

Supporting Information to:**Diterpenoids from *Casearia sylvestris***

Wei Wang¹, Jianpin Zhao¹, Yan-hong Wang¹, Troy A. Smillie¹, Xing-Cong Li¹, Ikhlas A. Khan^{1,2}

Affiliation

¹ National Center for Natural Products Research, Research Institute of Pharmaceutical Sciences, University of Mississippi, MS, USA

² Department of Pharmacognosy, School of Pharmacy, University of Mississippi, MS, USA

Correspondence

Prof. Dr. Ikhlas A. Khan

National Center for Natural Products Research

School of Pharmacy

University of Mississippi

MS 38677

USA

Tel.: +1/662/915 7821

Fax: +1/662/915 7989

ikhlan@olemiss.edu

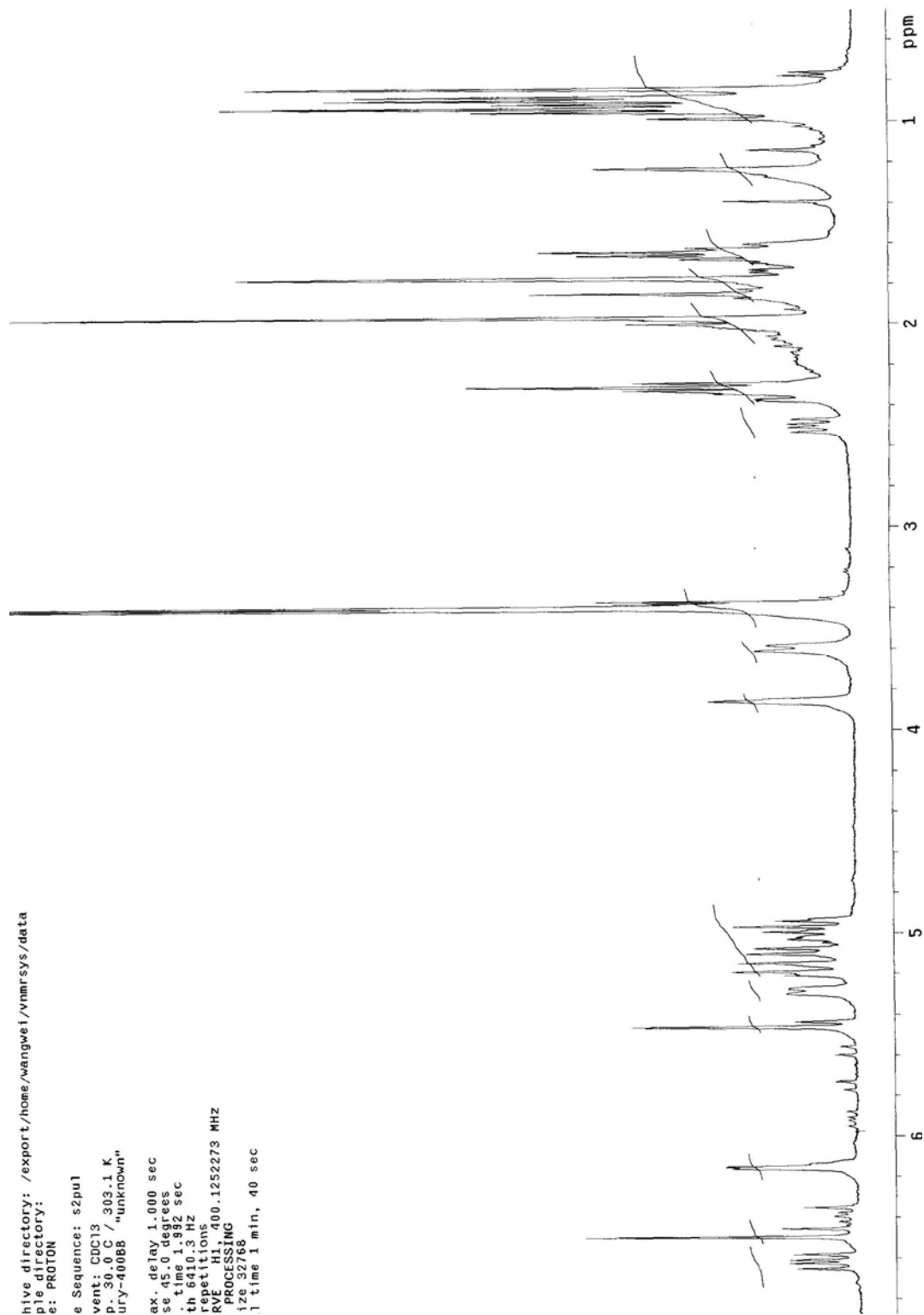


Fig. 1S $^1\text{H-NMR}$ spectrum of compound **1** (CDCl_3 , 400 MHz).

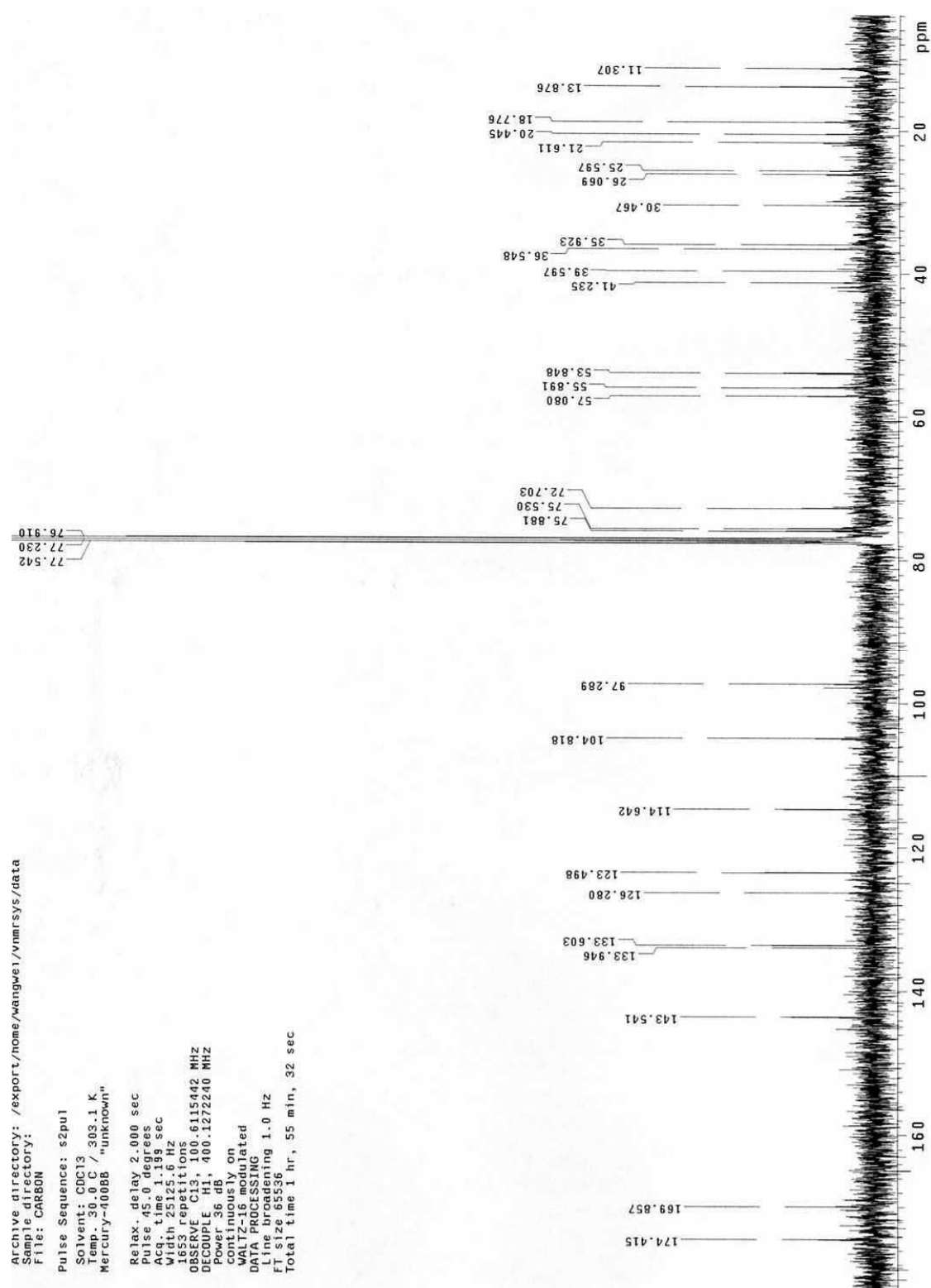


Fig. 2S ^{13}C -NMR spectrum of compound **1** (CDCl_3 , 100 MHz).

Archive directory: /export/home/wangwei/vnmrsys/data
 Sample directory:
 File: CARBON

Pulse Sequence: DEPT

Solvent: CDC13
 Temp. 30.0 C / 303.1 K
 Mercury-400BB "unknown"

Relax. delay 2.000 sec

Pulse 90.0 degrees

Acq. time 1.189 sec

Width 25125.6 Hz

1000 repetitions

OBSERVE C13, 100.6115420 MHz

DECOUPLE H1, 400.1272240 MHz

Low pass during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 58 min, 30 sec

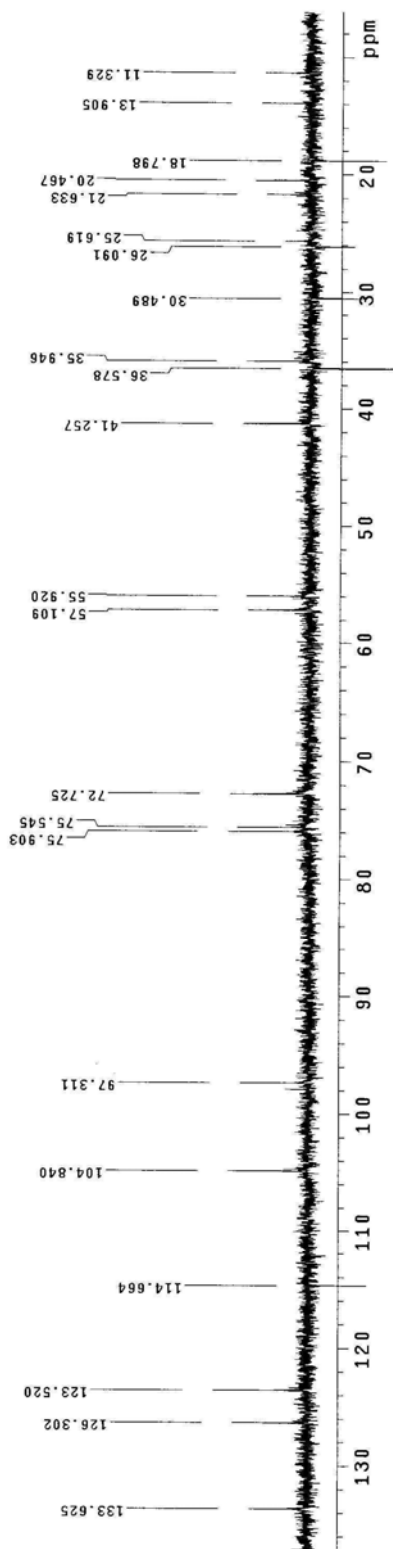


Fig. 3S DEPT spectrum of compound **1**.

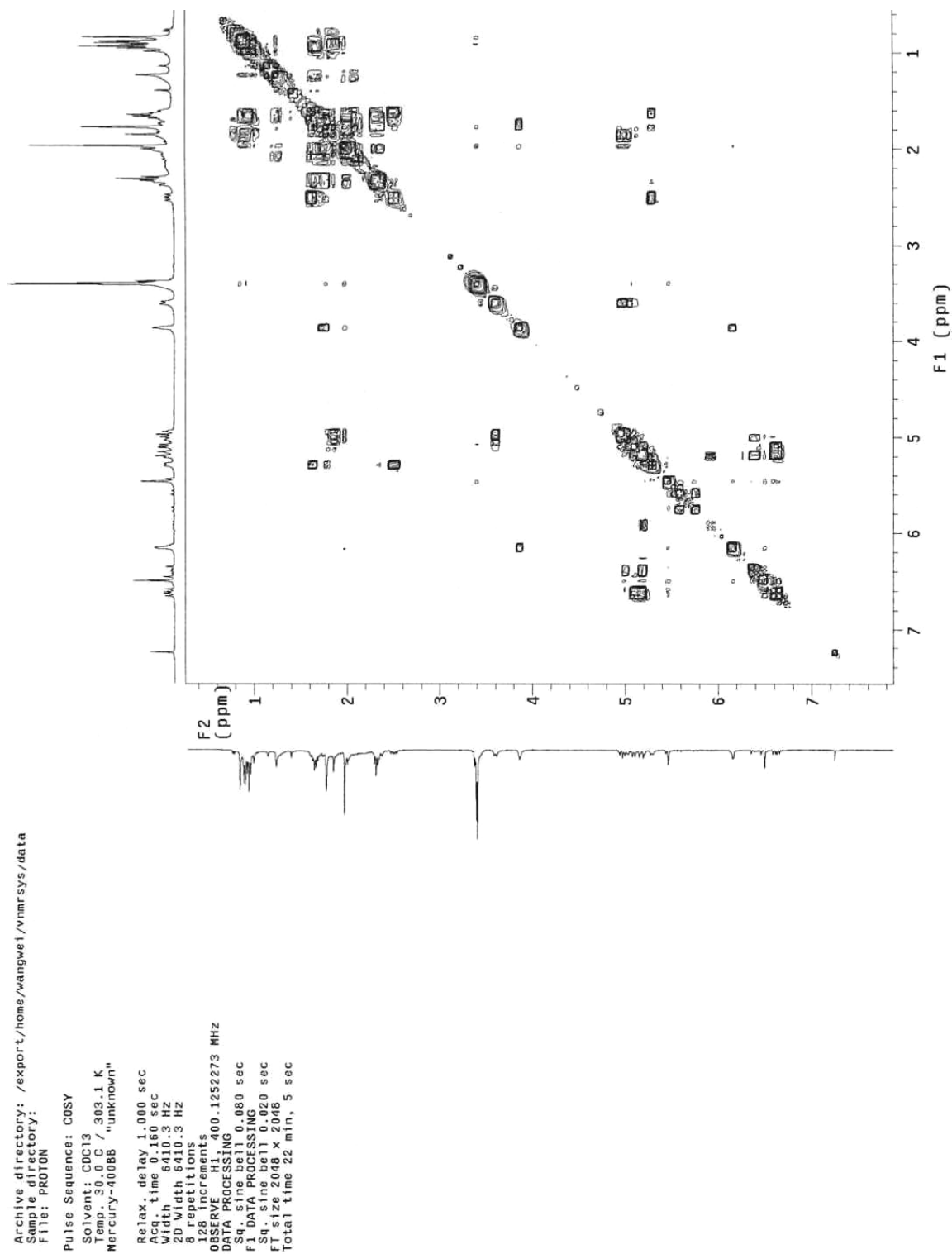


Fig. 4S COSY spectrum of compound **1**.

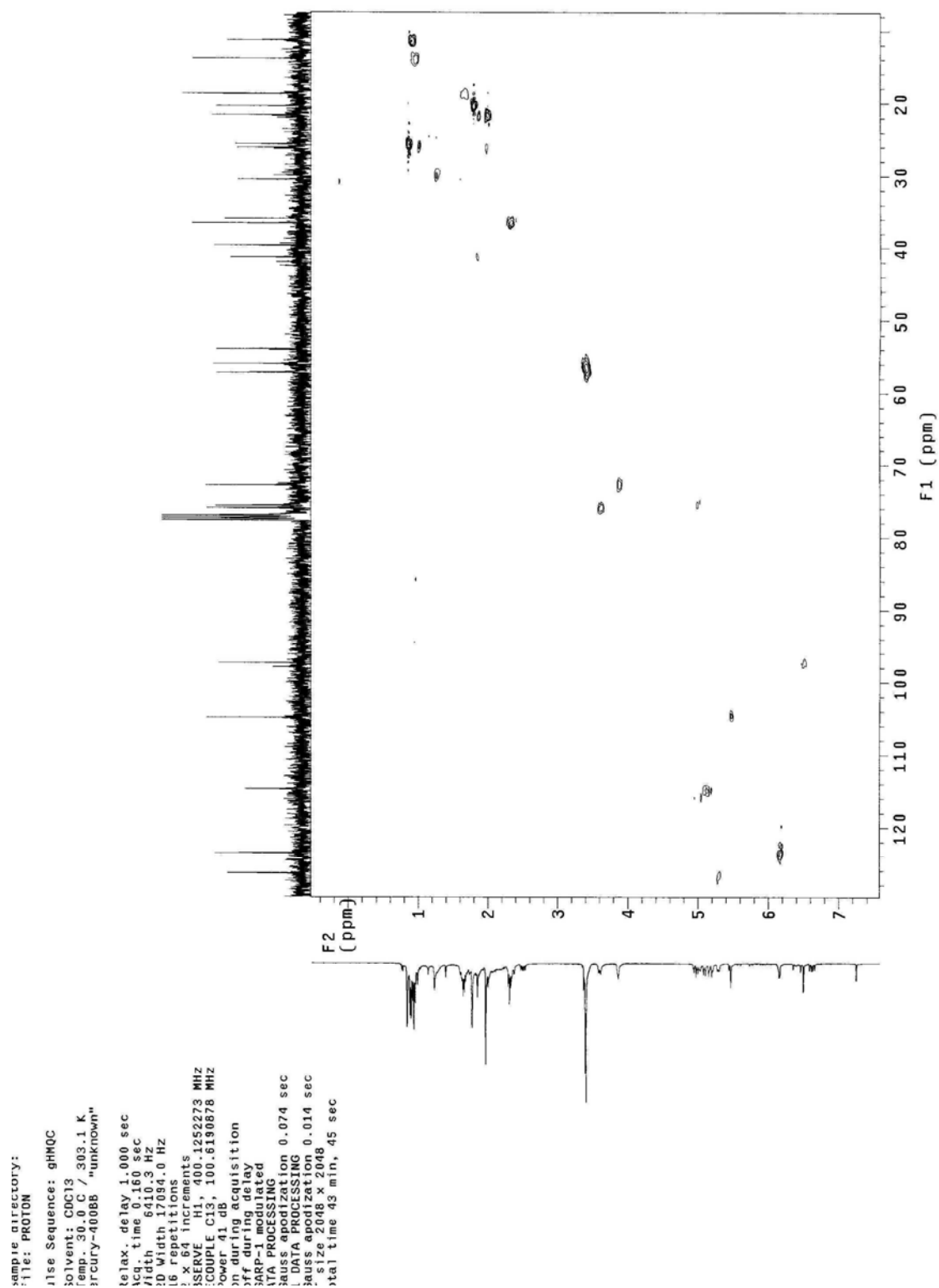


Fig. 5S HMQC spectrum of compound **1**.

```
-chive directory: /export/home/wangwei/vnmrSYS/data  
Sample directory:  
File: PROTON
```

```
Use Sequence: gHMBC
```

```
1) Solvent: CDC13
```

```
2) Temp.: 30.0 C / 303.1 K
```

```
3) Acq. File: "unknown"
```

```
4) Max. delay: 1.000 sec
```

```
5) Acq. time: 0.160 sec
```

```
6) Width: 6410.3 Hz
```

```
7) Width: 24154.6 Hz
```

```
8) Repetitions:
```

```
9) Repetition:
```

```
10) Frequency: 400.1252273 MHz
```

```
11) Name: "A PROCESSING"
```

```
12) Line: bell 0.080 sec
```

```
13) DATA PROCESSING
```

```
14) Line: bell 0.008 sec
```

```
15) Size: 2048 x 2048
```

```
16) .al time: 45 min, 0 sec
```

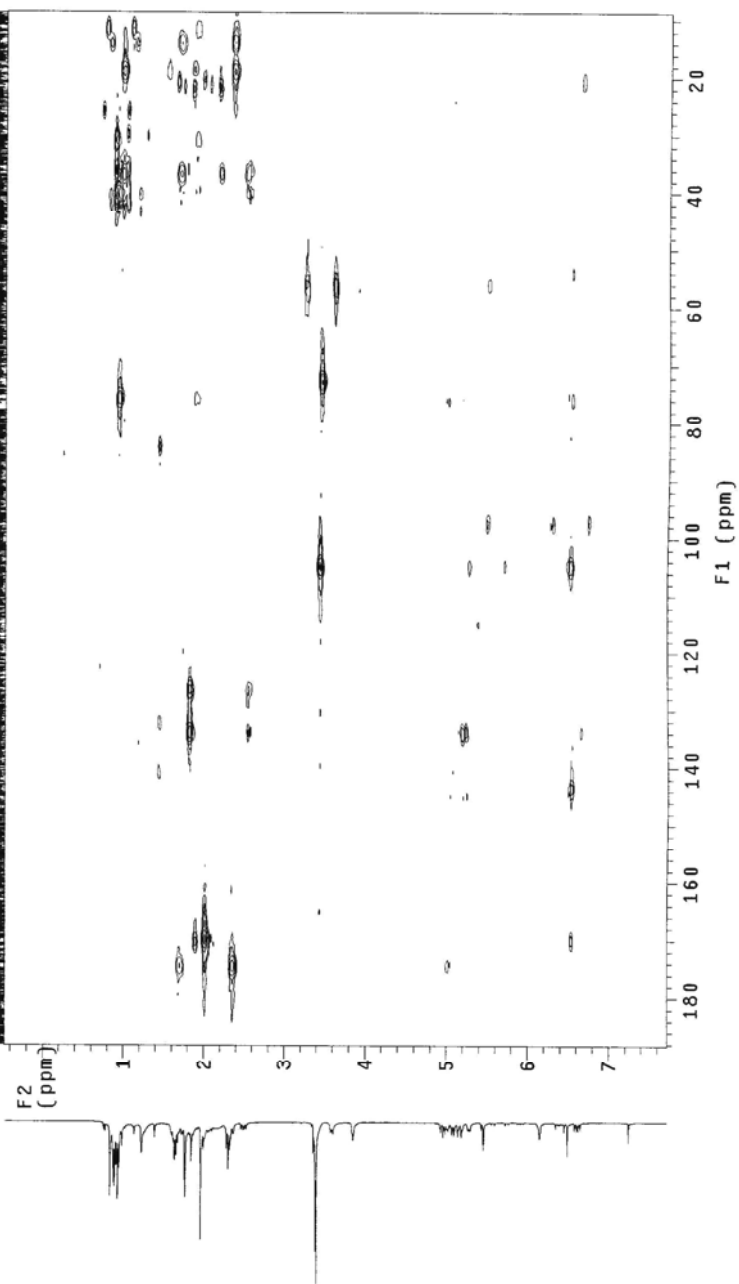


Fig. 6S HMBC spectrum of compound 1.

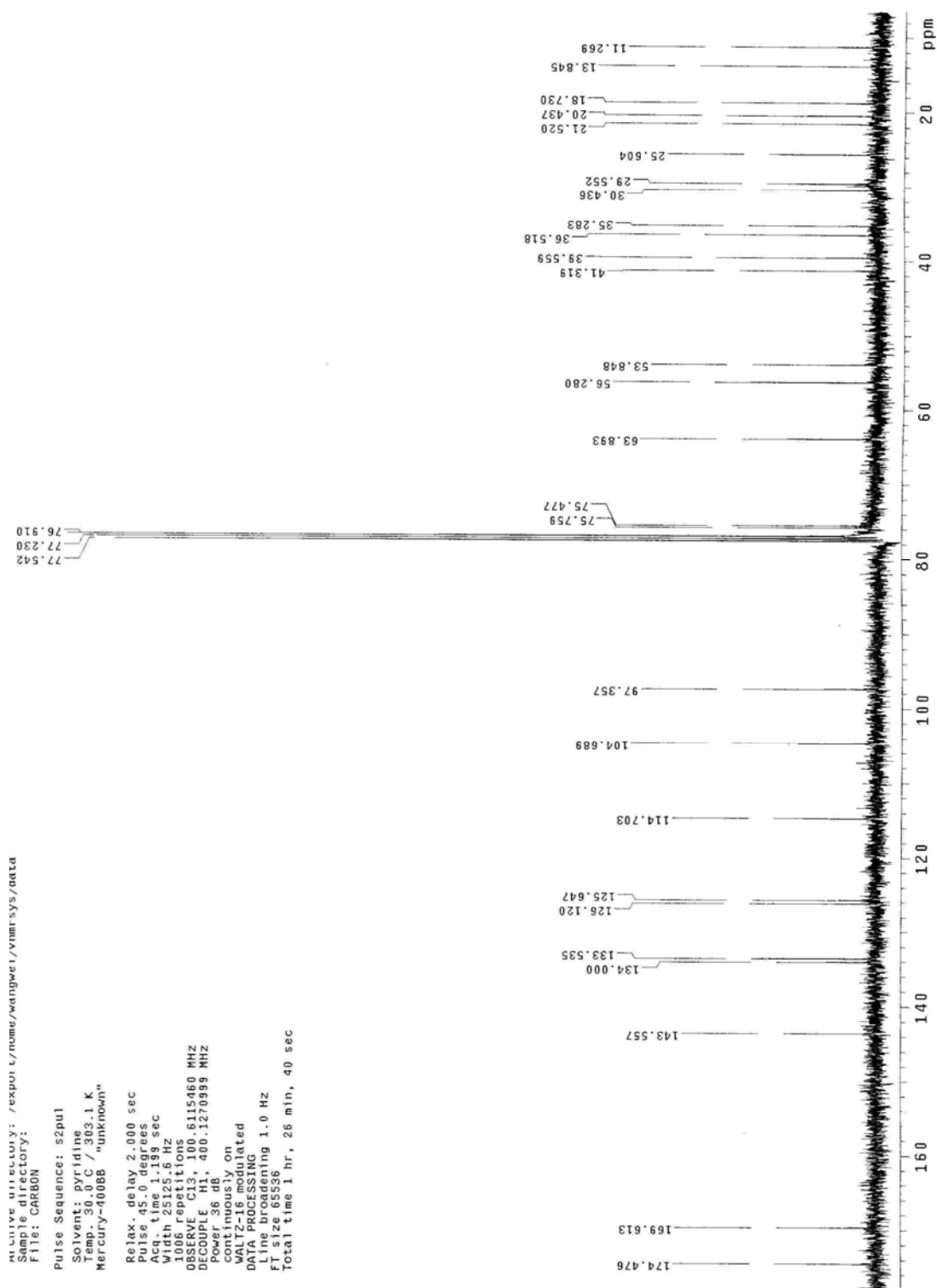


Fig. 8S ^{13}C -NMR spectrum of compound **2** (CDCl_3 , 100 MHz).

Archive directory: /export/home/wangwei/vnmr/sys/data
 Sample directory:
 File: CARBON

Pulse Sequence: DEPT

Solvent: pyridine
 Temp.: 30.0 C / 303.1 K
 Mercury-400BB "unknown"

Relax. delay 2.000 sec
 Pulse 90.0 degrees
 Acq. time 1.189 sec

Width 25125.6 Hz

302 repetitions

OBSERVE C13, 100.6115460 MHz

PROBHD 5mm QNP1HHR

Power 56 dB, 400.1270999 MHz

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 4.0 Hz

FT size 65536

Total time 35 min 18 sec

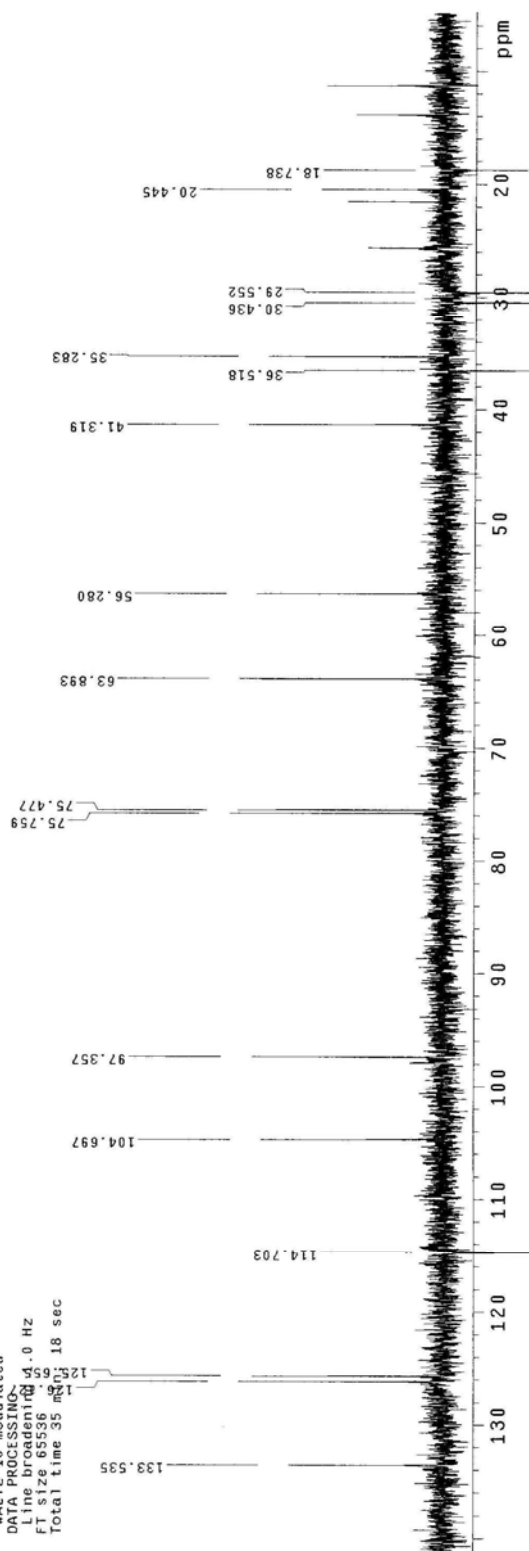


Fig. 9S DEPT spectrum of compound **2**.

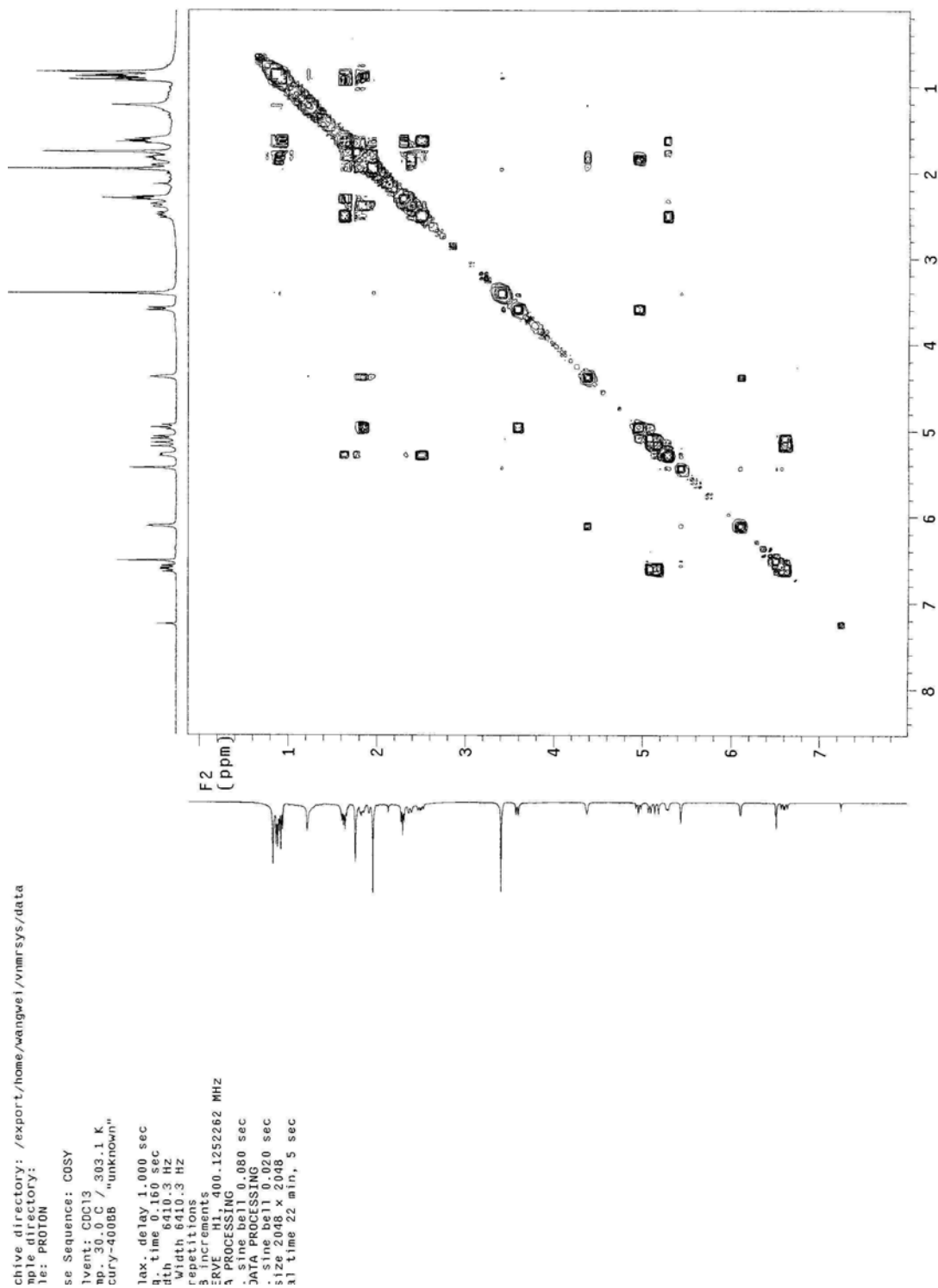


Fig. 10S COSY spectrum of compound 2.

hive directory: /export/home/wangwei/vnmrsys/data
 ple directory:
 e: PROTON

e Sequence: gHMQC

vent: CDC13
 p. 30.0 C / 303.1 K
 ury-40088 "unknown"

ax. delay 1.000 sec

time 0.150 sec

th 6410.3 Hz

Width 17094.0 Hz

repetitions

64 increments

RVE H1, 400.1252262 MHz

UPLE C13, 100.6190878 MHz

er 41 dB

during acquisition

during delay

P-1 modulated

PROCESSING

SS APROBATION 0.074 sec

STA PROCESSING

Size 2048 X 2048

l time 43 min, 45 sec

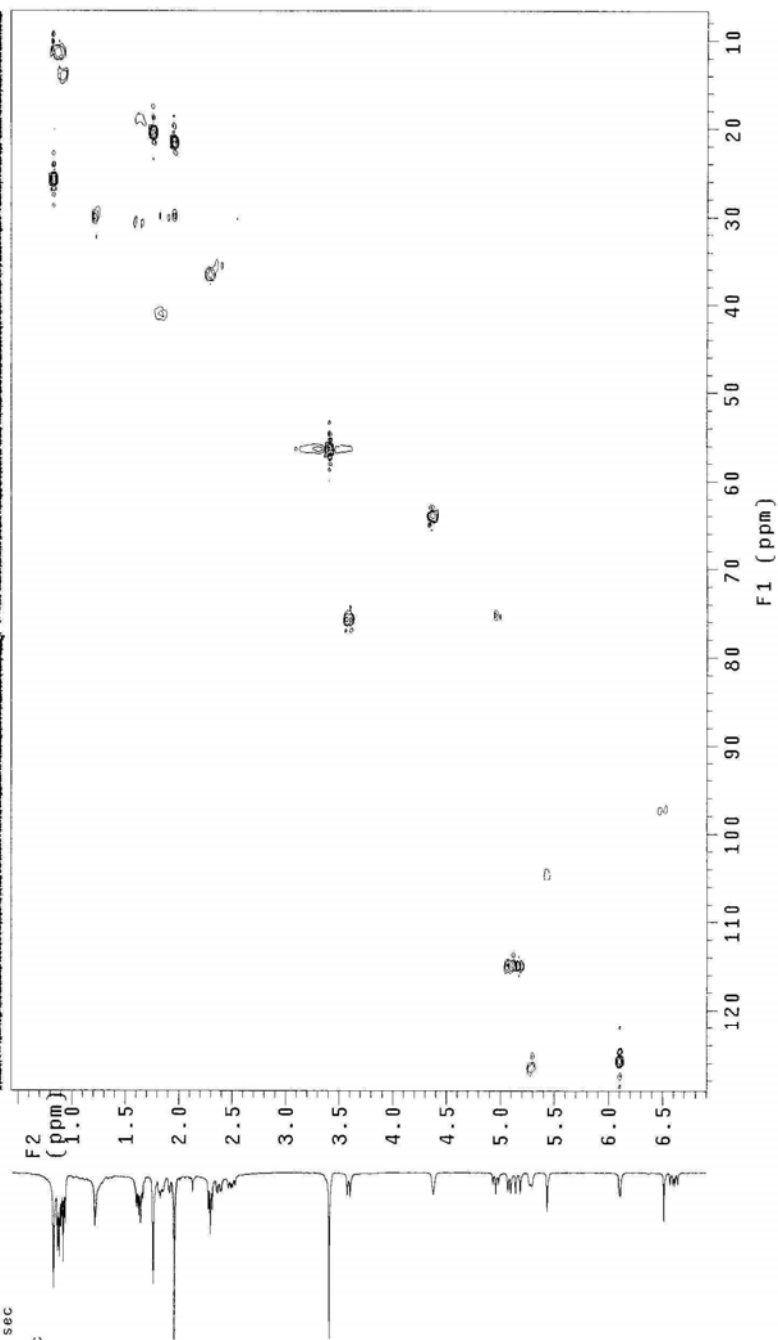


Fig. 11S HMQC spectrum of compound **2**.

```
:hive directory: /export/home/wangwei/vnmrsws/data
:pile directory:
:ie: PROTON
```

```
:ie Sequence: gHMBC
```

```
:ivent: CDC13
```

```
:ip: 30.0 C / 303.1 K
```

```
:isury: 400BB "unknown"
```

```
:iax. delay: 1.000 sec
```

```
:it. time: 0.160 sec
```

```
:lth: 6410.3 Hz
```

```
:Width: 24154.6 Hz
```

```
:repetitions:
```

```
:i increments:
```

```
:RVE: H1, 400.1252262 MHz
```

```
:A PROCESSING
```

```
:ie bell: 0.080 sec
```

```
:ATA PROCESSING
```

```
:t. be: 2048 X 2048
```

```
:il. time: 45 min, 0 sec
```

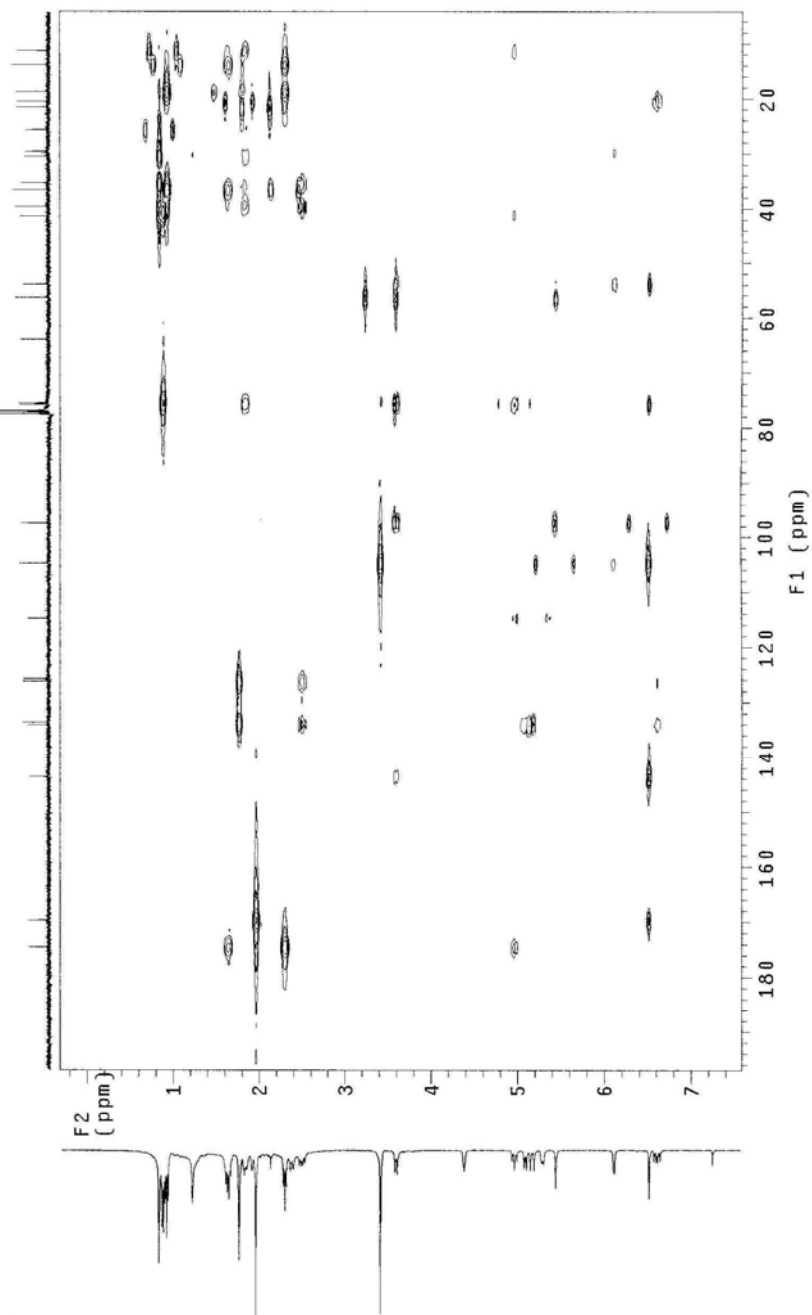


Fig. 12S HMBC spectrum of compound **2**.

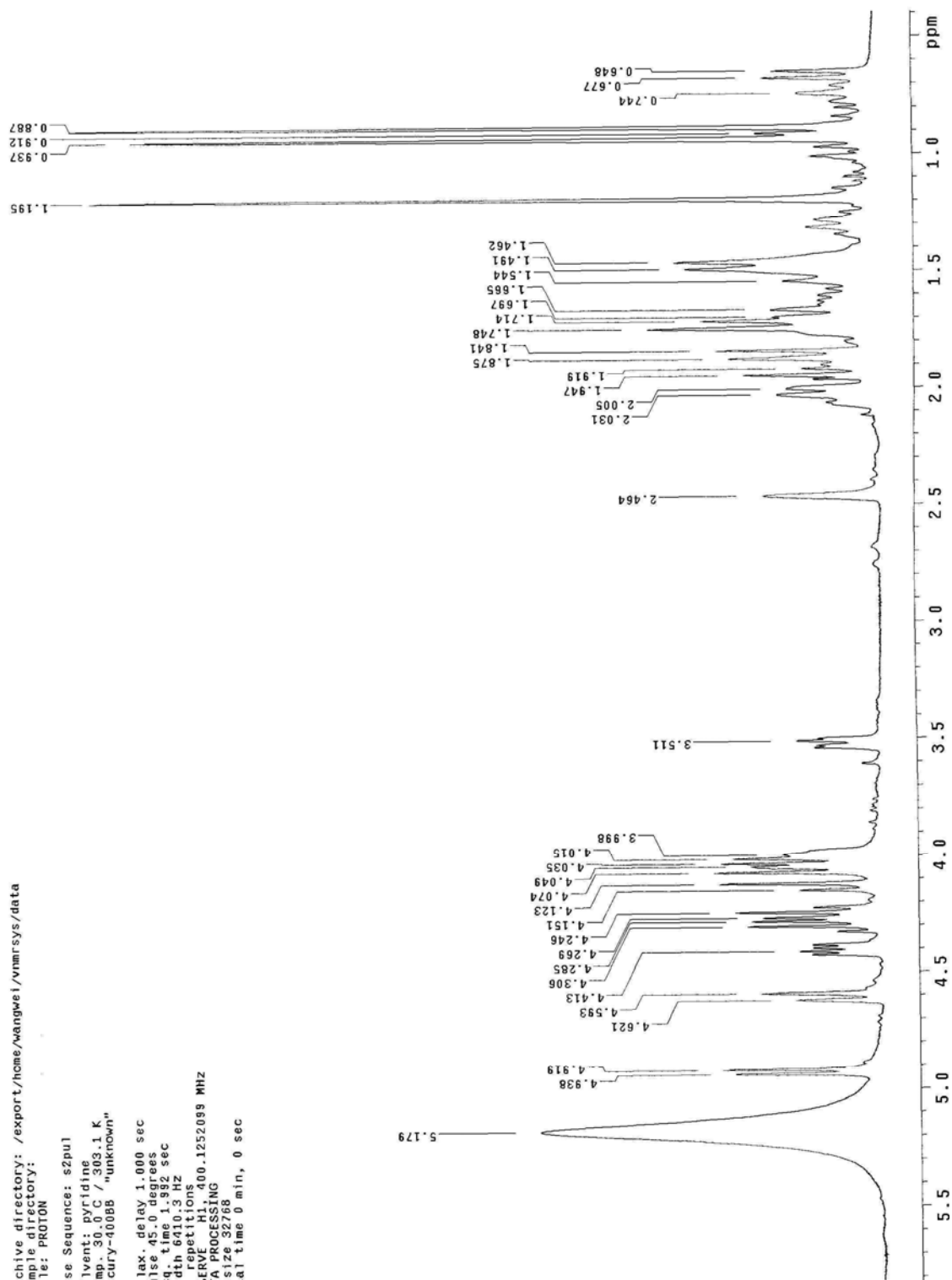


Fig. 13S $^1\text{H-NMR}$ spectrum of compound **3** (CDCl_3 , 400 MHz).

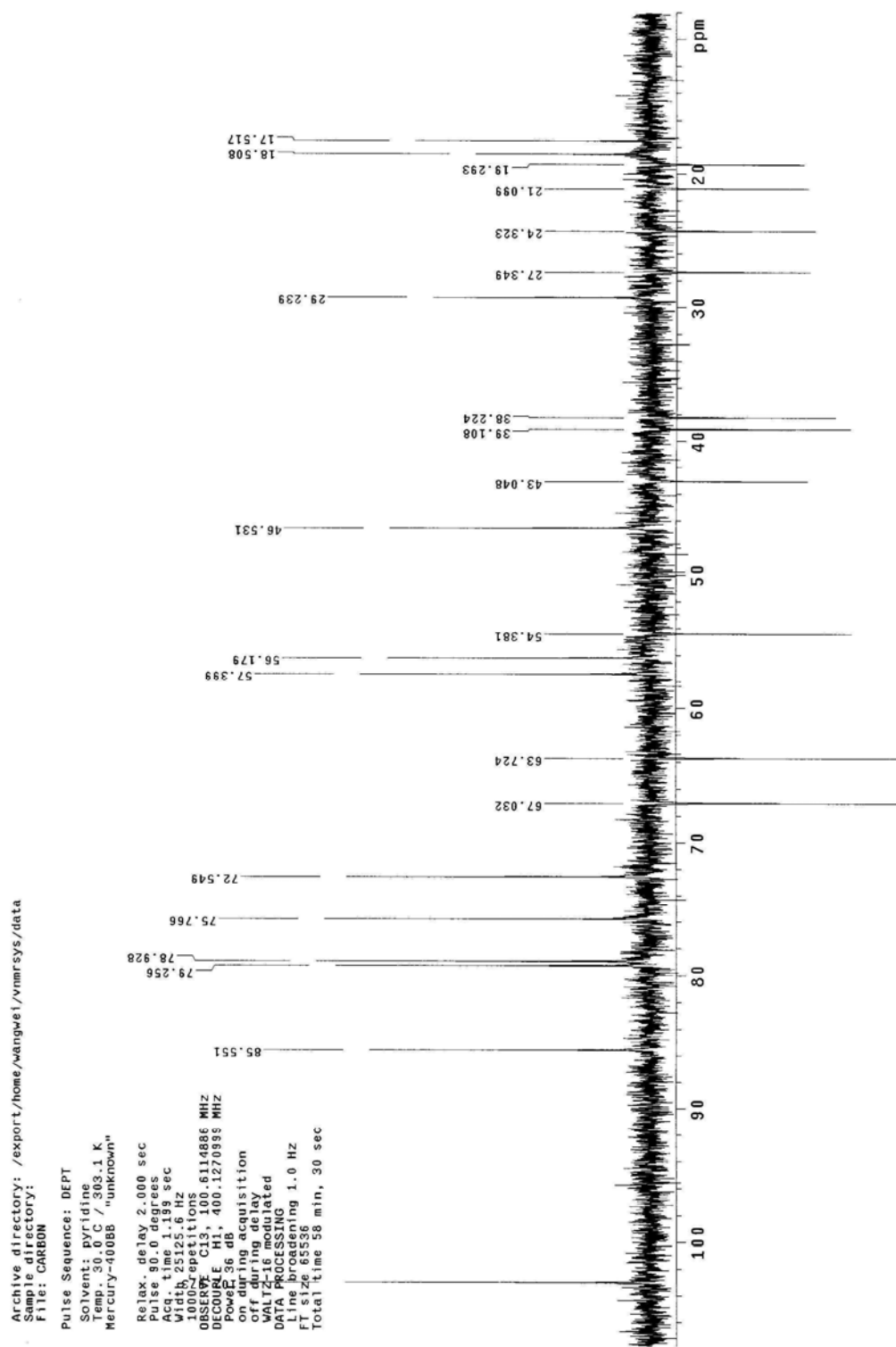


Fig. 15S DEPT spectrum of compound 3.

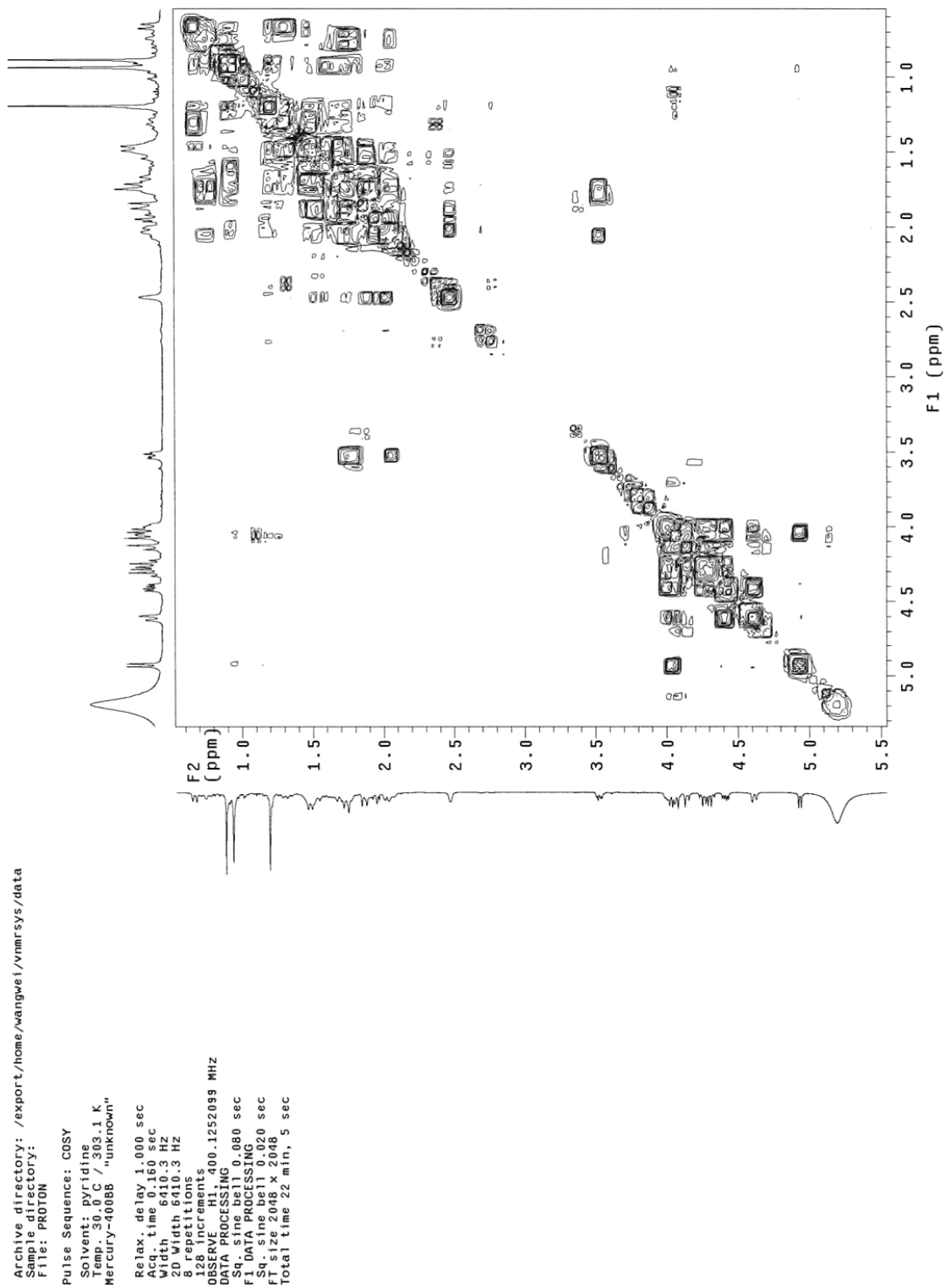


Fig. 16S COSY spectrum of compound **3**.

archive directory: /export/home/wangwei/vnmrsys/data
 sample directory:
 file: PROTON

file Sequence: ghmqc
 solvent: pyridine
 temp. 30.0 C / 303.1 K
 nucleus: mercury-400BBB "unknown"
 relax. delay 1.000 sec
 acq. time 0.160 sec
 fidh 6410.3 Hz
 D Width 17084.0 Hz
 d. repetitions
 4
 1st increments
 SERVE H1, 100.1252089 MHz
 COUPLE C13, 100.6180567 MHz
 power 41 dB
 on during acquisition
 off during delay
 IARP-1 modulated
 ITPA PROCESSING
 gauss apodization 0.074 sec
 ultra resolution 0.014 sec
 data size 2048 x 2048
 total time 2 hr, 52 min, 30 sec

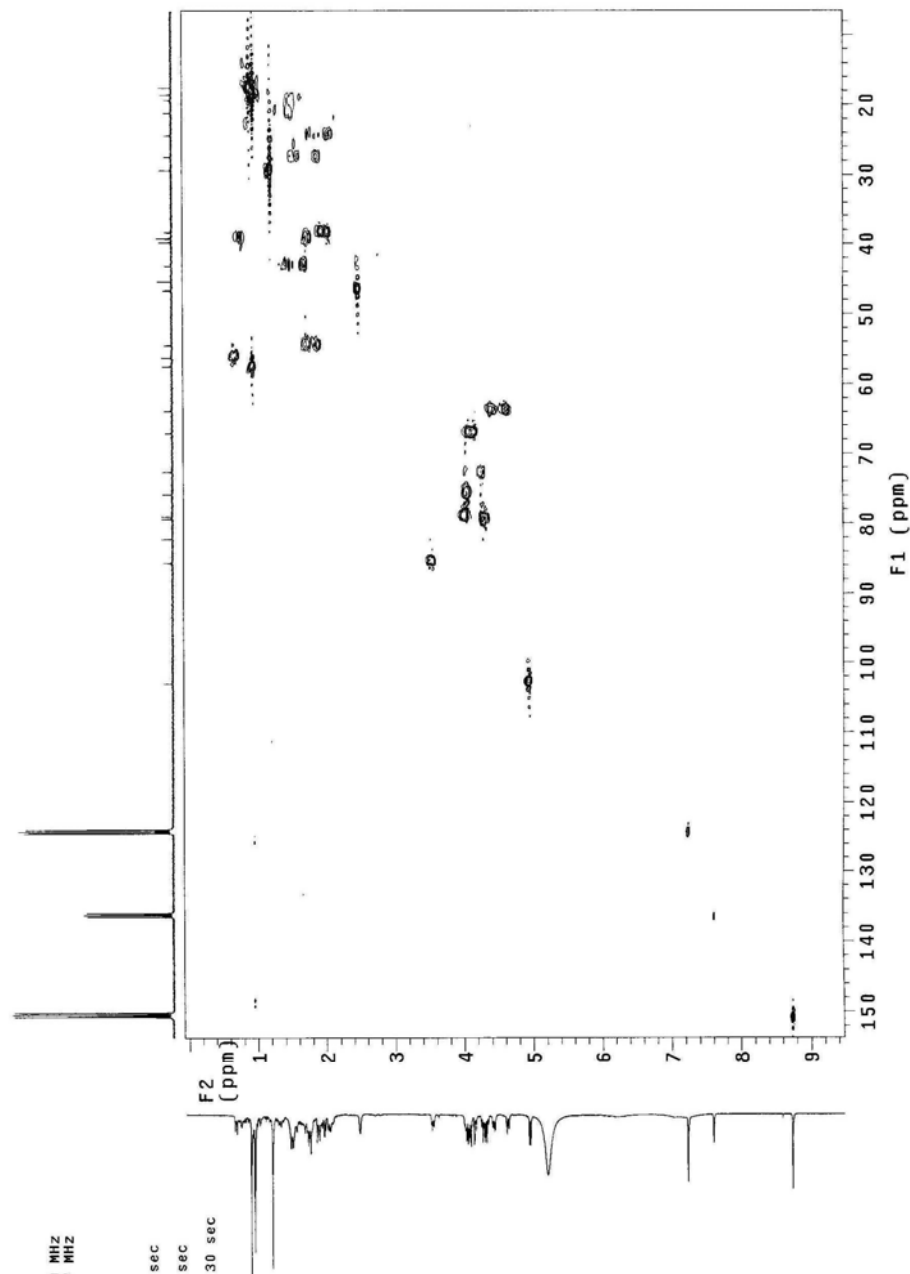


Fig. 17S HMQC spectrum of compound **3**.

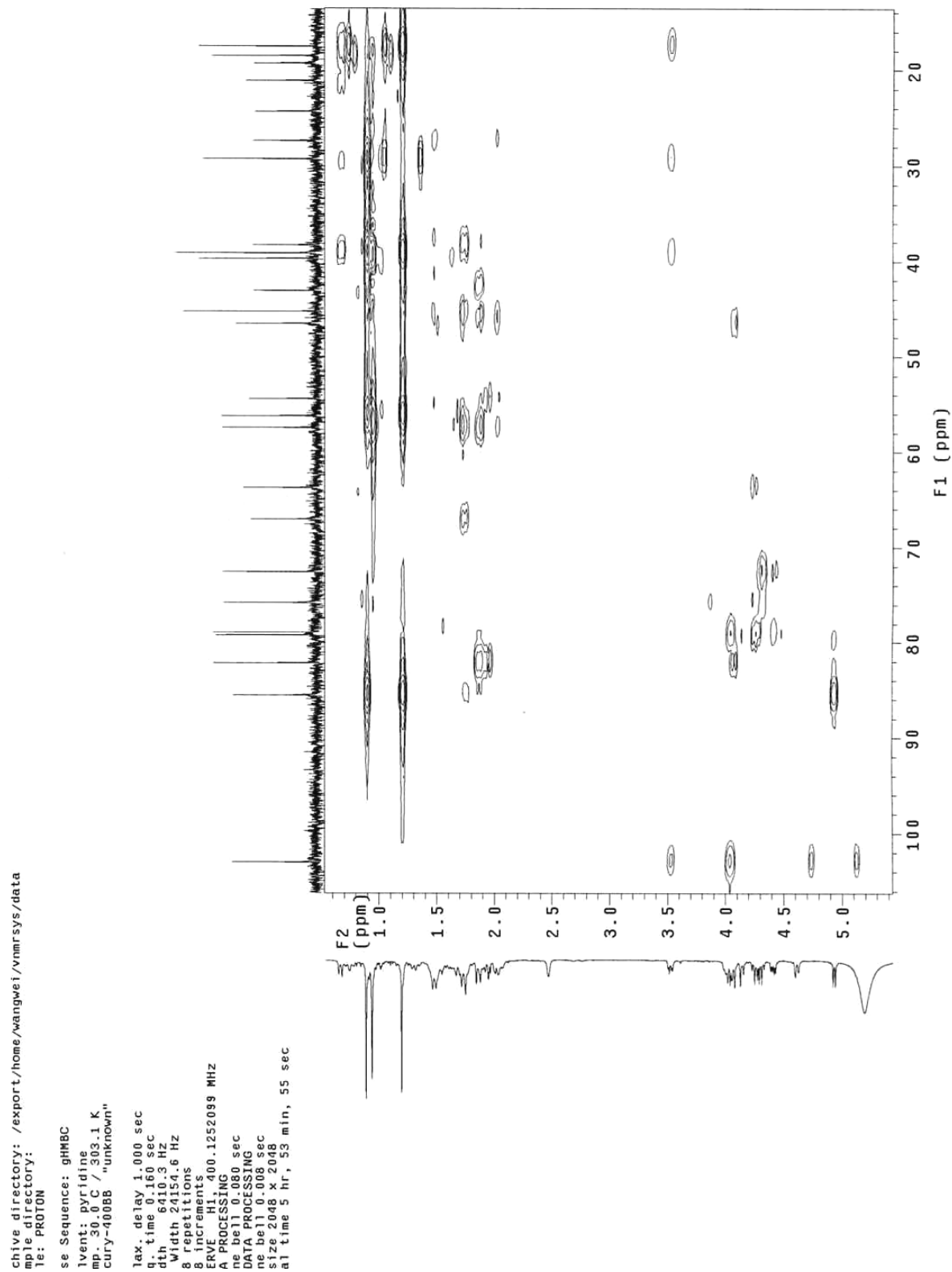


Fig. 18S HMBC spectrum of compound **3**.

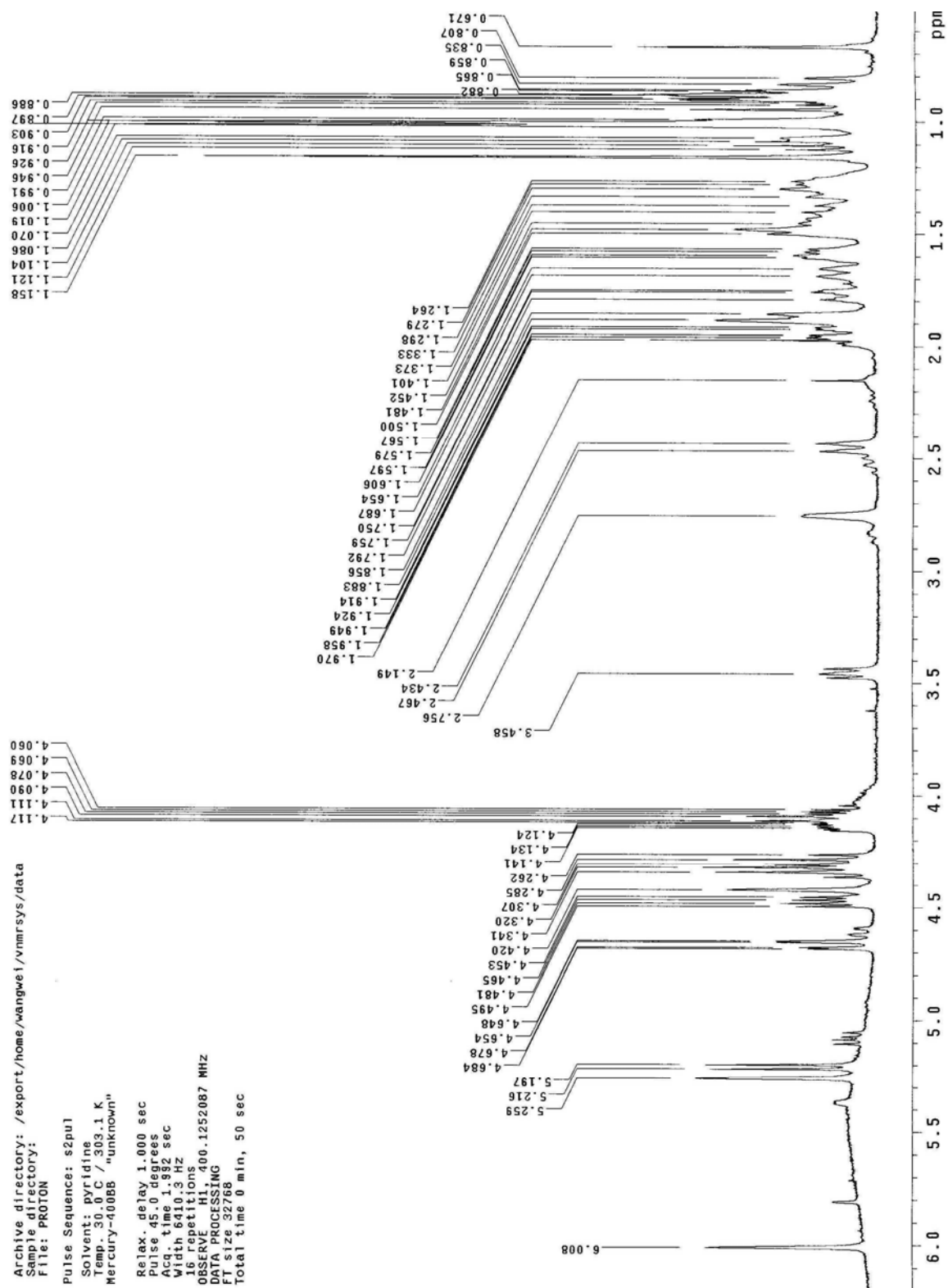


Fig. 19S $^1\text{H-NMR}$ spectrum of compound **4** (CDCl_3 , 400 MHz).

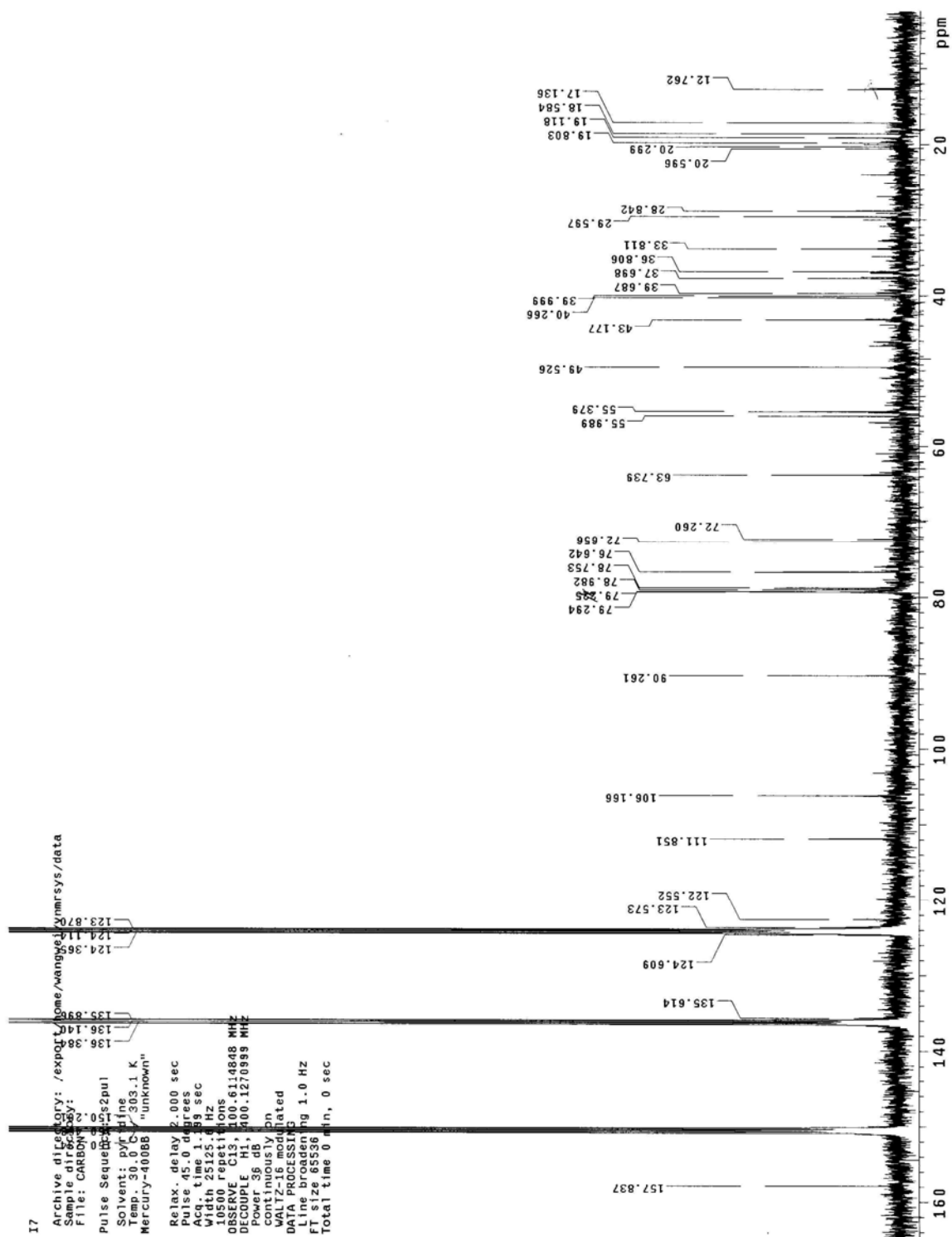


Fig. 20S ¹³C-NMR spectrum of compound 4 (CDCl₃, 100 MHz).

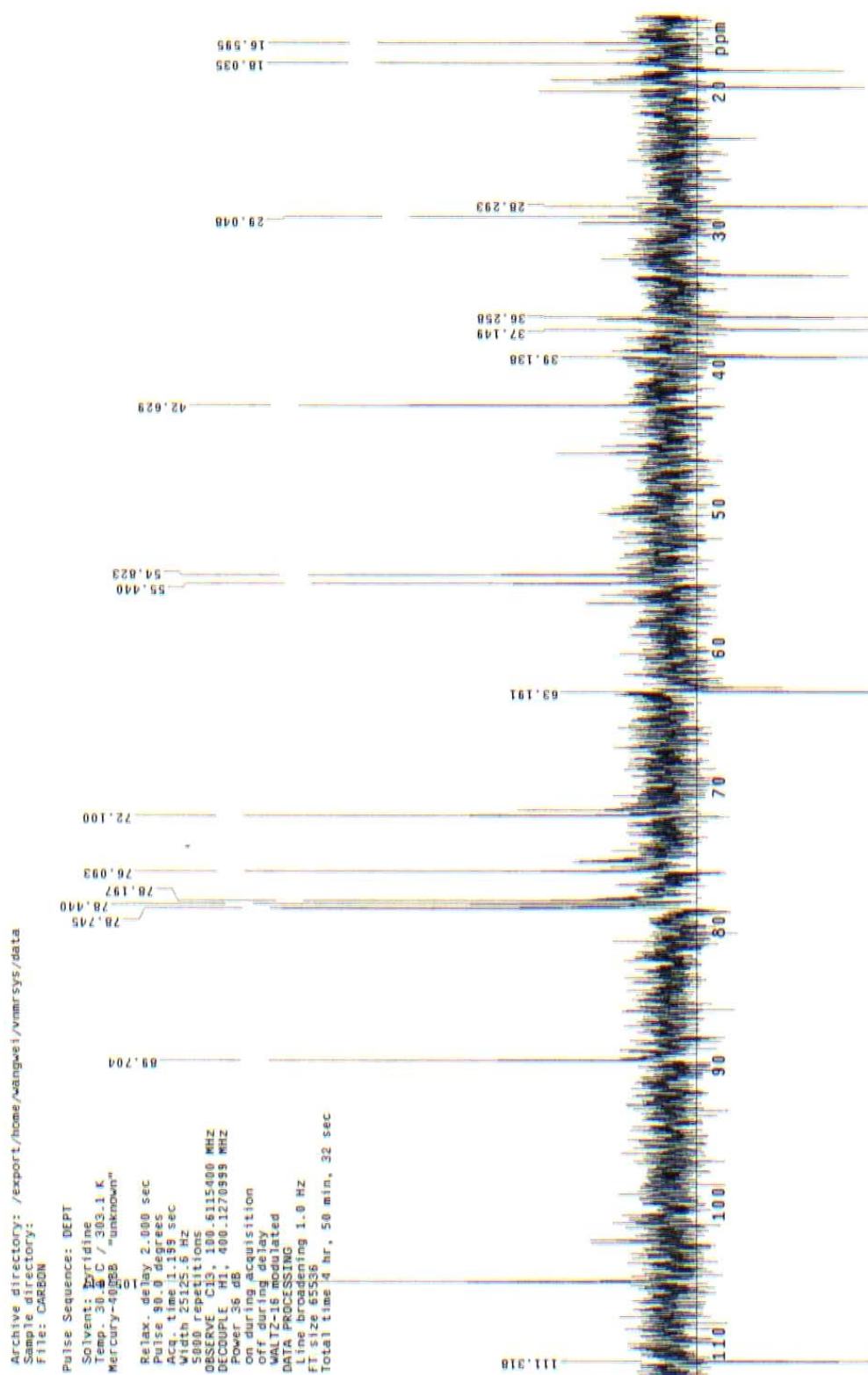


Fig. 21S DEPT spectrum of compound 4.

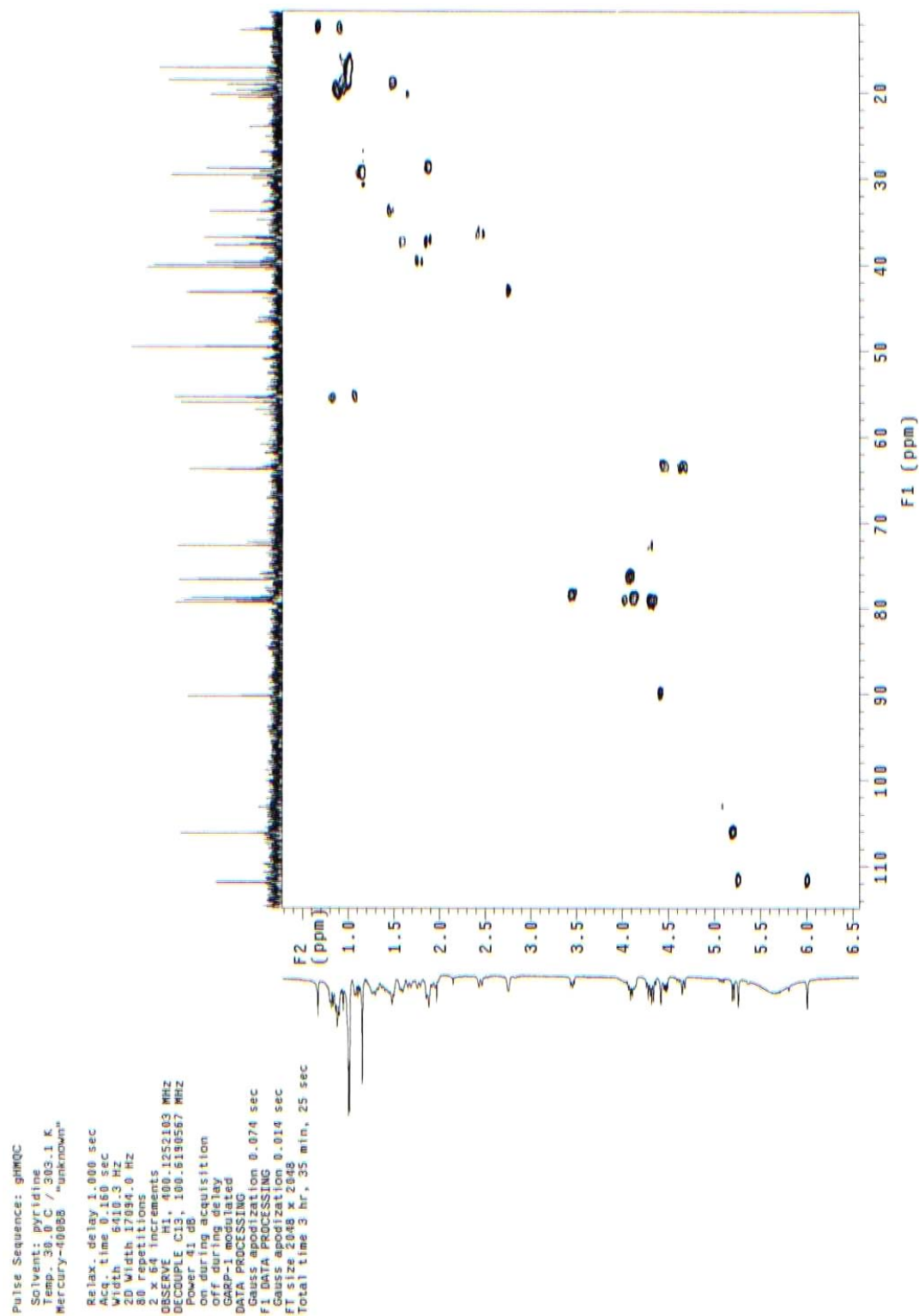


Fig. 22S HMQC spectrum of compound 4.

Archive directory: /export/home/wangwei/vnmr/sys/data
Sample directory:
File: PROTON
Pulse Sequence: gHMBC
Solvent: pyridine
Temp: 300.2 K
Mercury-400B5 "unknown"
Relax. delay 1.000 sec
Acq. time 0.160 sec
Width 5410.3 Hz
2D Width 24154.6 Hz
192 repetitions
128 increments
OBSERVE F1: 400.1252103 MHz
SOLVENT F1: 400.141 MHz
Sine bell 0.080 sec
F1 DATA PROCESSING
Sine bell 0.088 sec
FT size 2048 x 2048
Total time 8 hr, 48 min, 41 sec

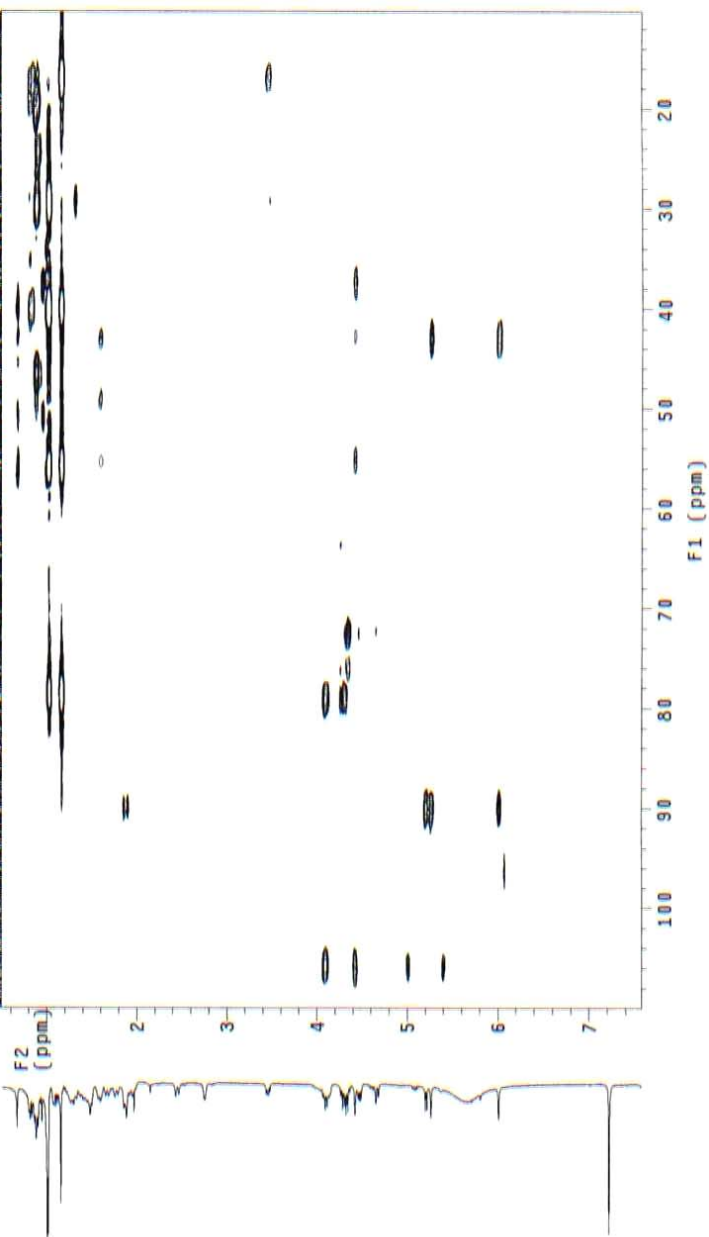


Fig. 23S HMBC spectrum of compound **4**.

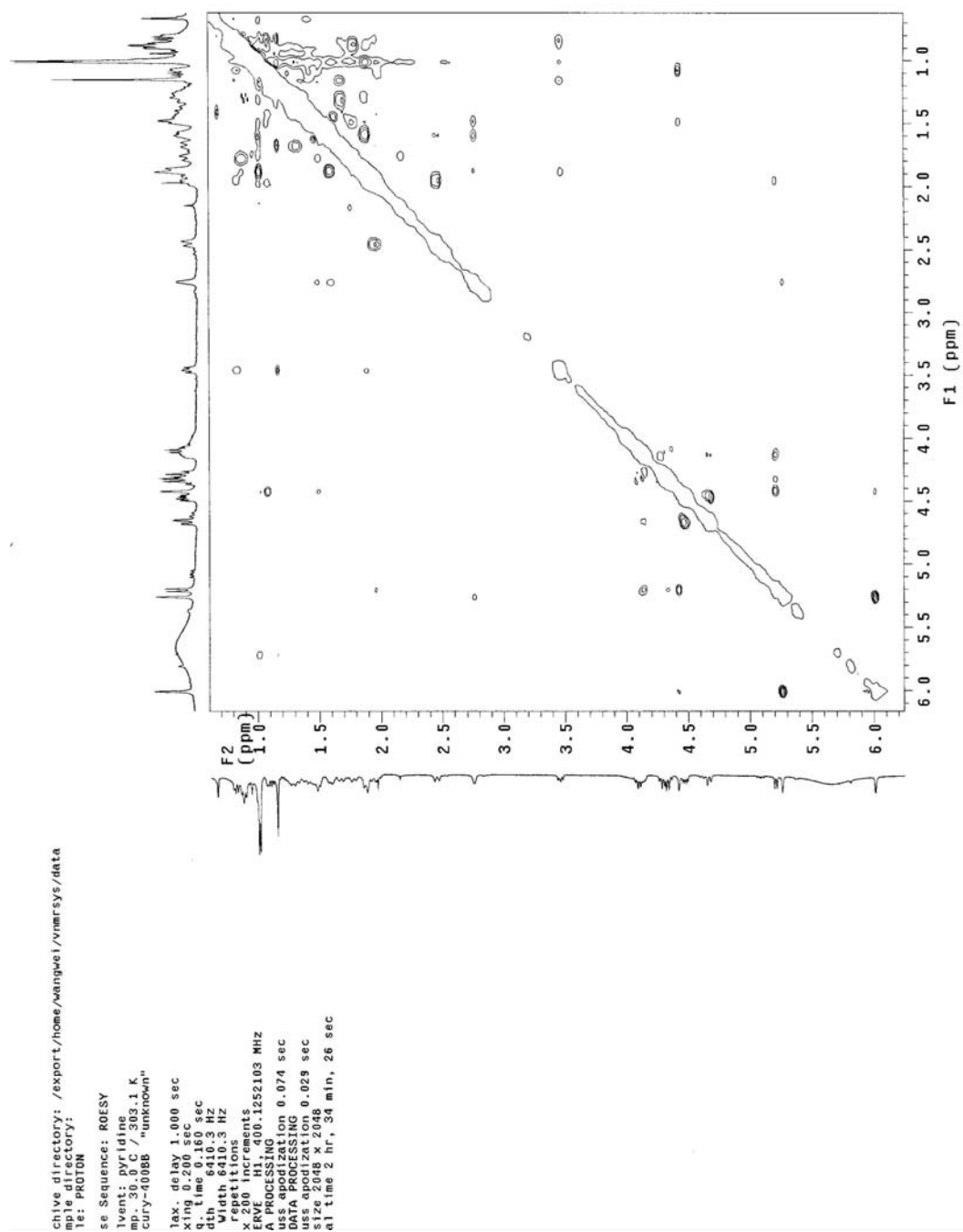


Fig. 24S ROESY spectrum of compound **4**.