Supplementary data for article:

Cinderella, A. P.; Vulovic, B.; Watson, D. A. Palladium-Catalyzed Cross-Coupling of Silyl Electrophiles with Alkylzinc Halides: A Silyl-Negishi Reaction. *Journal of the American Chemical Society* **2017**, *139* (23), 7741–7744. https://doi.org/10.1021/jacs.7b04364

Palladium-Catalyzed Cross-Coupling of Silyl Electrophiles with Alkylzinc Halides: A Silyl Negishi Reaction

Andrew P. Cinderella, Bojan Vulovic, and Donald A. Watson*

Department of Chemistry and Biochemistry, University of Delaware,
Newark, Delaware 19716, United States

Supporting Information

Index Page

1. General Experimental Details:

1,4-Dioxane, triethylamine, toluene, dichloromethane (DCM), acetonitrile (MeCN), and tetrahydrofuran (THF) were dried on alumina according to published procedures[1]. The following reagents were purchased from commercial suppliers and used as received: Tris(dibenzylideneacetone)dipalladium(0) chloroform adduct [$Pd_2(dba)_3$•$CHCl_3$] (Strem), allyl palladium(II) chloride dimer [$(allyl)PdCl]_2$ (Strem), palladium(II) iodide (Strem), palladium(II) chloride (Strem), triphenlyphosphine (Oakwood), tri-*ortho*-tolylphosphine (Strem), tri-*para*-tolylphosphine (Strem), tris(*para*-trifluoromethylphenyl)phosphine (Strem), tris(*para*-methoxyphenyl)phosphine (Alfa Aesar), 1-bromo-3,5-di-*tert*-butylbenzene (Combi Blocks), iodocyclohexane (Oakwood), 2-iodopropane (Oakwood), 1-iodo-2-methylpropane (Acros), 1-iodopropane (Acros), bromocyclopentane (Acros), 2-bromopropane (Aldrich), 1-bromo-2-methylpropane (Acros), 1-bromobutane (Acros), 3-bromopentane (Alfa Aesar), exo-2-bromonorbornane (Aldrich), 2-decanol (Acros), trimethylsilylchloride (Gelest), trimethylsilyliodide (Gelest), triethylsilyliodide (Gelest), methyldiphenylsilane (Gelest), dimethylphenylsilane (Gelest), iodomethane (Acros), zinc dust <10μm (Aldrich), 3-buten-2-ol (Matrix). Bis(3,5-di-*tert*-butylphenyl)(*tert*-butyl)phosphine (**JessePhos**)[2], (3-bromobutyl)benzene,[3] 1-(3-bromobutyl)-4-chlorobenzene,[4] and 1-(3-bromobutyl)-4-methoxybenzene,[5] were prepared according to the published procedures. 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 ^{1}H NMR with 1,3,5-trimethoxybenzene an internal standard and branched:linear (B:L) 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.

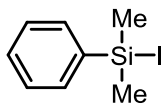
2. Instrumentation and Chromatography:

400 MHz ^{1}H, 101 MHz ^{13}C and 376 MHz ^{19}F spectra were obtained on a 400 MHz FT-NMR spectrometer equipped with a Bruker CryoPlatform. 600 MHz ^{1}H, 151 MHz ^{13}C, 119 MHz ^{29}Si, and 243 ^{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. ^{1}H 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. Product ^{29}Si spectra were calibrated using a hexamethyldisiloxane capillary standard at 7.32ppm. 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 4 Biotage unit with 40-63 μm silica gel, and the eluent reported in parentheses. Analytical thin-layer chromatography (TLC) was performed on silica gel (60 F_{254} Merck) pre-coated glass plates and visualized by UV or by staining with iodine, $KMnO_4$, or CAM.

3. Synthesis of Non-Commercial and Novel Reagents:

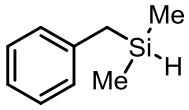
Silane Synthesis:

Silyl iodides were synthesized through a modified version of the procedure described by Kunai[6].
Note: These reactions are exothermic and generate an equivalent of methane as a byproduct.



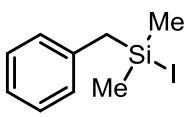
Me_2PhSiI

Dimethylphenylsilyl Iodide: An oven-dried 250 mL round bottom flask equipped with a magnetic stir bar, cold water condenser, and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the condenser was removed, and $PdCl_2$ (660 mg, 3.7 mmol, 0.01 equiv) was added. The condenser was replaced, the flask was attached to a double manifold then evacuated and backfilled with N_2 three times. The condenser was fitted with a vent tube attached to a mineral oil bubbler. A solution of dimethylphenylsilane (50.4 g, 370 mmol, 1 equiv) in iodomethane (69.1 mL, 1.11 mol, 3 equiv) was added dropwise via cannula over 1 h. The mixture was stirred 18 h at rt. The condenser was removed and the flask quickly connected to a flame-dried short-path distillation head. The excess iodomethane was removed in vacuo (30 °C/100 mm Hg) then the pressure was reduced and the product purified by fractional vacuum distillation (85 °C/5 mm Hg) to afford dimethylphenylsilyl iodide as a clear liquid that was stored over copper beads in a Schlenk tube sealed with a PTFE plug (86.2 g, 89%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 – 7.62 (m, 2H), 7.44 – 7.40 (m, 3H), 1.06 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 135.9, 133.5, 130.6, 128.2, 4.5; ^{29}Si NMR (119 MHz, $CDCl_3$) δ 0.21;. NMR spectra consistent with literature.[6b,7]



$BnMe_2SiH$

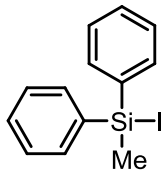
Benzyldimethylsilane: An oven-dried 1 L round bottom flask equipped with a magnetic stir bar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the septum removed, and magnesium turnings (14.6 g, 600 mmol, 1.5 equiv) and a chip of iodine were added. The septum was replaced, the flask was purged with N_2 approximately 10 minutes, then THF (200 mL) was added and the mixture stirred until clarity. The mixture was then cooled to 0 °C in an ice-water bath. A separate oven-dried 500 mL round bottom flask equipped with a rubber septum was attached to a double manifold, cooled under vacuum, backfilled with N_2, then charged with benzyl bromide (68.4g, 400 mmol, 1 equiv) and chlorodimethylsilane (37.9 g, 400 mmol, 1 equiv). This solution was then added dropwise via cannula to the magnesium mixture at 0 °C over approximately 1 h. After the addition was complete, the flask was allowed to warm to rt and stirred for 16 h. The solution was decanted away from the excess magnesium into a separatory funnel, rinsing 2X with 100 mL Et_2O. The organic layer was then washed 2X with saturated NH_4Cl solution (200 mL), dried over $MgSO_4$, filtered, and the solvent was removed in vacuo. The product was purified by fractional vacuum distillation (70 °C/15 mm Hg) to afford benzyldimethylsilane as a clear oil (49 g, 82%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.23 (t, *J* = 7.6 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 7.05 (d, *J* = 7.4 Hz, 2H), 3.96 (non, *J* = 3.5 Hz, 1H), 2.16 (d, *J* = 3.2 Hz, 2H), 0.07 (d, *J* = 3.6 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.2, 128.5, 128.3, 124.4, 24.4, -4.5; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -11.89; (cm^{-1}): 3453, 2119, 1657, 1641, 1493, 1250, 884, 698. HRMS (EI) m/z, calcd for $[C_9H_{14}Si]^+$: 150.0865; found: 150.0867. NMR spectra consistent with literature.[8]



$BnMe_2SiI$

Benzyldimethylsilyl Iodide: An oven-dried 250 mL round bottom flask equipped with a magnetic stir bar, cold water condenser, and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the condenser was removed, and $PdCl_2$ (580 mg, 3.3 mmol, 0.01 equiv) was added. The condenser was replaced, the flask was attached to a double manifold then evacuated and backfilled with N_2 three times. The condenser was fitted with a vent tube attached to a mineral oil bubbler. A solution of benzyldimethylsilane (49 g, 326 mmol, 1 equiv) in iodomethane (61 mL, 978 mmol, 3 equiv) was added dropwise via cannula over 1 h. The mixture was stirred 18 h at rt. The

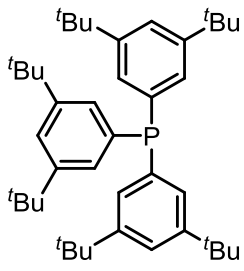
condenser was removed and the flask quickly connected to a flame-dried short-path distillation head. The excess iodomethane was removed in vacuo (30 °C/100 mm Hg) then the pressure was reduced and the product purified by fractional vacuum distillation (88 °C/3 mm Hg) to afford benzyldimethylsilyl iodide as a clear liquid that was stored over copper beads in a Schlenk tube sealed with a PTFE plug (85 g, 94%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (t, *J* = 7.6 Hz, 2H), 7.17 (t, *J* = 7.4 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 2H), 2.67 (s, 2H), 0.75 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 137.5, 128.67, 128.66, 125.4, 30.5, 3.5; ^{29}Si NMR (119 MHz, $CDCl_3$) δ 9.09. NMR spectra consistent with literature.[6b]



$MePh_2SiI$

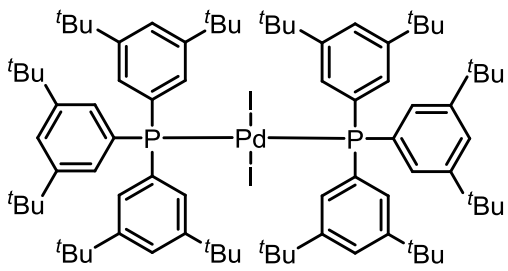
Methyldiphenylsilyl Iodide: An oven-dried 250 mL round bottom flask equipped with a magnetic stir bar, cold water condenser, and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the condenser was removed, and $PdCl_2$ (360 mg, 2 mmol, 0.01 equiv) was added. The condenser was replaced, the flask was attached to a double manifold then evacuated and backfilled with N_2 three times. The condenser was fitted with a vent tube attached to a mineral oil bubbler. A solution of methyldiphenylsilane (39.7 g, 200 mmol, 1 equiv) in iodomethane (49.8 mL, 800 mmol, 4 equiv) was added dropwise via cannula over 30 minutes. The mixture was stirred 18 h at rt. The condenser was removed and quickly replaced with a rubber septum. The excess iodomethane was removed in vacuo (30 °C/100 mm Hg). The remaining residue was then transferred via cannula to an oven dried 100 mL RBF equipped with a rubber septum and stirbar. The septum was removed and the flask quickly attached to a flame-dried short-path distillation head. The product was purified by fractional vacuum distillation (90 °C/0.1 mm Hg) to afford methyldiphenylsilyl iodide as a clear liquid that was stored over copper beads in a Schlenk tube sealed with a PTFE plug (57 g, 88%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 – 7.66 (m, 4H), 7.47 – 7.41 (m, 6H), 1.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.7, 134.2, 130.7, 128.3, 3.6; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -5.57; FTIR (cm^{-1}): 3069, 3050, 3024, 1486, 1428, 1253, 1117, 998, 799, 781, 728, 695. HRMS (LIFDI) m/z, calcd for $[C_{13}H_{13}SiI]^+$: 323.9831; found: 323.9843. NMR spectra consistent with literature.[7]

Ligand and Catalyst Synthesis:



DrewPhos

DrewPhos: An oven-dried 500 mL round bottom flask equipped with a magnetic stir bar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, 1-bromo-3,5-di-*tert*-butylbenzene (32.4 g, 120 mmol, 3.01 equiv) was added, and the septum replaced. The flask was then purged with N_2 for 15 minutes. THF (240 mL, [0.5 M]) was added and the flask was cool to –78 °C in a dry ice/acetone bath. While stirring, *n*BuLi (48.2 mL, 120 mmol, 3 equiv, [2.49 M] in hexanes) was added dropwise via syringe pump over 30 minutes. (**Note:** A large amount of solids can form and stall reaction stirring. Swirl flask by hand to break up clumps). PCl_3 (3.5 mL, 40 mmol, 1 equiv) was added dropwise via syringe pump over 15 minutes. (**Note:** Initial addition will begin to consume the aryl lithium and stirring will become easier, manual swirling may be necessary at the start of addition). After the addition was complete, the flask was warmed to 0 °C in an ice/water bath and stirred for 4 h. Flask was allowed to warm to rt, the septum was removed and the reaction was quenched by adding brine (100 mL). The reaction was poured into a separatory funnel and the product was extracted 2X with Et_2O (100 mL). The organic layer was dried over $MgSO_4$, filtered through a glass frit, and the solvent removed *in vacuo*. The product was purified by recrystallization from hot EtOH (200 mL), cooled under ambient conditions, then placed in a –20 °C freezer overnight. Collection of the solid via filtration and washing with EtOH resulted in white crystals (10.6 g, 44% yield): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 (t, *J* = 1.8 Hz, 3H), 7.12 (dd, *J* = 8.5, 1.8 Hz, 6H), 1.22 (s, 54H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 150.6 (d, *J* = 6.7 Hz), 137.3 (d, *J* = 9.4 Hz), 128.1 (d, *J* = 19.3 Hz), 122.4 , 35.0 , 31.5; ^{31}P NMR (243 MHz, $CDCl_3$) δ -3.59; FTIR (cm^{-1}): 2963, 1589, 1577, 1362, 1249, 1130, 875, 710; mp = 145–147 °C. HRMS (LIFDI) m/z, calcd for $[C_{42}H_{63}P]^+$: 598.4667; found: 598.4688.



(DrewPhos)$_2$PdI$_2$

(DrewPhos)$_2$PdI$_2$: A 50 mL round bottom flask equipped with a magnetic stirbar was charged with palladium(II) iodide (1.08 g, 3 mmol, 1.0 equiv) and DrewPhos (3.59 g, 6 mmol, 2.0 equiv). The flask was sealed with a rubber septum and purged 10 min with N_2. Toluene (24 mL) was added via syringe and the reaction was stirred for 24 hours at 85 °C. The reaction was cooled to rt, transferred to a 250 mL round bottom flask and the solvent evaporated *in vacuo*. The resulting solid was recrystallized from hot 3:1 ethanol:toluene (100 mL), cooled under ambient conditions, then placed in a –20 °C freezer overnight. Collection of the solid via filtration resulted in a stable, red solid (3.52 g, 75% yield). Subsequent recrystallization with same solvent system resulted in red crystals (900 mg, 19%). Total 4.42 g, 95%: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 – 7.54 (m, 12H), 7.29 (s, 6H), 1.21 (s, 108H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 149.2 (t, *J* = 5.1 Hz), 134.5 (t, *J* = 24.9 Hz), 129.9 (t, *J* = 6.2 Hz), 123.2, 35.1, 31.6; ^{31}P NMR (243 MHz, $CDCl_3$) δ 18.90 ; FTIR (cm^{-1}): 2953, 1589, 1384, 1247, 1087, 702, 584; mp = >250 °C. HRMS (LIFDI) m/z, calcd for $[C_{84}H_{126}P_2PdI]^+$:1429.7414; found: 1429.7373.

A small portion of **DrewPhos$_2$PdI$_2$** was dissolved in toluene under air and recrystallized via slow evaporation at rt to give an X-ray quality crystal (Figure S1, see below for full crystallographic details).

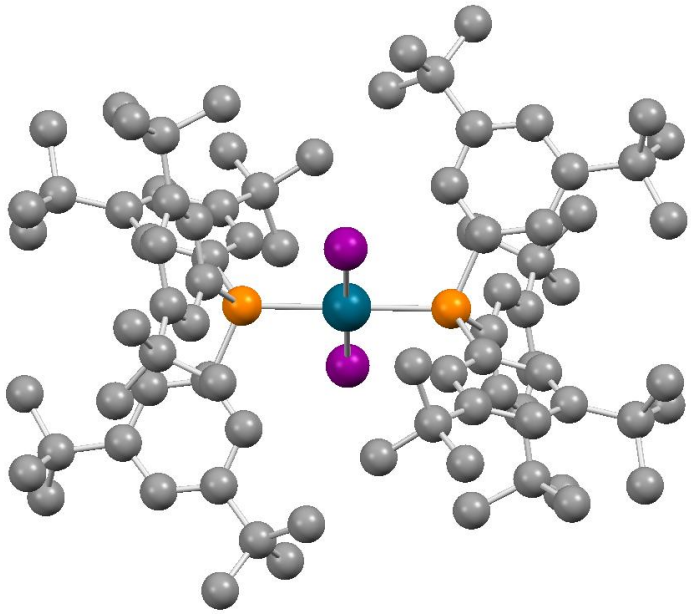
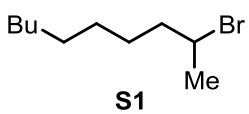


Figure S1. Crystal Structure of (**DrewPhos)$_2$PdI$_2$** (hydrogen atoms omitted for clarity).

Alkyl Halide Synthesis:



S1

(**S1**) A 500 mL round bottom flask equipped with a stirbar was charged with PPh_3 (15.7 g, 60 mmol, 1.2 equiv) and DCM (170 mL). The flask was then cooled to 0 °C in an ice-water bath. Once cool, Br_2 (9.6 g, 60 mmol, 1.2 equiv) was added dropwise via pipette. Once addition was complete, the flask was sealed with a rubber septum and purged with N_2 for approximately 10 minutes. A separate 100 mL round bottom flask was charged with 2-decanol (7.9 g, 50 mmol, 1 equiv), imidazole (4.1 g, 60 mmol, 1.2 equiv), and DCM (85 mL) then sealed with a rubber septum. The alcohol solution was then added dropwise via cannula to the 500 mL round bottom flask. After addition was complete, the flask was allowed to warm to rt and

continued stirring 20 h. The reaction was filtered through a glass frit then concentrated in vacuo to yield an orange crude mixture. Pentane (200 mL) was added and the mixture stirred 5 h then filtered through a glass frit. The filtrate was concentrated in vacuo and the product purified via short-path distillation under reduced pressure (85 °C/5 mm Hg) to yield 2-bromodecane as a clear oil (8.0 g, 73%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.13 (dq, *J* = 13.2, 6.6 Hz, 1H), 1.88 – 1.79 (m, 1H), 1.78 – 1.72 (m, 1H), 1.70 (d, *J* = 6.6 Hz, 3H), 1.53 – 1.45 (m, 1H), 1.44 – 1.35 (m, 1H), 1.33 – 1.23 (m, 10H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 52.0, 41.4, 32.0, 29.6, 29.4, 29.2, 27.9, 26.6, 22.8, 14.2; FTIR (cm^{-1}): 2956, 2926, 2855, 1457, 1378, 722, 541. HRMS (CI) m/z, calcd for $[C_{10}H_{20}Br]^+$: 219.0748; found: 219.0737.

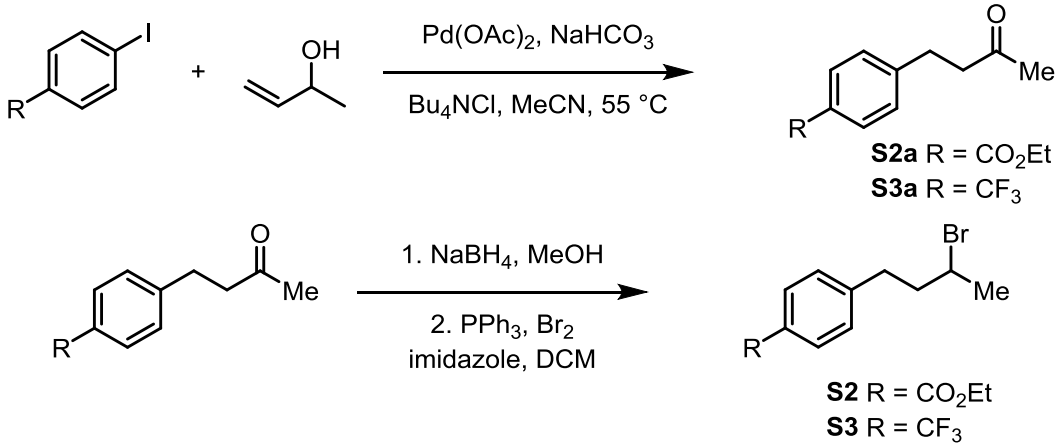
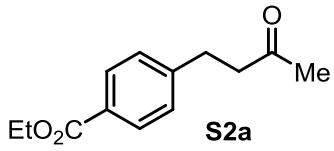


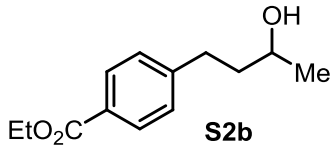
Figure S2. General route to (3-bromobutyl)arenes

Compounds **S2** and **S3** were synthesized through a Heck reaction under Jeffery conditions[9] (**S2a** and **S3a**), followed by sodium borohydride reduction[3] (**S2b** and **S3b**) and bromination[3] (**S2** and **S3**).



S2a

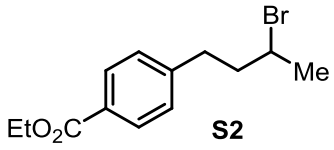
(**S2a**) An oven-dried 250 mL round bottom flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, $NaHCO_3$ (4.2 g, 50 mmol, 2.5 equiv), $Pd(OAc)_2$ (180 mg, 0.8 mmol, 0.04 equiv), tetrabutylammonium chloride (5.51 g, 20 mmol, 1 equiv,) and ethyl 4-iodobenzoate (3.40 mL, 5.58 g, 20 mmol, 1 equiv) were added. The septum was replaced, the flask was purged with N_2 for 15 minutes, and acetonitrile (100 mL, [0.2 M]) was added. 3-buten-2-ol (4.33 mL, 3.6 g, 50 mmol, 2.5 equiv) was added via syringe and the reaction mixture was stirred in an oil bath at 55 °C for 48 h. TLC indicated complete consumption of the starting aryl iodide. Silica gel was added, and volatiles removed *in vacuo*. Crude residue was purified via silica gel flash chromatography (hexanes:ethyl acetate = 4:1) to afford desired ketone **S2a** (4.04 g, 92%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 2.94 (t, *J* = 7.5 Hz, 2H), 2.78 (t, *J* = 7.5 Hz, 2H), 2.14 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 166.7, 146.5, 129.9, 128.6, 128.5, 61.0, 44.8, 30.3, 29.7, 14.5. NMR spectra consistent with literature.[10]



S2b

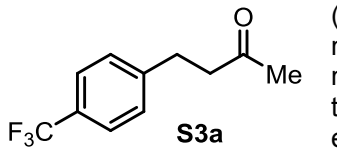
(**S2b**) A 250 mL round bottom flask equipped with a magnetic stirbar was charged with MeOH (70 mL, [0.25 M]) and ketone **S2a** (3.60 g, 16.3 mmol, 1 equiv). The solution was cooled to 0 °C in an ice-water bath. Once cool, $NaBH_4$ (940 mg, 24.8 mmol, 1.5 equiv) was added in portions with stirring. After 15 min at 0 °C TLC indicated complete consumption of the starting material. Water (50 mL) was added and methanol was removed *in vacuo*. The residue was extracted using DCM (3×40 mL). Combined organics were dried over $MgSO_4$, filtered through a glass frit, and solvent

removed *in vacuo* to afford alcohol **S2b** as a colorless viscous oil (3.5 g, 96%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 3.87 – 3.76 (m, 1H), 2.87 – 2.67 (m, 2H), 1.85 – 1.71 (m, 2H), 1.47 (s, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.23 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 147.7, 129.8, 128.5, 128.3, 67.5, 60.9, 40.6, 32.3, 23.9, 14.5; FTIR (cm^{-1}): 3420, 2968, 2930, 1717, 1611, 1416, 1368, 1279, 1107, 1022, 765. HRMS (ESI) m/z, calcd for $C_{13}H_{19}O_3^+$: 223.1329; found: 223.1329; calcd for $[C_{13}H_{17}O_2]^+$: 205.1223; found: 205.1224. NMR spectra consistent with literature.[11]



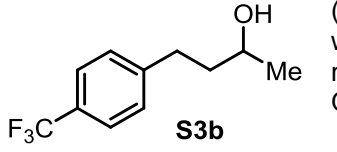
S2

(**S2**) 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 flask was backfilled with N_2, the rubber septum was removed, and PPh_3 (5.23 g, 20 mmol, 1.2 equiv) was added. The septum was replaced, the flask was purged with N_2 for 15 minutes, then DCM (35 mL) was added and the solution cooled to 0 °C in an ice-water bath. Br_2 (1.0 mL, 3.12 g, 20 mmol, 1.2 equiv) was added slowly to form an orange suspension. A separate oven-dried 100 mL round bottom flask equipped with a rubber septum was cooled under vacuum then backfilled with N_2. The septum was removed and imidazole (1.36 g, 20 mmol, 1.2 equiv) was added. The rubber septum was replaced and the flask purged with N_2 for 15 minutes. DCM (45 mL) and alcohol **S2b** (3.70 g, 16.6 mmol, 1 equiv) were added. The alcohol solution was added dropwise via cannula to the orange suspension held at 0 °C. After addition was complete, the flask was allowed to warm to rt and stirred 24 h. The reaction was quenched by addition of water (30 mL), poured into a separatory funnel, and the layers separated. The aqueous layer was extracted with DCM (30 mL), organics combined, dried over $MgSO_4$, and filtered through a glass frit. Silica gel was added and solvent removed *in vacuo*. The residue was purified via silica gel flash chromatography (hexanes:ethyl acetate = 2:1) to afford ethyl 4-(3-bromobutyl)benzoate **S2** as yellow oil (4.70 g, 90%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 4.05 (dqd, *J* = 9.0, 6.7, 4.3 Hz, 1H), 2.92 (ddd, *J* = 14.1, 9.0, 5.3 Hz, 1H), 2.80 (ddd, *J* = 13.8, 8.8, 7.3 Hz, 1H), 2.20 – 1.99 (m, 2H), 1.73 (d, *J* = 6.7 Hz, 3H), 1.39 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 146.4, 129.9, 128.64, 128.59, 61.0, 50.7, 42.3, 34.1, 26.7, 14.5; FTIR (cm^{-1}): 2981, 2926, 1713, 1611, 1444, 1415, 1366, 1275, 1178, 1105, 1021, 851, 764, 704. HRMS (CI) m/z, calcd for $[C_{13}H_{18}O_2Br]^+$: 285.0490; found: 285.0489.



S3a

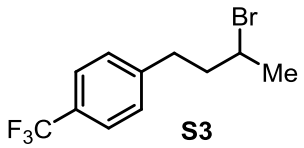
(**S3a**) An oven-dried 250 mL round bottom flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, $NaHCO_3$ (3.15 g, 37.5 mmol, 2.5 equiv), $Pd(OAc)_2$ (67 mg, 0.2 mmol, 0.04 equiv), tetrabutylammonium chloride (4.17 g, 15 mmol, 1 equiv,) and 4-iodobenzotrifluoride (2.20 mL, 4.07 g, 15 mmol, 1 equiv). The septum was replaced and the flask was then purged with N_2 for 15 minutes then acetonitrile (80 mL, [0.2 M]) was added. 3-buten-2-ol (3.25 mL, 2.7 g, 37.5 mmol, 2.5 equiv) was added via syringe and the reaction mixture was stirred in an oil bath at 55 °C for 18 h. TLC indicated complete consumption of the starting aryl iodide. Silica gel was added, and volatiles removed *in vacuo*. Crude residue was purified via silica gel flash chromatography (hexanes/ethyl acetate=7/1) to afford desired ketone **S3a** (2.60 g, 80%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.95 (t, *J* = 7.5 Hz, 2H), 2.78 (t, *J* = 7.4 Hz, 2H), 2.15 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.4, 145.3, 128.8, 128.9 (q, *J* = 32.5 Hz), 125.5 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 271.7 Hz), 44.7, 30.2, 29.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.37. FTIR (cm^{-1}): 2937, 1717, 1618, 1326, 1162, 1110, 1067, 1018, 823. HRMS (CI) m/z, calcd for $[C_{11}H_{12}OF_3]^+$: 217.0840; found: 217.0838.



S3b

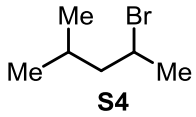
(**S3b**) A 100 mL round bottom flask equipped with a magnetic stirbar was charged with MeOH (15 mL, [0.8 M]) and ketone **S3a** (2.60 g, 12 mmol, 1 equiv). The solution was cooled to 0 °C in an ice-water bath. Once cool, $NaBH_4$ (700 mg, 18 mmol, 1.5 equiv) was added in

portions with stirring. After 15 min at 0 °C TLC indicated complete consumption of the starting material. Water (20 mL) was added and methanol was removed *in vacuo*. The residue was extracted using DCM (3×25 mL). Combined organics were dried over $MgSO_4$, filtered through a glass frit, and solvent removed *in vacuo* to afford alcohol **S3b** as a pale yellow oil (2.45 g, 94%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.83 (hept, *J* = 6.1 Hz, 1H), 2.83 (ddd, *J* = 15.3, 8.9, 6.6 Hz, 1H), 2.73 (dt, *J* = 13.8, 8.0 Hz, 1H), 1.85 – 1.70 (m, 2H), 1.43 – 1.37 (m, 1H), 1.24 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.2, 128.7, 128.2 (q, *J* = 32.5 Hz), 125.3 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 271.8 Hz), 67.3, 40.5, 32.0, 23.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.29; FTIR (cm^{-1}): 3346, 2969, 2931, 1618, 1418, 1327, 1163, 1124, 1067, 1019, 831. HRMS (CI) m/z, calcd for $[C_{11}H_{12}F_3]^+$: 201.0891; found: 201.0895.



S3

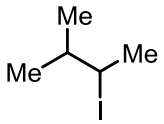
(**S3**) An oven-dried 100 mL round bottom flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, and PPh_3 (3.53 g, 13.5 mmol, 1.2 equiv) was added. The septum was replaced, the flask was purged with N_2 for 15 minutes, then DCM (25 mL) was added and the solution cooled to 0 °C in an ice-water bath. Br_2 (0.70 mL, 2.18 g, 13.5 mmol, 1.2 equiv) was added slowly to form an orange suspension. A separate oven-dried 50 mL round bottom flask equipped with a rubber septum was cooled under vacuum then backfilled with N_2. The septum was removed and imidazole (0.92 g, 13.5 mmol, 1.2 equiv) was added. The rubber septum was replaced and the flask purged with N_2 for 15 minutes. DCM (30 mL) and alcohol **S3b** (2.45 g, 11.2 mmol, 1 equiv) were added. The alcohol solution was added dropwise via cannula to the orange suspension held at 0 °C. After addition was complete, the flask was allowed to warm to rt and stirred 24 h. The reaction was quenched by addition of water (20 mL), poured into a separatory funnel, and the layers separated. The aqueous layer was extracted with DCM (20 mL), organics combined, dried over $MgSO_4$, and filtered through a glass frit. Silica gel was added and solvent removed *in vacuo*. The residue was purified via silica gel flash chromatography (hexanes:ethyl acetate = 4:1) to afford 1-(3-bromobutyl)-4-(trifluoromethyl)-benzene **S3** as a colorless oil (2.88 g, 91%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.05 (dqd, *J* = 9.1, 6.7, 4.3 Hz, 1H), 2.94 (ddd, *J* = 14.0, 9.0, 5.2 Hz, 1H), 2.81 (dt, *J* = 13.9, 7.9 Hz, 1H), 2.20 – 1.99 (m, 2H), 1.74 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.1, 129.1, 128.6 (q, *J* = 32.4 Hz), 125.6 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 271.8 Hz), 50.6, 42.4, 34.0, 26.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.34; FTIR (cm^{-1}): 2926, 1618, 1326, 1164, 1122, 1068, 1019, 839. HRMS (CI) m/z, calcd for $[C_{11}H_{12}F_3Br]^+$: 280.0074; found: 280.0063.



S4

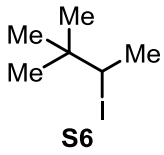
(**S4**) An oven-dried 250 mL round bottom flask equipped with a magnetic stirbar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, and PPh_3 (6.3 g, 24 mmol, 1.2 equiv) was added. The septum was replaced, the flask purged with N_2 for 15 minutes, then DCM (50 mL) was added and the solution cooled to 0 °C in an ice-water bath. Br_2 (1.23 mL, 3.84 g, 24 mmol, 1.2 equiv) was added slowly to form an orange suspension. A separate oven-dried 100 mL round bottom flask equipped with a rubber septum was cooled under vacuum then backfilled with N_2. The septum was removed and imidazole (1.64 g, 24 mmol, 1.2 equiv) was added. The rubber septum was replaced and the flask purged with N_2 for 15 minutes. DCM (40 mL) and alcohol 4-methyl-2-pentanol (2.55 mL, 2.04 g 20 mmol, 1 equiv) were added. The alcohol solution was added dropwise via cannula to the orange suspension held at 0 °C. After addition was complete, the flask was allowed to warm to rt and stirred 16 h. The reaction was quenched by addition of water (30 mL), poured into a separatory funnel, and the layers separated. The aqueous layer was extracted with DCM (30 mL), organics combined, dried over $MgSO_4$, filtered through a glass frit, and solvent removed *in vacuo*. The residue was purified via silica gel flash chromatography (pentane) then vacuum distillation (40 °C/30 mm Hg) to afford 2-bromo-4-methylpentane **S4** as a colorless oil (2.6 g, 78%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.22 – 4.14 (m, 1H), 1.88 – 1.78 (m, 2H), 1.71 (d, *J* = 6.6 Hz, 3H), 1.54 – 1.46 (m, 1H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.90 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 50.38, 50.36, 27.0, 26.8, 22.8, 21.5; FTIR (cm^{-1}): 2960, 2924, 2872,

1467, 1259, 1199, 1150. HRMS (CI) m/z, calcd for $[C_6H_{12}Br]^+$: 163.0122; found: 163.0131. NMR spectra consistent with literature.[12]



S5

(**S5**) Synthesized from a literature procedure for another compound[13]. An oven-dried 250 mL round bottom flask equipped with a magnetic stirbar and rubber septum was cooled under vacuum. The flask was backfilled with N_2, the rubber septum was removed, PPh_3 (3.82 g, 14.6 mmol, 2.7 equiv), and imidazole (1.4 g, 20.5 mmol, 3.8 equiv) were added. The septum was replaced, the flask was purged with N_2 for 15 minutes, then THF (60 mL) and 3-methyl-2-butanol (0.60 mL, 490 mg, 5.4 mmol, 1 equiv) were added. The solution was cooled to 0 °C in an ice-water bath. The septum was quickly remover, I_2 (3.56 g, 14.0 mmol, 2.6 equiv) was added in one portion, and the septum replaced. The flask continued stirring at 0 °C for 2 h then was allowed to warm to rt and stirred for 2 h. The reaction was quenched by the addition of saturated $Na_2S_2O_3 \cdot 5H_2O$ (150 mL), poured into a separatory funnel, and the layers separated. The aqueous layer was extracted using diethyl ether (2 X 100mL), combined organic layers were washed with water (100 mL) and brine (100 mL), then dried over $MgSO_4$ and filtered through a glass frit. The solvent was carefully removed *in vacuo*. The crude residue was purified via silica gel flash chromatography (pentane) then distillation under reduced pressure (22 °C/10 mm Hg, **note:** product decomposed if heated over 30–40 °C) to afford 2-iodo-3-methylbutane **S5** as a pale pink oil (446 mg, 42%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.28 (qd, *J* = 7.0, 3.8 Hz, 1H), 1.89 (d, *J* = 7.0 Hz, 3H), 1.24 (dtd, *J* = 13.0, 6.5, 3.8 Hz, 1H), 0.97 (d, *J* = 6.5 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 42.4, 36.7, 26.3, 22.5, 20.2; FTIR (cm^{-1}): 2968, 2927, 2871, 1457, 1386, 1376, 1184, 1138, 767, 577. HRMS (CI) m/z, calcd for $[C_5H_{10}I]^+$: 196.9827; found: 196.9827.



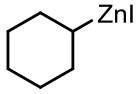
S6

(**S6**) 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 flask was backfilled with N_2, the rubber septum was removed, PPh_3 (4.53 g, 17.5 mmol, 1.2 equiv), and imidazole (1.18 g, 17.5 mmol, 1.2 equiv) were added. The septum was replaced, the flask was purged with N_2 for 15 minutes, then DCM (70 mL) was added and the solution cooled to 0 °C in an ice-water bath. The septum was quickly removed, I_2 (4.45 g, 17.5 mmol, 1.2 equiv) was added in one portion, and the septum replaced. The flask was then allowed to warm to rt and stirred for 48 h. The reaction was quenched by addition of water (25 mL), poured into a separatory funnel, and the layers separated. The aqueous layer was extracted with DCM (20 mL), organics combined, dried over $MgSO_4$, and filtered through a glass frit. The solvent was carefully removed *in vacuo*. The crude residue was purified via silica gel flash chromatography (hexanes:diethyl ether = 9:1) to afford 3-iodo-2,2-dimethylbutane **S6** as a pale yellow oil (1.31 g, 44%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 (q, *J* = 7.0 Hz, 1H), 1.92 (d, *J* = 7.0 Hz, 3H), 1.07 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 48.1, 36.1, 28.0, 24.9; FTIR (cm^{-1}): 2918, 2868, 1384, 1086. HRMS (CI) m/z, calcd for $[C_6H_{13}I]^+$: 212.0062; found: 212.0057.

4. Synthesis of Alkyl Zinc Halides:

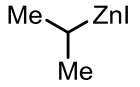
General Procedure A:

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) added. The septum was replaced; 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 rt under vacuum. The flask was backfilled with N_2 then dioxane [2 M], trimethylsilyl chloride (0.03 equiv), and alkyl bromide (1 equiv) were 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 filtered via cannula to a Schlenk tube. If insoluble particles persist, filtration through a 0.2 µm PTFE syringe filter was employed. Solutions were then titrated according to the literature procedure by Knochel.[14]



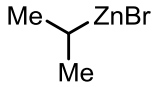
S4

(**S4**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (115 µL, 98 mg, 0.9 mmol), and cyclohexyl iodide (3.88 mL, 6.3 g, 30 mmol). The flask was heated to 100 °C for 12 h. Filtration and titration resulted in a [0.97 M] solution of cyclohexylzinc iodide in dioxane.



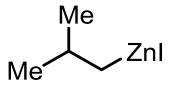
S5

(**S5**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (115 µL, 98 mg, 0.9 mmol), and isopropyl iodide (3.0 mL, 5.1 g, 30 mmol). The flask was heated to 100 °C for 20 h. Filtration and titration resulted in a [1.56 M] solution of isopropylzinc iodide in dioxane.



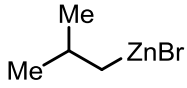
S6

(**S6**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (120 µL, 102 mg, 0.9 mmol), and isopropyl bromide (2.9 mL, 3.8 g, 31 mmol). The flask was heated to 100 °C for 20 h. Filtration and titration resulted in a [1.81 M] solution of isopropylzinc bromide.



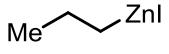
S7

(**S7**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (115 µL, 98 mg, 0.9 mmol), and isobutyl iodide (3.6 mL, 5.8 g, 30 mmol). The flask was heated to 100 °C for 17 h. Filtration and titration resulted in a [1.59 M] solution of isobutylzinc iodide in dioxane.



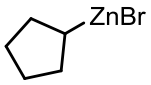
S8

(**S8**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (115 µL, 98 mg, 0.9 mmol), and isobutyl bromide (3.3 mL, 4.2 g, 30 mmol). The flask was heated to 100 °C for 17 h. Filtration and titration resulted in a [1.40 M] solution of isobutylzinc bromide in dioxane.



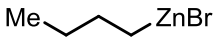
S9

(**S9**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (1.52 g, 23 mmol), dioxane (6 mL), trimethylsilyl chloride (50 µL, 45 mg, 0.4 mmol), and *n*-propyl iodide (1.5 mL, 2.61 g, 15 mmol). The flask was heated to 100 °C for 20 h. Filtration and titration resulted in a [2.25 M] solution of *n*-propylzinc iodide in dioxane.



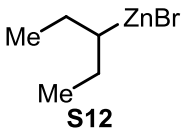
S10

(**S10**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), trimethylsilyl chloride (80 µL, 66 mg 0.6 mmol), and cyclopentyl bromide (2.2 mL, 20 mmol). The flask was heated to 100 °C for 18 h. Filtration and titration resulted in a [0.89 M] solution of cyclopentylzinc bromide.



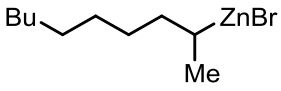
S11

(**S11**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (3.92 g, 60 mmol), dioxane (15 mL), trimethylsilyl chloride (115 µL, 98 mg, 0.9 mmol), and *n*-butyl bromide (3.3 mL, 4.2 g, 30 mmol). The flask was heated to 100 °C for 17 h. Filtration and titration resulted in a [1.51 M] solution of *n*-butylzinc bromide in dioxane.



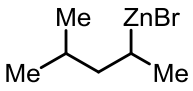
S12

(**S12**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), trimethylsilyl chloride (80 µL, 66 mg 60 µmol), and 3-bromopentane (2.5 mL,3.0 g, 20 mmol). The flask was heated to 100 °C for 4 h. Filtration and titration resulted in a [1.35 M] solution of pentan-3-ylzinc bromide.



S13

(**S13**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (2.1 g, 32 mmol), dioxane (8 mL), trimethylsilyl chloride (60 µL, 52 mg, 0.5 mmol), and 2-bromodecane (3.4 mL, 16 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.00 M] solution of 1-octylethylzinc bromide.

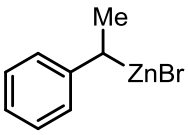


S14

(**S14**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (1.83 g, 28 mmol), dioxane (7 mL), trimethylsilyl chloride (50 µL, 43 mg, 0.4 mmol), and 2-bromo-4-methylpentane (2.29 g, 14 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [0.91 M] solution of (4-methylpentan-2-yl)zinc bromide in dioxane.

S15

(**S15**) According to general procedure A, a 25 mL Schlenk flask was charged with zinc dust (2.6 g, 40 mmol), dioxane (10 mL), trimethylsilyl chloride (80 µL, 65 mg, 0.6 mmol), and 2-exo-bromonorbornane (2.6 mL, 3.5 g, 20 mmol). The flask was heated to 100 °C for 3 h. Filtration and titration resulted in a [1.23 M] solution of (1*S*,4*R*)-bicyclo[2.2.1]heptan-2-ylzinc bromide.



S16

(**S16**) A 50 mL Schlenk flask equipped with a stirbar and rubber septum attached to a double manifold (with cold trap) was charged with titrated, [0.38 M] α-methylbenzylzinc bromide (Aldrich) in THF (25 mL, 9.5 mmol) and dioxane (8 mL). Solvent was removed in vacuo (26 °C/0.3 mm Hg) until total volume was reduced to approx. 2-3 mL (solution became viscous). Dioxane (12 mL) was added and solvents were removed again in vacuo (26 °C/0.2 mm Hg) until total volume was reduced to approx. 2-3 mL (viscous solution). Dioxane (8 mL) was added to afford a total volume of approximately 10 mL. Filtration via cannula to a Schlenk bomb and titration resulted in a [0.82 M] solution of α-methylbenzylzinc bromide in dioxane. See Figure S3 for solvent exchange setup diagram.

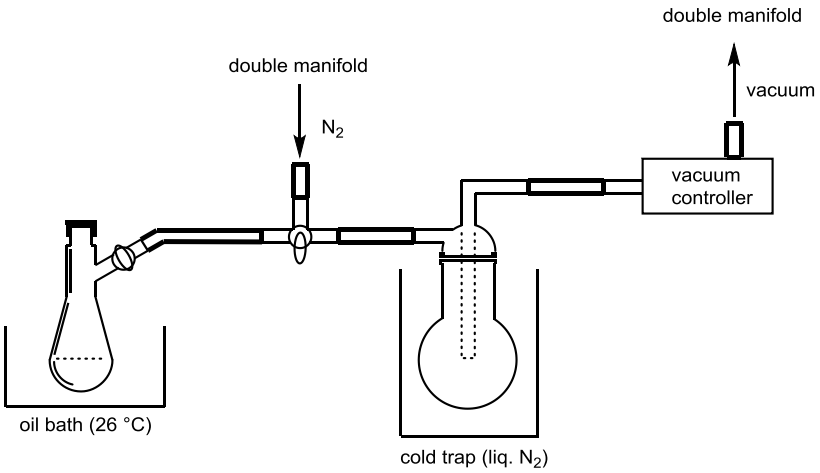
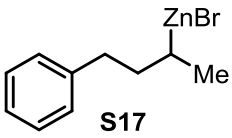
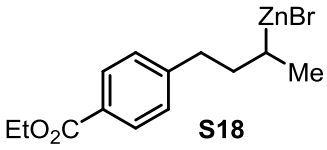


Figure S3: Experimental setup for solvent exchange



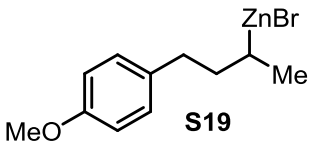
S17

(**S17**) According to general procedure A, a 10 mL Schlenk flask was charged with zinc dust (1.3 g, 20 mmol), dioxane (5 mL), trimethylsilyl chloride (40 µL, 33 mg 0.3 mmol), and 2-bromo-4-phenylbutane (2.1 g, 10 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.32 M] solution of (4-phenylbutan-2-yl)zinc bromide.



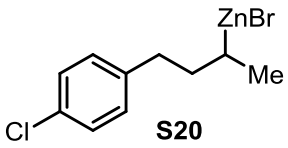
S18

(**S18**) According to general procedure A, a 10 mL Schlenk flask was charged with zinc dust (1.3 g, 20 mmol), dioxane (6 mL), trimethylsilyl chloride (100 µL, 86 mg, 0.8 mmol), and ethyl 4-(3-bromobutyl)benzoate (2.85 g, 10 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.09 M] solution of (4-(4-(ethoxycarbonyl)phenyl)butan-2yl)zinc bromide in dioxane.



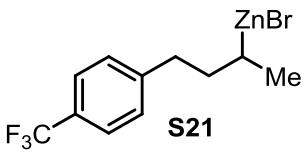
S19

(**S19**) According to general procedure A, a 10 mL Schlenk flask was charged with zinc dust (1.3 g, 20 mmol), dioxane (5 mL), trimethylsilyl chloride (40 µL, 33 mg, 0.3 mmol), and 1-(3-bromobutyl)-4-methoxybenzene (2.4 g, 10 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.26 M] solution of (4-(4-methoxyphenyl)butan-2yl)zinc bromide.



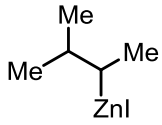
S20

(**S20**) According to general procedure A, a 10 mL Schlenk flask was charged with zinc dust (1.3 g, 20 mmol), dioxane (5 mL), trimethylsilyl chloride (40 µL, 33 mg, 0.3 mmol), and 1-(3-bromobutyl)-4-chlorobenzene (2.5 g, 10 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.25 M] solution of (4-(4-chlorophenyl)butan-2-yl)zinc bromide.



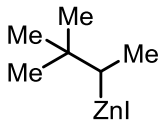
S21

(**S21**) According to general procedure A, a 10 mL Schlenk flask was charged with zinc dust (1.3 g, 20 mmol), dioxane (5 mL), trimethylsilyl chloride (40 µL, 33 mg, 0.3 mmol), and 1-(3-bromobutyl)-4-(trifluoromethyl)benzene (2.82 g, 10 mmol). The flask was heated to 100 °C for 2 h. Filtration and titration resulted in a [1.14 M] solution of (4-(4-(trifluoromethyl)phenyl)butan-2yl)zinc bromide in dioxane.



21

(**21**) According to a modified general procedure A, a 10 mL Schlenk flask was charged with zinc dust (294 mg, 4.5 mmol), dioxane (1 mL), trimethylsilyl chloride (20 µL, 17 mg, 0.1 mmol), and 2-iodo-3-methylbutane (446 mg, 2.3 mmol) was added dissolved in dry dioxane (1.1 mL). The flask was heated to 50 °C for 1 h. Filtration and titration resulted in a [1.05 M] solution of (3-methylbutan-2-yl)zinc iodide.



24

(**24**) According to a modified general procedure A, a 10 mL Schlenk flask was charged with zinc dust (0.8 g, 12 mmol), dioxane (5 mL), trimethylsilyl chloride (50 µL, 43 mg, 0.4 mmol), and 3-iodo-2,2-dimethylbutane (1.31 g, 6 mmol). The flask was heated to 50 °C for 2 h. Filtration and titration resulted in a [0.54 M] solution of (3,3-dimethylbutan-2-yl)zinc iodide in dioxane.

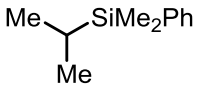
5. General Procedure for the Silyl-Negishi Reaction:

General Procedure B:

Note: Reactions are run at [0.5 M] overall concentration based on the sum of all liquid reagents.

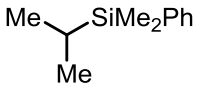
Note: THF quenches were performed for certain substrates due to inseparable disiloxane formed upon aqueous workup. This quench generates the more easily separated (4-iodobutoxy)silane through silyl iodide induced ring opening of THF.

An oven dried 10 mL 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 rubber septum was removed, and $(DrewPhos)_2PdI_2$ (0.01 equiv) was added. The septum was replaced and the flask purged with N_2 for 10 minutes. Dioxane, triethylamine (1 equiv), silyl iodide (2 equiv), and alkylzinc bromide (1 equiv) were added via syringe. The flask was then stirred at rt for the indicated time. The reaction was quenched as indicated, diluted with Et_2O (20 mL) or EtOAc (20 mL) then washed 2 times with brine (20 mL). The organic layer was dried over $MgSO_4$, filtered, and the solvent removed *in vacuo*. The crude material was purified via silica gel flash chromatography in the indicated solvent.



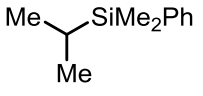
1

(**1**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 µmol), dioxane (820 µL), triethylamine (140 µL, 1 mmol), dimethylphenylsilyl iodide (360 µL, 2 mmol), and [1.56 M] isopropylzinc iodide **S5** (640 µL, 1 mmol) were combined under N_2 and stirred at rt for 1 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **1** as a clear volatile oil (165.1 mg, 93%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.45 (m, 2H), 7.44 – 7.31 (m, 3H), 1.02 – 0.92 (m, 7H), 0.25 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.7, 134.1, 128.9, 127.7, 17.7, 13.9, -5.2; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.40; FTIR (cm^{-1}): 2955, 2864, 1463, 1427, 1248, 1112, 882, 831, 812, 770, 733, 699. HRMS (CI) m/z, calcd for $[C_{11}H_{18}Si]^+$: 178.1178; found: 178.1179.



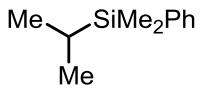
1

(**1**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 µmol), dioxane (940 µL), triethylamine (140 µL, 1 mmol), dimethylphenylsilyl iodide (360 µL, 2 mmol), and [1.79 M] isopropylzinc bromide **S6** (560 µL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **1** as a clear volatile oil (160.5 mg, 90%). NMR spectra matched previous isolation: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.45 (m, 2H), 7.40 – 7.32 (m, 3H), 1.07 – 0.86 (m, 7H), 0.25 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.7, 134.1, 128.9, 127.7, 17.7, 13.9, -5.2.



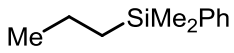
1

(**1**) A two dram vial with stirbar (open to air) was charged with $(DrewPhos)_2PdI_2$ (16 mg, 10 µmol), dioxane (910 µL), triethylamine (140 µL, 1 mmol) and dimethylphenylsilyl iodide (360 µL, 2 mmol). The vial was sealed with a Teflon lined cap. While stirring, the cap was removed, [1.70 M] solution isopropylzinc iodide **S5** (590 µL, 1 mmol) was added via syringe, and the cap was replaced. Stirring at rt was continued for 1 h. The reaction was quenched by removing the cap and adding wet EtOAc (0.5 mL) and brine (3 mL) via syringe and then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **1** as a clear volatile oil (156.1 mg, 88%). NMR spectra matched previous isolations: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.49 (m, 2H), 7.36-7.35 (m, 3H), 0.99-0.94 (m, 7H), 0.25 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.6, 133.9, 128.7, 127.6, 17.6, 13.8, -5.3.



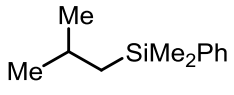
1

(**1**) According to general procedure B, $(DrewPhos)_2PdI_2$ (390 mg, 0.25 mmol), dioxane (21 mL), triethylamine (3.5 mL, 25 mmol), dimethylphenylsilyl iodide (9 mL, 50 mmol), and [1.52 M] isopropylzinc bromide **S6** (16.5 mL, 25 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with THF (10 mL), stirred for 15 min then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **1** as a clear volatile oil (4.35 g, 98%). NMR spectra matched previous isolation: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.49 (m, 2H), 7.37 – 7.33 (m, 3H), 0.97 – 0.94 (m, 7H), 0.25 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.8, 134.1, 128.9, 127.7, 17.7, 13.9, -5.2.



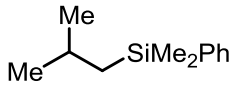
2

(**2**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (1.00 mL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [2.25 M] *n*-propylzinc bromide **S9** (440 μL, 1 mmol) were combined under N_2 and stirred at rt for 1 h. . The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **2** as a clear very volatile oil (169.5 mg, 96%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.49 (m, 2H), 7.39 – 7.31 (m, 3H), 1.42 – 1.31 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H), 0.79 – 0.72 (m, 2H), 0.26 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 133.7, 128.9, 127.8, 18.51, 18.48, 17.6, -2.8; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -3.37; FTIR (cm^{-1}): 2955, 2868, 1427, 1248, 1114, 1065, 997, 882, 834, 767, 727, 699. HRMS (CI) m/z, calcd for $[C_{10}H_{15}Si]^+$: 163.0943; found: 163.0941.



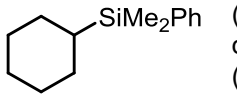
3

(**3**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (900 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.59 M] isobutylzinc iodide **S7** (640 μL, 1 mmol) were combined under N_2 and stirred at rt for 1 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **3** as a clear volatile oil (187.0 mg, 95%). NMR spectra matched previous isolation: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.44 (m, 2H), 7.43 – 7.31 (m, 3H), 1.77 (dh, *J* = 13.3, 6.6 Hz, 1H), 0.90 (d, *J* = 6.6 Hz, 6H), 0.77 (d, *J* = 6.9 Hz, 2H), 0.29 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.4, 133.7, 128.8, 127.8, 26.50, 26.48, 25.1, -1.9.



3

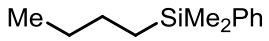
(**3**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (800 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.34 M] isobutylzinc bromide **S8** (750 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **3** as a clear volatile oil (183.3 mg, 95%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 2H), 7.38 – 7.32 (m, 3H), 1.77 (dh, *J* = 13.3, 6.7 Hz, 1H), 0.91 (d, *J* = 6.6 Hz, 6H), 0.78 (d, *J* = 6.9 Hz, 2H), 0.29 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.3, 133.7, 128.8, 127.8, 26.50, 26.48, 25.1, -1.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -4.17; FTIR (cm^{-1}): 2953, 2893, 2361, 2338, 1248, 1112, 838, 812, 790, 698. HRMS (CI) m/z, calcd for $[C_{12}H_{19}Si]^+$: 191.1256; found: 191.1253.



4

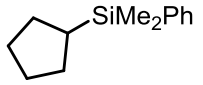
(**4**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (440 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [0.97 M] cyclohexylzinc bromide **S4** (1.00 mL, 0.97 mmol) were combined under N_2 and stirred at rt for 1 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **4** as a clear oil (205.6 mg, 97%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 2H), 7.40 – 7.32 (m, 3H), 1.75 – 1.61 (m, 5H), 1.27 – 1.02 (m, 5H), 0.84 – 0.73 (m, 1H), 0.24 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.8, 134.1, 128.8, 127.7, 28.2, 27.5, 27.0, 25.9, -5.1; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -2.20; FTIR (cm^{-1}): 2919, 2846, 1446, 1427, 1247, 1112, 850, 834, 818, 770, 699.

HRMS (CI) m/z, calcd for $[C_{14}H_{22}Si]^+$: 218.1491; found: 218.1486. NMR spectra consistent with literature.[15]



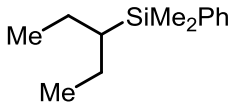
5

(**5**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (900 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.60 M] *n*-butylzinc bromide **S11** (630 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **5** as a clear oil (175.1 mg, 91%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 2H), 7.39 – 7.33 (m, 3H), 1.37 – 1.26 (m, 4H), 0.87 (t, *J* = 6.9 Hz, 3H), 0.80 – 0.73 (m, 2H), 0.26 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 133.7, 128.9, 127.8, 26.7, 26.2, 15.6, 13.9, -2.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -3.08; FTIR (cm^{-1}): 2956, 2922, 2872, 1427, 1248, 1113, 887, 837, 779, 727, 699. HRMS (CI) m/z, calcd for $[C_{11}H_{17}Si]^+$: 177.1100; found: 177.1102. NMR spectra consistent with literature.[16]



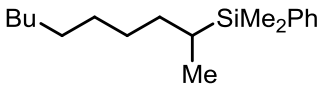
6

(**6**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (380 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [0.89 M] cyclopentylzinc bromide **S10** (1.1 mL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **6** as a clear oil (197 mg, 97%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 2H), 7.36 – 7.33 (m, 3H), 1.80 – 1.73 (m, 2H), 1.59 – 1.46 (m, 4H), 1.36 – 1.25 (m, 2H), 1.11 (tt, *J* = 8.4 Hz, 1H), 0.25 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.4, 133.9, 128.7, 127.6, 28.2, 27.0, 25.5, -4.5; ^{29}Si NMR (119 MHz, $CDCl_3$) δ –2.01; FTIR (cm^{-1}): 3068, 2950, 2862, 1427, 1248, 1114, 827, 811, 699, 415. HRMS (CI) m/z, calcd for $[C_{13}H_{20}Si]^+$: 204.1334; found: 204.1337.



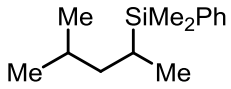
7

(**7**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (760 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.35 M] pentan-3-ylzinc bromide **S12** (740 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **7** as a clear oil (192 mg, 93%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 – 7.49 (m, 2H), 7.36 – 7.32 (m, 3H), 1.57 – 1.47 (m, 2H), 1.36 (dp, *J* = 14.7, 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 6H), 0.74 – 0.68 (m, 1H), 0.28 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.7, 133.8, 128.6, 127.6, 28.9, 21.7, 13.7, -3.6; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -1.04; FTIR (cm^{-1}):3069, 2959, 2871, 1427, 1248, 1029, 831, 810, 700, 471. HRMS (CI) m/z, calcd for $[C_{13}H_{22}Si]^+$: 206.1491; found: 206.1495.



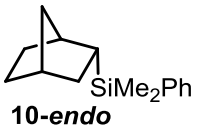
8

(**8**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (500 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.00 M] 1-octylethylzinc bromide **S13** (1 mL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with dry THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **8** as a clear oil (261 mg, 94%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 – 7.48 (m, 2H), 7.36 – 7.31 (m, 3H), 1.49 – 1.36 (m, 2H), 1.33 – 1.20 (m, 9H), 1.19 – 1.13 (m, 2H), 1.13 – 1.06 (m, 1H), 0.92 (d, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 7.1 Hz, 3H), 0.86 – 0.80 (m, 1H), 0.24 (d, *J* = 2.6 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.1, 134.1, 128.8, 127.7, 32.1, 31.7, 29.8, 29.7, 29.5, 28.7, 22.8, 19.2, 14.3, 14.2, -4.7; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.17; FTIR (cm^{-1}): 3069, 2955, 2924, 2853, 1466, 1427, 1248, 1112, 832, 813, 770, 733, 700. HRMS (CI) m/z, calcd for $[C_{18}H_{31}Si]^+$: 275.2195; found: 275.2206.



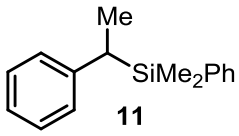
9

(**9**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (400 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [0.91 M] (4-methylpentan-2-yl)zinc bromide **S14** in dioxane (1.10 mL, 1 mmol) were combined under N_2 and stirred at rt for 15 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **9** as a clear volatile oil (170.1 mg, 77%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.52 – 7.49 (m, 2H), 7.37 – 7.33 (m, 3H), 1.67 (ttd, *J* = 13.2, 6.6, 4.4 Hz, 1H), 1.16 (ddd, *J* = 13.4, 9.7, 3.6 Hz, 1H), 1.09 (ddd, *J* = 13.5, 10.8, 4.4 Hz, 1H), 1.00 – 0.94 (m, 1H), 0.90 (d, *J* = 7.0 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H), 0.79 (d, *J* = 6.5 Hz, 3H), 0.25 (s, 3H), 0.24 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.9, 134.1, 128.8, 127.7, 40.9, 25.5, 24.1, 21.1, 16.5, 14.0, -4.8, -4.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ 0.16; FTIR (cm^{-1}): 2954, 2900, 2867, 1248, 1112, 830, 767, 733, 699. HRMS (CI) m/z, calcd for $[C_{13}H_{21}Si]^+$: 205.1413; found: 205.1419.

10-*exo* 80:20 

10-*endo*

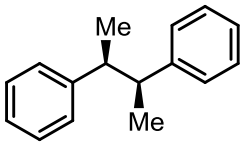
(**10**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (690 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.23 M] (1*S*,4*R*)-bicyclo[2.2.1]heptan-2ylzinc bromide **S15** (810 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **10** as an inseparable mixture of *exo*:*endo* (80:20) diastereomers as a clear oil (230 mg, 99%): Useful diagnostic peaks for each compound are listed, mixture matches previously reported spectra.[5,17] **10-*exo***: ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.21 (dd, *J* = 3.7, 2.0 Hz, 2H), 1.06 – 1.05 (m, 2H), 0.84 – 0.78 (m, 1H), 0.24 (s, 3H), 0.22 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.6, 134.1, 128.8, 127.8, 38.1, 37.9, 37.1, 34.5, 32.9, 29.1, 28.8, -3.9, -3.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -3.23. **10-*endo***: ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.33 – 2.25 (m, 2H), 1.78 – 1.69 (m, 1H), 1.03 – 0.98 (m, 1H), 0.31 (s, 3H), 0.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.4, 133.9, 128.7, 127.8, 41.9, 39.8, 37.3, 32.0, 30.0, 28.6, 27.6, -2.7, -2.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -2.81.



11

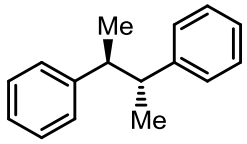
(**11**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (260 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [0.80 M] α-methylbenzylzinc bromide **S16** in dioxane (1.25 mL, 1 mmol) were combined under N_2 and stirred at rt for 16 h. The reaction was quenched with THF (0.4 mL), stirred for 15 min, and then wet EtOAc (0.5 mL) and brine (3 mL) were added via syringe and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford 233 mg of mixture: **11**, DL-2,3-diphenylbutane, *meso*-2,3-diphenylbutane, and $(PhMe_2Si)_2O$ in a relative ratio 78:8:5:9. This mixture was purified by reverse phase chromatography on Biotage instrument using SNAP Ultra C18 120 g column (50:50 MeCN:H_2O to 80:20 MeCN:H_2O linear gradient) to obtain **11** as a colorless oil (171.1 mg, 71%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 – 7.29 (m, 5H), 7.20 (t, *J* = 7.7 Hz, 2H), 7.09 (t, *J* = 7.3 Hz, 1H), 6.95 (d, *J* = 7.3 Hz, 2H), 2.39 (q, *J* = 7.5 Hz, 1H), 1.35 (d, *J* = 7.5 Hz, 3H), 0.25 (s, 3H), 0.21 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 145.4, 137.8, 134.3, 129.1, 128.1, 127.7, 127.5, 124.6, 29.7, 15.3, -4.2, -5.3; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -1.06; FTIR (cm^{-1}): 3023, 2957, 2870, 1495, 1450, 1427, 1248, 1112, 833, 817, 775, 735, 699. HRMS (CI) m/z, calcd for $[C_{16}H_{20}Si]^+$: 240.1334; found: 240.1345.

Notes: DL- and *meso*-2,3-diphenylbutane were present as impurities in purchased α-methylbenzylzinc bromide reagent, and their formation was not observed during the Pd coupling reaction. Reference spectra after reverse phase separation confirming their identity have been provided and match literature reports.[18]



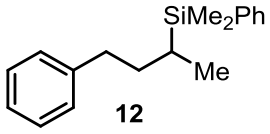
DL-2,3-diphenylbutane

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 – 7.15 (m, 4H), 7.10 – 7.07 (m, 2H), 7.02 – 7.00 (m, 4H), 2.97 – 2.92 (m, 2H), 1.29 – 1.27 (m, 6H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 146.0, 128.0, 127.9, 125.8, 46.6, 18.1.



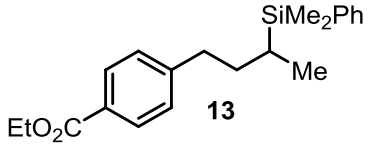
***meso*-2,3-diphenylbutane**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 4H), 7.25 – 7.19 (m, 6H), 2.85 – 2.76 (m, 2H), 1.06 – 1.00 (m, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.6, 128.4, 127.7, 126.2, 47.4, 21.2.



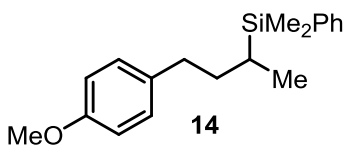
12

(**12**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (745 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.32 M] (4-phenylbutan-2-yl)zinc bromide **S17** (760 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with dry THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **12** as a clear oil (267 mg, 99%): ^{1}H NMR (600 MHz, CD_2Cl2) δ 7.49 – 7.46 (m, 2H), 7.36 – 7.30 (m, 3H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.14 (t, *J* = 7.4 Hz, 1H), 7.11 (d, *J* = 7.4 Hz, 2H), 2.76 (ddd, *J* = 14.6, 10.4, 4.9 Hz, 1H), 2.46 (ddd, *J* = 13.5, 10.1, 6.7 Hz, 1H), 1.79 (dddd, *J* = 13.7, 10.3, 6.6, 3.5 Hz, 1H), 1.44 – 1.36 (m, 1H), 1.02 (d, *J* = 7.3 Hz, 3H), 0.92 (ddp, *J* = 11.2, 7.3, 3.7 Hz, 1H), 0.26 (s, 3H), 0.25 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.0, 138.6, 134.1, 128.9, 128.6, 128.4, 127.8, 125.7, 35.0, 33.9, 19.0, 14.1, -4.6, -4.8; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.08; FTIR (cm^{-1}): 3067, 3025, 2953, 2864, 1454, 1427, 1248, 1112, 833, 812, 772, 699. HRMS (CI) m/z, calcd for $[C_{17}H_{21}Si]^+$: 253.1413; found: 253.1403. NMR spectra consistent with literature.[19]



13

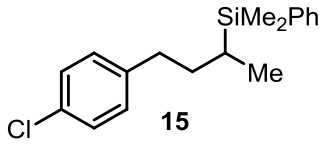
(**13**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (580 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.09 M] 4-(4-(ethoxycarbonyl)phenyl)butan-2yl)zinc bromide **S18** (920 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes:dichloromethane = 90:10 gradient to hexanes:dichloromethane = 80:20) and product dried (50 °C/0.1 mmHg) for 43 h to afford **13** as a clear oil (276.1 mg, 81%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.49 – 7.43 (m, 2H), 7.39 – 7.30 (m, 3H), 7.16 (d, *J* = 8.2 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 2.80 (ddd, *J* = 14.3, 9.9, 4.9 Hz, 1H), 2.52 (ddd, *J* = 13.6, 9.7, 7.0 Hz, 1H), 1.80 (dddd, *J* = 13.5, 10.2, 7.0, 3.5 Hz, 1H), 1.48 – 1.39 (m, 1H), 1.39 (t, *J* = 7.1 Hz, 3H), 1.02 (d, *J* = 7.2 Hz, 3H), 0.89 (dtq, *J* = 14.9, 7.6, 3.4 Hz, 1H), 0.26 (s, 3H), 0.25 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 148.4, 138.4, 134.0, 129.7, 129.0, 128.5, 128.0, 127.8, 60.9, 34.9, 33.5, 18.8, 14.5, 14.0, -4.6, -5.0; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.04; FTIR (cm^{-1}): 2954, 2864, 2361, 2340, 1427, 1718, 1610, 1275, 1248, 1107, 1021, 833, 701. HRMS (CI) m/z, calcd for $[C_{21}H_{29}O_2Si]^+$: 341.1937; found: 341.1926.



14

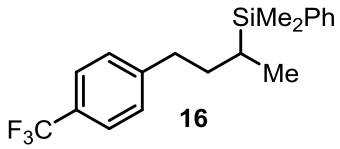
(**14**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (710 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.26 M] (4-(4-methoxyphenyl)butan-2yl)zinc bromide **S19** (795 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe

and worked up according to general procedure B and purified via silica gel flash chromatography (hexanes:DCM = 85:15;) to afford **14** as a clear oil (257 mg, 86%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50 – 7.44 (m, 2H), 7.38 – 7.30 (m, 3H), 7.03 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 3.79 (s, 3H), 2.71 (ddd, *J* = 14.4, 10.2, 4.8 Hz, 1H), 2.41 (ddd, *J* = 13.7, 9.9, 6.9 Hz, 1H), 1.82 – 1.73 (m, 1H), 1.42 – 1.34 (m, 1H), 1.01 (d, *J* = 7.3 Hz, 3H), 0.94 – 0.85 (m, 1H), 0.25 (s, 3H), 0.24 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 157.8, 138.7, 135.1, 134.1, 129.4, 128.9, 127.8, 113.9, 55.4, 34.08, 34.07, 18.9, 14.1, -4.6, -4.8; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.08; FTIR (cm^{-1}): 3068, 2998, 2952, 2864, 1612, 1512, 1246, 1177, 1113, 1038, 816, 771, 735, 702. HRMS (CI) m/z, calcd for $[C_{19}H_{26}OSi]^+$: 298.1753; found: 298.1741. NMR spectra consistent with literature.[5]



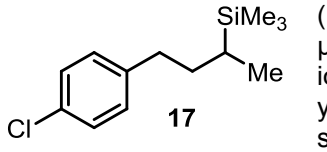
15

(**15**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (700 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.25 M] (4-(4-chlorophenyl)butan-2-yl)zinc bromide **S20** (800 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with dry THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **15** as a clear oil (285 mg, 94%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 7.3, 1.9 Hz, 2H), 7.34 (q, *J* = 5.6 Hz, 3H), 7.21 (d, *J* = 8.3 Hz, 2H), 7.02 (d, *J* = 8.3 Hz, 2H), 2.71 (ddd, *J* = 14.3, 10.0, 4.9 Hz, 1H), 2.43 (ddd, *J* = 13.7, 9.7, 7.0 Hz, 1H), 1.76 (dddd, *J* = 13.5, 10.2, 7.0, 3.5 Hz, 1H), 1.42 – 1.34 (m, 1H), 1.01 (d, *J* = 7.3 Hz, 3H), 0.87 (dqd, *J* = 10.9, 7.3, 3.5 Hz, 1H), 0.25 (s, 3H), 0.24 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.4, 138.5, 134.1, 131.4, 129.9, 129.0, 128.5, 127.8, 34.3, 33.8, 18.9, 14.1, -4.6, -4.9; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.05; FTIR (cm^{-1}):3068, 2953, 2864, 1492, 1427, 1248, 1111, 1092, 1015, 831, 812, 701, 522, 472. HRMS (CI) m/z, calcd for $[C_{18}H_{22}SiCl]^+$: 301.1179; found: 301.1166.



16

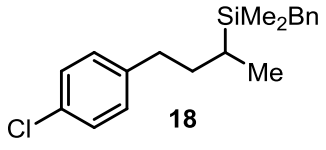
(**16**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (640 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.14 M] (4-(4-(trifluoromethyl)phenyl)butan-2yl)zinc bromide **S21** (880 μL, 1 mmol) were combined under N_2 and stirred at rt for 8 h. The reaction was quenched with THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes:ethyl acetate = 99:1) and product dried (45 °C/0.1 mmHg) for 7 h to afford **16** as a clear oil (276.1 mg, 81%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.0 Hz, 2H), 7.48 – 7.44 (m, 2H), 7.39 – 7.31 (m, 3H), 7.19 (d, *J* = 8.0 Hz, 2H), 2.80 (ddd, *J* = 14.4, 10.1, 5.0 Hz, 1H), 2.51 (ddd, *J* = 13.6, 9.8, 6.9 Hz, 1H), 1.79 (dddd, *J* = 13.6, 10.2, 6.9, 3.5 Hz, 1H), 1.47 – 1.36 (m, 1H), 1.02 (d, *J* = 7.2 Hz, 3H), 0.89 (ddp, *J* = 11.4, 7.6, 3.8 Hz, 1H), 0.26 (s, 3H), 0.25 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.0, 138.4, 134.0, 129.0, 128.8, 128.0 (q, *J* = 32.2 Hz), 127.8, 125.3 (q, J = 3.8 Hz), 124.5 (q, *J* = 271.9 Hz), 34f.8, 33.6, 18.9, 14.1, -4.6, -5.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.22; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.03; FTIR (cm^{-1}): 2954, 2866, 2361, 1618, 1427, 1326, 1249, 1163, 1124, 1068, 1018, 814, 701. HRMS (CI) m/z, calcd for $[C_{18}H_{20}F_3Si]^+$: 321.1286; found: 321.1271.



17

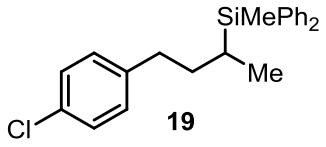
(**17**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (780 μL), triethylamine (140 μL, 1 mmol), trimethylsilyl iodide (290 μL, 2 mmol), and [1.25 M] (4-(4-chlorophenyl)butan-2-yl)zinc bromide **S20** (800 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **17** as a clear oil (232 mg, 96%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 – 7.22 (m, 2H), 7.13 – 7.08 (m, 2H), 2.76 (ddd, *J* = 14.4, 10.1, 4.8 Hz, 1H), 2.47 (ddd, *J* = 13.8, 10.1, 6.7 Hz, 1H), 1.74 (dddd, *J* = 13.8, 10.4, 6.7, 3.6 Hz, 1H), 1.38 (dtd, *J* = 13.5, 10.2, 4.8 Hz, 1H), 0.99 (d, *J* = 7.3 Hz, 3H), 0.65 – 0.56 (m, 1H), -0.04 (d, *J* = 1.7 Hz, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 131.4, 129.9, 128.5, 34.5, 34.0, 19.4, 14.0, -3.1; ^{29}Si NMR

(119 MHz, $CDCl_3$) δ 4.55; FTIR (cm^{-1}): 2953, 2865, 1492, 1248, 1093, 1016, 856, 834, 747, 521. HRMS (CI) m/z, calcd for $[C_{13}H_{20}SiCl]^+$: 239.1023; found: 239.1026.



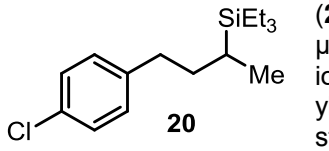
18

(**18**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (650 μL), triethylamine (140 μL, 1 mmol), benzyldimethylsilyl iodide (410 μL, 2 mmol), and [1.25 M] (4-(4-chlorophenyl)butan-2-yl)zinc bromide **S20** (800 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was diluted with dry THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **18** as a clear oil (313 mg, 99%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.3 Hz, 2H), 7.19 (t, *J* = 7.6 Hz, 2H), 7.09 (d, *J* = 8.3 Hz, 2H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 7.5 Hz, 2H), 2.77 (ddd, *J* = 14.3, 10.1, 4.8 Hz, 1H), 2.45 (ddd, *J* = 13.6, 9.8, 7.0 Hz, 1H), 2.08 (s, 2H), 1.74 (dddd, *J* = 13.4, 10.0, 6.9, 3.2 Hz, 1H), 1.43 – 1.35 (m, 1H), 1.01 (d, *J* = 7.4 Hz, 3H), 0.69 (dqd, *J* = 10.7, 7.3, 3.2 Hz, 1H), -0.08 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.2, 140.2, 131.3, 129.7, 128.4, 128.1, 123.9, 34.1, 33.7, 23.9, 17.8, 13.7, -5.2, -5.3; ^{29}Si NMR (119 MHz, $CDCl_3$) δ 5.24; FTIR (cm^{-1}): 3081, 3024, 2952, 2864, 1600, 1492, 1452, 1248, 1093, 1015, 830, 699. HRMS (CI) m/z, calcd for $[C_{19}H_{26}SiCl]^+$: 317.1492; found: 317.1490.



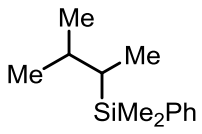
19

(**19**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (610 μL), triethylamine (140 μL, 1 mmol), methyldiphenylsilyl iodide (450 μL, 2 mmol), and [1.25 M] (4-(4-chlorophenyl)butan-2-yl)zinc bromide **S20** (800 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with dry THF (405 μL, 5 equiv, 5 mmol) via syringe and allowed to stir 15 minutes at rt then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **19** as a clear oil (291 mg, 80%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 – 7.45 (m, 4H), 7.41 – 7.30 (m, 6H), 7.22 (d, *J* = 8.3 Hz, 2H), 7.01 (d, *J* = 8.2 Hz, 2H), 2.75 (ddd, *J* = 13.9, 9.4, 4.8 Hz, 1H), 2.47 (dt, *J* = 13.7, 8.4 Hz, 1H), 1.91 – 1.79 (m, 1H), 1.49 – 1.40 (m, 1H), 1.35 – 1.23 (m, 1H), 1.08 (d, *J* = 7.3 Hz, 3H), 0.52 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.0, 136.4, 136.2, 134.8, 134.8, 131.3, 129.8, 129.1, 129.1, 128.3, 127.8, 127.8, 34.0, 33.6, 17.0, 14.1, -6.4; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -4.70; FTIR (cm^{-1}): 3068, 2952, 2864, 1491, 1427, 1252, 1111, 1015, 788, 737, 700, 490, 477. HRMS (CI) m/z, calcd for $[C_{23}H_{24}SiCl\ ]^+$: 363.1336; found: 363.1320.



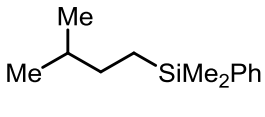
20

(**20**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (700 μL), triethylamine (140 μL, 1 mmol), triethylsilyl iodide (360 μL, 2 mmol), and [1.25 M] (4-(4-chlorophenyl)butan-2-yl)zinc bromide **S20** (800 μL, 1 mmol) were combined under N_2 and stirred at rt for 4 h. The reaction was quenched with wet Et_2O (3 mL) and H_2O (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **20** as a clear oil (85 mg, 30%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 2.79 (ddd, *J* = 14.3, 10.2, 4.8 Hz, 1H), 2.45 (ddd, *J* = 13.6, 9.9, 6.9 Hz, 1H), 1.75 (dddd, *J* = 13.4, 10.0, 6.9, 3.1 Hz, 1H), 1.46 – 1.38 (m, 1H), 1.02 (d, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.9 Hz, 9H), 0.82 – 0.74 (m, 1H), 0.53 (q, *J* = 7.9 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 131.4, 129.9, 128.5, 34.6, 34.2, 16.5, 14.2, 7.8, 2.3; ^{29}Si NMR (119 MHz, $CDCl_3$) δ 8.20; FTIR (cm^{-1}): 2952, 2909, 2874, 1492, 1456, 1239, 1093, 1016, 834, 806, 732, 521. HRMS (CI) m/z, calcd for $[C_{14}H_{22}SiCl\ ]^+$: 253.1179; found: 253.1177.



22

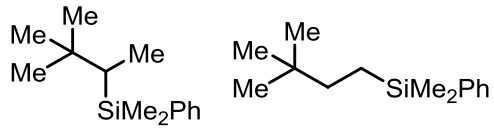
>99:1



23

(**22**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (500 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [1.05 M] (3-methylbutan-2-yl)zinc iodide **21** in dioxane (1.00 mL, 1.05 mmol) were combined under N_2 and stirred at rt for 4 h. The

reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford **22** as a clear volatile oil (175.1 mg, 81%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54 – 7.51 (m, 2H), 7.37 – 7.33 (m, 3H), 1.87 (heptd, *J* = 6.8, 3.4 Hz, 1H), 0.94 – 0.89 (m, 7H), 0.81 (d, *J* = 6.9 Hz, 3H), 0.30 (s, 3H), 0.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.9, 134.0, 128.7, 127.8, 29.0, 26.8, 23.2, 19.9, 9.7, -3.2, -3.3; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -0.85; FTIR (cm^{-1}): 2955, 2871, 1465, 1427, 1248, 1111, 834, 817, 768, 733, 700. HRMS (CI) m/z, calcd for $[C_{13}H_{22}Si]^+$: 206.1491; found: 206.1482.



25 62:38 **26**

(**25 : 26**) According to general procedure B, $(DrewPhos)_2PdI_2$ (16 mg, 10 μmol), dioxane (60 μL), triethylamine (140 μL, 1 mmol), dimethylphenylsilyl iodide (360 μL, 2 mmol), and [0.54 M] (3,3-dimethylbutan-2-yl)zinc iodide **24** (1.85 mL, 1 mmol) were combined under N_2 and stirred at rt for 8 h. The reaction was quenched with wet EtOAc (0.5 mL) and brine (3 mL) via syringe then worked up according to general procedure B and purified via silica gel flash chromatography (hexanes) to afford an inseparable mixture of isomers **25**:**26** (62:38) as a clear oil (123.0 mg, 56%): Useful diagnostic peaks for each compound are listed. **25:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.50 (m, 2H), 7.38 – 7.31 (m, 3H), 0.98 – 0.92 (m, 4H), 0.89 (s, 9H), 0.36 (s, 3H), 0.32 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.2, 134.0, 128.6, 127.7, 33.7, 32.3, 30.3, 11.9, -0.5, -2.0; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -2.06. HRMS (CI) m/z, calcd for $[C_{13}H_{21}Si]^+$: 205.1413; found: 205.1407.

26: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 2H), 7.38 – 7.31 (m, 3H), 1.22 – 1.15 (m, 2H), 0.85 (s, 9H), 0.72 – 0.65 (m, 2H), 0.25 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.8, 133.7, 128.9, 127.8, 37.9, 31.2, 28.9, 9.8, -3.0; ^{29}Si NMR (119 MHz, $CDCl_3$) δ -2.21. HRMS (CI) m/z, calcd for $[C_{13}H_{21}Si]^+$: 205.1413; found: 205.1406.

25 + 26: FTIR (cm^{-1}): 2955, 2911, 1466, 1427, 1363, 1249, 1112, 834, 815, 700.

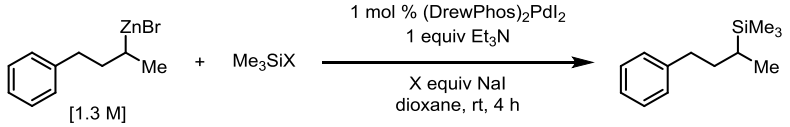
6. Additional Optimization Data:

Note: All reactions in this section were performed on 0.25 mmol in a nitrogen-filled glovebox with a [0.5 M] overall concentration based on the sum of all liquid reagents.

Examination of Silyl Electrophiles:
We also examined the use of other silyl electrophiles other than silyl iodides (Table S1). Previous studies have shown that the addition of iodide additives can be beneficial with use of silyl chlorides, bromides, and triflates, so the reaction was also examined with NaI additive. The results show minimal reactivity with Me_3SiBr in the absence of NaI, and modest reactivity with. For silyl chlorides and triflates, negligible reactivity was observed with or without NaI.

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with $(DrewPhos)_2PdI_2$ (0.01 equiv), dioxane, triethylamine (1 equiv), and trimethylsilyl halide (2 equiv). Vial was then sealed with a septum cap and removed from GB. (4-phenylbutan-2-yl)zinc bromide (1 equiv) was added via syringe and the flask was then stirred at rt for 4 h. The reaction was quenched with Et_2O (1 mL) then H_2O (0.5 mL) via syringe. Nonane (32 mg, 45 µL, 0.25 mmol, 1 equiv) and 1,3,5-trimethoxybenzene (TMB) (14 mg, 0.25 mmol, 0.33 equiv) were added as GC and NMR internal standards respectively. Brine (1 mL) and Et_2O (1 mL) were then added and the vials shaken. An aliquot was then filtered through a $MgSO_4$ and Silica plug. The solution was directly analyzed by GC. The solvent was removed *in vacuo* then analyzed by NMR.

Table S1. Examination of Silyl Electrophiles



Entry	Me_3SiX	0 equiv NaI[a]	3 equiv NaI[a]
1	Me_3SiI	98%	99%
2	Me_3SiBr	26%	61%
3	Me_3SiCl	1%	11%
4	Me_3SiOTf	2%	5%

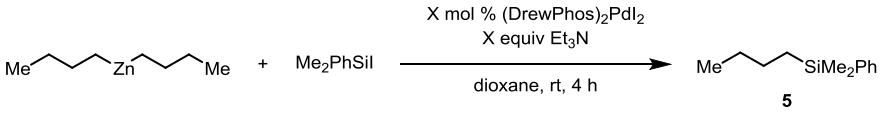
[a]Yields obtained by ^1H NMR with TMB as an internal standard.

Examination of Dibutyl Zinc Reactivity:

We also examined the use of dialkylzinc reagents in the coupling reaction. As can be seen in Table S2, with Bu_2Zn only trace background reaction is observed, which is comparable to the background reaction with BuZnBr. Under palladium-catalyzed conditions, quantitative alkylation resulted. Et_3N does not effect this reaction. It is notable that only 0.5 equiv of Bu_2Zn is required in this reaction, both alkyl groups transfer to the product.

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with $(DrewPhos)_2PdI_2$ (0.01 equiv), dioxane, triethylamine (1 equiv), and dimethylphenylsilyl iodide (2 equiv), and dibutylzinc (0.5 equiv). Vial was then sealed with a septum cap, removed from GB, and stirred at rt for 4 h. The reaction was quenched with Et_2O (1 mL) then H_2O (0.5 mL) via syringe. Nonane (32 mg, 45 µL, 0.25 mmol, 1 equiv) and 1,3,5-trimethoxybenzene (TMB) (14 mg, 0.25 mmol, 0.33 equiv) were added as GC and NMR internal standards respectively. Brine (1 mL) and Et_2O (1 mL) were then added and the vials shaken. An aliquot was then filtered through a $MgSO_4$ and Silica plug. The solution was directly analyzed by GC. The solvent was removed *in vacuo* then analyzed by NMR.

Table S2. Reactivity of Dibutylzinc



Entry	% Pd	1 equiv Et_3N[a]	0 equiv Et_3N[a]
1	1	99%	99%
2	0	7%	7%

[a]Yields obtained by ^{1}H NMR with TMB as an internal standard.

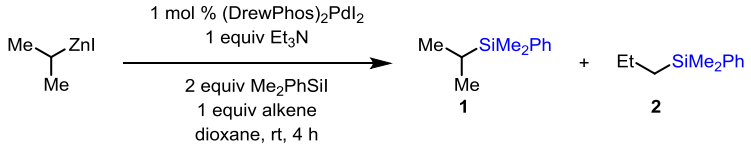
Isolation of the palladium catalyzed reaction in the presence of triethylamine via flash chromatography (hexanes) afforded **5** as a clear oil (42 mg, 88%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.50 (m, 2H), 7.37 – 7.34 (m, 3H), 1.36 – 1.26 (m, 4H), 0.88 (t, *J* = 6.9 Hz, 3H), 0.79 – 0.73 (m, 2H), 0.26 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 133.7, 128.9, 127.8, 26.7, 26.3, 15.6, 13.9, -2.9.

Examination of Alkene Additives:

As noted in the main text, alkenes interfered with the reaction, as is reflected in the additive study shown in Table S3. This appears to be a function of alkene substitution, as the effect is most notable with lower substituted alkenes.

In a nitrogen filled glovebox, a 1-dram vial equipped with a magnetic stirbar was charged with $(DrewPhos)_2PdI_2$ (0.01 equiv), dioxane, triethylamine (1 equiv), alkene (1 equiv) and trimethylsilyl halide (2 equiv). Vial was then sealed with a septum cap and removed from GB. Isopropylzinc bromide (1 equiv) was added via syringe and the flask was then stirred at rt for 4 h. The reaction was quenched with Et_2O (1 mL) then H_2O (0.5 mL) via syringe. Nonane (32 mg, 45 µL, 0.25 mmol, 1 equiv) and 1,3,5-trimethoxybenzene (TMB) (14 mg, 0.25 mmol, 0.33 equiv) were added as GC and NMR internal standards respectively. Brine (1 mL) and Et_2O (1 mL) were then added and the vials shaken. An aliquot was then filtered through a $MgSO_4$ and Silica plug. The solution was directly analyzed by GC. The solvent was removed *in vacuo* then analyzed by NMR.

Table S3. Impact of Alkene Additives



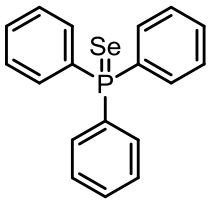
Entry	Alkene	1+2 (%)[a]	1:2[b]
1	None	98	>99:1
2	4-octene	55	98:2
3	1-methyl-cyclohexene	99	>99:1
4	(+)-limonene	75	>99:1

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

7. *In Situ* Phosphine Selenide Data:

General Procedure C:

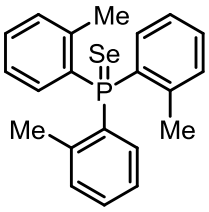
Using a modified literature procedure,[20] in a nitrogen-filled glovebox, a 1-dram vial equipped with a stirbar was charged with phosphine (1 equiv), selenium powder (5 equiv), and $CDCl_3$ [0.04 M]. The vial was sealed with a teflon lined capped and heated in a temperature controlled aluminum block at 70 °C on a stirplate for 16 h. The vial was allowed to cool to rt. The mixture was filtered directly into an NMR tube through a pipette fitted with a paper filter (small piece of laboratory paper wipe) to remove excess selenium metal. The tube was sealed with a standard NMR cap and the ^{31}P NMR spectrum acquired.



Se-1

(**Se-1**) According to general procedure C, triphenlyphosphine (5 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

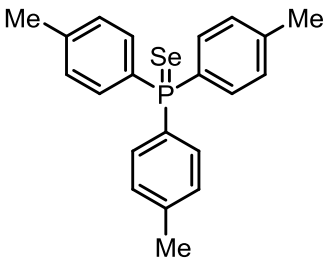
^{31}P NMR (243 MHz, $CDCl_3$) δ 35.3 ($^1J_{P\text{-}Se}$ = 729.9 Hz).



Se-2

(**Se-2**) According to general procedure C, tri-*ortho*-tolylphosphine (6 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

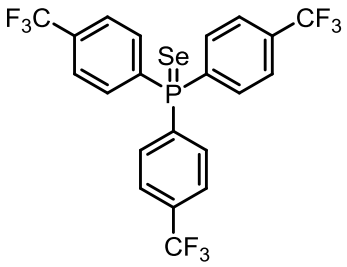
^{31}P NMR (243 MHz, $CDCl_3$) δ 28.1 ($^1J_{P\text{-}Se}$ = 704.6 Hz).



Se-3

(**Se-3**) According to general procedure C, tri-*para*-tolylphosphine (6 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

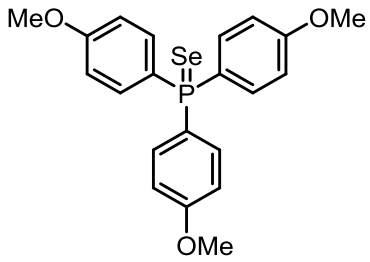
^{31}P NMR (243 MHz, $CDCl_3$) δ 33.7 ($^1J_{P\text{-}Se}$ = 719.3 Hz).



Se-4

(**Se-4**) According to general procedure C, tris(*para*-trifluoromethylphenyl)phosphine (9 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

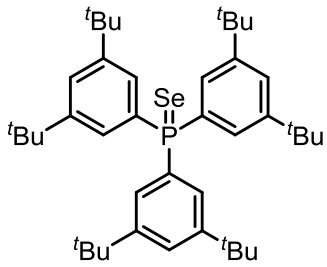
^{31}P NMR (243 MHz, $CDCl_3$) δ 34.1 ($^1J_{P\text{-}Se}$ = 766.3 Hz).



Se-5

(**Se-5**) According to general procedure C, tris(*para*-methoxyphenyl)phosphine (7 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

^{31}P NMR (243 MHz, $CDCl_3$) δ 31.6 ($^{1}J_{P\text{-}Se}$ = 712.5 Hz).



Se-6

(**Se-6**) According to general procedure C, DrewPhos (12 mg, 20 µmol, 1 equiv), selenium (8 mg, 100 µmol, 5 equiv), and $CDCl_3$ (500 µL, [0.04 M]) were combined and stirred at 70 °C for 16 h. The reaction was allowed to cool to rt and the mixture filtered directly into an NMR tube.

^{31}P NMR (243 MHz, $CDCl_3$) δ 37.4 ($^{1}J_{P\text{-}Se}$ = 714.0 Hz).

Table S4. Phosphine Selenide Coupling Constants

Ar_3P	Measured $J_{P\text{-}Se}$	Literature[20] $J_{P\text{-}Se}$
Ph_3P	729.9	729
(*o*-tol)$_3$P	704.6	704
(*p*-tol)$_3$P	719.3	719
(*p*-CF_3Ph)$_3$P	766.3	766
(*p*-OMePh)$_3$P	712.5	712
DrewPhos	714.0	N/A

The coupling values measured in our hands matched the literature within error. ^{31}P-^{77}Se coupling values for simple phosphines have been shown to have a degree of variability ±1 Hz.[21]

8. Crystallographic Details:

X-ray Structural Analysis for (DrewPhos)$_2$PdI$_2$. The crystal was mounted using viscous oil onto a plastic mesh and cooled to the data collection temperature (200 K). Data were collected on a Bruker-AXS APEX Duo CCD diffractometer. Unit cell parameters were obtained from 36 data frames, 0.5º ω, from three different sections of the Ewald sphere. No symmetry higher than triclinic was observed and the centrosymmetric space group option yielded chemically reasonable and computationally stable results of refinement. The data-set was treated with absorption corrections based on redundant multiscan data. The structure was solved using direct methods and refined with full-matrix, least-squares procedures on F^2.[22] The molecule is located at the inversion center.

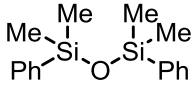
All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were treated as idealized contributions. Scattering factors are contained in the SHELXTL 6.12 program library.[22]

9. References:

(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
(2) McAtee, J. R.; Yap, G. P. A.; Watson, D. A. *J. Am. Chem. Soc.* **2014**, *136*, 10166.
(3) Denmark, S. E.; Cresswell, A. J. *J. Org. Chem.* **2013**, *78*, 12593.
(4) Powell, D. A.; Fu, G. C. *J. Am. Chem. Soc.* **2004**, *126*, 7788.
(5) Chu, C. K.; Liang, Y.; Fu, G. C. *J. Am. Chem. Soc.* **2016**, *138*, 6404.
(6) (a) Kunai, A.; Ohshita, J. *J. Organomet. Chem.* **2003**, *686*, 3; (b) McAtee, J. R.; Krause, S. B.; Watson, D. A. *Adv. Synth. Catal.* **2015**, *357*, 2317.
(7) Kunai, A.; Sakurai, T.; Toyoda, E.; Ishikawa, M.; Yamamoto, Y. *Organometallics* **1994**, *13*, 3233.
(8) Wilt, J. W.; Kolewe, O.; Kraemer, J. F. *J. Am. Chem. Soc.* **1969**, *91*, 2624.
(9) Jeffery, T. *Tetrahedron Lett.* **1991**, *32*, 2121.
(10) Condon, S.; Dupré, D.; Falgayrac, G.; Nédélec, J.-Y. *Eur. J. Org. Chem.* **2002**, *2002*, 105.
(11) Han, X.; Wu, J. *Angew. Chem., Int. Ed. Engl.* **2013**, *52*, 4637.
(12) Andrade, V. S. C. M., Marcio, C.S. *J. Braz. Chem. Soc.* **2014**, *25*, 975.
(13) Clive, D. L. J.; Pham, M. P.; Subedi, R. *J. Am. Chem. Soc.* **2007**, *129*, 2713.
(14) Krasovskiy, A.; Knochel, P. *Synthesis* **2006**, *2006*, 0890.
(15) Matsumoto, A.; Ito, Y. *J. Org. Chem.* **2000**, *65*, 5707.
(16) (a) Inubushi, H.; Kondo, H.; Lesbani, A.; Miyachi, M.; Yamanoi, Y.; Nishihara, H. *Chem. Commun.* **2013**, *49*, 134; (b) Negishi, E.; Akiyoshi, K. *J. Am. Chem. Soc.* **1988**, *110*, 646.
(17) Nakamura, S.; Uchiyama, M. *J. Am. Chem. Soc.* **2007**, *129*, 28.
(18) Sato, K.; Inoue, Y.; Mori, T.; Sakaue, A.; Tarui, A.; Omote, M.; Kumadaki, I.; Ando, A. *Org. Lett.* **2014**, *16*, 3756.
(19) Fleming, I.; Henning, R.; Parker, D. C.; Plaut, H. E.; Sanderson, P. E. J. *J. Chem. Soc., Perkin Trans. 1* **1995**, 317.
(20) Kawaguchi, S.-i.; Minamida, Y.; Okuda, T.; Sato, Y.; Saeki, T.; Yoshimura, A.; Nomoto, A.; Ogawa, A. *Adv. Synth. Catal.* **2015**, *357*, 2509.
(21) McFarlane, W.; Rycroft, D. S. *J. Chem. Soc., Dalton Trans.* **1973**, 2162.
(22) Sheldrick, G. *Acta Cryst. A* **2008**, *64*, 112.
(23) Hazra, C. K.; Oestreich, M. *Org. Lett.* **2012**, *14*, 4010.

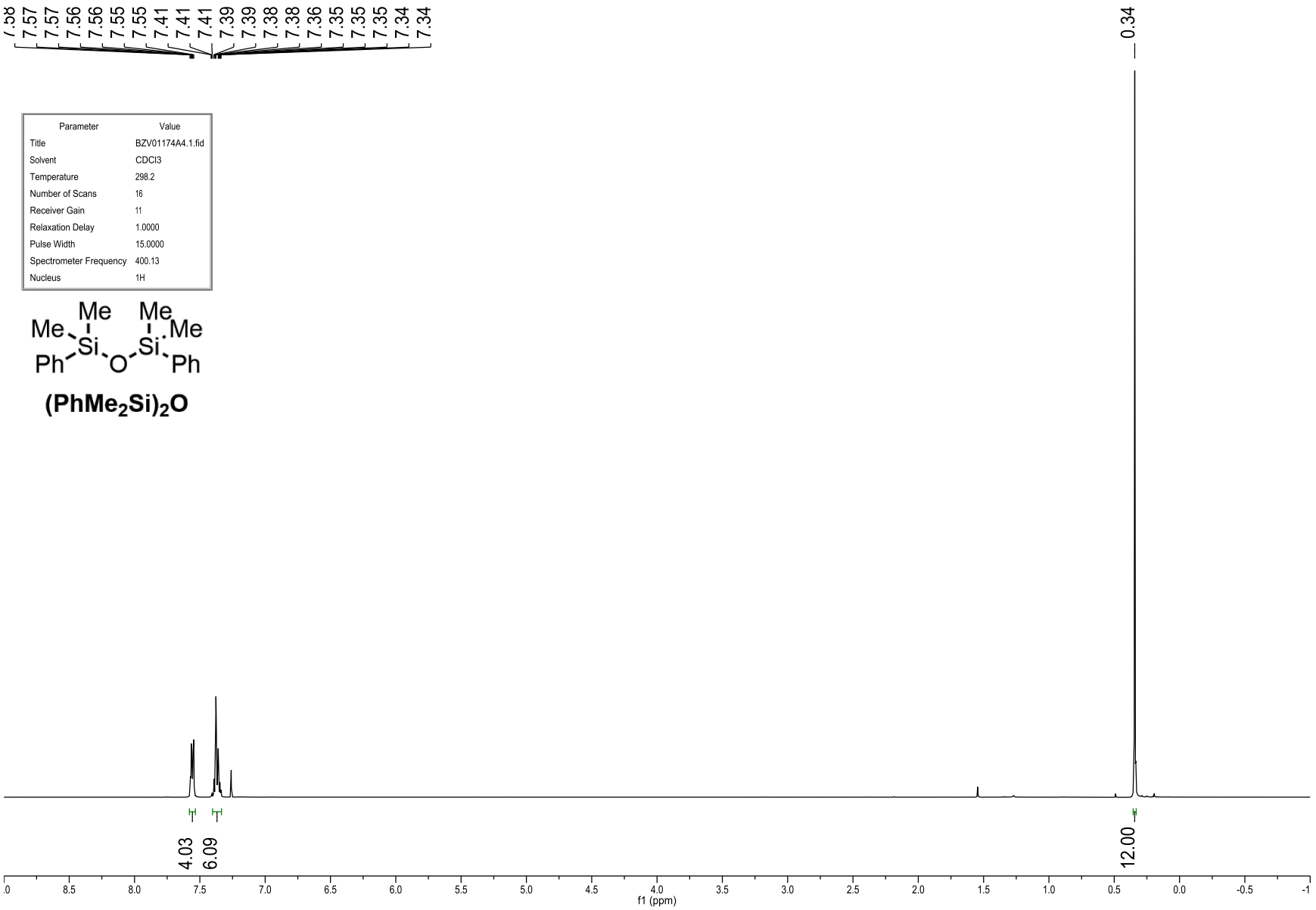
10. Spectral Data:

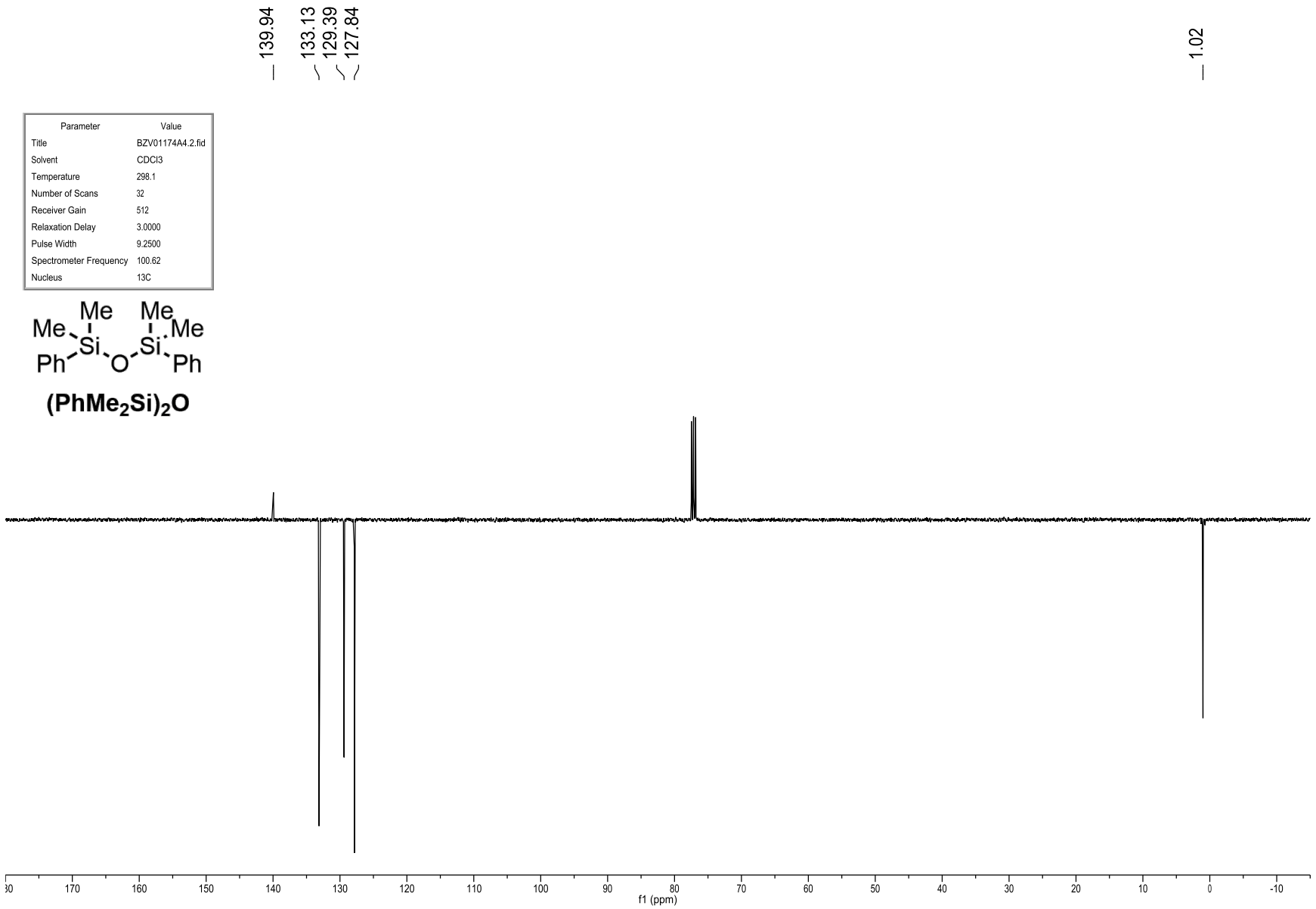
The main silicon by-product from the reaction is $(PhMe_2Si)_2O$.
For reference, spectra of $(PhMe_2Si)_2O$ have been provided matching literature reports.[23]



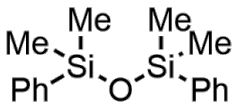
$(PhMe_2Si)_2O$

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.50 (m, 4H), 7.47 – 7.29 (m, 6H), 0.34 (s, 12H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 133.1, 129.4, 127.8, 1.0.
^{29}Si NMR (119 MHz, $CDCl_3$) δ -1.24.

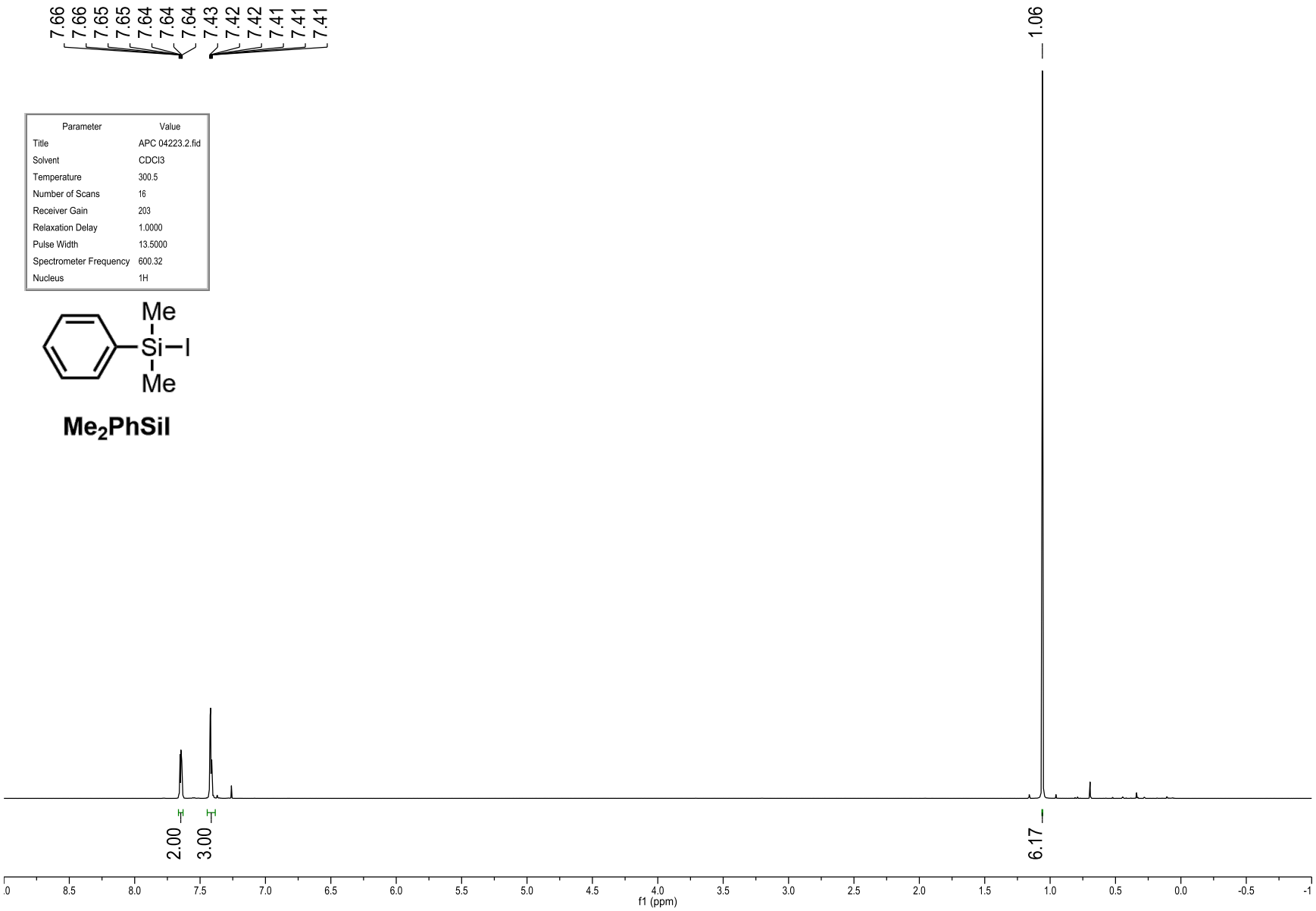

7.57
7.57
7.56
7.56
7.55
7.55
7.41
7.41
7.41
7.39
7.39
7.38
7.38
7.36
7.35
7.35
7.35
7.34
7.34
0.34
Parameter Value
Title BZV01174A4.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
Me
Me
Si
Si
O
Ph
Ph
(PhMe2Si)2O
4.03
6.09
12.00
f1 (ppm)


139.94
133.13
129.39
127.84
1.02
Parameter Value
Title BZV01174A4.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 32
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me Me
Me Si O Si Me
Ph Ph
(PhMe2Si)2O
f1 (ppm)

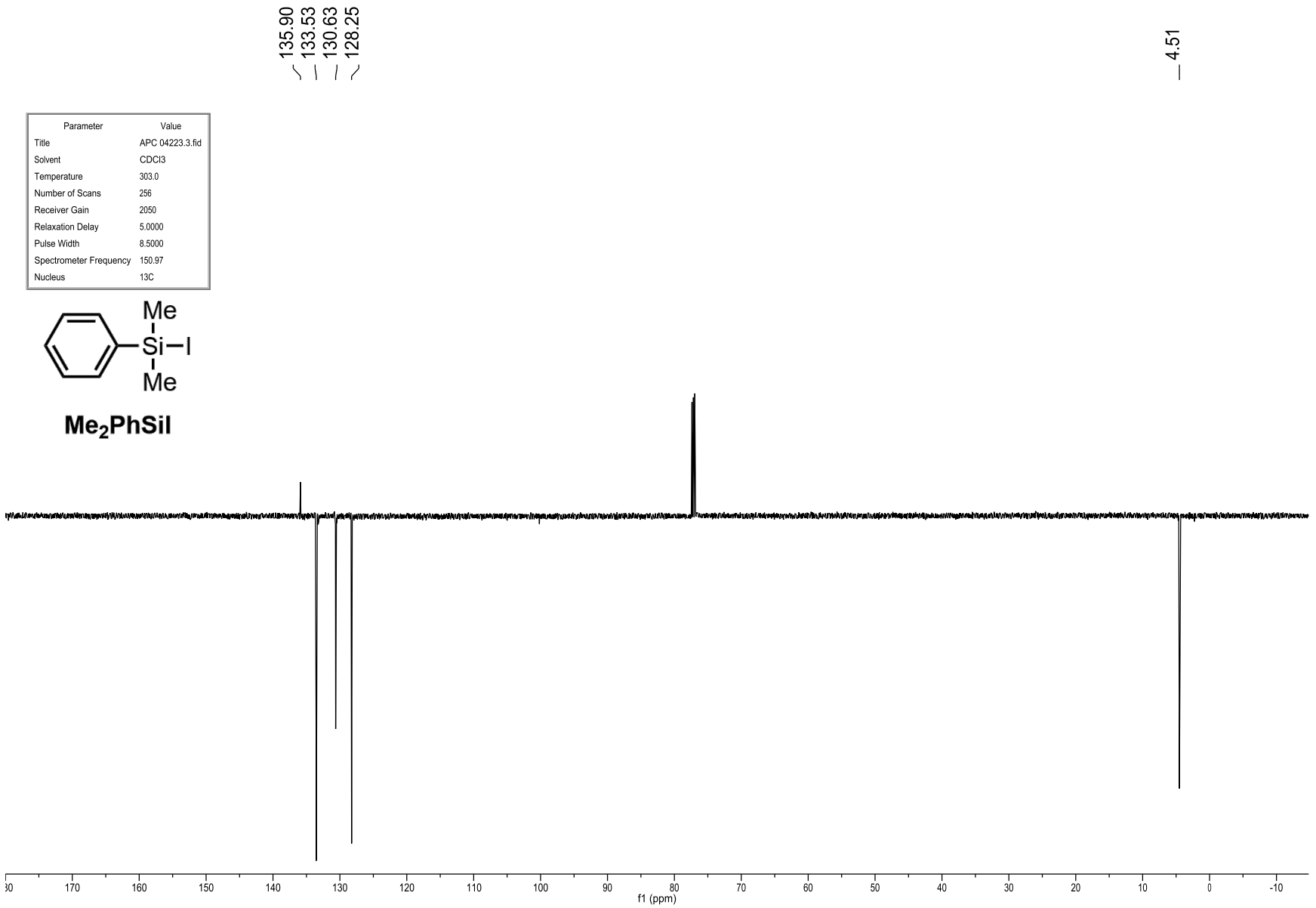
Parameter	Value
Title	BZV01174A4Si.1.fid
Solvent	CDCl3
Temperature	299.3
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si



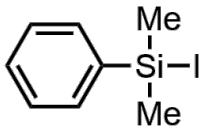
$(PhMe_2Si)_2O$


7.66
7.66
7.65
7.65
7.64
7.64
7.64
7.43
7.42
7.42
7.41
7.41
7.41
1.06
Parameter Value
Title APC 04223.2.fid
Solvent CDCl3
Temperature 300.5
Number of Scans 16
Receiver Gain 203
Relaxation Delay 1.0000
Pulse Width 13.5000
Spectrometer Frequency 600.32
Nucleus 1H
Me
Si
I
Me
2.00
3.00
6.17
f1 (ppm)

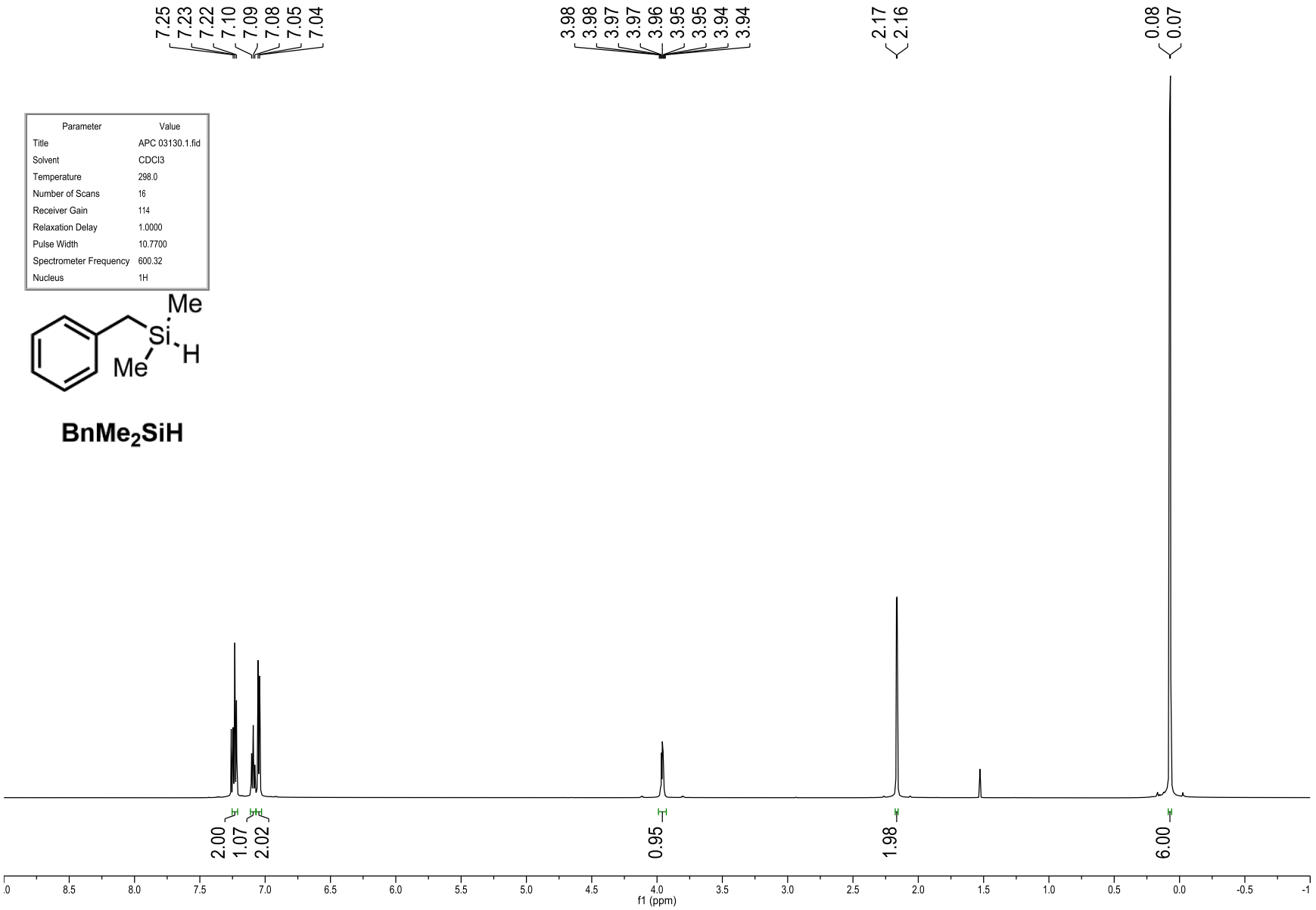
Me_2PhSiI


135.90
133.53
130.63
128.25
4.51
Parameter Value
Title APC 04223.3.fid
Solvent CDCl3
Temperature 303.0
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
Si
I
Me
Me2PhSiI
f1 (ppm)

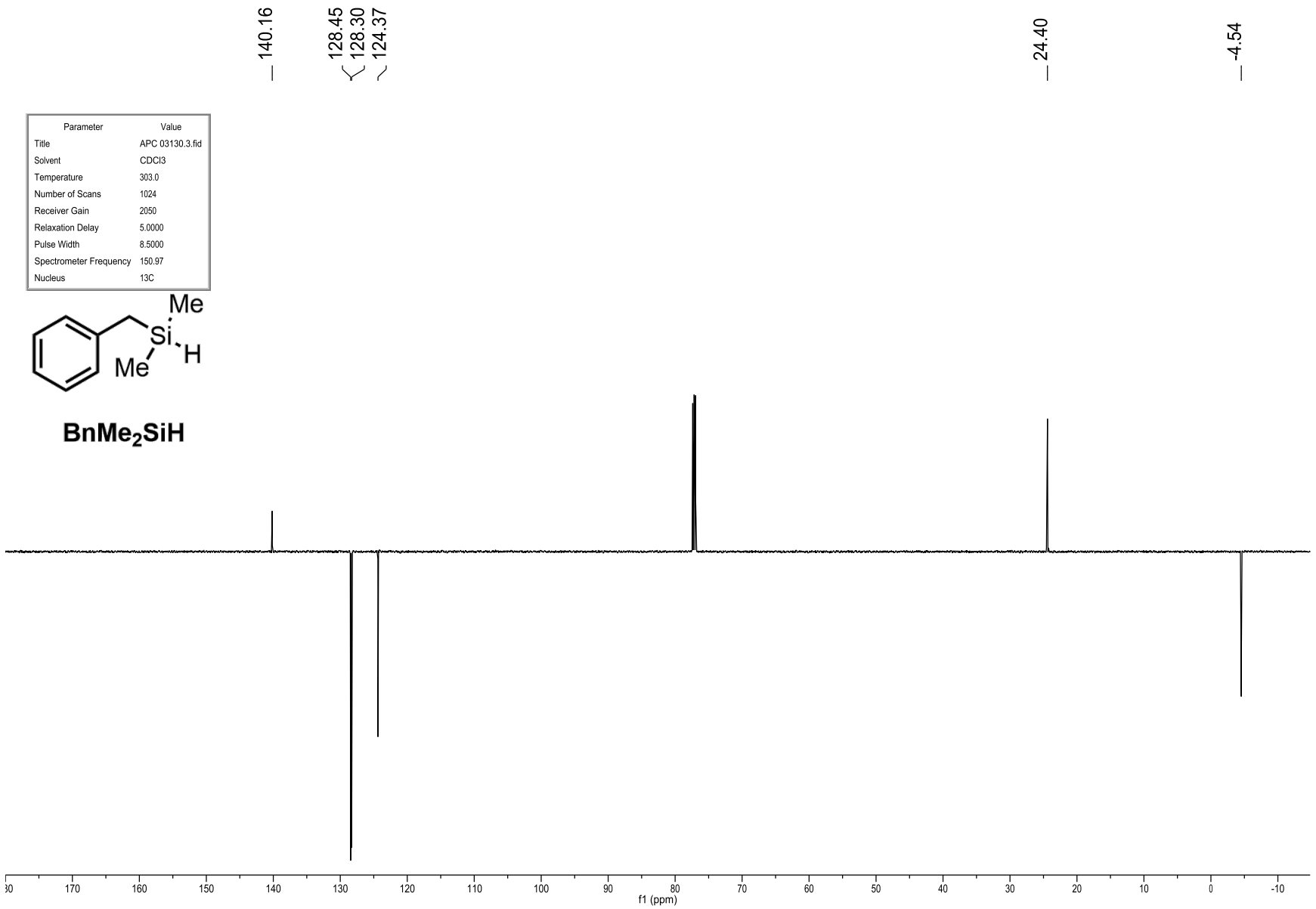
Parameter	Value
Title	APC 04223.4.fid
Solvent	CDCl3
Temperature	299.9
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si



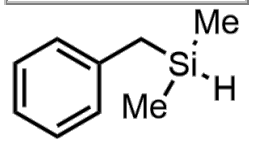
Me_2PhSiI


7.25
7.23
7.22
7.10
7.09
7.08
7.05
7.04
3.98
3.98
3.97
3.97
3.96
3.95
3.95
3.94
3.94
2.17
2.16
0.08
0.07
Parameter Value
Title APC 03130.1.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 114
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
Me
Si
H
Me
2.00
1.07
2.02
0.95
1.98
6.00
f1 (ppm)

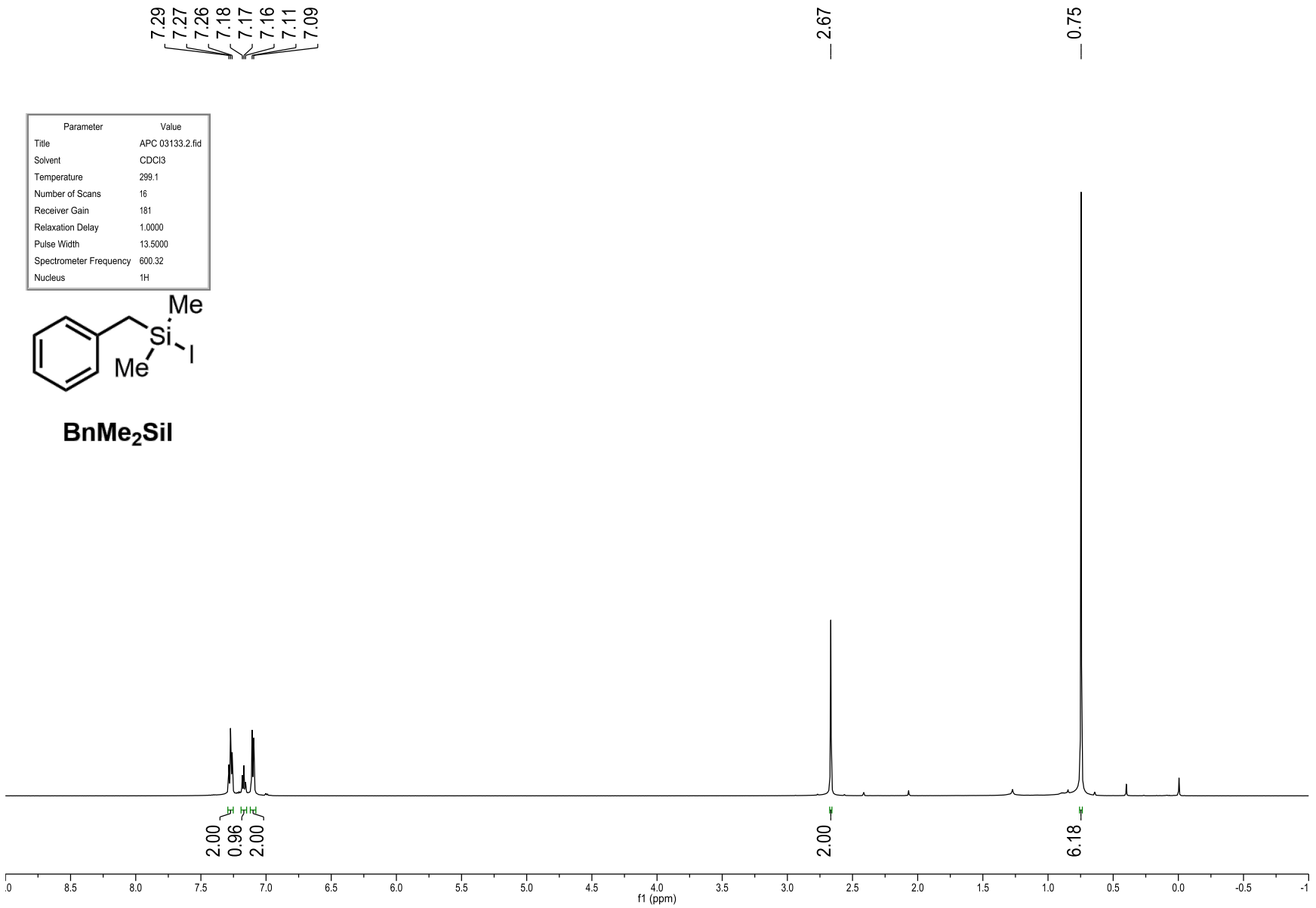
$BnMe_2SiH$

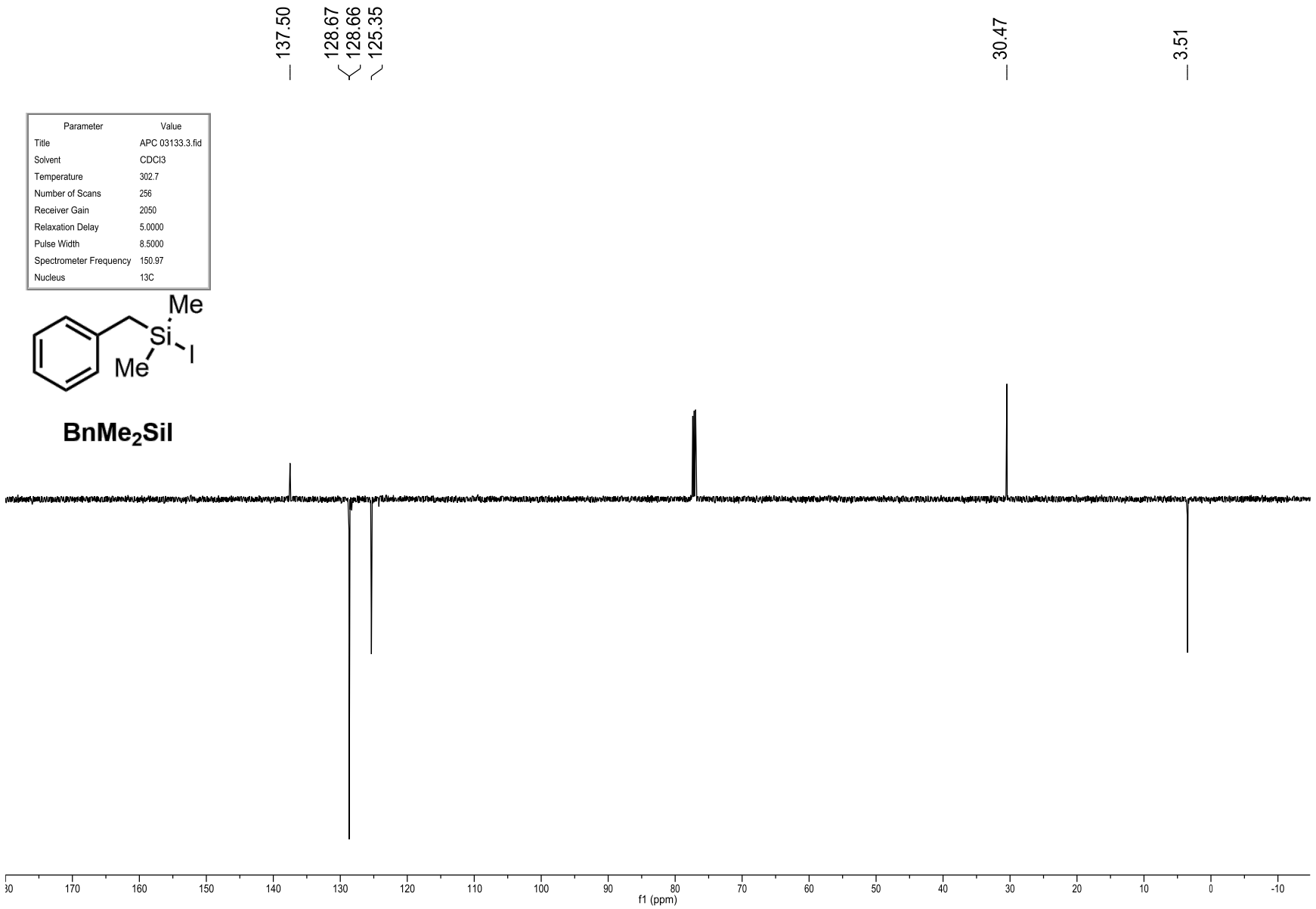

140.16
128.45
128.30
124.37
24.40
-4.54
Parameter Value
Title APC 03130.3.fid
Solvent CDCl3
Temperature 303.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
Si
H
Me
BnMe2SiH
f1 (ppm)

Parameter	Value
Title	APC 03130.4.fid
Solvent	CDCl3
Temperature	299.9
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si



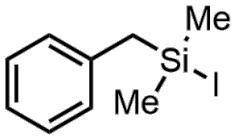
$BnMe_2SiH$


7.29
7.27
7.26
7.18
7.17
7.16
7.11
7.09
2.67
0.75
Parameter Value
Title APC 03133.2.fid
Solvent CDCl3
Temperature 299.1
Number of Scans 16
Receiver Gain 181
Relaxation Delay 1.0000
Pulse Width 13.5000
Spectrometer Frequency 600.32
Nucleus 1H
Me
Si
I
Me
BnMe2SiI
2.00
0.96
2.00
2.00
6.18
f1 (ppm)

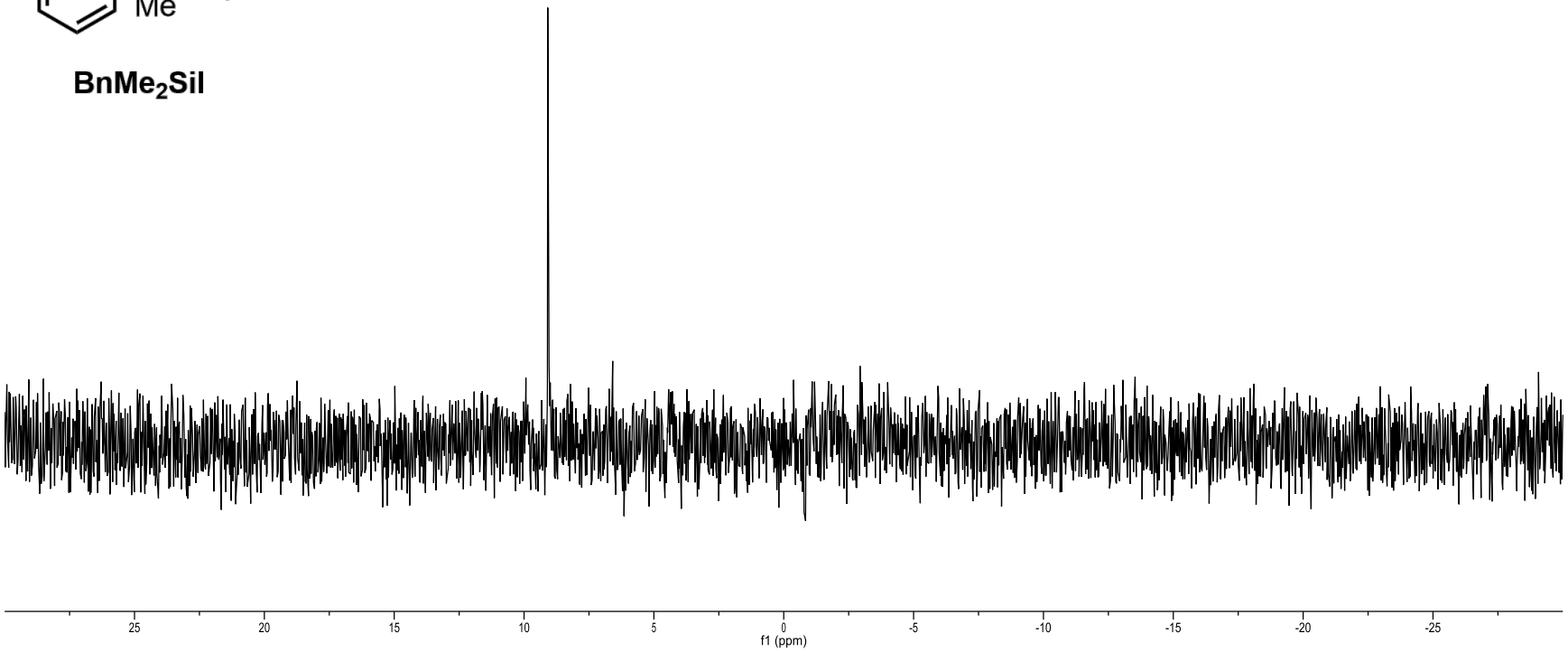

137.50
128.67
128.66
125.35
30.47
3.51
Parameter Value
Title APC 03133.3.fid
Solvent CDCl3
Temperature 302.7
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
Si
I
Me
BnMe2SiI
f1 (ppm)

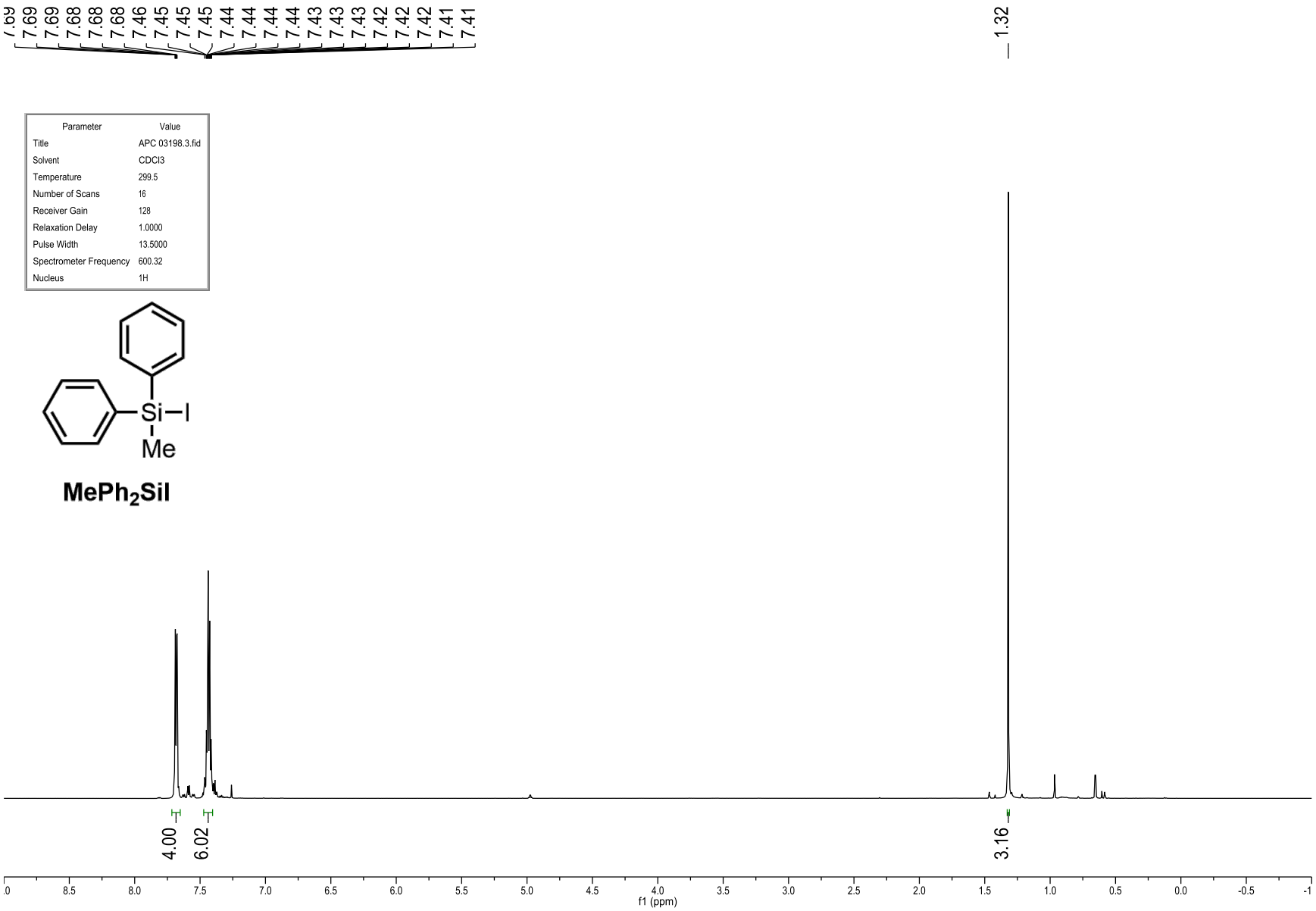
9.09

Parameter	Value
Title	APC 03133.4.fid
Solvent	CDCl3
Temperature	299.7
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si

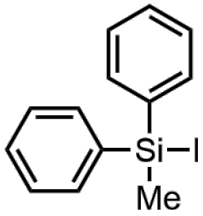


$BnMe_2SiI$




7.69
7.69
7.68
7.68
7.68
7.46
7.45
7.45
7.45
7.44
7.44
7.44
7.44
7.43
7.43
7.43
7.42
7.42
7.42
7.41
7.41
1.32
Parameter Value
Title APC 03198.3.fid
Solvent CDCl3
Temperature 299.5
Number of Scans 16
Receiver Gain 128
Relaxation Delay 1.0000
Pulse Width 13.5000
Spectrometer Frequency 600.32
Nucleus 1H
Si
I
Me
MePh2SiI
4.00
6.02
3.16
f1 (ppm)

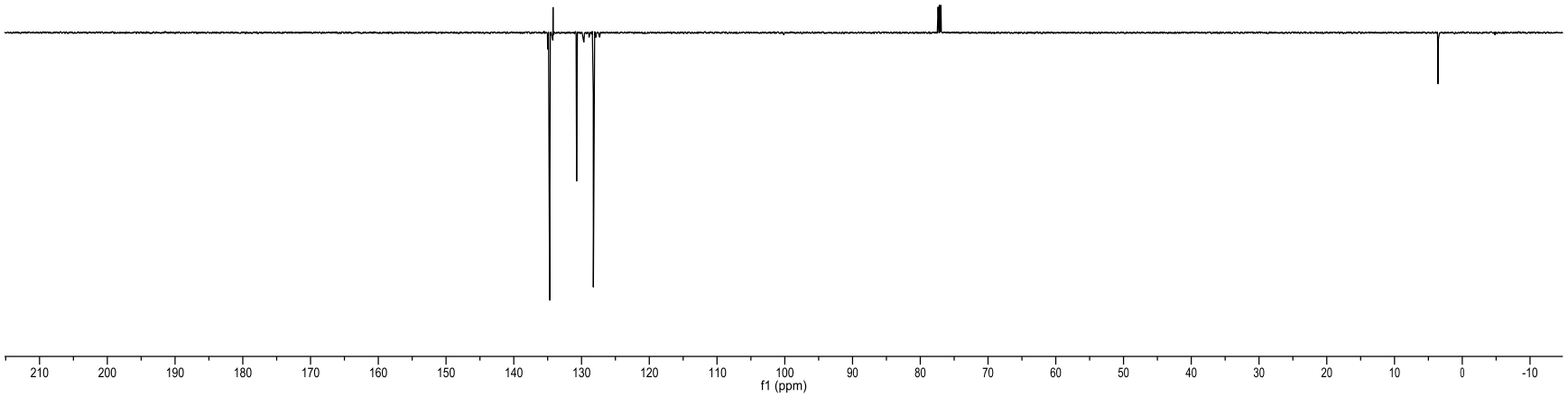
Parameter	Value
Title	APC 03198.4.fid
Solvent	CDCl3
Temperature	302.8
Number of Scans	256
Receiver Gain	2050
Relaxation Delay	5.0000
Pulse Width	8.5000
Spectrometer Frequency	150.97
Nucleus	13C



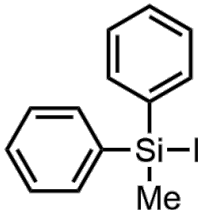
$MePh_2SiI$

134.71
134.21
130.73
128.25

3.56

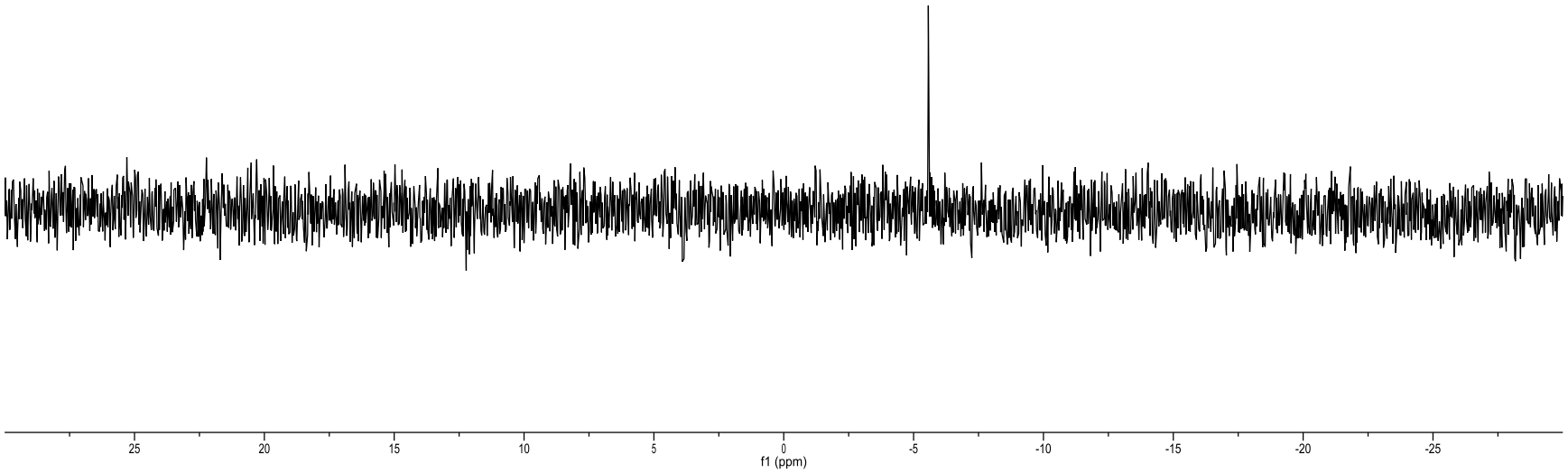


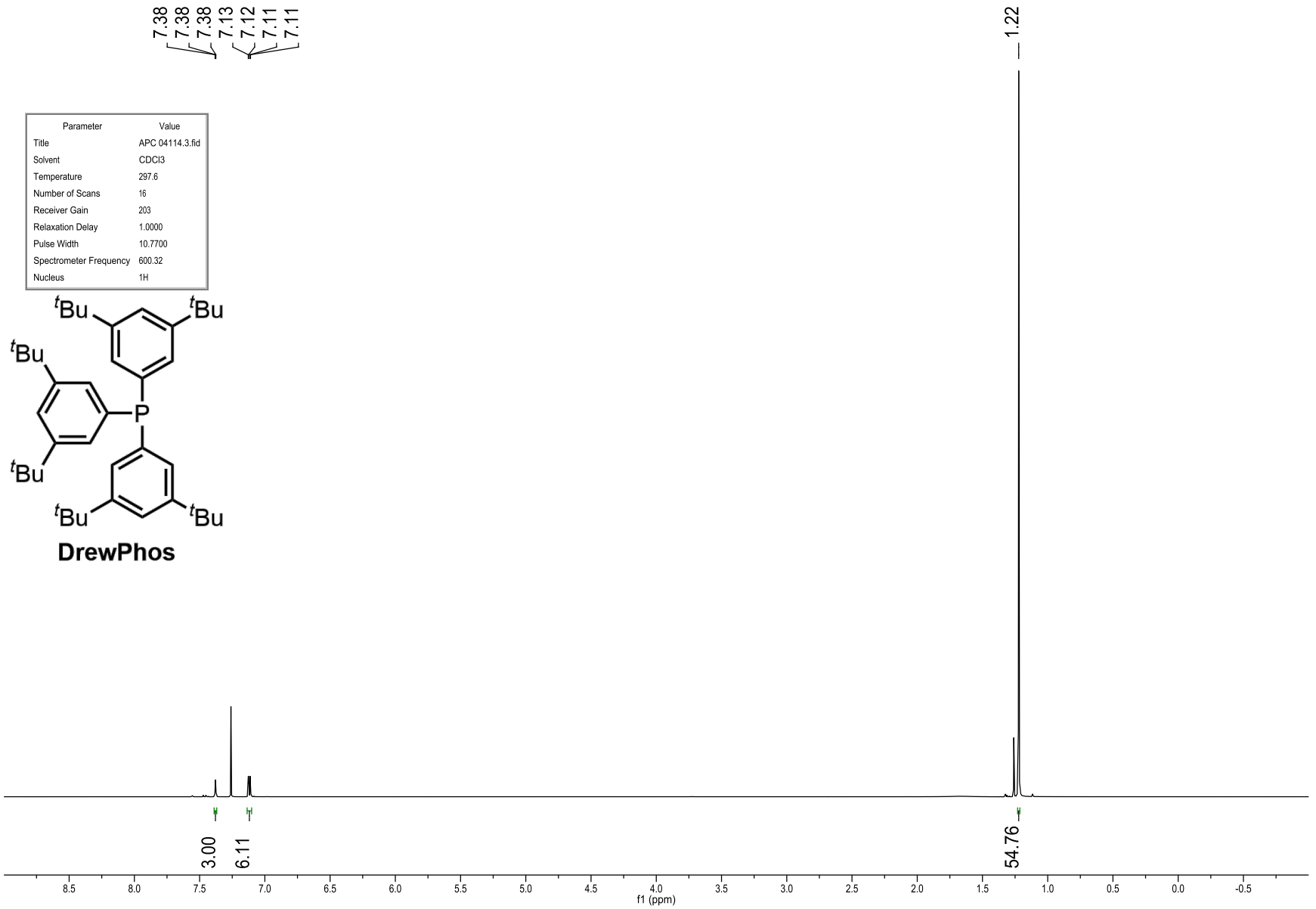
Parameter	Value
Title	APC 03198.2.fid
Solvent	CDCl3
Temperature	297.5
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

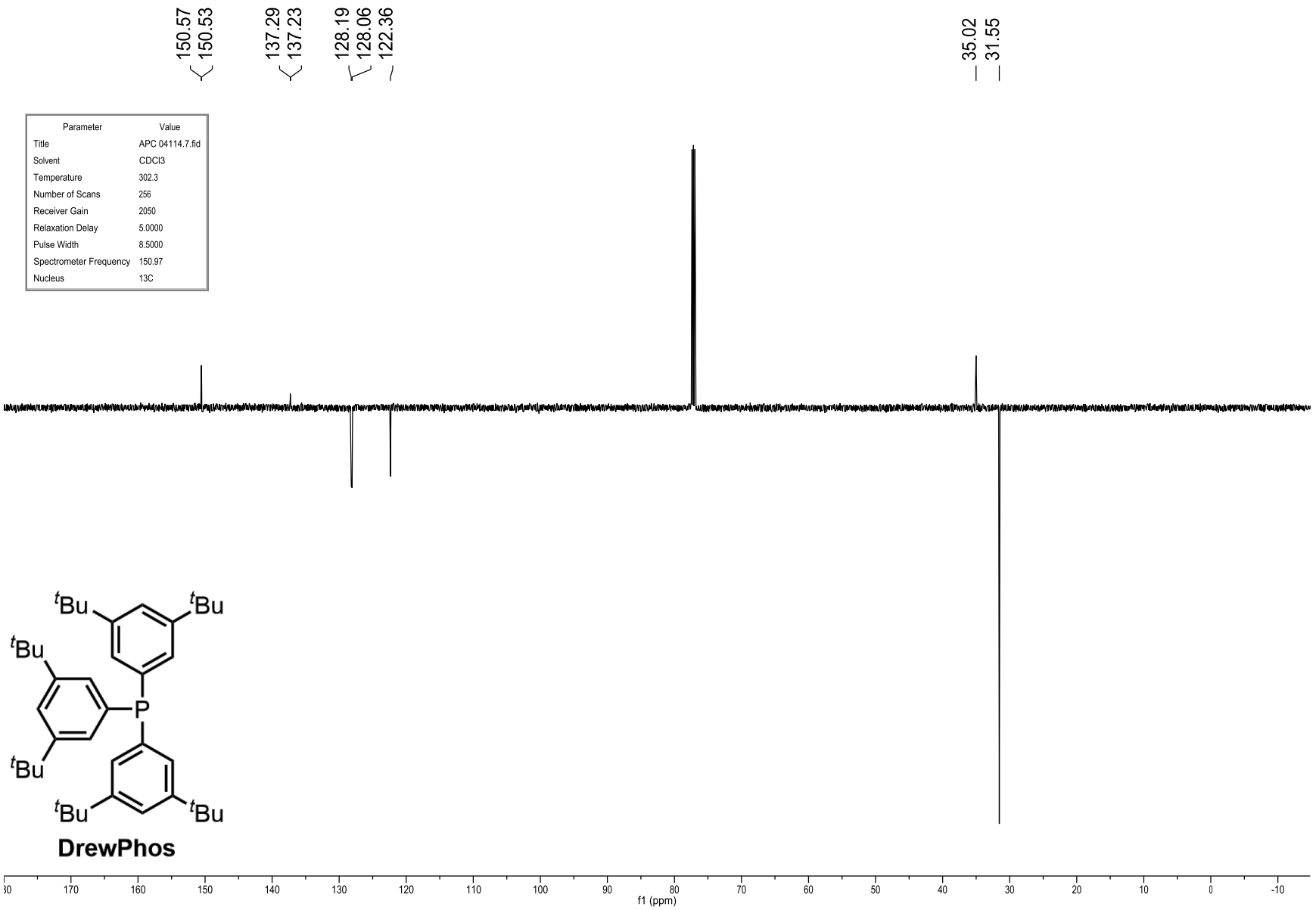


$MePh_2SiI$

-5.57

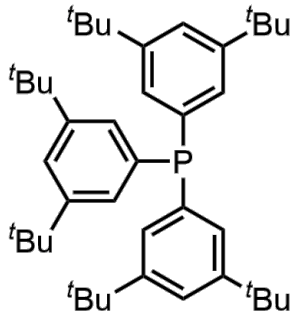



7.38
7.38
7.38
7.13
7.12
7.11
7.11
1.22
Parameter Value
Title APC 04114.3.fid
Solvent CDCl3
Temperature 297.6
Number of Scans 16
Receiver Gain 203
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
tBu
tBu
tBu
P
tBu
tBu
tBu
DrewPhos
3.00
6.11
54.76
8.5
8.0
7.5
7.0
6.5
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
-0.5
f1 (ppm)

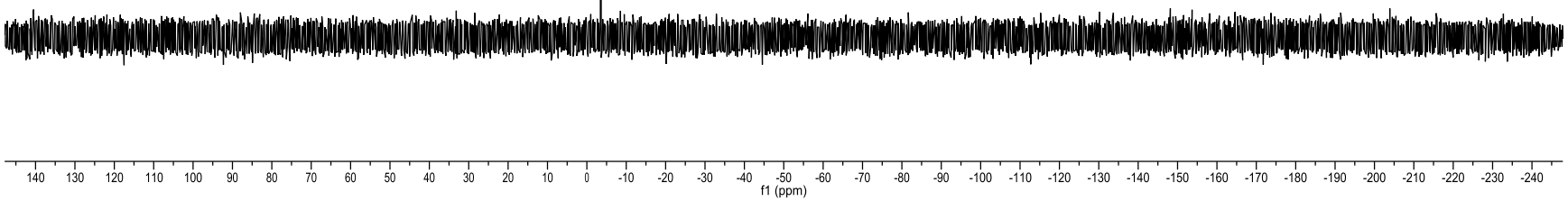

150.57
150.53
137.29
137.23
128.19
128.06
122.36
35.02
31.55
Parameter Value
Title APC 04114.7.fid
Solvent CDCl3
Temperature 302.3
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
tBu
P
DrewPhos
f1 (ppm)

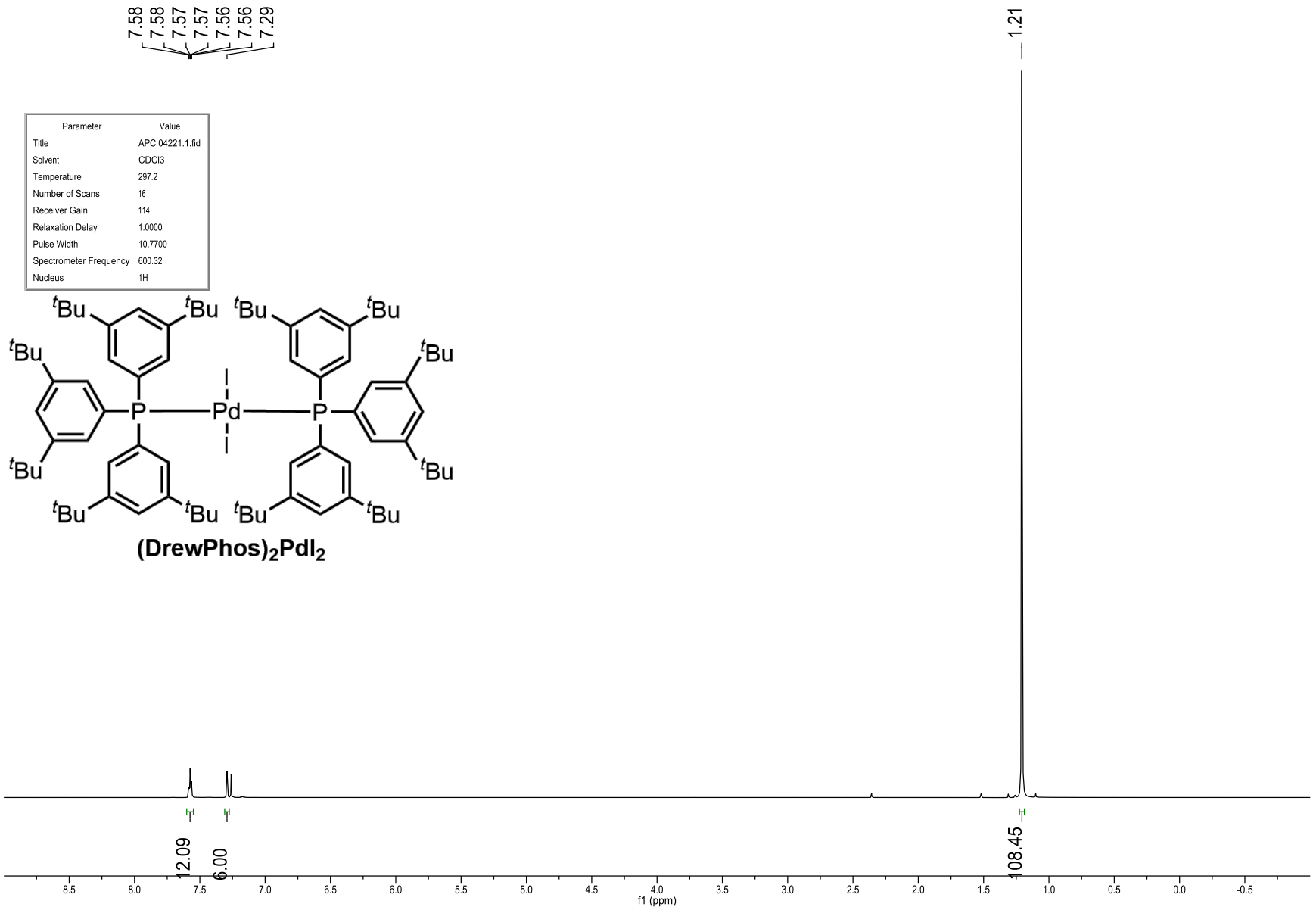
-3.59

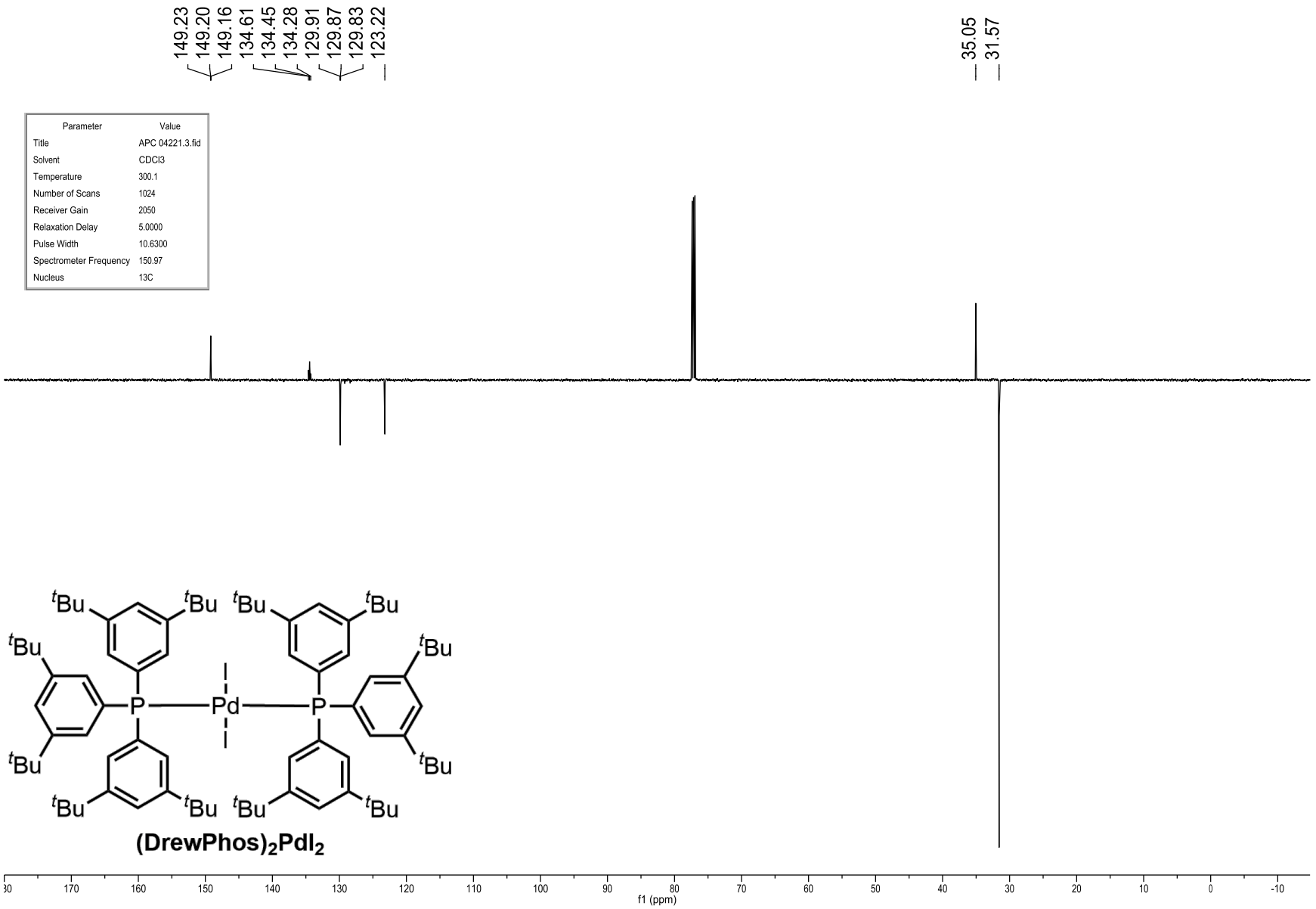
Parameter	Value
Title	APC 04114.4.fid
Solvent	CDCl3
Temperature	297.8
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P



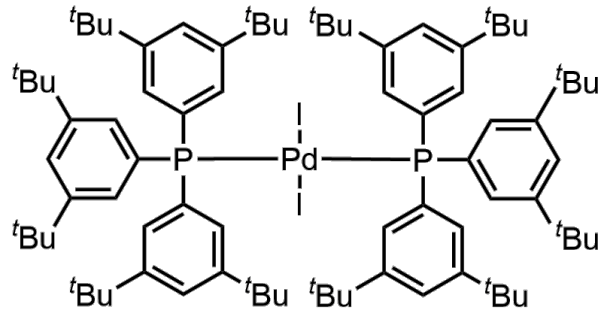
DrewPhos



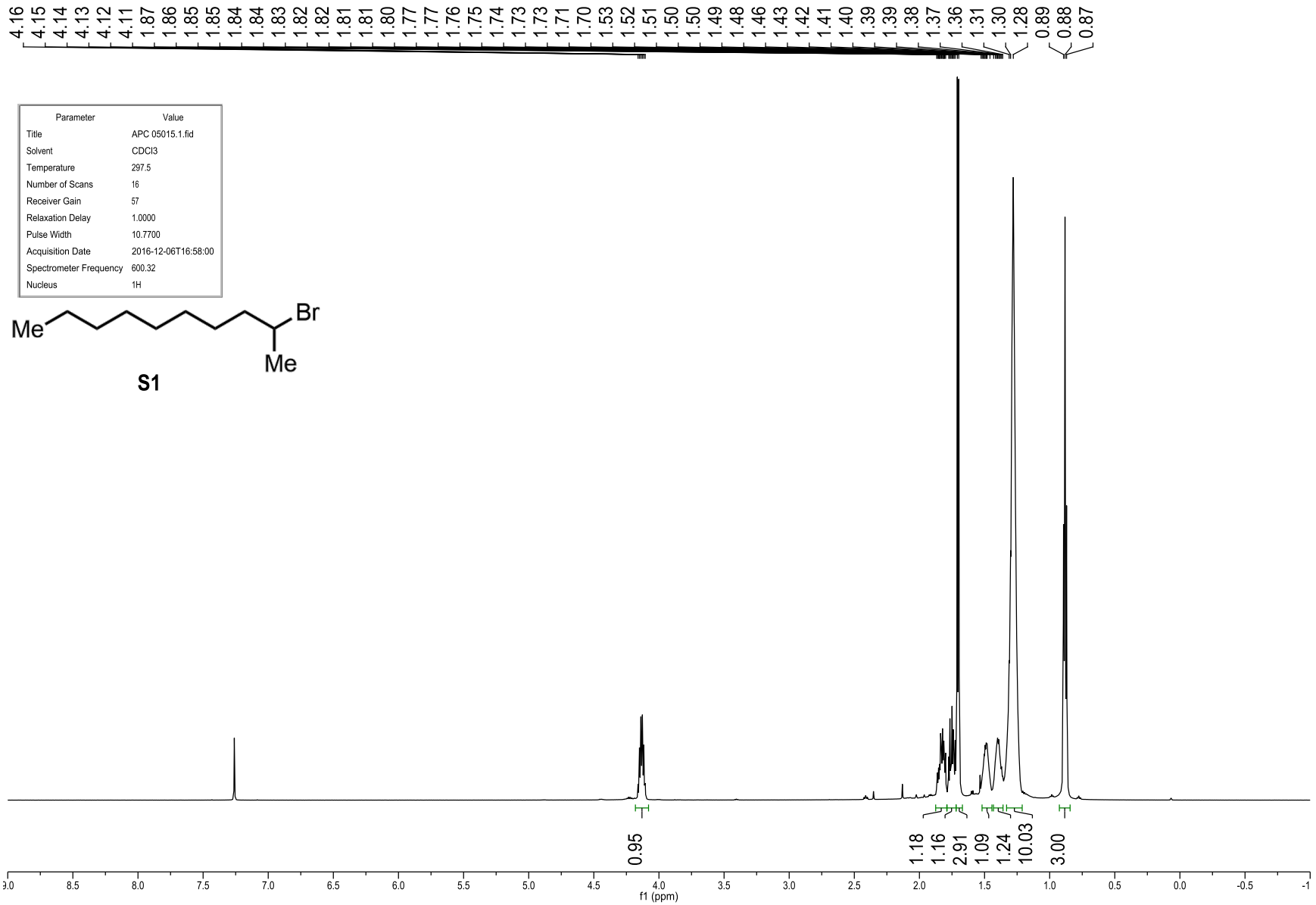

7.58
7.58
7.57
7.57
7.56
7.56
7.29
1.21
Parameter Value
Title APC 04221.1.fid
Solvent CDCl3
Temperature 297.2
Number of Scans 16
Receiver Gain 114
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
tBu
P
Pd
(DrewPhos)2PdI2
12.09
6.00
108.45
f1 (ppm)

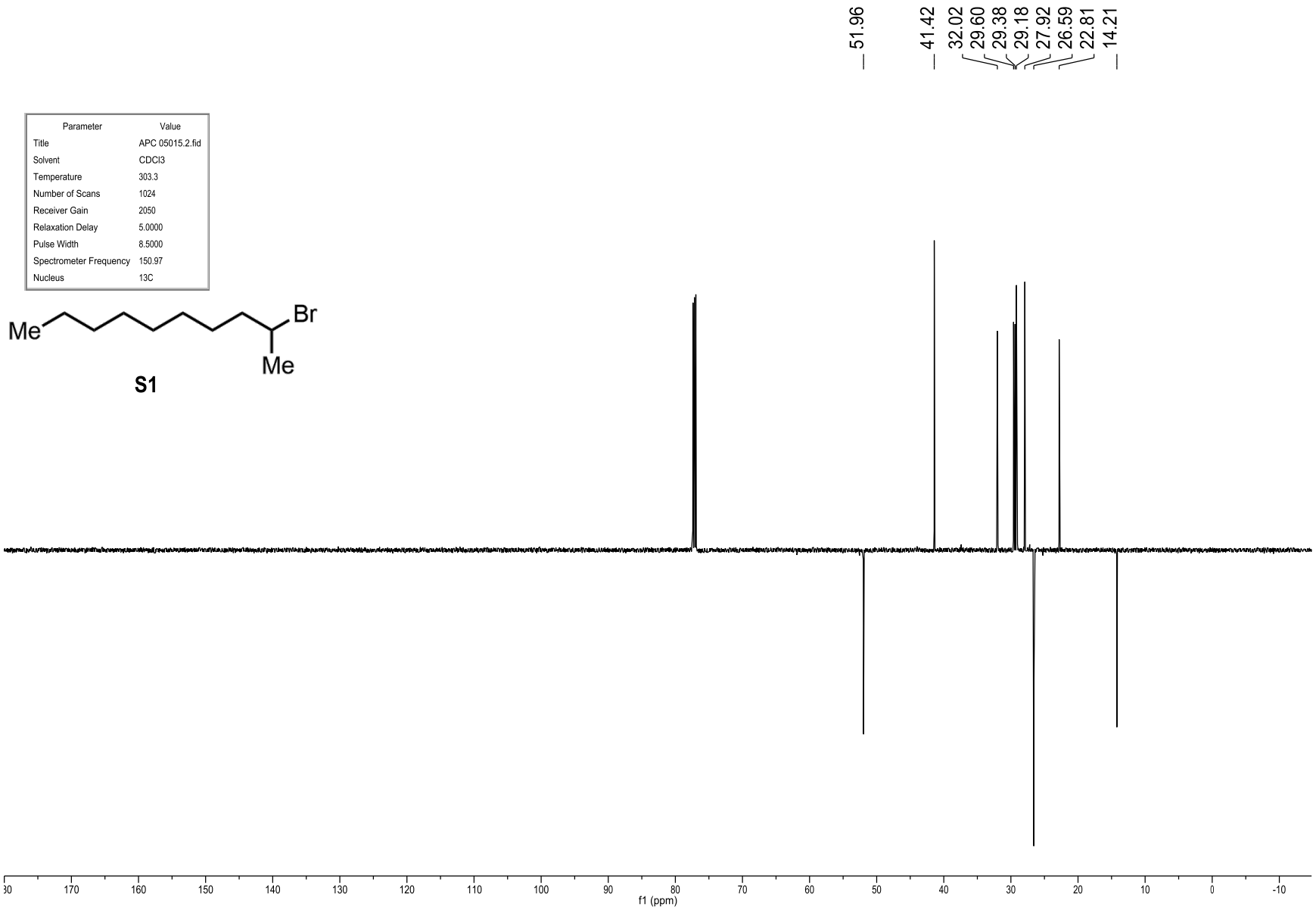

149.23
149.20
149.16
134.61
134.45
134.28
129.91
129.87
129.83
123.22
35.05
31.57
Parameter Value
Title APC 04221.3.fid
Solvent CDCl3
Temperature 300.1
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
tBu
P
Pd
(DrewPhos)2PdI2
f1 (ppm)

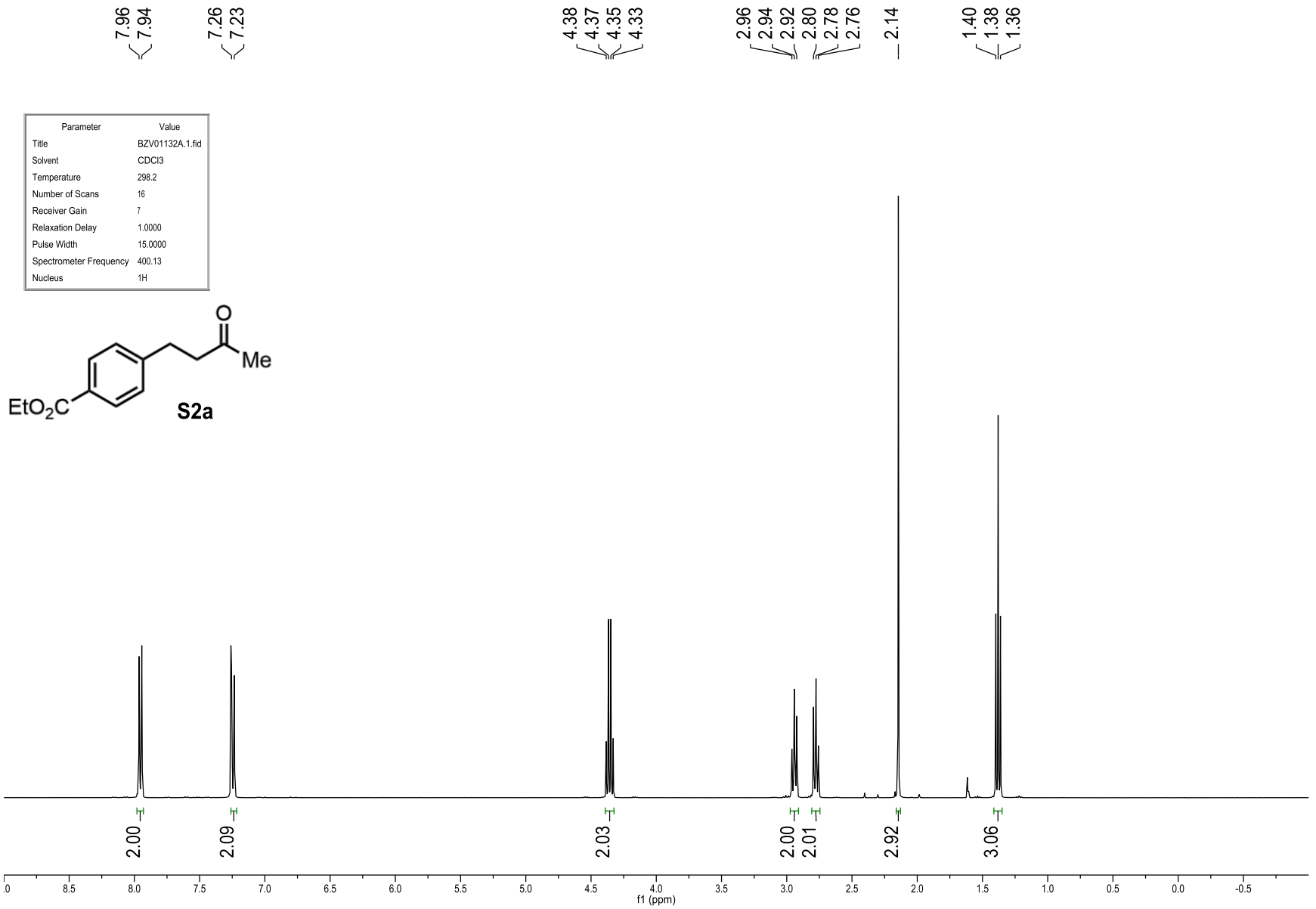
Parameter	Value
Title	APC 04221.2.fid
Solvent	CDCl3
Temperature	297.4
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P

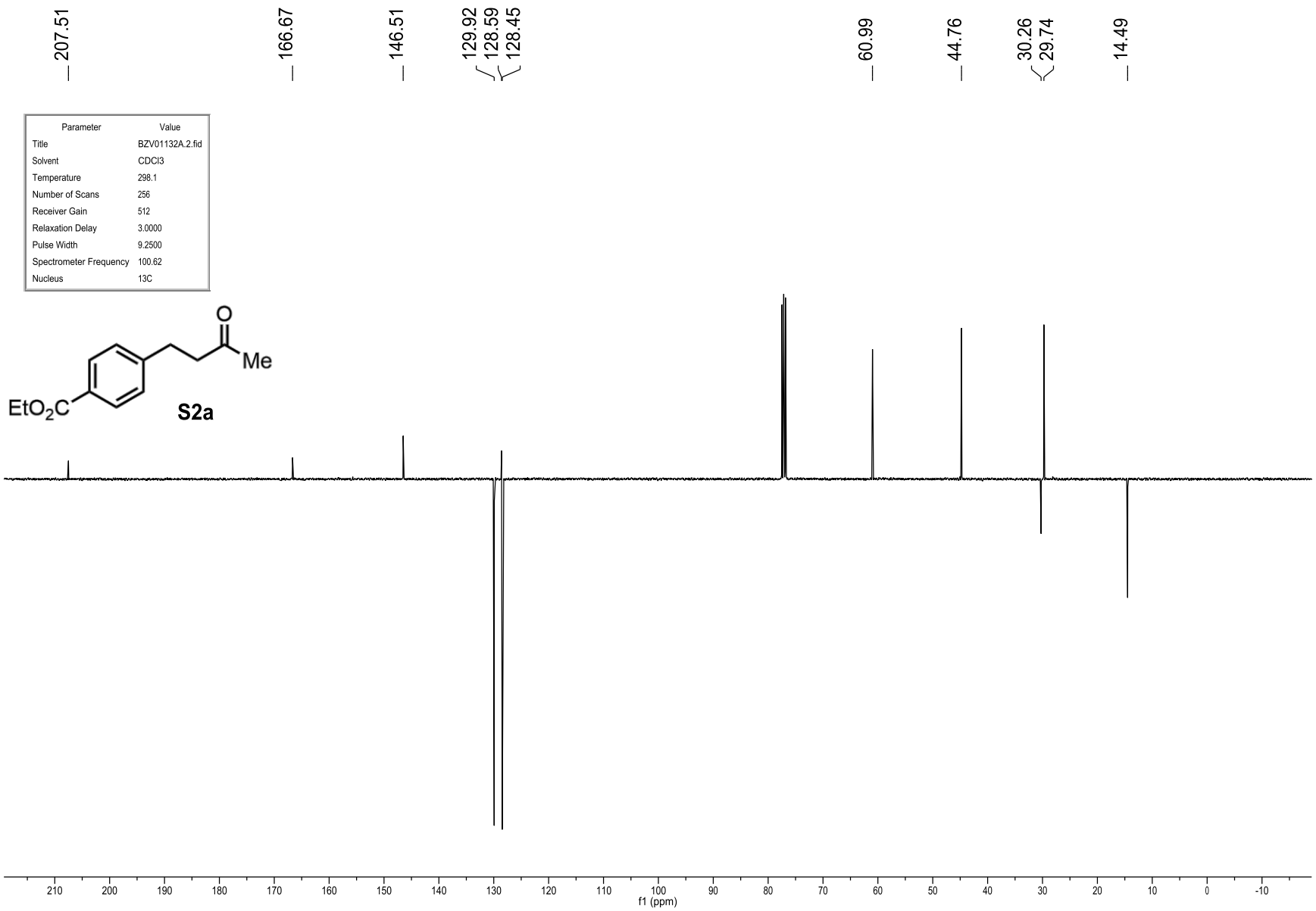


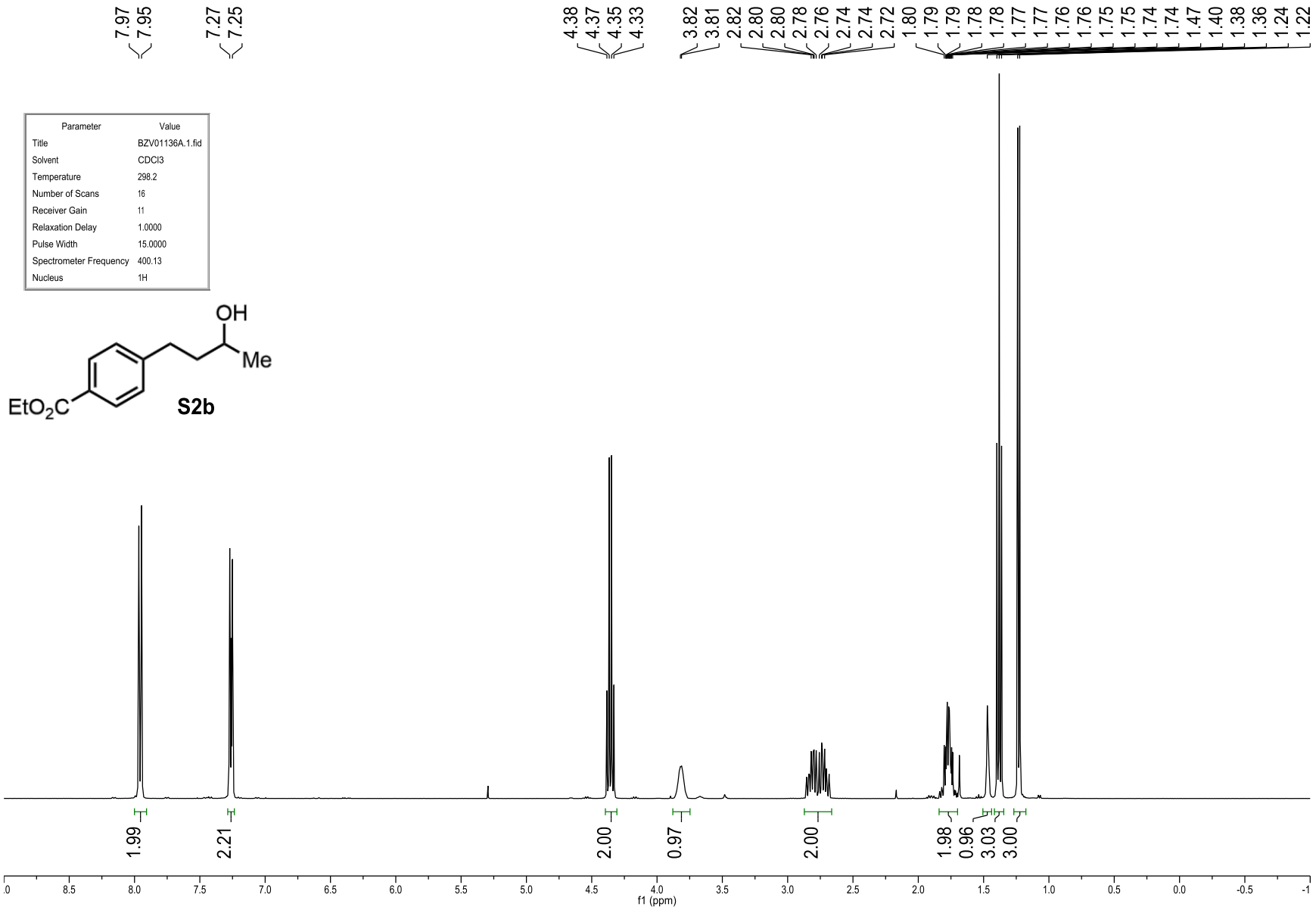
(DrewPhos)$_2$PdI$_2$

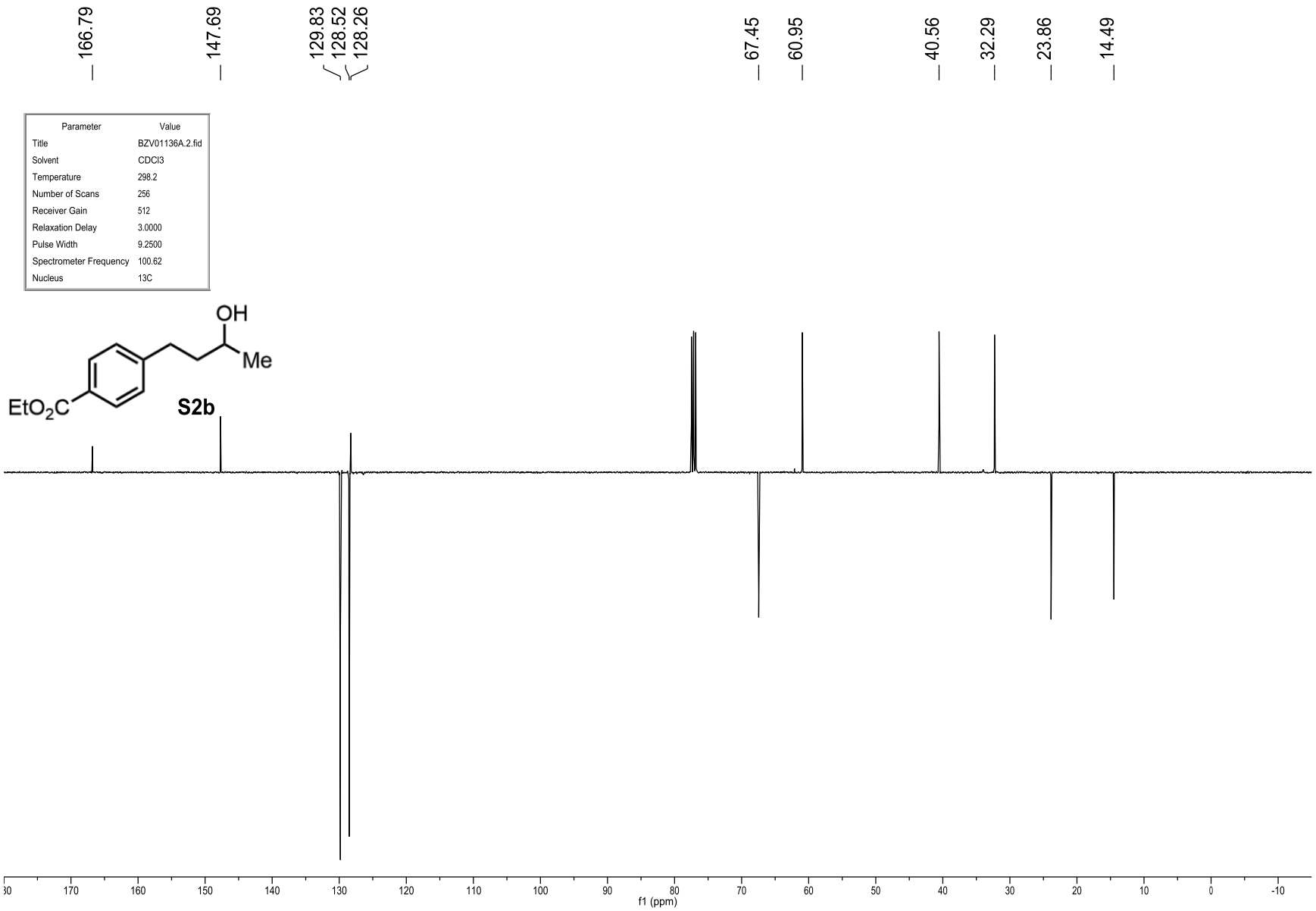

4.16 4.15 4.14 4.13 4.12 4.11 1.87 1.86 1.85 1.85 1.84 1.84 1.83 1.82 1.82 1.81 1.81 1.80 1.77 1.77 1.76 1.75 1.74 1.73 1.73 1.71 1.70 1.53 1.52 1.51 1.50 1.50 1.49 1.48 1.46 1.43 1.42 1.41 1.40 1.39 1.39 1.38 1.37 1.36 1.31 1.30 1.28 0.89 0.88 0.87
Parameter Value
Title APC 05015.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 16
Receiver Gain 57
Relaxation Delay 1.0000
Pulse Width 10.7700
Acquisition Date 2016-12-06T16:58:00
Spectrometer Frequency 600.32
Nucleus 1H
Me
Br
Me
S1
0.95
1.18
1.16
2.91
1.09
1.24
10.03
3.00
f1 (ppm)

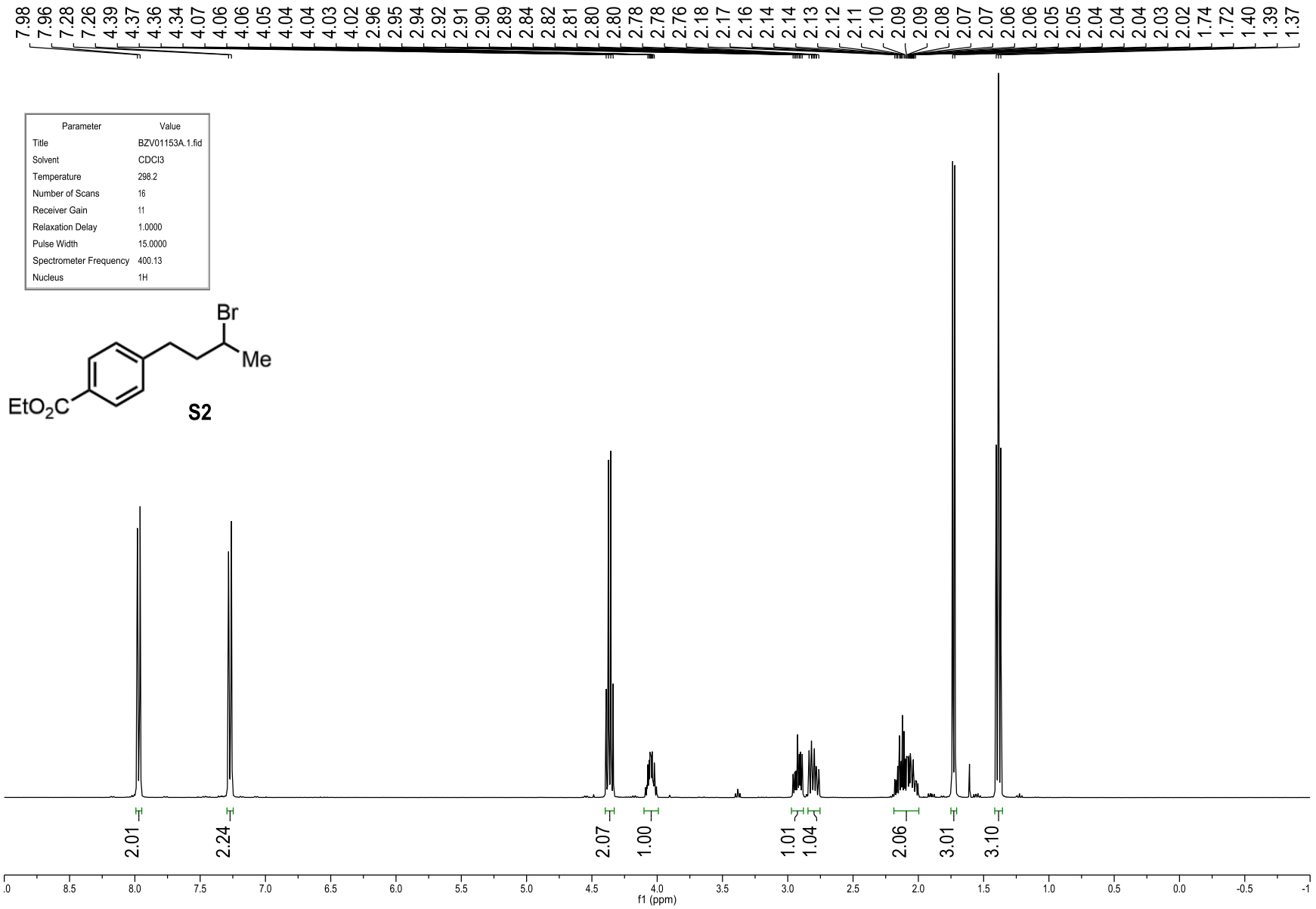

51.96
41.42
32.02
29.60
29.38
29.18
27.92
26.59
22.81
14.21
Parameter Value
Title APC 05015.2.fid
Solvent CDCl3
Temperature 303.3
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
Br
Me
S1
f1 (ppm)

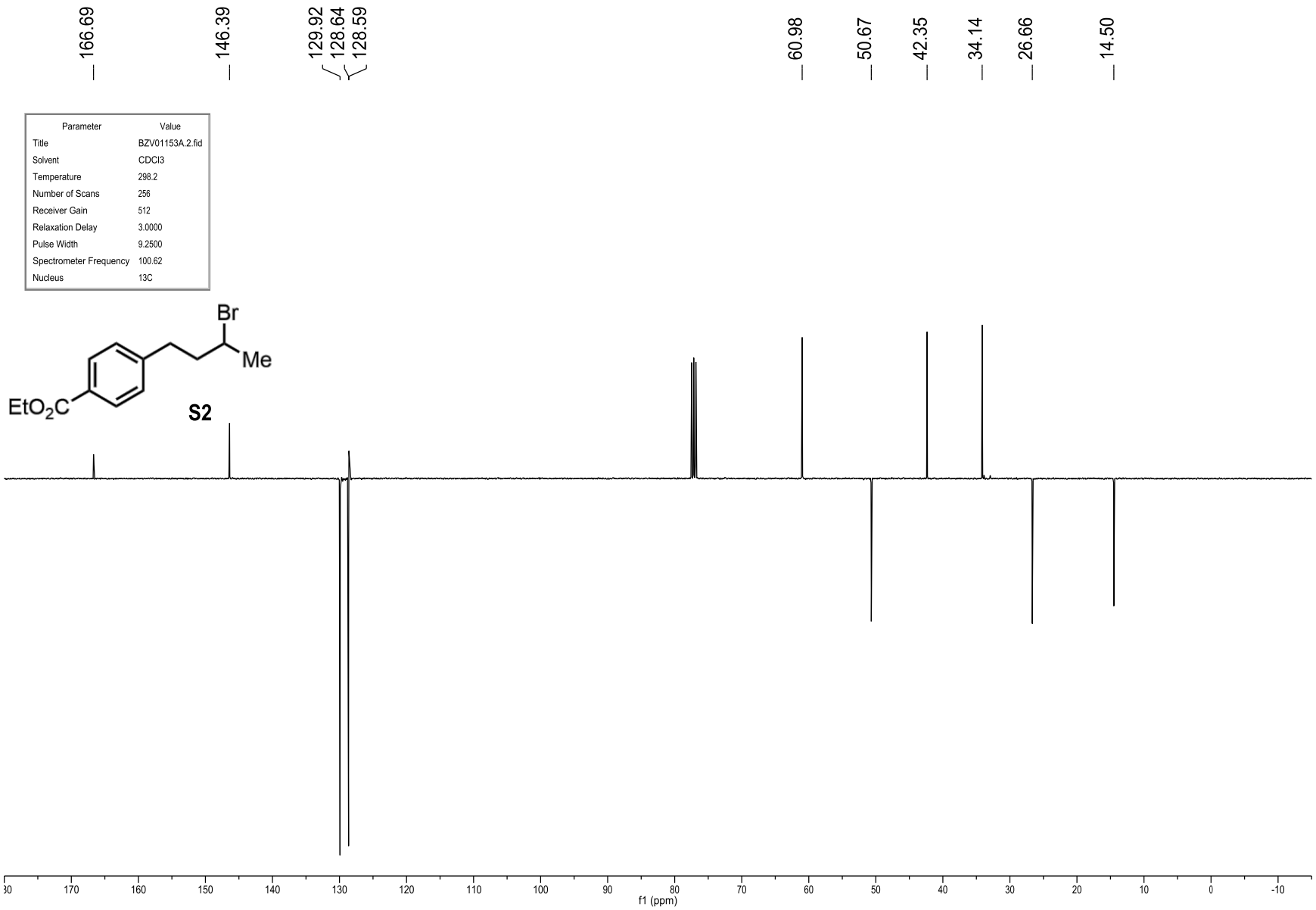

7.96
7.94
7.26
7.23
4.38
4.37
4.35
4.33
2.96
2.94
2.92
2.80
2.78
2.76
2.14
1.40
1.38
1.36
Parameter Value
Title BZV01132A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 7
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
O
Me
EtO2C
S2a
2.00
2.09
2.03
2.00
2.01
2.92
3.06
f1 (ppm)

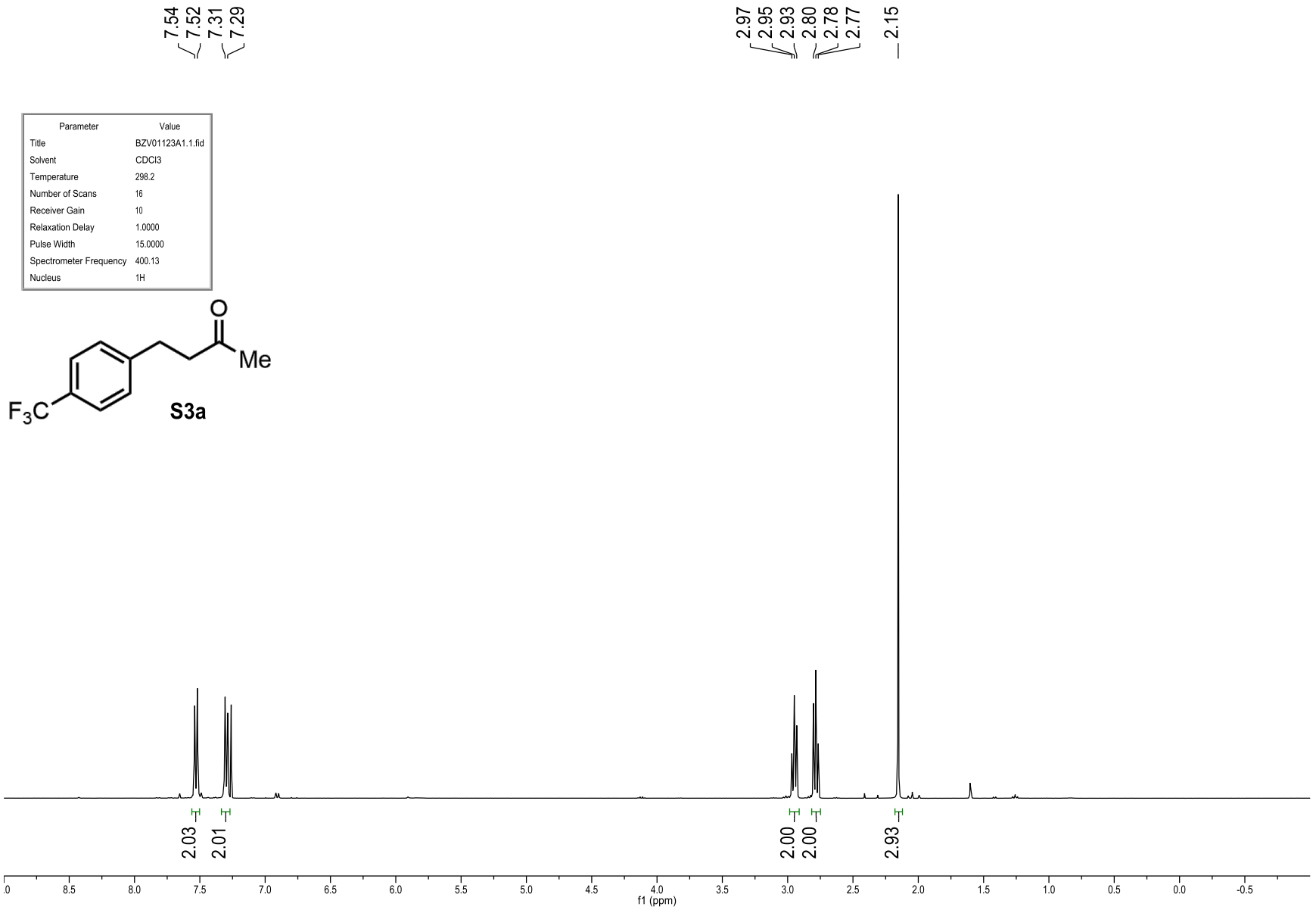

207.51
166.67
146.51
129.92
128.59
128.45
60.99
44.76
30.26
29.74
14.49
Parameter Value
Title BZV01132A.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
O
Me
EtO2C
S2a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

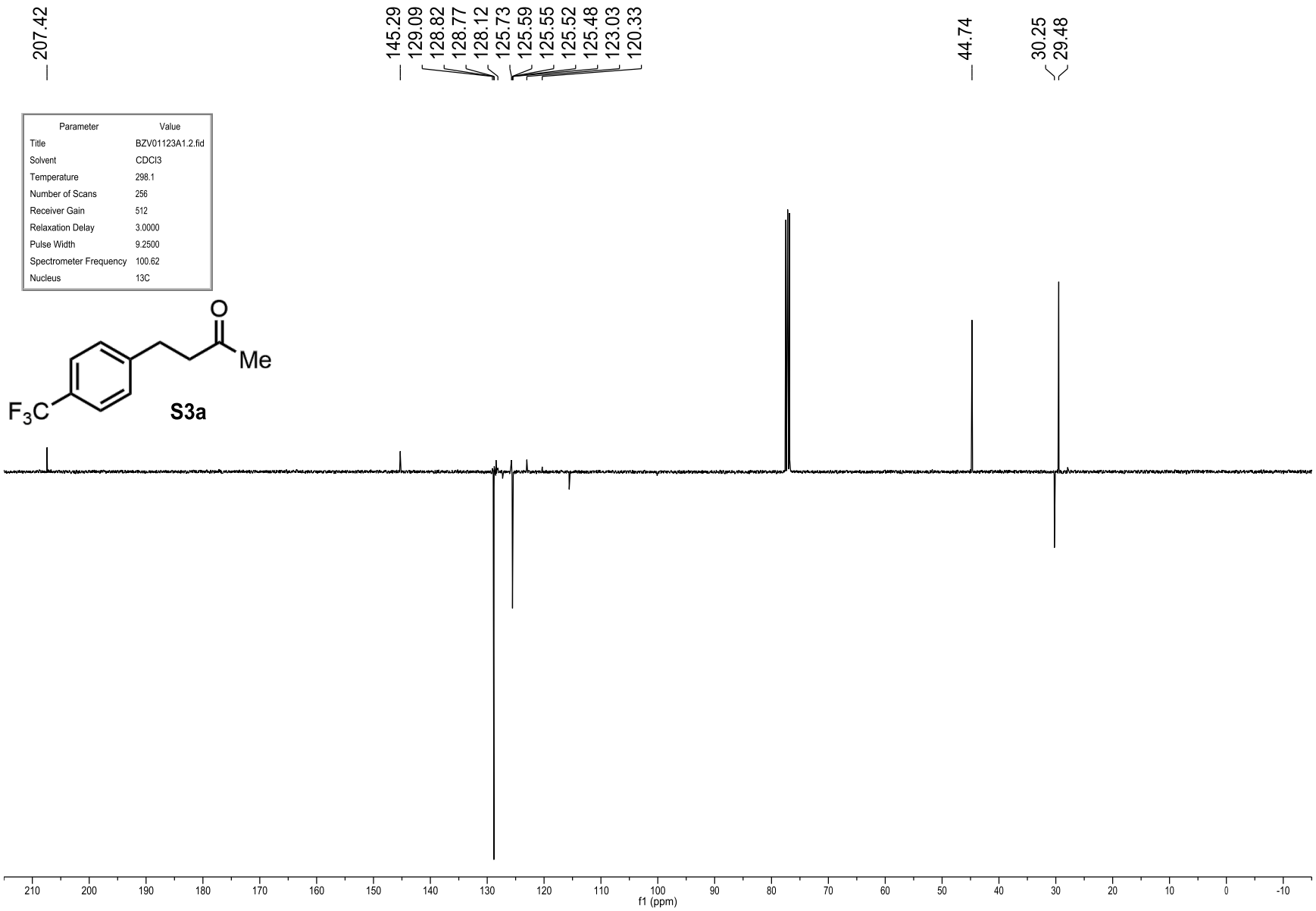

7.97
7.95
7.27
7.25
4.38
4.37
4.35
4.33
3.82
3.81
2.82
2.80
2.80
2.78
2.76
2.74
2.74
2.72
1.80
1.79
1.79
1.78
1.78
1.77
1.77
1.76
1.76
1.75
1.75
1.74
1.74
1.47
1.40
1.38
1.36
1.24
1.22
Parameter Value
Title BZV01136A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
OH
Me
EtO2C
S2b
1.99
2.21
2.00
0.97
2.00
1.98
0.96
3.03
3.00
f1 (ppm)


166.79
147.69
129.83
128.52
128.26
67.45
60.95
40.56
32.29
23.86
14.49
Parameter Value
Title BZV01136A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
OH
Me
EtO2C
S2b
f1 (ppm)

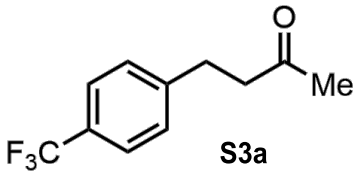

7.98 7.96 7.28 7.26 4.39 4.37 4.36 4.34 4.07 4.06 4.06 4.05 4.04 4.04 4.03 4.02 2.96 2.95 2.94 2.92 2.91 2.90 2.89 2.84 2.82 2.81 2.80 2.80 2.78 2.78 2.76 2.18 2.17 2.16 2.14 2.14 2.13 2.12 2.11 2.10 2.09 2.09 2.08 2.07 2.07 2.06 2.06 2.05 2.05 2.04 2.04 2.04 2.03 2.02 1.74 1.72 1.40 1.39 1.37
Parameter Value
Title BZV01153A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Br
Me
EtO2C
S2
2.01 2.24 2.07 1.00 1.01 1.04 2.06 3.01 3.10
f1 (ppm)


166.69
146.39
129.92
128.64
128.59
60.98
50.67
42.35
34.14
26.66
14.50
Parameter Value
Title BZV01153A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Br
Me
EtO2C
S2
f1 (ppm)

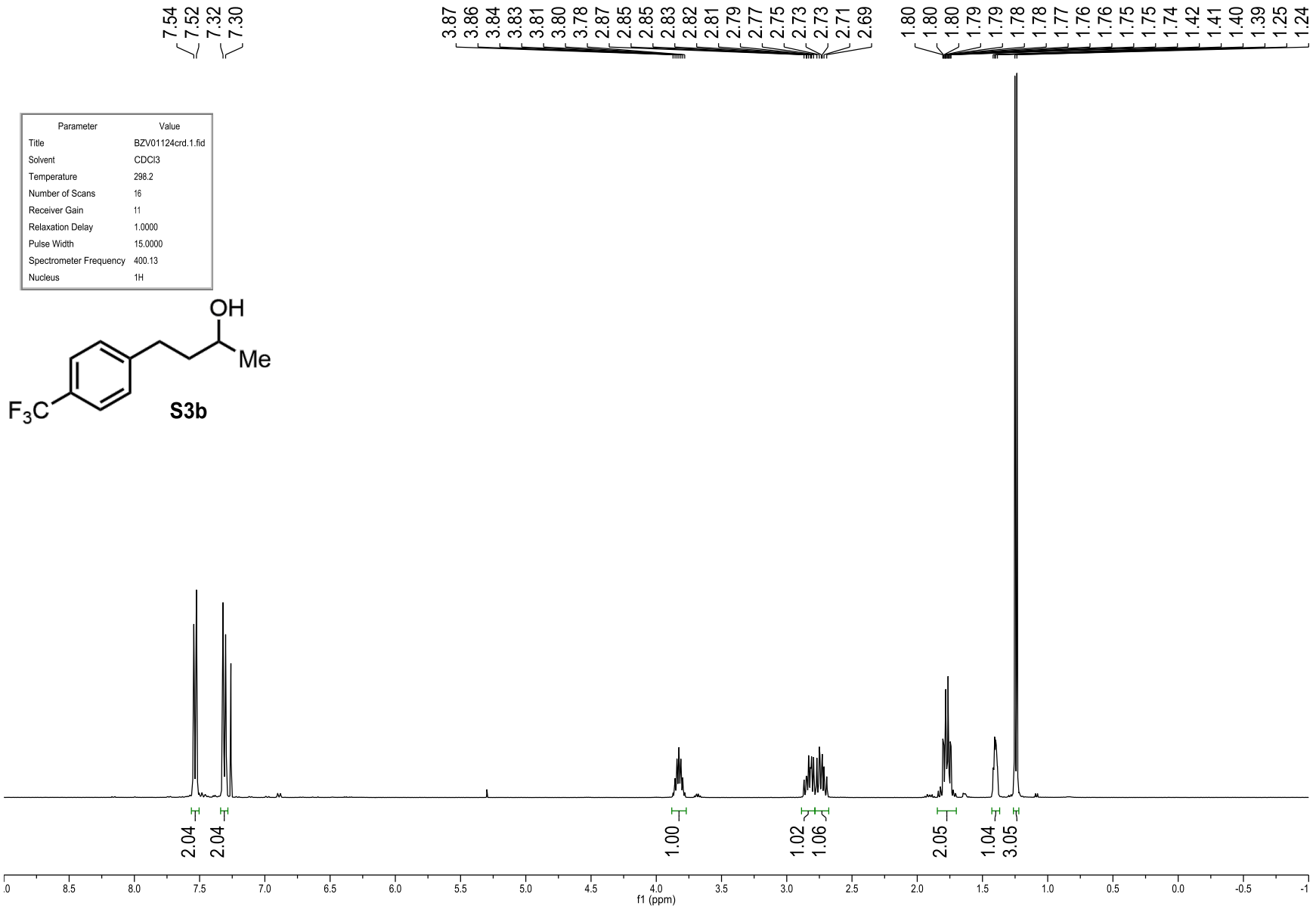

7.54
7.52
7.31
7.29
2.97
2.95
2.93
2.80
2.78
2.77
2.15
Parameter Value
Title BZV01123A1.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 10
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
O
Me
F3C
S3a
2.03
2.01
2.00
2.00
2.93
f1 (ppm)

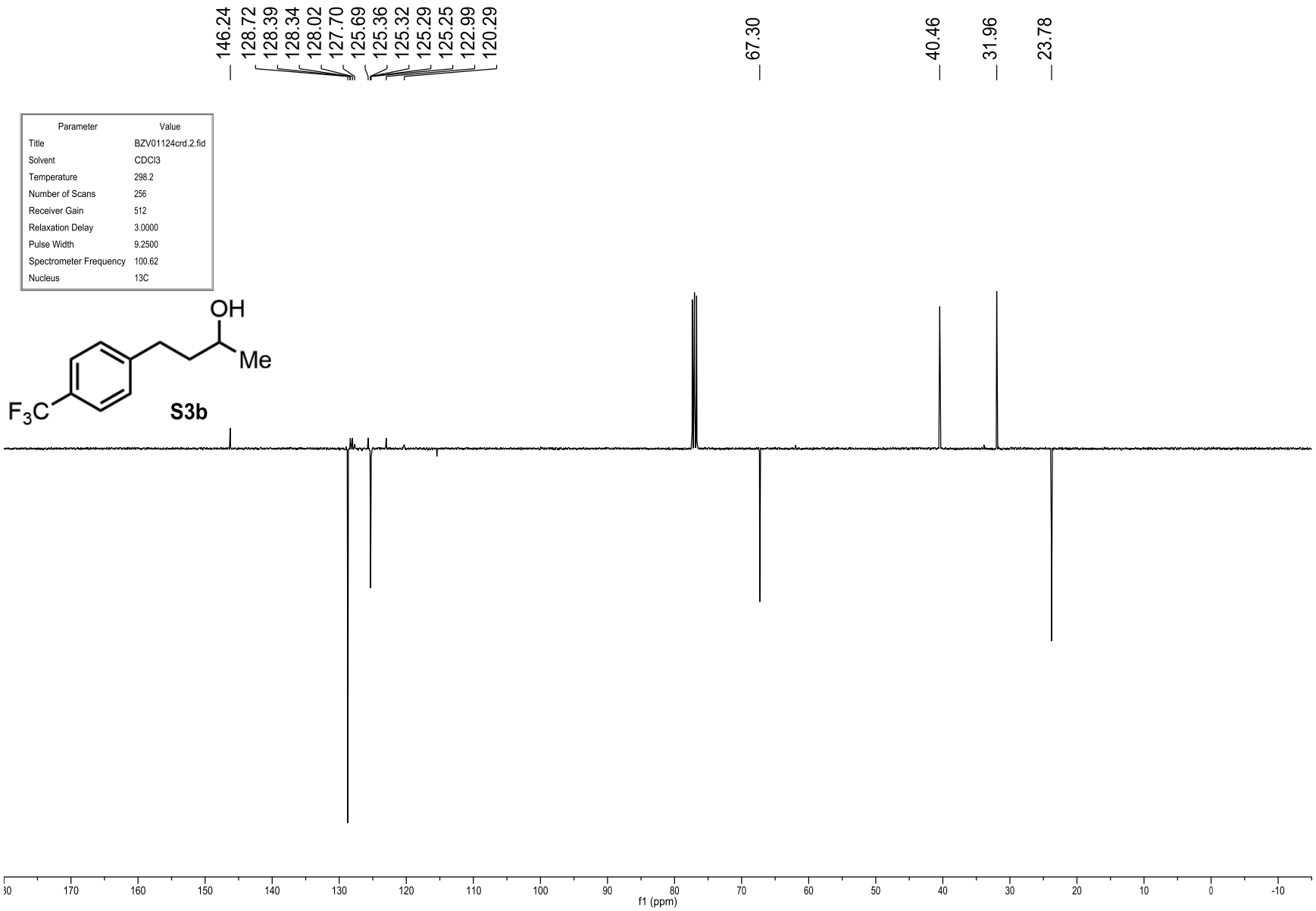

207.42
145.29
129.09
128.82
128.77
128.12
125.73
125.59
125.55
125.52
125.48
123.03
120.33
44.74
30.25
29.48
Parameter Value
Title BZV01123A1.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
O
Me
F3C
S3a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Parameter	Value
Title	BZV01123A1.7.fid
Solvent	CDCl3
Temperature	298.2
Number of Scans	16
Receiver Gain	813
Relaxation Delay	2.0000
Pulse Width	15.0300
Spectrometer Frequency	376.46
Nucleus	19F



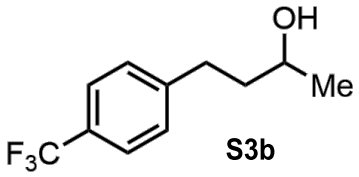
S3a


7.54
7.52
7.32
7.30
3.87
3.86
3.84
3.83
3.81
3.80
3.78
2.87
2.85
2.85
2.83
2.82
2.81
2.79
2.77
2.75
2.73
2.73
2.71
2.69
1.80
1.80
1.80
1.79
1.79
1.78
1.78
1.77
1.76
1.76
1.75
1.75
1.74
1.42
1.41
1.40
1.39
1.25
1.24
Parameter Value
Title BZV01124crd.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
OH
Me
F3C
S3b
2.04
2.04
1.00
1.02
1.06
2.05
1.04
3.05
f1 (ppm)

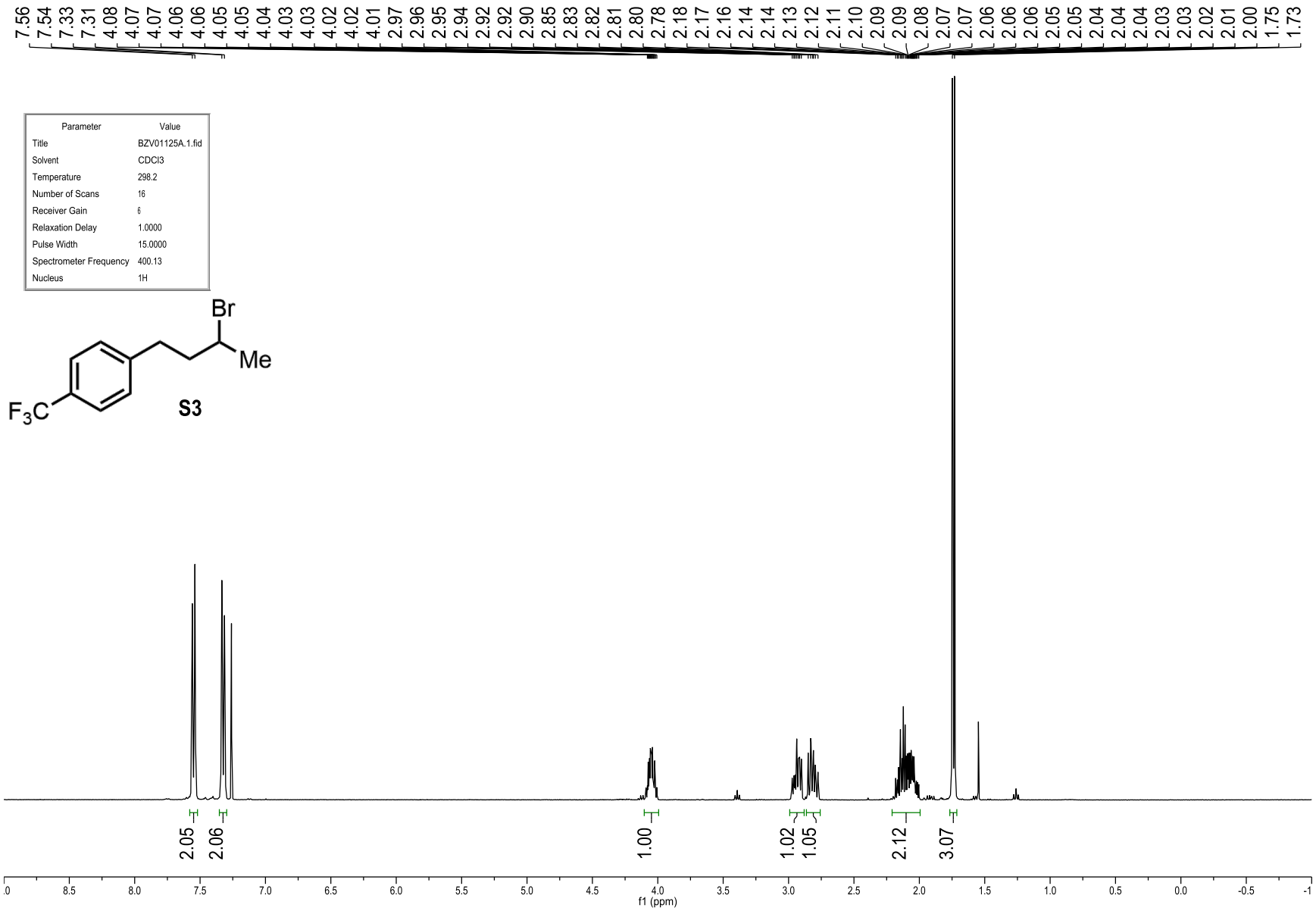

146.24
128.72
128.39
128.34
128.02
127.70
125.69
125.36
125.32
125.29
125.25
122.99
120.29
67.30
40.46
31.96
23.78
Parameter Value
Title BZV01124crd.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
OH
Me
F3C
S3b
f1 (ppm)

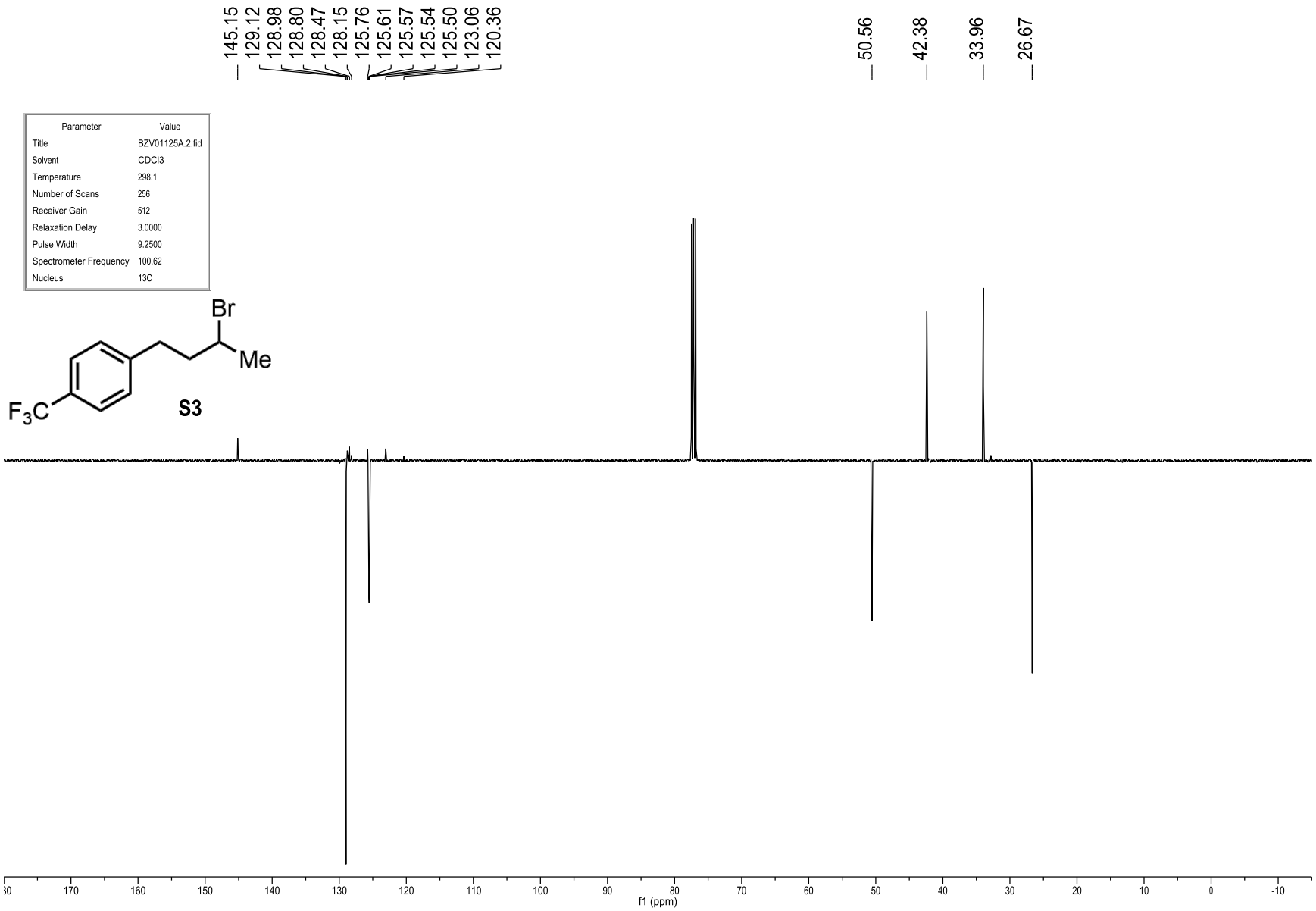
-62.29

Parameter	Value
Title	BZV01124crd.5.fid
Solvent	CDCl3
Temperature	298.2
Number of Scans	16
Receiver Gain	575
Relaxation Delay	2.0000
Pulse Width	15.0300
Spectrometer Frequency	376.46
Nucleus	19F

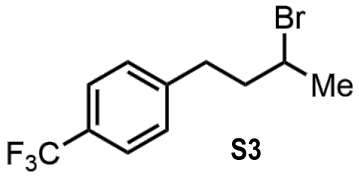


S3b

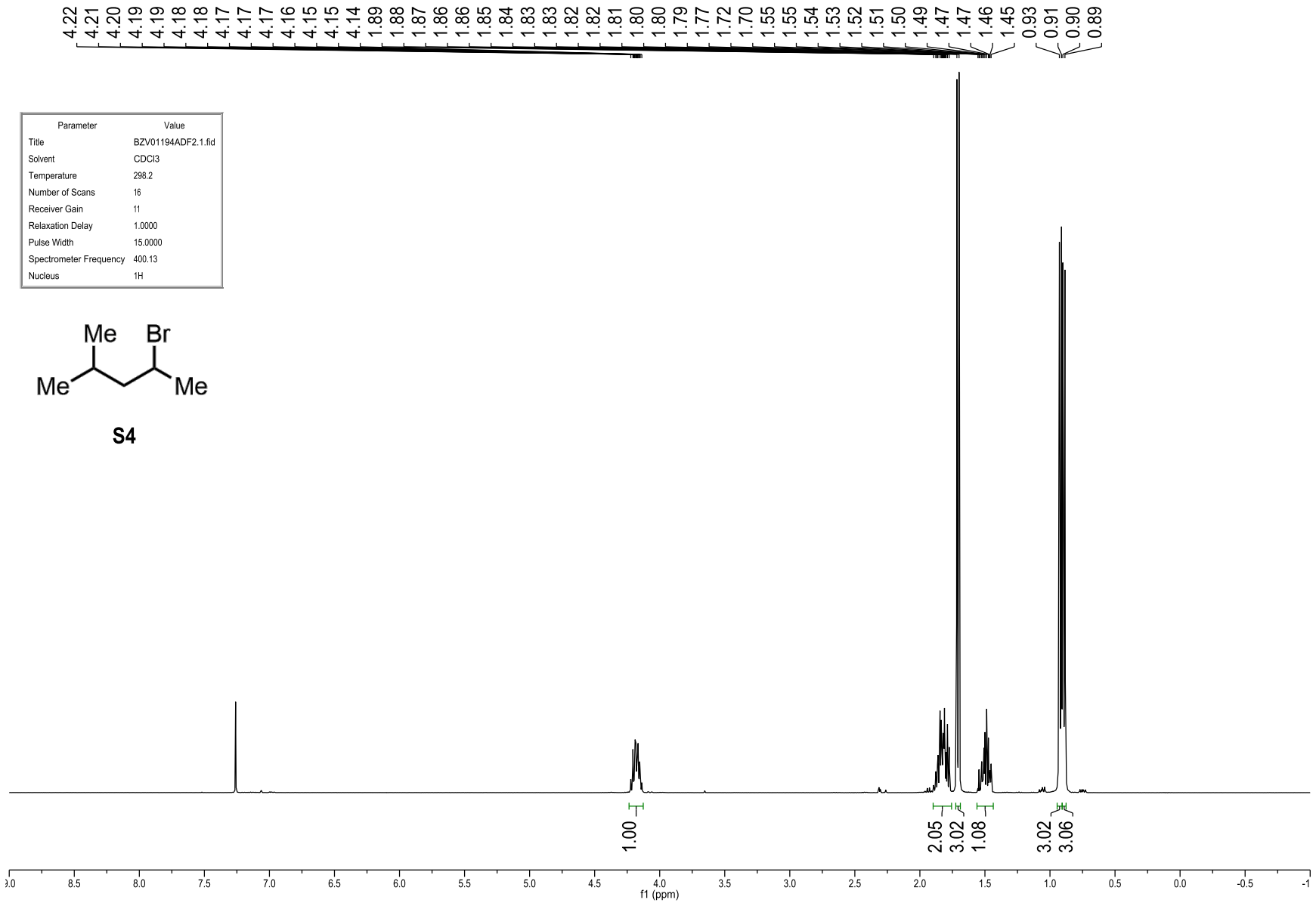

7.56 7.54 7.33 7.31 4.08 4.07 4.07 4.06 4.06 4.05 4.05 4.04 4.03 4.03 4.02 4.02 4.01 2.97 2.96 2.95 2.94 2.92 2.92 2.90 2.85 2.83 2.82 2.81 2.80 2.78 2.18 2.17 2.16 2.14 2.14 2.13 2.12 2.11 2.10 2.09 2.09 2.08 2.07 2.07 2.06 2.06 2.06 2.05 2.05 2.04 2.04 2.04 2.03 2.03 2.02 2.01 2.00 1.75 1.73
Parameter Value
Title BZV01125A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 6
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Br
Me
F3C
S3
2.05 2.06 1.00 1.02 1.05 2.12 3.07
f1 (ppm)


145.15
129.12
128.98
128.80
128.47
128.15
125.76
125.61
125.57
125.54
125.50
123.06
120.36
50.56
42.38
33.96
26.67
Parameter Value
Title BZV01125A.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Br
Me
F3C
S3
f1 (ppm)

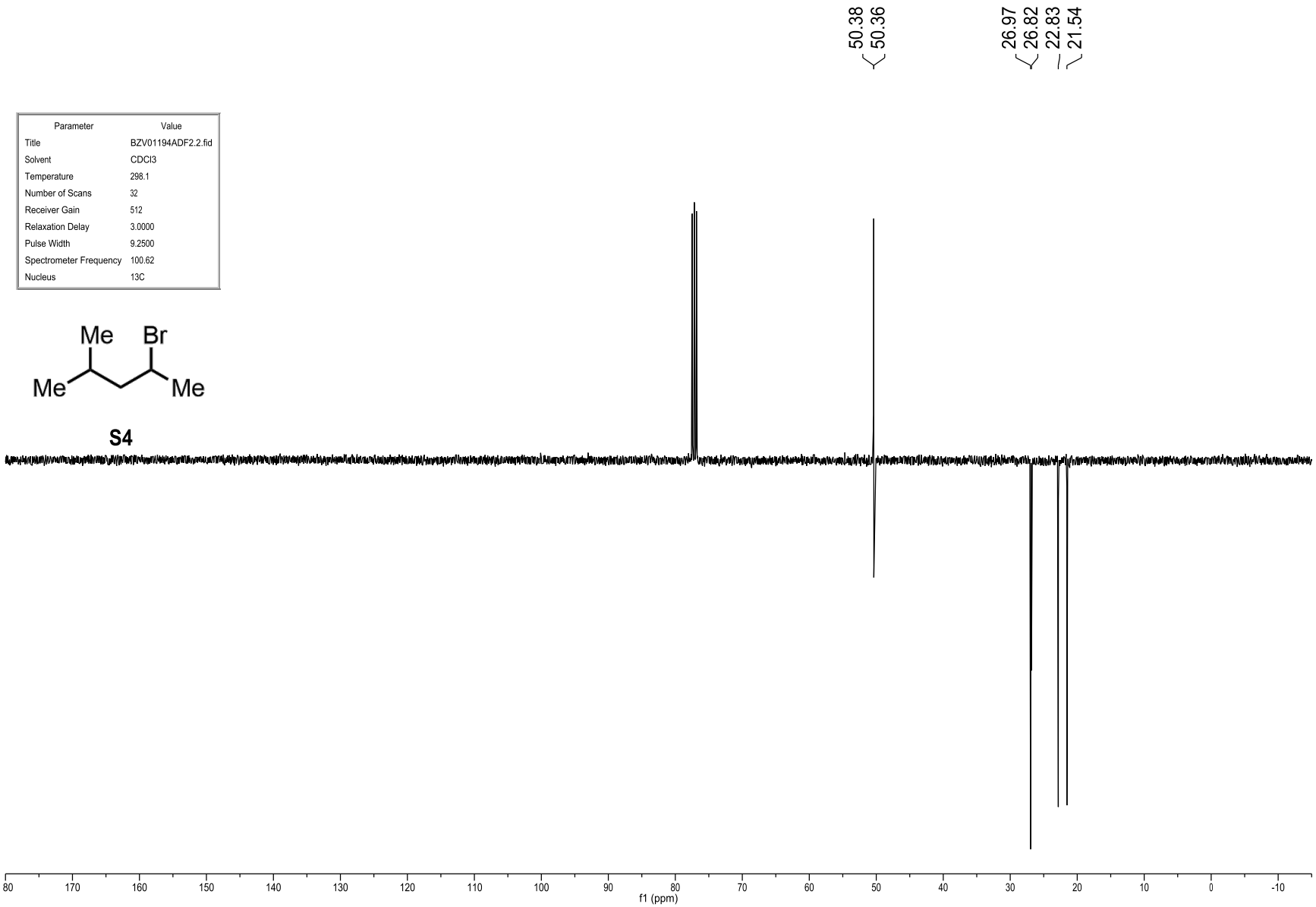
Parameter	Value
Title	BZV01125A.5.fid
Solvent	CDCl3
Temperature	298.1
Number of Scans	16
Receiver Gain	645
Relaxation Delay	2.0000
Pulse Width	15.0300
Spectrometer Frequency	376.46
Nucleus	19F

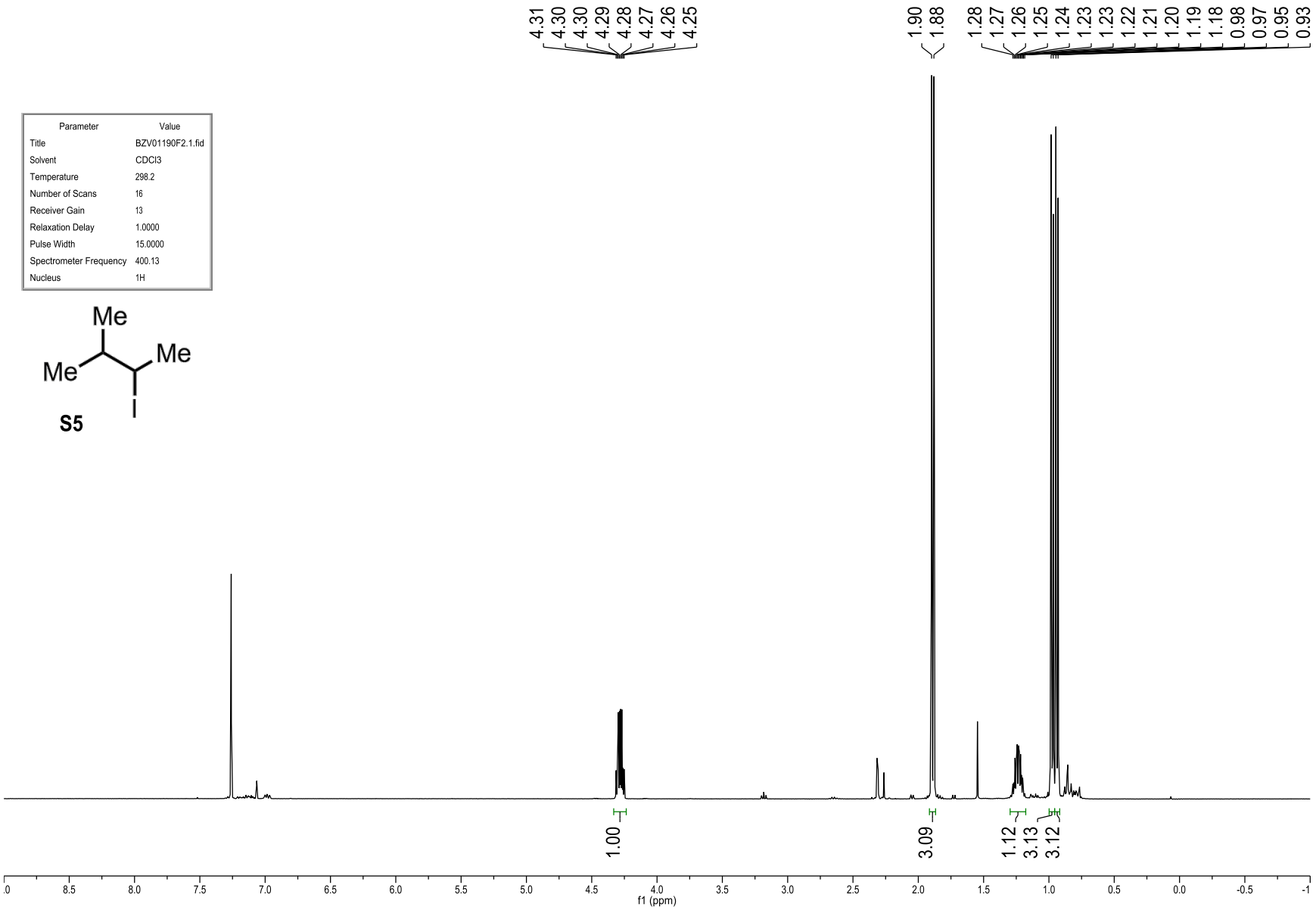


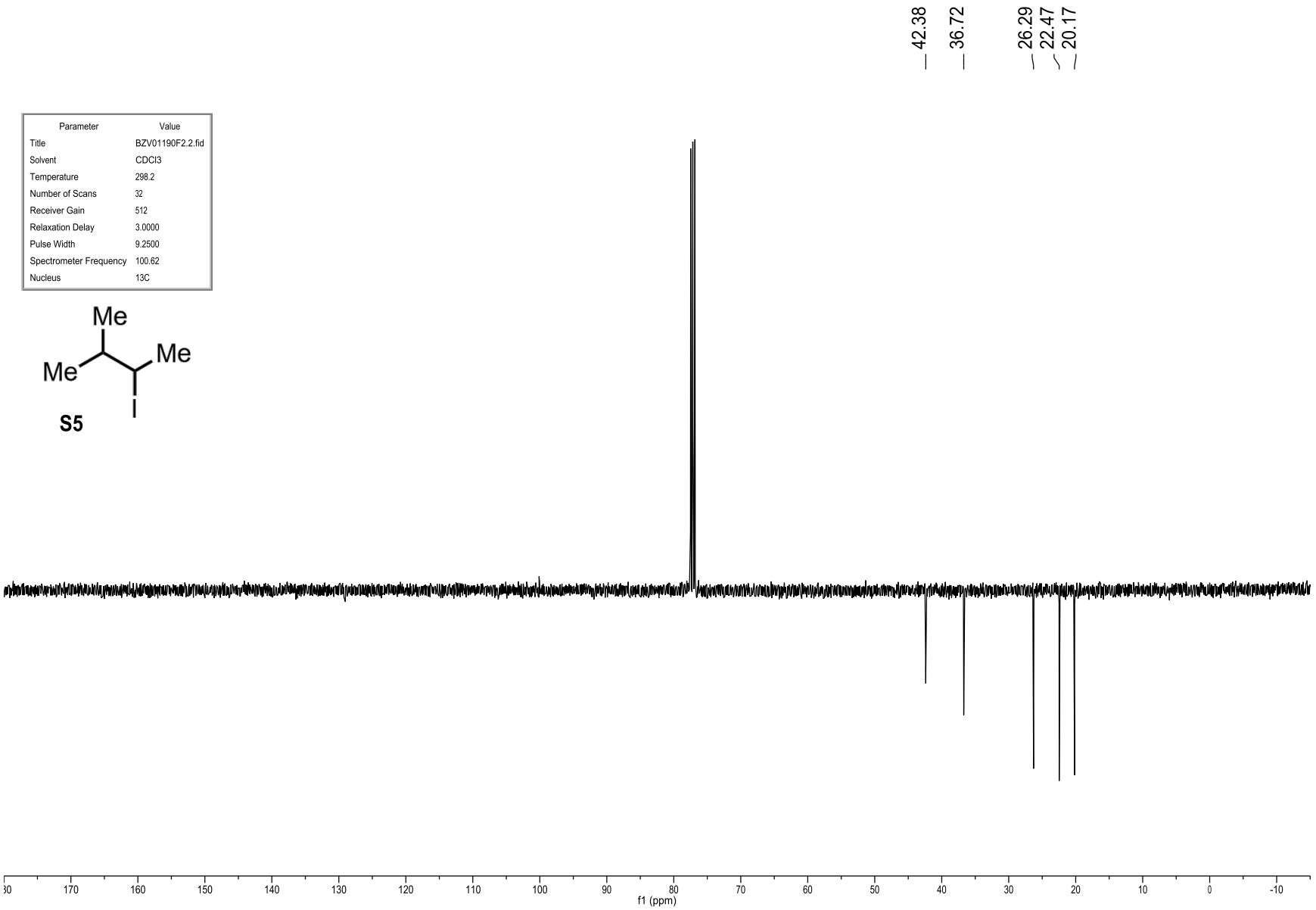
S3


4.22 4.21 4.20 4.19 4.19 4.18 4.18 4.17 4.17 4.17 4.16 4.15 4.15 4.14 1.89 1.88 1.87 1.86 1.86 1.85 1.84 1.83 1.83 1.82 1.82 1.81 1.80 1.80 1.79 1.77 1.72 1.70 1.55 1.55 1.54 1.53 1.52 1.51 1.50 1.49 1.47 1.47 1.46 1.45 0.93 0.91 0.90 0.89
Parameter Value
Title BZV01194ADF2.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me Br Me Me
1.00 2.05 3.02 1.08 3.02 3.06
f1 (ppm)

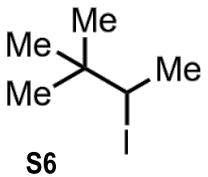
S4


50.38
50.36
26.97
26.82
22.83
21.54
Parameter Value
Title BZV01194ADF2.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 32
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Br
Me
Me
S4
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

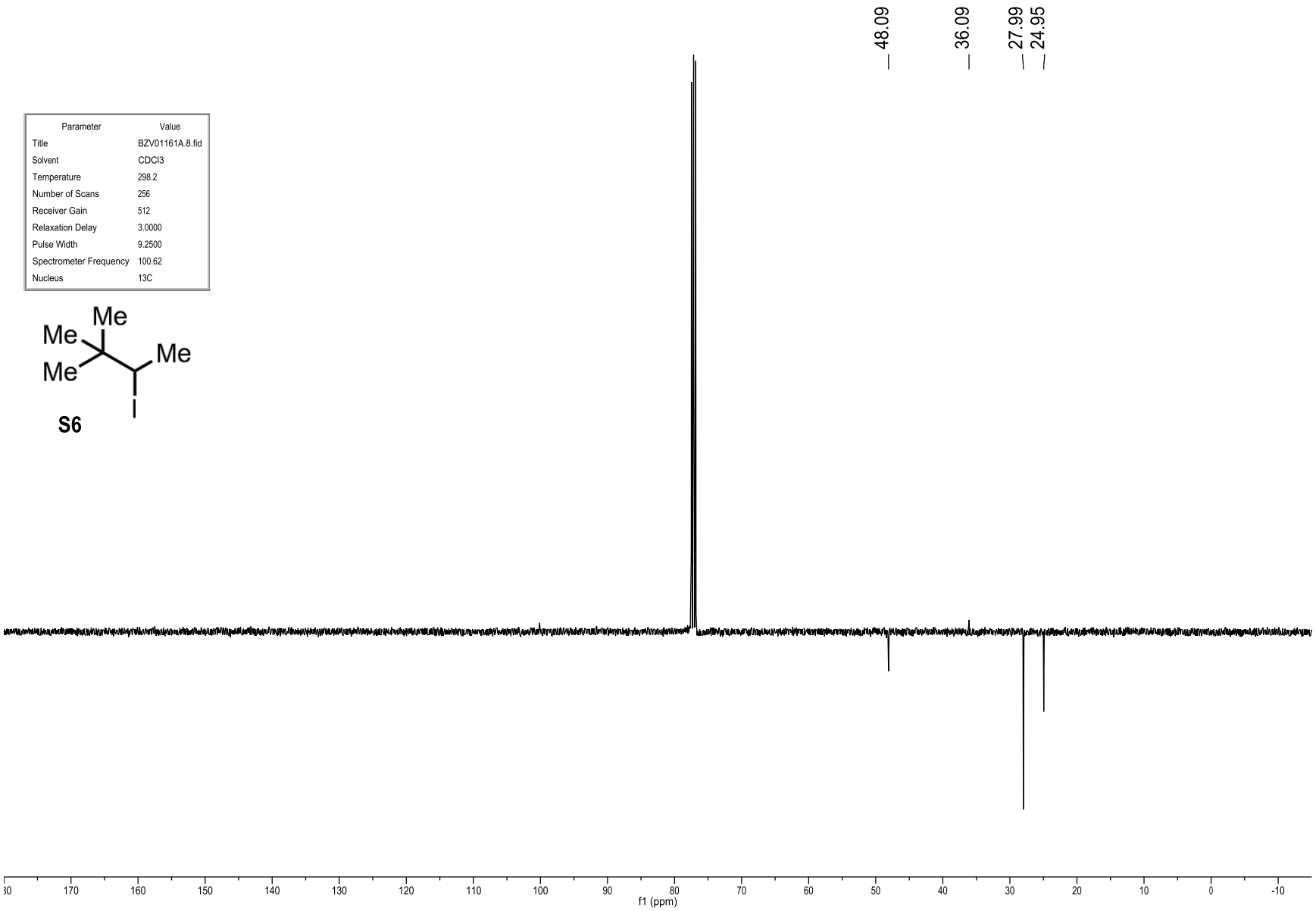

4.31
4.30
4.30
4.29
4.28
4.27
4.26
4.25
1.90
1.88
1.28
1.27
1.26
1.25
1.24
1.23
1.23
1.22
1.21
1.20
1.19
1.18
0.98
0.97
0.95
0.93
Parameter Value
Title BZV01190F2.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 13
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
Me
I
S5
1.00
3.09
1.12
3.13
3.12
f1 (ppm)

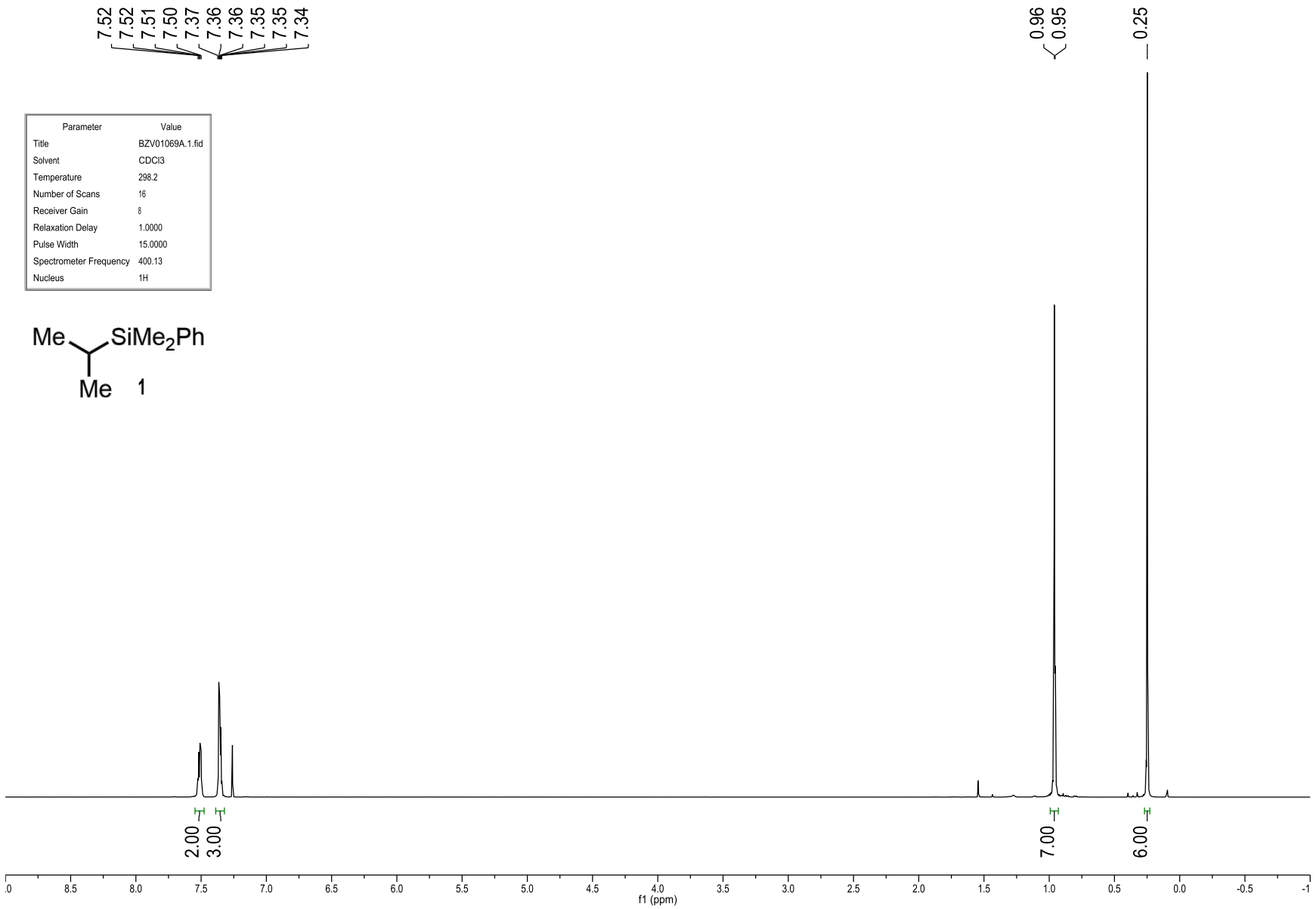

42.38
36.72
26.29
22.47
20.17
Parameter Value
Title BZV01190F2.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 32
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
Me
I
S5
f1 (ppm)

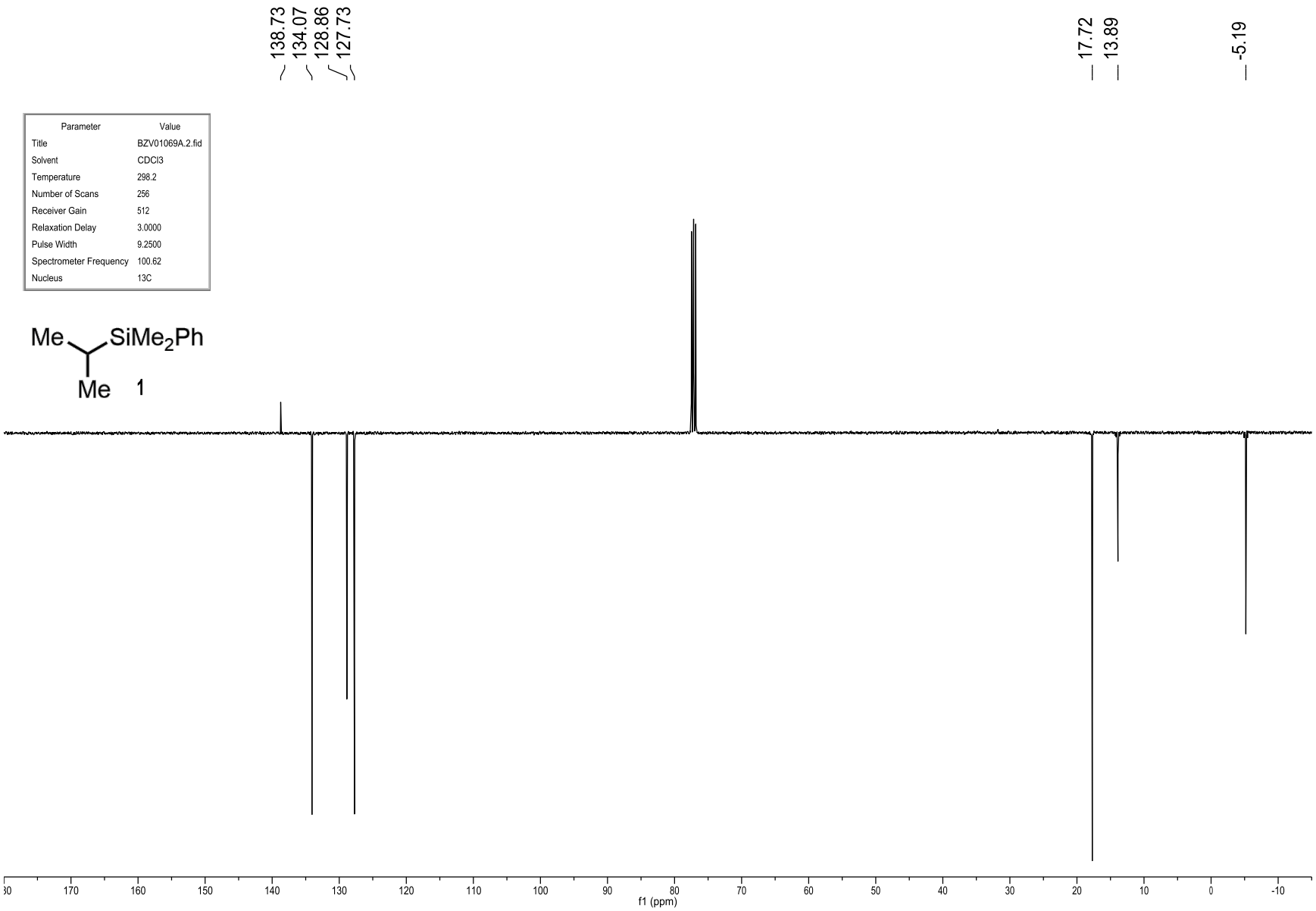
Parameter	Value
Title	BZV01161A.7.fid
Solvent	CDCl3
Temperature	298.2
Number of Scans	16
Receiver Gain	13
Relaxation Delay	1.0000
Pulse Width	15.0000
Spectrometer Frequency	400.13
Nucleus	1H



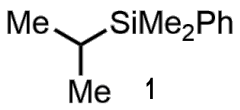
S6


48.09
36.09
27.99
24.95
Parameter Value
Title BZV01161A.8.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
Me
Me
I
S6
f1 (ppm)

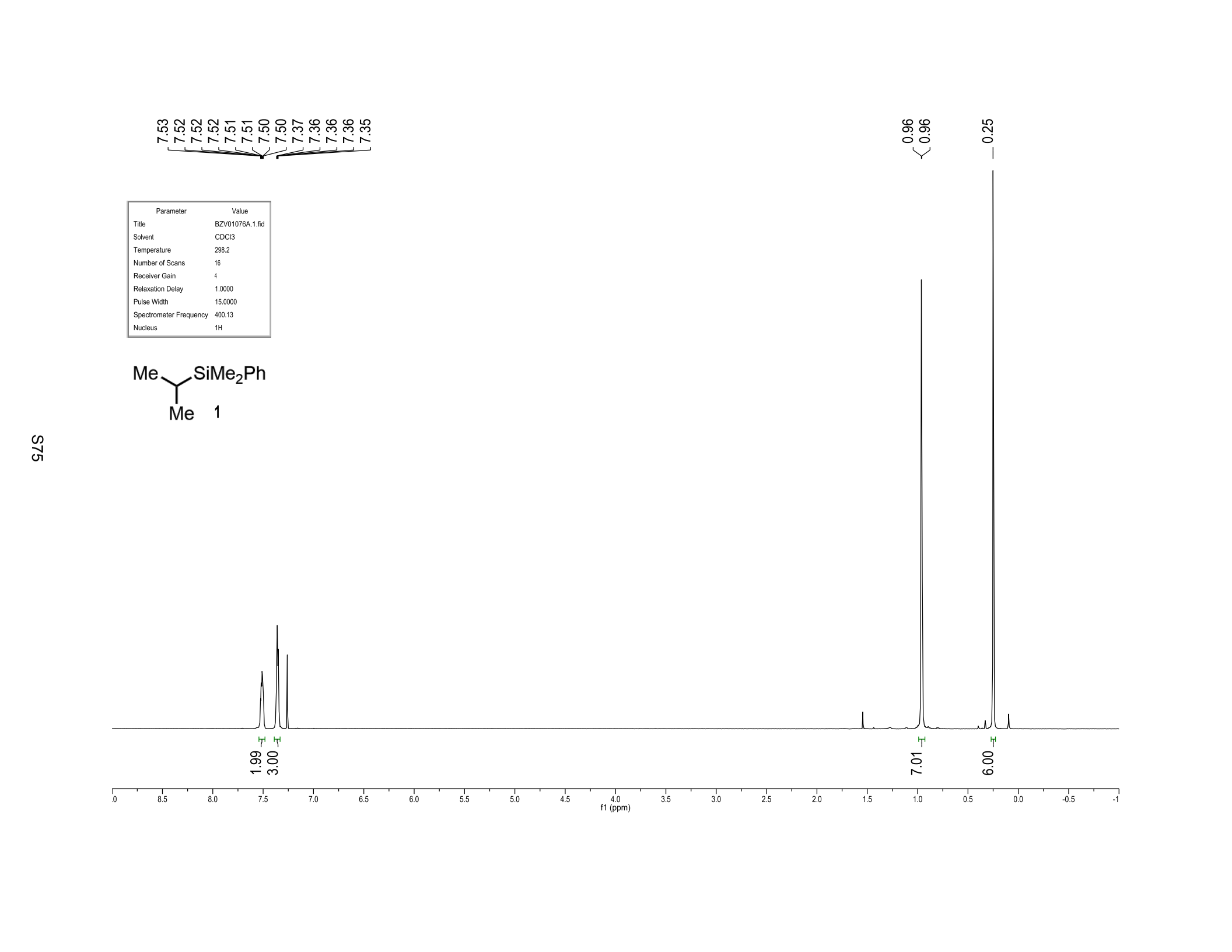

7.52
7.52
7.51
7.50
7.37
7.36
7.36
7.35
7.35
7.34
0.96
0.95
0.25
Parameter Value
Title BZV01069A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 8
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
SiMe2Ph
Me
1
2.00
3.00
7.00
6.00
f1 (ppm)

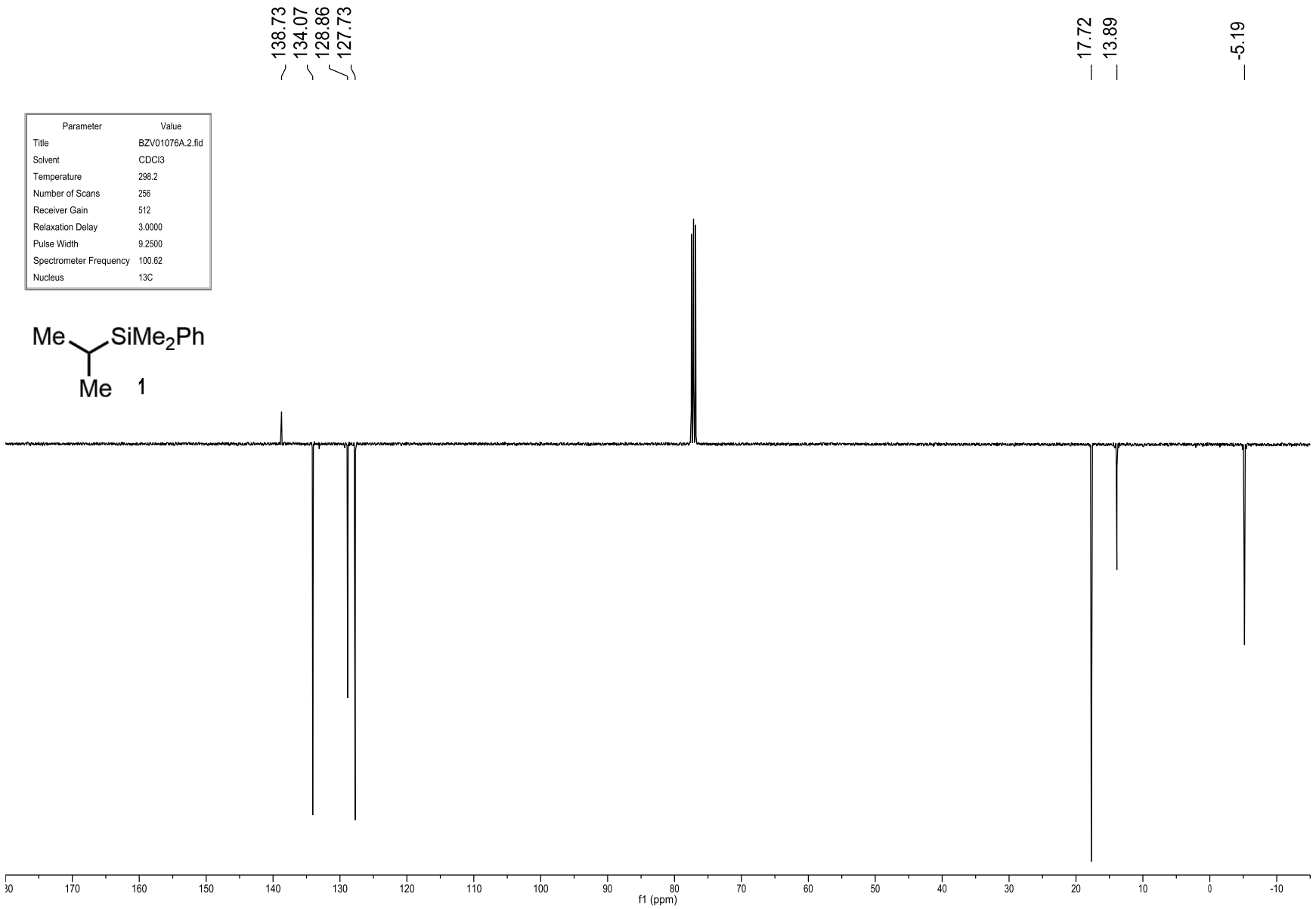

138.73
134.07
128.86
127.73
17.72
13.89
-5.19
Parameter Value
Title BZV01069A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me SiMe2Ph
Me
1
f1 (ppm)

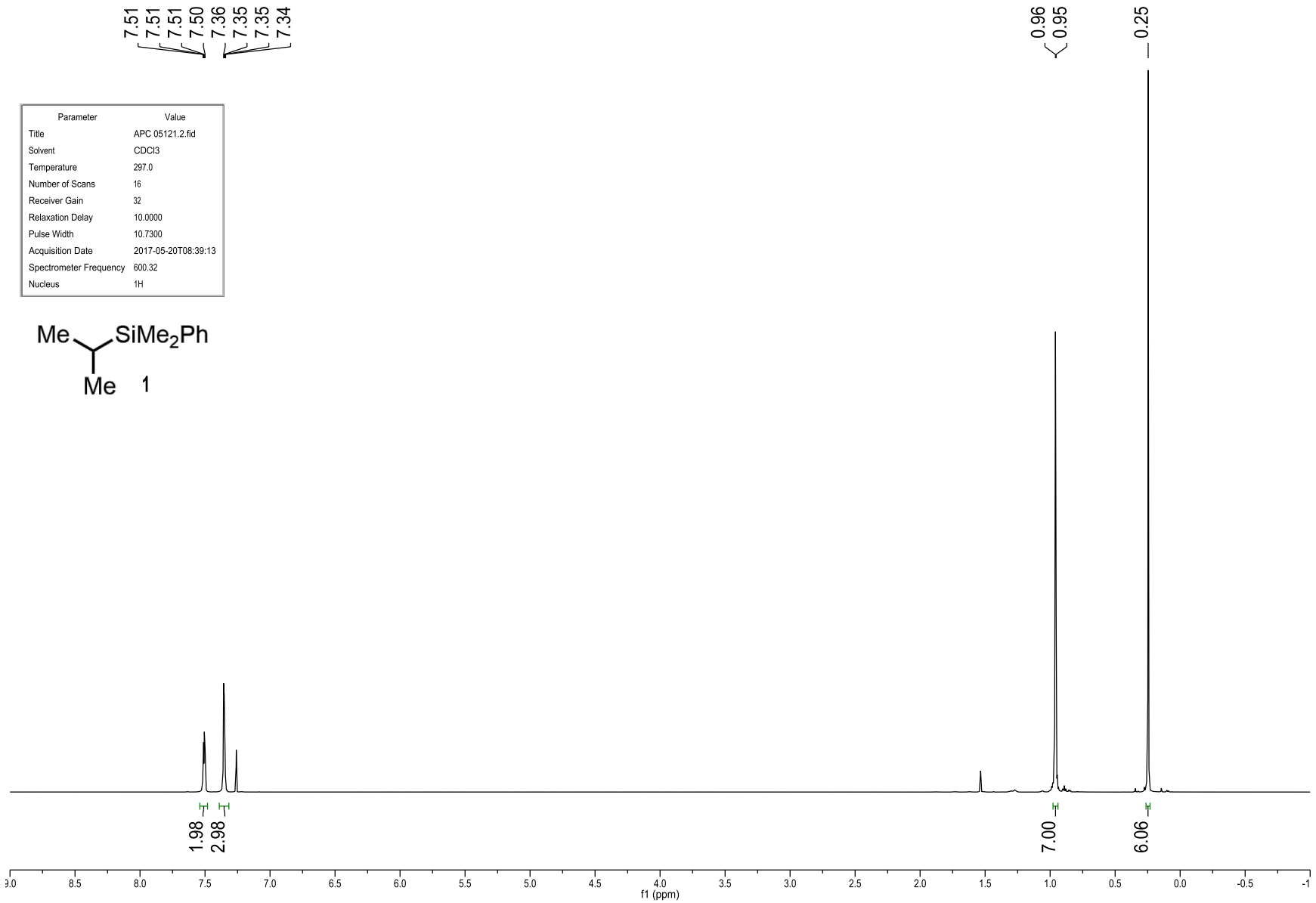
Parameter	Value
Title	BZV01069ASi1.1.fid
Solvent	CDCl3
Temperature	300.5
Number of Scans	128
Receiver Gain	1620
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

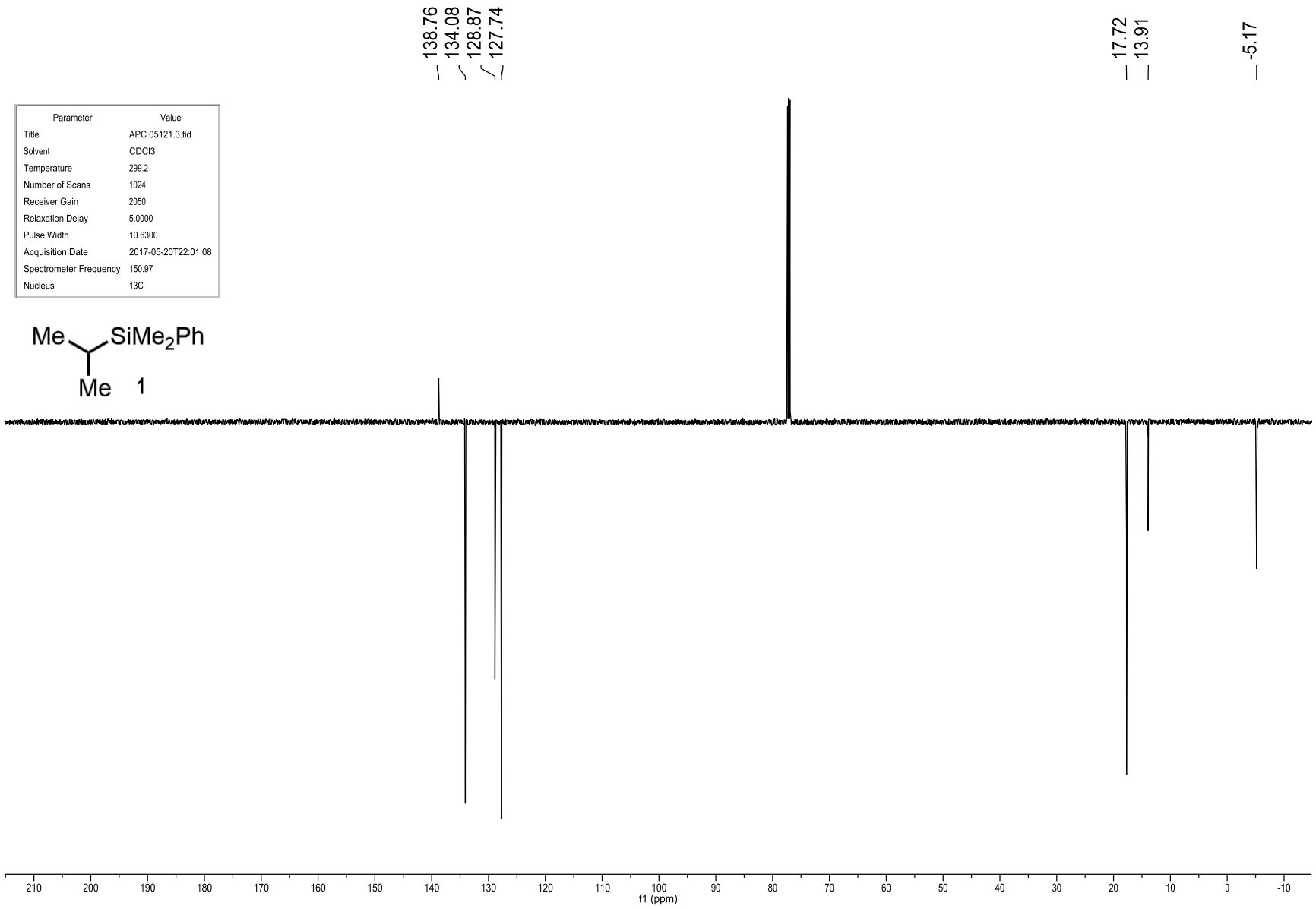


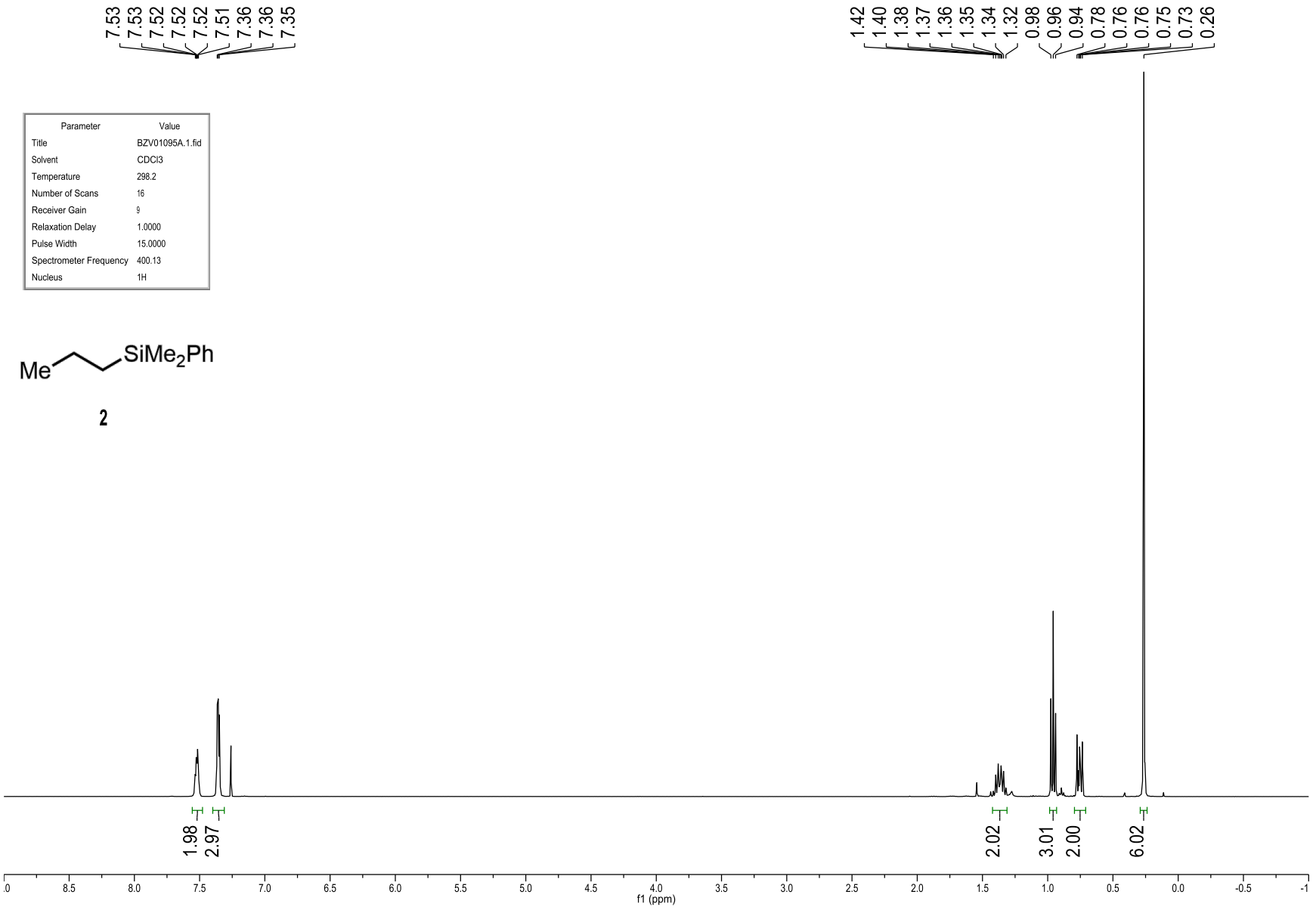
1

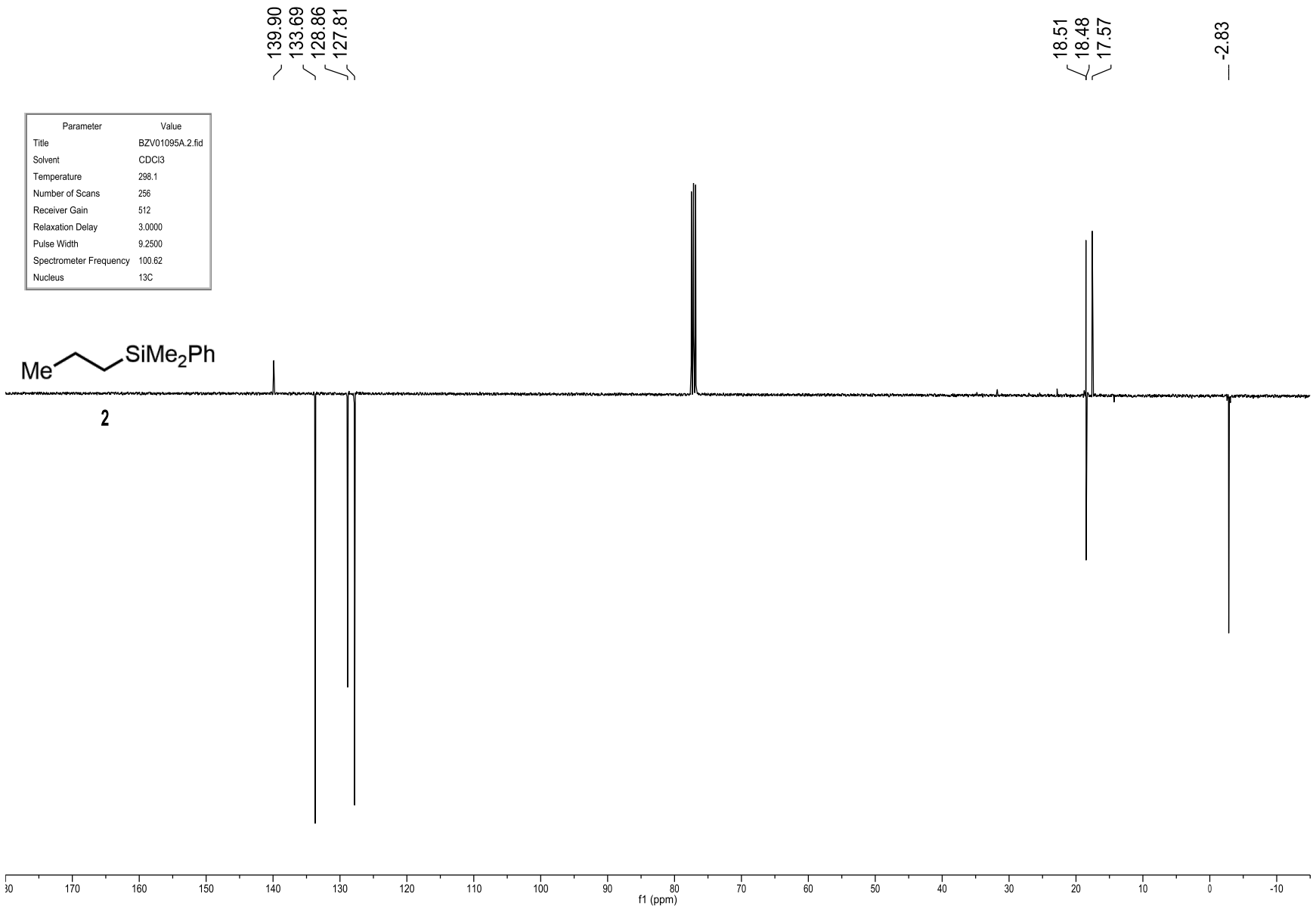

7.53
7.52
7.52
7.52
7.51
7.51
7.50
7.50
7.37
7.36
7.36
7.36
7.35
0.96
0.96
0.25
Parameter
Value
Title
BZV01076A.1.fid
Solvent
CDCl3
Temperature
298.2
Number of Scans
16
Receiver Gain
4
Relaxation Delay
1.0000
Pulse Width
15.0000
Spectrometer Frequency
400.13
Nucleus
1H
Me
SiMe2Ph
Me
1
1.99
3.00
7.01
6.00
f1 (ppm)

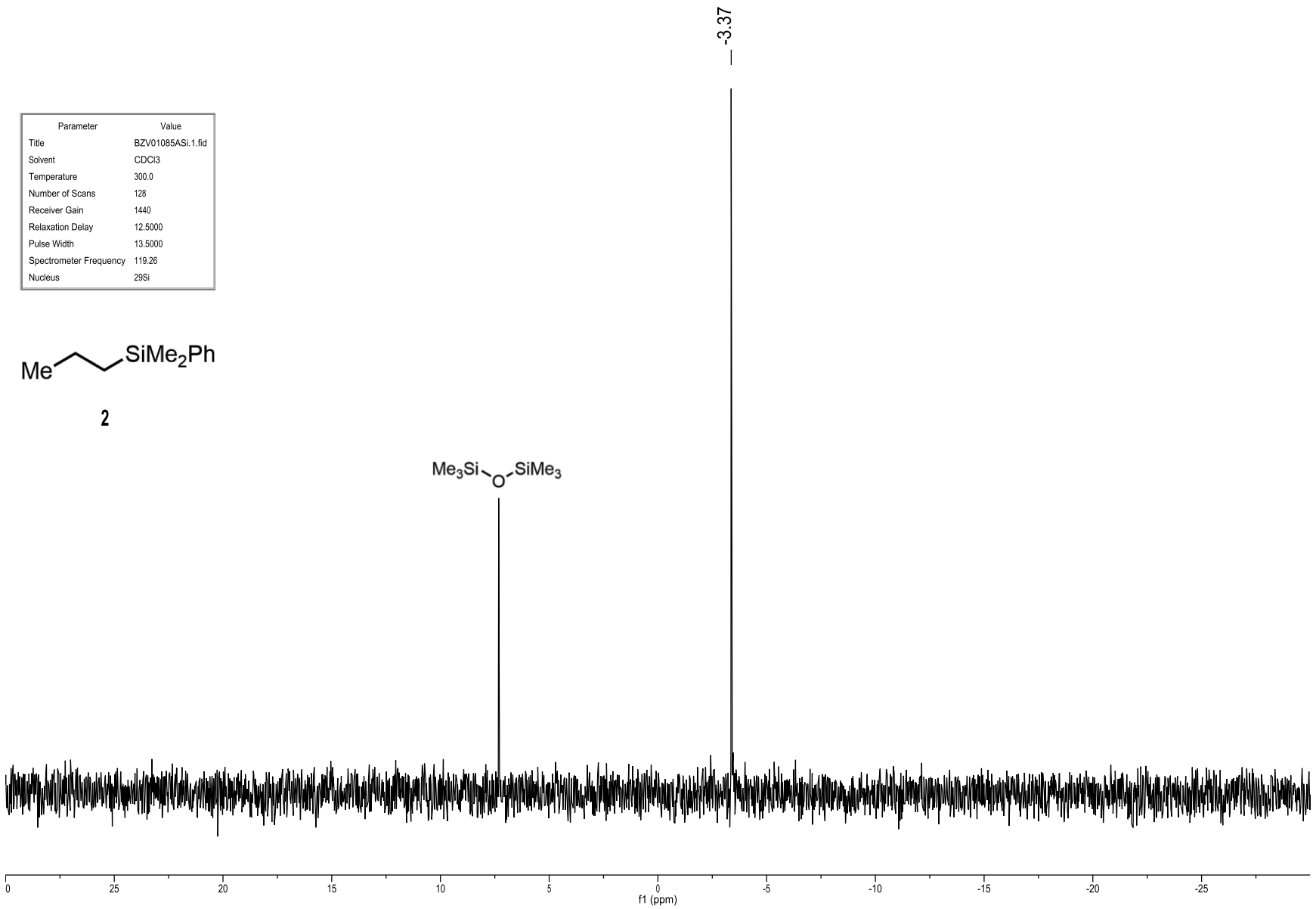

138.73
134.07
128.86
127.73
17.72
13.89
-5.19
Parameter Value
Title BZV01076A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me SiMe2Ph
Me
1
f1 (ppm)

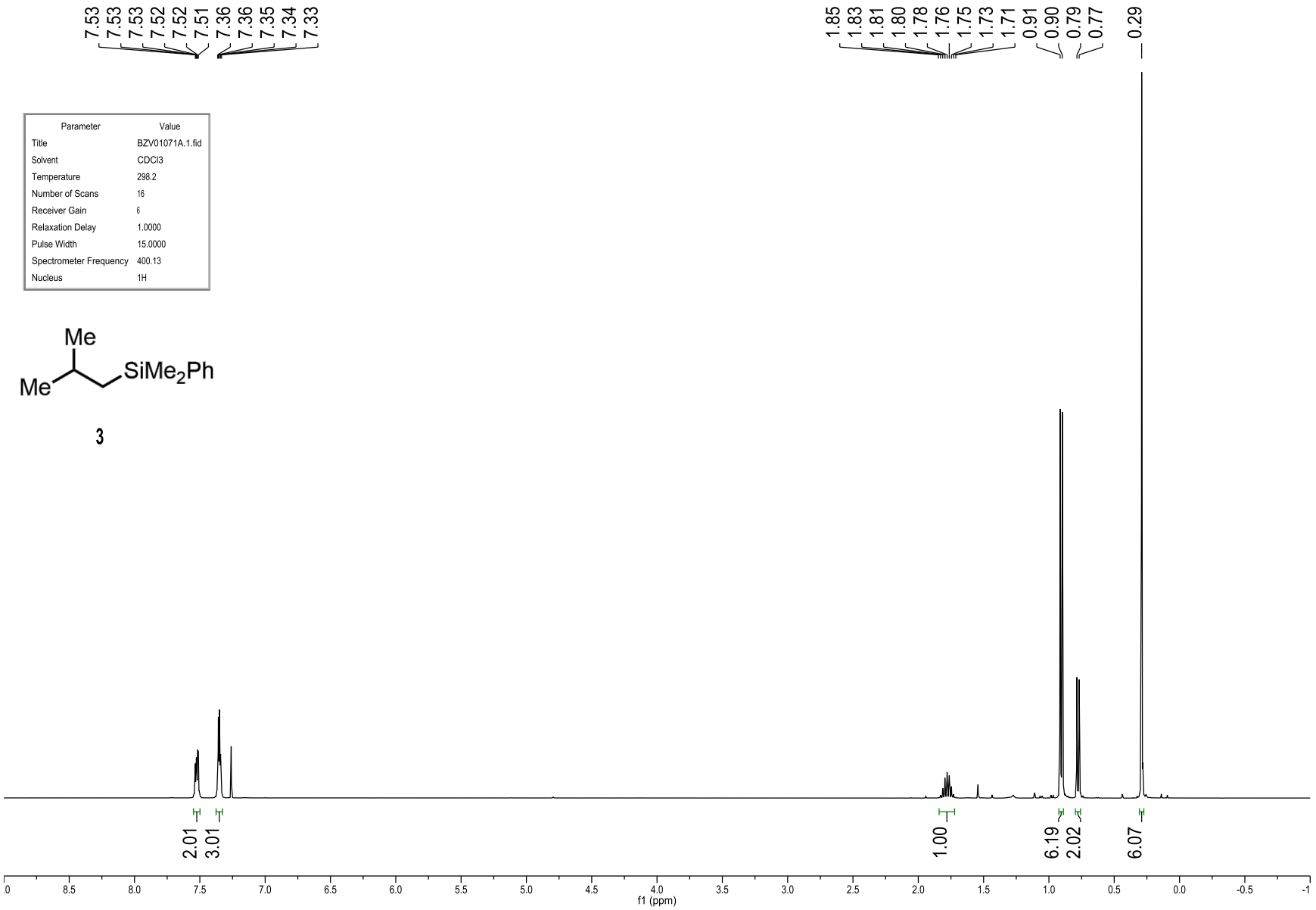

7.51
7.51
7.51
7.50
7.36
7.35
7.35
7.34
0.96
0.95
0.25
Parameter Value
Title APC 05121.2.fid
Solvent CDCl3
Temperature 297.0
Number of Scans 16
Receiver Gain 32
Relaxation Delay 10.0000
Pulse Width 10.7300
Acquisition Date 2017-05-20T08:39:13
Spectrometer Frequency 600.32
Nucleus 1H
Me
SiMe2Ph
Me
1
1.98
2.98
7.00
6.06
f1 (ppm)

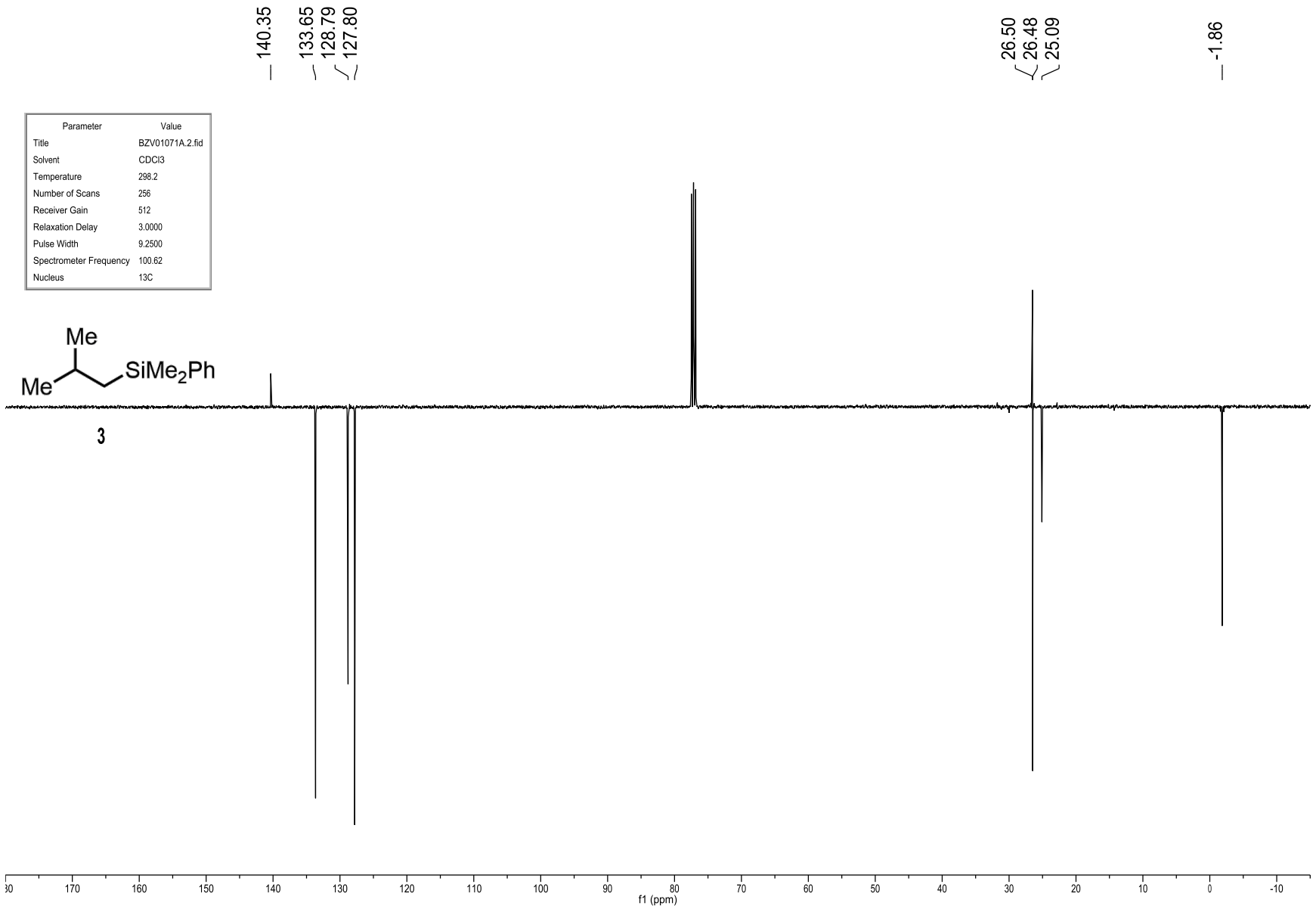

138.76
134.08
128.87
127.74
17.72
13.91
-5.17
Parameter Value
Title APC 05121.3.fid
Solvent CDCl3
Temperature 299.2
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Acquisition Date 2017-05-20T22:01:08
Spectrometer Frequency 150.97
Nucleus 13C
Me SiMe2Ph
Me
1
f1 (ppm)


7.53
7.53
7.52
7.52
7.52
7.51
7.36
7.36
7.35
1.42
1.40
1.38
1.37
1.36
1.35
1.34
1.32
0.98
0.96
0.94
0.78
0.76
0.76
0.75
0.73
0.26
Parameter Value
Title BZV01095A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 9
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
SiMe2Ph
2
1.98
2.97
2.02
3.01
2.00
6.02
f1 (ppm)

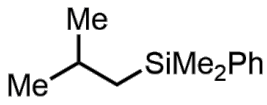

139.90
133.69
128.86
127.81
18.51
18.48
17.57
-2.83
Parameter Value
Title BZV01095A.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
SiMe2Ph
2
f1 (ppm)


-3.37
Parameter Value
Title BZV01085ASi.1.fid
Solvent CDCl3
Temperature 300.0
Number of Scans 128
Receiver Gain 1440
Relaxation Delay 12.5000
Pulse Width 13.5000
Spectrometer Frequency 119.26
Nucleus 29Si
Me
SiMe2Ph
2
Me3Si
O
SiMe3
0
25
20
15
10
5
0
-5
-10
-15
-20
-25
f1 (ppm)

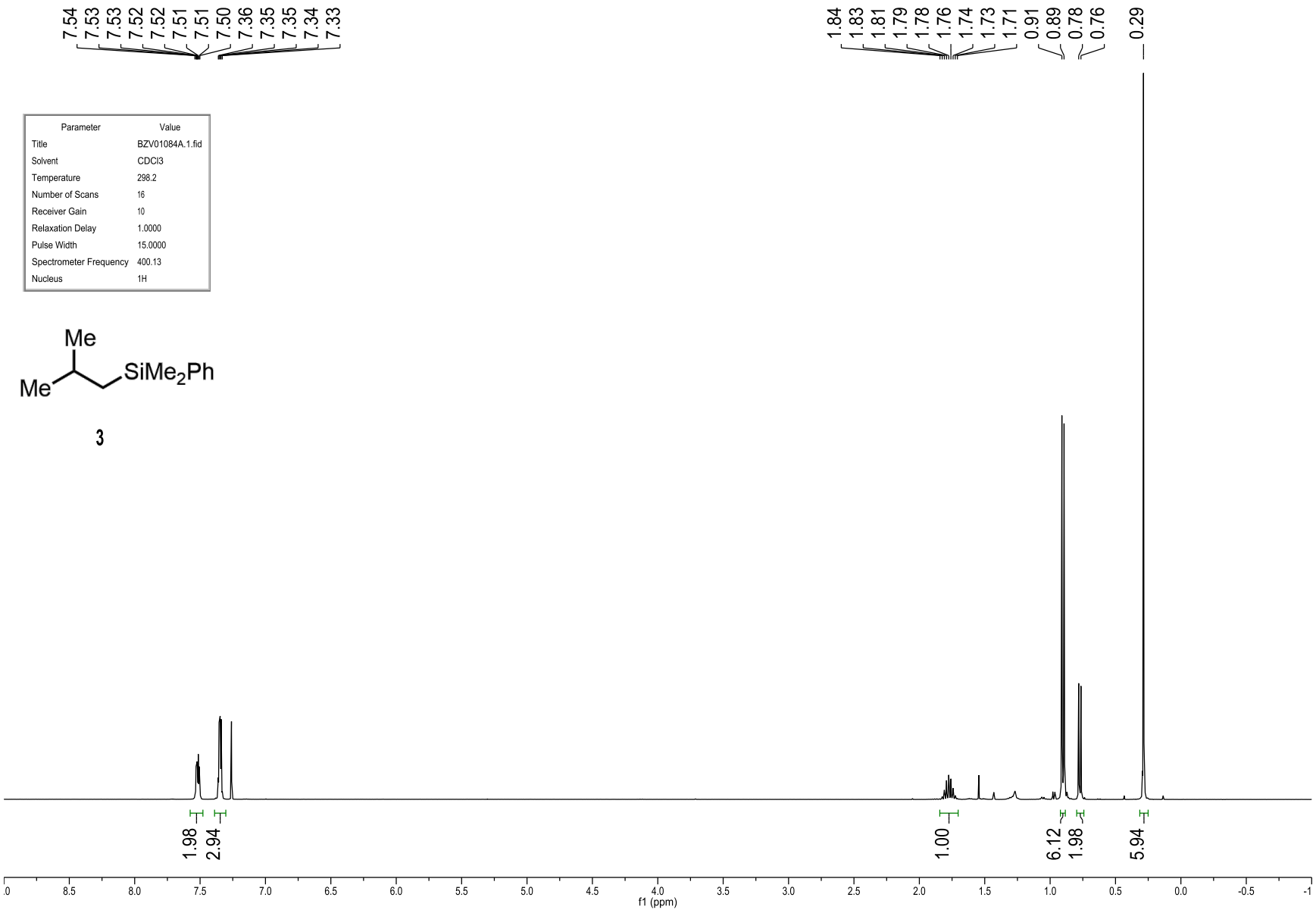

7.53
7.53
7.53
7.52
7.52
7.51
7.36
7.36
7.35
7.34
7.33
1.85
1.83
1.81
1.80
1.78
1.76
1.75
1.73
1.71
0.91
0.90
0.79
0.77
0.29
Parameter Value
Title BZV01071A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 6
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
SiMe2Ph
3
2.01
3.01
1.00
6.19
2.02
6.07
f1 (ppm)

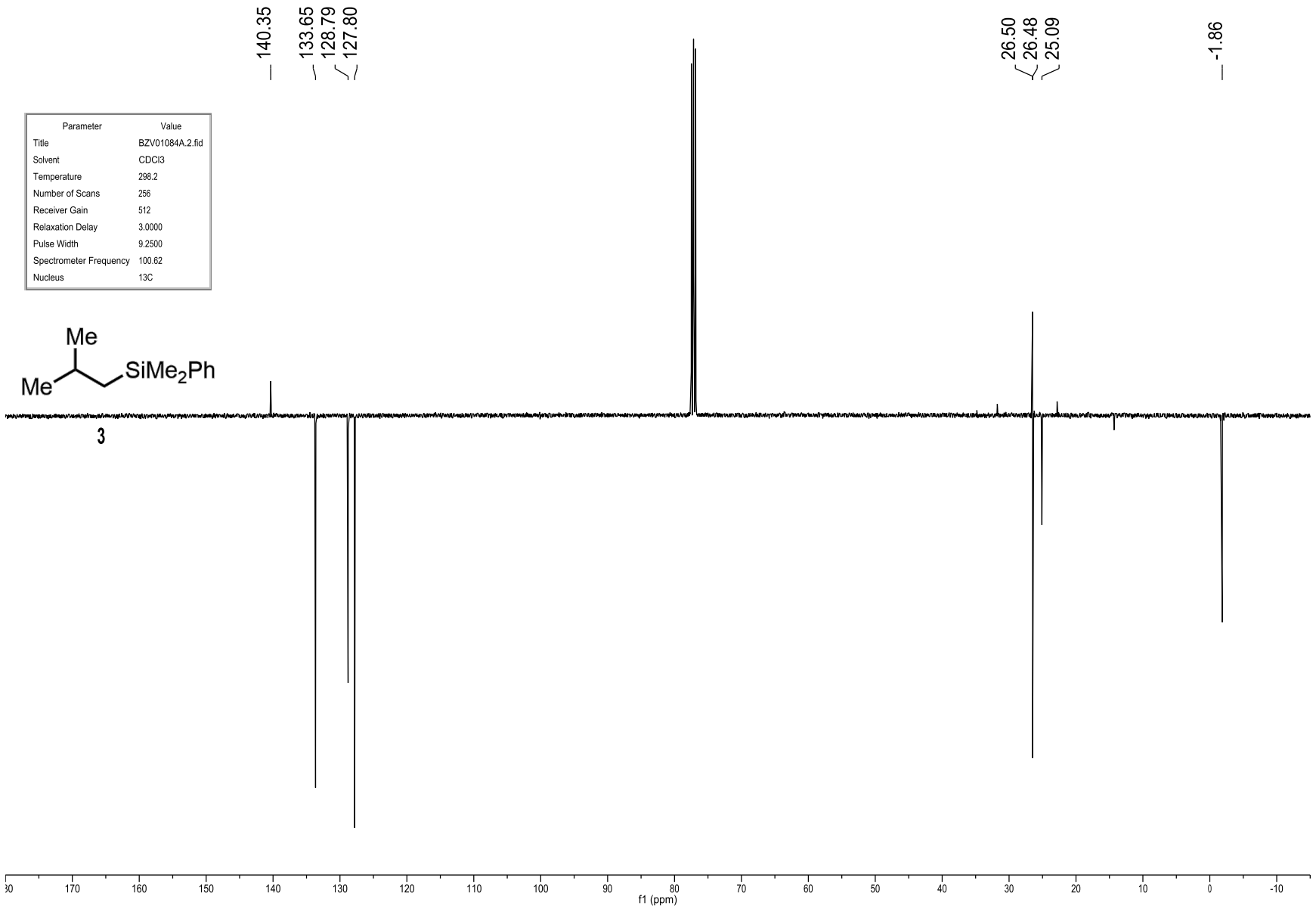

140.35
133.65
128.79
127.80
26.50
26.48
25.09
-1.86
Parameter Value
Title BZV01071A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
SiMe2Ph
3
f1 (ppm)

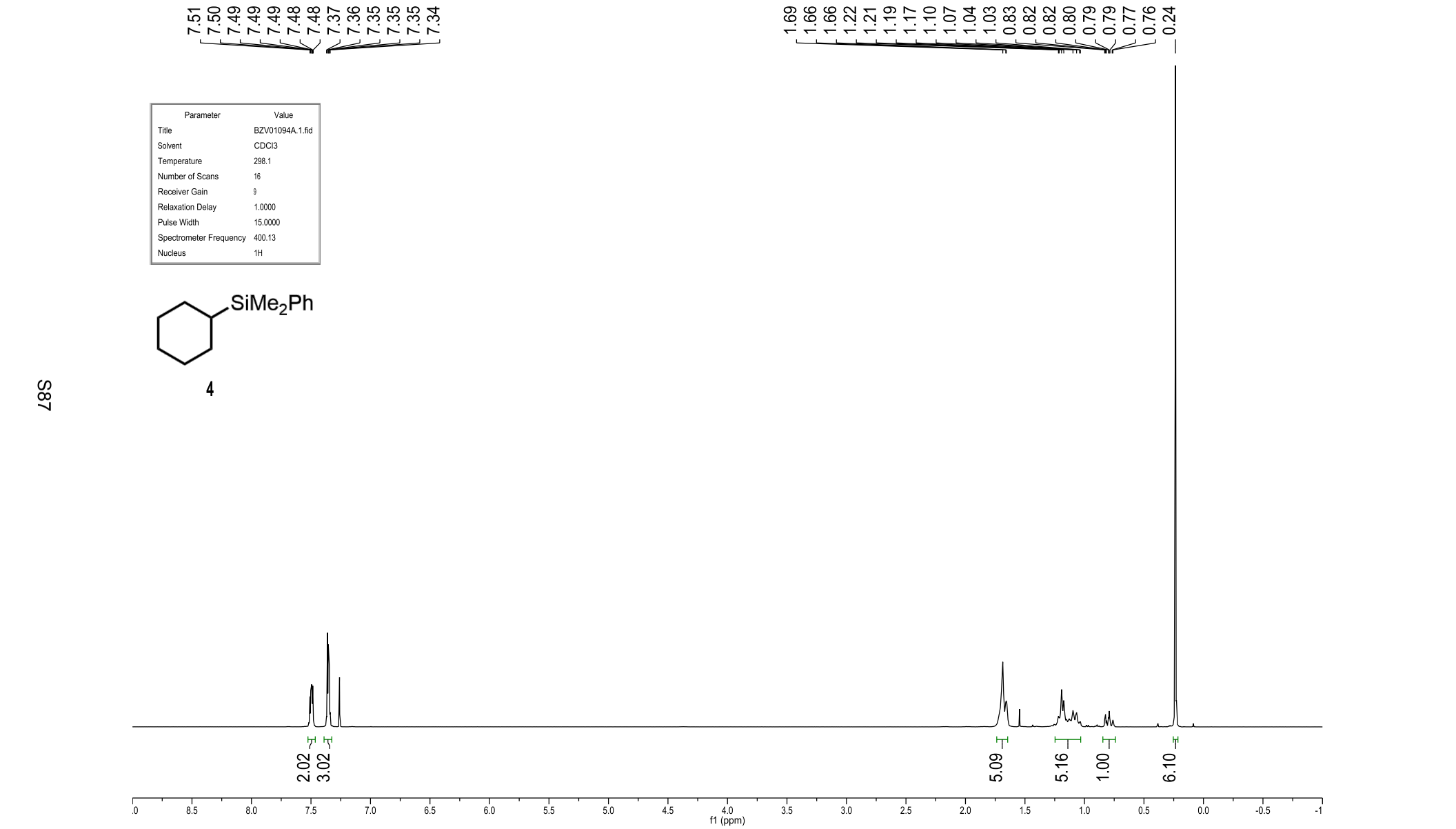
Parameter	Value
Title	BZV01071ASi.1.fid
Solvent	CDCl3
Temperature	298.1
Number of Scans	128
Receiver Gain	1440
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

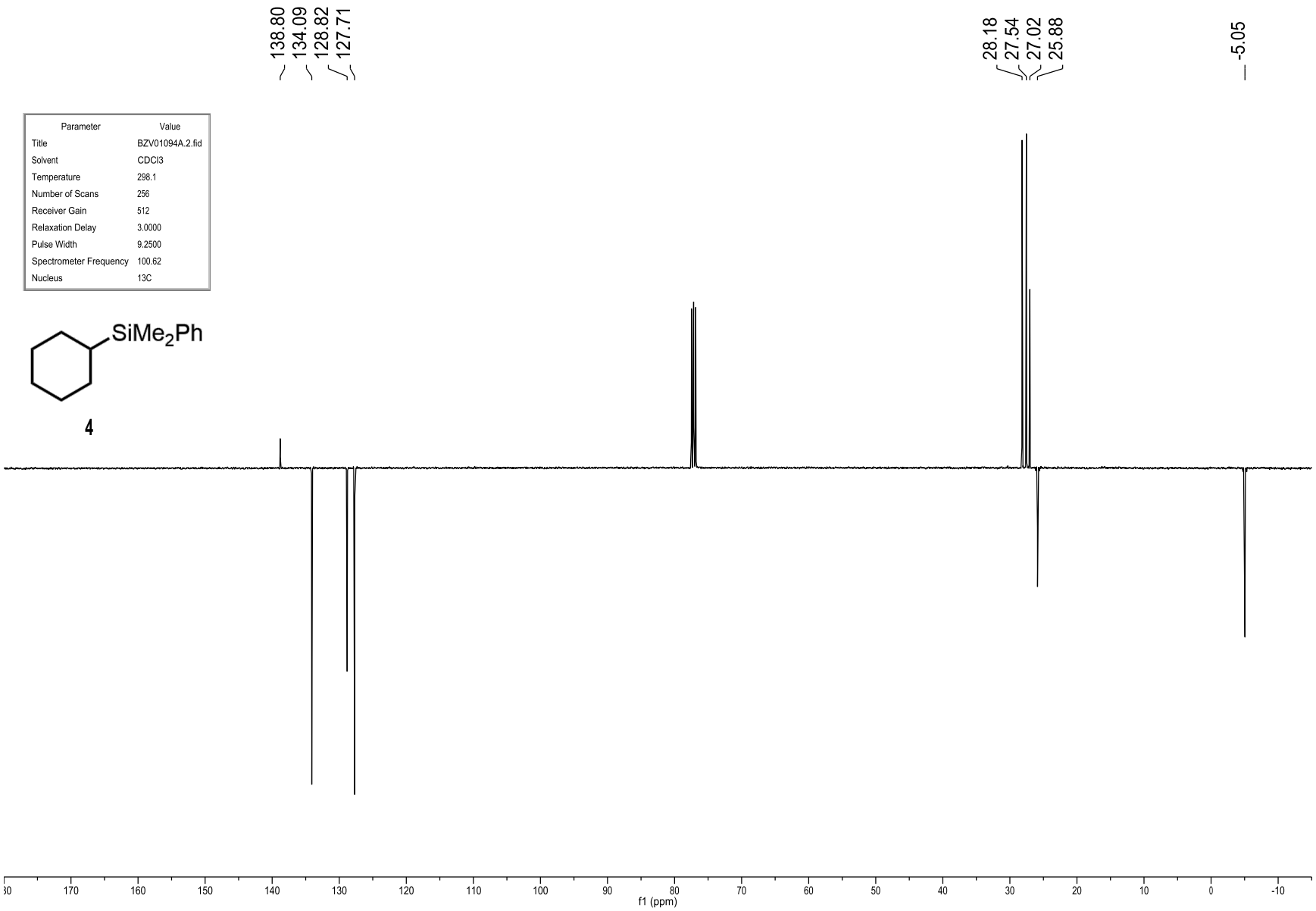


3

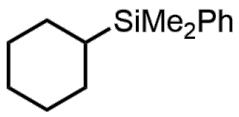

7.54
7.53
7.53
7.52
7.52
7.51
7.51
7.50
7.36
7.35
7.35
7.34
7.33
1.84
1.83
1.81
1.79
1.78
1.76
1.74
1.73
1.71
0.91
0.89
0.78
0.76
0.29
Parameter Value
Title BZV01084A.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 10
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
SiMe2Ph
3
1.98
2.94
1.00
6.12
1.98
5.94
f1 (ppm)


140.35
133.65
128.79
127.80
26.50
26.48
25.09
-1.86
Parameter Value
Title BZV01084A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
SiMe2Ph
3
f1 (ppm)

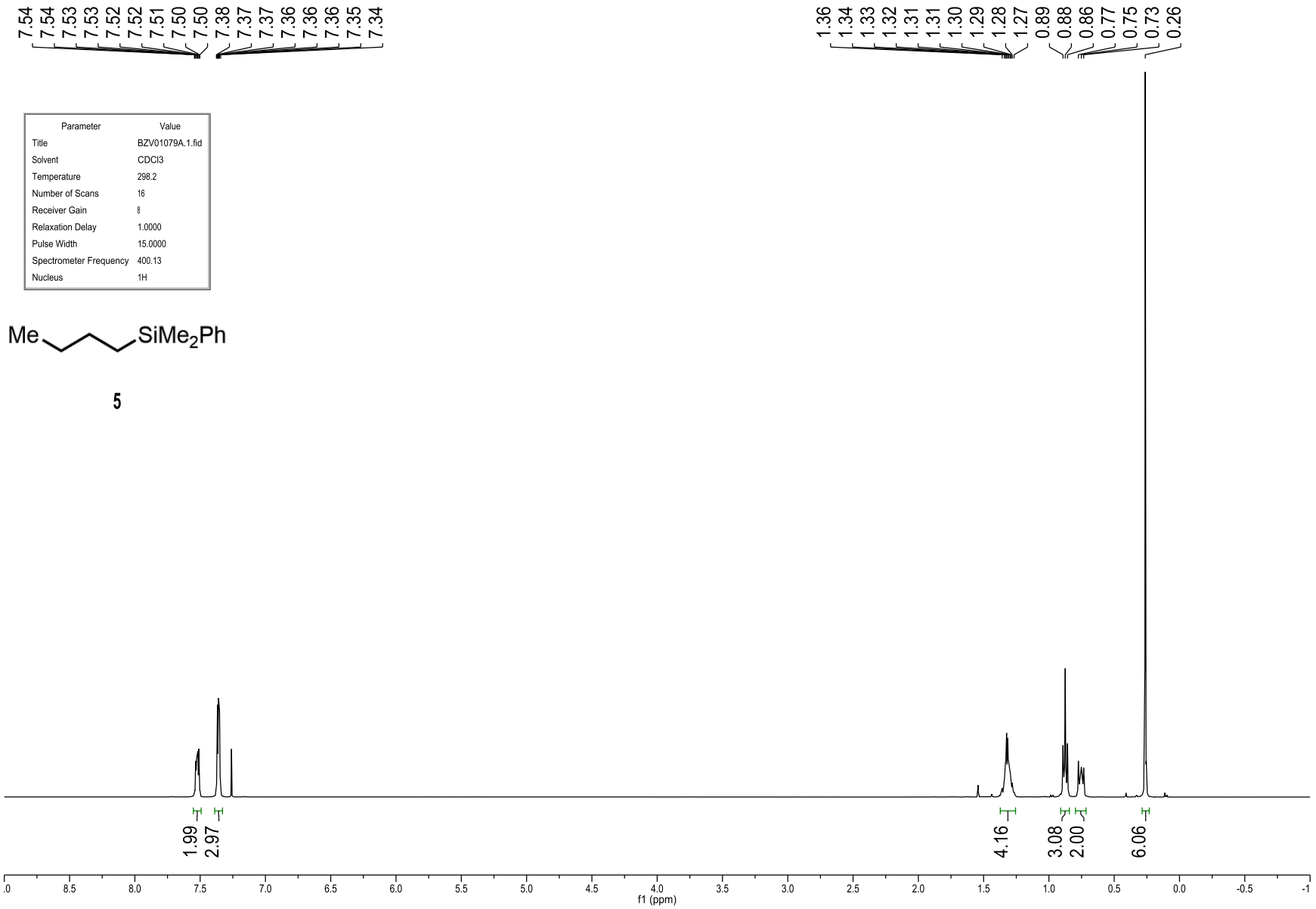

7.51
7.50
7.49
7.49
7.49
7.48
7.48
7.37
7.36
7.35
7.35
7.35
7.34
1.69
1.66
1.66
1.22
1.21
1.19
1.17
1.10
1.07
1.04
1.03
0.83
0.82
0.82
0.80
0.79
0.79
0.77
0.76
0.24
Parameter Value
Title BZV01094A.1.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 16
Receiver Gain 9
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
SiMe2Ph
4
2.02
3.02
5.09
5.16
1.00
6.10
f1 (ppm)


138.80
134.09
128.82
127.71
28.18
27.54
27.02
25.88
-5.05
Parameter Value
Title BZV01094A.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
SiMe2Ph
4
f1 (ppm)

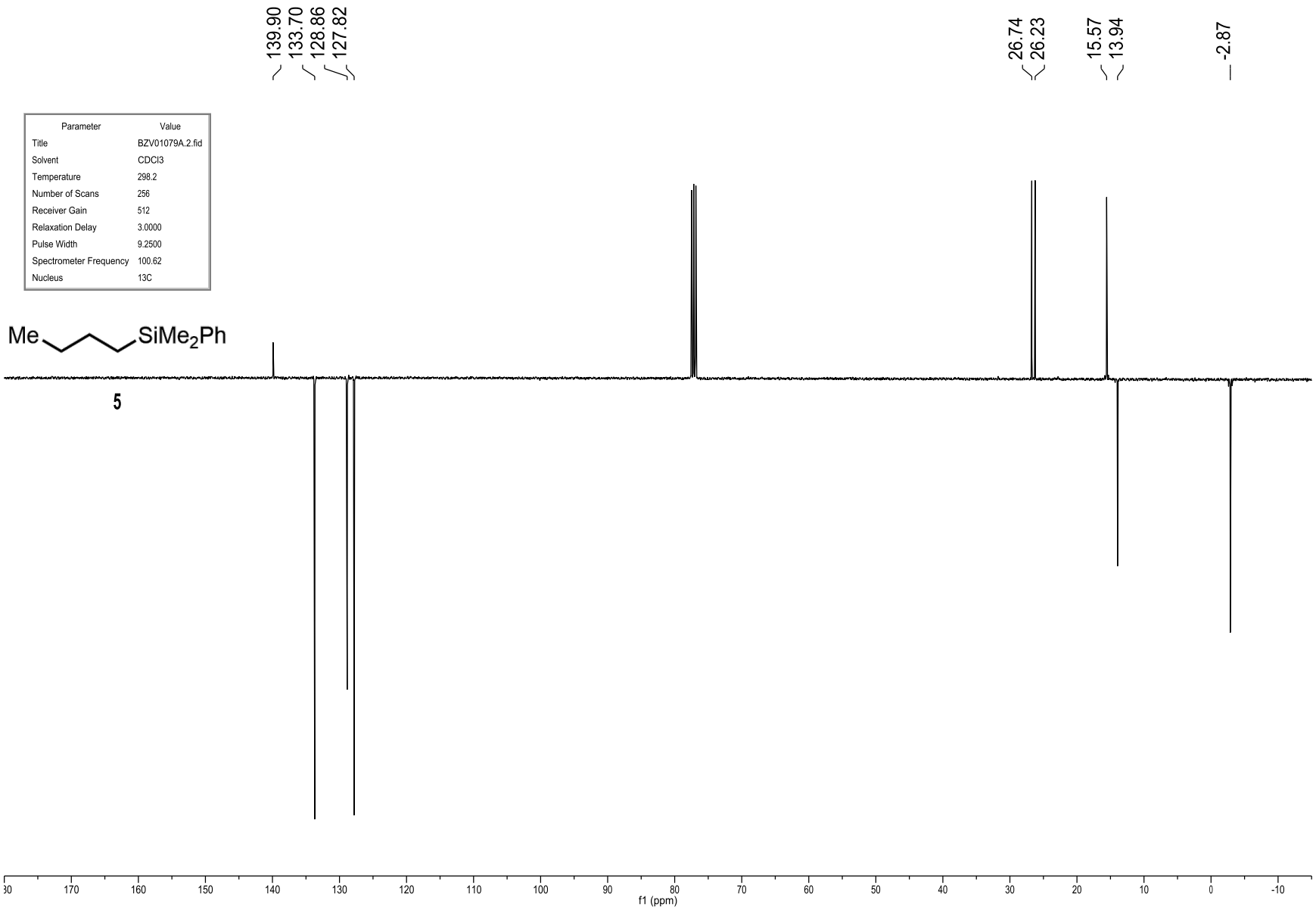
Parameter	Value
Title	BZV01094ASi.1.fid
Solvent	CDCl3
Temperature	300.0
Number of Scans	128
Receiver Gain	1440
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si



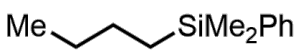
4


7.54
7.54
7.53
7.53
7.52
7.52
7.51
7.50
7.50
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.34
1.36
1.34
1.33
1.32
1.31
1.31
1.30
1.29
1.28
1.27
0.89
0.88
0.86
0.77
0.75
0.73
0.26
Parameter
Value
Title
BZV01079A.1.fid
Solvent
CDCl3
Temperature
298.2
Number of Scans
16
Receiver Gain
8
Relaxation Delay
1.0000
Pulse Width
15.0000
Spectrometer Frequency
400.13
Nucleus
1H
Me
SiMe2Ph
1.99
2.97
4.16
3.08
2.00
6.06
f1 (ppm)

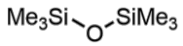
5

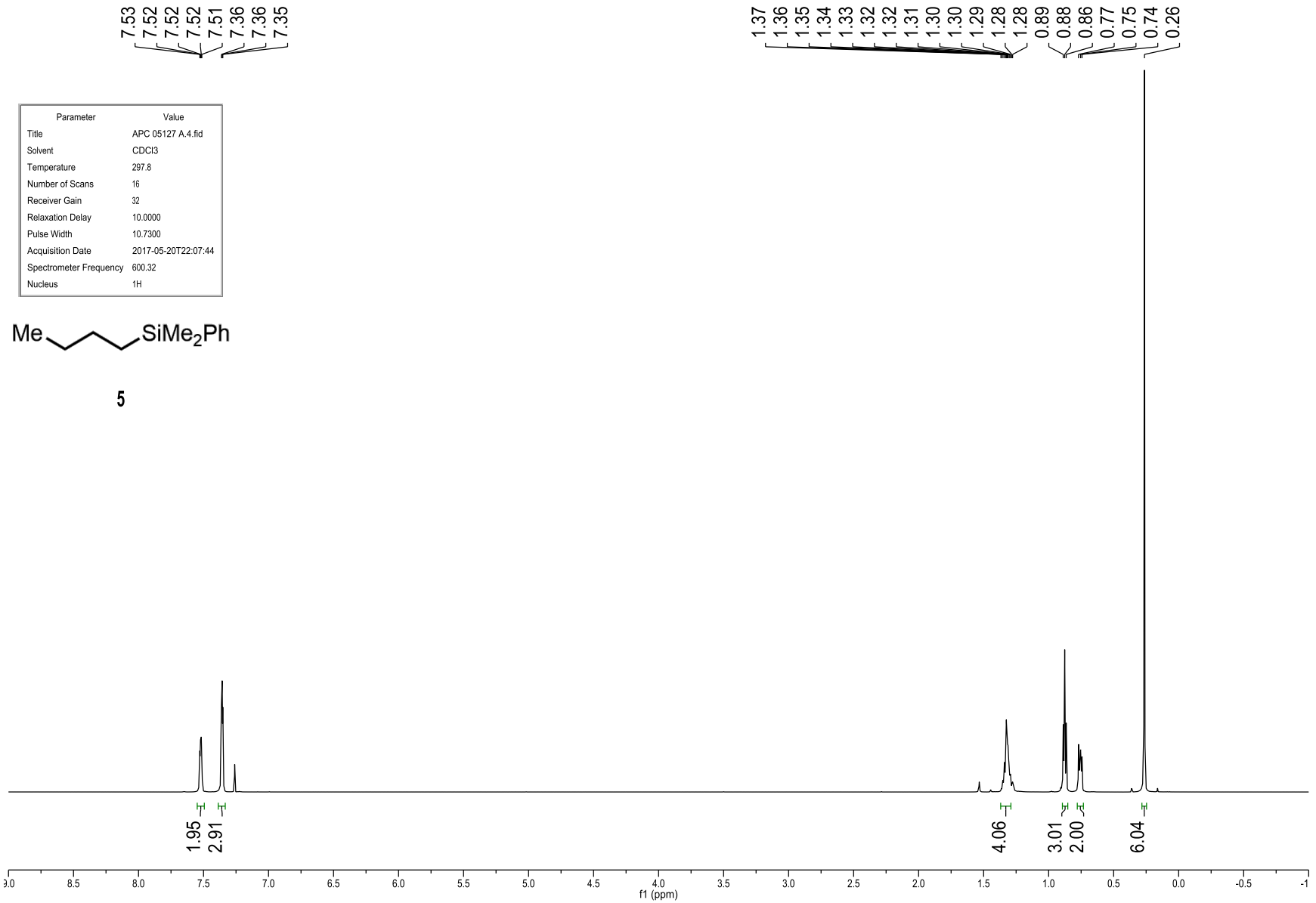

139.90
133.70
128.86
127.82
26.74
26.23
15.57
13.94
-2.87
Parameter Value
Title BZV01079A.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me SiMe2Ph
5
f1 (ppm)

Parameter	Value
Title	BZV01079ASi.1.fid
Solvent	CDCl3
Temperature	300.0
Number of Scans	128
Receiver Gain	1620
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

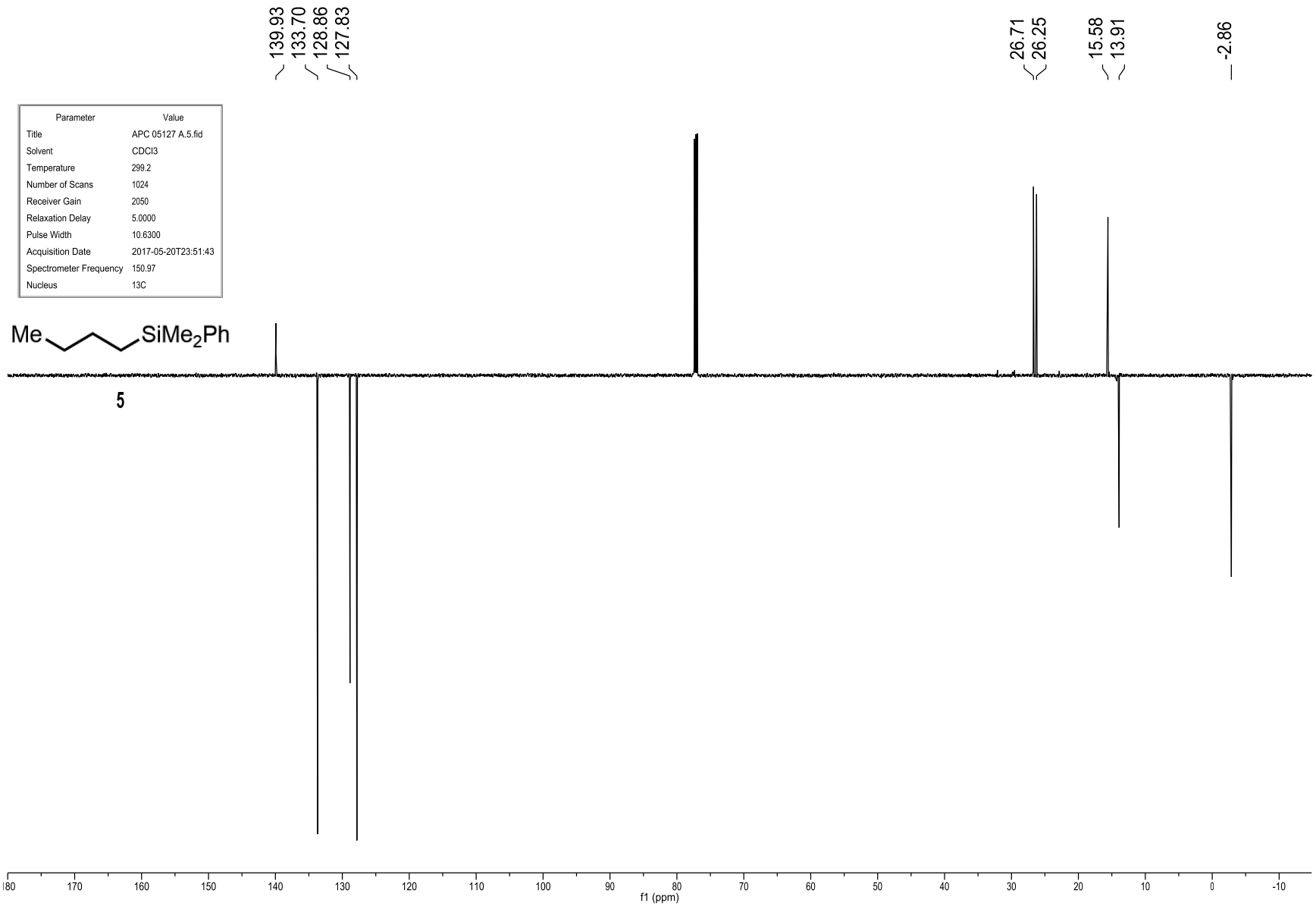


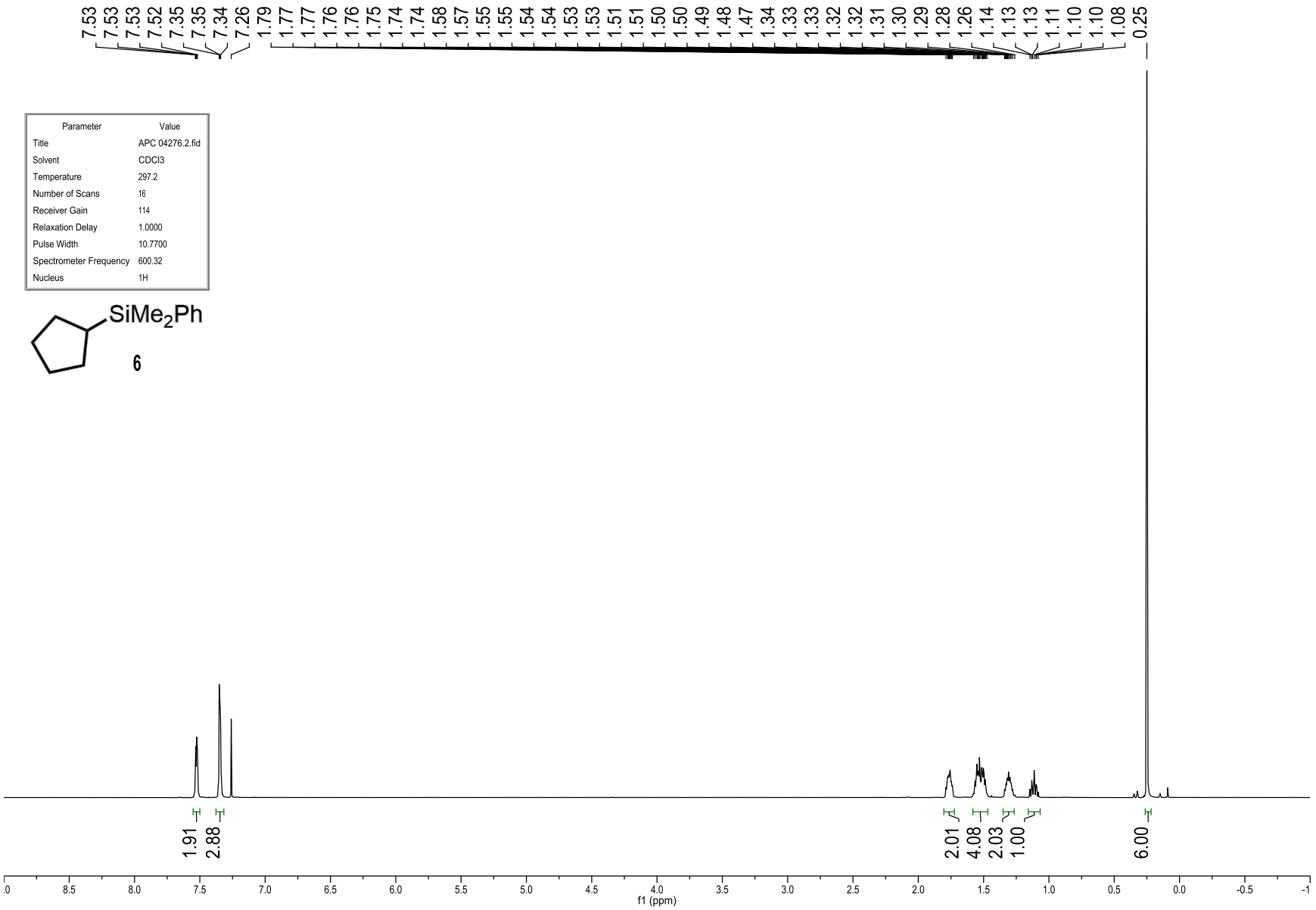
5

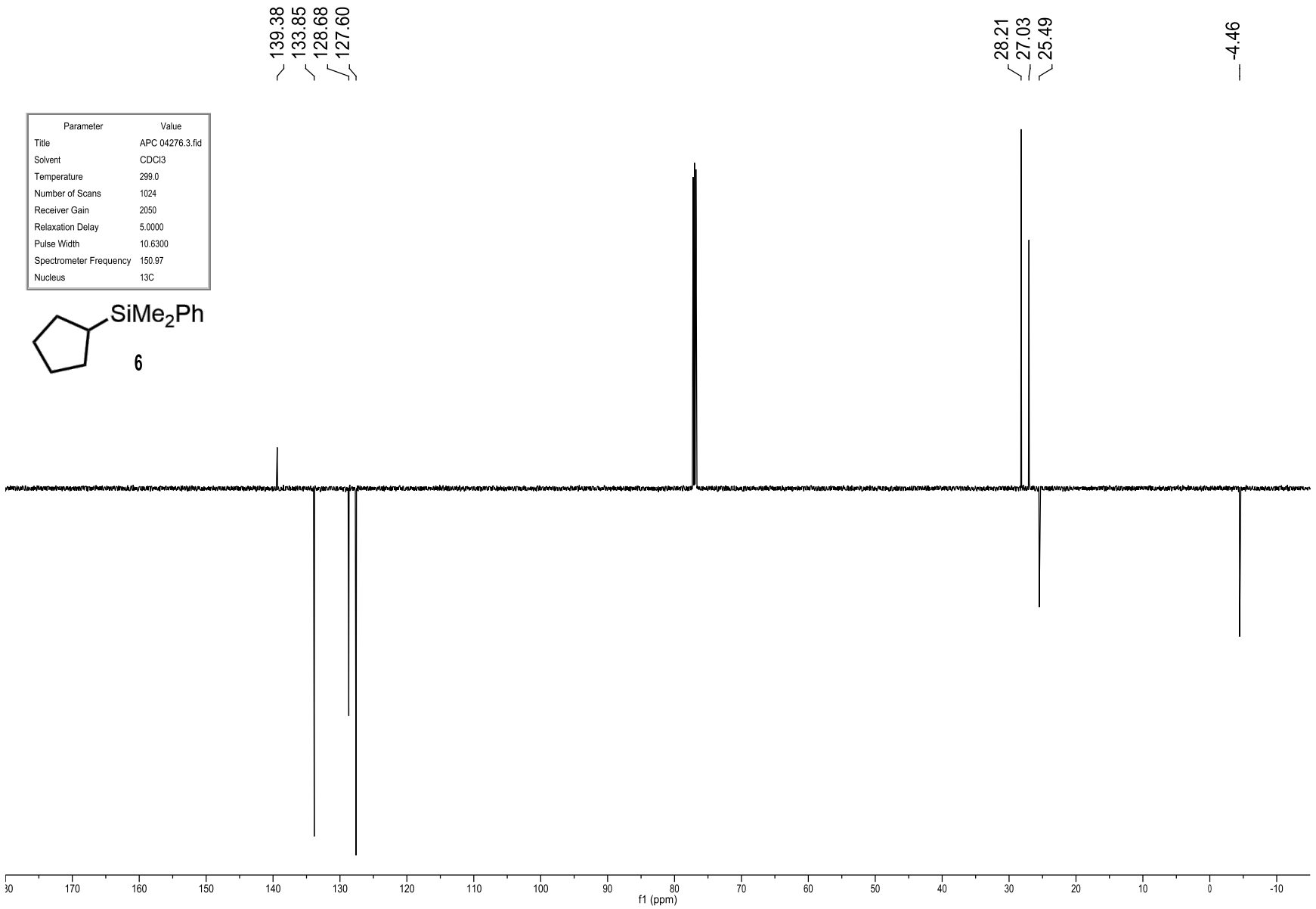



7.53
7.52
7.52
7.52
7.51
7.36
7.36
7.35
1.37
1.36
1.35
1.34
1.33
1.32
1.32
1.31
1.30
1.30
1.29
1.28
1.28
0.89
0.88
0.86
0.77
0.75
0.74
0.26
Parameter Value
Title APC 05127 A.4.fid
Solvent CDCl3
Temperature 297.8
Number of Scans 16
Receiver Gain 32
Relaxation Delay 10.0000
Pulse Width 10.7300
Acquisition Date 2017-05-20T22:07:44
Spectrometer Frequency 600.32
Nucleus 1H
Me
SiMe2Ph
1.95
2.91
4.06
3.01
2.00
6.04
f1 (ppm)

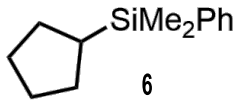
5


139.93
133.70
128.86
127.83
26.71
26.25
15.58
13.91
-2.86
Parameter Value
Title APC 05127 A.5.fid
Solvent CDCl3
Temperature 299.2
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Acquisition Date 2017-05-20T23:51:43
Spectrometer Frequency 150.97
Nucleus 13C
Me SiMe2Ph
5
f1 (ppm)

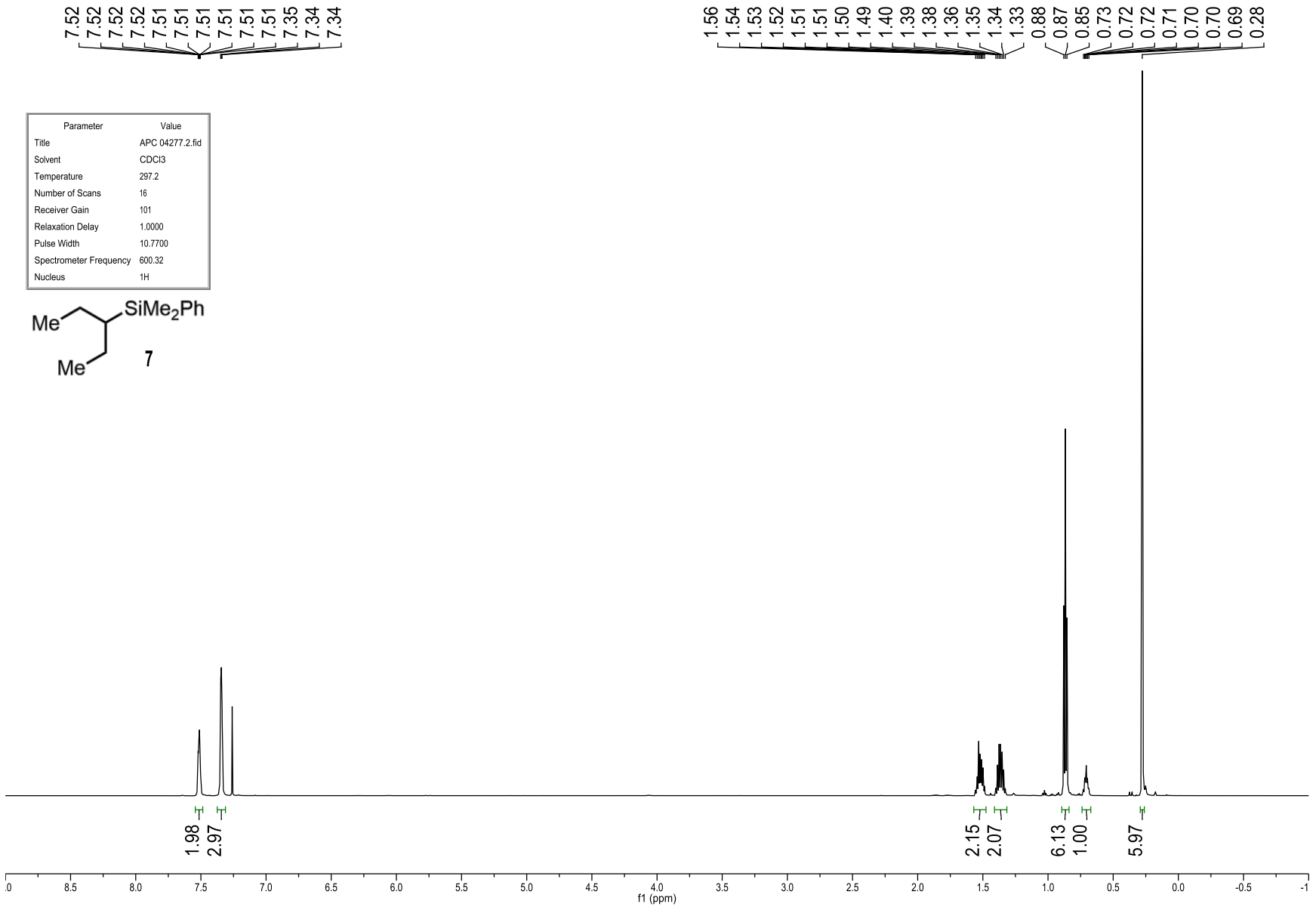

7.53 7.53 7.53 7.52 7.35 7.35 7.34 7.26 1.79 1.77 1.77 1.76 1.76 1.75 1.74 1.74 1.58 1.57 1.55 1.55 1.54 1.54 1.53 1.53 1.51 1.51 1.50 1.50 1.49 1.48 1.47 1.34 1.33 1.33 1.32 1.32 1.31 1.30 1.29 1.28 1.26 1.14 1.13 1.13 1.11 1.10 1.10 1.08 0.25
Parameter Value
Title APC 04276.2.fid
Solvent CDCl3
Temperature 297.2
Number of Scans 16
Receiver Gain 114
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Ph
6
1.91 2.88 2.01 4.08 2.03 1.00 6.00
f1 (ppm)

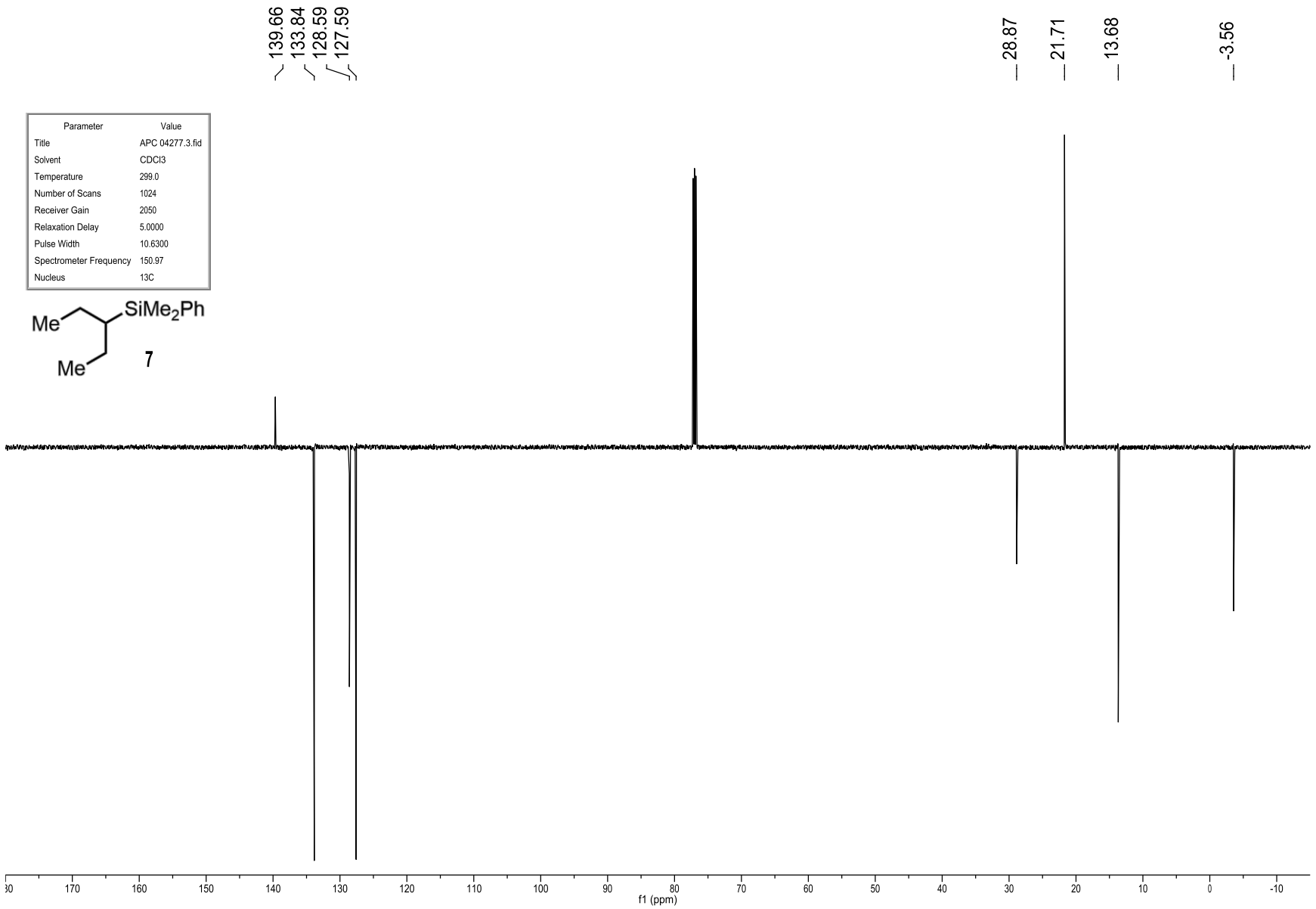

139.38
133.85
128.68
127.60
28.21
27.03
25.49
-4.46
Parameter Value
Title APC 04276.3.fid
Solvent CDCl3
Temperature 299.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Ph
6
f1 (ppm)

Parameter	Value
Title	APC 04276.4.fid
Solvent	CDCl3
Temperature	297.8
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

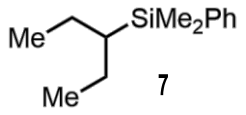


6


7.52 7.52 7.52 7.52 7.51 7.51 7.51 7.51 7.51 7.51 7.51 7.35 7.34 7.34
1.56 1.54 1.53 1.52 1.51 1.51 1.50 1.49 1.40 1.39 1.38 1.36 1.35 1.34 1.33 0.88 0.87 0.85 0.73 0.72 0.72 0.71 0.70 0.70 0.69 0.28
Parameter Value
Title APC 04277.2.fid
Solvent CDCl3
Temperature 297.2
Number of Scans 16
Receiver Gain 101
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
Me
SiMe2Ph
Me
7
1.98
2.97
2.15
2.07
6.13
1.00
5.97
f1 (ppm)

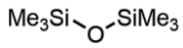

139.66
133.84
128.59
127.59
28.87
21.71
13.68
-3.56
Parameter Value
Title APC 04277.3.fid
Solvent CDCl3
Temperature 299.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
Me
SiMe2Ph
Me
7
f1 (ppm)

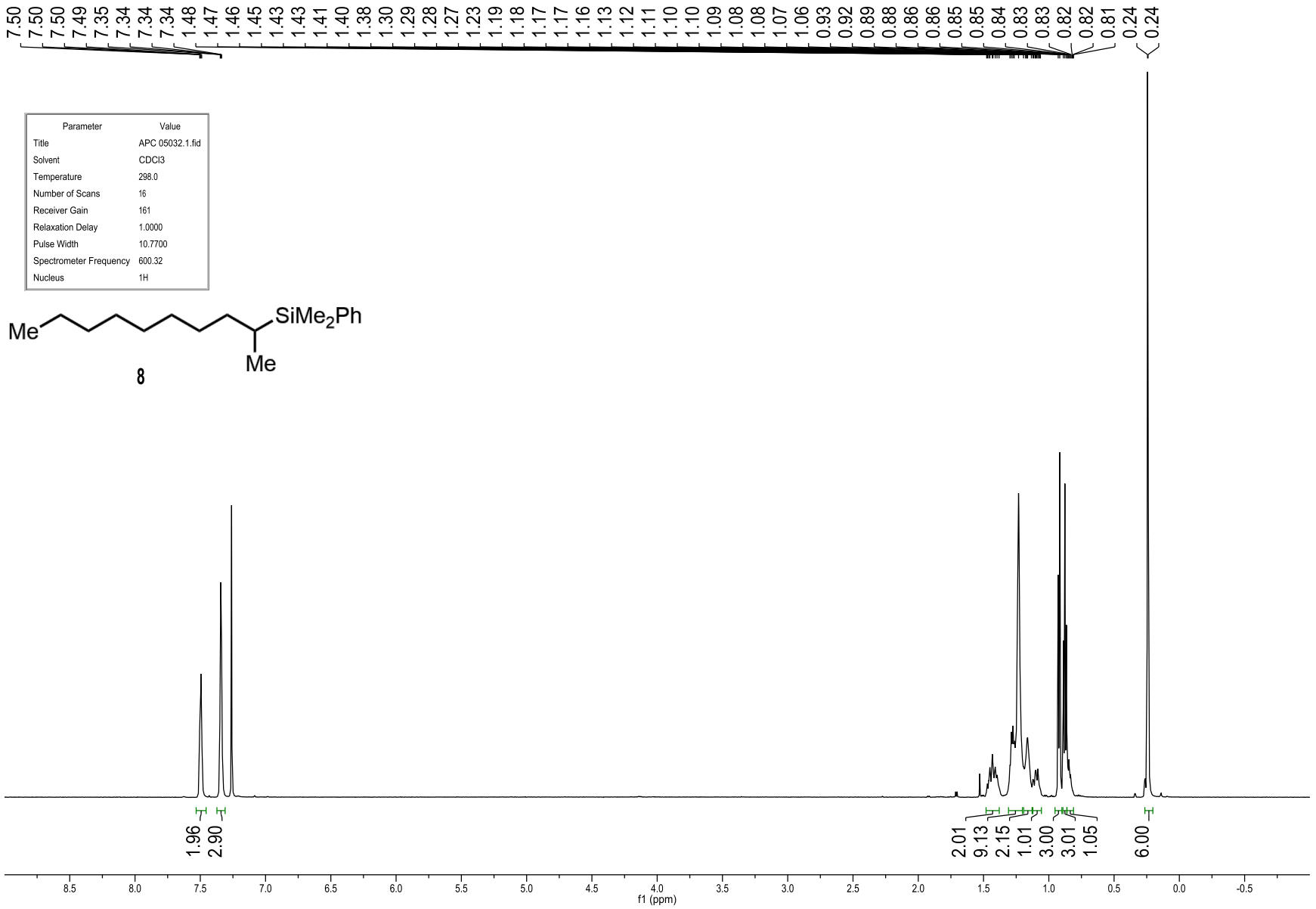
Parameter	Value
Title	APC 04277.4.fid
Solvent	CDCl3
Temperature	297.7
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

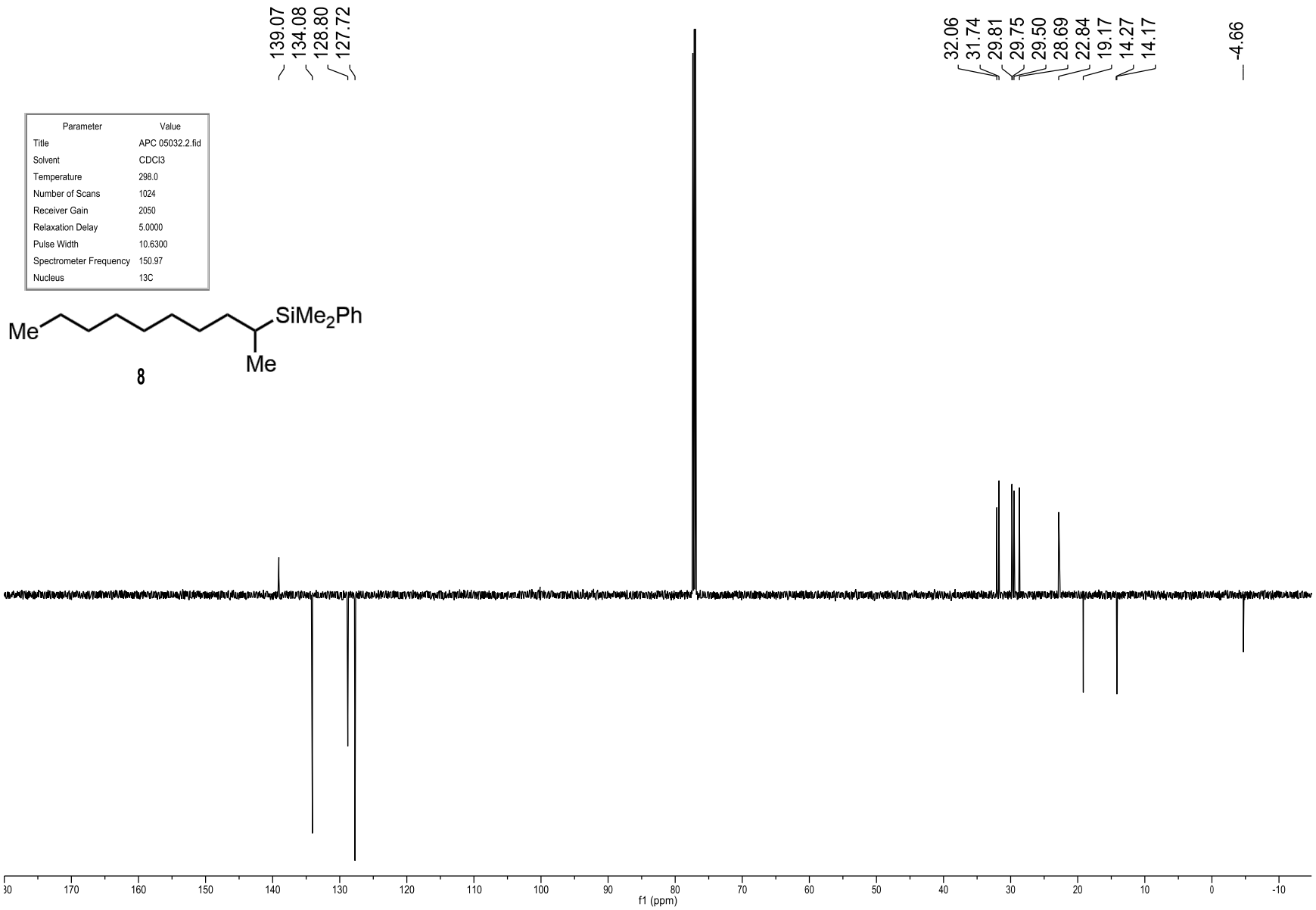


7

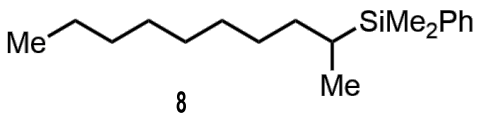
-1.04



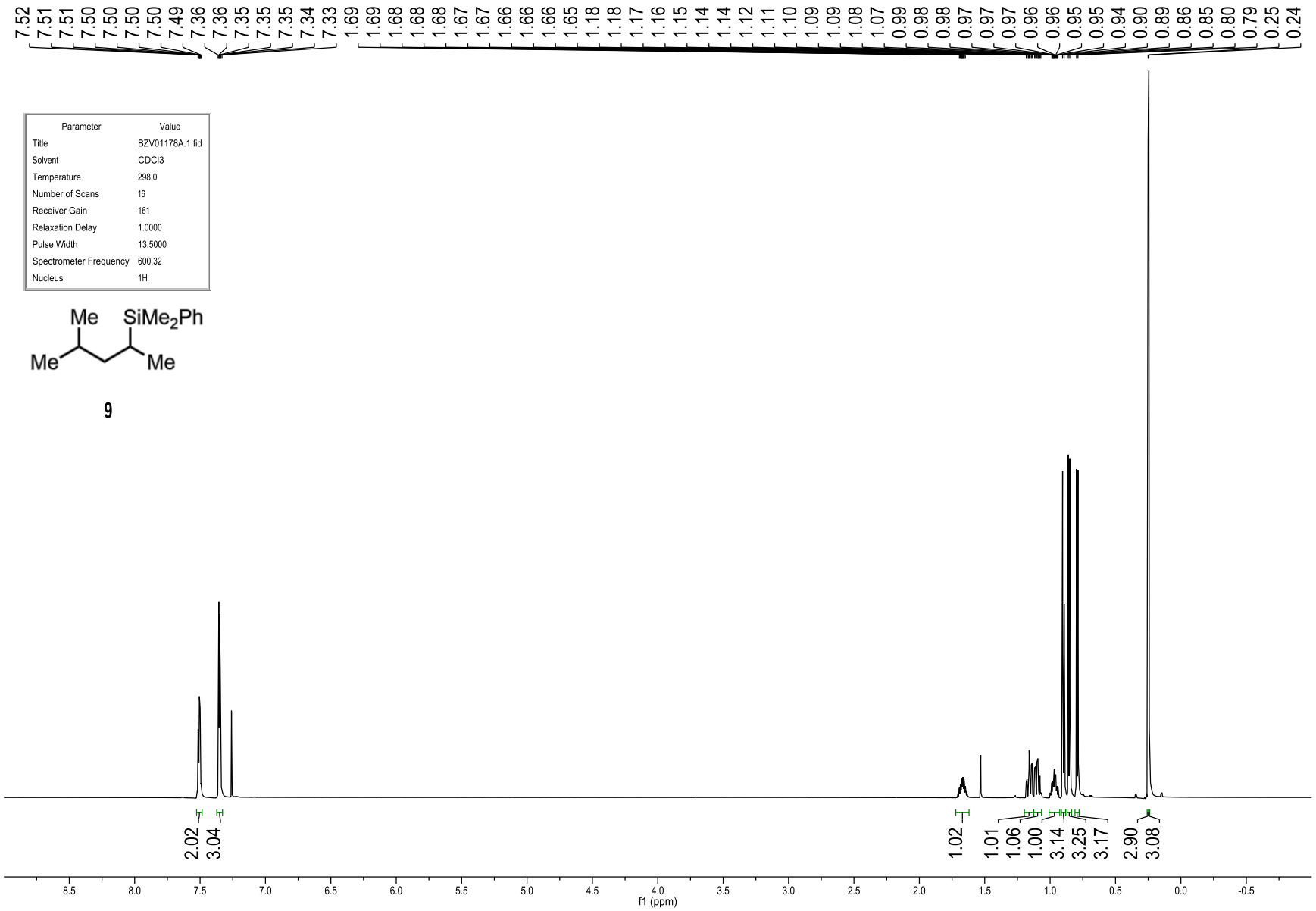

7.50 7.50 7.50 7.49 7.35 7.34 7.34 7.34 1.48 1.47 1.46 1.45 1.43 1.43 1.41 1.40 1.38 1.30 1.29 1.28 1.27 1.23 1.19 1.18 1.17 1.17 1.16 1.13 1.12 1.11 1.10 1.10 1.09 1.08 1.08 1.07 1.06 0.93 0.92 0.89 0.88 0.86 0.86 0.85 0.85 0.84 0.83 0.83 0.82 0.82 0.81 0.24 0.24
Parameter Value
Title APC 05032.1.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 161
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
Me
SiMe2Ph
Me
8
1.96 2.90
2.01 9.13 2.15 1.01 3.00 3.01 1.05 6.00
8.5 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 -0.5
f1 (ppm)

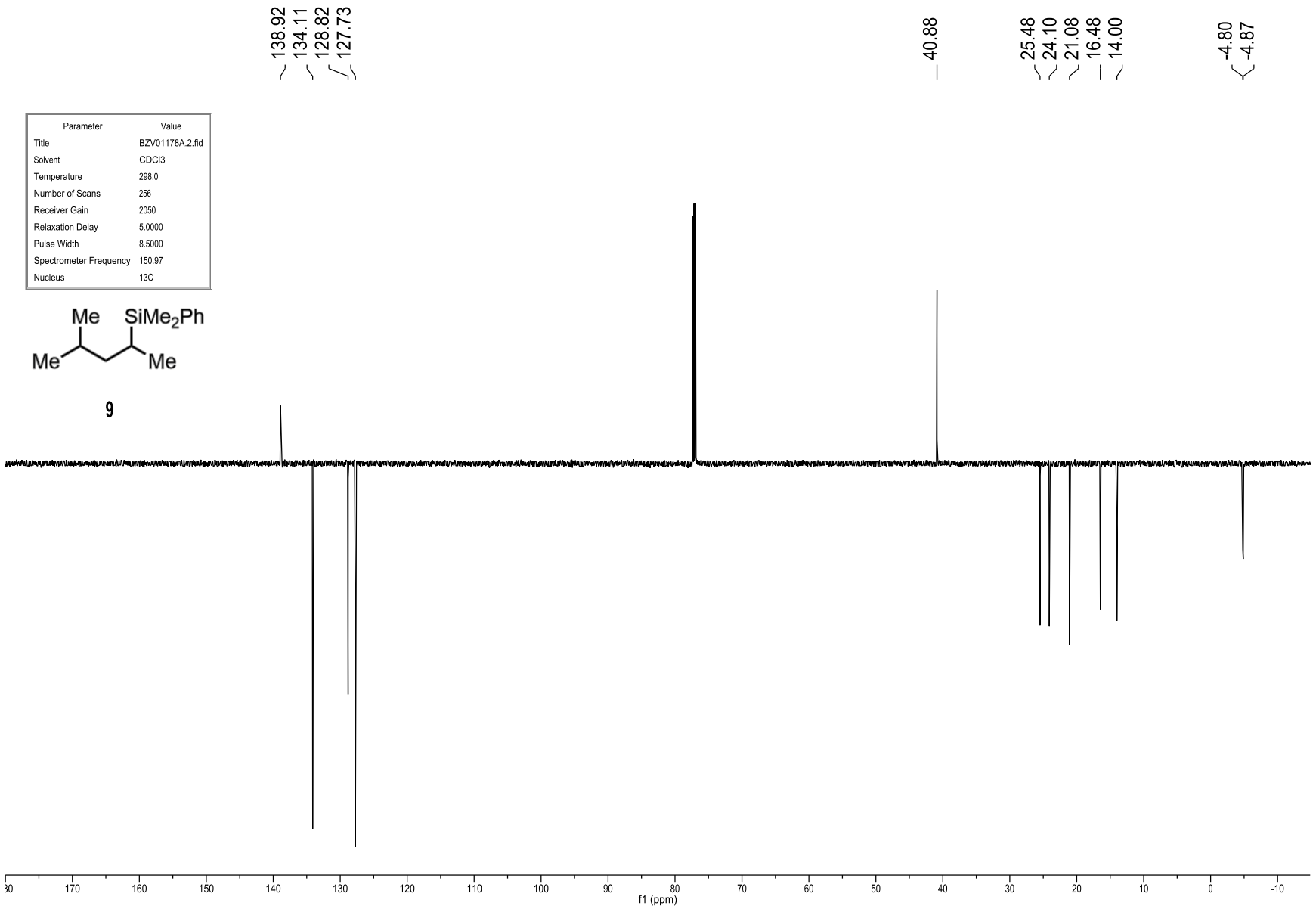

139.07
134.08
128.80
127.72
32.06
31.74
29.81
29.75
29.50
28.69
22.84
19.17
14.27
14.17
-4.66
Parameter Value
Title APC 05032.2.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
Me
SiMe2Ph
Me
8
f1 (ppm)

Parameter	Value
Title	APC 05032.3.fid
Solvent	CDCl3
Temperature	298.0
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

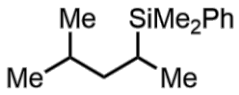


8

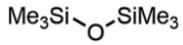

7.52 7.51 7.51 7.50 7.50 7.50 7.50 7.49 7.36 7.36 7.35 7.35 7.35 7.34 7.33
1.69 1.69 1.68 1.68 1.68 1.67 1.67 1.66 1.66 1.66 1.65 1.18 1.18 1.17 1.16 1.15 1.14 1.14 1.12 1.11 1.10 1.09 1.09 1.08 1.07 0.99 0.98 0.98 0.97 0.97 0.97 0.96 0.96 0.95 0.95 0.94 0.90 0.89 0.86 0.85 0.80 0.79 0.25 0.24
Parameter Value
Title BZV01178A.1.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 161
Relaxation Delay 1.0000
Pulse Width 13.5000
Spectrometer Frequency 600.32
Nucleus 1H
Me SiMe2Ph
Me Me
9
2.02 3.04 1.02 1.01 1.06 1.00 3.14 3.25 3.17 2.90 3.08
8.5 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 -0.5
f1 (ppm)


138.92
134.11
128.82
127.73
40.88
25.48
24.10
21.08
16.48
14.00
-4.80
-4.87
Parameter Value
Title BZV01178A.2.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
SiMe2Ph
Me
Me
9
f1 (ppm)

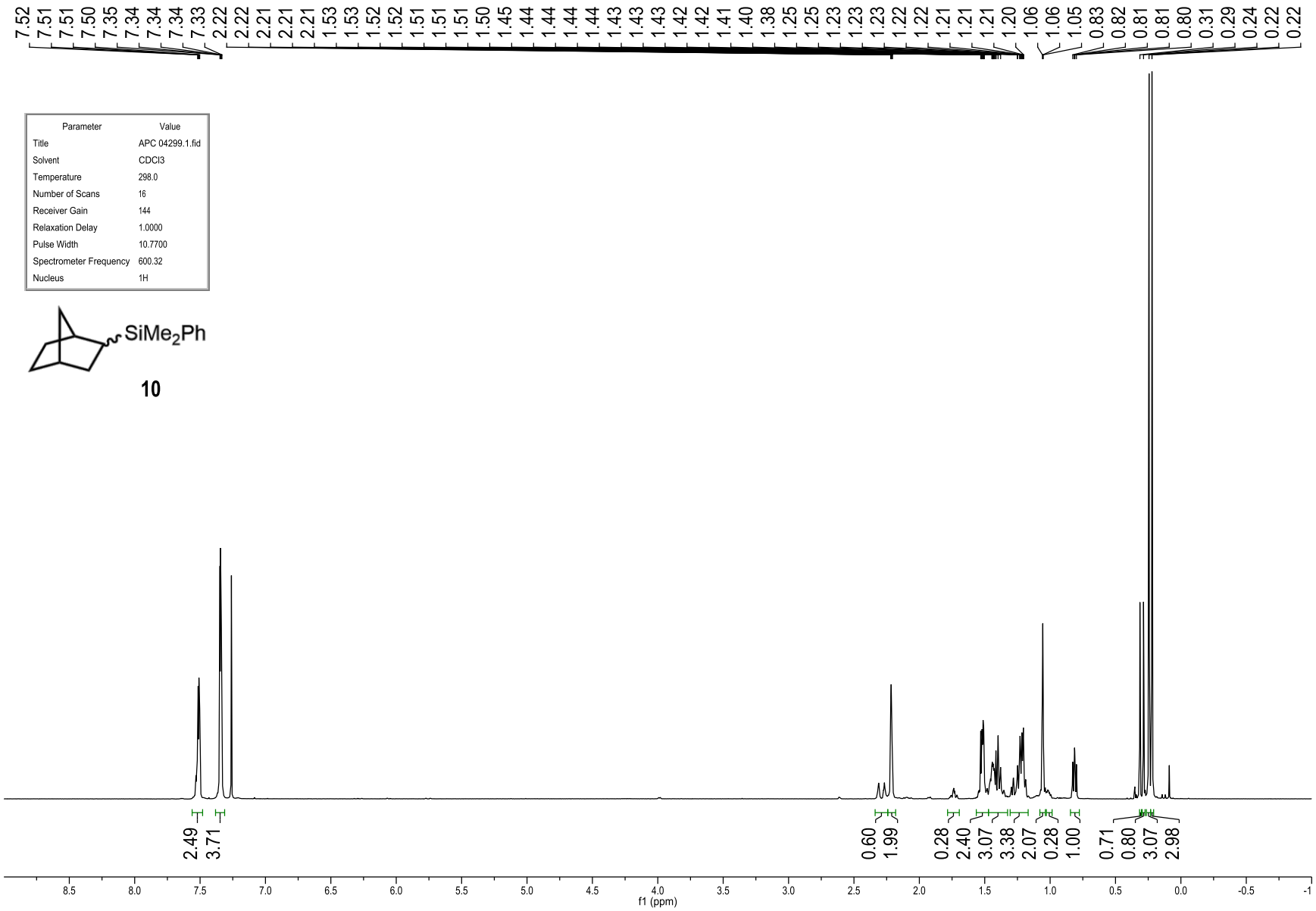
Parameter	Value
Title	BZV01178ASi.1.fid
Solvent	CDCl3
Temperature	298.0
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si

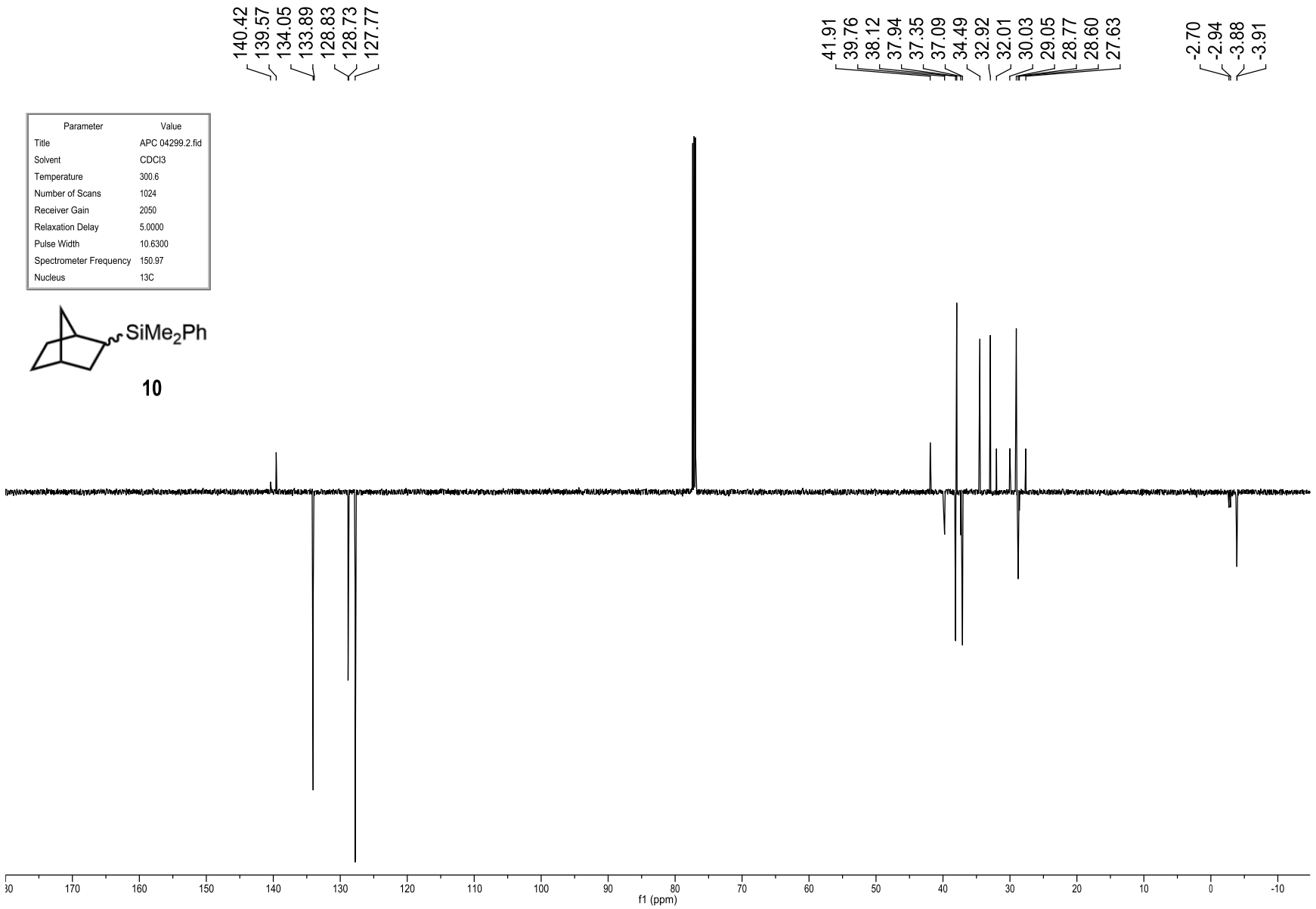


9

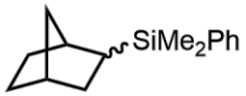


0.16

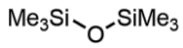

7.52 7.51 7.51 7.50 7.35 7.34 7.34 7.34 7.33 2.22 2.22 2.21 2.21 2.21 1.53 1.53 1.52 1.52 1.51 1.51 1.51 1.50 1.45 1.44 1.44 1.44 1.44 1.43 1.43 1.43 1.42 1.42 1.41 1.40 1.38 1.25 1.25 1.23 1.23 1.23 1.22 1.22 1.21 1.21 1.21 1.20 1.06 1.06 1.05 0.83 0.82 0.81 0.81 0.80 0.31 0.29 0.24 0.22 0.22
Parameter Value
Title APC 04299.1.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 144
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Ph
10
2.49 3.71 0.60 1.99 0.28 2.40 3.07 3.38 2.07 0.28 1.00 0.71 0.80 3.07 2.98
8.5 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 -0.5 -1
f1 (ppm)

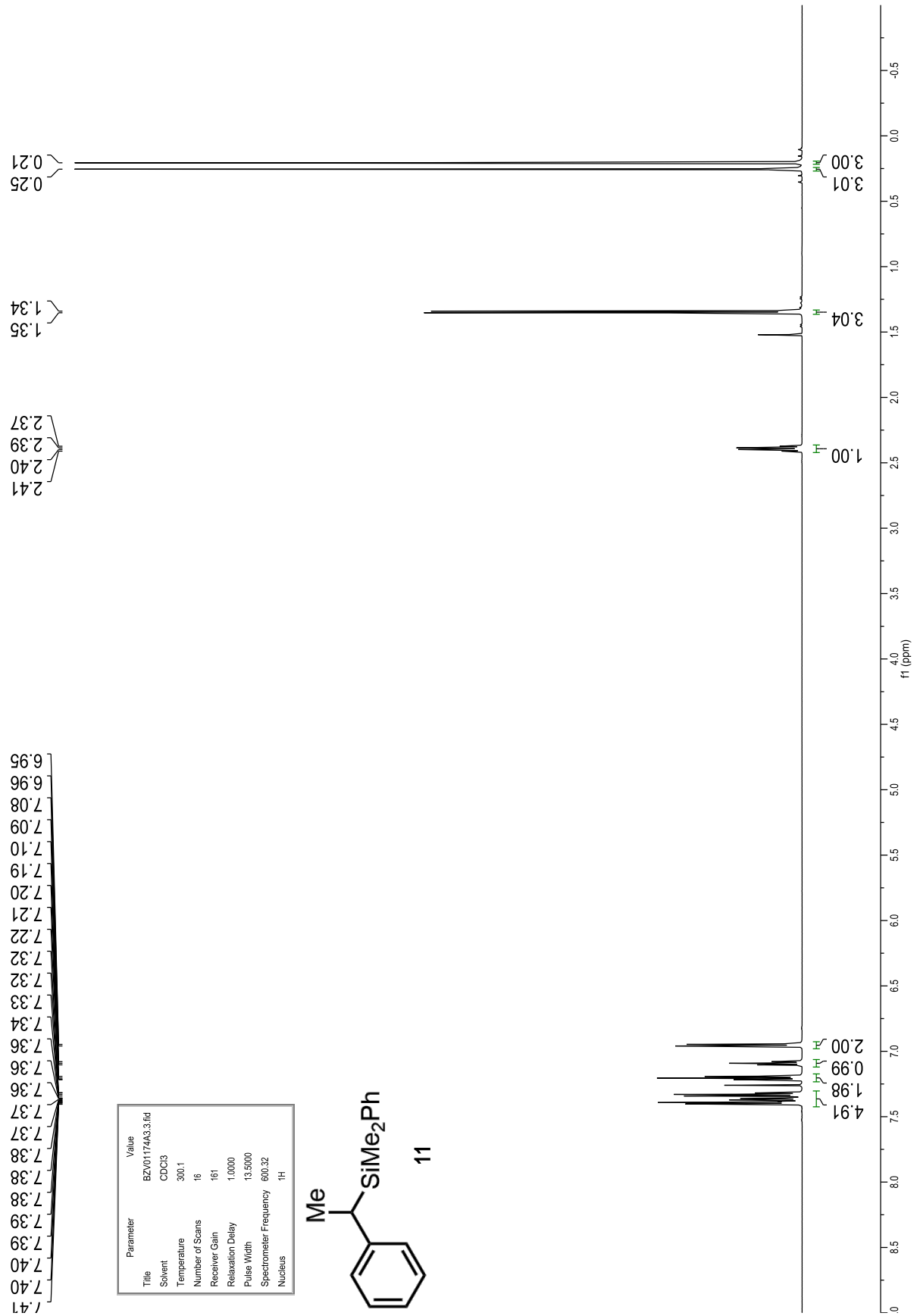

140.42
139.57
134.05
133.89
128.83
128.73
127.77
41.91
39.76
38.12
37.94
37.35
37.09
34.49
32.92
32.01
30.03
29.05
28.77
28.60
27.63
-2.70
-2.94
-3.88
-3.91
Parameter Value
Title APC 04299.2.fid
Solvent CDCl3
Temperature 300.6
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Ph
10
f1 (ppm)

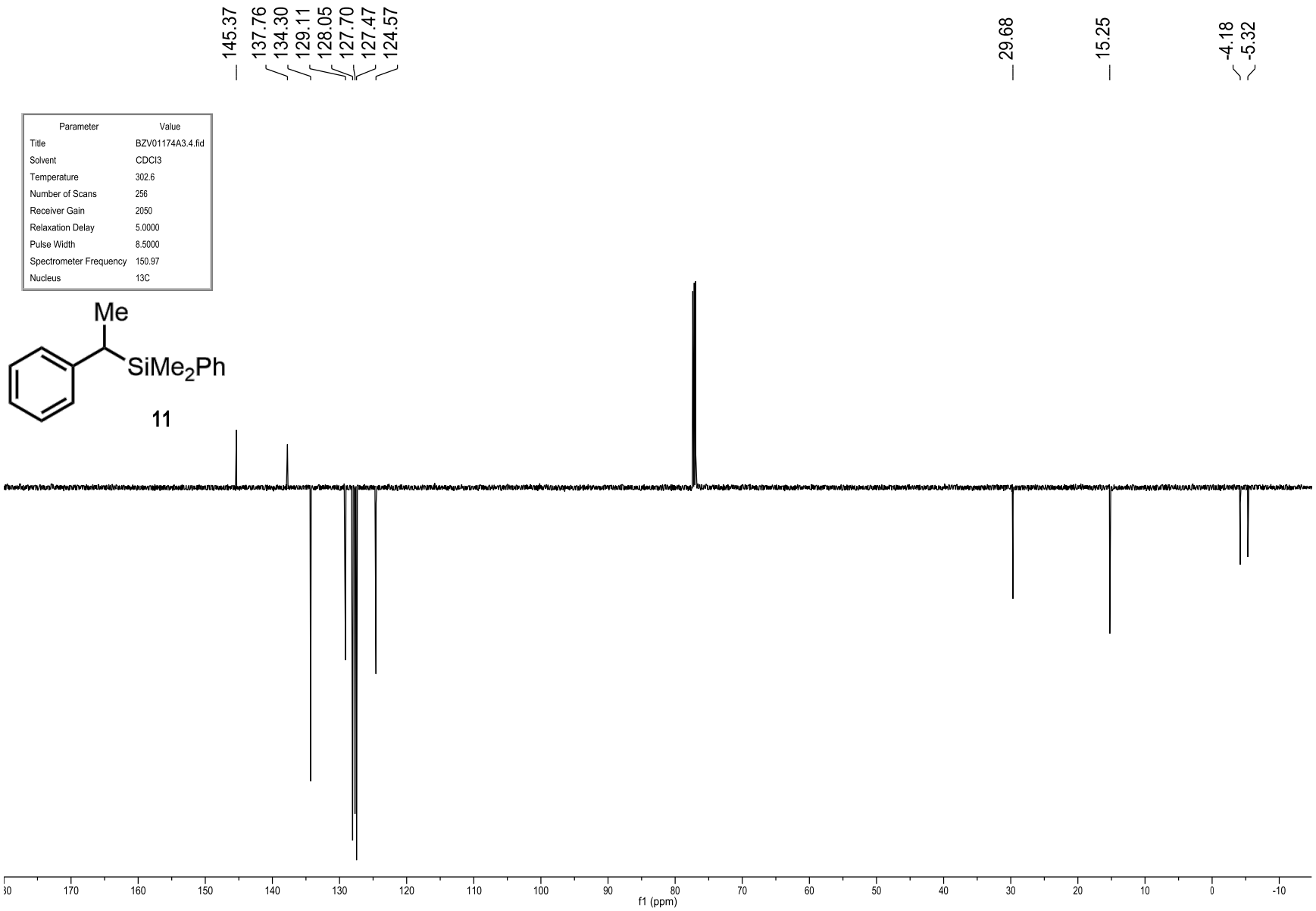
Parameter	Value
Title	APC 04299.3.fid
Solvent	CDCl3
Temperature	297.7
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si



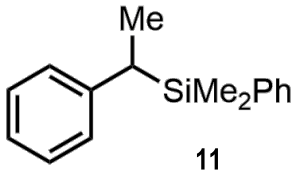
10



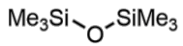

Parameter Value
Title BZV01174A3.3.fid
Solvent CDCl3
Temperature 300.1
Number of Scans 16
Receiver Gain 161
Relaxation Delay 1.0000
Pulse Width 13.5000
Spectrometer Frequency 600.32
Nucleus 1H
Me
SiMe2Ph
11
f1 (ppm)

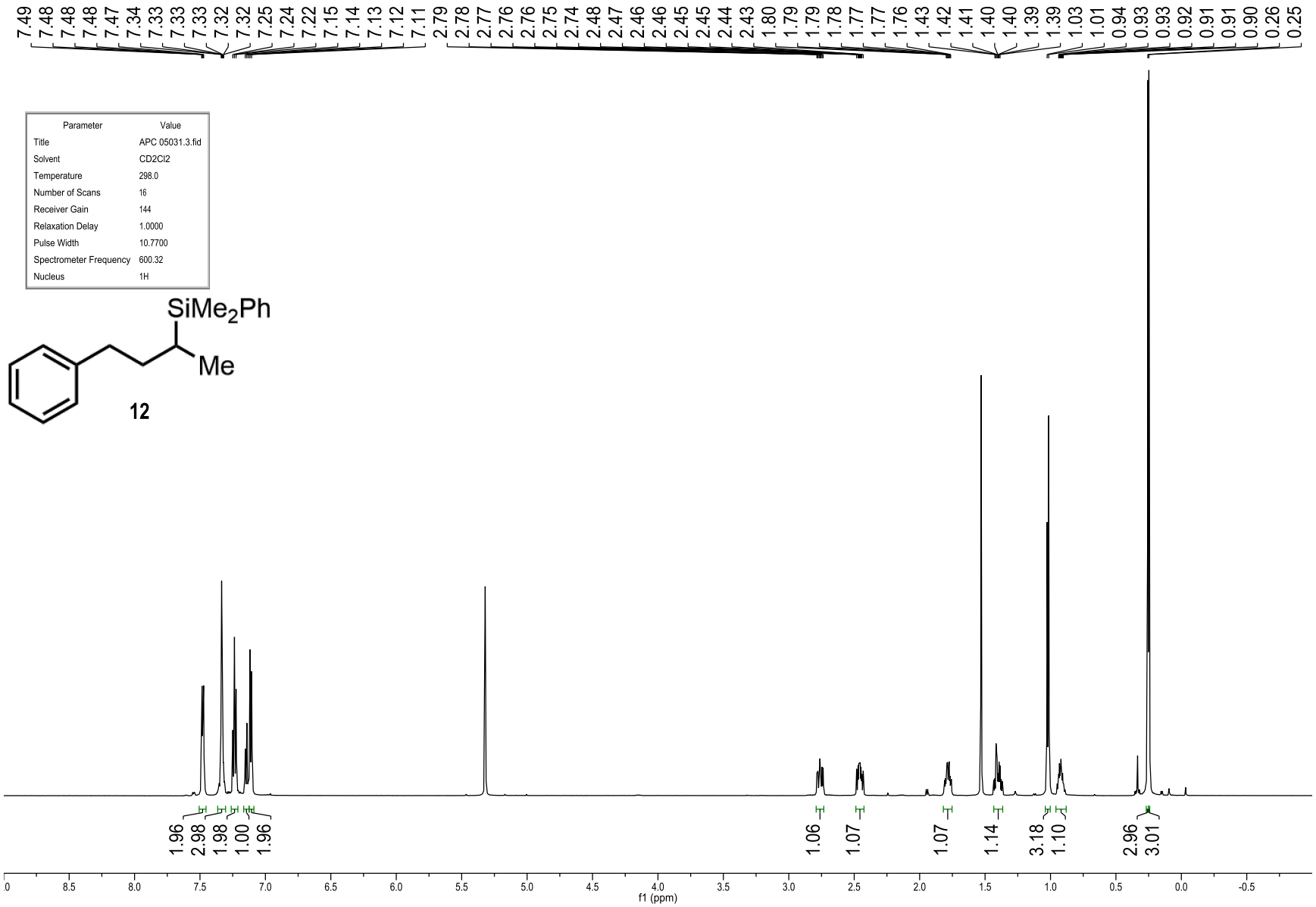

145.37
137.76
134.30
129.11
128.05
127.70
127.47
124.57
29.68
15.25
-4.18
-5.32
Parameter Value
Title BZV01174A3.4.fid
Solvent CDCl3
Temperature 302.6
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
SiMe2Ph
11
f1 (ppm)

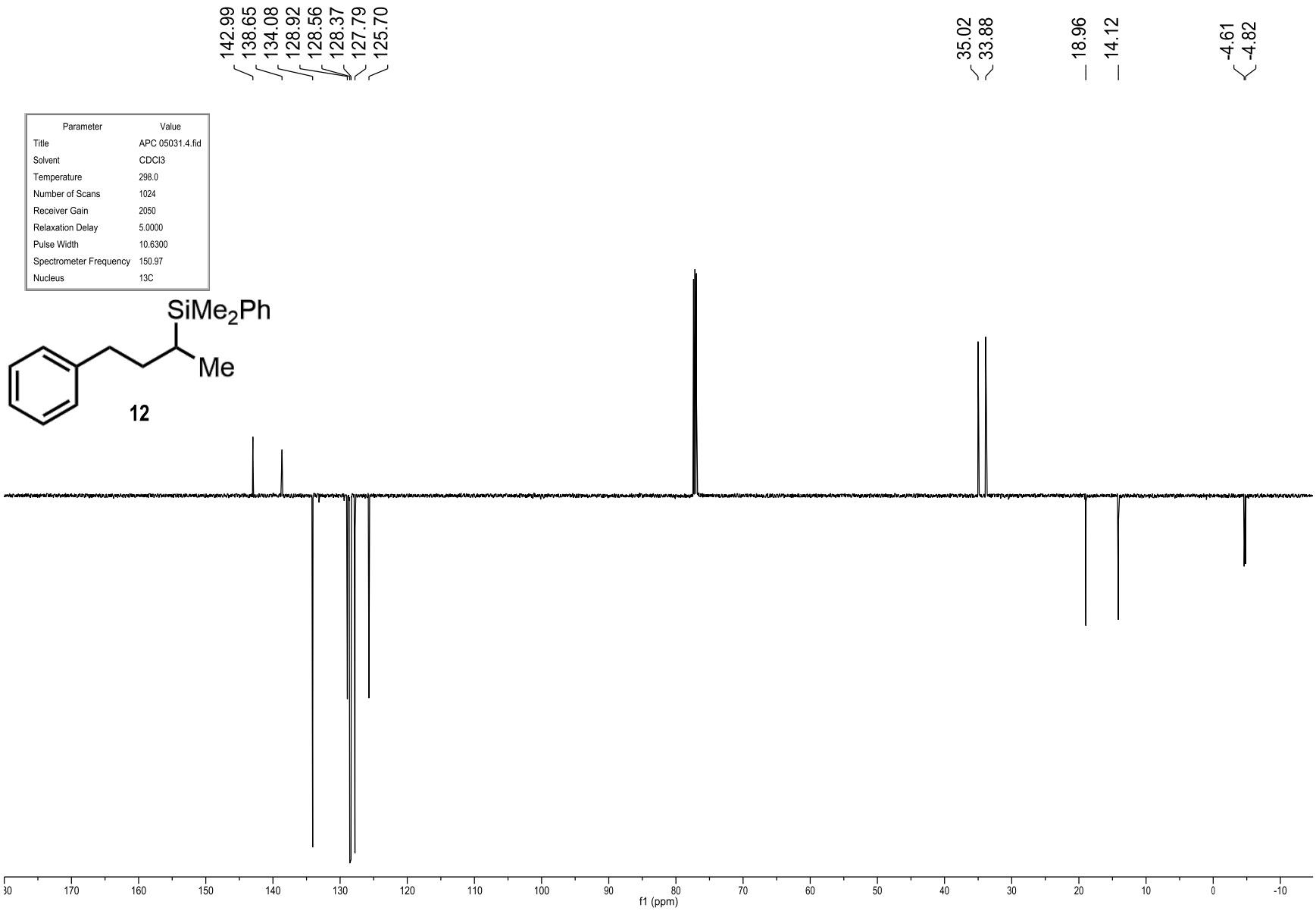
Parameter	Value
Title	BZV01174A3Si.1.fid
Solvent	CDCl3
Temperature	299.2
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si



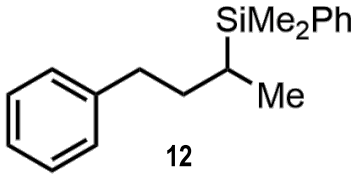
11



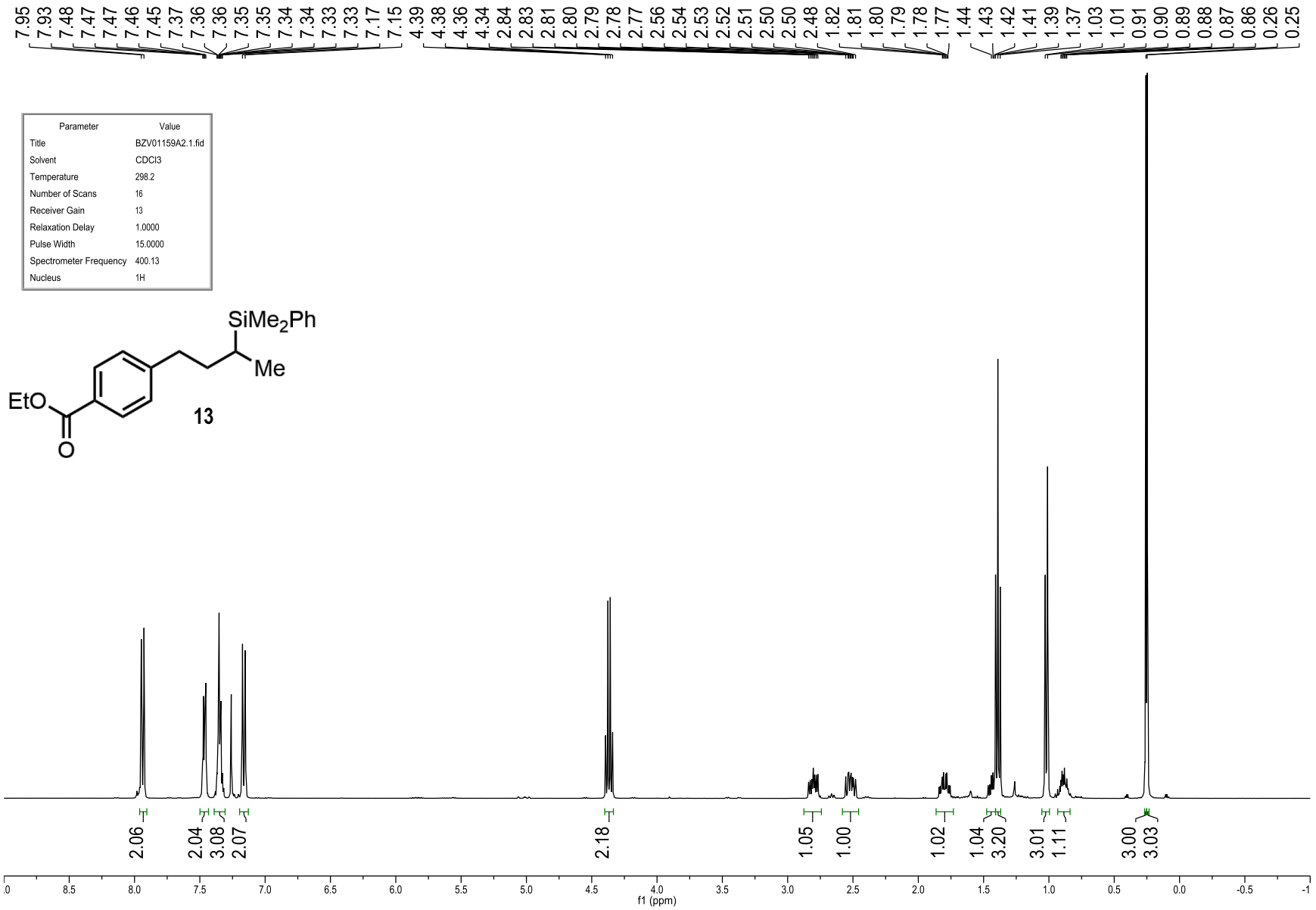

7.49 7.48 7.48 7.48 7.47 7.34 7.33 7.33 7.33 7.32 7.32 7.25 7.24 7.22 7.15 7.14 7.13 7.12 7.11
2.79 2.78 2.77 2.76 2.76 2.75 2.74 2.48 2.47 2.46 2.46 2.45 2.45 2.44 2.43 1.80 1.79 1.79 1.78 1.77 1.77 1.76 1.43 1.42 1.41 1.40 1.40 1.39 1.39 1.03 1.01 0.94 0.93 0.93 0.92 0.91 0.91 0.90 0.26 0.25
Parameter Value
Title APC 05031.3.fid
Solvent CD2Cl2
Temperature 298.0
Number of Scans 16
Receiver Gain 144
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Ph
Me
12
1.96 2.98 1.98 1.00 1.96
1.06 1.07 1.07 1.14 3.18 1.10 2.96 3.01
f1 (ppm)

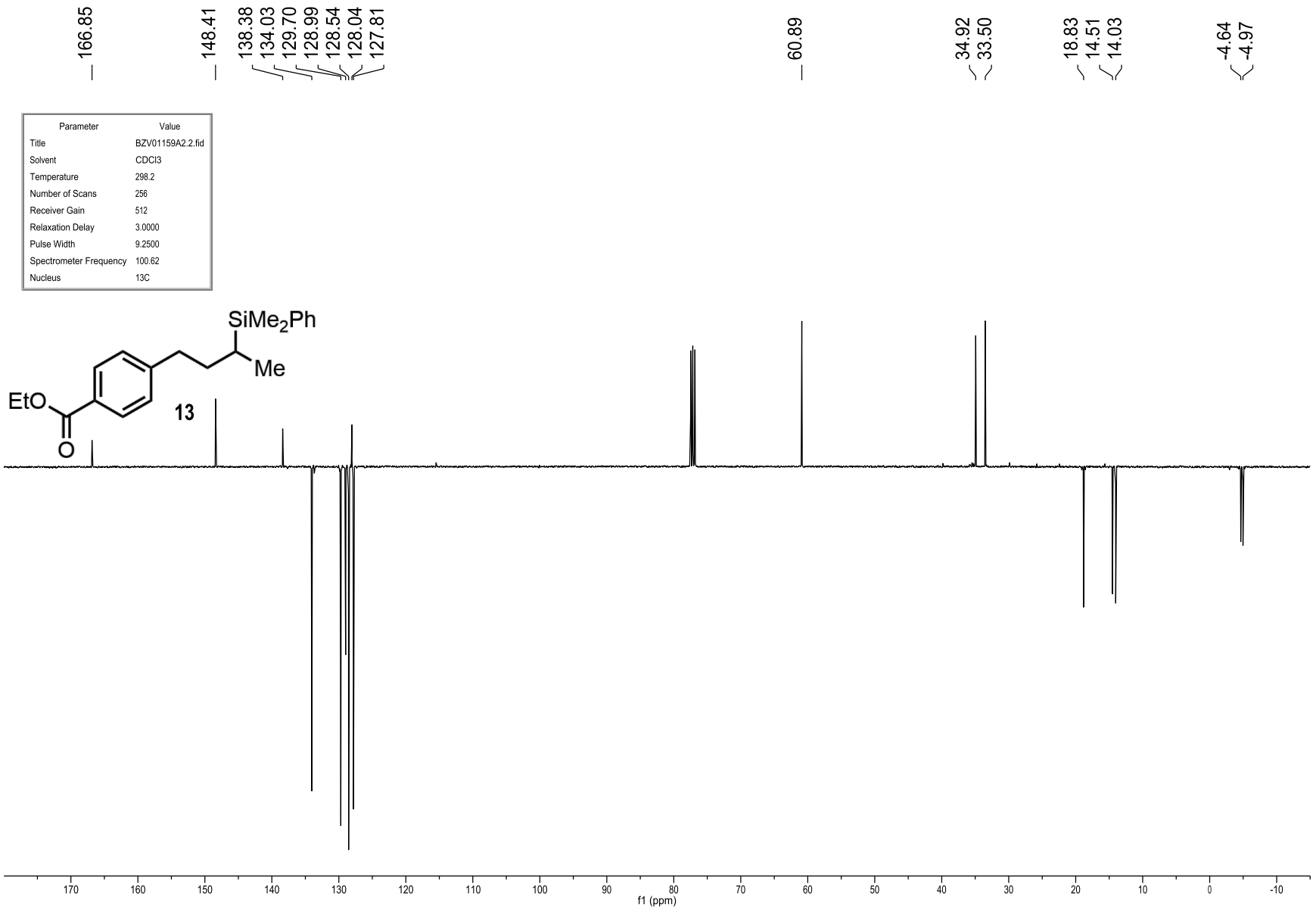

142.99
138.65
134.08
128.92
128.56
128.37
127.79
125.70
35.02
33.88
18.96
14.12
-4.61
-4.82
Parameter Value
Title APC 05031.4.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Ph
Me
12
f1 (ppm)

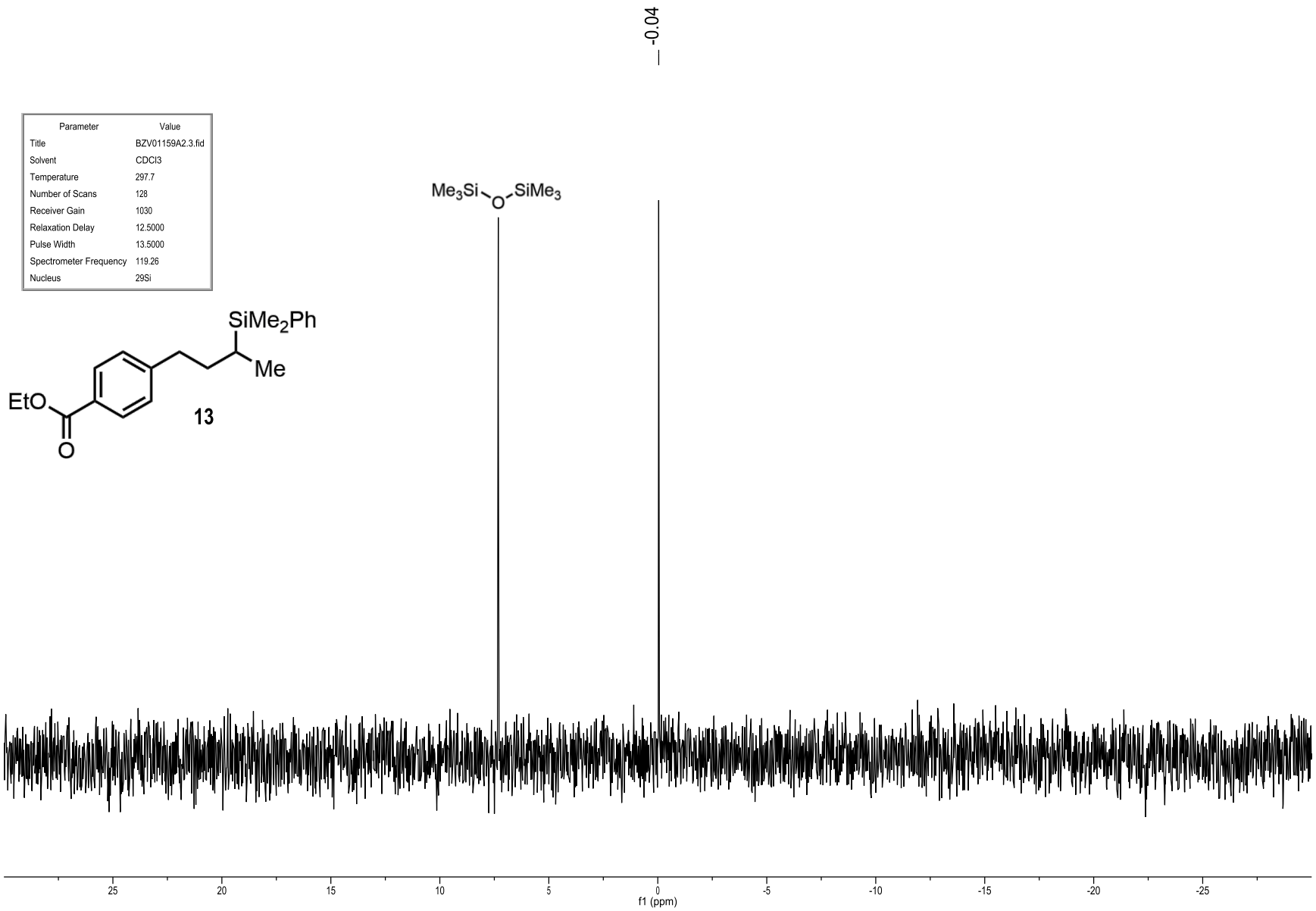
Parameter	Value
Title	APC 05031.5.fid
Solvent	CDCl3
Temperature	298.0
Number of Scans	128
Receiver Gain	1820
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

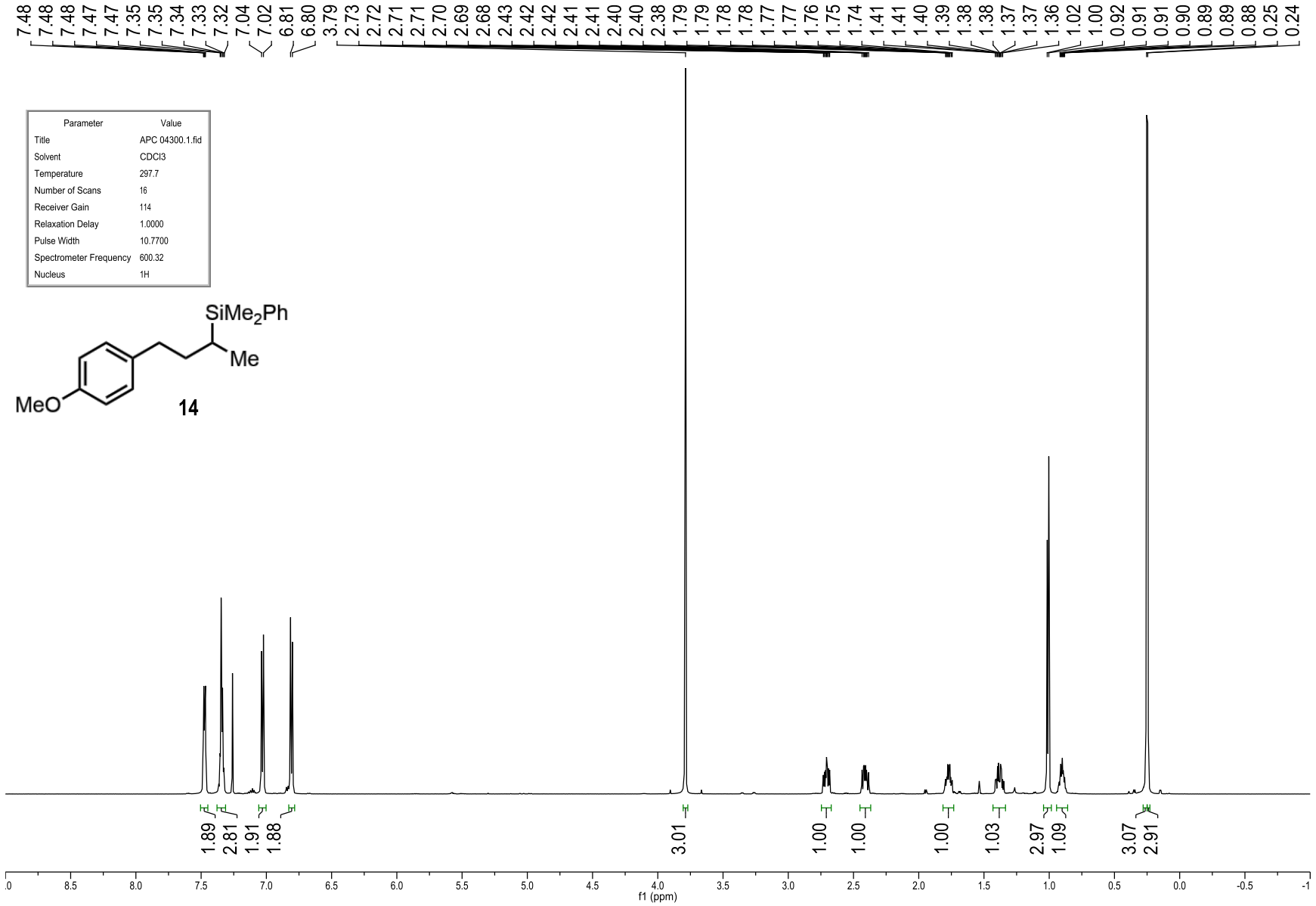


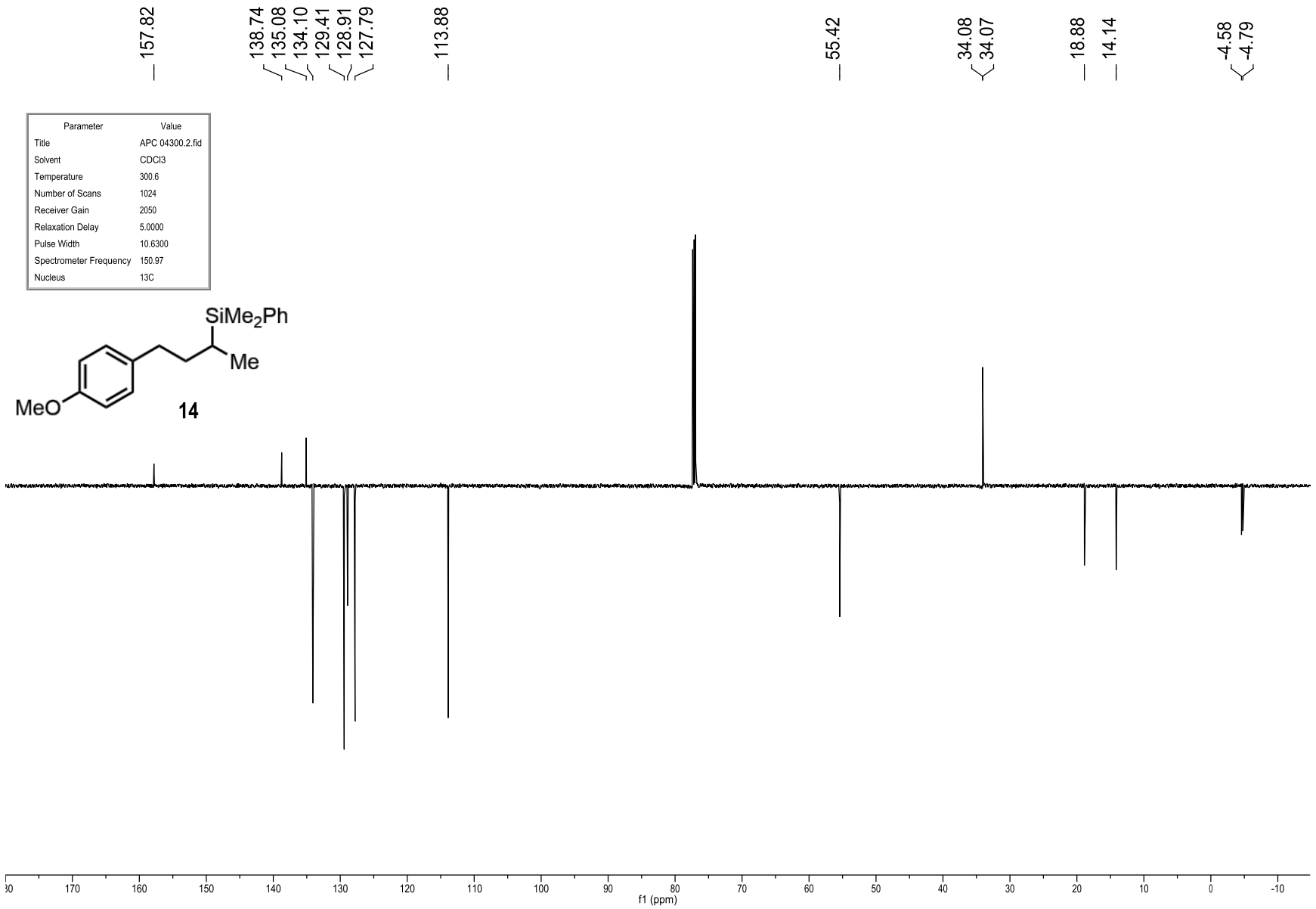
12


7.95 7.93 7.48 7.47 7.47 7.46 7.45 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.17 7.15
4.39 4.38 4.36 4.34 2.84 2.83 2.81 2.80 2.79 2.78 2.77 2.56 2.54 2.53 2.52 2.51 2.50 2.50 2.48 1.82 1.81 1.80 1.79 1.78 1.77
1.44 1.43 1.42 1.41 1.39 1.37 1.03 1.01 0.91 0.90 0.89 0.88 0.87 0.86 0.26 0.25
Parameter Value
Title BZV01159A2.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 13
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
SiMe2Ph
Me
EtO
O
13
2.06 2.04 3.08 2.07 2.18 1.05 1.00 1.02 1.04 3.20 3.01 1.11 3.00 3.03
f1 (ppm)


166.85
148.41
138.38
134.03
129.70
128.99
128.54
128.04
127.81
60.89
34.92
33.50
18.83
14.51
14.03
-4.64
-4.97
Parameter Value
Title BZV01159A2.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
SiMe2Ph
Me
EtO
O
13
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

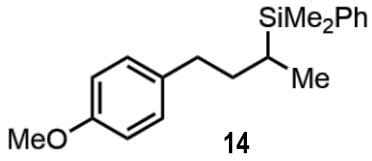

-0.04
Me3Si O SiMe3
Parameter Value
Title BZV01159A2.3.fid
Solvent CDCl3
Temperature 297.7
Number of Scans 128
Receiver Gain 1030
Relaxation Delay 12.5000
Pulse Width 13.5000
Spectrometer Frequency 119.26
Nucleus 29Si
SiMe2Ph
Me
EtO
O
13
25 20 15 10 5 0 -5 -10 -15 -20 -25
f1 (ppm)


7.48 7.48 7.48 7.47 7.47 7.35 7.35 7.34 7.33 7.32 7.04 7.02 6.81 6.80 3.79 2.73 2.72 2.71 2.71 2.70 2.69 2.68 2.43 2.42 2.42 2.41 2.41 2.40 2.40 2.38 1.79 1.79 1.78 1.78 1.77 1.77 1.76 1.75 1.74 1.41 1.41 1.40 1.39 1.38 1.38 1.37 1.37 1.36 1.02 1.00 0.92 0.91 0.91 0.90 0.89 0.89 0.88 0.25 0.24
Parameter Value
Title APC 04300.1.fid
Solvent CDCl3
Temperature 297.7
Number of Scans 16
Receiver Gain 114
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Ph
Me
MeO
14
1.89 2.81 1.91 1.88 3.01 1.00 1.00 1.00 1.03 2.97 1.09 3.07 2.91
f1 (ppm)

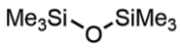

157.82
138.74
135.08
134.10
129.41
128.91
127.79
113.88
55.42
34.08
34.07
18.88
14.14
-4.58
-4.79
Parameter Value
Title APC 04300.2.fid
Solvent CDCl3
Temperature 300.6
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Ph
Me
MeO
14
f1 (ppm)

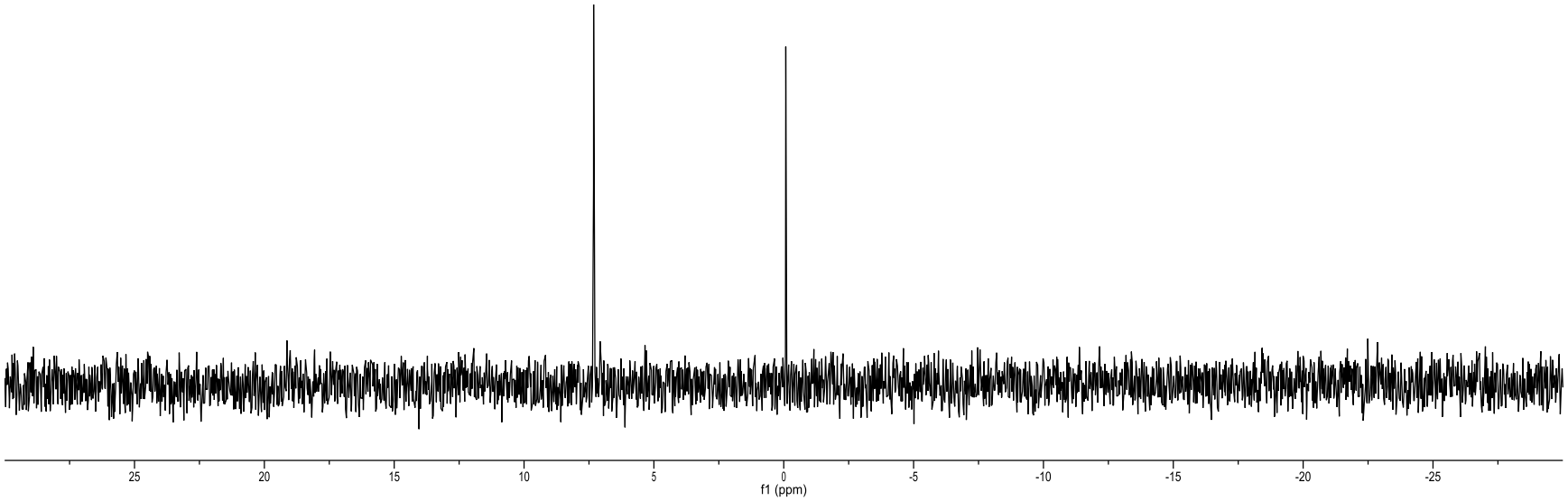
80.0

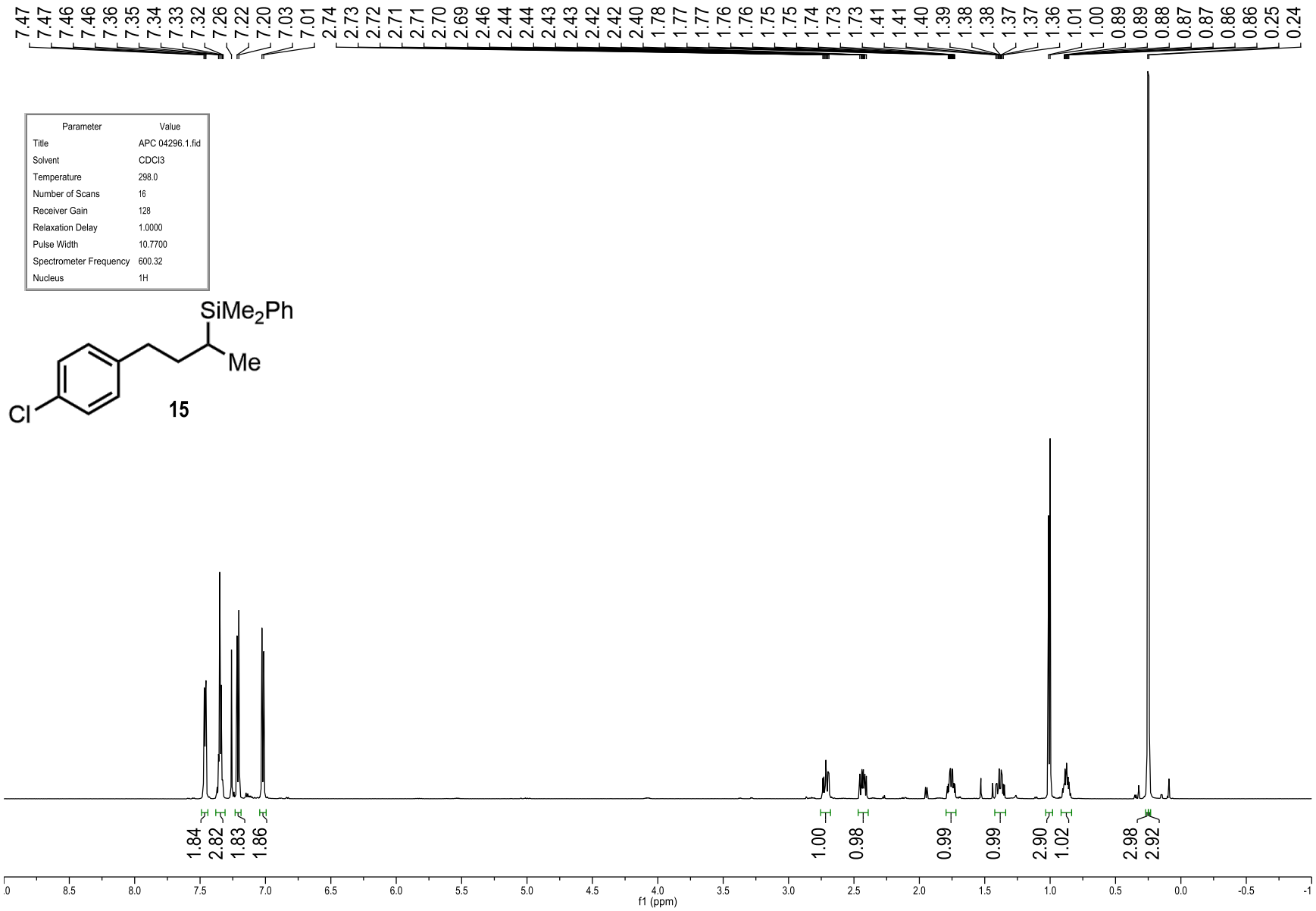
Parameter	Value
Title	APC 04300.3.fid
Solvent	CDCl3
Temperature	297.7
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

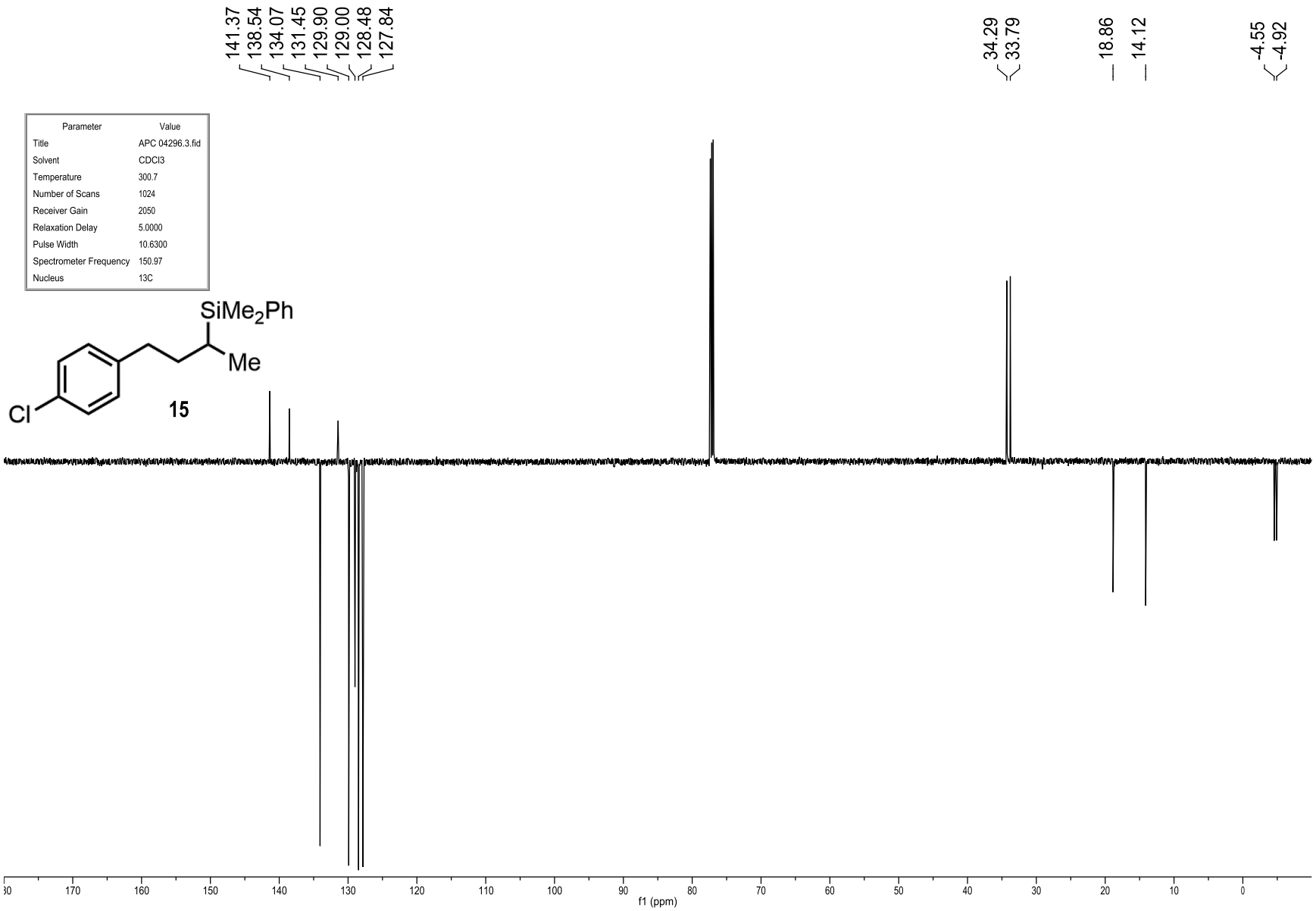


14

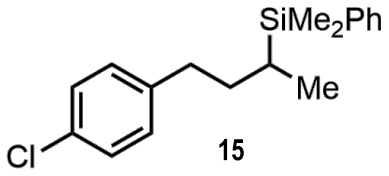




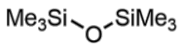

7.47 7.47 7.46 7.46 7.36 7.35 7.34 7.33 7.32 7.26 7.22 7.20 7.03 7.01 2.74 2.73 2.72 2.71 2.71 2.70 2.69 2.46 2.44 2.44 2.43 2.43 2.42 2.42 2.40 1.78 1.77 1.77 1.76 1.76 1.75 1.75 1.74 1.73 1.73 1.41 1.41 1.40 1.39 1.38 1.38 1.37 1.37 1.36 1.01 1.00 0.89 0.89 0.88 0.87 0.87 0.86 0.86 0.25 0.24
Parameter Value
Title APC 04296.1.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 128
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Ph
Me
Cl
15
1.84 2.82 1.83 1.86 1.00 0.98 0.99 0.99 2.90 1.02 2.98 2.92
f1 (ppm)

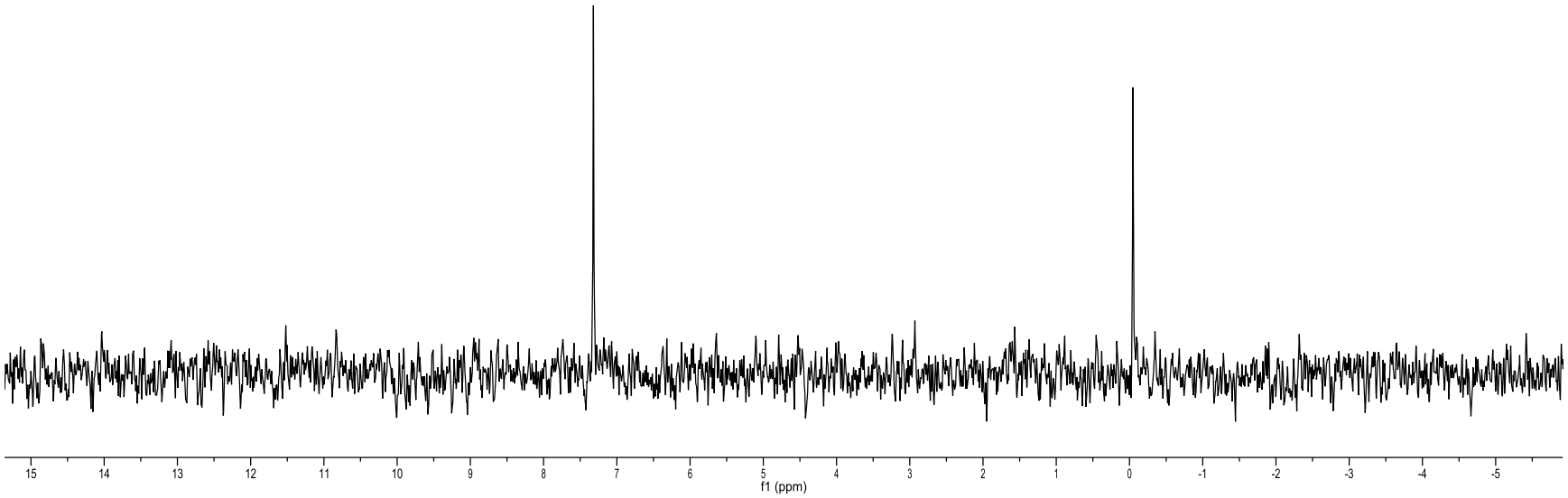

141.37
138.54
134.07
131.45
129.90
129.00
128.48
127.84
34.29
33.79
18.86
14.12
-4.55
-4.92
Parameter Value
Title APC 04296.3.fid
Solvent CDCl3
Temperature 300.7
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Ph
Me
Cl
15
f1 (ppm)

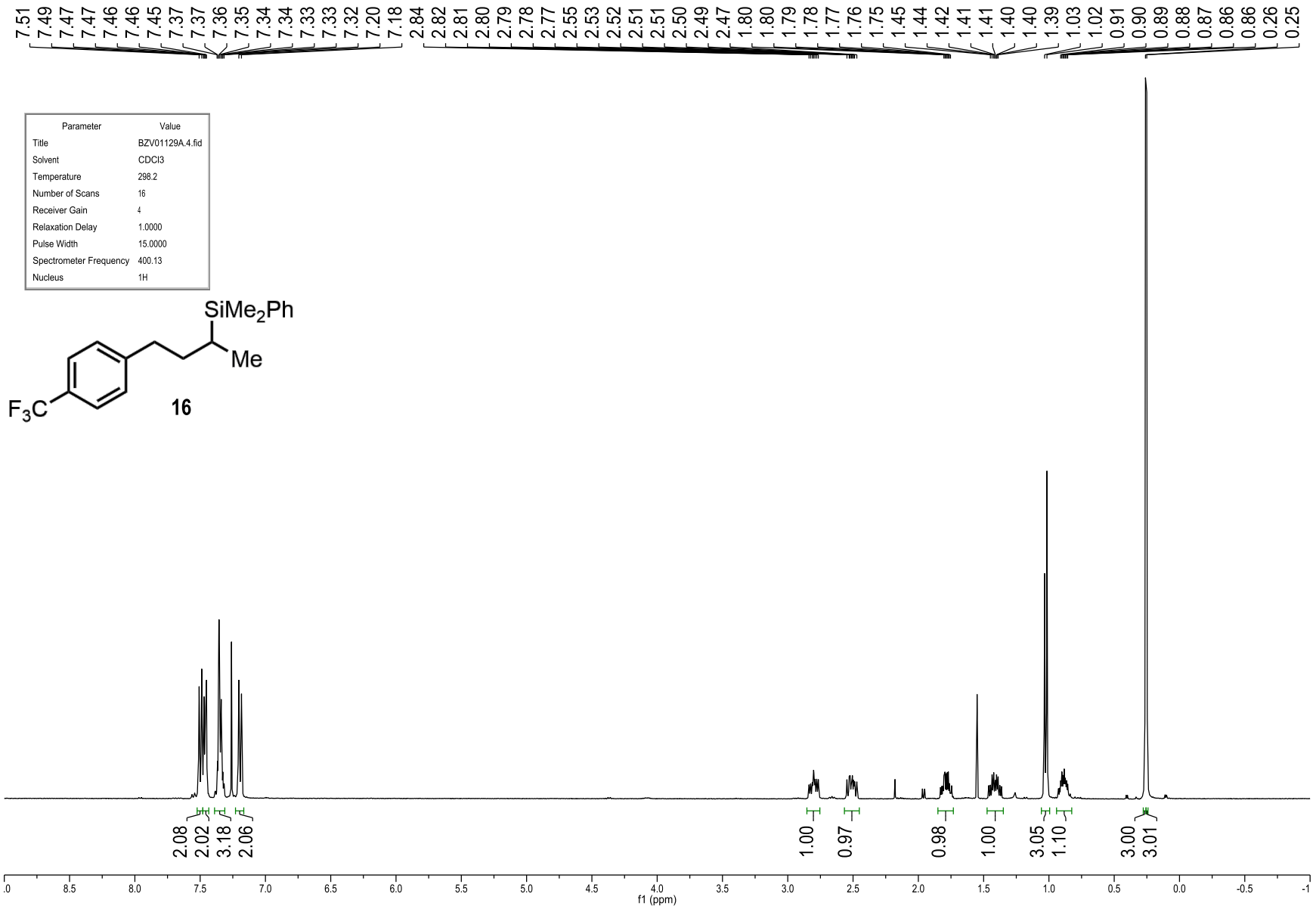
Parameter	Value
Title	APC 04296.4.fid
Solvent	CDCl3
Temperature	297.7
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

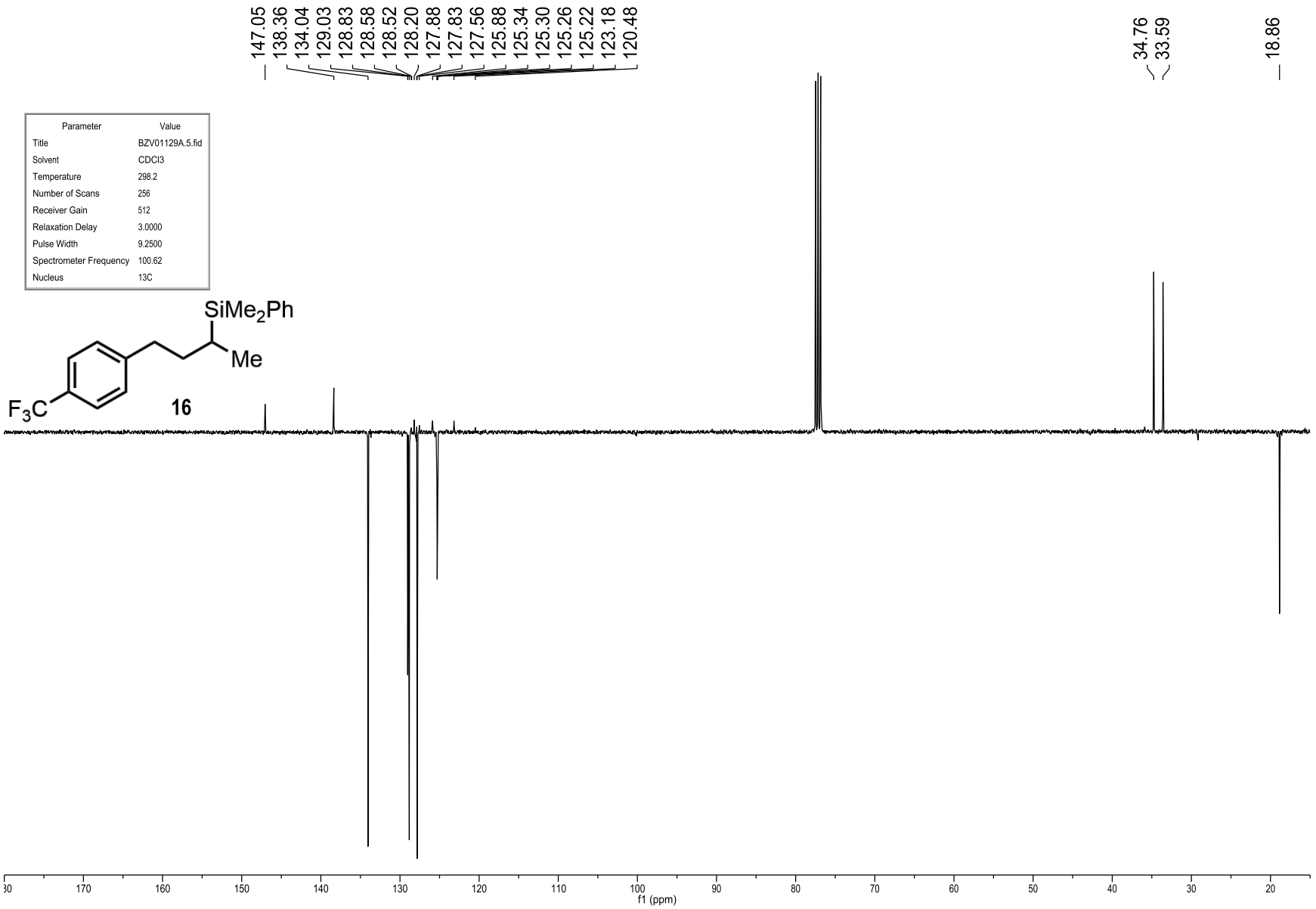


15

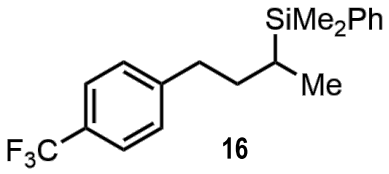





7.51 7.49 7.47 7.47 7.46 7.46 7.45 7.37 7.37 7.36 7.35 7.34 7.34 7.33 7.33 7.32 7.20 7.18
2.84 2.82 2.81 2.80 2.79 2.78 2.77 2.55 2.53 2.52 2.51 2.51 2.50 2.49 2.47 1.80 1.80 1.79 1.78 1.77 1.76 1.75 1.45 1.44 1.42 1.41 1.41 1.40 1.40 1.39 1.03 1.02 0.91 0.90 0.89 0.88 0.87 0.86 0.86 0.26 0.25
Parameter Value
Title BZV01129A.4.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 4
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
SiMe2Ph
Me
F3C
16
2.08 2.02 3.18 2.06
1.00 0.97 0.98 1.00 3.05 1.10 3.00 3.01
f1 (ppm)

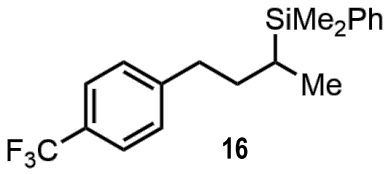

147.05
138.36
134.04
129.03
128.83
128.58
128.52
128.20
127.88
127.83
127.56
125.88
125.34
125.30
125.26
125.22
123.18
120.48
34.76
33.59
18.86
Parameter Value
Title BZV01129A.5.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
SiMe2Ph
Me
F3C
16
f1 (ppm)

Parameter	Value
Title	BZV01129A.6.fid
Solvent	CDCl3
Temperature	298.1
Number of Scans	16
Receiver Gain	1626
Relaxation Delay	2.0000
Pulse Width	15.0300
Spectrometer Frequency	376.46
Nucleus	19F

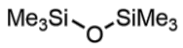


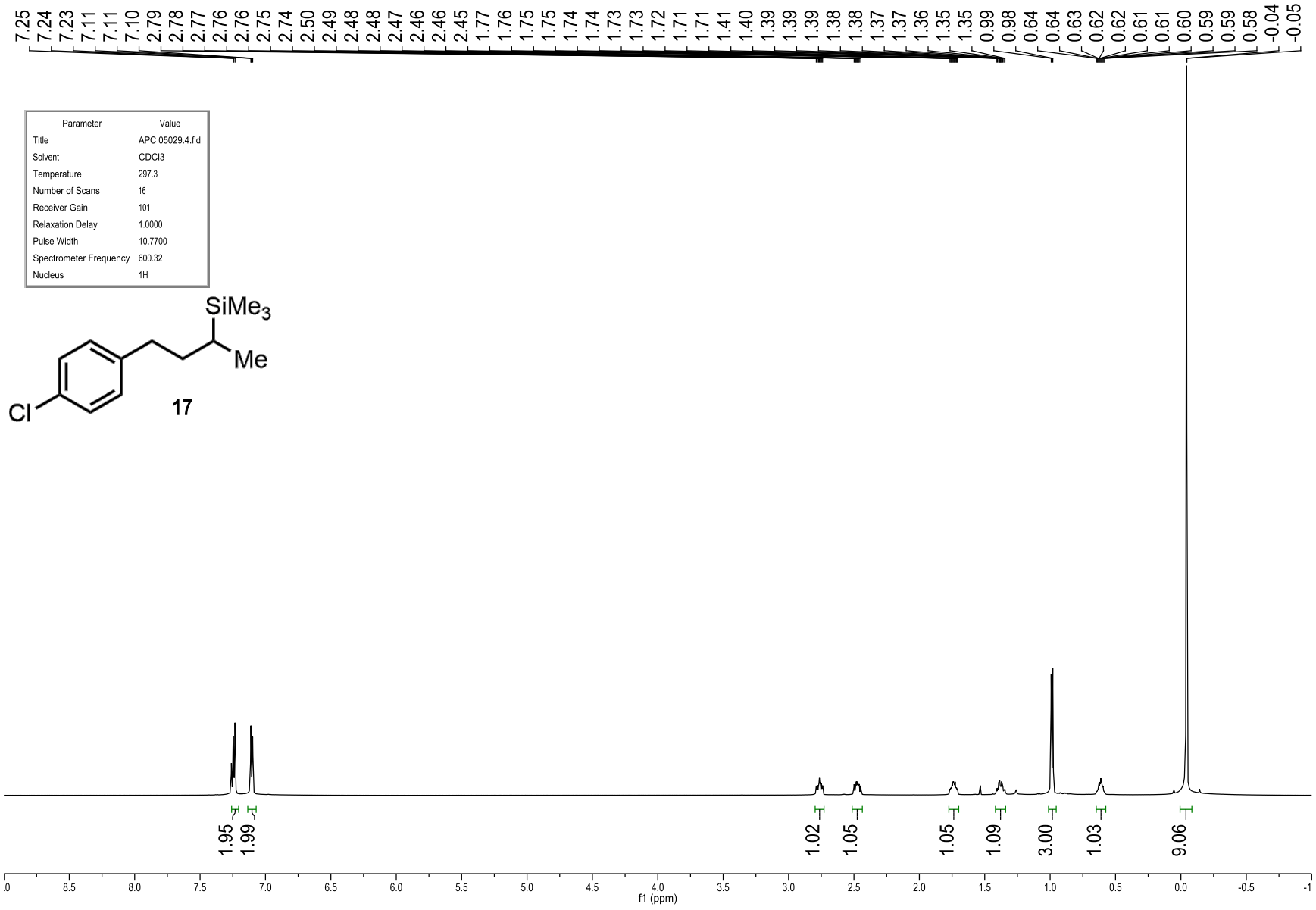
16

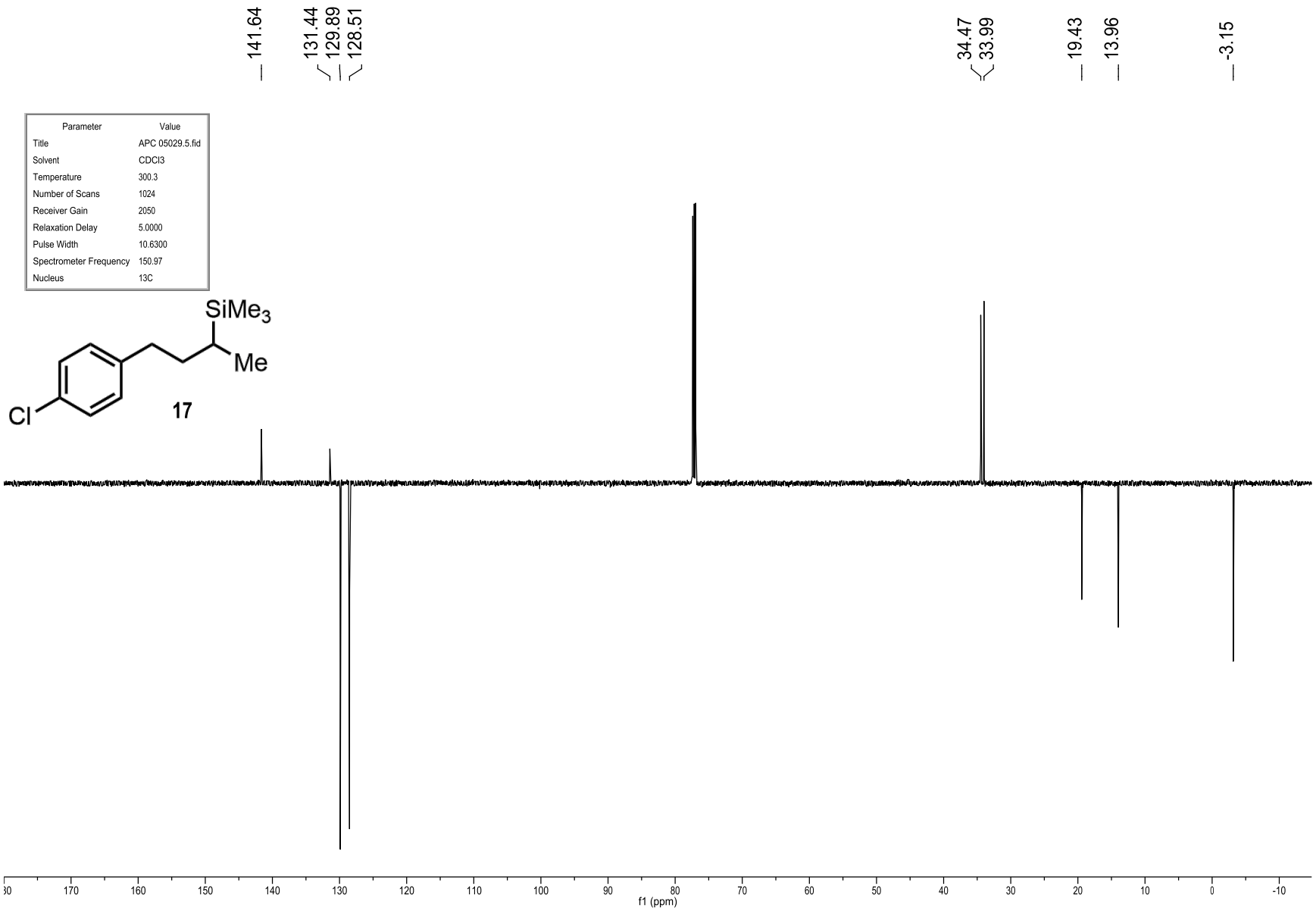
Parameter	Value
Title	BZV01129A.1.fid
Solvent	CDCl3
Temperature	297.5
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si



16

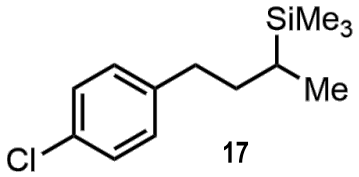



7.25 7.24 7.23 7.11 7.11 7.10 2.79 2.78 2.77 2.76 2.76 2.75 2.74 2.50 2.49 2.48 2.48 2.47 2.46 2.46 2.45 1.77 1.76 1.75 1.75 1.74 1.74 1.73 1.73 1.72 1.71 1.71 1.41 1.40 1.39 1.39 1.39 1.38 1.38 1.37 1.37 1.36 1.35 1.35 0.99 0.98 0.64 0.64 0.63 0.62 0.62 0.61 0.61 0.60 0.59 0.59 0.58 -0.04 -0.05
Parameter Value
Title APC 05029.4.fid
Solvent CDCl3
Temperature 297.3
Number of Scans 16
Receiver Gain 101
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe3
Me
Cl
17
1.95 1.99 1.02 1.05 1.05 1.09 3.00 1.03 9.06
f1 (ppm)

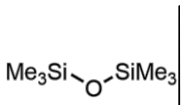

141.64
131.44
129.89
128.51
34.47
33.99
19.43
13.96
-3.15
Parameter Value
Title APC 05029.5.fid
Solvent CDCl3
Temperature 300.3
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe3
Me
Cl
17
f1 (ppm)

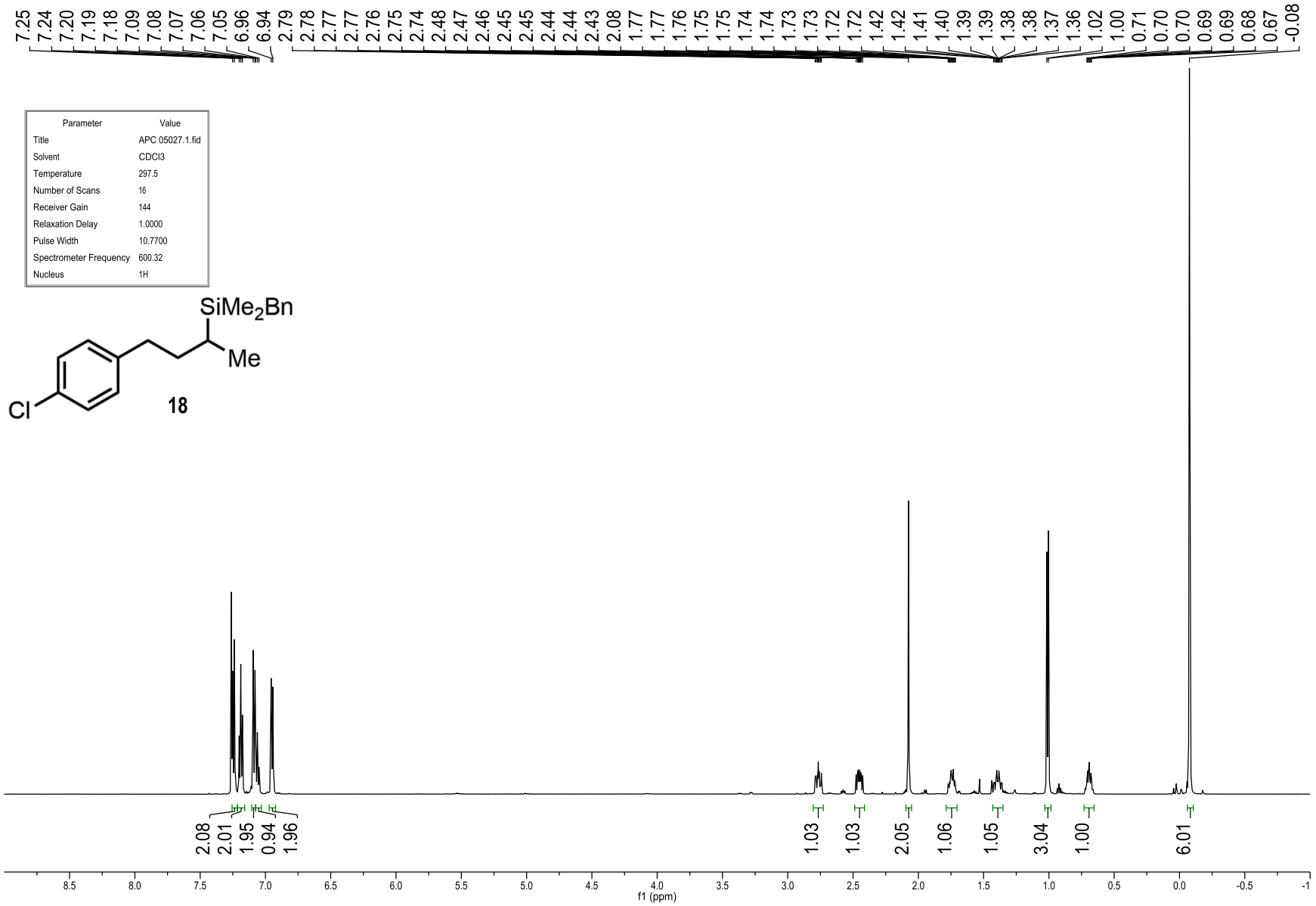
4.55

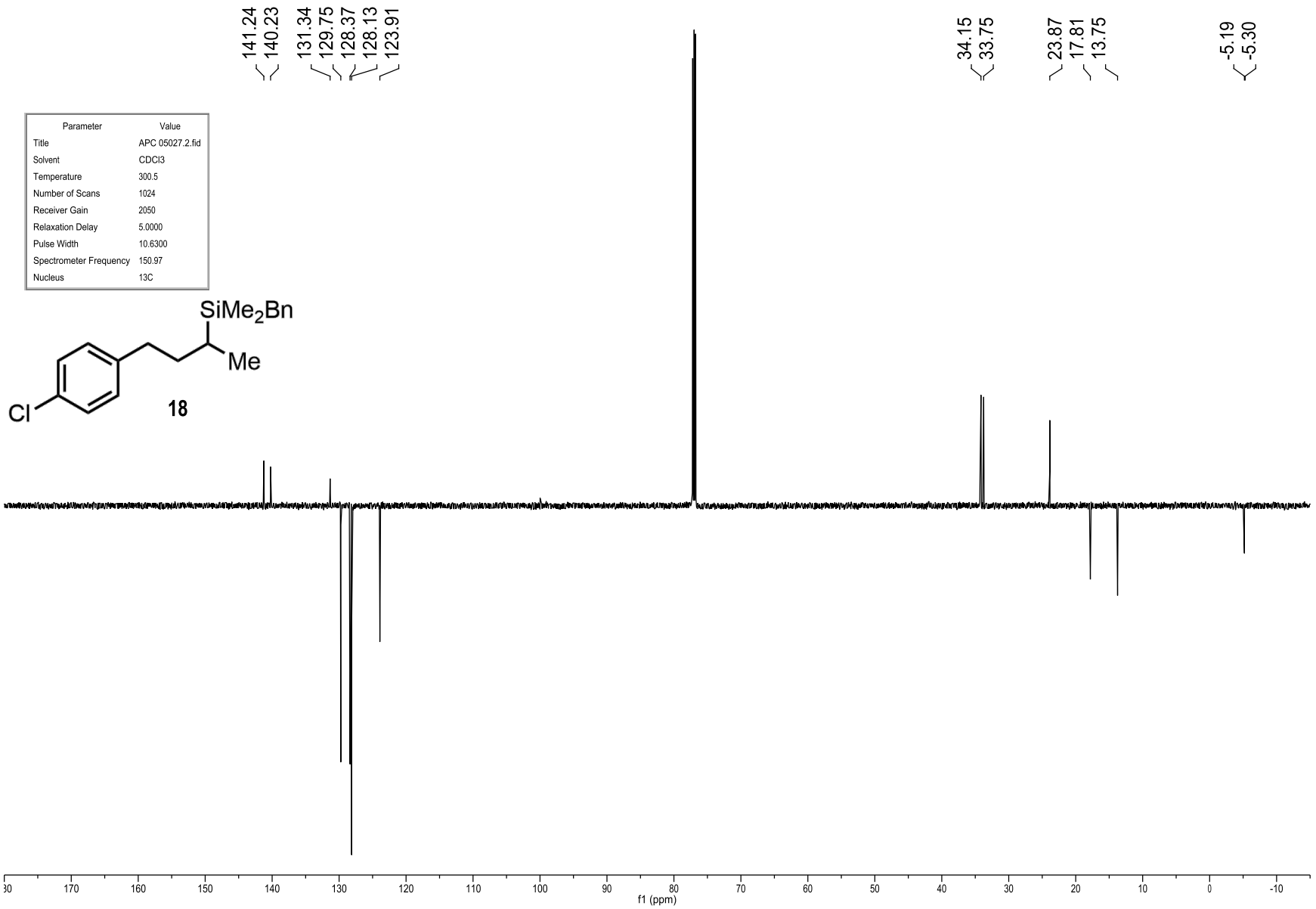
Parameter	Value
Title	APC 05029.6.fid
Solvent	CDCl3
Temperature	297.6
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si



17

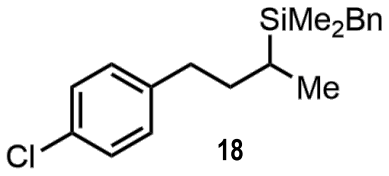



7.25 7.24 7.20 7.19 7.18 7.09 7.08 7.07 7.06 7.05 6.96 6.94 2.79 2.78 2.77 2.77 2.76 2.75 2.74 2.48 2.47 2.46 2.45 2.45 2.44 2.44 2.43 2.08 1.77 1.77 1.76 1.75 1.75 1.74 1.74 1.73 1.73 1.72 1.72 1.42 1.42 1.41 1.40 1.39 1.39 1.38 1.38 1.37 1.36 1.02 1.00 0.71 0.70 0.70 0.69 0.69 0.68 0.67 -0.08
Parameter Value
Title APC 05027.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 16
Receiver Gain 144
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiMe2Bn
Me
Cl
18
2.08 2.01 1.95 0.94 1.96 1.03 1.03 2.05 1.06 1.05 3.04 1.00 6.01
8.5 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 -0.5 -1
f1 (ppm)

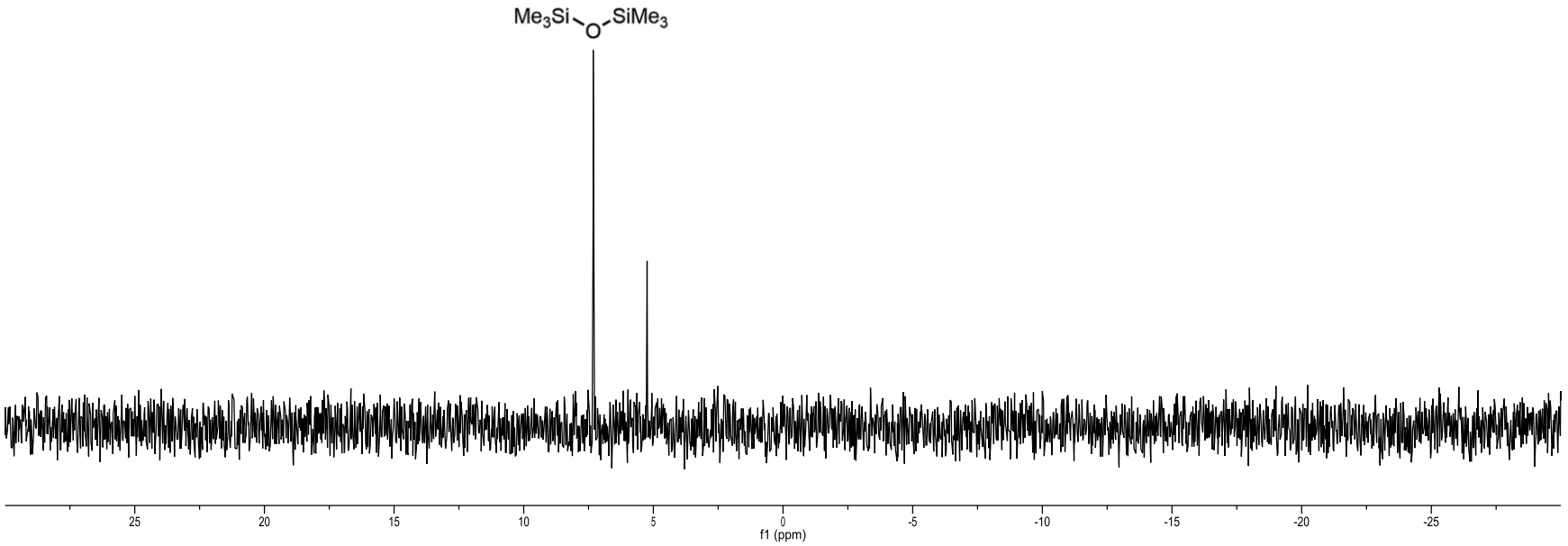

141.24
140.23
131.34
129.75
128.37
128.13
123.91
34.15
33.75
23.87
17.81
13.75
-5.19
-5.30
Parameter Value
Title APC 05027.2.fid
Solvent CDCl3
Temperature 300.5
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiMe2Bn
Me
Cl
18
f1 (ppm)

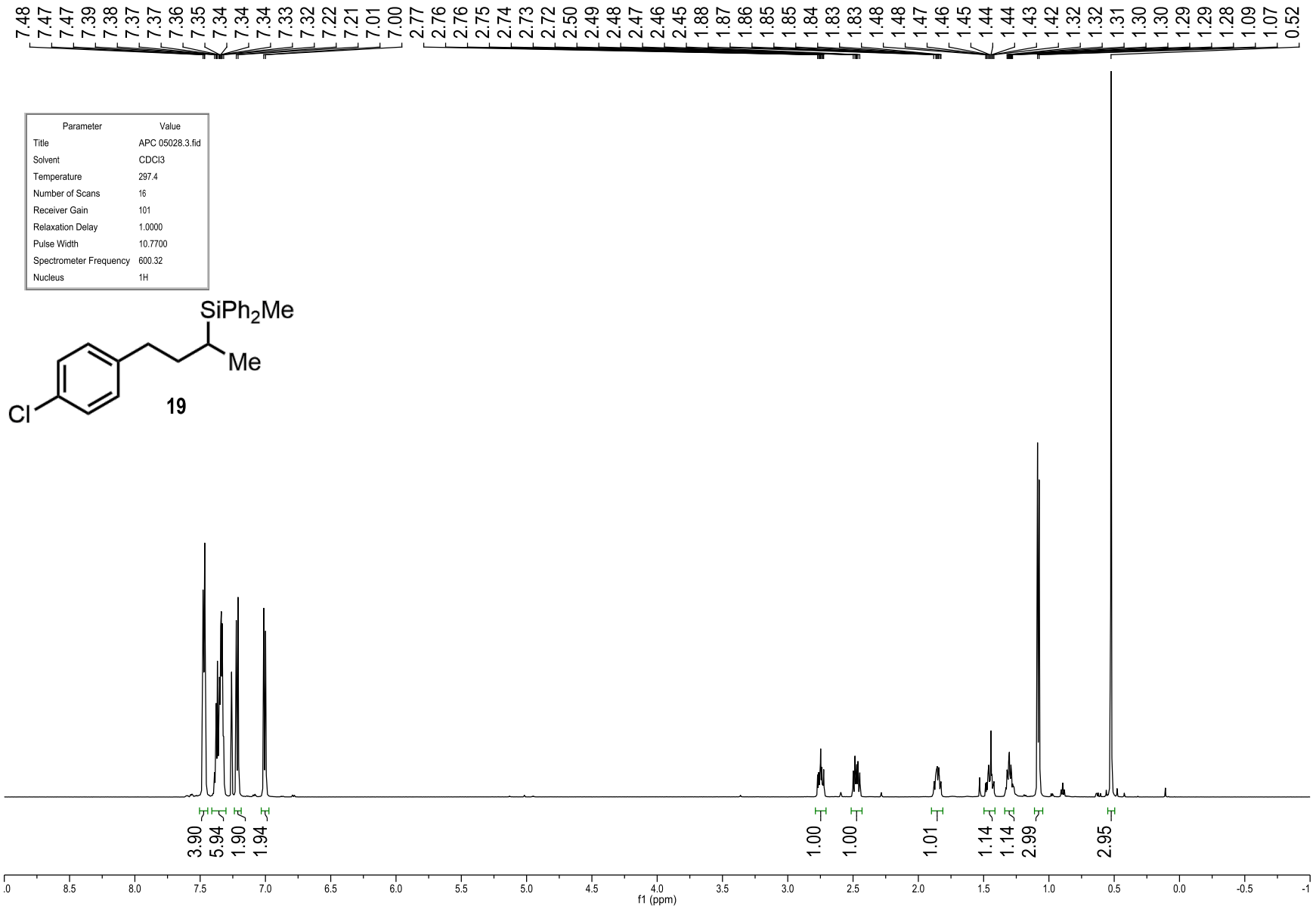
5.24

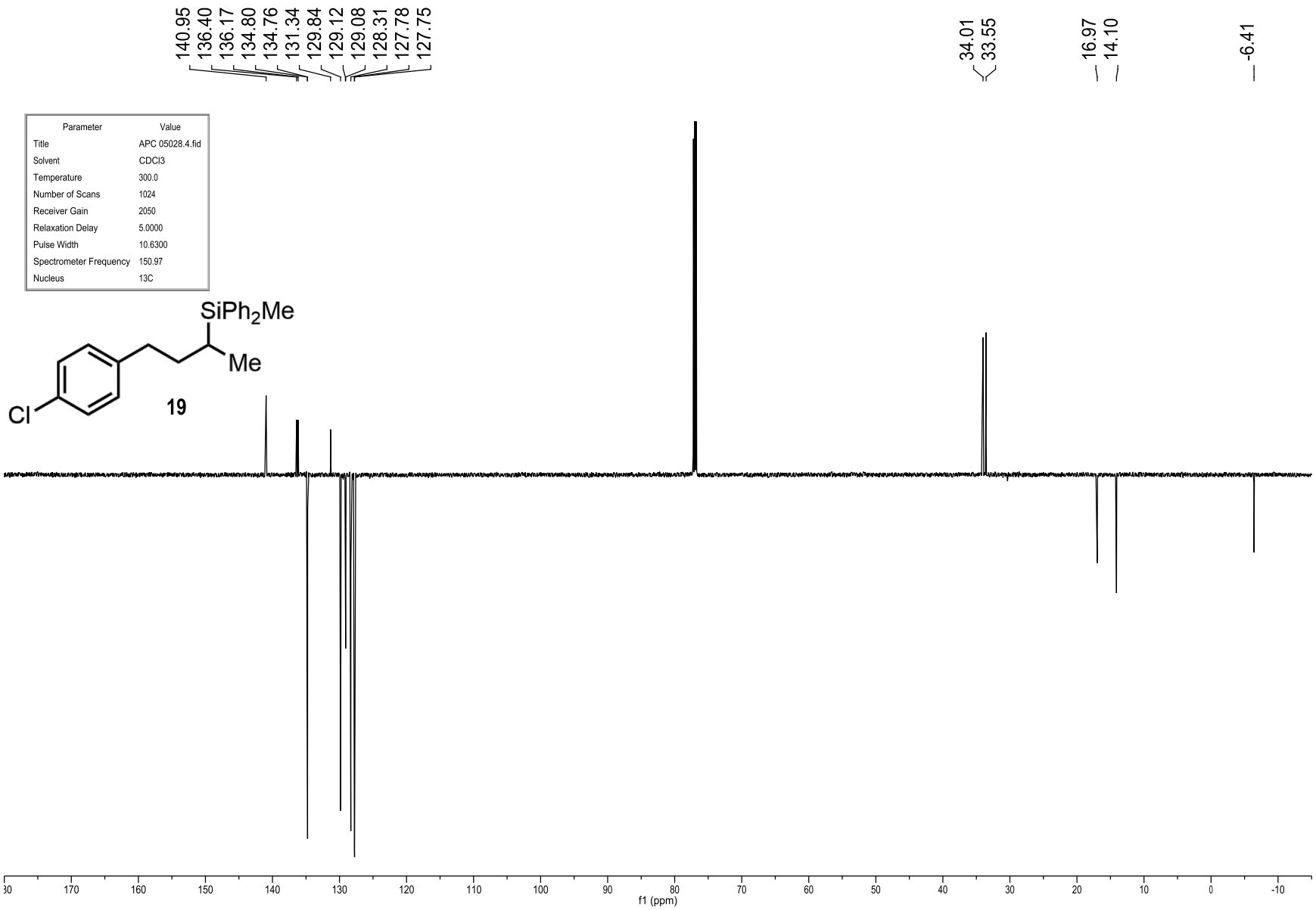
Parameter	Value
Title	APC 05027.3.fid
Solvent	CDCl3
Temperature	297.5
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si



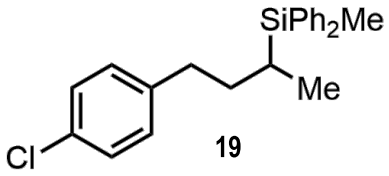
18




7.48 7.47 7.47 7.39 7.38 7.37 7.37 7.36 7.35 7.34 7.34 7.34 7.33 7.32 7.22 7.21 7.01 7.00
2.77 2.76 2.76 2.75 2.74 2.73 2.72 2.50 2.49 2.48 2.47 2.46 2.45 1.88 1.87 1.86 1.85 1.85 1.84 1.83 1.83 1.48 1.48 1.47 1.46 1.45 1.44 1.44 1.43 1.42 1.32 1.32 1.31 1.30 1.30 1.29 1.29 1.28 1.09 1.07 0.52
Parameter Value
Title APC 05028.3.fid
Solvent CDCl3
Temperature 297.4
Number of Scans 16
Receiver Gain 101
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiPh2Me
Me
Cl
19
3.90 5.94 1.90 1.94
1.00 1.00 1.01 1.14 1.14 2.99 2.95
f1 (ppm)

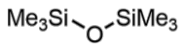

140.95
136.40
136.17
134.80
134.76
131.34
129.84
129.12
129.08
128.31
127.78
127.75
34.01
33.55
16.97
14.10
-6.41
Parameter Value
Title APC 05028.4.fid
Solvent CDCl3
Temperature 300.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 10.6300
Spectrometer Frequency 150.97
Nucleus 13C
SiPh2Me
Me
Cl
19
f1 (ppm)

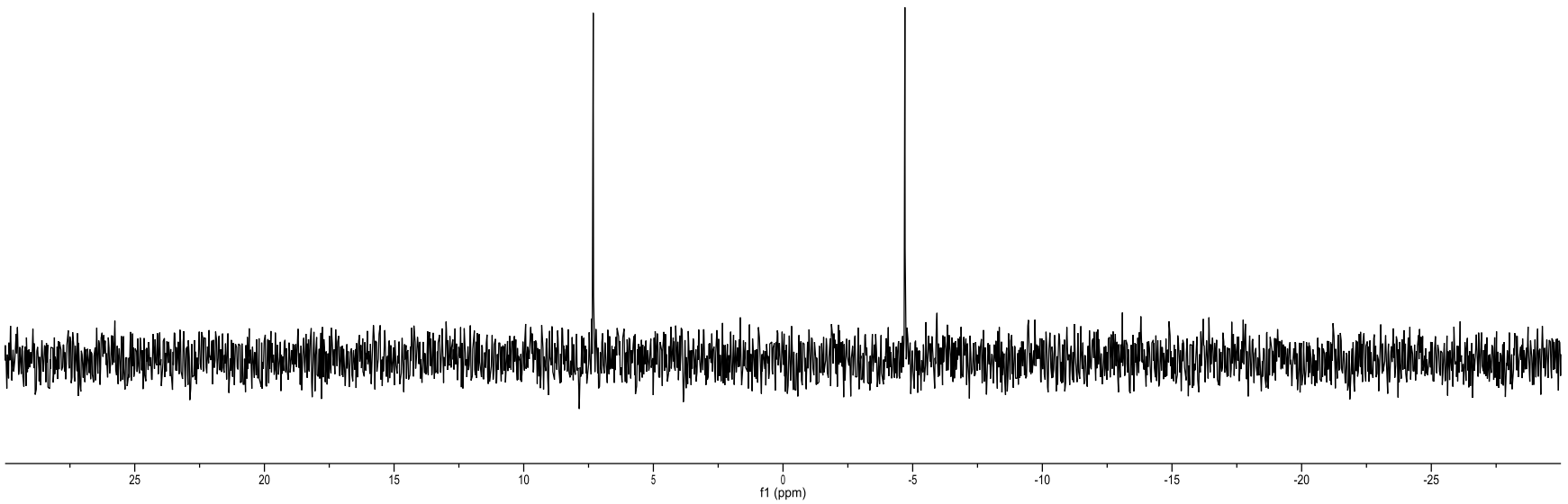
Parameter	Value
Title	APC 05028.5.fid
Solvent	CDCl3
Temperature	297.3
Number of Scans	128
Receiver Gain	1030
Relaxation Delay	12.5000
Pulse Width	13.5000
Spectrometer Frequency	119.26
Nucleus	29Si

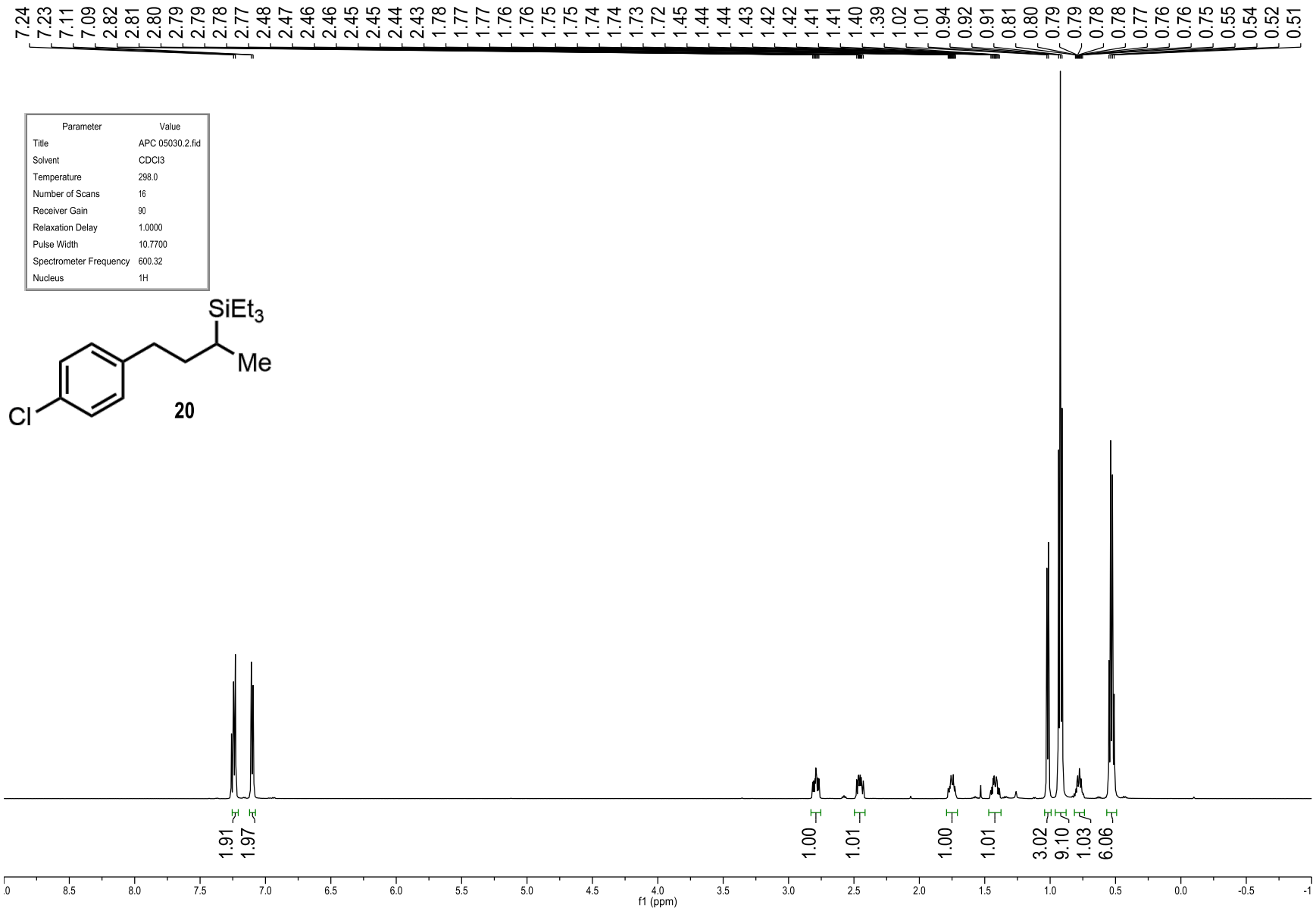


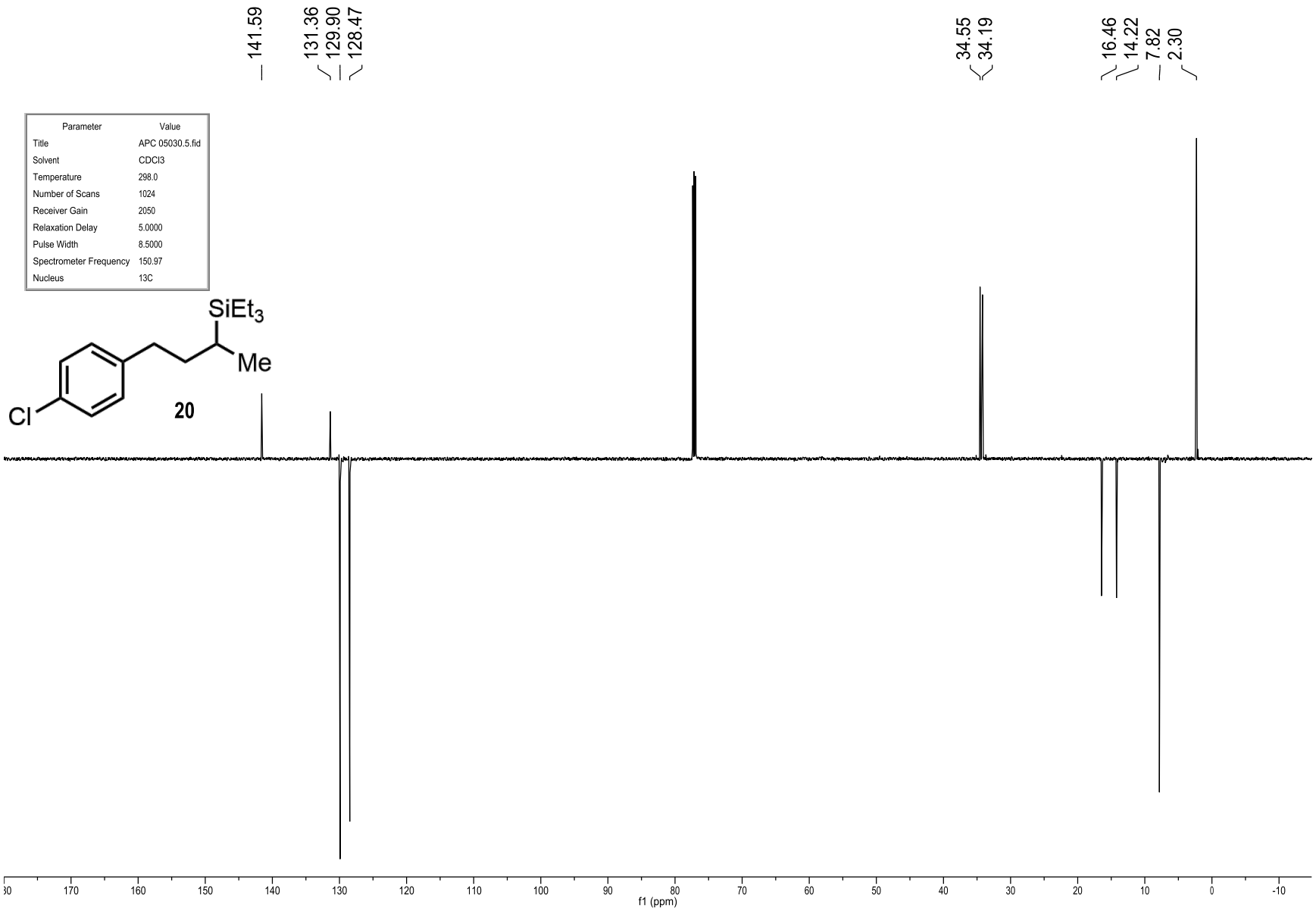
19

-4.70



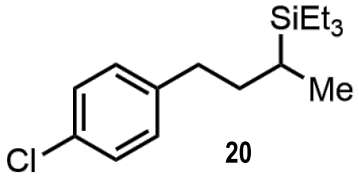



7.24 7.23 7.11 7.09 2.82 2.81 2.80 2.79 2.79 2.78 2.77 2.48 2.47 2.46 2.46 2.45 2.45 2.44 2.43 1.78 1.77 1.77 1.76 1.76 1.75 1.75 1.74 1.74 1.73 1.72 1.45 1.44 1.44 1.43 1.42 1.42 1.41 1.41 1.40 1.39 1.02 1.01 0.94 0.92 0.91 0.81 0.80 0.79 0.79 0.78 0.78 0.77 0.76 0.76 0.75 0.55 0.54 0.52 0.51
Parameter Value
Title APC 05030.2.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 16
Receiver Gain 90
Relaxation Delay 1.0000
Pulse Width 10.7700
Spectrometer Frequency 600.32
Nucleus 1H
SiEt3
Me
Cl
20
1.91 1.97 1.00 1.01 1.00 1.01 3.02 9.10 1.03 6.06
f1 (ppm)

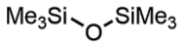

141.59
131.36
129.90
128.47
34.55
34.19
16.46
14.22
7.82
2.30
Parameter Value
Title APC 05030.5.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
SiEt3
Me
Cl
20
f1 (ppm)

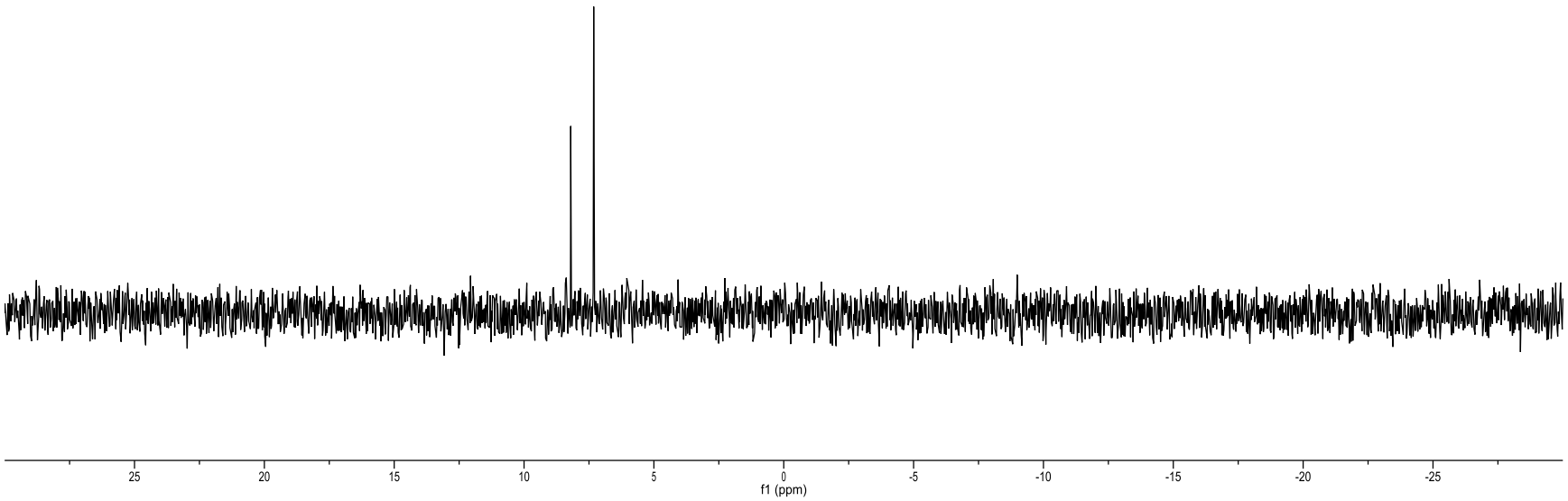
8.20
7.32

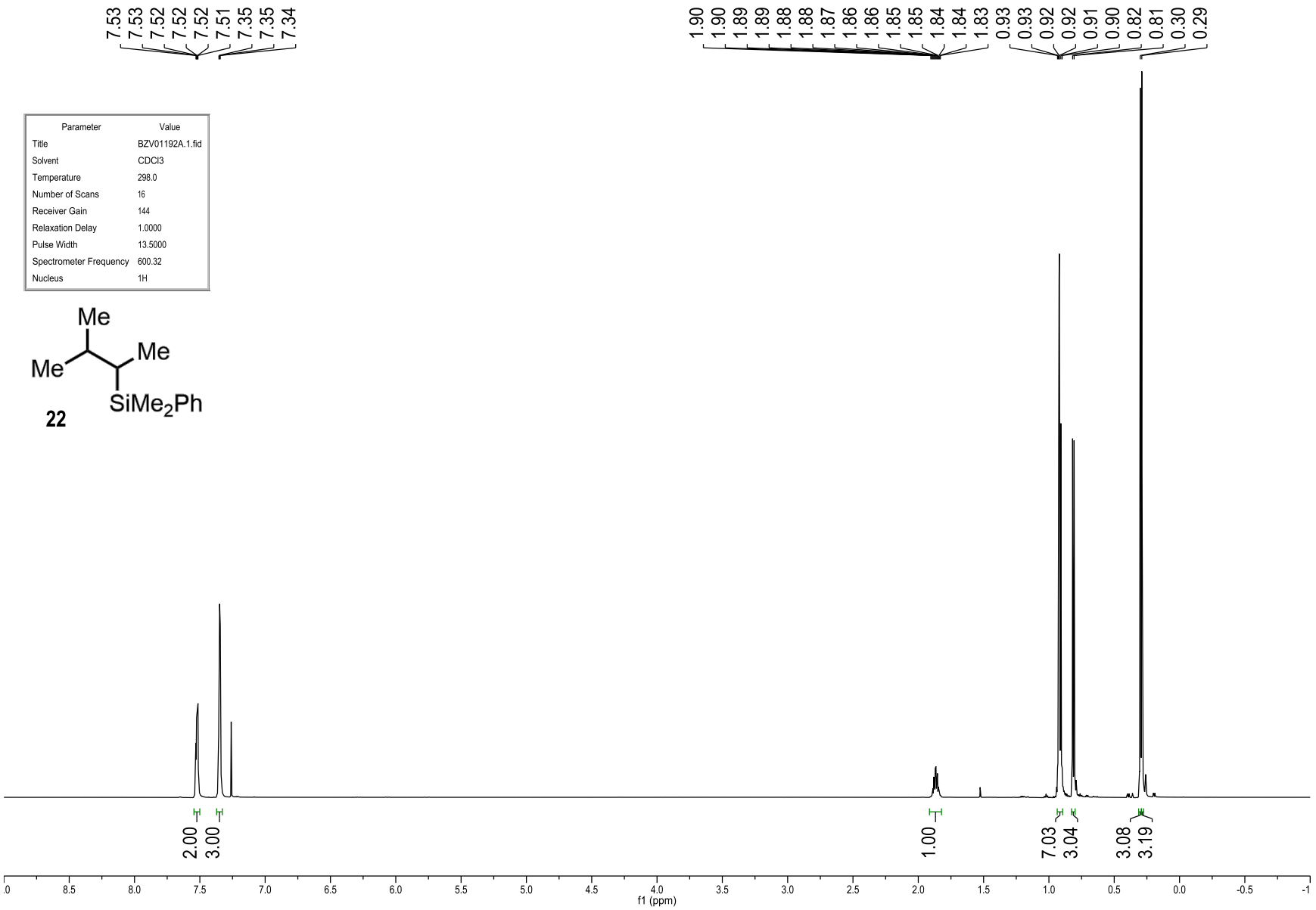
Parameter	Value
Title	APC 05030.4.fid
Solvent	CDCl3
Temperature	298.0
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si

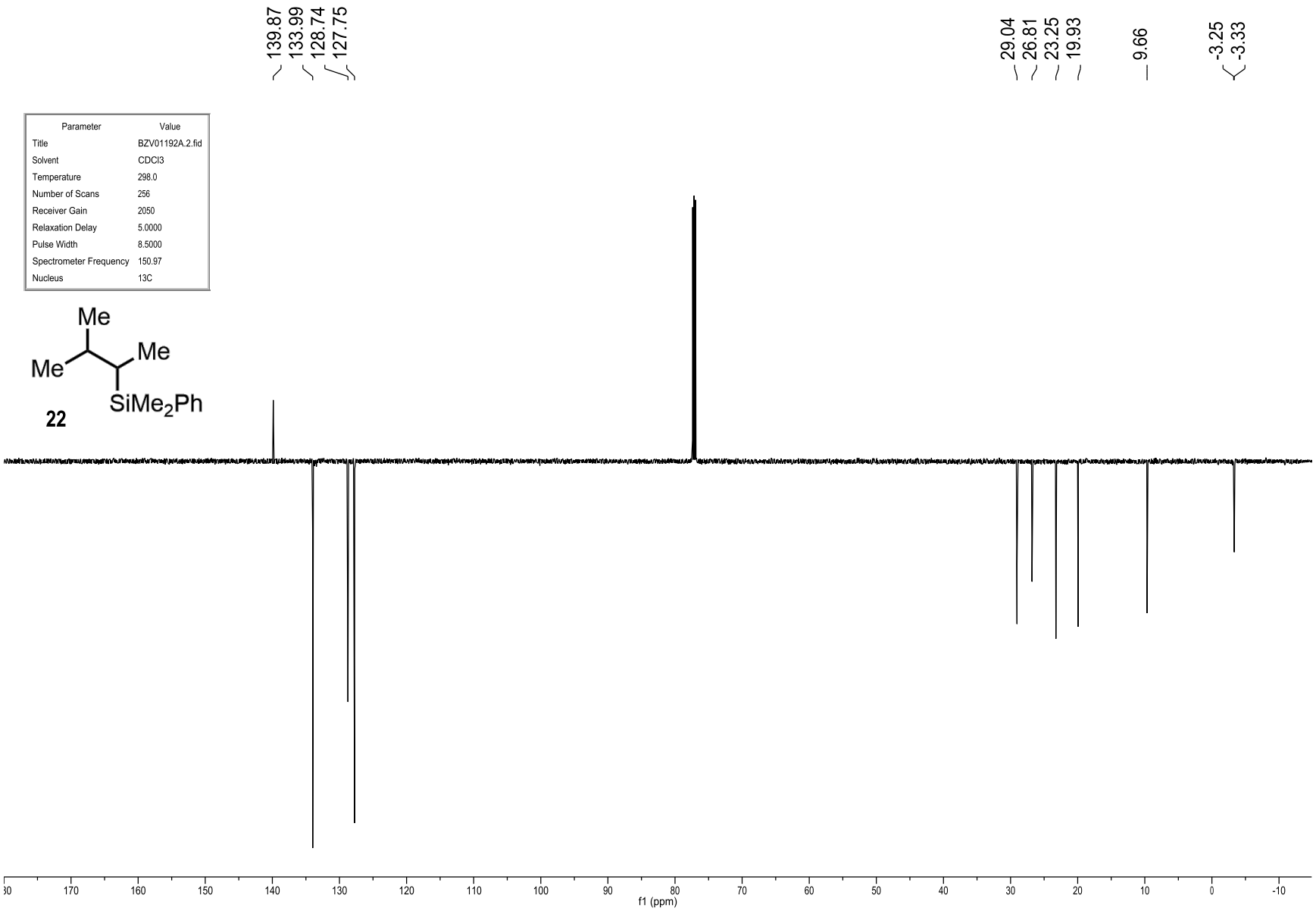


20

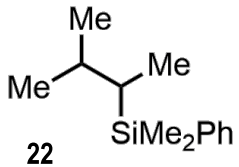




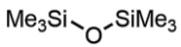

7.53
7.53
7.52
7.52
7.52
7.51
7.35
7.35
7.34
1.90
1.90
1.89
1.89
1.88
1.88
1.87
1.86
1.86
1.85
1.85
1.84
1.84
1.83
0.93
0.93
0.92
0.92
0.91
0.90
0.82
0.81
0.30
0.29
Parameter
Value
Title
BZV01192A.1.fid
Solvent
CDCl3
Temperature
298.0
Number of Scans
16
Receiver Gain
144
Relaxation Delay
1.0000
Pulse Width
13.5000
Spectrometer Frequency
600.32
Nucleus
1H
Me
Me
Me
SiMe2Ph
22
2.00
3.00
1.00
7.03
3.04
3.08
3.19
f1 (ppm)

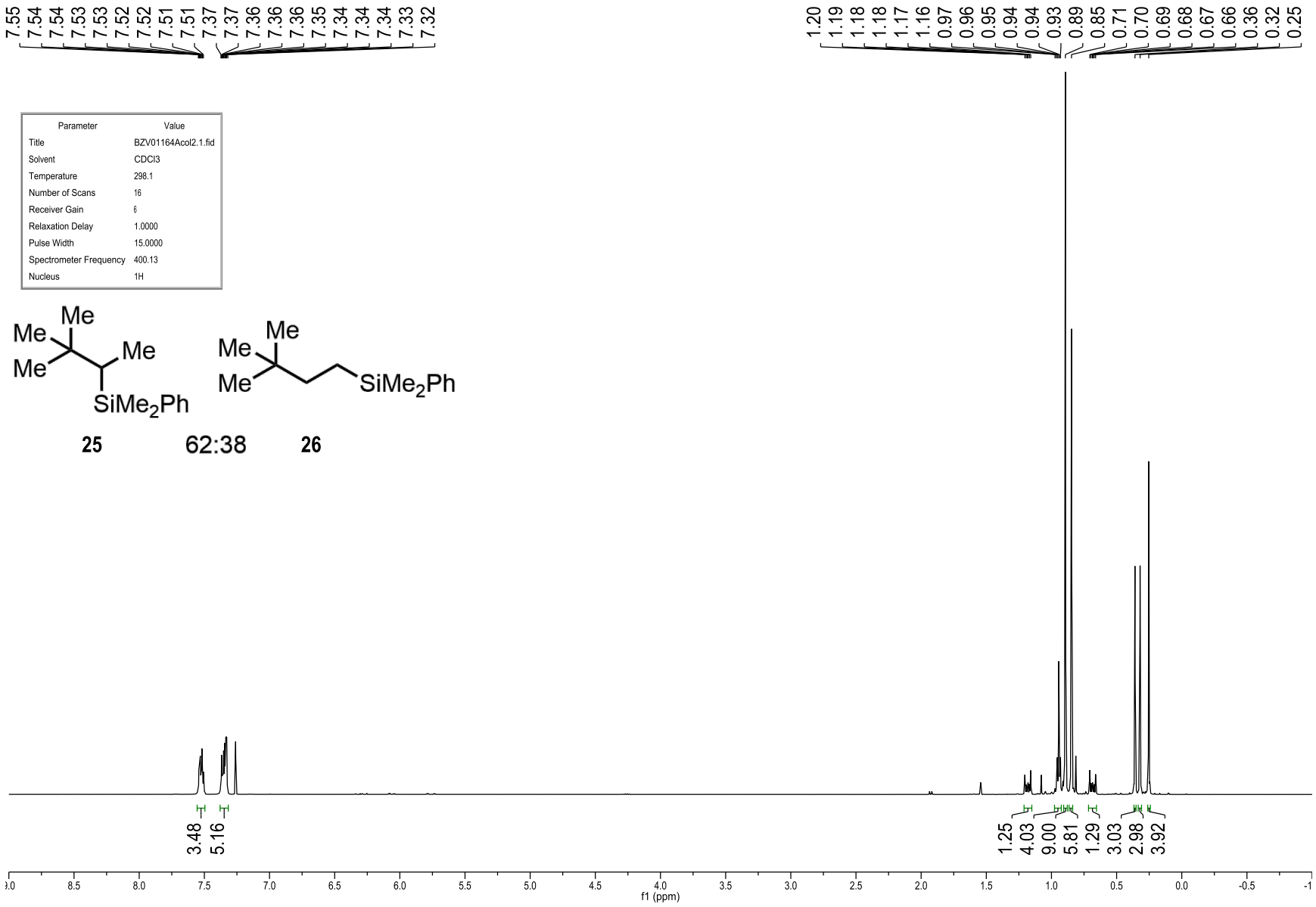

139.87
133.99
128.74
127.75
29.04
26.81
23.25
19.93
9.66
-3.25
-3.33
Parameter Value
Title BZV01192A.2.fid
Solvent CDCl3
Temperature 298.0
Number of Scans 256
Receiver Gain 2050
Relaxation Delay 5.0000
Pulse Width 8.5000
Spectrometer Frequency 150.97
Nucleus 13C
Me
Me
Me
SiMe2Ph
22
f1 (ppm)

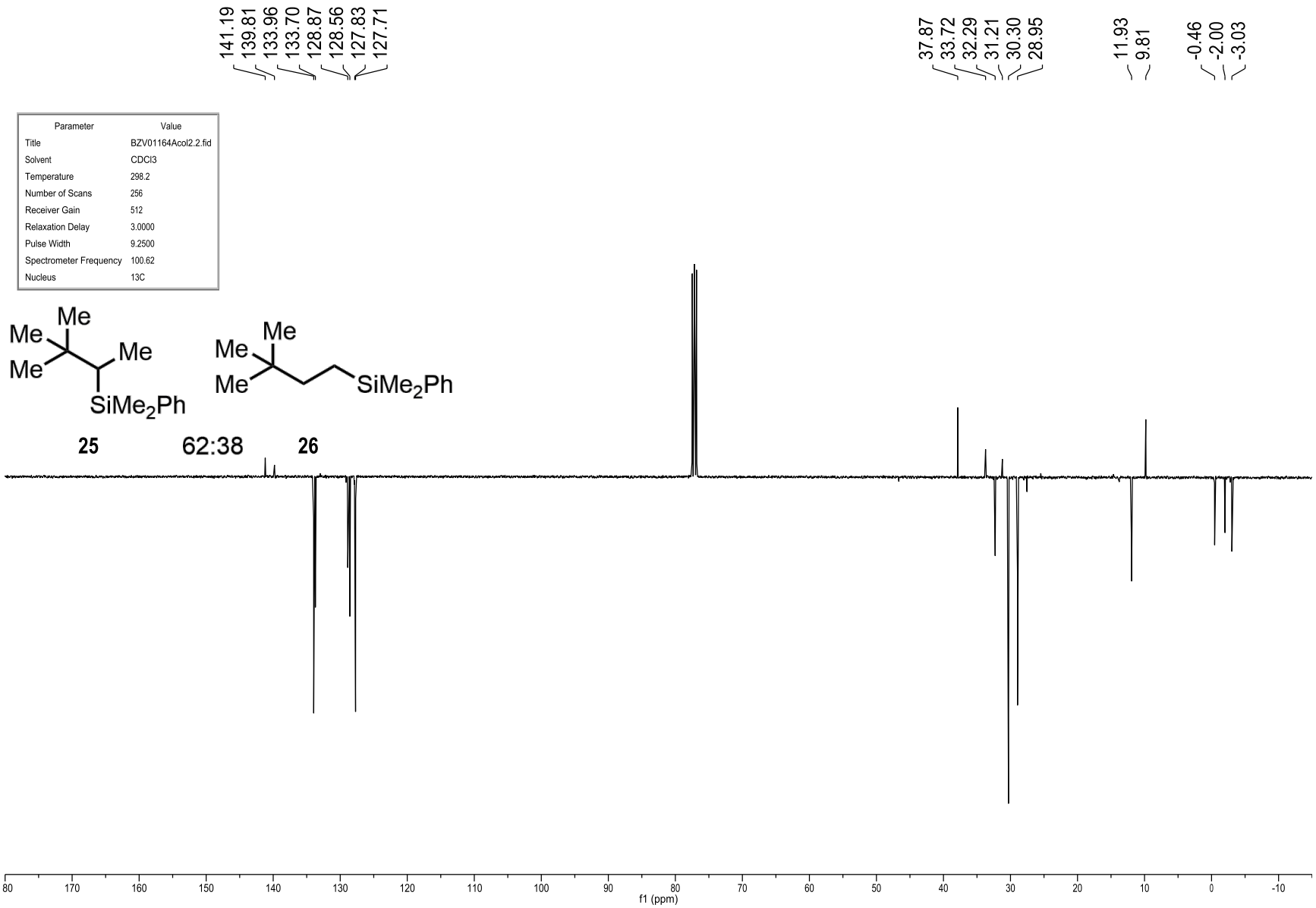
Parameter	Value
Title	BZV01192ASi.3.fid
Solvent	CDCl3
Temperature	298.0
Number of Scans	128
Receiver Gain	2050
Relaxation Delay	12.5000
Pulse Width	11.0000
Spectrometer Frequency	119.26
Nucleus	29Si

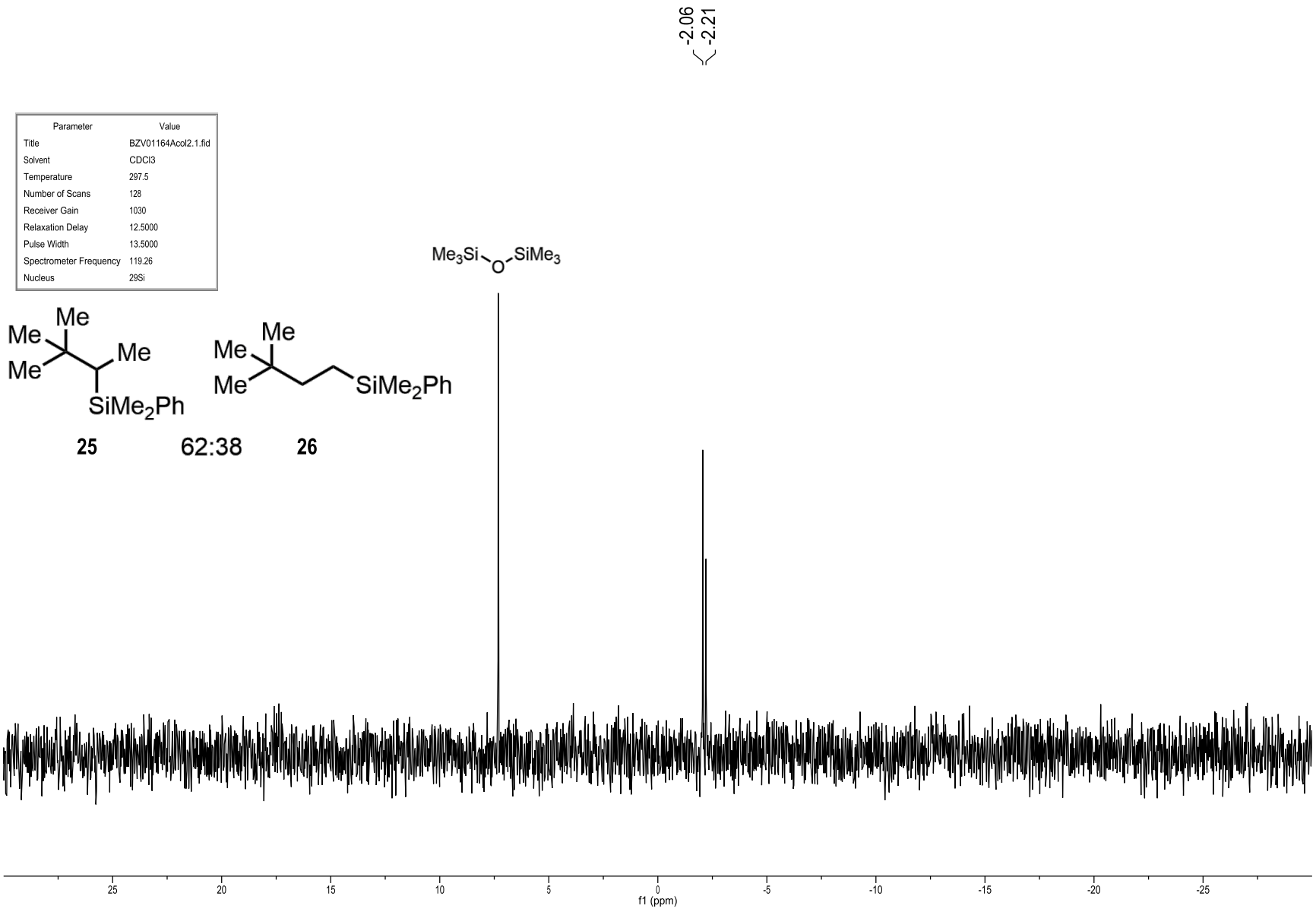


22

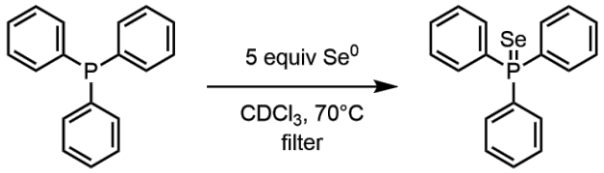



7.55 7.54 7.54 7.53 7.53 7.52 7.52 7.51 7.51 7.37 7.37 7.36 7.36 7.36 7.35 7.34 7.34 7.34 7.33 7.32
1.20 1.19 1.18 1.18 1.17 1.16 0.97 0.96 0.95 0.94 0.94 0.93 0.89 0.85 0.71 0.70 0.69 0.68 0.67 0.66 0.36 0.32 0.25
Parameter Value
Title BZV01164Acol2.1.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 16
Receiver Gain 6
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me Me Me SiMe2Ph
Me Me SiMe2Ph
25 62:38 26
3.48 5.16
1.25 4.03 9.00 5.81 1.29 3.03 2.98 3.92
f1 (ppm)


141.19
139.81
133.96
133.70
128.87
128.56
127.83
127.71
37.87
33.72
32.29
31.21
30.30
28.95
11.93
9.81
-0.46
-2.00
-3.03
Parameter Value
Title BZV01164Acol2.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 256
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
Me
Me
SiMe2Ph
Me
Me
Me
SiMe2Ph
25
62:38
26
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

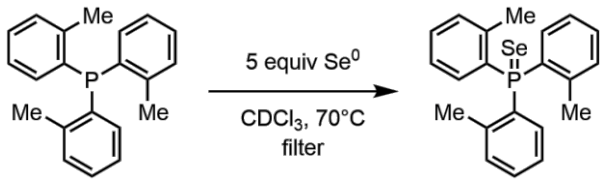

-2.06
-2.21
Parameter Value
Title BZV01164Acol2.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 128
Receiver Gain 1030
Relaxation Delay 12.5000
Pulse Width 13.5000
Spectrometer Frequency 119.26
Nucleus 29Si
Me3Si O SiMe3
Me
Me
Me
SiMe2Ph
25
62:38
Me
Me
Me
SiMe2Ph
26
25
20
15
10
5
0
-5
-10
-15
-20
-25
f1 (ppm)

Parameter	Value
Title	APC 05023 A.1.fid
Solvent	CDCl3
Temperature	297.6
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P

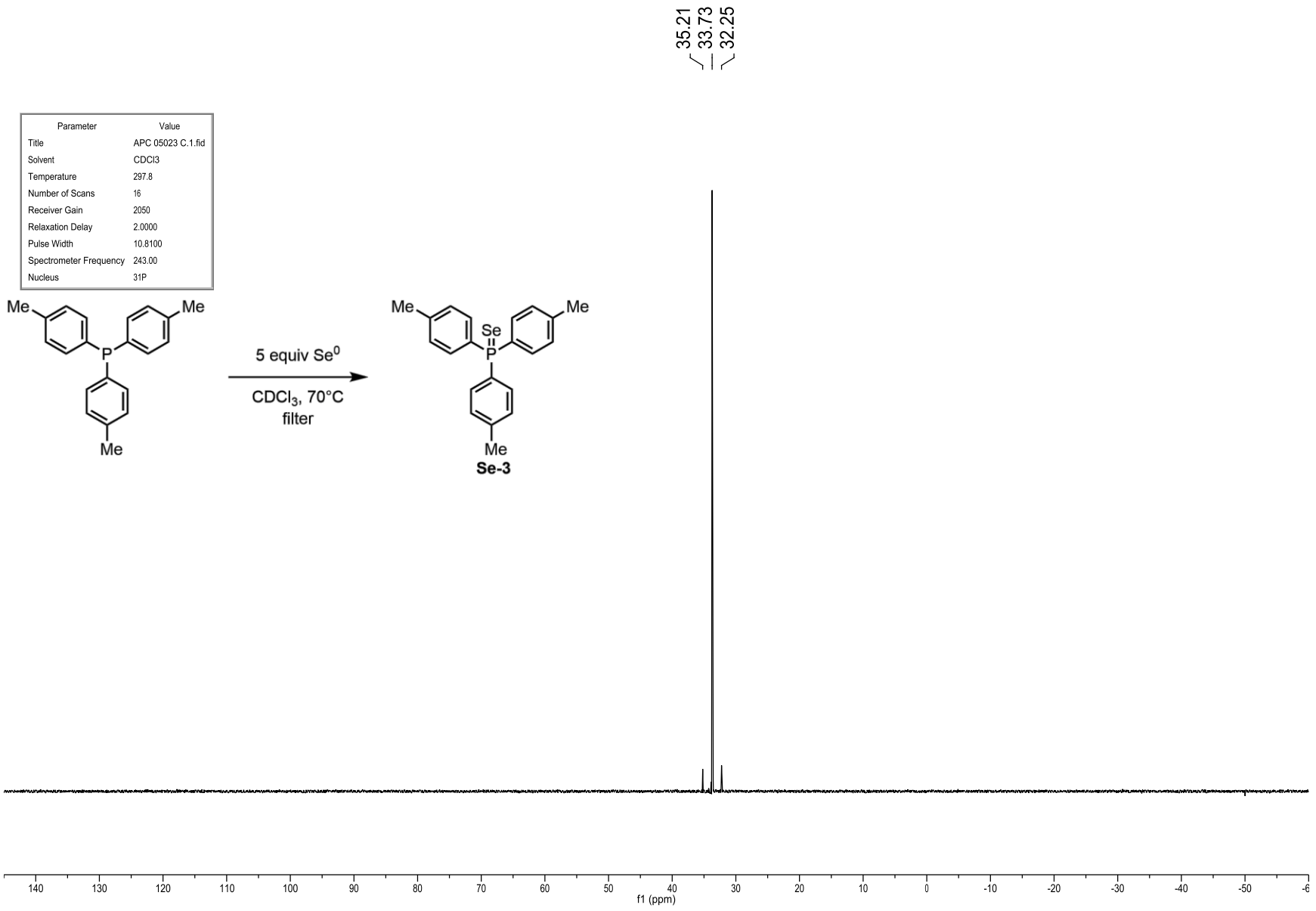


Se-1

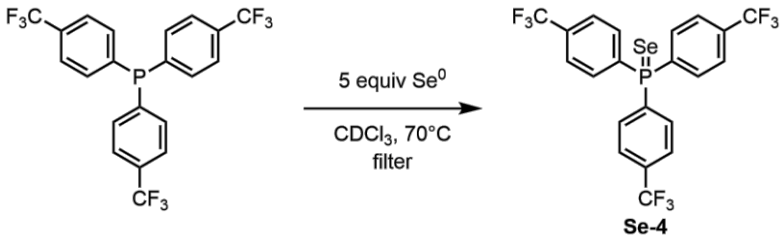
Parameter	Value
Title	APC 05023 B.1.fid
Solvent	CDCl3
Temperature	297.7
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P



Se-2

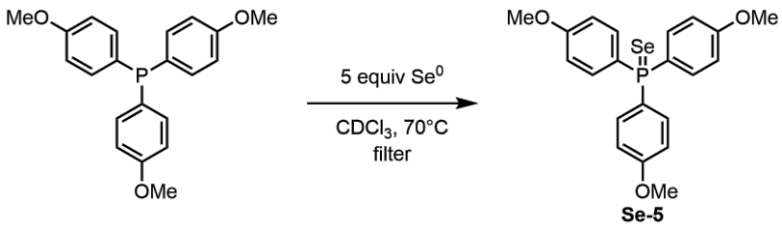

35.21
33.73
32.25
Parameter Value
Title APC 05023 C.1.fid
Solvent CDCl3
Temperature 297.8
Number of Scans 16
Receiver Gain 2050
Relaxation Delay 2.0000
Pulse Width 10.8100
Spectrometer Frequency 243.00
Nucleus 31P
Me
Me
P
Me
5 equiv Se0
CDCl3, 70°C
filter
Me
Me
Se
P
Me
Se-3
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

Parameter	Value
Title	APC 05023 D.1.fid
Solvent	CDCl3
Temperature	297.8
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P



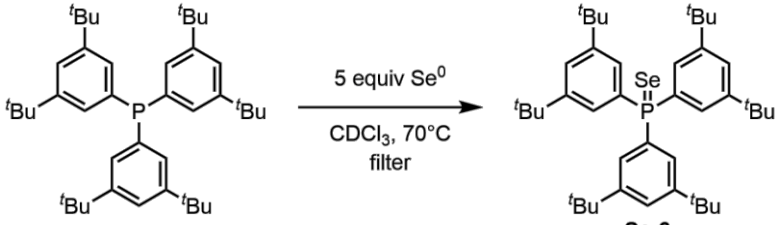
Se-4

Parameter	Value
Title	APC 05023 E.1.fid
Solvent	CDCl3
Temperature	297.8
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P



Se-5

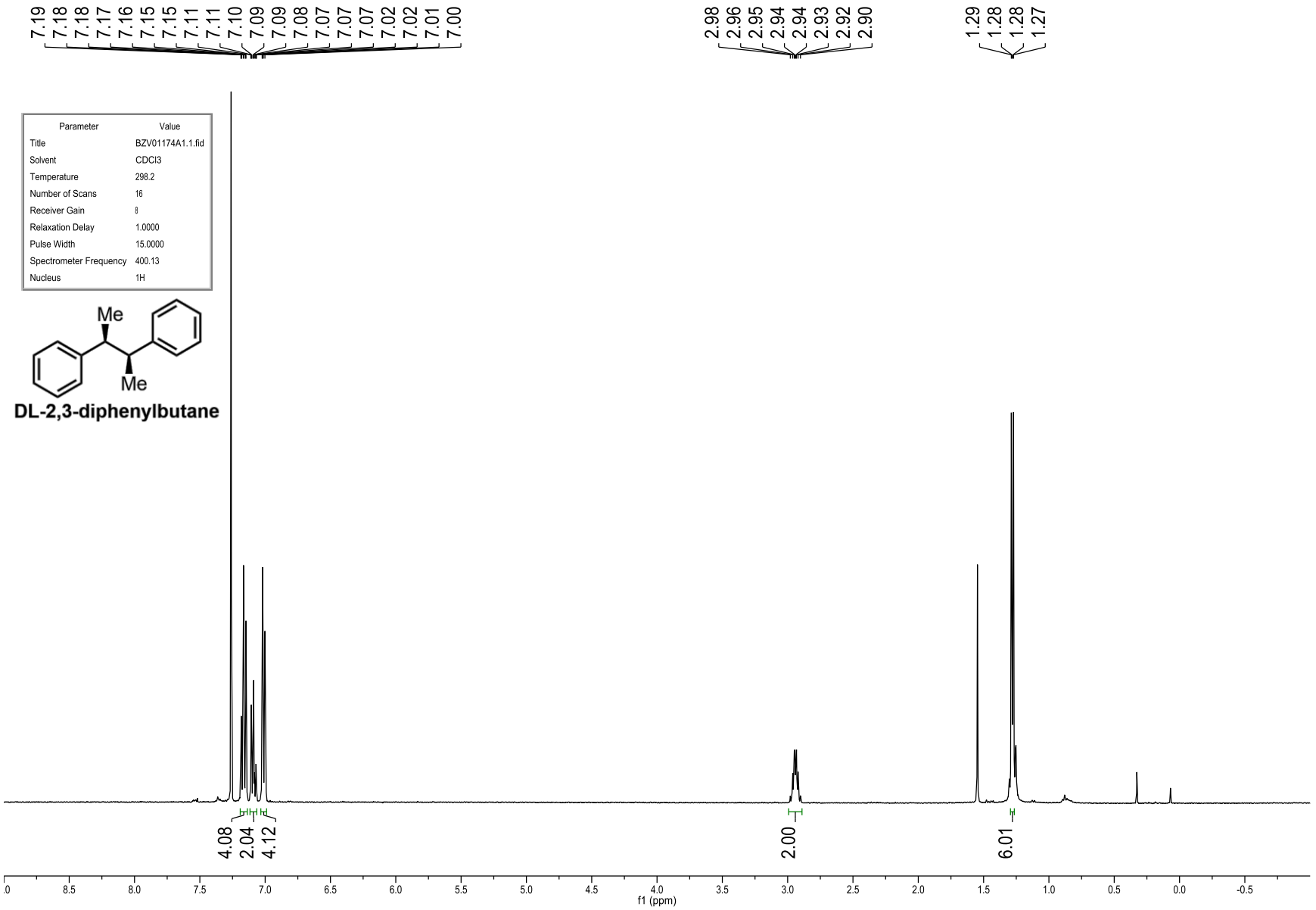
Parameter	Value
Title	APC 05023 F.1.fid
Solvent	CDCl3
Temperature	297.8
Number of Scans	16
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.8100
Spectrometer Frequency	243.00
Nucleus	31P

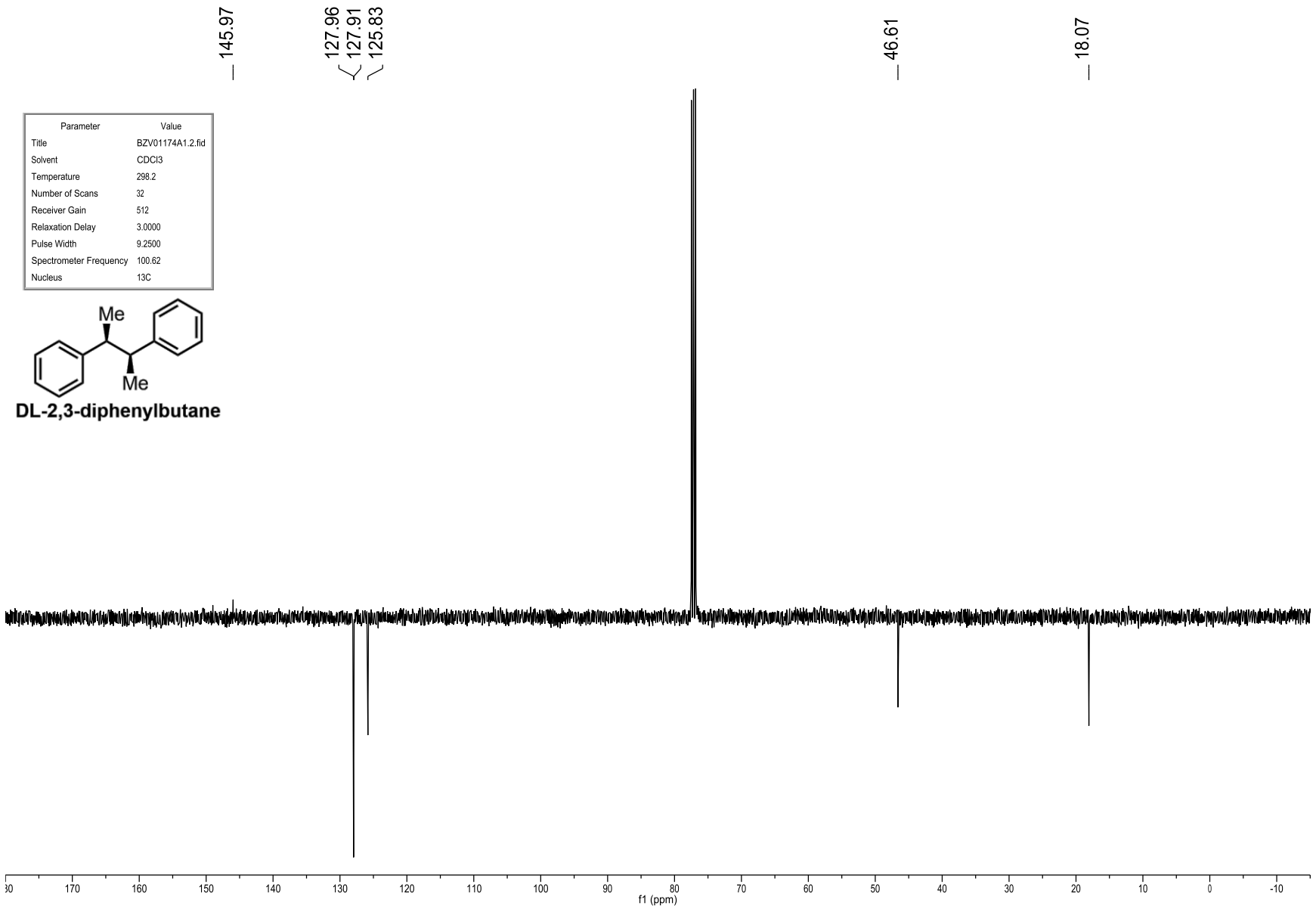


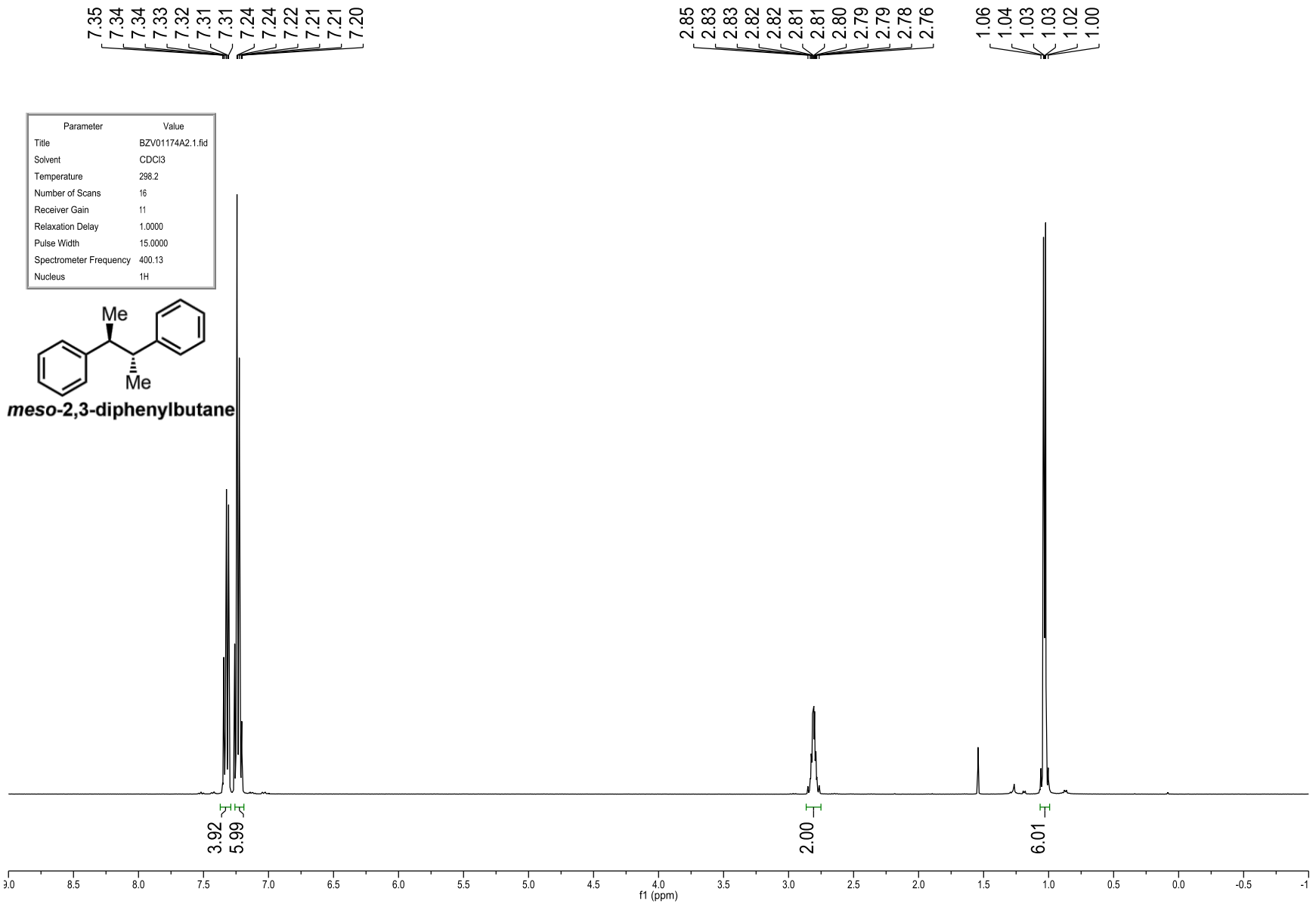
5 equiv Se^0

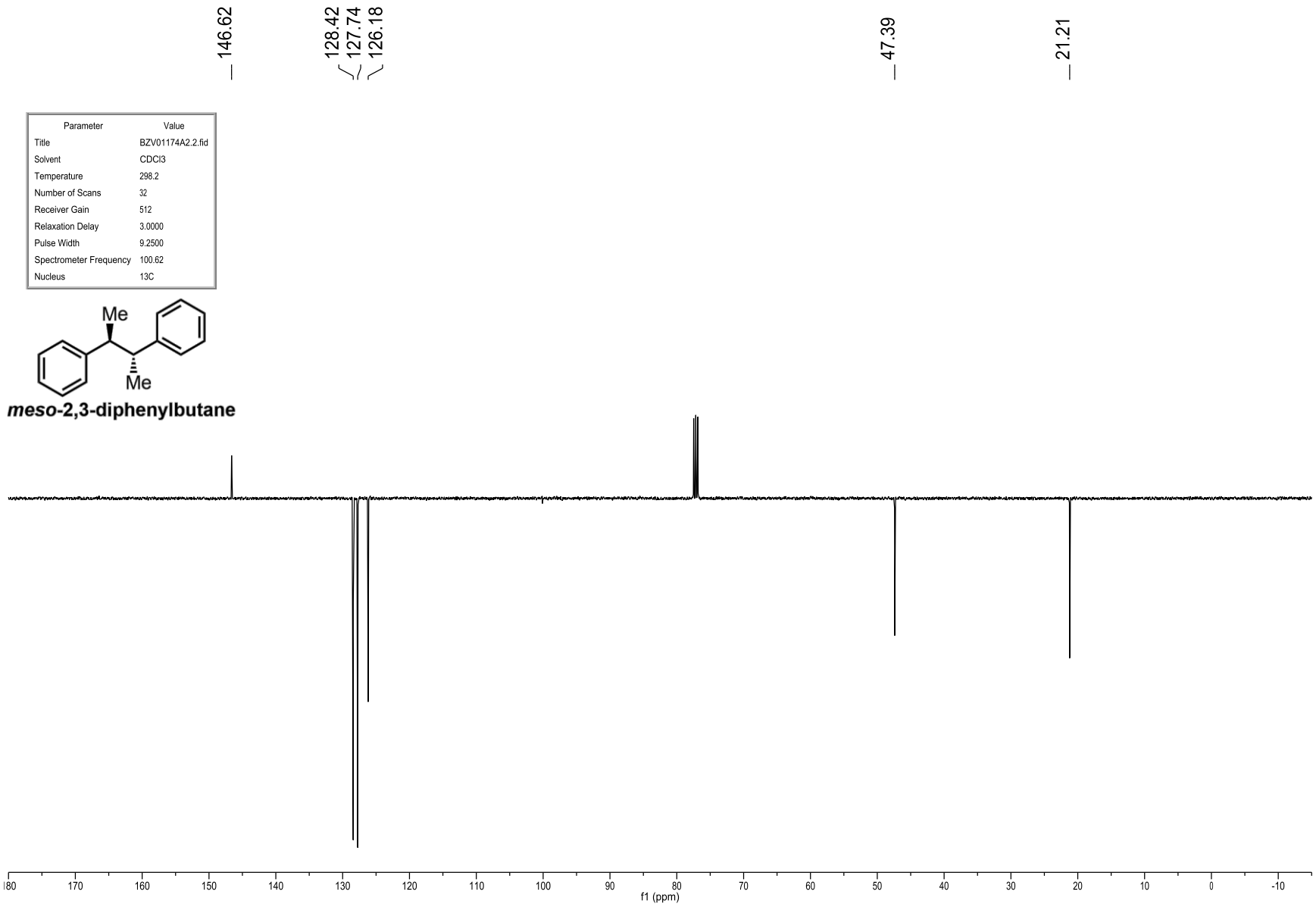
$CDCl_3$, 70°C
filter

Se-6


7.19
7.18
7.18
7.17
7.16
7.15
7.15
7.11
7.11
7.10
7.09
7.09
7.08
7.07
7.07
7.07
7.02
7.02
7.01
7.00
2.98
2.96
2.95
2.94
2.94
2.93
2.92
2.90
1.29
1.28
1.28
1.27
Parameter Value
Title BZV01174A1.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 8
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
DL-2,3-diphenylbutane
4.08
2.04
4.12
2.00
6.01
f1 (ppm)


145.97
127.96
127.91
125.83
46.61
18.07
Parameter Value
Title BZV01174A1.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 32
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
DL-2,3-diphenylbutane
f1 (ppm)


7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.24
7.24
7.22
7.21
7.21
7.20
2.85
2.83
2.83
2.82
2.82
2.81
2.81
2.80
2.79
2.79
2.78
2.76
1.06
1.04
1.03
1.03
1.02
1.00
Parameter Value
Title BZV01174A2.1.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 16
Receiver Gain 11
Relaxation Delay 1.0000
Pulse Width 15.0000
Spectrometer Frequency 400.13
Nucleus 1H
Me
Me
meso-2,3-diphenylbutane
3.92
5.99
2.00
6.01
f1 (ppm)


146.62
128.42
127.74
126.18
47.39
21.21
Parameter Value
Title BZV01174A2.2.fid
Solvent CDCl3
Temperature 298.2
Number of Scans 32
Receiver Gain 512
Relaxation Delay 3.0000
Pulse Width 9.2500
Spectrometer Frequency 100.62
Nucleus 13C
Me
Me
meso-2,3-diphenylbutane
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)