

¹³C NMR(2) of 2·5a

Current Data Parameters
 NAME tube3
 EXPNO 3333
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000921
 Time 14.19
 INSTRUM dirx500
 PROBHD 5 mm BBI 1H-B
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 9040
 DS 2
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 3251
 DW 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 d11 0.03000000 sec
 d12 0.00002000 sec
 PL13 24.00 dB
 D1 2.00000000 sec
 CDPORG2 waltz16
 PCPD2 100.00 usec
 SFO2 500.1320005 MHz
 NUC2 1H
 PL2 -1.00 dB
 PL12 24.00 dB
 P1 13.00 usec
 SFO1 125.7736214 MHz
 NUC1 13C
 PL1 -2.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7578237 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 172.000 ppm
 F1 21630.35 Hz
 F2P 120.000 ppm
 F2 15090.94 Hz
 PPMCM 2.60000 ppm/cm
 HZCM 326.97034 Hz/cm

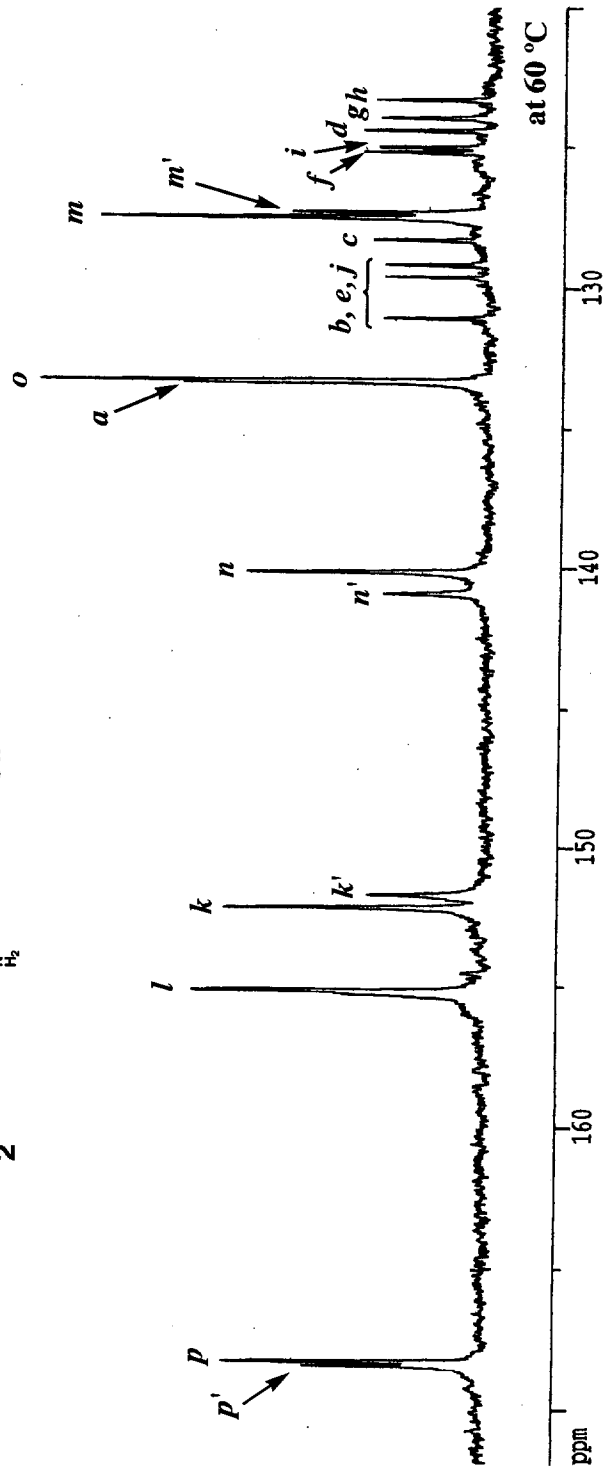
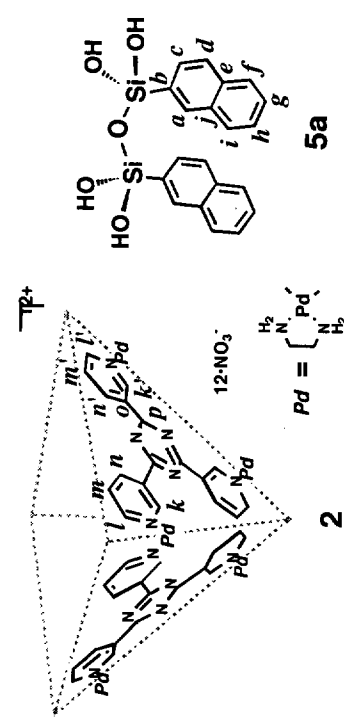
123.334
 124.004
 124.465
 125.037
 125.207
 127.323
 127.496
 128.275
 129.171
 129.611
 131.094
 133.359
 133.410

140.170
 140.960

151.743
 152.217
 154.533
 155.194

168.330
 168.471

ppm



DEPT of 2.5a

Current Data Parameters
 NAME bowl4
 EAPNO 6
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20001010
 Time 15.31
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG dept135
 TD 65536
 DZ0
 SOLVENT D2O
 NS 1355
 DS 4

SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 16384
 DM 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 P1 6.40 usec
 P2 12.80 usec
 P3 8.40 usec
 P4 16.80 usec

CNSTR2 145.0000000
 d2 0.00344828 sec
 d12 0.00002000 sec
 DELTA 0.00000815 sec
 DI 2.00000000 sec
 PL2 4.00 dB
 SFO2 500.1320005 MHz
 NUC2 1H
 SFO1 125.7736214 MHz
 NUC1 13C

PL1 2.00 dB
 PL12 16.00 dB
 CPDPRG2 waltz16
 PCPD2 81.00 usec

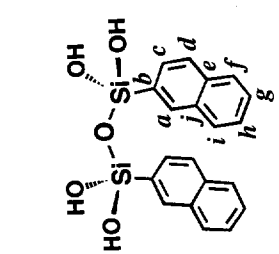
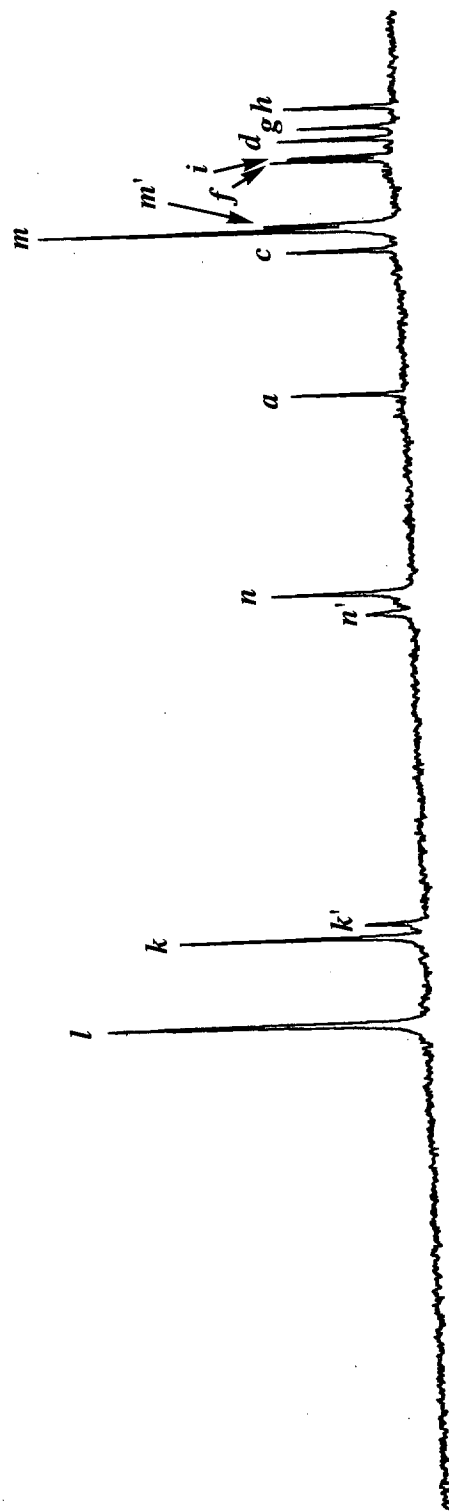
F2 - Processing parameters

SI 32768
 SP 125.7578471 MHz
 EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

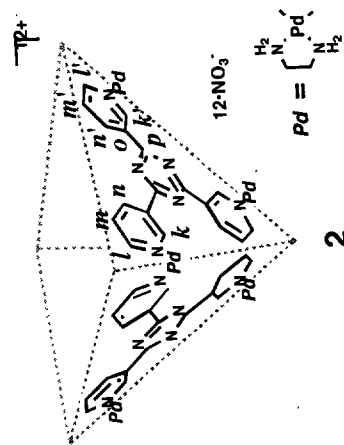
1D NMR plot parameters

CX 20.00 cm
 F1P 172.000 ppm
 F1 21630.35 Hz
 F2P 120.000 ppm
 F2 15090.34 Hz
 PPMCM 2.60000 ppm/cm
 HZCM 326.97040 Hz/cm

155.093
 152.092
 151.620
 140.846
 140.091
 134.014
 133.218
 128.182
 127.402
 127.241
 125.077
 124.938
 124.328
 123.907
 123.219

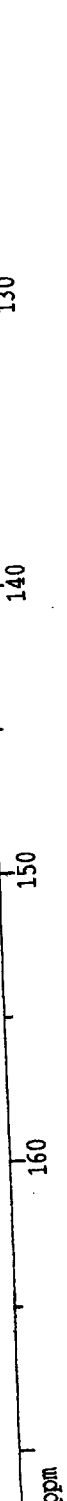


5a



2

at 60 °C



²⁹Si NMR of 2.5a

Current Data Parameters
 NAME bow14
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

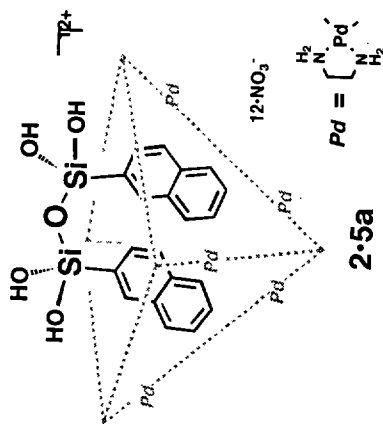
Date_ 20001004
 Time 19.33
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG dept45
 TD 65536
 SOLVENT CDCl3
 NS 3744
 DS 4
 SMH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 7298.2
 DM 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 P1 7.50 usec
 P2 15.00 usec
 P3 7.60 usec
 P4 15.20 usec
 CNST2 20.000000
 G2 0.0250000 sec
 d12 0.0002000 sec
 DELTA 0.0000955 sec
 D1 2.0000000 sec
 PL2 -4.00 dB
 SF02 500.1320005 MHz
 NUC2 ¹H
 SF01 99.3617370 MHz
 NUC1 ²⁹Si
 PL1 2.00 dB
 PL12 16.00 dB
 CDDPRG2 waltz16
 PCPD2 81.00 usec

F2 - Processing parameters

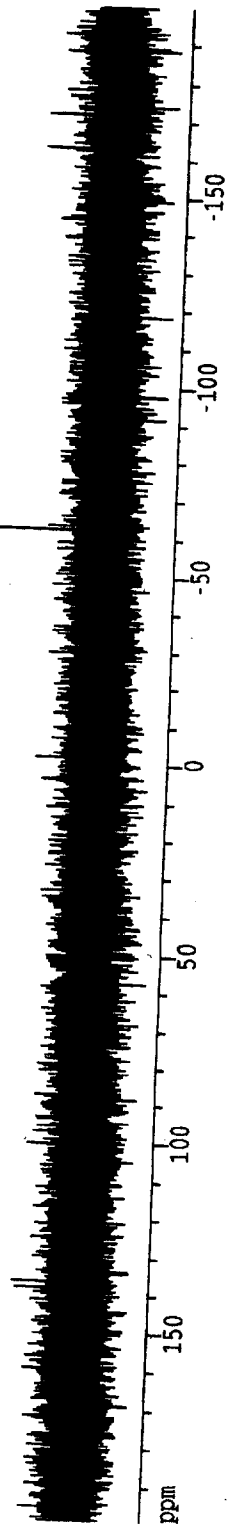
SI 32768
 SF 99.3617534 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 199.522 ppm
 F1 19824.83 Hz
 F2P -199.853 ppm
 F2 -19857.71 Hz
 PPMCM 19.96872 ppm/cm
 HZCM 1984.12708 Hz/cm

-62.372



at 60 °C



ppm

HH-COSY (1) of 2-5a

Current Data Parameters
 NAME bow13
 EXPNO 3
 PROCNO 1

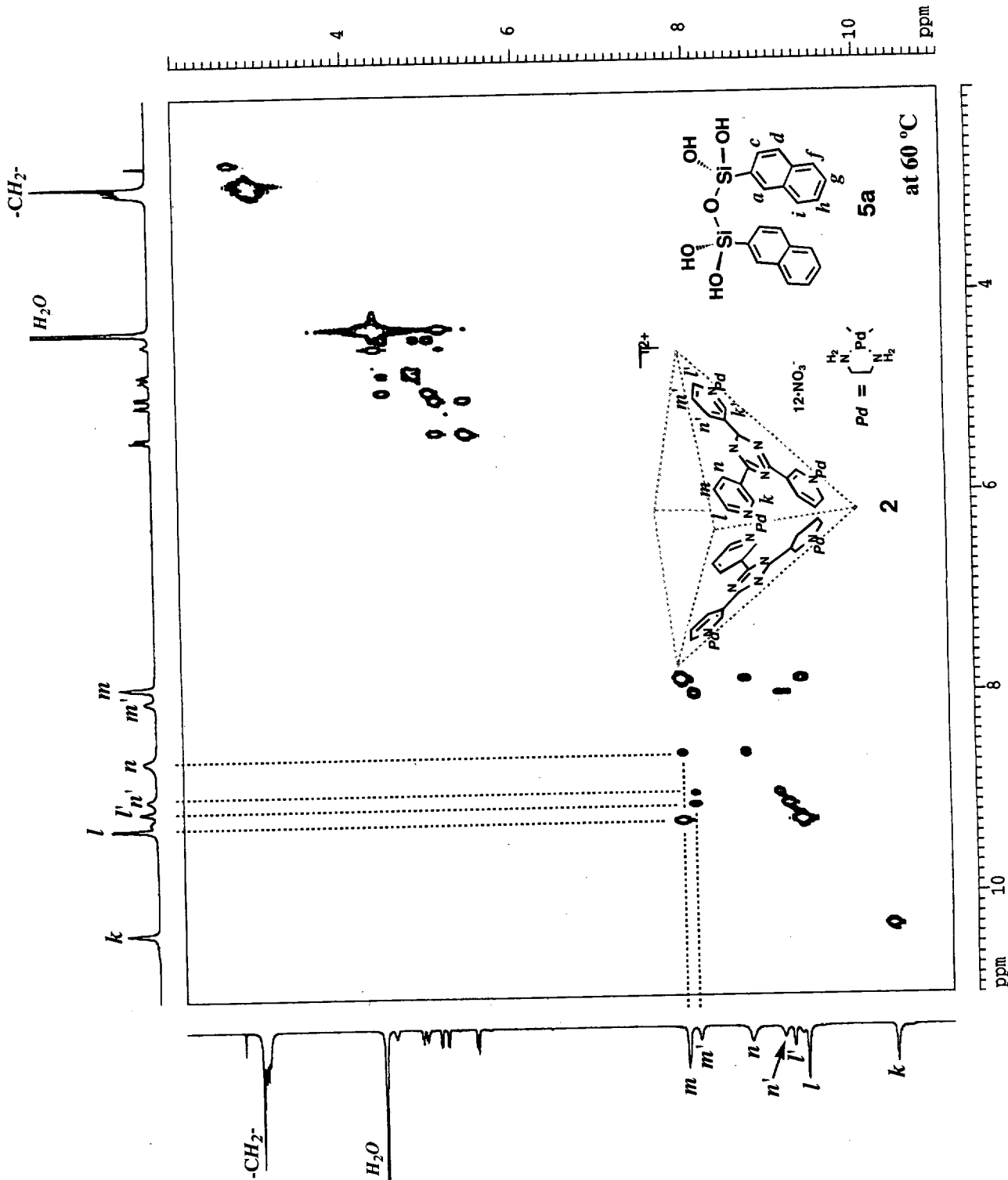
P2 - Acquisition Parameters
 Date_ 20000908
 Time 0.18
 INSTRUM drs500
 PROBD 5 mm BBI 1H-B
 PULPROG cosy90
 TD 2048
 SOLVENT D2O
 NS 64
 DS 4
 SWH 7002.801 Hz
 FIDRES 3.419337 Hz
 AQ 0.1462772 sec
 PC 512
 DM 71.400 usec
 DE 6.00 usec
 FE 333.0 K
 FI 0.0000300 sec
 DI 2.0000000 sec
 P1 7.10 usec
 SFO1 500.1324528 MHz
 NUC1 1H
 PL1 1.00 dB
 TNO 0.00014280 sec

P1 - Acquisition parameters
 ND0 1
 TD 256
 SFO1 500.1325 MHz
 FIDRES 27.354692 Hz
 SW 14.002 ppm

P2 - Processing parameters
 SI 1024
 SP 500.1301943 MHz
 WDM SINE
 SINE 0
 SB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

P1 - Processing parameters
 SI 1024
 WC2 QF
 SP 500.1301960 MHz
 WDM SINE
 SINE 0
 SB 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 15.00 cm
 CX1 15.00 cm
 F2P/LO 11.000 ppm
 F2LO 5501.43 Hz
 F2PHI 2.000 ppm
 F2PHI 1000.22 Hz
 F1P/LO 11.000 ppm
 F1LO 5501.43 Hz
 F1PHI 1.996 ppm
 F1PHI 998.46 Hz
 F2PRACH 0.60001 ppm/cm
 F2PRACH 300.08075 Hz/cm
 F1PRACH 0.60024 ppm/cm
 F1PRACH 300.19833 Hz/cm



HH-COSY (2) of 2-5a

Current Data Parameters
 NAME
 EXPNO 3
 PROCNO 1

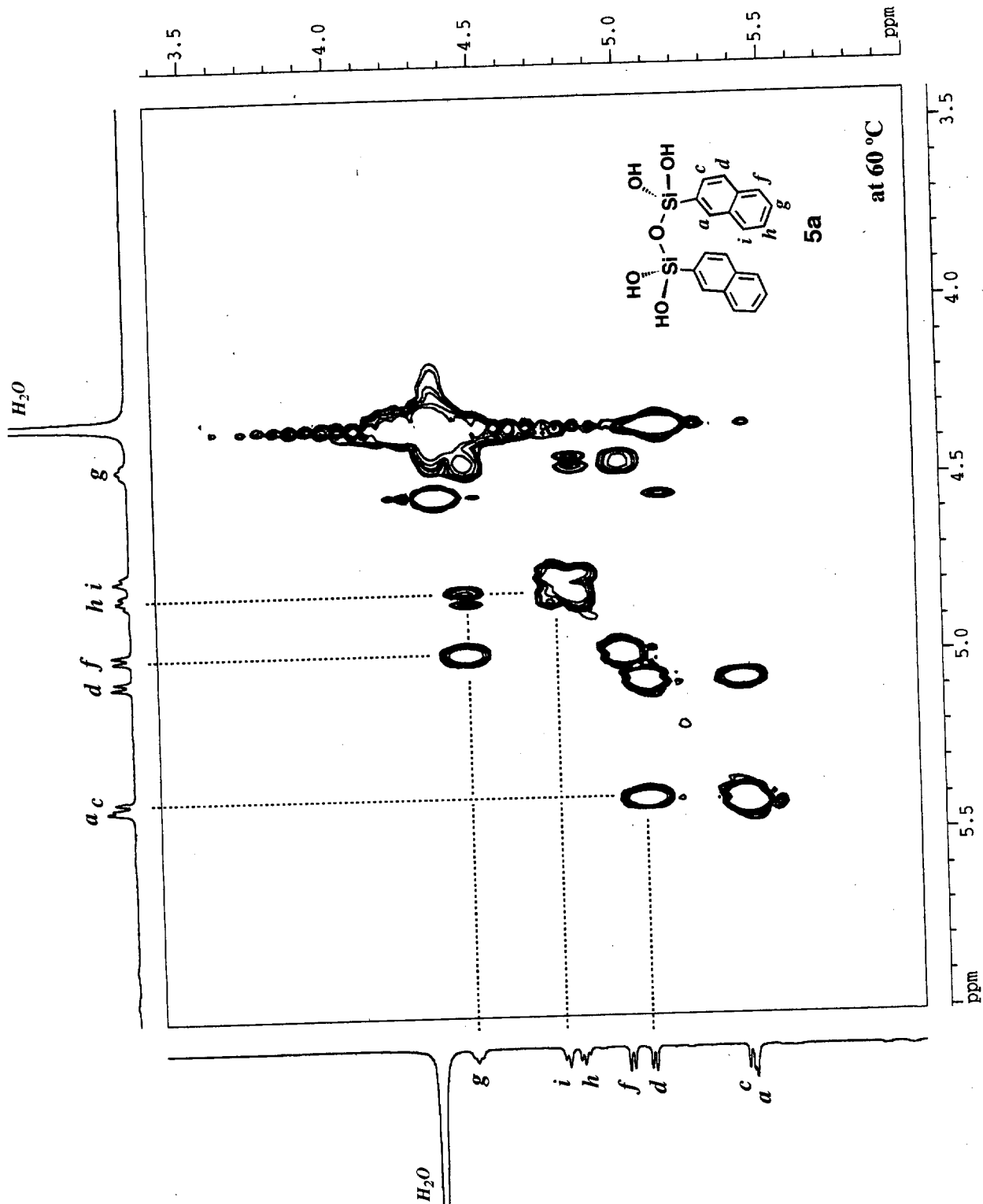
P2 - Acquisition Parameters
 Date_ 26000908
 Time 0.18
 INSTRUM dir500
 PROBRD 5 mm BBI 1H-B
 PULPROG cosy90
 TD 2048
 SOLVENT D2O
 NS 64
 DS 4
 SNR 7002.801 Hz
 FIDRES 3.41937 Hz
 AQC 0.1462772 sec
 RG 512
 DW 71.400 usec
 DE 6.00 usec
 TE 333.0 K
 d0 0.00000300 sec
 d1 2.00000000 sec
 P1 7.10 usec
 SFO1 500.1324528 MHz
 NUC1 1H
 PL1 -1.00 dB
 IN0 0.00014280 sec

F1 - Acquisition parameters
 ND0 254
 TD 500.1225 MHz
 SFO1 27.354692 Hz
 FIDRES 14.002 Dpm
 SN

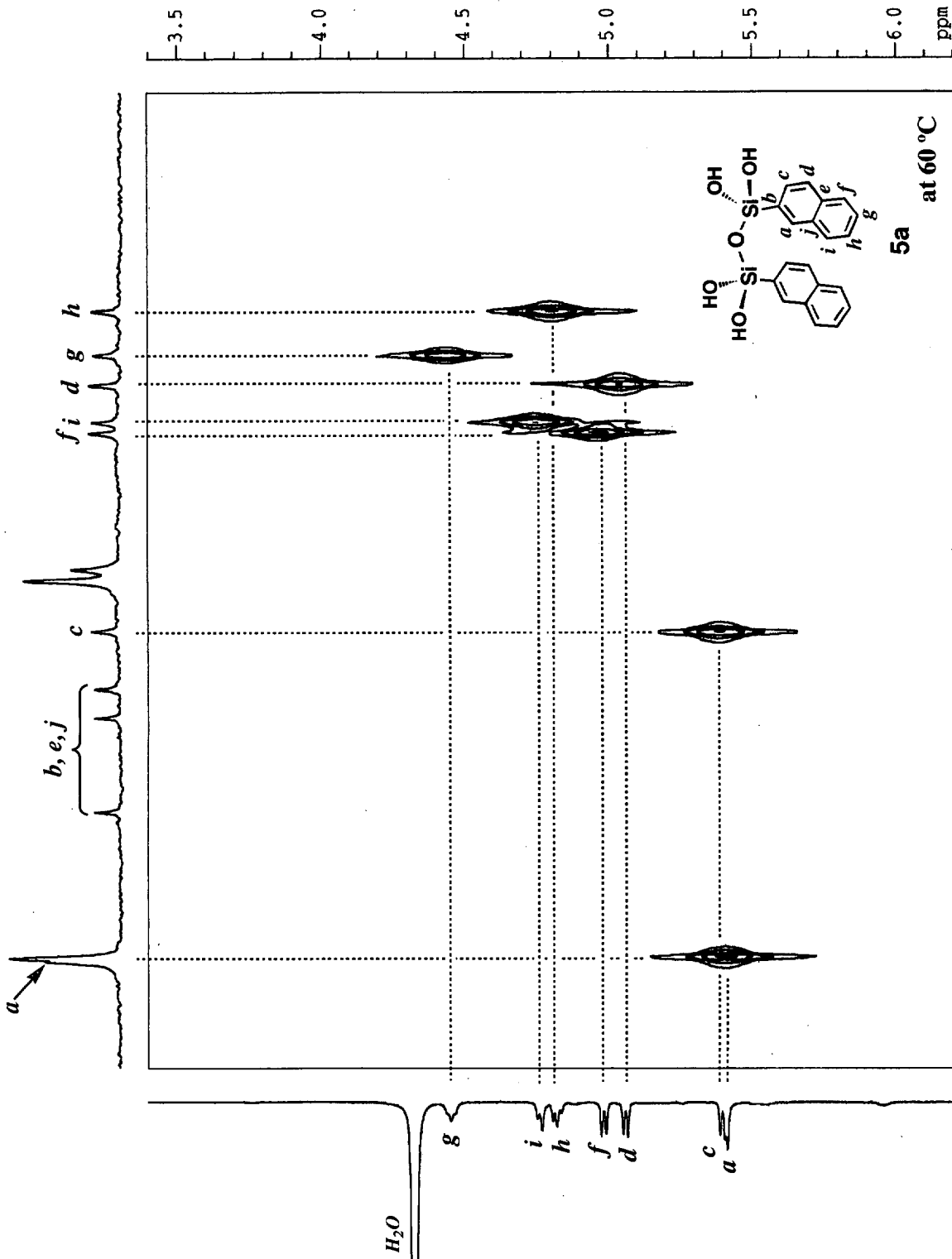
P2 - Processing parameters
 SI 1024
 SF 500.1301943 MHz
 SINE 0
 WDW 0
 SSB 0.00 Hz
 LB 0
 GB 0
 EC 1.00

F1 - Processing parameters
 SI 1024
 MC2 OF
 SF 500.1301960 MHz
 SINE 0
 WDW 0
 SSB 0.00 Hz
 LB 0
 GB 0

2D NMR plot parameters
 CY2 15.00 cm
 CX1 15.00 cm
 P2F0 6.011 ppm
 P2F1 3006.04 Hz
 P2F2 3.426 ppm
 P2F3 1713.53 Hz
 P2F4 6.000 ppm
 P2F5 3000.98 Hz
 P2F6 3.375 ppm
 P2F7 1687.96 Hz
 P2F8 0.17229 ppm/cm
 P2F9 86.16732 Hz/cm
 P2F10 0.17502 ppm/cm
 P2F11 87.53506 Hz/cm



CH-COSY (2) of 2·5a



Current Data Parameters
 NAME: bow14
 EXPNO: 9
 PROCNO: 1

F2 - Acquisition Parameters
 Date_ Time: 20000101 22:19
 INSTRUM: drs500
 PROBRD: 5 mm BBO BB-1
 PULPROG: zgpg30
 SOLVENT: D2O
 NS: 148
 DS: 4
 SFR: 6169.935 Hz
 ZONES: 2
 AQ: 0.1507951 sec
 RG: 16384
 DW: 61.200 usec
 DE: 6.00 usec
 TE: 300.0 K
 F1: 47.0 usec
 F2: 12.50 usec
 D0: 0.03000300 sec
 CHSF2: 145.000000 sec
 d2: 0.03344828 sec
 CHFT1: 3.000000 sec
 CHFT2: 0.03000000 sec
 d1: 0.03000000 sec
 d12: 0.03002000 sec
 d13: 0.03000000 sec
 d14: 2.03000000 sec
 F2: 4.00 dB
 F3: 8.40 usec
 F4: 500.13344828 MHz
 NUC2: 1H
 SF01: 125.7760832 MHz
 WPC1: 13C
 F1: 2.00 dB
 F2: 16.00 dB
 C12PC2: waltz16
 PCPC2: 81.00 usec
 INO: 0.03009840 sec

F1 - Acquisition parameters
 NU0: 128
 SF01: 500.1334 MHz
 FIDRES: 39.697662 Hz
 SW: 10.160 ppm

F2 - Processing parameters
 SI: 704
 SF: 125.7578293 MHz
 GPCINE: 2
 LB: 0.00 Hz
 GB: 0
 PC: 1.00

F1 - Processing parameters
 SI: 1024
 SF: 500.1302938 MHz
 GPCINE: 2
 LB: 0.00 Hz
 GB: 0

2D NMR plot parameters
 CZ1: 15.00 cm
 CZ2: 15.00 cm
 F2FLO: 135.032 ppm
 F1FLO: 16981.27 Hz
 F2H1: 1302.912 ppm
 F1H1: 6.202 ppm
 F2F0: 3101.83 Hz
 F1F0: 3.394 ppm
 F1H2: 1697.53 Hz
 F2F0H2: 134.59843 ppm/cm
 F1F0H2: 0.18719 ppm/cm
 F2F0CZ: 93.62032 Hz/cm

NOESY of 2.5a

Current Data Parameters
 NAME 2.5a
 EXPNO 12
 PROCNO 1

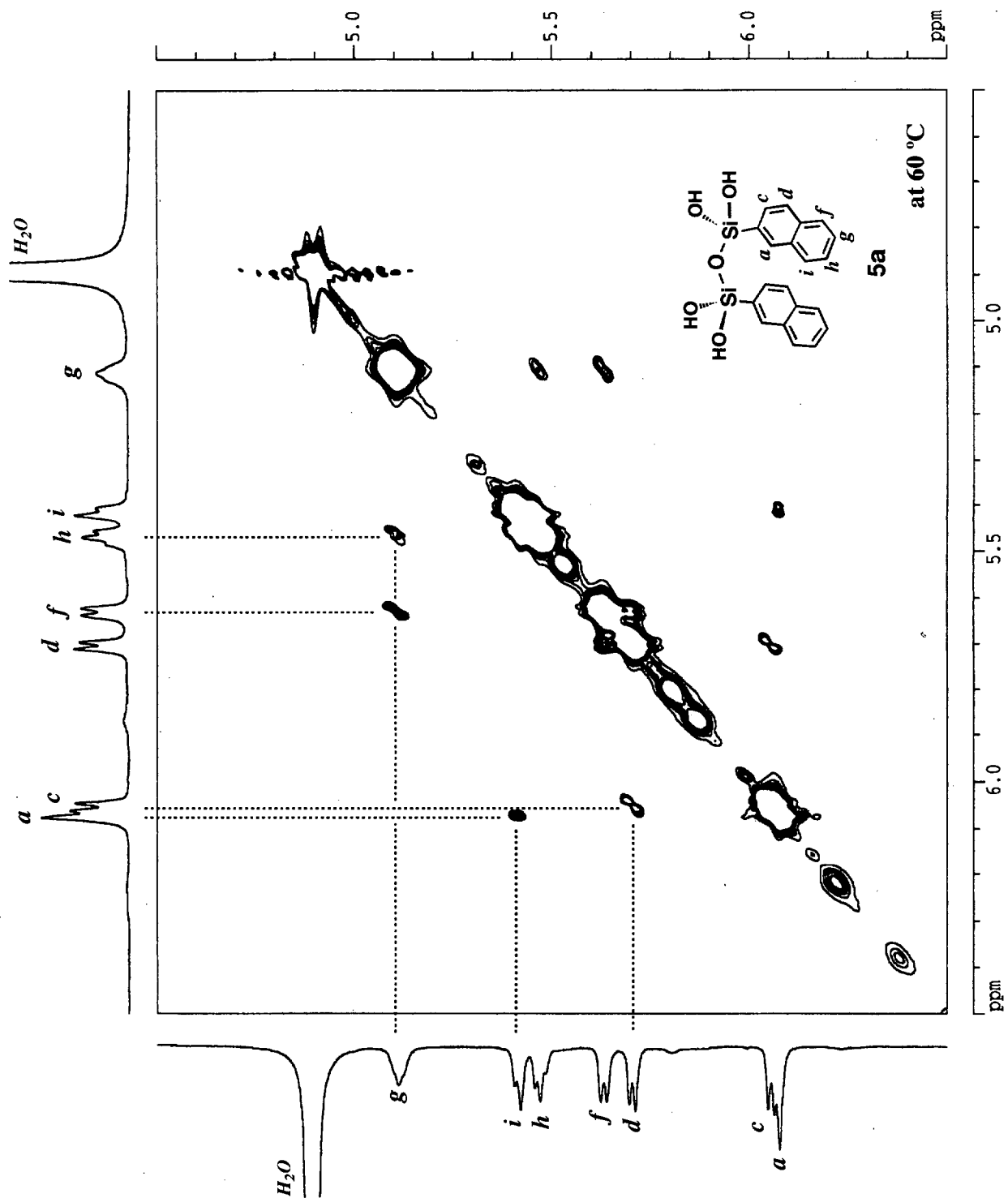
F2 - Acquisition Parameters
 Date_ 2000101
 Time 1.56
 INSTRUM dr500
 PROBRD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 2048
 SOLVENT D2O
 NS 32
 DS 4
 SWH 2042.484 Hz
 FIDRES 0.997306 Hz
 AQ 0.5014004 sec
 RG 256
 DW 244.800 usec
 DE 6.00 usec
 TE 300.0 K
 D0 0.00000000 sec
 D1 2.00000000 sec
 P1 8.40 usec
 SFO1 500.1323920 MHz
 NUC1 1H
 P11 -4.00 dB
 D8 0.30000001 sec
 INO 0.00024480 sec

F1 - Acquisition Parameters
 MD0 417
 TD 500.1324 MHz
 FIDRES 4.898042 Hz
 SW 4.084 ppm

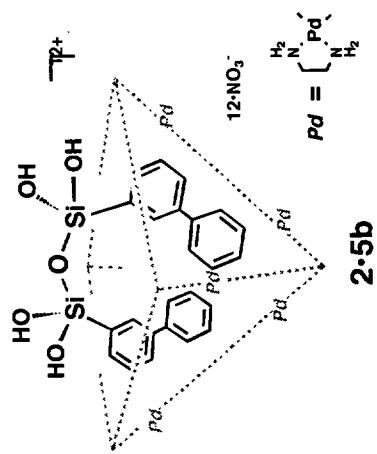
F2 - Processing parameters
 SI 1024
 SF 500.1299079 MHz
 WDW GEMME
 LB 0.00 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 TPR1
 SP 500.1299079 MHz
 WDW GEMME
 SSB 2
 LB 0.00 Hz
 GB 0

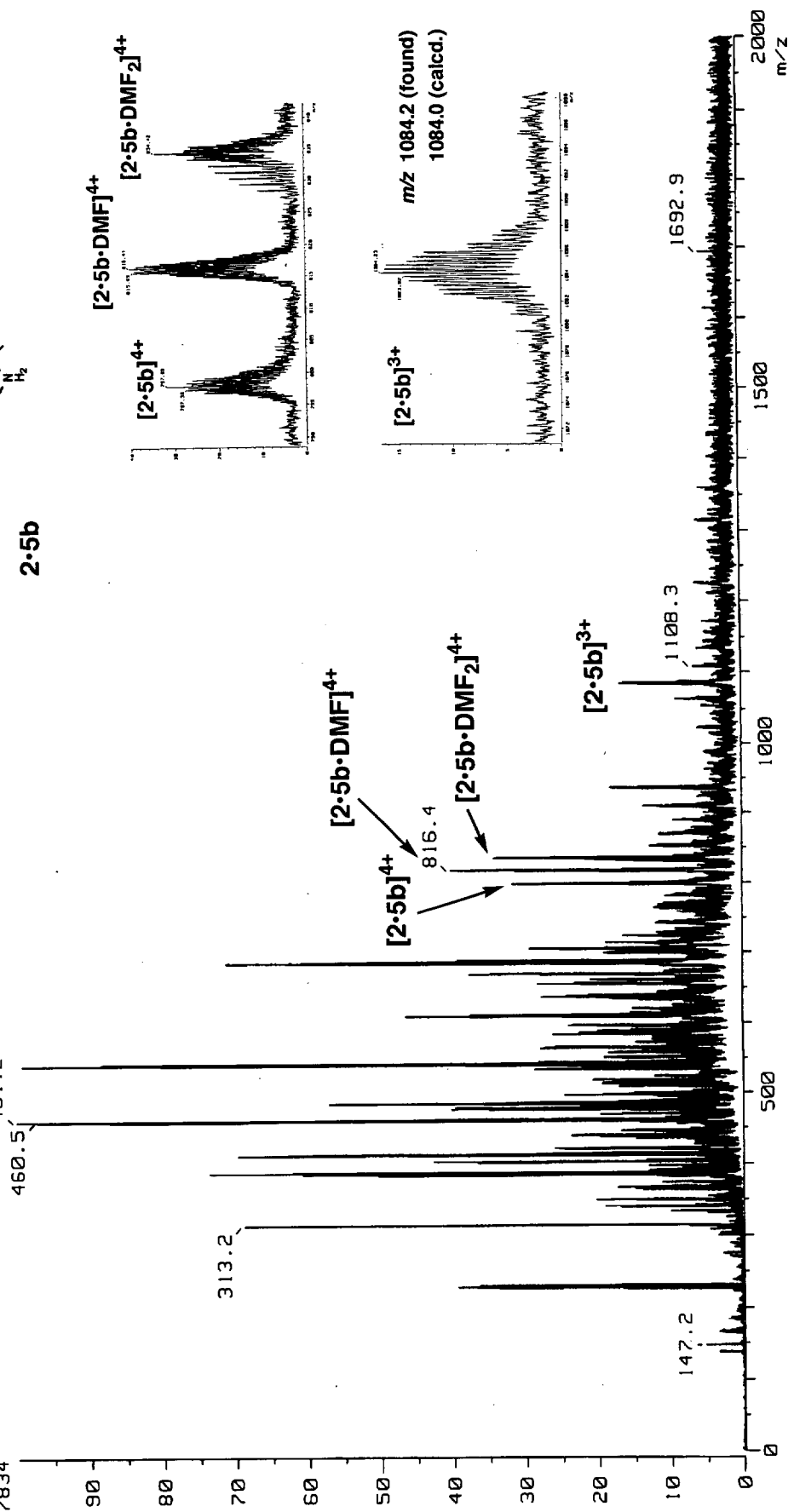
2D NMR plot parameters
 CX2 15.00 cm
 CX1 15.00 cm
 F2FLO 6.500 ppm
 F2LO 3250.84 Hz
 F2PHI 4.500 ppm
 F2HI 2250.58 Hz
 F1FLO 6.500 ppm
 F1LO 3250.84 Hz
 F1PHI 4.500 ppm
 F1HI 2250.58 Hz
 F2PCPKC 0.13333 ppm/cm
 F2HZCN 66.68399 Hz/cm
 F1PCPKC 0.13333 ppm/cm
 F1HZCN 66.68399 Hz/cm



CSI MS of 2-5b



[Mass Spectrum]
 Date : 30-Jun-2000 17:50
 Data : 000630-Yoshizawa-006
 Sample : -
 Note : -
 Inlet : Direct
 Ion Mode : ESI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 9.14 min
 Scan# : (1,59)
 BP : m/z 461.2257
 Int. : 77.07
 Output m/z range : 0.0000 to 2000.0000
 47677834
 Cut Level : 0.00 %



¹H NMR(1) of 2-5b

Current Data Parameters
 NAME bow16
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

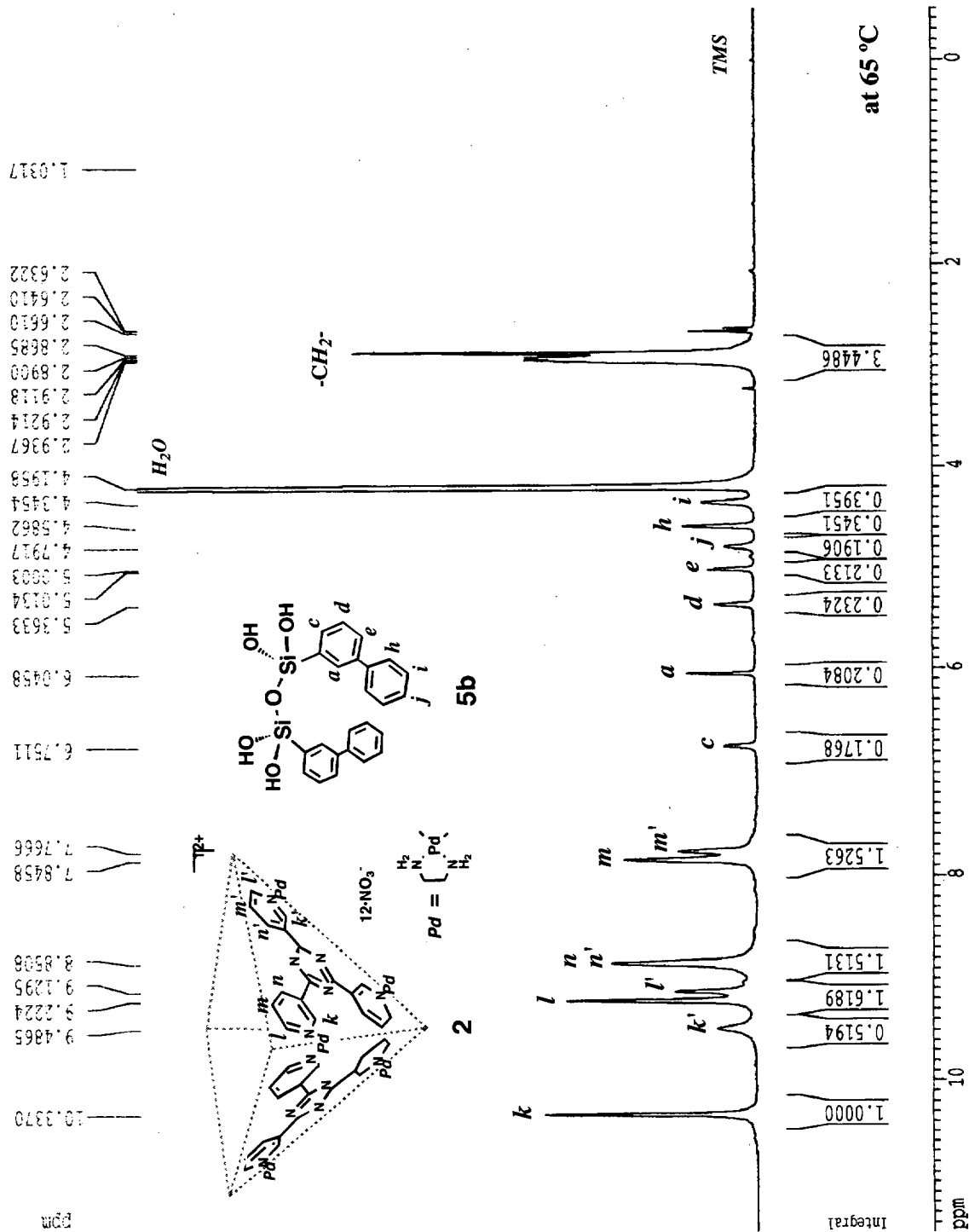
Date_ 20001109
 Time 11.09
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.315264 Hz
 AQ 1.5860212 sec
 RG 128
 DW 48.400 usec
 DE 6.00 usec
 TE 338.0 K
 D1 1.00000000 sec
 F1 8.40 usec
 SFO1 500.1330885 MHz
 NUC1 1H
 PL1 -4.00 dB

F2 - Processing parameters

SI 16384
 SF 500.1302587 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

ID NMR plot parameters

CX 20.00 cm
 F1P 11.500 ppm
 F1 5751.50 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07816 Hz/cm



¹H NMR(2) of 2-5b

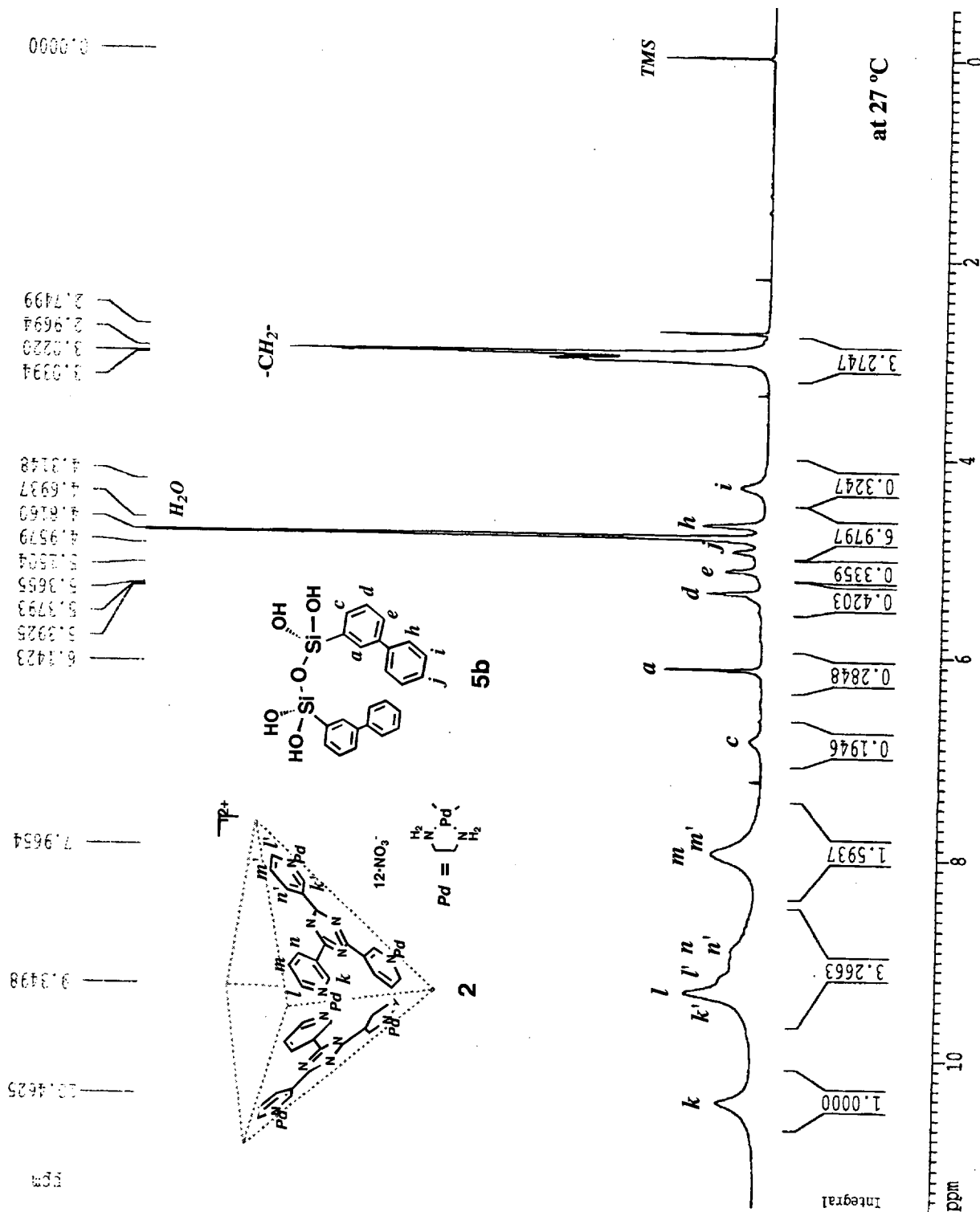
Current Data Parameters
 NAME yoshih4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20000301
 Time 13.26
 INSTRUM drx500
 PROBHD 5 mm BBI 1H-B
 PULPROG zg30
 TD 32768
 SOLVENT D2O
 NS 32
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.315264 Hz
 AQ 1.5860212 sec
 RG 181
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.10 usec
 SFOL 500.1330885 MHz
 NUCL 1H
 PL1 -1.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1299423 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 11.500 ppm
 F1 5751.49 Hz
 F2P -0.500 ppm
 F2 -250.06 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 300.07797 Hz/cm



¹³C NMR(1) of 2·5b

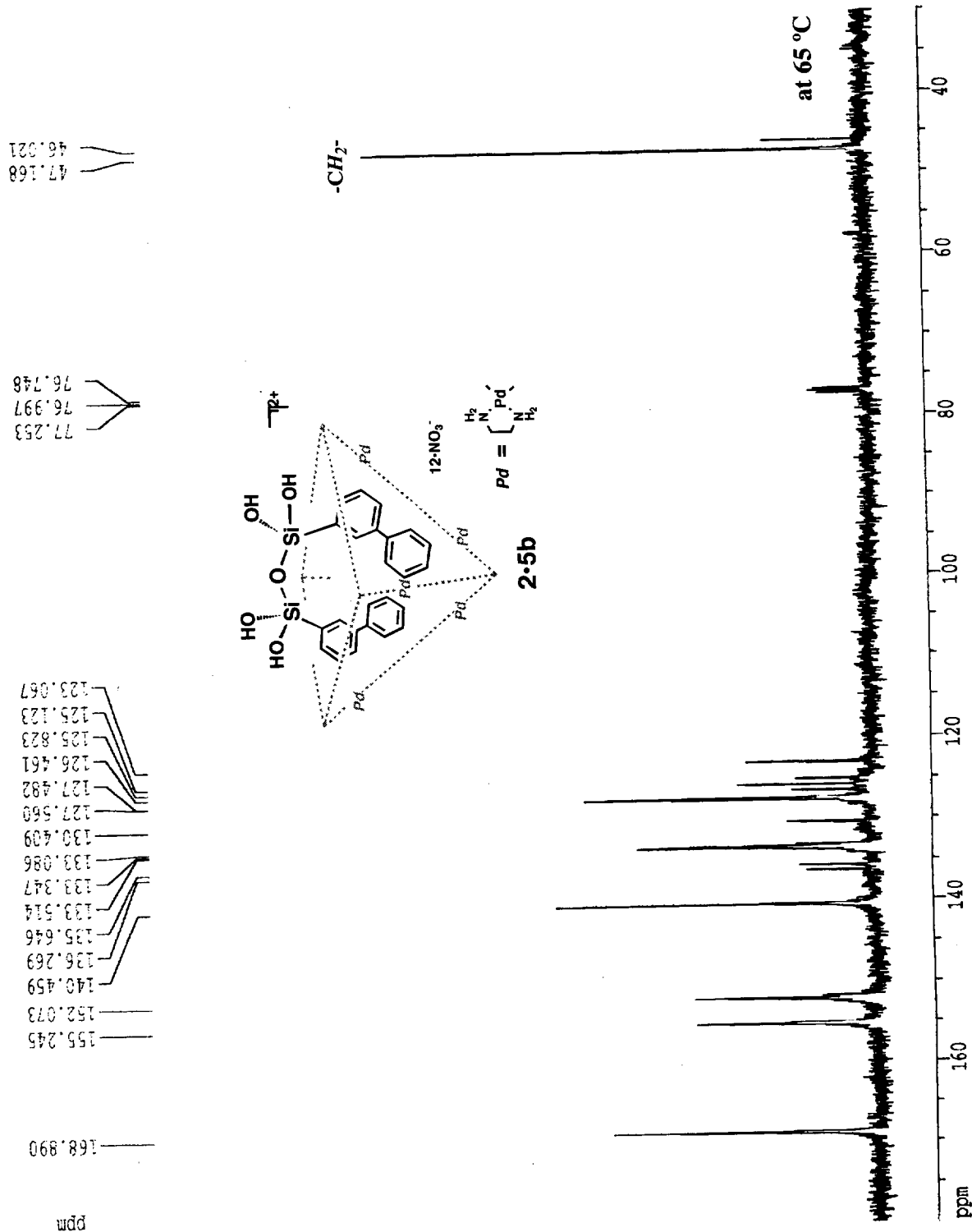
Current Data Parameters
 NAME bow16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20001109
 Time 11.18
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 4600
 DS 2
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 13004
 DW 12.600 usec
 DE 6.00 usec
 TE 338.0 K
 d11 0.0300000 sec
 d12 0.0002000 sec
 PL13 120.00 dB
 D1 2.0000000 sec
 CPDPRG2 waltz16
 PCPD2 81.00 usec
 SFO2 500.1320005 MHz
 NUC2 1H
 PL2 -4.00 dB
 PL12 16.00 dB
 P1 6.40 usec
 SFO1 125.7736214 MHz
 NUC1 13C
 PL1 2.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7578333 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CX 20.00 cm
 F1P 180.000 ppm
 F1 22636.41 Hz
 F2P 30.000 ppm
 F2 3772.74 Hz
 PPMCM 7.50000 ppm/cm
 HZCM 943.18365 Hz/cm



¹³C NMR(2) of 2-5b

Current Data Parameters
 NAME bow16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters

Date 20001109

Time 11.18

INSTRUM drx500

PROBHD 5 mm BBO BB-1

PULPROG zgpg30

TD 65536

SOLVENT D2O

NS 4600

DS 2

SWH 39682.539 Hz

FIDRES 0.605507 Hz

AQ 0.8258036 sec

RG 13004

DW 12.600 usec

DE 6.00 usec

TE 338.0 K

d11 0.03000000 sec

d12 0.00002000 sec

PL13 120.00 dB

D1 2.00000000 sec

CPDPRG2 waltz16

PCPD2 81.00 usec

SFO2 500.1320005 MHz

NUC2 1H

PL2 -4.00 dB

PL12 16.00 dB

P1 6.40 usec

SFO1 125.7736214 MHz

NUC1 13C

PL1 2.00 dB

F2 - Processing parameters

SI 32768

SF 125.7578333 MHz

WDW EM

SSB 0

LB 3.00 Hz

GB 0

PC 1.40

ID NMR plot parameters

CX 20.00 cm

F1P 175.000 ppm

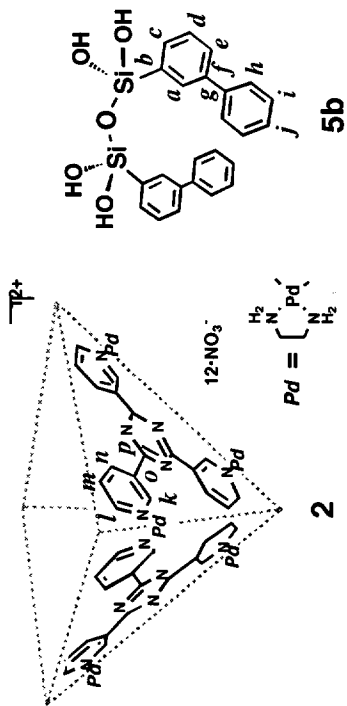
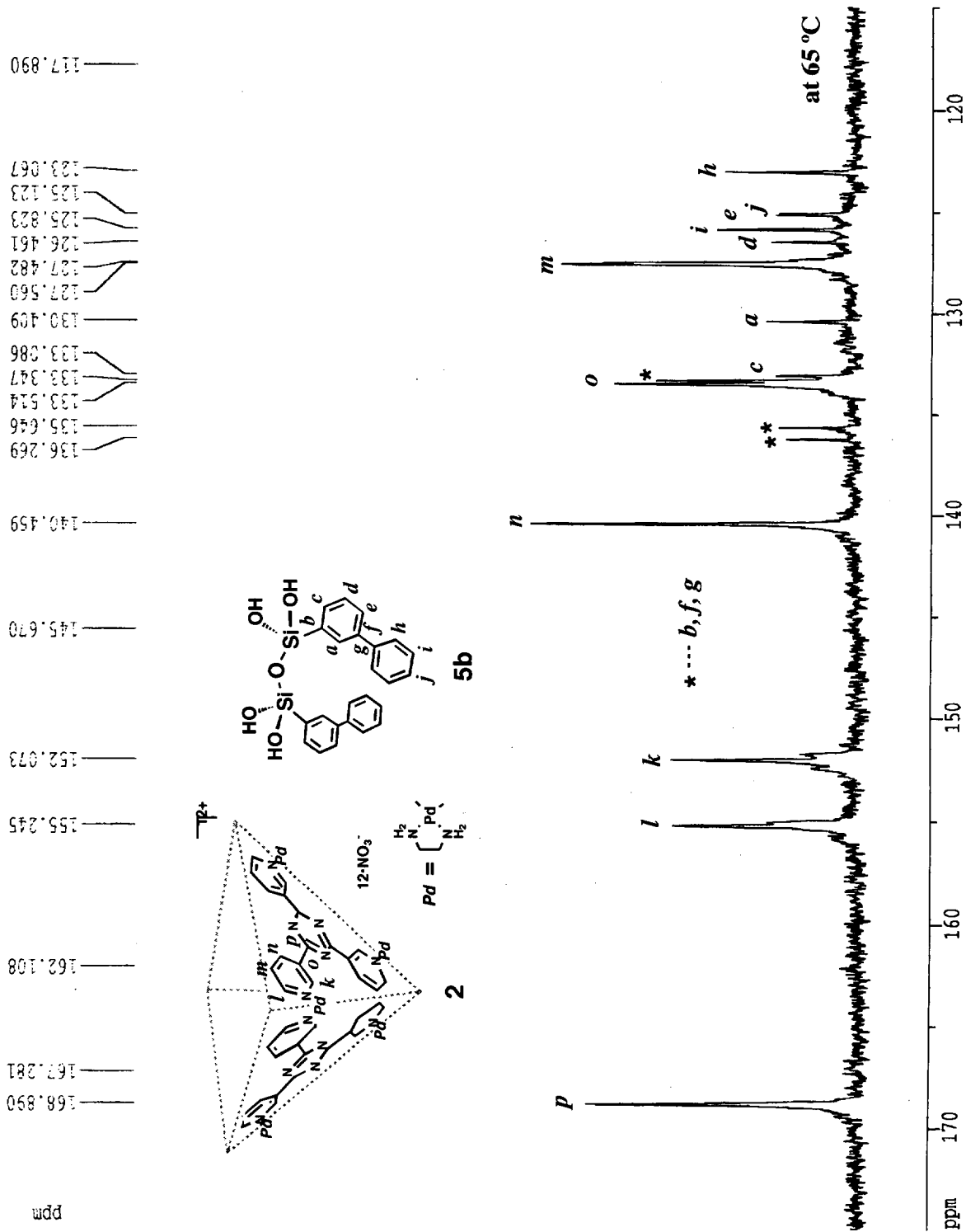
F1 22007.62 Hz

F2P 115.000 ppm

F2 14462.15 Hz

PPMCM 3.00000 ppm/cm

HZCM 377.27350 Hz/cm



Chemical Shift (ppm)	Assignment
168.890	p
167.281	
162.108	l
155.245	
152.073	k
145.670	
140.459	n
136.269	**
135.646	**
133.524	
133.347	o
133.086	
130.409	
127.560	m
127.482	
126.461	a
125.823	i
125.123	d
123.067	e
117.890	j
	h

²⁹Si NMR of 2-5b

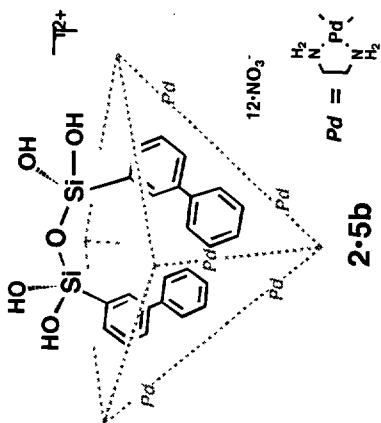
Current Data Parameters
 NAME bow11
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000613
 Time 15.04
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG dept45
 TD 65536
 SOLVENT CDC13
 NS 4043
 DS 4
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 3649.1
 DM 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 P1 7.50 usec
 P2 15.00 usec
 P3 7.50 usec
 P4 15.20 usec
 CNST2 20.0000000
 d2 0.02500000 sec
 d12 0.00002000 sec
 DELTA 0.00000955 sec
 D1 2.00000000 sec
 PL2 -4.00 dB
 SFO2 500.1320005 MHz
 NUCC2 1H
 SFO1 99.3617370 MHz
 NUCC1 29Si
 PL1 2.00 dB
 PL12 16.00 dB
 CPDPRG2 waltz16
 PCPD2 81.00 usec

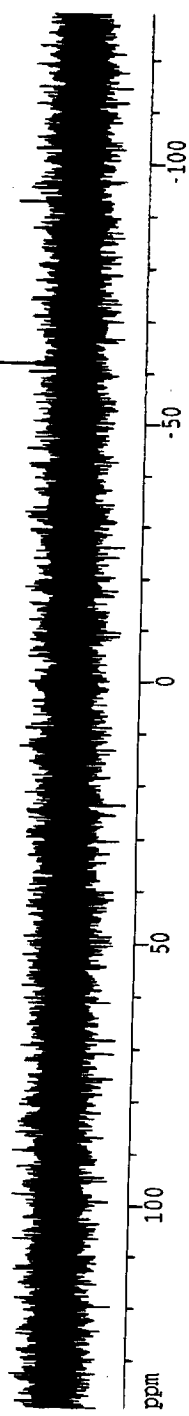
F2 - Processing parameters
 SI 32768
 SP 99.3617534 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 FIP 139.373 ppm
 F1 13848.34 Hz
 F2P -128.435 ppm
 F2 -12761.56 Hz
 PPMCM 13.39041 ppm/cm
 HZCM 1330.49513 Hz/cm

-61.837



at 27 °C



55
13

HH-COSY of 2.5b

Current Data Parameters
 NAME: 2.5b
 EXPNO: 11
 PROCNO: 1

P2 - Acquisition Parameters

Date: 20011111
 Time: 13:23
 INSTRUM: spect
 PROCESSOR: 5
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: D2O
 NS: 16
 DS: 4
 SWH: 5567.831 Hz
 F2: 2.630826 Hz
 AQ: 1.1961044 sec
 RG: 128
 DM: 92.900 usec
 DE: 6.00 usec
 TE: 300.10 K
 D1: 0.0000000 sec
 D2: 2.0000000 sec
 SFO1: 500.1354797 MHz
 NUC1: 1H
 P1A: 4.00 dB
 ENO: 0.0000000 sec

F1 - Acquisition Parameters

WDW: EM
 SSF: 256
 SFO2: 500.1354797 MHz
 F2RES: 21.046606 Hz
 SW: 10.773 ppr

F2 - Processing Parameters

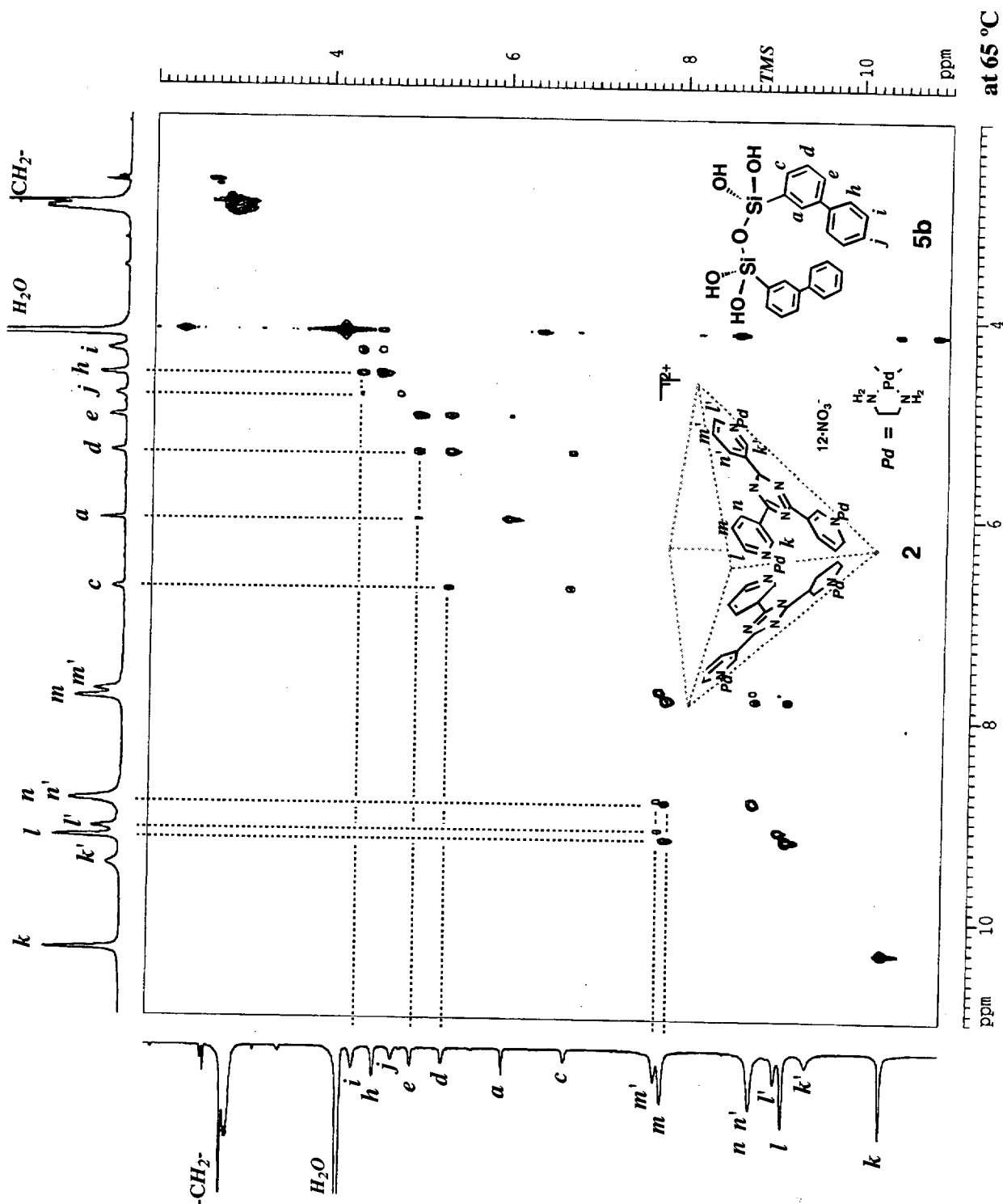
SI: 1024
 SF: 500.1302867 MHz
 GBW: 8.58E
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 1.00

F1 - Processing Parameters

SI: 1024
 SF: 500.1302867 MHz
 SSB: 0
 LB: 0.00 Hz
 GB: 0

2D NMR Plot Parameters

CH1: 25.00 cm
 CH2: 25.00 cm
 F2P2G: 11.000 ppm
 F2A0: 5567.43 Hz
 F2P1: 2.630826 Hz
 F2A1: 2.630826 Hz
 F2P2C: 11.000 ppm
 F2A2: 5567.43 Hz
 F2P1C: 2.630826 Hz
 F2P1MCH: 0.63000 ppm/cm
 F2P2MCH: 200.07816 Hz/cm
 F2P1MCC: 0.63000 ppm/cm
 F2P2MCC: 300.07816 Hz/cm



at 65 °C

CH-COSY of 2-5b

Current Data Parameters
 NAME: bowls
 EXPNO: 5
 PROCNO: 1

F2 - Acquisition Parameters

Date_: 20010717
 Time: 22:47
 INSTRUM: spect
 PROBRD: 5 mm BBO BB-1
 PULPROG: waltz16
 TD: 6556
 SOLVENT: D2O
 NS: 114
 DS: 4
 SWH: 7575.758 Hz
 FIDRES: 1.849550 Hz
 AQ: 0.2703860 sec
 RG: 16384
 EQ: 66.000 usec
 DE: 300.0
 TE: 6.40 usec
 F2: 12.80 usec
 SFO2: 0.00000000 sec
 ZG: 185.00000000 sec
 CHFT2: 0.00344828 sec
 GZ: 0.00000000 sec
 CHFT1: 0.00000000 sec
 d1: 0.00000000 sec
 d11: 0.00000000 sec
 d12: 0.00002500 sec
 d13: 2.00000000 sec
 D1: 4.00 usec
 D12: 4.00 usec
 F3: 6.40 usec
 SFO3: 500.132489 MHz
 MCH1: 132.489 MHz
 MCH2: 132.489 MHz
 MCH3: 132.489 MHz
 PL1: 2.00 dB
 PL12: 16.00 dB
 CPOPC2: waltz16
 PCPO2: 81.00 usec
 TNU: 0.0000470 sec

F1 - Acquisition Parameters

NUC1: 13C
 TD: 135
 SFO1: 500.132489 MHz
 FIDRES: 62.305298 Hz
 SW: 15.372 ppm

F2 - Processing parameters

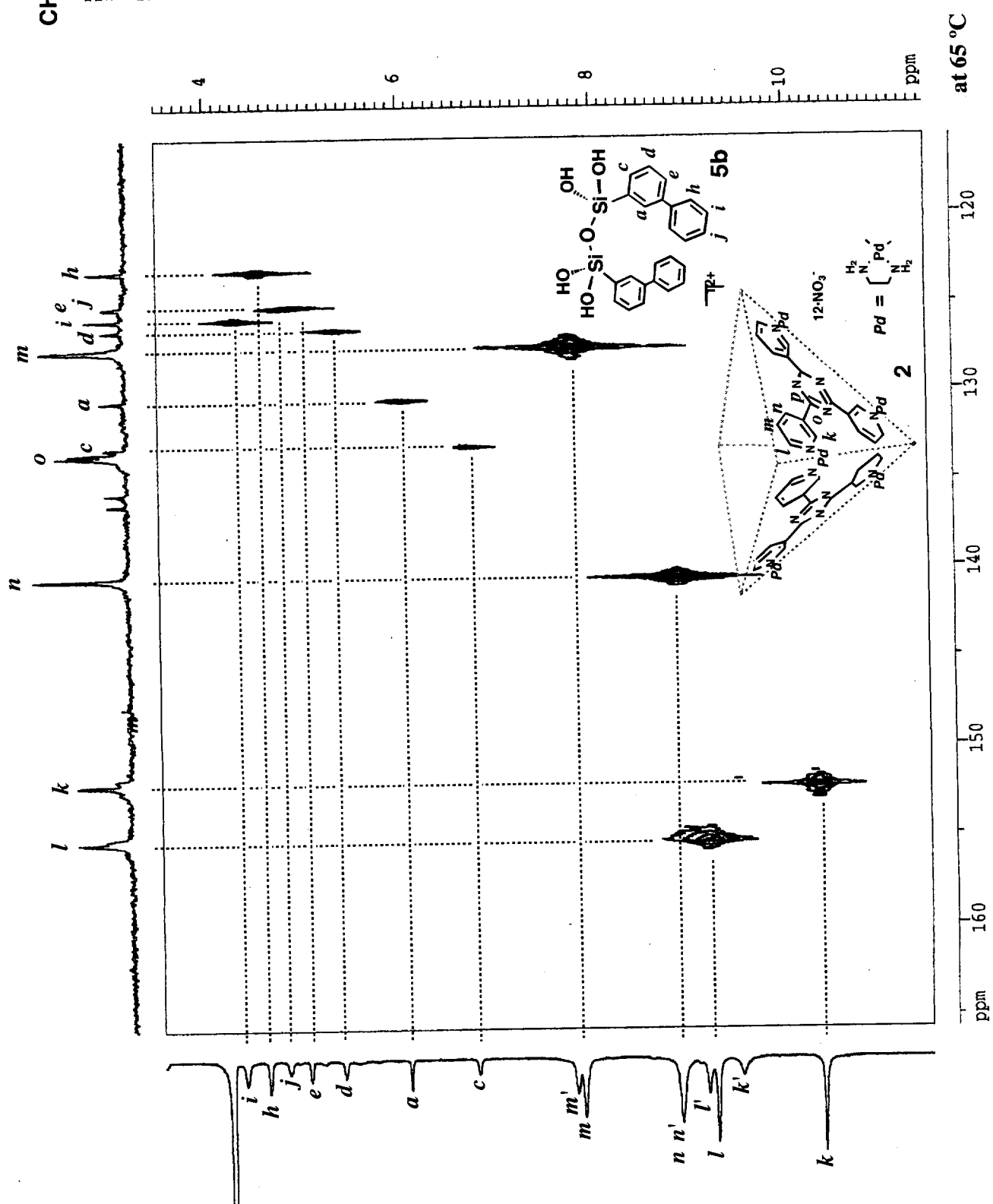
SI: 2648
 SF: 125.7576473 MHz
 SFO: 99.999999 MHz
 LB: 2.00 Hz
 GB: 0.00 Hz
 PC: 1.00

F3 - Processing parameters

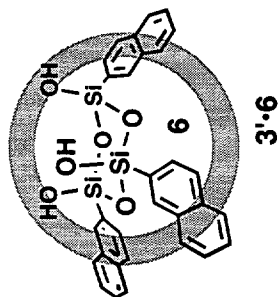
SI: 1024
 SF: 500.132489 MHz
 SFO: 99.999999 MHz
 LB: 0.30 Hz
 GB: 0.00 Hz

2D RMS proc parameters

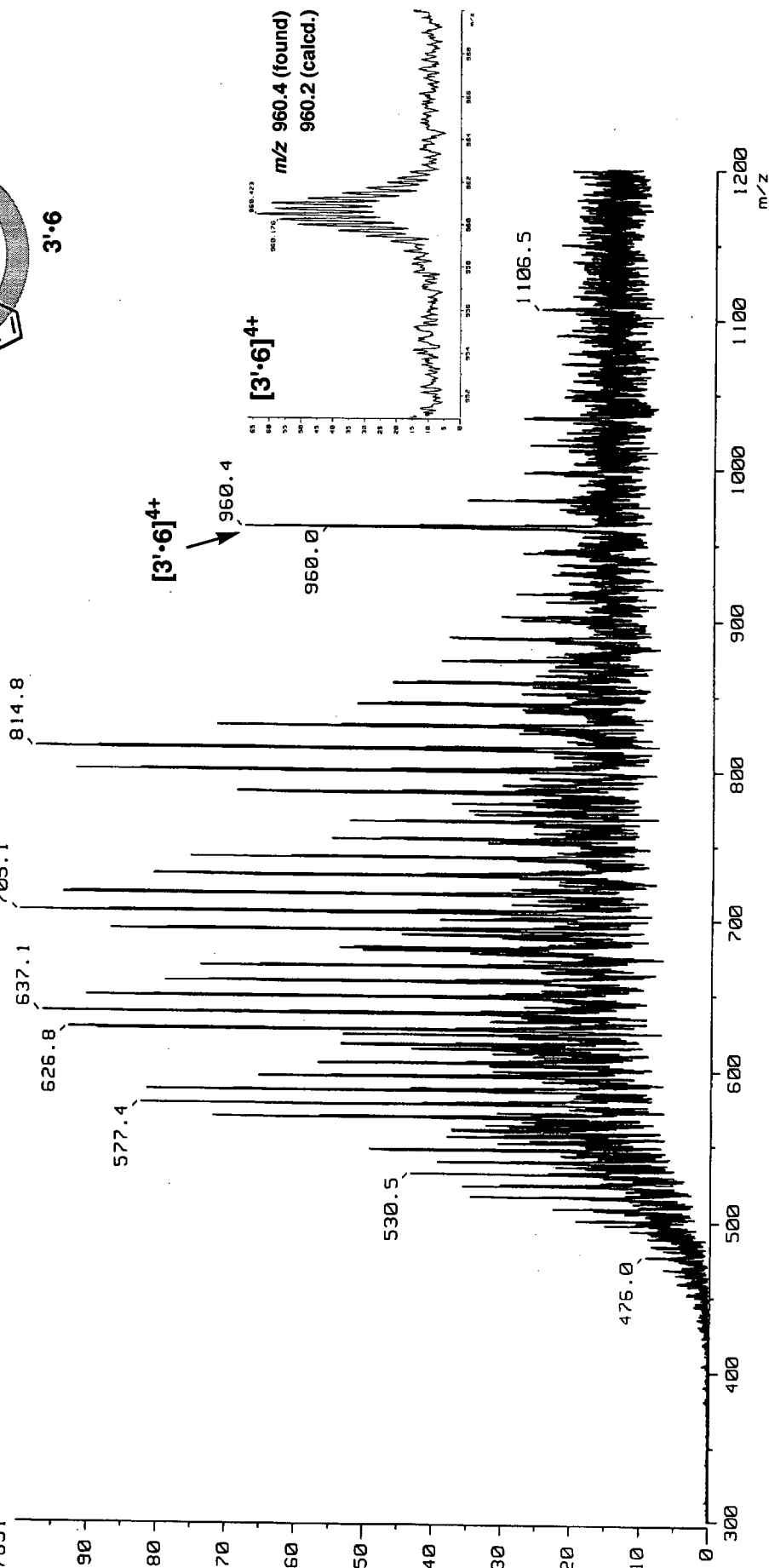
SI: 25.00 cm
 SF: 165.786 ppm
 SFO: 20848.85 Hz
 F1F1: 115.693 ppm
 F1F2: -3549.29 Hz
 F1F3: 11.467 ppm
 F1F4: 574.84 Hz
 F1F5: 1764.49 Hz
 F1F6: 3.53953 ppm/cm
 F1F7: 419.97086 Hz/cm
 F1F8: 0.52224 ppm/cm
 F1F9: 265.49056 Hz/cm



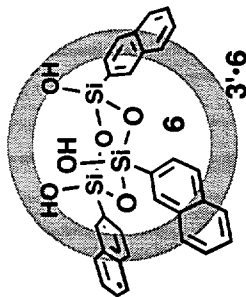
CSI MS(1) of 3'·6



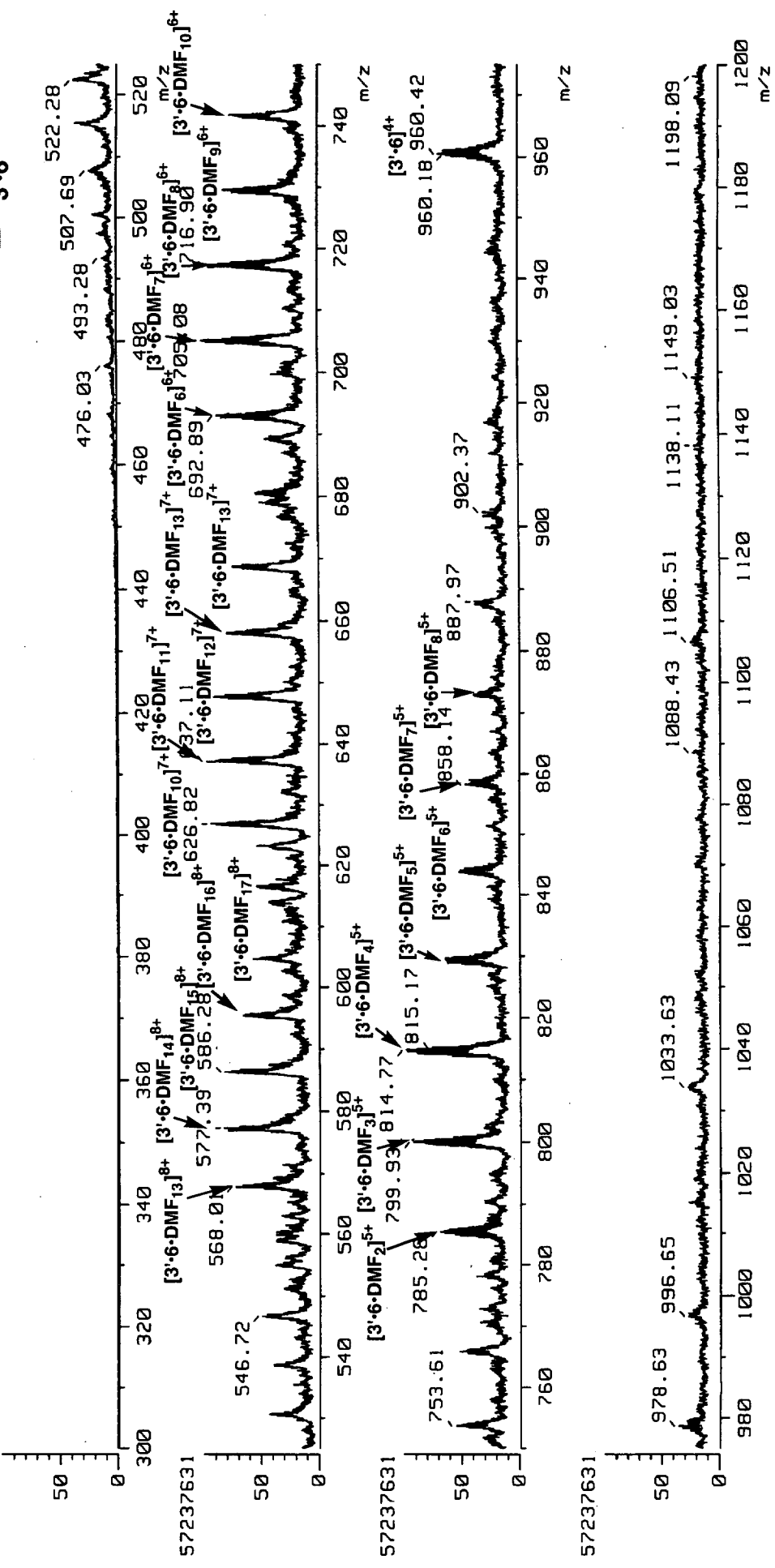
[Mass Spectrum]
 Date : 30-Jun-2000 12:12
 Sample : 000630-Yoshizawa-002
 Note : -
 Inlet : Direct Ion Mode : ESI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 19.85 min Scan# : (1,127)
 BP : m/z 705.0768 Int. : 42.98
 Output m/z range : 300.0000 to 1200.0000 Cut Level : 0.00 %
 57237631 705.1



CSI MS(2) of 3'·6



[Mass Spectrum]
 Date : 30-Jun-2000 12:12
 Sample : 000630-Yoshizawa-002
 Note : -
 Inlet : Direct Ion Mode : ESI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 19.85 min Scan# : (1,127)
 BP : m/z 705.0768 Int. : 42.98
 Output m/z range : 300.0000 to 1200.0000 Cut Level : 0.00 %
 57237631



¹H NMR of 3·6

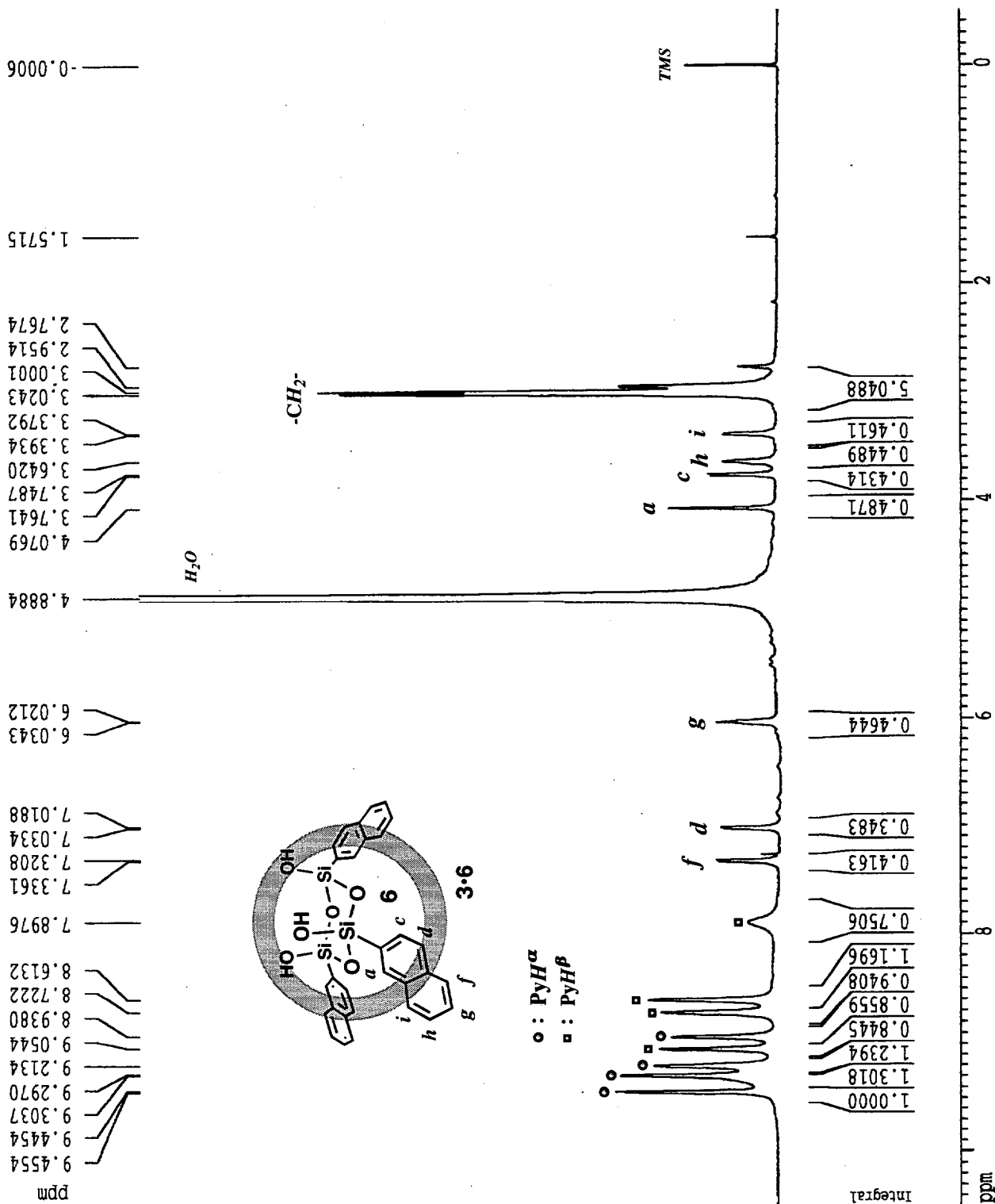
Current Data Parameters
 NAME M6LANAS11
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 2001121
 Time 20.51

INSTRUM dirx500
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 32768
 SOLVENT D2O
 NS 126
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.315264 Hz
 AQ 1.5860212 sec
 RG 128
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 8.40 usec
 SF01 500.1330885 MHz
 NUC1 ¹H
 PL1 -4.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1299069 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.500 ppm
 F1 5251.36 Hz
 F2P -0.500 ppm
 F2 -250.06 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07144 Hz/cm



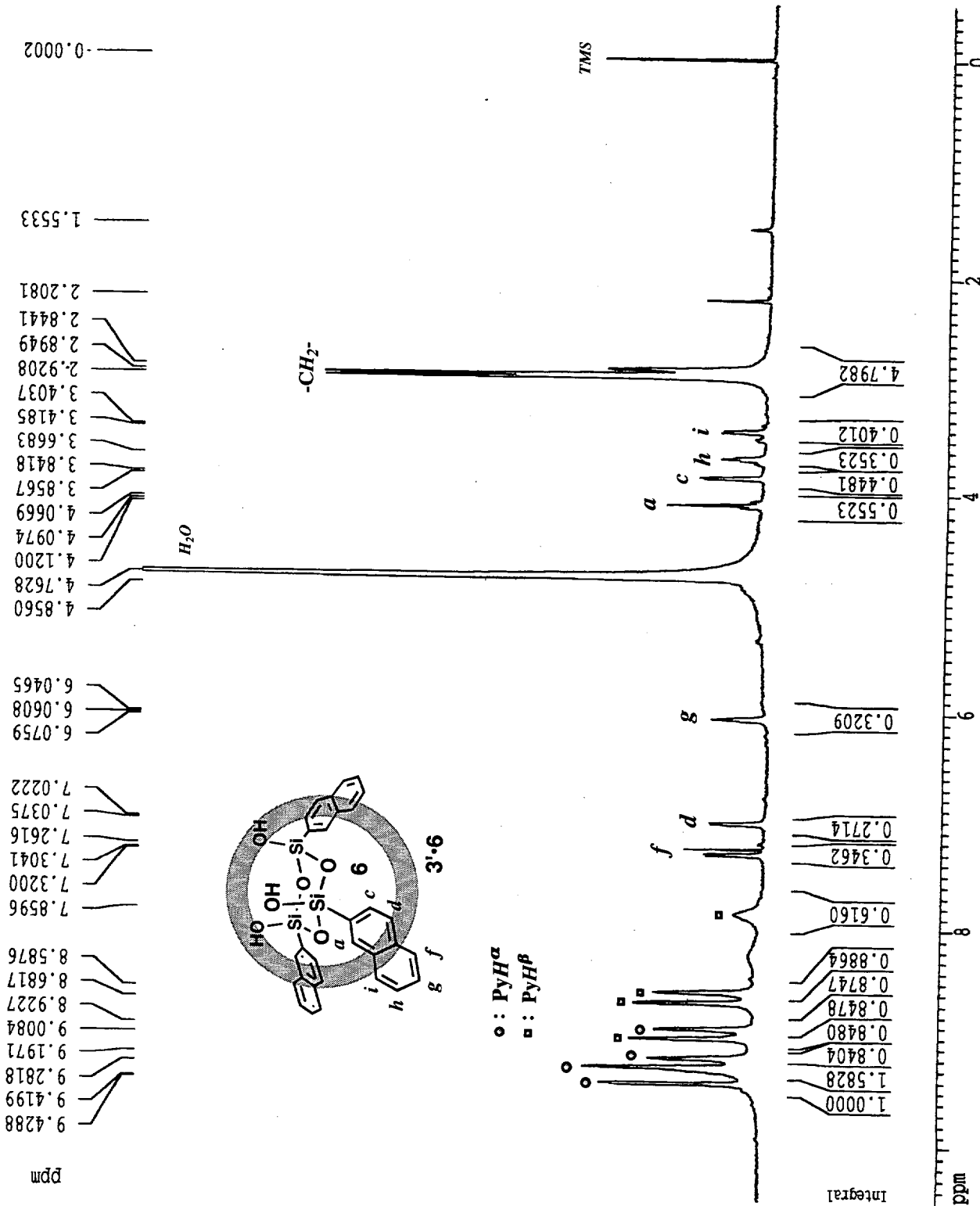
¹H NMR of 3'-6

Current Data Parameters
 NAME M6LANaSi2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010308
 Time 23.02
 INSTRUM drx500
 PROBHD 5 mm BBO BB-1
 PULPROG zg30
 TD 32768
 SOLVENT D2O
 NS 128
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.315264 Hz
 AQ 1.5860212 sec
 RG 362
 DW 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 F1 8.40 usec
 SFO1 500.1330885 MHz
 NUC1 1H
 FL1 -4.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1299705 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.500 ppm
 F1 5251.36 Hz
 F2P -0.500 ppm
 F2 -250.06 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 275.07147 Hz/cm

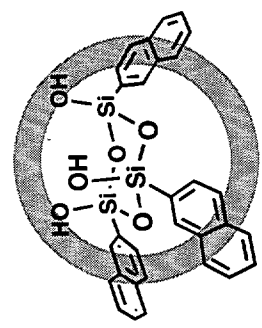


¹³C NMR(1) of 3-6

F2 - Acquisition Parameters
 Date_ 20001202
 Time 13.53
 INSTRUM drx500
 PROBHD 10 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 10784
 DS 2
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 3649.1
 DW 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 d11 0.03000000 sec
 d12 0.00002000 sec
 PL13 18.00 dB
 D1 2.00000000 sec
 CPDPRG2 waltz16
 PCPD2 85.00 usec
 SFO2 500.1320005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 10.00 dB
 P1 10.30 usec
 SFO1 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB
 F2 - Processing parameters
 SI 32768
 SF 125.7577789 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 FC 1.40
 ID NMR plot parameters
 CX 20.00 cm
 FIP 180.000 ppm
 F1 22636.40 Hz
 F2P 40.000 ppm
 F2 5030.31 Hz
 PPMCM 7.00000 ppm/cm
 HZCM 880.30444 Hz/cm

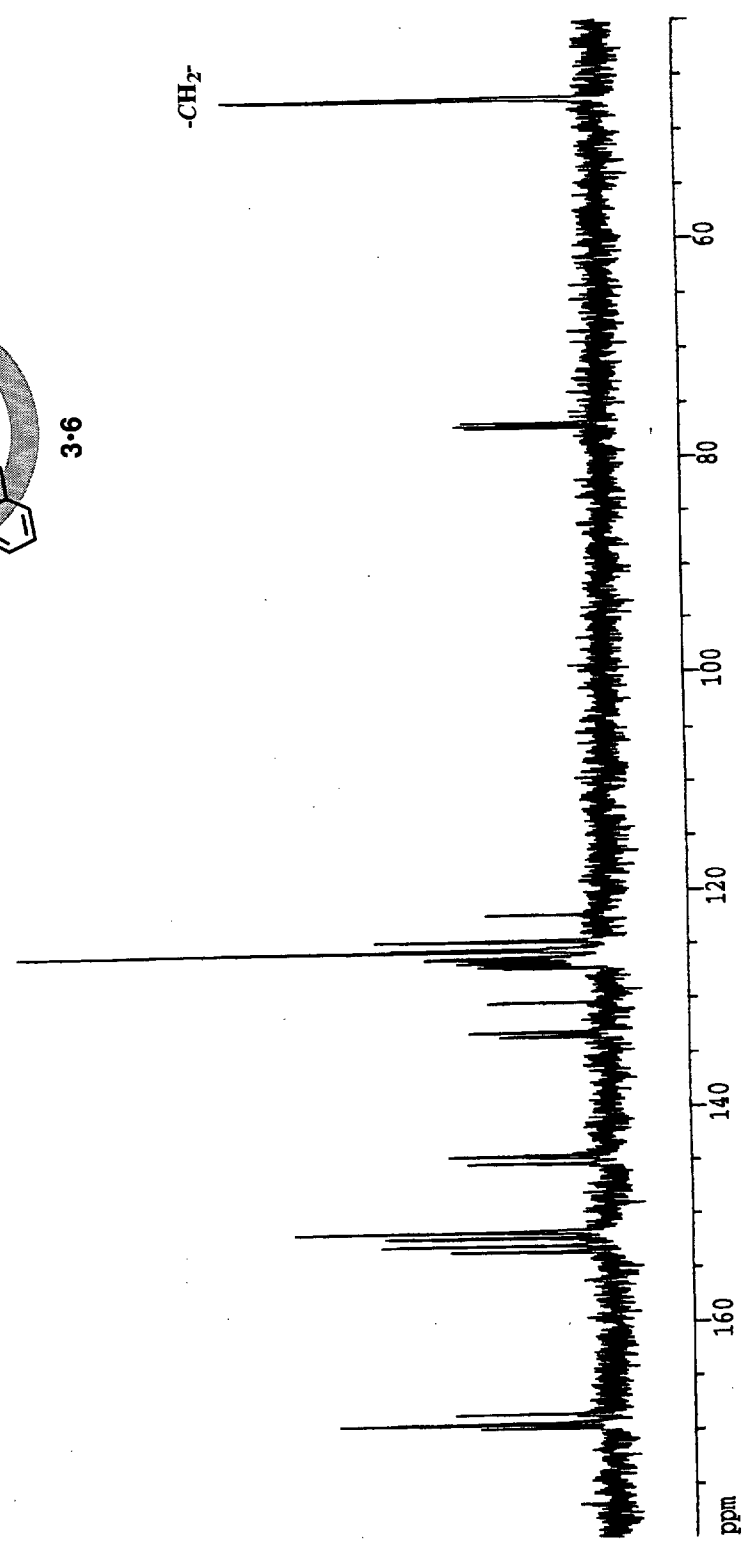
46.997
46.888

77.253
77.001
76.746



169.724
169.326
168.469
153.511
152.948
152.245
151.663
145.240
144.589
133.404
133.021
130.357
127.112
126.941
126.846
126.614
126.348
126.174
125.460
124.600
122.219

-CH₂-



¹³C NMR(2) of 3·6

F2 - Acquisition Parameters

Date_ 20001202
 Time 13.53
 INSTRUM dirx500
 PROBHD 10 mm BBO BB-1
 PULPROG zgpg30
 TD 65536
 SOLVENT D2O
 NS 10784
 DS 2
 SMH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 3649.1
 DM 12.600 usec
 DE 6.00 usec
 TE 300.0 K
 d11 0.03000000 sec
 d12 0.00020000 sec
 PL13 18.00 dB
 DI 2.00000000 sec
 CPDPRG2 waltz16
 PCPD2 85.00 usec
 SF02 500.1320005 MHz
 NUC2 ¹H
 PL2 -6.00 dB
 PL12 10.00 dB
 P1 10.30 usec
 SF01 125.7736214 MHz
 NUC1 ¹³C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577789 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

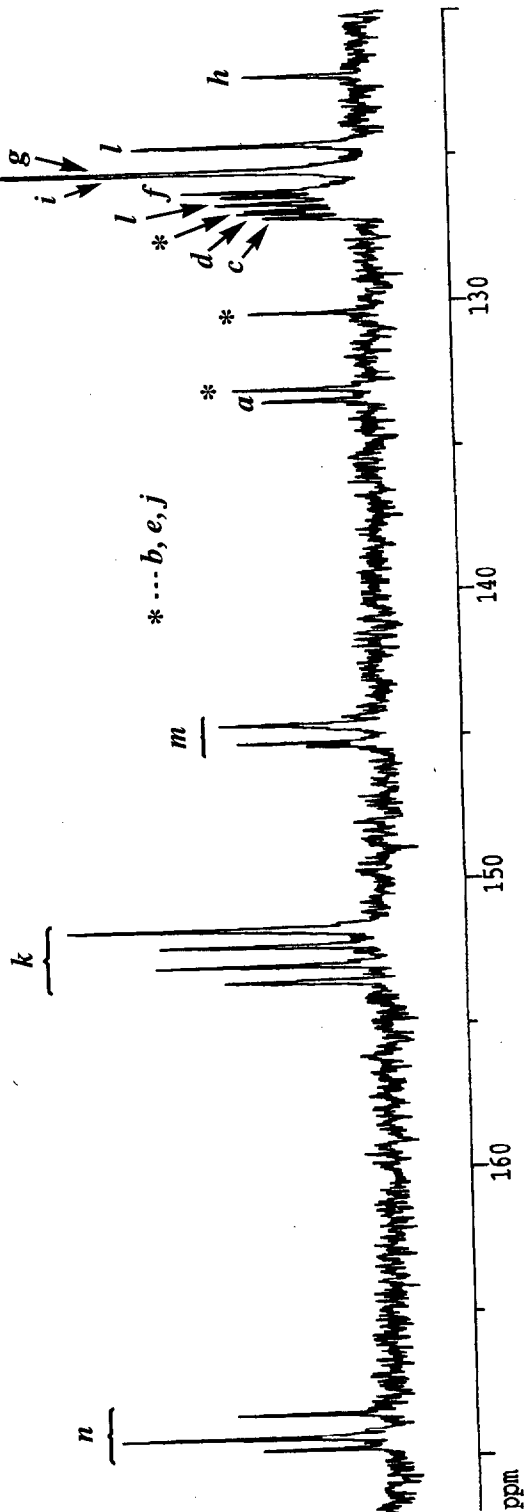
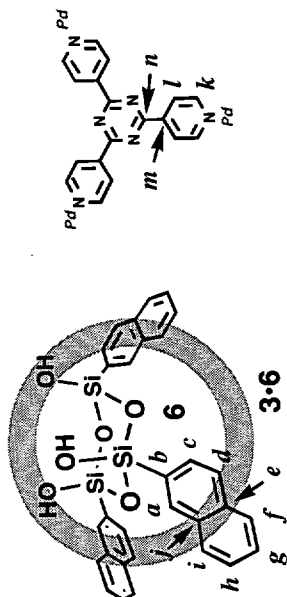
1D NMR plot parameters
 CX 20.00 cm
 FIP 172.000 ppm
 F1 21630.34 Hz
 F2P 120.000 ppm
 F2 15090.93 Hz
 PPMCM 2.60000 ppm/cm
 HZCM 326.97025 Hz/cm

133.404
 133.021
 130.357
 127.112
 126.941
 126.846
 126.614
 126.348
 126.174
 125.460
 124.600
 122.219

145.240
 144.589

153.511
 152.948
 152.245
 151.663

169.724
 169.326
 168.469



HH-COSY of 3·6

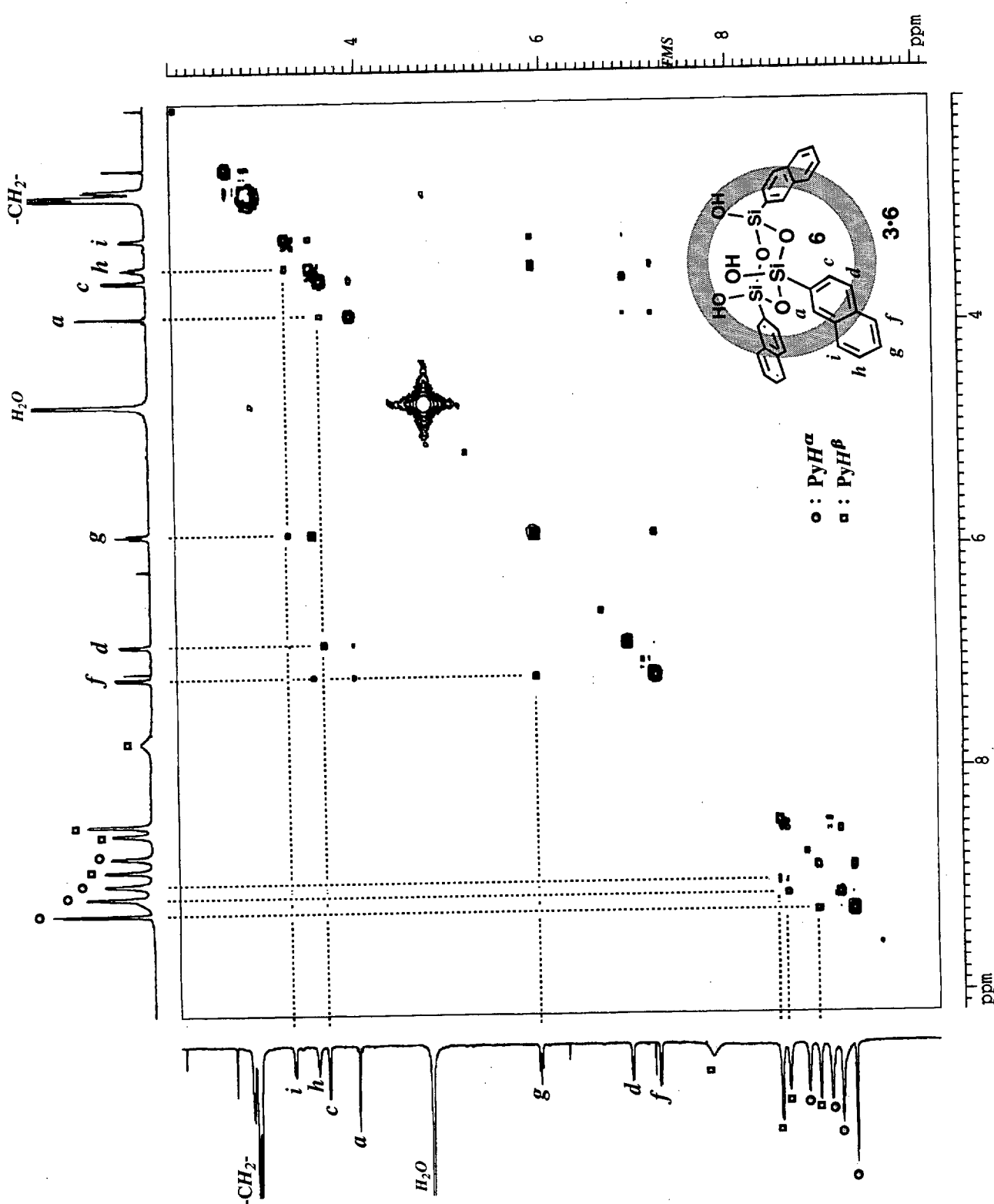
F2 - Acquisition Parameters
 Date_ 9/11/95
 Time 14:06
 INSTRON 415600
 PROBD 5 mm BBH-5
 PULPROG zgpg30
 COUPLER 2048
 SOLVENT D2O
 NS 64
 DS 8
 SWH 5482.456 Hz
 FIDRES 2.676980 Hz
 AQ 0.1868276 sec
 RG 5792.6
 DW 91.200 usec
 DE 6.00 usec
 TE 300.0 K
 d0 0.10000000 sec
 d13 0.00000000 sec
 D1 1.48689198 sec
 F1 7.10 usec
 SFO1 500.1323514 MHz
 NUC1 1H
 P1 1.00 dB
 PL1 1500.00 usec
 F16 0.00000000 sec
 D16 7.10 usec
 P0 0.00000000 sec
 INO 0.00018240 sec

F1 - Acquisition Parameters
 ND0 1
 TD 79
 SFO1 500.1324 MHz
 FIDRES 69.398178 Hz
 SW 10.962 ppa

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC 0
 SF 500.1300000 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX1 15.00 cm
 PZP10 10.183 ppm
 PZ10 5092.65 Hz
 PZPH 2.000 ppm
 PZHI 1000.26 Hz
 F1P10 10.183 ppm
 F1LO 5092.65 Hz
 F1PH 2.000 ppm
 F1HI 1000.26 Hz
 F2PMCM 0.56551 ppm/cm
 F2ZCM 272.82590 Hz/cm
 F1PMCM 0.56551 ppm/cm
 F1ZCM 272.82590 Hz/cm



CH-COSY of 3•6

F1 - Acquisition Parameters

Date_ 20001202
 Time 23:16
 INSTRUM spect
 PULPROG zgpg30
 TO 4096
 SOLVENT D2O
 NS 440
 DS 4
 SWH 10000.644 Hz
 FIDRES 3.161095 Hz
 AQ 0.2032116 sec
 SFO1 500.1324088 MHz
 DE 49.600 usec
 TE 298.0 K
 P1 6.00 usec
 PL 0.00 dB
 ZG 20.40 usec
 ZG2 20.40 usec
 SFO2 145.0000000 MHz
 CNST1 0.00344828 sec
 CNST11 3.0000000
 d3 0.00229885 sec
 d11 0.0002000 sec
 d12 0.0002000 sec
 d1 2.0000000 sec
 EL2 -6.00 dB
 P3 19.40 usec
 SFO2 500.1324088 MHz
 NUC2 1H
 SFO1 125.7753818 MHz
 P1 13.00 usec
 PL1 0.00 dB
 EL1 10.00 dB
 CDEPR2 walz16
 PCPD2 85.00 usec
 INO 0.0008320 sec

F1 - Acquisition Parameters

NUO 2
 TD 124
 SFO1 500.1324 MHz
 FIDRES 48.468462 Hz
 SW 12.016 ppm

F2 - Processing parameters

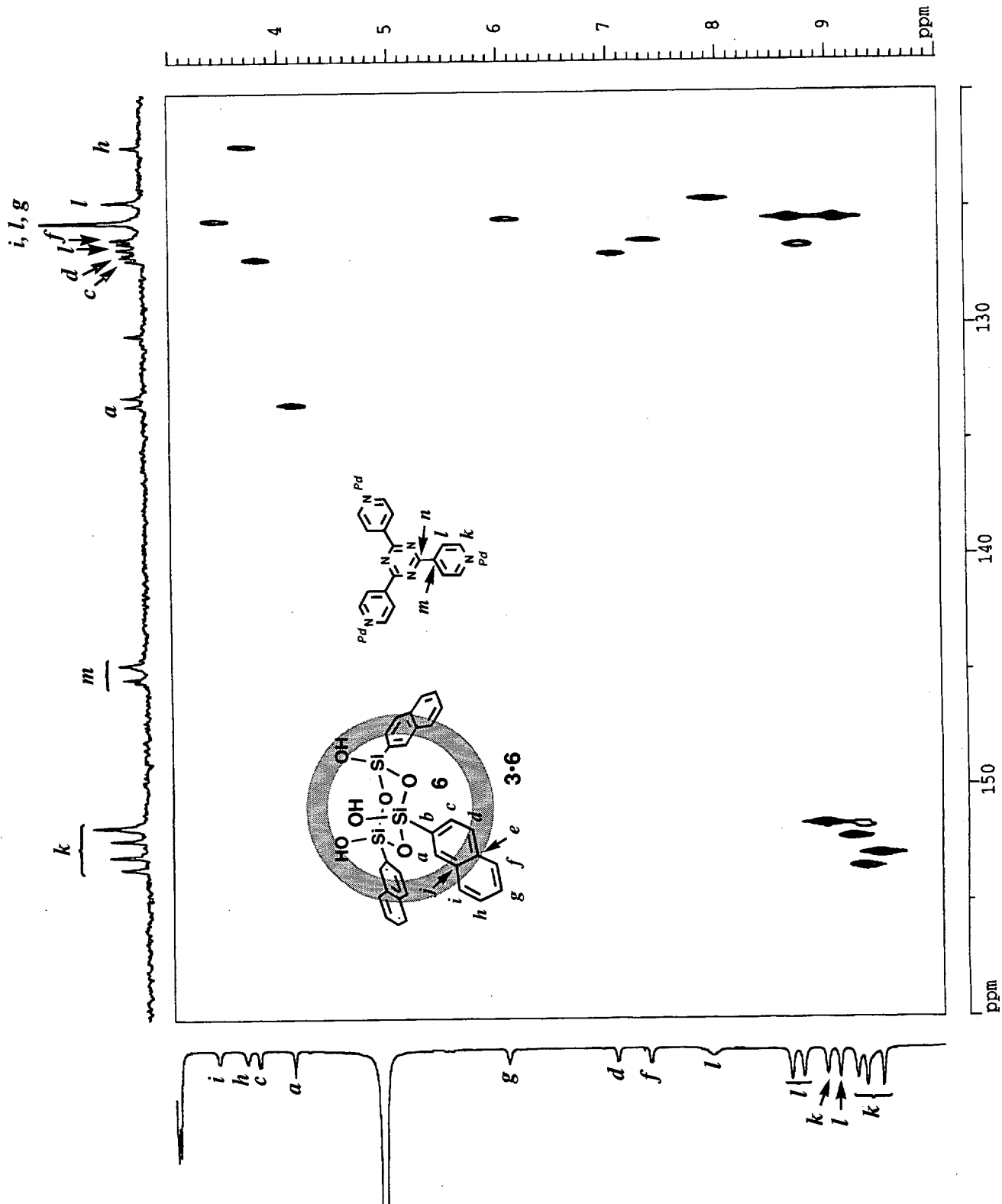
SI 2048
 SF 125.757773 MHz
 NDM 2
 SFO1 125.757773 MHz
 LB 0.00 Hz
 GB 1.40
 PC 1.40

F1 - Processing parameters

SI 1024
 SF 500.129483 MHz
 NDM 2
 SFO1 500.129483 MHz
 LB 0.00 Hz
 GB 0

2D NMR plot parameters

SI 15.00 cm
 CX2 15.00 cm
 CX1 15.00 cm
 F2LO 20127.80 Hz
 F2H1 119.933 ppm
 F1F0 15082.56 Hz
 F1LO 10.013 ppm
 F1H1 5007.85 Hz
 F1F2 1482.16 Hz
 F2BPMCH 2.67458 ppm/cm
 F1BPMCH 336.38773 Hz/cm
 F2PMCH 0.46660 ppm/cm
 F1PMCH 234.35931 Hz/cm



²⁹Si NMR of 3·6

F2 - Acquisition Parameters

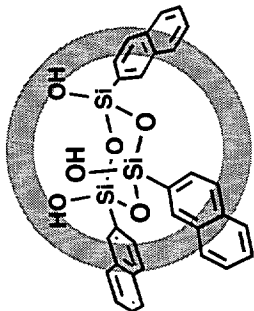
Date_ 20001120
 Time 22.33
 INSTRUM dirx500
 PROBHD 5 mm BBO BB-1
 PULPROG dept45
 TD 65536
 SOLVENT H2O
 NS 14699
 DS 4
 SWH 39682.539 Hz
 FIDRES 0.60507 Hz
 AQ 0.8258036 sec
 RG 7298.2
 DW 12.600 usec
 DE 6.00 usec
 TE 300.0 K 298
 P1 7.50 usec
 P2 15.00 usec
 P3 7.60 usec
 P4 15.20 usec
 CNST2 20.0000000
 d2 0.02500000 sec
 d12 0.00002000 sec
 DELTA 0.0000955 sec
 D1 2.00000000 sec
 PL2 -4.00 dB
 SFO2 500.1320005 MHz
 NUC2 ¹H
 SFO1 99.3617370 MHz
 NUC1 ²⁹Si
 PL1 2.00 dB
 PL12 16.00 dB
 CPDPRG2 waltz16
 PCPD2 81.00 usec

F2 - Processing parameters

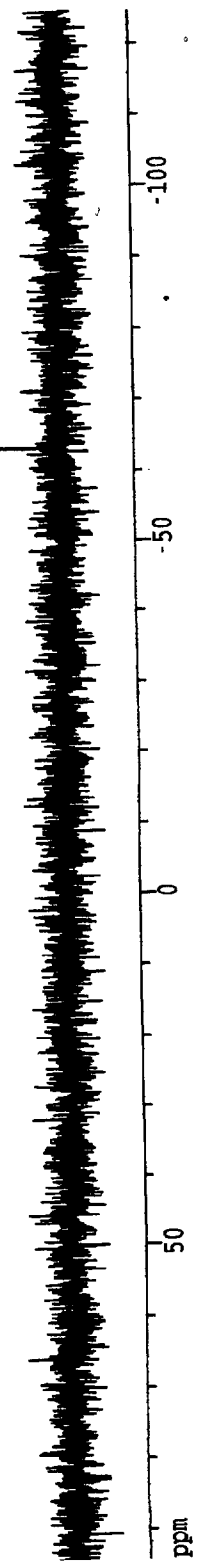
SI 32768
 SF 99.3617534 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 94.459 ppm
 F1 9385.60 Hz
 F2P -124.664 ppm
 F2 -12386.87 Hz
 PPMCM 10.95616 ppm/cm
 HZCM 1088.62317 Hz/cm

-63.054



3·6



ppm