

Additional File 7

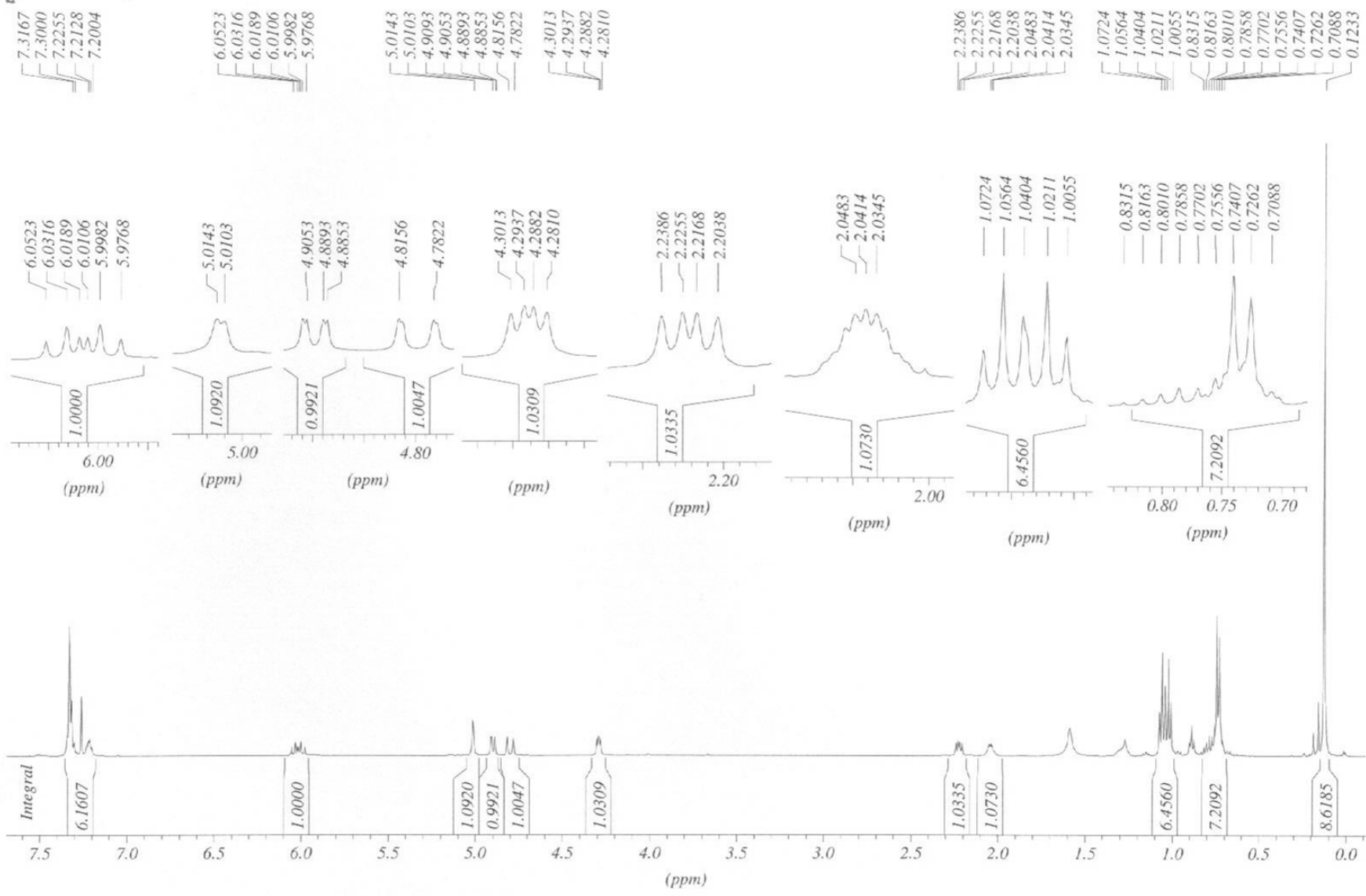
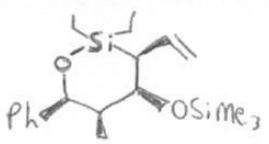
Tether-directed synthesis of highly substituted oxasilacycles *via* an intramolecular allylation employing allylsilanes

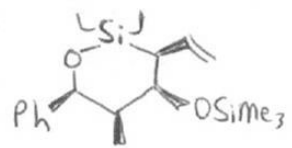
Peter J. Jervis and Liam R. Cox*

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

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

18b, 19b





— 143.0362

— 137.2709

— 127.8099

— 126.4697

— 125.5022

— 113.9714

— 76.2387

— 74.1134

— 46.0160

— 38.2919

7.6013

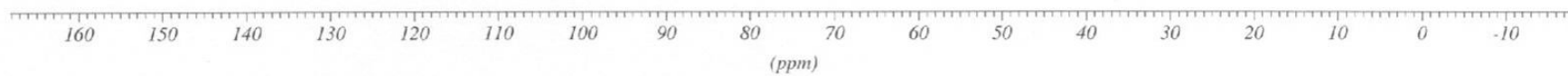
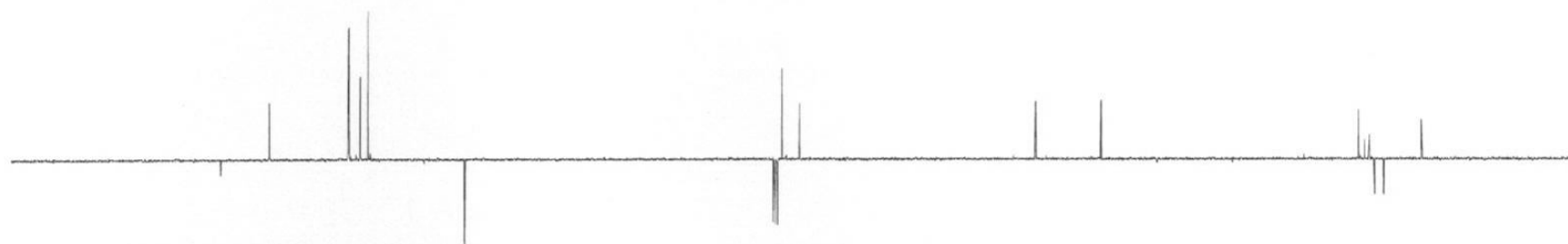
6.9351

6.3007

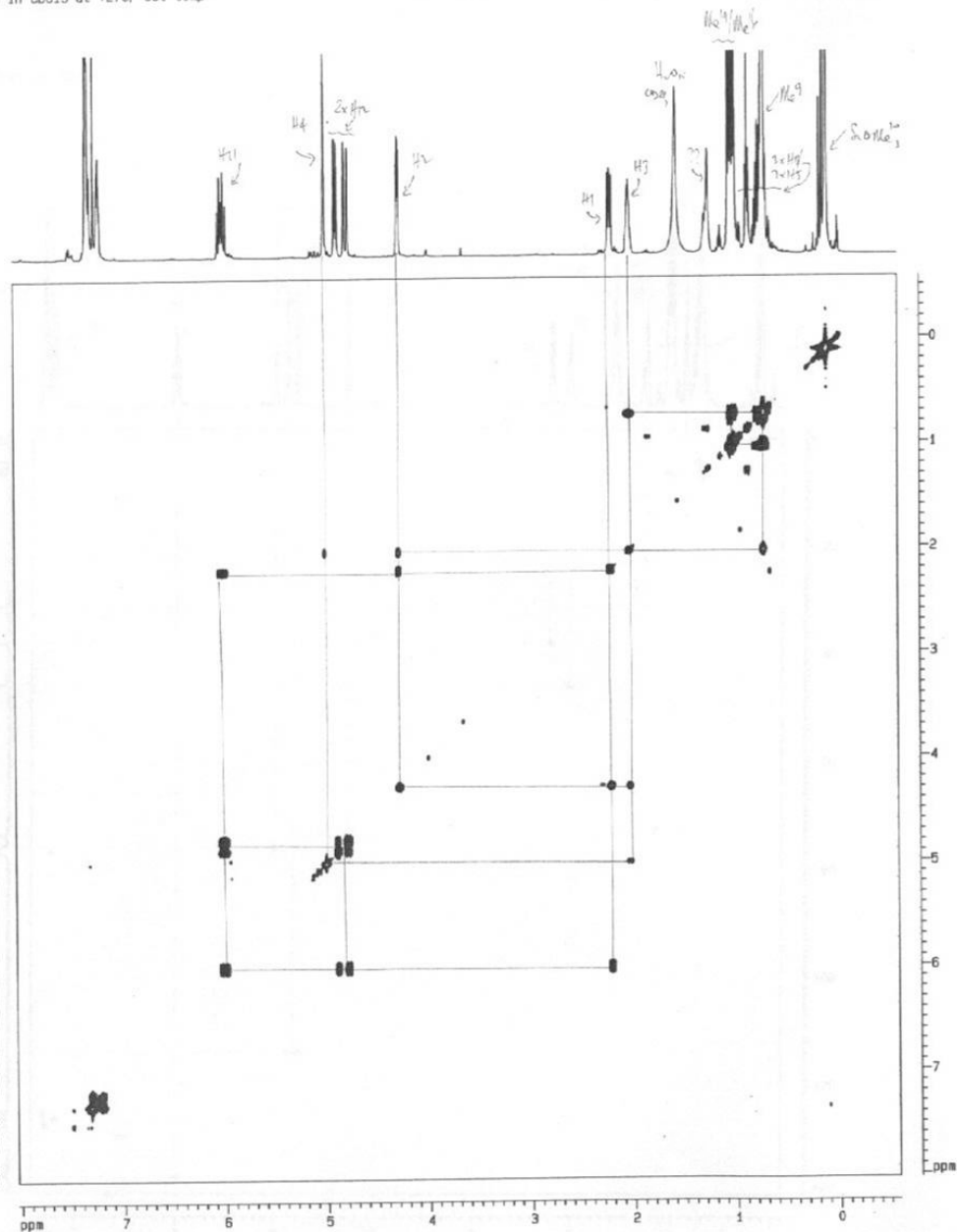
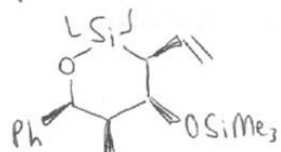
5.7139

4.6512

0.1864



Peter Jervis Sample 04/10/05 in CDCl3 at +27C, set temp
drex500, Gradient COSY90



Current User Parameters
 NAME oc040j10
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051005
 Time 12.17
 INSTRUM drex500
 PROBHD 5 mm TBI H/C
 PULPROG cosygp
 TD 2048
 SOLVENT CDCl3
 NS 8
 DS 16
 SWH 4310.345 Hz
 FIDRES 2.104661 Hz
 AQ 0.2378180 sec
 RG 128
 DM 118.000 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
 INO 0.0023200 sec

----- CHANNEL f1 -----
 NUC1 1H
 P0 10.70 usec
 P1 10.70 usec
 PL1 1.00 dB
 SFO1 500.1318867 MHz

----- GRADIENT CHANNEL -----
 GPMAN1 SINE.100
 GPMAN2 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
 MD 1
 TD 812
 SFO1 500.1318 MHz
 FIDRES 8.418642 Hz
 SW 8.618 ppm

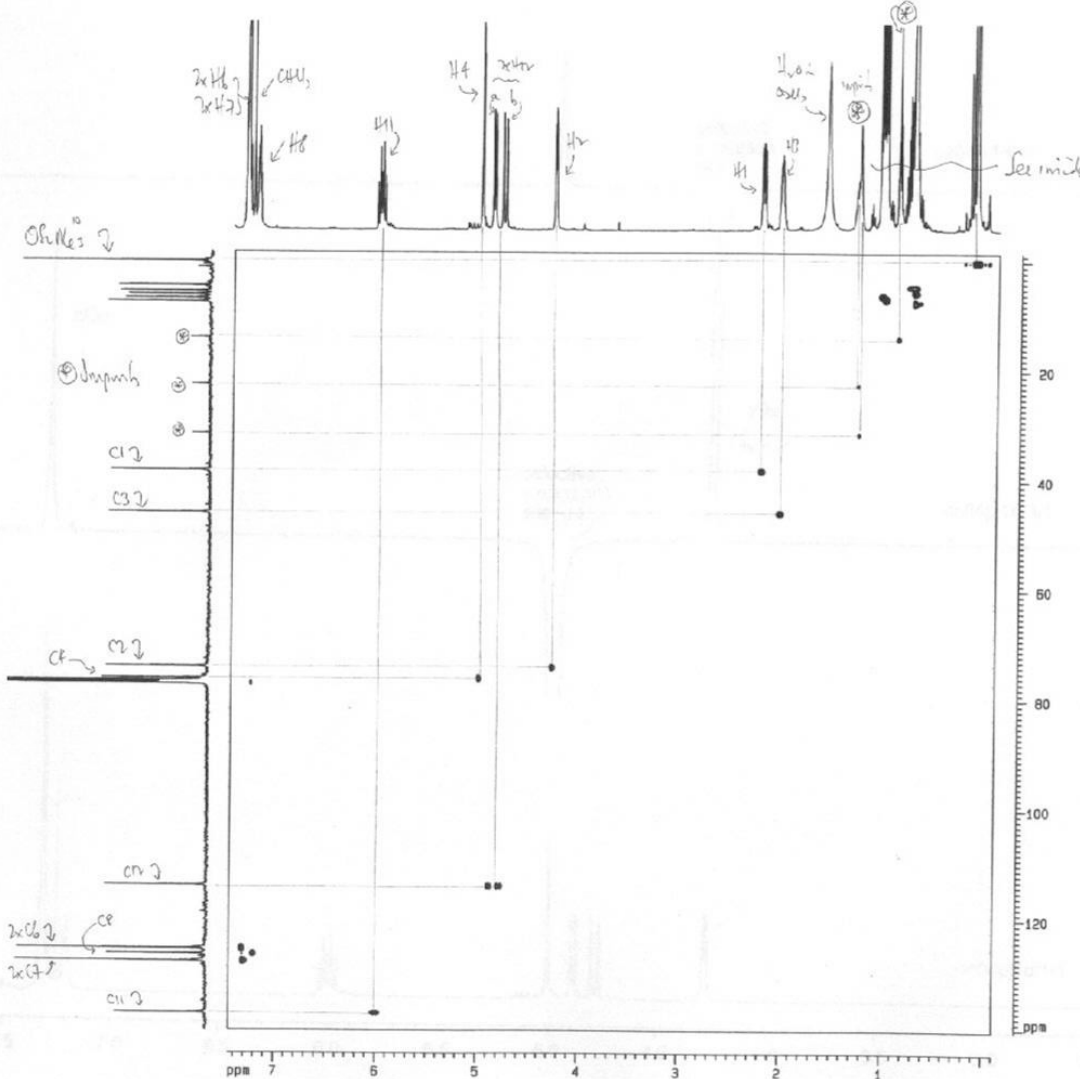
F2 - Processing parameters
 SI 2048
 SF 500.1300233 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

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
 CX2 20.00 cm
 CX1 20.00 cm
 F2PLD 8.035 ppm
 F2LD 4018.63 Hz
 F2PHI -0.583 ppm
 F2HI -251.71 Hz
 F1PLD 8.035 ppm
 F1LD 4018.63 Hz
 F1PHI -0.583 ppm
 F1HI -251.71 Hz
 F2PRMCH 0.43092 ppa/cm
 F2KZCH 215.51724 Hz/cm
 F1PRMCH 0.43092 ppa/cm
 F1KZCH 215.51724 Hz/cm

Peter Jervis Sample 04/10/05 in CDCl3 at +27C, set temp
drx500, Gradient HSGC

⊕ Impurity



Current Data Parameters
 NAME cc04p116
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20051004
 Time 15.35
 INSTRUM drx500
 PROBHD 5 mm TBI H/C
 PULPROG invgpgp
 TO 20.48
 SOLVENT CDCl3
 NS 8
 DS 8
 SWH 431.345 Hz
 FIDRES 2.104661 Hz
 AQ 0.2376180 sec
 RG 32788
 DM 150.000 usec
 DE 9.50 usec
 TE 300.0 K
 CHET2 145.000000
 d0 0.0000300 sec
 d1 2.0000000 sec
 d4 0.00172414 sec
 d11 0.0000000 sec
 d13 0.0000300 sec
 d18 0.0001000 sec
 d20 0.0011000 sec
 d21 0.00061714 sec
 dM0 0.00001140 sec

CHANNEL F1
 NUC1 1H
 P1 19.70 usec
 p2 21.40 usec
 PL1 1.00 dB
 SFO1 500.130087 MHz

CHANNEL F2
 CPDPRG2 gprp
 NUC2 13C
 P2 12.00 usec
 p4 24.00 usec
 PCPD2 78.00 usec
 PL2 -1.00 dB
 PL3 15.00 dB
 SFO2 125.7677693 MHz

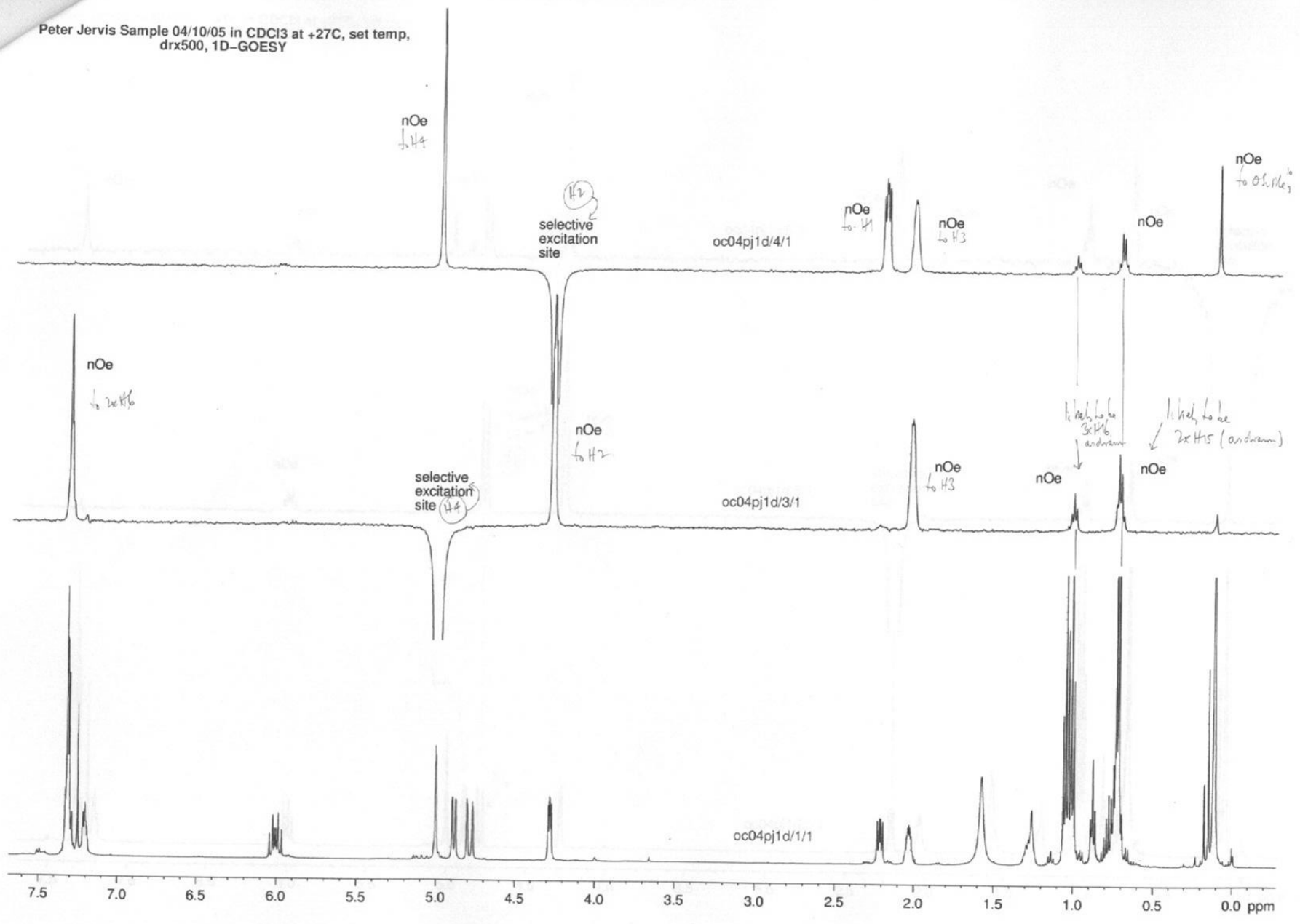
GRADIENT CHANNEL
 GRANA1 SINE 100
 GRANA2 SINE 100
 GRANA3 SINE 100
 GPC1 0.00 %
 GPC2 0.00 %
 GPC3 0.00 %
 GPT1 0.00 %
 GPT2 0.00 %
 GPT3 0.00 %
 GPT4 80.00 %
 GPT5 30.00 %
 GPC5 20.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 NS0 4
 TO 512
 SFO1 125.7698 MHz
 FIDRES 42.831888 Hz
 SW 174.389 pps

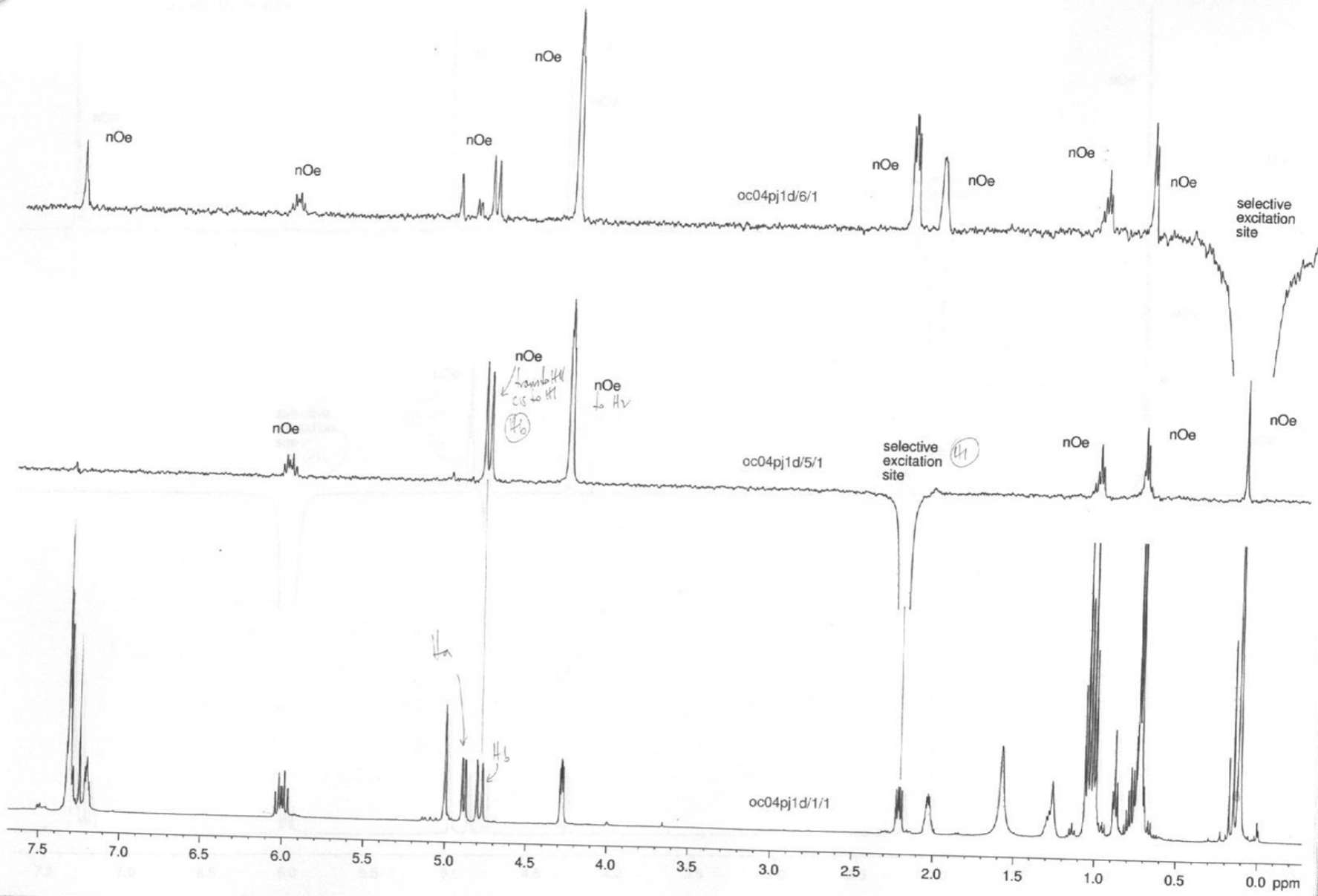
F2 - Processing parameters
 SI 20.48
 SF 500.1300233 MHz
 WM 95196
 SSB 2
 LB 0.00 Hz
 GB 0
 PC 1.00

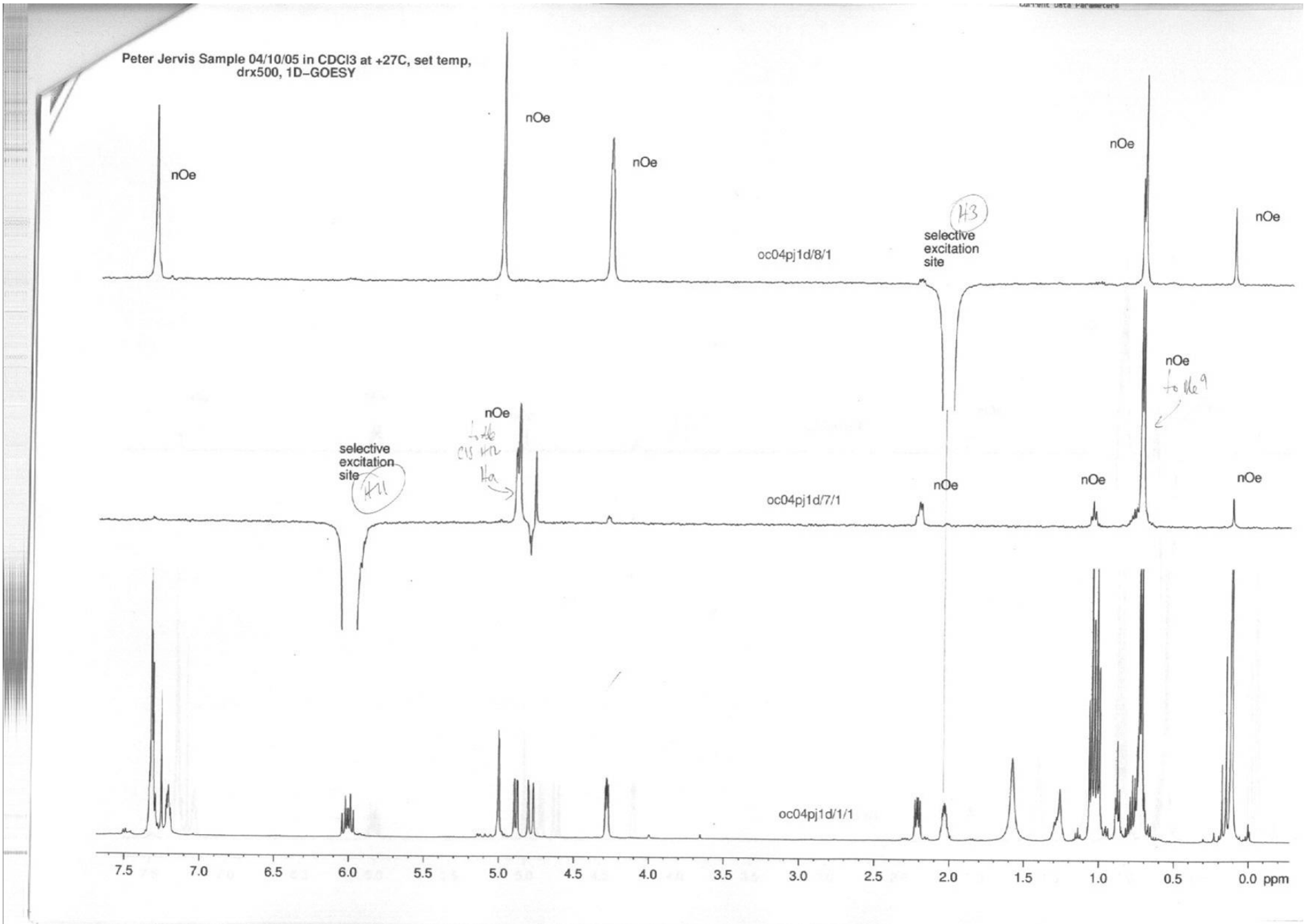
F1 - Processing parameters
 SI 1024
 MC2 TP2
 SF 125.7677640 MHz
 WM 95196
 SSB 2
 LB 0.00 Hz
 GB 0

2D NMR plot parameters
 CX2 17.00 ca
 CX1 17.00 ca
 F2PLD 7.490 ppm
 F2LD 3726.00 Hz
 F2PHI -9.104 ppm
 F2HG -51.78 Hz
 F2PLD 140.484 ppm
 F2LD 17088.20 Hz
 F2PHI -1.531 ppm
 F2HG -192.56 Hz
 F2PHICH 0.44434 pps/ca
 F2PHCN 232.22740 Hz/ca
 F2PHCH 8.25444 pps/ca
 F2HZCH 1000.03611 Hz/ca

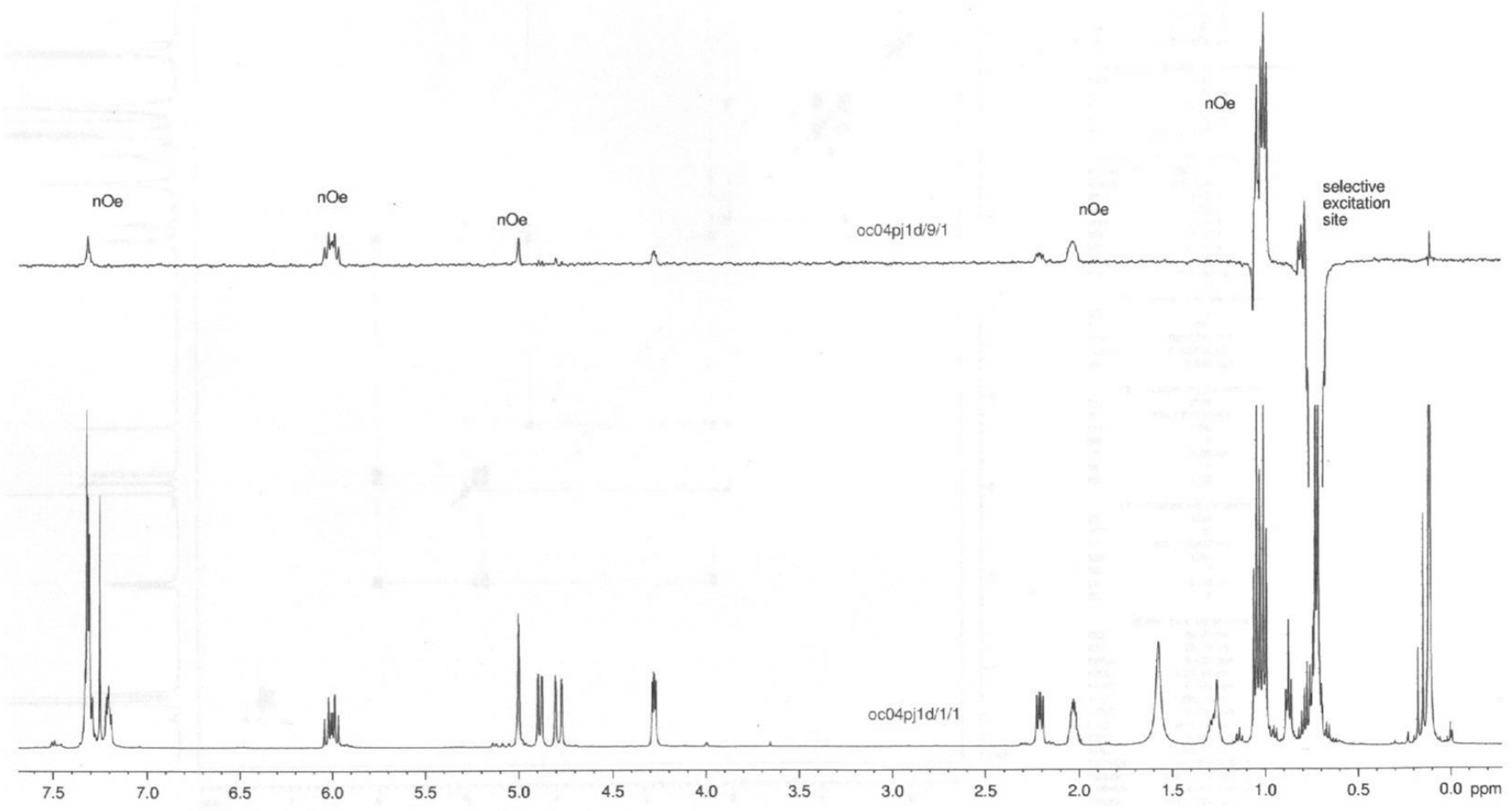


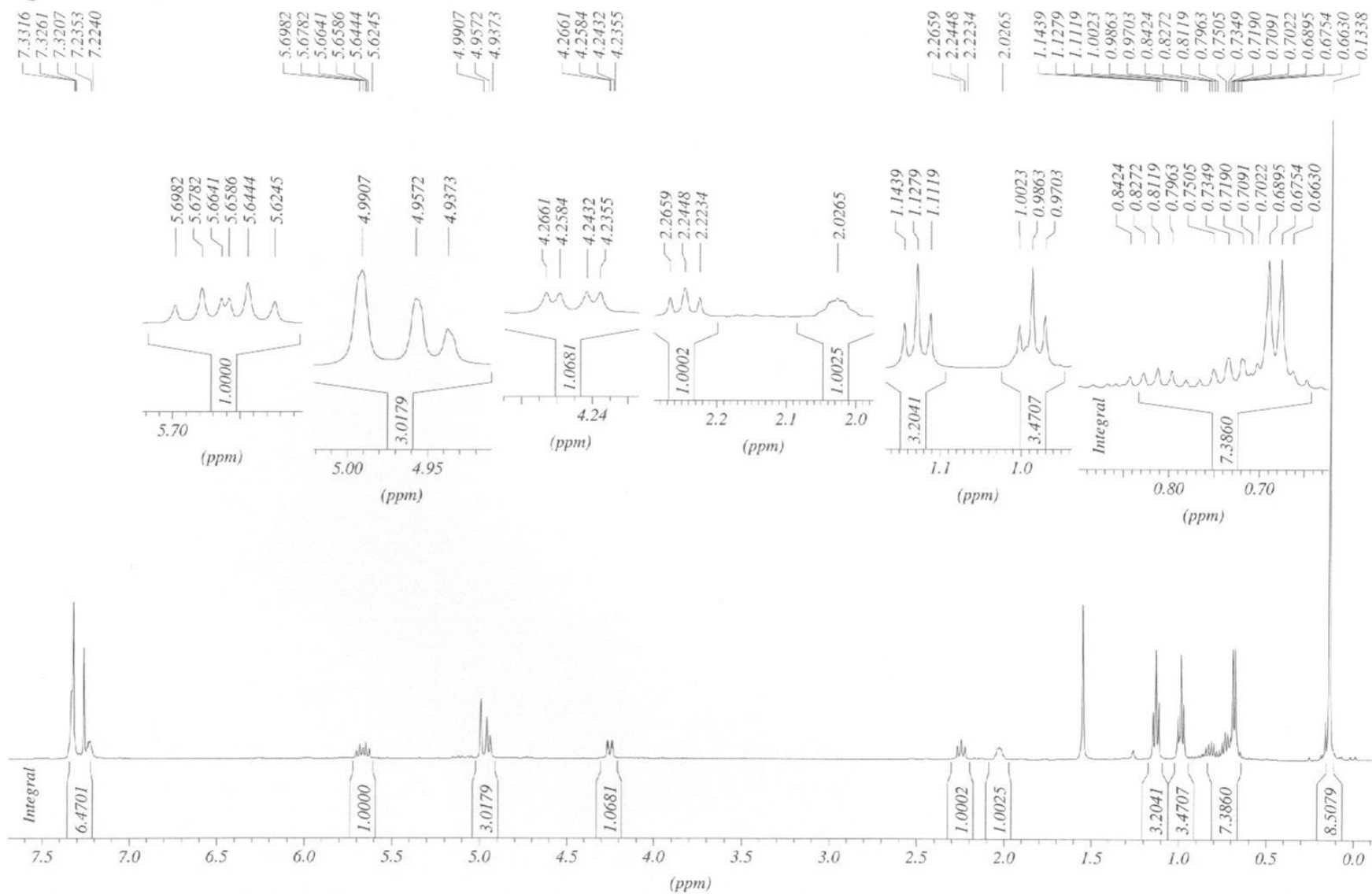
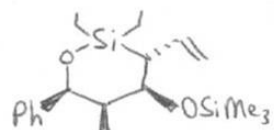
Peter Jervis Sample 04/10/05 in CDCl3 at +27C, set temp,
drx500, 1D-GOESY

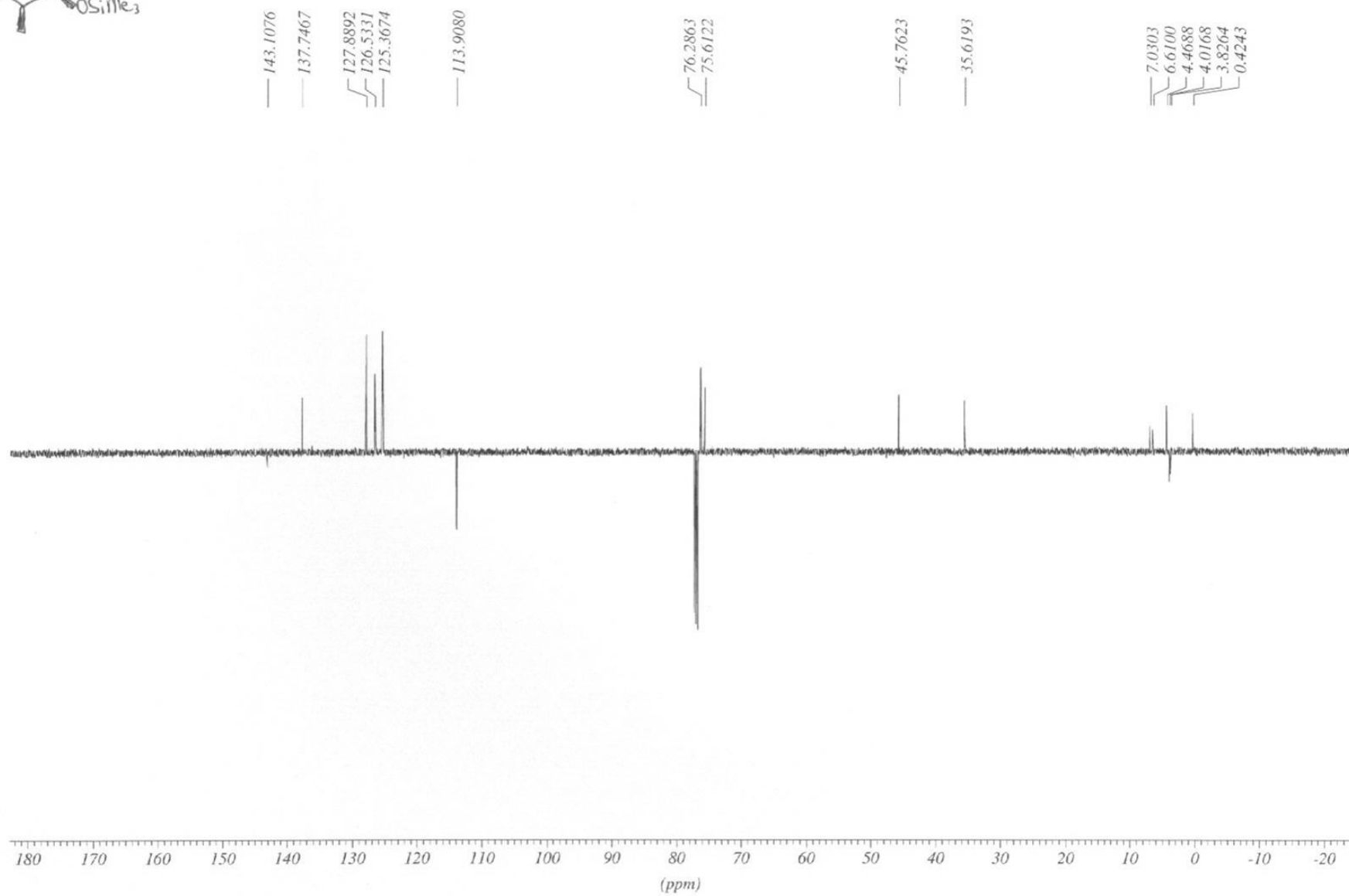
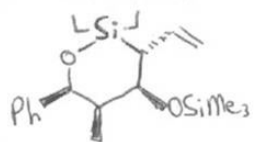




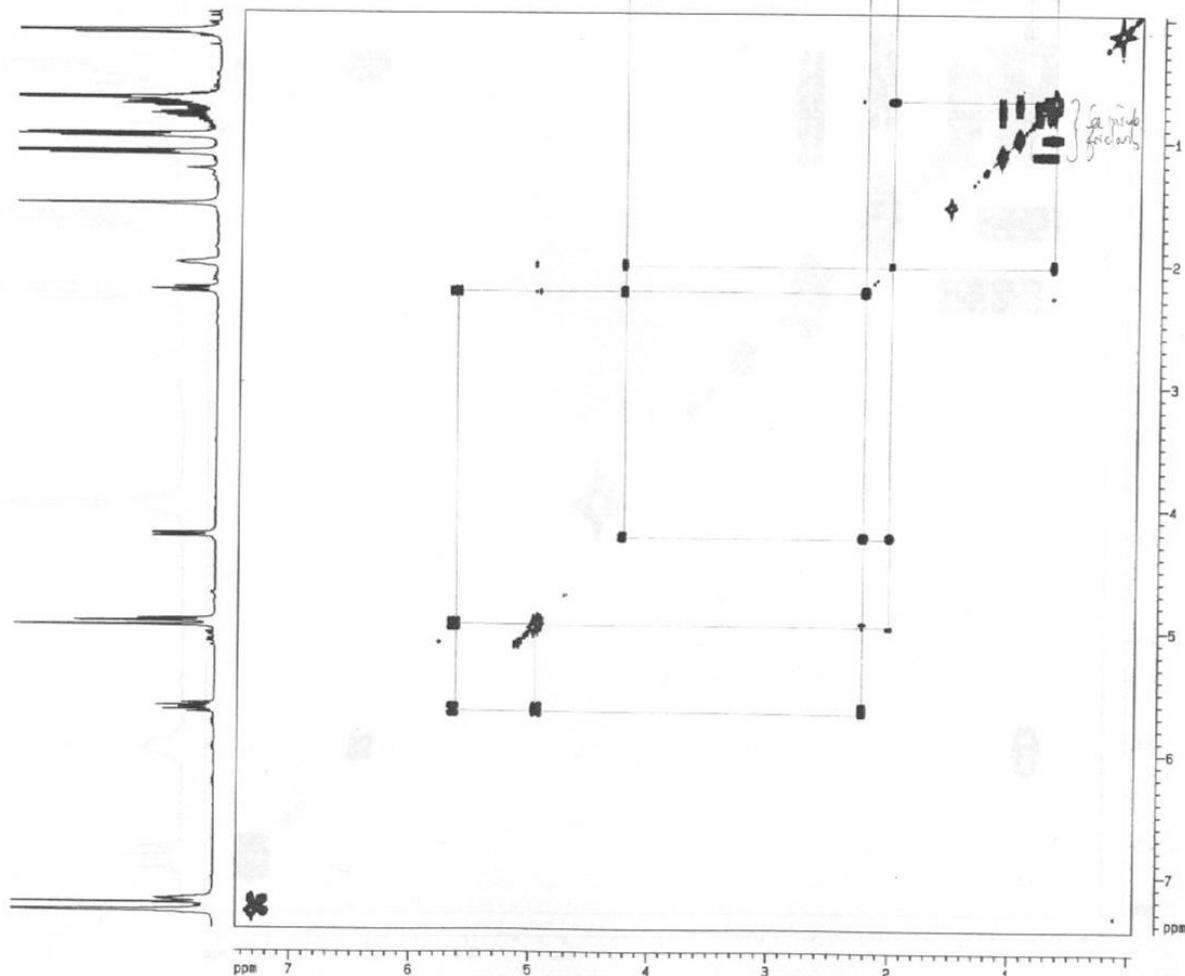
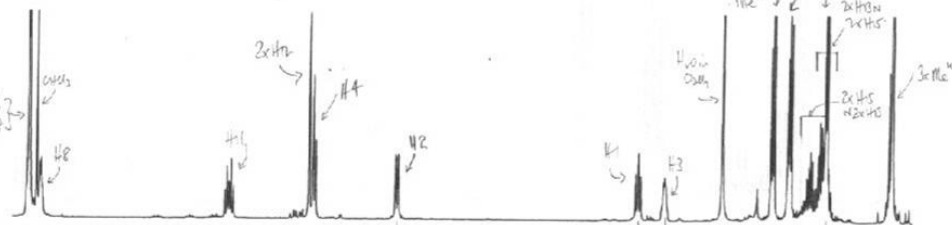
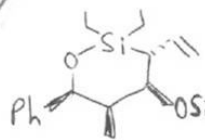
Peter Jervis Sample 04/10/05 in CDCl3 at +27C, set temp,
drx500, 1D-GOESY







Peter Jarvis Barcode 3905 in CDC13 at +27C, set temp
dix500, Gradient COSY90



Current Data Parameters
NAME j12epj1d
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050725
Time 19.47
INSTRUM dix500
PROBHD 5 mm TBI H/C
PULPROG cosygp
TD 2048
SOLVENT CDC13
NS 8
DS 16
SWH 4310.345 Hz
FIDRES 2.1046651 Hz
AQ 0.2375180 sec
RG 485.1
DM 115.000 usec
DE 5.50 usec
TE 300.0 K
d0 0.00000300 sec
D1 2.00000000 sec
d13 0.00000300 sec
D15 0.00010000 sec
IN0 0.00023200 sec

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

NUC1 1H
P0 10.80 usec
P1 10.80 usec
PL1 1.00 dB
SFO1 500.131867 MHz

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

GRNAM1 SINE.100
GRNAM2 SINE.100
SPX1 0.00 %
SPX2 0.00 %
SPY1 0.00 %
SPY2 0.00 %
SPZ1 10.00 %
SPZ2 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters

MD0 1
TD 512
SFO1 500.1318 MHz
FIDRES 8.418642 Hz
SW 8.618 ppm

F2 - Processing parameters

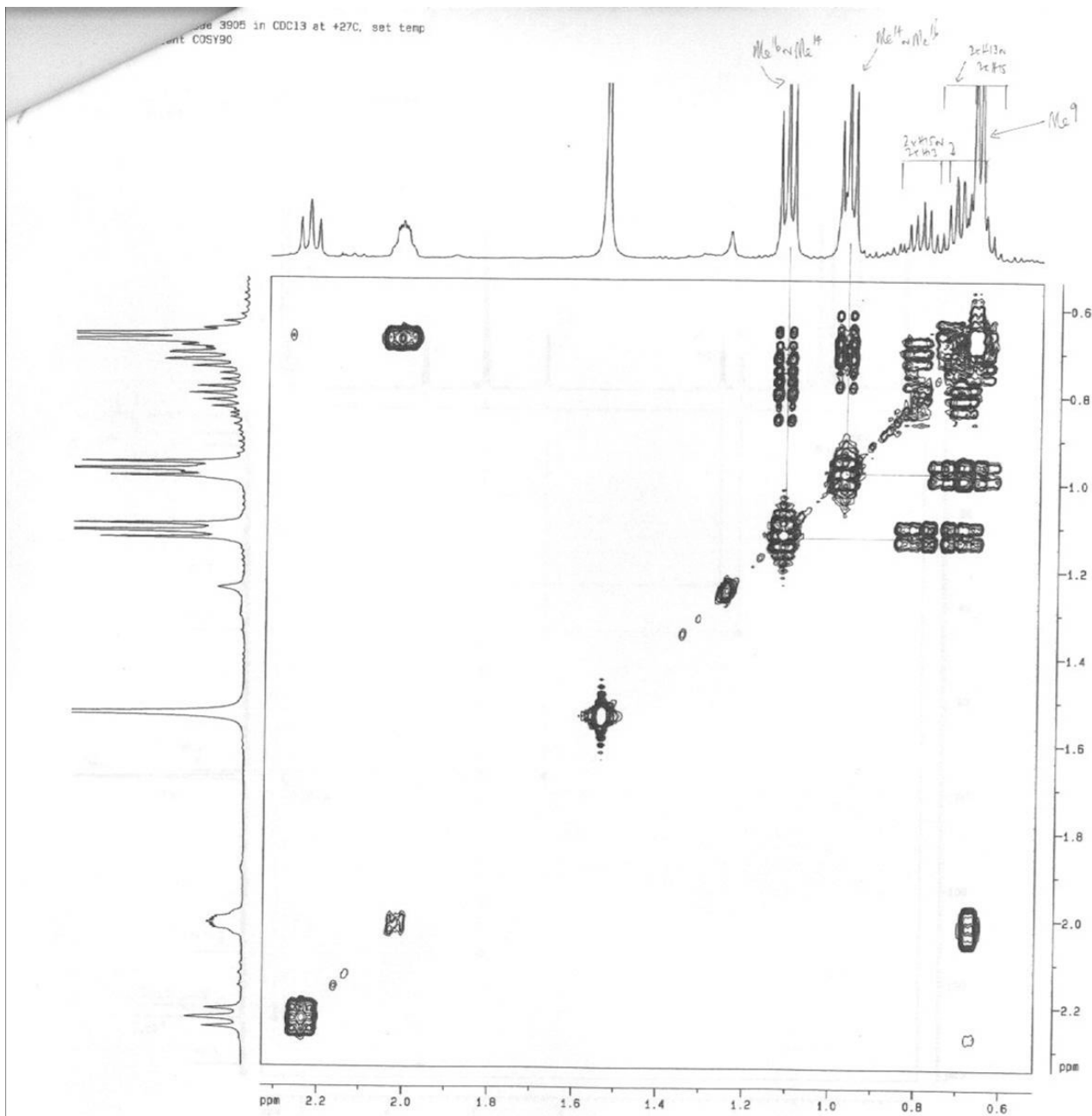
SI 2048
SF 500.1300233 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

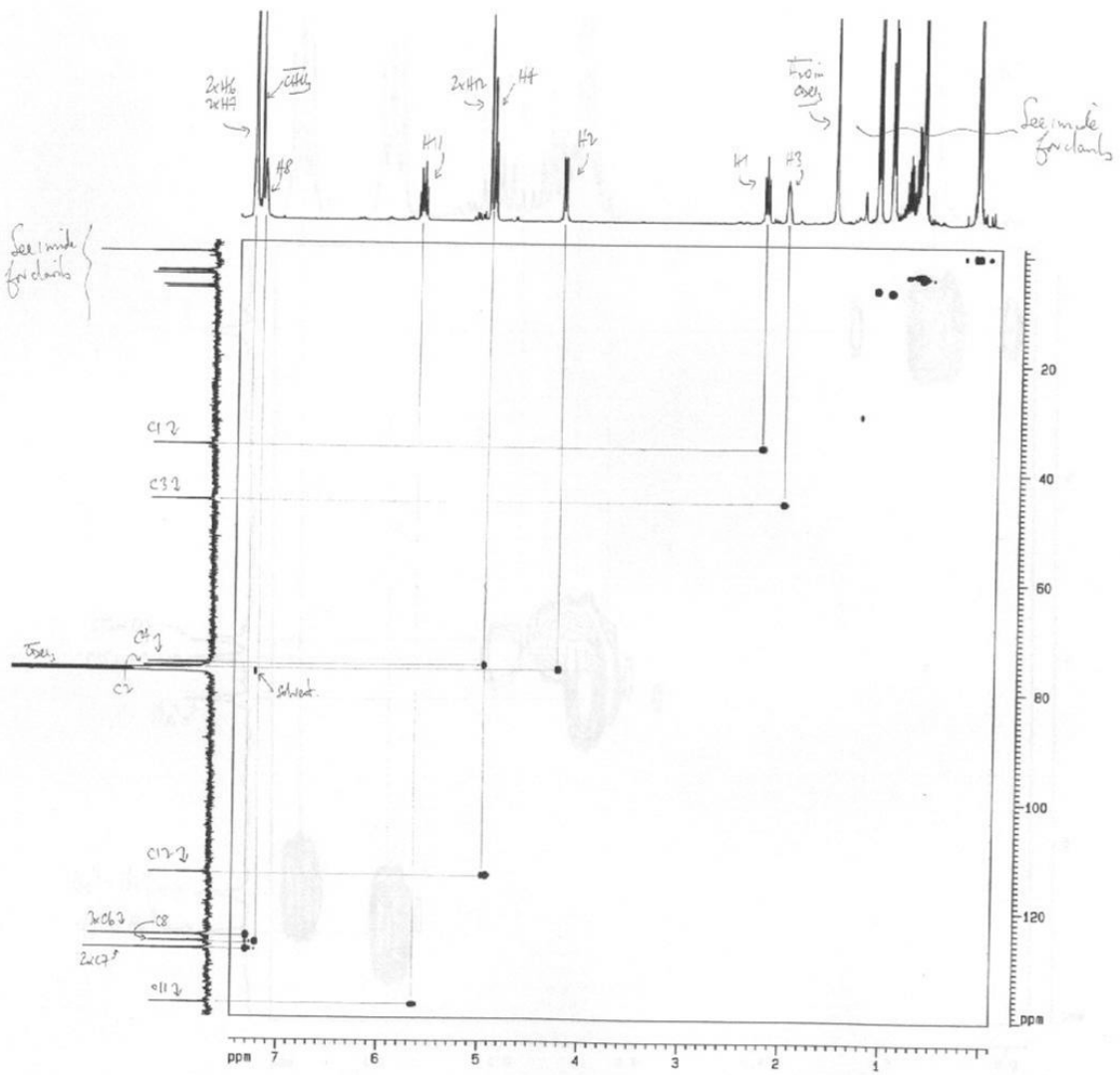
SI 1024
MC2 GF
SF 500.1300233 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters

CX2 20.00 cm
CX1 20.00 cm
F2PL0 7.450 ppm
F2L0 3726.06 Hz
F2PHI -0.049 ppm
F2PHI -24.42 Hz
F1PL0 7.454 ppm
F1L0 3728.19 Hz
F1PHI -0.036 ppm
F1PHI -18.11 Hz
F2PPMCM 0.37485 ppm/cm
F2HZCM 187.52527 Hz/cm
F1PPMCM 0.37453 ppm/cm
F1HZCM 187.31480 Hz/cm



Peter Jarvis Barcode 3905 in CDCl3 at +27C, set temp
dix500, Gradient HSGC



```

Current Data Parameters
NAME      11209j10
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20050705
Time     17.12
INSTRUM  spect
PROBHD   5 mm TBI H/C
PULPROG  invgpgp
TD        2048
SOLVENT  CDCl3
NS        8
DS        18
SFO1     4310.345 Hz
FIDRES   2.104681 Hz
AQ        0.2376180 sec
RG        32788
SWH       118.000 usec
DE        0.50 usec
TE        300.2 K
CMST2    1.45.000000
D0        0.0000000 sec
D1        2.0000000 sec
S4        0.00172414 sec
d11       0.0300000 sec
d15       0.0000000 sec
d16       0.0010000 sec
d20       0.0010000 sec
d21       0.00081714 sec
IND       0.00001140 sec

----- CHANNEL f1 -----
NUC1      1H
P1        10.00 usec
pR        21.20 usec
PL1       1.00 dB
SFO1     500.1319887 MHz

----- CHANNEL f2 -----
CPDPRG2   gpgp
NUC2      13C
P2        12.00 usec
pR        24.00 usec
PL2       78.00 usec
PL3       -1.00 dB
PL13      15.00 dB
SFO2     125.7687889 MHz

----- GRADIENT CHANNEL -----
GPMAX1    SINE.100
GPMAX2    SINE.100
GPMAX3    SINE.100
GPI1      0.00 %
GPI2      0.00 %
GPI3      0.00 %
GPI4      0.00 %
GPI5      0.00 %
GPI6      80.00 %
GPI7      30.00 %
GPI8      20.10 %
P16       1000.00 usec

F1 - Acquisition parameters
NOI        4
TO         512
SFO1     125.7688 MHz
FIDRES   42.831888 Hz
SW        174.369 ppm

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

F1 - Processing parameters
SI         1024
HC2       TWP1
SF        125.7577945 MHz
WDW        SINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CQ2       17.00 cm
CQ1       17.00 cm
F1PLD     7.483 ppm
F2C1     3732.40 Hz
F2PC1    -0.184 ppm
F2HC1    -81.78 Hz
F1PLD    140.324 ppm
F1LD     1740.84 Hz
F1PC1    -1.031 ppm
F1HC1    -180.86 Hz
F2PCMCN  0.4408 ppm/cm
F2HCMCN  22.5980 Hz/cm
F1PCMCN  0.34442 ppm/cm
F1HCMCN  1040.37634 Hz/cm

```

Peter Jervis Barcode 3905 in COC13 at +27C, set temp
dpx500, Gradient HSGC

[See coupling 11/15, 14/13 pairs]

