An Electrochemical Strategy to Synthesize Disilanes and Oligosilanes from Chlorosilanes

Weiyang Guan[1], Lingxiang Lu[1], Qifeng Jiang[2], Alexandra F. Gittens[2], Yi Wang[1], Luiz F. T. Novaes[1], Rebekka S. Klausen[2]*, Song Lin[1]*

[1]Department of Chemistry and Chemical Biology, Cornell University, Ithaca, NY 14853, USA
[2]Department of Chemistry, Johns Hopkins University, Baltimore, MD 21218, USA

*Correspondence to: klausen@jhu.edu; songlin@cornell.edu

Supporting Information

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1. General information

All electrochemical reactions were performed using an IKA Electrasyn vial unless otherwise noted. The threads on the vial were wrapped with PTFE tape to ensure sufficient sealing. All reactions were conducted under a nitrogen atmosphere unless otherwise noted. Flash chromatography was performed using silica gel P60 from SiliCycle. Commercial reagents were purchased from Sigma Aldrich, Alfa Aesar, Acros, TCI, AK Scientific, Combi-Blocks and Oakwood and used as received. Tetrahydrofuran (THF) was distilled over sodium metal and benzophenone and stored in the glove box. Dimethoxyethane (DME) and acetonitrile (MeCN) were dried with 3~4 Å molecular sieves and stored in the glove box.

Nuclear magnetic resonance (NMR) spectroscopy

All proton NMR spectra were recorded on either a Varian-mercury 300 (300 MHz), Varian-Mercury 400 (400 MHz), Inova 500 (500 MHz) or Inova 600 (600 MHz) spectrometers at 20 °C. Chemical shifts for proton are reported in parts per million and are reference to residual protium in the NMR solvent according to values reported in literature: $\delta(CDCl_3)$ = 7.26 ppm, $\delta(CD_3CN)$ = 2.33 ppm. Carbon ($^{13}C\{^1H\}$ NMR) was referenced to the carbon resonances of the solvent according to values reported in literature: $\delta(CDCl_3)$ = 77.16 ppm.

Mass spectrometry

The high-resolution mass spectral (HRMS) data were obtained on Thermo Fisher Scientific GC-Exactive Spectrometer using EI-Orbitrap as the method. The mass spectral (GC-MS) data were obtained on Agilent 8860 GC System with an Agilent 5977B Mass Selective Detector. Input temperature were set between 70 °C to 350 °C depending on the molecule. Methods used for the GC-MS measurements: **Method-LT**: temperature starts at 70 °C (for 0.5 min) and ends at 310 °C (for 4 min) with heating increase gradient: 30 °C/min; **Method-HT**: temperature starts at 70 °C (for 0.5 min) and ends at 325 °C (for 1 min) with heating increase gradient: 20 °C/min; **Method-Slow**: temperature starts at 70 °C (for 0.5 min) and ends at 310 °C (for 4 min) with heating increase gradient: 15 °C/min.

Gas chromatography (GC)

All GC analyses were performed on a Shimadzu Nexus GC-2030 gas chromatograph with an FID detector or an Agilent 6890A gas chromatograph with a MS detector.

Ultraviolet-visible spectroscopy (UV-Vis)

All UV-Vis absorption spectra were collected using a Shimadzu UV-2600i UV-Vis spectrophotometer equipped with an ISR-2600 Plus Integrating Sphere.

Electrolysis methods and preparation

Electrolysis experiments were performed in a 5-mL, 10-mL or 20-mL Electrasyn vial on an IKA Electrasyn 2.0. The magnetic stir bars and ElectraSyn vials were oven dried overnight prior to use. Mg electrodes were cut into 5.25 x 0.8 x 0.2 cm^3 plates from a Mg plate (99.95% Mg) and polished with a razor to remove the impurities on the surface before use. Graphite electrodes (5.25 x 0.8 x 0.2 cm^3) were supplied by IKA and could be reused. Molybdenum electrodes were cut from a Molybdenum plate (99.96% Mo) into 5.25 x 0.8 x 0.1 cm^3 and could be reused. To clean the graphite

electrodes after use: after reaction, the graphite was sequentially washed with 1 M HCl, water, and acetone, then immersed in acetone and sonicated for 5 minutes. After that, the graphite was rinsed with acetone and polished with sand paper, and dried in an oven. To clean the molybdenum electrodes: after the reaction, the electrode was sequentially washed with 1 M HCl, water, and acetone, immersed in acetone and sonicated for 5 minutes. The electrode was air dried for several hours before use. If necessary, the electrode could be further polished with sandpaper.

Abbreviations

Ar = aryl, Bn = benzyl, ^{t}Bu = *tert*-butyl, DMF = *N*,*N*-dimethylformamide, EA = ethyl acetate, F = faraday, Hex = hexanes, ^{i}Pr = isopropyl, MeCN = acetonitrile, Ph = phenyl, TBA = tetrabutylammonium, TEA = triethylamine, TES = triethylsilyl, TFSI = trifluoromethanesulfonimide, THF = tetrahydrofuran, TMS = trimethylsilyl.

Safety when handling chemicals and reaction setup

$TBAClO_4$: $TBAClO_4$ is classified as an oxidizer that should be handled with care and avoid heat or ignition. MSDS is available on https://www.sigmaaldrich.com/catalog/product/aldrich/86885.

Reaction setup and workup: The reaction mixtures are prepared in a N_2 glovebox before being transferred out to a bench top for electrolysis. THF was distilled over sodium/benzophenone or dried with 3~4 Å molecular sieves (lower yield observed) before use. In silyl cross-electrophile coupling reactions, Mg^0 will precipitate on the graphite cathode after 2 F/mol of charge or at the stage when chlorosilanes are almost consumed. In general, Mg will coat the carbon electrode uniformly as a shiny layer and is normal sign of a successful reaction and the workup of the reaction can be handled safely using routine procedures. However, if the solvent is not dry or if HCl is formed during the reaction (eg. alcohol substrates which react with chlorosilane first to form Si-O and release HCl), Mg^0 will plate out as black micro sized solids, which is combustible when the electrode is exposed to air and needs to be handled with care. In this case, a good way to quench Mg^0 is adding around 1 mL of water before working up the reaction.

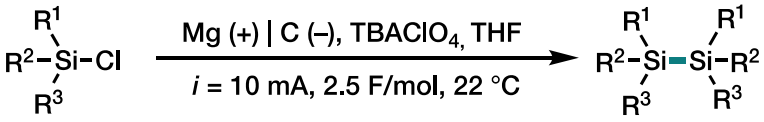
2. General procedures

2.1. Graphic guide for experimental setup

(a) **(b)** **(c)** **(d)**

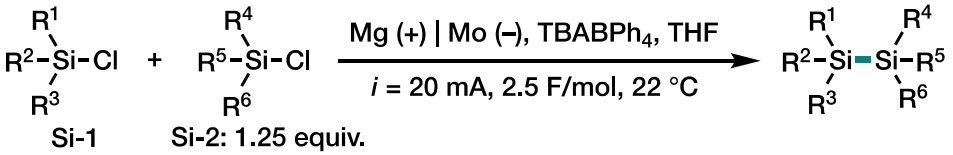
(a) Polished Mg anode and graphite cathode. (b) Assembled Electrasyn cap. The red cap on top can be further secured with parafilm if the sealing is not sufficient. (c) The threads on the vial were wrapped with PTFE tape to ensure sufficient sealing. (d) Electrolysis.

2.2. General procedure 1: disilane synthesis via homocoupling



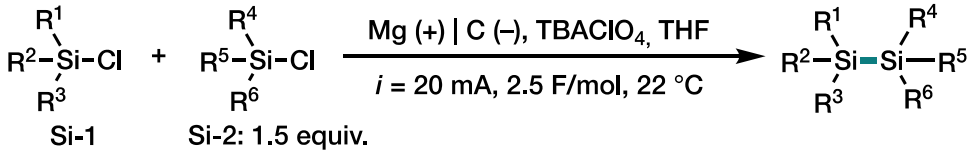
Magnesium electrodes were polished with a razor and graphite electrodes were polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Graphite electrodes were dried in oven for at least 10 min before use. In a glove box, $TBAClO_4$ (2 mmol, 2 equiv.) and chlorosilane (1 mmol, 1 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (graphite plate), and then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 10 mA until passing 2.5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

2.3. General procedure 2: disilane synthesis via hetero-coupling (for chlorosilanes WITH Si-H functionality)



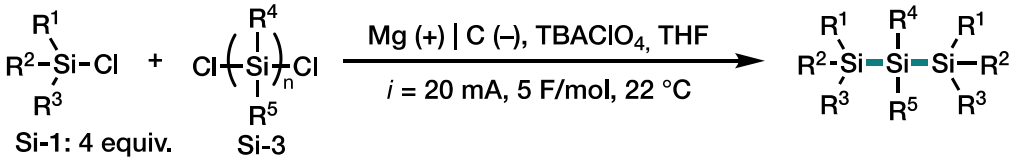
Magnesium electrode was polished with a razor until a shiny finish was obtained. Molybdenum electrode was used as received. In a glove box, $TBABPh_4$ (1 mmol, 1 equiv.), chlorosilane **Si-1** (1 mmol, 1 equiv.) and chlorosilane **Si-2** (1.25 mmol, 1.25 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (molybdenum plate), and then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 20 mA until passing 2.5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

2.4 General procedure 3: disilane synthesis via hetero-coupling (for chlorosilanes WITHOUT Si-H functionality)



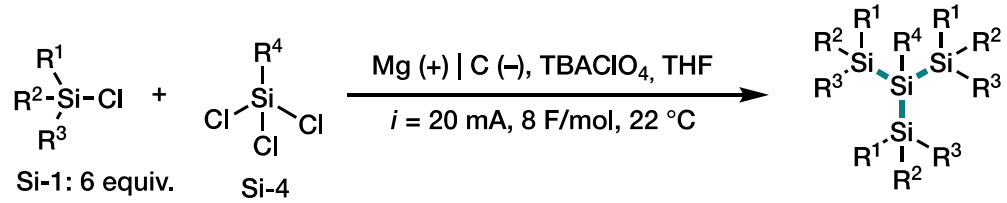
Magnesium electrodes were polished with a razor and graphite electrodes were polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Graphite electrodes were dried in oven for at least 10 min before use. In a glove box, $TBAClO_4$ (2 mmol, 2 equiv.), chlorosilane **Si-1** (1 mmol, 1 equiv.) and chlorosilane **Si-2** (1.5 mmol, 1.5 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (graphite plate), and then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 20 mA until passing 2.5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

2.5. General procedure 4: trisilane synthesis



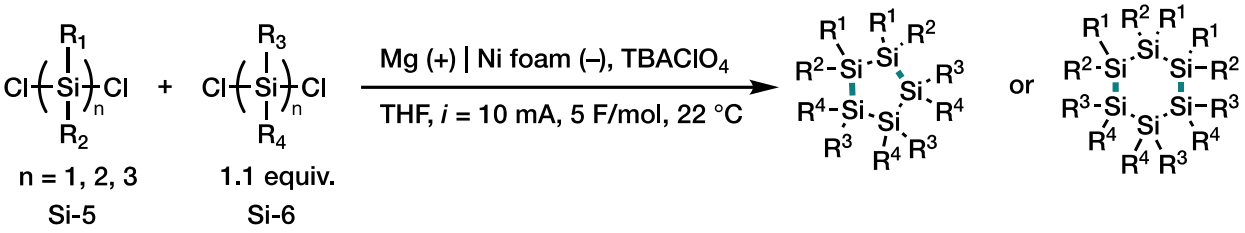
Magnesium electrodes were polished with a razor and graphite electrodes were polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Graphite electrodes were dried in oven for at least 10 min before use. In a glove box, $TBAClO_4$ (2 mmol, 2 equiv.), dichlorosilane **Si-3** (1 mmol, 1 equiv.) and mono-chlorosilane **Si-1** (4 mmol, 4 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (graphite plate), and then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 20 mA until passing 5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

2.6. General procedure 5: tetrasilane synthesis



Magnesium electrodes were polished with a razor and graphite electrodes were polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Graphite electrodes were dried in oven for at least 10min before use. In a glove box, $TBAClO_4$ (2 mmol, 2 equiv.), trichlorosilane **Si-4** (1 mmol, 1 equiv.) and mono-chlorosilane **Si-1** (6 mmol, 6 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (graphite plate), and then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 20 mA until passing 8 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

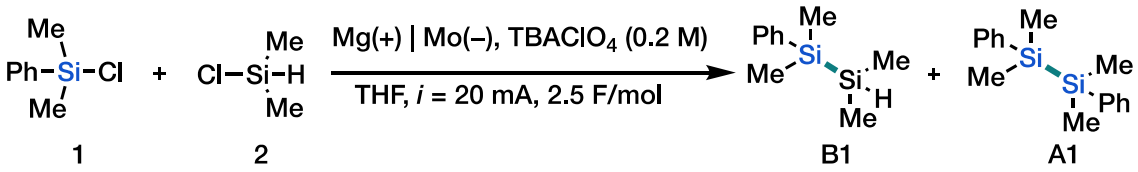
2.7. General procedure 6: cyclosilane synthesis



Magnesium electrode was polished with a razor until a shiny finish was obtained. Nickle foam electrode was used as received. In a glove box, $TBAClO_4$ (1 mmol, 2 equiv.), chlorosilane **Si-5** (0.5 mmol, 1 equiv.) and chlorosilane **Si-6** (0.55 mmol, 1.1 equiv.) were added into an oven-dried ElectraSyn vial (5 mL) equipped with a magnetic stir bar. Dried THF (4 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (nickle foam), then the assembly was brought out of the glove box. The reaction mixture was electrolyzed at a constant current of 10 mA until passing 5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was added to hexanes (10 mL) to precipitate $TBAClO_4$. The resultant mixture was then filtered through a short silica plug (8 cm thick, ca. 10 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and purified with column chromatography.

3. Reaction optimization

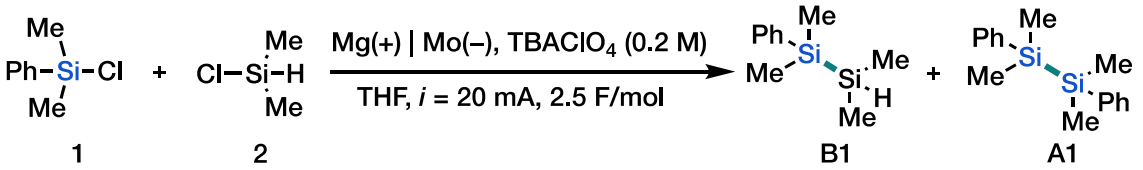
Table S1. Screening of electrode material for hetero-coupling



Entry	Electrode Variation	Yield% (B1)	Selectivity (B1 : A1)
1	**standard condition**	**70[a]**	**>20:1[b] (96:4)[c]**
2	Al (-) instead of Mo (-)	21	1:1
3	Cu (-) instead of Mo (-)	47 (42)	3:1
4	Pt (-) instead of Mo (-)	51 (47)	5:1
5	Sn (-) instead of Mo (-)	55 (50)	4:1
6	Ti (-) instead of Mo (-)	57 (55)	10:1
7	Glasy carbon (-) instead of Mo (-)	58 (60)	8:1
8	Ag (-) instead of Mo (-)	62 (58)	8:1
9	Mg (-) instead of Mo (-)	63 (55)	9:1
10	Zn (-) instead of Mo (-)	64 (61)	10:1
11	Graphite (-) instead of Mo (-)	65 (61)	10:1
12	Fe (-) instead of Mo (-)	67 (61)	10:1
13	**Ni (-) instead of Mo (-)**	**68**	**>20:1 (94:6)**
14	Cu (+) instead of Mg (+)	39	13:1
15	Al (+) instead of Mg (+)	n.d	n.d

[a] Reaction conditions: 1mmol chlorosilane 1, 1.5mmol of chlorosilane 2, $TBAClO_4$ (0.2 M), THF (8 mL), Mg anode, graphite cathode, undivided cell (10mL, ElectraSyn 2.0), constant current = 20 mA, 2.5 F/mol, 22°C. Yield determined by ^1H NMR using dibromomethane as internal standard. Yield in parentheses is isolated yield. [b] Selectivity determined by ^1H NMR. [c] Selectivity in parentheses determined by GC

Table S2. Screening of electrolytes and other parameters for hetero-coupling

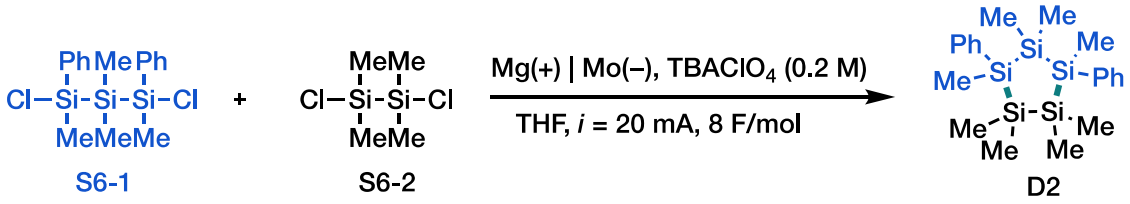


1 2 B1 A1

Entry	Variation from standard condition	Yield% (B1)[a]	Selectivity (B1:A1)[b]
1	standard condition	70	>20:1
2	$NaSbF_6$ or $NaBPh_4$ instead of $TBAClO_4$	0	n.d.
3	$TBABF_4$ instead of $TBAClO_4$	23	15:1
4	$LiNTf_2$ instead of $TBAClO_4$	29	14:1 (91:9)
5	TBAOTf instead of $TBAClO_4$	34	>20:1 (93:7)
6	$TBAPF_6$ instead of $TBAClO_4$	48	6:1
7	$TBABPh_4$ instead of $TBAClO_4$	66	>20:1 (94:4)
8	0.05 M or 0.1 M instead of 0.2 M $TBABPh_4$	65 / 67	>20:1 (95:5)
9[c]	**1.25 equiv of chlorosilane 2**	**65**	**>20:1 (93:6)**
10[c]	1 equiv of chlorosilane 2	53	10:1 (88:11)
11[d]	TMSCl instead of chlorosilane 2	50	6:1
12[e]	CH_3CN or DMF instead of THF	n.d.	n.d.
13	DME instead of THF	54	4:1
14	2F or 3F instead of 2.5F	65 / 67	>20:1
15[e]	10 mA or 40 mA instead of 20 mA	60	15:1
16[e]	1.25 equiv of chlorosilane 2	60	6:1

[a] Reaction conditions: 1mmol chlorosilane 1, 1.5mmol of chlorosilane 2, $TBAClO_4$ (0.2 M), THF (8 mL), Mg anode, Molybdenum cathode, undivided cell (10mL, ElectraSyn 2.0), constant current = 20 mA, Q = 2.5 F/mol, 22°C. Yield determined by ^1H NMR using dibromomethane as internal standard. Yield in parentheses is isolated yield. [b] Selectivity determined by ^1H NMR. Selectivity in parentheses determined by GC. [c] $TBABPh_4$ (0.1 M). [d] Hetero-coupling product in this entry is $Ph(Me)_2Si\text{-}Si(Me)_3$. Homo-coupling product remains the same. [e] Solvent polymerization observed for CH_3CN. [e]Graphite is used as the cathode.

Table S3. Screening of parameters for [3 + 2] annulation



Entry	Changes	Yield %
1	None	24
2	10 mA instead of 20 mA	39
3	$TBABPh_4$ (0.1 M) instead of $TBACIO_4$ (0.2 M)	26
4	6 F/mol instead of 5 F/mol	20
5	1.5 equiv instead of 1.1 equiv of chlorosilane S6-2	26
6	Ni foam (-) instead of Mo(-)	36
7	0.05 M instead of 0.1 M	27
8	**Ni foam (-) instead of Mo(-) and 10 mA instead of 20 mA**	**44**

[a]Reaction conditions: 0.5 mmol (1 equiv) chlorosilane S6-1, 0.55 mmol (1.1 equiv) of chlorosilane S6-1, $TBACIO_4$ (0.2 M), THF (4 mL), Mg anode, Molybdenum cathode, undivided cell (5 mL, ElectraSyn 2.0), constant current = 20 mA, Q= 5 F/mol, 22°C. Yield is isolated yield.

General comments on the reaction conditions:

The general procedures described herein proved applicable to a majority of substrates. For specific substrates when unsatisfying yields are obtained, the conditions can be further optimized by tuning simple parameters such as current (10-20 mA), cathode material (Ni, Mo, C), reaction time (increasing charge passed), which have proven effective in our study.

4. Control experiments

4.1. Divided cell electrolysis

A flame-dried, 10-mL H-type divided cell with glass frit was equipped with a stir bar in each well. A graphite cathode (1.0 × 0.5 cm^2) was attached to a septum using a silver wire. The anode (Mg bar or Zn plate) was attached to a septum using a three-inch long needle. The cell and electrodes were then transferred into the glovebox. Chlorodiphenylmethylsilane (**1**, 0.5 mmol, 1 equiv.), chlorodimethylsilane (**2**, 2 mmol, 4 equiv.), and $TBAClO_4$ (1.5 mmol, 3 equiv.) were added into cathode chamber. To the anodic chamber was added $TBAClO_4$ (1.5 mmol, 3 equiv.). 5.0 mL of THF was added to each chamber (10 mL in total). Subsequently, the chambers were capped with septa connected to the corresponding electrode before transferring out for electrolysis. The electrodes were connected to a Electrasyn device with a constant current of 3 mA. The reaction was terminated after passing 2.5 F/mol of charge. The reaction mixtures of each chamber were combined and washed with 5% diethyl ether/Hex (10.0 mL × 3). The combined mixture was then filtered through a silica plug (4 cm thick, ca. 5 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and the yield of the product was determined by ^1H NMR using dibromomethane as internal standard.

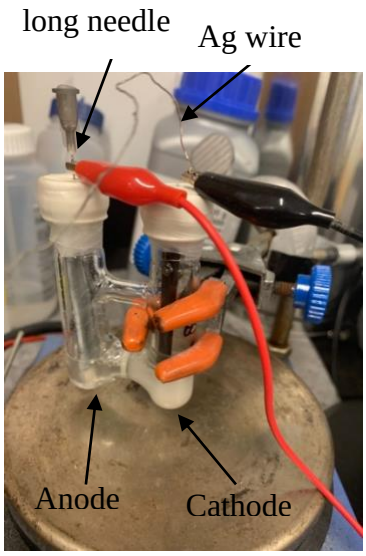
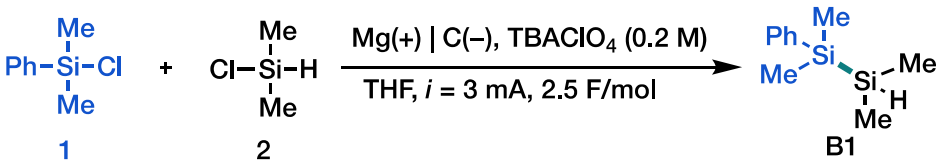


Table S4. Divided cell electrolysis



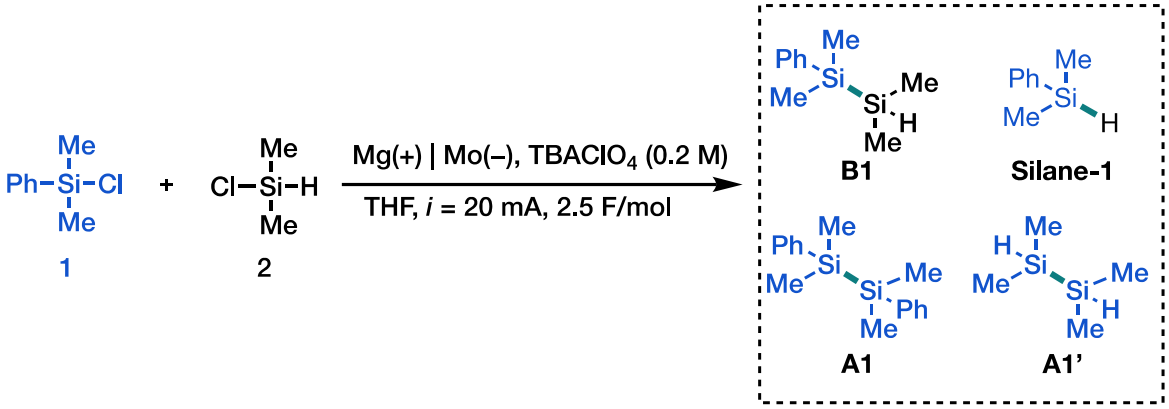
Entry	Changes	Yield (NMR)
1	None (undivided cell under standard condition)	65
2	Mg(+), C(-), divided cell	54
3	Zn(+) instead of Mg(+), divided cell	46
4	C(+) instead of Mg(+), 2 equiv Et_3N added to anode, divided cell	42
5	Zn(+) instead of Mg(+), $Mg(OTf)_2$ (1 equiv.) added to cathode, divided cell	38

Discussion:
Mg^{2+} generated from anodic oxidation of Mg is not necessary and thus its role is not invoked in the proposed reaction mechanism

4.2. Mass balance track by GC curve

Following general procedure 3, the reaction was set up and running inside of a glove box. Magnesium electrode was polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Molybdenum electrode was used as received. In glove box, $TBAClO_4$ (2 mmol, 2 equiv.), chlorosilane **1** (1 mmol, 1 equiv.), chlorosilane **2** (1.5 mmol, 1.5 equiv.) and hexadecane (1.0 mmol, 1 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (molybdenum plate). The reaction mixture was electrolyzed at a constant current of 20 mA until passing 2.5 F/mol of charge at room temperature. After electrolysis, an aliquot was added to a vial with 1 mL of diethyl ether (dried over molecular sieves) to precipitate $TBAClO_4$. The resultant mixture was then filtrated through a short cotton plug to a GC vial and flushed with 3 mL of diethyl ether. The crude mixture was analyzed by GC using standard curve (using hexadecane as internal standard) developed.

Table S5. Mass balance



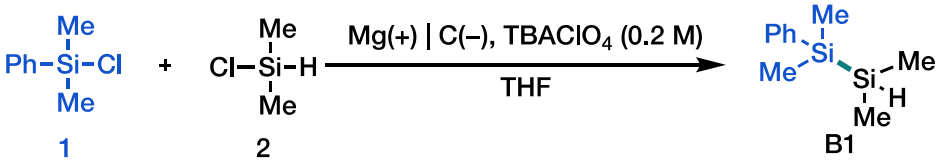
Reactions inside of GB

Entry	Changes	B1	A1	A1'	Silane-1	SM (1)
1	2.5 F/mol	75	1	2	2	7
2	3 F/mol	82	2	2	1	3
3	Add 0.25 equiv Si-H after 2.5 F/mol	72	2	4	2	5

4.3. Experiments without electricity or using magnesium powder

Following general procedure 3, in a glove box, $TBAClO_4$ (2 mmol, 2 equiv.), chlorosilane **1** (1 mmol, 1 equiv.) and chlorosilane **2** (1.5 mmol, 1.5 equiv.) were added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (graphite plate). For entry 2 below, 4 equiv. of Mg powder was also added to the reaction mixture. The reaction mixture was stirred overnight at room temperature. After electrolysis, the reaction mixture was collected and washed with 5% diethyl ether/hexanes (10 mL × 3). The combined mixture was then filtered through a silica plug (4 cm thick, ca. 5 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated under vacuum and the yield/selectivity of the product was determined by NMR with dibromomethane as IS.

Table S6. Control experiments

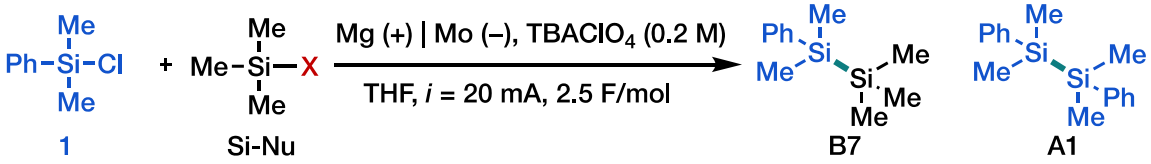


Entry	Changes	Yield (B1, NMR)
1	No electricity	n.d
2	Mg powder added instead of electricity	n.d

4.4. Selectivity with silanes with different leaving groups

Following general procedure 3, the reaction was set up inside of a glove box using different silane electrophiles. Magnesium electrode was polished with 500 grit silicon carbide sandpaper until a shiny finish was obtained. Molybdenum electrode was used as received. In a glove box, $TBAClO_4$ (2 mmol, 2 equiv.), chlorosilane **1** (1 mmol, 1 equiv.) and **TMS-X** (1.5 mmol, 1.5 equiv.) was added into an oven-dried ElectraSyn vial (10 mL) equipped with a magnetic stir bar. Dried THF (8 mL) was then added to the mixture. The vial was sealed with the ElectraSyn vial cap equipped with anode (Mg plate) and cathode (molybdenum plate). The reaction mixture was electrolyzed at a constant current of 20 mA until passing 2.5 F/mol of charge at room temperature. After electrolysis, the reaction mixture was collected and washed with 5% diethyl ether/hexanes (10 mL × 3). The combined mixture was then filtrated on a silica plug (4 cm thick, ca. 5 g) and flushed with 5% diethyl ether/hexanes (100 mL). The crude mixture was concentrated on vacuum and the yield/selectivity of the product was determined by isolation.

Table S7. Influence of the leaving group



Entry	Si-Nu (TMS-X)	Yield (B7)	Selectivity (B7 : A1)	Note
1	X=Cl	77	7:1	
2	X=Br	53	8:1	
3	X=OAc	19	No A1 isolated	Very High voltage

5. Cyclic voltammetry studies

All cyclic voltammetry studies were conducted inside the glovebox at room temperature with the Pine WaveNow® Potentiostat/Galvanostat System. Measurements were performed in 0.2 M $TBAClO_4$ in THF using a divided three-compartment cell. $Mg(OTf)_2$, which bears a redox innocent anion, was used as the Mg^{2+} source instead of $MgCl_2$ due to its higher solubility in THF. Control experiments revealed no difference of $Mg(OTf)_2$ and $MgCl_2$ in the scan range of -2.0 ~ 1.0 V vs $Mg^{2+/0}$. Scan rate is 50 mV/s unless specified.

Supporting Electrolyte: $TBAClO_4$ was recrystallized in ethyl acetate for three times and dried under vacuum at 60 °C overnight.

Solvent: THF was first dried overnight with potassium hydroxide. Then reflux in sodium metal and benzophenone under nitrogen for 5 h.

Working electrode: The working electrode is a 3 mm diameter glassy carbon working electrode. Polished with 0.3 μm aluminum oxide and then sonicated in distilled water before drying and transferring into the glovebox.

Reference electrode: The reference electrode consisted of a magnesium wire submerged in a saturated solution of magnesium chloride and THF. The Mg wire was polished with sand paper and washed with diethyl ether before transferring into the glovebox.

Counter electrode: The counter electrode is a platinum wire that was previous burnt for 30 seconds with a butane torch.

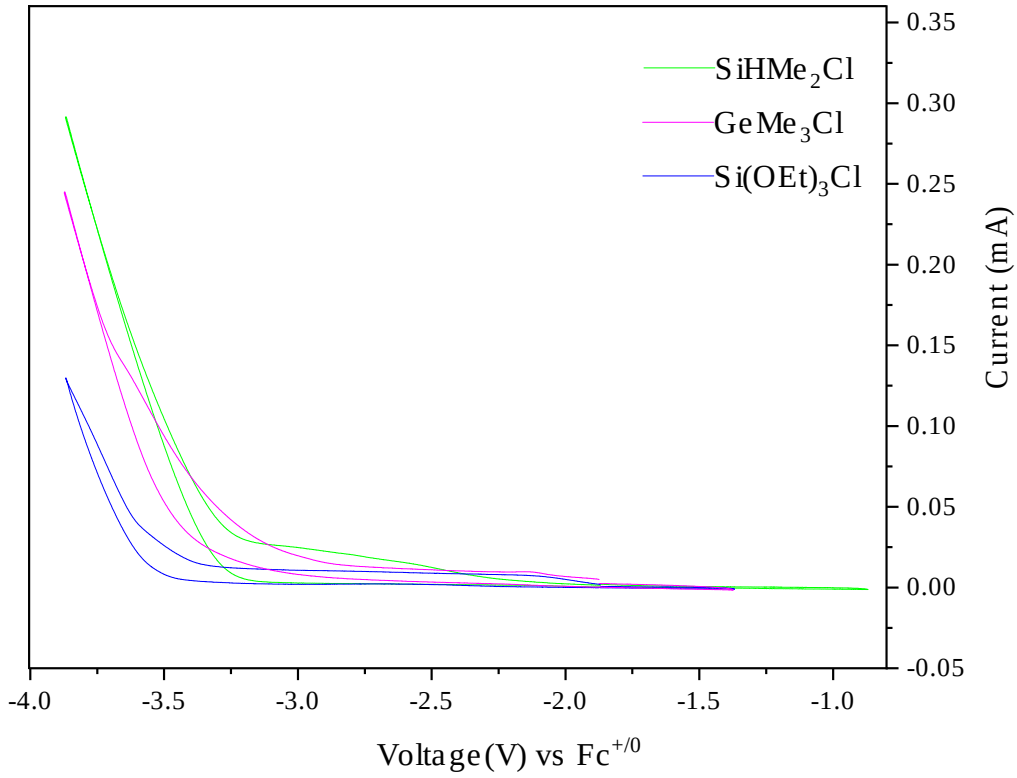


Fig. S1. Cyclic voltammetry of chlorosilanes (or chlorogermane): $SiHMe_2Cl$, $GeMe_3Cl$, and $Si(OEt)_3Cl$. The initial scan direction is reductive (i.e., from relatively positive potentials to negative potentials).

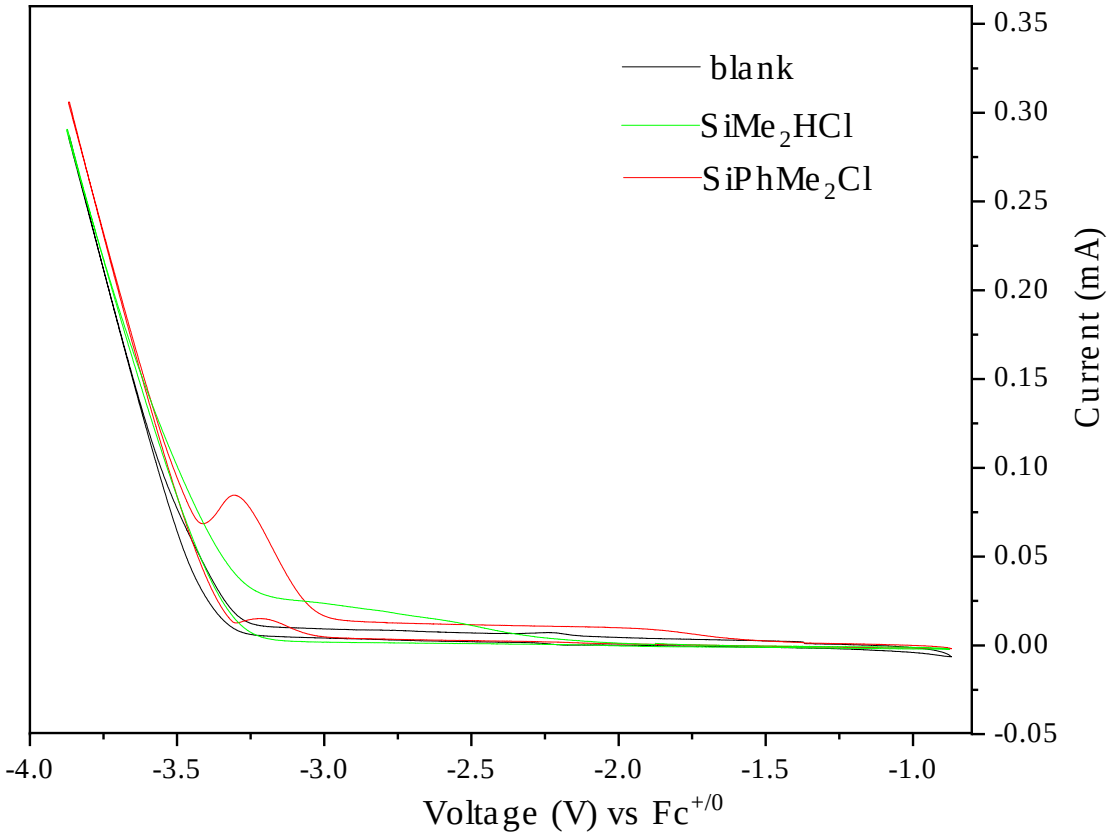


Fig. S2. Cyclic voltammetry of chlorosilanes: $SiHMe_2Cl$ and $SiPhMe_2Cl$. The initial scan direction is reductive (i.e., from relatively positive potentials to negative potentials).

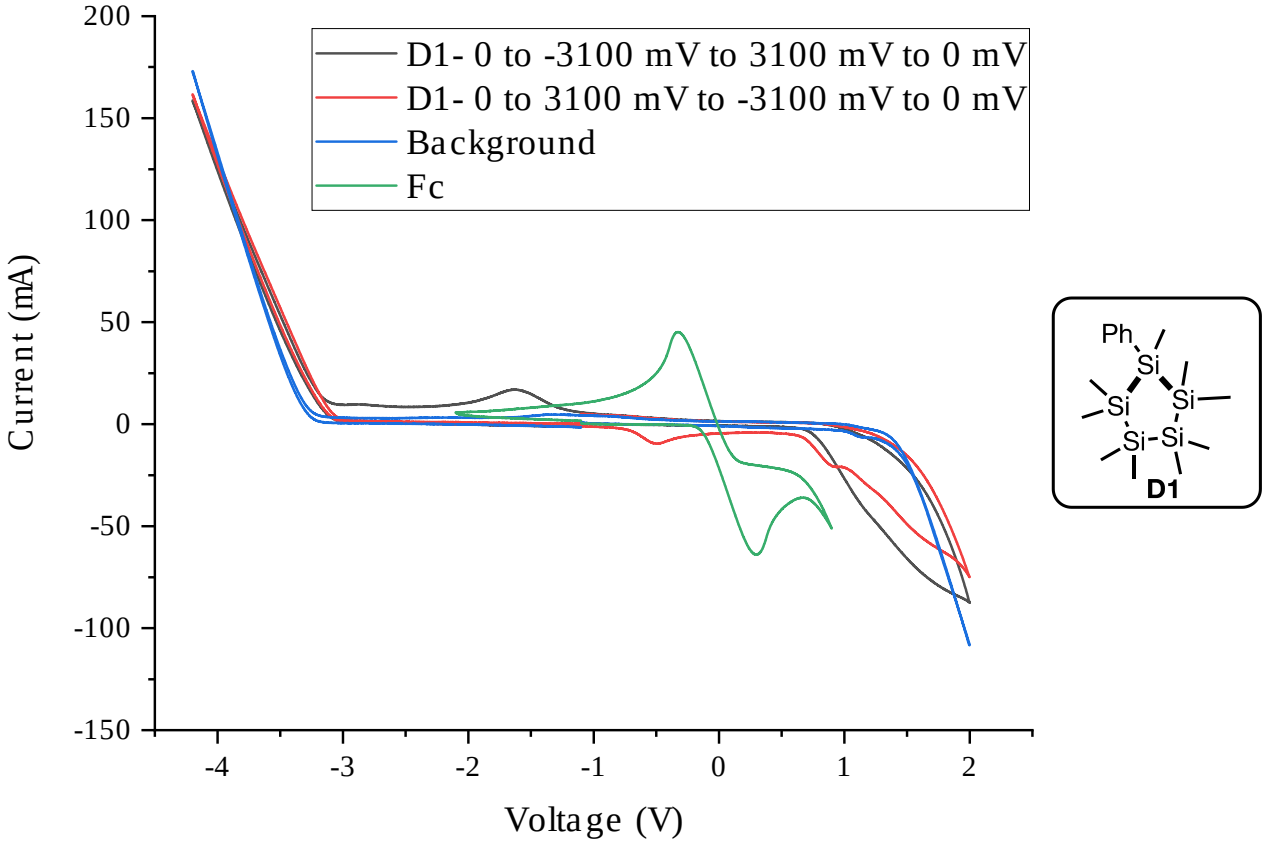


Fig. S3. Cyclic voltammetry of cyclosilane D1.

NOTES: black trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while red was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

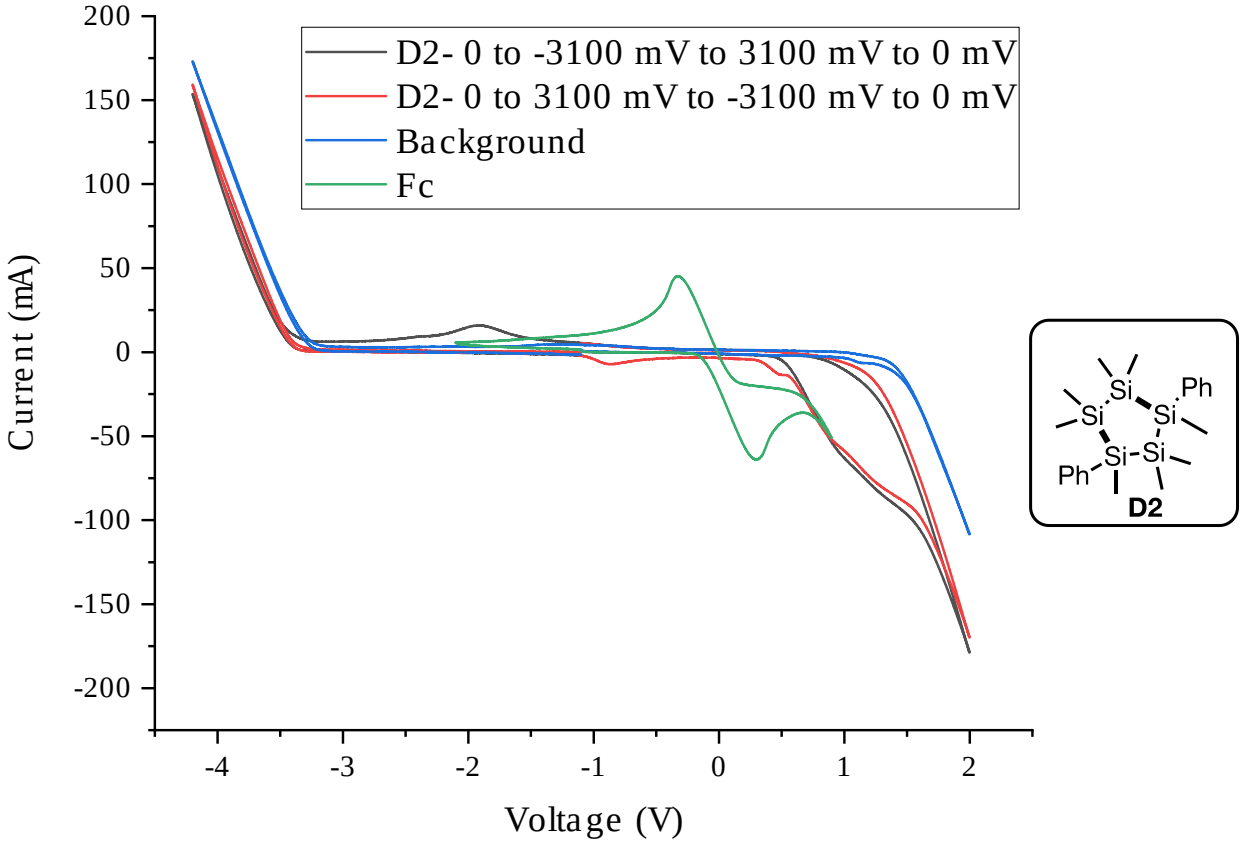


Fig. S4. Cyclic voltammetry of cyclosilane D2.

NOTES: black trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while red was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

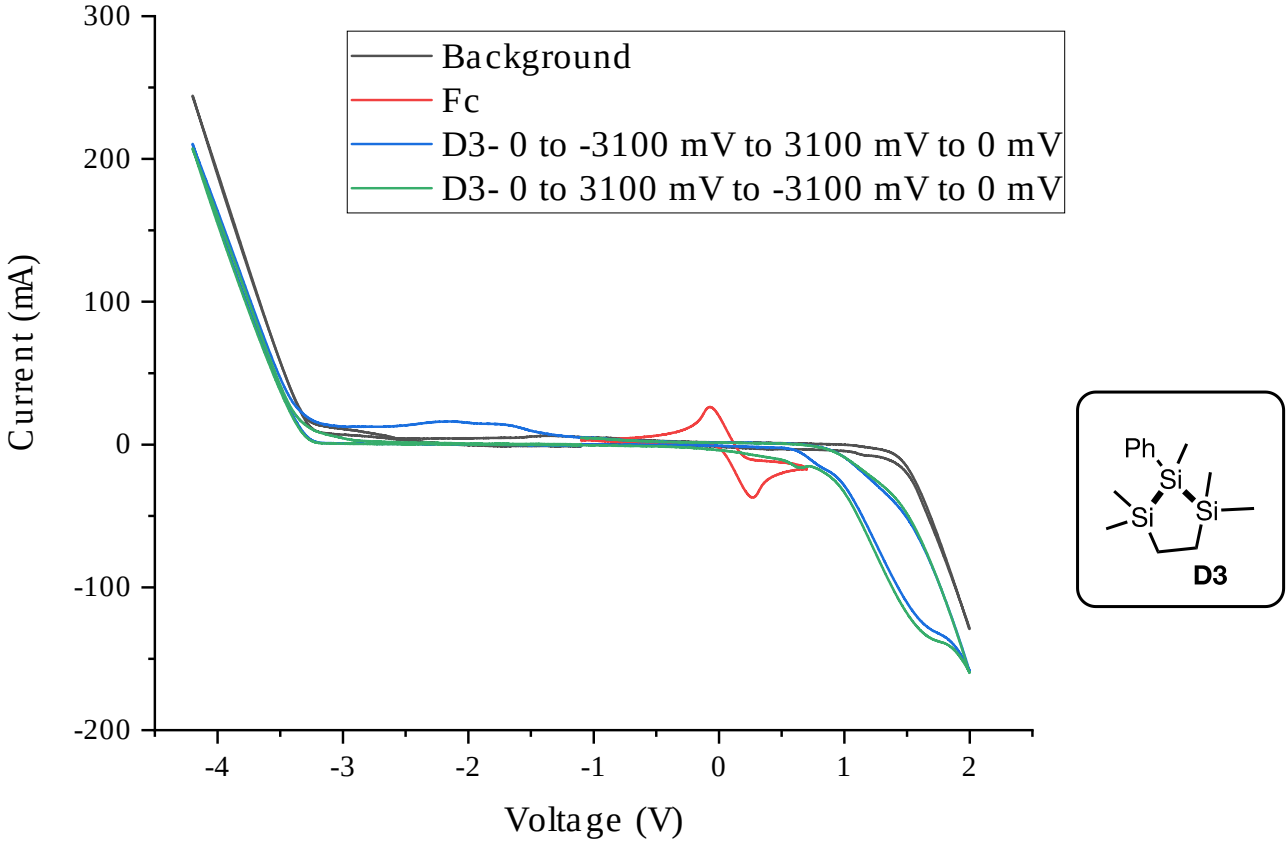


Fig. S5. Cyclic voltammetry of cyclosilane D3.

NOTES: blue trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while green was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

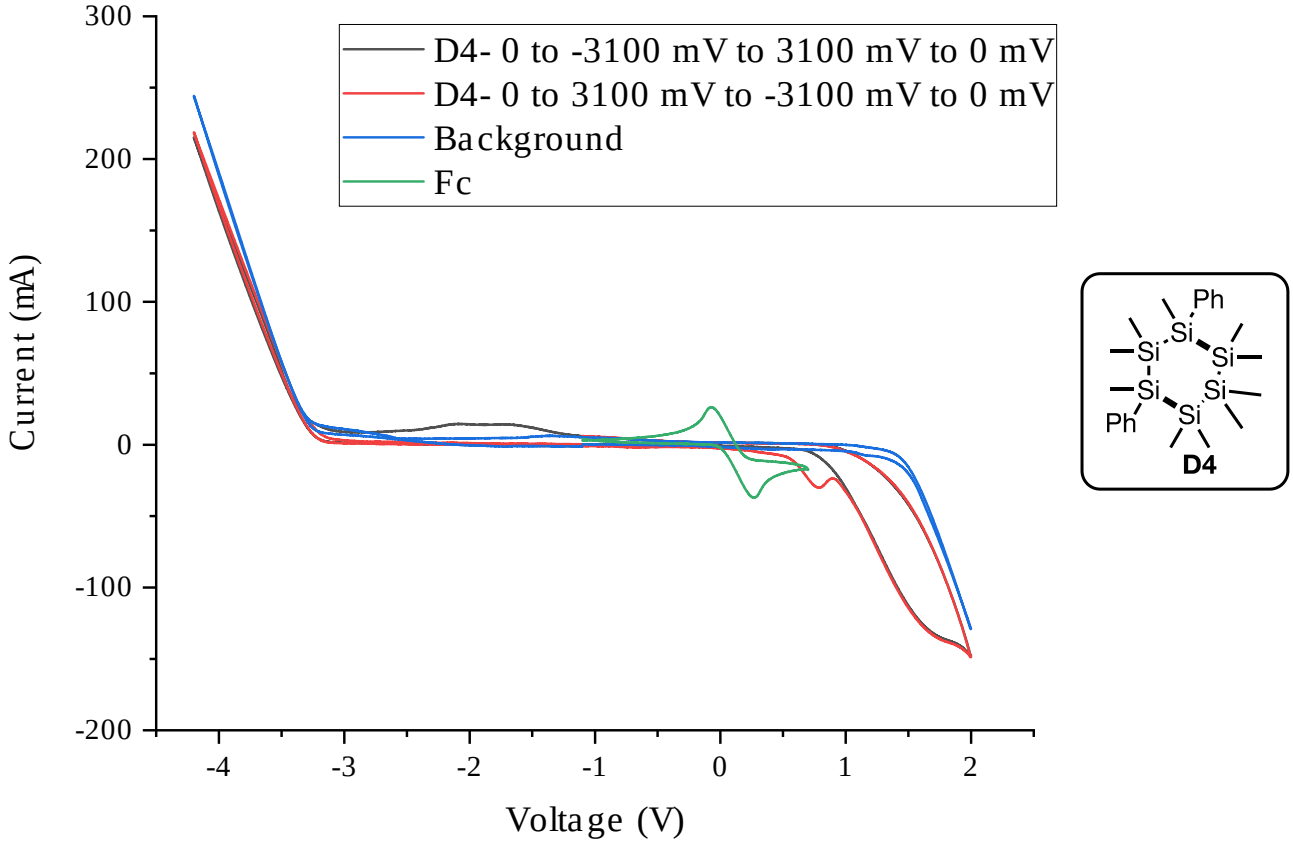


Fig. S6. Cyclic voltammetry of cyclosilane D4.

NOTES: black trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while red was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

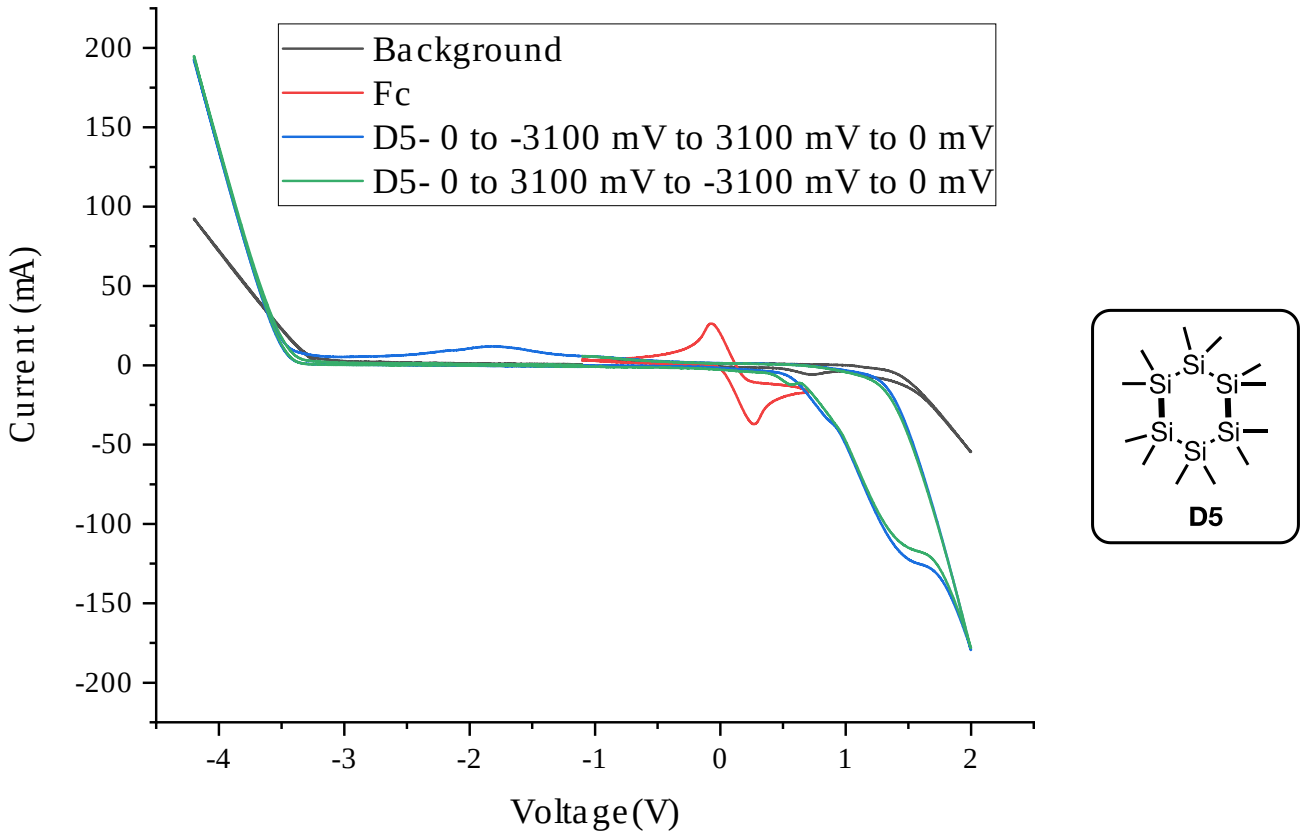


Fig. S7. Cyclic voltammetry of cyclosilane D5.

NOTES: blue trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while green was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

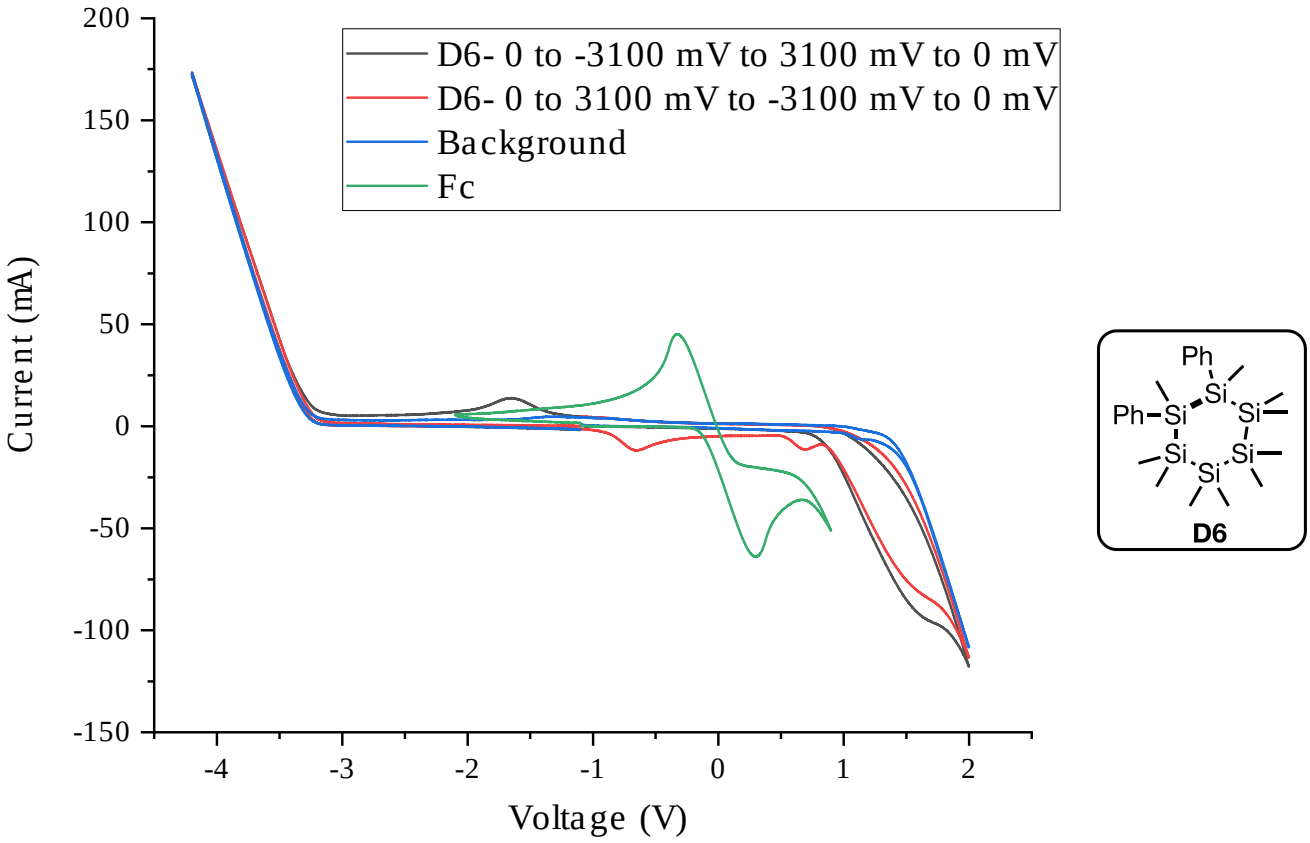
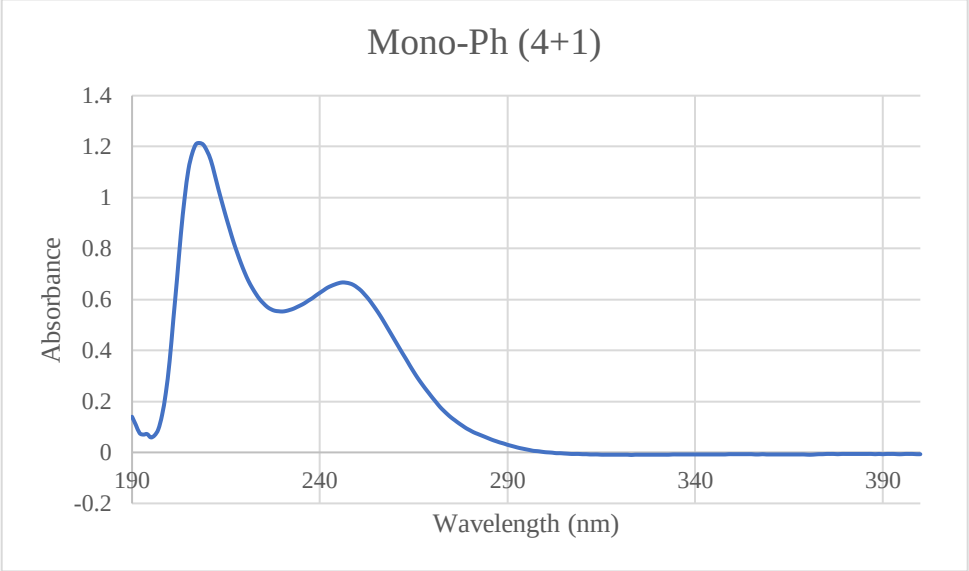


Fig. S8. Cyclic voltammetry of cyclosilane D6.

NOTES: black trace was scanned negatively then positively (0 mV to -3100 mV, then to 3100 mV and back to 0 mV), while red was the reverse (0 mV to 3100 mV, then to -3100 mV and back to 0 mV).

6. UV-Vis of cyclosilanes



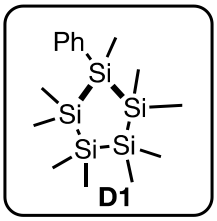
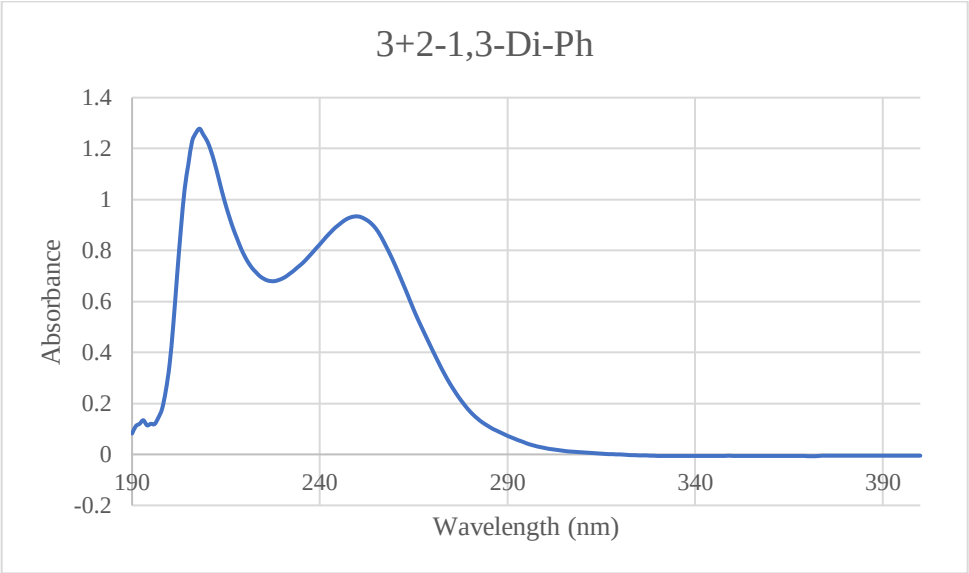


Fig. S9. UV-Vis of cyclosilane D1 in pentane ([D1]=1*10^{-5} mol/L).



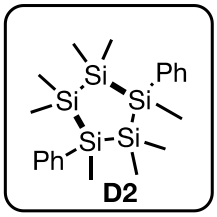
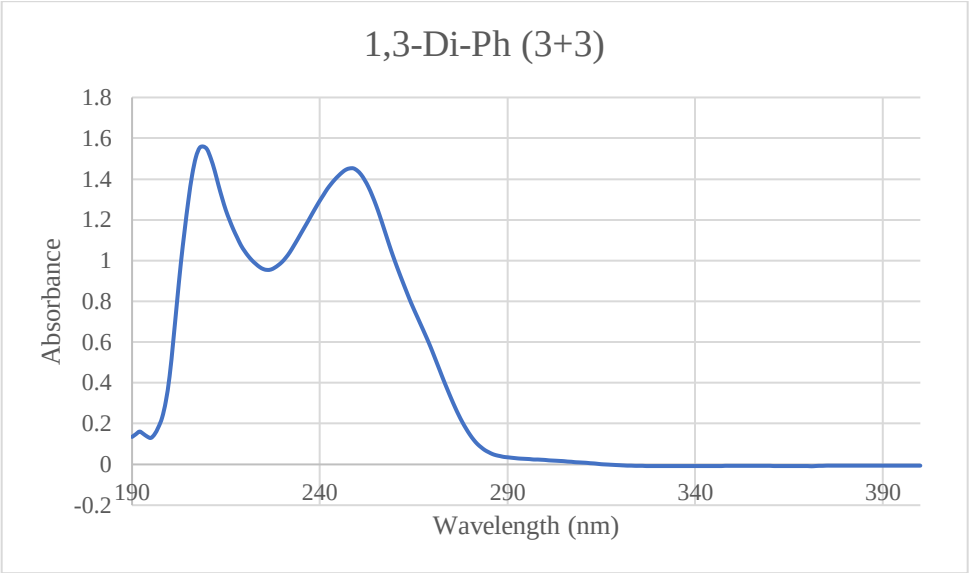


Fig. S10. UV-Vis of cyclosilane D2 in pentane ([D2]=1*10^{-5} mol/L).



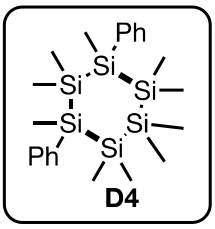
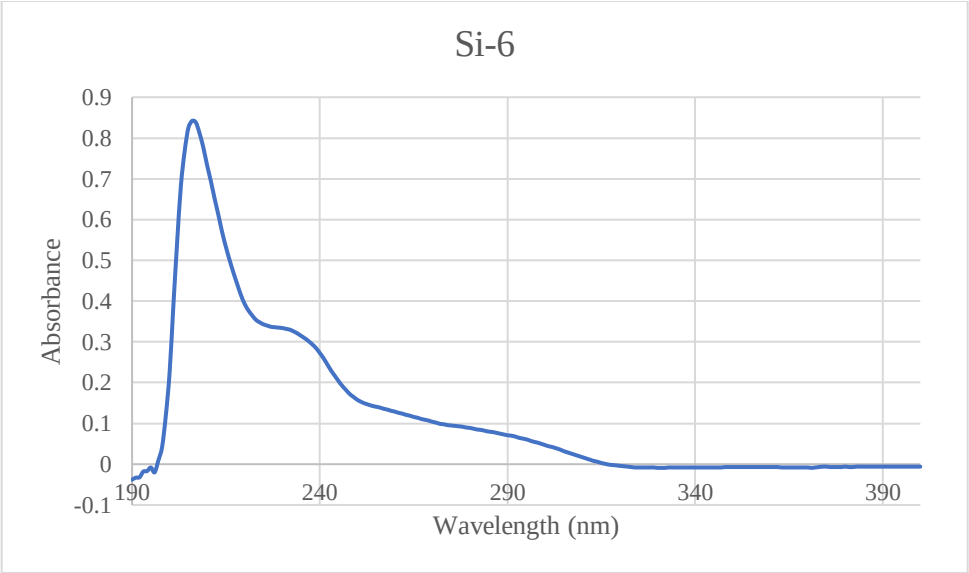


Fig. S11. UV-Vis of cyclosilane D4 in pentane ([D4]=1*10^{-5} mol/L).



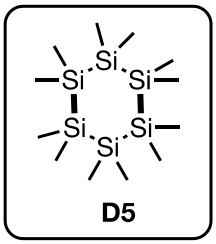
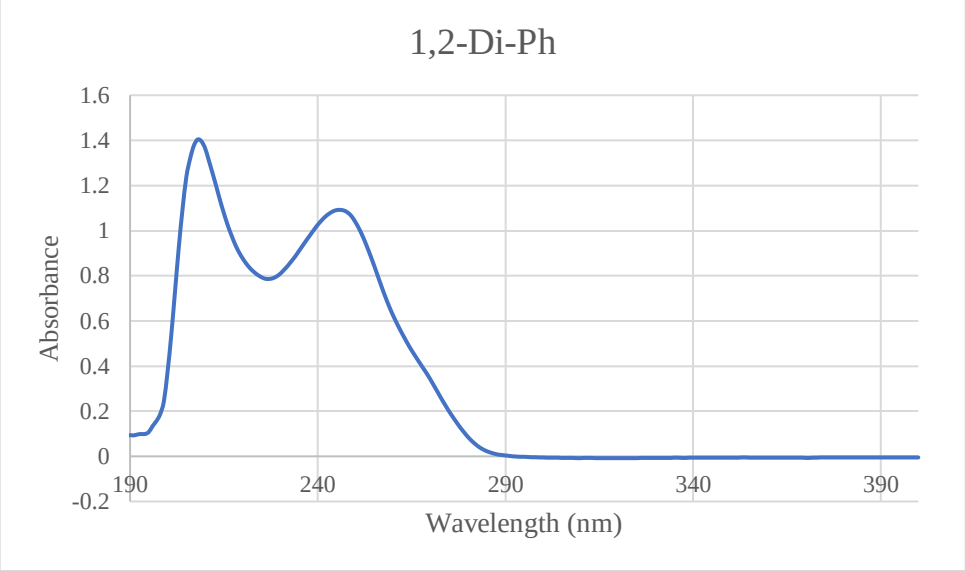


Fig. S12. UV-Vis of cyclosilane D5 in pentane ([D5]=1*10^{-5} mol/L).



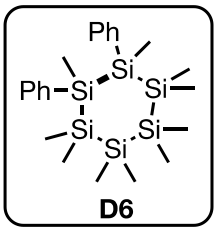


Fig. S13. UV-Vis of cyclosilane D6 in pentane ([D6]=1*10^{-5} mol/L).

7. DFT calculations

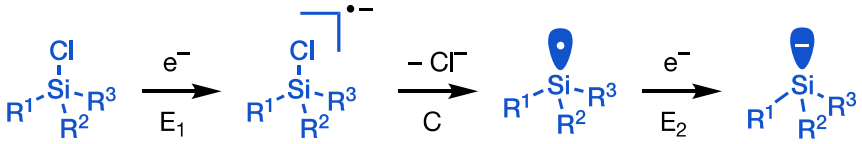
7.1. Reduction of chlorosilanes

Computational methods. All DFT calculations were performed with Gaussian 16.[1] Geometry optimizations were carried out in the gas phase using the (U)B3LYP (U = unrestricted) functional[2,3] and the 6-31+G(d) basis set.[4] Unscaled harmonic frequency calculations at the same level were performed to validate each structure as a minimum and to evaluate its zero-point energy and thermal corrections at 298 K. Quasiharmonic corrections were applied during the entropy calculations by setting all positive frequencies that are less than 100 cm^{-1} to 100 cm^{-1}.[5,6] On the basis of the optimized structures, single-point energy refinements were performed at the (U)B3LYP/maug-cc-pVTZ[7–11] level and the Gibbs energies of solvation were computed at the SMD(THF)/(U)B3LYP/6-31+G(d) level (standard state concentration: 1.0 M).[12]

Discussion. We proposed that the electroreduction of chlorosilanes proceeds through an ECE mechanism (Table S8). The reduction potential of the first cathodic reduction (E_1) is highly dependent on the substituent effect. Compared with unactivated chlorosilanes (e.g., Me_2HSiCl and Me_3SiCl), $PhMe_2SiCl$ and $(Me_3Si)_3SiCl$ show significantly less negative reduction potentials due to the presence of stabilizing phenyl and silyl groups. The sequential C−Cl bond cleavage step (C) is barrierless and exergonic, indicating that the silyl radical generation is irreversible. The second cathodic event (E_2) requires less reduction potential than the first one, indicating that the silyl anion generation should be very facile once the silyl radical is generated. Combining together, we conclude that the first reduction event determines which radical is more favorably generated and eventually which anion is prevalent for the nucleophilic substitution.

Though we had split the overall two-electron reduction into E_1, C, and E_2 steps computationally, the actual cathodic event can hardly be decoupled completely. Thus, the radical anion $R_3SiCl^{\bullet-}$ should be considered as a hypothetical species that resembles the reductive C−Cl cleavage transition state rather than a detectable intermediate. We envision that the computed $E°(E_1)$ among different chlorosilanes could reflect, to some extent, the trend in difficulty of the electroreduction.

Table S8. Reduction of chlorosilanes via an ECE mechanism



chlorosilane	$E°(E_1)$ (V)	ΔG(C) (kcal/mol)	$E°(E_1C)$ (V)	$E°(E_2)$ (V)
$PhMe_2SiCl$	−3.73	−8.7	−3.36	−2.38
Me_2HSiCl	−4.67	−28.9	−3.42	−2.27
Me_3SiCl	−4.56	−24.3	−3.51	−2.49
$(Me_3Si)_3SiCl$	−3.42	−13.4	−2.83	−2.16

Computed at the (U)B3LYP/maug-cc-pVTZ:SMD(THF)//(U)B3LYP/6-31+G(d) level. The reduction potentials were referenced to ferrocene/ferrocenium couple in THF.

Table S9. Computed energies of stationary points in Section 7.1

	TCG[a,b] (a.u.)	SPE[a] (a.u.)	SPE[c] (a.u.)	SPE[d] (a.u.)
ferrocene	0.138679	−1650.731059	−1651.013252	−1650.743363
ferrocenium	0.138050	−1650.471264	−1650.754612	−1650.551110
Cl^-	−0.015023	−460.274726	−460.310516	−460.367485
$PhMe_2SiCl$	0.130406	−1061.267656	−1061.438738	−1061.279293
$PhMe_2SiCl^{\bullet-}$	0.123387	−1061.257857	−1061.429156	−1061.325356
$PhMe_2Si^{\bullet}$	0.128475	−600.960747	−601.092236	−600.968809
$PhMe_2Si^-$	0.126801	−601.001049	−601.132299	−601.070484
Me_2HSiCl	0.054042	−830.198244	−830.299622	−830.203356
$Me_2HSiCl^{\bullet-}$	0.051474	−830.170331	−830.276274	−830.214960
$Me_2HSi^{\bullet}$	0.052463	−369.890001	−369.951144	−369.891526
Me_2HSi^-	0.050935	−369.923375	−369.984468	−369.996945
Me_3SiCl	0.081435	−869.529319	−869.645258	−869.534968
$Me_3SiCl^{\bullet-}$	0.079126	−869.508093	−869.628303	−869.551132
$Me_3Si^{\bullet}$	0.079873	−409.217958	−409.294042	−409.219507
Me_3Si^-	0.078359	−409.248434	−409.324270	−409.317340
$(Me_3Si)_3SiCl$	0.287262	−1977.585194	−1977.875879	−1977.591483
$(Me_3Si)_3SiCl^{\bullet-}$	0.283684	−1977.594578	−1977.883451	−1977.654661
$(Me_3Si)_3Si^{\bullet}$	0.286914	−1517.298882	−1517.550057	−1517.301654
$(Me_3Si)_3Si^-$	0.284845	−1517.358295	−1517.610812	−1517.409215

Cartesian coordinates are available upon request from one of the corresponding authors (Song Lin: songlin@cornell.edu). [a]Computed at the (U)B3LYP/6-31+G(d) level. [b]Computed at 298 K and 1 atm with quasiharmonic corrections. [c]Computed at the (U)B3LYP/maug-cc-pVTZ//(U)B3LYP/6-31+G(d) level. [d]Computed at the SMD(THF)/(U)B3LYP/6-31+G(d)//(U)B3LYP/6-31+G(d) level. TCG, thermal correction to Gibbs energy; SPE, single-point energy.

7.2. Nucleophilic substitution of chlorosilanes

Computational methods. Geometry optimizations in THF were carried out with Gaussian 16[1] using the SMD solvation model,[12] the B3LYP functional[2,3] and the 6-31+G(d) basis set.[4] Unscaled harmonic frequency calculations at the same level were performed to validate each structure as either a minimum or a transition state and to evaluate its zero-point energy and thermal corrections at 298 K. Quasiharmonic corrections were applied during the entropy calculations by setting all positive frequencies that are less than 100 cm^{-1} to 100 cm^{-1}.[5,6] On the basis of the optimized structures, single-point energy refinements were performed at the SMD(THF)/B3LYP/maug-cc-pVTZ[7−11] level. All discussed energy differences are based on Gibbs energies in THF at 298 K (standard state concentration: 1.0 M). Generalized Kohn−Sham energy decomposition analysis[13,14] was performed with GAMESS[15] and XEDA.[16] Three-dimensional structures were prepared with CYLview.[17]

Discussion. To shed light on the selectivity between homocoupling and hetero-coupling, we further investigated the reductive coupling of $PhMe_2SiCl$ and Me_2HSiCl. As mentioned above, $PhMe_2SiCl$ is easier to reduce than Me_2HSiCl due to the presence of a stabilizing phenyl group. Upon selective reduction of $PhMe_2SiCl$ and generation of silyl anion $PhMe_2Si^-$, it could react with either $PhMe_2SiCl$ or Me_2HSiCl via nucleophilic substitution. DFT calculations indicated that the Si−Si bond formation processes proceed through a concerted mechanism but involve highly asynchronous transition states

TS1 and **TS2** (Fig. S9). The computed Gibbs energy of activation for homocoupling and hetero-coupling is 17.4 and 13.7 kcal/mol, respectively, suggesting that the hetero-coupling will be kinetically favored over the homocoupling. These results are in good accordance with our experimental observations.

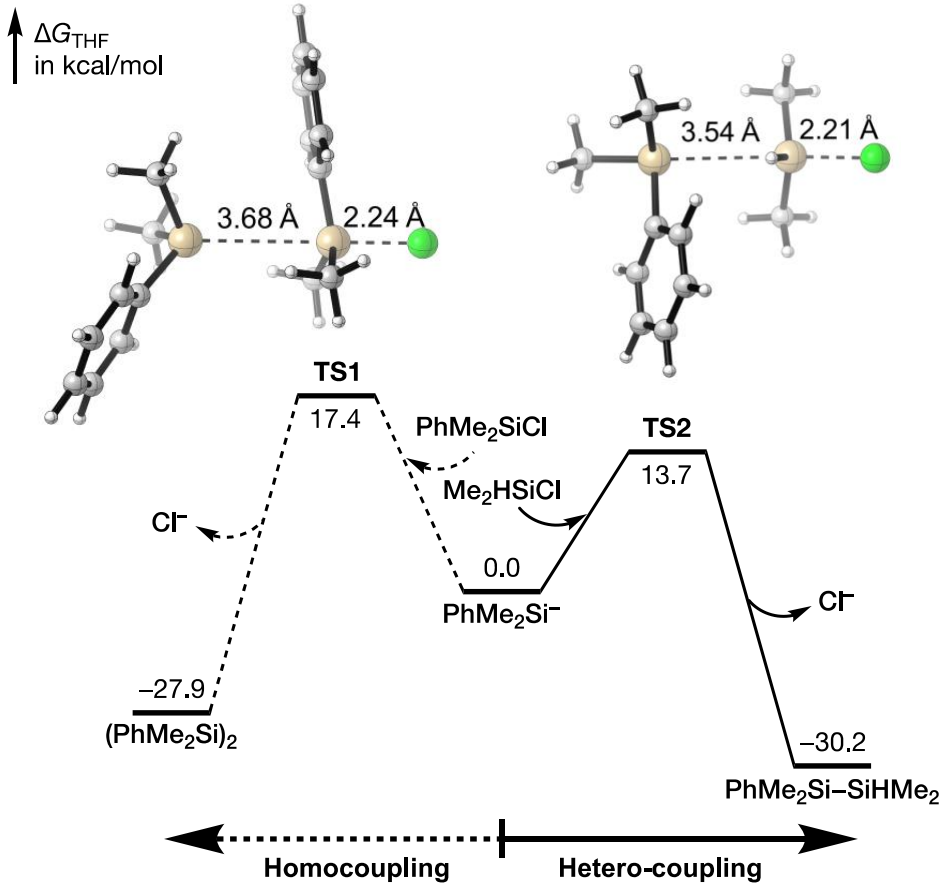


Fig. S14. Selectivity between homocoupling and hetero-coupling. Computed at the SMD(THF)/B3LYP/maug-cc-pVTZ//SMD(THF)/B3LYP/6-31+G(d) level. Hypothetical pentacoordinate siliconate intermediates in an alternative stepwise addition−elimination pathway are not stationary points on the potential energy surface.

To further understand the selectivity, we applied the distortion/interaction−activation strain model[18] on the Si−Si bond activation transition states (**TS1** and **TS2**). The electronic energy change (ΔE) was decomposed into six components: the distortion energy of the silyl anion [$\Delta E^{dist}(PhMe_2Si^-)$], the distortion energy of the chlorosilanes [ΔE^{dist}(chlorosilane)], electrostatic interaction (ΔE^{ele}), Pauli repulsion (ΔE^{Pauli}), polarization (ΔE^{pol}), and correlation (ΔE^{corr}). As shown in Table S10, the closed-shell Pauli repulsion term determines the selectivity between homocoupling and hetero-coupling—in other word, the sterically more accessible Me_2HSiCl will react with the silyl anion preferentially.

Table S10. Energy decomposition analysis

	TS1 (homocoupling)	**TS2** (hetero-coupling)	ΔE(**TS1**) − ΔE(**TS2**)
ΔE	−6.3	−8.3	2.0
ΔE^{dist}($PhMe_2Si^-$)	0.1	0.0	0.1
ΔE^{dist}(chlorosilane)	6.9	5.0	1.9
ΔE^{ele}	−30.1	−25.7	−4.4
ΔE^{Pauli}	35.5	27.4	8.1
ΔE^{pol}	−13.9	−13.1	−0.8
ΔE^{corr}	−4.8	−1.9	−2.9

Computed at the B3LYP/6-31+G(d)//SMD(THF)/B3LYP/6-31+G(d) level. Basis set superposition error was not taken into account.

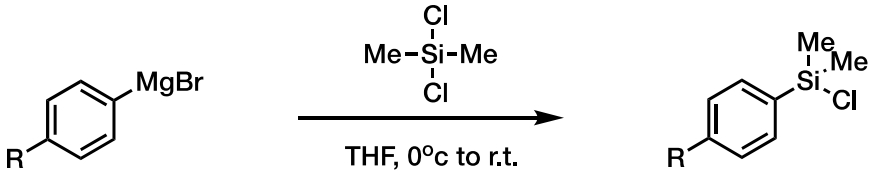
Table S11. Computed energies of stationary points in Section 7.2

	TCG[a,b] (a.u.)	SPE[a] (a.u.)	SPE[c] (a.u.)
$PhMe_2Si^-$	0.127901	−601.070711	−601.201601
$PhMe_2SiCl$	0.130372	−1061.279657	−1061.449725
TS1 (homocoupling)	0.280320	−1662.342403	−1662.642559
$(PhMe_2Si)_2$	0.284636	−1202.043988	−1202.303698
Cl^-	−0.015023	−460.367485	−460.403455
Me_2HSiCl	0.053501	−830.203593	−830.304404
TS2 (hetero-coupling)	0.202909	−1431.271722	−1431.502654
$PhMe_2Si{-}SiHMe_2$	0.206573	−970.970265	−971.160760

Cartesian coordinates are available upon request from one of the corresponding authors (Song Lin: songlin@cornell.edu). [a]Computed at the SMD(THF)/B3LYP/6-31+G(d) level. [b]Computed at 298 K and 1 atm with quasiharmonic corrections. [c]Computed at the SMD(THF)/B3LYP/maug-cc-pVTZ//SMD(THF)/B3LYP/6-31+G(d) level. TCG, thermal correction to Gibbs energy; SPE, single-point energy.

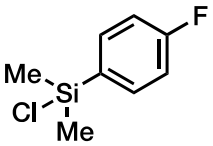
8. Synthesis and characterization of chlorosilanes

General procedure A [19,20]

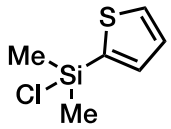


Chlorosilane synthesis using commercially available Grignard reagents:
A dry Schlenk flask under positive pressure of N_2 was charged with dry Et_2O (10 mL, dried over MS 4A) and Me_2SiCl_2 (3.65 mL, 1.5 equiv.), and the solution was cooled to 0 °C. Next, commercially available magnesium bromide solution (20 mL, 1 M in THF, 20 mmol, 1 equiv.) was added dropwise, after 10 min at 0 °C, the ice bath was removed and the solution was further stirred at rt for 15 h. Volatiles were removed directly in vacuo (~2 torr) at rt, and dry hexanes (30 mL, dried over MS 4A) was added to the crude chlorosilane. The suspension was filtered under inert atmosphere, and hexanes was removed directly in vacuo (~2 torr) at rt. The product was purified by Kugelrohr distillation.

Chlorosilane synthesis using freshly prepared Grignard reagents:
Grignard formation: A 100-mL round bottom flask was charged with Mg (583 mg, 24 mmol, 1.2 equiv.), corresponding bromoanisole (4.062 g, 20 mmol, 1 equiv.), and dry THF (40 mL). Next, the reaction mixture was heated to reflux for 2 hours. **Notes about Grignard formation**: 1.00 mL of the Grignard solution was titrated with I_2 (63.5 mg, 0.250 mmol) to calculate the ArMgBr concentration.
Chlorosilane synthesis: Similar to the procedure described above.

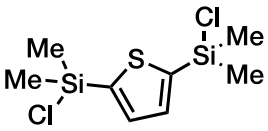


Prepared according to GPA using commercially available 4-fluorophenylmagnesium bromide solution (20.0 mL, 1 M in THF, 20 mmol, 1 equiv.) according to GP1 described above. Condition for Kugelrohr distillation: 185-190 °C, under nitrogen atmosphere ~1 atm. colorless oil (2.800 g, 14.8 mmol, 74% yield). Known compound.**^{1}H NMR** (500 MHz, C_6D_6) δ 0.32-0.42(s, 6H), 6.75-6.87(m, 2H), 7.22-7.29 (m, 2H) **^{13}C NMR** (101 MHz, C_6D_6) δ 164.29, 161.81, 133.96, 133.88, 130.38, 130.34, 113.87, 0.22. **^{29}Si NMR** (99 MHz, C_6D_6) δ 19.66. **^{19}F NMR** (376 MHz, C_6D_6) δ -109.63.

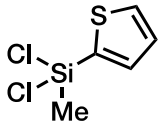


Prepared according to GPA with slight modification. A dry Schlenk flask under positive pressure of N_2 was charged with dry Et_2O (10 mL, dried over MS 4A) and thiophene (2.40 mL, 30 mmol, 1.0 equiv.), and the solution was cooled to 0 °C. *n*-BuLi in hexanes (18.8 mL, 1.6 M, 30 mmol, 1 equiv.) was slowly added to the solution, the reaction was further stirred at 0 °C for 10 min, and at rt for 3 h. A separate dry Schlenk flask under positive pressure of N_2 was charged with dry Et_2O (10 mL, dried over MS 4A) and Me_2SiCl_2 (5.43 mL, 1.5 equiv.), and the solution was cooled to 0 °C. The organolithium solution was slowly transferred via syringe to the chlorosilane solution. After 10 min

at 0 °C, the ice bath was removed, and the solution was further stirred at rt for 15 h. The suspension was filtered under inert atmosphere, and volatiles were removed directly in vacuo (~2 torr) at rt. The product was purified by Kugelrohr distillation (160-165 °C, under N_2 atmosphere ~1 atm) to obtain a colorless oil (3.810 g, 21.6 mmol, 72% yield). Known compound. **^{1}H NMR** (500 MHz, C_6D_6) δ 0.42-0.48(s, 6H), 6.83-6.89(m, 1H), 7.17-7.22 (m, 2H) **^{13}C NMR** (101 MHz, C_6D_6) δ 135.77, 135.52, 132.07, 128.20, 2.73. **^{29}Si NMR** (99 MHz, C_6D_6) δ 14.63.

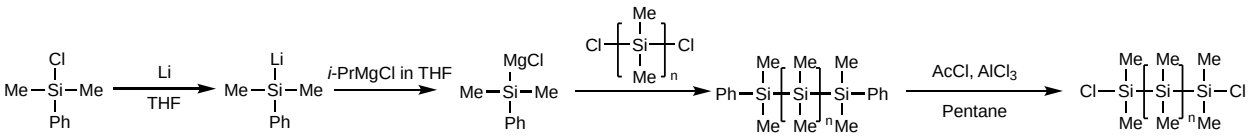


Prepared similarly to literature reports.[20] A solution of *n*-BuLi in hexanes (25.0 mL, 1.6 M, 40 mmol, 2 equiv.) was added dropwise at room temperature to a solution of thiophene (1.60 mL, 20 mmol, 1 equiv.) and dry TMEDA (6.00 mL, 40.0 mmol, 2 equiv.) in dry hexanes (5 mL). The resulting mixture was then heated under reflux (70 °C, oil bath temperature) for 90 min. After cooling to -78 °C, Me_2SiCl_2 (12.1 mL, 100 mmol, 5 equiv.) was added and the resulting solution was heated under reflux for 2 h (70 °C, oil bath temperature). The mixture was filtered under inert atmosphere, and the volatiles were removed in vacuo (~2 torr, rt). The residue obtained was distilled under N_2 atmosphere (~1 atm): the fraction collected between rt-200 °C contained TMEDA and small amounts of side products and was discarded, the fraction collected between 200-275 °C contained the desired product with residues of TMEDA. The desired product was further purified in a second distillation (under N_2 atmosphere ~1 atm), collecting the material distilled between 260-275 °C, affording product as a colorless oil (2.102 g, 7.80 mmol, 39% yield). **^{1}H NMR** (500 MHz, C_6D_6) δ 0.43-0.48(s, 12H), 7.24-7.29 (s, 2H) **^{13}C NMR** (101 MHz, C_6D_6) δ143.18, 136.76, 2.73. **^{29}Si NMR** (99 MHz, C_6D_6) δ 14.50.



Prepared according to GPA with modification. A dry Schlenk flask under positive pressure of N_2 was charged with dry Et_2O (10 mL, dried over MS 4A) and thiophene (2.40 mL, 30 mmol, 1.0 equiv.), and the solution was cooled to 0 °C. *n*-BuLi in hexanes (18.8 mL, 1.6 M, 30 mmol, 1 equiv.) was slowly added to the solution, the reaction was further stirred at 0 °C for 10 min, and at rt for 2 h. A separate dry Schlenk flask under positive pressure of N_2 was charged with dry Et_2O (10 mL, dried over MS 4A) and $MeSiCl_3$ (17.6 mL, 5 equiv.), and the solution was cooled to 0 °C. The organolithium solution was slowly transferred via syringe to the chlorosilane solution. After 10 min at 0 °C, the ice bath was removed and the solution was further stirred at rt for 15 h. The suspension was filtered under inert atmosphere, and volatiles were removed directly in vacuo (~2 torr) at rt. The product was purified by Kugelrohr distillation (140-160 °C, under N_2 atmosphere ~1 atm) to obtain a colorless oil (2.398 g, 12.2 mmol, 41% yield).
^{1}H NMR (500 MHz, C_6D_6) δ 0.58-0.67(s, 3H), 6.73-6.79(t, 1H), 7.09-7.13 (d, 1H), 7.21-7.26 (d, 1H), 7.24-7.29 (s, 2H) **^{13}C NMR** (101 MHz, C_6D_6) δ137.38, 133.68, 132.60, 128.34, 6.01. **^{29}Si NMR** (99 MHz, C_6D_6) δ 12.08

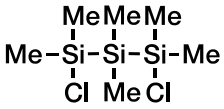
General Procedure B:[22,23]



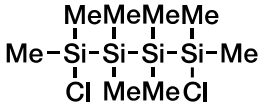
The following substrates were synthesized through optimized procedures adapted from known literature.[22,23]

Synthesis of Diphenyloligosilanes: An oven-dried Schlenk flask outfitted with a stir bar was cooled under an Argon atmosphere. To the flask was added granular lithium (8.0 equiv.), which was suspended in tetrahydrofuran (making 1 M THF solution based on dichlorosilane). Chloro(dimethyl)phenylsilane (2.0 equiv.) was added to the lithium suspension dropwise via syringe. The clear, colorless solution was observed to darken to a dark red color within one hour after addition of chloro(dimethyl)phenylsilane and the heterogeneous mixture was allowed to stir at room temperature overnight. Under argon, the reaction mixture was filtered through a fritted Schlenk tube into the receiving flask to remove excess lithium, and the dark red solution was cooled to 0 °C in an ice bath. Isopropylmagnesium chloride (2M in THF, 2.0 equiv.) was added dropwise via syringe, and the solution was stirred an additional 30 minutes at 0 °C. Dichlorosilane (1.0 equiv.) was added dropwise. The solution was allowed to warm to room temperature with stirring overnight. The reaction was cooled in an ice bath and quenched by the careful, dropwise addition of saturated NH_4Cl aqueous solution (half volume of THF), followed by DI water (half volume of THF). The aqueous and organic layers were separated, and the aqueous layer extracted twice with diethyl ether. The combined organic layers were dried with magnesium sulfate, filtered, and concentrated, yielding a yellow oil. The products are purified by automated column chromatography on silica gel eluting with hexanes.

Synthesis of Dichlorooligosilanes: An oven-dried Schlenk flask outfitted with a stir bar was cooled under an Argon atmosphere. To the flask was added anhydrous aluminum chloride (2.2 equiv.) under high flow of argon. After the addition, the flask was sealed with a septum, then purged under vacuum and backfilled with argon 3 times. Pentane (making 0.8 M solution based on diphenyloligosilane) and diphenyloligosilane (1.0 equiv.) were added yielding a suspension. The mixture was cooled to 0 °C in an ice bath and acetyl chloride (AcCl, 2.2 equiv.) with added slowly via syringe pump in 30 minutes. Orange precipitates were observed during the addition. The reaction mixture was allowed to warm to room temperature and stirred overnight. Then stirring was stopped and the top clear solution was transferred to a receiving flask through cannula under argon protection. The residual solids were washed 3 times by pentane and all the fractions were combined in the receiving flask. Pentane was distilled at 50 °C under argon protection and the product (dichlorooligosilane) was distilled under vacuum.

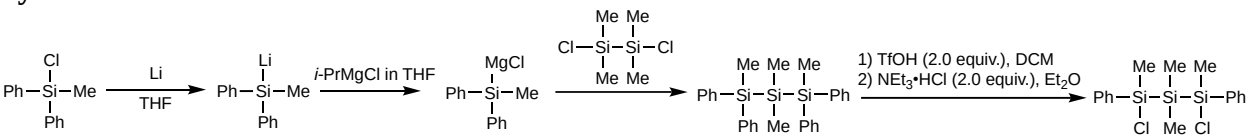


S7-1 synthesized according to GPB using diphenylhexamethyltrisilane at the scale of 30.4 mmol. The product was purified by vacuum distillation (0.05 Torr, 60 °C) yielding a clear colorless liquid (6.00 g, 80.4% yield). ^{1}H NMR (400 MHz, C6D6) δ 0.39 (s, 12H), 0.15 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 3.17, -7.51 **^{29}Si NMR** (99 MHz, C_6D_6) δ 25.69, -43.86.



S5-2 synthesized according to GPB using diphenyloctamethyltetrasilane at the scale of 33.8 mmol. The product was purified by vacuum distillation (0.05 Torr, 120 °C) yielding a clear, colorless liquid (7.40 g, 72.1 % yield). ^{1}H NMR (400 MHz, C6D6) δ 0.40 (s, 12H), 0.20 (s, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 3.39, -6.18 **^{29}Si NMR** (99 MHz, C_6D_6) δ 27.16, -42.84.

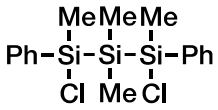
Synthesis of S6-1:[21]



The following substrates were synthesized through optimized procedures adapted from known literature.[21]

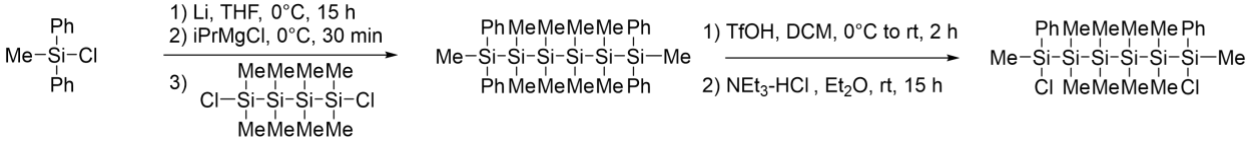
Synthesis of Tetraphenyltrisilane: An oven-dried 250 mL Schlenk flask outfitted with a stir bar was cooled under an Argon atmosphere. To the flask was added granular lithium (2.2 g, 320 mmol, 8.0 equiv.), which was suspended in tetrahydrofuran (80 mL). Chloromethyl(diphenyl)silane (18.9 mL, 80 mmol, 2.0 equiv.) was added to the lithium suspension dropwise via syringe. The clear, colorless solution was observed to darken to a dark red color within one hour after addition of chloromethyl(diphenyl)silane and the heterogeneous mixture was allowed to stir at room temperature overnight. Under argon, the reaction mixture was filtered through a fritted Schlenk tube into the receiving flask to remove excess liutium, and the dark red solution was cooled to 0 °C in an ice bath. Isopropylmagnesium chloride (2M in THF, 40.0 mL, 80 mmol, 2.0 equiv.) was added dropwise via syringe, and the solution was stirred an additional 30 minutes at 0 °C. Dichlorodimethylsilane (5.16 g, 40 mmol, 1.0 equiv.) was added dropwise. The solution was allowed to warm to room temperature with stirring overnight. The reaction was cooled in an ice bath and quenched by the careful, dropwise addition of saturated NH_4Cl aqueous solution (40 mL), followed by DI water (40 mL). The aqueous and organic layers were separated and the aqueous layer extracted twice with diethyl ether. The combined organic layers were dried with magnesium sulfate, filtered, and concentrated, yielding a white solid. The solid was subjected to automated column chromatography with hexanes to a gradient to 5% dichloromethane/95% hexanes, yielding a white solid (11.60 g, 78.5%).

Synthesis of Dichlorodiphenyltrisilane (S6-1): A 250 mL Schlenk flask equipped with a stir bar under argon was charged with **Tetraphenyltrisilane** (1.0 equiv., 20.0 mmol, 9.06 g) and DCM (40 mL). In an inert atmosphere glove box, trifluoromethanesulfonic acid (2.0 equiv., 40 mmol, 3.5 mL) was dispensed into a closed 25 mL addition funnel. The addition funnel was sealed and removed from the glove box. Under a high flow of argon, the addition funnel was attached to the Schlenk flask. The reaction mixture was cooled to 0 °C with an ice water bath and the trifluoromethanesulfonic acid was gradually added. 4 mL of DCM was added to the addition funnel to wash any residual trifluoromethanesulfonic acid into the reaction flask. After half an hour, the ice water bath was removed and the reaction mixture was stirred for 2 hours at room temperature. After 2 hours volatile materials were removed under vacuum, and the resulting yellow oil was dissolved in diethyl ether (48 mL). NEt_3-HCl (5.51 g 2.0 equiv., 40 mmol) was was quickly poured into the reaction mixture under a high flow of argon. The reaction mixture was stirred at room temperature overnight. Then the reaction mixture was cooled at -78°C. The bottom ionic layer was frozen and the top clear solution was transferred to a receiving flask through cannula under argon protection. The residual solids were washed 3 times by diethyl ether (10 mL) and all the fractions were combined in the receiving flask. Diethyl ether was removed under vacuum and the product was distilled at 150 °C under vacuum, yielding a a clear colorless liquid (7.27 g, 93.7%).



^{1}H NMR (400 MHz, C6D6) δ 7.49-7.55 (m, 2H), 7.43-7.47 (m, 2H).7.31-7.43 (m, 6H), 0.72 (s, 3H), 0.65 (s, 3H), 0.32-0.36 (d, *J*=7.9 Hz, 3H), 0.27-0.32 (d, *J*=7.9 Hz 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.93, 133.50, 133.47, 130.23, 130.19, 128.32, 128.29, 1.53, 1.49, -6.80. **^{29}Si NMR** (99 MHz, C_6D_6) δ 15.32, -42.65.

Synthesis of S6-2:[21]



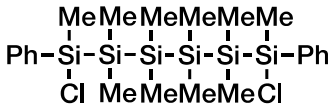
The following substrates were synthesized through optimized procedures adapted from known literature.[21]

Synthesis of Tetraphenylhexasilane: An oven-dried 250 mL Schlenk flask outfitted with a stir bar was cooled under an argon atmosphere. To the flask was added granular lithium (1.06 g, 153 mmol, 8.2 equiv.), which was suspended in tetrahydrofuran (38 mL). Chloromethyl(diphenyl)silane (8.5 mL, 40.4 mmol, 2.2 equiv.) was added to the lithium suspension dropwise via syringe. The clear, colorless solution was observed to darken to a dark red color within one hour after addition of chloromethyl(diphenyl)silane and the heterogeneous mixture was allowed to stir at room temperature overnight. Under argon, the reaction mixture was filtered through a fritted Schlenk tube into the receiving flask to remove excess lithium, and the dark red solution was cooled to 0 °C in an ice bath. Isopropylmagnesium chloride (2M in THF, 19.1 mL, 38.2 mmol, 2.0 equiv.) was added dropwise via syringe, and the solution was stirred an additional 30 minutes at 0 °C. **S5-2** (5.67 g, 18.7 mmol, 1.0 equiv.) was added dropwise. The solution was allowed to warm to room temperature with stirring overnight. The reaction was cooled in an ice bath and quenched by the careful, dropwise addition of saturated NH_4Cl aqueous solution (20 mL), followed by DI water (20 mL). The aqueous and organic layers were separated and the aqueous layer extracted twice with diethyl ether. The combined organic layers were dried with magnesium sulfate, filtered, and concentrated, yielding a white solid. The solid was subjected to automated column chromatography with hexanes to a gradient to 5% dichloromethane/95% hexanes, yielding a white solid (4.62 g, 38%).

Synthesis of Dichlorodiphenyltetrasilane (S6-2): A 250 mL Schlenk flask equipped with a stir bar under argon was charged with dichlorooctonethyltetrasilane **S5-2** (4.62 g, 7.37 mmol, 1.0 equiv.) and DCM (45 mL). In an inert atmosphere glove box, trifluoromethanesulfonic acid (1.3 mL, 40 mmol, 14.7 equiv.) was dispensed into a closed 10 mL addition funnel. The addition funnel was sealed and removed from the glove box. Under a high flow of argon, the addition funnel was attached to the Schlenk flask. The reaction mixture was cooled to 0 °C with an ice water bath and the trifluoromethanesulfonic acid was gradually added. 5 mL of DCM was added to the addition funnel to wash any residual trifluoromethanesulfonic acid into the reaction flask. After half an hour, the ice water bath was removed, and the reaction mixture was stirred for 2 hours at room temperature. After 2 hours volatile materials were removed under vacuum, and the resulting yellow oil was dissolved in diethyl ether (50 mL). $NEt_3{\cdot}HCl$ (2.15 g, 15.6 mmol, 2.1 equiv.) was quickly poured into the reaction

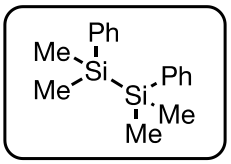
mixture. The reaction mixture was stirred for 15 hours. The $NEt_3 \cdot HCl$ is slow to dissolve, but a homogenous mixture was eventually observed. After 15 hours of stirring, the reaction mixture was cooled to -78 °C using an acetone/dry ice bath to solidify the bottom layer of ionic liquid. The top layer was transferred to a dry 100 mL Schlenk flask. Diethyl ether was removed under vacuum to produce **S6-2** as a white solid (2.00 g, 3.69 mmol, 50% yield).

Note: When the biphasic solution is cooled to at -78 °C for too long, the product in the top layer crashes out of solution. When this occurred, the solution was warmed to rt, then cooled again to -78 °C, long enough to solidify ionic byproduct, but not long enough to solidify the product. Care was taken to transfer the top layer quickly to collect product.

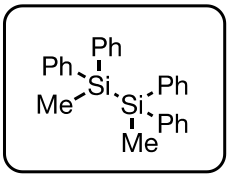


^{1}H NMR (400 MHz, C6D6) δ 7.52-7.59 (m, 4H), 7.35-7.42 (m, 6H), 0.75 (s, 6H), 0.23 (s, 12H), 0.13 (s, 3H), 0.10 (s, 3H), 0.08 (s, 3H), 0.07 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.07, 133.44, 129.96, 128.24, 77.48, 77.16, 76.84, 1.98, -4.52, -4.57, -5.35, -5.38. **^{29}Si NMR** (99 MHz, C_6D_6) δ 16.95, -39.40, -40.35.

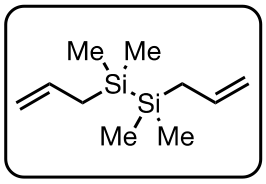
9. Synthesis and characterization of products



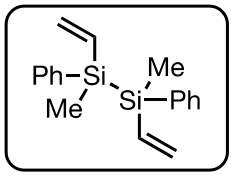
A1 Following general procedure 1 starting from chlorodimethylphenylsilane (1mmol). The crude mixture was purified with 0 to 5% Et_2O / Hexanes to afford a white solid (116mg, 86% isolated yield).
^{1}H NMR (500 MHz, CDCl3) δ 7.36 - 7.43 (m, 2H), 7.28 - 7.35 (m, 3H), 0.30 - 0.37 (s, 6H). **^{13}C NMR** (126 MHz, CDCl3) δ 133.82, 128.68, 123.23, 122.51, -9.11. **^{29}Si NMR** (99 MHz, CDCl3) δ -27.02.
HRMS: Calcd. for $C_{16}H_{22}Si_2^+$ $[M]^+$ m/z: 270.1260, found m/z: 270.1255. **GC-MS:** 270.1 (**Method-HT**, retention time: 9.692).



A2 Following general procedure 1 starting from chloromethyldiphenylsilane (1mmol). The crude mixture was purified with 0 to 5% Et_2O / Hexanes to afford a white solid (171mg, 87% isolated yield).
^{1}H NMR (500 MHz, CDCl3) δ 7.44-7.46 (m, 8H), 7.38-7.40 (m, 4H), 7.34-7.35 (m, 8H), 0.74 (s, 6H). **^{13}C NMR** (126 MHz, CDCl3) δ 136.62, 135.26, 128.99, 127.88, -4.02. **^{29}Si NMR** (99 MHz, CDCl3) δ -23.25.

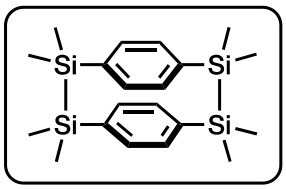


A3 Following general procedure 1 starting from chlorodimethylvinylsilane (1mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (23mg, 23% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.70-5.92 (ddt, *J* = 16.4, 9.8, 8.1 Hz, 2H), 4.78-4.94 (m, 4H), 1.59-1.70 (dt, *J* = 8.1, 1.2 Hz, 4H), 0.02-0.14 (s, 12H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ. 135.24, 112.72, 22.85, -4.23. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -18.25.
HRMS: Calcd. for $C_{10}H_{22}Si_2^+$ $[M\text{-Allyl}]^+$ m/z: 157.0869, found m/z: 157.0862. **GC-MS:** 198.1 (**Method-LT**, retention time: 2.720).

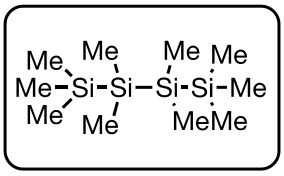


A4 Following general procedure 1 starting from chlorovinylmethylphenylsilane (1mmol). The crude mixture was purified with 0 to 5% Et_2O / Hexanes to afford a white solid (105mg, 72% isolated yield, dr 1:1 based on Si NMR).

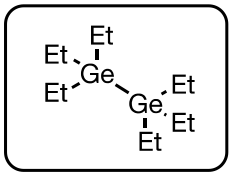
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30-7.52 (m, 5H), 6.32-6.48 (m, 1H), 6.07-6.19 (m, 1H), 5.71-5.84 (m, 1H), 0.47-0.51(s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ136.65, 136.64, 135.92, 135.89, 134.61, 133.66, 128.77, 127.80, 5.61, 5.57. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -27.07, -27.02.
HRMS: Calcd. for $C_{18}H_{22}Si_2^+$ $[M\text{-}Me]^+$ m/z: 279.1025, found m/z: 279.1021. **GC-MS:** 294.2 (**Method-LT**, retention time: 6.644).



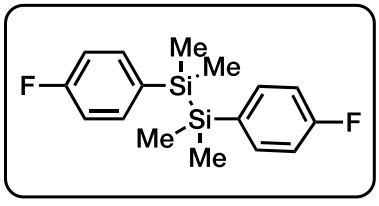
A5 Following general procedure 1 starting from 1,4-bis(chlorodimethylsilyl)benzene (1mmol). The crude mixture was purified with 0 to 5% Et_2O / Hexanes to afford a white solid (30% NMR yield). The product is partially inseparatable from the oxidized form.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (s, 8H), 6.32-6.48 (m, 1H), 0.38 (s, 24H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ140.87, 132.27, 0.94. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -1.26.
HRMS: Calcd. for $C_{20}H_{32}Si_4^+$ $[M]^+$ m/z: 384.1581, found m/z: 384.1574. **GC-MS:** 384.2 (**Method-LT**, retention time: 8.026). 400.2 shows as a minor peak (**Method-LT**, retention time: 7.570).



A6 Following general procedure 1 starting from chlorotriethyllgermane (1mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (102mg, 78% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 0.12-0.14 (s, 12H), 0.10-0.12 (s, 18H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ-1.3, -5.95. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.31, -45.04.
HRMS: Calcd. for $C_{10}H_{30}Si_4^+$ $[M]^+$ m/z: 262.1425, found m/z: 262.1420. **GC-MS:** 262.2 (**Method-LT**, retention time: 3.892).

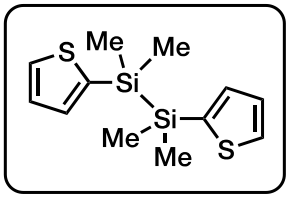


A7 Following general procedure 1 starting from chlorotriethyllgermane (1mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (128mg, 80% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 1.04-1.12 (t, J = 7.9 Hz 18H), 0.85-0.93 (q, J = 7.8 Hz, 12H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 10.06, 5.44.
HRMS: Calcd. for $C_{12}H_{30}Ge_2^+$ $[M]^+$ m/z: 322.0771, found m/z: 322.0764. **GC-MS:** 322.1 (**Method-LT**, retention time: 4.917).

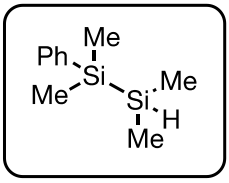


A8 Following general procedure 1 starting from chloro(4-fluorophenyl)dimethylsilane (1mmol) with slight modification: **10 mA, 2 F/mol** instead of 20 mA, 2.5 F/mol. The crude mixture was purified with pure hexanes to afford a colorless oil (116mg, 76% isolated yield).

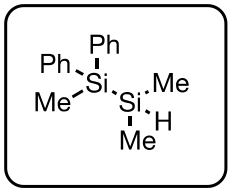
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31- 7.34 (m, 4H), 7.01-7.04 (m, 4H), 0.33 (s, 12H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.44, 162.47, 135.59, 135.53, 115.00, 114.85, -3.87. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -21.73.
HRMS: Calcd. for $C_{16}H_{20}F_2Si_2^+$ $[M]^+$ m/z: 306.1072, found m/z: 306.1060. **GC-MS:** 306.1 (**Method-LT**, retention time: 5.641).



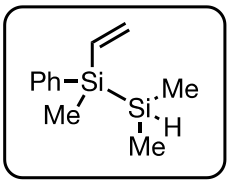
A9. Following general procedure 1 starting from chlorodimethyl(thiophen-2-yl)silane (1mmol) with slight modification: **10 mA, 2 F/mol** instead of 20 mA, 2.5 F/mol. The crude mixture was purified with pure hexanes to afford a colorless oil (91mg, 65% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62-7.63 (m, 2H), 7.20-7.21 (m,4H), 0.44 (s, 12H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 137.70, 134.50, 130.70, 128.27, -2.67. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -24.47
HRMS: Calcd. for $C_{12}H_{18}S_2Si_2^+$ $[M]^+$ m/z: 282.0388, found m/z: 282.0385. **GC-MS:** 282.1 (**Method-LT**, retention time: 5.682).



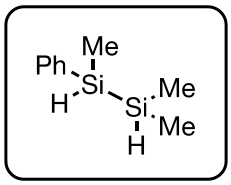
B1. Following general procedure 2 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (128mg, 66 % isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.52 (m, 2H), 7.32-7.38 (m, 3H), 3.69-3.78 (hept, *J* = 4.5 Hz, 1H), 0.37-0.40 (s, 6H), 0.12-0.16 (d, *J* = 4.5 Hz, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.13, 133.82, 128.54, 127.82, -3.54, -6.62. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -20.79, -39.13.
HRMS: Calcd. for $C_{10}H_{18}Si_2^+$ $[M]^+$ m/z: 194.0947, found m/z: 194.0942 **GC-MS:** 194.1 (**Method-LT**, retention time: 3.642).



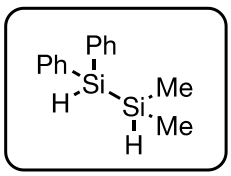
B2. Following general procedure 2 starting from chloromethyldiphenylsilane (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (153mg, 60% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ7.54 (m, 4H), 7.38 (m, 6H), 4.00 (hept, *J* = 4.5 Hz, 1H), 0.67 (s, 3H), 0.23 (d, *J* = 4.5 Hz, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 136.97, 134.86, 128.93, 127.95, -4.59, -6.29. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -21.33, -39.82.
HRMS: Calcd. for $C_{15}H_{20}Si_2^+$ $[M]^+$ m/z: 256.1104, found m/z: 256.1098 **GC-MS:** 256.1 ((**Method-LT**, retention time: 5.917).



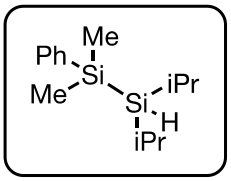
B3. Following general procedure 2 starting from chlorovinylmethylphenylsilane (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (123mg, 60% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51-7.55(m, 2H), 7.36-7.40 (m, 3H), 6.31-6.46 (dd, 1H), 6.08-6.19 (m, *J* = 14.5, 3.5 Hz, 1H), 5.74-5.87 (dd, 1H), 3.83-3.88 (hept, *J* = 4.5 Hz, 1H), 0.47-0.50 (s, 3H), 0.17-0.22 (t, *J* = 4.5 Hz, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 137.03, 136.24, 134.40, 133.43, 128.79, -5.50, -6.49, -6.52. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -25.16, -39.95.
HRMS: Calcd. for $C_{11}H_{18}Si_2^+$ [M-Me]$^+$ m/z: 191.0712 found m/z: 191.0706. **GC-MS**: 206.1 (**Method-LT**, retention time: 4.017).



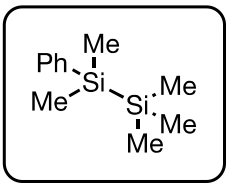
B4. Following general procedure 2 starting from chloromethylphenylsilane (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (75mg, 42% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50-7.57 (m, 2H), 7.33-7.40 (m, 3H), 4.28-4.35 (qd, *J* = 4.7, 2.2 Hz, 1H), 3.82-3.90 (dq, *J* = 4.4, 2.2 Hz, 1H), 0.45-0.50 (d, *J* = 4.6 Hz, 3H), 0.20-0.22 (d, *J* = 4.5 Hz, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ.135.40, 134.76, 128.85, 127.95, -6.30, -6.32, -7.65 **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -35.88, -39.33.
HRMS: Calcd. for $C_9H_{16}Si_2^+$ [M-H]$^+$ m/z: 179.0712 found m/z: 179.0706 **GC-MS**: 180.1 (**Method-LT**, retention time: 3.411).



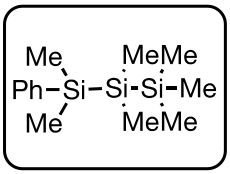
B5. Following general procedure 2 starting from chlorodiphenylsilane (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (176mg, 73% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.62 (m, 4H), 7.37-7.43 (m, 6H), 4.90-4.94 (d, *J* = 2.5 Hz, 1H), 4.06-4.13 (heptd, *J* = 4.5, 2.5 Hz, 1H), 0.27-0.31 (d, *J* = 4.5 Hz, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ. 135.76, 133.51, 129.22, 128.12, -5.98. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -31.82, -40.16.
HRMS: Calcd. for $C_{14}H_{18}Si_2^+$ [M]$^+$ m/z: 242.0947 found m/z: 242.0944 **GC-MS**: 242.1 (**Method-LT**, retention time: 5.739).



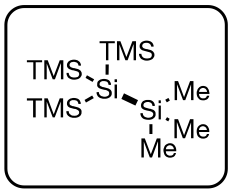
B6. Following general procedure 2 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chlorodiisopropylsilane (Chlorosilane B) (1.25mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (180mg, 72% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52-7.57 (m, 2H), 7.34-7.38 (m, 3H), 3.43-3.50 (t, *J* = 3.0 Hz, 1H), 1.14-1.21 (qd, *J* = 7.2, 2.9 Hz, 2H), 1.02-1.06 (d, *J* = 7.0 Hz,12H), 0.46-0.50 (s, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 140.13, 133.95, 128.42, 127.76, 20.44, 11.06, -1.22. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -21.63.
HRMS: Calcd. for $C_{14}H_{26}Si_2^+$ $[M]^+$ m/z: 250.1573 found m/z: 250.1568 **GC-MS**: 250.2 (**Method-LT**, retention time: 5.236).



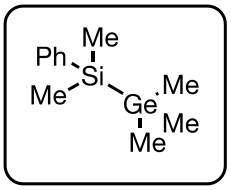
B7. Following general procedure 3 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chlorotrimethylsilane (Chlorosilane B) (1.5mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (160mg, 77% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46-7.51 (m, 2H), 7.33-7.40 (m, 3H), 0.34-0.39 (s, 6H), 0.06-0.12 (s, 9H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ137.20, 131.29, 125.82, 125.25, -4.74, -6.49. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -21.86, -24.14.
HRMS: Calcd. for $C_{11}H_{20}Si_2^+$ $[M]^+$ m/z: 208.1104 found m/z: 208.1098 **GC-MS**: 208.1 (**Method-LT**, retention time: 3.799).



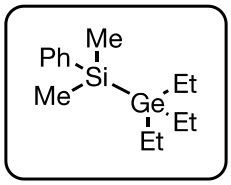
B8. Following general procedure 3 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chloropentamethyldisilane (Chlorosilane B) (1.5mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (199mg, 75% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.51 (m, 2H), 7.32-7.40 (m, 3H), 0.39-0.41 (s, 6H), 0.09-0.12 (s, 6H), 0.02-0.05 (s, 9H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.98, 133.72, 128.30, 127.70, -1.58, -3.13, -6.82. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.84, -18.6, -48.4.
HRMS: Calcd. for $C_{13}H_{26}Si_3^+$ $[M]^+$ m/z: 266.1342 found m/z: 266.1337 **GC-MS**: 266.2 (**Method-LT**, retention time: 4.978).



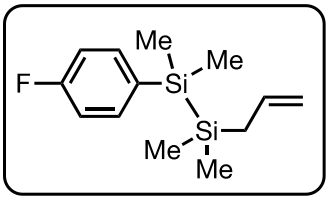
B9. Following general procedure 3 starting from chlorotri(trimethyl)silane (Chlorosilane A) (1mmol) and chlorotrimethylsilane (Chlorosilane B) (1.5mmol). The crude mixture was purified with pure hexanes to afford a white solid (320mg, quantitative isolated yield)
^{1}H NMR (500 MHz, $CDCl_3$) δ 0.23 (s, 36H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 2.67. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -9.85, -135.38.
HRMS: Calcd. for $C_{12}H_{36}Si_5^+$ $[M]^+$ m/z: 320.1663 found m/z: 320.1665 **GC-MS**: 320.2 (**Method-LT**, retention time: 5.144).



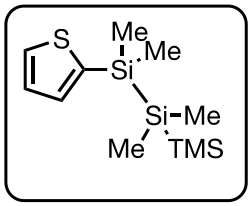
B10. Following general procedure 3 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chlorotrimethylgermane (Chlorosilane B) (1.5mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (157mg, 62% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.51 (m, 2H), 7.33-7.40 (m, 3H), 0.39-0.44 (s, 6H), 0.15-0.21 (s, 9H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.26, 133.65, 128.54, 127.79, -3.08, -3.38. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -14.84.
HRMS: Calcd. for $C_{11}H_{20}SiGe^+$ $[M]^+$ m/z: 254.0546 found m/z: 254.0540 **GC-MS**: 254.1 (**Method-LT**, retention time: 4.057).



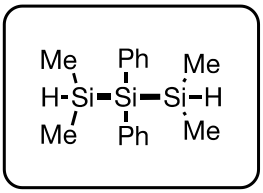
B11. Following general procedure 3 starting from chlorodimethylphenylsilane (Chlorosilane A) (1mmol) and chlorotriethylgermane (Chlorosilane B) (1.5mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (130mg, 44% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44-7.50 (m, 2H), 7.29-7.37 (m, 3H), 0.97-1.02 (t, *J*=7.9 Hz, 9H), 0.77-0.83 (q, *J*=7.9 Hz, 6H), 0.41-0.46 (s, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 140.11, 133.65, 128.39, 127.75, 10.03, 4.00, -1.90. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.19.
HRMS: Calcd. for $C_{14}H_{26}SiGe^+$ $[M]^+$ m/z: 296.1016 found m/z: 296.1008 **GC-MS**: 296.1(**Method-LT**, retention time: 5.491).



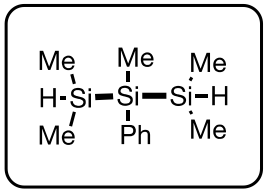
B12. Following general procedure 3 starting from chloro(4-fluorophenyl)dimethylsilane (Chlorosilane A) (0.5 mmol) and chlorodimethylallylsilane (Chlorosilane B) (0.75 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (44mg, 35% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42-7.46 (m, 2H), 7.05-7.08 (m, 2H), 5.72-5.76 (m, 1H), 4.81-4.85 (m, 2H), 1.56-1.60 (m, 2H), 0.37 (s, 6H), 0.06 (s, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.38, 162.42, 135.54, 135.48, 134.97, 115.02, 114.87, 112.91, 22.48, -3.47, -4.51. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -18.66, -21.14.
HRMS: Calcd. for $C_{13}H_{21}Si_2F^+$ $[M\text{-}Me]^+$ m/z: 237.0931 found m/z: 237.0925 **GC-MS**: 252.1 (**Method-LT**, retention time: 4.362).



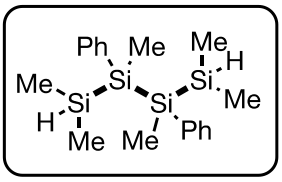
B13. Following general procedure 3 starting from chlorodimethyl(thiophen-2-yl)silane (Chlorosilane A) (0.5 mmol) and chloropentamethyldisilane (Chlorosilane B) (0.75 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (91mg, 57% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.61 (m, 1H), 7.20-7.23 (m, 2H), 0.43 (s, 6H), 0.13 (s, 6H), 0.06 (s, 9H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.33, 133.92, 130.29, 128.15, -1.59, -1.73, -6.93. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.90, -20.90, -48.24.
HRMS: Calcd. for $C_{11}H_{24}Si_3S^+$ $[M]^+$ m/z: 272.0907 found m/z: 272.0902 **GC-MS**: 272.1 (**Method-LT**, retention time: 4.615).



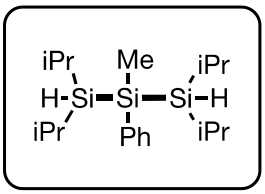
C1. Following general procedure 4 starting from dichlorodiphenylsilane (Chlorosilane A) (1mmol) and chlorotrimethylsilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (156mg, 52% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46-7.57 (m, 4H), 7.32-7.43 (m, 6H), 4.08-4.22 (hept, *J* = 4.5 Hz, 2H), 0.22-0.33 (d, *J* = 4.65Hz, 12H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ135.98, 128.60, 128.07, -5.52. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -38.13, -39.65.
HRMS: Calcd. for $C_{16}H_{24}Si_3^+$ $[M]^+$ m/z: 300.1186 found m/z: 300.1172 **GC-MS**: 300.1 (**Method-LT**, retention time: 6.521).



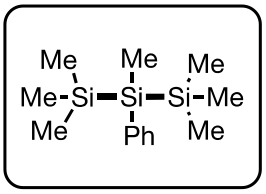
C2. Following general procedure 4 starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and chlorodiisopropylsilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (116mg, 49% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44-7.51(m, 2H), 7.31-7.39 (m, 3H), 3.87-3.97 (sept, *J* = 4.4 Hz, 2H), 0.46-0.51 (s, 3H), 0.23-0.25 (d, *J* = 4.4 Hz, 6H), 0.19-0.22(d, *J* = 4.4 Hz, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ136.60, 134.68, 128.18, 127.89, -5.83, -5.86, -8.38. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -37.06, -45.30.
HRMS: Calcd. for $C_{11}H_{22}Si_3^+$ $[M-H]^+$ m/z: 237.0951 found m/z: 237.0941 **GC-MS**: 238.1(**Method-LT**, retention time: 4.588).



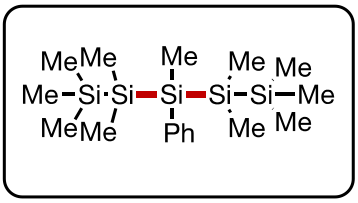
C2'. Isolated as side product for c2 on **4mmol** scale. The crude mixture was purified with pure hexanes to afford a colorless oil (143mg, 10% isolated yield, dr 1:1.3 by Si NMR).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.46 (m, 4H), 7.25-7.33 (m, 6H), 3.88-3.96 (m, 2H), 0.50 (s, 6H), 0.05-0.13 (m, 12H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 136.69, 136.65, 134.83, 134.73, 128.18, 127.80, 127.74, -5.68, -5.75, -5.77, -5.79, -7.50, -7.65. **^{29}Si NMR** (99 MHz, $CDCl_3$) -43.19, -43.30
HRMS: Calcd. for $C_{18}H_{30}Si_4^+$ $[M-H]^+$ m/z: 358.1425 **GC-MS**: 358.1.



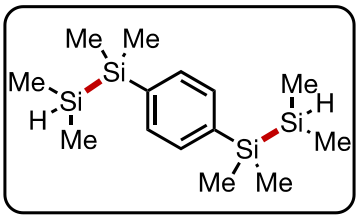
C3. Following general procedure 4 starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and chlorodiisopropylsilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (98mg, 28% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52-7.58 (m, 2H), 7.29-7.34 (m, 3H), 3.66-3.71 (t, *J* = 2.7 Hz, 2H), 1.18-1.31 (m, 4H), 1.11-1.13 (d, *J* = 7.3 Hz, 6H), 1.07-1.10 (d, *J* = 7.3 Hz, 6H), 1.03-1.05 (d, *J* = 7.3 Hz, 6H),0.97-1.01(d, *J* = 7.3 Hz, 6H), 0.68 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 137.68, 134.95, 127.90, 127.71, 21.01, 20.98, 20.53, 11.83, 11.75, -4.91. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -10.16, -47.84.
HRMS: Calcd. for $C_{19}H_{38}Si_3^+$ $[M\text{-iPr}]^+$ m/z: 307.1734 found m/z:307.1723 **GC-MS**: 350.3(**Method-LT**, retention time: 7.095).



C4. Following general procedure 4 starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and chlorotrimethylsilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (175mg, 46% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39-7.44 (m, 2H), 7.29-7.34 (m, 3H), 0.40 (s, 3H), 0.14 (s, 18H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 137.70, 134.48, 127.69, 127.68, -1.08, -9.02. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.89, -46.27.
HRMS: Calcd. for $C_{13}H_{26}Si_3^+$ $[M]^+$ m/z: 266.1342 **GC-MS:** 266.2 (**Method-LT**, retention time: 4.879).



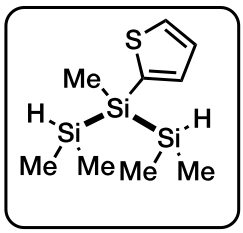
C5. Following general procedure 4 starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and chloropentamethyldisilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (176mg, 46% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38-7.44 (m, 2H), 7.25-7.32 (m, 3H), 0.48 (s, 3H), 0.24 (s, 12H), -0.1 (s, 18H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ138.45, 134.35, 127.73, 127.60, -1.50, -4.92, -5.23, -7.04. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -14.81, -41.12, -43.15.
HRMS: Calcd. for $C_{17}H_{38}Si_5^+$ $[M]^+$ m/z: 382.1820 found m/z: 382.1809 **GC-MS**: 382.3 (**Method-LT**, retention time: 6.781).



C6. Following general procedure 4 starting from 1,4-bis(chlorodimethylsilyl)benzene (Chlorosilane A) (1mmol) and chlorodimethylsilane (Chlorosilane B) (4 mmol). The crude mixture was purified with pure hexanes to afford a colorless oil (155mg, 50% isolated yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45(s, 4H), 3.69-3.77 (sept, *J* = 4.5 Hz, 2H), 0.36 (s, 12H), 0.13 (d, *J* = 4.5 Hz, 12H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ139.37, 133.18, -3.59, -6.58. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -20.96, -39.12.

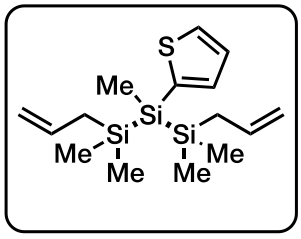
HRMS: Calcd. for $C_{14}H_{30}Si_4^+$ $[M]^+$ m/z: 310.1425 found m/z: 310.1417 **GC-MS**: 310.2 (**Method-LT**, retention time: 5.698).



C7. Following general procedure 4 starting from dichloro(methyl)(thiophen-2-yl)silane (Chlorosilane A) (0.5 mmol) and chlorodimethylsilane (Chlorosilane B) (2 mmol) with slight modification: **10 mA** instead of 20 mA. The crude mixture was purified with pure hexanes to afford a colorless oil (48mg, 39% isolated yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (m, 1H), 7.22 (m, 2H), 3.93 (hept, *J* = 4.5 Hz, 2H), 0.51 (s, 3H), 0.24-0.25 (d, *J* = 4.5 Hz, 6H), 0.21-0.23 (d, *J* = 4.5 Hz, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.06, 134.93, 130.73, 128.36, -5.93, -5.95, -6.71. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -36.43, -40.73.

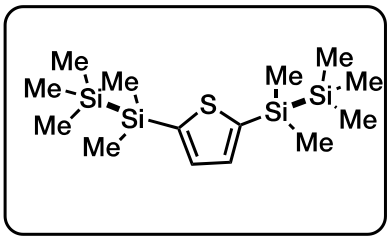
HRMS: Calcd. for $C_9H_{20}SSi_3^+$ $[M]^+$ m/z: 244.0594 found m/z: 244.0589 **GC-MS**: 244.1(**Method-LT**, retention time: 4.288).



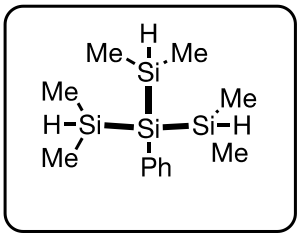
C8. Following general procedure 4 starting from dichloro(methyl)(thiophen-2-yl)silane (Chlorosilane A) (0.5 mmol) and chlorodimethyallyllsilane (Chlorosilane B) (2 mmol) with slight modification: **10 mA** instead of 20 mA. The crude mixture was purified with pure hexanes to afford a colorless oil (94mg, 58% isolated yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60-7.61 (m, 1H), 7.17-7.20 (m, 2H), 5.77-5.84 (m, 1H), 4.83-4.86 (m, 4H), 1.66-1.67 (d, *J* = 8.1 Hz, 4H), 0.50 (s, 3H), 0.16 -0.17(d, *J* = 5.5 Hz, 12H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.40, 134.67, 134.58, 130.36, 128.29, 113.18, 22.99, -3.56, -6.85. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -15.45, -49.11.

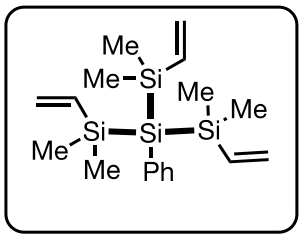
HRMS: Calcd. for $C_{15}H_{28}SSi_3^+$ $[M]^+$ m/z: 283.0828 found m/z: 283.0823 **GC-MS**: 324.2 (**Method-LT**, retention time: 5.911).



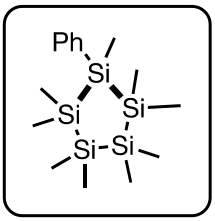
C9. Following general procedure 4 starting from 2,5-bis(chlorodimethylsilyl)thiophene (Chlorosilane A) (0.5 mmol) and chlorodtrimethylsilane (Chlorosilane B) (2 mmol) with slight modification: **10 mA** instead of 20 mA. The crude mixture was purified with pure hexanes to afford a colorless oil (144mg, 42% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 (s, 2H), 0.36 (s, 12H), 0.08 (s, 18H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 144.64, 135.06, -2.40, -2.58. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -19.32, -24.65.
HRMS: Calcd. for $C_{14}H_{32}SSi_4^+$ $[M]^+$ m/z: 344.1302 found m/z: 344.1296 **GC-MS**: 344.1(**Method-LT**, retention time: 5.327).



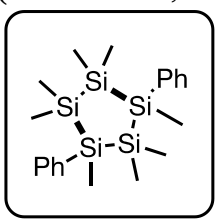
C10. Following general procedure 5 starting from trichlorophenylsilane (Chlorosilane A) (1 mmol) and chlorodimethylsilane (Chlorosilane B) (6 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (68mg, 24% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43-7.52 (m, 2H), 4.08-4.17 (sept, *J* = 4.4 Hz, 3H), 0.28-0.32 (d, *J* = 4.4 Hz, 18H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 136.53, 133.53, 128.00, 127.93, -4.33. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -35.62, -78.42.
HRMS: Calcd. for $C_{12}H_{26}Si_4^+$ $[M-H]^+$ m/z: 281.1033 found m/z: 281.1029 **GC-MS:** 282.1(**Method-HT**, retention time: 5.394).



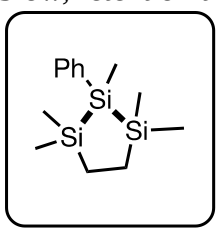
C11. Following general procedure 5 starting from trichlorophenylsilane (Chlorosilane A) (1 mmol) and chlorovinyldimethylsilane (Chlorosilane B) (6 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (82mg, 23% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.53 (m, 2H), 7.22-7.32 (m, 3H), 6.25-6.38 (m, 3H), 5.93-6.03 (dd, *J* = 14.5, 3.5 Hz, 3H), 5.61-5.5.73 (m, 3H), 0.30 (s, 18H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 140.20, 136.78, 134.52, 130.96, 127.65, 127.55, -1.30. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -18.83, -78.25.
HRMS: Calcd. for $C_{18}H_{32}Si_4^+$ $[M-Me]^+$ m/z: 345.1346 found m/z: 345.1335 **GC-MS**: 360.1 (**Method-HT**, retention time: 6.653).



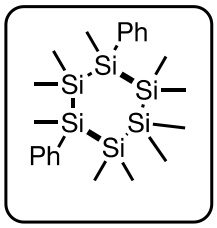
Following general procedure 6 starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and 1,4-dichloro-1,1,2,2,3,3,4,4-octamethyltetrasilane (Chlorosilane B) (1.1 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (104mg, 30% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40-7.44 (m, 2H), 7.30-7.37 (m, 2H), 0.41-0.45 (s, 3H), 0.20-0.24 (m, 18H), 0.16-0.19 (m, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ138.09, 134.94, 127.80, 127.70, -5.39, -6.07, -6.36, -6.38, -7.30. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -39.84, -41.50, -42.73.
HRMS: Calcd. for $C_{15}H_{32}Si_5^+$ $[M\text{-}Me]^+$ m/z: 352.1350 found m/z: 352.1342 **GC-MS**: 352.2 (**Method-HT**, retention time: 4.993).



Following general procedure 6 starting from 1,3-dichloro-1,2,2,3-tetramethyl-1,3-diphenyltrisilane (Chlorosilane A) (1mmol) and1,2-dichloro-1,1,2,2-tetramethyldisilane (Chlorosilane B) (1.1 mmol). The crude mixture was purified with pure Hexanes to afford a white solid (182mg, 44% isolated yield, dr 1:1 based on Si NMR).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.47 (m, 4H), 7.31-7.38 (m, 6H), 0.46-0.52 (m, 6H),0.26-0.29(m, 6H), 0.17-0.25 (m, 12H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ137.70, 137.59, 135.03, 135.01, 127.92, 127.76, -4.44, -5.15, -5.21, -5.30, -6.04, -6.39, -6.42, -6.91, -7.18. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -40.15, -40.39, -40.49, -41.21, -41.69, -41.96.
HRMS: Calcd. for $C_{20}H_{34}Si_5^+$ $[M]^+$ m/z: 414.1507 found m/z: 414.1501 **GC-MS**: 414.2 (**Method-Slow**, retention time: 14.458, 14.386).



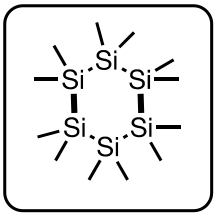
Following general procedure **6** starting from dichloromethylphenylsilane (Chlorosilane A) (1mmol) and (2,3-dimethylbutane-2,3-diyl)bis(chlorodimethylsilane) (Chlorosilane B) (1.1 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (110mg, 42% isolated yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.46 (m, 2H), 7.27-7.36 (m, 3H), 0.82-0.93 (m, 4H), 0.47 (s, 3H), 0.16 (s, 6H), 0.12 (s, 6H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ136.91, 134.95, 127.71, 127.69, 12.76, -3.16, -3.46, -9.38. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -7.03, -51.66.
GC-MS: Calcd. for $C_{13}H_{24}Si_3^+$ $[M]^+$ m/z: 264.1186 found m/z: 264.1 (**Method-HT**, retention time: 7.018)



Following general procedure 6 starting from 1,3-dichloro-1,2,2,3-tetramethyl-1,3-diphenyltrisilane (Chlorosilane A) (1mmol) and 1,3-dichloro-1,1,2,2,3,3-hexamethyltrisilane (Chlorosilane B) (1.1 mmol). The crude mixture was purified with pure Hexanes to afford a colorless oil (141mg, 30% isolated yield, dr 2:1 based on ^{1}H NMR).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43-7.50 (m, 4H), 7.30-7.38 (m, 6H), 0.51-0.54 (s, 3H), 0.41-0.44 (s, 3H), 0.27-0.32 (m, 9H), 0.18-0.25 (m, 15H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ137.77, 137.64, 135.03, 134.83, 128.00, 127.93, 127.74, 127.70, -3.85, -4.37, -4.79, -4.91, -5.10, -5.69, -5.79, -6.36, -6.38, -6.46, -6.73, -6.98. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ-39.91, -40.89, -41.11, -41.29, -41.49, -41.70, -42.10, -42.36.

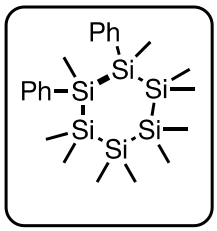
HRMS: Calcd. for $C_{22}H_{40}Si_6^+$ $[M]^+$ m/z: 472.1746 found m/z: 472.1743 **GC-MS**: 472.1 (**Method-HT**, retention time: 8.508).



Following general procedure 6 starting from 1,3-dichloro-1,1,2,2,3,3-hexamethyltrisilane (Chlorosilane A) (1mmol). The crude mixture was purified with pure hexanes to afford a colorless crystal (132mg, 38% isolated yield). The spectra are consistent with literature data.[27]

^{1}H NMR (500 MHz, $CDCl_3$) δ 0.16 (s, 36H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ -6.12. **^{29}Si NMR** (99 MHz, $CDCl_3$) δ -42.18.

GC-MS: Calcd. for $C_{12}H_{36}Si_6^+$ $[M]^+$ m/z: 348.1433 found m/z: 348.2 (**Method-HT**, retention time: 3.715).

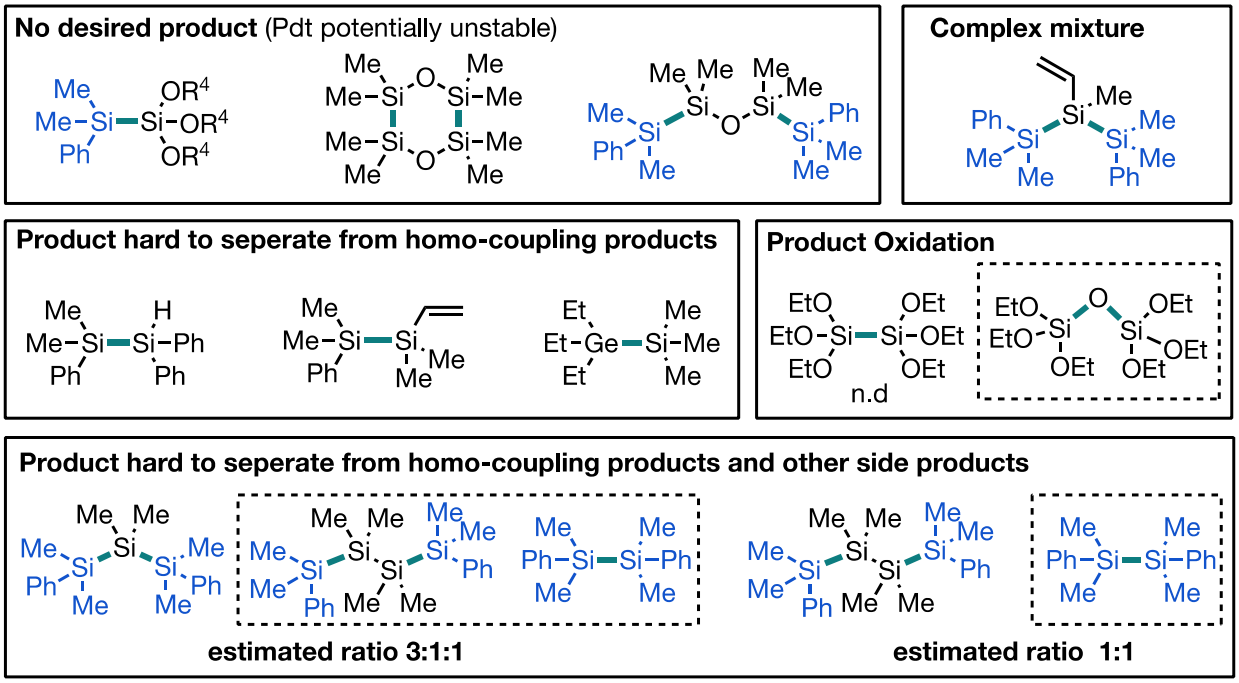


Following general procedure 6 starting from 1,6-dichloro-1,2,2,3,3,4,4,5,5,6-decamethyl-1,6-diphenylhexasilane (Chlorosilane A) (1mmol). The crude mixture was purified with pure Hexanes to afford a white solid (202mg, 43% isolated yield, dr 1:1 by ^{1}H NMR). The spectra are consistent with literature data.[21]

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.36 (m, 2H), 7.24-7.28 (m, 6H), 7.20-7.24 (m, 2H), 0.52 (s, 3H), 0.48 (s, 3H), 0.28 (s, 3H), 0.25 (s, 3H), 0.24 (s, 3H), 0.23 (s, 3H), 0.22 (s, 3H), 0.18 (s, 3H), 0.15 (s, 3H) **^{13}C NMR** (126 MHz, $CDCl_3$) δ 137.48, 137.24, 135.24, 134.80, 128.01, 127.91, 127.64, 127.51, -4.36, -5.10, -5.24, -5.54, -5.56, -5.76, -5.93, -6.55, -6.78, -6.82.**^{29}Si NMR** (99 MHz, $CDCl_3$) δ -40.06, -40.96, -41.28, -41.76, -41.86, -42.07.

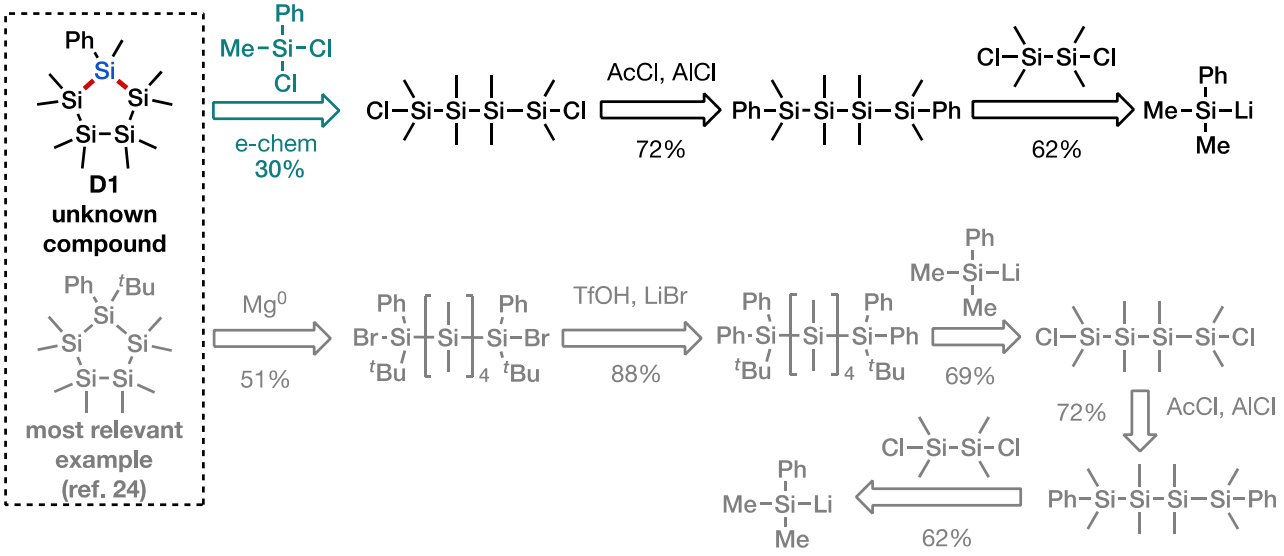
GC-MS: Calcd. for $C_{22}H_{40}Si_6^+$ $[M]^+$ m/z: 472.1746 found m/z: 472.1.

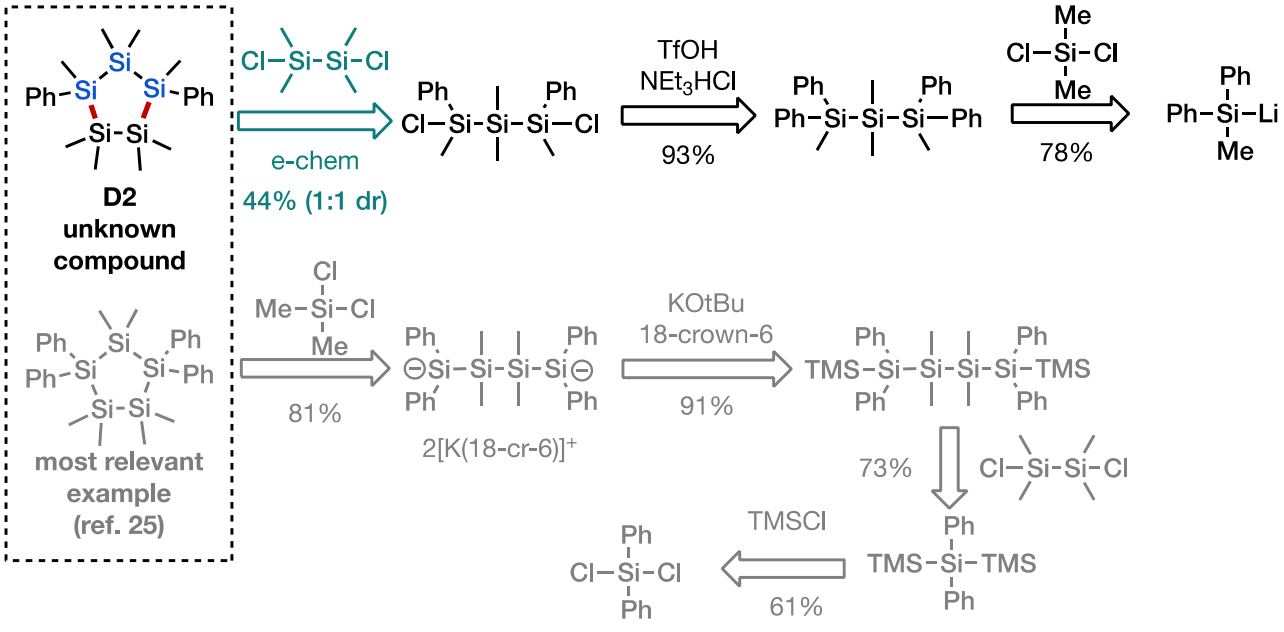
10. Unsuccessful and suboptimal substrates

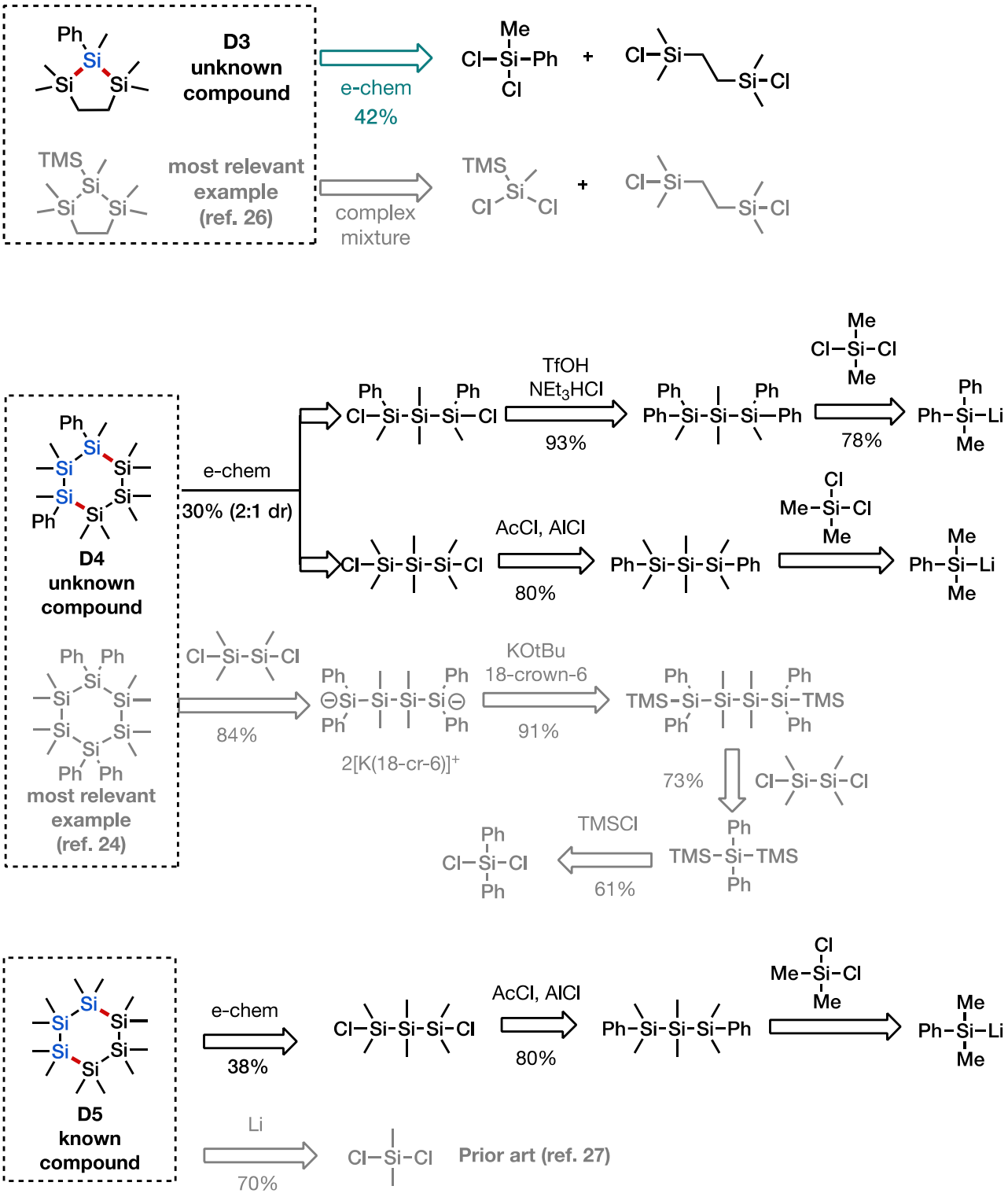


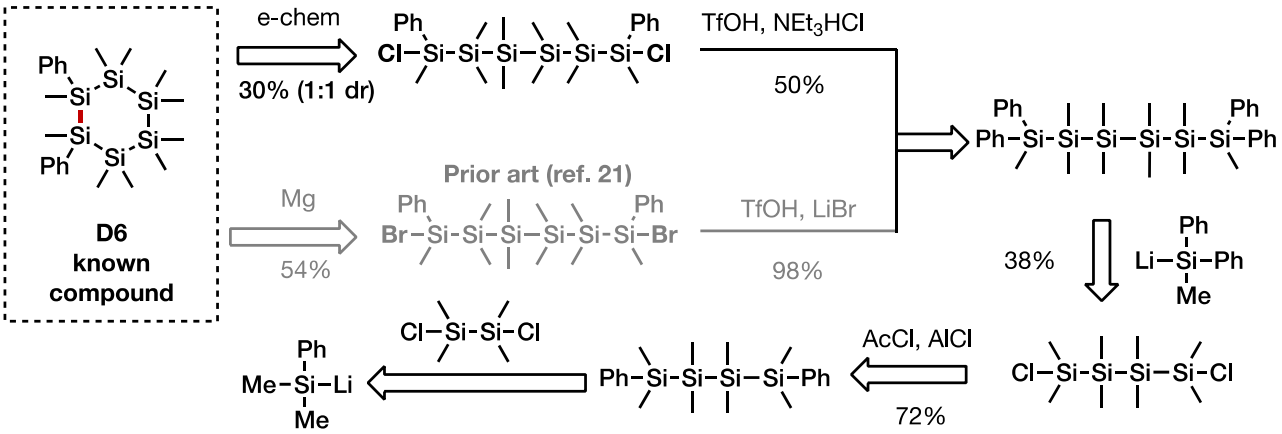
11. Comparison of cyclosilane synthesis from this work to prior routes

Following is a detailed comparison of cyclosilane synthesis from this work and from the known precedence. Compounds **D1**-**D4** have no known precedence, and the most relevant example were shown with their synthetic routes. Compounds **D5** and **D6** were synthesized previously using different routes. Overall, previous synthetic methods of cyclosilanes rely on different strategy while our methods provide a uniformed method for cyclosilane synthesis.






D3
unknown
compound
e-chem
42%
most relevant
example
(ref. 26)
complex
mixture
D4
unknown
compound
e-chem
30% (2:1 dr)
TfOH
NEt3HCl
93%
78%
AcCl, AlCl
80%
most relevant
example
(ref. 24)
84%
KOtBu
18-crown-6
91%
2[K(18-cr-6)]+
73%
TMSCl
61%
D5
known
compound
e-chem
38%
AcCl, AlCl
80%
Li
70%
Prior art (ref. 27)


D6
known
compound
e-chem
30% (1:1 dr)
TfOH, NEt3HCl
50%
Prior art (ref. 21)
Mg
54%
TfOH, LiBr
98%
38%
AcCl, AlCl
72%

12. References

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5986.
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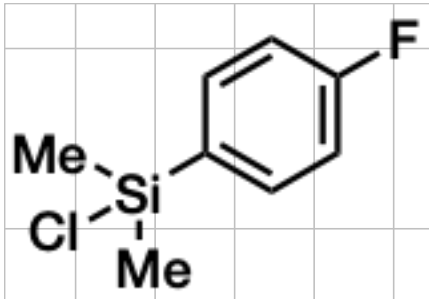
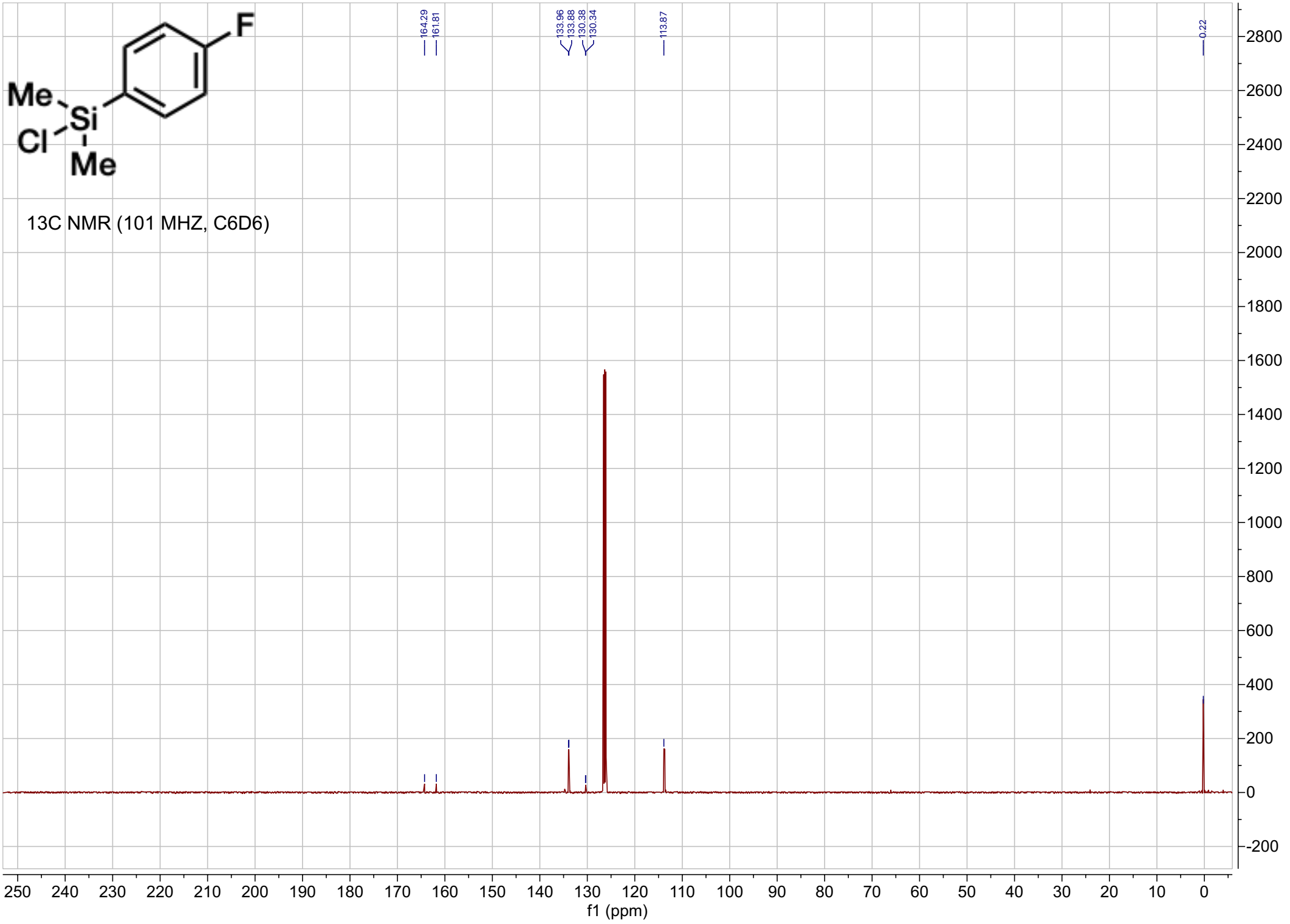
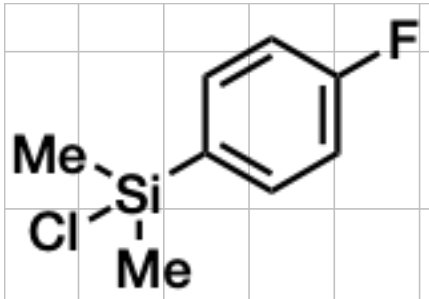
13. Copies of NMR of chlorosilanes starting materials and product



1H NMR (500 MHZ, C6D6)


F
Me
Si
Cl
Me
13C NMR (101 MHZ, C6D6)
164.29
161.81
133.96
133.88
130.38
130.34
113.87
0.22
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



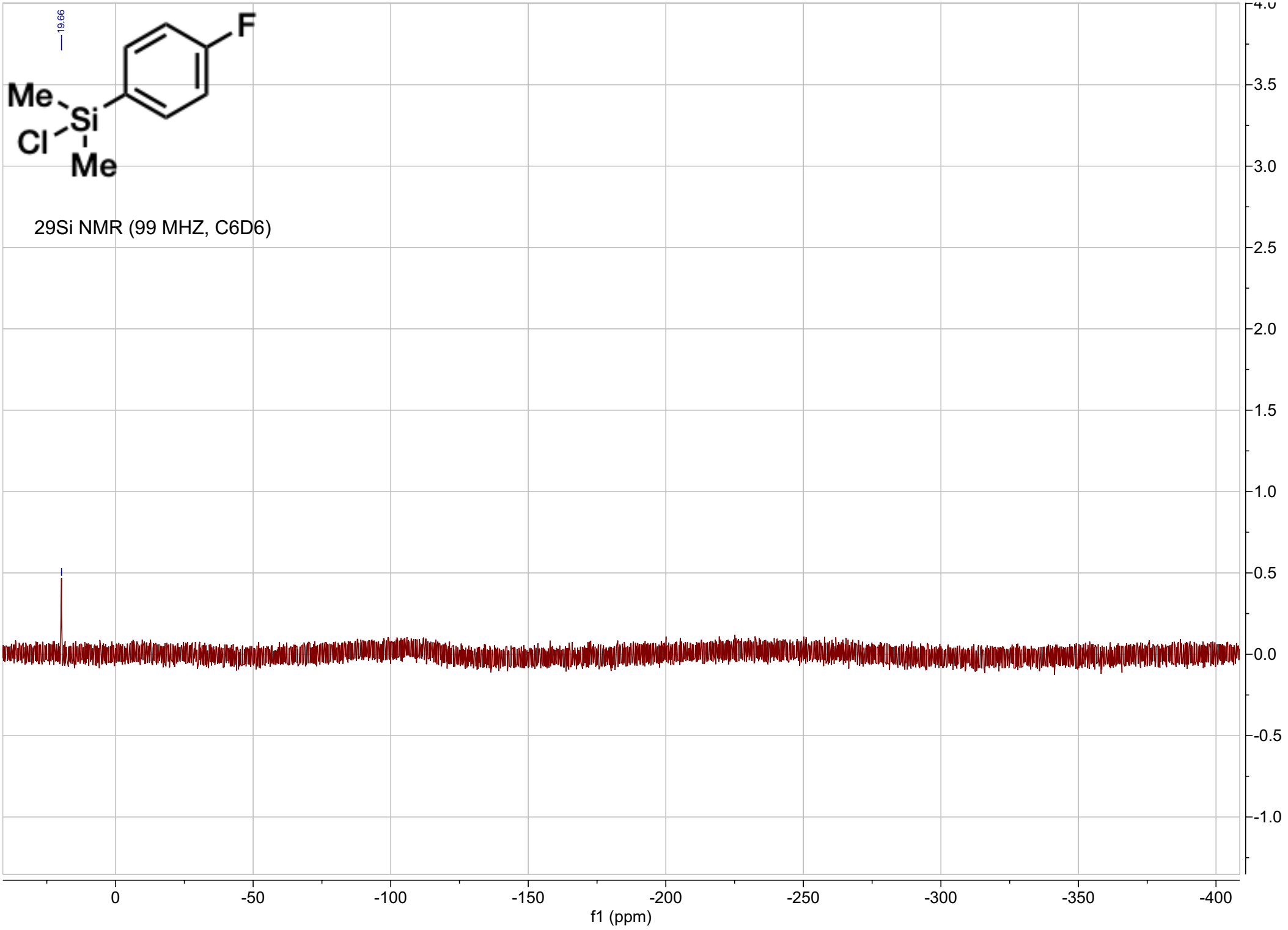
19F NMR (376 MHZ, C6D6)

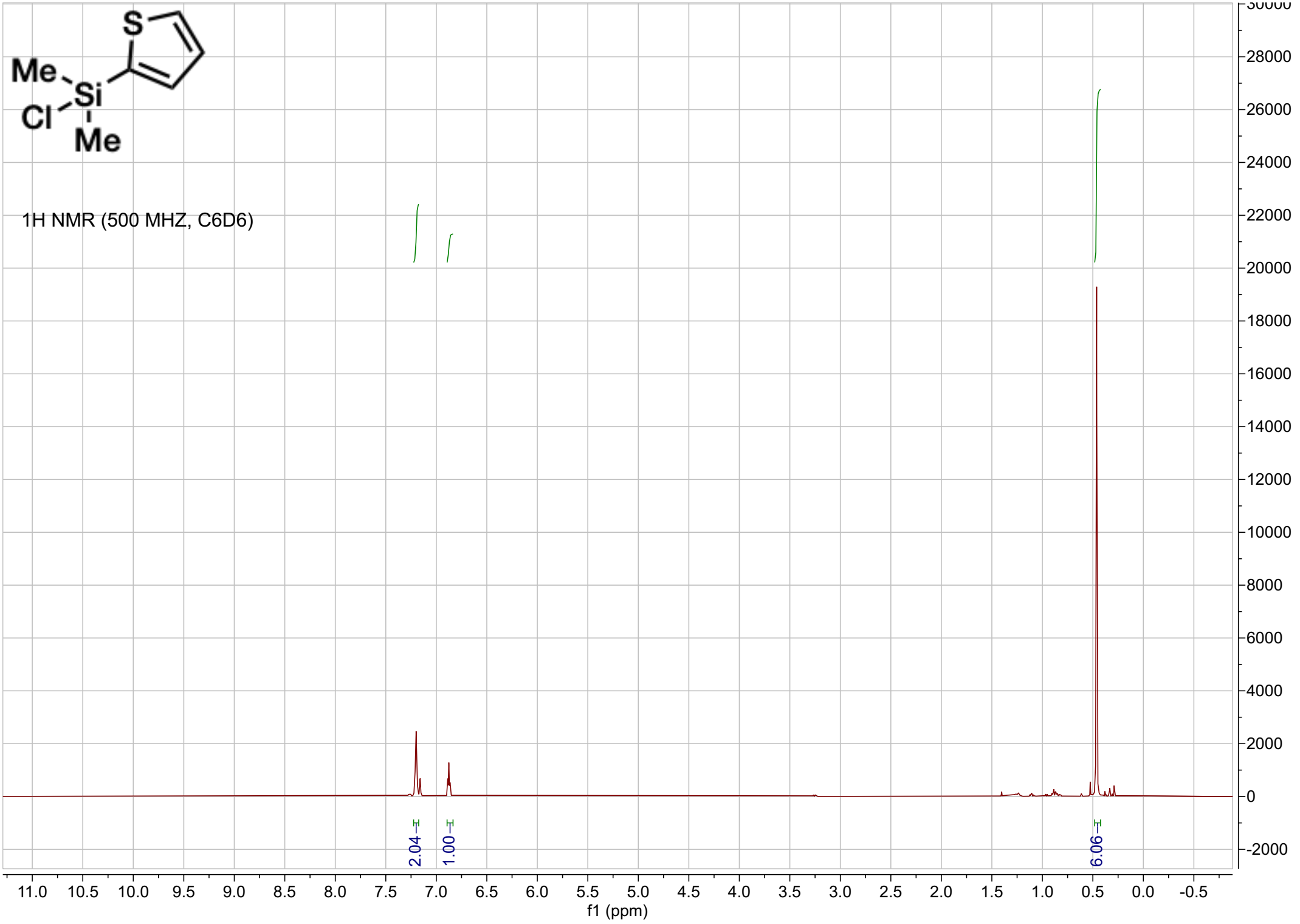
-109.63

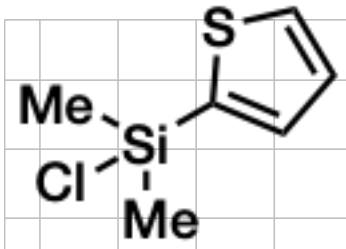
40000
35000
30000
25000
20000
15000
10000
5000
0

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

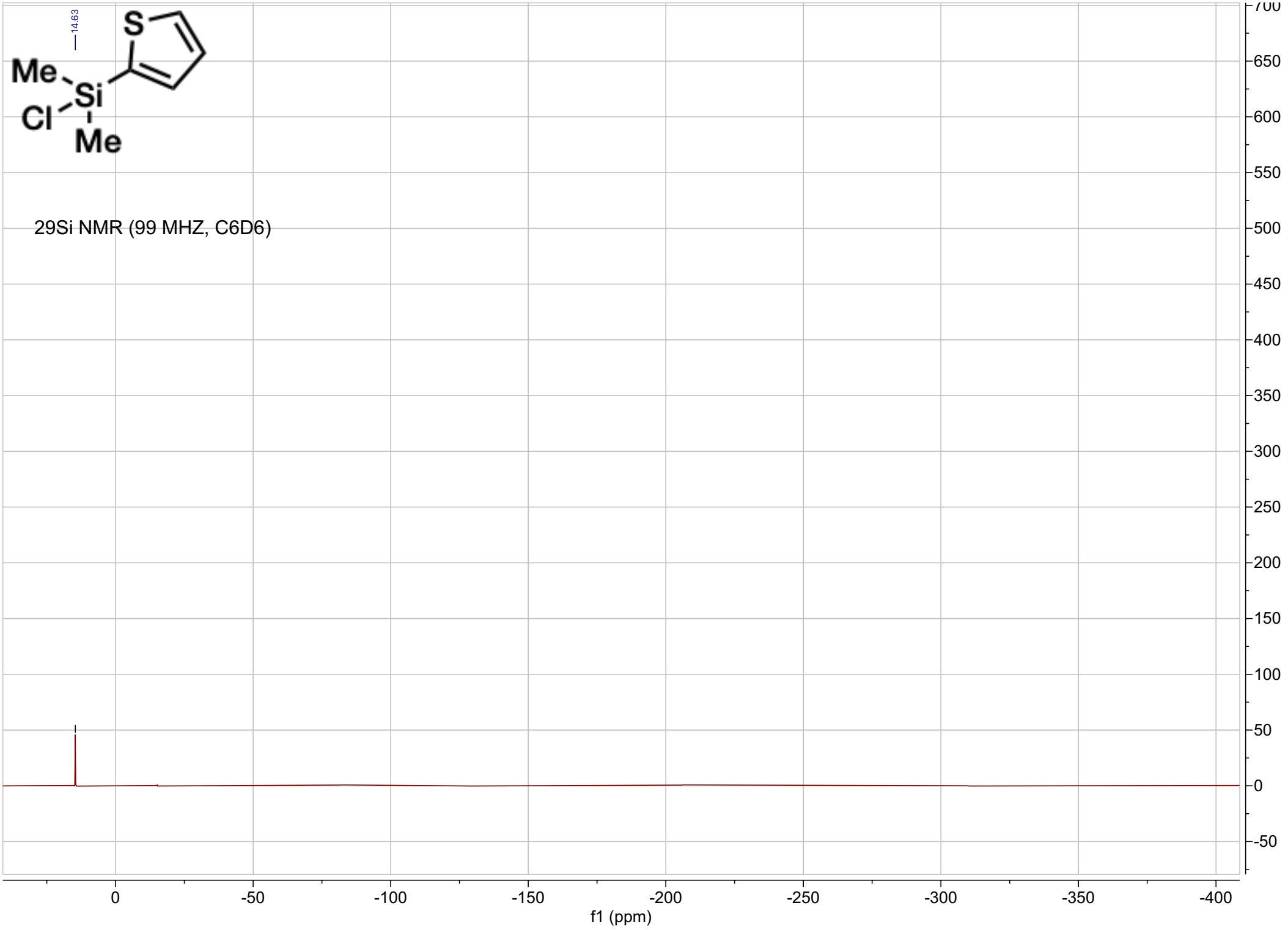
f1 (ppm)

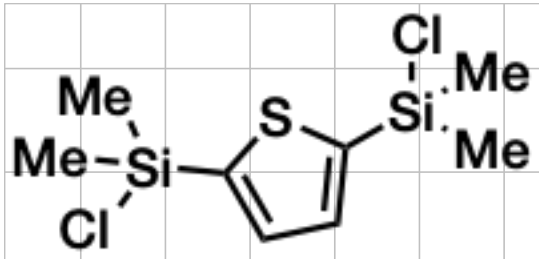

19.66
F
Me
Si
Cl
Me
29Si NMR (99 MHZ, C6D6)
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)


Me
Si
Cl
Me
S
1H NMR (500 MHZ, C6D6)
2.04
1.00
6.06
f1 (ppm)

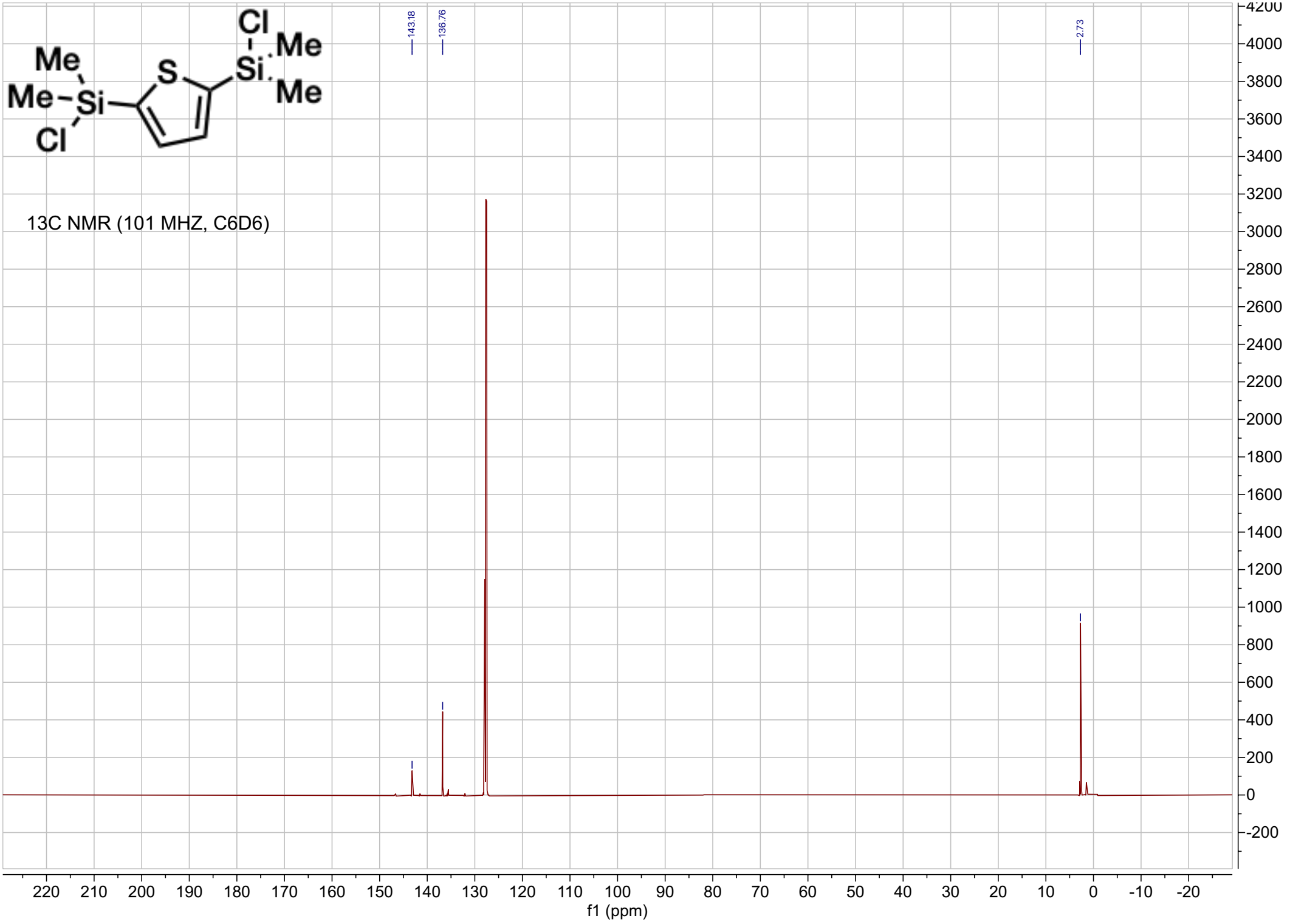


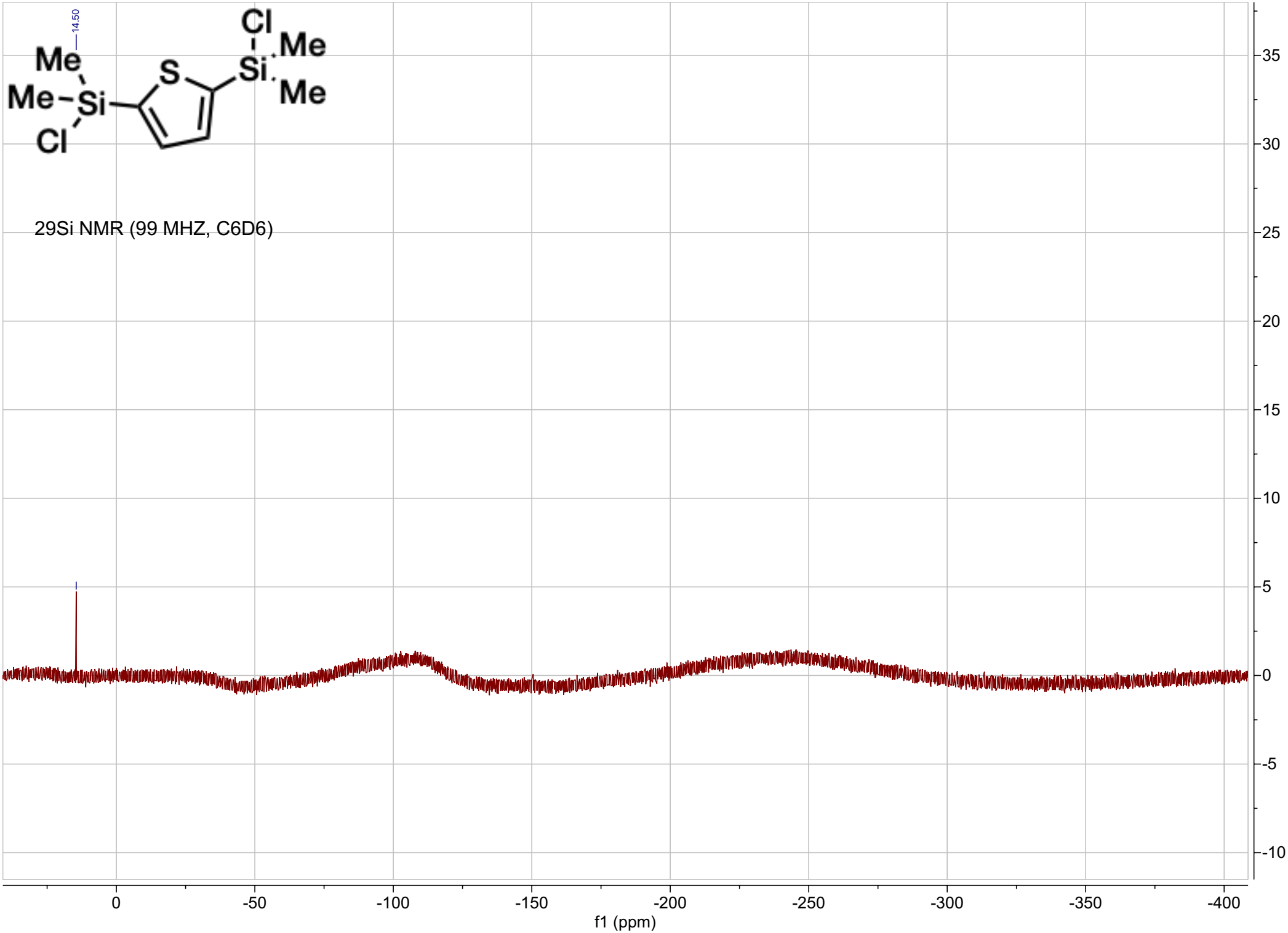
13C NMR (101 MHZ, C6D6)

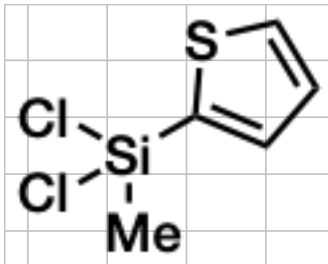

14.63
S
Me
Si
Cl
Me
29Si NMR (99 MHZ, C6D6)
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)



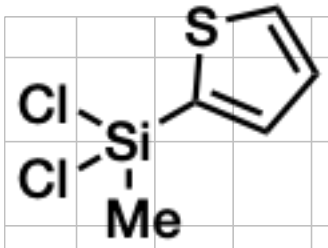
1H NMR (500 MHZ, C6D6)


Me
Me
Si
Cl
S
Cl
Si
Me
Me
13C NMR (101 MHZ, C6D6)
143.18
136.76
2.73
4200
4000
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

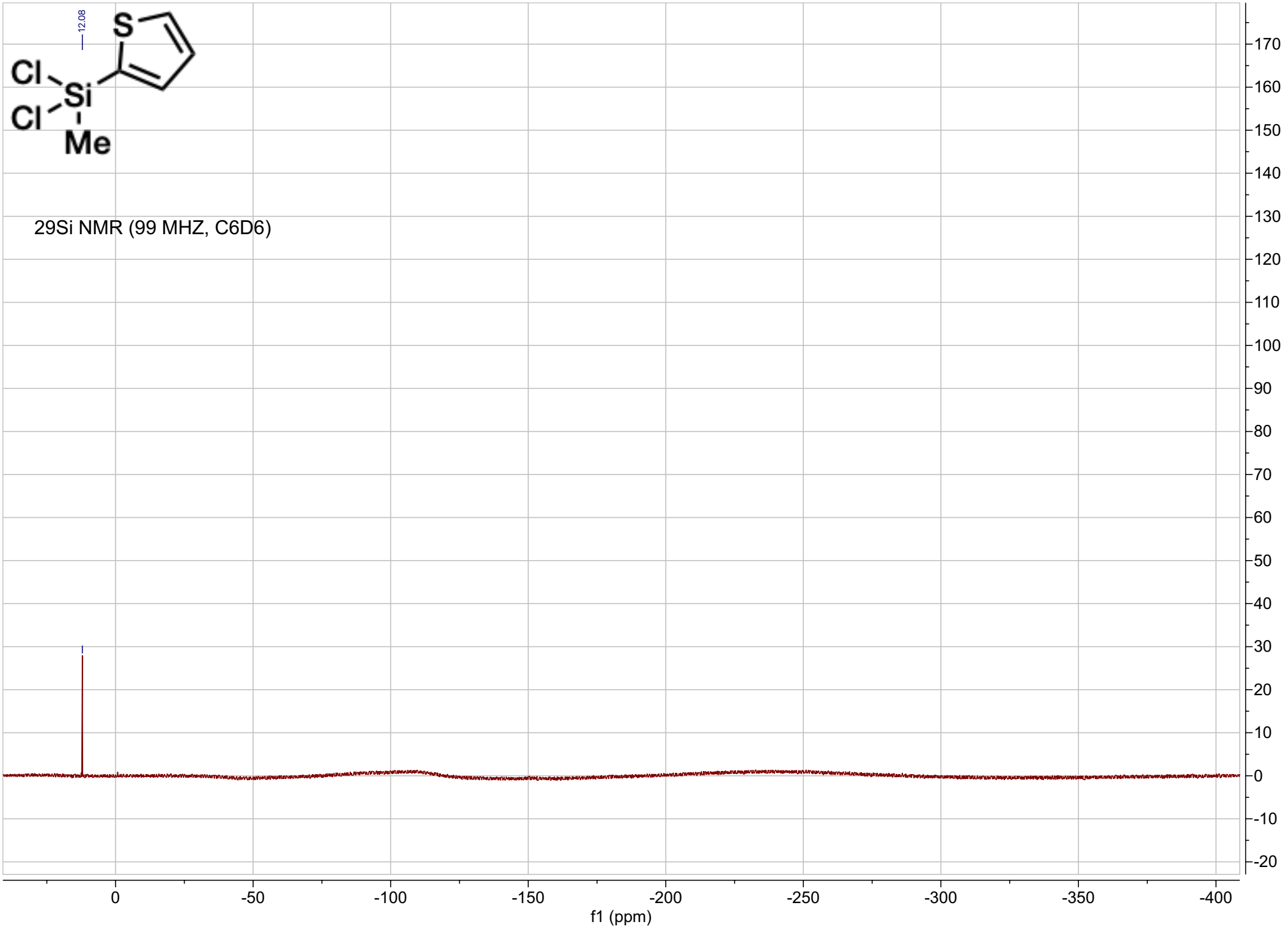

14.50
Me
Me
Si
Cl
S
Cl
Si
Me
Me
29Si NMR (99 MHZ, C6D6)
35
30
25
20
15
10
5
0
-5
-10
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

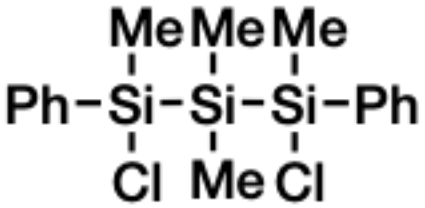


1H NMR (500 MHZ, C6D6)

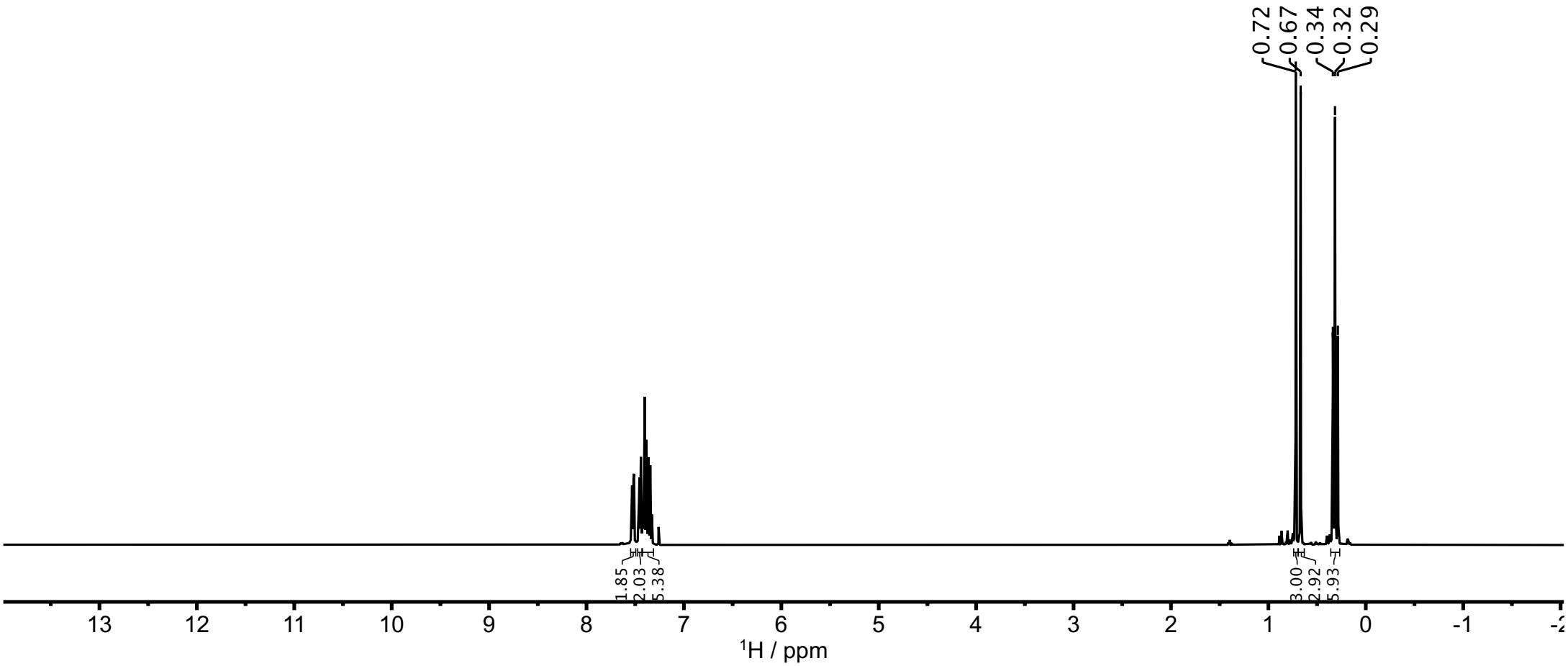


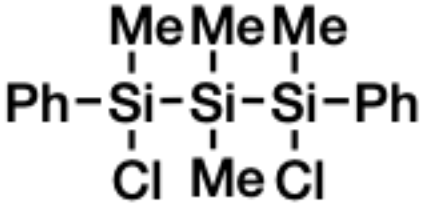
13C NMR (101 MHZ, C6D6)


12.08
S
Cl
Si
Cl
Me
29Si NMR (99 MHZ, C6D6)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

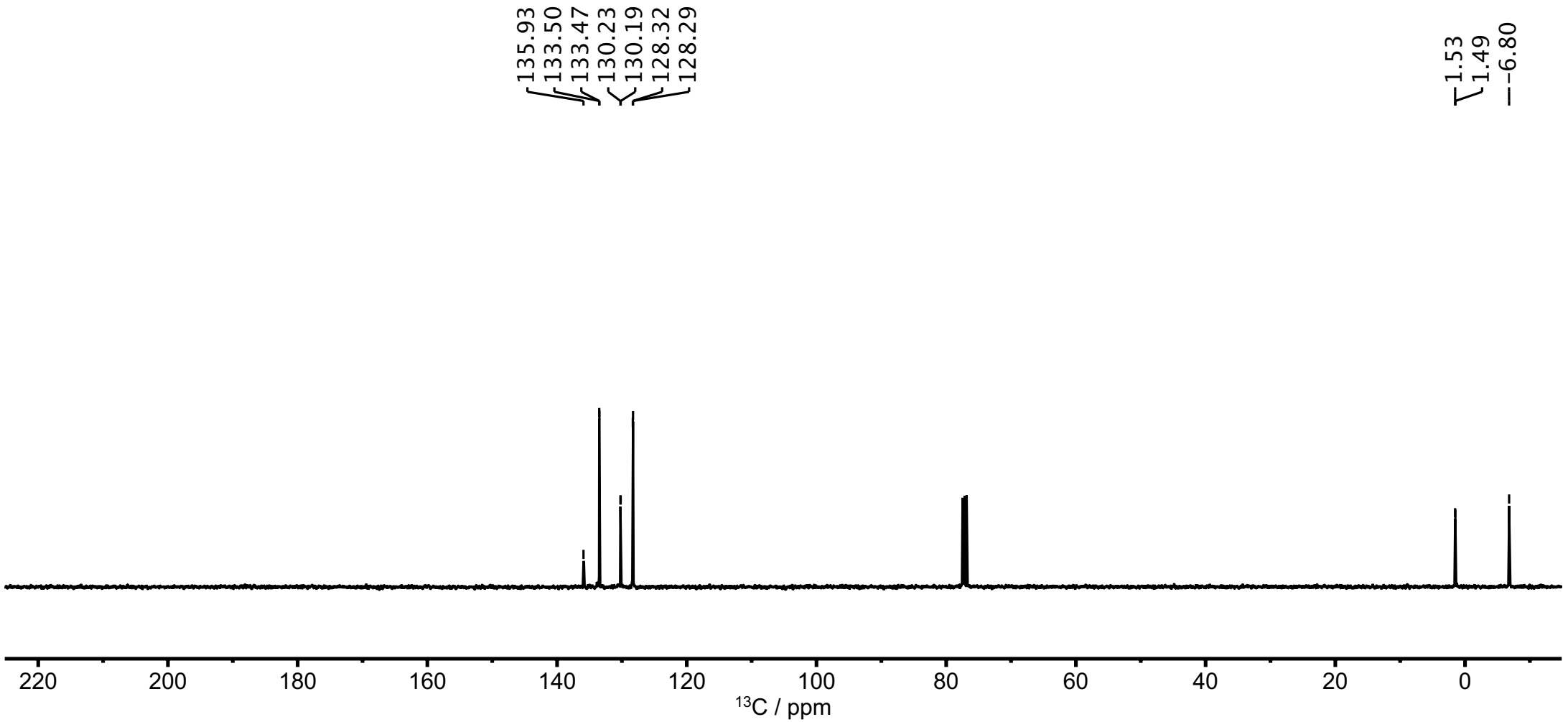


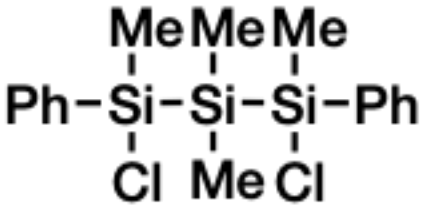
1H NMR (400 MHZ, C6D6)



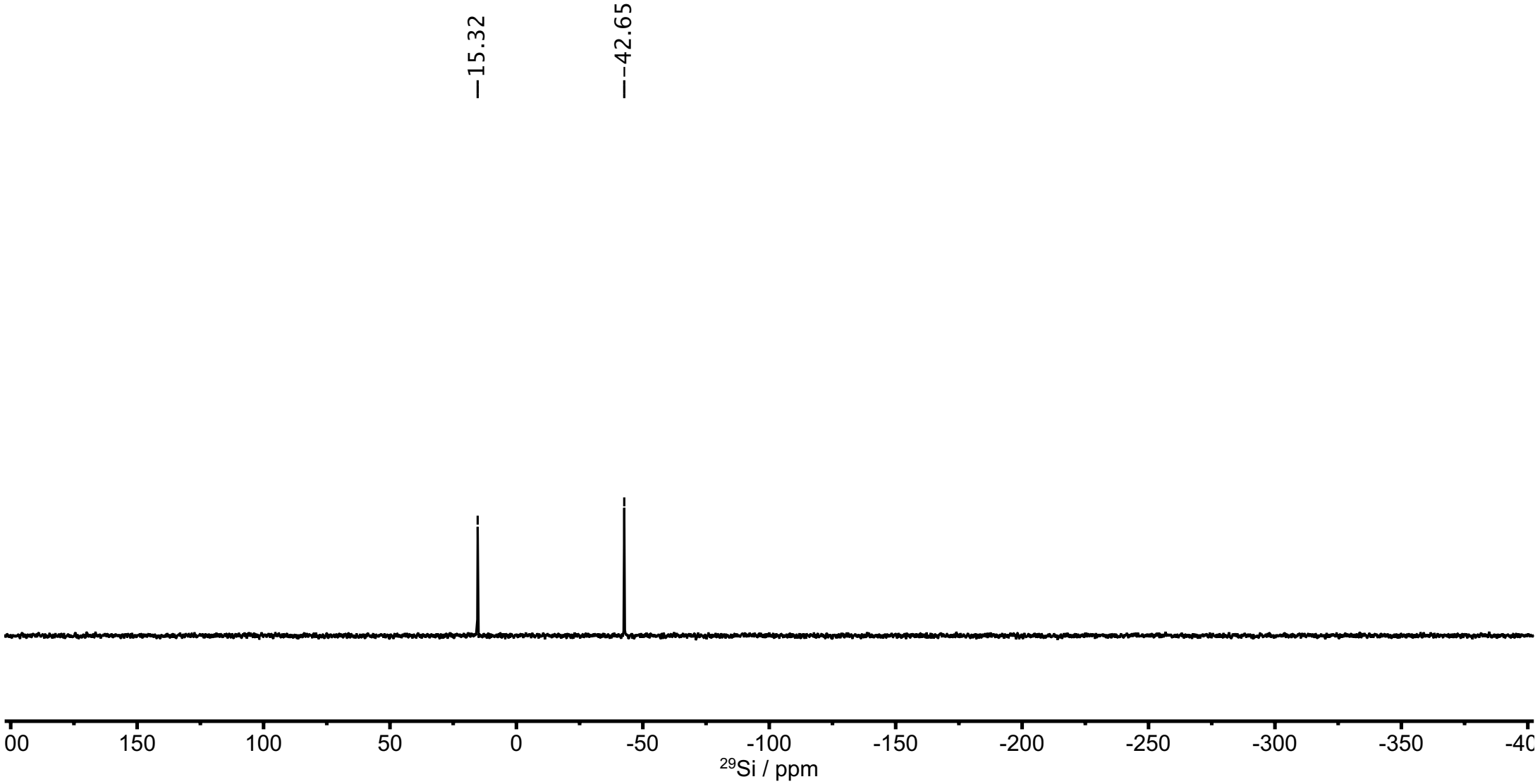


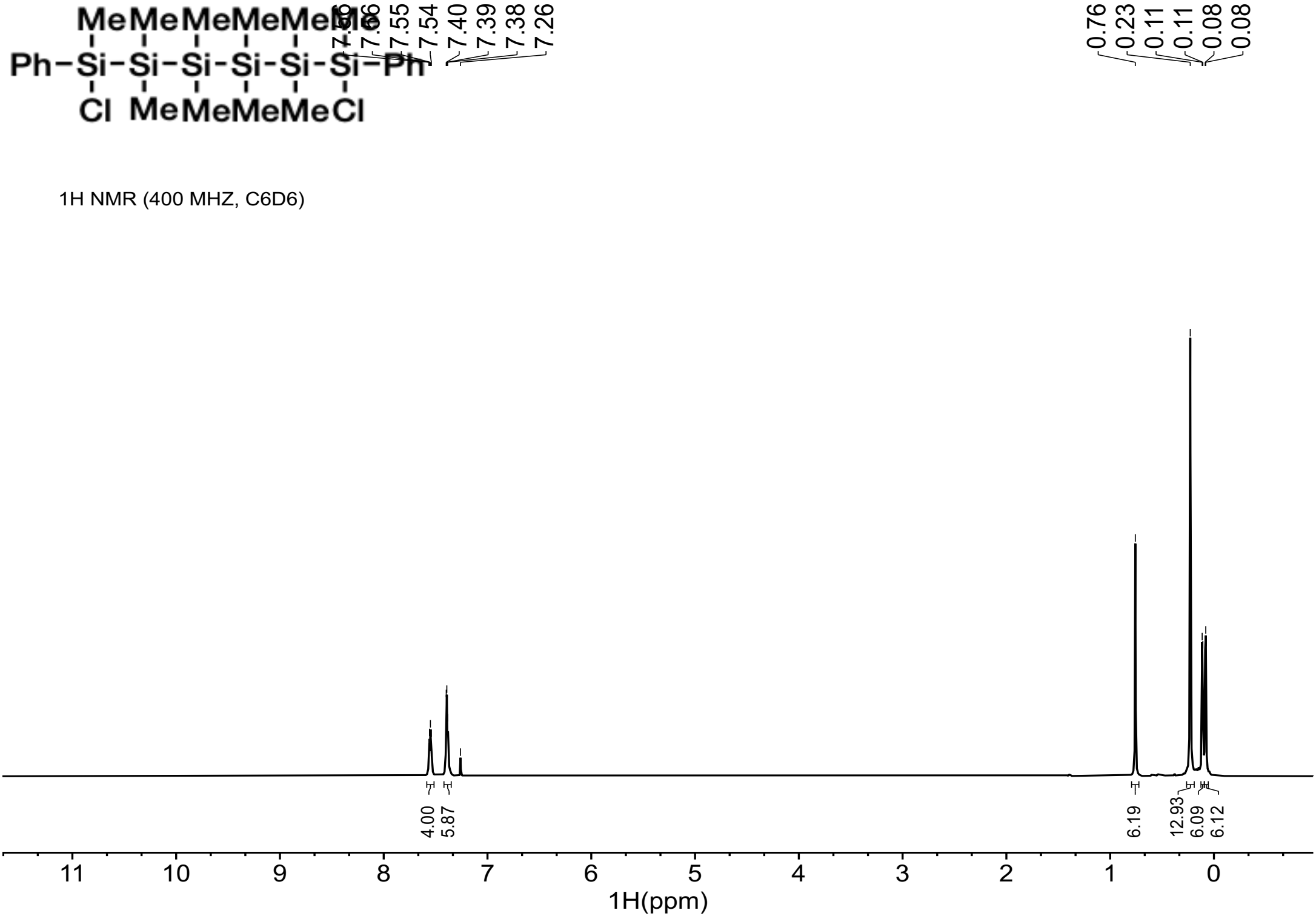
13C NMR (101 MHZ, CDCl3)





29Si NMR (99 MHZ, CDCl3)



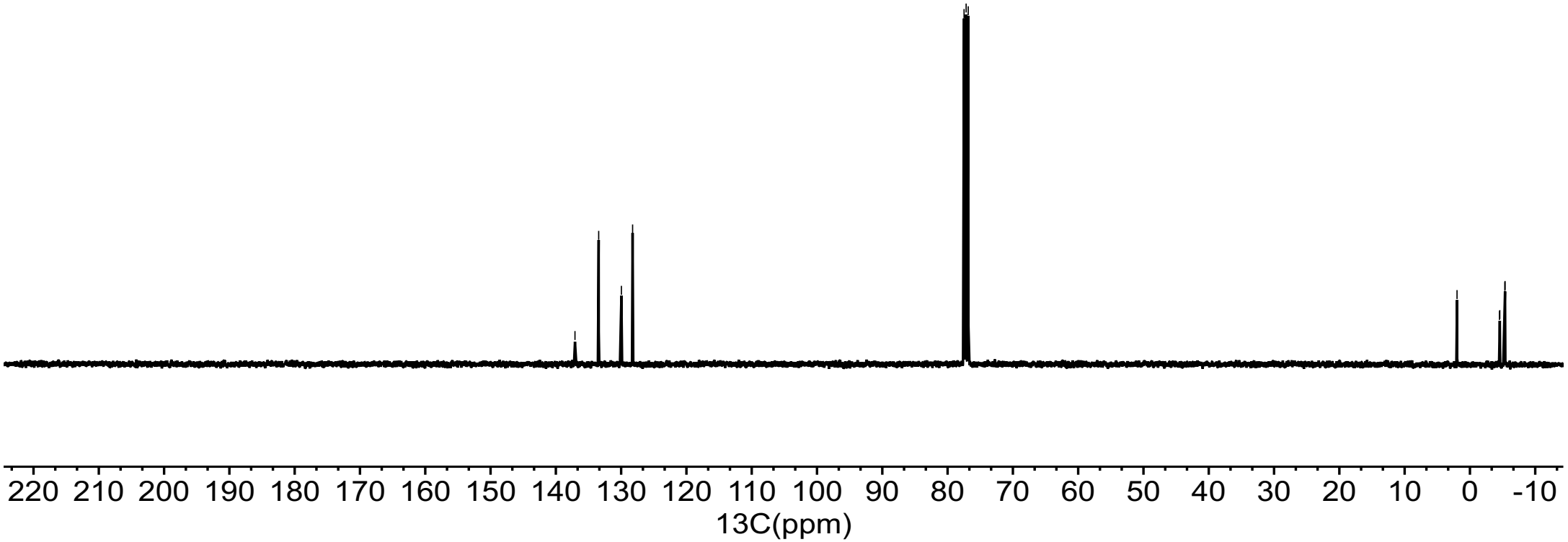

Me Me Me Me Me Me
Ph–Si–Si–Si–Si–Si–Si–Ph
Cl Me Me Me Me Cl
7.56
7.55
7.54
7.40
7.39
7.38
7.26
0.76
0.23
0.11
0.11
0.08
0.08
1H NMR (400 MHZ, C6D6)
4.00
5.87
6.19
12.93
6.09
6.12
11
10
9
8
7
6
5
4
3
2
1
0
1H(ppm)

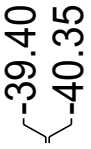
13C NMR (101 MHZ, C6D6)

137.07
133.44
129.96
128.24

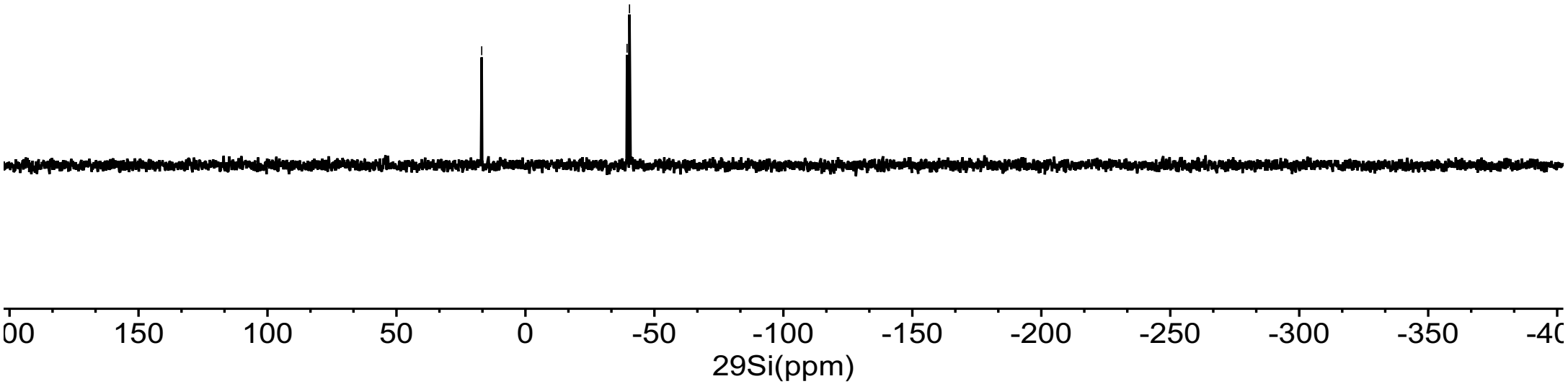
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3

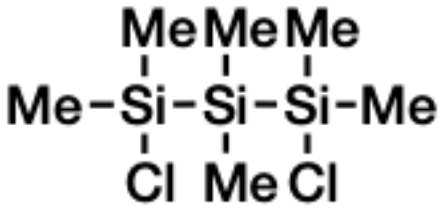
1.98
-4.52
-4.57
-5.35
-5.38



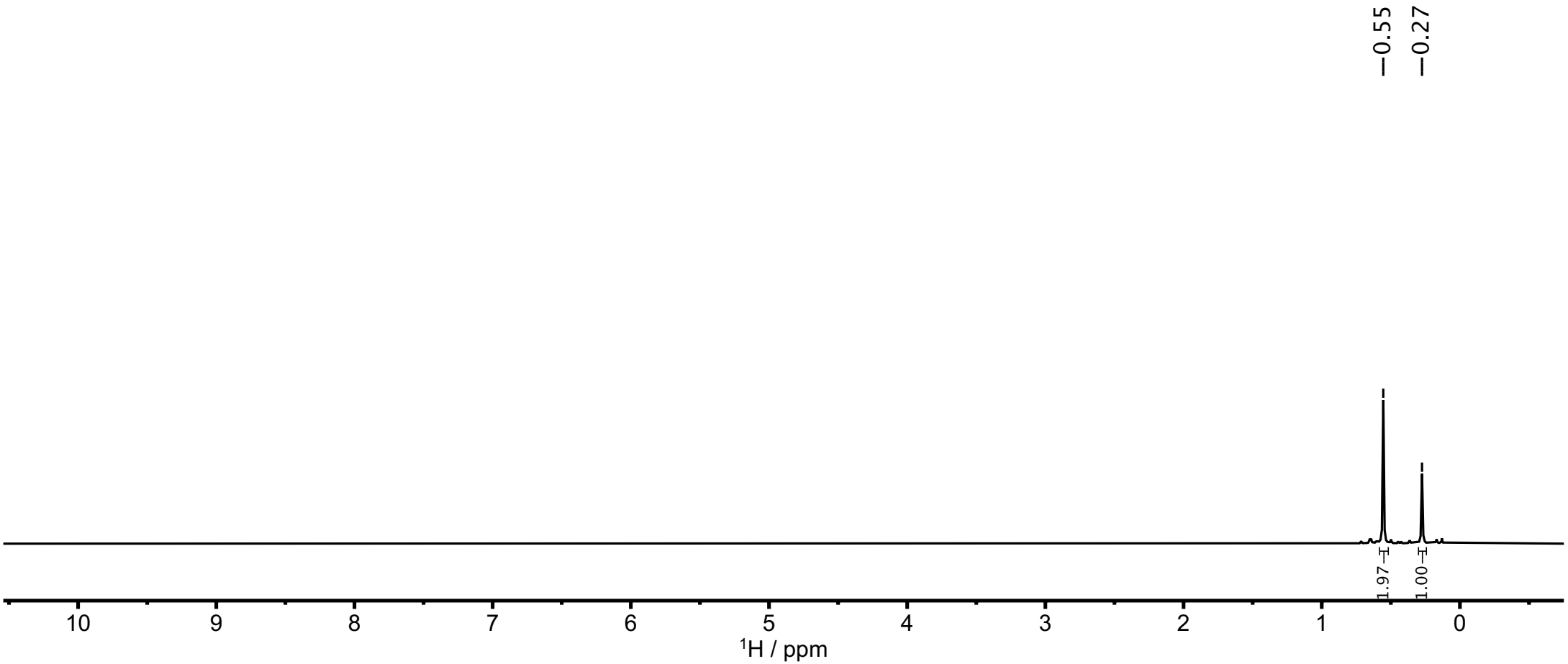


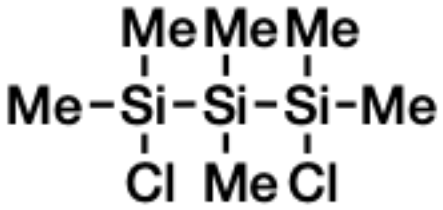
29Si NMR (99 MHZ, CDCl3)



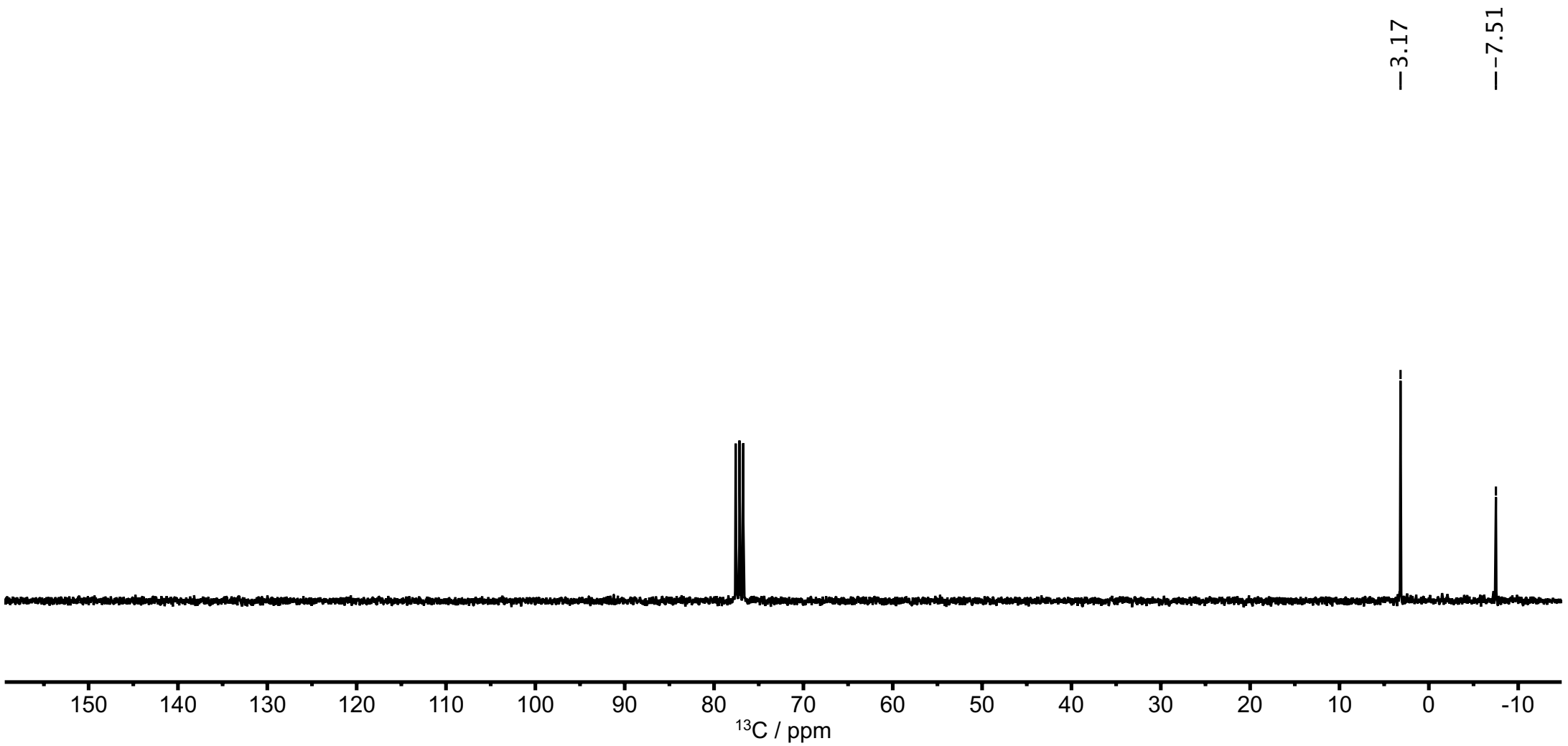


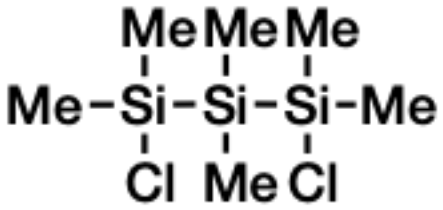
1H NMR (400 MHZ, C6D6)



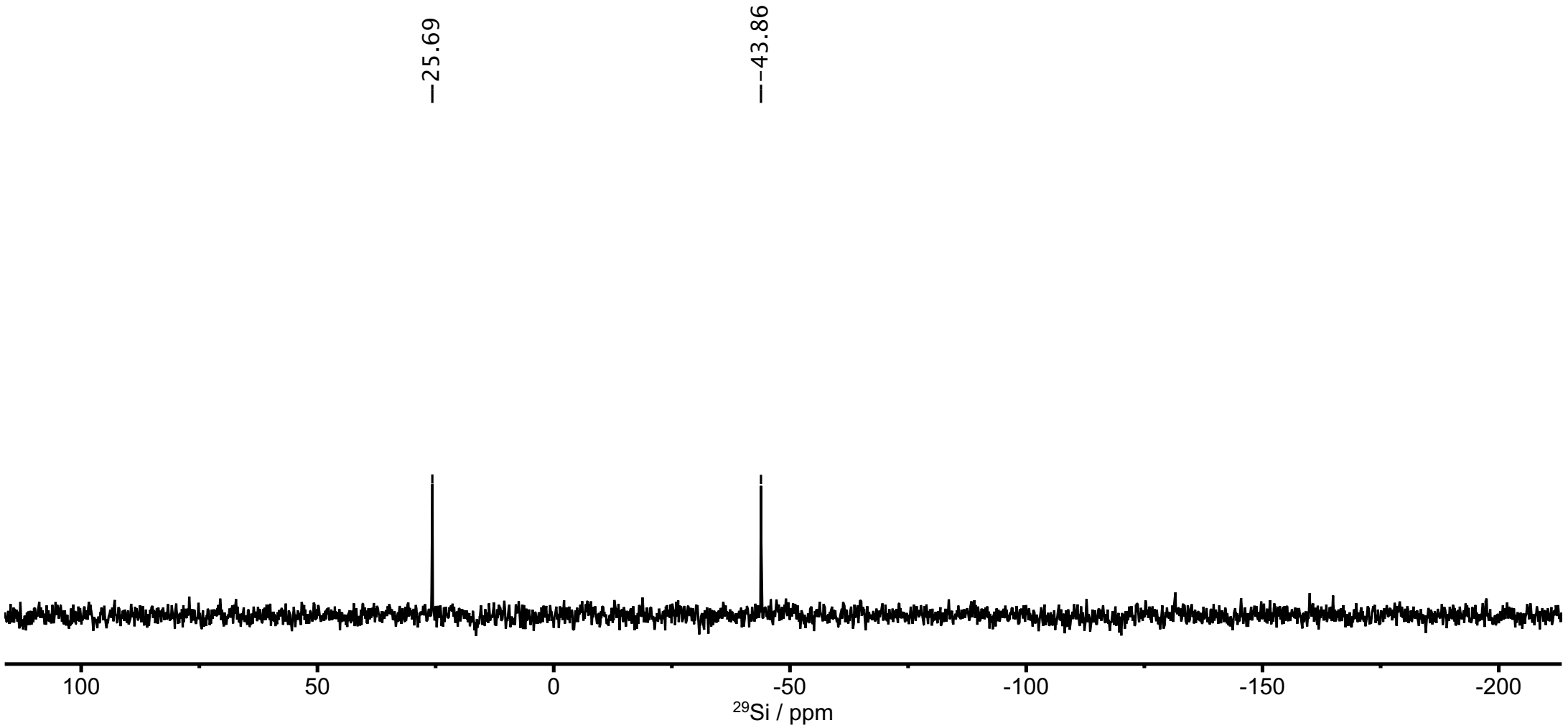


13C NMR (101 MHZ, C6D6)

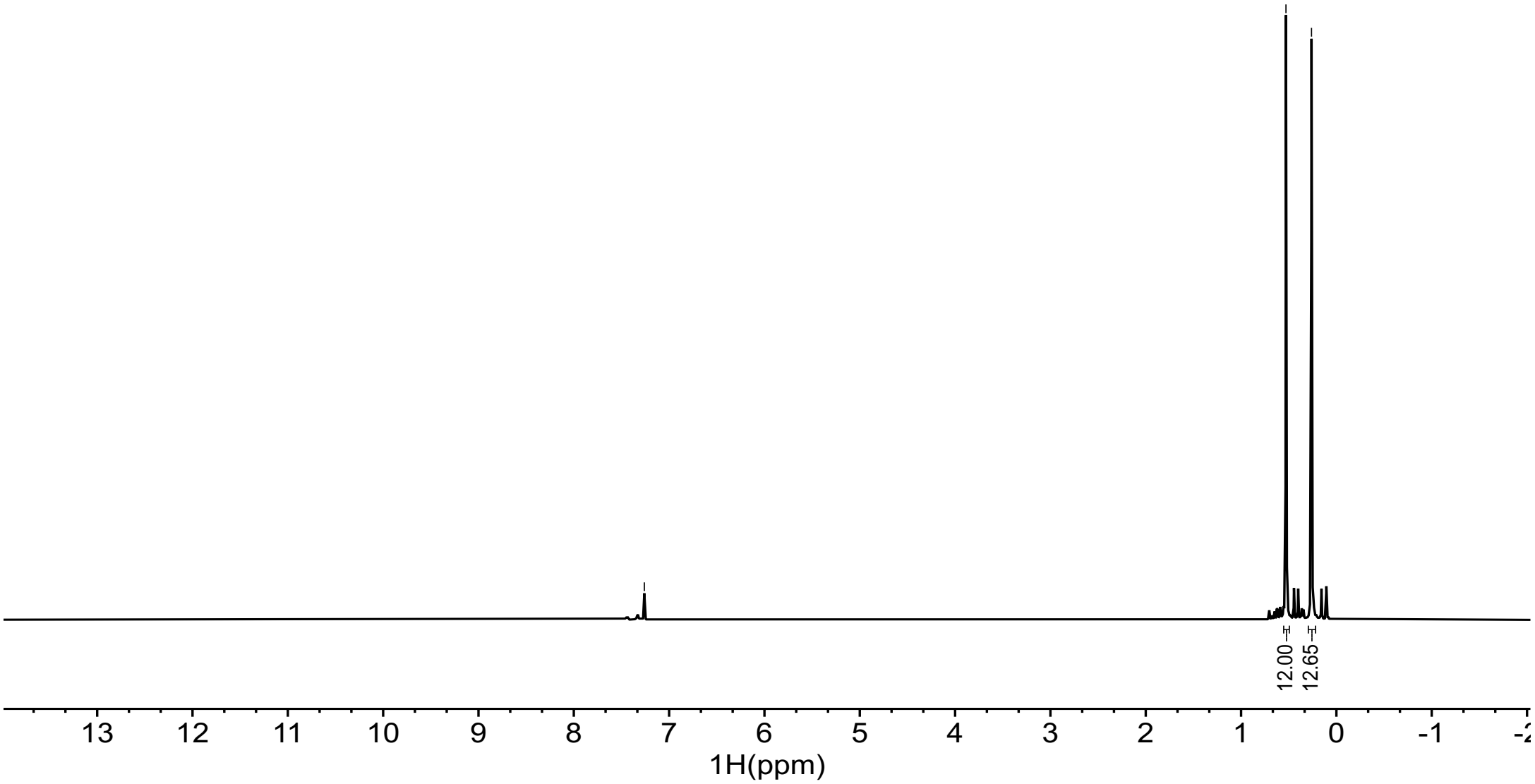




29Si NMR (99 MHZ, CDCl3)



1H NMR (400 MHZ, C6D6)

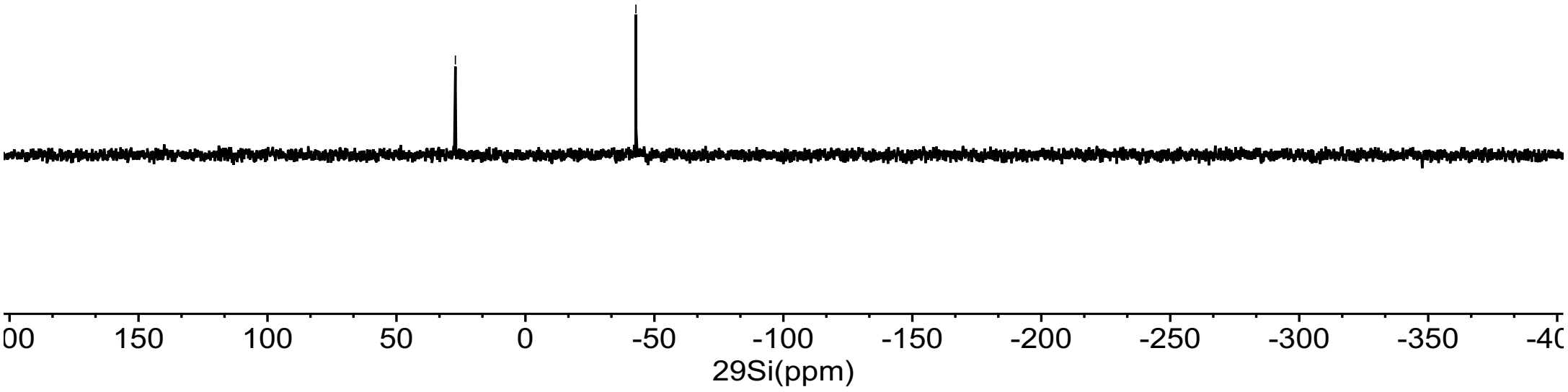


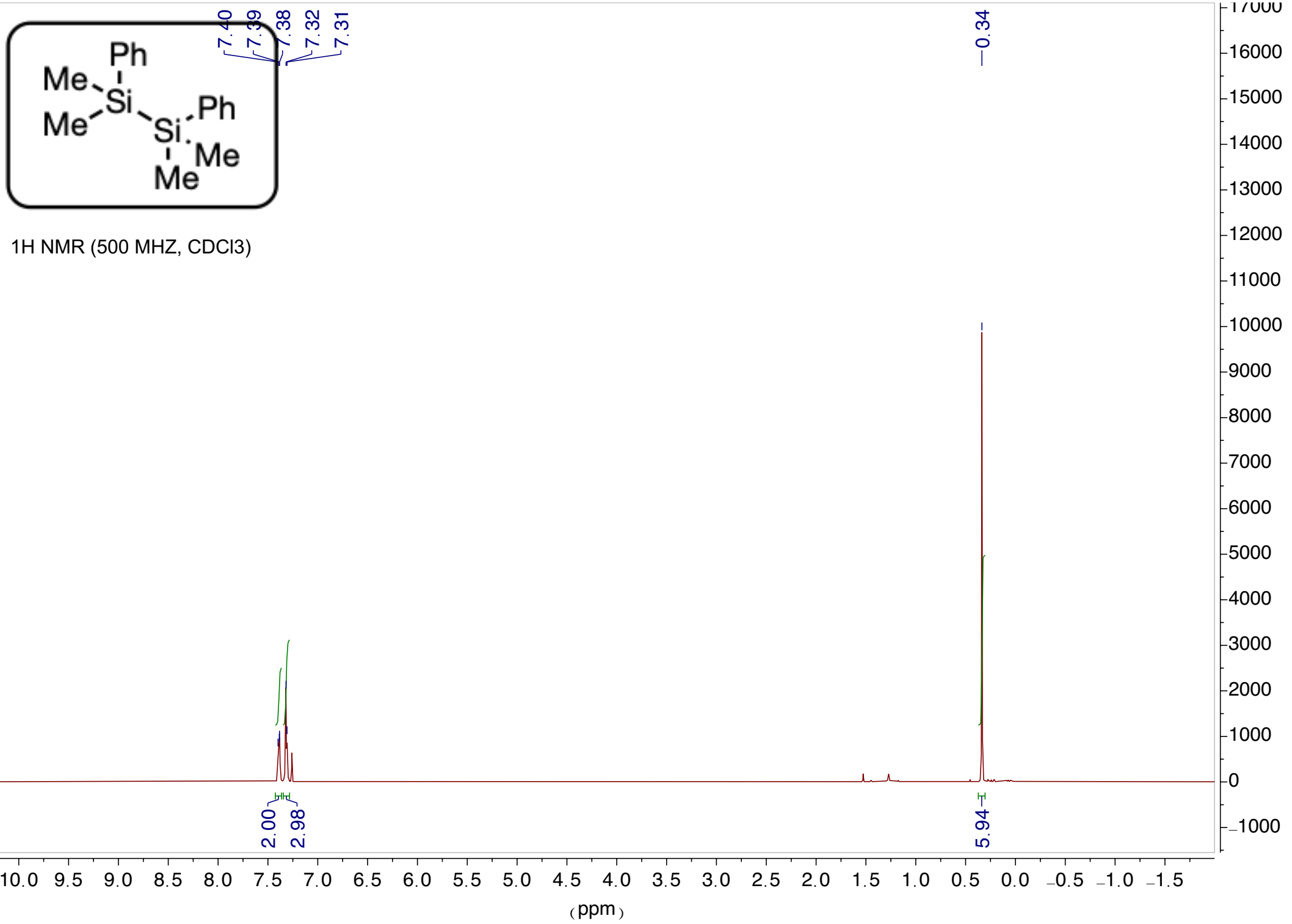
13C NMR (101 MHZ, C6D6)

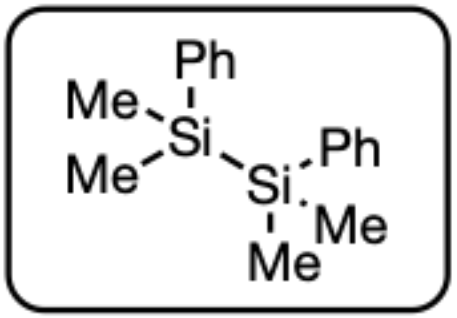
-27.16

-42.84

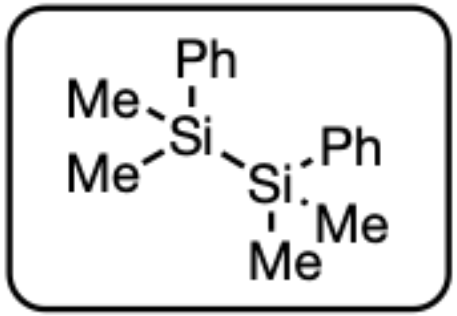
29Si NMR (99 MHZ, CDCl3)



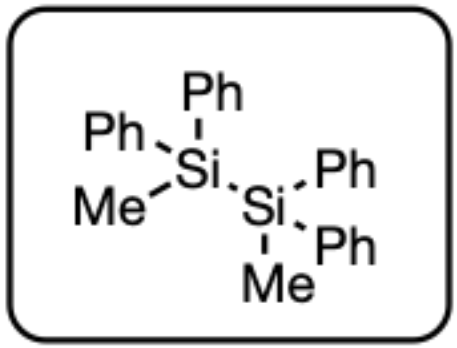

Ph
Me
Si
Me
Si
Ph
Me
Me
7.40
7.39
7.38
7.32
7.31
0.34
1H NMR (500 MHZ, CDCl3)
2.00
2.98
5.94
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
(ppm)



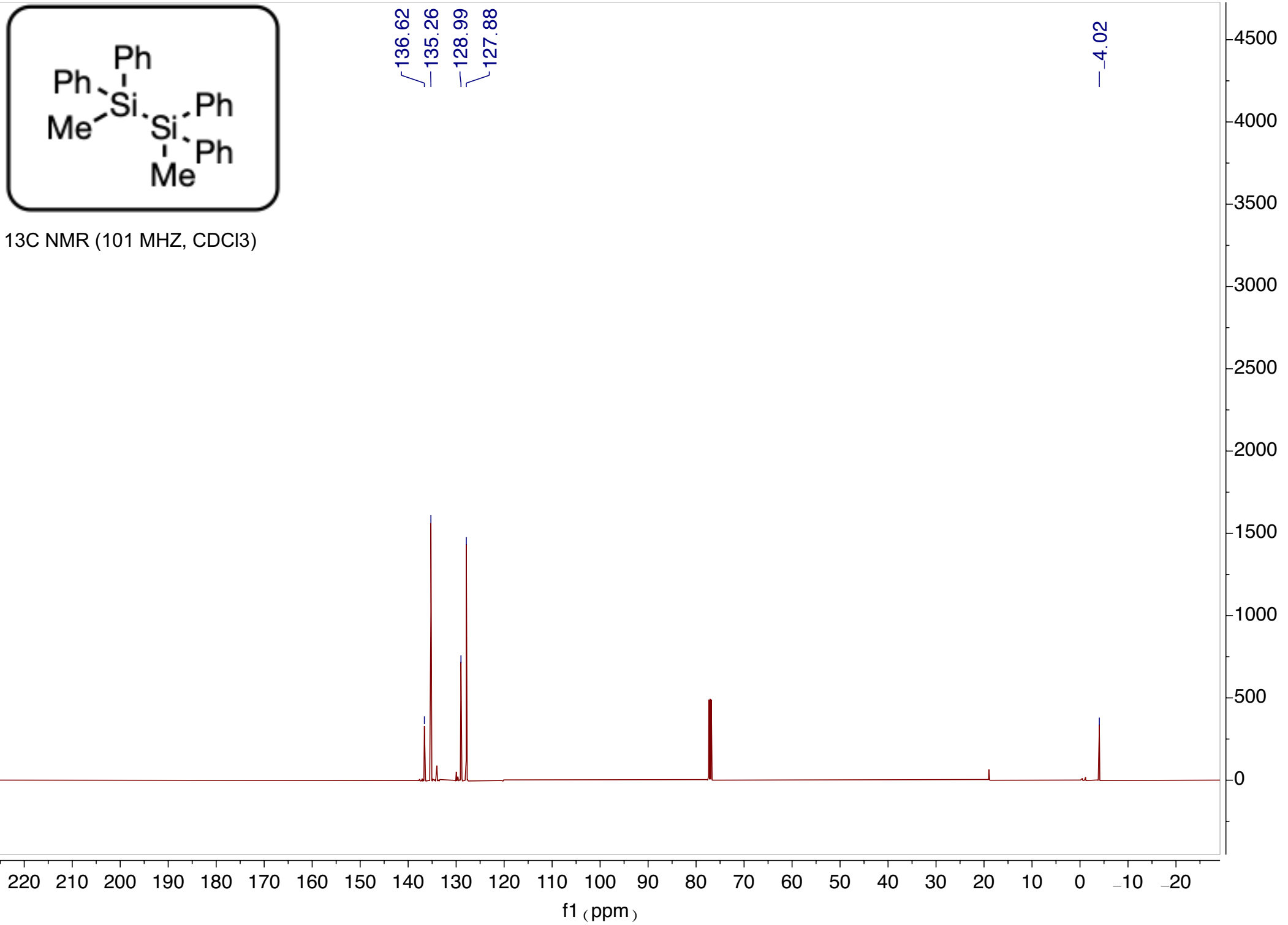
13C NMR (101 MHZ, CDCl3)

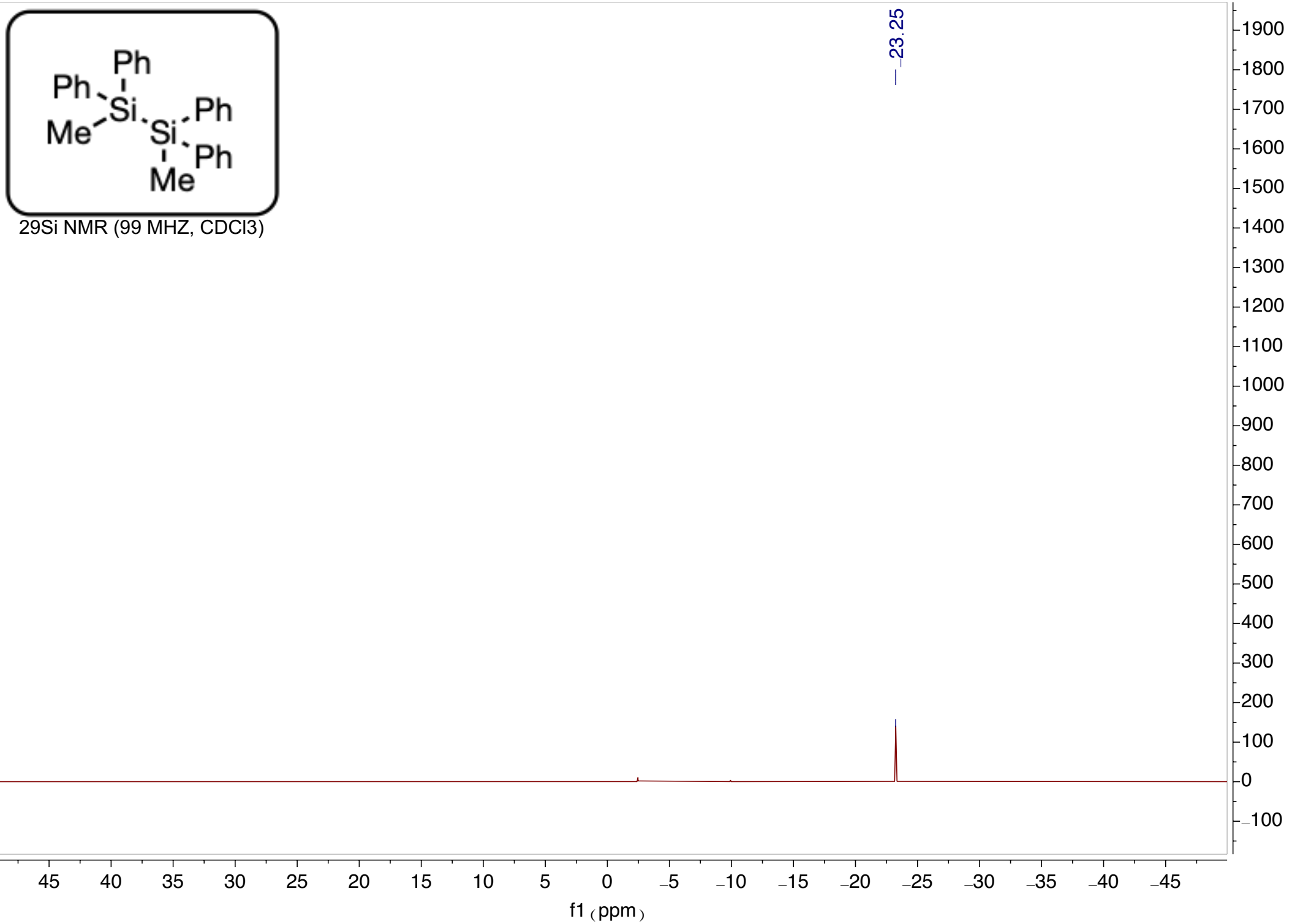


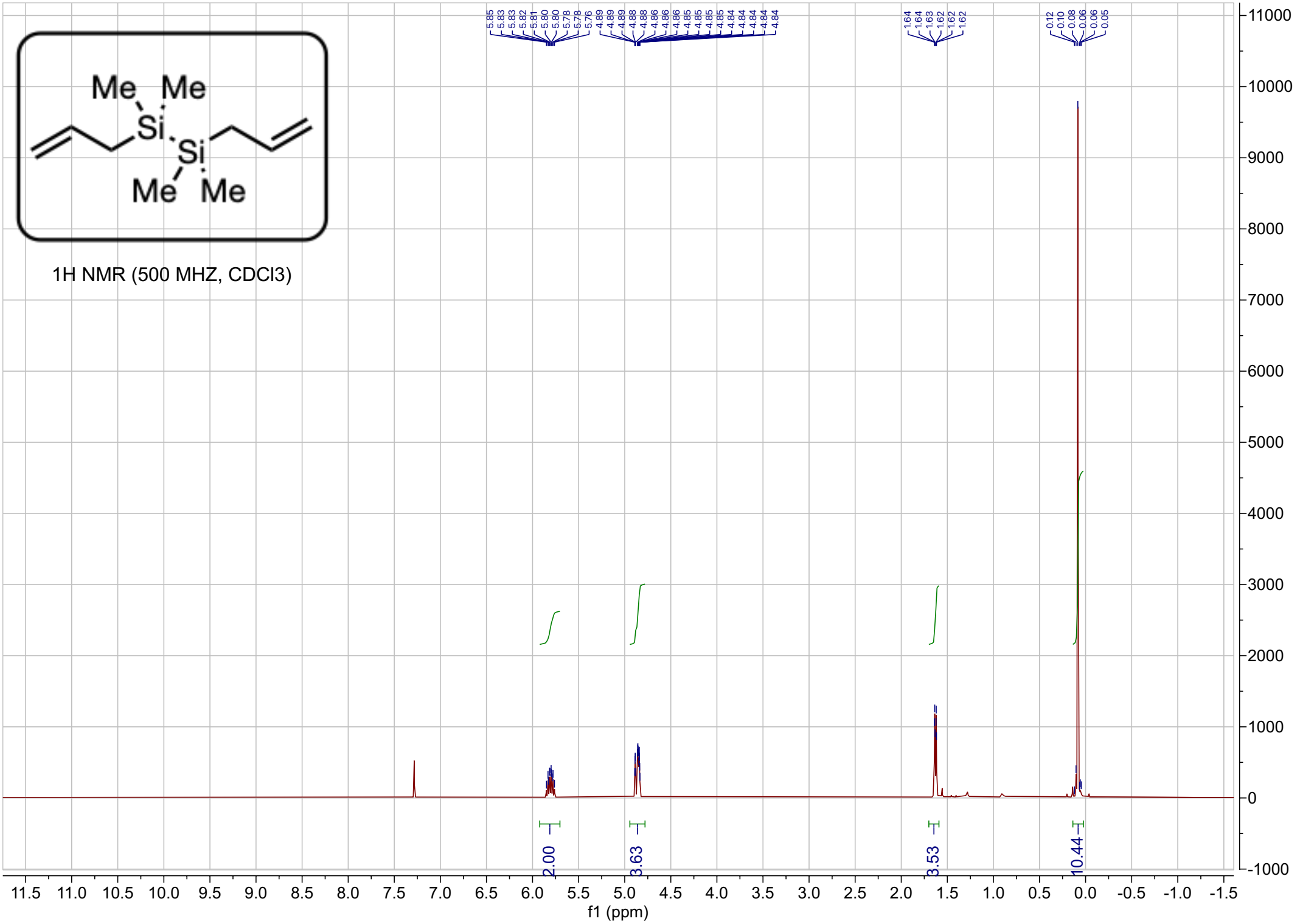
29Si NMR (99 MHZ, CDCl3)

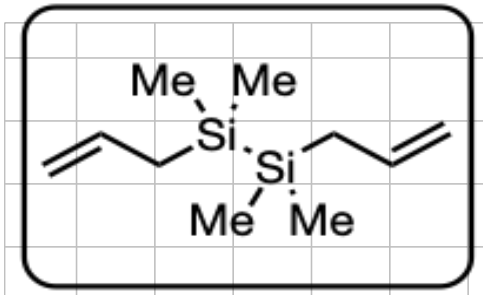


1H NMR (500 MHZ, CDCl3)

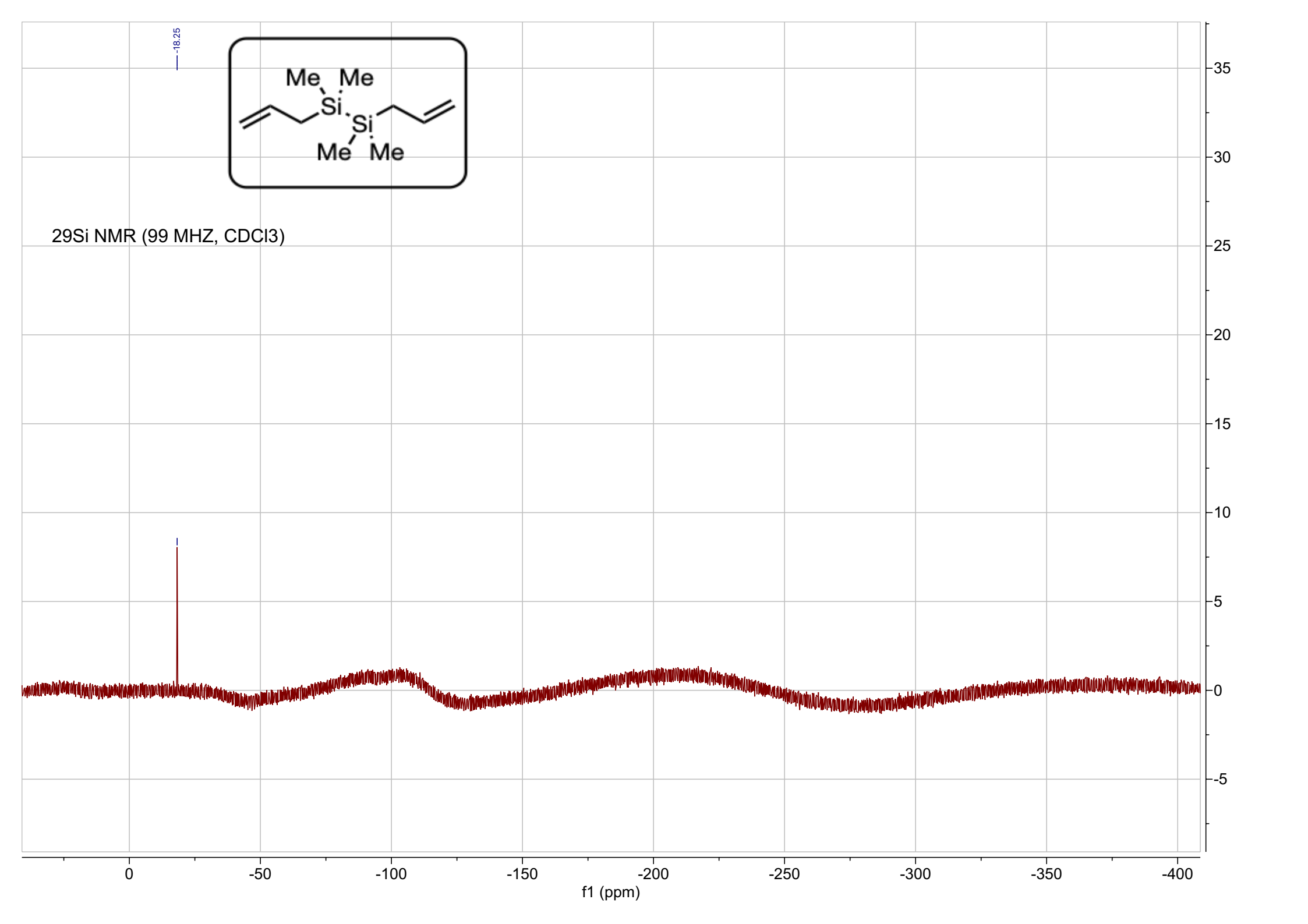

Ph
Ph
Si
Ph
Me
Si
Ph
Me
136.62
135.26
128.99
127.88
4.02
13C NMR (101 MHZ, CDCl3)
4500
4000
3500
3000
2500
2000
1500
1000
500
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

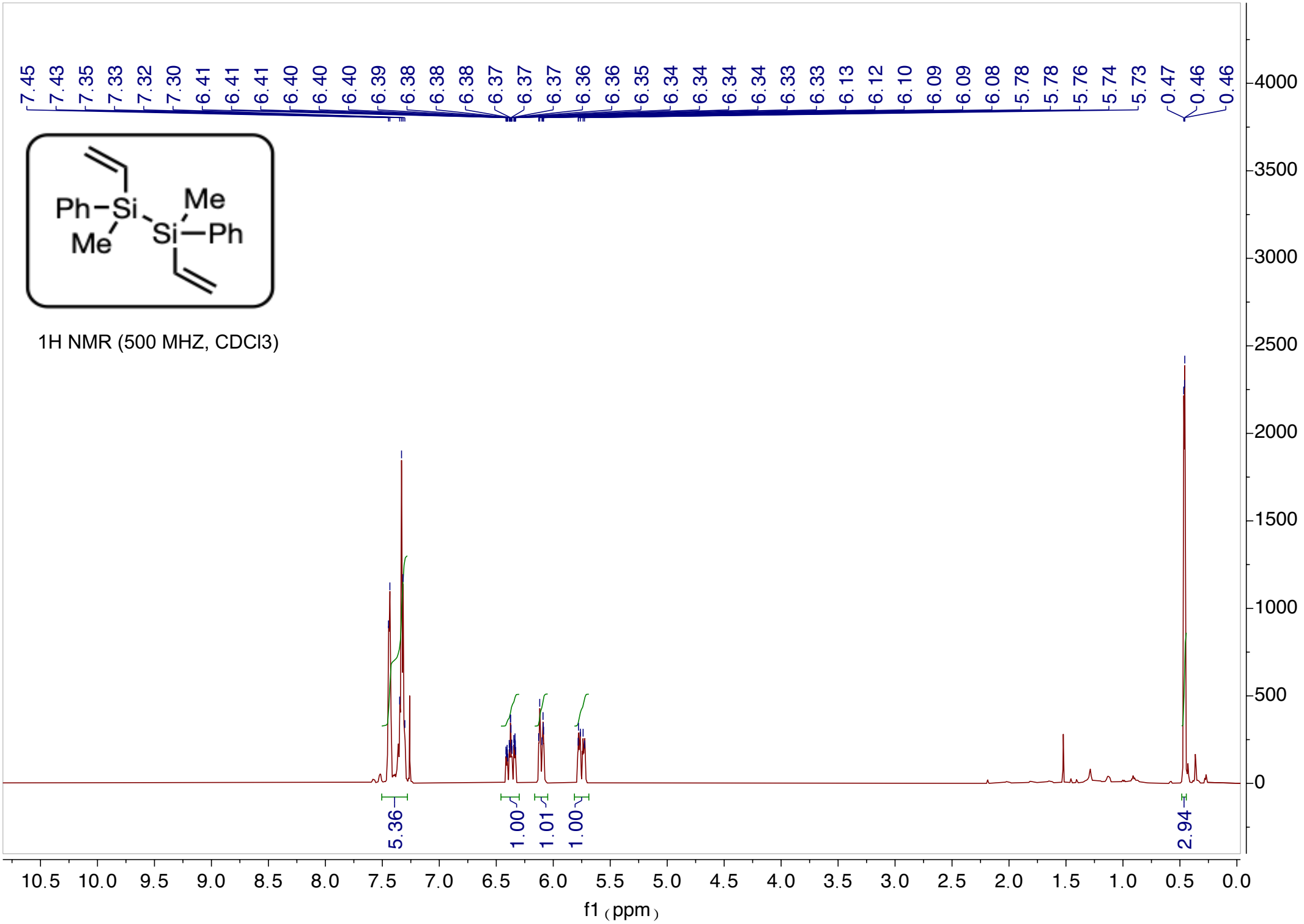

Ph
Ph
Si
Me
Si
Ph
Ph
Me
29Si NMR (99 MHZ, CDCl3)
-23.25
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

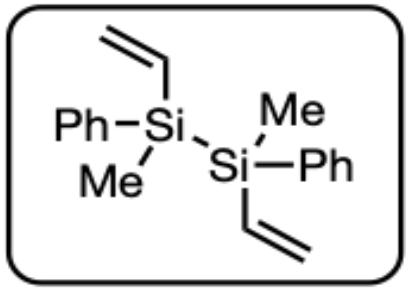

Me Me
Si
Si
Me Me
1H NMR (500 MHZ, CDCl3)
5.85 5.83 5.83 5.82 5.81 5.80 5.80 5.78 5.78 5.76
4.89 4.89 4.89 4.88 4.88 4.86 4.86 4.86 4.85 4.85 4.85 4.84 4.84 4.84 4.84 4.84
1.64 1.64 1.63 1.62 1.62 1.62
0.12 0.10 0.08 0.06 0.06 0.06 0.05
2.00
3.63
3.53
10.44
11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
11.5 11.0 10.5 10.0 9.5 9.0 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 -1.0 -1.5
f1 (ppm)



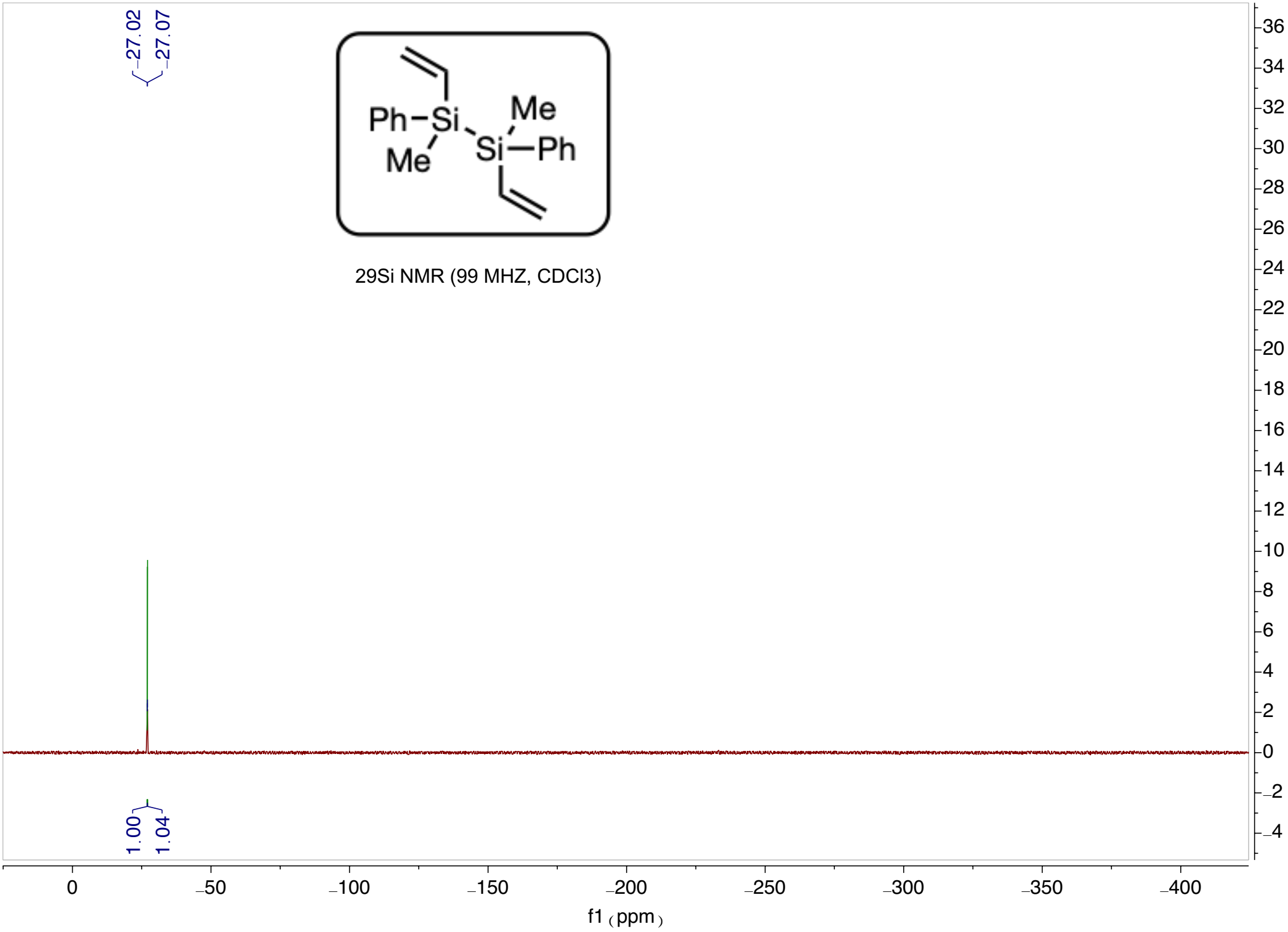
13C NMR (101 MHZ, CDCl3)

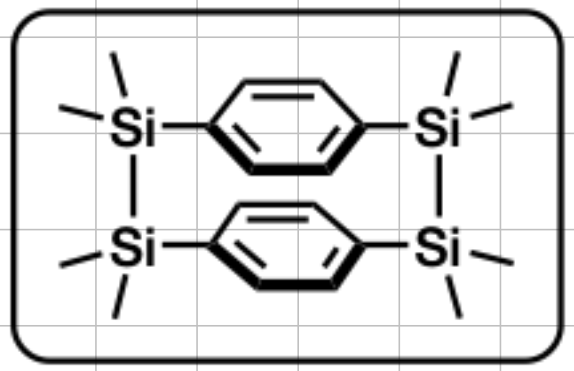

-18.25
Me Me
Si
Si
Me Me
29Si NMR (99 MHZ, CDCl3)
35
30
25
20
15
10
5
0
-5
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)


7.45 7.43 7.35 7.33 7.32 7.30 6.41 6.41 6.41 6.40 6.40 6.40 6.39 6.38 6.38 6.38 6.37 6.37 6.37 6.36 6.36 6.35 6.34 6.34 6.34 6.34 6.33 6.33 6.13 6.12 6.10 6.09 6.09 6.08 5.78 5.78 5.76 5.74 5.73 0.47 0.46 0.46
Ph
Si
Me
Me
Si
Ph
1H NMR (500 MHZ, CDCl3)
5.36
1.00
1.01
1.00
2.94
4000
3500
3000
2500
2000
1500
1000
500
0
10.5 10.0 9.5 9.0 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
f1 (ppm)



13C NMR (101 MHZ, CDCl3)


27.02
27.07
Ph–Si
Me
Me
Si–Ph
29Si NMR (99 MHZ, CDCl3)
1.00
1.04
36
34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
–2
–4
0
–50
–100
–150
–200
–250
–300
–350
–400
f1 (ppm)



1H NMR (500 MHZ, CDCl3)

760

0.38

from impurity: siloxane

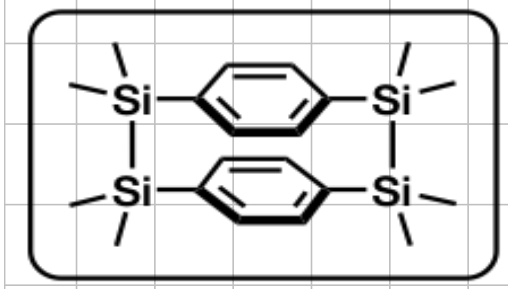
8.00

23.59

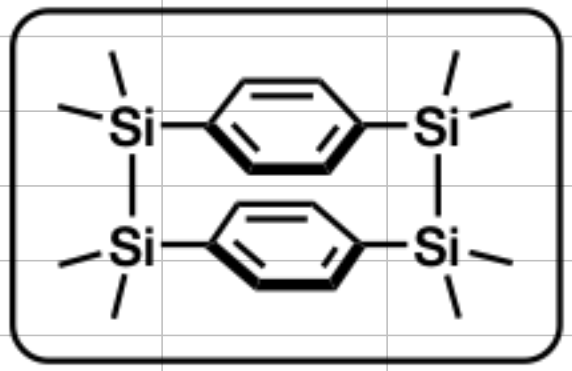
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

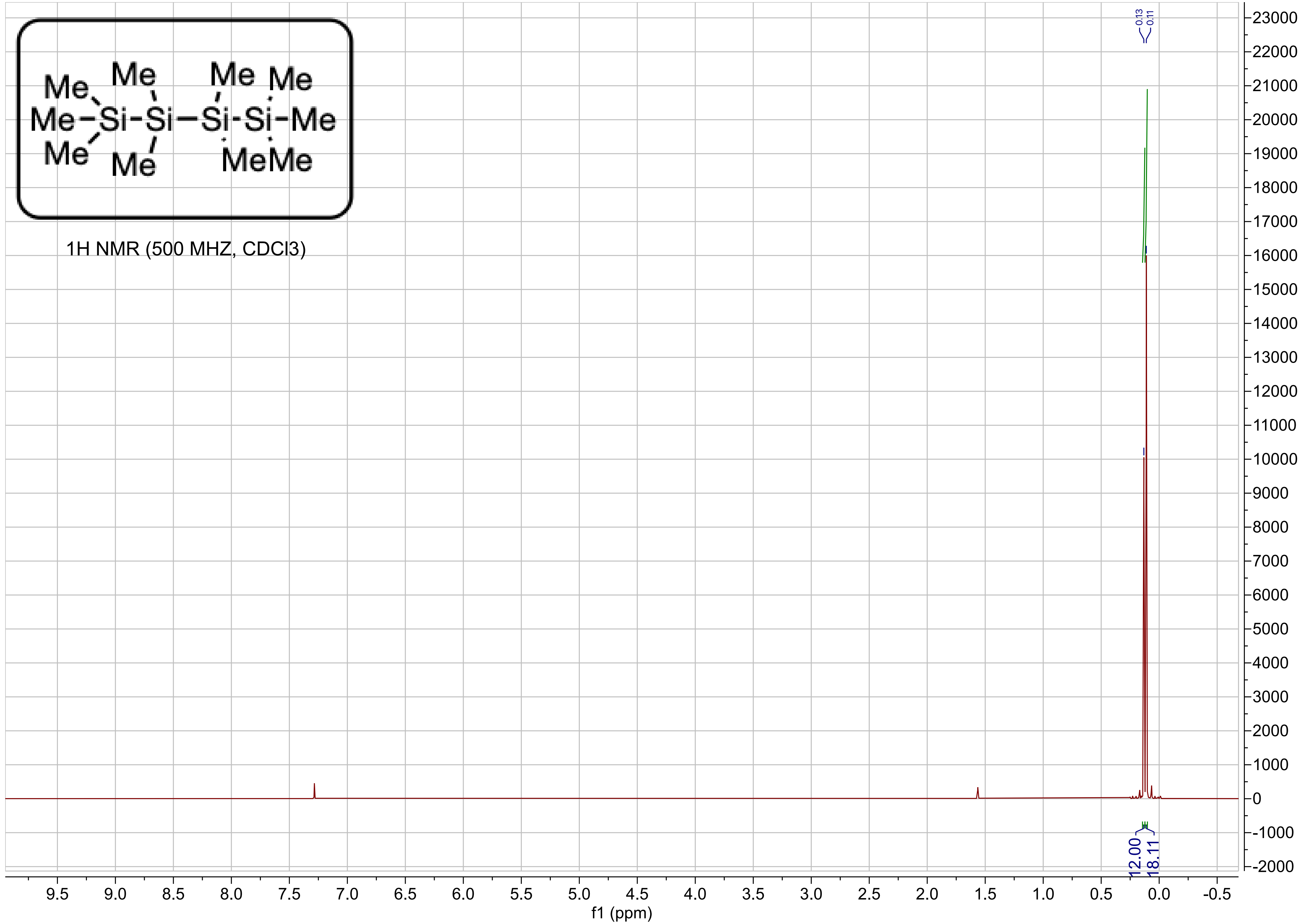
f1 (ppm)



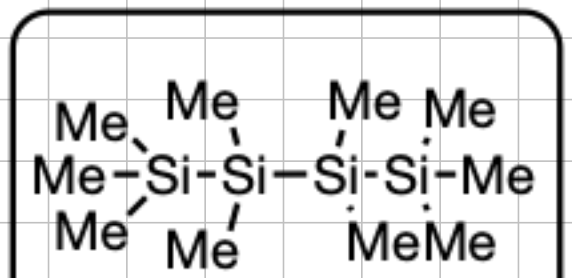
13C NMR (101 MHZ, CDCl3)



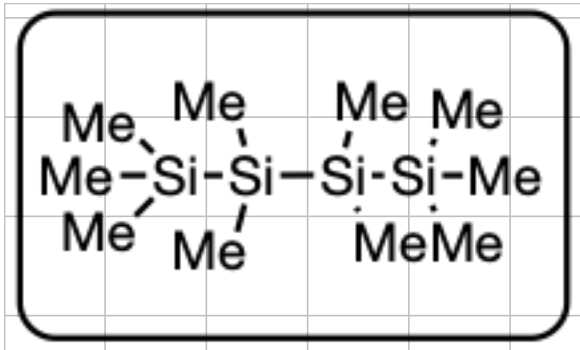
29Si NMR (99 MHZ, CDCl3)



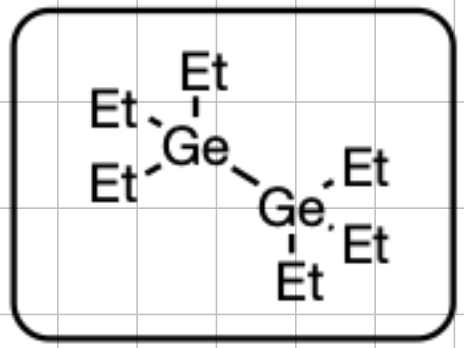
Me
Si
1H NMR (500 MHZ, CDCl3)
0.13
0.11
12.00
18.11
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
23000 22000 21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000



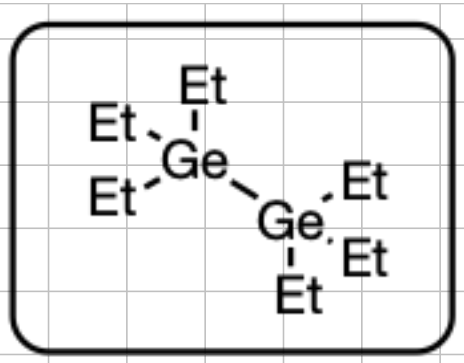
13C NMR (101 MHZ, CDCl3)



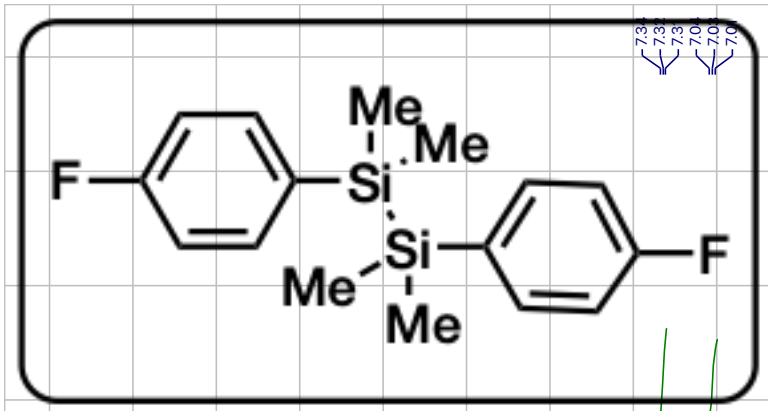
29Si NMR (99 MHZ, CDCl3)



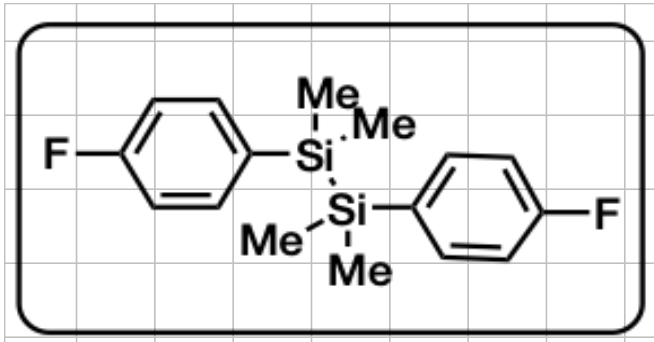
1H NMR (500 MHZ, CDCl3)



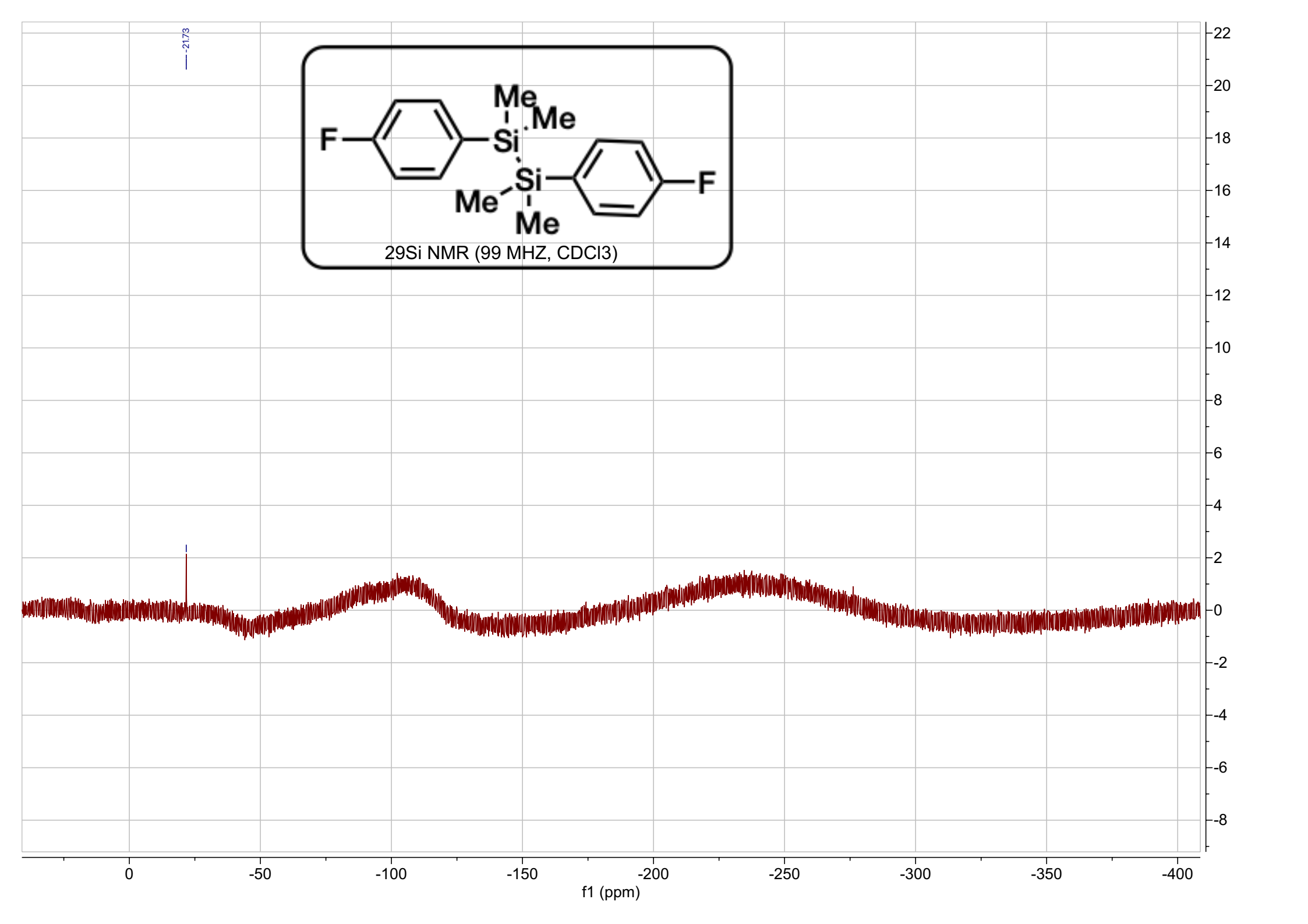
13C NMR (101 MHZ, CDCl3)

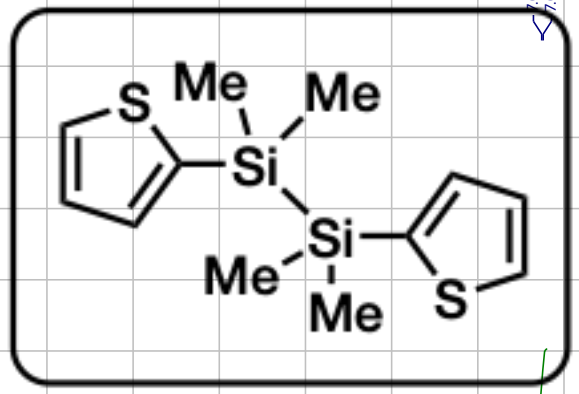


1H NMR (500 MHZ, CDCl3)



13C NMR (101 MHZ, CDCl3)


-21.73
Me
Me
F
Si
Si
F
Me
Me
29Si NMR (99 MHZ, CDCl3)
22
20
18
16
14
12
10
8
6
4
2
0
-2
-4
-6
-8
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

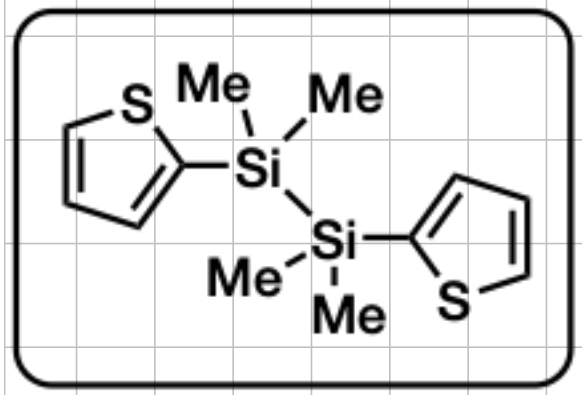


1H NMR (500 MHZ, CDCl3)

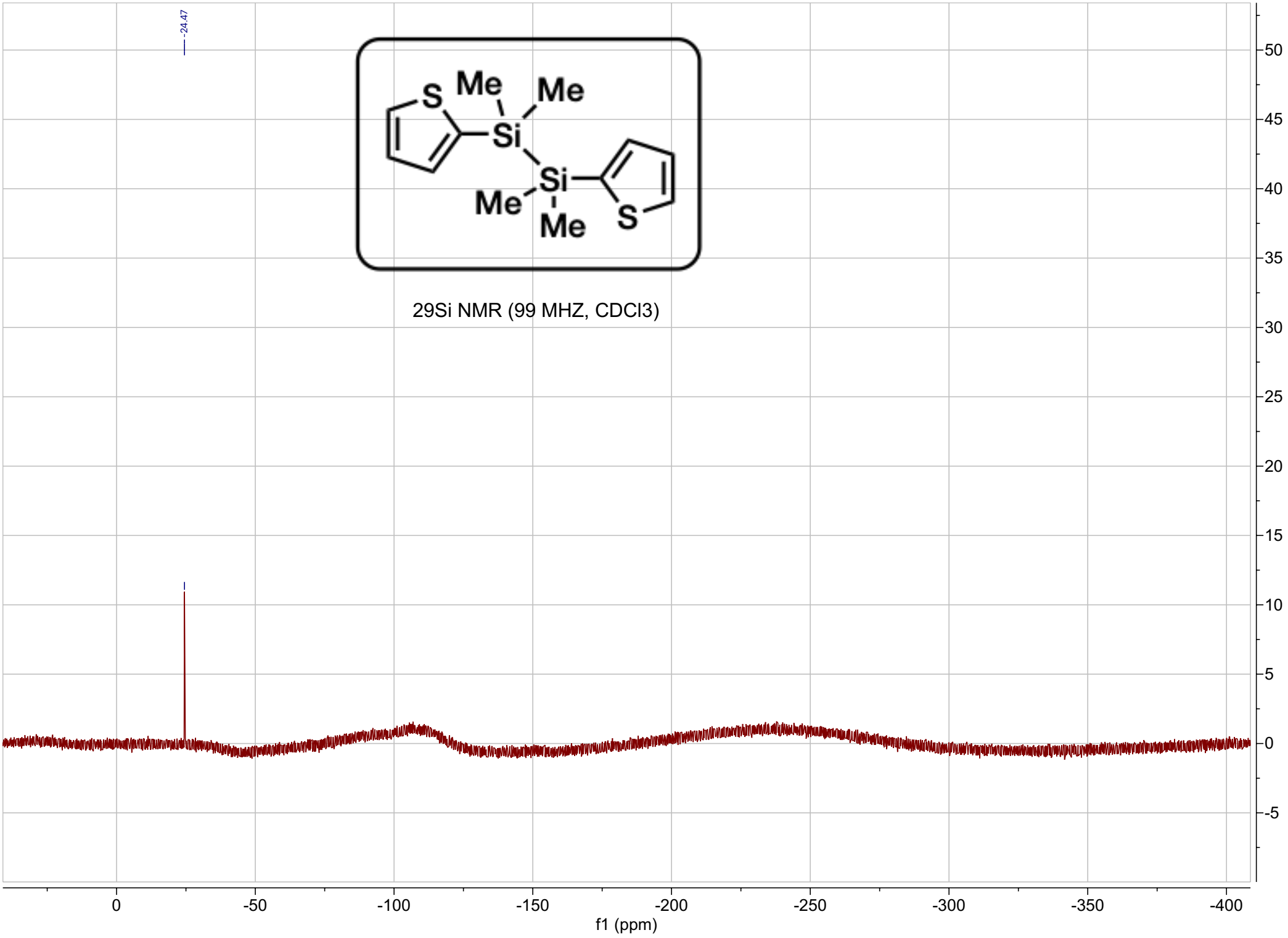
7.63
7.62
7.21
7.20
0.44

1.98
4.00
11.66

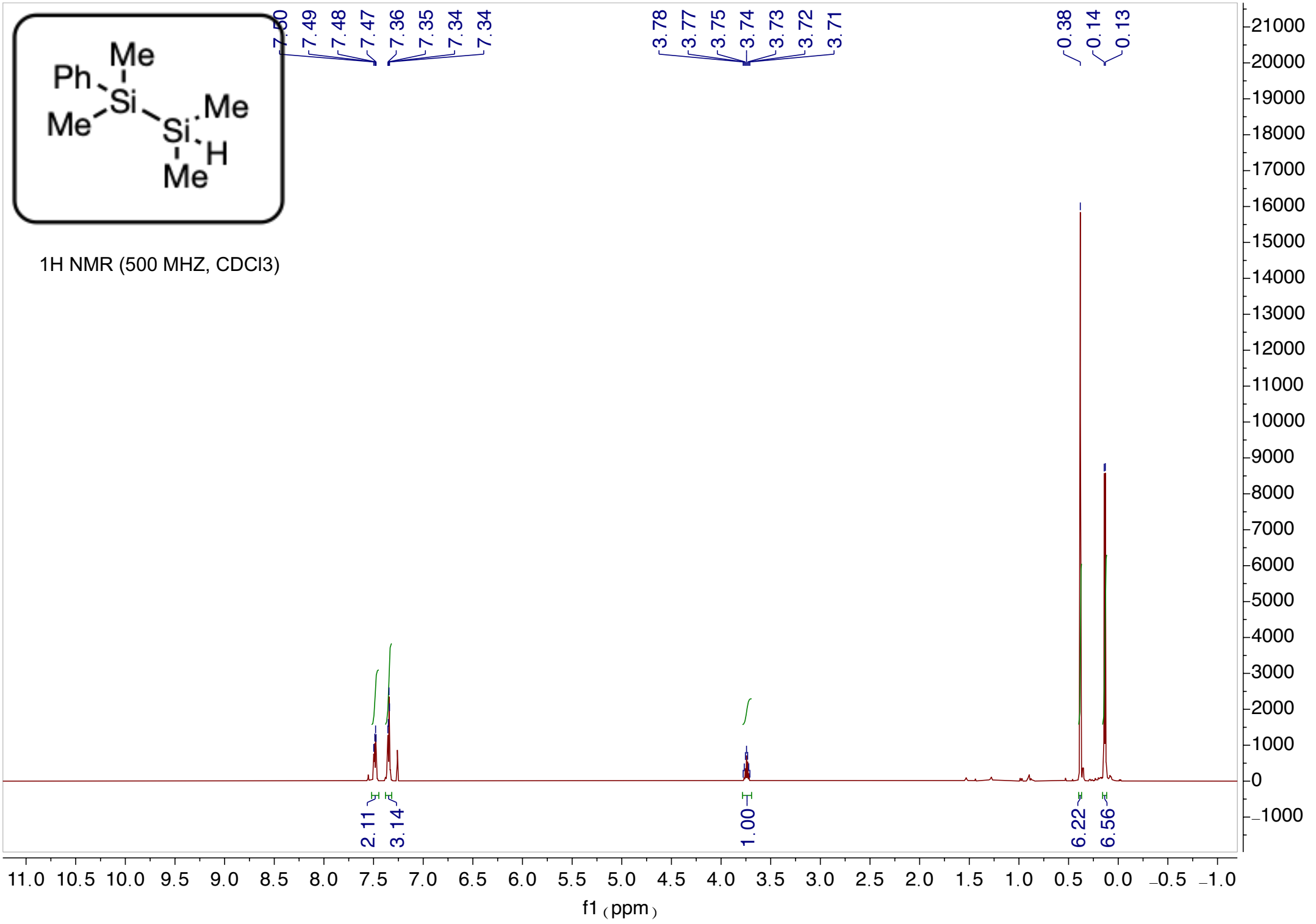
f1 (ppm)

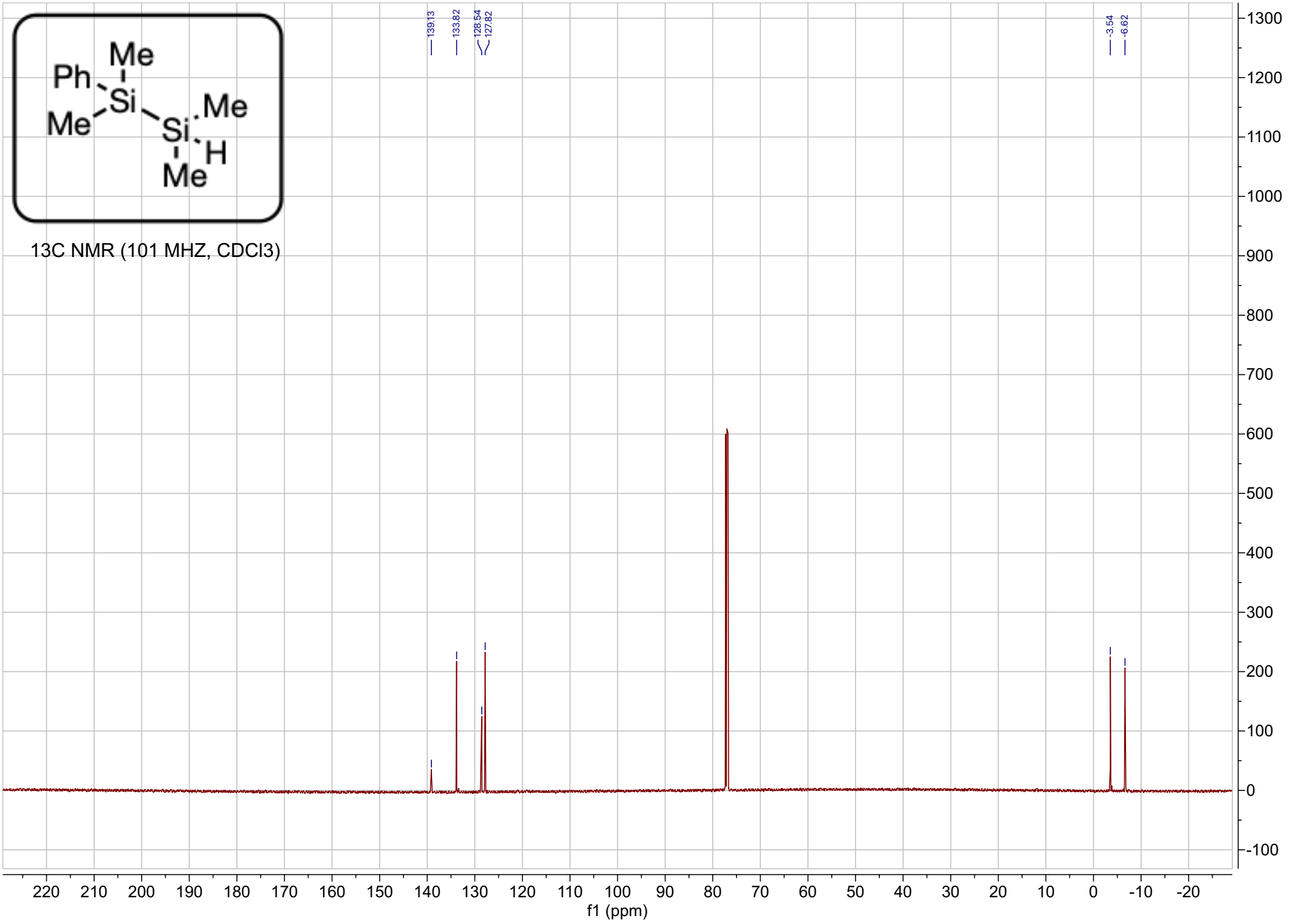


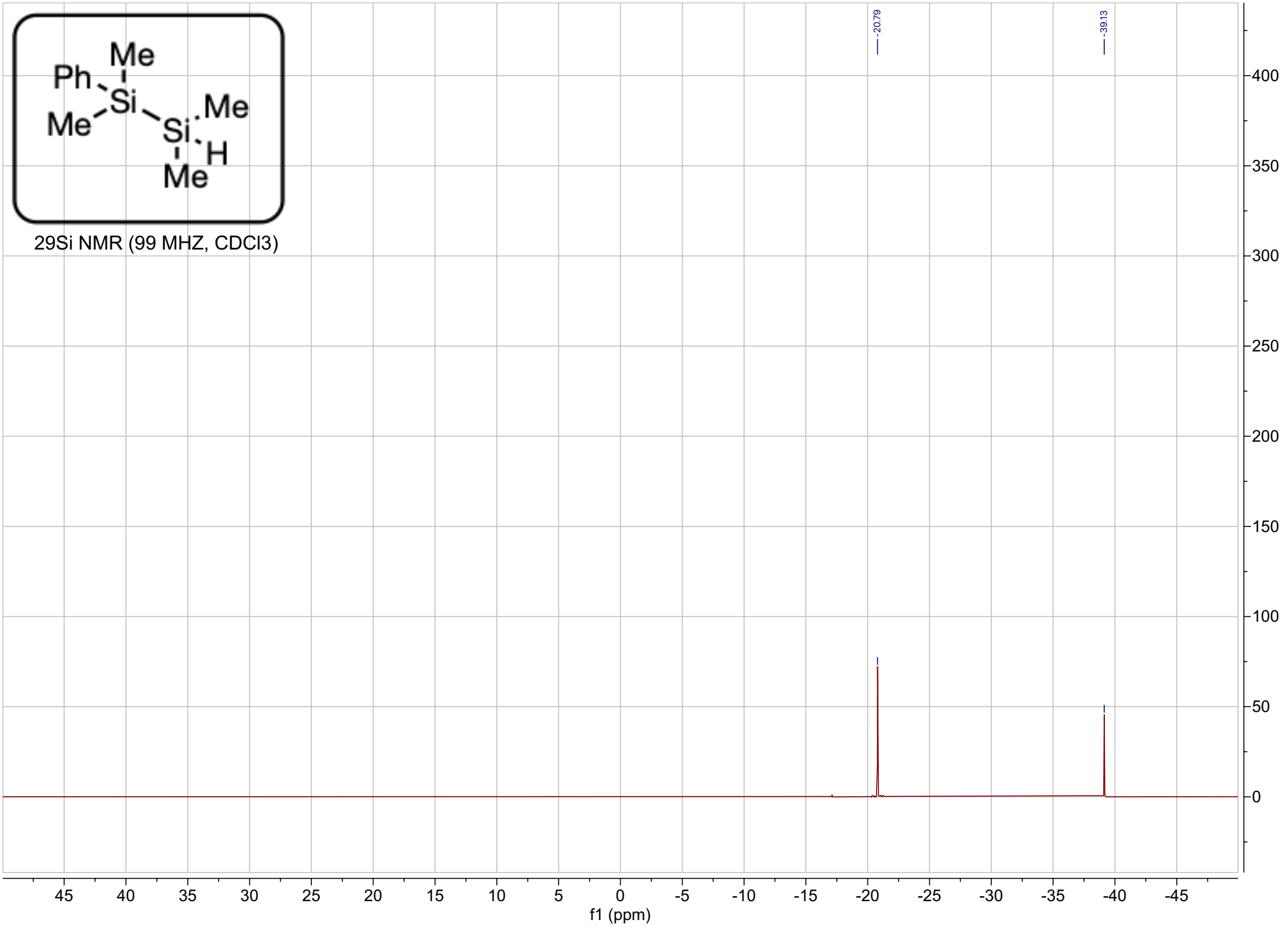
13C NMR (101 MHZ, CDCl3)

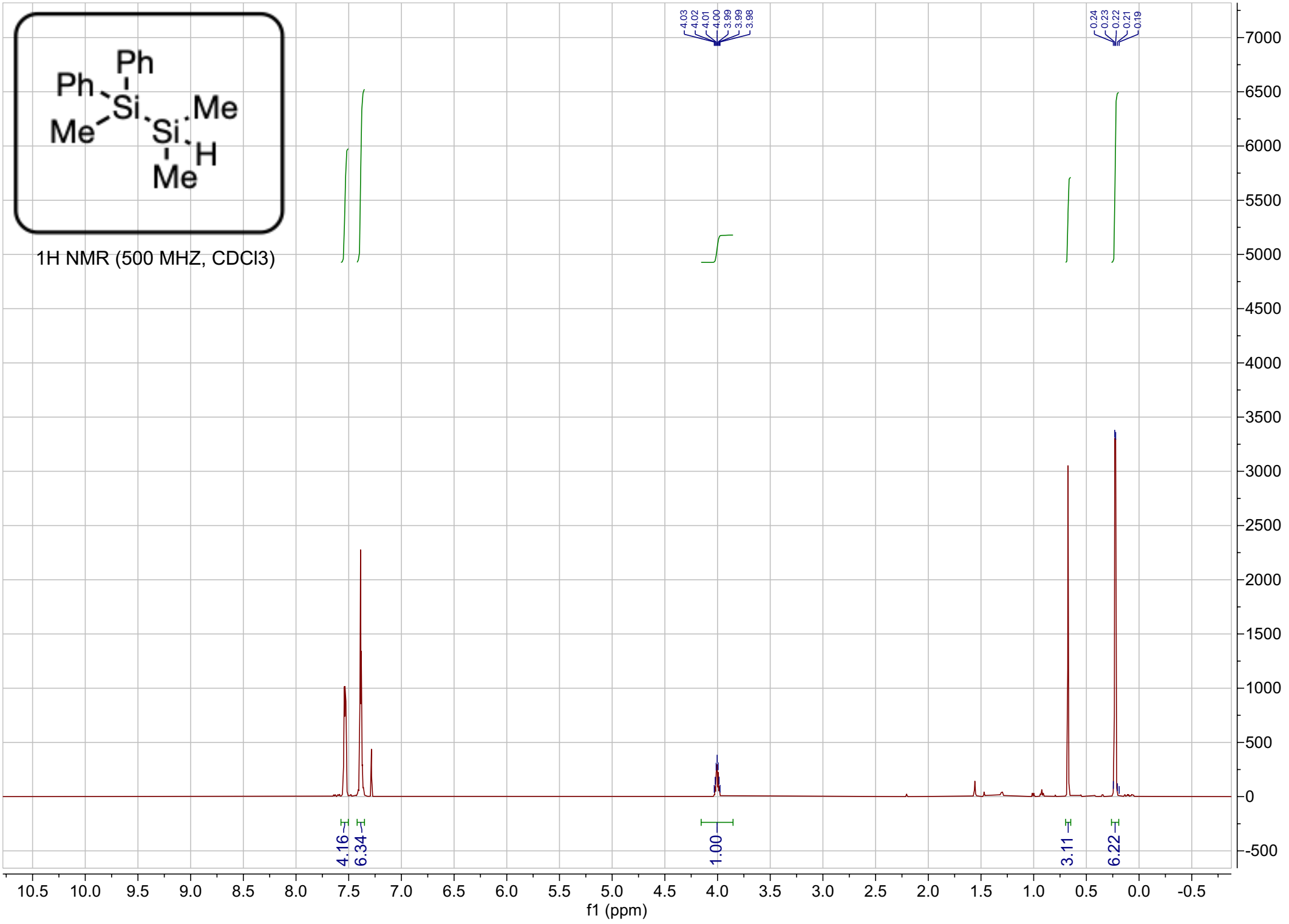


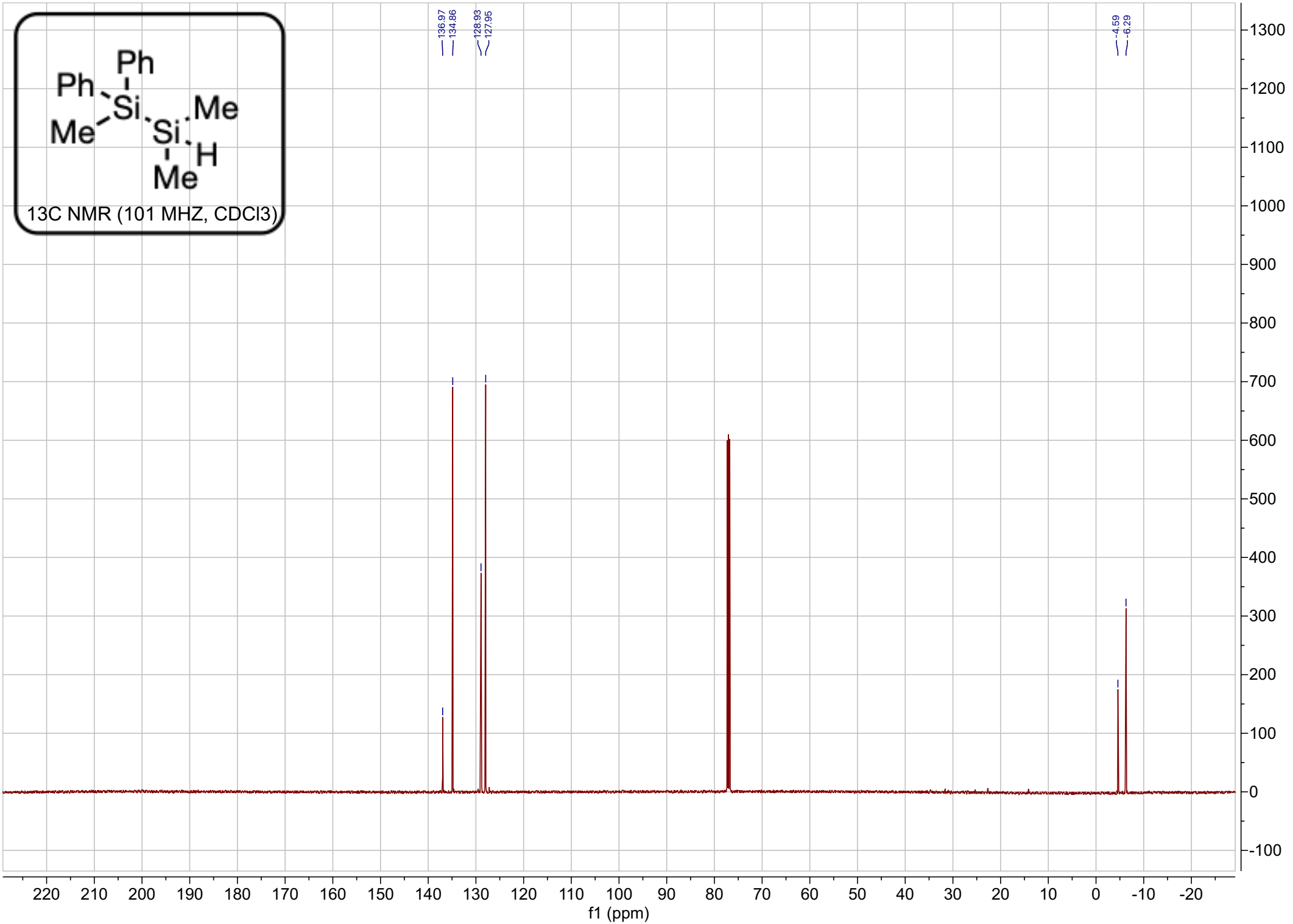
29Si NMR (99 MHZ, CDCl3)

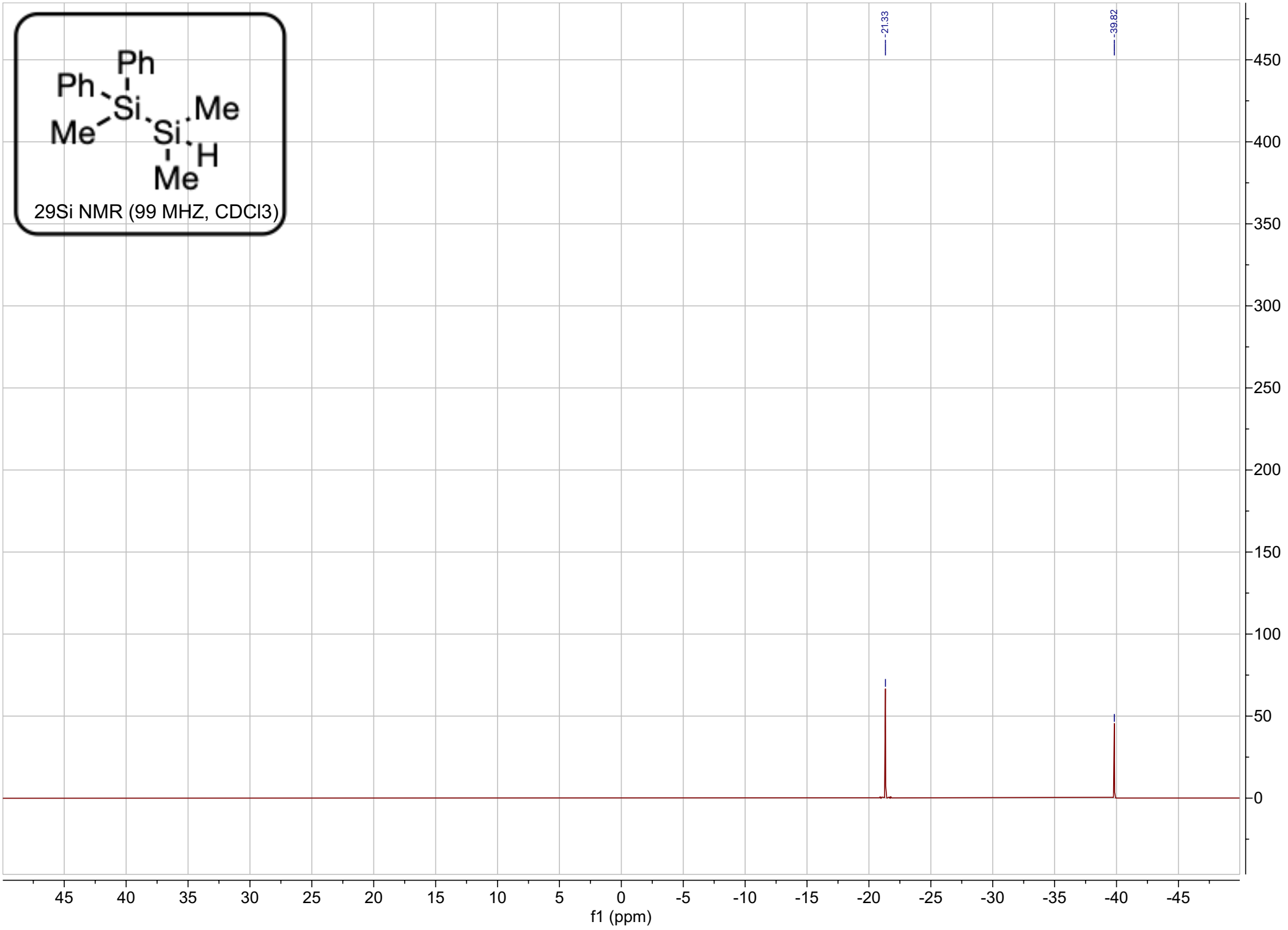

Me
Ph
Si
Me
Si
Me
H
Me
1H NMR (500 MHZ, CDCl3)
7.50
7.49
7.48
7.47
7.36
7.35
7.34
7.34
3.78
3.77
3.75
3.74
3.73
3.72
3.71
0.38
0.14
0.13
2.11
3.14
1.00
6.22
6.56
f1 (ppm)

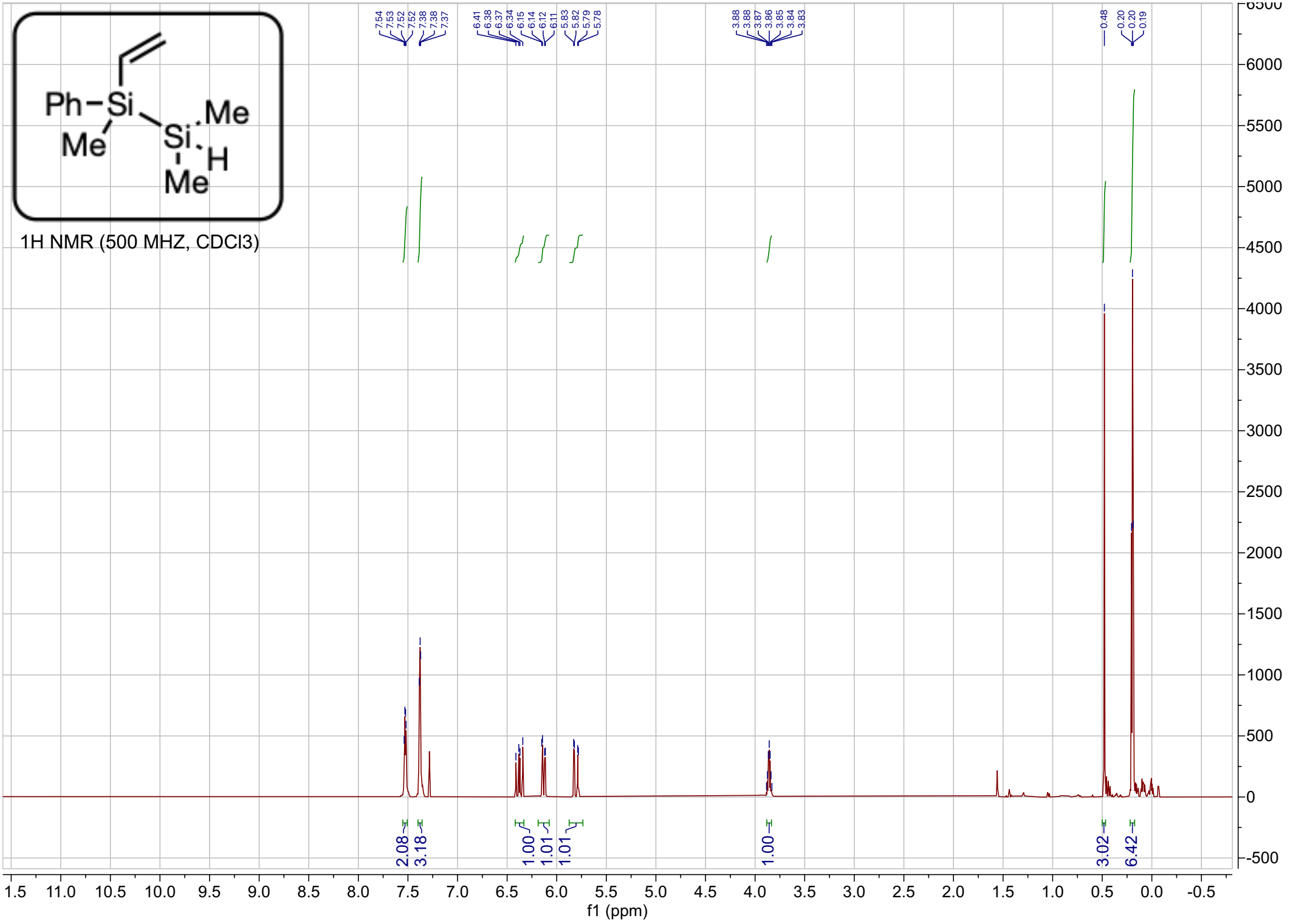

Ph
Me
Si
Me
Me
Si
Me
H
Me
13C NMR (101 MHZ, CDCl3)
139.13
133.82
128.54
127.82
-3.54
-6.62
f1 (ppm)

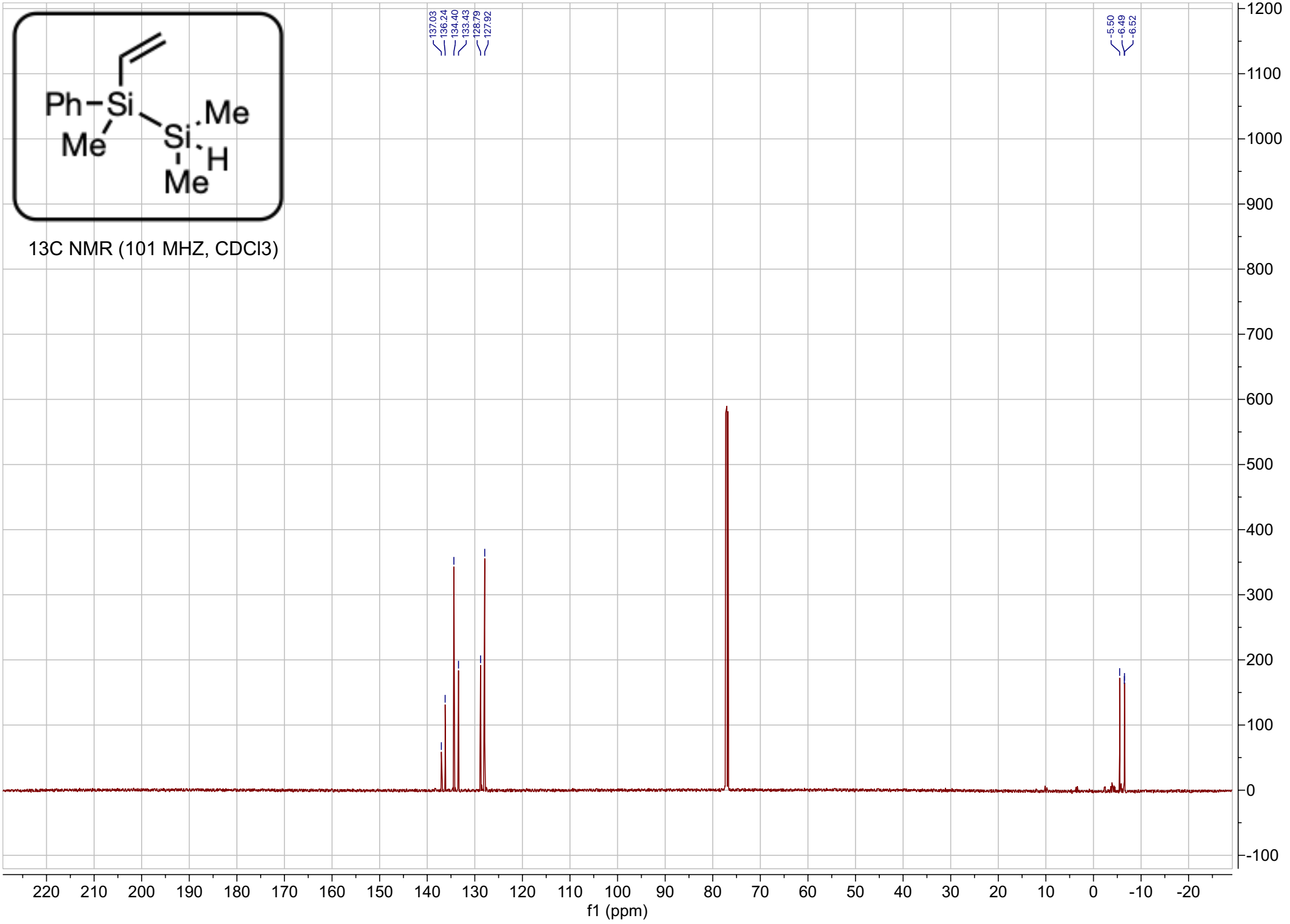

Me
Ph
Si
Me
Si
Me
H
Me
29Si NMR (99 MHZ, CDCl3)
-20.79
-39.13
400
350
300
250
200
150
100
50
0
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

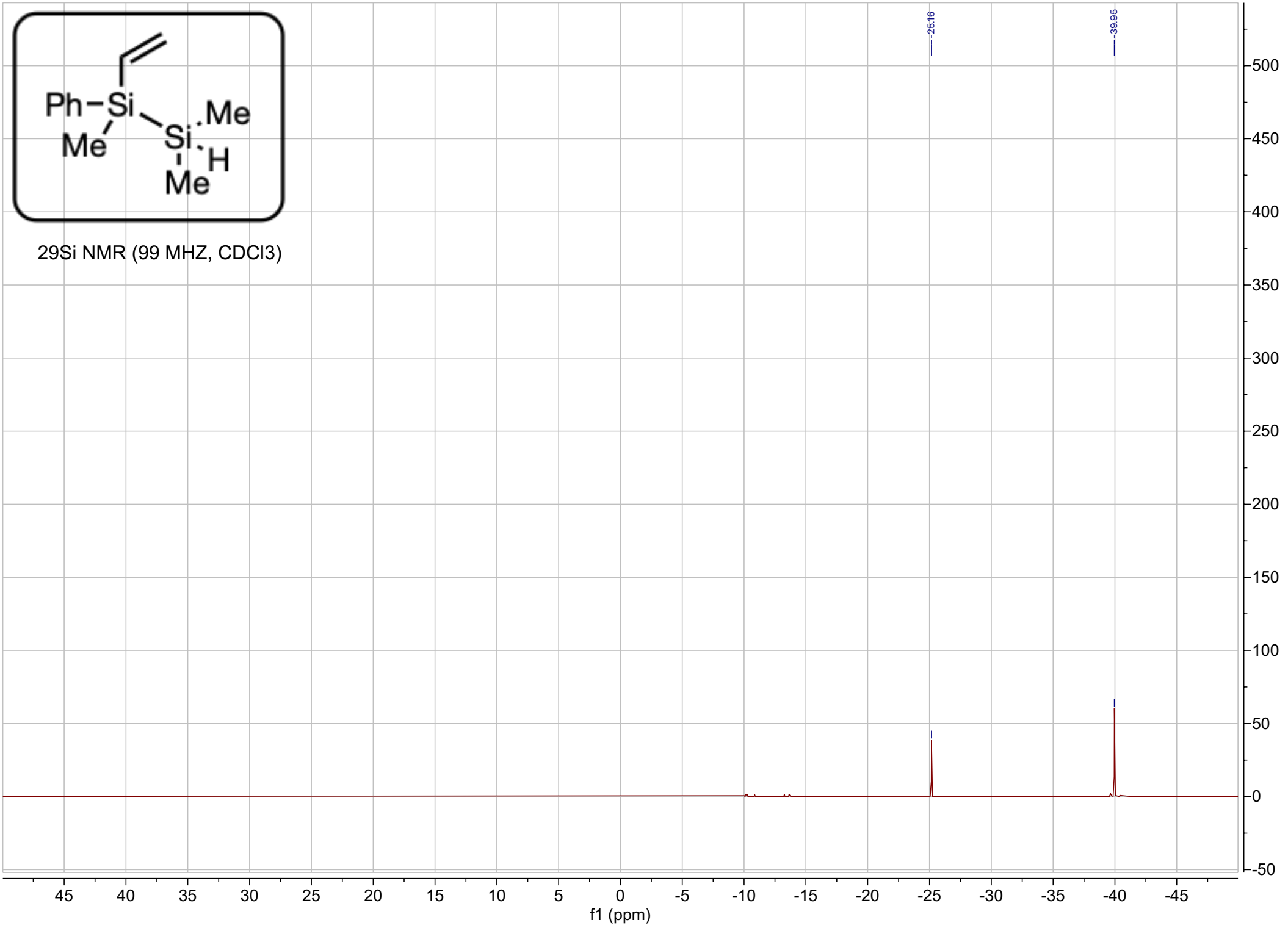

Ph
Ph
Si
Me
Me
Si
H
Me
1H NMR (500 MHZ, CDCl3)
4.03
4.02
4.01
4.00
3.99
3.99
3.98
0.24
0.23
0.22
0.21
0.19
4.16
6.34
1.00
3.11
6.22
f1 (ppm)

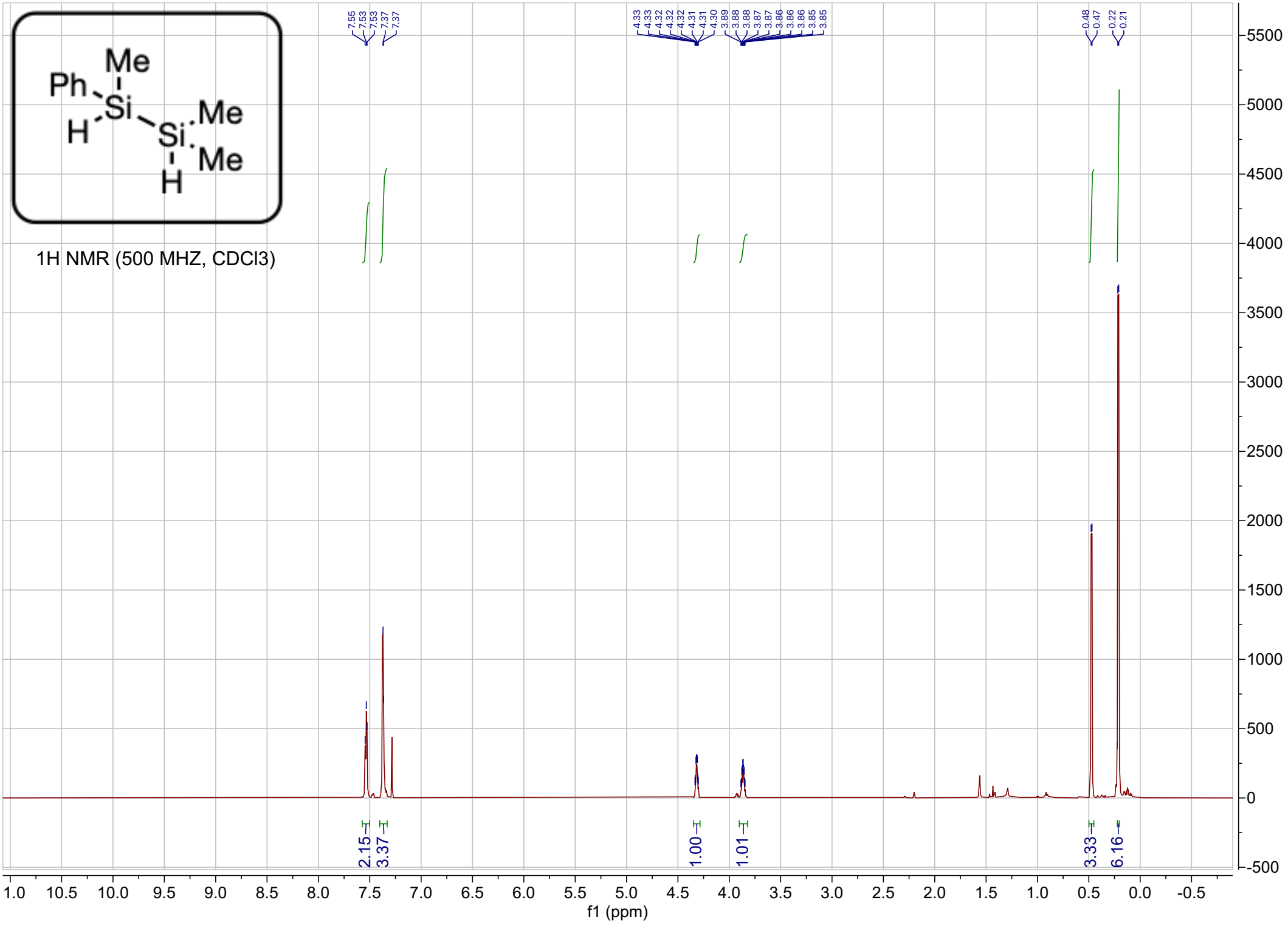

Ph
Ph
Si
Me
Me
Si
Me
H
Me
13C NMR (101 MHZ, CDCl3)
136.97
134.86
128.93
127.95
-4.59
-6.29
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

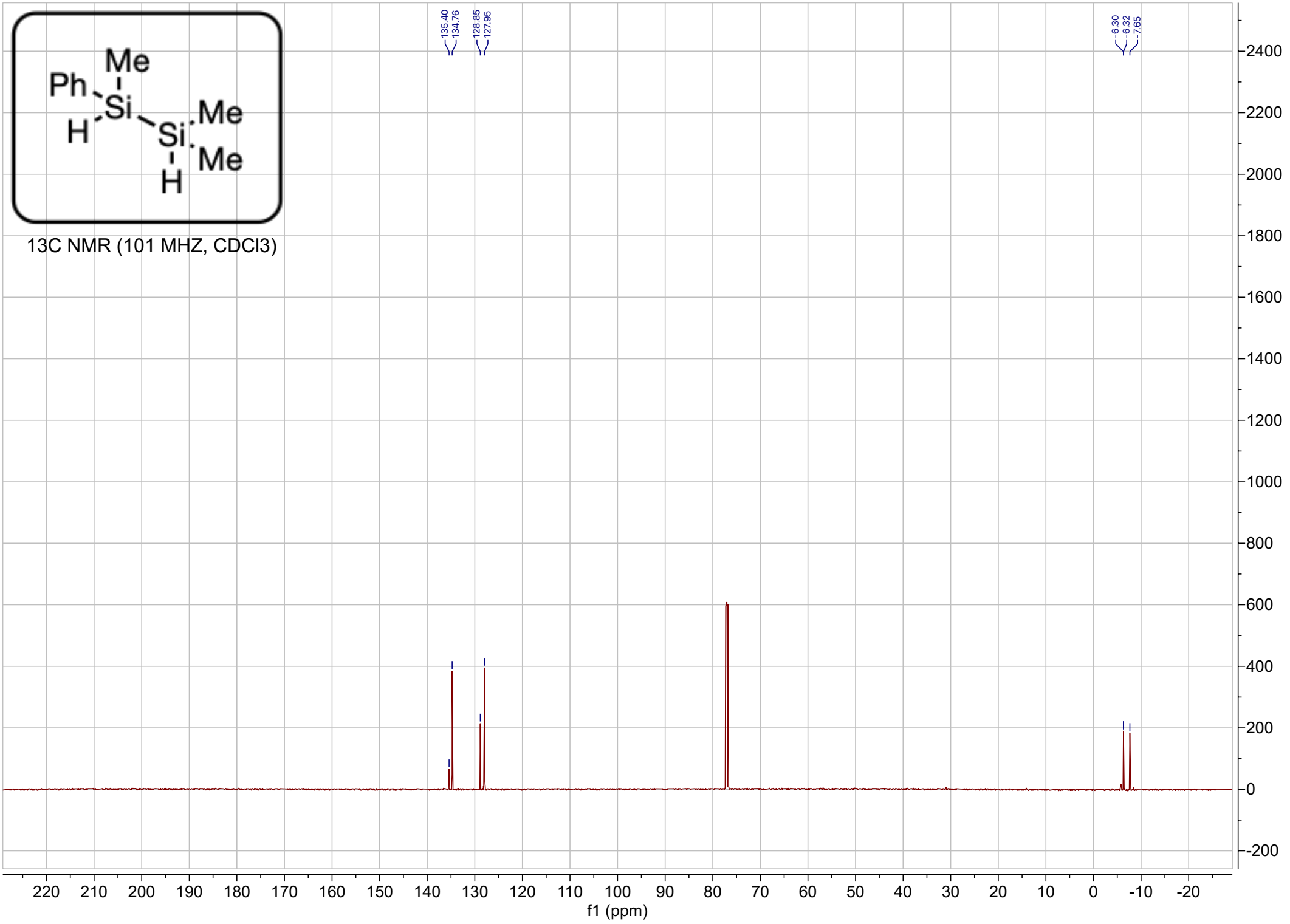

Ph
Ph
Si
Me
Me
Si
H
Me
29Si NMR (99 MHZ, CDCl3)
-21.33
-39.82
450
400
350
300
250
200
150
100
50
0
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

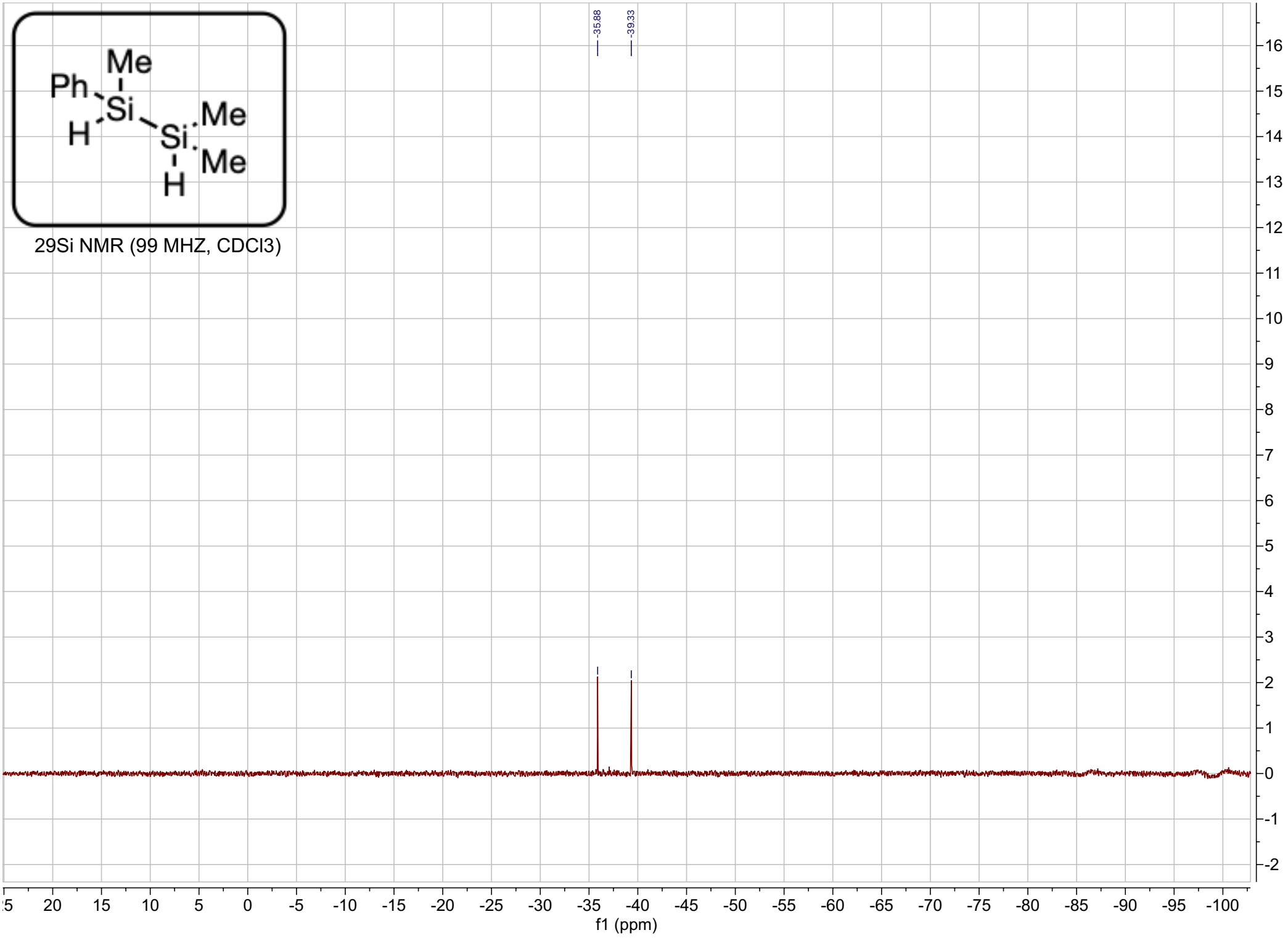

Ph
Si
Me
Me
Si
Me
H
1H NMR (500 MHZ, CDCl3)
7.54
7.53
7.52
7.52
7.38
7.38
7.37
6.41
6.38
6.37
6.34
6.15
6.14
6.12
6.11
5.83
5.82
5.79
5.78
3.88
3.88
3.87
3.86
3.85
3.84
3.83
0.48
0.20
0.20
0.19
2.08
3.18
1.00
1.01
1.01
1.00
3.02
6.42
f1 (ppm)

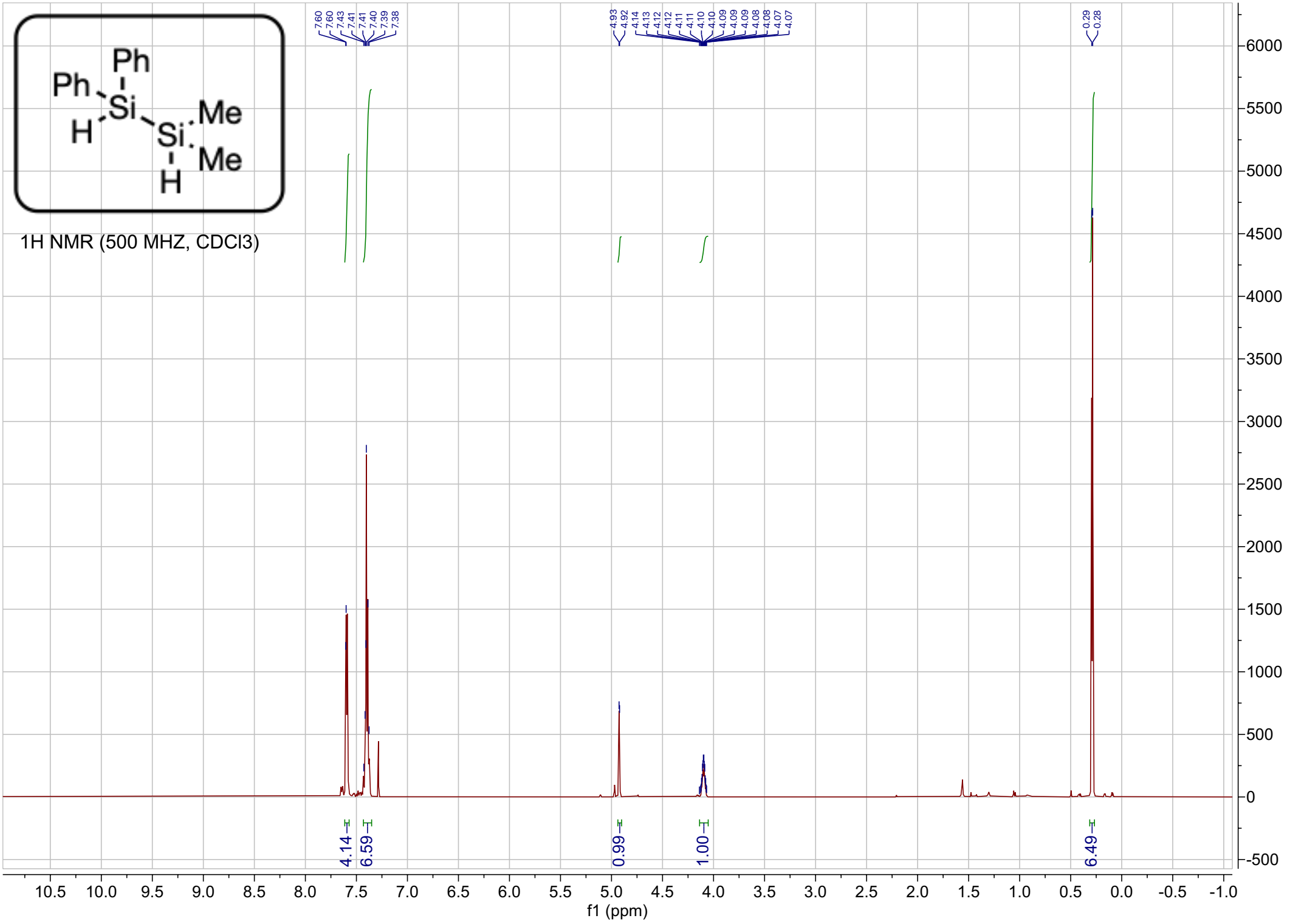

Ph
Si
Me
Me
Si
Me
H
Me
13C NMR (101 MHZ, CDCl3)
137.03
136.24
134.40
133.43
128.79
127.92
-5.50
-6.49
-6.52
f1 (ppm)

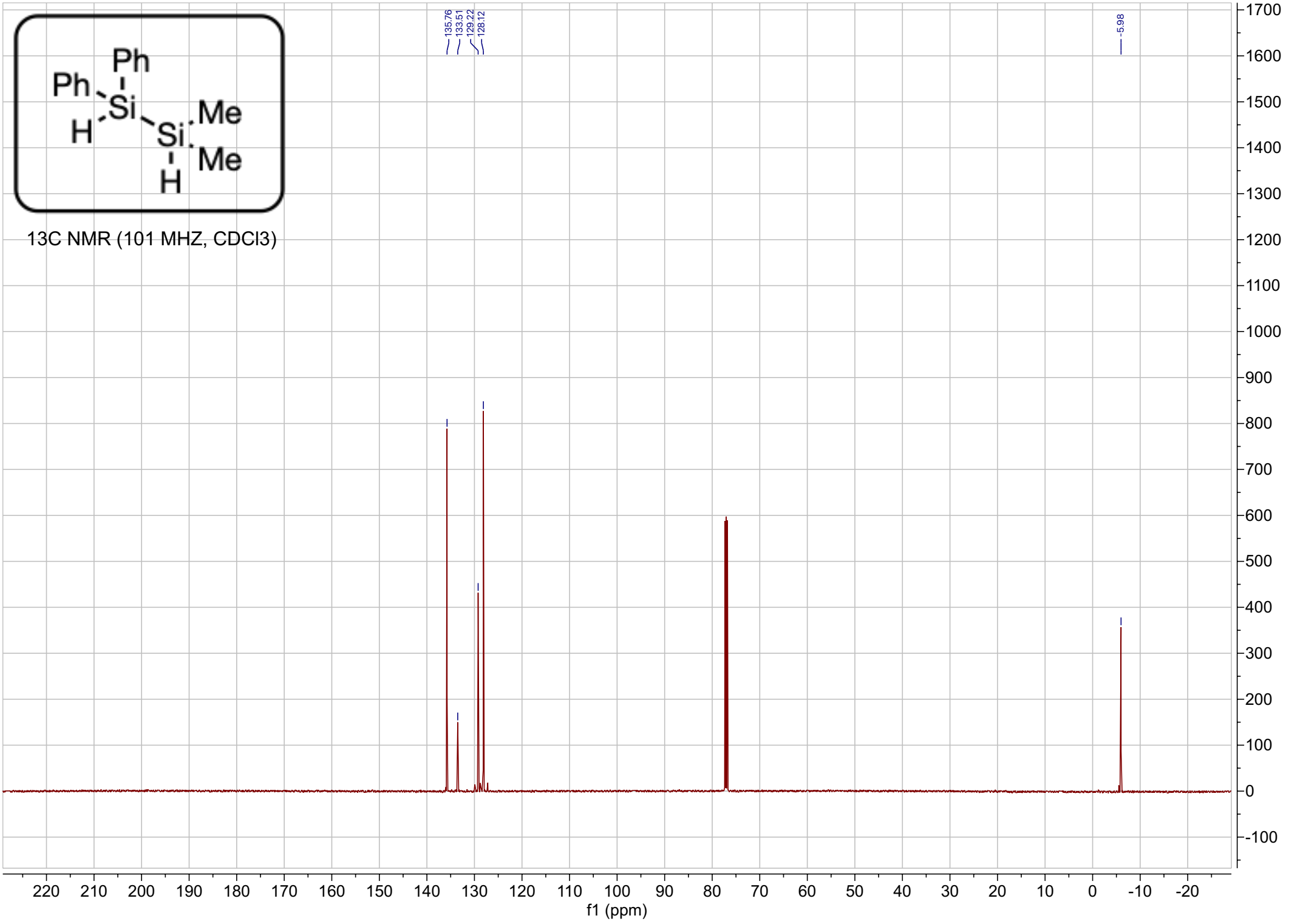

Ph
Si
Me
Me
Si
Me
H
Me
29Si NMR (99 MHZ, CDCl3)
-25.16
-39.95
500
450
400
350
300
250
200
150
100
50
0
-50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

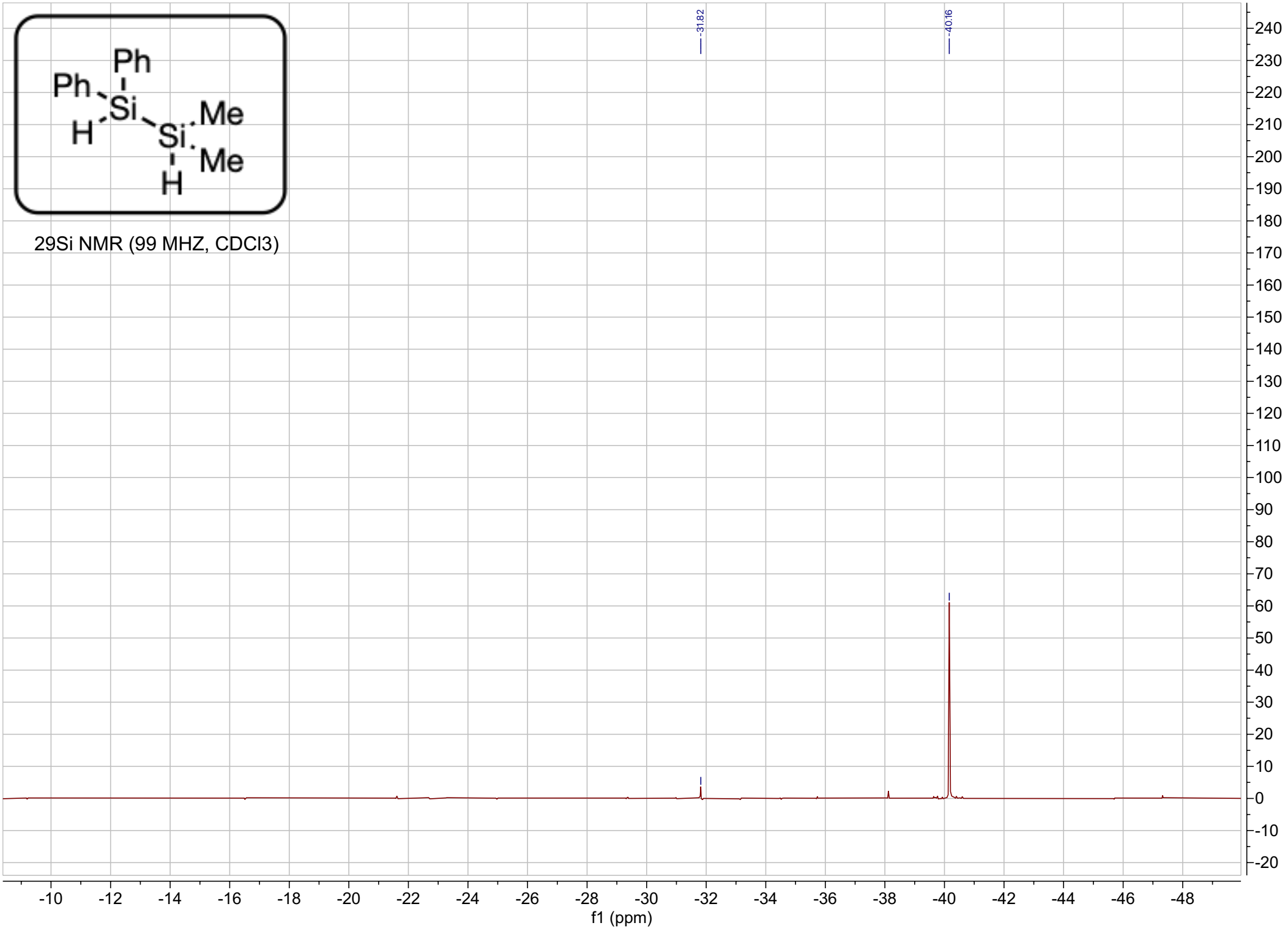

Me
Ph
Si
H
Si
Me
Me
H
1H NMR (500 MHZ, CDCl3)
7.55
7.53
7.53
7.37
7.37
4.33
4.33
4.32
4.32
4.32
4.31
4.31
4.30
3.89
3.88
3.88
3.87
3.87
3.86
3.86
3.86
3.85
3.85
0.48
0.47
0.22
0.21
2.15
3.37
1.00
1.01
3.33
6.16
f1 (ppm)

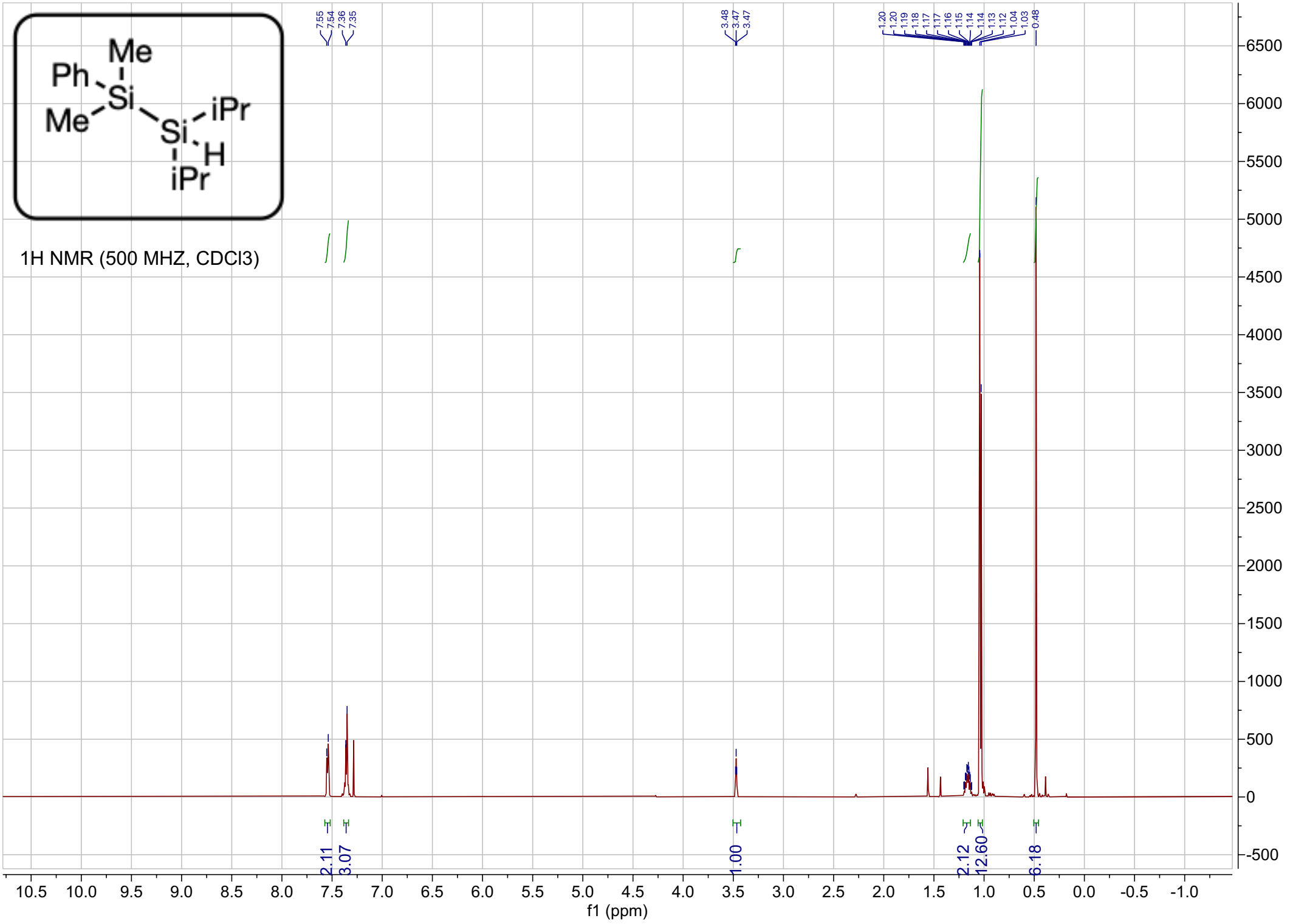

Me
Ph
Si
H
Si
Me
Me
H
13C NMR (101 MHZ, CDCl3)
135.40
134.76
128.85
127.95
-6.30
-6.32
-7.65
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

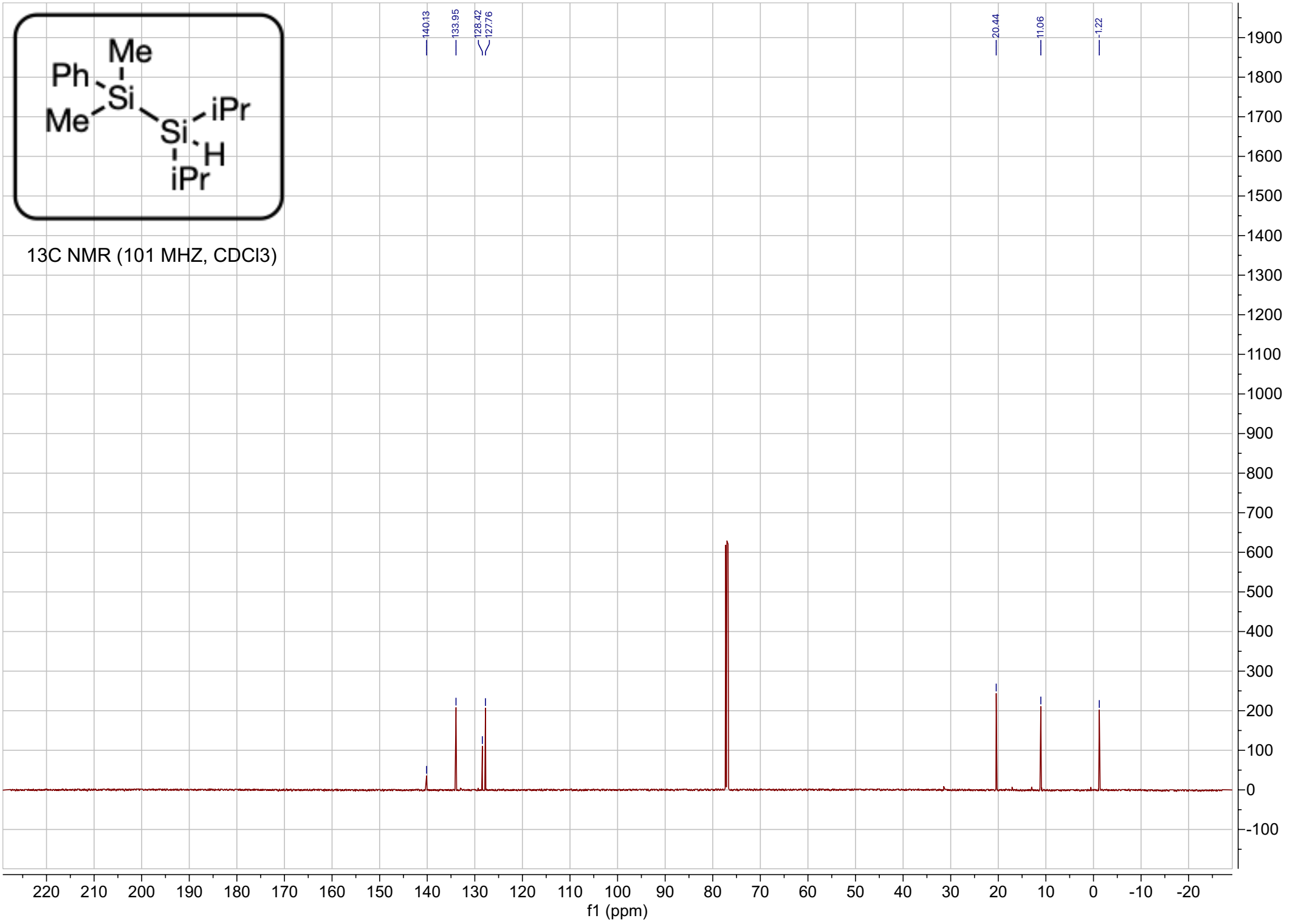

Me
Ph
Si
H
Si
Me
Me
H
29Si NMR (99 MHZ, CDCl3)
-35.88
-39.33
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
f1 (ppm)

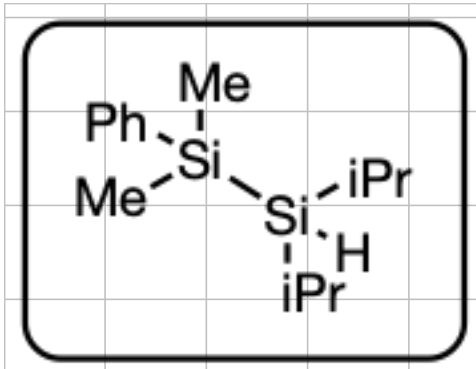

Ph
Ph
Si
H
Si
Me
Me
H
1H NMR (500 MHZ, CDCl3)
7.60
7.60
7.43
7.41
7.41
7.40
7.39
7.38
4.93
4.92
4.14
4.13
4.12
4.12
4.11
4.11
4.10
4.10
4.09
4.09
4.09
4.08
4.08
4.07
4.07
0.29
0.28
4.14
6.59
0.99
1.00
6.49
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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
-1.0
f1 (ppm)


Ph
Ph
Si
H
Si
Me
Me
H
13C NMR (101 MHZ, CDCl3)
135.76
133.51
129.22
128.12
-5.98
f1 (ppm)

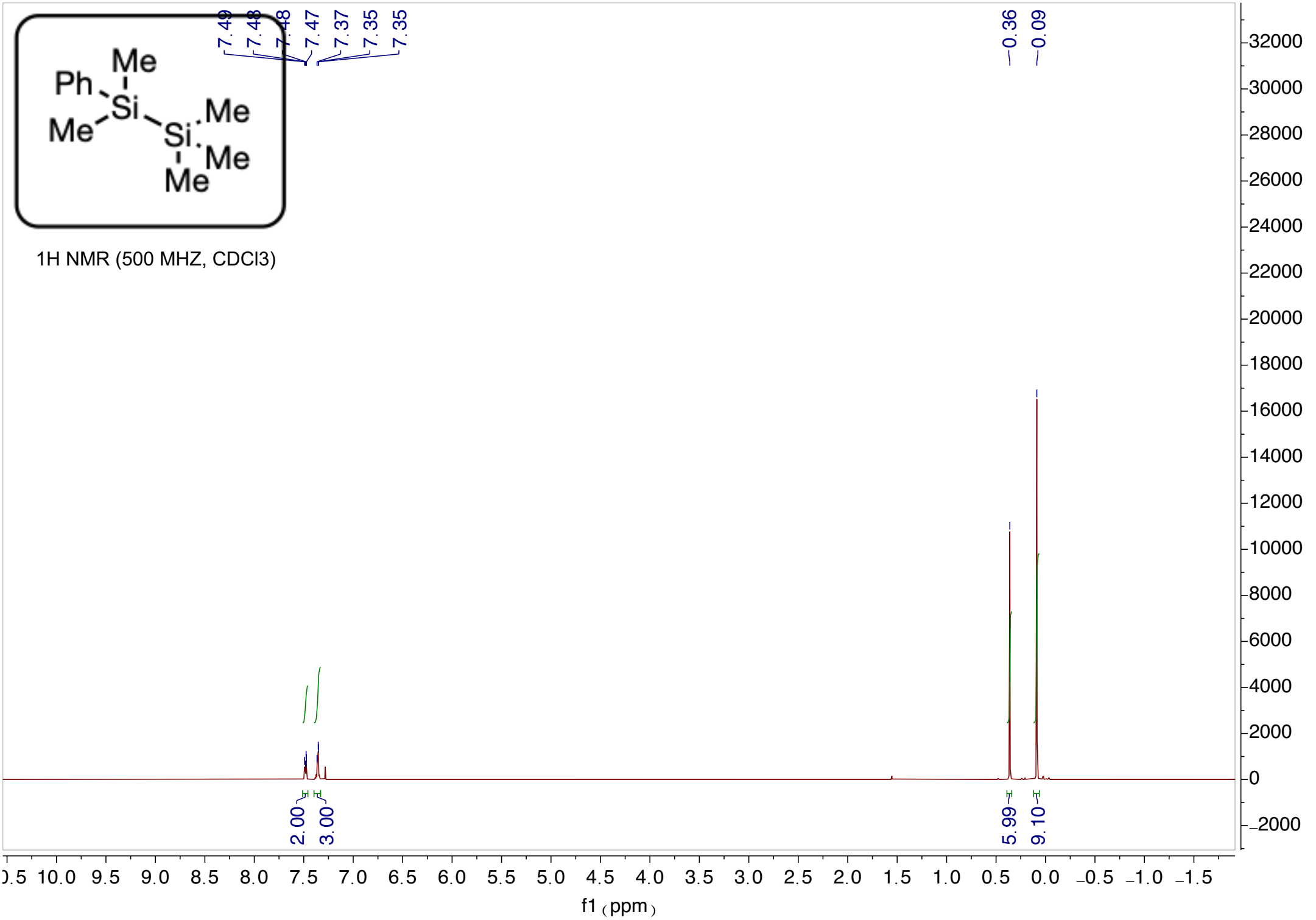

Ph
Ph
Si
H
Si
Me
Me
H
29Si NMR (99 MHZ, CDCl3)
-31.82
-40.16
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-10
-12
-14
-16
-18
-20
-22
-24
-26
-28
-30
-32
-34
-36
-38
-40
-42
-44
-46
-48
f1 (ppm)


Me
Ph
Si
Me
Si
iPr
H
iPr
1H NMR (500 MHZ, CDCl3)
7.55
7.54
7.36
7.35
3.48
3.47
3.47
1.20
1.20
1.19
1.18
1.17
1.17
1.16
1.15
1.14
1.14
1.13
1.12
1.04
1.03
0.48
2.11
3.07
1.00
2.12
12.60
6.18
f1 (ppm)

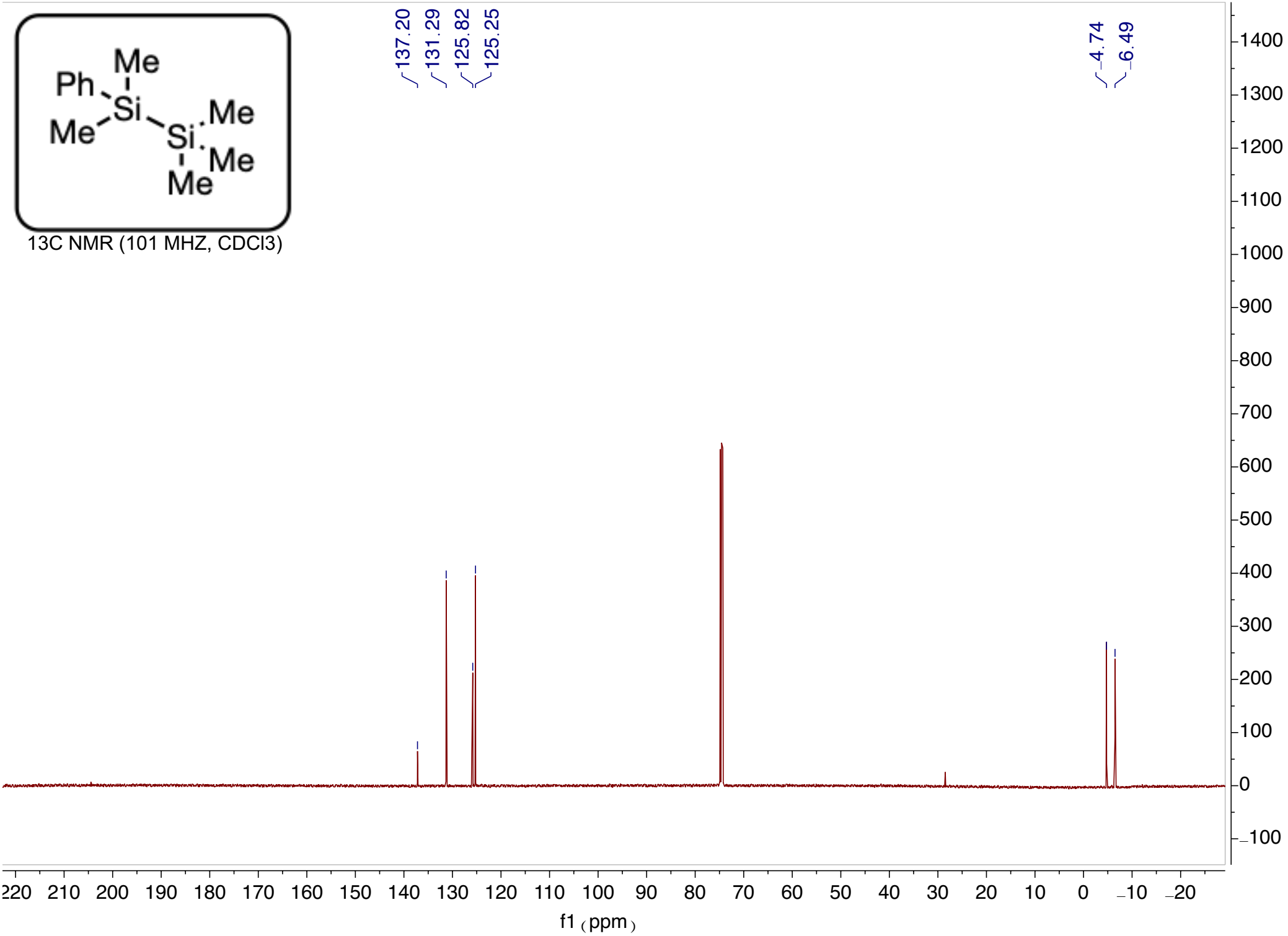

Me
Ph
Si
Me
Si
iPr
H
iPr
13C NMR (101 MHZ, CDCl3)
140.13
133.95
128.42
127.76
20.44
11.06
-1.22
f1 (ppm)

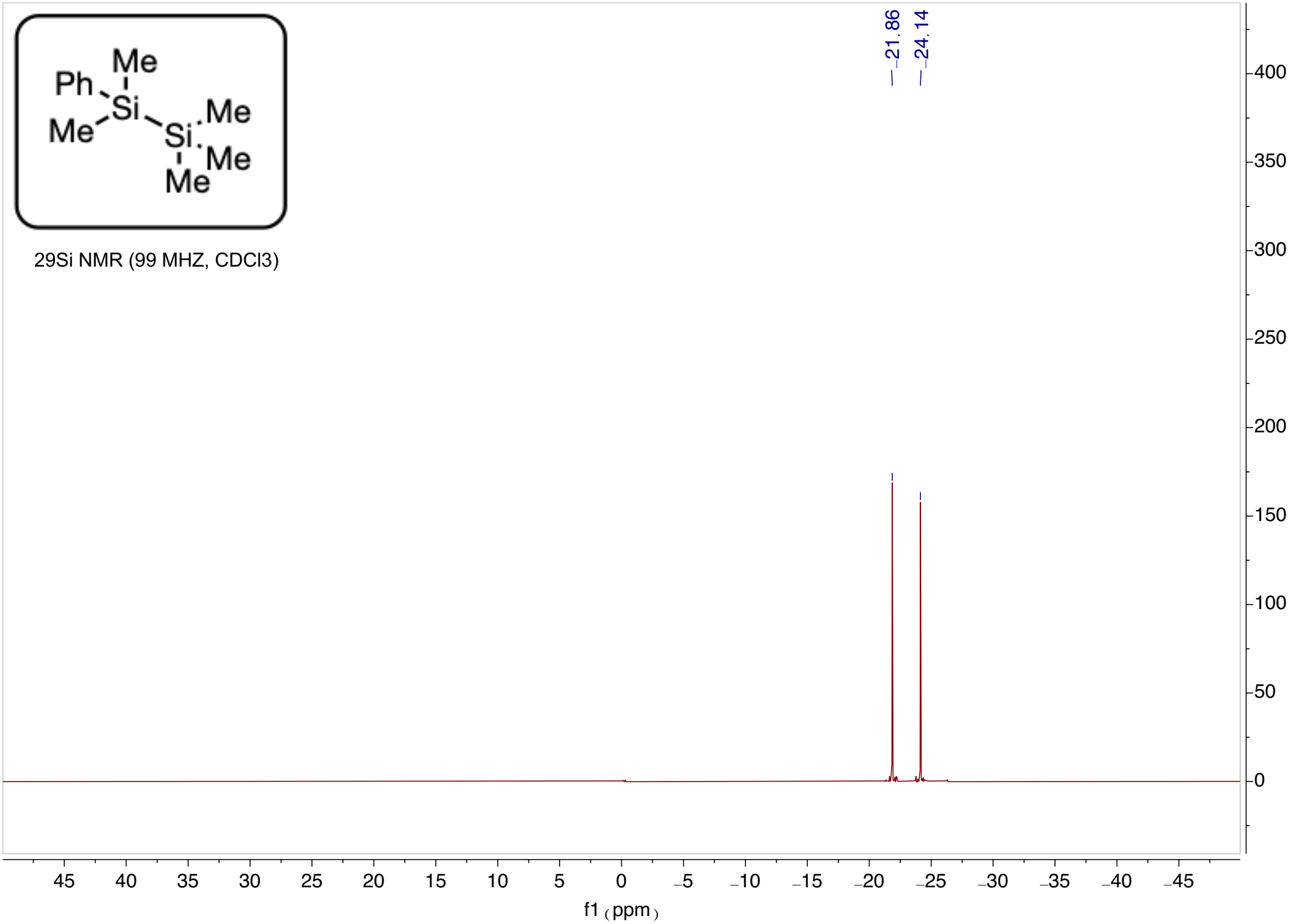


29Si NMR (99 MHZ, CDCl3)

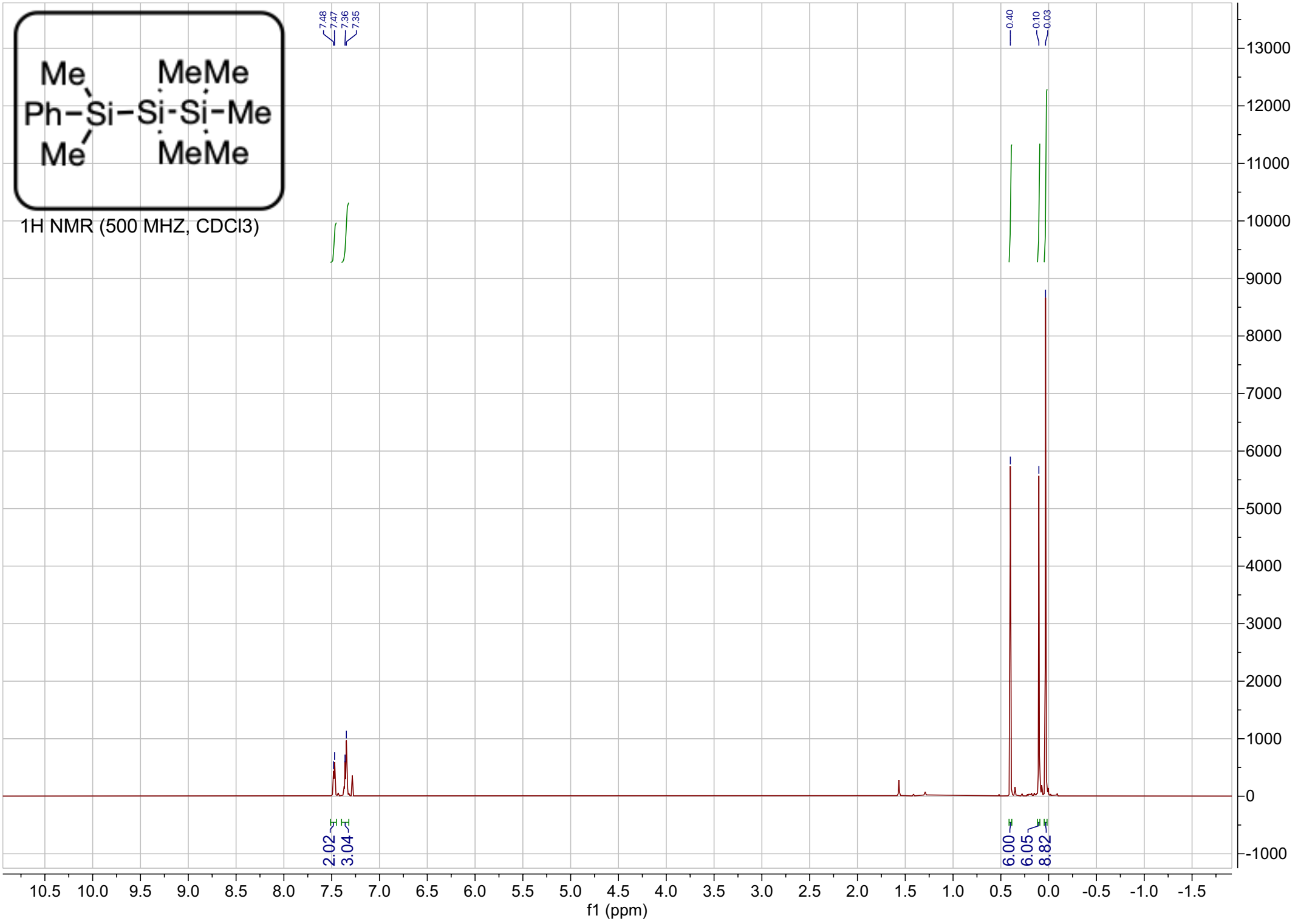


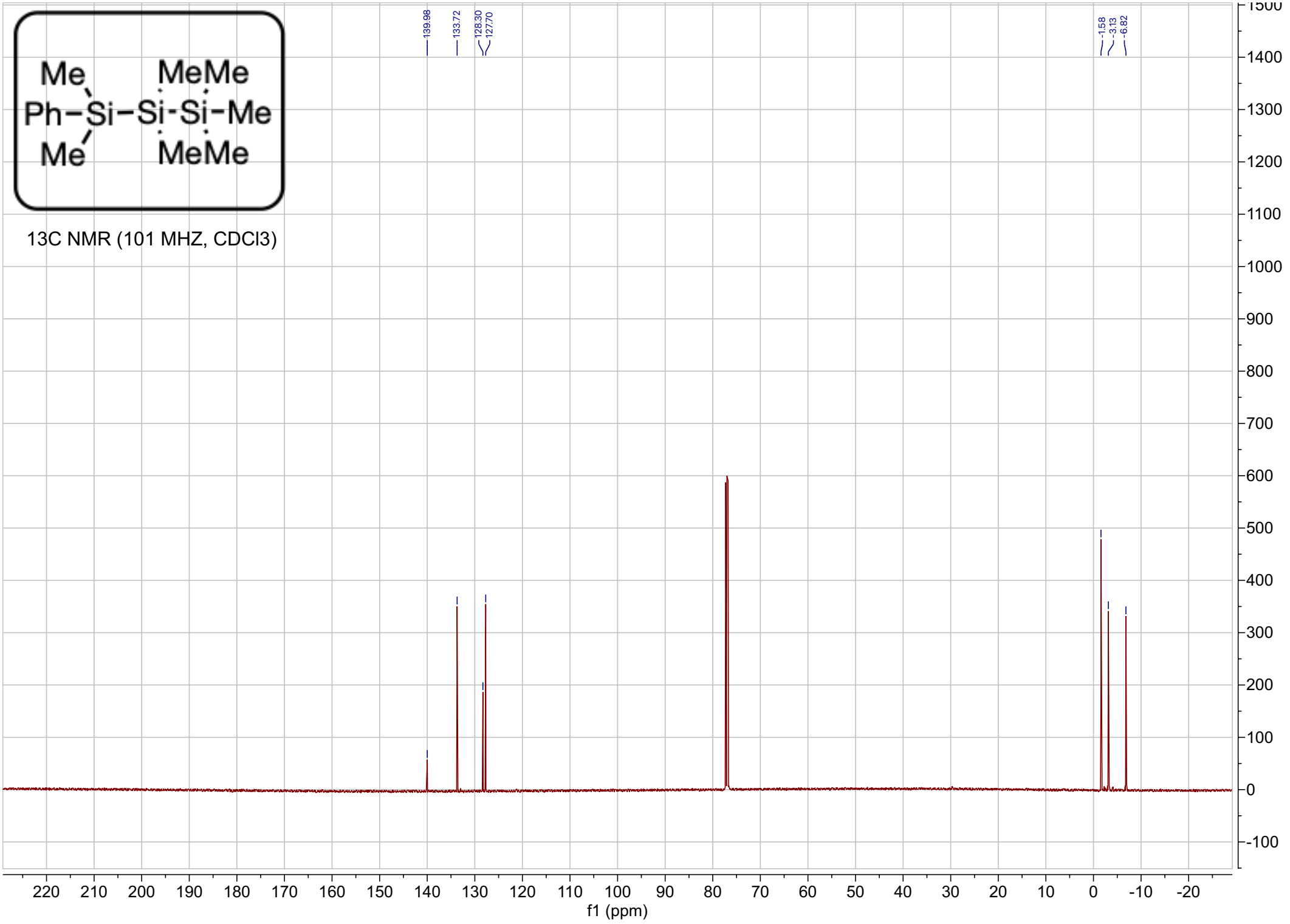
1H NMR (500 MHZ, CDCl3)

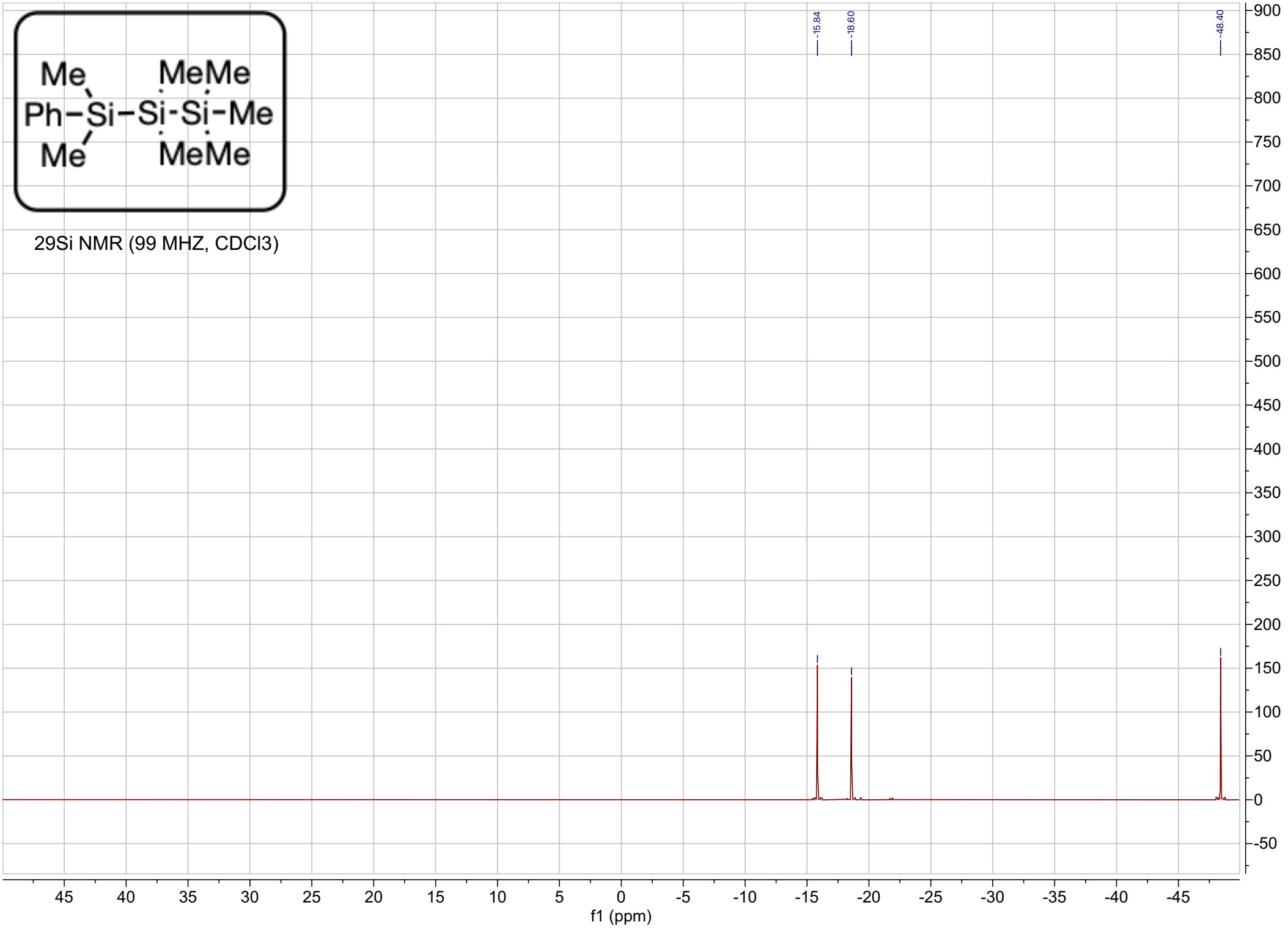

Ph
Me
Si
Me
Me
Si
Me
Me
Me
13C NMR (101 MHZ, CDCl3)
137.20
131.29
125.82
125.25
4.74
6.49
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

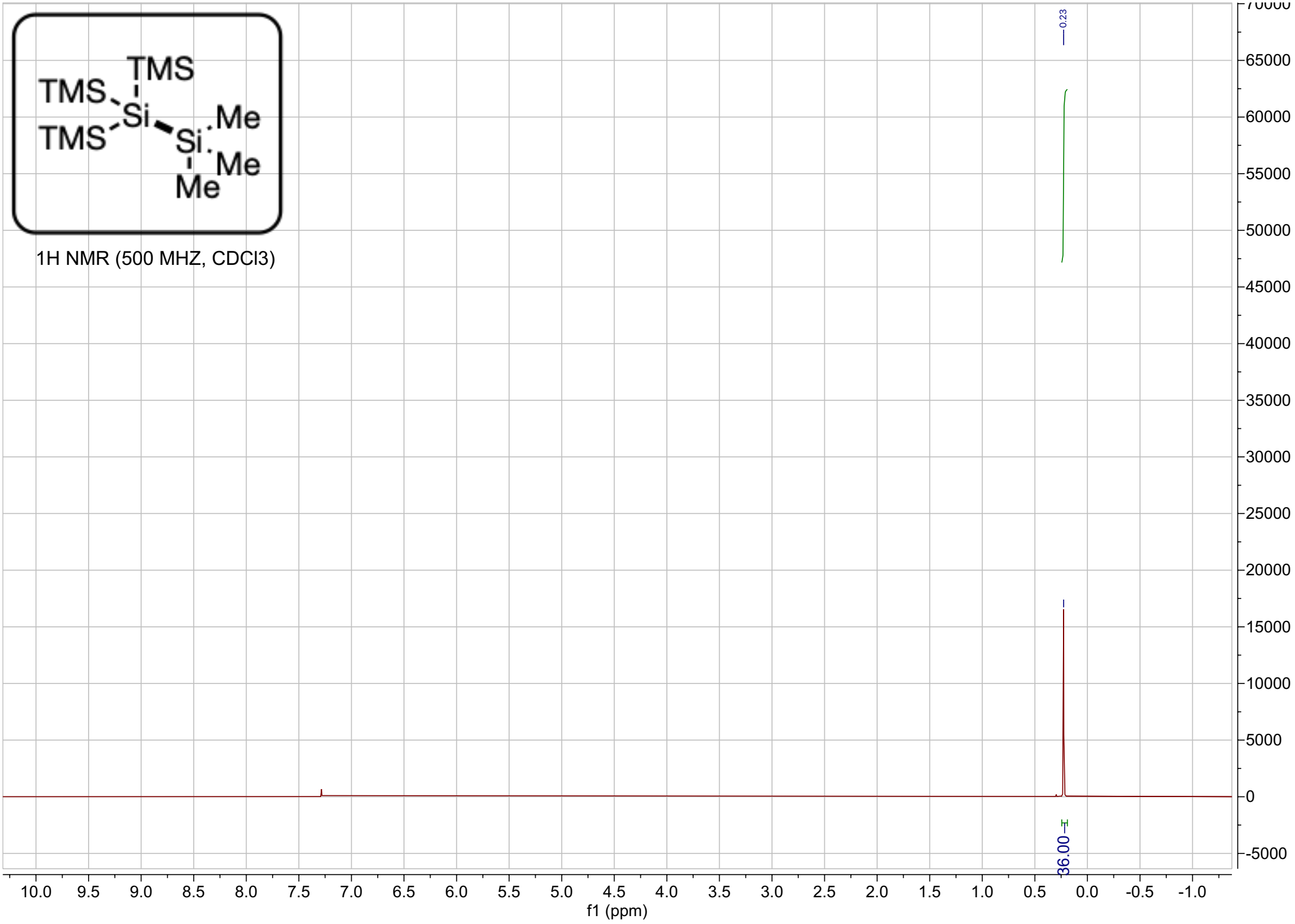


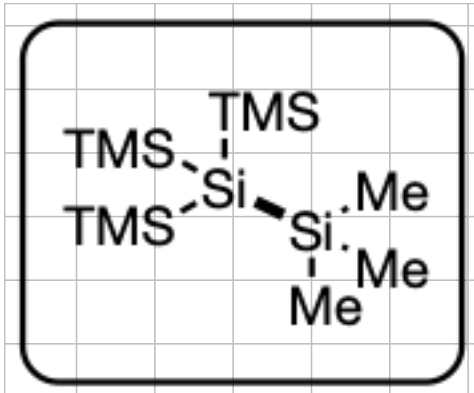
29Si NMR (99 MHZ, CDCl3)


Me MeMe
Ph–Si–Si-Si–Me
Me MeMe
1H NMR (500 MHZ, CDCl3)
7.48
7.47
7.36
7.35
0.40
0.10
0.03
2.02
3.04
6.00
6.05
8.82
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.5 10.0 9.5 9.0 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 -1.0 -1.5
f1 (ppm)

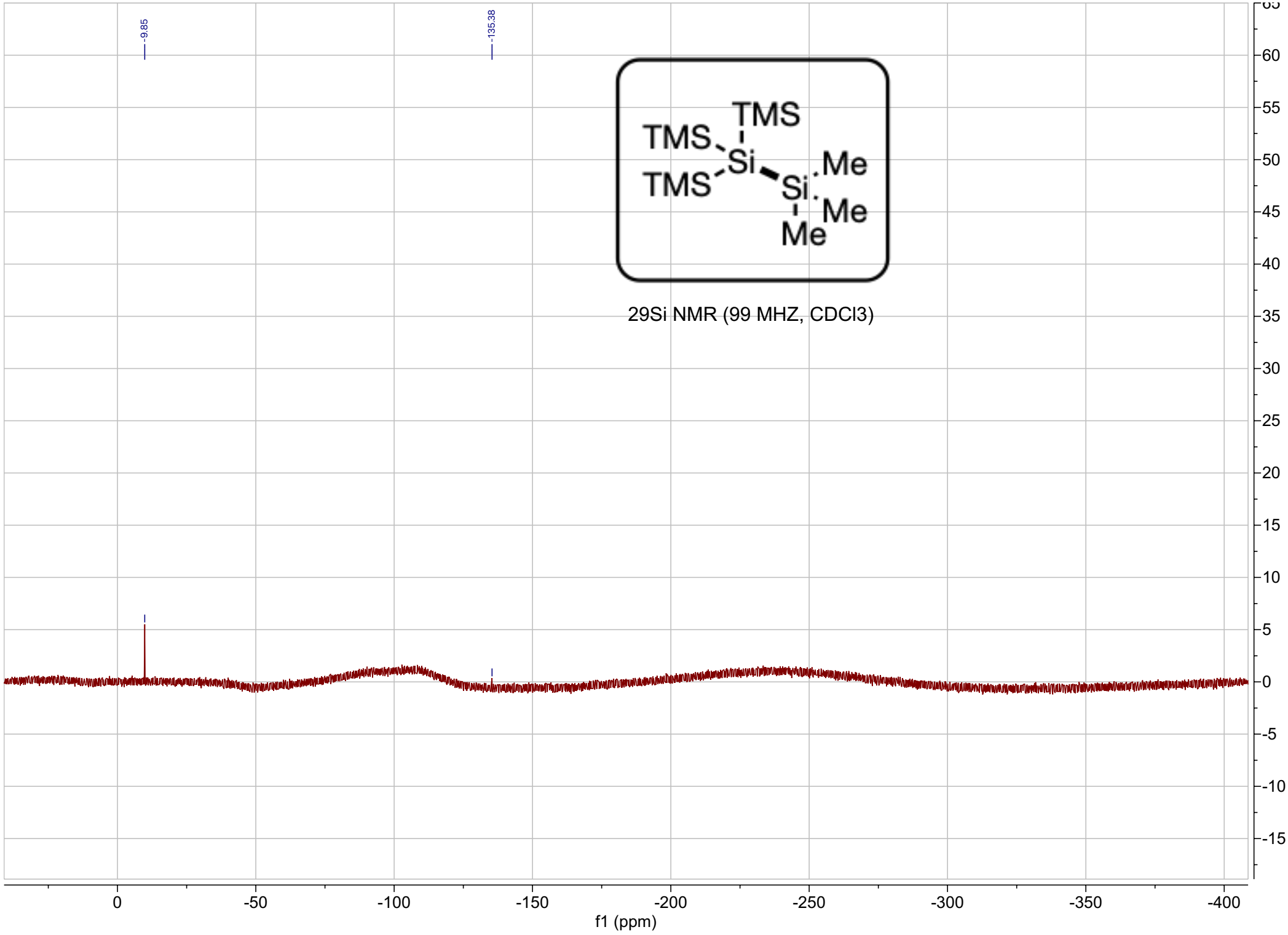

Me
MeMe
Ph–Si–Si-Si–Me
Me
MeMe
13C NMR (101 MHZ, CDCl3)
139.98
133.72
128.30
127.70
-1.58
-3.13
-6.82
f1 (ppm)

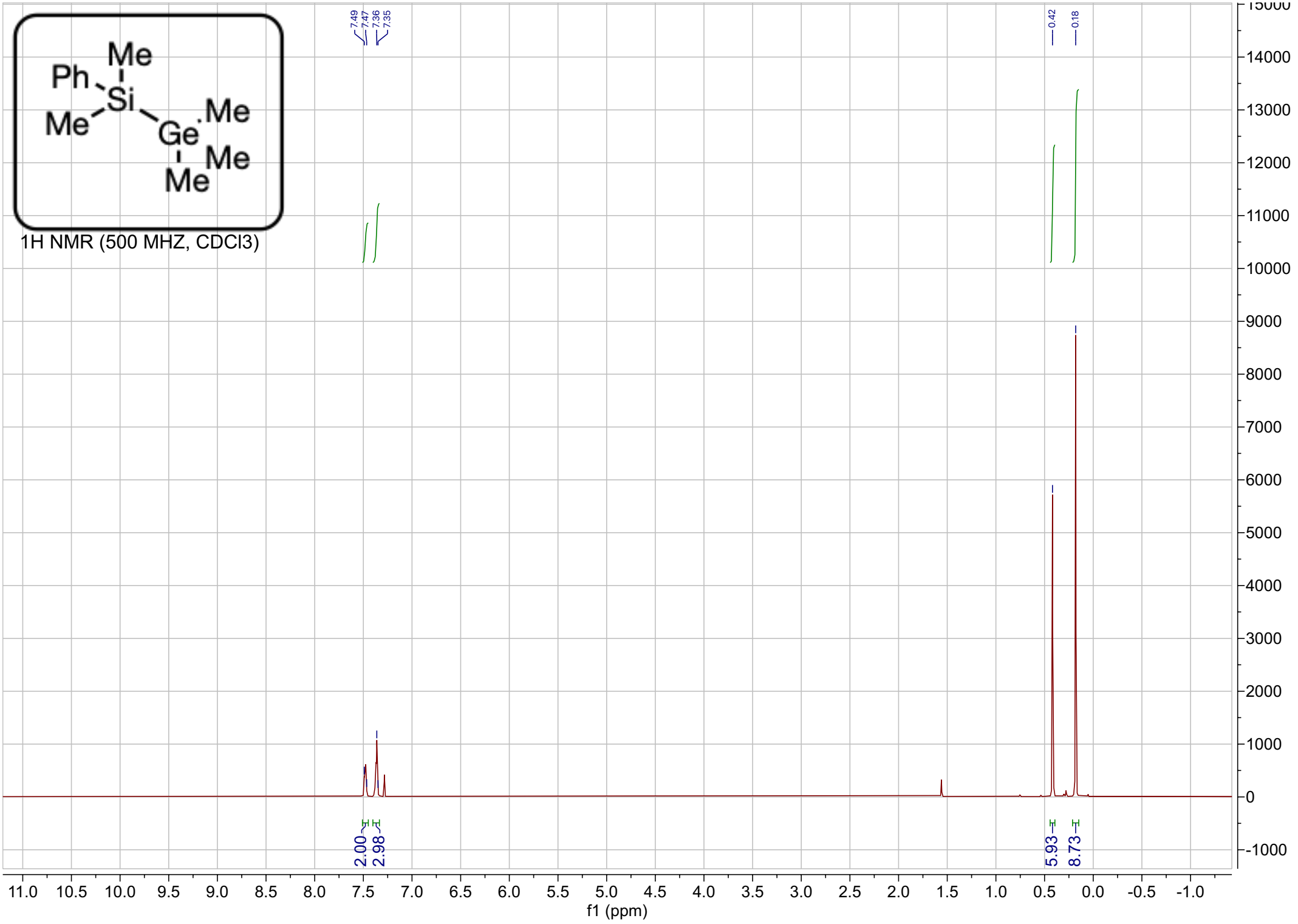

Me
MeMe
Ph–Si–Si-Si-Me
Me
MeMe
29Si NMR (99 MHZ, CDCl3)
-15.84
-18.60
-48.40
900
850
800
750
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

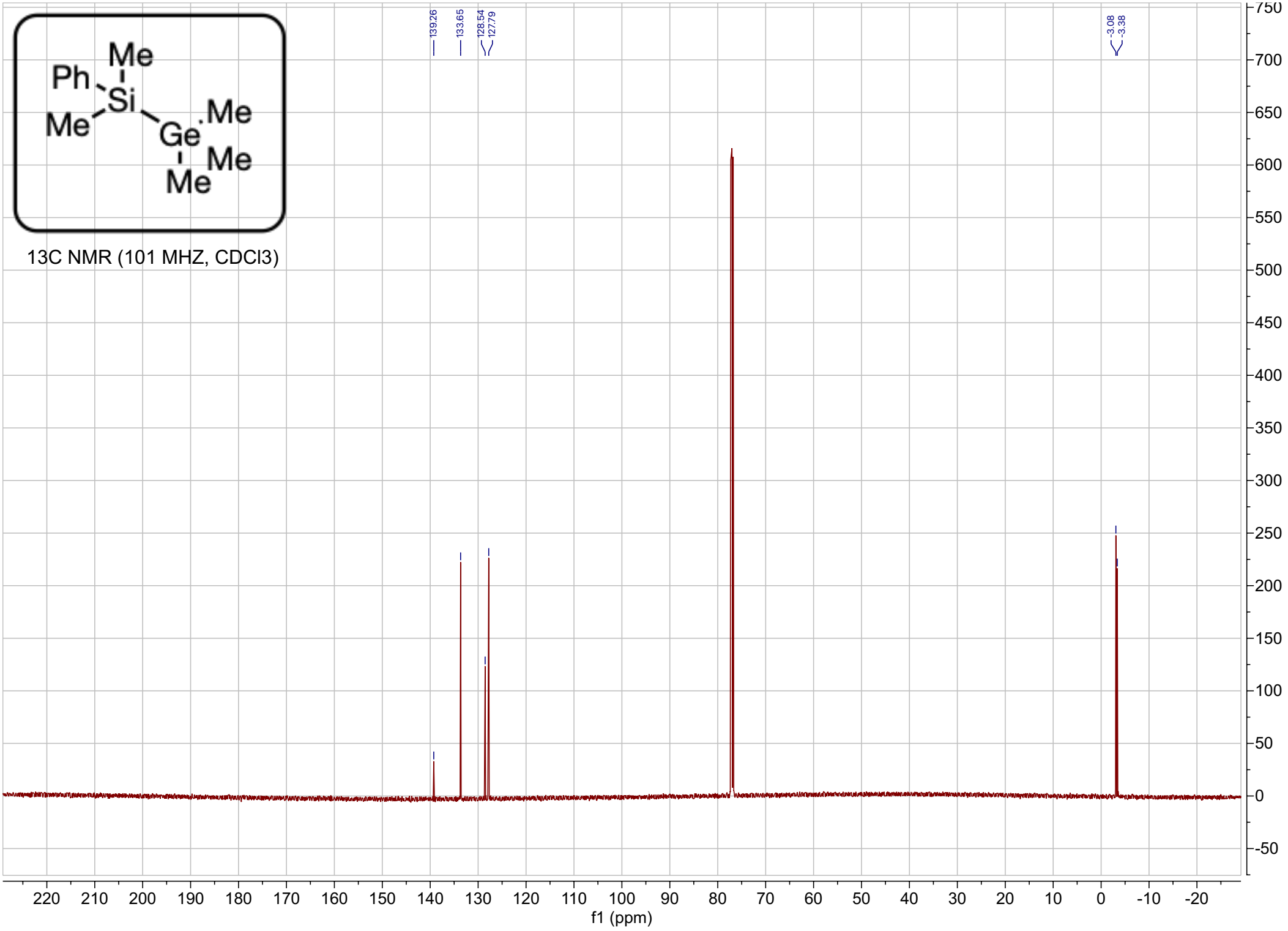

TMS
TMS
Si
TMS
Si
Me
Me
Me
1H NMR (500 MHZ, CDCl3)
0.23
36.00
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

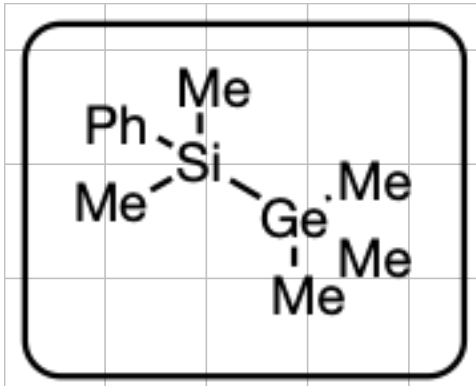


13C NMR (101 MHZ, CDCl3)

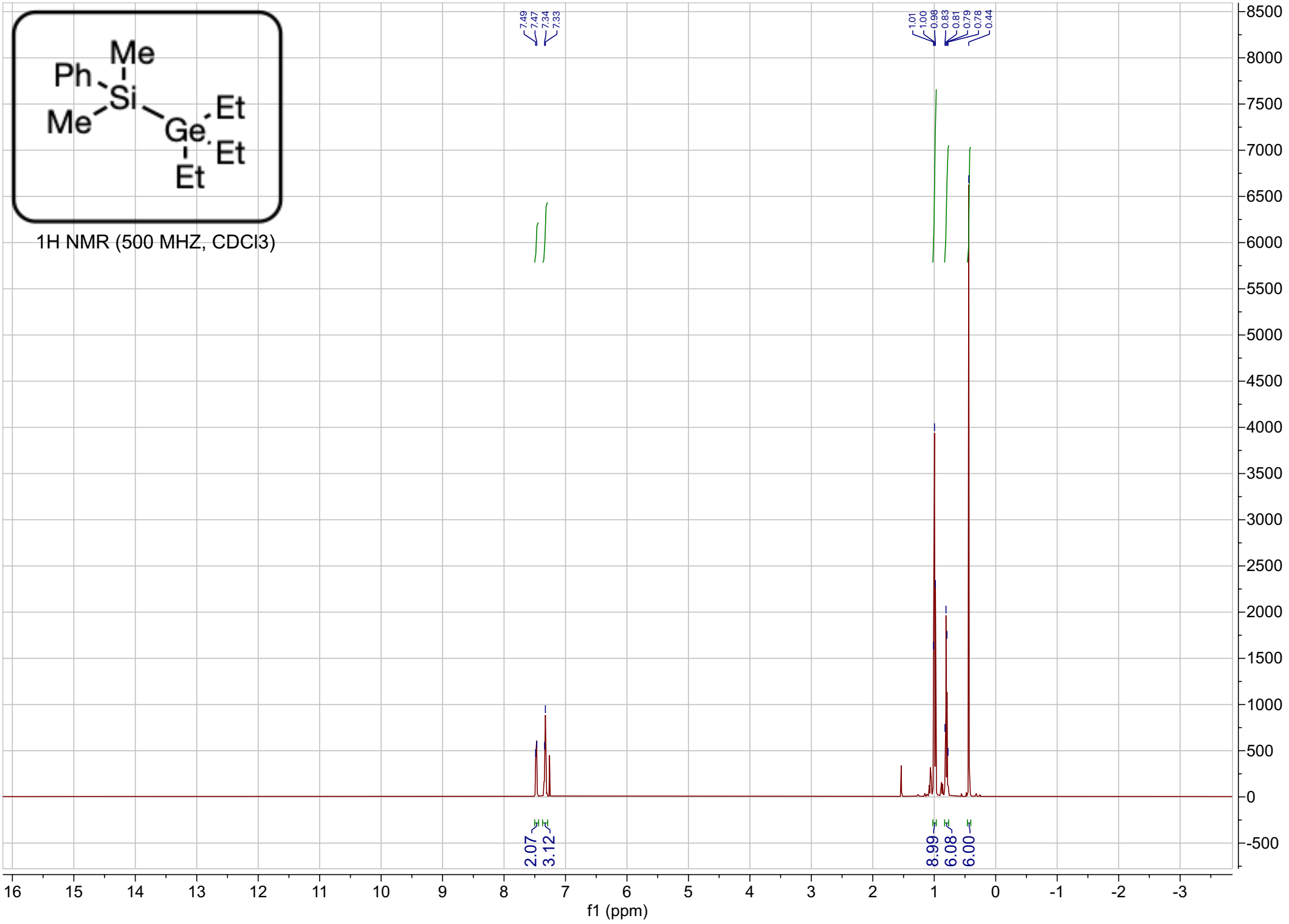

-9.85
-135.38
TMS
TMS
TMS
Si
Si
Me
Me
Me
29Si NMR (99 MHZ, CDCl3)
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

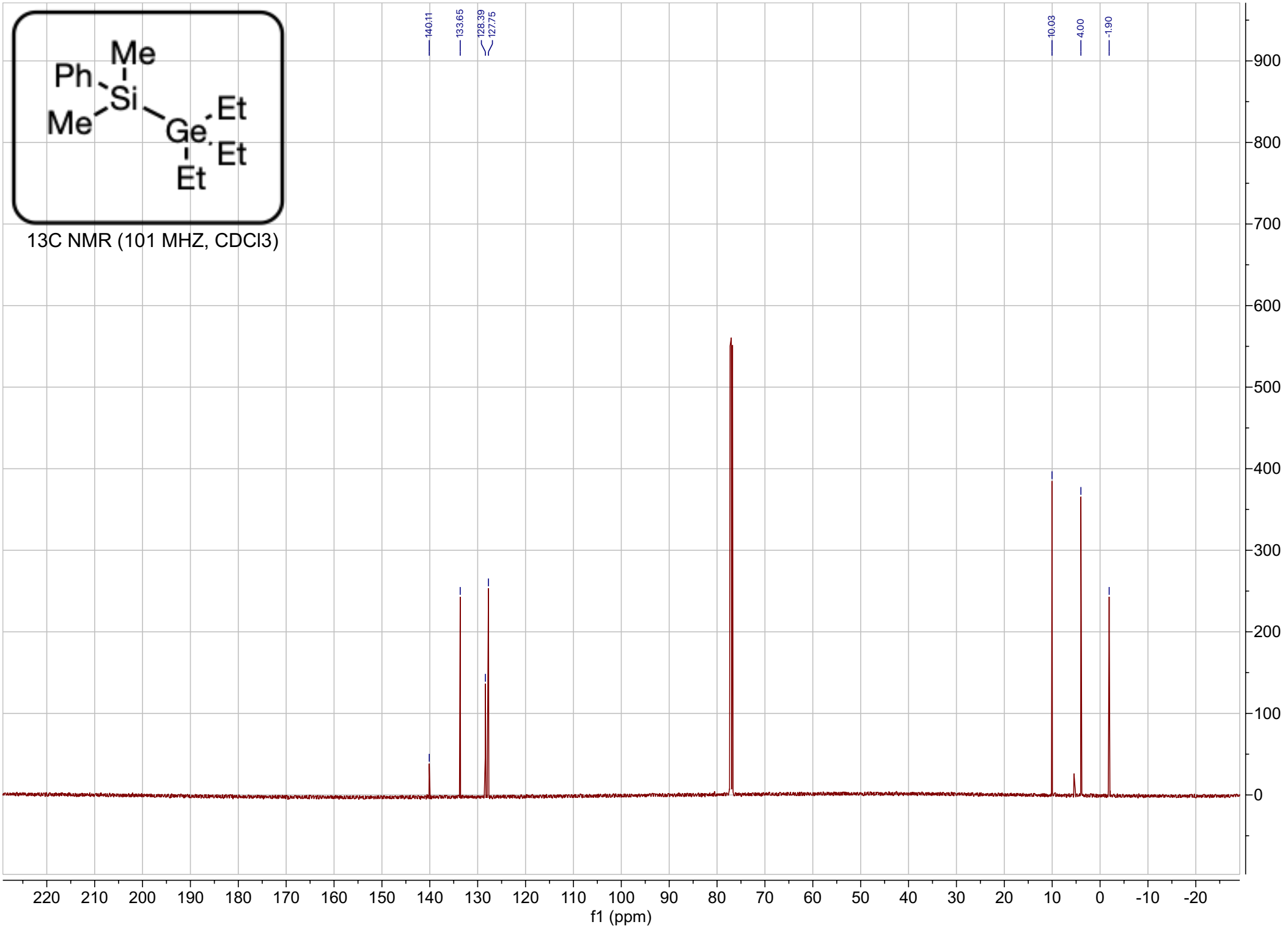

Me
Ph
Si
Me
Ge
Me
Me
Me
1H NMR (500 MHZ, CDCl3)
7.49
7.47
7.36
7.35
0.42
0.18
2.00
2.98
5.93
8.73
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

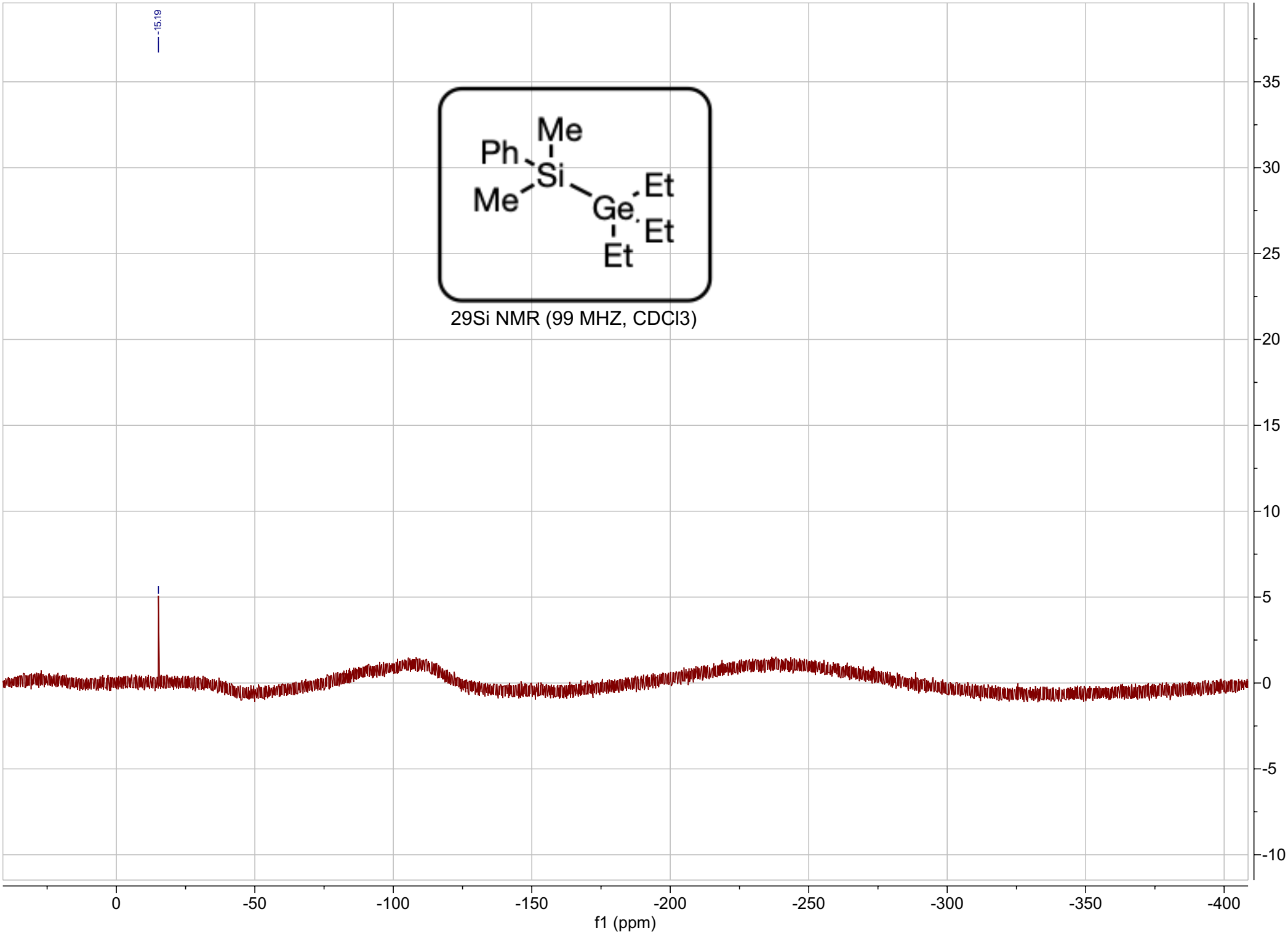

Ph
Me
Si
Me
Ge
Me
Me
Me
13C NMR (101 MHZ, CDCl3)
139.26
133.65
128.54
127.79
-3.08
-3.38
f1 (ppm)

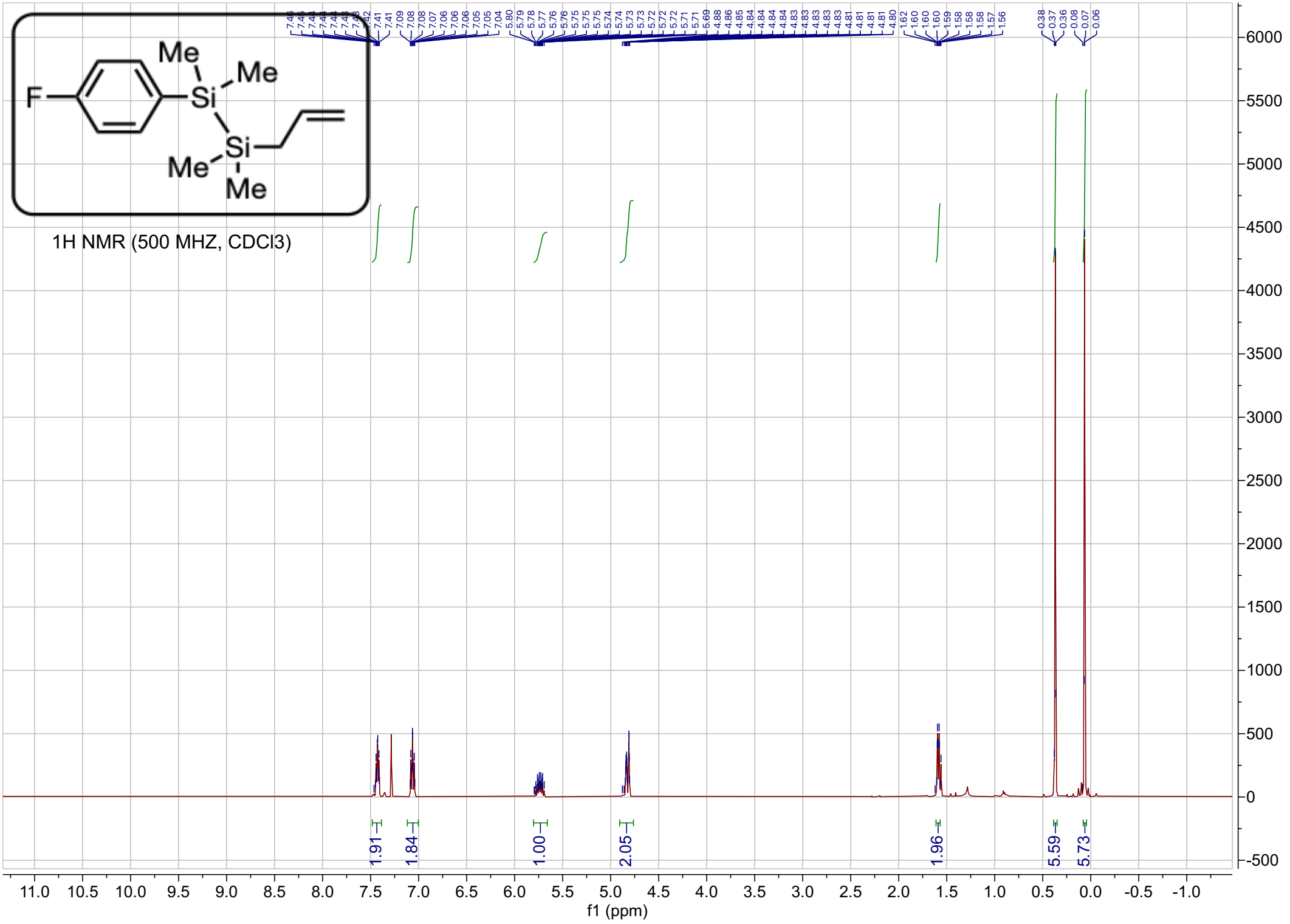


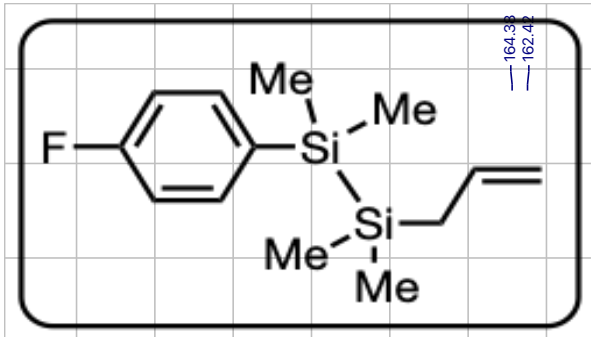
29Si NMR (99 MHZ, CDCl3)


Me
Ph
Si
Me
Ge
Et
Et
Et
1H NMR (500 MHZ, CDCl3)
7.49
7.47
7.34
7.33
1.01
1.00
0.98
0.83
0.81
0.79
0.78
0.44
2.07
3.12
8.99
6.08
6.00
f1 (ppm)

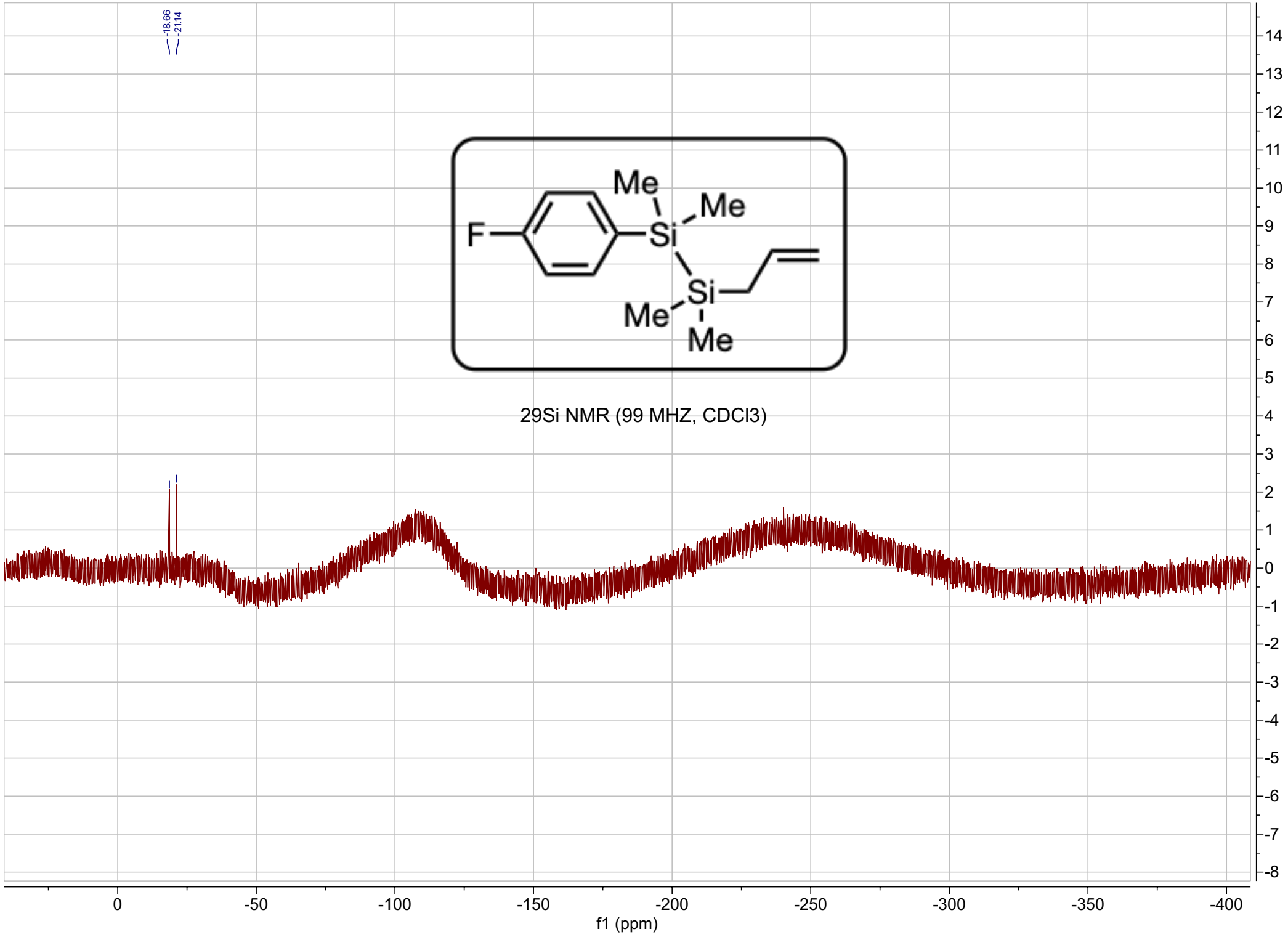

Ph
Me
Si
Me
Ge
Et
Et
Et
13C NMR (101 MHZ, CDCl3)
140.11
133.65
128.39
127.75
10.03
4.00
-1.90
900
800
700
600
500
400
300
200
100
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

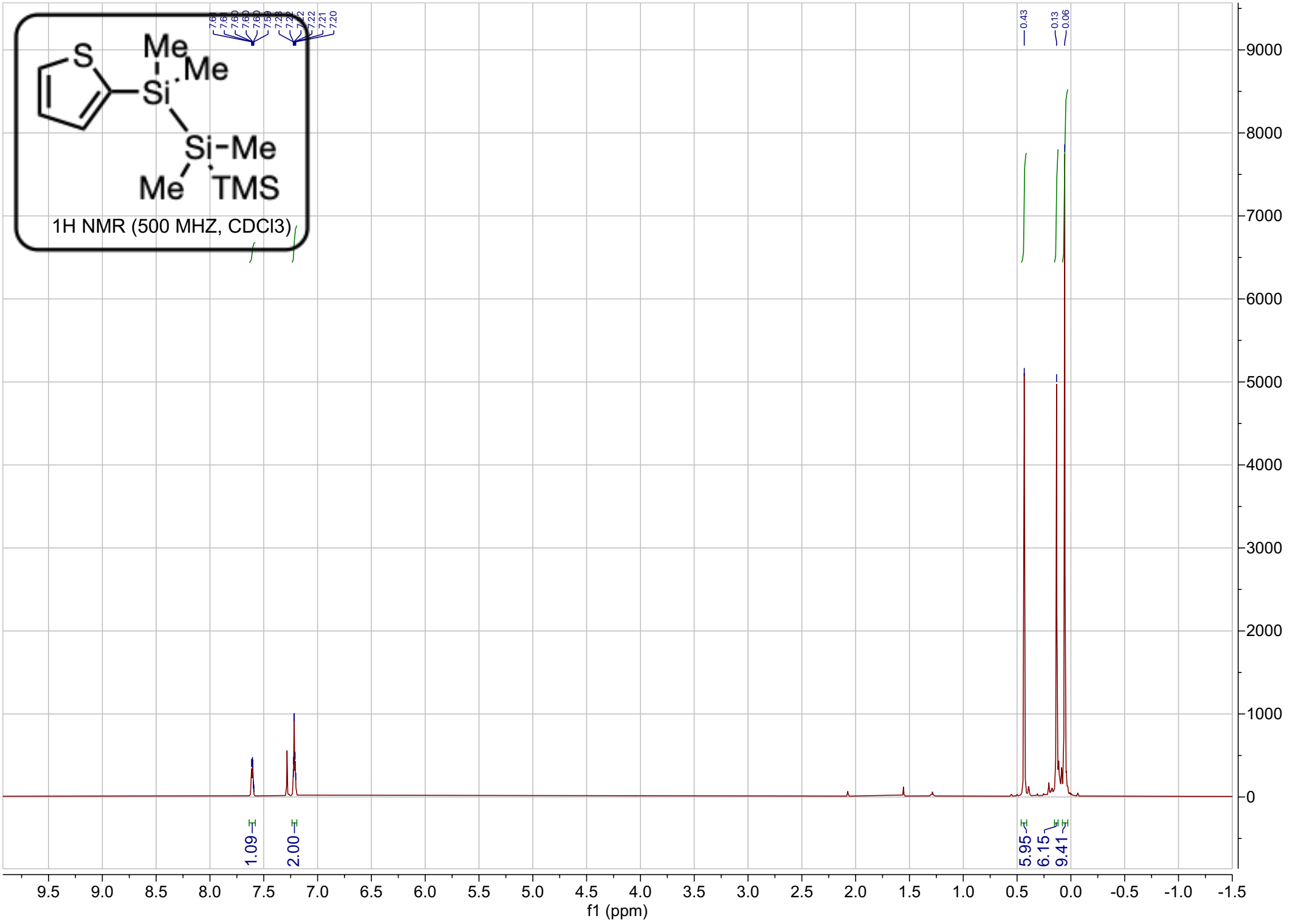

-15.19
Me
Ph
Si
Me
Ge
Et
Et
Et
29Si NMR (99 MHZ, CDCl3)
35
30
25
20
15
10
5
0
-5
-10
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

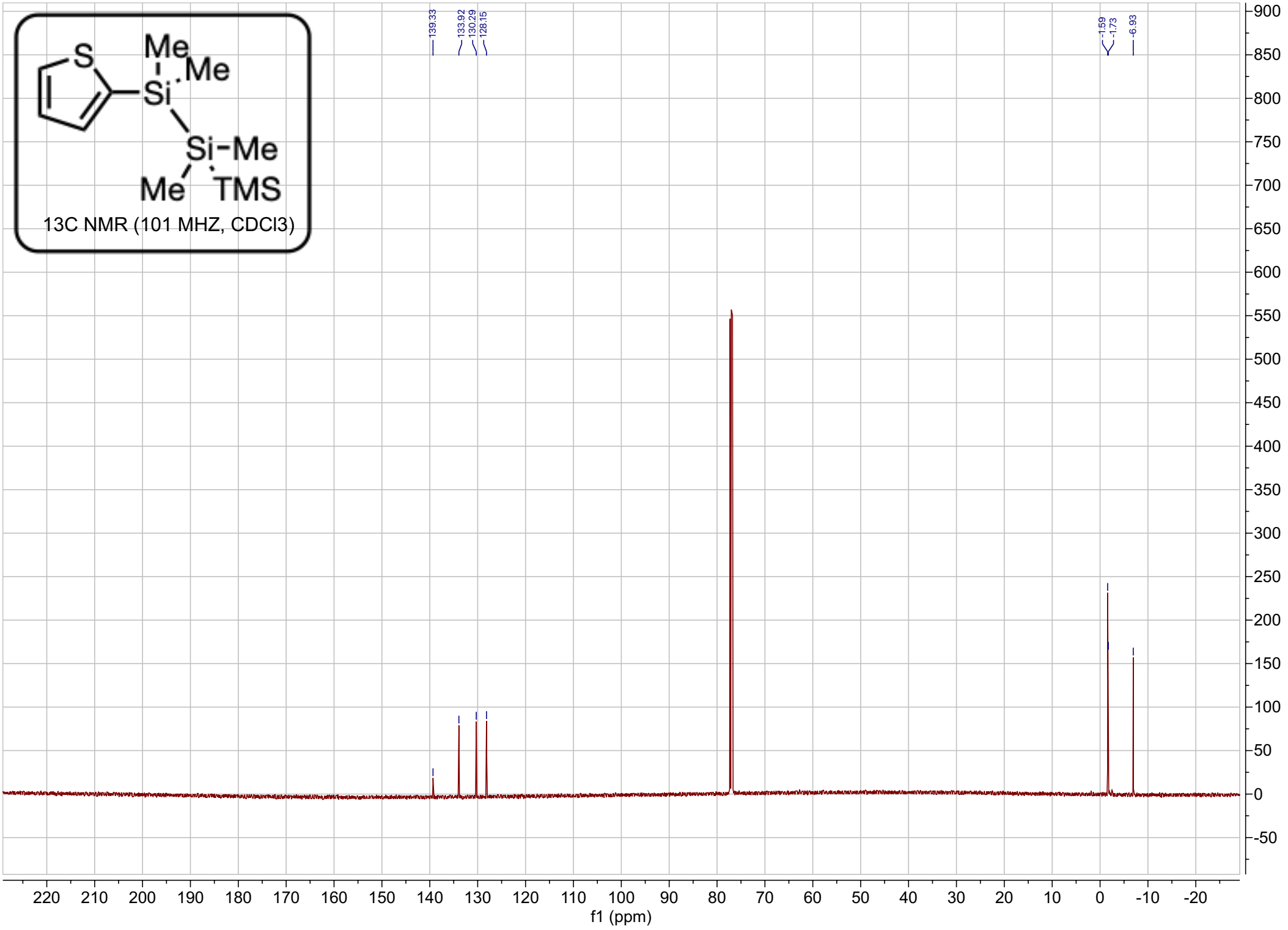

Me
Me
F
Si
Si
Me
Me
1H NMR (500 MHZ, CDCl3)
1.91
1.84
1.00
2.05
1.96
5.59
5.73
f1 (ppm)

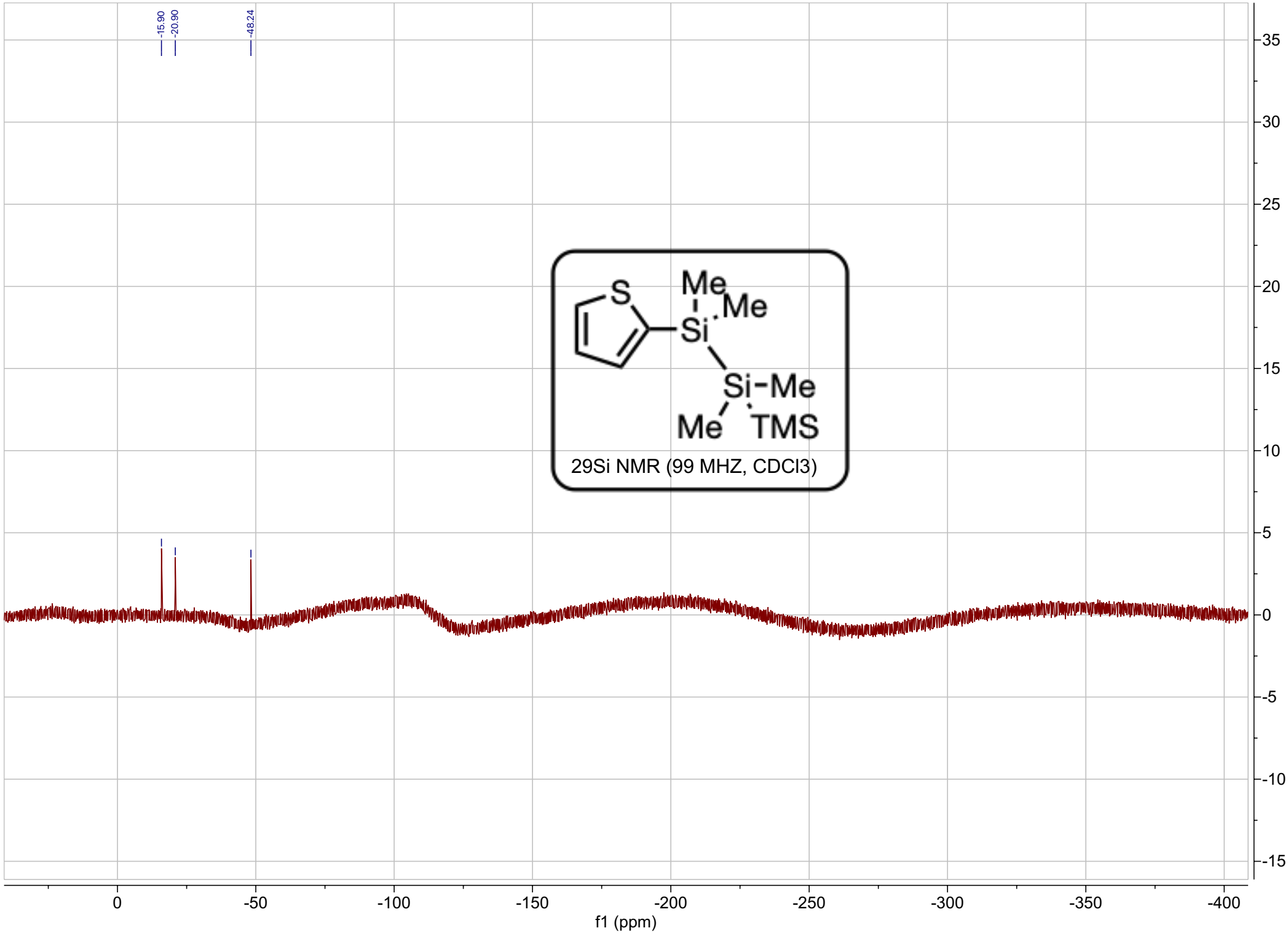


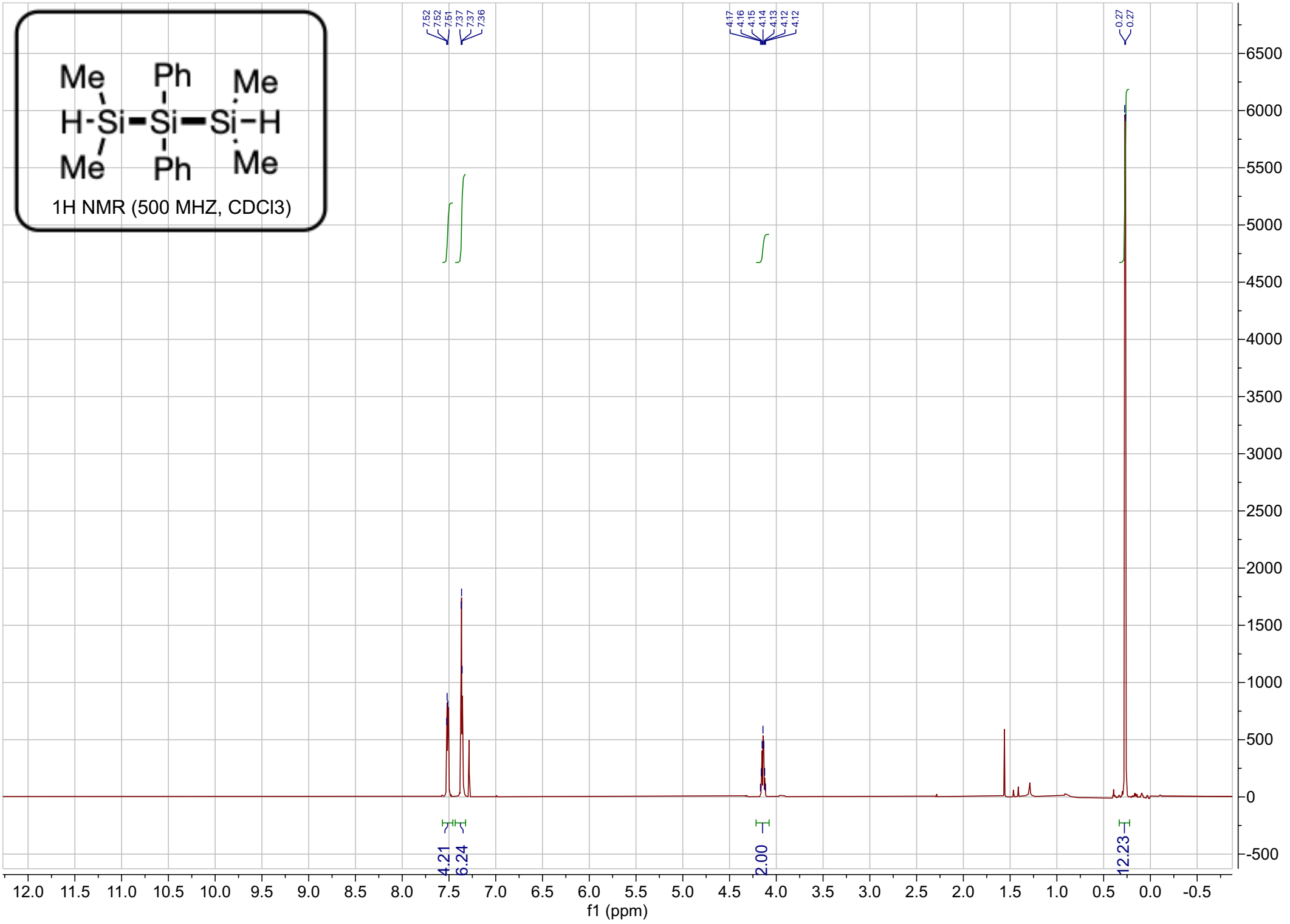
13C NMR (101 MHZ, CDCl3)

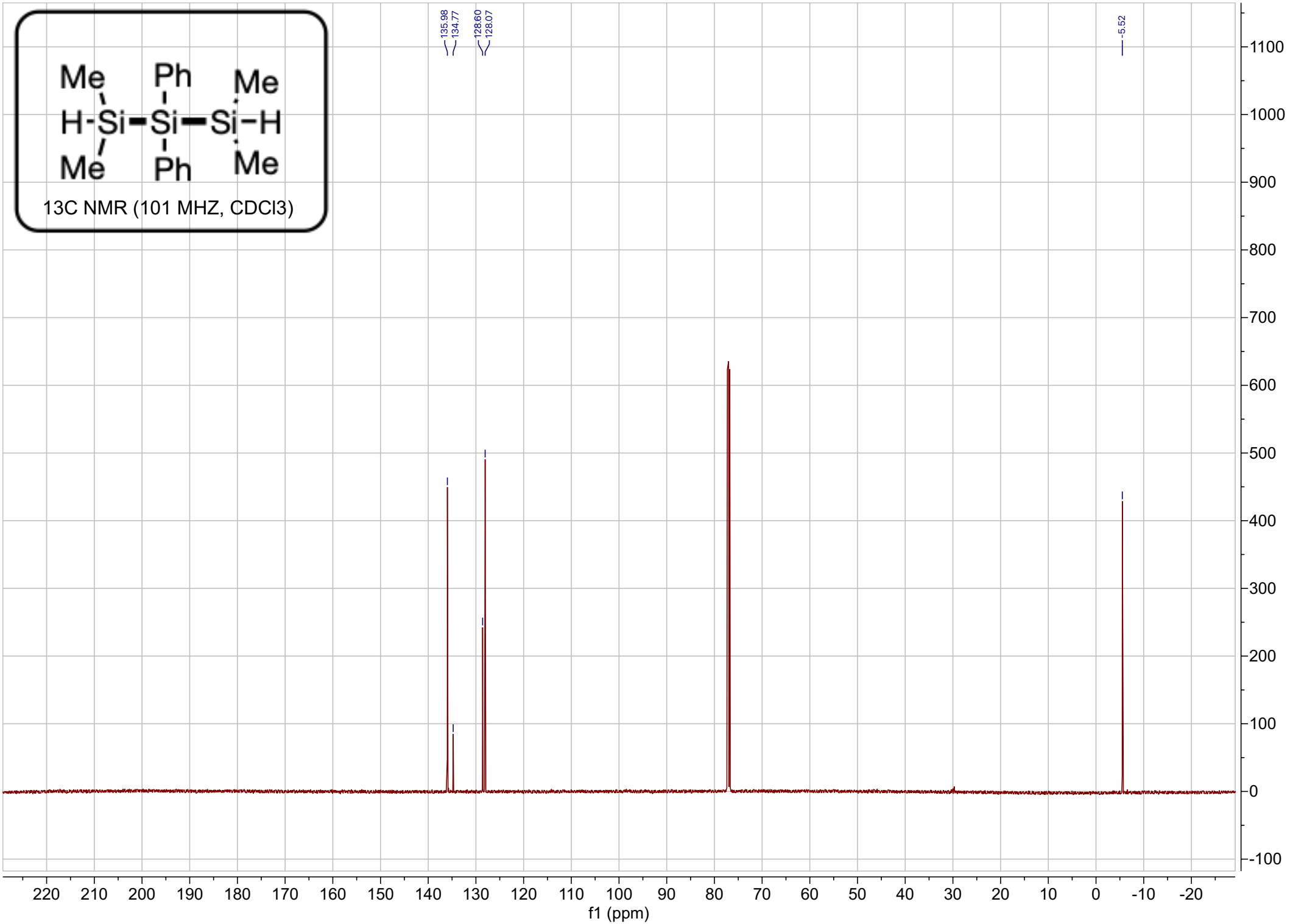

-18.66
-21.14
Me
Me
F
Si
Si
Me
Me
29Si NMR (99 MHZ, CDCl3)
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
-5
-6
-7
-8
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

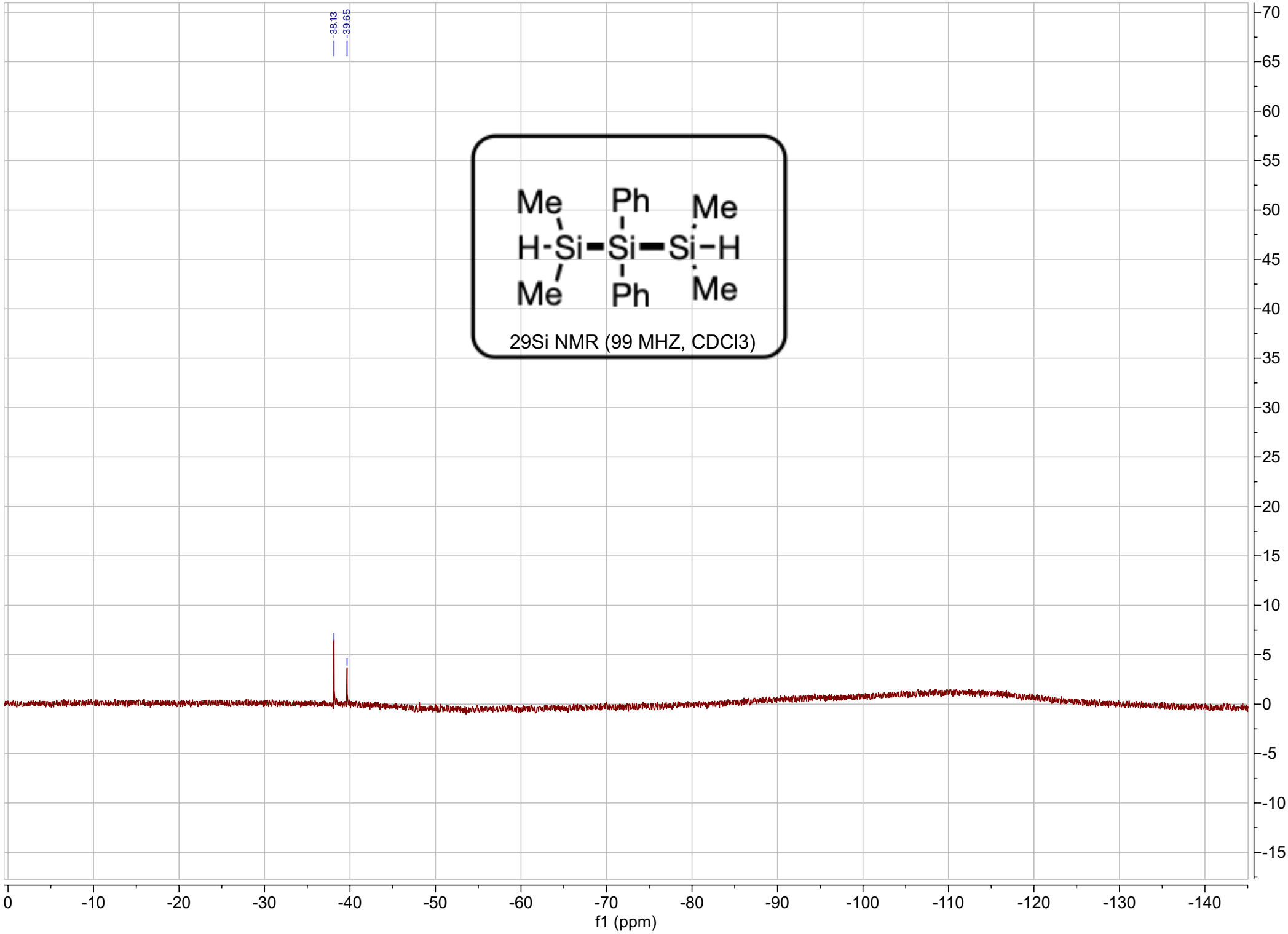

S
Me
Me
Si
Si-Me
Me
TMS
1H NMR (500 MHZ, CDCl3)
0.43
0.13
0.06
1.09
2.00
5.95
6.15
9.41
f1 (ppm)

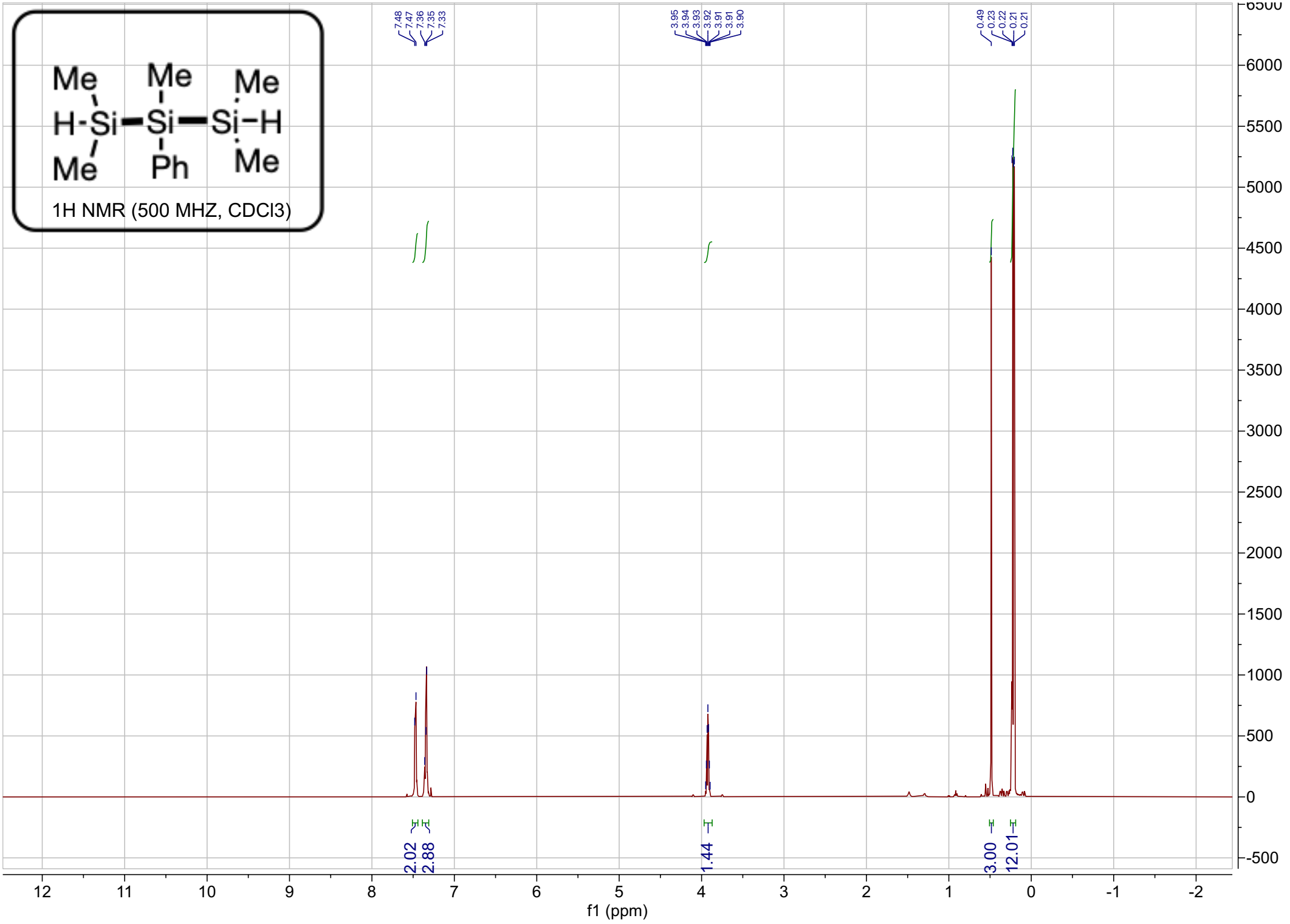

S
Me
Me
Si
Si-Me
Me
TMS
13C NMR (101 MHZ, CDCl3)
139.33
133.92
130.29
128.15
-1.59
-1.73
-6.93
f1 (ppm)

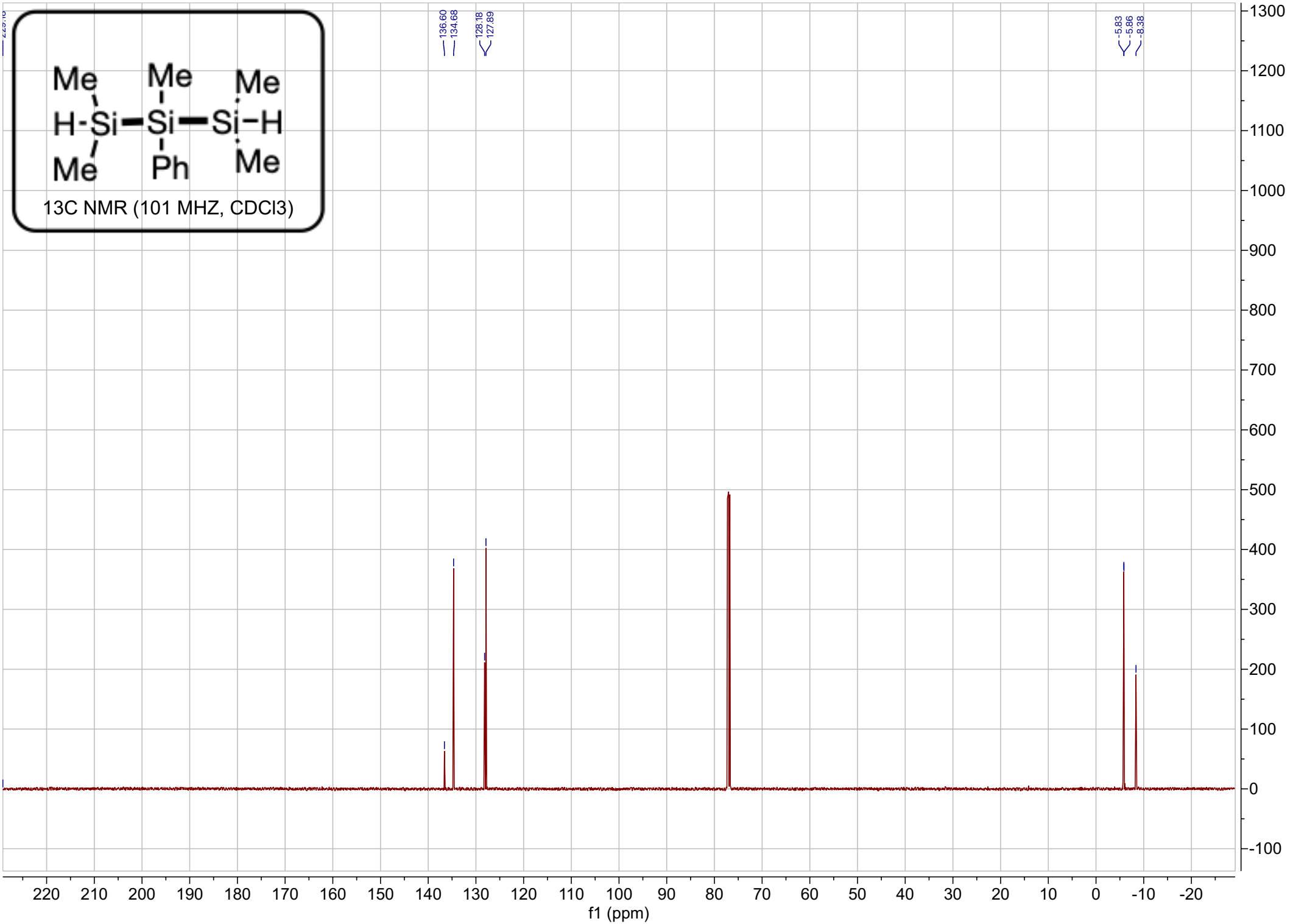

-15.90
-20.90
-48.24
S
Me
Me
Si
Si-Me
Me
TMS
29Si NMR (99 MHZ, CDCl3)
35
30
25
20
15
10
5
0
-5
-10
-15
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

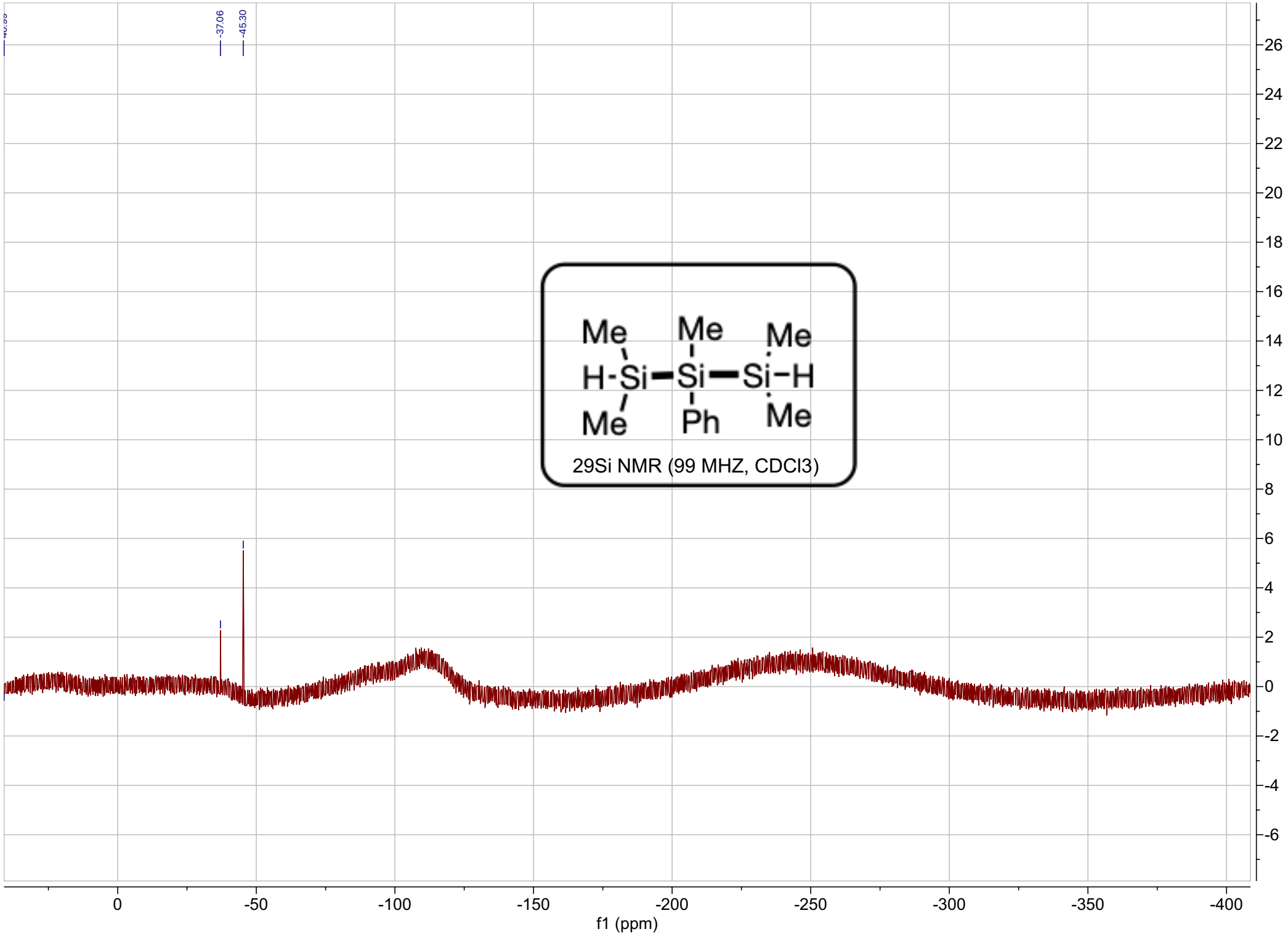

Me Ph Me
H-Si-Si-Si-H
Me Ph Me
1H NMR (500 MHZ, CDCl3)
7.52
7.52
7.51
7.37
7.37
7.36
4.17
4.16
4.15
4.14
4.13
4.12
4.12
4.12
0.27
0.27
4.21
6.24
2.00
12.23
f1 (ppm)

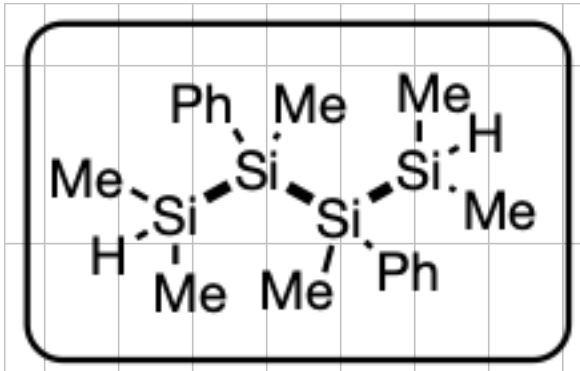

Me
Ph
Me
H-Si-Si-Si-H
Me
Ph
Me
13C NMR (101 MHZ, CDCl3)
135.98
134.77
128.60
128.07
-5.52
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


-38.13
-39.65
Me
Ph
Me
H-Si-Si-Si-H
Me
Ph
Me
29Si NMR (99 MHZ, CDCl3)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
f1 (ppm)

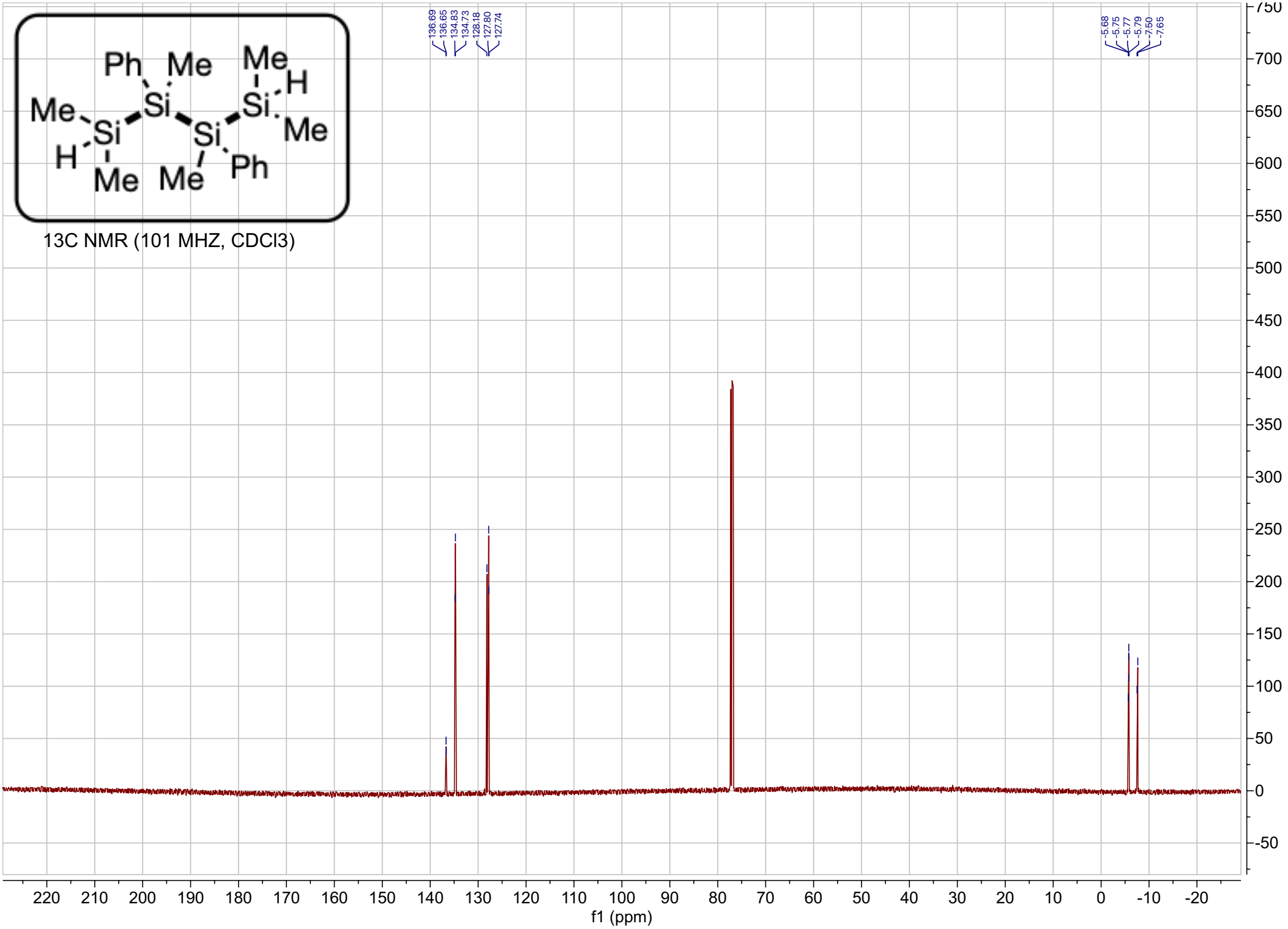

Me
Me
Me
H-Si
Si
Si-H
Me
Ph
Me
1H NMR (500 MHZ, CDCl3)
7.48
7.47
7.36
7.35
7.33
3.95
3.94
3.93
3.92
3.91
3.91
3.90
0.49
0.23
0.22
0.21
0.21
2.02
2.88
1.44
3.00
12.01
f1 (ppm)


Me
Me
Me
H-Si
Si
Si-H
Me
Ph
Me
13C NMR (101 MHZ, CDCl3)
136.60
134.68
128.18
127.89
-5.83
-5.86
-8.38
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

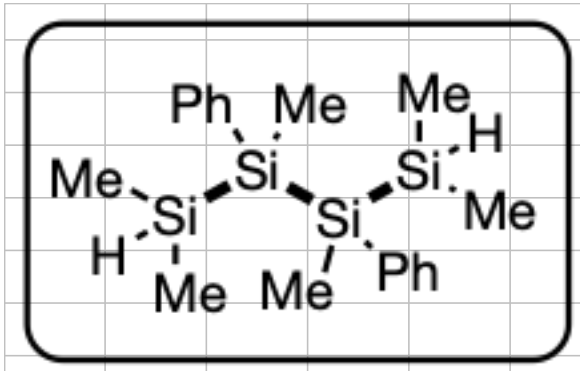

-37.06
-45.30
Me
Me
Me
H-Si
Si
Si-H
Me
Ph
Me
29Si NMR (99 MHZ, CDCl3)
26
24
22
20
18
16
14
12
10
8
6
4
2
0
-2
-4
-6
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)



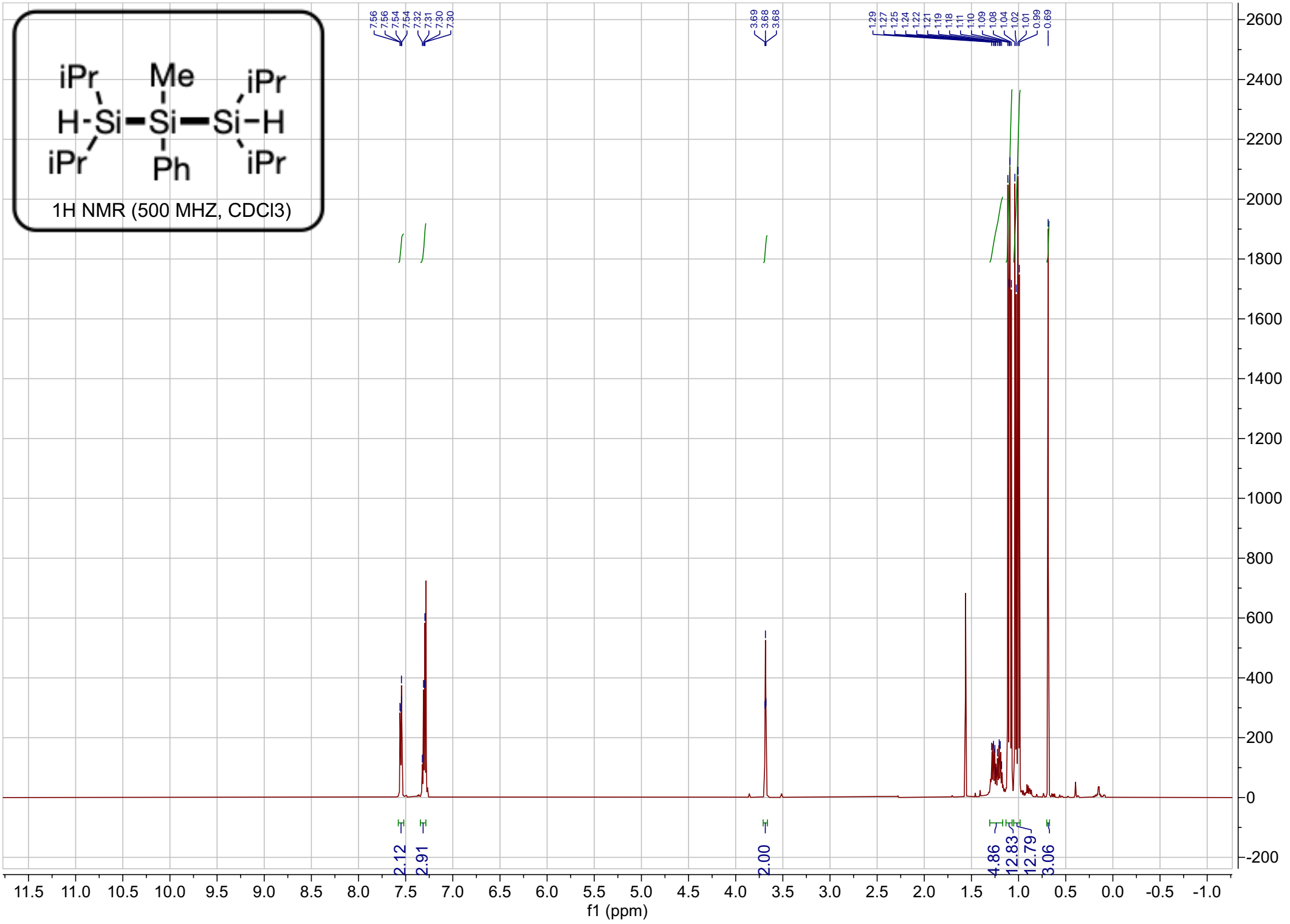
1H NMR (500 MHZ, CDCl3)

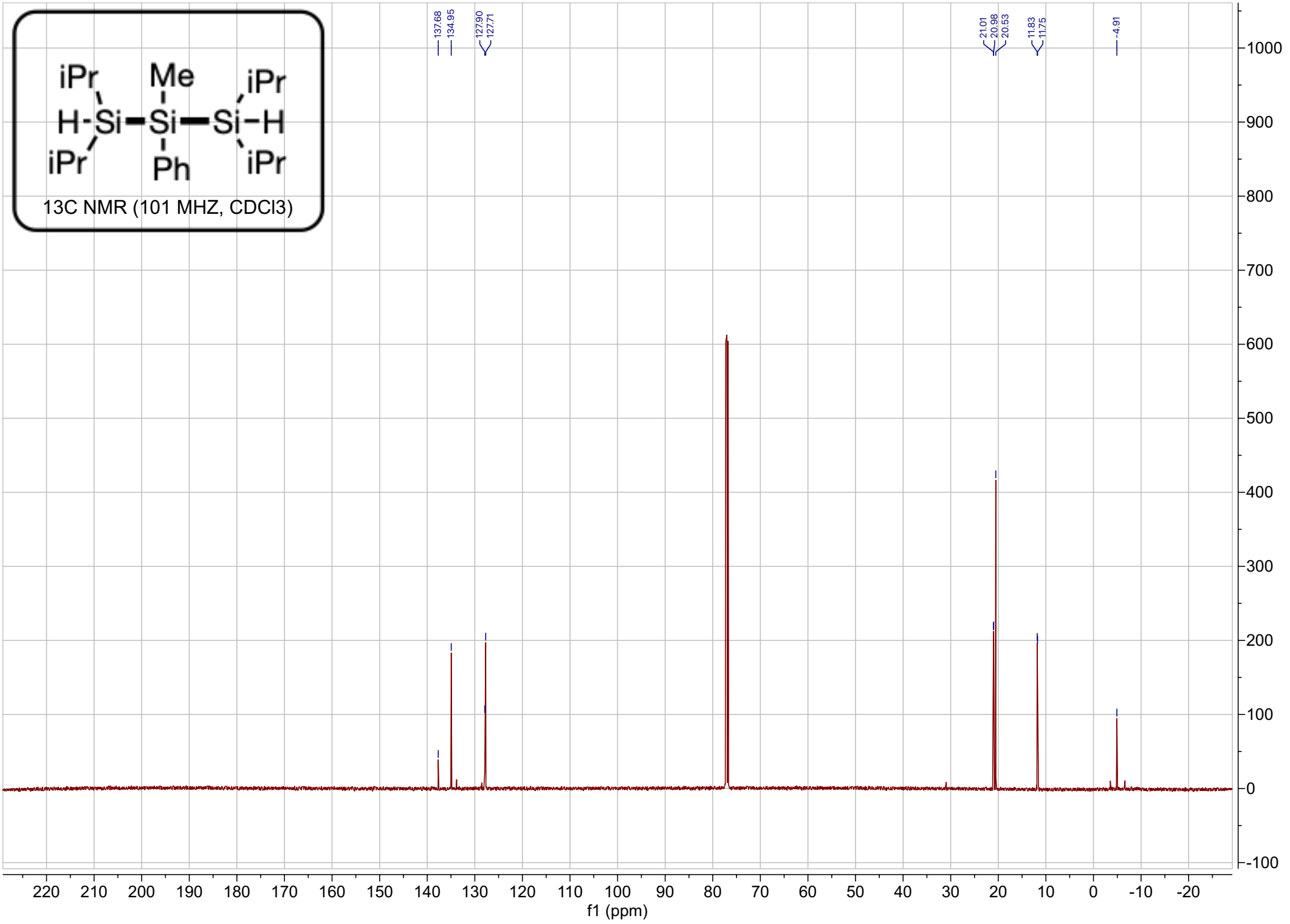


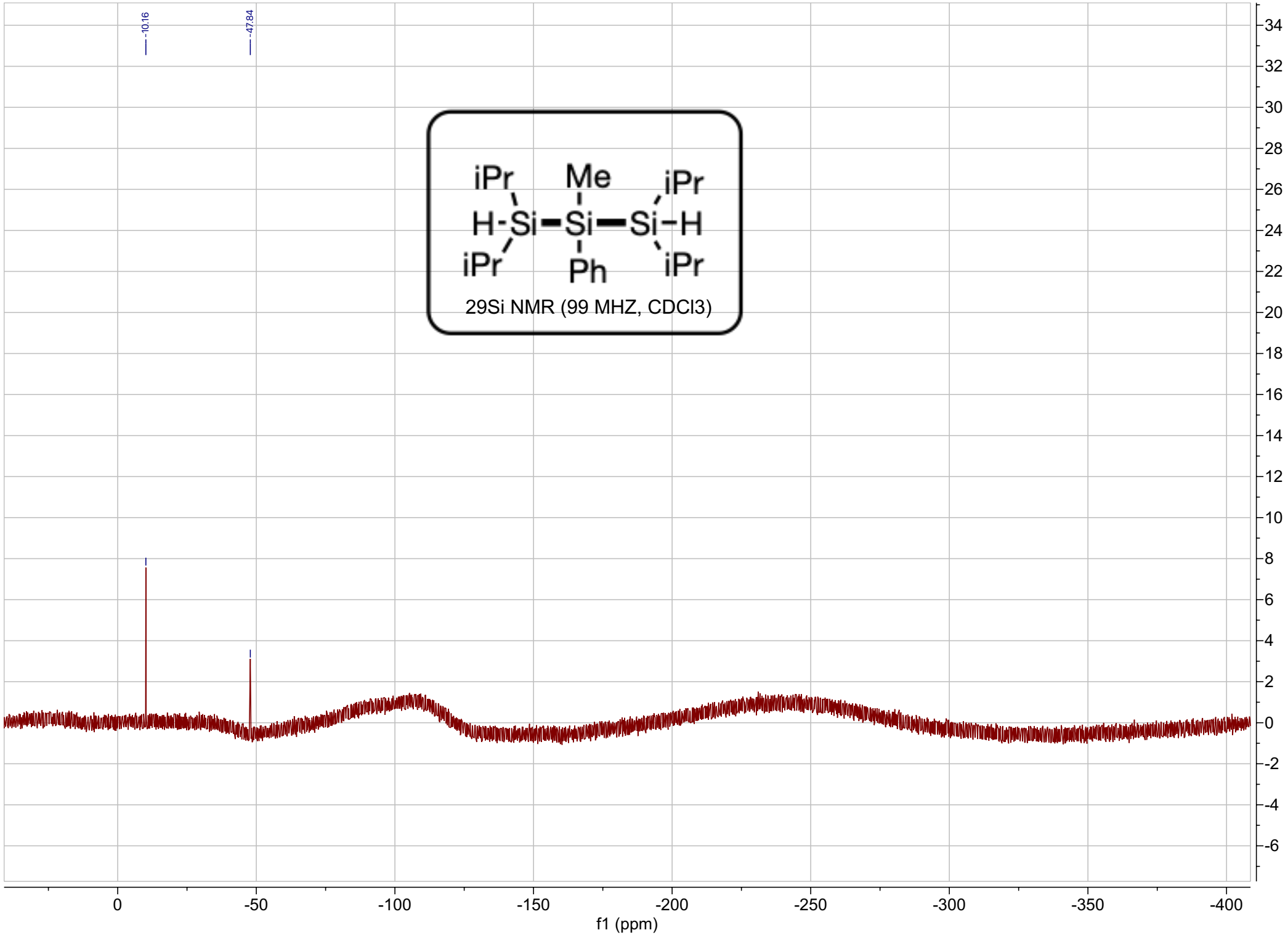
13C NMR (101 MHZ, CDCl3)

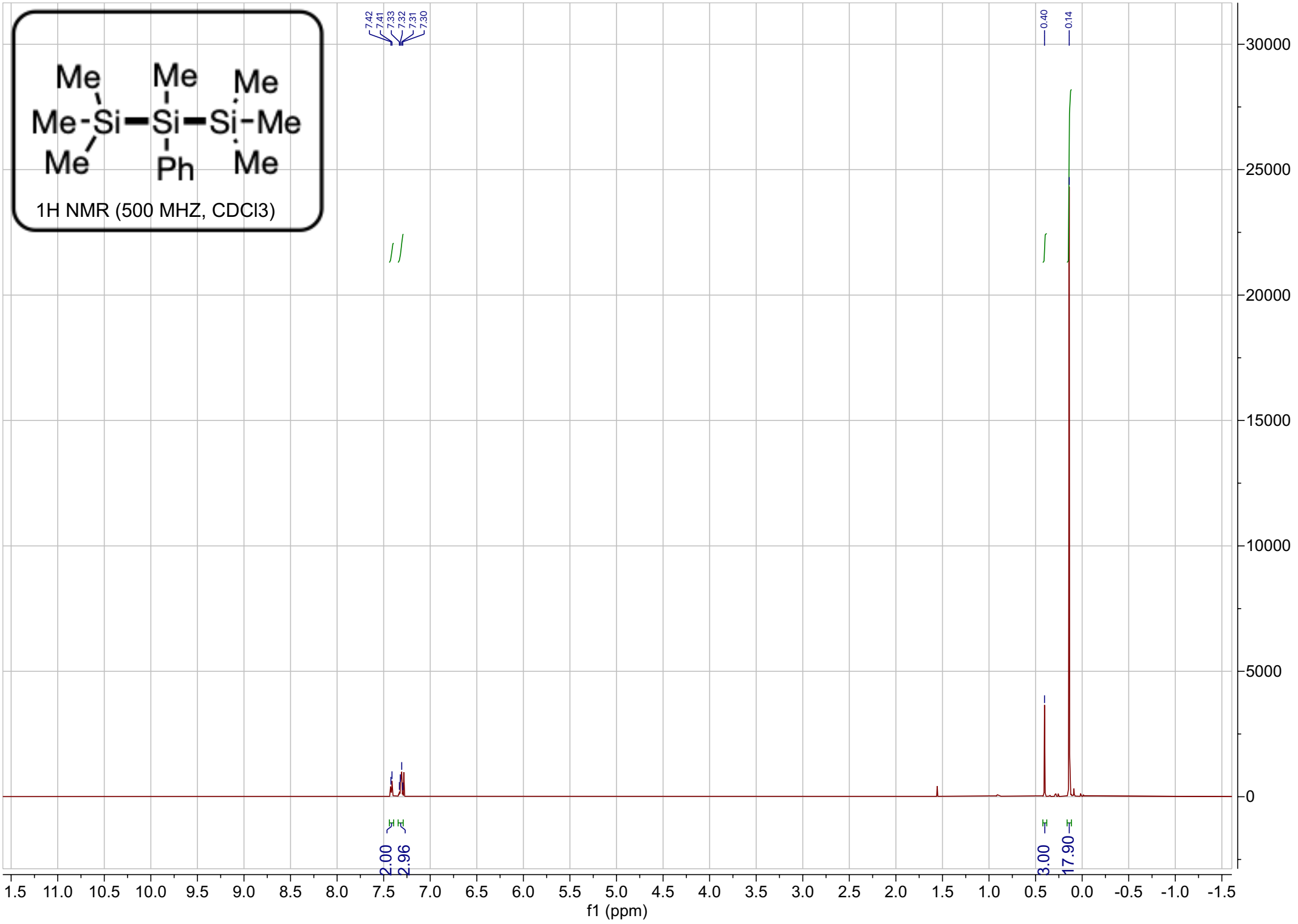


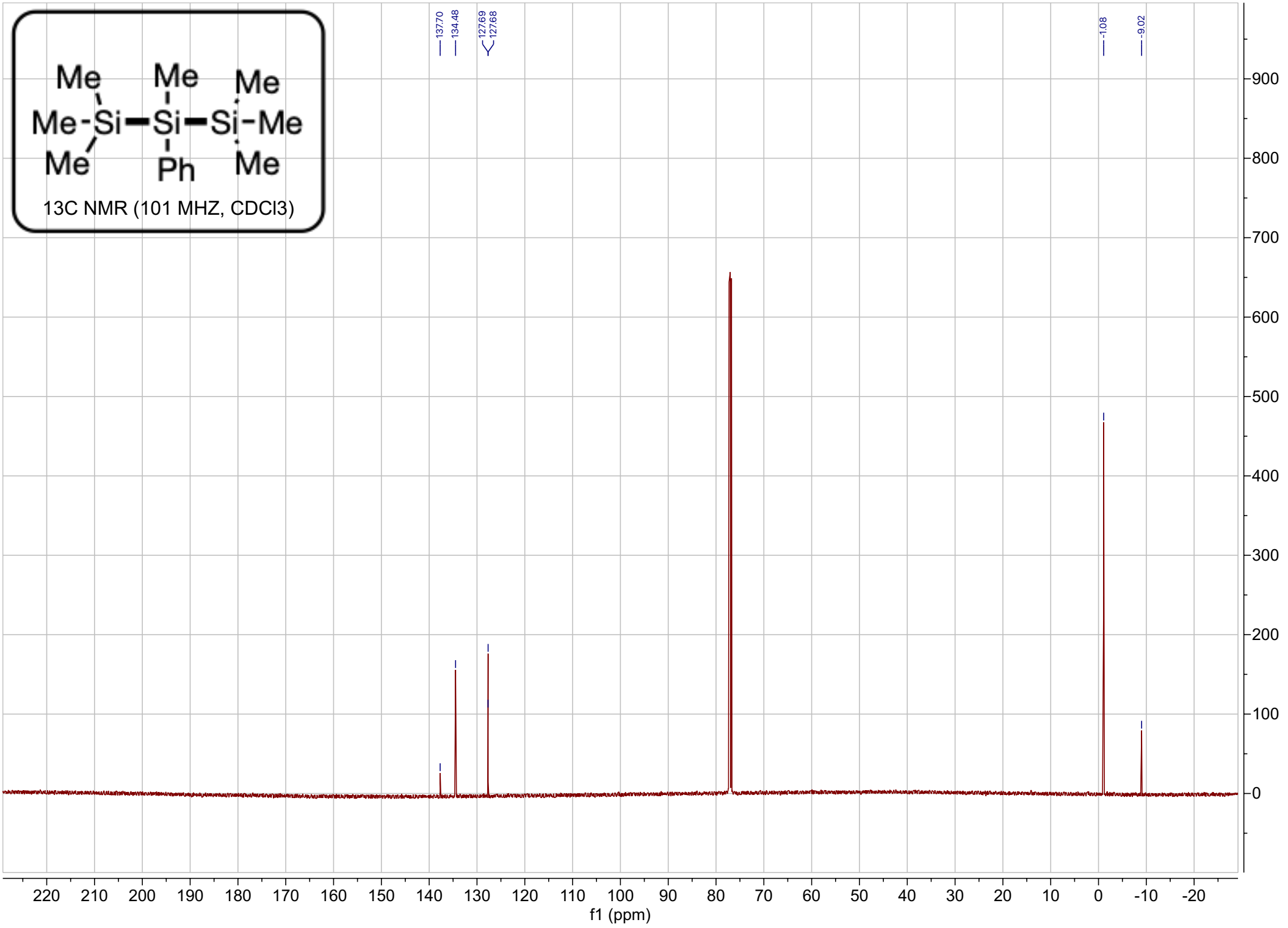
29Si NMR (99 MHZ, CDCl3)

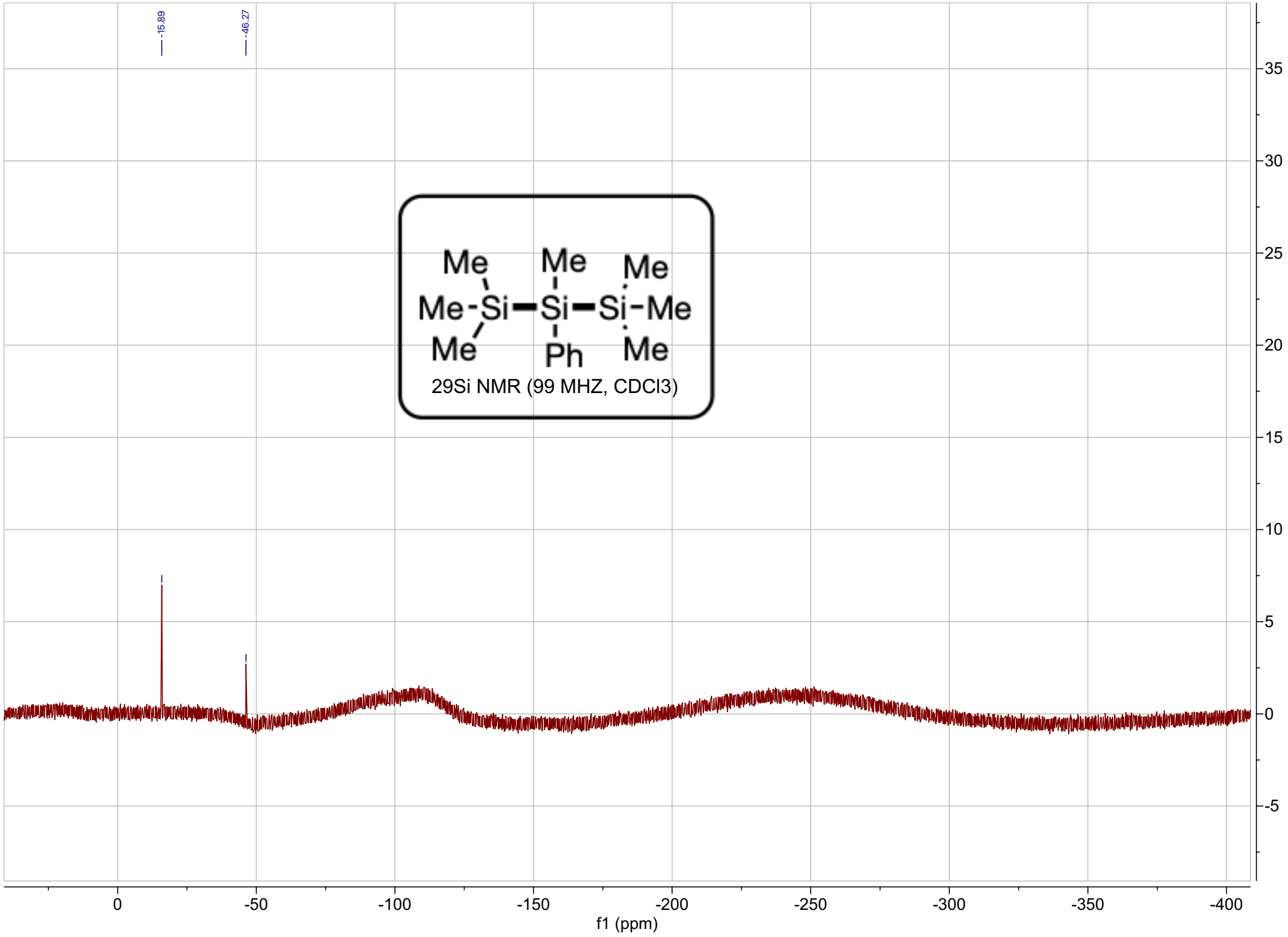

iPr
Me
iPr
H-Si-Si-Si-H
iPr
Ph
iPr
1H NMR (500 MHZ, CDCl3)
7.56
7.56
7.54
7.54
7.32
7.31
7.30
7.30
3.69
3.68
3.68
1.29
1.27
1.25
1.24
1.22
1.21
1.19
1.18
1.11
1.10
1.09
1.08
1.04
1.02
1.01
0.99
0.69
2.12
2.91
2.00
4.86
12.83
12.79
3.06
f1 (ppm)

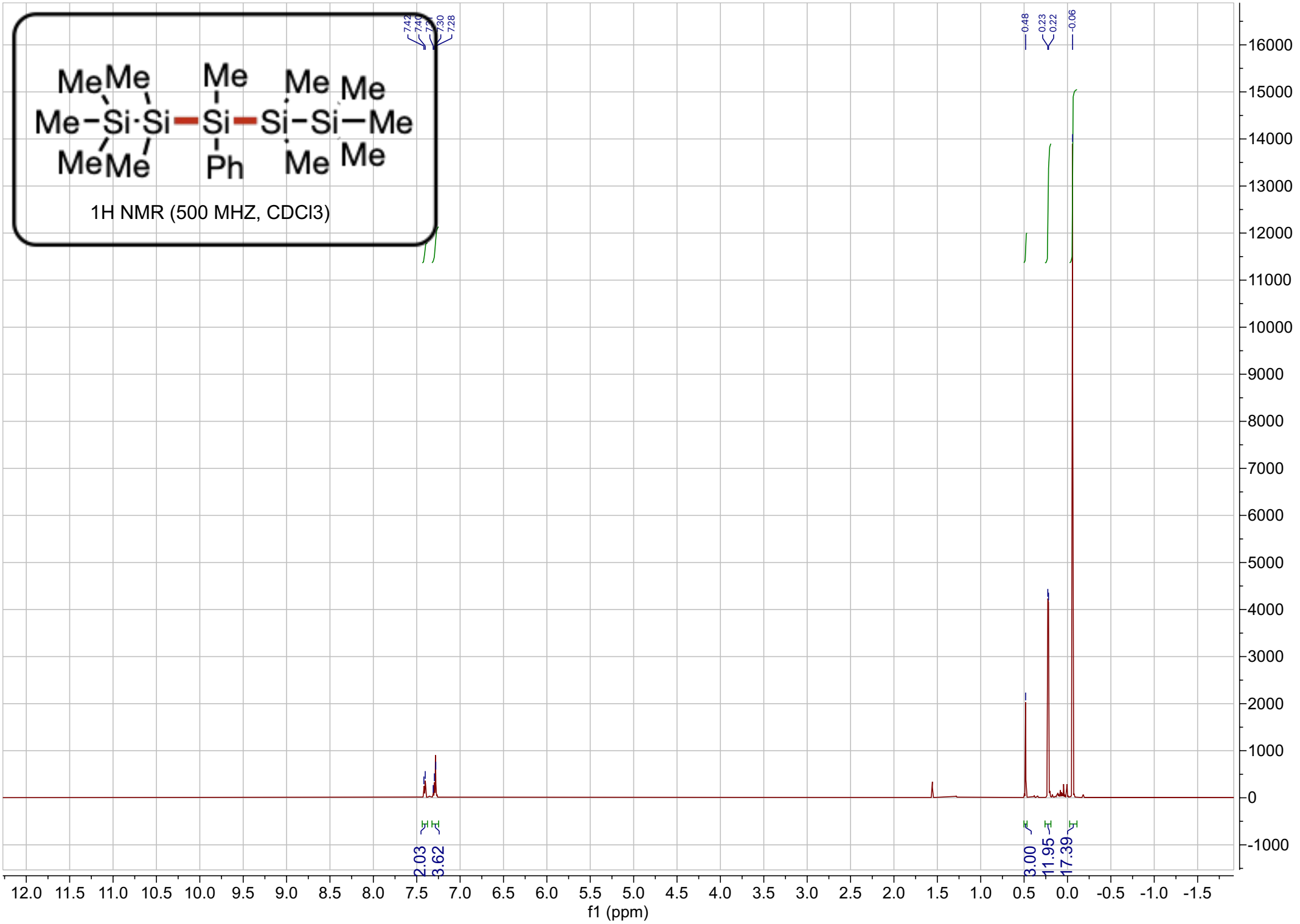

iPr
Me
iPr
H-Si-Si-Si-H
iPr
Ph
iPr
13C NMR (101 MHZ, CDCl3)
137.68
134.95
127.90
127.71
21.01
20.98
20.53
11.83
11.75
-4.91
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

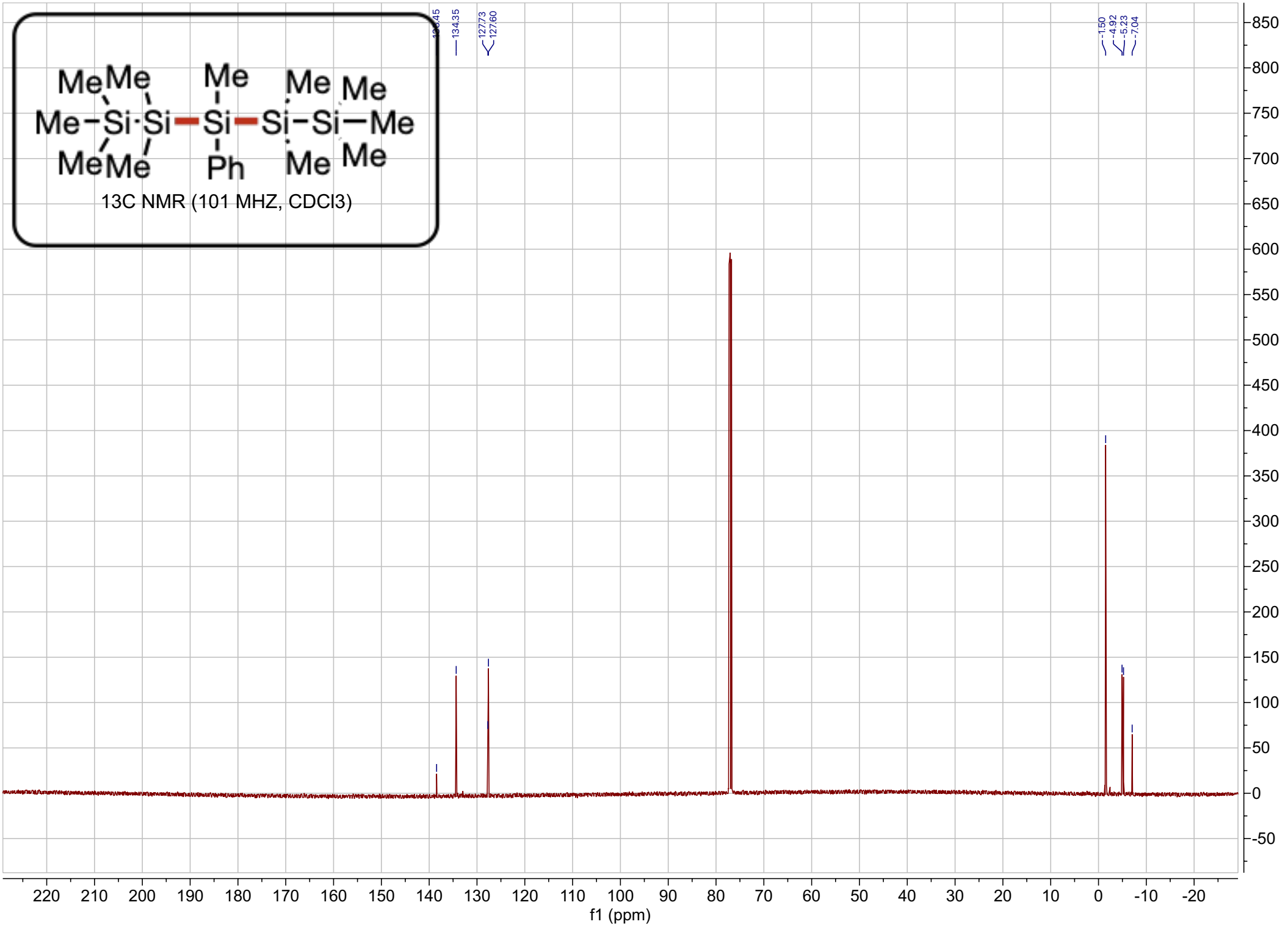

-10.16
-47.84
iPr
Me
iPr
H-Si
Si
Si-H
iPr
Ph
iPr
29Si NMR (99 MHZ, CDCl3)
34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
-2
-4
-6
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

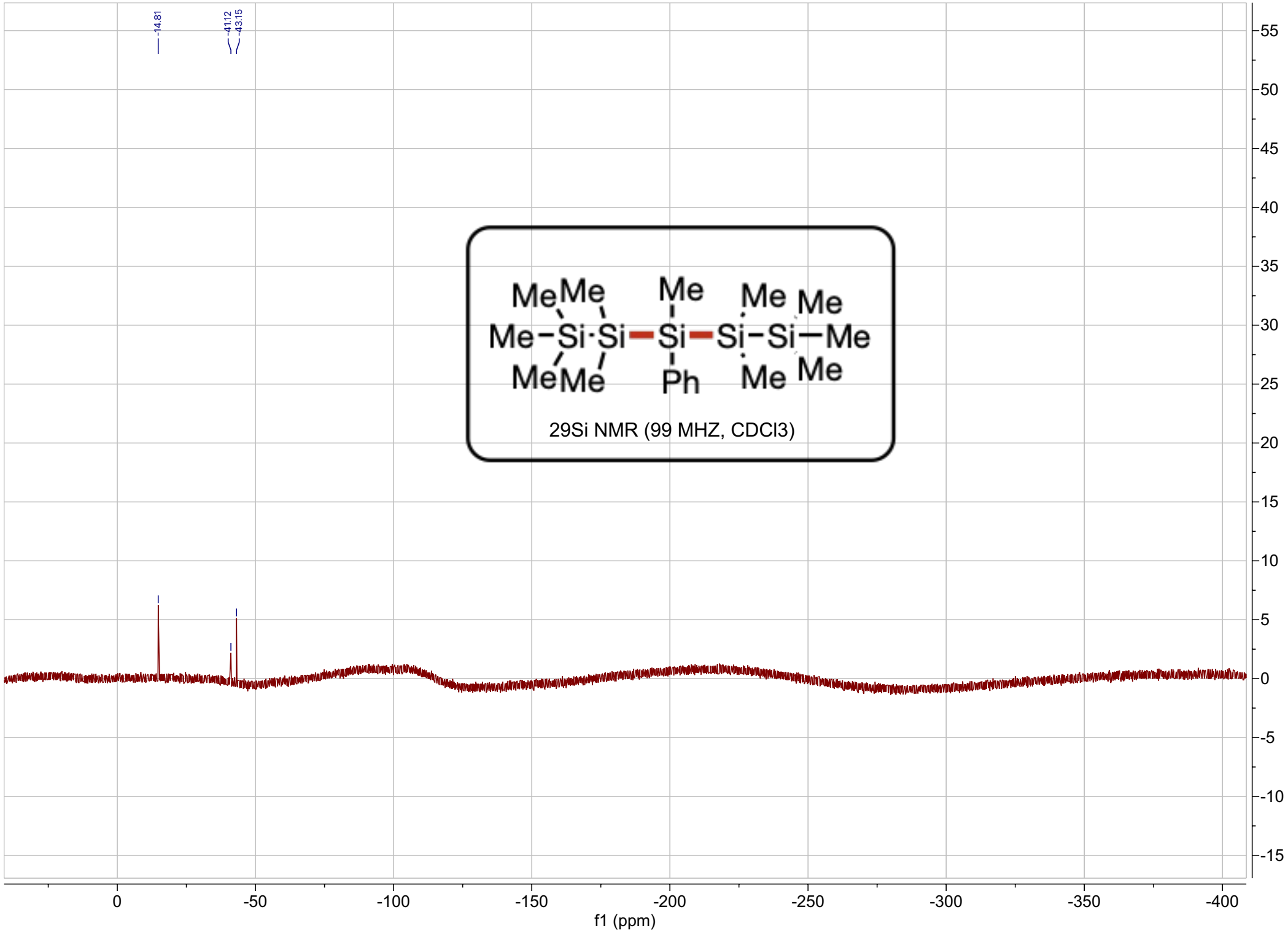

Me
Me
Me
Me-Si-Si-Si-Me
Me
Ph
Me
1H NMR (500 MHZ, CDCl3)
7.42
7.41
7.33
7.32
7.31
7.30
0.40
0.14
30000
25000
20000
15000
10000
5000
0
2.00
2.96
3.00
17.90
1.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

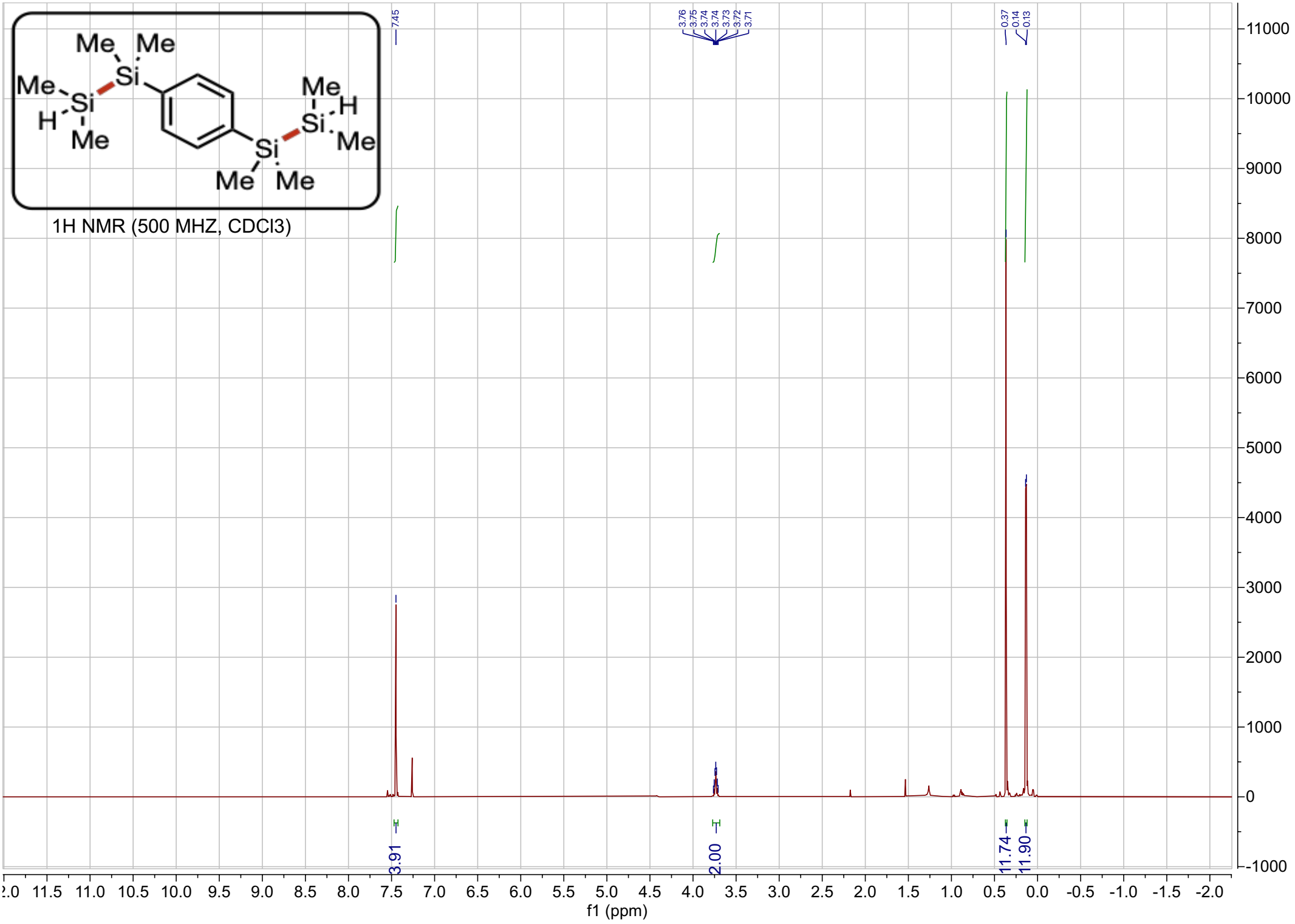

Me
Me
Me
Me-Si-Si-Si-Me
Me
Ph
Me
13C NMR (101 MHZ, CDCl3)
137.70
134.48
127.69
127.68
-1.08
-9.02
900
800
700
600
500
400
300
200
100
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

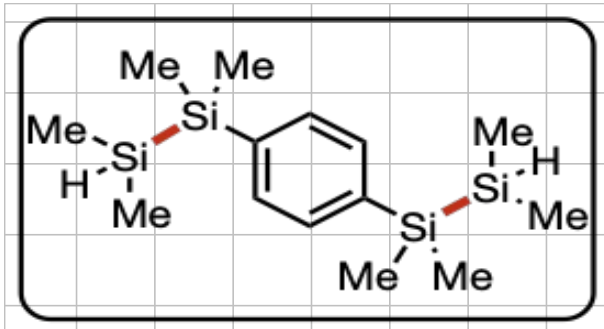

-15.89
-46.27
Me
Me
Me
Me-Si-Si-Si-Me
Me
Ph
Me
29Si NMR (99 MHZ, CDCl3)
35
30
25
20
15
10
5
0
-5
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)


1H NMR (500 MHZ, CDCl3)
7.42
7.40
7.30
7.28
0.48
0.23
0.22
-0.06
2.03
3.62
3.00
11.95
17.39
f1 (ppm)

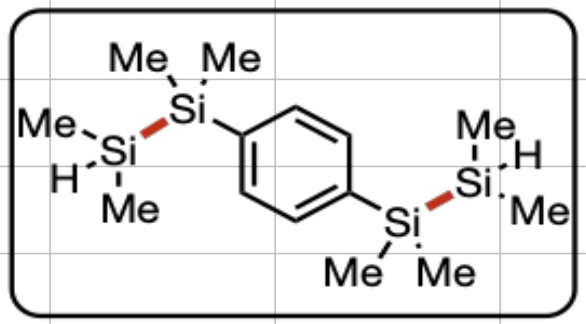

13C NMR (101 MHZ, CDCl3)
134.35
127.73
127.60
-1.50
-4.92
-5.23
-7.04
f1 (ppm)


-14.81
-41.12
-43.15
Me
Me
Me
Me
Me
Me
Si
Si
Si
Si
Si
Me
Me
Me
Me
Ph
Me
Me
29Si NMR (99 MHZ, CDCl3)
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

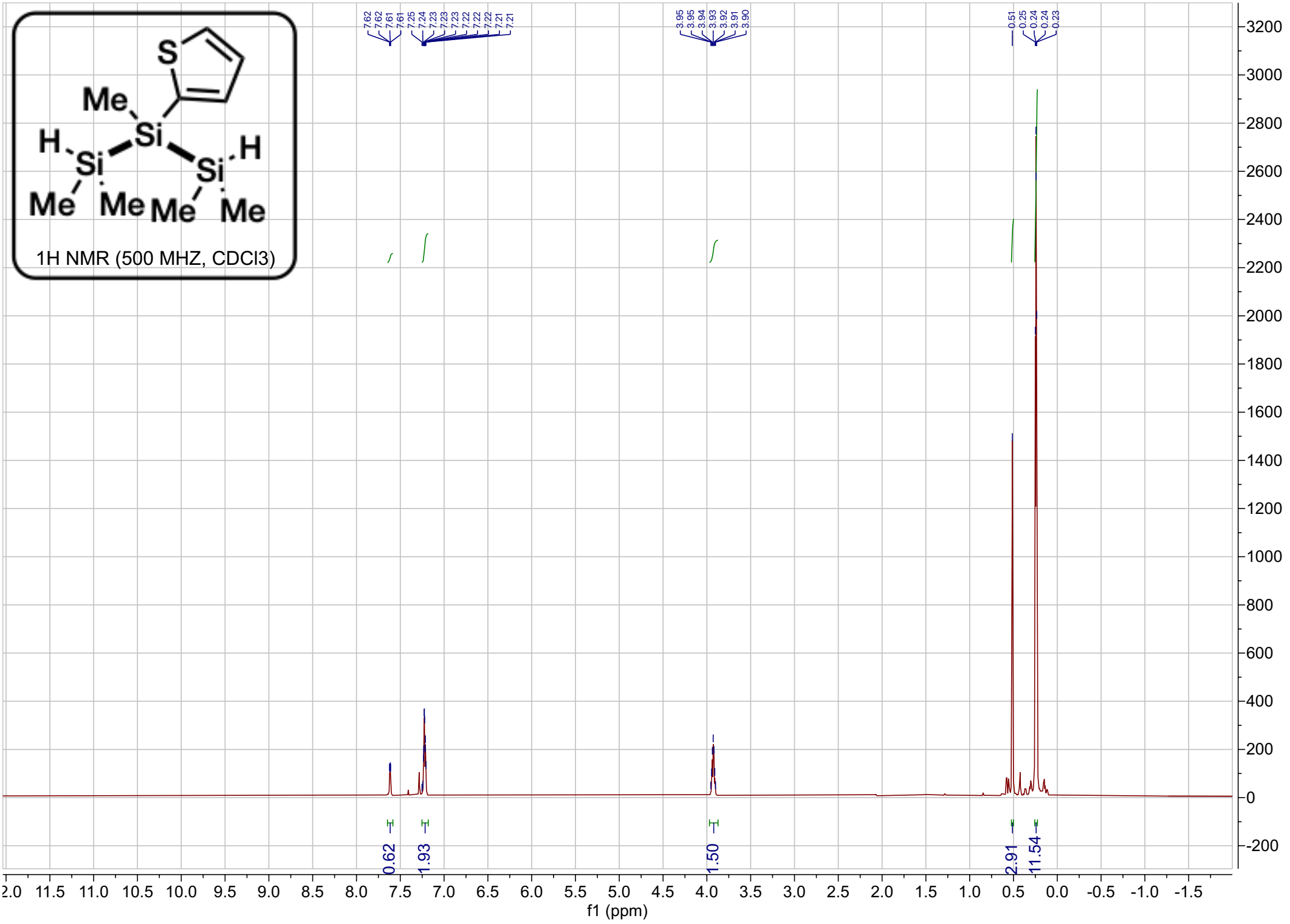

Me
Me
Si
Me
Si
H
Me
Si
Me
Si
H
Me
Me
Me
1H NMR (500 MHZ, CDCl3)
7.45
3.76
3.75
3.74
3.74
3.73
3.72
3.71
0.37
0.14
0.13
3.91
2.00
11.74
11.90
f1 (ppm)

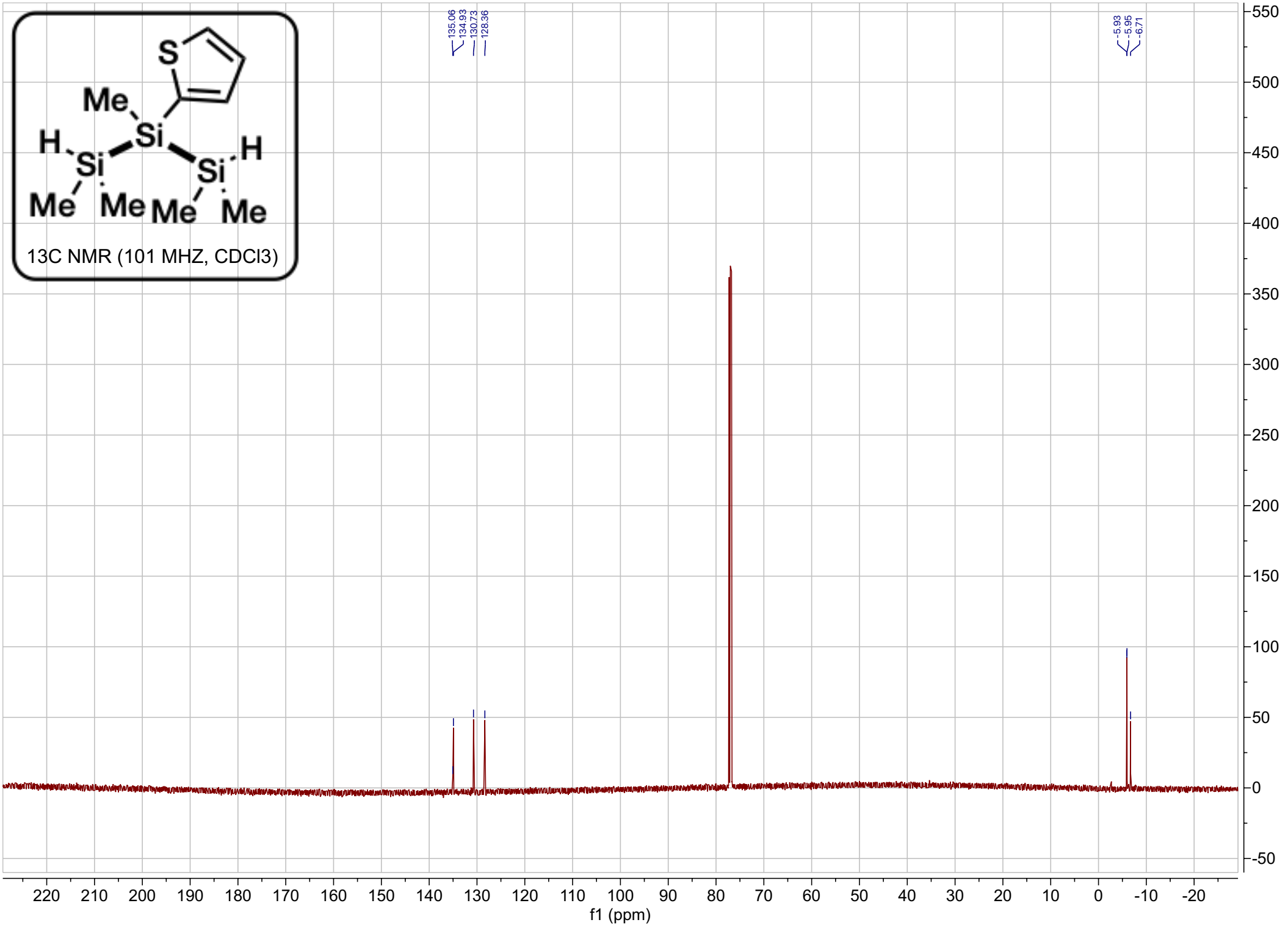


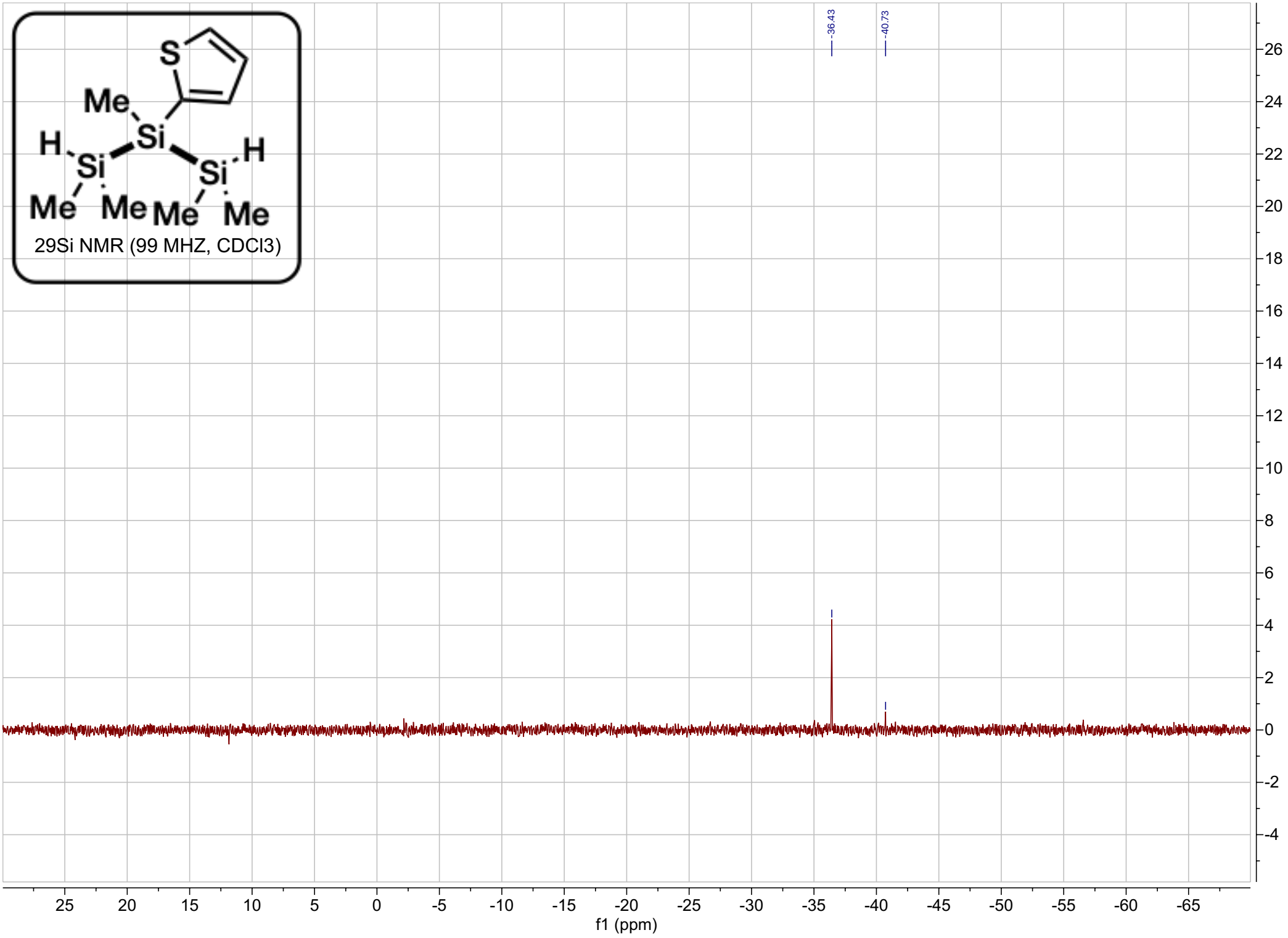
13C NMR (101 MHZ, CDCl3)

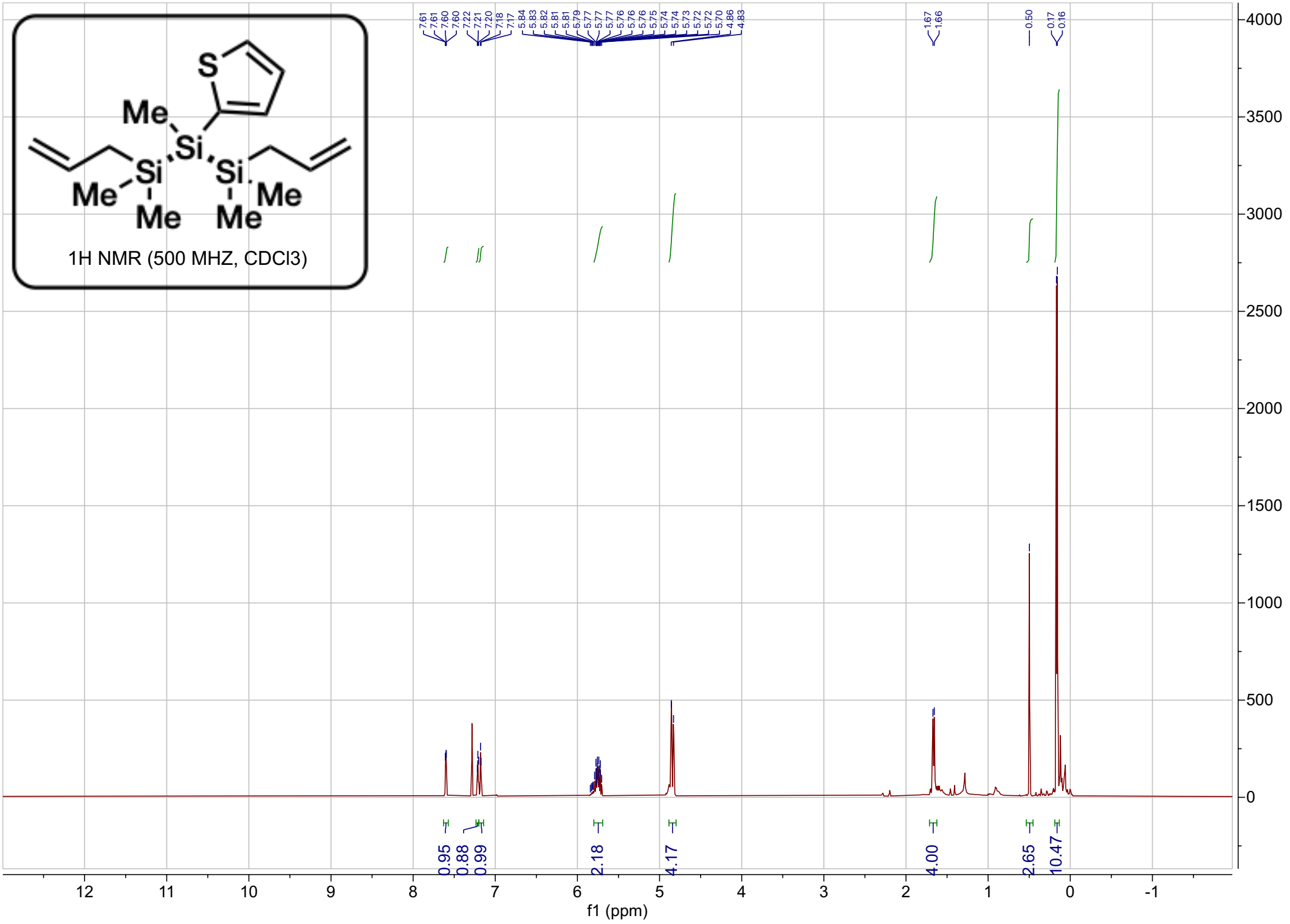


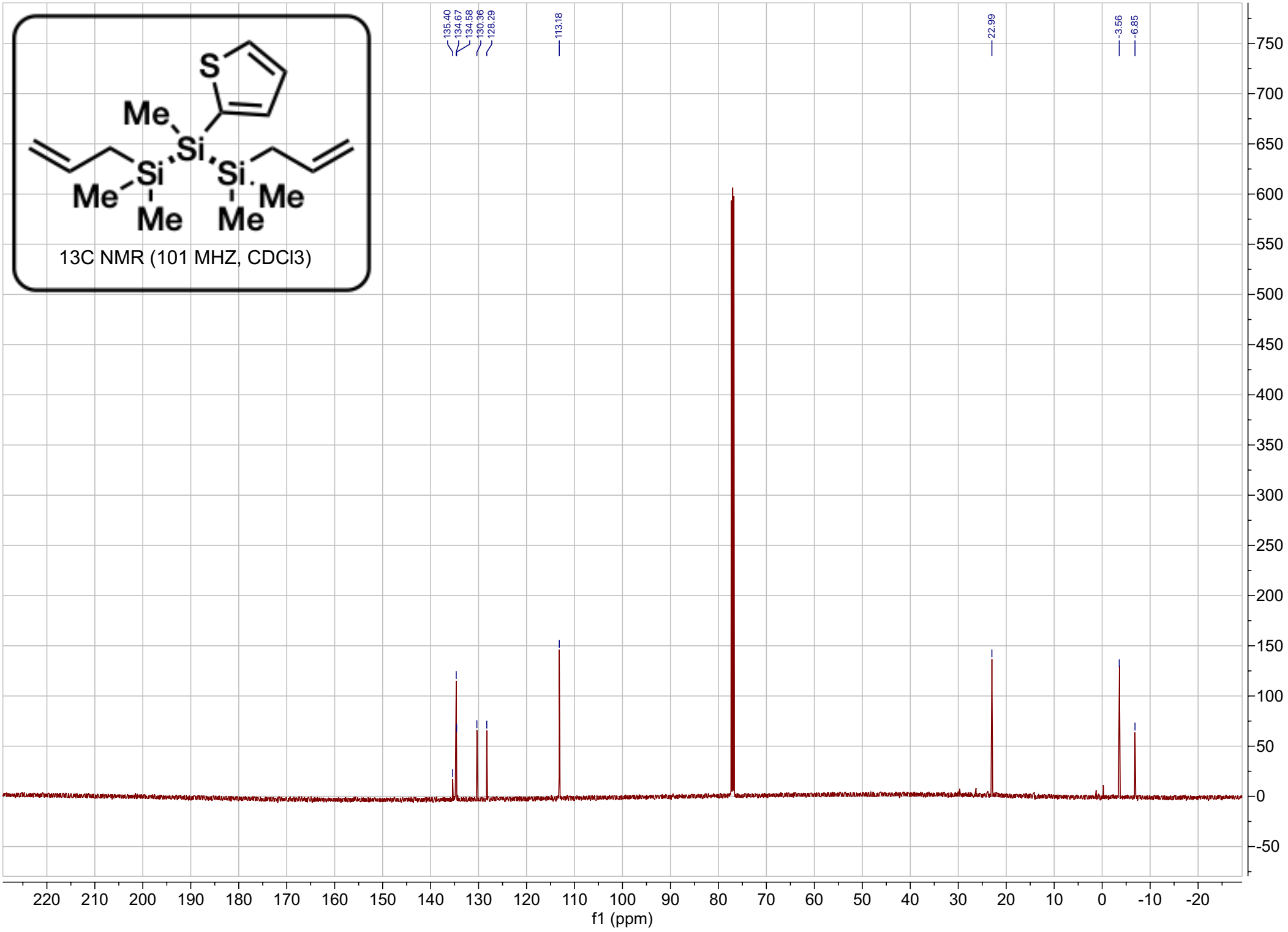
29Si NMR (99 MHZ, CDCl3)

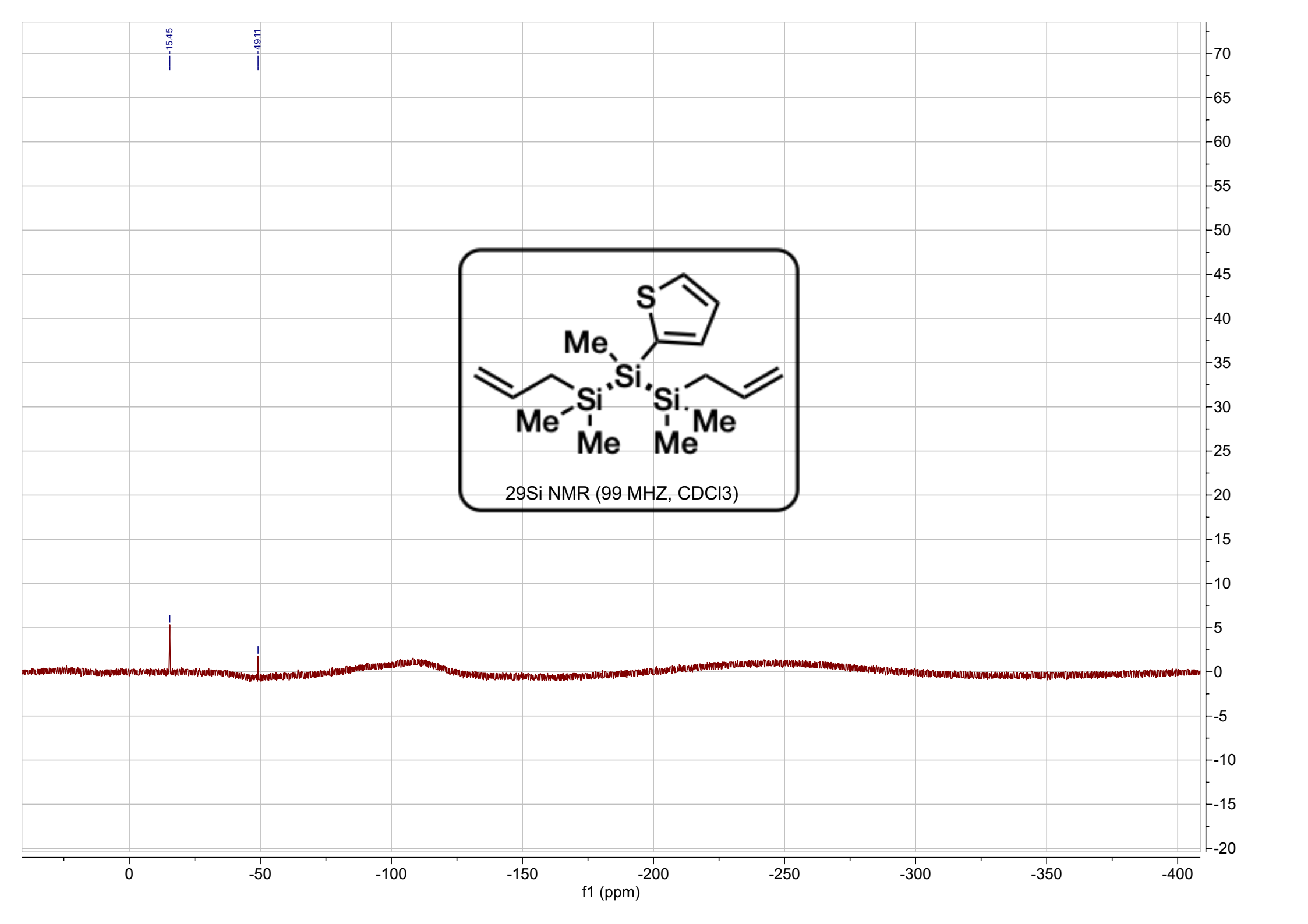

1H NMR (500 MHZ, CDCl3)
f1 (ppm)

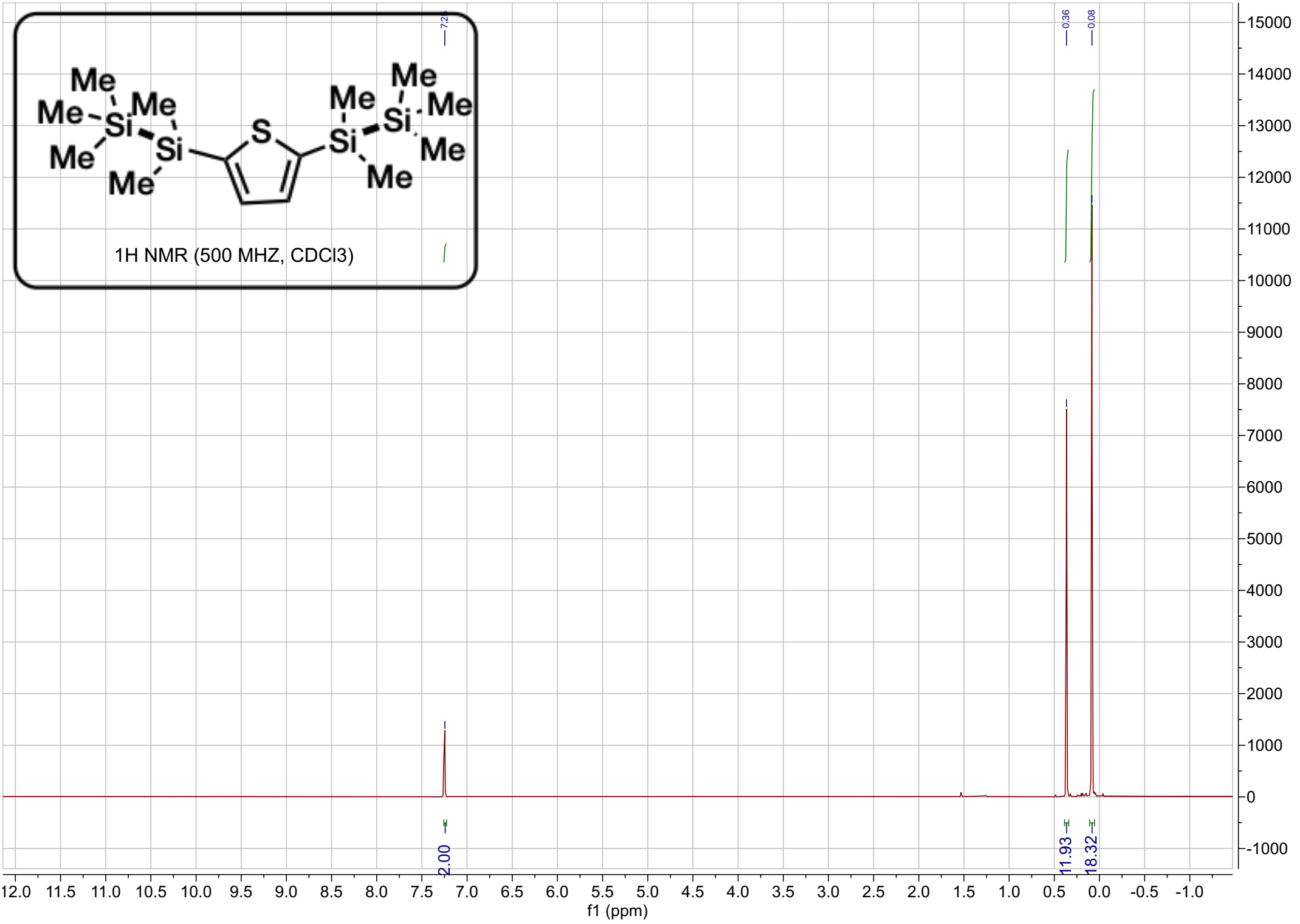

S
Me
Si
H
Si
Si
H
Me
Me
Me
Me
13C NMR (101 MHZ, CDCl3)
135.06
134.93
130.73
128.36
-5.93
-5.95
-6.71
550
500
450
400
350
300
250
200
150
100
50
0
-50
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

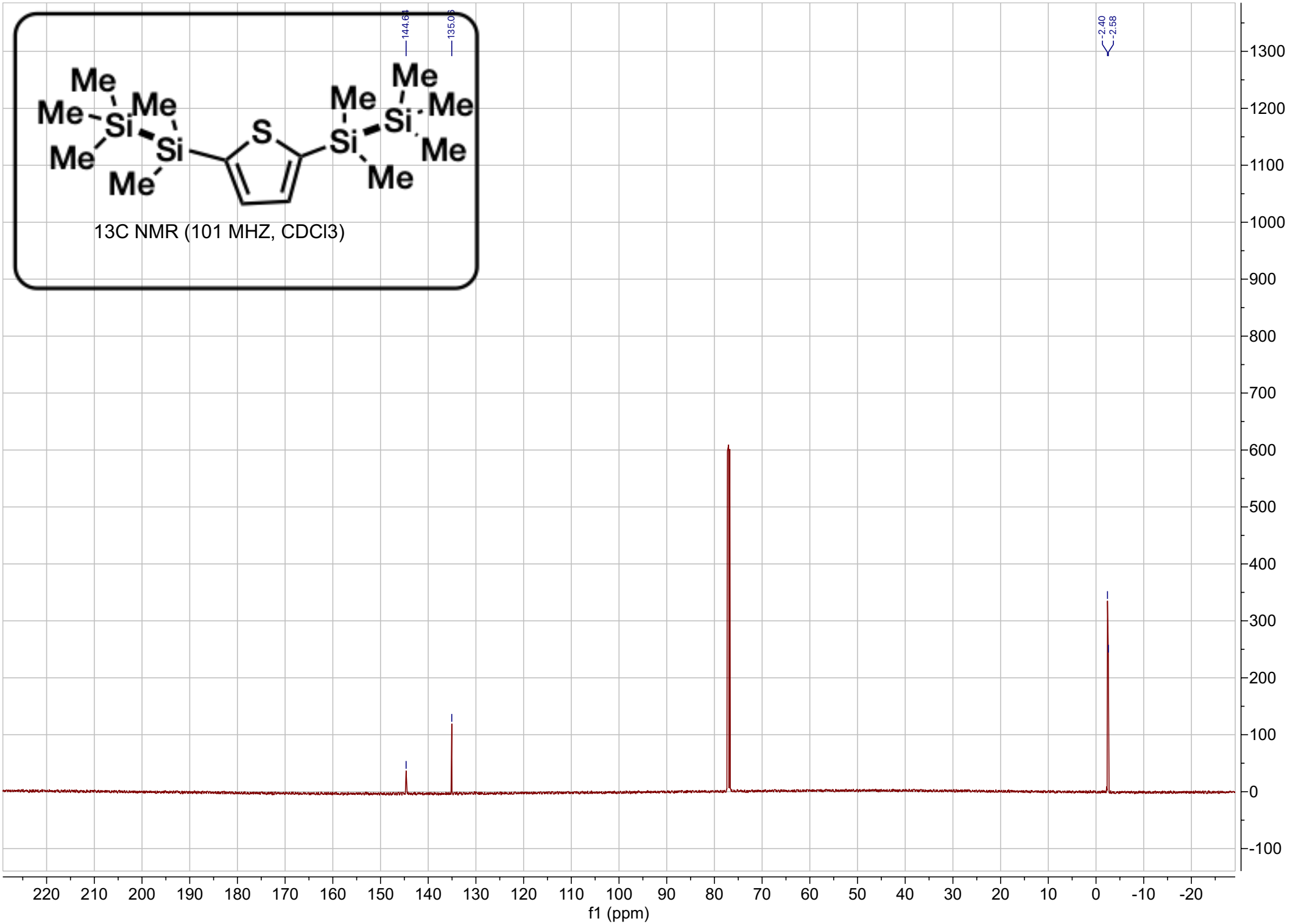

S
Me
Si
H
Si
Si
H
Me
Me
Me
Me
29Si NMR (99 MHZ, CDCl3)
-36.43
-40.73
26
24
22
20
18
16
14
12
10
8
6
4
2
0
-2
-4
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
f1 (ppm)

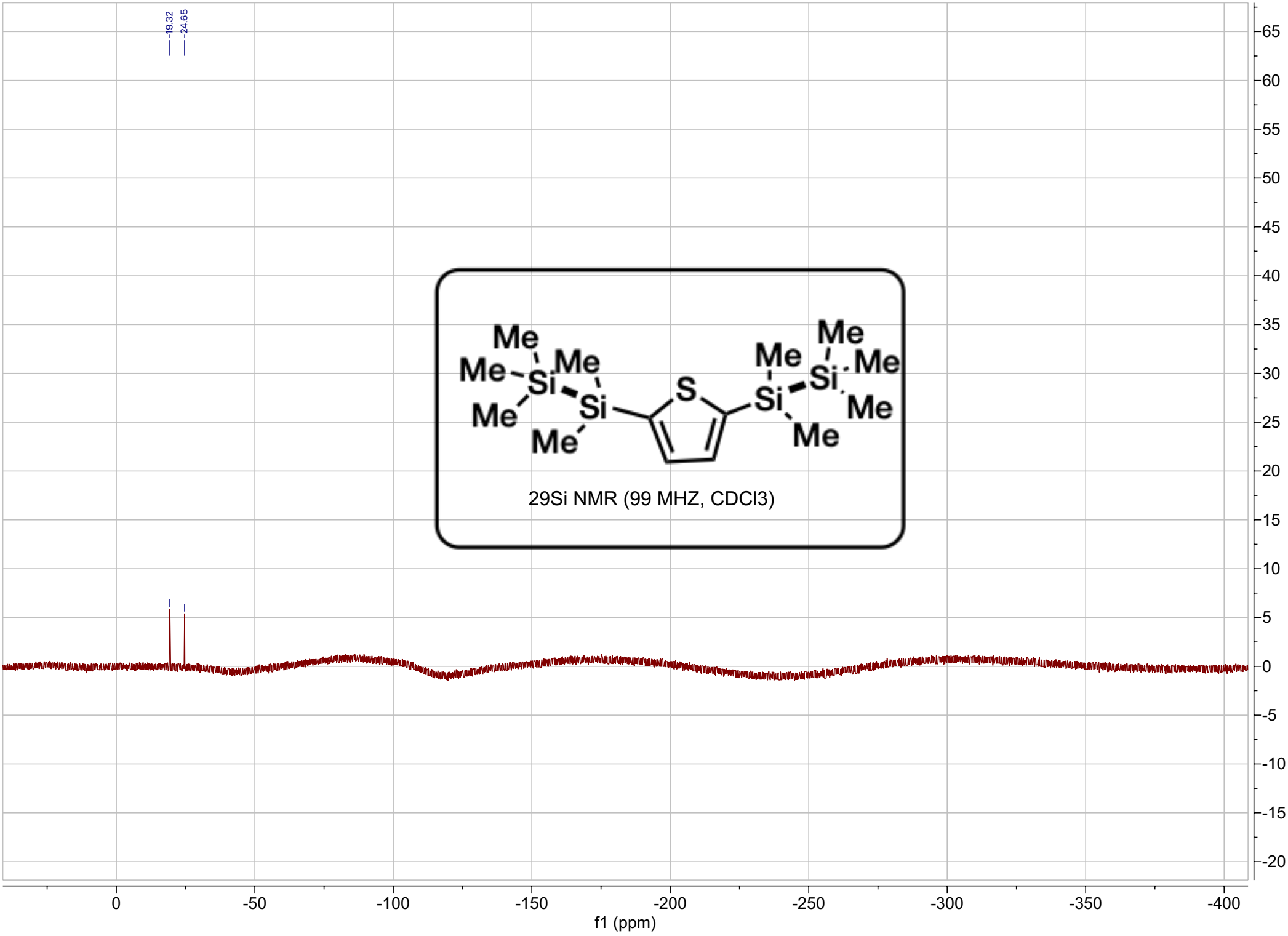

1H NMR (500 MHZ, CDCl3)
f1 (ppm)

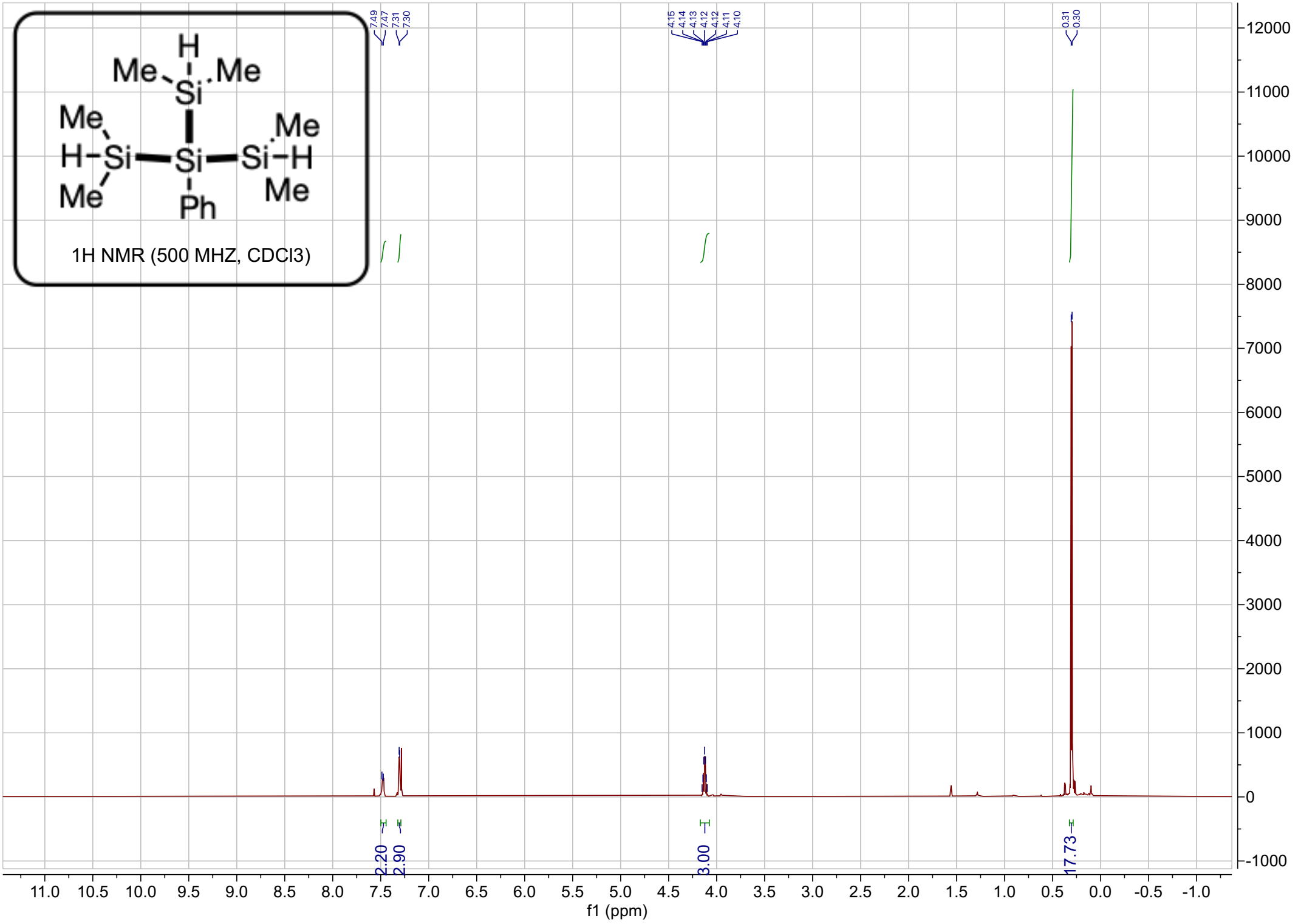

S
Me
Si
Si
Si
Me
Me
Me
Me
Me
13C NMR (101 MHZ, CDCl3)
135.40
134.67
134.58
130.36
128.29
113.18
22.99
-3.56
-6.85
750
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

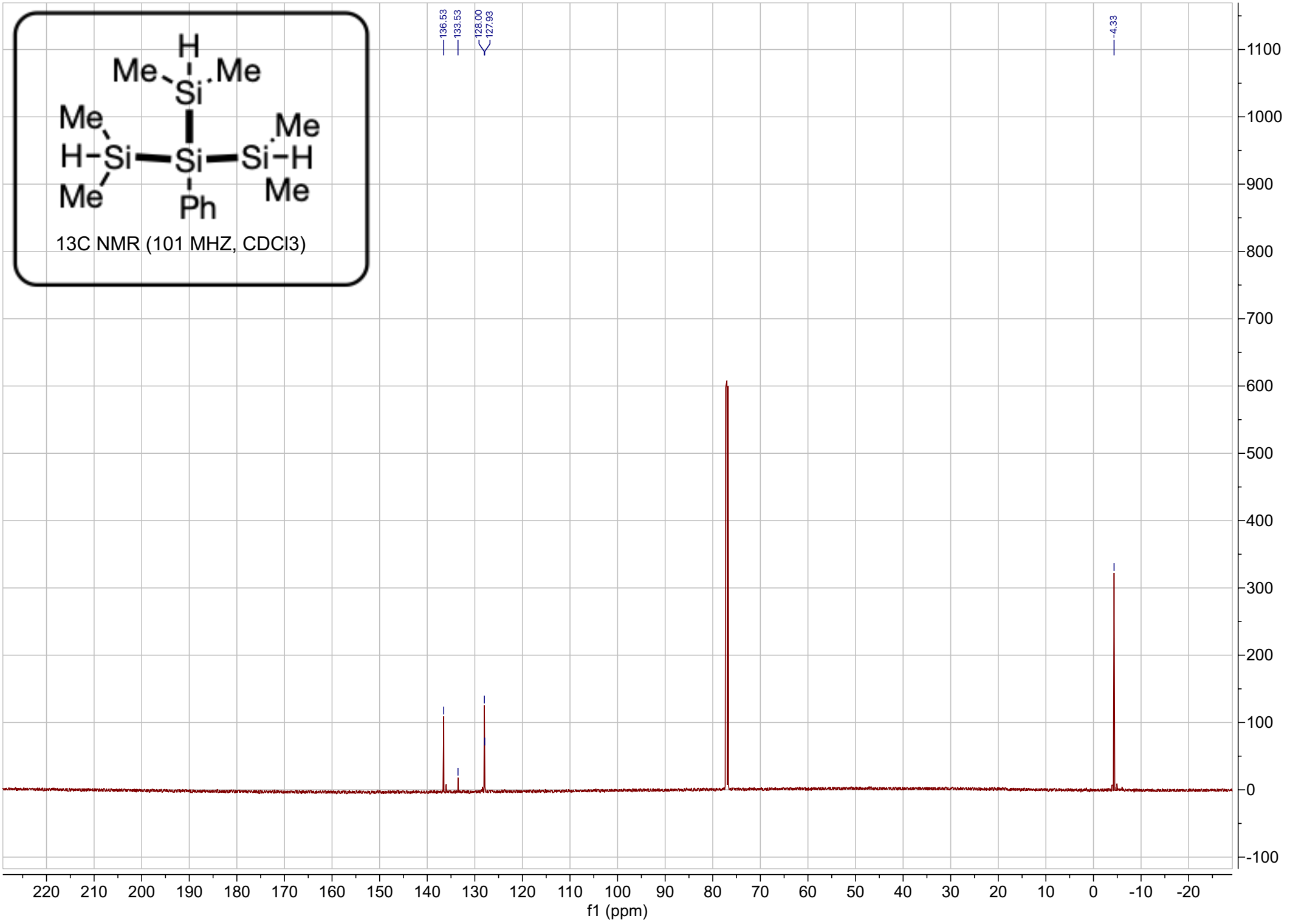

-15.45
-49.11
S
Me
Si
Si
Si
Me
Me
Me
Me
29Si NMR (99 MHZ, CDCl3)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

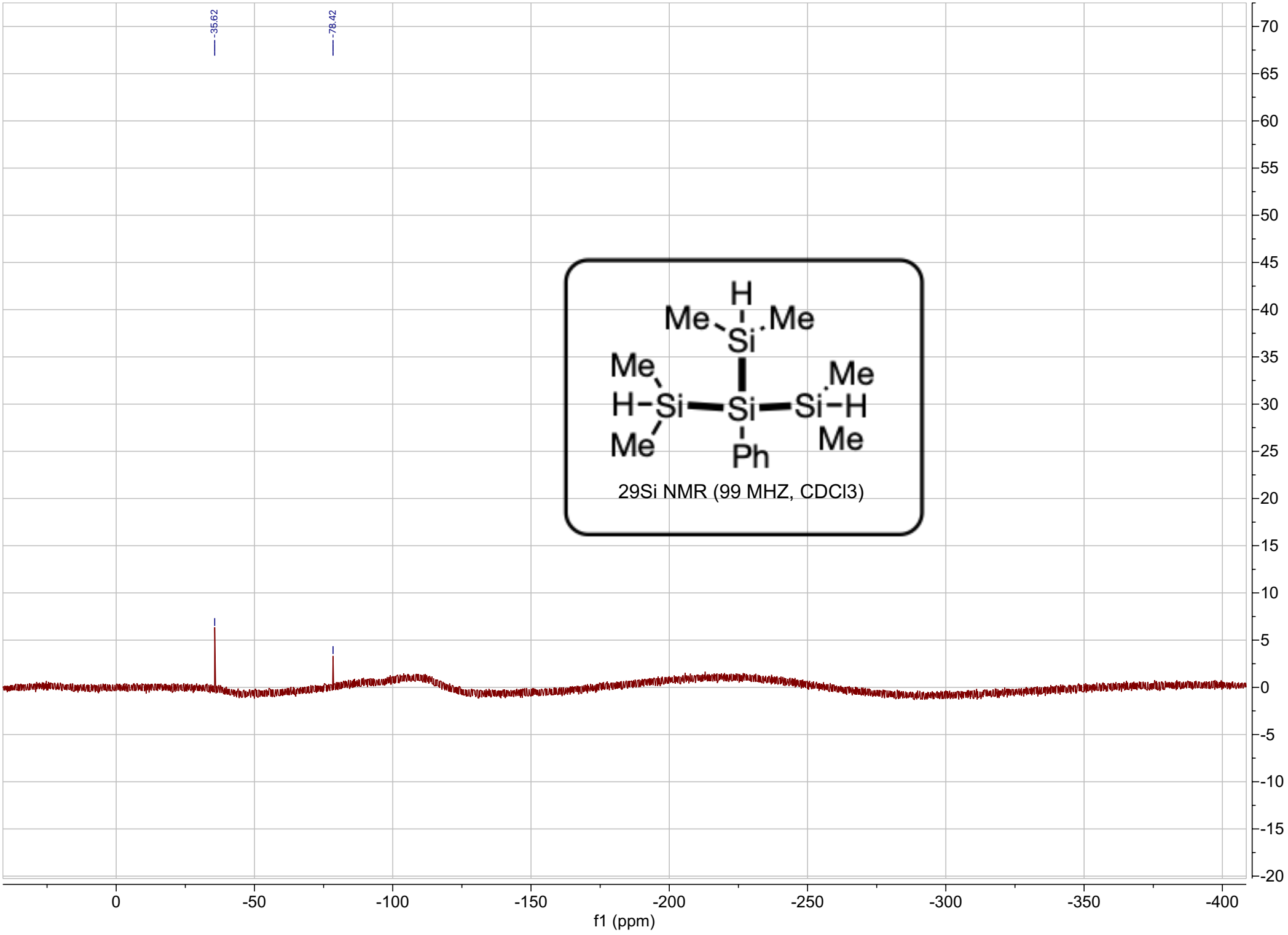

Me
Me
Me
Si
Si
Me
Me
S
Me
Si
Si
Me
Me
Me
1H NMR (500 MHZ, CDCl3)
7.25
0.36
0.08
2.00
11.93
18.32
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

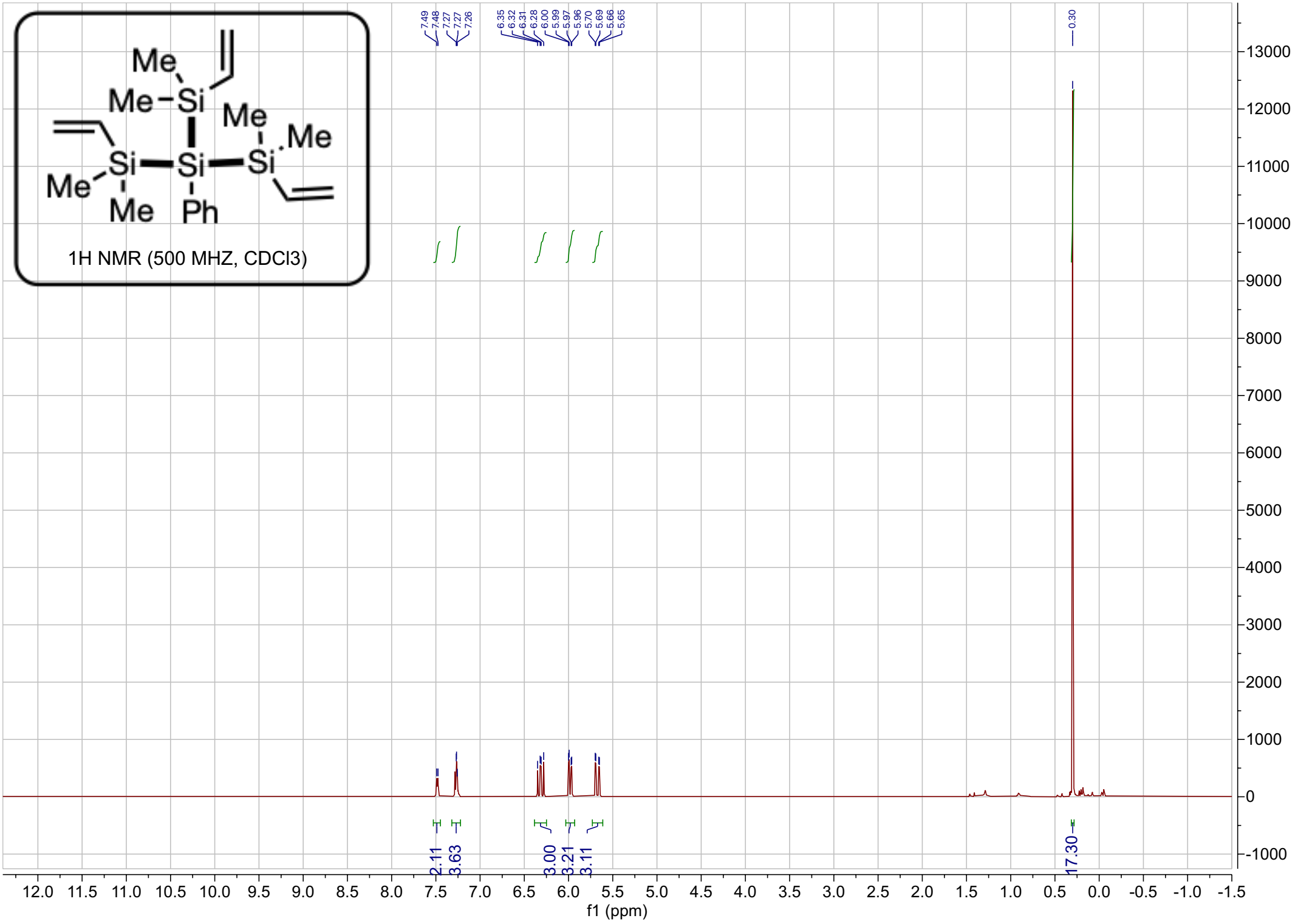

144.61
135.06
2.40
2.58
Me
Me
Me
Si
Si
Me
Me
S
Si
Me
Si
Me
Me
Me
13C NMR (101 MHZ, CDCl3)
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

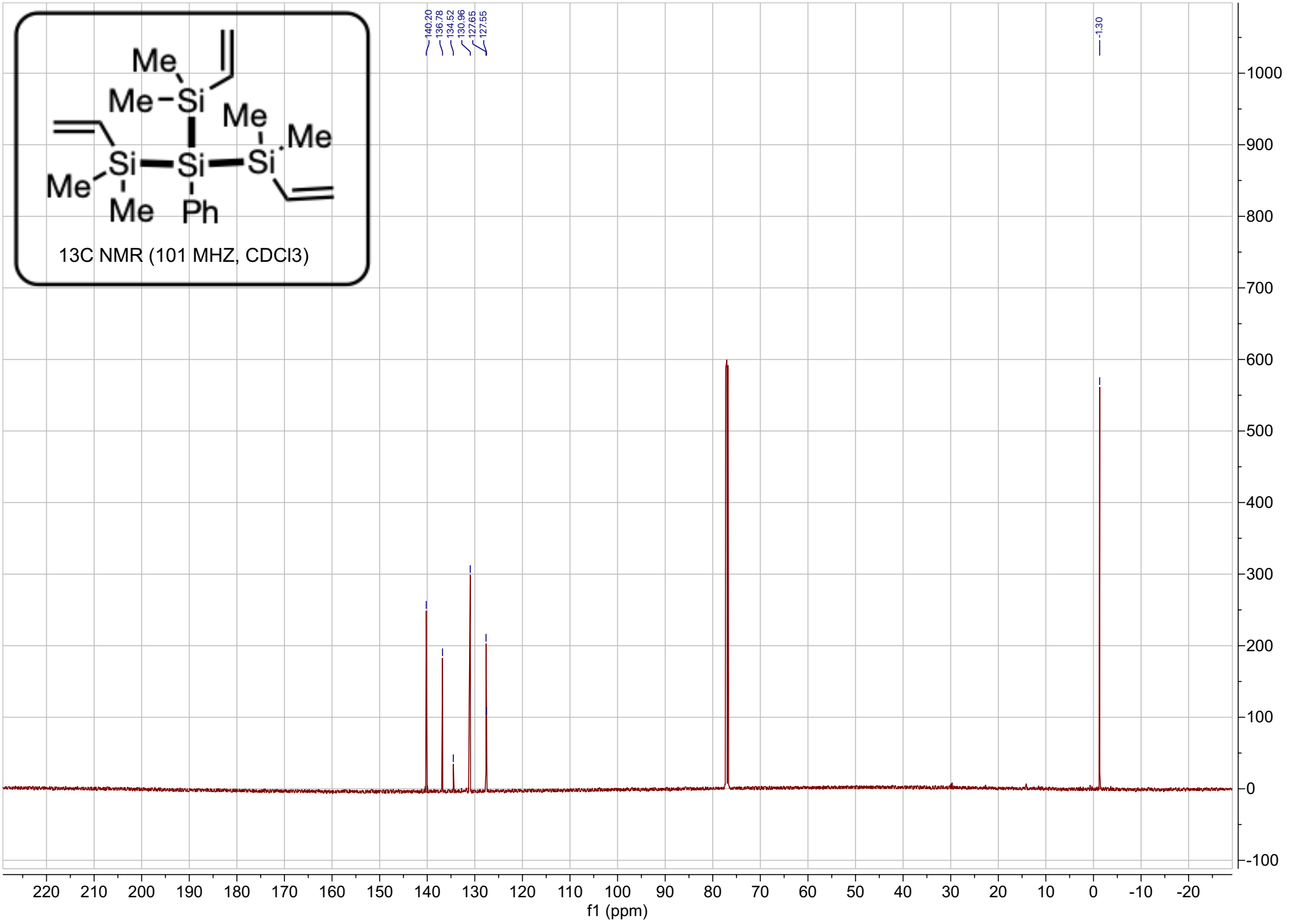

-19.32
-24.65
Me
Me
Me
Si
Si
Me
Me
S
Si
Me
Me
Si
Me
Me
29Si NMR (99 MHZ, CDCl3)
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

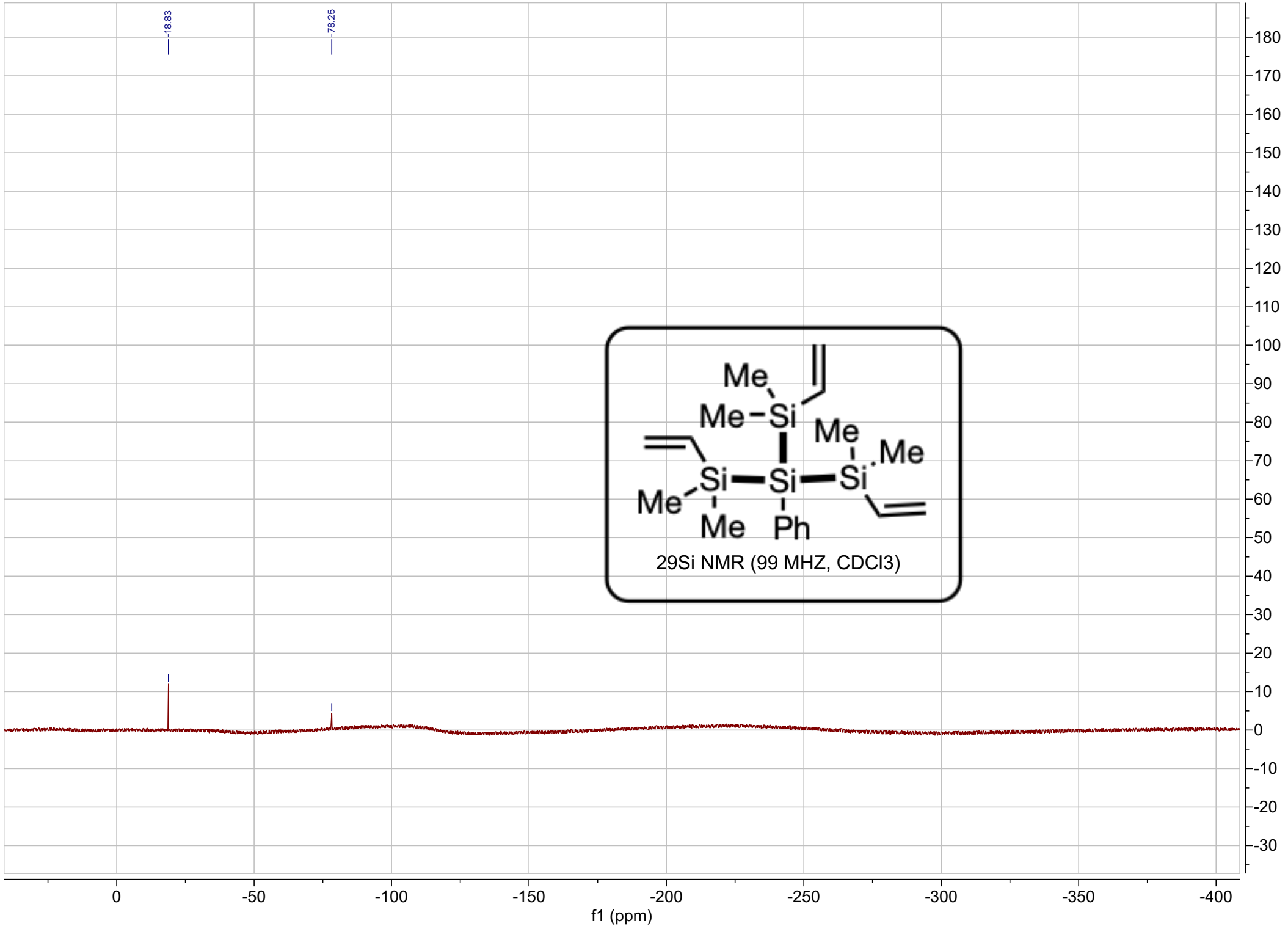

1H NMR (500 MHZ, CDCl3)
7.49
7.47
7.31
7.30
4.15
4.14
4.13
4.12
4.12
4.11
4.10
0.31
0.30
2.20
2.90
3.00
17.73
f1 (ppm)

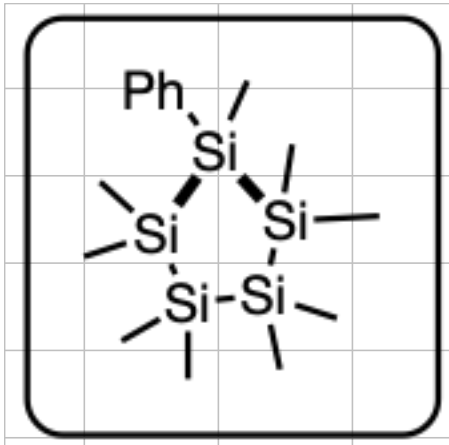

136.53
133.53
128.00
127.93
-4.33
13C NMR (101 MHZ, CDCl3)
f1 (ppm)


-35.62
-78.42
H
Me
Me
Si
Me
Me
H
Si
Si
Si
H
Me
Me
Ph
29Si NMR (99 MHZ, CDCl3)
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)

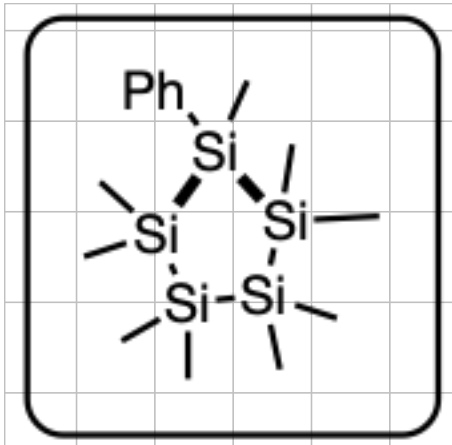

Me
Me
Si
Me
Me
Si
Si
Si
Me
Me
Me
Ph
1H NMR (500 MHZ, CDCl3)
7.49
7.48
7.27
7.27
7.26
6.35
6.32
6.31
6.28
6.00
5.99
5.97
5.96
5.70
5.69
5.66
5.65
0.30
2.11
3.63
3.00
3.21
3.11
17.30
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 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 -1.0 -1.5
f1 (ppm)


Me
Me
Si
Me
Me
Si
Si
Si
Me
Me
Me
Ph
13C NMR (101 MHZ, CDCl3)
140.20
136.78
134.52
130.96
127.65
127.55
-1.30
1000
900
800
700
600
500
400
300
200
100
0
-100
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

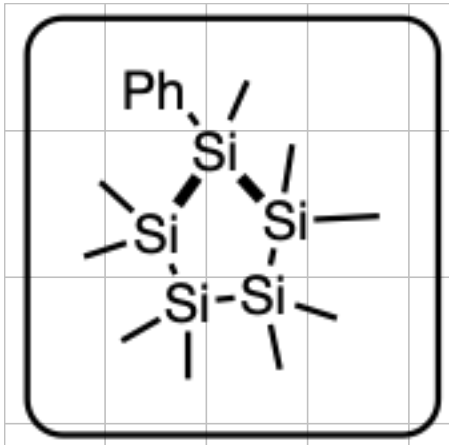

-18.83
-78.25
Me
Me
Si
Me
Me
Si
Si
Si
Me
Me
Ph
29Si NMR (99 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)



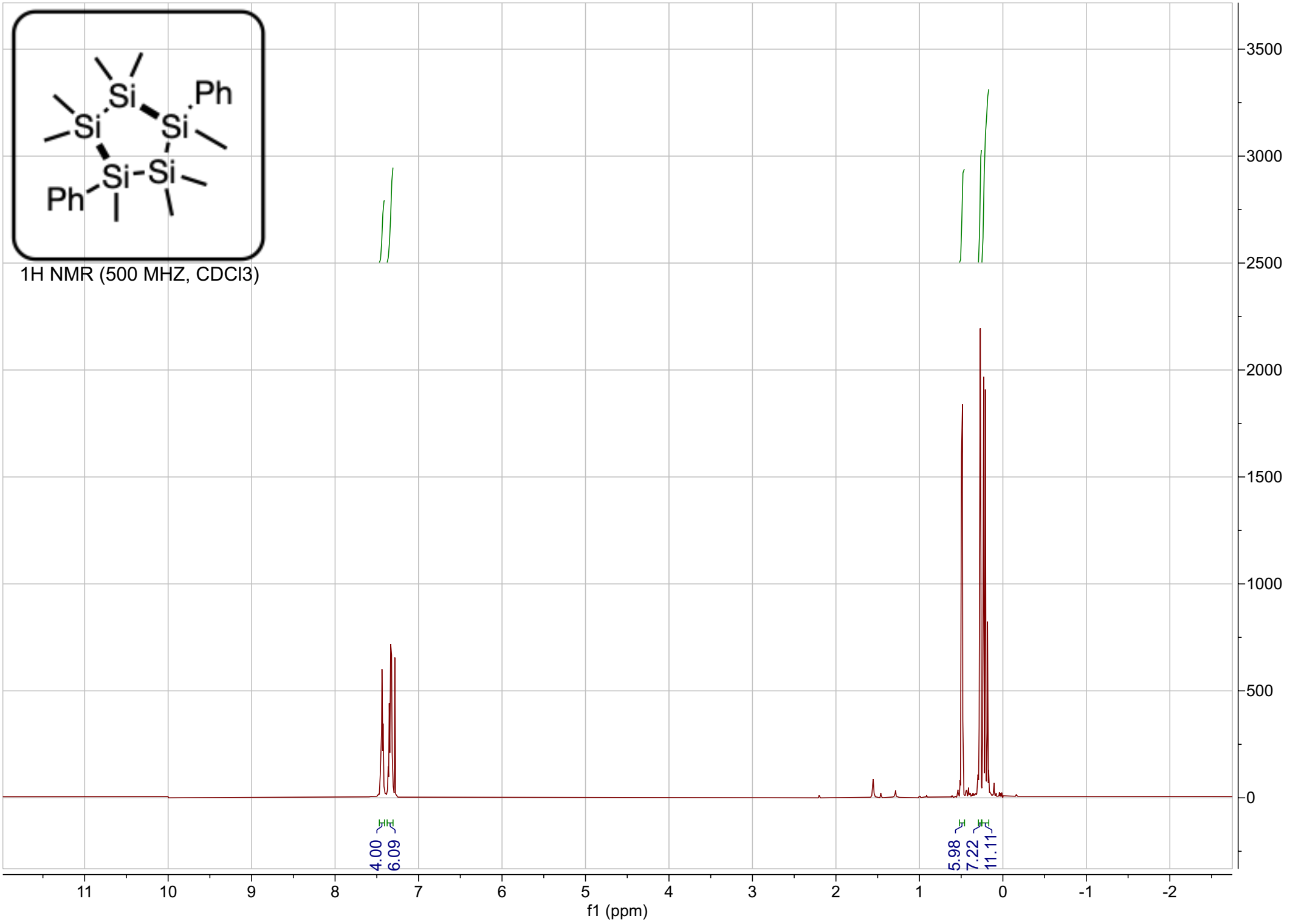
1H NMR (500 MHZ, CDCl3)

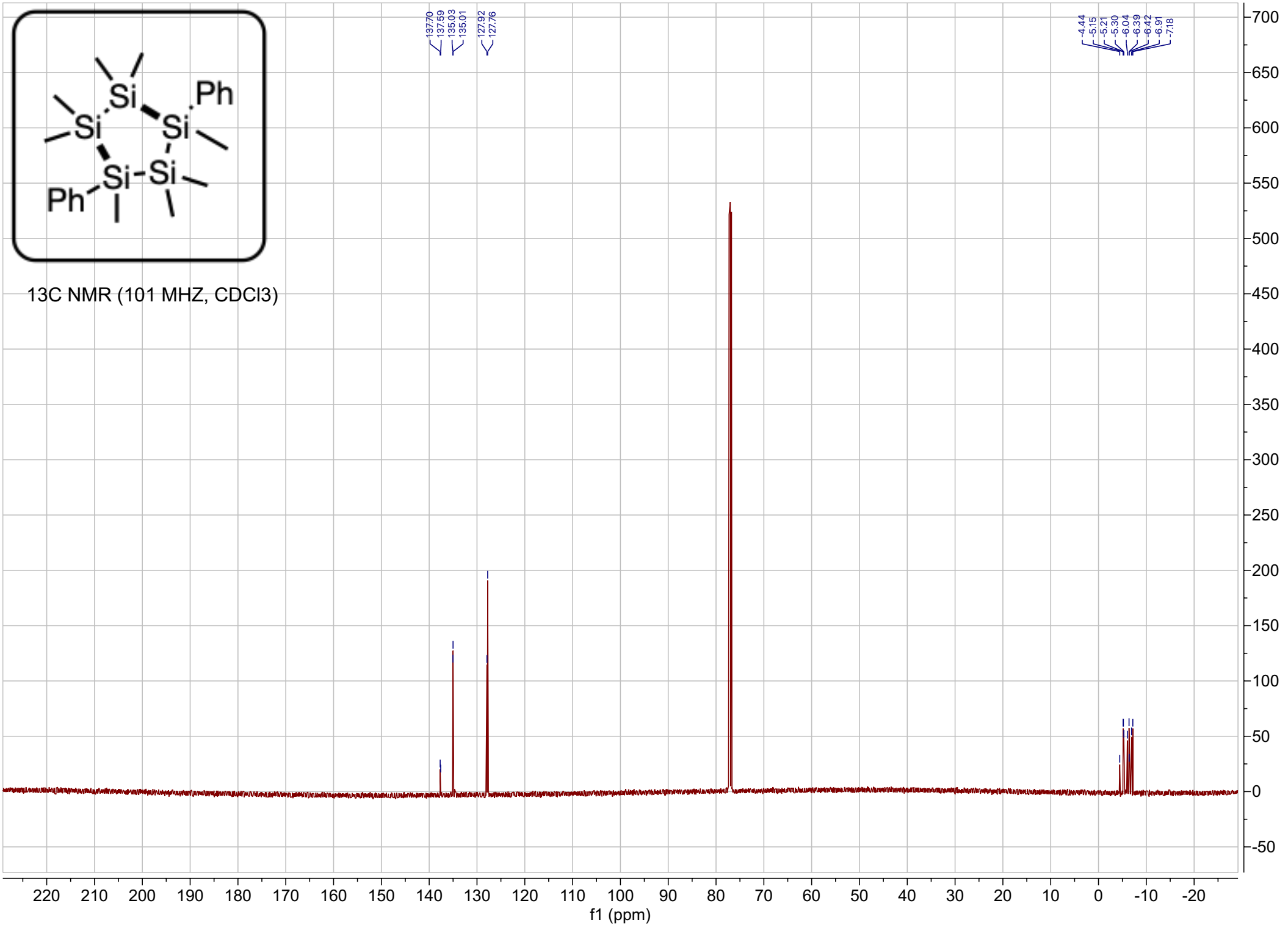


13C NMR (101 MHZ, CDCl3)

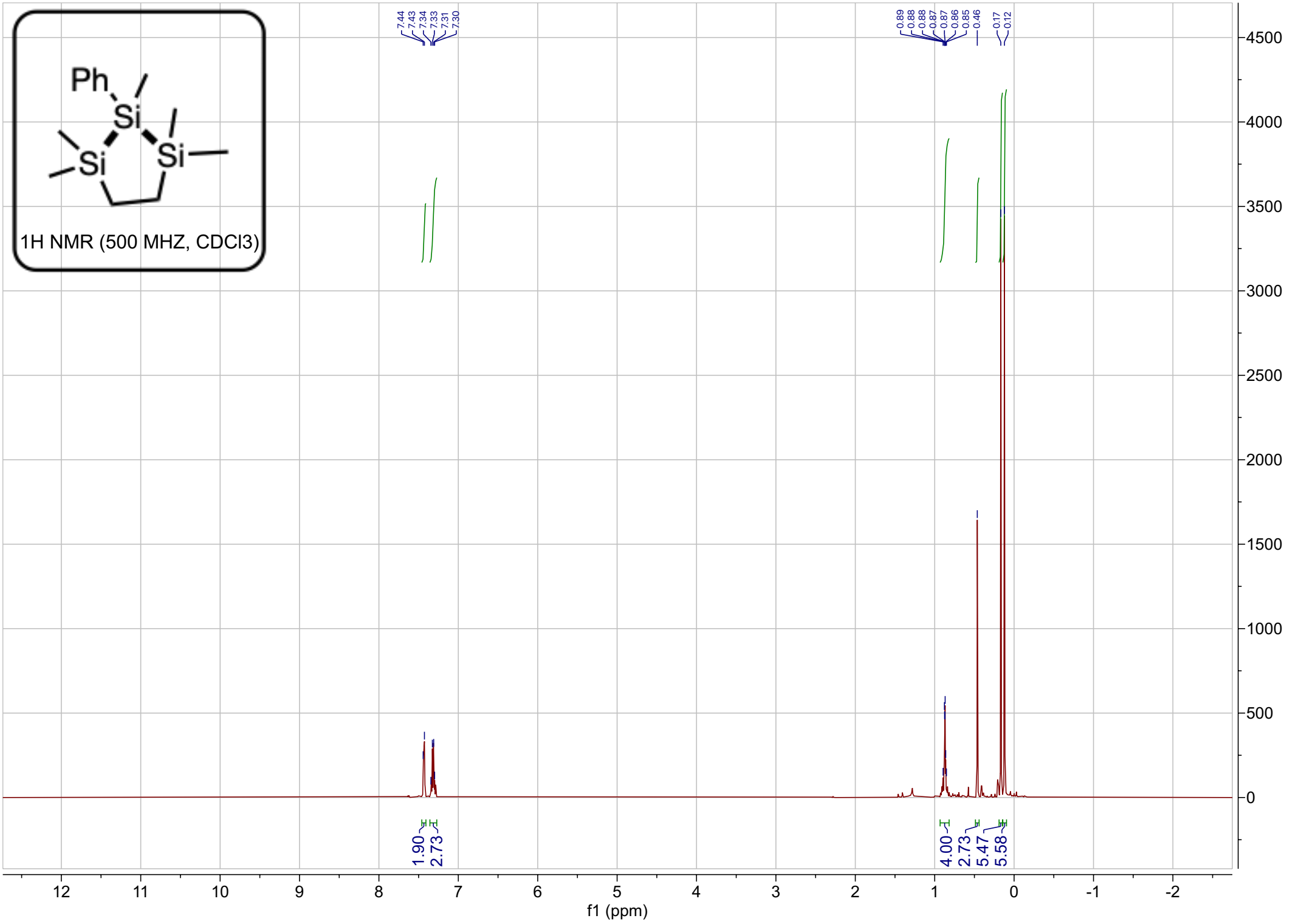


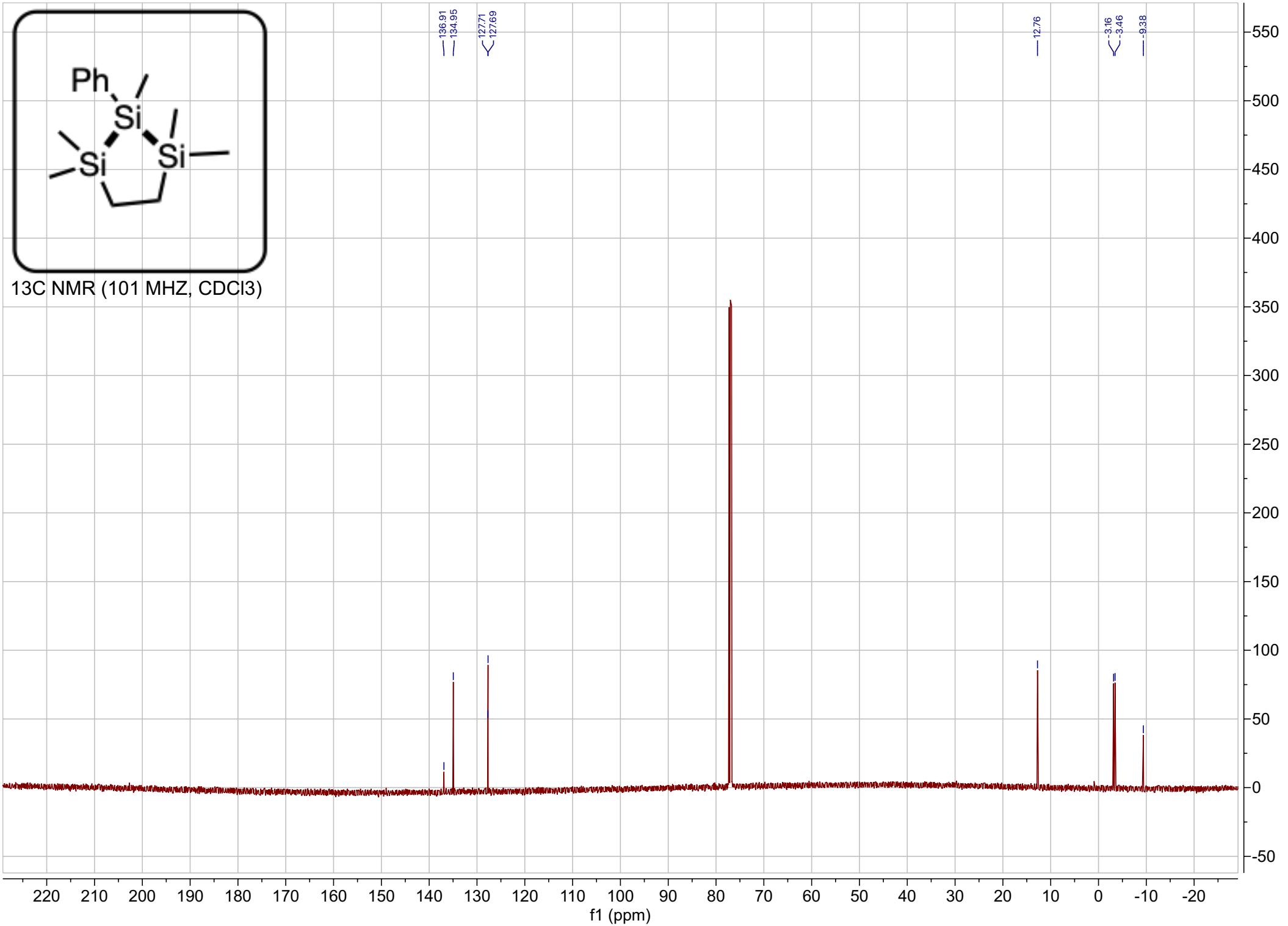
29Si NMR (99 MHZ, CDCl3)

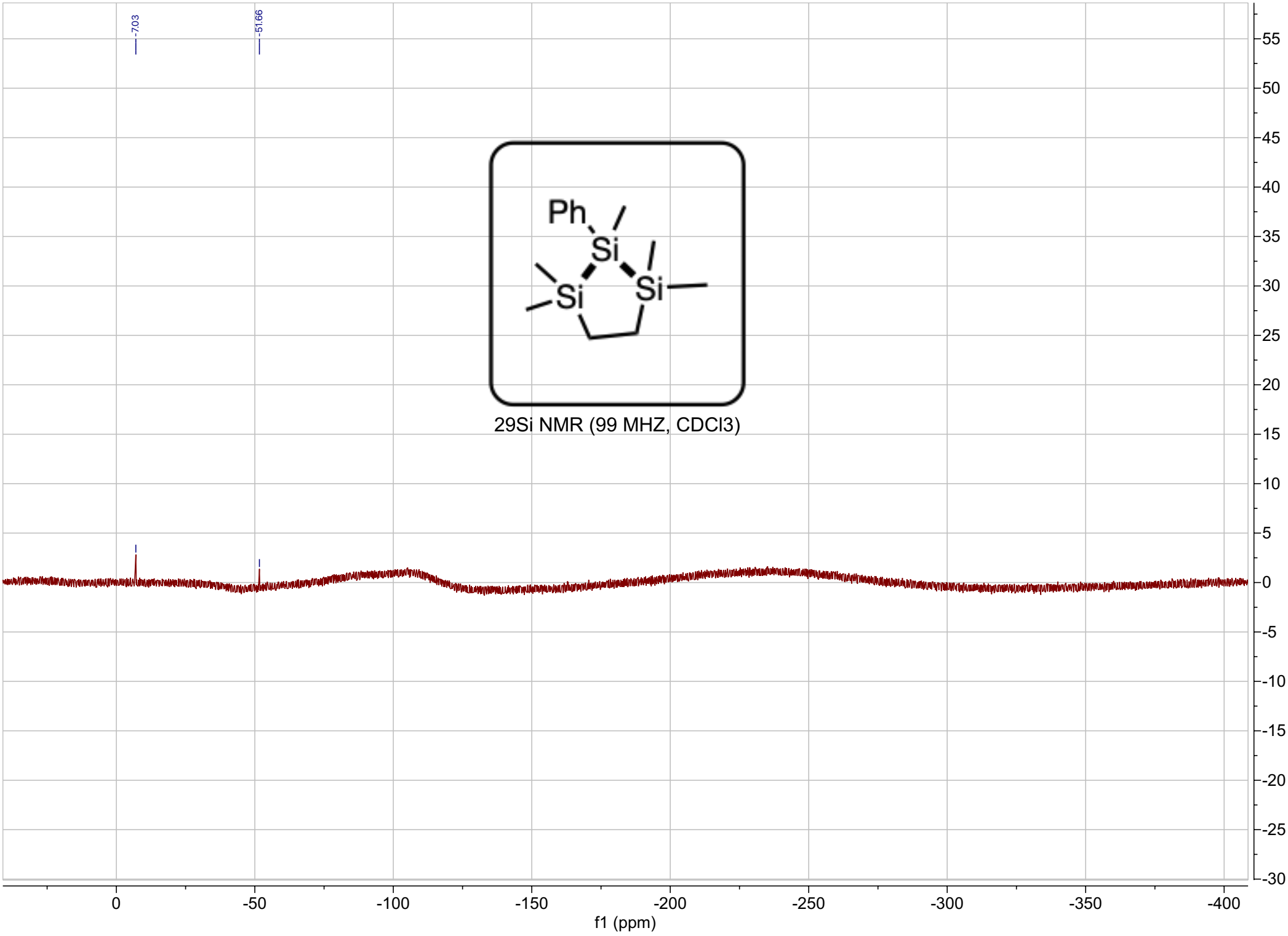

Si
Ph
Si
Si
Si
Si
Ph
1H NMR (500 MHZ, CDCl3)
3500
3000
2500
2000
1500
1000
500
0
4.00
6.09
5.98
7.22
11.11
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

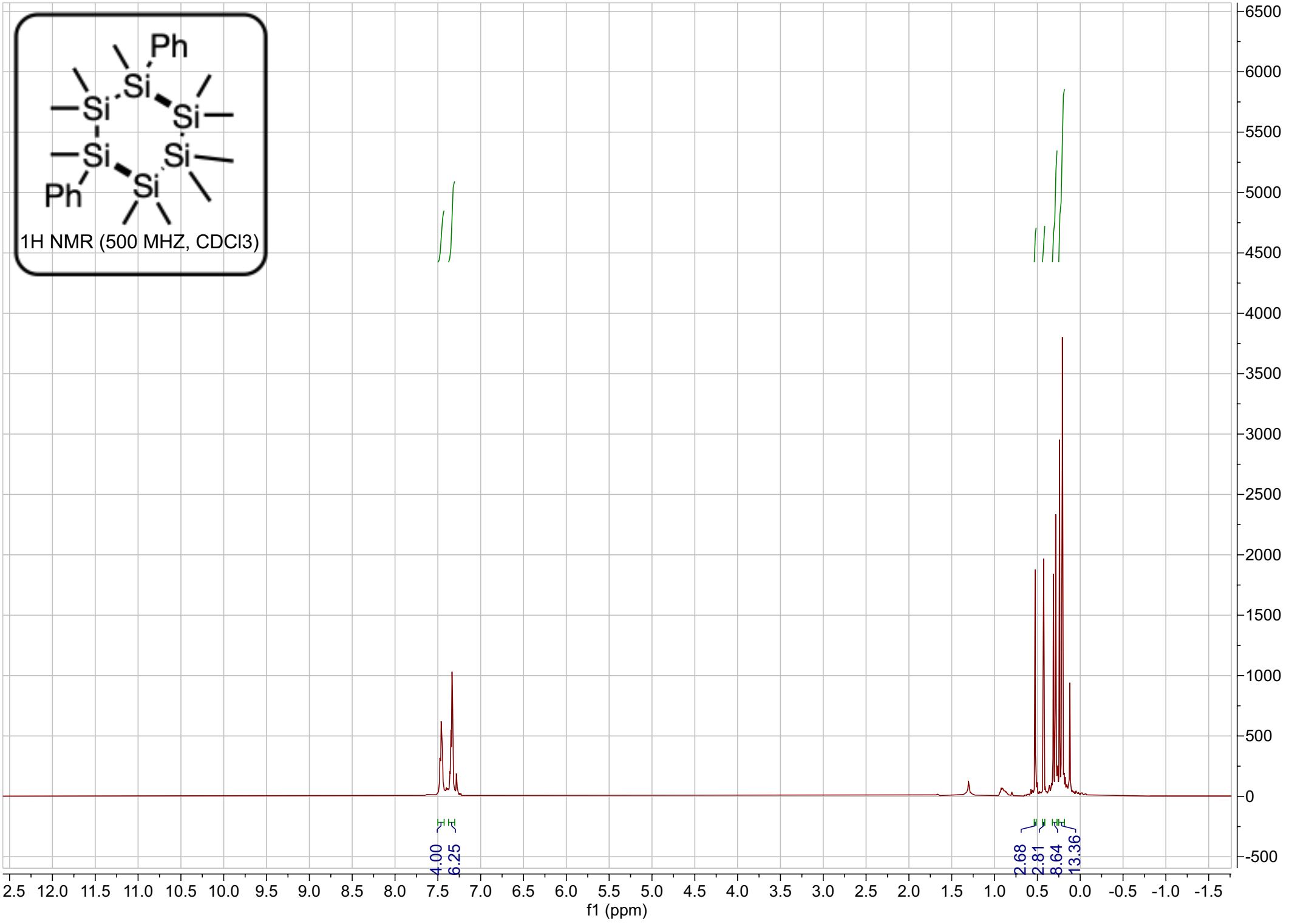

Si
Ph
Si
Si
Si
Si
Ph
13C NMR (101 MHZ, CDCl3)
137.70
137.59
135.03
135.01
127.92
127.76
-4.44
-5.15
-5.21
-5.30
-6.04
-6.39
-6.42
-6.91
-7.18
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

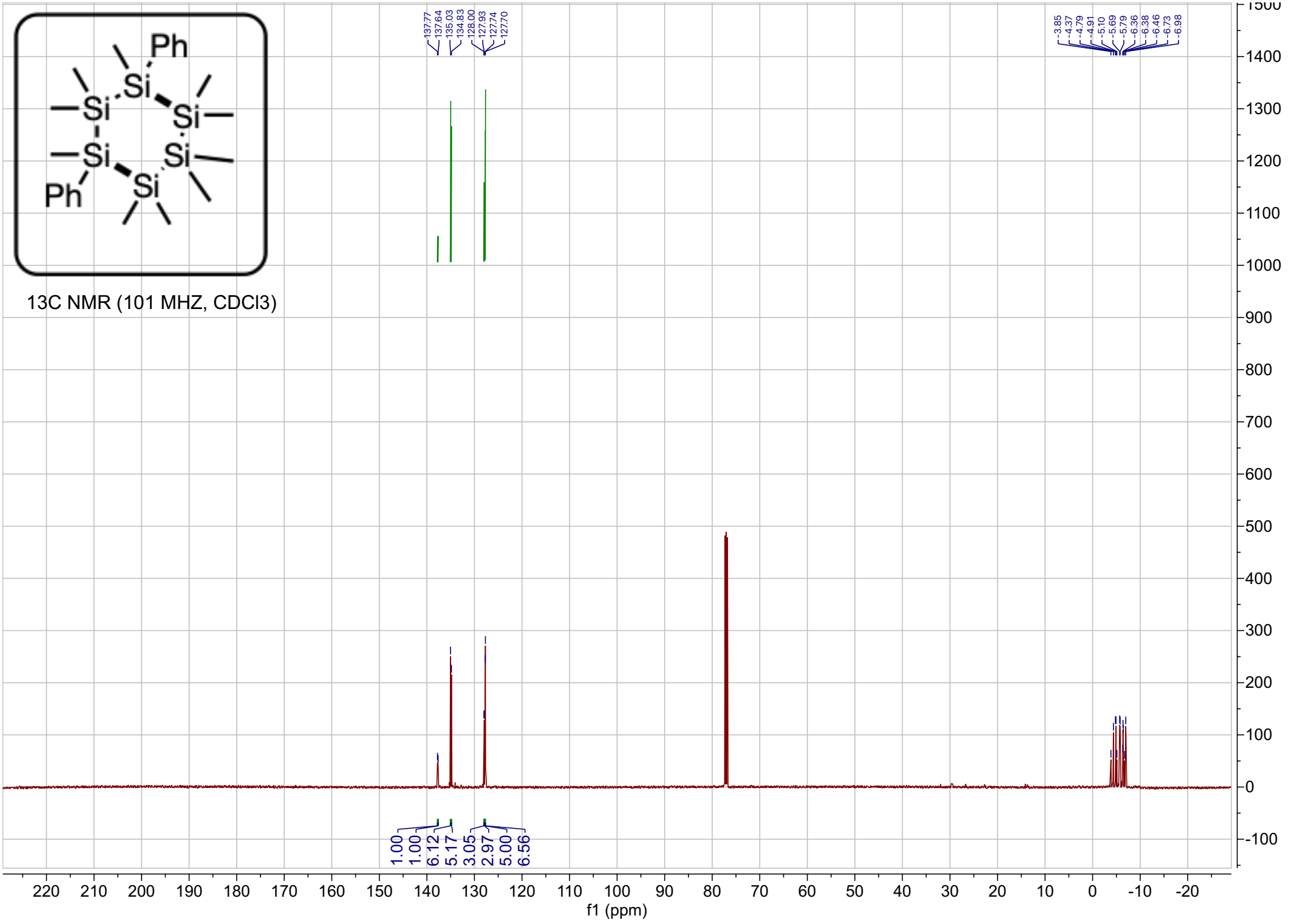

Si
Ph
Si
Si
Si
Si
Ph
29Si NMR (99 MHZ, CDCl3)
-40.15
-40.39
-40.49
-41.21
-41.69
-41.96
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)

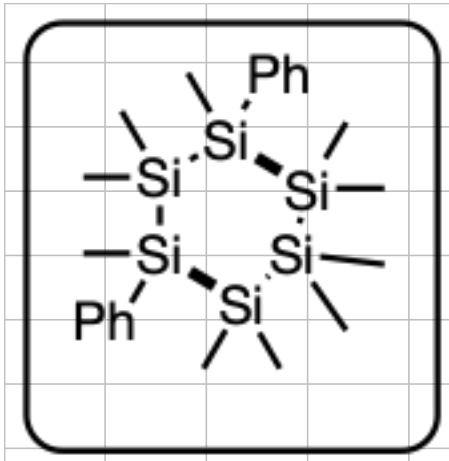

Ph
Si
Si
Si
1H NMR (500 MHZ, CDCl3)
7.44
7.43
7.34
7.33
7.31
7.30
0.89
0.88
0.88
0.87
0.87
0.86
0.85
0.46
0.17
0.12
1.90
2.73
4.00
2.73
5.47
5.58
f1 (ppm)


Ph
Si
Si
Si
13C NMR (101 MHZ, CDCl3)
136.91
134.95
127.71
127.69
12.76
-3.16
-3.46
-9.38
550
500
450
400
350
300
250
200
150
100
50
0
-50
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

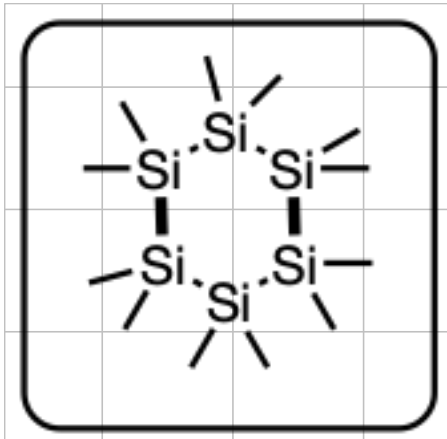

-7.03
-51.66
Ph
Si
Si
Si
29Si NMR (99 MHZ, CDCl3)
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
0
-50
-100
-150
-200
-250
-300
-350
-400
f1 (ppm)


Ph
Si
Si
Si
Si
Si
Si
Ph
1H NMR (500 MHZ, CDCl3)
4.00
6.25
2.68
2.81
8.64
13.36
f1 (ppm)

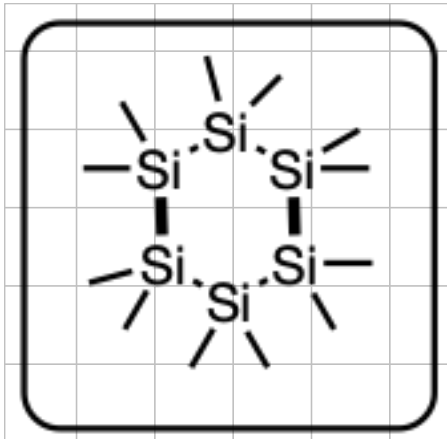

Ph
Si
Si
Si
Si
Si
Si
Ph
Si
13C NMR (101 MHZ, CDCl3)
137.77
137.64
135.03
134.83
128.00
127.93
127.74
127.70
3.85
4.37
4.79
4.91
5.10
5.69
5.79
6.36
6.38
6.46
6.73
6.98
1.00
1.00
6.12
5.17
3.05
2.97
5.00
6.56
f1 (ppm)



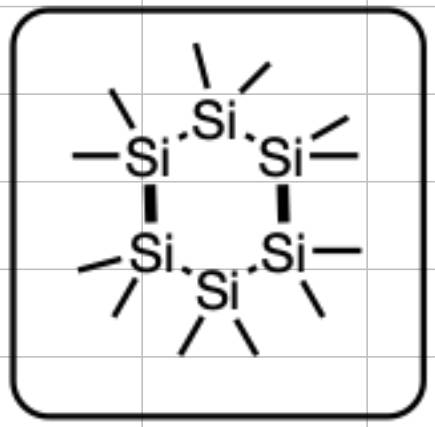
29Si NMR (99 MHZ, CDCl3)



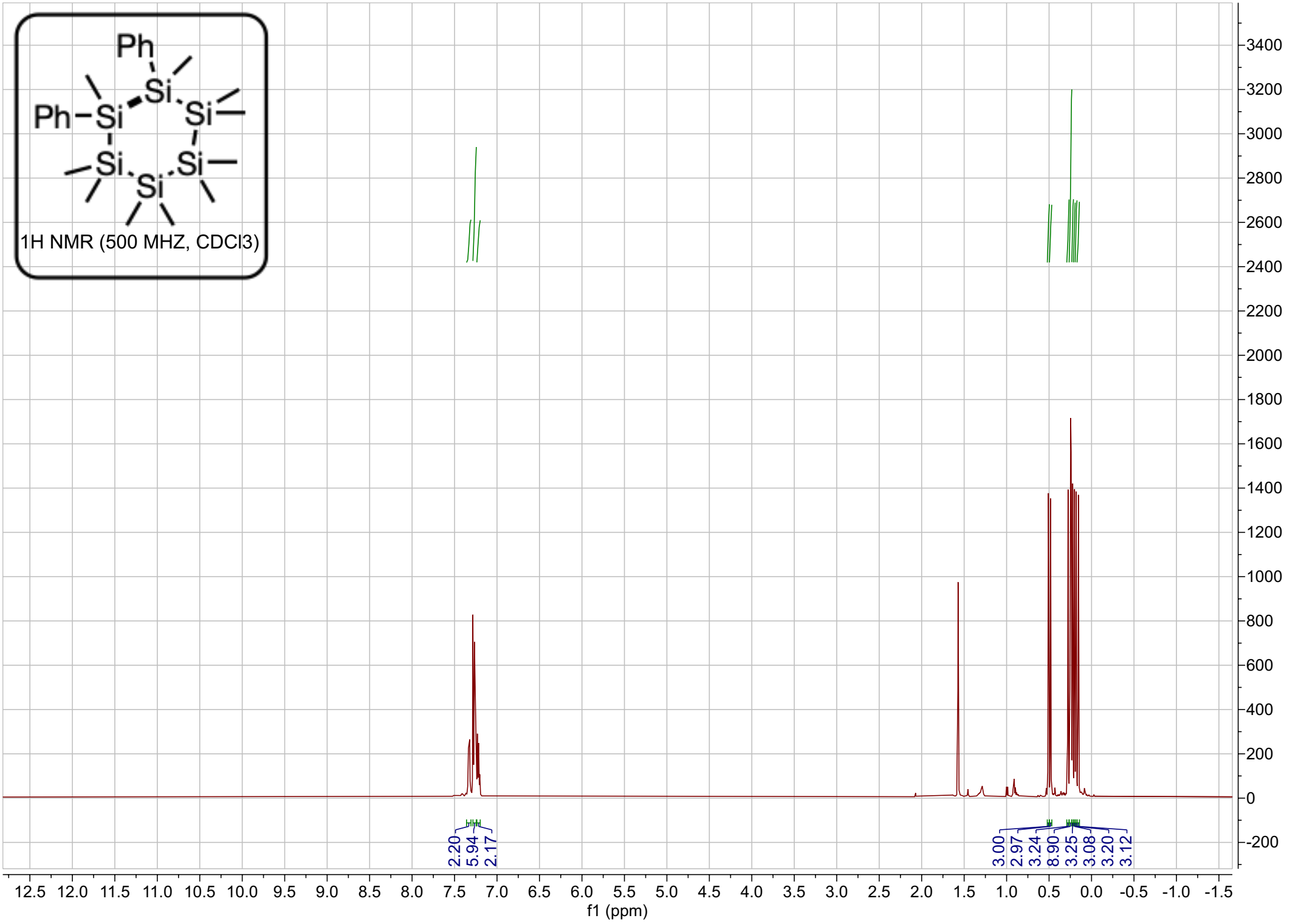
1H NMR (500 MHZ, CDCl3)

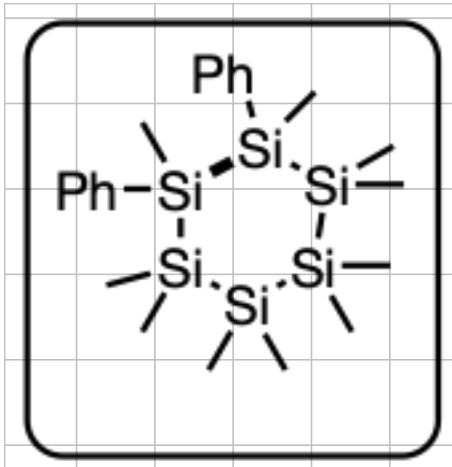


13C NMR (101 MHZ, CDCl3)



29Si NMR (99 MHZ, CDCl3)


Ph
Si
Ph
Si
Si
Si
Si
Si
1H NMR (500 MHZ, CDCl3)
2.20
5.94
2.17
3.00
2.97
3.24
8.90
3.25
3.08
3.20
3.12
f1 (ppm)



13C NMR (101 MHZ, CDCl3)

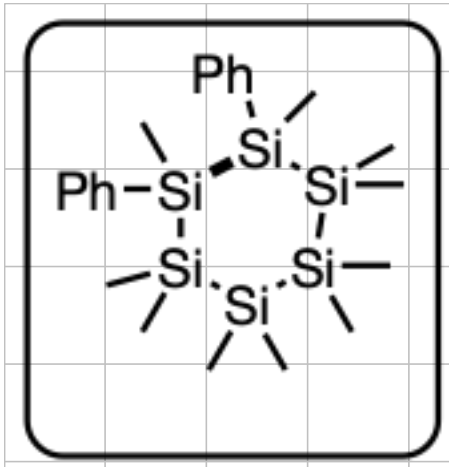
137.48
137.24
135.24
134.80
128.01
127.91
127.64
127.51

-4.36
-5.10
-5.24
-5.54
-5.56
-5.76
-5.93
-6.55
-6.78
-6.82

750 700 650 600 550 500 450 400 350 300 250 200 150 100 50 0 -50

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

f1 (ppm)



29Si NMR (99 MHZ, CDCl3)