

Name: _____

Chem 203
December 20, 2014

Final Exam Part II
Problem 2 of 3 (30 points)

Select and submit TWO OUT OF THE THREE PROBLEMS FROM PART II for grading.
Do not submit three problems.

If you wish to unstaple the pages, please initial each page.

Books, notes, lecture videos, calculators, rulers, and laptop computers are permitted as is wireless (or wired) internet access and appropriate software (e.g, Pymol, Macromodel/Maestro, Excel, NMRPrediction, Chemdraw, ElComp, MolE, etc.). Catalogs of molecular structures (e.g., the Aldrich catalog, the Merck Index, etc.) or databases of molecular structures (such as wireless access to SciFinder Scholar, the Sigma-Aldrich website, etc.) are NOT PERMITTED. USE OF SUCH ITEMS CONSTITUTES ACADEMIC DISHONESTY, WILL RESULT IN A FAILING GRADE (F) IN THE CLASS, AND MAY RESULT IN EXPULSION FROM THE Ph.D. PROGRAM.

The following spectral data are provided for a compound with a molecular formula $C_{18}H_{21}NO_4$: IR (KBr pellet), 500 MHz 1H NMR, 125.8 MHz ^{13}C NMR, DEPT-90, DEPT-135, COSY, TOCSY (60 ms mixing time), HMQC, HMBC, HSQC-TOCSY spectra with increasing mixing times (5, 10, 25, and 50 ms), and NOESY. All NMR spectra were measured in $CDCl_3$ solution.

Using these data, determine the structure of the compound and assign all of the 1H and ^{13}C resonances to their respective atoms in the structure. Make sure to determine the stereochemistry of the compound and to clearly assign 1H resonances to the appropriate diastereotopic protons.

MAKE SURE TO COMPLETELY ANSWER THE QUESTIONS **a-f** ON PAGES 2-4.

a. Write the structure of the molecule.

If there are any aspects of your structure that don't seem to make sense, please feel free to comment on them below:

b. Build an energy-minimized molecular model of the molecule using PyMOL and the "clean" function. Use the side menu (S) to show each molecule as sticks (Show-sticks). Use the pulldown menu to display each molecule in maximum quality with (Display-Quality-Maximum Quality). Save the .pse files as Problem_II.2.pse. E-mail the .pse files to me (jsnowick@uci.edu).

Alternatively, you may build an energy-minimized model of the correct diastereomer using Maestro/MacroModel with the MMFF force field. E-mail me (jsnowick@uci.edu) the -out.maegz file associated with that model.

c. Tabulate the shifts of the ^1H resonances (a-q) in order of descending chemical shift. Provide an appropriate name for each multiplet (e.g., s, d, t, q, quintet, sextet, septet, octet, dd, ddd, dt, td, dddd, tt, qd, dq, etc.) and list its coupling constants in appropriate order; also list its integral [e.g., a: 3.30 (t, $J = 7.3$ Hz, 3 H)]. If a resonance is not described by a well-defined multiplet, name it as m and give its range [e.g., c: 4.43-4.37 (m, 2 H)].

a:

b:

c:

d:

e:

f:

g:

h:

i:

j:

k:

l:

m:

n:

o:

p:

q:

d. Redraw the structure of the molecule below and write the letter (a-q) of each ^1H resonance next to the proton or protons to which it corresponds. If the assignment of a particular resonance is uncertain, please indicate this.

e. Tabulate the shifts of the ^{13}C resonances (1-18) in order of descending chemical shift. Make sure to report the chemical shift of each carbon resonances to the nearest tenth of ppm, unless two have the same value, in which case you may report it to the nearest hundredth.

1:

2:

3:

4:

5:

6:

7:

8:

9:

10:

11:

12:

13:

14:

15:

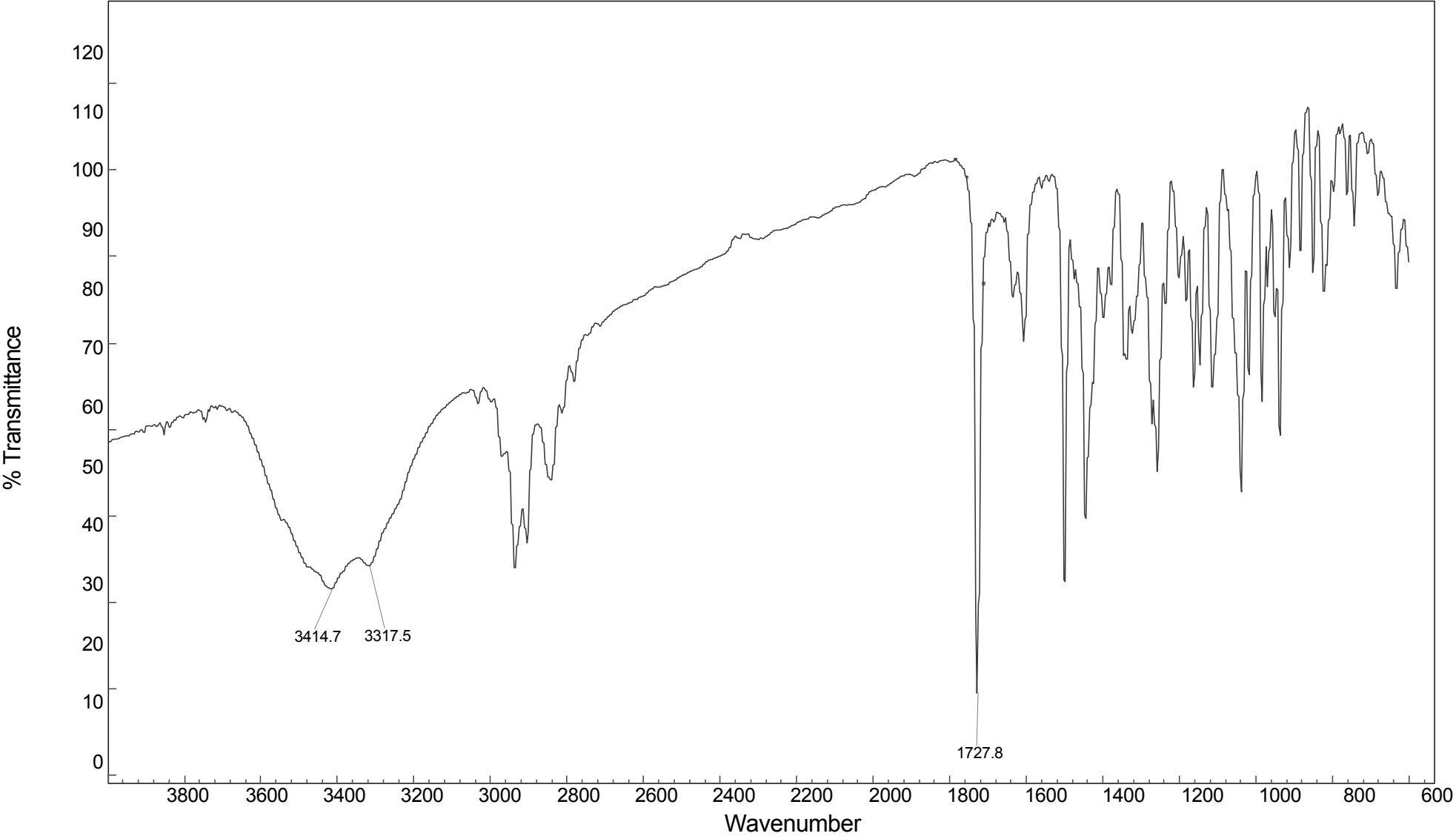
16:

17:

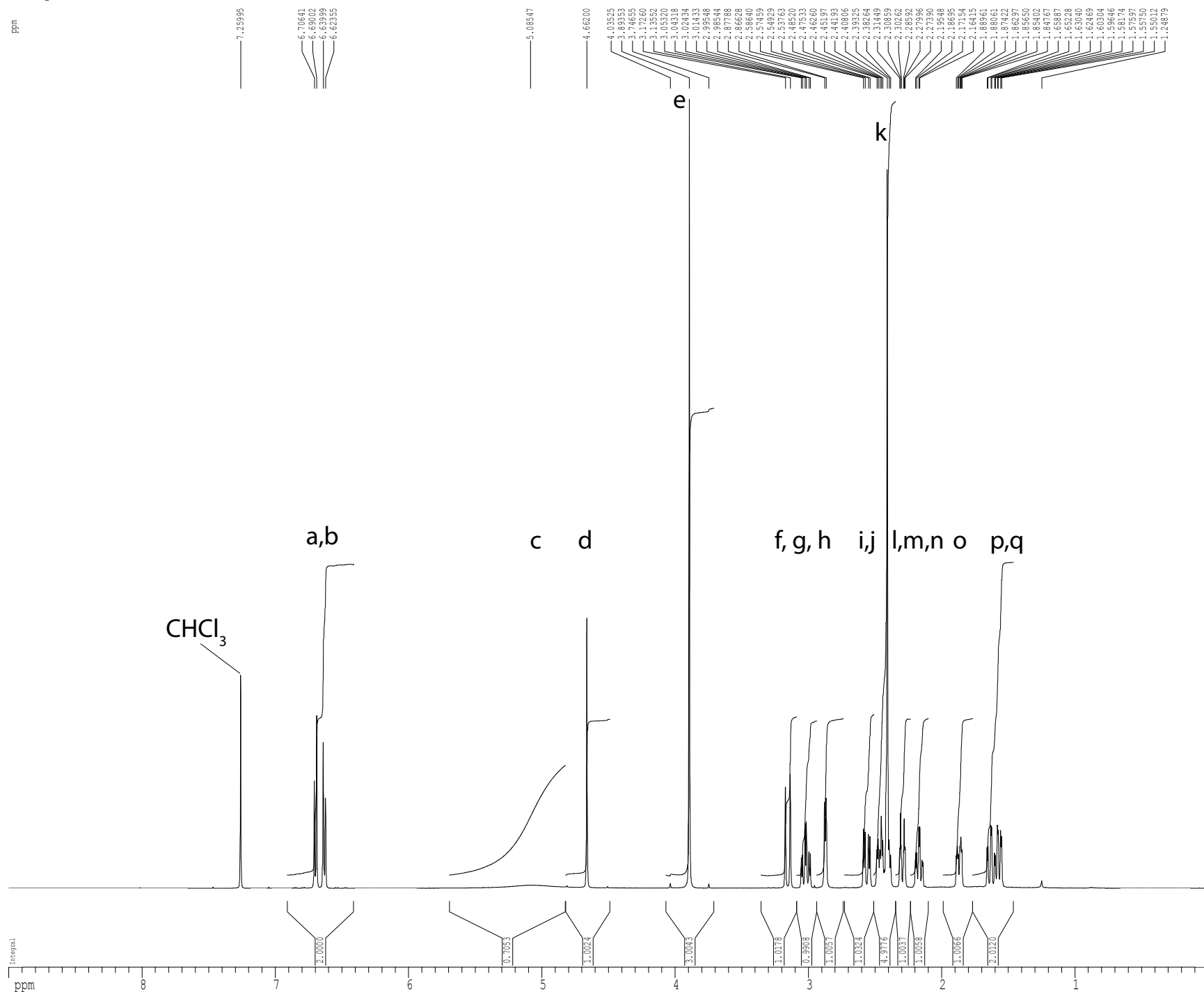
18:

f. Redraw the structure of the molecule below and write the number (1-18) of each ^{13}C resonance next to the carbon or carbons to which it corresponds. If the assignment of particular resonance is uncertain, please indicate this.

Varian Resolutions Pro



¹H spectrum



Current Data Parameters

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NAME	partII_C.h
EXPNO	22
PROCNO	1

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PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	64
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	5.7
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
DL	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

F2 - Processing parameters

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SF	500.2200340 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

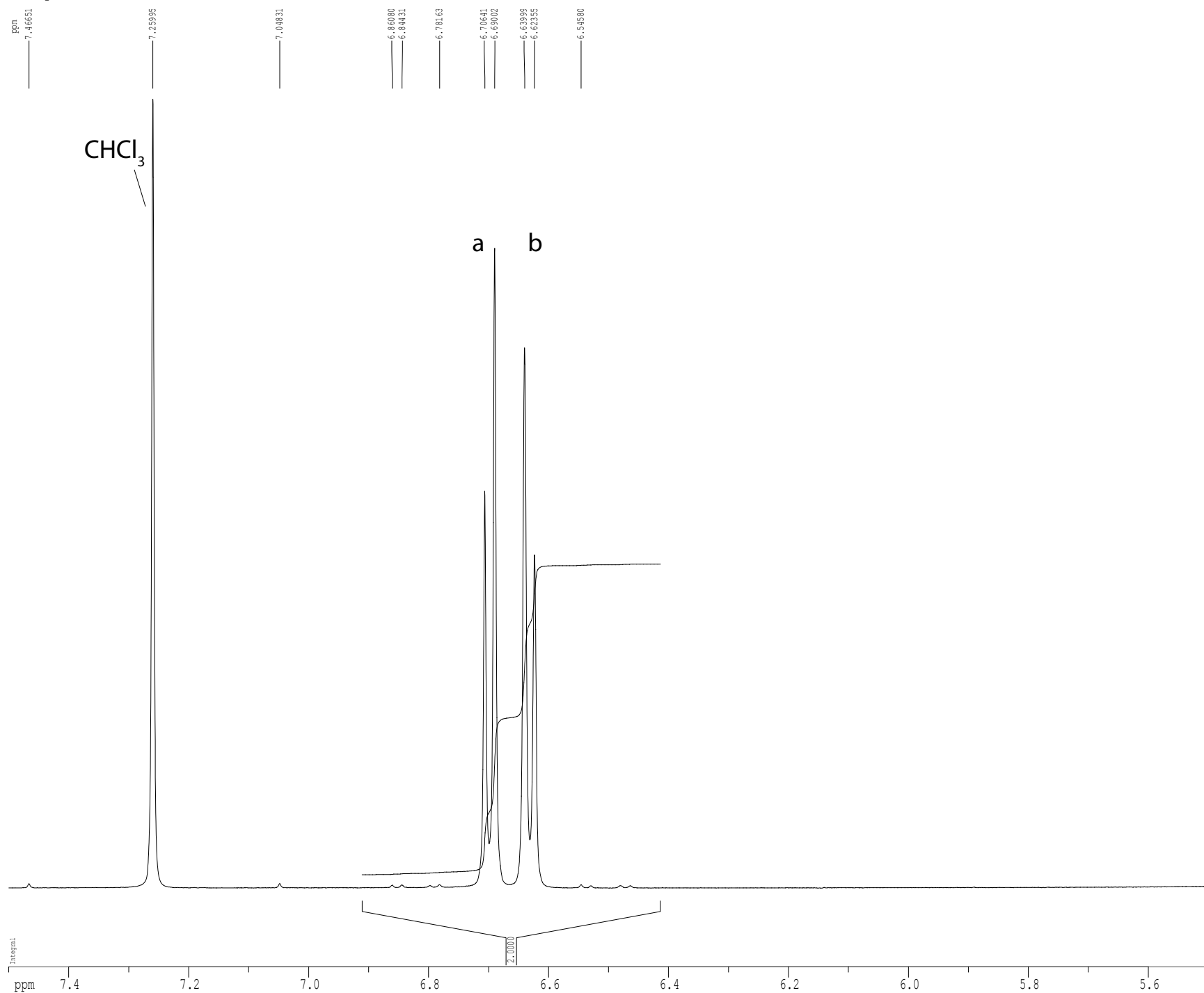
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F2P	0.000 ppm
F2	0.00 Hz
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HZCM	197.45528 Hz/cm

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#	ADDRESS	FREQUENCY		INTENSITY
		[Hz]	[PPM]	
1	31426.2	3631.570	7.2599	4.05
2	33690.8	3354.681	6.7064	2.04
3	33757.9	3346.480	6.6900	3.29
4	33962.6	3321.453	6.6400	2.78
5	34029.8	3313.231	6.6235	1.71
6	42055.0	2332.025	4.6620	5.13
7	45199.0	1947.622	3.8935	15.00
8	48148.5	1587.000	3.1726	1.92
9	48300.2	1568.451	3.1355	2.17
10	48637.0	1527.272	3.0532	0.58
11	48678.0	1522.261	3.0432	0.63
12	48755.1	1512.836	3.0243	1.25
13	48796.1	1507.828	3.0143	1.27
14	48873.2	1498.398	2.9955	0.70
15	48914.2	1493.378	2.9854	0.66
16	49354.3	1439.573	2.8779	1.64
17	49401.8	1433.771	2.8663	1.72
18	50546.8	1293.769	2.5864	1.13
19	50595.1	1287.863	2.5746	1.07
20	50698.6	1275.208	2.5493	1.03
21	50746.3	1269.374	2.5376	0.99
22	50960.9	1243.144	2.4852	0.73
23	51001.2	1238.210	2.4753	0.94
24	51053.3	1231.840	2.4626	0.74
25	51096.8	1226.525	2.4520	1.38
26	51137.9	1221.501	2.4419	0.92
27	51276.5	1204.559	2.4081	13.67
28	51337.0	1197.152	2.3933	0.93
29	51380.4	1191.846	2.3826	0.63
30	51659.3	1157.754	2.3145	0.77
31	51683.4	1154.800	2.3086	1.43
32	51707.8	1151.816	2.3026	0.82
33	51776.2	1143.462	2.2859	0.73
34	51800.6	1140.479	2.2800	1.33
35	51825.3	1137.450	2.2739	0.79
36	52146.2	1098.224	2.1955	0.67
37	52181.1	1093.955	2.1869	0.68
38	52244.1	1086.246	2.1715	1.16
39	52274.3	1082.551	2.1642	1.18
40	52342.0	1074.273	2.1476	0.54
41	52367.4	1071.171	2.1414	0.50
42	53397.5	945.222	1.8896	0.62
43	53434.4	940.719	1.8806	0.80
44	53460.5	937.521	1.8742	0.65
45	53506.5	931.896	1.8630	0.81
46	53533.0	928.658	1.8565	0.98
47	53543.1	927.419	1.8540	0.97
48	53569.2	924.239	1.8477	0.74
49	54341.6	829.802	1.6589	0.78
50	54368.5	826.505	1.6523	0.81

51	54458.0	815.560	1.6304	1.20
52	54481.4	812.704	1.6247	1.18
53	54570.0	801.873	1.6030	0.67
54	54596.9	798.580	1.5965	0.62
55	54657.1	791.219	1.5817	1.20
56	54680.7	788.332	1.5760	1.11
57	54756.3	779.094	1.5575	1.10
58	54786.5	775.402	1.5501	0.99

¹H spectrum



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Current Data Parameters
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NAME          partII_C.h
EXPNO         22
PROCNO        1

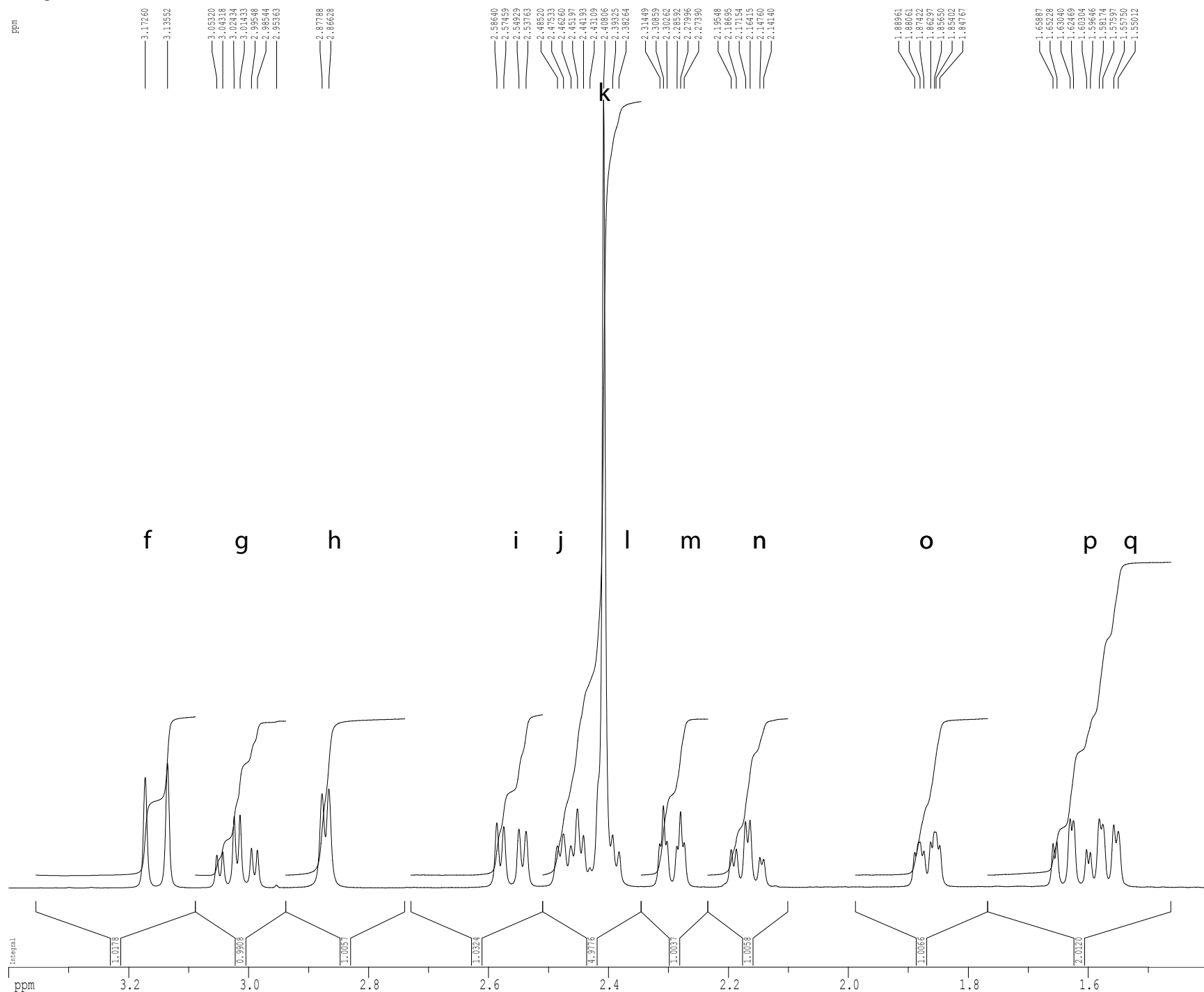
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PULPROG       zg30
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SOLVENT       CDCl3
NS            64
DS            2
SWH           8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0998774 sec
RG            5.7
DW            62.400 usec
DE            6.00 usec
TE            298.0 K
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MCREST        0.00000000 sec
MCWRK         0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            7.50 usec
PL1           1.60 dB
SF01          500.2235015 MHz

F2 - Processing parameters
SI            65536
SF            500.2200340 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

1D NMR plot parameters
CX            22.80 cm
CY            15.00 cm
F1P           7.500 ppm
F1            3751.65 Hz
F2P           5.500 ppm
F2            2751.21 Hz
PPMCM         0.08772 ppm/cm
HZCM          43.87895 Hz/cm
    
```

¹H spectrum



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Current Data Parameters
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NAME          partII_C.h
EXPNO         22
PROCNO        1

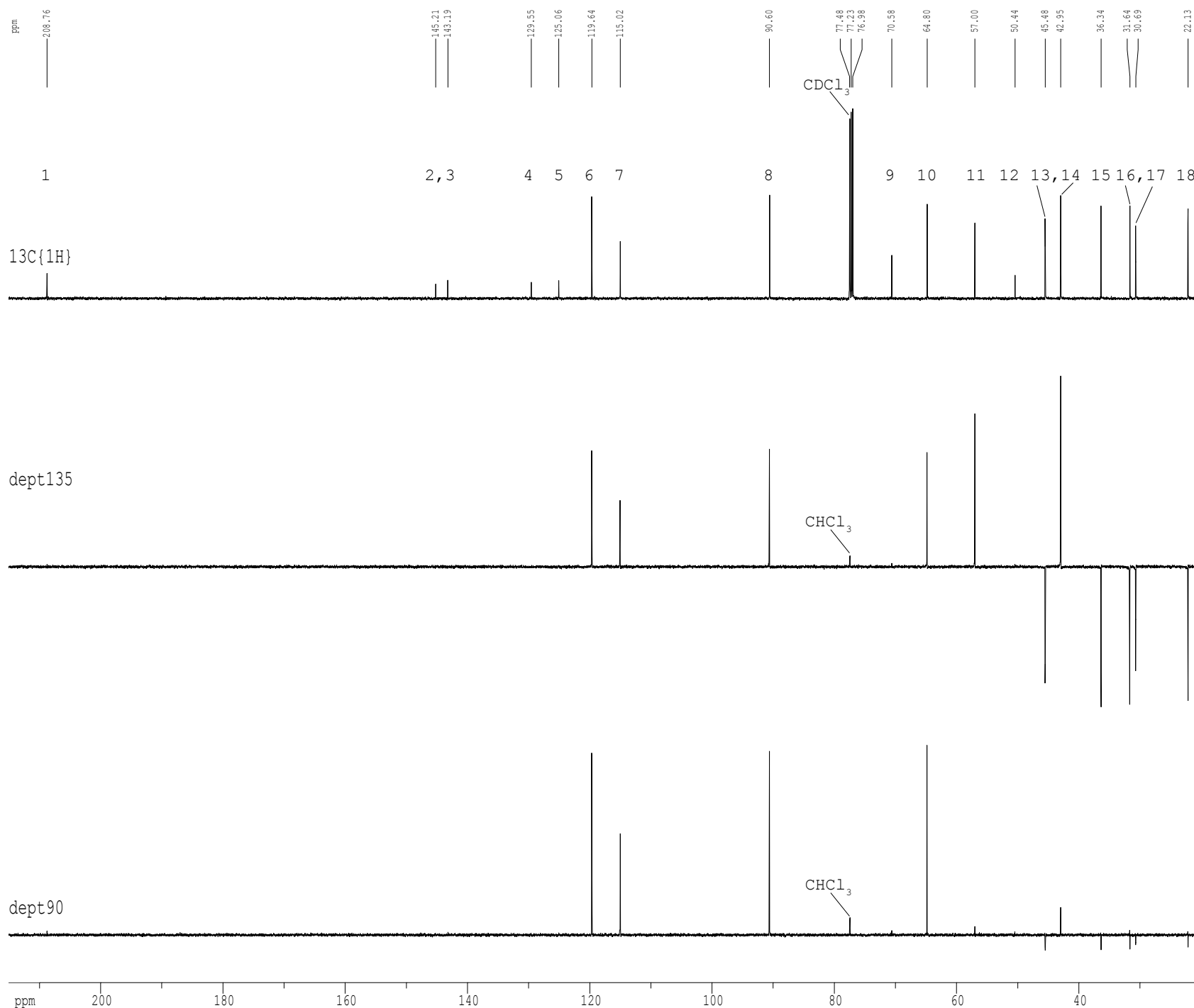
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Date_         20141216
Time          22.07
INSTRUM       cryo500
PROBHD        5 mm CPTCI 1H-
PULPROG       zg30
TD            81728
SOLVENT       CDCl3
NS            64
DS            2
SWH           8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0998774 sec
RG            5.7
DW            62.400 usec
DE            6.00 usec
TE            298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

===== CHANNEL f1 =====
NUC1           1H
P1             7.50 usec
PL1           1.60 dB
SF01          500.2235015 MHz

F2 - Processing parameters
SI            65536
SF            500.2200340 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

1D NMR plot parameters
CX            22.80 cm
CY            15.00 cm
F1P           3.400 ppm
F1            1700.75 Hz
F2P           1.400 ppm
F2            700.31 Hz
PPMCM         0.08772 ppm/cm
HZCM          43.87895 Hz/cm
    
```

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
 USER nm14t
 NAME partII_C_h
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141215
 Time_ 22.11
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 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchopg30gp.prd
 TD 65536
 SOLVENT CDCl₃
 NS 1024
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 7298.2
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.25000000 sec
 d11 0.03000000 sec
 D16 0.00020000 sec
 d17 0.00019600 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec
 P2 33.10 usec

===== CHANNEL f1 =====
 NUC1 13C
 P1 16.55 usec
 P11 500.00 usec
 P12 2000.00 usec
 PL0 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SP1 2.70 dB
 SP2 2.70 dB
 SPNAM1 Crp60,0.5,20.1
 SPNAM2 Crp60comp,4
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 SPOFF2 0.00 Hz

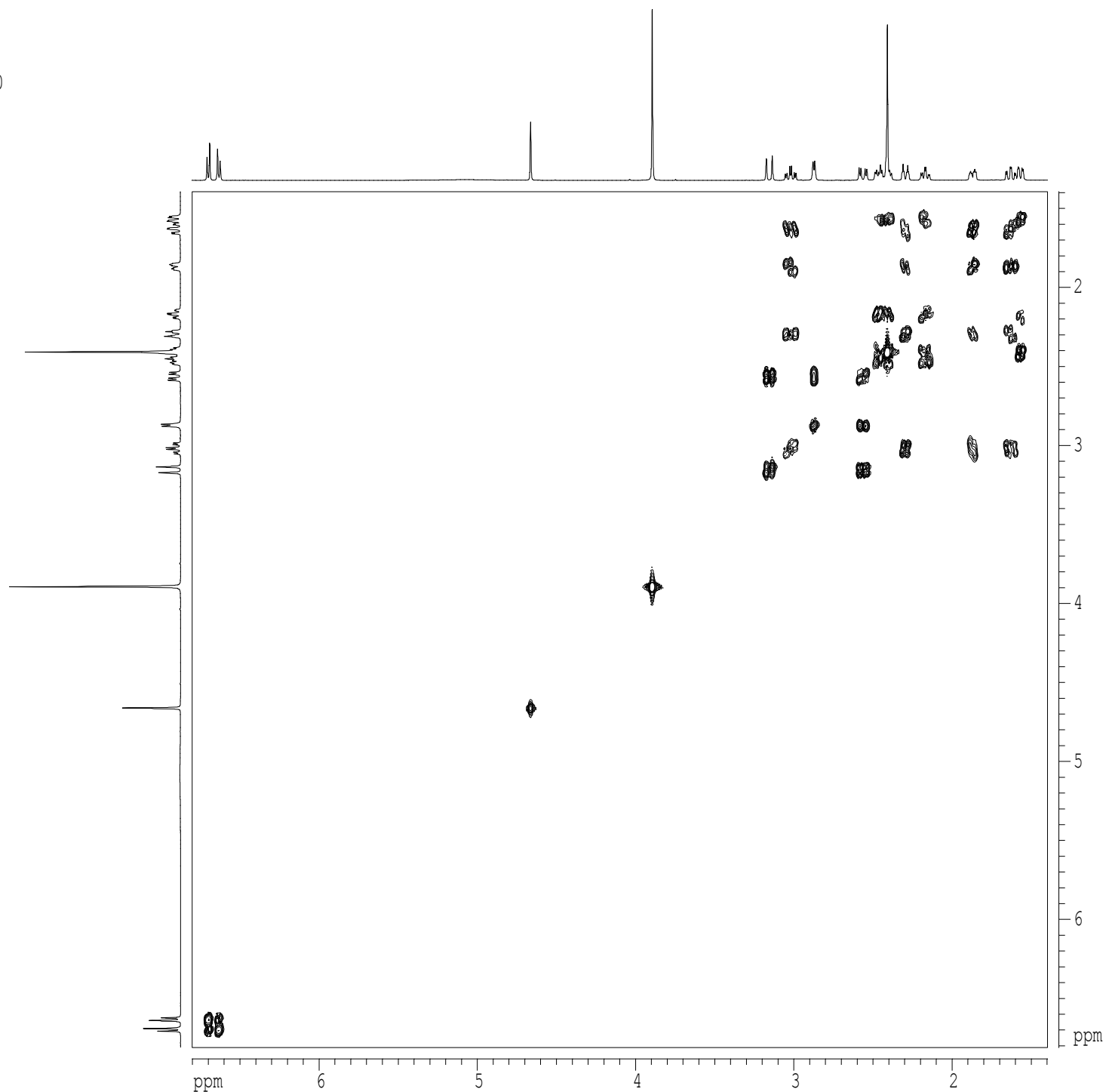
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 NUC2 1H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.50 dB
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SINE.100
 GPNAM2 SINE.100
 GFX1 0.00 %
 GFX2 0.00 %
 GPT1 0.00 %
 GPT2 0.00 %
 GP21 30.00 %
 GP22 50.00 %
 p15 500.00 usec
 p16 1000.00 usec

F2 - Processing parameters
 SI 65536
 SF 125.7804012 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 9.56 cm
 F1P 215.000 ppm
 F1 27042.79 Hz
 F2P 15.000 ppm
 F2 1886.71 Hz
 PPMCM 8.77193 ppm/cm
 HCM 1103.33691 Hz/cm

gcosy60



Current Data Parameters
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NAME partII_C_h
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
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Time 23.21
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PROBHD 5 mm CPTCI 1H-
PULPROG cosygp60.prd
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 5482.456 Hz
FIDRES 2.676980 Hz
AQ 0.1868276 sec
RG 5
DW 91.200 usec
DE 6.00 usec
TE 298.0 K
d0 0.00000300 sec
D1 1.00000000 sec
d13 0.00000300 sec
D16 0.00020000 sec
IN0 0.00018240 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2227500 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 17.00 %
GPZ2 17.00 %
P16 1000.00 usec

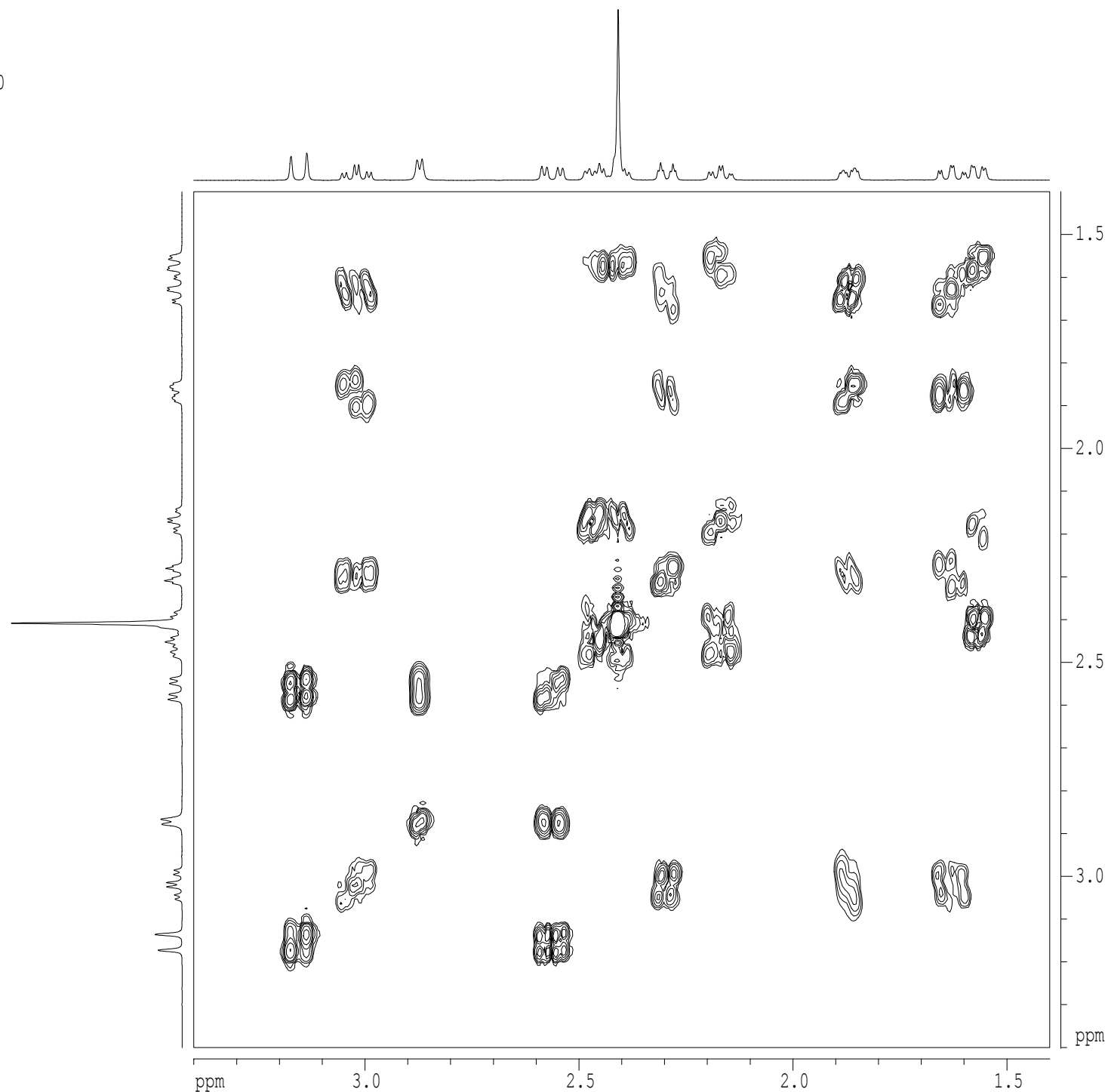
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FIDRES 21.415844 Hz
SW 10.960 ppm
FnMODE undefined

F2 - Processing parameters
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SF 500.2200352 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.2200340 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 6.803 ppm
F2LO 3402.75 Hz
F2PHI 1.397 ppm
F2HI 699.00 Hz
F1PLO 6.810 ppm
F1LO 3406.63 Hz
F1PHI 1.394 ppm
F1HI 697.53 Hz
F2PPMCM 0.36034 ppm/cm
F2HZCM 180.25002 Hz/cm
F1PPMCM 0.36106 ppm/cm
F1HZCM 180.60695 Hz/cm

gcosy60



Current Data Parameters
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NAME partII_C_h
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141215
Time 23.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG cosygp60.prd
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 5482.456 Hz
FIDRES 2.676980 Hz
AQ 0.1868276 sec
RG 5
DW 91.200 usec
DE 6.00 usec
TE 298.0 K
d0 0.00000300 sec
D1 1.00000000 sec
d13 0.00000300 sec
D16 0.00020000 sec
IN0 0.00018240 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2227500 MHz

===== GRADIENT CHANNEL =====
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GPNAM2 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 17.00 %
GPZ2 17.00 %
P16 1000.00 usec

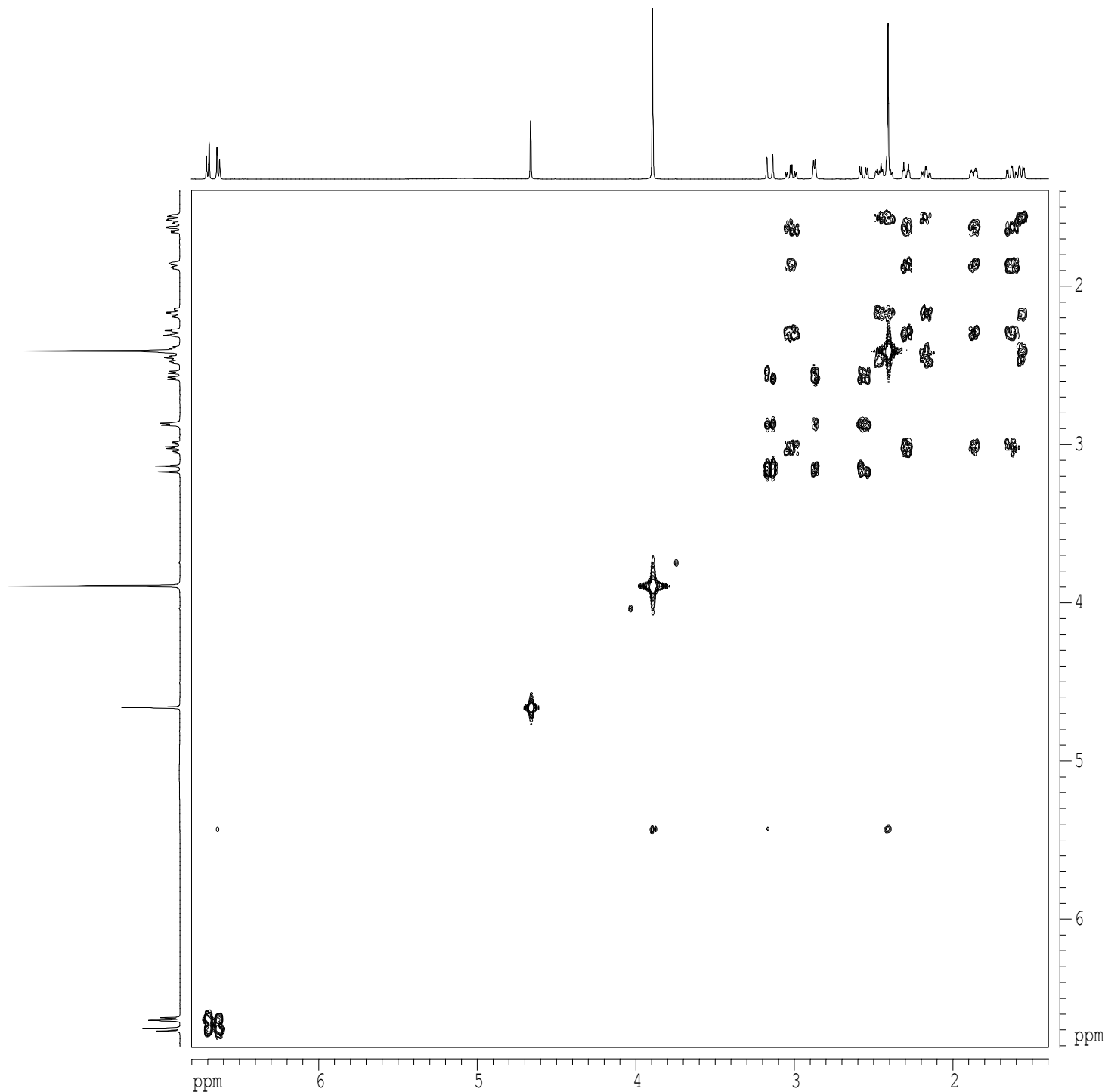
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FIDRES 21.415844 Hz
SW 10.960 ppm
FnMODE undefined

F2 - Processing parameters
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SF 500.2200352 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.2200340 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 3.400 ppm
F2LO 1700.75 Hz
F2PHI 1.400 ppm
F2HI 700.31 Hz
F1PLO 3.400 ppm
F1LO 1700.75 Hz
F1PHI 1.400 ppm
F1HI 700.31 Hz
F2PPMCM 0.13333 ppm/cm
F2HZCM 66.69601 Hz/cm
F1PPMCM 0.13333 ppm/cm
F1HZCM 66.69601 Hz/cm

gtocsy



```
Current Data Parameters
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NAME      partII_C_h
EXPNO     6
PROCNO    1

F2 - Acquisition Parameters
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Time      0.03
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PULPROG   mlevpg mo
TD        2048
SOLVENT   CDCl3
NS         8
DS        16
SWH        5482.456 Hz
FIDRES     2.676980 Hz
AQ         0.1868276 sec
RG         16
DW         91.200 usec
DE         6.00 usec
TE         298.0 K
d0         0.00000300 sec
d1         2.00000000 sec
d9         0.06000000 sec
d12        0.00002000 sec
d16        0.00020000 sec
FACTOR1    4
IN0        0.00018240 sec
l1         24
SCALEF     6

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p5         23.34 usec
P6         35.00 usec
p7         70.00 usec
P17        2500.00 usec
PL1        1.60 dB
PL10       15.20 dB
SFO1       500.2227500 MHz

===== GRADIENT CHANNEL =====
GPNAM1     sine.100
GPNAM2     sine.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPE1       10.00 %
GPE2       10.00 %
F16        1000.00 usec

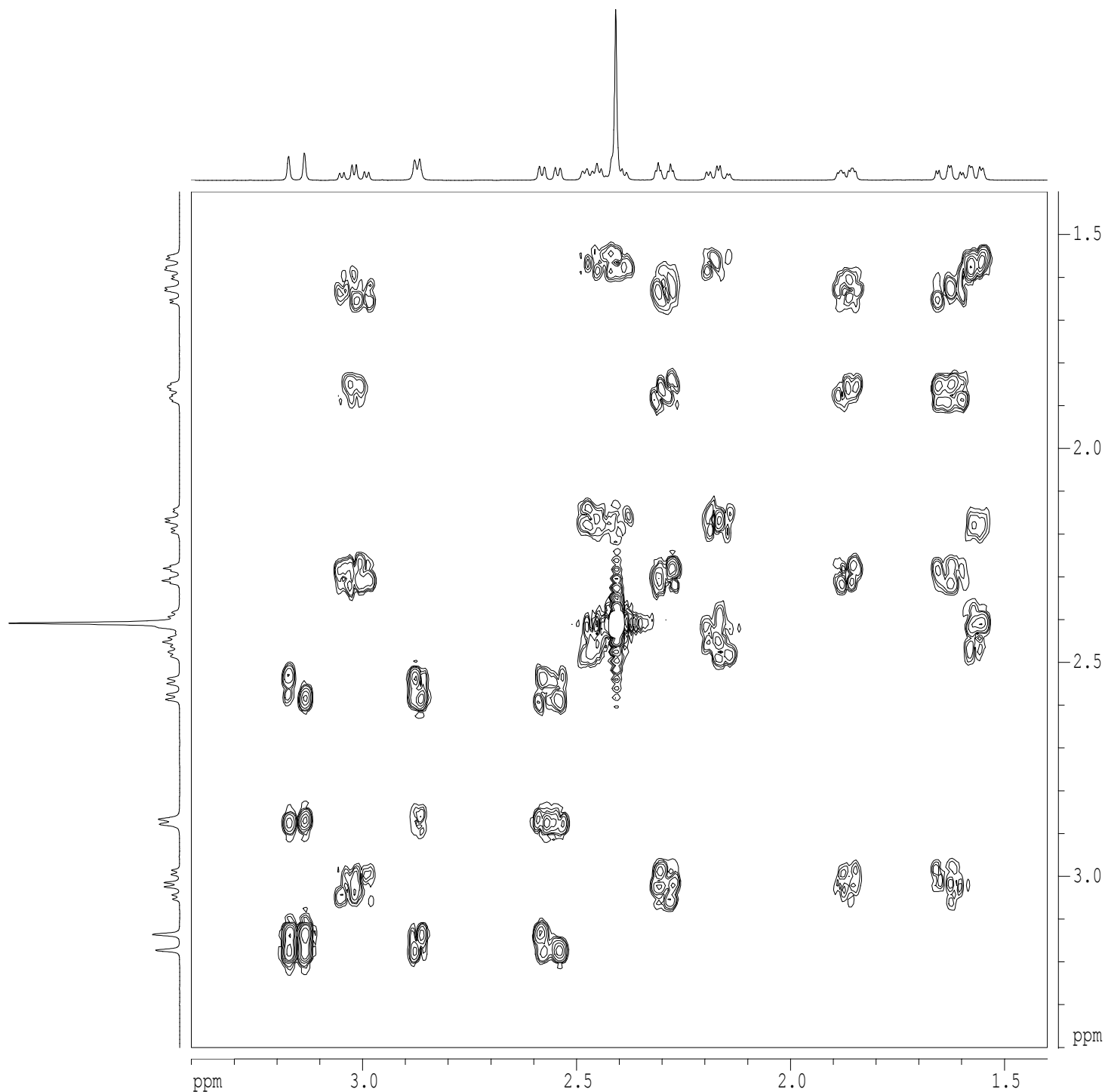
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SW         10.960 ppm
FnMODE     undefined

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SF         500.2200352 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        QF
SF         500.2200340 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        15.00 cm
CX1        15.00 cm
F2PLO      6.803 ppm
F2LO       3402.75 Hz
F2EHI      1.397 ppm
F2EHL      699.00 Hz
F1PLO      6.810 ppm
F1LO       3406.63 Hz
F1EHI      1.394 ppm
F1EHL      697.53 Hz
F2PPMCM    0.36034 ppm/cm
F2HZCM     180.25002 Hz/cm
F1PPMCM    0.36106 ppm/cm
F1HZCM     180.60695 Hz/cm
```

gtocsy



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Current Data Parameters
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NAME      partII_C_h
EXPNO     6
PROCNO    1

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Time      0.03
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PROBHD    5 mm CPTCI 1H-
PULPROG   mlevpg mo
TD        2048
SOLVENT   CDCl3
NS        8
DS        16
SWH       5482.456 Hz
FIDRES    2.676980 Hz
AQ        0.1868276 sec
RG        16
DW        91.200 usec
DE        6.00 usec
TE        298.0 K
d0        0.00000300 sec
D1        2.00000000 sec
D9        0.06000000 sec
d12       0.00002000 sec
D16       0.00020000 sec
FACTOR1   4
IN0       0.00018240 sec
l1        24
SCALEF    6

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p5         23.34 usec
P6         35.00 usec
p7         70.00 usec
P17        2500.00 usec
PL1        1.60 dB
PL10       15.20 dB
SFO1       500.2227500 MHz

===== GRADIENT CHANNEL =====
GPNAM1    sine.100
GPNAM2    sine.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GPE1      10.00 %
GPE2      10.00 %
P16       1000.00 usec

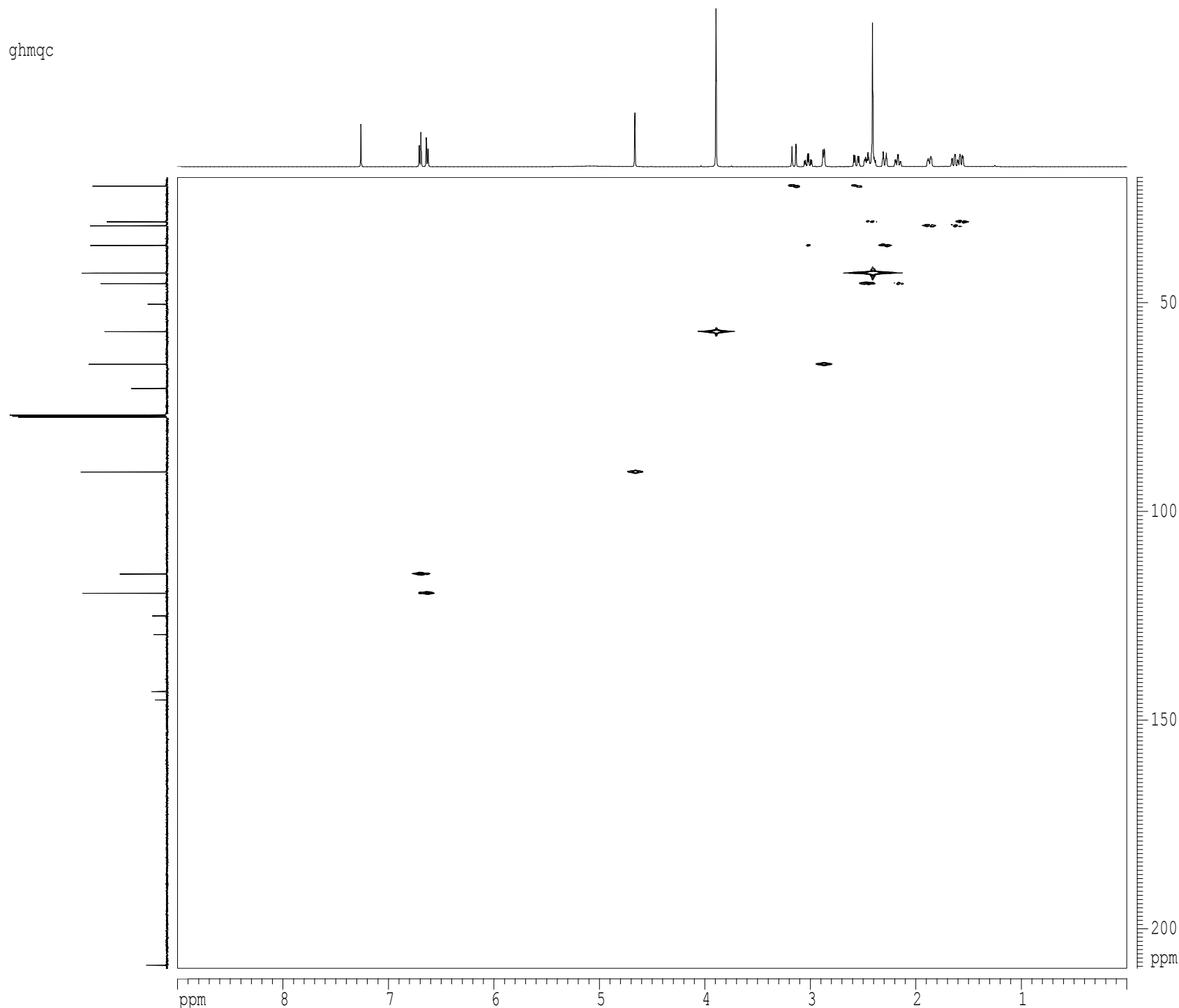
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FIDRES    21.415844 Hz
SW        10.960 ppm
FnMODE    undefined

F2 - Processing parameters
SI        2048
SF        500.2200352 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       QF
SF        500.2200340 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0

2D NMR plot parameters
CX2       15.00 cm
CX1       15.00 cm
F2PLO     3.400 ppm
F2LO      1700.75 Hz
F2PHI     1.400 ppm
F2H1      700.31 Hz
F1PLO     3.400 ppm
F1LO      1700.75 Hz
F1PHI     1.400 ppm
F1H1      700.31 Hz
F2PPMCM   0.13333 ppm/cm
F2HZCM    66.69601 Hz/cm
F1PPMCM   0.13333 ppm/cm
F1HZCM    66.69601 Hz/cm
```

ghmqc



```
Current Data Parameters
USER      nmrl4t
NAME      partII_C_h
EXPNO     7
PROCNO    1

F2 - Acquisition Parameters
Date_     20141216
Time      1.21
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   inv4gp.wu
TD         2048
SOLVENT   CDCl3
NS         8
DS         16
SWH        5482.456 Hz
FIDRES     2.676980 Hz
AQ         0.1868276 sec
RG         11585.2
DW         91.200 usec
DE         6.50 usec
TE         298.0 K
CMST2     145.0000000
d0         0.00000300 sec
d1         1.00000000 sec
d2         0.00344828 sec
d12        0.00002000 sec
d13        0.00000300 sec
d16        0.00020000 sec
d20        0.00242528 sec
IN0        0.00001806 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p2         15.00 usec
PL1        1.60 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
CPDPRG2    gqrp
NUC2       13C
P3         15.50 usec
PCPD2      65.00 usec
PL2        -1.00 dB
PL12       11.30 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPNAM1     sine.100
GPNAM2     sine.100
GPNAM3     sine.100
GPX1        0.00 %
GPX2        0.00 %
GPX3        0.00 %
GPY1        0.00 %
GPY2        0.00 %
GPY3        0.00 %
GPZ1        30.00 %
GPZ2        18.00 %
GPZ3        24.00 %
P16         1000.00 usec

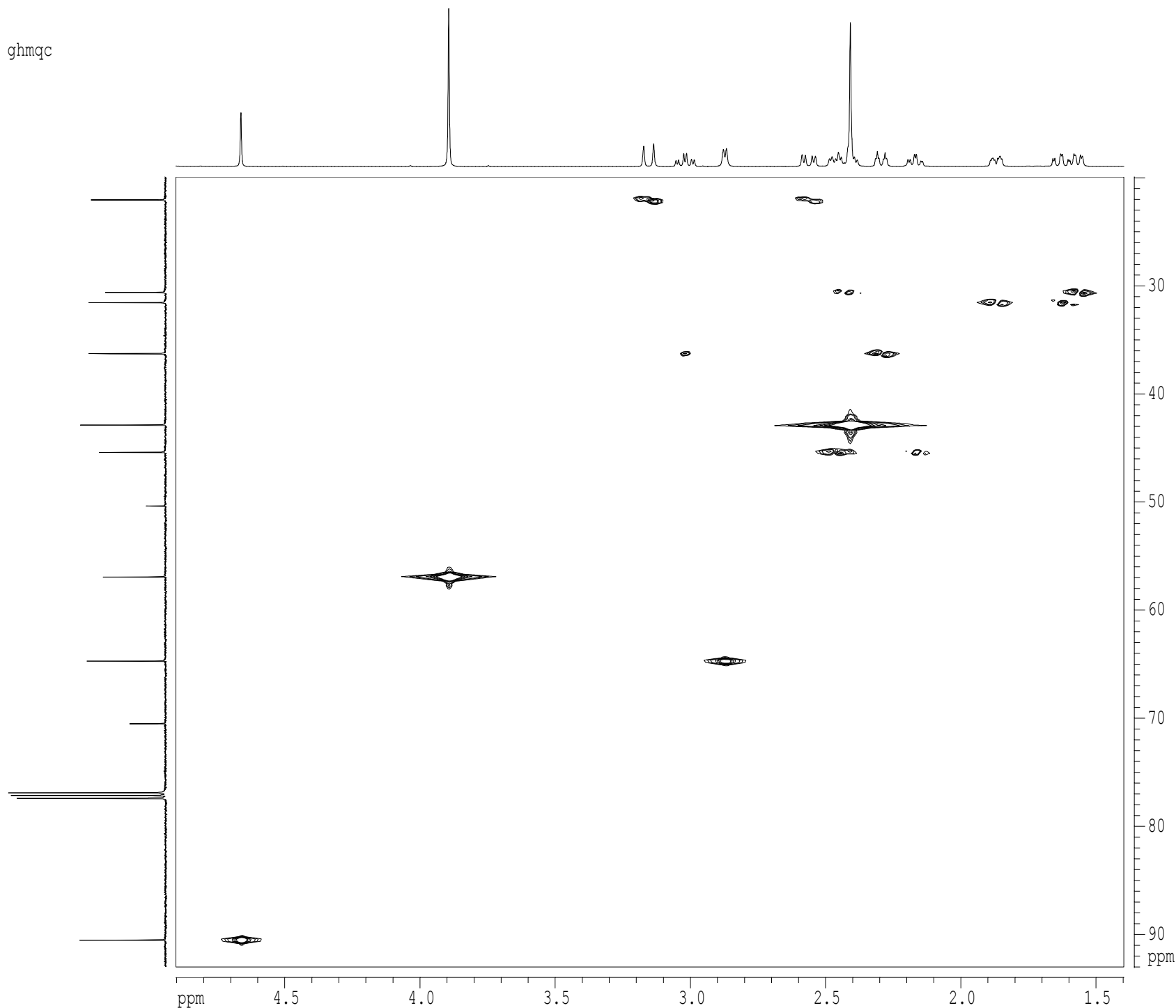
F1 - Acquisition parameters
ND0         2
TD          512
SFO1        125.7929 MHz
FIDRES      54.065742 Hz
SW          220.057 ppm
FnMODE      undefined

F2 - Processing parameters
SI          2048
SF          500.2200352 MHz
WDW         EM
SSB         0
LB          7.00 Hz
GB          0
PC          1.40

F1 - Processing parameters
SI          1024
MC2         QF
SF          125.7804099 MHz
WDW         QSINE
SSB         3
LB          0.00 Hz
GB          0

2D NMR plot parameters
CX2         18.00 cm
CX1         15.00 cm
F2PL0       9.000 ppm
F2LO        4501.98 Hz
F2PHI       0.000 ppm
F2H1        0.000 Hz
F1PL0       209.492 ppm
F1LO        26349.96 Hz
F1PHI       20.000 ppm
F1H1        2515.61 Hz
F2PPMCM     0.50000 ppm/cm
F2HZCM      250.11002 Hz/cm
F1PPMCM     12.63279 ppm/cm
F1HZCM      1588.95715 Hz/cm
```


ghmqc



Current Data Parameters

USER nmrl4t
NAME partII_C_h
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters

Date 20141216
Time 1.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG inv4gp.wu
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 5482.456 Hz
FIDRES 2.676980 Hz
AQ 0.1868276 sec
RG 11585.2
DW 91.200 usec
DE 6.50 usec
TE 298.0 K
CMST2 145.000000
d0 0.00000300 sec
d1 1.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000300 sec
d16 0.00020000 sec
d20 0.00242528 sec
IN0 0.00001806 sec

===== CHANNEL f1 =====

NUC1 1H
P1 7.50 usec
p2 15.00 usec
PL1 1.60 dB
SFO1 500.2227500 MHz

===== CHANNEL f2 =====

CPDPRG2 gqrp
NUC2 13C
P3 15.50 usec
PCPD2 65.00 usec
PL2 -1.00 dB
PL12 11.30 dB
SFO2 125.7929190 MHz

===== GRADIENT CHANNEL =====

GPVAM1 sine.100
GPVAM2 sine.100
GPVAM3 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 30.00 %
GPZ2 18.00 %
GPZ3 24.00 %
P16 1000.00 usec

F1 - Acquisition parameters

ND0 2
TD 512
SFO1 125.7929 MHz
FIDRES 54.065742 Hz
SW 220.057 ppm
FMODE undefined

F2 - Processing parameters

SI 2048
SF 500.2200352 MHz
WDW EM
SSB 0
LB 7.00 Hz
GB 0
PC 1.40

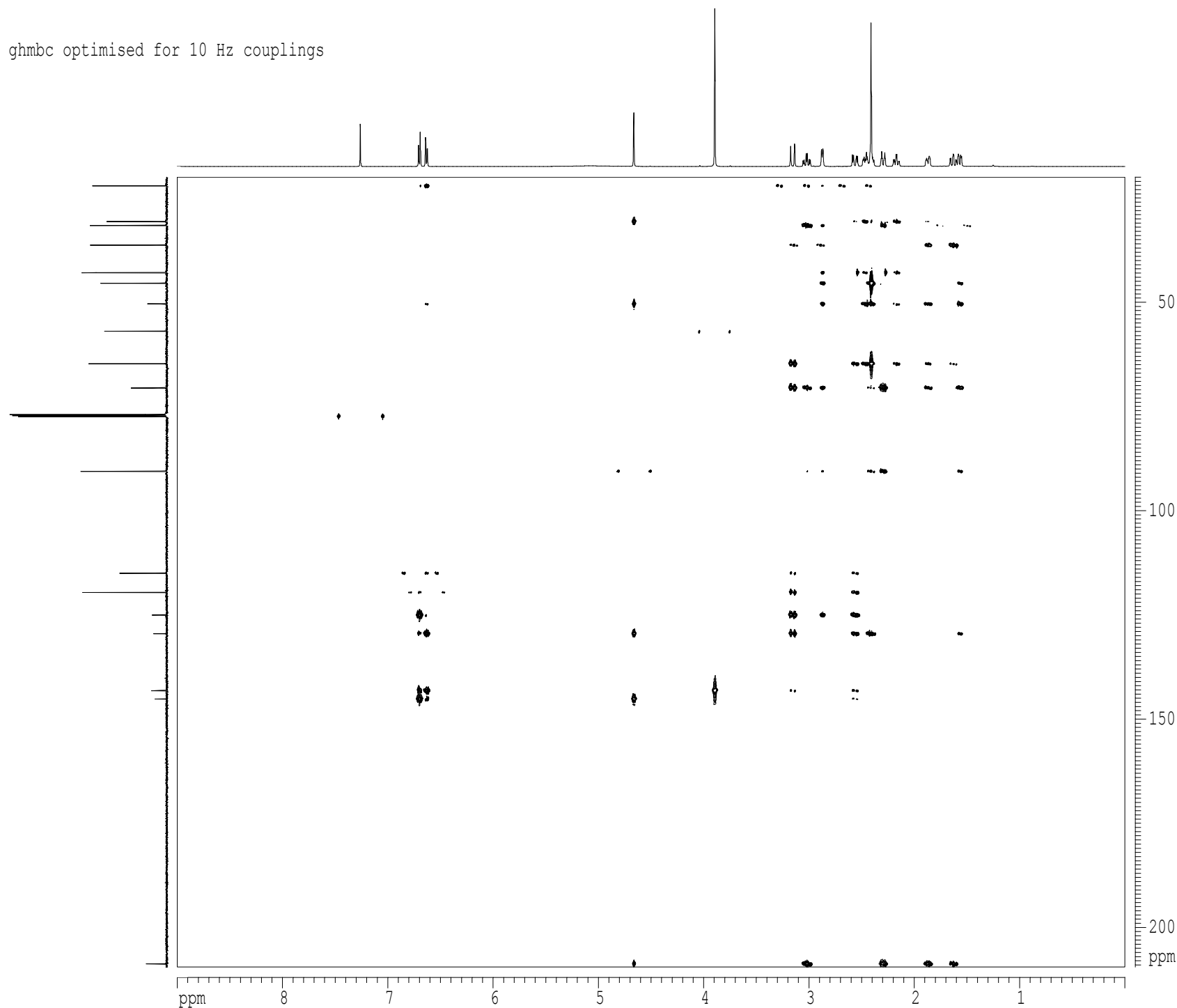
F1 - Processing parameters

SI 1024
MC2 QF
SF 125.7804099 MHz
WDW QSINE
SSB 3
LB 0.00 Hz
GB 0

2D NMR plot parameters

CX2 18.00 cm
CX1 15.00 cm
F2PLO 4.903 ppm
F2LO 2452.42 Hz
F2PHI 1.397 ppm
F2H1 699.00 Hz
F1PLO 93.005 ppm
F1LO 11698.15 Hz
F1PHI 19.931 ppm
F1H1 2506.97 Hz
F2PPMCM 0.19474 ppm/cm
F2HZCM 97.41235 Hz/cm
F1PPMCM 4.87155 ppm/cm
F1HZCM 612.74512 Hz/cm

ghmbc optimised for 10 Hz couplings



```
Current Data Parameters
USER      nmr14t
NAME      partII_C_h
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20141216
Time      2.45
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   ghmbc.wu
TD         4096
SOLVENT   CDCl3
NS         8
DS         16
SWH        5482.456 Hz
FIDRES     1.338490 Hz
AQ         0.3736052 sec
RG         14596.5
DW         91.200 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.000000
d0         0.00000300 sec
D1         1.25000000 sec
d2         0.00344828 sec
D6         0.05000000 sec
d13        0.00000300 sec
D16        0.00020000 sec
IN0        0.0001806 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
P2         15.00 usec
PL1        1.60 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
NUC2       13C
P3         15.50 usec
PL2        -1.00 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPNAM1     sine.100
GPNAM2     sine.100
GPNAM3     sine.100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.00 %
P16        1000.00 usec

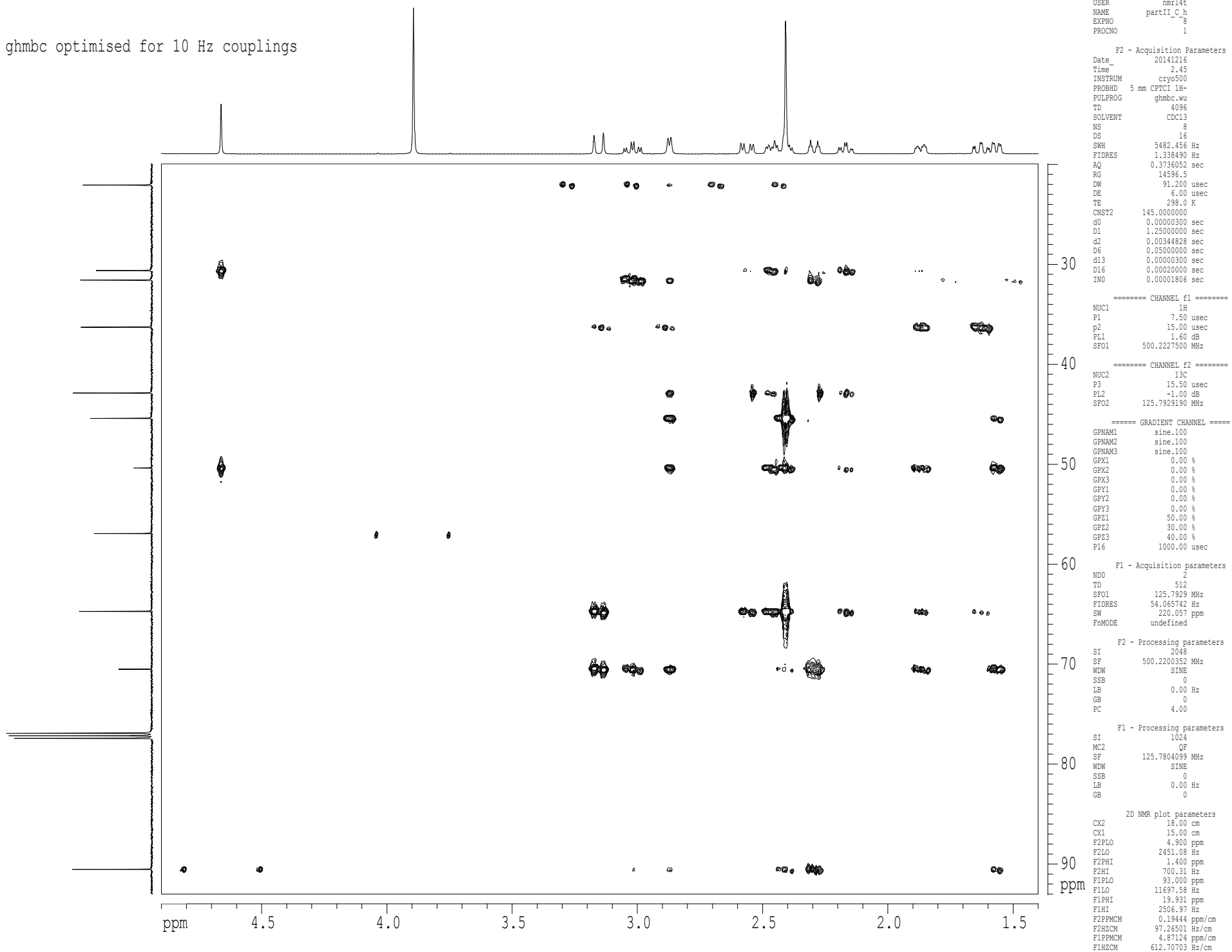
F1 - Acquisition parameters
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FnMODE     undefined

F2 - Processing parameters
SI         2048
SF         500.2200352 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         4.00

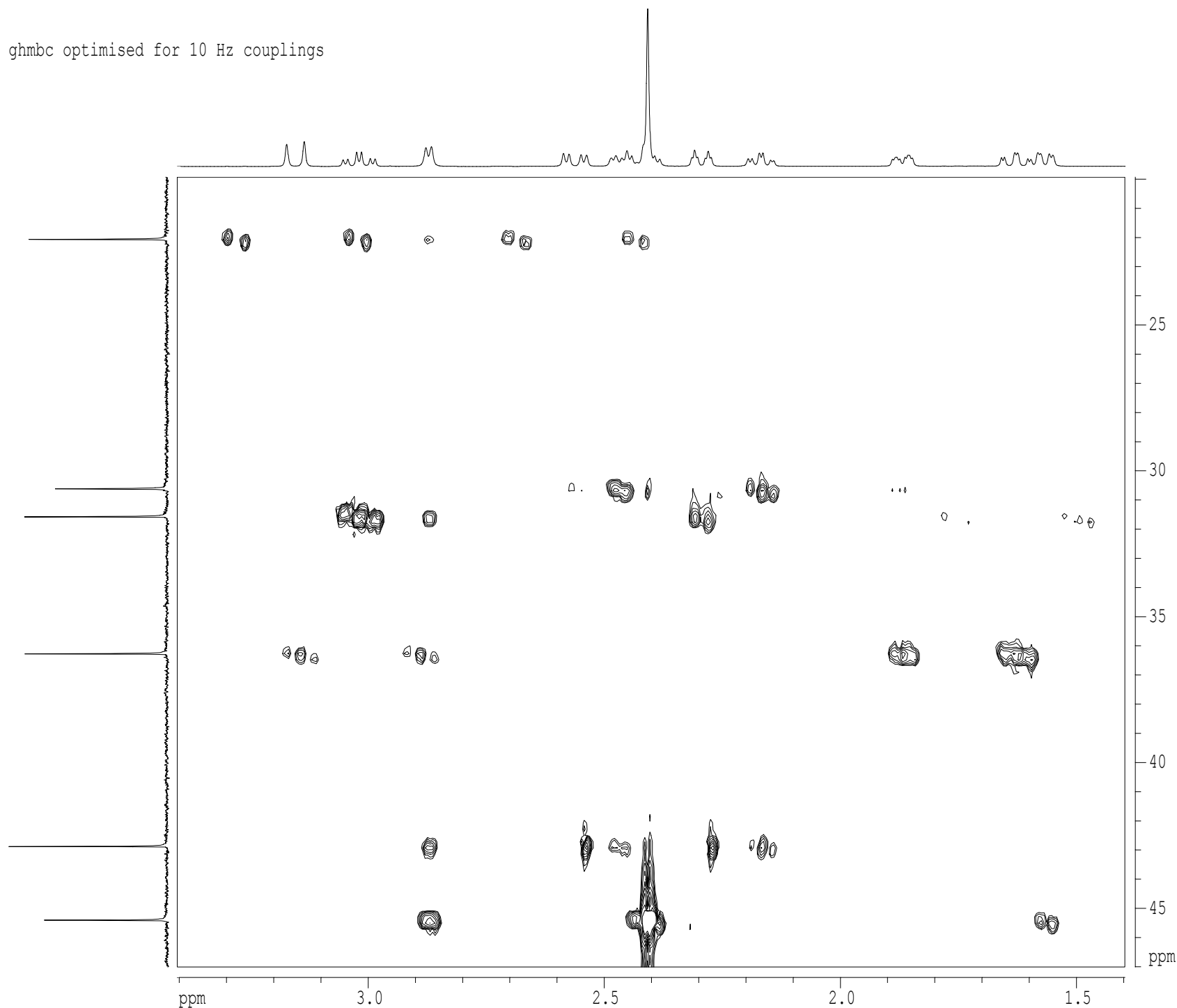
F1 - Processing parameters
SI         1024
MC2        QF
SF         125.7804099 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        18.00 cm
CX1        15.00 cm
F2PLO      9.000 ppm
F2LO       4501.98 Hz
F2PHI      0.000 ppm
F2HI       0.00 Hz
F1PLO      209.492 ppm
F1LO       26349.96 Hz
F1PHI      20.000 ppm
F1HI       2515.61 Hz
F2PPMCM    0.50000 ppm/cm
F2HZCM     250.11002 Hz/cm
F1PPMCM    12.63279 ppm/cm
F1HZCM     1588.95715 Hz/cm
```

ghmbc optimised for 10 Hz couplings



ghmbc optimised for 10 Hz couplings



```
Current Data Parameters
USER      nmr14t
NAME      partII_C_h
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20141216
Time      2.45
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   ghmbc.wu
TD         4096
SOLVENT   CDCl3
NS         8
DS         16
SWH        5482.456 Hz
FIDRES     1.338490 Hz
AQ         0.3736052 sec
RG         14596.5
DW         91.200 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.000000
d0         0.00000300 sec
D1         1.25000000 sec
d2         0.00344828 sec
D6         0.05000000 sec
d13        0.00000300 sec
D16        0.00020000 sec
IN0        0.00001806 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
P2         15.00 usec
PL1        1.60 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
NUC2       13C
P3         15.50 usec
PL2        -1.00 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPNAM1     sine.100
GPNAM2     sine.100
GPNAM3     sine.100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.00 %
P16        1000.00 usec

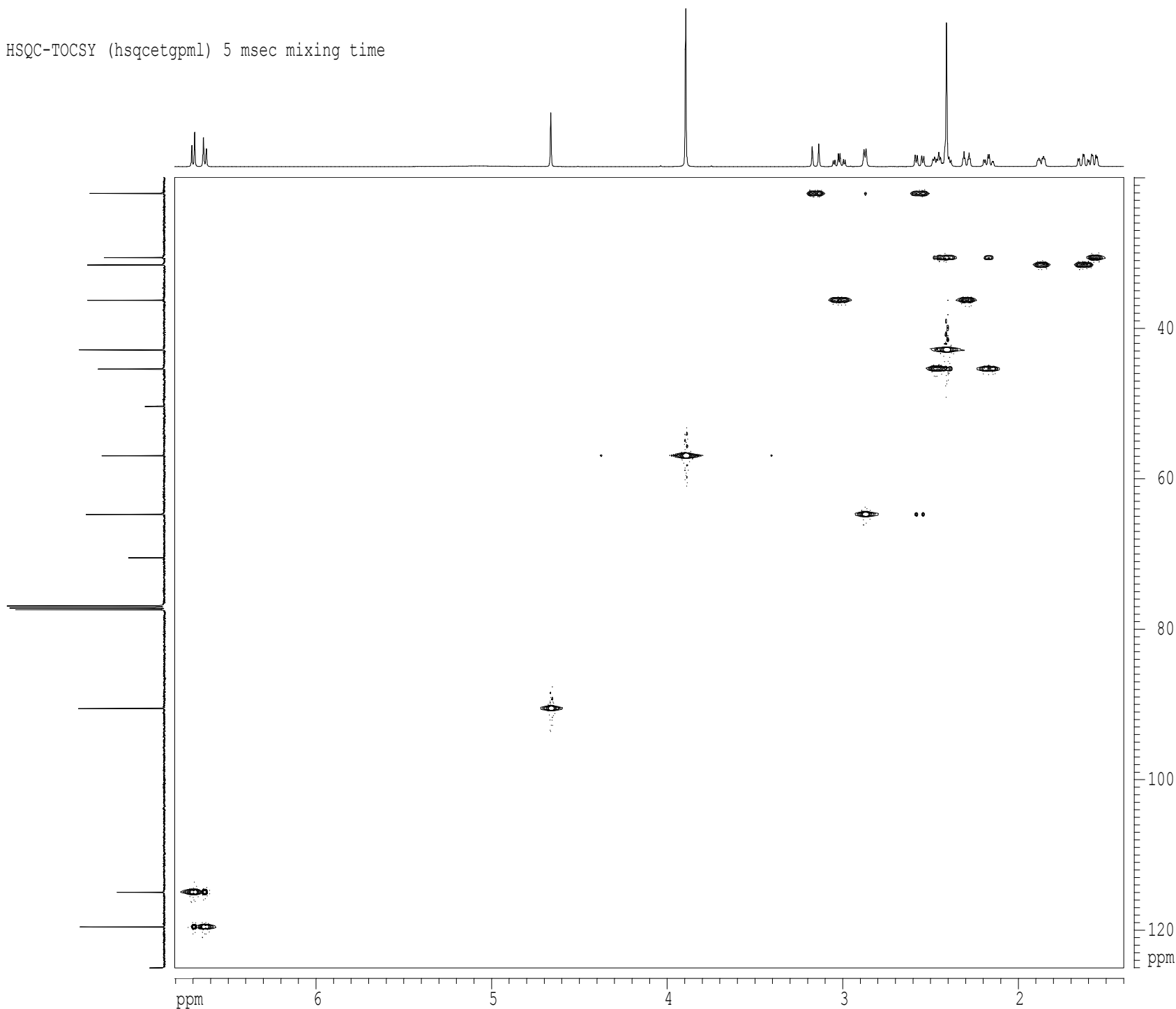
F1 - Acquisition parameters
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FnMODE     undefined

F2 - Processing parameters
SI         2048
SF         500.2200352 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0
PC         4.00

F1 - Processing parameters
SI         1024
MC2        QF
SF         125.7804099 MHz
WDW        SINE
SSB        0
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        16.00 cm
CX1        15.00 cm
F2PLO      3.404 ppm
F2LO       1702.87 Hz
F2PHI      1.397 ppm
F2HI       699.00 Hz
F1PLO      47.011 ppm
F1LO       5913.11 Hz
F1PHI      19.931 ppm
F1HI       2506.97 Hz
F2PPMCM    0.11149 ppm/cm
F2HZCM     55.77043 Hz/cm
F1PPMCM    1.80534 ppm/cm
F1HZCM     227.07614 Hz/cm
```

HSQC-TOCSY (hsgcetgpm1) 5 msec mixing time



```

Current Data Parameters
USER      rnr14t
NAME      part11_C_1
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20141217
Time      0.00
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   hsgcetgpm1
TD        2048
SOLVENT   CDCl3
NS         8
DS         32
SWH        5482.456 Hz
FIDRES     2.678980 Hz
AQ         0.1868276 sec
RG         1024
DW         91.200 usec
DE         6.50 usec
TE         298.0 K
CNST2     145.0000000
d0         0.00000300 sec
d1         2.00000000 sec
d4         0.00172414 sec
d9         0.00500000 sec
d11        0.03000000 sec
d12        0.00020000 sec
d13        0.00000400 sec
d16        0.00020000 sec
DELTA     0.0012100 sec
DELTA1    0.00120800 sec
FACTOR1   0
IN0        0.00001806 sec
l1         0
MCREST    0.00000000 sec
MCWRK     0.40000001 sec
SCALEF    6
STCNT     256

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p2         15.00 usec
p5         23.34 usec
p6         35.00 usec
p7         70.00 usec
P17        2500.00 usec
P28        1000.00 usec
PL1        1.60 dB
PL10       15.20 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
CPDPRG2    9atp
NUC2       13C
P3         15.50 usec
p4         31.00 usec
PCPD2      65.00 usec
PL2        -1.00 dB
PL12       11.30 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPRM1      SINE.100
GPRM2      SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GEZ1       80.00 %
GEZ2       20.10 %
P16        1000.00 usec

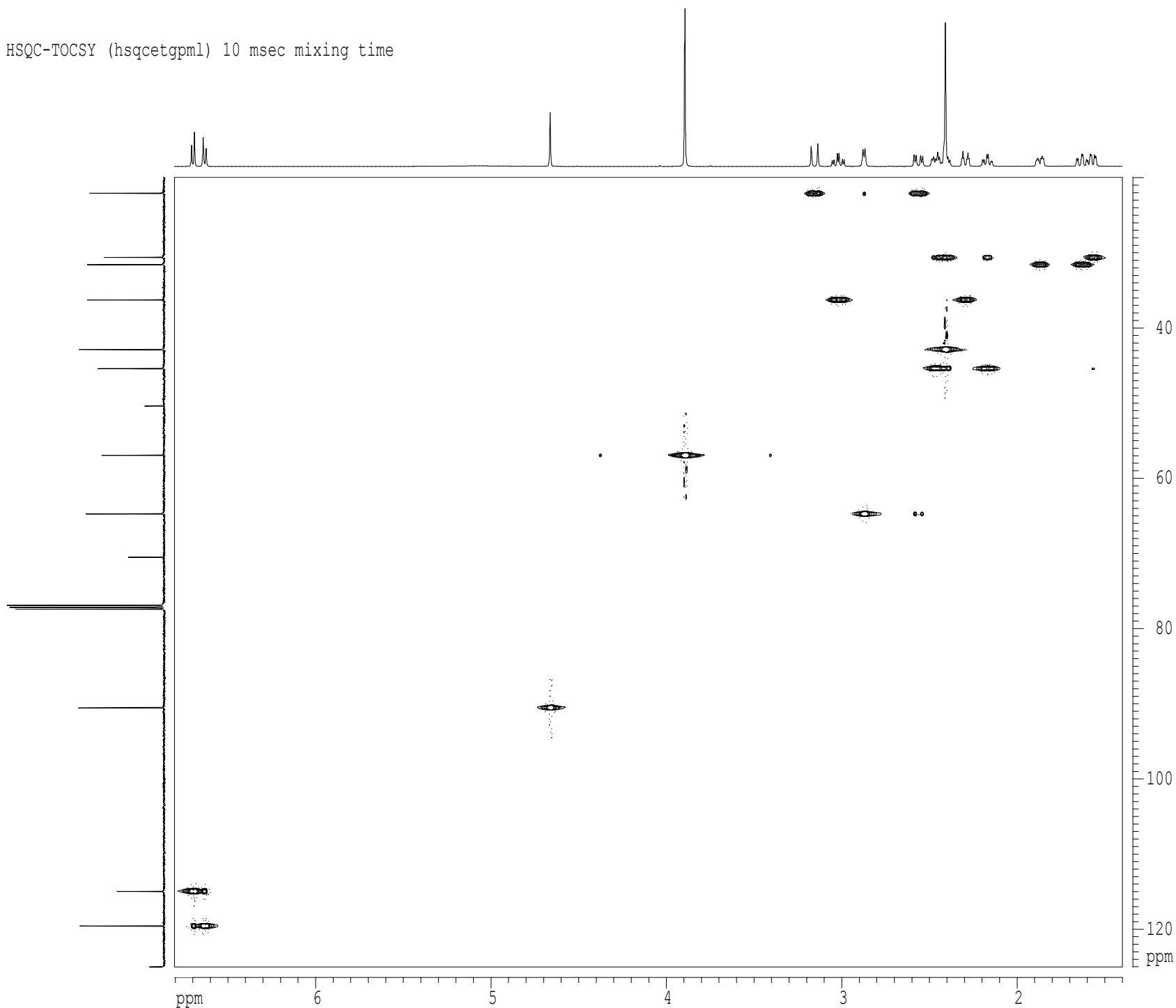
F1 - Acquisition parameters
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FAMODE     Echo-Antiecho

F2 - Processing parameters
SI         2048
SF         500.2200340 MHz
WDW        EM
SSB        0
LB         5.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        echo-antiecho
SF         125.7804099 MHz
WDW        QSINE
SSB        3
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        18.00 cm
CX1        15.00 cm
F2PLO      6.805 ppm
F2ZLO      3403.96 Hz
F2ZHI      1.400 ppm
F2ZL1      700.21 Hz
F1PLO      125.028 ppm
F1ZLO      15726.05 Hz
F1ZHI      19.331 ppm
F1ZL1      2504.97 Hz
F2PZMCM    0.30028 ppm/cm
F2HZMCM    150.20836 Hz/cm
F1PZMCM    7.00643 ppm/cm
F1HZMCM    881.27167 Hz/cm
    
```

HSQC-TOCSY (hsgcetgpm1) 10 msec mixing time



```
Current Data Parameters
USER      rnr14t
NAME      part11_C 1
EXPNO     75
PROCNO    1

F2 - Acquisition Parameters
Date_     20141217
Time      2.33
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   hsgcetgpm1
TD        2048
SOLVENT   CDCl3
NS         8
DS         32
SWH        5482.456 Hz
FIDRES     2.678980 Hz
AQ         0.1868276 sec
RG         1024
DW         91.200 usec
DE         6.50 usec
TE         298.0 K
CNST2     145.0000000
d0         0.00000300 sec
d1         2.00000000 sec
d4         0.00172414 sec
d9         0.01000000 sec
d11        0.03000000 sec
d12        0.00020000 sec
d13        0.00000400 sec
d16        0.00020000 sec
DELTA      0.0012100 sec
DELTA1     0.00120800 sec
FACTOR1    0
IN0        0.00001806 sec
l1         0
MCREST     0.00000000 sec
MCNMR      0.40000001 sec
SCALEF     6
STICNT     256

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p2         15.00 usec
p5         23.34 usec
p6         35.00 usec
p7         70.00 usec
P17        2500.00 usec
P28        1000.00 usec
PL1        1.60 dB
PL10       15.20 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
CPDPRG2    9atp
NUC2       13C
P3         15.50 usec
p4         31.00 usec
PCPD2      65.00 usec
PL2        -1.00 dB
PL12       11.30 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPRAM1     SINE.100
GPRAM2     SINE.100
GPX1        0.00 %
GPX2        0.00 %
GPY1        0.00 %
GPY2        0.00 %
GEZ1        80.00 %
GPZ2        20.10 %
P16        1000.00 usec

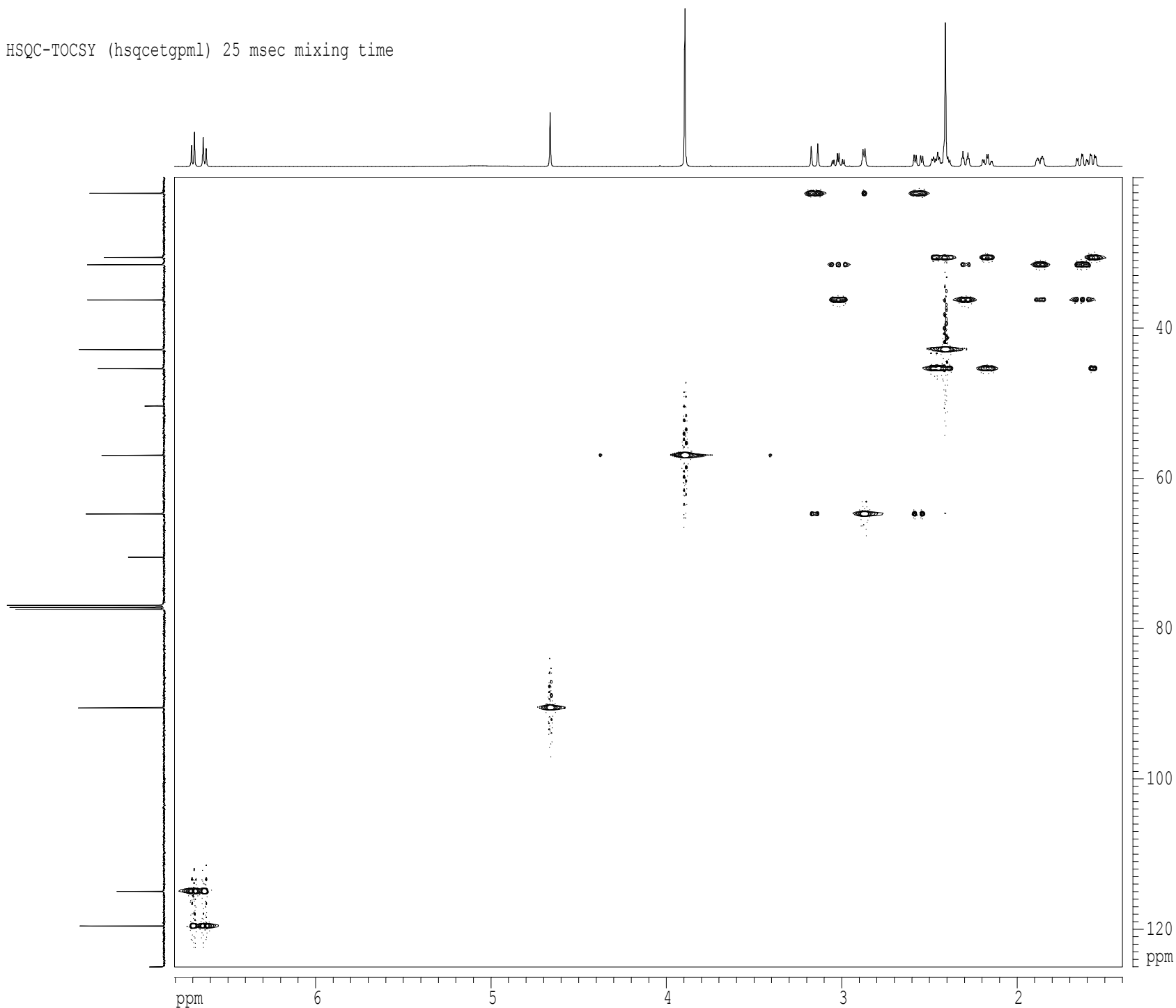
f1 - Acquisition parameters
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FAMODE     Echo-Antiecho

F2 - Processing parameters
SI         2048
SF         500.2200340 MHz
WDW        EM
SSB        0
LB         5.00 Hz
GB         0
PC         1.40

f1 - Processing parameters
SI         1024
MC2        echo-antiecho
SF         125.7804099 MHz
WDW        QSINE
SSB        3
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        18.00 cm
CX1        15.00 cm
F2PLO      6.805 ppm
F2PLO      3403.96 Hz
F2PHI      1.400 ppm
F2HI       700.21 Hz
F1PLO      125.028 ppm
F1LO       15726.05 Hz
F1HI       19.331 ppm
F1HI       2504.97 Hz
F2P2MCM    0.30028 ppm/cm
F2H2MCM    150.20836 Hz/cm
F1P2MCM     7.00643 ppm/cm
F1H2MCM    881.27167 Hz/cm
```

HSQC-TOCSY (hsgcetgpm1) 25 msec mixing time



```
Current Data Parameters
USER      rnr14t
NAME      part11_C 1
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20141217
Time      5.05
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   hsgcetgpm1
TD         2048
SOLVENT   CDCl3
NS         8
DS         32
SWH        5482.456 Hz
FIDRES     2.678980 Hz
AQ         0.1868276 sec
RG         1024
DW         91.200 usec
DE         6.50 usec
TE         298.0 K
CNST2     145.0000000
d0         0.0000300 sec
d1         2.00000000 sec
d4         0.00172414 sec
d9         0.02500000 sec
d11        0.03000000 sec
d12        0.00020000 sec
d13        0.00004000 sec
d16        0.00020000 sec
DELTA     0.0012100 sec
DELTA1    0.00120800 sec
FACTOR1   1
IN0       0.00001806 sec
l1
MCREST    0.00000000 sec
MCWRK     0.40000001 sec
SCALEF    6
STCNT     256

===== CHANNEL f1 =====
NUC1      1H
P1         7.50 usec
p2        15.00 usec
p5        23.34 usec
p6        35.00 usec
p7        70.00 usec
P17       2500.00 usec
P28       1000.00 usec
PL1       1.60 dB
PL10      15.20 dB
SFO1      500.2227500 MHz

===== CHANNEL f2 =====
CPDPRG2   gprp
NUC2      13C
P3         15.50 usec
p4         31.00 usec
PCPD2     65.00 usec
PL2       -1.00 dB
PL12      11.30 dB
SFO2      125.7929190 MHz

===== GRADIENT CHANNEL =====
GPRAM1    SINE.100
GPRAM2    SINE.100
GPX1      0.00 %
GPX2      0.00 %
GPY1      0.00 %
GPY2      0.00 %
GEZ1      80.00 %
GEZ2      20.10 %
P16       1000.00 usec

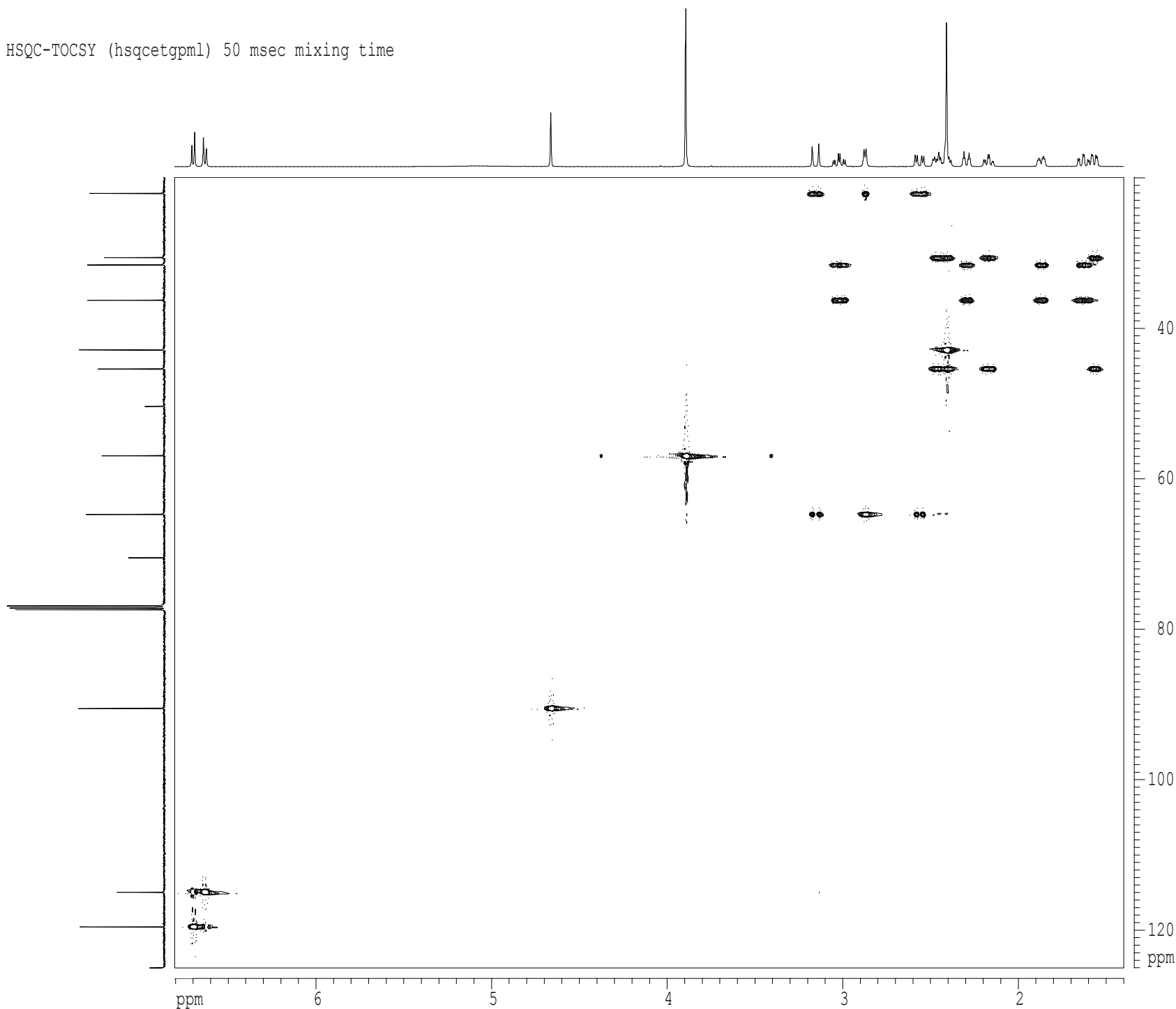
F1 - Acquisition parameters
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FAMODE     Echo-Antiecho

F2 - Processing parameters
SI         2048
SF         500.2200340 MHz
WDW        EM
SSB        0
LB         5.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MC2        echo-antiecho
SF         125.7804099 MHz
WDW        QSINE
SSB        3
LB         0.00 Hz
GB         0

2D NMR plot parameters
CX2        18.00 cm
CX1        15.00 cm
F2PLO      6.805 ppm
F2PLO      3403.96 Hz
F2PHI      1.400 ppm
F2HI       700.21 Hz
F1PLO      125.028 ppm
F1LO       15726.05 Hz
F1HI       19.331 ppm
F1HI       2504.97 Hz
F2P2MCM    0.30028 ppm/cm
F2H2MCM    150.20836 Hz/cm
F1P2MCM    7.00643 ppm/cm
F1H2MCM    881.27167 Hz/cm
```

HSQC-TOCSY (hsgcetgpm1) 50 msec mixing time



```
Current Data Parameters
=====
USER      nmrl4t
NAME      part11_C h
EXPNO     27
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20141217
Time      7.39
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   hsgcetgpm1
TD         2048
SOLVENT   CDCl3
NS         8
DS         32
SWH        5482.456 Hz
FIDRES     2.678980 Hz
AQ         0.1868276 sec
RG         1024
DW         91.200 usec
DE         6.50 usec
TE         298.0 K
=====
CNST2     145.0000000
d0         0.00000300 sec
d1         2.00000000 sec
d4         0.00172414 sec
d9         0.05000000 sec
d11        0.03000000 sec
d12        0.00020000 sec
d13        0.00000400 sec
d16        0.00020000 sec
DELTA      0.0012100 sec
DELTA1     0.00120800 sec
FACTOR1    3
IN0        0.00001806 sec
l1         18
MCREST     0.00000000 sec
MCNMRK     0.40000001 sec
SCALEF     6
ST1CNT     256

===== CHANNEL f1 =====
NUC1       1H
P1         7.50 usec
p2         15.00 usec
p5         23.34 usec
p6         35.00 usec
p7         70.00 usec
P17        2500.00 usec
P28        1000.00 usec
PL1        1.60 dB
PL10       15.20 dB
SFO1       500.2227500 MHz

===== CHANNEL f2 =====
CPDPRG2    9atp
NUC2       13C
P3         15.50 usec
p4         31.00 usec
PCPD2      65.00 usec
PL2        -1.00 dB
PL12       11.30 dB
SFO2       125.7929190 MHz

===== GRADIENT CHANNEL =====
GPRM1      SINE.100
GPRM2      SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GEZ1       80.00 %
GPZ2       20.10 %
P16        1000.00 usec

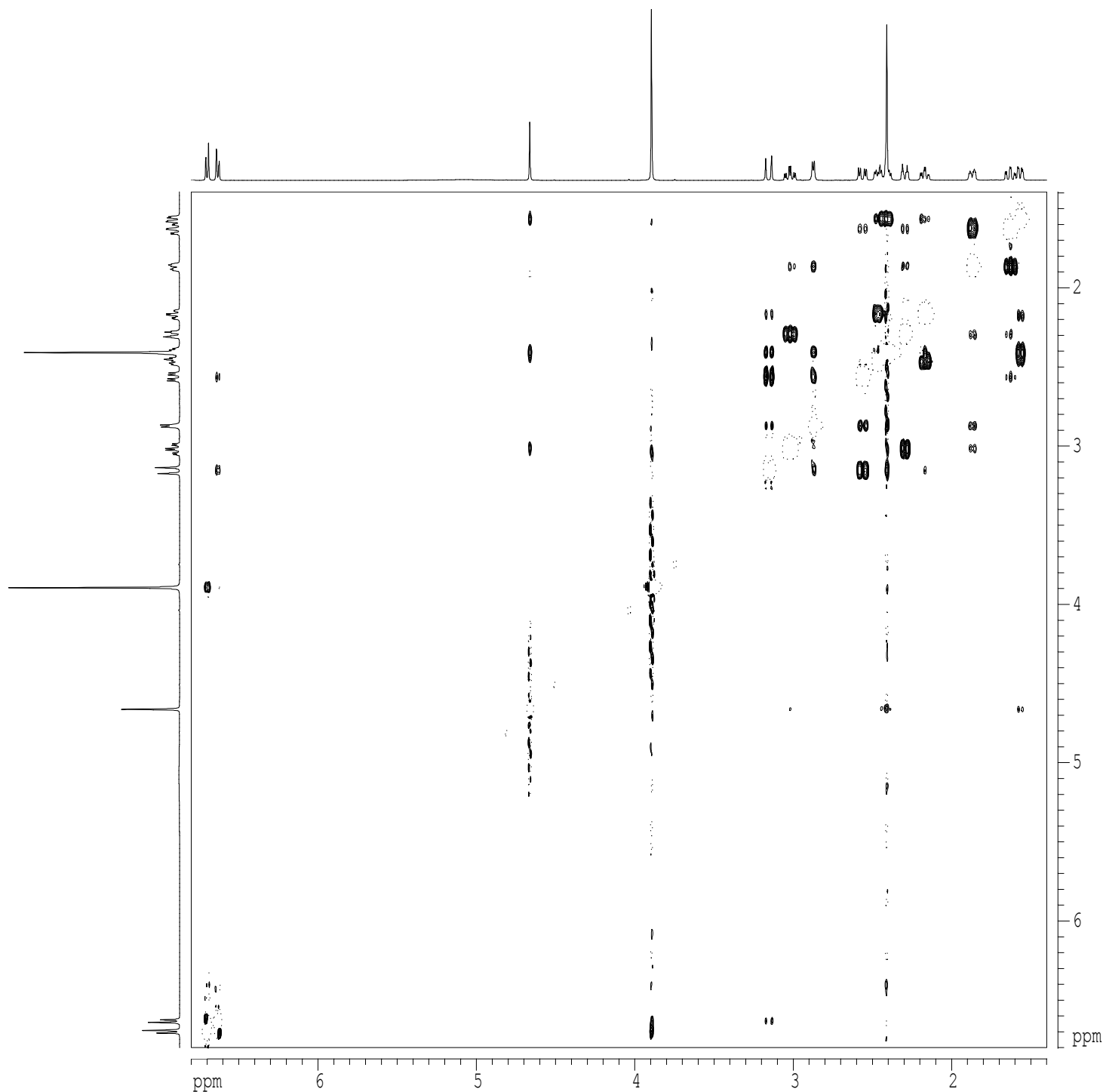
F1 - Acquisition parameters
=====
ND0        2
TD         512
SFO1       125.7929 MHz
FIDRES     54.065742 Hz
SW         220.057 ppm
FAMODE     Echo-Antiecho

F2 - Processing parameters
=====
SI         2048
SF         500.2200340 MHz
WDW        EM
SSB        0
LB         5.00 Hz
GB         0
PC         1.40

F1 - Processing parameters
=====
SI         1024
MC2        echo-antiecho
SF         125.7804099 MHz
WDW        QSINE
SSB        3
LB         0.00 Hz
GB         0

2D NMR plot parameters
=====
CX2        18.00 cm
CX1        15.00 cm
F2PLO      6.805 ppm
F2PLO      3403.96 Hz
F2PHI      1.400 ppm
F2HI       700.21 Hz
F1PLO      125.028 ppm
F1LO       15726.05 Hz
F1PHI      19.331 ppm
F1HI       2504.97 Hz
F2P2MCM    0.30028 ppm/cm
F2H2MCM    150.20836 Hz/cm
F1P2MCM     7.00643 ppm/cm
F1H2MCM    881.27167 Hz/cm
```


noesy



Current Data Parameters

USER nmrl4t
NAME partII_C h
EXPNO 23
PROCNO 1

F2 - Acquisition Parameters

Date_ 20141216
Time_ 22.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG noesyrvtp
TD 2048
SOLVENT CDC13
NS 8
DS 16
SWH 5482.456 Hz
FIDRES 2.676980 Hz
AQ 0.1868276 sec
RG 5
DW 91.200 usec
DE 6.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D8 1.00000000 sec
IN0 0.00009120 sec

===== CHANNEL f1 =====

NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SF01 500.2227500 MHz

F1 - Acquisition parameters

ND0 2
TD 256
SF01 500.2228 MHz
FIDRES 21.415844 Hz
SW 10.960 ppm
FnMODE undefined

F2 - Processing parameters

SI 2048
SF 500.2200340 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

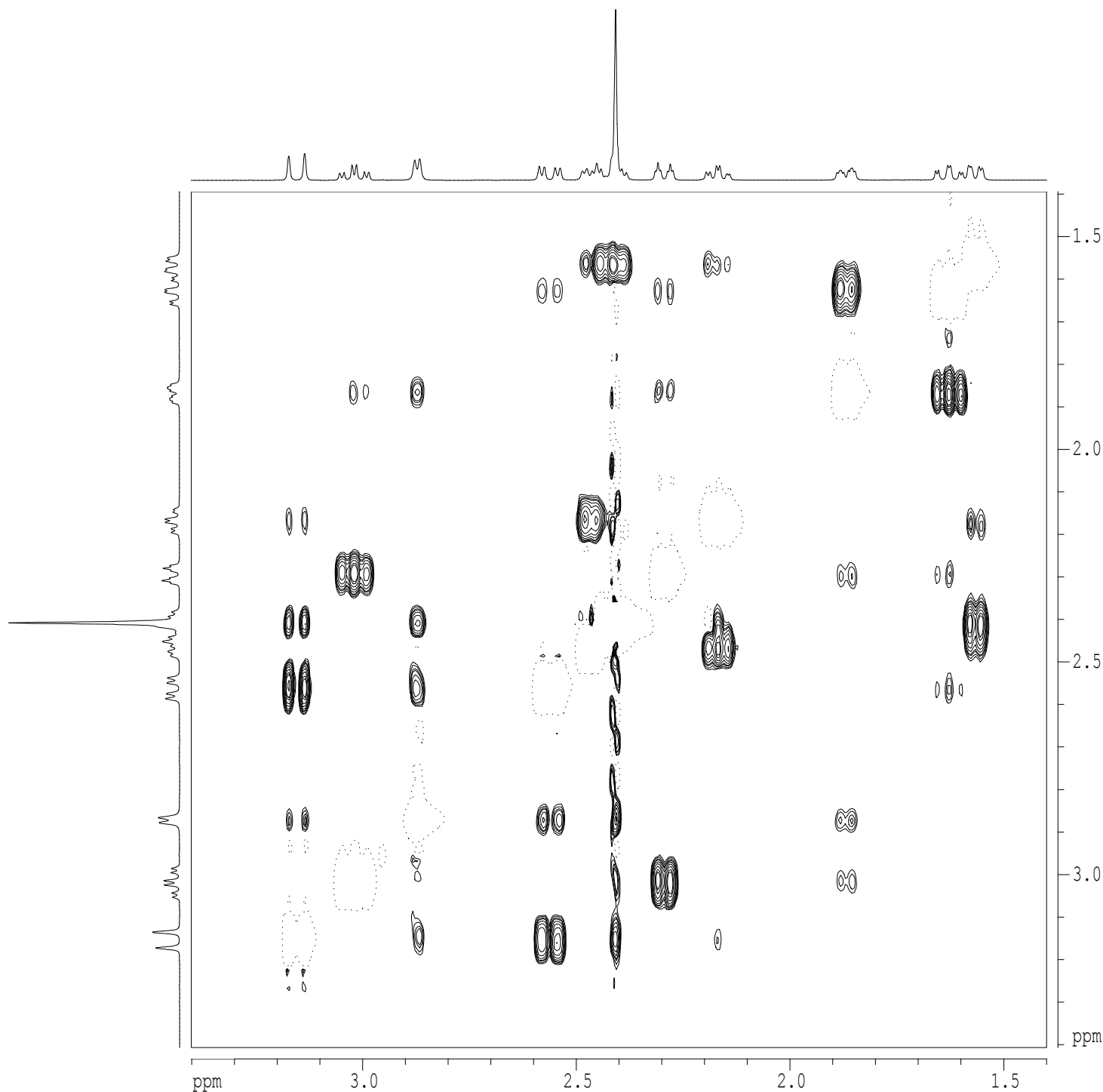
F1 - Processing parameters

SI 1024
MC2 TPPI
SF 500.2200340 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters

CX2 15.00 cm
CX1 15.00 cm
F2PLO 6.800 ppm
F2LO 3401.50 Hz
F2PHI 1.400 ppm
F2HI 700.21 Hz
F1PLO 6.800 ppm
F1LO 3401.50 Hz
F1PHI 1.394 ppm
F1HI 697.53 Hz
F2PPMCM 0.36001 ppm/cm
F2HZCM 180.08600 Hz/cm
F1PPMCM 0.36037 ppm/cm
F1HZCM 180.26447 Hz/cm

noesy



Current Data Parameters

USER	nmrl4t
NAME	partII_C h
EXPNO	23
PROCNO	1

F2 - Acquisition Parameters

Date_	20141216
Time_	22.10
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	noesyrvtp
TD	2048
SOLVENT	CDC13
NS	8
DS	16
SWH	5482.456 Hz
FIDRES	2.676980 Hz
AQ	0.1868276 sec
RG	5
DW	91.200 usec
DE	6.00 usec
TE	298.0 K
D0	0.0000300 sec
D1	2.0000000 sec
D8	1.0000000 sec
IN0	0.00009120 sec

===== CHANNEL f1 =====

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2227500 MHz

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	500.2228 MHz
FIDRES	21.415844 Hz
SW	10.960 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200340 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	1024
MC2	TPPI
SF	500.2200340 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	3.401 ppm
F2LO	1701.40 Hz
F2PHI	1.400 ppm
F2HI	700.21 Hz
F1PLO	3.407 ppm
F1LO	1704.07 Hz
F1PHI	1.394 ppm
F1HI	697.53 Hz
F2PPMCM	0.13343 ppm/cm
F2HZCM	66.74605 Hz/cm
F1PPMCM	0.13415 ppm/cm
F1HZCM	67.10298 Hz/cm