Name: _____

Chem 203
December 15, 2012

Final Exam Part II
Problem 3 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 DISHNONESTY, WILL RESULT IN A FAILING GRADE (F) IN THE CLASS, AND MAY RESULT IN EXPULSION FROM THE Ph.D. PROGRAM.

If you wish to use a laptop computer, please be willing to share briefly with others when needed.

The following spectral data are provided for a compound with a molecular formula $C_{10}H_{19}N$: IR (thin film on NaCl plates), 500 MHz ^{1}H NMR, 125.7 MHz ^{13}C NMR, DEPT-90, DEPT-135, COSY, HMQC, HMBC, TOCSY and NOESY. Using these data, determine the structure of the compound and assign all of the ^{1}H and ^{13}C resonances to their respective atoms in the structure. Make sure to determine the stereochemistry of the compound and to clearly assign ^{1}H resonances to the appropriate diastereotopic protons.

All NMR spectra were measured in $CDCl_3$.

MAKE SURE TO COMPLETELY ANSWER THE QUESTIONS **a-e** 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. Tabulate the shifts of the ^{1}H resonances (a-k) 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:

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

d. Tabulate the shifts of the ^{13}C resonances (1-10) 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:

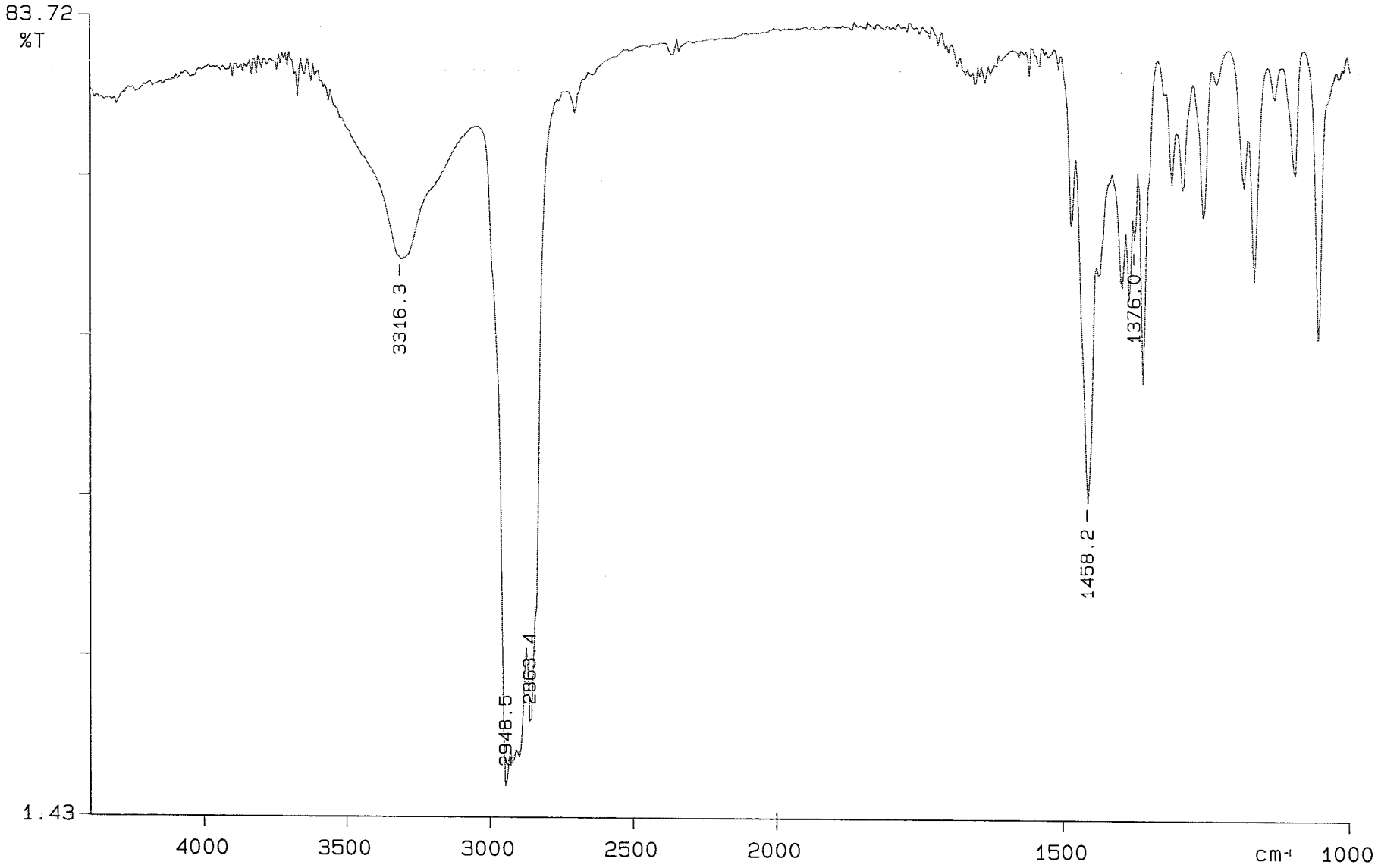
8:

9:

10:

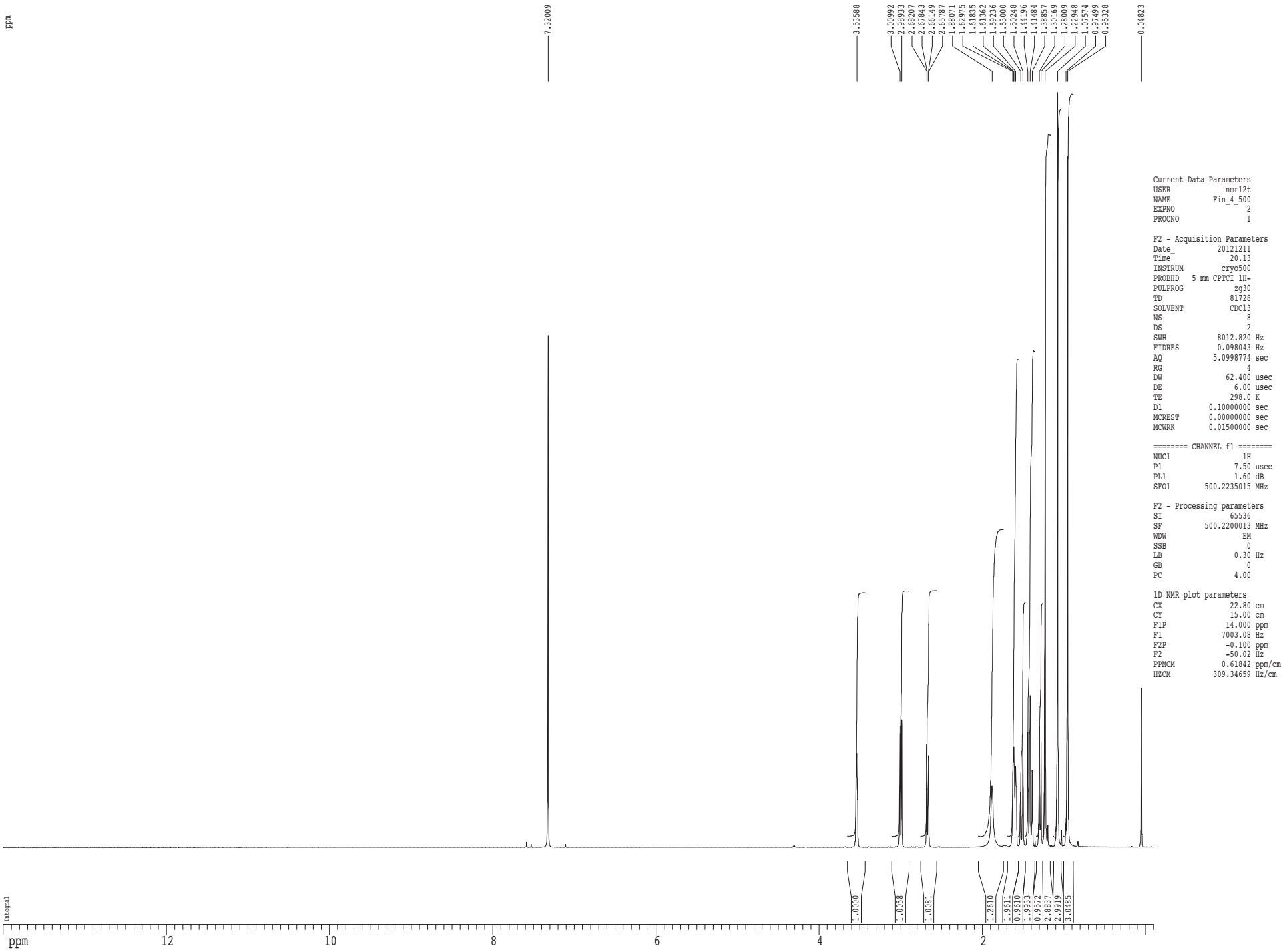
e. Redraw the structure of the molecule below and write the number (1-10) of each ^{13}C resonance next to the carbon or carbons to which it corresponds. If the assignment of particular resonance is uncertain, please indicate this.

PERKIN ELMER



12/12/12 15:00
X: 4 scans, 4.0cm-1

1H spectrum



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PROCNO 1

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PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
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
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200013 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 14.000 ppm
F1 7003.08 Hz
F2P -0.100 ppm
F2 -50.02 Hz
PPMCM 0.61842 ppm/cm
HZCM 309.34659 Hz/cm

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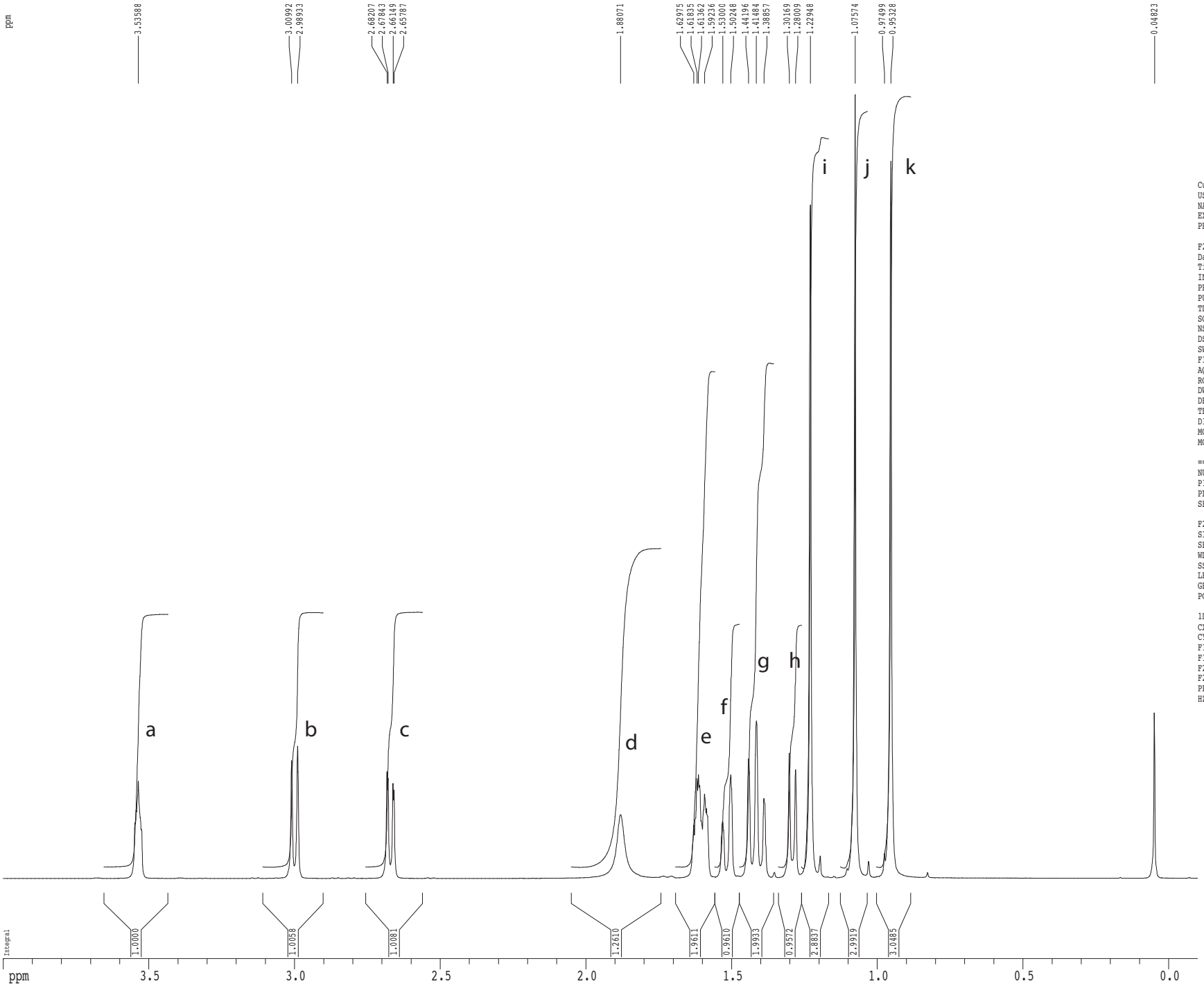
#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	46929.6	1768.719	3.5359	1.89
2	49081.4	1505.621	3.0099	2.29
3	49165.6	1495.323	2.9893	2.56
4	50422.7	1341.623	2.6821	2.07
5	50437.6	1339.802	2.6784	1.94
6	50506.9	1331.330	2.6615	1.84
7	50521.7	1329.518	2.6579	1.72
8	53701.3	940.767	1.8807	1.25
9	54728.0	815.232	1.6297	1.05
10	54774.7	809.529	1.6183	1.94
11	54794.0	807.163	1.6136	2.01
12	54881.0	796.530	1.5924	1.64
13	55136.1	765.337	1.5300	1.12
14	55248.7	751.572	1.5025	2.01
15	55496.3	721.298	1.4420	2.31
16	55607.3	707.733	1.4148	3.04
17	55714.8	694.588	1.3886	1.56
18	56070.2	651.129	1.3017	2.42
19	56158.6	640.325	1.2801	2.11
20	56365.6	615.008	1.2295	12.89
21	56994.6	538.106	1.0757	15.00
22	57406.8	487.710	0.9750	0.51
23	57495.6	476.848	0.9533	13.73
24	61198.4	24.124	0.0482	3.20

1H spectrum

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NAME            Fin_4_500
EXPNO                   2
PROCNO                  1

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Time                20.13
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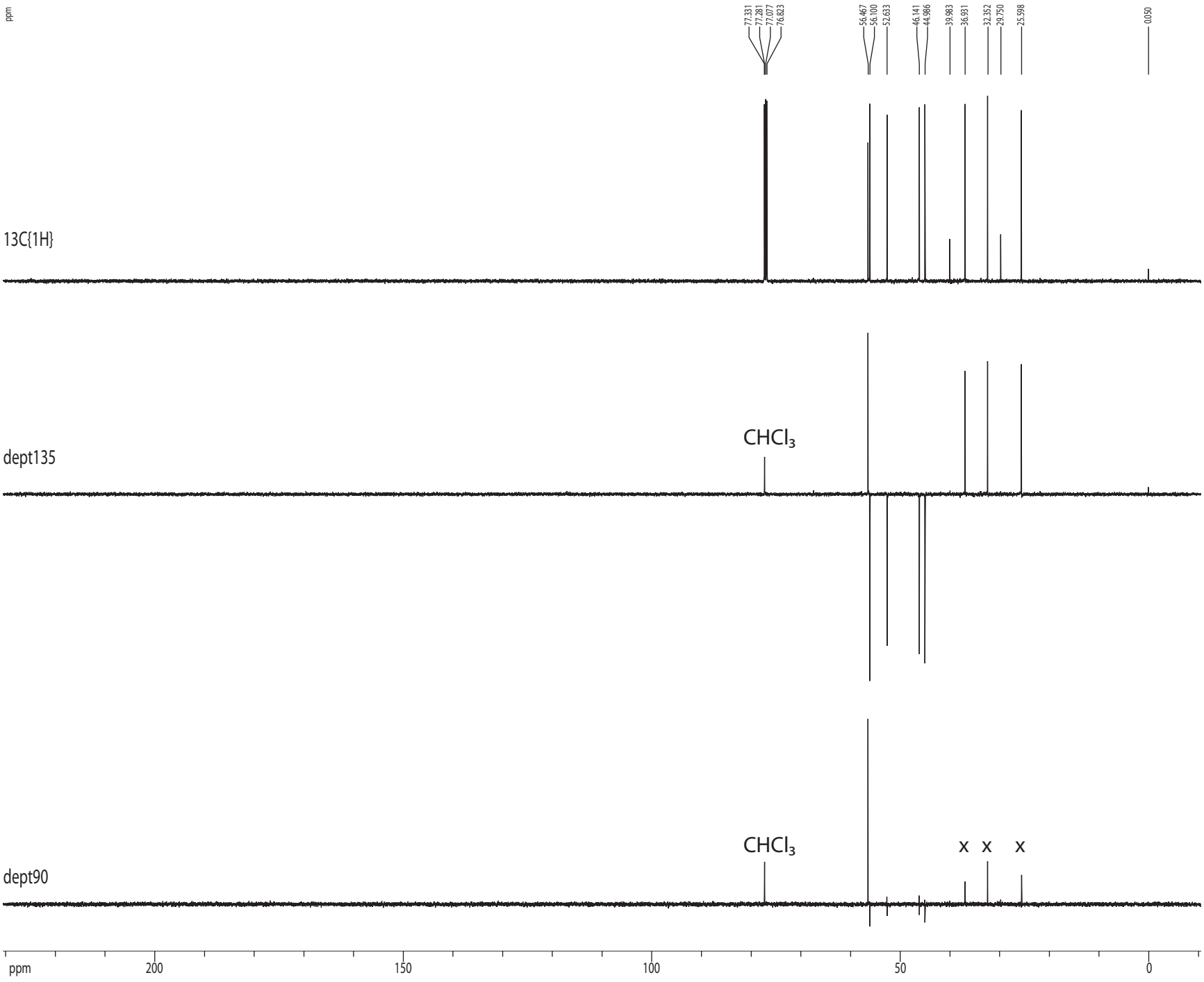
PULPROG               zg30
TD                   81728
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                       4
DW                 62.400 usec
DE                  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
SFO1          500.2235015 MHz

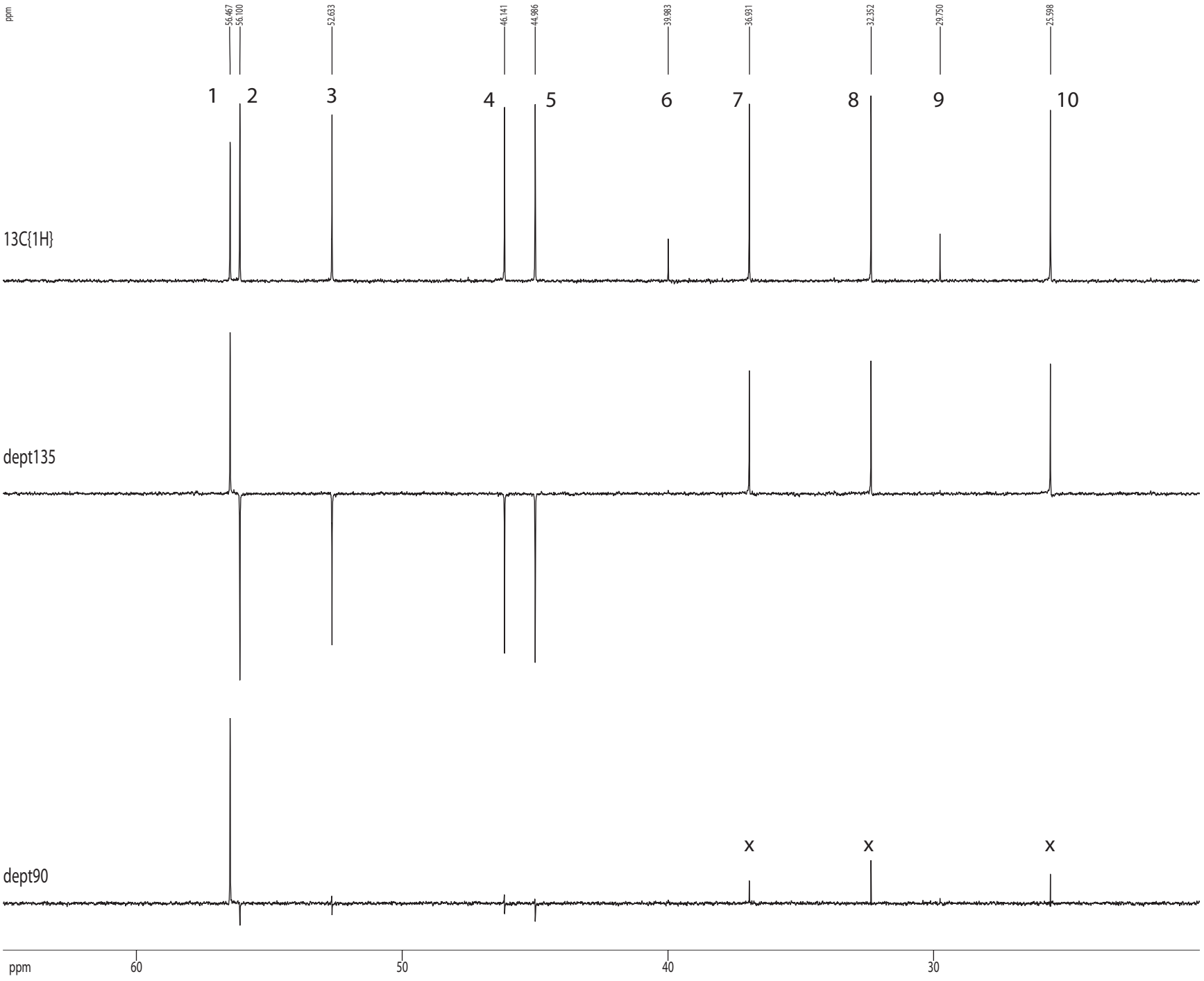
F2 - Processing parameters
SI                   65536
SF            500.2200013 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   4.00

1D NMR plot parameters
CX                   22.80 cm
CY                   15.00 cm
F1P                  4.000 ppm
F1                2000.88 Hz
F2P                 -0.100 ppm
F2                  -50.02 Hz
PPMCM             0.17982 ppm/cm
HZCM             89.95184 Hz/cm
```

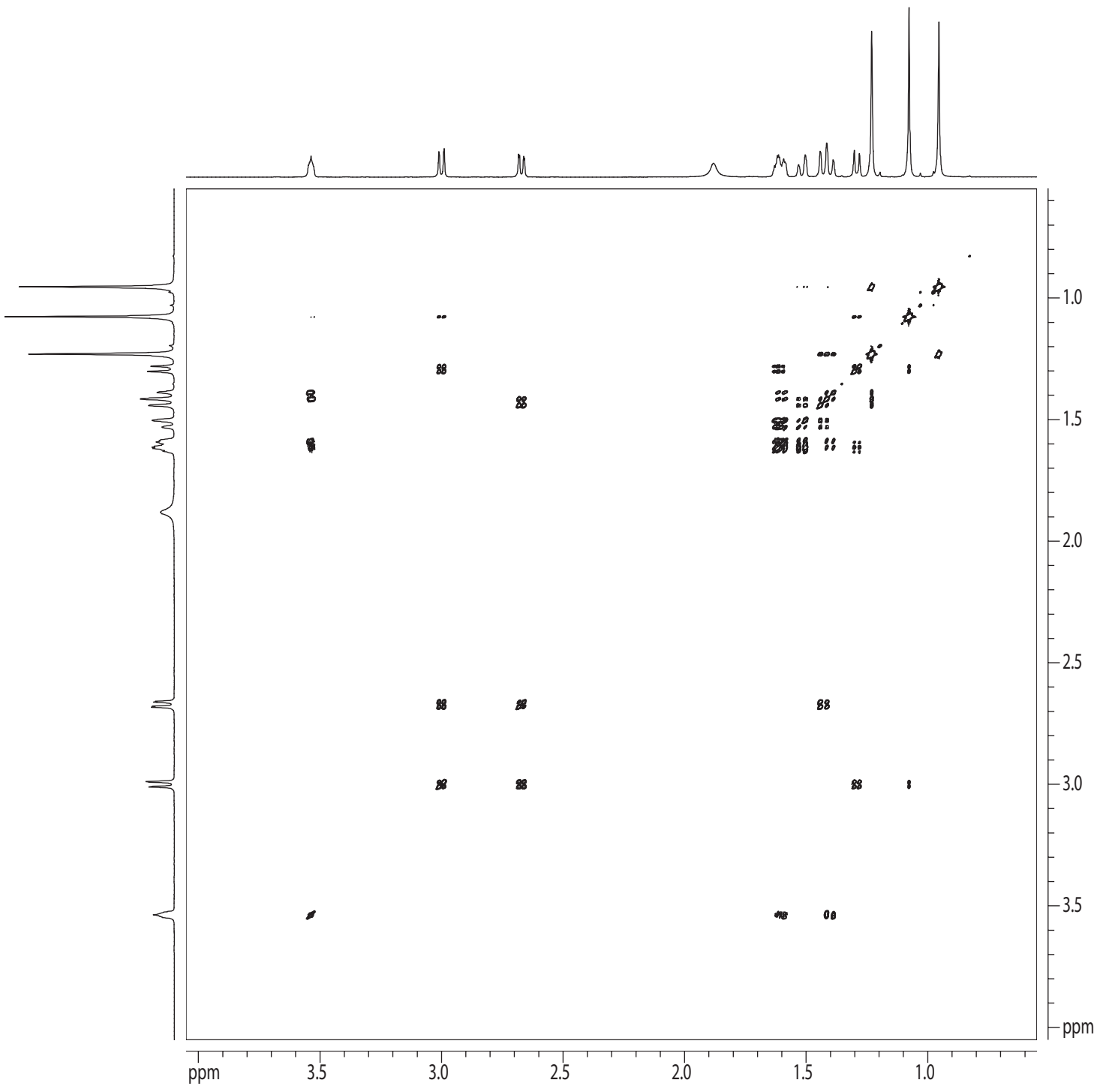
Z-restored spin-echo 13C spectrum with 1H decoupling



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



gcosy60



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PROCNO 1

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PULPROG cosygp60.prd
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 1750.700 Hz
FIDRES 0.854834 Hz
AQ 0.5849588 sec
RG 4
DW 285.600 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 0.00057120 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2211510 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

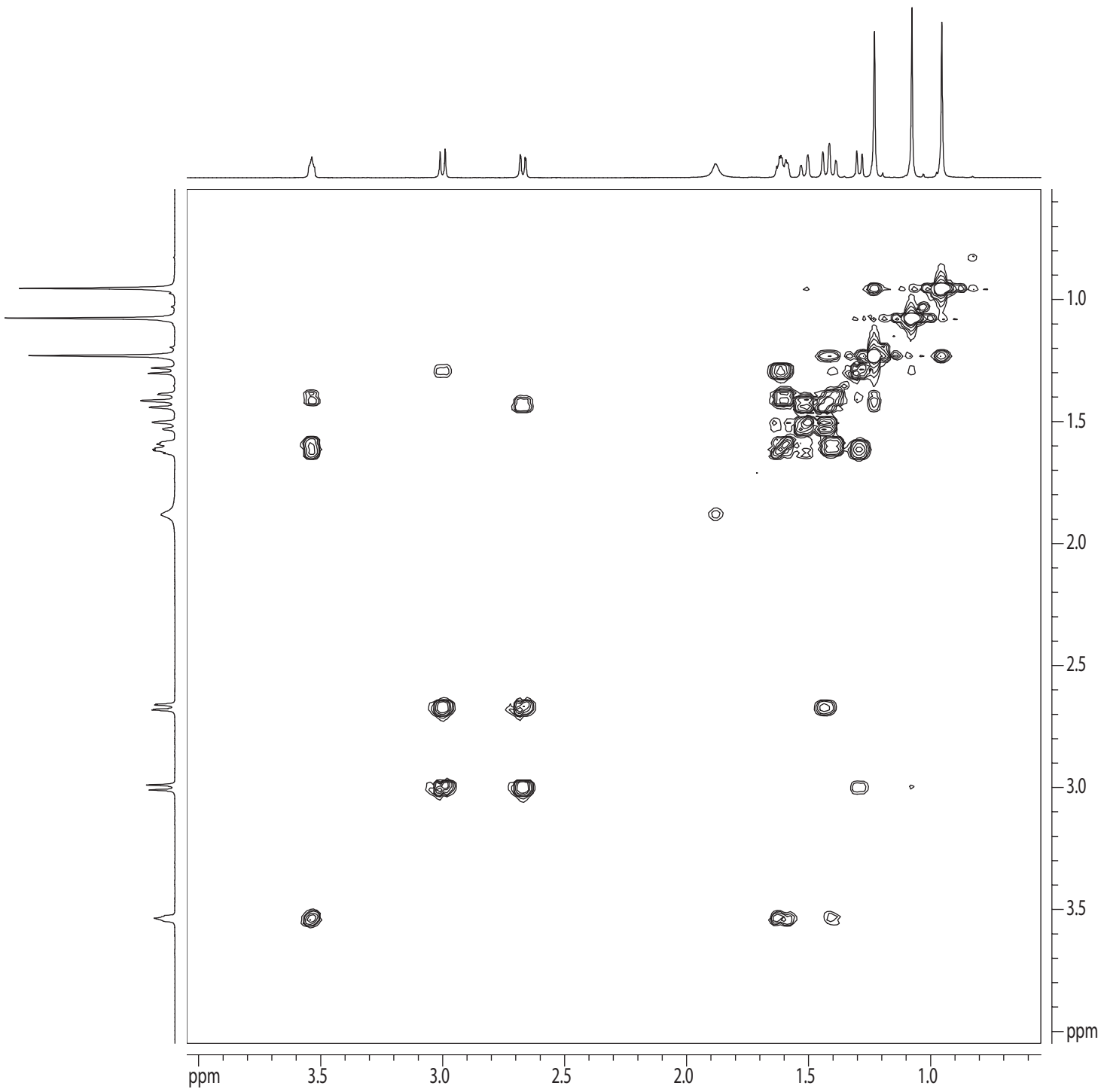
F1 - Acquisition parameters
ND0 1
TD 512
SFO1 500.2212 MHz
FIDRES 3.419337 Hz
SW 3.500 ppm
FnMODE undefined

F2 - Processing parameters
SI 1024
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 4.051 ppm
F2LO 2026.36 Hz
F2PHI 0.551 ppm
F2HI 275.66 Hz
F1PLO 4.051 ppm
F1LO 2026.36 Hz
F1PHI 0.551 ppm
F1HI 275.66 Hz
F2PPMCM 0.23332 ppm/cm
F2HZCM 116.71336 Hz/cm
F1PPMCM 0.23332 ppm/cm
F1HZCM 116.71336 Hz/cm

TOCSY



Current Data Parameters
USER nmr12t
NAME Fin_4_500
EXPNO 6
PROCNO 2

F2 - Acquisition Parameters
Date_ 20121211
Time 20.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG cosygp60.prd
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 1750.700 Hz
FIDRES 0.854834 Hz
AQ 0.5849588 sec
RG 4
DW 285.600 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 0.00057120 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2211510 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

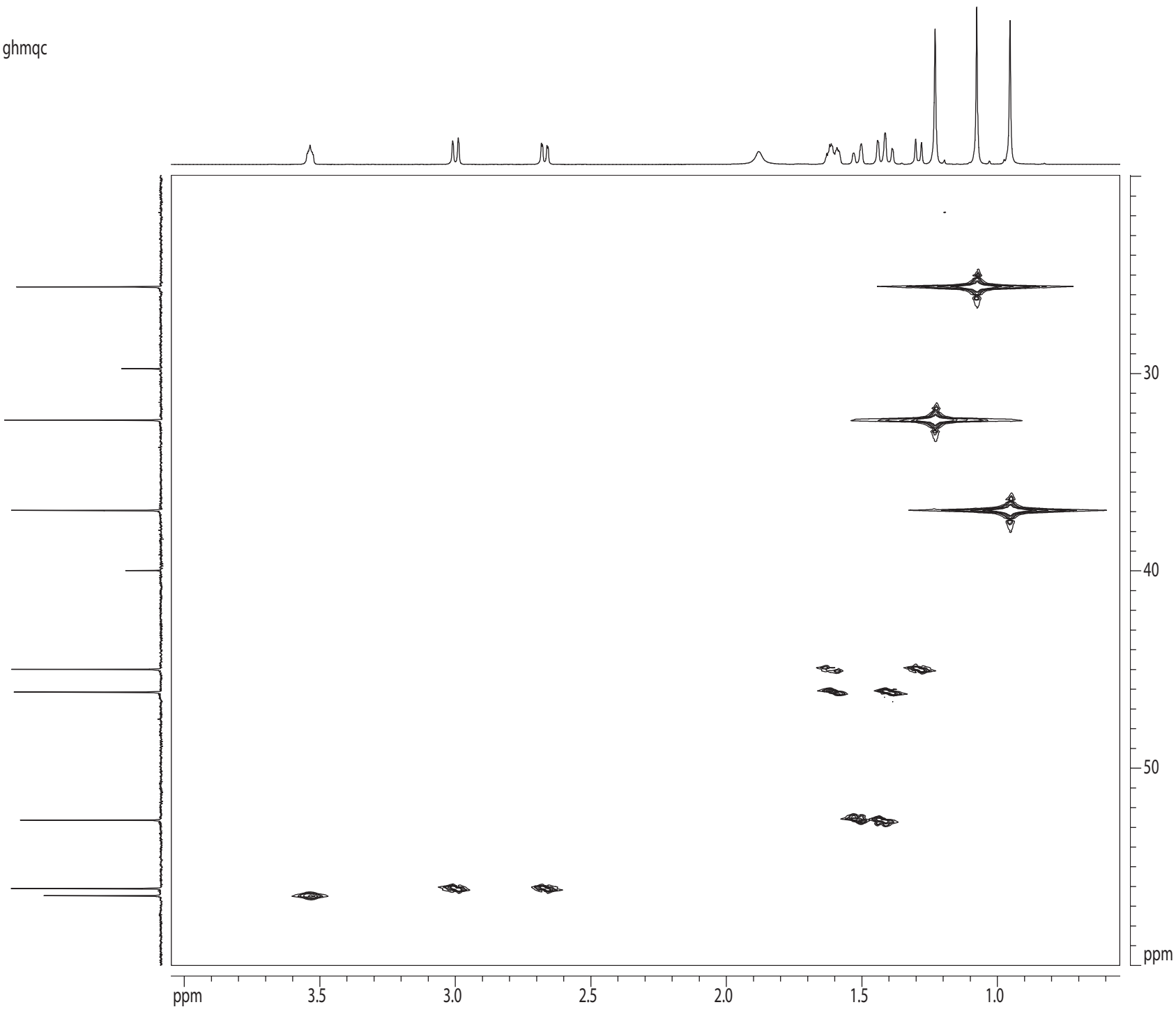
F1 - Acquisition parameters
ND0 1
TD 512
SFO1 500.2212 MHz
FIDRES 3.419337 Hz
SW 3.500 ppm
FnMODE undefined

F2 - Processing parameters
SI 256
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 256
MC2 QF
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 4.048 ppm
F2LO 2025.02 Hz
F2PHI 0.548 ppm
F2HI 274.32 Hz
F1PLO 4.048 ppm
F1LO 2025.02 Hz
F1PHI 0.548 ppm
F1HI 274.32 Hz
F2PPMCM 0.23332 ppm/cm
F2HZCM 116.71336 Hz/cm
F1PPMCM 0.23332 ppm/cm
F1HZCM 116.71336 Hz/cm

ghmqc



Current Data Parameters
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NAME Fin_4_500
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PROCNO 1

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PULPROG inv4gp.wu
TD 2048
SOLVENT CDCl3
NS 4
DS 16
SWH 1750.700 Hz
FIDRES 0.854834 Hz
AQ 0.5849588 sec
RG 8192
DW 285.600 usec
DE 6.50 usec
TE 298.0 K
CNST2 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 0.00004960 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p2 15.00 usec
PL1 1.60 dB
SFO1 500.2211510 MHz

======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 15.50 usec
PCPD2 65.00 usec
PL2 -1.00 dB
PL12 11.30 dB
SFO2 125.7867080 MHz

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

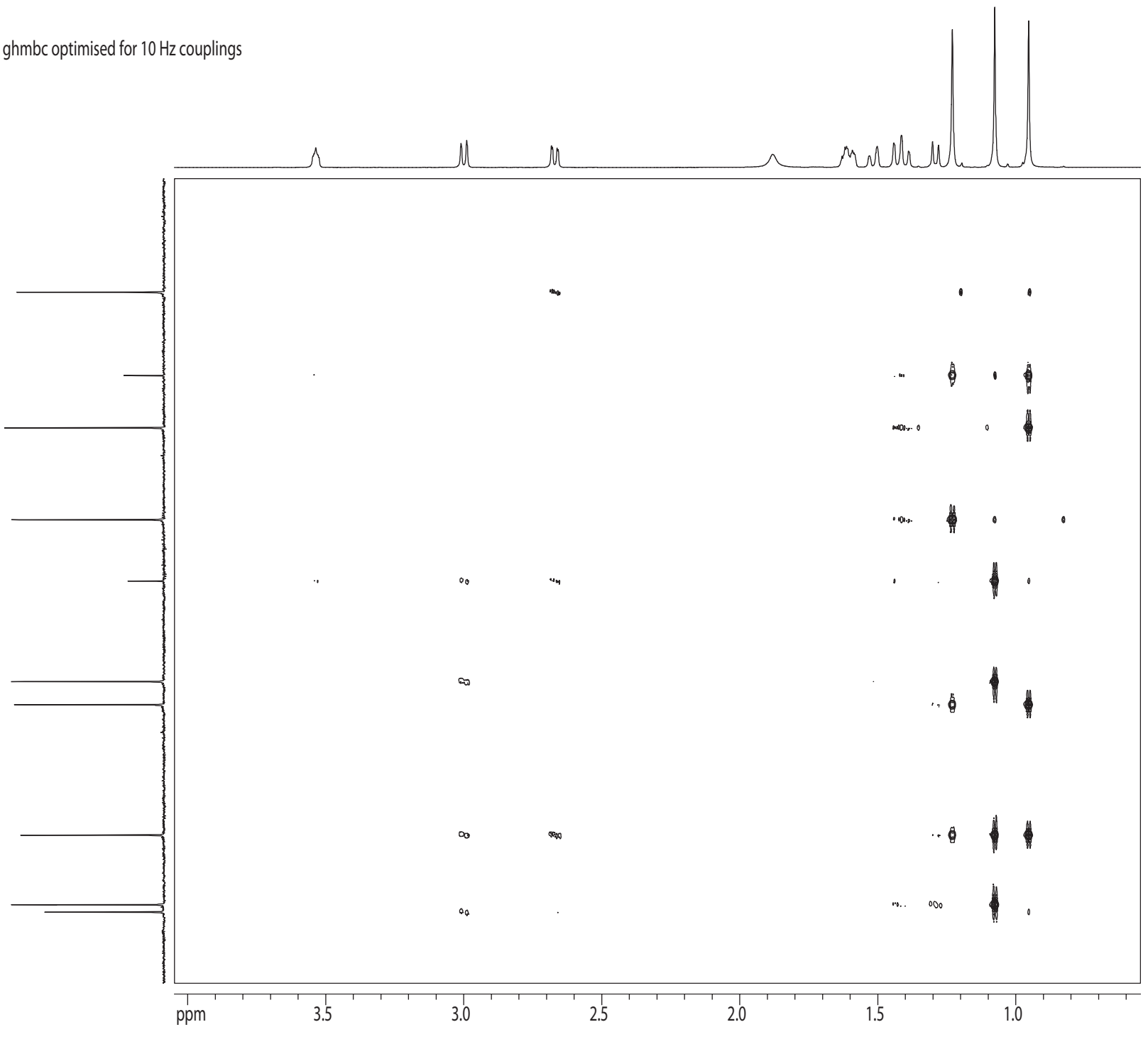
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7867 MHz
FIDRES 19.688761 Hz
SW 80.141 ppm
FnMODE undefined

F2 - Processing parameters
SI 1024
SF 500.2200013 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7804190 MHz
WDW QSINE
SSB 3
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 18.00 cm
CX1 15.00 cm
F2PLO 4.048 ppm
F2LO 2025.02 Hz
F2PHI 0.548 ppm
F2HI 274.32 Hz
F1PLO 60.000 ppm
F1LO 7546.83 Hz
F1PHI 19.946 ppm
F1HI 2508.77 Hz
F2PPMCM 0.19444 ppm/cm
F2HZCM 97.26112 Hz/cm
F1PPMCM 2.67029 ppm/cm
F1HZCM 335.87018 Hz/cm

ghmbc optimised for 10 Hz couplings



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PROCNO 1

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TD 4096
SOLVENT CDCl3
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DS 16
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FIDRES 0.427417 Hz
AQ 1.1698676 sec
RG 5792.6
DW 285.600 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
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D1 1.25000000 sec
d2 0.00344828 sec
D6 0.05000000 sec
d13 0.00000300 sec
D16 0.00020000 sec
IN0 0.00004960 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p2 15.00 usec
PL1 1.60 dB
SFO1 500.2211510 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 15.50 usec
PL2 -1.00 dB
SFO2 125.7867080 MHz

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

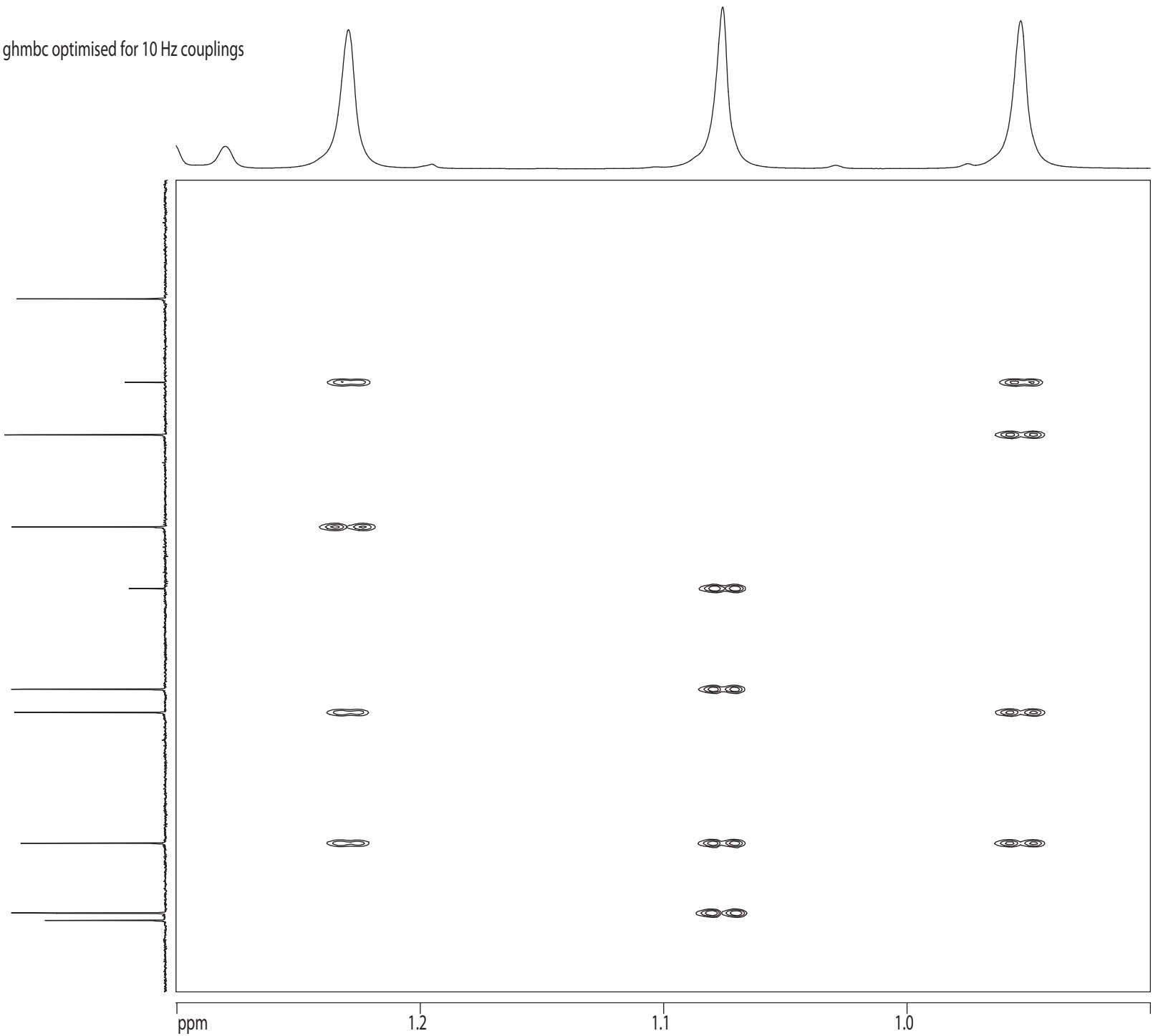
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7867 MHz
FIDRES 19.688761 Hz
SW 80.141 ppm
FnMODE undefined

F2 - Processing parameters
SI 2048
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7804190 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 18.00 cm
CX1 15.00 cm
F2PLO 4.048 ppm
F2LO 2025.02 Hz
F2PHI 0.548 ppm
F2HI 274.32 Hz
F1PLO 60.018 ppm
F1LO 7549.09 Hz
F1PHI 19.946 ppm
F1HI 2508.77 Hz
F2PPMCM 0.19444 ppm/cm
F2HZCM 97.26112 Hz/cm
F1PPMCM 2.67149 ppm/cm
F1HZCM 336.02151 Hz/cm

ghmbc optimised for 10 Hz couplings



Current Data Parameters
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NAME Fin_4_500
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
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Time 2.47
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PULPROG ghmbc.wu
TD 4096
SOLVENT CDCl3
NS 8
DS 16
SWH 1750.700 Hz
FIDRES 0.427417 Hz
AQ 1.1698676 sec
RG 5792.6
DW 285.600 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
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 0.00004960 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p2 15.00 usec
PL1 1.60 dB
SFO1 500.2211510 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 15.50 usec
PL2 -1.00 dB
SFO2 125.7867080 MHz

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

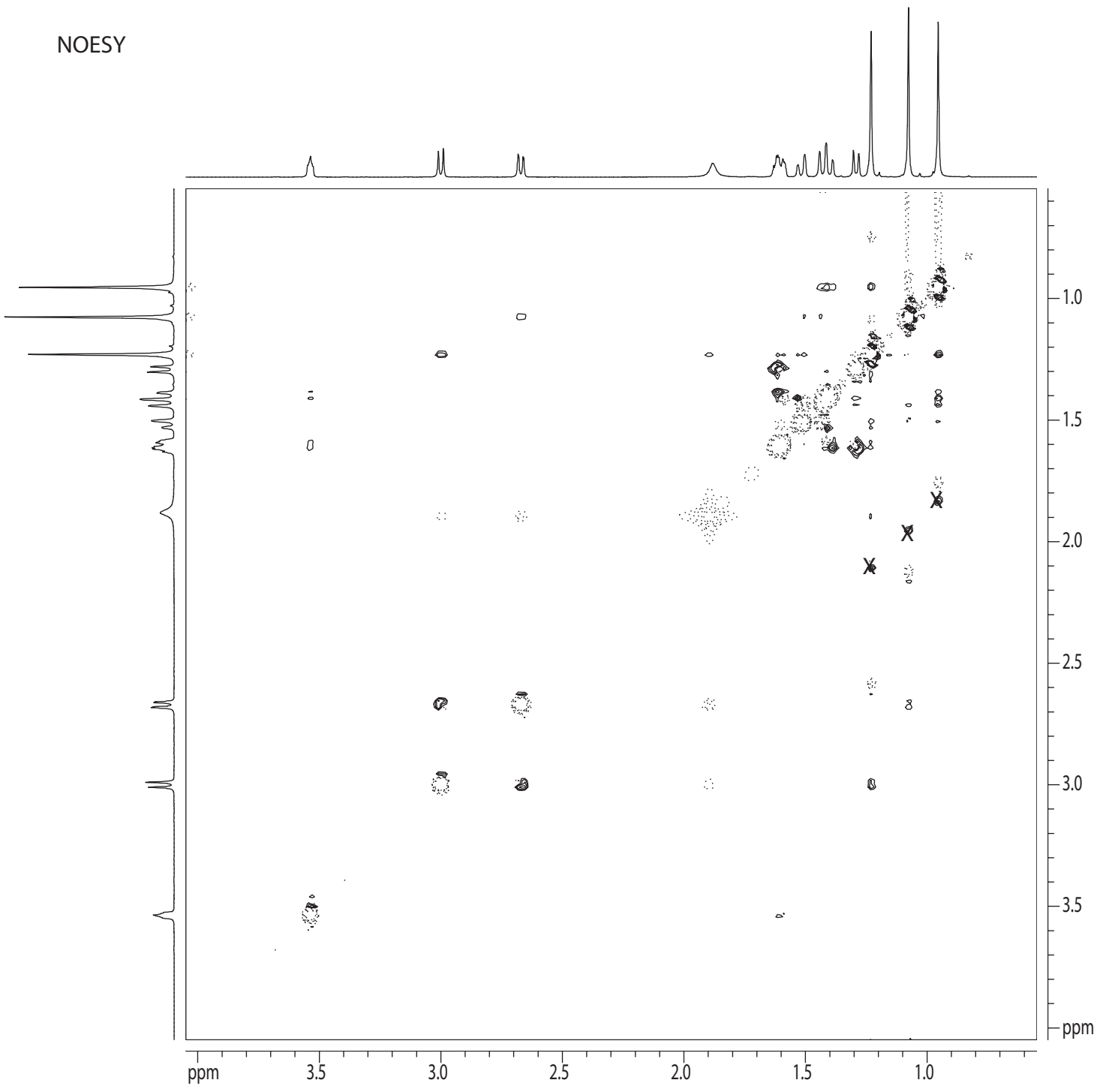
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7867 MHz
FIDRES 19.688761 Hz
SW 80.141 ppm
FnMODE undefined

F2 - Processing parameters
SI 2048
SF 500.2200013 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7804190 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 18.00 cm
CX1 15.00 cm
F2PLO 1.300 ppm
F2LO 650.45 Hz
F2PHI 0.900 ppm
F2HI 450.20 Hz
F1PLO 60.018 ppm
F1LO 7549.10 Hz
F1PHI 19.711 ppm
F1HI 2479.24 Hz
F2PPMCM 0.02224 ppm/cm
F2HZCM 11.12518 Hz/cm
F1PPMCM 2.68715 ppm/cm
F1HZCM 337.99039 Hz/cm

NOESY



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PROCNO 2

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PULPROG noesygptp
TD 2048
SOLVENT CDCl3
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DS 16
SWH 1750.700 Hz
FIDRES 0.854834 Hz
AQ 0.5849588 sec
RG 4
DW 285.600 usec
DE 6.00 usec
TE 298.0 K
D0 0.00000300 sec
D1 2.00000000 sec
D8 0.80000001 sec
D16 0.00020000 sec
d20 0.39880002 sec
IN0 0.00028560 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
P2 15.00 usec
PL1 1.60 dB
SFO1 500.2211510 MHz

====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 40.00 %
GPZ2 -40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 500.2212 MHz
FIDRES 3.419337 Hz
SW 3.500 ppm
FnMODE undefined

F2 - Processing parameters
SI 256
SF 500.2200013 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 256
MC2 TPPI
SF 500.2200013 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 4.048 ppm
F2LO 2025.02 Hz
F2PHI 0.548 ppm
F2HI 274.32 Hz
F1PLO 4.048 ppm
F1LO 2025.02 Hz
F1PHI 0.548 ppm
F1HI 274.32 Hz
F2PPMCM 0.23332 ppm/cm
F2HZCM 116.71336 Hz/cm
F1PPMCM 0.23332 ppm/cm
F1HZCM 116.71336 Hz/cm