

Electronic Supplementary Information

Total Synthesis and Reassignment of the Structures of the Antimicrobial Lipodepsipeptides: Circulocins γ and δ

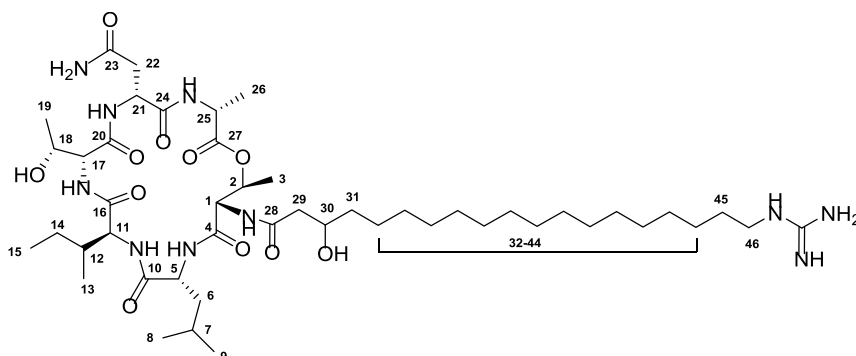
James R. Cochrane, Claudia J. Exner and Katrina A. Jolliffe*

School of Chemistry, The University of Sydney, 2006, NSW, Australia

kate.jolliffe@sydney.edu.au

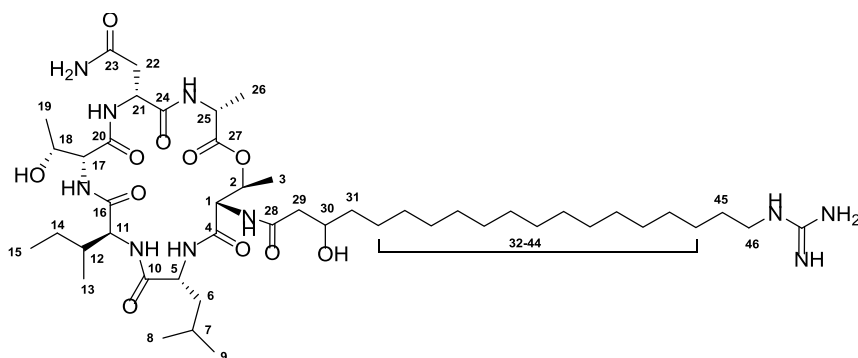
	Page
Comparison of Spectroscopic Data for Synthetic and Natural Products	S2
Reaction Schemes S1-S4	S6
^1H and ^{13}C NMR Spectra	S10
HPLC traces of compounds (R)-4, (S)-4 and (R)-6	S66
References	S68

Table S1: ^1H NMR comparison of (*R*)-4 and (*S*)-4 with reported data¹



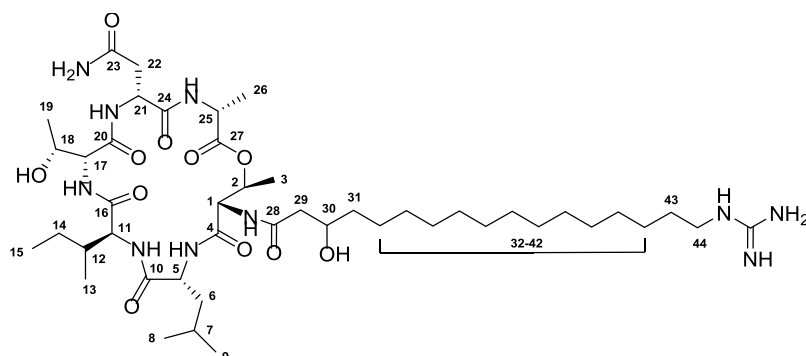
Reported Data (D) ¹		(R)-4		(S)-4	
Assignment	δ_{H} (J in Hz)	δ_{H} (J in Hz)	$\Delta\delta_{\text{H}}$ (D-R)	δ_{H} (J in Hz)	$\Delta\delta_{\text{H}}$ (D-S)
L-Threonine (1)					
1	4.39 (dd, 8.5, 2.5)	4.39 (dd, 8.5, 2.2)	0	4.36 (dd, 8.5, 2.2)	0.03
2	5.30 (dq, 6.5, 2.5)	5.30 (dq, 6.5, 2.5)	0	5.29 (dq, 6.5, 2.5)	0.01
3	1.12 (d, 6.5)	1.12 (d, 6.6)	0	1.17 (d, 6.6)	-0.05
1-NH	8.05 (d, 8.5)	8.06 (d, 8.5)	-0.01	8.02 (d, 7.0)	0.03
D-Leucine (2)					
5	4.65 (dd, 7.8, 7.8)	4.66 (dd, 7.7, 7.8)	-0.01	4.70 (dd, 7.7, 7.8)	-0.05
6	1.39 (m)	1.39 (m)	0	1.39 (m)	0
7	1.43 (m)	1.43 (m)	0	1.44 (m)	-0.01
8	0.83 (d, 6.5)	0.84 (d, 6.5)	-0.01	0.84 (d, 6.5)	-0.01
9	0.83 (d, 6.5)	0.84 (d, 6.5)	-0.01	0.84 (d, 6.5)	-0.01
5-NH	7.48 (d, 7.8)	7.45 (d, 8.0)	0.03	7.47 (br m)	0.01
L-Isoleucine (3)					
11	4.20 (dd, 7.7, 7.7)	4.20 (m)	0	4.13 (m)	0.07
12	1.76 (m)	1.77 (m)	-0.01	1.75 (m)	0.01
13	0.80 (d, 7.0)	0.81 (d, 7.0)	-0.01	0.81 (d, 7.0)	-0.01
14a	1.40 (m)	1.40 (m)	0	1.44 (m)	-0.04
14b	1.17 (m)	1.18 (m)	-0.01	1.19 (m)	-0.02
15	0.79 (t, 7)	0.79 (t, 7.3)	0	0.79 (t, 7.3)	0
11-NH	8.36 (d, 7.7)	8.37 (br m)	-0.01	8.43 (d, 7.4)	-0.07
D-allo-Threonine (4)					
17	3.92 (m)	3.92 (m)	0	3.95 (m)	-0.03
18	3.92 (m)	3.92 (m)	0	3.95 (m)	-0.03
19	1.09 (d, 7.0)	1.10 (d, 7.0)	-0.01	1.09 (d, 7.0)	0
17-NH	8.38 (d, 5.8)	8.38 (br m)	0	8.39 (d, 4.2)	-0.01
D-Asparagine (5)					
21	4.28 (dt, 7.0, 7.0)	4.28 (m)	0	4.30 (m)	-0.02
22a	2.59 (dd, 15, 7.2)	2.59 (15, 7.3)	0	2.57 (15.0, 7.2)	0.02
22b	2.77 (dd, 15, 6.2)	2.78 (15, 6.2)	-0.01	2.85 (15.0, 6.6)	-0.08
21-NH	8.09 (d, 7.0)	8.09 (d, 7.0)	0	8.07 (d, 8.3)	0.02
23-NHa	7.41 (br, s)	7.42 (br s)	-0.01	7.45 (br, s)	-0.04
23-NHb	7.00 (br s)	7.00 (br s)	0	7.00 (br s)	0
D-Alanine (6)					
25	3.95 (dq, 7.0, 7.0)	3.97 (m)	-0.02	3.91 (m)	0.04
26	1.17 (d, 7.0)	1.18 (d, 7.3)	-0.01	1.19 (d, 7.3)	-0.02
25-NH	7.32 (d, 7.0)	7.35 (d, 7.3)	-0.03	7.28 (d, 8.2)	0.04
Guanidino-3-hydroxy-nonadecanoic acid (GHND)					
29a	2.32 (dd, 13.2, 6.2)	2.32 (dd, 13.2, 6.4)	0	2.24 (dd, 13.1, 3.7)	0.08
29b	2.45 (dd, 13.2, 4.9)	2.45 (dd, 13.2, 5.4)	0	2.43 (dd, 13.1, 9.0)	0.02
30	3.80 (m)	3.81 (m)	-0.01	3.79 (m)	0.01
31	1.36 (m)	1.37 (m)	-0.01	1.37 (m)	-0.01
32	1.35 (m)	1.37 (m)	-0.02	1.37 (m)	-0.02
33-44	1.25 (m)	1.25 (m)	0	1.24 (m)	0.01
45	1.42 (m)	1.42 (m)	0	1.44 (m)	-0.02
46a	3.07 (7.0, 7.0)	3.07 (dd, 7.0, 7.0)	0	3.10 (dd, 7.0, 7.0)	-0.03
46b	3.05 (dd, 7.0, 7.0)	3.05 (dd, 7.0, 7.0)	0	3.05 (dd, 7.0, 7.0)	0
NHCNHNH ₂	7.52 (t, 7.0)	7.46 (br m)	0.06	7.47 (br m)	0.05

Table S2: ^{13}C NMR comparison of (*R*)-4 and (*S*)-4 with reported data¹



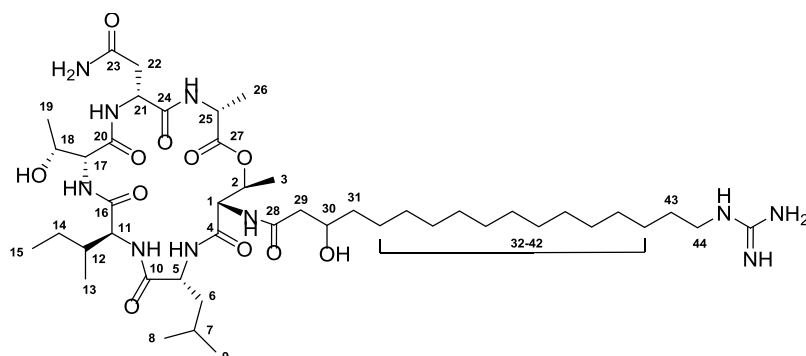
Reported Data (D) ¹		<i>(R)</i> -4		<i>(S)</i> -4	
Assignment	δ_{C}	δ_{C}	$\Delta\delta_{\text{C}} (\text{D-R})$	δ_{C}	$\Delta\delta_{\text{C}} (\text{D-S})$
L-Threonine (1)					
1	56.6	56.6	0.00	56.8	-0.20
2	70.3	70.3	0.00	70.1	0.20
3	16.0	16.0	0.00	15.8	0.20
4	168.0	167.9	0.10	168.1	-0.10
D-Leucine (2)					
5	50.1	49.9	0.20	49.7	0.40
6	42.3	42.3	0.00	42.2	0.10
7	24.0	23.9	0.10	23.6	0.40
8	22.6	22.6	0.00	22.6	0.00
9	22.2	22.2	0.00	21.9	0.30
10	171.6	171.4	0.20	171.1	0.50
L-Isoleucine (3)					
11	56.7	56.6	0.10	56.9	-0.20
12	35.8	35.8	0.00	35.4	0.40
13	15.3	15.3	0.00	15.0	0.30
14	24.2	24.0	0.20	23.7	0.50
15	10.6	10.6	0.00	10.3	0.30
16	173.0	172.7	0.30	173.0	0.00
D-allo-Threonine (4)					
17	60.1	60.1	0.00	59.7	0.40
18	65.6	65.4	0.20	65.4	0.20
19	19.3	19.3	0.00	18.9	0.40
20	170.2	170.2	0.00	170.4	-0.20
D-Asparagine (5)					
21	50.4	50.3	0.10	50.3	0.10
22	36.5	36.4	0.10	36.5	0.00
23	169.7	169.7	0.00	169.7	0.00
24	172.4	172.4	0.00	172.3	0.10
D-Alanine (6)					
25	47.8	47.6	0.20	47.7	0.10
26	16.9	17.0	-0.10	16.8	0.10
27	170.6	170.6	0.00	170.6	0.00
Guanidino-3-hydroxy-nonadecanoic acid (GHND)					
28	171.6	171.5	0.10	172.2	-0.60
29	42.8	42.7	0.10	43.0	-0.20
30	67.5	67.5	0.00	68.4	-0.90
31	36.4	36.4	0.00	36.4	0.00
32	25.3	25.4	-0.10	25.6	-0.30
33-43	28.5-29.0	28.5-29.0	0.00	28.5-29.0	0.00
44	25.9	25.9	0.00	25.8	0.10
45	28.3	28.3	0.00	28.2	0.10
46	40.6	40.5	0.10	40.5	0.10
NHCNHNH ₂	156.6	156.5	0.10	156.6	0.00

Table S3: ^1H NMR comparison of (*R*)-**6** with reported data¹



Assignment	Reported Data (D) ¹	(R)-6	
	δ_{H} (J in Hz)	δ_{H} (J in Hz)	$\Delta\delta_{\text{H}}$ (N-R)
L-Threonine (1)			
1	4.39 (dd, 8.5, 2.5)	4.41 (dm, 8.3)	-0.02
2	5.30 (dq, 2.5, 6.5)	5.30 (m)	0
3	1.12 (d, 6.5)	1.13 (d, 6.2)	-0.01
1-NH	8.05 (d, 8.5)	8.13 (d, 8.1)	-0.06
D-Leucine (2)			
5	4.65 (br dd, 7.8, 7.8)	4.64 (m)	0.01
6	1.39 (m)	1.38 (m)	0.01
7	1.43 (m)	1.45 (m)	-0.02
8	0.83 (d, 6.5)	0.83 (d, 5.8)	0
9	0.83 (d, 6.5)	0.83 (d, 5.8)	0
5-NH	7.48 (d, 7.8)	7.49 (d, 9.2)	-0.01
L-Isoleucine (3)			
11	4.20 (dd, 7.7, 7.7)	4.20 (m)	0
12	1.76 (m)	1.77 (m)	-0.01
13	0.80 (d, 7.0)	0.81 (d, 5.6)	-0.01
14a	1.40 (m)	1.40 (m)	0
14b	1.17 (m)	1.16 (m)	0.01
11-NH	8.36 (d, 7.7)	8.39 (d, 6.6)	-0.03
D-allo-Threonine (4)			
17	3.92 (m)	3.95 (m)	-0.03
18	3.92 (m)	3.95 (m)	-0.03
19	1.09 (d, 7.0)	1.10 (d, 5.0)	-0.01
17-NH	8.38 (d, 5.8)	8.50 (m)	-0.12
D-Asparagine (5)			
21	4.28 (dt, 7, 7)	4.31 (m)	-0.03
22a	2.77 (dd, 15, 6.2)	2.76 (dd, 14.5, 5.6)	0.01
22b	2.59 (dd, 15, 7.2)	2.61 (dd, 14.5, 6.6)	-0.02
23-NHa	7.41 (br s)	7.47 (br)	-0.06
23-NHb	7.00 (br s)	7.02 (s)	-0.02
D-Alanine (6)			
25	3.95 (dq, 7, 7)	3.95 (m)	0
26	1.17 (d, 7)	1.19 (d, 7.0)	-0.02
25-NH	7.32 (d, 7)	7.45 (m)	-0.07
Guanidino-3-hydroxy-heptadecanoic acid (GHND)			
29a	2.45 (dd, 13.2, 4.9)	2.46 (dd, 13.3, 4.7)	-0.01
29b	2.32 (dd, 13.2, 6.2)	2.31 (dd, 13.6, 6.5)	0.01
30	3.80 (m)	3.81 (m)	-0.01
31	1.36 (2H, m)	1.37 (2H, m)	-0.01
32	1.35 (2H, m)	1.34 (2H, m)	0.01
33-42	1.25 (20H, m)	1.24 (20H, m)	0.01
43	1.42 (2H, m)	1.44 (2H, m)	-0.02
44a	3.07 (dd, 7, 7)	3.06 (br s)	0.01
44b	3.05 (dd, 7, 7)	3.06 (br s)	-0.01
NHCNHNH ₂	7.52 (t, 7)	7.45 (m)	0.07

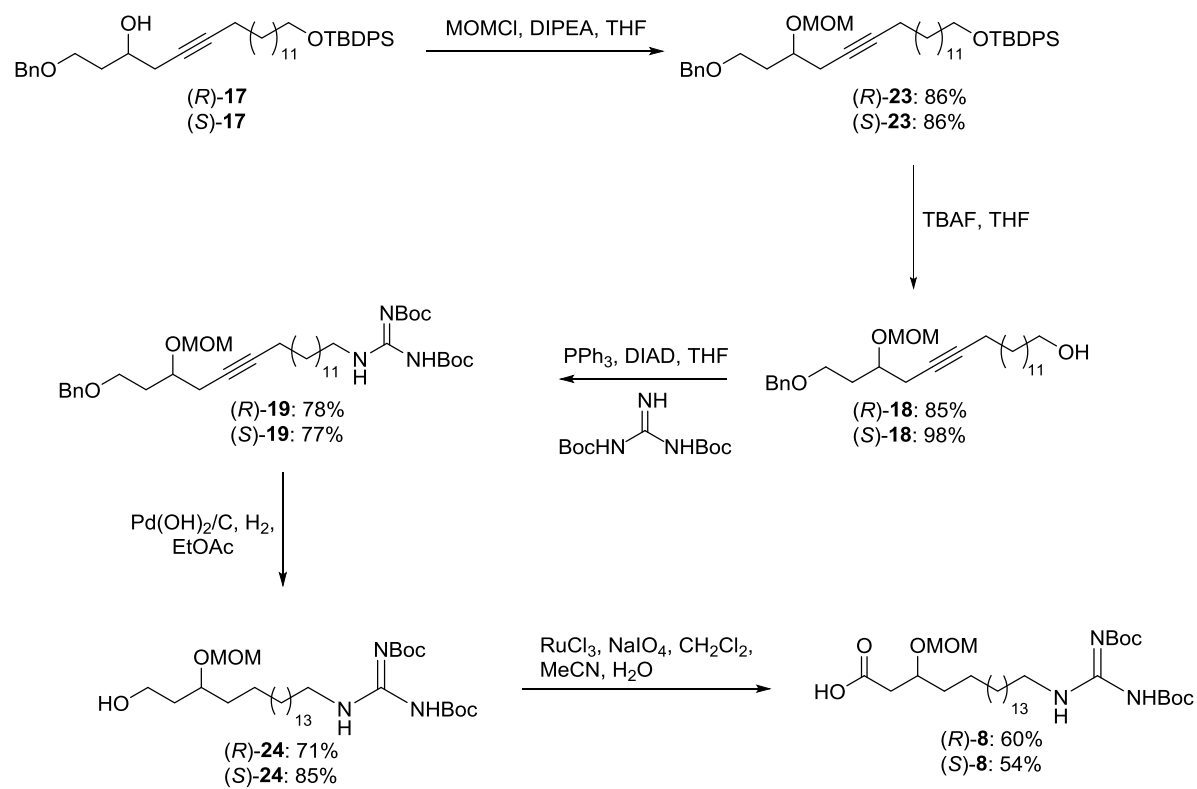
Table S4: ^{13}C NMR comparison of (*R*)-**6** with reported data¹



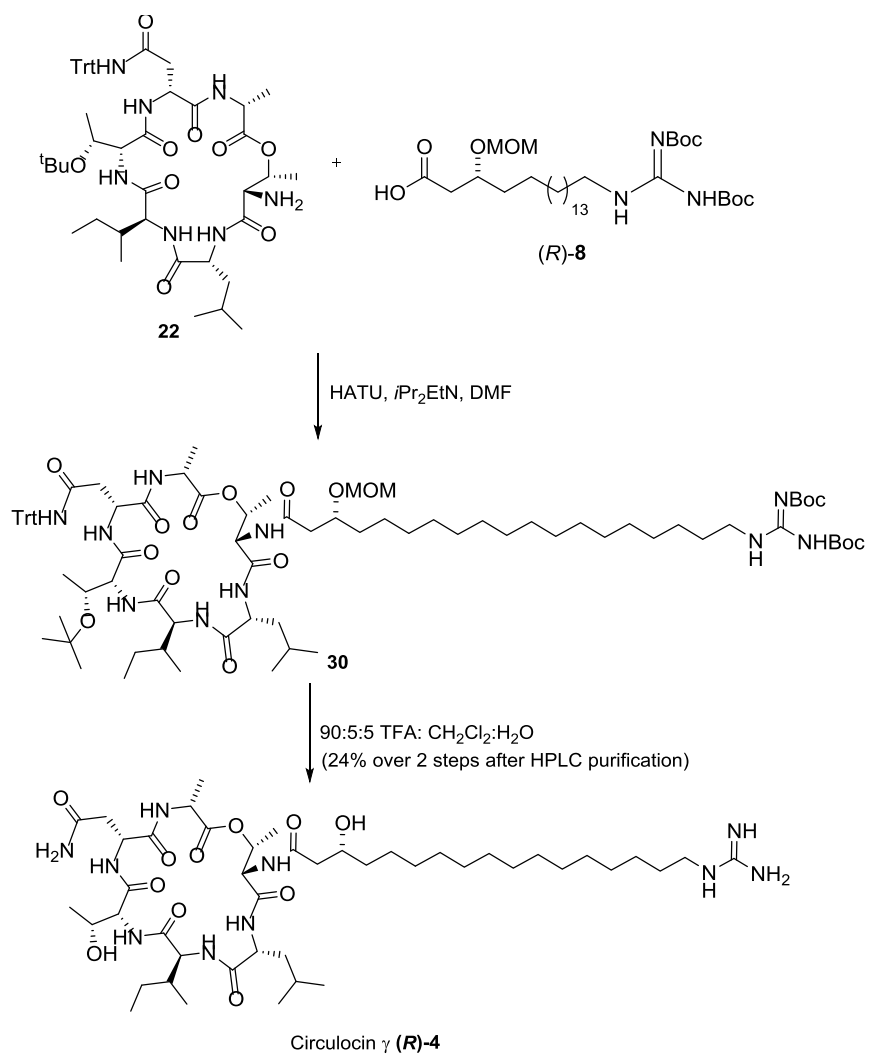
Assignment	Reported Data (D) ¹		(R)-6	
	δ_{C}		δ_{C}	$\Delta\delta_{\text{C}}$ (N-R)
L-Threonine (1)				
1	56.6		56.5	0.0
2	70.3		70.3	0.0
3	16.0		15.9	0.1
4	168.0		167.8	0.2
D-Leucine (2)				
5	50.1		50.1	0.0
6	42.3		42.1	0.2
7	24.0		23.9	0.1
8	22.6		22.3	0.3
9	22.2		22.3	-0.1
10	171.6		171.6	0.0
L-Isoleucine (3)				
11	56.7		56.7	0.0
12	35.8		35.6	0.2
13	15.3		15.2	0.1
14	24.2		24.1	0.1
15	10.6		10.6	0.0
16	173.0		172.9	0.1
D-allo-Threonine (4)				
17	60.1		60.1	0.0
18	65.6		65.4	0.2
19	19.3		19.2	0.1
20	170.2		170.1	0.1
D-Asparagine (5)				
21	50.4		50.3	0.1
22	36.5		36.5	0.0
23	172.4		172.3	0.1
24	169.7		169.5	0.2
D-Alanine (6)				
25	47.8		47.6	0.2
26	16.9		16.7	0.2
27	170.6		170.4	0.2
Guanidino-3-hydroxy-heptadecanoic acid (GHHD)				
28	171.6		171.5	0.1
29	42.8		42.6	0.2
30	67.5		67.5	0.0
31	36.4		36.5	-0.1
32	25.3		25.1	0.2
33-41	28.5 - 29.0		28.6	0.2
42	25.9		25.8	0.1
43	28.3		28.3	0.0
44	40.6		40.4	-0.2
NHCNHNH ₂	156.6		n.d.	n.d.

Reaction Schemes S1-S4

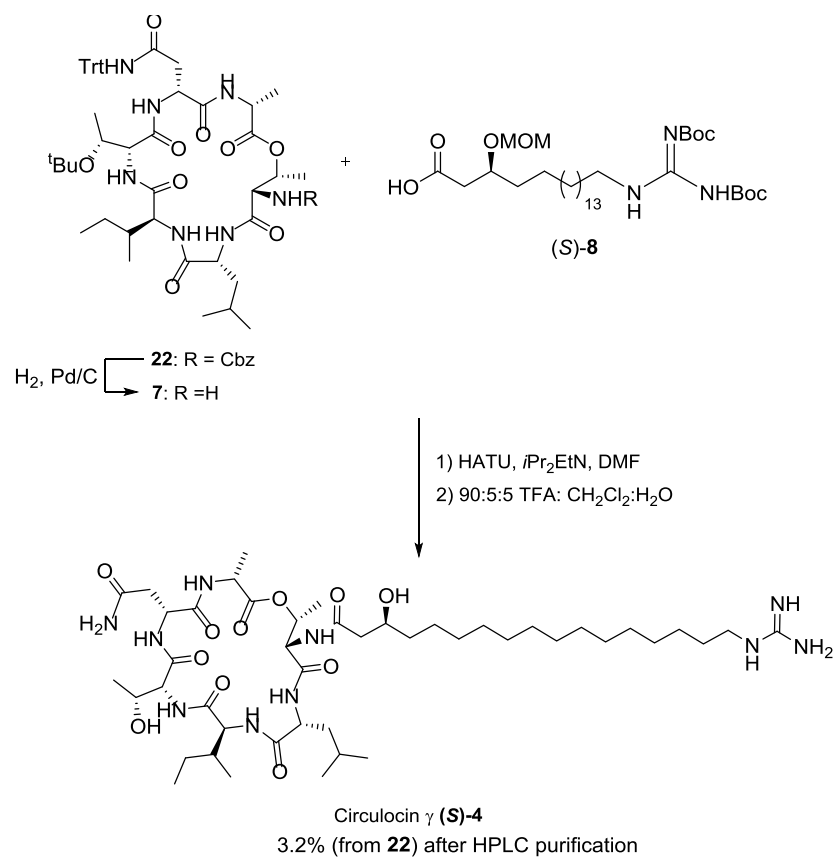
Scheme S1



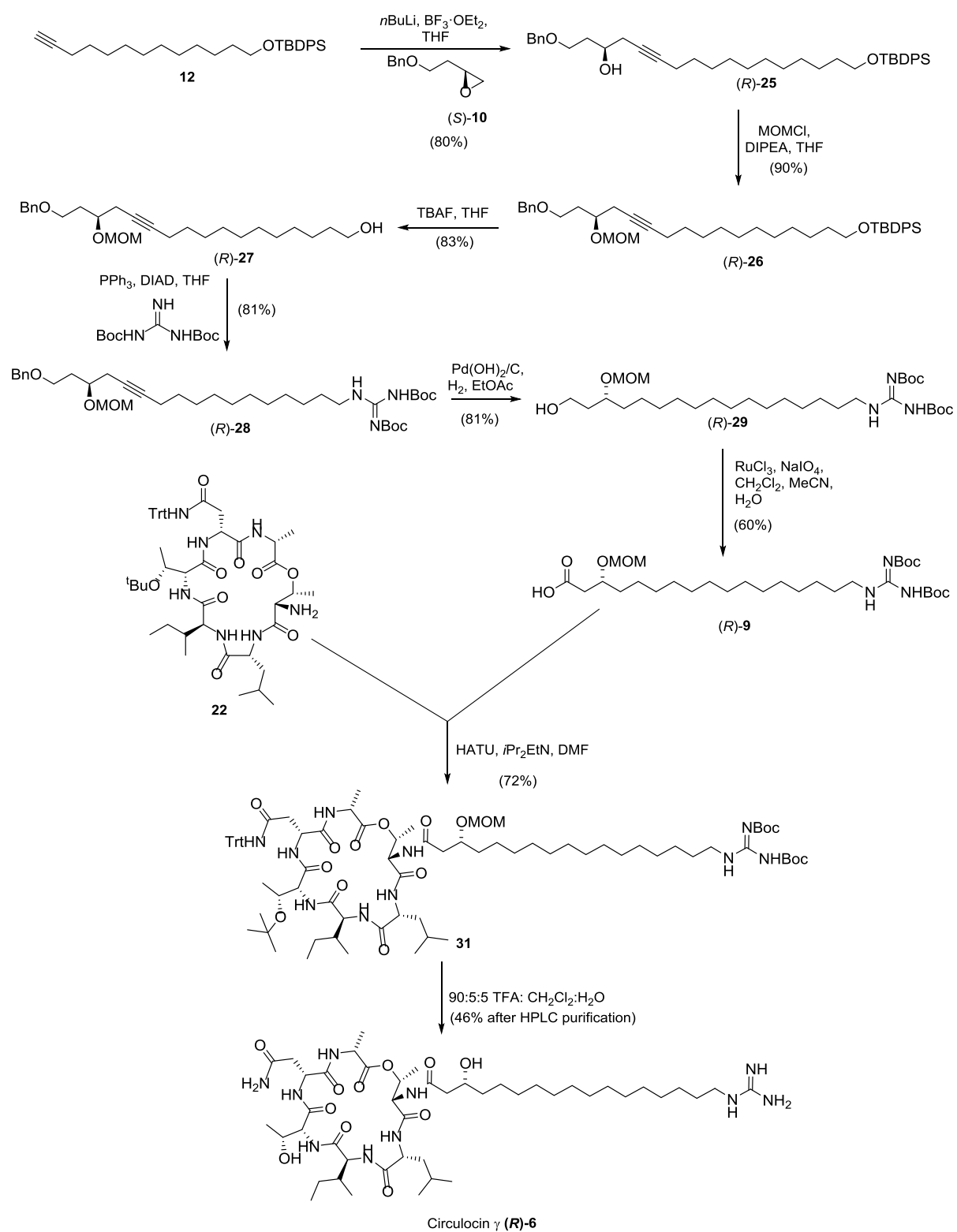
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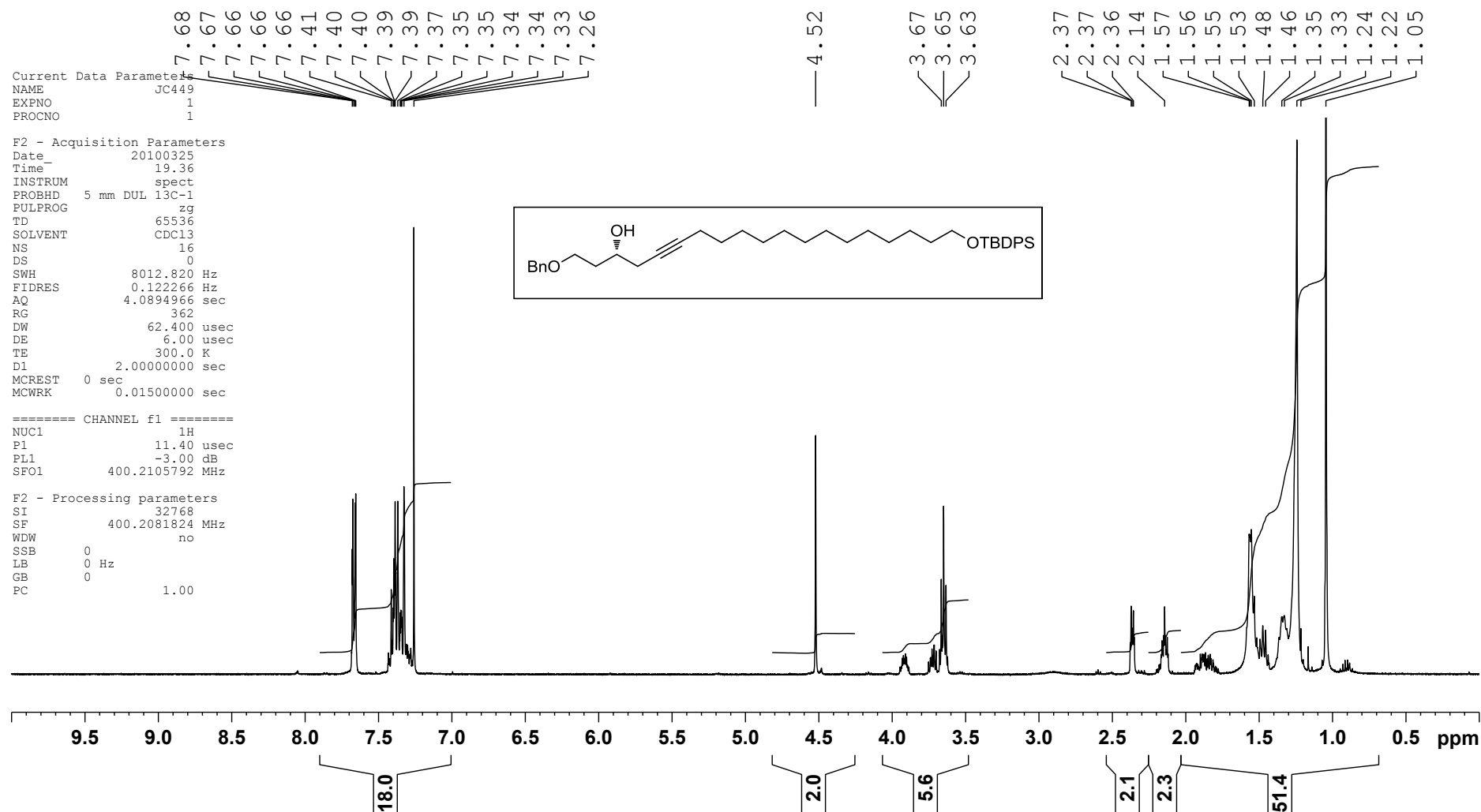
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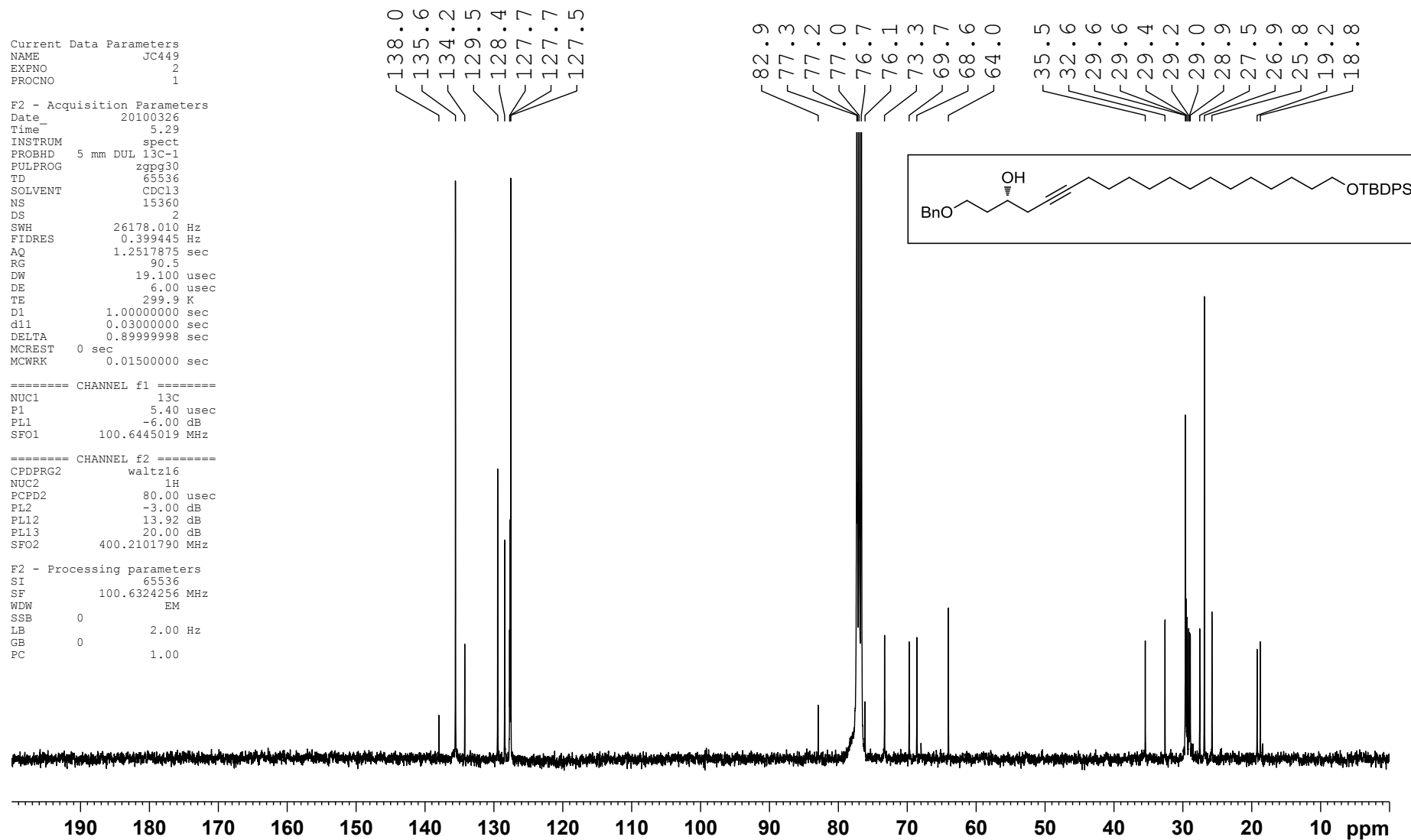
Scheme S4:



1-(Benzyloxy)-19-[(*tert*-butyldiphenylsilyl)oxy]nonadec-5-yn-3-ol (*R*)-17 (400 MHz, CDCl₃)



(R)-1-(Benzyloxy)-19-[(tert-butyldiphenylsilyl)oxy]nonadec-5-yn-3-ol (R)-17 (¹³C NMR 100 MHz, CDCl₃)



(R)-1-(Benzyloxy)-19-[(*tert*-butyldiphenylsilyl)oxy]nonadec-5-yn-3-ol (R)-17 (DEPT-135, CDCl₃)

Current Data Parameters
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EXPNO 3
PROCNO 1

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Time_ 10.09
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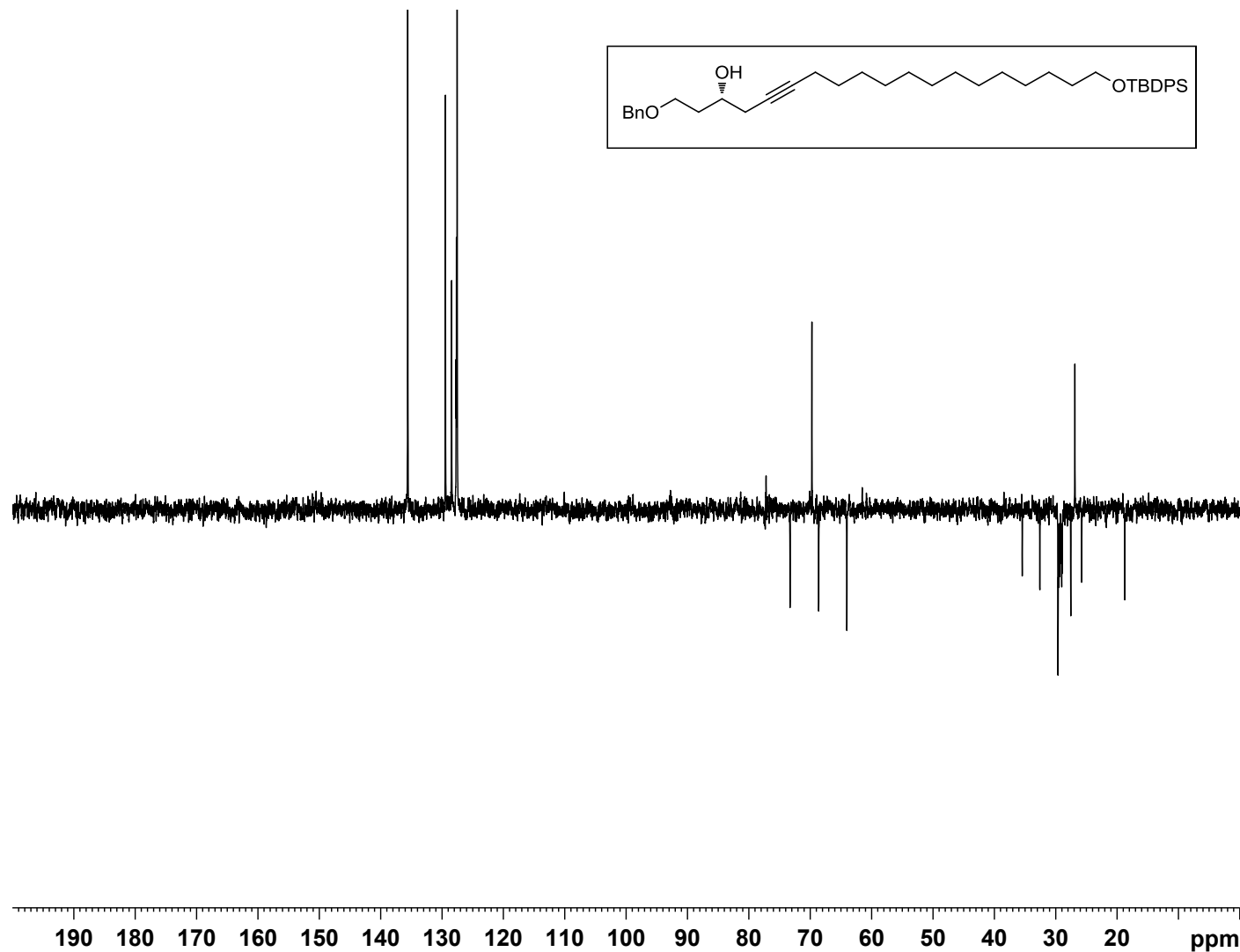
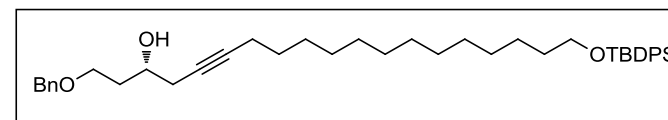
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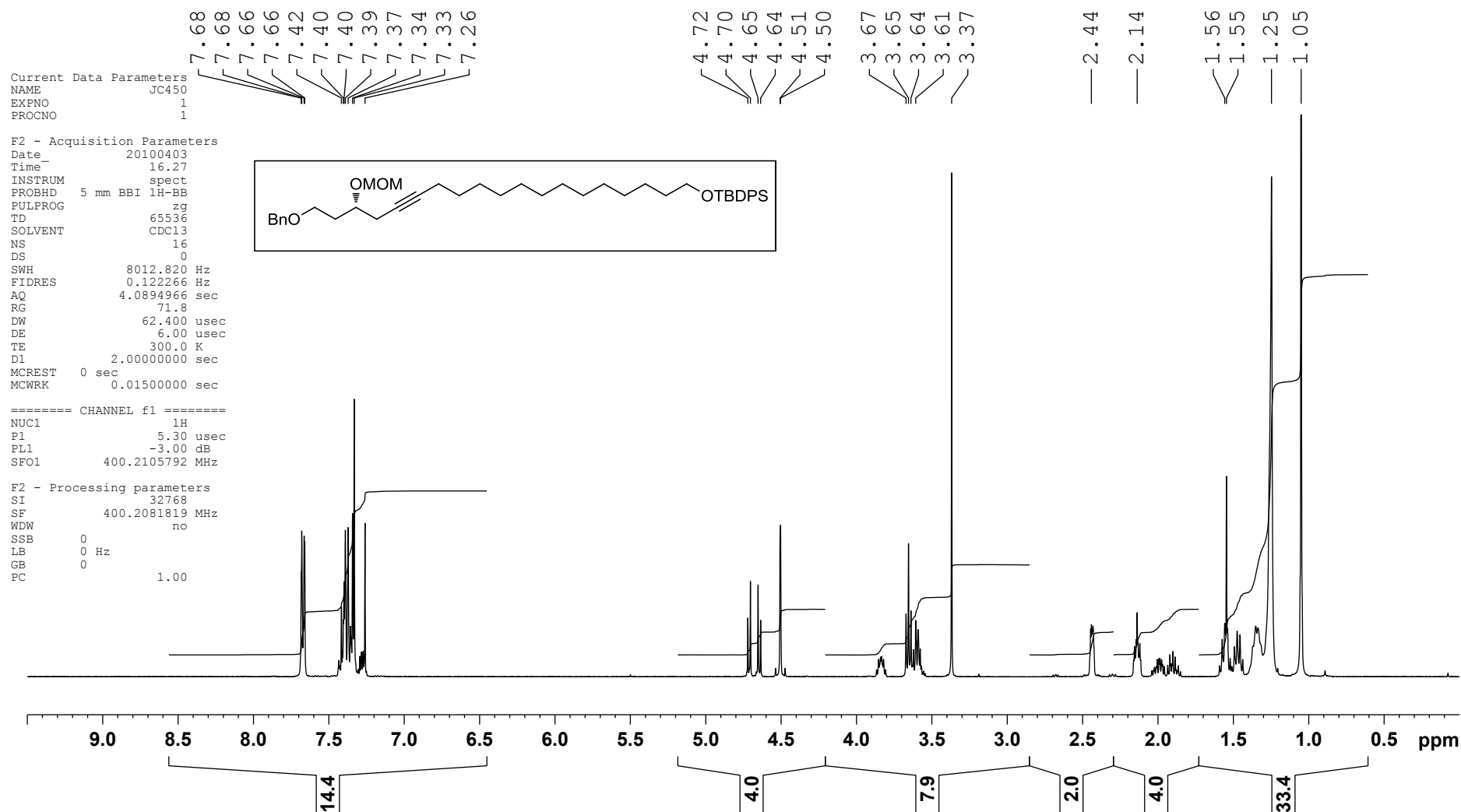
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PCPD2 80.00 usec
PL2 -3.00 dB
PL12 13.92 dB
SFO2 400.2101790 MHz

F2 - Processing parameters

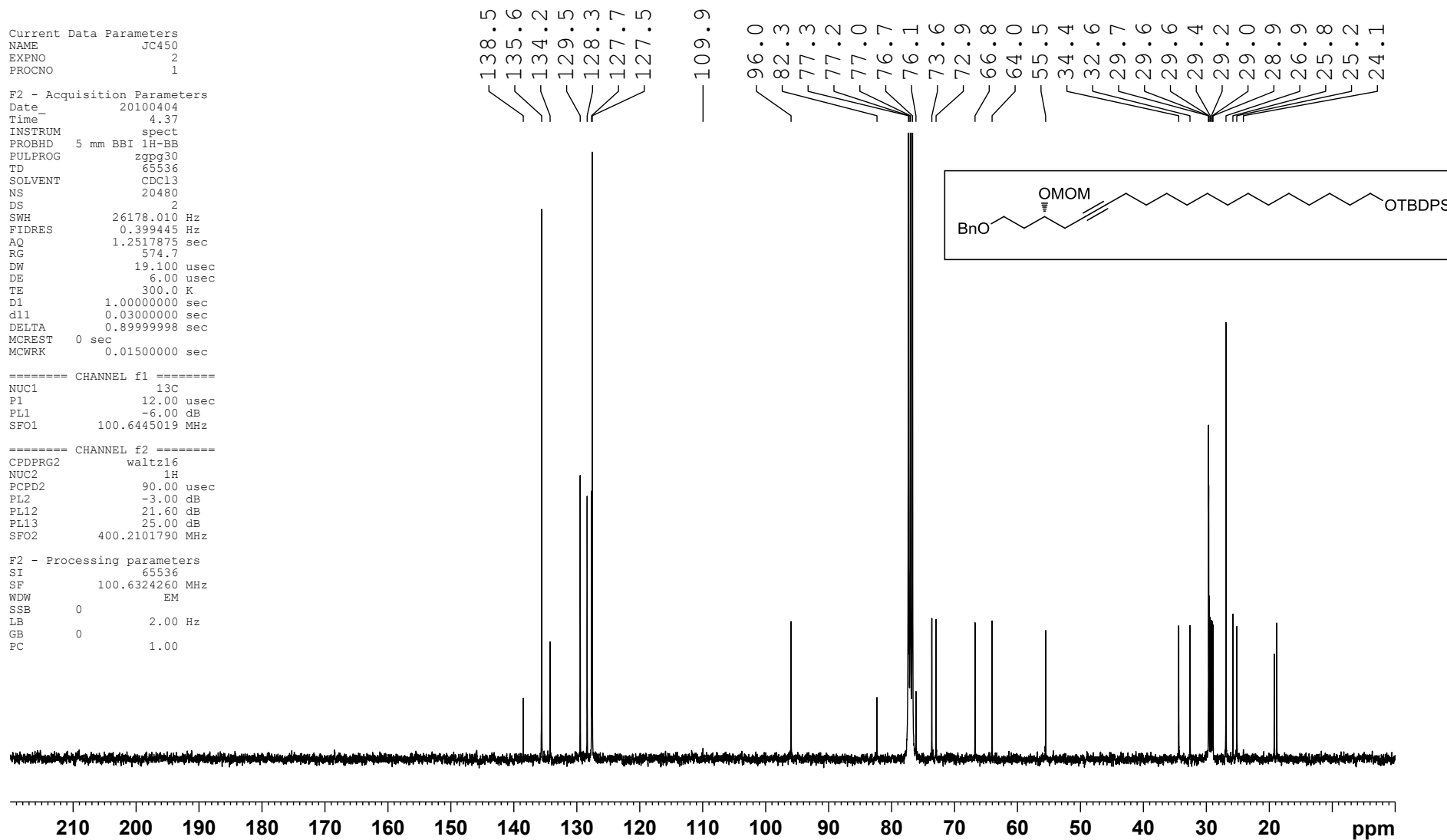
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WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00



1-Benzyloxy-19-(*tert*-butyldiphenylsiloxy)-3-(methoxymethoxy)-nonadec-5-yne (*R*)-23 (¹H NMR 400 MHz, CDCl₃)



1-Benzyloxy-19-(*tert*-butyldiphenylsiloxy)-3-(methoxymethoxy)-nonadec-5-yne (*R*)-23 (¹³C NMR100 MHz, CDCl₃)



(R)-1-Benzyloxy-19-(tert-butyldiphenylsiloxy)-3-(methoxymethoxy)-nonadec-5-yne 9 (R)-23 (DEPT-135, CDCl₃)

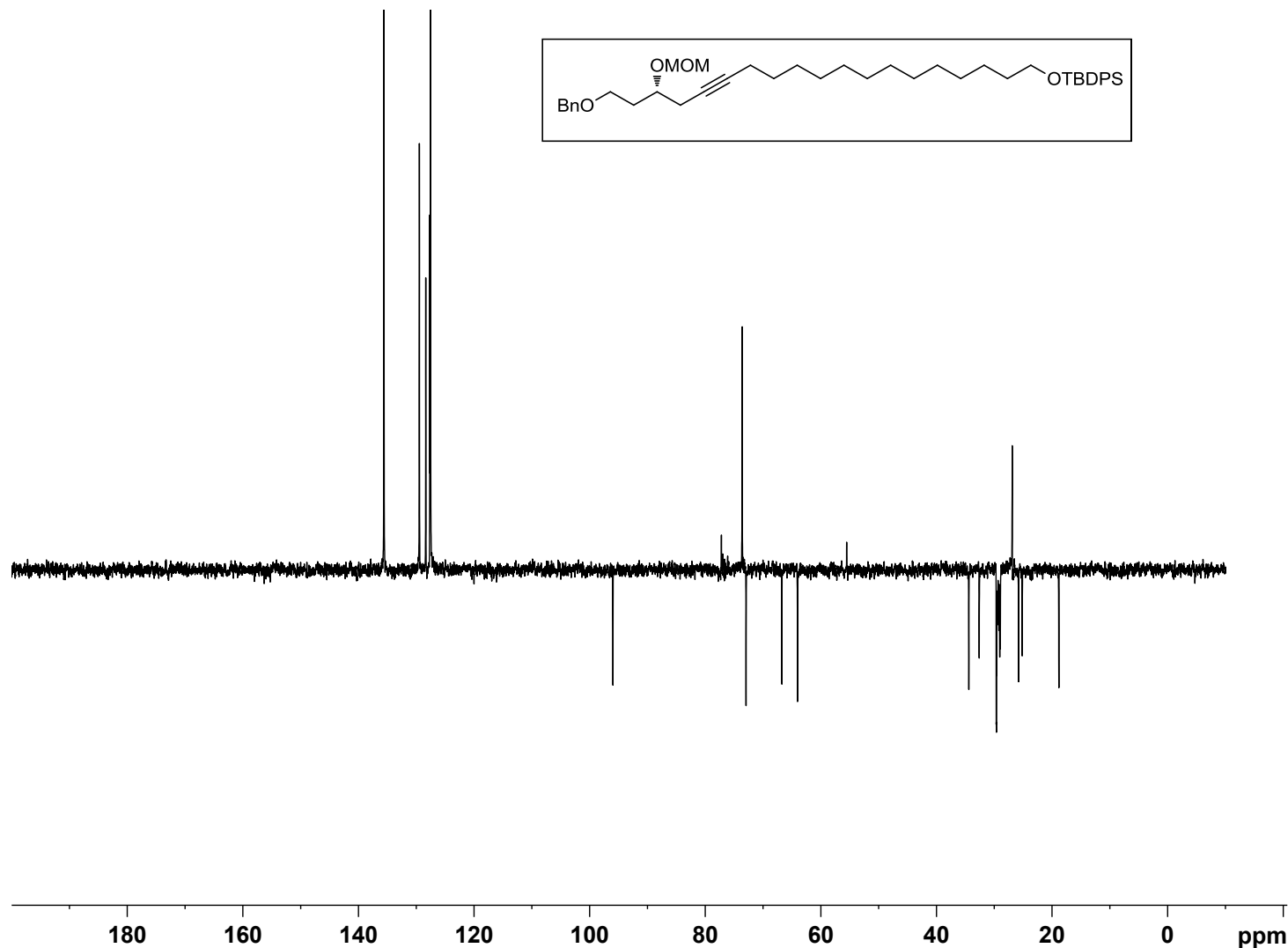
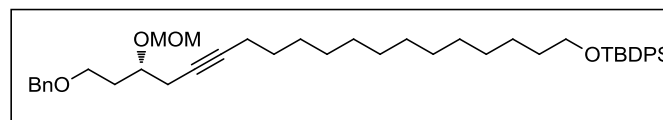
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 DE 6.00 usec
 TE 300.0 K
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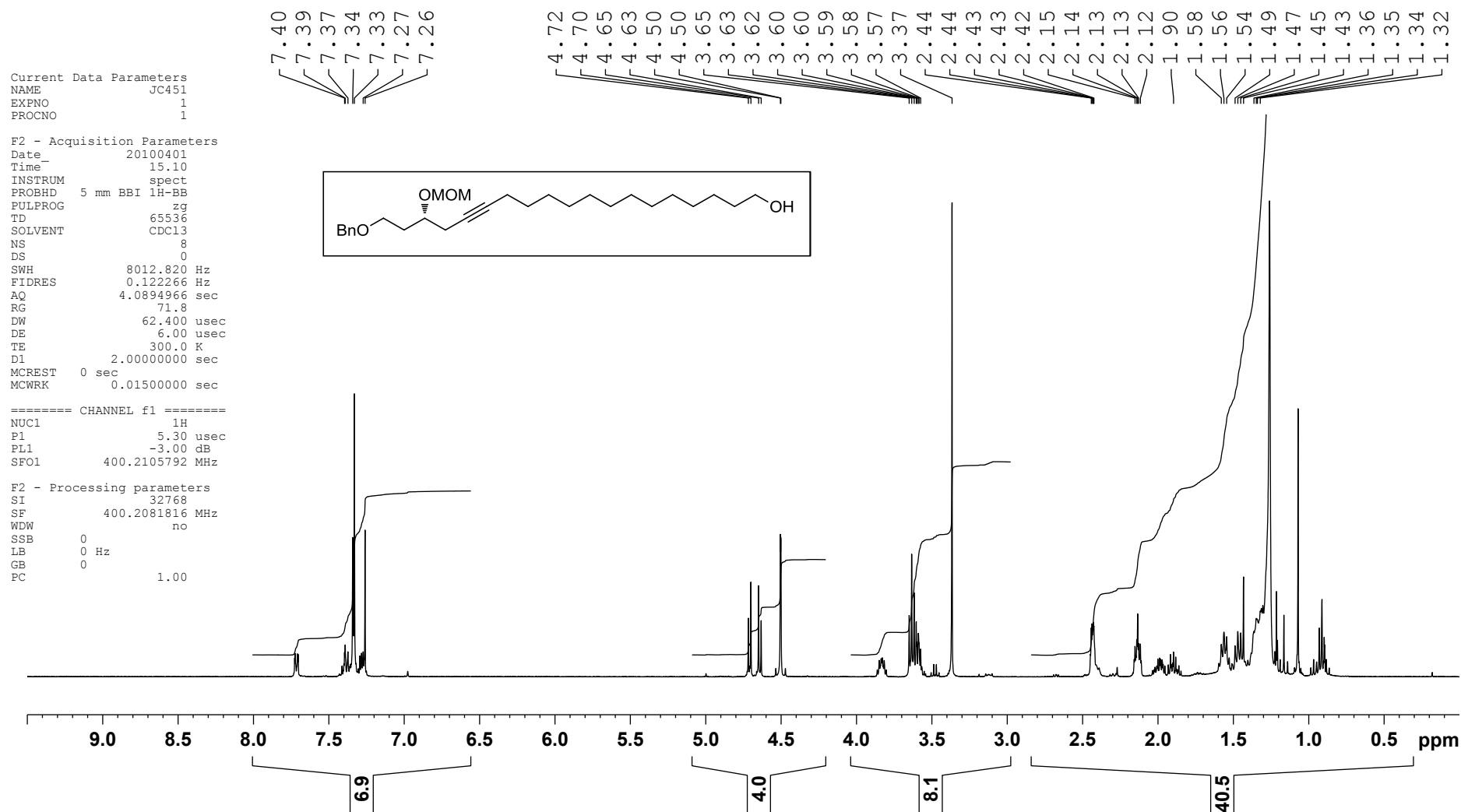
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 NUC2 1H
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 p4 10.60 usec
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 21.60 dB
 SFO2 400.2101790 MHz

F2 - Processing parameters
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 SF 100.6324260 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00



19-(Benzyloxy)-13-(methoxymethoxy)-pentadec-14-yn-1-ol (*R*)-18 (¹H NMR 400 MHz, CDCl₃)



19-(Benzyloxy)-13-(methoxymethoxy)-pentadec-14-yn-1-ol (*R*)-18 (^{13}C NMR 100 MHz, CDCl_3)

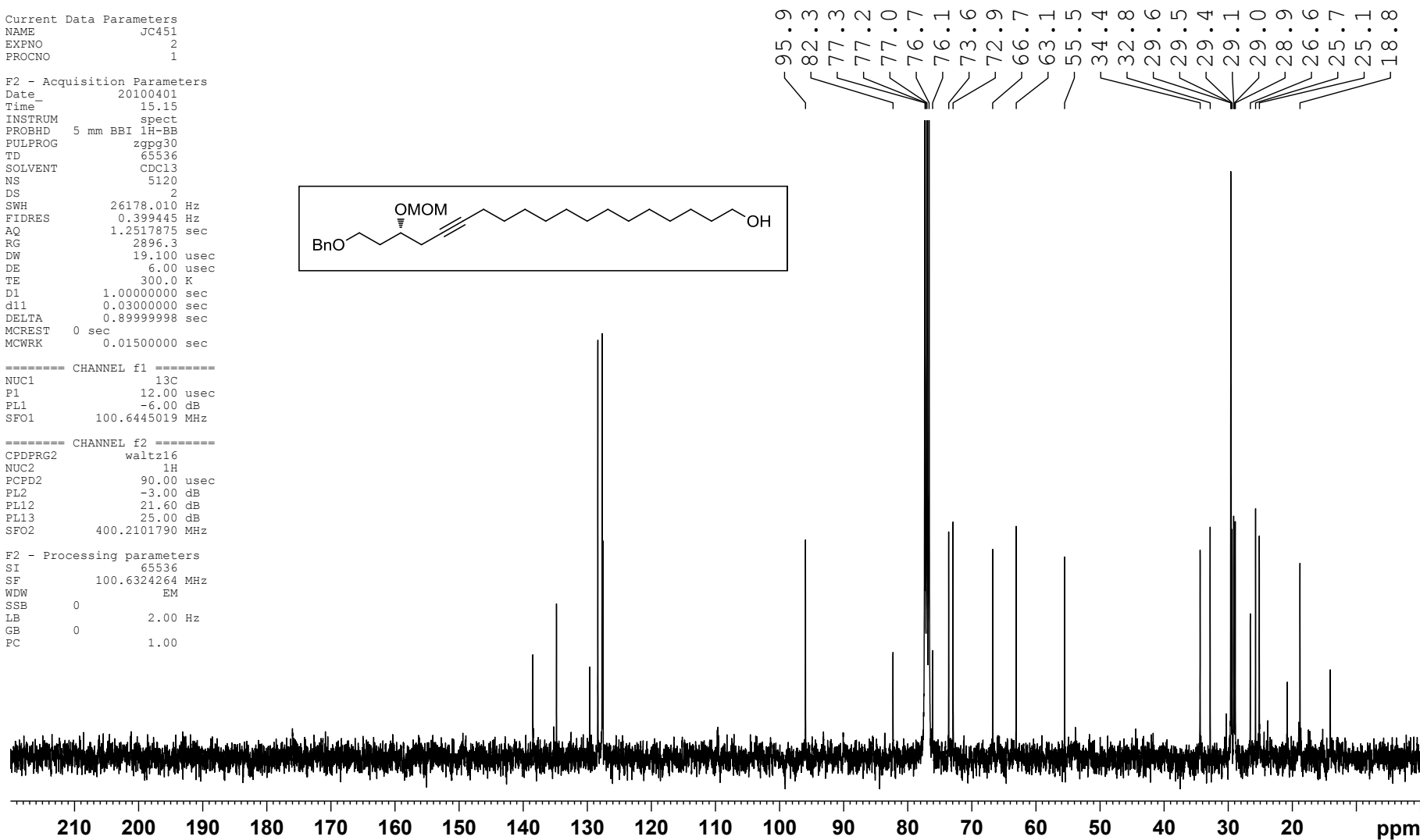
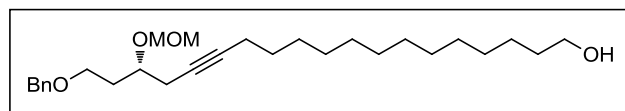
Current Data Parameters
NAME JC451
EXPNO 2
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl_3
NS 5120
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 2896.3
DW 19.100 usec
DE 6.00 usec
TE 300.0 K
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===== CHANNEL f1 =====
NUC1 ^{13}C
P1 12.00 usec
PL1 -6.00 dB
SFO1 100.6445019 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 21.60 dB
PL13 25.00 dB
SFO2 400.2101790 MHz

F2 - Processing parameters
SI 65536
SF 100.6324264 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



19-(Benzyloxy)-13-(methoxymethoxy)-pentadec-14-yn-1-ol (*R*)-18 (DEPT-135, CDCl₃)

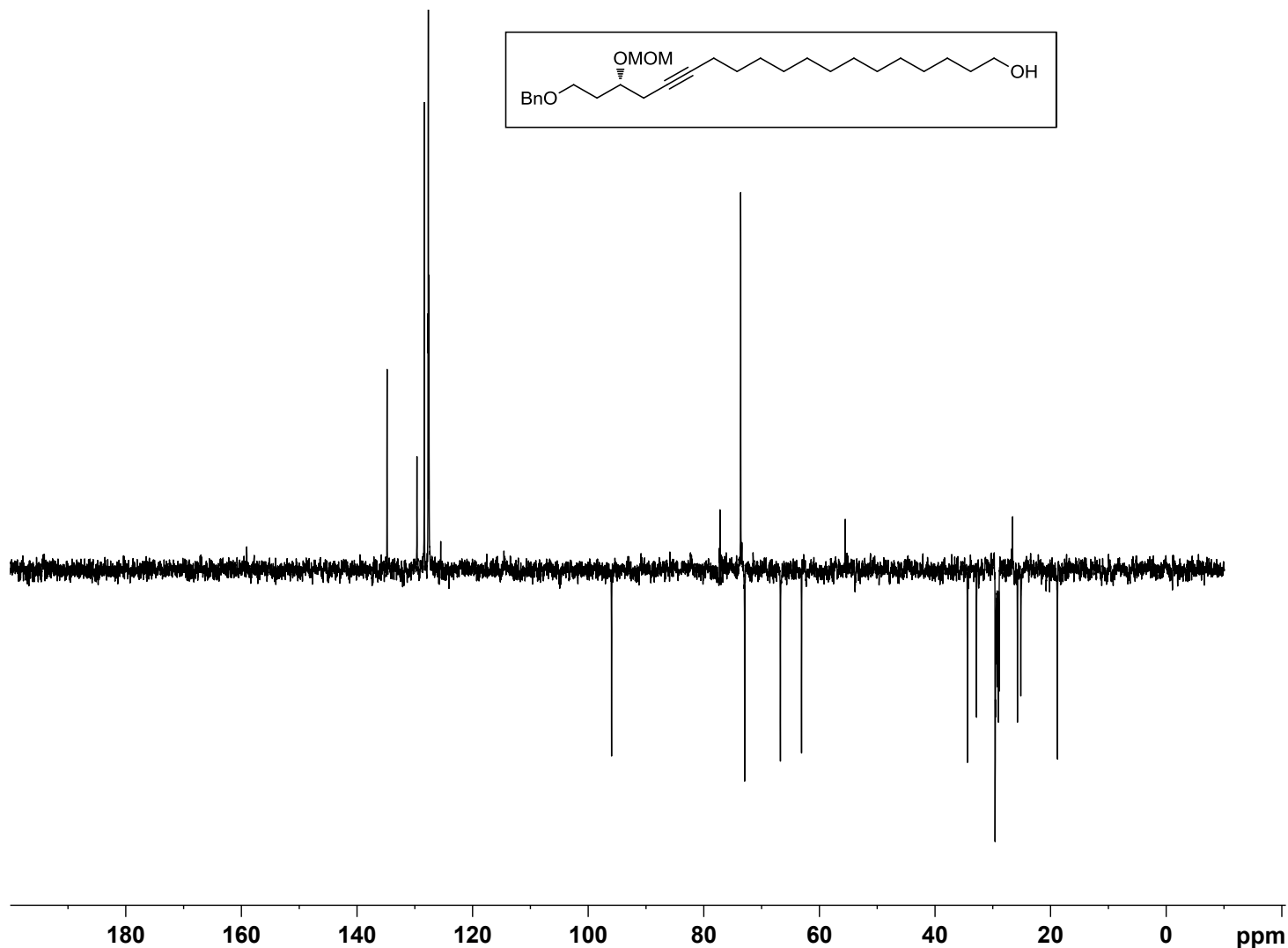
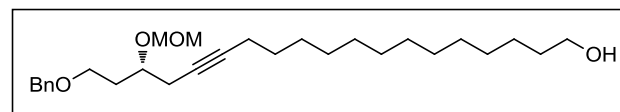
Current Data Parameters
 NAME JC451
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100402
 Time_ 4.35
 INSTRUM spect
 PROBHD 5 mm BBI 1H-BB
 PULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 10240
 DS 2
 SWH 26178.010 Hz
 FIDRES 0.399445 Hz
 AQ 1.2517875 sec
 RG 5160.6
 DW 19.100 usec
 DE 6.00 usec
 TE 300.0 K
 CNST2 145.0000000
 D1 2.00000000 sec
 d2 0.00344828 sec
 d12 0.00002000 sec
 DELTA 0.00001528 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.00 usec
 p2 24.00 usec
 PL1 -6.00 dB
 SFO1 100.6445019 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P3 5.30 usec
 p4 10.60 usec
 PCPD2 90.00 usec
 PL2 -3.00 dB
 PL12 21.60 dB
 SFO2 400.2101790 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6324260 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00



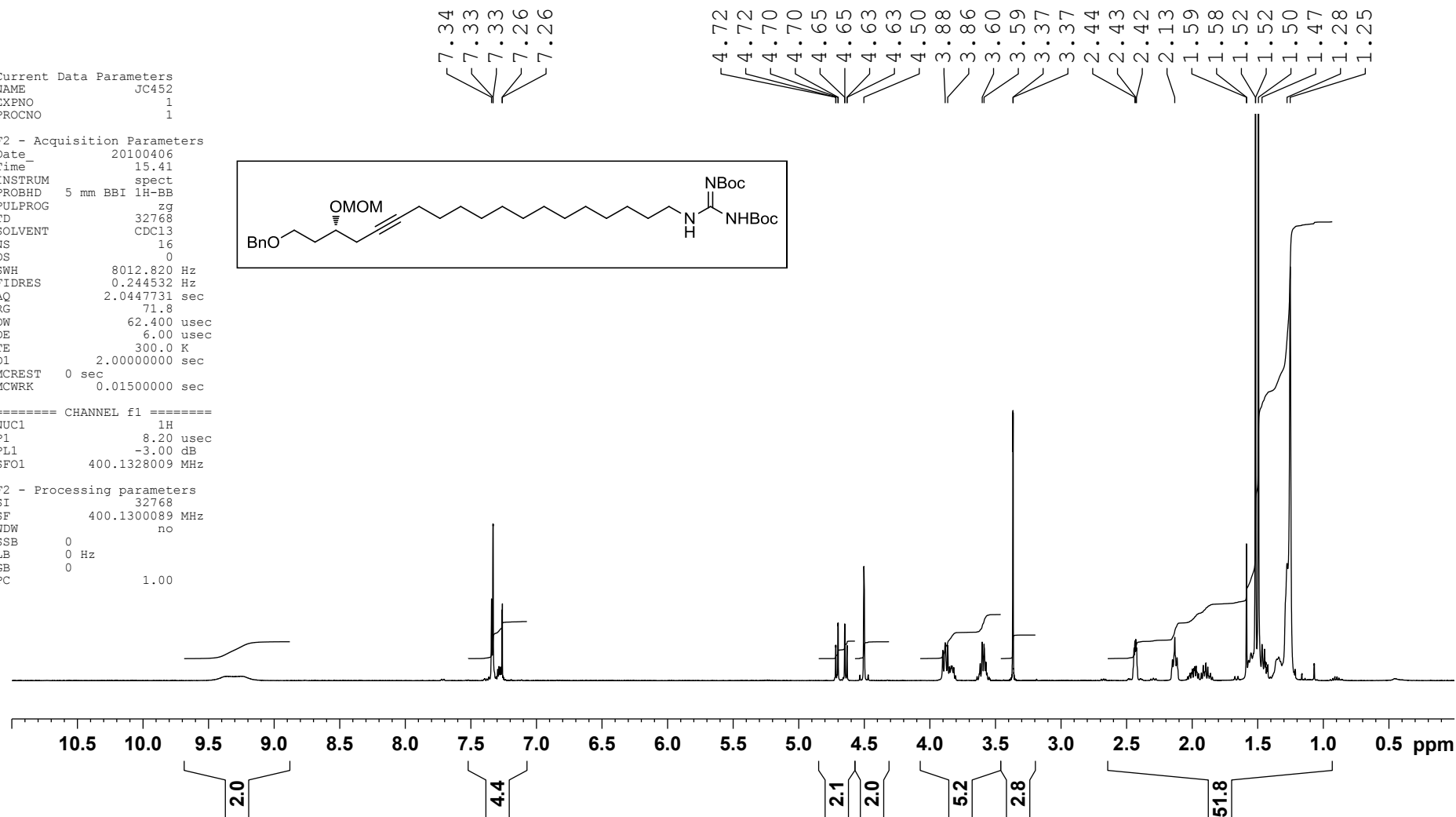
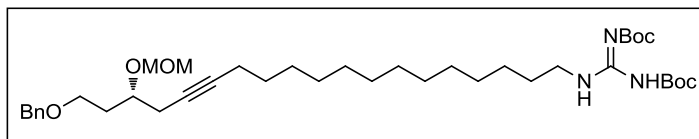
1-(Benzyloxy)-19-{[N,N'-bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-nonadec-5-yne (*R*)-19 (¹H NMR 400 MHz, CDCl₃)

Current Data Parameters
NAME JC452
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100406
Time_ 15.41
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl₃
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 71.8
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 32768
SF 400.1300089 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



1-(Benzyloxy)-19- $\{[N,N'$ -bis(*tert*-butoxycarbonyl)]guanidino $\}$ -3-(methoxymethoxy)-nonadec-5-yne (*R*)-19 (^{13}C NMR 100 MHz, CDCl_3)

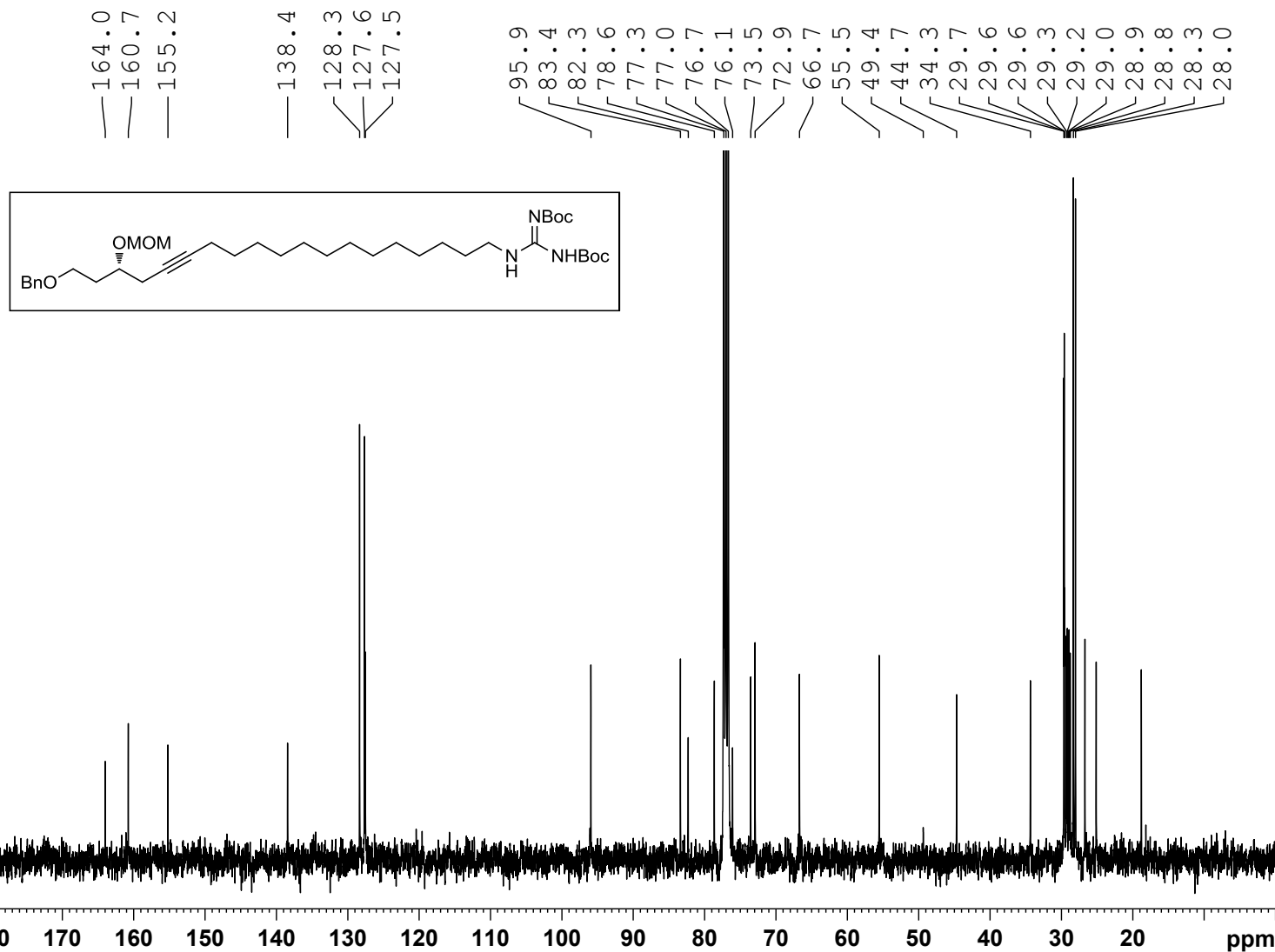
Current Data Parameters
NAME JC452
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100406
Time 15.48
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 4898
DS 0
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^{13}C
P1 12.60 usec
PL1 -6.00 dB
SFO1 100.6248025 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



1-(Benzyloxy)-19- $\{[N,N'$ -bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-nonadec-5-yne (*R*)-19 (DEPT-135, CDCl₃)

Current Data Parameters

NAME JC452
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100407
Time 5.24
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG dept135
TD 65536
SOLVENT CDCl₃
NS 10240
DS 4
SWH 28248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 5792.6
DW 17.700 usec
DE 6.00 usec
TE 300.0 K
CNST2 140.0000000
D1 2.50000000 sec
d2 0.00357143 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
MCREST 0 sec
MCWRK 0.01500000 sec

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

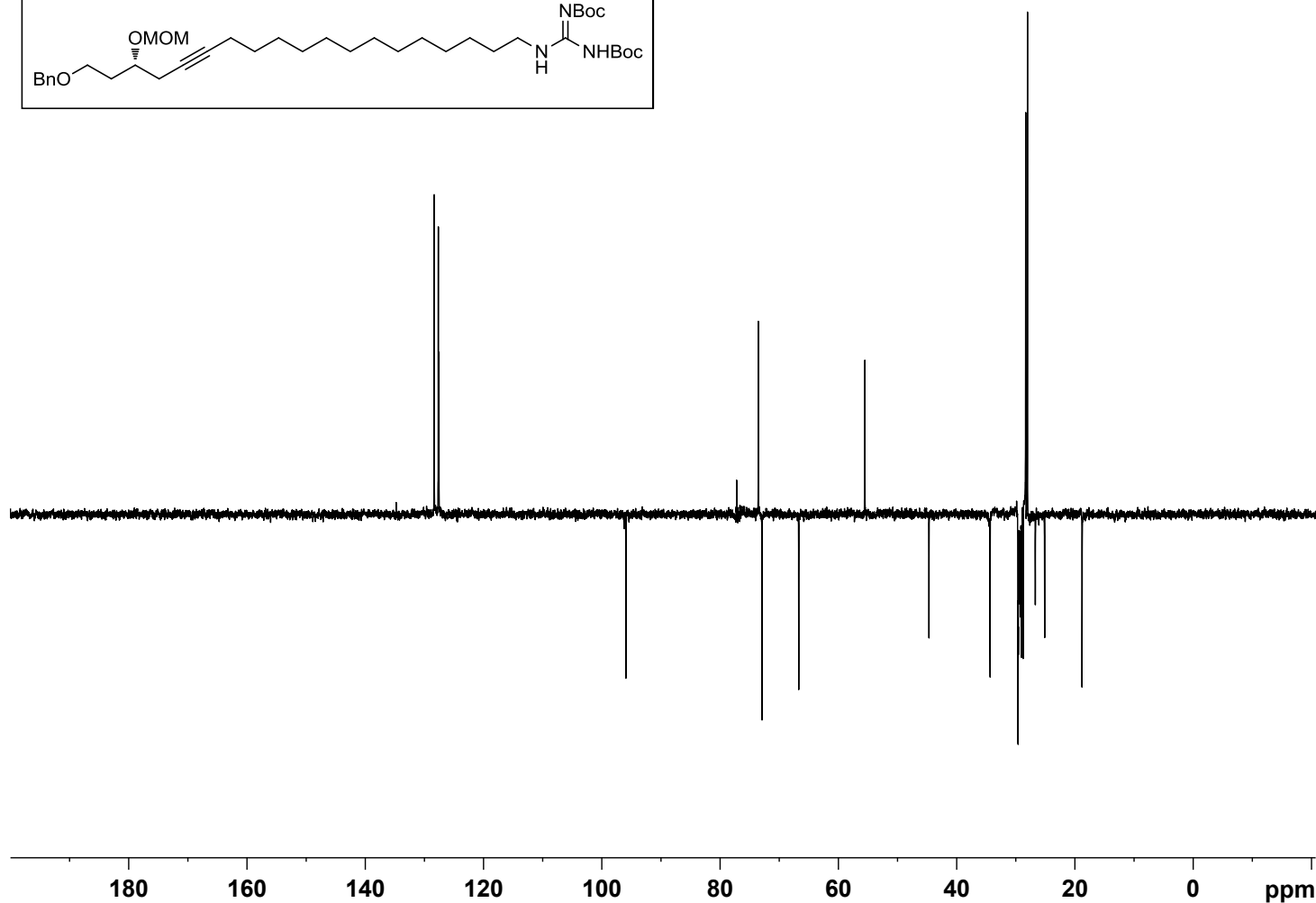
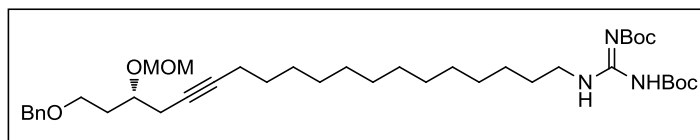
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 100.6248025 MHz

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

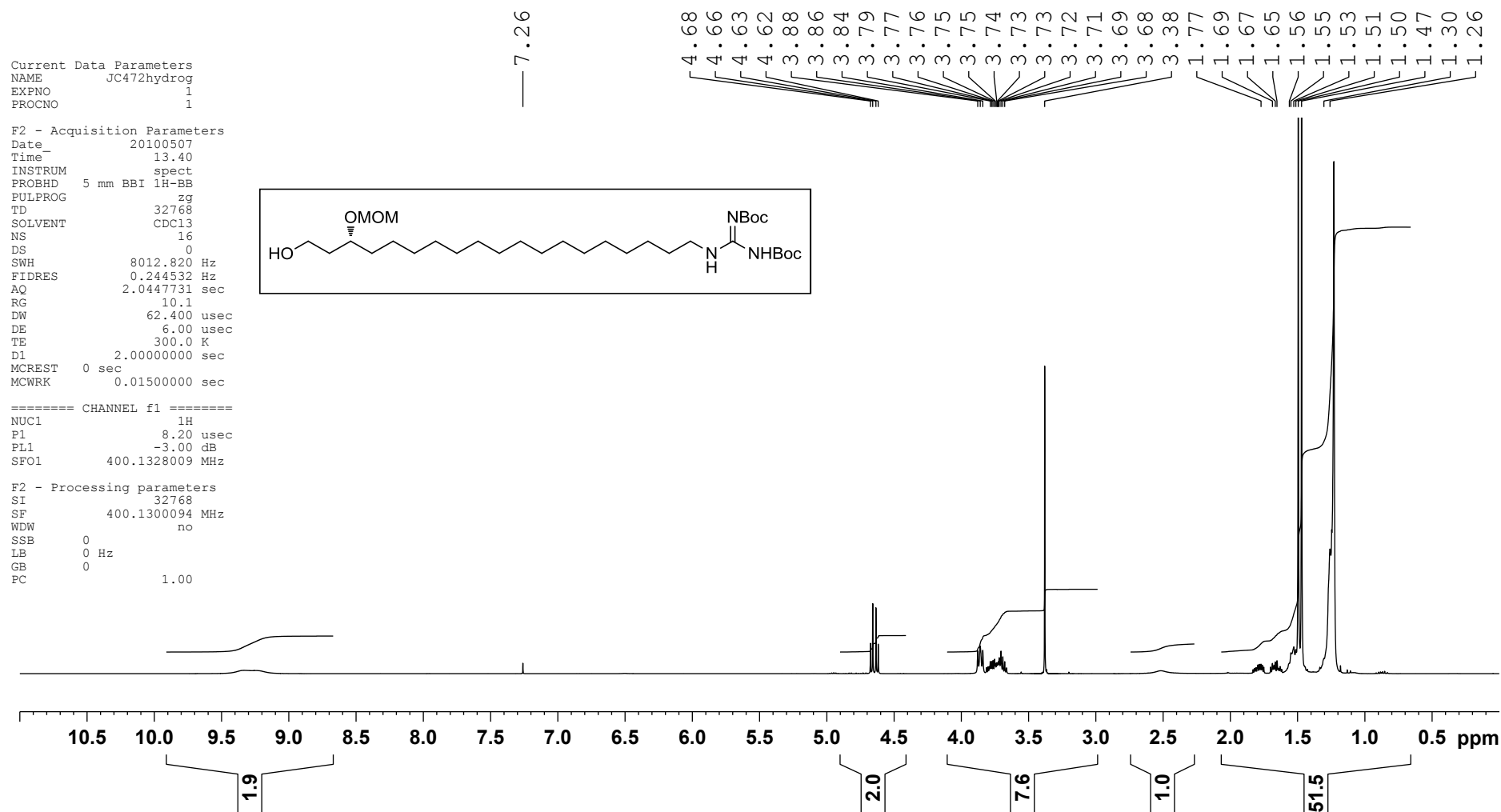
CPDPRG2 waltz16
NUC2 1H
P3 6.90 usec
p4 13.80 usec
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 400.1328009 MHz

F2 - Processing parameters

SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



19- $\{[N,N'$ -Bis(*tert*-butoxycarbonyl)]guanidino $\}$ 3-(methoxymethoxy)-nonadecan-1-ol (*R*)-24 (^1H NMR 400 MHz, CDCl_3)



(R)-19-{[N,N'-Bis(*tert*-butoxycarbonyl)]guanidino}3-(methoxymethoxy)-nonadecan-1-ol (R)-24 (^{13}C NMR 100 MHz, CDCl_3)

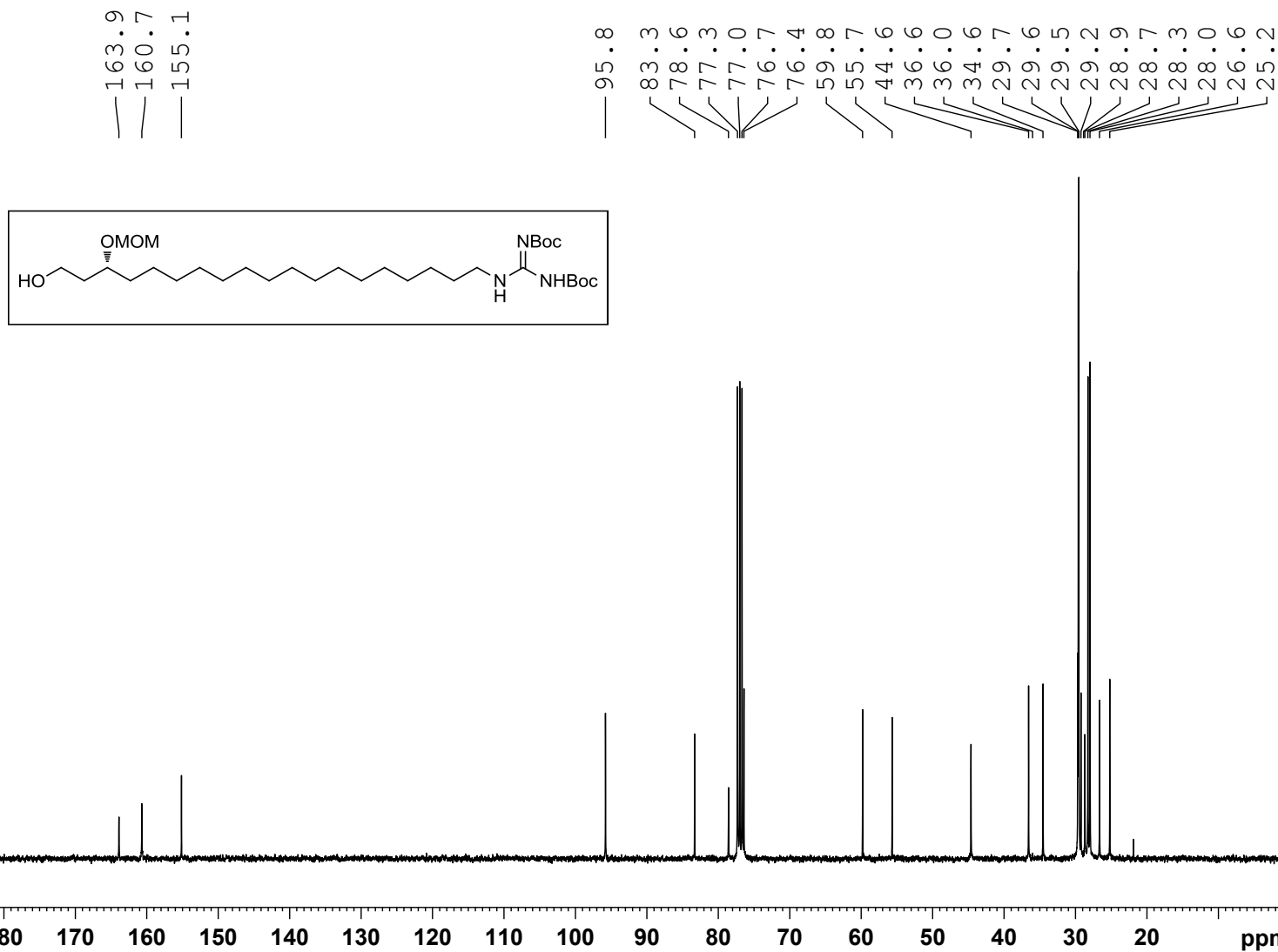
Current Data Parameters
NAME JC472hydrog
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100507
Time_ 13.44
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zgdc30
TD 65536
SOLVENT C6D6
NS 1956
DS 0
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 4096
DW 19.100 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^{13}C
P1 12.60 usec
PL1 -6.00 dB
SFO1 100.6248025 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



(R)-19-{[N,N'-Bis(*tert*-butoxycarbonyl)]guanidino}3-(methoxymethoxy)-nonadecan-1-ol (R)-24 (DEPT-135, CDCl₃)

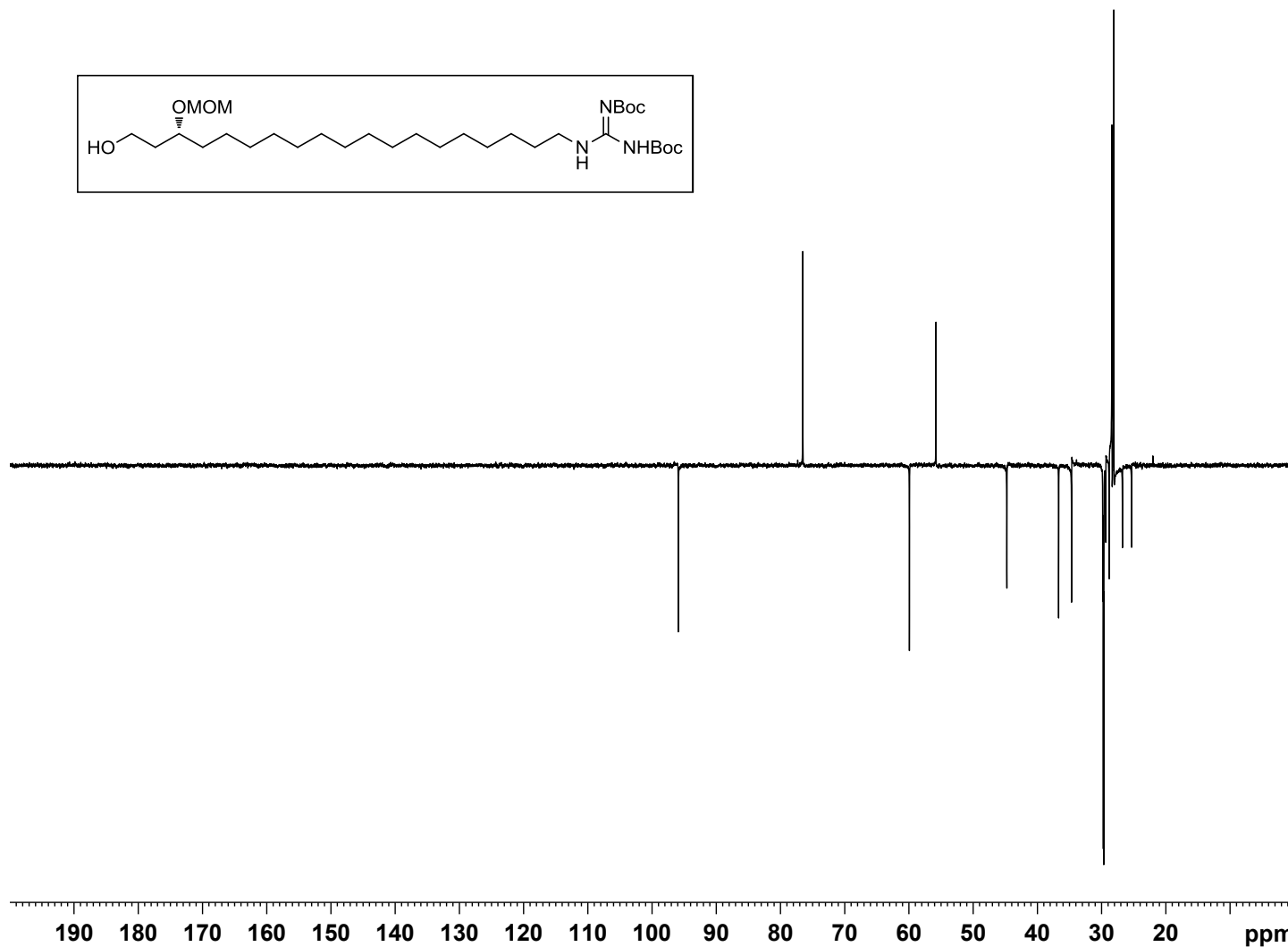
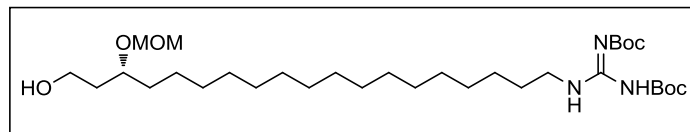
Current Data Parameters
NAME JC472hydrog
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100507
Time 16.13
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 1270
DS 4
SWH 28248.588 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 14596.5
DW 17.700 usec
DE 6.00 usec
TE 300.0 K
CNST2 140.0000000
D1 2.50000000 sec
d2 0.00357143 sec
d12 0.00002000 sec
DELTA 0.00001604 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.60 usec
p2 25.20 usec
PL1 -6.00 dB
SFO1 100.6248025 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 6.90 usec
p4 13.80 usec
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 32768
SF 100.6127617 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



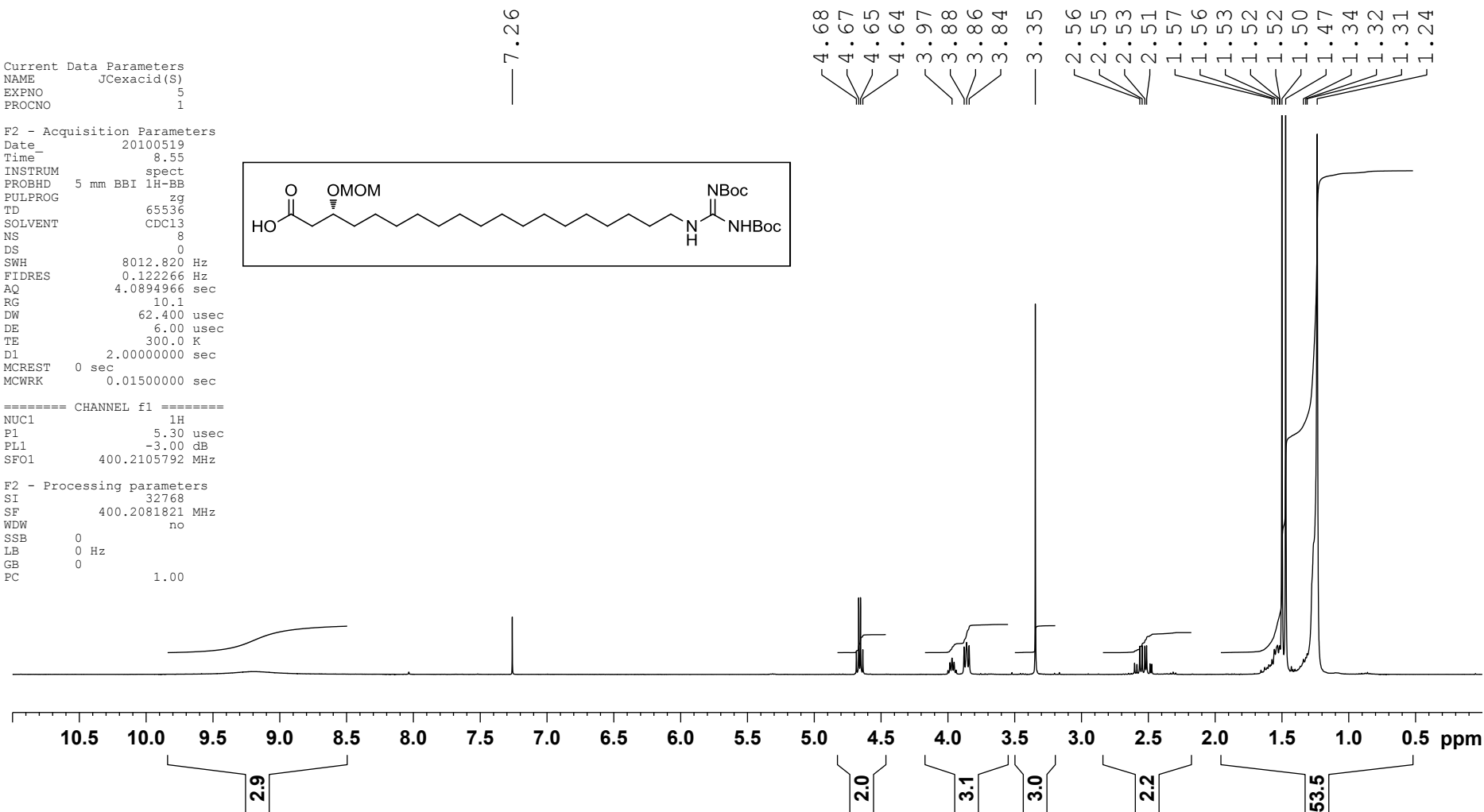
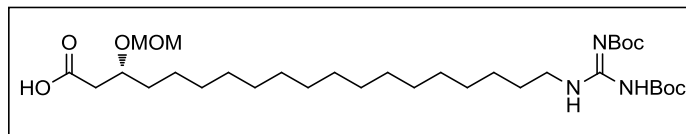
19- $\{[N,N'$ -Bis(*tert*-butoxycarbonyl)]guanidino $\}$ 3-(methoxymethylenoxy)-nonadecanoic acid (*R*)-8 (^1H NMR 400 MHz, CDCl_3)

Current Data Parameters
NAME JCexacid(S)
EXPNO 5
PROCNO 1

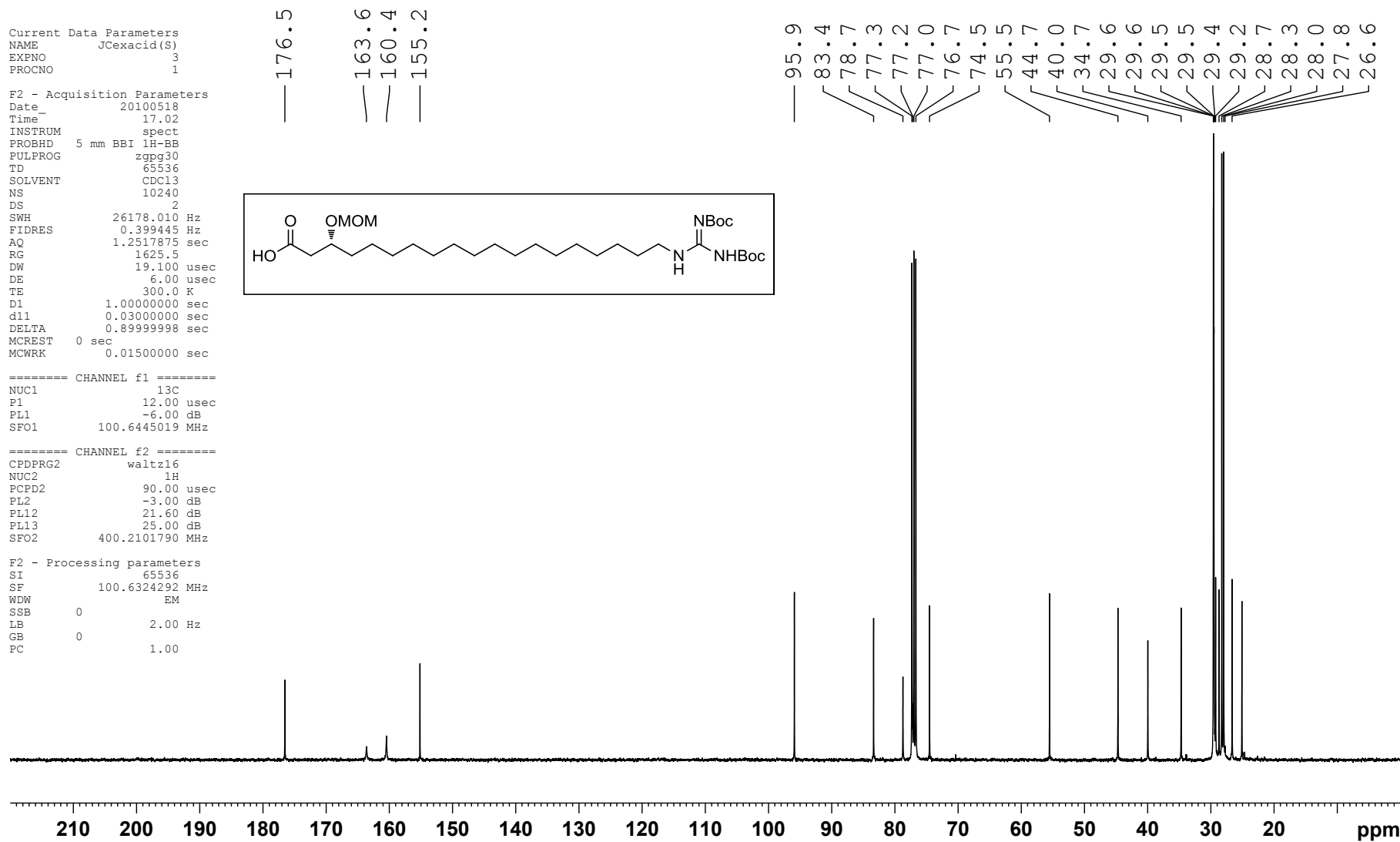
F2 - Acquisition Parameters
Date_ 20100519
Time 8.55
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 65536
SOLVENT CDCl_3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 10.1
DW 62.400 μsec
DE 6.00 μsec
TE 300.0 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^1H
P1 5.30 μsec
PL1 -3.00 dB
SFO1 400.2105792 MHz

F2 - Processing parameters
SI 32768
SF 400.2081821 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



19- $\{[N,N'$ -Bis(*tert*-butoxycarbonyl)]guanidino $\}$ 3-(methoxymethylenoxy)-nonadecanoic acid (*R*)-8 (^{13}C NMR 100 MHz, CDCl_3)



19- $\{[N,N'$ -Bis(*tert*-butoxycarbonyl)]guanidino $\}$ 3-(methoxymethylenoxy)-nonadecanoic acid (*R*)-8 (DEPT-135, CDCl₃)

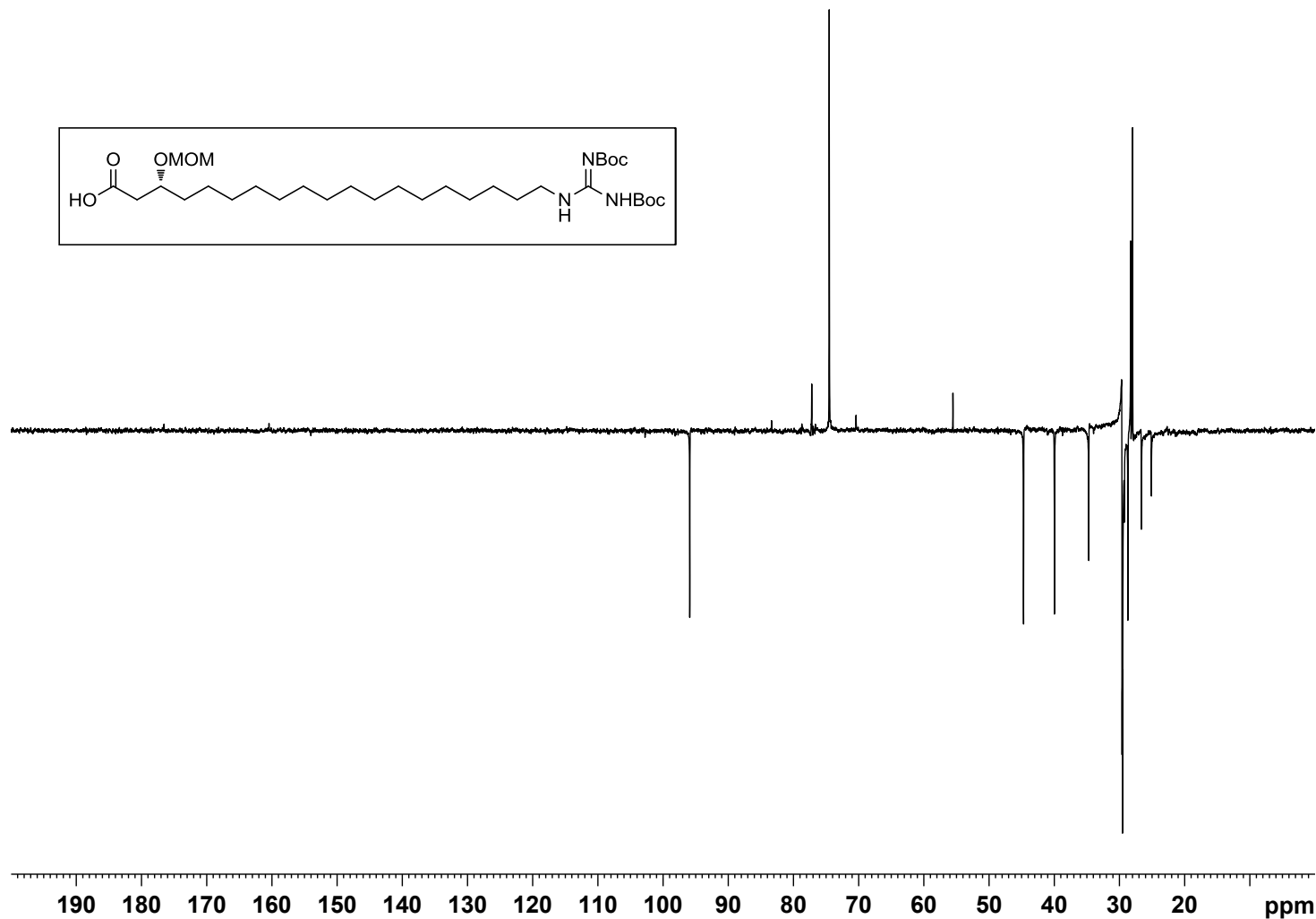
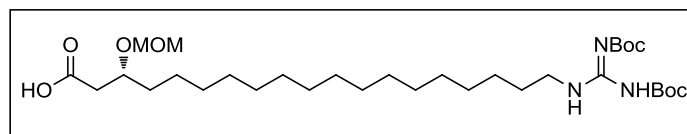
Current Data Parameters
NAME JCexacid(S)
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100519
Time_ 8.54
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG dept135
TD 65536
SOLVENT CDC13
NS 10240
DS 2
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 7298.2
DW 19.100 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001528 sec
MCREST 0 sec
MCWRK 0.01500000 sec

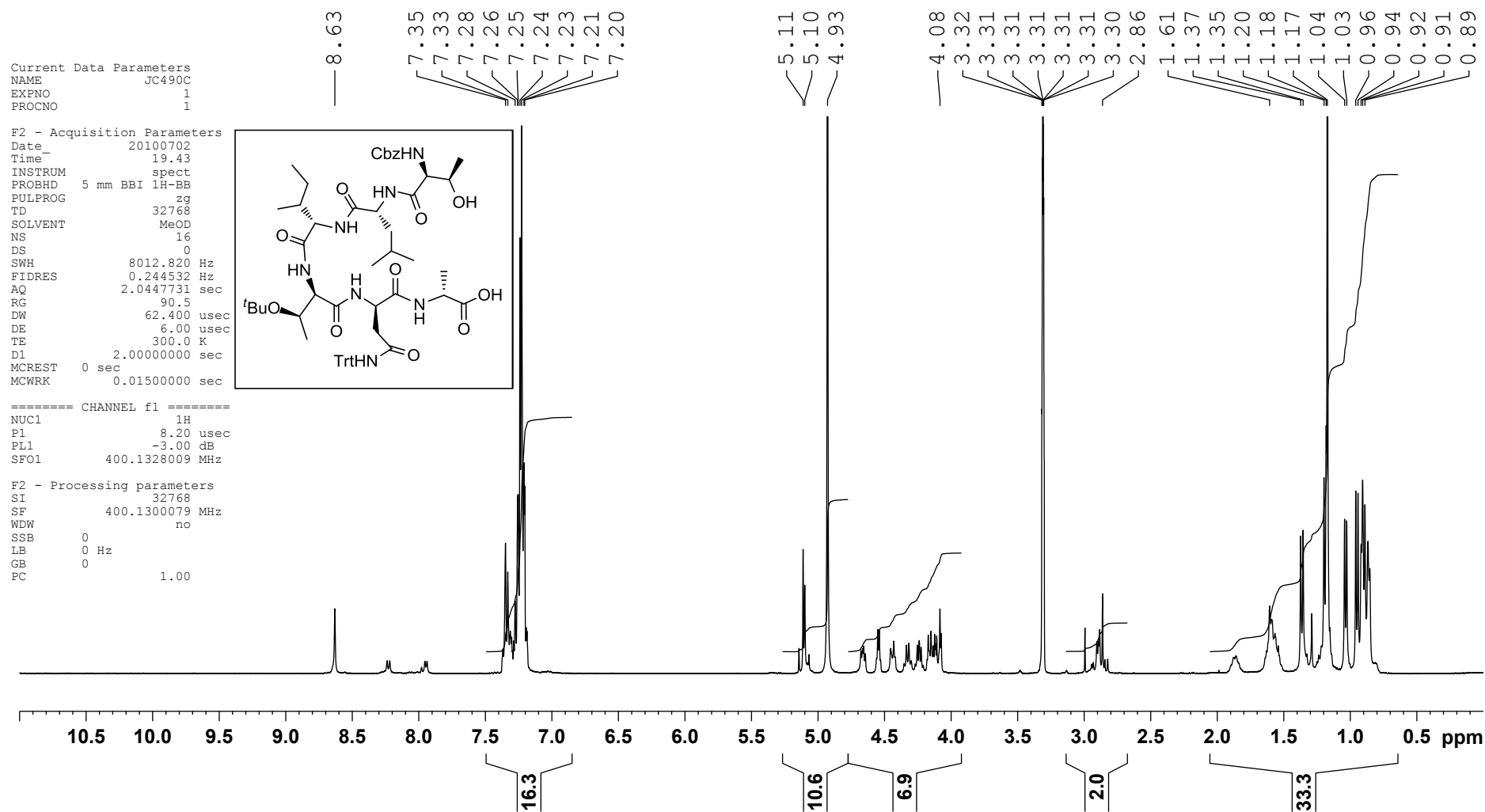
===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
p2 24.00 usec
PL1 -6.00 dB
SFO1 100.6445019 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 5.30 usec
p4 10.60 usec
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 21.60 dB
SFO2 400.2101790 MHz

F2 - Processing parameters
SI 32768
SF 100.6324292 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00



Cbz-L-Thr-D-Leu-L-Ile-Val-D-*allo*-Thr(*tert*-butyl)-D-Asn(Trt)-D-Ala-OH 13 (¹H NMR 400 MHz, MeOD-*d*₄)



Cbz-L-Thr-D-Leu-L-Ile-Val-D-*allo*-Thr(*tert*-butyl)-D-Asn(Trt)-D-Ala-OH 13 (COSY, MeOD-*d*₄)

```

Current Data Parameters
NAME          JC490C
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20100702
Time_         19.44
INSTRUM       spect
PROBHD        5 mm BBI 1H-BB
PULPROG       cosygpgf
TD            2048
SOLVENT       MeOH
NS            16
DS            4
SWH           4006.410 Hz
FIDRES        1.956255 Hz
AQ            0.2556404 sec
RG            80.625
DW            124.800 usec
DE            6.00 usec
TE            300.0 K
d0            0.00000300 sec
D1            2.25000000 sec
d13           0.00000400 sec
D16           0.00015000 sec
IN0           0.00024991 sec
MCREST        0 sec
MCWRK         2.25000000 sec

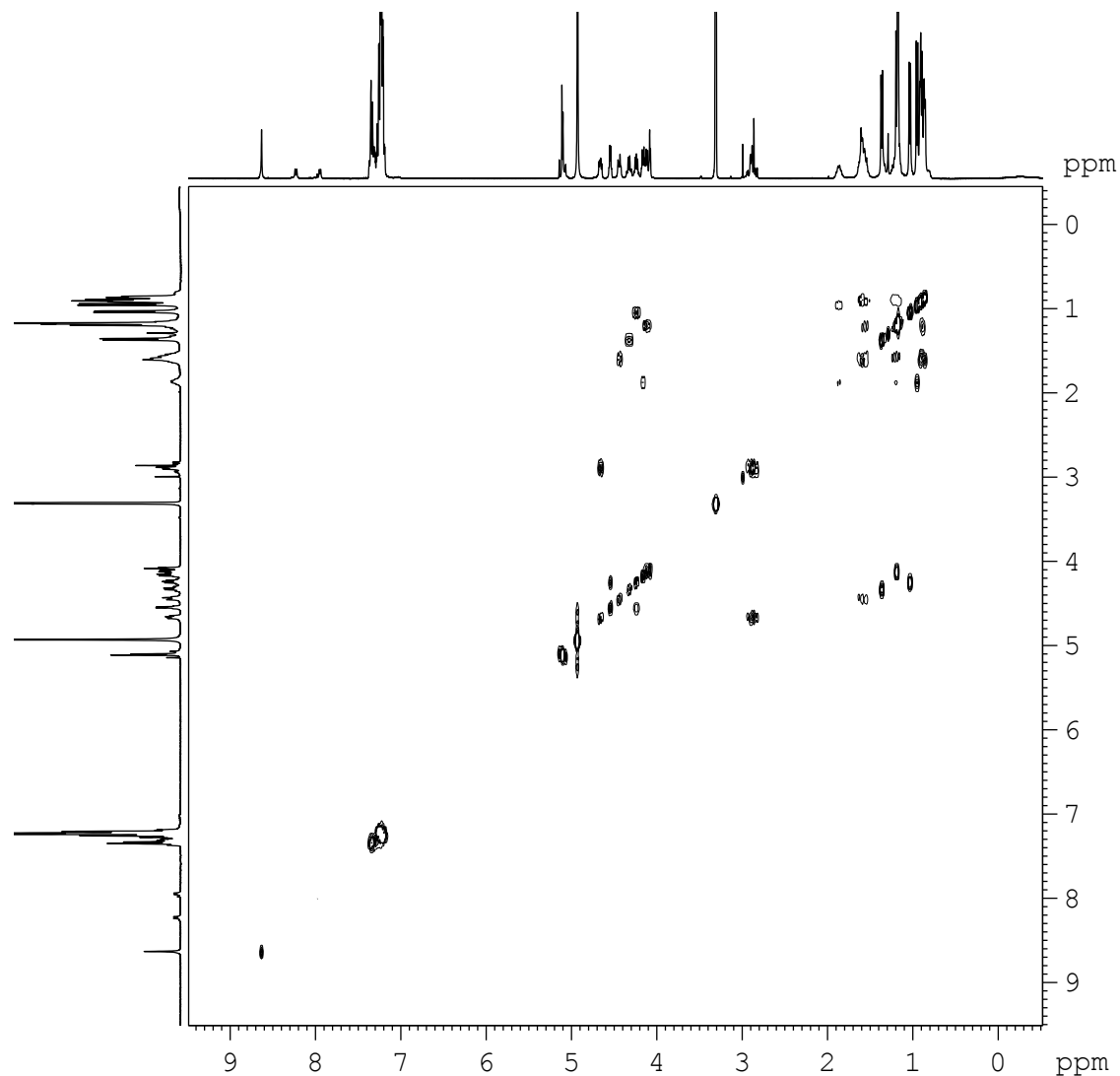
===== CHANNEL f1 =====
NUC1           1H
P0             4.19 usec
P1             8.38 usec
PL1           -3.00 dB
SFO1          400.1318006 MHz

===== GRADIENT CHANNEL =====
GPNAM1        sine.100
GPNAM2        sine.100
GPX1          0 %
GPX2          0 %
GPY1          0 %
GPY2          0 %
GPZ1          10.00 %
GPZ2          10.00 %
P16           1500.00 usec

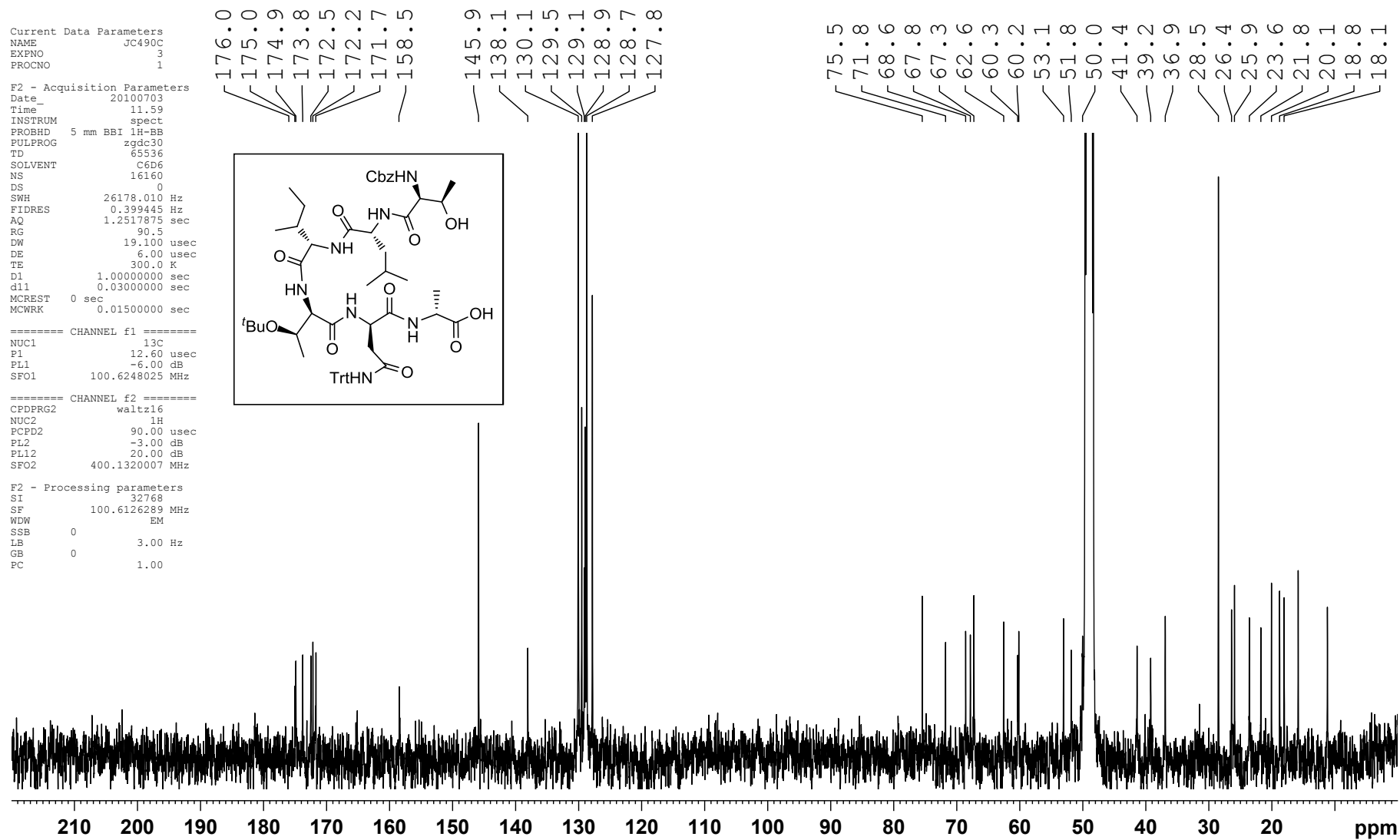
F1 - Acquisition parameters
TD            512
SFO1          400.1318 MHz
FIDRES        7.815235 Hz
SW            10.000 ppm
FnMODE        QF

F2 - Processing parameters
SI            2048
SF            400.1300078 MHz
WDW           QSINE
SSB           1
LB            0 Hz
GB            0
PC            1.00

F1 - Processing parameters
SI            256
MC2           QF
SF            400.1299955 MHz
WDW           States-TPPI
SSB           1
LB            0 Hz
GB            0
    
```



Cbz-L-Thr-D-Leu-L-Ile-Val-D-*allo*-Thr(*tert*-butyl)-D-Asn(Trt)-D-Ala-OH 13 (^{13}C NMR 100 MHz, MeOD- d_4)



Cbz-L-Thr-D-Leu-L-Ile-Val-D-*allo*-Thr(*tert*-butyl)-D-Asn(Trt)-D-Ala-OH 13 (HSQC, MeOD-*d*₄)

Current Data Parameters
NAME JC490C
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100704
Time_ 14.51
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG invietgps1
TD 4096
SOLVENT CDCl3
NS 32
DS 4
SWH 4006.410 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 912.275
DW 124.800 usec
DE 6.00 usec
TE 301.0 K
CNST2 140.0000000
d0 0.0000300 sec
D1 2.00000000 sec
d4 0.00178571 sec
d11 0.03000000 sec
d13 0.0000400 sec
D16 0.00015000 sec
D24 0.00178500 sec
DELTA 0.00172276 sec
DELTA1 0.00165800 sec
INO 0.00002485 sec
MCREST 0 sec
MCWRK 0.33333400 sec
ST1CNT 0

===== CHANNEL f1 =====
NUC1 1H
P1 8.38 usec
P2 16.76 usec
P28 0.50 usec
PL1 -3.00 dB
SFO1 400.1318006 MHz

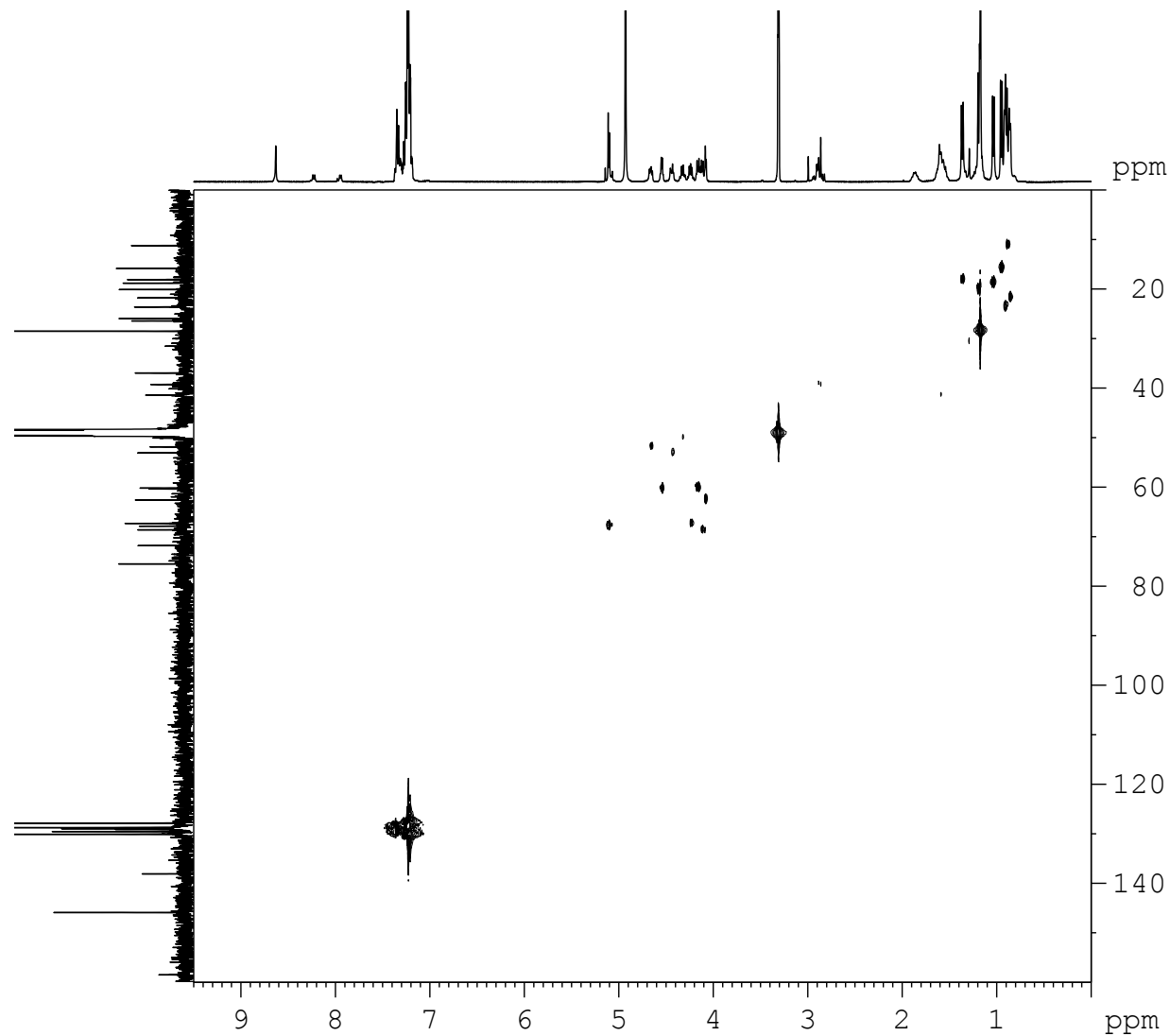
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 12.00 usec
P4 24.00 usec
PCPD2 70.00 usec
PL2 -6.00 dB
PL12 10.70 dB
SFO2 100.6227903 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1500.00 usec

F1 - Acquisition parameters
TD 188
SFO1 100.6228 MHz
FIDRES 107.025131 Hz
SW 199.962 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 400.1300055 MHz
WDW SINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 512
MC2 echo-antiecho
SF 100.6126317 MHz
WDW P
SSB 2
LB 0 Hz
GB 0



Cbz-L-Thr-D-Leu-L-Ile-Val-D-*allo*-Thr(*tert*-butyl)-D-Asn(Trt)-D-Ala-OH 13 (HMBC, MeOD-*d*₄)

```

Current Data Parameters
NAME          JC490C
EXPNO         6
PROCNO        1

F2 - Acquisition Parameters
Date_         20100711
Time          19.09
INSTRUM       spect
PROBHD        5 mm BBI 1H-BB
PULPROG       inv4gplirndgf
TD            4096
SOLVENT       CD3CN
NS            32
DS            8
SWH           4006.410 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG            5792.63
DW            124.800 usec
DE            6.00 usec
TE            0 K
CNST2         145.0000000
d0            0.00000300 sec
d1            2.00000000 sec
d2            0.00344828 sec
d6            0.06250000 sec
d13           0.00000400 sec
d16           0.00015000 sec
IN0           0.00002484 sec
MCREST        0 sec
MCWRK         2.00000000 sec

===== CHANNEL f1 =====
NUC1           1H
P1            6.12 usec
P2           12.24 usec
PL1           -3.00 dB
SFO1          400.2101790 MHz

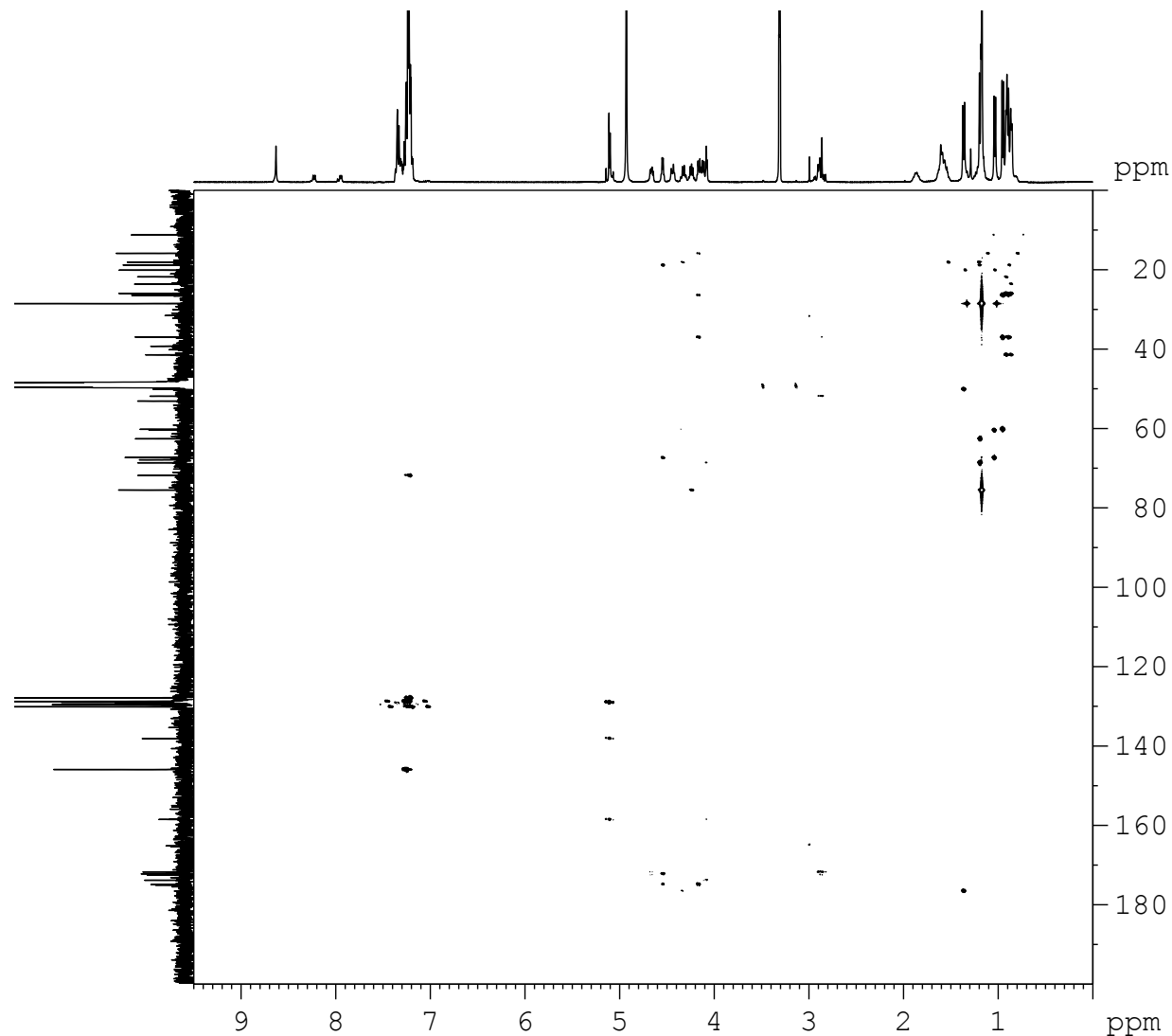
===== CHANNEL f2 =====
NUC2           13C
P3           12.00 usec
PL2           -6.00 dB
SFO2          100.6424892 MHz

===== GRADIENT CHANNEL =====
GPNAM1         sine.100
GPNAM2         sine.100
GPNAM3         sine.100
GPX1           0 %
GPX2           0 %
GPX3           0 %
GPY1           0 %
GPY2           0 %
GPY3           0 %
GPZ1           50.00 %
GPZ2           30.00 %
GPZ3           40.10 %
P16            1500.00 usec

F1 - Acquisition parameters
TD            512
SFO1          100.6425 MHz
FIDRES        39.318066 Hz
SW            200.023 ppm
FnMODE        QF

F2 - Processing parameters
SI            2048
SF            400.2081779 MHz
WDW           SINE
SSB           2
LB            0 Hz
GB            0
PC            1.00

F1 - Processing parameters
SI            1024
MC2           QF
SF            100.6322781 MHz
WDW           States
SSB           2
LB            0 Hz
GB            0
    
```



Cyclic peptide 7 (¹H NMR 400 MHz, DMSO-d₆)

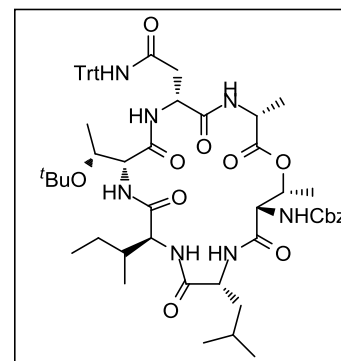
Current Data Parameters
NAME JC498P
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100704
Time 18.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 287.4
DW 62.400 usec
DE 6.00 usec
TE 0 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.40 usec
PL1 -3.00 dB
SFO1 400.2105792 MHz

F2 - Processing parameters
SI 32768
SF 400.2081750 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

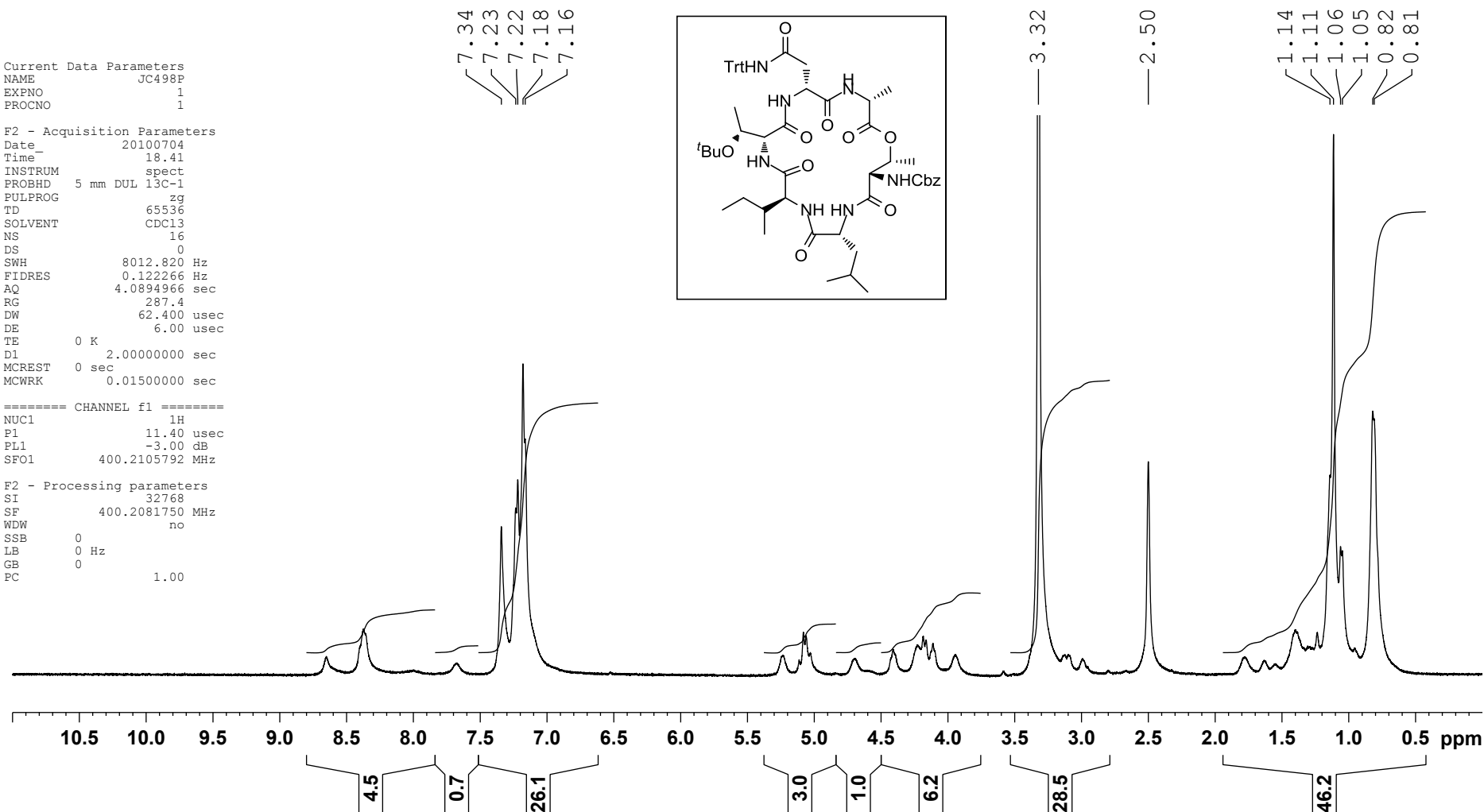
7.34
7.23
7.22
7.18
7.16



3.32

2.50

1.14
1.11
1.06
1.05
0.82
0.81



```

Current Data Parameters
NAME          JC498P
EXPO          4
PROCNO        1

F2 - Acquisition Parameters
Date_         20100714
Time_         13.30
INSTRUM       spect
PROBHD        5 mm BBI 1H-BB
PULPROG       cosygpgf
TD            2048
SOLVENT       CDC13
NS            16
DS            16
SWH           4006.410 Hz
ZFDRES        1.956255 Hz
AQ            0.2556404 sec
RG            143.675
DW            124.800 usec
DE            6.00 usec
TE            0 K
d0            0.00000300 sec
D1            2.00000000 sec
d13           0.00000400 sec
D16           0.00015000 sec
INO           0.00024988 sec
MCREST        0 sec
MCWRK         2.00000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            2.91 usec
PL1           5.82 usec
PL1           -3.00 dB
SFO1          400.2101790 MHz

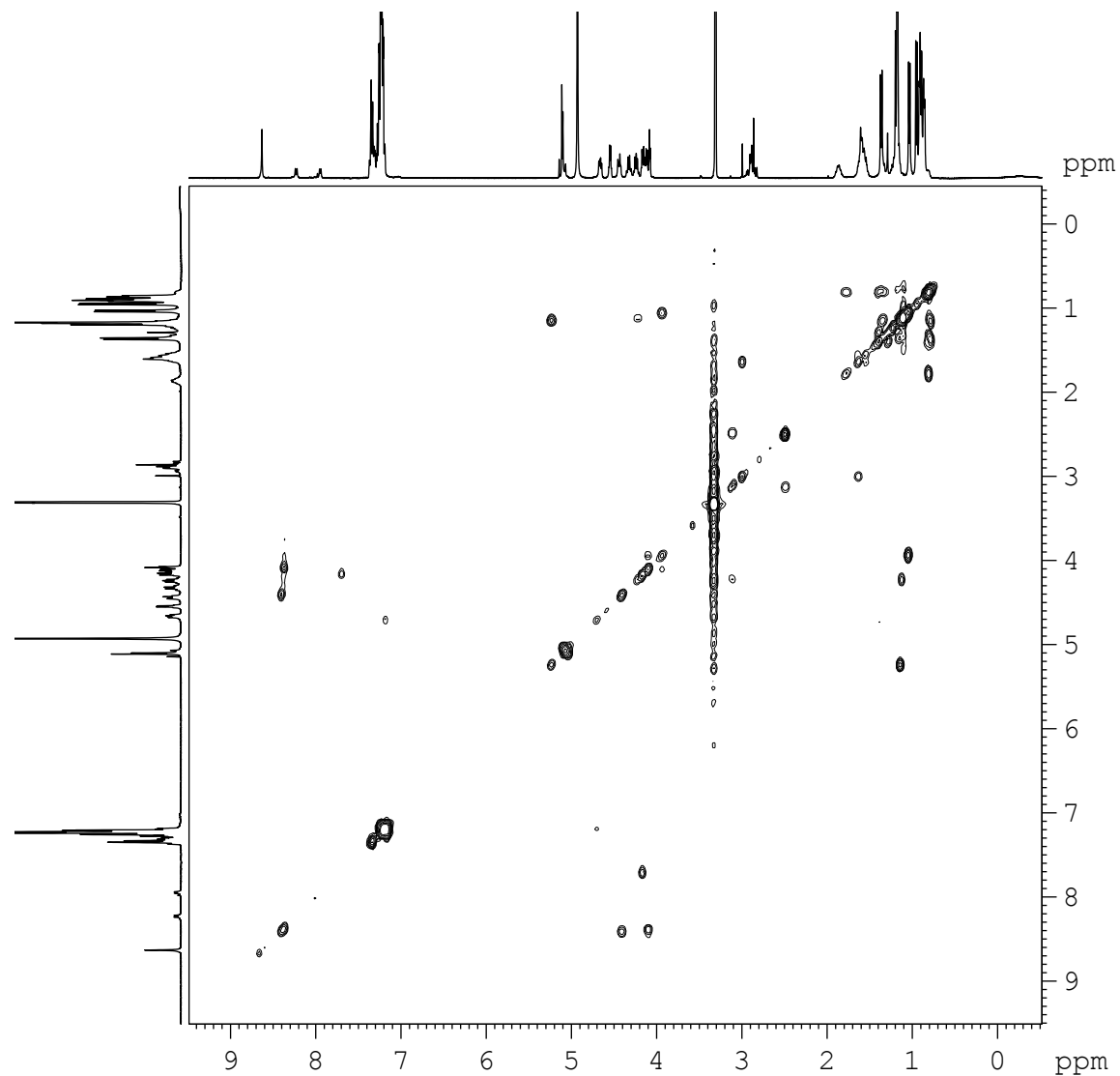
===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPX1          0 %
GPX2          0 %
GPY1          0 %
GPY2          0 %
GPZ1          10.00 %
GPZ2          10.00 %
P16           1500.00 usec

F1 - Acquisition parameters
TD            512
SFO1          400.2102 MHz
FIDRES        7.816408 Hz
SW            10.000 ppm
FnMODE        QF

F2 - Processing parameters
SI            512
SF            400.2081737 MHz
WDW           QSINE
SSB           0
LB            0 Hz
GB            0
PC            1.00

F1 - Processing parameters
SI            256
MC2           QF
P1            400.2081672 MHz
WDW           States-TPPI
SSB           0
LB            0 Hz
GB            0

```



Cyclic peptide 7 (¹³C NMR 100 MHz, DMSO-*d*₆)

```

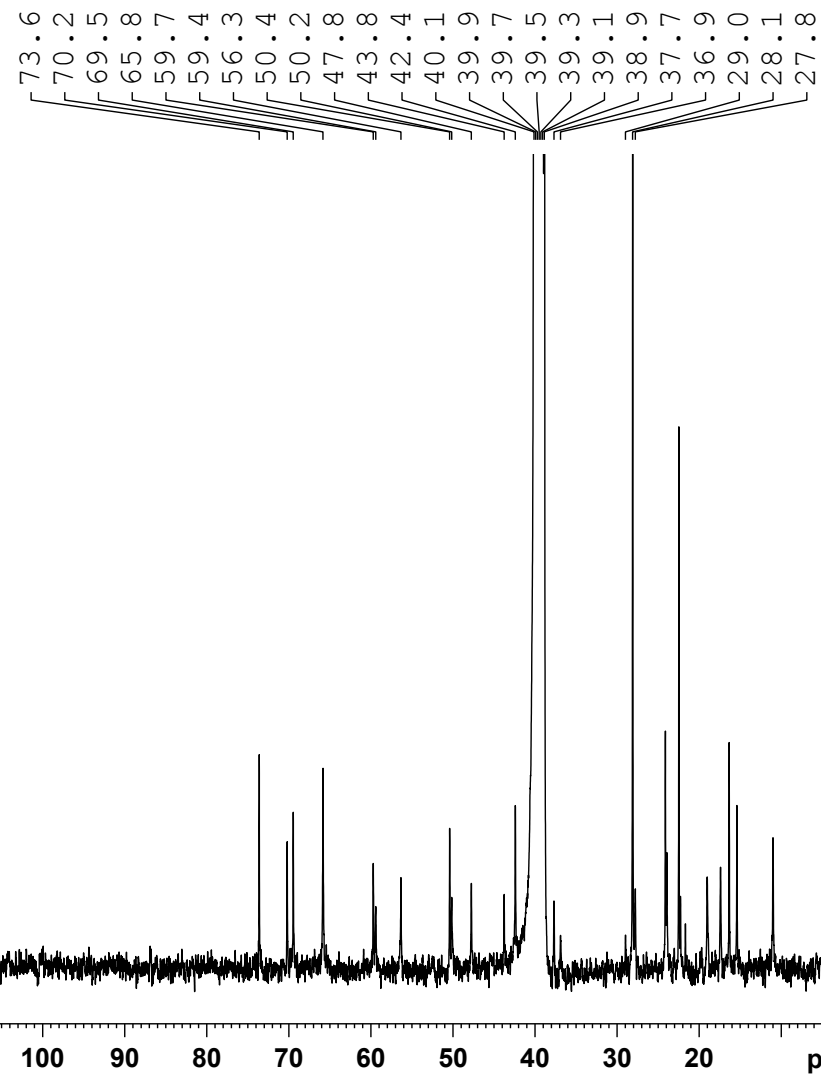
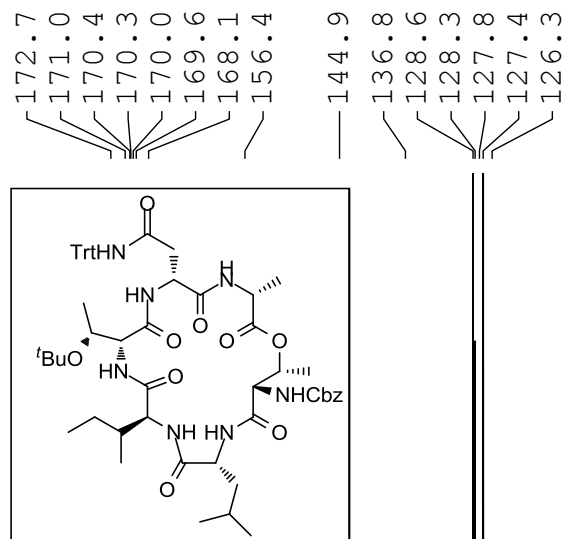
Current Data Parameters
NAME          JC498P
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20100704
Time          19.00
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            30786
DS            2
SWH           26178.010 Hz
FIDRES        0.399445 Hz
AQ            1.2517875 sec
RG            57
DW            19.100 usec
DE            6.00 usec
TE            0 K
D1            1.00000000 sec
d11           0.03000000 sec
DELTA         0.89999998 sec
MCREST        0 sec
MCWRK         0.01500000 sec

===== CHANNEL f1 =====
NUC1           13C
P1             5.40 usec
PL1            -6.00 dB
SFO1          100.6445019 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2            -3.00 dB
PL12          13.92 dB
PL13          20.00 dB
SFO2          400.2101790 MHz

F2 - Processing parameters
SI            65536
SF            100.6324759 MHz
WDW           EM
SSB           0
LB            3.00 Hz
GB            0
PC            1.00
  
```



Cyclic peptide 7 (HSQC, DMSO-*d*₆)

Current Data Parameters
NAME JC498P
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100714
Time 18.49
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG invietgpsi
TD 4096
SOLVENT DMSO
NS 32
DS 8
SWH 4006.410 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 1149.4
DW 124.800 usec
DE 6.00 usec

TE 0 K
CNST2 145.0000000
d0 0.0000300 sec
D1 2.0000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.0000400 sec
D16 0.00015000 sec
D24 0.00172000 sec
DELTA 0.00171764 sec
DELTA1 0.00165800 sec
INO 0.00002484 sec
MCREST 0 sec
MCWRK 0.33333400 sec
ST1CNT 0

===== CHANNEL f1 =====
NUC1 1H
P1 5.82 usec
P2 11.64 usec
P28 2000.00 usec
PL1 -3.00 dB
SFO1 400.2101790 MHz

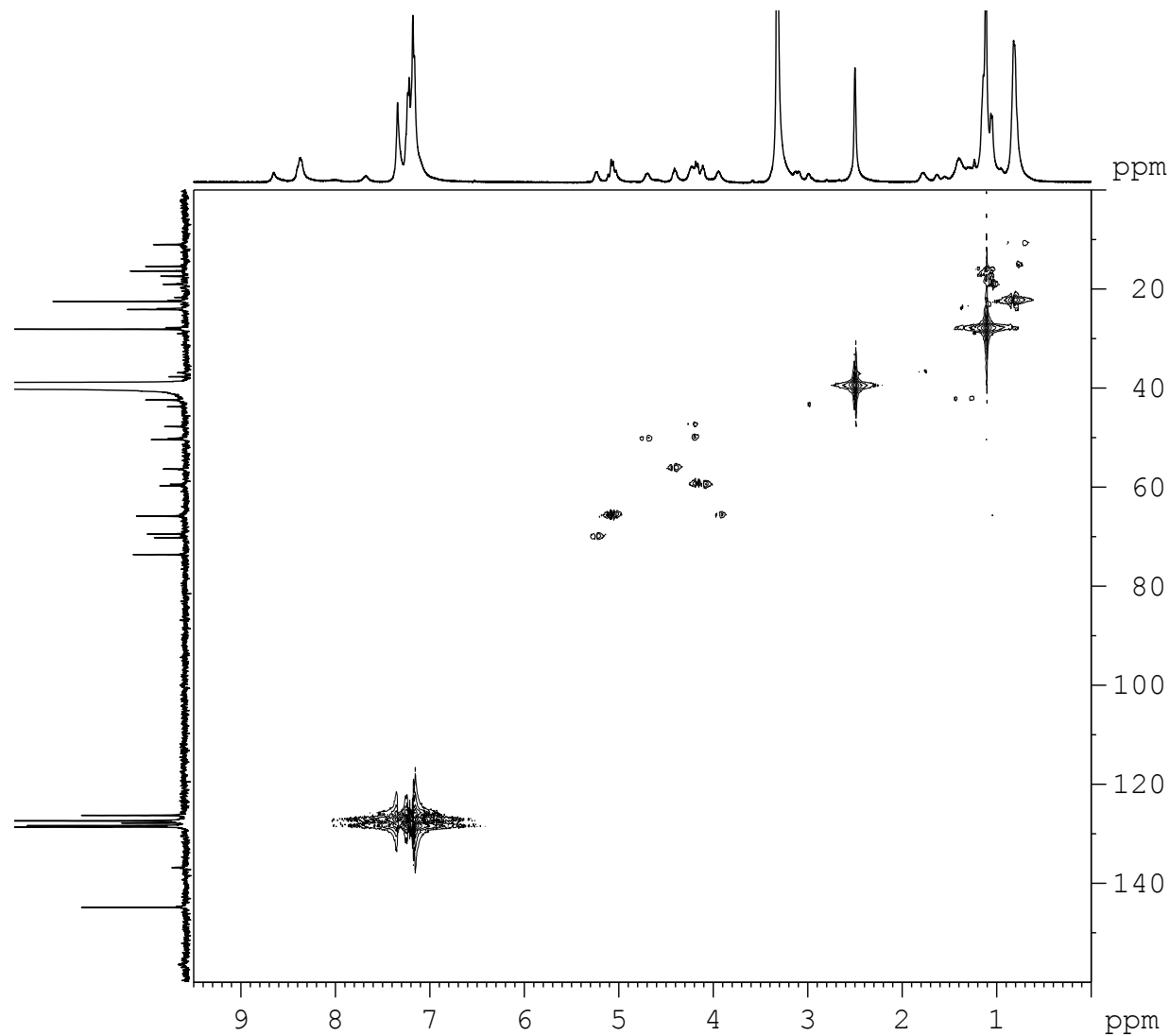
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 12.00 usec
P4 24.00 usec
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 10.48 dB
SFO2 100.6424892 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1500.00 usec

F1 - Acquisition parameters
TD 256
SFO1 100.6425 MHz
FIDRES 78.636131 Hz
SW 200.023 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 400.2081757 MHz
WDW SINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 512
MC2 echo-antiecho
SF 100.6324842 MHz
WDW
SSB 2
LB 0 Hz
GB 0



Cyclic peptide 7 (HMBC, DMSO-*d*₆)

Current Data Parameters
NAME JC498P
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100715
Time_ 0.36
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG inv4gplirndgf
TD 4096
SOLVENT CD3CN
NS 32
DS 8
SWH 4006.410 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 724.075
DW 124.800 usec
DE 6.00 usec
TE 0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 2.00000000 sec
d2 0.00344828 sec
D6 0.06250000 sec
d13 0.00000400 sec
D16 0.00015000 sec
IN0 0.00002484 sec
MCREST 0 sec
MCWRK 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 5.82 usec
P2 11.64 usec
PL1 -3.00 dB
SFO1 400.2101790 MHz

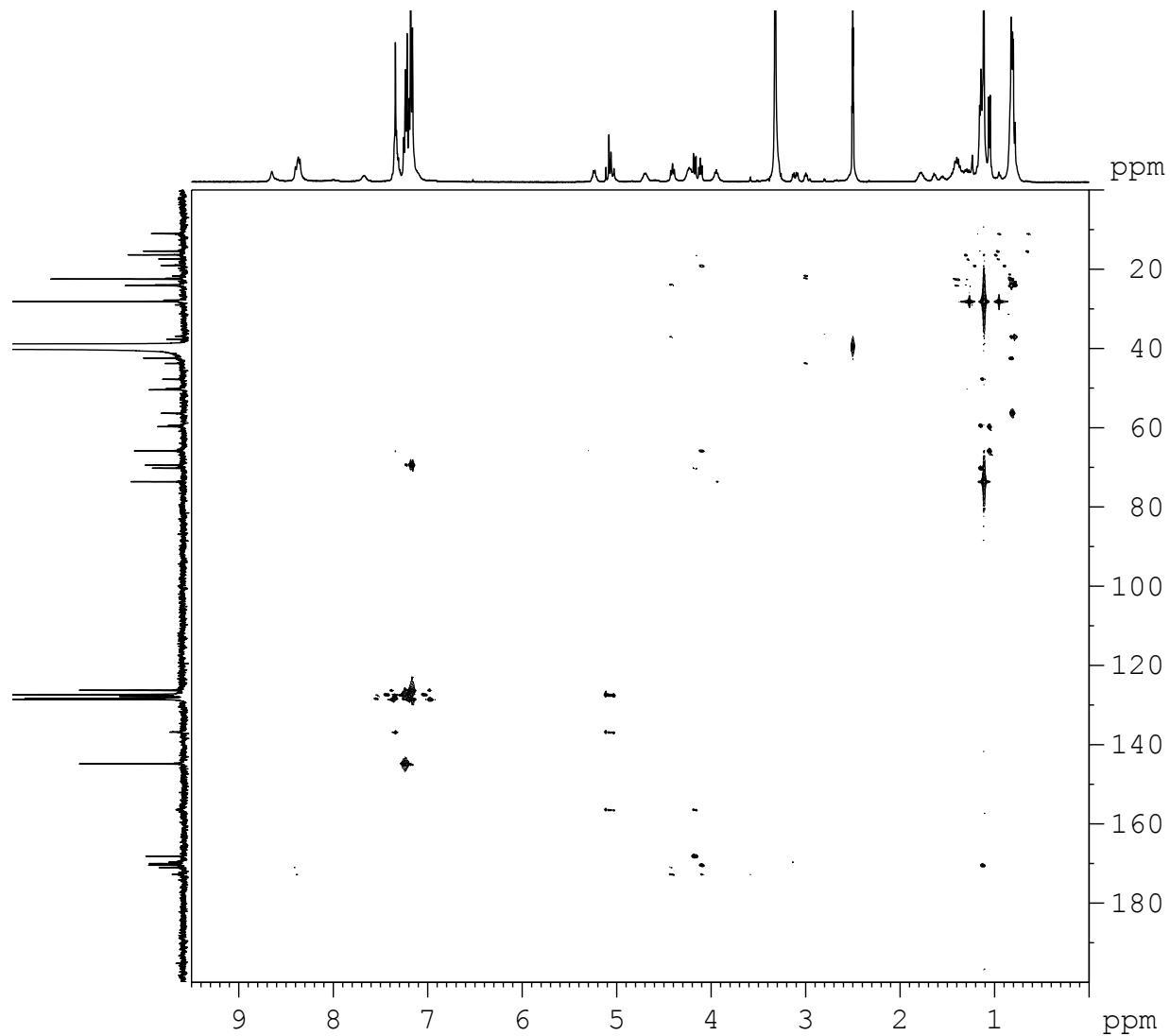
===== CHANNEL f2 =====
NUC2 13C
P3 12.00 usec
PL2 -6.00 dB
SFO2 100.6424892 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPX1 0 %
GPX2 0 %
GPX3 0 %
GPY1 0 %
GPY2 0 %
GPY3 0 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1500.00 usec

F1 - Acquisition parameters
TD 462
SFO1 100.6425 MHz
FIDRES 43.573269 Hz
SW 200.023 ppm
FMODE QF

F2 - Processing parameters
SI 2048
SF 400.2081747 MHz
WDW SINE
SSB 2
LB 0 Hz
GB 0
FC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6324617 MHz
WDW States
SSB 2
LB 0 Hz
GB 0



Current Data Parameters

NAME	circulocindpure
EXPNO	1
FPROCNO	1

F2 - Acquisition Parameters

Date_	20100908
Time	11.42
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zg
TD	32768
SOLVENT	CDCl ₃
NS	64
DS	0
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	2.0447731 sec
RG	143.7
DW	62.400 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
MCREST	0 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	8.20 usec
PL1	-3.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters

SI	32768
SF	400.1300035 MHz
WDW	no
SSB	0
LB	0 Hz
GB	0
PC	1.00

Chemical Structure:

C[C@H](O)[C@@H](NC(=O)CC(C)[C@H](N)C(=O)OC(C)(C)C)[C@@H](C)C(=O)NCCC(N)=N

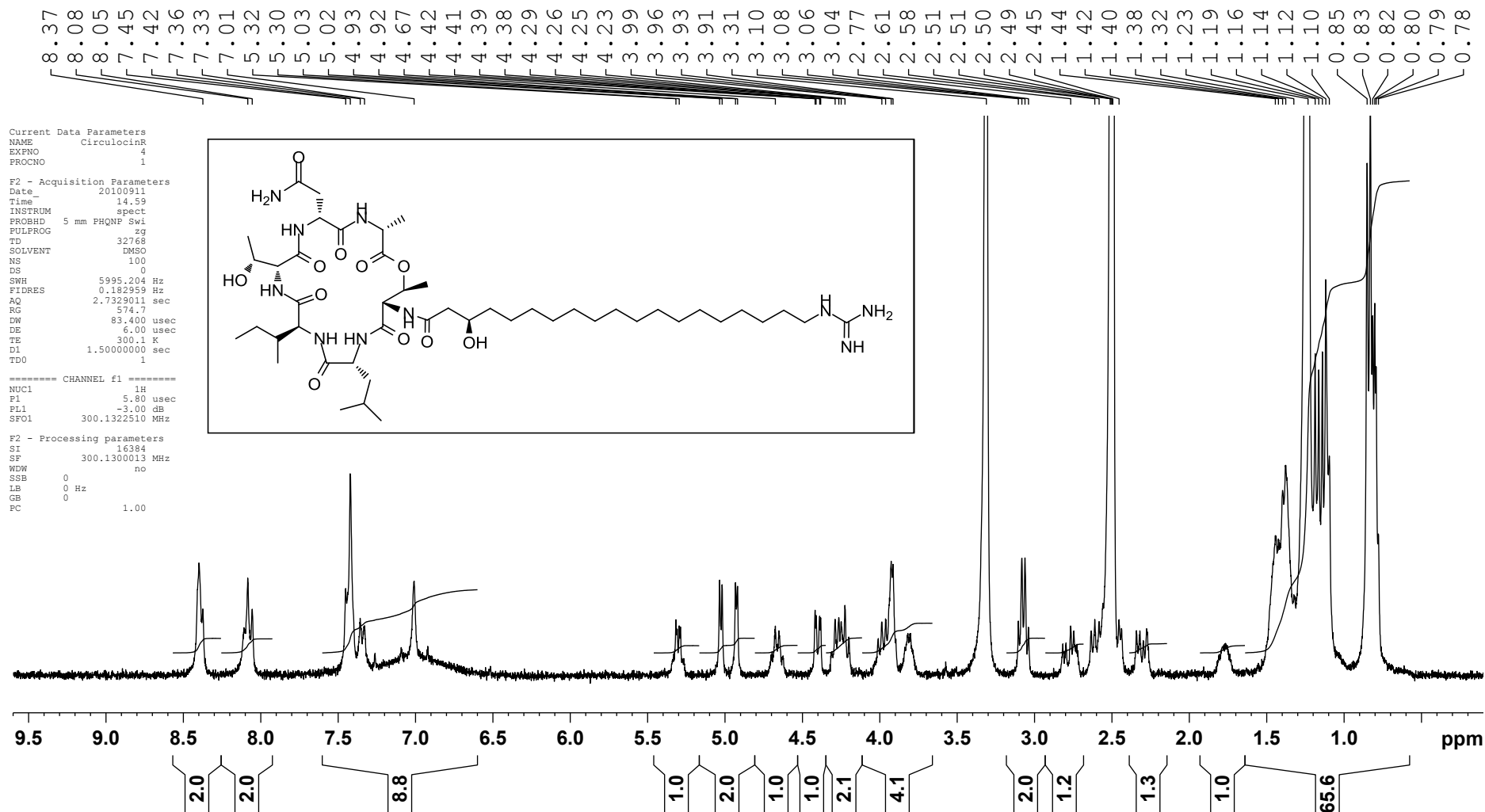
Peak List (ppm):

Chemical Shift (ppm)
8.46
8.45
8.42
8.13
8.11
7.49
7.47
7.45
7.44
7.04
4.40
4.40
4.38
4.38
4.25
4.23
3.91
3.89
3.39
3.07
3.05
2.54
2.50
2.50
1.45
1.44
1.42
1.41
1.37
1.36
1.22
1.16
1.15
1.13
1.12
1.10
1.09
0.84

Integration Values:

Integration Value
2.1
2.0
9.8
1.0
1.9
1.0
7.4
2.3
21.4
1.1
64.6

Compound (R)-4 (¹H NMR 300 MHz, DMSO-d₆)



Compound (R)-4 (COSY, DMSO-*d*₆)

Current Data Parameters
NAME circulocinR
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100806
Time_ 17.14
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG cosygpgf
TD 2048
SOLVENT MeOH
NS 32
DS 4
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 456.15
DW 124.800 usec
DE 6.00 usec
TE 300.0 K
d0 0.00000300 sec
D1 1.50000000 sec
d13 0.00000400 sec
D16 0.00015000 sec
IN0 0.00024991 sec
MCREST 0 sec
MCWRK 1.50000000 sec

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

NUC1 1H
P0 3.63 usec
P1 7.25 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

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

GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1500.00 usec

F1 - Acquisition parameters

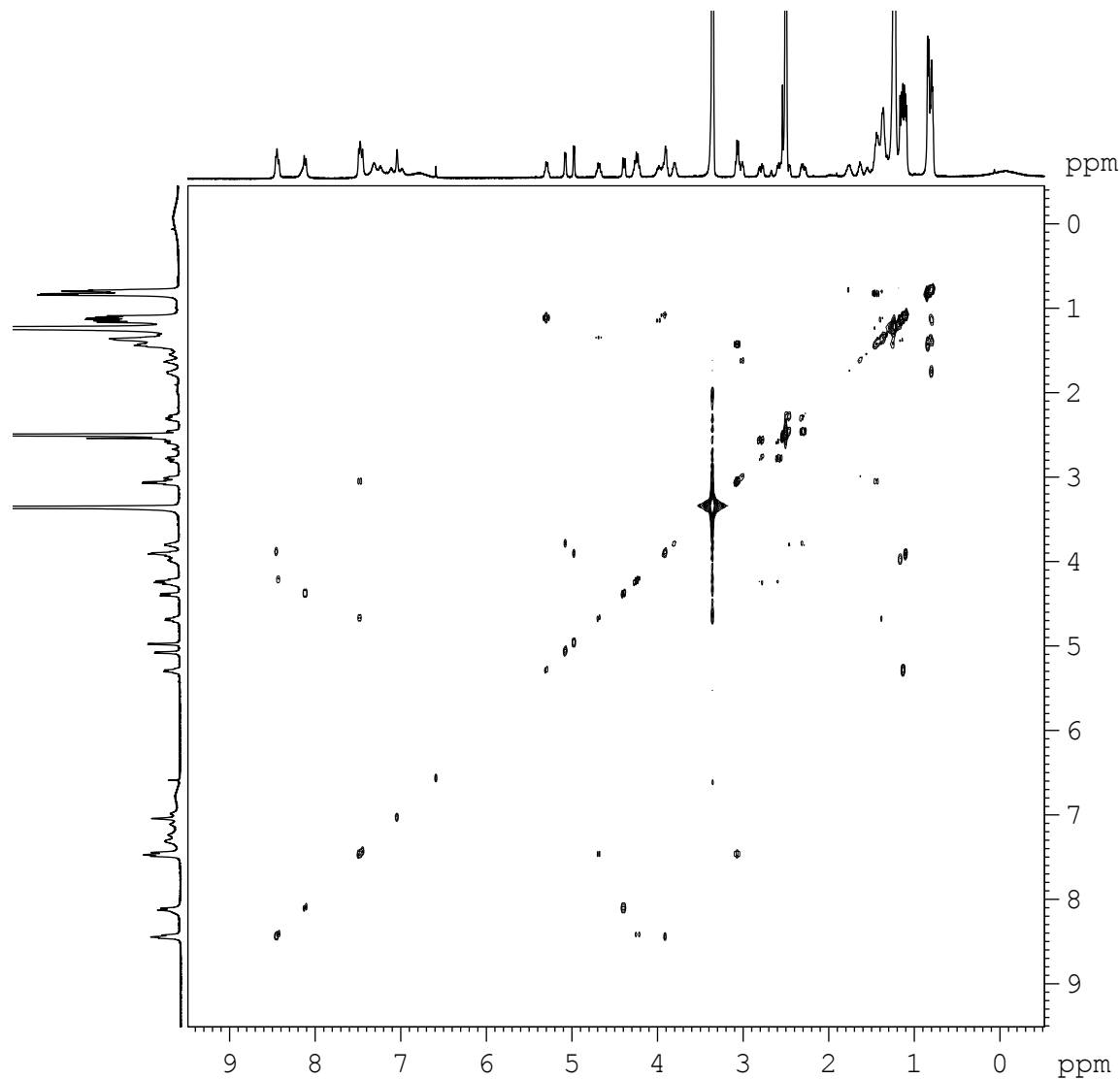
TD 512
SFO1 400.132 MHz
FIDRES 7.815235 Hz
SW 10.000 ppm
FnMODE QF

F2 - Processing parameters

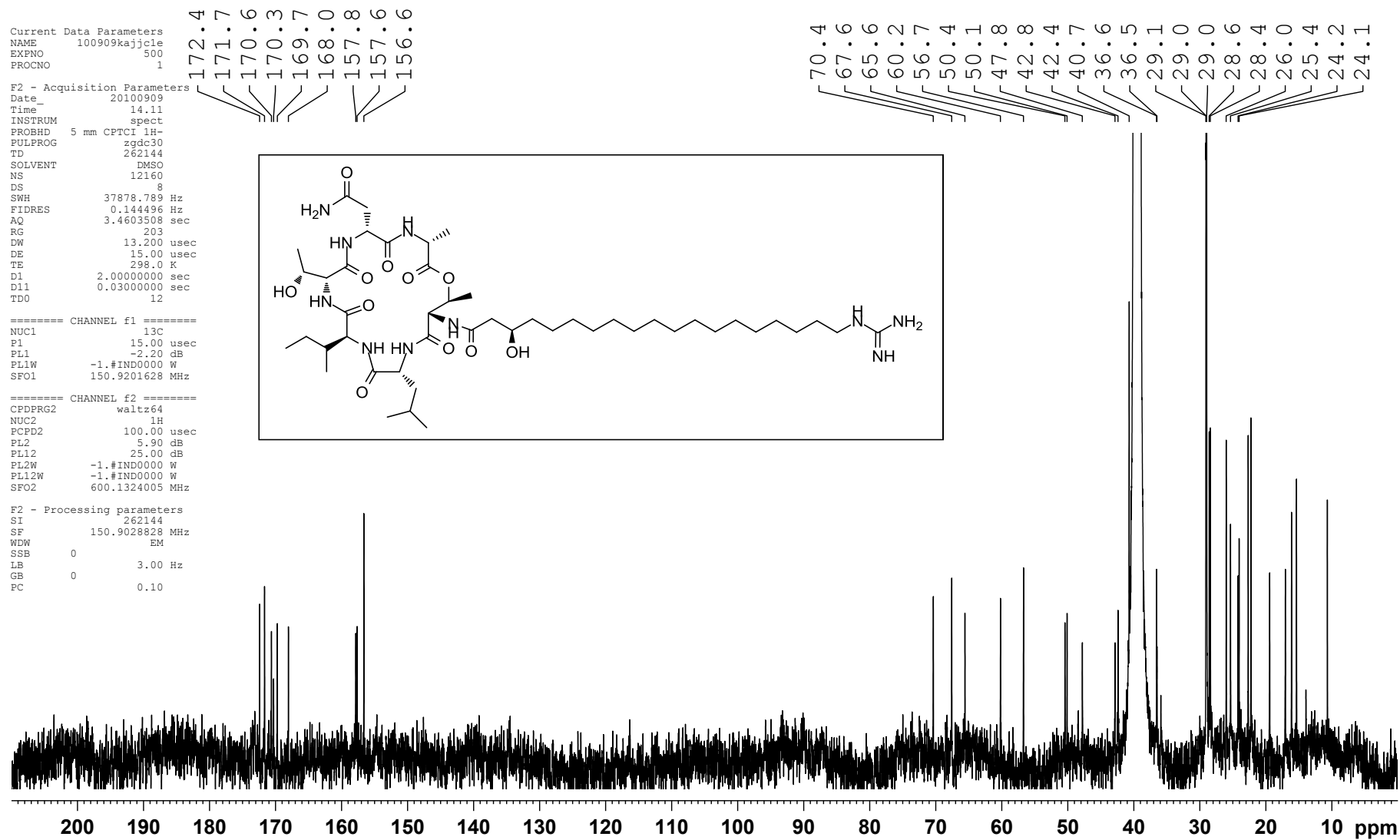
SI 2048
SF 400.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters

SI 256
MC2 QF
SF 400.1300000 MHz
WDW States
SSB 0
LB 0 Hz
GB 0



Compound (R)-4 (^{13}C NMR 150 MHz, DMSO- d_6)



Compound (R)-4 (HSQC, DMSO-*d*₆)

Current Data Parameters
NAME circuloctnR
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100807
Time_ 1.34
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG invietgpsi
TD 2048
SOLVENT DMSO
NS 64
DS 8
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 912.275
DW 124.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 140.000000
d0 0.0000300 sec
D1 1.5000000 sec
d4 0.00178571 sec
d11 0.03000000 sec
d13 0.0000400 sec
D16 0.00015000 sec
D24 0.00178500 sec
DELTA 0.00172050 sec
DELTA1 0.00165800 sec
INO 0.00004969 sec
MCREST 0 sec
MCWRK 0.25000051 sec
ST1CNT 0

===== CHANNEL f1 =====
NUC1 1H
P1 7.25 usec
P2 14.50 usec
P28 0.50 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

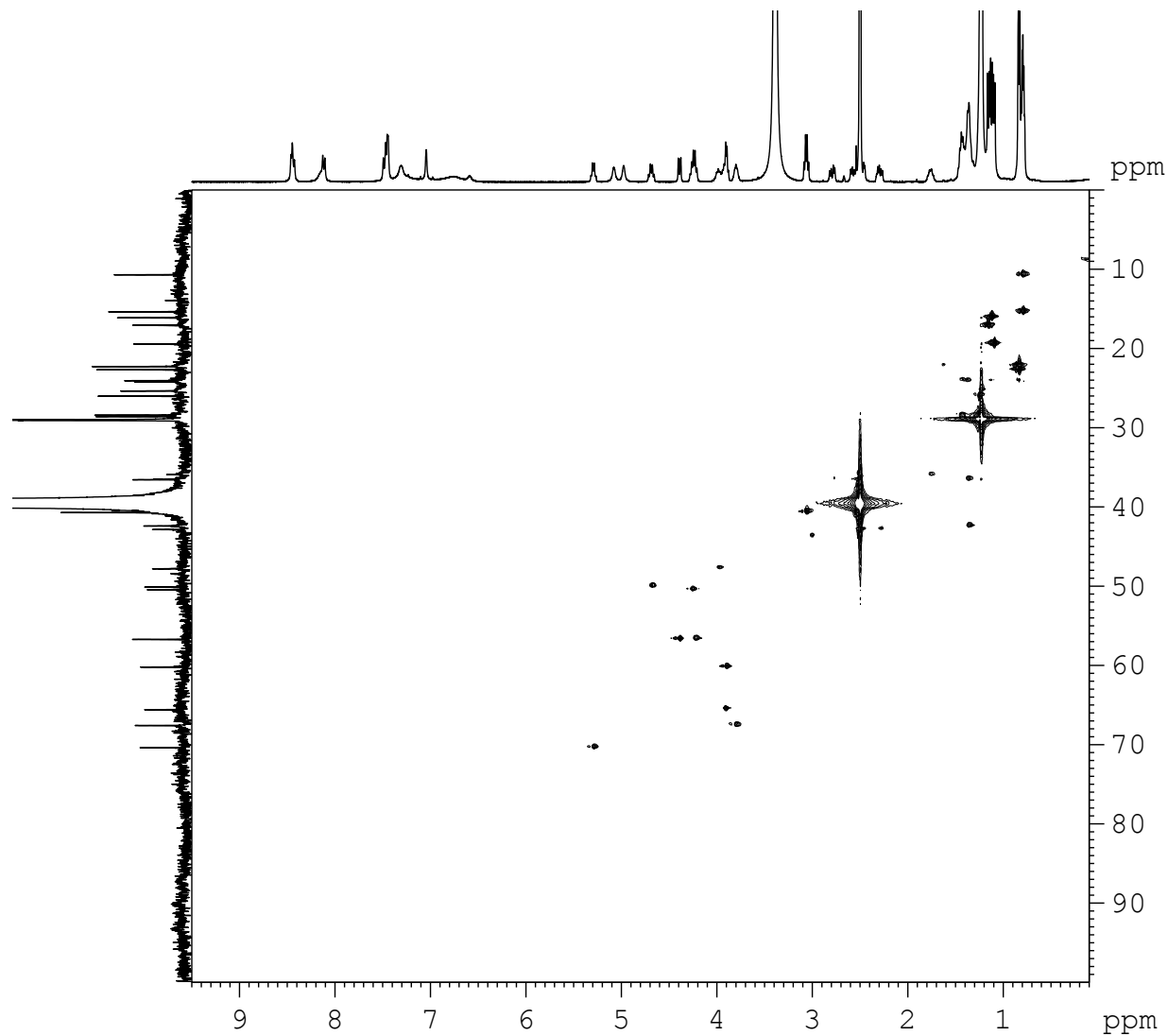
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 12.00 usec
P4 24.00 usec
PCPD2 70.00 usec
PL2 -6.00 dB
PL12 10.70 dB
SFO2 100.6177596 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1500.00 usec

F1 - Acquisition parameters
TD 512
SFO1 100.6178 MHz
FIDRES 19.654089 Hz
SW 100.013 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 400.1299986 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 512
MC2 echo-antiecho
SF 100.6128254 MHz
WDW
SSB 2
LB 0 Hz
GB 0



Compound (R)-4 (HMBC, DMSO-*d*₆)

Current Data Parameters
NAME circuloctnR
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100807
Time_ 1.34
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG invietgpsi
TD 2048
SOLVENT DMSO
NS 64
DS 8
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 912.275
DW 124.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 140.0000000
d0 0.0000300 sec
D1 1.50000000 sec
d4 0.00178571 sec
d11 0.03000000 sec
d13 0.0000400 sec
D16 0.00015000 sec
D24 0.00178500 sec
DELTA 0.00172050 sec
DELTA1 0.00165800 sec
INO 0.00004969 sec
MCREST 0 sec
MCWRK 0.25000051 sec
ST1CNT 0

===== CHANNEL f1 =====
NUC1 1H
P1 7.25 usec
P2 14.50 usec
P28 0.50 usec
PL1 -3.00 dB
SFO1 400.1320007 MHz

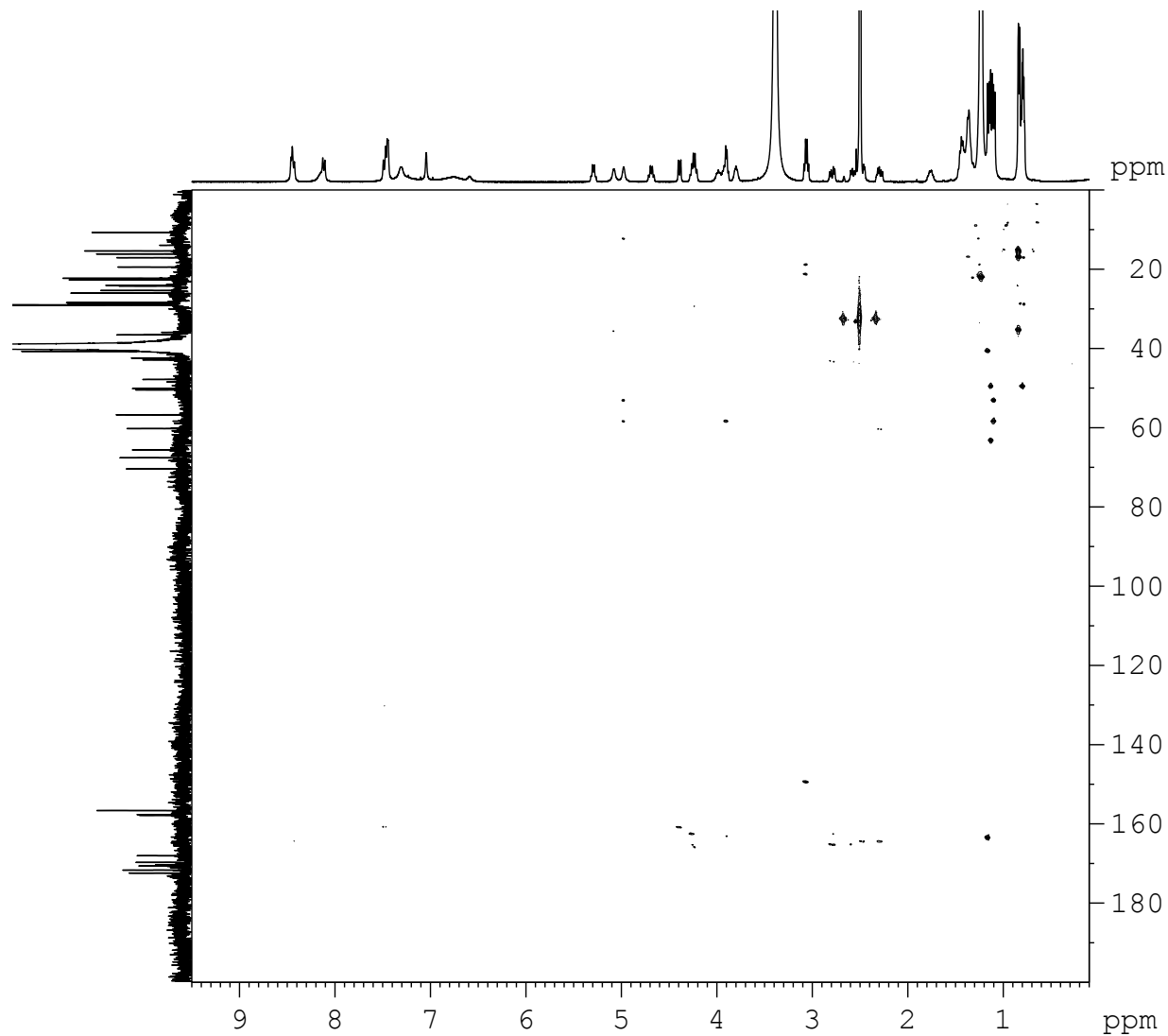
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 12.00 usec
P4 24.00 usec
PCPD2 70.00 usec
PL2 -6.00 dB
PL12 10.70 dB
SFO2 100.6177596 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 80.00 %
GPZ2 20.10 %
P16 1500.00 usec

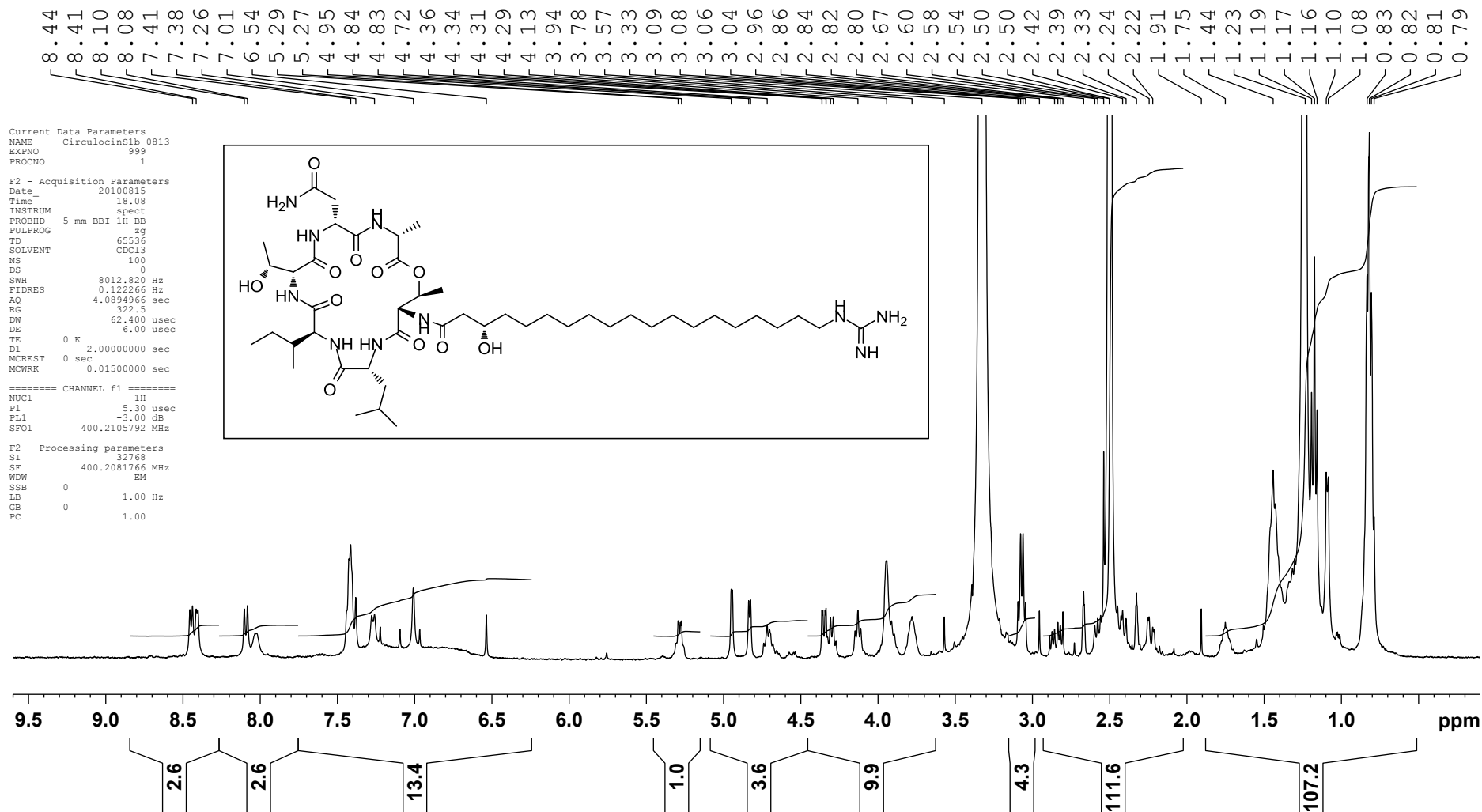
F1 - Acquisition parameters
TD 512
SFO1 100.6178 MHz
FIDRES 19.654089 Hz
SW 100.013 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 400.1299986 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

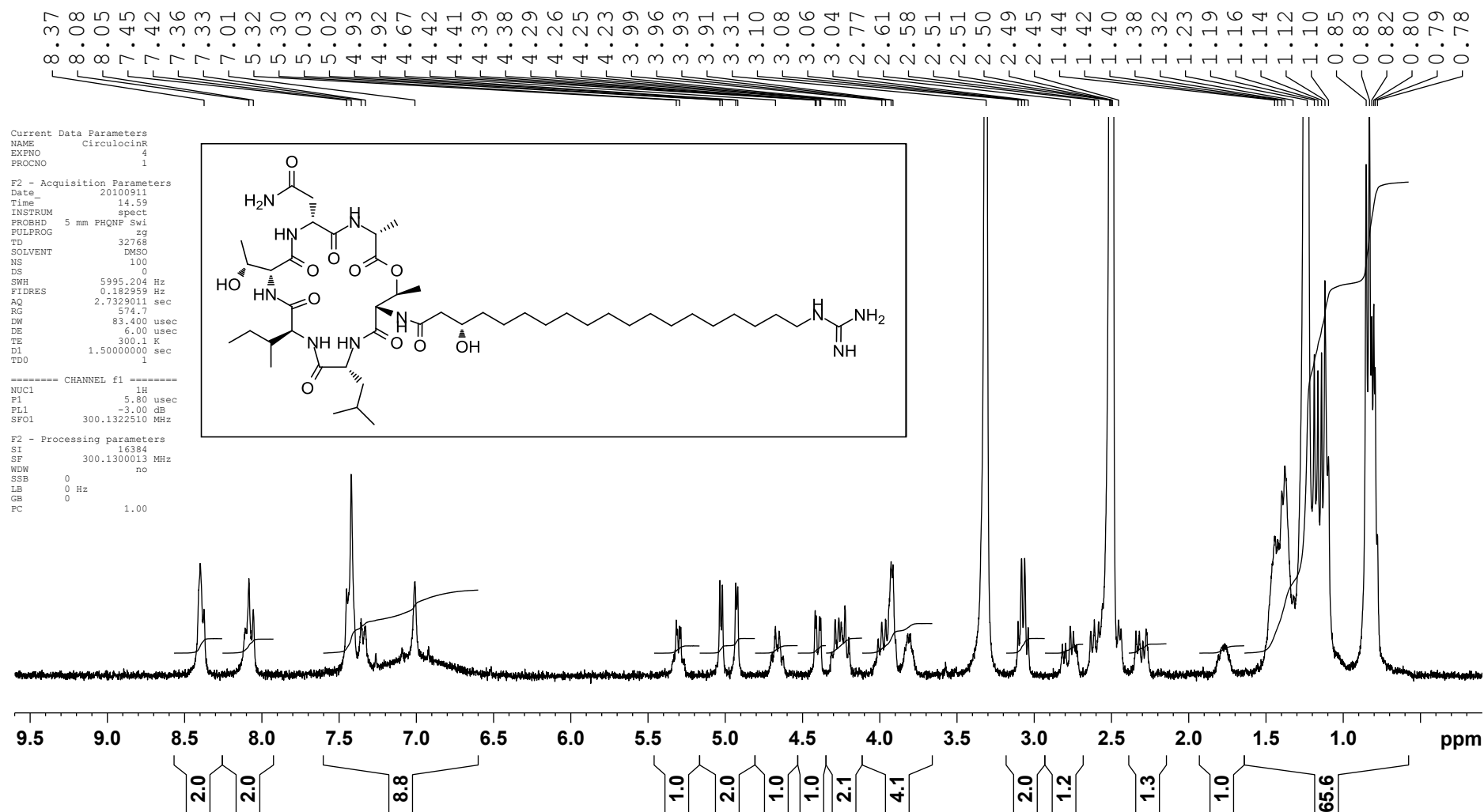
F1 - Processing parameters
SI 512
MC2 echo-antiecho
SF 100.6128254 MHz
WDW
SSB 2
LB 0 Hz
GB 0



Compound (S)-4 (¹H NMR 400 MHz, DMSO-*d*₆)



Compound (S)-4 (¹H NMR 300 MHz, DMSO-*d*₆)



Compound (S)-4 (COSY, DMSO-*d*₆)

Current Data Parameters
NAME CirculocinS1b
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100809
Time_ 17.13
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG cosygpgf
TD 2048
SOLVENT DMSO
NS 128
DS 8
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 203.175
DW 124.800 usec
DE 6.00 usec
TE 0 K
d0 0.00000300 sec
D1 0.80000001 sec
d13 0.00000400 sec
D16 0.00015000 sec
IN0 0.00024988 sec
MCREST 0 sec
MCWRK 0.80000001 sec

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

NUC1 1H
P0 5.80 usec
P1 5.30 usec
PL1 -3.00 dB
SFO1 400.2101790 MHz

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

GPNAME1 SINE.100
GPNAME2 SINE.100
GPX1 0 %
GPX2 0 %
GPY1 0 %
GPY2 0 %
GPZ1 10.00 %
GPZ2 10.00 %
PL6 1500.00 usec

F1 - Acquisition parameters

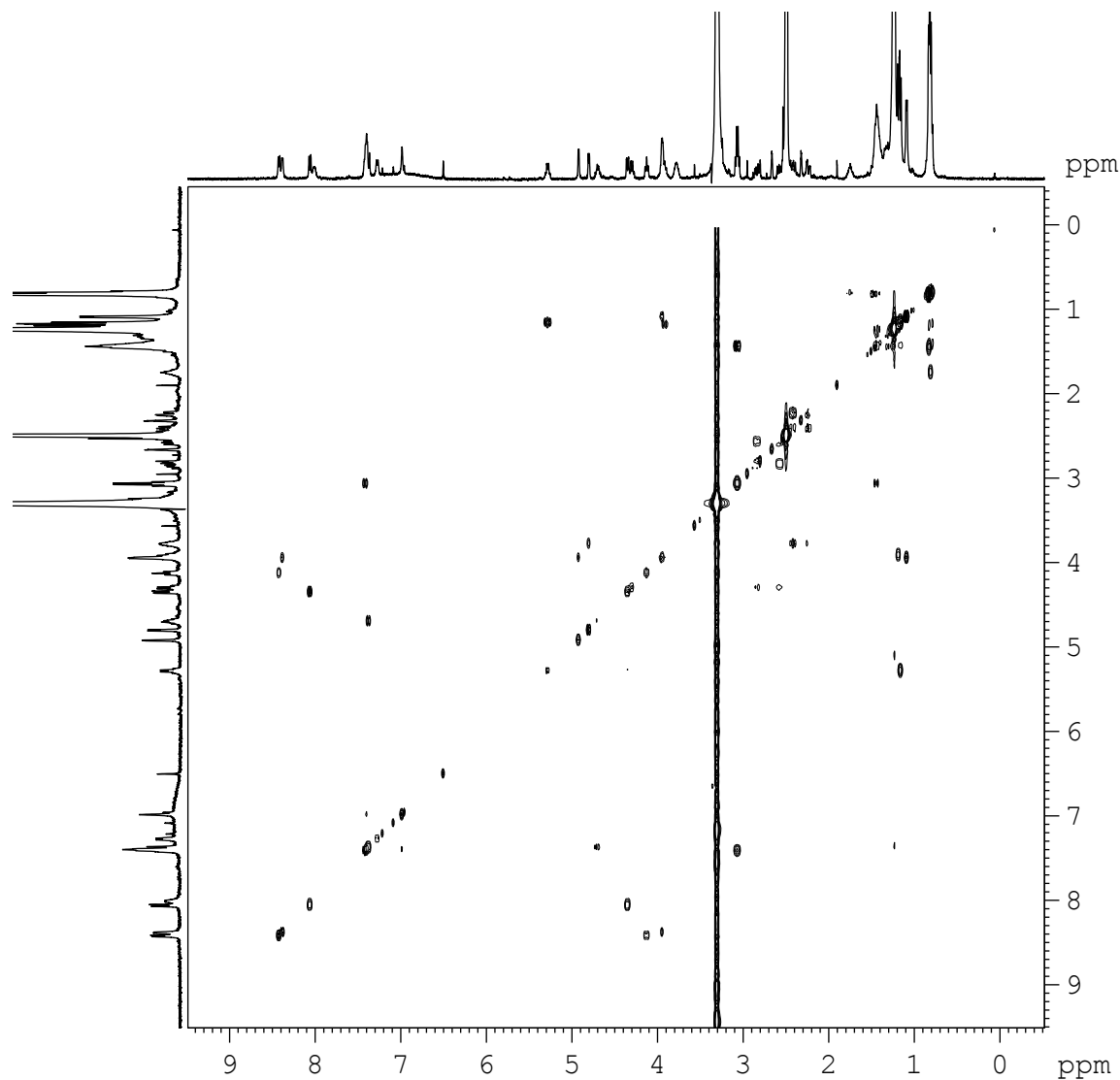
TD 424
SFO1 400.2102 MHz
FIDRES 9.438682 Hz
SW 10.000 ppm
FnMODE QF

F2 - Processing parameters

SI 1024
SF 400.2081747 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters

SI 256
MC2 QF
SF 400.2081728 MHz
WDW States-TPFI
SSB 0
LB 0 Hz
GB 0



Compound (S)-4 (HSQC, DMSO-*d*₆)

Current Data Parameters
NAME Circulocins1b
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100808
Time_ 17.15
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG invietgpsi
TD 2048
SOLVENT DMSO
NS 128
DS 8
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 1290.15
DW 124.800 usec
DE 6.00 usec
TE 0 K
CNST2 145.000000
d0 0.0000300 sec
d1 0.8000001 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.0000400 sec
d16 0.00015000 sec
D24 0.00172000 sec
DELTA 0.00171760 sec
DELTA1 0.00165800 sec
INO 0.00004969 sec
MCREST 0 sec
MCWRK 0.13333361 sec
ST1CNT 0

===== CHANNEL f1 =====
NUC1 1H
P1 5.80 usec
P2 11.60 usec
P28 2000.00 usec
PL1 -3.00 dB
SFO1 400.2101790 MHz

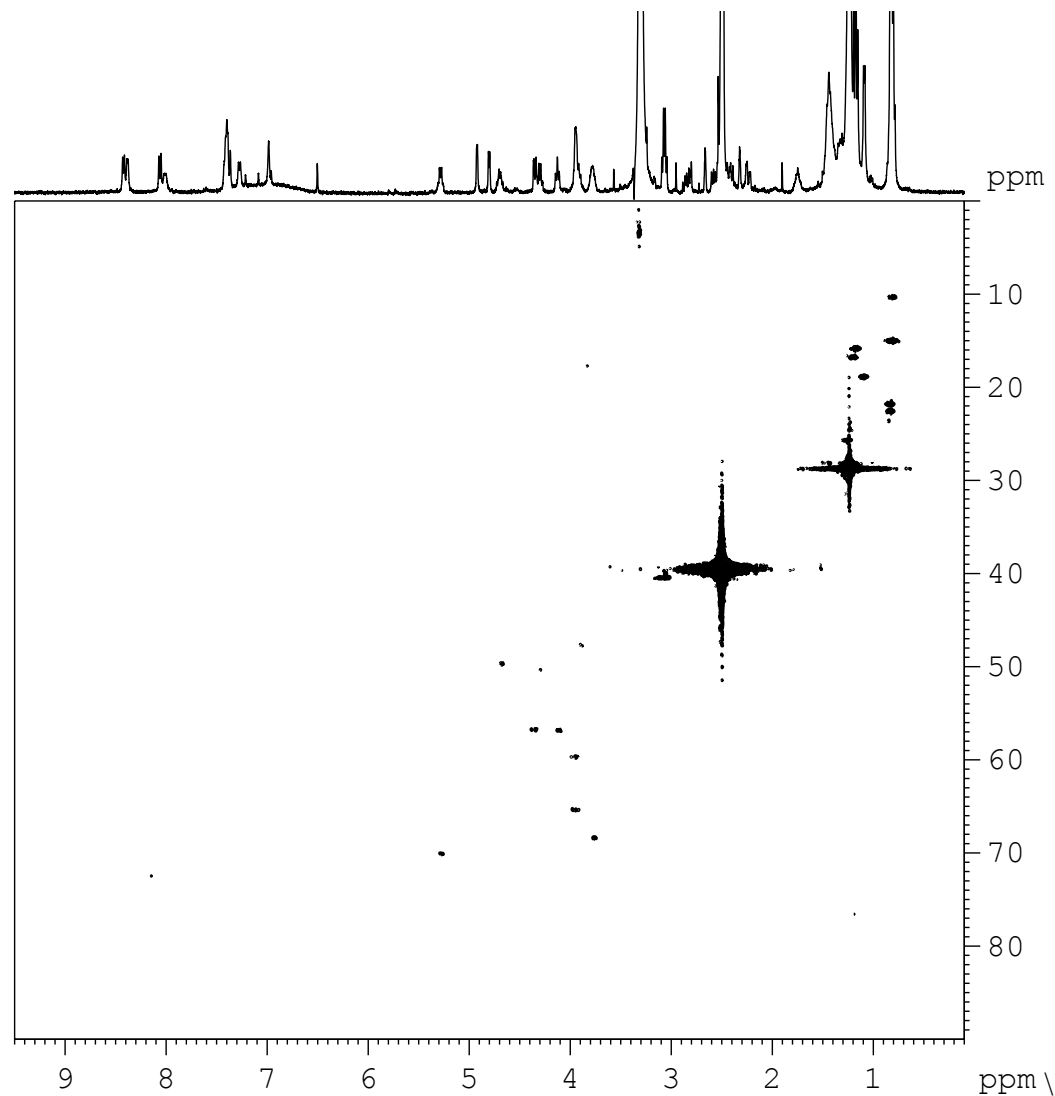
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 12.00 usec
P4 24.00 usec
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 10.48 dB
SFO2 100.6374576 MHz

===== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GFX1 0 %
GFX2 0 %
GPY1 0 %
GPY2 0 %
GFX1 80.00 %
GFX2 20.10 %
P16 1500.00 usec

F1 - Acquisition parameters
TD 512
SFO1 100.6375 MHz
FIDRES 19.654089 Hz
SW 99.992 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 400.2081739 MHz
WDW SINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 100.6324983 MHz
WDW
SSB 2
LB 0 Hz
GB 0



Compound (S)-4 (HMBC, DMSO-*d*₆)

```

Current Data Parameters
NAME      CirculocinSib-1222
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20101222
Time      13.52
INSTRUM   spect
PROBHD    5 mm BBI 1H-BS
PULPROG   inv4gplndgfp
TD        4096
SOLVENT   CD3CN
NS        300
DS        8
SWH        5592.841 Hz
FIDRES    1.365440 Hz
AQ        0.3662324 sec
RG        20642.5
DW        89.400 usec
DE        6.00 usec
TE        0.0 K
CNST2     145.0000000
d0         0.00000300 sec
d1         2.00000000 sec
d2         0.00344828 sec
d6         0.06250000 sec
d11        0.03000000 sec
d12        0.00002000 sec
d13        0.00004000 sec
d16        0.00015000 sec
IN0        0.00001988 sec
MCREST     0.00000000 sec
MCWRK     0.03000000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         5.85 usec
p2         11.70 usec
PL1        -3.00 dB
PL9        55.00 dB
SFO1       400.2095334 MHz

===== CHANNEL f2 =====
NUC2       13C
P3         12.00 usec
PL2        -6.00 dB
SFO2       100.6445019 MHz

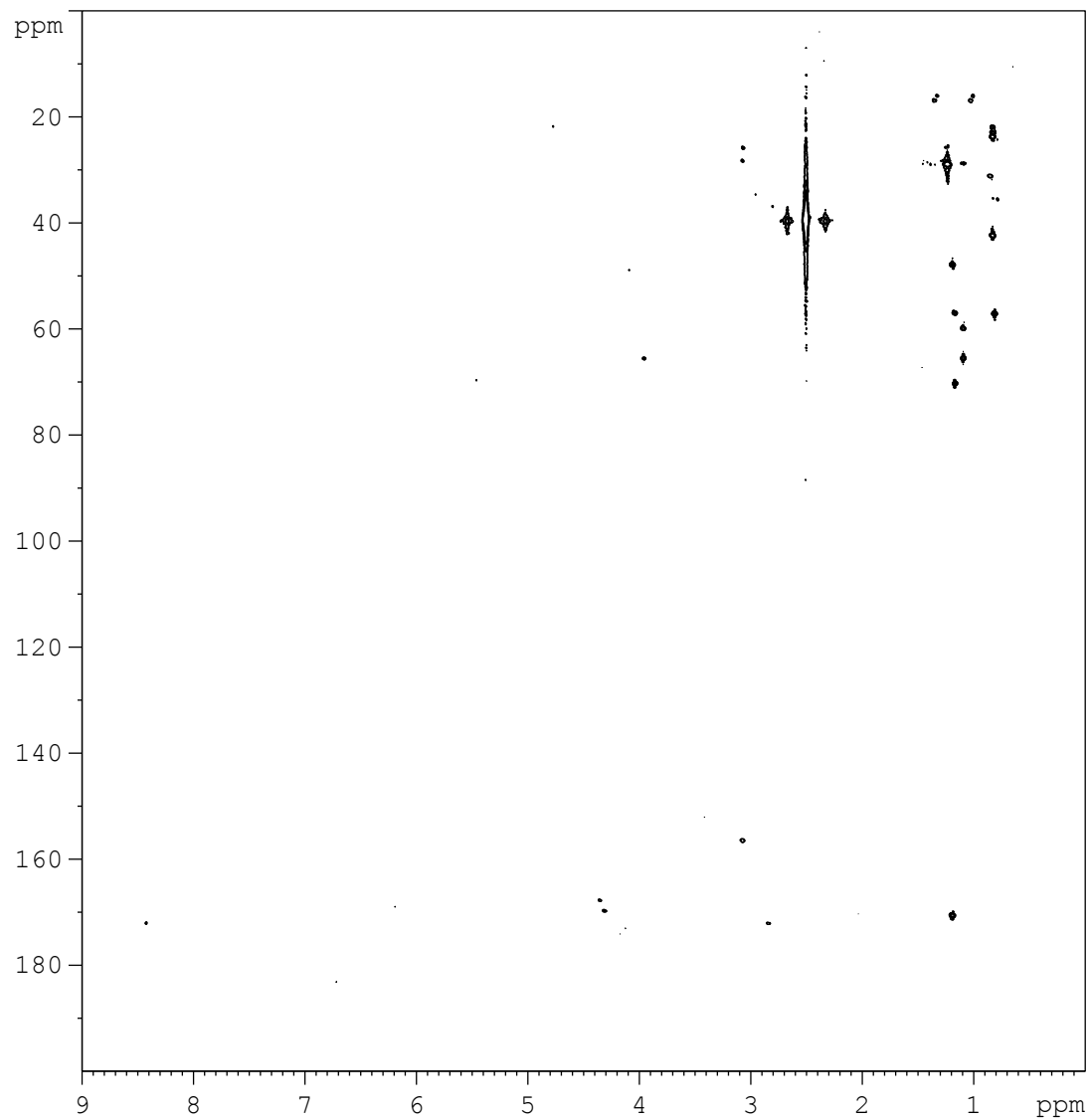
===== GRADIENT CHANNEL =====
GPNAM1     sine.100
GPNAM2     sine.100
GPNAM3     sine.100
GFX1       0.00 %
GFX2       0.00 %
GFX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       50.00 %
GPZ2       30.00 %
GPZ3       40.10 %
P16        1500.00 usec

F1 - Acquisition parameters
ND0        2
TD         512
SFO1       100.6445 MHz
FIDRES     49.135220 Hz
SW         249.961 ppm
FAMODE     QF

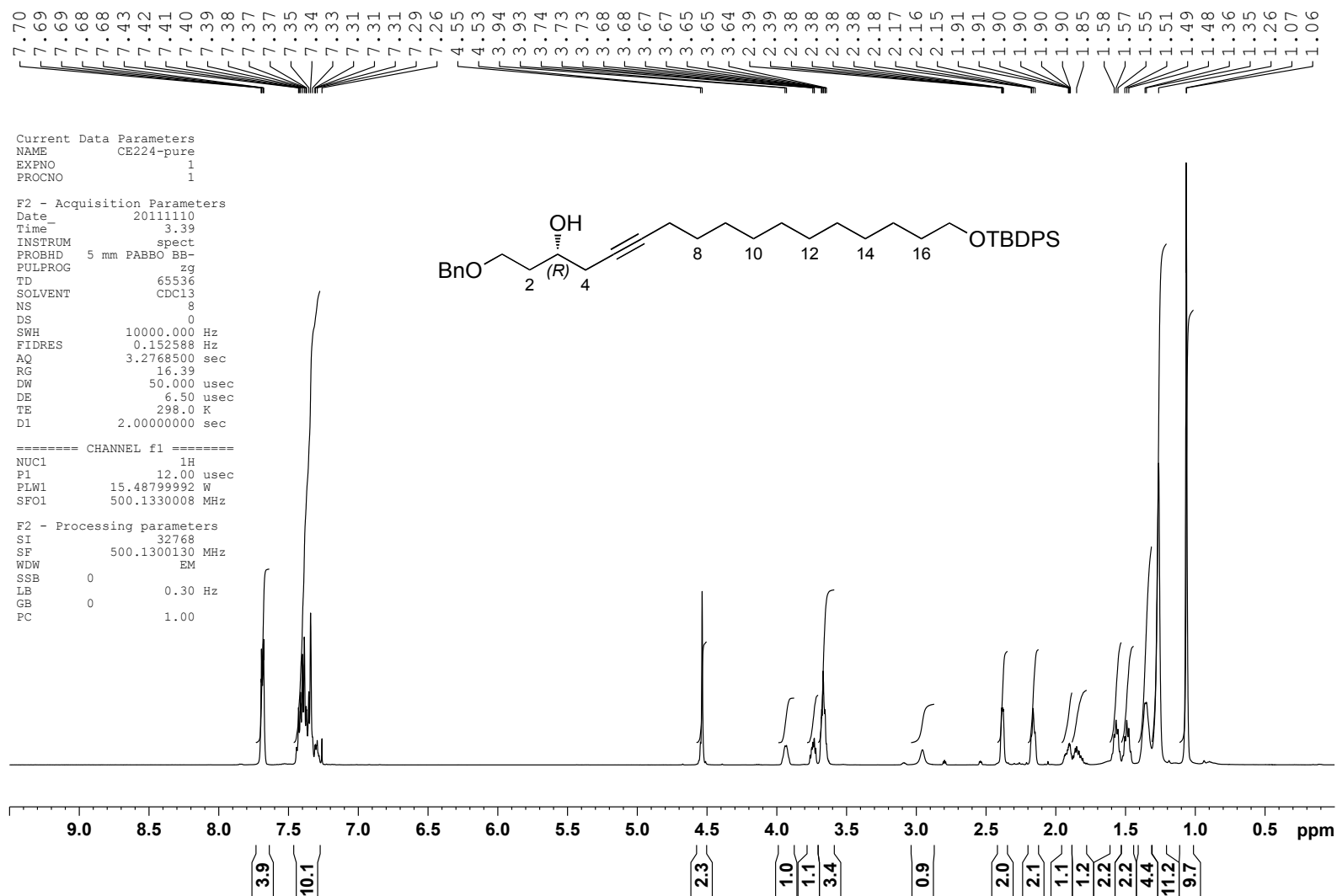
F2 - Processing parameters
SI         2048
SF         400.2081778 MHz
WDW        EM
SSB        0
LB         3.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         2048
MC2        QF
SF         100.6323439 MHz
WDW        EM
SSB        0
LB         12.00 Hz
GB         0

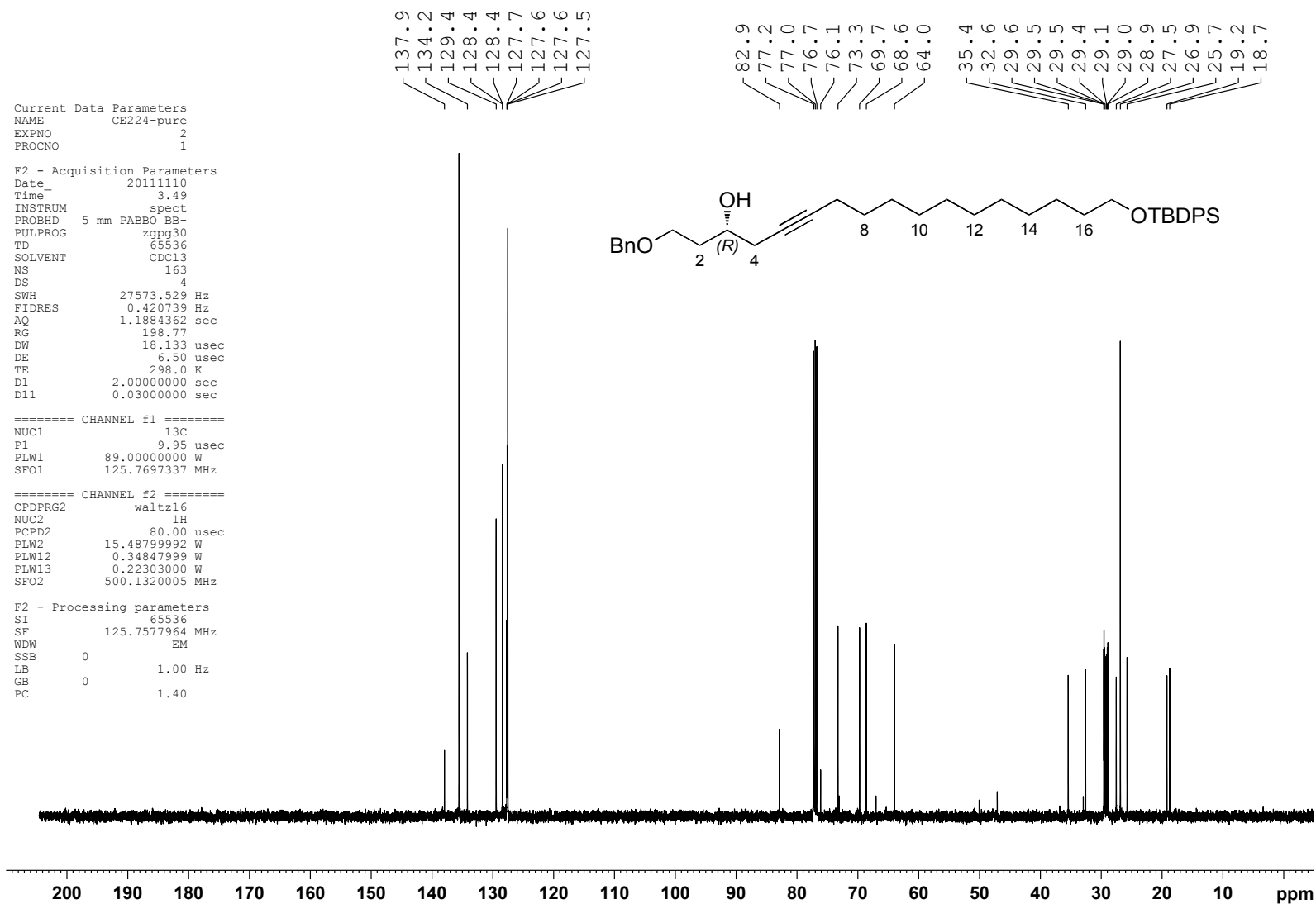
```



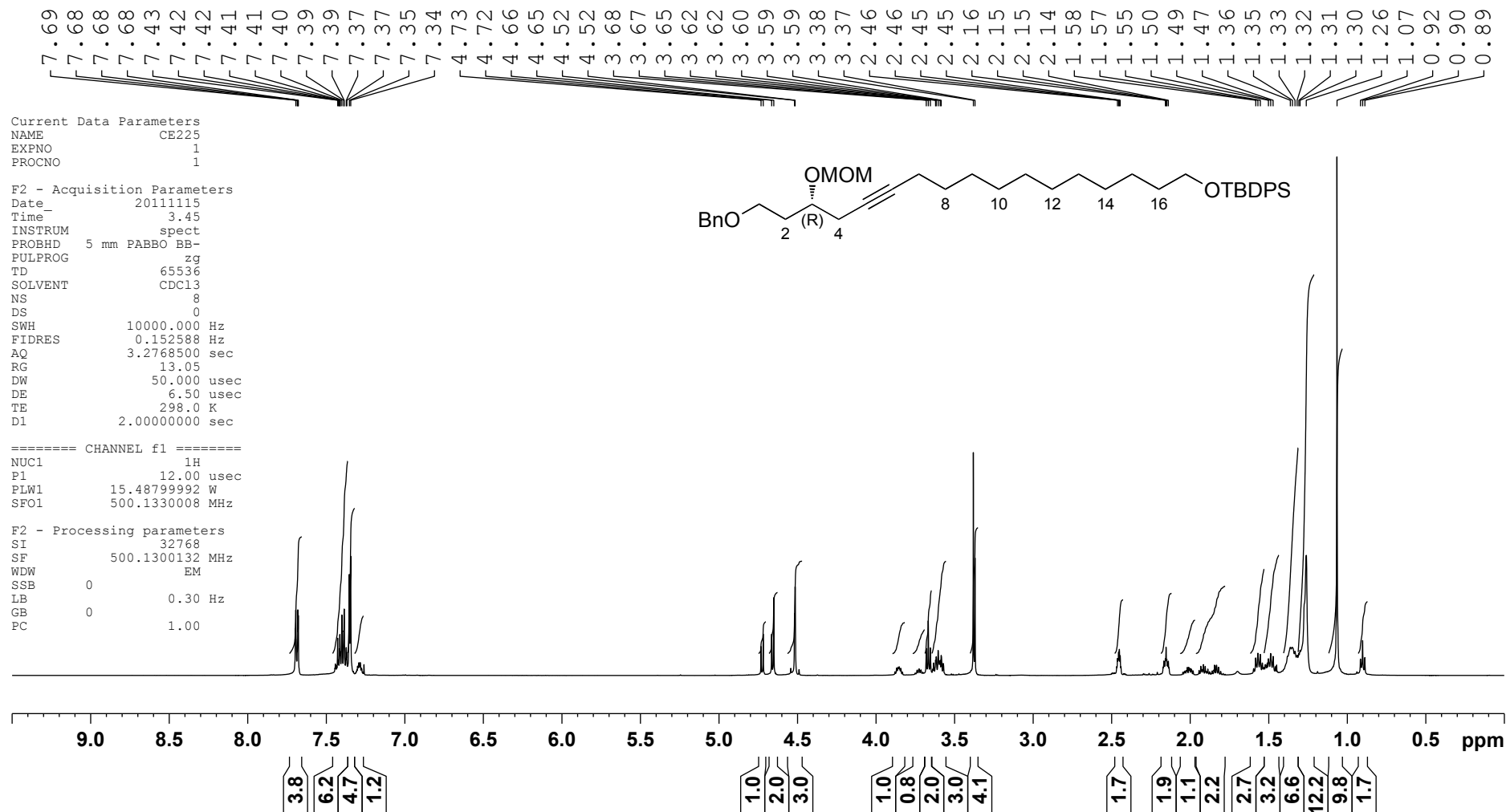
(R)-1-(Benzyloxy)-17-[(*tert*-butyldiphenylsilyl)oxy]heptadec-5-yn-3-ol (R)-25 (¹H NMR 400 MHz, CDCl₃)



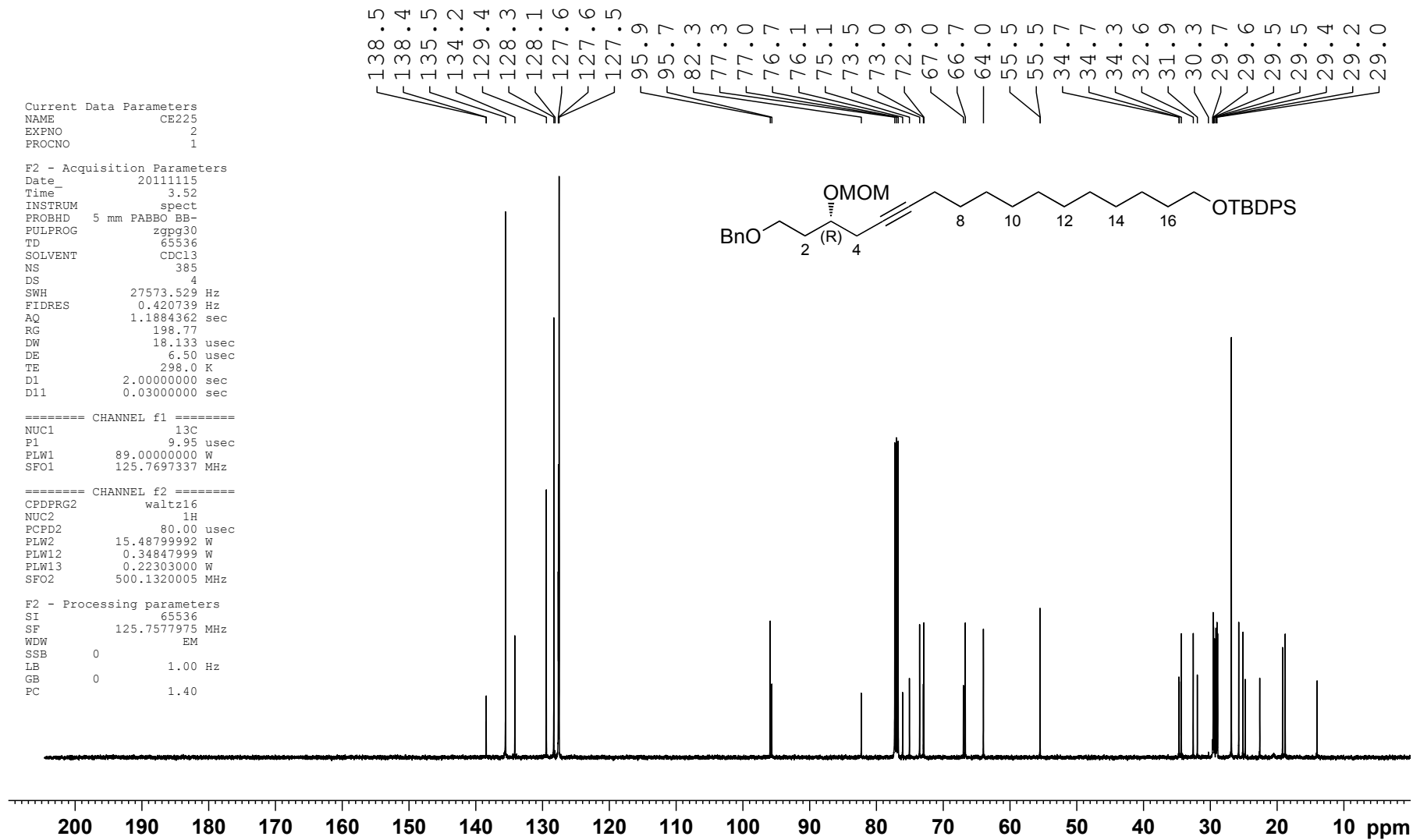
(R)-1-(Benzyloxy)-17-[(*tert*-butyldiphenylsilyl)oxy]heptadec-5-yn-3-ol (R)-25 (¹³CNMR 100 MHz, CDCl₃)



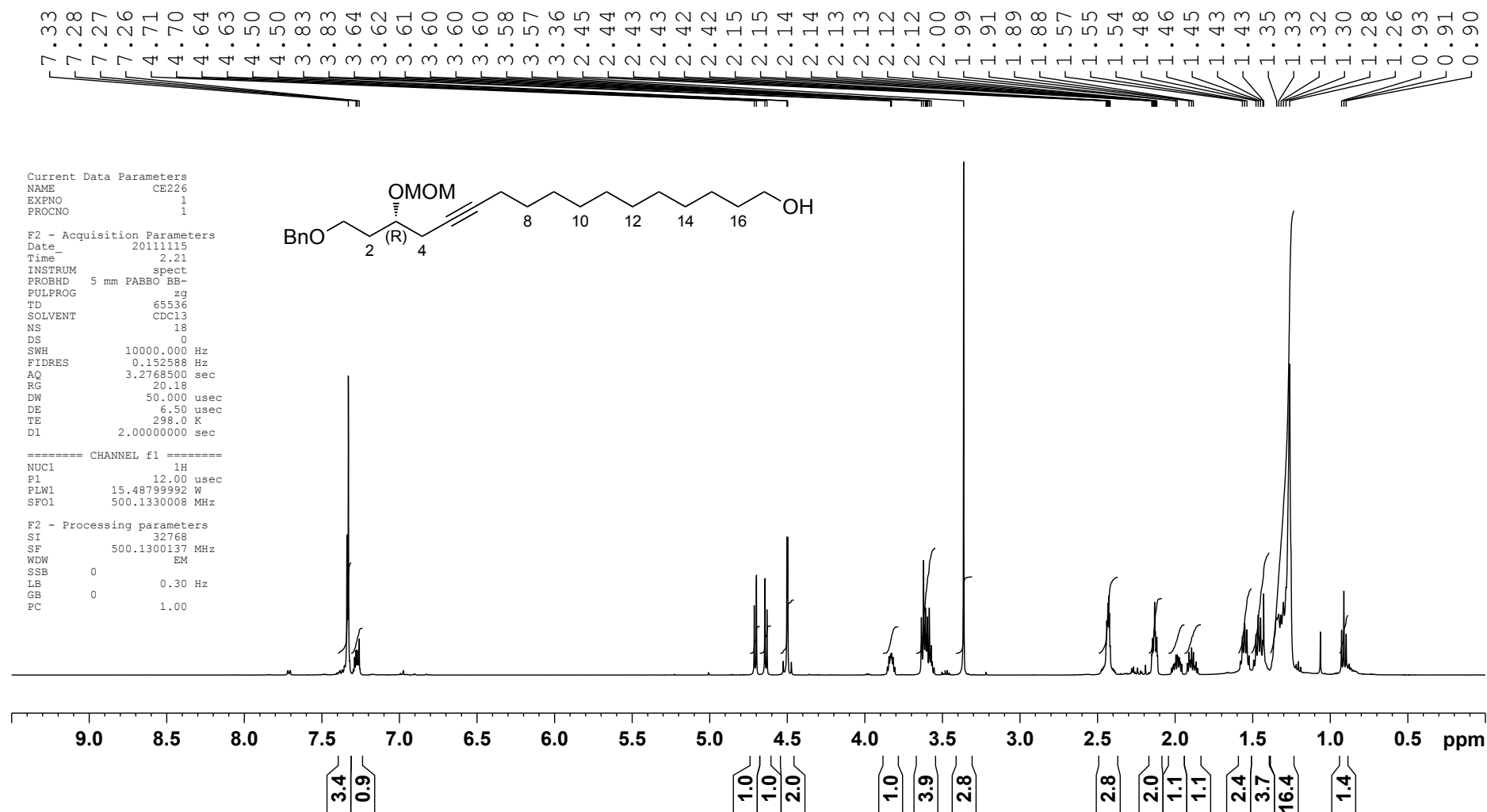
(R)-1-Benzyloxy-17-(tert-butylidiphenylsiloxy)-3-(methoxymethoxy)-heptadec-5-yne (R)-26 (¹H NMR 400 MHz, CDCl₃)



(R)-1-Benzyloxy-17-(tert-butyldiphenylsiloxy)-3-(methoxymethoxy)-heptadec-5-yne (R)-26 (^{13}C NMR 100 MHz, CDCl_3)



(R)-17-(Benzyloxy)-15-(methoxymethoxy)-heptadec-12-yn-1-ol (R)-27 (¹H NMR 400 MHz, CDCl₃)



(R)-17-(Benzyloxy)-15-(methoxymethoxy)-heptadec-12-yn-1-ol (R)-27 (^{13}C NMR 100 MHz, CDCl_3)

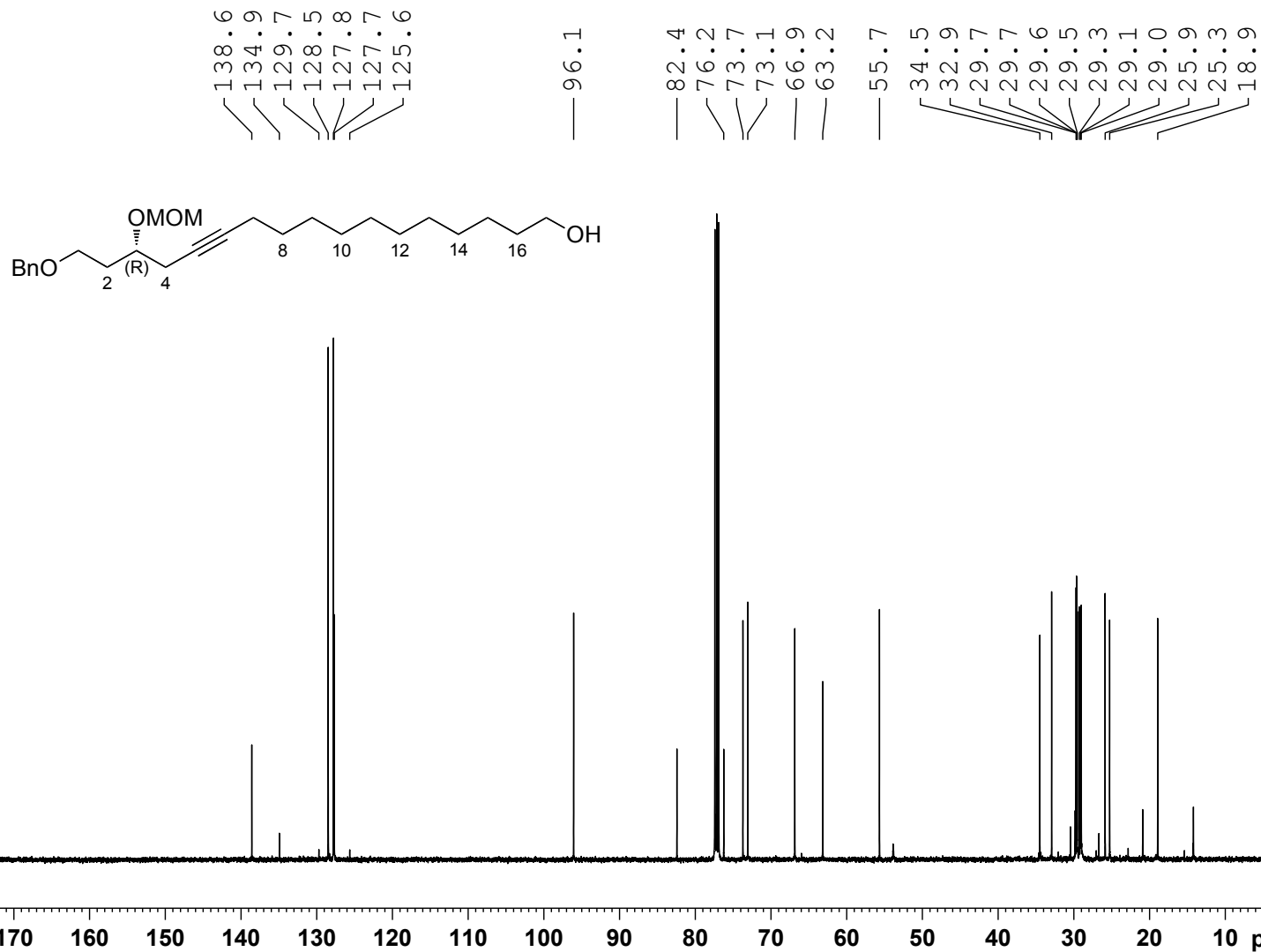
Current Data Parameters
NAME CE226
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111115
Time_ 2.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl_3
NS 919
DS 4
SWH 27573.529 Hz
FIDRES 0.420739 Hz
AQ 1.1884362 sec
RG 198.77
DW 18.133 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec

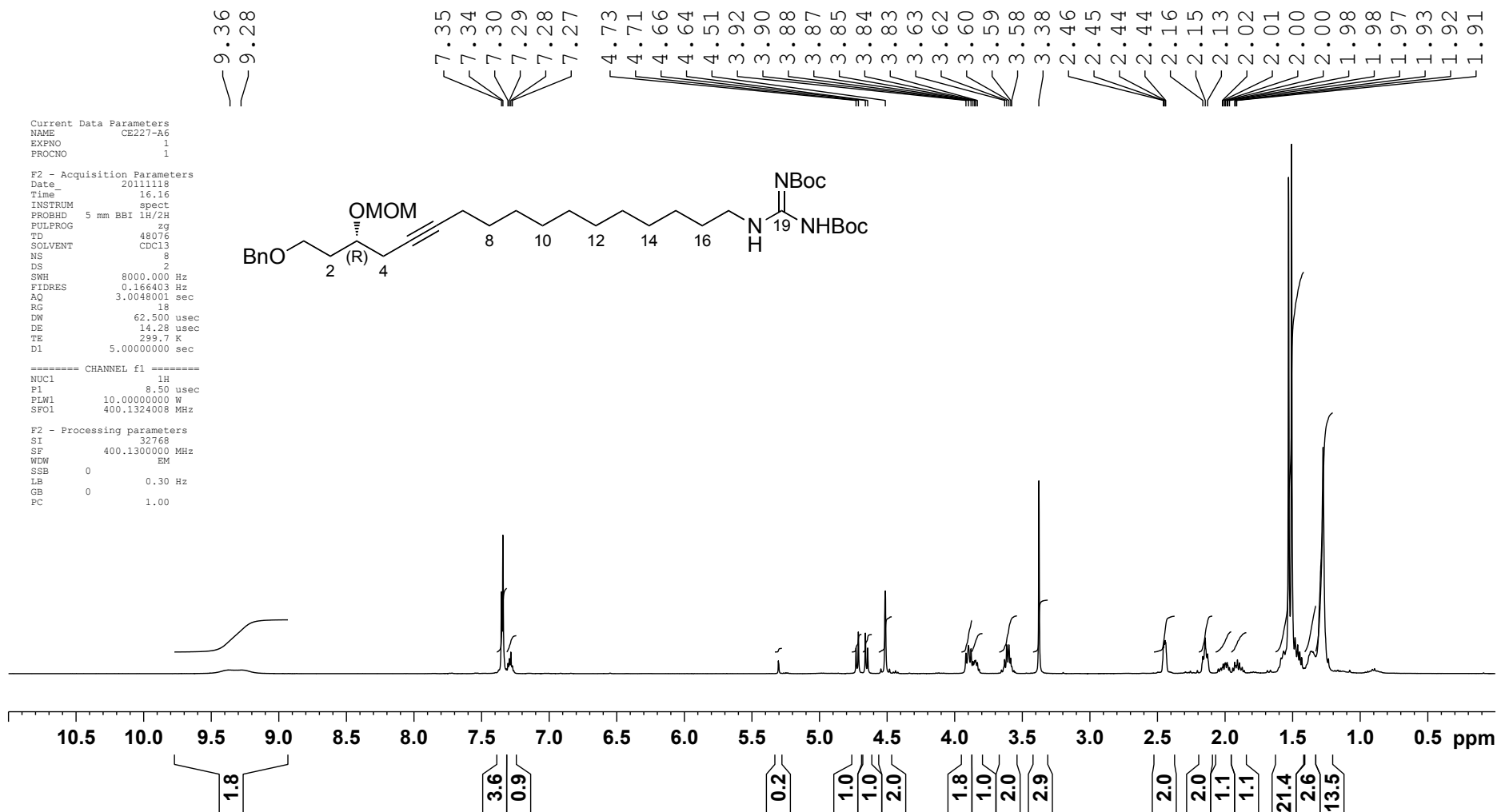
===== CHANNEL f1 =====
NUC1 ^{13}C
P1 9.95 usec
PLW1 89.00000000 W
SFO1 125.7697337 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 80.00 usec
PLW2 15.48799992 W
PLW12 0.34847999 W
PLW13 0.22303000 W
SFO2 500.1320005 MHz

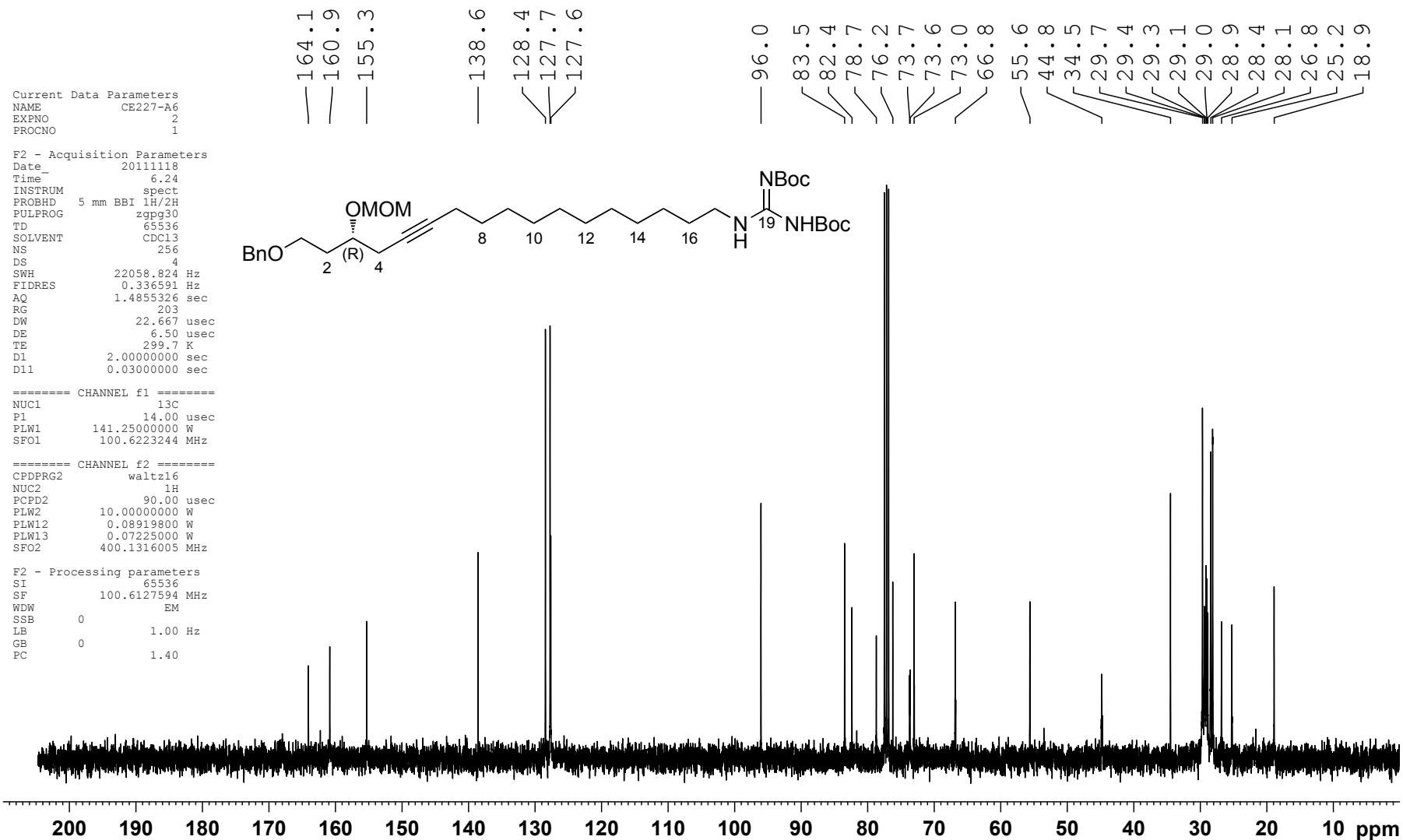
F2 - Processing parameters
SI 65536
SF 125.7577745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



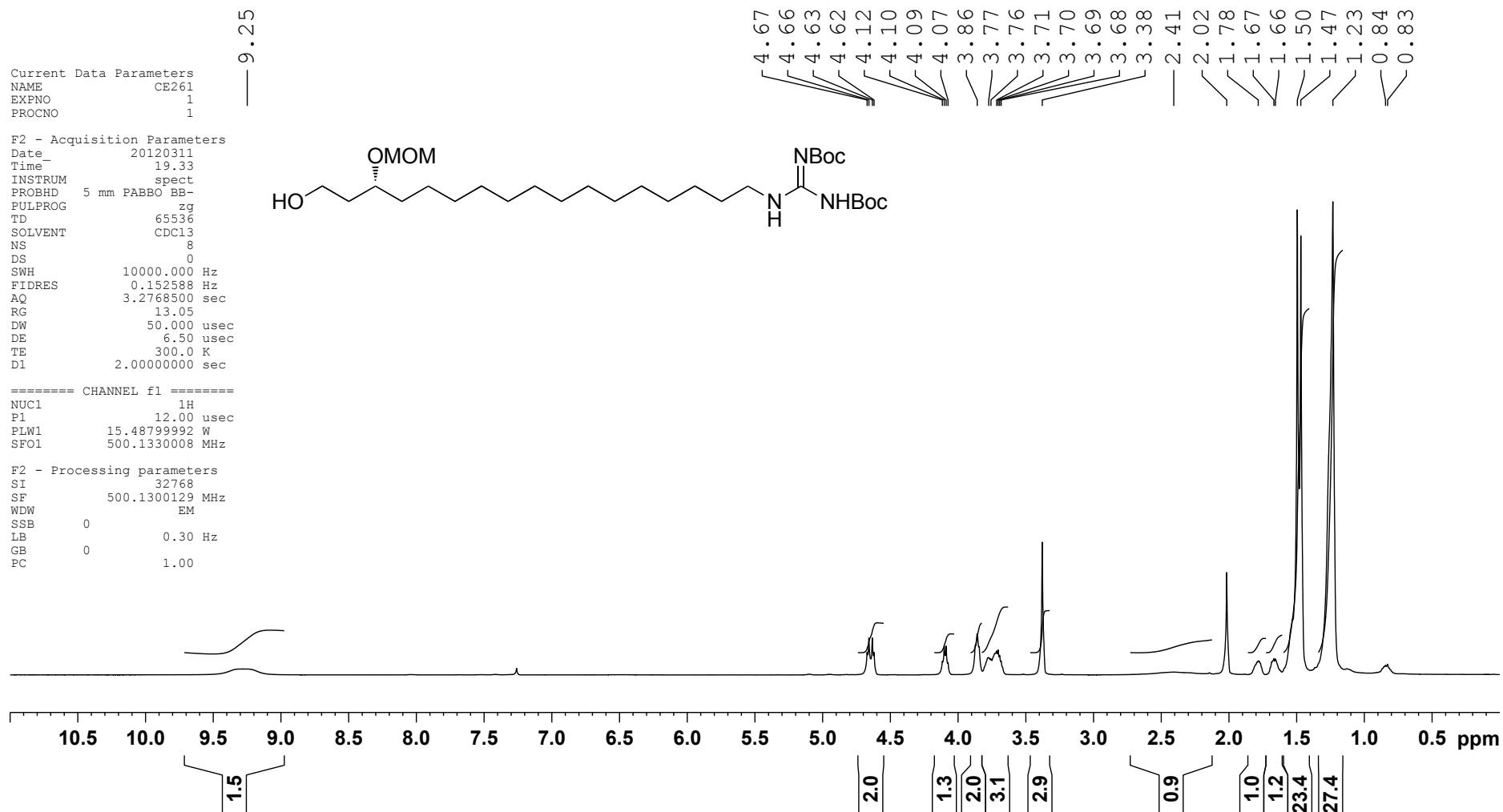
(R)-1-(Benzyloxy)-17-{[N,N'-bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-heptadec-5-yne (R)-28 (¹H NMR 400 MHz, CDCl₃)



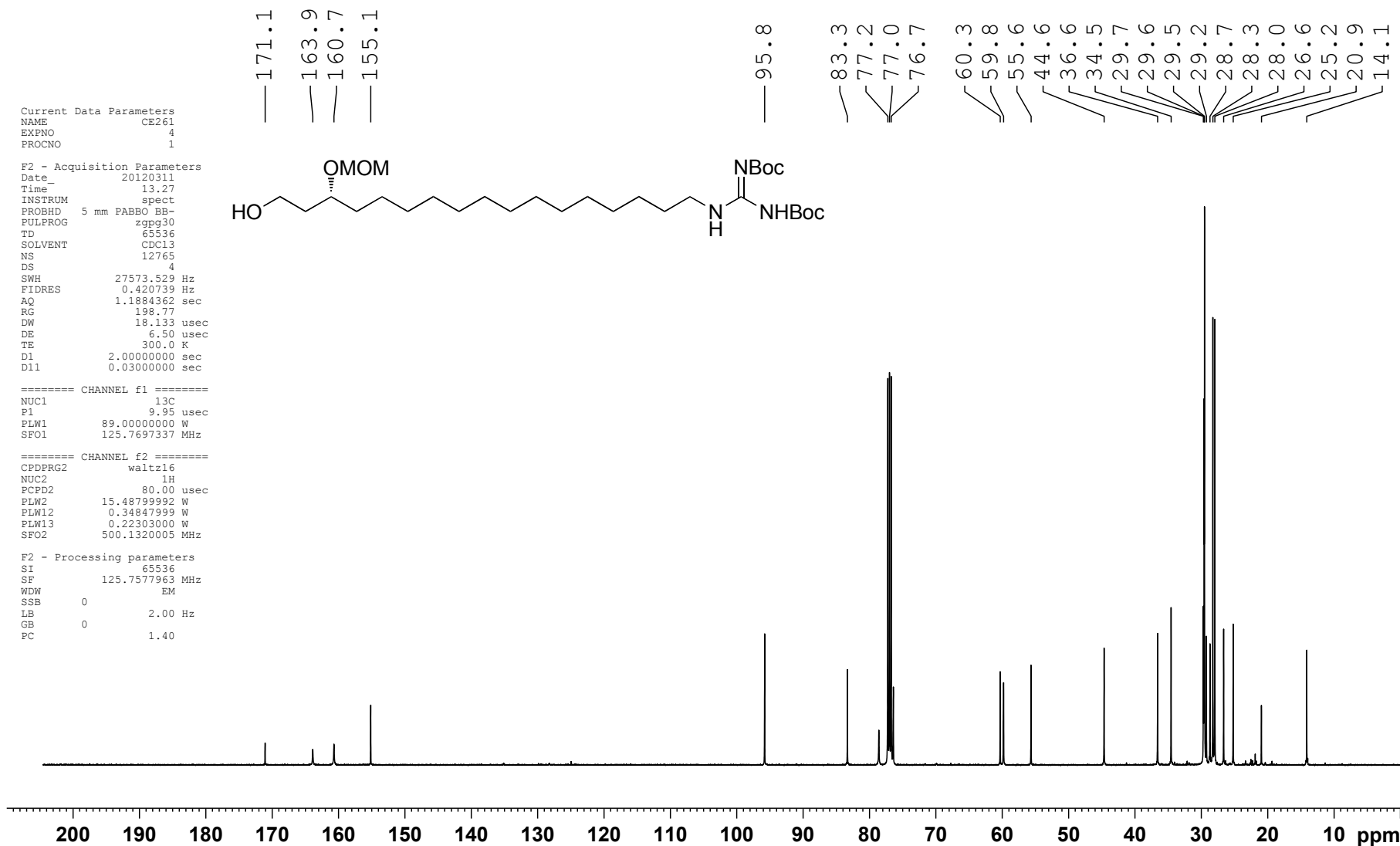
(R)-1-(Benzyloxy)-17-{[N,N'-bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-heptadec-5-yne (R)-28 (¹³C NMR 100 MHz, CDCl₃)



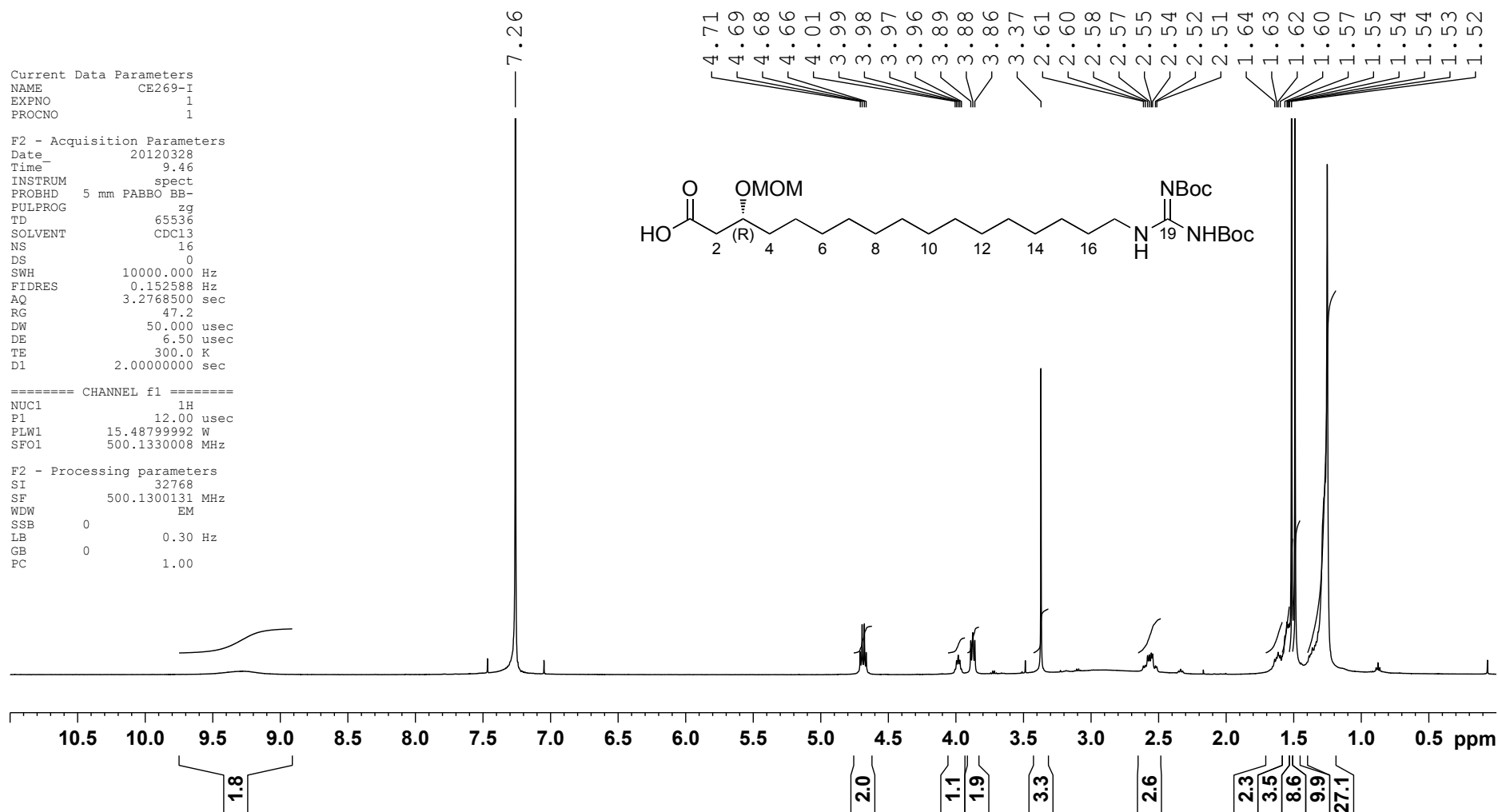
(R)-17-([N,N'-Bis(*tert*-butoxycarbonyl)]guanidino)3-(methoxymethoxy)-heptadecan-1-ol (R)-29 (¹H NMR 400 MHz, CDCl₃)



(R)-17-{[N,N'-Bis(*tert*-butoxycarbonyl)]guanidino}3-(methoxymethoxy)-heptadecan-1-ol (R)-29 (¹³C NMR 100 MHz, CDCl₃)



(R)-17-[N,N'-Bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-heptadecanoic acid (R)-9 (¹H NMR 400 MHz, CDCl₃)



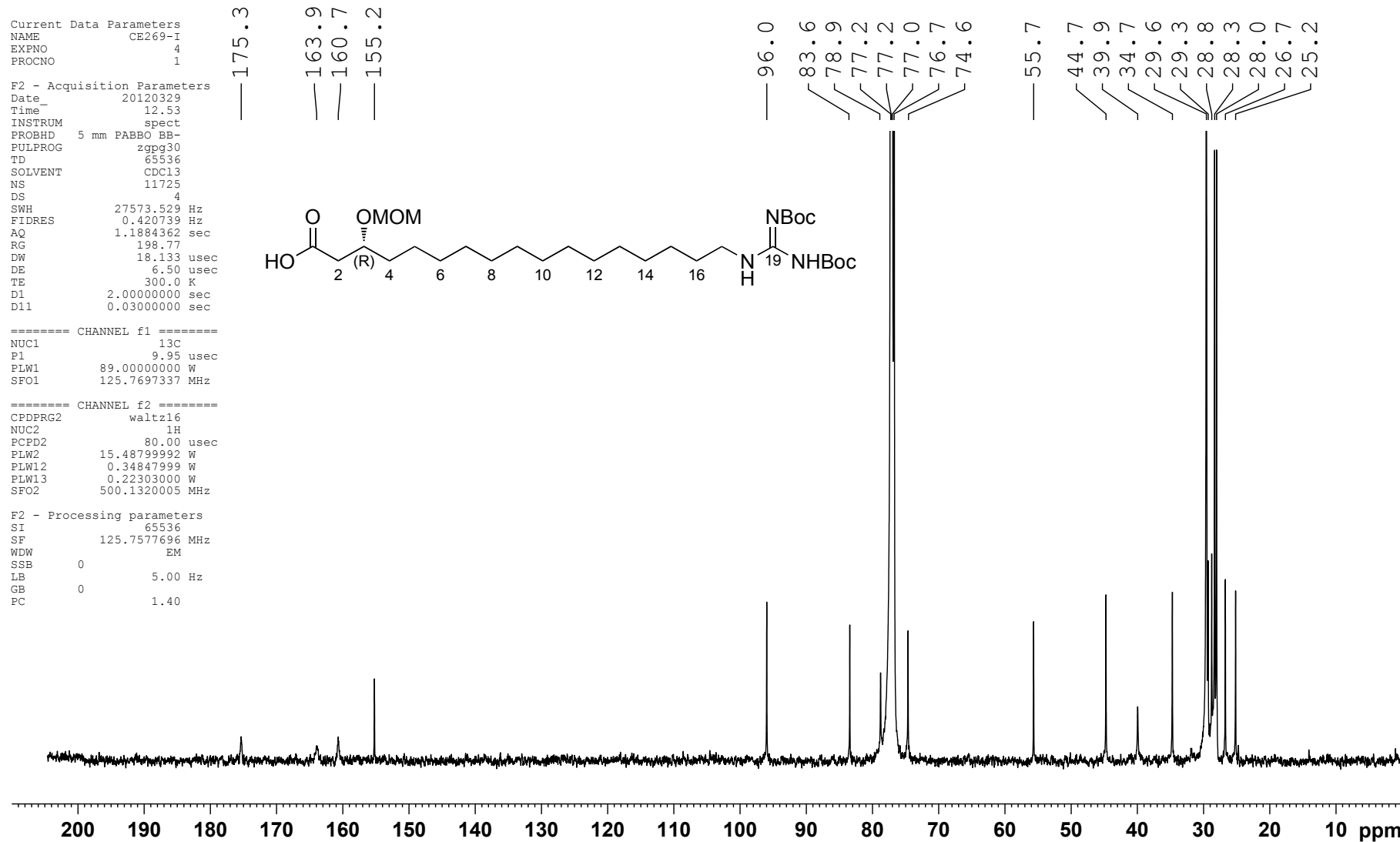
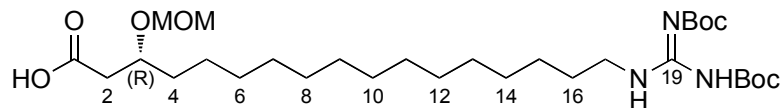
(R)-17-{[N,N'-Bis(*tert*-butoxycarbonyl)]guanidino}-3-(methoxymethoxy)-heptadecanoic acid (R)-9 (¹³C NMR 100 MHz, CDCl₃)

Current Data Parameters
 NAME CE269-I
 EXPNO 4
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120329
 Time_ 12.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 11725
 DS 4
 SWH 27573.529 Hz
 FIDRES 0.420739 Hz
 AQ 1.1884362 sec
 RG 198.77
 DW 18.133 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec

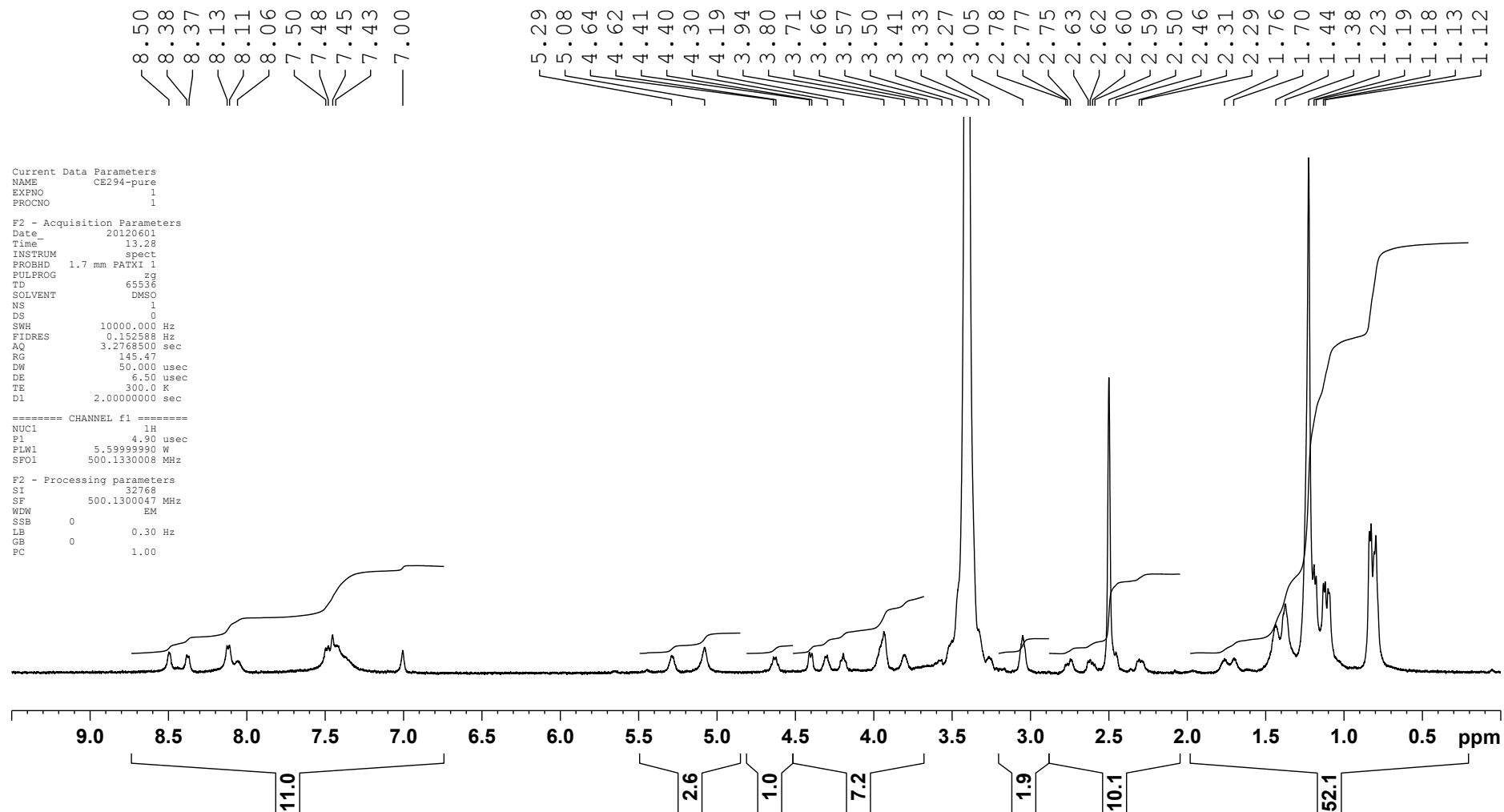
===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 9.95 usec
 PLW1 89.0000000 W
 SFO1 125.7697337 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 80.00 usec
 PLW2 15.48799992 W
 PLW12 0.34847999 W
 PLW13 0.22303000 W
 SFO2 500.1320005 MHz

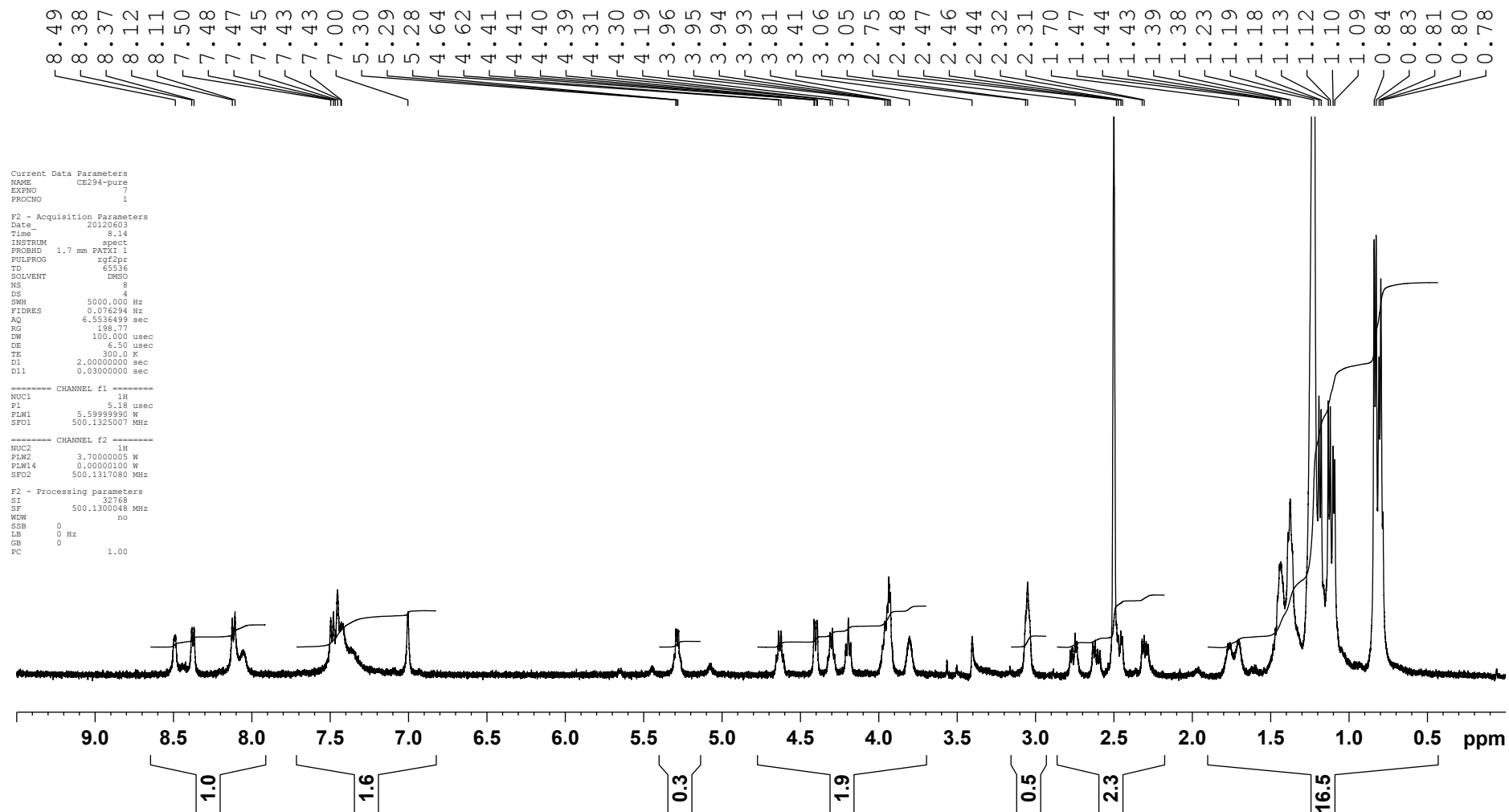
F2 - Processing parameters
 SI 65536
 SF 125.7577696 MHz
 WDW EM
 SSB 0
 LB 5.00 Hz
 GB 0
 PC 1.40



Compound (R)-6 (¹H NMR 500 MHz, DMSO-d₆)



Compound (R)-6 (¹H NMR with solvent suppression, 500 MHz, DMSO-d₆)



Compound (R)-6 (COSY, DMSO-*d*₆)

Current Data Parameters
 NAME CE294-pure
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120601
 Time_ 14.17
 INSTRUM spect
 PROBHD 1.7 mm PATXI 1
 PULPROG cosygpqff3pr.nmr
 TD 2048
 SOLVENT DMSO
 NS 40
 DS 16
 SWH 5000.000 Hz
 FIDRES 2.441406 Hz
 AQ 0.2048500 sec
 RG 198.77
 DW 100.000 usec
 DE 6.50 usec
 TE 300.0 K
 D0 0.00000300 sec
 D1 2.50000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D16 0.00020000 sec
 IN0 0.00019995 sec

===== CHANNEL f1 =====
 NUC1 1H
 P0 2.59 usec
 P1 5.18 usec
 PLW1 5.59999990 W
 SFO1 500.1325007 MHz

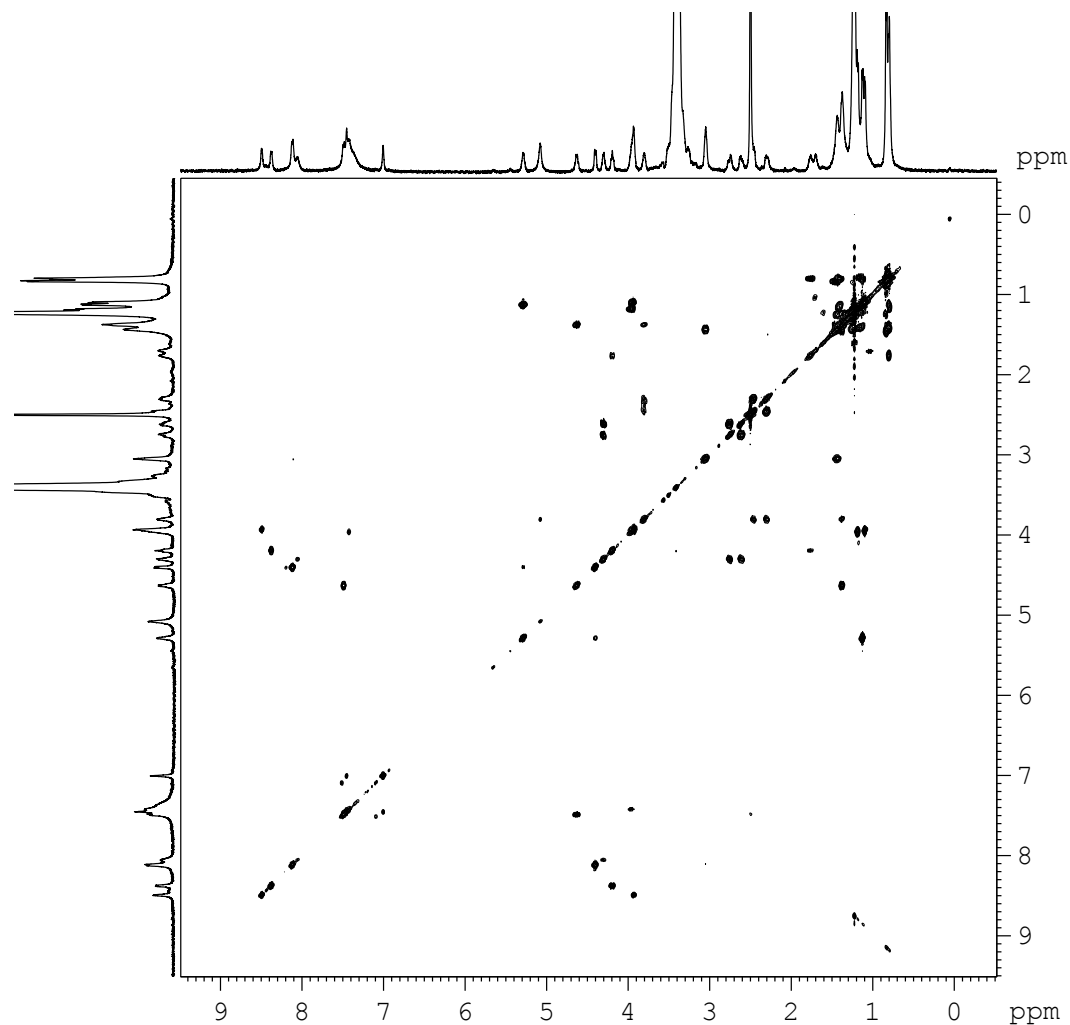
===== CHANNEL f3 =====
 NUC3 1H
 PLW3 3.70000005 W
 PLW9 0.00000100 W
 SFO3 500.1317080 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 256
 SFO1 500.1325 MHz
 FIDRES 19.536427 Hz
 SW 10.000 ppm
 FnmODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300014 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300030 MHz
 WDW
 SSB 0
 LB 0 Hz
 GB 0



Compound (R)-6 (HSQC, DMSO-*d*₆)

```
Current Data Parameters
NAME      CE294-pure
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20120602
Time      7.49
INSTRUM    spect
PROBHD     1.7 mm PAXXI 1
PULPROG    hsqcetgp5pr.nmr
TD          2048
SOLVENT    DMSO
NS          68
DS          16
SWH         5000.000 Hz
FIDRES     2.441406 Hz
AQ          0.2048500 sec
RG          198.77
DW          100.000 usec
DE          6.50 usec
TE          300.0 K
CNS2       145.0000000
D0          0.0000300 sec
D1          3.00000000 sec
D4          0.00172414 sec
D11         0.13000000 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00020000 sec
D21         0.00344828 sec
INO         0.00002210 sec
ZGPGTMS

===== CHANNEL f1 =====
NUC1        1H
P1          5.18 usec
P2          10.36 usec
P2B         1000.00 usec
PLW1        5.59999990 W
SFO1        500.1325007 MHz

===== CHANNEL f2 =====
CPDPRG2     gaird4
NUC2         13C
P3           9.80 usec
P4          19.60 usec
PCPD2       70.00 usec
PLW2        29.79999924 W
PLW12       0.58407998 W
SFO2        125.7684784 MHz

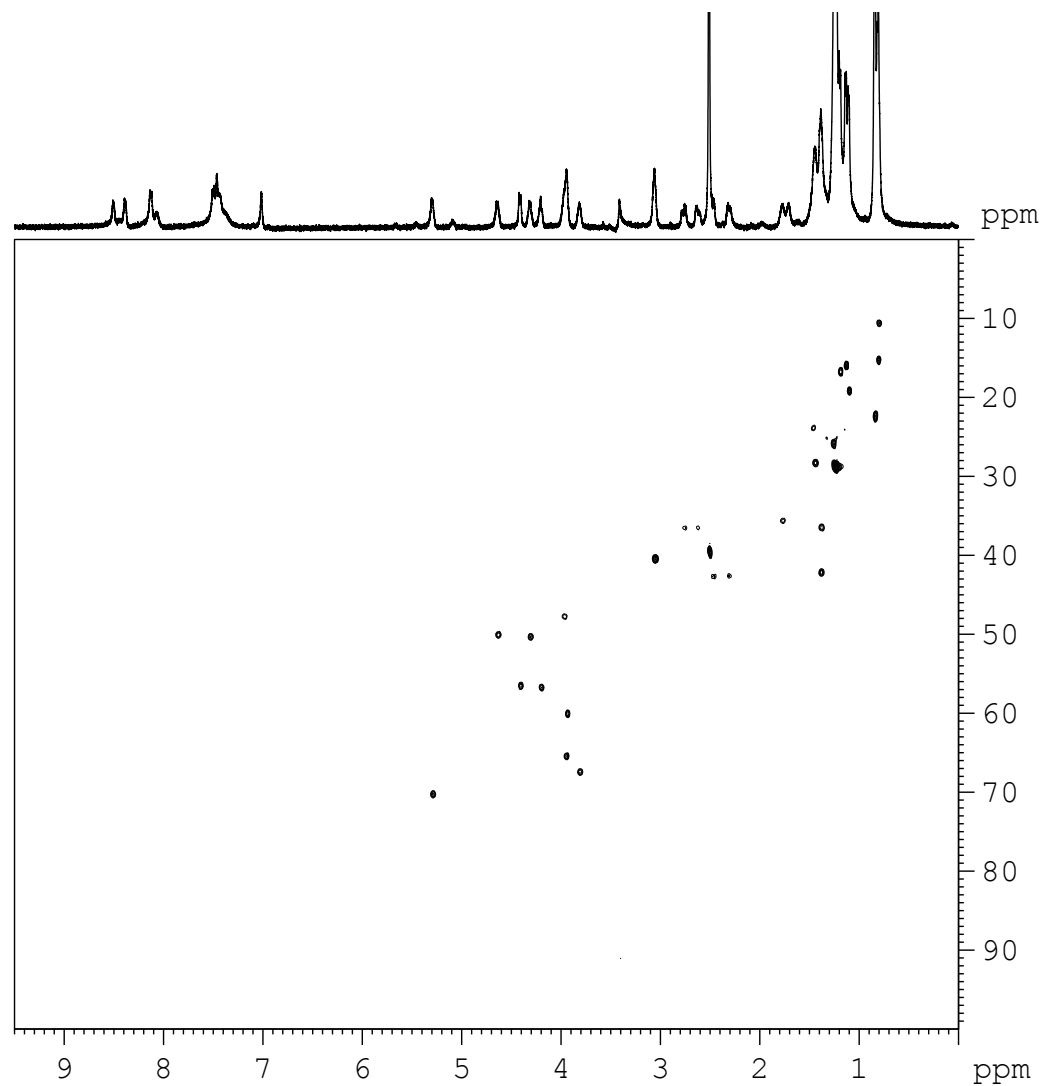
===== CHANNEL f3 =====
NUC3         1H
PLW3        3.70000005 W
PLW9        0.00000100 W
SFO3        500.1317080 MHz

===== GRADIENT CHANNEL =====
GPNAM1      SMSQ10.100
GPNAM2      SMSQ10.100
GP21        80.00 %
GP22        20.10 %
P16         1000.00 usec

F1 - Acquisition parameters
TD          256
SFO1        125.7685 MHz
FIDRES      88.430962 Hz
SW          180.000 ppm
F2MODE      Echo-Antiecho

F2 - Processing parameters
SI          1024
SF          500.1300021 MHz
WDW         QSINE
SSB         2
LB           0 Hz
GB           0
PC           1.00

F1 - Processing parameters
SI          1024
WC2         echo-antiecho
SF          125.7578572 MHz
WDW         SSB
LB           0 Hz
GB           0
```



Compound (R)-6 (HMBC, DMSO-*d*₆)

```
Current Data Parameters
NAME      CE294-pure
EXPNO     6
PROCNO    1

F2 - Acquisition Parameters
Date_     20120603
Time      0.28
INSTRUM    spect
PROBHD     1.7 mm PATXI 1
PULPROG    hmbcgp1pndqf3pr.nmr
TD         2048
SOLVENT    DMSO
NS         108
DS         16
SWH        5000.000 Hz
FIDRES     2.441406 Hz
AQ         0.2048500 sec
RG         198.77
DW         100.000 usec
DE         6.50 usec
TE         300.0 K
CNST2      145.0000000
CNST13     8.0000000
D0         0.00000300 sec
D1         3.00000000 sec
D2         0.00344828 sec
D6         0.06250000 sec
D11        0.03000000 sec
D12        0.00002000 sec
D16        0.00020000 sec
IN0        0.0001803 sec

===== CHANNEL f1 =====
NUC1       1H
P1         5.18 usec
P2         10.36 usec
PLW1       5.59999990 W
SFO1       500.1325007 MHz

===== CHANNEL f2 =====
NUC2       13C
P3         9.80 usec
PLW2       29.79999924 W
SFO2       125.7709936 MHz

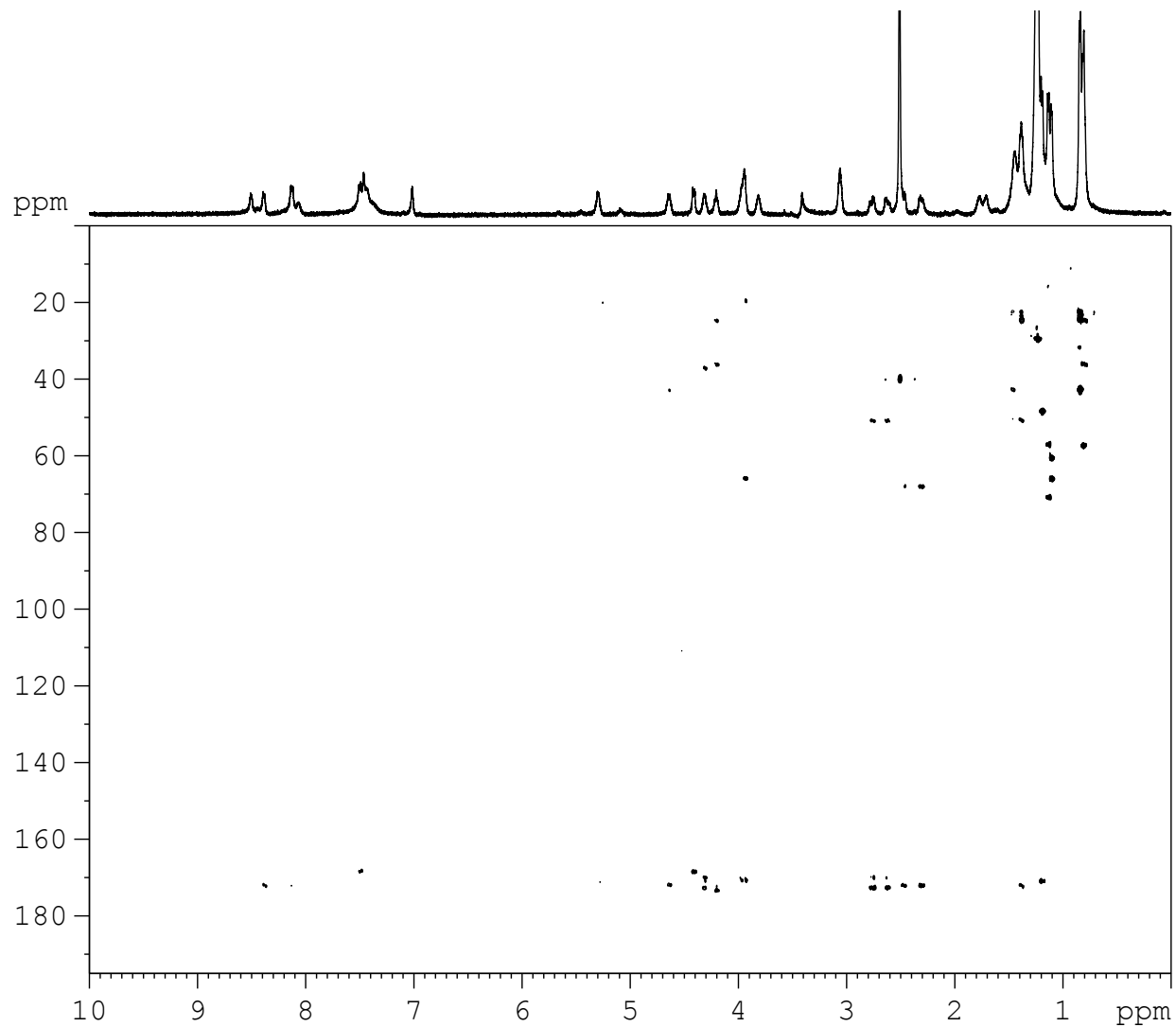
===== CHANNEL f3 =====
NUC3       1H
PLW3       3.70000005 W
PLW9       0.00000100 W
SFO3       500.1317079 MHz

===== GRADIENT CHANNEL =====
GPNAM1     SMSQ10.100
GPNAM2     SMSQ10.100
GPNAM3     SMSQ10.100
GP21       50.00 %
GP22       30.00 %
GP23       40.10 %
P16        1000.00 usec

F1 - Acquisition parameters
TD         256
SFO1       125.771 MHz
FIDRES     108.081726 Hz
SW         219.994 ppm
FhMODE     QF

F2 - Processing parameters
SI         4096
SF         500.1300000 MHz
WDW        SINE
SSB        0
LB         0 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        QF
SF         125.7577890 MHz
WDW        States
SSB        0
LB         0 Hz
GB         0
```



Analytical HPLC Traces of (R)-4, (S)-4, (R)-6

Analytical HPLC employed a Waters Sunfire C18 column (2.1×150 mm column, $5 \mu\text{m}$ particle size, flow rate of 0.8 mL min^{-1}). The mobile phase consisted of eluents A (0.1% TFA in water) and B (0.1% TFA in acetonitrile). A gradient of 26-60 % B over 45 min was used.

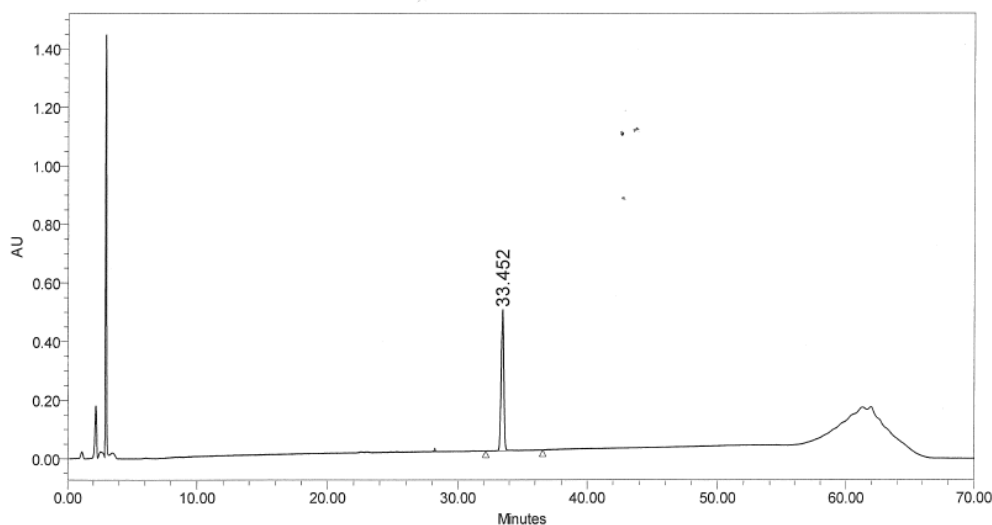
Analytical HPLC trace of compound (R)-4



The University of Sydney, School of Chemistry Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	G_26to60ov45_P210to300_D2mL
Sample Name:	CirculocinR	Sample Set Name:	circulocina
Sample Type:	Unknown	Date Acquired:	3/08/2010 7:46:03 PM EST
Vial:	2	Proc. Method:	NoPeaks
Injection #:	1	Date Processed:	4/08/2010 9:50:14 AM EST
Injection Volume:	10.00 ul	Channel Name:	PDA Max Plot 190.0 - 800.0
Run Time:	70.0 Minutes	Proc. Chnl. Descr.:	PDA MaxPlot (190.0 nm to 800.0 nm)
Chromatogram_Info:	Sunfire narrow bore		



	RT	Area	% Area	Height
1	33.452	6922226	100.00	481168

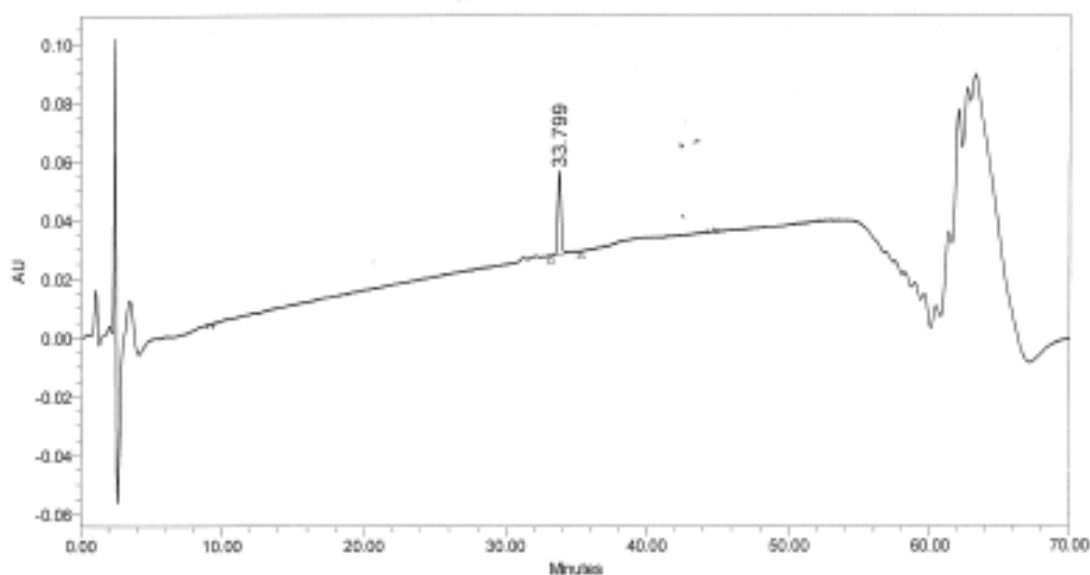
Analytical HPLC trace of compound (S)-4



The University of Sydney, School of Chemistry
Single Channel Report

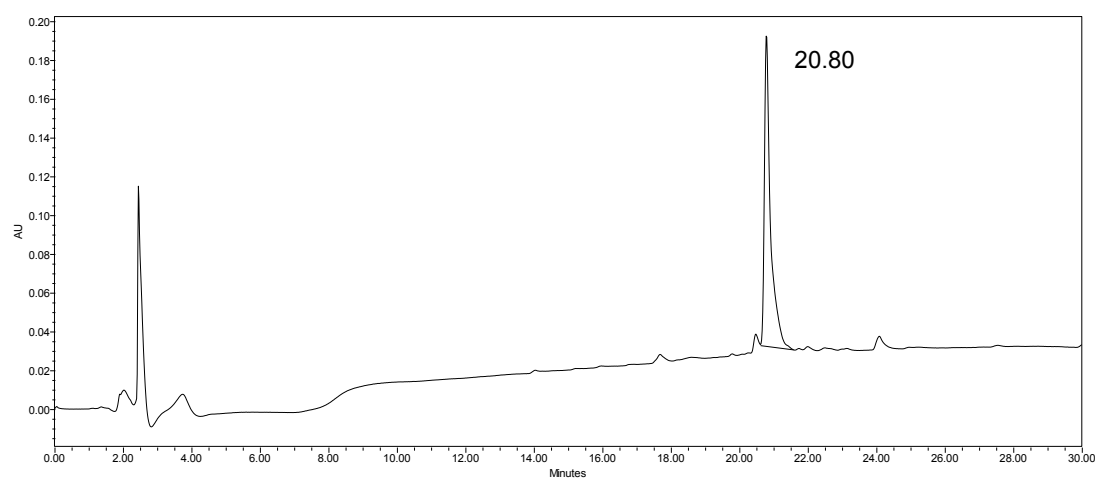
SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	G_26to80ov45_P210to300_D2mL
Sample Name:	CirculocinS	Sample Set Name:	JC_KF_04082010
Sample Type:	Unknown	Date Acquired:	4/08/2010 9:39:56 PM EST
Vial:	6	Proc. Method:	NoPeaks
Injection #:	1	Date Processed:	5/08/2010 11:20:38 AM EST
Injection Volume:	10.00 ul	Channel Name:	214.0nm@1
Run Time:	70.0 Minutes	Proc. Chnl. Descr.:	PDA.214.0 nm
Chromatogram_Info:	sunfire C18 analytical		



	RT	Area	% Area	Height
1	33.799	423557	100.00	28285

Analytical HPLC trace of compound (*R*)-6



References

1. He, H.; Shen, B.; Korshalla, J.; Carter, G. T. *Tetrahedron* **2001**, 57, 1189.