (cm^{-1}): 2956, 2928, 1483, 1249, 1110, 1072, 1010, 835, 812, 796, 732, 701, 515.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{28}Br_2Si]^+$: 526.0327; found: 526.0328.



8
Chemical Formula: $C_{28}H_{34}Si$
Exact Mass: 398.2430
Molecular Weight: 398.6650

(**8**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(3-methylphenyl)acetylene (413 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.9 M], 1.6 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **8** as a clear colorless oil (621 mg, 81%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 7.4, 1.8 Hz, 2H), 7.47 – 7.34 (m, 3H), 6.92 (q, *J* = 7.6 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 2H), 6.77 – 6.66 (m, 3H), 6.66 – 6.55 (m, 1H), 2.42 – 2.34 (m, 2H), 2.17 (s, 6H), 1.15 – 0.86 (m, 4H), 0.68 – 0.62 (m, 3H), 0.27 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 156.0, 144.5, 143.1, 140.5, 137.9, 136.5, 133.9, 130.2, 129.5, 128.8, 127.9, 127.2, 127.0, 126.5, 126.4, 125.9, 125.2, 38.7, 30.8, 22.9, 21.5, 21.5, 14.0, 0.0.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.8.

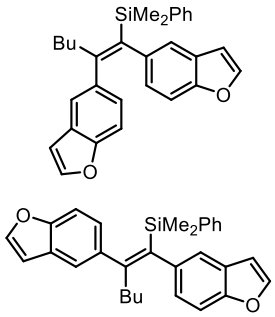
^{1}H NMR (400 MHz, C_6D_6) δ 7.81 (dd, *J* = 8.0, 1.4 Hz, 2H), 7.36 – 7.29 (m, 3H), 7.28 – 7.21 (m, 1H), 7.02 – 6.93 (m, 1H), 6.94 – 6.78 (m, 5H), 6.76 – 6.62 (m, 2H), 2.66 – 2.50 (m, 1H), 2.01 (d, *J* = 13.5 Hz, 6H), 1.24 (p, *J* = 7.7 Hz, 2H), 1.02 (h, *J* = 7.3 Hz, 2H), 0.65 (t, *J* = 7.3 Hz, 1H), 0.42 (s, 6H).

^{13}C NMR (101 MHz, C_6D_6) δ 156.4, 144.9, 143.3, 140.6, 138.4, 137.0, 136.9, 134.2, 134.2, 130.3, 129.6, 129.2, 128.4, 127.8, 127.6, 127.1, 126.9, 126.4, 125.9, 39.1, 31.2, 23.1, 21.5, 21.4, 14.1, 0.2.

^{29}Si NMR (119 MHz, C_6D6) δ -11.8.

FTIR (cm^{-1}): 3049, 3009, 2955, 2925, 2859, 1600, 1483, 1427, 1248, 1112, 826, 780, 702, 469.

HRMS (LIFDI) m/z, calcd for $[C_{28}H_{34}Si]^+$: 398.2430; found: 398.2431.



***anti*-9**
Chemical Formula: $C_{30}H_{30}O_2Si$
Exact Mass: 450.2015
Molecular Weight: 450.6530

(**9**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(5-benzofuran)acetylene (516 mg, 2 mmol, 1 equiv), dioxane (20 mL, 0.1 [M]), triethylamine (836 µL, 6 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.9 M], 1.57 mL, 3 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 70:30 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford the separable products ***syn*-9** as a clear pale yellow oil (353 mg, 40%) and ***anti*-9** as a clear pale yellow oil (130 mg, 15 %).

***Syn*-9:**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 – 7.71 (m, 2H), 7.45 (t, *J* = 2.4 Hz, 2H), 7.42 (dd, *J* = 5.9, 1.2 Hz, 3H), 7.19 (d, *J* = 1.3 Hz, 1H), 7.15 (t, *J* = 8.7 Hz, 2H), 7.04 (d, *J* = 1.3 Hz, 1H), 6.87 (dd, *J* = 8.5, 1.6 Hz, 1H), 6.79 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.57 (ddd, *J* = 5.8, 2.1, 0.8 Hz, 2H), 2.53 – 2.46 (m, 2H), 1.13 – 0.92 (m, 4H), 0.64 (t, *J* = 7.0 Hz, 3H), 0.28 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.5, 153.2, 152.8, 144.7, 144.5, 140.4, 139.3, 138.1, 137.9, 133.9, 128.9, 128.0, 127.9, 126.8, 126.6, 126.2, 125.7, 121.2, 120.9, 110.2, 110.1, 106.7, 106.6, 39.3, 30.8, 22.9, 0.0.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.5.

FTIR (cm^{-1}): 2955, 2926, 2857, 1592, 1535, 1465, 1428, 1260, 1200, 1130, 1109, 1031, 832, 770, 737, 701.

HRMS (LIFDI) m/z, calcd for $[C_{30}H_{30}O_2Si]^+$: 450.2015; found: 450.2021.

***Anti*-9**

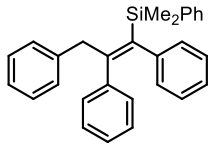
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (t, *J* = 2.2 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.26 – 7.13 (m, 7H), 7.03 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.97 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.76 (dd, *J* = 2.1, 0.8 Hz, 1H), 6.66 (dd, *J* = 2.1, 0.8 Hz, 1H), 2.22 – 2.14 (m, 2H), 1.13 – 1.04 (m, 2H), 0.99 (dt, *J* = 14.3, 6.9 Hz, 2H), 0.59 (t, *J* = 7.2 Hz, 3H), -0.19 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.6, 154.2, 153.4, 145.2, 145.0, 140.4, 140.2, 139.1, 138.5, 133.8, 128.3, 127.3, 127.3, 126.8, 125.4, 125.4, 121.8, 120.6, 110.7, 110.5, 106.8, 106.8, 37.3, 30.4, 22.6, 14.0, -0.9.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -12.9.

FTIR (cm^{-1}): 2955, 2928, 2858, 1591, 1537, 1461, 1428, 1261, 1228, 1130, 1109, 1032, 883, 853, 833, 770, 735, 701.

HRMS (LIFDI) m/z, calcd for $[C_{30}H_{30}O_2Si]^+$: 450.2015; found: 450.2025.



10
Chemical Formula: $C_{29}H_{28}Si$
Exact Mass: 404.1960
Molecular Weight: 404.6280

(**10**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), diphenyl acetylene (356 mg, 2 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6 mmol, 3 equiv) were combined under nitrogen. Benzylzinc iodide **S6** in dioxane ([1.9 M], 1.58 mL, 3 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **10** as a white crystalline solid (634 mg, 78%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.42 – 7.39 (m, 3H), 7.06 (dd, *J* = 10.3, 7.6 Hz, 5H), 6.95 (d, *J* = 7.3 Hz, 1H), 6.93 – 6.83 (m, 7H), 6.79 – 6.74 (m, 2H), 3.84 (s, 2H), 0.34 (s, 6H).

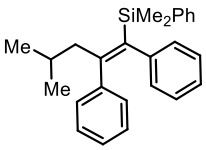
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.3, 144.5, 142.4, 140.9, 139.9, 138.9, 134.0, 129.3, 129.1, 129.1, 129.1, 128.2, 128.0, 127.5, 127.1, 125.9, 125.8, 124.8, 44.3, 0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.4.

FTIR (cm^{-1}): 3063, 3024, 2954, 1597, 1494, 1427, 1249, 1111, 834, 810, 786, 731, 698.

mp = 72 – 73 °C.

HRMS (LIFDI) m/z, calcd for $[C_{29}H_{28}Si]^+$: 404.1960; found: 404.1957.



11
Chemical Formula: $C_{26}H_{30}Si$
Exact Mass: 370.2117
Molecular Weight: 370.6110

(**11**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), diphenylacetylene (356 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. Isobutylzinc iodide **S4** in dioxane ([1.0 M], 2.9 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 92:8 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **11** as a white solid (687 mg, 93%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.61 (m, 2H), 7.41 (dd, *J* = 4.9, 1.8 Hz, 3H), 7.11 – 7.02 (m, 4H), 7.02 – 6.94 (m, 2H), 6.92 (dd, *J* = 8.2, 1.4 Hz, 2H), 6.82 (dd, *J* = 8.1, 1.2 Hz, 2H), 2.40 (d, *J* = 7.3 Hz, 2H), 1.33 (p, *J* = 7.1 Hz, 1H), 0.64 (d, *J* = 6.6 Hz, 6H), 0.30 (s, 6H).

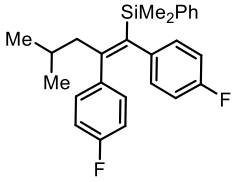
^{13}C NMR (101 MHz, $CDCl_3$) δ 154.8, 144.7, 142.8, 140.3, 139.8, 134.1, 129.5, 128.9, 128.7, 127.9, 127.4, 127.3, 125.7, 124.6, 46.6, 26.4, 22.1, 0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.9.

FTIR (cm^{-1}): 2955, 1586, 1487, 1427, 1248, 1112, 833, 810, 699, 471.

mp = 71 °C.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{30}Si]^+$: 370.2117; found: 370.2119.



12
Chemical Formula: $C_{26}H_{28}F_2Si$
Exact Mass: 406.1928
Molecular Weight: 406.5918

(**12**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-fluorophenyl)acetylene (428 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. Isobutylzinc iodide **S4** in dioxane ([0.7 M], 4.6 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **12** as a white solid (667 mg, 82%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, *J* = 6.5, 2.9 Hz, 2H), 7.46 – 7.33 (m, 3H), 6.83 (dd, *J* = 8.6, 5.6 Hz, 2H), 6.78 – 6.68 (m, 6H), 2.36 (d, *J* = 7.3 Hz, 2H), 1.31 (hept, *J* = 7.0 Hz, 1H), 0.62 (d, *J* = 6.6 Hz, 6H), 0.29 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.9 (d, *J* = 44.9 Hz), 159.5 (d, *J* = 43.2 Hz), 154.5, 140.4 (d, *J* = 3.4 Hz), 139.8, 139.5, 138.5 (d, *J* = 3.4 Hz), 134.0, 130.7 (d, *J* = 7.7 Hz), 130.1 (d, *J* = 7.8 Hz), 129.1, 128.0, 114.5 (d, *J* = 7.0 Hz), 114.3 (d, *J* = 7.0 Hz), 46.5, 26.4, 22.0, 0.1.

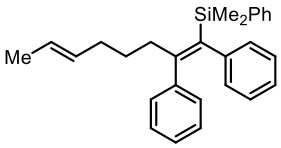
^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -116.6, -118.6.

FTIR (cm^{-1}): 3068, 2957, 2869, 1602, 1503, 1249, 1221, 1157, 835, 816, 798, 700, 531.

mp = 73 – 74 °C.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{28}F_2Si]^+$: 406.1928; found: 406.1945.



13
Chemical Formula: $C_{28}H_{32}Si$
Exact Mass: 396.2273
Molecular Weight: 396.6490

(**13**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), diphenyl acetylene (356 mg, 2 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6 mmol, 3 equiv) were combined under nitrogen. (*E*)-2-Hexenezinc iodide **S5** in dioxane ([0.93 M], 3.3 mL, 3 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **13** as a clear pale yellow oil (453 mg, 57%).

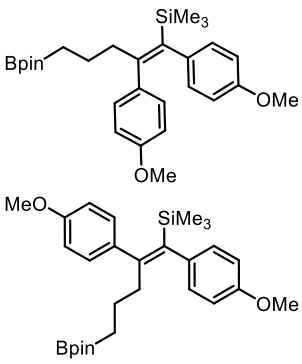
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.69 (m, 2H), 7.43 (dd, *J* = 5.0, 1.9 Hz, 3H), 7.10 – 7.04 (m, 4H), 7.00 (t, *J* = 7.3 Hz, 1H), 6.98 – 6.91 (m, 3H), 6.87 – 6.83 (m, 2H), 5.31 – 5.19 (m, 1H), 5.19 – 5.10 (m, 1H), 2.57 – 2.25 (m, 2H), 1.67 (q, *J* = 7.3 Hz, 2H), 1.58 (d, *J* = 7.2 Hz, 3H), 1.24 – 1.06 (m, 2H), 0.31 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 155.9, 144.5, 143.0, 140.3, 138.4, 133.9, 131.1, 129.3, 128.9, 128.8, 128.0, 127.4, 127.3, 125.8, 125.0, 124.6, 38.4, 32.7, 28.5, 18.0, -0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.6.

FTIR (cm^{-1}): 3067, 3019, 2954, 2856, 1586, 1486, 1440, 1427, 1249, 1112, 966, 834, 811, 730, 699.

HRMS (LIFDI) m/z, calcd for $[C_{28}H_{32}Si]^+$: 396.2273; found: 396.2256.



15
Chemical Formula: $C_{28}H_{41}O_2Si$
Exact Mass: 353.1937
Molecular Weight: 353.5570

(**15**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-methoxyphenyl)acetylene (476 mg, 2 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6 mmol, 3 equiv), and trimethylsilyl iodide (853 µL, 6 mmol, 3 equiv) were combined under nitrogen. 3-(4,4,5,5,-tetramethyl-1,3,2-dioxaborolan-2-yl)zinc iodide **S7** in dioxane ([0.55 M], 5.46 mL, 3 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 50:50 *syn*:*ant*i ratio. The product was purified by silica gel chromatography (1 : 99 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **15** as a clear pale yellow oil (688 mg, 97%, mixture of *syn* and *anti* isomers).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.7 Hz, 2H), 6.93 – 6.78 (m, 8H), 6.64 – 6.53 (m, 6H), 3.83 (s, 3H), 3.81 (s, 3H), 3.69 (s, 6H), 2.65 – 2.49 (m, 2H), 2.19 – 2.08 (m, 2H), 1.39 (p, *J* = 8.0 Hz, 2H), 1.21 (s, 14H), 1.11 (s, 12H), 0.76 (t, *J* = 7.8 Hz, 2H), 0.52 (t, *J* = 7.8 Hz, 2H), 0.11 (s, 9H), -0.36 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 157.2, 157.2, 156.5, 154.0, 153.8, 141.2, 139.6, 137.6, 137.1, 136.9, 135.8, 130.1, 130.0, 130.0, 129.3, 113.4, 113.1, 112.7, 112.6, 83.0, 82.9, 55.4, 55.2, 55.1, 40.9, 39.5, 25.0, 24.8, 23.3, 22.3, 1.2, 0.5.

^{11}B NMR (193 MHz, $CDCl_3$) δ -34.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -7.8, -7.6.

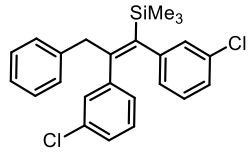
FTIR (cm^{-1}): 2976, 2951, 1608, 1508, 1372, 1243, 1173, 1145, 1036, 838.

mp = 72 – 73 °C.

HRMS (FD) m/z, calcd for $[C_{28}H_{41}BO_4Si]^+$: 480.2867; found: 480.2879.

FTIR (cm^{-1}): 3067, 3019, 2956, 2928, 2859, 1486, 1427, 1248, 1112, 834, 810, 730, 699.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{28}SiCl_2]^+$: 438.1337; found: 438.1315.



16
Chemical Formula: $C_{24}H_{24}Cl_2Si$
Exact Mass: 410.1024
Molecular Weight: 411.4410

(**16**) According to the general procedure B, $(Ph_3P)_2PdCl_2$ (28 mg, 0.04 mmol, 2 mol %), 1,2-bis(3-chlorophenyl)acetylene (494 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and trimethylsilyl iodide (853 µL, 6.0 mmol, 3 equiv) were combined under nitrogen. Benzylzinc iodide **S6** in dioxane ([0.8 M], 3.8 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 94:6 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 1 : 9 water : acetonitrile) to afford **16** as a white solid (622 mg, 76%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.5 Hz, 2H), 7.13 (dd, *J* = 16.0, 7.3 Hz, 3H), 7.00 (t, *J* = 7.8 Hz, 1H), 6.95 (dt, *J* = 8.0, 1.5 Hz, 1H), 6.89 – 6.74 (m, 4H), 6.67 (dd, *J* = 7.5, 1.2 Hz, 1H), 6.60 – 6.54 (m, 1H), 3.98 (s, 5H), 0.20 (s, 9H).

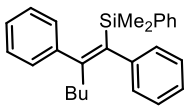
^{13}C NMR (151 MHz, $CDCl_3$) δ 151.0, 146.2, 144.1, 143.0, 138.3, 133.5, 133.2, 129.0, 128.9, 128.8, 128.7, 128.5, 128.4, 127.4, 127.1, 126.4, 126.2, 125.2, 43.7, 1.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -6.5.

FTIR (cm^{-1}): 2953, 1589, 1560, 1251, 933, 839, 789, 762, 696.

mp = 82 – 83 °C.

HRMS (LIFDI) m/z, calcd for $[C_{24}H_{24}Cl_2Si]^+$: 410.1024; found: 410.1024.



28
Chemical Formula: $C_{26}H_{30}Si$
Exact Mass: 370.2117
Molecular Weight: 370.6110

(**28**) According to the general procedure B, $(JessePhos)_2PdCl_2$ (44 mg, 0.04 mmol, 2 mol %), diphenylacetylene (357 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and dimethylphenylsilyl iodide (1.12 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([2.0 M], 1.6 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 13:87 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column

chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **28** as a clear colorless oil (565 mg, 76%).

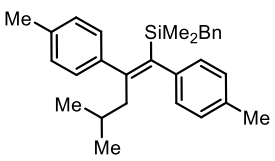
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (q, *J* = 6.0, 4.7 Hz, 5H), 7.28 – 7.23 (m, 5H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.16 – 7.09 (m, 2H), 7.04 – 7.00 (m, 2H), 2.26 – 2.11 (m, 2H), 1.10 (ddt, *J* = 35.4, 14.7, 7.1 Hz, 4H), 0.67 (t, *J* = 7.2 Hz, 3H), -0.12 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 156.0, 144.4, 143.9, 140.3, 139.9, 134.0, 129.1, 128.8, 128.4, 127.9, 127.8, 127.4, 126.9, 125.3, 37.1, 30.3, 22.6, 13.9, -1.0.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -12.6.

FTIR (cm^{-1}): 3068, 3022, 2956, 2927, 2858, 1596, 1442, 1427, 1247, 1110, 833, 812, 770, 701, 471.

HRMS (LIFDI) m/z, calcd for $[C_{26}H_{30}Si]^+$: 370.2117; found: 370.2173.



29
Chemical Formula: $C_{29}H_{36}Si$
Exact Mass: 412.2586
Molecular Weight: 412.6920

(**29**) According to the general procedure B, $(JessePhos)_2PdCl_2$ (44 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-methylphenyl)acetylene (413 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (0.836 mL, 6.0 mmol, 3 equiv), and dimethylbenzylsilyl iodide (1.13 mL, 6.0 mmol, 3 equiv) were combined under nitrogen. Isobutylzinc iodide **S4** in dioxane ([1.3 M], 2.5 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a <5:95 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **29** as a white solid (208 mg, 50%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.2 – 7.1 (m, 6H), 7.1 (d, *J* = 8.0 Hz, 2H), 7.0 – 7.0 (m, 1H), 6.9 – 6.8 (m, 4H), 2.4 (d, *J* = 5.6 Hz, 6H), 2.1 (d, *J* = 7.2 Hz, 2H), 1.8 (s, 2H), 1.3 (hept, *J* = 6.8 Hz, 1H), 0.7 (d, *J* = 6.6 Hz, 6H), -0.5 (s, 6H).

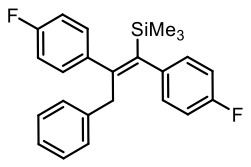
^{13}C NMR (101 MHz, $CDCl_3$) δ 154.4, 141.5, 141.3, 140.8, 140.7, 136.6, 134.5, 128.8, 128.7, 128.6, 128.6, 128.5, 128.1, 123.9, 45.9, 26.4, 26.0, 22.5, 21.4, 21.3, -1.6.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -7.4.

FTIR (cm^{-1}): 3024, 2955, 2928, 2869, 1600, 1493, 1452, 1248, 1205, 1154, 833, 761, 698.

mp = 89 – 90 °C

HRMS (LIFDI) m/z, calcd for $[C_{24}H_{24}F_2Si]^+$: 412.2586; found: 412.2586.



30
Chemical Formula: $C_{24}H_{24}F_2Si$
Exact Mass: 378.1615
Molecular Weight: 378.5378

(**30**) According to the general procedure B, $(JessePhos)_2PdCl_2$ (44 mg, 0.04 mmol, 2 mol %), 1,2-bis(4-fluorophenyl)acetylene (428 mg, 2.0 mmol, 1 equiv), dioxane (1 mL, 2.0 [M]), triethylamine (836 µL, 6.0 mmol, 3 equiv), and trimethylsilyl iodide (853 µL, 6.0 mmol, 3 equiv) were combined under nitrogen. Benzylzinc iodide **S6** in dioxane ([0.8 M], 3.8 mL, 3.0 mmol, 1.5 equiv) was added over 4 h. The reaction was quenched with water, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a <5:95 *syn:anti* ratio. The product was purified by silica gel chromatography (1 : 99 to 3 : 47 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (7 : 13 to 0 : 100 water : acetonitrile) to afford **30** as a white solid (544 mg, 72%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.14 – 7.02 (m, 7H), 6.96 (dd, *J* = 8.7, 5.6 Hz, 2H), 6.90 (t, *J* = 8.7 Hz, 2H), 6.80 – 6.71 (m, 2H), 3.48 (s, 3H), -0.32 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.8 (d, *J* = 84.9 Hz), 160.4 (d, *J* = 83.2 Hz), 151.7, 142.5, 140.1 (d, *J* = 3.4 Hz), 139.1 (d, *J* = 3.3 Hz), 138.9, 130.8 (d, *J* = 7.9 Hz), 129.7 (d, *J* = 7.7 Hz), 129.2, 128.1, 125.9, 115.2 (d, *J* = 21.1 Hz), 114.6 (d, *J* = 21.1 Hz), 43.4, 0.3.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -6.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -115.6, -117.9.

FTIR (cm^{-1}): 3029, 2956, 2896, 1601, 1506, 1246, 1223, 1155, 838, 826, 762, 700.

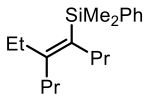
mp = 93 – 94 °C.

HRMS (LIFDI) m/z, calcd for $[C_{24}H_{24}F_2Si]^+$: 378.1615; found: 378.1612.

General Procedure C: Alkyl Alkynes

Note: THF was used to quench certain reactions to avoid disiloxane formation upon aqueous workup. This quench generates the more easily separated (4-iodobutoxy)silane through silyl-iodide induced ring opening of THF.

A 25 mL Schlenk flask equipped with a magnetic stirbar and rubber septum was flame-dried and allowed to cool to room temperature under vacuum and refilled with nitrogen. The flask was briefly opened, charged with palladium precatalyst (0.05 equiv), and the septum was replaced. The flask was evacuated and refilled with N_2 three times. Dioxane (0.5 mL), triethylamine (5 equiv), silyl-iodide (5 equiv), and alkyne (1 equiv) were added sequentially via syringe at room temperature with stirring. A solution of alkylzinc iodide in dioxane (5 equiv) was then added dropwise over 4 h via syringe pump. The reaction was allowed to stir at room temperature for 0.25 h after the addition was completed. The reaction was quenched as indicated and opened to air. The resultant mixture was transferred to a separatory funnel and partitioned between 1 N aqueous hydrogen chloride solution (3 mL) to solubilize the zinc salts, water (10 mL), and diethyl ether (20 mL). The aqueous layer was extracted with diethyl ether (3 x 15 mL). The combined organic layers were washed with saturated aqueous sodium chloride solution (30 mL) and dried over anhydrous magnesium sulfate, filtered, and concentrated *in vacuo*. A small aliquot of the crude reaction mixture (~20 µL) was analyzed by gas chromatography or NMR spectroscopy to determine the *syn:anti* ratio. The crude material was purified via silica column chromatography in the indicated solvent combination followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column in the indicated



16
Chemical Formula: $C_{18}H_{30}Si$
Exact Mass: 274.2117
Molecular Weight: 274.5230

(**16**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (156 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), dimethylphenylsilyl iodide (1.80 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 92:8 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **16** as a clear colorless oil (502 mg, 91%).

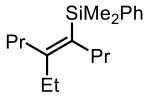
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.42 (m, 2H), 7.36 – 7.27 (m, 3H), 2.11 (td, *J* = 8.2, 2.9 Hz, 4H), 1.99 (q, *J* = 7.4 Hz, 2H), 1.42 (dd, 2H), 1.30 (dd, *J* = 15.9, 7.6 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.77 (t, *J* = 7.4 Hz, 3H), 0.35 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 155.0, 141.5, 133.9, 130.3, 128.5, 127.7, 34.2, 32.8, 29.9, 24.7, 22.4, 14.7, 14.5, 13.4, 0.2.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.1.

FTIR (cm^{-1}): 2959, 2930, 2870, 1595, 1464, 1428, 1248, 1107, 819, 770, 700.

HRMS (LIFDI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 274.2117; found: 274.2108.



17
Chemical Formula: $C_{18}H_{30}Si$
Exact Mass: 274.2117
Molecular Weight: 274.5230

(**17**) According to the general procedure C, $(JessePhos)_2PdCl_2$ (111 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), dimethylphenylsilyl iodide (1.80 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a <5 : 95 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **17** as a clear colorless oil (420 mg, 77%).

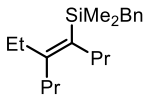
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 – 7.46 (m, 2H), 7.33 – 7.29 (m, 3H), 2.14 (q, *J* = 7.6 Hz, 2H), 2.12 – 2.07 (m, 2H), 1.96 – 1.89 (m, 2H), 1.29 (dt, *J* = 15.0, 7.4 Hz, 2H), 1.19 (dt, 2H), 1.00 (t, *J* = 7.5 Hz, 3H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.64 (t, *J* = 7.3 Hz, 3H), 0.36 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 155.4, 141.5, 133.9, 130.2, 128.5, 127.7, 38.9, 34.2, 24.7, 24.2, 22.3, 14.5, 14.2, 13.9, 0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.2.

FTIR (cm^{-1}): 2959, 2930, 2871, 1597, 1464, 1427, 1248, 1108, 818, 770, 700.

HRMS (LIFDI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 274.2117; found: 274.2119.



18
Chemical Formula: $C_{19}H_{32}Si$
Exact Mass: 288.2273
Molecular Weight: 288.5500

(**18**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (156 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), dimethylbenzylsilyl iodide (1.80 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3:17 water : acetonitrile) to afford **18** as a clear colorless oil (496 mg, 86%).

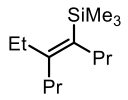
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 (t, *J* = 7.7 Hz, 2H), 7.04 (tt, *J* = 7.3, 1.3 Hz, 1H), 7.01 – 6.95 (m, 2H), 2.19 (s, 2H), 2.11 (q, *J* = 7.4 Hz, 2H), 2.09 – 2.02 (m, 2H), 1.97 – 1.86 (m, 2H), 1.47 – 1.29 (m, 2H), 1.13 (ddd, *J* = 12.4, 9.3, 5.7 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H), 0.85 (t, *J* = 7.3 Hz, 3H), 0.08 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 154.2, 140.8, 131.1, 128.5, 128.1, 123.9, 34.2, 33.0, 29.4, 27.5, 24.4, 22.3, 14.7, 14.5, 14.1, -0.8.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -6.8.

FTIR (cm^{-1}): 2959, 2930, 2871, 1600, 1493, 1453, 1248, 1206, 1155, 829, 760, 698, 477.

HRMS (LIFDI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 288.2273; found: 288.2263.



19
Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520

(**19**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (156 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), trimethylsilyl iodide (1.40 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **19** as a clear colorless oil (312 mg, 73%).

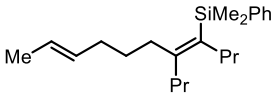
^{1}H NMR (600 MHz, $CDCl_3$) δ 2.13 (q, *J* = 7.4 Hz, 2H), 2.08 – 2.03 (m, 2H), 2.03 – 1.98 (m, 2H), 1.42 – 1.32 (m, 2H), 1.28 – 1.15 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.90 (dt, *J* = 10.9, 7.3 Hz, 6H), 0.12 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 153.0, 132.8, 34.1, 32.9, 29.3, 24.6, 22.4, 14.6, 14.5, 14.1, 1.3.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -7.9.

FTIR (cm^{-1}): 2959, 2931, 2871, 1465, 1248, 849, 835, 757.

HRMS (ESI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 213.2033; found: 213.1841.



20
Chemical Formula: $C_{22}H_{36}Si$
Exact Mass: 328.2586
Molecular Weight: 328.6150

(**20**) According to the general procedure C, $(DrewPhos)_2PdCl_2$ (78 mg, 0.05 mmol, 5 mol %), triethylamine (700 µL, 5 mmol, 5 equiv), dimethylphenylsilyl iodide (940 µL, 5 mmol, 5 equiv), and 4-octyne (146 µL, 1 mmol, 1 equiv) were combined under nitrogen. *No dioxane was added to the initial reaction mixture.* (*E*)-2-Hexenezinc iodide **S5** in dioxane ([1.26M], 3.9 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Palladium scavenger 1-pyrrolidinecarbodithioic acid ammonium salt was added to the drying organic solution. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (0 : 100 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (1 : 3 to 0 : 1 water : acetonitrile) to afford **20** as a clear colorless oil (220 mg, 67%).

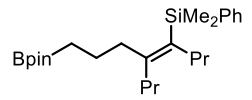
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, J = 6.4, 3.1 Hz, 2H), 7.35 – 7.28 (m, 3H), 5.33 – 5.15 (m, 2H), 2.15 – 2.07 (m, 4H), 1.97 – 1.89 (m, 2H), 1.65 (q, J = 7.4, 6.9 Hz, 2H), 1.59 (d, J = 5.6 Hz, 3H), 1.47 – 1.36 (m, 2H), 1.36 – 1.25 (m, 2H), 1.26 – 1.13 (m, 2H), 0.91 (dt, J = 14.3, 7.3 Hz, 6H), 0.36 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.7, 141.5, 133.8, 131.4, 131.0, 128.5, 127.7, 124.9, 36.8, 34.2, 33.4, 32.9, 29.1, 24.7, 22.4, 18.0, 14.6, 14.5, 0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.2.

FTIR (cm^{-1}): 2958, 2931, 2870, 1465, 1252, 849, 835, 750.

HRMS (FD) m/z, calcd for $[C_{22}H_{36}Si]^+$: 328.2586; found: 328.2594.



21
Chemical Formula: $C_{25}H_{43}BO_2Si$
Exact Mass: 414.3125
Molecular Weight: 414.5120

(**21**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (78 mg, 0.05 mmol, 5 mol %), triethylamine (700 µL, 5 mmol, 5 equiv), dimethylphenylsilyl iodide (935 µL, 5 mmol, 5 equiv), and 4-octyne (146 µL, 1 mmol, 1 equiv) were combined under nitrogen. *No dioxane was added to the initial reaction mixture*. Alkylzinc iodide **S7** in dioxane ([1.1 M], 4.5 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Palladium scavenger 1-pyrrolidinecarbodithioic acid ammonium salt was added to the drying organic solution. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (1 : 3 to 0 : 1 water : acetonitrile) to afford **21** as a clear colorless oil (228 mg, 55%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 2H), 7.29 (dd, J = 4.3, 2.1 Hz, 3H), 2.13 – 2.04 (m, 4H), 1.97 – 1.90 (m, 2H), 1.47 – 1.36 (m, 2H), 1.33 – 1.22 (m, 4H), 1.19 (s, 12H), 0.92 (t, J = 7.4 Hz, 3H), 0.86 (t, J = 7.3 Hz, 3H), 0.49 (t, J = 7.9 Hz, 2H), 0.35 (s, 6H).

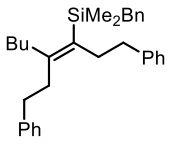
^{13}C NMR (151 MHz, $CDCl_3$) δ 153.9, 141.4, 133.9, 130.8, 128.4, 127.6, 82.9, 39.7, 34.3, 33.3, 25.0, 24.9, 24.6, 23.6, 22.4, 14.6, 14.6, 0.2.

^{11}B NMR (193 MHz, $CDCl_3$) δ 34.2.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.2.

FTIR (cm^{-1}): 2956, 2930, 2870, 1372, 1318, 1248, 1146, 1109, 834, 815, 770, 728, 700.

HRMS (FD) m/z, calcd for $[C_{25}H_{43}O_2BSi]^+$: 414.3125 ; found: 414.3144.



22
Chemical Formula: $C_{31}H_{40}Si$
Exact Mass: 440.2899
Molecular Weight: 440.7460

(**22**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (78 mg, 0.05 mmol, 5 mol %), dioxane (0.5 mL), triethylamine (700 µL, 5 mmol, 5 equiv), dimethylbenzylsilyl iodide (0.94 mL, 5 mmol, 5 equiv), and 4-octyne (146 µL, 1 mmol, 1 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([1.0 M], 5.0 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Palladium scavenger 1-pyrrolidinecarbodithioic acid ammonium salt was added to the drying organic solution. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (1 : 3 to 0 : 1 water : acetonitrile) to afford **22** as a colorless oil (238 mg, 54%).

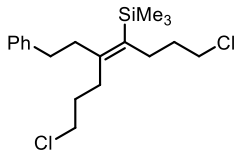
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 3H), 7.25 – 7.14 (m, 7H), 7.14 – 7.10 (m, 2H), 7.08 – 7.03 (m, 1H), 7.02 – 6.98 (m, 2H), 2.73 – 2.66 (m, 2H), 2.45 – 2.38 (m, 2H), 2.37 – 2.31 (m, 2H), 2.30 – 2.25 (m, 2H), 2.24 (s, 2H), 2.21 – 2.15 (m, 2H), 1.48 – 1.31 (m, 4H), 0.95 (t, *J* = 7.0 Hz, 3H), 0.17 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.1, 142.6, 142.5, 140.5, 131.4, 128.5, 128.5, 128.5, 128.4, 128.3, 128.2, 126.0, 125.9, 124.1, 37.3, 36.8, 35.5, 34.3, 33.7, 31.8, 27.4, 23.4, 14.3, -0.6.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -6.3.

FTIR (cm^{-1}): 3024, 2955, 2929, 2871, 1600, 1493, 1453, 1248, 1205, 1154, 832, 761, 747, 698.

HRMS (LIFDI) m/z, calcd for $[C_{31}H_{40}Si]^+$: 440.2899; found: 440.2904.



23
Chemical Formula: $C_{19}H_{30}Cl_2Si$
Exact Mass: 356.1494
Molecular Weight: 357.4340

(**23**) According to the general procedure C, $(DrewPhos)_2PdI_2$ (78 mg, 0.05 mmol, 5 mol %), dioxane (0.5 mL), triethylamine (700 µL, 5 mmol, 5 equiv), trimethylsilyl iodide (710 µL, 5 mmol, 5 equiv), and 1,8-dichloro-oct-4-yne (178 µL, 1 mmol, 1 equiv) were combined under nitrogen. Phenethylzinc iodide **S8** in dioxane ([1.2 M], 4.2 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Palladium scavenger 1-pyrrolidinecarbodithioic acid ammonium salt was added to the drying organic solution. Analysis of the crude reaction mixture via gas chromatography revealed a >95:5 *syn:anti* ratio. The product was purified by silica gel chromatography (0 : 100 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **23** as a clear colorless oil (330 mg, 92%).

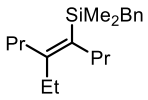
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.20 (t, *J* = 6.6 Hz, 3H), 3.55 (dt, *J* = 12.7, 6.5 Hz, 4H), 2.72 – 2.64 (m, 2H), 2.48 – 2.39 (m, 2H), 2.38 – 2.31 (m, 2H), 2.28 – 2.21 (m, 2H), 1.90 (dt, *J* = 14.1, 6.5 Hz, 2H), 1.72 (dt, *J* = 14.5, 6.6 Hz, 2H), 0.15 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2, 142.0, 134.4, 128.6, 128.4, 126.1, 45.3, 45.2, 38.6, 35.8, 34.0, 32.1, 29.4, 28.6, 1.3.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -7.1.

FTIR (cm^{-1}): 3027, 2954, 1602, 1496, 1454, 1307, 1249, 836, 755, 699.

HRMS (LIFDI) m/z, calcd for $[C_{19}H_{30}Cl_2Si]^+$: 356.1494; found: 356.1477.



24
Chemical Formula: $C_{19}H_{32}Si$
Exact Mass: 288.2273
Molecular Weight: 288.5500

(**24**) According to the general procedure C, $(JessePhos)_2PdCl_2$ (111 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), dimethylbenzylsilyl iodide (1.80 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a <5:95 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **24** as a colorless oil (532 mg, 92%).

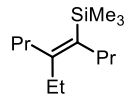
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.18 (t, *J* = 7.7 Hz, 2H), 7.08 – 7.02 (m, 1H), 7.02 – 6.97 (m, 2H), 2.19 (s, 2H), 2.11 (q, *J* = 7.5 Hz, 2H), 2.08 – 2.02 (m, 2H), 1.97 – 1.89 (m, 2H), 1.47 – 1.30 (m, 2H), 1.24 – 1.07 (m, 2H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.85 (t, *J* = 7.3 Hz, 3H), 0.08 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 154.5, 140.8, 130.8, 128.4, 128.1, 123.9, 38.5, 34.1, 27.5, 24.5, 24.2, 22.7, 14.6, 14.5, 13.8, -0.8.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -6.7.

FTIR (cm^{-1}): 2959, 2930, 2871, 1600, 1493, 1453, 1249, 1206, 1154, 829, 761, 698, 477.

HRMS (ESI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 288.2273; found: 288.2274.



25
Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520

(**25**) According to the general procedure C, $(JessePhos)_2PdCl_2$ (111 mg, 0.1 mmol, 5 mol %), dioxane (1 mL), triethylamine (1.4 mL, 10 mmol, 5 equiv), trimethylsilyl iodide (1.40 mL, 10 mmol, 5 equiv), and 4-octyne (293 µL, 2 mmol, 1 equiv) were combined under nitrogen. Ethylzinc iodide **S2** in dioxane ([1.6 M], 6.25 mL, 10 mmol, 5 equiv) was added over 4 h. The reaction was worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed an 8:92 *syn:anti* ratio. The product was purified by silica gel chromatography (hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **25** as a clear colorless oil (318 mg, 75%).

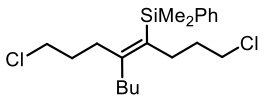
^{1}H NMR (600 MHz, $CDCl_3$) δ 2.15 – 2.05 (m, 4H), 2.04 – 1.98 (m, 2H), 1.46 – 1.31 (m, 2H), 1.30 – 1.15 (m, 2H), 0.96 (t, *J* = 7.5 Hz, 3H), 0.90 (dt, *J* = 9.4, 7.3 Hz, 6H), 0.12 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 153.4, 132.7, 38.5, 34.1, 24.6, 24.2, 22.8, 14.5, 14.4, 13.8, 1.4.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -7.8.

FTIR (cm^{-1}): 2960, 2931, 2872, 1599, 1465, 1248, 849, 834, 758.

HRMS (ESI) m/z, calcd for $[C_{16}H_{27}OSi]^+$: 213.2033; found: 213.1842.



26
Chemical Formula: $C_{20}H_{32}Cl_2Si$
Exact Mass: 370.1650
Molecular Weight: 371.4610

(**26**) According to the general procedure C, $(JessePhos)_2PdCl_2$ (56 mg, 0.05 mmol, 5 mol %), dioxane (0.5 mL), Et_3N (700 µL, 5 mmol, 5 equiv), Me_2PhSiI (935 µL, 5 mmol, 5 equiv), and 1,8-dichloro-oct-4-yne (178 µL, 1 mmol, 1 equiv) were combined under nitrogen. *n*-Butylzinc iodide **S3** in dioxane ([2.0 M], 2.5 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a 17:83 *syn:anti* ratio. The product was purified by silica gel chromatography (0 : 100 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (3 : 17 water : acetonitrile) to afford **26** as a clear colorless oil (221 mg, 60%).

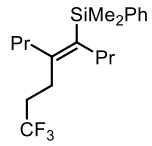
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.6 – 7.4 (m, 2H), 7.4 – 7.3 (m, 3H), 3.5 (t, *J* = 6.5 Hz, 2H), 3.1 (t, *J* = 6.8 Hz, 2H), 2.4 – 2.2 (m, 2H), 2.2 – 2.0 (m, 4H), 1.8 – 1.7 (m, 2H), 1.6 – 1.5 (m, 2H), 1.4 (td, *J* = 3.7, 1.9 Hz, 4H), 1.0 – 0.8 (m, 3H), 0.4 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.5, 140.7, 133.7, 130.8, 128.9, 127.9, 45.3, 44.9, 34.4, 34.0, 32.0, 31.4, 31.1, 29.4, 23.2, 14.2, -0.1.

^{29}Si NMR (119 MHz, $CDCl_3$) δ -10.9.

FTIR (cm^{-1}): 2956, 2871, 1595, 1457, 1428, 1305, 1250, 1109, 832, 814, 772, 730, 701.

HRMS (FD) m/z, calcd for $[C_{19}H_{30}Cl_2Si]^+$: 370.1650; found: 370.1639.



27
Chemical Formula: $C_{19}H_{29}F_3Si$
Exact Mass: 342.1991
Molecular Weight: 342.5212

(**27**) According to the general procedure C, $(JessePhos)_2PdCl_2$ (56 mg, 0.05 mmol, 5 mol %), dioxane (0.5 mL), triethylamine (700 µL, 5 mmol, 5 equiv), dimethylphenylsilyl iodide (940 µL, 5 mmol, 5 equiv), and 4-octyne (146 µL, 1 mmol, 1 equiv) were combined under nitrogen. 3,3,3-trifluorozinc iodide **S9** in dioxane ([1.2M], 4.1 mL, 5 mmol, 5 equiv) was added over 4 h. The reaction was quenched with THF, stirred for 0.25 h, and worked up according to the general procedure. Analysis of the crude reaction mixture via gas chromatography revealed a <5:95 *syn:anti* ratio. The product was purified by silica gel chromatography (0 : 100 to 5 : 95 ethyl acetate : hexanes) followed by reverse phase column chromatography on a Biotage instrument using a SNAP Ultra C18 60 g column (1 : 3 to 0 : 1 water : acetonitrile) to afford **27** as a clear colorless oil (208 mg, 61%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.45 (m, 2H), 7.32 (dd, *J* = 4.7, 1.9 Hz, 3H), 2.46 – 2.26 (m, 2H), 2.23 – 2.02 (m, 4H), 1.99 – 1.80 (m, 2H), 1.39 – 1.23 (m, 2H), 1.23 – 1.07 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.63 (t, *J* = 7.3 Hz, 3H), 0.37 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 149.4, 140.7, 133.7, 134.0, 128.7, 127.8, 127.1 (q, *J* = 276.9 Hz), 38.9, 34.2, 33.4 (q, *J* = 28.0 Hz), 24.5, 23.4 (q, *J* = 2.8 Hz), 22.2, 14.5, 14.0, -0.1.

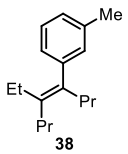
^{29}Si NMR (119 MHz, $CDCl_3$) δ -10.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -66.9.

FTIR (cm^{-1}): 2960, 2932, 2873, 1428, 1377, 1302, 1259, 1249, 1139, 1111, 1072, 978, 835, 813, 772, 729, 701.

HRMS (LIFDI) m/z, calcd for $[C_{19}H_{30}Cl_2Si]^+$: 342.199; found: 342.1983.

6. Synthesis of Tetrasubstituted Alkenes via Hiyama Cross-Coupling



38
Chemical Formula: $C_{17}H_{26}$
Exact Mass: 230.2035
Molecular Weight: 230.3950

(**38**) A 25 mL Schlenk flask equipped with a magnetic stirbar and rubber septum was flame-dried, allowed to cool to room temperature under vacuum, and refilled with N_2. The flask was briefly opened, charged with potassium trimethylsilanolate (257 mg, 2 mmol, 2 equiv) and 18-crown-6 (528 mg, 2 mmol, 2 equiv) and the septum was replaced. The flask was evacuated and refilled with N_2 3 times. THF (2 mL) and vinylsilane **16** (315 uL, 1 mmol, 1 equiv) were added by syringe. The reaction was stirred at 65 °C for 0.5 h. At this time, a solution of $[(allyl)PdCl]_2$ (9 mg, 0.025 mmol, 2.5 mol %), SPhos (21 mg, 0.05 mmol, 5 mol %), and 3-chlorotoluene (177 uL, 1.5 mmol, 1.5 equiv) in THF (2 mL) was added by syringe. The reaction was allowed to stir at 65 °C for 18 h. The reaction was then cooled to room temperature, opened to air, quenched with brine (5 mL), and diluted with Et_2O (5 mL). The aqueous layer was extracted with Et_2O (3 x 3 mL). The combined organic layers were washed with brine (10 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude material was purified via silica column chromatography (hexanes) to yield alkene **38** as a clear oil (142 mg, 62%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 6.88 – 6.85 (m, 2H), 2.34 (s, 3H), 2.28 – 2.24 (m, 2H), 2.17 – 2.12 (m, 2H), 1.82 (q, *J* = 7.5 Hz, 2H), 1.47 (dq, *J* = 15.0, 7.4 Hz, 2H), 1.30 – 1.19 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.85 (q, *J* = 7.3 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 137.3, 137.2, 136.0, 129.6, 127.7, 126.5, 126.1, 36.4, 32.6, 25.9, 22.4, 21.7, 14.6, 14.2, 13.9.

FTIR (cm^{-1}): 2961, 2932, 2871, 1463, 1377, 1053, 784, 715.

HRMS (ESI) m/z, calcd for $[C_{19}H_{30}Cl_2Si]^+$: 230.2035; found: 230.2035.

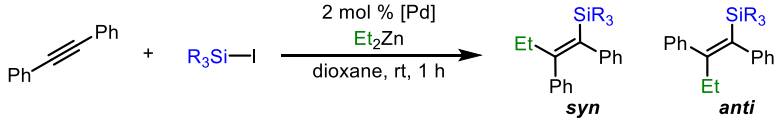
7. Additional Optimization Data

Note: All reactions in this section were performed on a 0.25 mmol scale in a nitrogen filled glovebox.

Examination of Addition of Reagents in the Multicomponent Carbosilylation Reaction of Diaryl Alkynes:

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with [Pd] (2 mol %), diphenylacetylene (1 equiv), dioxane (125 µL), diethylzinc (1 equiv) and silyl-iodide (1 equiv). The vial was sealed with a septum cap and removed from the glovebox. The reaction was quenched with diethyl ether (1 mL) and water (1 mL) via syringe. 1,3,5-trimethoxybenzene (TMB) (0.33 equiv) and nonane (1 equiv) were added as an NMR standard and a GC standard, respectively. An aliquot of the organic layer was then filtered through $MgSO_4$ and silica gel for NMR and GC analysis.

Table S1: Examination of Order of Addition and Slow Addition:



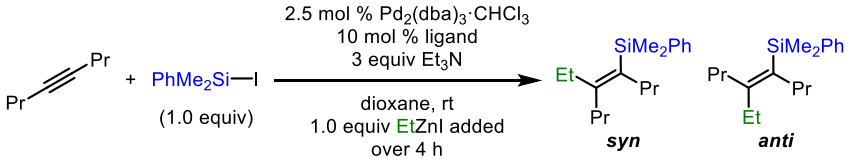
Entry	[Pd] Source	Silyl Iodide (equiv)	Nucleophile (equiv)	Et_3N (equiv)	Conditions	Yield [a]	*syn:anti* [b]
1	None	TMSI (1)	Et_2Zn (1)	0	TMSI added last, over 0.5 min	trace	>95:5
2	None	$PhMe_2SiI$ (1)	Et_2Zn (1)	0	$PhMe_2SiI$ added last, over 0.5 min	<1	>95:5
3	$(Ph_3P)_4Pd$	TMSI (1)	Et_2Zn (1)	0	TMSI added last, over 0.5 min	6	>95:5
4	$(Ph_3P)_4Pd$	TMSI (1)	Et_2Zn (1)	0	Et_2Zn added last, over 0.5 min	5	>95:5
5	$(Ph_3P)_4Pd$	$PhMe_2SiI$ (1)	Et_2Zn (1)	0	Et_2Zn added last, over 0.5 min	6	>95:5
6	$(Ph_3P)_4Pd$	$PhMe_2SiI$ (1)	Et_2Zn (1)	0	Et_2Zn added last, over 1h	25	>95:5
7	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (1)	Et_2Zn (1)	0	Et_2Zn added last, over 1h	70	>95:5
8	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (1)	Et_2Zn (1)	0	Et_2Zn added last, over 4 h	89	>95:5
9	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (1)	EtZnI (1)	1	EtZnI added last, over 4h	89	>95:5
10	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (1)	BuZnI (1)	1	BuZnI added last, over 4h	95	>95:5
11	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (3)	EtZnI (1.5)	3	EtZnI added last, over 4h	95	>95:5

12	$(Ph_3P)_2PdCl_2$	$PhMe_2SiI$ (3)	BuZnI (1.5)	3	BuZnI added last, over 4 h	96	>95:5

[a]Yields obtained by ^1H NMR with TMB as an internal standard. [b]Ratio determined by GC.

Examination of Ligands in the Multicomponent Carbosilylation Reaction of Dialkyl Alkynes:

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with Pd_2dba_3 $CHCl_3$ (2.5 mol %), ligand (10 mol %), dioxane (125 uL), triethylamine (3 equiv), 4-octyne (1 equiv), and dimethylphenylsilyl iodide (1 equiv). The vial was sealed with a septum cap and removed from the glovebox. A solution of ethylzinc iodide (1.0 equiv) in dioxane was then added dropwise over 4 h via syringe pump, with stirring. The reaction was quenched with diethyl ether (1 mL) and water (1 mL) via syringe. 1,3,5-trimethoxybenzene (TMB) (0.33 equiv) and nonane (1 equiv) were added as an NMR standard and a GC standard, respectively. An aliquot of the organic layer was then filtered through $MgSO_4$ and silica gel for NMR and GC analysis.

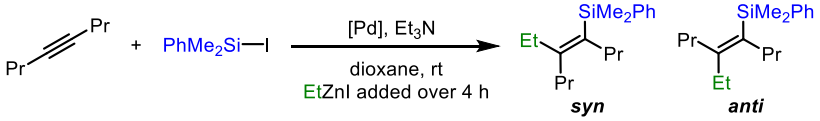


Entry	Ligand	Yield[a]	syn:anti[b]
1	Ph_3P	88	65:35
2	$(3,5\text{-}CF_3C_6H_3)_3P$	13	50:50
3	$(4\text{-}MeOC_6H_4)_3P$	66	60:40
4	DrewPhos	76	90:10
5	$(o\text{-tol})_3P$	65	18:82
6	Ph_2P^tBu	60	3:97
7	JessePhos	67	5:95

[a]Yields obtained by ^1H NMR with TMB as an internal standard. [b]Ratio determined by GC.

Further Optimization of Conditions for the Multicomponent Carbosilylation Reaction of Dialkyl Alkynes:

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with palladium precatalyst, dioxane (125 uL), triethylamine, 4-octyne (1 equiv), and dimethylphenylsilyl iodide. The vial was sealed with a septum cap and removed from the glovebox. A solution of ethylzinc iodide in dioxane was then added dropwise over 4 h via syringe pump, with stirring. The reaction was quenched with diethyl ether (1 mL) and water (1 mL) via syringe. 1,3,5-trimethoxybenzene (TMB) (0.33 equiv) and nonane (1 equiv) were added as an NMR standard and a GC standard, respectively. An aliquot of the organic layer was then filtered through $MgSO_4$ and silica gel for NMR and GC analysis.

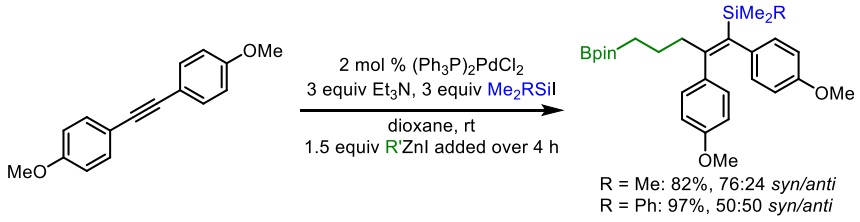


Entry	[Pd] (mol %)	Me_2PhSiI (equiv)	Et_3N (equiv)	EtZnI (equiv)	Yield (%)[a]	syn:anti[b]
1	$(DrewPhos)_2PdI_2$ (2 mol %)	3	3	1.5	53	>95:5
2	$(DrewPhos)_2PdI_2$ (5 mol %)	3	3	1.5	60	>95:5
3	$(DrewPhos)_2PdI_2$ (5 mol %)	5	5	1.5	65	>95:5
4	$(DrewPhos)_2PdI_2$ (5 mol %)	5	5	2	74	>95:5
5	$(DrewPhos)_2PdI_2$ (5 mol %)	5	5	5	99	>95:5
6	$(JessePhos)_2PdI_2$ (5 mol %)	5	5	5	94	<5:95

[a]Yields obtained by ^1H NMR with TMB as an internal standard. [b]Ratio determined by GC.

8. Examination of Silicon Substitution

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with $(Ph_3P)_2PdCl_2$ (3.5 mg, 0.005 mmol, 2 mol %), 1,2-bis(4-methoxyphenyl)acetylene (59 mg, 0.25 mmol, 1 equiv), dioxane (125 µL), and silyl-iodide (0.75 mmol, 3 equiv). The vial was sealed with a septum cap and removed from the glovebox. Alkylzinc iodide **S7** (0.6M, 0.68 mL, 0.38 mmol,1.5 equiv) was added over 4 h via syringe pump. The reaction was quenched with diethyl ether (1 mL) and water (1 mL) via syringe. 1,3,5-trimethoxybenzene (TMB) (14 mg, 0.33 equiv) and nonane (32 mg, 1 equiv) were added as an NMR standard and a GC standard, respectively. An aliquot of the organic layer was then filtered through $MgSO_4$ and silica gel for NMR and GC analysis.



9. References

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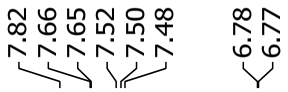
10. Spectral Data



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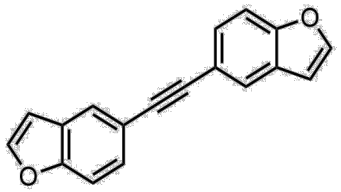
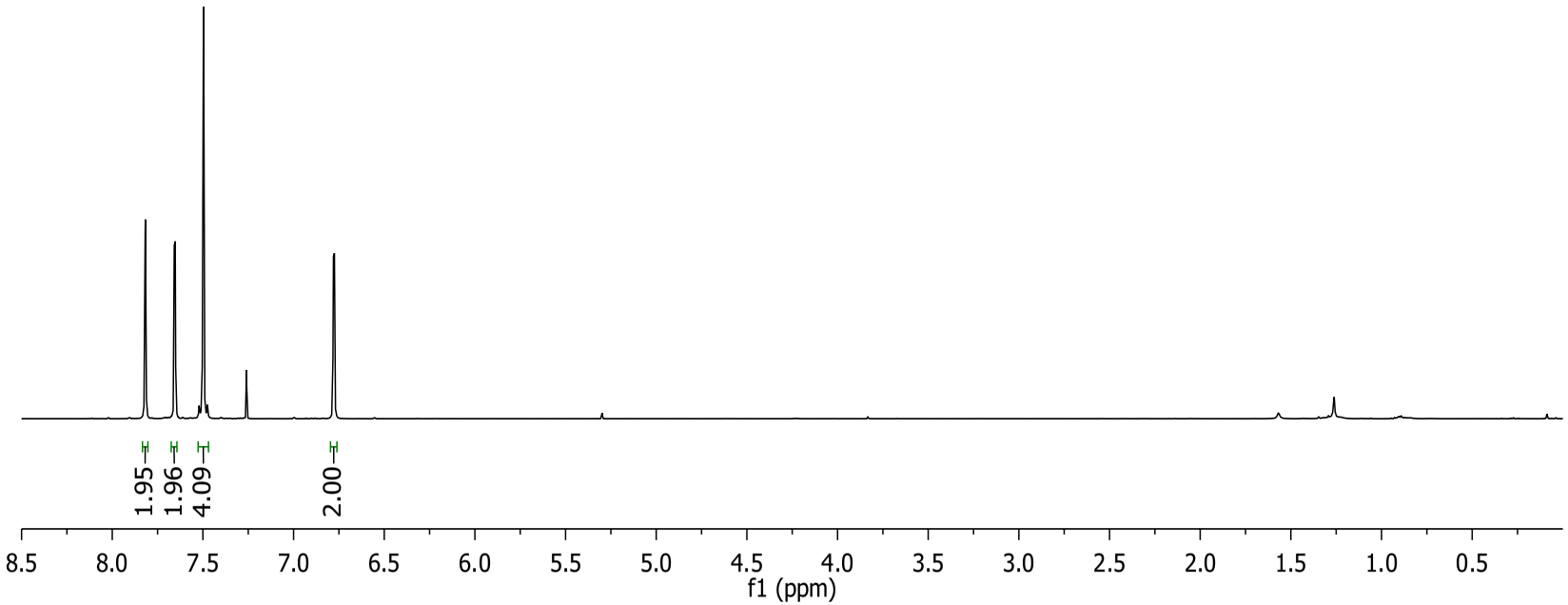


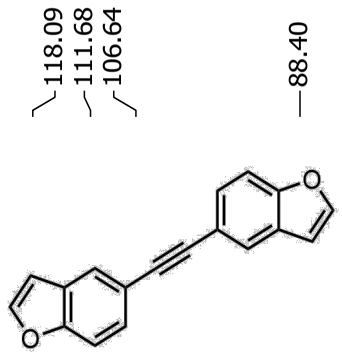
S1

Chemical Formula: $C_{18}H_{10}O_2$

Exact Mass: 258.0681

Molecular Weight: 258.2760





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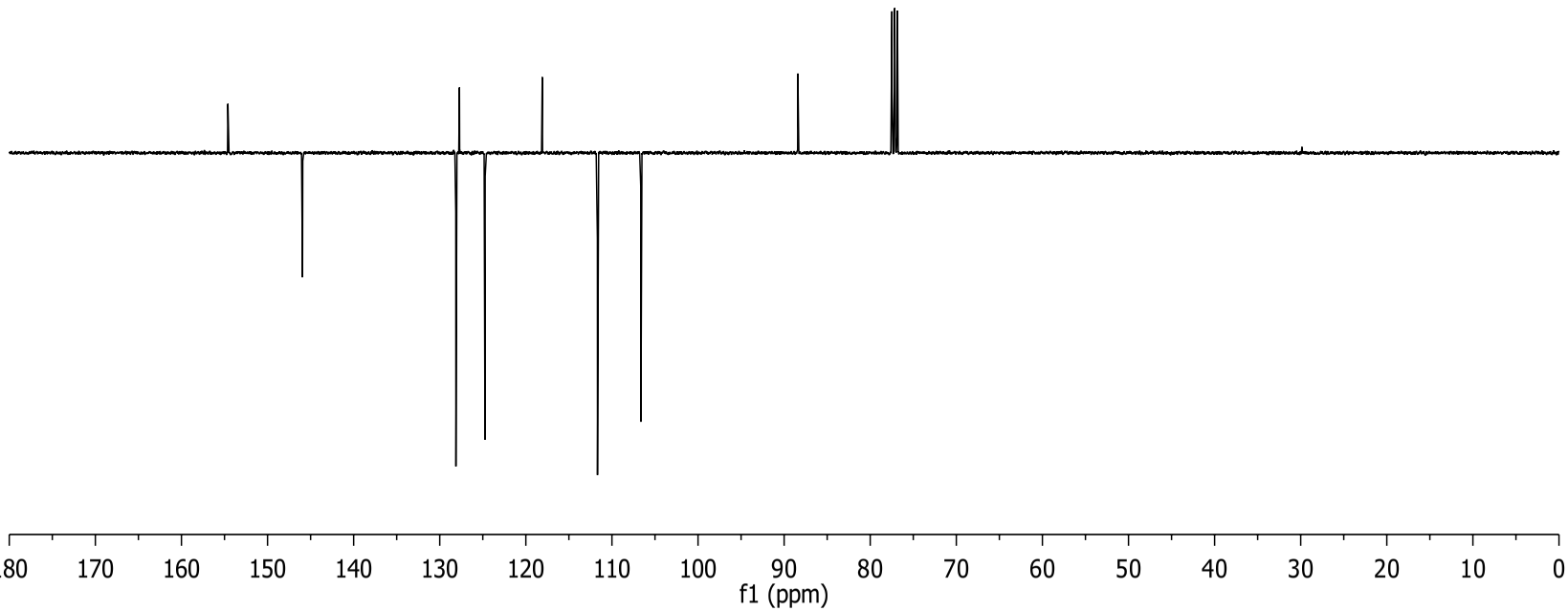
S1

Chemical Formula: $C_{18}H_{10}O_2$

Exact Mass: 258.0681

Molecular Weight: 258.2760



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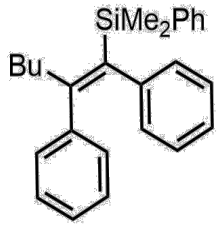
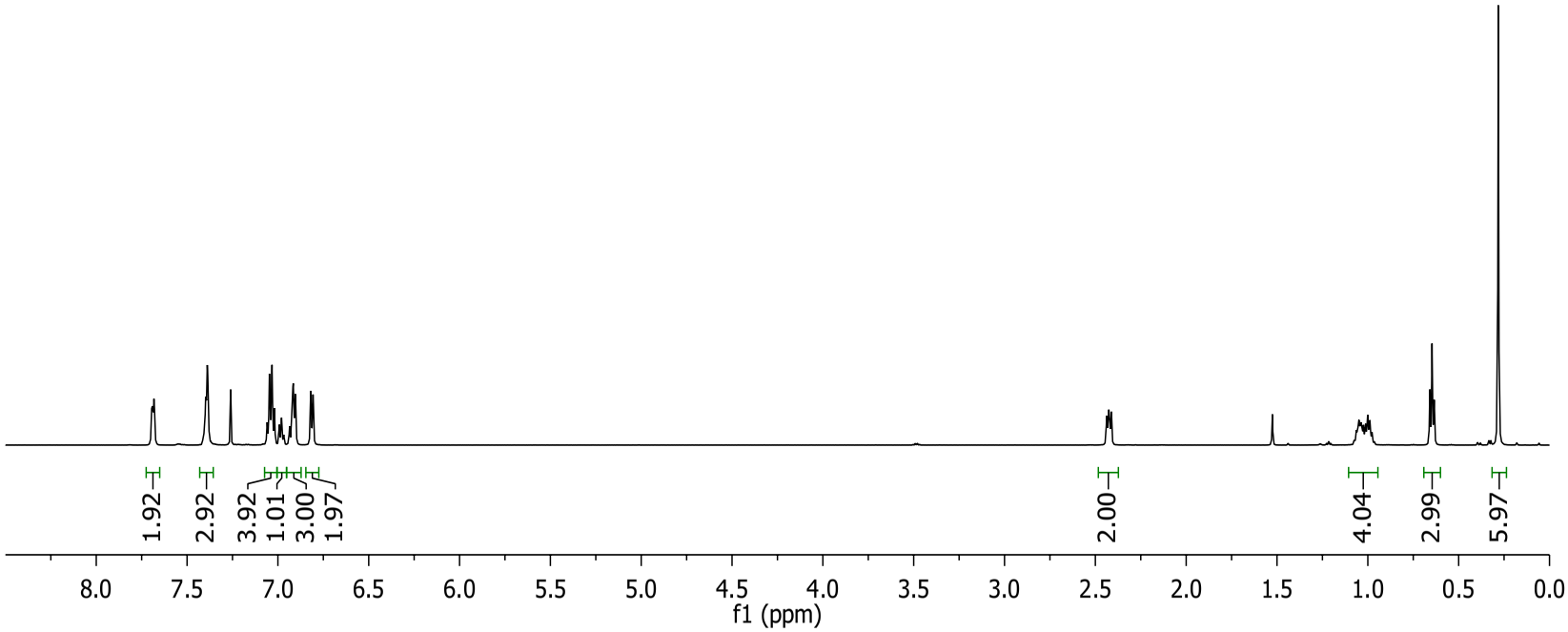


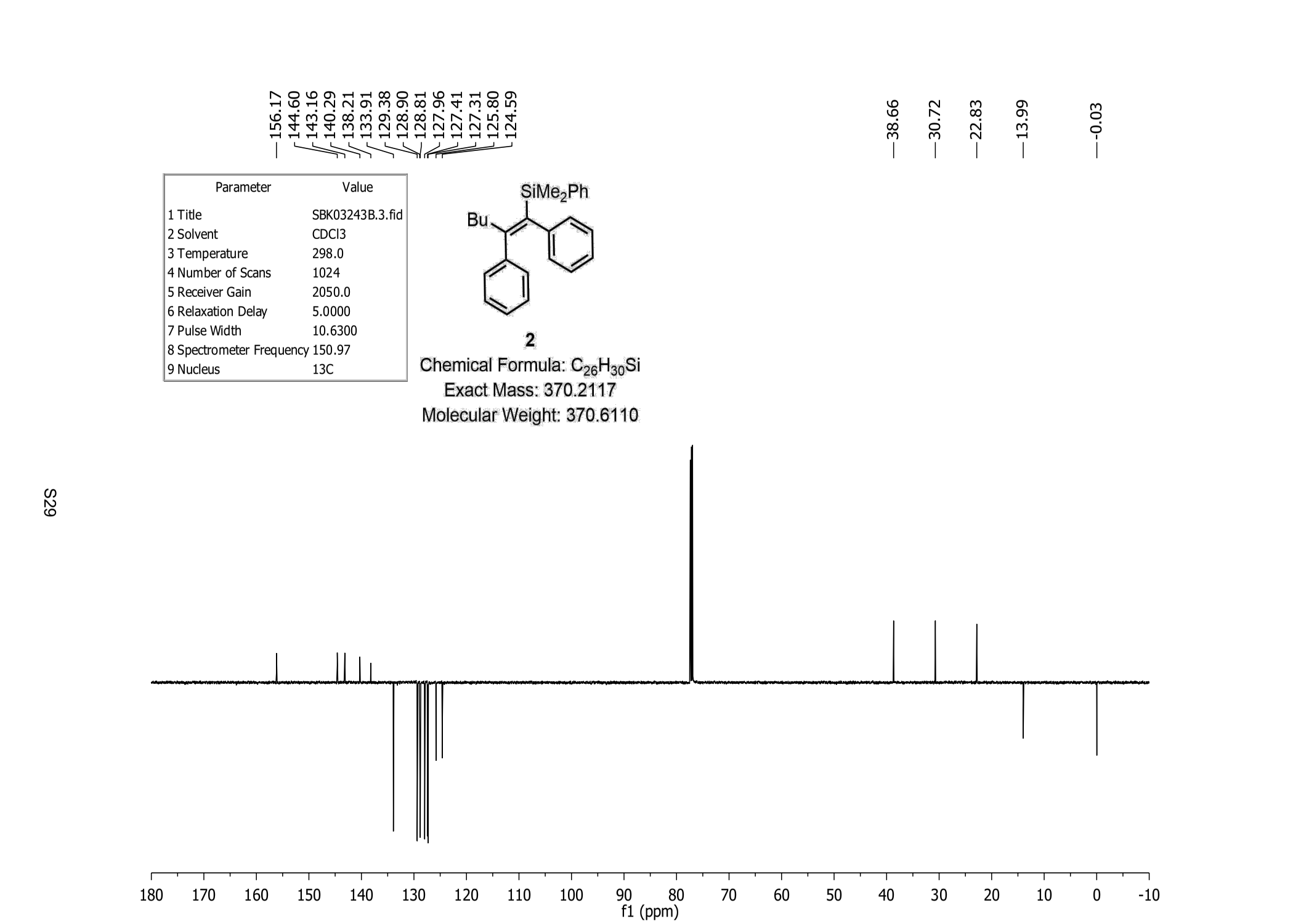
2

Chemical Formula: $C_{26}H_{30}Si$

Exact Mass: 370.2117

Molecular Weight: 370.6110

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8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Ph
Bu
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Exact Mass: 370.2117
Molecular Weight: 370.6110
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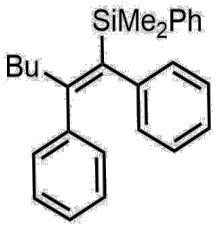
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



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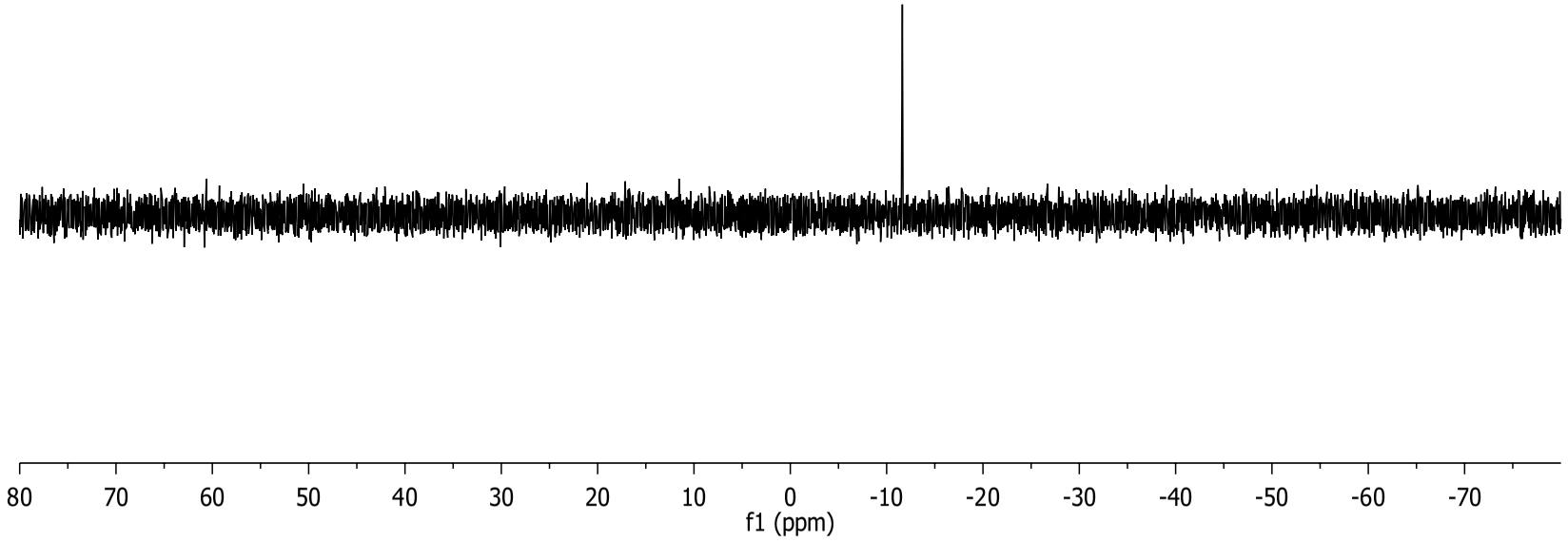
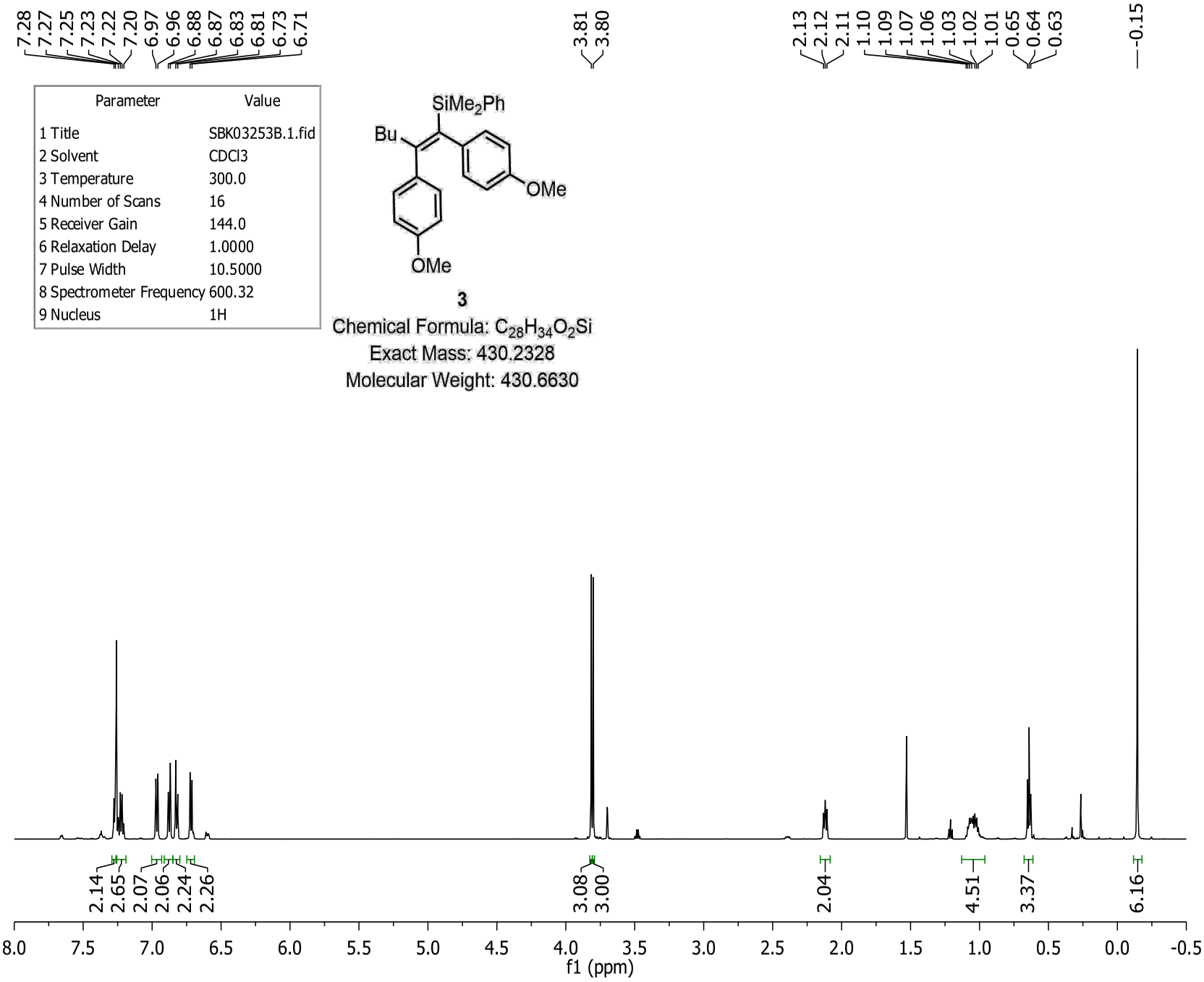
Molecular Weight: 370.6110

-11.63

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2.12
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1.07
1.06
1.03
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9 Nucleus 1H
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OMe
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2.06
2.24
2.26
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6.0
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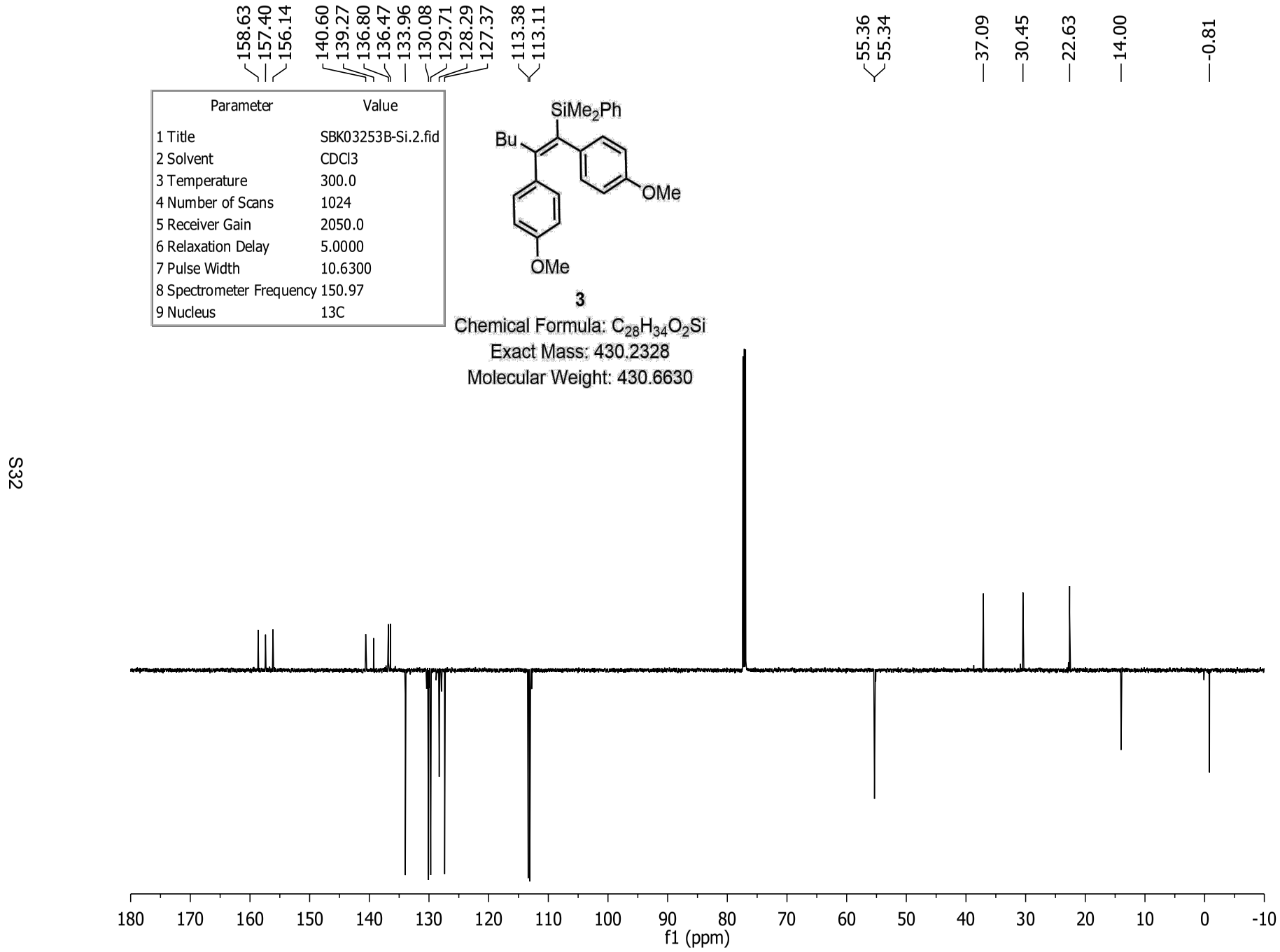
f1 (ppm)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9 Nucleus 13C
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Chemical Formula: C28H34O2Si
Exact Mass: 430.2328
Molecular Weight: 430.6630
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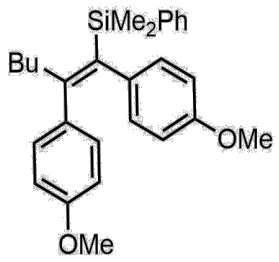

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Chemical Formula: $C_{28}H_{34}O_2Si$
Exact Mass: 430.2328
Molecular Weight: 430.6630

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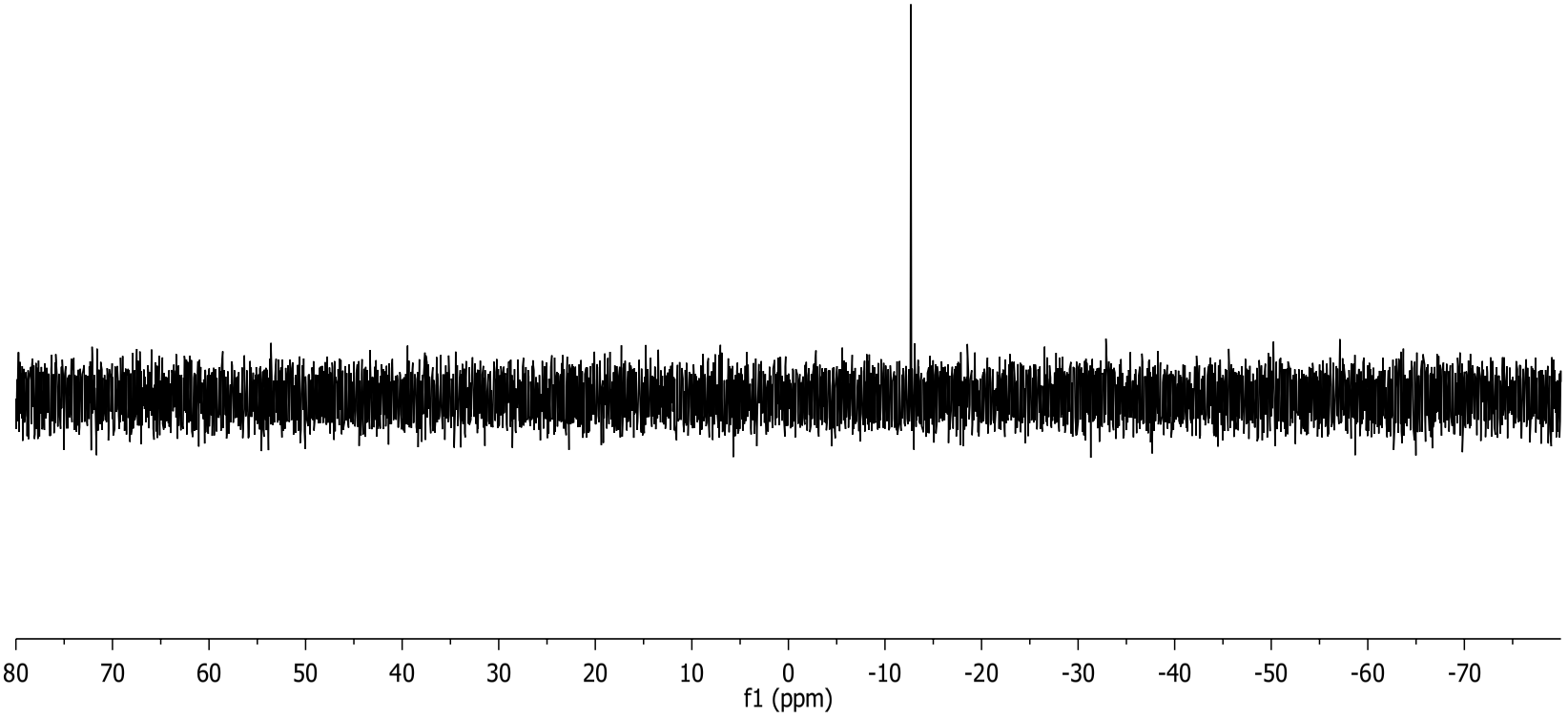

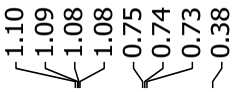

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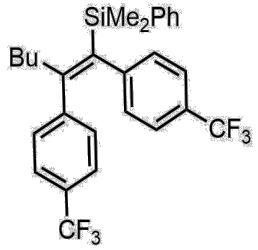

4

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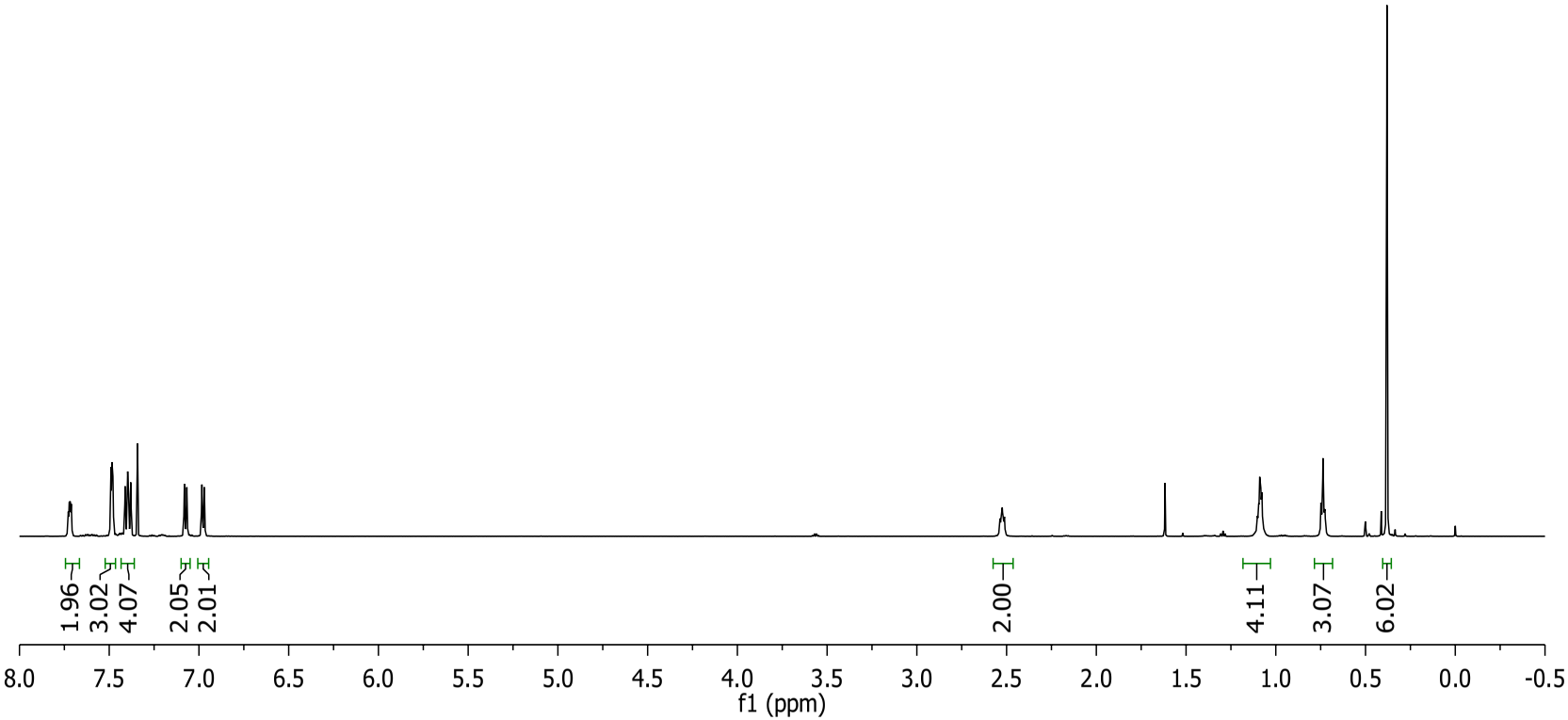

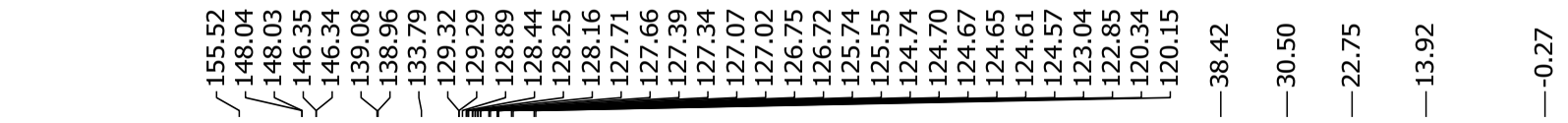

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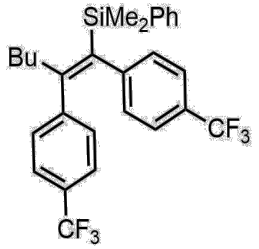

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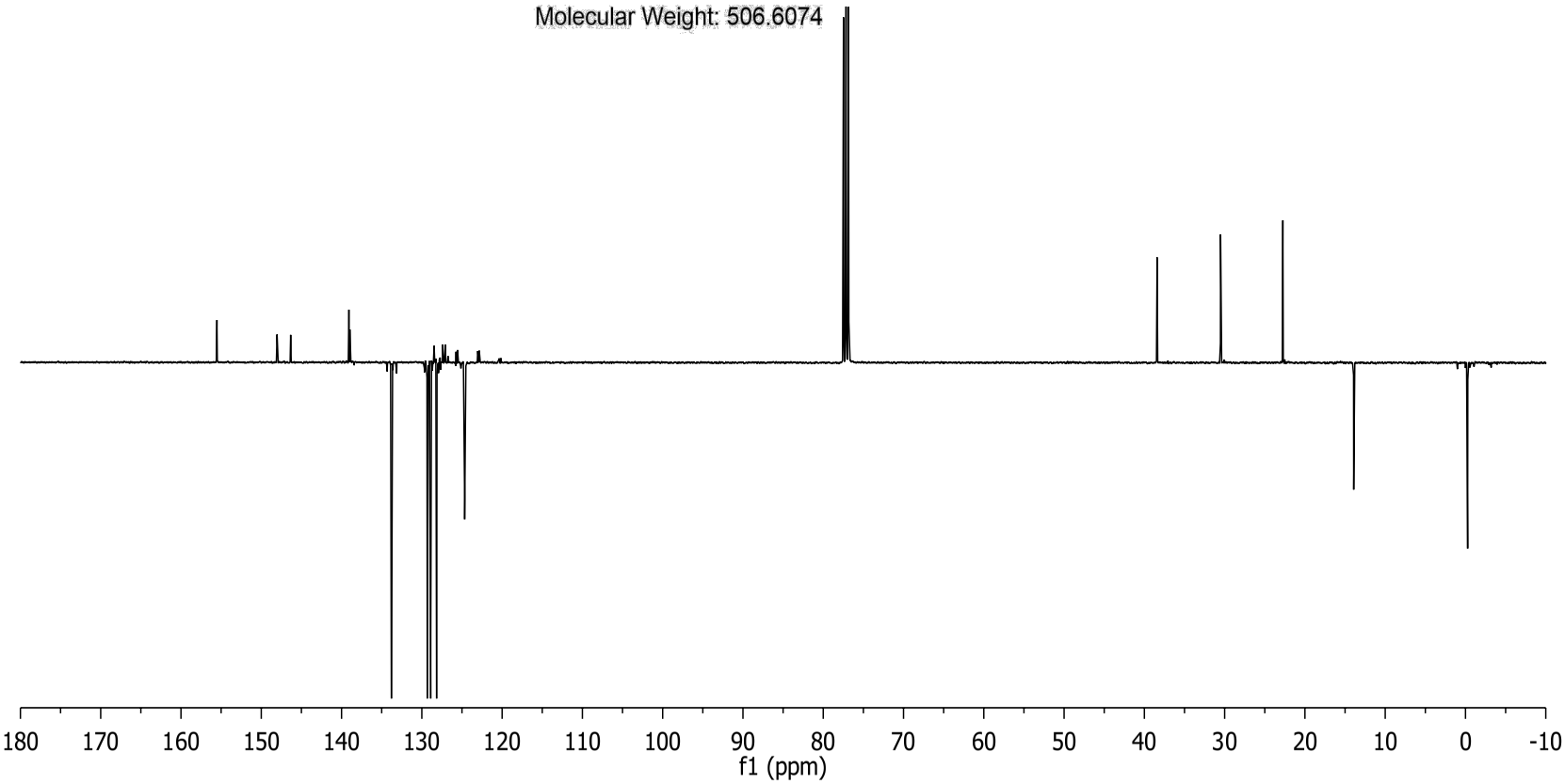
Exact Mass: 506.1864

Molecular Weight: 506.6074



Parameter	Value
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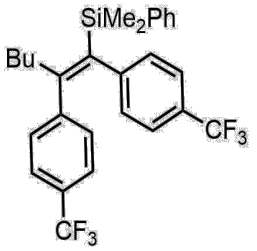
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



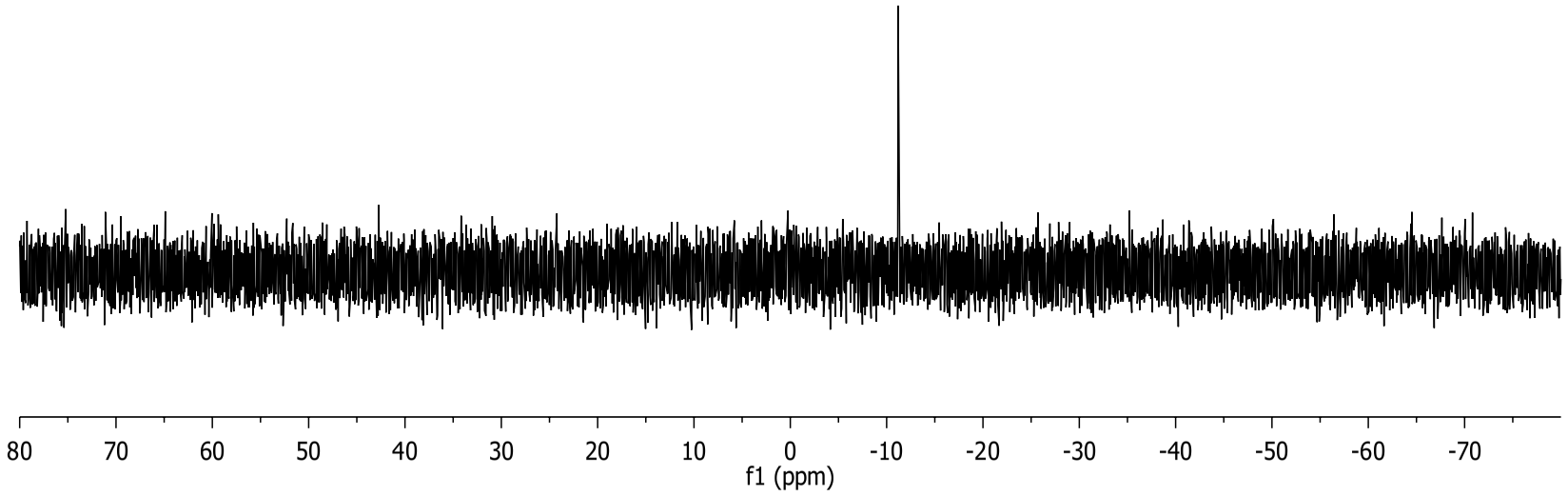
4

Chemical Formula: $C_{28}H_{28}F_6Si$

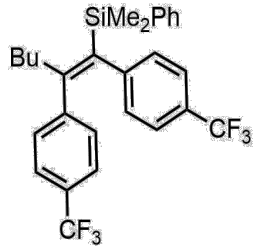
Exact Mass: 506.1864

Molecular Weight: 506.6074

-11.20



Parameter	Value
1 Title	SBK03263B-19F.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	322.0
6 Relaxation Delay	3.0000
7 Pulse Width	11.4000
8 Spectrometer Frequency	564.81
9 Nucleus	19F

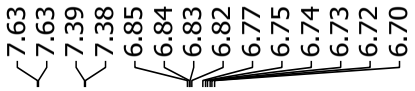


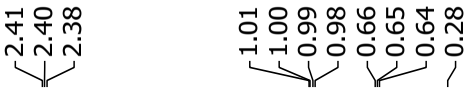
4

Chemical Formula: $C_{28}H_{28}F_6Si$

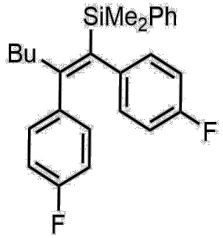
Exact Mass: 506.1864

Molecular Weight: 506.6074





Parameter	Value
1 Title	SBK03245B.1.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	16
5 Receiver Gain	144.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

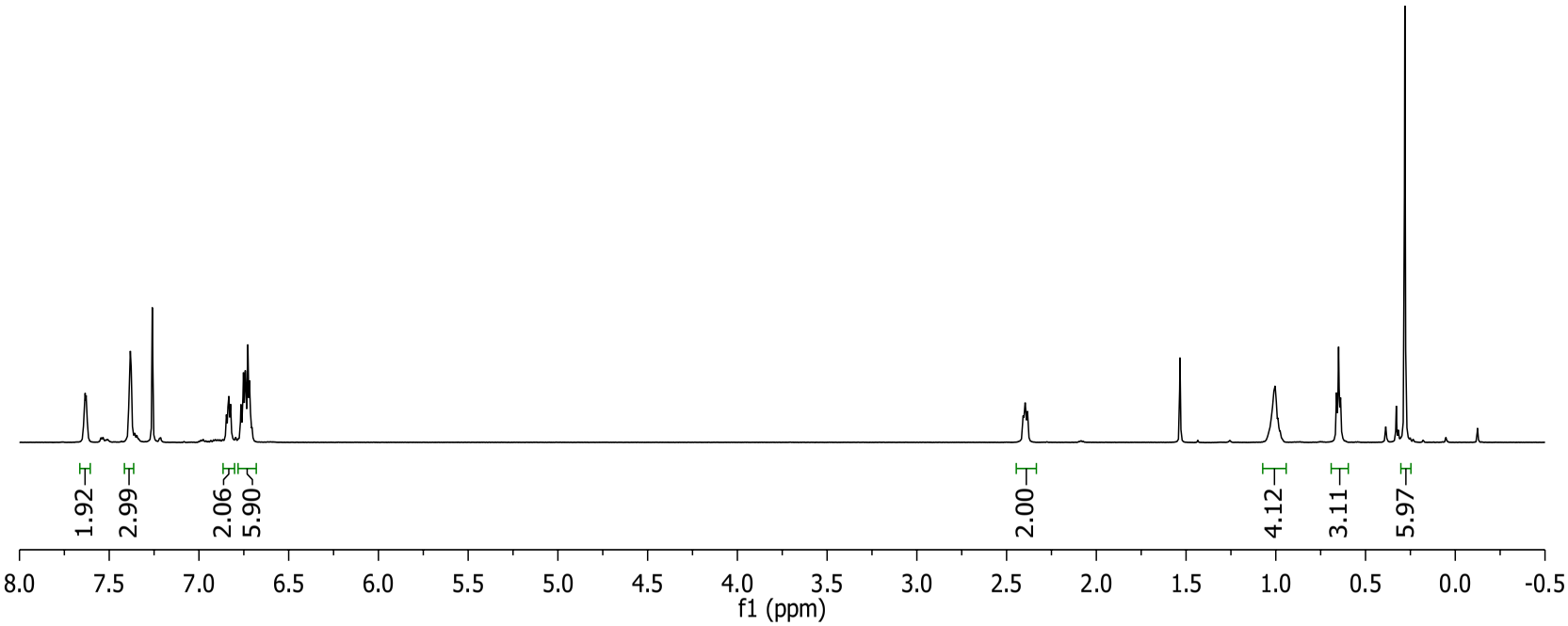


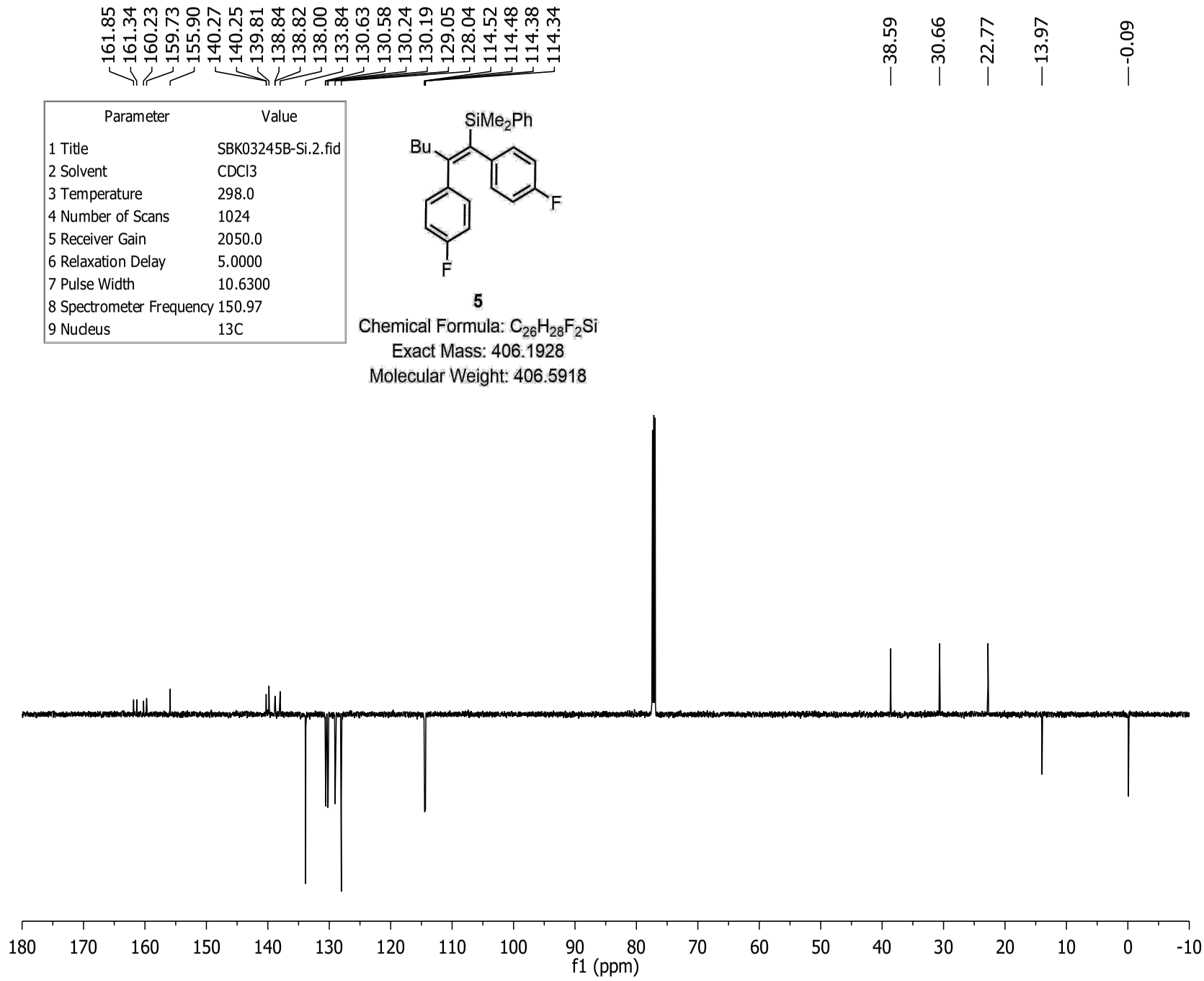
5

Chemical Formula: $C_{26}H_{28}F_2Si$

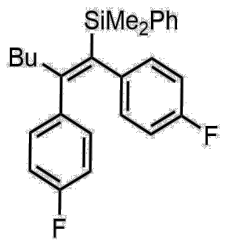
Exact Mass: 406.1928

Molecular Weight: 406.5918




161.85
161.34
160.23
159.73
155.90
140.27
140.25
139.81
138.84
138.82
138.00
133.84
130.63
130.58
130.24
130.19
129.05
128.04
114.52
114.48
114.38
114.34
38.59
30.66
22.77
13.97
-0.09
Parameter Value
1 Title SBK03245B-Si.2.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Ph
Bu
F
F
5
Chemical Formula: C26H28F2Si
Exact Mass: 406.1928
Molecular Weight: 406.5918
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Parameter	Value
1 Title	SBK03245B-Si.1.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



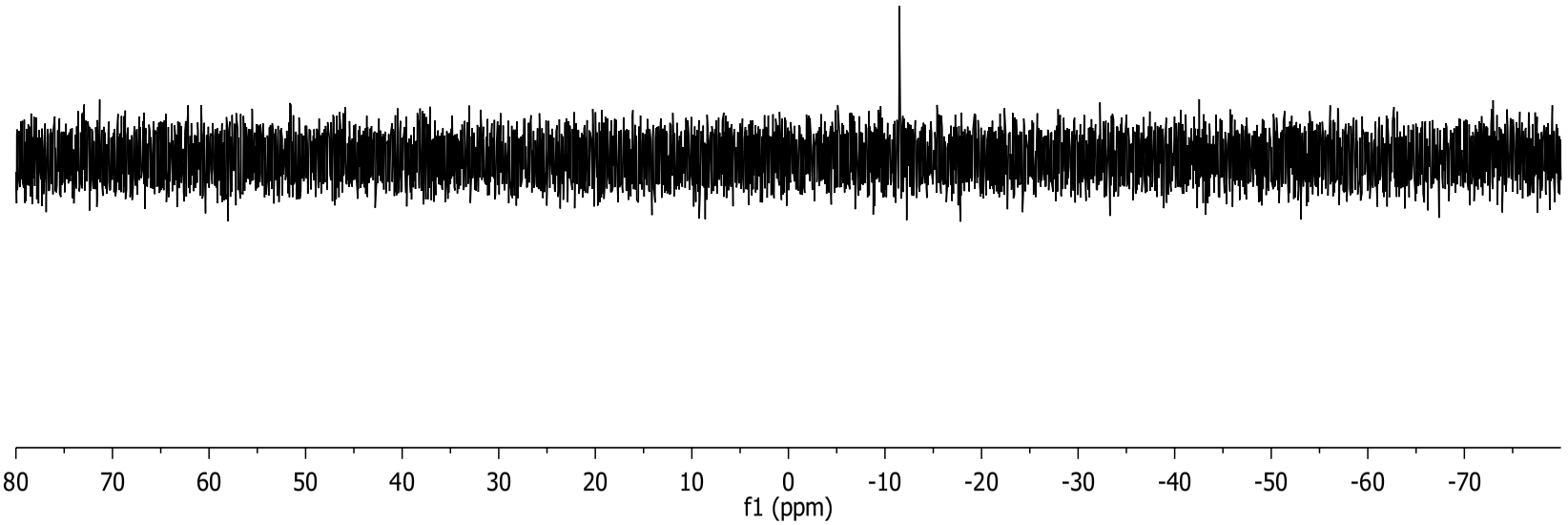
5

Chemical Formula: $C_{26}H_{28}F_2Si$

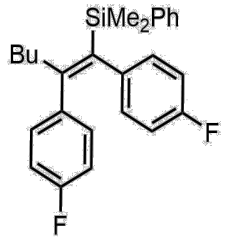
Exact Mass: 406.1928

Molecular Weight: 406.5918

-11.51

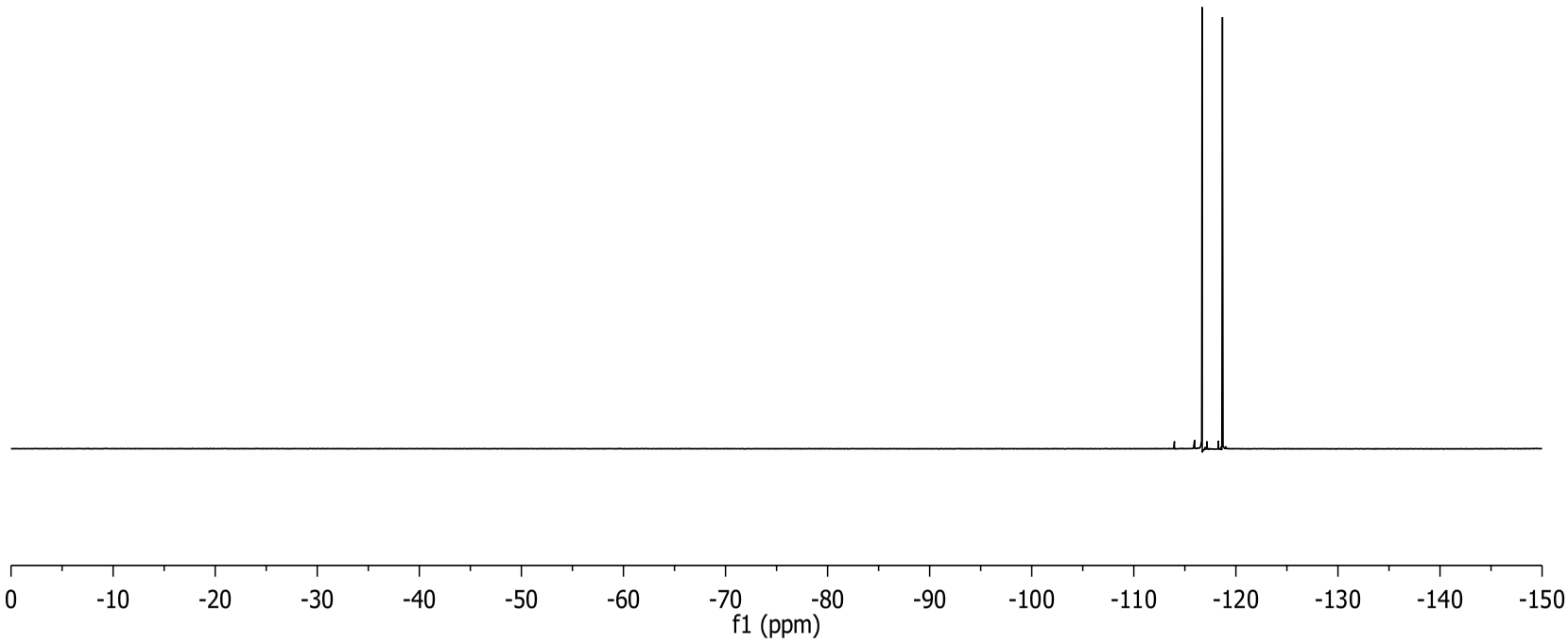


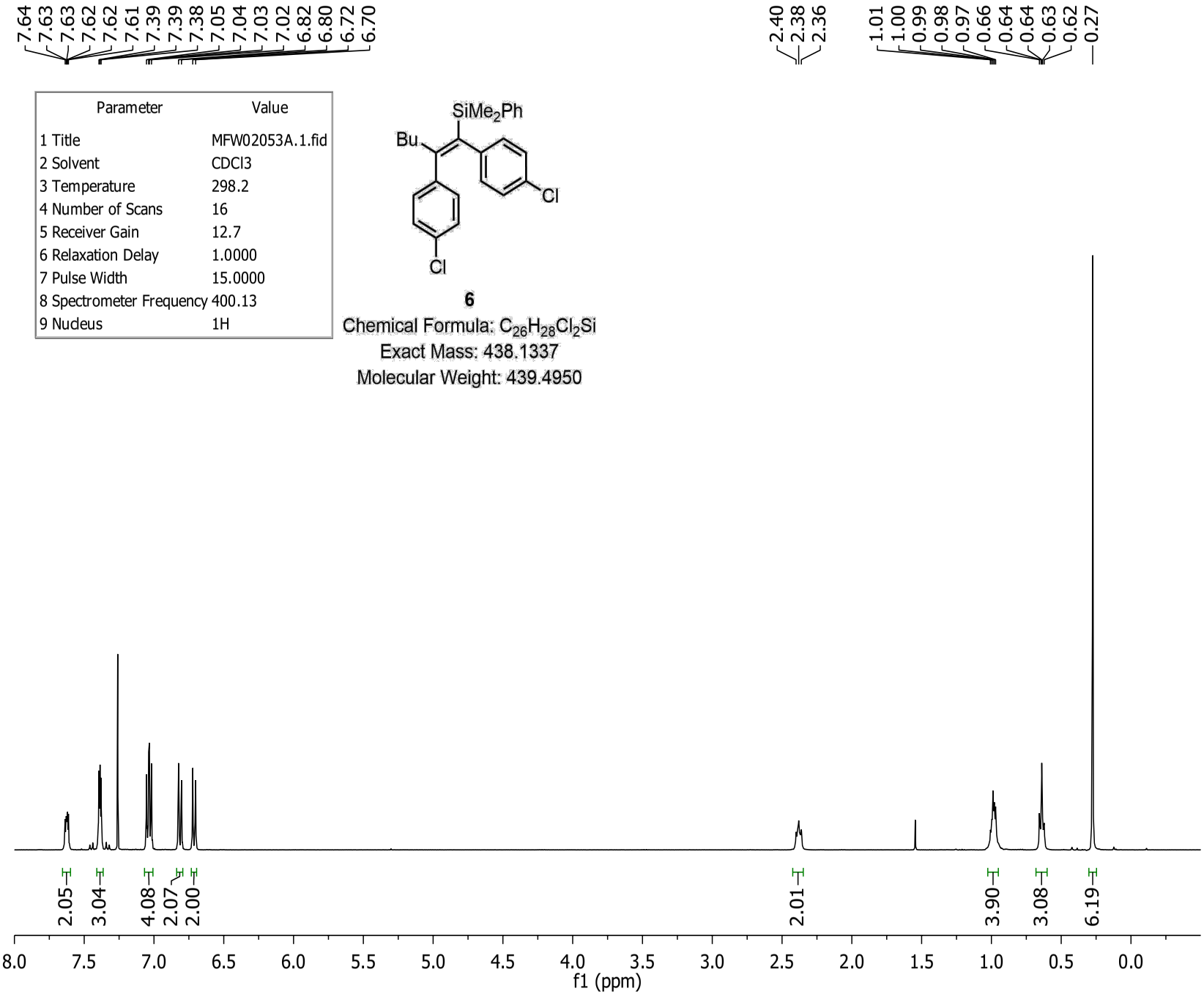
Parameter	Value
1 Title	SBK03245B.4.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	322.0
6 Relaxation Delay	3.0000
7 Pulse Width	11.4000
8 Spectrometer Frequency	564.81
9 Nucleus	19F

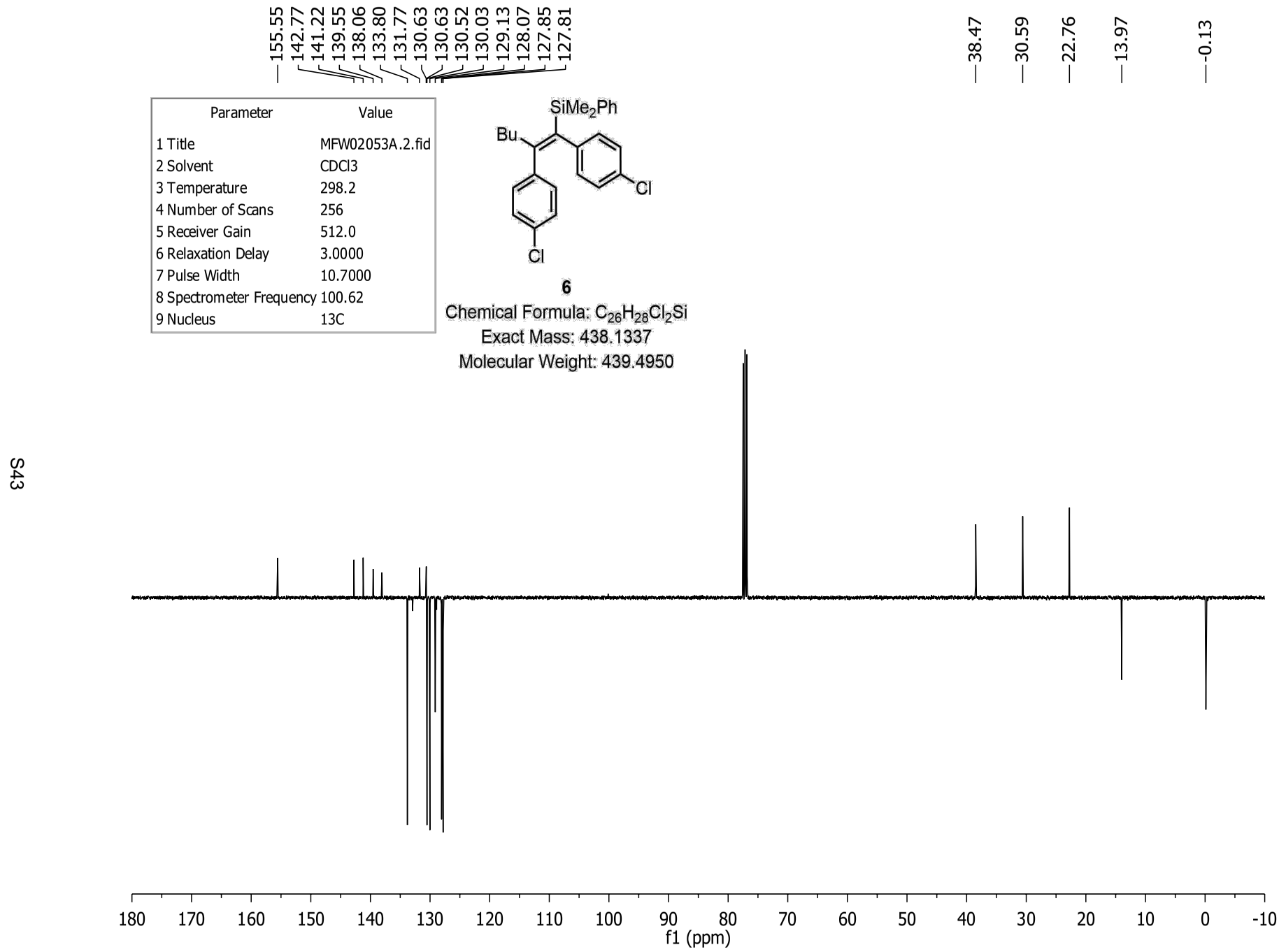


5

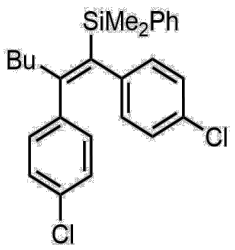
Chemical Formula: $C_{26}H_{28}F_2Si$
Exact Mass: 406.1928
Molecular Weight: 406.5918




7.64
7.63
7.63
7.62
7.62
7.61
7.39
7.39
7.38
7.05
7.04
7.03
7.02
6.82
6.80
6.72
6.70
2.40
2.38
2.36
1.01
1.00
0.99
0.98
0.97
0.66
0.64
0.64
0.63
0.62
0.27
Parameter
Value
1 Title
MFW02053A.1.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
16
5 Receiver Gain
12.7
6 Relaxation Delay
1.0000
7 Pulse Width
15.0000
8 Spectrometer Frequency
400.13
9 Nucleus
1H
SiMe2Ph
Bu
Cl
Cl
6
Chemical Formula: C26H28Cl2Si
Exact Mass: 438.1337
Molecular Weight: 439.4950
2.05
3.04
4.08
2.07
2.00
2.01
3.90
3.08
6.19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

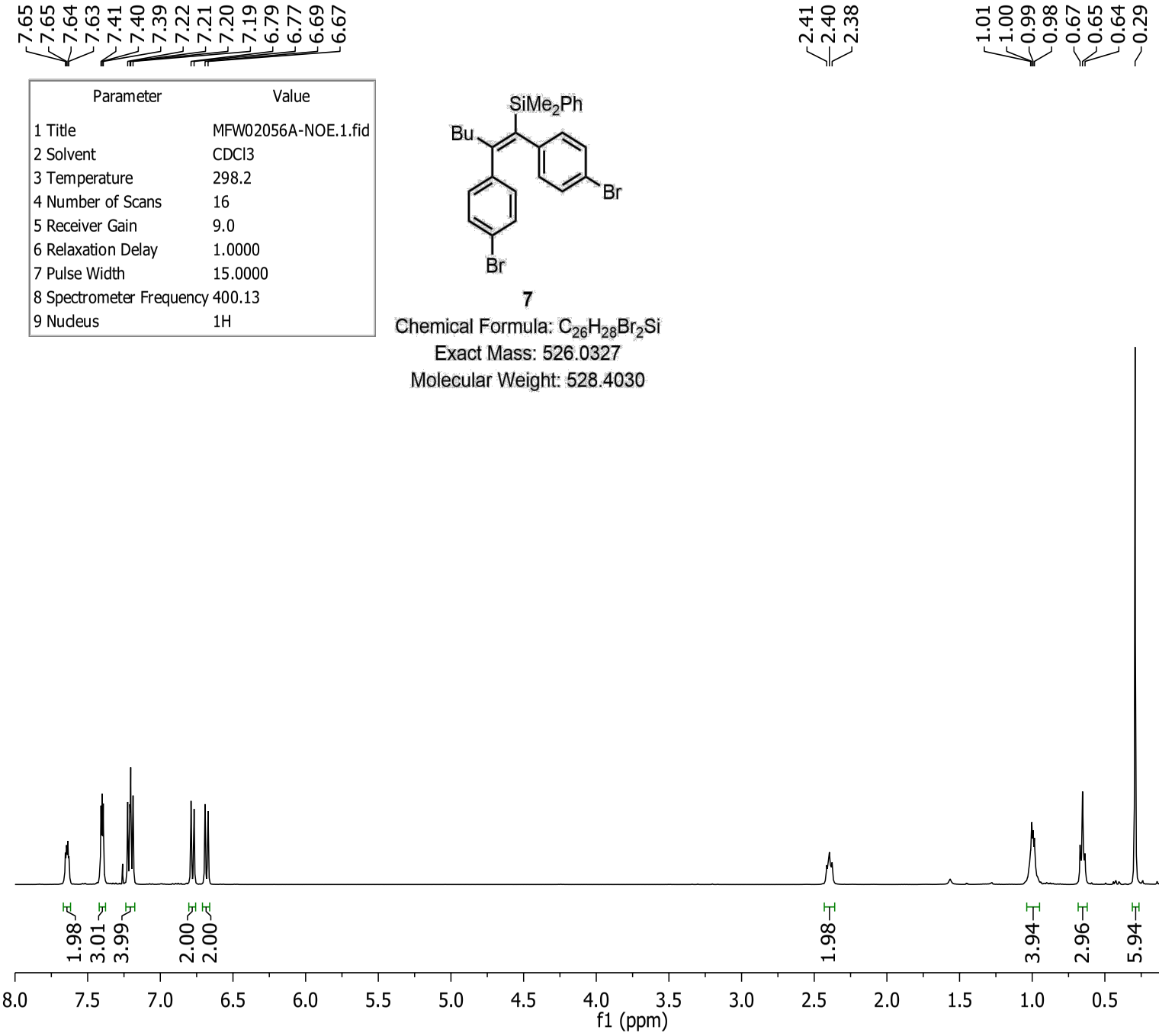

155.55
142.77
141.22
139.55
138.06
133.80
131.77
130.63
130.63
130.52
130.03
129.13
128.07
127.85
127.81
38.47
30.59
22.76
13.97
-0.13
Parameter
Value
1 Title
MFW02053A.2.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Ph
Bu
Cl
Cl
6
Chemical Formula: C26H28Cl2Si
Exact Mass: 438.1337
Molecular Weight: 439.4950
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

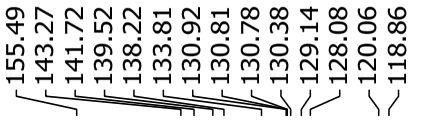
Parameter	Value
1 Title	MFW02053col2A-Si.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	128
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

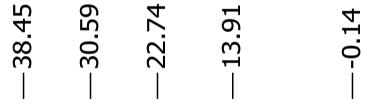


6

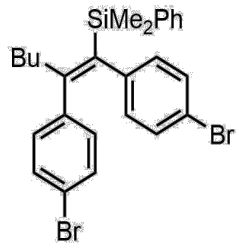
Chemical Formula: $C_{26}H_{28}Cl_2Si$
Exact Mass: 438.1337
Molecular Weight: 439.4950


7.65
7.65
7.64
7.63
7.41
7.40
7.39
7.22
7.21
7.20
7.19
6.79
6.77
6.69
6.67
2.41
2.40
2.38
1.01
1.00
0.99
0.98
0.67
0.65
0.64
0.29
Parameter
Value
1 Title
MFW02056A-NOE.1.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
16
5 Receiver Gain
9.0
6 Relaxation Delay
1.0000
7 Pulse Width
15.0000
8 Spectrometer Frequency
400.13
9 Nucleus
1H
SiMe2Ph
Bu
Br
Br
7
Chemical Formula: C26H28Br2Si
Exact Mass: 526.0327
Molecular Weight: 528.4030
1.98
3.01
3.99
2.00
2.00
1.98
3.94
2.96
5.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)





Parameter	Value
1 Title	MFW02056A.4.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	5.0000
7 Pulse Width	10.6300
8 Spectrometer Frequency	150.97
9 Nucleus	13C

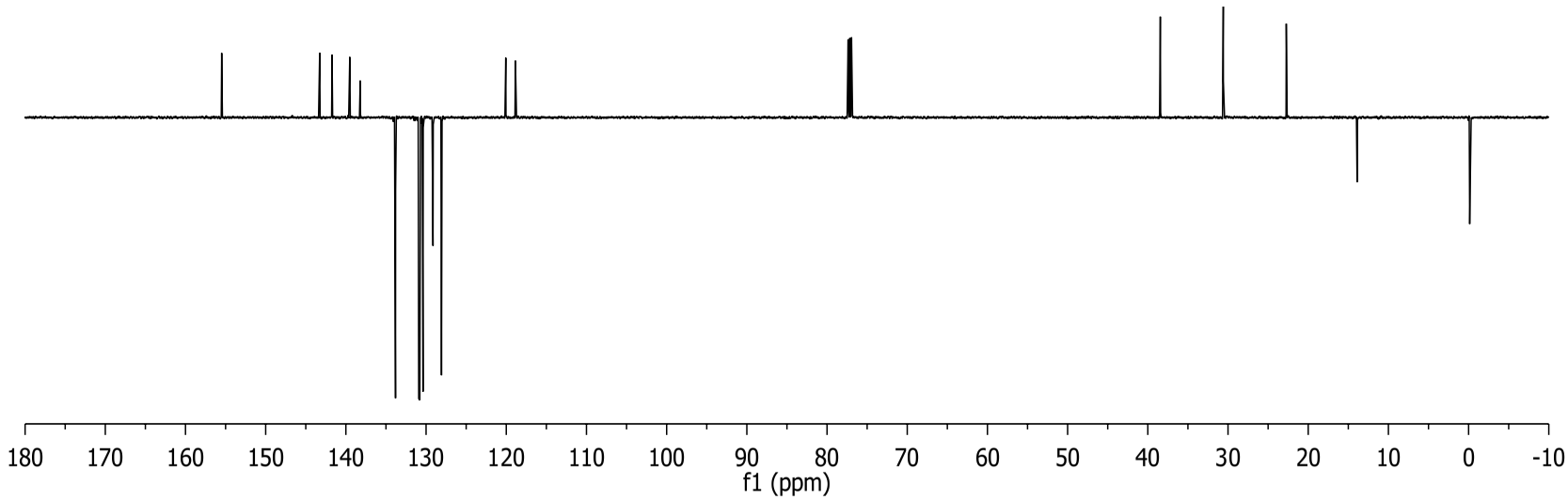


7

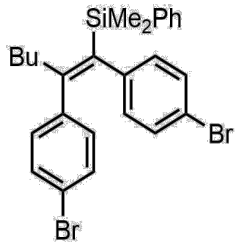
Chemical Formula: $C_{26}H_{28}Br_2Si$

Exact Mass: 526.0327

Molecular Weight: 528.4030



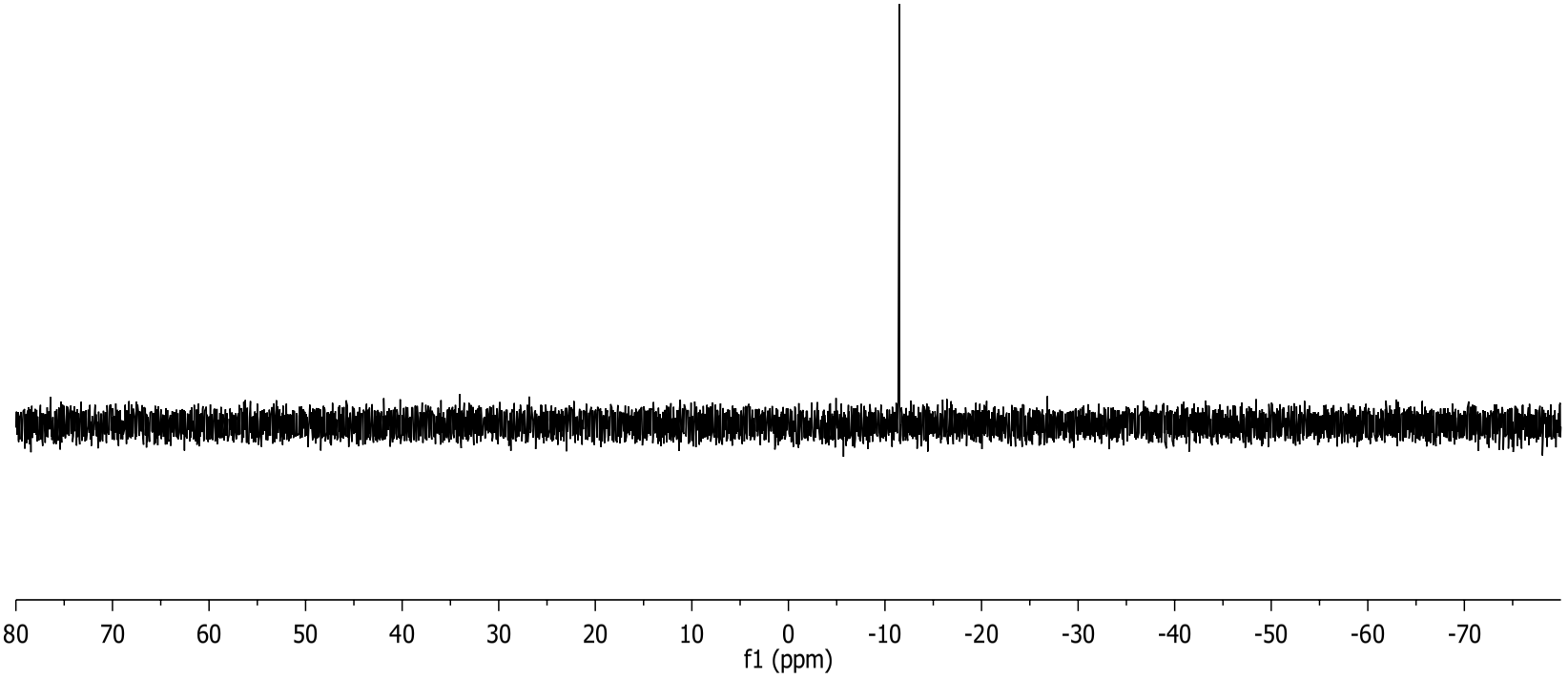
Parameter	Value
1 Title	MFW02056col2A-Si.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	128
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

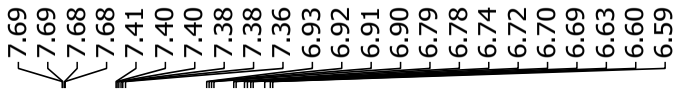


7

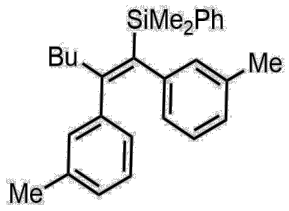
Chemical Formula: $C_{26}H_{28}Br_2Si$
Exact Mass: 526.0327
Molecular Weight: 528.4030

-11.49





Parameter	Value
1 Title	SBK03259B.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	128.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

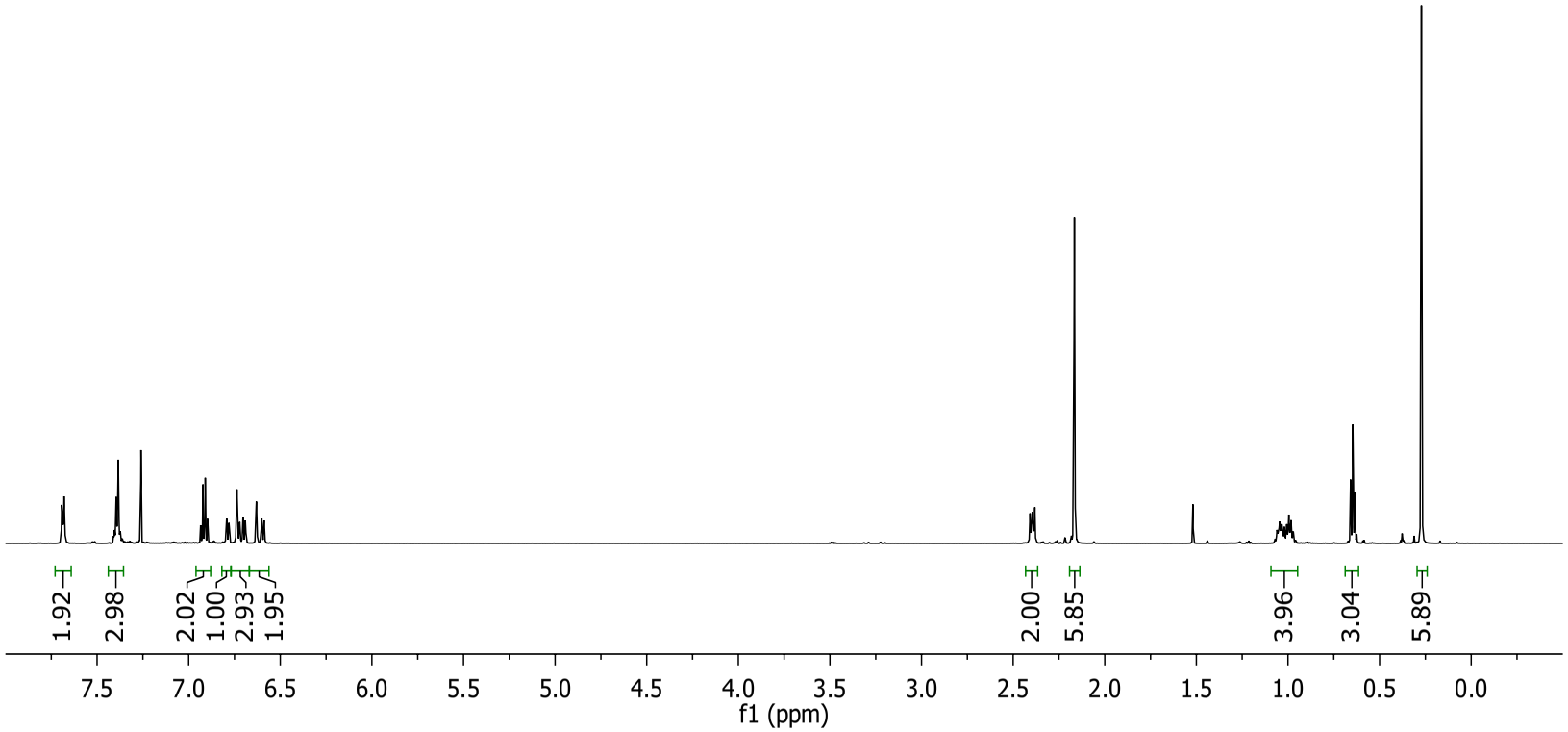


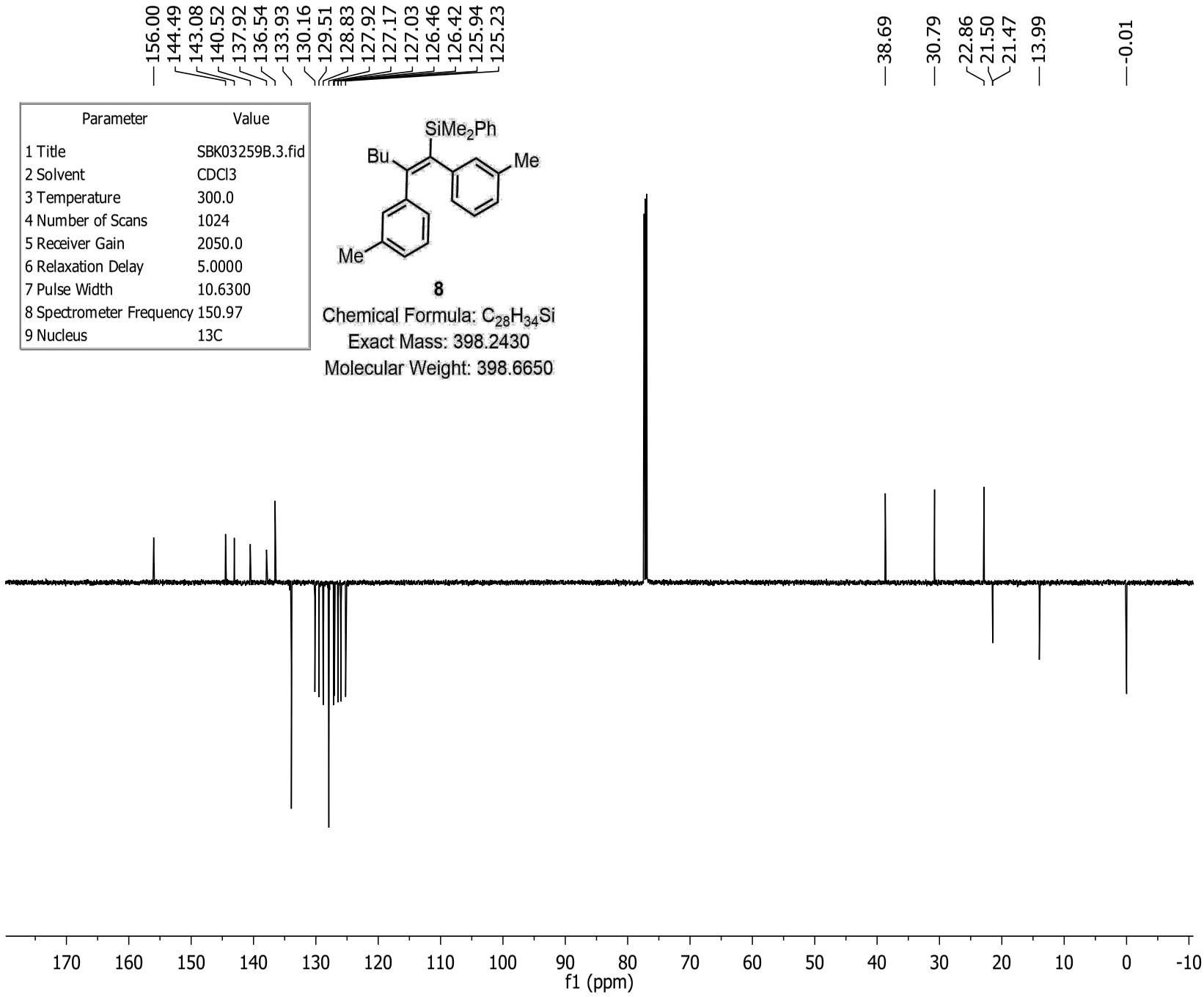
8

Chemical Formula: $C_{28}H_{34}Si$

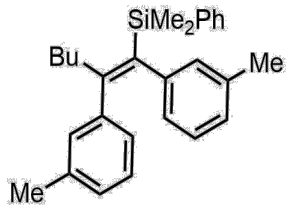
Exact Mass: 398.2430

Molecular Weight: 398.6650




156.00
144.49
143.08
140.52
137.92
136.54
133.93
130.16
129.51
128.83
127.92
127.17
127.03
126.46
126.42
125.94
125.23
38.69
30.79
22.86
21.50
21.47
13.99
-0.01
Parameter Value
1 Title SBK03259B.3.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Ph
Bu
Me
Me
8
Chemical Formula: C28H34Si
Exact Mass: 398.2430
Molecular Weight: 398.6650
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

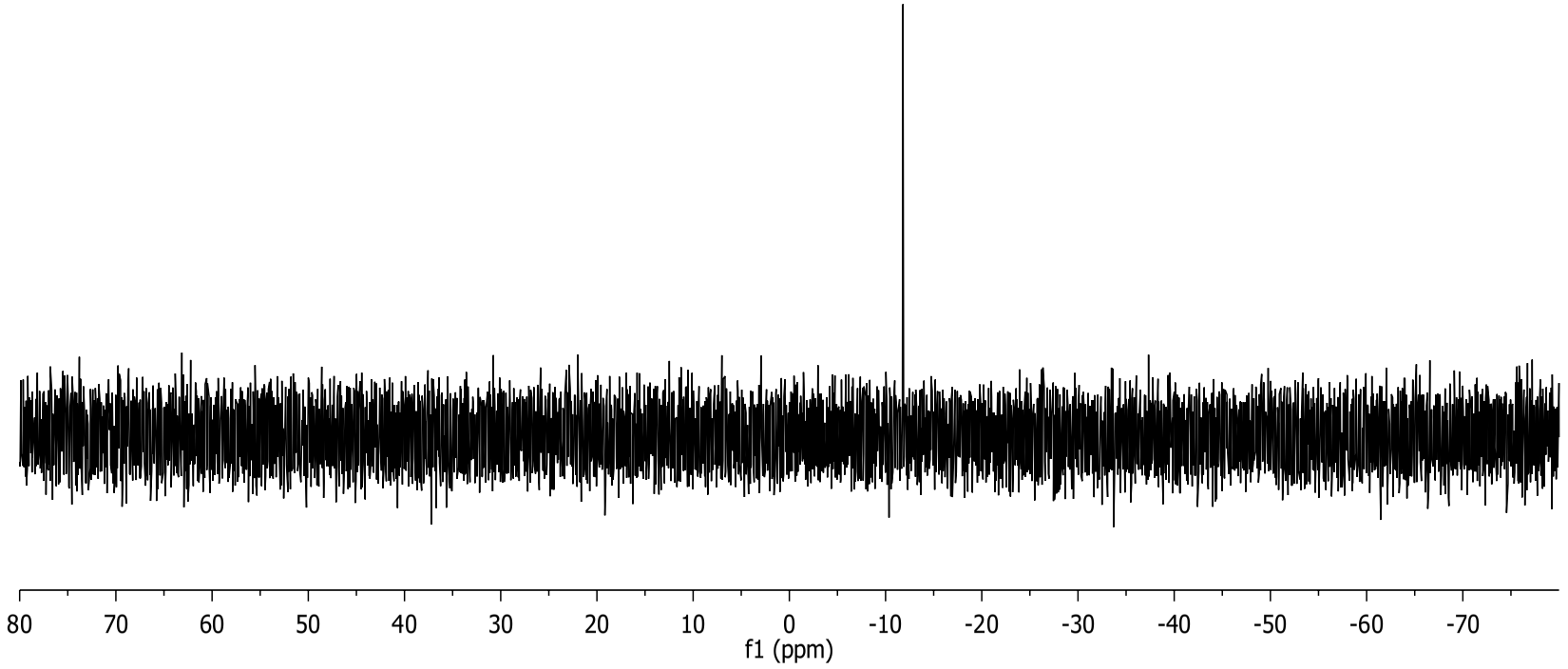
Parameter	Value
1 Title	SBK03259B.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

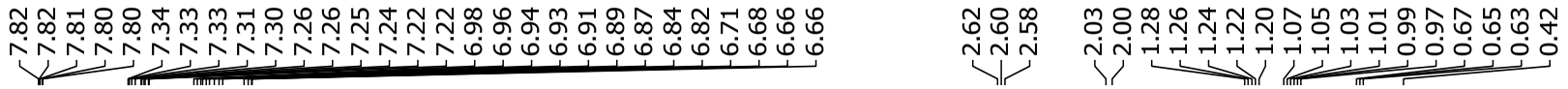


8

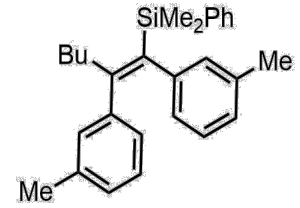
Chemical Formula: $C_{28}H_{34}Si$
Exact Mass: 398.2430
Molecular Weight: 398.6650

-11.81





Parameter	Value
1 Title	SBK03259B-C6D6.1.fid
2 Solvent	C6D6
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	12.7
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

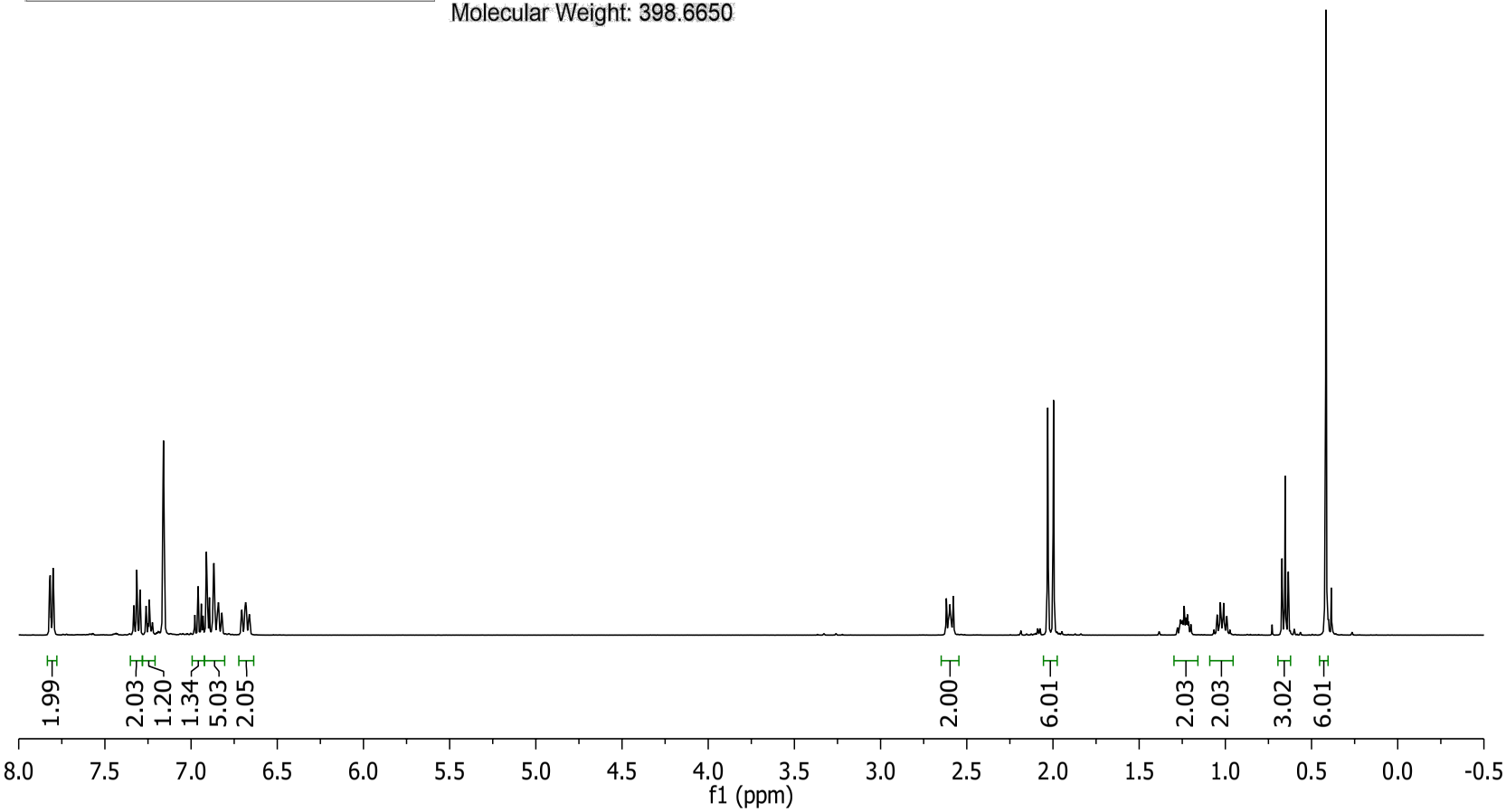


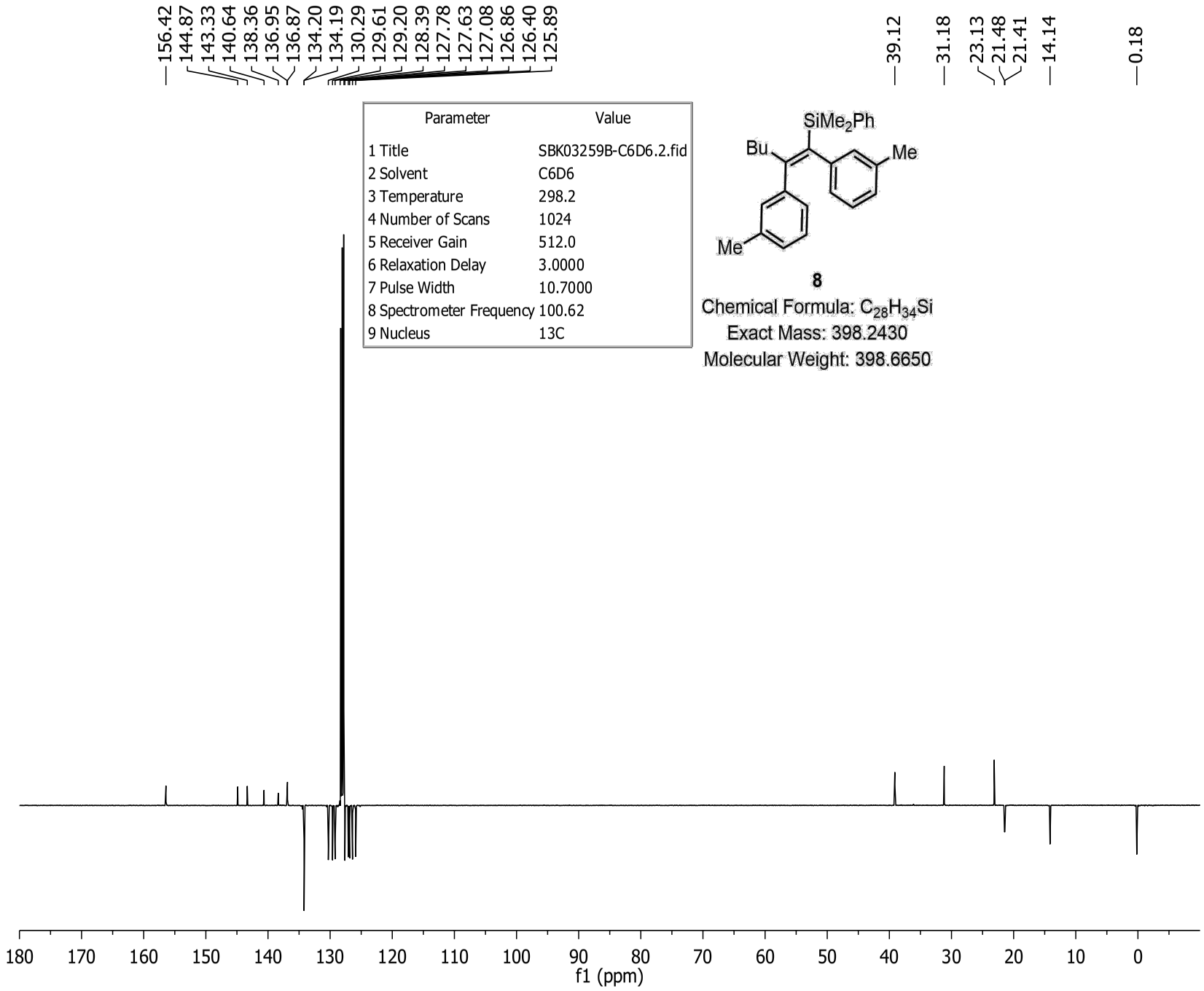
8

Chemical Formula: $C_{28}H_{34}Si$

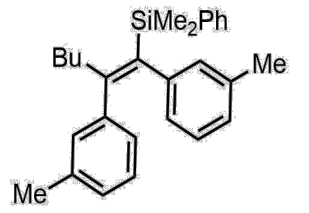
Exact Mass: 398.2430

Molecular Weight: 398.6650




156.42
144.87
143.33
140.64
138.36
136.95
136.87
134.20
134.19
130.29
129.61
129.20
128.39
127.78
127.63
127.08
126.86
126.40
125.89
39.12
31.18
23.13
21.48
21.41
14.14
0.18
Parameter Value
1 Title SBK03259B-C6D6.2.fid
2 Solvent C6D6
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Bu
Me
Me
8
Chemical Formula: C28H34Si
Exact Mass: 398.2430
Molecular Weight: 398.6650
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

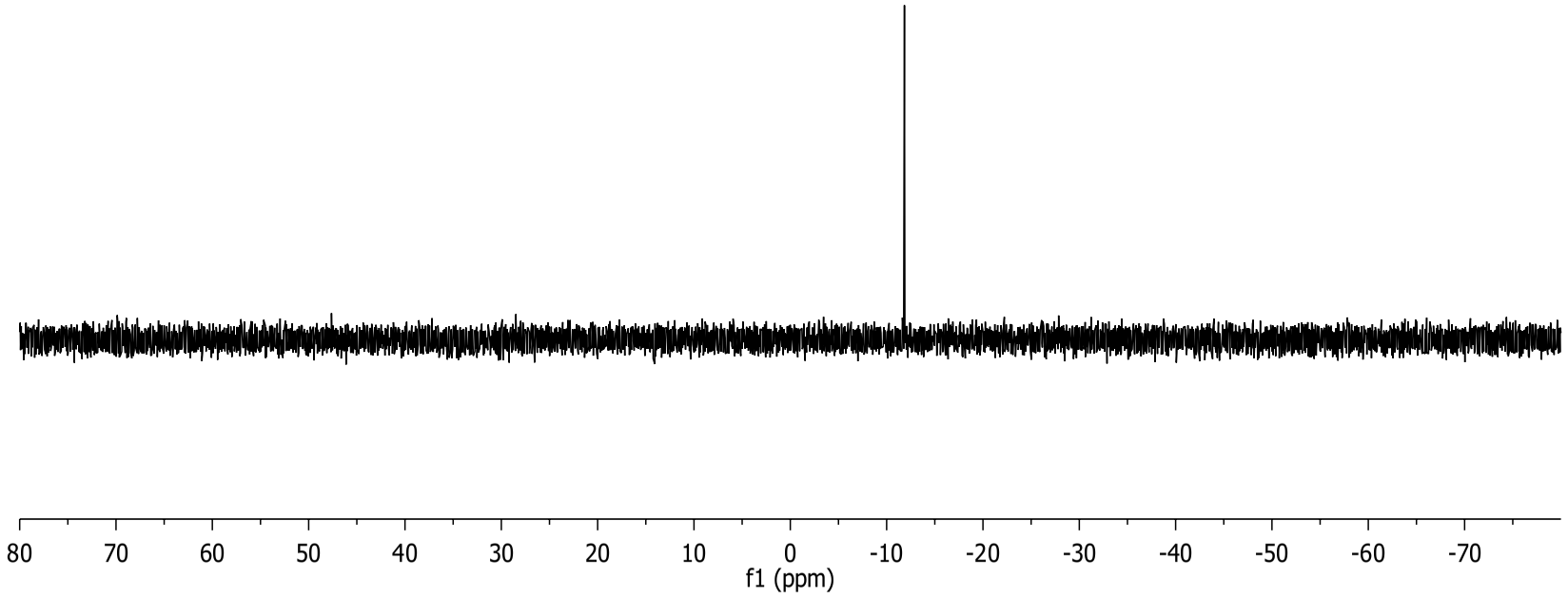
Parameter	Value
1 Title	SBK03259B.4.fid
2 Solvent	C6D6
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

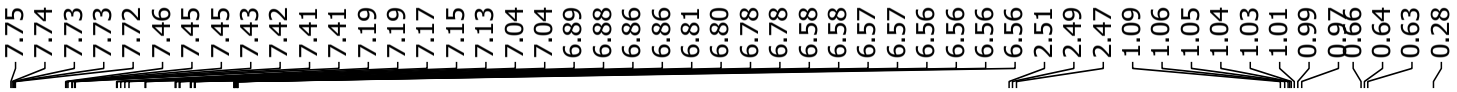


8

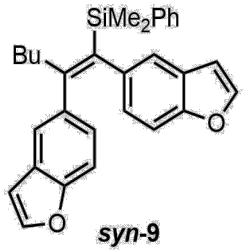
Chemical Formula: $C_{28}H_{34}Si$
Exact Mass: 398.2430
Molecular Weight: 398.6650

-11.85





Parameter	Value
1 Title	MFW02093col2C-dry2.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	11.3
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

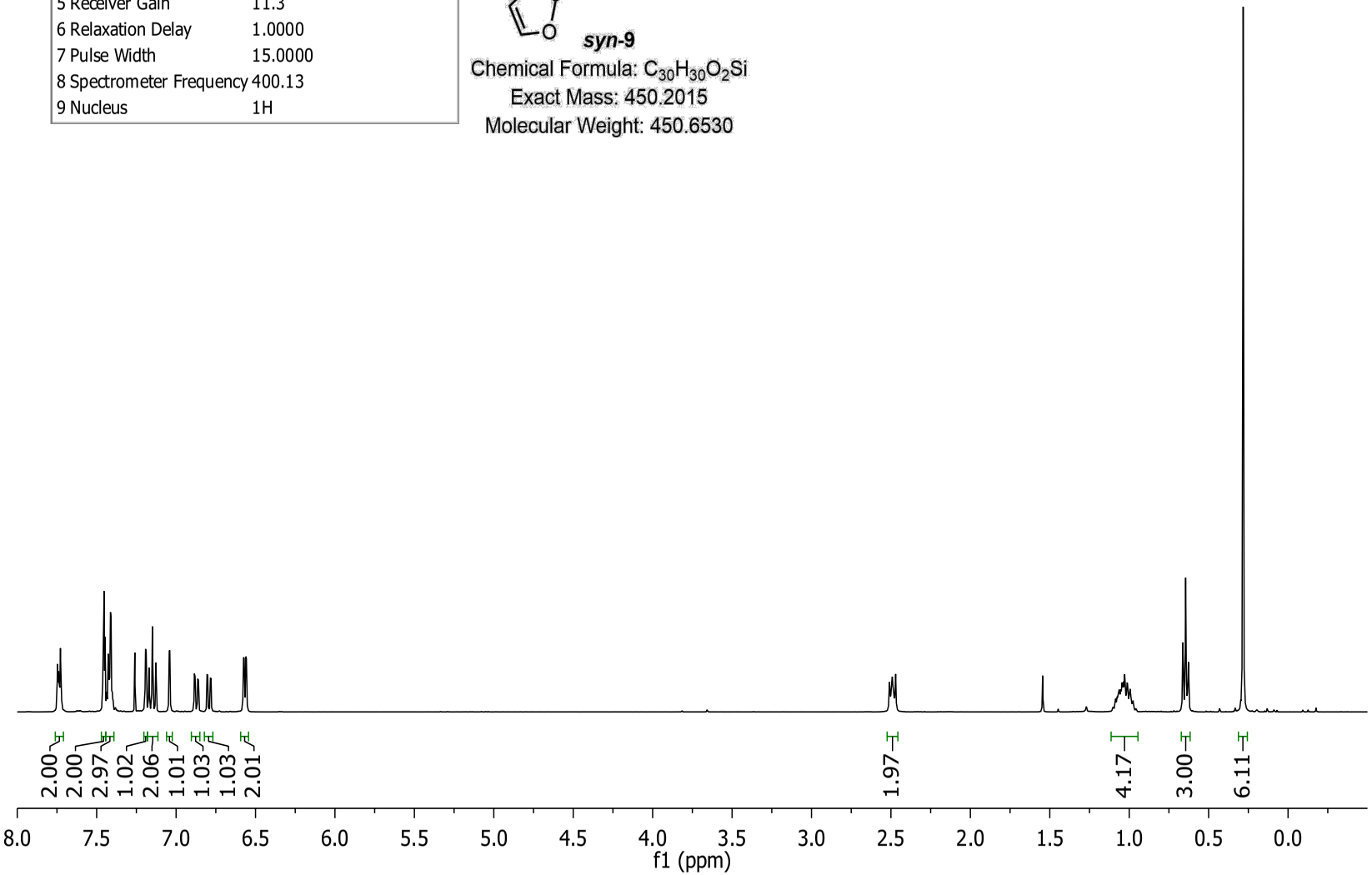


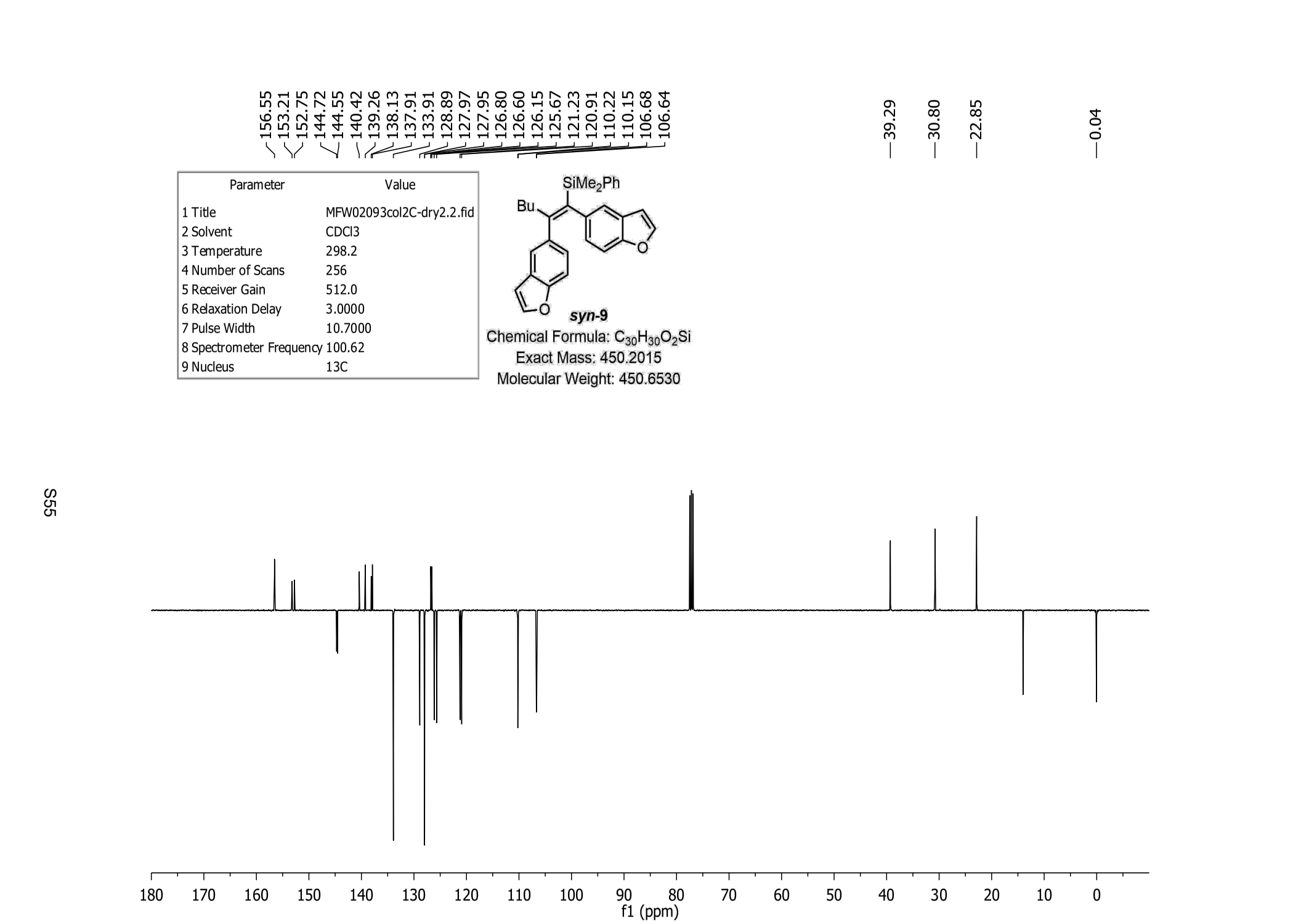
***syn*-9**

Chemical Formula: $C_{30}H_{30}O_2Si$

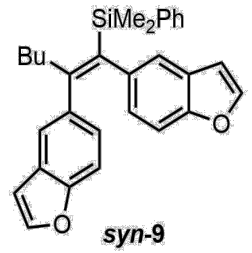
Exact Mass: 450.2015

Molecular Weight: 450.6530




156.55
153.21
152.75
144.72
144.55
140.42
139.26
138.13
137.91
133.91
128.89
127.97
127.95
126.80
126.60
126.15
125.67
121.23
120.91
110.22
110.15
106.68
106.64
39.29
30.80
22.85
0.04
Parameter
Value
1 Title
MFW02093col2C-dry2.2.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Ph
Bu
O
O
syn-9
Chemical Formula: C30H30O2Si
Exact Mass: 450.2015
Molecular Weight: 450.6530
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Parameter	Value
1 Title	MFW02093col2C-dry3.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

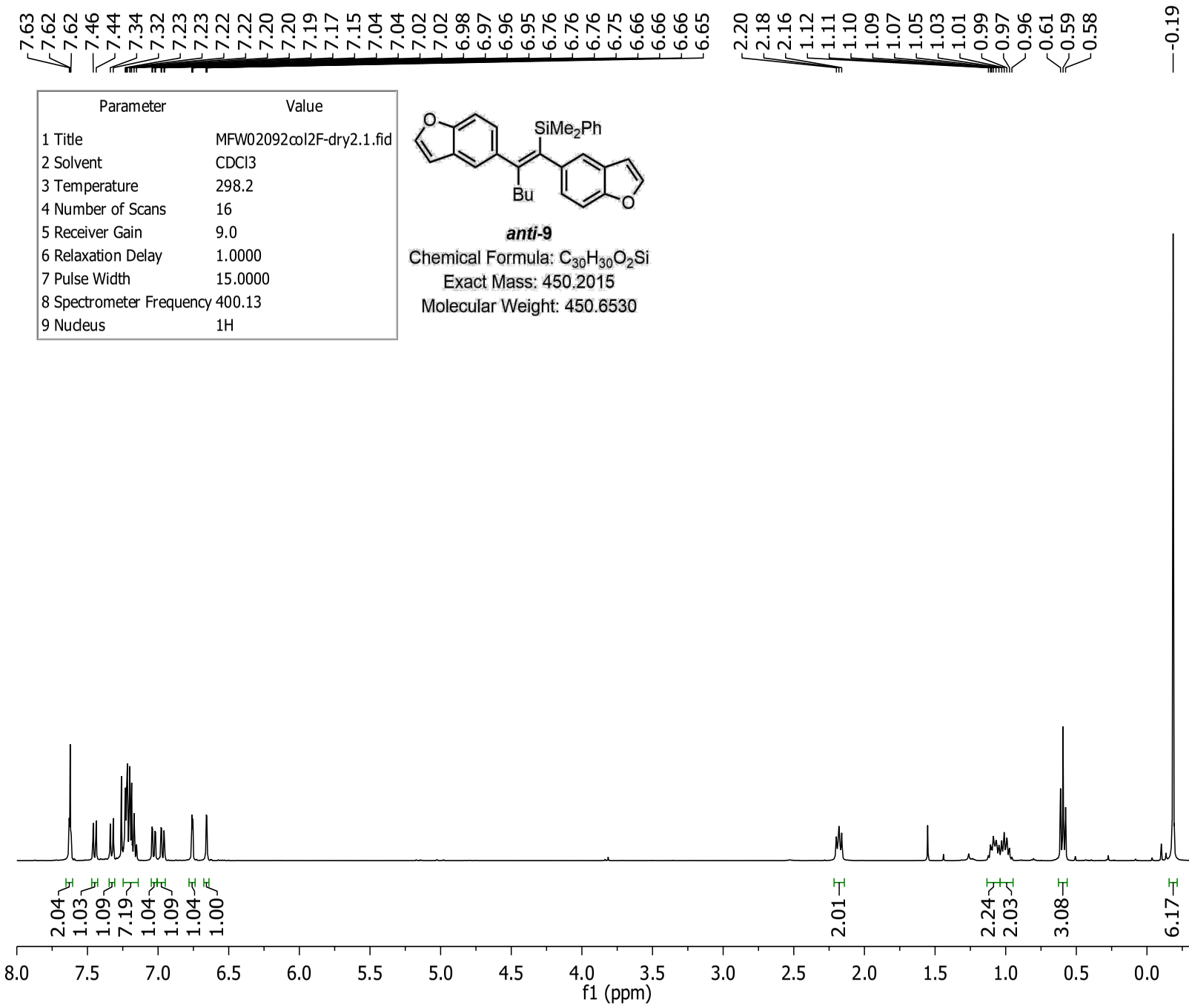


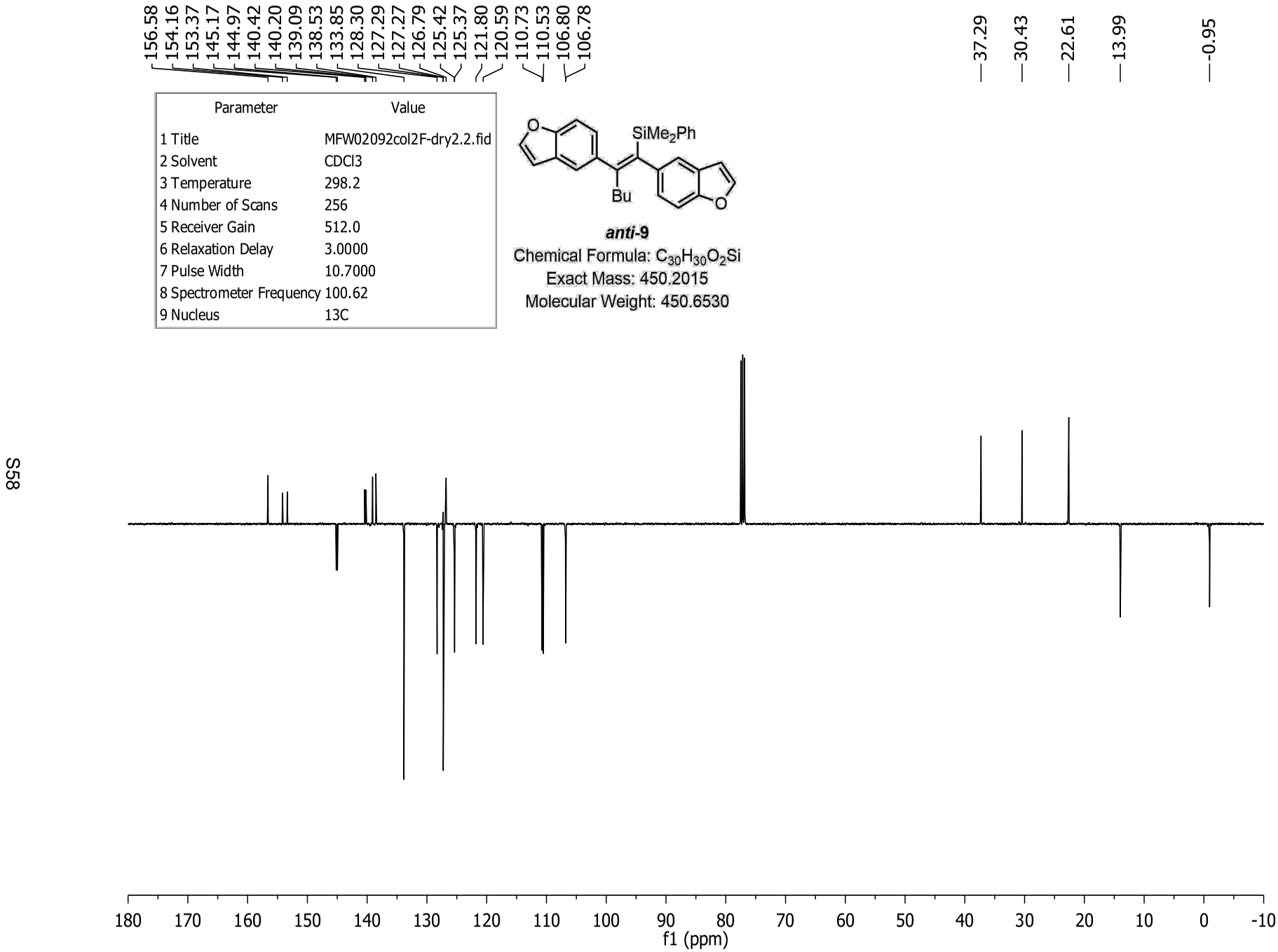
***syn*-9**

Chemical Formula: $C_{30}H_{30}O_2Si$

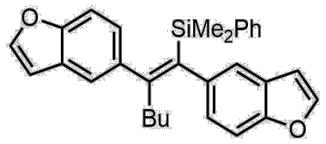
Exact Mass: 450.2015

Molecular Weight: 450.6530


7.63 7.62 7.62 7.46 7.44 7.34 7.32 7.23 7.23 7.22 7.22 7.20 7.20 7.19 7.17 7.15 7.04 7.04 7.02 7.02 6.98 6.97 6.96 6.95 6.76 6.76 6.76 6.75 6.66 6.66 6.66 6.65
2.20 2.18 2.16 1.12 1.11 1.10 1.09 1.07 1.05 1.03 1.01 0.99 0.97 0.96 0.61 0.59 0.58
-0.19
Parameter Value
1 Title MFW02092col2F-dry2.1.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 16
5 Receiver Gain 9.0
6 Relaxation Delay 1.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 400.13
9 Nucleus 1H
SiMe2Ph
Bu
O
O
anti-9
Chemical Formula: C30H30O2Si
Exact Mass: 450.2015
Molecular Weight: 450.6530
2.04 1.03 1.09 7.19 1.04 1.09 1.04 1.00
2.01 2.24 2.03 3.08 6.17
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


156.58
154.16
153.37
145.17
144.97
140.42
140.20
139.09
138.53
133.85
128.30
127.29
127.27
126.79
125.42
125.37
121.80
120.59
110.73
110.53
106.80
106.78
37.29
30.43
22.61
13.99
-0.95
Parameter Value
1 Title MFW02092col2F-dry2.2.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 256
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Bu
O
O
anti-9
Chemical Formula: C30H30O2Si
Exact Mass: 450.2015
Molecular Weight: 450.6530
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Parameter	Value
1 Title	MFW02092col2F-dry3.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

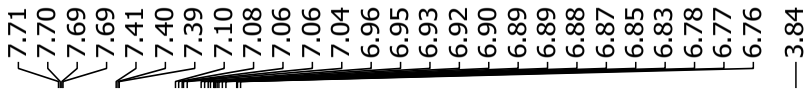


***anti*-9**

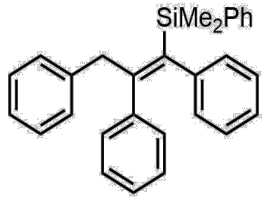
Chemical Formula: $C_{30}H_{30}O_2Si$

Exact Mass: 450.2015

Molecular Weight: 450.6530



Parameter	Value
1 Title	MFW02062col2A.3.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	12.7
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

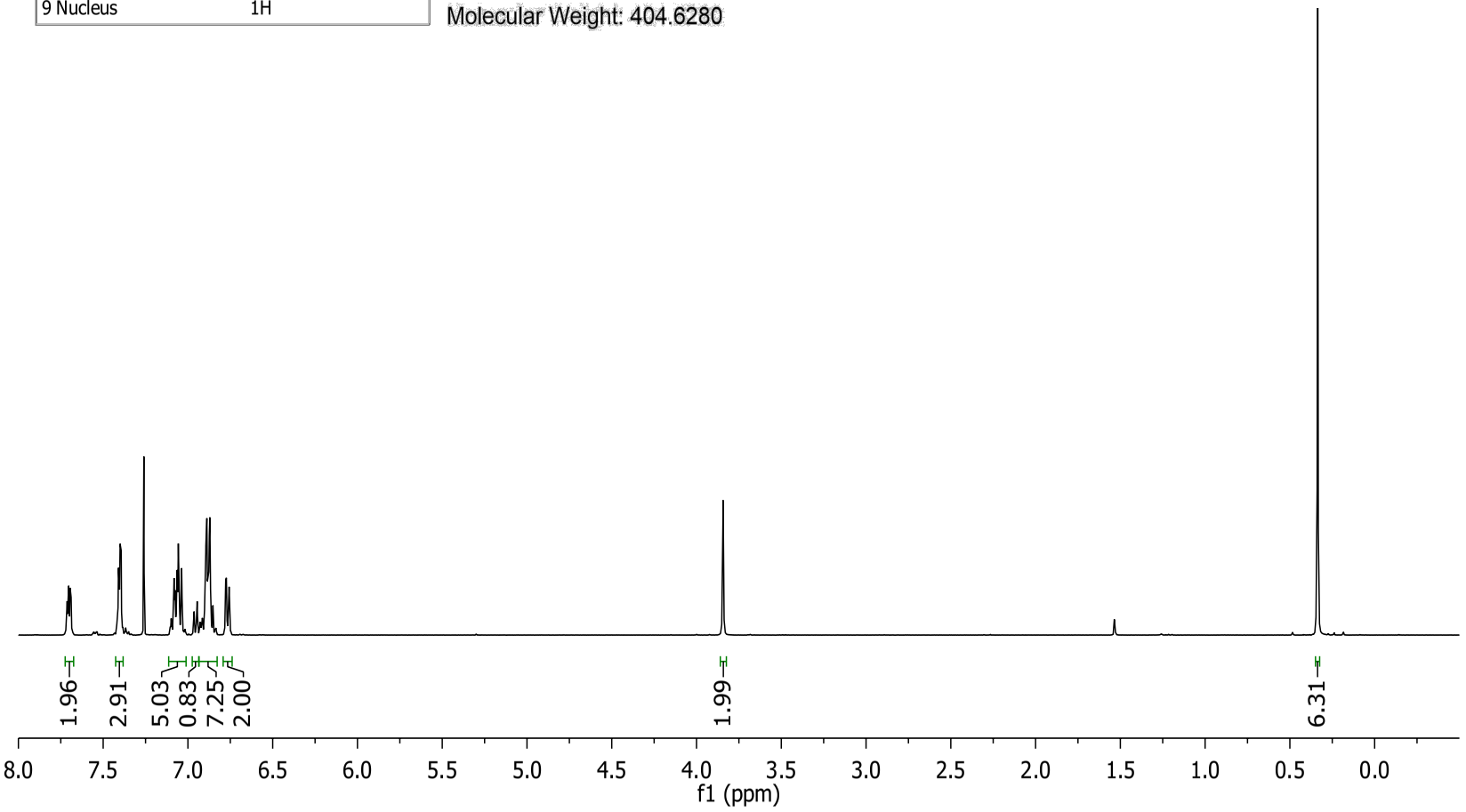


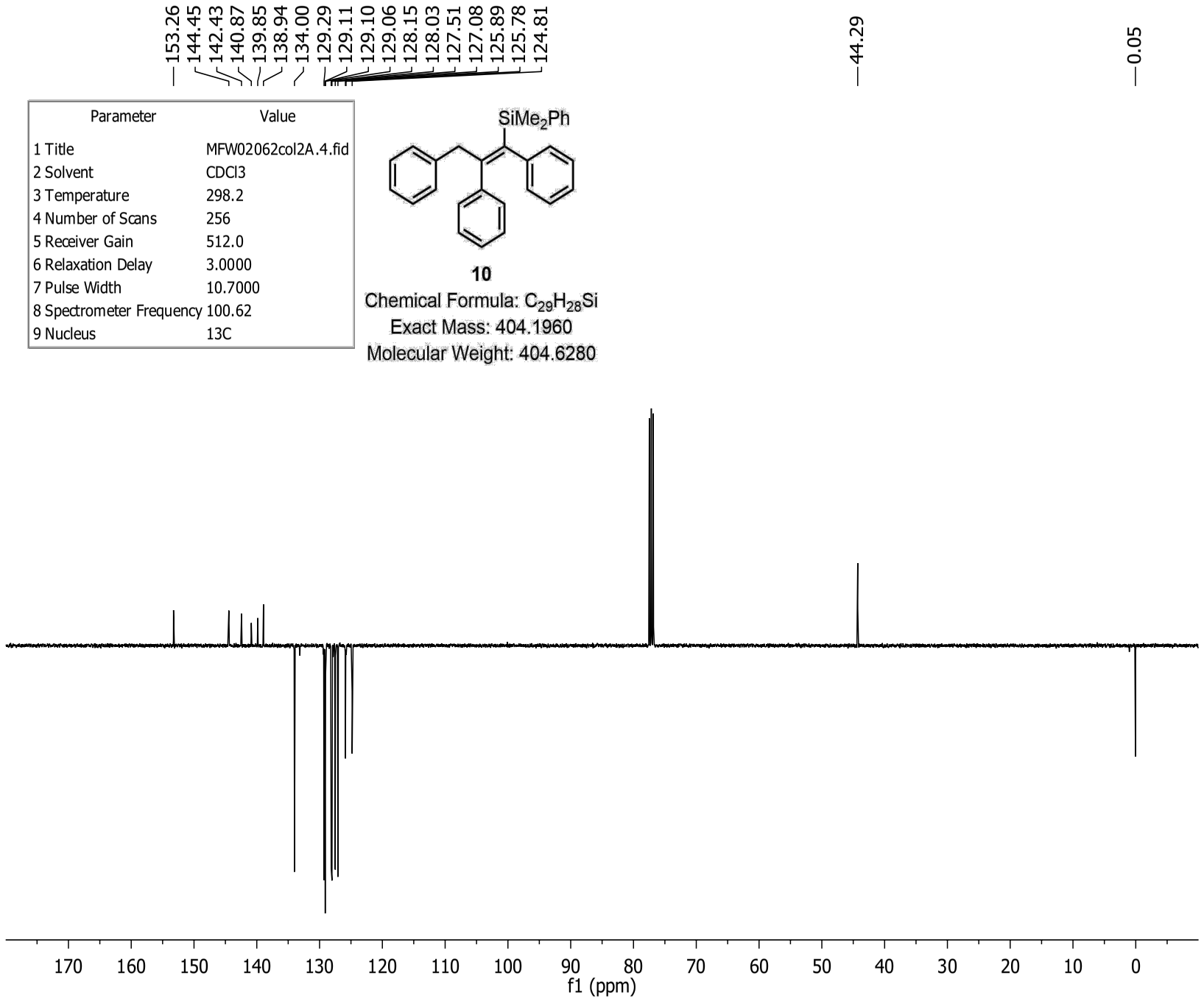
10

Chemical Formula: $C_{29}H_{28}Si$

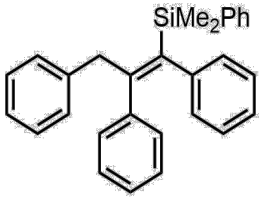
Exact Mass: 404.1960

Molecular Weight: 404.6280




153.26
144.45
142.43
140.87
139.85
138.94
134.00
129.29
129.11
129.10
129.06
128.15
128.03
127.51
127.08
125.89
125.78
124.81
44.29
0.05
Parameter Value
1 Title MFW02062col2A.4.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 256
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
10
Chemical Formula: C29H28Si
Exact Mass: 404.1960
Molecular Weight: 404.6280
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

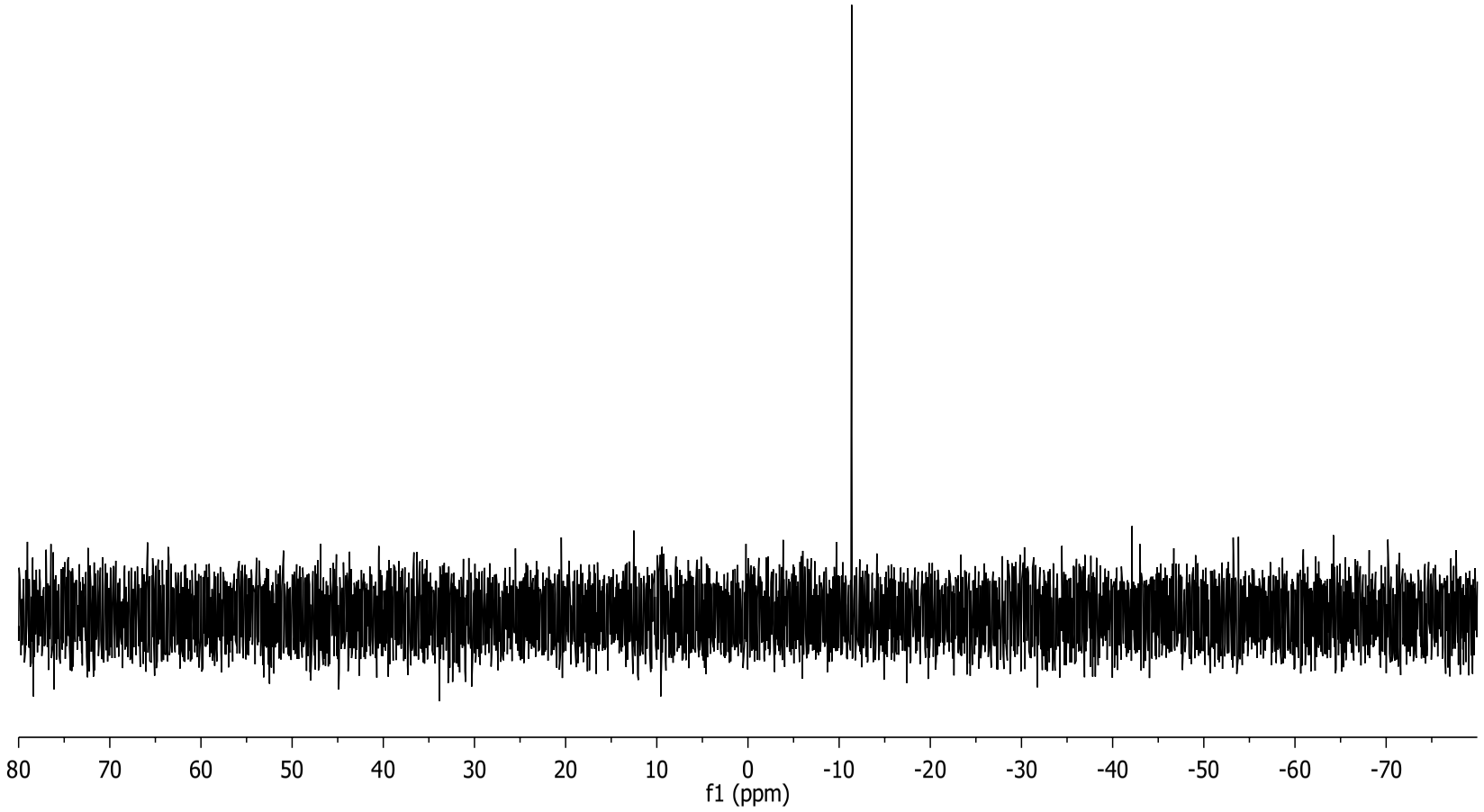
Parameter	Value
1 Title	MFW02062col2A-Si.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	128
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

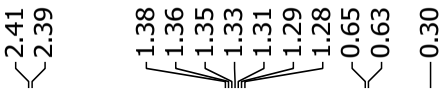


10

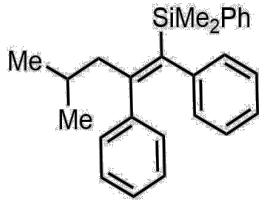
Chemical Formula: $C_{29}H_{28}Si$
Exact Mass: 404.1960
Molecular Weight: 404.6280

-11.38





Parameter	Value
1 Title	SBK03258B.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	9.0
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

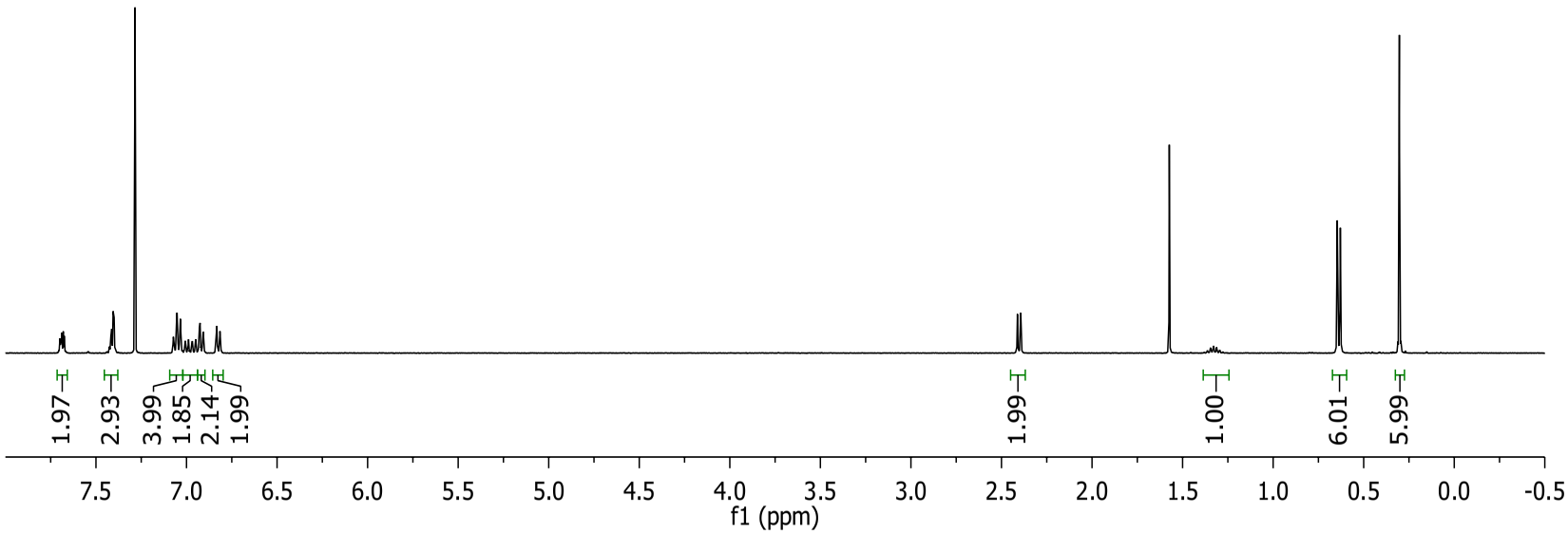


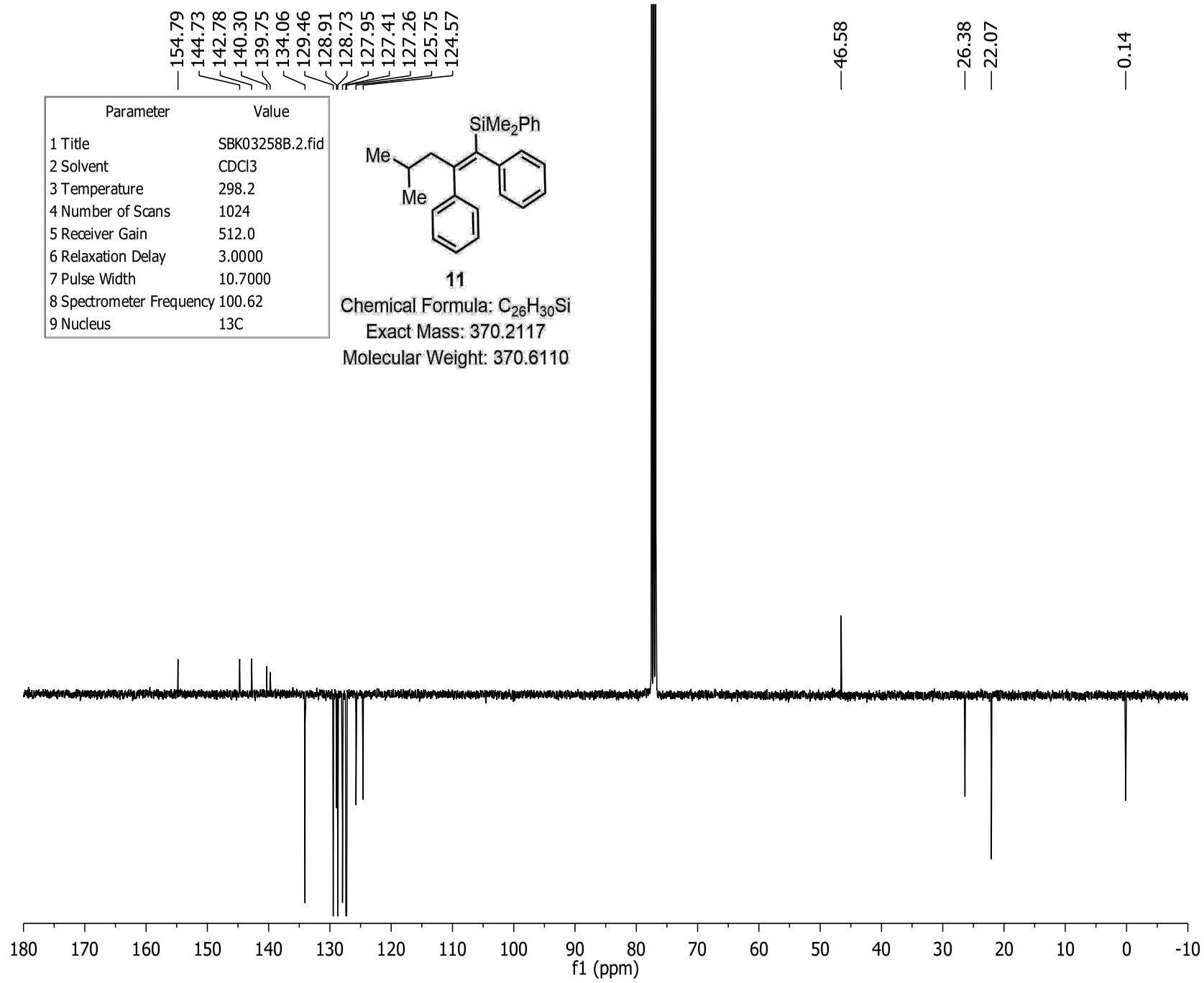
11

Chemical Formula: $C_{26}H_{30}Si$

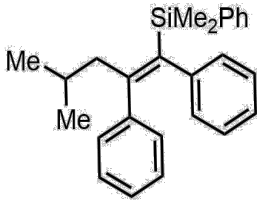
Exact Mass: 370.2117

Molecular Weight: 370.6110




154.79
144.73
142.78
140.30
139.75
134.06
129.46
128.91
128.73
127.95
127.41
127.26
125.75
124.57
46.58
26.38
22.07
0.14
Parameter Value
1 Title SBK03258B.2.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Me
Me
11
Chemical Formula: C26H30Si
Exact Mass: 370.2117
Molecular Weight: 370.6110
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

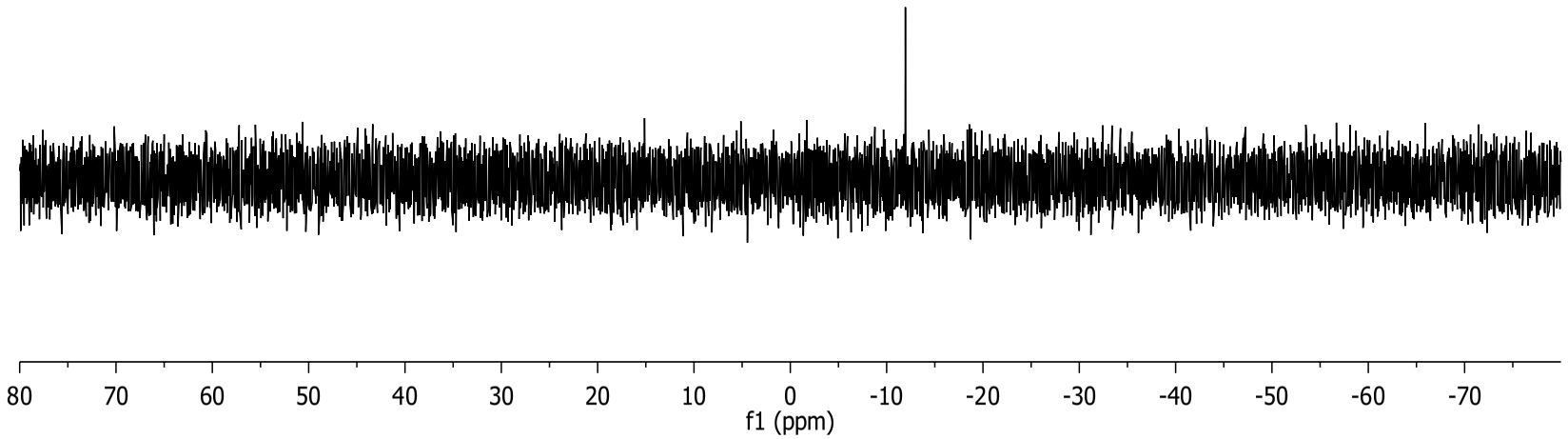
Parameter	Value
1 Title	SBK03258B-Si.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



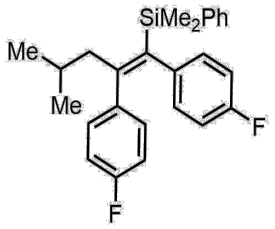
11

Chemical Formula: $C_{26}H_{30}Si$
Exact Mass: 370.2117
Molecular Weight: 370.6110

-11.95



Parameter	Value
1 Title	SBK03276C.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	203.0
6 Relaxation Delay	1.0000
7 Pulse Width	12.2500
8 Spectrometer Frequency	400.15
9 Nucleus	1H

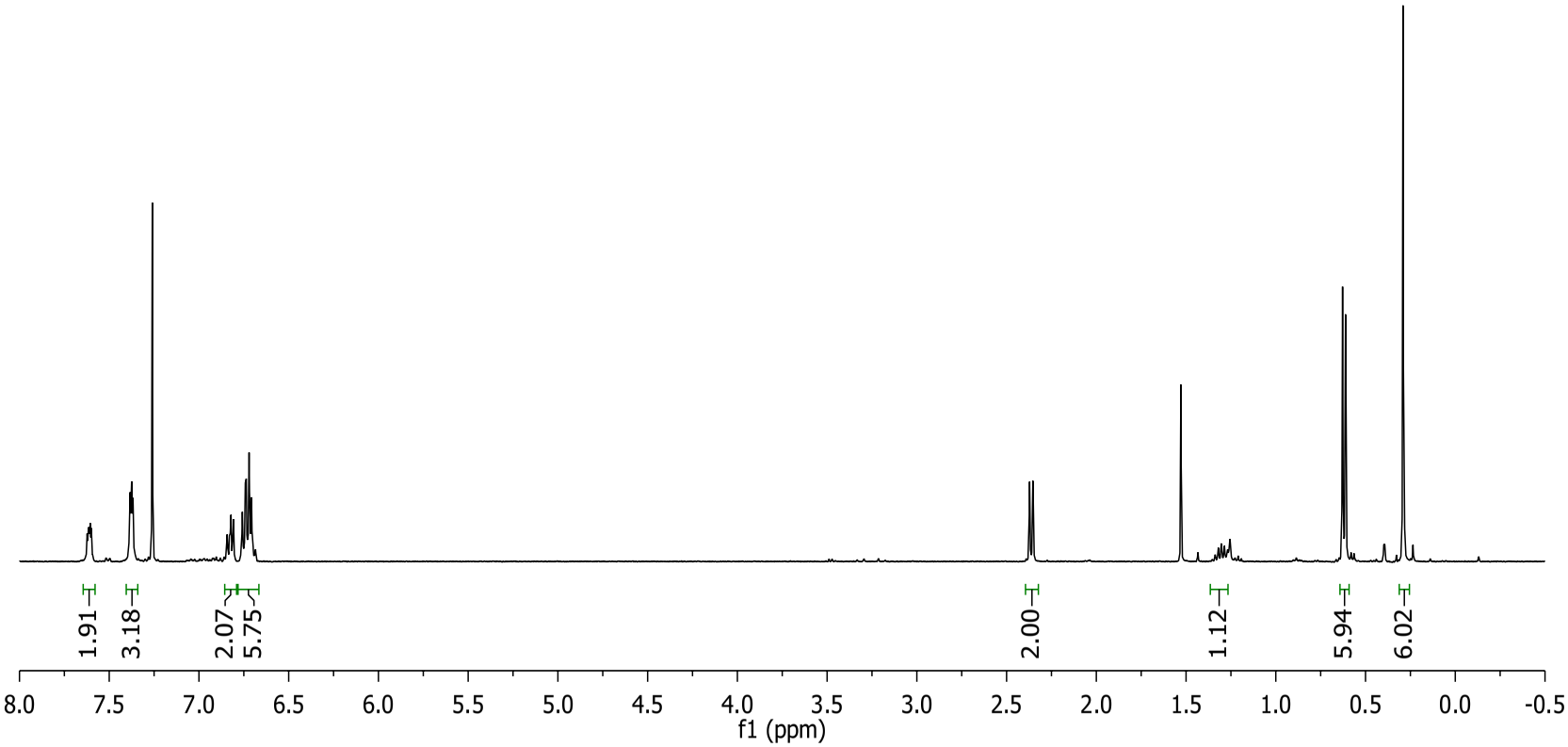


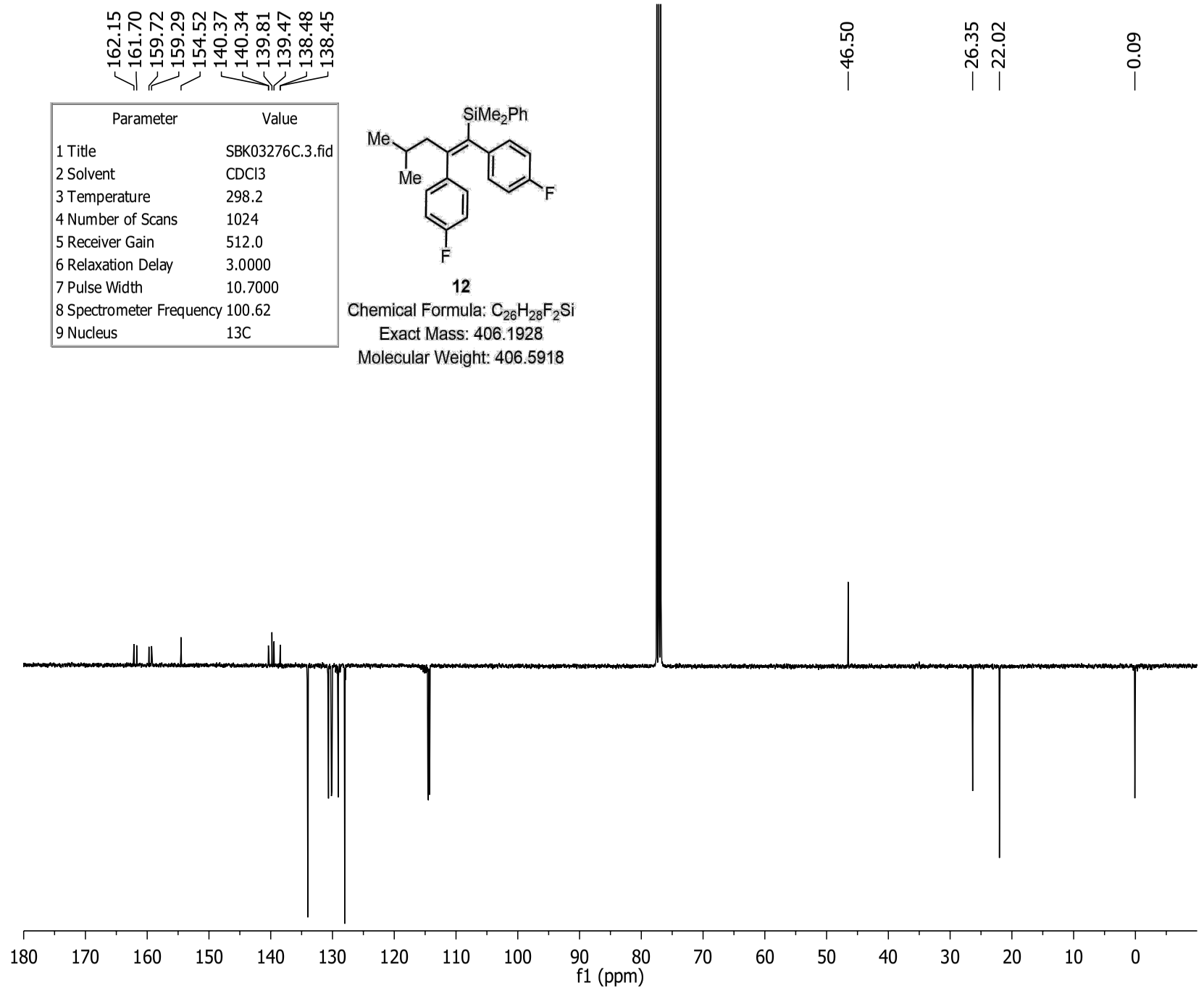
12

Chemical Formula: $C_{26}H_{28}F_2Si$

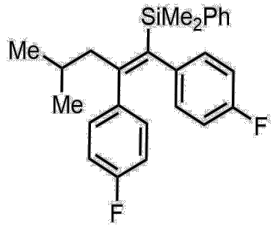
Exact Mass: 406.1928

Molecular Weight: 406.5918




162.15
161.70
159.72
159.29
154.52
140.37
140.34
139.81
139.47
138.48
138.45
46.50
26.35
22.02
0.09
Parameter
Value
1 Title
SBK03276C.3.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
1024
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Ph
Me
Me
F
F
12
Chemical Formula: C26H28F2Si
Exact Mass: 406.1928
Molecular Weight: 406.5918
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Parameter	Value
1 Title	SBK03276C-Si.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



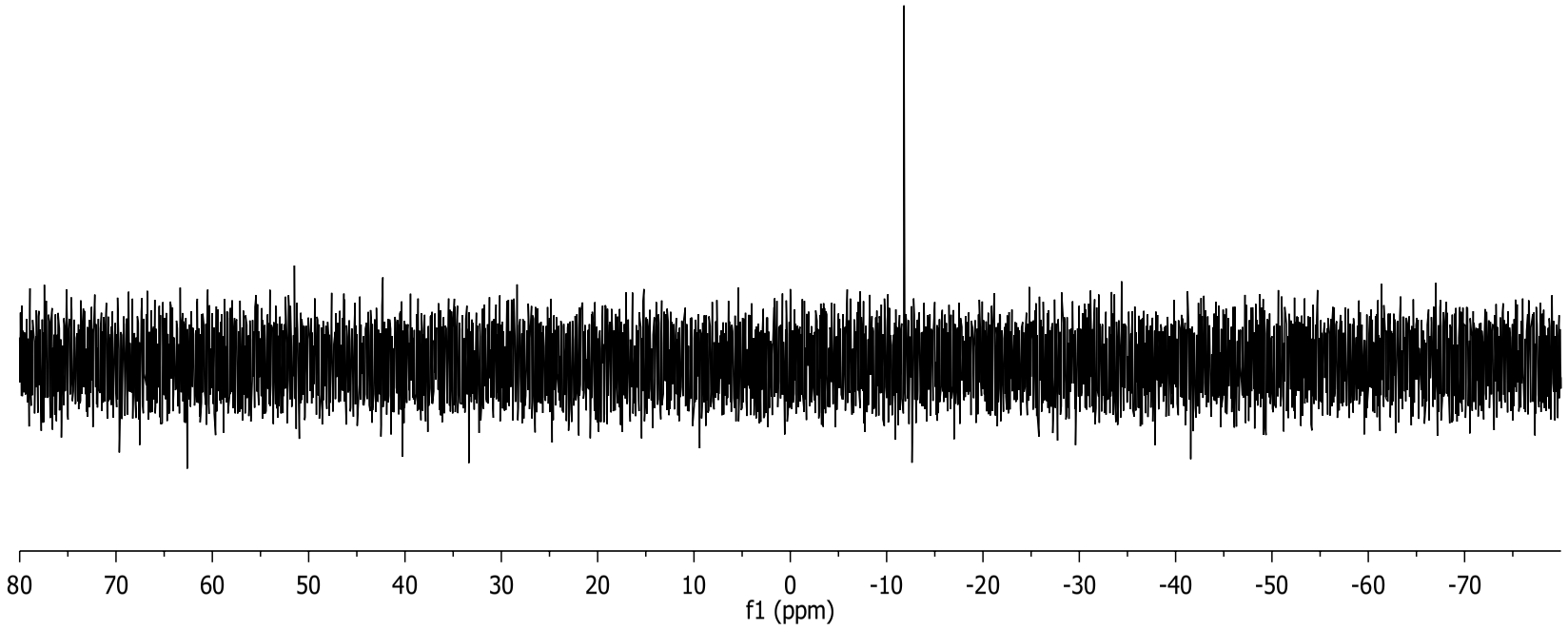
12

Chemical Formula: $C_{26}H_{28}F_2Si$

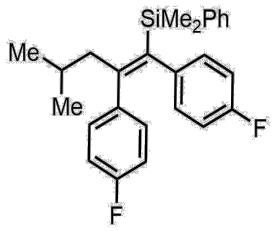
Exact Mass: 406.1928

Molecular Weight: 406.5918

-11.80



Parameter	Value
1 Title	SBK03276.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	322.0
6 Relaxation Delay	3.0000
7 Pulse Width	11.4000
8 Spectrometer Frequency	564.81
9 Nucleus	19F

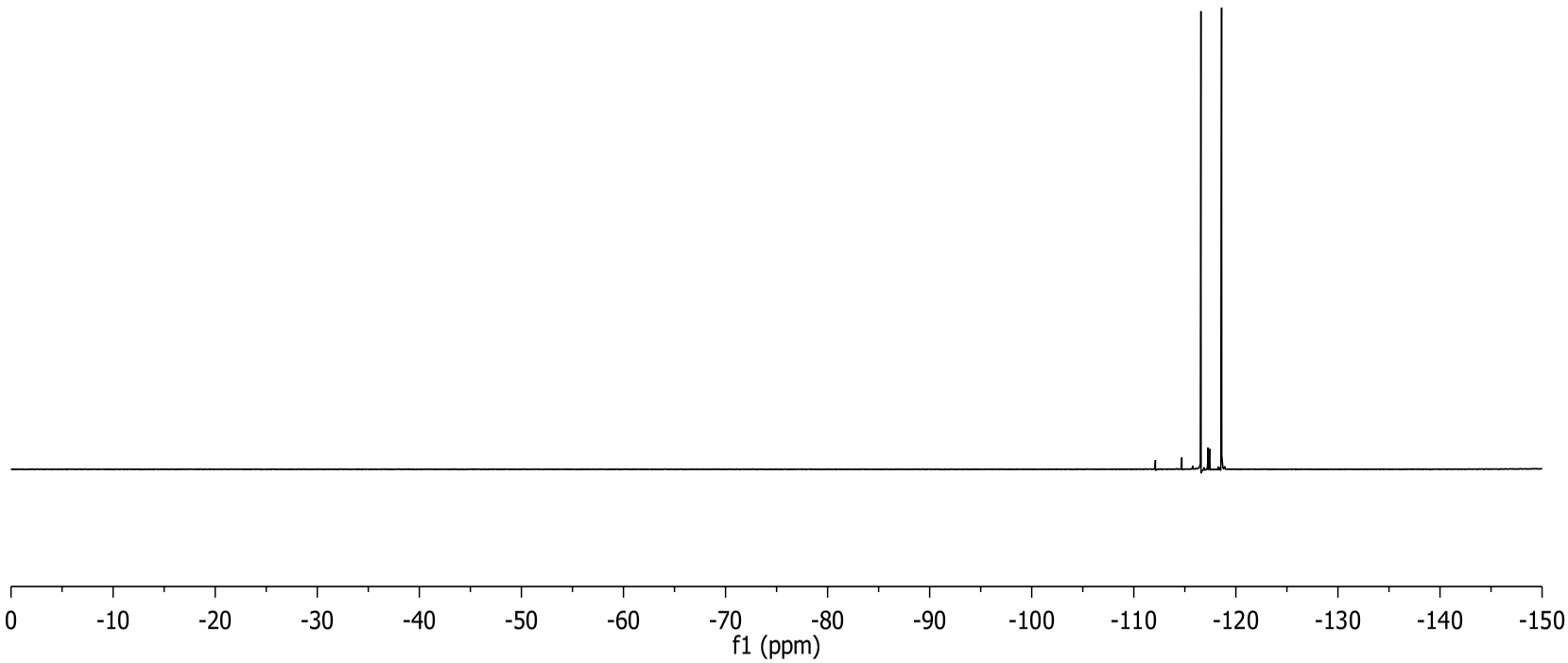


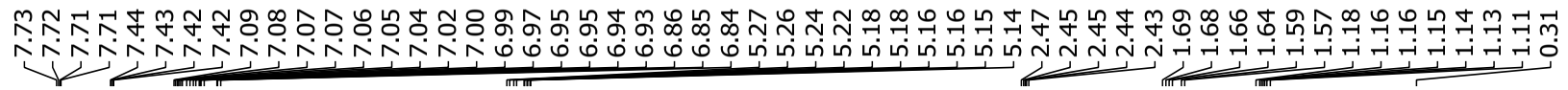
12

Chemical Formula: $C_{26}H_{28}F_2Si$

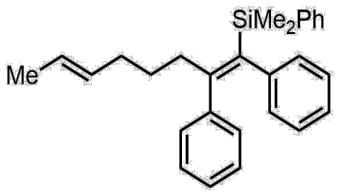
Exact Mass: 406.1928

Molecular Weight: 406.5918





Parameter	Value
1 Title	MFW02088col2E-dry2.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	10.1
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

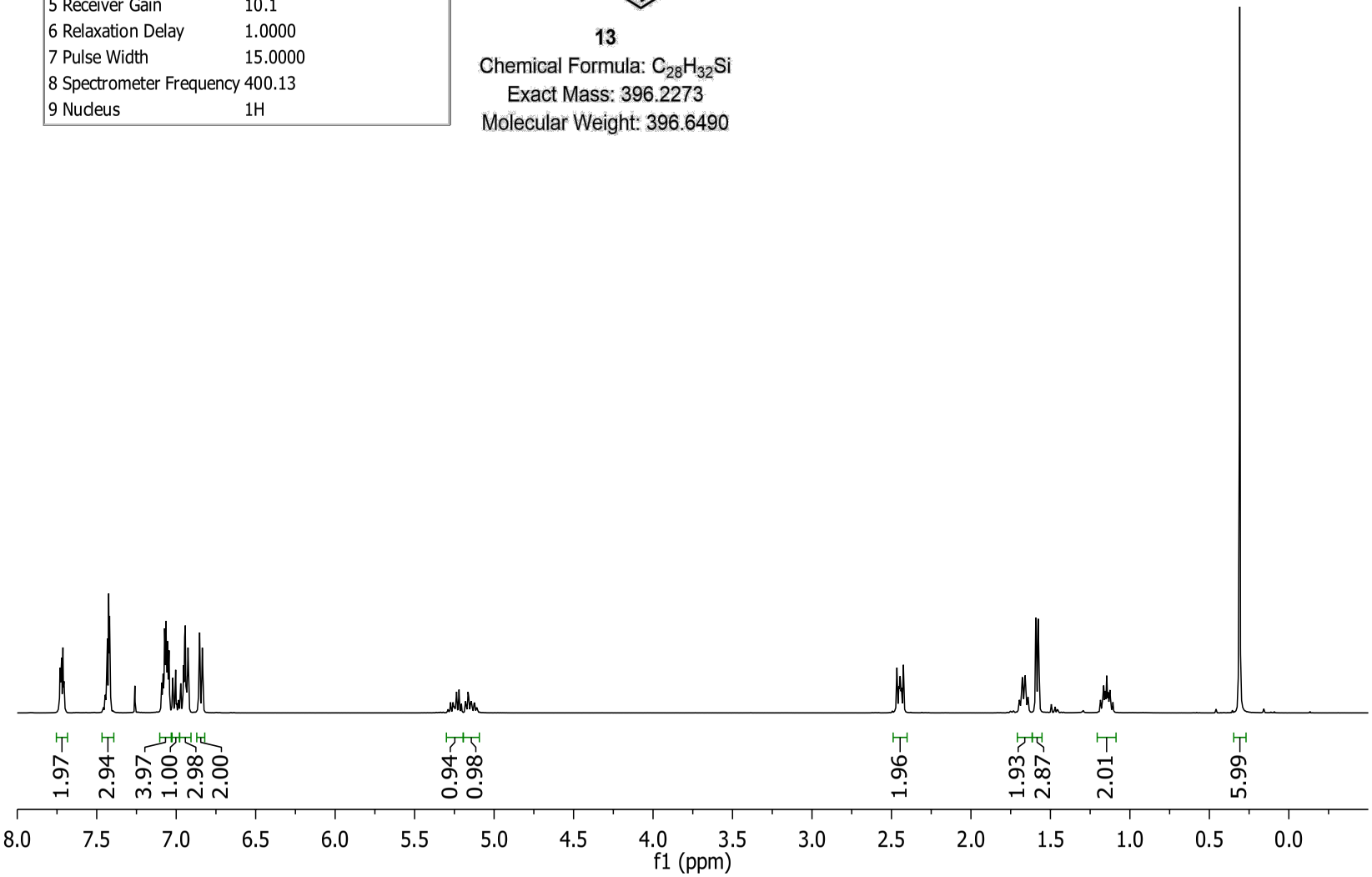


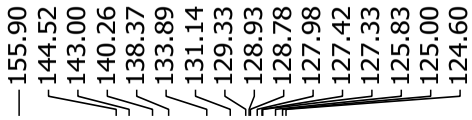
13

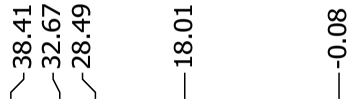
Chemical Formula: $C_{28}H_{32}Si$

Exact Mass: 396.2273

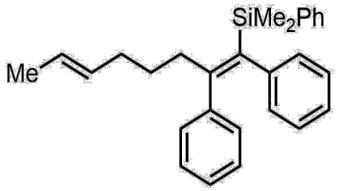
Molecular Weight: 396.6490







Parameter	Value
1 Title	MFW02088col2E-dry2.2.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	256
5 Receiver Gain	512.0
6 Relaxation Delay	3.0000
7 Pulse Width	10.7000
8 Spectrometer Frequency	100.62
9 Nucleus	13C

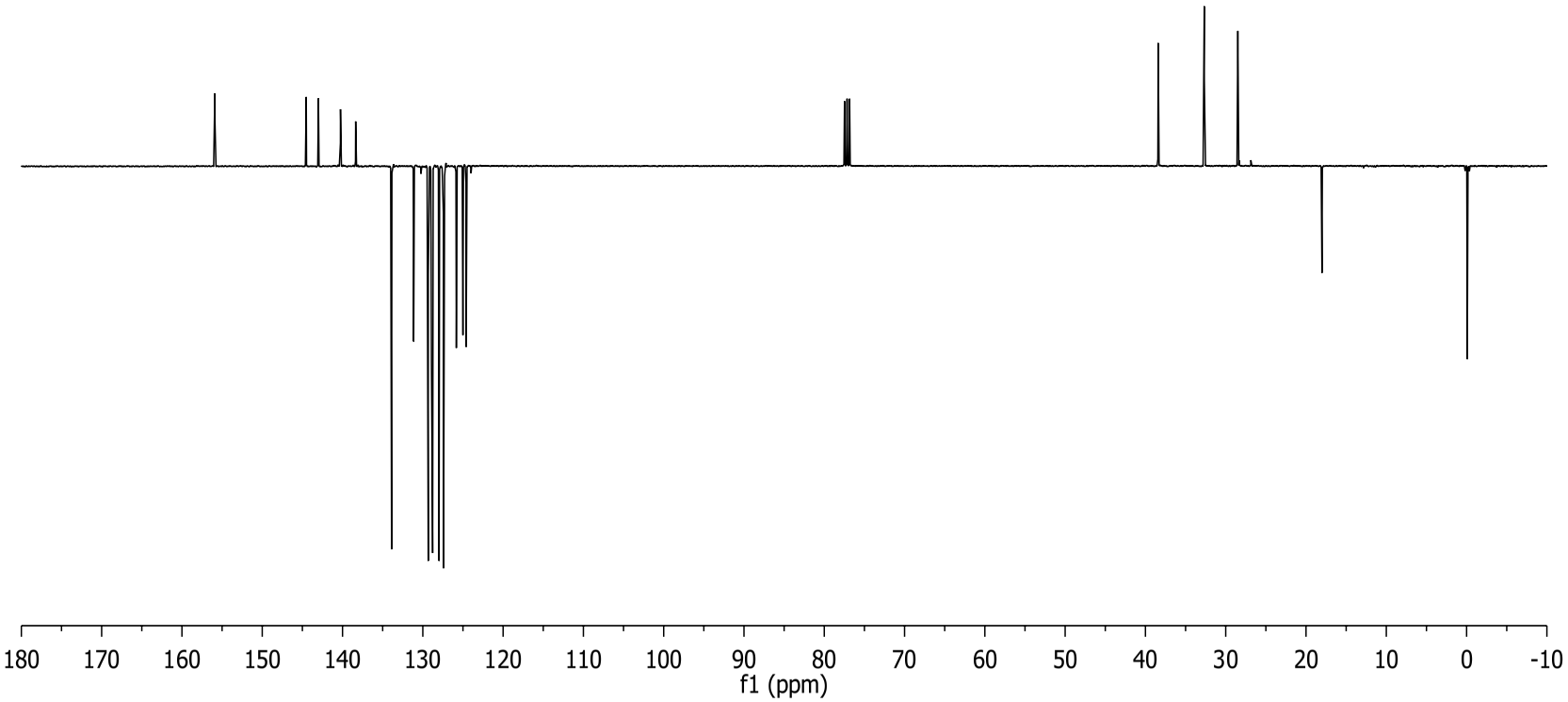


13

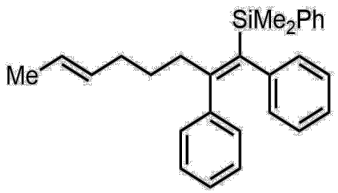
Chemical Formula: $C_{28}H_{32}Si$

Exact Mass: 396.2273

Molecular Weight: 396.6490

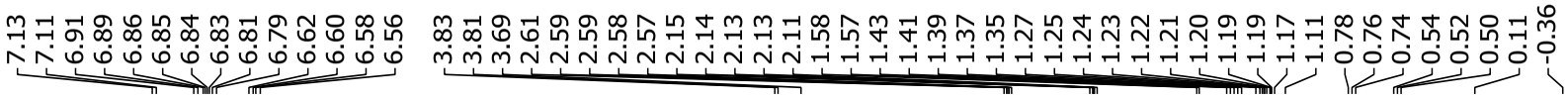


Parameter	Value
1 Title	MFW02088col2E-dry3.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

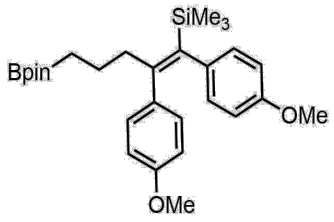


13

Chemical Formula: $C_{28}H_{32}Si$
Exact Mass: 396.2273
Molecular Weight: 396.6490

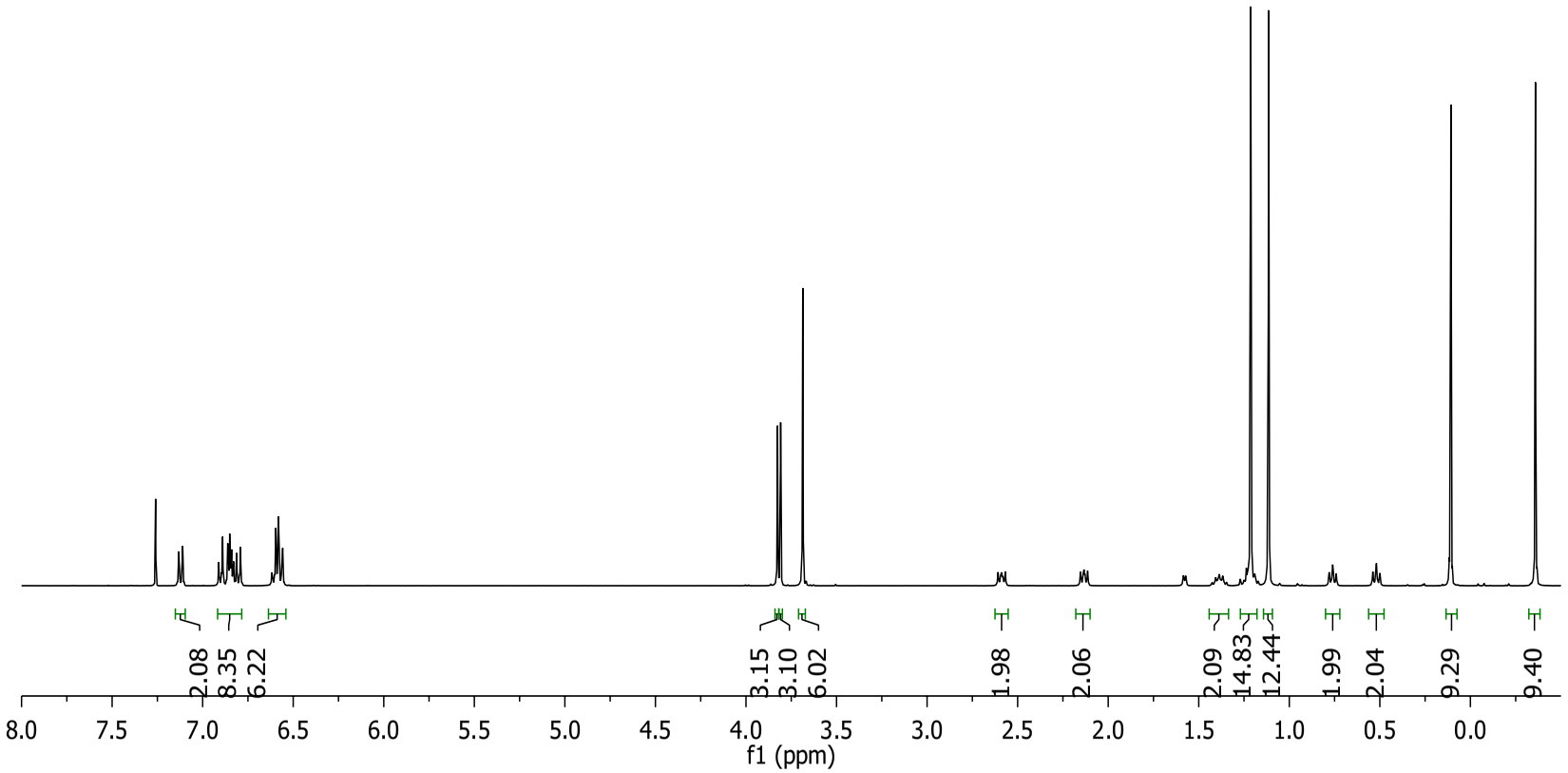


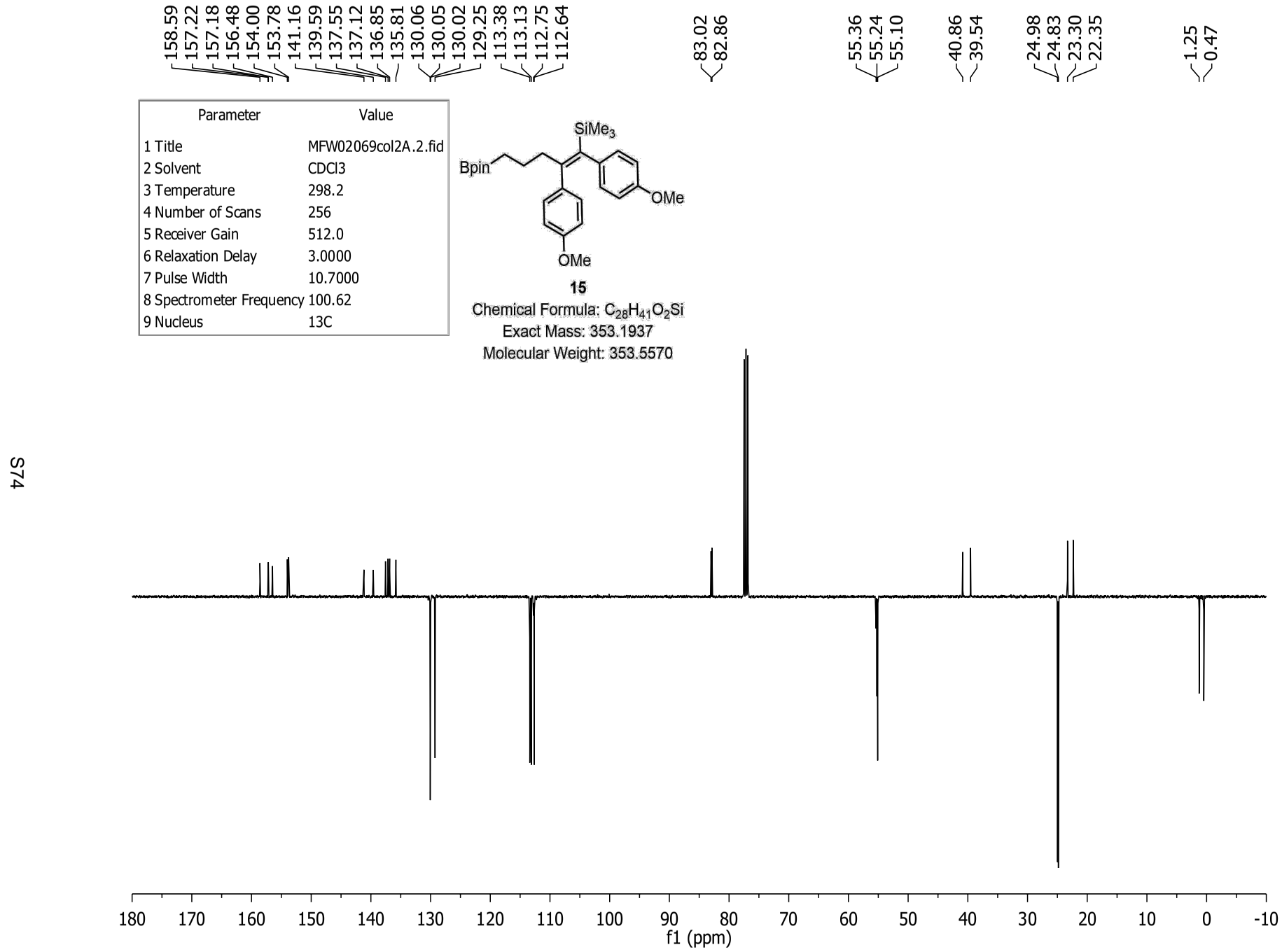
Parameter	Value
1 Title	MFW02069col2A.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	9.0
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H



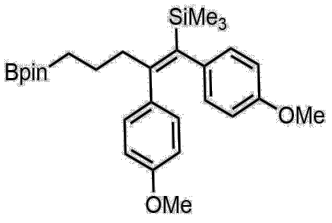
15

Chemical Formula: $C_{28}H_{41}O_2Si$
Exact Mass: 353.1937
Molecular Weight: 353.5570




158.59
157.22
157.18
156.48
154.00
153.78
141.16
139.59
137.55
137.12
136.85
135.81
130.06
130.05
130.02
129.25
113.38
113.13
112.75
112.64
83.02
82.86
55.36
55.24
55.10
40.86
39.54
24.98
24.83
23.30
22.35
1.25
0.47
Parameter
Value
1 Title
MFW02069col2A.2.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe3
Bpin
OMe
OMe
15
Chemical Formula: C28H41O2Si
Exact Mass: 353.1937
Molecular Weight: 353.5570
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

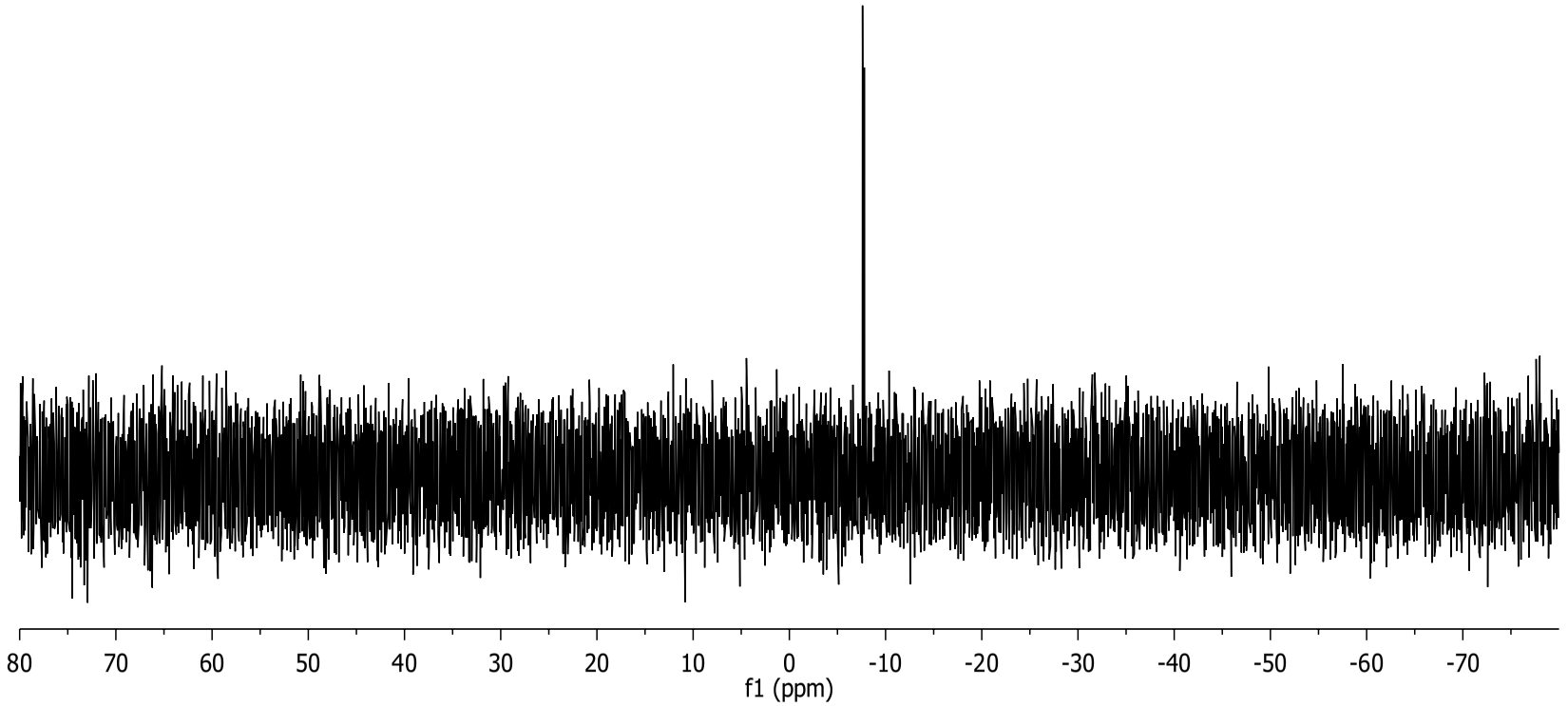
Parameter	Value
1 Title	MFW02069col2A.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	128
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



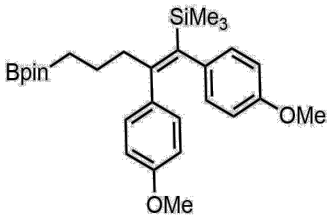
15

Chemical Formula: $C_{28}H_{41}O_2Si$
Exact Mass: 353.1937
Molecular Weight: 353.5570

-7.60
-7.79



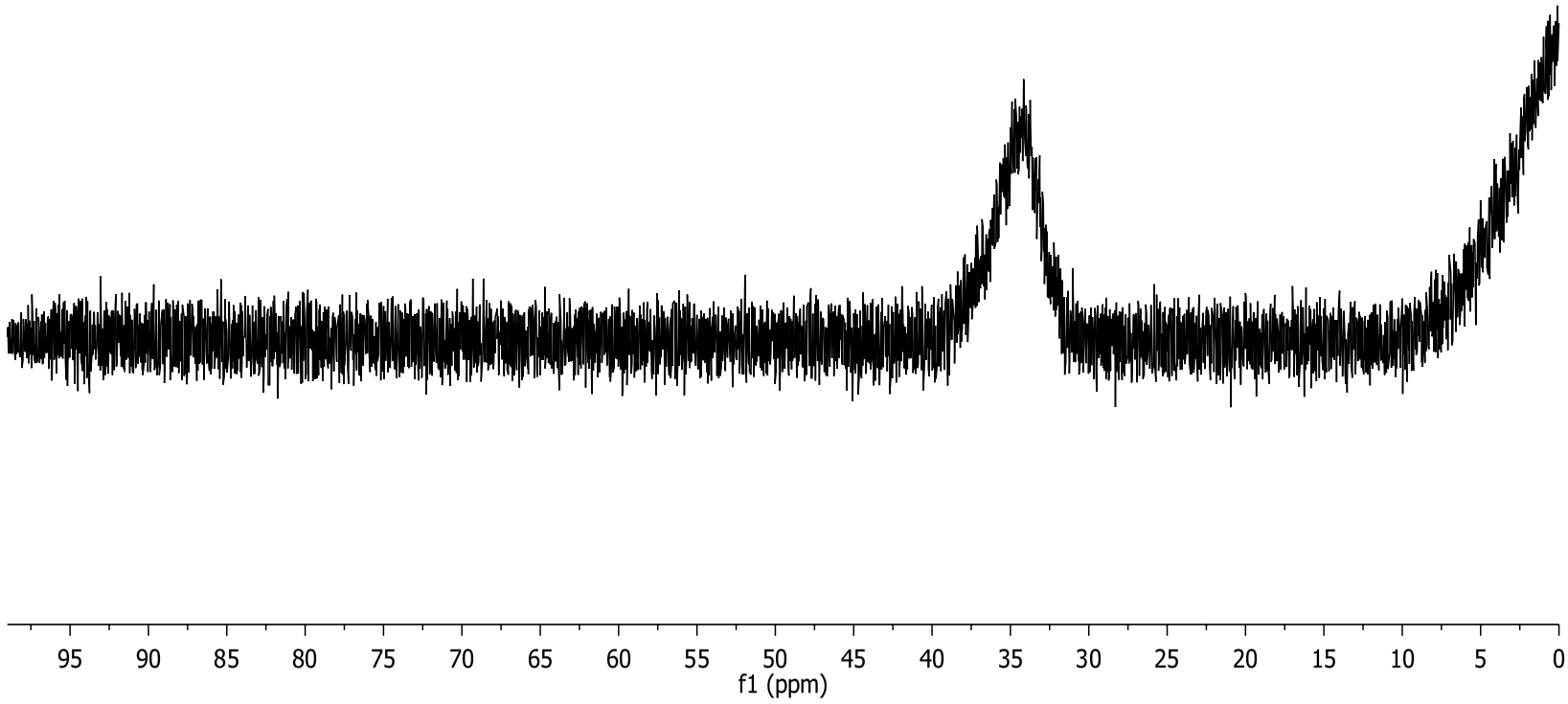
Parameter	Value
1 Title	MFW02069-11B-2.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	128
5 Receiver Gain	203.0
6 Relaxation Delay	1.0000
7 Pulse Width	8.3000
8 Spectrometer Frequency	128.38
9 Nucleus	11B

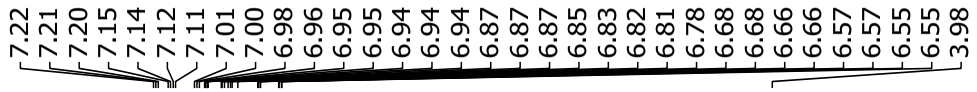


15

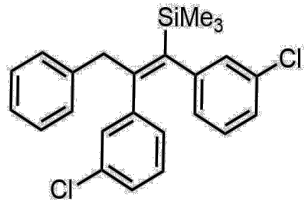
Chemical Formula: $C_{28}H_{41}O_2Si$
Exact Mass: 353.1937
Molecular Weight: 353.5570

34.10





Parameter	Value
1 Title	SBK03283B-hv.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	161.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

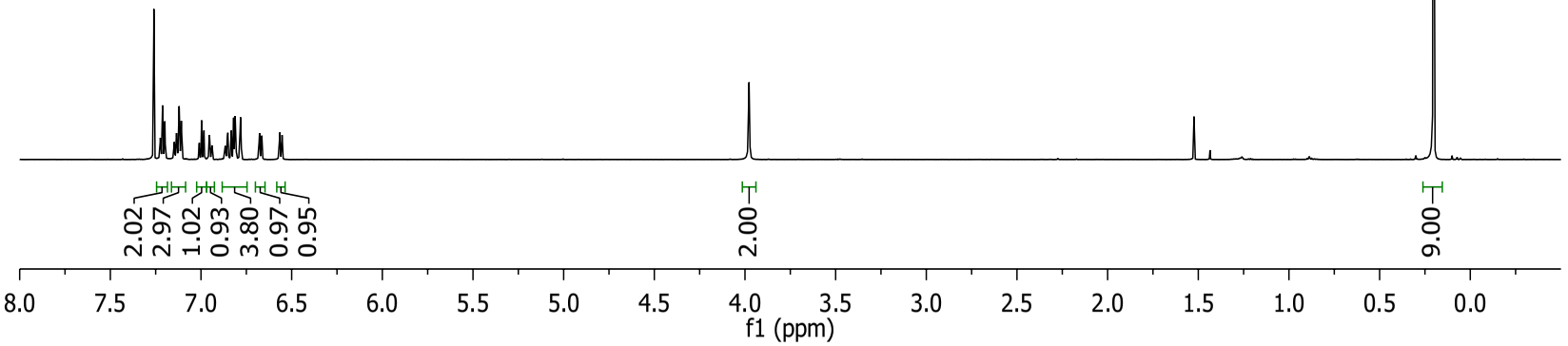


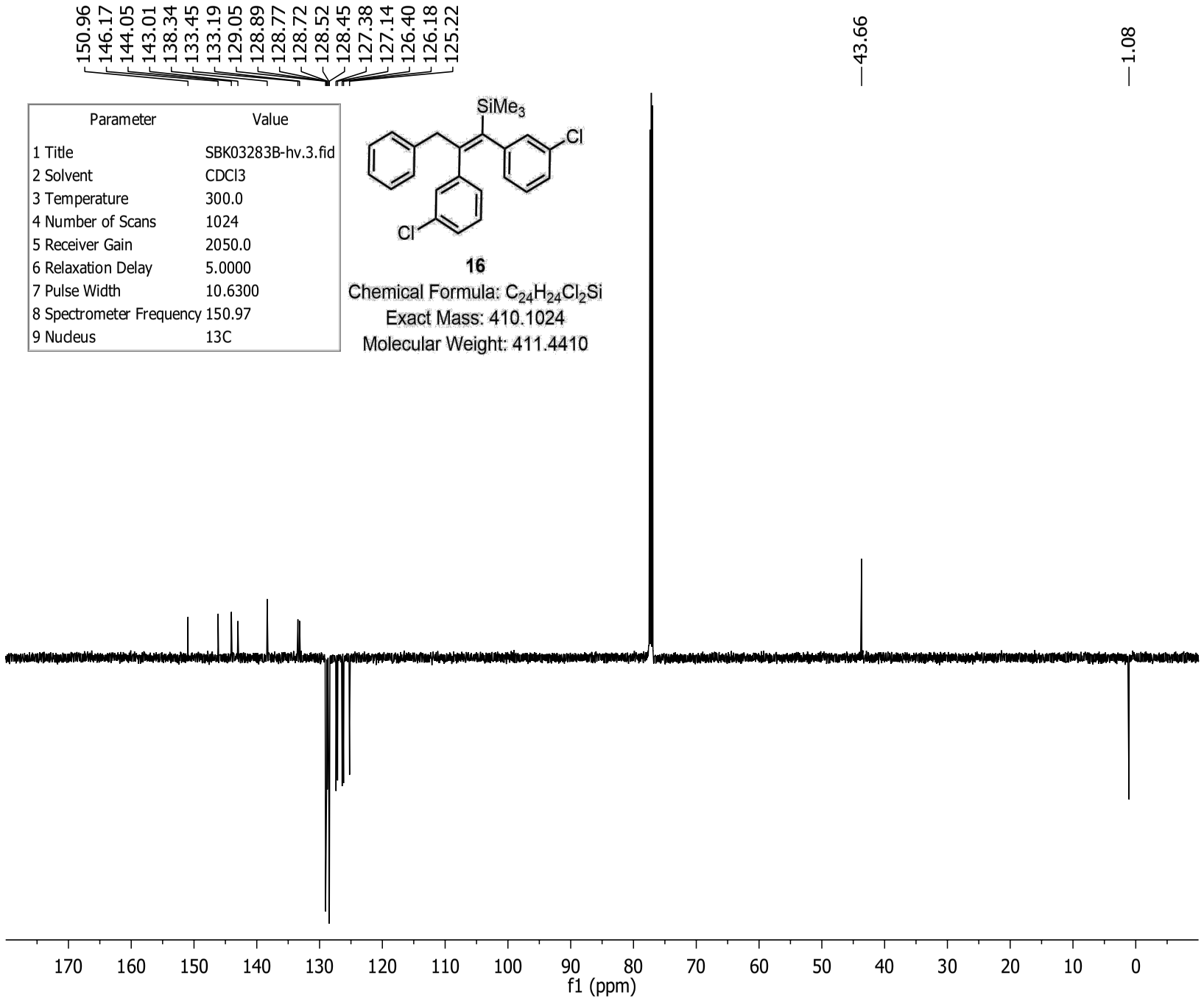
16

Chemical Formula: $C_{24}H_{24}Cl_2Si$

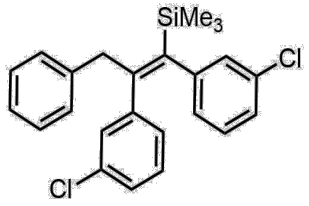
Exact Mass: 410.1024

Molecular Weight: 411.4410




150.96
146.17
144.05
143.01
138.34
133.45
133.19
129.05
128.89
128.77
128.72
128.52
128.45
127.38
127.14
126.40
126.18
125.22
43.66
1.08
Parameter Value
1 Title SBK03283B-hv.3.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe3
Cl
Cl
16
Chemical Formula: C24H24Cl2Si
Exact Mass: 410.1024
Molecular Weight: 411.4410
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Parameter	Value
1 Title	SBK03283B-hv.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



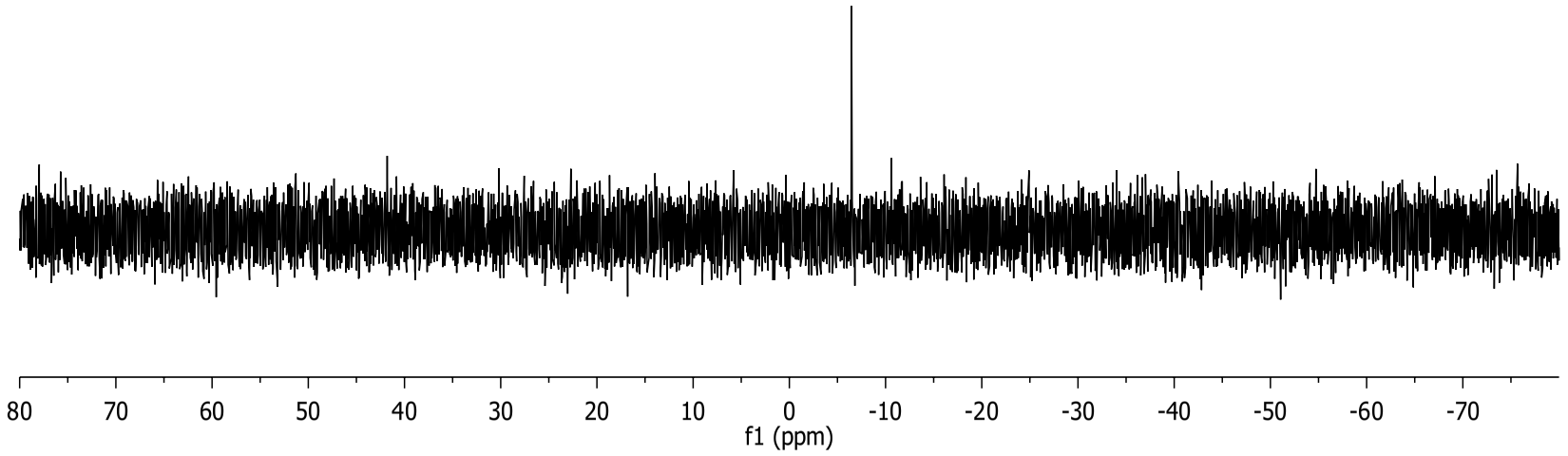
16

Chemical Formula: $C_{24}H_{24}Cl_2Si$

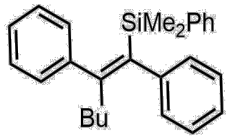
Exact Mass: 410.1024

Molecular Weight: 411.4410

-6.46

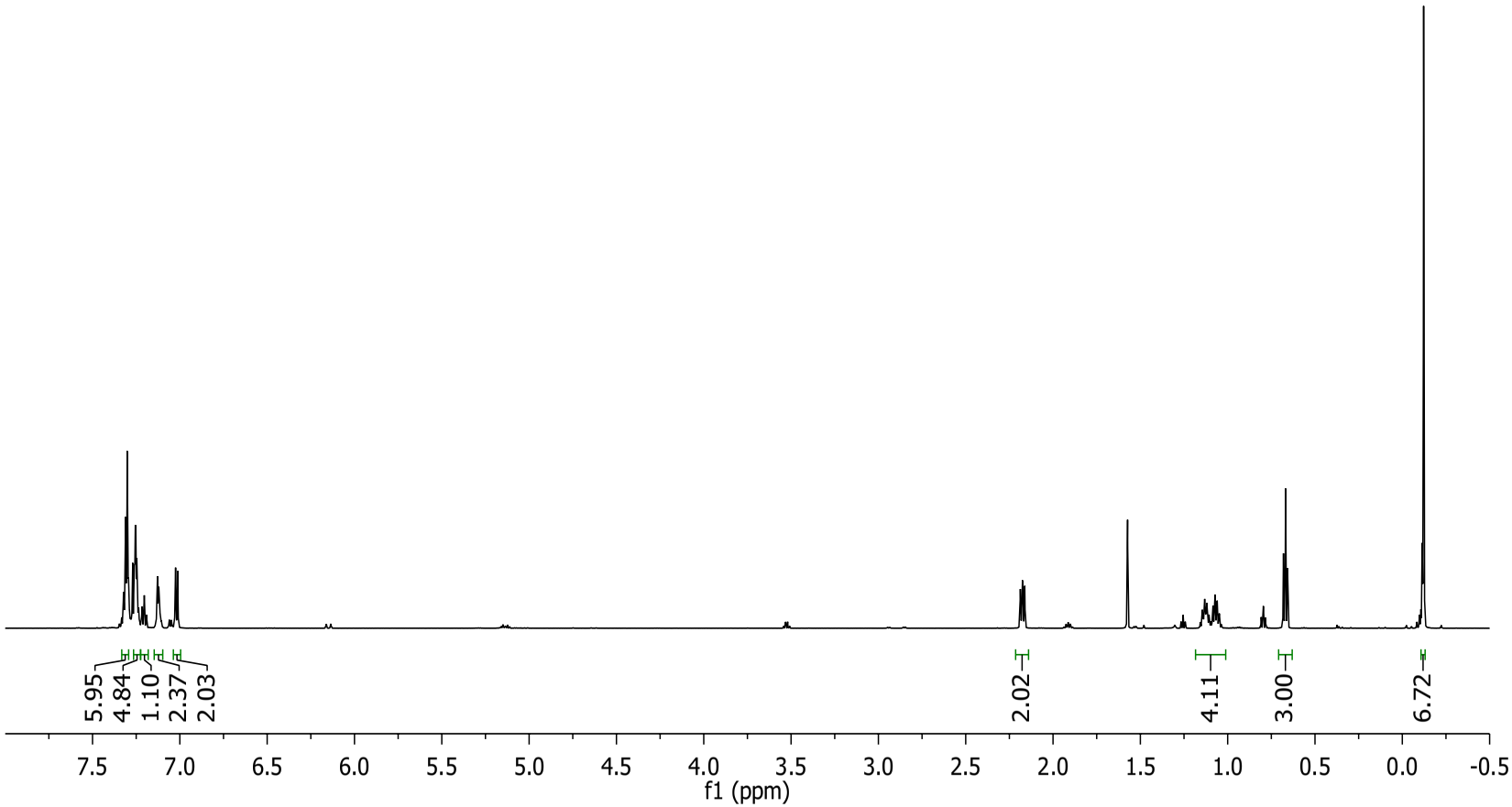


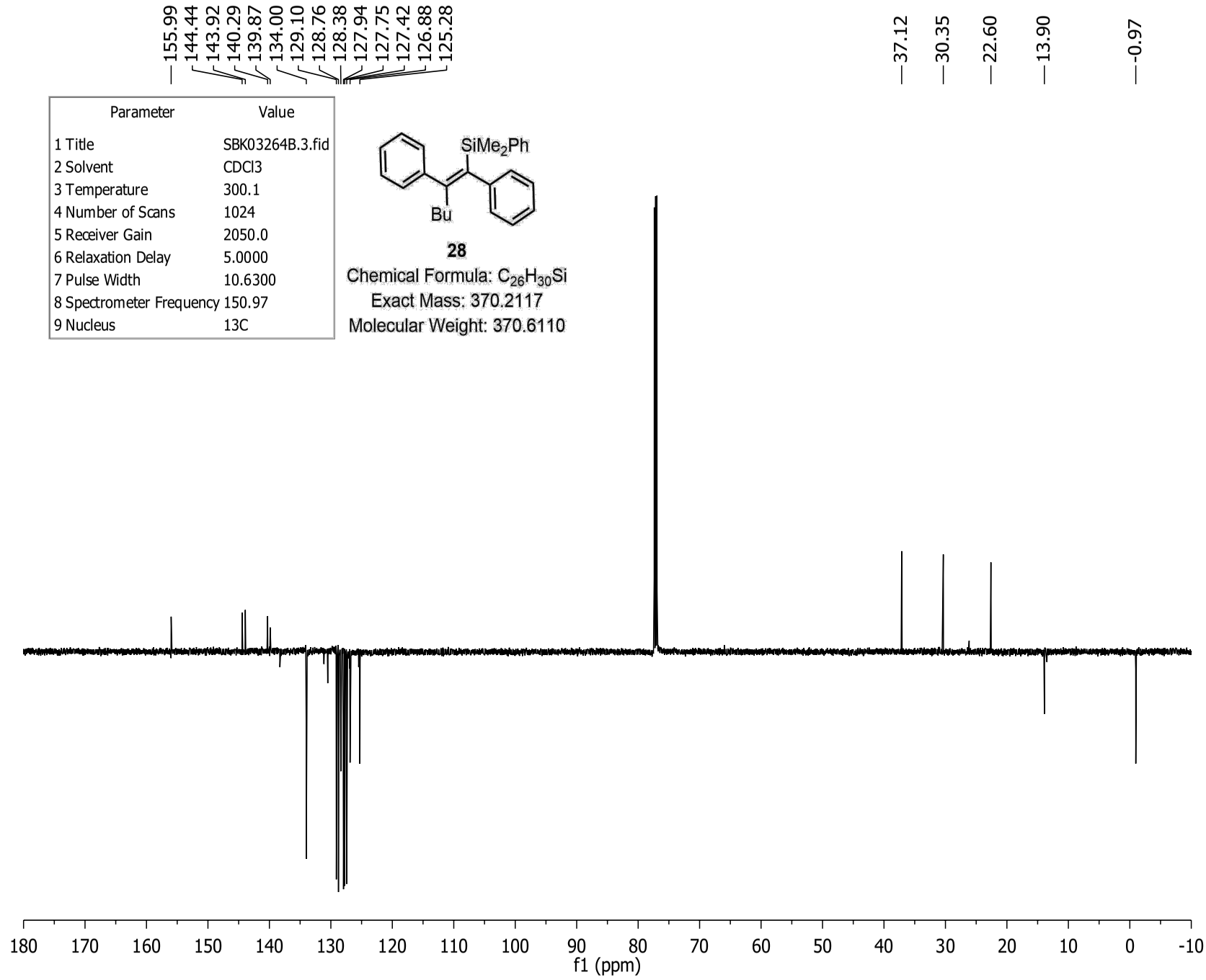
Parameter	Value
1 Title	SBK03264B.1.fid
2 Solvent	CDCl3
3 Temperature	298.8
4 Number of Scans	16
5 Receiver Gain	128.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H



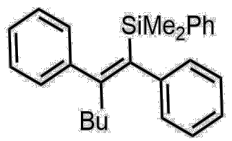
28

Chemical Formula: $C_{26}H_{30}Si$
Exact Mass: 370.2117
Molecular Weight: 370.6110




155.99
144.44
143.92
140.29
139.87
134.00
129.10
128.76
128.38
127.94
127.75
127.42
126.88
125.28
37.12
30.35
22.60
13.90
-0.97
Parameter Value
1 Title SBK03264B.3.fid
2 Solvent CDCl3
3 Temperature 300.1
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Ph
Bu
28
Chemical Formula: C26H30Si
Exact Mass: 370.2117
Molecular Weight: 370.6110
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Parameter	Value
1 Title	SBK03264B.2.fid
2 Solvent	CDCl3
3 Temperature	297.4
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



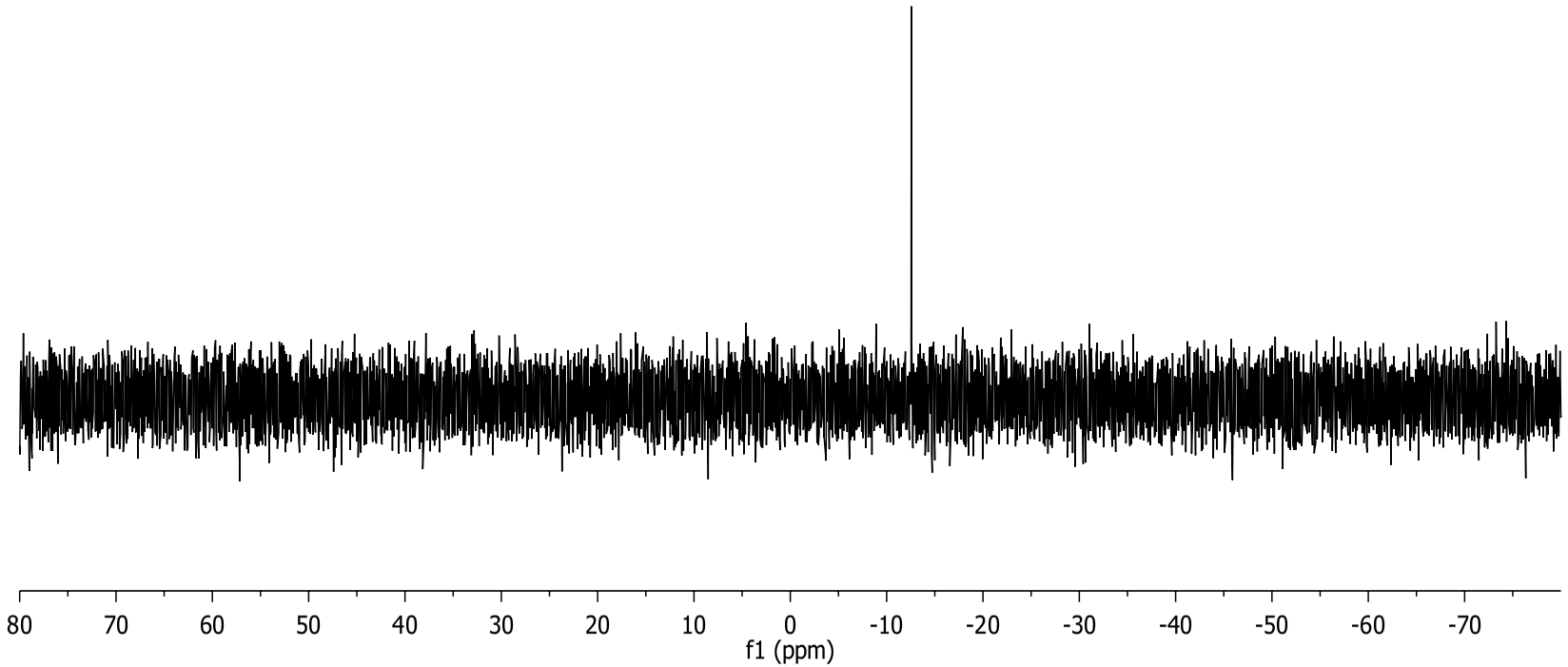
28

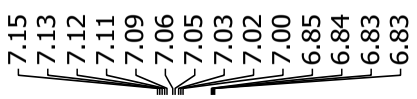
Chemical Formula: $C_{26}H_{30}Si$

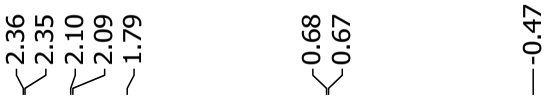
Exact Mass: 370.2117

Molecular Weight: 370.6110

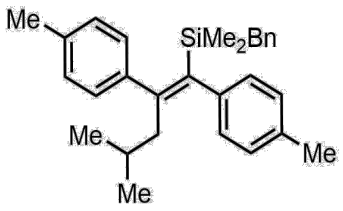
-12.57







Parameter	Value
1 Title	SBK04015B.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	161.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

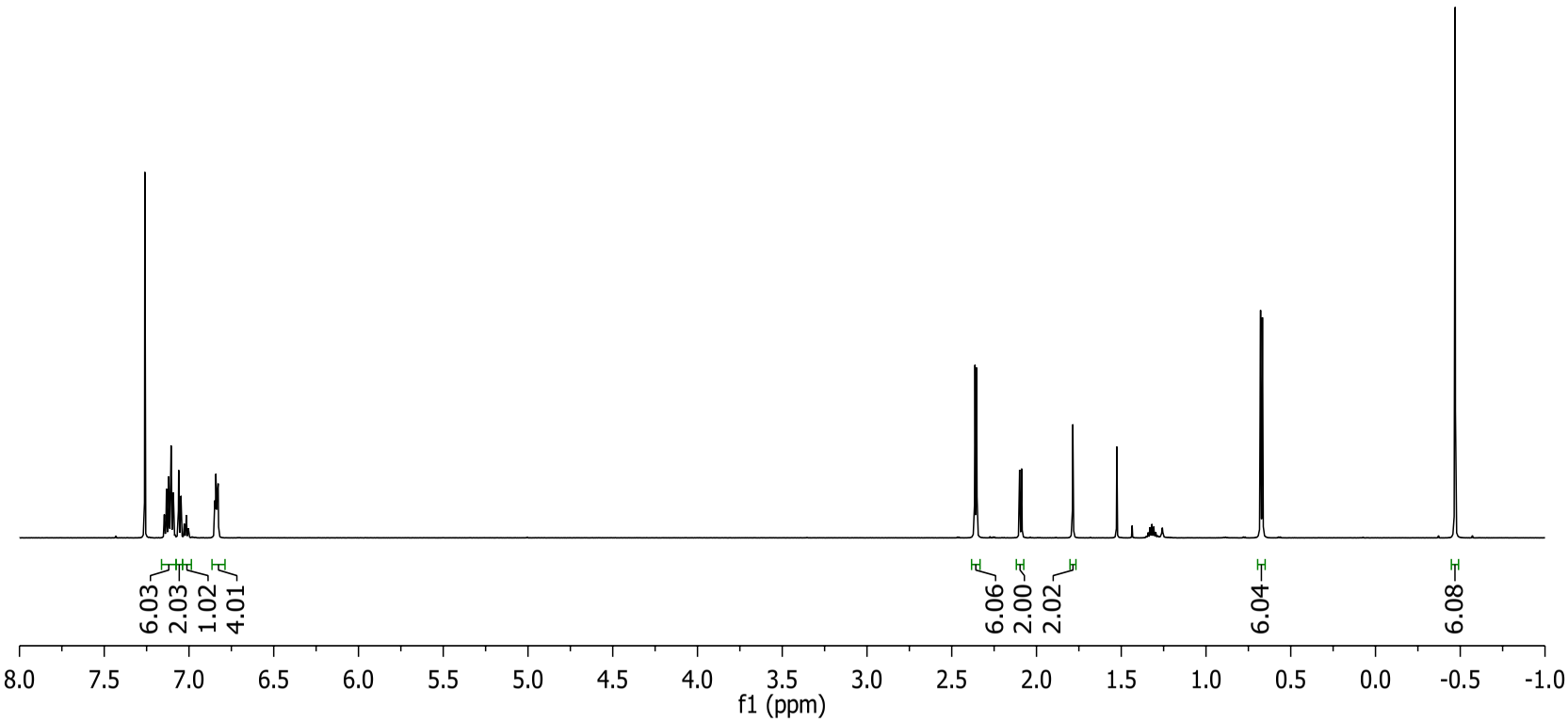


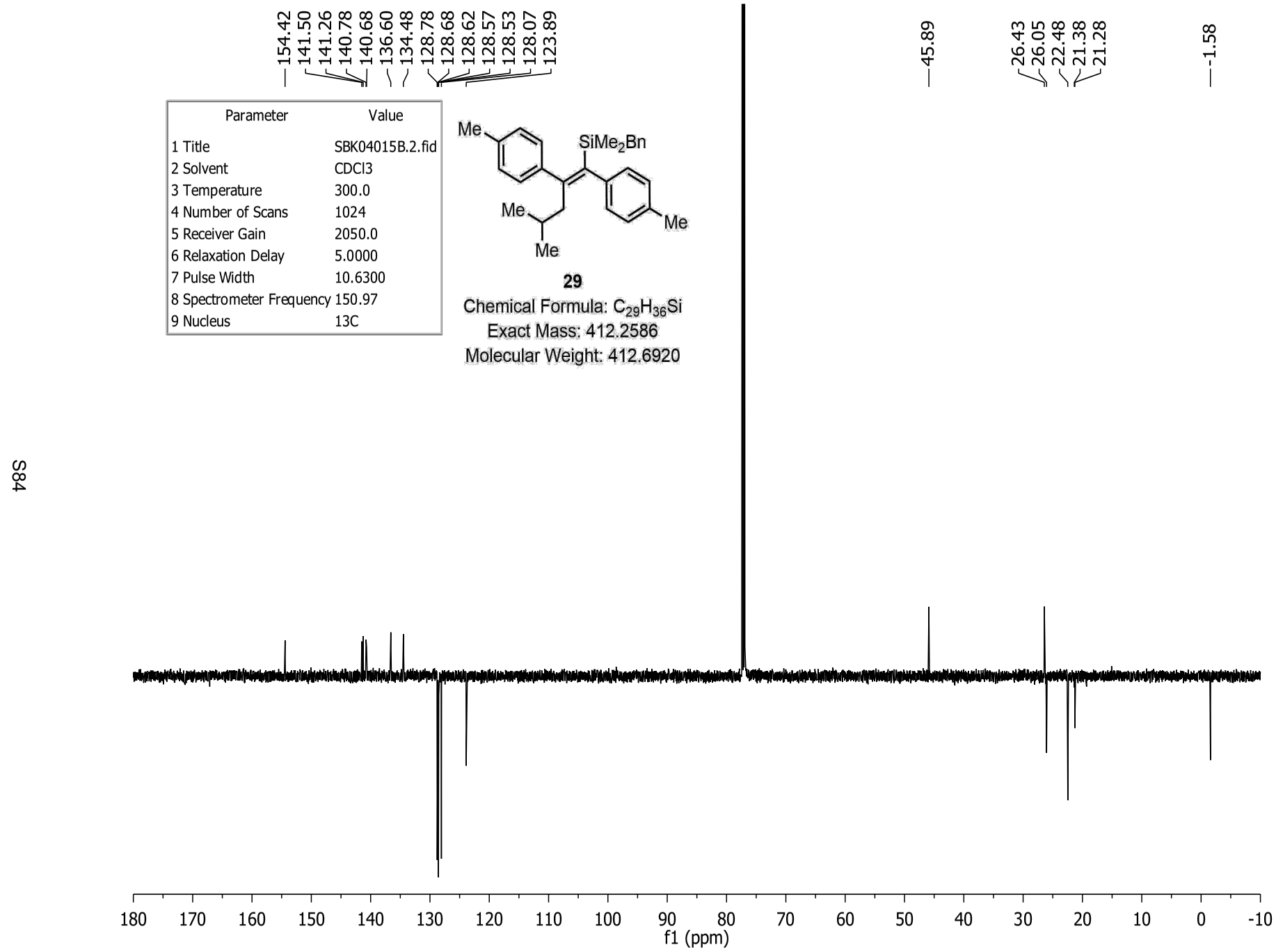
29

Chemical Formula: $C_{29}H_{36}Si$

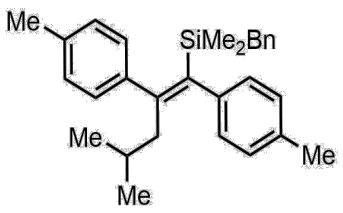
Exact Mass: 412.2586

Molecular Weight: 412.6920




154.42
141.50
141.26
140.78
140.68
136.60
134.48
128.78
128.68
128.62
128.57
128.53
128.07
123.89
45.89
26.43
26.05
22.48
21.38
21.28
-1.58
Parameter Value
1 Title SBK04015B.2.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
Me
SiMe2Bn
Me
Me
Me
29
Chemical Formula: C29H36Si
Exact Mass: 412.2586
Molecular Weight: 412.6920
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

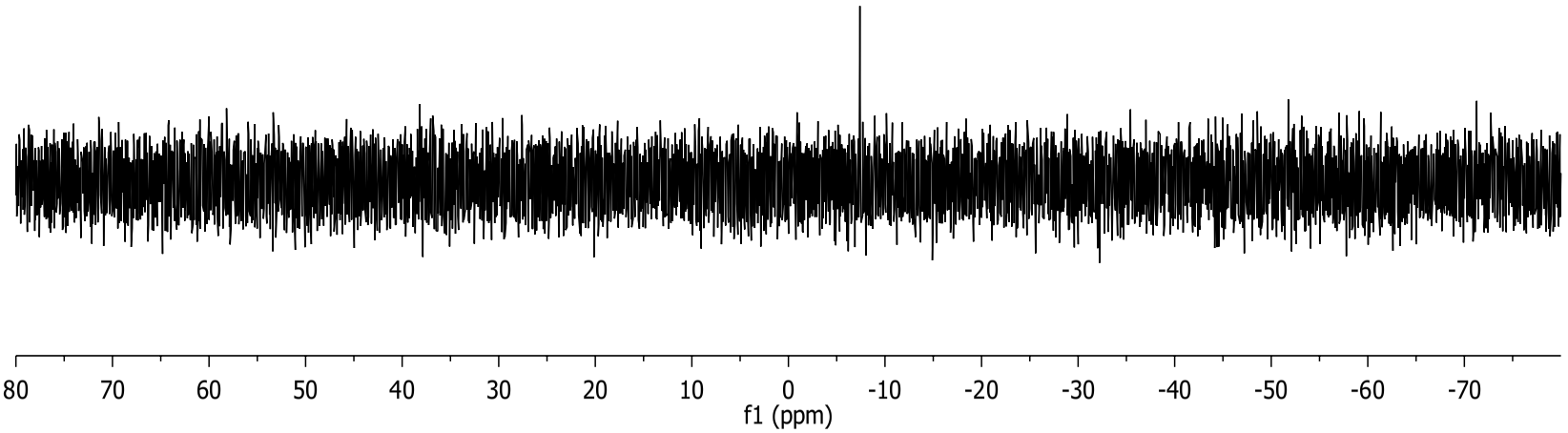
Parameter	Value
1 Title	SBK04015B.3.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

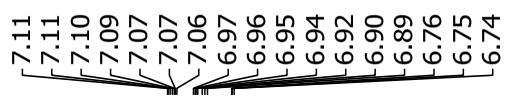


29

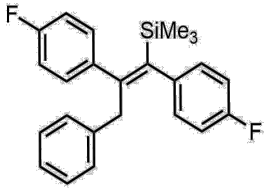
Chemical Formula: $C_{29}H_{36}Si$
Exact Mass: 412.2586
Molecular Weight: 412.6920

-7.40



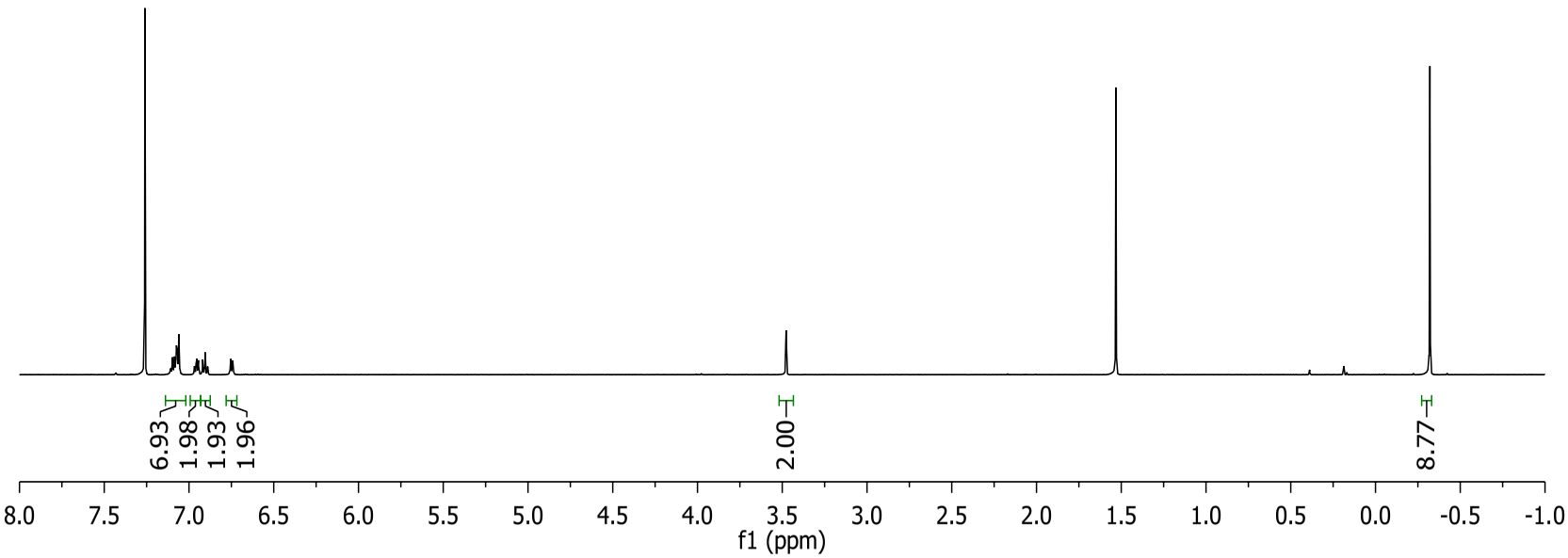


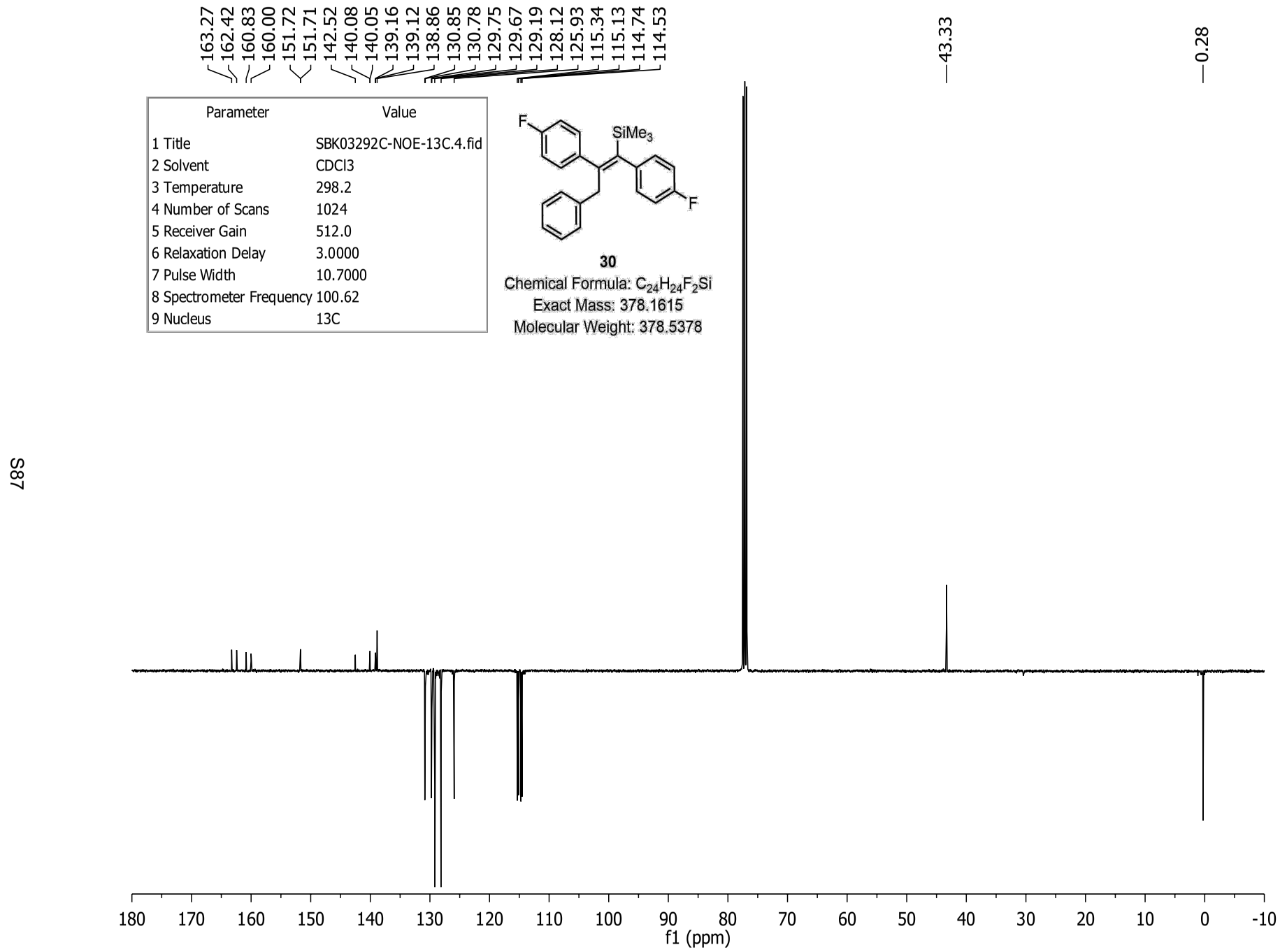
Parameter	Value
1 Title	SBK03292C-2.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	181.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H



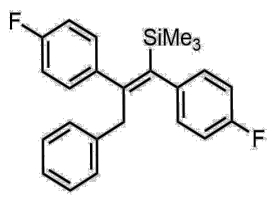
30

Chemical Formula: $C_{24}H_{24}F_2Si$
Exact Mass: 378.1615
Molecular Weight: 378.5378




163.27
162.42
160.83
160.00
151.72
151.71
142.52
140.08
140.05
139.16
139.12
138.86
130.85
130.78
129.75
129.67
129.19
128.12
125.93
115.34
115.13
114.74
114.53
43.33
0.28
Parameter
Value
1 Title
SBK03292C-NOE-13C.4.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
1024
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
F
SiMe3
F
30
Chemical Formula: C24H24F2Si
Exact Mass: 378.1615
Molecular Weight: 378.5378
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

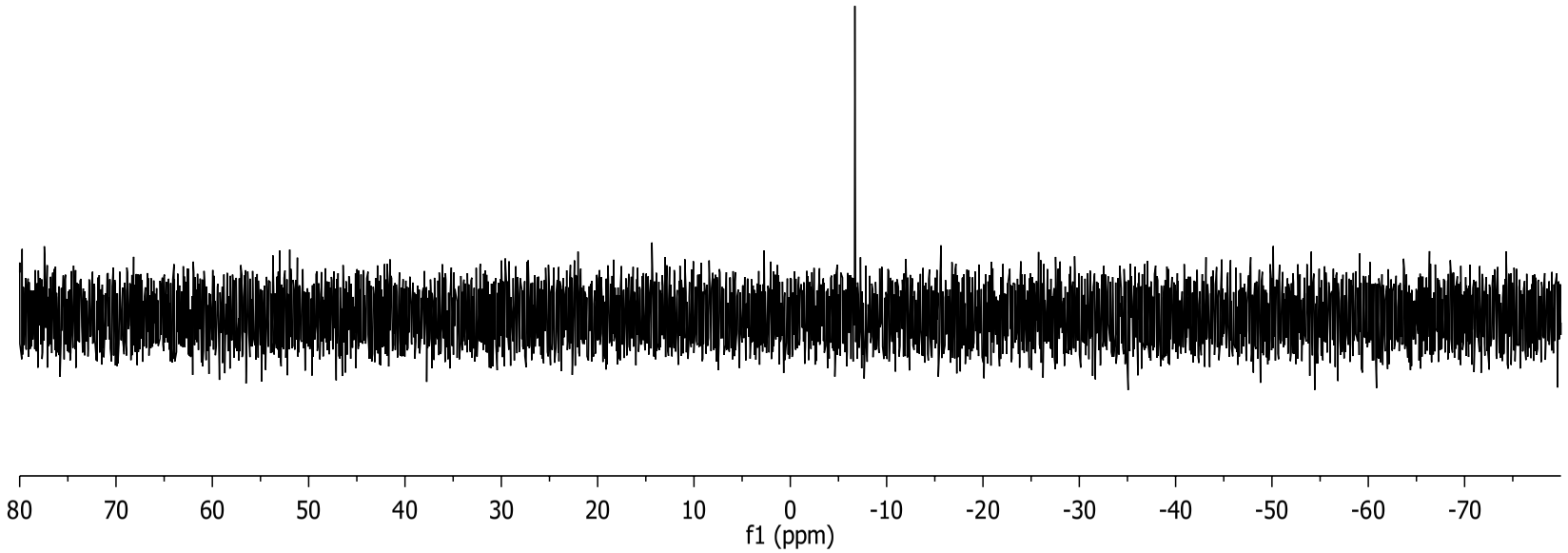
Parameter	Value
1 Title	SBK03292C-Si.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



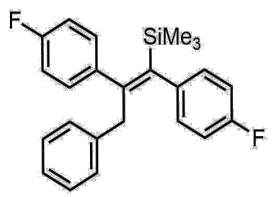
30

Chemical Formula: $C_{24}H_{24}F_2Si$
Exact Mass: 378.1615
Molecular Weight: 378.5378

-6.70



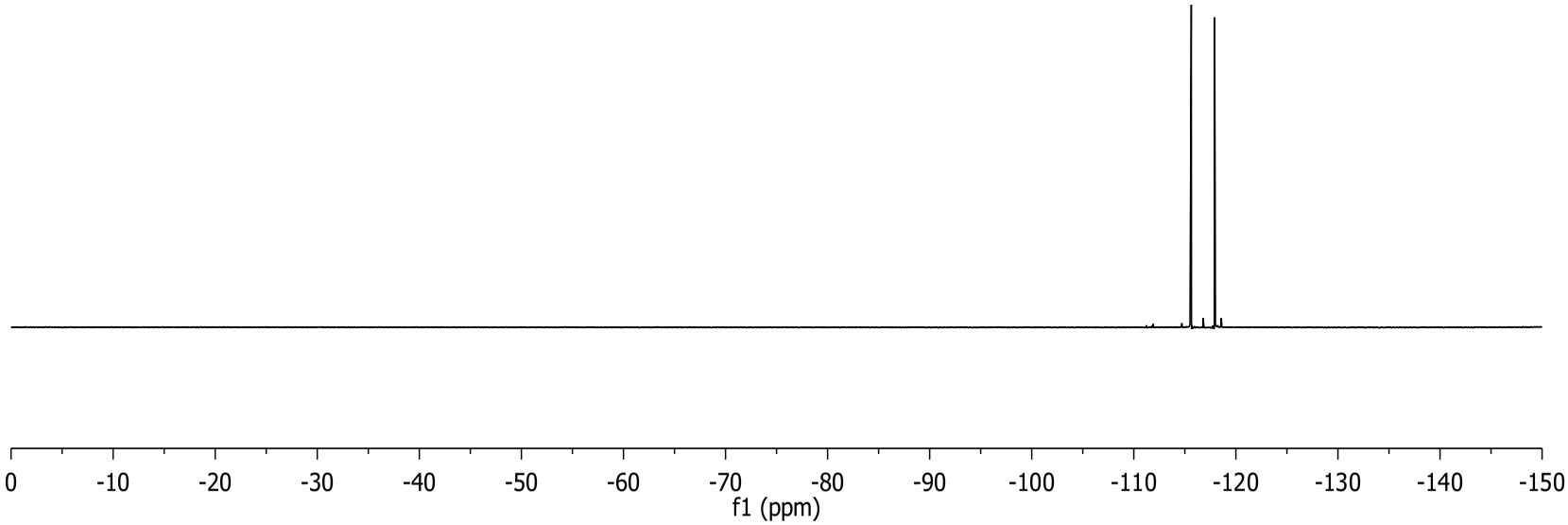
Parameter	Value
1 Title	SBK03292C-19F.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	362.0
6 Relaxation Delay	3.0000
7 Pulse Width	11.4000
8 Spectrometer Frequency	564.81
9 Nucleus	19F

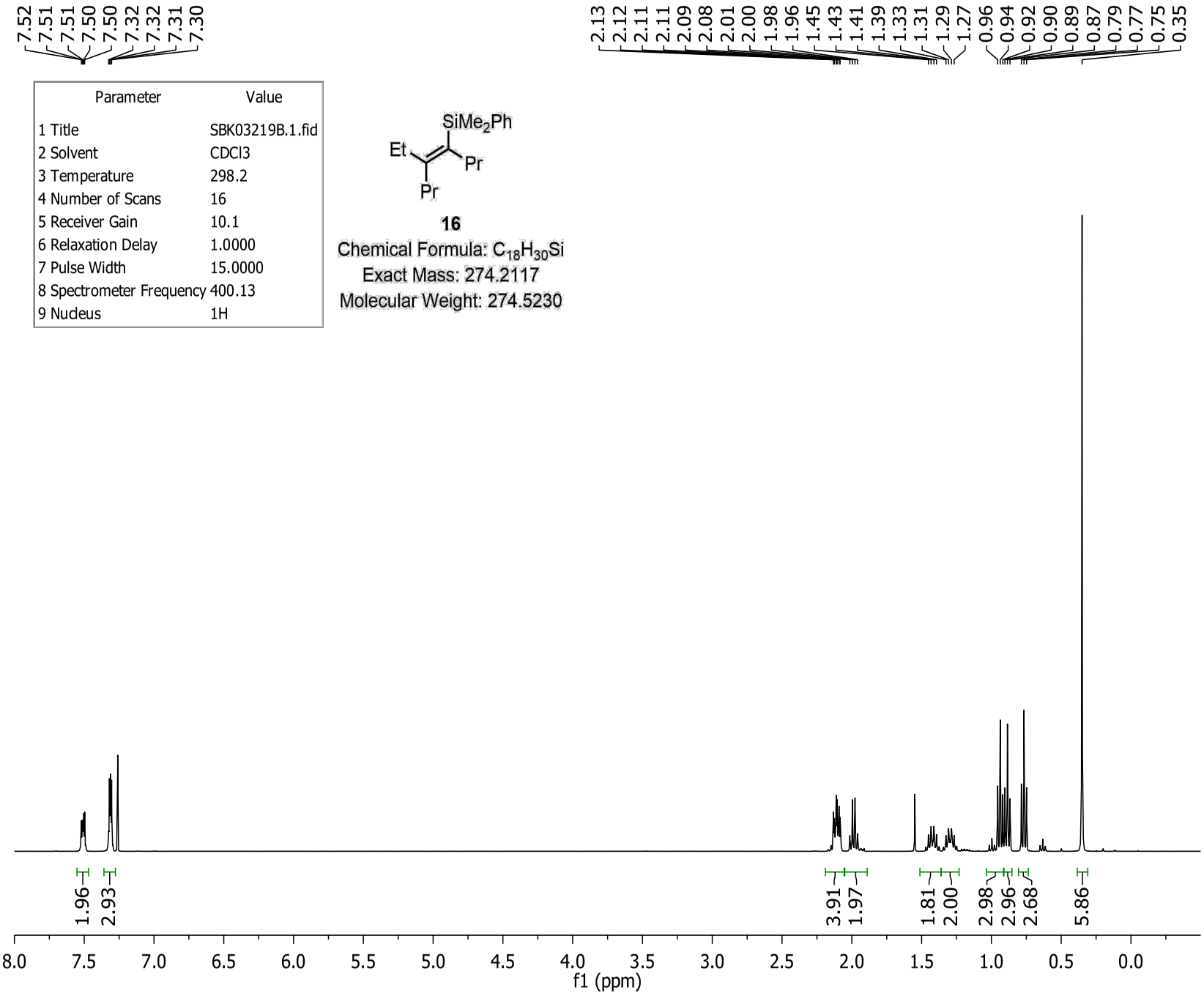


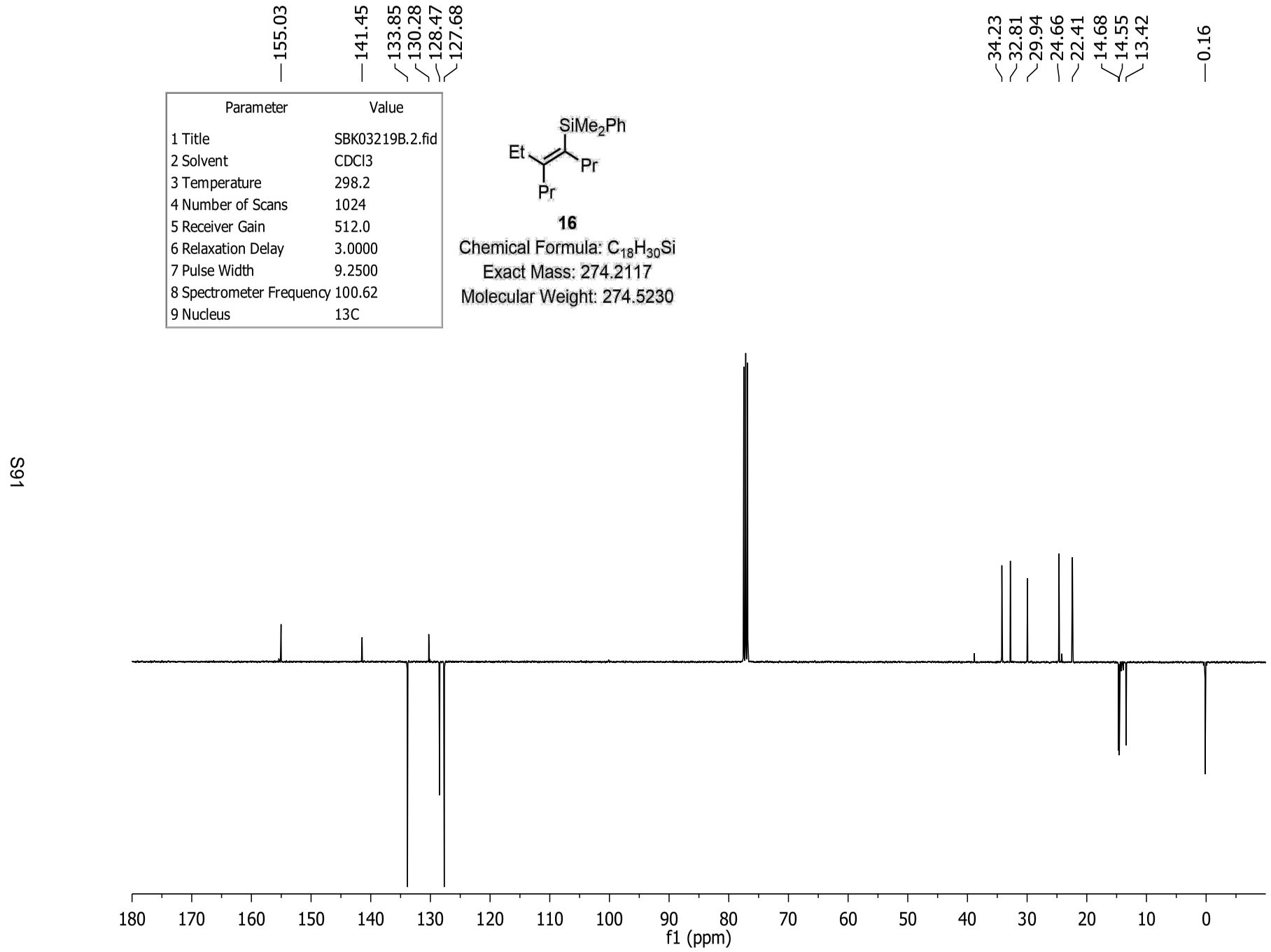
30

Chemical Formula: $C_{24}H_{24}F_2Si$
Exact Mass: 378.1615
Molecular Weight: 378.5378

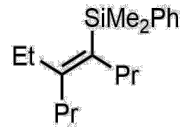
-115.63
-117.92




7.52
7.51
7.51
7.50
7.50
7.32
7.32
7.31
7.30
2.13
2.12
2.11
2.11
2.09
2.08
2.01
2.00
1.98
1.96
1.45
1.43
1.41
1.39
1.33
1.31
1.29
1.27
0.96
0.94
0.92
0.90
0.89
0.87
0.79
0.77
0.75
0.35
Parameter
Value
1 Title
SBK03219B.1.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
16
5 Receiver Gain
10.1
6 Relaxation Delay
1.0000
7 Pulse Width
15.0000
8 Spectrometer Frequency
400.13
9 Nucleus
1H
SiMe2Ph
Et
Pr
Pr
16
Chemical Formula: C18H30Si
Exact Mass: 274.2117
Molecular Weight: 274.5230
1.96
2.93
3.91
1.97
1.81
2.00
2.98
2.96
2.68
5.86
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


155.03
141.45
133.85
130.28
128.47
127.68
34.23
32.81
29.94
24.66
22.41
14.68
14.55
13.42
0.16
Parameter Value
1 Title SBK03219B.2.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 9.2500
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Et
Pr
Pr
16
Chemical Formula: C18H30Si
Exact Mass: 274.2117
Molecular Weight: 274.5230
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

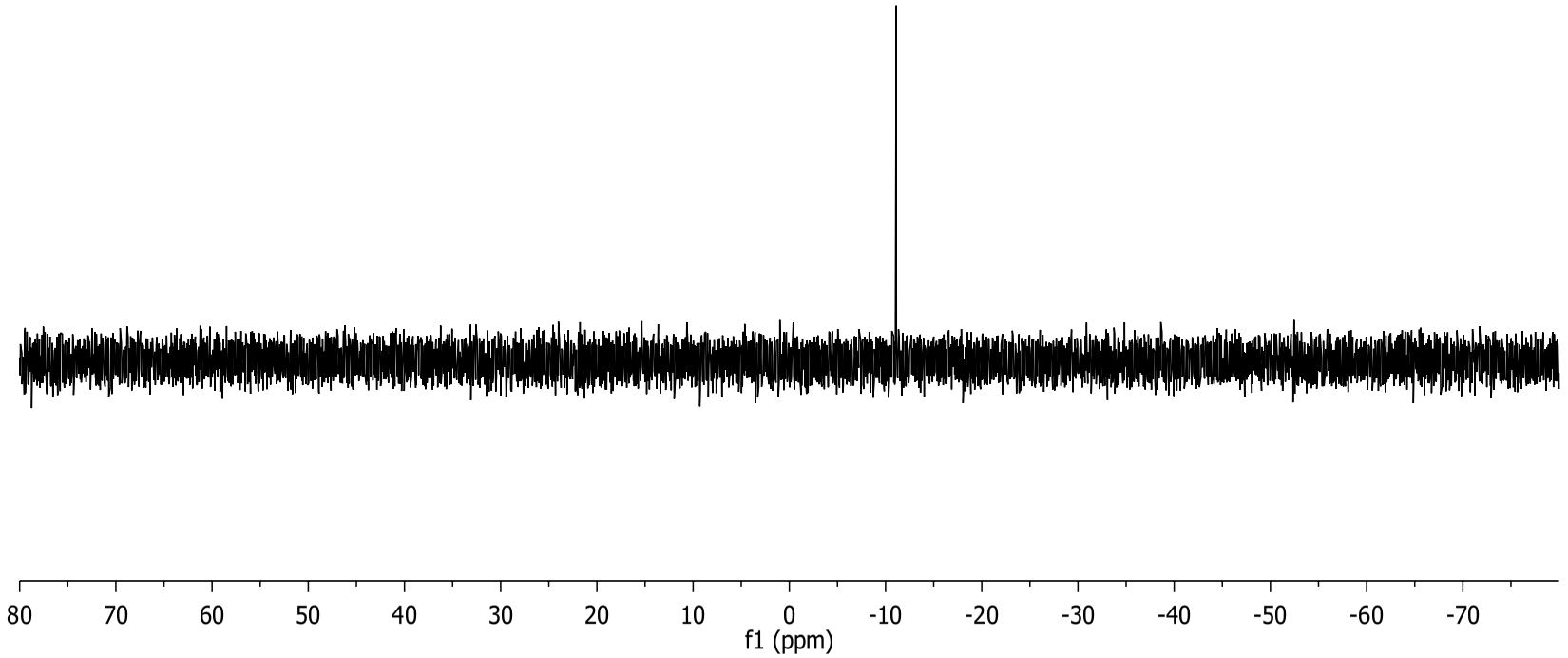
Parameter	Value
1 Title	SBK03219B-29Si.1.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

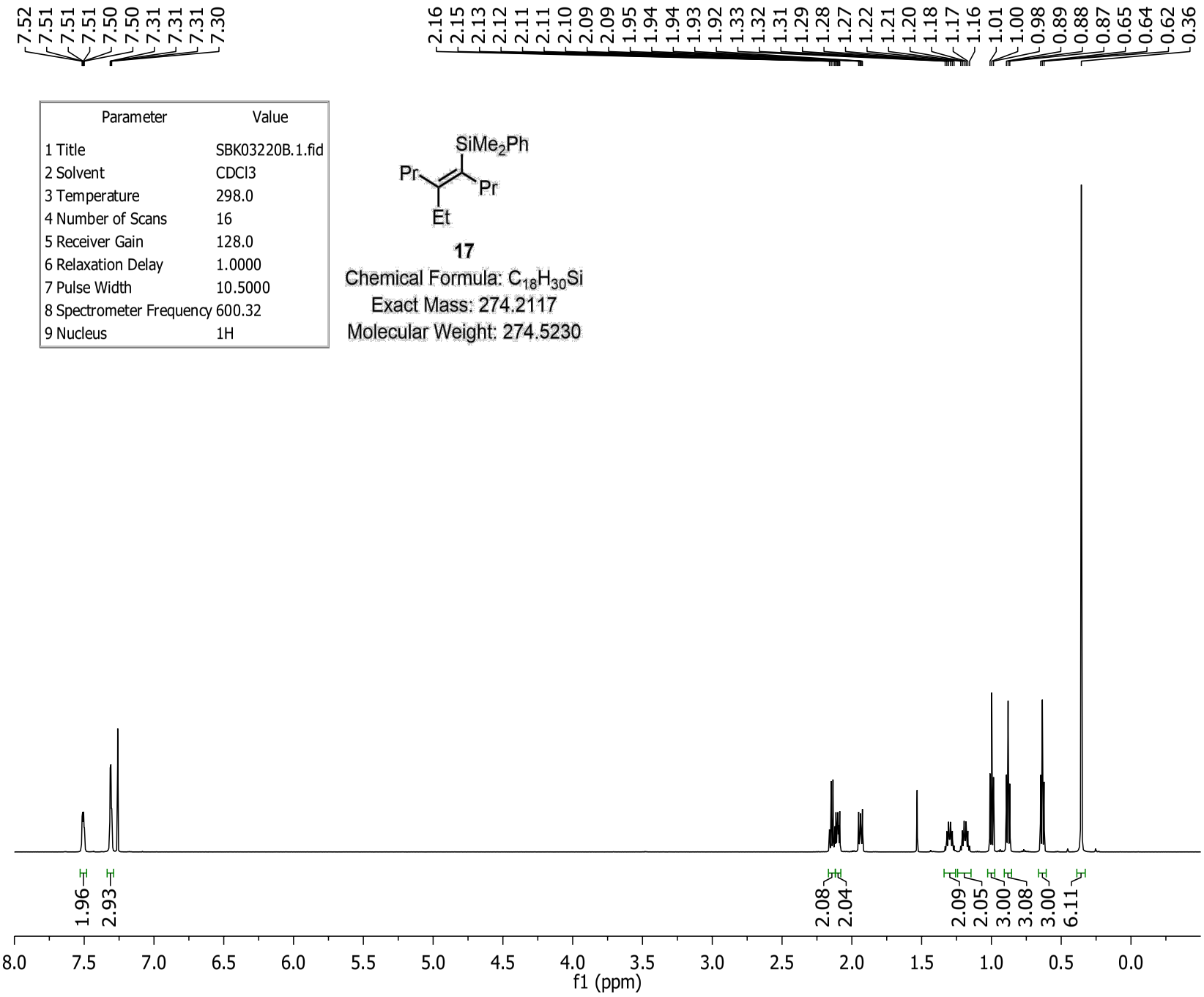


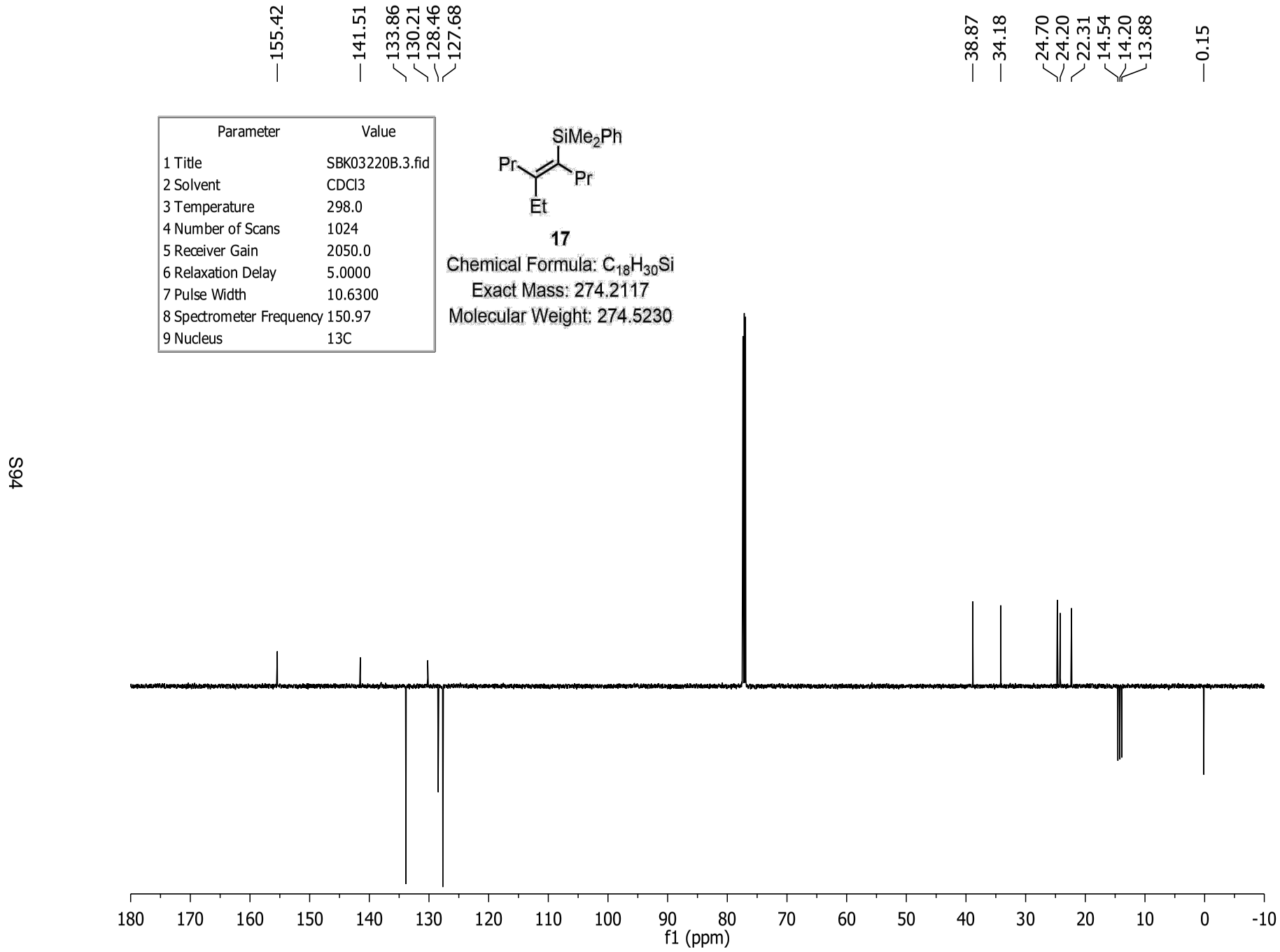
16

Chemical Formula: $C_{18}H_{30}Si$
Exact Mass: 274.2117
Molecular Weight: 274.5230

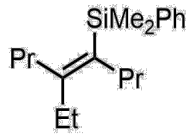
-11.11




7.52 7.51 7.51 7.51 7.50 7.50 7.31 7.31 7.31 7.30
2.16 2.15 2.13 2.12 2.11 2.11 2.10 2.09 2.09 1.95 1.94 1.94 1.93 1.92 1.33 1.32 1.31 1.29 1.28 1.27 1.22 1.21 1.20 1.18 1.17 1.16 1.01 1.00 0.98 0.89 0.88 0.87 0.65 0.64 0.62 0.36
Parameter Value
1 Title SBK03220B.1.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 128.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
SiMe2Ph
Pr
Pr
Et
17
Chemical Formula: C18H30Si
Exact Mass: 274.2117
Molecular Weight: 274.5230
1.96 2.93 2.08 2.04 2.09 2.05 3.00 3.08 3.00 6.11
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


155.42
141.51
133.86
130.21
128.46
127.68
38.87
34.18
24.70
24.20
22.31
14.54
14.20
13.88
0.15
Parameter Value
1 Title SBK03220B.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Ph
Pr
Pr
Et
17
Chemical Formula: C18H30Si
Exact Mass: 274.2117
Molecular Weight: 274.5230
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

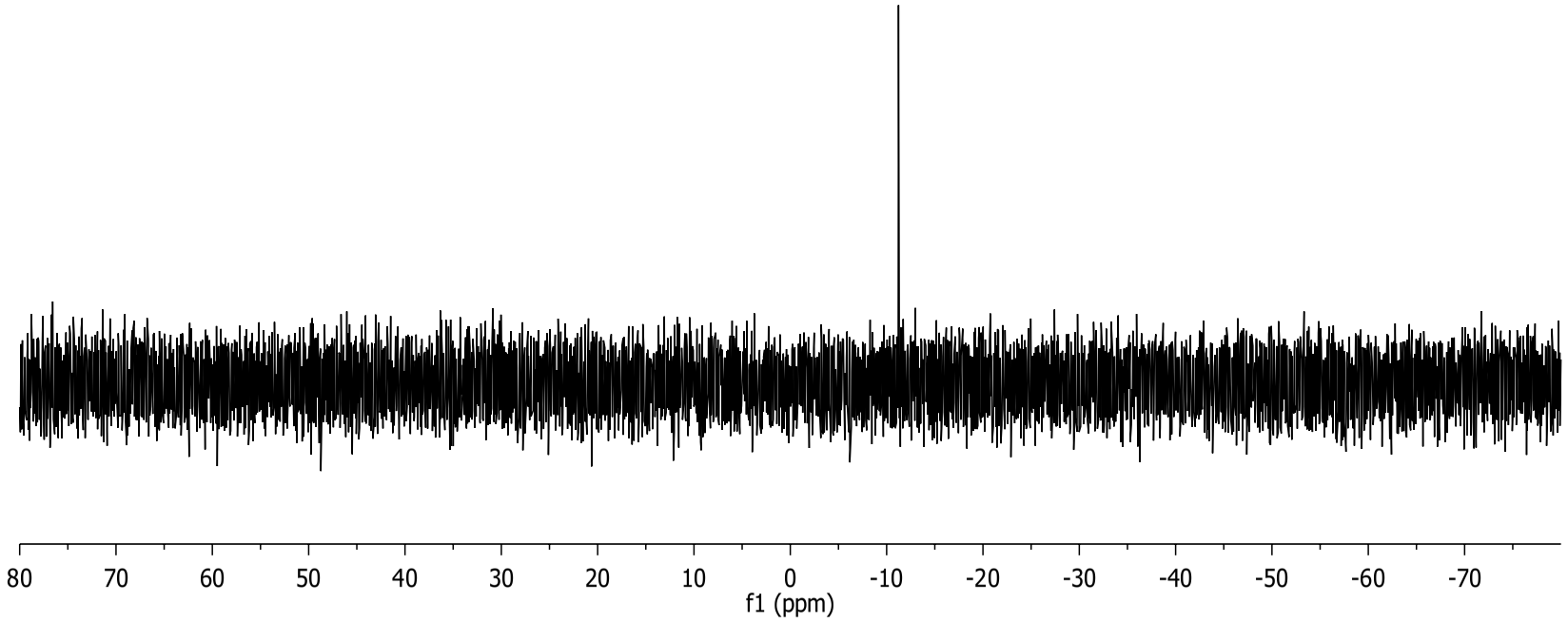
Parameter	Value
1 Title	SBK03220B.2.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

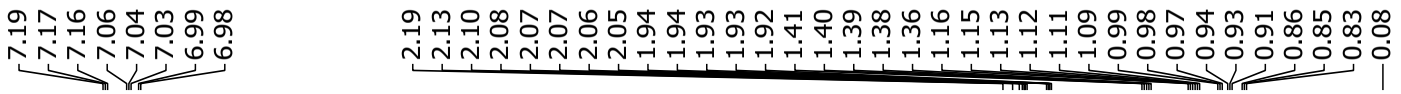


17

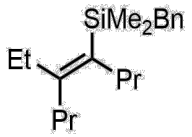
Chemical Formula: $C_{18}H_{30}Si$
Exact Mass: 274.2117
Molecular Weight: 274.5230

-11.23





Parameter	Value
1 Title	SBK03215B.1.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	16
5 Receiver Gain	128.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

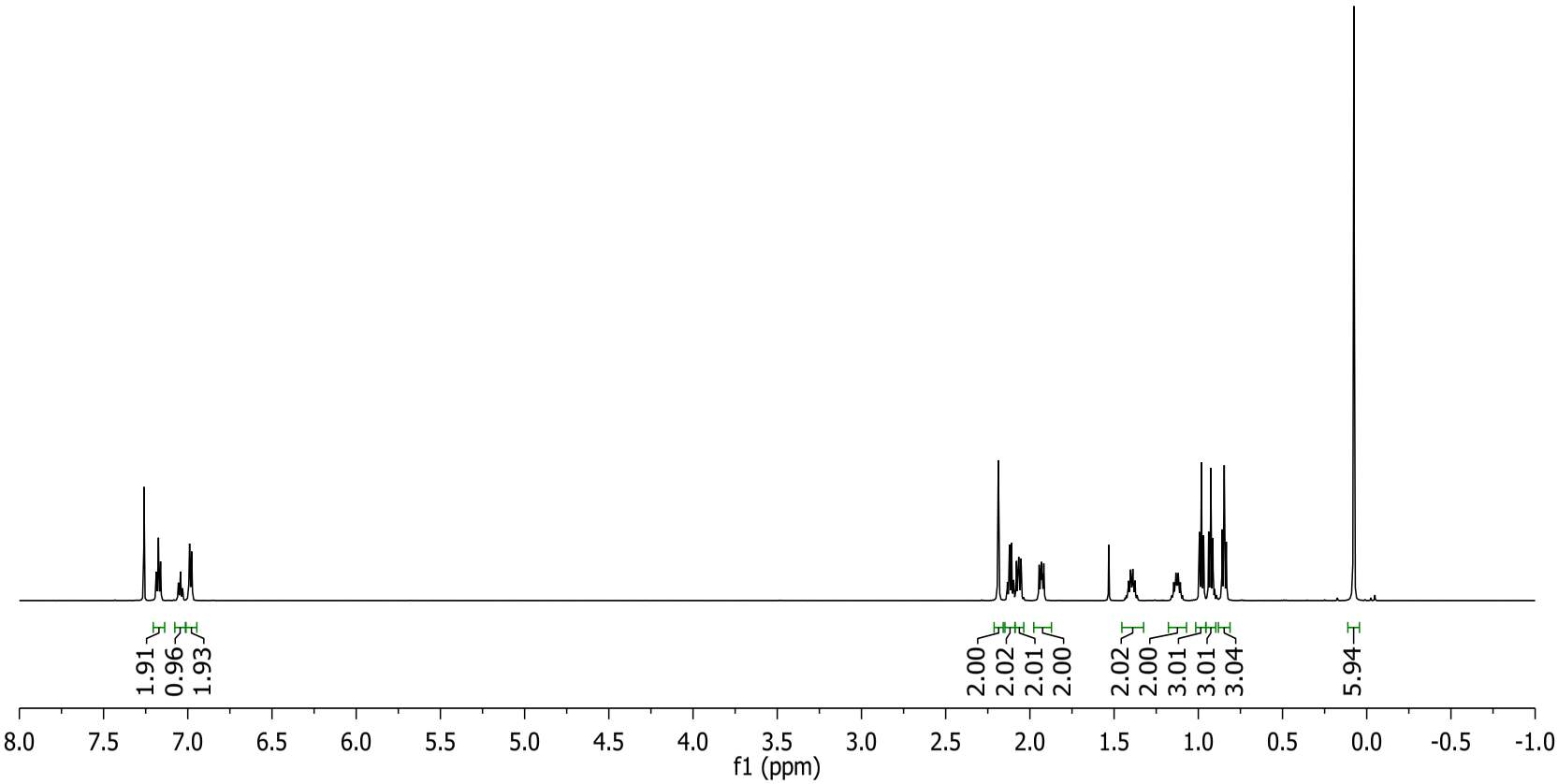


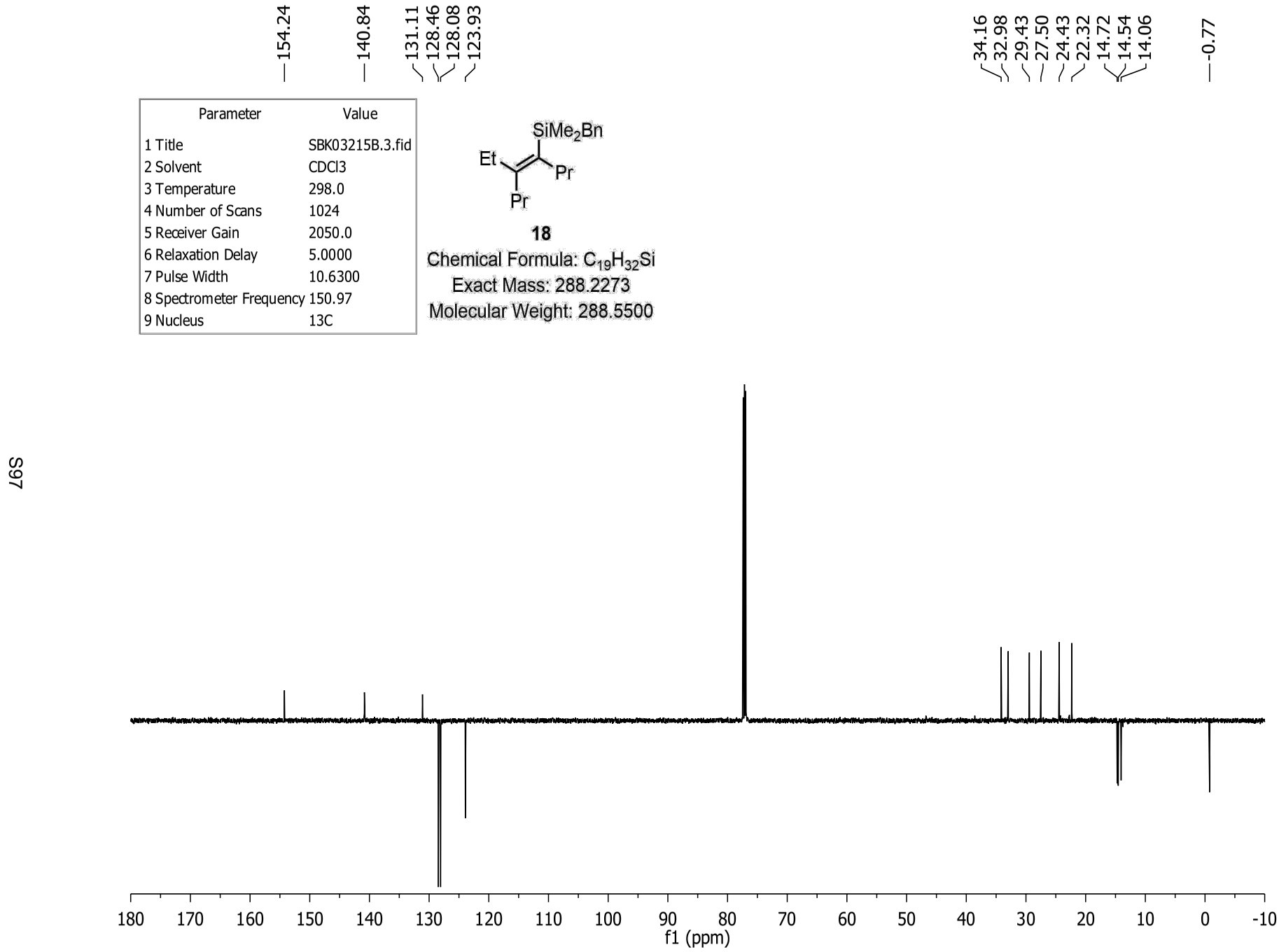
18

Chemical Formula: $C_{19}H_{32}Si$

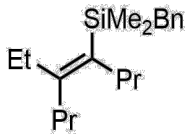
Exact Mass: 288.2273

Molecular Weight: 288.5500




154.24
140.84
131.11
128.46
128.08
123.93
34.16
32.98
29.43
27.50
24.43
22.32
14.72
14.54
14.06
-0.77
Parameter Value
1 Title SBK03215B.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe2Bn
Et
Pr
Pr
18
Chemical Formula: C19H32Si
Exact Mass: 288.2273
Molecular Weight: 288.5500
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

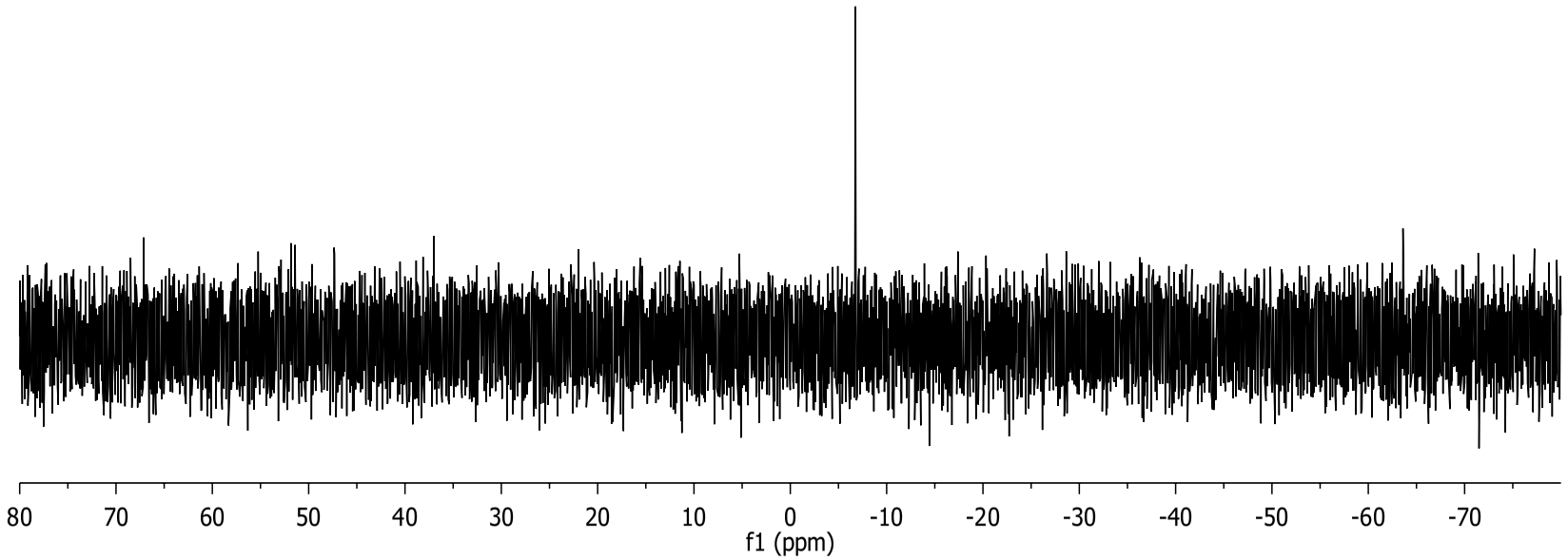
Parameter	Value
1 Title	SBK03215B.2.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



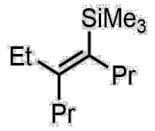
18

Chemical Formula: $C_{19}H_{32}Si$
Exact Mass: 288.2273
Molecular Weight: 288.5500

-6.76

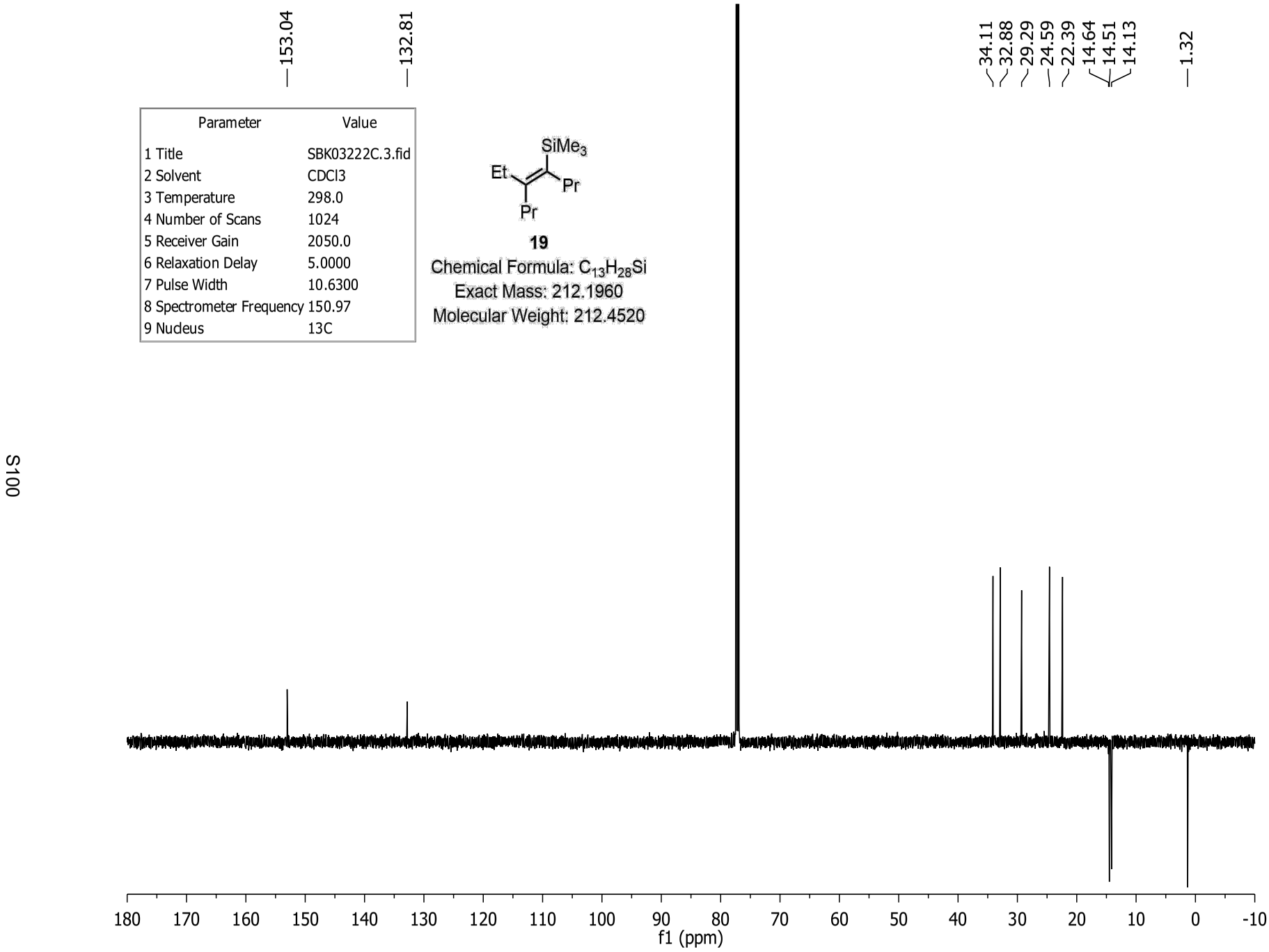


Parameter	Value
1 Title	SBK03222C.1.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	16
5 Receiver Gain	144.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

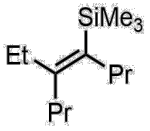


19

Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520


153.04
132.81
34.11
32.88
29.29
24.59
22.39
14.64
14.51
14.13
1.32
Parameter Value
1 Title SBK03222C.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe3
Et
Pr
Pr
19
Chemical Formula: C13H28Si
Exact Mass: 212.1960
Molecular Weight: 212.4520
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

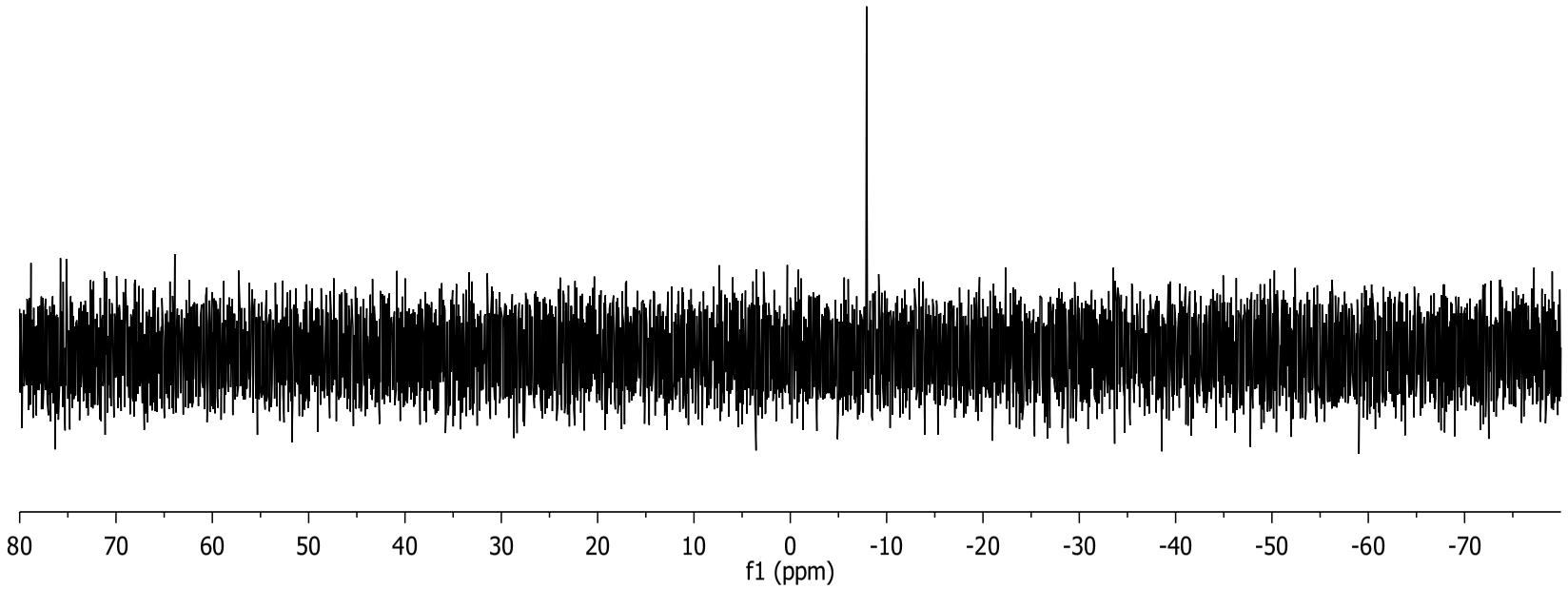
Parameter	Value
1 Title	SBK03222C.2.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

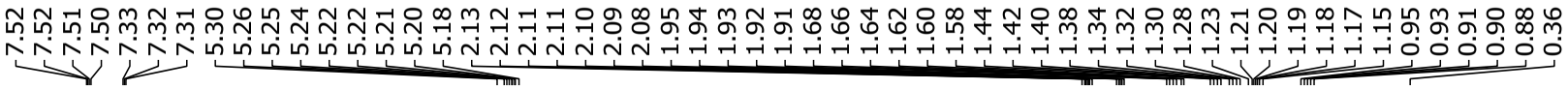


19

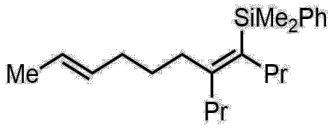
Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520

-7.92





Parameter	Value
1 Title	MFW02184col4A.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	4.0
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

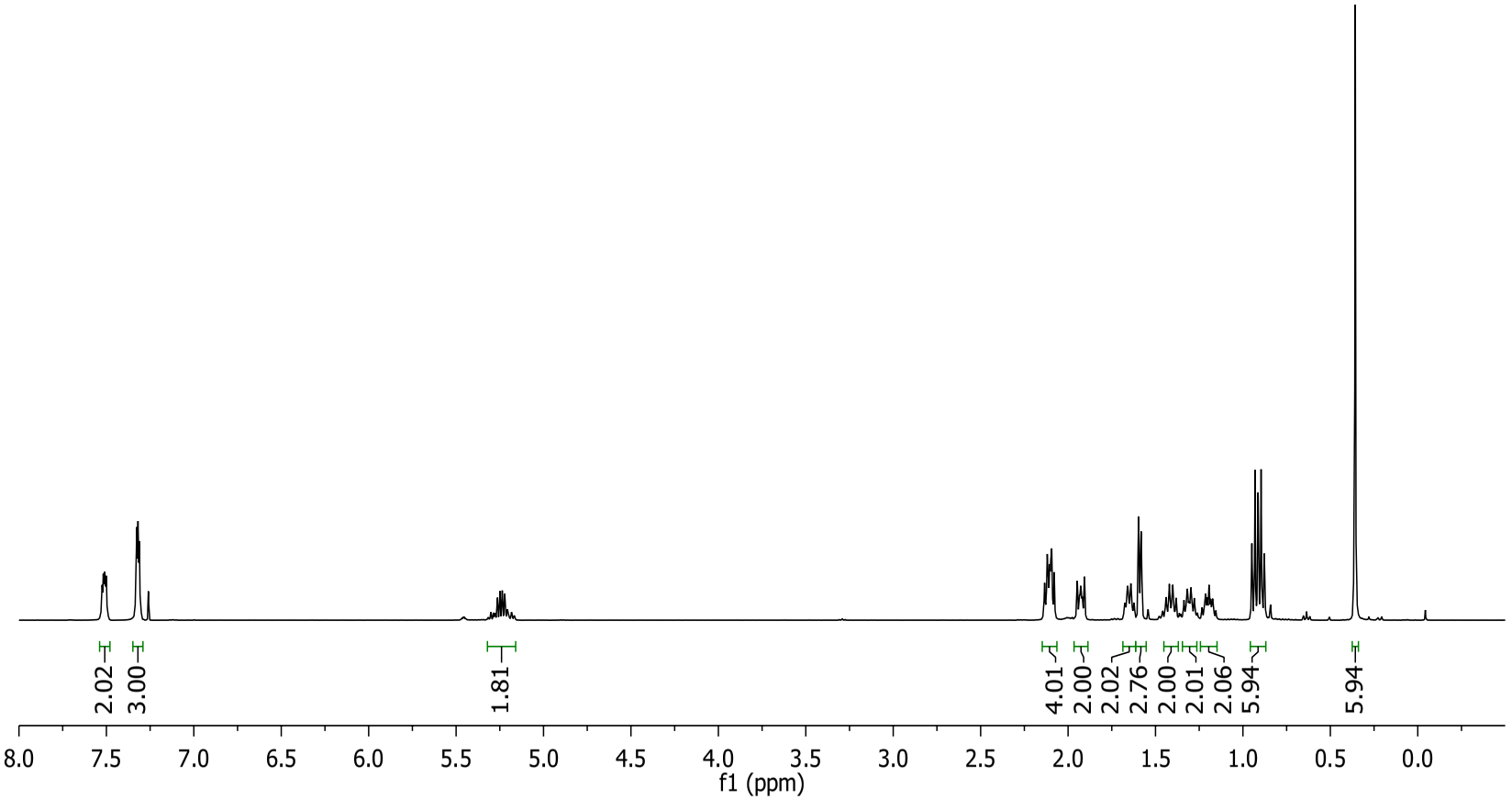


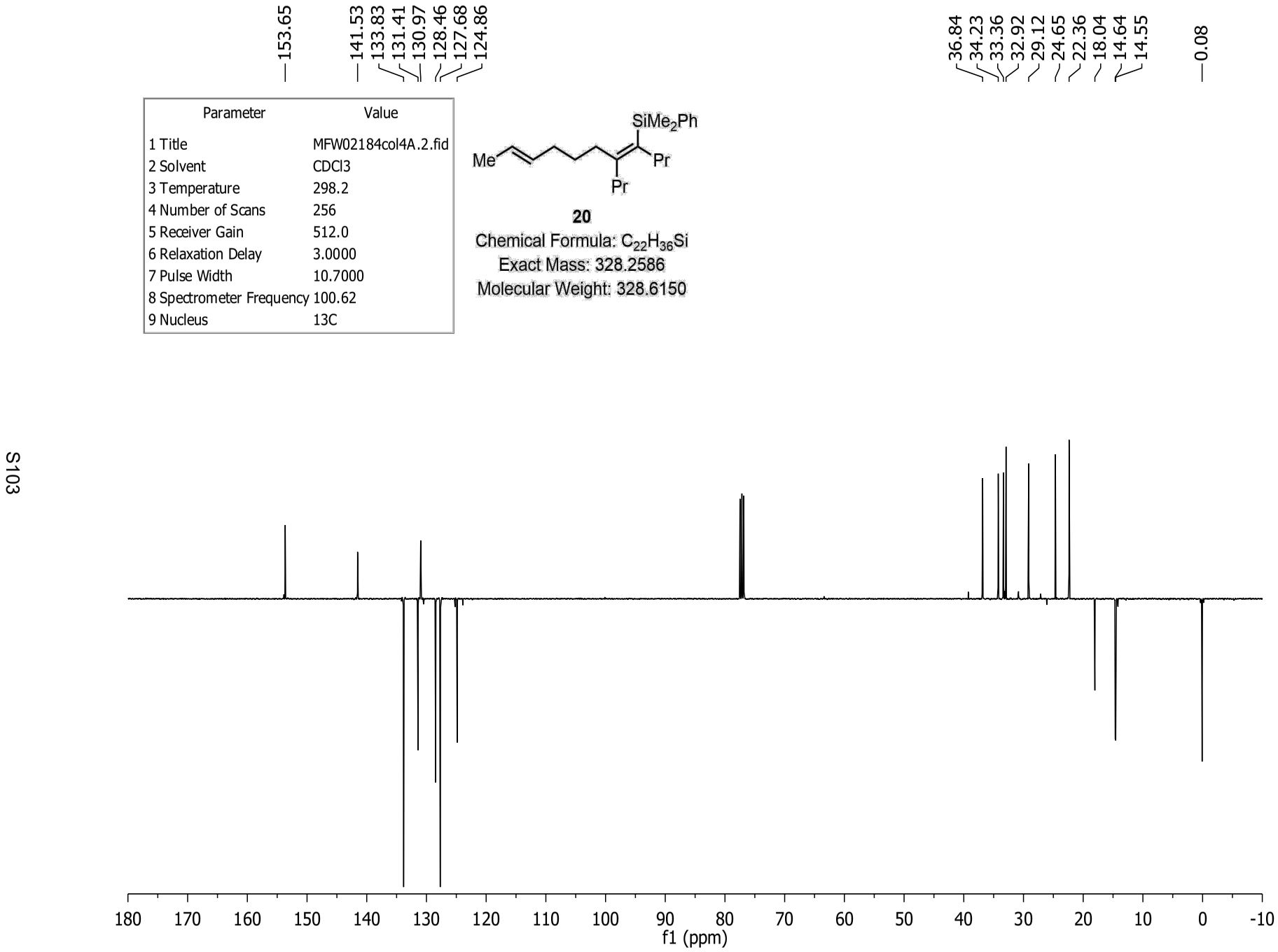
20

Chemical Formula: $C_{22}H_{36}Si$

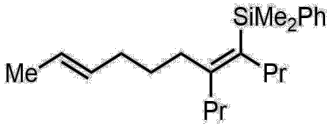
Exact Mass: 328.2586

Molecular Weight: 328.6150




153.65
141.53
133.83
131.41
130.97
128.46
127.68
124.86
36.84
34.23
33.36
32.92
29.12
24.65
22.36
18.04
14.64
14.55
0.08
Parameter
Value
1 Title
MFW02184col4A.2.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Ph
Me
Pr
Pr
20
Chemical Formula: C22H36Si
Exact Mass: 328.2586
Molecular Weight: 328.6150
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

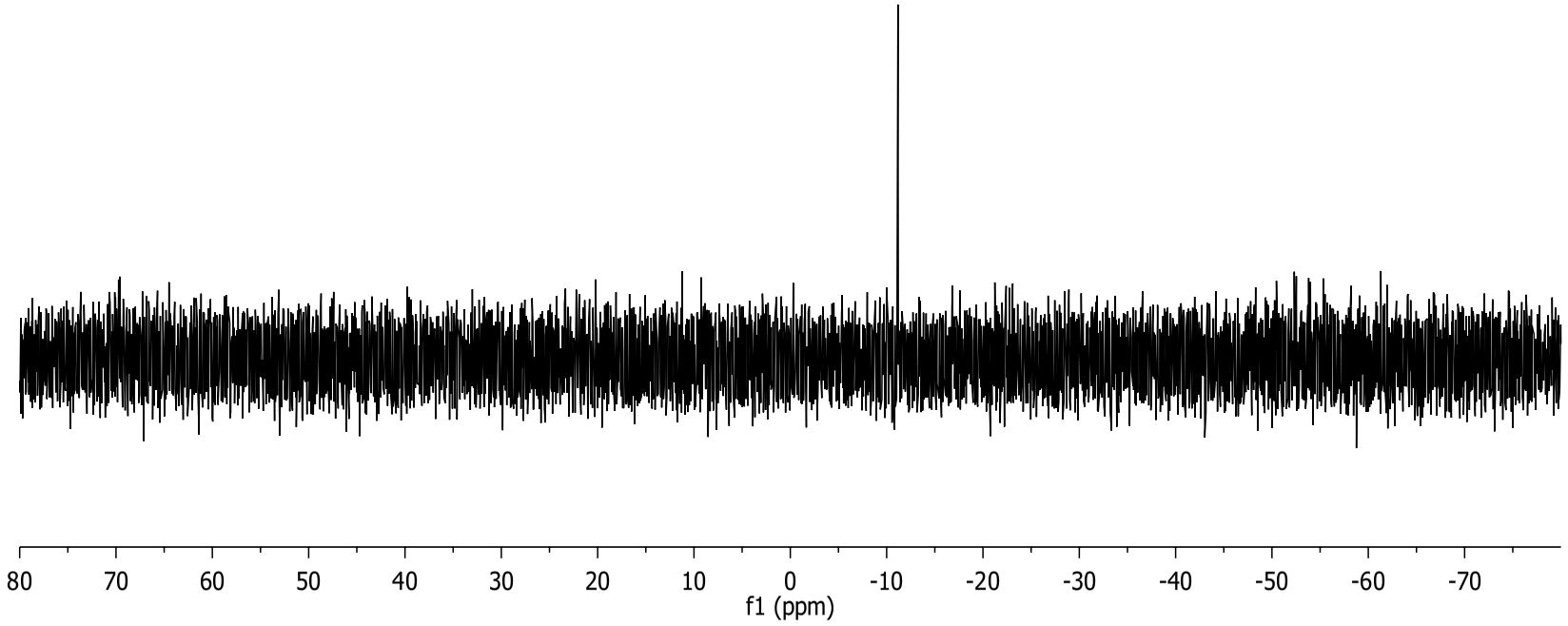
Parameter	Value
1 Title	MFW02184col3A.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

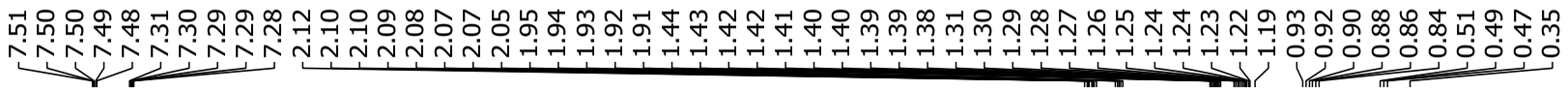


20

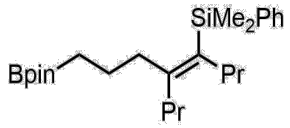
Chemical Formula: $C_{22}H_{36}Si$
Exact Mass: 328.2586
Molecular Weight: 328.6150

-11.17





Parameter	Value
1 Title	MFW02181col3A-NoE.1.fid
2 Solvent	CDCl3
3 Temperature	298.1
4 Number of Scans	16
5 Receiver Gain	8.0
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

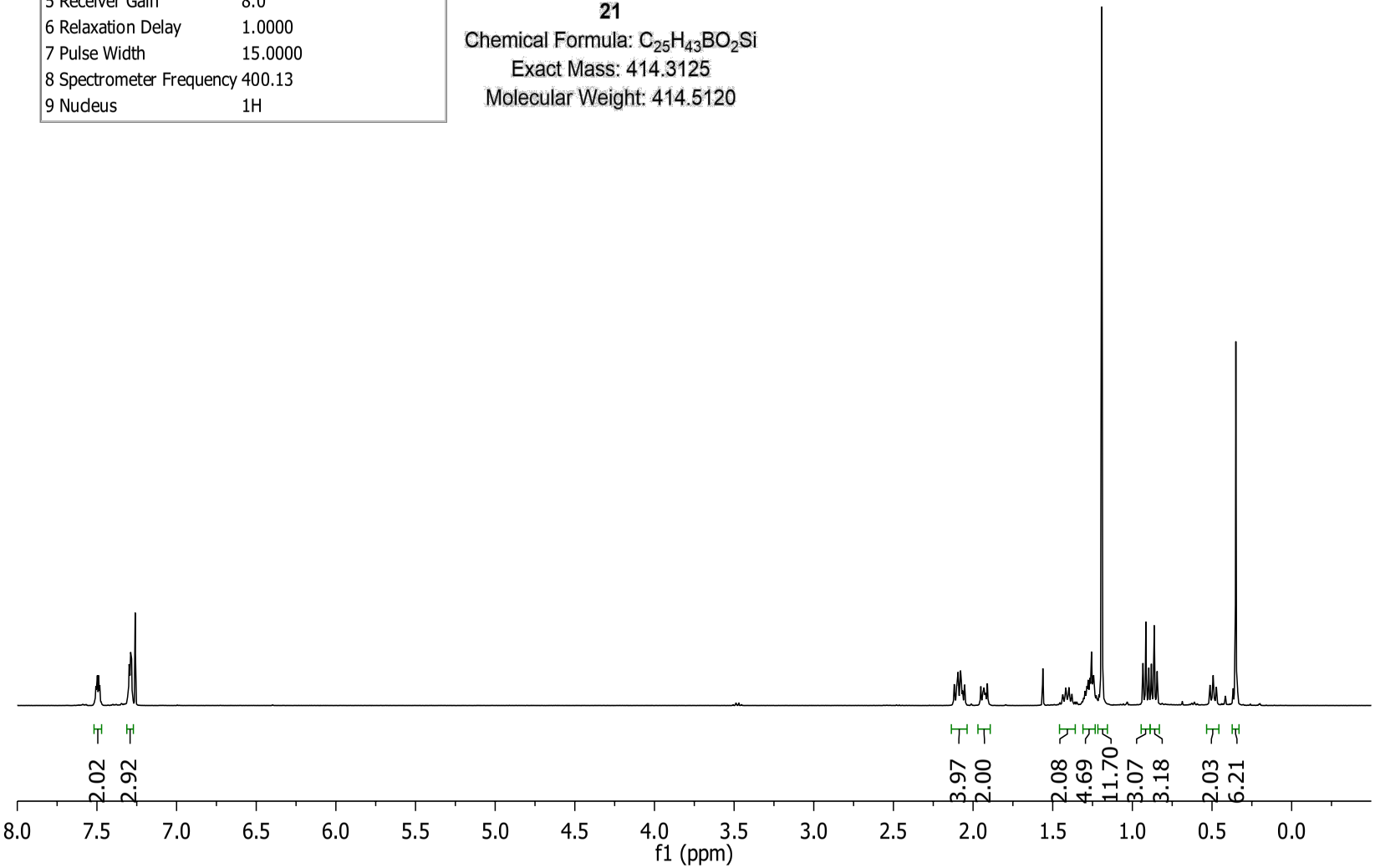


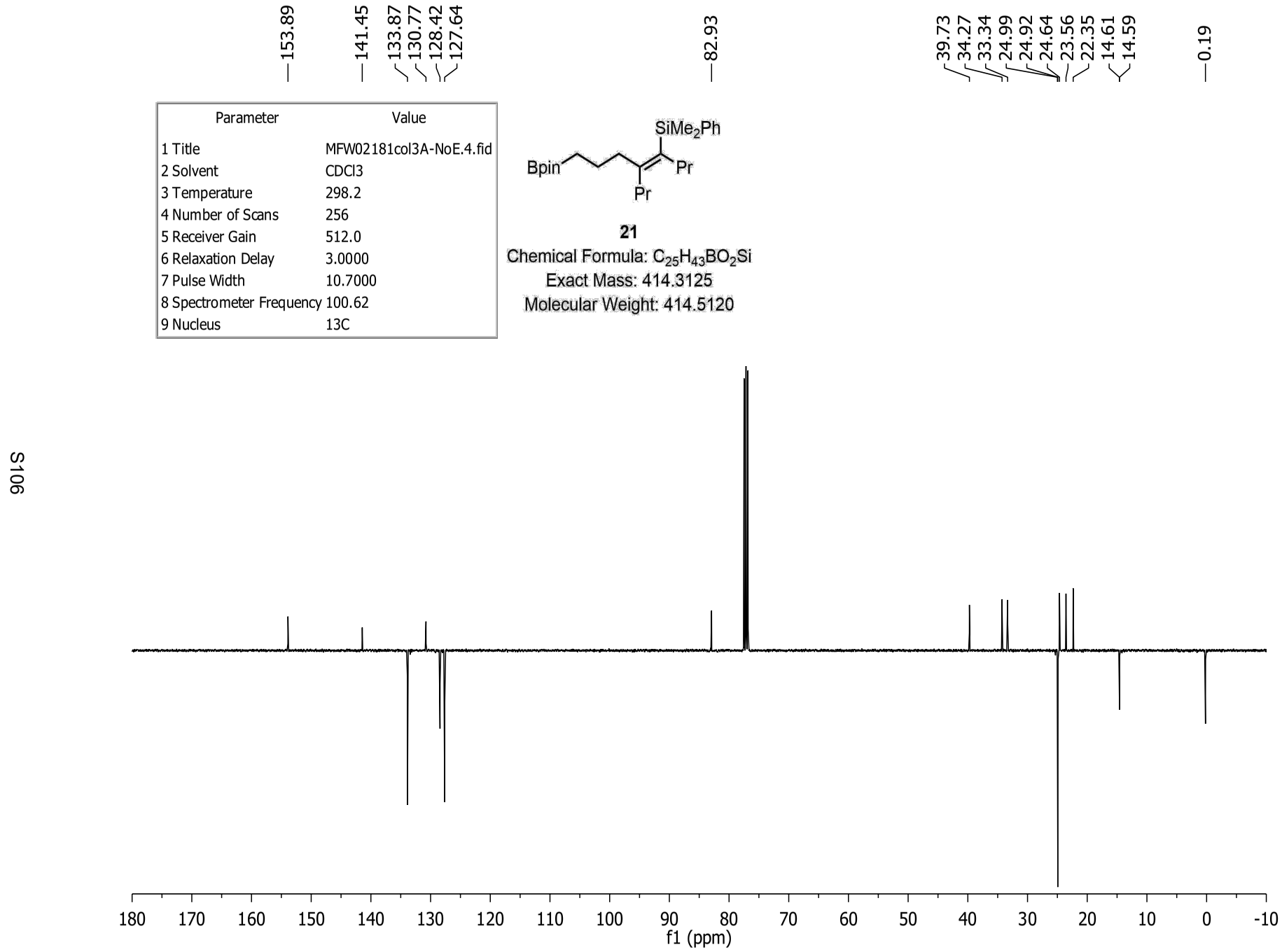
21

Chemical Formula: $C_{25}H_{43}BO_2Si$

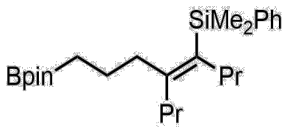
Exact Mass: 414.3125

Molecular Weight: 414.5120




153.89
141.45
133.87
130.77
128.42
127.64
82.93
39.73
34.27
33.34
24.99
24.92
24.64
23.56
22.35
14.61
14.59
0.19
Parameter
Value
1 Title
MFW02181col3A-NoE.4.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Ph
Bpin
Pr
Pr
21
Chemical Formula: C25H43BO2Si
Exact Mass: 414.3125
Molecular Weight: 414.5120
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

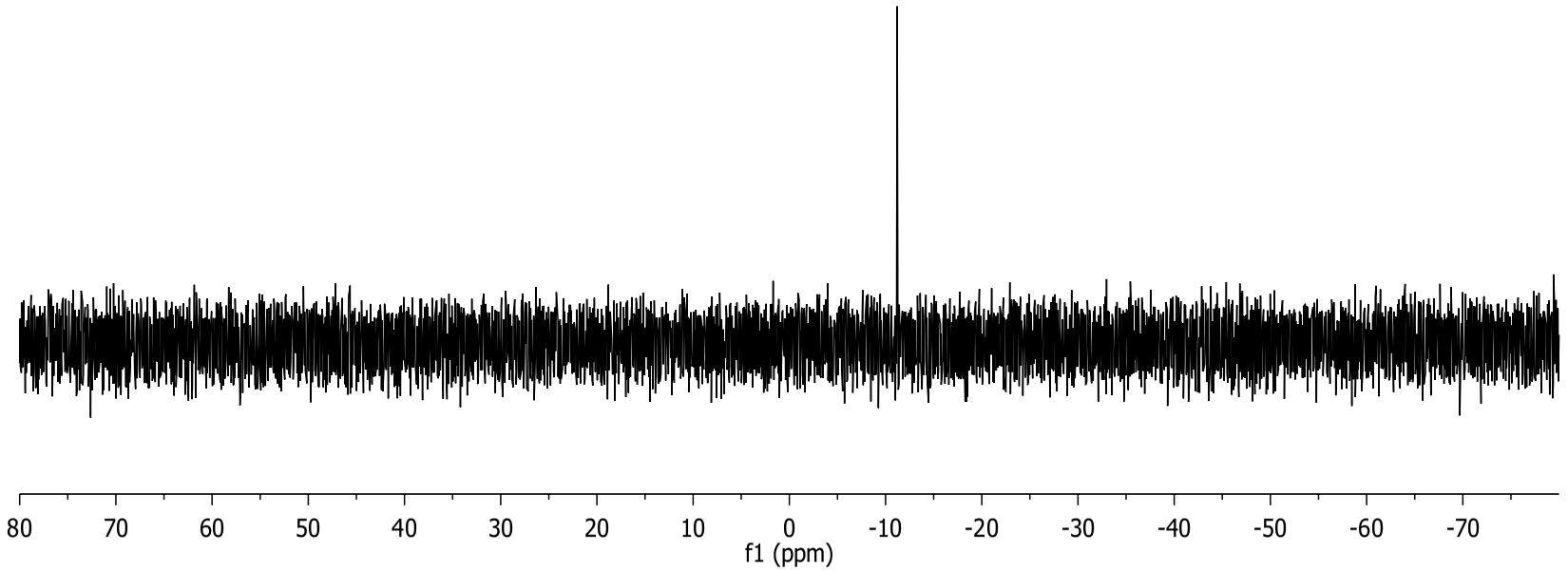
Parameter	Value
1 Title	MFW02181col3A.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



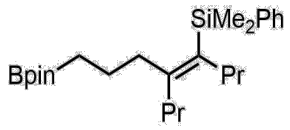
21

Chemical Formula: $C_{25}H_{43}BO_2Si$
Exact Mass: 414.3125
Molecular Weight: 414.5120

-11.21



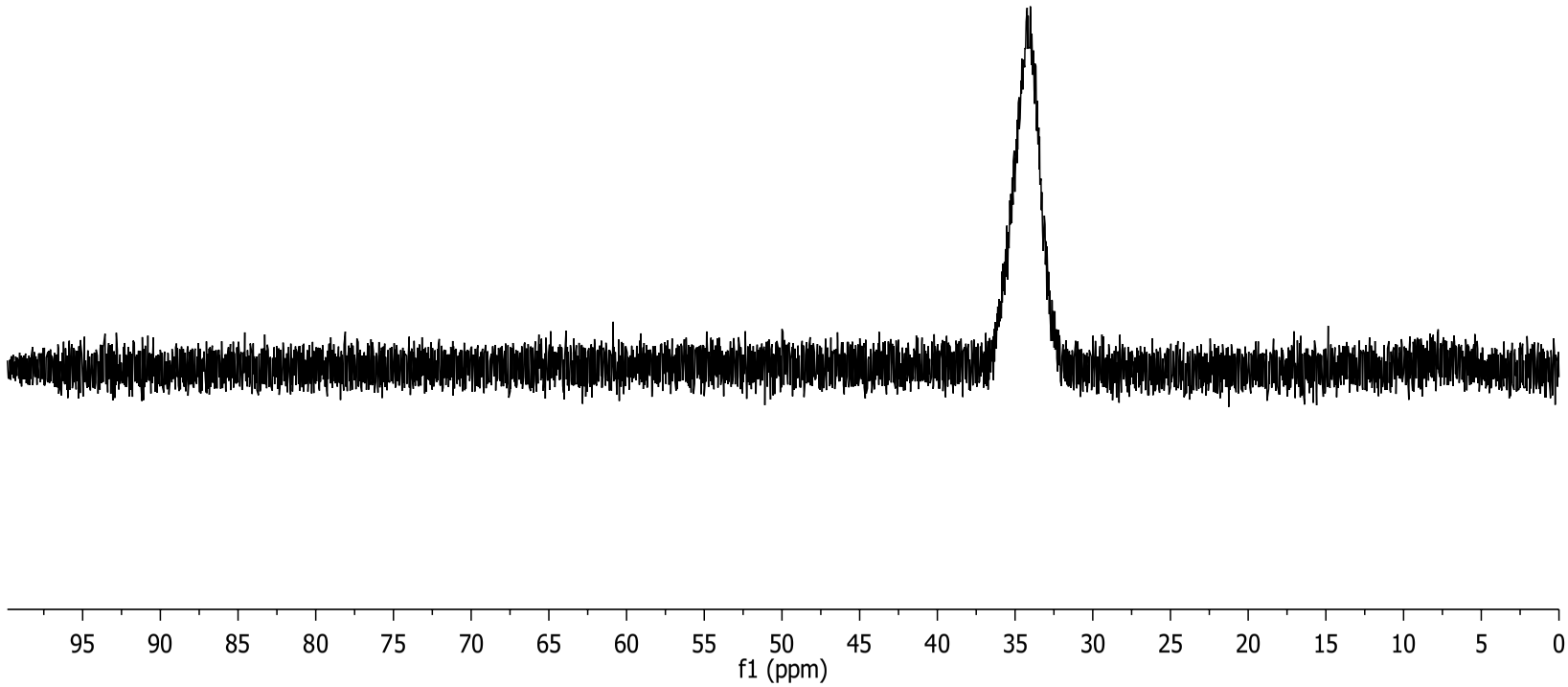
Parameter	Value
1 Title	MFW02181col3A.4.fid
2 Solvent	CDCl3
3 Temperature	297.2
4 Number of Scans	254
5 Receiver Gain	322.0
6 Relaxation Delay	1.0000
7 Pulse Width	11.0000
8 Spectrometer Frequency	192.61
9 Nucleus	11B

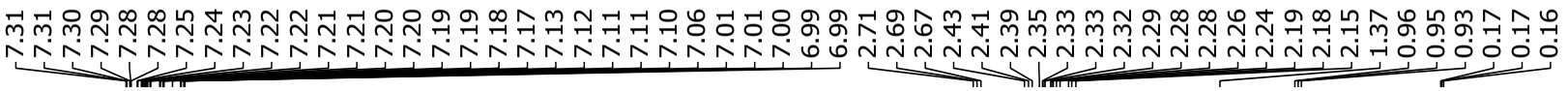


21

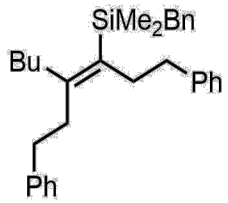
Chemical Formula: $C_{25}H_{43}BO_2Si$
Exact Mass: 414.3125
Molecular Weight: 414.5120

34.18





Parameter	Value
1 Title	MFW02119col2A-dry.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	11.3
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

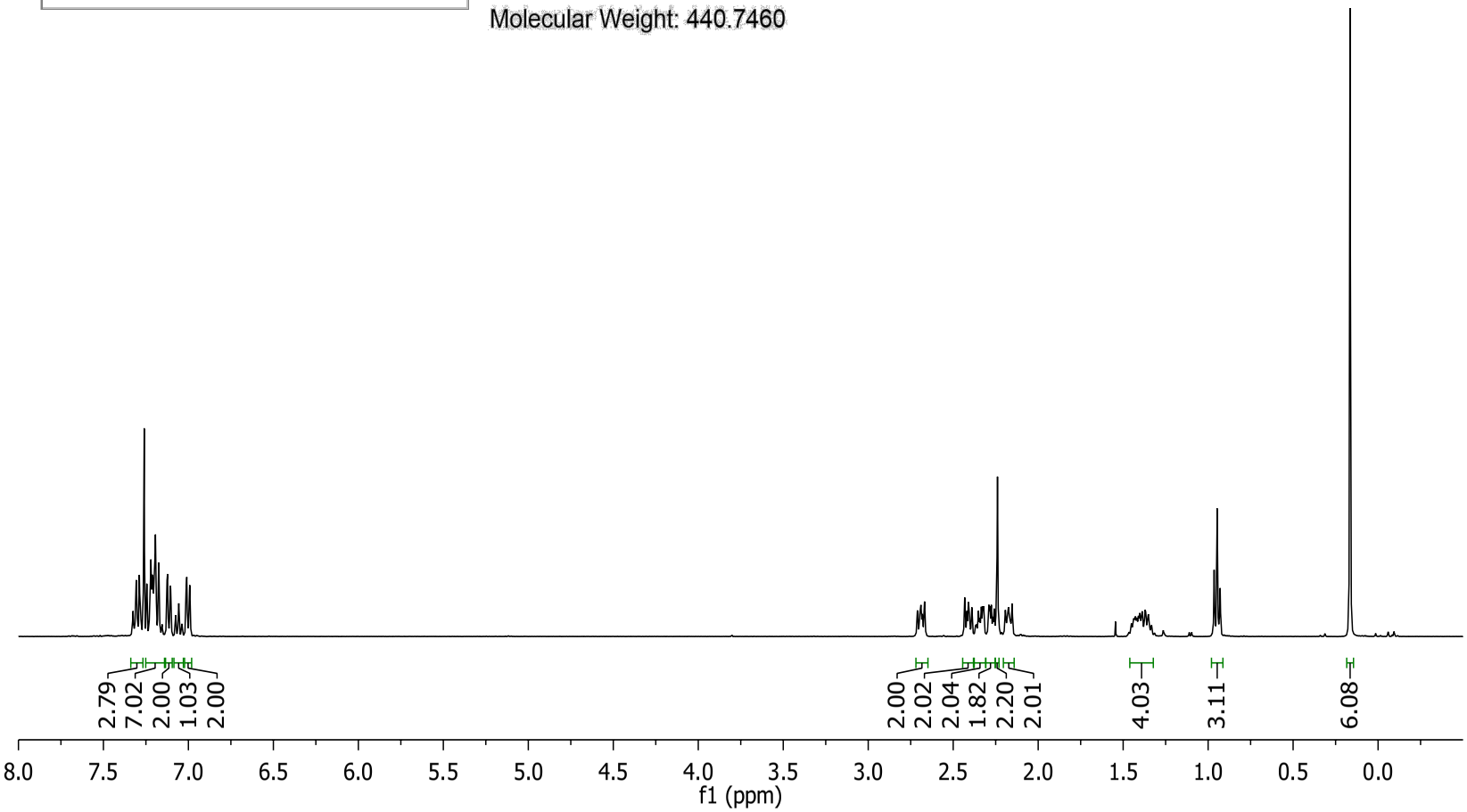


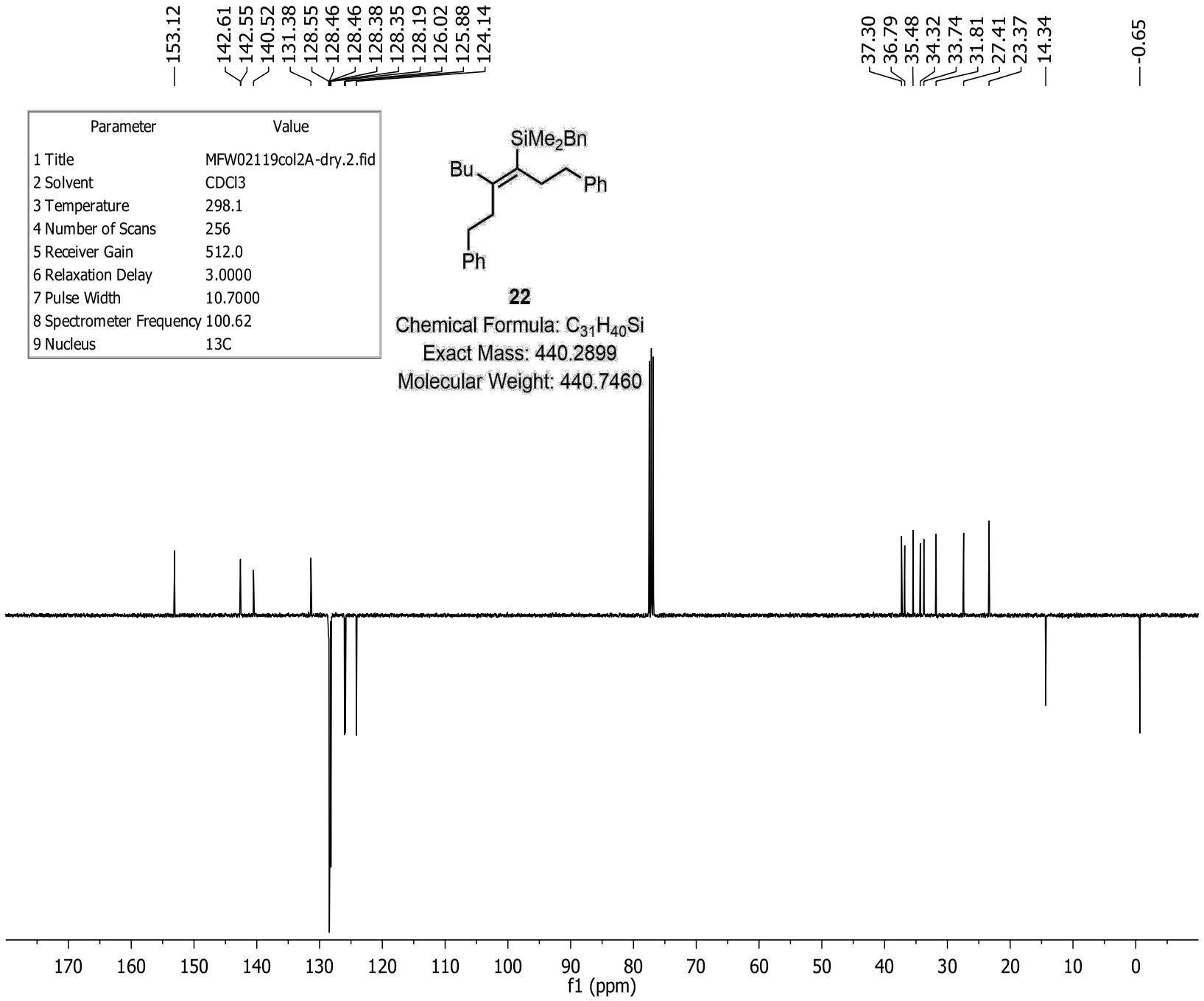
22

Chemical Formula: $C_{31}H_{40}Si$

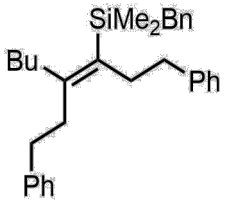
Exact Mass: 440.2899

Molecular Weight: 440.7460




153.12
142.61
142.55
140.52
131.38
128.55
128.46
128.46
128.38
128.35
128.19
126.02
125.88
124.14
37.30
36.79
35.48
34.32
33.74
31.81
27.41
23.37
14.34
-0.65
Parameter
Value
1 Title
MFW02119col2A-dry.2.fid
2 Solvent
CDCl3
3 Temperature
298.1
4 Number of Scans
256
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
10.7000
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Bn
Bu
Ph
Ph
22
Chemical Formula: C31H40Si
Exact Mass: 440.2899
Molecular Weight: 440.7460
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Parameter	Value
1 Title	MFW02119col2A.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

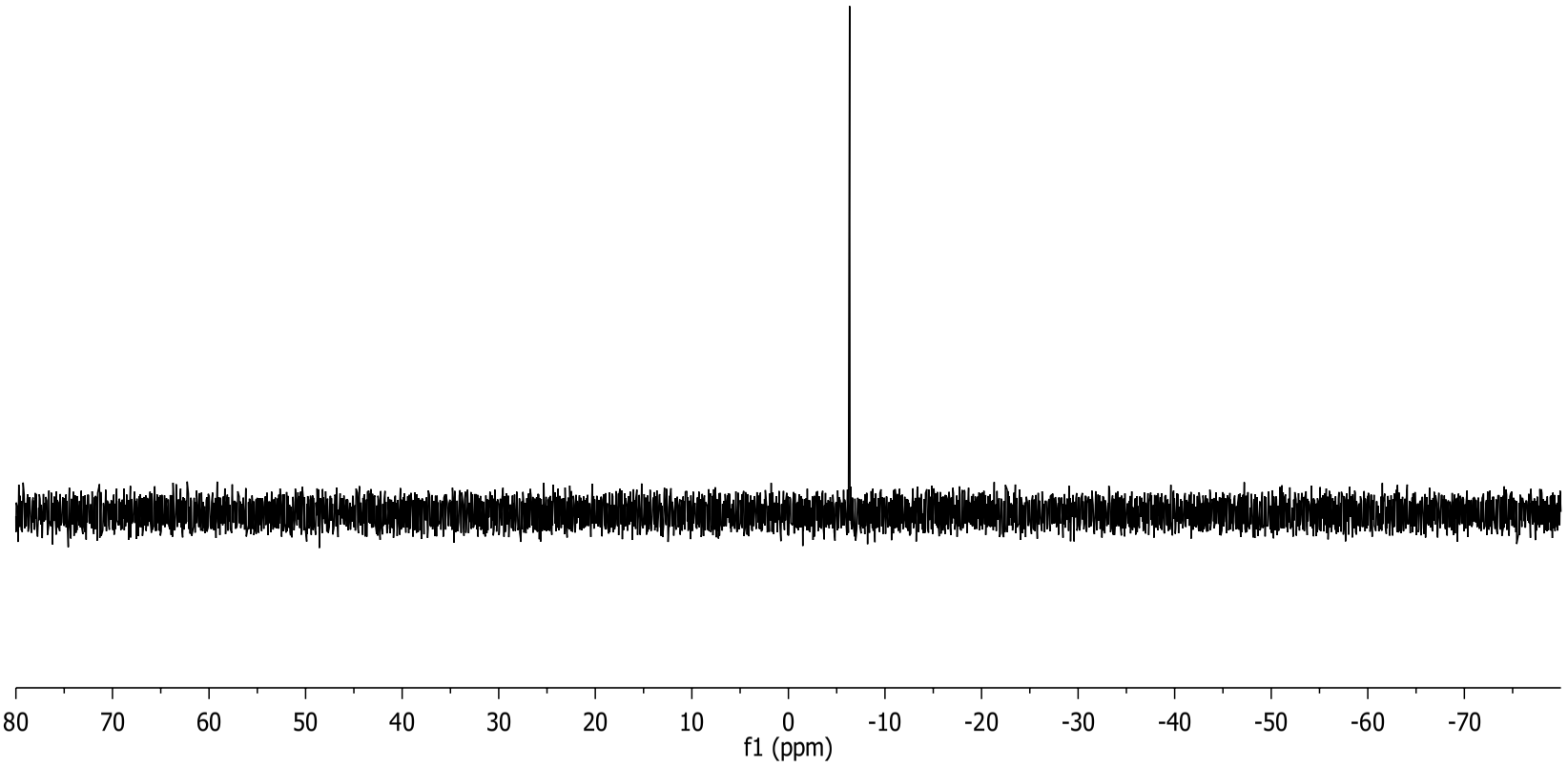


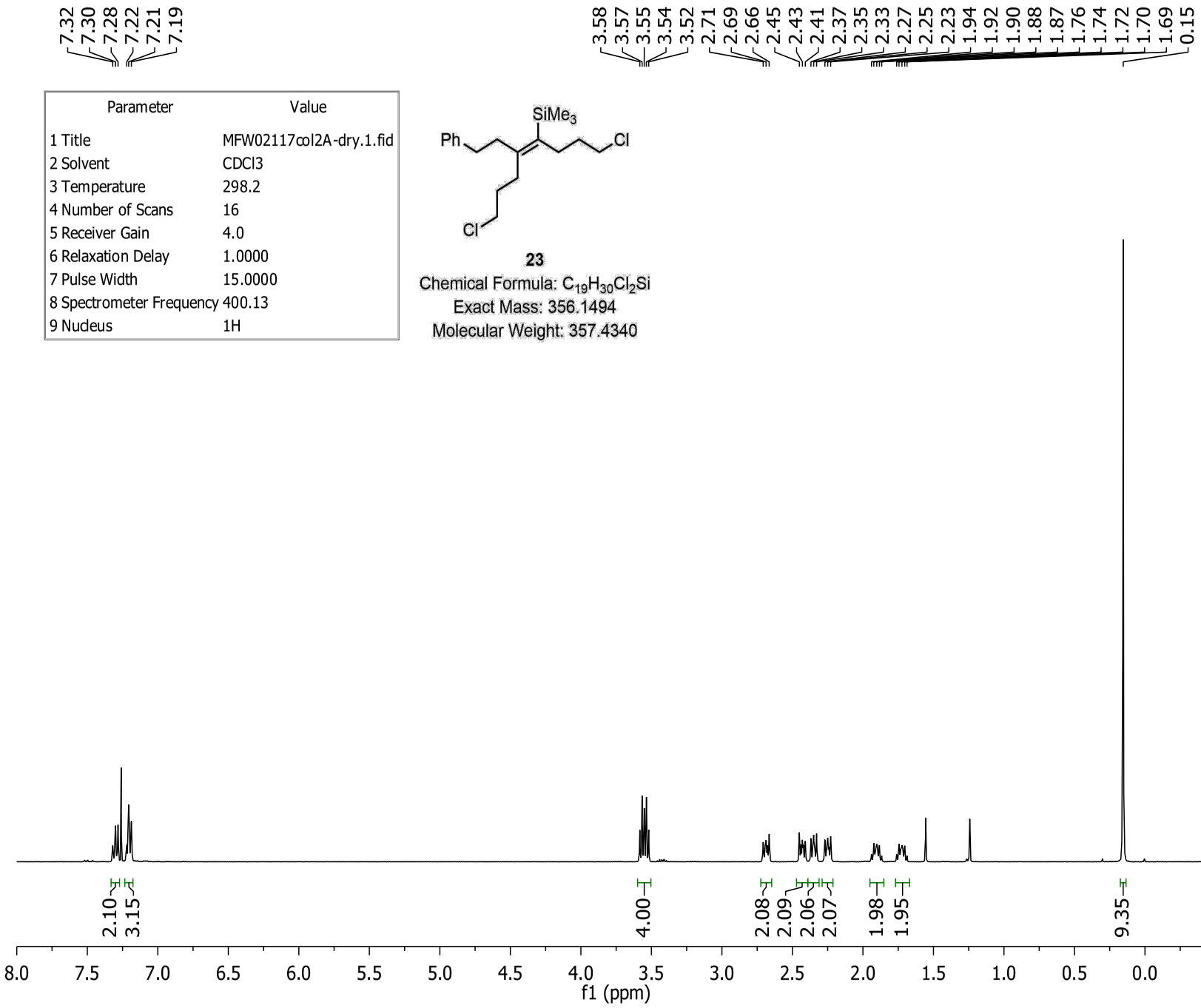
22

Chemical Formula: $C_{31}H_{40}Si$

Exact Mass: 440.2899

Molecular Weight: 440.7460



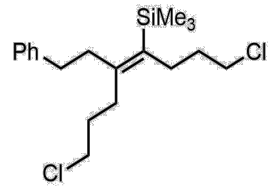

7.32
7.30
7.28
7.22
7.21
7.19
3.58
3.57
3.55
3.54
3.52
2.71
2.69
2.66
2.45
2.43
2.41
2.37
2.35
2.33
2.27
2.25
2.23
1.94
1.92
1.90
1.88
1.87
1.76
1.74
1.72
1.70
1.69
0.15
Parameter
Value
1 Title
MFW02117col2A-dry.1.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
16
5 Receiver Gain
4.0
6 Relaxation Delay
1.0000
7 Pulse Width
15.0000
8 Spectrometer Frequency
400.13
9 Nucleus
1H
SiMe3
Ph
Cl
Cl
23
Chemical Formula: C19H30Cl2Si
Exact Mass: 356.1494
Molecular Weight: 357.4340
2.10
3.15
4.00
2.08
2.09
2.06
2.07
1.98
1.95
9.35
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

150.19
142.04
134.41
128.61
128.38
126.11

45.30
45.22
38.57
35.80
34.00
32.12
29.35
28.60

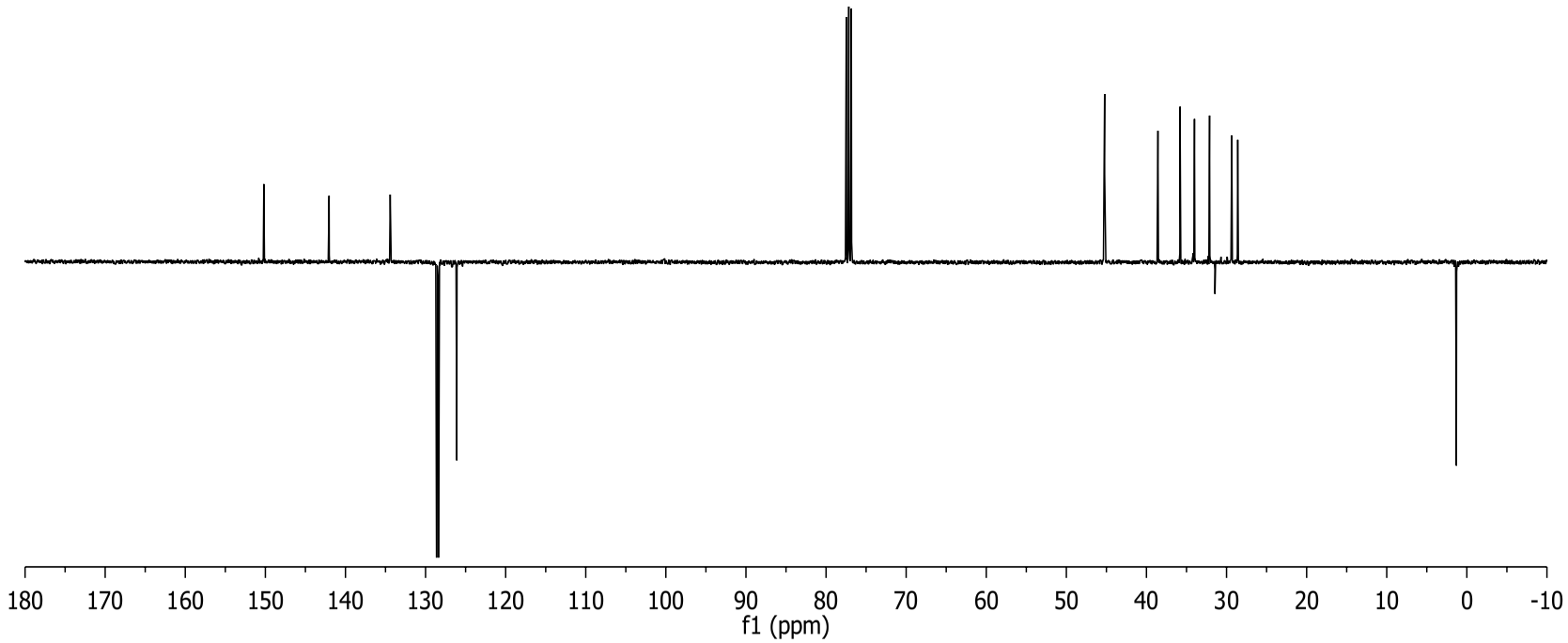
1.31

Parameter	Value
1 Title	MFW02117col2A-dry.2.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	256
5 Receiver Gain	512.0
6 Relaxation Delay	3.0000
7 Pulse Width	10.7000
8 Spectrometer Frequency	100.62
9 Nucleus	13C

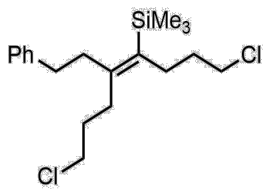


23

Chemical Formula: $C_{19}H_{30}Cl_2Si$
Exact Mass: 356.1494
Molecular Weight: 357.4340

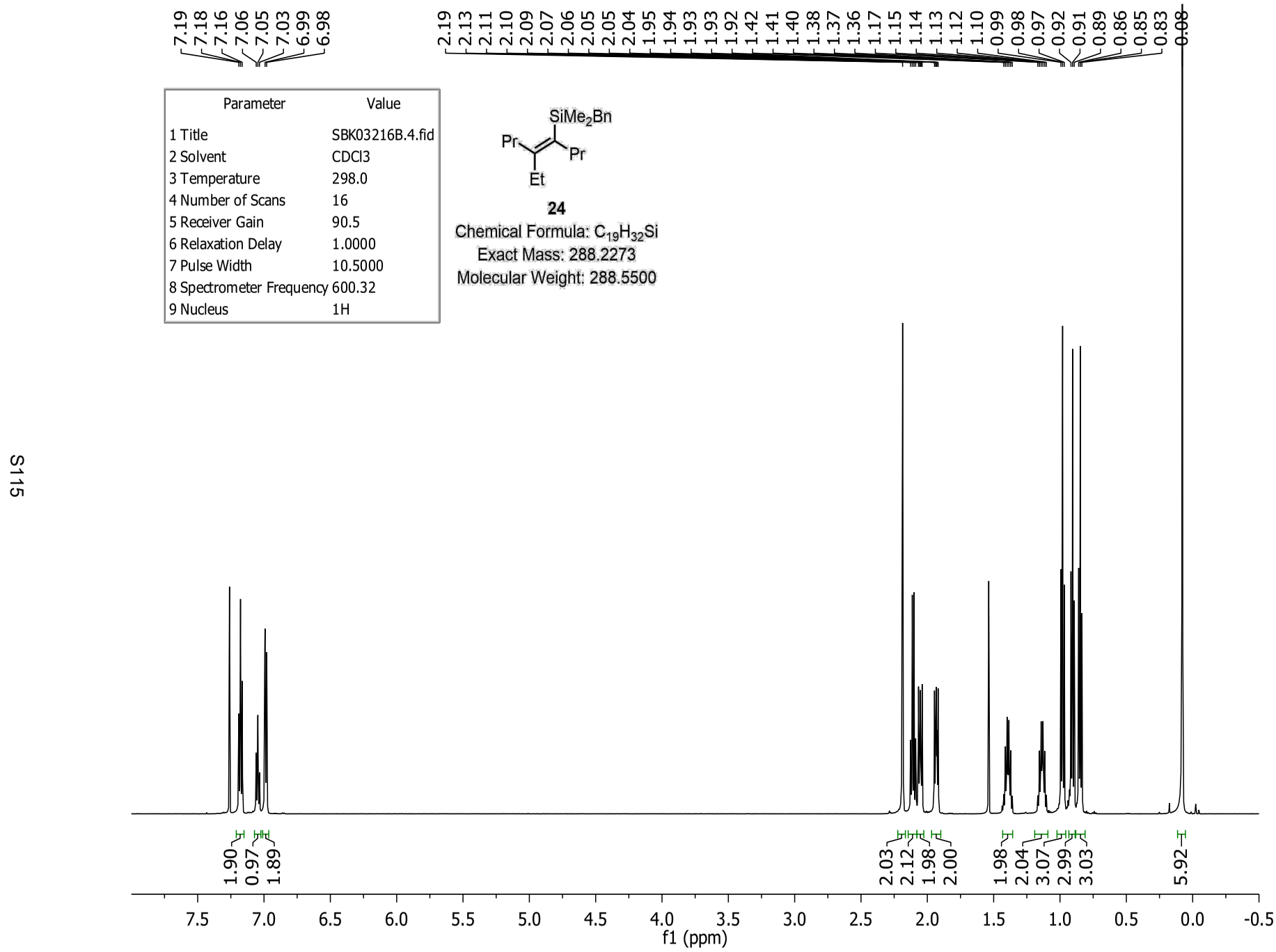


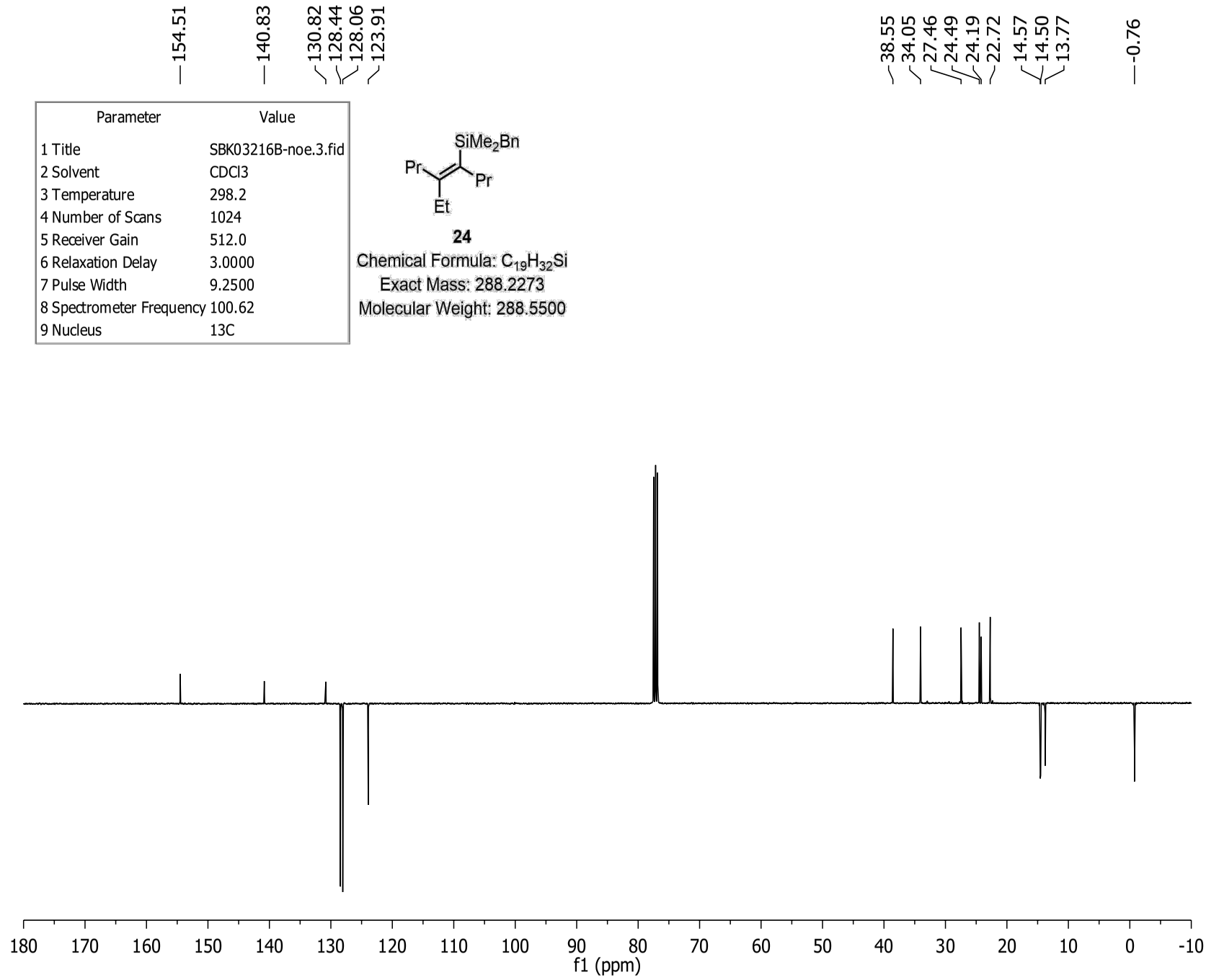
Parameter	Value
1 Title	MFW02117col2A.2.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	254
5 Receiver Gain	2050.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



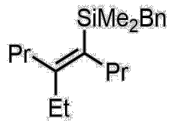
23

Chemical Formula: $C_{19}H_{30}Cl_2Si$
Exact Mass: 356.1494
Molecular Weight: 357.4340


7.19
7.18
7.16
7.06
7.05
7.03
6.99
6.98
2.19
2.13
2.11
2.10
2.09
2.07
2.06
2.05
2.05
2.04
1.95
1.94
1.93
1.93
1.92
1.42
1.41
1.40
1.38
1.37
1.36
1.17
1.15
1.14
1.13
1.12
1.10
0.99
0.98
0.97
0.92
0.91
0.89
0.86
0.85
0.83
0.08
Parameter
Value
1 Title
SBK03216B.4.fid
2 Solvent
CDCl3
3 Temperature
298.0
4 Number of Scans
16
5 Receiver Gain
90.5
6 Relaxation Delay
1.0000
7 Pulse Width
10.5000
8 Spectrometer Frequency
600.32
9 Nucleus
1H
SiMe2Bn
Pr
Pr
Et
24
Chemical Formula: C19H32Si
Exact Mass: 288.2273
Molecular Weight: 288.5500
1.90
0.97
1.89
2.03
2.12
1.98
2.00
1.98
2.04
3.07
2.99
3.03
5.92
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


154.51
140.83
130.82
128.44
128.06
123.91
38.55
34.05
27.46
24.49
24.19
22.72
14.57
14.50
13.77
-0.76
Parameter
Value
1 Title
SBK03216B-noe.3.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
1024
5 Receiver Gain
512.0
6 Relaxation Delay
3.0000
7 Pulse Width
9.2500
8 Spectrometer Frequency
100.62
9 Nucleus
13C
SiMe2Bn
Pr
Pr
Et
24
Chemical Formula: C19H32Si
Exact Mass: 288.2273
Molecular Weight: 288.5500
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

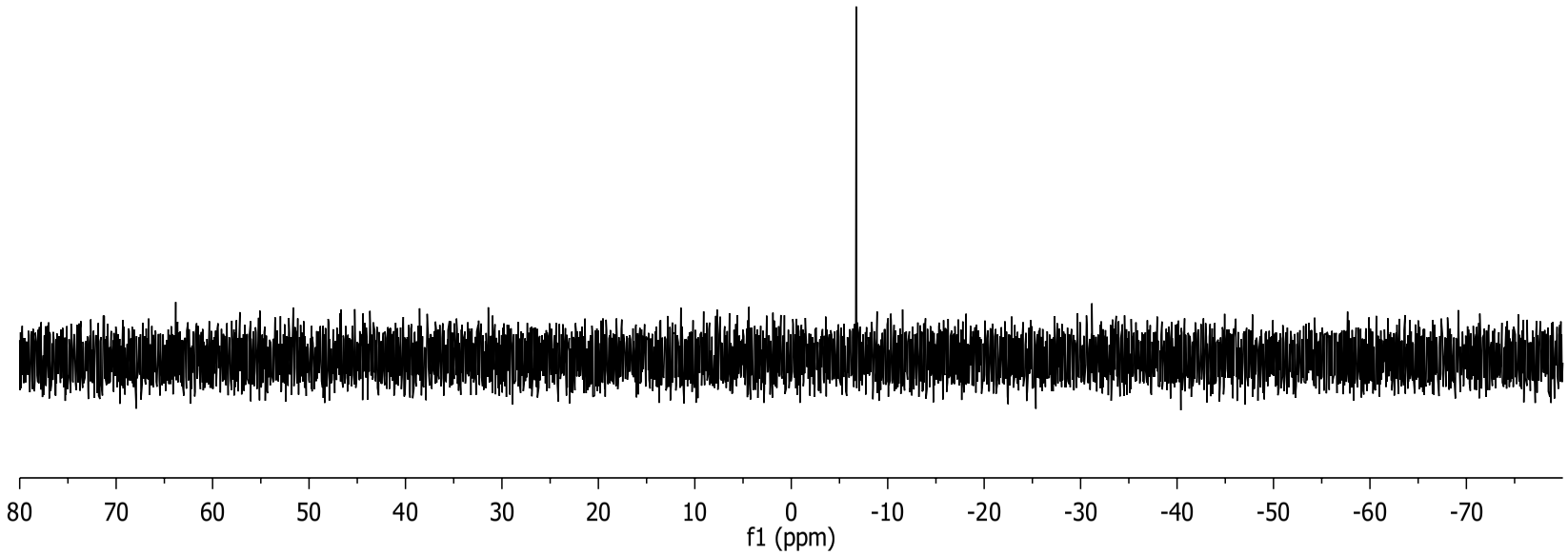
Parameter	Value
1 Title	SBK03216B.2.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



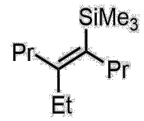
24

Chemical Formula: $C_{19}H_{32}Si$
Exact Mass: 288.2273
Molecular Weight: 288.5500

-6.73

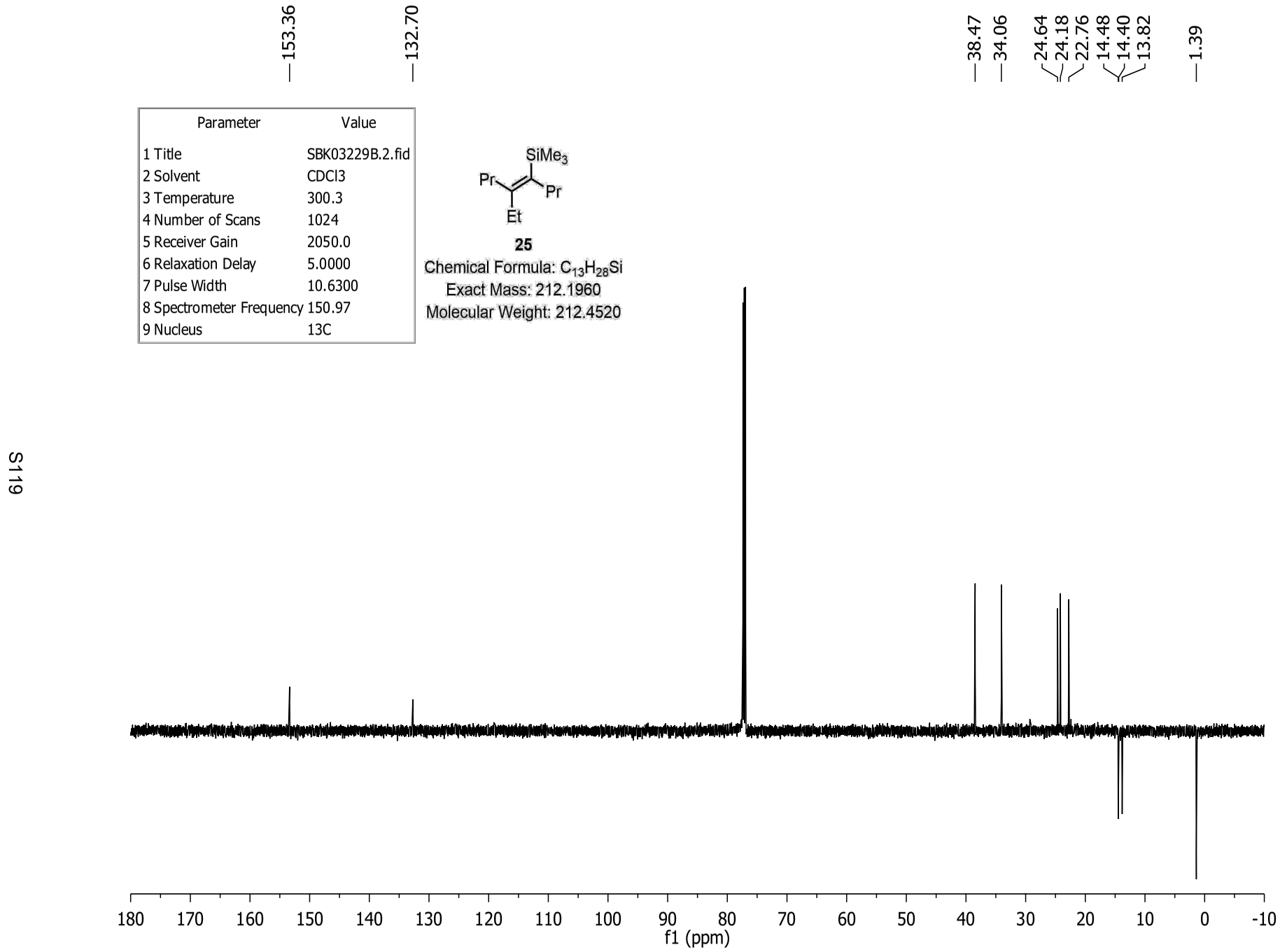


Parameter	Value
1 Title	SBK03229B.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	114.0
6 Relaxation Delay	1.0000
7 Pulse Width	10.5000
8 Spectrometer Frequency	600.32
9 Nucleus	1H

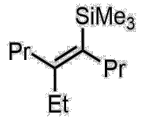


25

Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520


153.36
132.70
38.47
34.06
24.64
24.18
22.76
14.48
14.40
13.82
1.39
Parameter Value
1 Title SBK03229B.2.fid
2 Solvent CDCl3
3 Temperature 300.3
4 Number of Scans 1024
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
SiMe3
Pr
Pr
Et
25
Chemical Formula: C13H28Si
Exact Mass: 212.1960
Molecular Weight: 212.4520
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

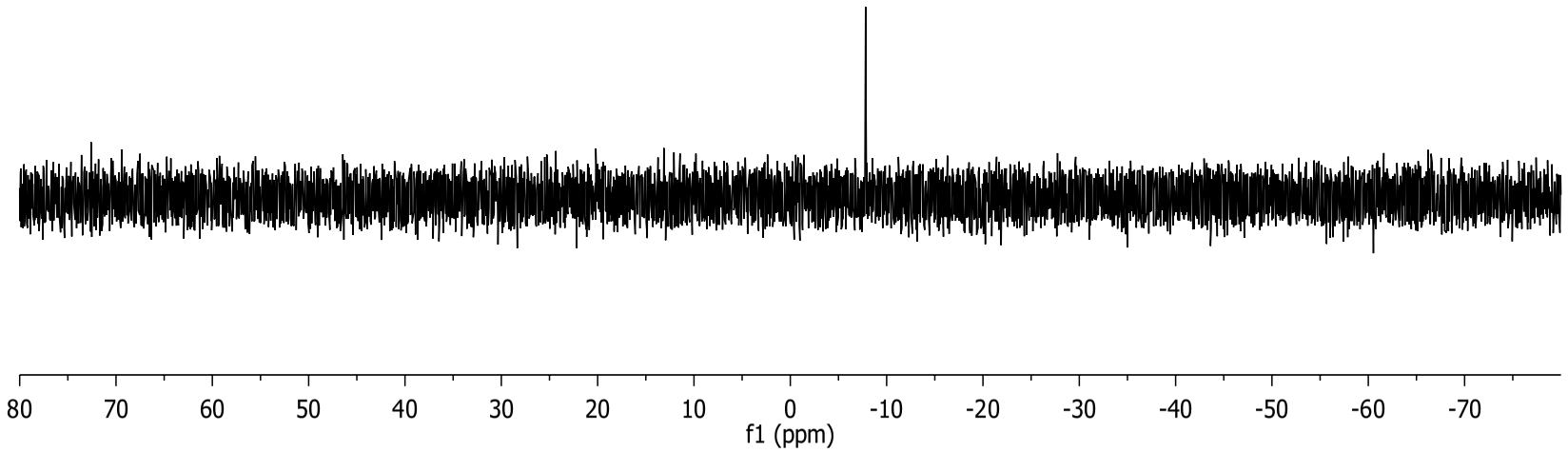
Parameter	Value
1 Title	SBK03229B.3.fid
2 Solvent	CDCl3
3 Temperature	297.7
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



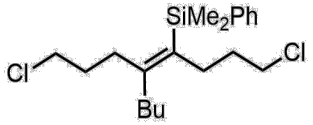
25

Chemical Formula: $C_{13}H_{28}Si$
Exact Mass: 212.1960
Molecular Weight: 212.4520

-7.81



Parameter	Value
1 Title	SBK04017B.1.fid
2 Solvent	CDCl3
3 Temperature	298.2
4 Number of Scans	16
5 Receiver Gain	5.7
6 Relaxation Delay	1.0000
7 Pulse Width	15.0000
8 Spectrometer Frequency	400.13
9 Nucleus	1H

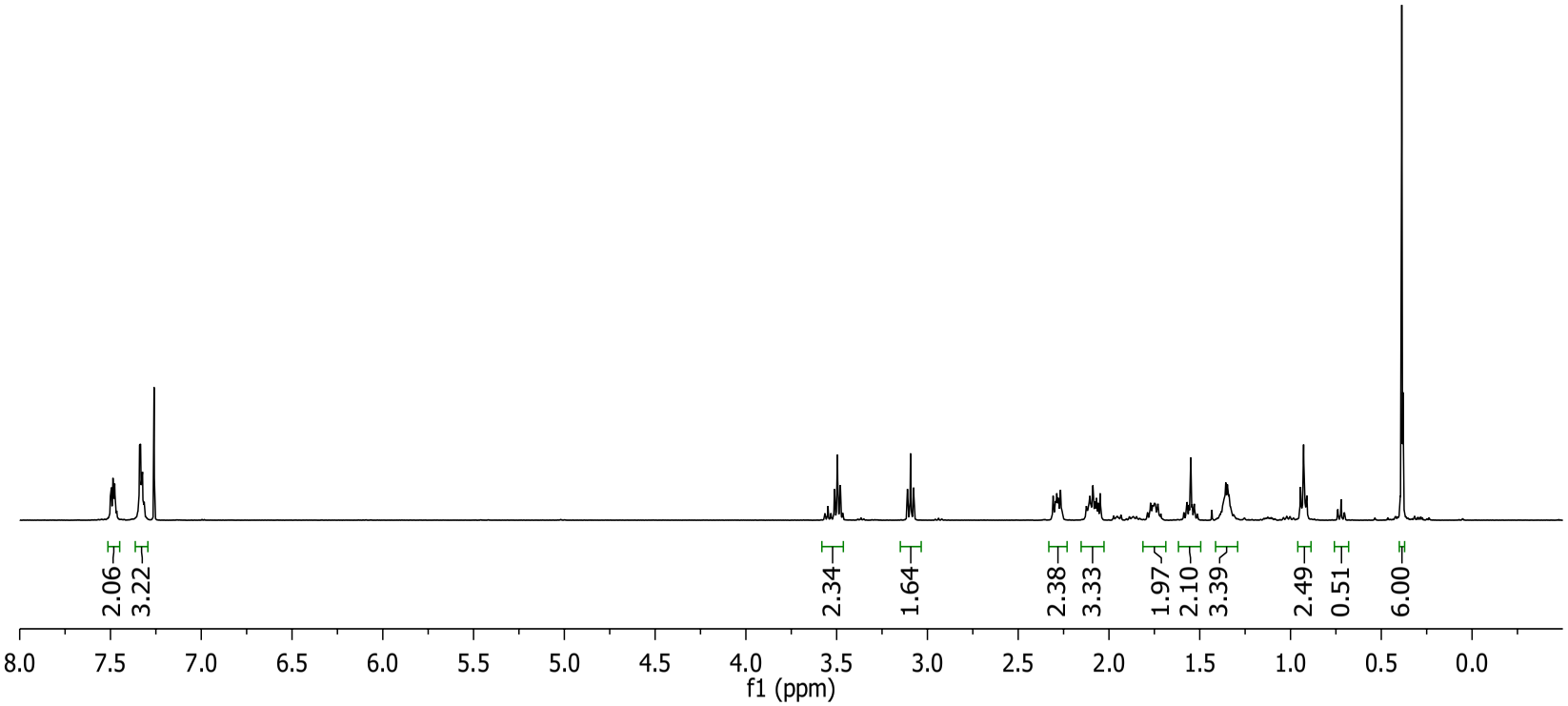


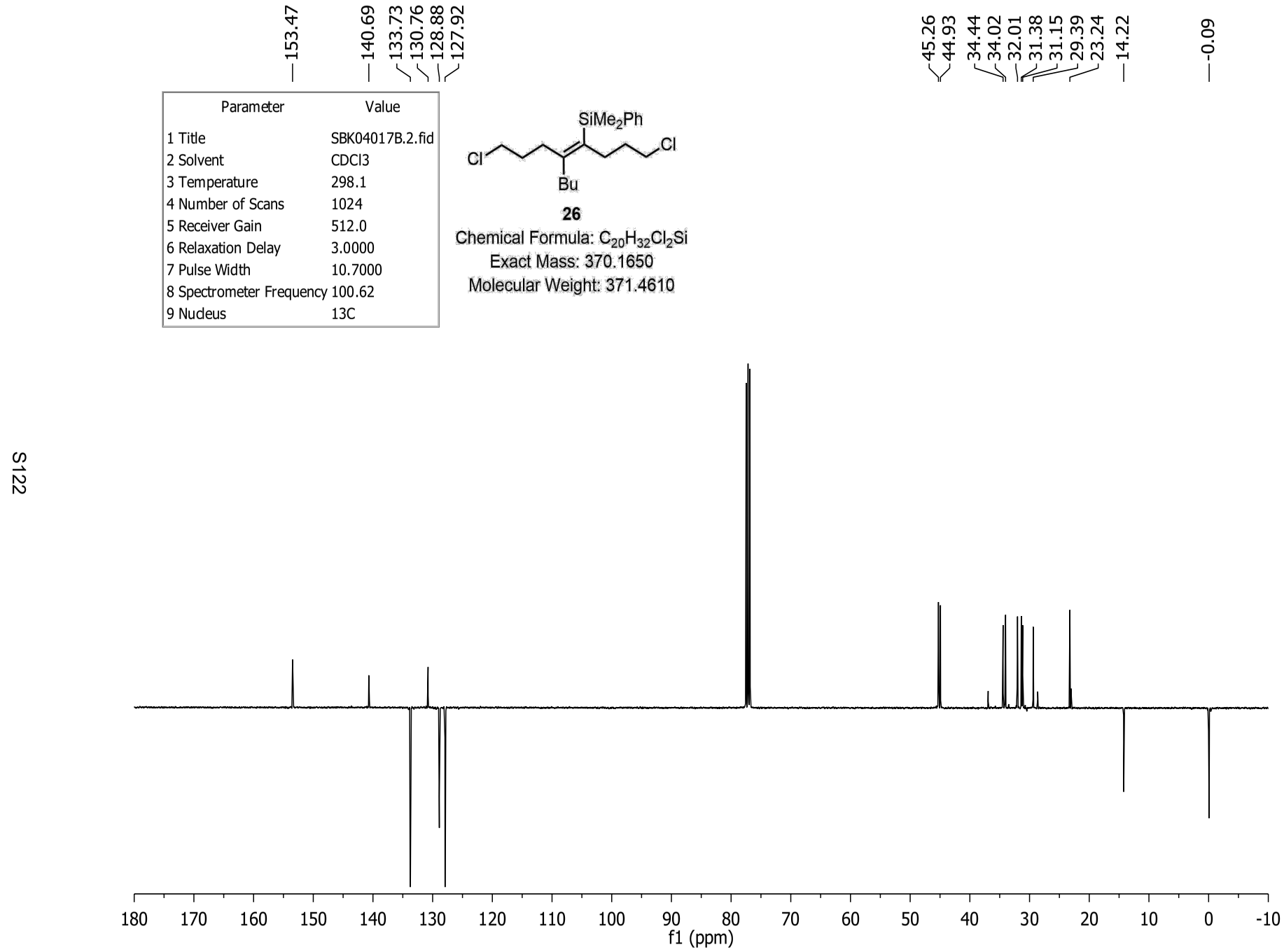
26

Chemical Formula: $C_{20}H_{32}Cl_2Si$

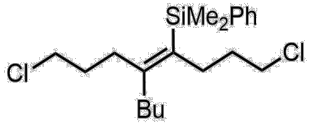
Exact Mass: 370.1650

Molecular Weight: 371.4610



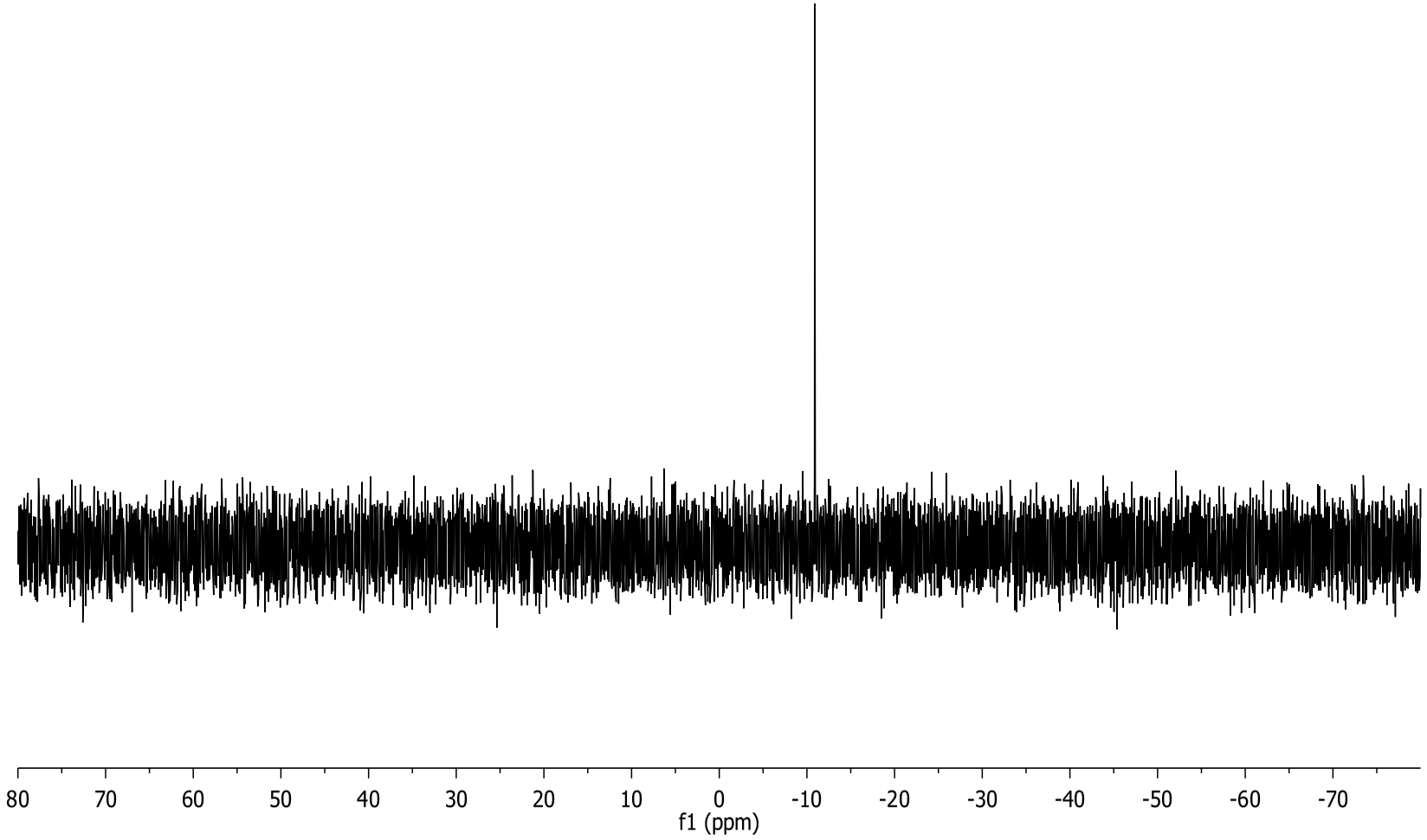

153.47
140.69
133.73
130.76
128.88
127.92
45.26
44.93
34.44
34.02
32.01
31.38
31.15
29.39
23.24
14.22
-0.09
Parameter Value
1 Title SBK04017B.2.fid
2 Solvent CDCl3
3 Temperature 298.1
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Cl
Cl
Bu
26
Chemical Formula: C20H32Cl2Si
Exact Mass: 370.1650
Molecular Weight: 371.4610
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

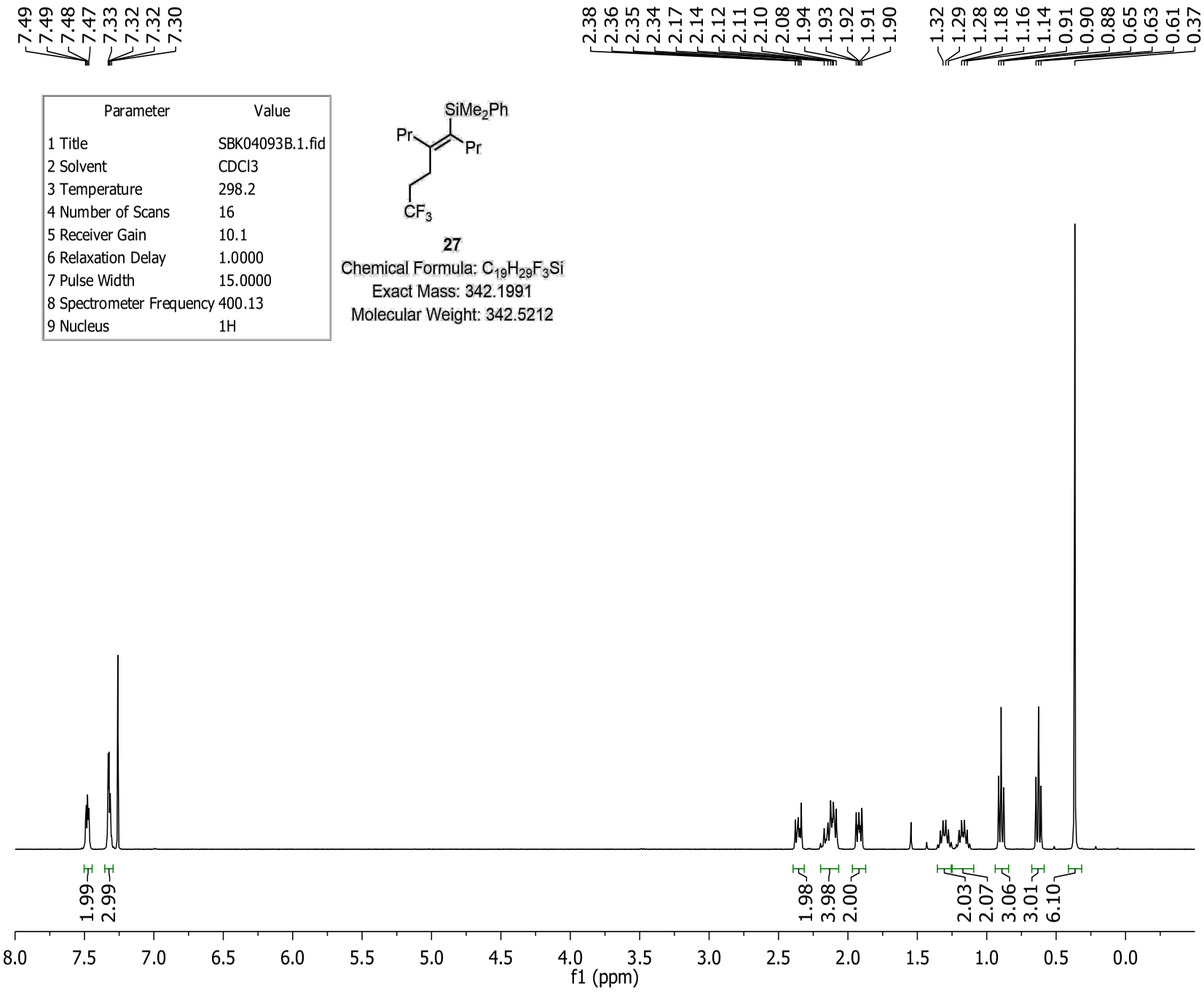
Parameter	Value
1 Title	SBK04017B-Si.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1620.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si

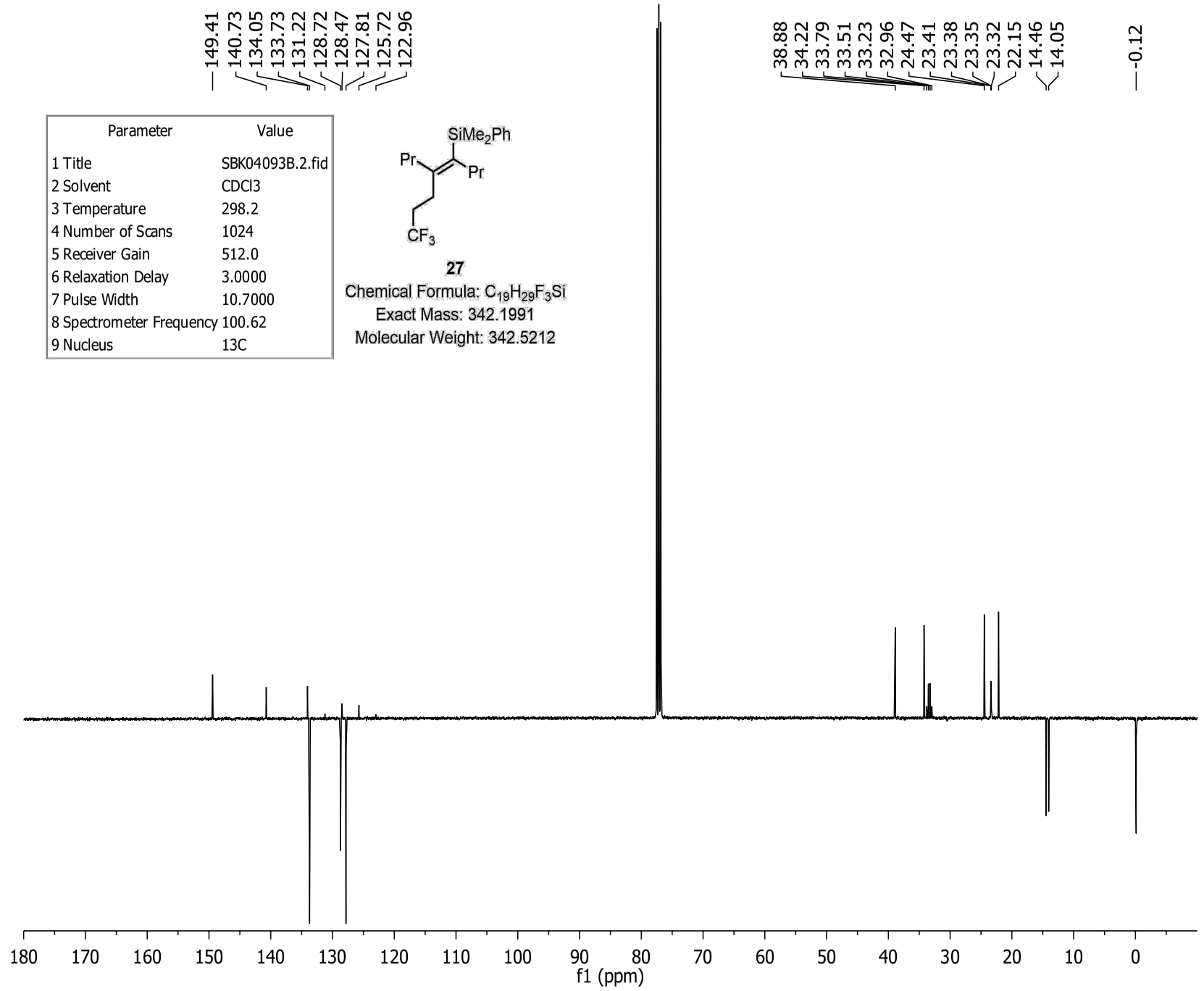


26

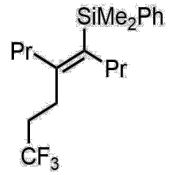
Chemical Formula: $C_{20}H_{32}Cl_2Si$
Exact Mass: 370.1650
Molecular Weight: 371.4610




7.49
7.49
7.48
7.47
7.33
7.32
7.32
7.30
2.38
2.36
2.35
2.34
2.17
2.14
2.12
2.11
2.10
2.08
1.94
1.93
1.92
1.91
1.90
1.32
1.29
1.28
1.18
1.16
1.14
0.91
0.90
0.88
0.65
0.63
0.61
0.37
Parameter
Value
1 Title
SBK04093B.1.fid
2 Solvent
CDCl3
3 Temperature
298.2
4 Number of Scans
16
5 Receiver Gain
10.1
6 Relaxation Delay
1.0000
7 Pulse Width
15.0000
8 Spectrometer Frequency
400.13
9 Nucleus
1H
SiMe2Ph
Pr
Pr
CF3
27
Chemical Formula: C19H29F3Si
Exact Mass: 342.1991
Molecular Weight: 342.5212
1.99
2.99
1.98
3.98
2.00
2.03
2.07
3.06
3.01
6.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


149.41
140.73
134.05
133.73
131.22
128.72
128.47
127.81
125.72
122.96
38.88
34.22
33.79
33.51
33.23
32.96
24.47
23.41
23.38
23.35
23.32
22.15
14.46
14.05
-0.12
Parameter Value
1 Title SBK04093B.2.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
SiMe2Ph
Pr
Pr
CF3
27
Chemical Formula: C19H29F3Si
Exact Mass: 342.1991
Molecular Weight: 342.5212
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

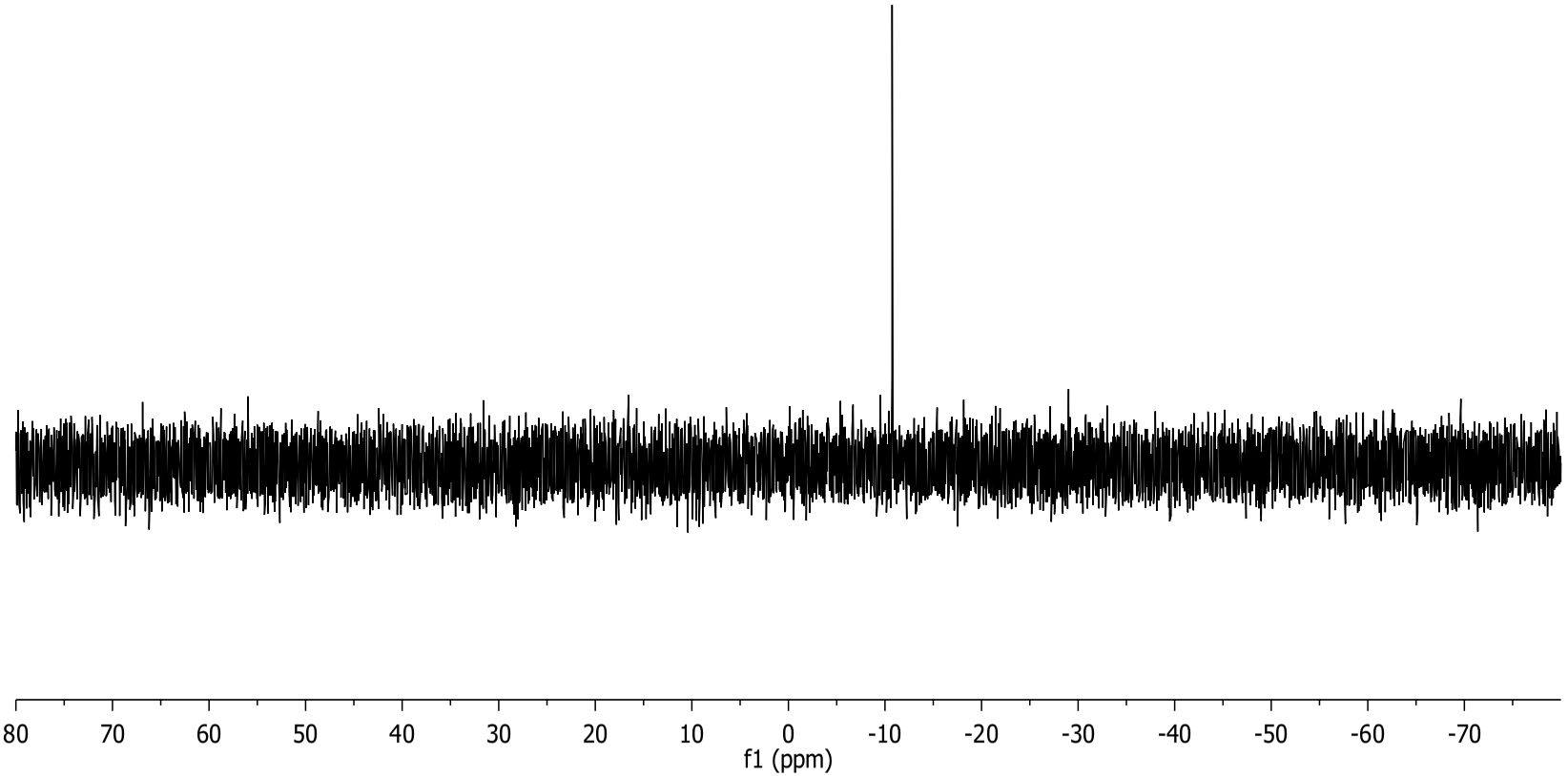
Parameter	Value
1 Title	SBK04093B-Si.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	256
5 Receiver Gain	1030.0
6 Relaxation Delay	12.5000
7 Pulse Width	13.5000
8 Spectrometer Frequency	119.26
9 Nucleus	29Si



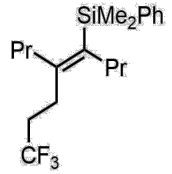
27

Chemical Formula: $C_{19}H_{29}F_3Si$
Exact Mass: 342.1991
Molecular Weight: 342.5212

-10.74

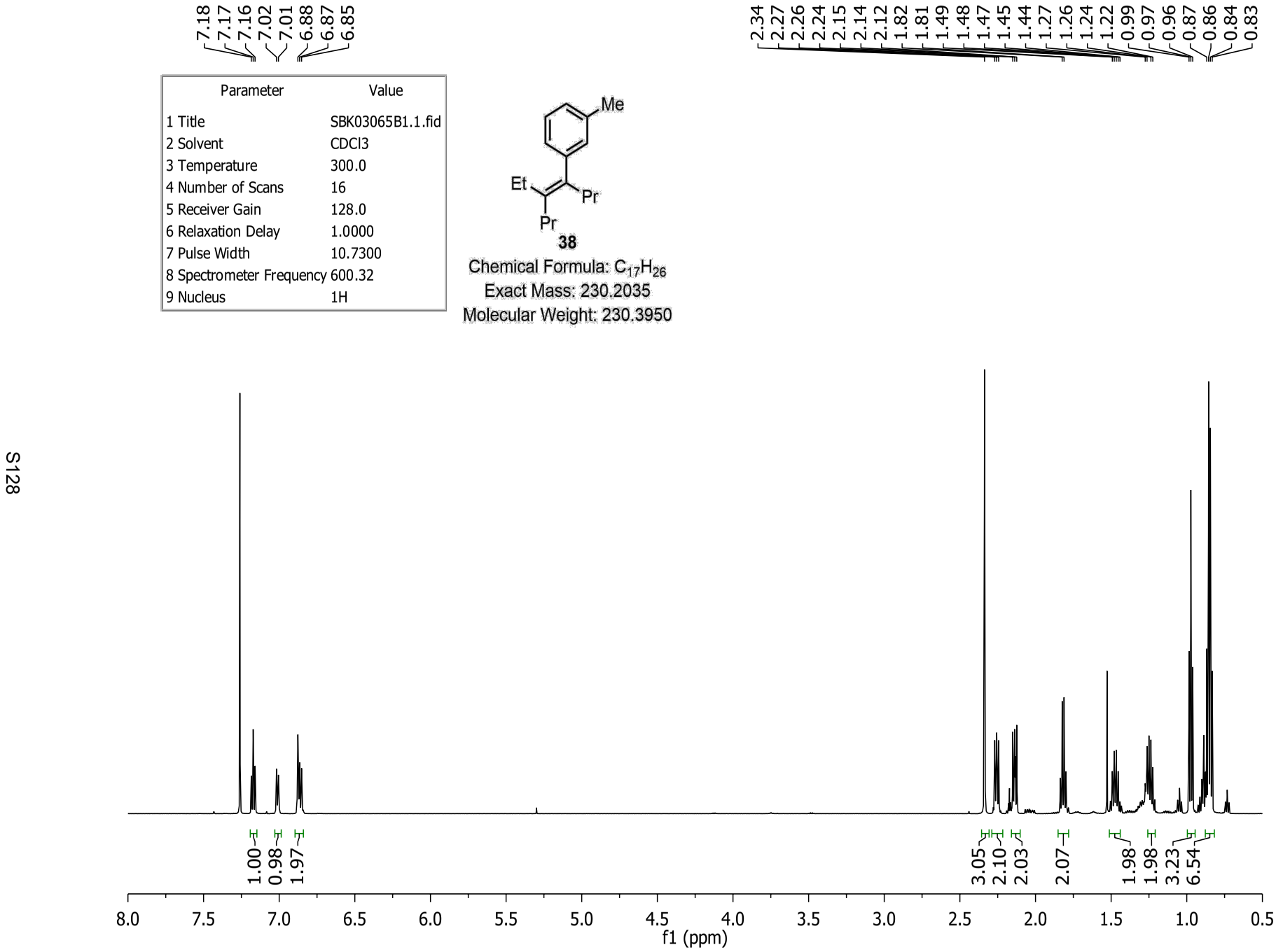


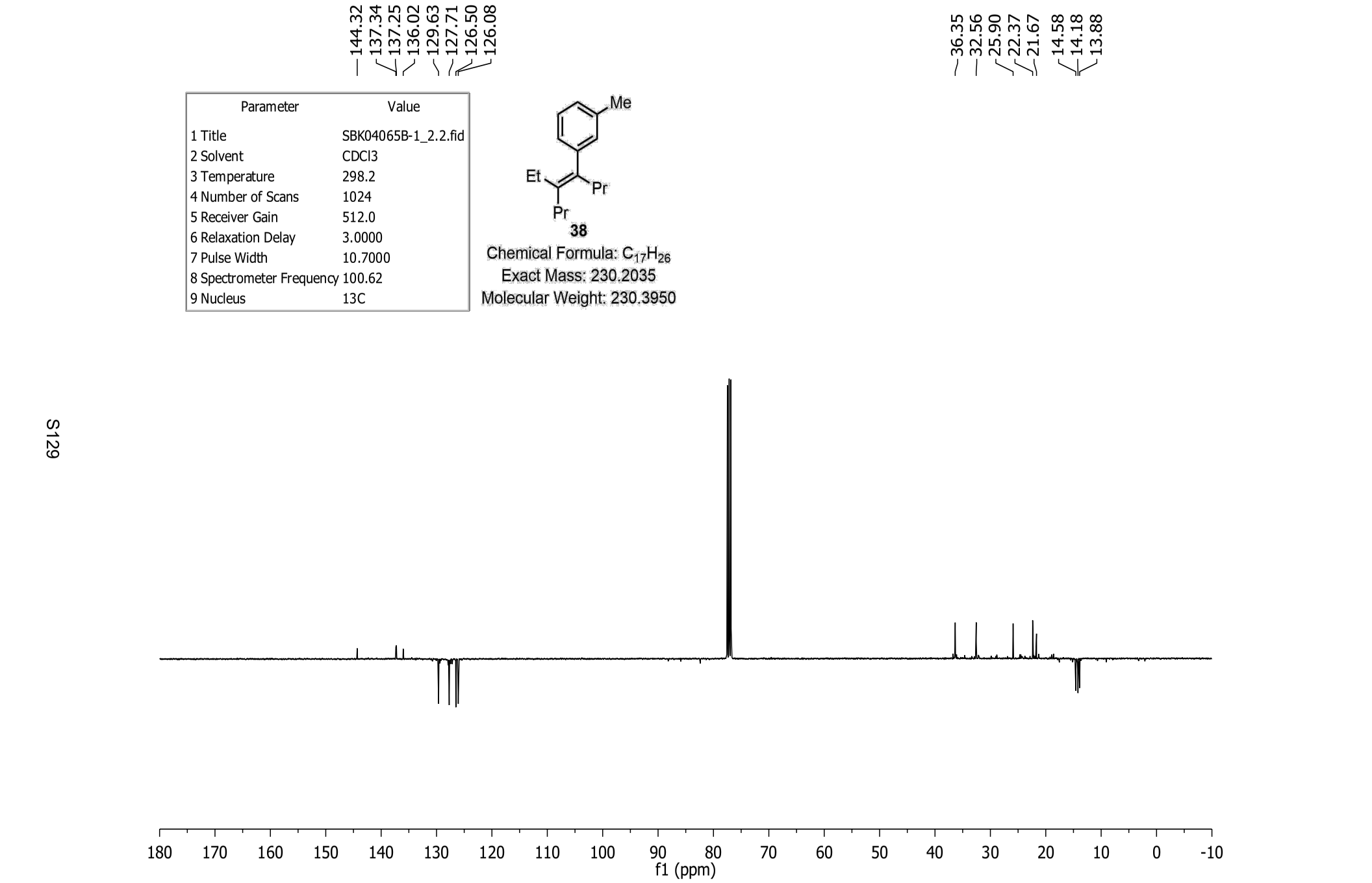
Parameter	Value
1 Title	SBK04093B-19F.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Number of Scans	16
5 Receiver Gain	322.0
6 Relaxation Delay	3.0000
7 Pulse Width	11.4000
8 Spectrometer Frequency	564.81
9 Nucleus	19F



27

Chemical Formula: $C_{19}H_{29}F_3Si$
Exact Mass: 342.1991
Molecular Weight: 342.5212


7.18
7.17
7.16
7.02
7.01
6.88
6.87
6.85
2.34
2.27
2.26
2.24
2.15
2.14
2.12
1.82
1.81
1.49
1.48
1.47
1.45
1.44
1.27
1.26
1.24
1.22
0.99
0.97
0.96
0.87
0.86
0.84
0.83
Parameter
Value
1 Title
SBK03065B1.1.fid
2 Solvent
CDCl3
3 Temperature
300.0
4 Number of Scans
16
5 Receiver Gain
128.0
6 Relaxation Delay
1.0000
7 Pulse Width
10.7300
8 Spectrometer Frequency
600.32
9 Nucleus
1H
Me
Et
Pr
Pr
38
Chemical Formula: C17H26
Exact Mass: 230.2035
Molecular Weight: 230.3950
1.00
0.98
1.97
3.05
2.10
2.03
2.07
1.98
1.98
3.23
6.54
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


144.32
137.34
137.25
136.02
129.63
127.71
126.50
126.08
36.35
32.56
25.90
22.37
21.67
14.58
14.18
13.88
Parameter Value
1 Title SBK04065B-1_2.2.fid
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
Me
Et
Pr
Pr
38
Chemical Formula: C17H26
Exact Mass: 230.2035
Molecular Weight: 230.3950
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)