# 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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# List of Supplementary Materials

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

| Atom | $\delta_H$ | $\delta_C$ | $^{1}$H–$^{1}$H COSY | HMBC[c] | NOESY |
|---|---|---|---|---|---|
| 1 | 3.60 | 36.8 | H2αβ, H14 | | Me20 |
| 2α | 1.22 | 40.2 | H1, H2β, H3 | H1, Me18 | H4 |
| 2β | 1.94 | | H1, H2α | | |
| 3 | 1.21 | 34.0 | H2α, H4, Me18 | H5β, Me18 | H5β, Me18 |
| 4 | 2.01 | 44.6 | H3, H5α | H2αβ, H3, H6αβ, Me18 | H2α |
| 5α | 0.95 | 27.7 | H4, H5β, H6αβ | | Me18 |
| 5β | 2.03 | | H5α, H6β | | H3, H7 |
| 6α | 2.15 | 32.0 | H5α, H6β, H7 | H7, Me19 | |
| 6β | 1.30 | | H5αβ, H6α, H7 | | |
| 7 | 3.20 | 28.6 | H6αβ, Me19 | H5β, H6αβ, Me19 | H5β, 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, H5β, 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, H5α |
| 19 | 1.25 | 23.1 | H7 | H7 | H7 |
| 20 | 1.96 | 15.6 | | | H1 |
| 9-OH | 5.97 | | | | |
| 1' | | 61.6 | | H5α', H6', H8α' | |
| 2' | | 141.1 | | H3', H4α', Me18', C17'-OH | |
| 3' | 3.14 | 28.7 | H4β', Me18' | H5α', Me18' | |
| 4α' | 1.46 | 24.3 | H4β', H5αβ' | H3', H5β', H6', Me18' | |
| 4β' | 1.78 | | H3', H4α', H5α' | | |
| 5α' | 1.55 | 19.3 | H4αβ', H5β' | H3' | |
| 5β' | 1.80 | | H4α', H5α', H6' | | |
| 6' | 2.35 | 40.3 | H5β', H7' | H8α', Me19' | Me18', Me19' |
| 7' | 2.05 | 41.7 | H6', H8αβ', Me19' | H6', H8β', Me19' | |
| 8α' | 2.22 | 44.5 | H7', H8β' | Me19' | |
| 8β' | 0.97 | | H7', H8α' | | |
| 9' | —[b] | 53.4[b] | | H8αβ' | |
| 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 $^{2,3}J$ = 6 and 8 Hz.

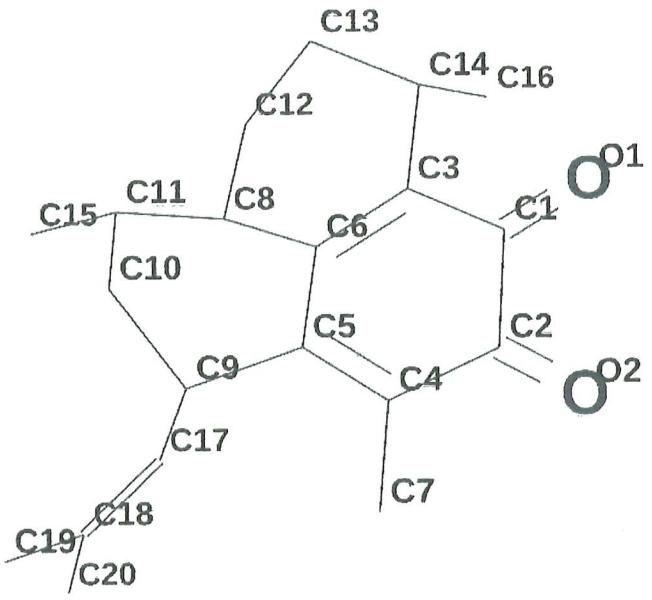
**Table S2.** $^{1}H$ NMR (500 MHz), $^{13}C$ NMR (125 MHz), $^{1}H$–$^{1}H$ COSY, HMBC, and NOESY spectroscopic data for biselisabethoxane B (2) in $CDCl_3$[a]

| Atom | $\delta_H$ | $\delta_C$ | $^{1}H$–$^{1}H$ COSY | HMBC[b] | NOESY |
|---|---|---|---|---|---|
| 1 | 3.65 | 36.7 | H2αβ, H14 | | Me20 |
| 2α | 1.18 | 40.3 | H1, H2β, H3 | H1, Me18 | H4 |
| 2β | 1.94 | | H1, H2α | | |
| 3 | 1.20 | 34.2 | H2α, H4, Me18 | H5β, Me18 | H5β, Me18 |
| 4 | 1.98 | 44.4 | H3, H5α | H2αβ, H3, H6αβ, Me18 | H2α |
| 5α | 0.97 | 28.3 | H4, H5β, H6αβ | | Me18 |
| 5β | 2.07 | | H5α, H6β | | H3, H7 |
| 6α | 2.17 | 31.8 | H5α, H6β, H7 | H7, Me19 | |
| 6β | 1.30 | | H5αβ, H6α, H7 | | |
| 7 | 3.35 | 27.6 | H6αβ, Me19 | H5β, H6αβ, Me19 | H5β, 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, H5β, 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, H5α |
| 19 | 1.21 | 23.3 | H7 | H7 | H7, Me20' |
| 20 | 1.84 | 11.5 | | | H1 |
| 1' | | 144.7 | | H2αβ' | |
| 2α' | 1.82 | 42.2 | H2β', H3' | Me18' | H2β', Me18' |
| 2β' | 2.12 | | H2α' | | H2α', Me18' |
| 3' | 1.36 | 32.8 | H2α', H4', Me18' | Me18' | H5αβ', Me18' |
| 4' | 1.80 | 44.2 | H3', H5α' | H2β', Me18' | H5β', H6β', Me18' |
| 5α' | 1.17 | 25.3 | H4', H5β', H6α' | | H3', H7' |
| 5β' | 2.00 | | H5α', H6β' | | H4', H6β' |
| 6α' | 2.09 | 31.0 | H5α', H6β', H7' | Me19' | H7' |
| 6β' | 1.12 | | H5β', H6α', H7' | | H4', H5β' |
| 7' | 2.82 | 29.3 | H6αβ', Me19' | Me19' | H5β', H6α', 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 | | H2αβ, H14', Me20' | |
| 13' | | 157.9 | | H5β' | |
| 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' | | H2αβ', 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 $^{2,3}J = 6$ and 8 Hz.

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

Job type: Equilibrium Geometry
Method: ωB97X-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) |
|---|---|---|
| *ortho*-Quinone 7 | –8.23 | –1.06 |
| Enol 7a | –7.28 | –0.45 |

# Table S5. Crystal and structure refinement data for 1



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 A |
| Crystal system, space group | Monoclinic, P2~1~ |
| Unit cell dimensions | a = 12.1072(6) A alpha = 90 deg.<br>b = 10.5771(6) A beta = 95.6550(10) deg.<br>c = 27.4452(14) A gamma = 90 deg. |
| Volume | 3497.5(3) A^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 / 8141 [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.A^-3 |

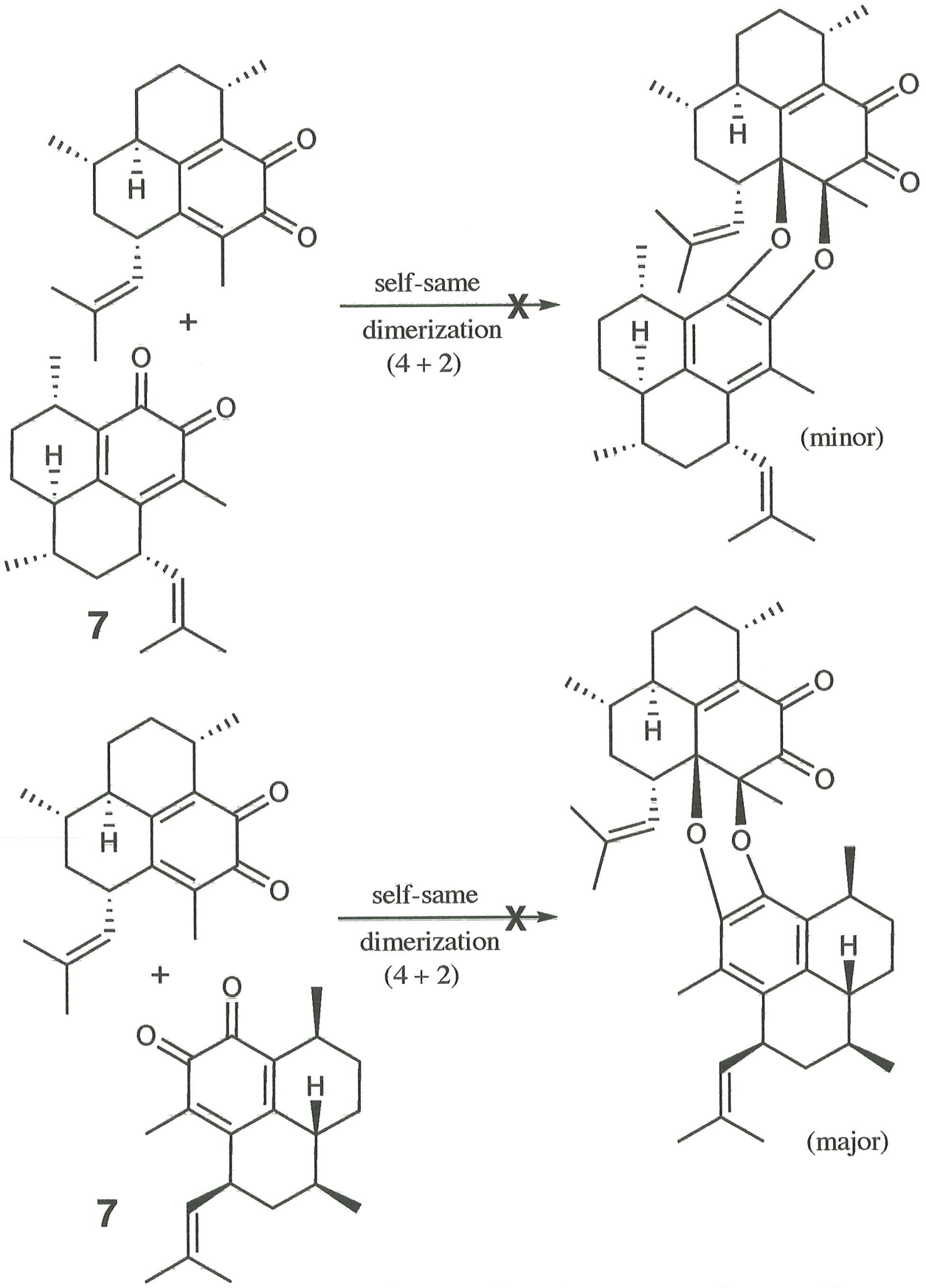
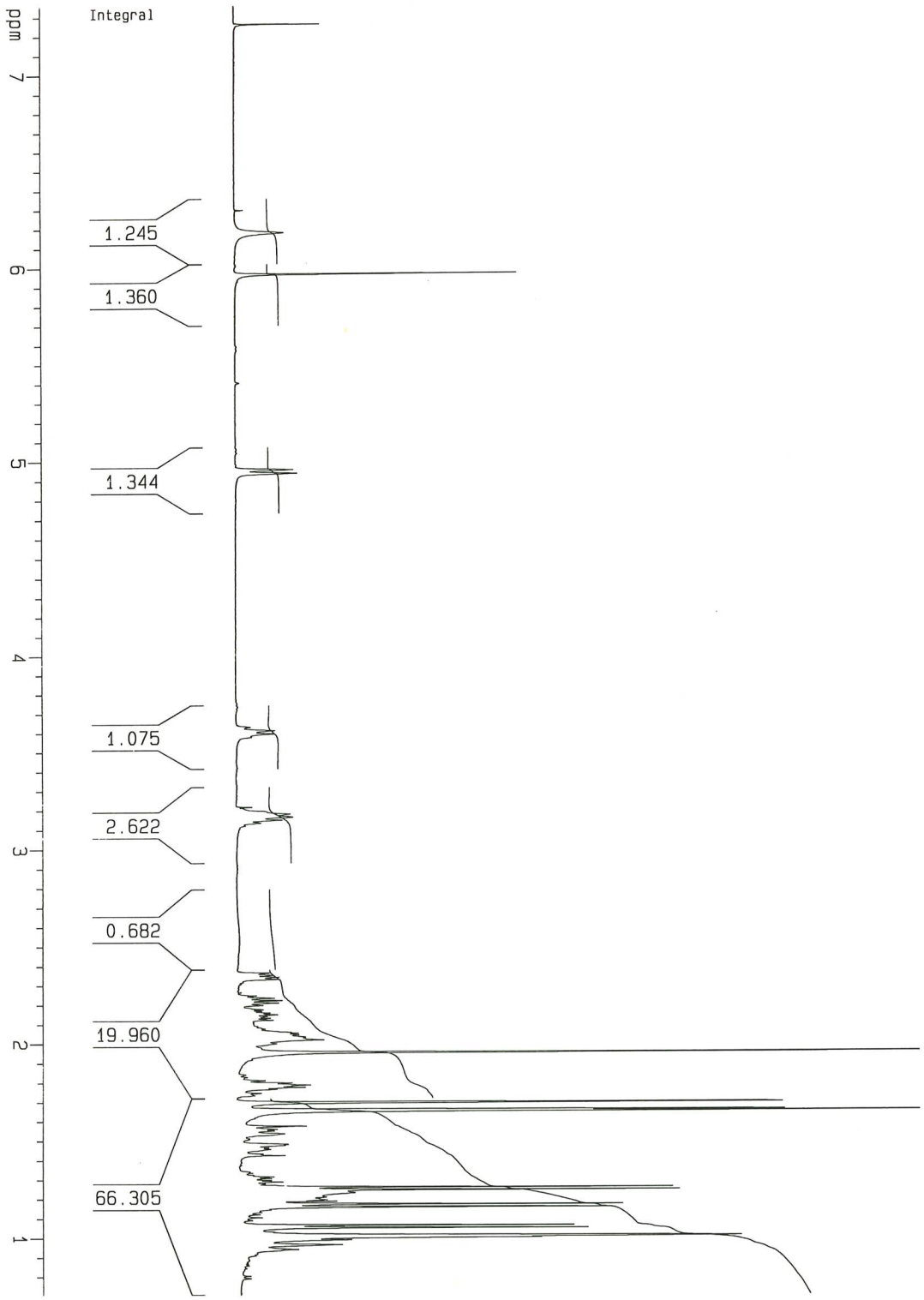



**Figure S1.** Predicted structures from self-same dimerization of *ortho*-benzoquinone **7** (not detected).

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




Bisditerpene A

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PULPROG                 zg30
TD                      32768
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FIDRES              0.221142 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          500.1328885 MHz
NUC1                      1H
PL1                  0.00 dB

F2 - Processing parameters
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WDW                       EM
SSB                        0
LB                   0.30 Hz
GB                         0
PC                    1.00

1D NMR plot parameters
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F1P                  7.357 ppm
F1                 3679.48 Hz
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HZCM             166.28813 Hz/cm
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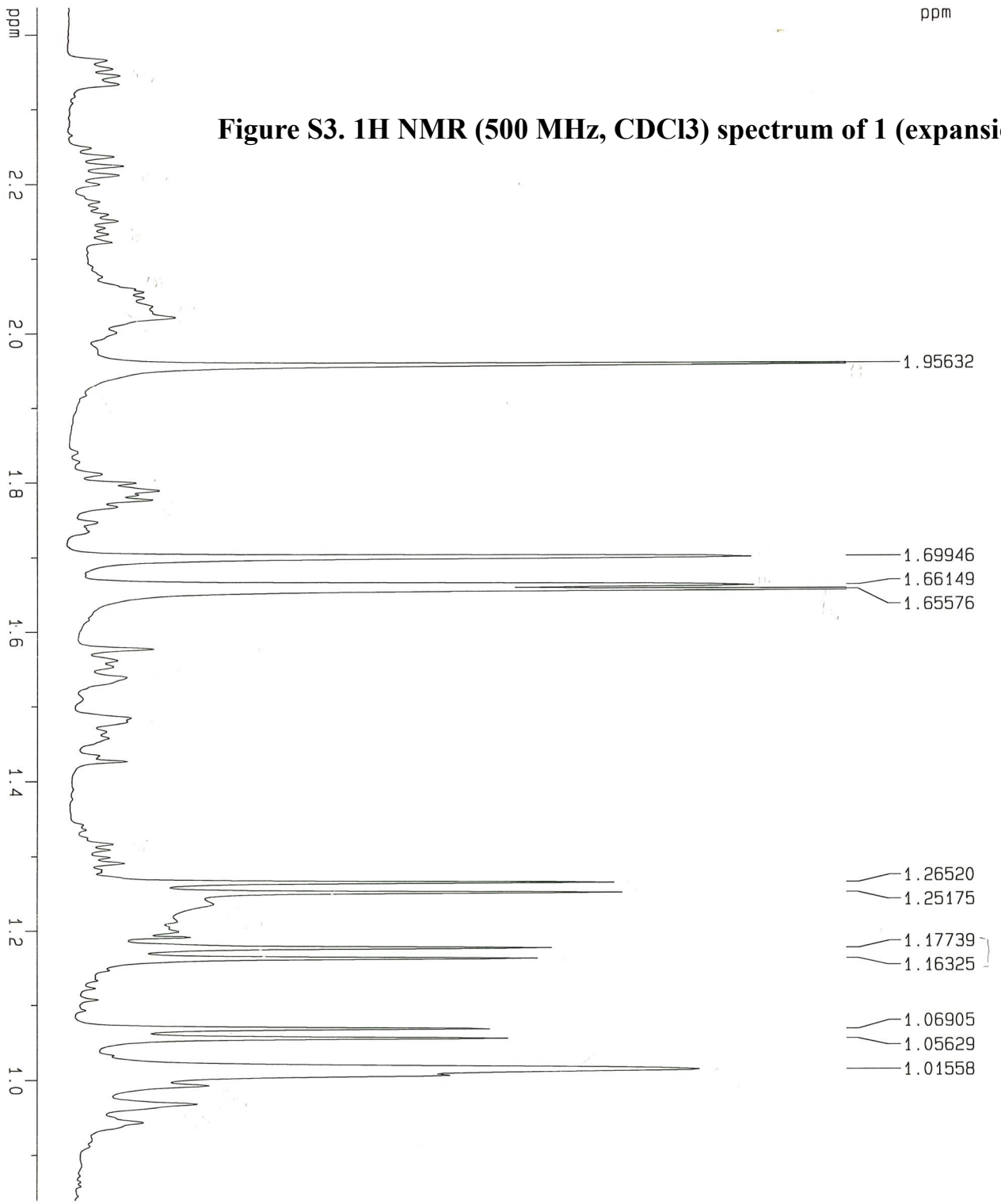


**Figure S3. 1H NMR (500 MHz, CDCl3) spectrum of 1 (expansion)**

Bisditerpene A

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PROCNO                        1

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FIDRES             0.221142 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           500.1328885 MHz
NUC1                       1H
PL1                 0.00 dB

F2 - Processing parameters
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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                 1218.69 Hz
F2P                  0.839 ppm
F2                   419.66 Hz
PPMCM             0.07990 ppm/cm
HZCM              39.96180 Hz/cm
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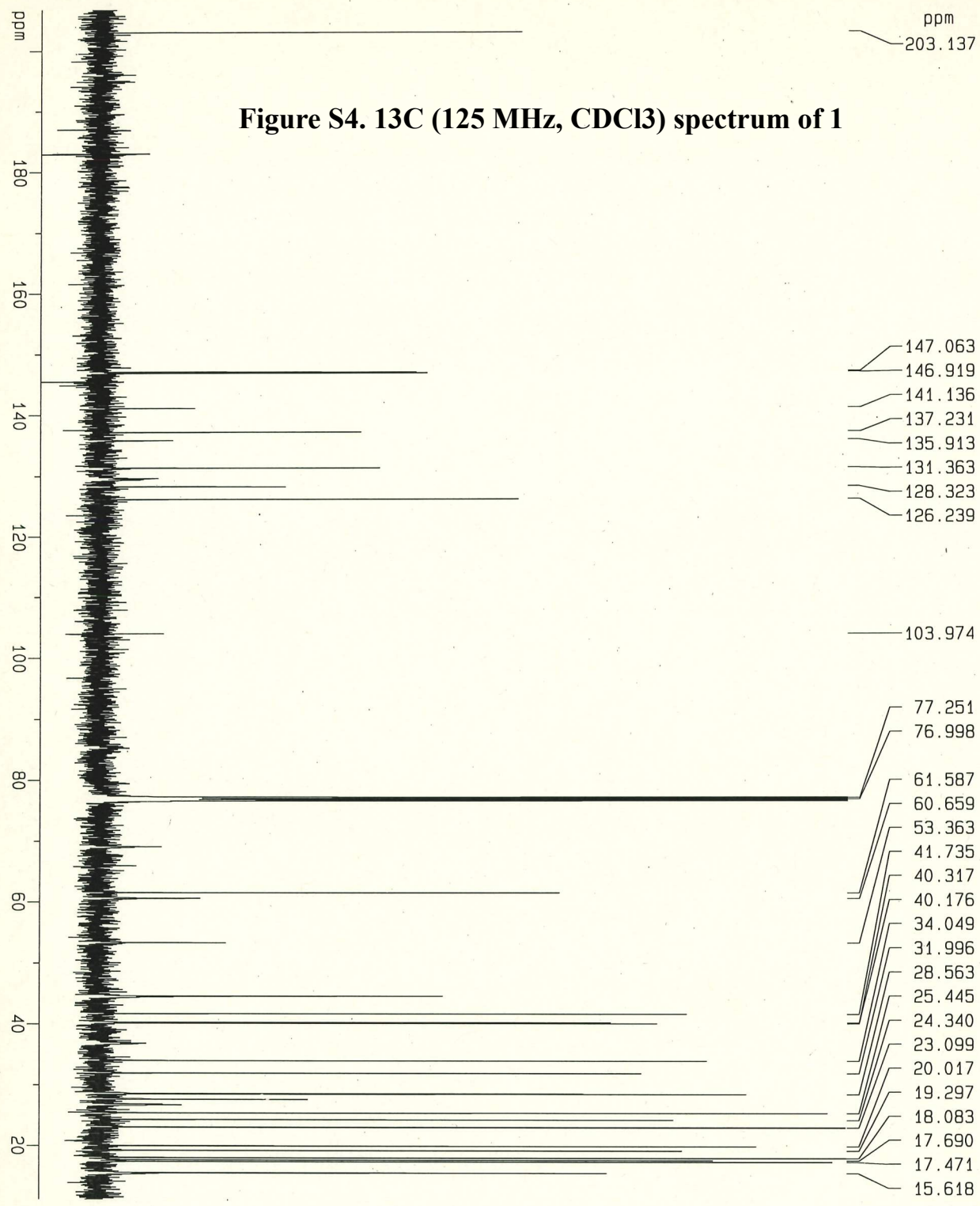
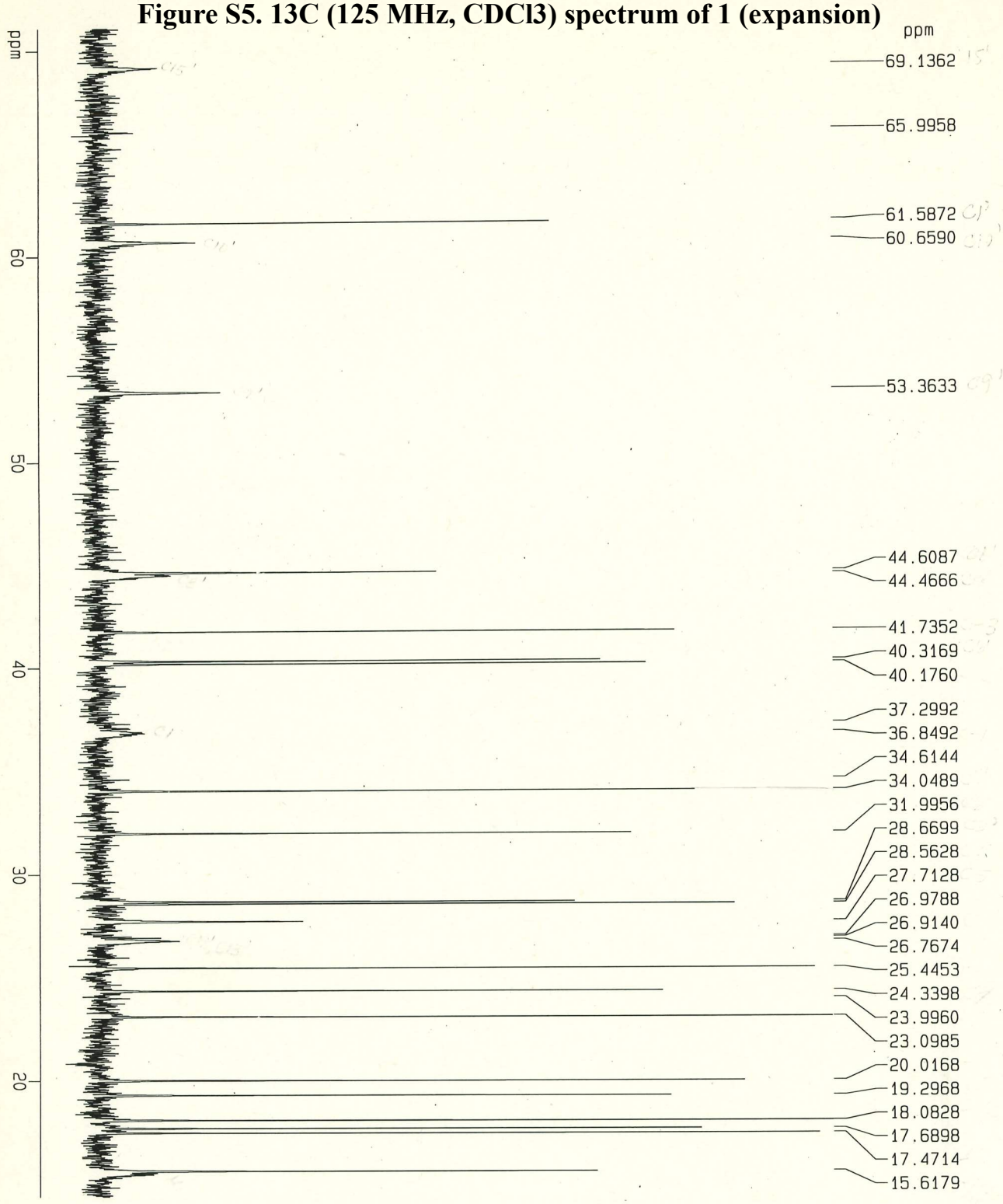



**Figure S4. 13C (125 MHz, CDCl3) spectrum of 1**

**Figure S5. 13C (125 MHz, CDCl3) spectrum of 1 (expansion)**

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DW 16.500 usec
DE 6.00 usec
TE 300.0 K
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PL13 22.00 dB
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NUC2 1H
PL2 0.00 dB
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NUC1 13C
PL1 2.00 dB
D11 0.03000000 sec

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GB 0
PC 1.00

1D NMR plot parameters
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F1 8941.96 Hz
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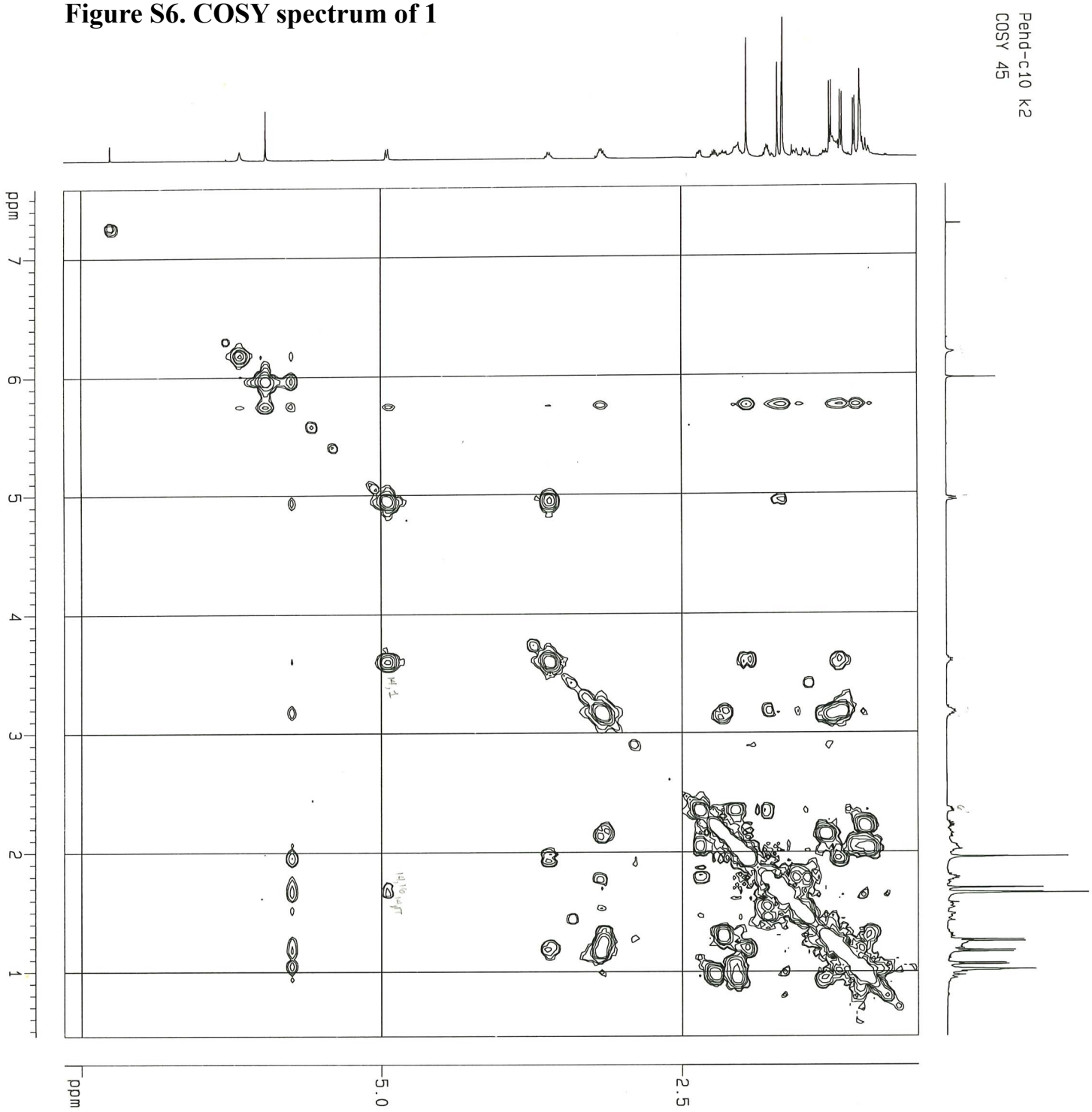
F2 1807.25 Hz
PPMCM 2.83669 ppm/cm
HZCM 356.73535 Hz/cm

# Figure S6. COSY spectrum of 1

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| SOLVENT | CDCl3 |
| NS | 8 |
| DS | 16 |
| SWH | 7246.377 Hz |
| FIDRES | 7.076540 Hz |
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| RG | 101.6 |
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| DE | 6.00 usec |
| TE | 300.0 K |
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| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

F1 - Processing parameters

| | |
|---|---|
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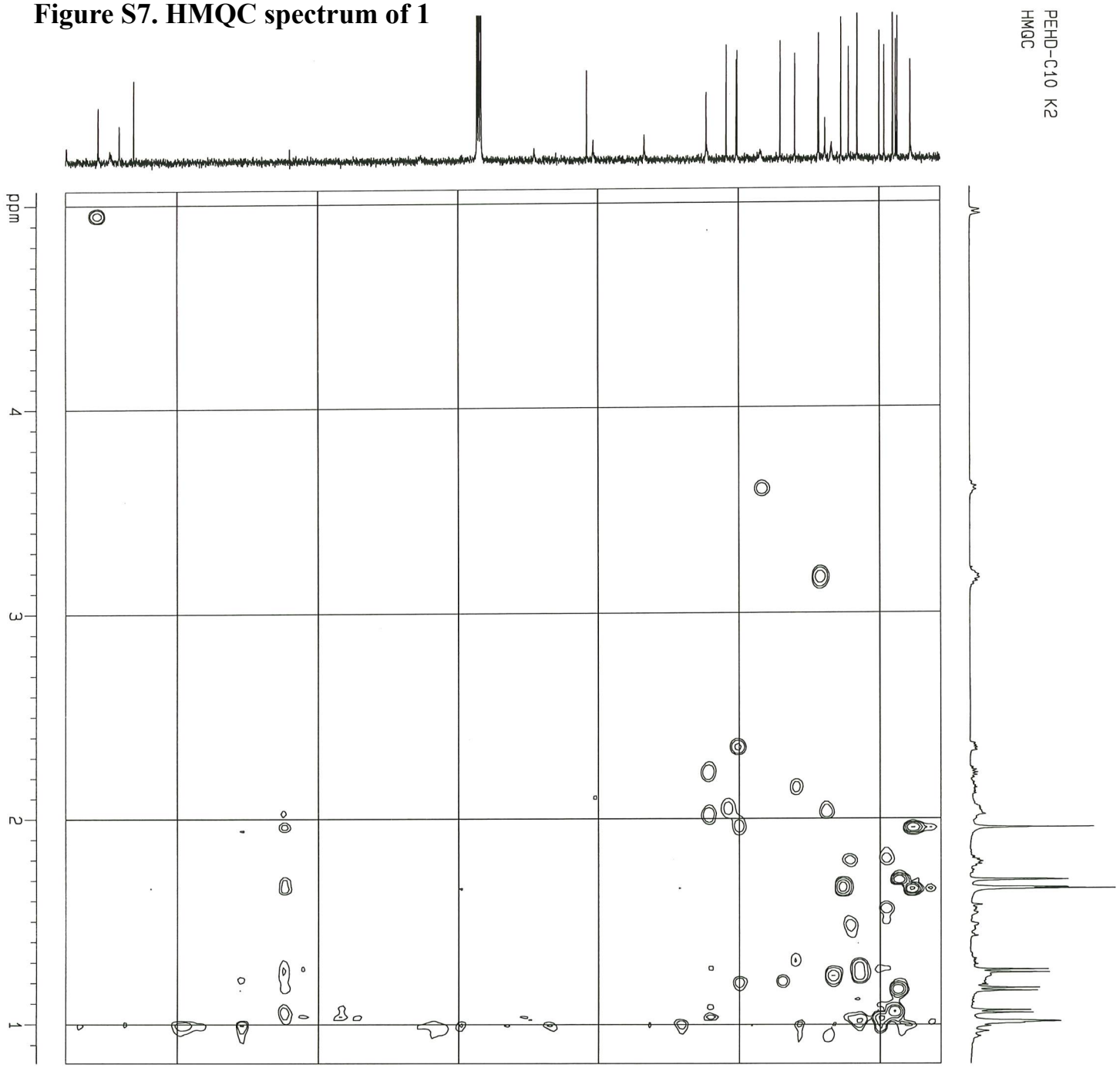
| SF | 500.1300135 MHz |
| WDW | SINE |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |

2D NMR plot parameters

| | |
|---|---|
| CX2 | 15.00 cm |
| CX1 | 15.00 cm |
| F2LO | 7.588 ppm |
| F2LO | 3794.99 Hz |
| F2PHI | 0.457 ppm |
| F2HI | 228.41 Hz |
| F1PLO | 7.645 ppm |
| F1LO | 3823.29 Hz |
| F1HI | 0.542 ppm |
| F1HI | 270.87 Hz |
| F2PPMCM | 0.47542 ppm/cm |
| F2HZCM | 237.77173 Hz/cm |
| F1PPMCM | 0.47353 ppm/cm |
| F1HZCM | 236.82820 Hz/cm |

**Figure S7. HMQC spectrum of 1**

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EXPNO 15
PROCNO 1

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SWH 7246.377 Hz
FIDRES 7.076540 Hz
AQ 0.0707060 sec
RG 574.7
DW 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
P1 10.50 usec
SFO1 500.1328885 MHz
NUC1 1H
PL1 0.00 dB
D2 0.00345000 sec
PL2 2.00 dB
P2 21.00 usec
P4 17.00 usec
SFO2 125.709955 MHz
NUC2 13C
D7 0.40000001 sec
P3 8.50 usec
D0 0.00000300 sec
PL12 20.00 dB
CPDPRG2 garp
PCPD2 66.00 usec
INO 0.00000825 sec

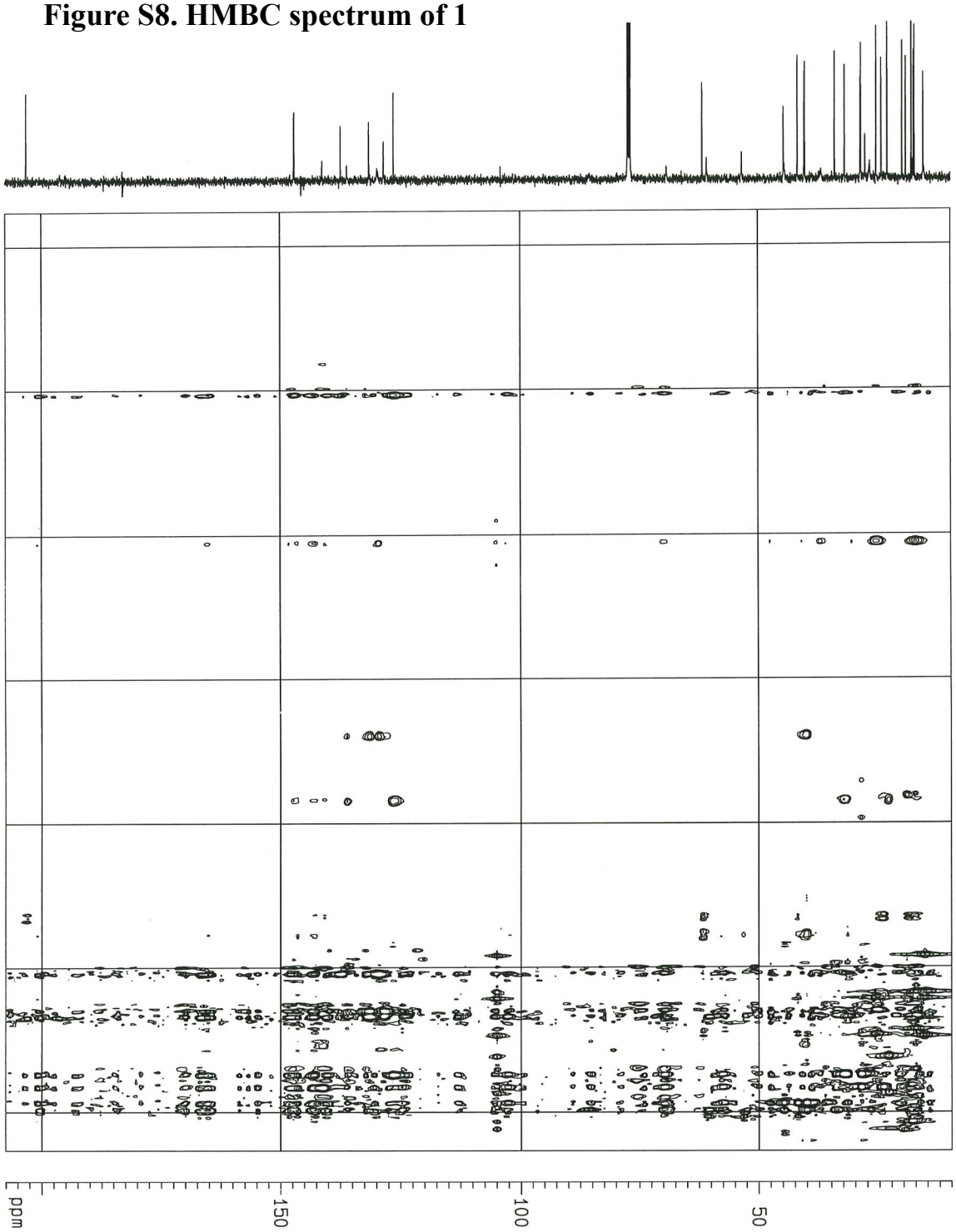
F1 - Acquisition parameters
NDO 4
TD 256
SFO1 125.771 MHz
FIDRES 118.371216 Hz
SW 240.938 ppm

F2 - Processing parameters
SI 1024
SF 500.1300135 MHz
WDW GSINE
SSB 2
LB 0.00 Hz
GB 0
PC 0.00

F1 - Processing parameters
SI 512
MC2 TPPI
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 5.069 ppm
F2LO 2535.36 Hz
F2PHI 0.810 ppm
F2HI 405.32 Hz
F1PLO 136.065 ppm
F1LO 17111.19 Hz
F1PHI 11.347 ppm
F1HI 1427.01 Hz
F2PPMCM 0.28393 ppm/cm
F2HZCM 142.00258 Hz/cm
F1PPMCM 8.31449 ppm/cm
F1HZCM 1045.61230 Hz/cm

# Figure S8. HMBC spectrum of 1




Current Data Parameters

| | |
|---|---|
| NAME | pehdc10k2 |
| EXPNO | 10 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20020710 |
| Time | 22.46 |
| INSTRUM | spect |
| PROBHD | 5 mm Multinu |
| PULPROG | inv4ipirnd |
| TD | 4096 |
| SOLVENT | CDCl3 |
| NS | 72 |
| DS | 32 |
| SWH | 7246.377 Hz |
| FIDRES | 1.769135 Hz |
| AQ | 0.2826740 sec |
| RG | 256 |
| DW | 69.000 usec |
| DE | 6.00 usec |
| TE | 300.0 K |
| D1 | 1.50000000 sec |
| P1 | 10.00 usec |
| SFO1 | 500.1320885 MHz |
| NUC1 | 1H |
| PL1 | 0.00 dB |
| D2 | 0.00300000 sec |
| P3 | 10.00 usec |
| SFO2 | 125.7709955 MHz |
| NUC2 | 13C |
| PL2 | 2.00 dB |
| D6 | 0.05000000 sec |
| D0 | 0.00000300 sec |
| P2 | 20.00 usec |
| IN0 | 0.00001650 sec |

F1 - Acquisition parameters

| | |
|---|---|
| NDO | 2 |
| TD | 256 |
| SFO1 | 125.771 MHz |
| FIDRES | 118.371216 Hz |
| SW | 240.938 ppm |

F2 - Processing parameters

| | |
|---|---|
| SI | 2048 |
| SF | 500.1300135 MHz |
| WDW | GSINE |
| SSB | 4 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

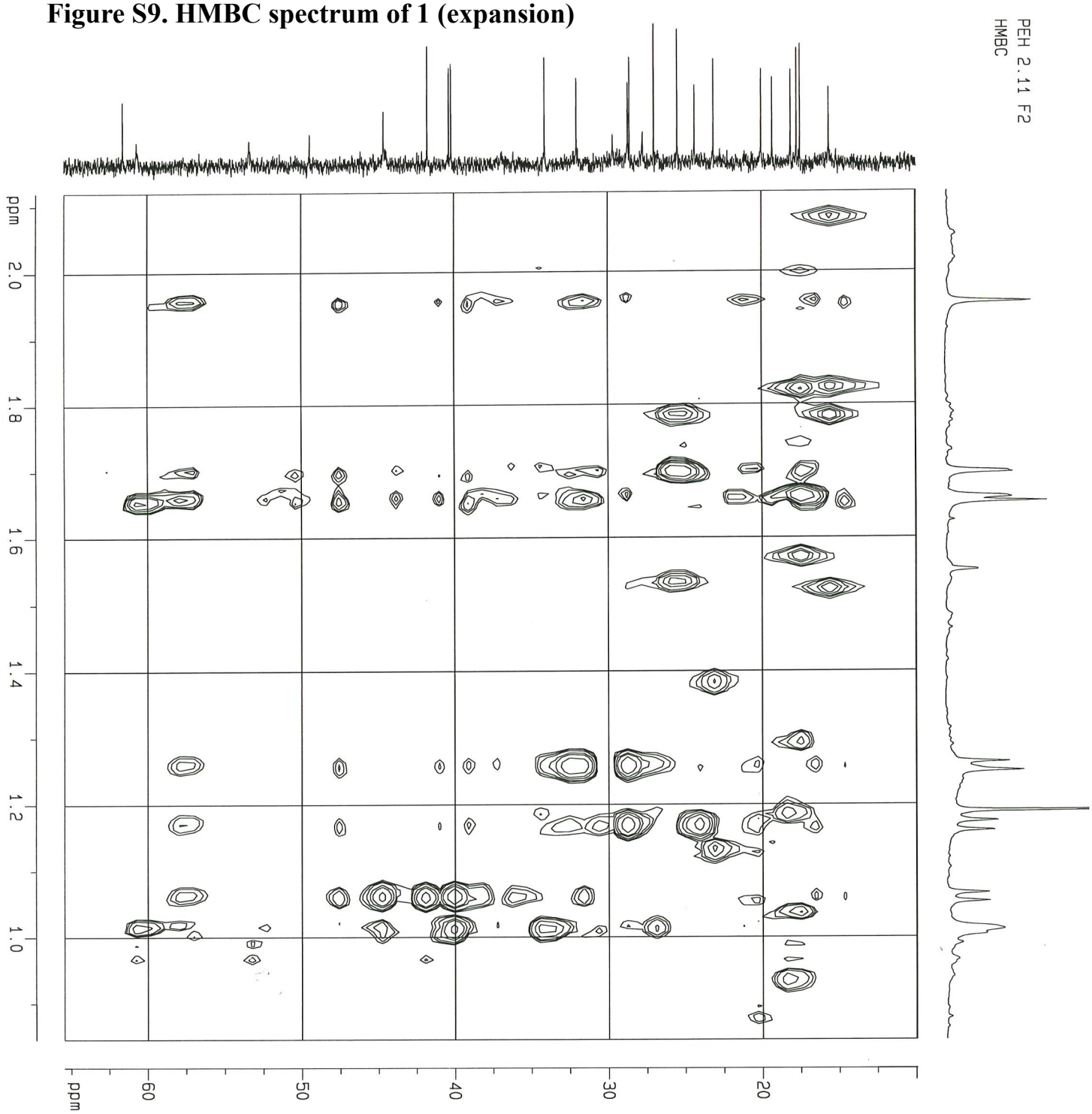
F1 - Processing parameters

| | |
|---|---|
| SI | 256 |
| MC2 | QF |
| 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 |
| F2PHI | 0.733 ppm |
| F2HI | 366.40 Hz |
| F1PLO | 207.601 ppm |
| F1LO | 26107.40 Hz |
| F1PHI | 9.935 ppm |
| F1HI | 1249.45 Hz |
| F2PPMCM | 0.43391 ppm/cm |
| F2HZCM | 217.01389 Hz/cm |
| F1PPMCM | 13.17769 ppm/cm |
| F1HZCM | 1657.19590 Hz/cm |

**Figure S9. HMBC spectrum of 1 (expansion)**




Current Data Parameters

| | |
|---|---|
| NAME | pehdc10k2 |
| EXPNO | 10 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20020710 |
| Time | 22.46 |
| INSTRUM | spect |
| PROBHD | 5 mm Multinu |
| PULPROG | inv4ip1rnd |
| TD | 4096 |
| SOLVENT | CDCl3 |
| NS | 72 |
| DS | 32 |
| SWH | 7246.377 Hz |
| FIDRES | 1.769135 Hz |
| AQ | 0.2826740 sec |
| RG | 256 |
| DW | 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 |
| D2 | 0.00300000 sec |
| P3 | 10.00 usec |
| SFO2 | 125.7709955 MHz |
| NUC2 | 13C |
| PL2 | 2.00 dB |
| D6 | 0.05000000 sec |
| D0 | 0.00000300 sec |
| P2 | 20.00 usec |
| IN0 | 0.00001650 sec |

F1 - Acquisition parameters

| | |
|---|---|
| NDO | 2 |
| TD | 256 |
| SFO1 | 125.771 MHz |
| FIDRES | 118.371216 Hz |
| SW | 240.938 ppm |

F2 - Processing parameters

| | |
|---|---|
| SI | 2048 |
| SF | 500.1300135 MHz |
| WDW | GSINE |
| SSB | 4 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

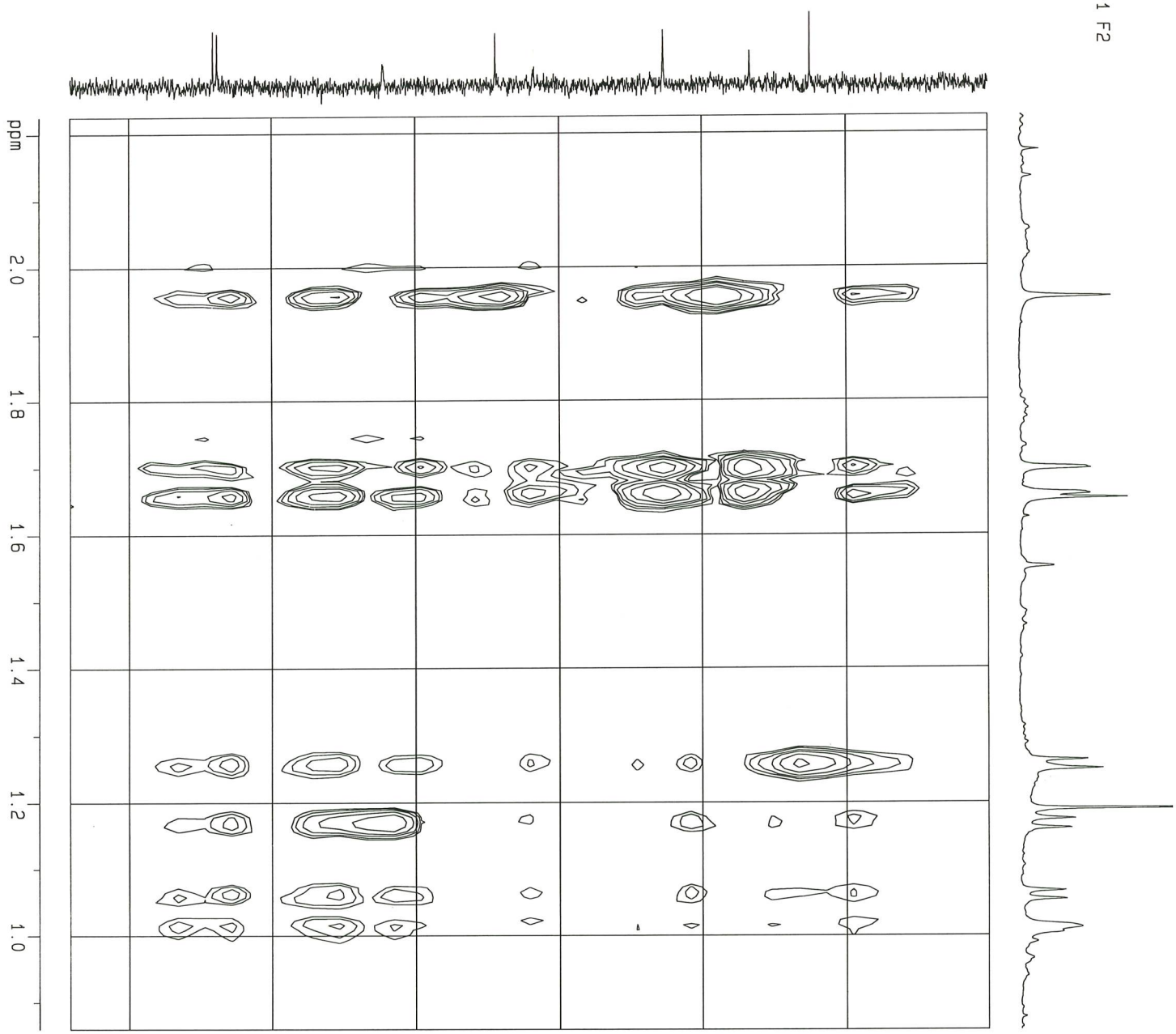
F1 - Processing parameters

| | |
|---|---|
| SI | 256 |
| MC2 | QF |
| 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 | 2.119 ppm |
| F2LO | 1059.90 Hz |
| F2PHI | 0.846 ppm |
| F2HI | 423.01 Hz |
| F1PLO | 65.470 ppm |
| F1LO | 8233.35 Hz |
| F1PHI | 9.935 ppm |
| F1HI | 1249.45 Hz |
| F2PPMCM | 0.08490 ppm/cm |
| F2HZCM | 42.45924 Hz/cm |
| F1PPMCM | 3.70230 ppm/cm |
| F1HZCM | 465.59344 Hz/cm |

# Figure S10. HMBC spectrum of 1 (expansion)




Current Data Parameters
NAME pehdc10k2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020710
Time 22.46
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG inv4ip1rnd
TD 4096
SOLVENT CDCl3
NS 72
DS 32
SWH 7246.377 Hz
FIDRES 1.769135 Hz
AQ 0.2826740 sec
RG 256
DW 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.50000000 sec
P1 10.00 usec
SFO1 500.1328865 MHz
NUC1 1H
PL1 0.00 dB
D2 0.00300000 sec
P3 10.00 usec
SFO2 125.7709955 MHz
NUC2 13C
PL2 2.00 dB
D6 0.05000000 sec
D0 0.00000300 sec
P2 20.00 usec
IN0 0.00001650 sec

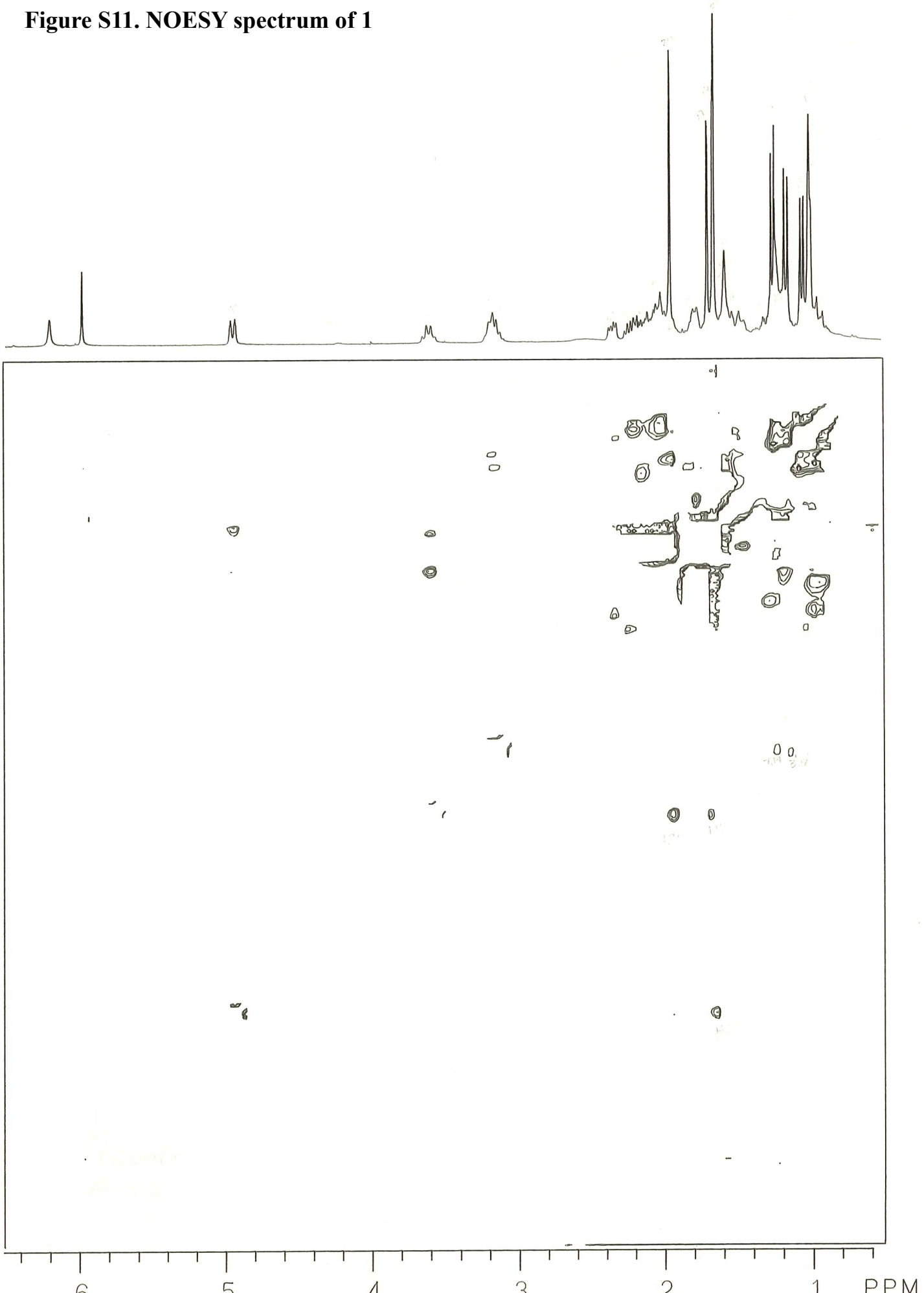
F1 - Acquisition parameters
NDO 2
TD 256
SFO1 125.771 MHz
FIDRES 118.371215 Hz
SW 240.938 ppm

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

F1 - Processing parameters
SI 256
MC2 QF
SF 125.7577905 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 2.225 ppm
F2LO 1112.98 Hz
F2PHI 0.860 ppm
F2HI 430.09 Hz
F1PLO 152.066 ppm
F1LO 19123.50 Hz
F1PHI 120.063 ppm
F1HI 15098.88 Hz
F2PPMCM 0.09103 ppm/cm
F2HZCM 45.52574 Hz/cm
F1PPMCM 2.13353 ppm/cm
F1HZCM 268.30811 Hz/cm

**Figure S11. NOESY spectrum of 1**



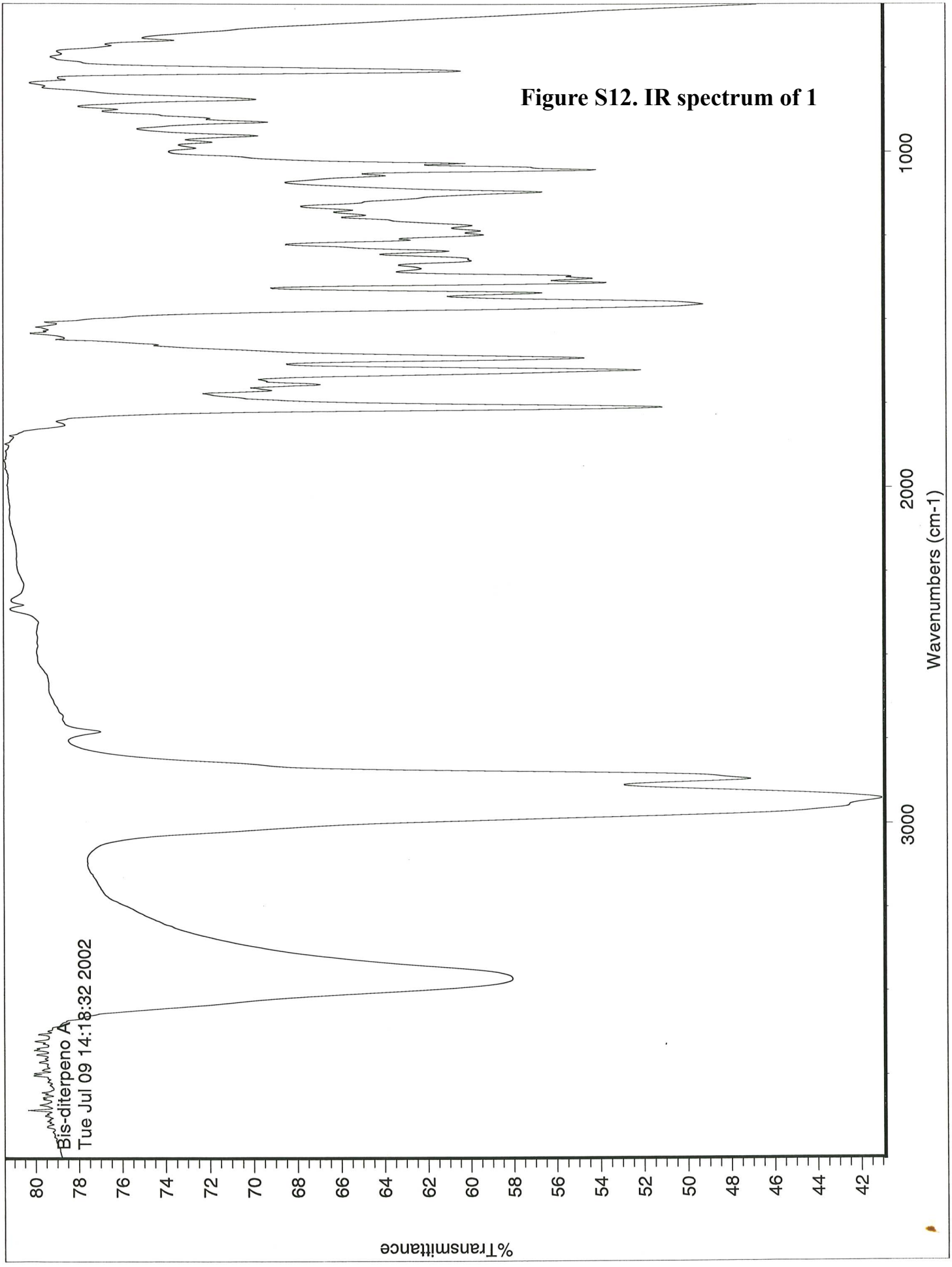
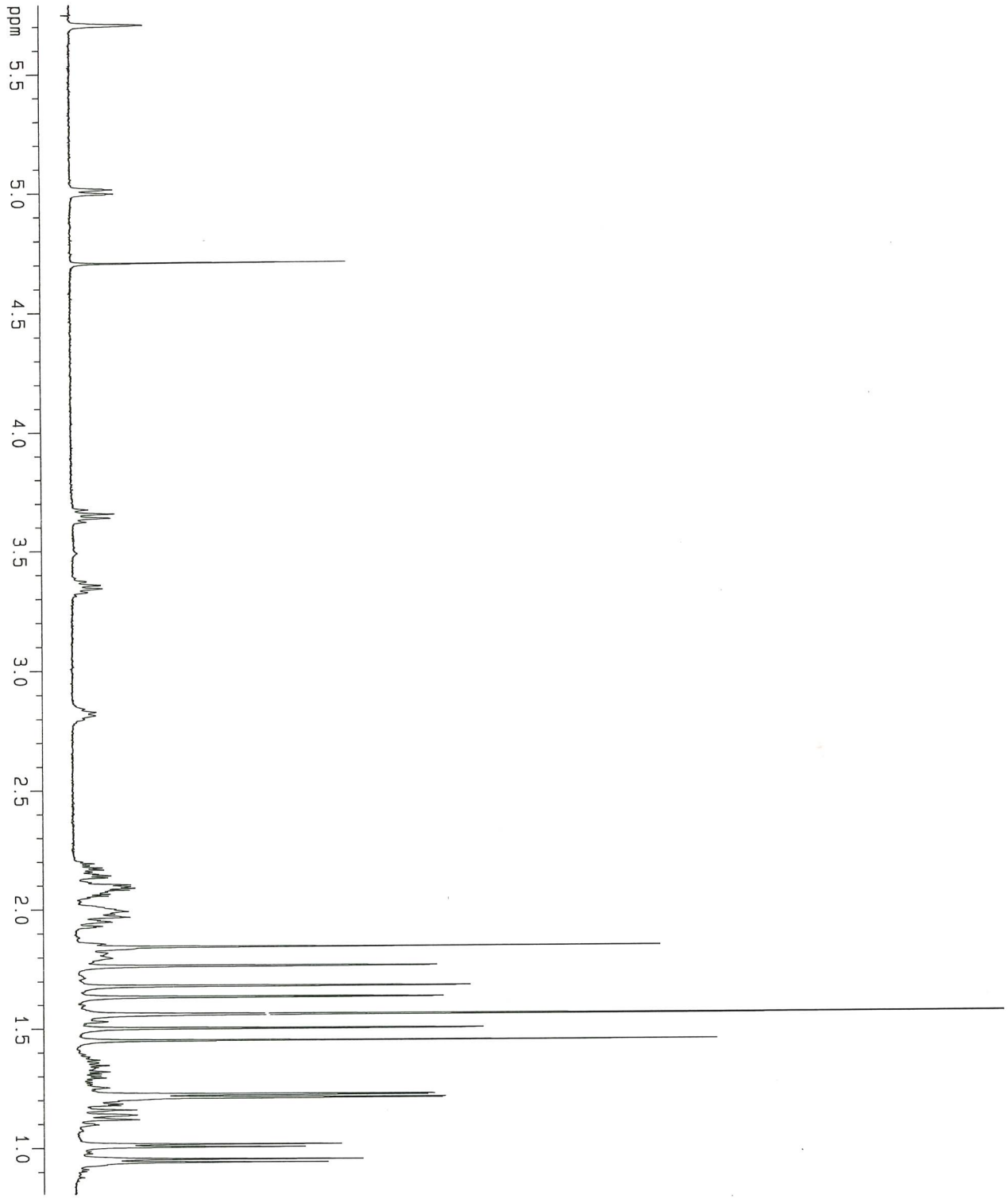


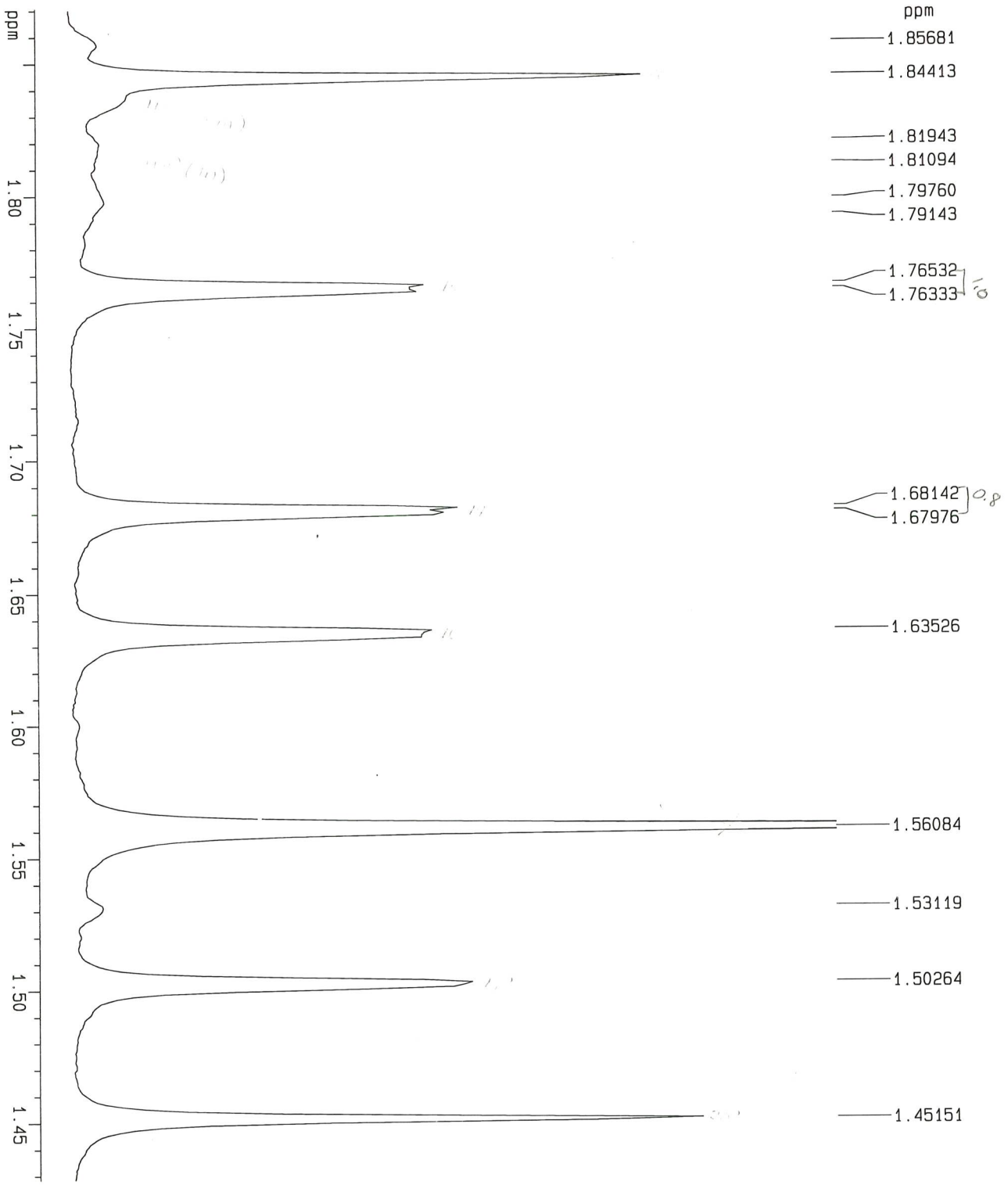

**Figure S12. IR spectrum of 1**

**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
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                  7246.377 Hz
FIDRES               0.221142 Hz
AQ                   2.2610421 sec
RG                       724.1
DW                   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)**



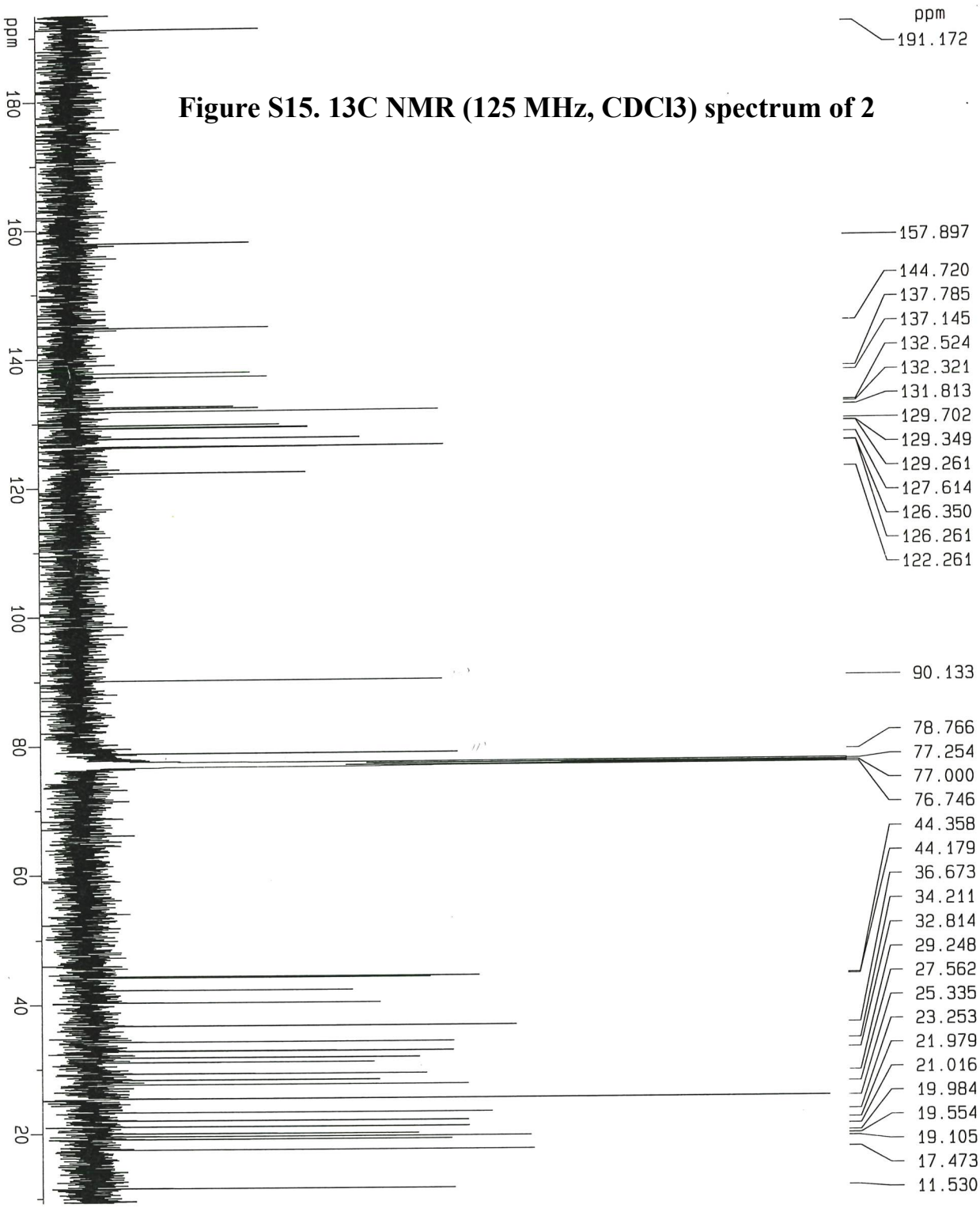



**Figure S15. 13C NMR (125 MHz, CDCl3) spectrum of 2**

PEH 2 R2 M2 y

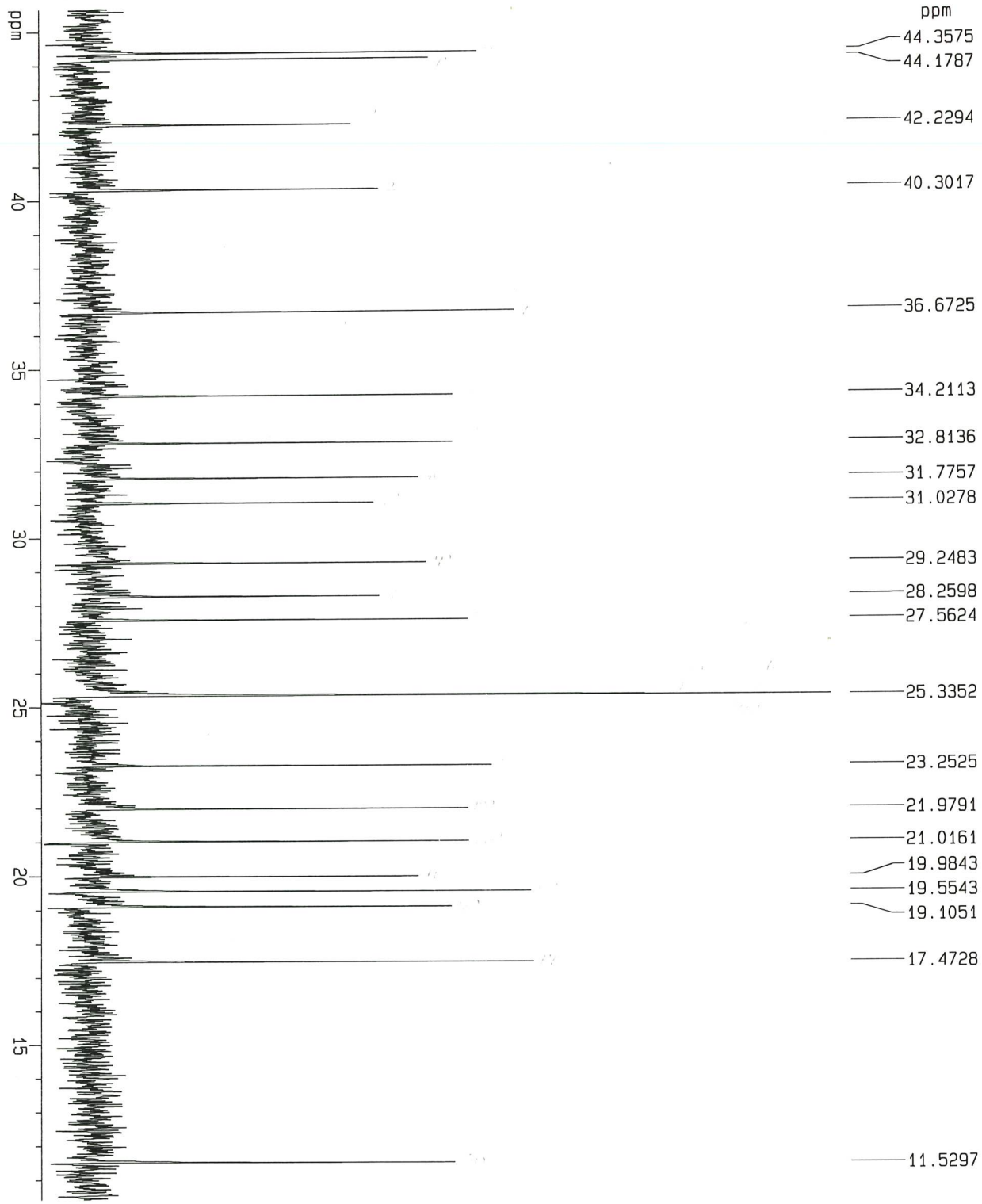
Current Data Parameters
NAME peh2r2m2y
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020221
Time 16.44
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 25204
DS 2
SWH 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 1H
PL2 0.00 dB
PL12 22.00 dB
P1 7.87 usec
SFO1 125.7709955 MHz
NUC1 13C
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

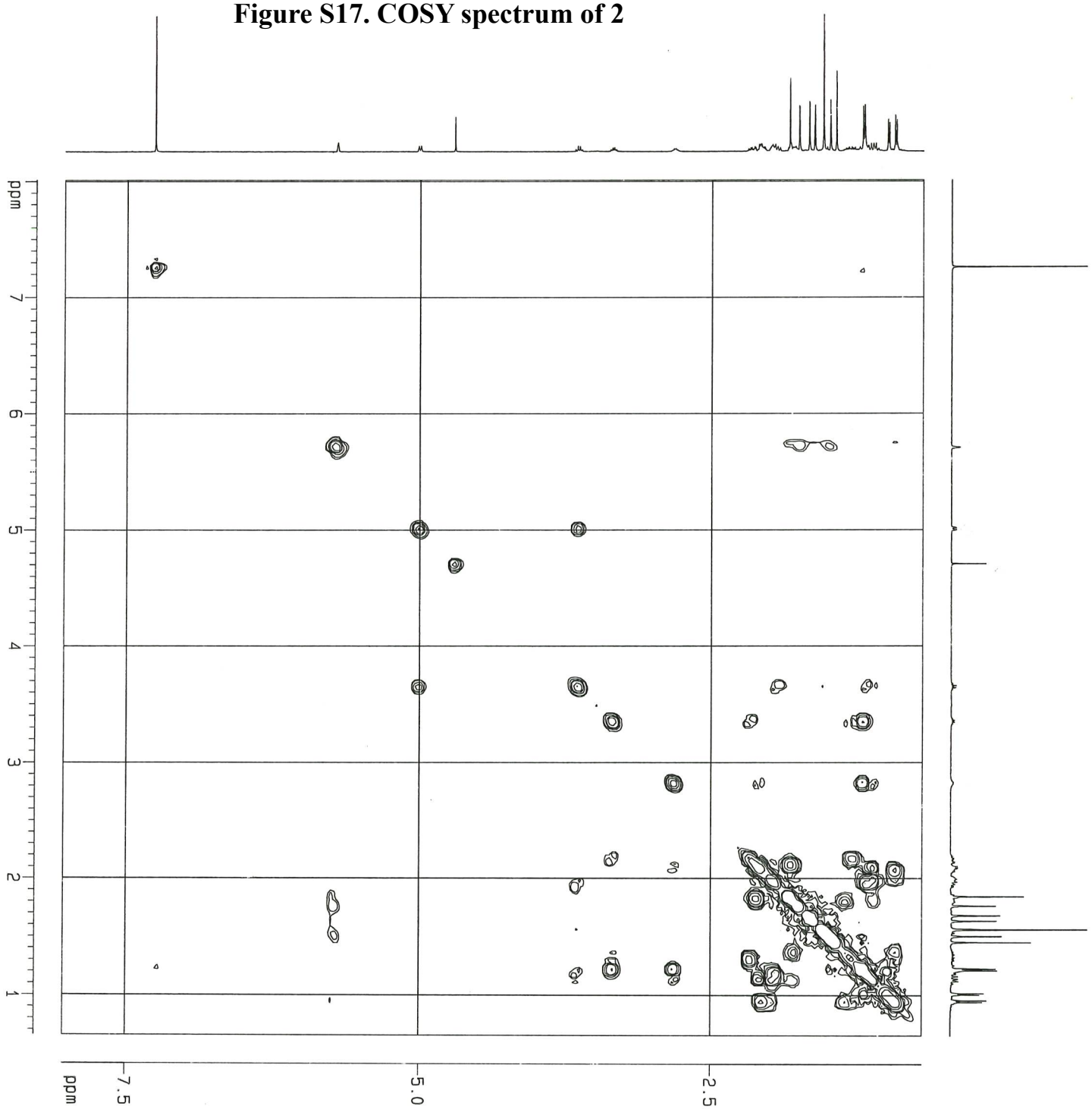
1D NMR plot parameters
CX 20.00 cm
F1P 193.428 ppm
F1 24325.07 Hz
F2P 9.305 ppm
F2 1170.14 Hz
PPMCM 9.20616 ppm/cm
HZCM 1157.74695 Hz/cm

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




```
Current Data Parameters
NAME                  peh2r2m2y
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
SWH                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                        1H
PL2                     0.00 dB
PL12                   22.00 dB
P1                      7.87 usec
SFO1               125.7709955 MHz
NUC1                        13C
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 | peh2r2m2y |
| EXPNO | 5 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20202220 |
| Time | 14.10 |
| INSTRUM | spect |
| PROBHD | 5 mm Multinu |
| PULPROG | cosy45 |
| TD | 1024 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 16 |
| SWH | 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 | 500.1328885 MHz |
| NUC1 | 1H |
| PL1 | 0.00 dB |
| D0 | 0.00000300 sec |
| IN0 | 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 | 500.1300135 MHz |
| WDW | SINE |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 0.00 |

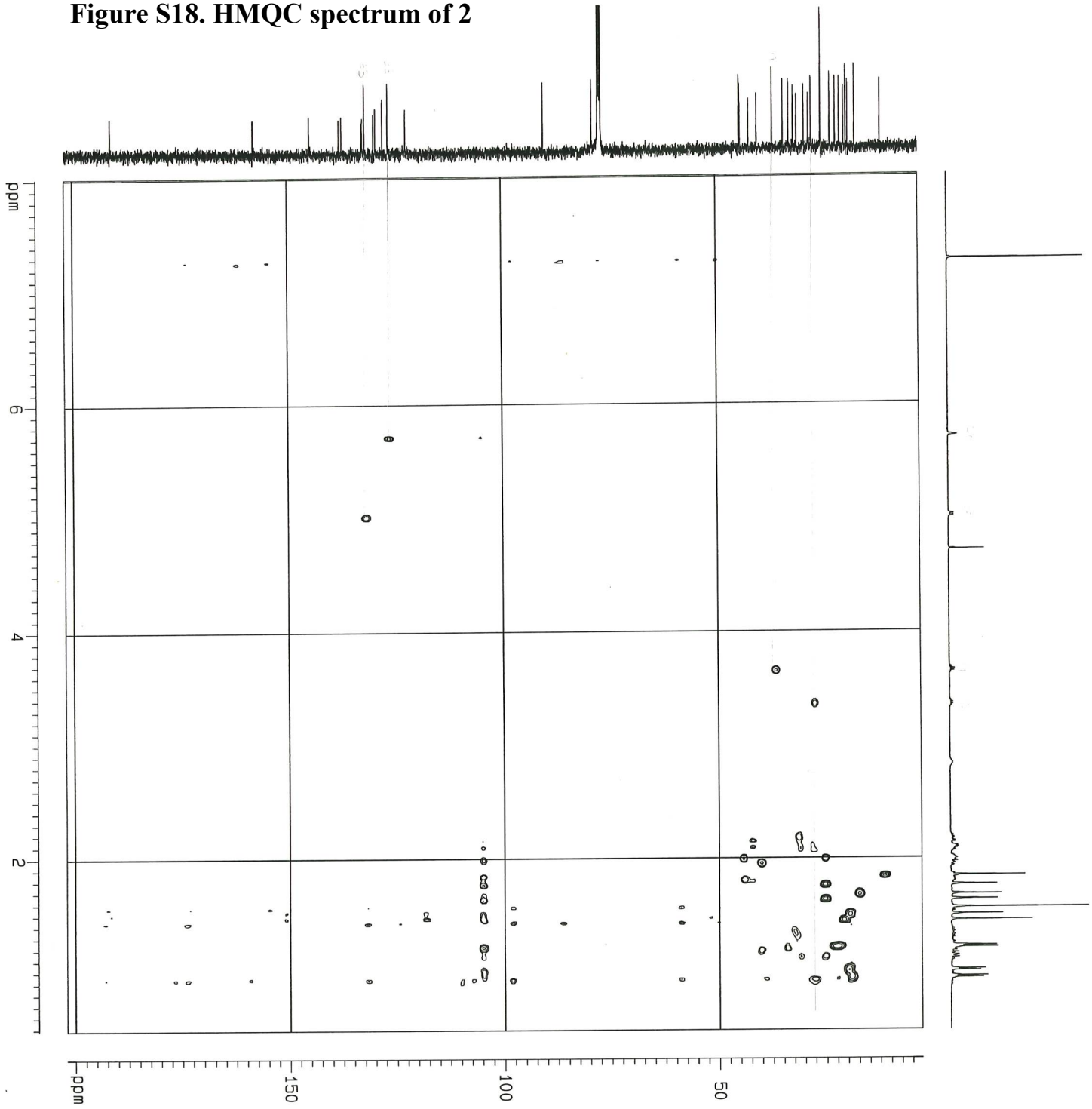
F1 - Processing parameters

| | |
|---|---|
| SI | 512 |
| MC2 | QF |
| SF | 500.1300135 MHz |
| WDW | SINE |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |

2D NMR plot parameters

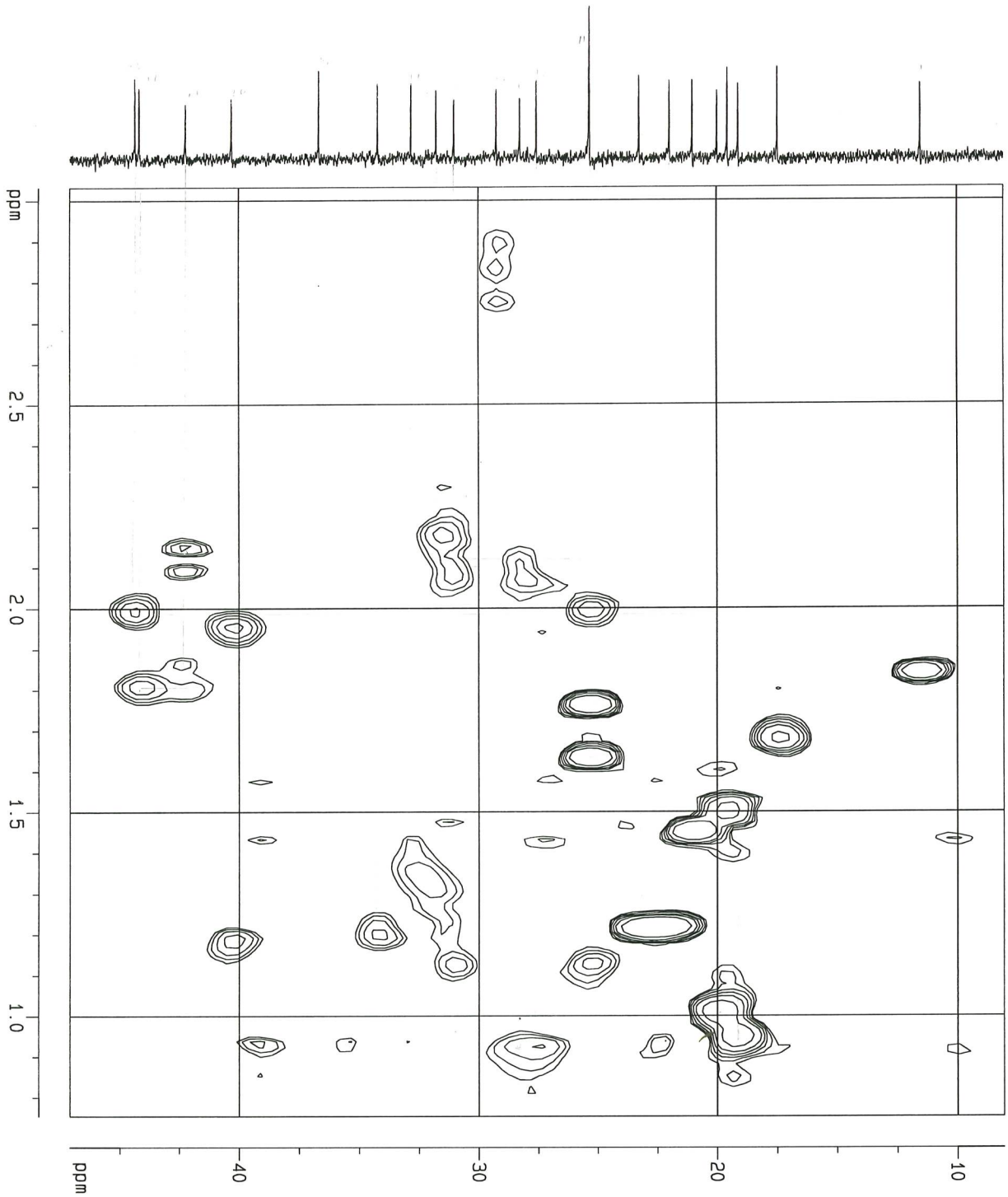
| | |
|---|---|
| CX2 | 15.00 cm |
| CX1 | 15.00 cm |
| F2PLO | 8.012 ppm |
| F2LO | 4007.28 Hz |
| F2PHI | 0.655 ppm |
| F2HI | 327.48 Hz |
| F1PLO | 8.041 ppm |
| F1LO | 4021.44 Hz |
| F1PHI | 0.711 ppm |
| F1HI | 355.79 Hz |
| F2PPMCM | 0.49051 ppm/cm |
| F2HZCM | 245.32004 Hz/cm |
| F1PPMCM | 0.48863 ppm/cm |
| F1HZCM | 244.37650 Hz/cm |

**Figure S18. HMQC spectrum of 2**



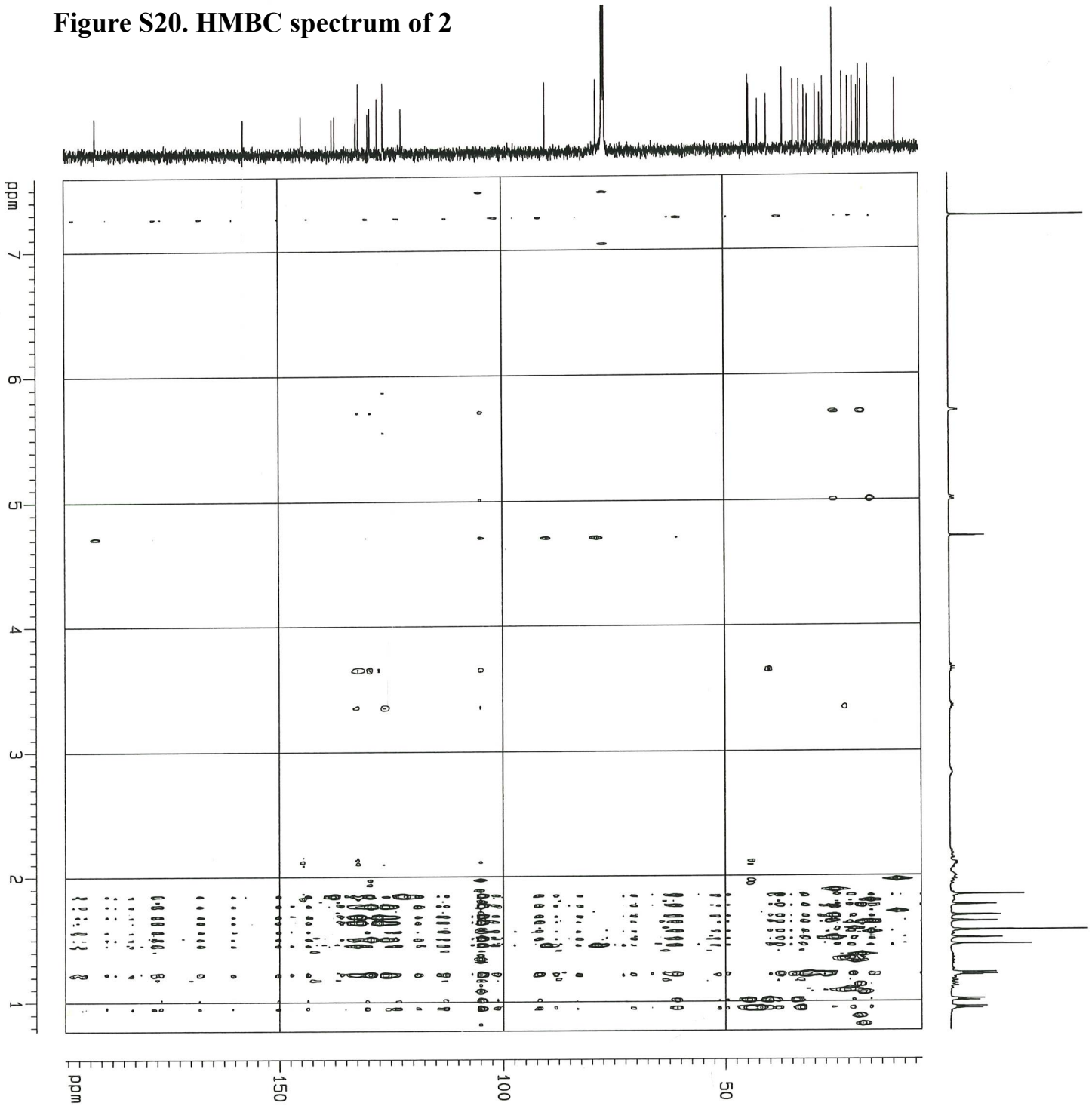

```
Current Data Parameters
NAME                  pen2r2m2y
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_               20020220
Time                    16.02
INSTRUM                 spect
PROBHD     5 mm Multinu
PULPROG                 invbtb
TD                      1024
SOLVENT                 CDCl3
NS                        16
DS                        32
SWH                  7246.377 Hz
FIDRES               7.076540 Hz
AQ                   0.07070560 sec
RG                      3649.1
DW                     69.000 usec
DE                      6.00 usec
TE                     300.0 K
D1                   1.25000000 sec
P1                     10.00 usec
SFO1                500.1328885 MHz
NUC1                      1H
PL1                   0.00 dB
D2                    0.00345000 sec
PL2                   2.00 dB
P2                    20.00 usec
P4                    14.20 usec
SFO2                125.7709955 MHz
NUC2                      13C
D7                    0.40000001 sec
P3                    7.10 usec
D0                    0.00000300 sec
PL12                  22.00 dB
CPDPRG2                   garp
PCPD2                 74.00 usec
IN0                   0.00000825 sec

F1 - Acquisition parameters
NDO                        4
TD                       256
SFO1                125.771 MHz
FIDRES              118.371216 Hz
SW                  240.938 ppm

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

F1 - Processing parameters
SI                        512
MC2                       TPPI
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                 8.012 ppm
F2LO                4007.28 Hz
F2PHI                 0.485 ppm
F2HI                  242.56 Hz
F1PLO                 201.953 ppm
F1LO                25397.18 Hz
F1PHI                 2.876 ppm
F1HI                  361.66 Hz
F2PPMCM              0.50183 ppm/cm
F2HZCM             250.98128 Hz/cm
F1PPMCM             13.27182 ppm/cm
F1HZCM            1669.03406 Hz/cm
```

# Figure S19. HMQC spectrum of 2 (expansion)



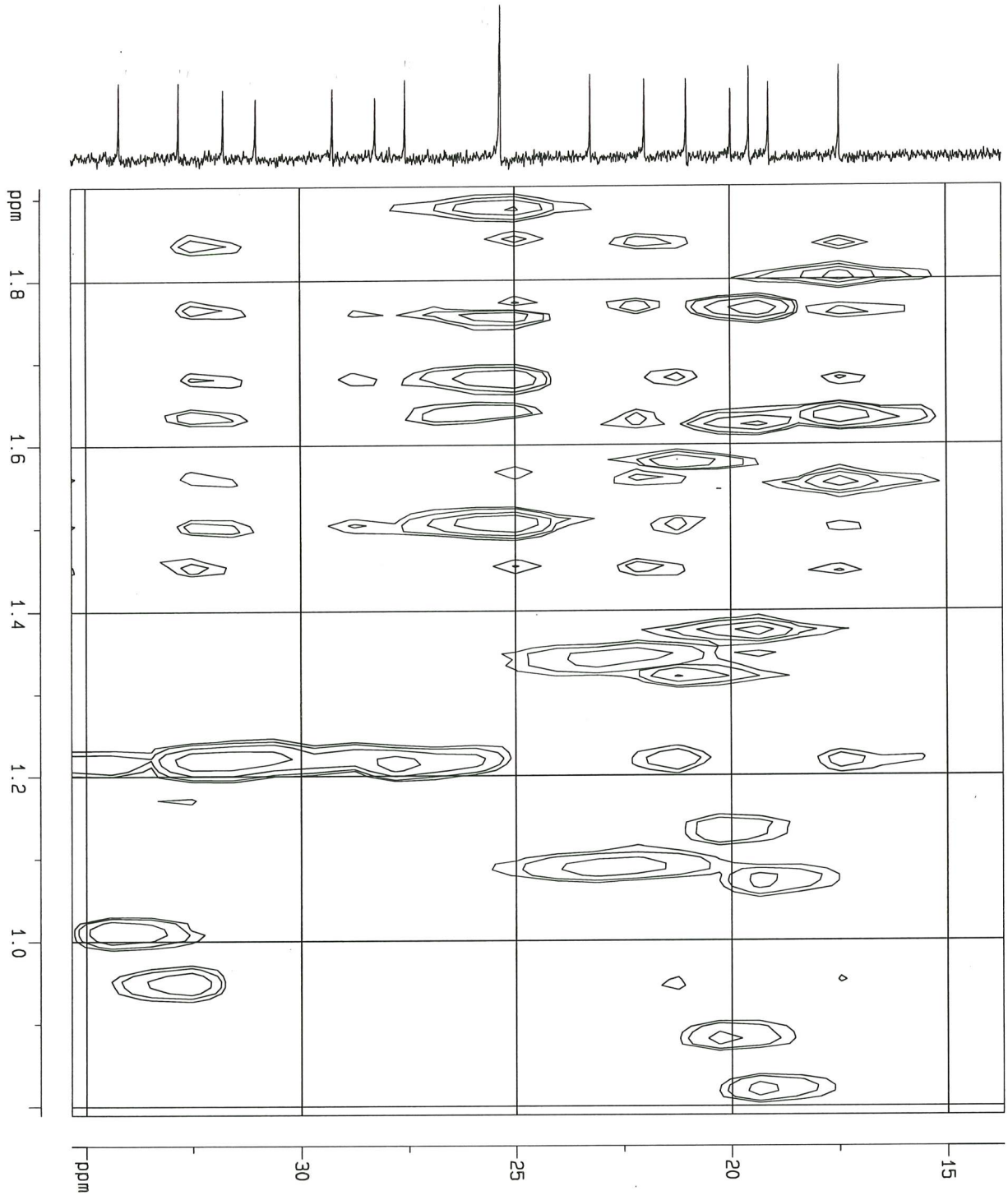

```
Current Data Parameters
NAME                  peh2r2m2y
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_              20020220
Time                   16.02
INSTRUM                spect
PROBHD     5 mm Multinu
PULPROG                inv4gp
TD                       1024
SOLVENT                  CDCl3
NS                         16
DS                         32
SWH                  7246.377 Hz
FIDRES               7.076540 Hz
AQ                   0.0707060 sec
RG                        3649.1
DW                      69.000 usec
DE                       6.00 usec
TE                     300.0 K
D1                     1.25000000 sec
D11                      10.00 msec
P1                     10.00 usec
SFO1                 500.1328885 MHz
NUC1                        1H
PL1                     0.00 dB
PL1W                0.00345000 sec
PL2                     2.00 dB
PL2W                   20.00 usec
P4                     14.20 usec
SFO2                 125.7709955 MHz
NUC2                       13C
D7                   0.40000001 sec
P3                      7.10 usec
D0                   0.00000300 sec
PL12                   22.00 dB
CPDPRG2                garp
PCPD2                  74.00 usec
IN0                  0.00000825 sec

F1 - Acquisition parameters
NDO                        4
TD                       256
SFO1                 125.771 MHz
FIDRES               118.371216 Hz
SW                   240.938 ppm

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

F1 - Processing parameters
SI                        512
MC2                      TPPI
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                    3.032 ppm
F2LO                  1516.34 Hz
F2PHI                    0.754 ppm
F2HI                  377.02 Hz
F1PLO                  47.115 ppm
F1LO                 5925.11 Hz
F1PHI                    8.053 ppm
F1HI                 1012.71 Hz
F2PPMCM                  0.15187 ppm/cm
F2HZCM                75.95486 Hz/cm
F1PPMCM                  2.60416 ppm/cm
F1HZCM               327.49368 Hz/cm
```

**Figure S20. HMBC spectrum of 2**




```
Current Data Parameters
NAME                  peh2r2m2y
EXPNO                        15
PROCNO                        1

F2 - Acquisition Parameters
Date_               20020220
Time                    18.12
INSTRUM                 spect
PROBHD     5 mm Multinu
PULPROG             inv4ip1rnd
TD                        4096
SOLVENT                 CDCl3
NS                          104
DS                           32
SWH                 7246.377 Hz
FIDRES              1.769135 Hz
AQ                  0.2826740 sec
RG                       1149.4
DW                     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
D2                  0.00300000 sec
P3                    7.10 usec
SFO2               125.7709955 MHz
NUC2                       13C
PL2                   2.00 dB
D6                  0.05000000 sec
D0                  0.00000300 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                  240.938 ppm

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

F1 - Processing parameters
SI                         256
MC2                         QF
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                 7.595 ppm
F2LO                3798.52 Hz
F2PHI                 0.768 ppm
F2HI                384.09 Hz
F1PLO               198.188 ppm
F1LO                24923.69 Hz
F1PHI                 6.170 ppm
F1HI                775.96 Hz
F2PPMCM             0.45514 ppm/cm
F2HZCM              227.62871 Hz/cm
F1PPMCM              12.80118 ppm/cm
F1HZCM              1609.84651 Hz/cm
```

# Figure S21. HMBC spectrum of 2 (expansion)




Current Data Parameters

| | |
|---|---|
| NAME | peh2m2y |
| EXPNO | 15 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20020220 |
| Time | 18.12 |
| INSTRUM | spect |
| PROBHD | 5 mm Multinu |
| PULPROG | inv4ip1rnd |
| TD | 4096 |
| SOLVENT | CDCl3 |
| NS | 104 |
| DS | 32 |
| SWH | 7246.377 Hz |
| FIDRES | 1.769135 Hz |
| RG | 1149.4 |
| DW | 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 |
| D2 | 0.00300000 sec |
| P3 | 7.10 usec |
| SFO2 | 125.7709955 MHz |
| NUC2 | 13C |
| PL2 | 2.00 dB |
| D6 | 0.05000000 sec |
| D0 | 0.00000300 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 | 240.938 ppm |

F2 - Processing parameters

| | |
|---|---|
| SI | 2048 |
| SF | 500.1300135 MHz |
| WDW | GSINE |
| SSB | 4 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

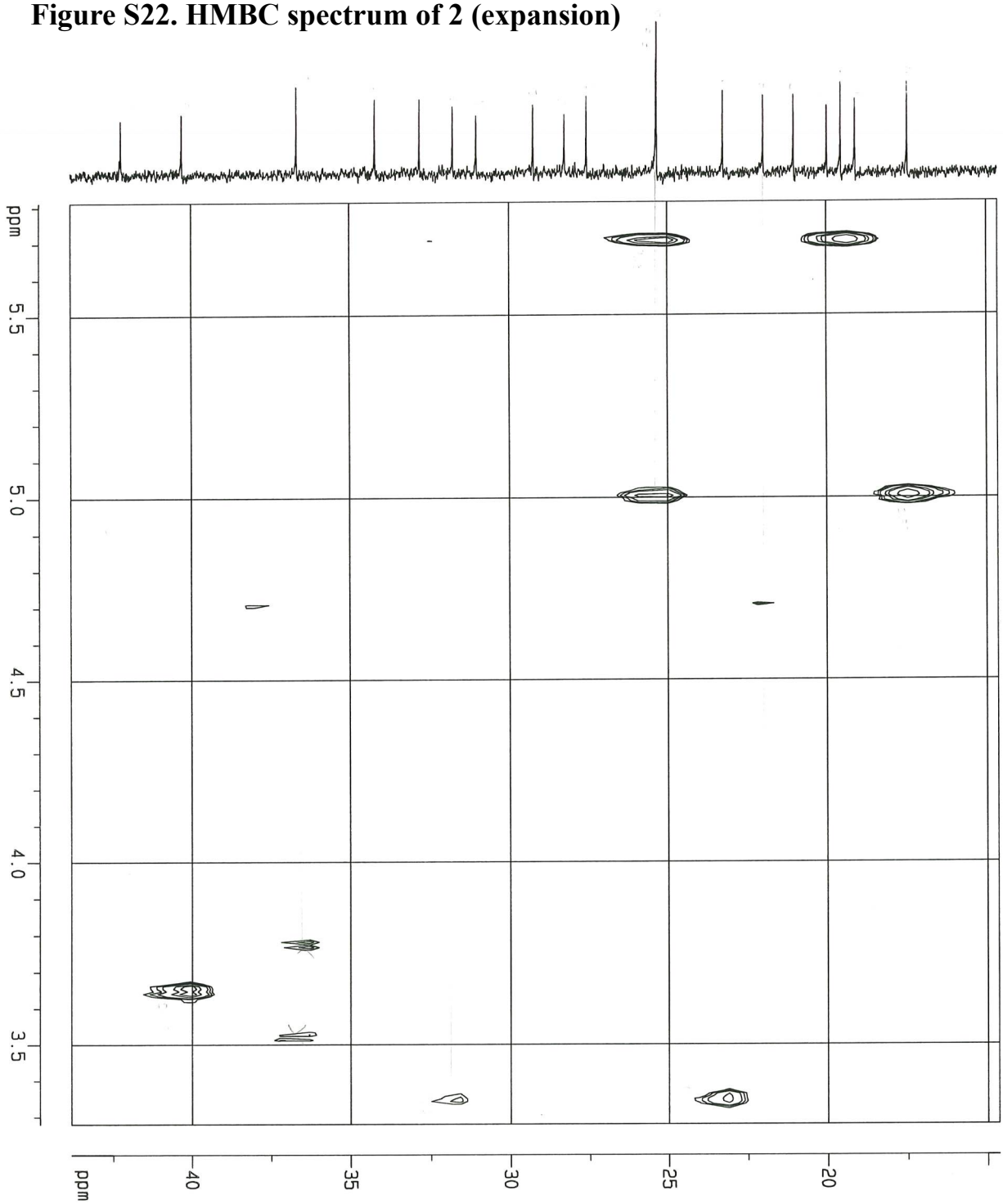
F1 - Processing parameters

| | |
|---|---|
| SI | 256 |
| MC2 | QF |
| SF | 125.7577906 MHz |
| WDW | SINE |
| SSB | 2 |
| LB | 0.00 Hz |
| GB | 0 |

2D NMR plot parameters

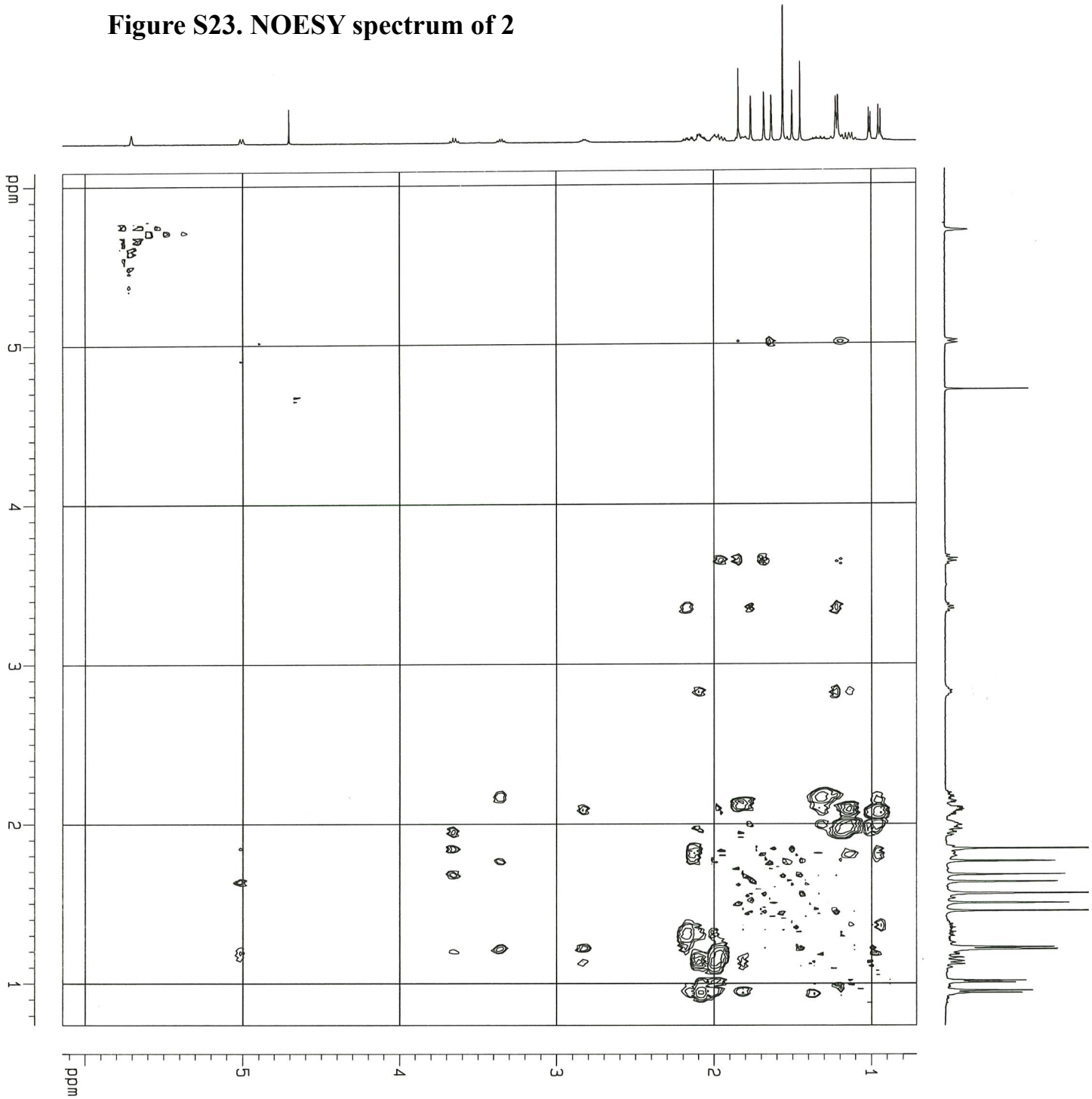
| | |
|---|---|
| CX2 | 15.00 cm |
| CX1 | 15.00 cm |
| F2PLO | 1.914 ppm |
| F2LO | 957.28 Hz |
| F2PHI | 0.789 ppm |
| F2HI | 394.71 Hz |
| F1PLO | 35.349 ppm |
| F1LO | 4445.47 Hz |
| F1PHI | 13.700 ppm |
| F1HI | 1722.93 Hz |
| F2PPMCM | 0.07499 ppm/cm |
| F2HZCM | 37.50566 Hz/cm |
| F1PPMCM | 1.44327 ppm/cm |
| F1HZCM | 181.50252 Hz/cm |

# Figure S22. HMBC spectrum of 2 (expansion)




```
                    Current Data Parameters
NAME                      peh2m2y
EXPNO                          15
PROCNO                          1

                F2 - Acquisition Parameters
Date_                    20020220
Time_                       18.12
INSTRUM                      spect
PROBHD     5 mm Multinu
PULPROG                 inv4ip1rnd
TD                            4096
SOLVENT                      CDCl3
NS                             104
DS                              32
SWH                      7246.377 Hz
FIDRES                   1.769135 Hz
AQ                       0.2826740 sec
RG                          1149.4
DW                       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
D2                       0.00300000 sec
P3                         7.10 usec
SFO2                  125.7709955 MHz
NUC2                           13C
PL2                         2.00 dB
D6                       0.05000000 sec
D0                       0.00000300 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                       240.938 ppm

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

                F1 - Processing parameters
SI                           256
MC2                           QF
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                      5.812 ppm
F2LO                    2906.66 Hz
F2PHI                     3.280 ppm
F2HI                    1640.18 Hz
F1PLO                     43.821 ppm
F1LO                    5510.81 Hz
F1PHI                     14.642 ppm
F1HI                    1841.31 Hz
F2PPMCM                 0.16885 ppm/cm
F2HZCM                  84.44671 Hz/cm
F1PPMCM                 1.94528 ppm/cm
F1HZCM                  244.63383 Hz/cm
```

**Figure S23. NOESY spectrum of 2**




Current Data Parameters
NAME pen2m2y
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020222
Time 18.49
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG noesytp
TD 1024
SOLVENT CDC13
NS 72
DS 16
SWH 7246.377 Hz
FIDRES 7.076540 Hz
AQ 0.0707060 sec
RG 128
DW 69.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
P1 10.00 usec
SFO1 500.1328885 MHz
NUC1 1H
PL1 0.00 dB
D0 0.00000300 sec
DB 0.41000000 sec
INO 0.00006900 sec

F1 - Acquisition parameters
NDO 2
TD 256
SFO1 500.1329 MHz
FIDRES 2B.306160 Hz
SW 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
F2PLO 6.090 ppm
F2LO 3045.56 Hz
F2PHI 0.741 ppm
F2HI 370.63 Hz
F1PLO 6.146 ppm
F1LO 3073.88 Hz
F1PHI 0.713 ppm
F1HI 356.49 Hz
F2PPMCM 0.35656 ppm/cm
F2HZCM 178.32881 Hz/cm
F1PPMCM 0.36222 ppm/cm
F1HZCM 181.15941 Hz/cm

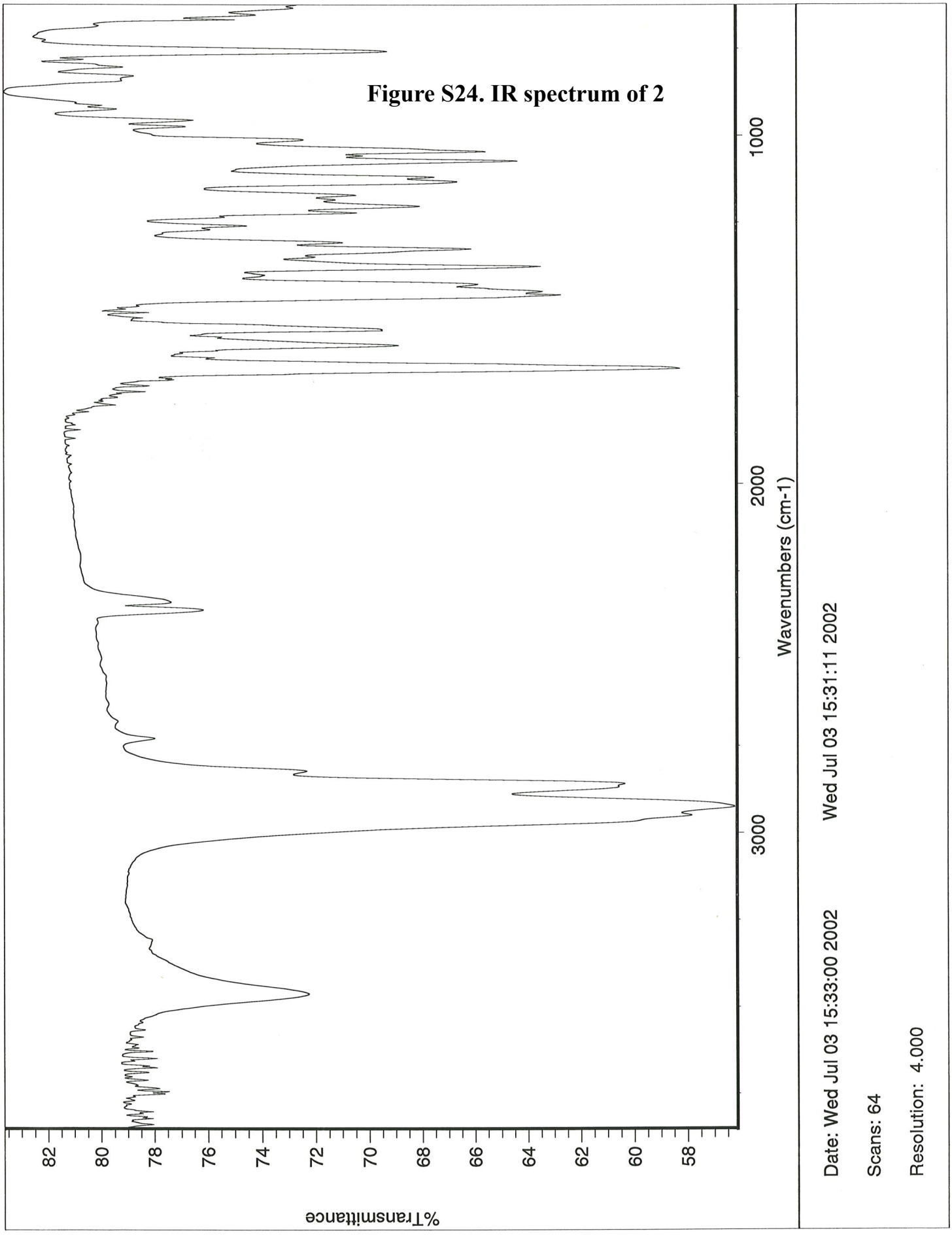



**Figure S24. IR spectrum of 2**