

Aromatic C–H silylation of arenes with 1-hydrosilatrane catalyzed by an iridium(I)/2,9-dimethylphenanthroline (dmphen) complex

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General. All the experiments were carried out under a nitrogen atmosphere. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 solutions using a JEOL JNM-A400II spectrometer (400 or 100 MHz) and Me_4Si or residual protiated solvent as an internal standard. Low- and high-resolution mass spectra were obtained on a JEOL JMS-DX303. GC analyses were conducted on a Hitachi G-3500 instrument equipped with a glass column (OV-101 on Uniport B, 2 m). $[\text{Ir}(\text{OMe})(\text{cod})]_2$,¹ 2,9-diisopropyl-1,10-phenanthroline,² 2,9-di-*tert*-butyl-1,10-phenanthroline,² 1-hydro-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane,³ and 2,9-dimethyl-4,7-dichloro-1,10-phenanthroline⁴ were synthesized by the reported procedure. 2,9-Dimethyl-4,7-bis(dimethylamino)-1,10-phenanthroline and 2,9-dimethyl-4,7-dimethoxy-1,10-phenanthroline were prepared by the methods similar to those for 4,7-bis(dimethylamino)-1,10-phenanthroline⁵ and 4,7-dimethoxy-1,10-phenanthroline,⁵ respectively. 2,9-Dimethyl-4,7-bis(trifluoromethyl)-1,10-phenanthroline was obtained by chlorine-iodine exchange⁶ of 2,9-dimethyl-4,7-dichloro-1,10-phenanthroline and coupling⁷ of the iodide with in situ generated (trifluoromethyl)copper. Arenes were purified by distillation from appropriate drying agents. All of other compounds were used as received.

2,9-Dimethyl-4,7-bis(dimethylamino)-1,10-phenanthroline. ^1H NMR (400 MHz, CDCl_3) δ 2.84 (s, 6H), 3.03 (s, 12H), 6.88 (s, 2H), 7.89 (s, 2H); ^{13}C NMR (100 MHz) δ 26.32, 44.11, 109.93, 119.85, 120.64, 147.34, 157.72, 158.92; LRMS (EI) m/z 294 (M^+ , 100), 279 (9.5), 263 (6.3), 250 (4.9), 236 (3.6), 147 (5.9), 139 (4.5); HRMS (EI) calcd for $\text{C}_{18}\text{H}_{22}\text{N}_4$ 294.1844, found 294.1833.

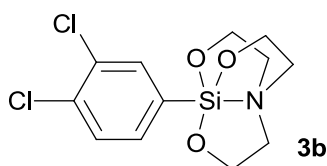
2,9-Dimethyl-4,7-dimethoxy-1,10-phenanthroline. ^1H NMR (400 MHz, CDCl_3) δ 2.89 (s, 6H), 4.07 (s, 6H), 6.86 (s, 2H), 8.08 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 26.63, 55.66, 102.74, 117.95, 119.42, 145.97, 160.26, 162.28; LRMS (EI) m/z 268 (M^+ , 100), 253 (14.1), 225 (22.8), 210 (4), 182 (7.5), 134 (4.9); HRMS (EI) calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2$ 268.1212, found 268.1207.

2,9-Dimethyl-4,7-bis(trifluoromethyl)-1,10-phenanthroline. ^1H NMR (400 MHz, CDCl_3) δ 3.05 (s, 6H), 7.86 (s, 2H), 8.19 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 26.13, 120.81 (q, $J = 5.2$ Hz),

121.90 (q, $J = 1.7$ Hz), 122.78 (q, $J = 1.7$ Hz), 123.30 (q, $J = 274.2$ Hz), 134.74 (q, $J = 319.4$), 146.09, 159.94; LRMS (EI) m/z 344 (M^+ , 100), 325 (4.7), 274 (6.7), 172 (5.5), 40 (9.3); HRMS (EI) calcd for $C_{16}H_{10}N_2F_6$ 344.0748, found 344.0750.

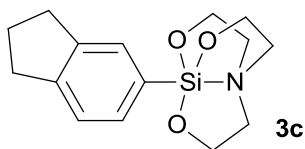
General procedure for the C–H silylation (Table 2). An oven-dried flask fitted with a condenser and a nitrogen bubbler was charged with 1-hydrosilatrane (1.0 mmol), $[\{Ir(OMe)(cod)\}_2]$ (0.015 mmol) and dmphen (0.03 mmol), and then flushed with nitrogen. Under a positive flow of nitrogen, an arene (60 mmol) was added. The reaction mixture was stirred at 120°C for 32 h. The product was isolated by Kugelrohr distillation to give an analytically pure sample.

1-(3,4-Dichlorophenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3b).



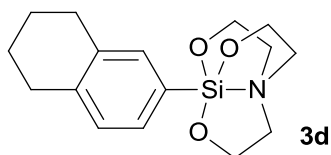
1H NMR (400 MHz, $CDCl_3$) δ 2.92(t, $J = 5.9$ Hz, 6 H), 3.89 (t, $J = 5.9$ Hz, 6 H), 7.32 (d, $J = 7.8$ Hz, 1 H), 7.53 (dd, $J = 1.5, 7.8$ Hz, 1H), 7.79 (d, $J = 1.5$ Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.95, 57.48, 129.22, 131.23, 131.28, 133.57, 136.19, 143.66; LRMS (EI) m/z 319 (M^+ , 8), 174 (100); HRMS (EI) calcd for $C_{12}H_{15}NO_3SiCl_2$ 319.0198, found 319.0198.

1-(5-Indanyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3c).



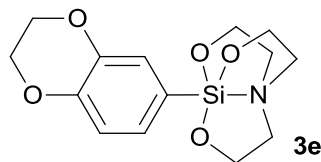
1H NMR (400 MHz, $CDCl_3$) δ 1.94-2.01 (m, 2 H), 2.81-2.90 (m, 7 H), 3.88 (t, $J = 5.9$ Hz, 6 H), 7.13 (d, $J = 7.3$ Hz, 1 H), 7.50 (d, $J = 7.3$ Hz, 1 H), 7.59 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.31, 32.76, 32.81, 51.08, 57.80, 123.33, 129.81, 131.82, 138.77, 142.82, 143.61; LRMS (EI) m/z 291 (M^+ , 19.7), 174 (100); HRMS (EI) calcd for $C_{15}H_{21}NO_3Si$ 291.1291, found 291.1293.

1-(6-Tetralinyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3d).



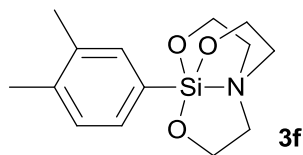
^1H NMR (400 MHz, CDCl_3) δ 1.72-1.75 (m, 4 H), 2.69 (br s, 2 H), 2.75 (br s, 2 H), 2.89 (t, $J = 5.9$ Hz, 6 H), 3.88 (t, $J = 5.9$ Hz, 6 H), 6.96 (d, $J = 7.3$ Hz, 1 H), 7.41 (s, 1 H), 7.42 (d, $J = 7.8$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 23.45, 23.56, 29.40, 29.49, 51.18, 57.86, 128.10, 131.14, 134.74, 135.65, 136.63, 137.98; LRMS (EI) m/z 305 (M^+ , 18.5), 174 (100); HRMS (EI) calcd for $\text{C}_{16}\text{H}_{23}\text{NO}_3\text{Si}$ 305.1447, found 305.1449.

1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3e).



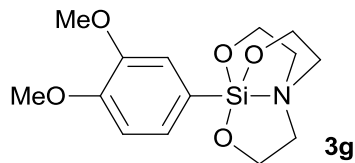
^1H NMR (400 MHz, CDCl_3) δ 2.90 (t, $J = 5.9$ Hz, 6 H), 3.88 (t, $J = 5.9$ Hz, 6 H), 4.20 (s, 3 H), 6.78 (d, $J = 8.0$ Hz, 1 H), 7.20 (dd, $J = 1.5$ and 8.0 Hz, 1 H), 7.25 (d, $J = 1.5$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 51.06, 57.72, 64.26, 64.53, 116.28, 123.06, 127.38, 134.79, 142.78, 143.42; LRMS(EI) m/z 311 (M^+ , 59.2), 174 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{21}\text{NO}_5\text{Si}$ 309.1032, found 309.1031.

1-(3,4-Dimethylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3f).



^1H NMR (400 MHz, CDCl_3) δ 2.19 (s, 3 H), 2.29 (s, 3 H), 2.87 (t, $J = 6.0$ Hz, 6 H), 3.87 (t, $J = 5.9$ Hz, 6 H), 7.03 (d, $J = 7.3$ Hz, 1 H), 7.44 (d, $J = 7.6$ Hz, 1 H), 7.47 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.68, 19.79, 51.11, 57.83, 128.66, 131.67, 134.96, 135.28, 135.85, 138.67; LRMS (EI) m/z 279 (M^+ , 23), 236 (3.2), 206 (3.1), 174 (100), 119 (3); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{21}\text{O}_3\text{NSi}$ 279.1291, found 279.1301.

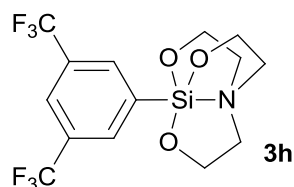
1-(3,4-Dimethoxyphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3g).



^1H NMR (400 MHz, CDCl_3) δ 2.92 (t, $J = 6.1$ Hz, 6 H), 3.84 (s, 3 H), 3.90 (t, $J = 5.9$ Hz, 6 H), 3.91 (s, 3 H), 6.83 (d, $J = 7.8$, 1 H), 7.28 (d, $J = 1.5$ Hz, 1 H), 7.30 (dd, $J = 1.4$ and 7.8 Hz, 1 H); ^{13}C NMR (100

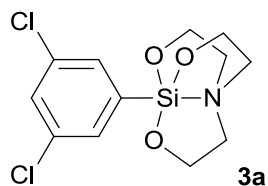
MHz, CDCl₃) δ 51.09, 55.59, 55.68, 57.81, 110.78, 116.65, 127.11, 133.67, 147.98, 148.88; LRMS(EI) m/z 311 (M⁺, 59.2), 174 (100); HRMS (EI) calcd for C₁₄H₂₁NO₅Si 311.1189, found 311.1194.

1-[3,5-Bis(trifluoromethyl)phenyl]-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3h).



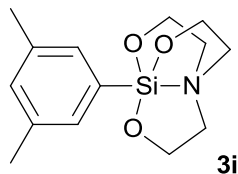
¹H NMR (400 MHz, CDCl₃) δ 2.95 (t, *J* = 6.1 Hz, 6 H), 3.92 (t, *J* = 6.1 Hz, 6 H), 7.71 (s, 1 H), 8.18 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 50.97, 57.44, 121.09-121.25 (m), 124.18 (q, *J* = 272.3 Hz), 129.45 (q, *J* = 31.9 Hz), 134.25-134.50 (m), 146.26; LRMS (EI) m/z 387 (M⁺, 3.2), 174 (100); HRMS (EI) calcd for C₁₄H₁₅NO₃F₆Si 387.0725, found 387.0722.

1-(3,5-Dichlorophenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3a).



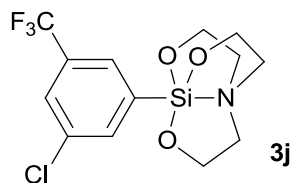
¹H NMR (400 MHz, CDCl₃) δ 2.94 (t, *J* = 6.1 Hz, 6 H), 3.91 (t, *J* = 6.1 Hz, 6 H), 7.21 (t, *J* = 2.0 Hz, 1 H), 7.58 (d, *J* = 2.0 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃) δ 51.01, 57.49, 127.42, 132.31, 133.93, 147.25; LEMS (EI) m/z 319 (M⁺, 5.4), 174 (100), 130 (3); HRMS (EI) calcd for C₁₂H₁₅NO₃SiCl₂ 319.0198, found 319.0204.

1-(3,5-Dimethylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3i).



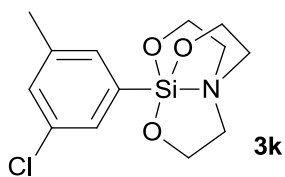
¹H NMR (400 MHz, CDCl₃) δ 2.27 (s, 6 H), 2.90 (t, *J* = 5.9 Hz, 6 H), 3.90 (t, *J* = 5.9 Hz, 6 H), 6.88 (s, 1 H), 7.35 (s, 2 H); ¹³C NMR (100 MHz, CDCl₃) δ 21.45, 51.09, 57.82, 129.58, 131.68, 136.26, 141.12; LRMS (EI) m/z 279 (M⁺, 19), 174 (100), 148 (3.1), 119 (3.1), 105 (3.2); HRMS (EI) calcd for C₁₄H₂₁O₃NSi 279.1291, found 279.1298.

1-(3-Chloro-5-trifluoromethylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3j).



^1H NMR (400 MHz, CDCl_3) δ 2.93 (t, $J = 6.1$ Hz, 6 H), 3.91 (t, $J = 5.9$ Hz, 6 H), 7.44 (s, 1 H), 7.87 (s, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ 50.94, 57.44, 124.01 (q, $J = 272.3$ Hz), 124.26 (q, $J = 4.1$ Hz), 128.97 (q, $J = 3.9$ Hz), 130.53 (q, $J = 31.9$ Hz), 133.59, 137.68, 147.12; LRMS (EI) m/z 353 (M^+ , 4), 174 (100); HRMS (EI) calcd for $\text{C}_{13}\text{H}_{15}\text{NO}_3\text{F}_3\text{SiCl}$ 353.0462, found 353.0461.

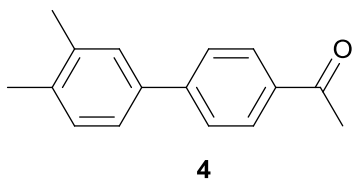
1-(3-Chloro-5-methylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (3k).



^1H NMR (400 MHz, CDCl_3) δ 2.28 (s, 3 H), 2.92 (t, $J = 5.9$ Hz, 6 H), 3.90 (t, $J = 5.9$ Hz, 6 H), 7.02 (s, 1 H), 7.40 (s, 1 H), 7.51 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.20, 50.98, 57.60, 128.28, 131.04, 132.83, 133.20, 138.38, 144.61; LRMS (EI) m/z 299 (M^+ , 10.3), 174 (100); HRMS (EI) calcd for $\text{C}_{13}\text{H}_{18}\text{NClO}_3\text{Si}$ 299.0744, found 299.0737.

Cross-coupling of 1-(3,4-dimethylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane with 4-iodoacetophenone (Scheme 3). A mixture of $\text{Pd}(\text{OAc})_2$ (0.1 mmol), PPh_3 (0.2 mmol), 1-(3,4-dimethylphenyl)-2,8,9-trioxa-5-aza-1-silabicyclo[3.3.3]undecane (2.0 mmol), 4-iodoacetophenone (1.0 mmol), and DMF (10 ml) was stirred at r.t. for 10 min. 1.0 M TBAF in THF (2.0 mmol) was added and the resulting mixture was stirred at 90 °C for 2 h. Isolation by column chromatography over silica gel gave an analytically pure sample.

4-(3,4-Dimethylphenyl)acetophenone 4.

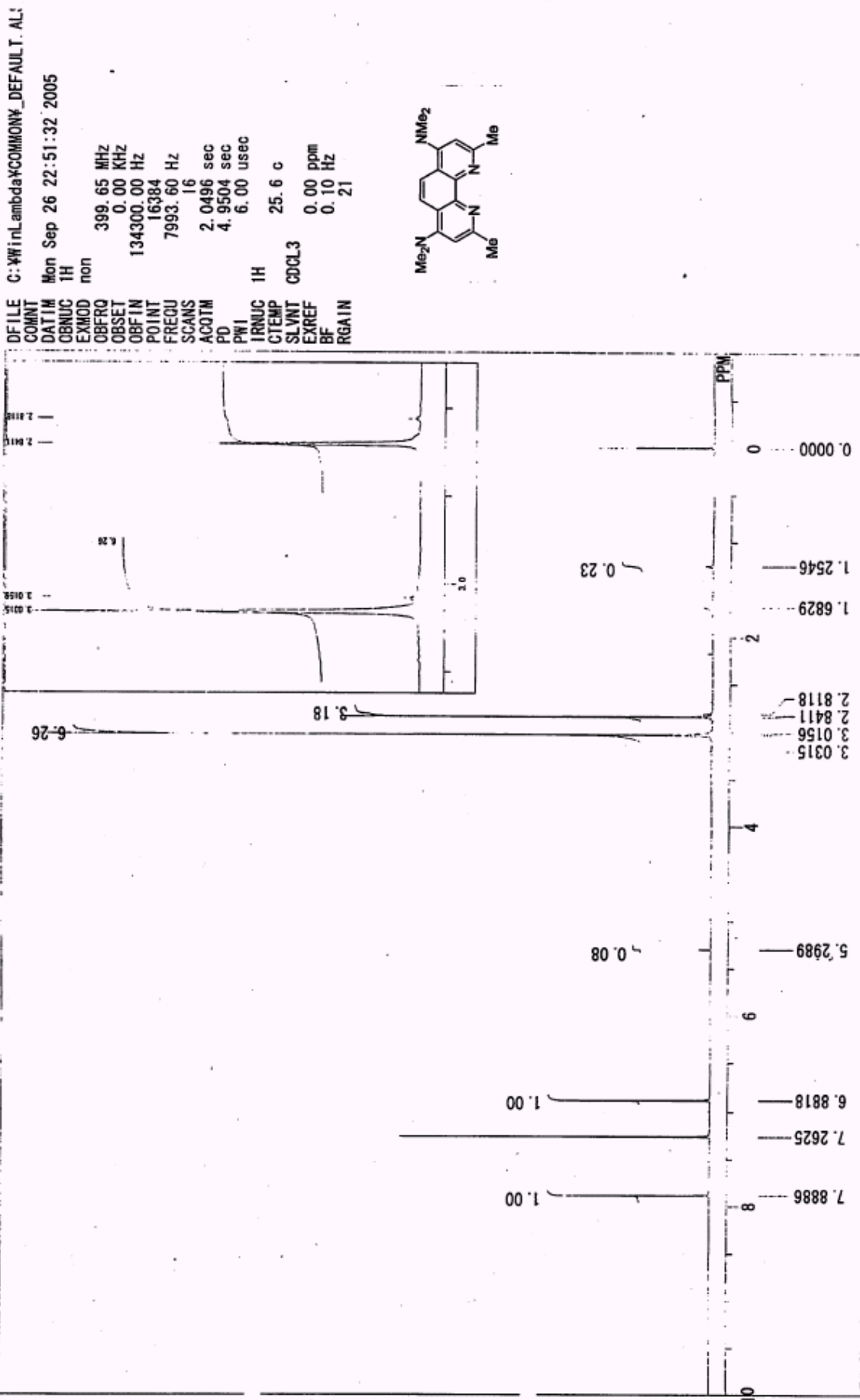


^1H NMR (400 MHz, CDCl_3) δ 2.32 (s, 3 H), 2.35 (s, 3 H), 2.63 (s, 3 H), 7.24 (t, $J = 6.6$ Hz, 1 H), 7.38 (dd, $J = 2.0$ and 7.8 Hz, 1 H), 7.42 (d, $J = 1.5$ Hz, 1 H), 7.67 (dt, $J = 2.0$ and 8.3 Hz, 2 H), 8.02 (dt, $J =$

2.0 and 8.8 Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ 19.48, 19.90, 26.61, 124.59, 126.92, 128.43, 128.84, 130.22, 135.50, 136.89, 137.16, 137.38, 145.84, 197.74; HRMS (EI) calcd for $\text{C}_{16}\text{H}_{16}\text{O}$ 224.1201, found 224.1199.

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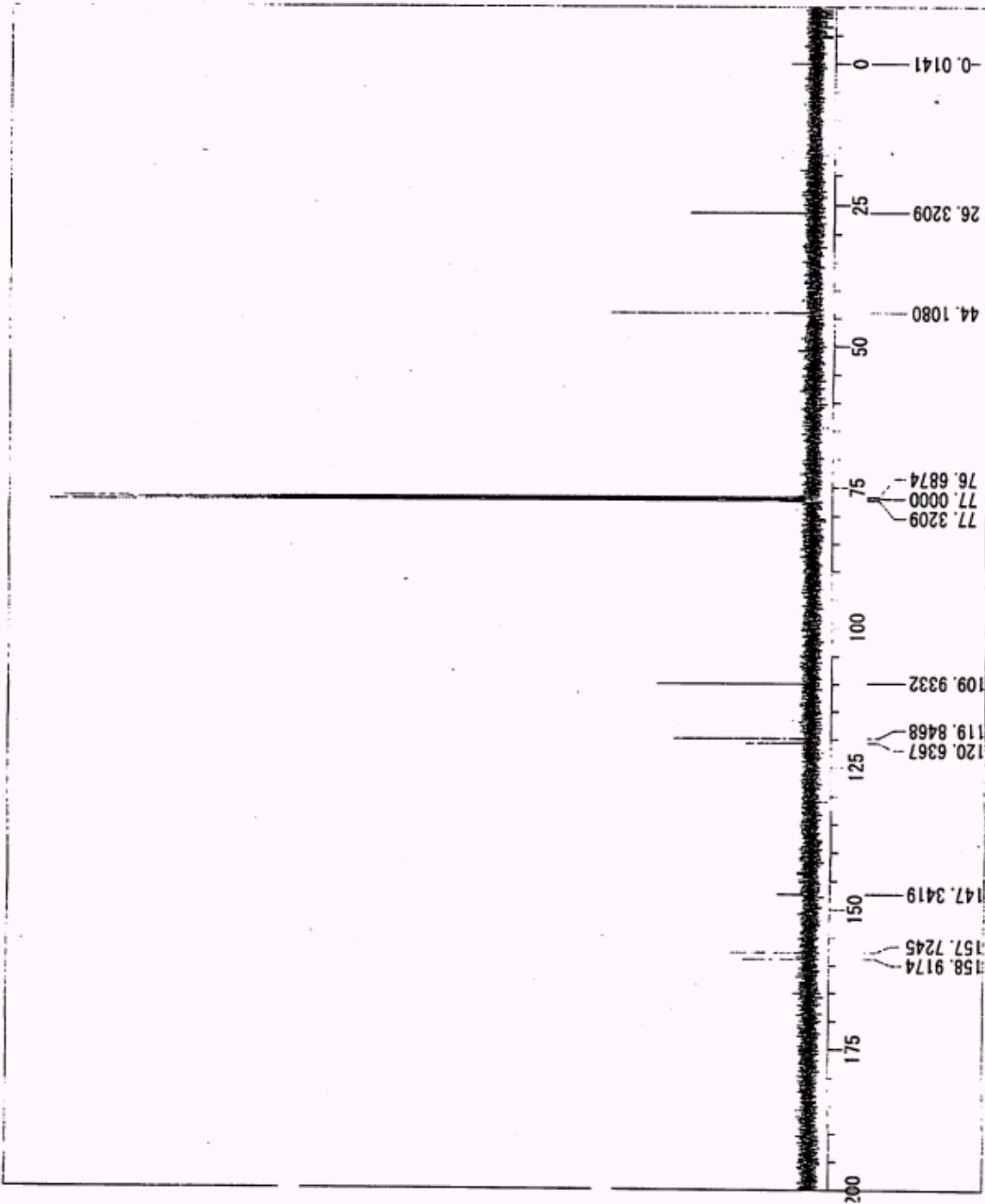
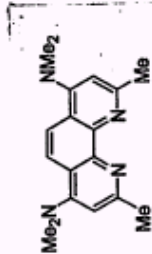
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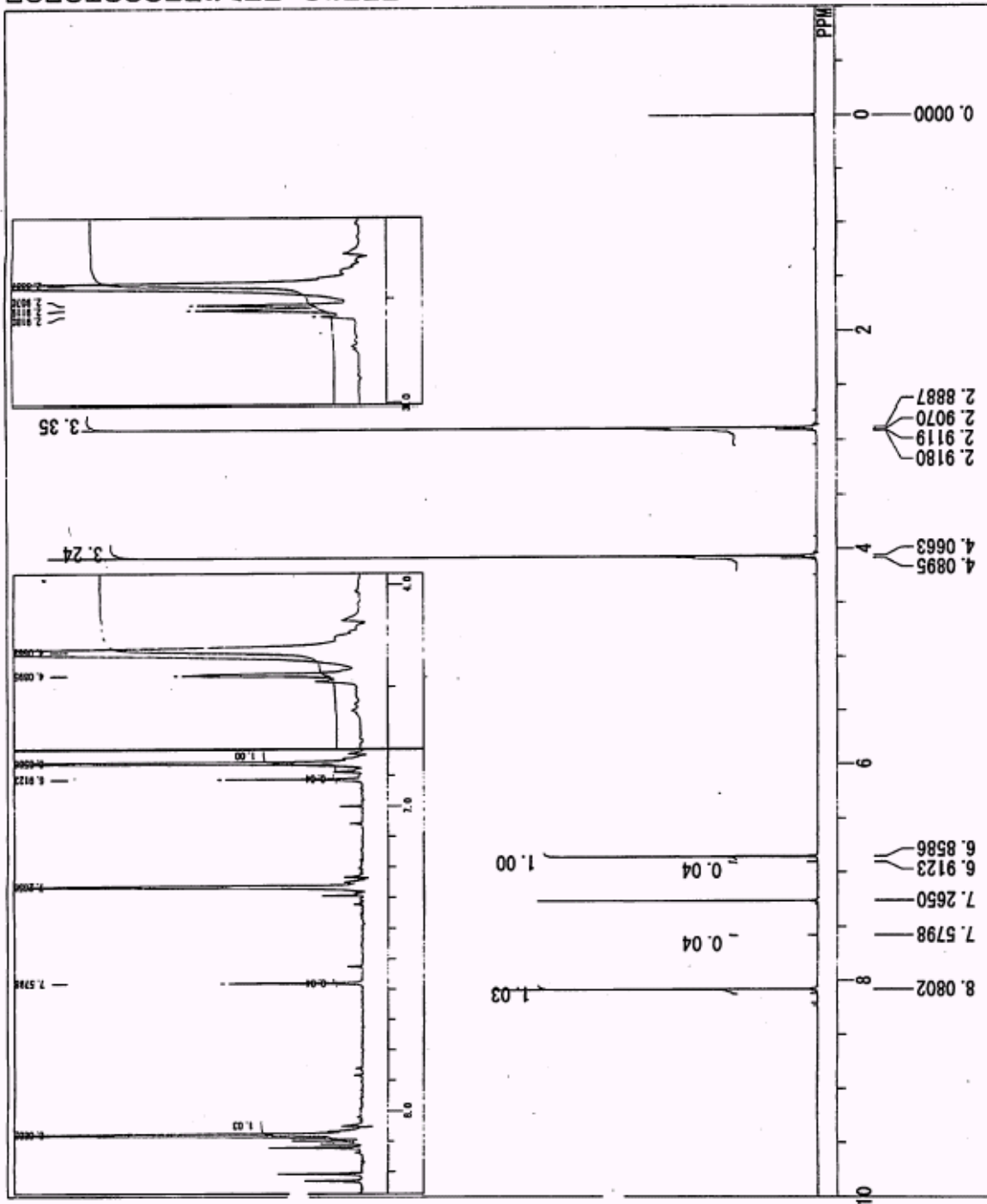
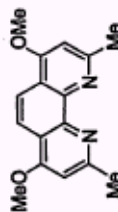
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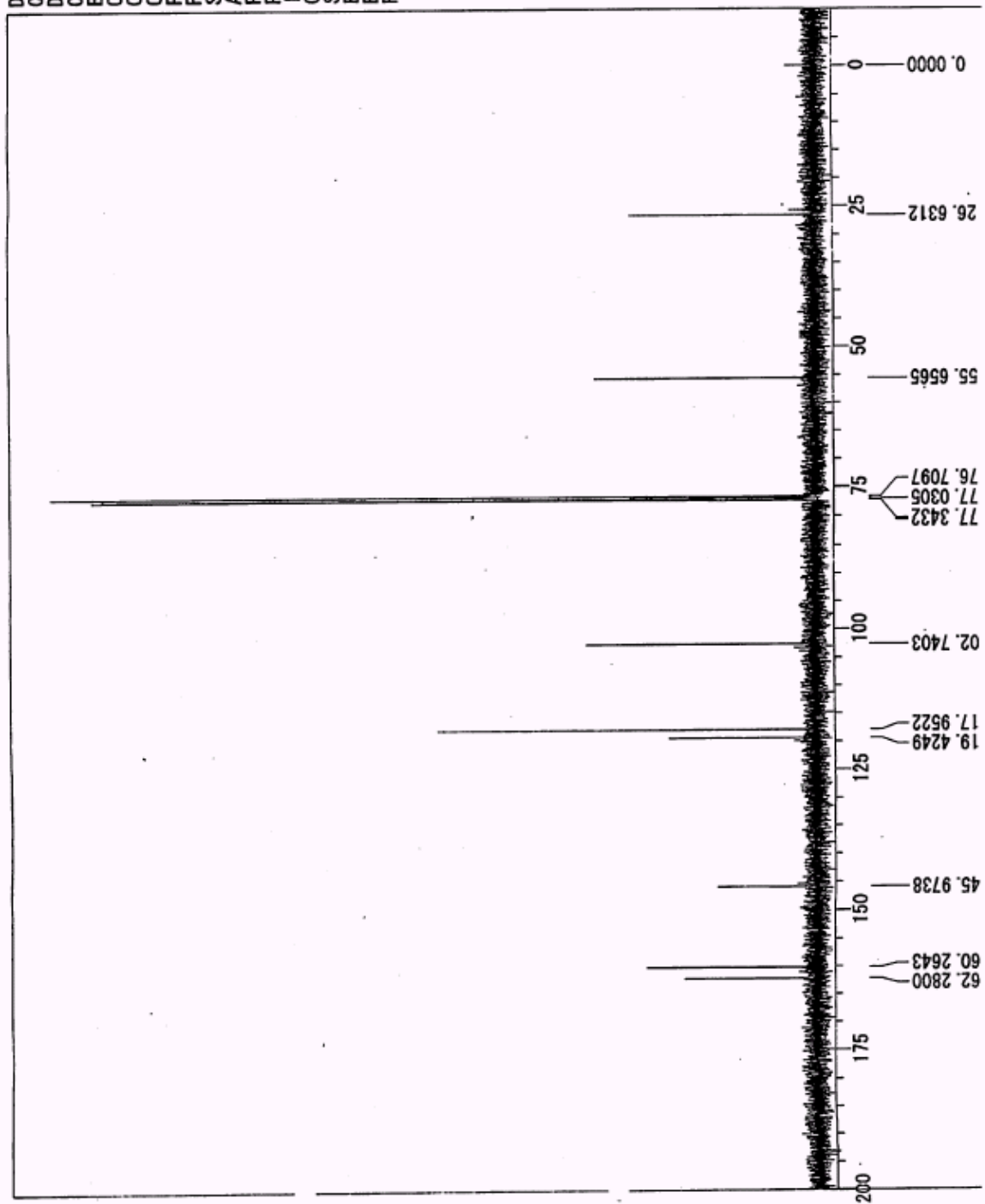
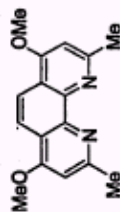


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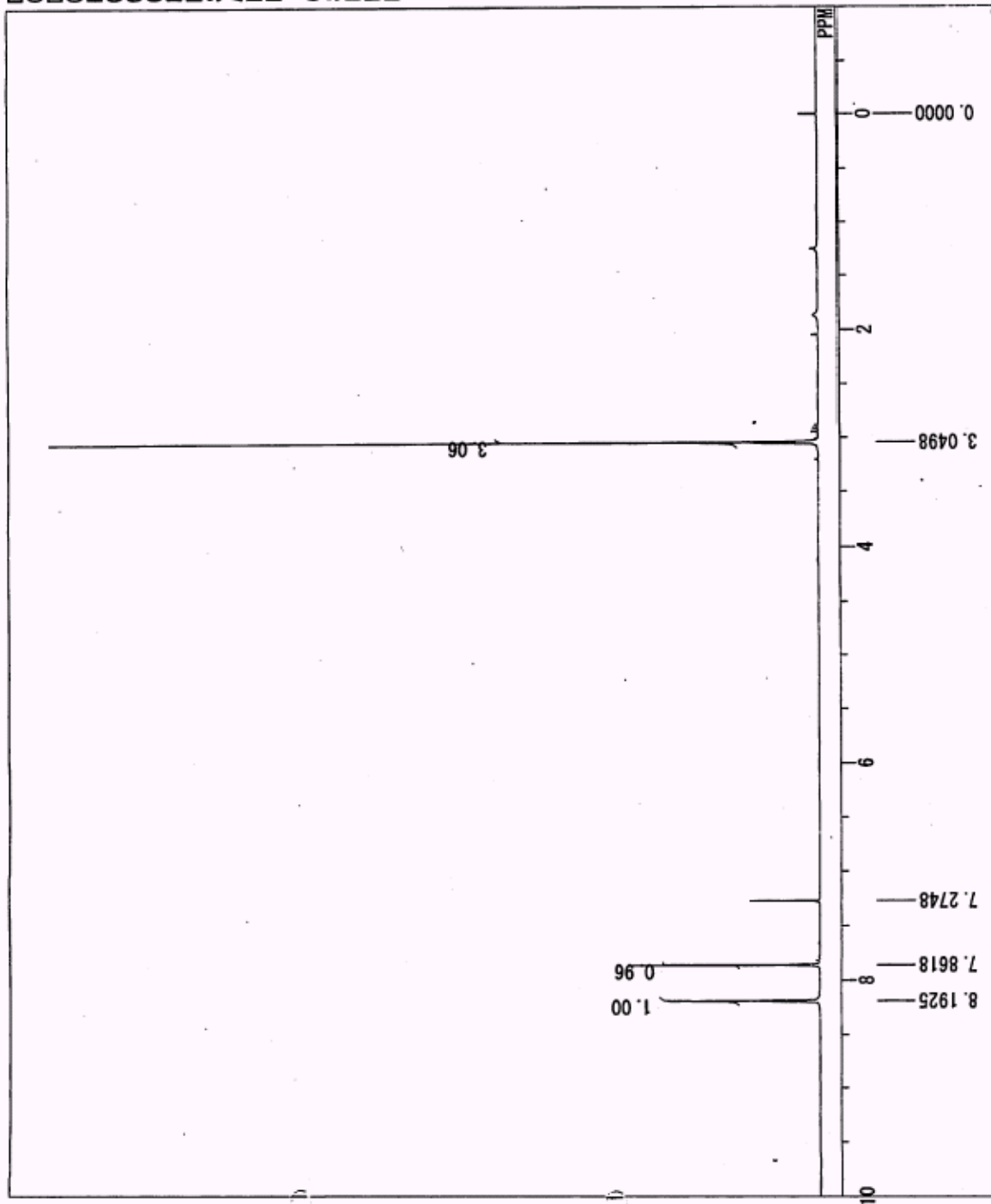
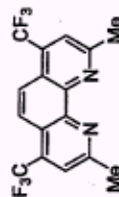


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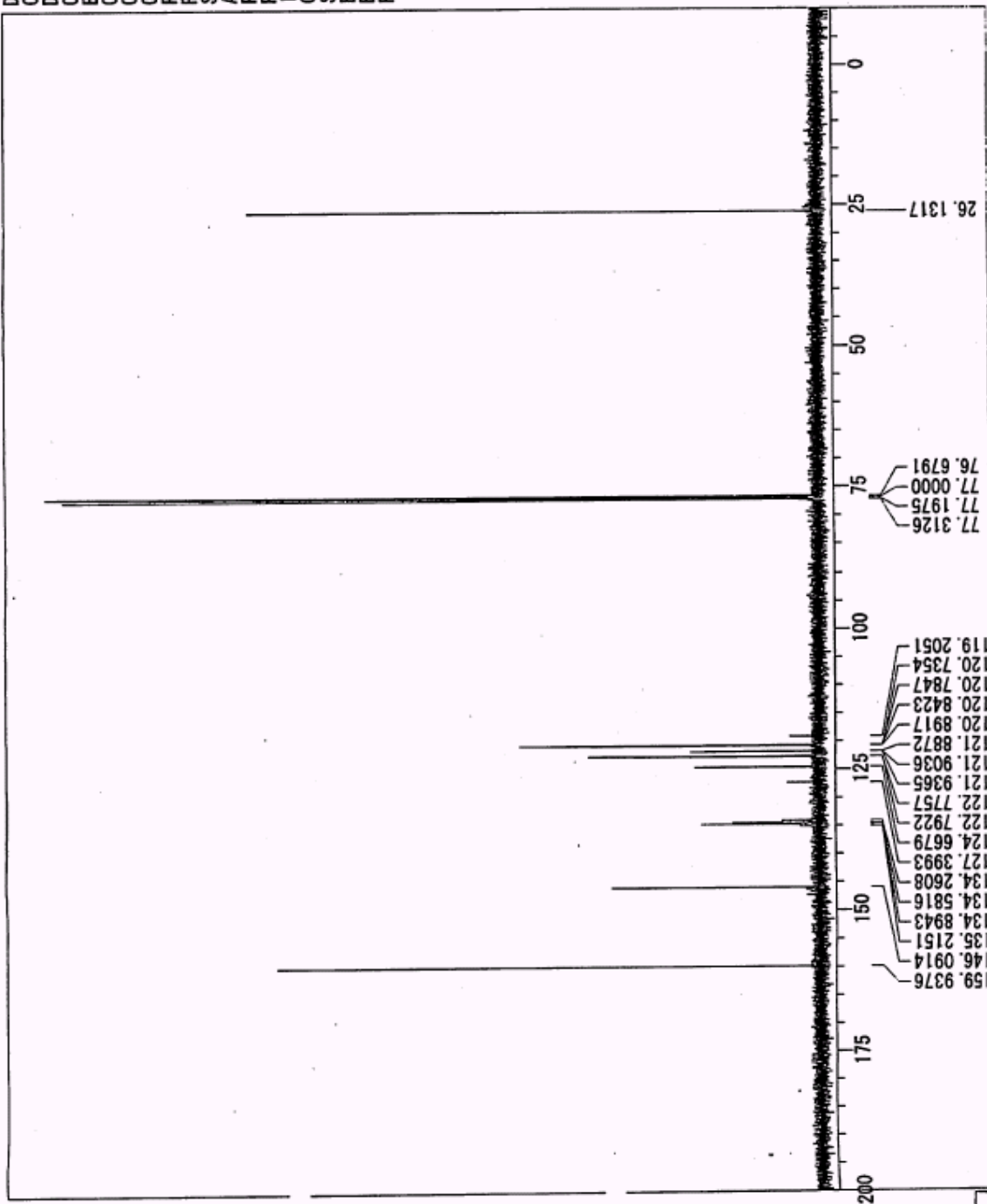
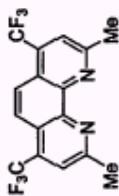
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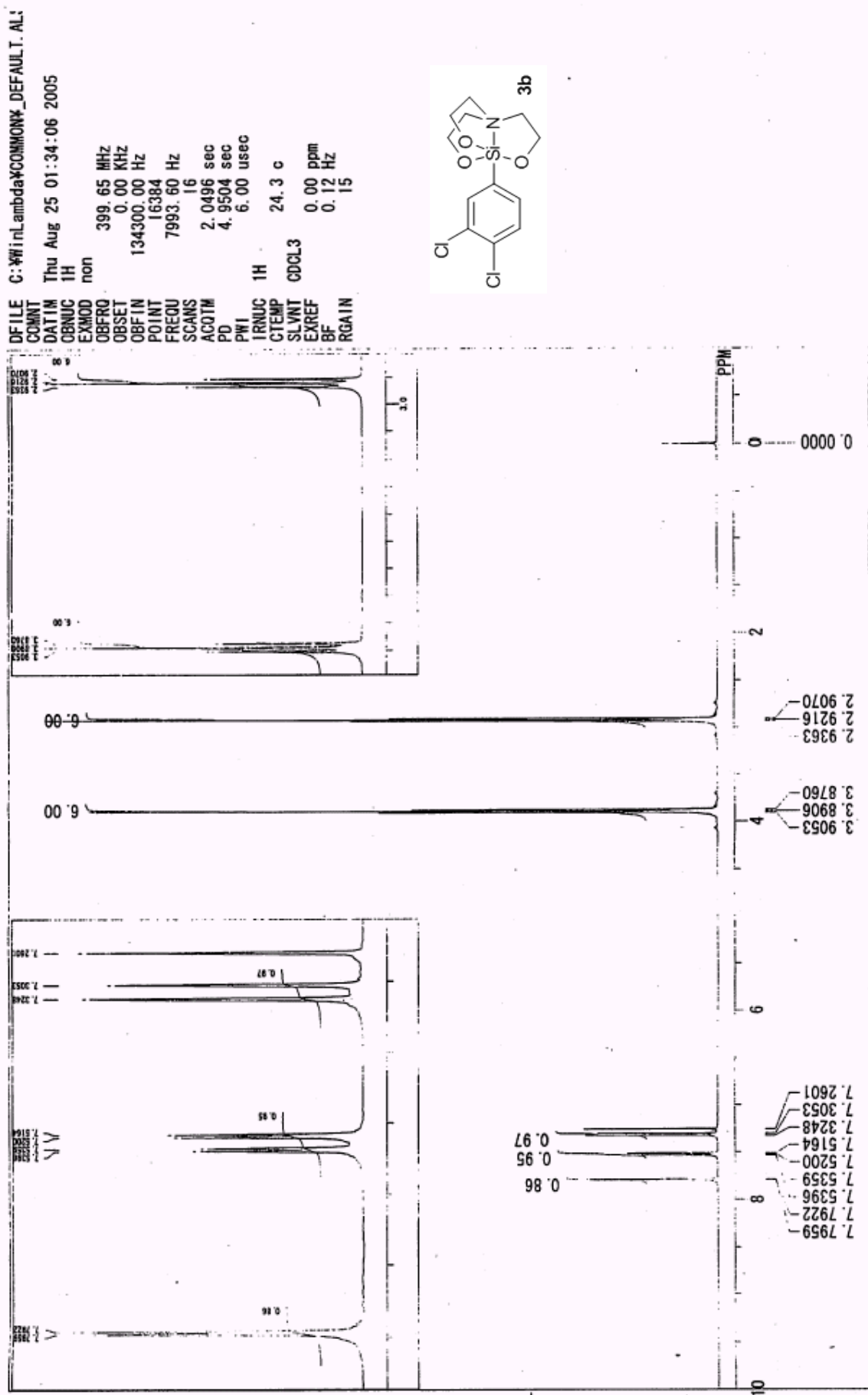


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0.12 Hz
30

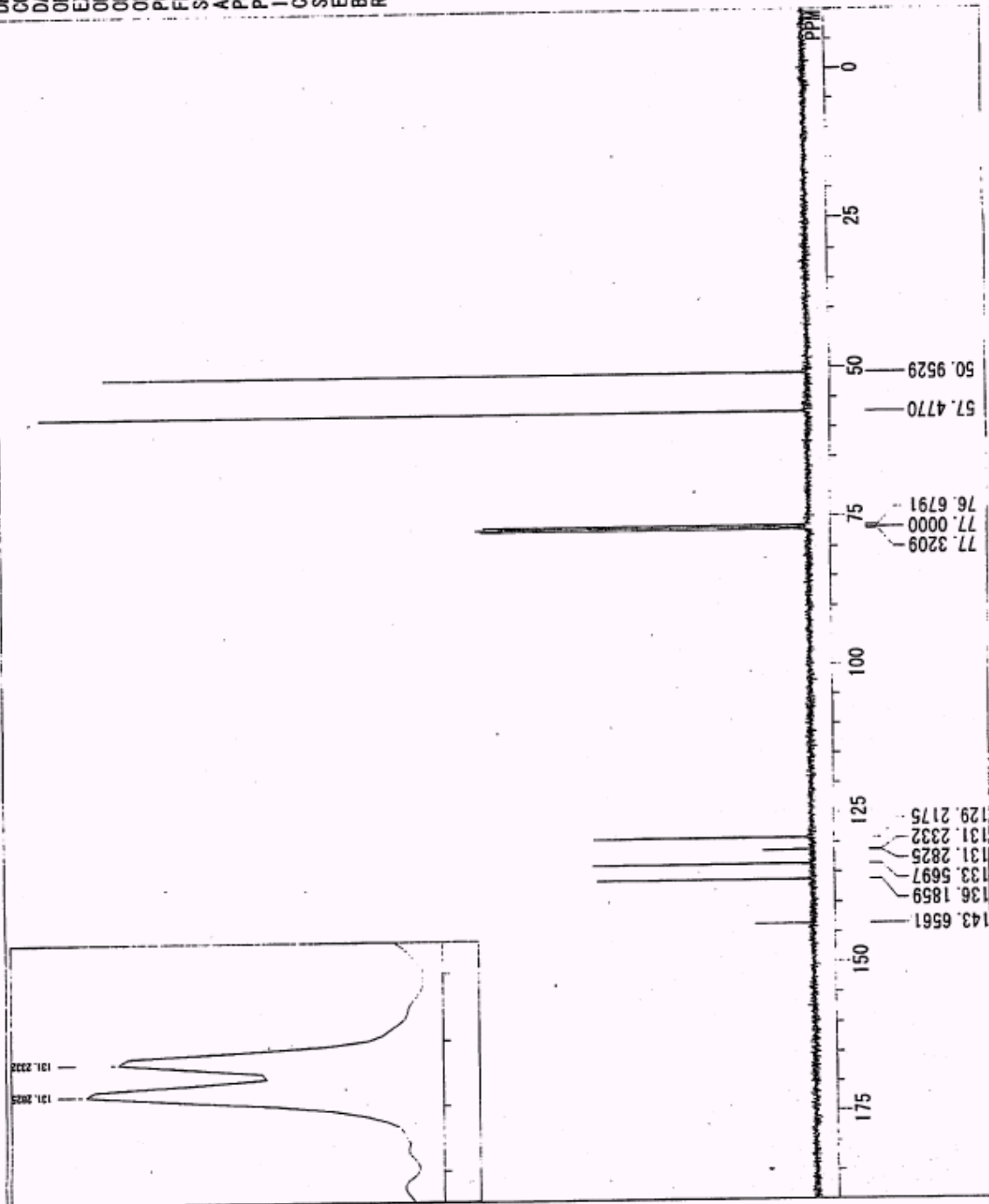
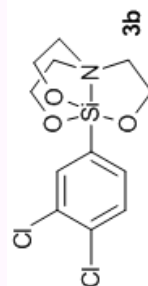


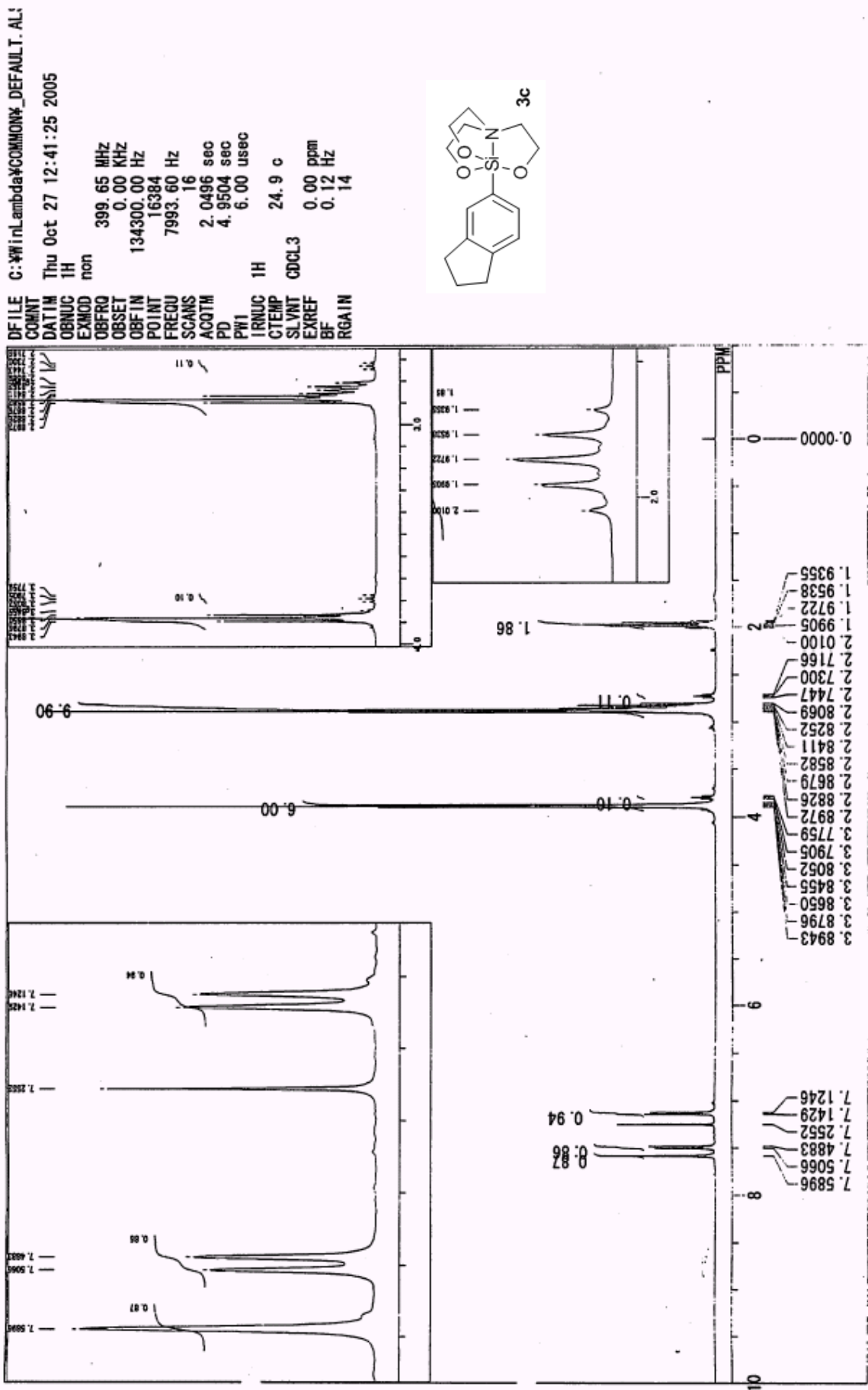


C:\Documents and Settings\lambdi

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Thu Aug 25 00:41:53 2005
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
512
1.2091 sec
1.7909 sec
4.50 usec
1H
24.6 c
CDCL3
77.00 ppm
0.12 Hz
29





C:\WIN\lambda\COMMON\DEFAULT.AL:

Thu Oct 27 13:08:40 2005

13C

bcm

100.40 MHz

0.00 KHz

135500.00 Hz

32768

27100.27 Hz

350

1.2091 sec

1.7909 sec

4.50 usec

1H

25.9 c

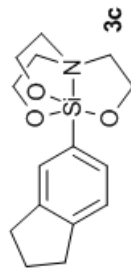
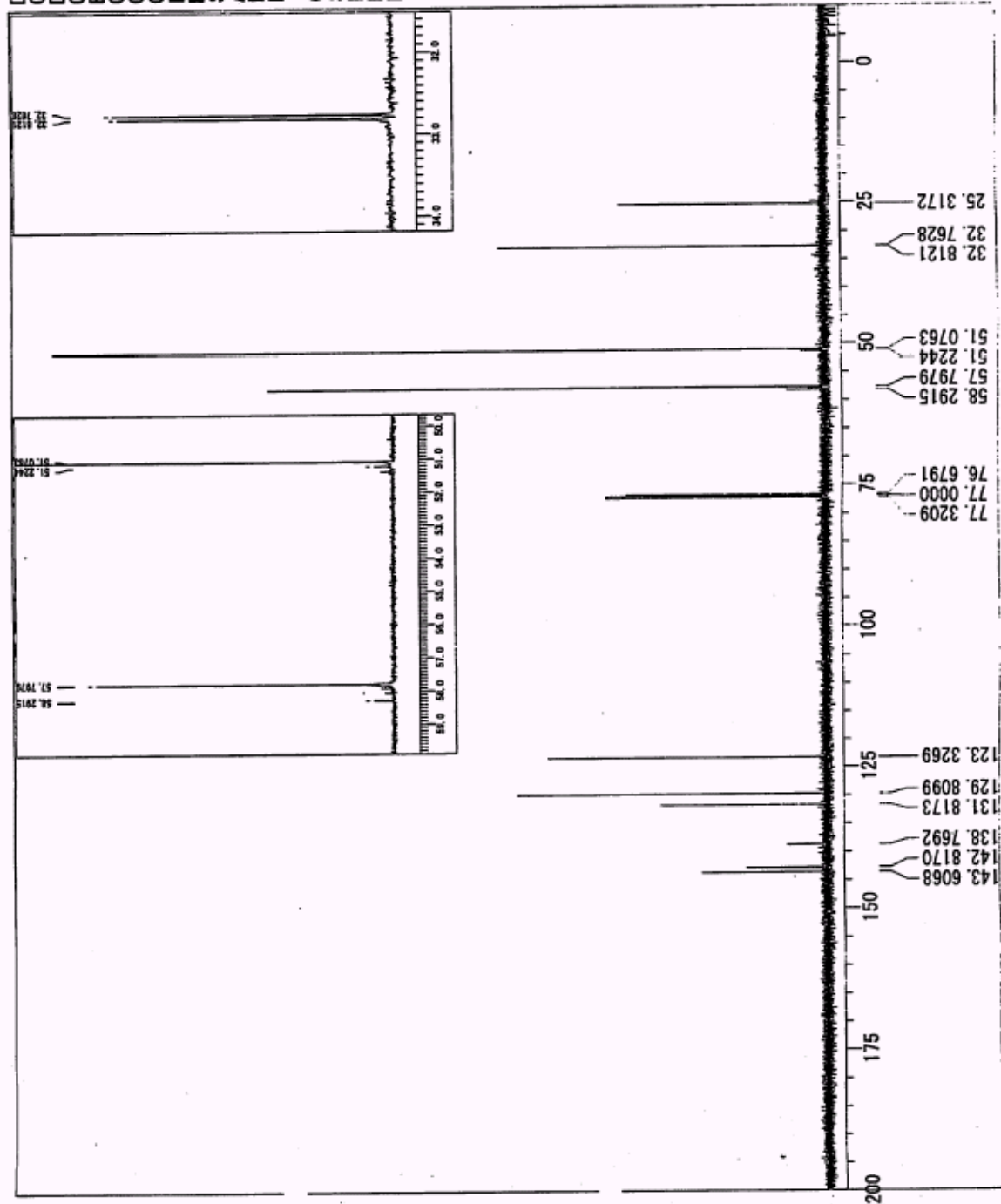
CDCL3

77.00 ppm

0.12 Hz

29

DFILE
COMNT
DATIN
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



C:\W1\inLambda\COMMON\DEFAULT.AL:

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRO
OBSST
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Thu Oct 13 19:33:15 2005

1H

non

399.65 MHz

0.00 KHz

134300.00 Hz

16384

7993.60 Hz

16

2.0496 sec

4.9504 sec

6.00 usec

1H

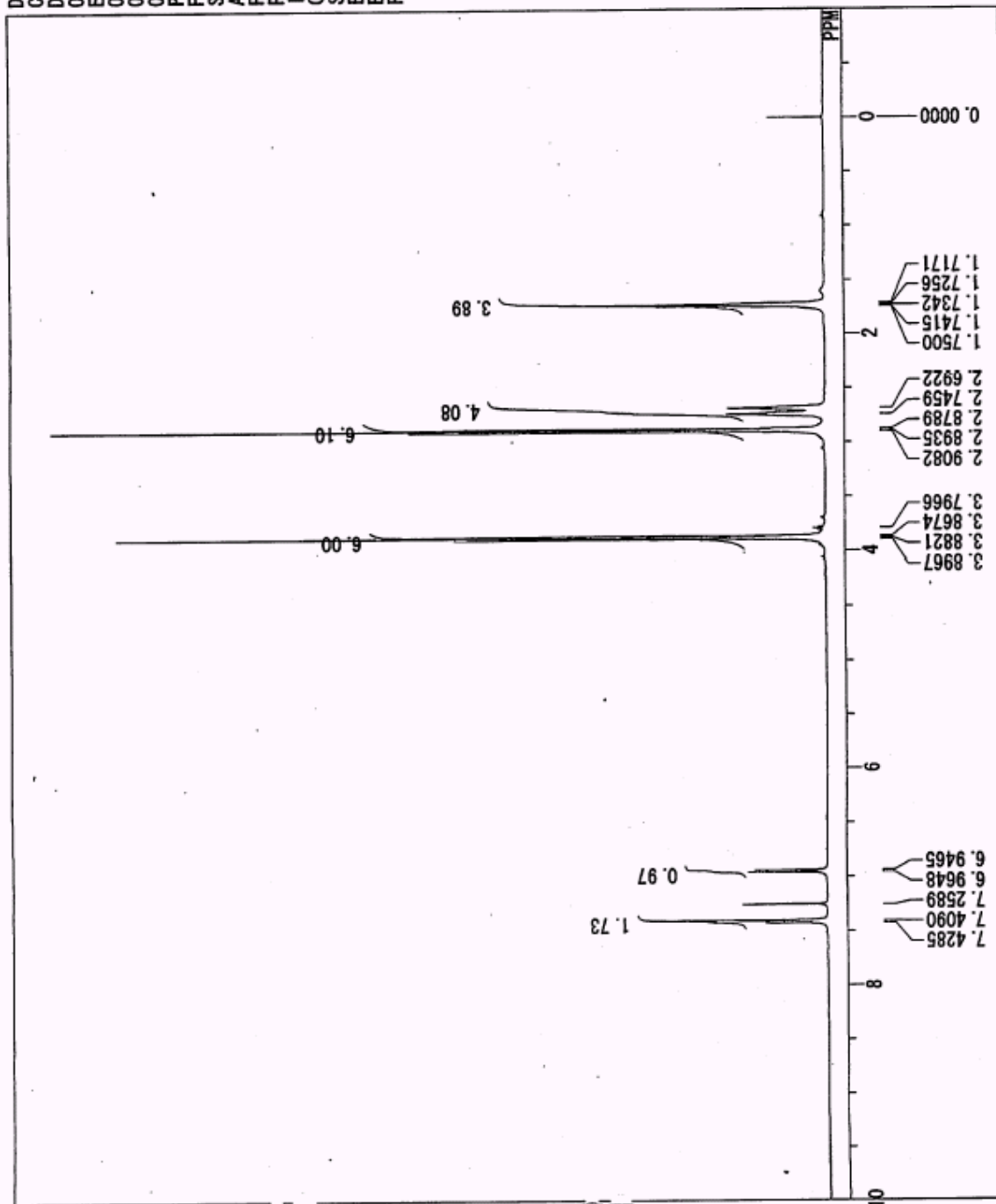
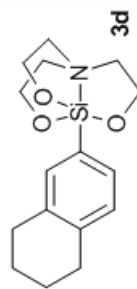
CDCL3

24.8 c

0.00 ppm

0.00 Hz

18

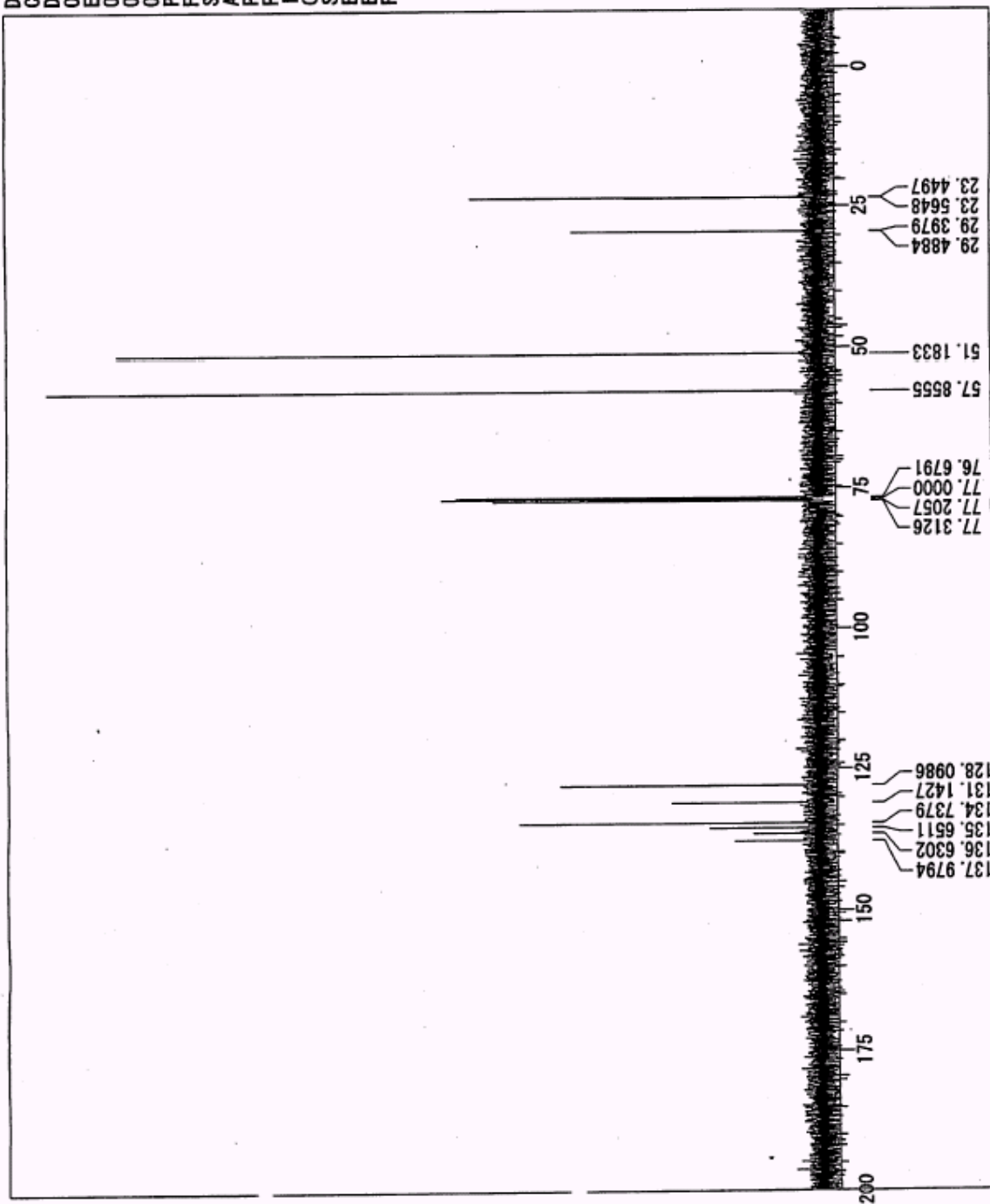
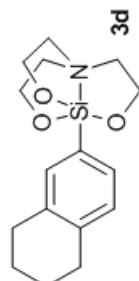


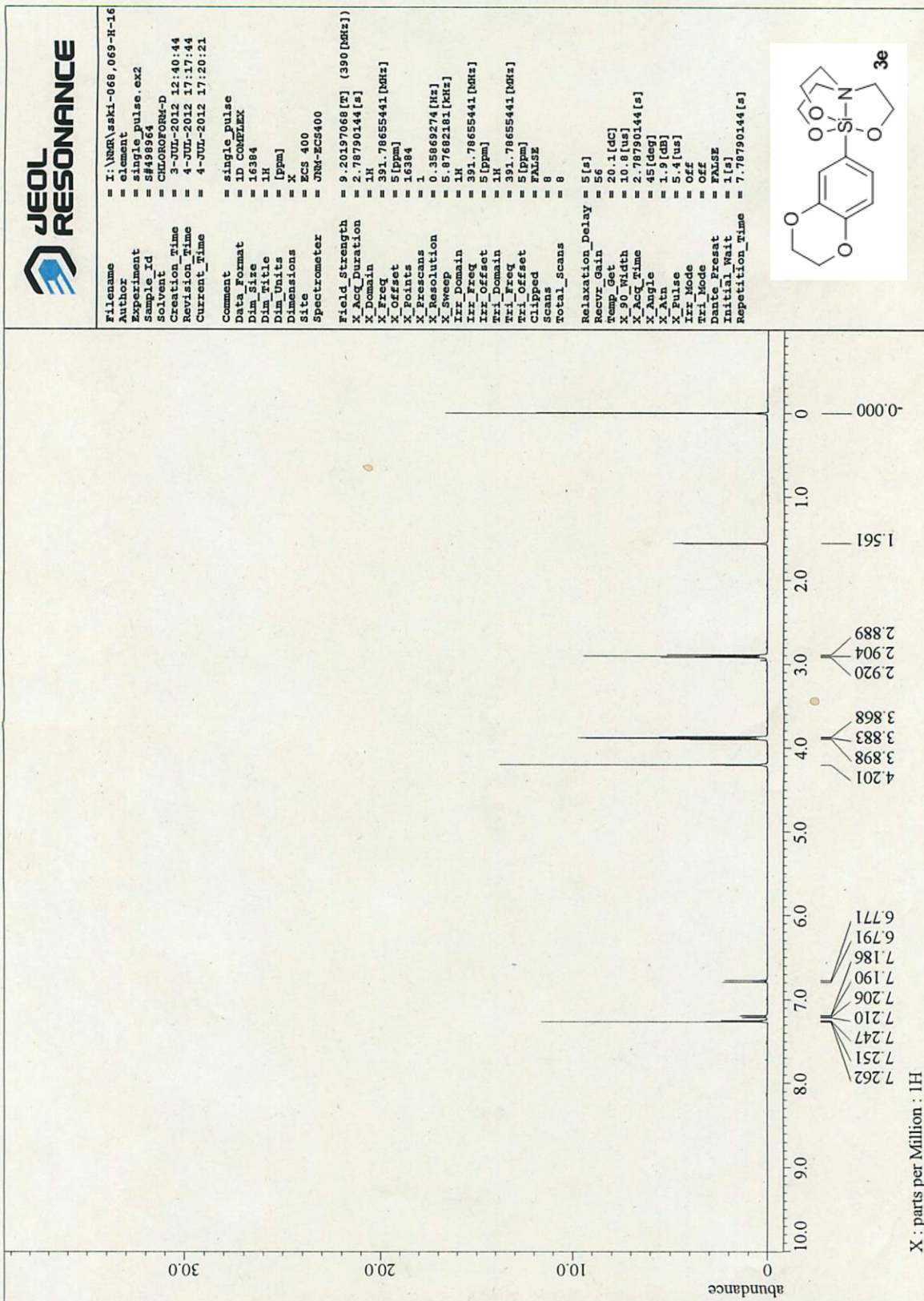
DFILE C:\Documents and Settings\lambdi

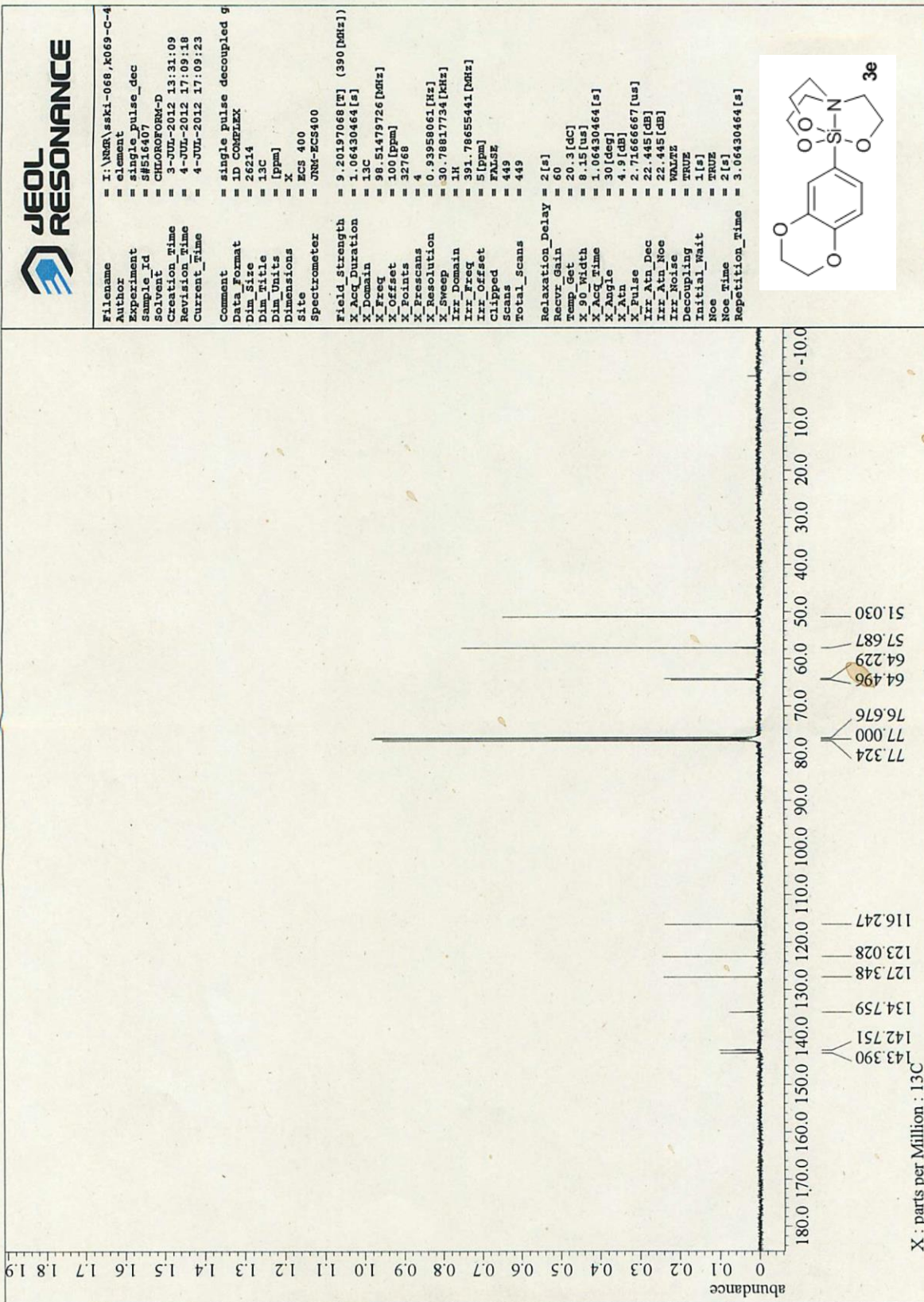
CCMINT
DATIN
OBNUC 13C
EXMDD
OBFREQ
OBFSET
OBFIN
POINT
FREQ
SCANS
ACQTH
PD
PWI
IRNUC 1H
CTEMP
SLVNT
EXREF
BF
RGAIN

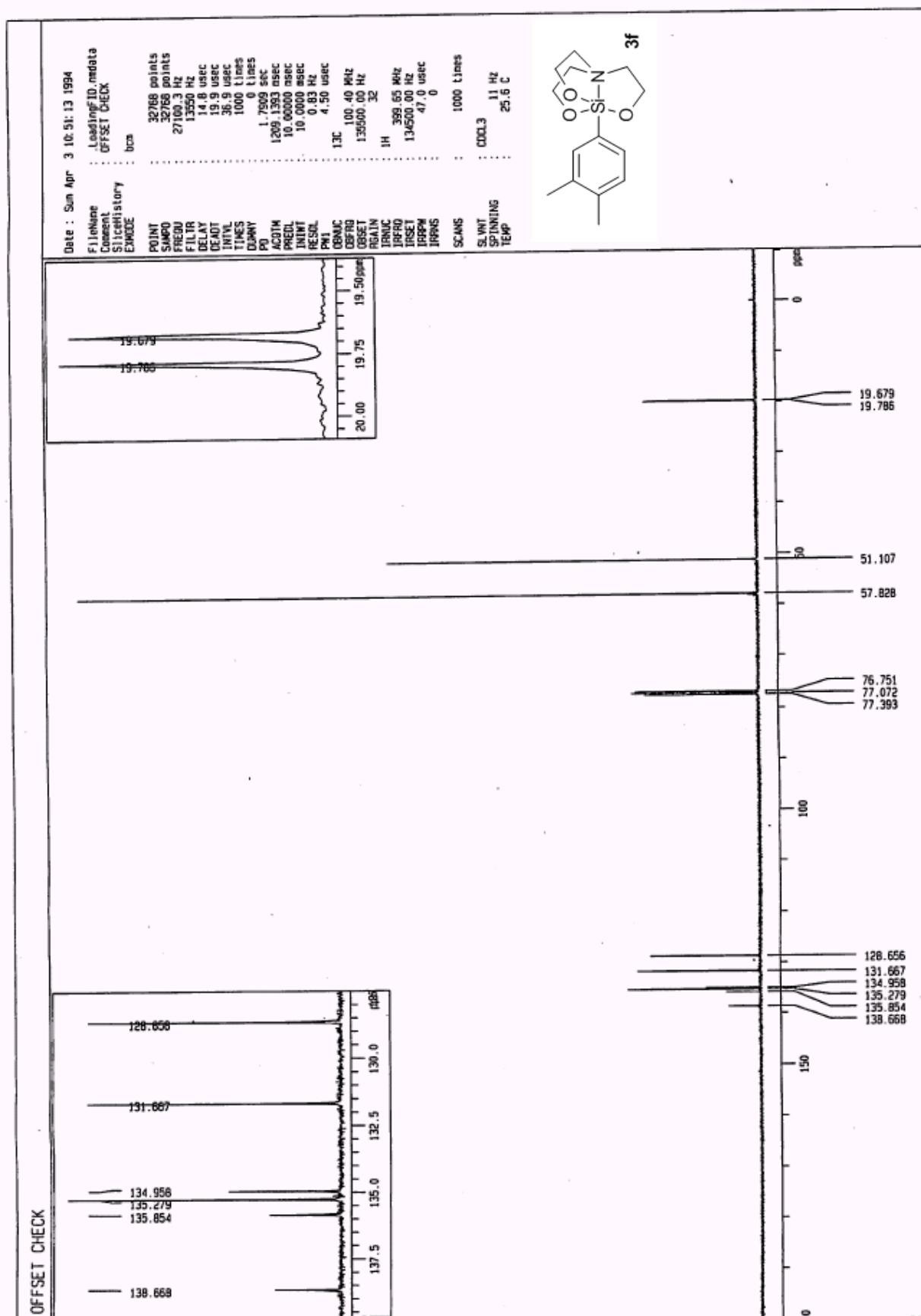
Thu Oct 13 20:05:49 2005

13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
512
1.2091 sec
1.7909 sec
4.50 usec
26.2 c
CDCL3
77.00 ppm
0.00 Hz
29



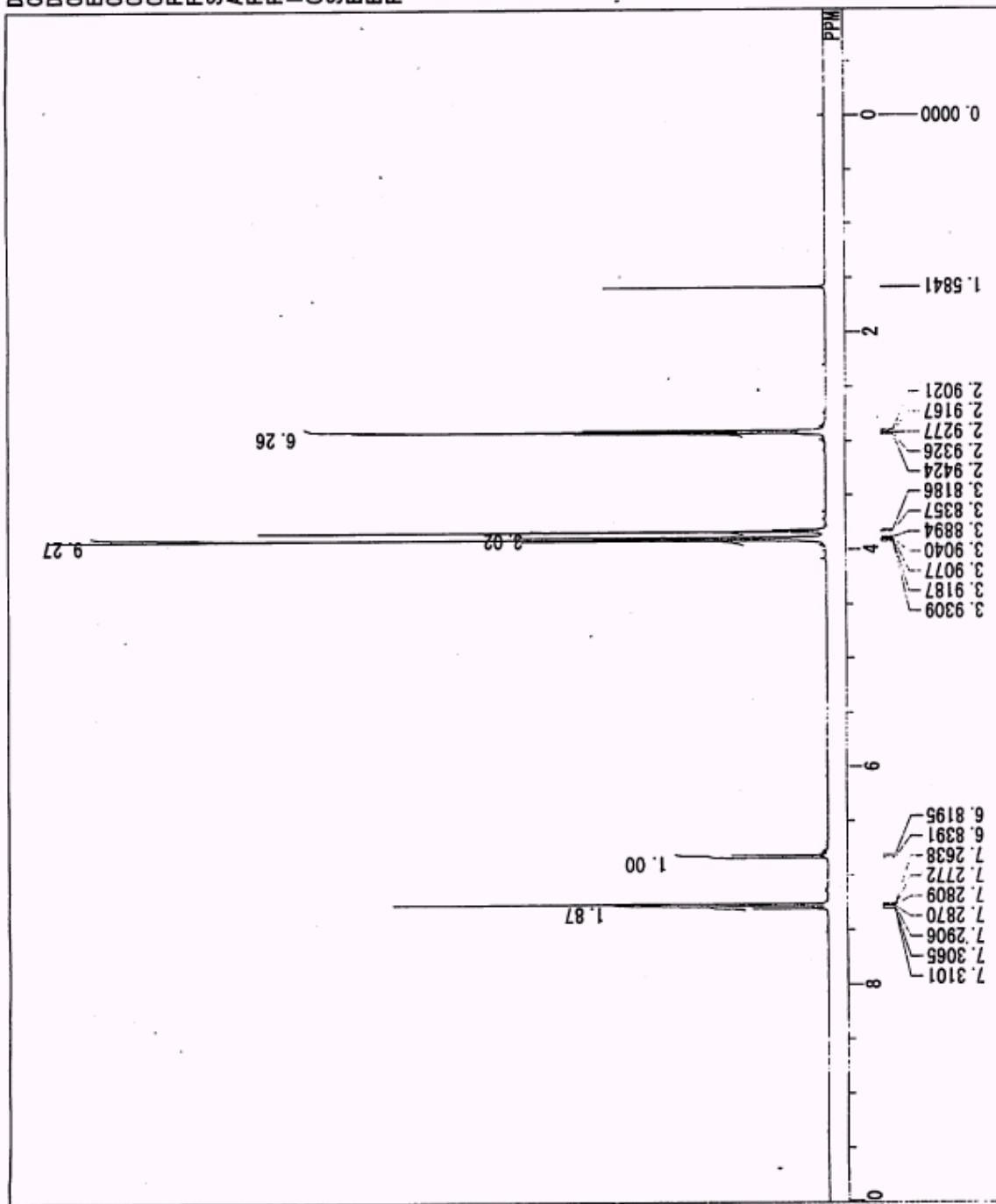
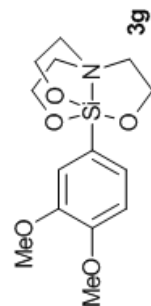






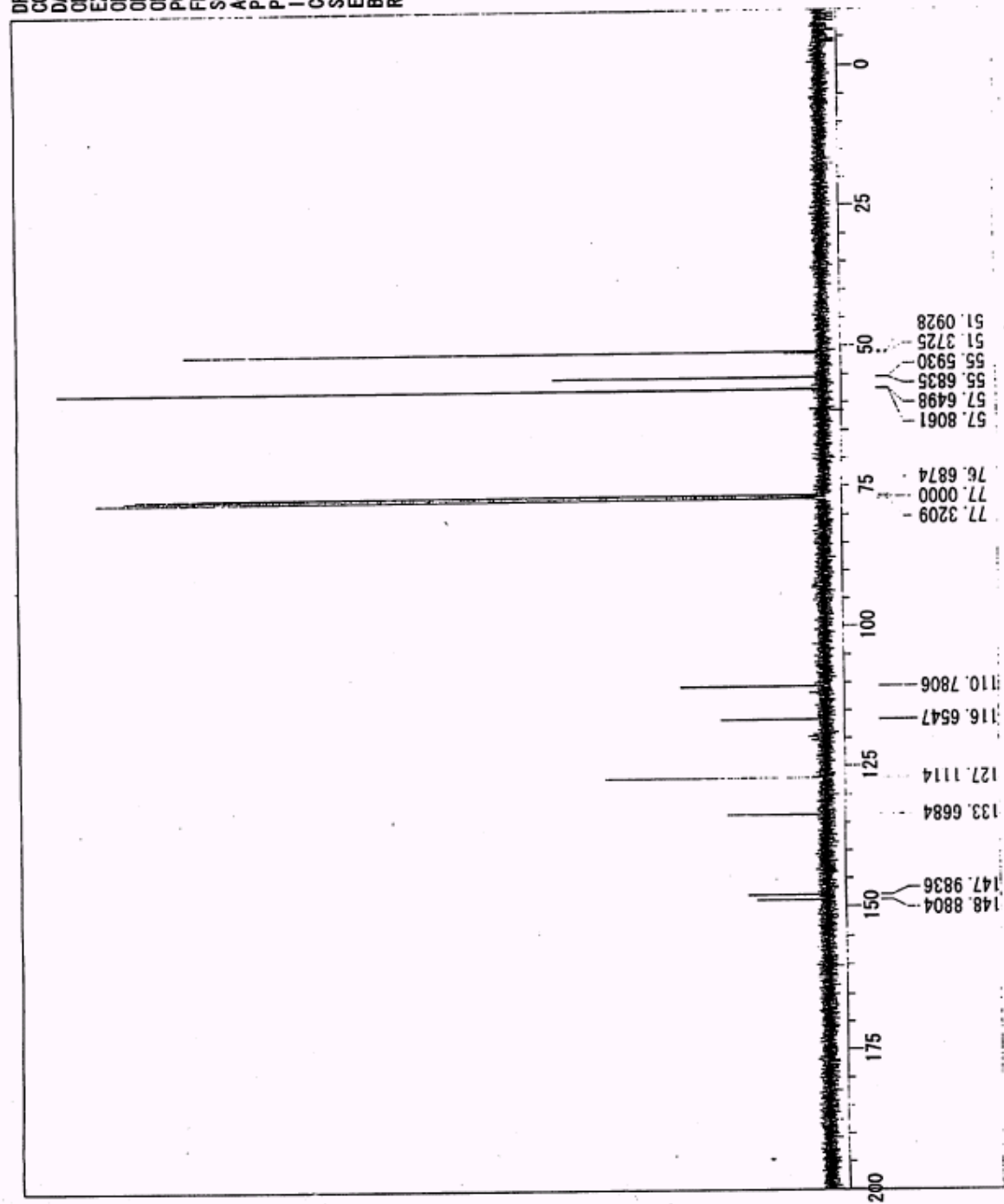
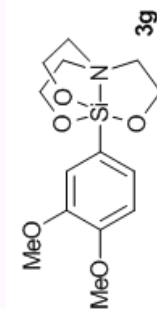
DFILE C:\Documents and Settings\lambdr

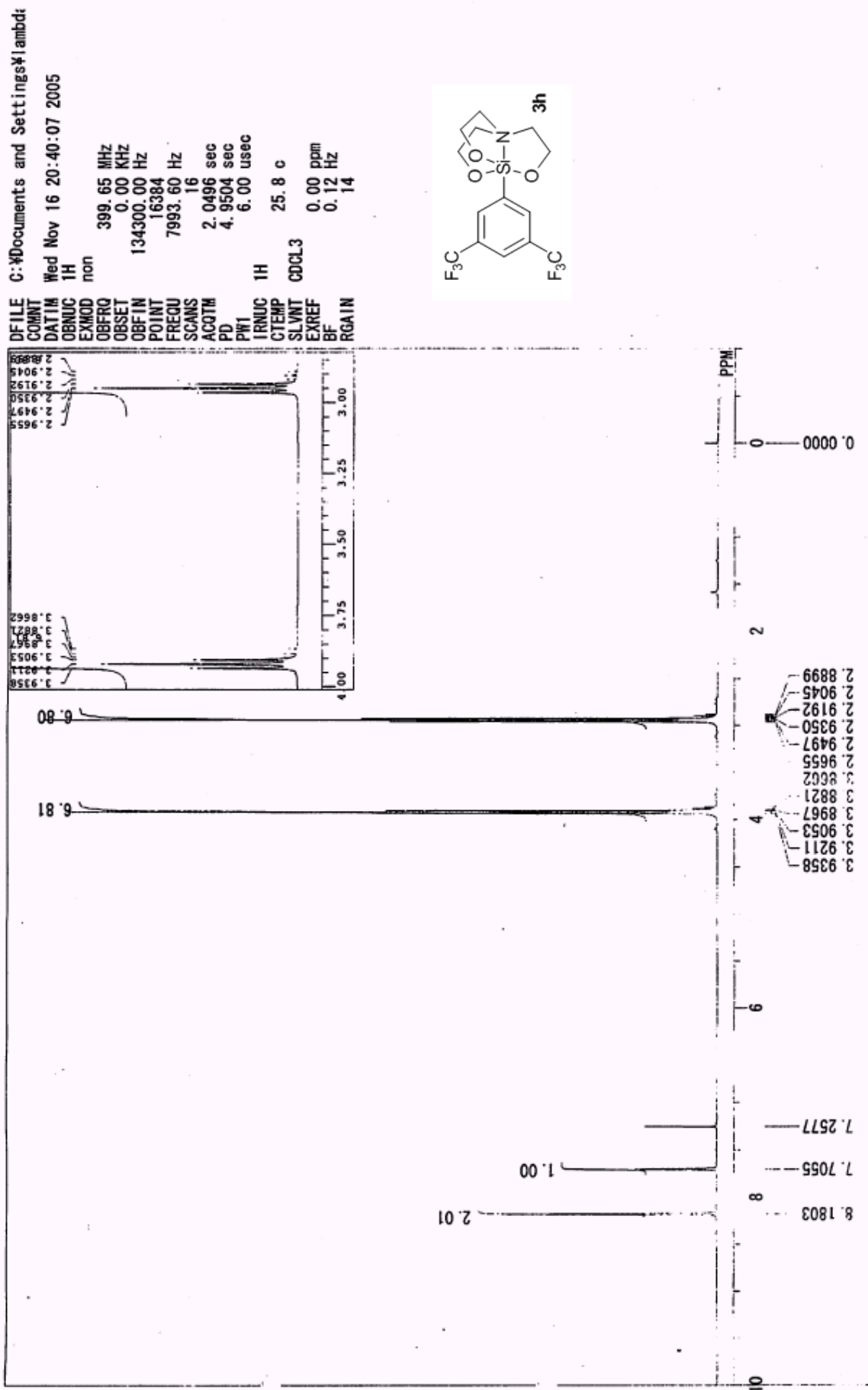
CONVT
DATIM Fri Dec 09 13:15:37 2005
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 16384
FREQU 7993.60 Hz
SCANS 16
ACQTM 2.0496 sec
PD 4.9504 sec
PWI 6.00 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18



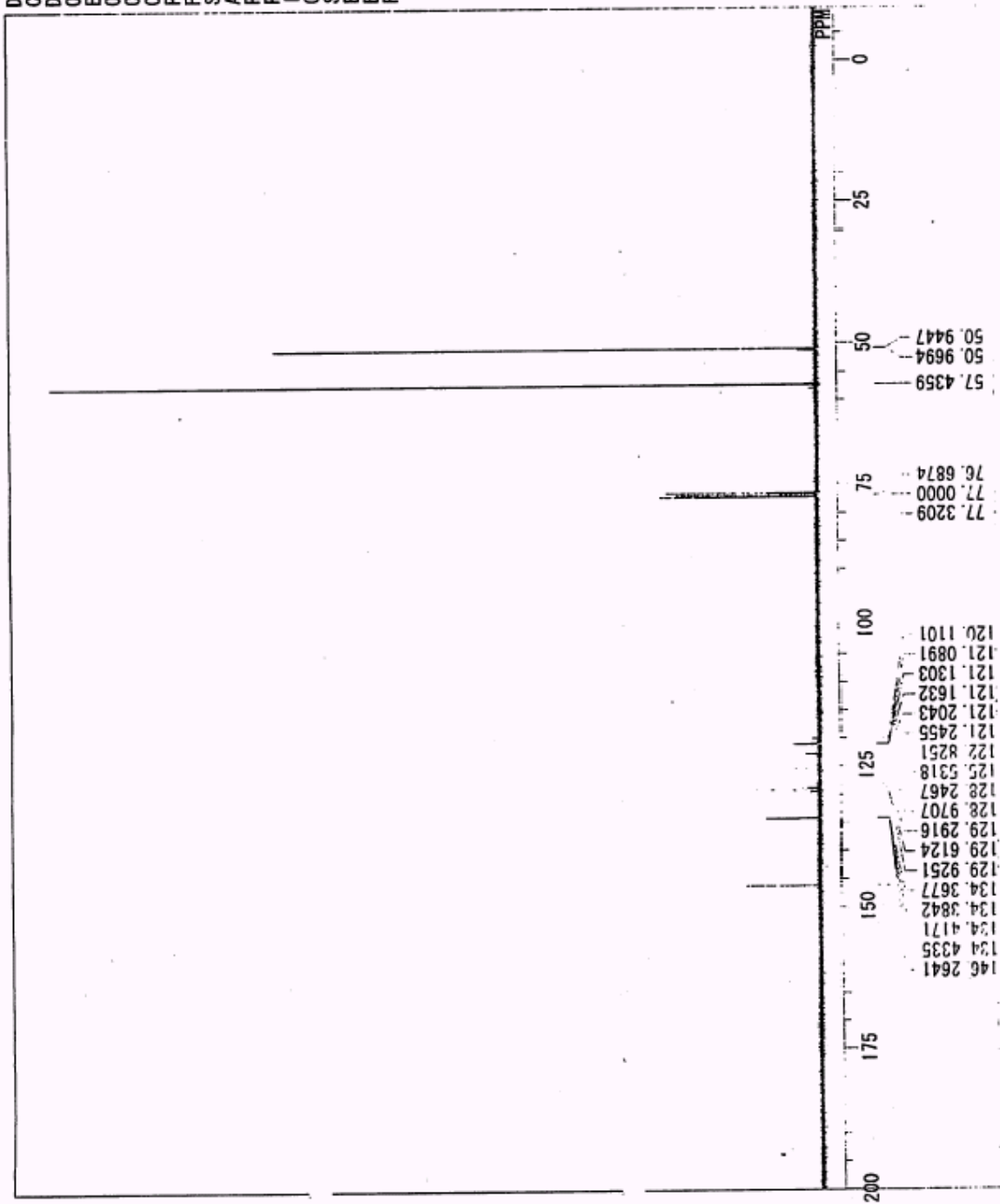
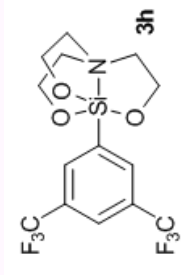
DF1LE C:\P\inl\lambdambda\COMMON\DEFAULT.T.AL

COMNT
DATIM Fri Dec 09 14:08:01 2005
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFTN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1024
ACQTH 1.2091 sec
PD 1.7909 sec
PHI 4.50 usec
IRNUC 1H
GTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30

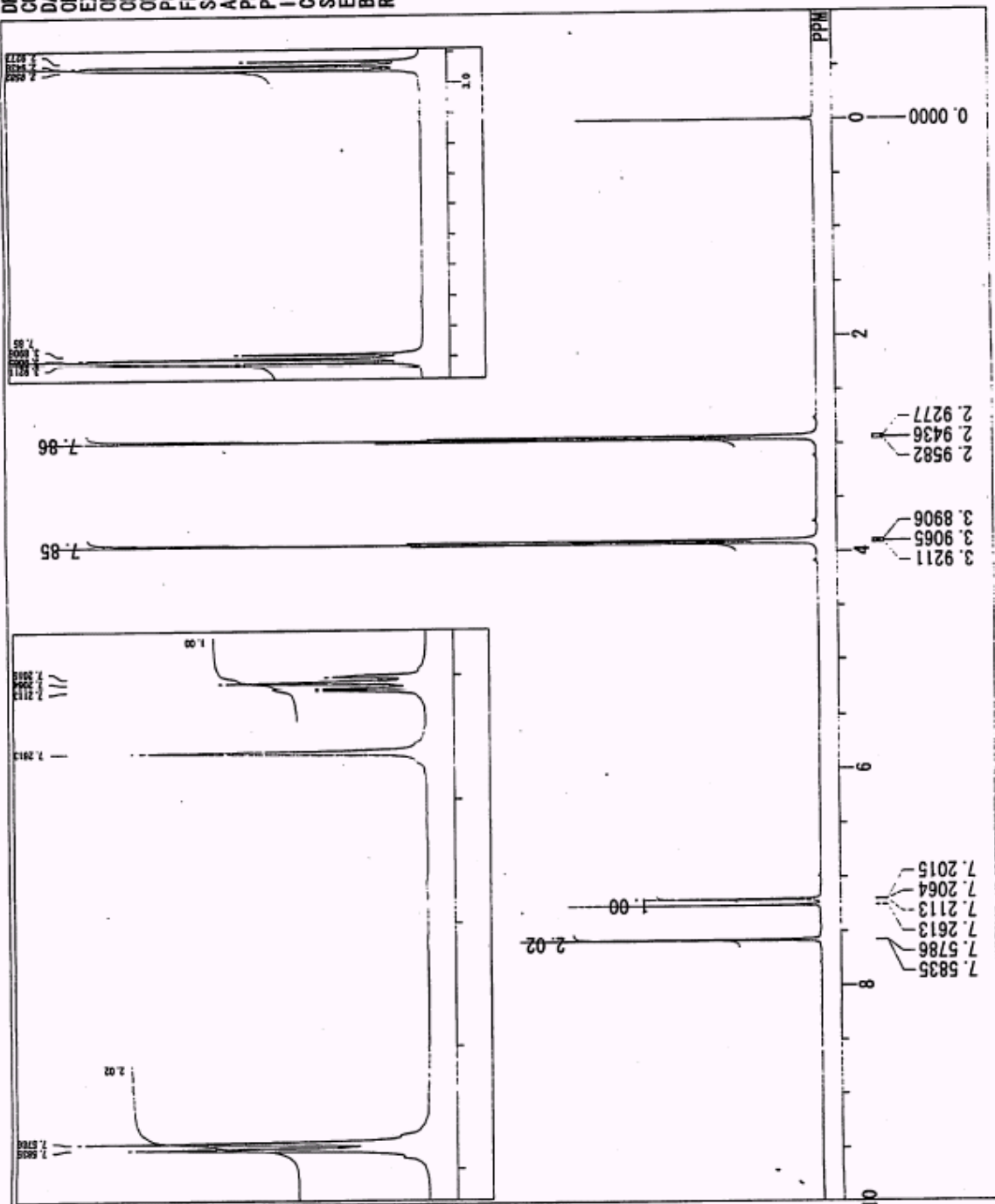
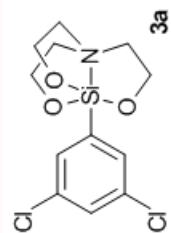




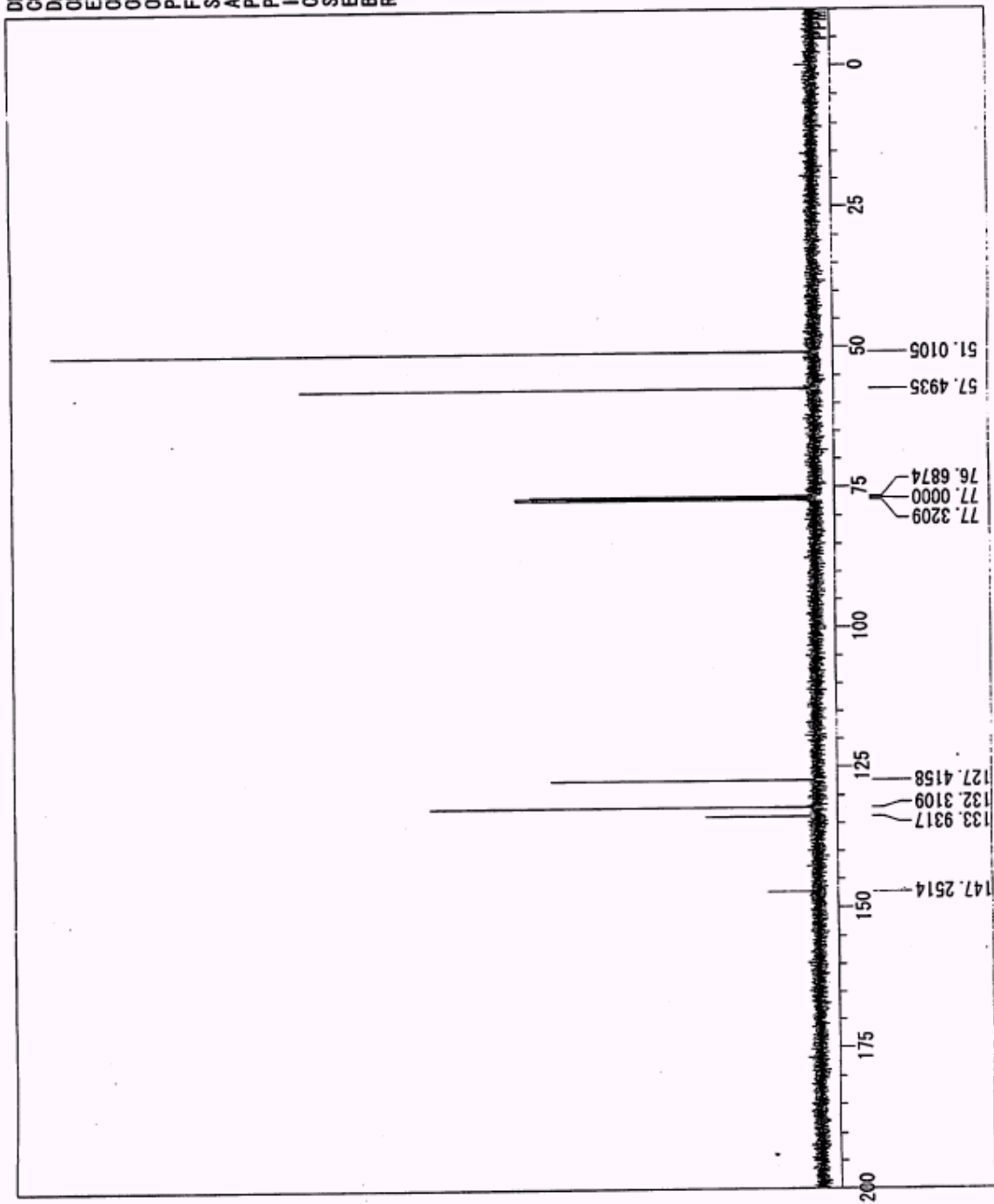
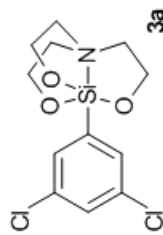
DF ILE E:\biscf3sillatrac.c ALS
COMNT Wed Nov 16 21:06:42 2005
DATIM 13C
OBNUC bom
EXMOD 100.40 MHz
OBFRQ 0.00 KHz
OBSET 135500.00 Hz
OBFIN 32768
POINT 27100.27 Hz
FREQU 512
SCANS 1.2091 sec
ACQTM 1.7909 sec
PD 4.50 usec
PHI 1H
IRNUC 26.6 C
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 0.12 Hz
BF 30
RGAIN



DFILE E:\13diclsilatrane-H. ALS
COMMT
DATIM Fri Aug 19 18:39:19 2005
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 kHz
OBFIN 134300.00 Hz
POINT 16384
FREQU 7993.60 Hz
SCANS 16
ACQTM 2.0496 sec
PD 4.9504 sec
PWT 6.00 usec
IRNUC 1H
CTEMP 23.8 C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18

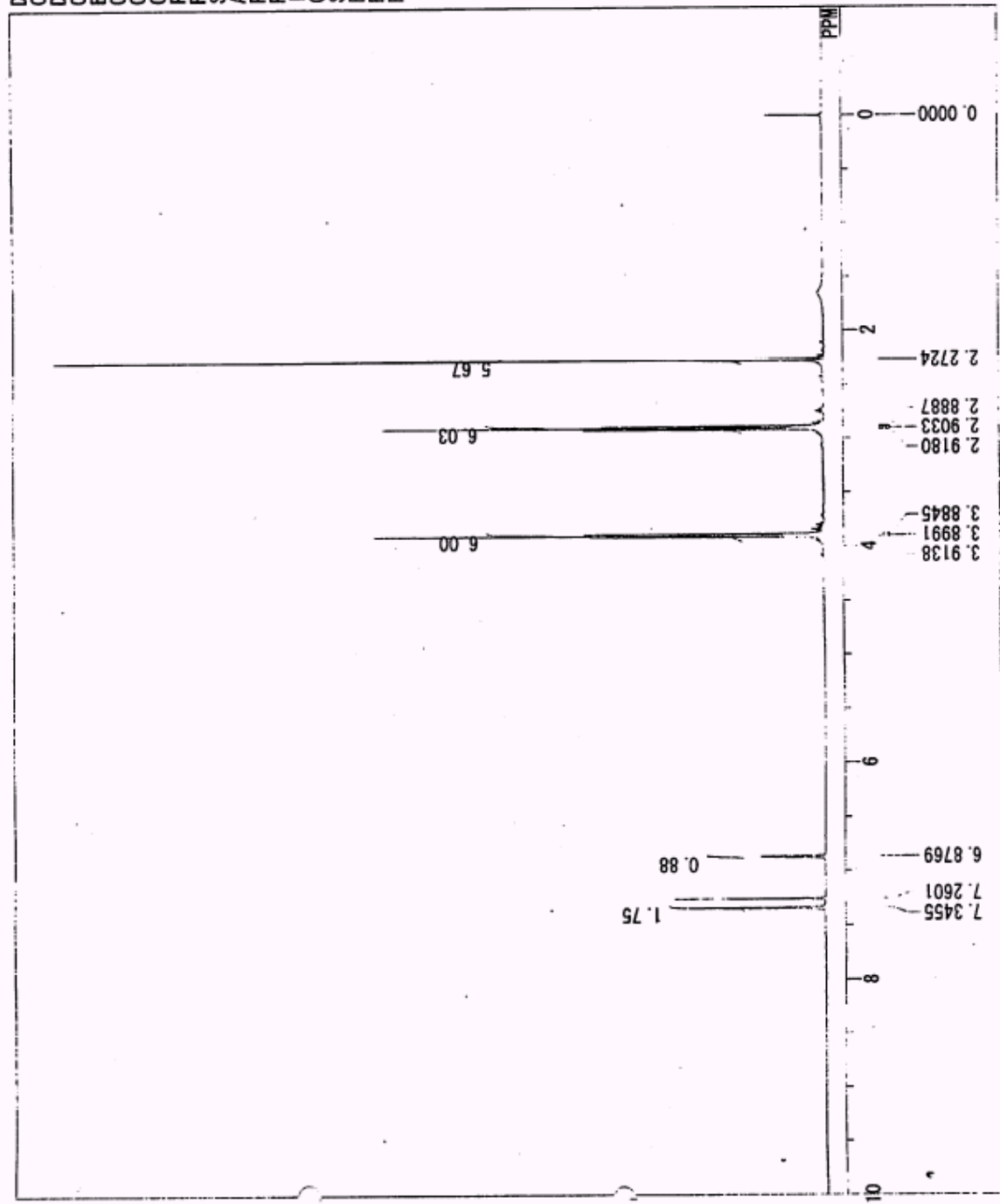
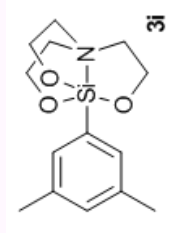


DFILE E:\13diclsilatrane-C. ALS
COMINT
DATIM Fri Aug 19 19:06:31 2005
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 512
ACQTM 1.2091 sec
PD 1.7909 sec
PWI 4.50 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 30



C:\P\inl\lambdambda\COMMON\DEFAULT.T.AL

DFILE CONNT
DATIM Wed Sep 28 20:07:27 2005
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 16384
FREQU 7993.60 Hz
SCANS 16
ACQTH 2.0496 sec
PD 4.9504 sec
PWI 6.00 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16



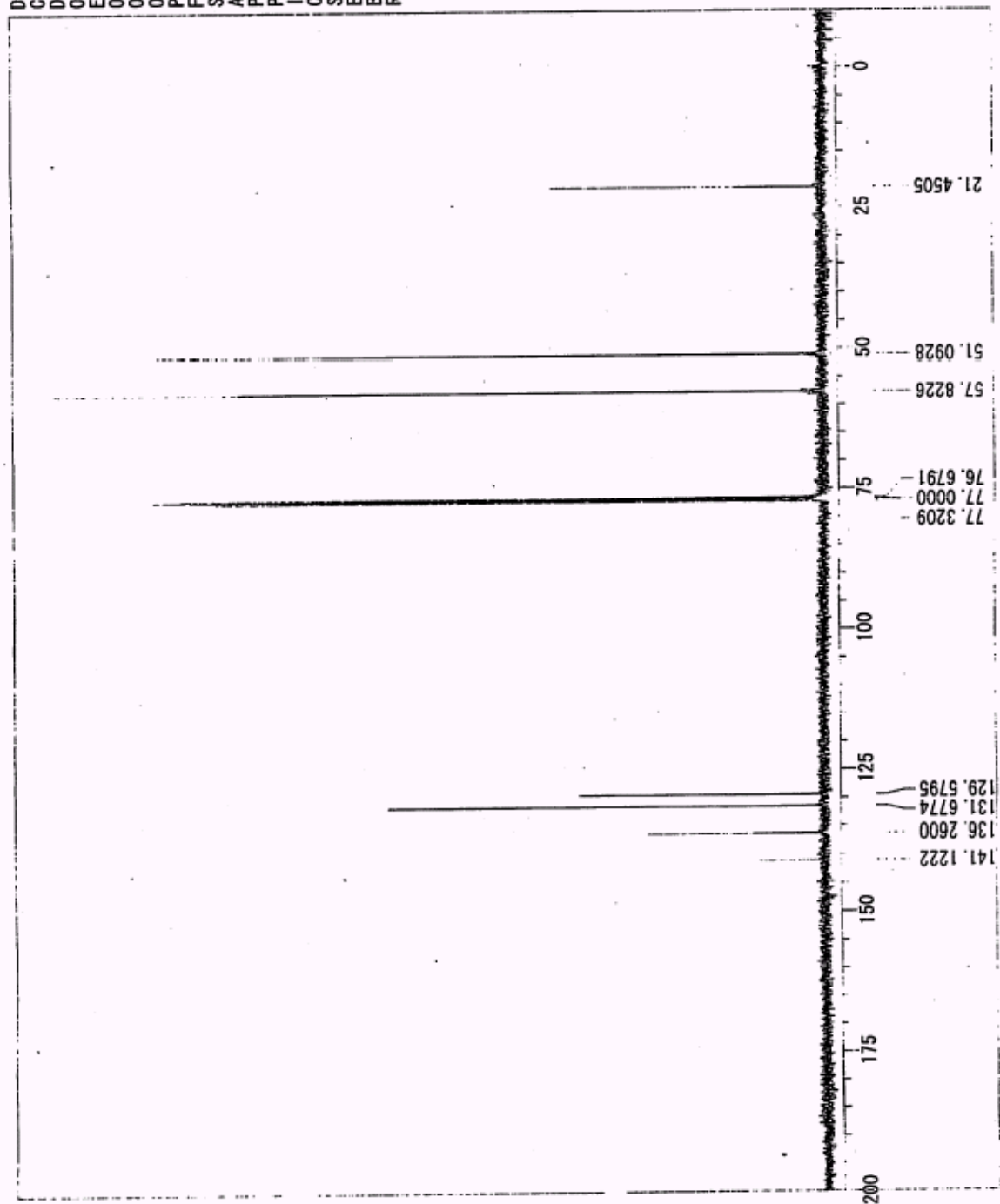
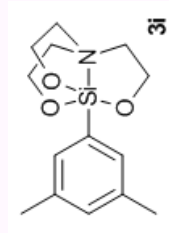
C:\W1\mlambda\COMMONY_DEFAULT.AL

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Wed Sep 28 23:27:27 2005
13C
bcm

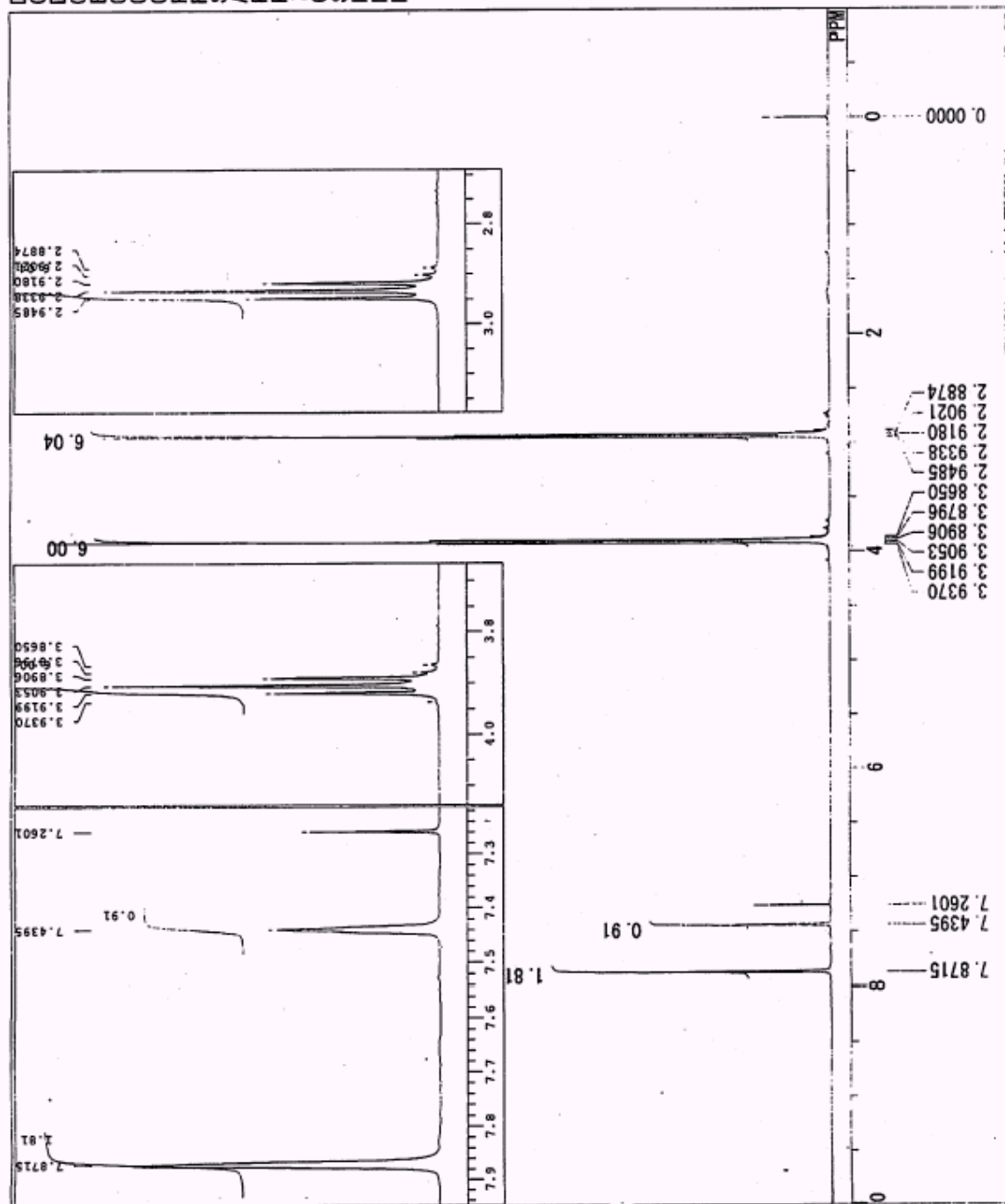
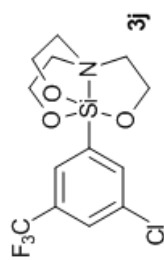
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
788
1.2091 sec
1.7909 sec
4.50 usec

1H
25.8 c
CDCL3
77.00 ppm
1.20 Hz
30



C:\Documents and Settings\lambdi

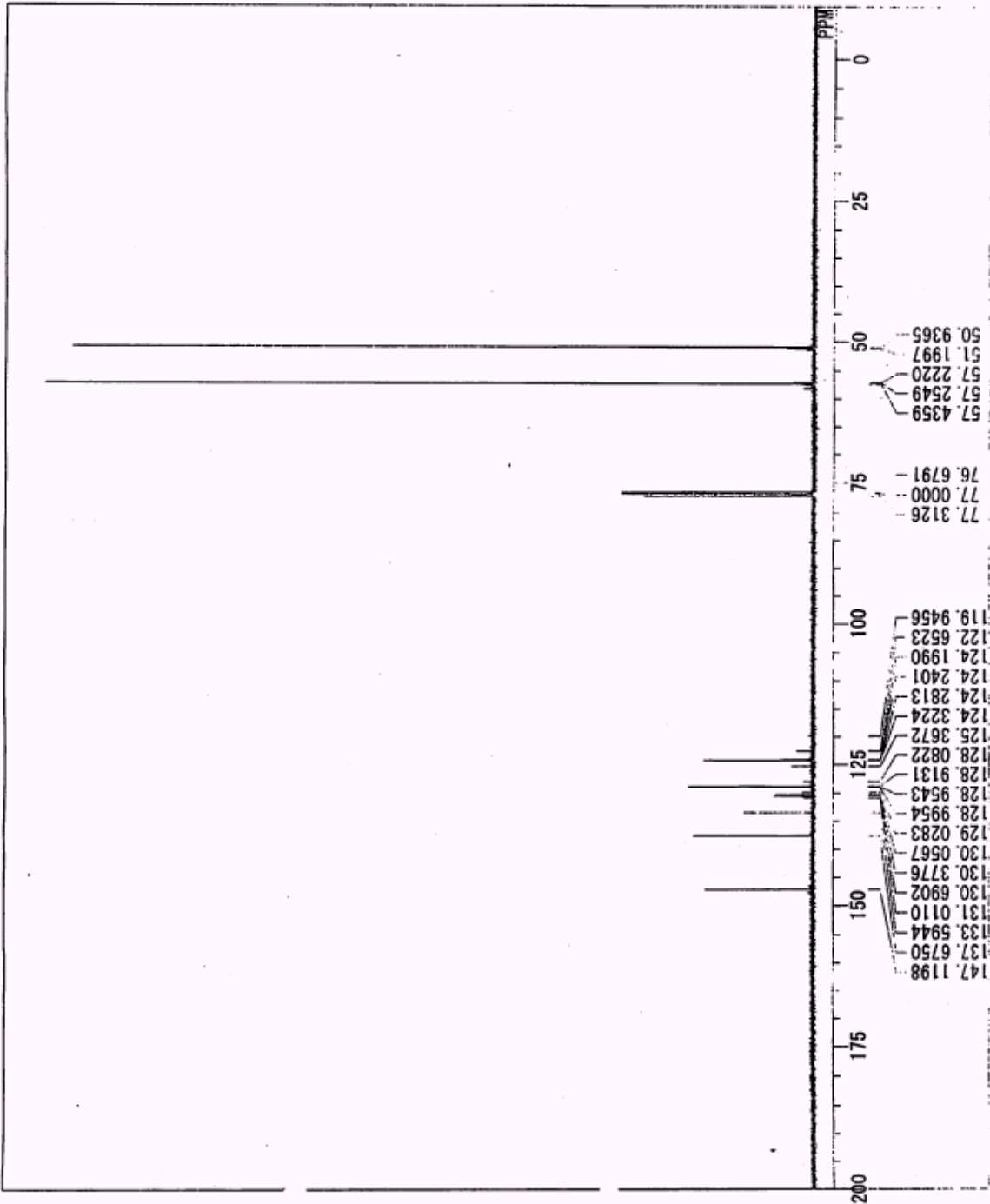
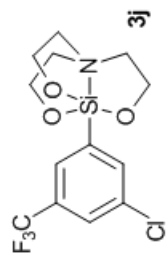
DFILE
 COMNT
 DATIM Thu Dec 08 10:39:29 2005
 OBNUC 1H
 EXMOD non
 OBFRO 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 16384
 FREQU 7993.60 Hz
 SCANS 16
 ACQTH 2.0496 sec
 PD 4.9504 sec
 PW1 6.00 usec
 TRNUC 1H
 CTEMP 25.5 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 12

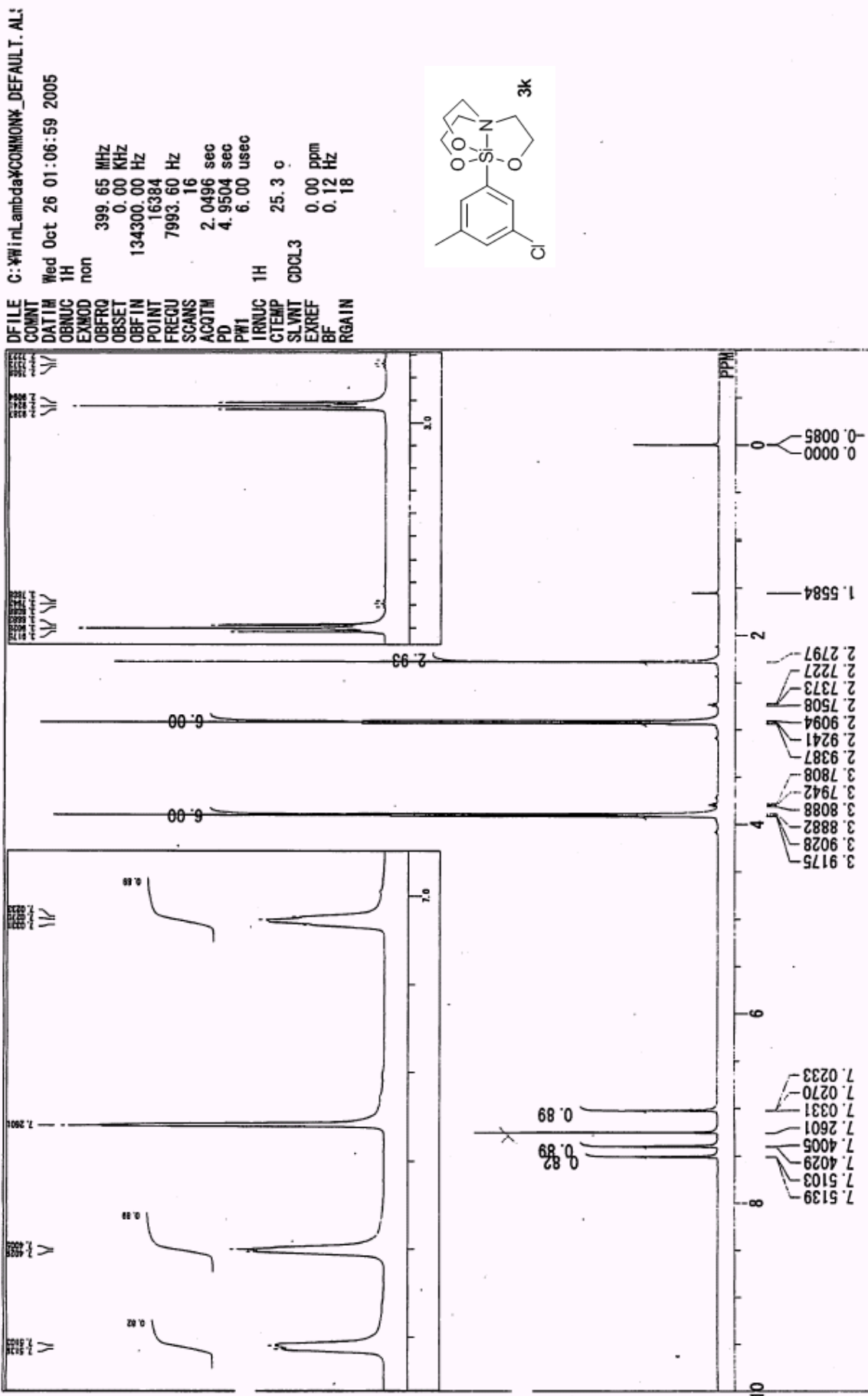


C:\Documents and Settings\lambdi

DFILE
COMINT
DAT1M
OBNUC
EXMOD
OBFRQ
OBSET
OBF IN
POINT
FREQU
SCANS
ACQTH
PD
PHT
IRNUC
GTEMP
SLVNT
EXREF
BF
RGAIN

Thu Dec 08 15:40:55 2005
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
1024
1.2091 sec
1.7909 sec
4.50 usec
1H
26.3 C
CDCL3
77.00 ppm
0.12 Hz
29





C:\Documents and Settings\lambdambda

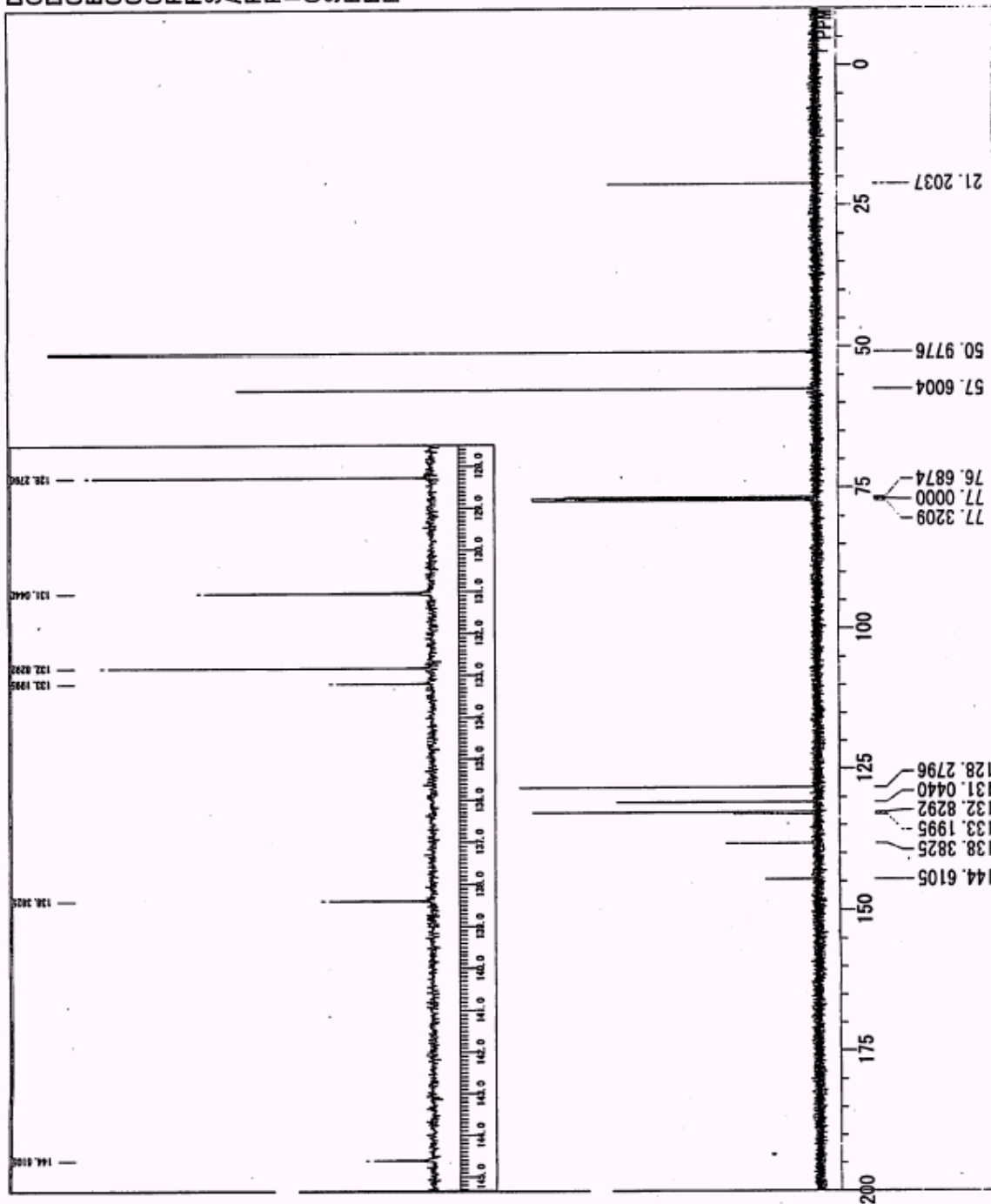
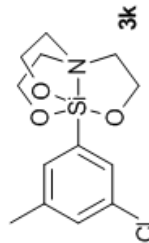
DFILE
COMNT
DATIN
OBNUC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Tue Oct 25 23:16:52 2005

13C

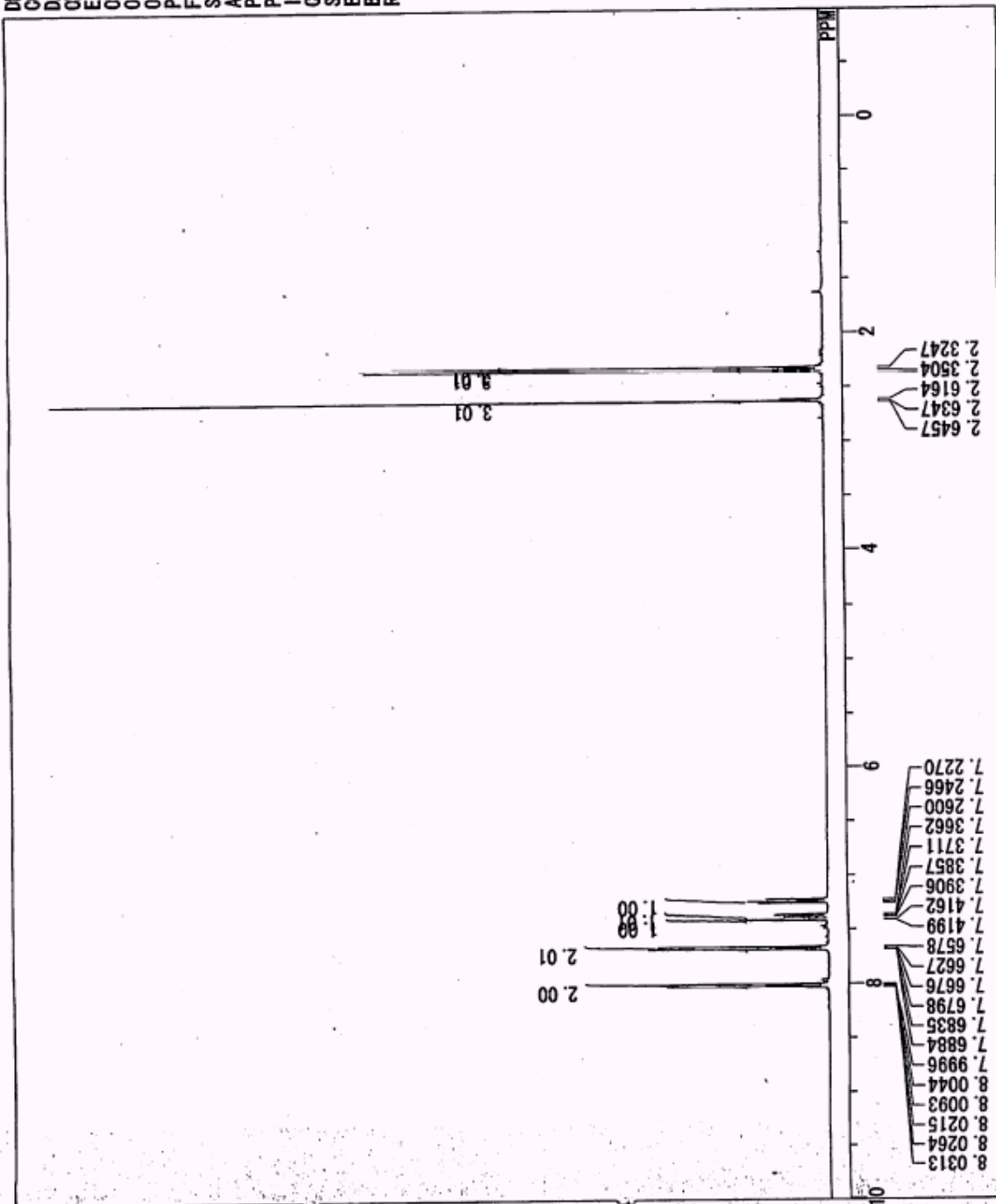
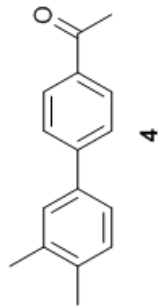
bcm

100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
258
1.2091 sec
1.7909 sec
4.50 usec
1H
26.2 °C
CDCL3
77.00 ppm
0.12 Hz
29



C:\Documents and Settings\lambdt

DFILE COMINT
DATIM Thu Feb 09 10:35:23 2006
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 16384
FREQU 7993.60 Hz
SCANS 16
ACQTM 2.0496 sec
PD 4.9504 sec
PRT 6.00 usec
IRNUC 1H
GTEMP 25.0 °C
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.15 Hz
RGAIN 15



C:\NW1\InLambda\COMMONW_DEFAULT.AL

DF ILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTH
PD
PHI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Thu Feb 09 10:51:17 2006
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
300
1.2091 sec
1.7909 sec
4.50 usec
26.0 c
CDCL3
77.00 ppm
0.12 Hz
30

