Supporting Information for

Palladium-Catalyzed Disilylation of *ortho*-Quinodimethanes: Synthesis of 9- and 10-Membered Disilacarbocycles

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Contents

General remarks.

All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (^{1}H, 270 MHz; ^{13}C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (^{1}H, 400 MHz; ^{13}C, 99.5 MHz; ^{29}Si, 78.5 MHz) spectrometer using residual chloroform (^{1}H, δ = 7.26), benzene (^{1}H, δ = 7.15), $CDCl_3$ (^{13}C, δ = 77.0) or C_6D_6 (^{13}C, δ = 128.0) as an internal standard, and tetramethylsilane (^{29}Si, δ = 0.00) as an external standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, br = broad, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra were obtained with a JEOL JMS-SX102A spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. The preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (benzene as an eluent). Column chromatography was carried out using Merck Aluminium oxide 90 active neutral. Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. 1,4-Dioxane was distilled from sodium/benzophenone ketyl, and purified by passing through activated aluminium oxide column (5 g/8 mL of 1,4-dioxane) prior to use. MeCN was distilled from phosphorus pentoxide.

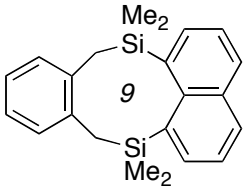
Chemicals.

o-Quinodimethane precursors (**1**)[1] and disilanes (**2**)[2] were prepared according to literature procedures.

Disilylation of *o*-quinodimethanes: a general procedure.

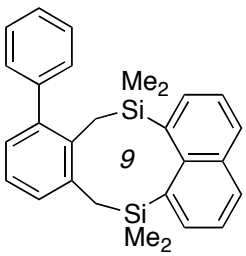
A 1,4-dioxane solution (1.0 mL) of 18-crown-6 (0.195 g, 0.738 mmol), KF (42.8 mg, 0.738 mmol), diphenyl-2-pyridylphosphine (2.6 mg, 0.010 mmol) and $Pd(dba)_2$ (5.8 mg, 0.010 mmol) was degassed by two freeze-thaw cycles, and the mixture was stirred at room temperature for 0.5 h. To this mixture was added **1** (0.396 mmol) and **2** (0.198 mmol), and the resulting mixture was stirred at 40 °C. After the time specified in Table 1 and Scheme 1, the mixture was diluted with ethyl acetate, filtered through a Celite plug, and concentrated. Alumina column chromatography (hexane as an eluent, Activity IV) followed by gel permeation chromatography (benzene as an eluent) gave the corresponding product.

7,7,14,14-Tetramethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3aa)



Isolated in 46% yield as a white solid: mp 61–65 °C; ^{1}H NMR ($CDCl_3$) δ 0.19 (s, 12 H), 2.66 (s, 4 H), 6.93-7.10 (m, 4 H), 7.33-7.42 (m, 2 H), 7.76 (dd, J = 8.0, 1.2 Hz, 2 H), 7.81 (dd, J = 6.8, 1.2 Hz, 2 H); ^{13}C NMR (C_6D_6) δ 2.5, 25.3, 124.3, 125.3, 129.6, 131.0, 134.1, 136.2, 138.4, 139.4, 140.1; ^{29}Si NMR (C_6D_6) δ –2.6; HRMS Calcd for $C_{22}H_{26}Si_2$: M^+, 346.1573. Found: *m*/*z* 346.1567.

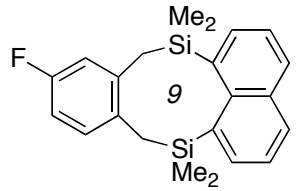
7,7,14,14-Tetramethyl-9-phenyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3ba)



Isolated in 64% yield as a white solid: mp 147–148 °C; ^{1}H NMR (C_6D_6) δ 0.07 (s, 6 H), 0.16 (s, 6 H), 2.73 (s, 2 H), 2.75 (s, 2 H), 6.90-7.41 (m, 10 H), 7.52-7.64 (m, 2 H), 7.66-7.80 (m, 2 H); ^{13}C NMR (C_6D_6) δ 2.2, 2.4, 21.4, 25.5, 124.2, 124.3, 124.7, 126.9, 127.0, 128.5, 129.1, 129.9, 131.09, 131.12, 134.1, 135.9, 136.2, 136.5, 139.1, 139.4, 139.6, 139.9,

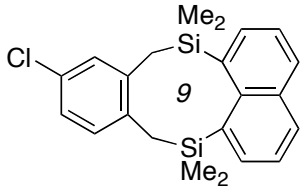
142.1, 143.8; ^{29}Si NMR (C_6D_6) δ –2.9, -2.3; Anal Calcd for $C_{28}H_{30}Si_2$: C, 79.56; H, 7.15. Found: C, 79.23; H, 6.88.

10-Fluoro-7,7,14,14-tetramethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3ca)



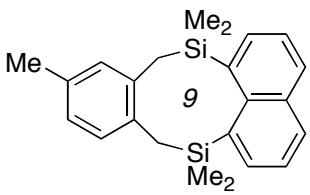
Isolated in 59% yield as a white solid: mp 93–95 °C; ^{1}H NMR ($CDCl_3$) δ 0.20 (s, 6 H), 0.21 (s, 6 H), 2.62 (s, 2 H), 2.66 (s, 2 H), 6.69 (td, *J* = 8.5, 2.9 Hz, 1 H), 6.78 (dd, *J* = 10.0, 2.7 Hz, 1 H), 6.98 (dd, *J* = 8.3, 6.1 Hz, 1 H), 7.41 (t, *J* = 7.8 Hz, 2 H), 7.66-7.94 (m, 4 H); ^{13}C NMR (C_6D_6) δ 2.38, 2.42. 24.3, 25.5, 111.6 (d, $J_{C\text{-}F}$ = 20.7 Hz), 116.1 (d, $J_{C\text{-}F}$ = 20.8 Hz), 124.3, 128.5, 130.5 (d, $J_{C\text{-}F}$ = 8.5 Hz), 133.86, 133.90, 134.1, 136.2, 138.9, 139.1, 140.0, 140.7 (d, $J_{C\text{-}F}$ = 7.3 Hz), 161.2 (d, $J_{C\text{-}F}$ = 241.7 Hz); ^{29}Si NMR ($CDCl_3$) δ –3.2, -3.1; Anal Calcd for $C_{22}H_{25}FSi_2$: C, 72.47; H, 6.91. Found: C, 72.07; H, 6.90.

10-Chloro-7,7,14,14-tetramethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3da)



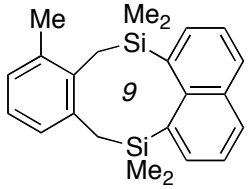
Isolated in 57% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.20 (s, 6 H), 0.21 (s, 6 H), 2.63 (s, 2 H), 2.65 (s, 2 H), 6.97 (s, 2 H), 7.06 (s, 1 H), 7.36-7.44 (m, 2 H), 7.76-7.83 (m, 4 H); ^{13}C NMR (C_6D_6) δ 2.3, 2.4, 24.6, 25.2, 124.27, 124.31, 125.2, 129.3, 130.6, 130.7, 131.12, 131.14, 134.1, 136.16, 136.23, 137.0, 138.8, 138.9, 139.9, 140.7; ^{29}Si NMR (C_6D_6) δ –3.0, -2.7; Anal Calcd for $C_{22}H_{25}ClSi_2$: C, 69.34; H, 6.61. Found: C, 69.17; H, 6.91.

7,7,10,14,14-Pentamethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3ea)



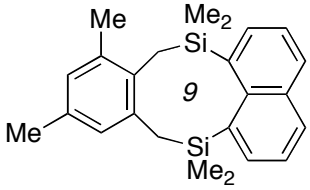
Isolated in 43% yield as a white solid: mp 74–76 °C; ^{1}H NMR ($CDCl_3$) δ 0.18 (s, 6 H), 0.19 (s, 6 H), 2.27 (s, 3 H), 2.62 (s, 2 H), 2.63 (s, 2 H), 6.82 (d, *J* = 7.6 Hz, 1 H), 6.87 (s, 1 H), 6.94 (d, *J* = 7.6 Hz, 1 H), 7.41 (t, *J* = 7.5 Hz, 2 H), 7.76 (d, *J* = 7.8 Hz, 2 H), 7.82 (d, *J* = 6.8 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ 2.5, 20.9, 24.5, 25.0, 123.8, 125.4, 129.1, 130.0, 130.5, 133.4, 133.9, 134.9, 135.7, 135.79, 135.83, 137.9, 139.2, 139.3, 139.5; ^{29}Si NMR (C_6D_6) δ –2.64, -2.59; Anal Calcd for $C_{23}H_{28}Si_2$: C, 76.60; H, 7.83. Found: C, 76.42; H, 8.08.

7,7,9,14,14-Pentamethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3fa)



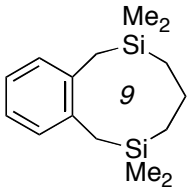
Isolated in 36% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.18 (s, 6 H), 0.20 (s, 6 H), 2.32 (s, 3 H), 2.66 (s, 2 H), 2.68 (s, 2 H), 6.86-7.00 (m, 3 H), 7.36-7.48 (m, 2 H), 7.70-7.93 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 2.5, 2.9, 20.0, 21.5, 25.3, 123.9, 124.0, 126.7, 127.3, 130.6, 133.4, 134.7, 135.79, 135.82, 137.1, 138.1, 139.0, 139.5, 139.6; ^{29}Si NMR (C_6D_6) δ –2.8, -2.3; HRMS Calcd for $C_{23}H_{28}Si_2$: M^+, 360.1730. Found: *m*/*z* 360.1721.

7,7,9,11,14,14-Hexamethyl-7,8,13,14-tetrahydrobenzo[*g*]naphtho[1,8-*bc*][1,5]disilonine (3ga)



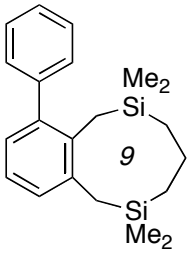
Isolated in 36% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.19 (s, 6 H), 0.20 (s, 6 H), 2.26 (s, 3 H), 2.30 (s, 3 H), 2.64 (s, 4 H), 6.77 (s, 1 H), 6.80 (s, 1 H), 7.31-7.50 (m, 2 H), 7.71-7.95 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 2.5, 2.8, 19.4, 20.8, 21.4, 25.3, 123.8, 123.9, 127.5, 128.1, 130.5, 130.6, 133.1, 133.4, 133.8, 134.5, 135.75, 135.79, 137.9, 139.2, 139.56, 139.58; ^{29}Si NMR ($CDCl_3$) δ –3.4, -2.7; HRMS Calcd for $C_{24}H_{30}Si_2$: M^+, 374.1886. Found: *m*/*z* 374.1881.

2,2,6,6-Tetramethyl-2,3,4,5,6,7-hexahydro-1*H*-benzo[*g*][1,5]disilonine (3ab)



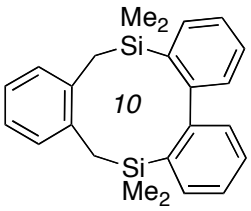
Isolated in 50% yield as a white solid: mp 51–52 °C; ^{1}H NMR (C_6D_6) δ 0.02 (s, 12 H), 0.36-0.46 (m, 4 H), 1.46-1.60 (m, 2 H), 2.15 (s, 4 H), 6.91-7.05 (m, 4 H); ^{13}C NMR (C_6D_6) δ –2.3, 14.9, 19.2, 25.0, 124.4, 129.6, 137.4; ^{29}Si NMR (C_6D_6) δ 3.5; HRMS Calcd for $C_{15}H_{26}Si_2$: M^+, 262.1573. Found: *m/z* 262.1570.

2,2,6,6-Tetramethyl-8-phenyl-2,3,4,5,6,7-hexahydro-1*H*-benzo[*g*][1,5]disilonine (3bb)



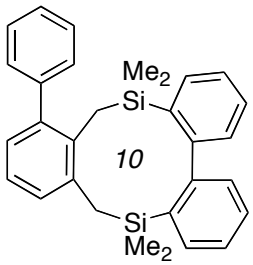
Isolated in 54% yield as a white solid: mp 82–83 °C; ^{1}H NMR (C_6D_6) δ –0.23 (s, 6 H), 0.10 (s, 6 H), 0.35 (t, *J* = 6.8 Hz, 2 H), 0.60 (t, *J* = 6.8 Hz, 2 H), 1.46-1.66 (m, 2 H), 2.12 (s, 2 H), 2.41 (s, 2 H), 6.92-7.25 (m, 6 H), 7.27-7.39 (m, 2 H); ^{13}C NMR (C_6D_6) δ –2.3, -1.4, 13.4, 16.7, 19.2, 21.8, 24.8, 123.9, 126.6, 127.3, 128.1, 128.6, 130.3, 135.3, 138.3, 142.3, 144.4; ^{29}Si NMR (C_6D_6) δ 3.6, 4.3; HRMS Calcd for $C_{21}H_{30}Si_2$: M^+, 338.1886. Found: *m/z* 338.1883.

5,5,12,12-Tetramethyl-5,6,11,12-tetrahydrotribenzo[*b,d,h*][1,6]disilecine (3ac)



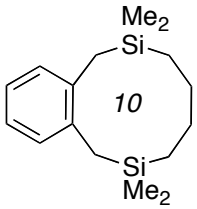
Isolated in 44% yield as a white solid: mp 114–115 °C; ^{1}H NMR (C_6D_6) δ –0.19 (s, 6 H), 0.23 (s, 6 H), 1.82 (d, *J* = 14.4 Hz, 2 H), 2.21 (d, *J* = 14.9 Hz, 2 H), 6.85 (dd, *J* = 5.4, 3.7 Hz, 2 H), 6.90-7.00 (m, 2 H), 7.02-7.22 (m, 6 H), 7.40 (dd, *J* = 6.0, 1.5 Hz, 2 H); ^{13}C NMR ($CDCl_3$) δ –1.7, 0.0, 25.8, 124.0, 126.6, 128.0, 130.0, 130.2, 133.7, 137.6, 138.6, 149.4; ^{29}Si NMR (C_6D_6) δ –3.9; Anal Calcd for $C_{24}H_{28}Si_2$: C, 77.35; H, 7.57. Found: C, 77.09; H, 7.57.

5,5,12,12-Tetramethyl-7-phenyl-5,6,11,12-tetrahydrotribenzo[*b,d,h*][1,6]disilecine (3bc)



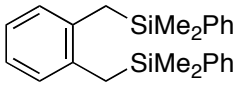
Isolated in 73% yield as a white solid: mp 161–163 °C; ^{1}H NMR (C_6D_6) δ –0.27 (s, 3 H), -0.07 (s, 3 H), 0.18 (s, 3 H), 0.31 (s, 3 H), 1.82 (d, *J* = 14.5 Hz, 1 H), 2.27 (s, 2 H), 2.57 (d, *J* = 14.5 Hz, 1 H), 6.87-7.29 (m, 14 H), 7.36-7.44 (m, 1 H), 7.45-7.54 (m, 1 H); ^{13}C NMR (C_6D_6) δ –2.5, -1.6, 0.4, 1.0, 21.7, 27.2, 124.2, 126.6, 127.1, 127.3, 128.1, 128.5, 128.8, 129.8, 129.9, 130.5, 131.2, 133.6, 134.1, 136.3, 138.6, 138.9, 140.1, 142.7, 144.1, 149.3, 150.3; ^{29}Si NMR (C_6D_6) δ –3.9, -3.4; Anal Calcd for $C_{30}H_{32}Si_2$: C, 80.30; H, 7.19. Found: C, 80.22; H, 7.12.

2,2,7,7-Tetramethyl-1,2,3,4,5,6,7,8-octahydrobenzo[*c*][1,6]disilecine (3ad)



Isolated in 8% yield as a colorless oil: ^{1}H NMR (C_6D_6) δ 0.08 (s, 12 H), 0.37 (brs, 4 H), 1.25-1.45 (m, 4 H), 2.08 (s, 4 H), 6.96-7.12 (m, 4 H); ^{13}C NMR (C_6D_6) δ –1.9, 10.4, 22.2, 25.0, 124.5, 129.7, 137.6; ^{29}Si NMR (C_6D_6) δ 3.5; Anal Calcd for $C_{16}H_{28}Si_2$: C, 69.49; H, 10.20. Found: C, 69.35; H, 10.31.

α,α'-Bis[dimethyl(phenyl)silyl]-*o*-xylene (3ae)



Isolated in 35% yield as a white solid: mp 31–32 °C; ^{1}H NMR (C_6D_6) δ 0.17 (s, 12 H), 2.11 (s, 4 H), 6.84-6.90 (m, 2 H), 6.92-6.98 (m, 2 H), 7.12-7.22 (m, 6 H), 7.32-7.39 (m, 4 H); ^{13}C NMR (C_6D_6) δ –3.0, 23.7, 124.7, 129.3, 129.6, 133.9, 136.8, 139.1; ^{29}Si NMR (C_6D_6) δ –3.4; HRMS Calcd for $C_{24}H_{30}Si_2$: M^+, 374.1886. Found: *m/z* 374.1890.

References

1. Yoshida, H.; Nakano, S. Yamaryo, Y.; Ohshita, J.; Kunai, A. *Org. Lett.* **2006**, *8*, 4157.

2. Yoshida, H.; Ikadai, J.; Shudo, M.; Ohshita, J.; Kunai, A. *Organometallics* **2005**, *24*, 156.

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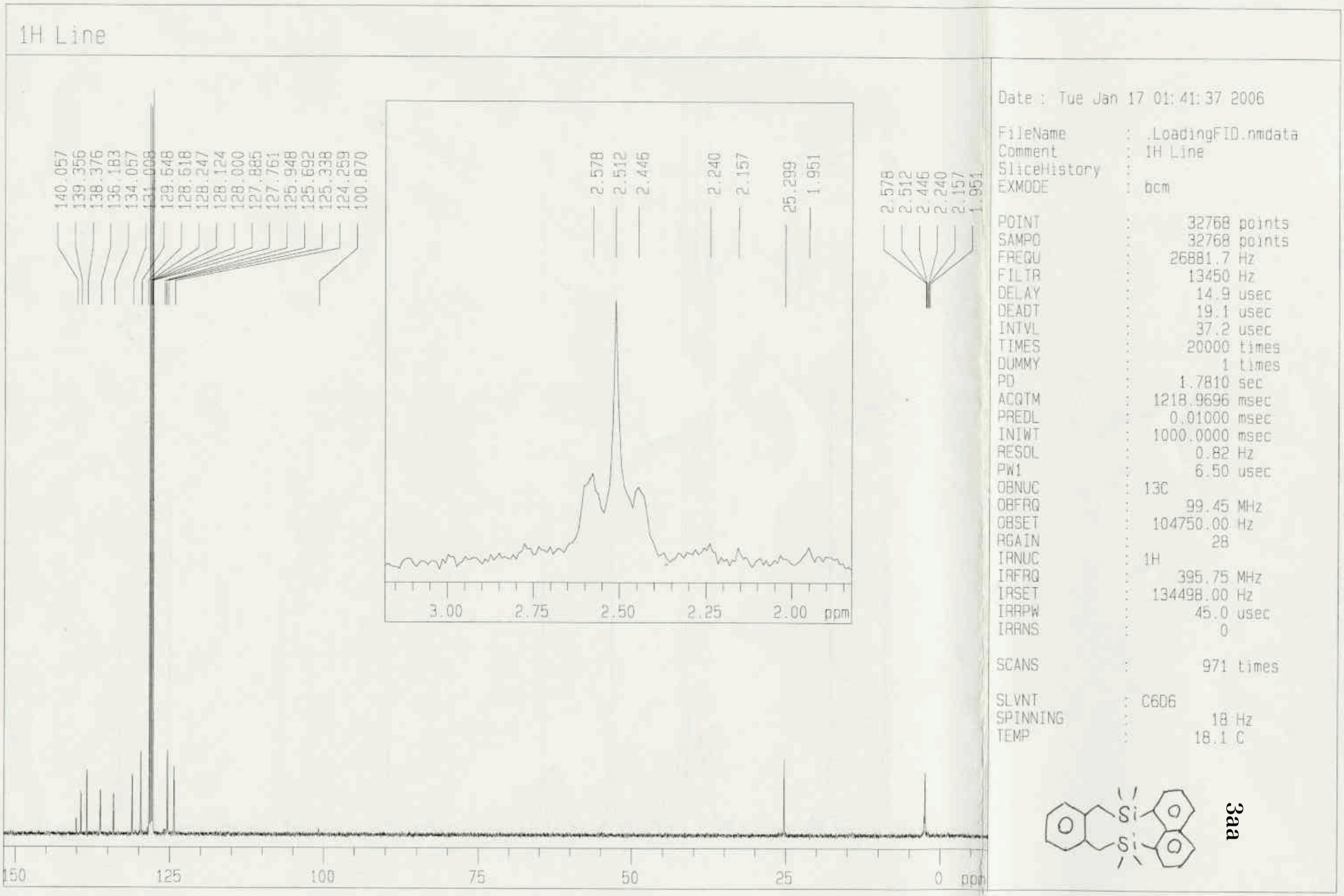
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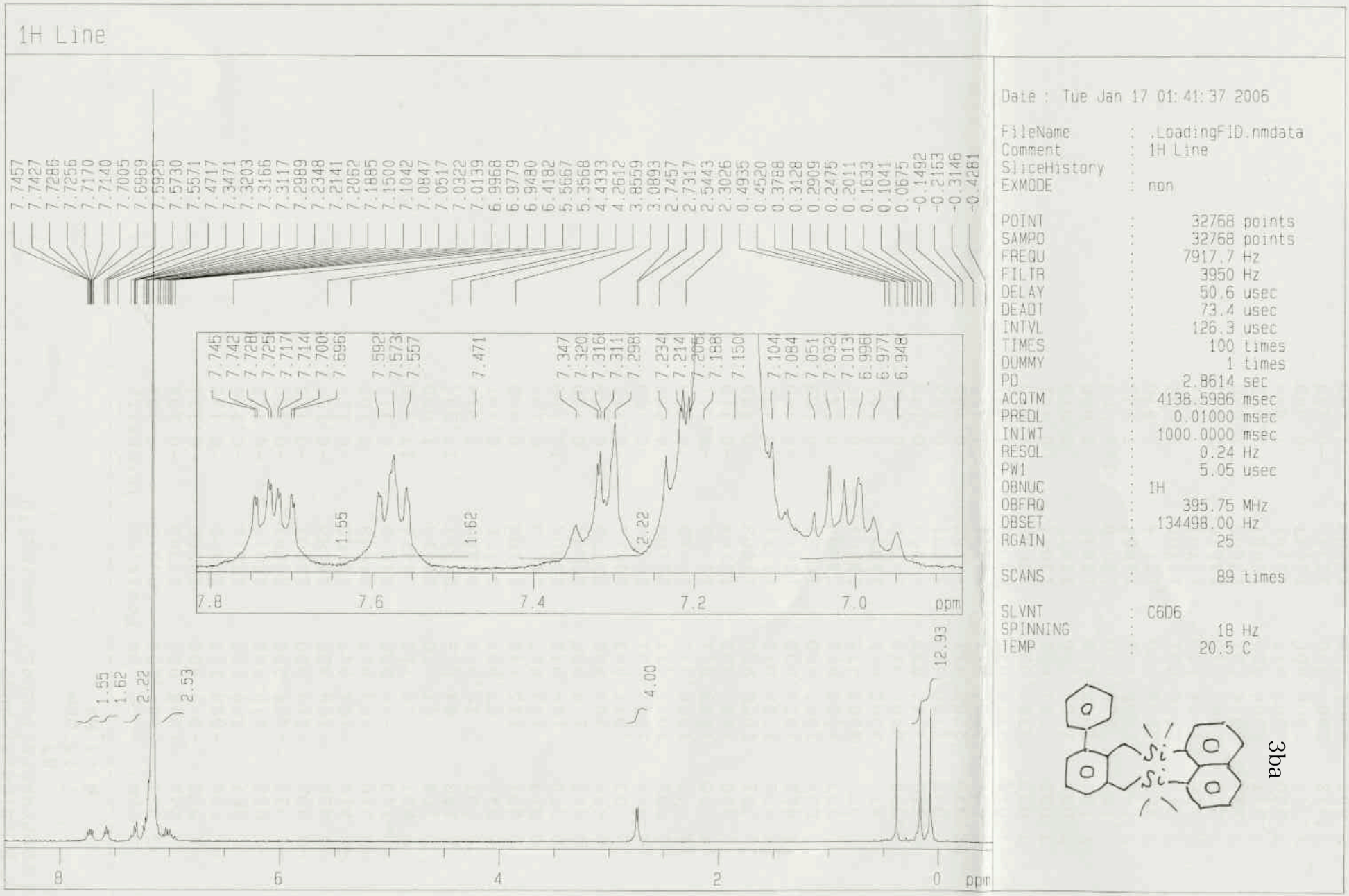
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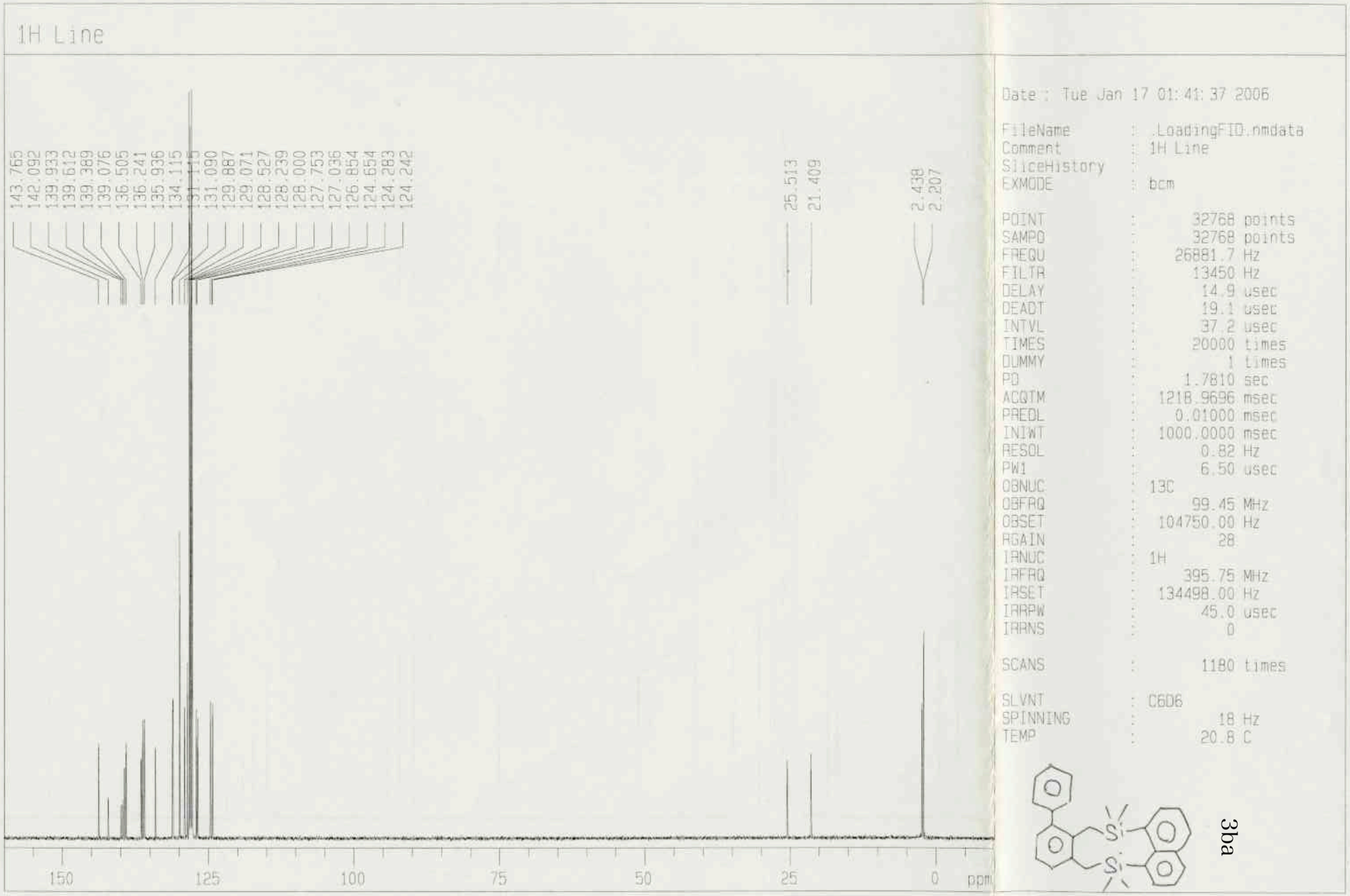
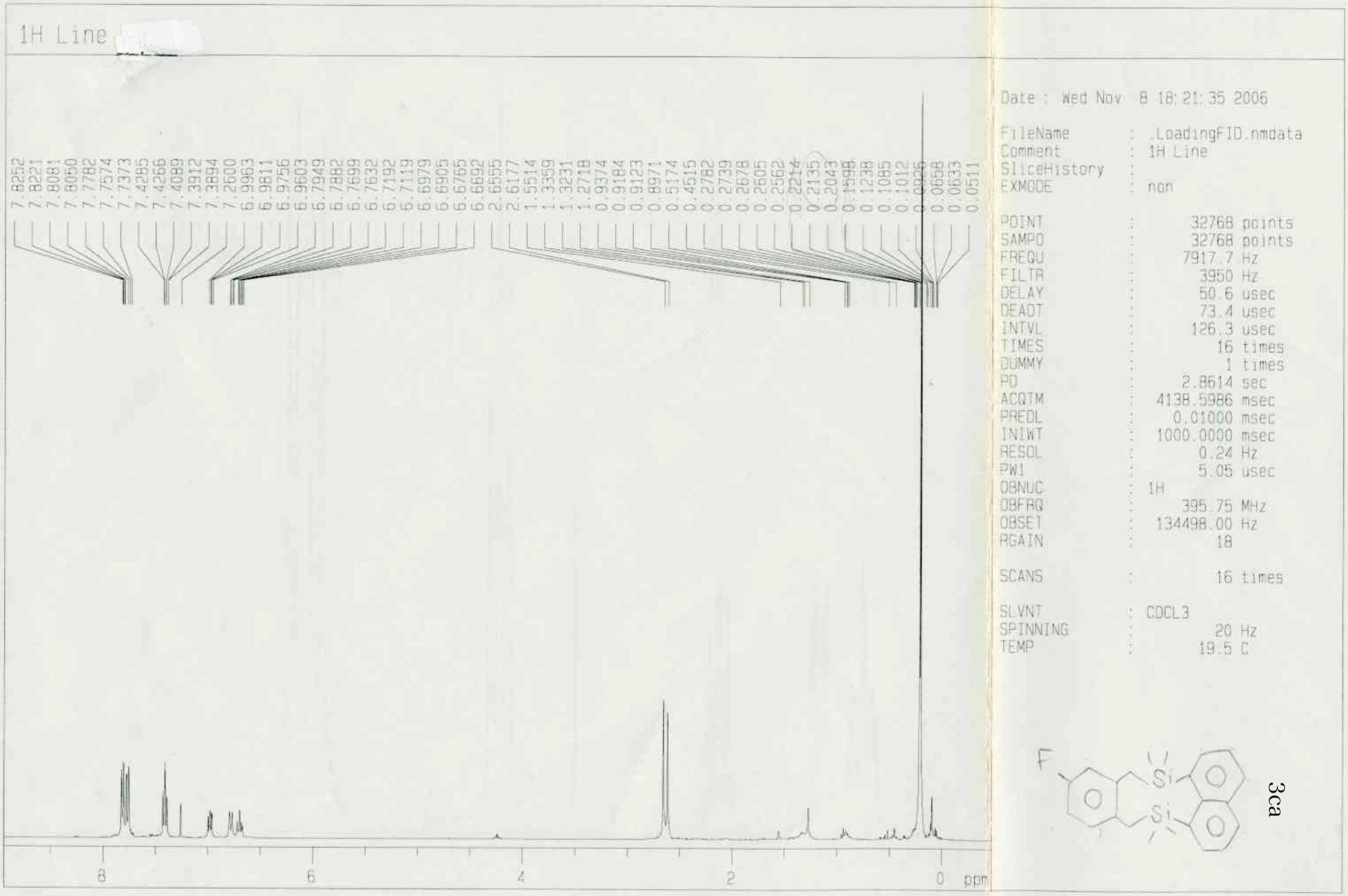
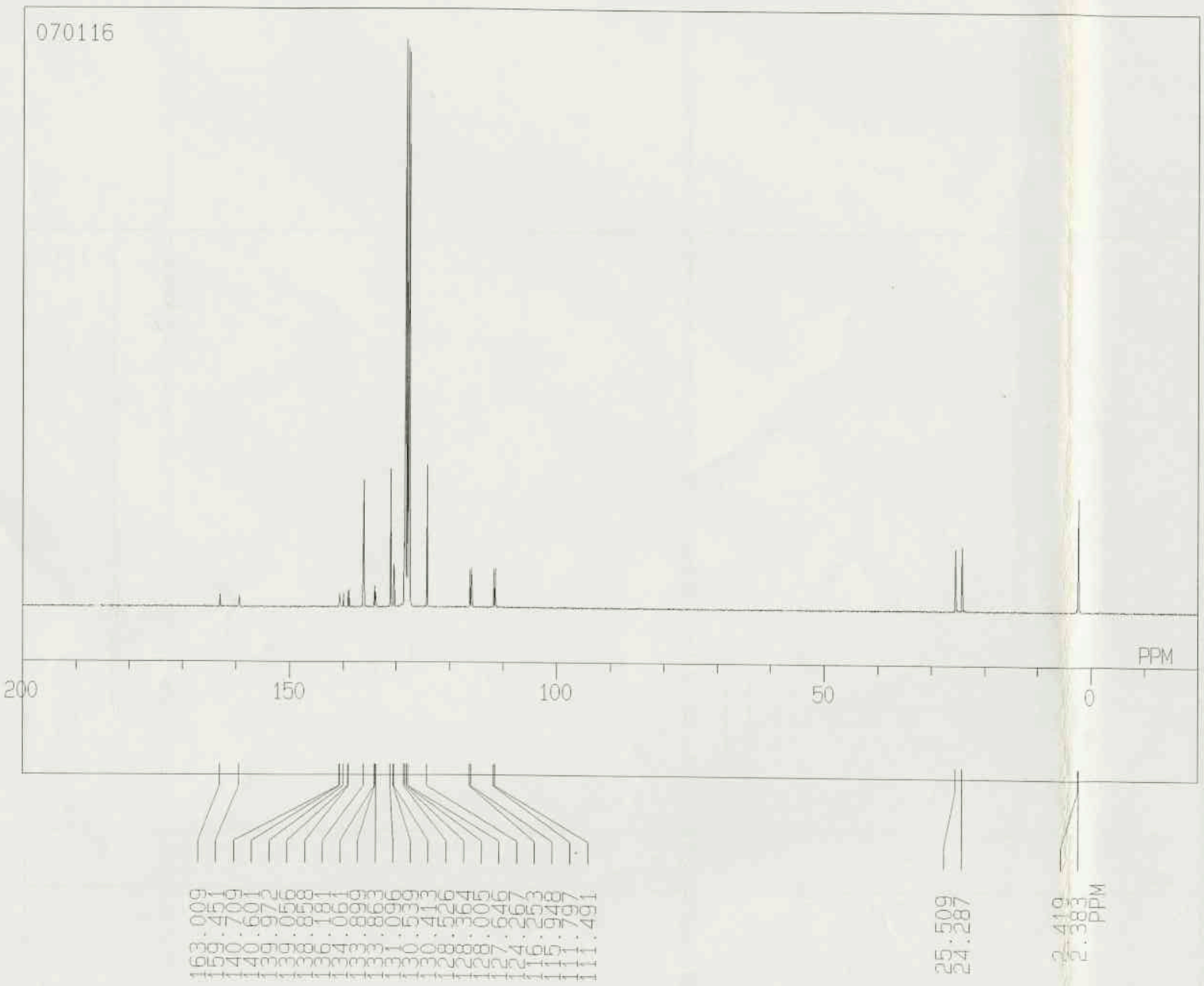
-0.2153
-0.3146
-0.4281
1.55
1.62
2.22
2.53
4.00
12.93
8
6
4
2
0
ppm
7.8
7.6
7.4
7.2
7.0
ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 100 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 25
SCANS : 89 times
SLVNT : C6D6
SPINNING : 18 Hz
TEMP : 20.5 C
Si
Si

3ba

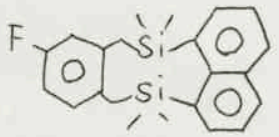

1H Line
143.765
142.092
139.933
139.612
139.389
139.076
136.505
136.241
135.936
134.115
131.115
131.090
129.887
129.071
128.527
128.239
128.000
127.753
127.036
126.854
124.654
124.283
124.242
25.513
21.409
2.438
2.207
150
125
100
75
50
25
0
ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IANUC : 1H
IAFRQ : 395.75 MHz
IASET : 134498.00 Hz
IARPW : 45.0 usec
IARNS : 0
SCANS : 1180 times
SLVNT : C6D6
SPINNING : 18 Hz
TEMP : 20.8 C
Si
Si

3ba

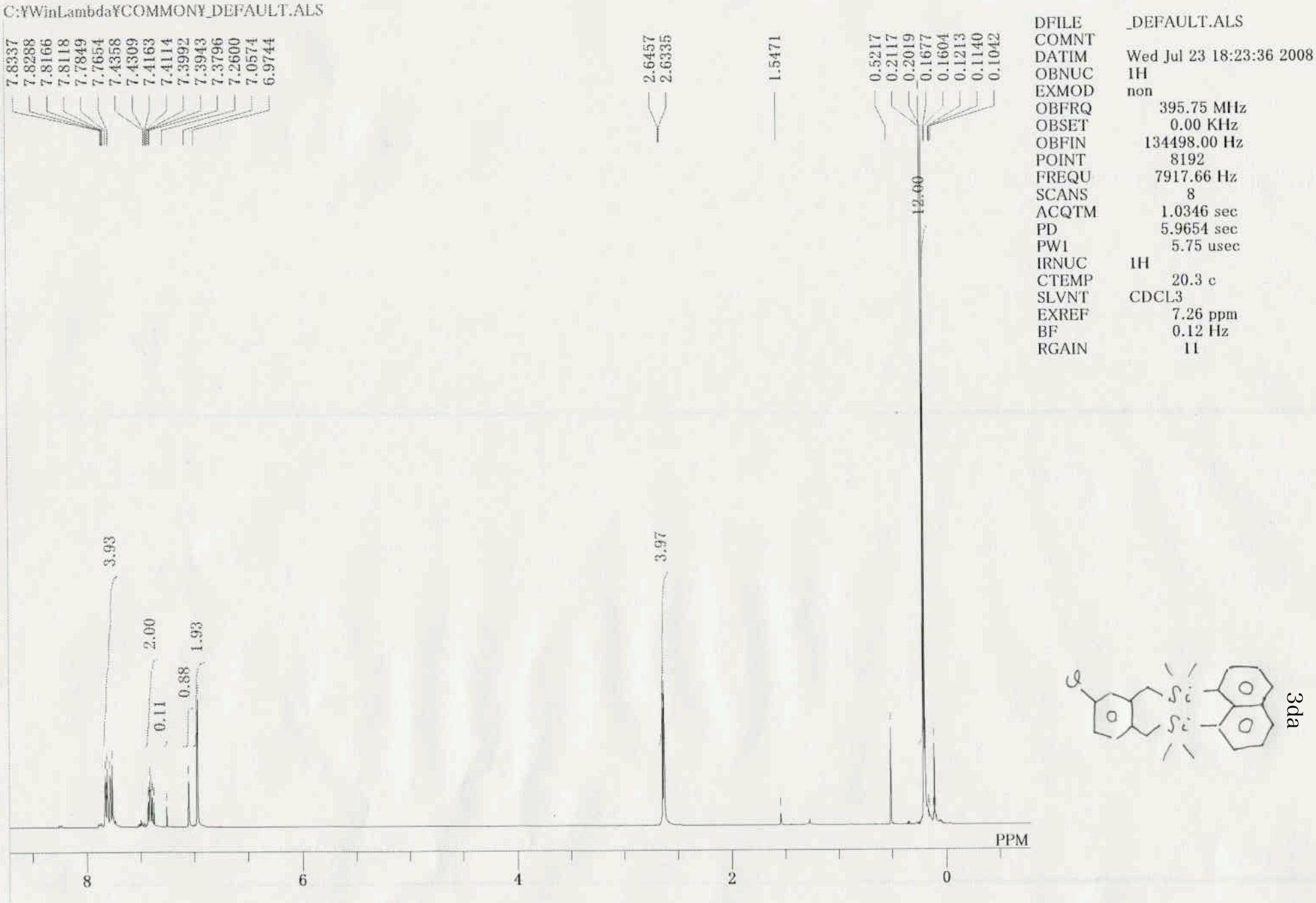

1H Line
7.8252
7.8221
7.8081
7.8050
7.7782
7.7574
7.7373
7.4285
7.4266
7.4089
7.3912
7.3894
7.2600
6.9963
6.9811
6.9756
6.9603
6.7949
6.7882
6.7699
6.7632
6.7192
6.7119
6.6979
6.6905
6.6765
6.6692
2.6555
2.6177
1.5514
1.3359
1.3231
1.2718
0.9374
0.9184
0.9123
0.8971
0.5174
0.4515
0.2782
0.2739
0.2678
0.2605
0.2562
0.2214
0.2135
0.2043
0.1598
0.1238
0.1085
0.1012
0.0926
0.0658
0.0633
0.0511
8
6
4
2
0
ppm
Date : Wed Nov 8 18:21:35 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 18
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 20 Hz
TEMP : 19.5 C
F
Si
Si
3ca

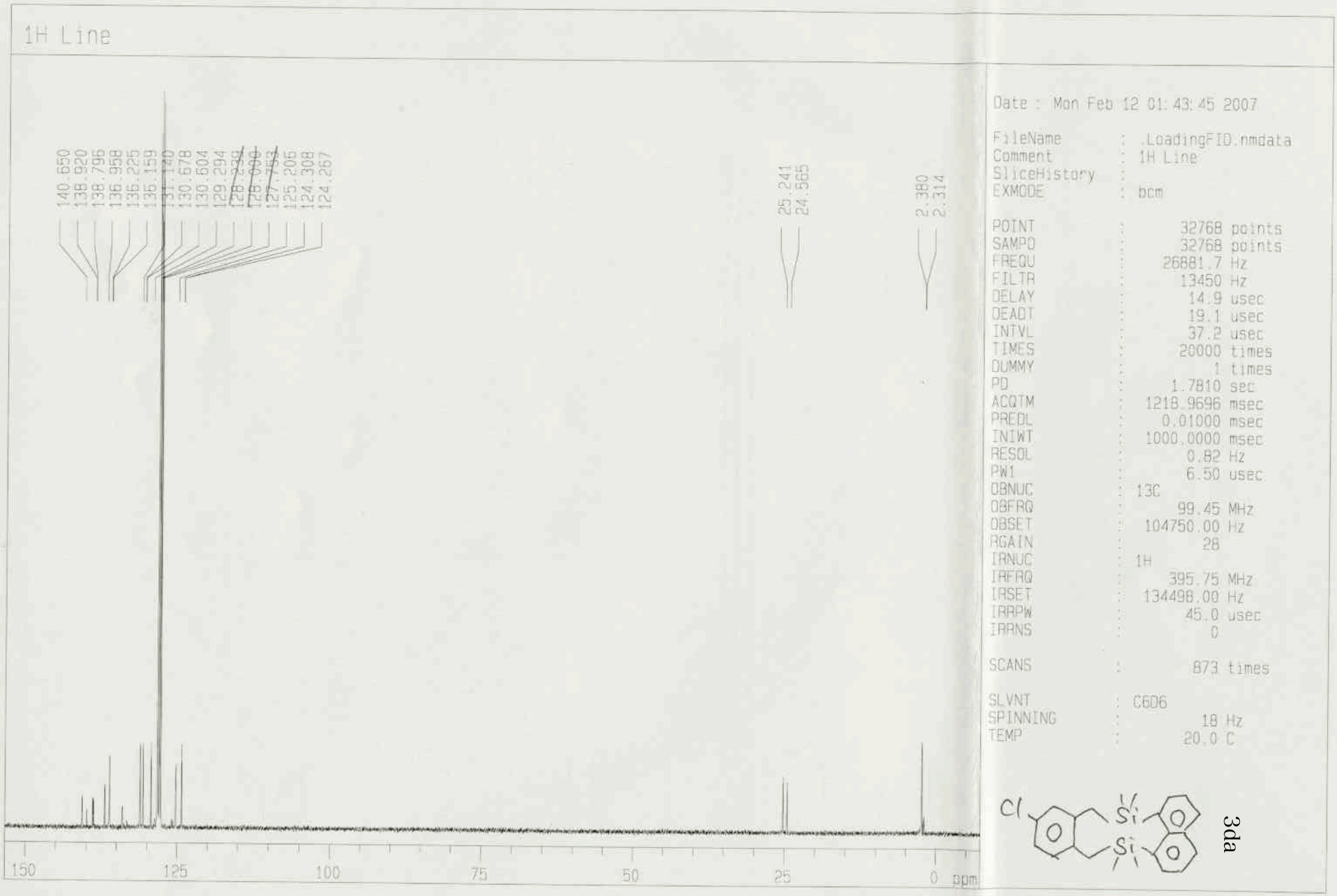


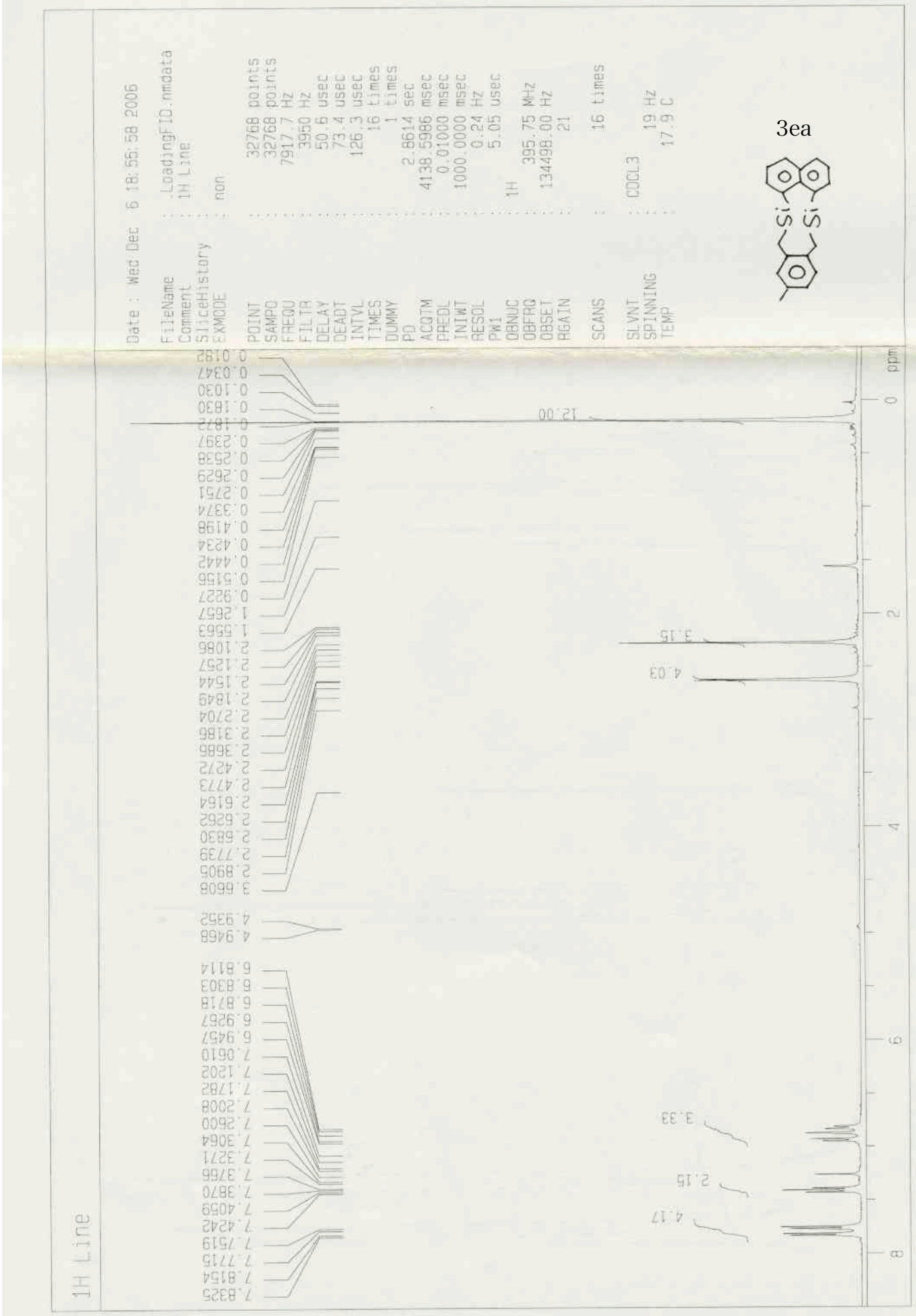
08-MAR-95 13:46:28
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 10766
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 28.6 c
SLVNT C6D6
EXREF 128.00 ppm
BF 1.22 Hz
RGAIN 24
OPERATOR : _____

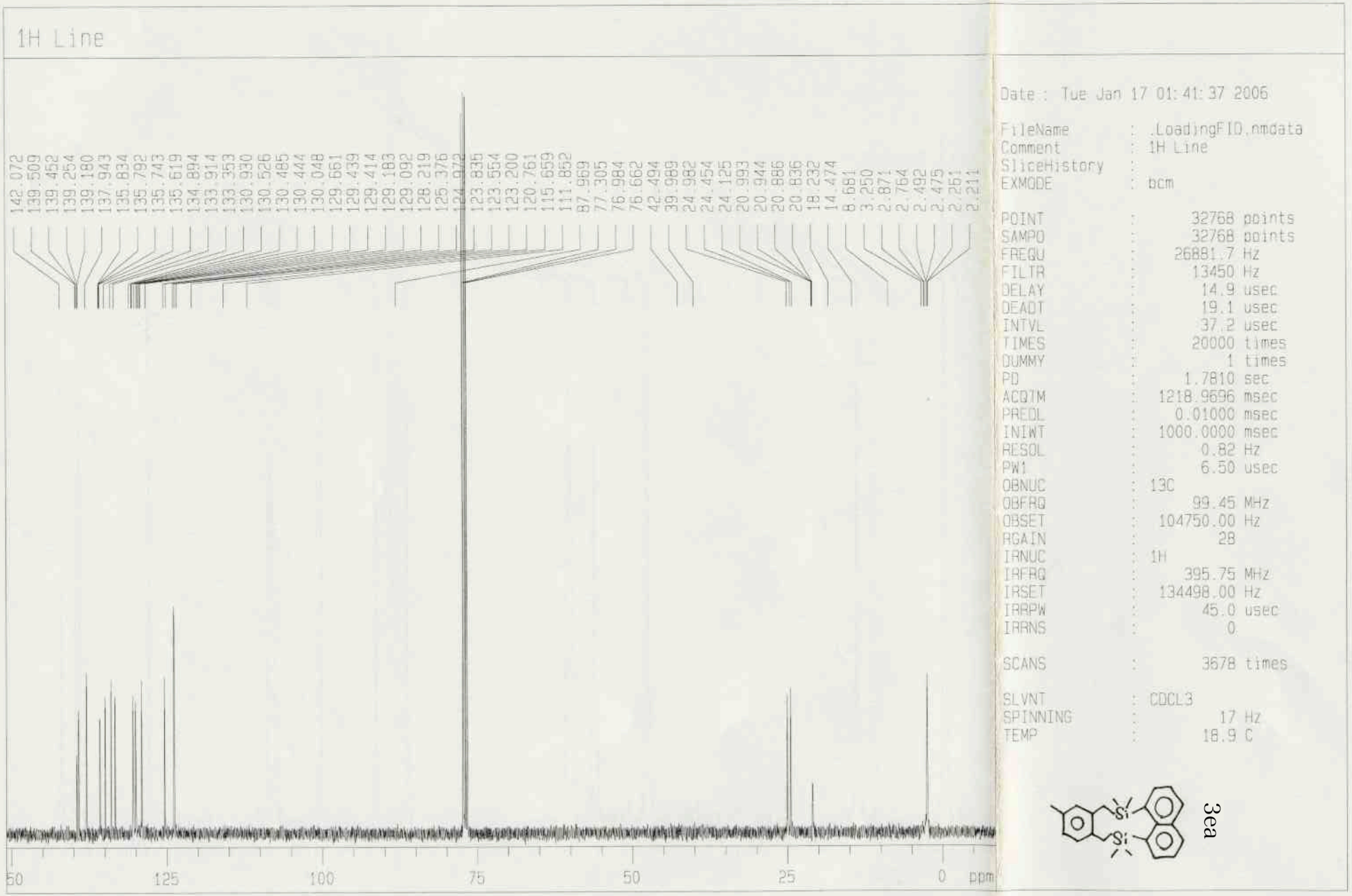


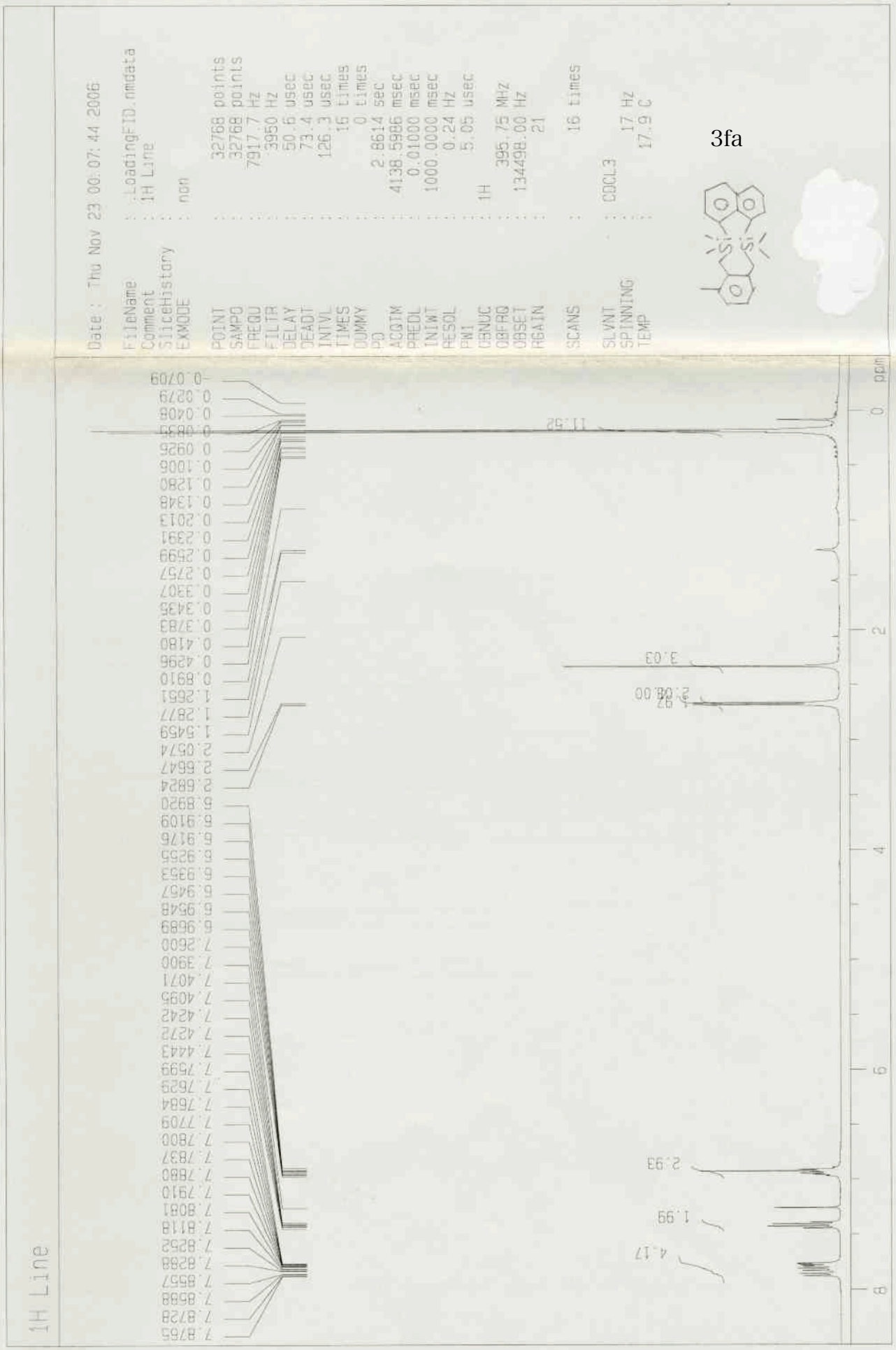
3ca

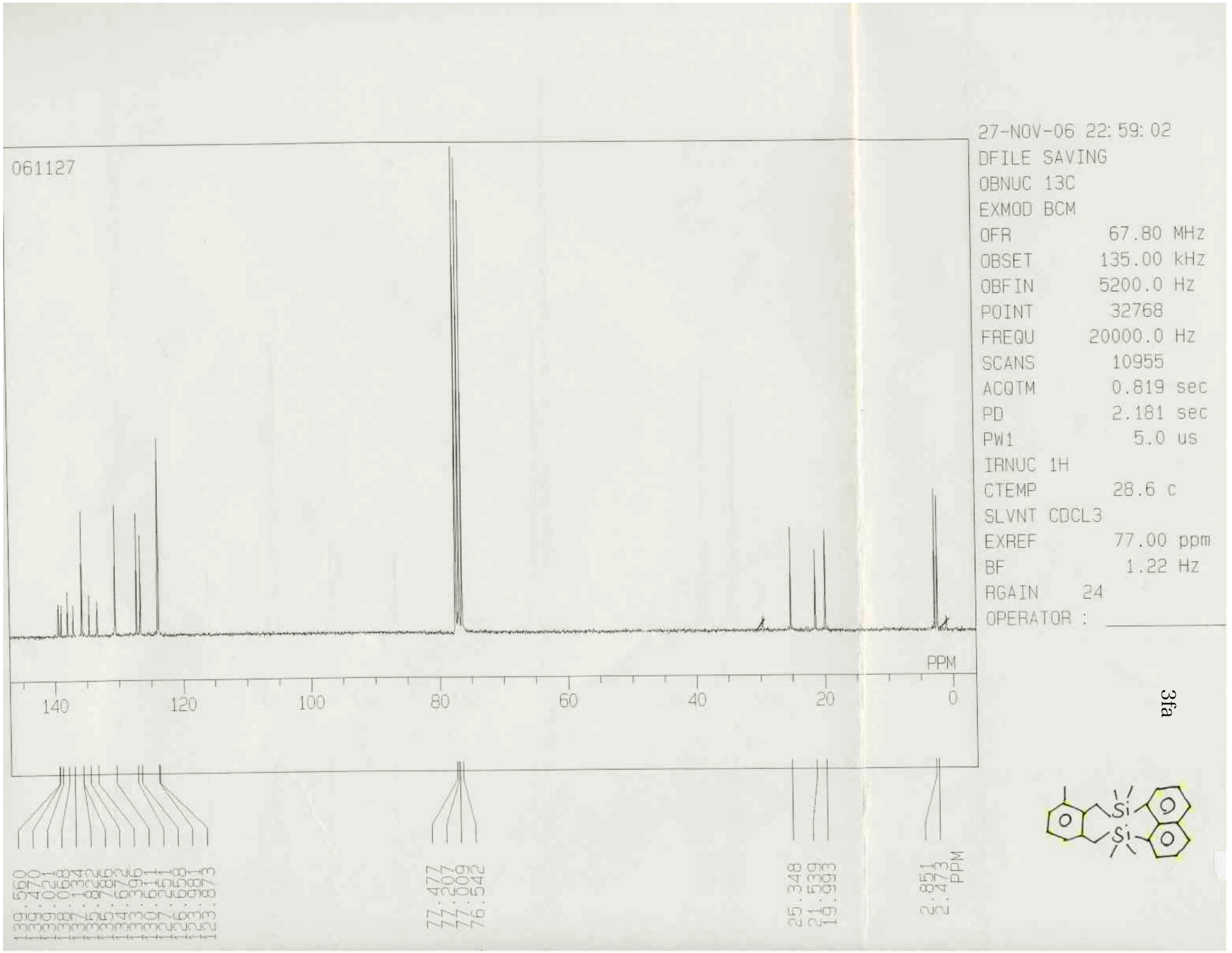

C:¥WinLambda¥COMMONY_DEFAULT.ALS
7.8337
7.8288
7.8166
7.8118
7.7849
7.7654
7.4358
7.4309
7.4163
7.4114
7.3992
7.3943
7.3796
7.2600
7.0574
6.9744
2.6457
2.6335
1.5471
0.5217
0.2117
0.2019
0.1677
0.1604
0.1213
0.1140
0.1042
12.00
3.93
2.00
0.11
0.88
1.93
3.97
PPM
8
6
4
2
0
DFILE _DEFAULT.ALS
COMNT
DATIM Wed Jul 23 18:23:36 2008
OBNUC 1H
EXMOD non
OBFRQ 395.75 MHz
OBSET 0.00 KHz
OBFIN 134498.00 Hz
POINT 8192
FREQU 7917.66 Hz
SCANS 8
ACQTM 1.0346 sec
PD 5.9654 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 11
Si
Si
3da


1H Line
140.650
138.920
138.796
136.958
136.225
135.159
131.140
130.678
130.604
129.294
128.239
128.000
127.753
125.205
124.308
124.267
25.241
24.565
2.380
2.314
150
125
100
75
50
25
0 ppm
Date : Mon Feb 12 01:43:45 2007
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 873 times
SLVNT : C6D6
SPINNING : 18 Hz
TEMP : 20.0 C
Cl
Si
Si
O
O
3da


3ea
Si
Si
1H Line
Date : Wed Dec 6 18:55:58 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory : non
EXMODE
POINT 32768 points
SAMPO 32768 points
FREQU 7917.7 Hz
FILTR 3950 Hz
DELAY 50.6 usec
DEADT 73.4 usec
INTVL 126.3 usec
TIMES 16 times
DUMMY 1 times
PD 2.8614 sec
ACQTM 4138.5986 msec
PREDL 0.01000 msec
INIWT 1000.0000 msec
RESOL 0.24 Hz
PW1 5.05 usec
OBNUC 1H
OBFRQ 395.75 MHz
OBSET 134498.00 Hz
RGAIN 21
SCANS 16 times
SLVNT CDCL3
SPINNING 19 Hz
TEMP 17.9 C
12.00
3.15
4.03
3.33
2.15
4.17
0 ppm
2
4
6
8

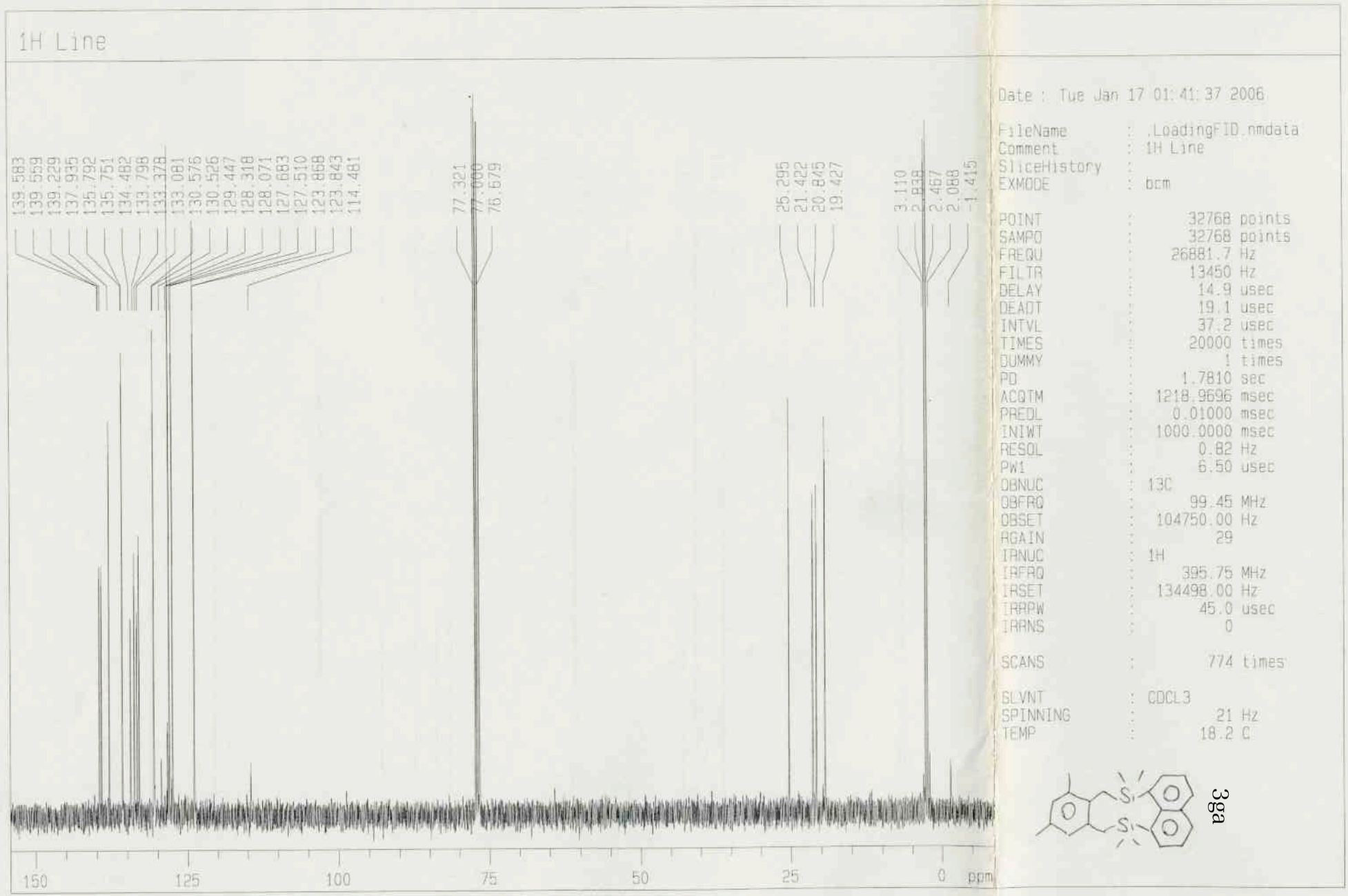

1H Line
142.072
139.509
139.452
139.254
139.180
137.943
135.834
135.792
135.743
135.619
134.894
133.914
133.353
130.930
130.526
130.485
130.444
130.048
129.661
129.439
129.414
129.183
129.092
128.219
125.376
124.972
123.835
123.554
123.200
120.761
115.659
111.852
87.969
77.305
76.984
76.662
42.494
39.989
24.982
24.454
24.125
20.993
20.944
20.886
20.836
18.232
14.474
8.681
3.250
2.871
2.764
2.492
2.475
2.261
2.211
50
125
100
75
50
25
0
ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 3678 times
SLVNT : CDCL3
SPINNING : 17 Hz
TEMP : 18.9 C
Si
Si
3ea


1H Line
Date : Thu Nov 23 00:07:44 2006
FileName : LoadingFID.nmdata
Comment : 1H Line
SliceHistory : 1
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 0 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
CBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 21
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 17 Hz
TEMP : 17.9 C
3fa
25.11
3.03
2.93
1.99
4.17
ppm

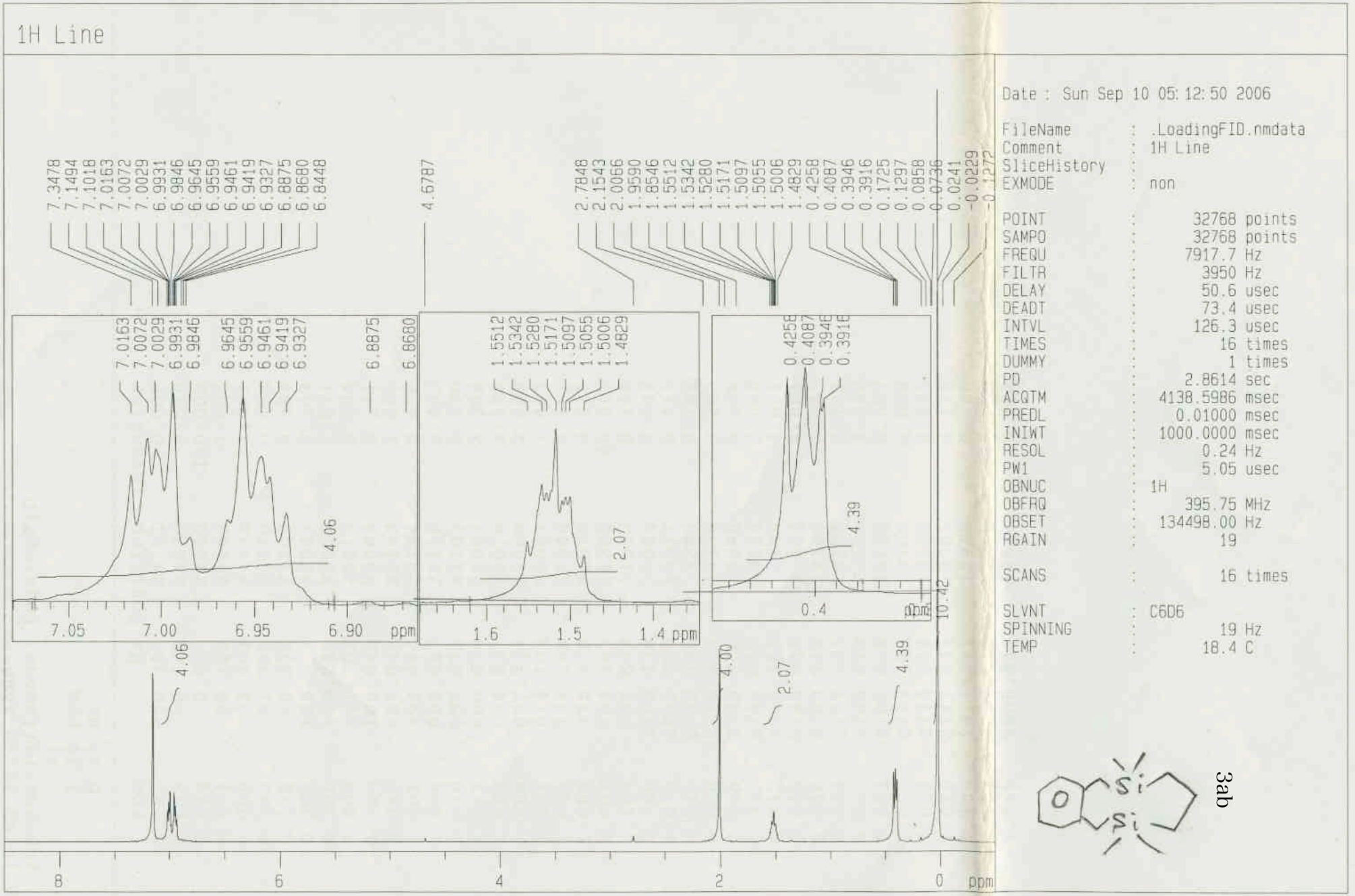

061127
27-NOV-06 22:59:02
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 10955
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 28.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 24
OPERATOR :
PPM
140
120
100
80
60
40
20
0
77.477
77.207
76.542
25.348
21.539
19.963
2.851
2.473
PPM
Si
Si
O
O
O

3fa

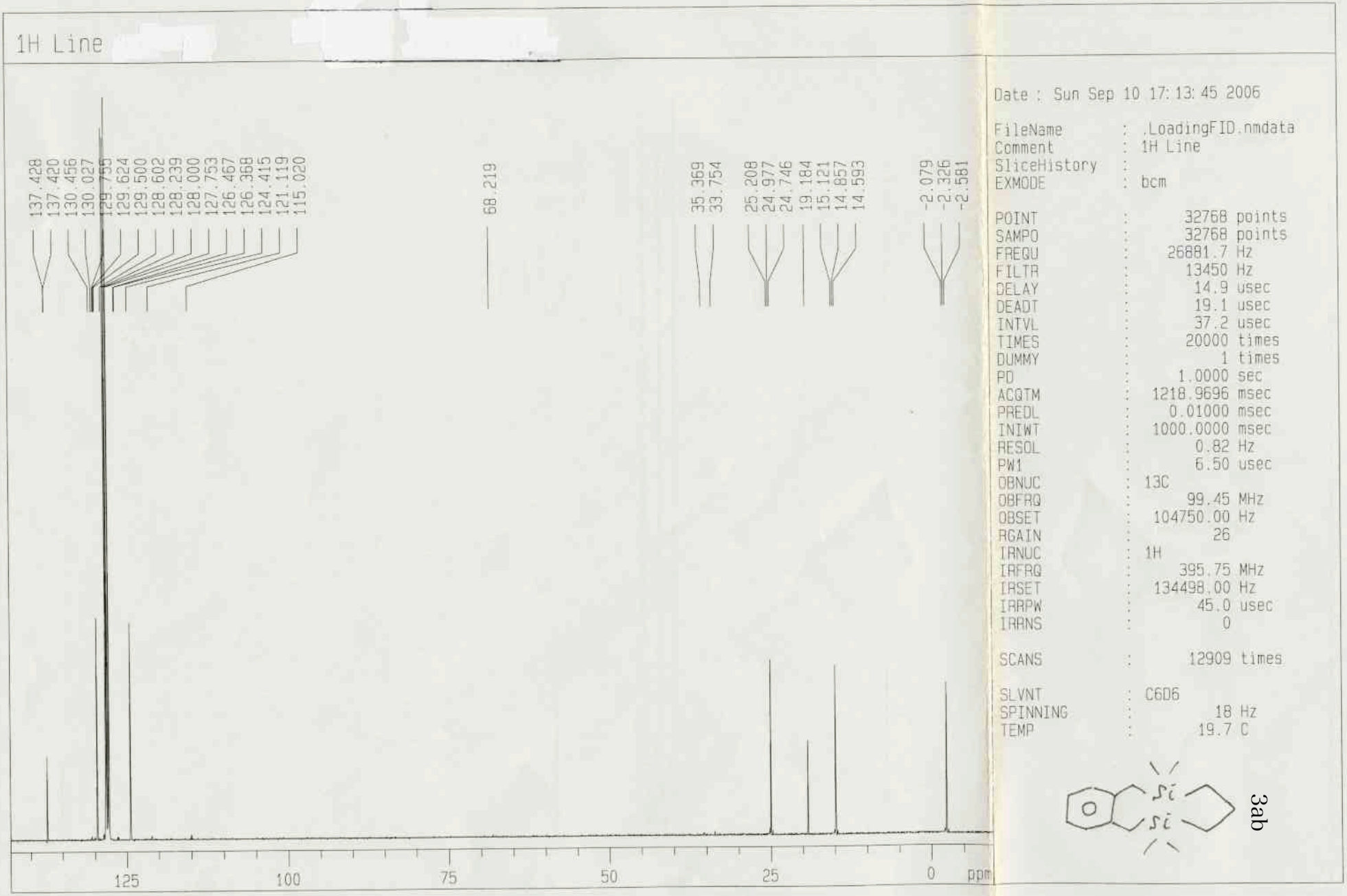
3ga
Si
Si
1H Line
Date : Tue Nov 28 05:48:38 2006
FileName : LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 16
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 19 Hz
TEMP : 18.6 C
9.89
3.50
1.88
2.57
3.89
ppm


1H Line
139.583
139.559
139.229
137.935
135.792
135.751
134.482
133.798
133.378
133.081
130.576
130.526
129.447
128.318
128.071
127.683
127.510
123.868
123.843
114.481
77.321
77.000
76.679
25.295
21.422
20.845
19.427
3.110
2.838
2.467
2.088
-1.415
150
125
100
75
50
25
0
ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 29
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 774 times
SLVNT : CDCL3
SPINNING : 21 Hz
TEMP : 18.2 C

3ga


1H Line
7.3478
7.1494
7.1018
7.0163
7.0072
7.0029
6.9931
6.9846
6.9645
6.9559
6.9461
6.9419
6.9327
6.8875
6.8680
6.8448
4.6787
2.7848
2.1543
2.0066
1.9590
1.8546
1.5512
1.5342
1.5280
1.5171
1.5097
1.5055
1.5006
1.4829
0.4258
0.4087
0.3946
0.3916
0.1725
0.1297
0.0858
0.0736
0.0241
-0.0229
-0.1272
4.06
2.07
4.39
10.42
4.00
7.05
7.00
6.95
6.90
ppm
1.6
1.5
1.4 ppm
0.4
8
6
4
2
0 ppm
Date : Sun Sep 10 05:12:50 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 19
SCANS : 16 times
SLVNT : C6D6
SPINNING : 19 Hz
TEMP : 18.4 C
O
Si
Si

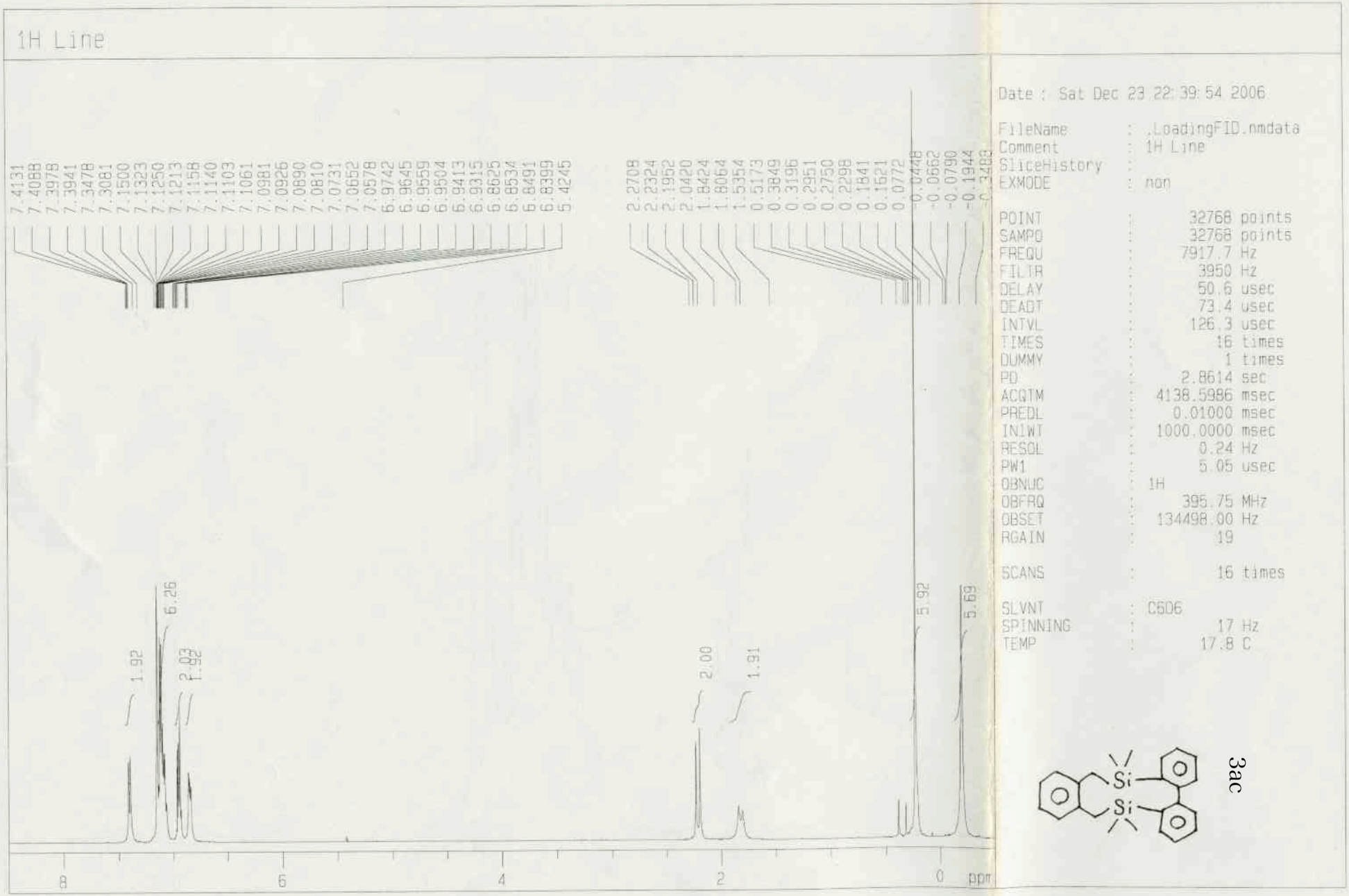
3ab

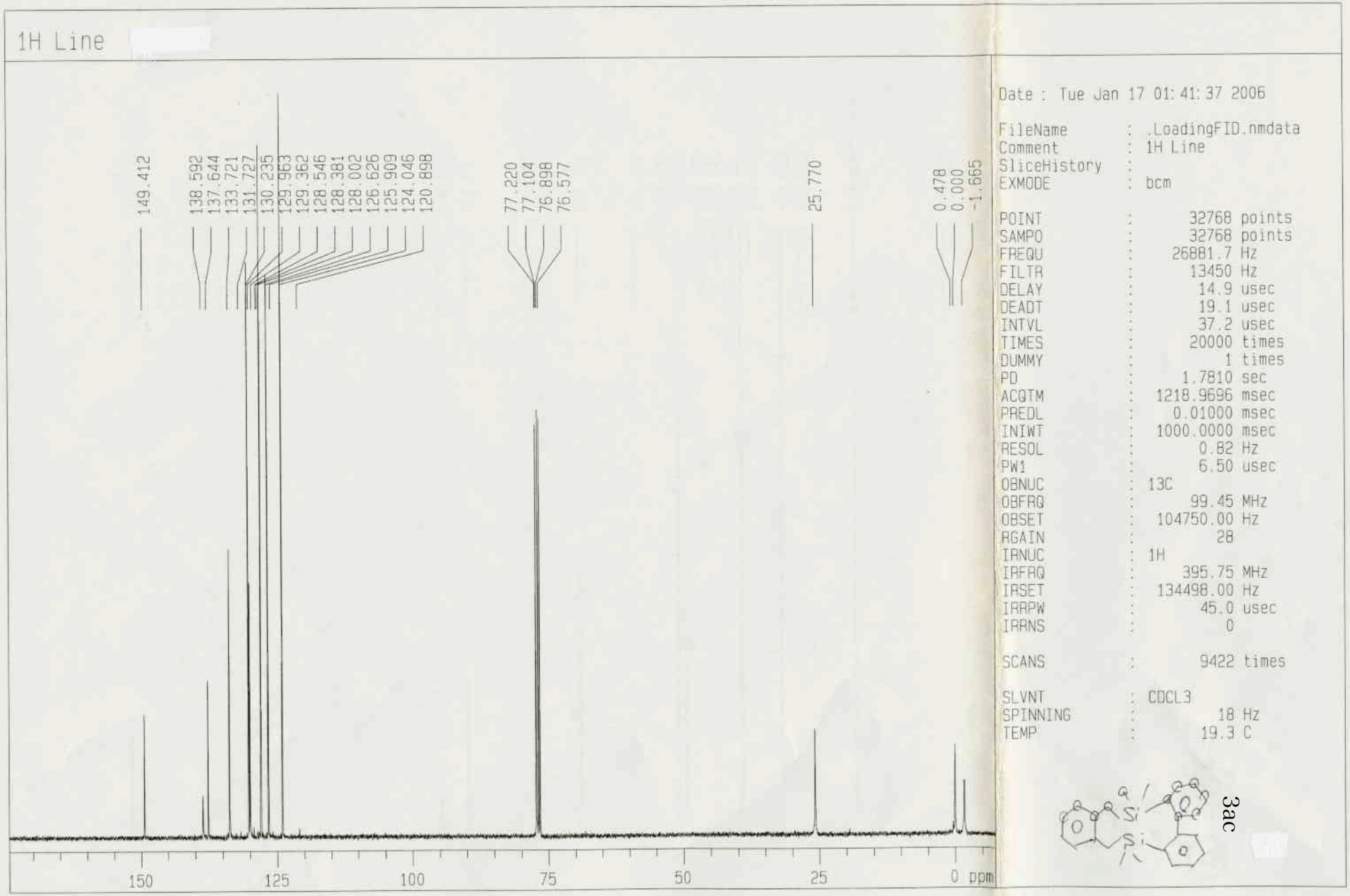

1H Line
137.428
137.420
130.456
130.027
129.755
129.624
129.500
128.602
128.239
128.000
127.753
126.467
126.368
124.415
121.119
115.020
68.219
35.369
33.754
25.208
24.977
24.746
19.184
15.121
14.857
14.593
-2.079
-2.326
-2.581
125
100
75
50
25
0
ppm
Date : Sun Sep 10 17:13:45 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 12909 times
SLVNT : C6D6
SPINNING : 18 Hz
TEMP : 19.7 C
Si
Si
3ab

1H Line
Date : Mon Jan 22 02:45:34 2007
FileName : LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3960 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 18
SCANS : 16 times
SLVNT : C6D6
SPINNING : 20 Hz
TEMP : 19.4 C
3bb
Si
Si
5.96
6.00
2.03
2.04
2.13
2.09
2.05
2.08
4.07
4.38
ppm


1H Line
144.367
142.274
138.260
135.335
130.316
128.619
128.239
128.124
128.000
127.753
127.308
126.624
123.896
24.771
21.813
19.225
16.744
13.439
-1.411
-2.260
150
125
100
75
50
25
0
ppm
Date : Mon Feb 12 01:43:45 2007
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 859 times
SLVNT : C6D6
SPINNING : 19 Hz
TEMP : 18.9 C

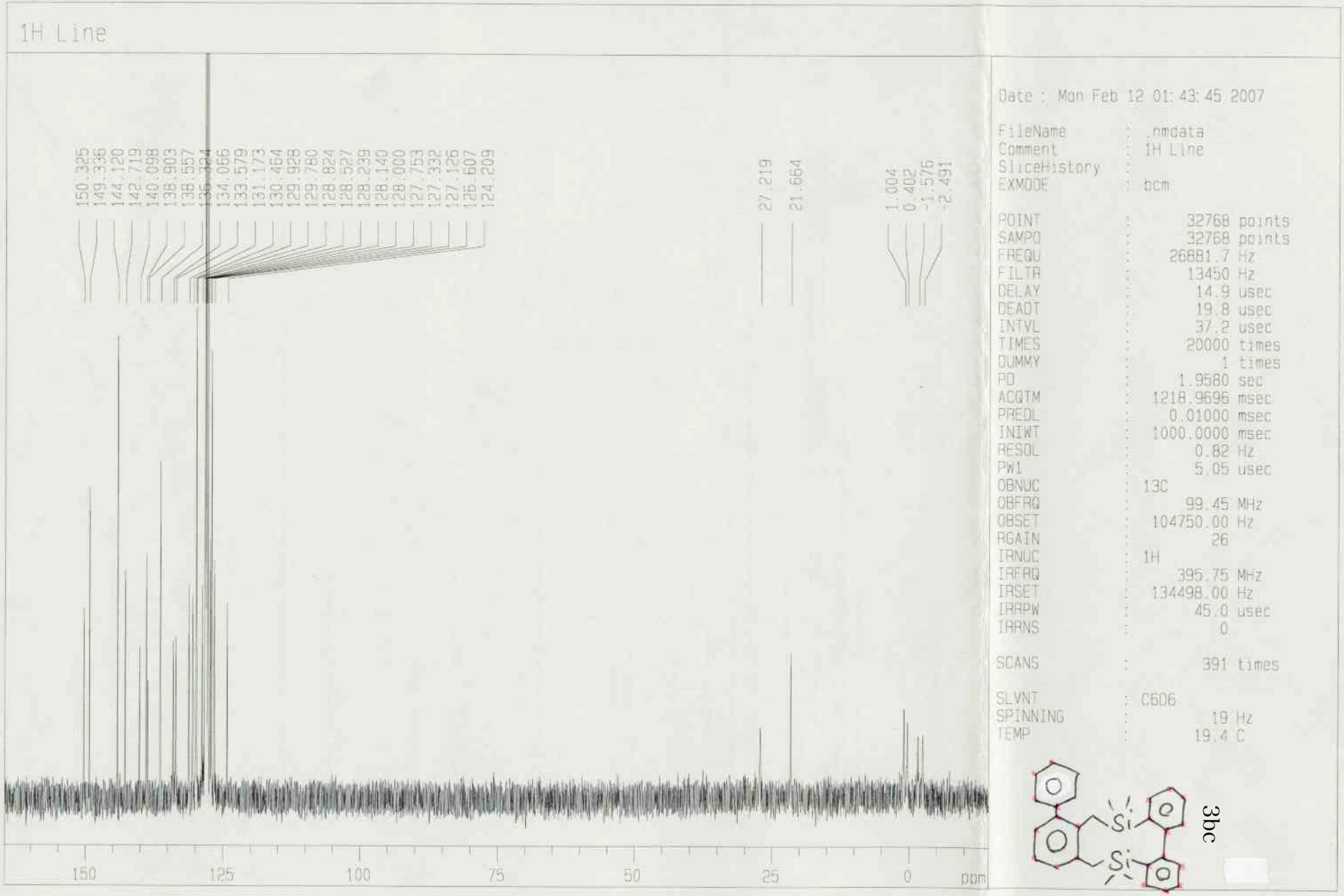
3bb

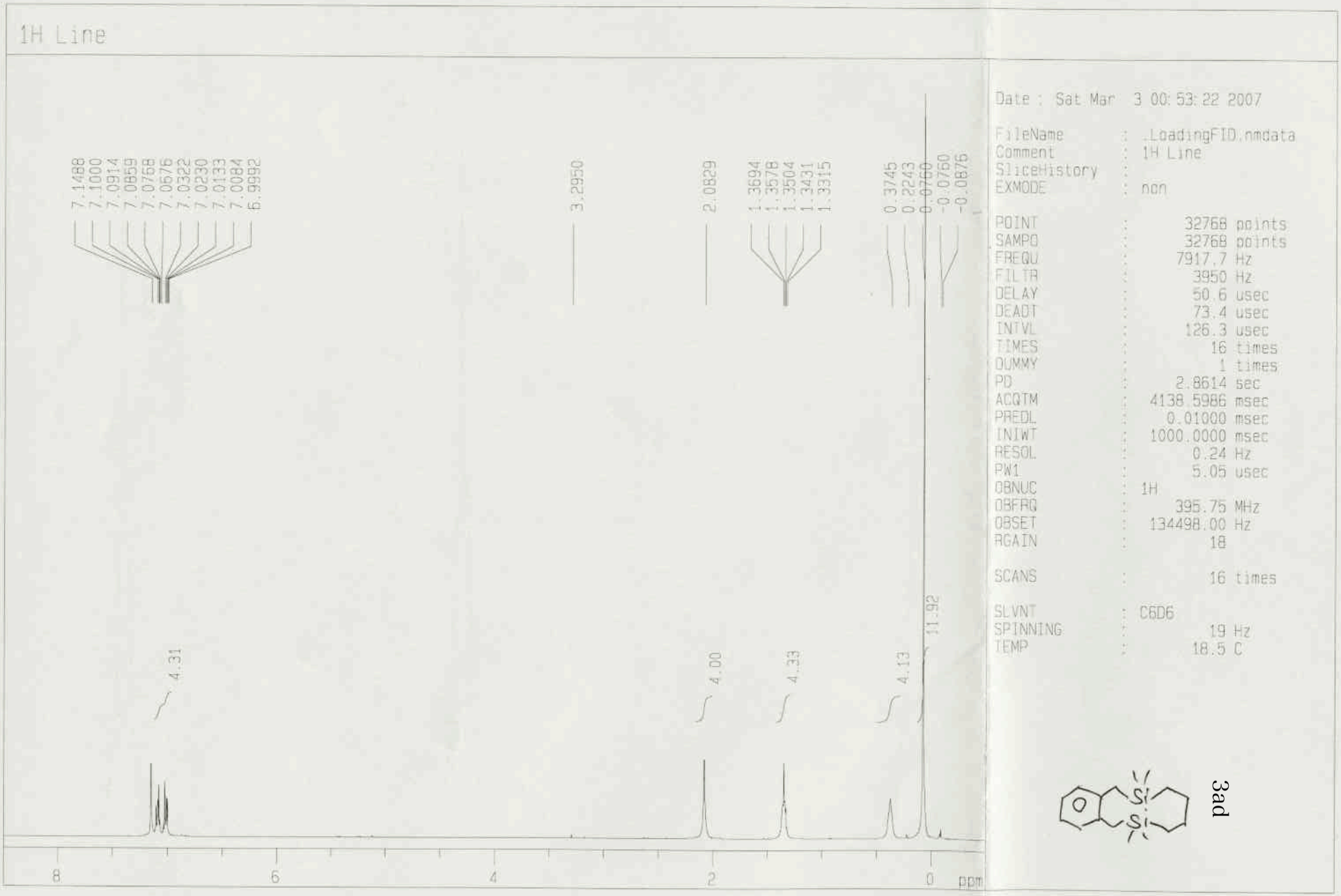

1H Line
7.4131
7.4088
7.3978
7.3941
7.3478
7.3081
7.1500
7.1323
7.1250
7.1213
7.1158
7.1140
7.1103
7.1061
7.0981
7.0926
7.0890
7.0810
7.0731
7.0652
7.0578
6.9742
6.9645
6.9559
6.9504
6.9413
6.9315
6.8625
6.8534
6.8491
6.8399
5.4245
2.2708
2.2324
2.1952
2.0420
1.8424
1.8064
1.5354
0.5173
0.3849
0.3196
0.2951
0.2750
0.2298
0.1841
0.1521
0.0772
-0.0448
-0.0662
-0.0790
-0.1944
-0.3489
1.92
6.26
2.03
1.92
2.00
1.91
5.92
5.69
8
6
4
2
0
ppm
Date : Sat Dec 23 22:39:54 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 19
SCANS : 16 times
SLVNT : C6D6
SPINNING : 17 Hz
TEMP : 17.8 C
Si
Si
3ac

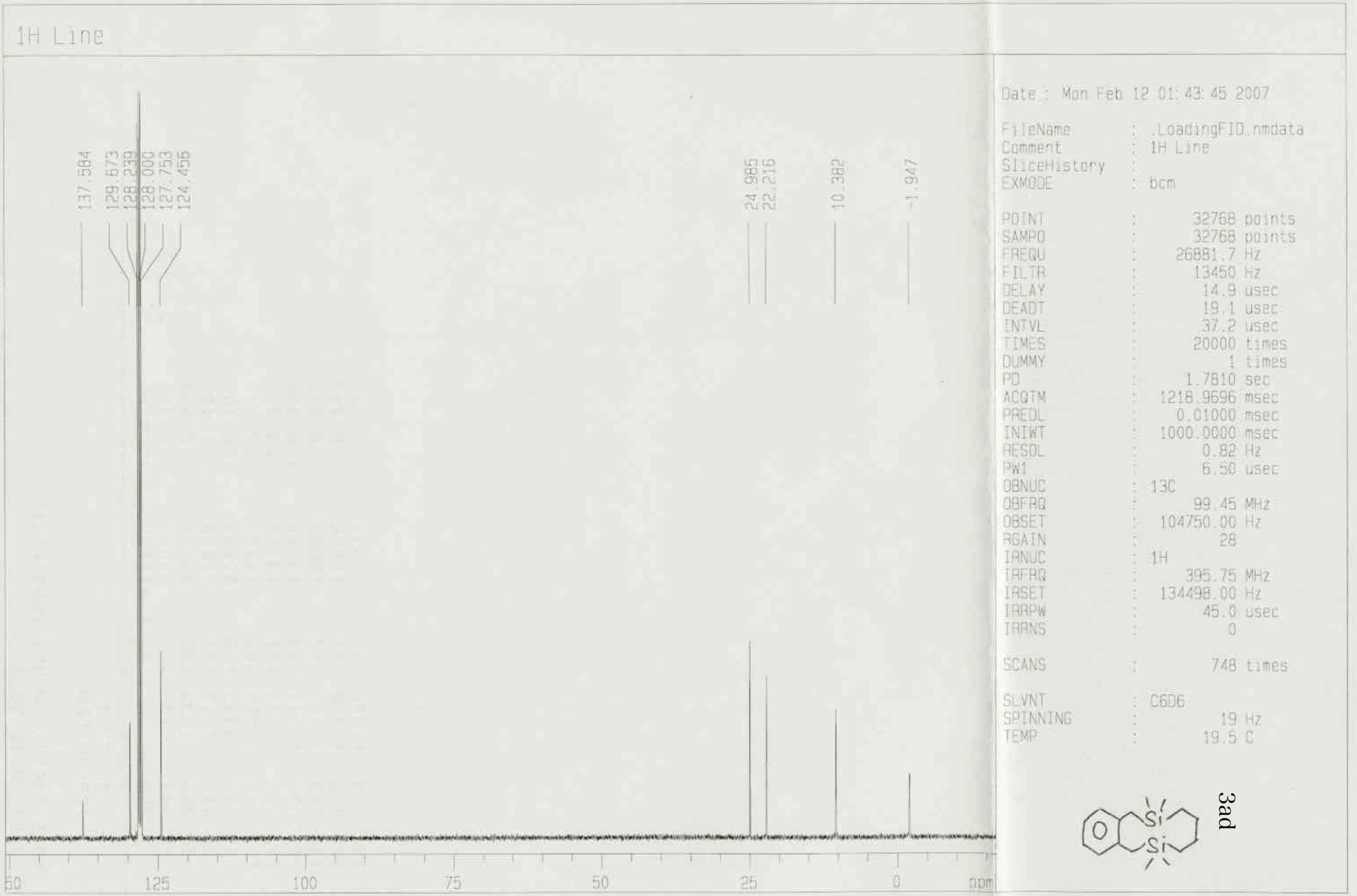

1H Line
149.412
138.592
137.644
133.721
131.727
130.235
129.963
129.362
128.546
128.381
128.002
126.626
125.909
124.046
120.898
77.220
77.104
76.898
76.577
25.770
0.478
0.000
-1.665
150
125
100
75
50
25
0 ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 9422 times
SLVNT : CDCL3
SPINNING : 18 Hz
TEMP : 19.3 C
3ac

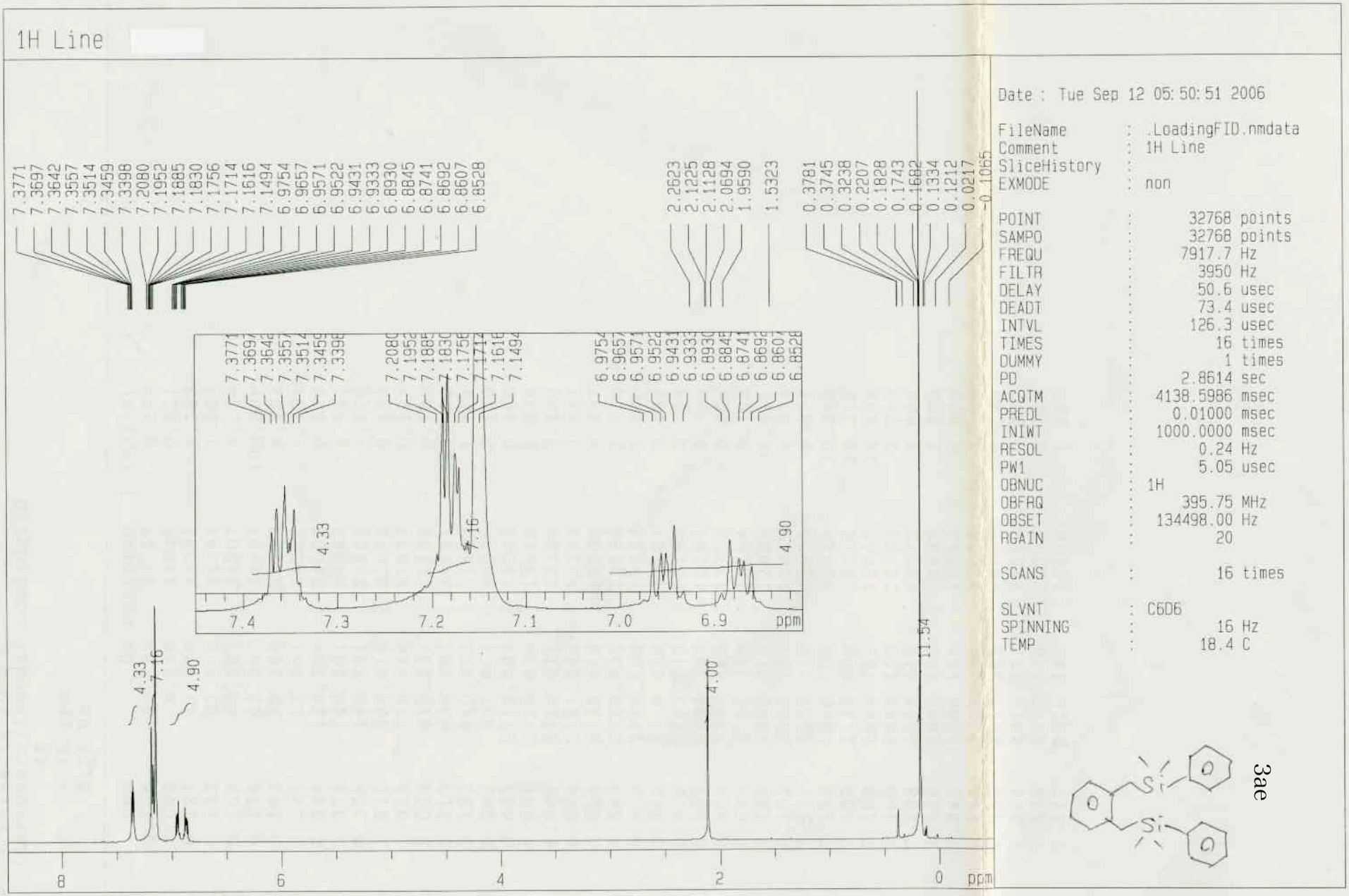
1H Line
Date : Mon Feb 26 06:56:45 2007
FileName : .LoadingFID.nmrdata
Comment : 1H Line
SliceHistory : 1
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.3 usec
INTVL : 126.3 usec
TIMES : 4 times
DUMMY : 4 times
PD : 2.9000 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134998.00 Hz
RGAIN : 18
SCANS : 4 times
SLVNT : C6D6
SPINNING : 22 Hz
TEMP : 18.7 C
2.94
3.00
3.01
3.29
0.98
2.00
1.00
2.11
ppm
0
2
4
6
8

3bc

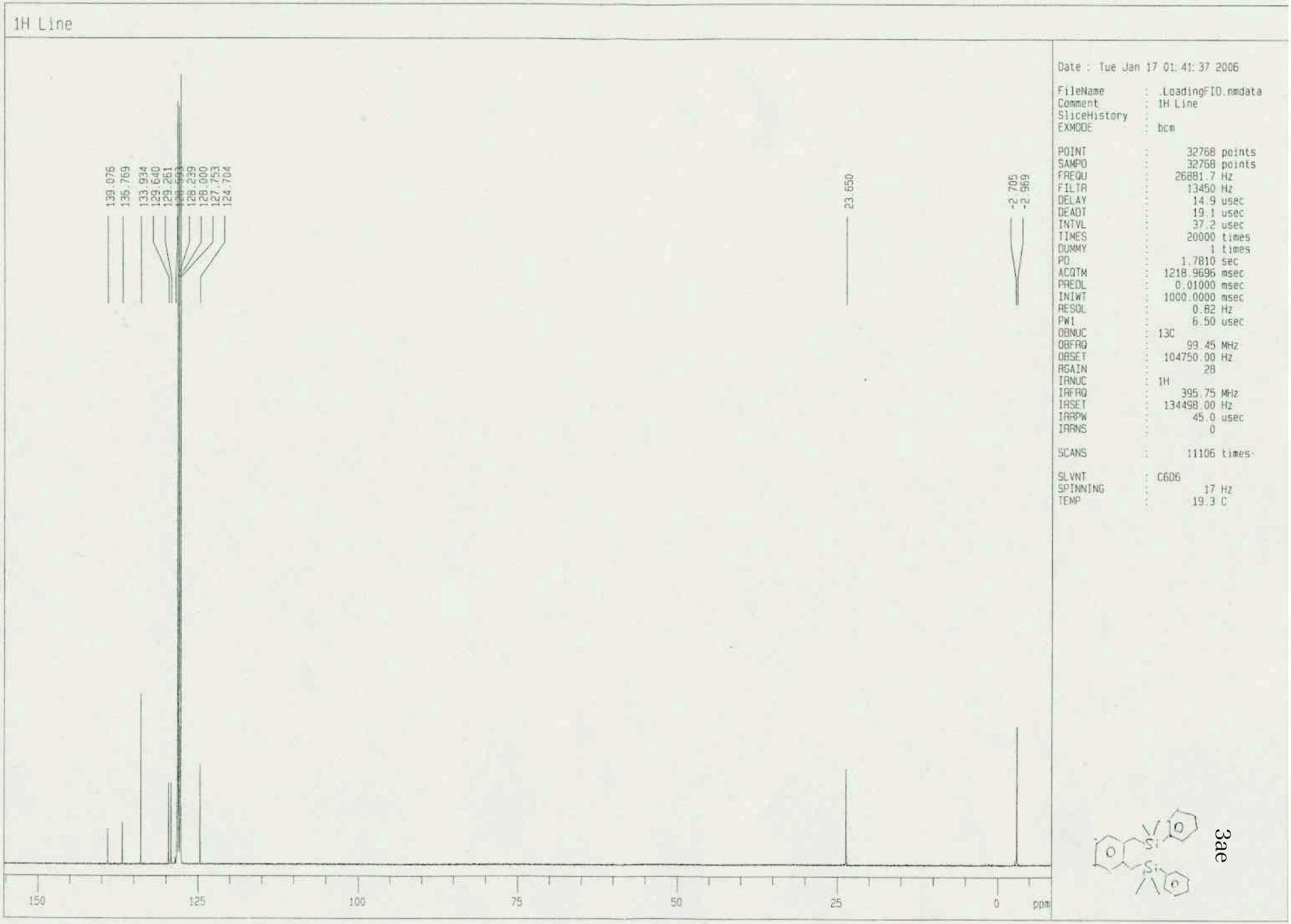

1H Line
150.325
149.336
144.120
142.719
140.098
138.903
138.557
135.324
134.066
133.579
131.173
130.464
129.928
129.760
128.824
128.527
128.239
128.140
128.000
127.753
127.332
127.126
125.607
124.209
27.219
21.664
1.004
0.402
-1.576
-2.491
150
125
100
75
50
25
0
ppm
Date : Mon Feb 12 01:43:45 2007
FileName : .nmdata
Comment : 1H Line
SliceHistory :
EXMODE : acm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.8 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.9580 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 5.05 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 391 times
SLVNT : C6D6
SPINNING : 19 Hz
TEMP : 19.4 C
Si
Si
3bc


1H Line
7.1488
7.1000
7.0914
7.0859
7.0758
7.0576
7.0322
7.0230
7.0133
7.0084
6.9992
3.2950
2.0829
1.3694
1.3578
1.3504
1.3431
1.3315
0.3745
0.2243
0.0760
-0.0760
-0.0876
4.31
4.00
4.33
4.13
11.92
8
6
4
2
0
ppm
Date : Sat Mar 3 00:53:22 2007
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 18
SCANS : 16 times
SLVNT : C6D6
SPINNING : 19 Hz
TEMP : 18.5 C
O
Si
Si
3ad


1H Line
137.564
129.673
128.239
128.000
127.753
124.456
24.985
22.216
10.382
-1.947
50
125
100
75
50
25
0
ppm
Date : Mon Feb 12 01:43:45 2007
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 748 times
SLVNT : C6D6
SPINNING : 19 Hz
TEMP : 19.5 C
O
Si
Si
3ad


1H Line
7.3771
7.3697
7.3642
7.3557
7.3514
7.3459
7.3398
7.2080
7.1952
7.1685
7.1830
7.1756
7.1714
7.1616
7.1494
6.9754
6.9657
6.9571
6.9522
6.9431
6.9333
6.8930
6.8845
6.8741
6.8692
6.8607
6.8528
2.2623
2.1225
2.1128
2.0694
1.9590
1.5323
0.3781
0.3745
0.3238
0.2207
0.1828
0.1743
0.1682
0.1334
0.1212
0.0217
-0.1065
4.33
7.16
4.90
4.00
11.54
7.4
7.3
7.2
7.1
7.0
6.9
ppm
8
6
4
2
0
ppm
Date : Tue Sep 12 05:50:51 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 73.4 usec
INTVL : 126.3 usec
TIMES : 16 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.05 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 20
SCANS : 16 times
SLVNT : C6D6
SPINNING : 16 Hz
TEMP : 18.4 C
Si
Si
O
O
O
O

3ae


1H Line
139.076
136.769
133.934
129.640
129.261
128.239
128.000
127.753
124.704
23.650
-2.705
-2.969
150
125
100
75
50
25
0
ppm
Date : Tue Jan 17 01:41:37 2006
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IANUC : 1H
IAFRQ : 395.75 MHz
IASET : 134498.00 Hz
IARPW : 45.0 usec
IARNS : 0
SCANS : 11106 times
SLVNT : C6D6
SPINNING : 17 Hz
TEMP : 19.3 C
O
Si
Si
O
O

3ae