Testing the Functional Tolerance of the Piers-Rubinsztajn Reaction: A New Strategy for Functional Silicones

John B. Grande,[a] David B. Thompson,[a,b] Ferdinand Gonzaga[a] and Michael A. Brook[a]*

Experimental Section

1. Materials and Methods

Allyltriethoxysilane, allyltrimethoxysilane, vinyltriethoxysilane, methylphenylvinylsilane, dimethylphenylsilane, pentamethyldisiloxane, 1,1,1,3,5,5,5-heptamethyltrisiloxane, (3-glycidoxy)propyltri-methoxysilane and (3-iodopropyl)trimethoxysilane were purchased from Gelest and used as received. Vinyltrimethoxysilane (98%), tetramethyl orthosilicate (98%), tetraethyl orthosilicate (98%), (3-chloropropyl)trimethoxysilane (97%), (3-chloropropyl)triethoxysilane (95%), dodecanethiol (>98%), (3-mercaptopropyl)trimethoxysilane (95%), (3-aminopropyl)trimethoxysilane (97%), diethyl acetylenedicarboxylate (95%), chromium (III) acetylacetonate, anhydrous dimethylformamide, sodium azide and tris(pentafluorophenyl)borane (95%) were purchased from Aldrich and used as received.

Commercial solvents: hexane, dichloromethane and toluene were dried over activated alumina prior to use.

^{1}H NMR, ^{13}C NMR and ^{29}Si NMR experiments were recorded at room temperature and performed on Bruker Avance 200, 500 and 600 MHz nuclear magnetic resonance spectrometers. High-resolution mass Spectrometry was performed with a Hi-Res Waters/Micromass Quattro Global Ultima (Q-TOF mass spectrometer).

2. General Procedure for exploring functional group tolerance:

Initially, survey experiments using the Piers-Rubinsztajn reaction were tested on a series of silicone compounds with different functional groups on an NMR scale. When the initial results were positive, the reaction was scaled up and compounds were isolated.

This general procedure applies to the reaction of pentamethyldisiloxane with (3-aminopropyl)triethoxysilane, (3-glycidoxypropyl)trimethoxysilane, and (3-mercaptopropyl)trimethoxysilane,.

A. Reaction of pentamethyldisiloxane with (3-aminopropyl)triethoxysilane (Figure 4A)

To a solution of (3-aminopropyl)triethoxysilane (0.150 g, 0.67 mmol) in dry hexane (5 ml), was added pentamethyldisiloxane (0.402 g, 2.7 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (10 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 7.8×10^{-4} mmol). The reaction was monitored for 1 h with no evidence of the typical exotherm associated with the Piers-Rubinsztajn reaction. The mixture was left for an additional 12 h, and resulting solution was examined via NMR. The spectrum obtained was that of starting material, indicating no reaction had taken place.

B. Reaction of pentamethyldisiloxane with (3-mercaptopropyl)trimethoxysilane 4 (Figure 4B)

To a solution of (3-mercaptopropyl)trimethoxysilane (0.250 g, 1.27 mmol) in dry hexane (10 ml), was added pentamethyldisiloxane (0.848 g, 5.72 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (20 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 1.6×10^{-3} mmol). After a 45 s induction time, slight bubbling from the solution occurred, and continued for 5 min. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1.0 g). The resulting solution was filtered, purged with nitrogen, to remove residual solvent and subjected to Kugelrohr distillation (2 mm Hg, 120 degrees centigrade). The product, obtained as a colorless oil was then examined via NMR. The obtained spectrum had shown complete loss of the methoxy moieties, with slight evidence of an SH signal still present (~ 10 %), signifying slight cleavage of the prepared thiol-silicon linkage during filtration.

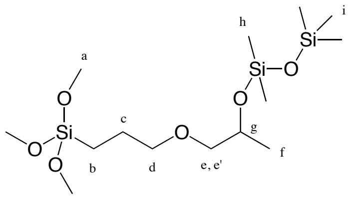
Data for protected thiol

^{1}H NMR (CDCl$_3$, 500 MHz): δ 2.55 (t, 2 H's, $O_3SiCH_2CH_2C\mathbf{H_2}SR$, *J* = 7.6 Hz), 1.67-1.74 (m, 2 H's, $O_3SiCH_2C\mathbf{H_2}CH_2SR$), 0.62-0.65 (m, 2 H's, $O_3SiC\mathbf{H_2}CH_2CH_2SR$), 0.35 (s, 6 H's, $CH_2SSi(C\mathbf{H_3})_2SiO(CH_3)_3$), 0.14 (s, 9 H's, $CH_2SSi(CH_3)_2SiO(C\mathbf{H_3})_3$), 0.11 (s, 27 H's, $H_2CSi(OSi(CH_3)_2OSi(C\mathbf{H_3})_3)_3$, 0.08 ppm (s, 18 H's, $H_2CSi(OSi(C\mathbf{H_3})_2OSi(CH_3)_3)_3$. ^{13}C NMR (CDCl$_3$ 125 MHz): δ 29.57, 27.01, 14.26, 2.67, 1.95, 1.26 ppm.

C. Reaction of pentamethyldisiloxane with (3-glycidoxypropyl)trimethoxysilane (Figure 4C)

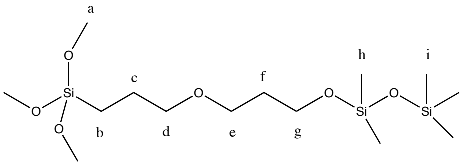
To a solution of (3-glycidoxypropyl)trimethoxysilane (0.150 g, 0.63 mmol) in dry hexane (5 ml), was added pentamethyldisiloxane (0.376 g, 2.5 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (10 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 7.8 x 10^{-4} mmol). After a 30 s induction time, the reaction flask becomes warm, signifying the onset of an exothermic reaction, lasting roughly 10 min. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1.5 g). The resulting solution was filtered and examined via NMR. The obtained spectrum was had shown evidence of complete loss epoxide, with no evidence of reaction at the methoxy moieties. The major and minor product deduced by proton and carbon NMR experiments are shown below.

Major Product: (~ 88% by NMR)



^{1}H NMR (CDCl$_3$, 500 MHz): δ 4.01-4.05 (m, 1 H, **g**, *J* = 6.0 Hz) 3.56 (s, 9 H's, **a**), 3.39-3.39-3.43 (m, 2 H's, **d**), 3.37 (dd, 1 H, **e,** *J* = 6.0, 9.6 Hz), 3.24 (dd, 1 H, **e',** *J* = 6.0, 9.6 Hz), 1.66 (m, 2 H's, **c**), 1.17 (d, 3 H's, **f**, *J* = 6 Hz), 0.65-0.68 (m, 2 H's, **b**), 0.09 (s, 9 H's, **i**), 0.07 ppm (s, 6 H's, **h**). ^{13}C NMR (CDCl$_3$ 125 MHz): δ 76.53, 73.56, 67.23, 50.64, 22.93, 20.94, 5.39, 1.92, -0.15 ppm. ^{29}Si NMR (CDCl$_3$, 99 MHz, 1 % w/v Cr(acac)$_3$): δ 7.07 (M), -13.56 (D), -42.01 ppm (T). HRMS (ES Positive mode): m/z [M+H$^+$] calculated = 385.1898, found = 385.1878.

Minor Product: (~ 12% by NMR)

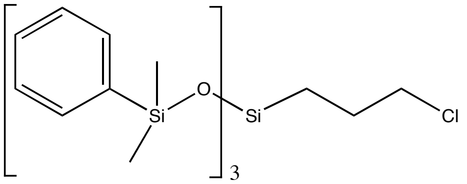


^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.72 (t, 2 H, **g**, *J* = 6.4 Hz), 3.48 (t, 2 H, **e**, *J* = 6.4 Hz), 3.56 (s, 9 H's, **a**), 3.39-3.43 (m, 2 H's, **d**), 1.79 (m, 2 H, **f**) 1.66 (m, 2 H's, **c**), 0.67 (m, 2 H's, **b**), 0.09 (s, 9 H's, **i**), 0.06 ppm (s, 6 H's, **h**). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 73.12, 67.56, 59.33, 50.64, 33.61, 22.93, 5.39, 1.92, -0.17 ppm. ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ 7.31 (M), -13.02 (D), -42.01 ppm (T).

3. Scaled Experimental Procedures (Table 1)

The compounds synthesized below have been scaled as preliminary small-scale studies have shown that the Piers-Rubinsztajn reaction has been functionally tolerant to chloro, iodo, vinyl and allyl containing species.

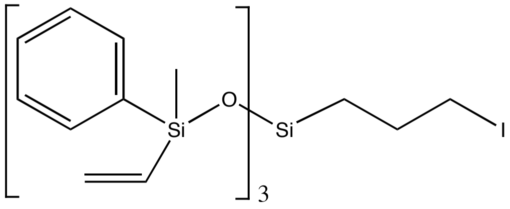
Synthesis of (3-chloropropyl)tris(dimethylphenylsilyloxy)silane. (Table 1, Entry **1**):



To a solution of chloropropyltriethoxysilane (0.100 g, 0.5 mmol) in dry hexane (4 ml), was added dimethylphenylsilane (0.308 g, 2.3 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (10 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 7.8×10^{-4} mmol). After a 2 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1 g). The resulting solution was filtered and concentrated on a rotary evaporator. The remaining solvent and excess reagents were removed *in vacuo,* affording pure chloropropyltris(dimethylphenylsilyl)silane (0.260 g, 91 % yield).

^{1}H NMR ($CDCl_3$, 200 MHz): δ 7.38-7.54 (m, 6 H's, phenyl) 7.27-7.33 (m, 9 H's, phenyl), 3.36 (t, 2 H's, $O_2SiCH_2CH_2C\mathbf{H_2}Cl$, *J* = 6.8 Hz), 1.54-1.67 (m, 2 H's, $O_2SiCH_2C\mathbf{H_2}CH_2Cl$), 0.31-0.57 (m, 2 H's, $O_2SiC\mathbf{H_2}CH_2CH_2Cl$), 0.31 (s, 18 H's, $OSi(C\mathbf{H_3})_2(C_6H_5)$). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 139.3, 133.2, 129.5, 127.9, 47.7, 26.9, 12.1, 0.64 ppm. ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ -1.98 (M), -66.90 ppm (T). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 576.2009, found = 576.2003.

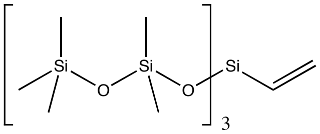
Synthesis of (3-iodopropyl)tris(methylphenylvinylsilyloxy)silane (Table 1, Entry **2**):



To a solution of iodopropyltrimethoxysilane (0.500 g, 1.7 mmol) in dry hexane (10 ml), was added methylphenylvinylsilane (1.14 g, 7.75 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (20 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 1.6 x 10^{-3} mmol). After a 1 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1.5 g). The resulting solution was filtered and concentrated under reduced pressure affording pure iodopropyltris(methylphenylvinylsilyloxy)silane (1.1 g , 93% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.49-7.51 (m, 6 H's, phenyl), 7.36-7.39 (m, 3 H's, phenyl), 7.29-7.32 (m, 6 H's, phenyl), 6.15-6.23 (m, 3 H, $R_3SiC\mathbf{H}CHH$), 6.02-6.06 (m, 3 H's, $R_3SiCHC\mathbf{H}H$), 5.75-5.80 (m, 3 H's, $R_3SiCHCH\mathbf{H}$), 3.03 (t, 2 H's, $O_3SiCH_2CH_2C\mathbf{H_2}I$, *J* = 7.0 Hz), 1.71-1.77 (m, 2 H's, $O_3SiCH_2C\mathbf{H_2}CH_2I$), 0.57-0.60 (m, 2 H's, $O_3SiC\mathbf{H_2}CH_2CH_2I$), 0.371 ppm (t, 9 H's, $OSi(C\mathbf{H_3})(C_6H_5)(CHCH_2)$, *J* = 3.5 Hz). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 137.36, 136.89, 134.20, 133.74, 129.69, 127.84, 27.93, 16.16, 10.94, -1.18 ppm. ^{29}Si NMR ($CDCl_3$, 99MHz, 1 % w/v $Cr(acac)_3$): δ -13.38 (M), -67.49 ppm (T). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 704.1365, found = 704.1349.

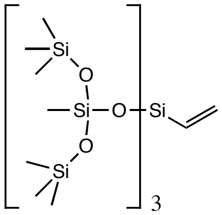
Synthesis of tris(pentamethyldisiloxy)vinylsilane (Table 1: Entry **3**):



To a solution of vinyltriethoxysilane (2 g, 10.5 mmol) in dry hexane (15 ml), was added pentamethyldisiloxane (7.01 g, 47 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (50 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 3.9×10^{-3} mmol). After a 5 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (2.5 g). The resulting solution was filtered and concentrated under reduced pressure. The remaining solvent and excess reagents were removed *in vacuo,* affording pure tris(pentamethyldisiloxy)vinylsilane (5.06 g, 9.3 mmol, 89% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.86-6.0 (m, 3 H's, $O_3SiC\mathbf{H}C\mathbf{H_2}$), 0.09 (s, 27 H's ($\mathbf{H_3}C)_3SiO$), 0.07 ppm (s, 18 H's ($C\mathbf{H_3})_2SiO_2$). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 133.97 (CH vinyl), 133.53 (CH_2 vinyl), 1.95 ($Si(\mathbf{C}H_3)_3$), 1.24 ppm ($O_2Si(\mathbf{C}H_3)_2$). ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ 7.0 (M), -21.6 (D), -82.2 ppm (D). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 562.2180, found = 562.2153.

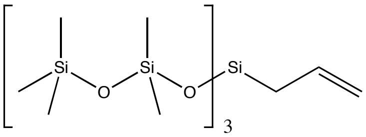
Synthesis of tris(1,1,1,3,5,5,5-heptamethyltrisiloxy)vinylsilane (Table 1: Entry **4**):



To a solution of vinyltrimethoxysilane (0.513 g, 3.5 mmol) in dry hexane (10 ml) was added 1,1,1,3,5,5,5-heptamethyltrisiloxane (3.85 g, 17.3 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (40 μl of a prepared solution containing 40 mg dissolved in 1 ml of toluene, 3.1×10^{-3} mmol). After a 3 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1 g). The resulting solution was filtered and concentrated under reduced pressure. The remaining solvent and excess reagents were removed *in vacuo,* affording pure tris(1,1,1,3,5,5,5-heptamethyltrisiloxy)vinylsilane (2.40 g, 89.5% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.89-5.98 (m, 3 H's, $O_3SiC\mathbf{H}C\mathbf{H_2}$), 0.09 (s, 54 H's ($\mathbf{H_3}C)_3SiO$), 0.05 ppm (s, 9 H's ($C\mathbf{H_3})SiO_3$); ^{13}C NMR: δ ($CDCl_3$ 125 MHz) 134.75 (CH vinyl), 133.74 (CH_2 vinyl), 2.48 ($Si(\mathbf{C}H_3)_3$), -1.33 ppm ($O_3Si\mathbf{C}H_3$); ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ 7.16 (M), -66.43 (D), -84.02 ppm (D). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 784.2743, found = 784.2771.

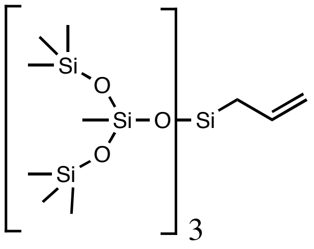
<u>Synthesis of allyltris(pentamethyldisiloxy)silane (Table 1: Entry **5**):</u>



To a solution of allyltriethoxysilane (0.500 g, 2.4 mmol) in dichloromethane (10 ml), was added pentamethyldisiloxane (1.63 g, 11 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (20 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 1.6 x 10^{-3} mmol). After a 3 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1 g). The resulting solution was filtered and concentrated on a rotary evaporator. The remaining solvent and excess reagents were removed *in vacuo,* affording pure allyltris(pentamethyldisiloxy)silane (1.18 g, 87% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.74-5.83 (m, 1 H, $O_3SiCH_2C\mathbf{H}CH_2$), 4.93 (dd, 1 H, $O_3SiCH_2CHC\mathbf{H}H$, *J* = 2.0, 17.0 Hz), 4.87 (dd, 1 H, $O_3SiCH_2CHCH\mathbf{H}$, *J* = 2.0, 10.0 Hz), 1.55 (d, 2 H's $O_3SiC\mathbf{H_2}CHCH_2$, *J* = 7.5 Hz), 0.09 (s, 27 H's, $((C\mathbf{H_3})_3SiO)$, 0.06 ppm (s, 18 H's, $(C\mathbf{H_3})_2SiO)$. ^{13}C NMR ($CDCl_3$ 125 MHz): δ 133.78 ($O_3SiCH_2\mathbf{C}HCH_2$), 114.31 ($O_3SiCH_2CH\mathbf{C}H_2$), 22.3 ($O_3Si\mathbf{C}H_2CHCH_2$), 1.97 ($(\mathbf{C}H_3)_3SiO$), 1.24 ppm ($(\mathbf{C}H_3)_2SiO_2$). ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ 6.95 (M), -21.96 (D), -74.08 ppm (T). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 576.2336, found = 576.2369

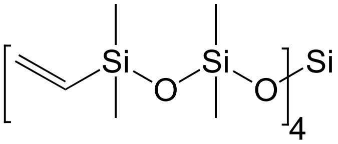
<u>Synthesis of allyltris(1,1,1,3,5,5,5-heptamethyltrisiloxy)silane (Table 1: Entry **6**):</u>



To a solution of allyltriethoxysilane (0.500 g, 2.4 mmol) in dry hexane (10 ml), was added 1,1,1,3,5,5,5-heptamethyltrisiloxane (2.72 g, 12 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (65 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 5.1 x 10^{-3} mmol). After a 5 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1 g). The resulting solution was filtered and concentrated under reduced pressure. The remaining solvent and excess reagents were removed *in vacuo,* affording pure allyltris(1,1,1,3,5,5,5-heptamethyltrisiloxy)silane (1.78 g, 94% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.78-5.86 (m, 1 H, $O_3SiCH_2C\mathbf{H}CH_2$), 4.95 (dd, 1 H, $O_3SiCH_2CHC\mathbf{H}H$, *J* = 1.8, 17.0 Hz), 4.87 (dd, 1 H, $O_3SiCH_2CHCH\mathbf{H}$ *J* = 1.8, 10.5 Hz), 1.57 (d, 2 H's, $O_3SiC\mathbf{H_2}CHCH_2$ *J* = 7.5 Hz), 0.10 (s, 54 H's, $(C\mathbf{H_3})_3SiO)$)), 0.04 (s, 9 H's, $C\mathbf{H_3}SiO_3$). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 133.63 ($O_3SiCH_2\mathbf{C}HCH_2$), 114.48 ($O_3SiCH_2CH\mathbf{C}H_2$), 22.04 ($O_3Si\mathbf{C}H_2CHCH_2$), 1.91 ($\mathbf{C}H_3)_3SiO$), -1.94 ppm ($\mathbf{C}H_3SiO_3$). ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ 7.14 (M), -66.57 (D), -75.67 ppm (D). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 798.2900, found = 798.2928.

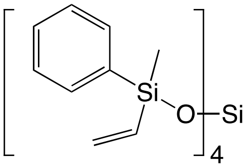
<u>Synthesis of tetrakis(vinyltetramethyldisiloxy)silane (Table 1: Entry **7**)</u>**:**



To a solution of tetraethyl orthosilicate (0.200 g, 0.96 mmol) in dry hexane (5 ml), was added vinyltetramethyldisiloxane (0.85 g, 5.28 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (10 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 7.81 x 10^{-4} mmol). After a 2 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1 g). The resulting solution was filtered and concentrated under reduced pressure. The remaining solvent and excess reagents were removed *in vacuo,* affording pure tetrakis(vinyltetramethyldisiloxy)silane (0.520 g, 75% yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 6.13 (dd, 4 H's, $R_3SiC\mathbf{H}CHH$, *J* = 15.0, 20.5 Hz), 5.93 (dd, 4 H's, $R_3SiCHCH\mathbf{H}$, *J* = 4.0, 15.0 Hz), 5.74 (dd, 4 H's, $R_3SiCHC\mathbf{H}H$, *J* = 4.0, 20.5 Hz), 0.166 (s, 24H's $O(\mathbf{H_3}C)_2Si(CHCH_2)$), 0.09 ppm (s, 24 H's $(C\mathbf{H_3})_2SiO_2$)). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 139.47 (CH vinyl), 131.82 (CH_2 vinyl), 1.17 ($SiCH_3$), 0.45 ppm ($SiCH_3$). ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ -4.34 (M), -20.95 (D), -110.18 ppm (D). HRMS (ES Positive mode): m/z [$M+NH_4^+$] calculated = 746.2555, found = 746.2564.

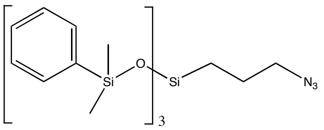
Synthesis of tetrakis(methylphenylvinylsiloxy)silane (Table 1: Entry **8**):



To a solution of tetramethyl orthosilicate (0.500 g, 3.3 mmol) in dry hexane (15 ml), was added methylphenylvinylsilane (2.92 g, 19.7 mmol). The mixture was stirred at room temperature for 5 min before the addition of tris(pentafluorophenyl)borane (25 μl of a solution containing 40 mg dissolved in 1 ml of toluene, 1.95 x 10^{-3} mmol). After a 2.5 min induction time, rapid evolution of gas and heat from the solution occurred. The mixture was allowed to cool to room temperature before the addition of neutral alumina (1.5 g). The resulting solution was filtered and concentrated on a rotary evaporator. The remaining solvent and excess reagents were removed *in vacuo,* affording pure tetrakis(methylphenylvinylsiloxy)silane (1.98 g, 2.9 mmol, 89 % yield).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.49-7.51 (d, 8 H's, *J* = 7.5 Hz, phenyl), 7.36-7.39 (m, 4 H's, phenyl), 7.26-7.29 (m, 8 H's, phenyl), 6.16 (dd, 4 H's, $R_3SiC\mathbf{H}CHH$, *J* = 14.5, 20.0 Hz), 6.0 (dd, 4 H's, $R_3SiCHCH\mathbf{H}$, *J* = 3.5, 14.5 Hz), 5.76 (dd, 4 H's, $R_3SiCHC\mathbf{H}H$, *J* = 3.5, 20 Hz), 0.34 ppm (s, 12 H's, $OSi(C\mathbf{H_3})(C_6H_5)(CHCH_2)$). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 137.34, 136.86, 133.97, 133.80, 129.48, 127.67, -1.37 ppm. ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v Cr(acac)$_3$): δ -12.84 (M), -106.12 ppm (Q). HRMS (ES Positive mode): m/z [M+NH$_4^+$] calculated = 698.2430, found = 698.2447.

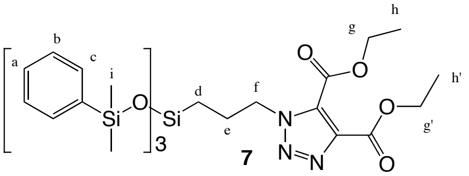
Synthesis of (3-azidopropyl)tris(dimethylphenylsiloxy)silane **6**:



To a 10 ml round bottom flask equipped with a magnetic stir bar was added a solution of (3-chloropropyl)tris(dimethylphenylsiloxy)silane **5** (0.500 g, 0.89 mmol) in anhydrous DMF (2.5 ml). To the solution was added sodium azide (0.116 g, 1.78 mmol). The mixture was then allowed to stir overnight at 90 °C. To the mixture was added to 40 ml of water and extracted with 25 ml of hexane. The aqueous layer was washed once more to maximize product recovery. The organic layers were combined and dried over magnesium sulfate (10 g). The resulting solution was filtered and concentrated under reduced pressure affording pure pale yellow (3-azidopropyl)tris(dimethylphenylsiloxy)-silane **6** (0.489 g, 97% yield).

^{1}H NMR (CDCl$_3$, 200 MHz): δ 7.48-7.53 (m, 6 H's, phenyl) 7.29-7.38 (m, 9 H's, , phenyl), 3.04 (t, 2 H's, $O_3SiCH_2CH_2C\mathbf{H_2}N_3$, *J* = 7.1 Hz), 1.45-1.53 (m, 2 H's, $O_3SiCH_2C\mathbf{H_2}CH_2N_3$), 0.43-0.52 (m, 2 H's, $O_3SiC\mathbf{H_2}CH_2CH_2N_3$), 0.30 (s, 18 H's, $OSi(C\mathbf{H_3})_2(C_6H_5)$). ^{13}C NMR (CDCl$_3$ 125 MHz): δ 139.3, 133.1, 129.5, 127.9, 53.9, 23.1, 11.6, 0.62 ppm. ^{29}Si NMR (CDCl$_3$, 99 MHz, 1 % w/v Cr(acac)$_3$): δ -1.97 (M), -66.94 ppm (T). HRMS (ES Positive mode): m/z [M+NH$_4^+$] calculated = 583.2412, found = 583.2407.

Synthesis of **7:**

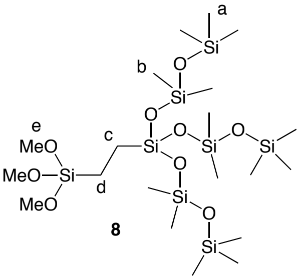


7

To a 5 ml round bottom flask equipped with a magnetic stir bar and previously prepared (3-azidopropyl)tris(dimethylphenylsiloxy)silane (0.250 g, 0.44 mmol), was added diethyl acetylenedicarboxylate (0.13 g, 0.88 mmol). The mixture was then stirred at room temperature overnight. Once completed, excess staring materials were removed under reduced pressure readily affording pure **7** (0.324 g, 99.6% yield)

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.46-7.48 (m, 6 H's, **c**) 7.34-7.37 (m, 3 H's, **a**), 7.29-7.31 (m, 6 H's, **b**), 4.43 (q, 2 H's, **g**, *J* = 7.0 Hz), 4.39 (t, 2 H's, **f**, *J* = 7.5 Hz), 4.35 (q, 2 H's, **g'**, *J* = 7.0 Hz), 1.77 (m, 2 H's, **c**), 1.42 (t, 3 H's, **h**, *J* = 7.0 Hz), 1.33 (t, 3 H's, **h'**, *J* = 7.0 Hz), 0.41-0.44 (m, 2 H's, **d**), 0.28 ppm (s, 18 H's, **i**). ^{13}C NMR ($CDCl_3$ 125 MHz): δ 160.42, 158.58, 140.22, 139.07, 133.05, 129.48, 127.81, 62.78, 61.87, 52.87, 24.57, 14.29, 13.95, 11.41, 0.50 ppm. ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ -1.68 (M), -67.75 ppm (T). HRMS (ES Positive mode): m/z [M $^+$] calculated = 736.2726, found = 736.2702.

Synthesis of **8**:



8

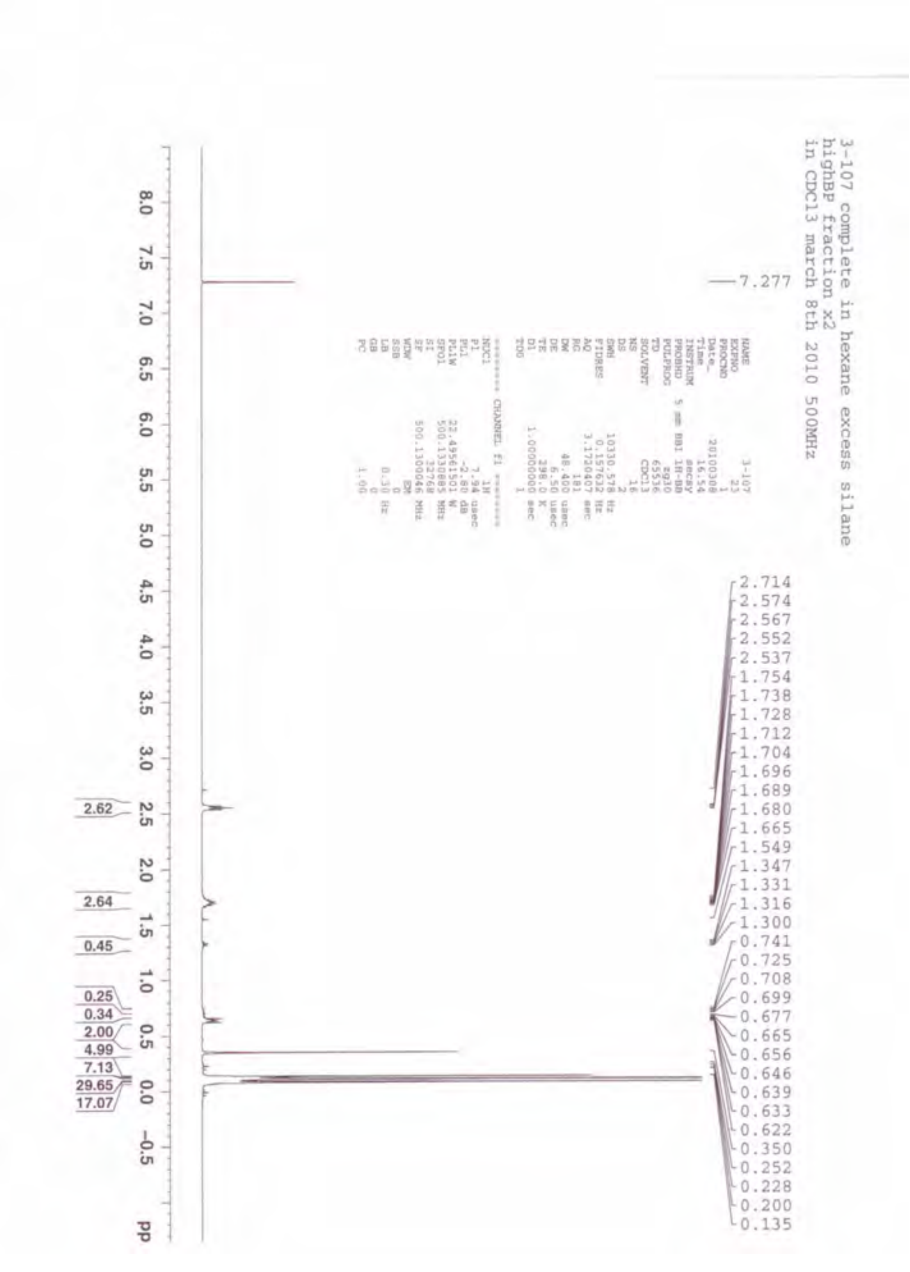
In a round bottom flask equipped with a stir bar and water jacket condenser with drying tube (Drierite desiccant) was dissolved tris(pentamethyldisiloxy)vinylsilane (0.250 g, 0.46 mmol) in toluene (5 ml). Trimethoxysilane (0.073 g, 0.59 mmol) was then added, followed by Karstedt's platinum complex (15 μl; 2% solution in xylenes). The reaction flask was then immersed in an oil bath at 80 °C. The reaction was allowed to proceed for 18 h, at which time activated charcoal was added (0.25 g), and the resulting solution was stirred for an additional 2 h. The crude reaction mixture was filtered over celite, and residual solvent and excess hydrosilane was removed *in vacuo*, affording **8**. **(**0.214 g, 70% yield)

^{1}H NMR ($CDCl_3$, 600 MHz): δ 3.56 (s, 9 H's, **e**), 0.639 (m, 2 H's, **c**), 0.55 (m, 2 H's, **d**), 0.09 (s, 27 H's, **f**), 0.60 ppm (s, 18 H's, **b**). ^{13}C NMR ($CDCl_3$, 150 MHz): δ 50.70, 5.39, 1.97, 1.30, 0.95 ppm. ^{29}Si NMR ($CDCl_3$, 99 MHz, 1 % w/v $Cr(acac)_3$): δ -6.90 (M), -22.12 (D), -41.89 (T), -69.16 ppm (T)

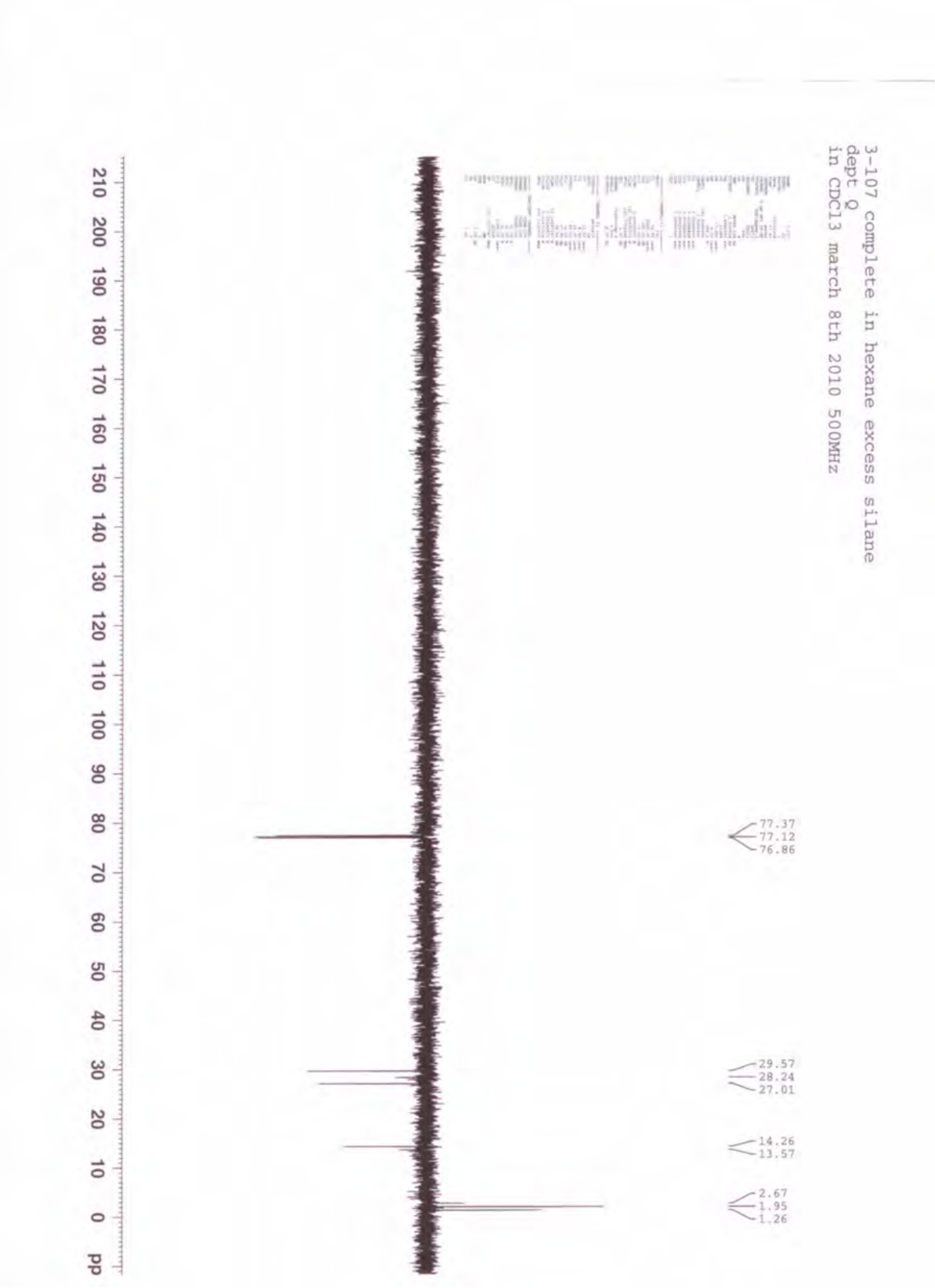
3. Spectroscopic Data from NMR experiments

Mercaptopropyltrimethoxysilane + pentamethyldisiloxane

^{1}H NMR

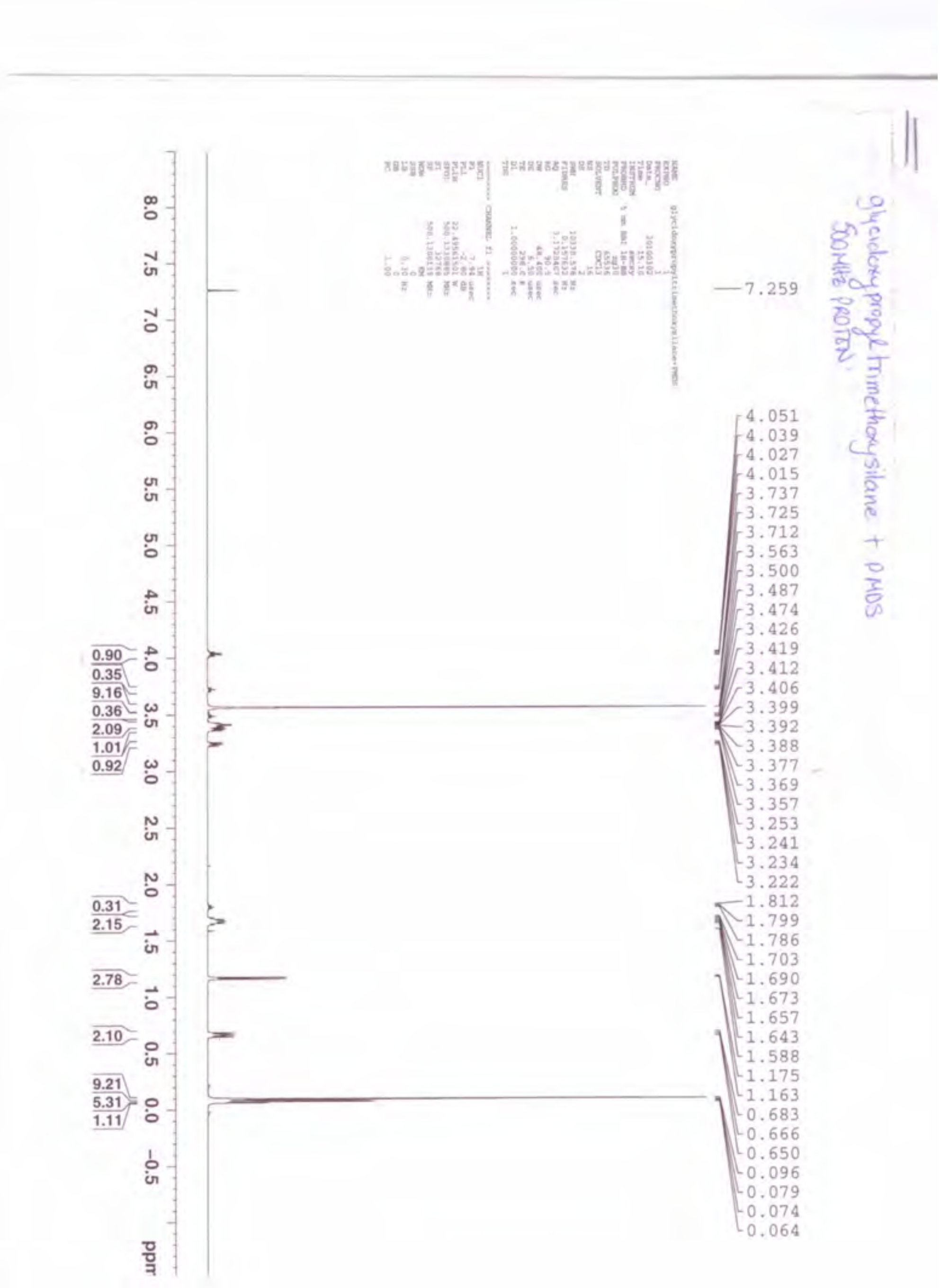


^{13}C NMR

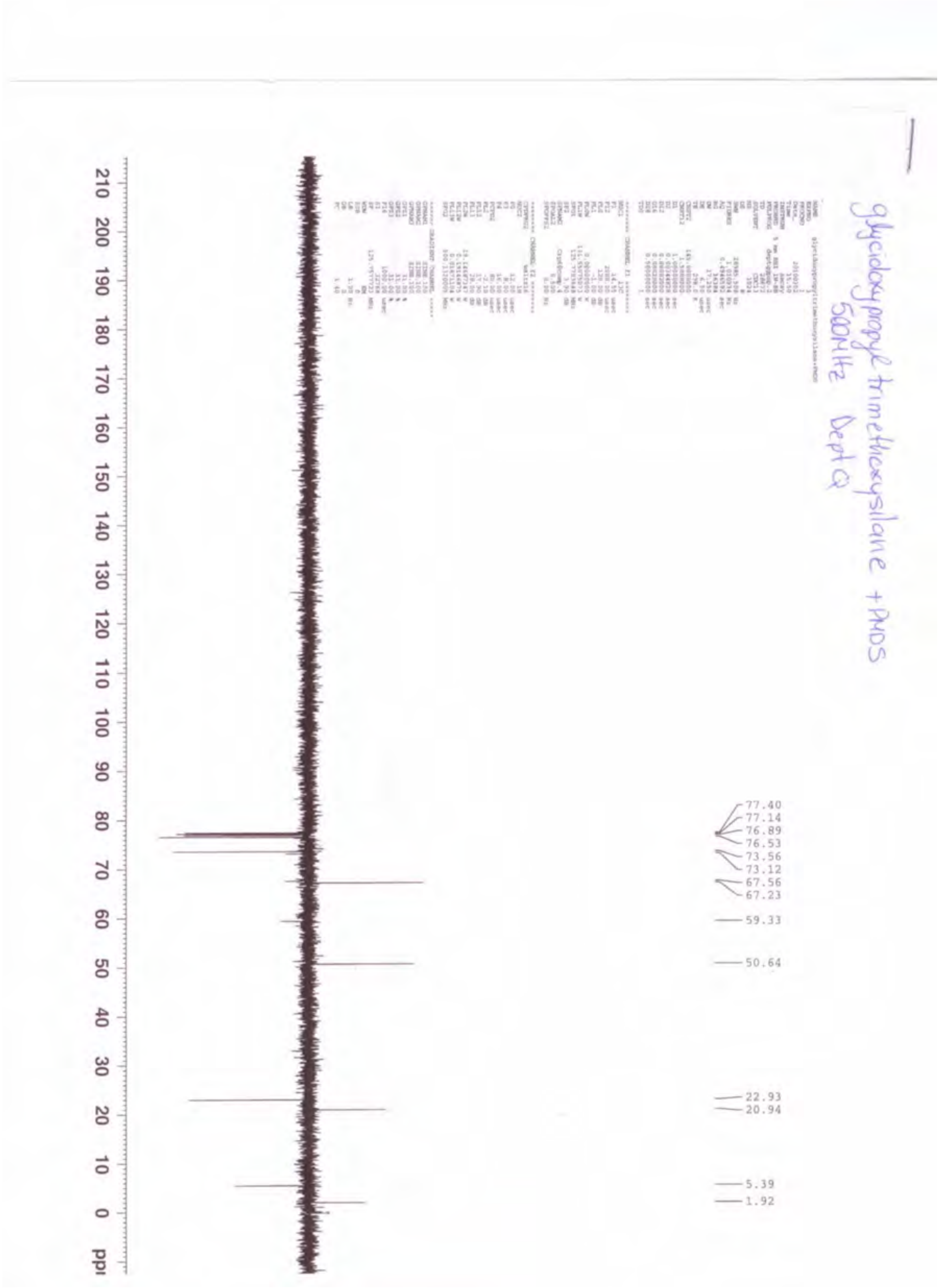

3-107 complete in hexane excess silane
dept Q
in CDCl3 march 8th 2010 500MHz
77.37
77.12
76.86
29.57
28.24
27.01
14.26
13.57
2.67
1.95
1.26
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
pp

Pentamethyldisiloxane + (3-glycidoxypropyl)trimethoxysilane

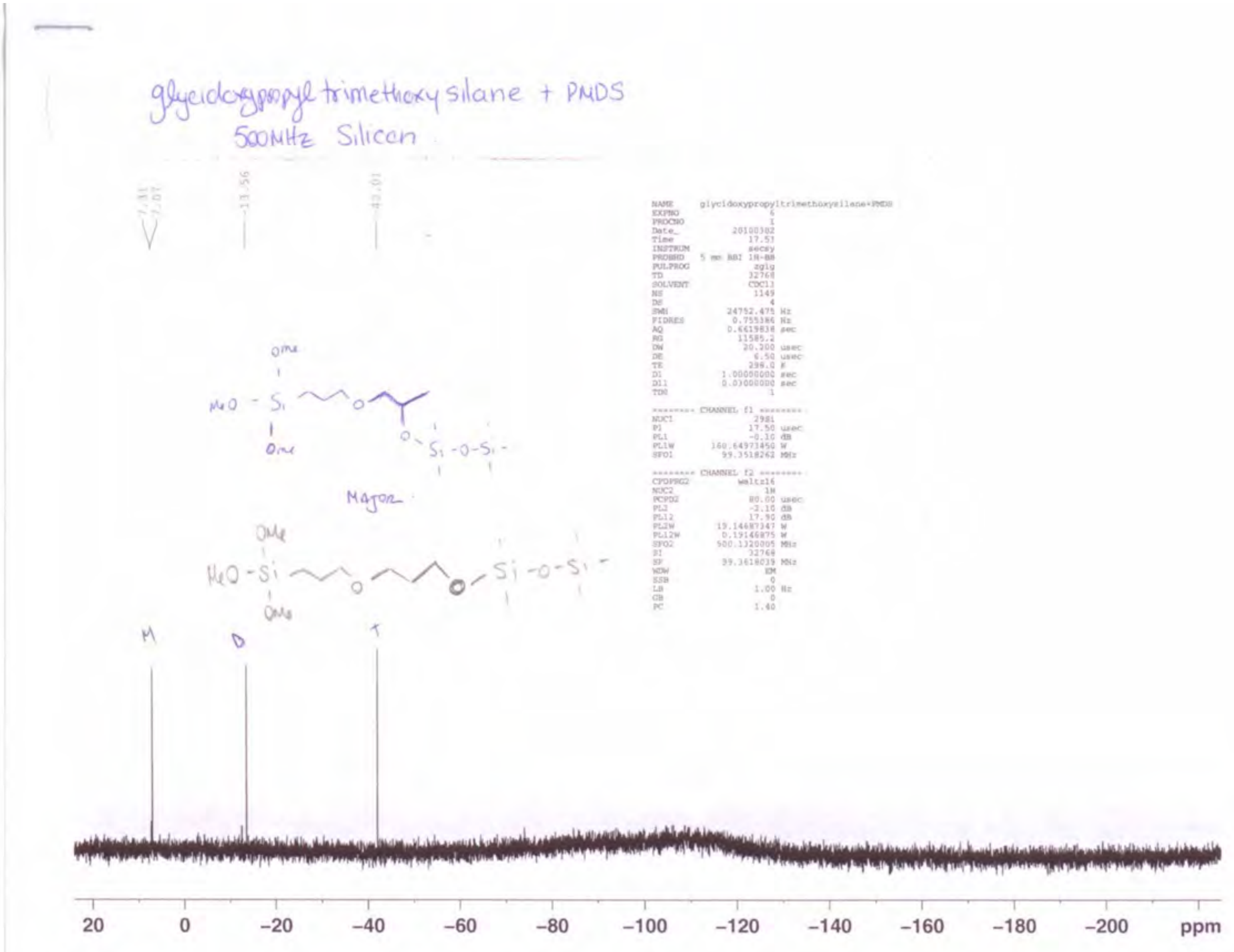
^{1}H NMR



^{13}C NMR


glycidoxypropyl trimethoxysilane +PMDS
500MHz DeptQ
77.40
77.14
76.89
76.53
73.56
73.12
67.56
67.23
59.33
50.64
22.93
20.94
5.39
1.92
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

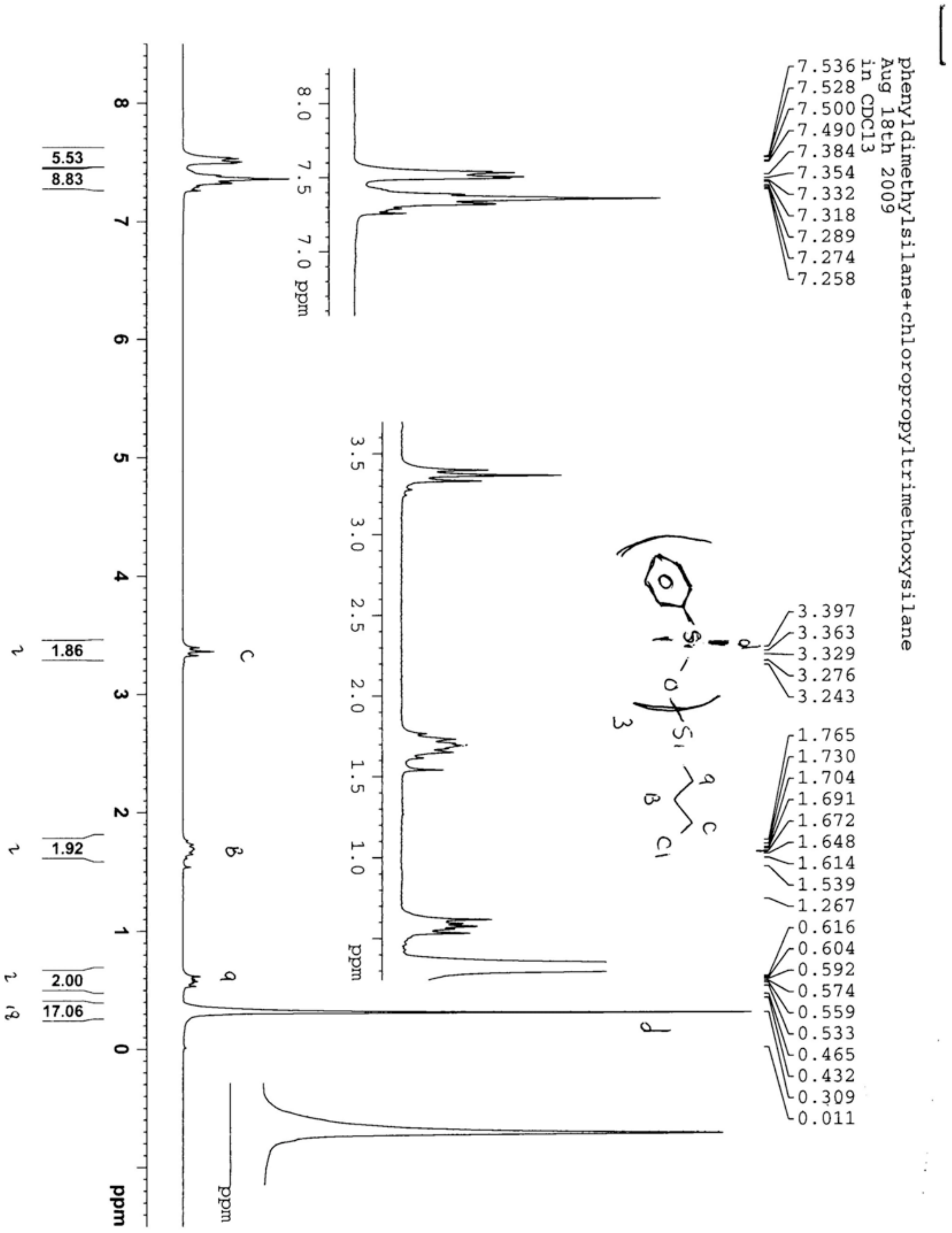
^{29}Si NMR

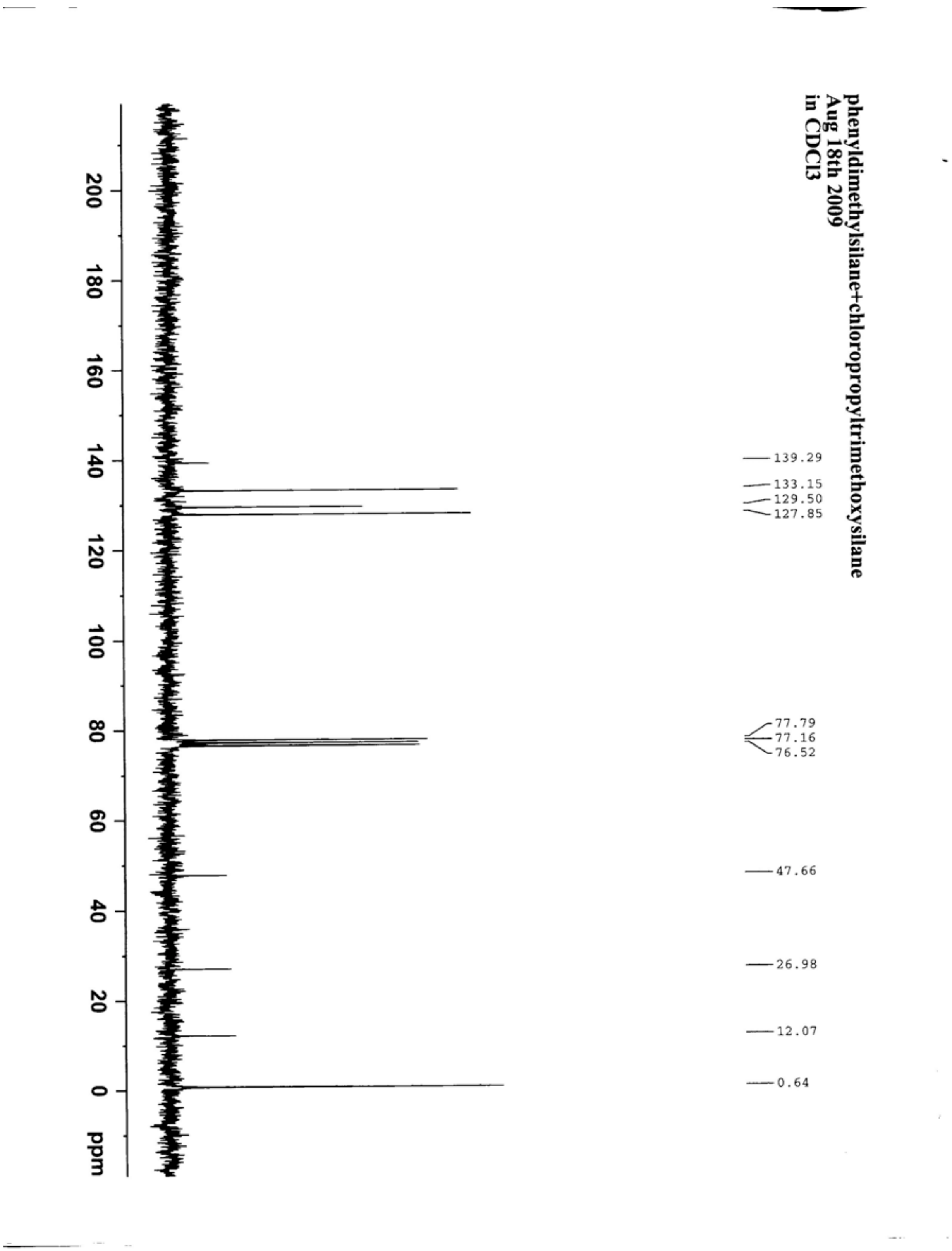


```
NAME       glycidoxypropyltrimethoxysilane+PMDS
EXPNO                  6
PROCNO                 1
Date_           20100302
Time               17.53
INSTRUM            secsy
PROBHD     5 mm BBI 1H-BB
PULPROG             zgig
TD                 32768
SOLVENT            CDCl3
NS                  1149
DS                     4
SWH            24752.475 Hz
FIDRES          0.755386 Hz
AQ             0.6619838 sec
RG               11585.2
DW                20.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                29Si
P1                 17.50 usec
PL1                -0.10 dB
PL1W        160.64973450 W
SFO1         99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.10 dB
PL12               17.90 dB
PL2W         15.14687347 W
PL12W         0.19146875 W
SFO2         500.1320005 MHz
SI                 32768
SF            99.3618039 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

4. Spectroscopic Data from Table 1

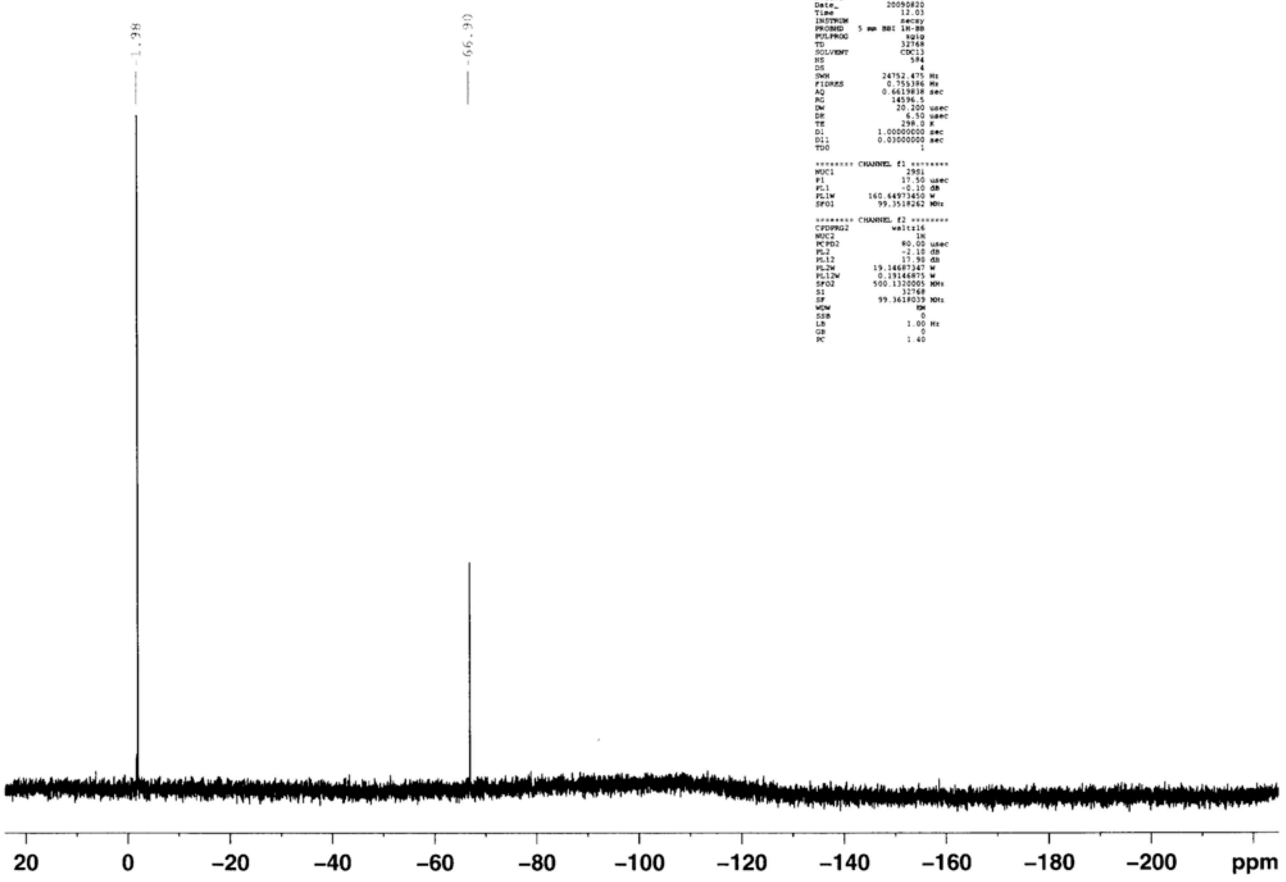
Table 1 Entry #1:




phenyldimethylsilane+chloropropyltrimethoxysilane
Aug 18th 2009
in CDCl3
139.29
133.15
129.50
127.85
77.79
77.16
76.52
47.66
26.98
12.07
0.64
200
180
160
140
120
100
80
60
40
20
0
ppm

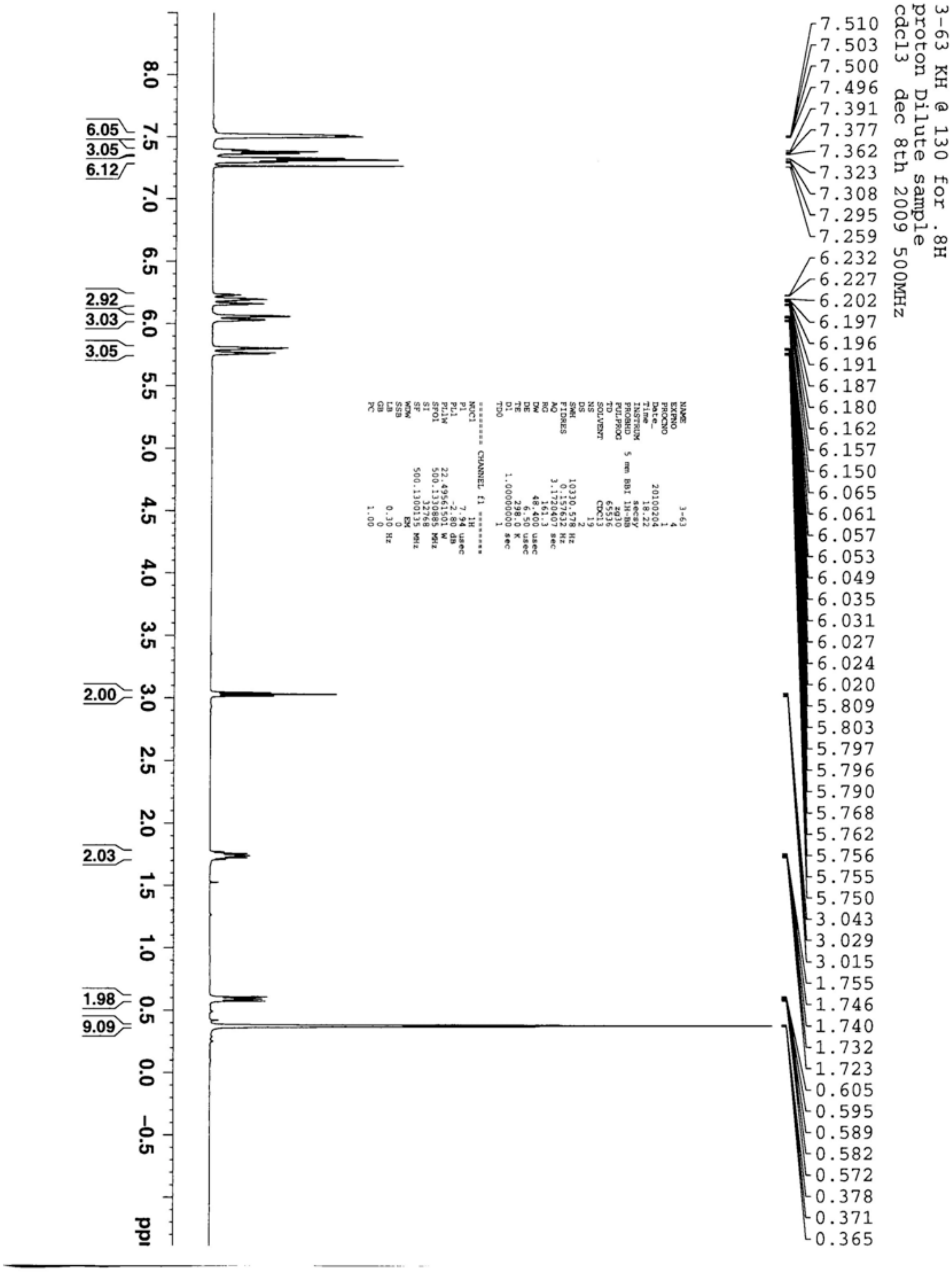
500 MHz

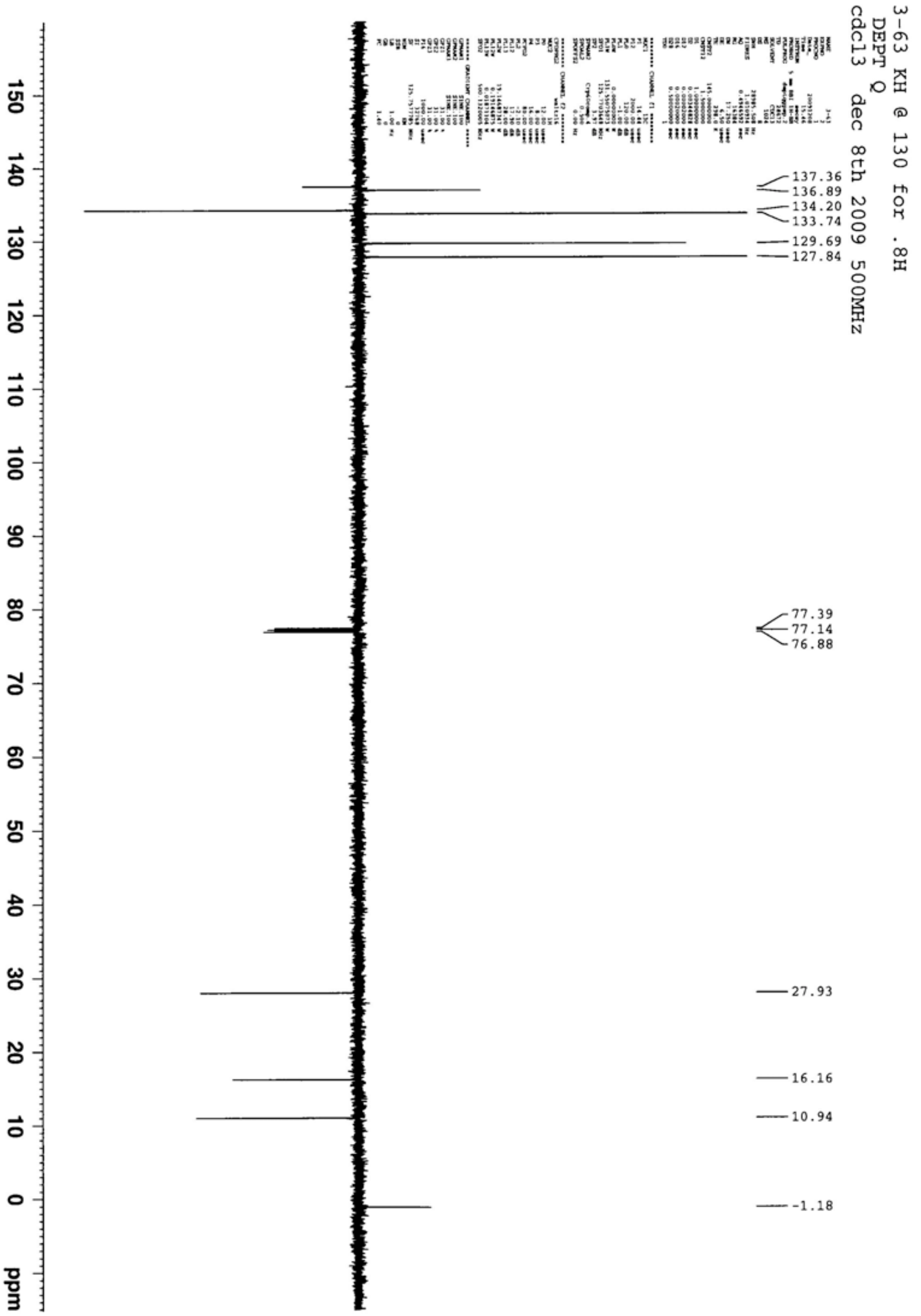
phenyldimethylsilane+chloropropyltrimethoxysilane
cdcl3 august 17th 2009 Silicon 29 NMR

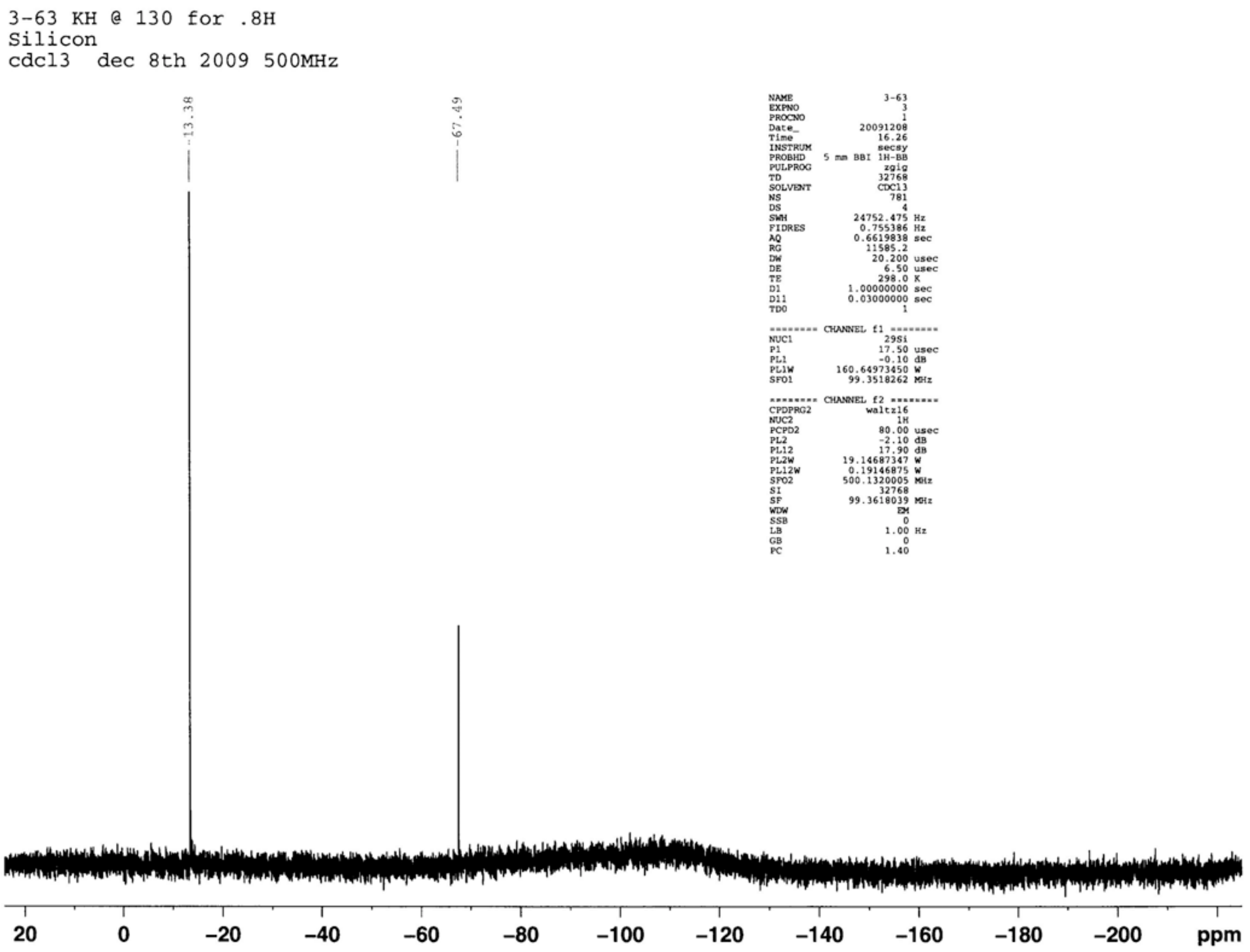


```
NAME     phenyldimethylsilane+chloropropyltrimethoxysilane
EXPNO                  2
PROCNO                 1
Date_           20090820
Time               12.03
INSTRUM            secsy
PROBHD   5 mm BBI 1H-BB
PULPROG             zgig
TD                 32768
SOLVENT            CDCl3
NS                   594
DS                     4
SWH            24752.475 Hz
FIDRES          0.755386 Hz
AQ             0.6619838 sec
RG               14596.5
DW                20.200 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                29Si
P1                 17.50 usec
PL1                -0.10 dB
PL1W        160.64973450 W
SFO1          99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.10 dB
PL12               17.90 dB
PL2W         19.14687347 W
PL12W         0.19146875 W
SFO2         500.1320005 MHz
SI                 32768
SF            99.3618039 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Table 1 Entry #2:



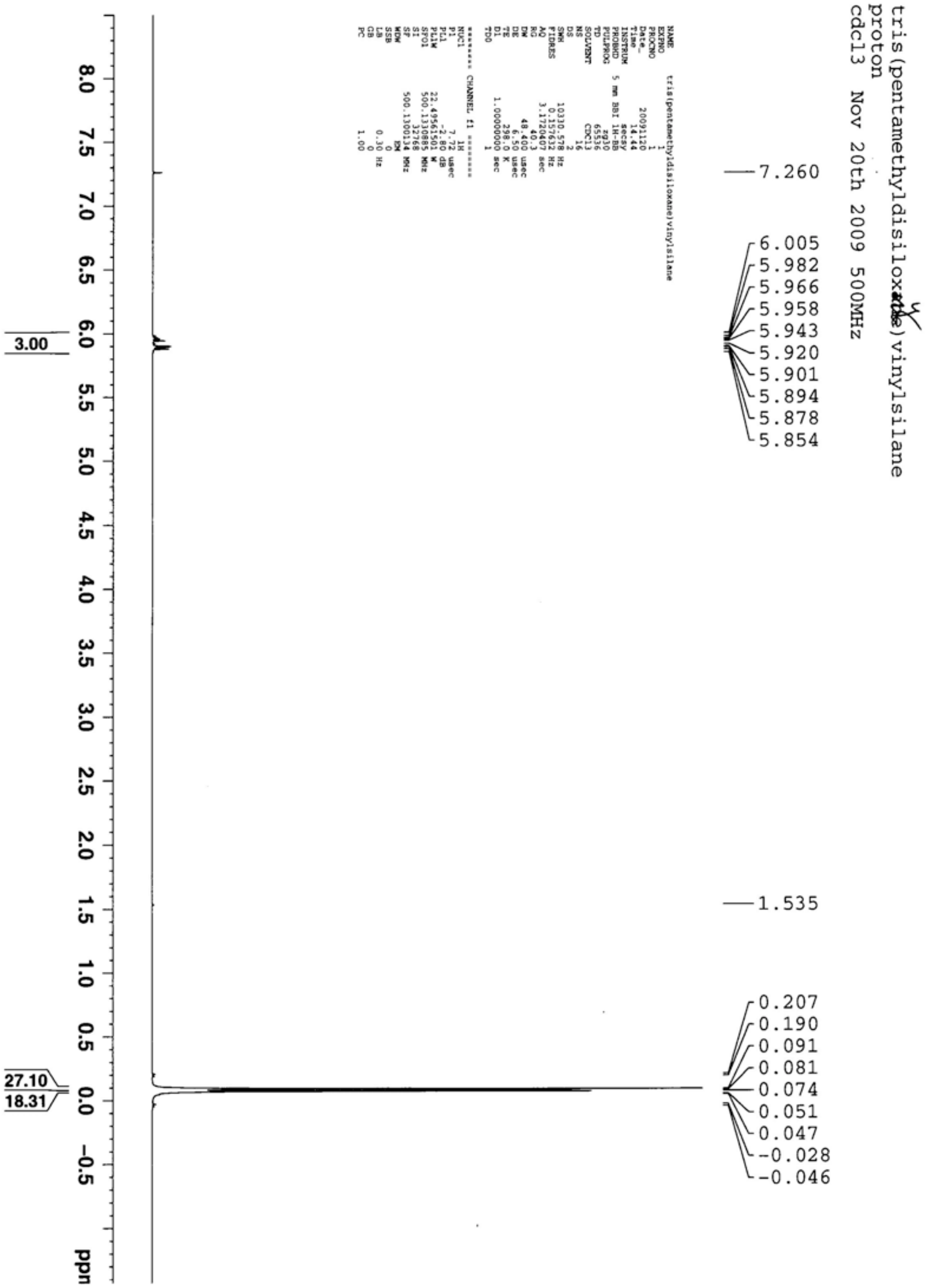

3-63 KH @ 130 for .8H
DEPT Q
cdcl3 dec 8th 2009 500MHz
137.36
136.89
134.20
133.74
129.69
127.84
77.39
77.14
76.88
27.93
16.16
10.94
-1.18
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

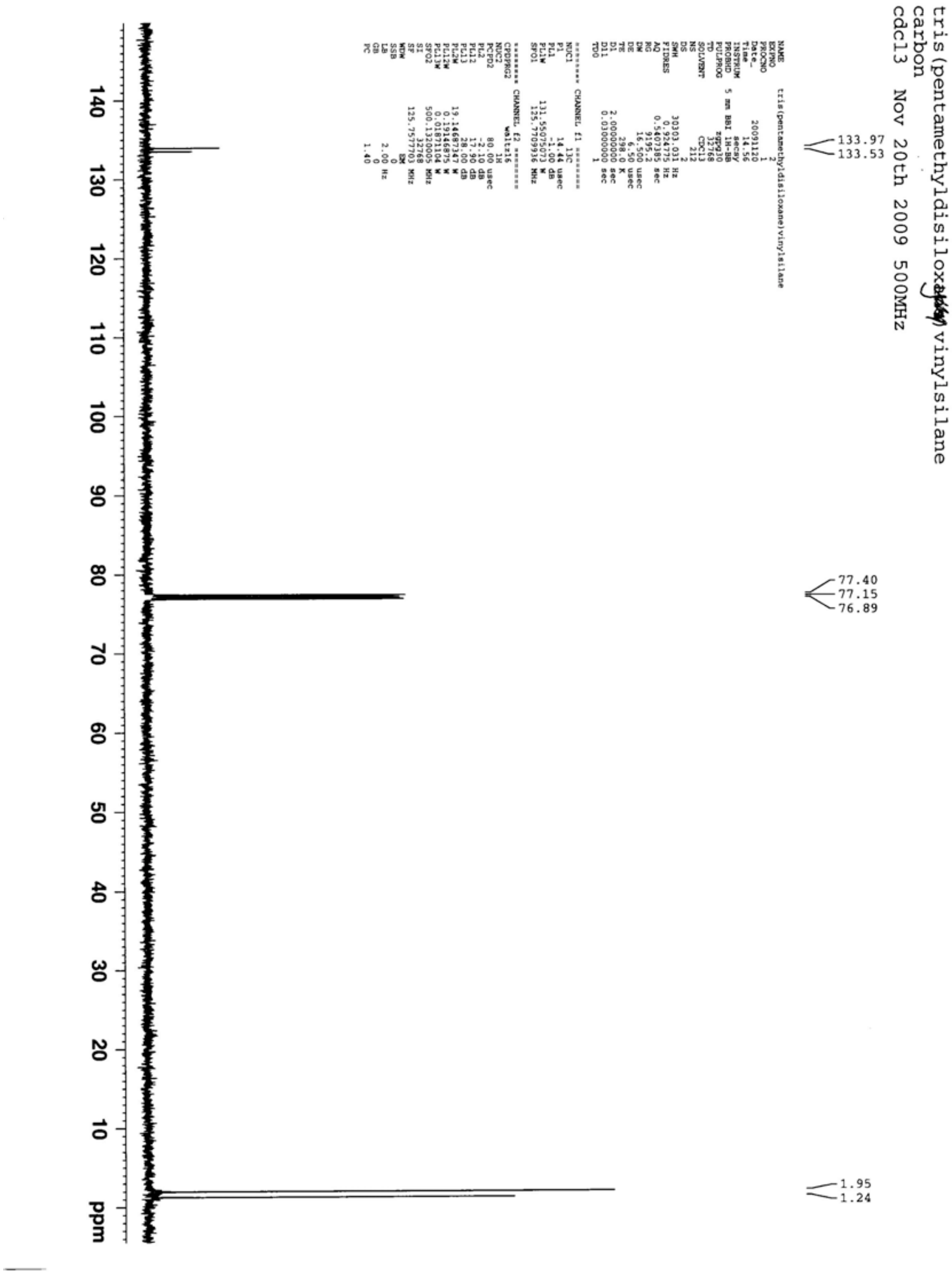


3-63 KH @ 130 for .8H
Silicon
cdcl3 dec 8th 2009 500MHz

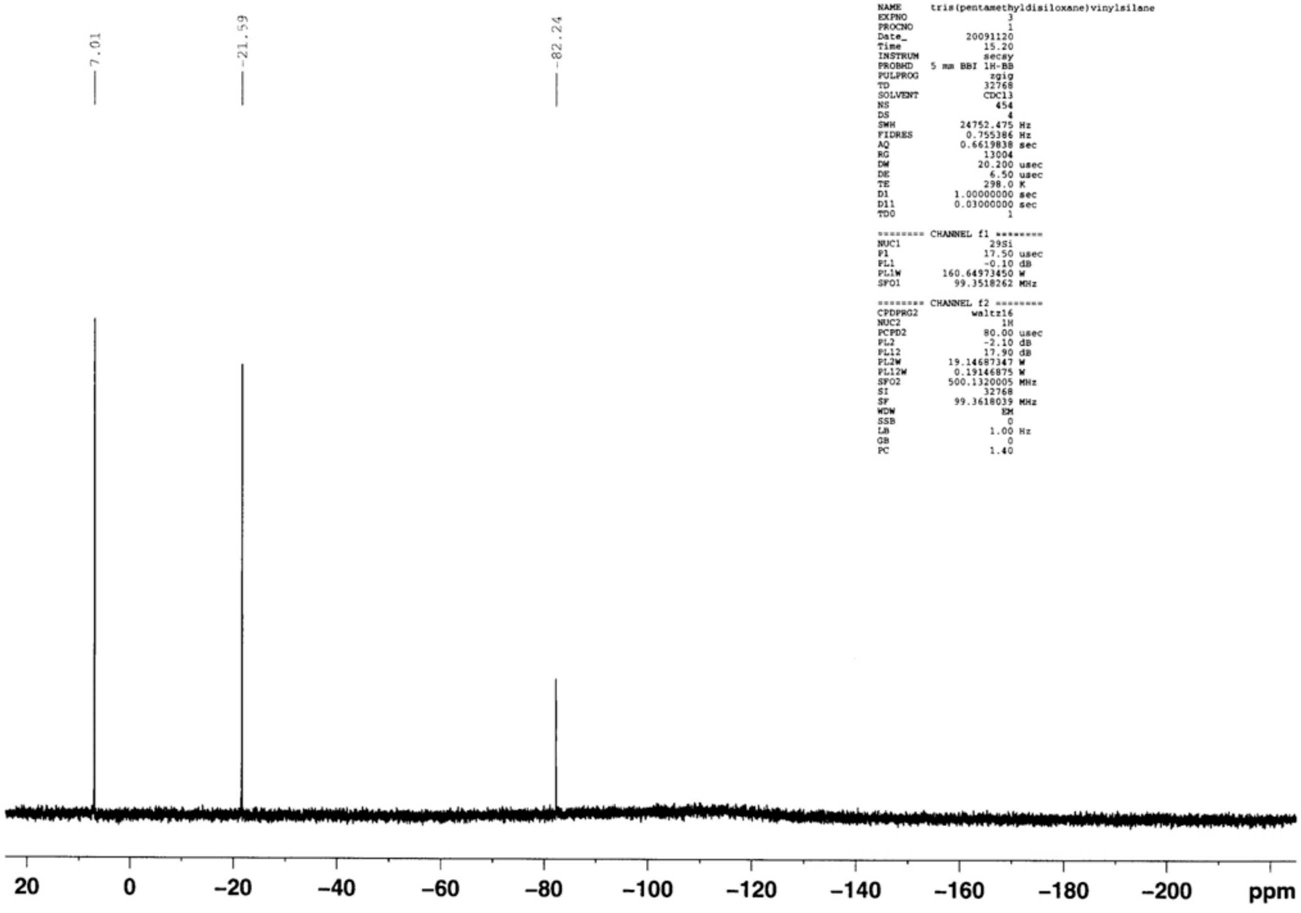
```
NAME             3-63
EXPNO               3
PROCNO              1
Date_        20091208
Time            16.26
INSTRUM         secsy
PROBHD   5 mm BBI 1H-BB
PULPROG          zgig
TD              32768
SOLVENT         CDCl3
NS                781
DS                  4
SWH         24752.475 Hz
FIDRES       0.755386 Hz
AQ          0.6619838 sec
RG            11585.2
DW             20.200 usec
DE               6.50 usec
TE              298.0 K
D1         1.00000000 sec
D11        0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1             29Si
P1              17.50 usec
PL1             -0.10 dB
PL1W     160.64973450 W
SFO1       99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2       waltz16
NUC2               1H
PCPD2           80.00 usec
PL2             -2.10 dB
PL12            17.90 dB
PL2W      19.14687347 W
PL12W      0.19146875 W
SFO2      500.1320005 MHz
SI              32768
SF         99.3618039 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

Table 1 Entry #3:



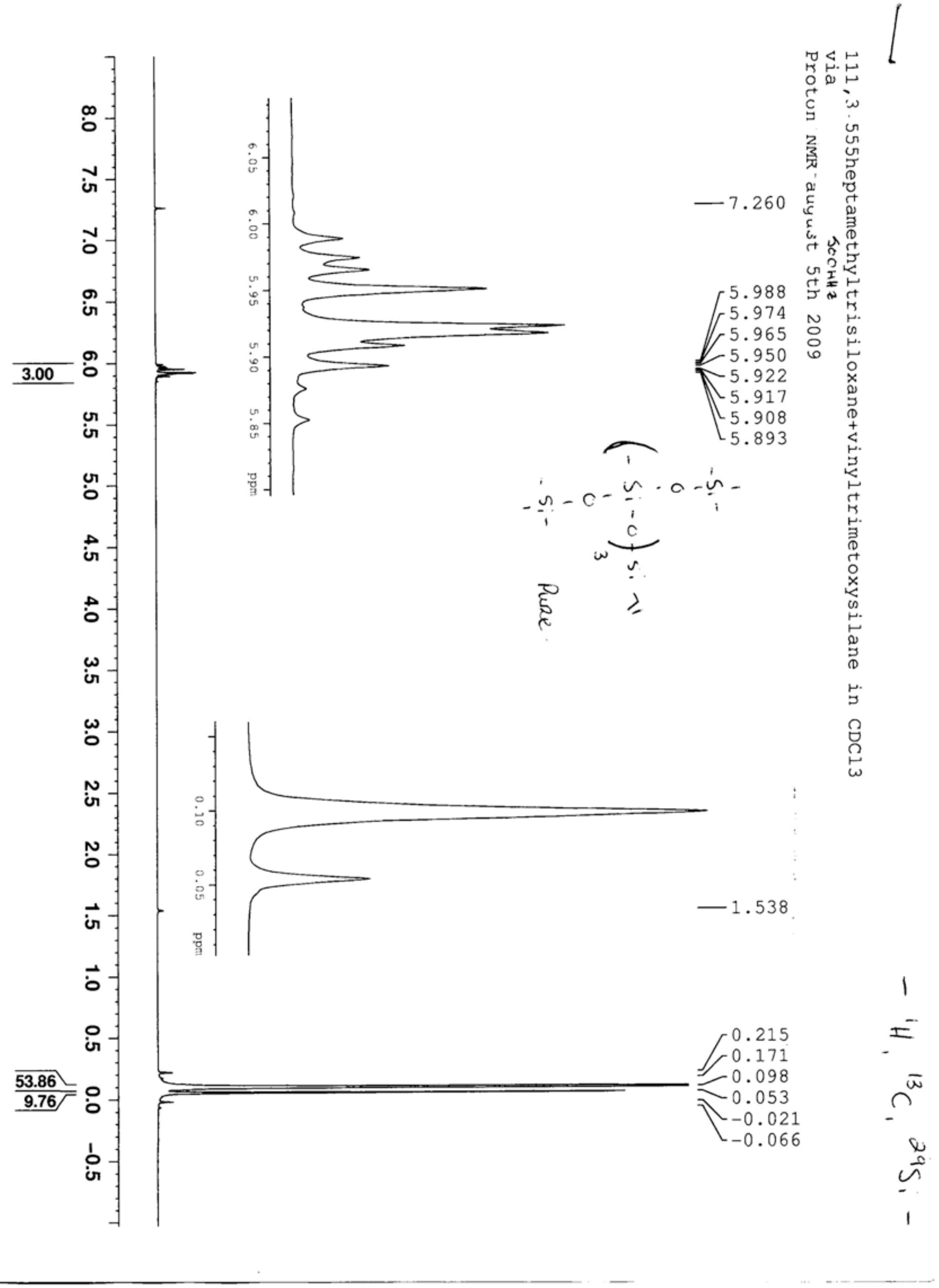

tris(pentamethyldisiloxane)vinylsilane
carbon
cdcl3 Nov 20th 2009 500MHz
NAME tris(pentamethyldisiloxane)vinylsilane
EXPNO 2
PROCNO 1
Date_ 20091120
Time 14.56
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 212
DS 2
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407385 sec
RG 9195.2
DW 16.500 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.44 usec
PL1 -1.00 dB
PL1W 131.55075073 W
SFO1 125.7709936 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 17.90 dB
PL13 28.00 dB
PL2W 19.14687347 W
PL12W 0.19146875 W
PL13W 0.01871104 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577703 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
133.97
133.53
77.40
77.15
76.89
1.95
1.24
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

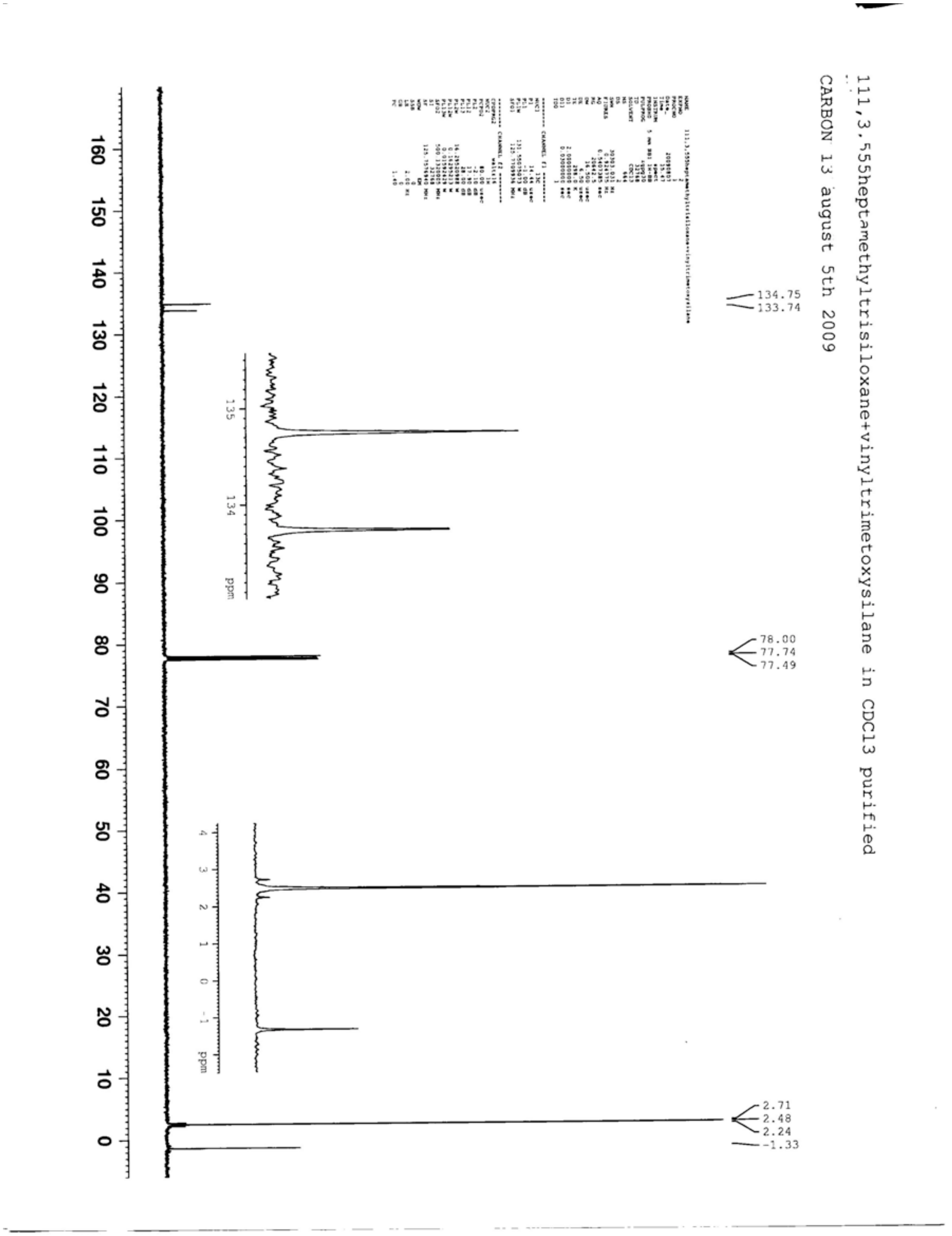
tris(pentamethyldisilox~~ane~~y)vinylsilane
silicon
cdcl3 Nov 20th 2009 500MHz



```
NAME     tris(pentamethyldisiloxane)vinylsilane
EXPNO                   3
PROCNO                  1
Date_            20091120
Time                15.20
INSTRUM             secsy
PROBHD   5 mm BBI 1H-BB
PULPROG              zgig
TD                  32768
SOLVENT             CDCl3
NS                    454
DS                      4
SWH             24752.475 Hz
FIDRES           0.755386 Hz
AQ              0.6619838 sec
RG                  13004
DW                 20.200 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                 29Si
P1                  17.50 usec
PL1                 -0.10 dB
PL1W         160.64973450 W
SFO1           99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.10 dB
PL12                17.90 dB
PL2W          19.14687347 W
PL12W          0.19146875 W
SFO2          500.1320005 MHz
SI                  32768
SF             99.3618039 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Table 1 Entry #4:




111,3.555heptamethyltrisiloxane+vinyltrimetoxysilane in CDCl3 purified
CARBON 13 august 5th 2009
134.75
133.74
78.00
77.74
77.49
2.71
2.48
2.24
-1.33
135
134
ppm
4
3
2
1
0
-1
ppm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

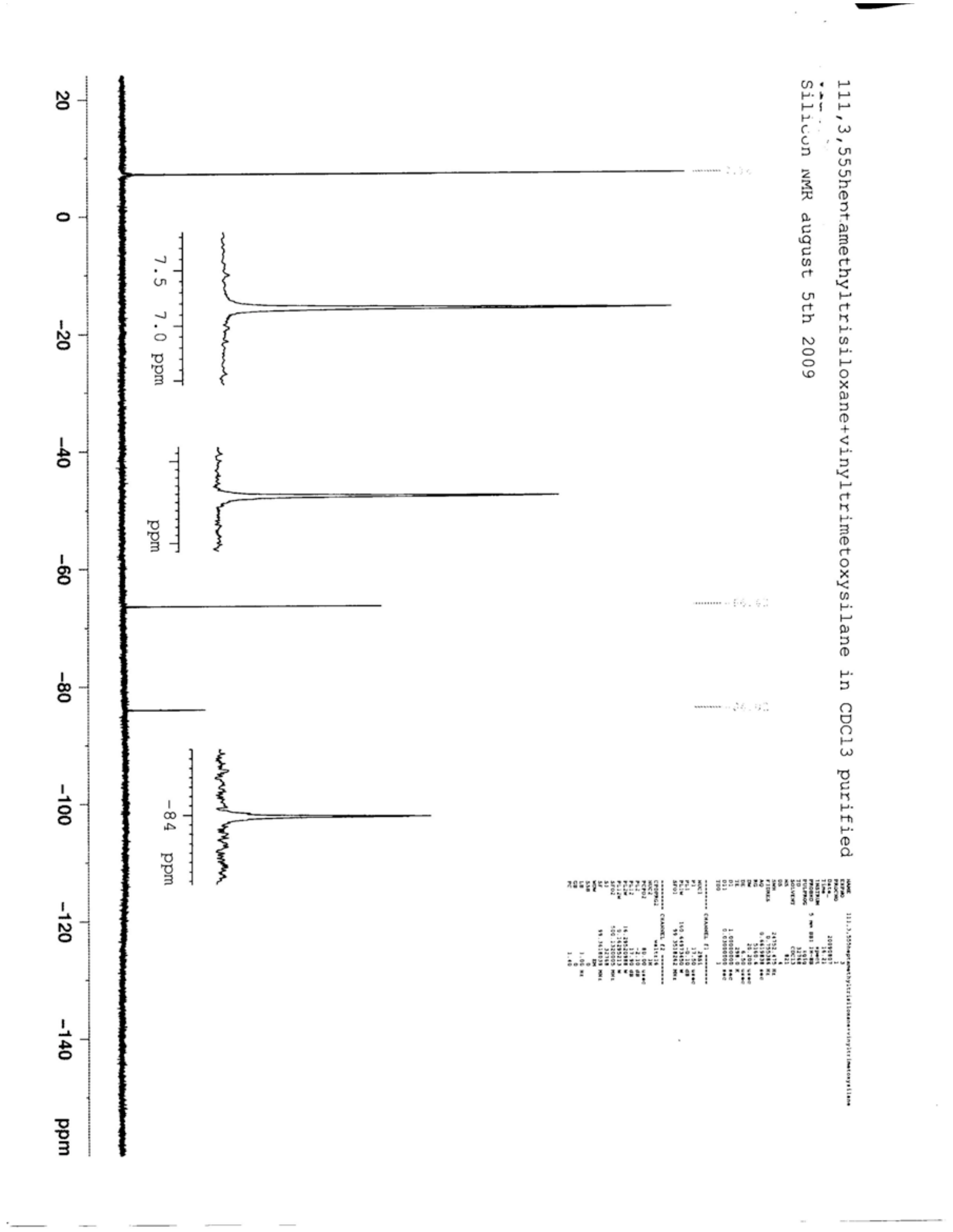
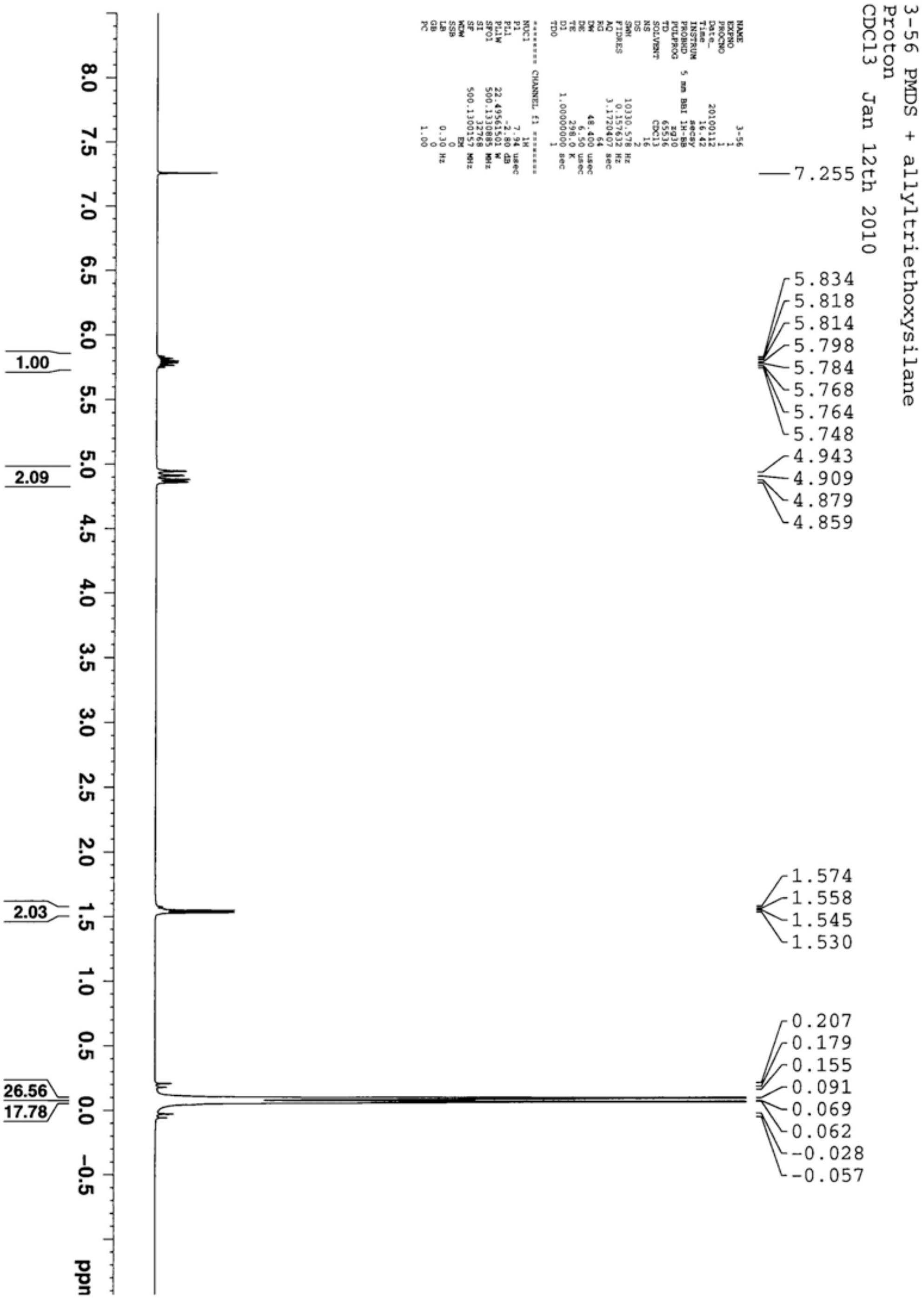
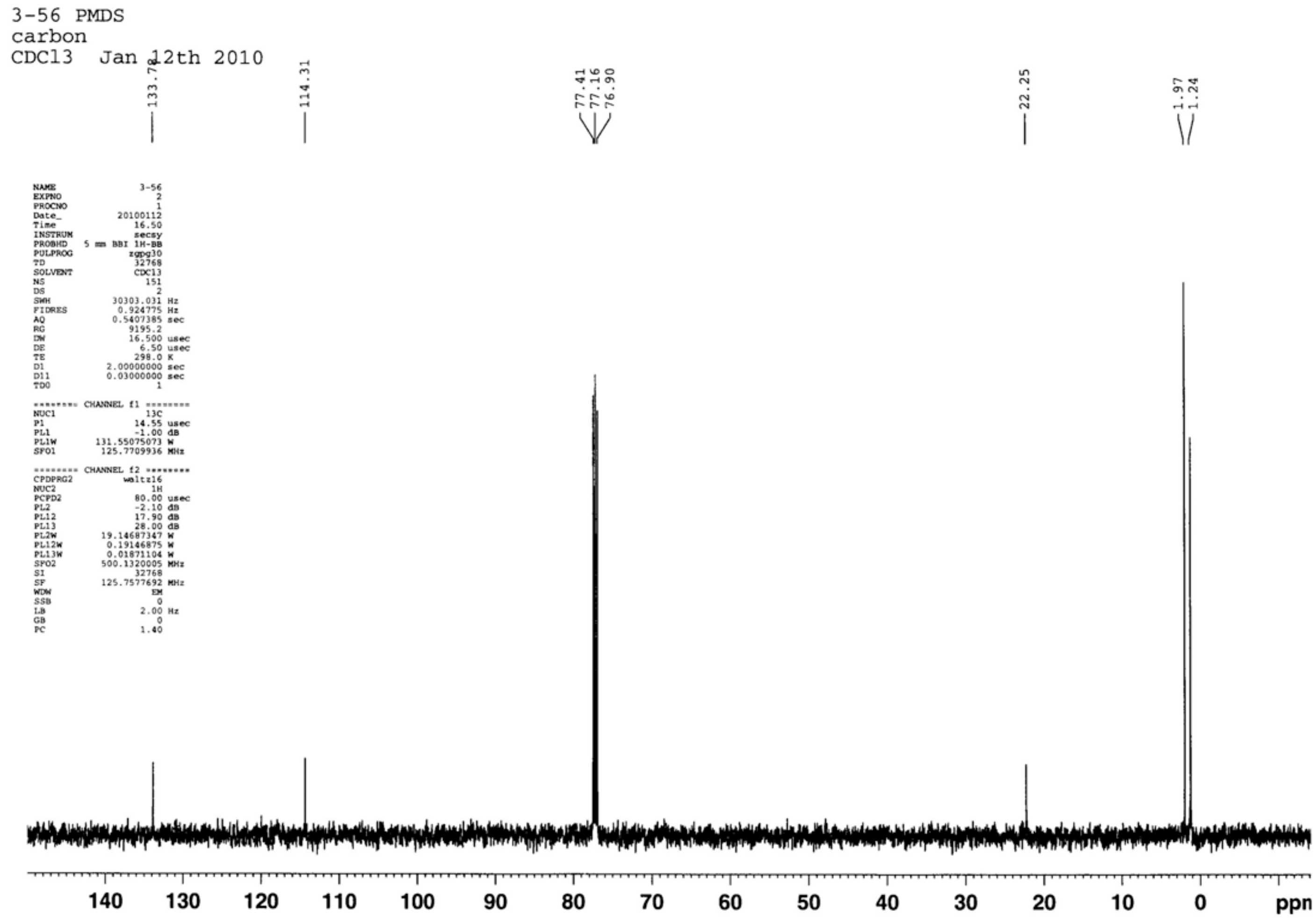

111,3,555heptamethyltrisiloxane+vinyltrimetoxysilane in CDCl3 purified
Sillicon NMR august 5th 2009
7.5 7.0 ppm
ppm
-84 ppm
20
0
-20
-40
-60
-80
-100
-120
-140
ppm

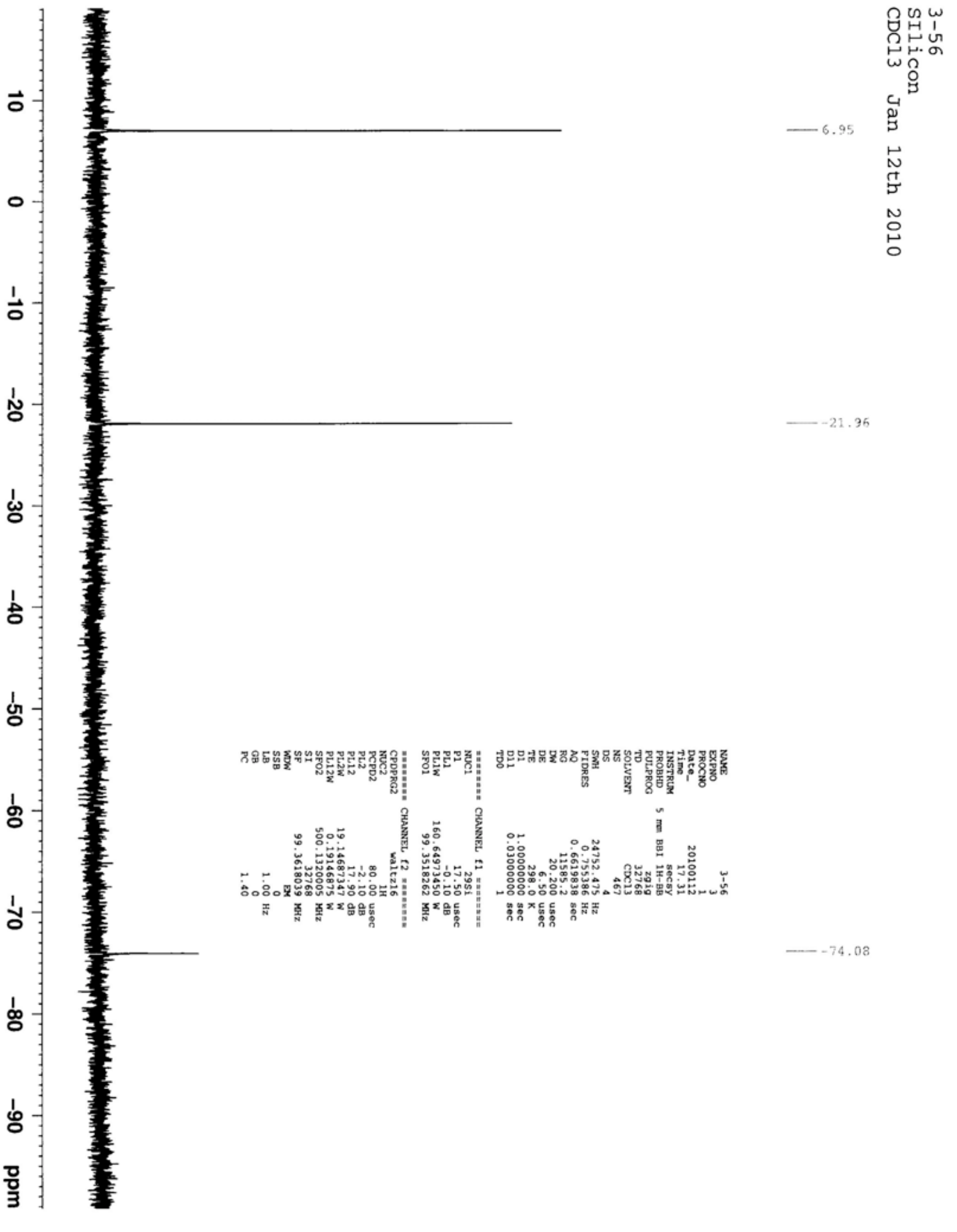
Table 1 Entry #5:





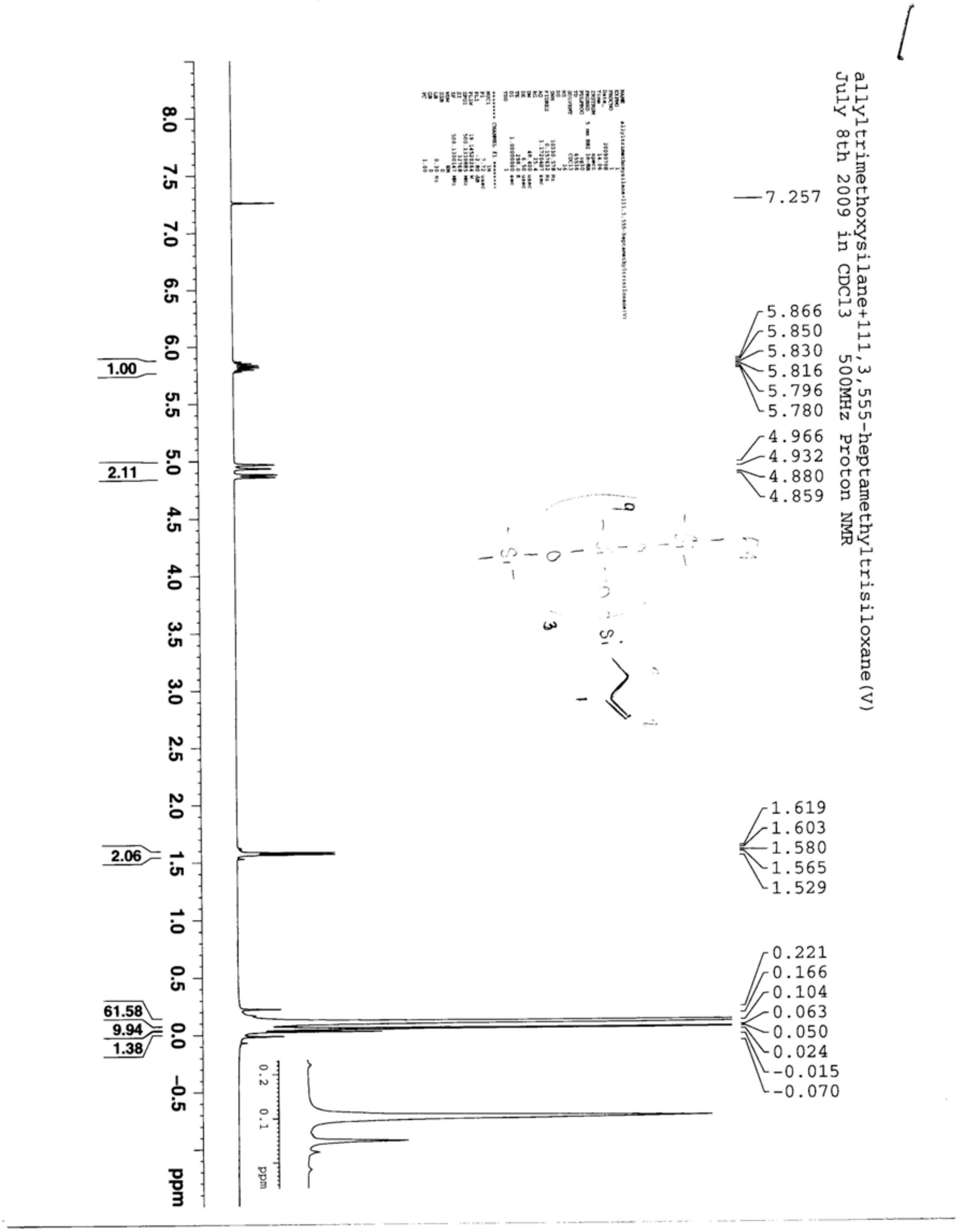
```
3-56 PMDS
carbon
CDCl3  Jan 12th 2010
```

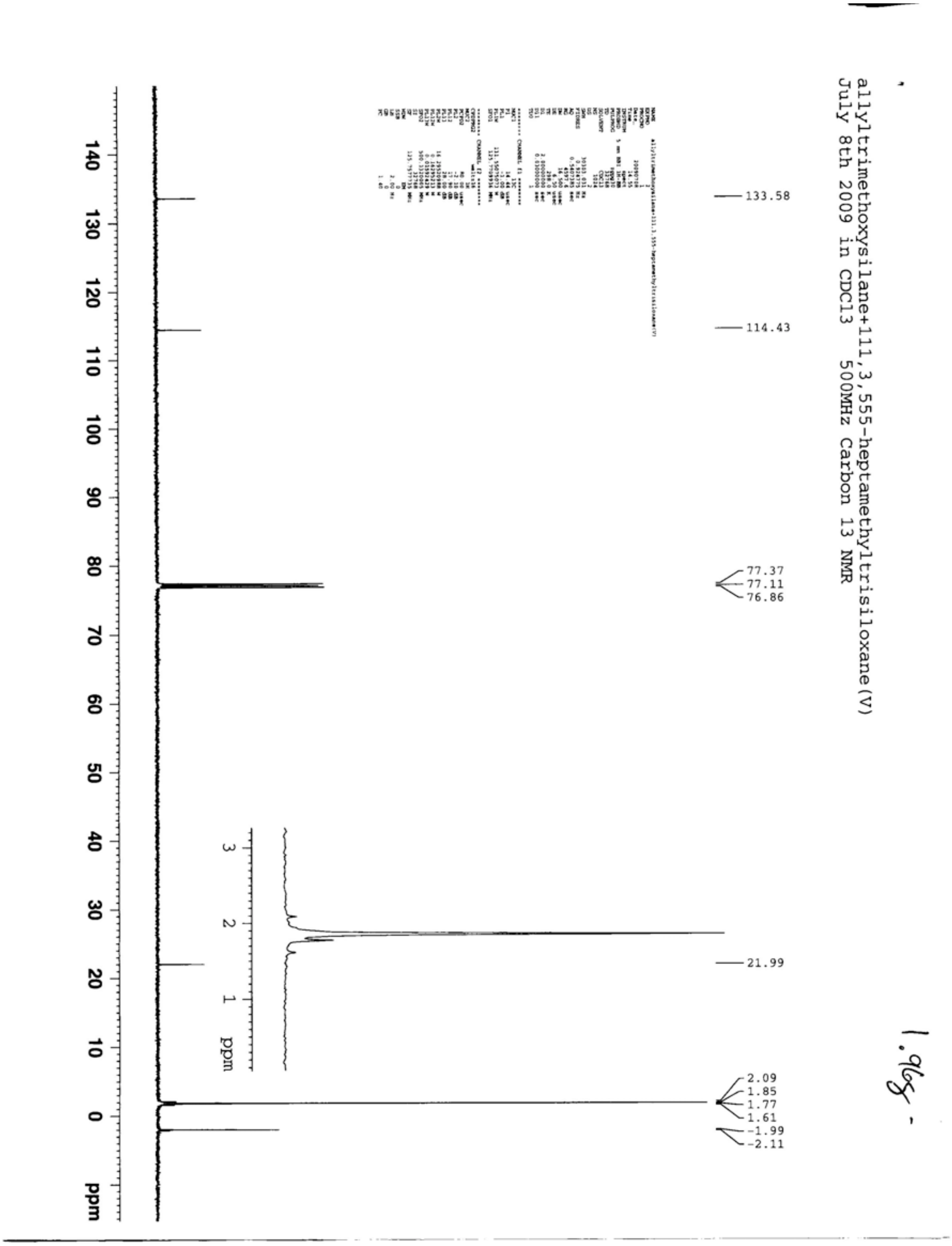
```
NAME              3-56
EXPNO                2
PROCNO               1
Date_         20100112
Time             16.50
INSTRUM          secsy
PROBHD   5 mm BBI 1H-BB
PULPROG        zgpg30
TD               32768
SOLVENT          CDCl3
NS                 151
DS                   2
SWH          30303.031 Hz
FIDRES        0.924775 Hz
AQ           0.5407385 sec
RG              9195.2
DW              16.500 usec
DE                6.50 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               14.55 usec
PL1              -1.00 dB
PL1W      131.55075073 W
SFO1       125.7709936 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.10 dB
PL12             17.90 dB
PL13             28.00 dB
PL2W       19.14687347 W
PL12W       0.19146875 W
PL13W       0.01871104 W
SFO2       500.1320005 MHz
SI               32768
SF         125.7577692 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.40
```



```
NAME              3-56
EXPNO                3
PROCNO               1
Date_         20100112
Time             17.31
INSTRUM          secsy
PROBHD   5 mm BBI 1H-BB
PULPROG           zgig
TD               32768
SOLVENT          CDCl3
NS                 467
DS                   4
SWH          24752.475 Hz
FIDRES        0.755386 Hz
AQ           0.6619838 sec
RG             11585.2
DW              20.200 usec
DE                6.50 usec
TE               298.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1              29Si
P1               17.50 usec
PL1              -0.10 dB
PL1W      160.64973450 W
SFO1       99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.10 dB
PL12             17.90 dB
PL2W       19.14687347 W
PL12W       0.19146875 W
SFO2       500.1320005 MHz
SI               32768
SF          99.3618039 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

Table 1 Entry #6:




allyltrimethoxysilane+111,3,555-heptamethyltrisiloxane(V)
July 8th 2009 in CDCl3   500MHz Carbon 13 NMR
133.58
114.43
77.37
77.11
76.86
21.99
2.09
1.85
1.77
1.61
-1.99
-2.11
1.965 -
3
2
1
ppm
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

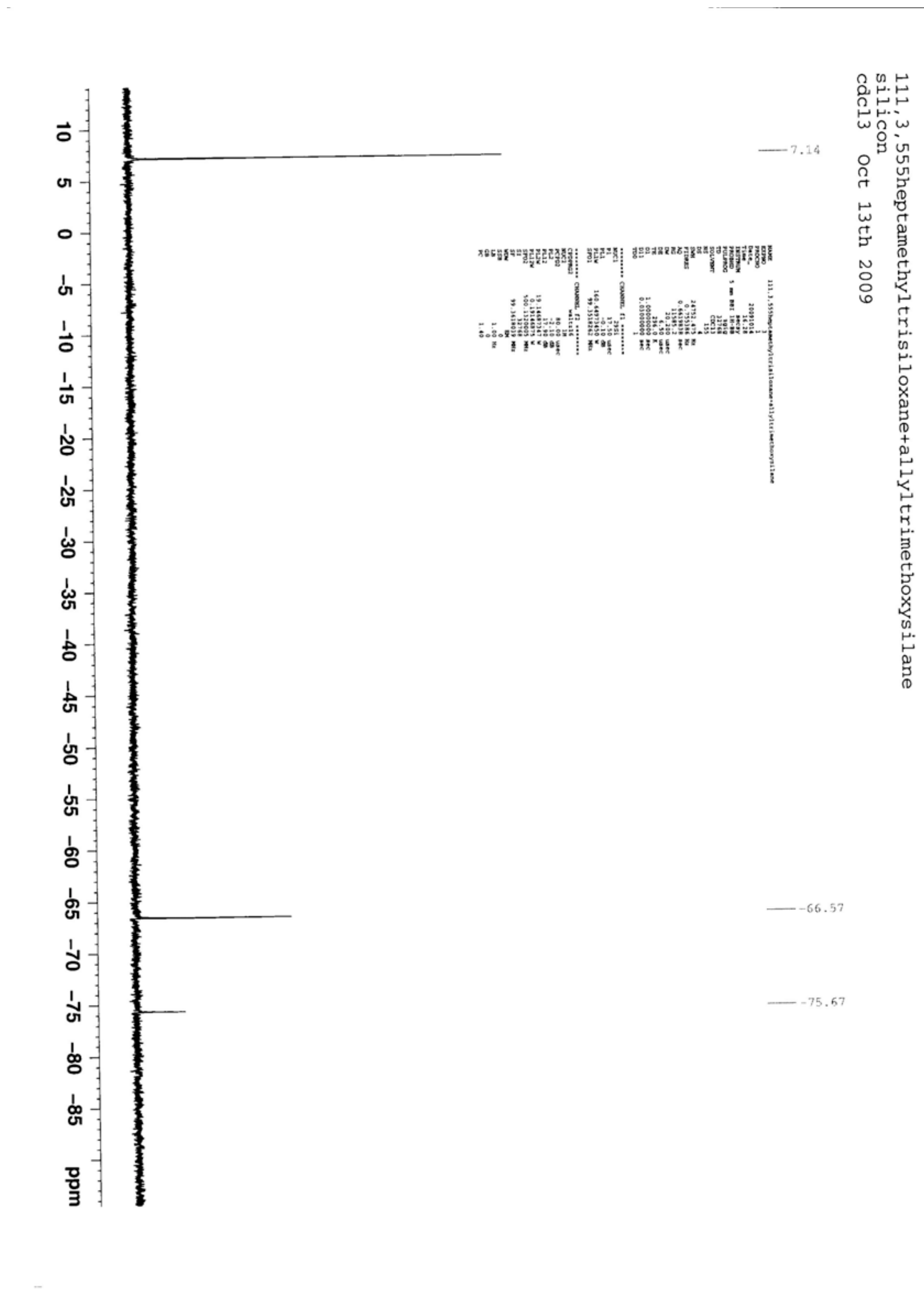
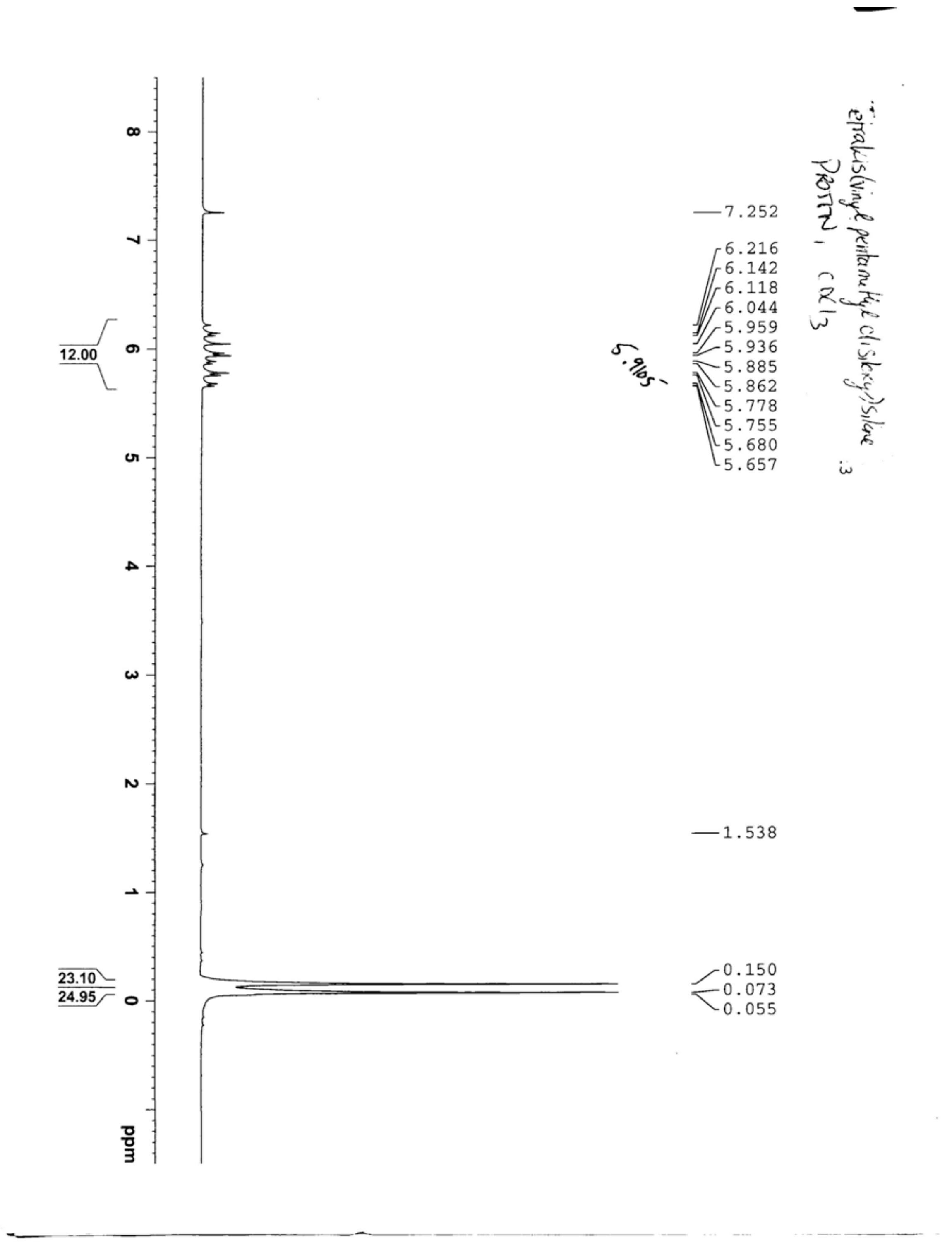
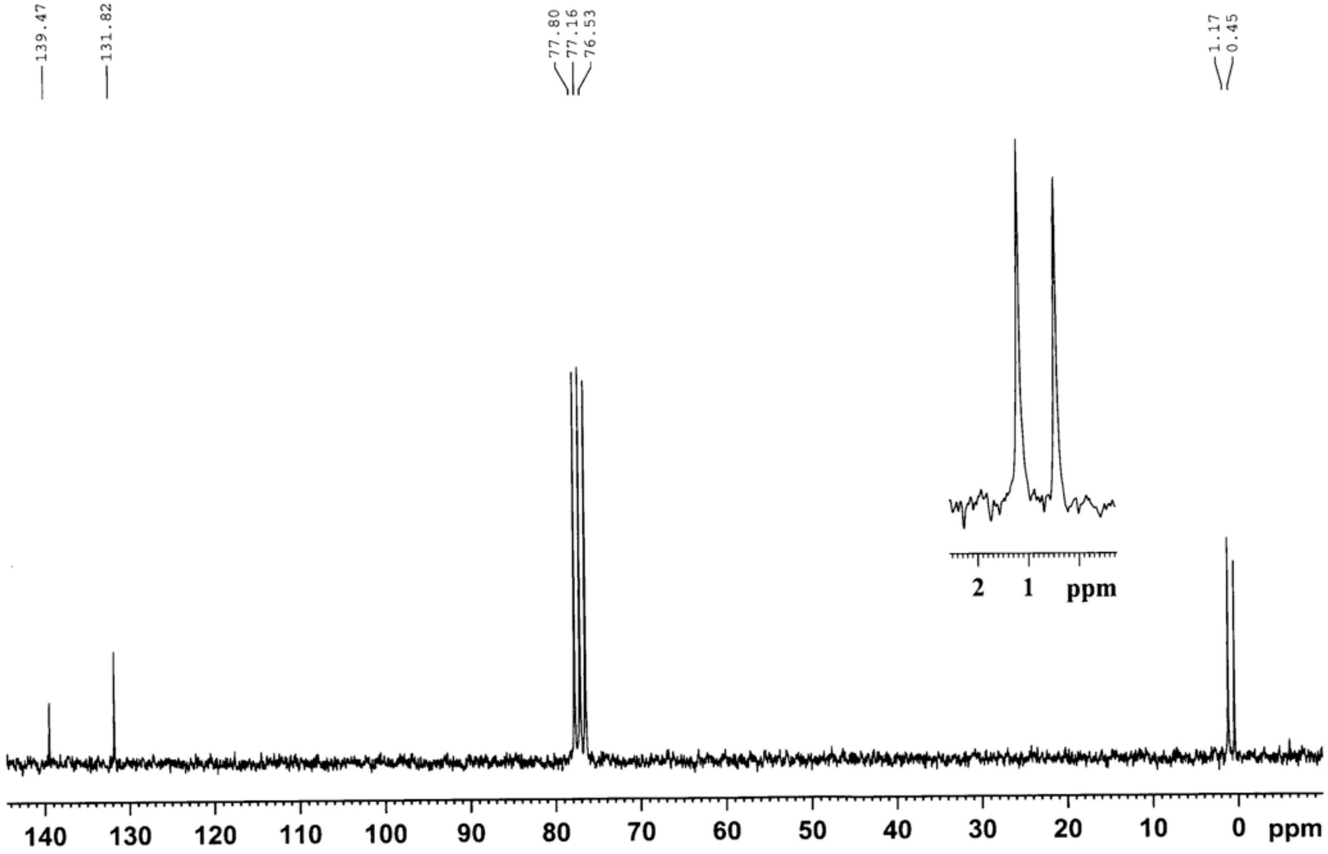

111,3,555heptamethyltrisiloxane+allyltrimethoxysilane
silicon
cdcl3 Oct 13th 2009
7.14
-66.57
-75.67
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
ppm

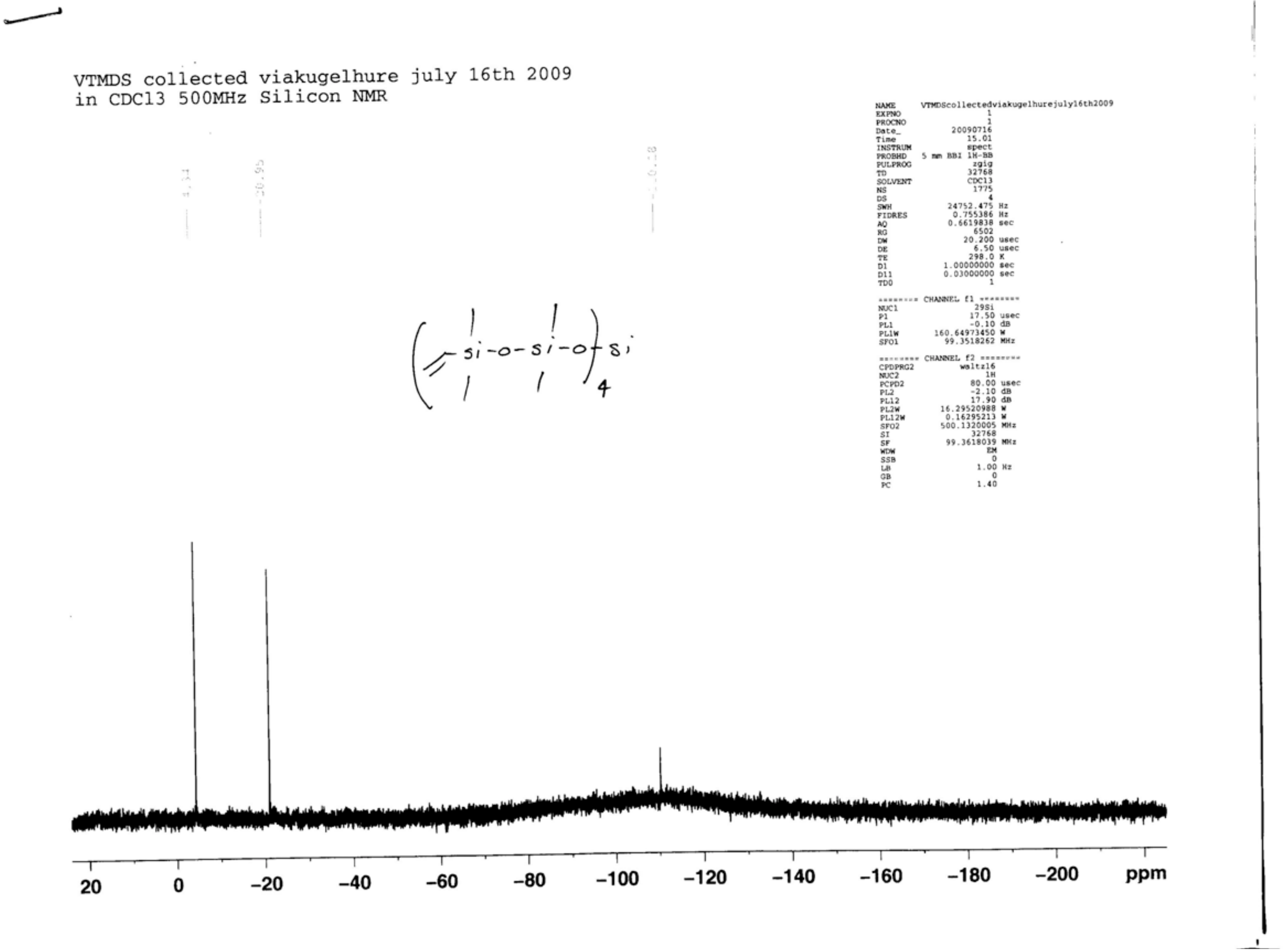
Table 1 Entry #7:



Tetrakis(vinyl(tetramethyl(disiloxy)silane
CDCl$_3$ Carbon

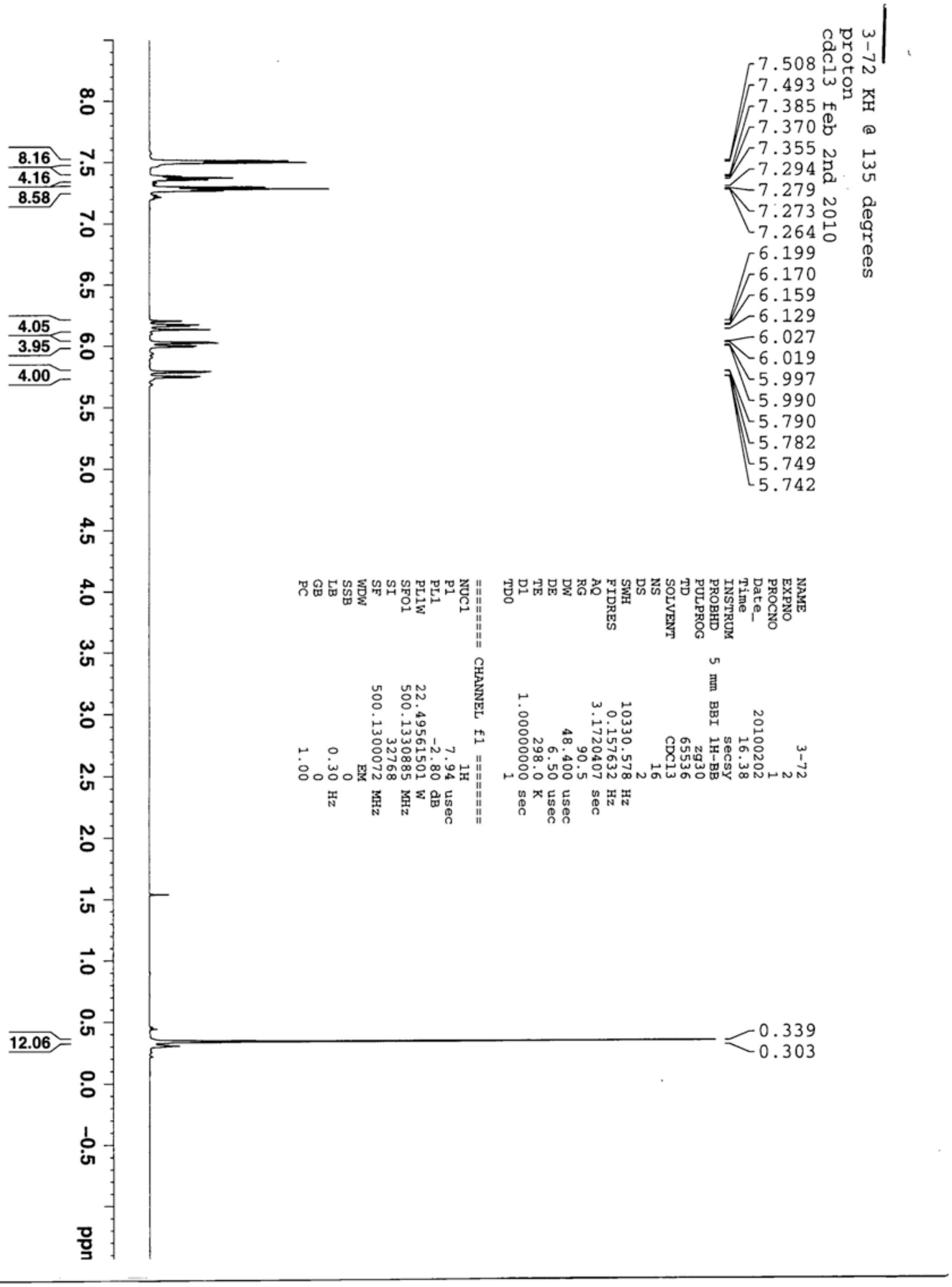


VTMDS collected viakugelhure july 16th 2009
in CDCl3 500MHz Silicon NMR

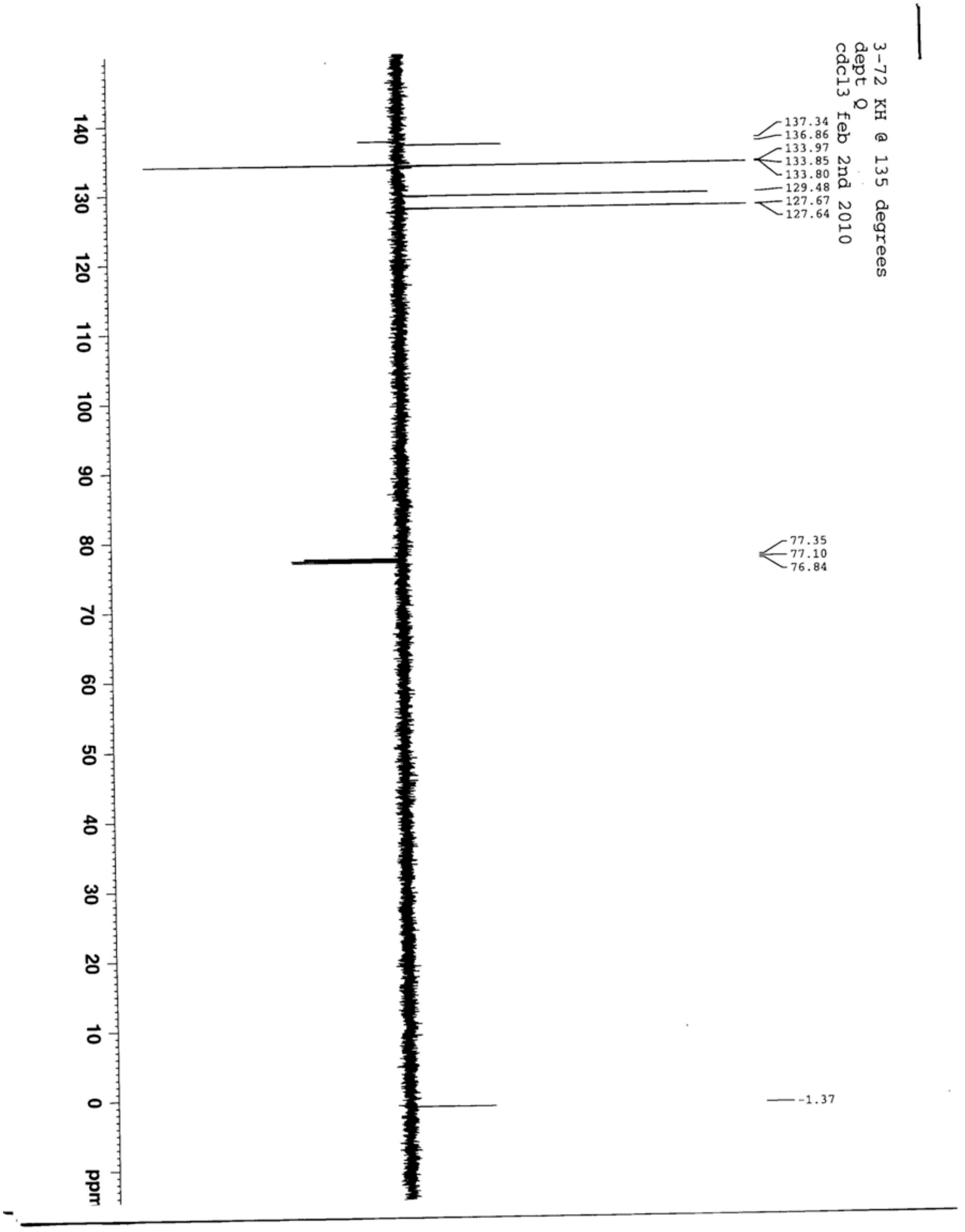


```
NAME      VTMDScollectedviakugelhurejuly16th2009
EXPNO                    1
PROCNO                   1
Date_             20090716
Time                 15.01
INSTRUM              spect
PROBHD      5 mm BBI 1H-BB
PULPROG               zgig
TD                   32768
SOLVENT              CDCl3
NS                    1775
DS                       4
SWH              24752.475 Hz
FIDRES            0.755386 Hz
AQ               0.6619838 sec
RG                    6502
DW                  20.200 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  29Si
P1                   17.50 usec
PL1                  -0.10 dB
PL1W          160.64973450 W
SFO1            99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 17.90 dB
PL2W           16.29520988 W
PL12W           0.16295213 W
SFO2           500.1320005 MHz
SI                   32768
SF              99.3618039 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

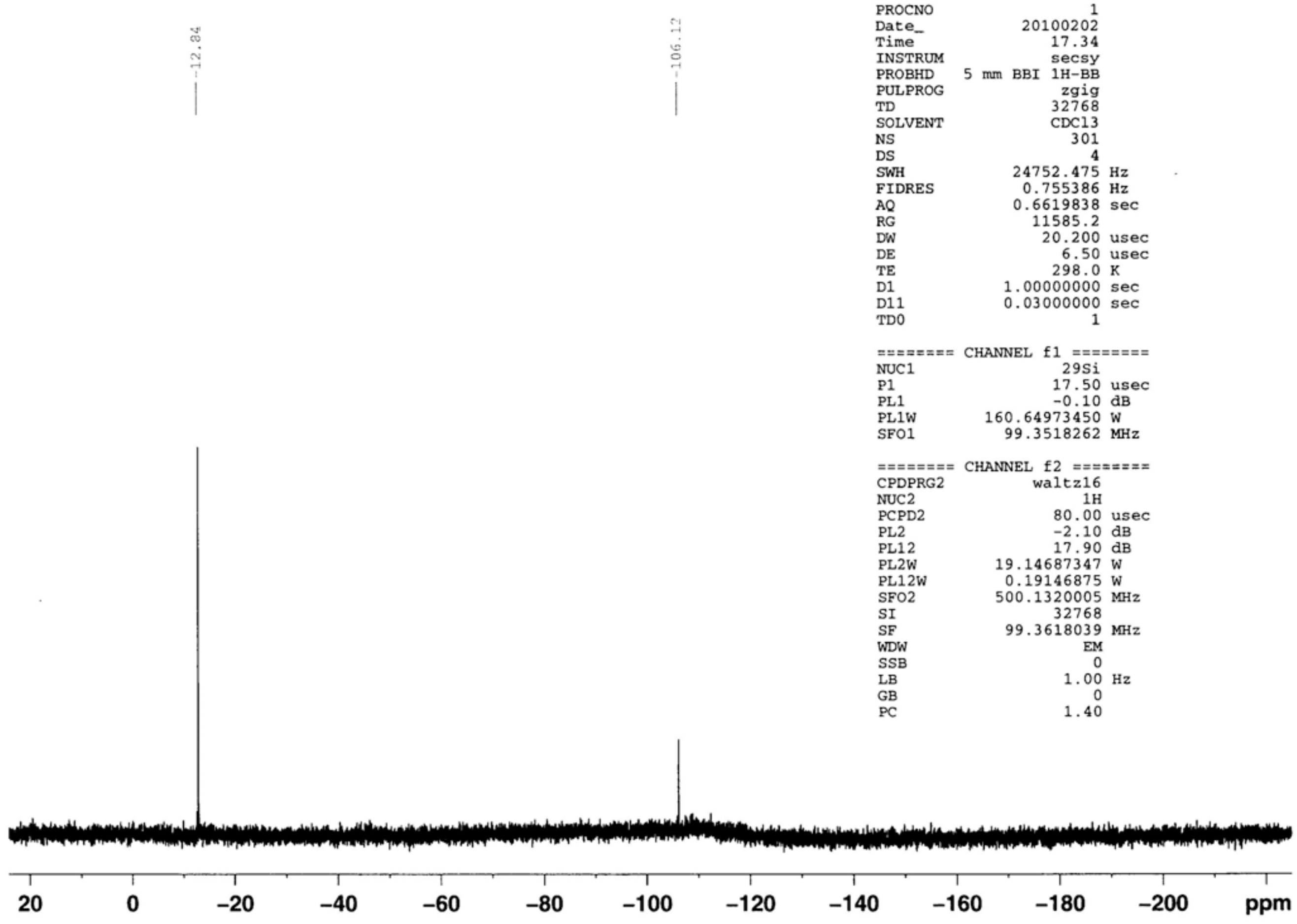
Table 1 Entry #8:



```
NAME                3-72
EXPNO                  2
PROCNO                 1
Date_           20100202
Time               16.38
INSTRUM            secsy
PROBHD   5 mm BBI 1H-BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1720407 sec
RG                  90.5
DW                48.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.94 usec
PL1                -2.80 dB
PL1W         22.49561501 W
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300072 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

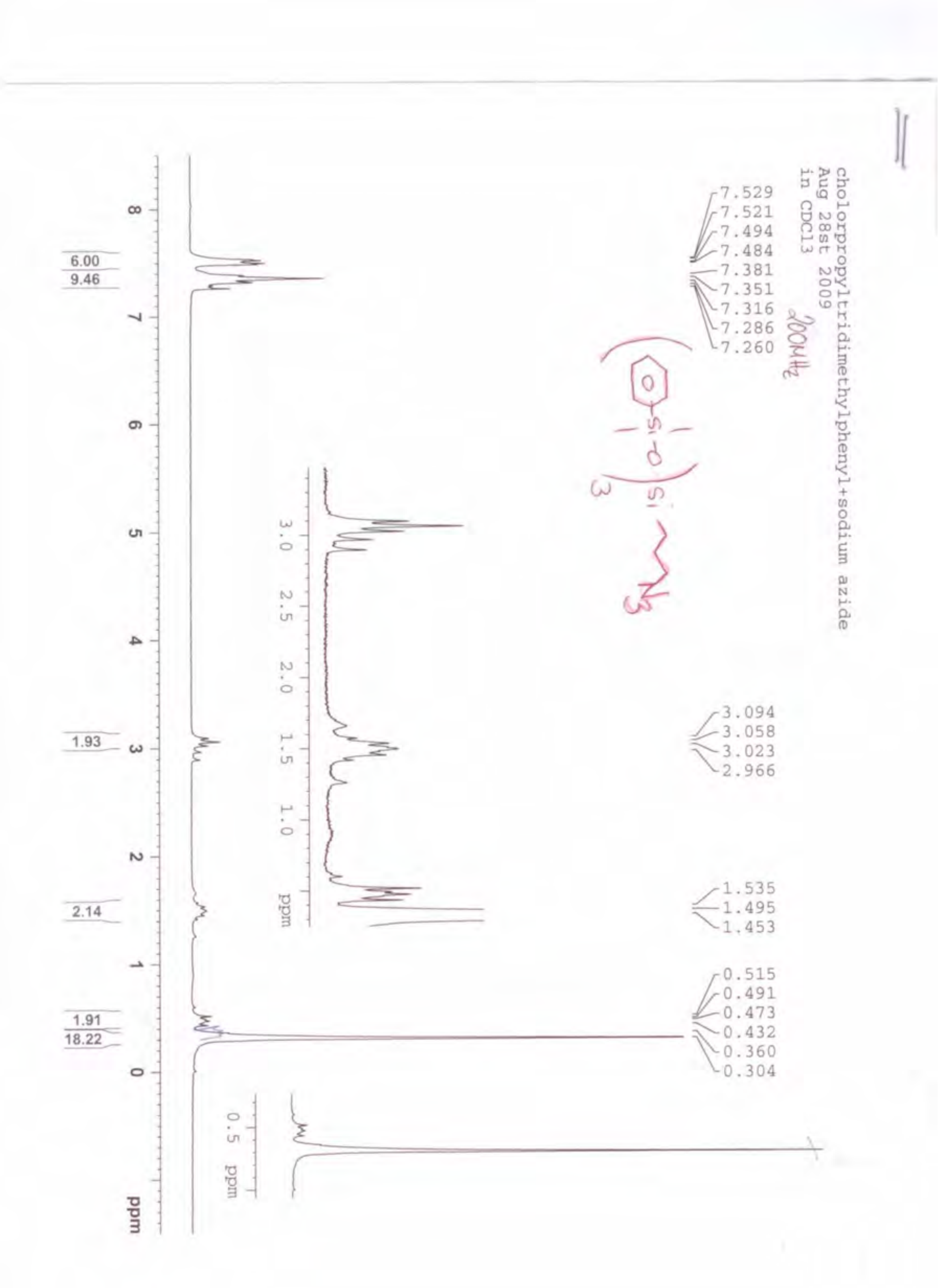

3-72 KH @ 135 degrees
dept Q
cdcl3 feb 2nd 2010
137.34
136.86
133.97
133.85
133.80
129.48
127.67
127.64
77.35
77.10
76.84
-1.37
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

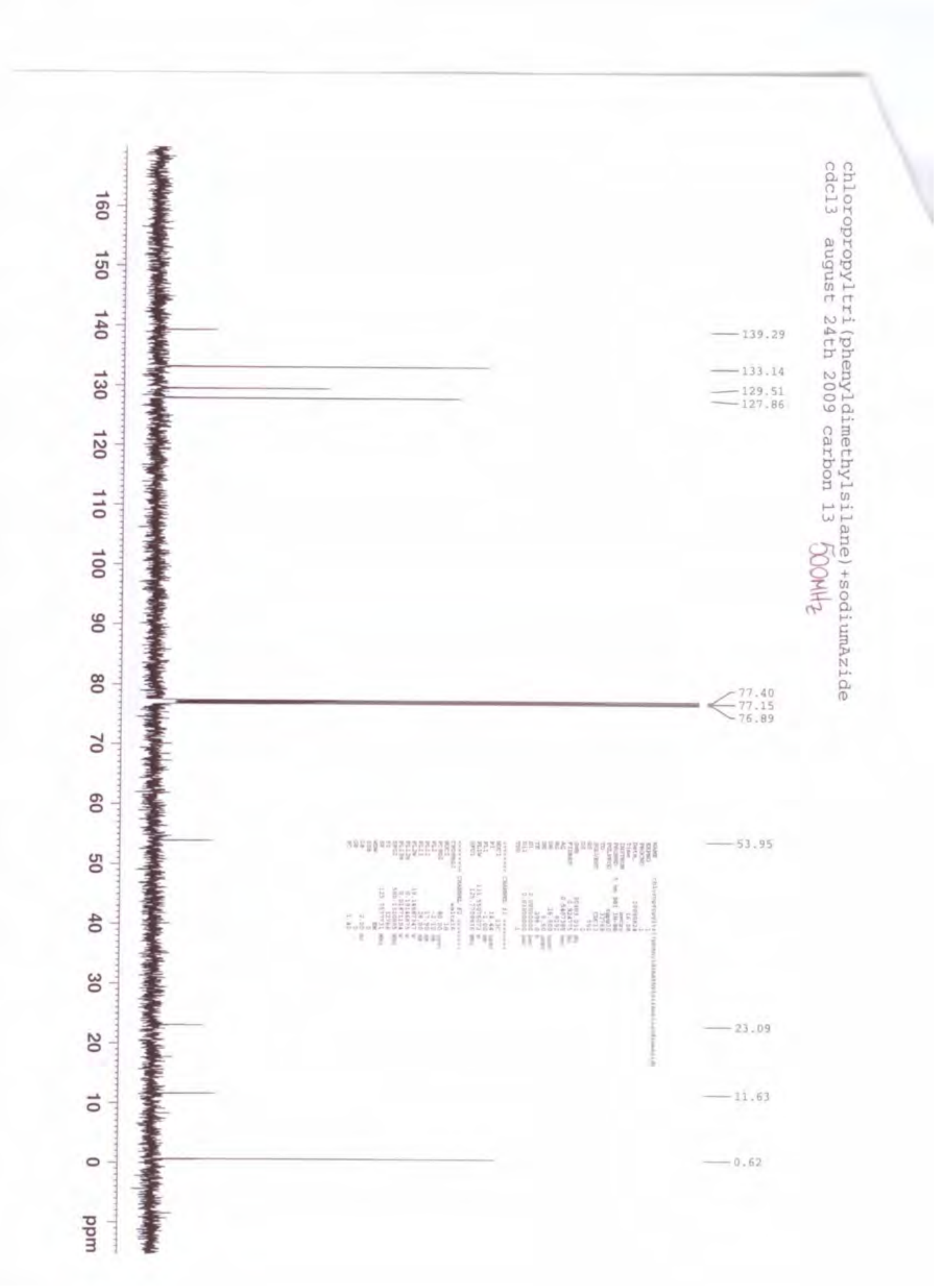
3-72 KH @ 135 degrees
silicon nmr
cdcl3 feb 2nd 2010

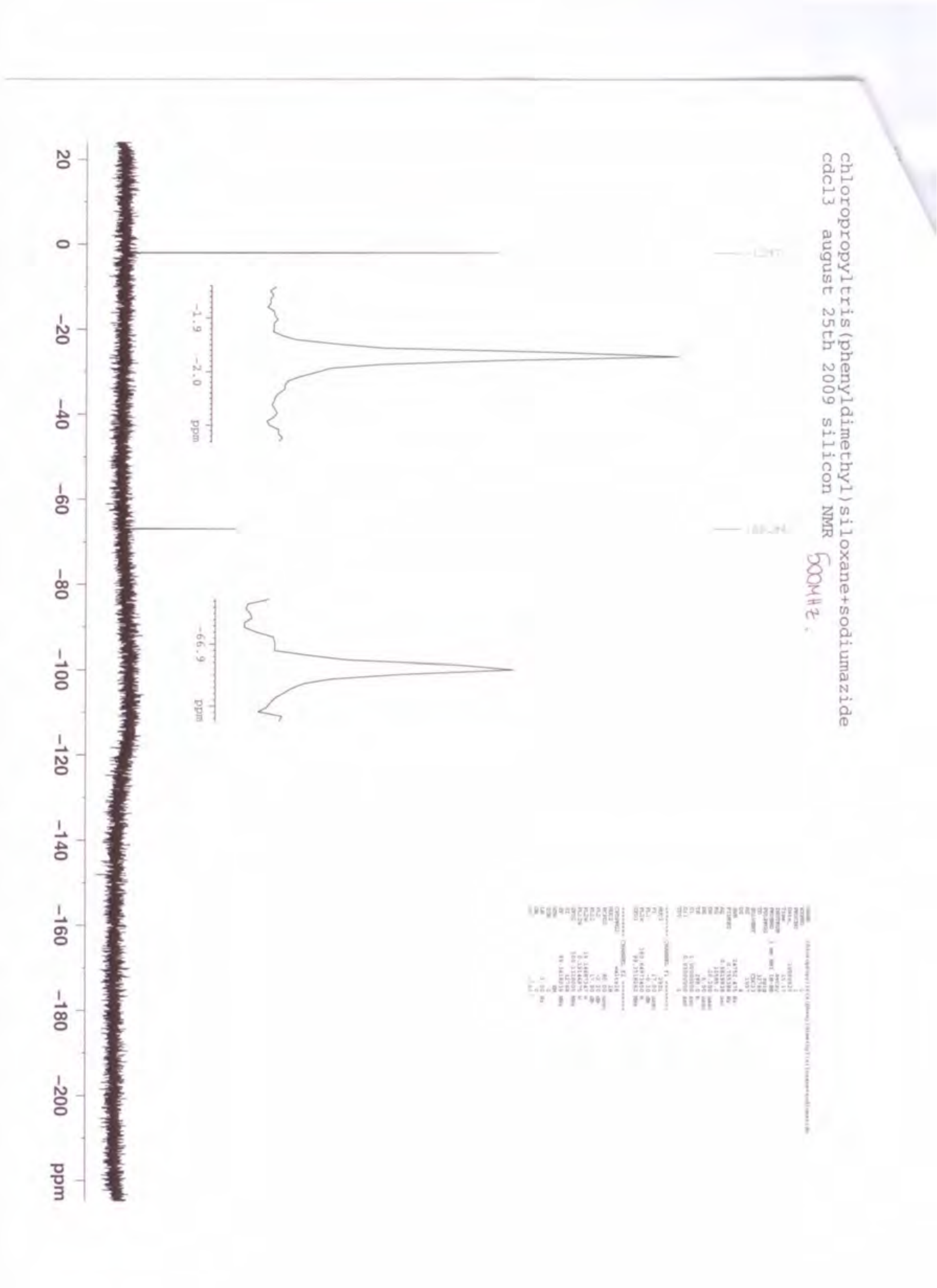


```
NAME              3-72
EXPNO                4
PROCNO               1
Date_         20100202
Time             17.34
INSTRUM          secsy
PROBHD   5 mm BBI 1H-BB
PULPROG           zgig
TD               32768
SOLVENT          CDCl3
NS                 301
DS                   4
SWH          24752.475 Hz
FIDRES        0.755386 Hz
AQ           0.6619838 sec
RG             11585.2
DW              20.200 usec
DE                6.50 usec
TE               298.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1              29Si
P1               17.50 usec
PL1              -0.10 dB
PL1W      160.64973450 W
SFO1       99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.10 dB
PL12             17.90 dB
PL2W       19.14687347 W
PL12W       0.19146875 W
SFO2      500.1320005 MHz
SI               32768
SF          99.3618039 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

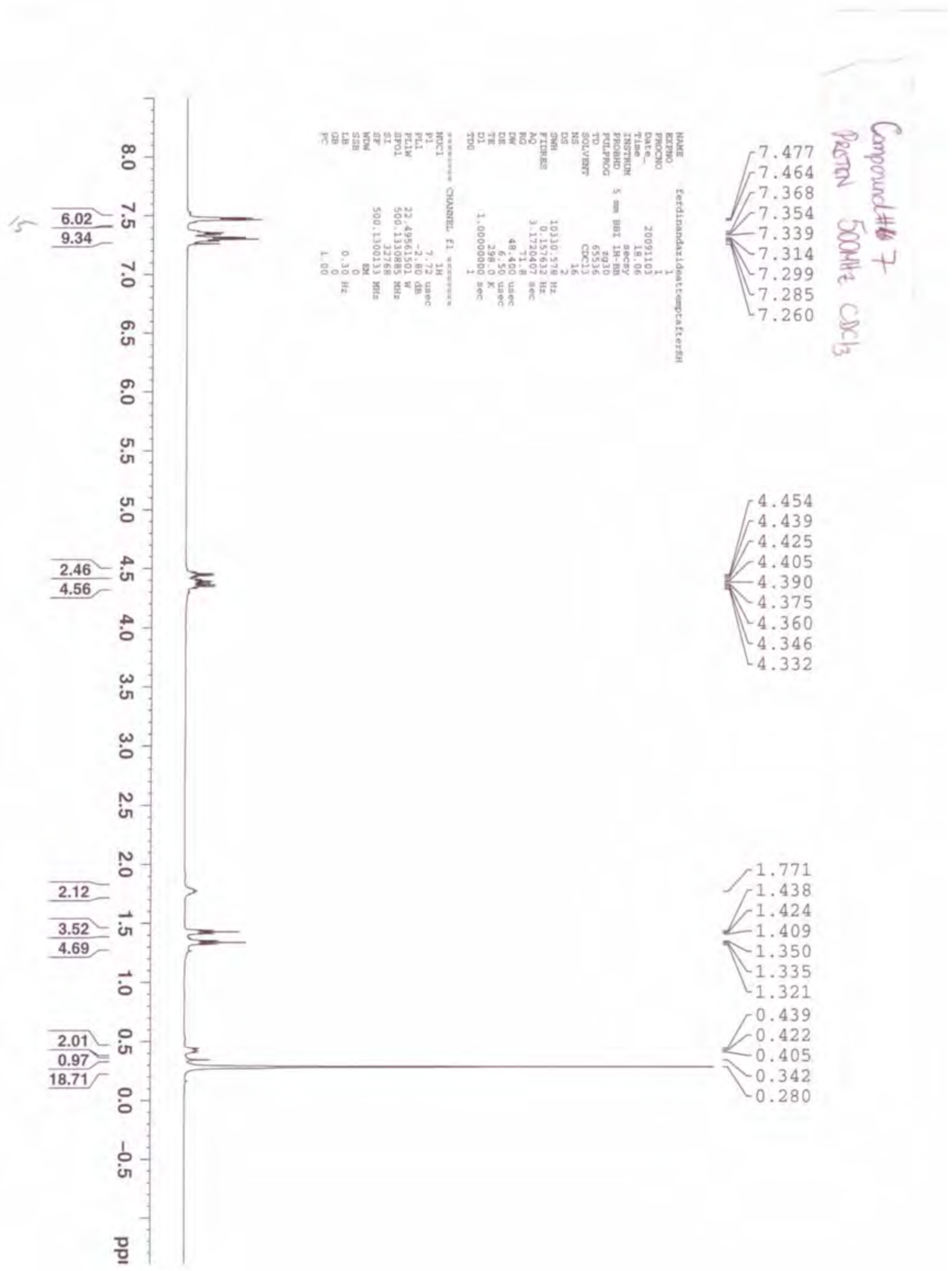
Compound **6** Spectra:

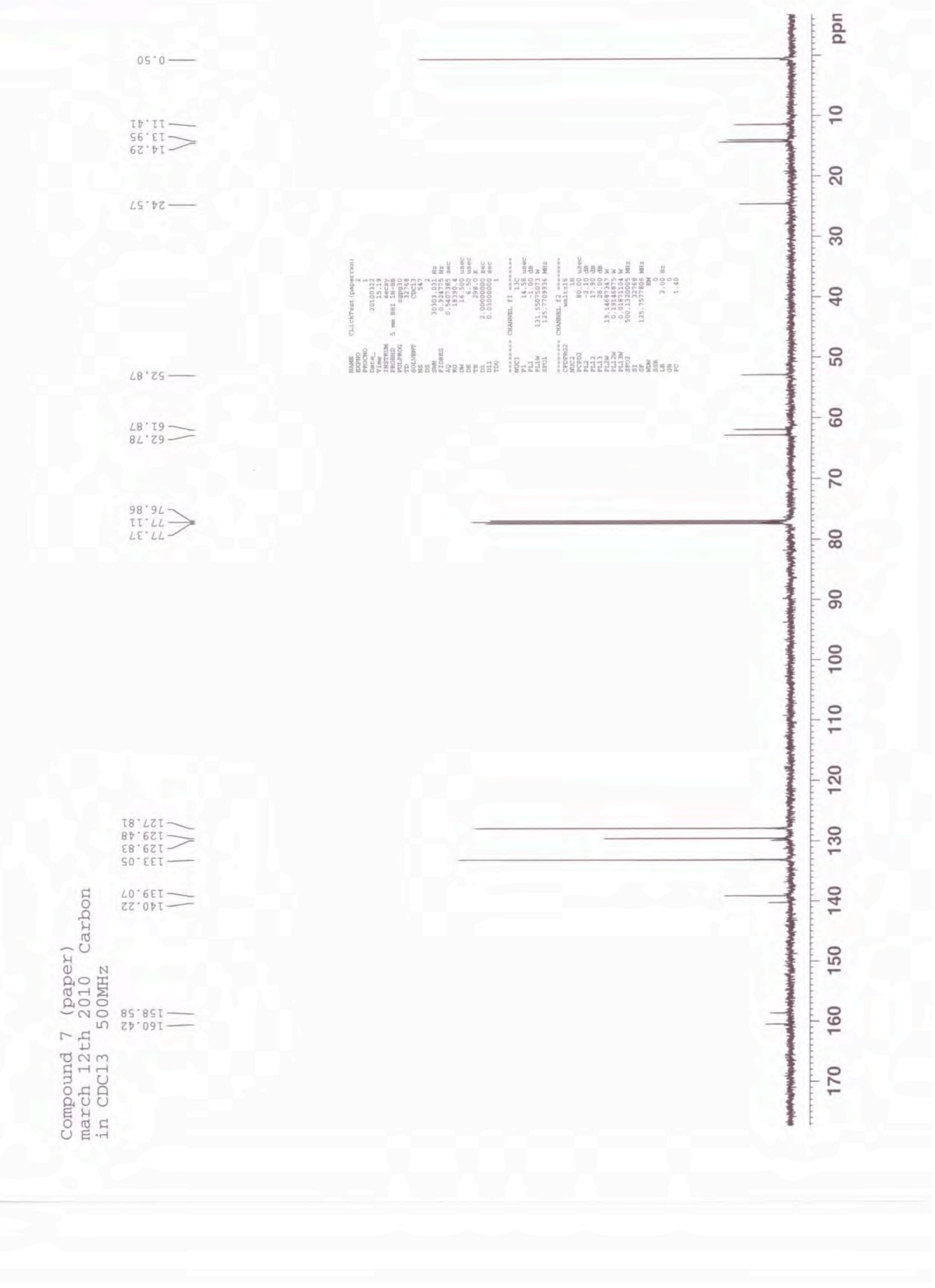



chloropropyltri(phenyldimethylsilane)+sodiumAzide
cdcl3 august 24th 2009 carbon 13
500MHz
139.29
133.14
129.51
127.86
77.40
77.15
76.89
53.95
23.09
11.63
0.62
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

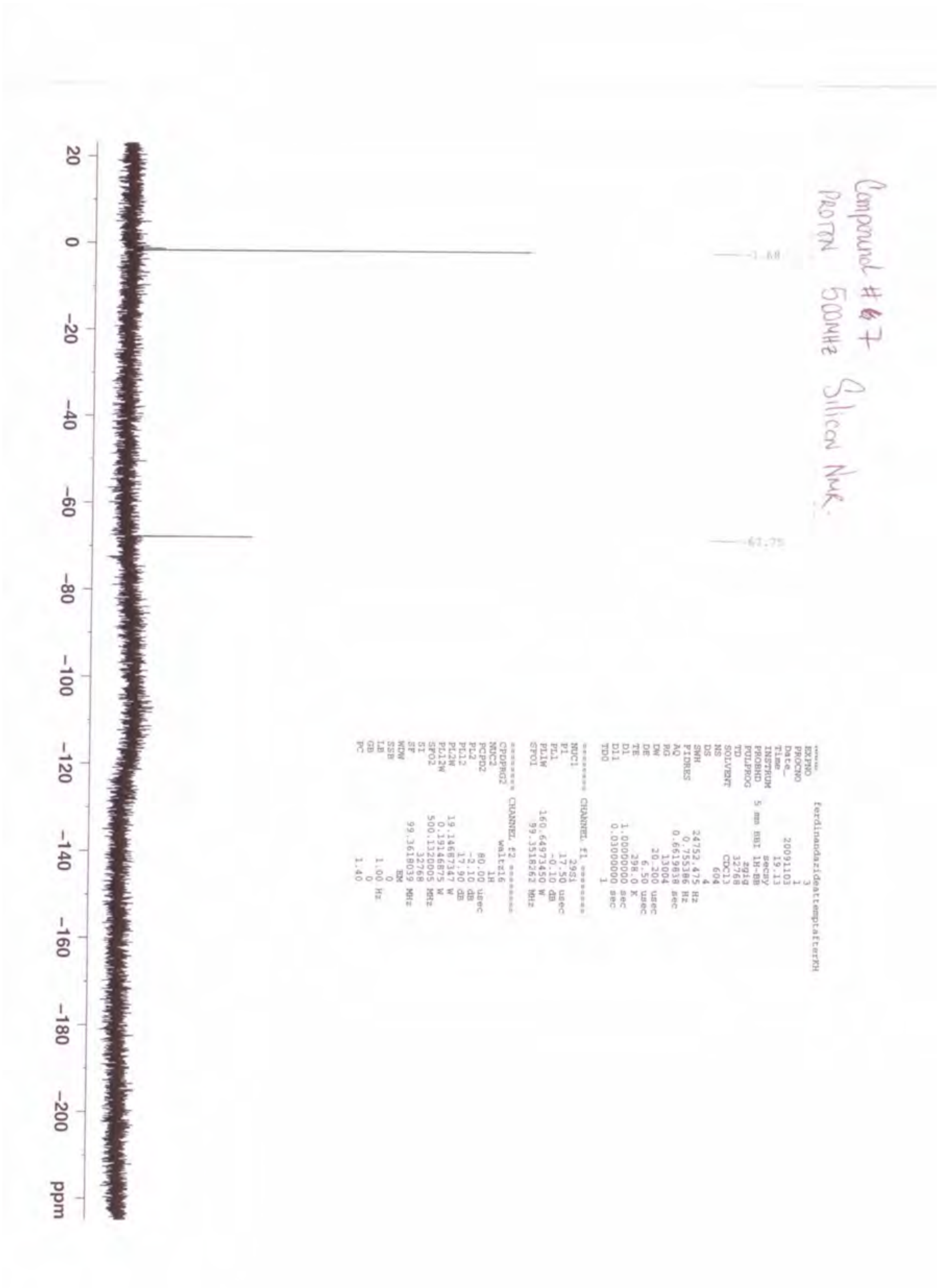

chloropropyltris(phenyldimethyl)siloxane+sodiumazide
cdcl3 august 25th 2009 silicon NMR
500MHz.
-1.9
-2.0
ppm
-66.9
ppm
20
0
−20
−40
−60
−80
−100
−120
−140
−160
−180
−200
ppm

Compound **7** Spectra**:**





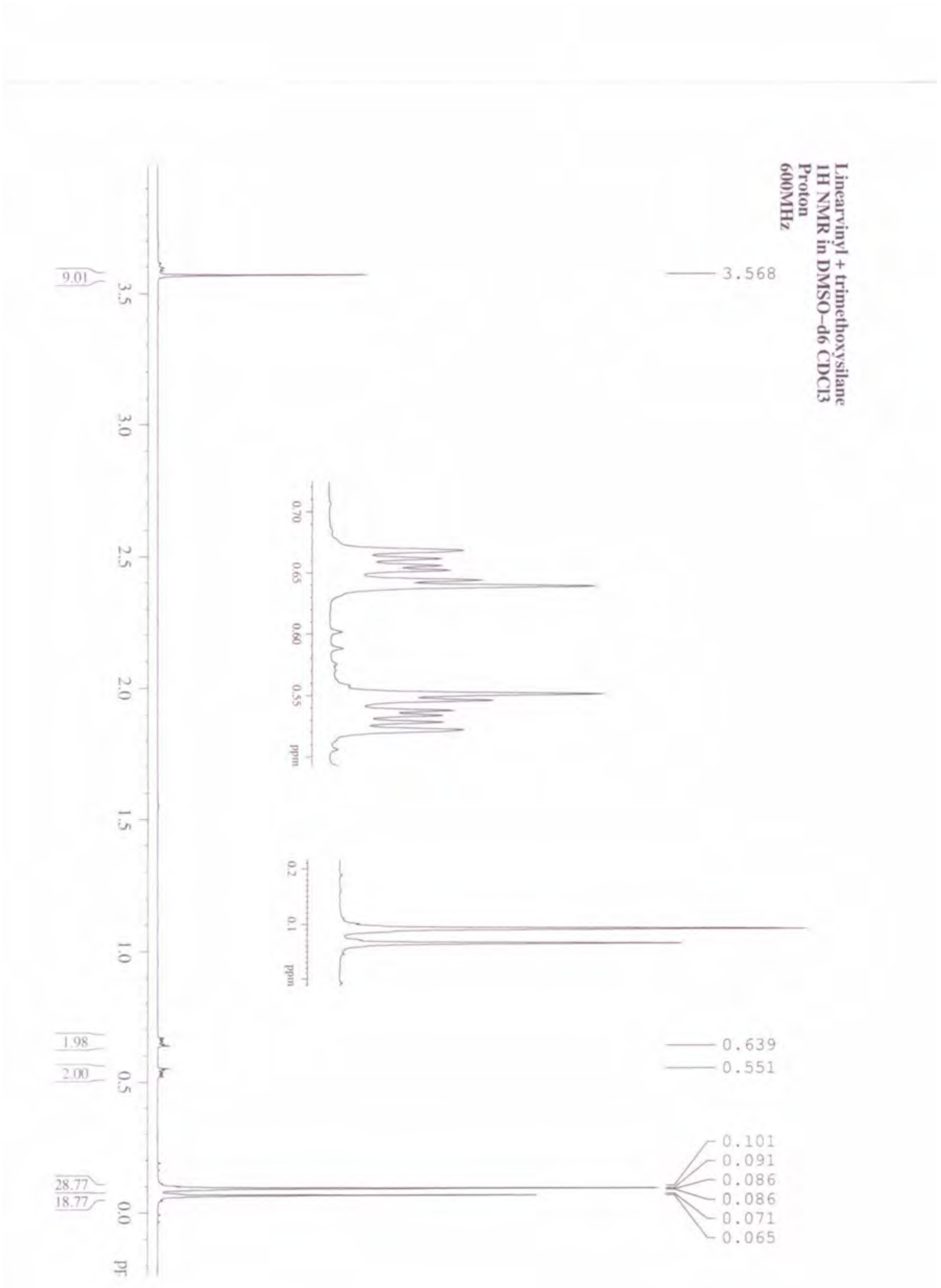
```
NAME        ClickTest(paperxn)
EXPNO                2
PROCNO               1
Date_         20100322
Time             15.19
INSTRUM          spect
PROBHD   5 mm BBI 1H-BB
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 547
DS                   2
SWH          30303.031 Hz
FIDRES        0.924775 Hz
AQ           0.5407385 sec
RG             18390.4
DW              16.500 usec
DE                6.50 usec
TE               298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1               14.58 usec
PL1              -1.00 dB
PL1W      131.55075073 W
SFO1       125.7709936 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.10 dB
PL12             17.90 dB
PL13             28.00 dB
PL2W       19.14687347 W
PL12W       0.19146875 W
PL13W       0.01871104 W
SFO2       500.1320005 MHz
SI               32768
SF         125.7577806 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.40
```

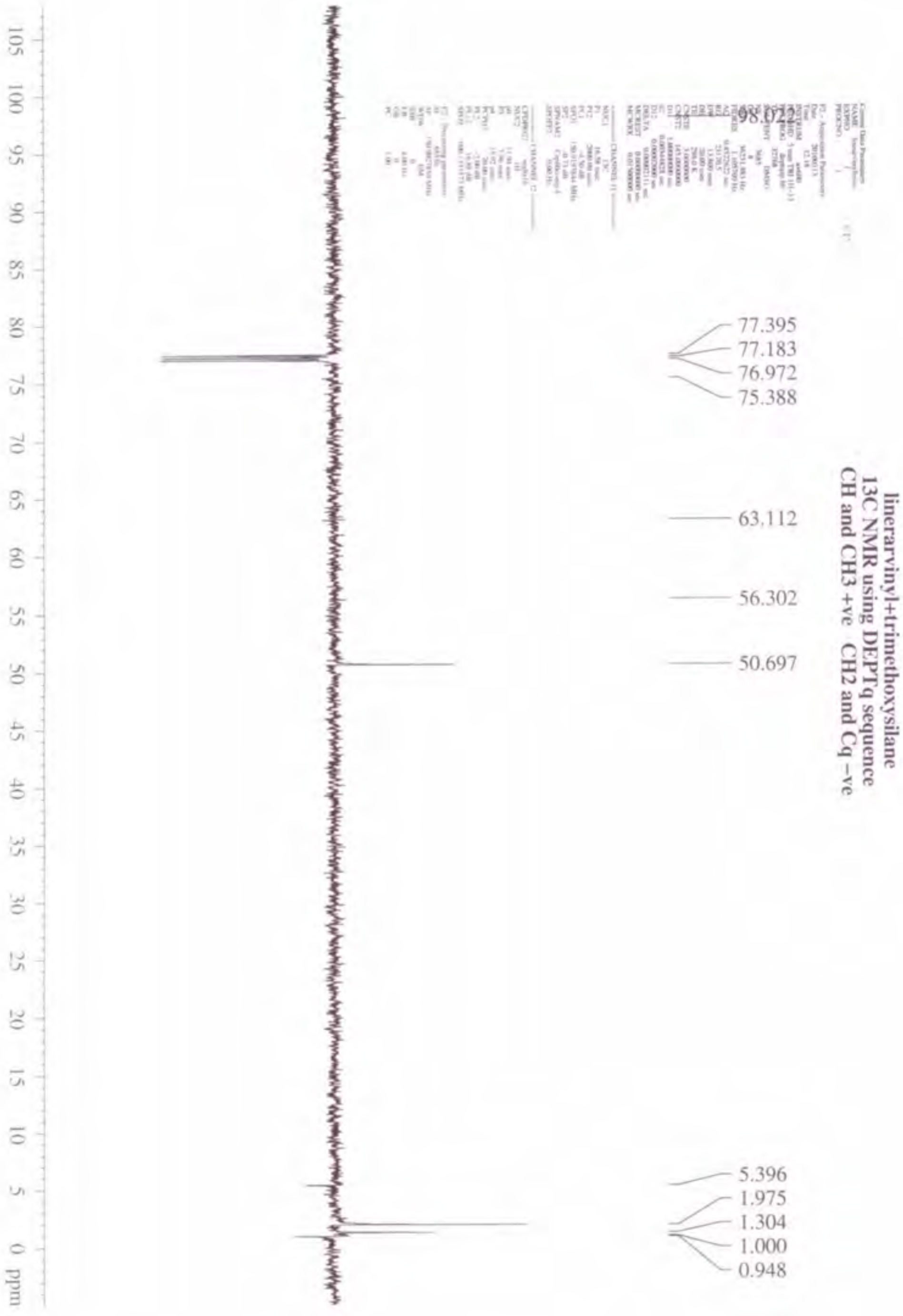


Compound #67
PROTON 500MHz Silicon NMR.

```
          ferdinandazideattemptafterKH
EXPNO                    3
PROCNO                   1
Date_             20091103
Time                 19.13
INSTRUM              secsy
PROBHD     5 mm BBI 1H-BB
PULPROG               zgig
TD                   32768
SOLVENT              CDCl3
NS                     604
DS                       4
SWH              24752.475 Hz
FIDRES            0.755386 Hz
AQ               0.6619838 sec
RG                   13004
DW                  20.200 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  29Si
P1                   17.50 usec
PL1                  -0.10 dB
PL1W          160.64973450 W
SFO1           99.3518262 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 17.90 dB
PL2W           19.14687347 W
PL12W           0.19146875 W
SFO2          500.1320005 MHz
SI                   32768
SF             99.3618039 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Compound **8** Spectra:


Linearvinyl + trimethoxysilane
1H NMR in DMSO–d6 CDCl3
Proton
600MHz
3.568
0.639
0.551
0.101
0.091
0.086
0.086
0.071
0.065
9.01
1.98
2.00
28.77
18.77
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
PF
0.70
0.65
0.60
0.55
ppm
0.2
0.1
ppm


linearvinyl+trimethoxysilane
13C NMR using DEPTq sequence
CH and CH3 +ve CH2 and Cq −ve
98.022
77.395
77.183
76.972
75.388
63.112
56.302
50.697
5.396
1.975
1.304
1.000
0.948
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0 ppm