

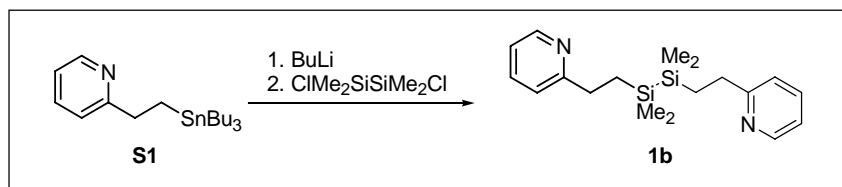
Generation of Pyridyl Coordinated Organosilicon Cation Pool
by Oxidative Si-Si Bond Dissociation

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Jun-ichi Yoshida*

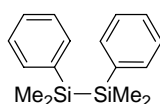
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General. ^1H and ^{13}C NMR spectra were recorded on Varian MERCURY plus-400 (^1H 400 MHz, ^{13}C 100 MHz), JEOL ECA-600P (^1H 600 MHz, ^{13}C 150 MHz) using the residual proton (CDCl_2 : 5.32 ppm, CDCl_3 : 7.26 ppm) or carbon (CD_2Cl_2 : 53.80 ppm, CDCl_3 : 77.00 ppm) as an internal standard. ^{29}Si NMR spectra were recorded on Varian Gemini 2000 (60 MHz), JEOL JNM-A400 (80 MHz), JEOL JNM-A500 (100 MHz) using Me_4Si as an internal standard. EI mass spectra were recorded on JMS-SX102A spectrometer. FAB mass spectra were recorded on JMS-HX110A spectrometer. CSI mass spectra were recorded on JMS-T100CSK spectrometer. Unless otherwise noted, all materials, dried diethyl ether and tetrahydrofuran were obtained from commercial suppliers and used without further purification. Dichloromethane was washed with water, distilled from P_2O_5 , redistilled from dried K_2CO_3 to remove a trace amount of acid, and stored over molecular sieves 4A. Starting materials, such as tributyl(2-pyridylethyl)stannane **S1**¹ and **1c**² were prepared according to the reported procedure. Rotating-disk electrode voltammetry was carried out using BAS 100B and Nikko Keisoku RRDE-1 rotating disk electrode with Nikko Keisoku SC-5 controller. Measurements were carried out in 0.1 M $\text{Bu}_4\text{NClO}_4/\text{CH}_3\text{CN}$ using a glassy carbon disk working electrode, a platinum wire counter electrode, and an SCE reference electrode with sweep rate of 10 mV/s at 5000 r.p.m.

Preparation of 1,1,2,2-Tetramethyl-1,2-bis(2-pyridylethyl)disilane (1b).

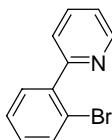
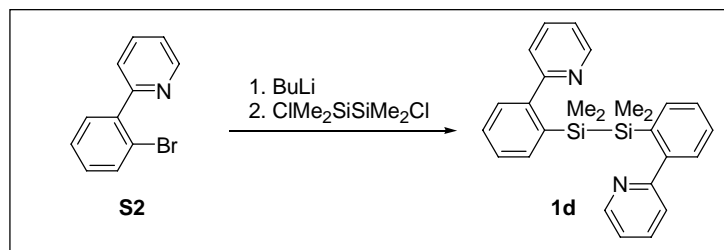


To a solution of tributyl(2-pyridylethyl)stannane **S1**¹ (15.9 g, 40.1 mmol) in THF (100 mL) was added dropwise a hexane solution of *n*-BuLi (44 mmol) at -78°C under argon. Additional stirring (-78°C , 30 min) of the mixture afforded a THF solution of 2-pyridylethyl lithium. To this solution, was added 1,2-dichloro-1,1,2,2-tetramethyldisilane (4.32 g, 23.1 mmol) dropwise and the mixture was stirred for 1 h at -78°C and an additional 1 h at room temperature. The resulting mixture was treated with saturated aq NaHCO_3 (40 mL). After extracted with Et_2O (40 mL \times 3), organic phase was dried over MgSO_4 . The solvent was removed under reduced pressure and the crude product was purified with flash chromatography (hexane / ethyl acetate 10:1, 1:1, 2:1 and 1:2) to give the title compound in 42% yield (2.79 g). ^1H NMR (400 MHz, CDCl_3) δ 0.09 (s, 12H), 1.02-1.06 (m, 4H), 2.76-2.80 (m, 4H), 7.02 (dd, $J = 7.6, 4.8$ Hz, 2H), 7.12 (d, $J = 7.6$ Hz, 2H), 7.52 (dd, $J = 7.6, 0.8$ Hz, 2H), 8.46 (dd, $J = 4.8, 0.8$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ -4.0, 15.4, 33.2, 120.6, 121.7, 136.1, 148.8, 164.1. ^{29}Si NMR (100 MHz, CDCl_3) δ -17.1. HRMS (FAB) m/z calcd for $\text{C}_{18}\text{H}_{29}\text{N}_2\text{Si}_2$ (M+H): 329.1869, found 329.1868.

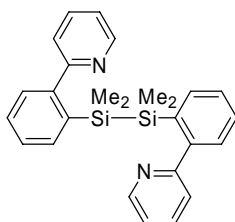


1,1,2,2-Tetramethyl-1,2-diphenyldisilane (1c).² 80 % yield from PhMgBr and 1,2-dichlorotetramethyldisilane. ^1H NMR (400 MHz, CDCl_3) δ 0.37 (s, 12H), 7.32-7.35 (m, 6H), 7.40-7.43 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ -3.7, 127.6, 128.3, 133.7, 138.8. ^{29}Si NMR (60 MHz, CDCl_3) δ -21.8.

Preparation of 1,1,2,2-Tetramethyl-1,2-bis[*o*-(2-pyridyl)phenyl]disilane (**1d**).

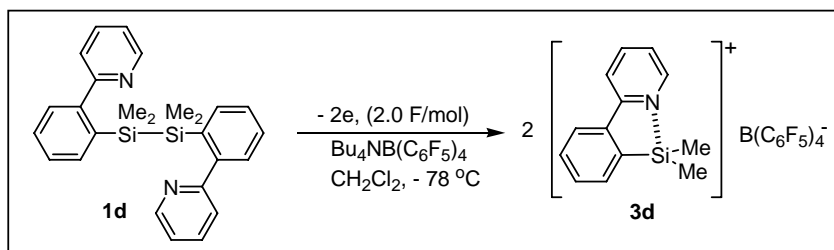


***o*-(2-Pyridyl)phenylbromide (S2).** A solution of 2-bromopyridine (2.83 g, 17.9 mmol), Pd(PPh₃)₄ (564 mg, 0.48 mmol), *o*-(2-bromo)phenylboronic acid (2.71 g, 12 mmol), K₂CO₃ (8.3 g, 60 mmol) and H₂O (6.5 g, 300 mmol) in DME (15 mL) was stirred at 100 °C (reflux) for 24 h under argon. Then, the mixture was cooled to room temperature and treated with saturated aq NaHCO₃ (10 mL). After extracted with Et₂O (15 mL × 3), organic phase was dried over MgSO₄. The solvent was removed under reduced pressure and the crude product was purified by distillation (0.067 mmHg, 123 °C) to give the title compound in 62% (1.95 g) yield. ¹H NMR (400 MHz, CDCl₃) δ 7.23-7.31 (m, 2H), 7.40 (td, *J* = 7.6, 1.2 Hz, 1H), 7.53 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.60 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.67 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.76 (td, *J* = 7.6, 2.0 Hz, 1H), 8.71 (dm, *J* = 4.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 121.6, 122.2, 124.5, 127.3, 129.5, 131.2, 133.0, 135.5, 141.0, 149.1, 158.0. HRMS (EI) *m/z* calcd for C₁₁H₈NBr (M⁺): 232.9840, found 232.9842.

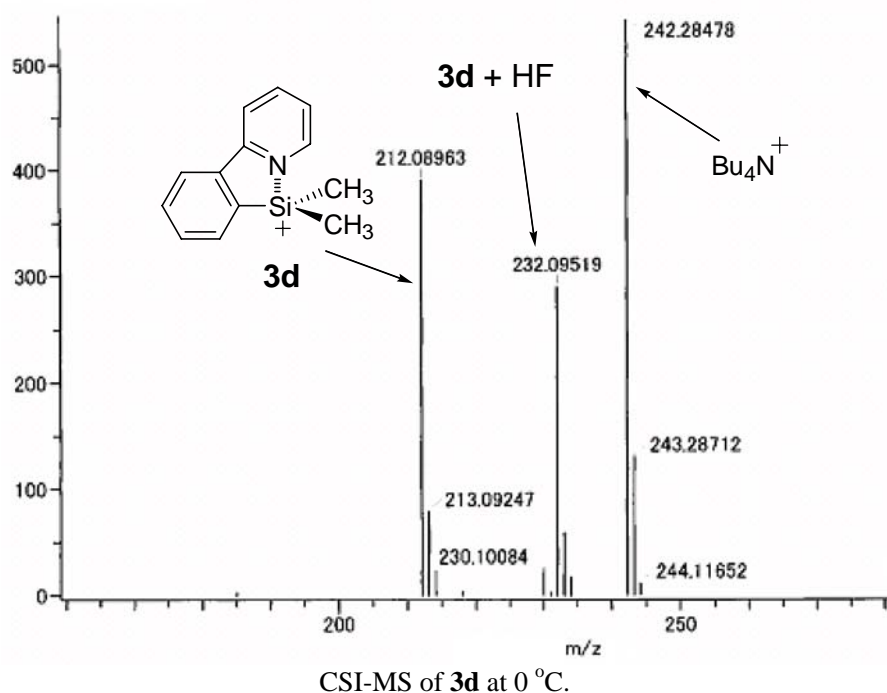


1,1,2,2-Tetramethyl-1,2-bis[*o*-(2-pyridyl)phenyl]disilane (1d**).** To a solution of *o*-(2-pyridyl)phenylbromide **S2** (1.32 g, 5.6 mmol) in Et₂O (10 mL) was added dropwise a *n*-hexane solution of *n*-BuLi (5.7 mmol) at -78 °C under argon. Additional stirring (-78 °C, 90 min) of the mixture afforded a Et₂O solution of *o*-(2-pyridyl)phenyl lithium. To this solution, was added 1,2-dichloro-1,1,2,2-tetramethyldisilane (505.5 mg, 2.7 mmol) dropwise and stirred at -78 °C to room temperature for 20 h. The resulting mixture was treated with saturated aq NaHCO₃ (10 mL). After extracted with Et₂O (10 mL × 3), organic phase was dried over MgSO₄. The solvent was removed under reduced pressure and the crude product was purified with flash chromatography (hexane / ethyl acetate 5:1) to give the title compound in 85% yield (974.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 0.07 (s, 12H), 7.08 (ddd, *J* = 6.4, 4.8, 1.6 Hz, 2H), 7.39 (td, *J* = 7.2, 1.6 Hz, 2H), 7.46 (td, *J* = 7.2, 1.6 Hz, 2H), 7.60-7.66 (m, 6H), 7.71 (dd, *J* = 7.8, 1.6 Hz, 2H), 8.18 (dt, *J* = 4.8, 0.8 Hz, 2H). ¹H NMR (600 MHz, CH₂Cl₂ containing 10% CD₂Cl₂, 0 °C) δ -0.05 (s, 12H), 7.06 (ddd, *J* = 7.5, 6.3, 1.2 Hz, 2H), 7.34 (td, *J* = 7.5, 1.2 Hz, 2H), 7.42 (td, *J* = 7.5, 1.7 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.61-7.64 (m, 6H), 8.08 (d, *J* = 5.2 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 0.2, 121.3, 122.0, 127.3, 127.8, 128.0, 135.9, 136.2, 141.1, 145.2, 148.0, 160.1. ²⁹Si NMR (80 MHz, CDCl₃) δ -21.0. HRMS (FAB) *m/z* calcd for C₂₆H₂₉N₂Si₂ (M+H): 425.1869, found 425.1874.

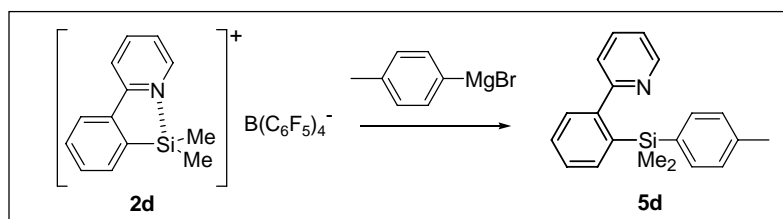
Preparation of Dimethyl[*o*-(2-pyridyl)phenyl]silylium ion (**3d**).



The anodic oxidation was carried out in an H-type divided cell equipped with a 4G glass filter (separator), a carbon felt anode (Nippon Carbon JF-20-P7, ca. 160 mg, dried at 250 °C/1 mmHg for 2.5 h before use) and a platinum plate cathode (10 mm x 10 mm). In the anodic chamber was placed a solution of **1d** (84.9 mg, 0.2 mmol) in 0.1 M Bu₄NB(C₆F₅)₄/CH₂Cl₂ (10% CD₂Cl₂) (4 mL). In the cathodic chamber was placed 0.1 M Bu₄NB(C₆F₅)₄/CH₂Cl₂ (10% CD₂Cl₂) (4 mL). The constant current electrolysis (5 mA) was carried out at 0 °C with magnetic stirring until 2 F/mol of electricity was consumed. The resulting solution in the anodic chamber was analyzed by NMR. ¹H NMR (600 MHz, CH₂Cl₂ containing 10% CD₂Cl₂, 0 °C) δ 0.87 (s, 6H), 7.73 (ddd, *J* = 7.5, 6.6, 0.6 Hz, 1H), 7.78 (ddd, *J* = 7.8, 7.5, 0.6 Hz, 1H), 7.83 (ddd, *J* = 6.6, 6.0, 0.6 Hz, 1H), 7.92 (d, *J* = 6.6 Hz, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 8.36 (d, *J* = 7.8 Hz, 1H), 8.50 (ddd, *J* = 7.8, 6.0, 0.6 Hz, 1H), 8.52 (d, *J* = 6.0 Hz, 1H). ¹³C NMR (150 MHz, CD₂Cl₂, 0 °C) δ -2.3, 121.8, 125.1, 126.4, 132.1, 133.6, 133.9, 134.1, 137.1, 143.6, 148.1, 157.3. ²⁹Si NMR (80 MHz, CD₂Cl₂) δ 37.7. HRMS (CSI) *m/z* calcd for C₁₃H₁₄NSi (M⁺): 212.08955, found 212.08963.



Preparation of Dimethyl[*o*-(2-pyridyl)phenyl]-*p*-tolylsilane (**5d**).

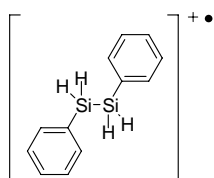


The electrochemical oxidation of **1d** (61.1 mg, 0.14 mmol) was carried out in 0.1 M Bu₄NB(C₆F₅)₄/CH₂Cl₂ as described above. A Et₂O solution of *p*-tolylmagnesium bromide (0.375 mmol) was added to the anodic chamber and the mixture was stirred at 0 °C for 1 h. The solution in the anodic chamber was treated with saturated aq

NaHCO₃ (4 mL). After extracted with CHCl₃ (4 mL × 3), organic phase was dried over MgSO₄. Removal of the solvent under reduced pressure and the residue was quickly filtered through a short column (2 x 3 cm) of silica gel to remove Bu₄NB(C₆F₅)₄. The silica gel was washed with diethyl ether (50 mL). The solvent was removed from the combined filtrate under reduced pressure. ¹H NMR analysis of the crude product using 1,1,2,2-tetrachloroethane as internal standard indicated that the title compound was formed in 90% yield. The product was purified with flash chromatography (hexane / ethyl acetate 10:1) to give the title compound in 68% (58.7 mg) yield. ¹H NMR (400 MHz, CDCl₃) δ 0.32 (s, 6H), 2.33 (s, 3H), 7.09 (d, *J* = 7.6 Hz, 2H), 7.16 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.32-7.38 (m, 4H), 7.44 (td, *J* = 7.6, 1.6 Hz, 1H), 7.50 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.59 (td, *J* = 7.6, 1.6 Hz, 1H), 7.62 (dd, *J* = 7.6, 1.6 Hz, 1H), 8.51 (dd, *J* = 4.8, 1.2 Hz, 1H). ¹³C NMR (150 MHz, CDCl₃) δ -0.4, 21.4, 121.9, 122.9, 127.4, 128.3, 128.6, 129.0, 133.8, 136.2, 136.7, 137.0, 137.5, 138.0, 147.0, 148.2, 160.6. HRMS (FAB) *m/z* calcd for C₂₀H₂₅N₂Si₁ (M+H): 304.1522, found 304.1524.

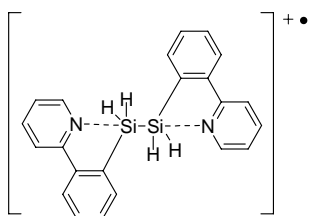
DFT Calculations.

The DFT calculations were carried out at B3LYP/LANL2DZ level using the Gaussian 2003W, Revision-B.05.⁷ Geometries were fully optimized. The local minima were verified to have no negative eigenvalue by the vibration analysis. Cartesian coordinates and energies of computationally characterized species are as follows:



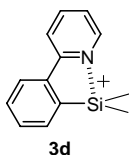
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4	6	0	1.886531	1.337262	-0.941782
5	6	0	-1.692778	-1.864633	2.273778
6	6	0	-2.313688	-3.058227	2.656262
7	6	0	-3.113040	-3.768108	1.726085
8	6	0	-3.292910	-3.272614	0.410696
9	6	0	-2.673616	-2.079375	0.024521
10	6	0	2.680729	2.081401	-0.010519
11	6	0	3.299812	3.274707	-0.396819
12	6	0	3.120042	3.769929	-1.712328
13	6	0	2.321003	3.059701	-2.642511
14	6	0	1.700301	1.866047	-2.259898
15	1	0	-1.819359	0.956370	-0.647582
16	1	0	-0.831280	1.172546	1.617980
17	1	0	0.839148	-1.171145	-1.603631
18	1	0	1.826989	-0.954356	0.661994
19	1	0	-1.082498	-1.327845	2.996394
20	1	0	-2.185911	-3.441015	3.664436
21	1	0	-3.585681	-4.701349	2.021297
22	1	0	-3.912239	-3.818965	-0.294428
23	1	0	-2.819637	-1.708164	-0.987230
24	1	0	2.826677	1.710399	1.001319
25	1	0	3.918909	3.821329	0.308300
26	1	0	3.592527	4.703224	-2.007620
27	1	0	2.193309	3.442274	-3.650777
28	1	0	1.090257	1.328991	-2.982516

Zero-point correction=	0.216454 (Hartree/Particle)
Thermal correction to Energy=	0.230809
Thermal correction to Enthalpy=	0.231753
Thermal correction to Gibbs Free Energy=	0.171505
Sum of electronic and zero-point Energies=	-472.886084
Sum of electronic and thermal Energies=	-472.871728
Sum of electronic and thermal Enthalpies=	-472.870784
Sum of electronic and thermal Free Energies=	-472.931033



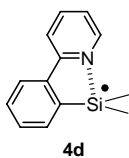
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4	6	0	-1.270497	-2.107509	0.586037
5	6	0	0.198774	3.028793	-0.485673
6	6	0	0.244325	4.409975	-0.764854
7	6	0	1.482306	5.059717	-0.927531
8	6	0	2.677295	4.331285	-0.811687
9	6	0	2.629093	2.950143	-0.532443
10	6	0	3.831088	2.104672	-0.393174
11	6	0	5.174385	2.515320	-0.510430
12	6	0	6.198855	1.571635	-0.346801
13	6	0	5.876094	0.226158	-0.067598
14	6	0	4.527336	-0.126844	0.038197
15	7	0	3.543536	0.793047	-0.121697
16	6	0	-2.517287	-2.778531	0.753094
17	6	0	-2.565489	-4.159638	1.032515
18	6	0	-1.370495	-4.888019	1.148643
19	6	0	-0.132510	-4.238258	0.986085
20	6	0	-0.086961	-2.857110	0.706732
21	6	0	-3.719284	-1.933103	0.613591
22	6	0	-5.062590	-2.343787	0.730645
23	6	0	-6.087058	-1.400133	0.566831
24	6	0	-5.764291	-0.054656	0.287637
25	6	0	-4.415525	0.298379	0.182037
26	7	0	-3.431726	-0.621483	0.342103
27	1	0	1.612165	-0.595367	-1.081475
28	1	0	1.675727	-0.096813	1.394517
29	1	0	-1.563894	0.268409	-1.173920
30	1	0	-1.500414	0.766957	1.302077
31	1	0	-0.768119	2.552506	-0.364890
32	1	0	-0.679296	4.975427	-0.854869
33	1	0	1.514498	6.124127	-1.142608
34	1	0	3.627823	4.841930	-0.938944
35	1	0	5.411910	3.550744	-0.725022
36	1	0	7.237696	1.875787	-0.434782
37	1	0	6.647163	-0.524659	0.063718
38	1	0	4.217030	-1.145129	0.251184
39	1	0	-3.516020	-4.670293	1.159709
40	1	0	-1.402691	-5.952399	1.363859
41	1	0	0.791118	-4.803663	1.076335
42	1	0	0.879939	-2.380807	0.586066
43	1	0	-5.300120	-3.379213	0.945217
44	1	0	-7.125906	-1.704312	0.654653
45	1	0	-6.535359	0.696138	0.156181
46	1	0	-4.105214	1.316660	-0.030960

Zero-point correction=	0.358910 (Hartree/Particle)
Thermal correction to Energy=	0.381025
Thermal correction to Enthalpy=	0.381969
Thermal correction to Gibbs Free Energy=	0.304229
Sum of electronic and zero-point Energies=	-966.915950
Sum of electronic and thermal Energies=	-966.893836
Sum of electronic and thermal Enthalpies=	-966.892892
Sum of electronic and thermal Free Energies=	-966.970631



Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
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3	6	0	0.317469	2.005770	-2.975299
4	6	0	0.217309	2.145601	-1.580555
5	6	0	0.104064	0.991354	-0.779149
6	6	0	0.090418	-0.308140	-1.368130
7	6	0	-0.007472	1.005604	0.691084
8	14	0	-0.069911	-1.631294	-0.046051
9	6	0	-0.021040	2.133923	1.532824
10	6	0	-0.134440	1.967063	2.920003
11	6	0	-0.234741	0.668852	3.469991
12	6	0	-0.218038	-0.425427	2.607247
13	7	0	-0.107313	-0.256338	1.256309
14	6	0	1.442324	-2.694580	0.283541
15	6	0	-1.703478	-2.552940	0.044016
16	1	0	0.183828	-1.406324	-3.240528
17	1	0	0.382798	0.629458	-4.644766
18	1	0	0.405114	2.888937	-3.601363
19	1	0	0.228583	3.138941	-1.140659
20	1	0	0.056268	3.126043	1.103978
21	1	0	-0.145238	2.835337	3.572012
22	1	0	-0.323358	0.512750	4.538774
23	1	0	-0.291760	-1.443485	2.974430
24	1	0	2.356712	-2.093306	0.337400
25	1	0	1.573060	-3.424744	-0.526376
26	1	0	1.344065	-3.264344	1.216265
27	1	0	-1.798443	-3.123119	0.976826
28	1	0	-1.775139	-3.273775	-0.781499
29	1	0	-2.558105	-1.872020	-0.036529

Zero-point correction= 0.239061 (Hartree/Particle)
 Thermal correction to Energy= 0.252719
 Thermal correction to Enthalpy= 0.253663
 Thermal correction to Gibbs Free Energy= 0.199003
 Sum of electronic and zero-point Energies= -561.976619
 Sum of electronic and thermal Energies= -561.962961
 Sum of electronic and thermal Enthalpies= -561.962017
 Sum of electronic and thermal Free Energies= -562.016678



Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
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3	6	0	0.318117	1.993586	-2.993413
4	6	0	0.219501	2.153943	-1.606264
5	6	0	0.104092	1.014230	-0.765986
6	6	0	0.090047	-0.298339	-1.356233
7	6	0	-0.005430	1.036997	0.682933
8	14	0	-0.070808	-1.589228	-0.009463
9	6	0	-0.020859	2.147594	1.540335

10	6	0	-0.133708	1.998418	2.931417
11	6	0	-0.235194	0.668850	3.477221
12	6	0	-0.220176	-0.421209	2.637314
13	7	0	-0.108114	-0.282790	1.250203
14	6	0	1.409691	-2.740062	0.248736
15	6	0	-1.670138	-2.601438	0.013750
16	1	0	0.181081	-1.421027	-3.211720
17	1	0	0.381995	0.593087	-4.656853
18	1	0	0.406248	2.871070	-3.630867
19	1	0	0.231761	3.153320	-1.177396
20	1	0	0.057006	3.140550	1.105551
21	1	0	-0.144559	2.864985	3.584862
22	1	0	-0.323911	0.513048	4.548095
23	1	0	-0.294799	-1.436478	3.016431
24	1	0	2.343449	-2.167975	0.299204
25	1	0	1.493562	-3.453883	-0.582608
26	1	0	1.311396	-3.322695	1.174838
27	1	0	-1.765544	-3.184351	0.939978
28	1	0	-1.690676	-3.310424	-0.825670
29	1	0	-2.545903	-1.947886	-0.073653

Zero-point correction=	0.235103 (Hartree/Particle)
Thermal correction to Energy=	0.249091
Thermal correction to Enthalpy=	0.250035
Thermal correction to Gibbs Free Energy=	0.194115
Sum of electronic and zero-point Energies=	-562.152616
Sum of electronic and thermal Energies=	-562.138629
Sum of electronic and thermal Enthalpies=	-562.137684
Sum of electronic and thermal Free Energies=	-562.193604

References

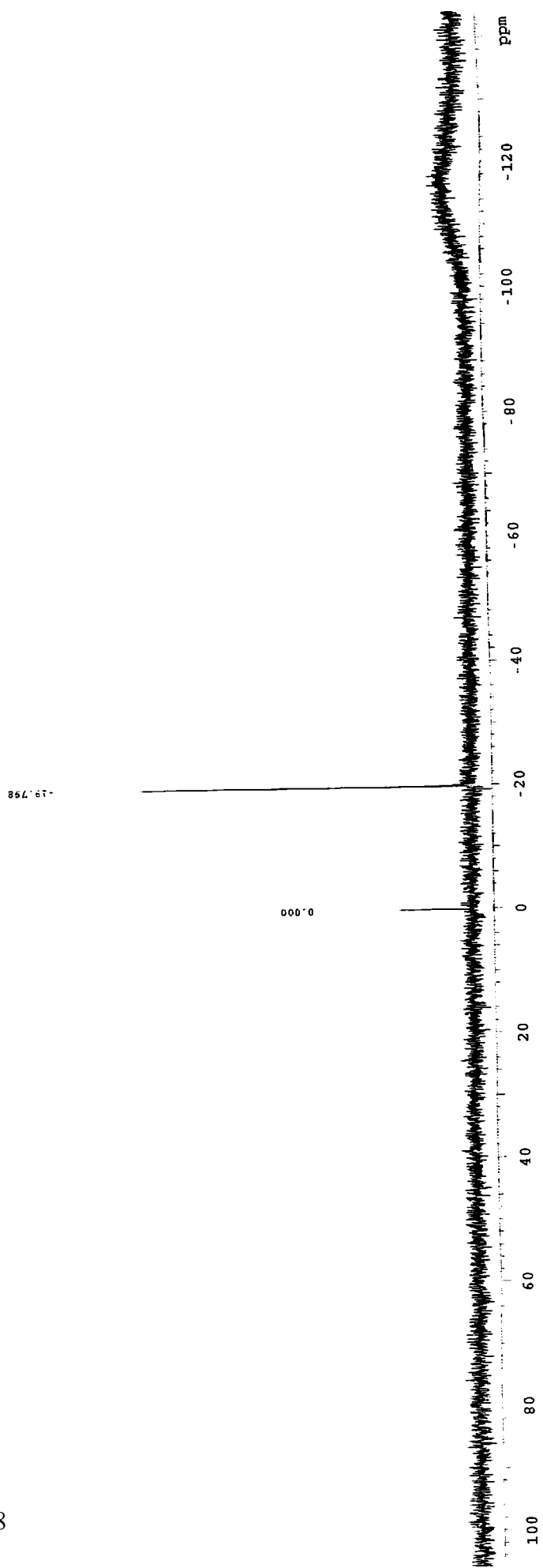
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^{29}Si NMR

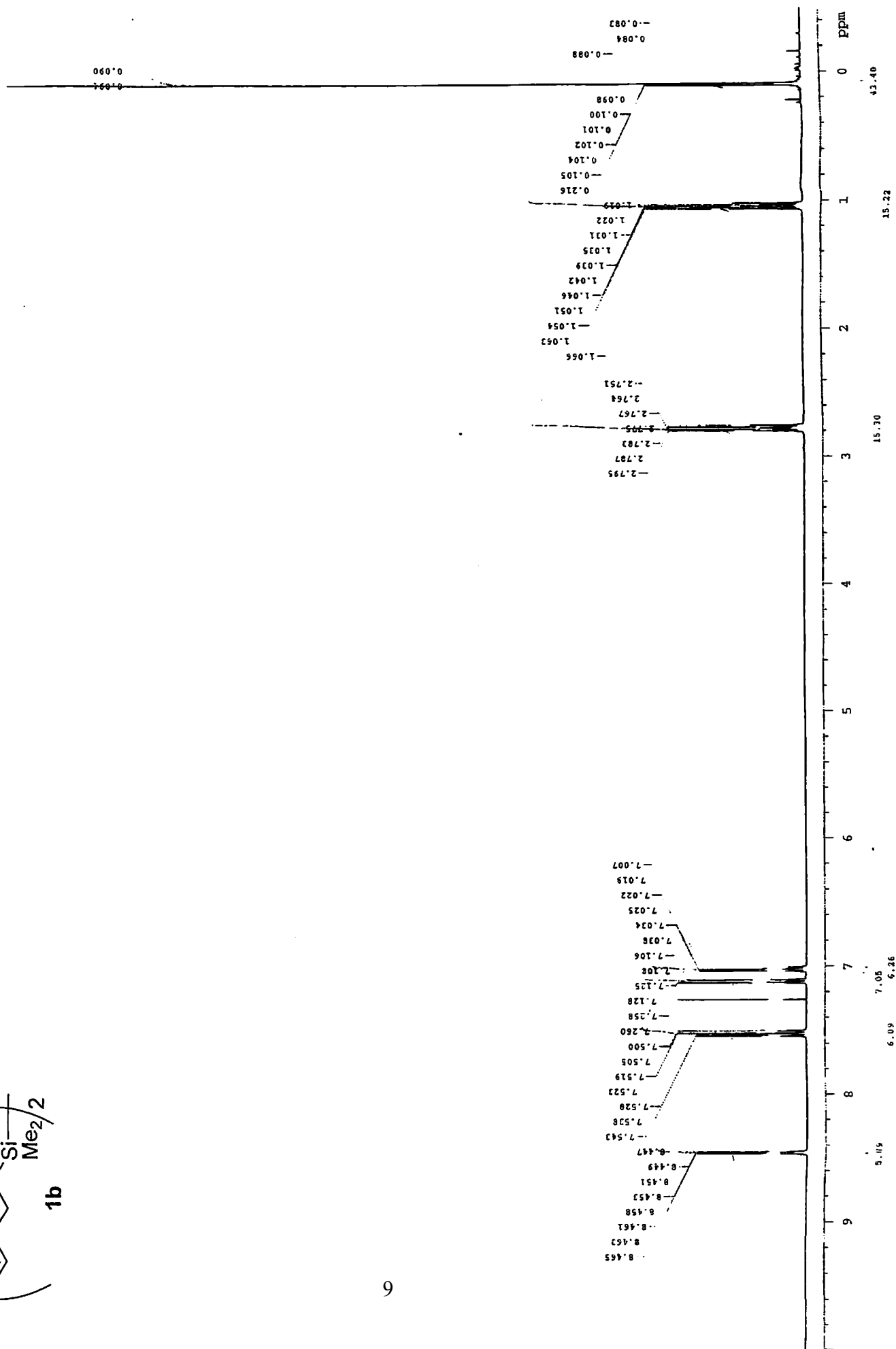
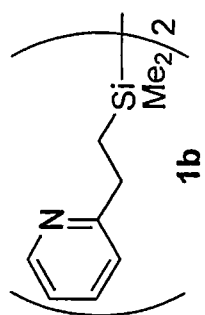
$\text{Me}_3\text{Si}-\text{SiMe}_3$

1a

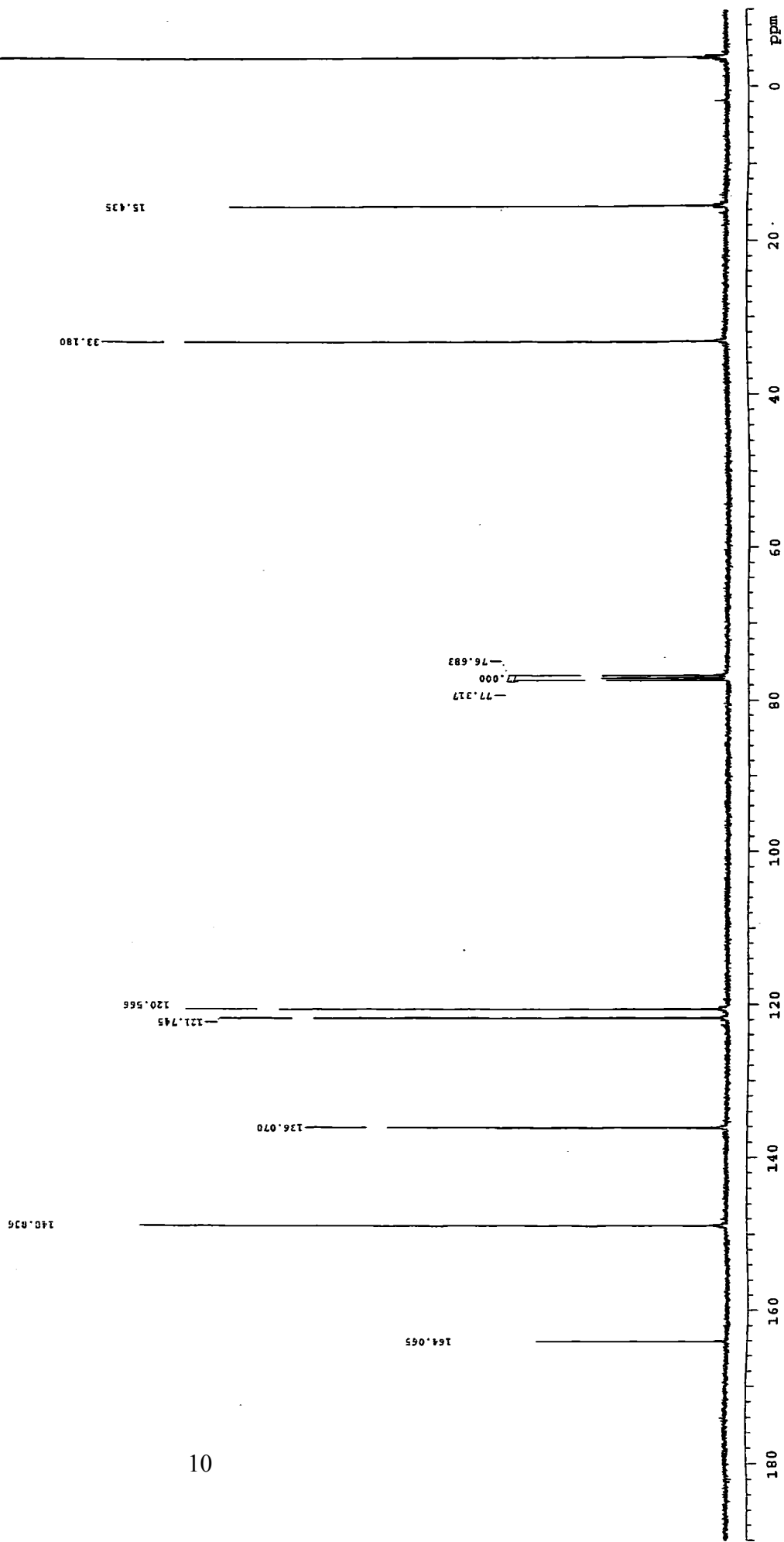
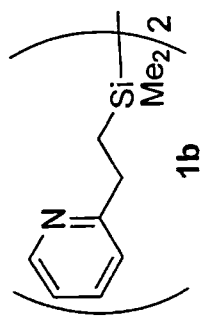
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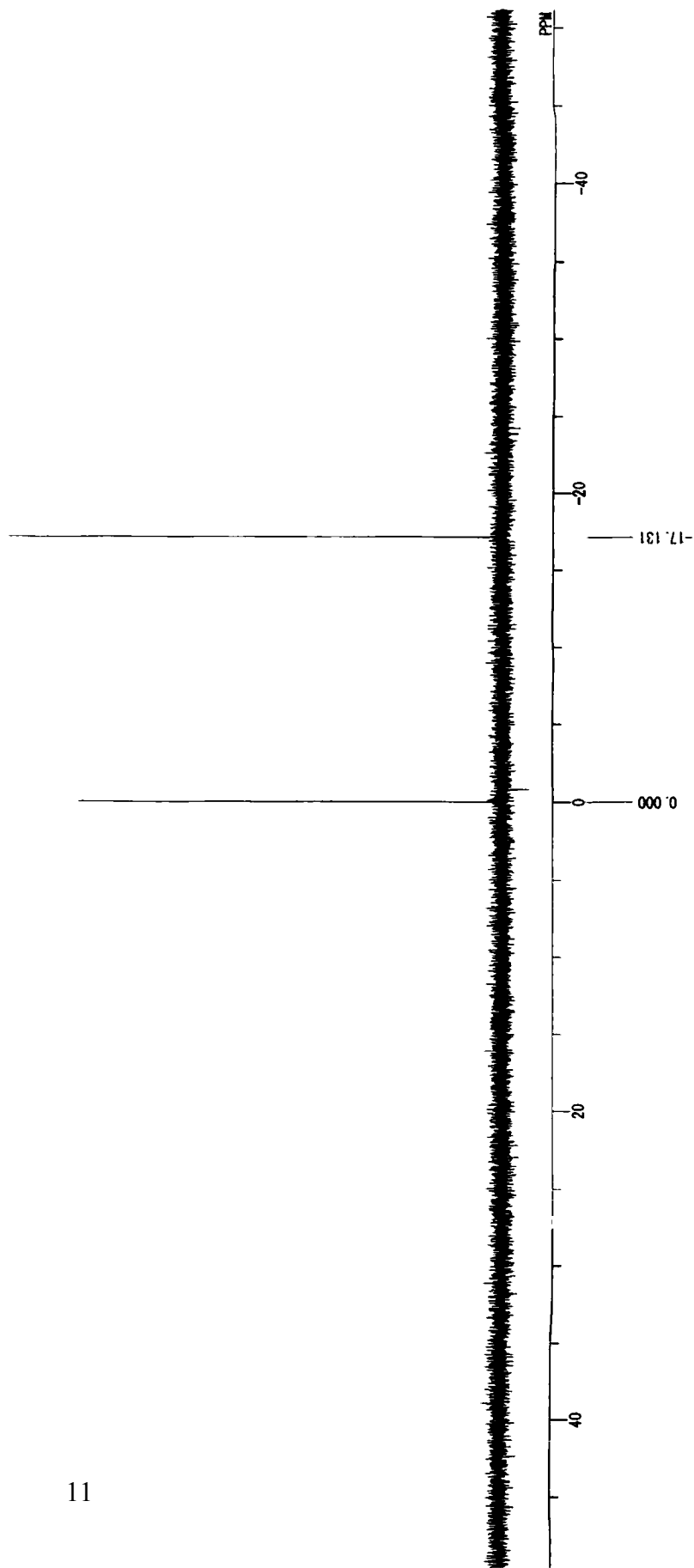
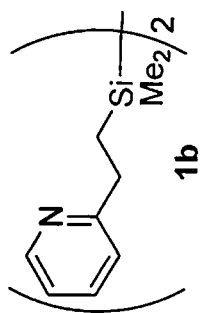
¹H NMR



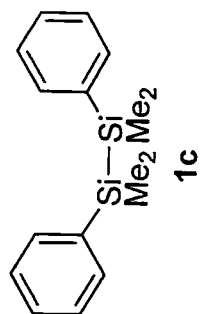
¹³C NMR



^{29}Si NMR

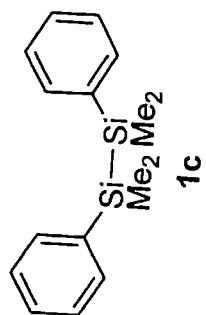


¹H NMR

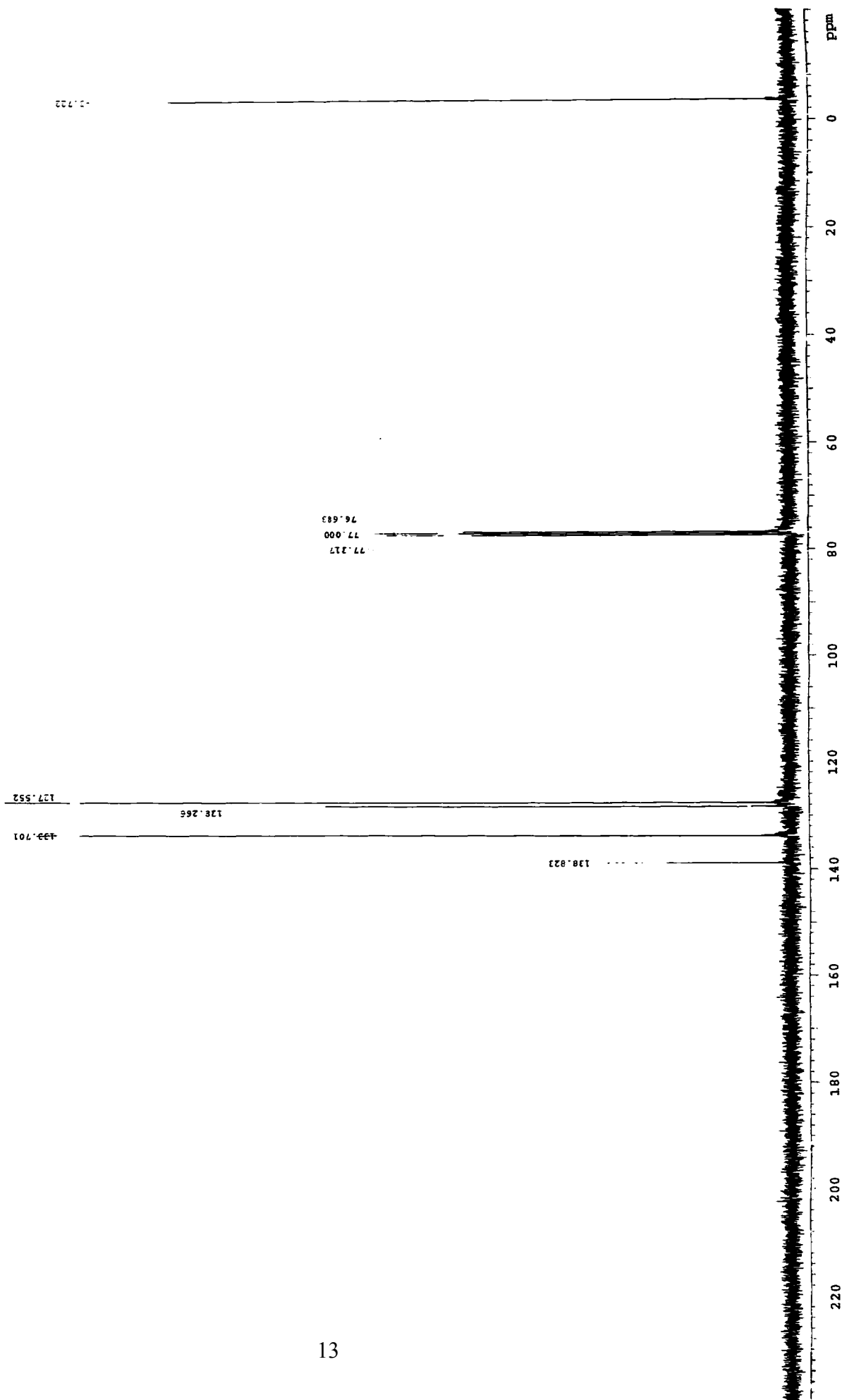


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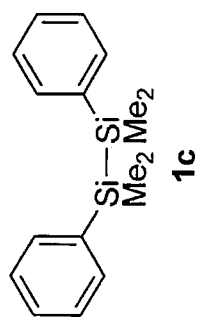
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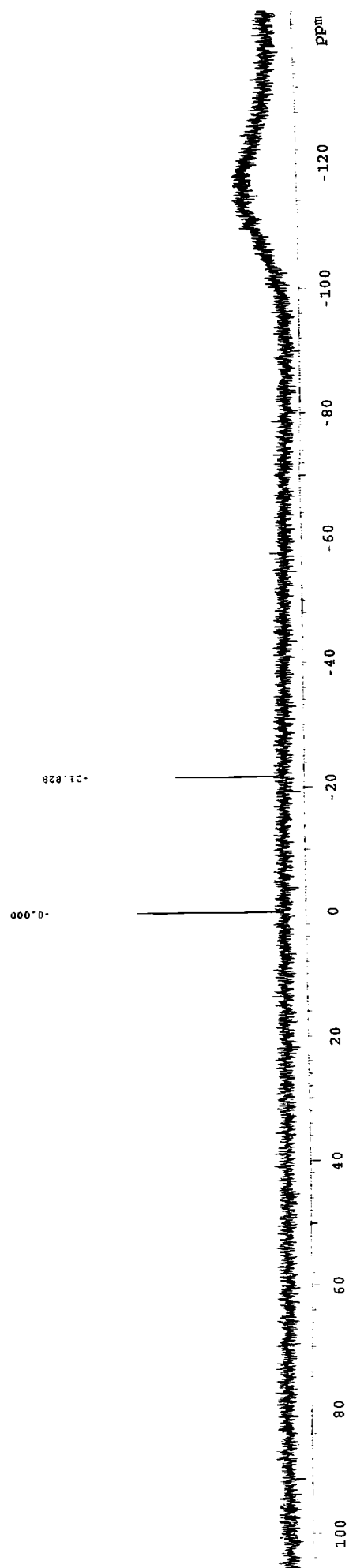
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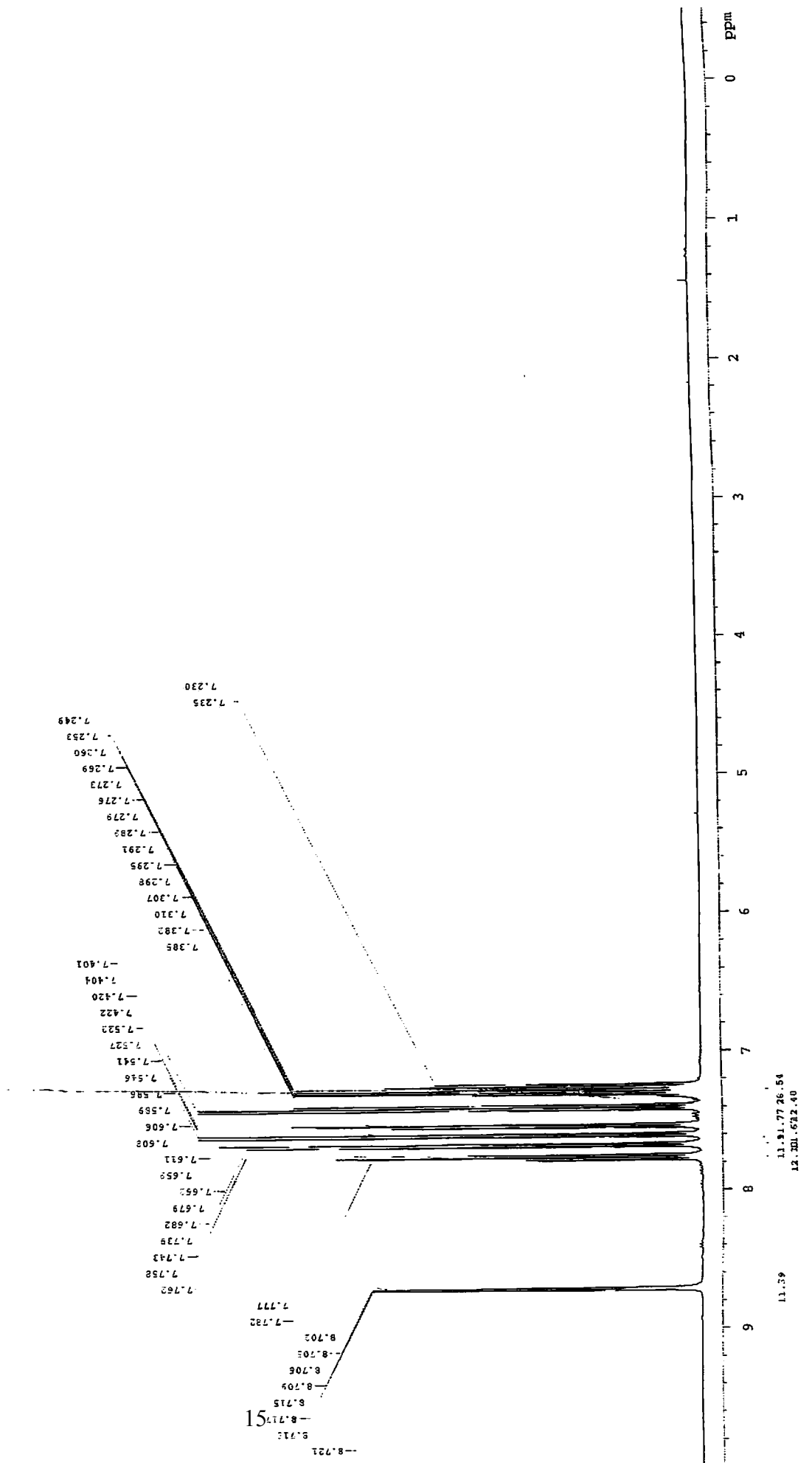
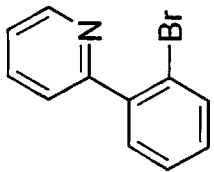
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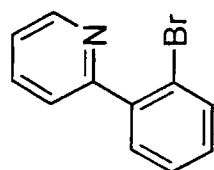


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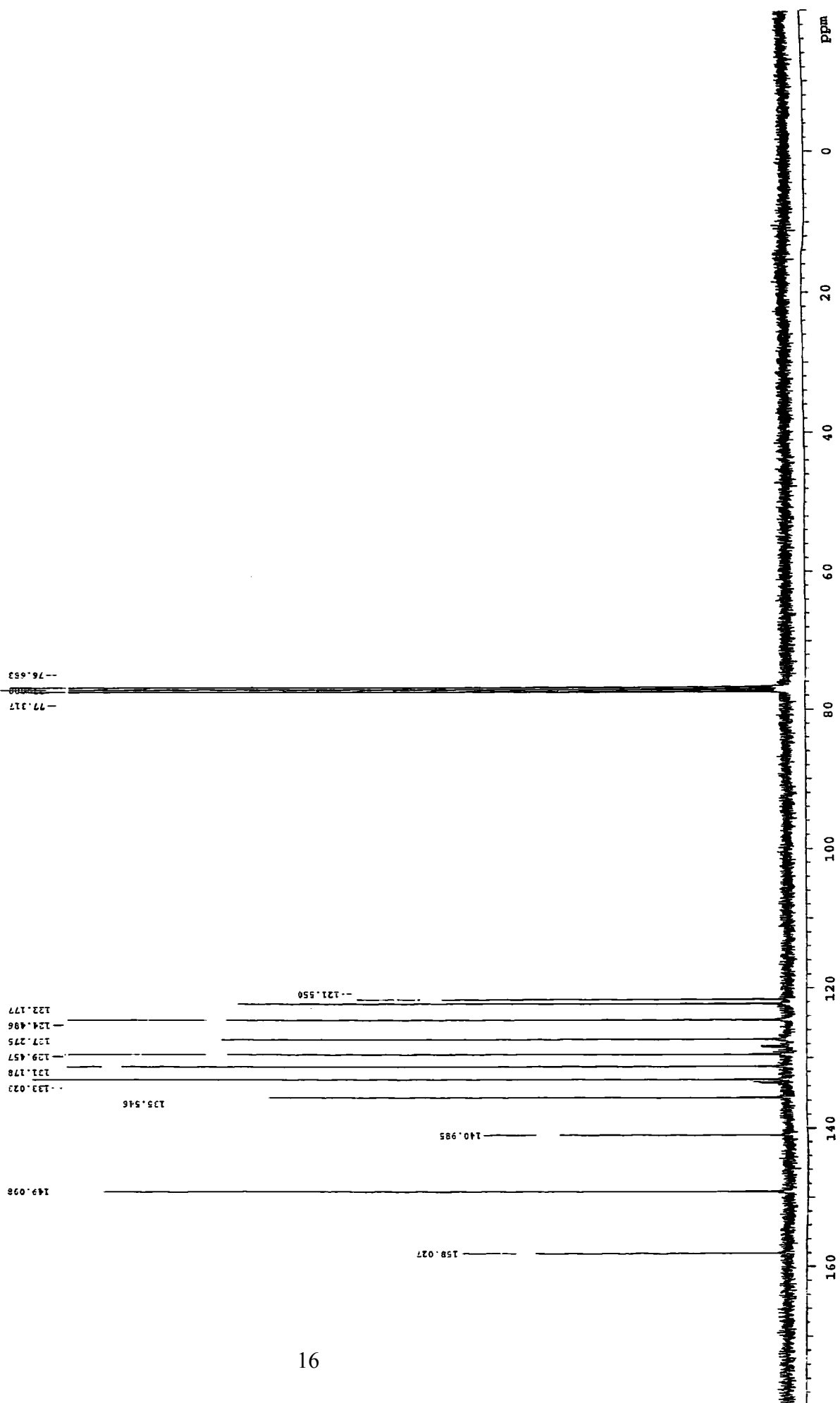


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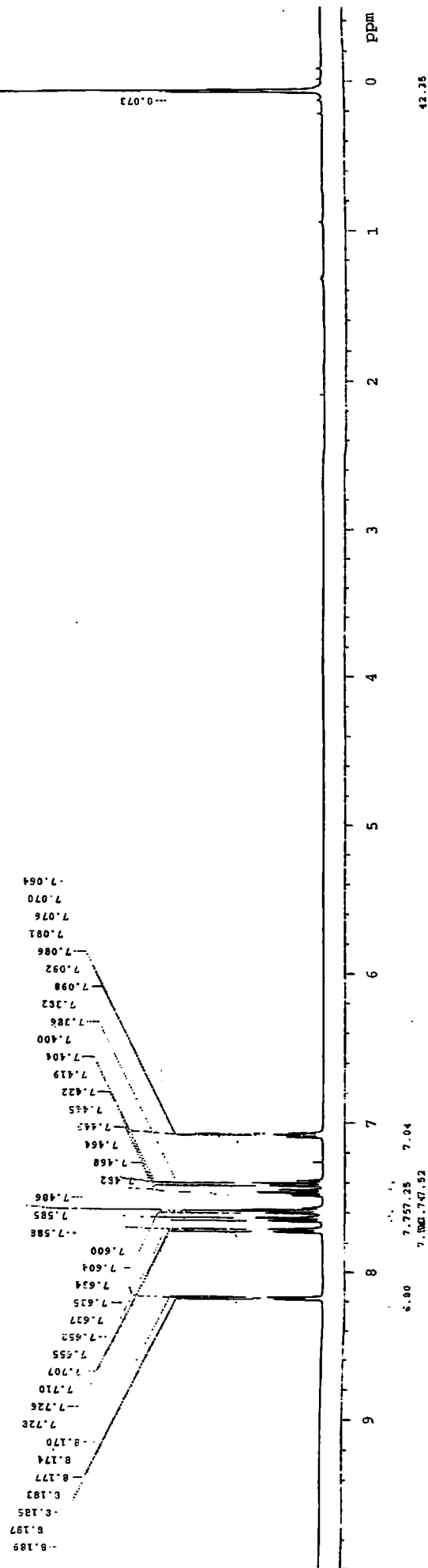
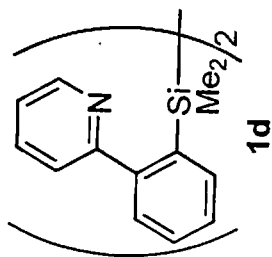


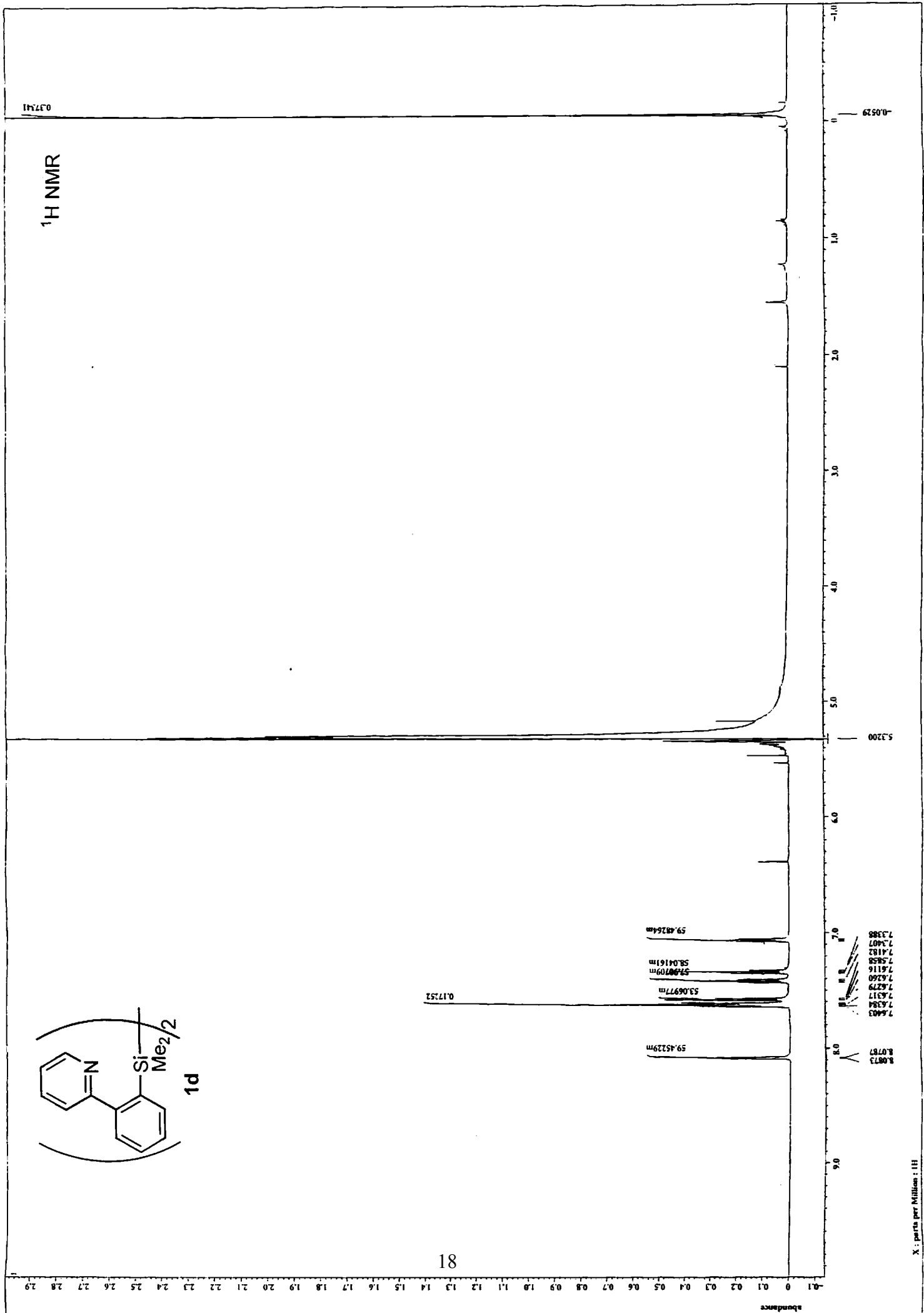


¹³C NMR



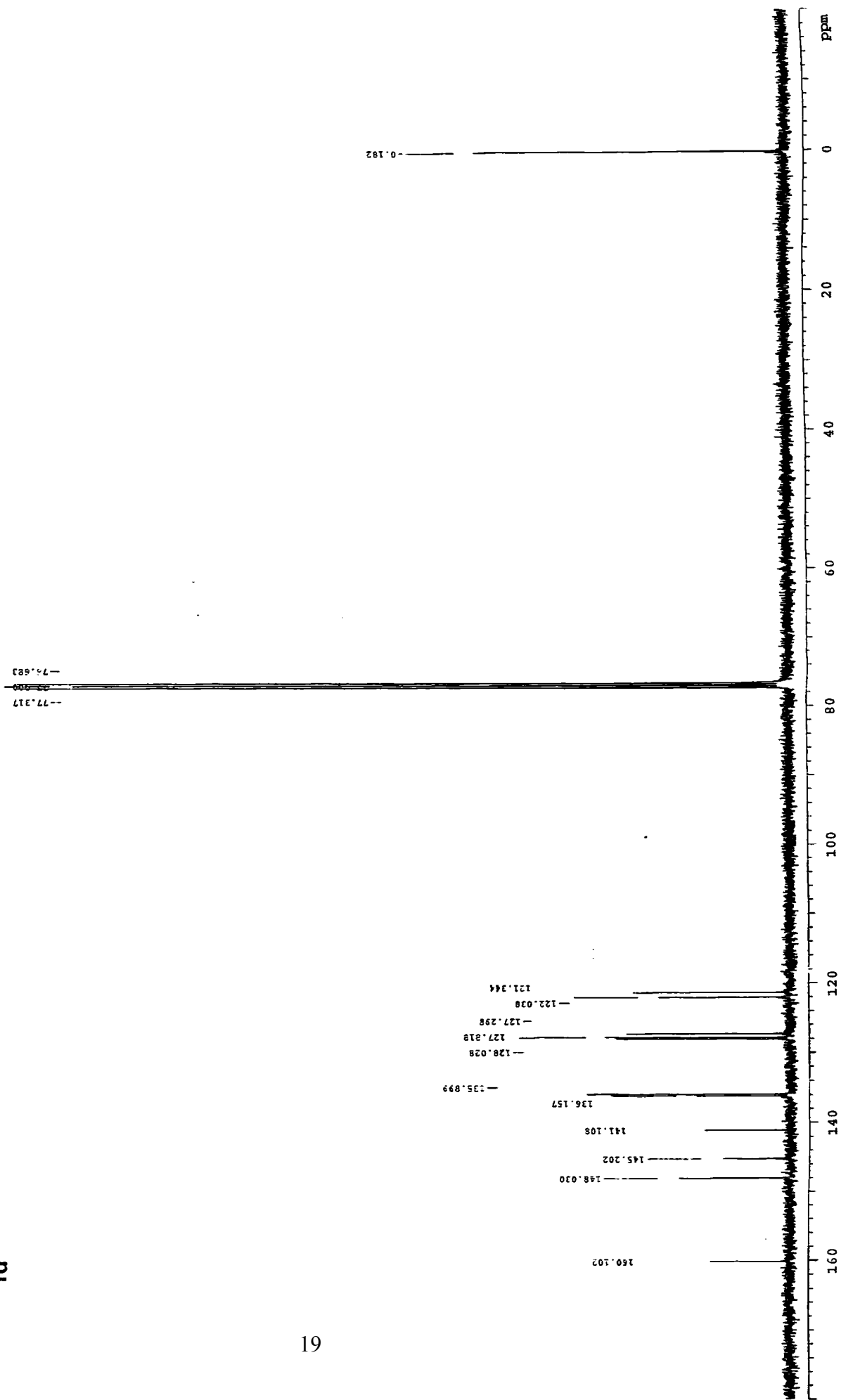
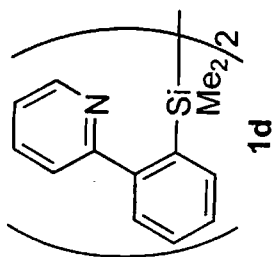
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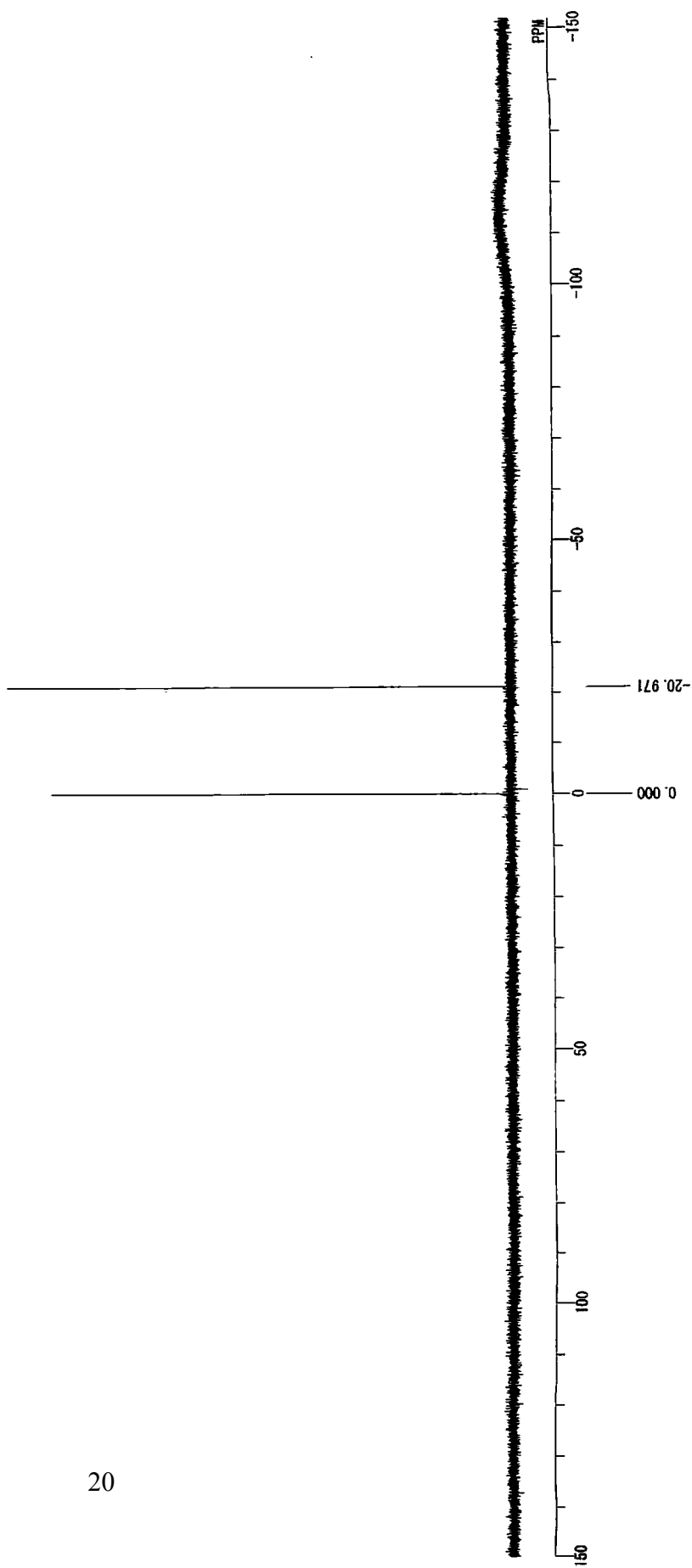
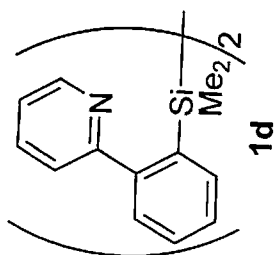


X : parts per Million : Hz

^{13}C NMR

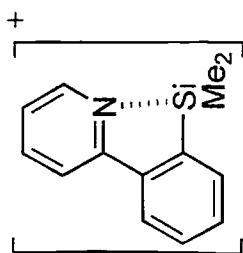


^{29}Si NMR



¹H NMR

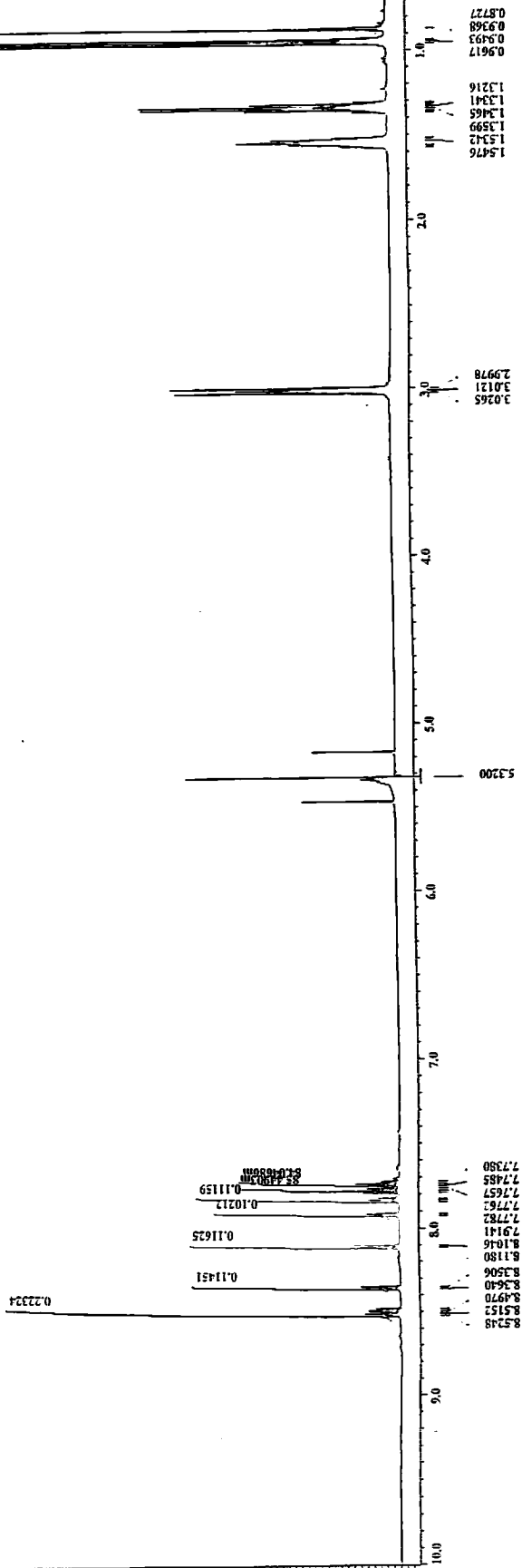
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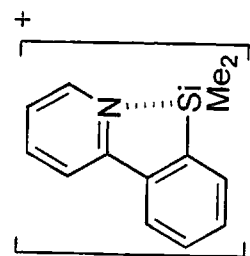


with Bu₄NB(C₆F₅)₄
3d

18.0 17.0 16.0 15.0 14.0 13.0 12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0

abundance

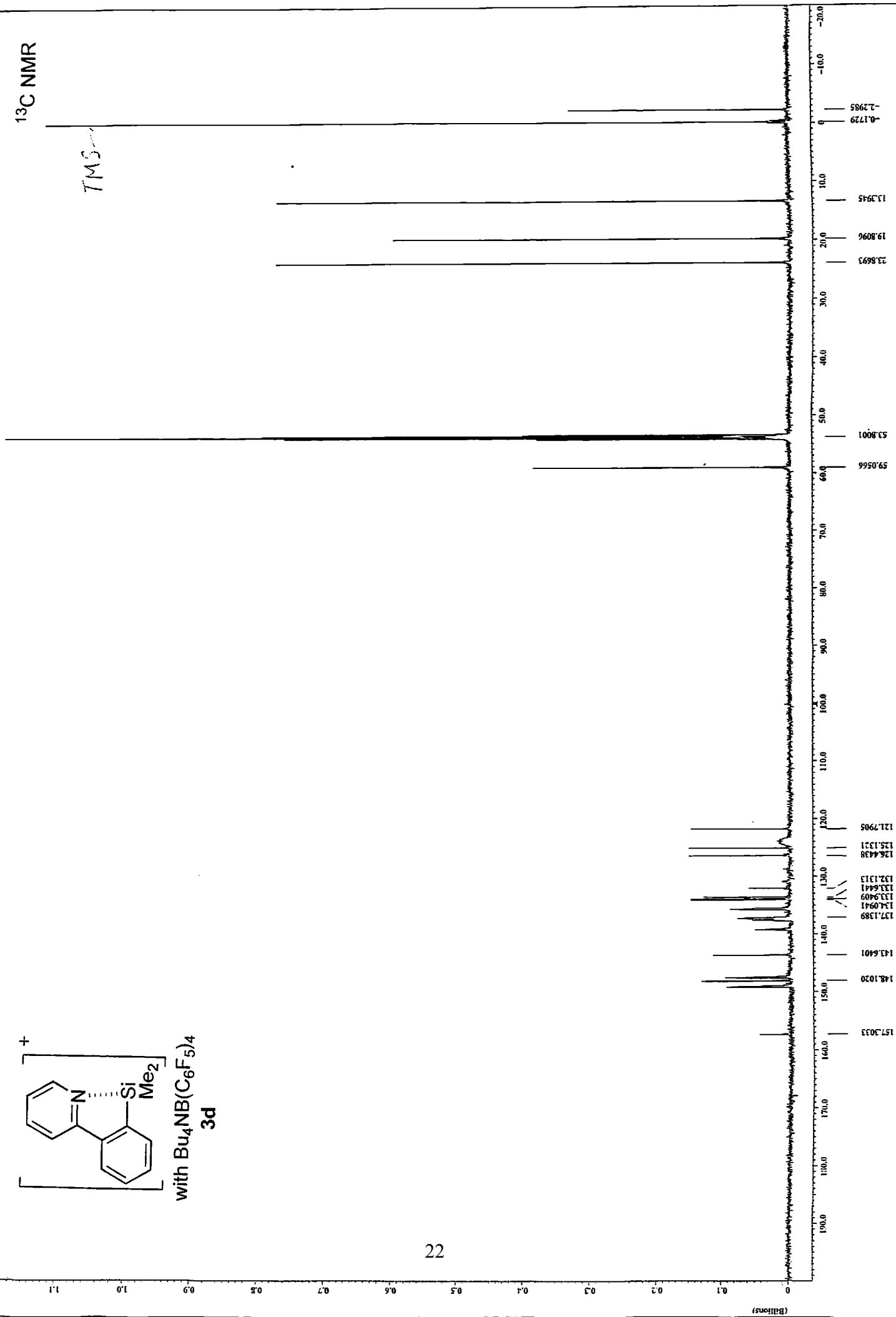


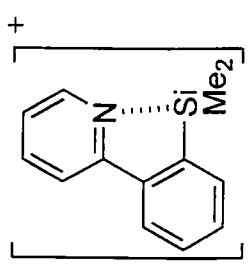


with $\text{Bu}_4\text{NB}(\text{C}_6\text{F}_5)_4$
3d

^{13}C NMR

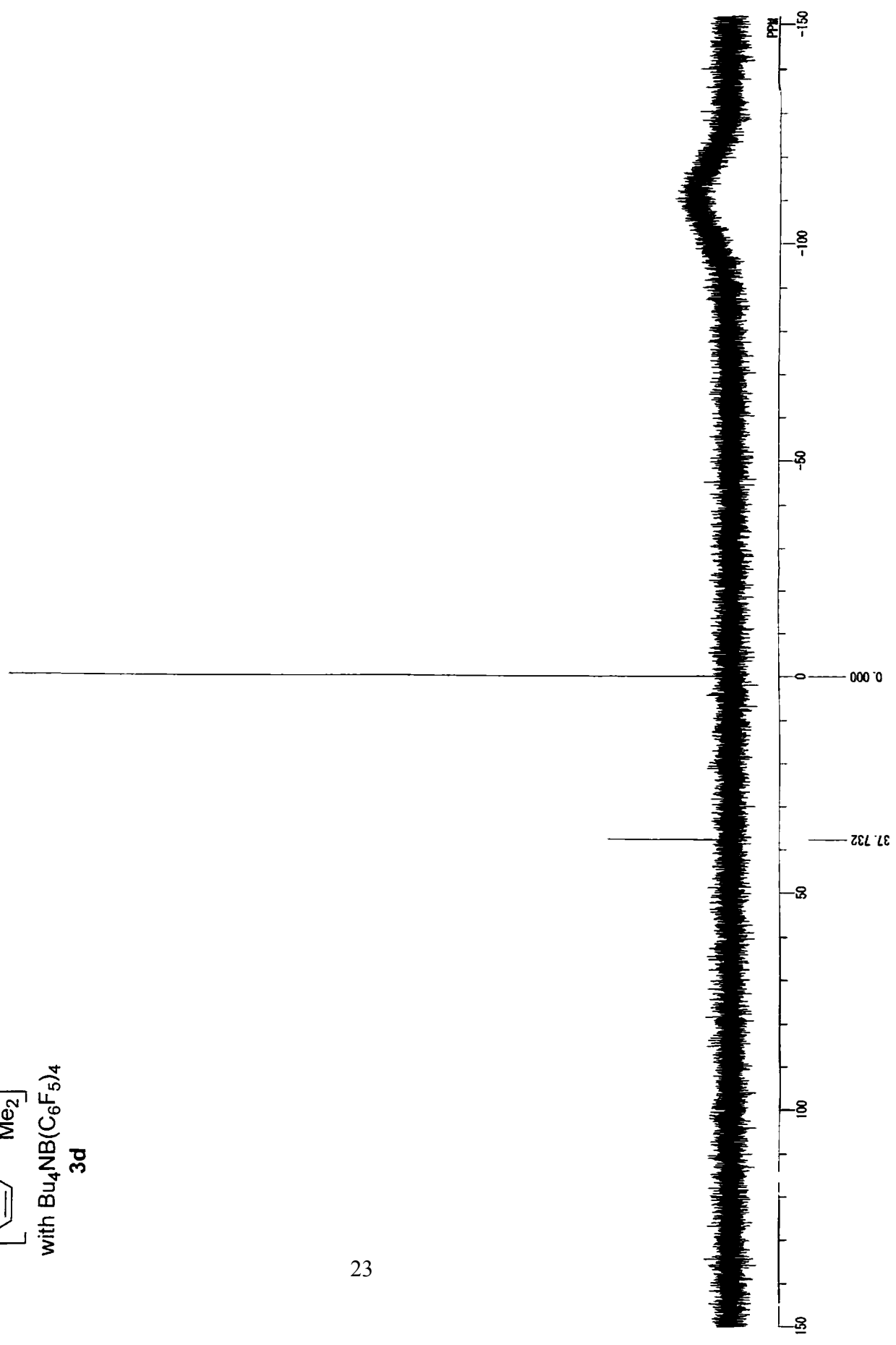
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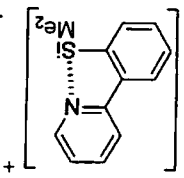
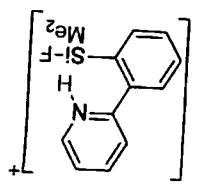
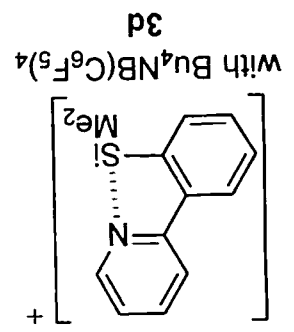
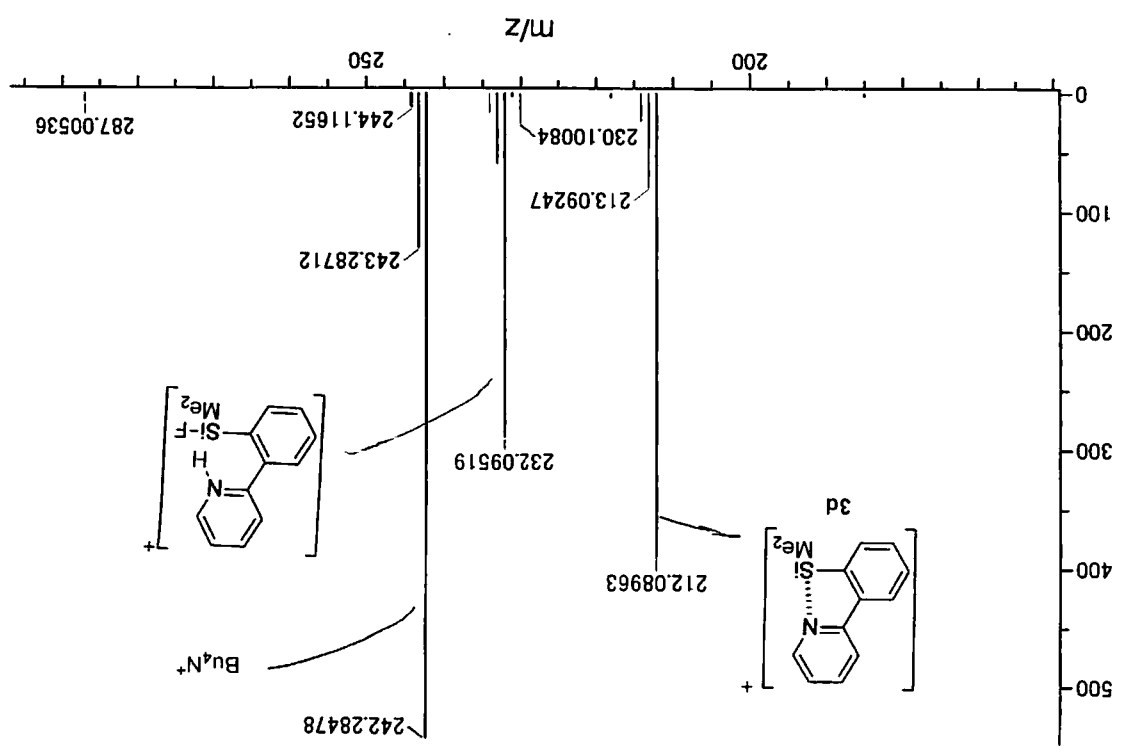




with $\text{Bu}_4\text{NB}(\text{C}_6\text{F}_5)_4$
3d

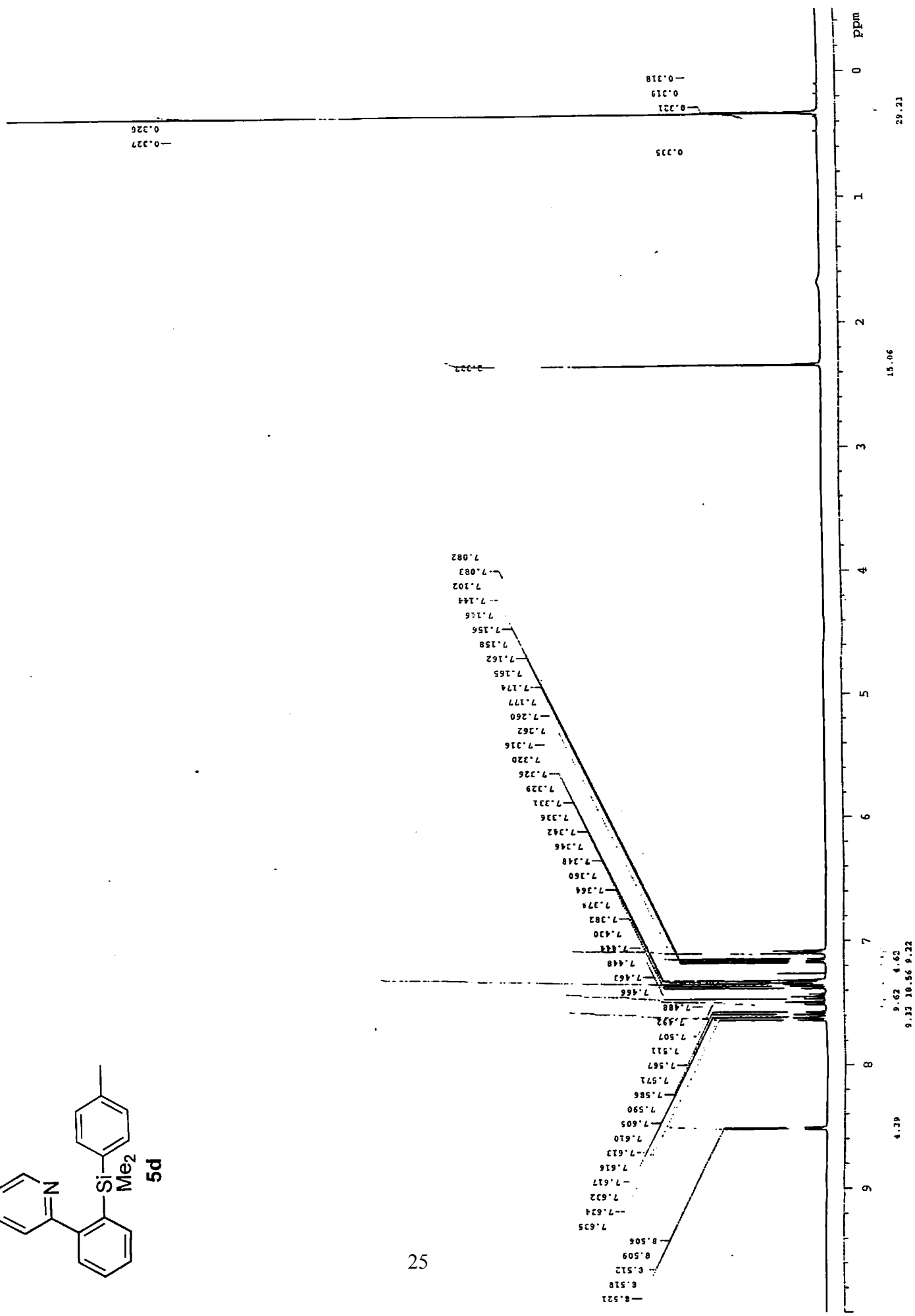
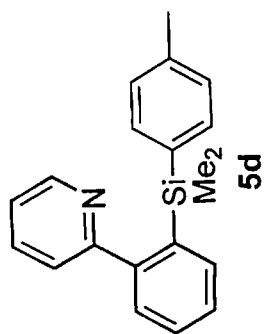
^{29}Si NMR





Bu⁴N⁺

¹H NMR



¹³C NMR

