

SUPPLEMENTARY MATERIALS

Isolation, Structural Analysis and Biological Activity Assays of Biselisabethoxanes A and B: Two Dissymmetric *Bis*-Diterpenes from the Southwestern Caribbean Sea Gorgonian Coral *Pseudopterogorgia elisabethae*

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Table S1. ^1H NMR (500 MHz), ^{13}C NMR (125 MHz), ^1H - ^1H COSY, HMBC, and NOESY spectroscopic data for biselisabethoxane A (1) in CDCl_3^a

Atom	δ_{H}	δ_{C}	^1H - ^1H COSY	HMBC ^c	NOESY
1	3.60	36.8	H2 α β , H14		Me20
2 α	1.22	40.2	H1, H2 β , H3	H1, Me18	H4
2 β	1.94		H1, H2 α		
3	1.21	34.0	H2 α , H4, Me18	H5 β , Me18	H5 β , Me18
4	2.01	44.6	H3, H5 α	H2 α β , H3, H6 α β , Me18	H2 α
5 α	0.95	27.7	H4, H5 β , H6 α β		Me18
5 β	2.03		H5 α , H6 β		H3, H7
6 α	2.15	32.0	H5 α , H6 β , H7	H7, Me19	
6 β	1.30		H5 α β , H6 α , H7		
7	3.20	28.6	H6 α β , Me19	H5 β , H6 α β , Me19	H5 β , Me19
8		126.2		H7, Me19, C9-OH	
9		147.1		H7, C9-OH	
10		137.2		Me20, C9-OH	
11		129.6		H1, Me20	
12		129.6		H1, H14, Me20	
13		135.9		H1, H5 β , H7	
14	4.94	131.4	H1, Me16, Me17	H1, Me16, Me17	Me16
15		128.3		H1, Me16, Me17	
16	1.66	25.4	H14	H14, Me17	H14
17	1.70	17.5	H14	H14, Me16	
18	1.02	20.0	H3	H3	H3, H5 α
19	1.25	23.1	H7	H7	H7
20	1.96	15.6			H1
9-OH	5.97				
1'		61.6		H5 α ', H6', H8 α '	
2'		141.1		H3', H4 α ', Me18', C17'-OH	
3'	3.14	28.7	H4 β ', Me18'	H5 α ', Me18'	
4 α '	1.46	24.3	H4 β ', H5 α β '	H3', H5 β ', H6', Me18'	
4 β '	1.78		H3', H4 α ', H5 α '		
5 α '	1.55	19.3	H4 α β ', H5 β '	H3'	
5 β '	1.80		H4 α ', H5 α ', H6'		
6'	2.35	40.3	H5 β ', H7'	H8 α ', Me19'	Me18', Me19'
7'	2.05	41.7	H6', H8 α β ', Me19'	H6', H8 β ', Me19'	
8 α '	2.22	44.5	H7', H8 β '	Me19'	
8 β '	0.97		H7', H8 α '		
9'	— ^b	53.4 ^b		H8 α β '	
10'	— ^b	60.7 ^b		Me12', Me13', Me20'	Me19', Me20'
11'		~85.0 ^b		Me12', Me13'	
12'	1.01	26.8 ^b		Me13'	Me13'
13'	1.02	26.9 ^b		Me12'	Me12'
14'		203.1		H6', H9', Me20'	
15'		69.2 ^b		Me20'	
16'		~196.0 ^b		Me20'	
17'		146.9		H3', C17'-OH	
18'	1.16	17.7	H3'		H6'
19'	1.06	18.1	H7'	H6'	H6', H10'
20'	1.66	15.6			H10'
17'-OH	6.18				

^a Spectra were recorded at 25°C. Chemical shift values are in ppm relative to TMS (0.00 ppm) or CDCl_3 (77.0 ppm) signals.

^b Detection of this signal was complicated by the increased linewidths associated with slower tumbling, and the spectral overlap from the large number of unique signals. Either this signal was not detected, or it appeared as a broad low intensity signal. ^c Protons correlated to carbon resonances in ^{13}C column. Parameters were optimized for $^2J = 6$ and 8 Hz.

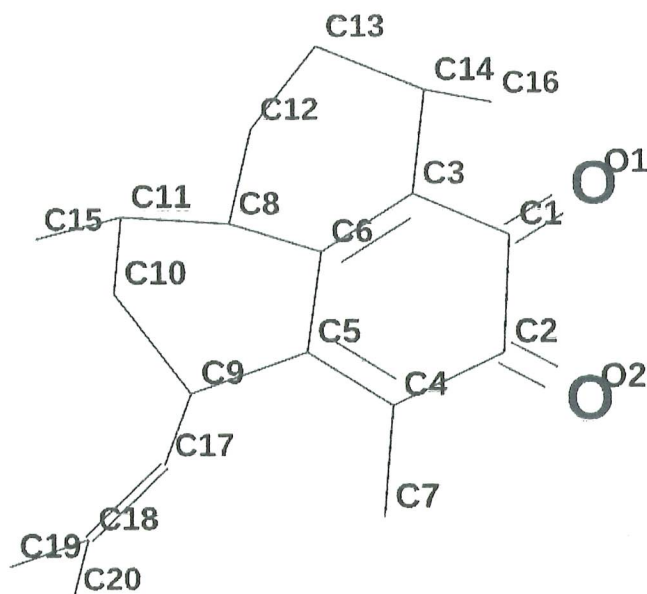
Table S2. ^1H NMR (500 MHz), ^{13}C NMR (125 MHz), ^1H - ^1H COSY, HMBC, and NOESY spectroscopic data for biselisabethoxane B (**2**) in CDCl_3 ^a

Atom	δ_{H}	δ_{C}	^1H - ^1H COSY	HMBC ^b	NOESY
1	3.65	36.7	H2 α β , H14		Me20
2 α	1.18	40.3	H1, H2 β , H3	H1, Me18	H4
2 β	1.94		H1, H2 α		
3	1.20	34.2	H2 α , H4, Me18	H5 β , Me18	H5 β , Me18
4	1.98	44.4	H3, H5 α	H2 α β , H3, H6 α β , Me18	H2 α
5 α	0.97	28.3	H4, H5 β , H6 α β		Me18
5 β	2.07		H5 α , H6 β		H3, H7
6 α	2.17	31.8	H5 α , H6 β , H7	H7, Me19	
6 β	1.30		H5 α β , H6 α , H7		
7	3.35	27.6	H6 α β , Me19	H5 β , H6 α β , Me19	H5 β , H14', Me16', Me-17'
8		126.3		H7, Me19, C9-OH	
9		137.2		H7, C9-OH	
10		137.8		Me20, C9-OH	
11		122.3		H1, Me20	
12		129.7		H1, H14, Me20	
13		132.3		H1, H5 β , H7	
14	5.00	131.8	H1, Me16, Me17	H1, Me16, Me17	Me16
15		127.6		H1, Me16, Me17	
16	1.63	25.3	H14	H14, Me17	H14
17	1.68	17.5	H14	H14, Me16	
18	1.00	20.0	H3	H3	H3, H5 α
19	1.21	23.3	H7	H7	H7, Me20'
20	1.84	11.5			H1
1'		144.7		H2 α β '	
2 α '	1.82	42.2	H2 β ', H3'	Me18'	H2 β ', Me18'
2 β '	2.12		H2 α '		H2 α ', Me18'
3'	1.36	32.8	H2 α ', H4', Me18'	Me18'	H5 α β ', Me18'
4'	1.80	44.2	H3', H5 α '	H2 β ', Me18'	H5 β ', H6 β ', Me18'
5 α '	1.17	25.3	H4', H5 β ', H6 α '		H3', H7'
5 β '	2.00		H5 α ', H6 β '		H4', H6 β '
6 α '	2.09	31.0	H5 α ', H6 β ', H7'	Me19'	H7'
6 β '	1.12		H5 β ', H6 α ', H7'		H4', H5 β '
7'	2.82	29.3	H6 α β ', Me19'	Me19'	H5 β ', H6 α ', Me19'
8'		129.3		Me19'	
9'		191.2		C10'-OH	
10'		90.1		C10'-OH, Me20'	
11'		78.8		C10'-OH, Me20'	
12'		132.5		H2 α β , H14', Me20'	
13'		157.9		H5 β '	
14'	5.71	126.4	Me16', Me17'	Me16', Me17'	H7, Me16', Me20'
15'		129.4		Me16', Me17'	
16'	1.76	25.3	H14'	H14', Me17'	H7, H14'
17'	1.50	19.6	H14'	H14', Me16'	H7
18'	0.95	19.1	H3'		H2 α β ', H3', H4'
19'	1.22	22.0	H7'		H7'
20'	1.45	21.0			10'-OH, H14', Me19
10'-OH	4.70				Me20'

^a Spectra were recorded at 25°C. Chemical shift values are in ppm relative to TMS (0.00 ppm) or CDCl_3 (77.0 ppm) signals. ^b Protons correlated to carbon resonances in ^{13}C column. Parameters were optimized for $^2,3J = 6$ and 8 Hz.

Table S3. Charge distributions of *ortho*-benzoquinone 7.

Job type: Equilibrium Geometry
 Method: ω B97X-D Basis set: 6-31G*
 Energy: -927.944232 hartrees



Atom Label	Natural charge	Mulliken charge	Electrostatic charge
C1	+0.490	+0.358	+0.465
C2	+0.486	+0.362	+0.354
C3	-0.102	-0.010	-0.366
C4	-0.096	+0.013	+0.190
C5	+0.035	+0.052	-0.555
C6	+0.024	+0.066	+0.243
C7	-0.728	-0.583	-0.597
C8	-0.284	-0.195	-0.157
C9	-0.310	-0.240	+0.957
C10	-0.456	-0.319	-1.108
C11	-0.254	-0.108	+0.748
C12	-0.464	-0.327	-0.189
C13	-0.466	-0.316	-0.519
C14	-0.284	-0.173	+0.485
C15	-0.691	-0.509	-0.888
C16	-0.688	-0.505	-0.708
C17	-0.247	-0.188	-0.671
C18	+0.000	+0.153	+0.544
C19	-0.719	-0.561	-0.800
C20	-0.708	-0.554	-0.798
H1	+0.252	+0.184	+0.155
H2	+0.250	+0.156	-0.010
H3	+0.251	+0.183	+0.164

H4	+0.273	+0.214	+0.190
H5	+0.240	+0.165	+0.209
H6	+0.266	+0.171	+0.089
H7	+0.240	+0.158	+0.128
H8	+0.246	+0.162	+0.098
H9	+0.271	+0.172	-0.026
H10	+0.244	+0.173	+0.217
H11	+0.250	+0.166	+0.215
H12	+0.244	+0.164	+0.109
H13	+0.250	+0.161	+0.168
H14	+0.239	+0.171	+0.220
H15	+0.278	+0.184	+0.019
H16	+0.238	+0.164	+0.165
H17	+0.235	+0.163	+0.186
H18	+0.250	+0.164	+0.267
H19	+0.227	+0.157	+0.190
H20	+0.256	+0.195	+0.201
H21	+0.231	+0.150	+0.209
H22	+0.240	+0.176	+0.184
H23	+0.249	+0.178	+0.220
H24	+0.250	+0.180	+0.219
H25	+0.245	+0.175	+0.215
H26	+0.244	+0.176	+0.202
O1	-0.500	-0.440	-0.428
O2	-0.496	-0.439	-0.403

Table S4. HOMO-LUMO energies for the heterodiene **7** and heterodienophile **7a**.

Reactant	HOMO Energy (eV)	LUMO Energy (eV)
<i>ortho</i> -Quinone 7	−8.23	−1.06
Enol 7a	−7.28	−0.45

Table S5. Crystal and structure refinement data for 1

8

Table 1. Crystal data and structure refinement

Identification code	Ito-f4126
Empirical formula	C20 H27 O2.50 ✓
Formula weight	307.42 ✓
Temperature	173(2) K ✓
Wavelength	0.71073 Å ✓
Crystal system, space group	Monoclinic, P2~1~
Unit cell dimensions	a = 12.1072(6) Å alpha = 90 deg. b = 10.5771(6) Å beta = 95.6550(10) deg.
Volume	c = 27.4452(14) Å gamma = 90 deg. 3497.5(3) Å ³ ✓
Z, Calculated density	8, 1.168 Mg/m ³ ✓
Absorption coefficient	0.075 mm ⁻¹ ✓
F(000)	1336
Crystal size	0.50 x 0.25 x 0.05 mm ✓
Theta range for data collection	1.69 to 27.13 deg.
Limiting indices	-14<=h<=15, -13<=k<=13, -35<=l<=28
Reflections collected / unique	22255 / <u>8141</u> [R(int) = 0.0757] ✓
Completeness to theta = 27.13	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.98 and 0.85
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8141 / 1 / 848 ✓
Goodness-of-fit on F ²	0.847 ✓
Final R indices [I>2sigma(I)]	R1 = 0.0440, wR2 = 0.0689 ✓
R indices (all data)	R1 = 0.1356, wR2 = 0.0872
Absolute structure parameter	0(10)
Extinction coefficient	0.00038(10)
Largest diff. peak and hole	0.181 and -0.182 e.Å ⁻³

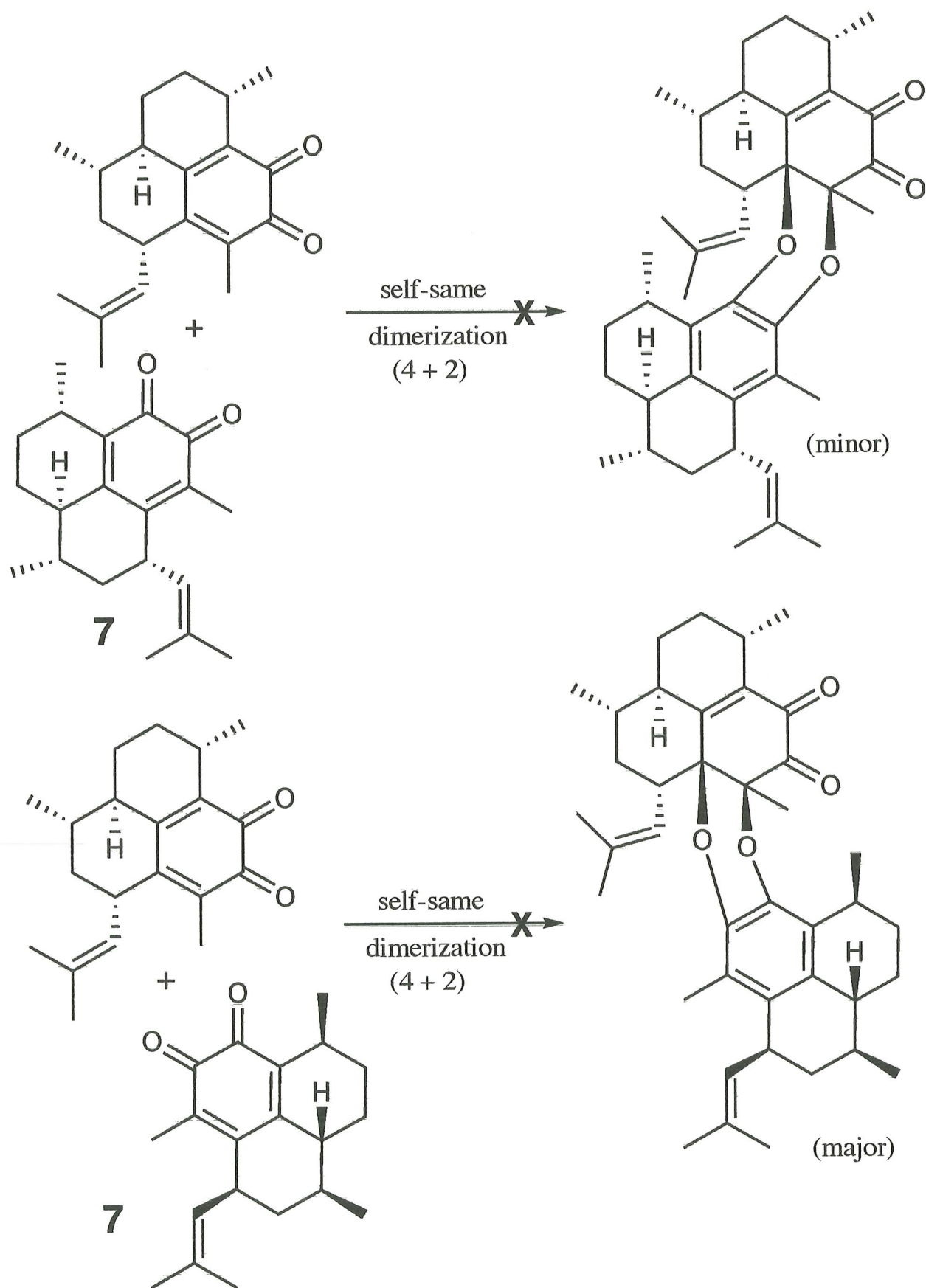
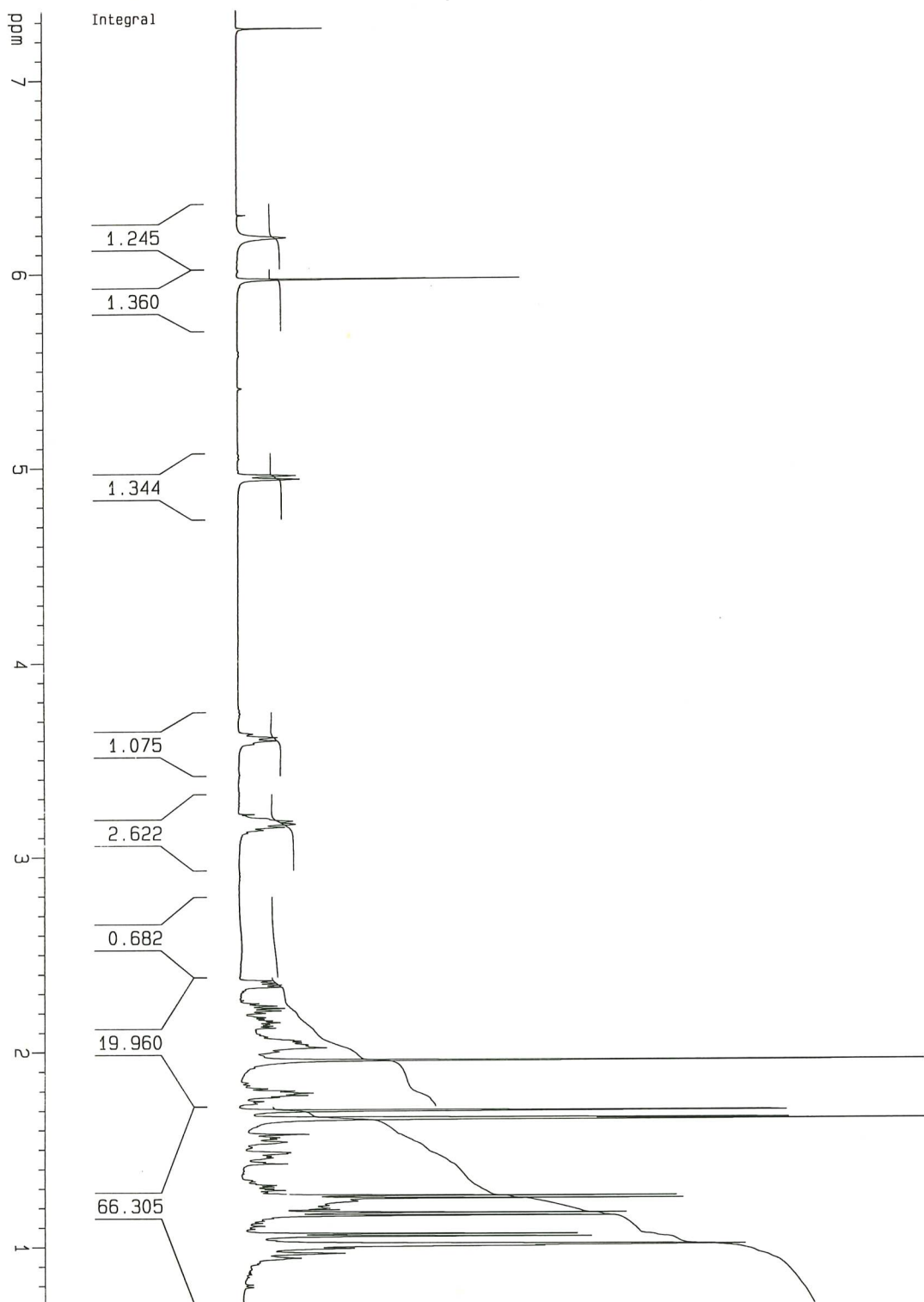


Figure S1. Predicted structures from self-same dimerization of *ortho*-benzoquinone **7** (not detected).

Bisditerpene A

Figure S2. ¹H NMR (500 MHz, CDCl₃) spectrum of 1

Current Data Parameters

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PROCNO	1

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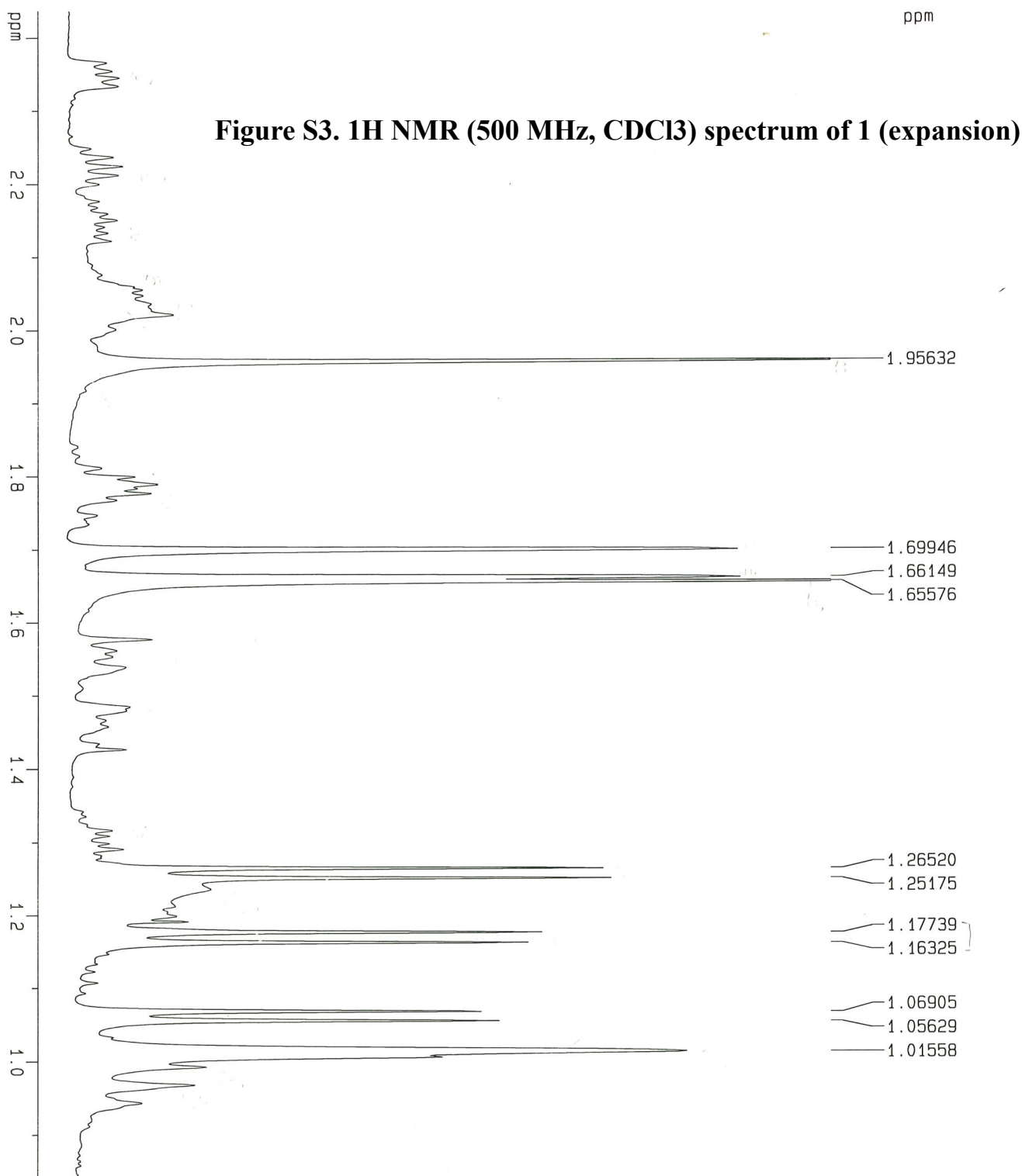
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SOLVENT	CDCl3
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DS	2
SWH	7246.377 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	203.2
DW	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	10.00 usec
SFO1	500.1328865 MHz
NUC1	¹ H
PL1	0.00 dB

F2 - Processing parameters

SI	16384
SF	500.1300135 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	7.357 ppm
F1	3679.48 Hz
F2P	0.707 ppm
F2	353.72 Hz
PPMCM	0.33249 ppm/cm
HZCM	166.28813 Hz/cm



Current Data Parameters

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PROCNO	1

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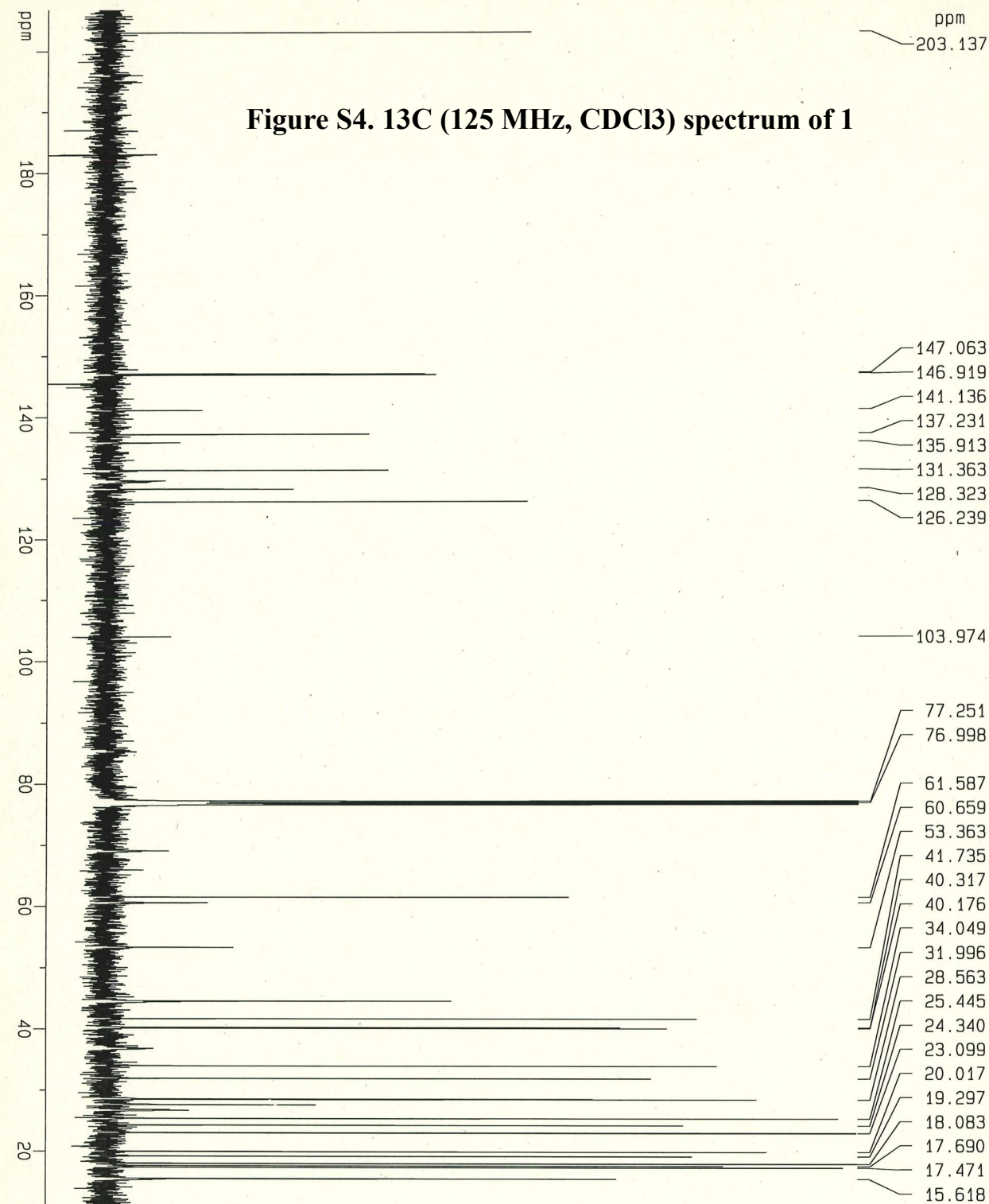
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PROBHD	5 mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	CDCl_3
NS	32
DS	2
SWH	7246.377 Hz
FIDRES	0.22142 Hz
AQ	2.2610421 sec
RG	203.2
DM	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	10.00 usec
SFO1	500.1328885 MHz
NUC1	^1H
PL1	0.00 dB

F2 - Processing parameters

SI	16384
SF	500.1300135 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	500.00

1D NMR plot parameters

CX	20.00 cm
F1P	2.437 ppm
F1	1218.89 Hz
F2P	0.839 ppm
F2	419.66 Hz
PPMCM	0.07990 ppm/cm
HZCM	39.96180 Hz/cm

Figure S4. ¹³C (125 MHz, CDCl₃) spectrum of 1

Pend-c10 k2

Current Data Parameters

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PROCNO	1

F2 - Acquisition Parameters

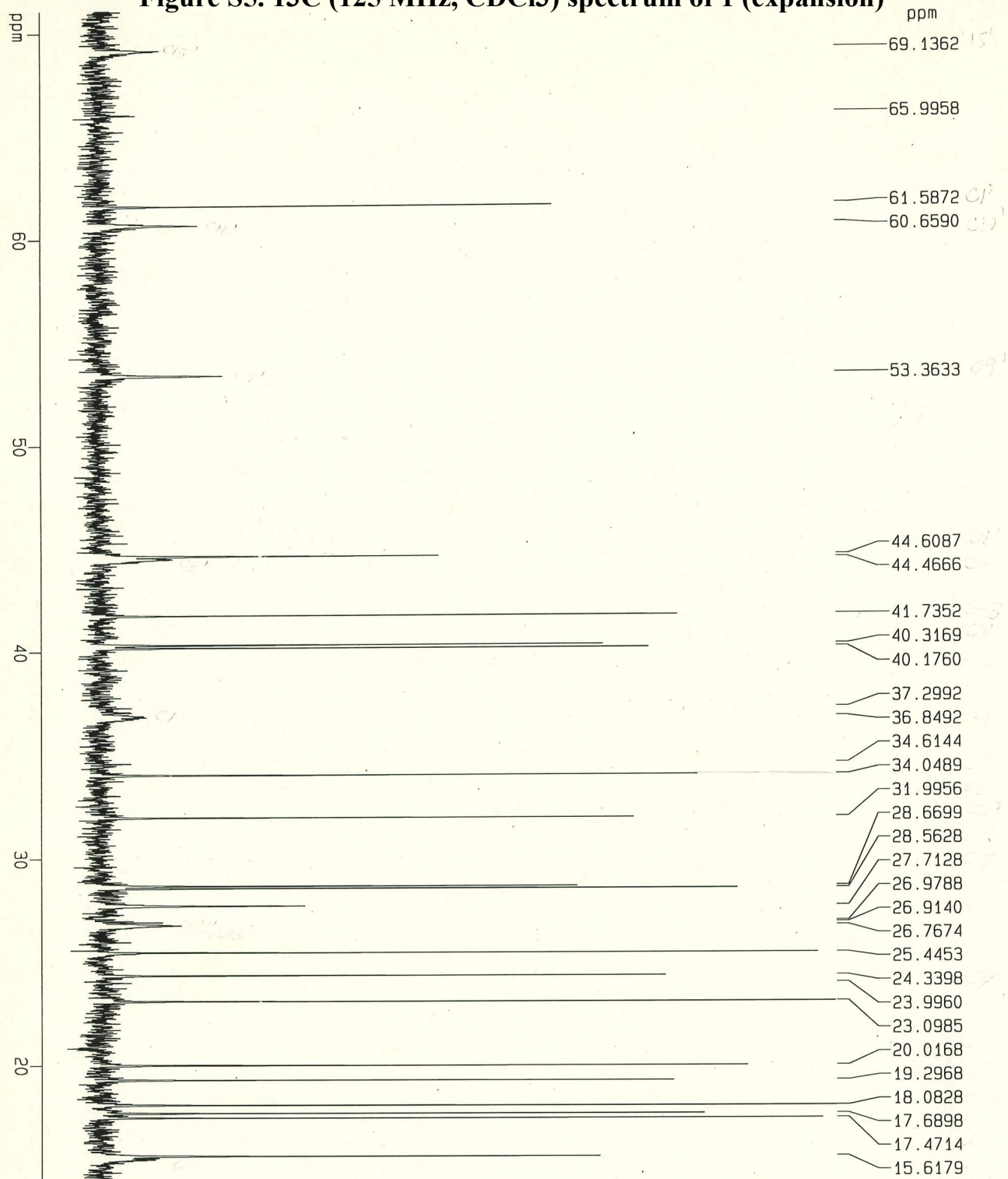
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PL13	22.00 dB
D1	1.00000000 sec
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PCPD2	102.00 usec
SFO2	500.132005 MHz
NUC2	¹ H
PL2	0.00 dB
PL12	22.00 dB
P1	10.00 usec
SFO1	125.770995 MHz
NUC1	¹³ C
PL1	2.00 dB
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	125.7577906 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

10 NMR plot parameters

CX	20.00 cm
F1P	206.828 ppm
F1	26010.20 Hz
F2P	11.270 ppm
F2	1417.25 Hz
PPMCM	9.77790 ppm/cm
HZCM	1229.64709 Hz/cm

Figure S5. ^{13}C (125 MHz, CDCl_3) spectrum of 1 (expansion)

Pend-c10 k2

Current Data Parameters

NAME	pendc10k2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

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PROBHD	5 mm Multinu
PULPROG	zgpg30
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SOLVENT	CDCl_3
NS	4703
DS	2
SWH	30303.031 Hz
FIDRES	0.462388 Hz
AQ	1.0813940 sec
RG	8192
DM	16.500 usec
DE	6.00 usec
TE	300.0 K
D12	0.00002000 sec
PL13	22.00 dB
D1	1.00000000 sec
CPDPRG2	waltz16
PCPD2	102.00 usec
SFO2	500.132005 MHz
NUC2	^1H
PL2	0.00 dB
PL12	22.00 dB
P1	10.00 usec
SFO1	125.7709955 MHz
NUC1	^{13}C
PL1	2.00 dB
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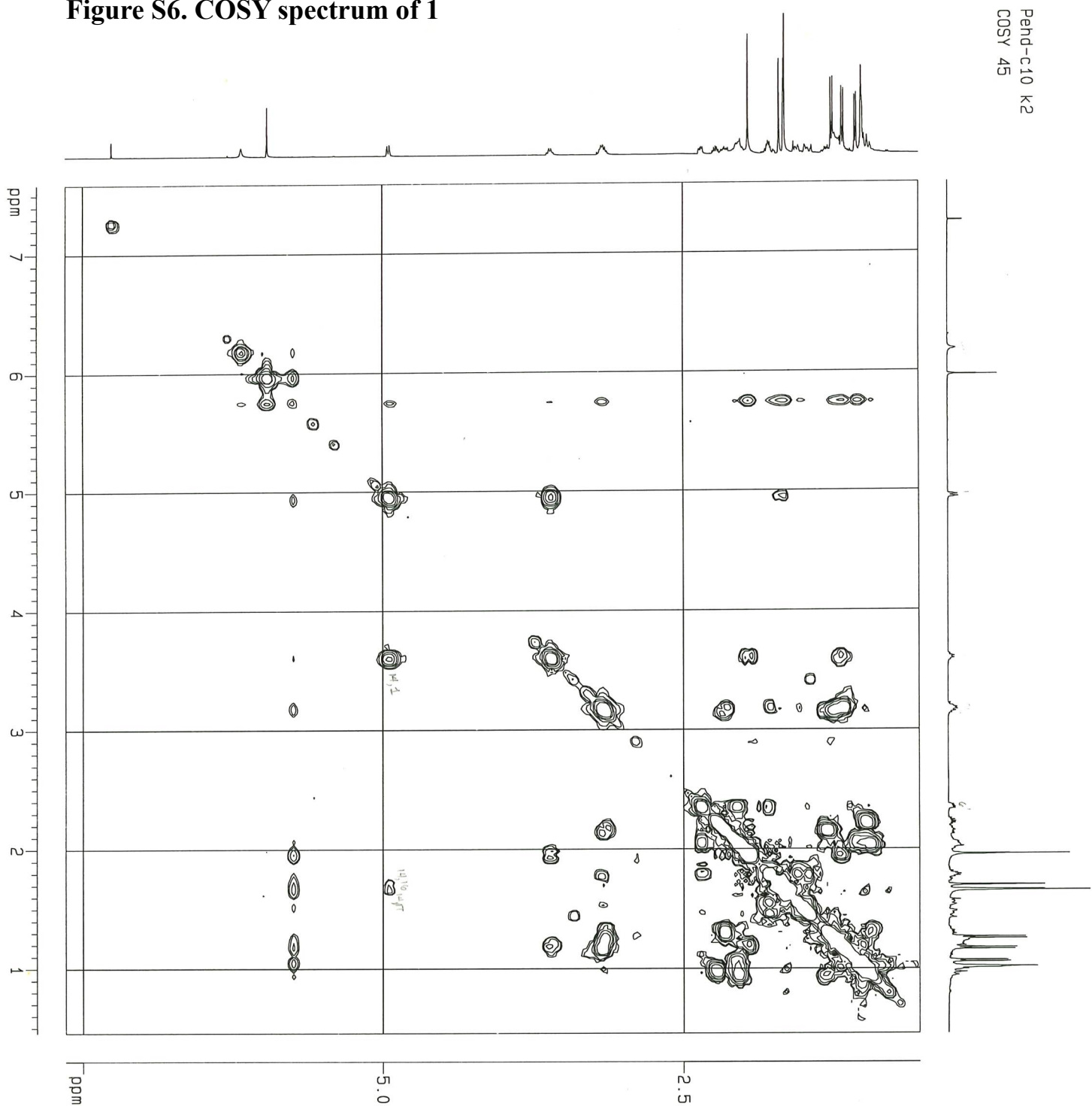
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LB	1.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	71.105 ppm
F1	8941.96 Hz
F2P	14.371 ppm
F2	1807.25 Hz
PPMCK	2.83669 ppm/cm
HZCM	356.73535 Hz/cm

Pend-c10 k2
COSY 45



Current Data Parameters

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PROCNO	1

F2 - Acquisition Parameters

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PROBHD	5 mm MultiNU
PULPROG	cosy45
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SMH	7246.377 Hz
FIDRES	7.076540 Hz
AQ	0.0707060 sec
RG	101.6
DW	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	-1.00000000 sec
P1	10.00 usec
SFO1	500.132885 MHz
NUC1	1H
PL1	0.00 dB
DO	0.00000300 sec
INO	0.00013800 sec

F1 - Acquisition Parameters

ND0	1
TD	256
SFO1	500.1328 MHz
FIDRES	28.306160 Hz
SM	14.489 ppm

F2 - Processing parameters

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GB	0
PC	1.00

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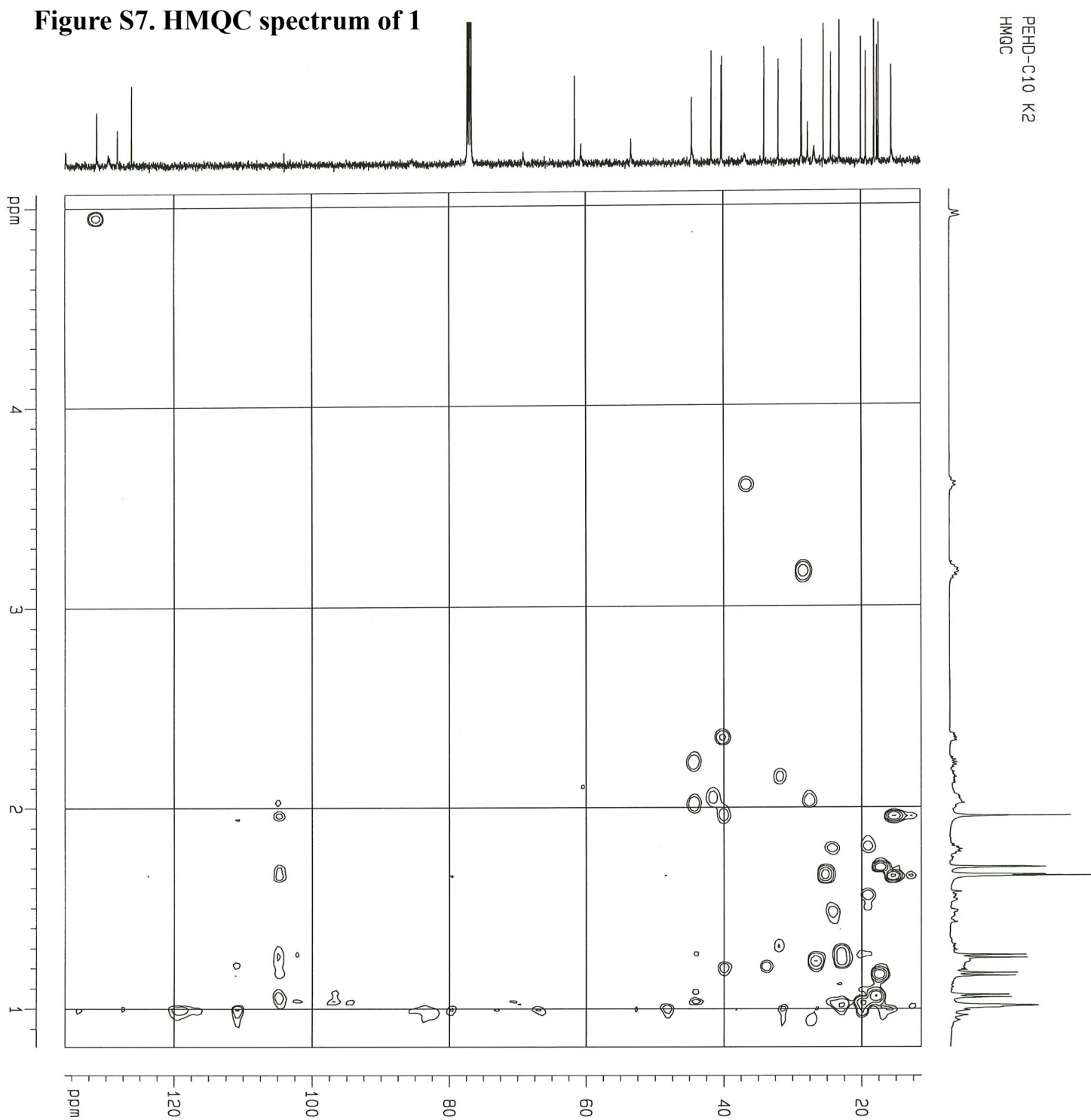
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WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PL0	7.588 ppm
F2L0	3794.99 Hz
F2PH1	0.457 ppm
F2H1	228.41 Hz
F1PL0	7.645 ppm
F1L0	3823.29 Hz
F1PH1	0.542 ppm
F1H1	270.87 Hz
F2PMCM	0.47542 ppm/cm
F2HZCM	237.77173 Hz/cm
F1PMCM	0.47353 ppm/cm
F1HZCM	236.82820 Hz/cm

Figure S6. COSY spectrum of 1

PEHD-C10 K2
HMQC



Current Data Parameters

NAME	EXPNO	PROCNO
pehd-c10k2	15	1

F2 - Acquisition Parameters

Date_	Time
20020711	16.40

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG invtdp
TD 1024
SOLVENT CDCl3
NS 8
DS 32
SMH 7246.377 Hz
FIDRES 7.075540 Hz
AQ 0.0707060 sec
RG 574.7
DM 69.000 usec
DE 5.00 usec
TE 300.0 K
D1 1.2500000 sec
P1 10.50 usec
SFO1 500.132885 MHz
NUC1 1H
P11 0.00 dB
D2 0.0034600 sec
P12 2.00 dB
P2 21.00 usec
P4 17.00 usec
SFO2 125.770985 MHz
NUC2 13C
D7 0.4000001 sec
P3 8.50 usec
D0 0.0000300 sec
P112 20.00 dB
COPR02 9999
PCPR02 66.00 usec
IN0 0.00000825 sec

F1 - Acquisition Parameters

NO	TD
4	256

SFO1 125.771 MHz
FIDRES 118.371216 Hz
SM 240.938 ppm

F2 - Processing parameters

SI	SF
1024	500.130135 MHz

WDW 0SINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.00

F1 - Processing parameters

SI	MC2
142	125.7577908 MHz

WDW SINE
SSB 2
LB 0.00 Hz
GB 0

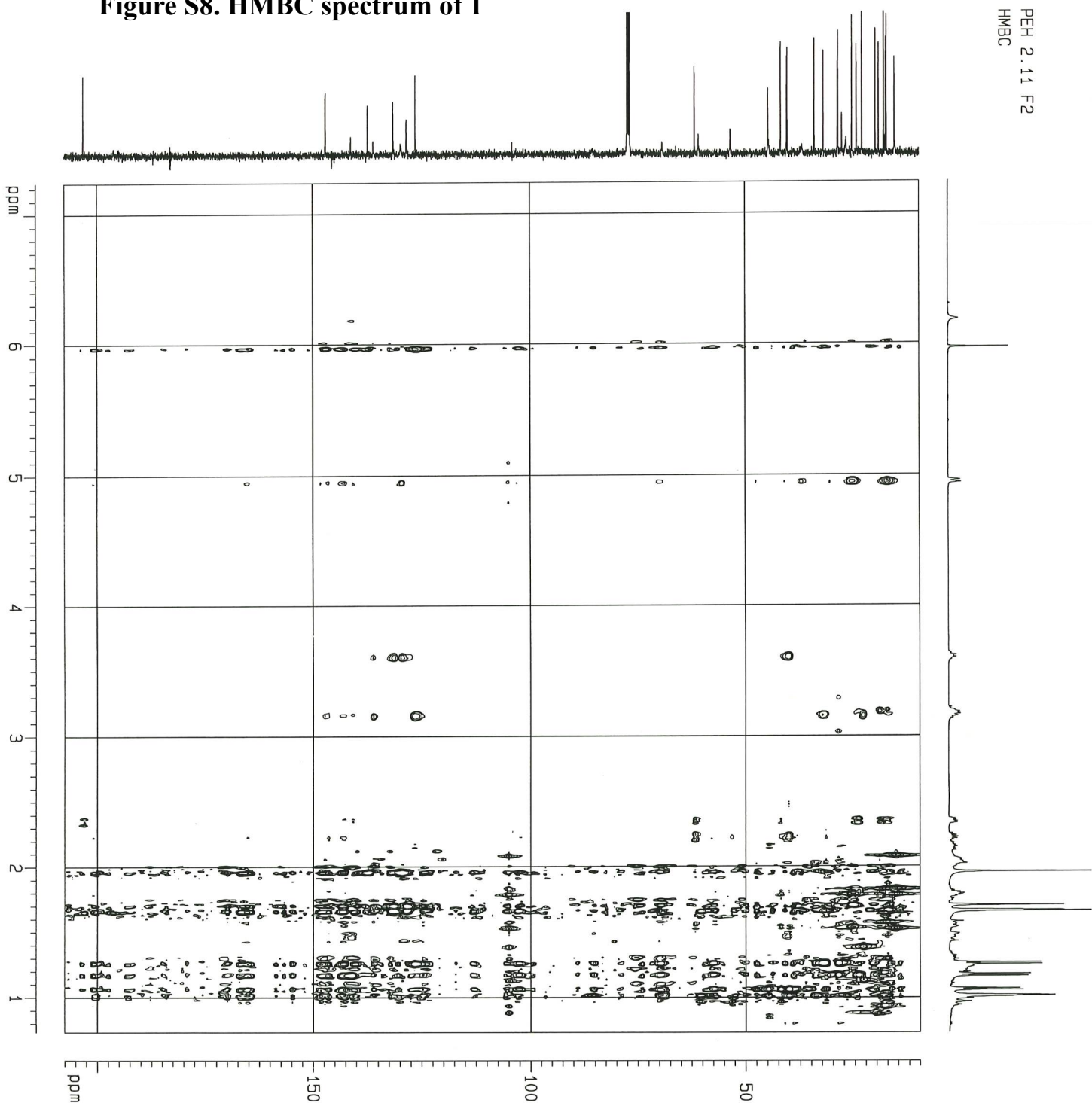
2D NMR plot parameters

CK2	CK1
15.00 cm	15.00 cm

F2H0 5.069 ppm
F2L0 2555.36 Hz
F2H1 0.810 ppm
F2L1 465.32 Hz
F2H0 156.065 ppm
F2L0 17111.19 Hz
F2H1 11.347 ppm
F2H0 0.26593 ppm/cm
F2L0 142.00256 Hz/cm
F2H0 8.31449 ppm/cm
F2L0 1045.61230 Hz/cm

PEH 2.11 F2
HMBC

Figure S8. HMBC spectrum of 1



Current Data Parameters
NAME peridolK2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020710
Time 22:46
INSTRUM spect
PROBHD 5 mm MUlti
PULPROG invalid
TD 4096
SOLVENT CDCl3
NS 72
DS 32
SMH 7246.377 Hz
FIDRES 1.769135 Hz
AQ 0.2826740 sec
RG 256
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.5000000 sec
P1 10.00 usec
SFO1 500.1328865 MHz
NUC1 1H
PL1 0.00 dB
D2 0.0030000 sec
P2 0.00 usec
SFO2 125.7709353 MHz
NUC2 13C
PL2 2.00 dB
D6 0.0500000 sec
D0 0.0000300 sec
P2 20.00 usec
INO 0.00001650 sec

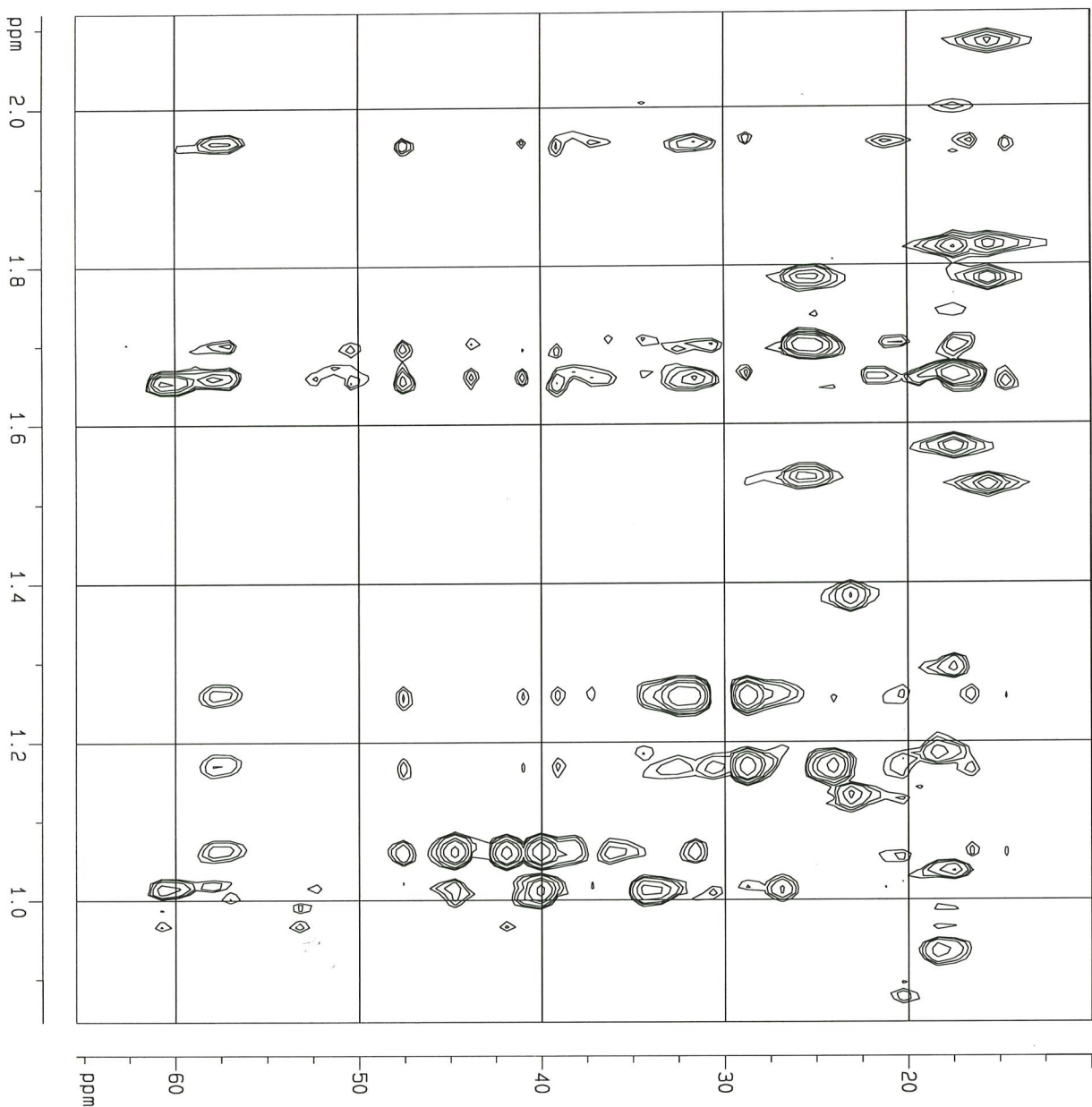
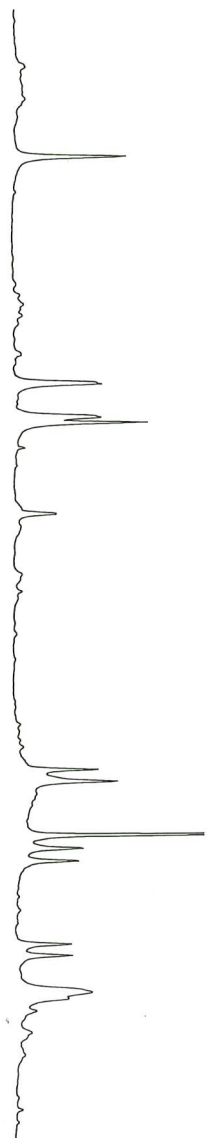
F1 - Acquisition Parameters
N00 2
TD 256
SFO1 125.771 MHz
FIDRES 118.371216 Hz
SM 240.938 ppm

F2 - Processing parameters
SI 2048
SF 500.1300135 MHz
WDW 0SINE
SSB 4
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 256
MC2 0F
SF 125.7577906 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 7.241 ppm
F2LO 3621.61 Hz
F2PH 0.733 ppm
F2H1 366.40 Hz
F1PLO 207.601 ppm
F1LO 26107.40 Hz
F1PH 9.935 ppm
F1H1 1249.45 Hz
F2PPOCH 0.43391 ppm/cm
F2H2CM 217.01389 Hz/cm
F1PPOCH 13.17769 ppm/cm
F1H2CM 1657.19690 Hz/cm

PEH 2.11 F2
HMBC



Current Data Parameters
NAME peh2c10k2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020710
Time 22:46
INSTRUM spect
PROBHD 5 mm Multino
PULPROG invalid
TD 4096
SOLVENT CDCl3
NS 72
DS 32
SHH 7246.377 Hz
FIDRES 1.769135 Hz
AQ 0.2826740 sec
RG 256
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.5000000 sec
P1 10.00 usec
SF01 500.132885 MHz
NUC1 1H
PL1 0.00 dB
D2 0.0030000 sec
P3 10.00 usec
SF02 125.770955 MHz
NUC2 13C
PL2 2.00 dB
D6 0.0500000 sec
D0 0.0000300 sec
P2 20.00 usec
INO 0.00001650 sec

F1 - Acquisition Parameters
NO 2
TD 256
SF01 125.771 MHz
FIDRES 118.371216 Hz
SM 240.938 ppm

F2 - Processing parameters
SI 2048
SF 500.130135 MHz
WDW GSSINE
SSB 4
LB 0.00 Hz
GB 0
PC 1.00

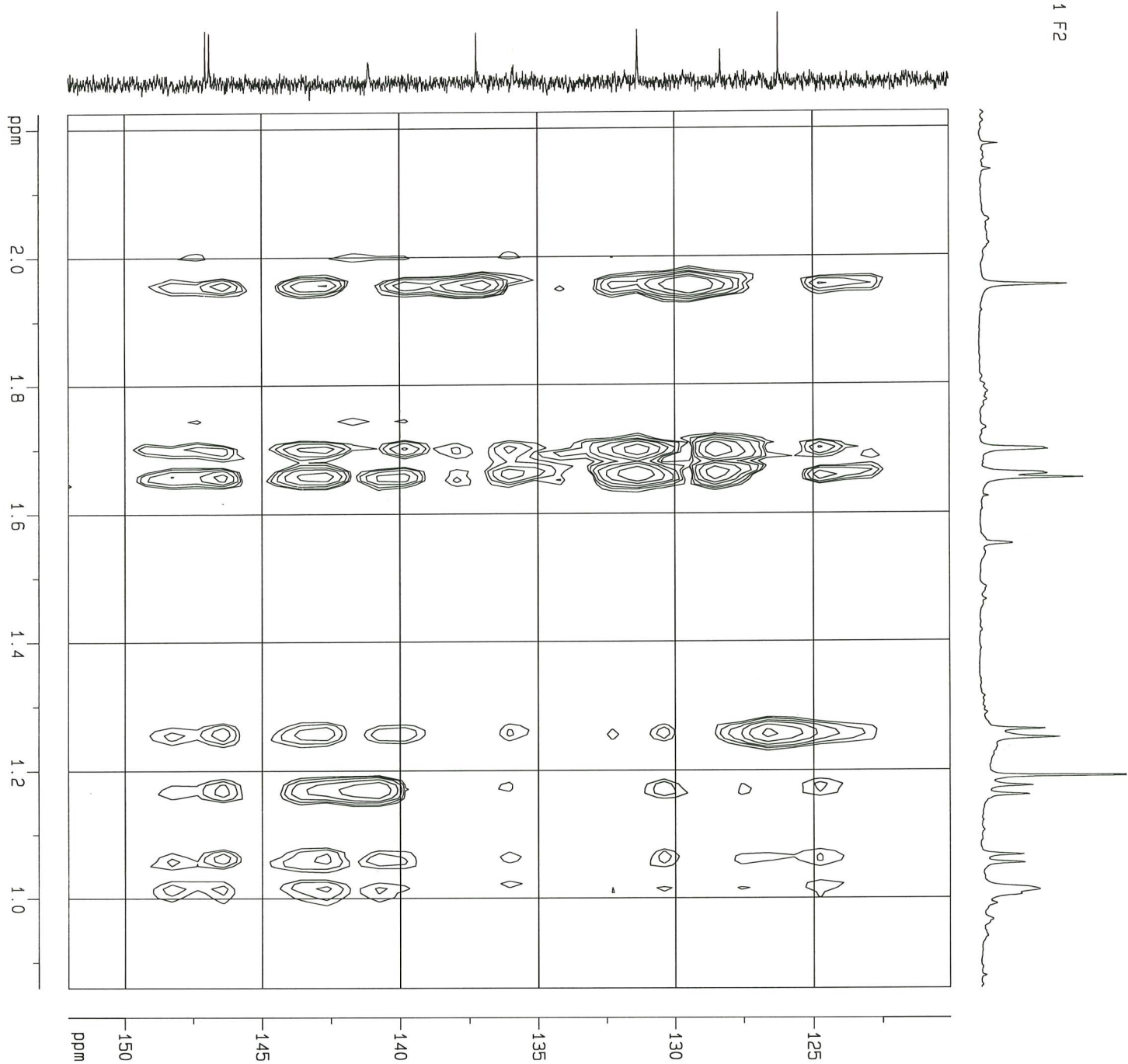
F1 - Processing parameters
SI 256
MC2 OF
SF 125.7577906 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2FLO 2.119 ppm
F2LO 1059.90 Hz
F2PHI 0.846 ppm
F2H1 423.01 Hz
F1FLO 65.470 ppm
F1LO 8233.35 Hz
F1PHI 9.935 ppm
F1H1 1249.45 Hz
F2PHICM 0.08460 ppm/cm
F2H2CM 42.45924 Hz/cm
F1PHICM 3.70230 ppm/cm
F1H2CM 465.59344 Hz/cm

Figure S9. HMBC spectrum of 1 (expansion)

PEH 2.11 F2
HMBC

Figure S10. HMBC spectrum of 1 (expansion)



Current Data Parameters
NAME percd10k2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020710
Time 22.46
INSTRUM spect
PROBHD 5 mm Multino
PULPROG invalidprog
TD 4096
SOLVENT CDCl3
NS 72
DS 32
SHH 7246.377 Hz
FIDRES 1.769135 Hz
AQ 0.2826740 sec
RG 256
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.5000000 sec
P1 10.00 usec
SFO1 500.1328885 MHz
NUC1 1H
PL1 0.00 dB
D2 0.0030000 sec
P3 10.00 usec
SFO2 125.7709553 MHz
NUC2 13C
PL2 2.00 dB
D6 0.0500000 sec
D0 0.0000300 sec
P2 20.00 usec
INO 0.00001650 sec

F1 - Acquisition Parameters
NDO 2
TD 256
SFO1 125.771 MHz
FIDRES 118.371216 Hz
SM 240.938 ppm

F2 - Processing parameters
SI 2048
SF 500.130135 MHz
WDW 63LINE
SSB 4
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 256
MC2 OF
SF 125.757906 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CY1 15.00 cm
F2PL0 2.225 ppm
F2PL1 1112.58 Hz
F2PH1 0.660 ppm
F2PL0 430.09 Hz
F2PL1 152.065 ppm
F2LO 19123.50 Hz
F2PH1 120.063 ppm
F2HI 15098.88 Hz
F2PRMCM 0.09103 ppm/cm
F2HZCM 45.52574 Hz/cm
F1PRMCM 2.13353 ppm/cm
F1HZCM 268.30811 Hz/cm

Figure S11. NOESY spectrum of 1

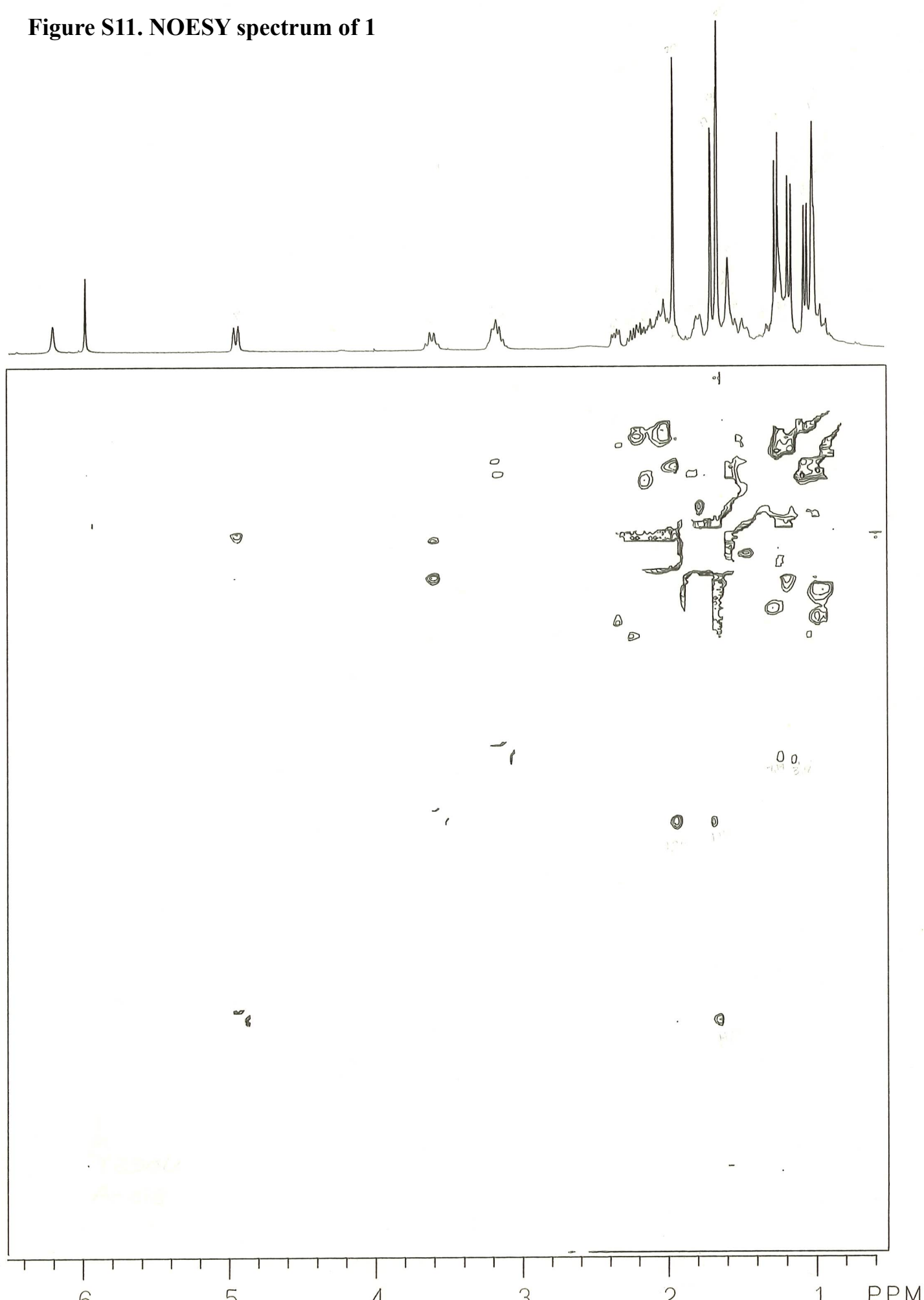
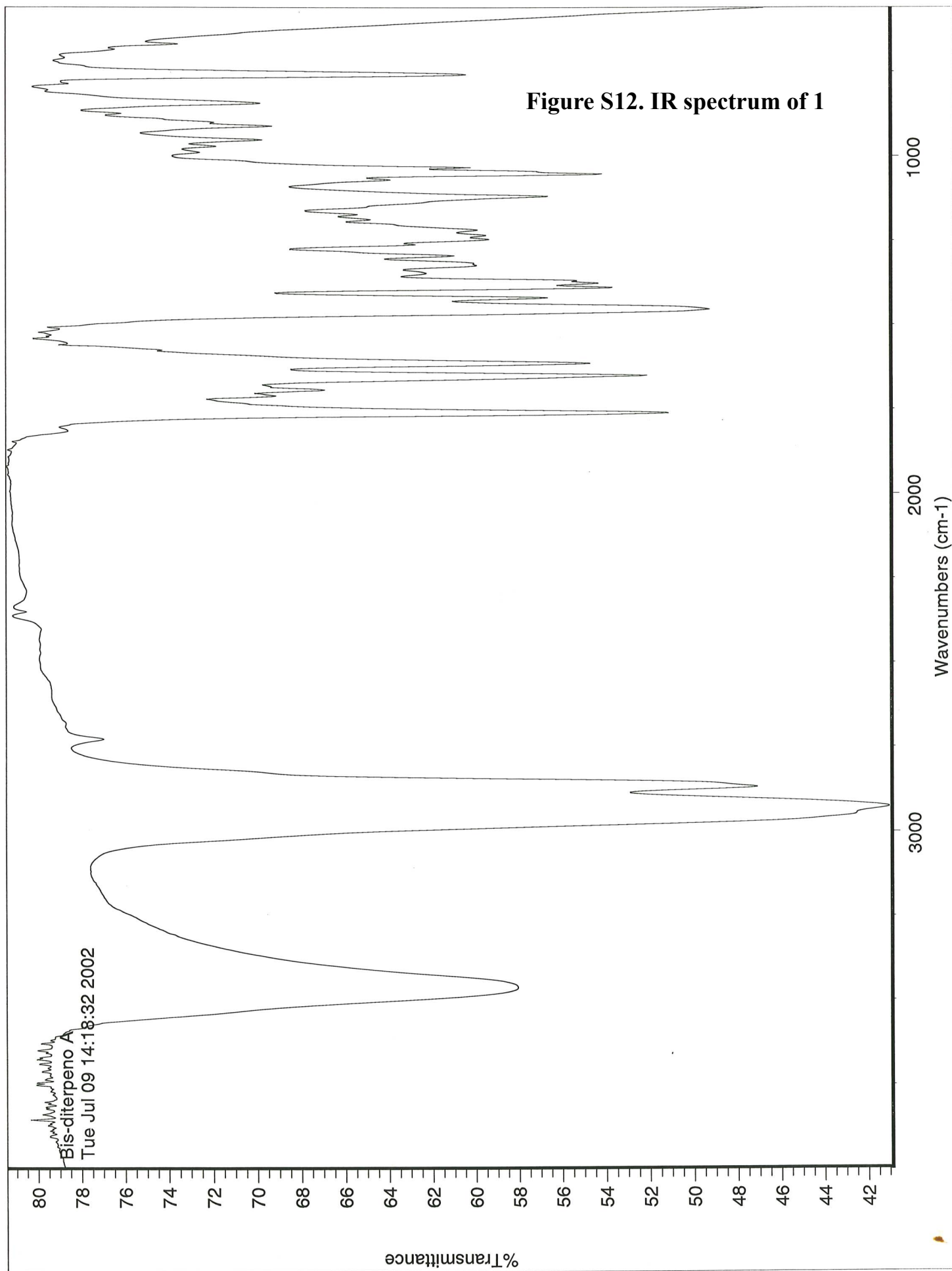
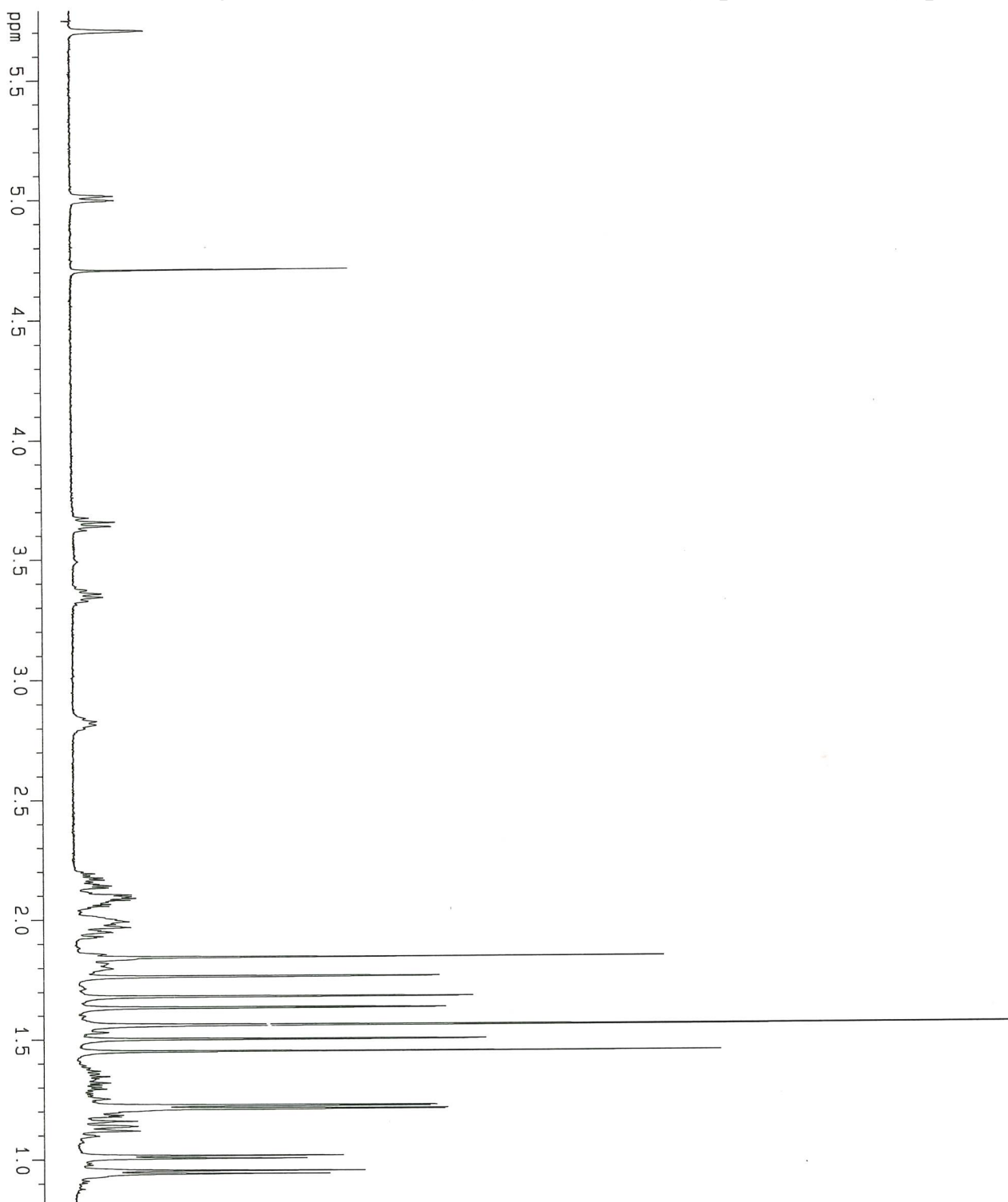


Figure S12. IR spectrum of 1



PEH 2 R2 M2
bis-diterpene

Figure S13. ¹H NMR (500 MHz, CDCl₃) spectrum of 2 (expansion)

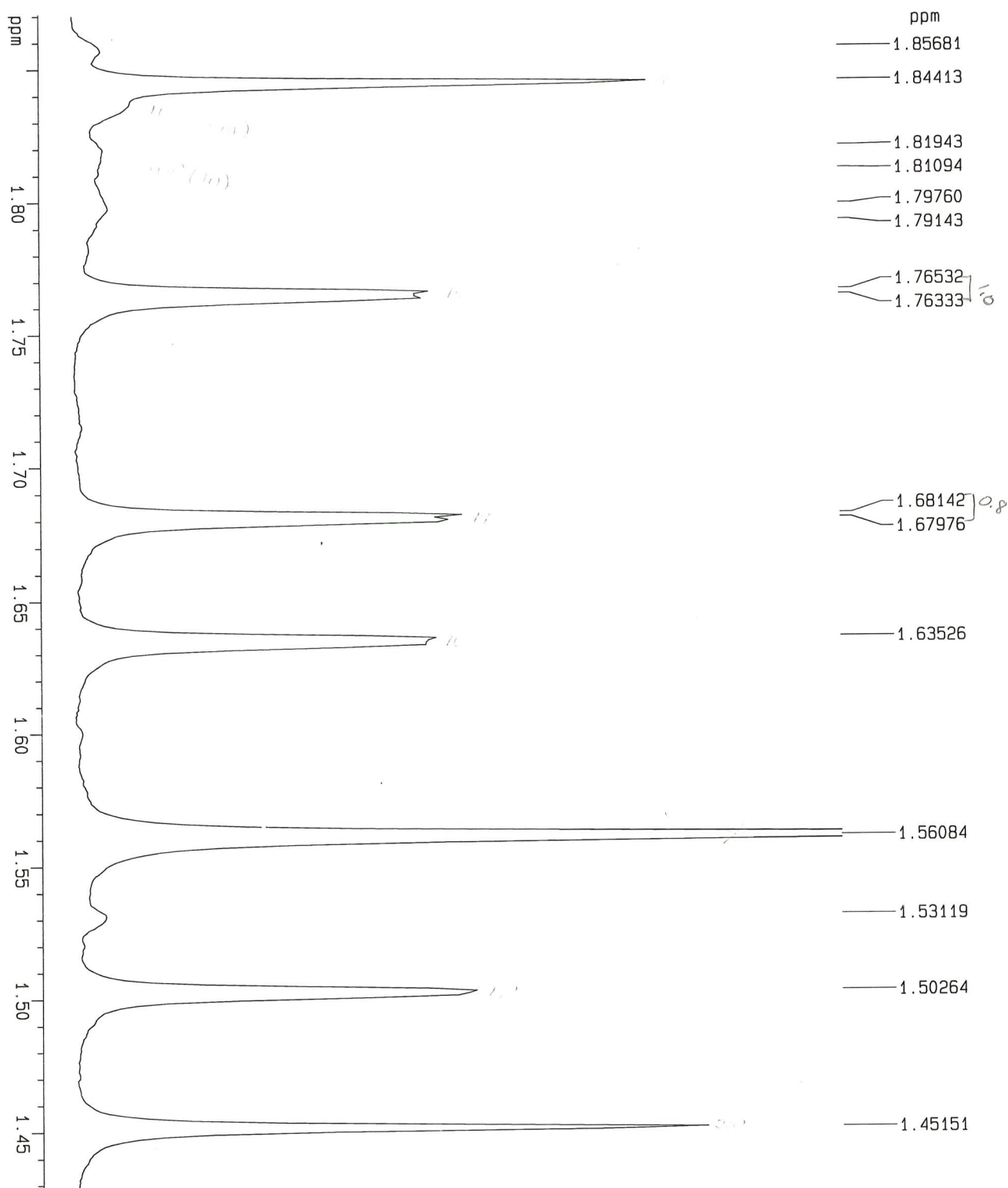


Current Data Parameters
NAME peh2r2m2y
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020220
Time 9.36
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
FIDRES 0.22142 Hz
AQ 2.2610421 sec
RG 724.1
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 10.00 usec
SFO1 500.1328885 MHz
NUC1 ¹H
PL1 0.00 dB

F2 - Processing parameters
SI 16384
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 5.792 ppm
F1 2896.57 Hz
F2P 0.809 ppm
F2 404.49 Hz
PPMCM 0.24914 ppm/cm
HZCM 124.60435 Hz/cm

Figure S14. ¹H NMR (500 MHz, CDCl₃) spectrum of 2 (expansion)

PEH 2 R2 M2
bis-diterpene

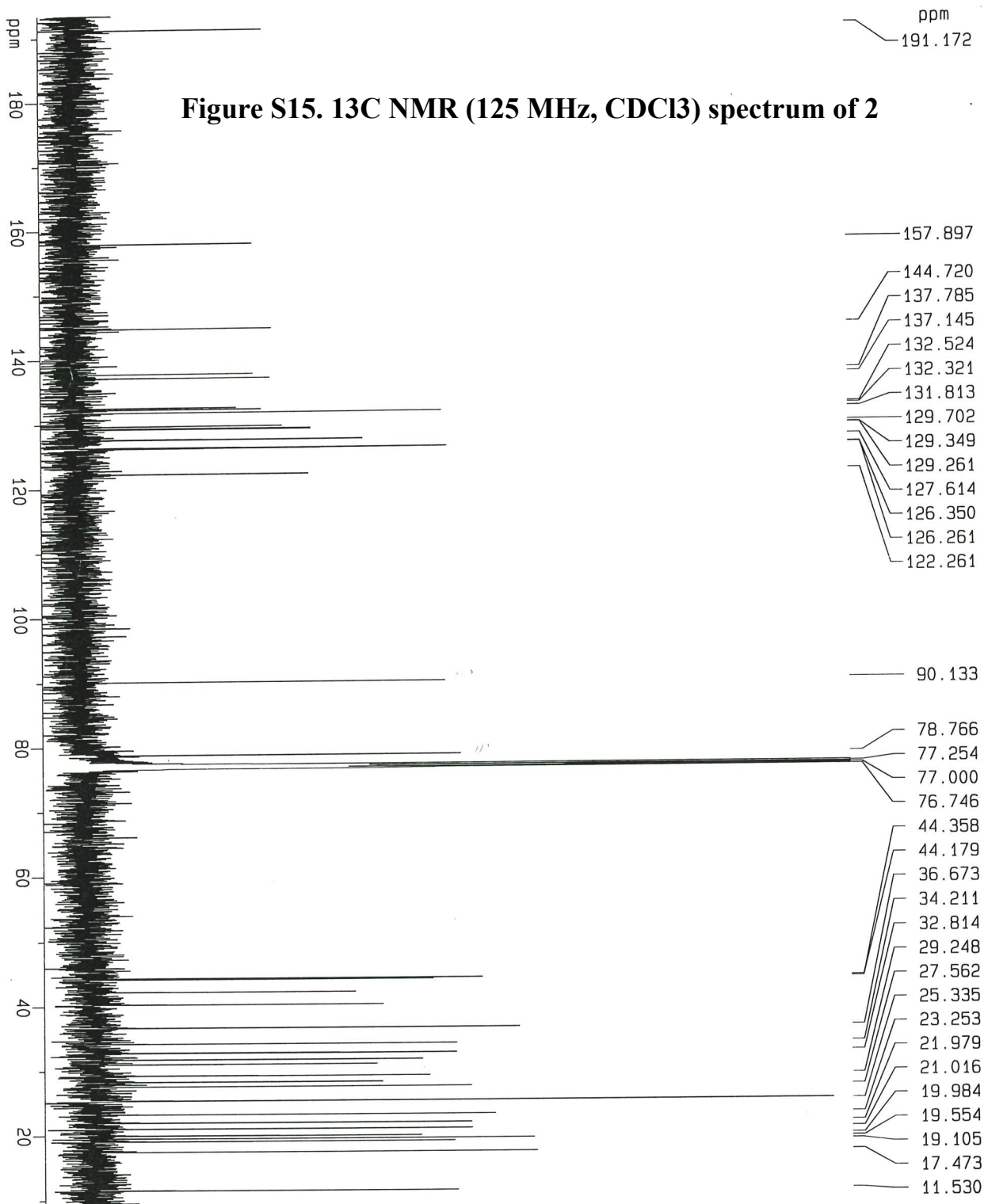
Current Data Parameters
NAME pen2r2m2y
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020220
Time 9.36
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SMH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 724.1
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 10.00 usec
SF01 500.1328885 MHz
NUC1 ¹H
PL1 0.00 dB

F2 - Processing parameters
SI 16384
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC * 0.00

1D NMR plot parameters
CX 20.00 cm
F1P 1.871 ppm
F1 935.64 Hz
F2P 1.428 ppm
F2 714.37 Hz
PPMCM 0.02212 ppm/cm
HZCM 11.06349 Hz/cm

PEH 2 R2 M2 Y



Current Data Parameters

NAME	peh2r2m2y
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

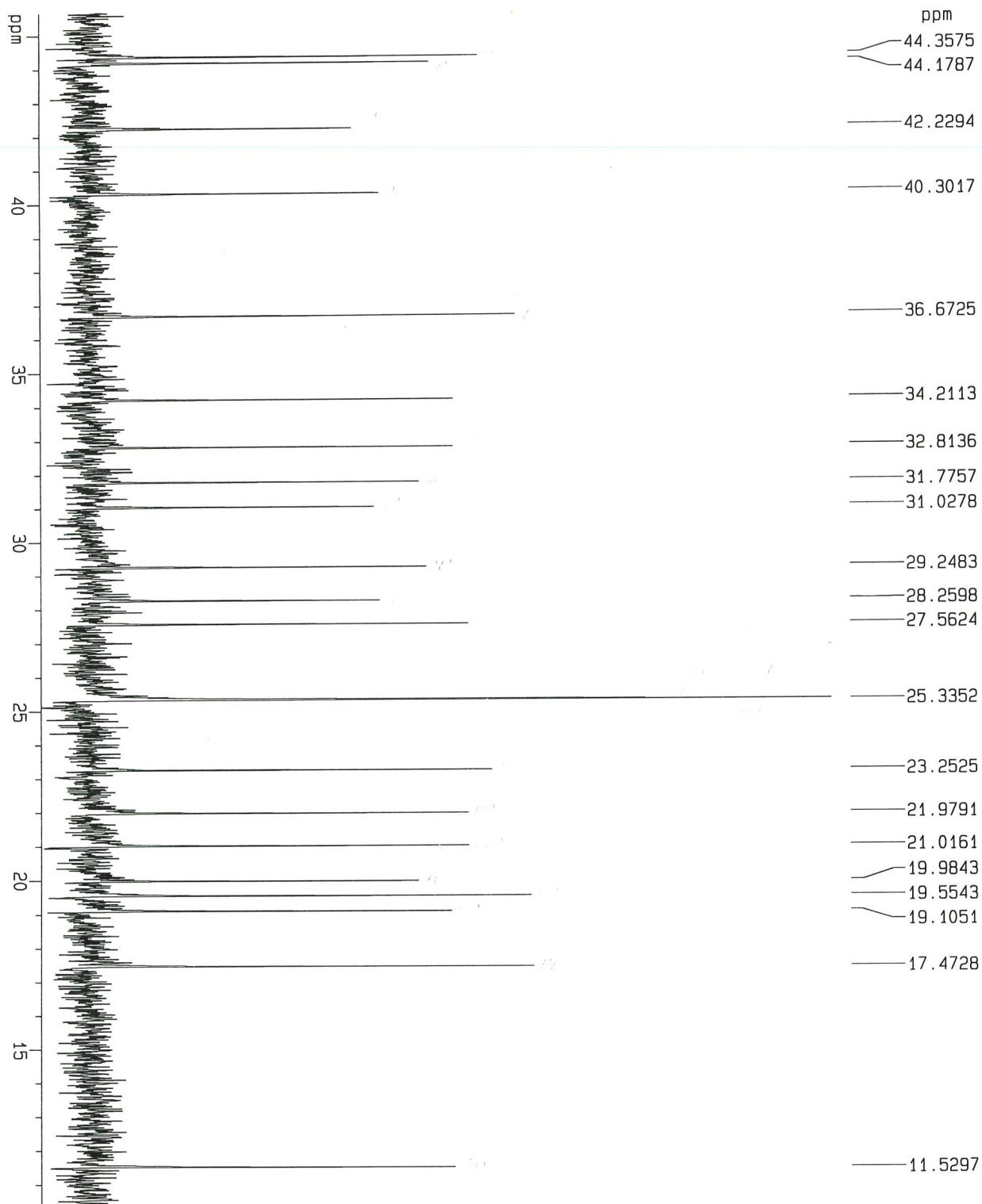
Date_	20020221
Time	16.44
INSTRUM	5 mm Multinu
PROBHD	z9p930
PULPROG	65536
TD	CDCl3
SOLVENT	25204
NS	2
DS	30303.031 Hz
SMH	0.462388 Hz
FIDRES	1.0813940 sec
RG	9195.2
DW	16.500 usec
DE	6.00 usec
TE	300.0 K
D12	0.0000200 sec
PL13	22.00 dB
D1	1.00000000 sec
CPDPRG2	waitz16
PCPD2	102.00 usec
SFO2	500.132005 MHz
NUC2	^1H
PL2	0.00 dB
PL12	22.00 dB
P1	7.87 usec
SFO1	125.7709955 MHz
NUC1	^{13}C
PL1	2.00 dB
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	125.7577897 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	193.428 ppm
F1	24325.07 Hz
F2P	9.305 ppm
F2	1170.14 Hz
PRMCM	9.20616 ppm/cm
HZCM	1157.74695 Hz/cm

Figure S16. ¹³C NMR (125 MHz, CDCl₃) spectrum of 2 (expansion)

PEH 2 R2 M2 Y

Current Data Parameters

NAME	pen2r2m2y
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20020221
Time	16.44
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	25204
DS	2
SMH	30303.031 Hz
FIDRES	0.462388 Hz
AQ	1.0813940 sec
RG	9195.2
DW	16.500 usec
DE	6.00 usec
TE	300.0 K
D12	0.00002000 sec
PL13	22.00 dB
D1	1.00000000 sec
CPDPRG2	waltz16
PCPD2	102.00 usec
SFO2	500.1320005 MHz
NUC2	¹ H
PL2	0.00 dB
PL12	22.00 dB
P1	7.87 usec
SFO1	125.7709955 MHz
NUC1	¹³ C
PL1	2.00 dB
D11	0.03000000 sec

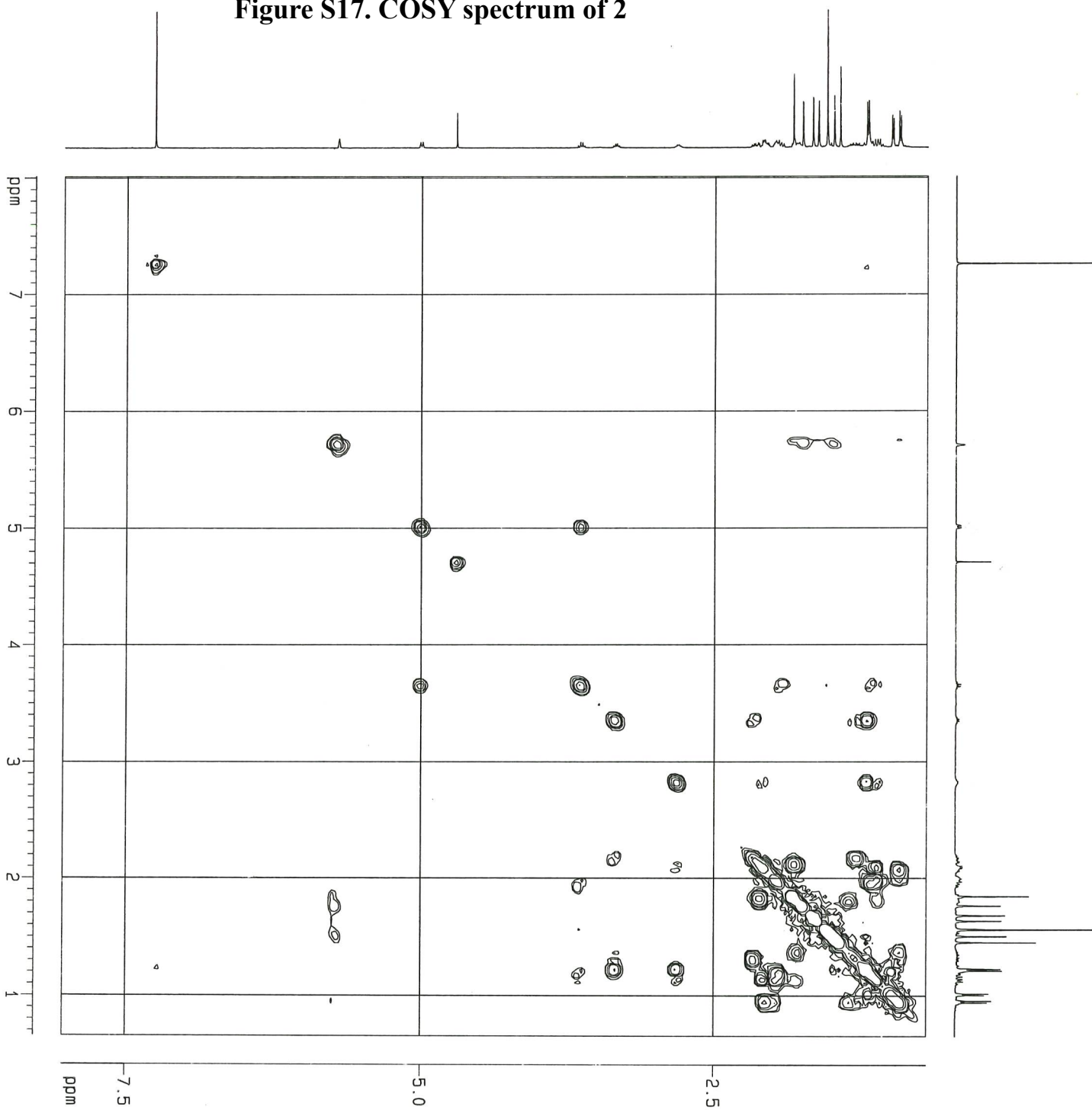
F2 - Processing parameters

SI	32768
SF	125.7577897 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	45.684 ppm
F1	5745.10 Hz
F2P	10.418 ppm
F2	1310.19 Hz
PPMCM	1.76328 ppm/cm
HZCM	221.74588 Hz/cm

Figure S17. COSY spectrum of 2



Current Data Parameters

NAME	perchancy
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20020220
Time	14.10
INSTRUM	5 mm Multinu
PROBHD	cosy/45
PULPROG	1024
TD	1024
SOLVENT	CDCl3
NS	16
DS	16
SMU	7246.377 Hz
FIDRES	7.076540 Hz
AQ	0.0707060 sec
RG	512
DW	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
P1	10.00 usec
SFO1	500.132885 MHz
NUC1	1H
PL1	0.00 dB
DO	0.00000300 sec
INO	0.00013800 sec

F1 - Acquisition Parameters

NDO	1
TD	256
SFO1	500.1329 MHz
FIDRES	28.306160 Hz
SW	14.489 ppm

F2 - Processing parameters

SI	512
SF	500.130135 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	0.00

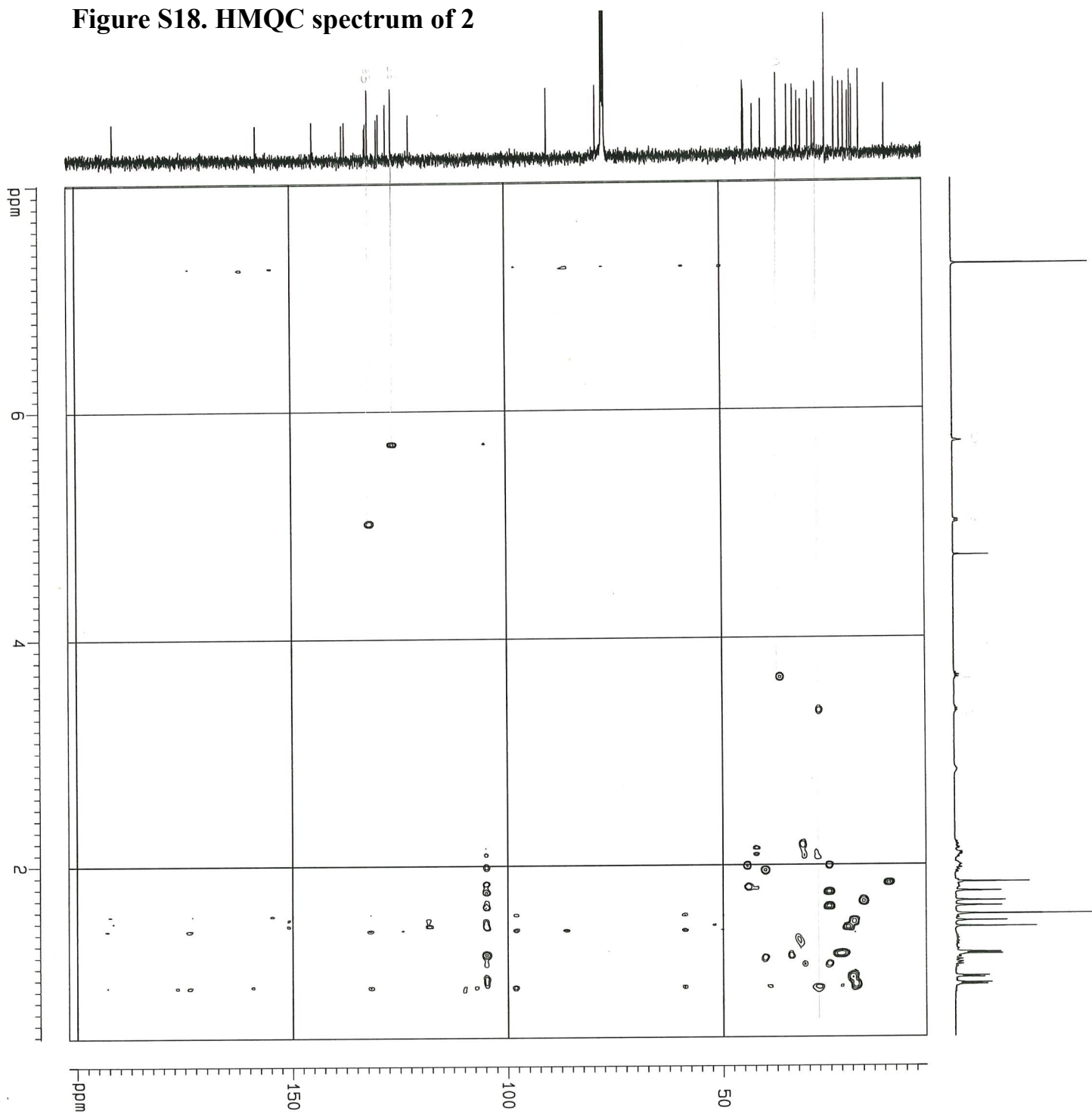
F1 - Processing parameters

SI	512
MC2	OF
SF	500.1300135 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PULO	8.012 ppm
F2LO	4007.28 Hz
F2PHI	0.655 ppm
F2HI	327.48 Hz
F1PULO	8.041 ppm
F1LO	4021.44 Hz
F1PHI	0.711 ppm
F1HI	355.79 Hz
F2PCKM	0.49051 ppm/cm
F2HCKM	245.32004 Hz/cm
F1PCKM	0.48863 ppm/cm
F1HCKM	244.37650 Hz/cm

Figure S18. HMQC spectrum of 2



Current Data Parameters
NAME pentramy
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020220
Time 16.02
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG invtp
TD 1024
SOLVENT CDCl3
NS 16
DS 32
SMH 7246.377 Hz
FIDRES 7.076540 Hz
AQ 0.0707060 sec
RG 3649.1
DM 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
P1 10.00 usec
SF01 500.132885 MHz
NUC1 1H
PL1 0.00 dB
D2 0.00345000 sec
PL2 2.00 dB
P2 20.00 usec
P4 14.20 usec
SF02 125.770955 MHz
NUC2 13C
D7 0.40000001 sec
P3 7.10 usec
D0 0.00000300 sec
PL12 22.00 dB
CPOPRG2 garp
PCPD2 74.00 usec
IN0 0.00000825 sec

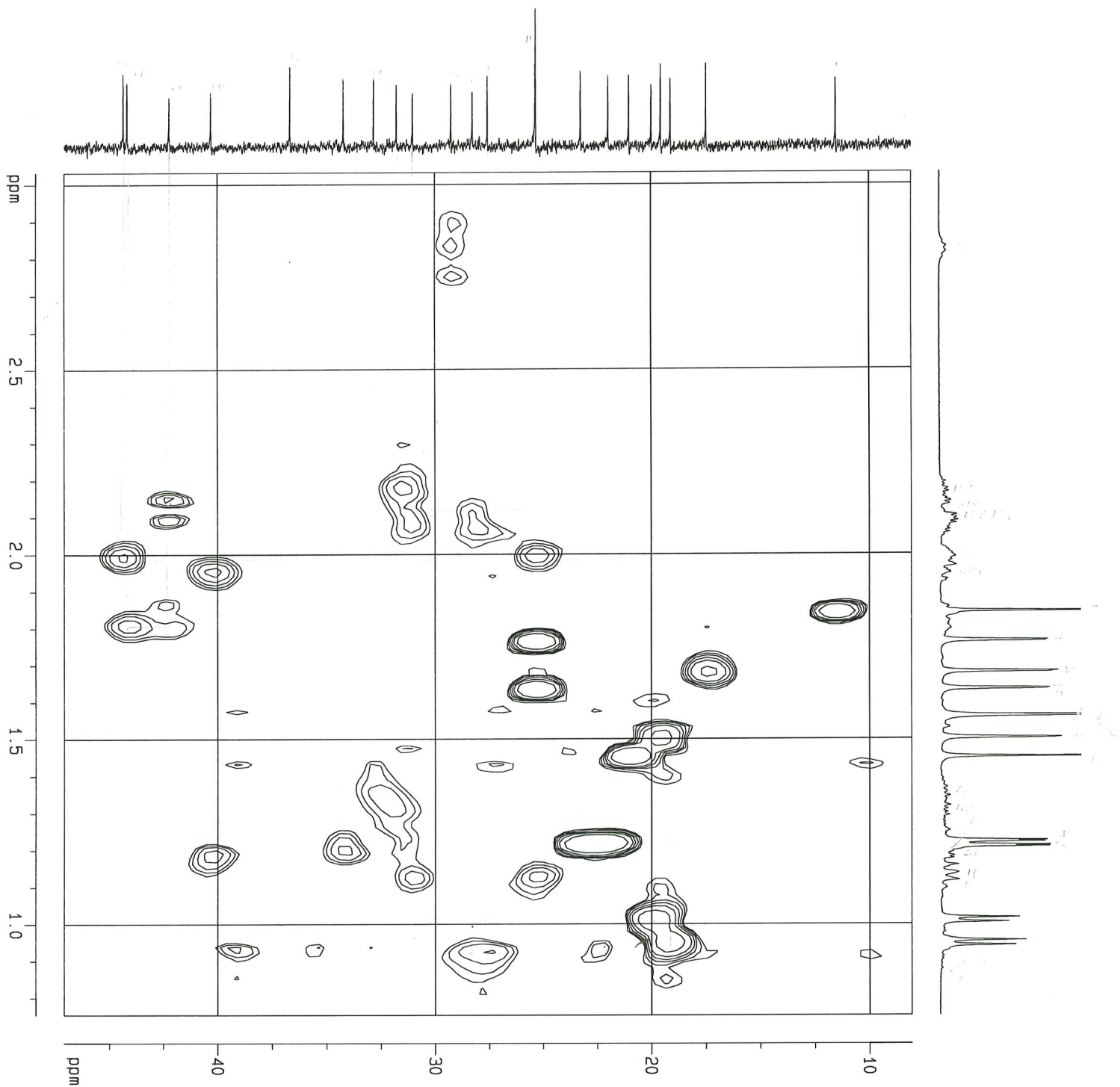
F1 - Acquisition Parameters
NDD 4
TD 256
SF01 125.771 MHz
FIDRES 118.371216 Hz
SM 240.939 ppm

F2 - Processing Parameters
SI 1024
SF 500.1300135 MHz
WDW DSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing Parameters
SI 512
MC2 TPT
SF 125.7577906 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PL0 8.012 ppm
F2LO 4007.28 Hz
F2PH1 0.465 ppm
F2PH1 242.56 Hz
F1PL0 201.953 ppm
F1LO 23397.18 Hz
F1PH1 2.876 ppm
F1PH1 361.66 Hz
F2PRCK 0.50183 ppm/cm
F2PRCK 250.98128 Hz/cm
F1PRCK 13.27182 ppm/cm
F1HZCK 1669.03406 Hz/cm

Figure S19. HMQC spectrum of 2 (expansion)



Current Data Parameters

NAME	pentan-3-ol
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20020220
Time	16.02
INSTRUM	spect
PROBHD	5 mm Nujol
PULPROG	zgpg30
TD	1024
SOLVENT	CDCl3
NS	16
DS	32
SWH	7246.377 Hz
FIDRES	7.076540 Hz
AQ	0.0707060 sec
RG	3549.1
DM	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.2500000 sec
P1	10.00 usec
SFO1	500.1328985 MHz
NUC1	1H
PL1	0.00 dB
D2	0.0034500 sec
PL2	2.00 dB
P2	20.00 usec
P4	14.20 usec
SFO2	125.7709595 MHz
NUC2	13C
D7	0.4000001 sec
P3	7.10 usec
D0	0.0000030 sec
PL12	22.00 dB
CPDPRG2	garp
PCPD2	74.00 usec
IND	0.00000825 sec

F1 - Acquisition Parameters

NO	4
TD	256
SFO1	125.771 MHz
FIDRES	118.37216 Hz
SM	240.938 ppm

F2 - Processing parameters

SI	1024
SF	500.130135 MHz
WDW	OSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00

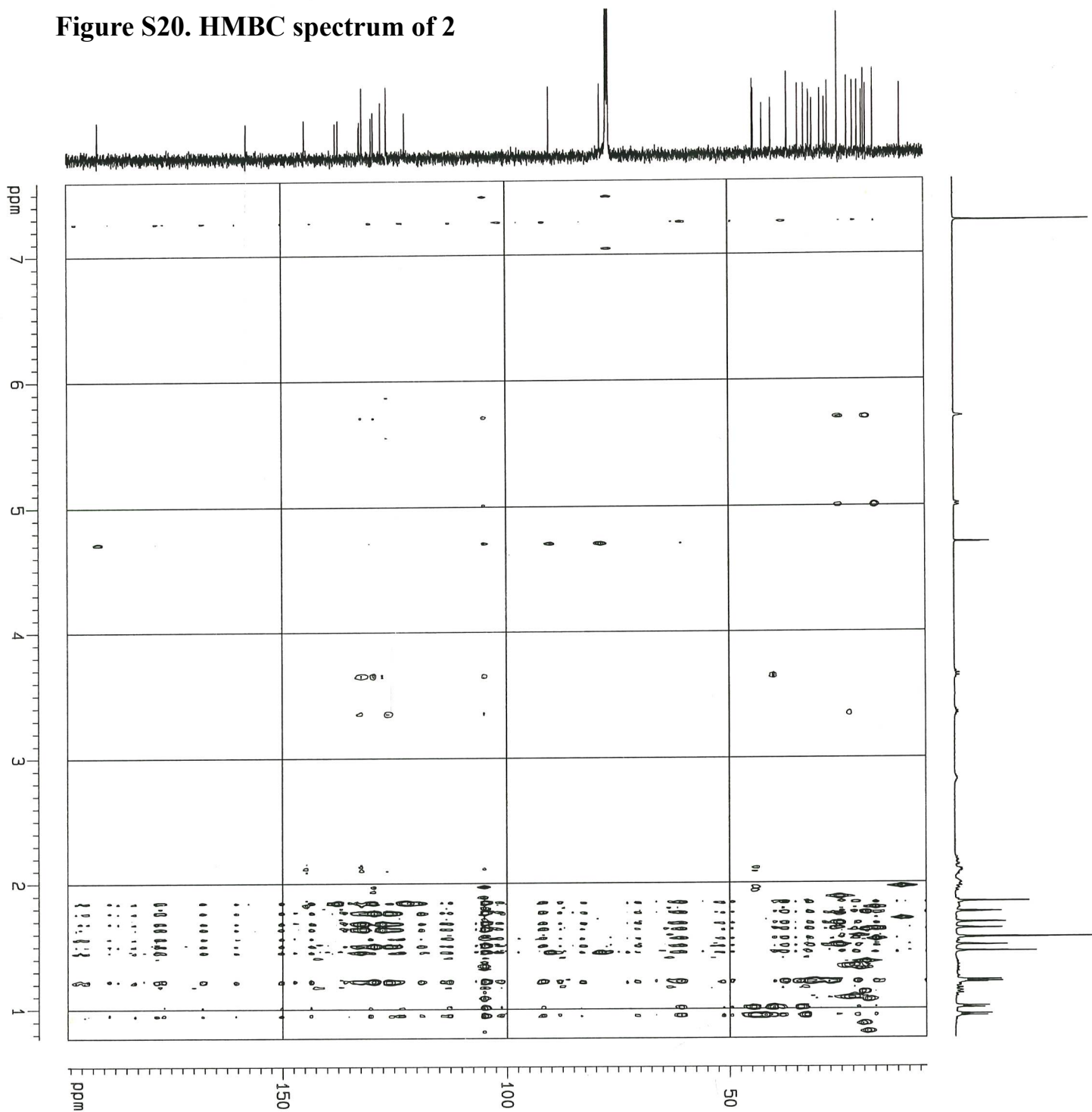
F1 - Processing parameters

SI	512
MC2	125.757790 MHz
SF	125.757790 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

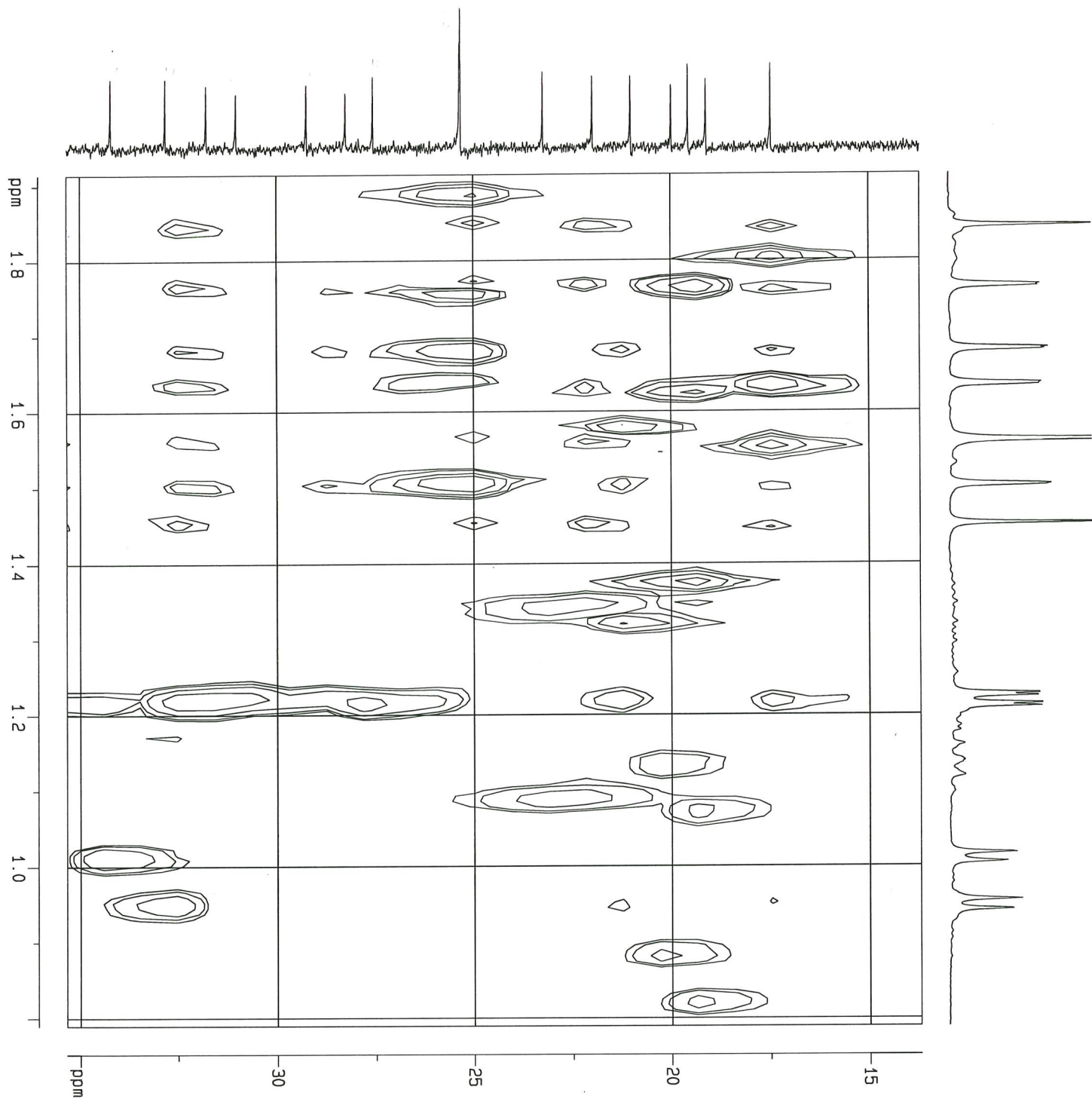
2D NMR plot parameters

CK2	15.00 cm
CK1	15.00 cm
FEHL0	3.032 ppm
FEHL	1516.34 Hz
FEH1	0.754 ppm
FEH2	37.102 Hz
FHLO	47.115 ppm
FHLO	5525.11 Hz
FH1	6.035 ppm
FH1	1012.71 Hz
FEPRCK	3.15167 ppm/cm
FEPRCK	75.55486 Hz/cm
F1PRCK	2.60416 ppm/cm
F1PRCK	327.49388 Hz/cm

Figure S20. HMBC spectrum of 2

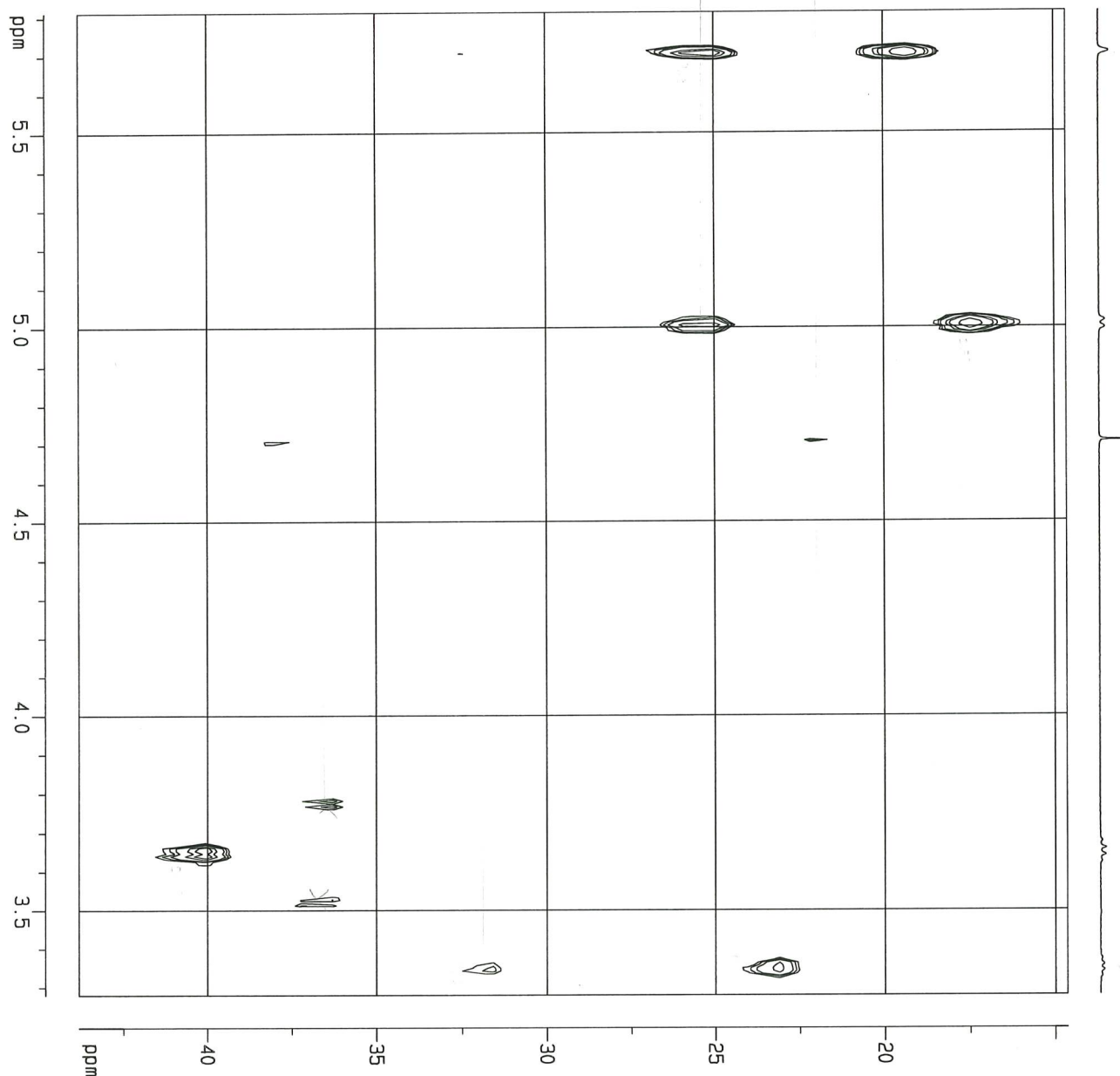


Current Data Parameters	
NAME	pen-2-en-1-ol
EXPNO	15
PROCNO	1
F2 - Acquisition Parameters	
Date_	20202220
Time	18.12
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	inv4dprnd
TD	4096
SOLVENT	CDCl3
NS	104
DS	32
SWH	7246.377 Hz
FIDRES	1.765135 Hz
AQ	0.265740 sec
RG	1149.4
DM	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.50000000 sec
P1	10.00 usec
SFO1	500.136885 MHz
NUC1	1H
PL1	0.00 dB
P2	0.00300000 sec
P3	7.10 usec
SFO2	125.770955 MHz
NUC2	13C
PL2	2.00 dB
D6	0.05000000 sec
D0	0.00003000 sec
P2	20.00 usec
INO	0.00001650 sec
F1 - Acquisition Parameters	
NDO	2
TD	256
SFO1	125.771 MHz
FIDRES	118.371216 Hz
SW	240.538 ppm
F2 - Processing parameters	
SI	2048
SF	500.1300135 MHz
WDW	Q5INE
SSB	4
LB	0.00 Hz
GB	0
PC	1.00
F1 - Processing parameters	
SI	256
MC2	GF
SF	125.757306 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0
2D NMR plot parameters	
CX2	15.00 cm
CX1	15.00 cm
F2FLO	7.3593 ppm
F2LO	3796.52 Hz
F2PH1	0.766 ppm
F2H1	364.09 Hz
F1FLO	199.188 ppm
F1LO	24923.69 Hz
F1PH1	6.170 ppm
F1H1	775.96 Hz
F2PCHM	0.45514 ppm/cm
F2H2CM	227.62871 Hz/cm
F1PCHM	12.80118 ppm/cm
F1H2CM	1609.84851 Hz/cm



Current Data Parameters		
NAME	penh-rmly	15
EXPNO	1	1
PROCNO	1	1
F2 - Acquisition Parameters		
Date_	20020220	18.12
Time	18.12	15
INSTRUM	spec	1
PROBHD	5 mm NMR/1H	1
PULPROG	zgpg30	1
TD	4096	1
SOLVENT	CDCl3	104
NS	104	1
DS	32	1
SH1	7246.377 Hz	1
FIDRES	1.69135 Hz	1
F1 - Acquisition Parameters		
RG	1149.4	1
DM	69.000 usec	1
D2	6.00 usec	1
D3	300.0 K	1
TE	1.5000000 sec	1
T1	10.00 usec	1
P1	500.1320885 MHz	1
SFO1	500.1320885 MHz	1
NULC1	1H	1
PL1	0.00 dB	1
D2	0.0030000 sec	1
D3	7.10 usec	1
SFO2	125.7705955 MHz	1
NULC2	13C	1
PL2	2.00 dB	1
D6	0.0500000 sec	1
D2	0.0000300 sec	1
P2	20.00 usec	1
IN0	0.00001650 sec	1
F1 - Acquisition Parameters		
TD	256	1
SFO1	125.771 MHz	1
FIDRES	118.371216 Hz	1
SN	240.938 ppm	1
F2 - Processing Parameters		
SI	2048	1
SF	500.1300135 MHz	1
MDM	OSINE	1
SSB	4	1
LB	0.00 Hz	1
GB	0	1
PC	1.00	1
F1 - Processing Parameters		
SI	256	1
MC2	OF	1
SF	125.7577905 MHz	1
MDM	SINE	1
SSB	2	1
LB	0.00 Hz	1
GB	0	1
2D NMR plot parameters		
CX2	15.00 cm	1
CX1	15.00 cm	1
F2P2D	1.914 ppm	1
F2P1D	957.29 Hz	1
F2PHT	0.789 ppm	1
F2H1	364.71 Hz	1
F1P1D	35.349 ppm	1
F1H1	4445.47 Hz	1
F1H1T	13.700 ppm	1
F1H1M	0.0739 ppm	1
F2P2H2	1722.93 Hz	1
F2P2H1	0.0739 ppm/cm	1
F2P2H2	37.50566 Hz/cm	1
F2P2H1	1.44321 ppm/cm	1
F1P2H2	161.50252 Hz/cm	1

Figure S22. HMBC spectrum of 2 (expansion)



Current Data Parameters

NAME	pen2-2m2y
EXPNO	15
PROCNO	1

F2 - Acquisition Parameters

Date_	20020220
Time	18.12
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	inv4iprd
TD	4096
SOLVENT	CDCl3
NS	104
DS	32
SWH	7246.377 Hz
FIDRES	1.769135 Hz
AQ	0.2826740 sec
RG	1149.4
DM	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.50000000 sec
P1	10.00 usec
SFO1	500.1328885 MHz
NUC1	1H
PL1	0.00 dB
PD	0.00300000 sec
P2	7.10 usec
SFO2	125.7705955 MHz
NUC2	13C
PL2	2.00 dB
PD	0.05000000 sec
D0	0.00000300 sec
P2	20.00 usec
INO	0.00001650 sec

F1 - Acquisition Parameters

ND0	2
TD	256
SFO1	125.771 MHz
FIDRES	118.37216 Hz
SW	240.938 ppm

F2 - Processing Parameters

SI	2048
SF	500.1300135 MHz
WDW	OSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.00

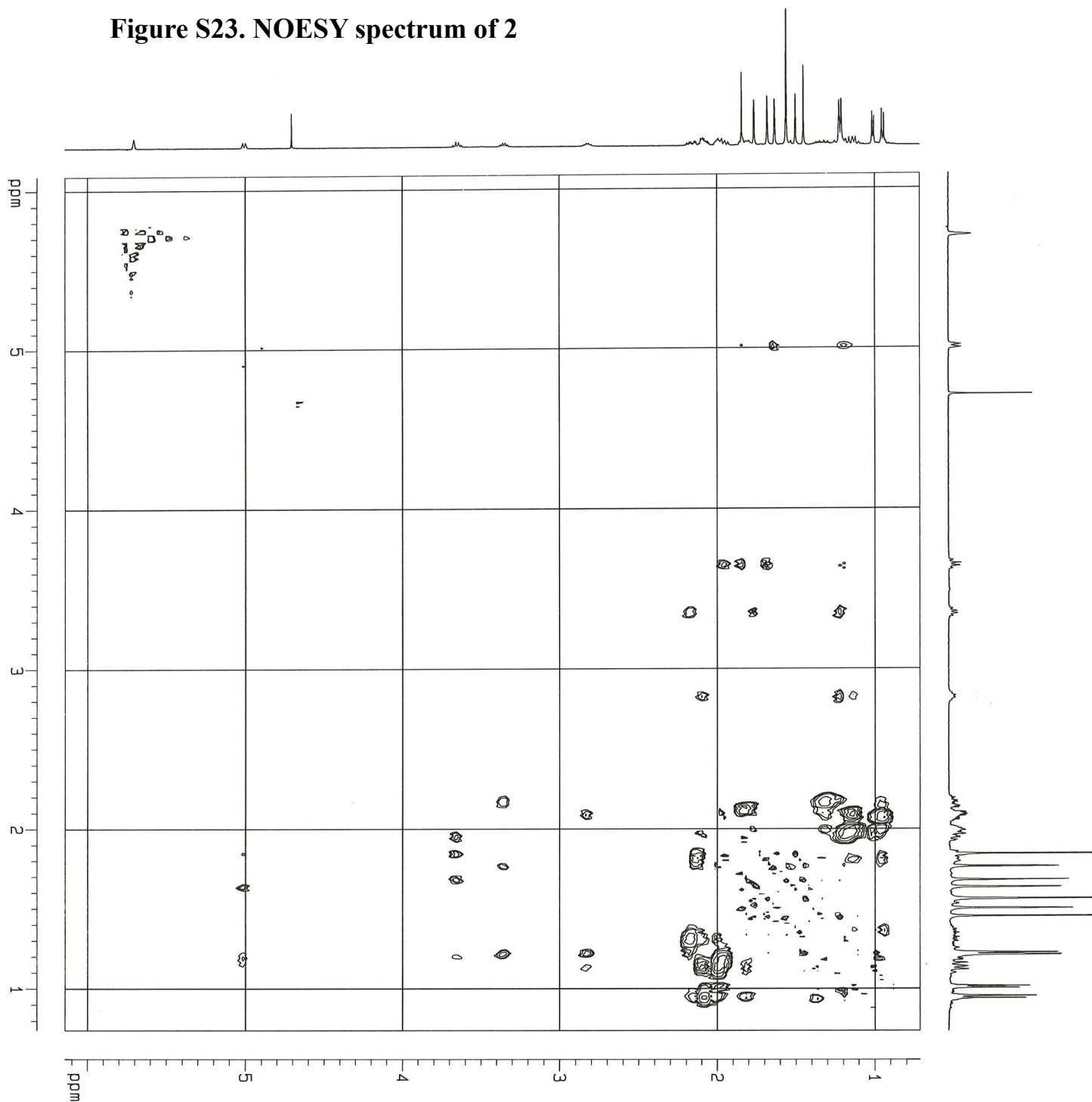
F1 - Processing Parameters

SI	256
MC2	GF
SF	125.7577906 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PUL	3.812 ppm
F2LO	2306.88 Hz
F2PHI	3.280 ppm
F2H1	1640.18 Hz
F1PUL	43.821 ppm
F1LO	5510.81 Hz
F1PHI	14.642 ppm
F1H1	1841.31 Hz
F2PPOCK	0.16865 ppm/cm
F2HZCM	84.44671 Hz/cm
F1PPOCK	1.94528 ppm/cm
F1HZCM	244.63383 Hz/cm

Figure S23. NOESY spectrum of 2



Current Data Parameters

NAME	pen2r2m2y
EXPNO	25
PROCNO	1

F2 - Acquisition Parameters

Date_	20020222
Time	18.49
INSTRUM	spect
PROBHD	5 mm MUltinu
PULPROG	noesyld
TD	1024
SOLVENT	CDCl3
NS	72
DS	16
SMH	7246.377 Hz
FIDRES	7.076540 Hz
RG	0.0707060 sec
RG	128
DW	69.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.0000000 sec
P1	10.00 usec
SFO1	500.132885 MHz
NUC1	1H
PL1	0.00 dB
DO	0.0000000 sec
DB	0.4100000 sec
INQ	0.0000500 sec

F1 - Acquisition Parameters

NDO	2
TD	256
SFO1	500.1329 MHz
FIDRES	28.306160 Hz
SM	14.489 ppm

F2 - Processing Parameters

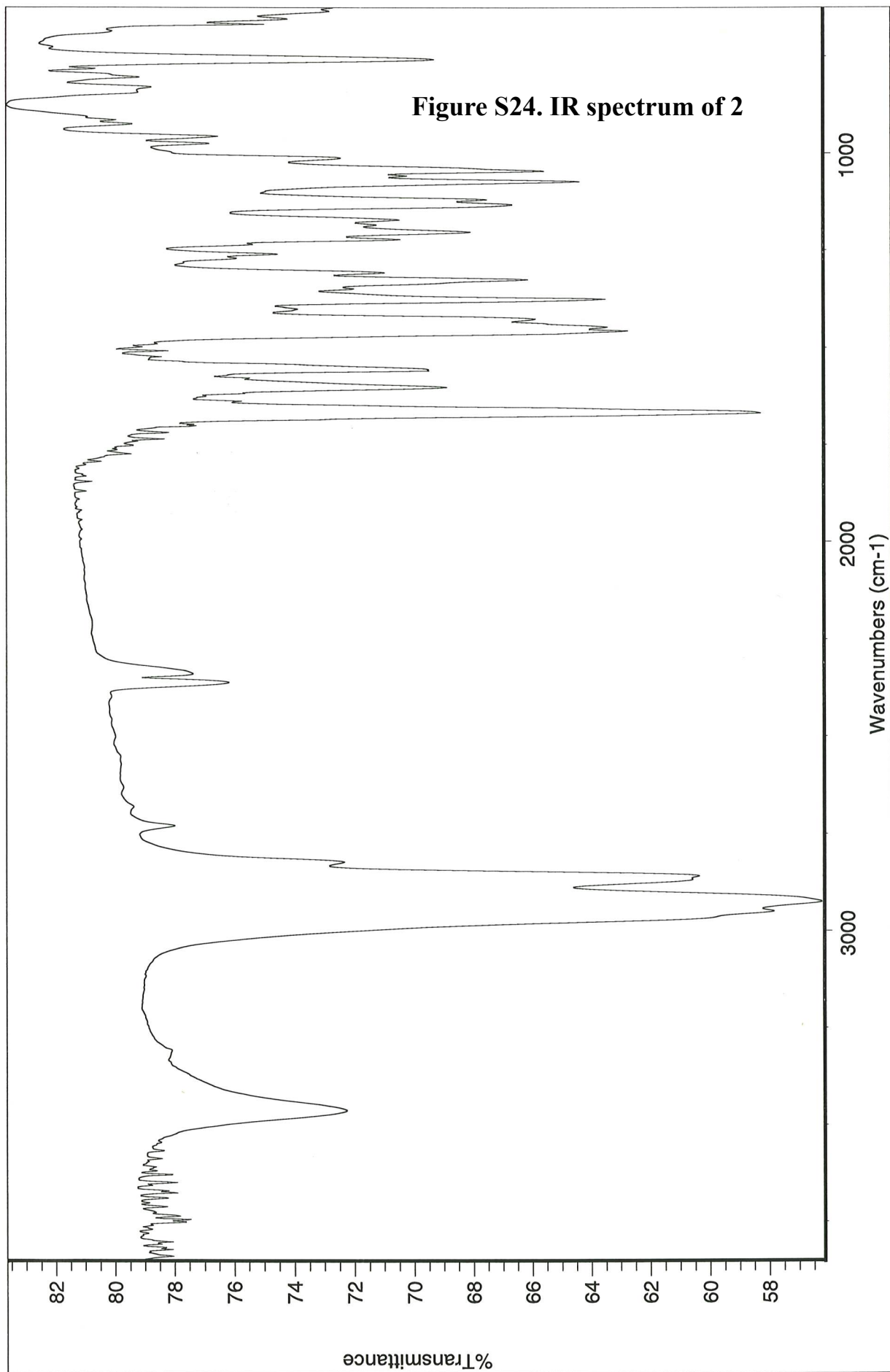
SI	1024
SF	500.1300128 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0
PC	0.00

F1 - Processing Parameters

SI	512
MC2	TPPI
SF	500.1300128 MHz
WDW	SINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PULO	6.090 ppm
F2LO	3045.56 Hz
F2PHI	0.741 ppm
F2HI	370.63 Hz
F1PULO	6.146 ppm
F1LO	3073.88 Hz
F1PHI	0.713 ppm
F1HI	356.49 Hz
F2PNUC	0.35656 ppm/cm
F2H2CM	178.32881 Hz/cm
F1PNUC	0.36222 ppm/cm
F1H2CM	181.15941 Hz/cm



Date: Wed Jul 03 15:33:00 2002

Scans: 64

Resolution: 4.000

Wed Jul 03 15:31:11 2002