Design, Synthesis and Functional Activity of Labeled CD1d Glycolipid Agonists

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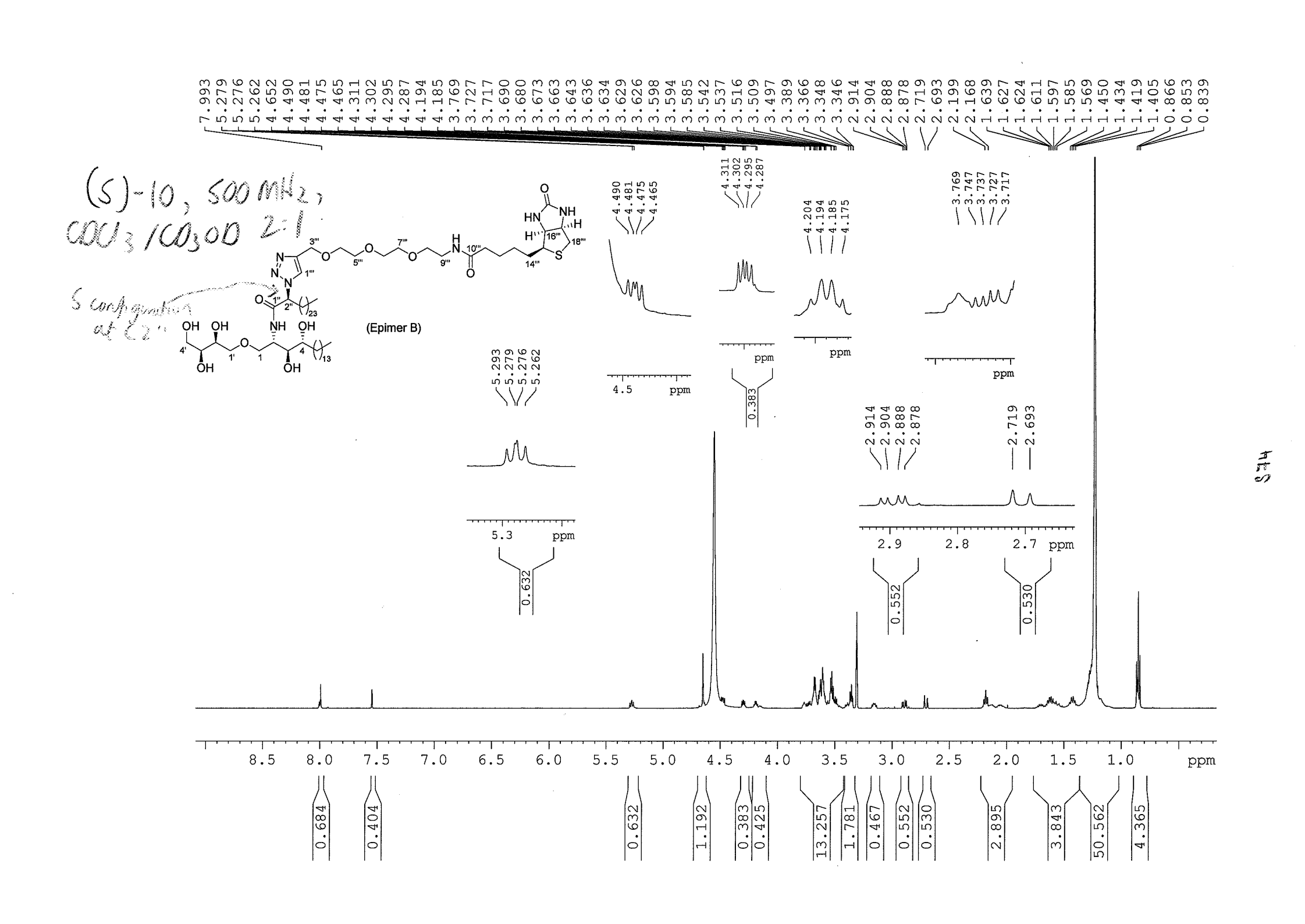
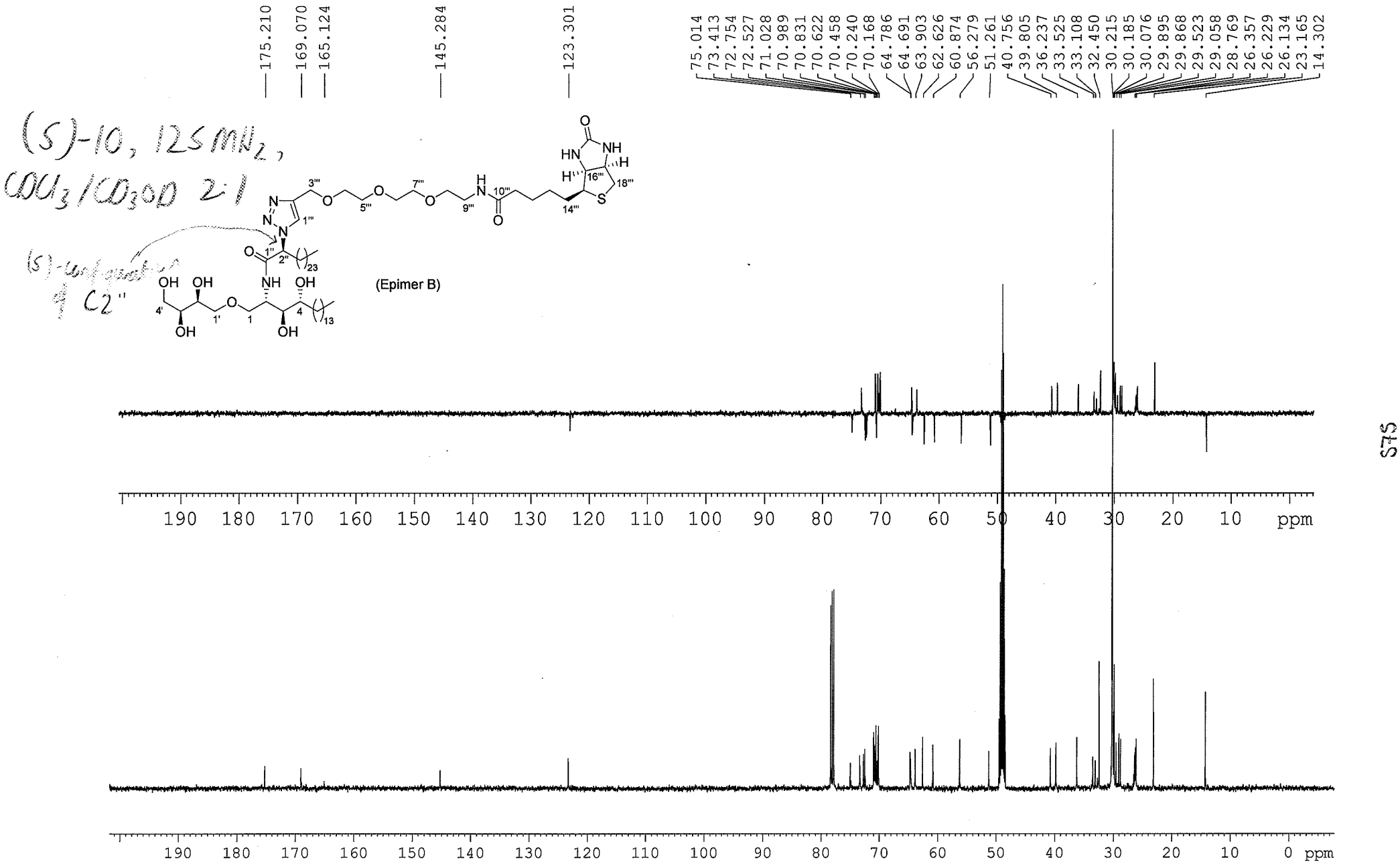
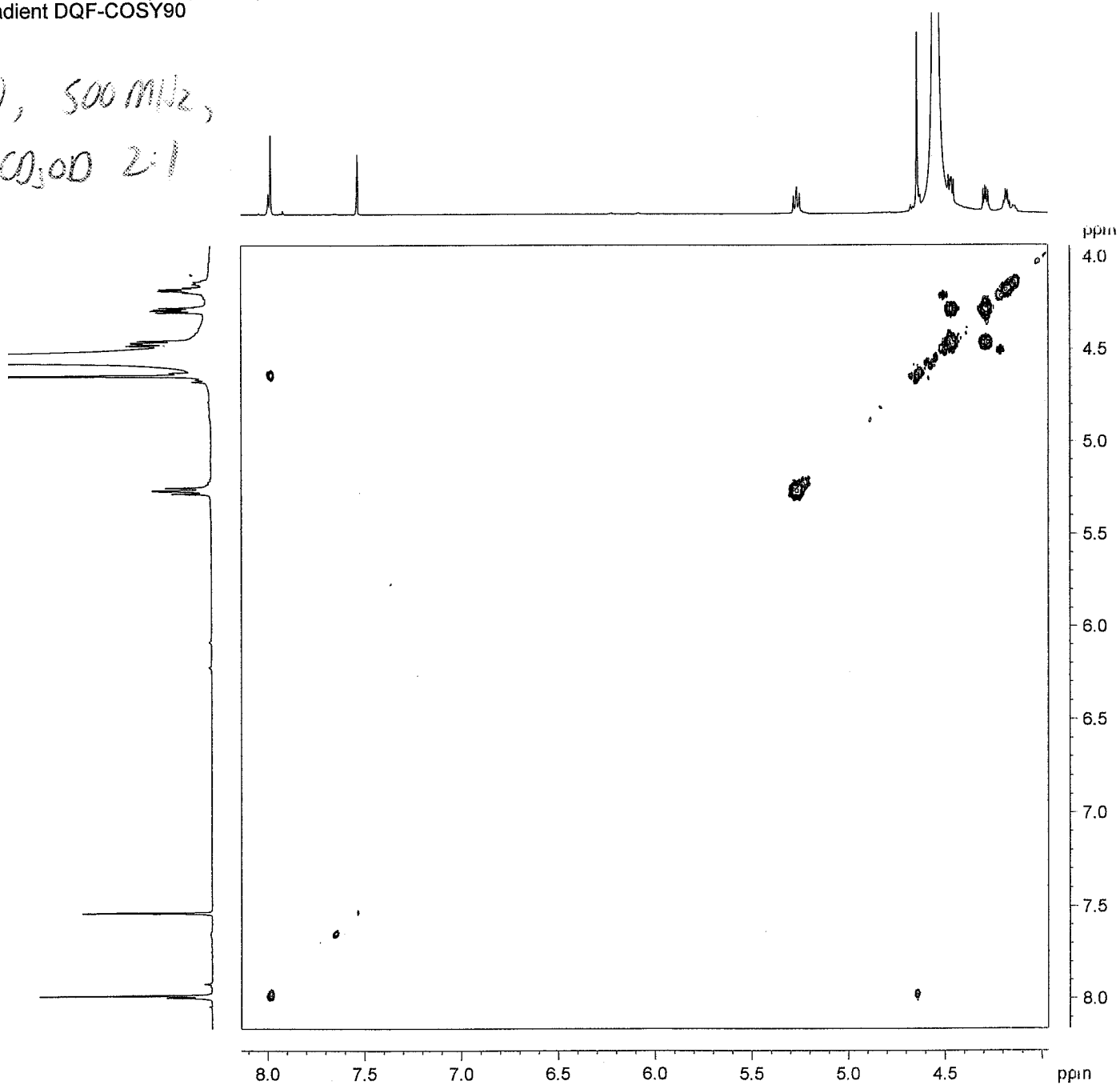
Supporting Information IV


(S)-10, 500 MHz, CDCl3/CD3OD 2:1
S configuration at C2''
(Epimer B)
7.993 5.279 5.276 5.262 4.652 4.490 4.481 4.475 4.465 4.311 4.302 4.295 4.287 4.194 4.185 3.769 3.727 3.717 3.690 3.680 3.673 3.663 3.643 3.636 3.634 3.629 3.626 3.598 3.594 3.585 3.542 3.537 3.516 3.509 3.497 3.389 3.366 3.348 3.346 2.914 2.904 2.888 2.878 2.719 2.693 2.199 2.168 1.639 1.627 1.624 1.611 1.597 1.585 1.569 1.450 1.434 1.419 1.405 0.866 0.853 0.839
ppm
0.684 0.404 0.632 1.192 0.383 0.425 13.257 1.781 0.467 0.552 0.530 2.895 3.843 50.562 4.365
0.632
0.383
0.552
0.530


175.210
169.070
165.124
145.284
123.301
75.014
73.413
72.754
72.527
71.028
70.989
70.831
70.622
70.458
70.240
70.168
64.786
64.691
63.903
62.626
60.874
56.279
51.261
40.756
39.805
36.237
33.525
33.108
32.450
30.215
30.185
30.076
29.895
29.868
29.523
29.058
28.769
26.357
26.229
26.134
23.165
14.302
(S)-10, 125 mM2,
CDCl3/CD3OD 2:1
(S)-configuration
@ C2"
(Epimer B)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

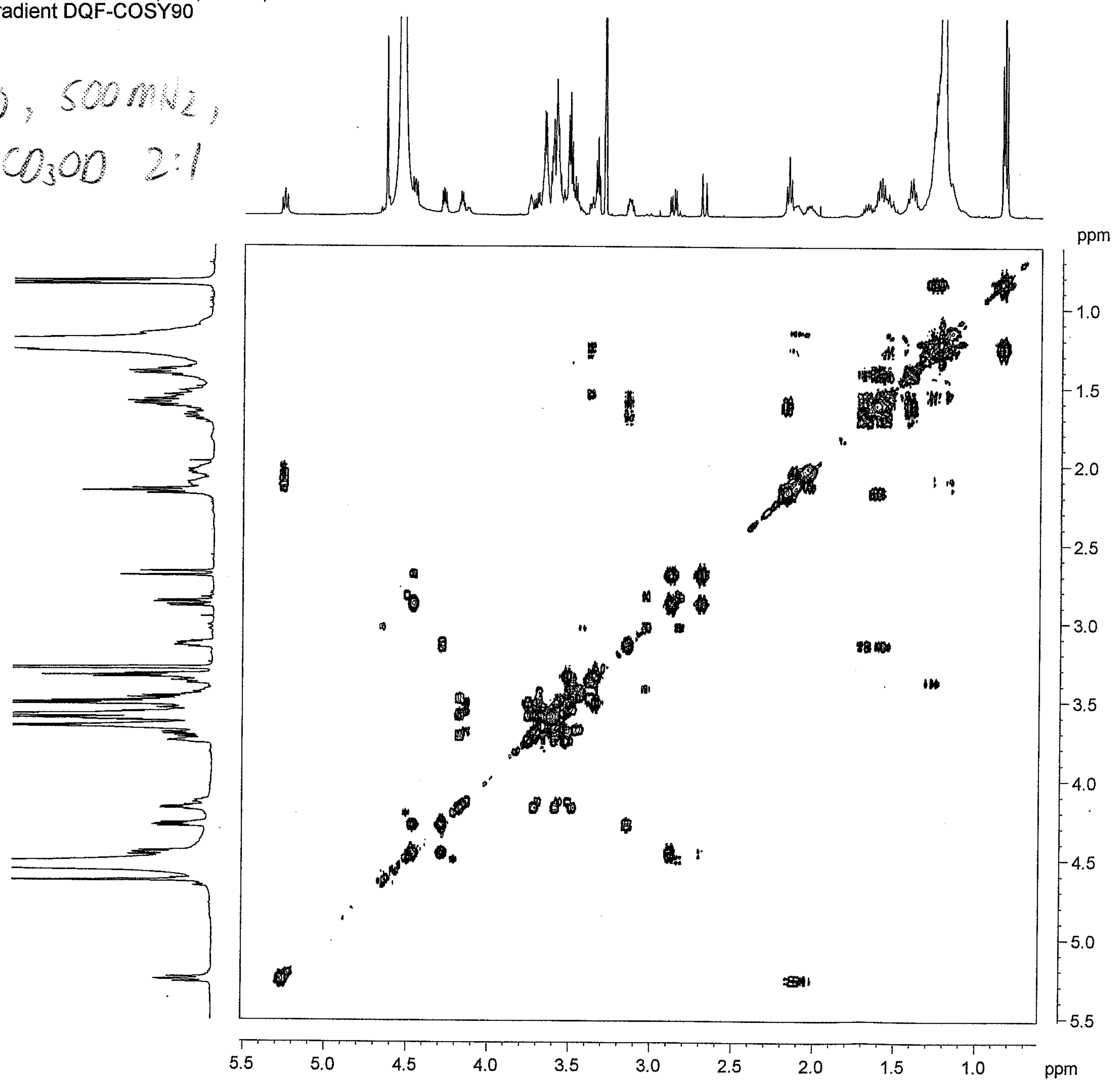
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient DQF-COSY90

(S)-10, 500 MHz,
$CDCl_3/CD_3OD$ 2:1



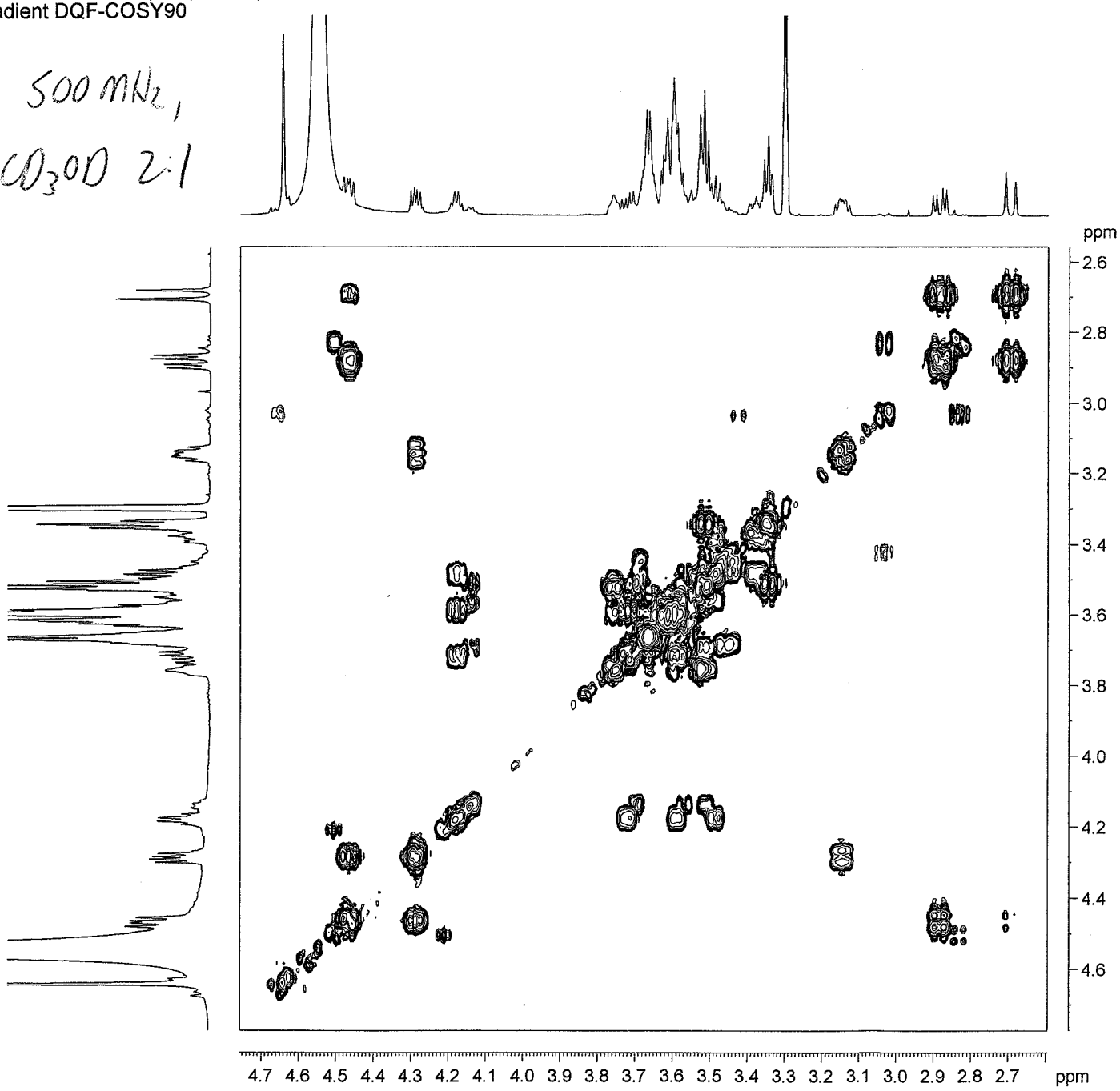
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient DQF-COSY90

(S)-10, 500 MHz,
CDCl3/CD3OD 2:1



Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient DQF-COSY90

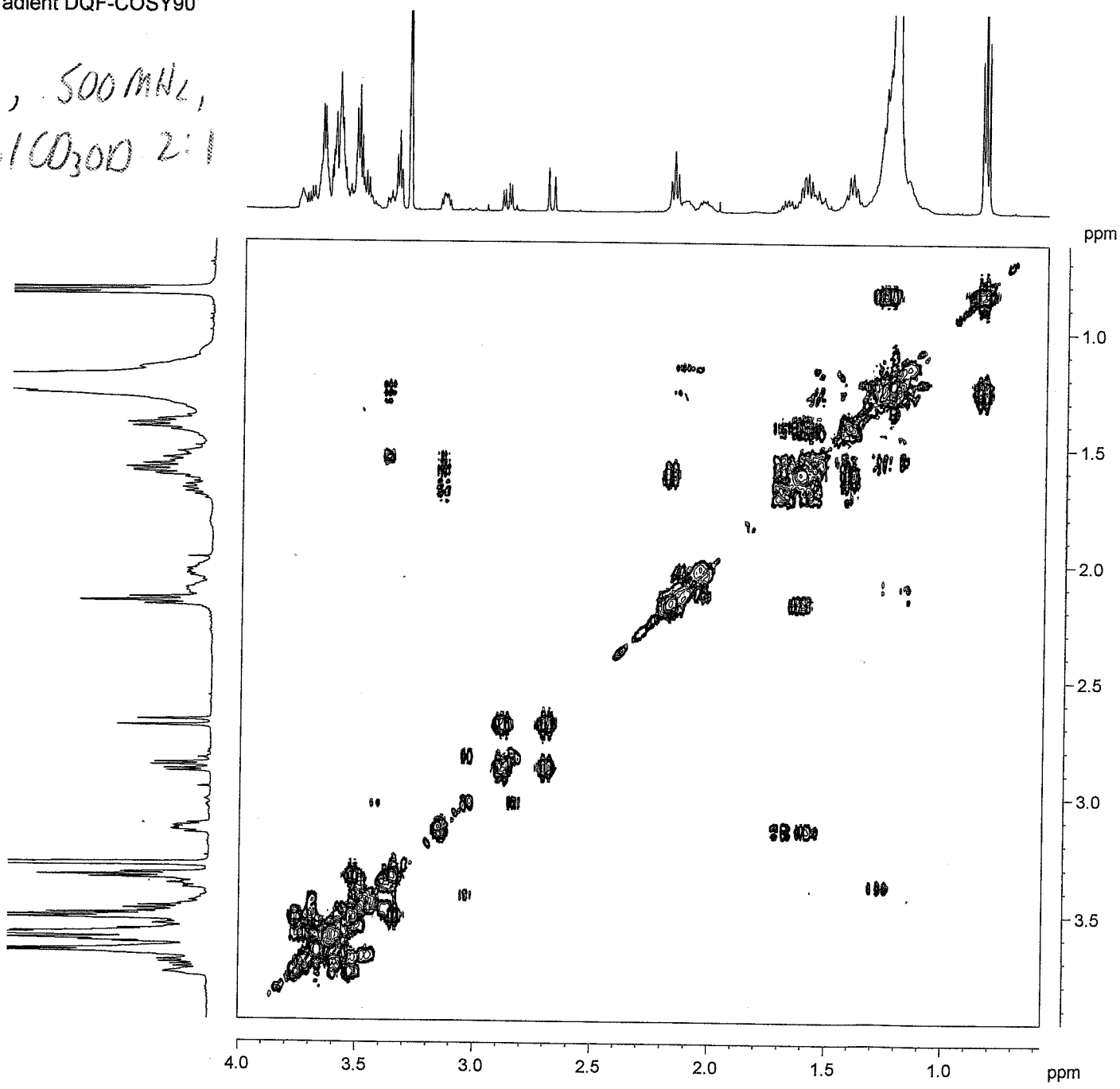
(S)-10, 500 MHz,
$CDCl_3/CD_3OD$ 2:1



S76

Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient DQF-COSY90

(S)-10, 500 MHz,
$CDCl_3$/CD_3OD 2:1

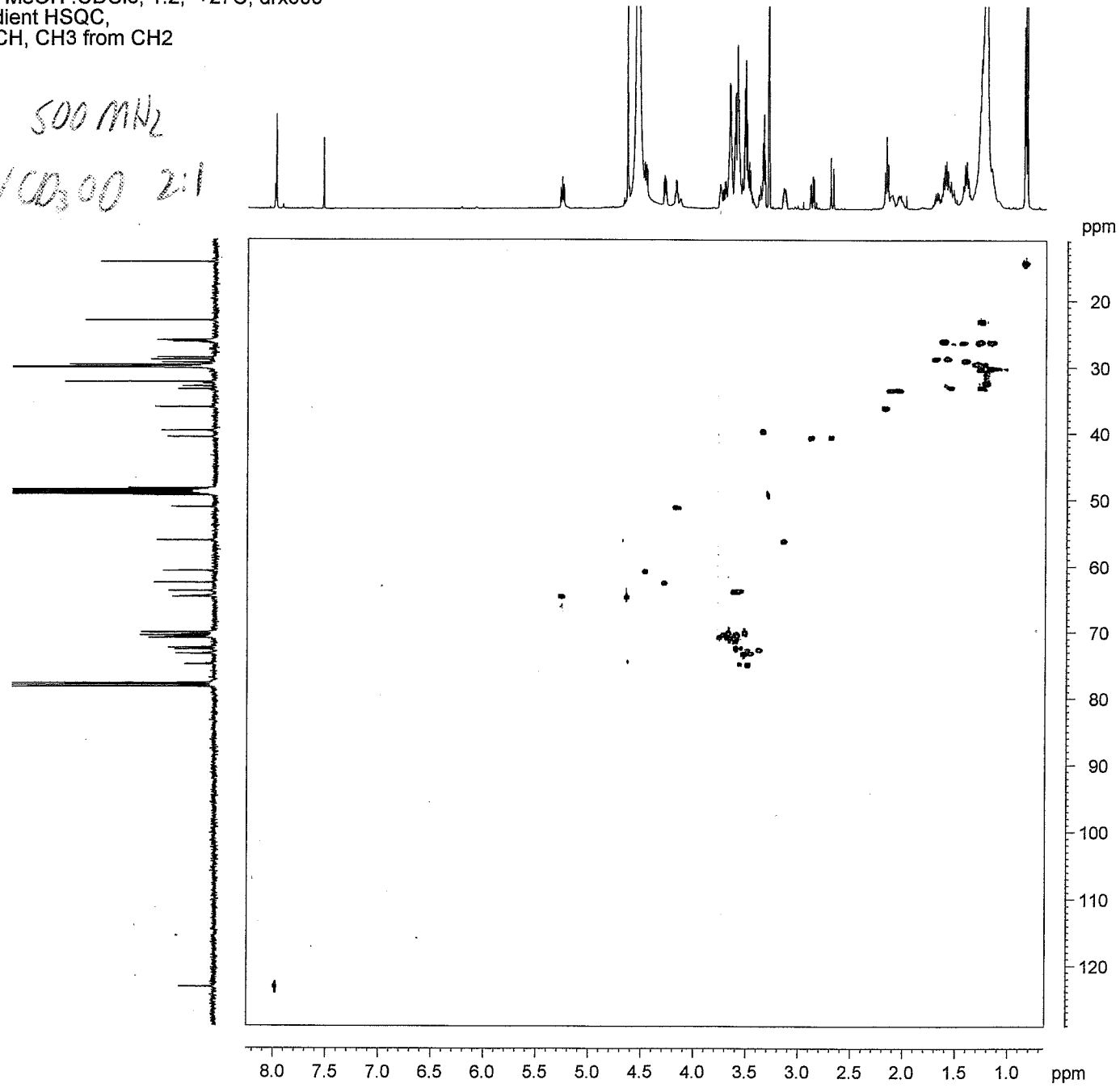


S65

red : CH
black : CH_2

Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

(S)-10, 500 MHz
$CDCl_3$ / CD_3OD 2:1



Current Data Parameters
NAME ap04pj1d
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110404
Time 12.17
INSTRUM drx500
PROBHD 5 mm TBI H/C-B
PULPROG hsqcedetgpsp.3
TD 2048
SOLVENT MeOD
NS 8
DS 16
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2294260 sec
RG 32768
DW 112.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 2.00000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
D16 0.00010000 sec
d21 0.00344828 sec
DELTA 0.00231087 sec
DELTA1 0.00036614 sec
DELTA2 0.00147414 sec
DELTA3 0.00343088 sec
IN0 0.00002260 sec
ST1CNT 256

======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
p2 24.40 usec
P28 0.10 usec
PL1 1.00 dB
SFO1 500.1320450 MHz

======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 11.00 usec
P14 500.00 usec
P31 1900.00 usec
PCPD2 76.00 usec
PL0 120.00 dB
PL2 -2.00 dB
PL12 14.60 dB
SFO2 125.7678780 MHz
SP3 5.33 dB
SP18 11.54 dB
SPNAM3 Crp60,0.5,20.1
SPNAM18 Crp60_xfilt.2
SPOAL3 0.500
SPOAL18 0.500
SPOFFS3 0.00 Hz
SPOFFS18 0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

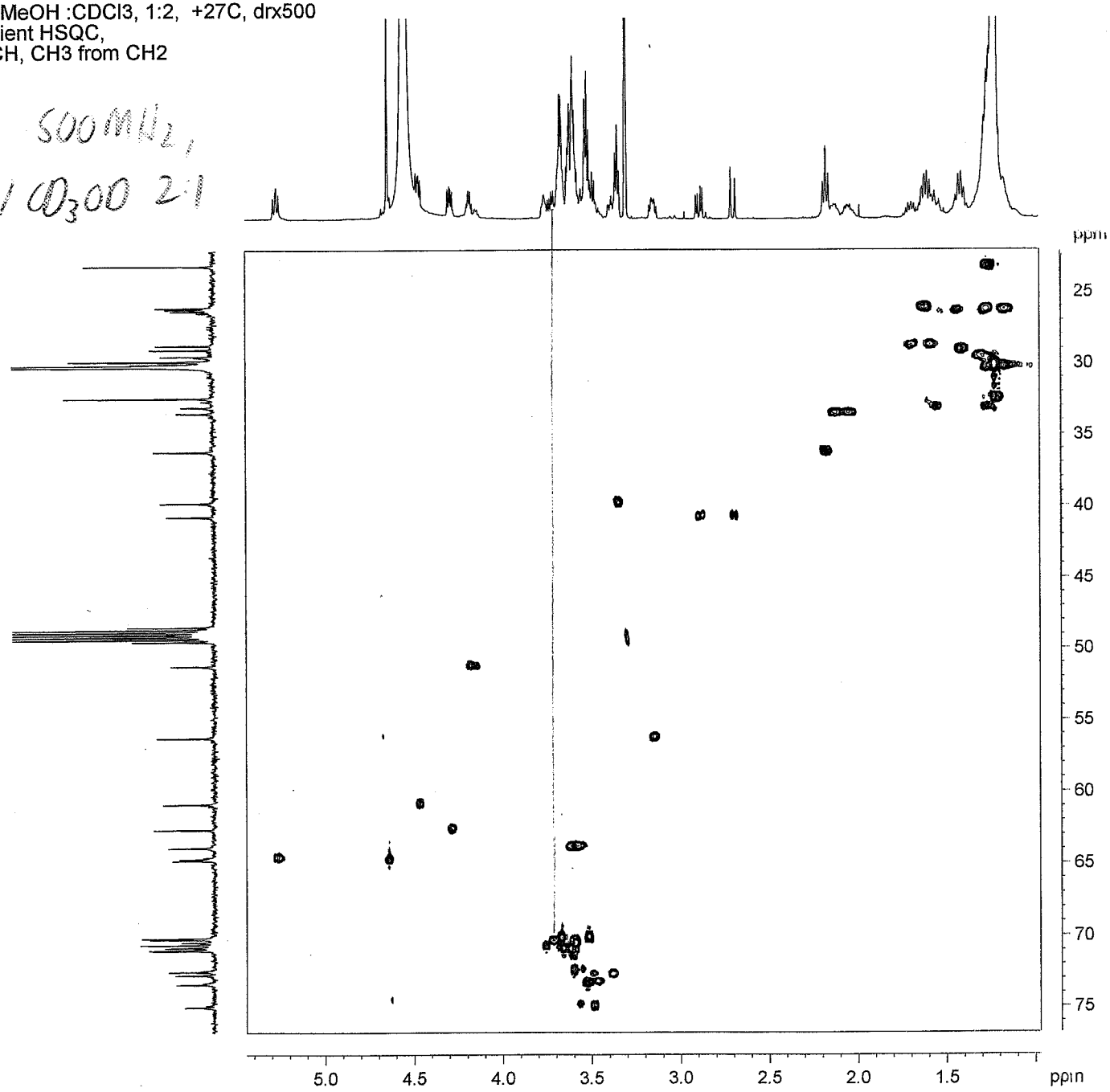
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7679 MHz
FIDRES 43.210732 Hz
SW 175.911 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 500.1300164 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 125.7577042 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

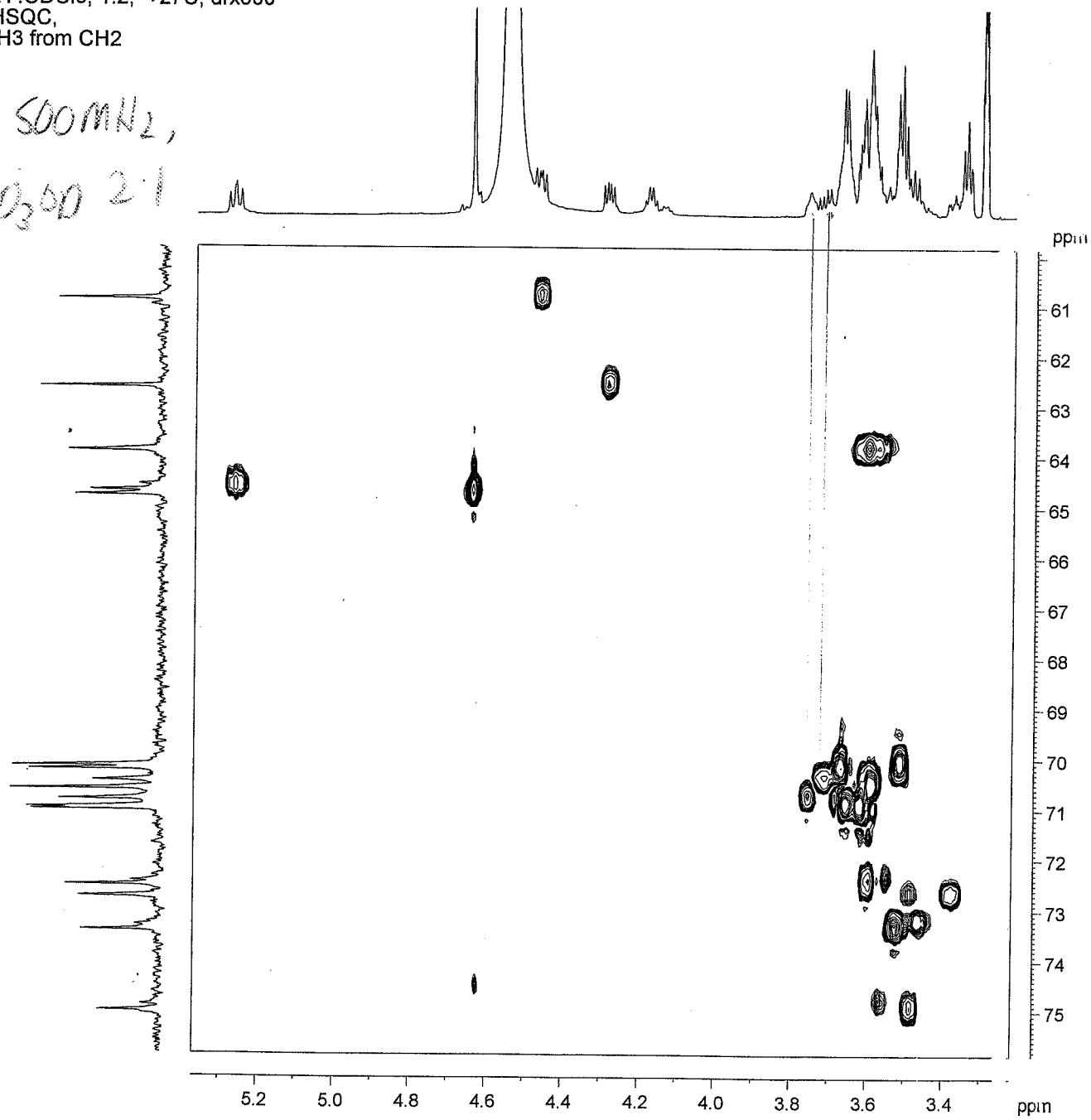
(S)-10, 500MHz,
$CDCl_3$ / CD_3OD 2:1



S81

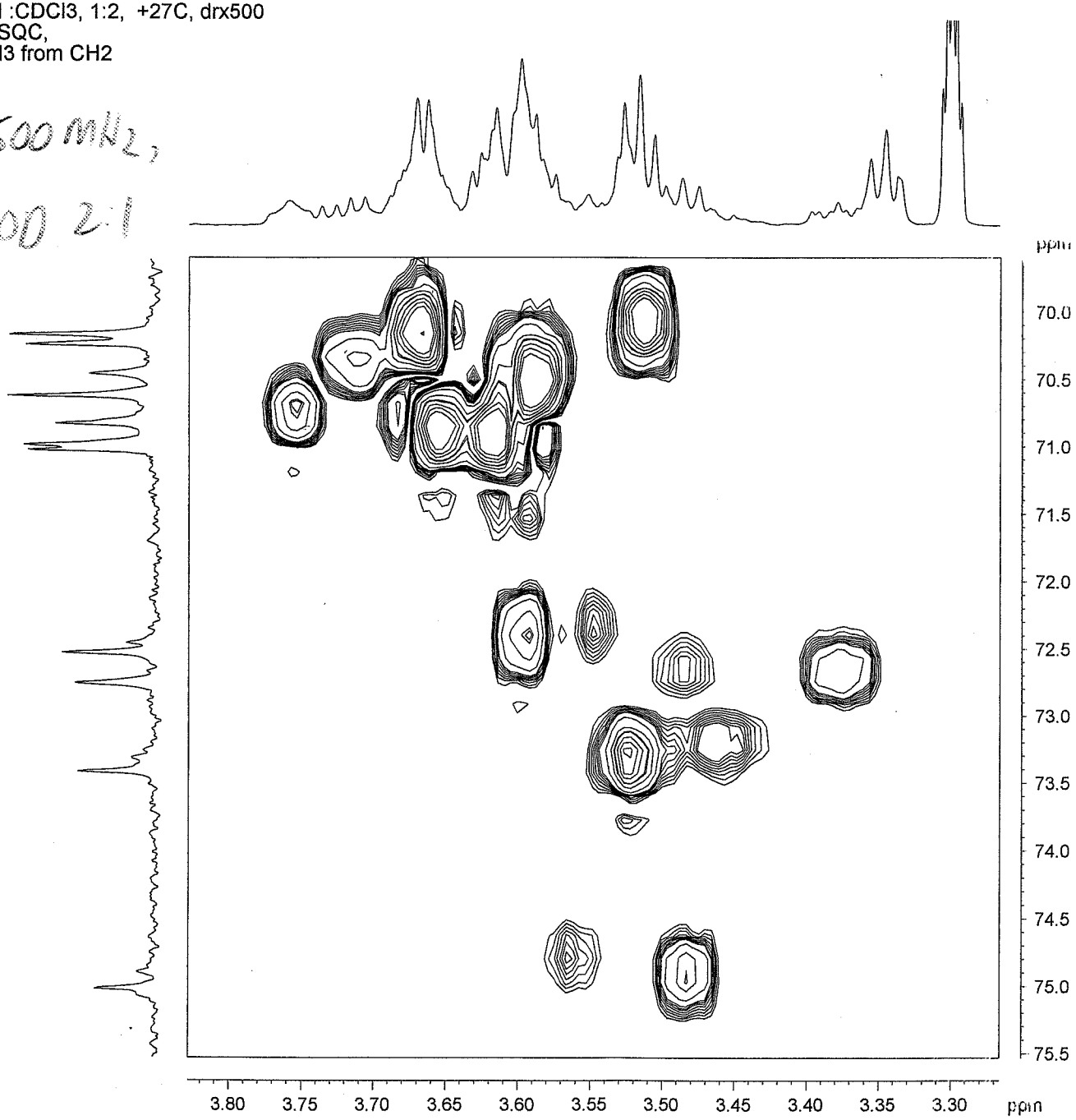
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

(S)-10, 500MHz,
$CDCl_3$ / CD_3OD 2:1



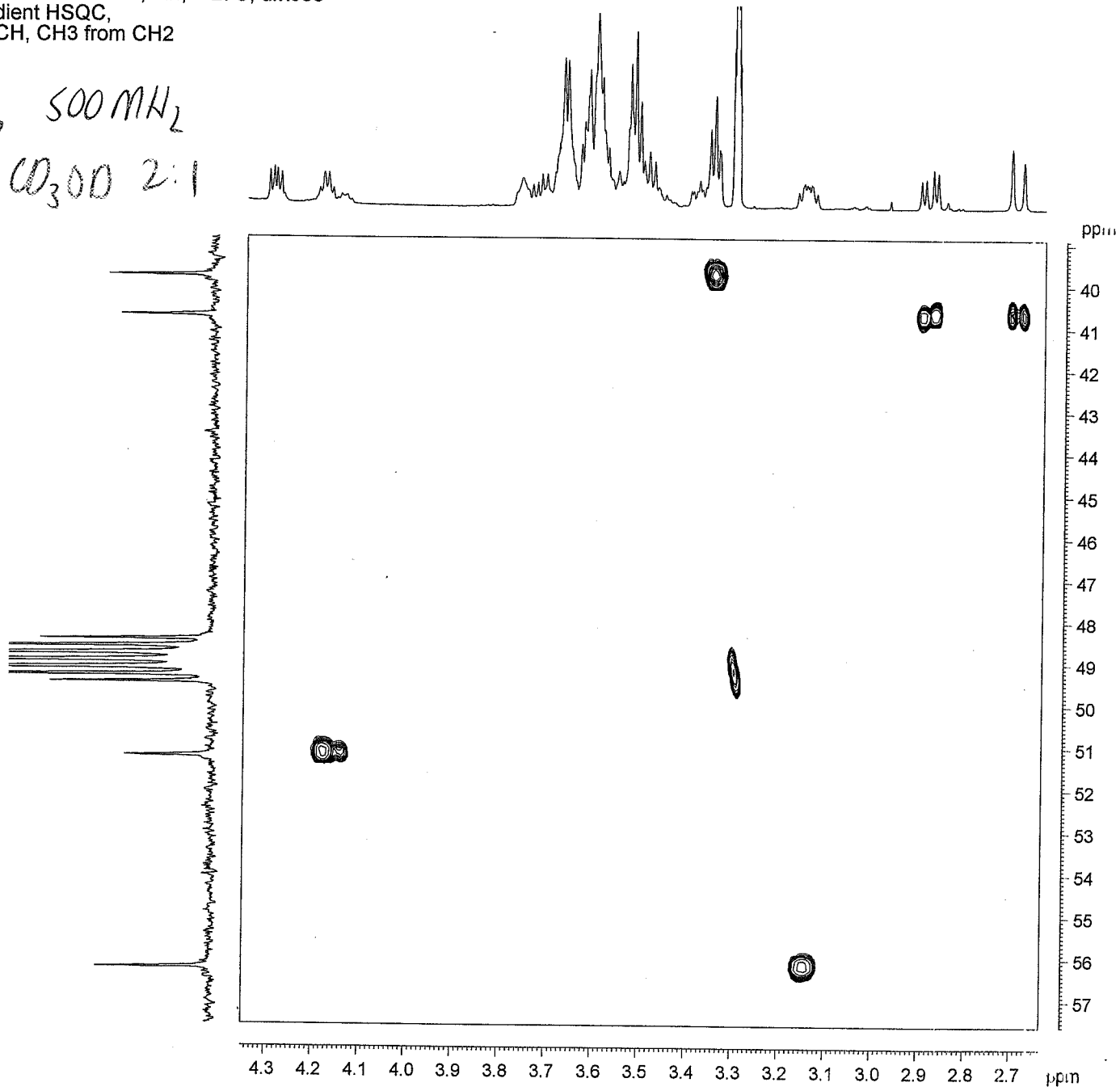
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

(S)-10, 500 mM, $CDCl_3/CD_3OD$ 2:1



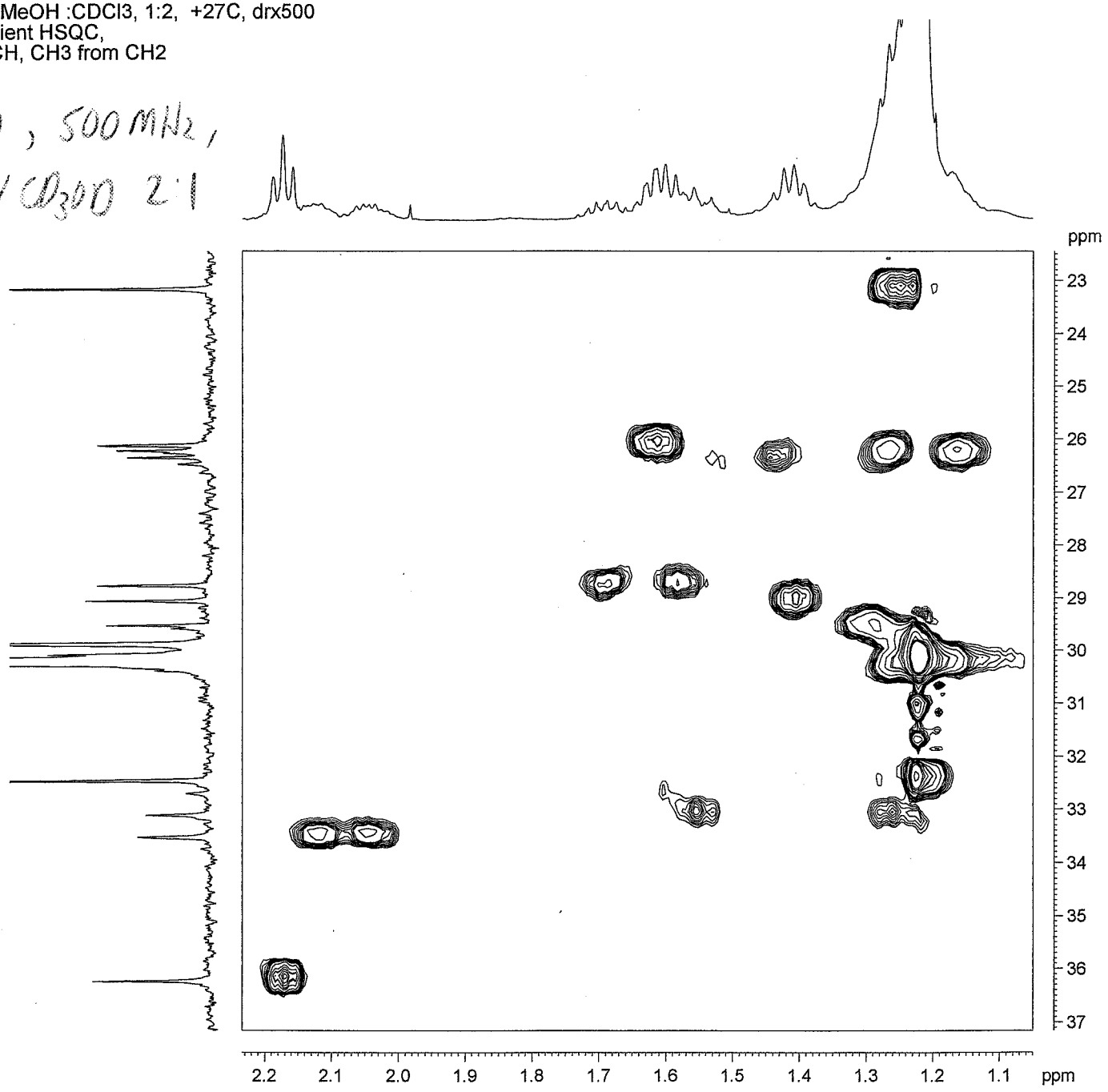
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

(S)-10, 500 MHz
$CDCl_3$/CD_3OD 2:1



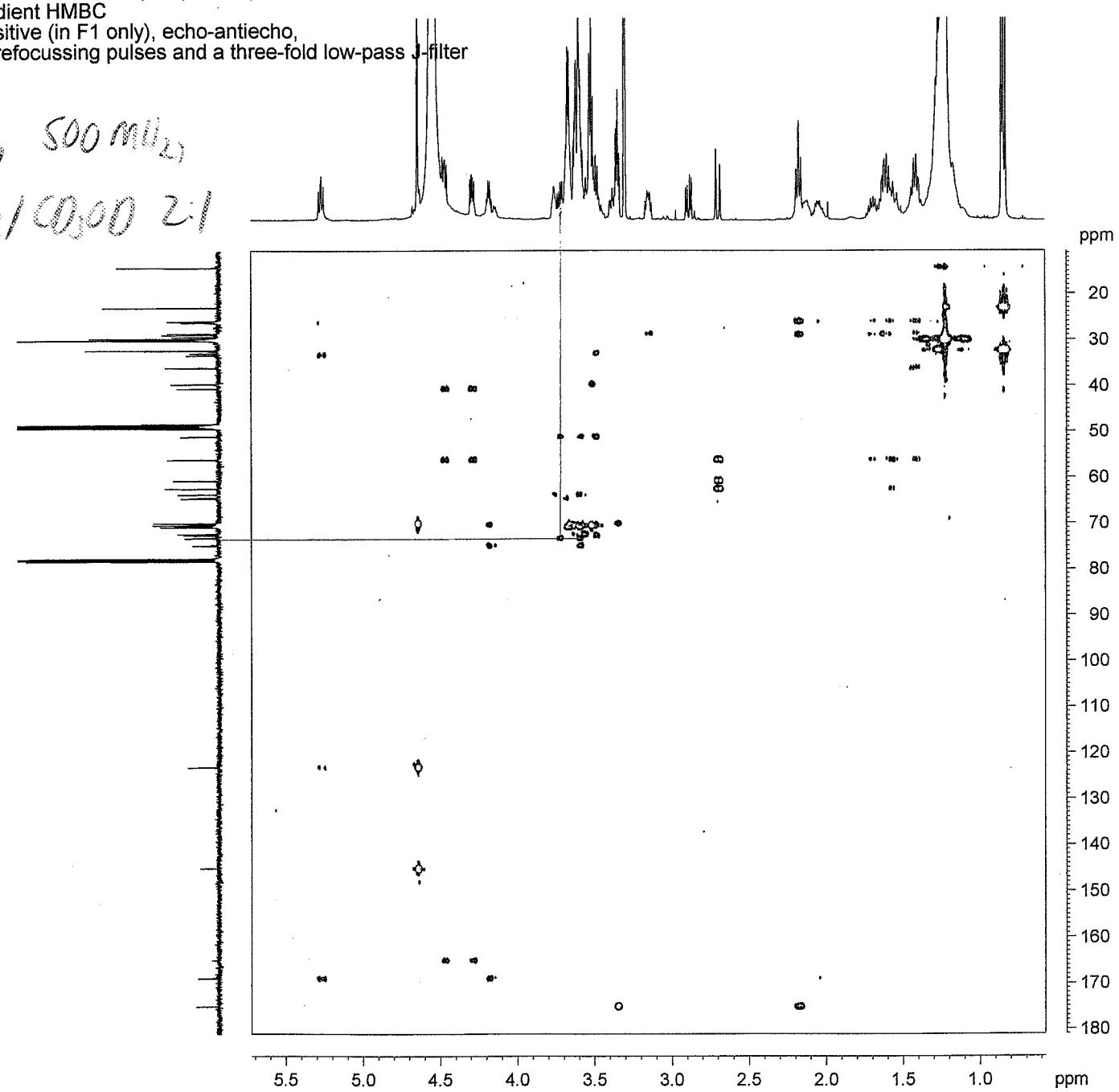
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HSQC,
with editing of CH, CH3 from CH2

(S)-10 , 500 MHz,
CDCl3/CD3OD 2:1



Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HMBC
run phase sensitive (in F1 only), echo-antiecho,
with adiabatic refocussing pulses and a three-fold low-pass J-filter

(S)-10, 500 mM, $CD_3OD/CDCl_3$ 2:1



Current Data Parameters
NAME ap04pj1d
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110404
Time 14.54
INSTRUM drx500
PROBHD 5 mm TBI H/C-B
PULPROG hmbcetgpl3nd
TD 2048
SOLVENT MeOD
NS 8
DS 16
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2294260 sec
RG 32768
DW 112.000 usec
DE 6.00 usec
TE 300.0 K
CNST6 120.0000000
CNST7 160.0000000
CNST13 5.0000000
CNST30 0.5981140
d0 0.00000300 sec
D1 2.00000000 sec
d6 0.10000000 sec
D16 0.00010000 sec
DELTA1 0.00297166 sec
DELTA2 0.00247143 sec
DELTA3 0.00208066 sec
DELTA4 0.09889600 sec
DELTA5 0.00003040 sec
IN0 0.00001785 sec
ST1CNT 256

======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
p2 24.40 usec
PL1 1.00 dB
SFO1 500.1320450 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 11.00 usec
P24 2000.00 usec
PL2 -2.00 dB
SFO2 125.7707920 MHz
SP7 5.33 dB
SPNAM7 Crp60comp.4
SPOAL7 0.500
SPOFFS7 0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPNAM5 SINE.100
GPNAM6 SINE.100
GPZ1 80.00 %
GPZ3 14.00 %
GPZ4 -8.00 %
GPZ5 -4.00 %
GPZ6 -2.00 %
P16 1000.00 usec

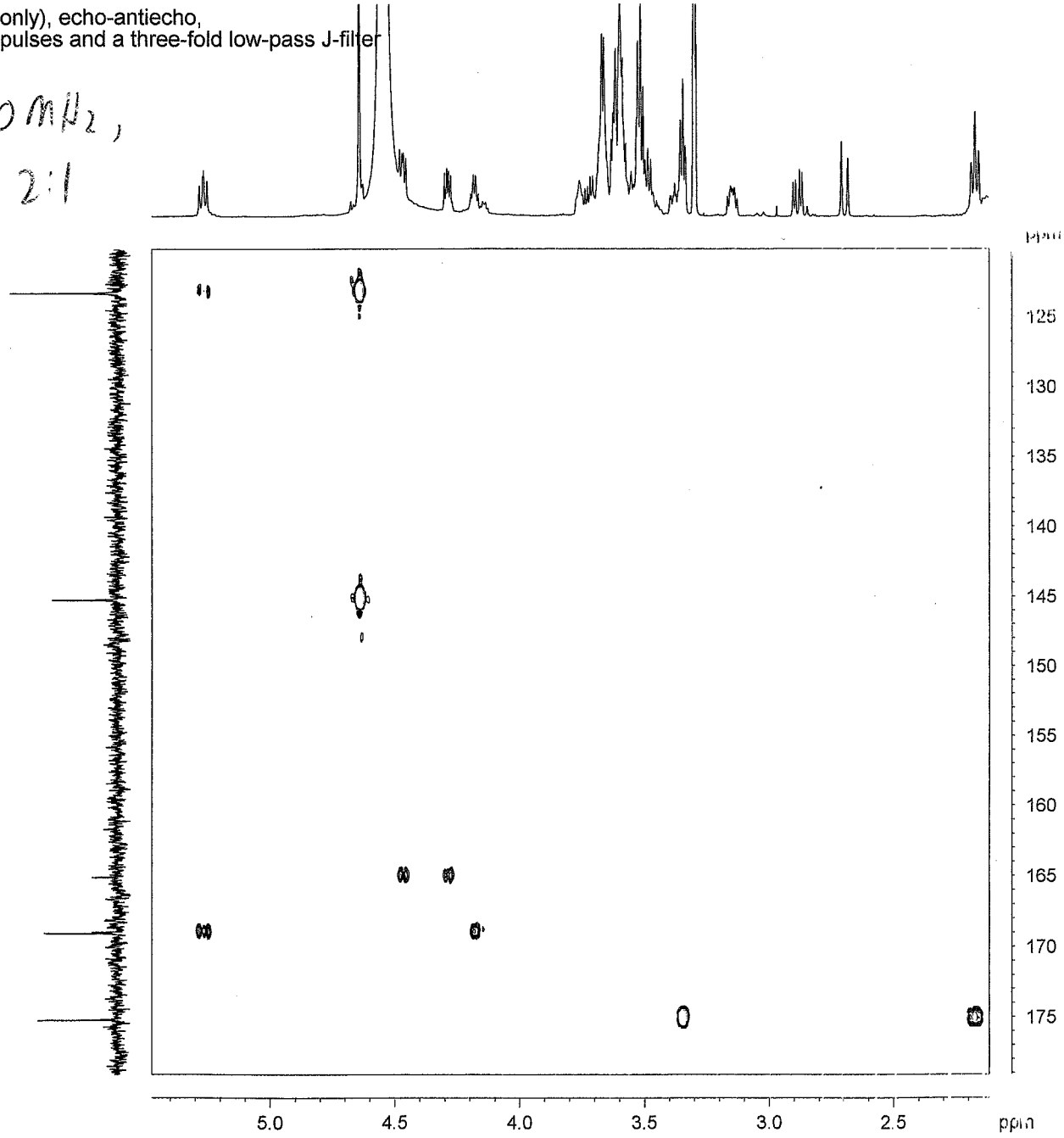
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 125.7708 MHz
FIDRES 54.709385 Hz
SW 222.716 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 500.1300164 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 125.7577042 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

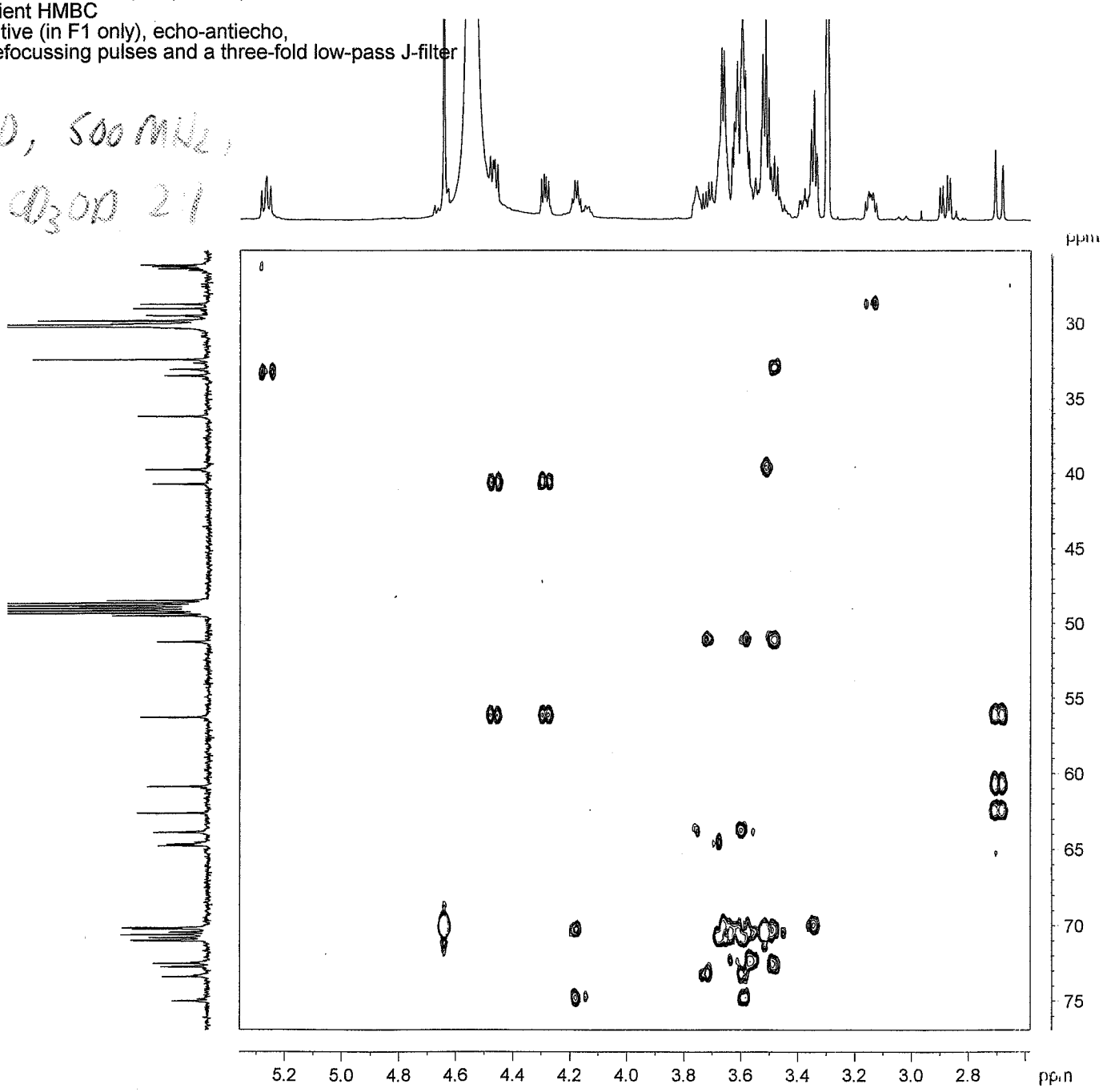
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HMBC
run phase sensitive (in F1 only), echo-antiecho,
with adiabatic refocussing pulses and a three-fold low-pass J-filter

(S)-10, 500 mM,
$CDCl_3$/CD_3OD 2:1



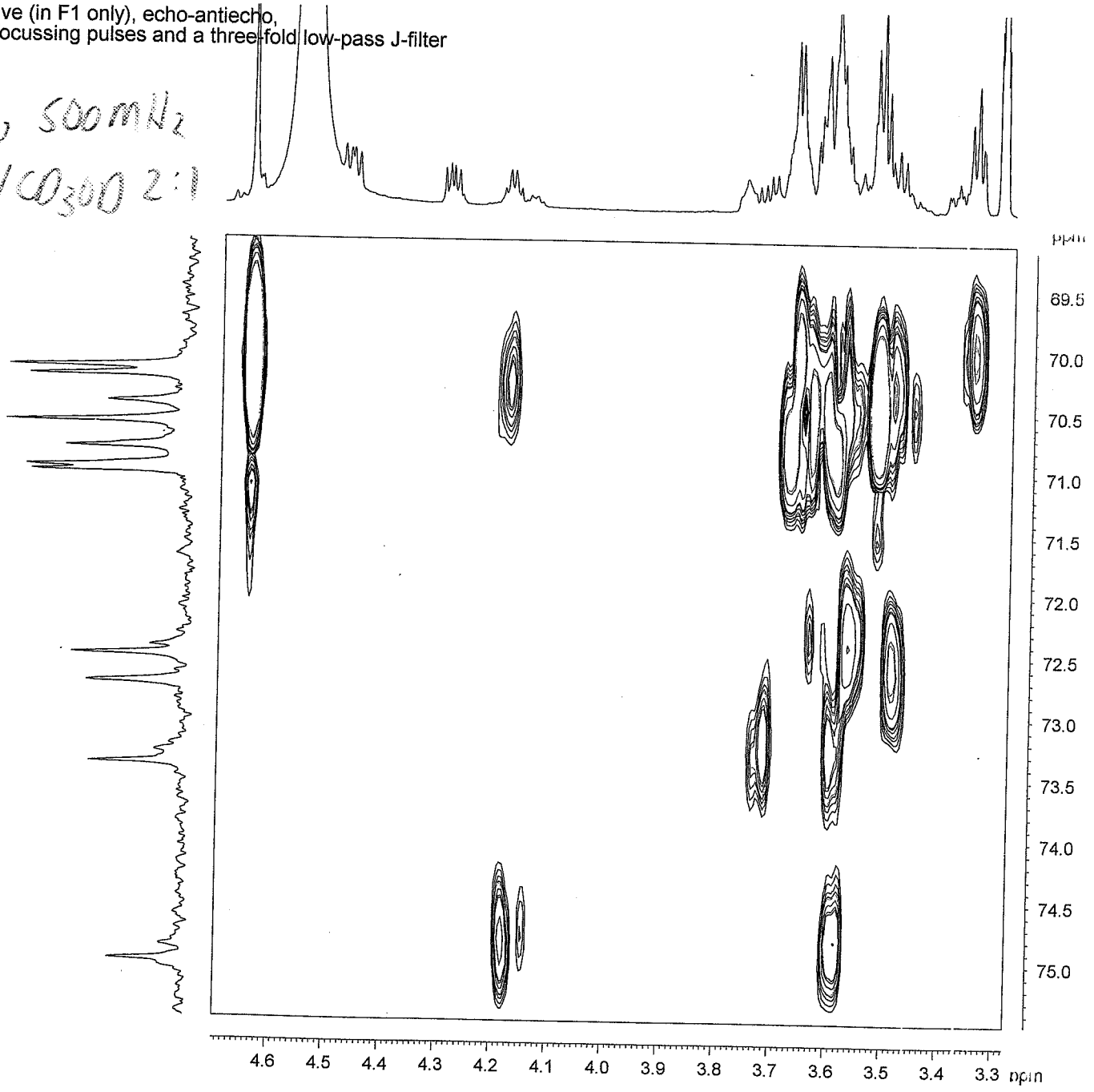
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HMBC
run phase sensitive (in F1 only), echo-antiecho,
with adiabatic refocussing pulses and a three-fold low-pass J-filter

(6)-10, 500 MHz,
$CDCl_3/CD_3OD$ 2:1



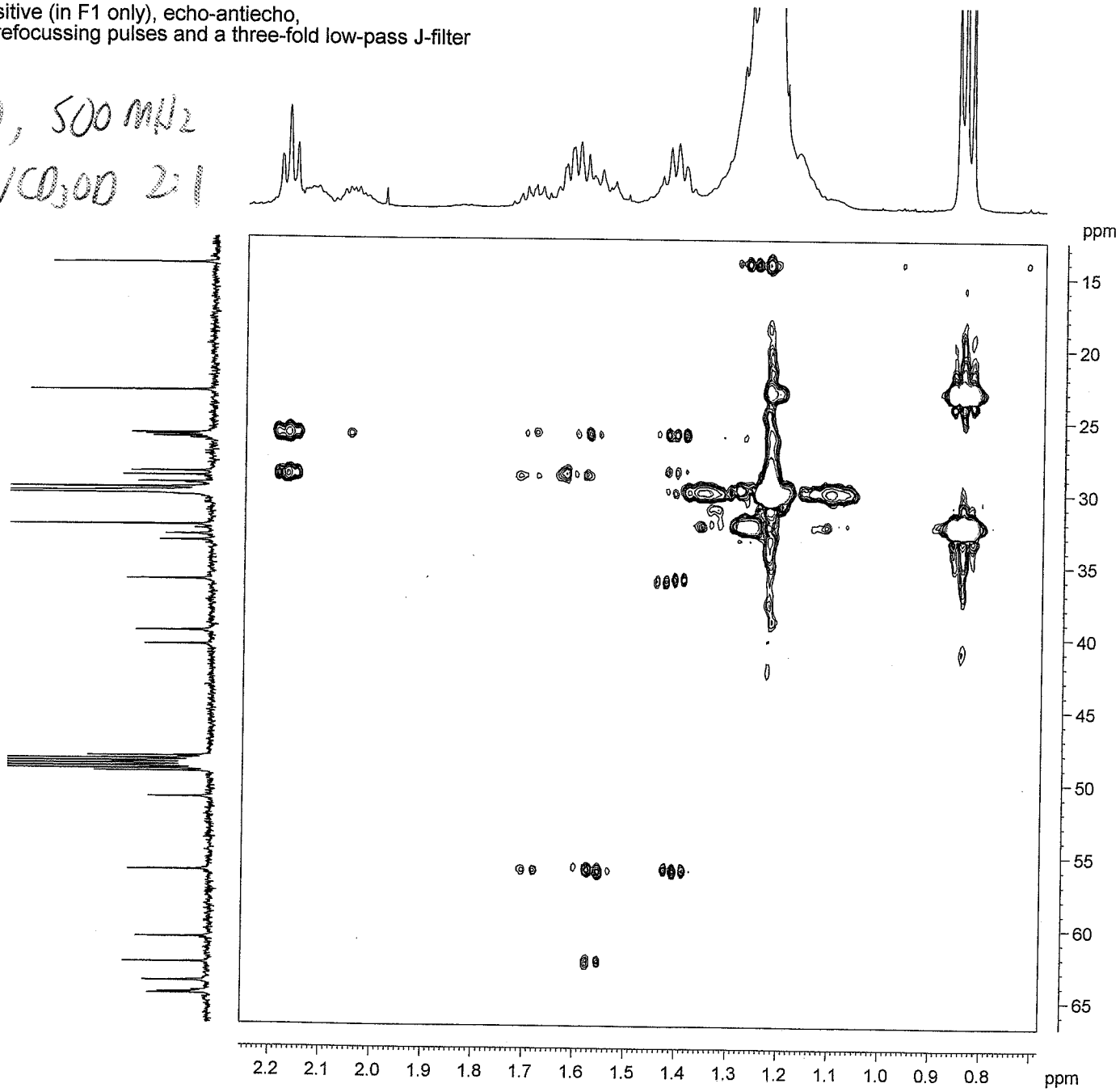
Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HMBC
run phase sensitive (in F1 only), echo-antiecho,
with adiabatic refocussing pulses and a three-fold low-pass J-filter

(S)-10, 500 MHz
$CDCl_3/CD_3OD$ 2:1



Pete Jervis, d4-MeOH :CDCl3, 1:2, +27C, drx500
Epimer B, Gradient HMBC
run phase sensitive (in F1 only), echo-antiecho,
with adiabatic refocussing pulses and a three-fold low-pass J-filter

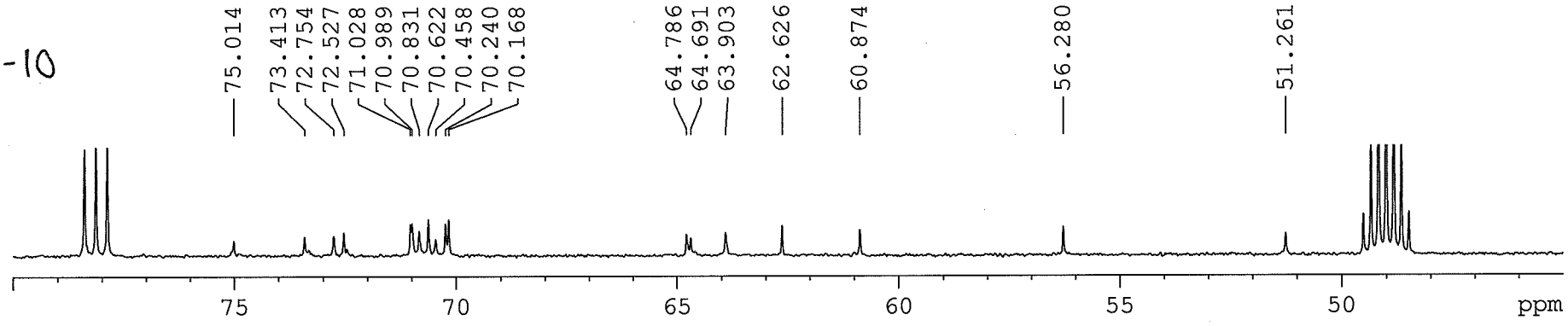
(S)-10, 500 MHz
$CDCl_3$ / CD_3OD 2:1



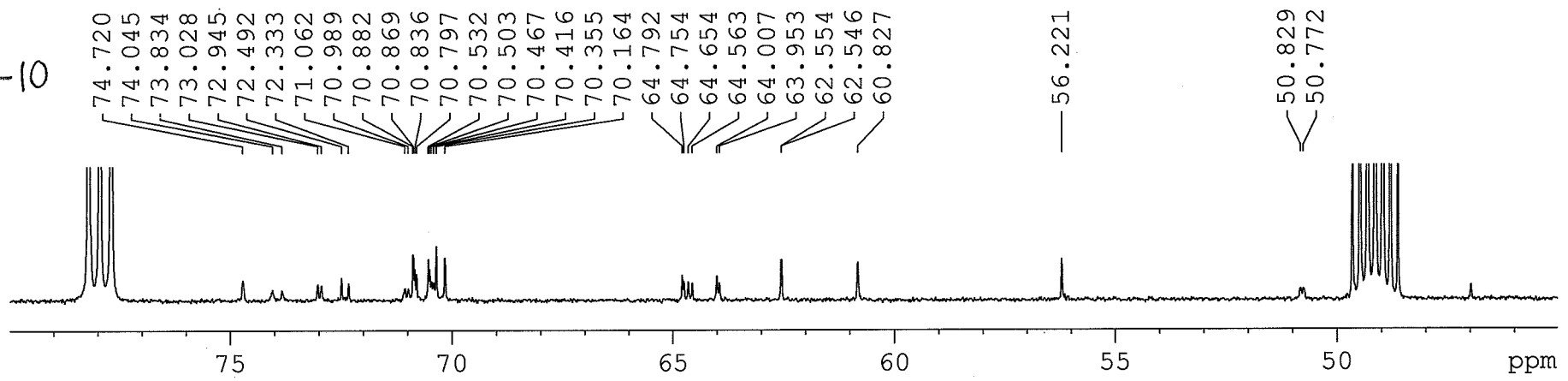
S90

^{13}C NMR COMPARISON OF INDIVIDUAL EPIMERS OF 10 WITH EPIMERIC MIXTURE

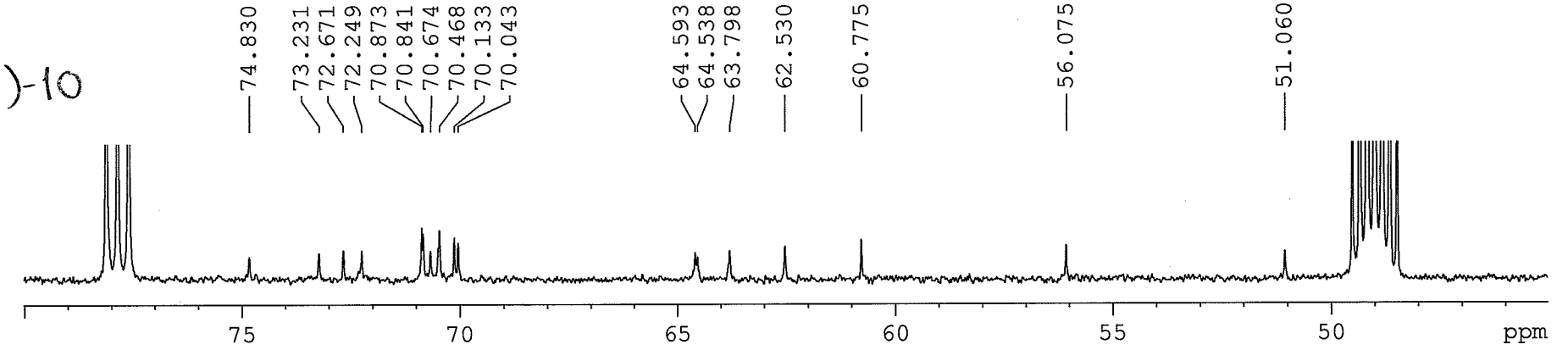
(S)-10

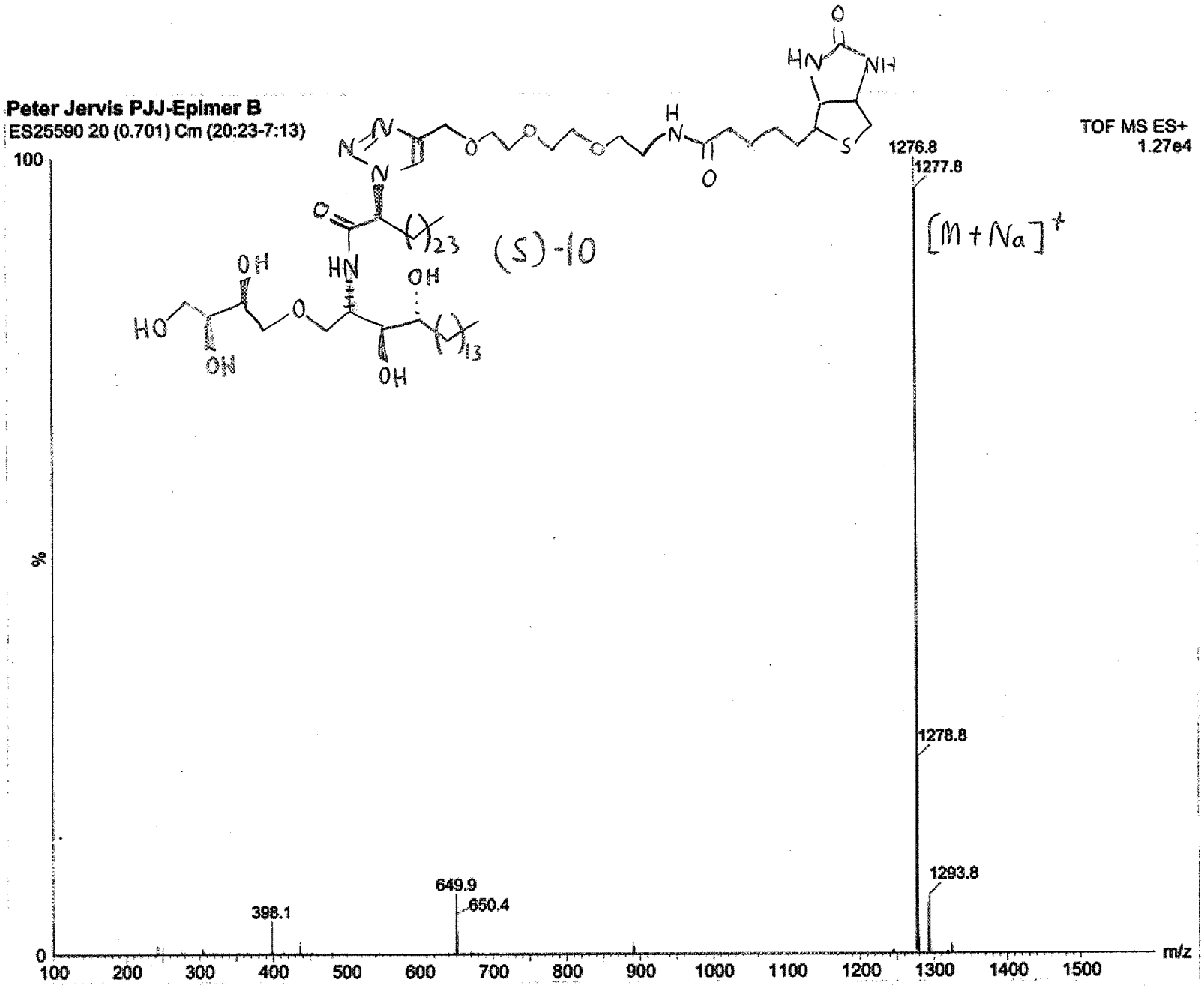


(R/S)-10



(R)-10




Peter Jervis PJJ-Epimer B
ES25590 20 (0.701) Cm (20:23-7:13)
TOF MS ES+
1.27e4
(S)-10
[M + Na]+
1276.8
1277.8
1278.8
1293.8
649.9
650.4
398.1
100
%
0
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500

(S)-10

Elemental Composition Report **Peter Jervis PJJ-Epimer B**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 99.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
94 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Minimum:				-1.5	
Maximum:		5.0	10.0	99.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
1276.9166	1276.9161	0.5	0.4	7.5	C67 H127 N7 O12 Na S