

## Additional File 5

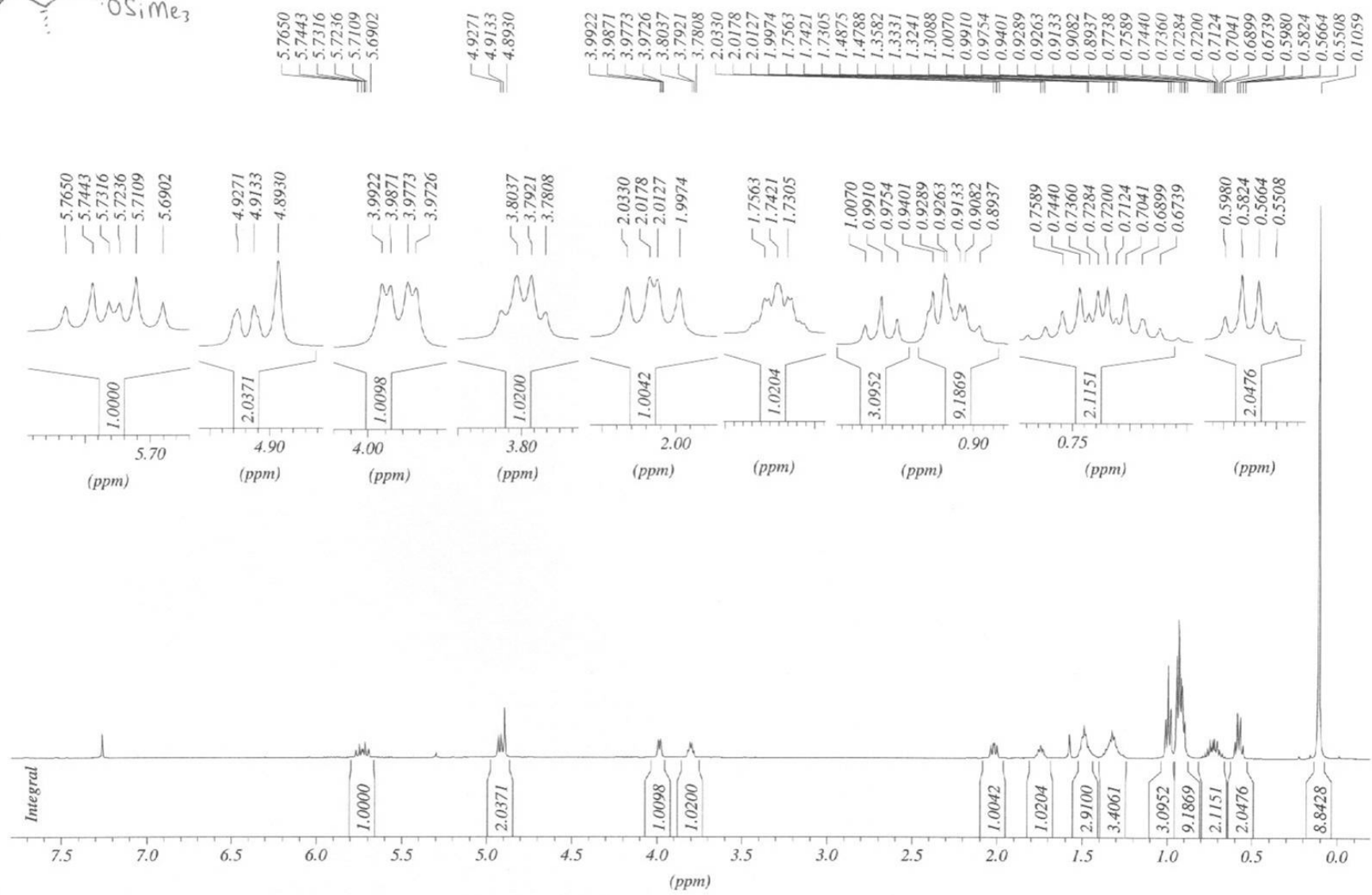
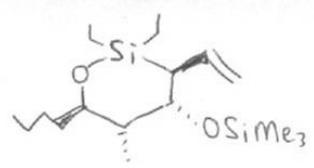
**Tether-directed synthesis of highly substituted oxasilacycles *via* an intramolecular allylation employing allylsilanes**

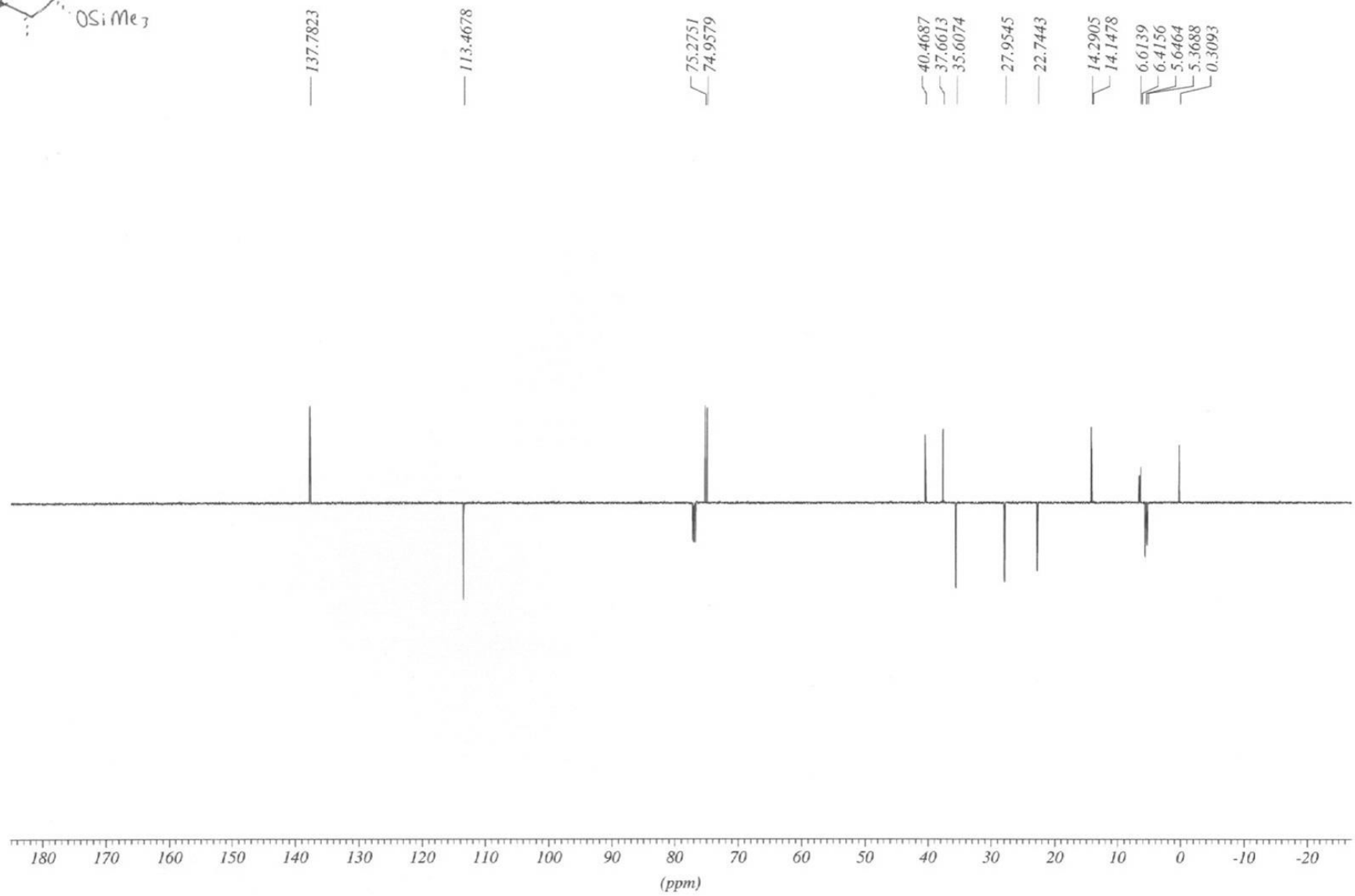
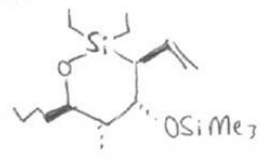
Peter J. Jervis and Liam R. Cox\*

email: l.r.cox@bham.ac.uk

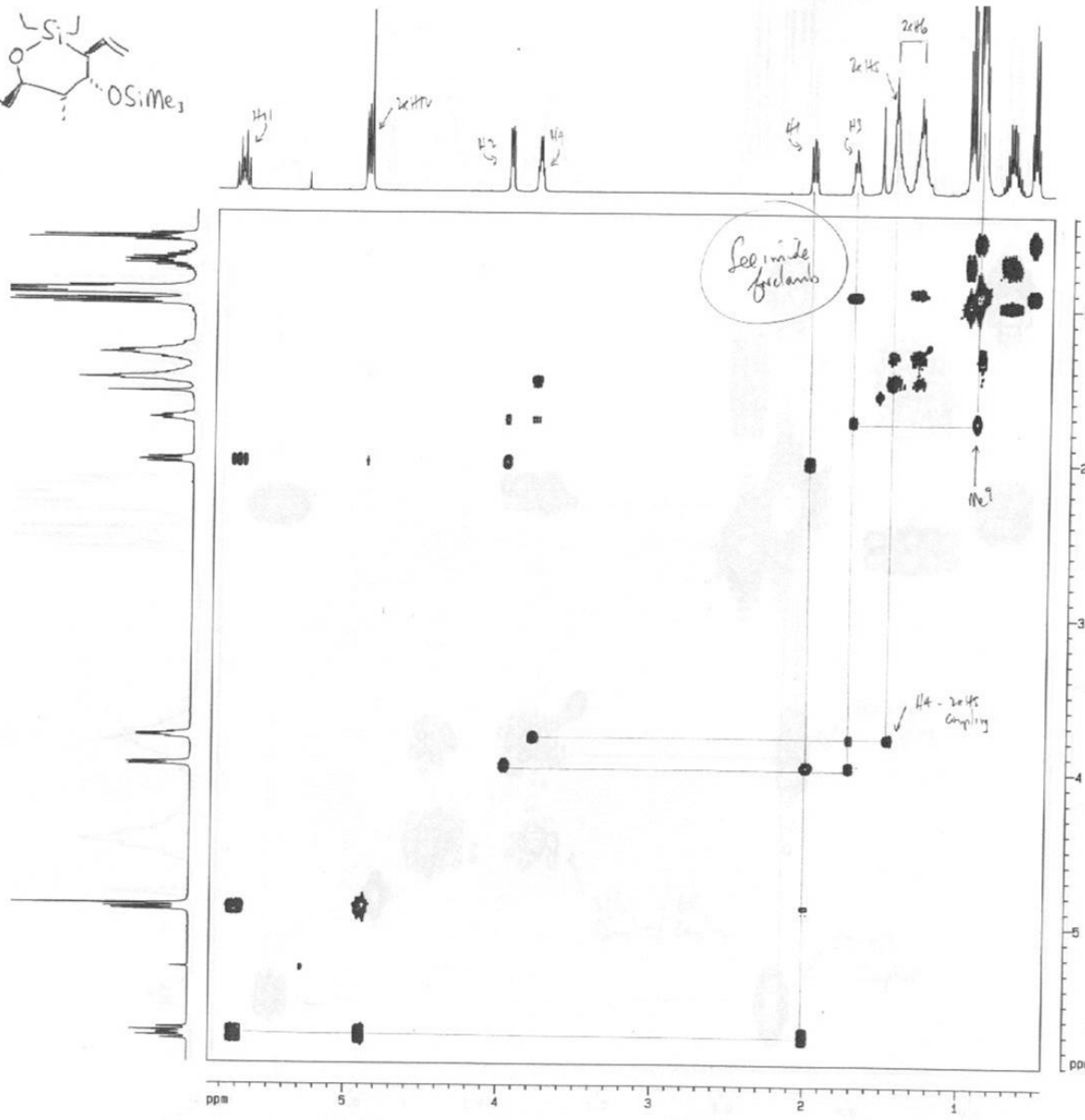
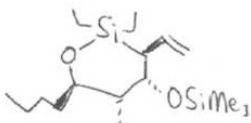
**$^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  Spectra for the following compounds:**

**14a, 15a**





Peter Jervis Sample B, 13/12/05 in CDC13 at +27C, set temp  
drx500, Gradient COSY90



Current Data Parameters

NAME dc13p1d  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20051212  
 Time 22:50  
 INSTRUM drx500  
 PROBNM 5 == TBI H/C  
 PULPROG cosy90  
 TD 2048  
 SOLVENT CDC13  
 NS 8  
 DS 16  
 SMH 4310.345 Hz  
 FIDRES 2.104661 Hz  
 AQ 0.2376180 sec  
 RG 57  
 DW 116.000 usec  
 DE 5.50 usec  
 TE 300.0 K  
 d0 0.00000300 sec  
 d1 2.00000000 sec  
 d13 0.00000300 sec  
 d16 0.00010000 sec  
 INO 0.00023200 sec

----- CHANNEL f1 -----

NUC1 <sup>13</sup>C  
 P0 10.70 usec  
 P1 10.70 usec  
 PL1 1.00 dB  
 SFO1 500.1318867 MHz

----- GRADIENT CHANNEL -----

GRANM1 SINE.100  
 GRANM2 SINE.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 SPZ1 10.00 %  
 SPZ2 10.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters

ND0 1  
 TD 512  
 SFO1 500.1319 MHz  
 FIDRES 8.418642 Hz  
 SM 8.518 ppm

F2 - Processing parameters

SI 2048  
 SF 500.1300233 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

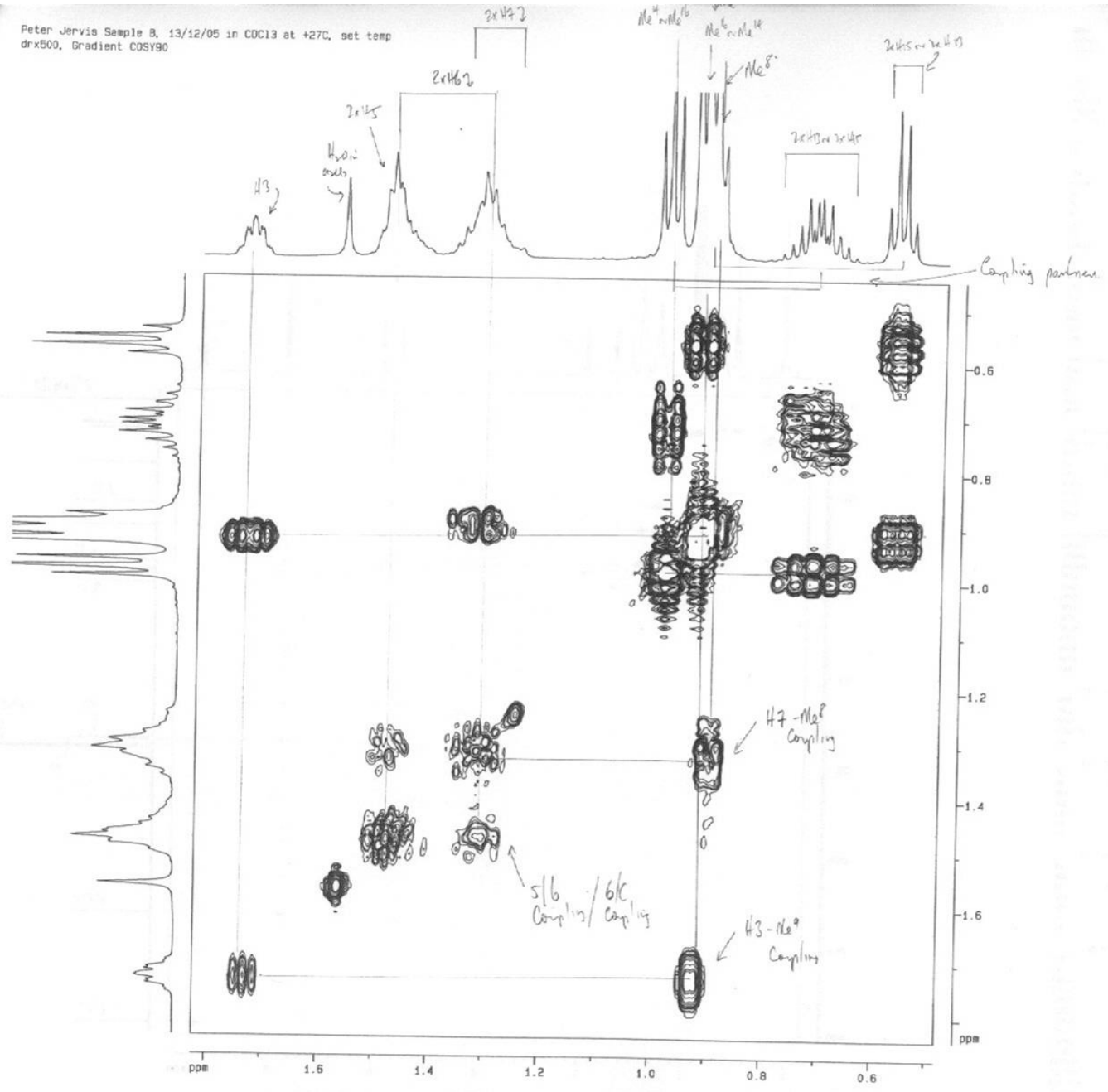
F1 - Processing parameters

SI 1024  
 MC2 GF  
 SF 500.1300233 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

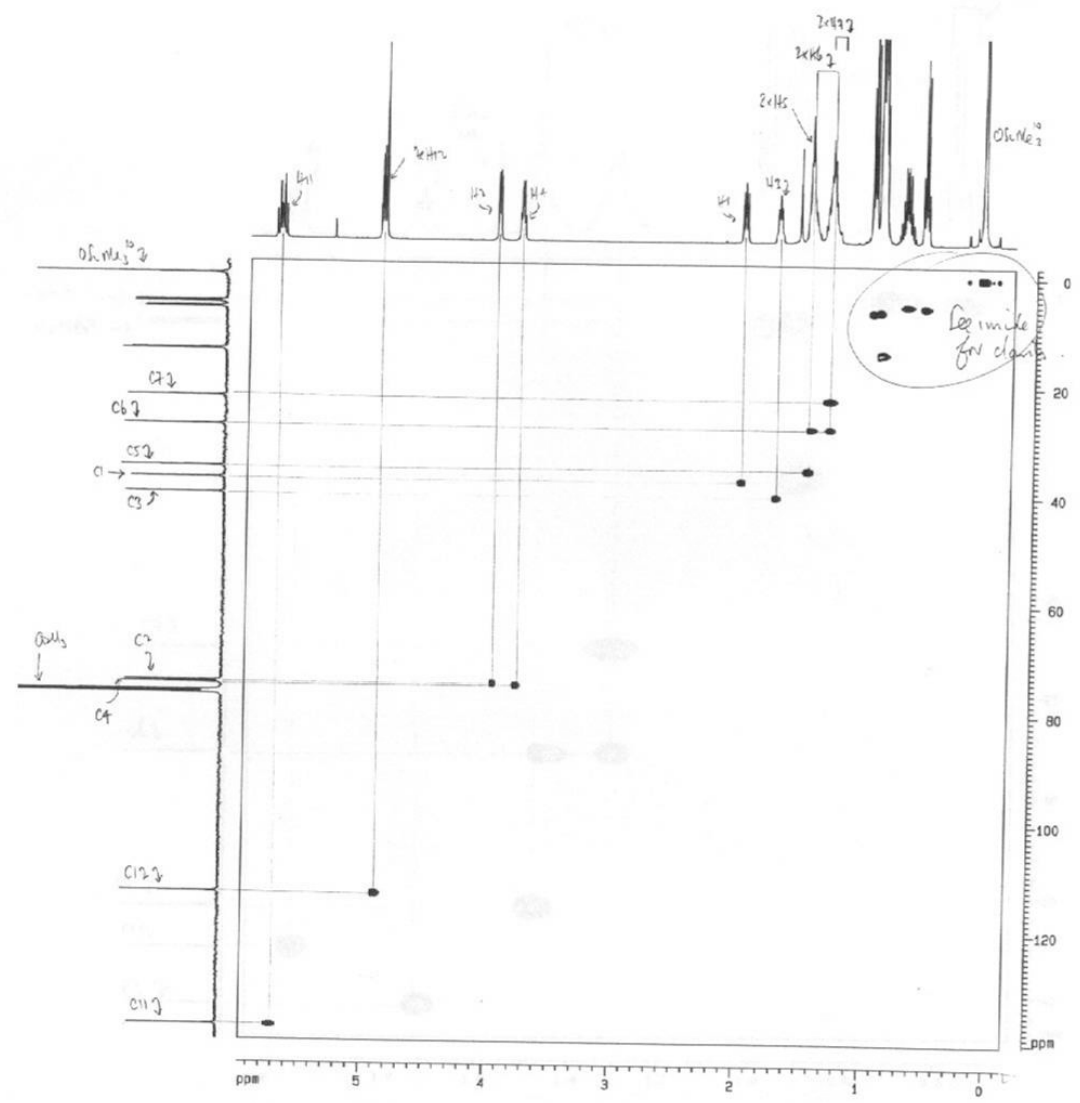
2D NMR plot parameters

CX2 20.00 cm  
 CX1 20.00 cm  
 F2PLO 5.881 ppm  
 F2L0 2941.05 Hz  
 F2PHI 0.444 ppm  
 F2H1 221.82 Hz  
 F1PLO 5.906 ppm  
 F1L0 2953.67 Hz  
 F1PHI 0.393 ppm  
 F1H1 196.57 Hz  
 F2PPMCM 0.27185 ppm/cm  
 F2HZCM 135.96109 Hz/cm  
 F1PPMCM 0.27054 ppm/cm  
 F1HZCM 137.85927 Hz/cm

Peter Jervis Sample B, 13/12/05 in CDCl3 at +27C, set temp  
drx500, Gradient COSY90



Peter Jervis Sample B, 13/12/05 in CDCl3 at +27C, set temp  
 drx500, Gradient HSQC



Current Data Parameters  
 NAME d:130110  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20051212  
 Time 17.34  
 INETRM drx500  
 PROBHD 5 mm TBI H/C  
 PULPROG invigpro  
 TO 2046  
 SOLVENT CDCl3  
 NS 8  
 DS 16  
 SWH 4312.345 Hz  
 FIDRES 2.154851 Hz  
 AQ 0.2376160 sec  
 RG 32768  
 DM 116.000 usec  
 DE 5.50 usec  
 TE 300.2 K  
 CNGT2 145.0000000  
 D0 0.0000300 sec  
 D1 2.0000000 sec  
 D4 0.0012414 sec  
 d11 0.0300000 sec  
 d13 0.0000300 sec  
 D16 0.0001000 sec  
 d20 0.0011000 sec  
 d21 0.0008714 sec  
 INO 0.0001140 sec

CHANNEL F1  
 NUC1 1H  
 P1 19.70 usec  
 p2 21.40 usec  
 PL1 1.00 dB  
 SFO1 500.1318667 MHz

CHANNEL F2  
 CPDPRG2 gpr2  
 NUC2 13C  
 P3 12.00 usec  
 p4 24.00 usec  
 PCPD2 76.00 usec  
 PL2 -1.00 dB  
 PL12 15.00 dB  
 SFO2 125.7667893 MHz

GRADIENT CHANNEL  
 GRAM1 SINE.100  
 GRAM2 SINE.100  
 GRAM3 SINE.100  
 GRX1 0.00 %  
 GRX2 0.00 %  
 GRX3 0.00 %  
 GRX4 0.00 %  
 GRX5 0.00 %  
 GRX6 0.00 %  
 GRX7 0.00 %  
 GRX8 0.00 %  
 GRX9 0.00 %  
 GRX10 0.00 %  
 GRX11 0.00 %  
 GRX12 0.00 %  
 GRX13 0.00 %  
 GRX14 0.00 %  
 GRX15 0.00 %  
 GRX16 0.00 %  
 GRX17 0.00 %  
 GRX18 0.00 %  
 GRX19 0.00 %  
 GRX20 0.00 %  
 GRX21 80.00 %  
 GRX22 30.00 %  
 GRX23 20.10 %  
 P16 1000.00 usec

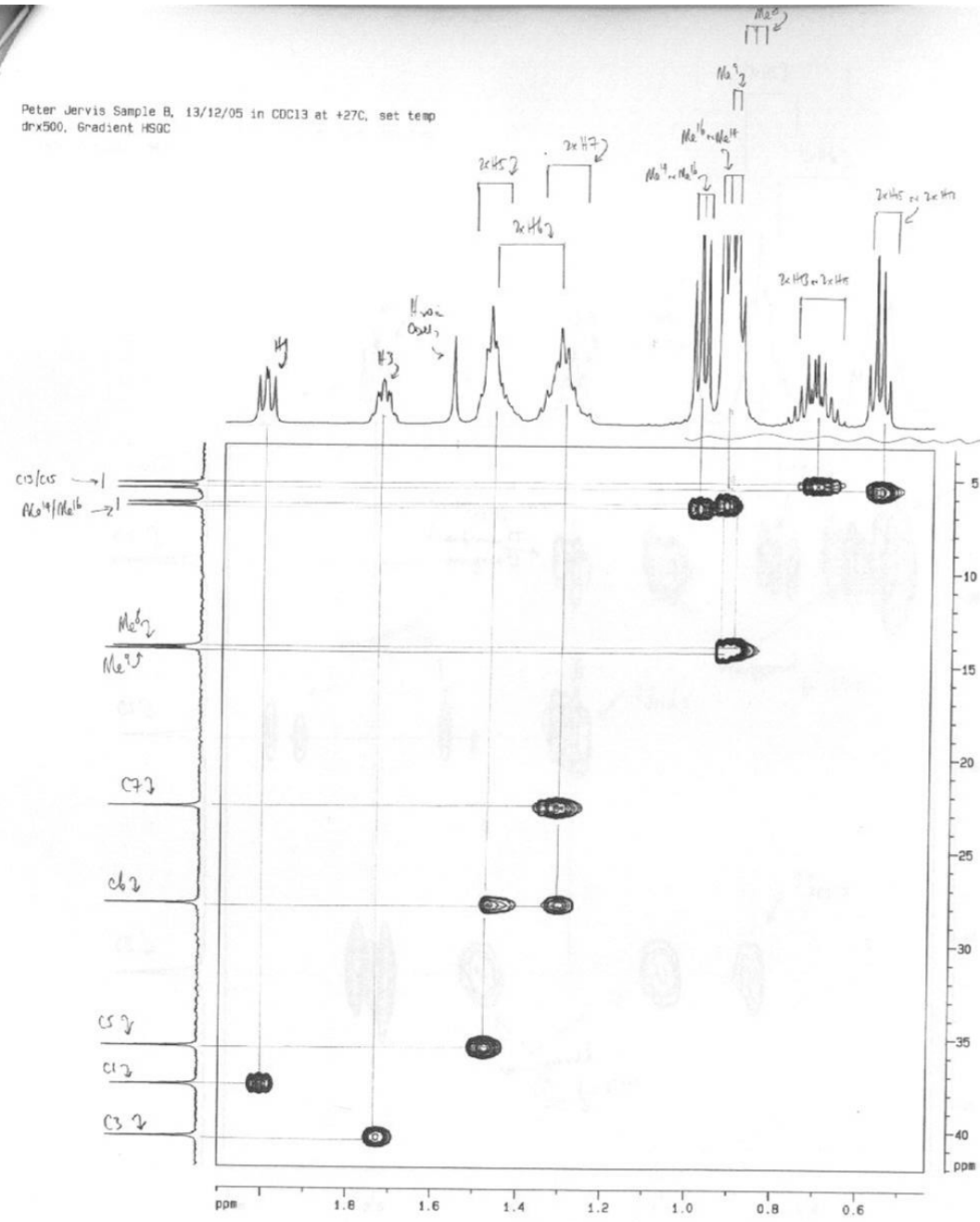
F1 - Acquisition parameters  
 MD 4  
 TO 512  
 SFO1 125.7668 MHz  
 FIDRES 42.631688 Hz  
 SM 174.389 ppm

F2 - Processing parameters  
 SI 2048  
 SF 500.1300233 MHz  
 WMW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 NUC1 1H  
 SF 125.7577910 MHz  
 WMW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

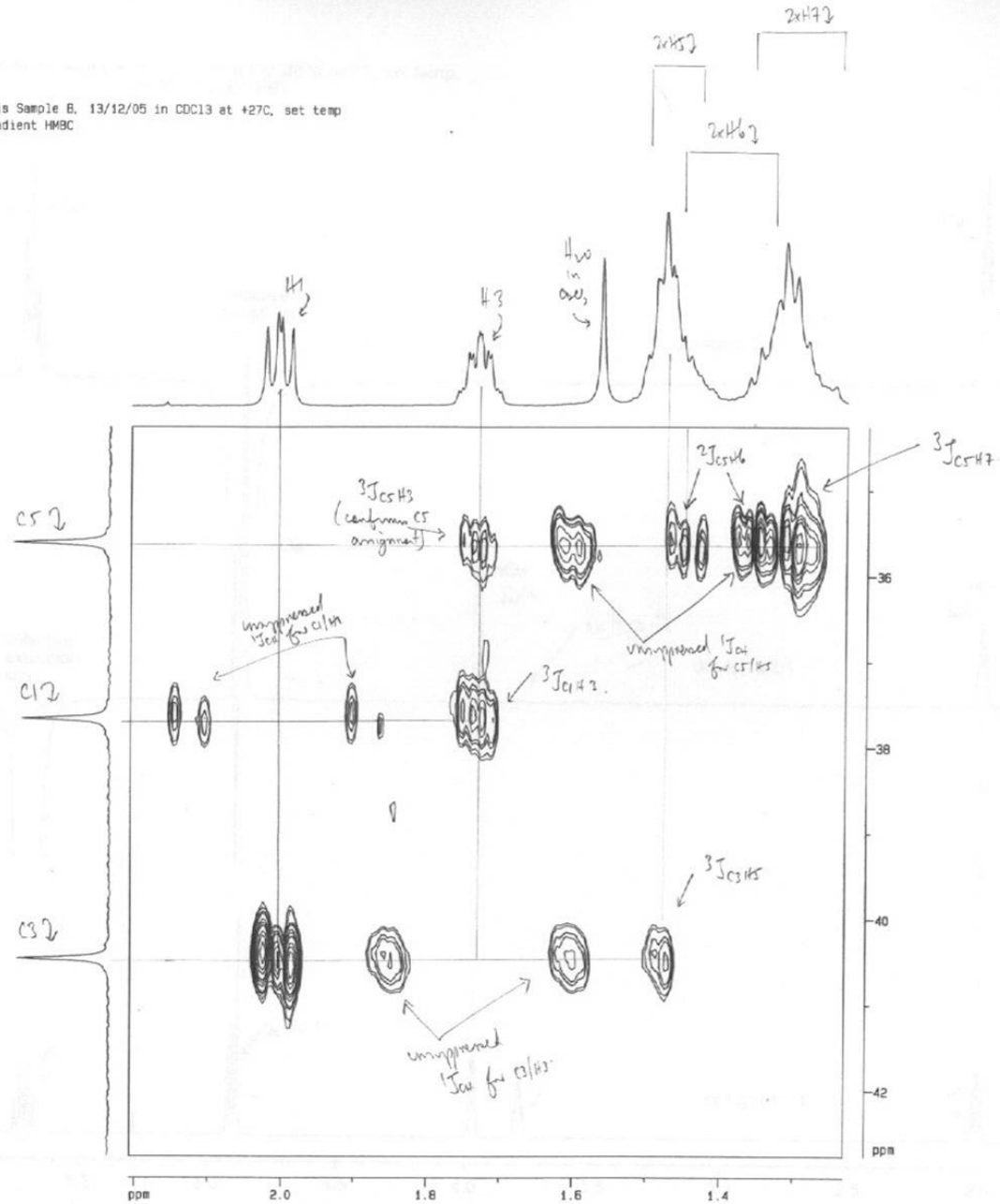
2D NMR plot parameters  
 CX2 17.00 cm  
 CX1 17.00 cm  
 F2PLD 5.985 ppm  
 F2LO 2083.14 Hz  
 F2SHE -0.154 ppm  
 F2KH -77.04 Hz  
 F2PL 140.522 ppm  
 F2LO 1767.75 Hz  
 F2SHE -0.014 ppm  
 F2KH -253.31 Hz  
 F2PANH 0.35693 ppm/cm  
 F2NDCH 180.01030 Hz/cm  
 F1PANH 8.39448 ppm/cm  
 F1NDCH 1054.41593 Hz/cm

Peter Jervis Sample B, 13/12/05 in CDCl3 at +27C, set temp  
drx500, Gradient HSQC



See Coug. 90 for coupling patterns  
(though not absolutely assigned)

Peter Jarvis Sample B, 13/12/05 in CDCl3 at +27C, set temp  
 drx500, Gradient HMBC



Current Data Parameters

NAME	dc13p1d
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20051212
Time	20.09
INSTRUM	gpc450
PROBHD	5 mm 1H/13C
PULPROG	zgpg30
TD	2048
SOLVENT	CDCl3
NS	16
DS	16
SWH	4310.345 Hz
FIDRES	2.104681 Hz
AQ	0.2378190 sec
RG	32768
DM	118.000 usec
DE	9.50 usec
TE	300.0 K
CHST2	160.0000000
DO	0.0000000 sec
D1	2.0000000 sec
SD	0.0010000 sec
DS	0.1000000 sec
D13	0.0000000 sec
D16	0.0001000 sec
END	0.0000280 sec

CHANNEL f1

NUC1	13C
P1	10.70 usec
PC	21.40 usec
PL1	1.00 dB
SFO1	500.1318857 MHz

CHANNEL f2

NUC2	13C
P2	12.00 usec
PL2	-1.00 dB
SFO2	125.7667893 MHz

GRADIENT CHANNEL

GRAN1	SINE.100
GRAN2	SINE.100
GRAN3	SINE.100
GRX1	0.00 %
GRX2	0.00 %
GRX3	0.00 %
GRY1	0.00 %
GRY2	0.00 %
GRY3	0.00 %
GRZ1	50.00 %
GRZ2	30.00 %
GRZ3	40.10 %
P16	1000.00 usec

F1 - Acquisition parameters

MD	2
TD	642
SFO1	125.7668 MHz
FIDRES	42.831688 Hz
SW	174.969 ppm

F2 - Processing parameters

SI	2048
SF	500.1300233 MHz
WDW	SSINE
SSB	2
LB	0.00 Hz
GB	0
PC	0.20

F1 - Processing parameters

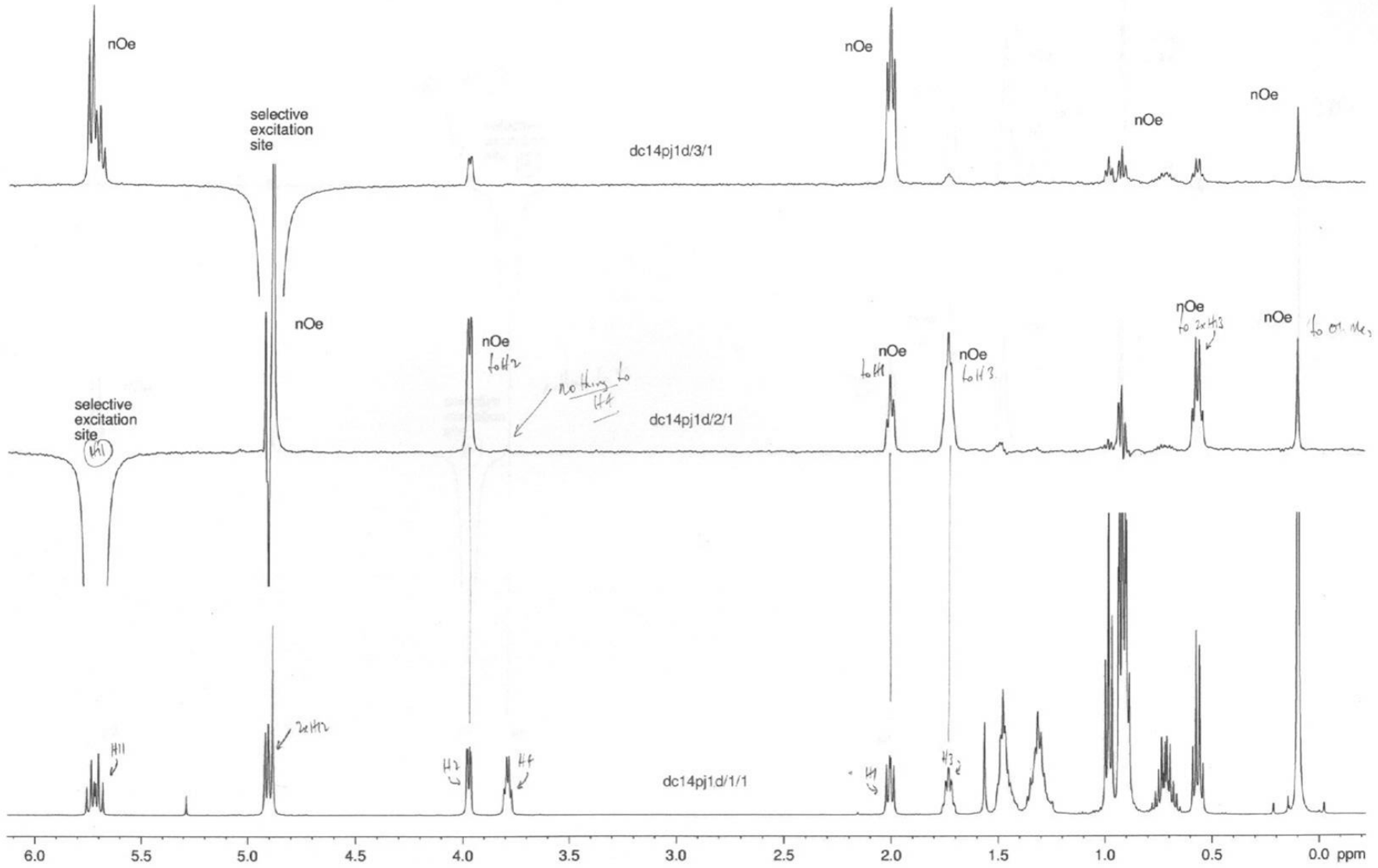
SI	1024
MC2	0F
SF	125.7677910 MHz
WDW	SSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

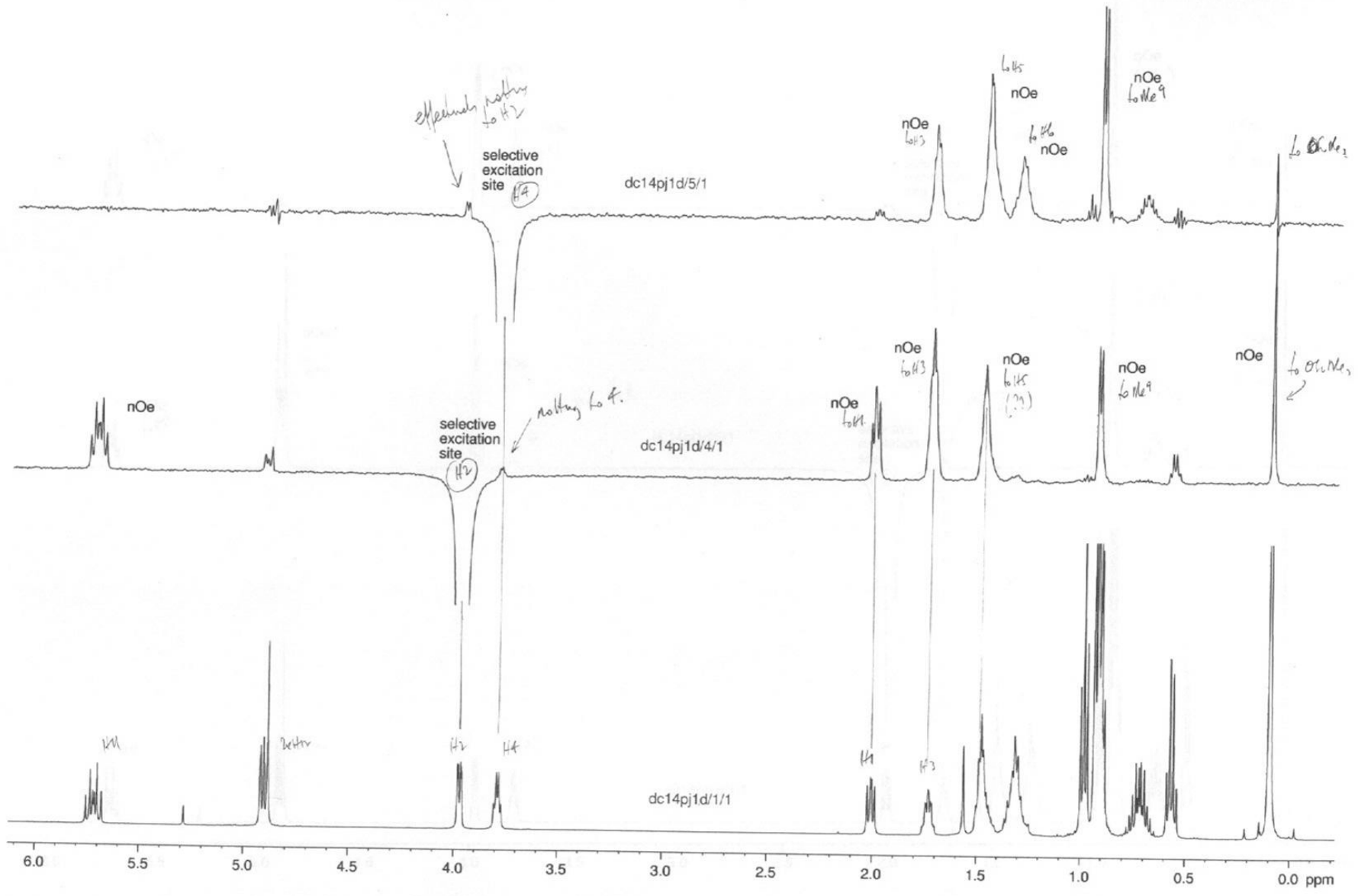
CH2	17.00 cm
CH1	17.00 cm
F2PLD	2.207 ppm
F2LD	1103.06 Hz
F2PH1	1.230 ppm
F2H1	815.40 Hz
F2PLD	42.773 ppm
F2LD	5370.06 Hz
F2PH1	34.298 ppm
F2H1	4306.26 Hz
F2PMCH	0.00743 ppm/cm
F2HCH	26.72643 Hz/cm
F1PMCH	0.50087 ppm/cm
F1HCH	62.98777 Hz/cm

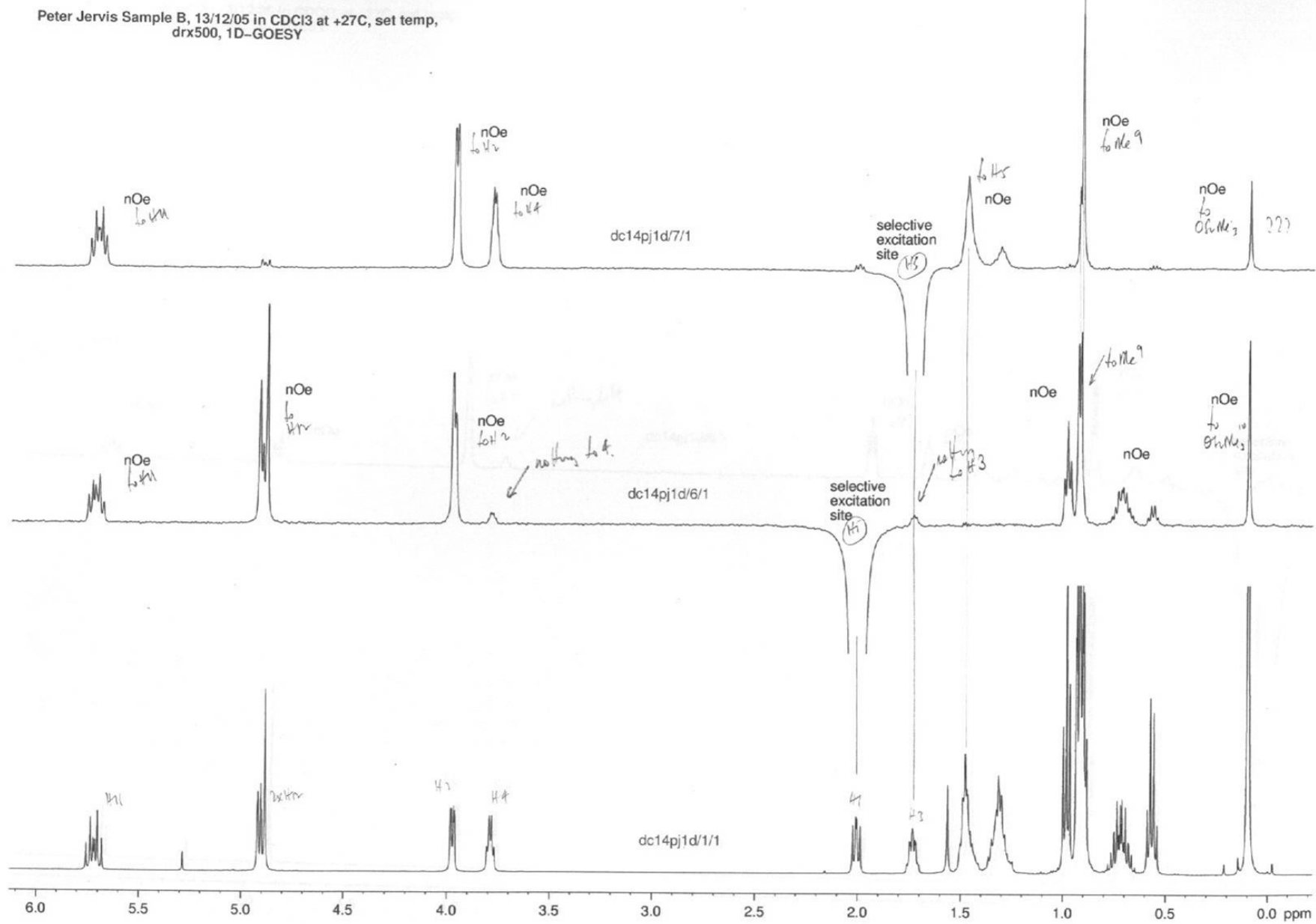


Peter Jarvis Sample B, 13/12/05 in CDCl3 at +27C, set temp, drx500, 1D-GOESY

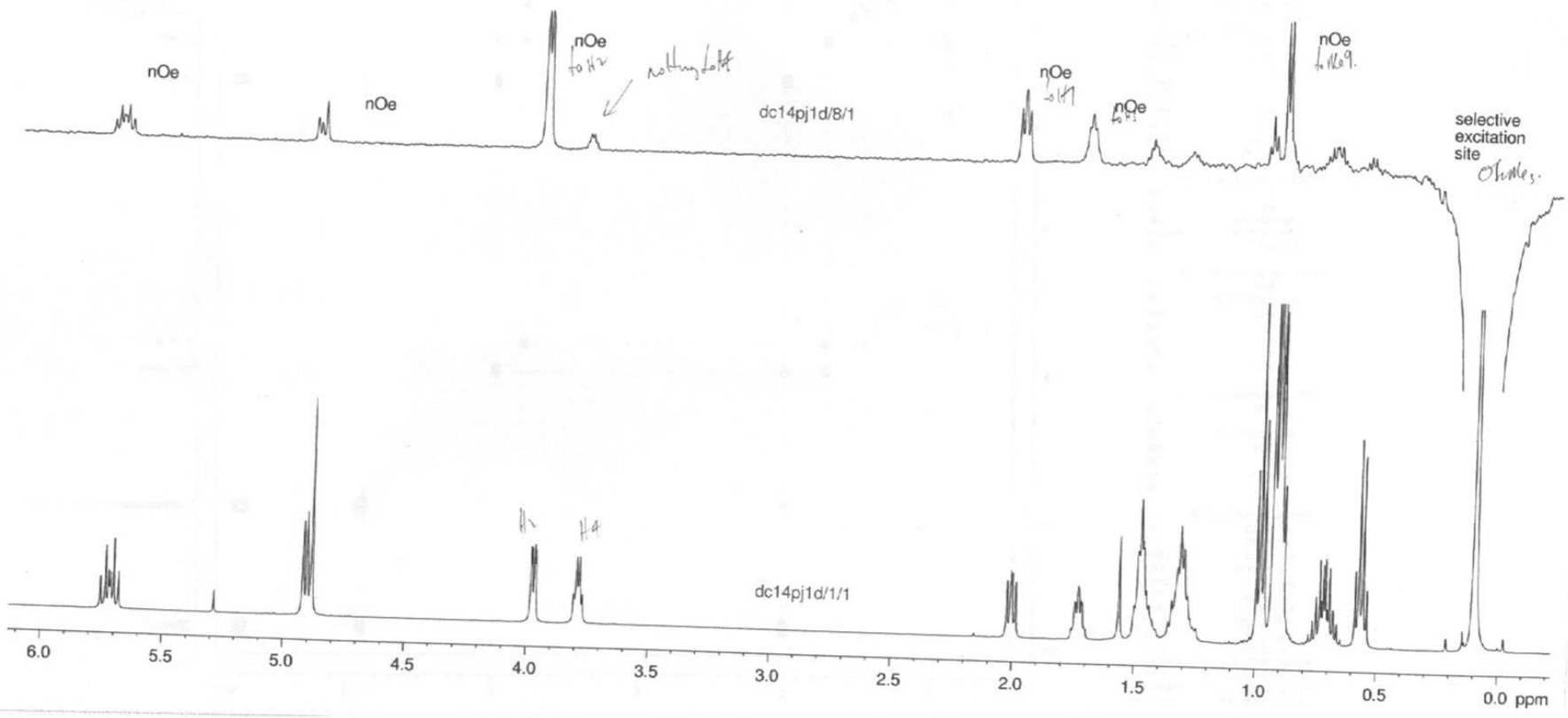


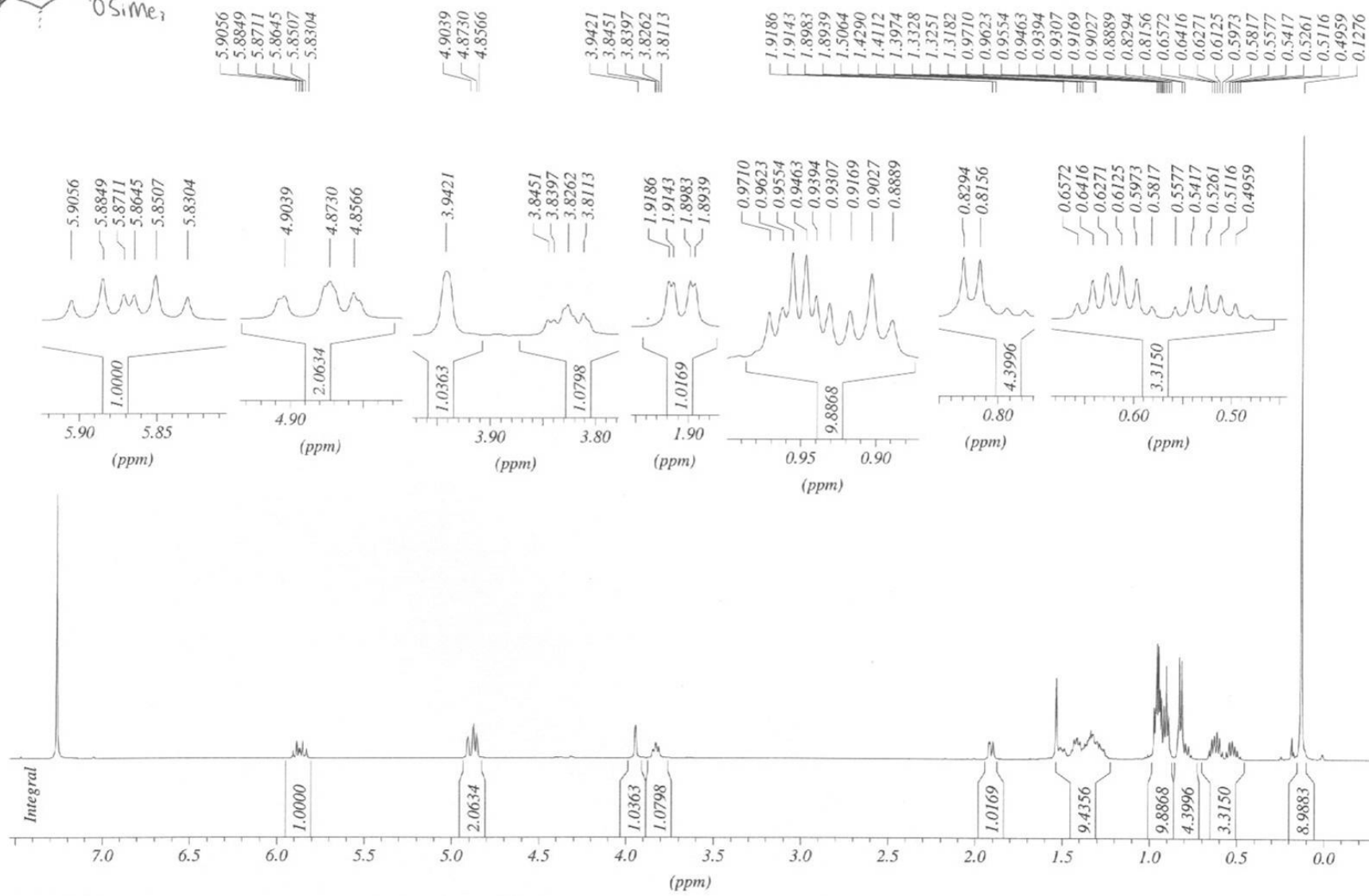
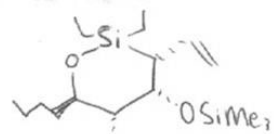
Peter Jervis Sample B, 13/12/05 in CDCl3 at +27C, set temp,  
drx500, 1D-GOESY

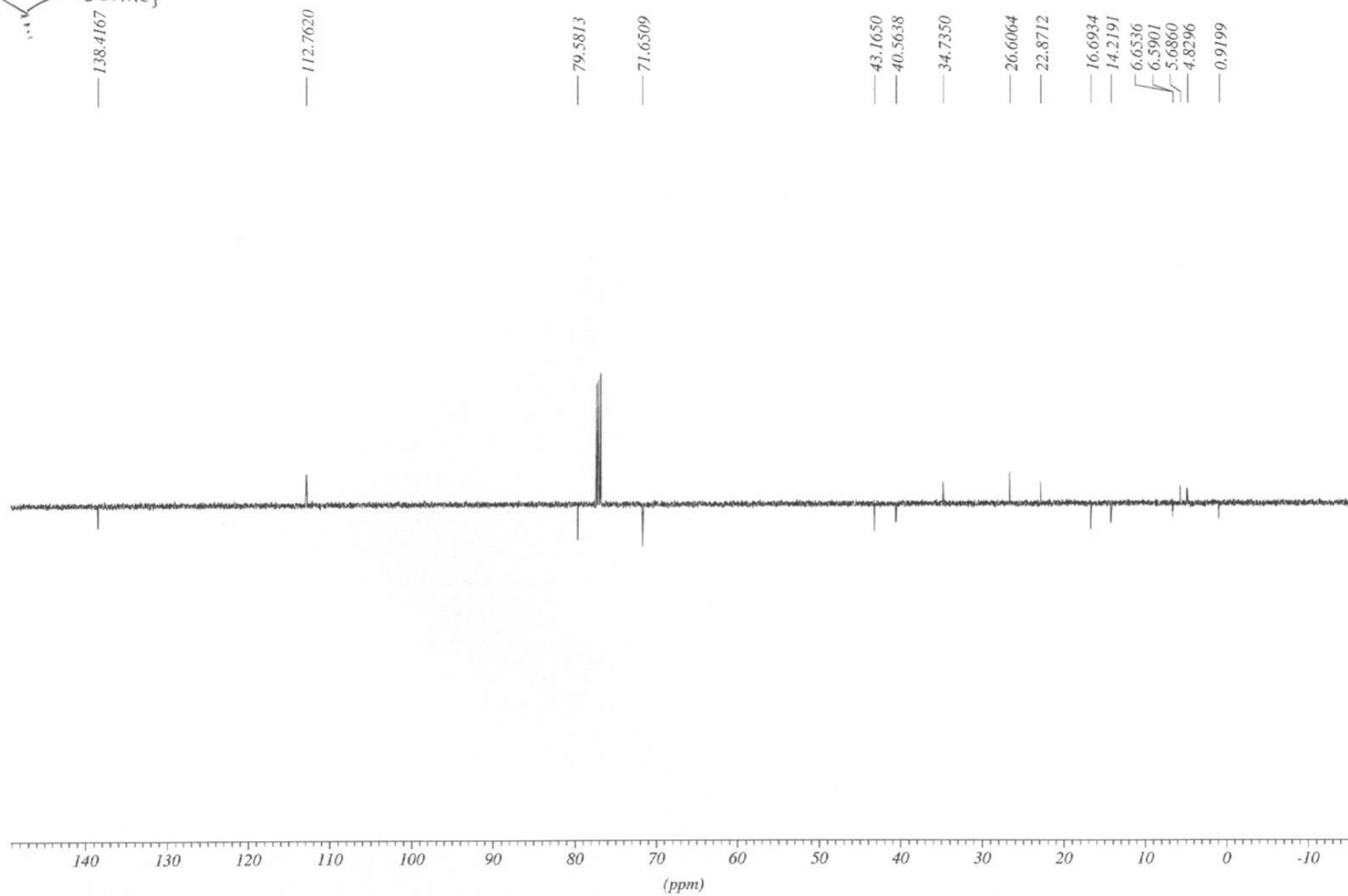
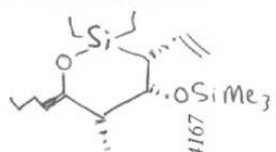




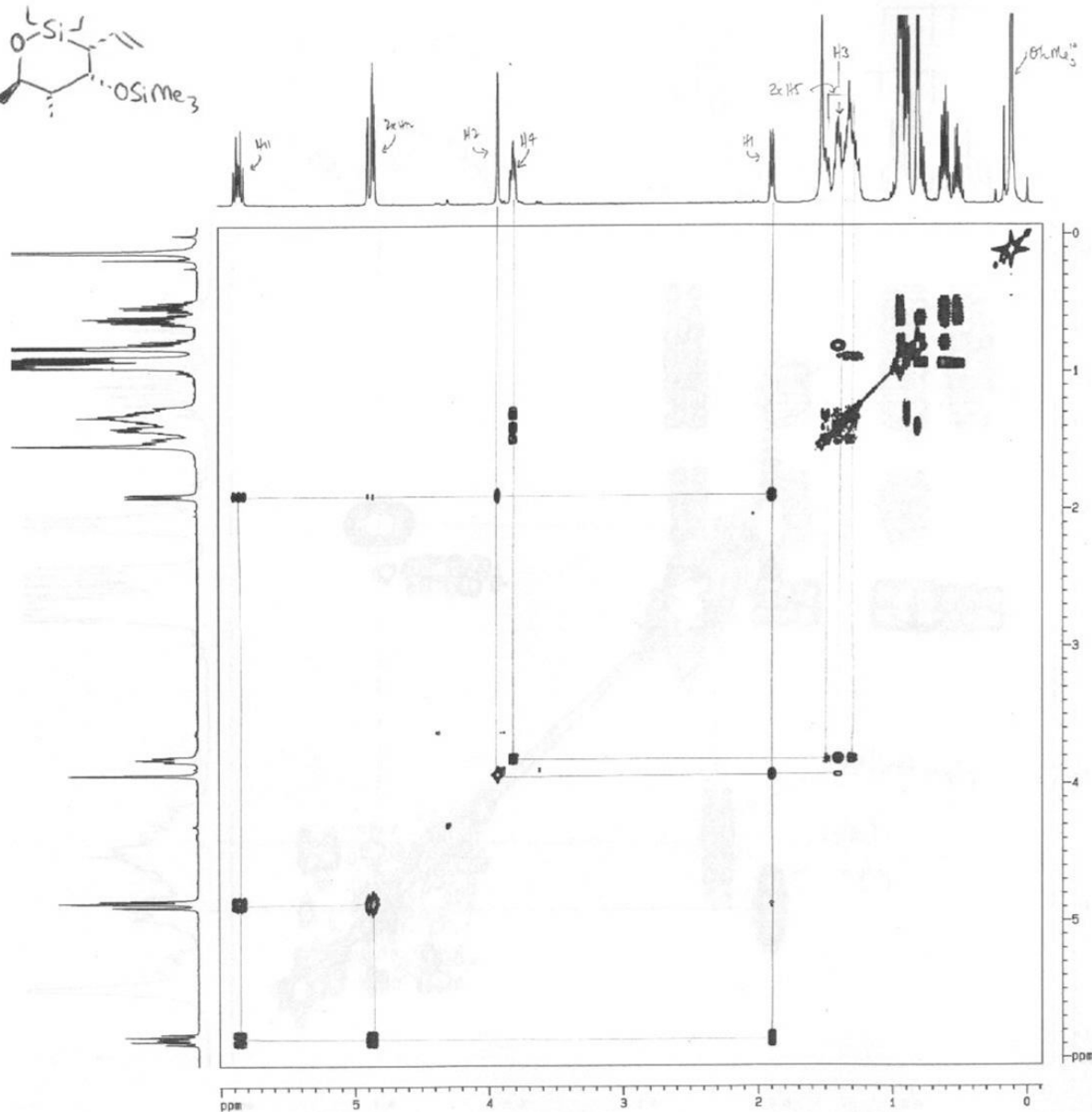
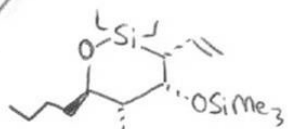
Peter Jervis Sample B, 13/12/05 in CDCl3 at +27C, set temp,  
drx500, 1D-GOESY







Peter Jervis 09/01/06 in CDC13 at +27C, set temp  
drx500, Gradient COSY90



## Current Data Parameters

NAME ja09j10  
EXPNO 4  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090109  
Time 21.49  
INSTRUM drx500  
PROBHD 5 mm TBI H/C  
PULPROG cosygp  
TD 2048  
SOLVENT CDC13  
NS 8  
DS 16  
SWH 4310.345 Hz  
FIDRES 2.104661 Hz  
AQ 0.2376180 sec  
RG 574.7  
DW 116.000 usec  
DE 5.50 usec  
TE 300.0 K  
d0 0.0000300 sec  
d1 2.0000000 sec  
d13 0.0000300 sec  
d16 0.0001000 sec  
IN0 0.0002300 sec

## CHANNEL f1

MUC1 1H  
PC 10.70 usec  
P1 10.70 usec  
PL1 1.00 dB  
SFO1 500.131867 MHz

## GRADIENT CHANNEL

GRAN1 SINE.100  
GRAN2 SINE.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 10.00 %  
GPZ2 10.00 %  
P16 1000.00 usec

## F1 - Acquisition parameters

NDO 1  
TD 512  
SFO1 500.1319 MHz  
FIDRES 8.418642 Hz  
SW 8.618 ppm

## F2 - Processing parameters

SI 2048  
SF 500.1300233 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

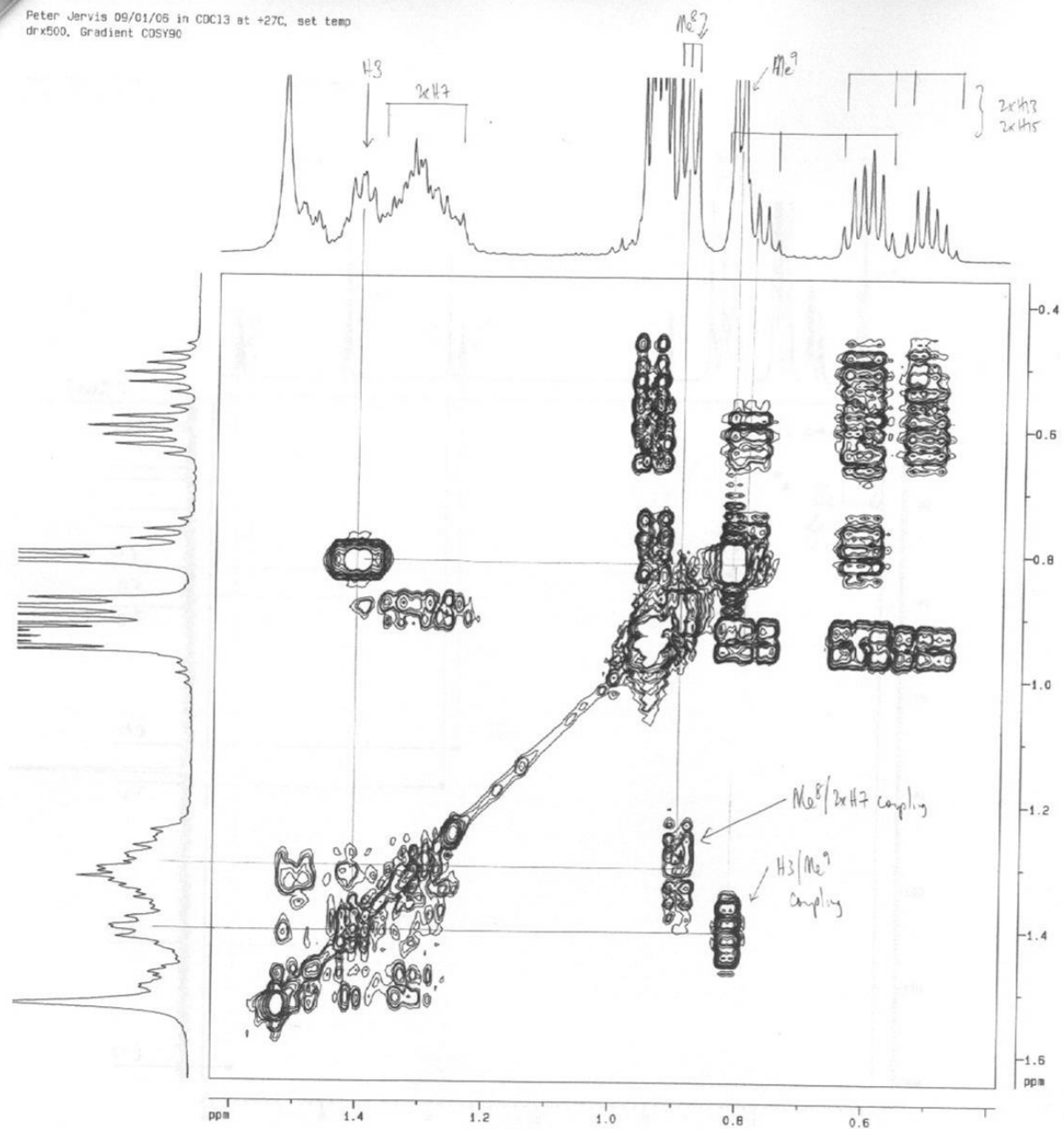
## F1 - Processing parameters

SI 1024  
MC2 GF  
SF 500.1300233 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

## 2D NMR plot parameters

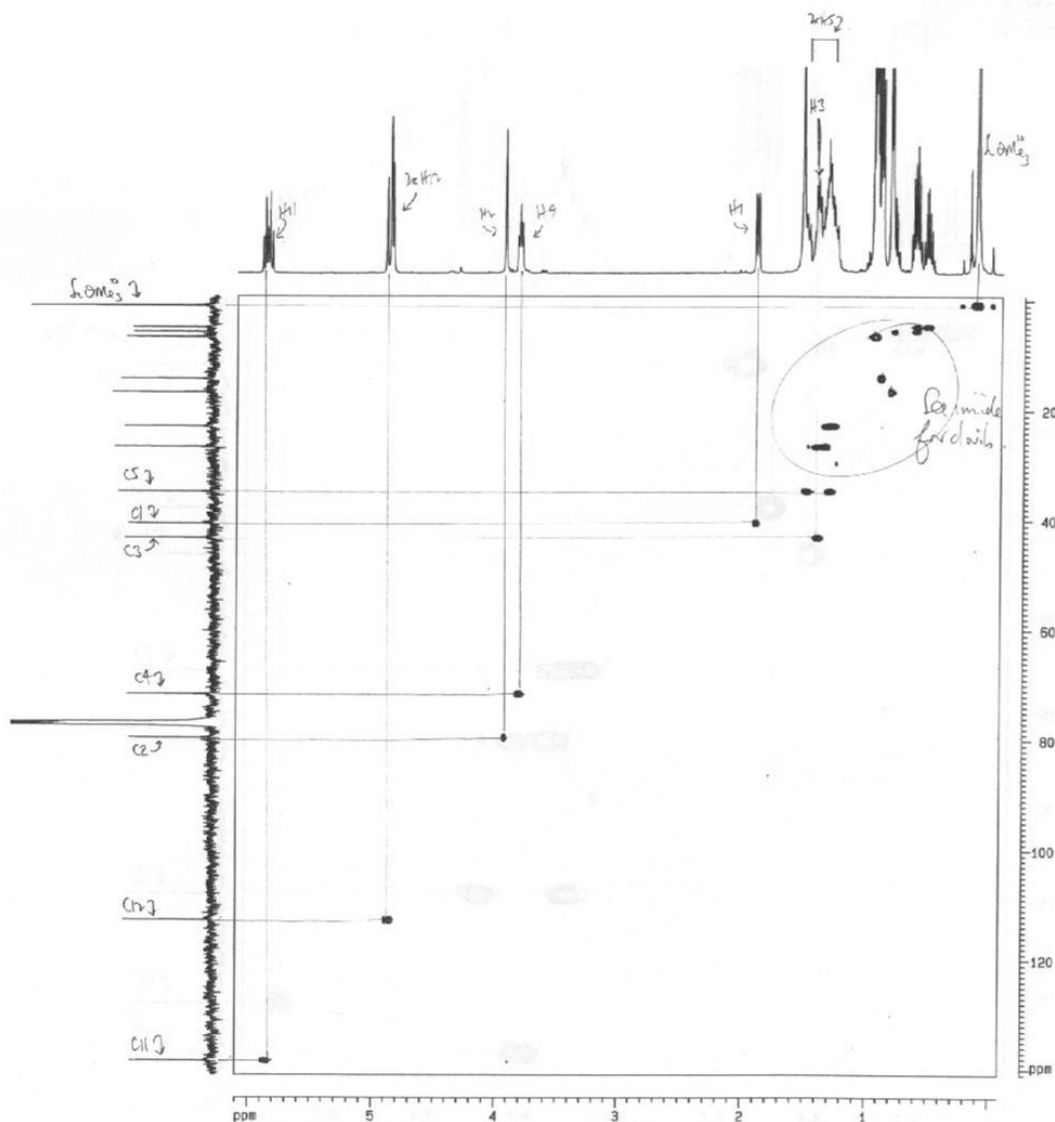
CK2 20.00 cm  
CK1 20.00 cm  
F2PL0 6.911 ppm  
F2PL 3006.29 Hz  
F2PHI -0.115 ppm  
F2NI -99.10 Hz  
F1PL0 6.057 ppm  
F1PL 3029.44 Hz  
F1PHI -0.070 ppm  
F1NI -34.94 Hz  
F2PPMCH 0.30636 ppm/cm  
F2HZCM 153.21930 Hz/cm  
F1PPMCH 0.30636 ppm/cm  
F1HZCM 153.21930 Hz/cm

Peter Jarvis 09/01/06 in CDCl<sub>3</sub> at +27C, set temp  
drx500, Gradient COSY90





Peter Jervis 09/01/06 in CDC13 at +27C, set temp  
drx500, Gradient HSGC



Current Data Parameters  
NAME 1690j1d  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050109  
Time 16.32  
INSTRUM drx500  
PROBHD 5 mm 1H1 H/C  
PULPROG invgpcg3  
TD 2048  
SOLVENT CDC13  
NS 8  
DS 16  
SWH 4319.345 Hz  
FIDRES 2.154661 Hz  
AQ 0.2378180 sec  
RG 32788  
DH 119.000 usec  
DE 0.50 usec  
TE 300.0 K  
CNS12 145.0000000  
d0 0.0000300 sec  
d1 2.0000000 sec  
d4 0.0017244 sec  
d11 0.0300000 sec  
d13 0.0000300 sec  
d16 0.0001000 sec  
d20 0.0011000 sec  
d21 0.0008174 sec  
dN0 0.0000140 sec

CHANNEL f1  
NUC1 1H  
P1 10.70 usec  
p2 21.40 usec  
PL1 1.00 dB  
SFO1 500.1318887 MHz

CHANNEL f2  
CPOPRG2 gwrp  
NUC2 13C  
P2 12.00 usec  
p4 24.00 usec  
PCPG2 78.00 usec  
PL2 -1.00 dB  
PL12 15.00 dB  
SFO2 125.7677853 MHz

GRADIENT CHANNEL  
GPMAN1 SINE 100  
GPMAN2 SINE 100  
GPMAN3 SINE 100  
GPA1 0.00 %  
GPA2 0.00 %  
GPA3 0.00 %  
GPA4 0.00 %  
GPA5 0.00 %  
GPA6 0.00 %  
GPA7 80.00 %  
GPA8 30.00 %  
GPA9 20.10 %  
P18 1000.00 usec

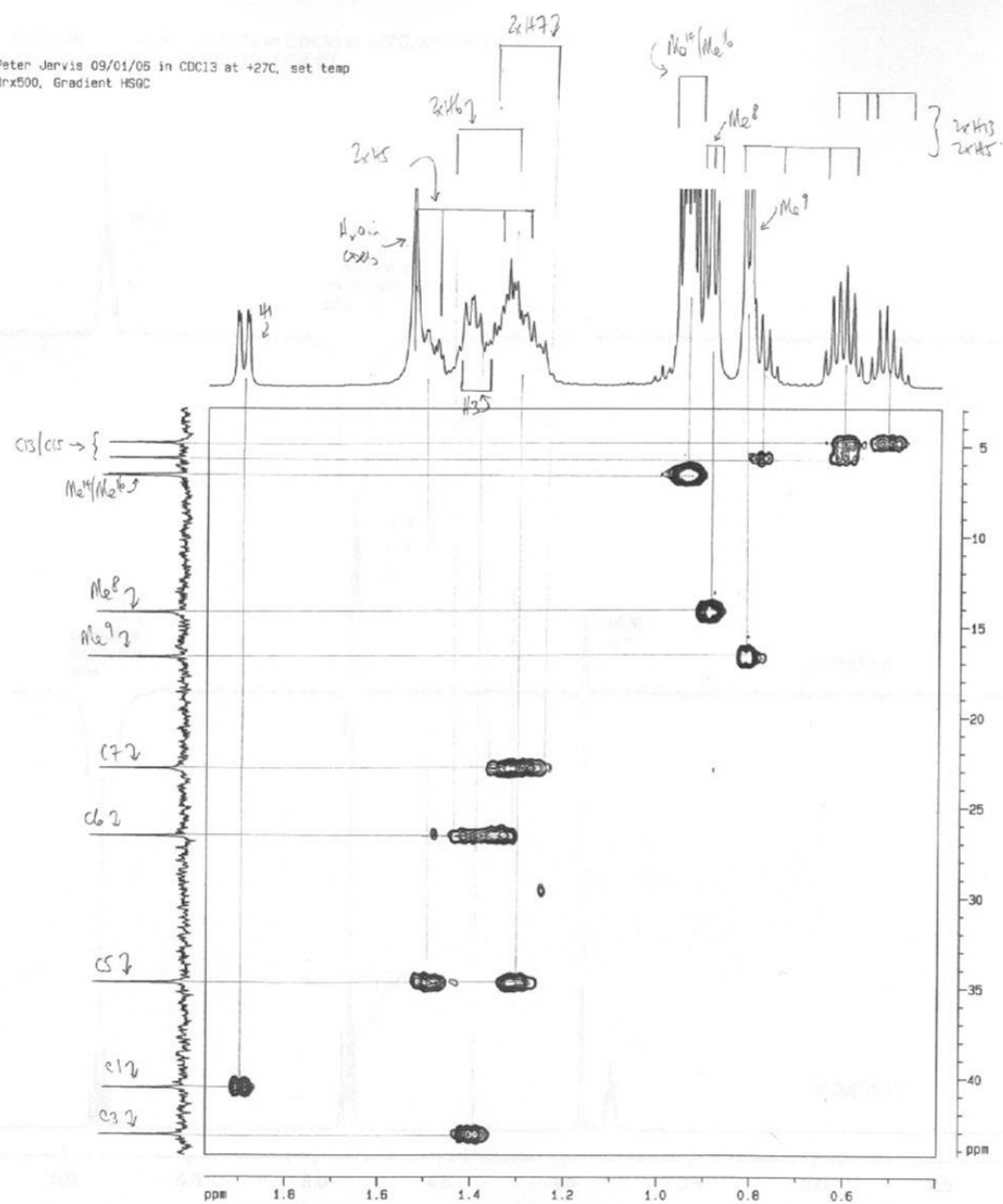
F1 - Acquisition parameters  
ND0 4  
TD 812  
SFO1 125.7698 MHz  
FIDRES 42.831698 Hz  
SW 174.389 ppm

F2 - Processing parameters  
SI 2048  
SF 500.1300233 MHz  
WDW 981HC  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

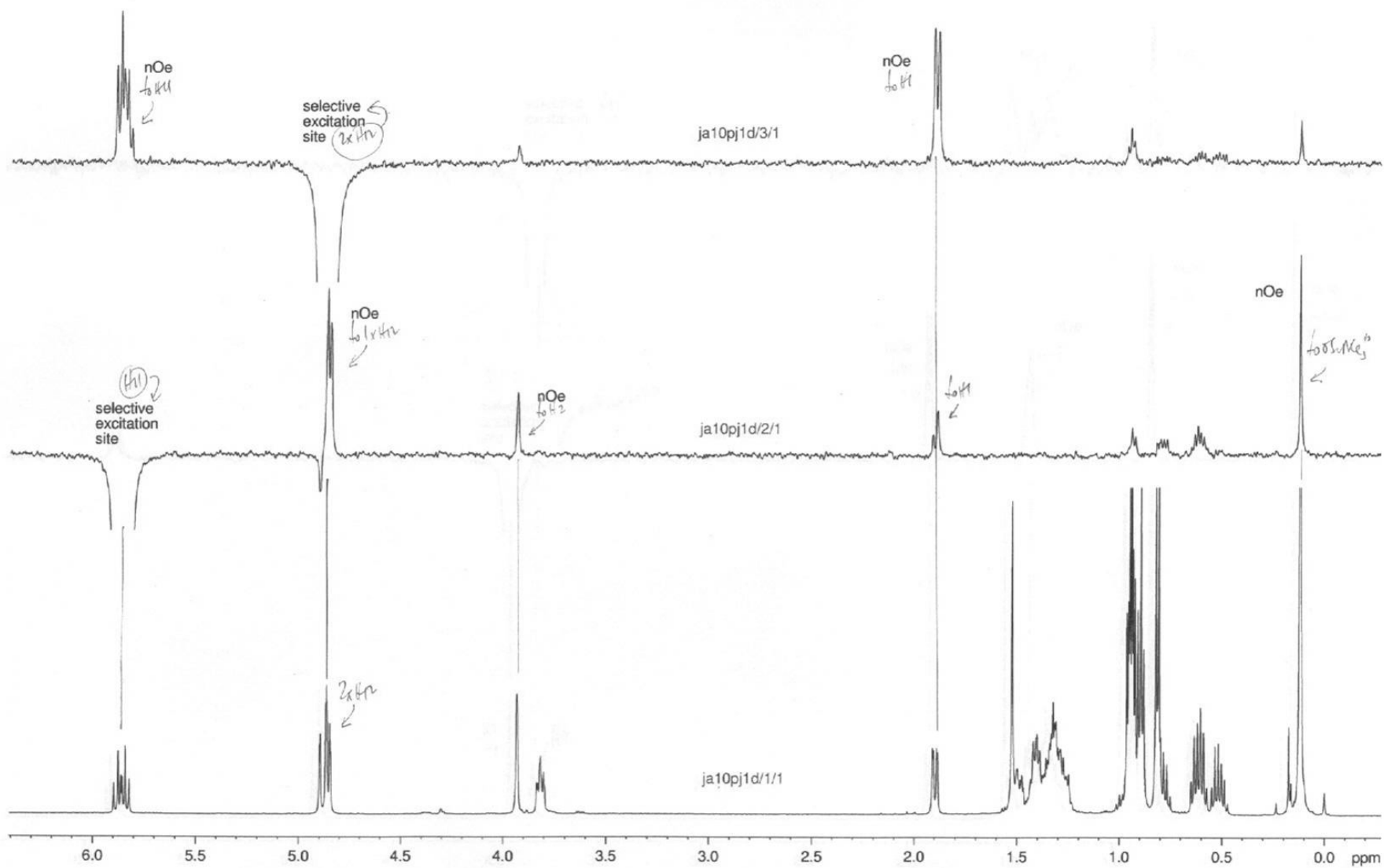
F1 - Processing parameters  
SI 1024  
MC2 TPP1  
SF 125.7577910 MHz  
WDW 981HC  
SSB 2  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 17.00 cm  
CX1 17.00 cm  
F2PL0 0.104 ppm  
F1L0 3050.00 Hz  
F2PHI -0.002 ppm  
F2M1 -41.26 Hz  
F1PL0 141.033 ppm  
F1L1 17736.00 Hz  
F1PHI -0.522 ppm  
F1M1 -103.40 Hz  
F2PHICH 0.36389 ppm/c  
F2M2CH 181.99104 Hz/c  
F1PHICH 0.54442 ppm/c  
F1M2CH 1049.37634 Hz/c

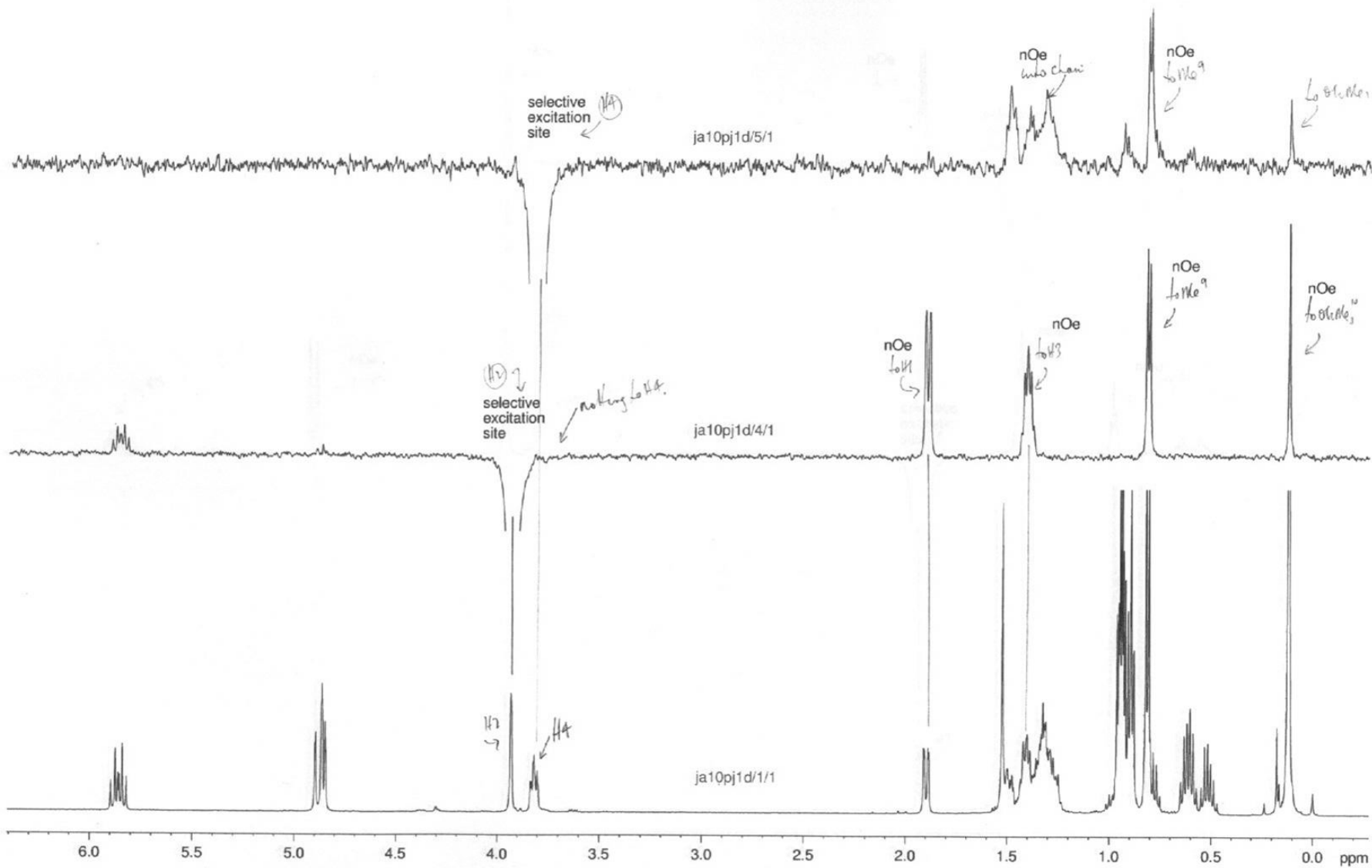
Peter Jarvis 09/01/06 in CDCl<sub>3</sub> at +27C, set temp  
 drx500, Gradient HSGC



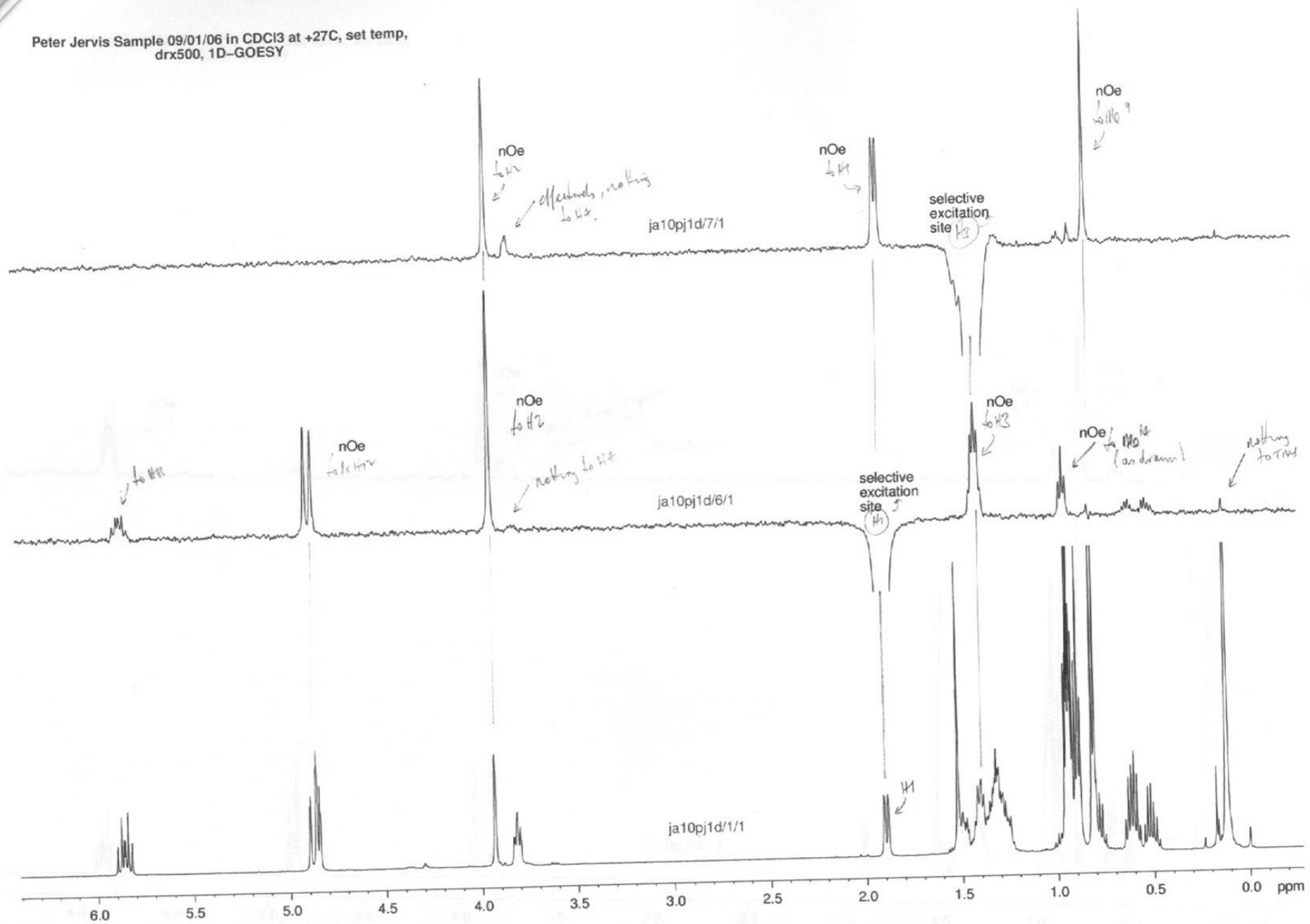
Peter Jervis Sample 09/01/06 in CDCl<sub>3</sub> at +27C, set temp,  
drx500, 1D-GOESY



Peter Jarvis Sample 09/01/06 in CDCl3 at +27C, set temp,  
drx500, 1D-GOESY



Peter Jervis Sample 09/01/06 in CDCl<sub>3</sub> at +27C, set temp,  
drx500, 1D-GOESY



Peter Jervis Sample 09/01/06 in CDCl<sub>3</sub> at +27C, set temp,  
drx500, 1D-GOESY

