Name: _____

Chem 203
December 20, 2014

Final Exam Part I
(40 points)

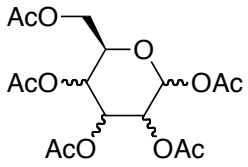
THIS FINAL EXAM CONSISTS OF PART I
AND TWO OUT OF THE THREE PROBLEMS FROM PART II

IF THREE PROBLEMS FROM PART II ARE SUBMITTED,
ONLY THE FIRST TWO (Part II, PROBLEMS 1-2) WILL BE GRADED

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, Maestro/MacroModel, Excel, ChemDoodle, Chemdraw, ElComp, MolE, etc.). Communication with other students by e-mail, text, or in person is not permitted. 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. INAPPROPRIATE COMMUNICATION OR 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.

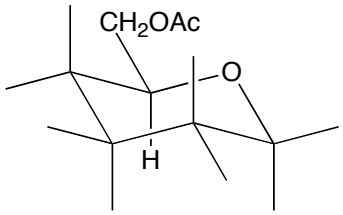
The following spectral data are provided for two diastereomeric L-hexopyranose pentaacetates, **A** and **B**: 500 MHz ^{1}H NMR, 125.8 MHz ^{13}C NMR, DEPT-90, DEPT-135, COSY, HMQC, NOESY, and a 1D NOE experiment. All NMR spectra were measured in C_6D_6 solution.



L-hexopyranose pentaacetate
(AcO = CH_3CO_2)

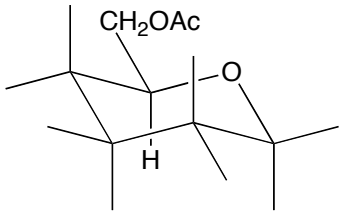
Using these data, determine the stereochemistry of diastereomers **A** and **B** and assign all of the ^{1}H resonances except those of the acetate groups (a-g) to the corresponding protons in the molecules.

a. Using the template below, write the structure of diastereomer **A**.



Explain how you determined the stereochemistry of the molecule.

b. Using the template below, write the structure of diastereomer **B**.



Explain how you determined the stereochemistry of the molecule.

c. Tabulate the shifts of the ^{1}H resonances a-g of diastereomer **A** 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:

d. Tabulate the shifts of the ^{1}H resonances a-g of diastereomer **B** 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:

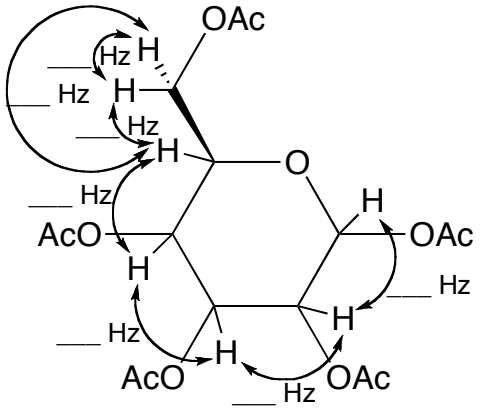
d:

e:

f:

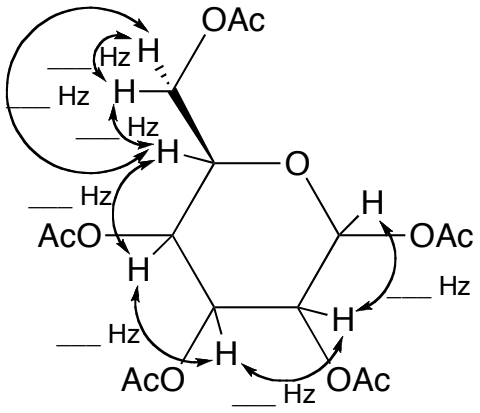
g:

e. For diastereomer **A**, write each geminal ($^2J_{HH}$) or vicinal ($^3J_{HH}$) coupling constant on the line by the corresponding arrow.



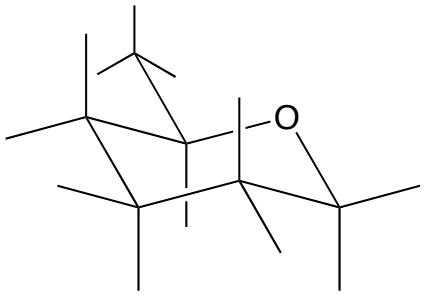
If you are uncertain about any of the coupling constants, please explain.

f. For diastereomer **B**, write each geminal ($^2J_{HH}$) or vicinal ($^3J_{HH}$) coupling constant on the line by the corresponding arrow.



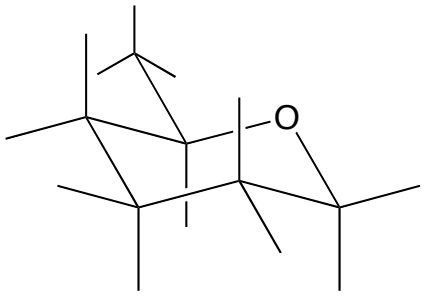
If you are uncertain about any of the coupling constants, please explain.

g. For diastereomer **A**, label the following template to show the positions of H_a, H_b, H_c, H_d, H_e, H_g, H_g, and the OAc groups. Show the orientation of the CH_2OAc group that is observed by NMR and correctly assign the diastereotopic CH_2 protons.



If you are uncertain about any of the assignments, please explain.

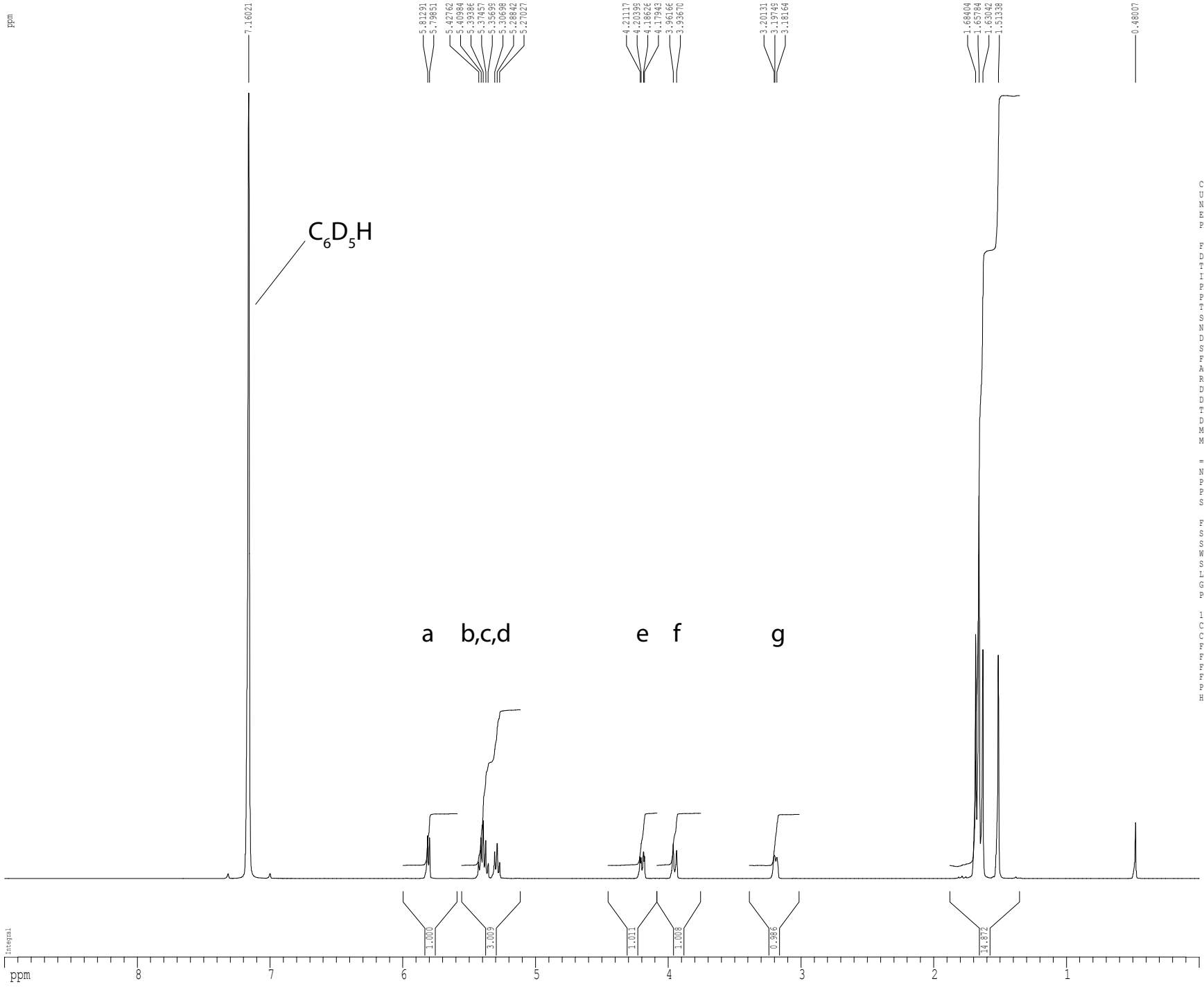
h. For diastereomer **B**, label the following template to show the positions of H_a, H_b, H_c, H_d, H_e, H_g, H_g, and the OAc groups. Show the orientation of the CH_2OAc group that is observed by NMR and correctly assign the diastereotopic CH_2 protons.



If you are uncertain about any of the assignments, please explain.

Diastereomer A

1H spectrum

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EXPNO                 9
PROCNO                1

F2 - Acquisition Parameters
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PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                160254
SOLVENT            CDCl3
NS                    64
DS                     2
SWH            8012.820 Hz
FIDRES         0.050001 Hz
AQ             9.9998999 sec
RG                     5
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          500.2200031 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                9.000 ppm
F1              4501.98 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          0.39474 ppm/cm
HZCM          197.45528 Hz/cm
```

Diastereomer A

DU=/v, USER=nmr14t, NAME=ACT_I_A, EXPNO=9, PROCNO=1
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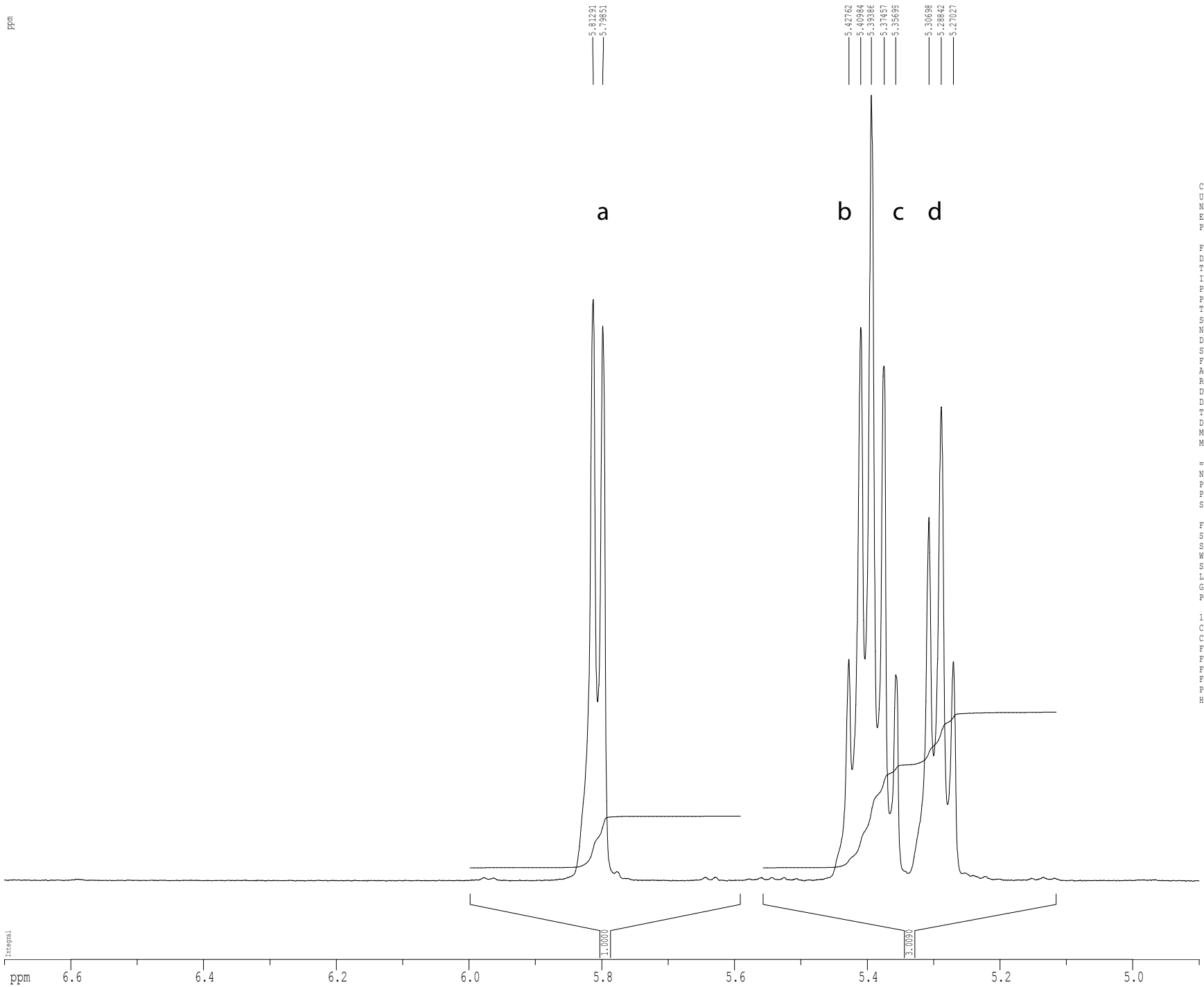
#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	32087.1	3581.681	7.1602	15.00
2	37599.3	2907.731	5.8129	0.82
3	37658.2	2900.529	5.7985	0.78
4	39175.6	2715.003	5.4276	0.31
5	39248.3	2706.113	5.4098	0.78
6	39313.7	2698.115	5.3939	1.11
7	39392.6	2688.469	5.3746	0.73
8	39464.6	2679.674	5.3570	0.29
9	39669.1	2654.660	5.3070	0.51
10	39745.1	2645.372	5.2884	0.67
11	39819.4	2636.295	5.2703	0.31
12	44152.4	2106.514	4.2112	0.43
13	44181.8	2102.919	4.2040	0.40
14	44254.3	2094.051	4.1863	0.51
15	44282.3	2090.634	4.1794	0.43
16	45173.2	1981.704	3.9617	0.67
17	45275.3	1969.218	3.9367	0.54
18	48284.0	1601.360	3.2013	0.45
19	48299.6	1599.446	3.1975	0.44
20	48364.5	1591.518	3.1816	0.41
21	54491.5	842.393	1.6840	4.66
22	54598.7	829.284	1.6578	7.31
23	54710.9	815.567	1.6304	4.37
24	55189.7	757.023	1.5134	4.27
25	59417.3	240.139	0.4801	1.07

Diastereomer A

1H spectrum



Current Data Parameters

USER	nmr14t
NAME	ACT_I_A
EXPNO	9
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
Time	9.34
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PULPROG	zg30
TD	160254
SOLVENT	CDCl3
NS	64
DS	2
SWH	8012.820 Hz
FIDRES	0.050001 Hz
AQ	9.9998999 sec
RG	5
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

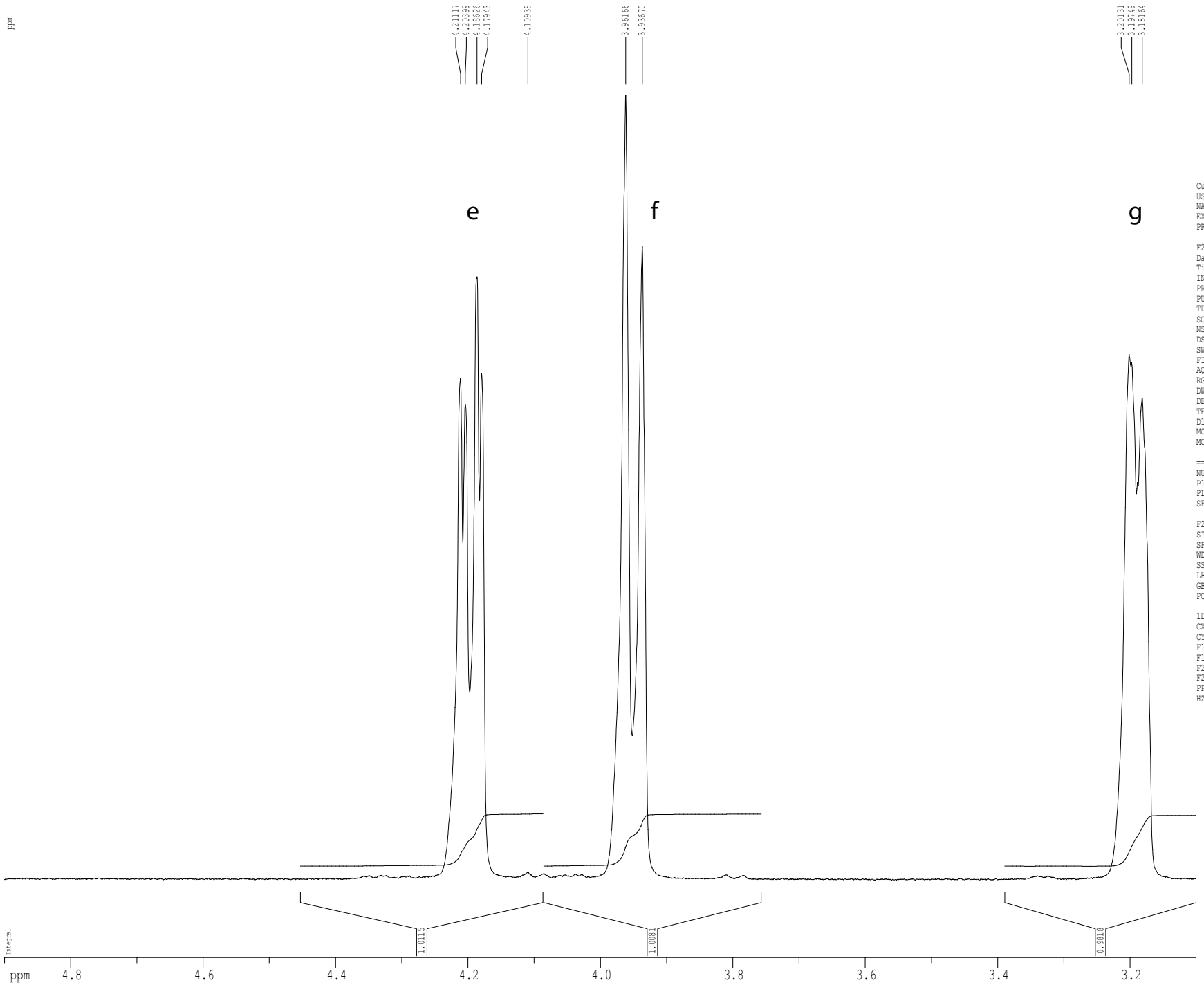
SI	65536
SF	500.2200031 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	6.700 ppm
F1	3351.47 Hz
F2P	4.900 ppm
F2	2451.08 Hz
PPMCM	0.07895 ppm/cm
HZCM	39.49105 Hz/cm

Diastereomer A

1H spectrum



Current Data Parameters

USER	nmr14t
NAME	ACT_I_A
EXPNO	9
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
Time	9.34
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	160254
SOLVENT	CDCl3
NS	64
DS	2
SWH	8012.820 Hz
FIDRES	0.050001 Hz
AQ	9.9998999 sec
RG	5
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

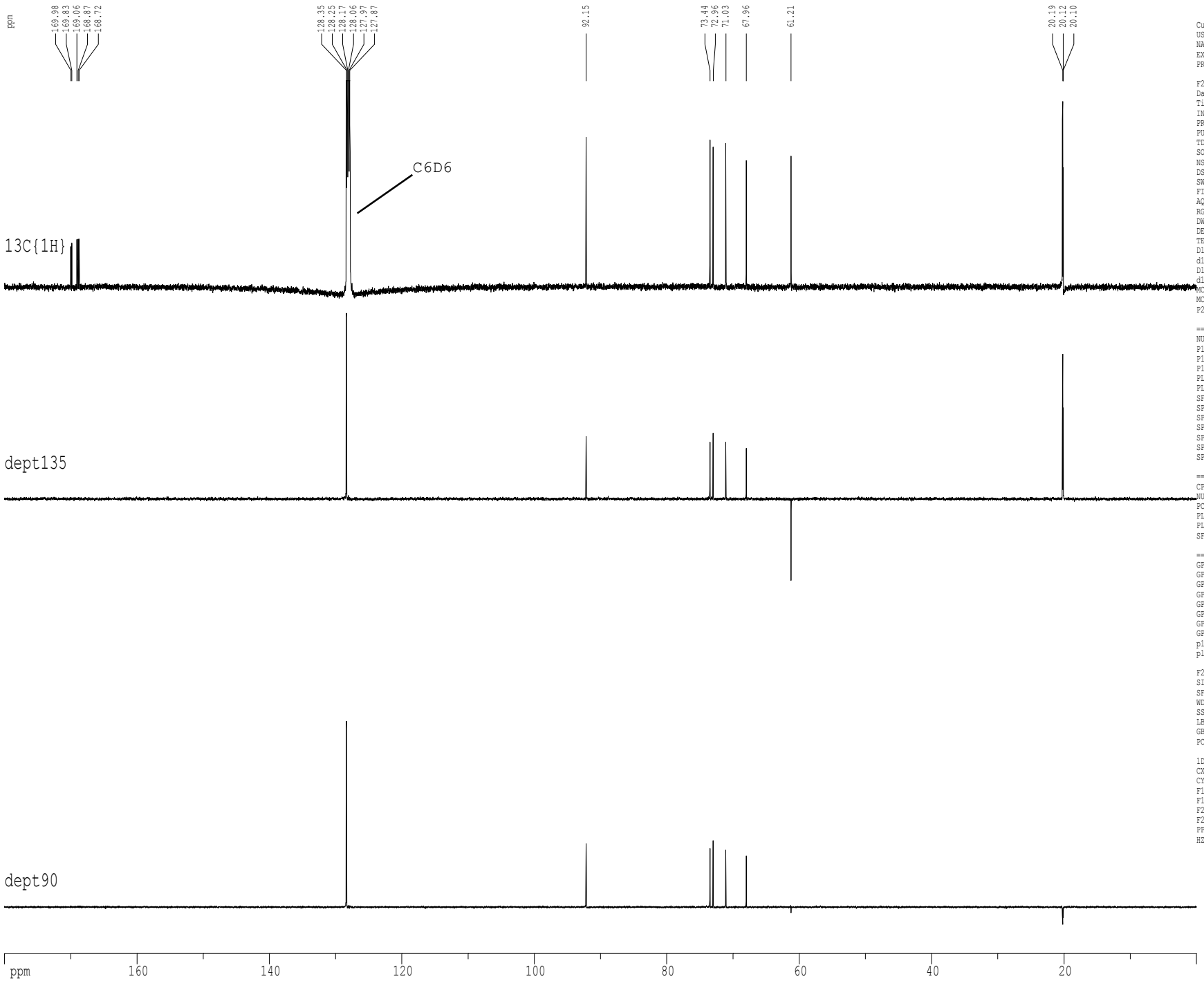
SI	65536
SF	500.2200031 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	4.900 ppm
F1	2451.08 Hz
F2P	3.100 ppm
F2	1550.68 Hz
PPMCM	0.07895 ppm/cm
HZCM	39.49106 Hz/cm

Diastereomer A

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



Current Data Parameters
USER nmr14t
NAME ACT_I_A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141219
Time 2.33
INSTRUM cryo500
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 3649.1
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
MCWRK 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
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2225011 MHz

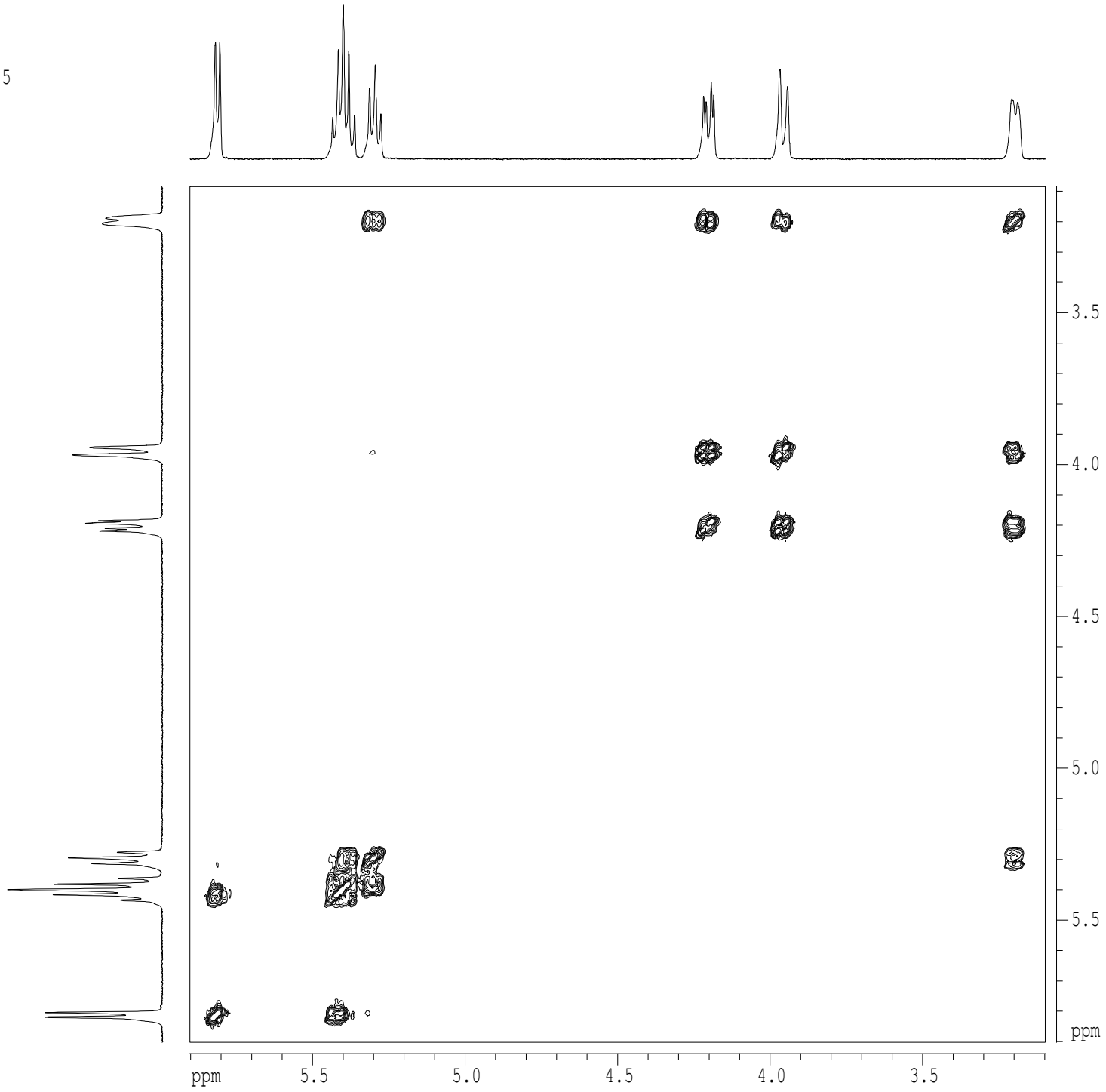
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803766 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 3.56 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 7.89474 ppm/cm
HZCM 993.00305 Hz/cm

Diastereomer A

gCOSY-45



Current Data Parameters

USER	nmr14t
NAME	ACT_I_A
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
Time	3.28
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	cosygp45.prd
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	5
DW	111.200 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	0.00022240 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2222500 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	500.2222 MHz
FIDRES	8.782037 Hz
SW	8.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

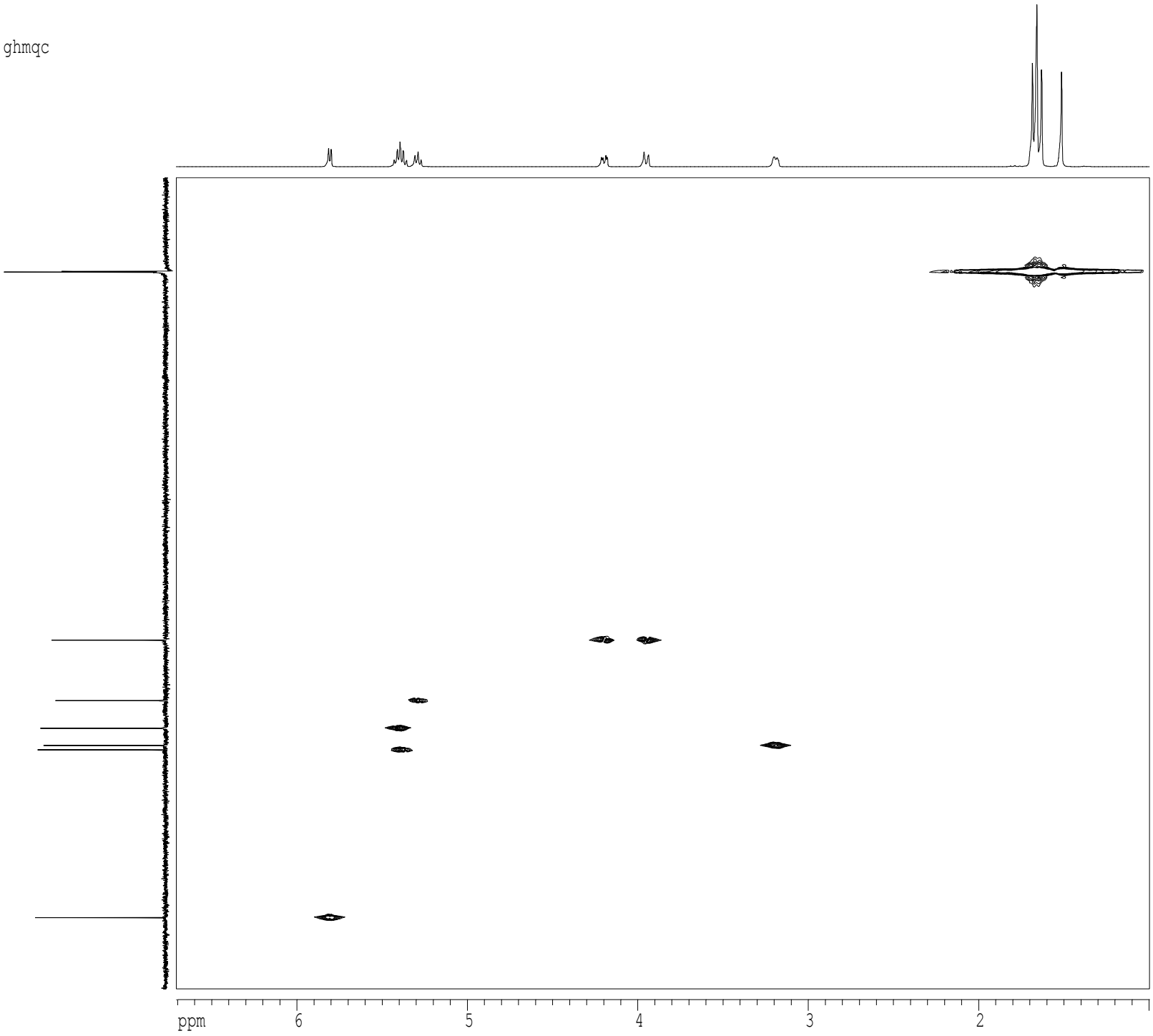
SI	1024
MC2	QF
SF	500.2200000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	5.903 ppm
F2LO	2952.56 Hz
F2PHI	3.098 ppm
F2HI	1549.63 Hz
F1PLO	5.903 ppm
F1LO	2952.56 Hz
F1PHI	3.085 ppm
F1HI	1543.05 Hz
F2PPMCM	0.18698 ppm/cm
F2HZCM	93.52869 Hz/cm
F1PPMCM	0.18785 ppm/cm
F1HZCM	93.96780 Hz/cm

Diastereomer A

ghmqc



Current Data Parameters

USER	nmr14t
NAME	ACT_I_A
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
Time	4.56
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	inv4gp.wu
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	11585.2
DW	111.200 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	0.00001988 sec

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

NUC1	1H
P1	7.50 usec
p2	15.00 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

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

CPDPRG2	garp
NUC2	13C
P3	15.50 usec
PCPD2	65.00 usec
PL2	-1.00 dB
PL12	11.30 dB
SFO2	125.7929190 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	49.135220 Hz
SW	199.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200031 MHz
WDW	EM
SSB	0
LB	7.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

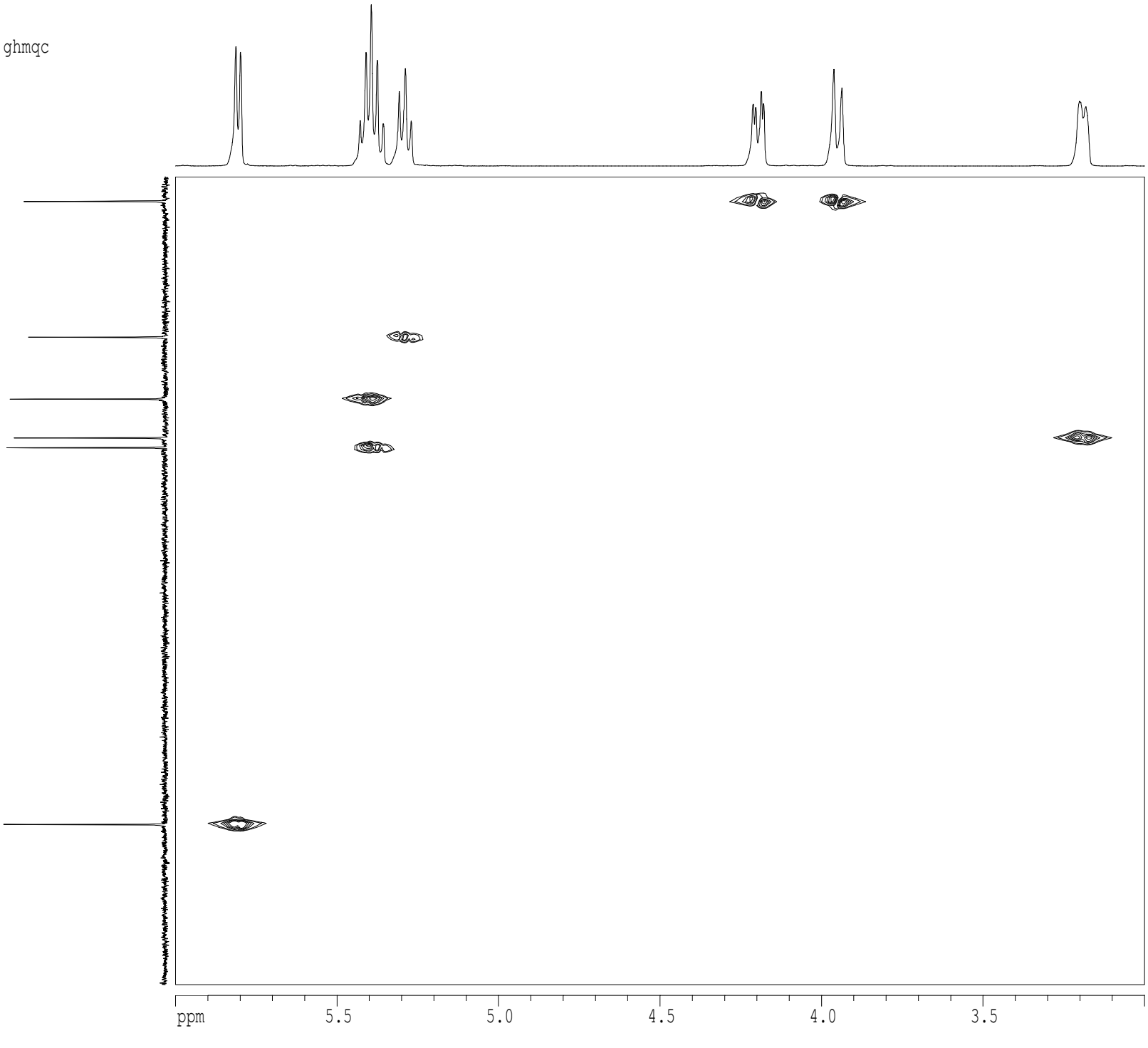
SI	1024
MC2	QF
SF	125.7803766 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	18.00 cm
CX1	15.00 cm
F2PLO	6.708 ppm
F2LO	3355.62 Hz
F2PHI	0.998 ppm
F2HI	499.26 Hz
F1PLO	100.107 ppm
F1LO	12591.54 Hz
F1PHI	9.674 ppm
F1HI	1216.74 Hz
F2PPMCM	0.31723 ppm/cm
F2HZCM	158.68654 Hz/cm
F1PPMCM	6.02892 ppm/cm
F1HZCM	758.32025 Hz/cm

Diastereomer A

ghmqc



Current Data Parameters

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NAME	ACT_I_A
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
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INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	inv4gp.wu
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	11585.2
DW	111.200 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	0.00001988 sec

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

NUC1	1H
P1	7.50 usec
p2	15.00 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

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

CPDPRG2	garp
NUC2	13C
P3	15.50 usec
PCPD2	65.00 usec
PL2	-1.00 dB
PL12	11.30 dB
SFO2	125.7929190 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	49.135220 Hz
SW	199.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200031 MHz
WDW	EM
SSB	0
LB	7.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

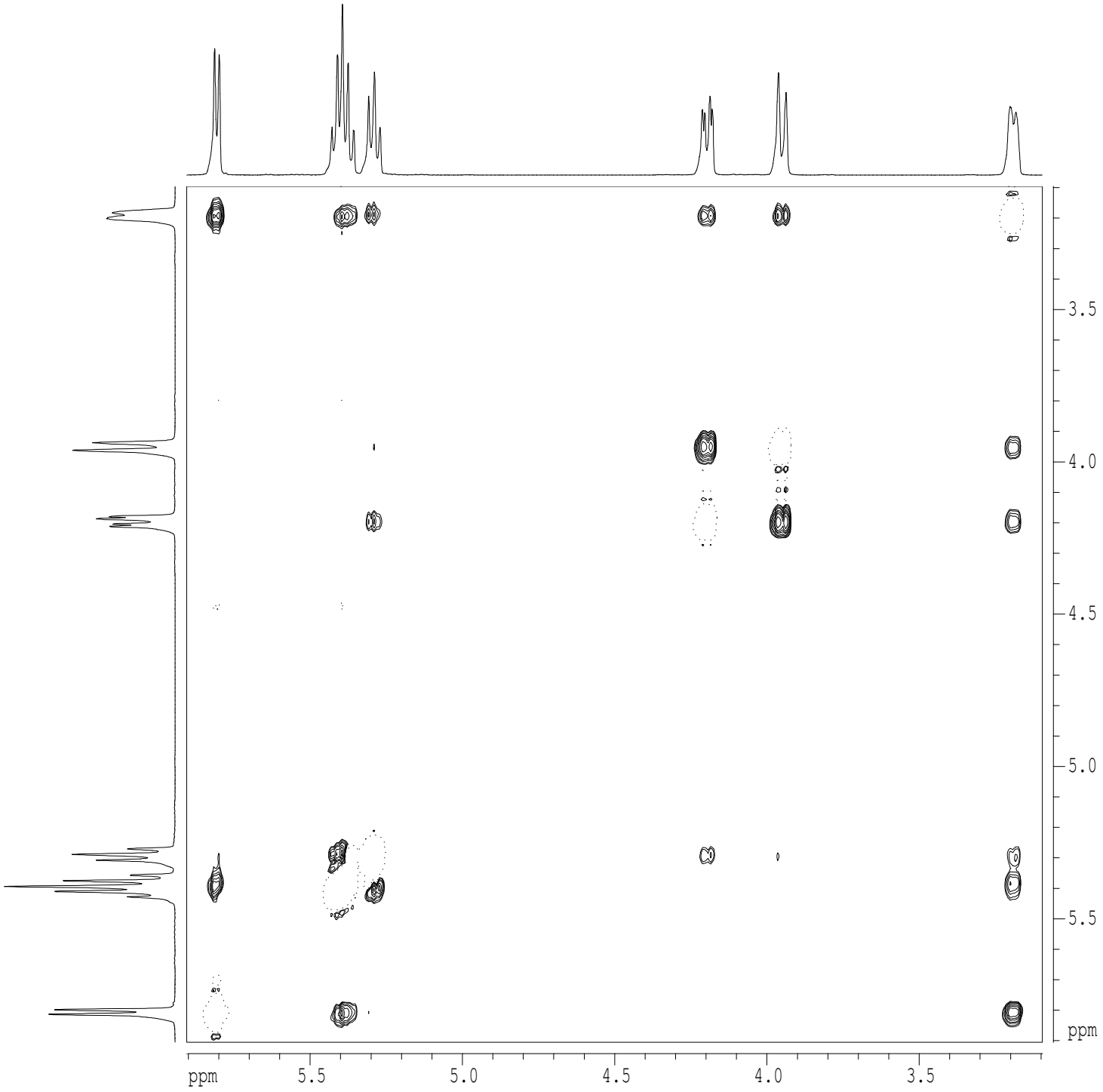
SI	1024
MC2	QF
SF	125.7803766 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	18.00 cm
CX1	15.00 cm
F2PLO	6.000 ppm
F2LO	3001.32 Hz
F2PHI	3.000 ppm
F2HI	1500.66 Hz
F1PLO	100.107 ppm
F1LO	12591.54 Hz
F1PHI	60.000 ppm
F1HI	7546.82 Hz
F2PPMCM	0.16667 ppm/cm
F2HZCM	83.37000 Hz/cm
F1PPMCM	2.67382 ppm/cm
F1HZCM	336.31445 Hz/cm

Diastereomer A

noesy



Current Data Parameters

USER	nmr14t
NAME	ACT_I_A
EXPNO	7
PROCNO	1

F2 - Acquisition Parameters

Date_	20141219
Time	6.24
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	noesyrvtp
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	5
DW	111.200 usec
DE	6.00 usec
TE	298.0 K
D0	0.00000300 sec
D1	2.00000000 sec
D8	2.00000000 sec
IN0	0.00011120 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	500.2222 MHz
FIDRES	17.564074 Hz
SW	8.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	1024
SF	500.2200031 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

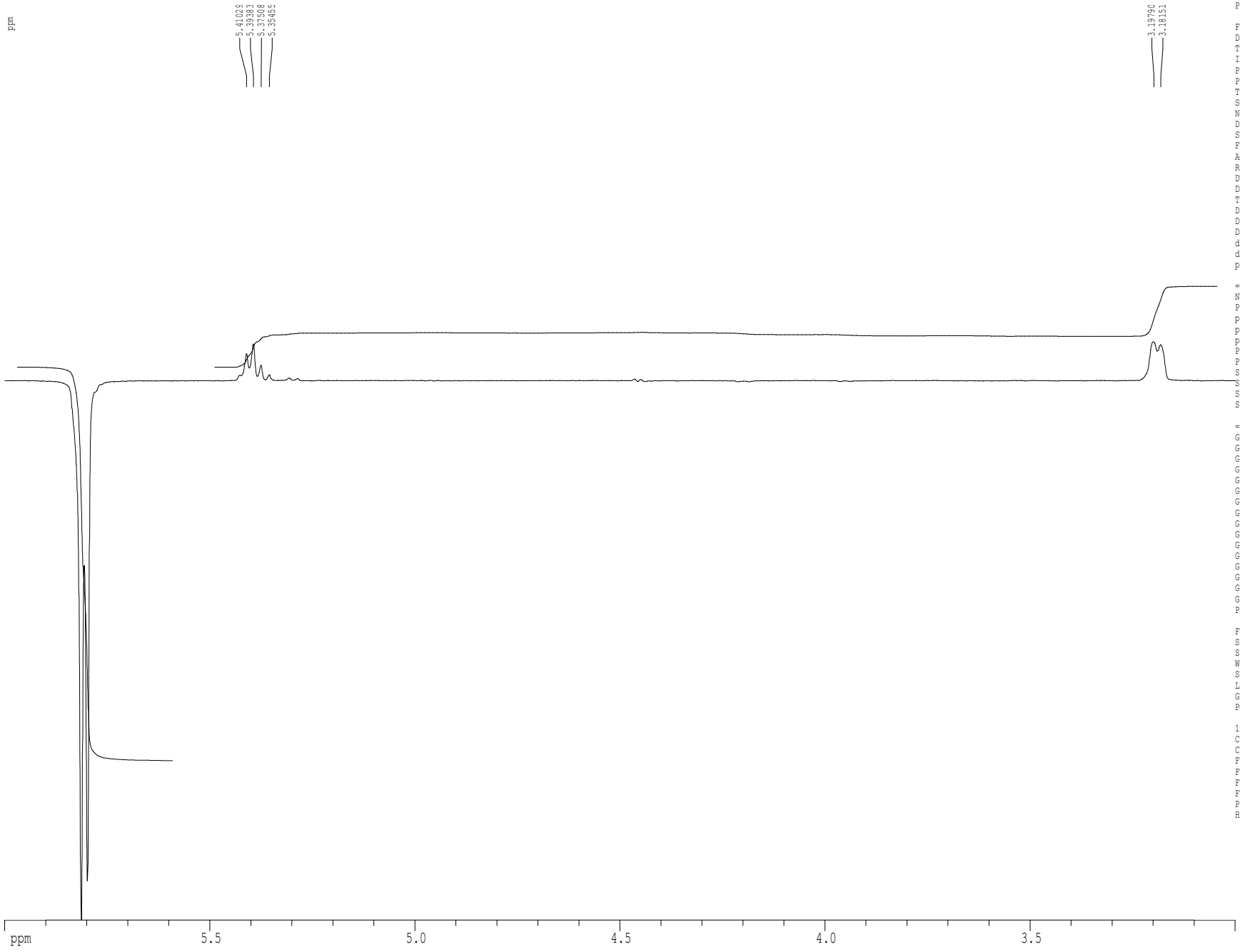
SI	1024
MC2	TPPI
SF	500.2200031 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	5.905 ppm
F2LO	2953.84 Hz
F2PHI	3.096 ppm
F2HI	1548.72 Hz
F1PLO	5.905 ppm
F1LO	2953.84 Hz
F1PHI	3.096 ppm
F1HI	1548.72 Hz
F2PPMCM	0.18727 ppm/cm
F2HZCM	93.67506 Hz/cm
F1PPMCM	0.18727 ppm/cm
F1HZCM	93.67506 Hz/cm

Diastereomer A

gnoe



Current Data Parameters
USER nmr14t
NAME ACT_I_A
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141219
Time 9.22
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG gnoelcc.wu
TD 65536
SOLVENT CDC13
NS 256
DS 8
SWH 7002.801 Hz
FIDRES 0.106854 Hz
AQ 4.6793203 sec
RG 10
DW 71.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D8 2.00000000 sec
D16 0.00020000 sec
d21 1.33876503 sec
d22 0.65899700 sec
p2 15.00 usec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p3 22.50 usec
p4 30.00 usec
p5 20.00 usec
P12 40000.00 usec
PL1 1.60 dB
SFO1 500.2229078 MHz
SP1 61.60 dB
SPNAM1 gauss1.512
SPOFF1 0.00 Hz

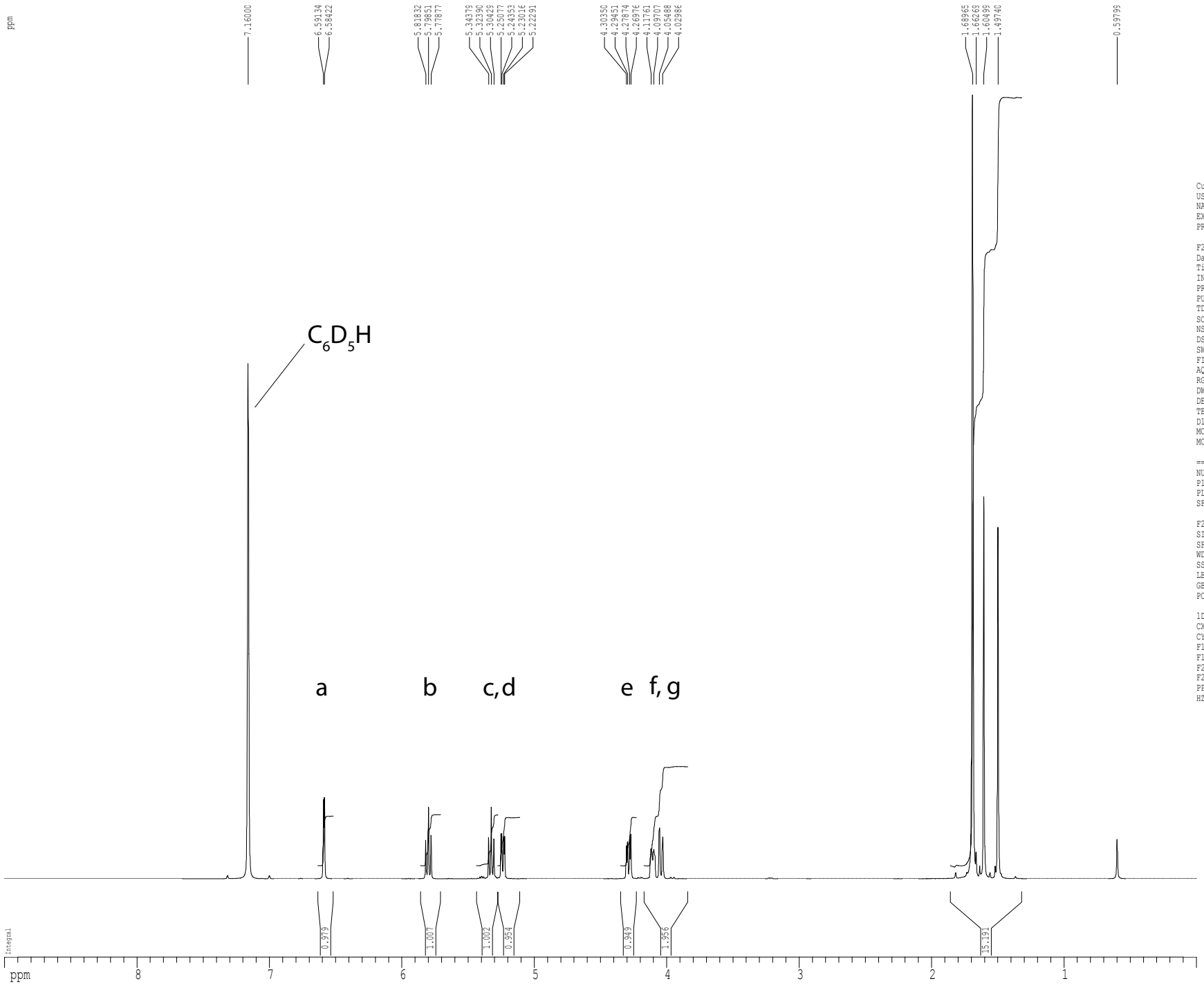
====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPNAM4 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPX4 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPY4 0.00 %
GPZ1 7.00 %
GPZ2 3.00 %
GPZ3 2.30 %
GPZ4 -2.30 %
P16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 500.2200031 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 6.000 ppm
F1 3001.32 Hz
F2P 3.000 ppm
F2 1500.66 Hz
PPMCM 0.13158 ppm/cm
HZCM 65.81842 Hz/cm

Diastereomer B

1H spectrum



Current Data Parameters
USER nmr14t
NAME ACT_I_B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141217
Time 21.22
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 160254
SOLVENT C6D6
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.050001 Hz
AQ 9.9998999 sec
RG 4.5
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 500.2200035 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 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm

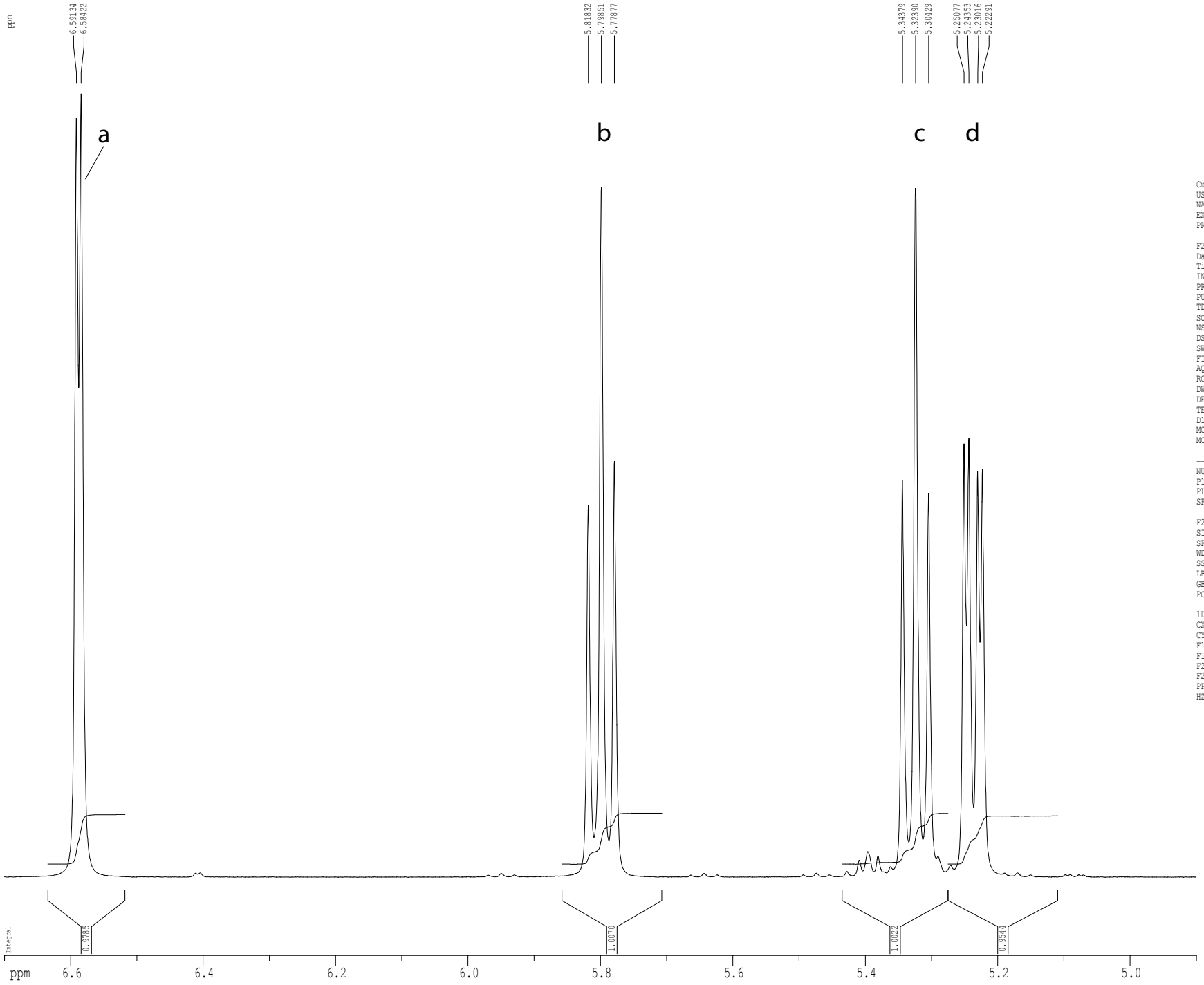
Diastereomer B

DU=/v, USER=nmr14t, NAME=ACT_I_B, EXPNO=1, PROCNO=1
F1=9.000ppm, F2=0.000ppm, MI=0.50cm, MAXI=10000.00cm, PC=4.000

#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	32085.0	3581.575	7.1600	9.86
2	34411.5	3297.119	6.5913	1.51
3	34440.6	3293.558	6.5842	1.56
4	37574.1	2910.438	5.8183	0.74
5	37655.2	2900.531	5.7985	1.37
6	37735.9	2890.657	5.7788	0.83
7	39515.5	2673.072	5.3438	0.79
8	39596.9	2663.121	5.3239	1.37
9	39677.2	2653.311	5.3043	0.76
10	39896.1	2626.539	5.2508	0.86
11	39925.8	2622.916	5.2435	0.87
12	39980.4	2616.229	5.2302	0.81
13	40010.1	2612.603	5.2229	0.81
14	43771.6	2152.696	4.3035	0.64
15	43808.4	2148.202	4.2945	0.72
16	43872.9	2140.313	4.2787	0.78
17	43909.7	2135.819	4.2698	0.86
18	44532.2	2059.709	4.1176	0.58
19	44616.2	2049.438	4.0971	0.57
20	44788.8	2028.333	4.0549	0.98
21	44891.2	2015.814	4.0299	0.80
22	54465.5	845.195	1.6896	15.00
23	54575.8	831.711	1.6627	0.51
24	54811.9	802.848	1.6050	7.31
25	55252.1	749.030	1.4974	6.73
26	58931.8	299.127	0.5980	0.75

Diastereomer B

1H spectrum




```
Current Data Parameters
USER                nmr14t
NAME              ACT_I_B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20141217
Time                21.22
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG               zg30
TD                  160254
SOLVENT               C6D6
NS                      64
DS                       2
SWH              8012.820 Hz
FIDRES           0.050001 Hz
AQ               9.9998999 sec
RG                     4.5
DW                62.400 usec
DE                 6.00 usec
TE                 298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

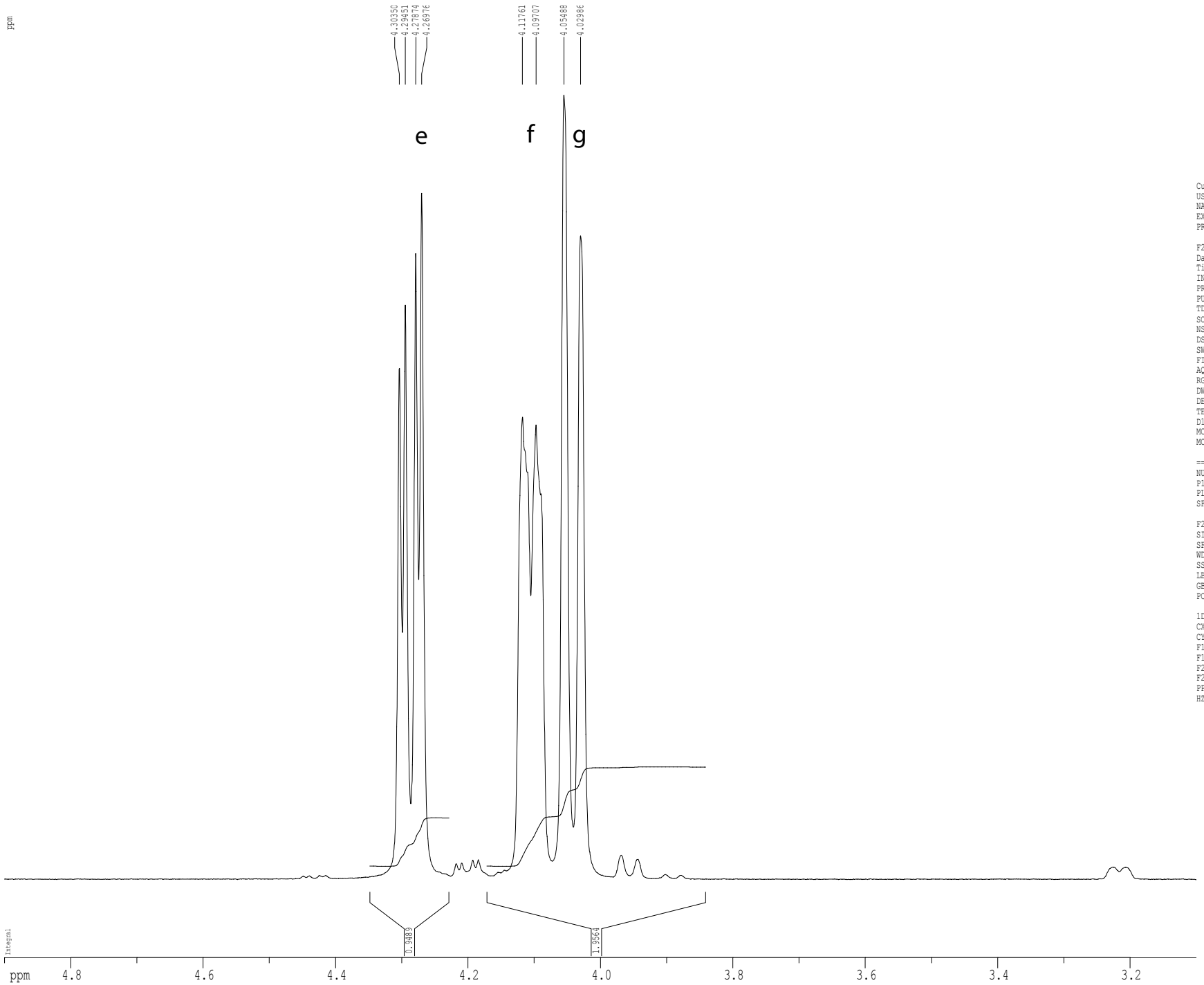
======== CHANNEL f1 ========
NUC1                    1H
P1                   7.50 usec
PL1                  1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                    65536
SF            500.2200035 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                   6.700 ppm
F1                 3351.47 Hz
F2P                   4.900 ppm
F2                 2451.08 Hz
PPMCM             0.07895 ppm/cm
HZCM             39.49105 Hz/cm
```

Diastereomer B

1H spectrum




```
Current Data Parameters
USER              nmr14t
NAME            ACT_I_B
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20141217
Time               21.22
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                160254
SOLVENT             C6D6
NS                    64
DS                     2
SWH            8012.820 Hz
FIDRES         0.050001 Hz
AQ             9.9998999 sec
RG                   4.5
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

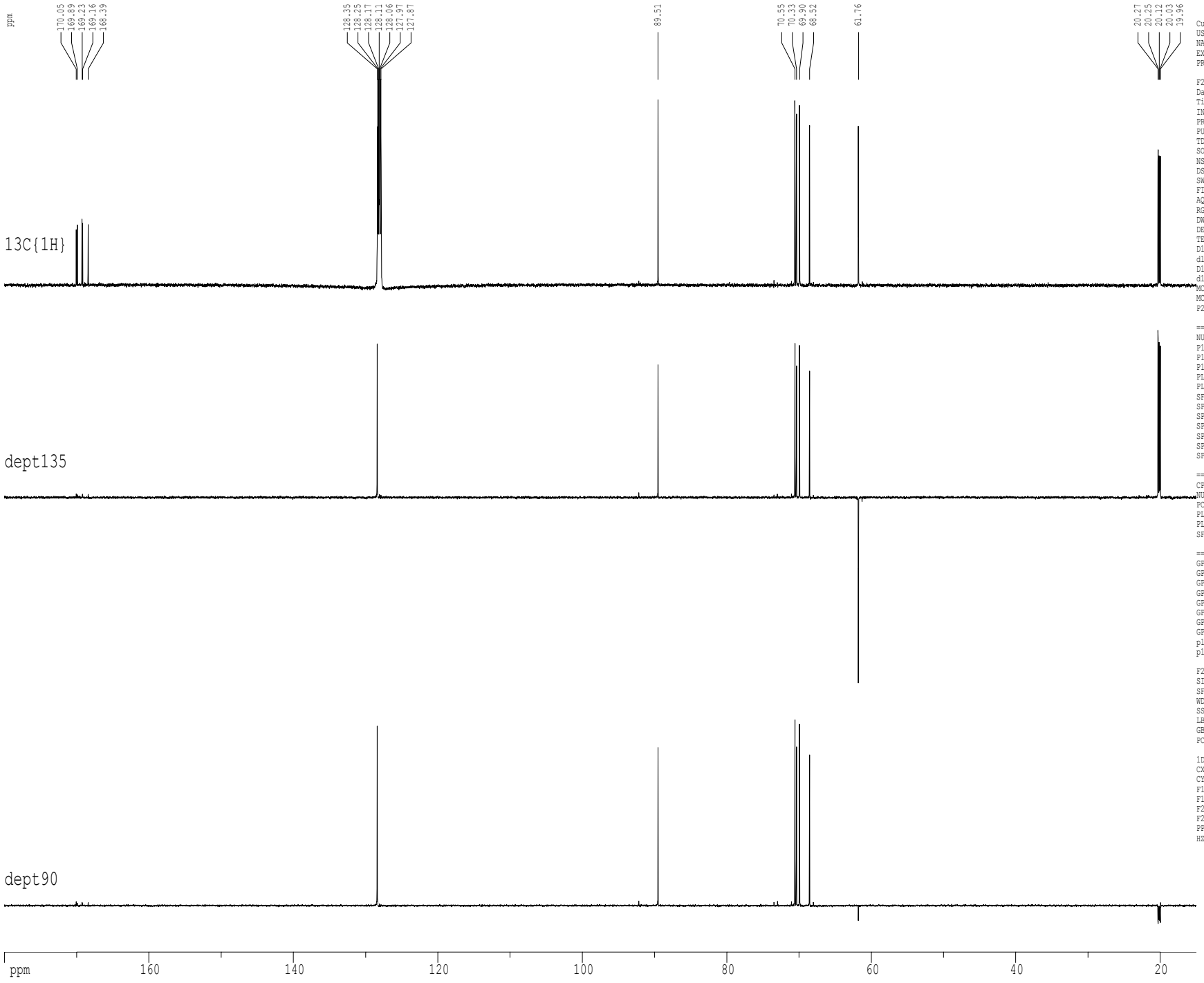
======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1                1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF          500.2200035 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               4.900 ppm
F1              2451.08 Hz
F2P               3.100 ppm
F2              1550.68 Hz
PPMCM          0.07895 ppm/cm
HZCM          39.49106 Hz/cm
```

Diastereomer B

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



Current Data Parameters
USER nmr14t
NAME ACT_I_B
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141217
Time 21.43
INSTRUM cryo500
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 2580.3
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
MCWRK 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
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.50 dB
SFO2 500.2225011 MHz

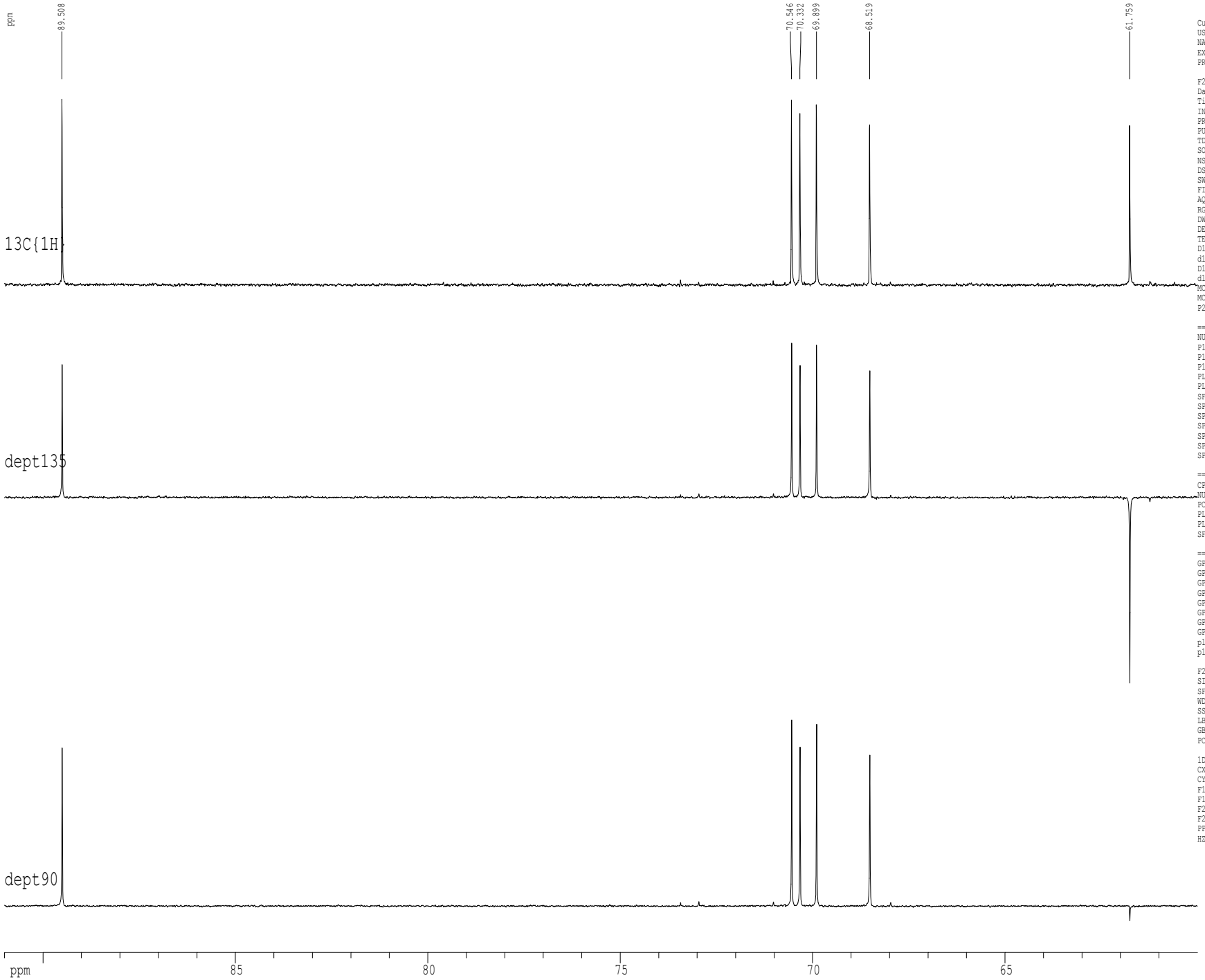
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803766 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 3.56 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P 15.000 ppm
F2 1886.71 Hz
PPMCM 7.23684 ppm/cm
HZCM 910.25269 Hz/cm

Diastereomer B

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




```
Current Data Parameters
USER              nmr14t
NAME            ACT_I_B
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20141217
Time              21.43
INSTRUM         cryo500
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              2580.3
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
MCWRK       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
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2          100.00 usec
PL2             1.60 dB
PL12           24.50 dB
SFO2       500.2225011 MHz

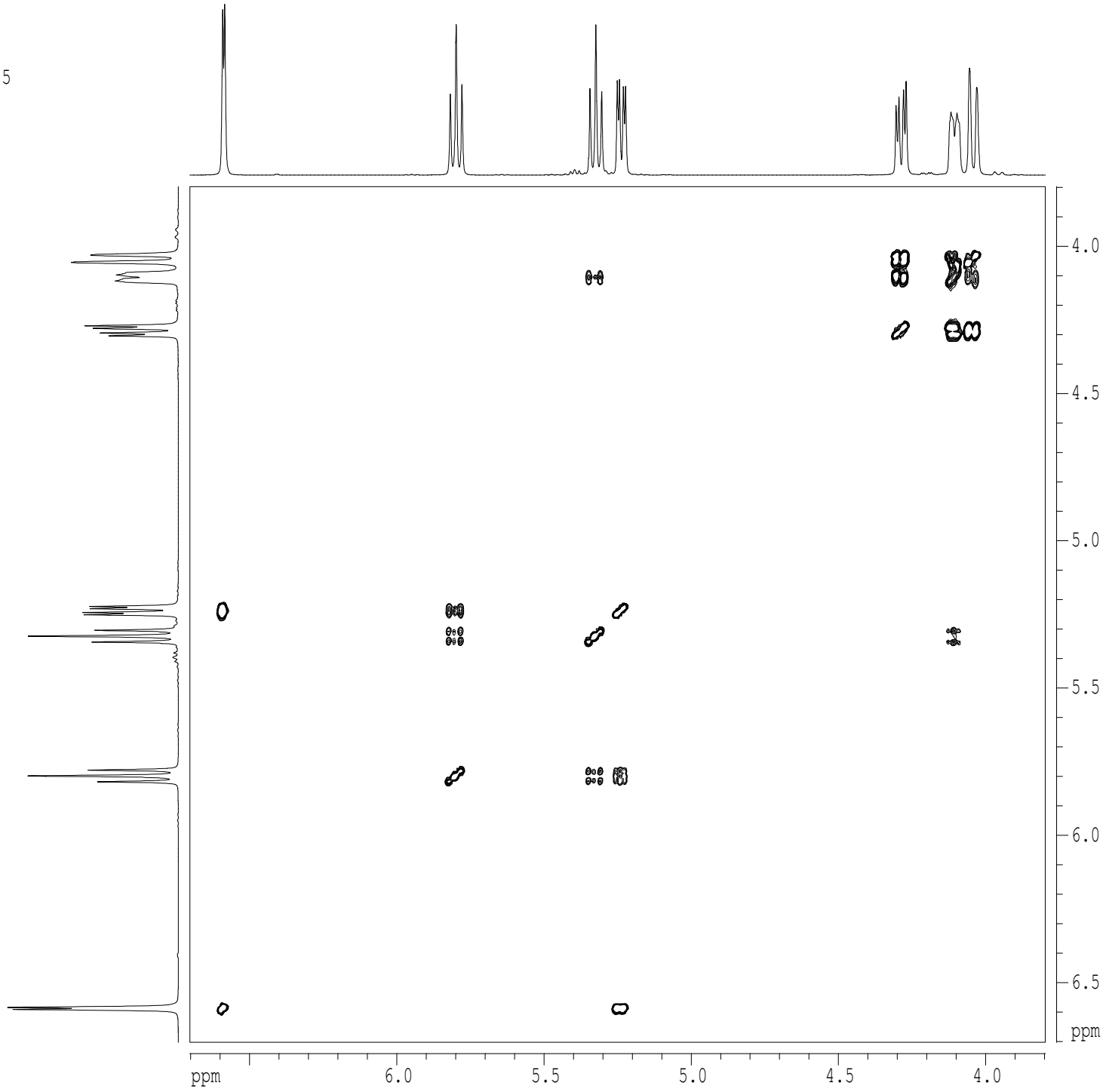
====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1              0.00 %
GPX2              0.00 %
GPY1              0.00 %
GPY2              0.00 %
GPZ1             30.00 %
GPZ2             50.00 %
p15            500.00 usec
p16           1000.00 usec

F2 - Processing parameters
SI                65536
SF          125.7803766 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                 3.56 cm
F1P              91.000 ppm
F1             11446.02 Hz
F2P              60.000 ppm
F2              7546.82 Hz
PPMCM           1.35965 ppm/cm
HZCM          171.01720 Hz/cm
```

Diastereomer B

gCOSY-45



Current Data Parameters

USER	nmr14t
NAME	ACT_I_B
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20141217
Time	22.57
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	cosygp45.prd
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	4.5
DW	111.200 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	0.00022240 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2222500 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	500.2222 MHz
FIDRES	8.782037 Hz
SW	8.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200035 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

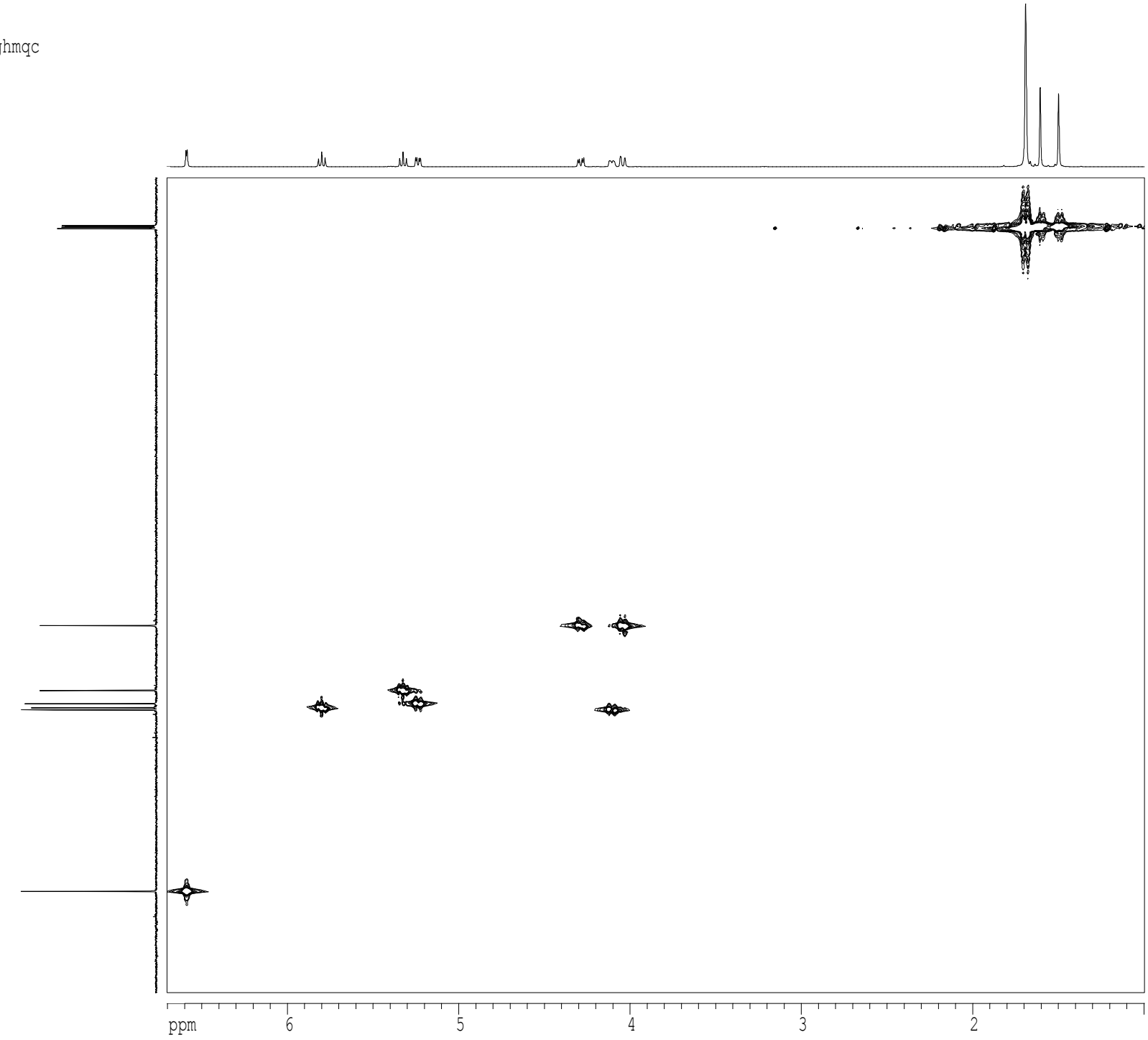
SI	1024
MC2	QF
SF	500.2200035 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	6.703 ppm
F2LO	3353.07 Hz
F2PHI	3.798 ppm
F2HI	1899.64 Hz
F1PLO	6.703 ppm
F1LO	3353.07 Hz
F1PHI	3.798 ppm
F1HI	1899.64 Hz
F2PPMCM	0.19371 ppm/cm
F2HZCM	96.89514 Hz/cm
F1PPMCM	0.19371 ppm/cm
F1HZCM	96.89514 Hz/cm

Diastereomer B

ghmqc



Current Data Parameters

USER	nmr14t
NAME	ACT_I_B
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20141218
Time	0.25
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	inv4gp.wu
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	11585.2
DW	111.200 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	0.00001988 sec

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

NUC1	1H
P1	7.50 usec
p2	15.00 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

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

CPDPRG2	garp
NUC2	13C
P3	15.50 usec
PCPD2	65.00 usec
PL2	-1.00 dB
PL12	11.30 dB
SFO2	125.7929190 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	49.135220 Hz
SW	199.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200035 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

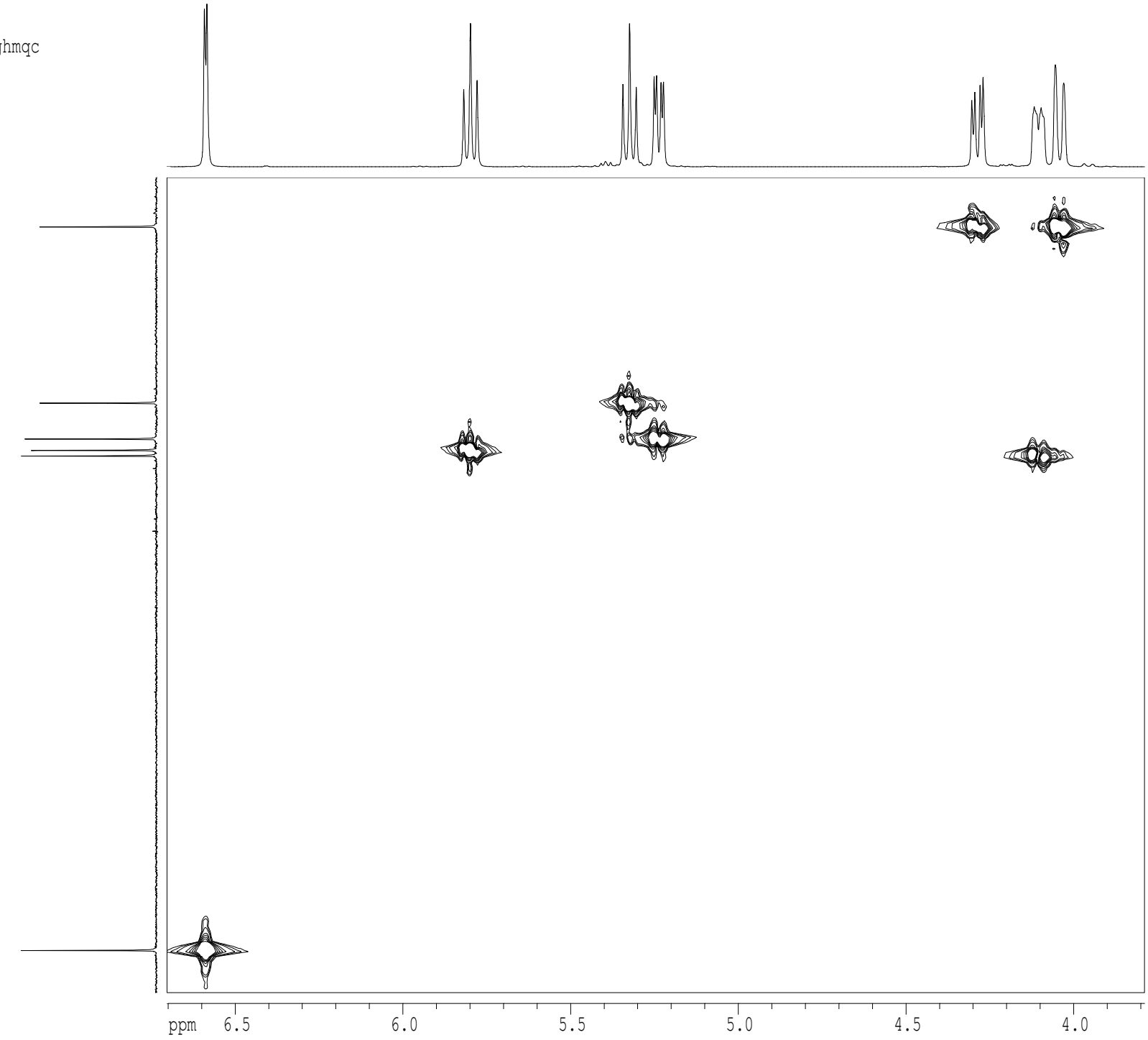
SI	1024
MC2	QF
SF	125.7803766 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	18.00 cm
CX1	15.00 cm
F2PLO	6.703 ppm
F2LO	3353.07 Hz
F2PHI	0.997 ppm
F2HI	498.90 Hz
F1PLO	100.107 ppm
F1LO	12591.54 Hz
F1PHI	14.947 ppm
F1HI	1880.06 Hz
F2PPMCM	0.31699 ppm/cm
F2HZCM	158.56456 Hz/cm
F1PPMCM	5.67734 ppm/cm
F1HZCM	714.09857 Hz/cm

Diastereomer B

ghmqc



Current Data Parameters

USER	nmr14t
NAME	ACT_I_B
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20141218
Time	0.25
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	inv4gp.wu
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	11585.2
DW	111.200 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	0.00001988 sec

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

NUC1	1H
P1	7.50 usec
p2	15.00 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

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

CPDPRG2	garp
NUC2	13C
P3	15.50 usec
PCPD2	65.00 usec
PL2	-1.00 dB
PL12	11.30 dB
SFO2	125.7929190 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	49.135220 Hz
SW	199.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200035 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

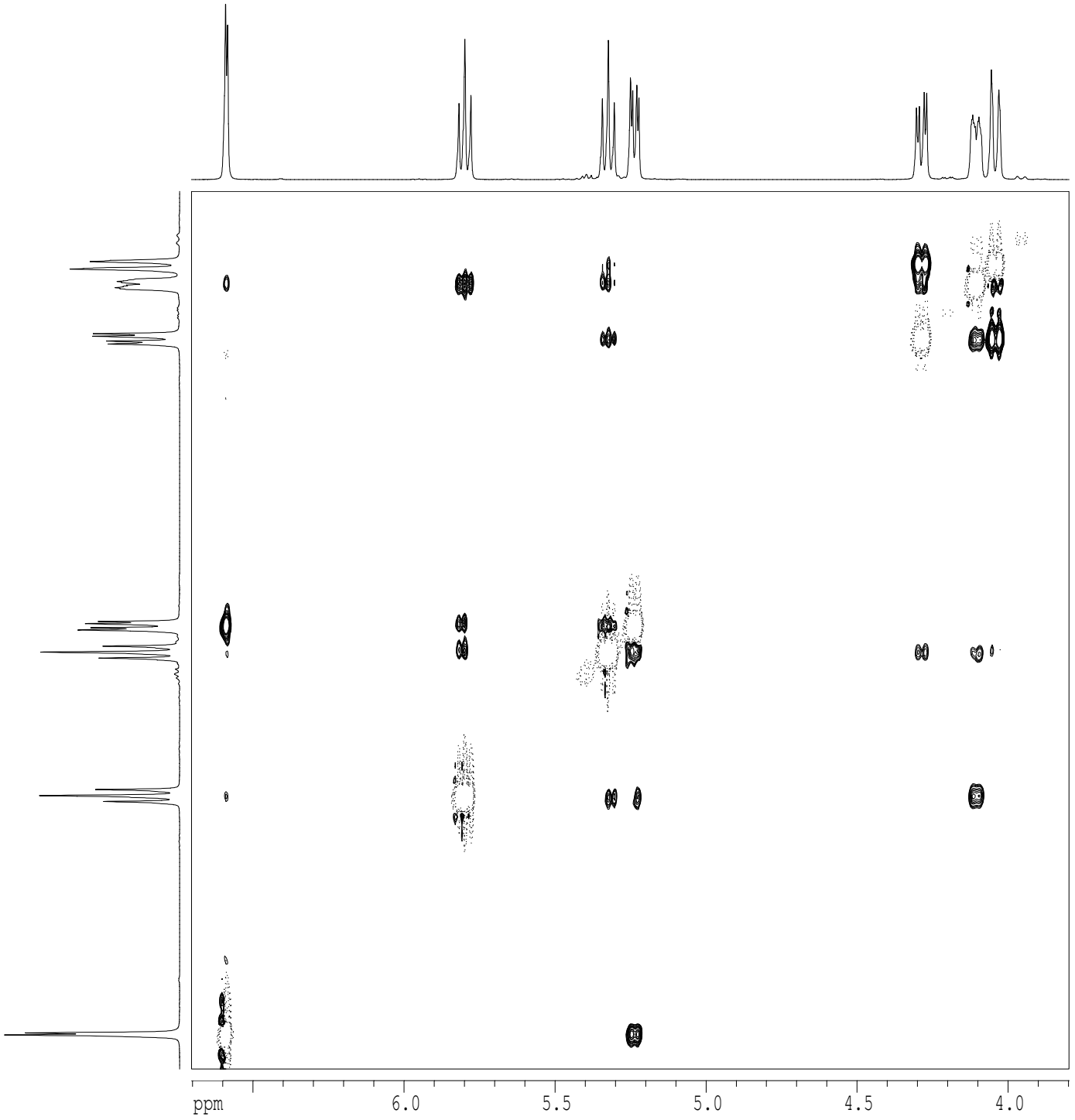
SI	1024
MC2	QF
SF	125.7803766 MHz
WDW	QSINE
SSB	3
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	18.00 cm
CX1	15.00 cm
F2PLO	6.703 ppm
F2LO	3353.07 Hz
F2PHI	3.789 ppm
F2HI	1895.25 Hz
F1PLO	91.123 ppm
F1LO	11461.43 Hz
F1PHI	59.871 ppm
F1HI	7530.61 Hz
F2PPMCM	0.16191 ppm/cm
F2HZCM	80.98989 Hz/cm
F1PPMCM	2.08343 ppm/cm
F1HZCM	262.05450 Hz/cm

Diastereomer B

noesy



Current Data Parameters

USER	nmr14t
NAME	ACT_I_B
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20141218
Time	21.57
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	noesyrvtp
TD	2048
SOLVENT	C6D6
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	5
DW	111.200 usec
DE	6.00 usec
TE	298.0 K
D0	0.00000300 sec
D1	2.00000000 sec
D8	2.00000000 sec
IN0	0.00011120 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2222500 MHz

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	500.2222 MHz
FIDRES	17.564074 Hz
SW	8.989 ppm
FnMODE	undefined

F2 - Processing parameters

SI	2048
SF	500.2200033 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	4.00

F1 - Processing parameters

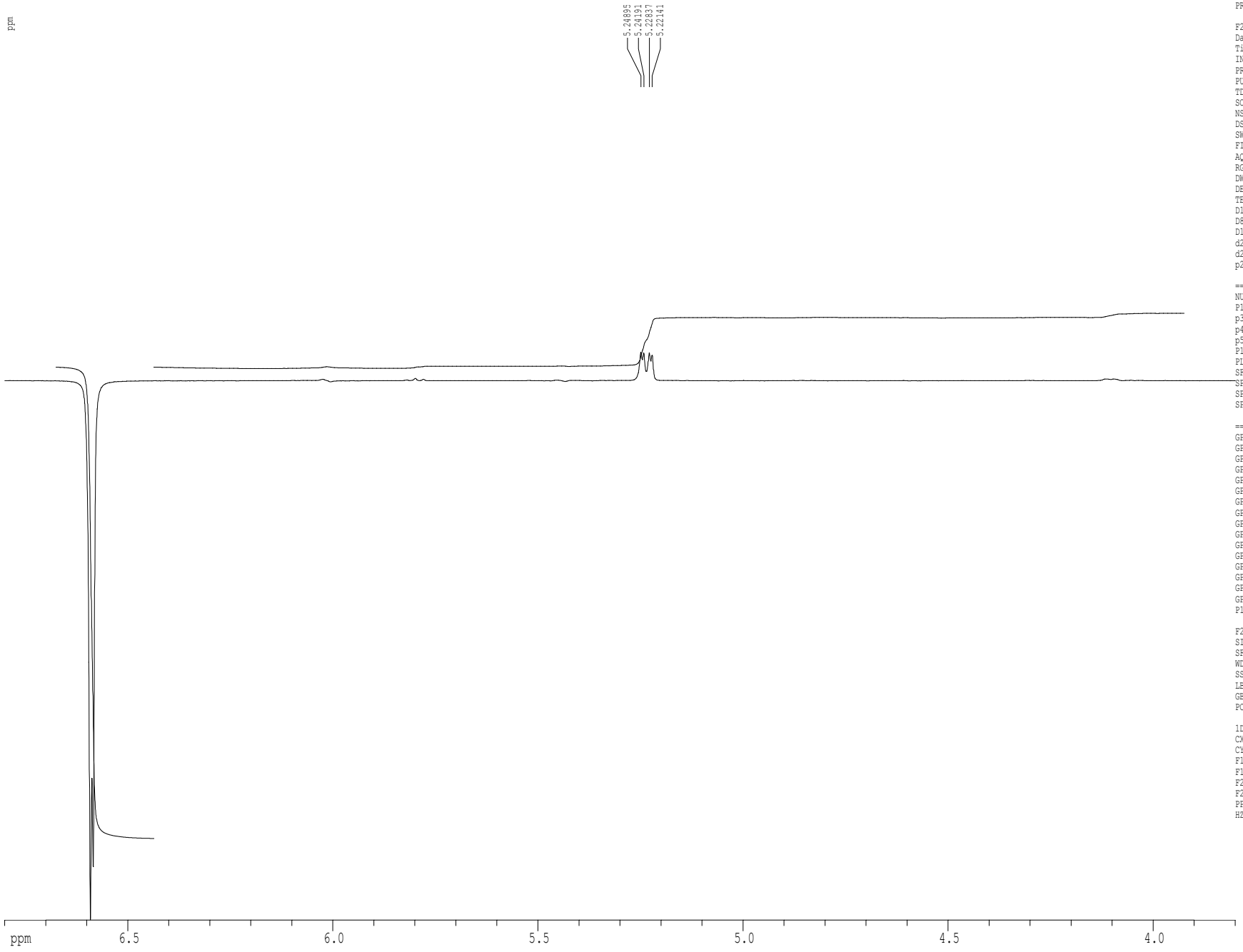
SI	1024
MC2	TPPI
SF	500.2200033 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	6.704 ppm
F2LO	3353.28 Hz
F2PHI	3.798 ppm
F2HI	1899.85 Hz
F1PLO	6.704 ppm
F1LO	3353.28 Hz
F1PHI	3.798 ppm
F1HI	1899.85 Hz
F2PPMCM	0.19371 ppm/cm
F2HZCM	96.89514 Hz/cm
F1PPMCM	0.19371 ppm/cm
F1HZCM	96.89514 Hz/cm

Diastereomer B

gnoe



Current Data Parameters
USER nmr14t
NAME ACT_I_B
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141219
Time 0.56
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG gnoelcc.wu
TD 65536
SOLVENT C6D6
NS 256
DS 8
SWH 7002.801 Hz
FIDRES 0.106854 Hz
AQ 4.6793203 sec
RG 5
DW 71.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D8 2.00000000 sec
D16 0.00020000 sec
d21 1.33876503 sec
d22 0.65899700 sec
p2 15.00 usec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p3 22.50 usec
p4 30.00 usec
p5 20.00 usec
P12 40000.00 usec
PL1 1.60 dB
SFO1 500.2232989 MHz
SP1 61.60 dB
SPNAM1 gauss1.512
SPOFF1 0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPNAM4 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPX4 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPY4 0.00 %
GPZ1 7.00 %
GPZ2 3.00 %
GPZ3 2.30 %
GPZ4 -2.30 %
P16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 500.2200033 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 6.800 ppm
F1 3401.50 Hz
F2P 3.800 ppm
F2 1900.84 Hz
PPMCM 0.13158 ppm/cm
HZCM 65.81843 Hz/cm