

## Minor oxygenated cannabinoids from high potency *Cannabis sativa* L.

Safwat A. Ahmed,<sup>a,b</sup> Samir A. Ross,<sup>a,c\*</sup> Desmond Slade,<sup>a</sup> Mohamed M. Radwan,<sup>a,d</sup> Ikhlas A. Khan<sup>a,c</sup>,  
Mahmoud A. ElSohly<sup>a,e\*</sup>

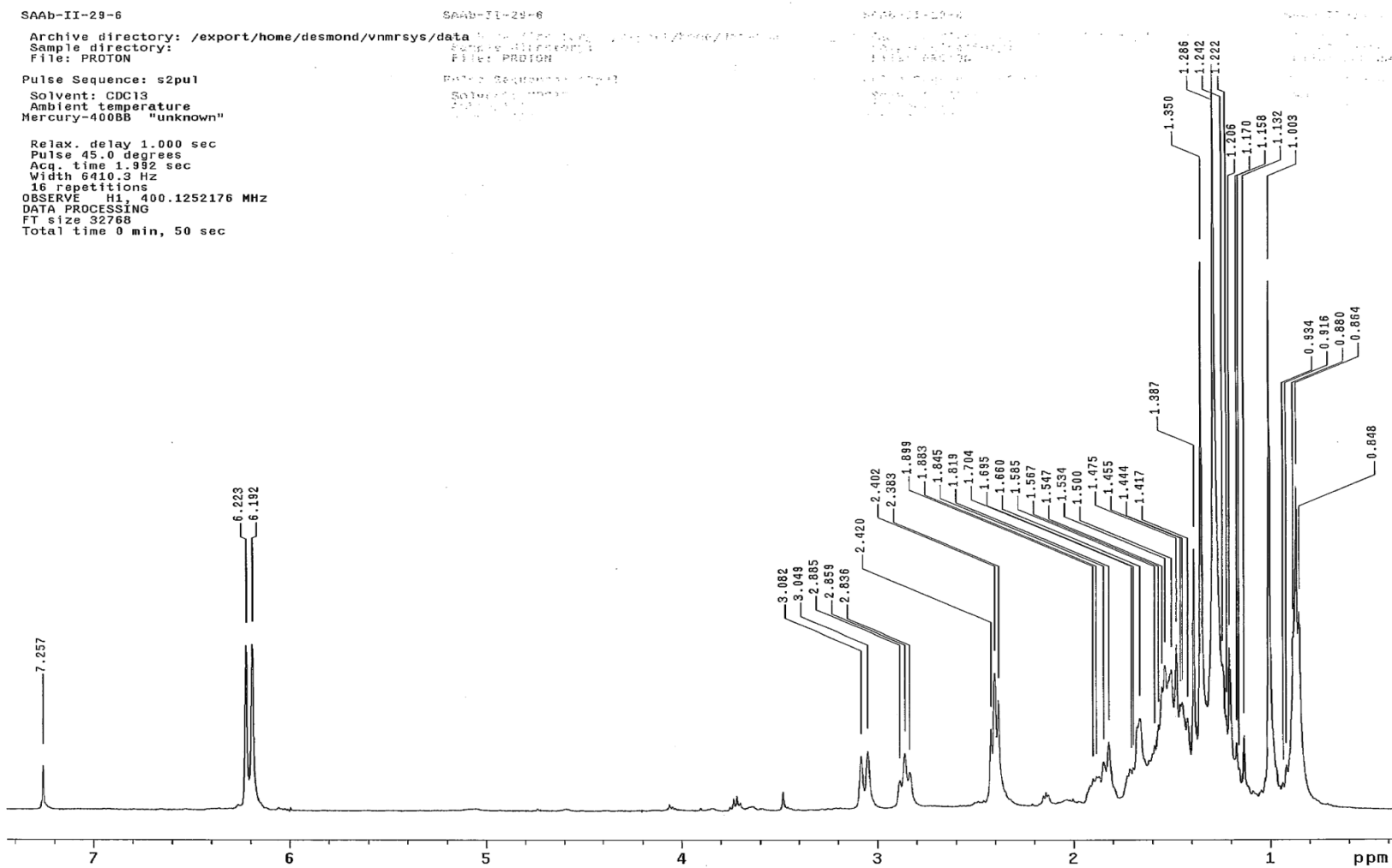
<sup>a</sup>*National Center for Natural Products Research, <sup>c</sup>Department of Pharmacognosy and <sup>e</sup>Department of  
Pharmaceutics,*

*School of Pharmacy, The University of Mississippi, University, MS 38677.*

<sup>b</sup>*Department of Pharmacognosy, Faculty of Pharmacy, Suez Canal University, Ismailia, Egypt.*

<sup>d</sup>*Department of Pharmacognosy, Faculty of Pharmacy, Alexandria University, Alexandria, Egypt.*

\*Corresponding authors. Tel.: +1-662-915-1031; fax: +1-662-915-7989 (S.A. Ross), tel.: +1-662-915-5928; fax: +1-662-915-5587 (M.A. ElSohly). *E-mail addresses:* [sross@olemiss.edu](mailto:sross@olemiss.edu) (S.A. Ross), [melsohly@olemiss.edu](mailto:melsohly@olemiss.edu) (M.A. ElSohly).



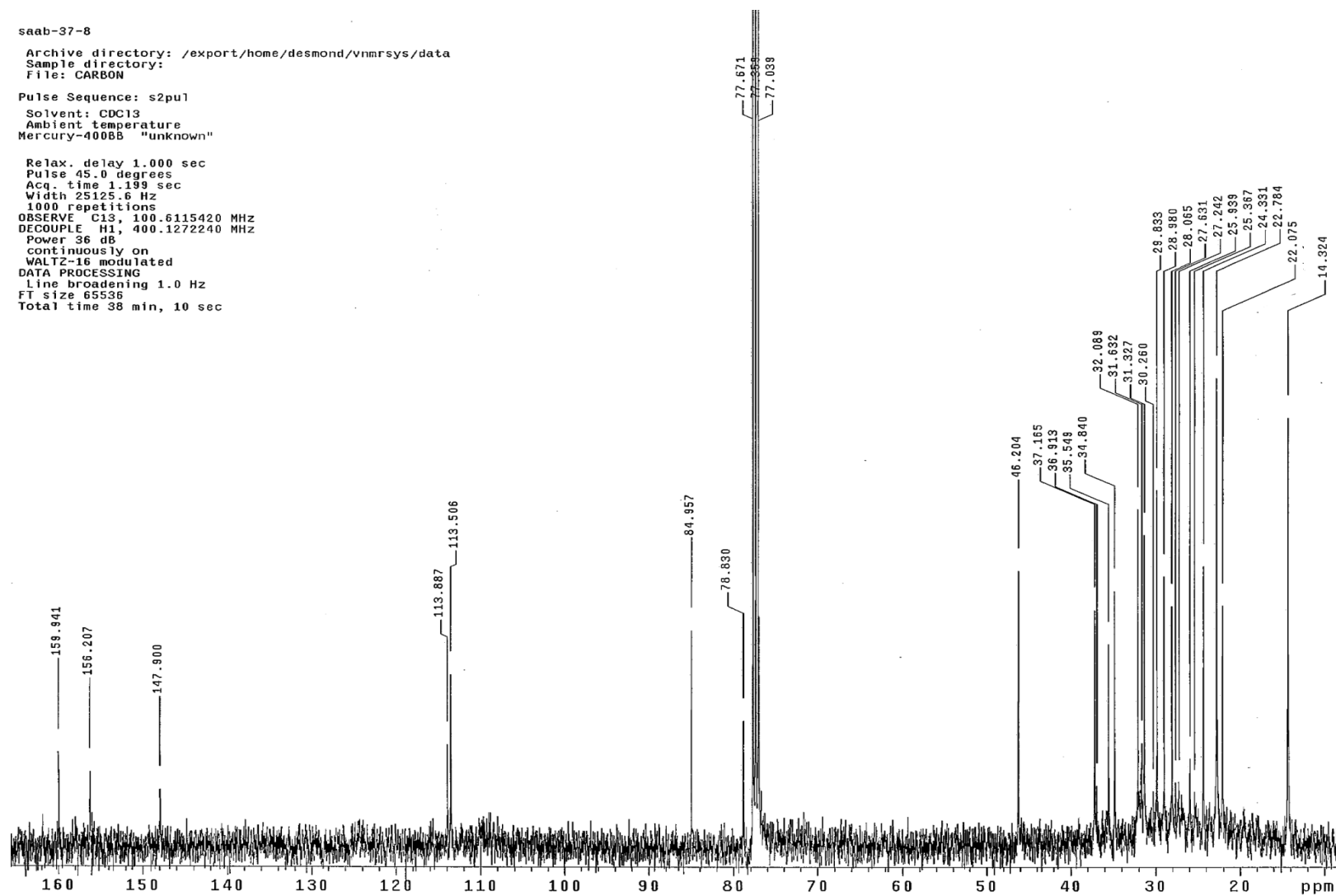
$^1\text{H-NMR}$  spectrum of compound 1 in ( $\text{CDCl}_3$ , 400 MHz)

saab-37-8

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

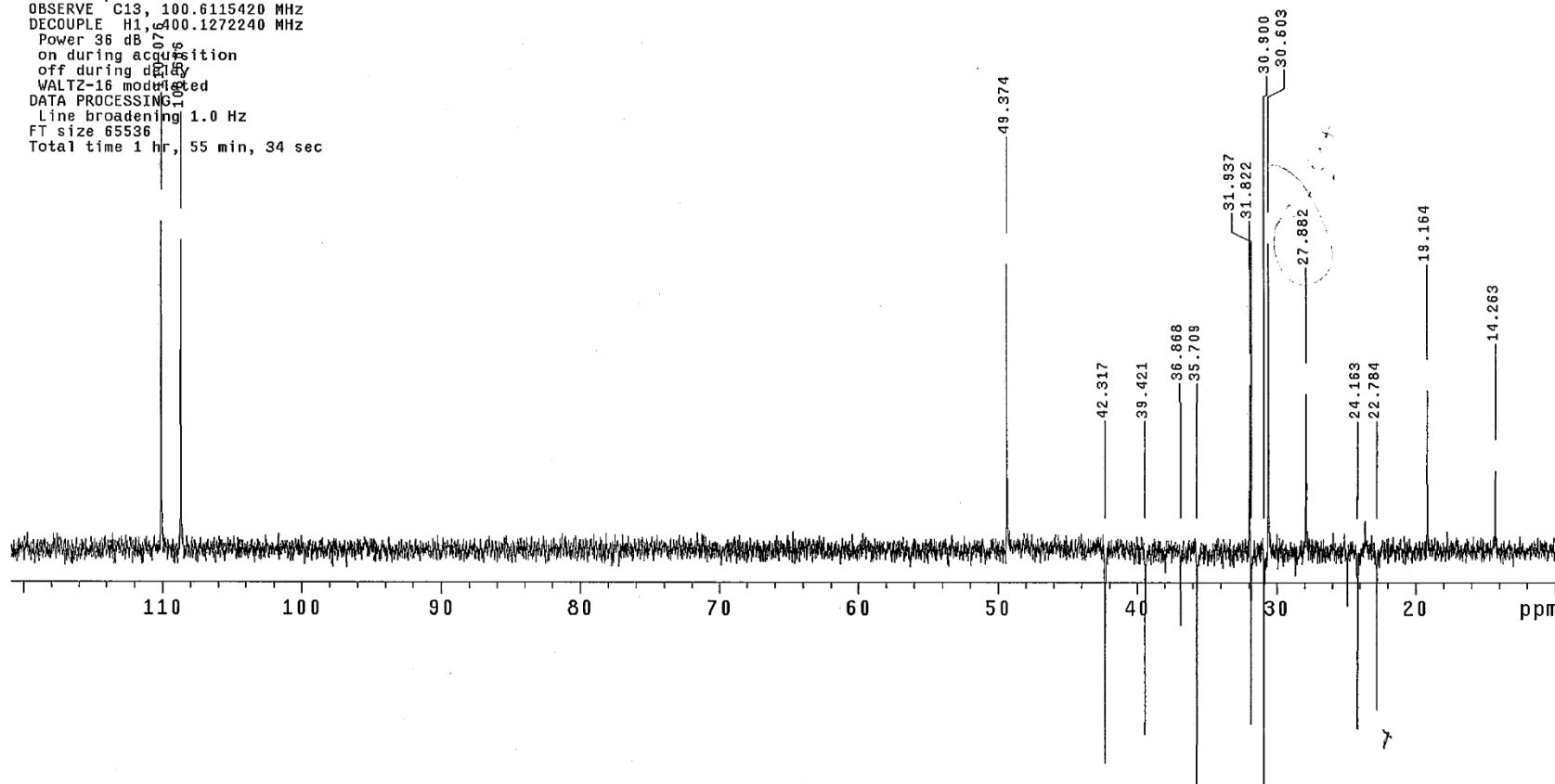
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
1000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 38 min, 10 sec



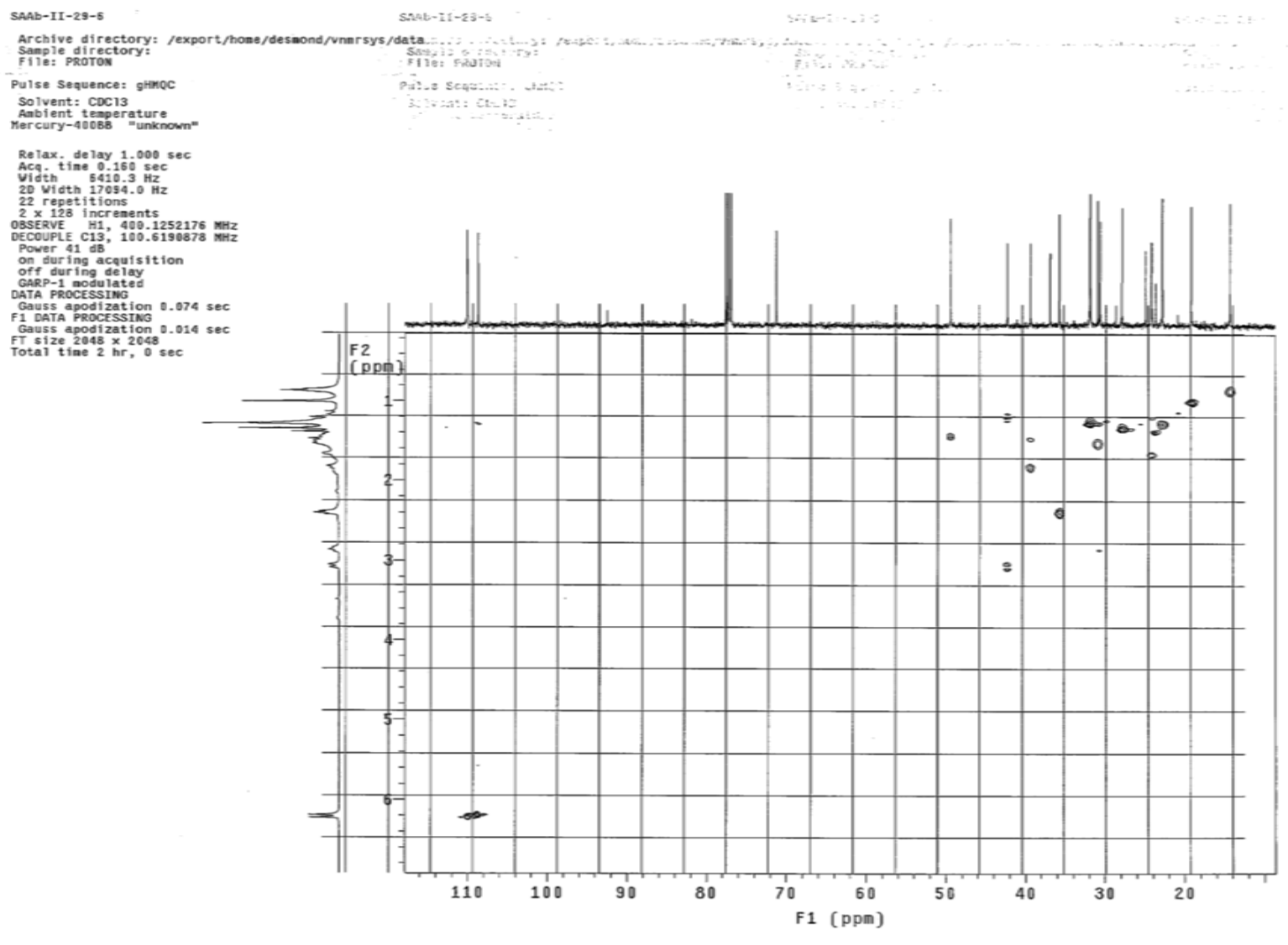
$^{13}\text{C}$ -NMR spectrum of compound 1 in ( $\text{CDCl}_3$ , 100 MHz)

saabII-29-6  
Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: CARBON  
Pulse Sequence: DEPT  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Pulse 90.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
3000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
on during acquisition  
off during DEPT  
WALTZ-16 modified  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 1 hr, 55 min, 34 sec

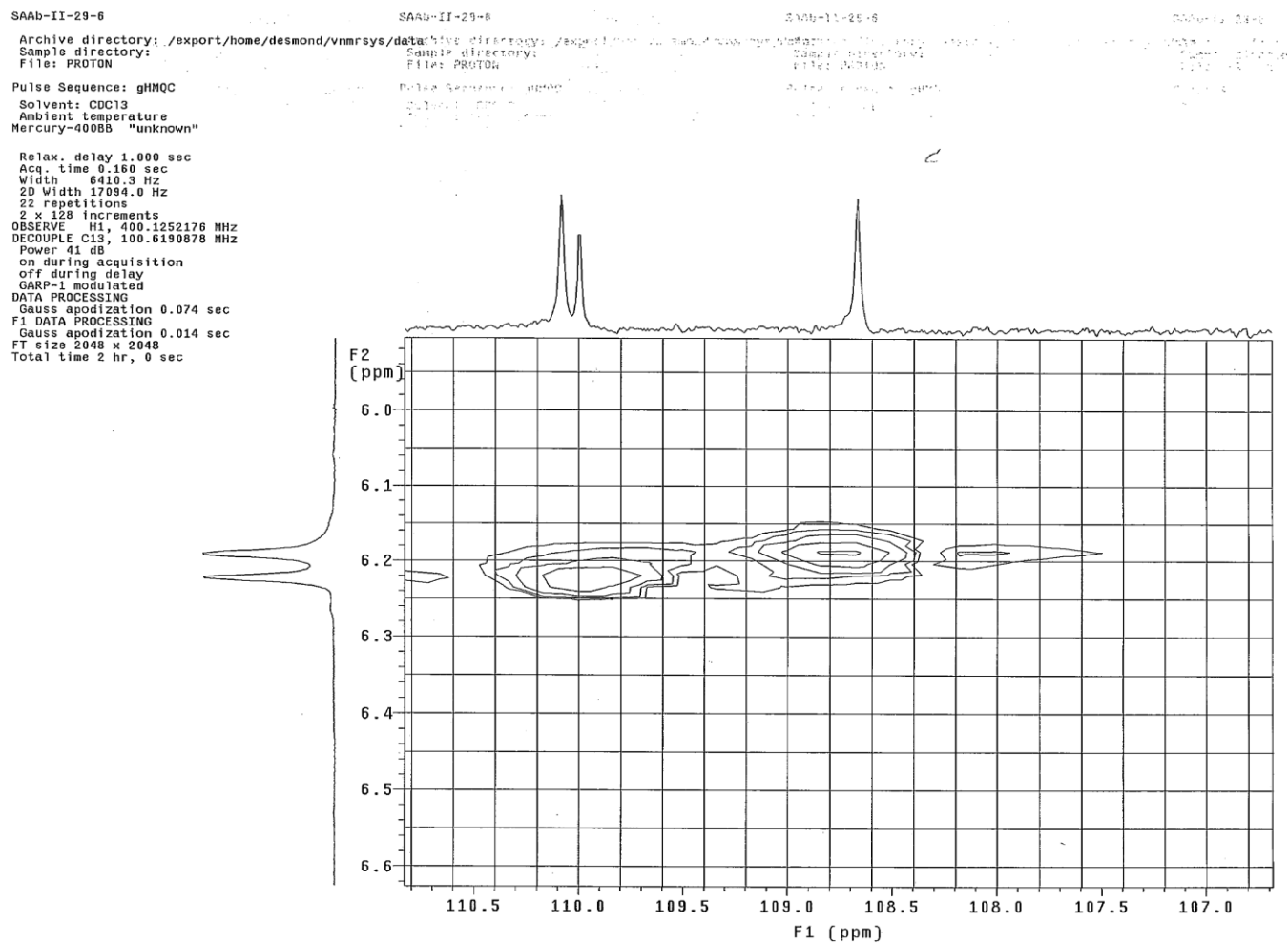


DEPT spectrum of compound 1 in (CDCl<sub>3</sub>, 100 MHz)

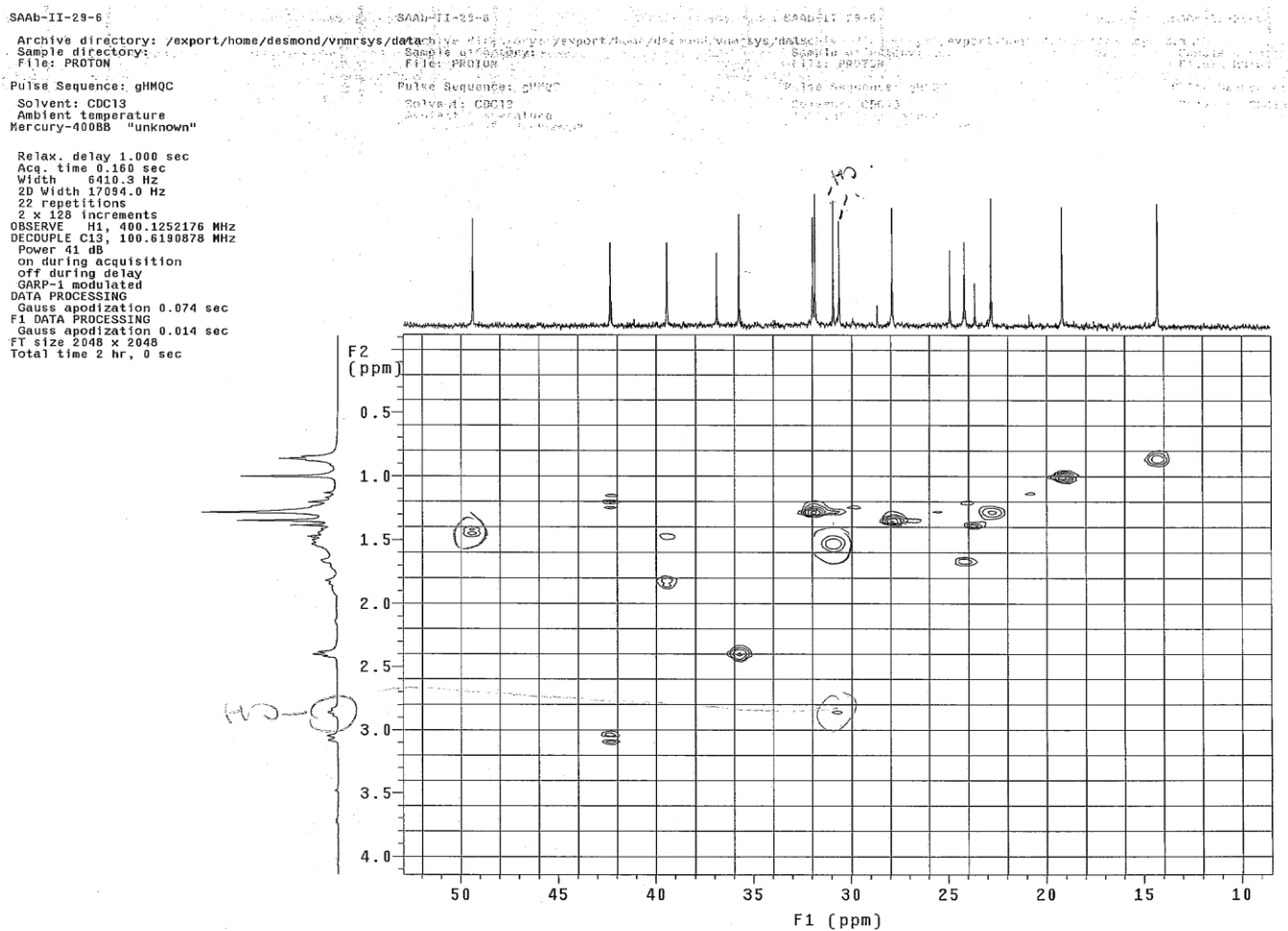




2D-HMOC spectrum of compound 1



Partial expansions of the 2D-HMQC spectrum of compound 1



Partial expansions of the 2D-HMQC spectrum of compound 1



SAAb-II-29-6

SAAb-II-29-6

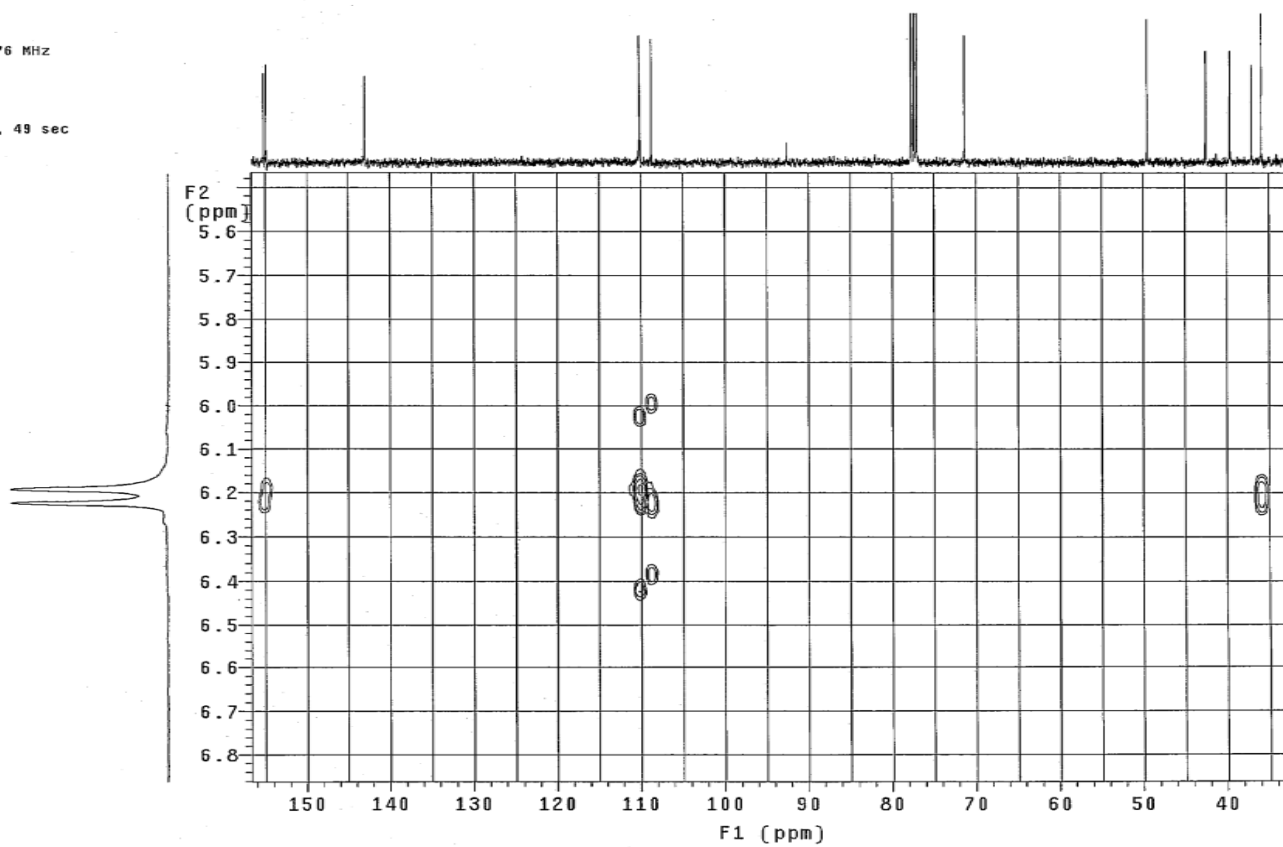
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec



Partial expansions of the 2D-HMBC spectrum of compound 1

SAAB-II-29-6

SAAB-II-29-6

SAAB-II-29-6

Archive directory: /export/home/desmond/vmarsys/data

Sample directory:

File: PROTON

Sample directory:

File: PROTON

Pulse Sequence: gHMBC

Solvent: CDCl<sub>3</sub>

Ambient temperature

Mercury-40055 "unknown"

Relax. delay 1.000 sec

Acq. time 0.160 sec

Width 6410.3 Hz

2D Width 24154.6 Hz

22 repetitions

400 increments

OBSERVE H1, 400.1252176 MHz

DATA PROCESSING

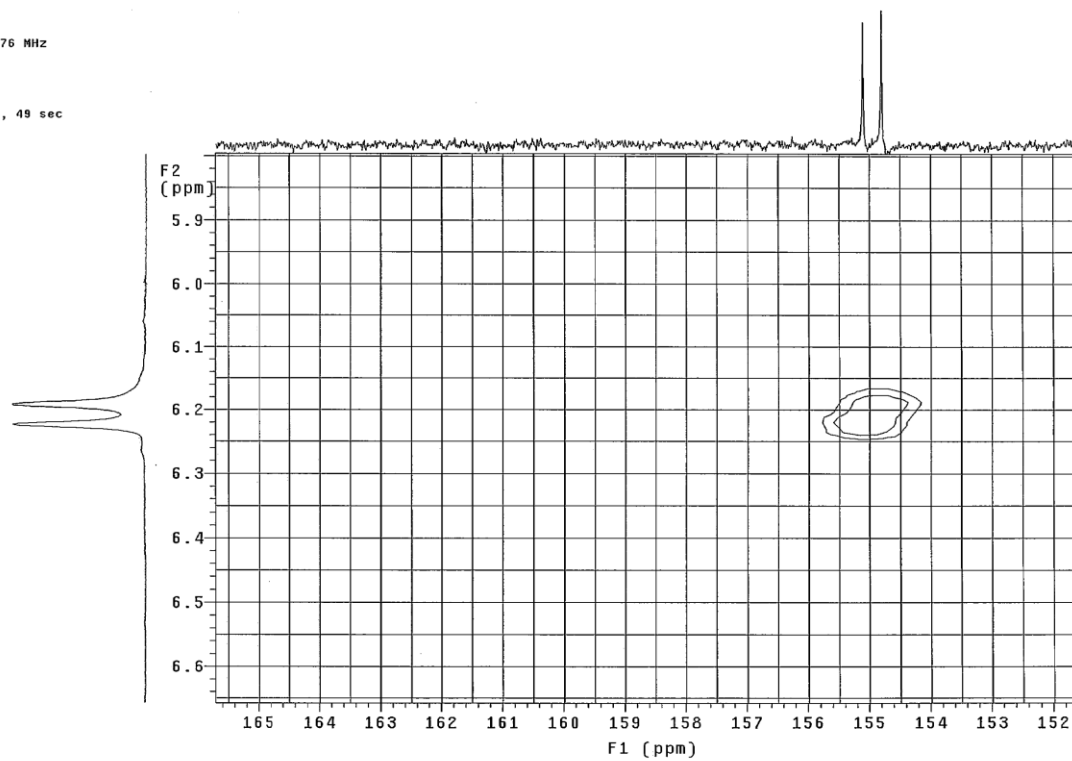
Sine bell 0.080 sec

F1 DATA PROCESSING

Sine bell 0.008 sec

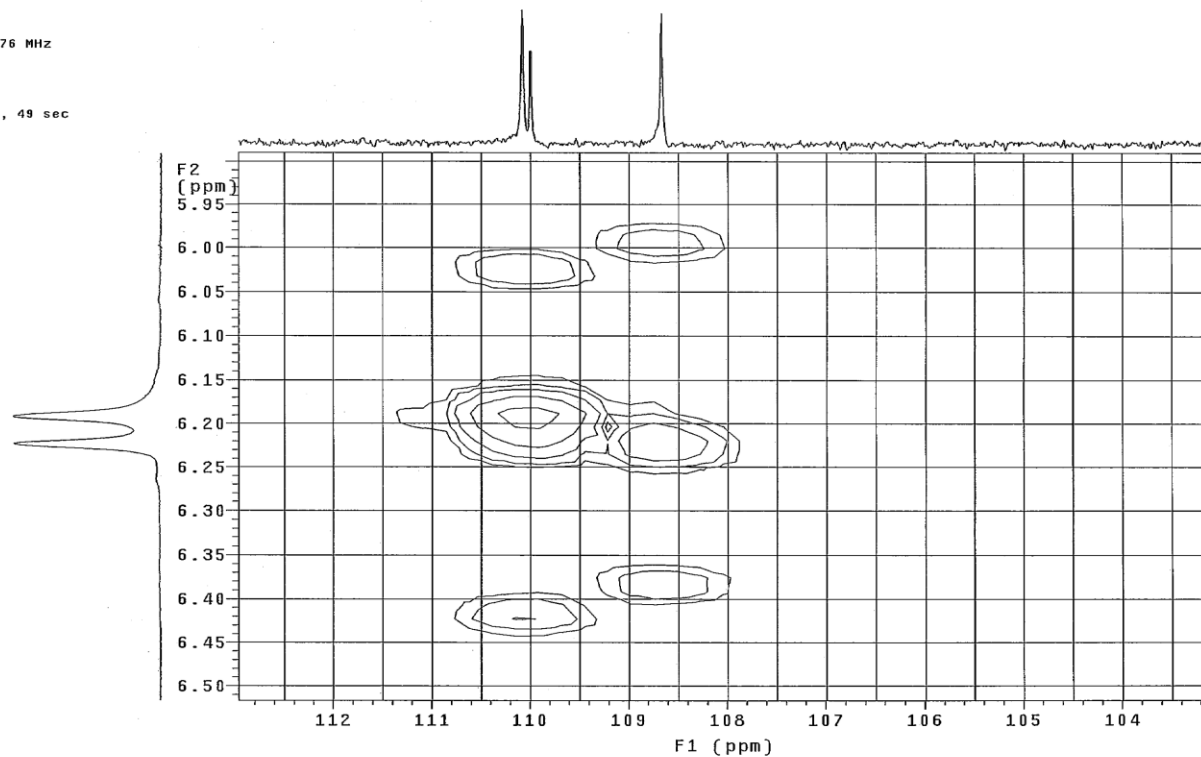
FT size 2048 x 2048

Total time 3 hr, 12 min, 49 sec



Partial expansions of the 2D-HMBC spectrum of compound 1

SAAb-II-29-6  
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec



Partial expansions of the 2D-HMBC spectrum of compound 1

SAAB-II-29-6

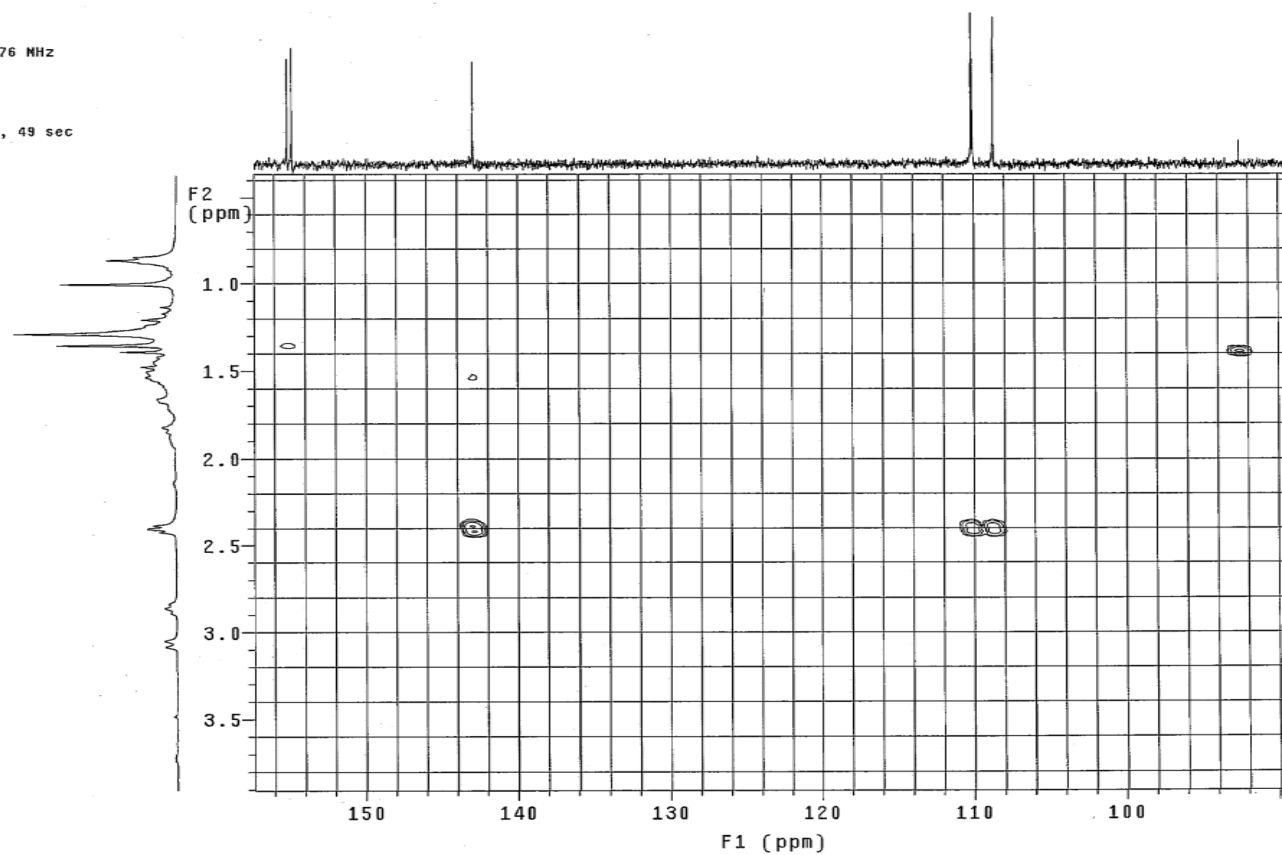
Archive directory: /export/home/desmond/vnmrSYS/data

Sample directory:  
File: PROTON

Pulse Sequence: gHMBC

Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec



Partial expansions of the 2D-HMBC spectrum of compound 1



SAAb-II-29-6

Archive directory: /export/home/desmond/vnmr/sys/data

Sample directory:

File: PROTON

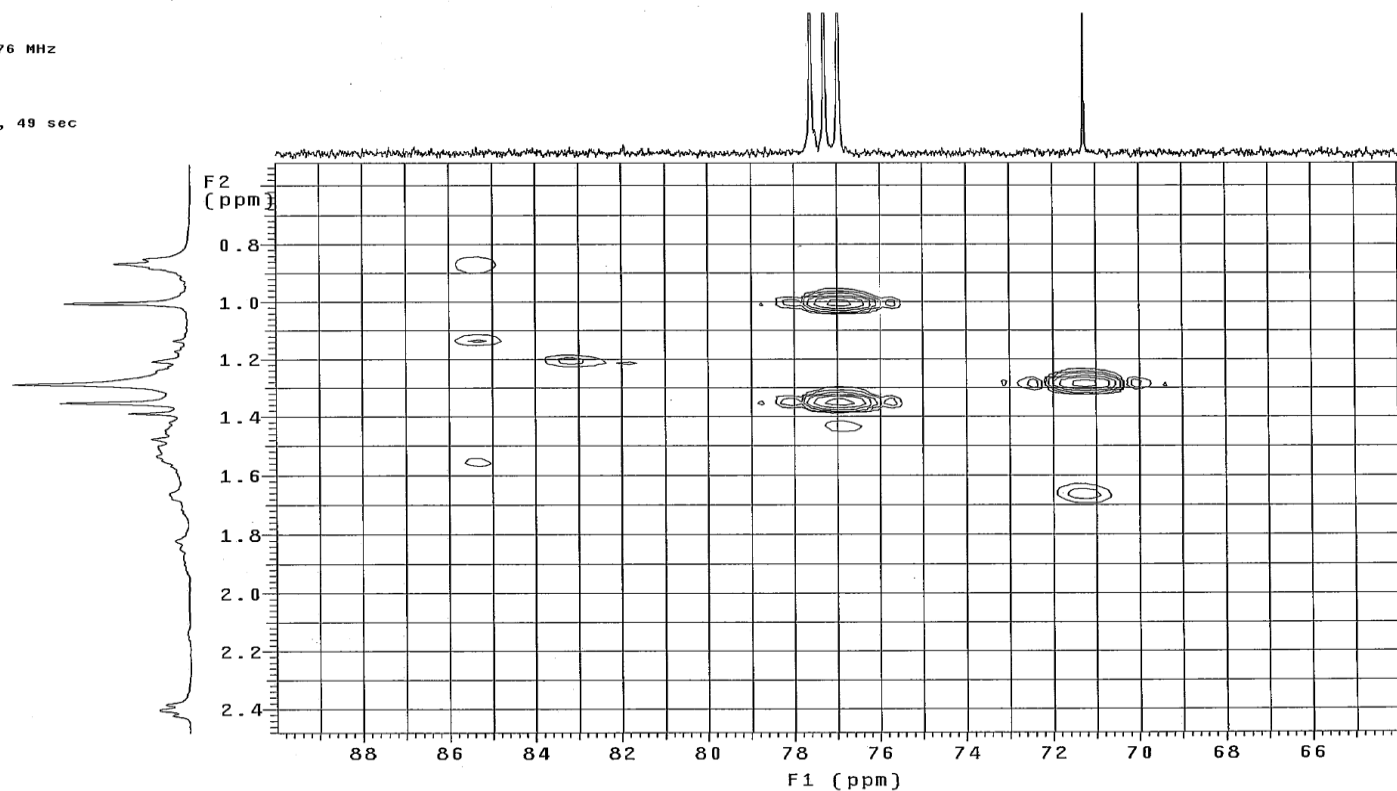
Pulse Sequence: gHMBC

Solvent: CDC13

Ambient temperature

Mercury-400SB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec

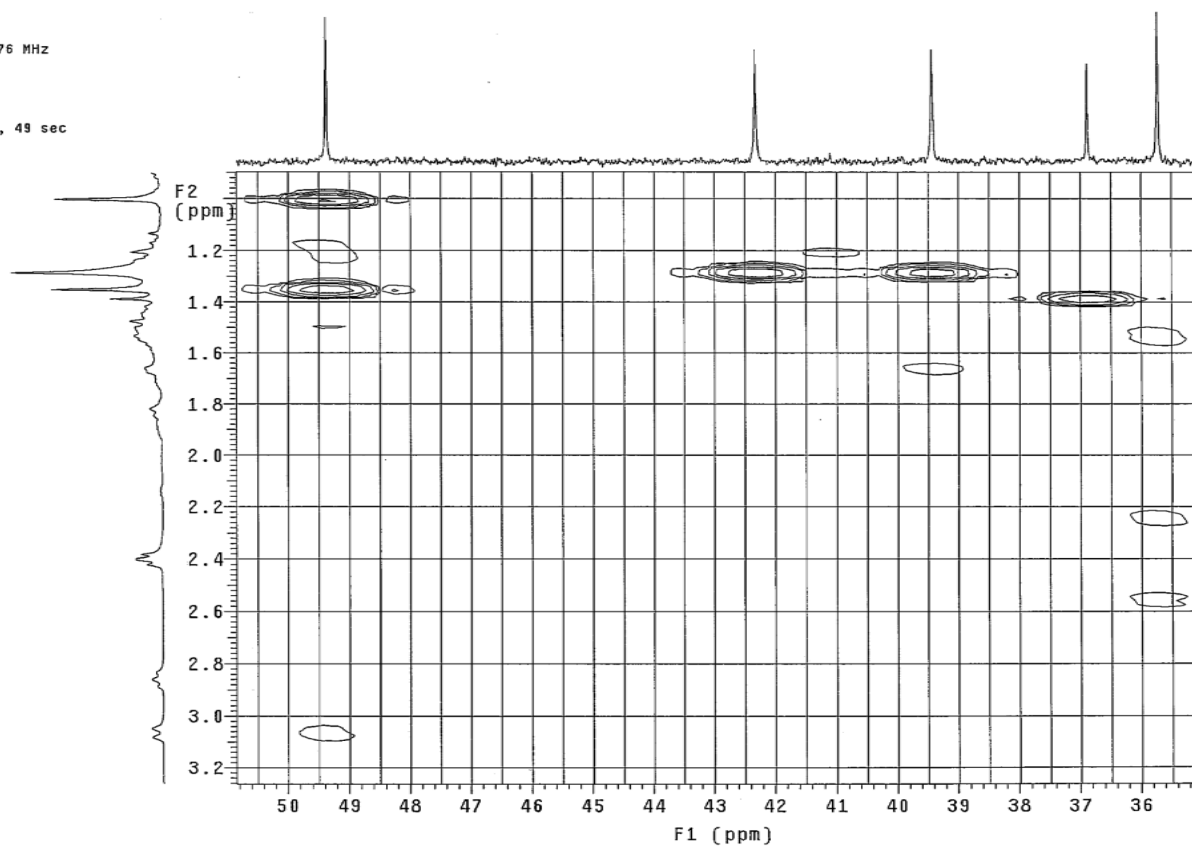


Partial expansions of the 2D-HMBC spectrum of compound 1

SAAb-II-29-6

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec



Partial expansions of the 2D-HMBC spectrum of compound 1

SAAb-II-29-6

SAAb-II-29-6

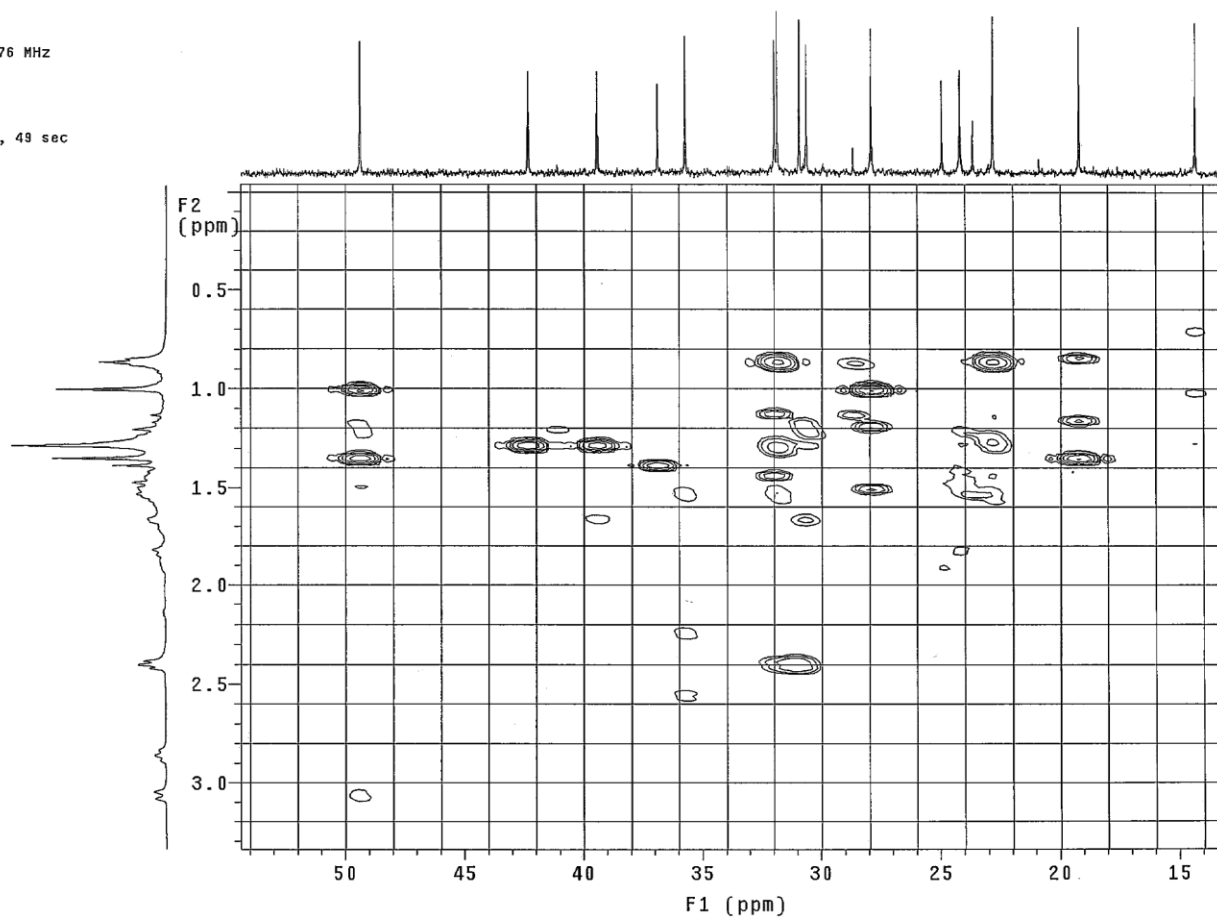
SAAb-II-29-6

SAAb-II-29-6

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
22 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 min, 49 sec



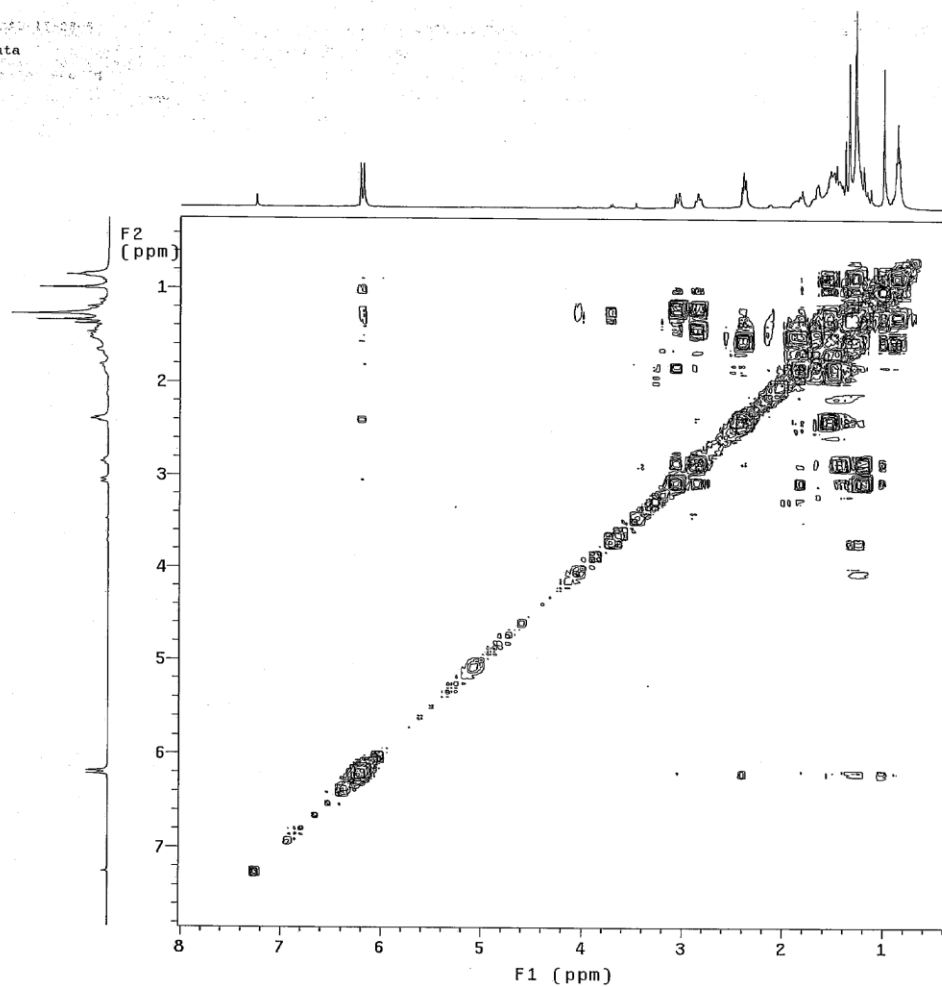
Partial expansions of the 2D-HMBC spectrum of compound 1

SAAB-II-29-6

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13  
Ambient temperature  
Mercury-40055 "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
9 repetitions  
128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
F1 size 2048 x 2048  
Total time 21 min, 55 sec



2D-COSY spectrum of compound 1

SAABII-29-6

Archive directory: /export/home/mradwan/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: pyridine  
Temp. 35.0 C / 308.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
20 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252091 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
FT size 2048 x 2048  
Total time 0 min, 0 sec

SAABII-29-6

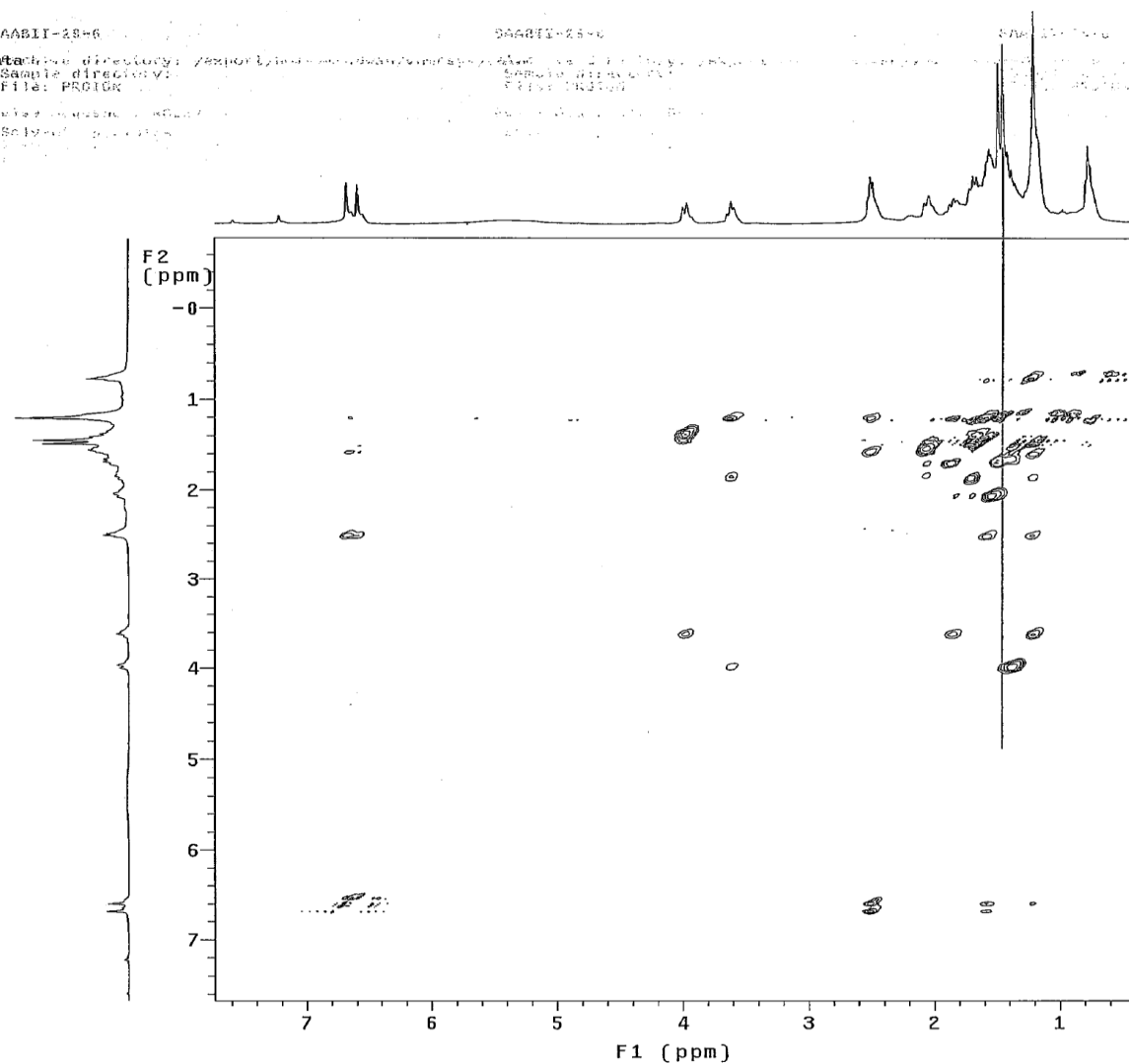
Archive directory: /export/home/mradwan/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: pyridine  
Temp. 35.0 C / 308.1 K  
Mercury-400BB "unknown"

SAABII-29-6

Archive directory: /export/home/mradwan/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: pyridine  
Temp. 35.0 C / 308.1 K  
Mercury-400BB "unknown"



2D-ROESY spectrum of compound 1

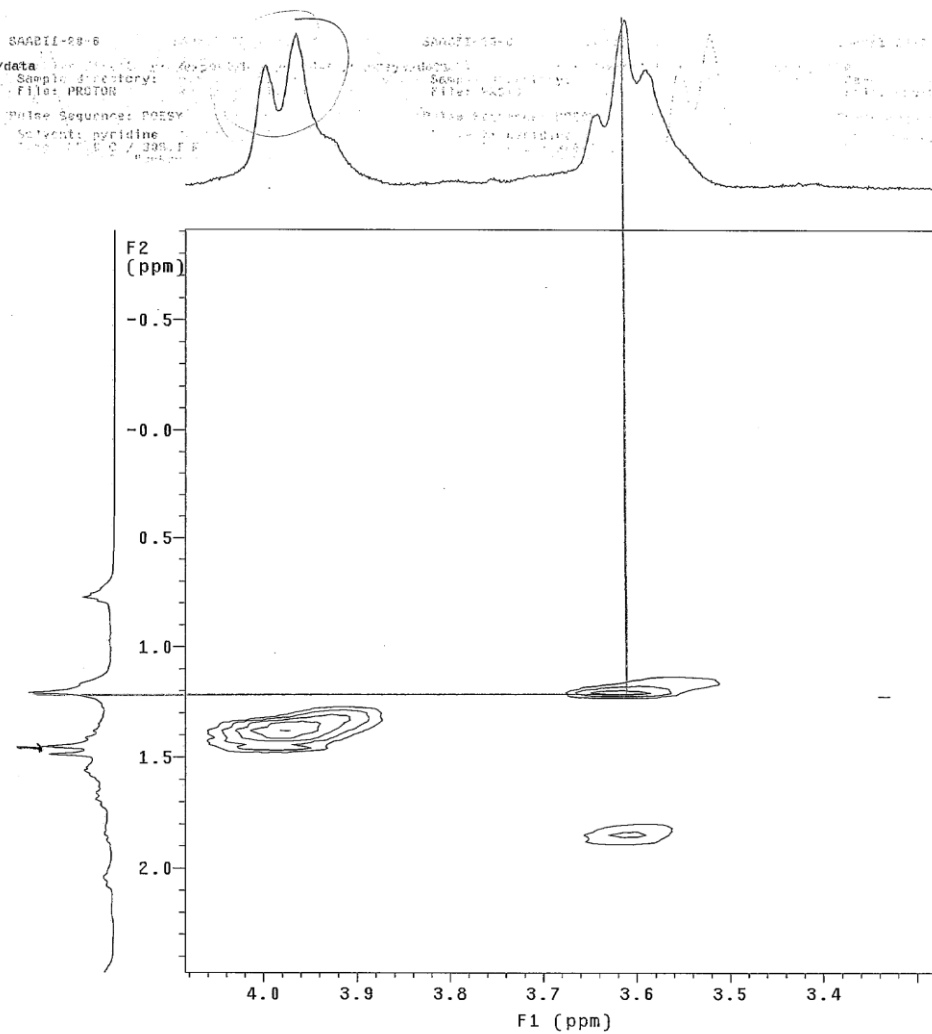
SAABII-29-6

Archive directory: /export/home/mradwan/vnmrsws/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY

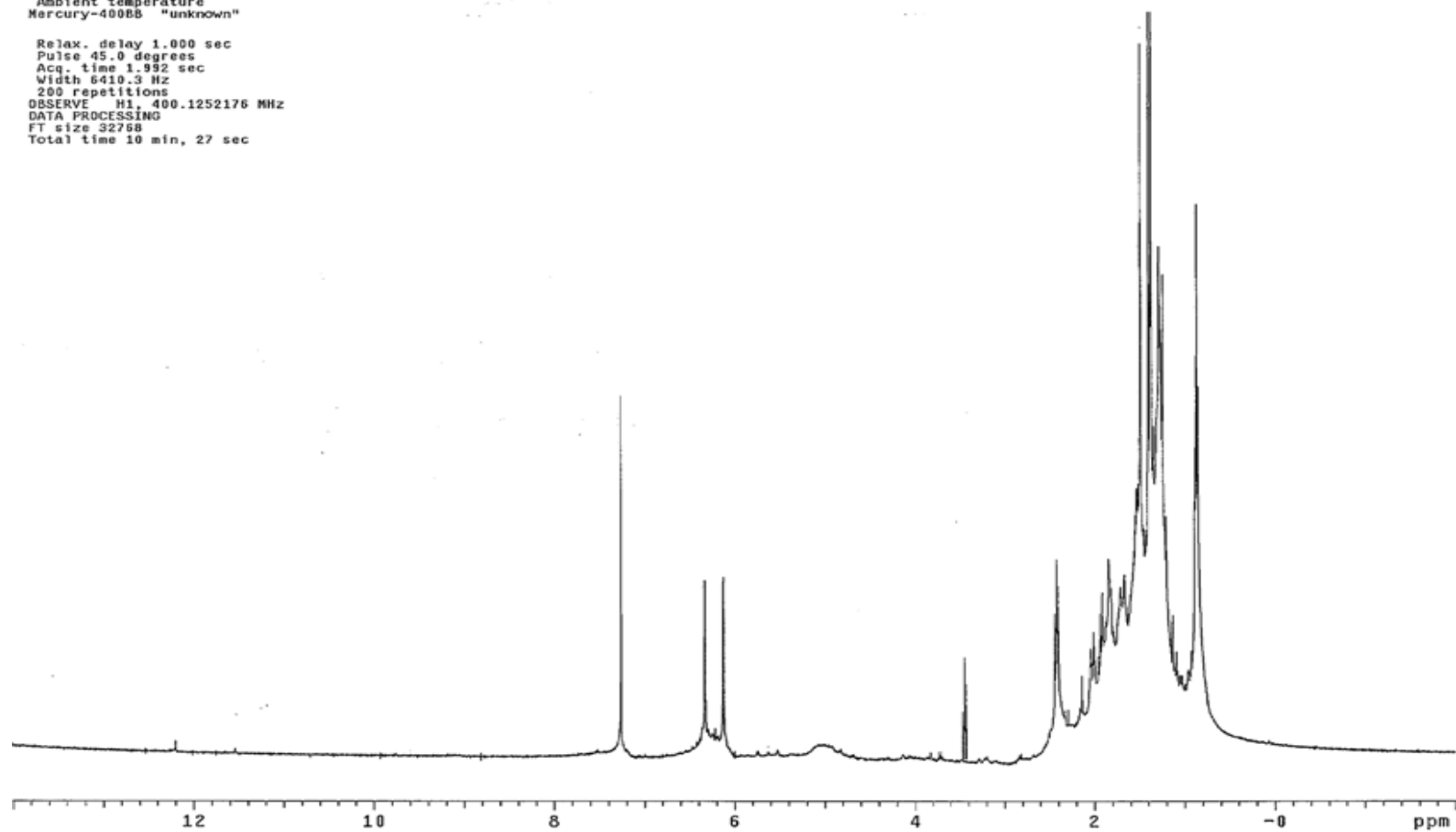
Solvent: pyridine  
Temp: 35.0 C / 308.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
20 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252091 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
F1 size 2048 x 2048  
Total time 2 hr, 56 min, 26 sec



2D-ROESY spectrum of compound 1

SAAB\_II-30-8  
Archive directory: /export/home/desmond/vnmrsys/data/priv-4  
Sample directory: /export/home/desmond/vnmrsys/data/priv-4  
File: PROTON  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
200 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
FT size 32768  
Total time 10 min, 27 sec



<sup>1</sup>H-NMR spectrum of compound 2 in (CDCl<sub>3</sub>, 400 MHz)

SAABII-30-8

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

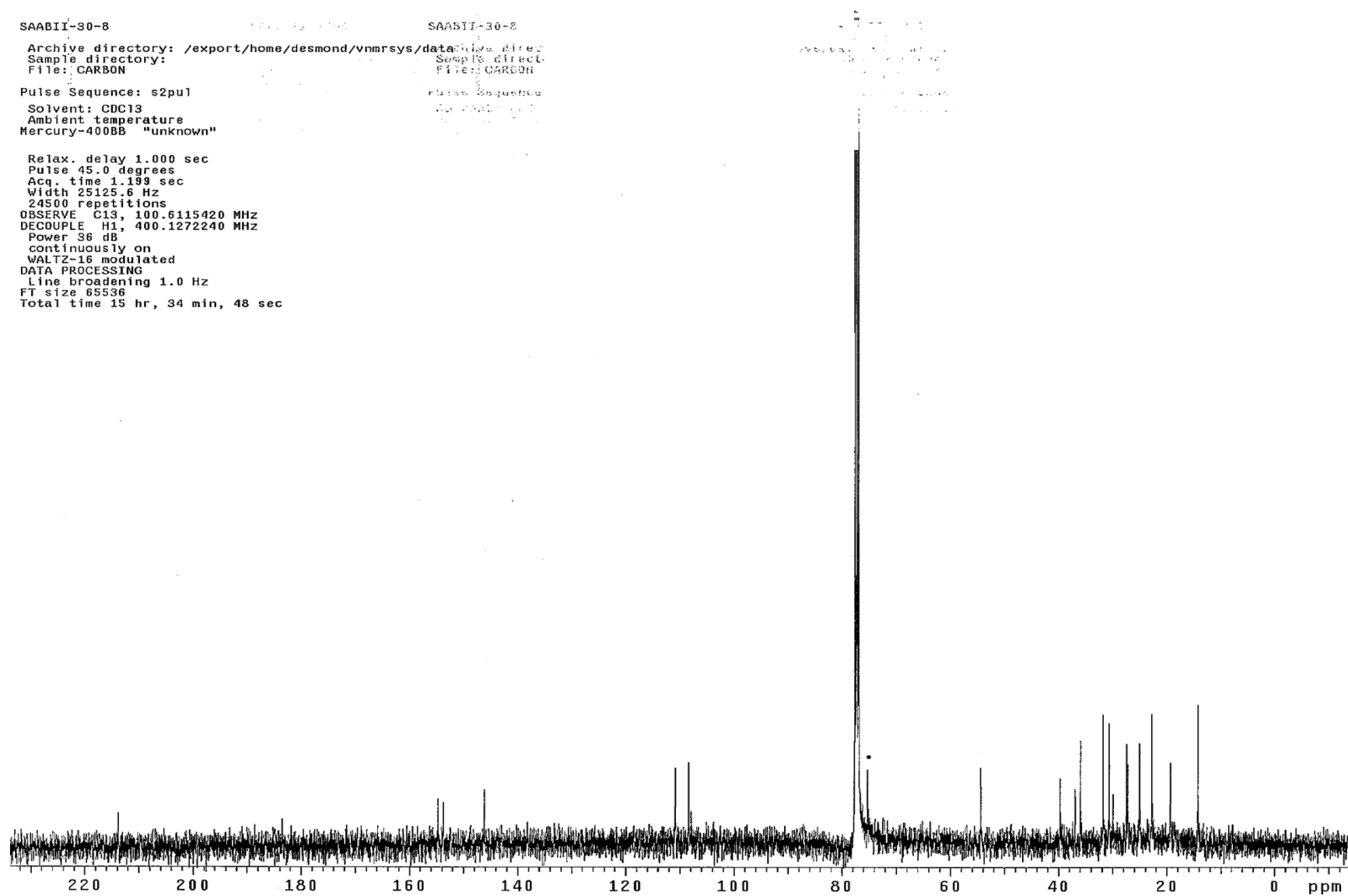
Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
24500 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 15 hr, 34 min, 48 sec



$^{13}\text{C}$ -NMR spectrum of compound 2 in ( $\text{CDCl}_3$ , 100 MHz)



SAABII-30-8

SAABII-30-8

SAABII-30-8

SAABII-30-8

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

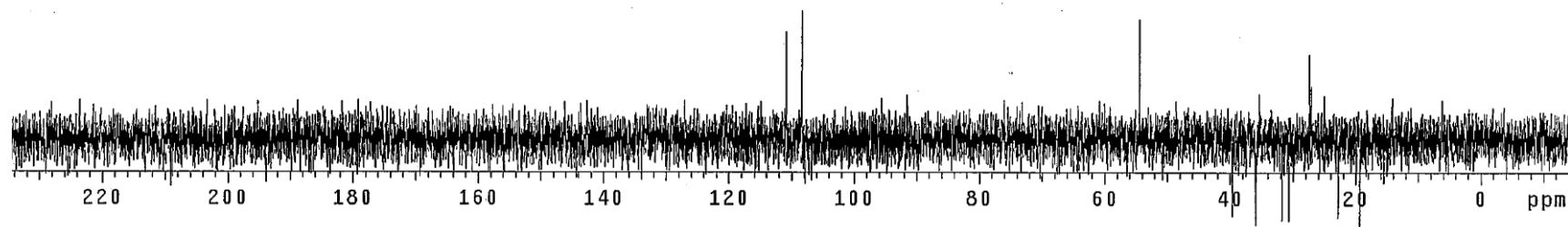
Pulse Sequence: DEPT

Solvent: CDCl<sub>3</sub>

Ambient temperature

Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 90.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
3753 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 0 min, 0 sec



**DEPT spectrum of compound 2 in (CDCl<sub>3</sub>, 100 MHz)**

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
48 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6198878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 4 hr, 18 min, 43 sec

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

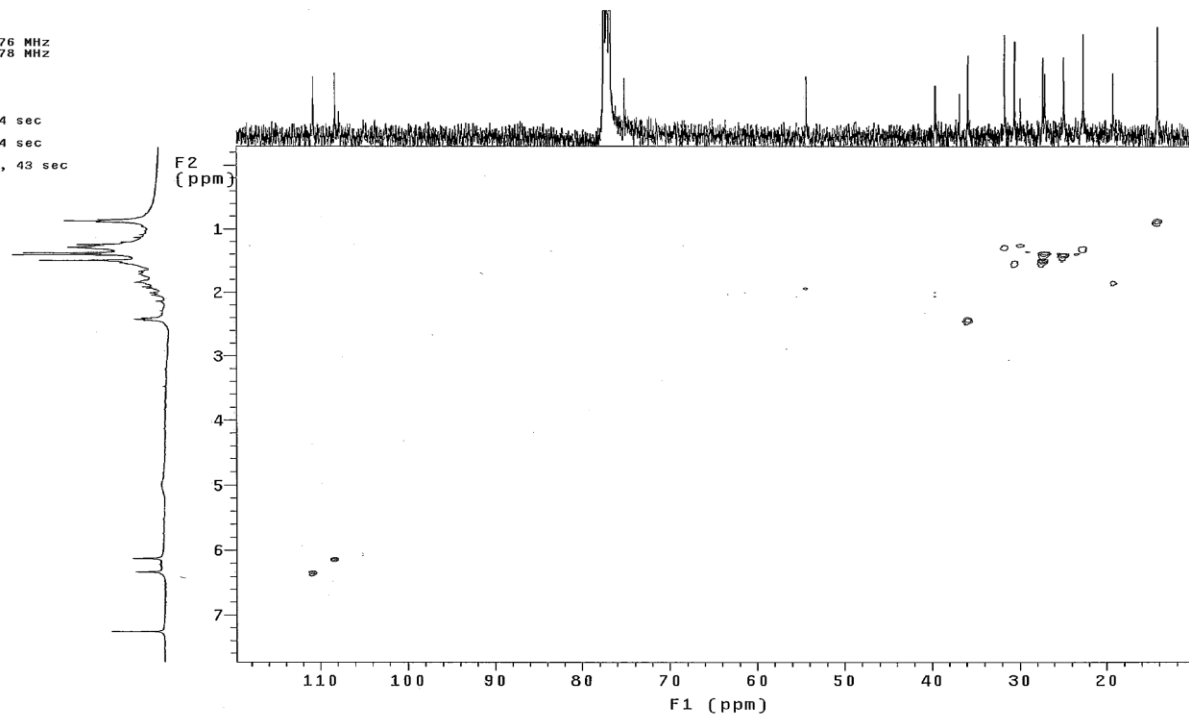
Pulse Sequence: gHMQC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON



2D-HMPC spectrum of compound 2

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory: /export/home/desmond/vnmrsys/data  
 File: PROTON

Pulse Sequence: gHMOC

Solvent: CDC13  
 Ambient temperature  
 Mercury-400BB "unknown"

Relax. delay 1.000 sec  
 Acq. time 0.160 sec  
 Width 6410.3 Hz  
 2D Width 17094.0 Hz  
 48 repetitions  
 2 x 128 increments  
 OBSERVE H1, 400.1252176 MHz  
 DECOUPLE C13, 100.6190878 MHz  
 Power 41 dB  
 on during acquisition  
 off during delay  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization 0.074 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.014 sec  
 FT size 2048 x 2048  
 Total time 4 hr, 18 min, 43 sec

SAAB-II-30-d

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory: /export/home/desmond/vnmrsys/data  
 File: PROTON

Pulse Sequence: gHMOC

Solvent: CDC13  
 Ambient temperature  
 Mercury-400BB

SAAB-II-06-8

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory: /export/home/desmond/vnmrsys/data  
 File: PROTON

Pulse Sequence: gHMOC

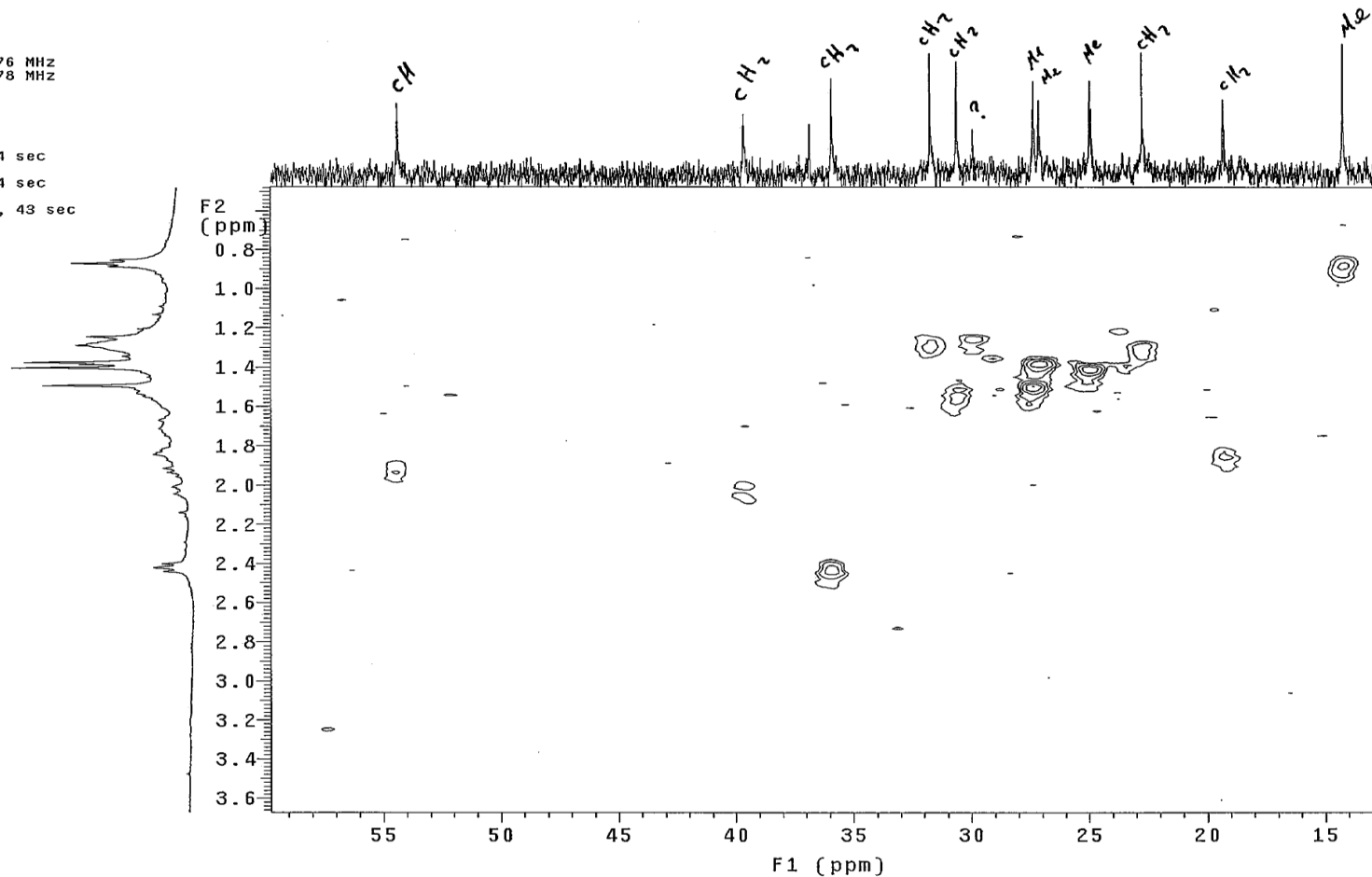
Solvent: CDC13  
 Ambient temperature  
 Mercury-400BB

SAAB-II-26-8

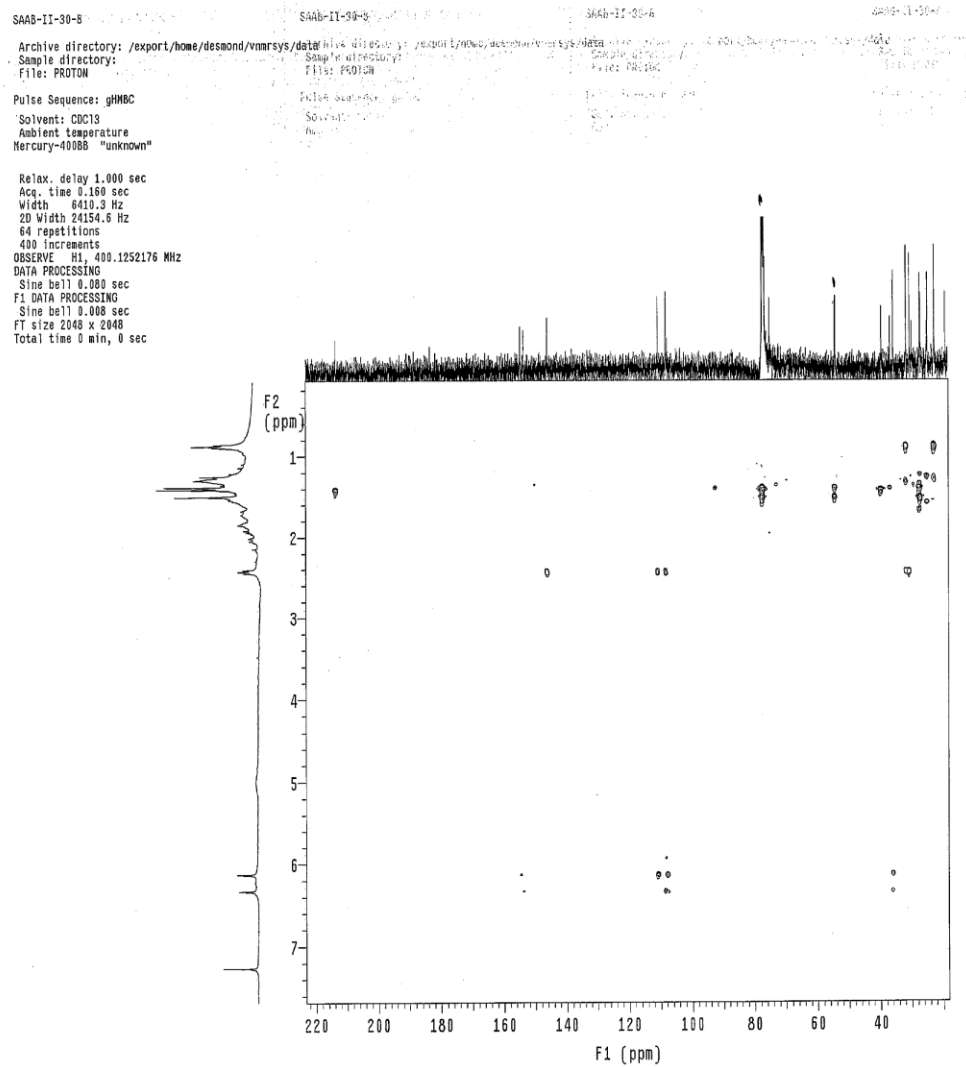
Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory: /export/home/desmond/vnmrsys/data  
 File: PROTON

Pulse Sequence: gHMOC

Solvent: CDC13  
 Ambient temperature  
 Mercury-400BB

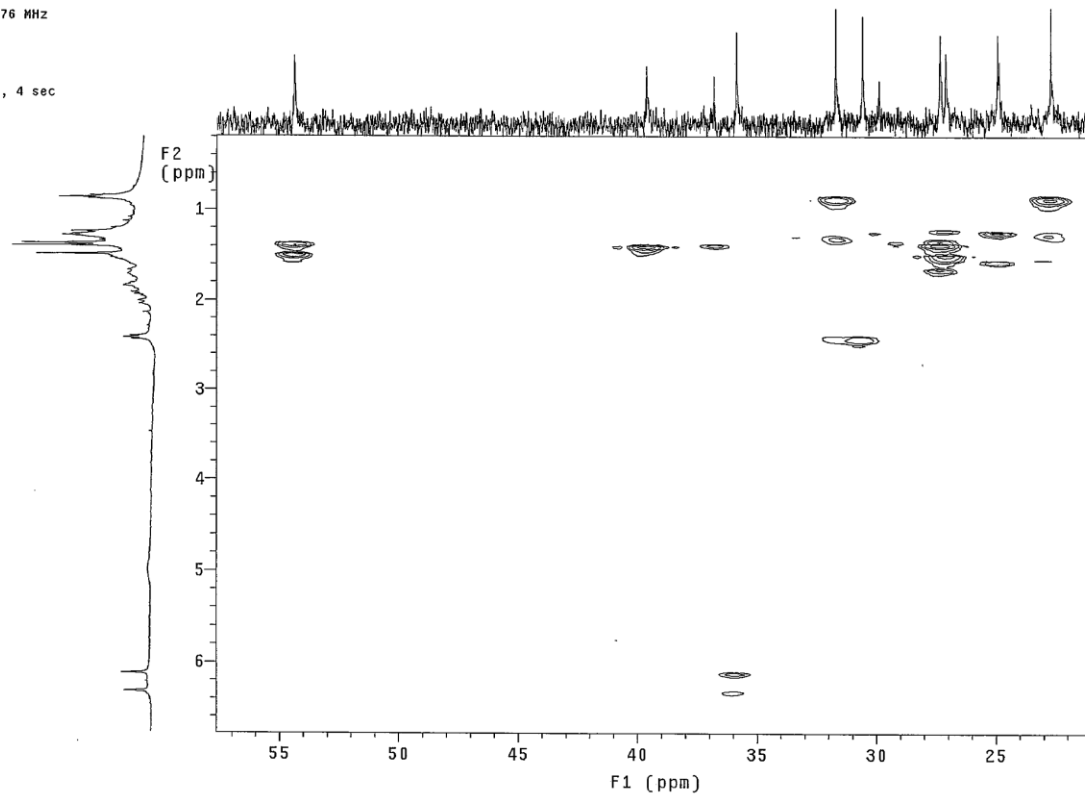


Partial expansions of the 2D-HMOC spectrum of compound 2



2D-HMBC spectrum of compound 2

SAAB-II-30-8	SAAB-II-30-8	SAAB-II-30-8	SAAB-II-30-8
Archive directory: /export/home/desmond/vnmrsys/data	Archive directory: /export/home/desmond/vnmrsys/data	Archive directory: /export/home/desmond/vnmrsys/data	Archive directory: /export/home/desmond/vnmrsys/data
Sample directory:	Sample directory:	Sample directory:	Sample directory:
File: PROTON	File: PROTON	File: PROTON	File: PROTON
Pulse Sequence: gHMBC	Pulse Sequence: gHMBC	Pulse Sequence: gHMBC	Pulse Sequence: gHMBC
Solvent: CDCl3	Solvent: CDCl3	Solvent: CDCl3	Solvent: CDCl3
Ambient temperature	Ambient temperature	Ambient temperature	Ambient temperature
Mercury-400BB "unknown"	Mercury-400BB "unknown"	Mercury-400BB "unknown"	Mercury-400BB "unknown"
Relax. delay 1.000 sec	Acq. time 0.160 sec	Width 6410.3 Hz	2D Width 24154.6 Hz
64 repetitions	400 increments	OBSERVE H1, 400.1252176 MHz	DATA PROCESSING
Sine bell 0.000 sec	F1 DATA PROCESSING	Sine bell 0.000 sec	FT size 2048 x 2048
Total time 9 hr, 13 min, 4 sec			



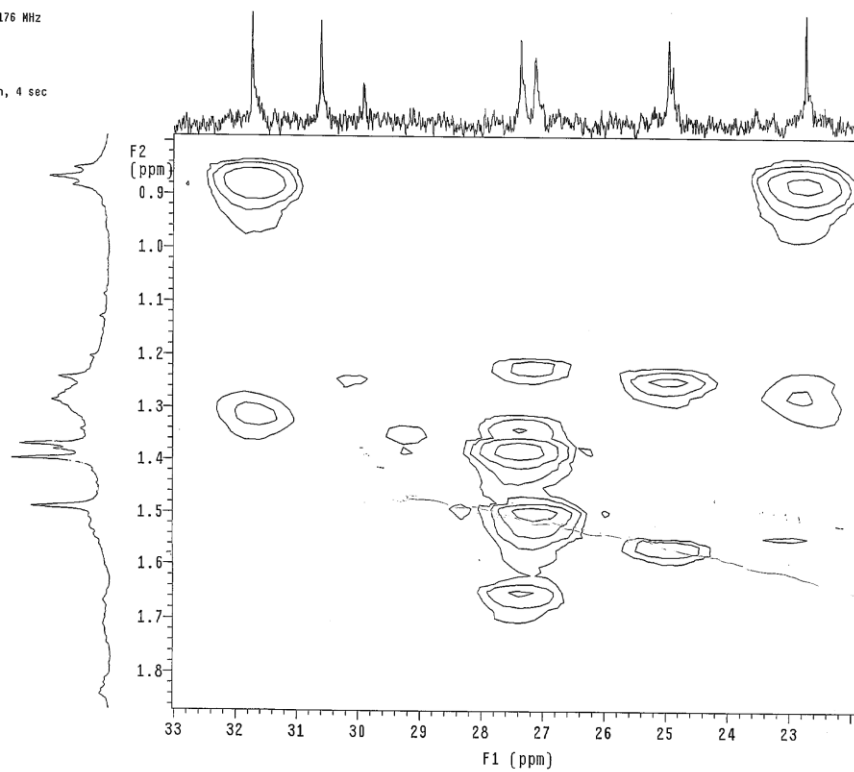
Partial expansions of the 2D-HMBC spectrum of compound 2

SAAB-II-30-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

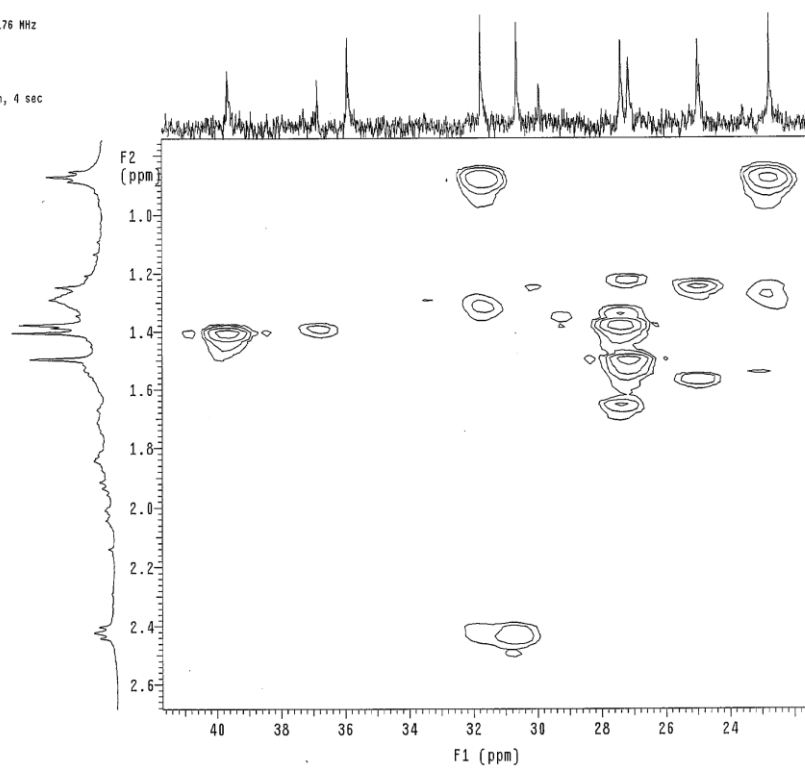
Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient Temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
ZD Width 24154.6 Hz  
64 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.000 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
F1 size 2048 x 2048  
Total time 9 hr, 13 min, 4 sec

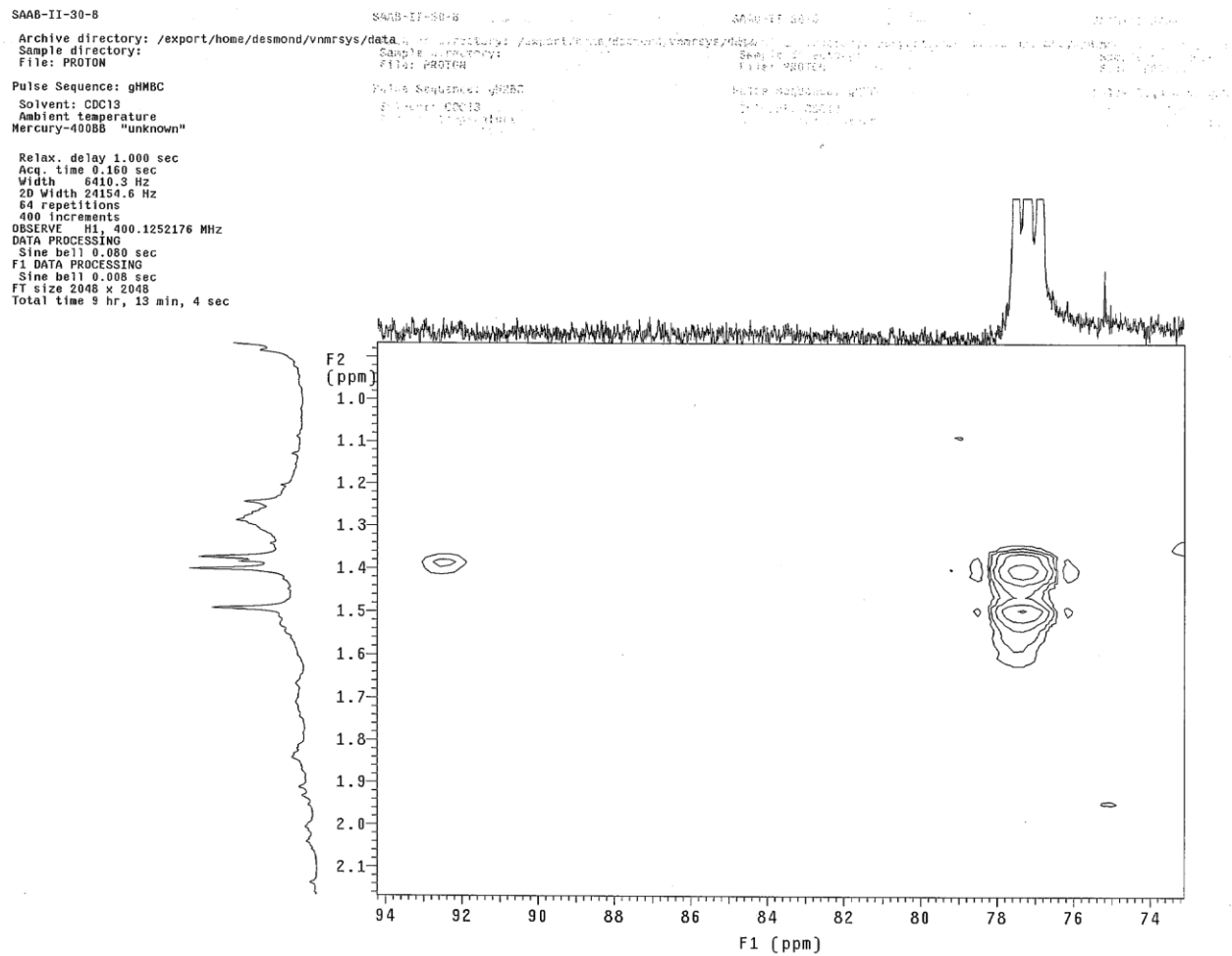


Partial expansions of the 2D-HMBC spectrum of compound 2

SAAB-II-30-8 SAAB-II-30-8 SAAB-II-30-8 SAAB-II-30-8  
Archive directory: /export/home/desmond/vmarsys/data/...  
Sample directory: ...  
File: PROTON  
Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-40068 "unknown"  
Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6419.3 Hz  
2D Width 24154.6 Hz  
64 repetitions  
400 increments  
OBSERVE F1: 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 13 min, 4 sec



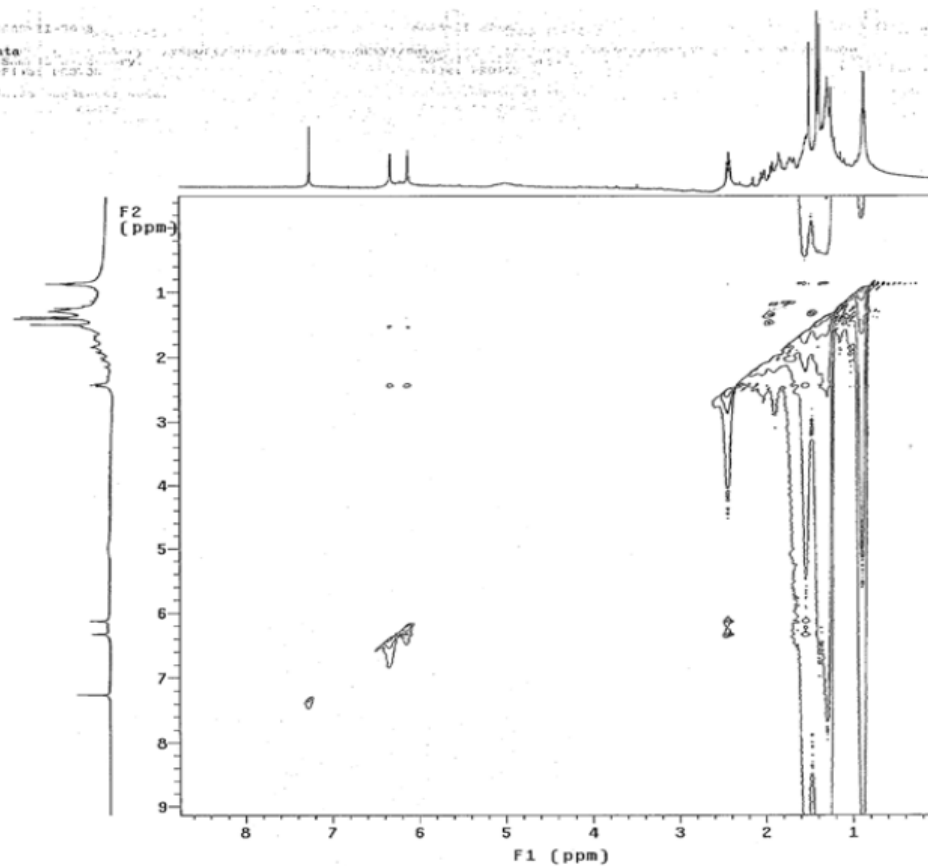
Partial expansions of the 2D-HMBC spectrum of compound 2



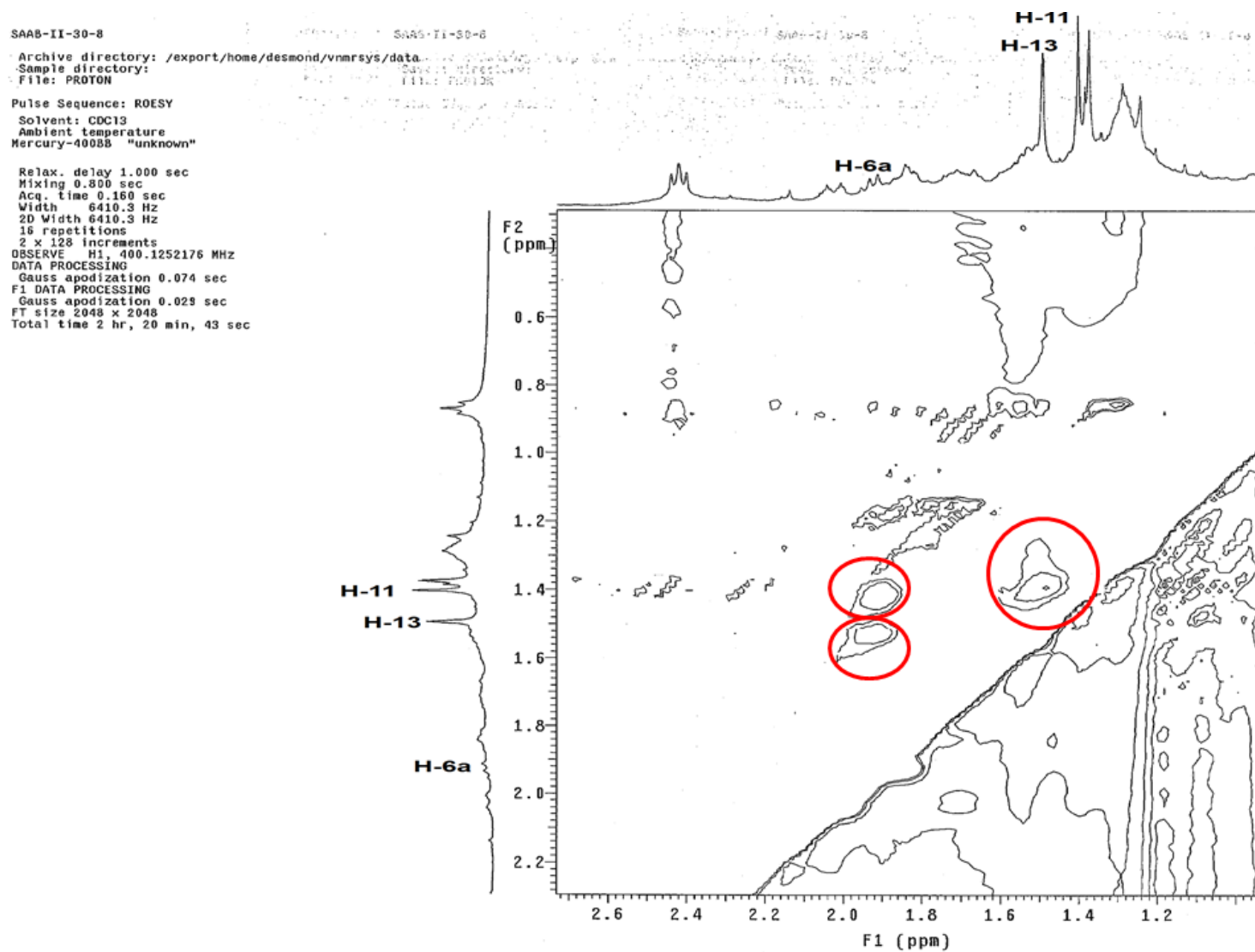
Partial expansions of the 2D-HMBC spectrum of compound 2



SAAB-II-30-8  
Archive directory: /export/hose/desmond/vnmrsys/data  
Sample directory: /export/hose/desmond/vnmrsys/data  
File: PROTON  
Pulse Sequence: ROESY  
Solvent: CDC13  
Ambient temperature  
Mercury-4095B "unknown"  
Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
18 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.028 sec  
F1 size 2048 x 2048  
Total time 2 hr, 20 min, 43 sec



2D-ROESY spectrum of compound 2

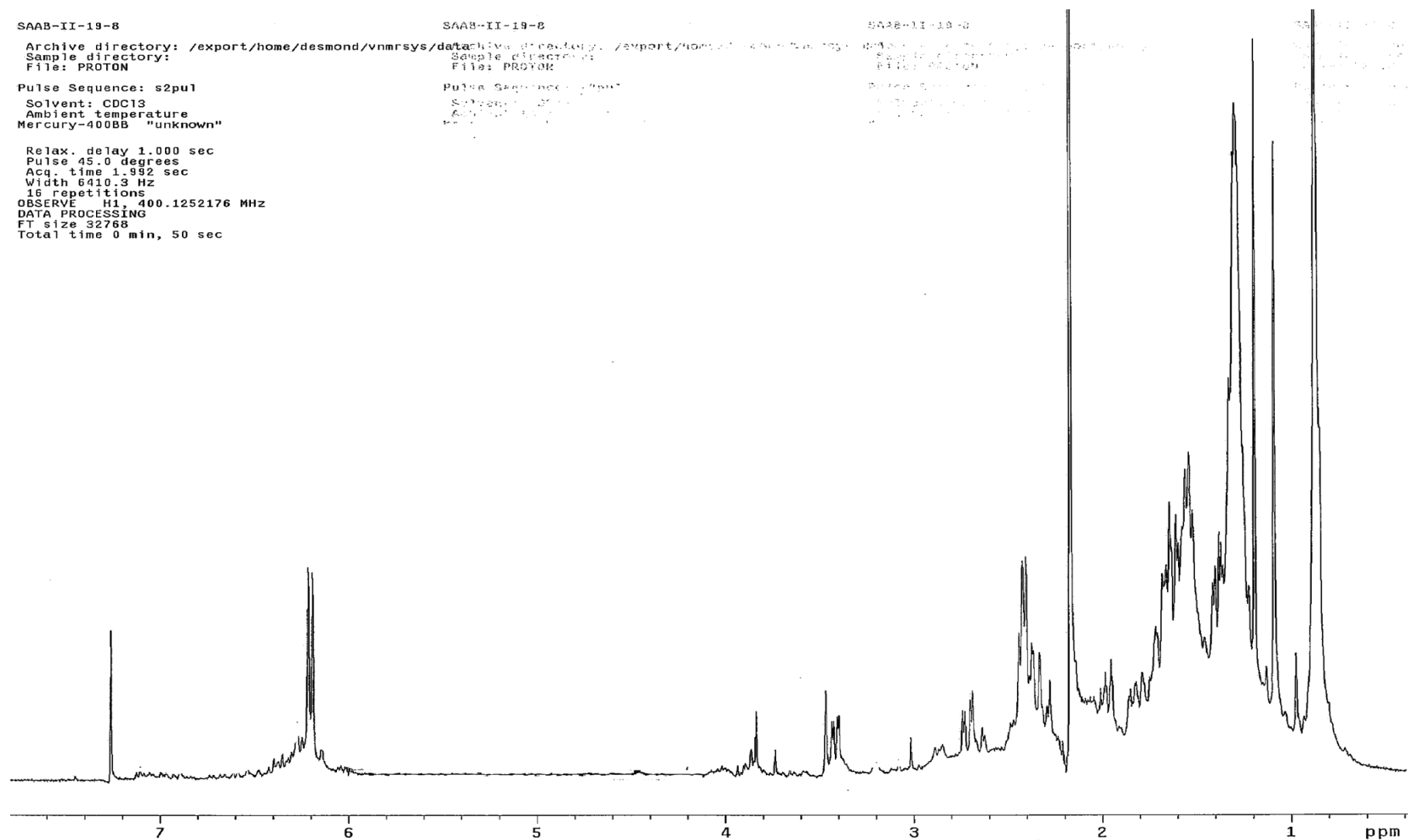


2D-ROESY spectrum of compound 2

SAAB-II-19-8  
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
16 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 50 sec

SAAB-II-19-8  
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

SAAB-II-19-8  
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"



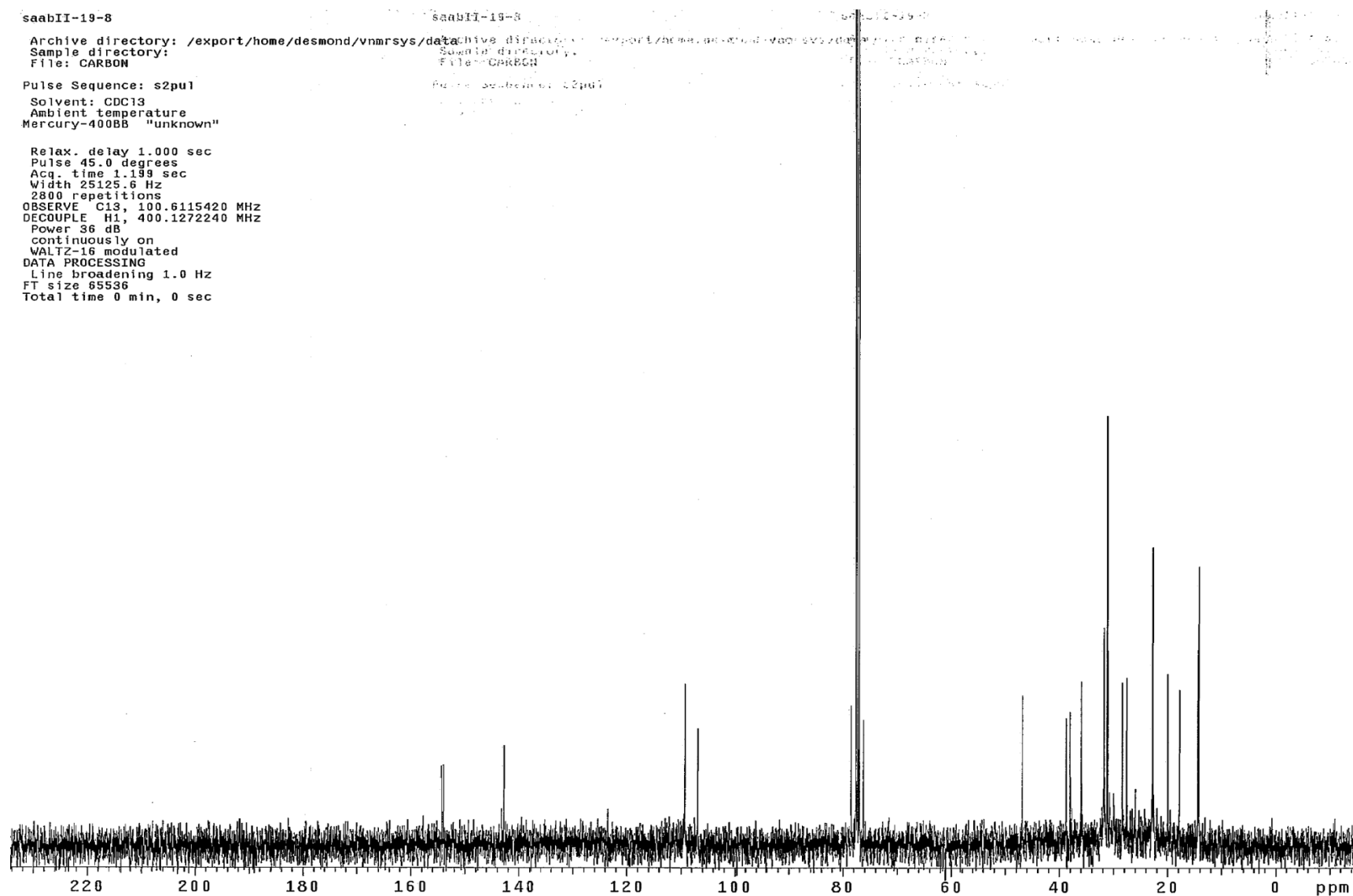
**$^1\text{H}$ -NMR spectrum of compound 3 in ( $\text{CDCl}_3$ , 400 MHz)**

saabII-19-8

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

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
2800 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 0 min, 0 sec



$^{13}\text{C}$ -NMR spectrum of compound 3 in ( $\text{CDCl}_3$ , 100 MHz)

SAAB-II-19-8

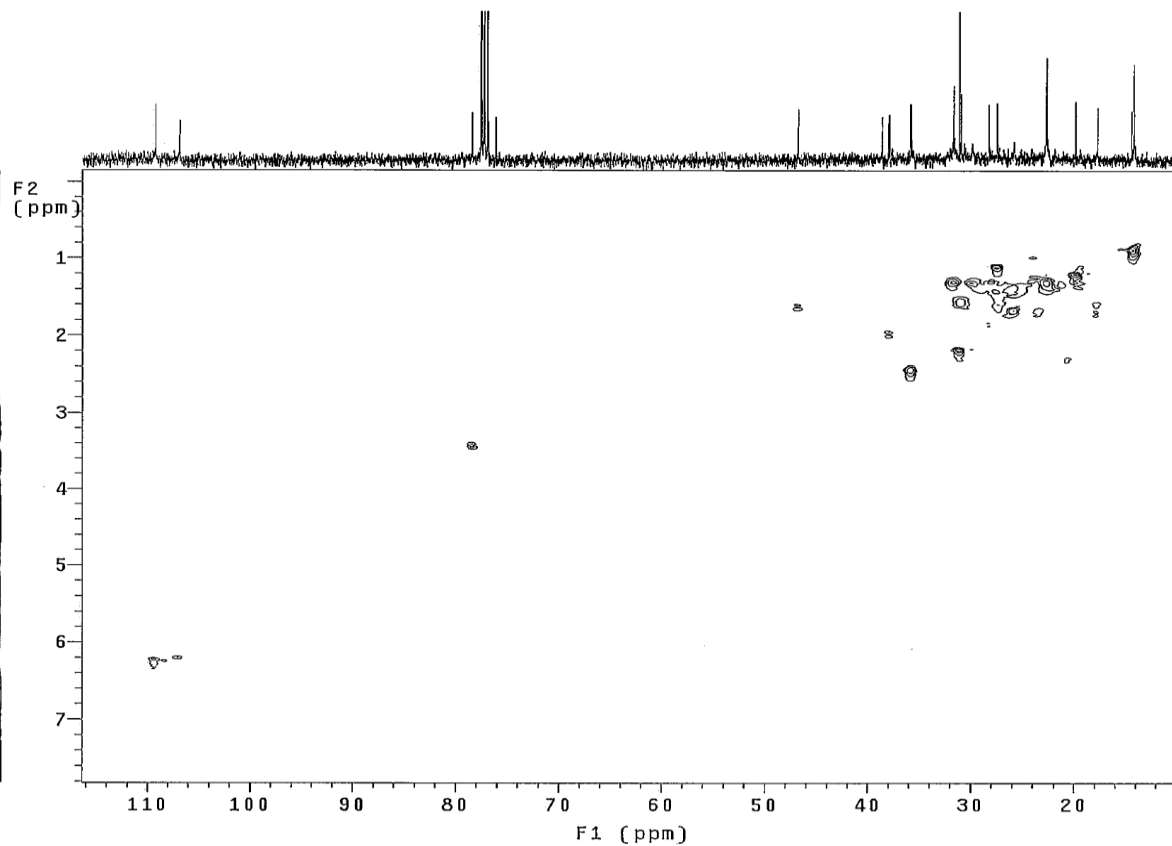
SAAB-II-19-8

SAAB-II-19-8

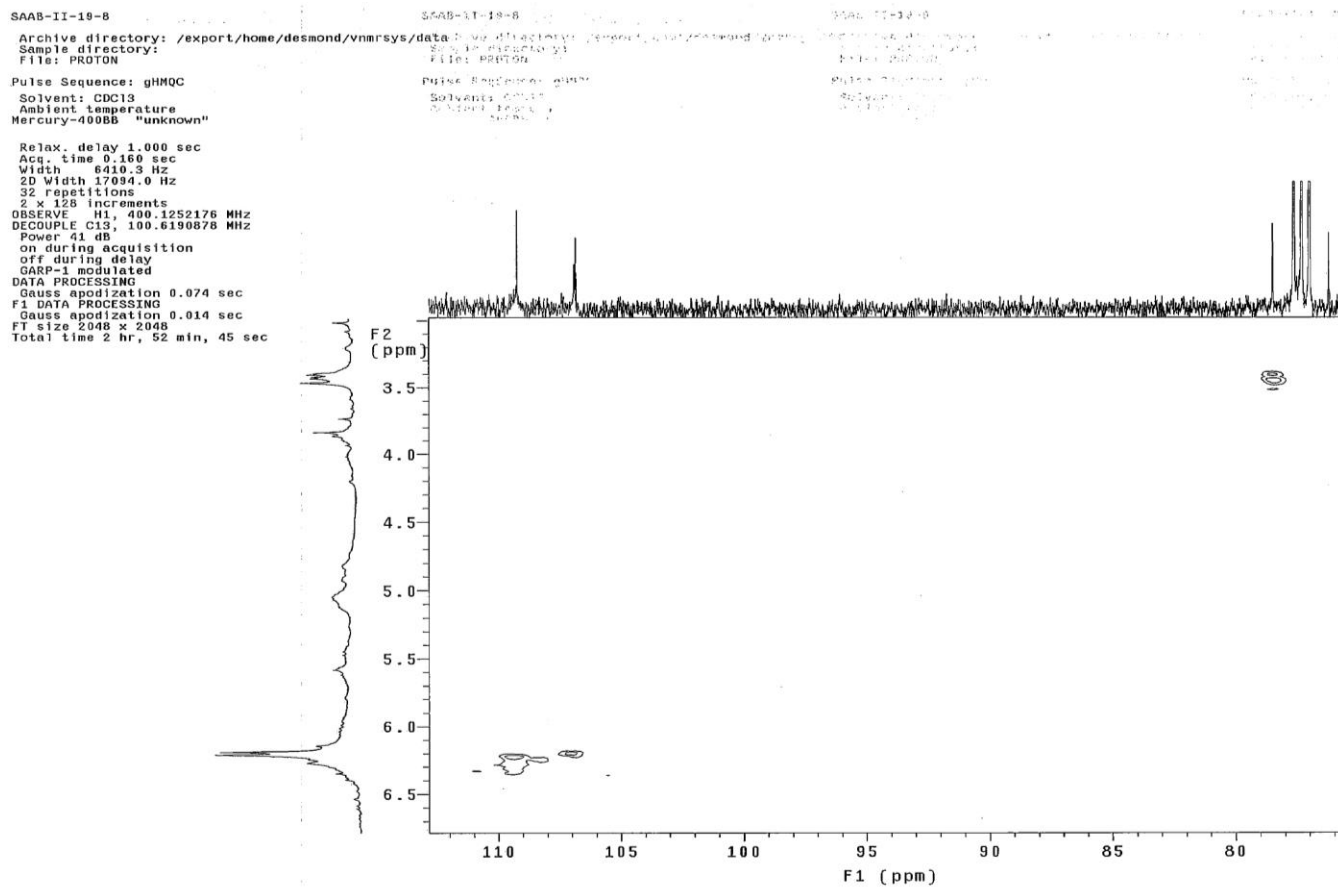
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



2D-HMQC spectrum of compound 3



Partial expansions of the 2D-HMPC spectrum of compound 3

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
OFF during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"

SAAB-II-19-8

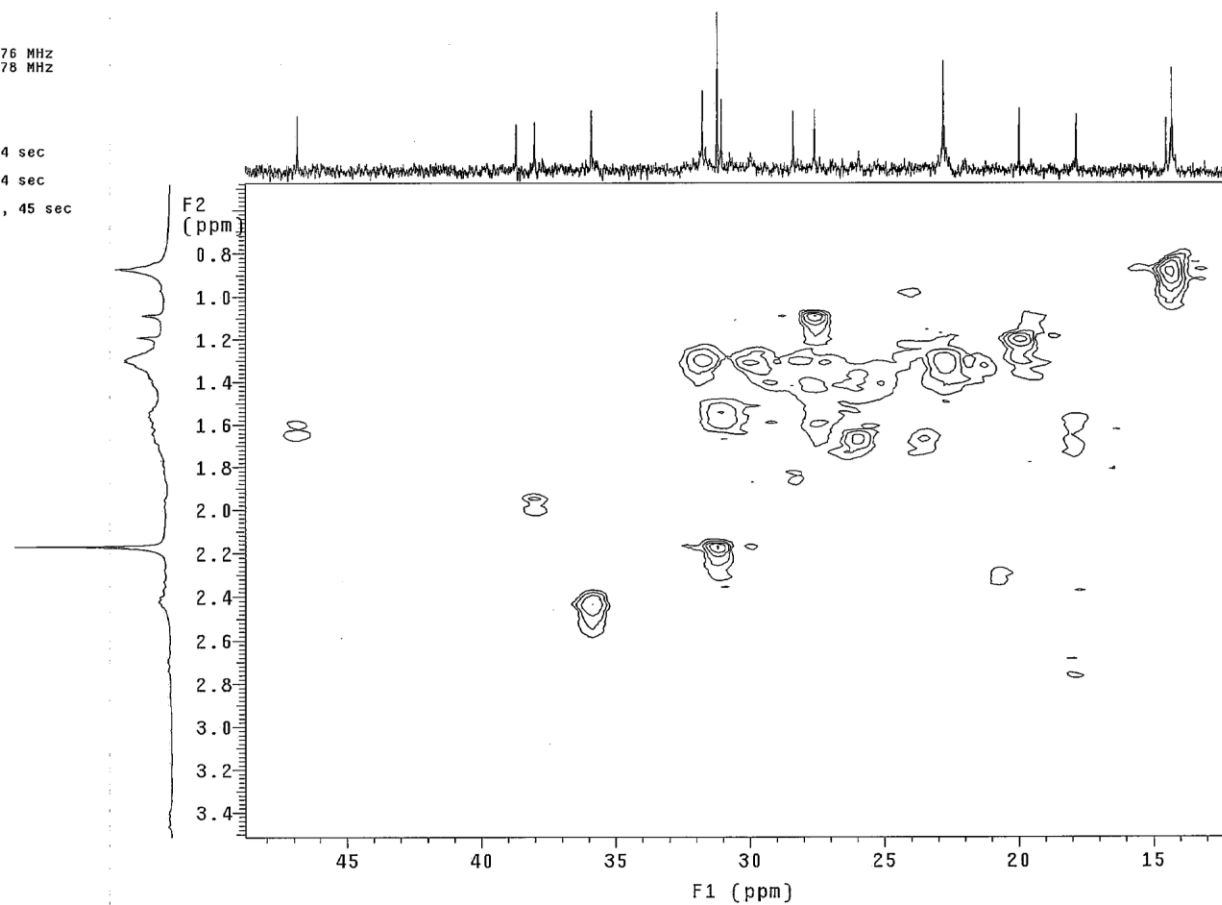
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"



Partial expansions of the 2D-HMPC spectrum of compound 3

SAAB-II-19-8

SAAB-II-19-8

SAAB-II-19-8

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

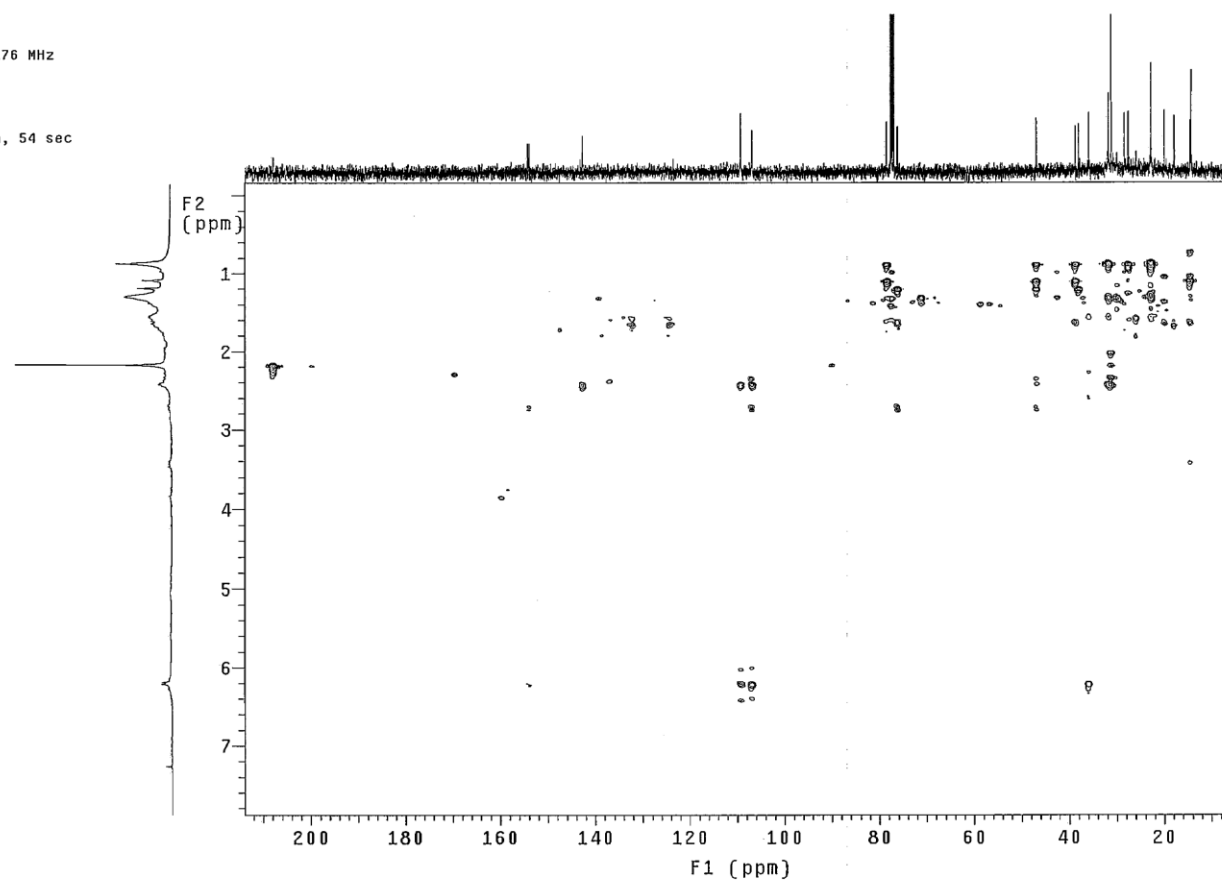
Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Pulse Sequence: gHMBC

Pulse Sequence: gHMBC

Pulse Sequence: gHMBC

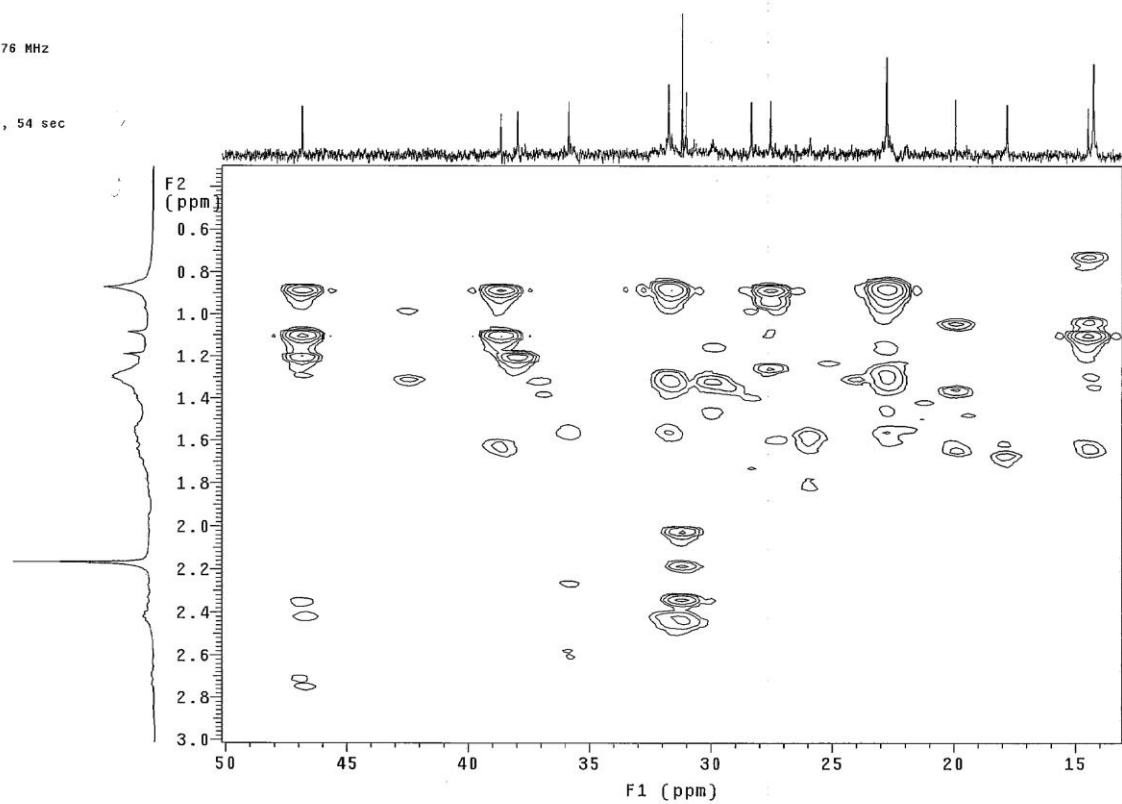
Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



2D-HMBC spectrum of compound 3



SAAB-II-19-8 SAAB-II-19-8 SAAB-II-19-8 SAAB-II-19-8  
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON  
Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



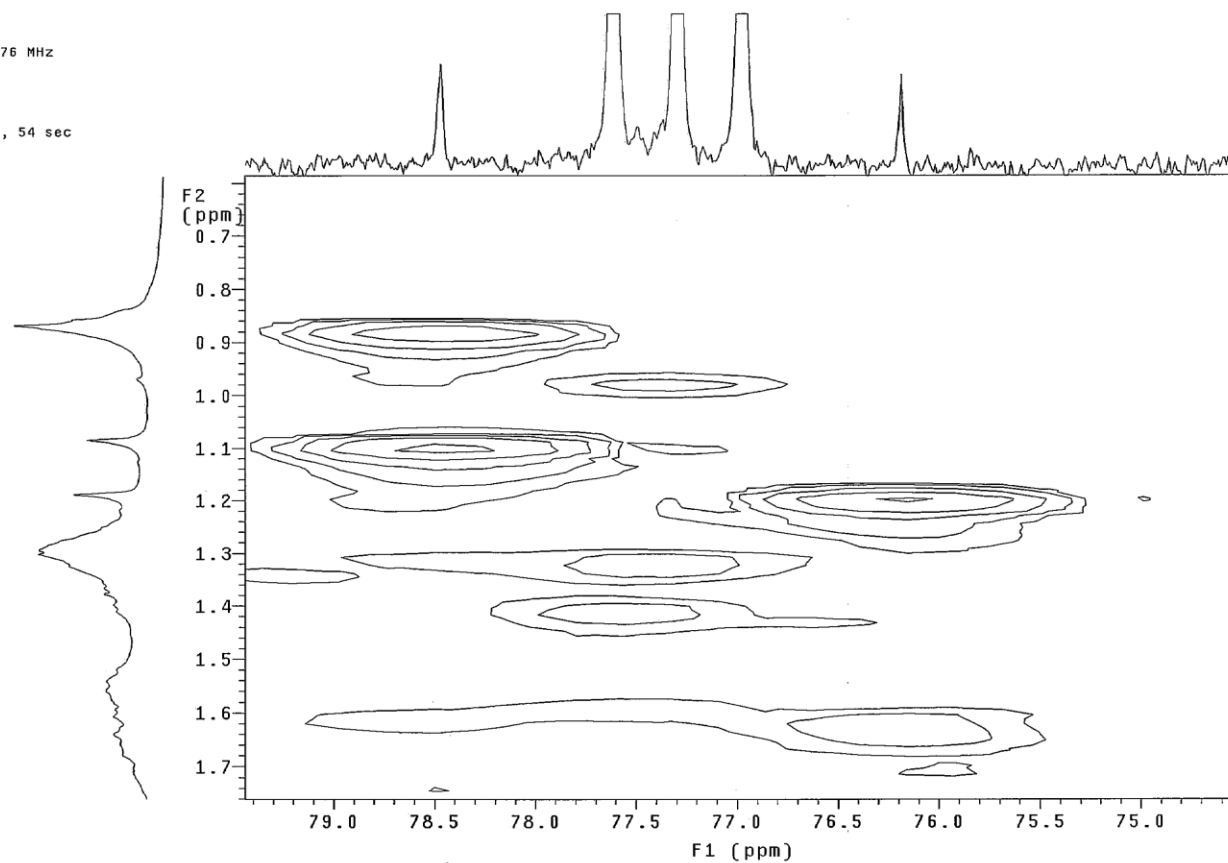
Partial expansions of the 2D-HMBC spectrum of compound 3

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsws/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



Partial expansions of the 2D-HMBC spectrum of compound 3

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory:  
 File: PROTON

Pulse Sequence: gHMBC  
 Solvent: CDCl3  
 Ambient temperature  
 Mercury-400BB "unknown"

Relax. delay 1.000 sec  
 Acq. time 0.160 sec  
 Width 6410.3 Hz  
 2D Width 24154.6 Hz  
 32 repetitions  
 400 increments  
 OBSERVE H1, 400.1252176 MHz  
 DATA PROCESSING  
 Sine bell 0.080 sec  
 F1 DATA PROCESSING  
 Sine bell 0.008 sec  
 FT size 2048 x 2048  
 Total time 4 hr, 37 min, 54 sec

SAAB-II-19-0

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory:  
 File: PROTON

Pulse Sequence: gHMBC  
 Solvent: CDCl3  
 Ambient temperature  
 Mercury-400BB "unknown"

SAAB-II-19-8

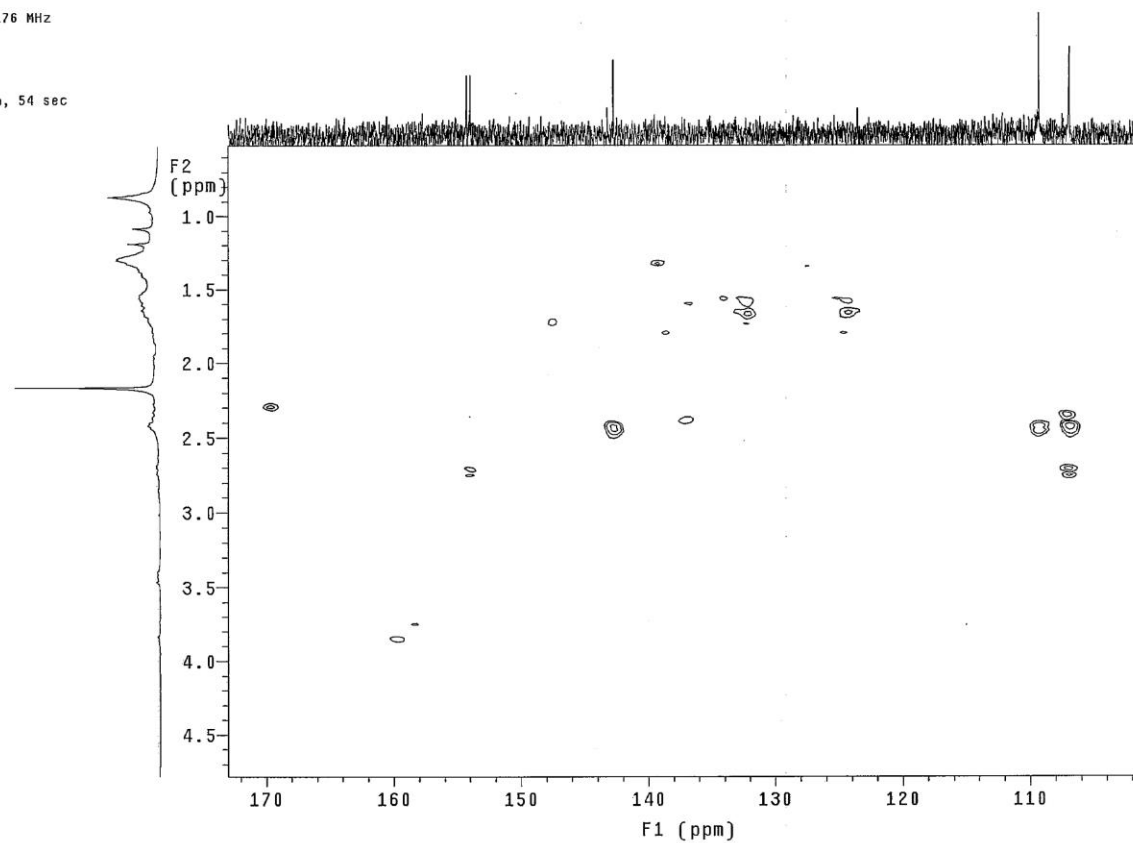
Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory:  
 File: PROTON

Pulse Sequence: gHMBC  
 Solvent: CDCl3  
 Ambient temperature  
 Mercury-400BB "unknown"

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
 Sample directory:  
 File: PROTON

Pulse Sequence: gHMBC  
 Solvent: CDCl3  
 Ambient temperature  
 Mercury-400BB "unknown"



Partial expansions of the 2D-HMBC spectrum of compound 3

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsws/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
ZD Width 6410.3 Hz  
16 repetitions  
128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 42 min, 39 sec

SAAB-II-19-8

Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13

SAAB-II-19-8

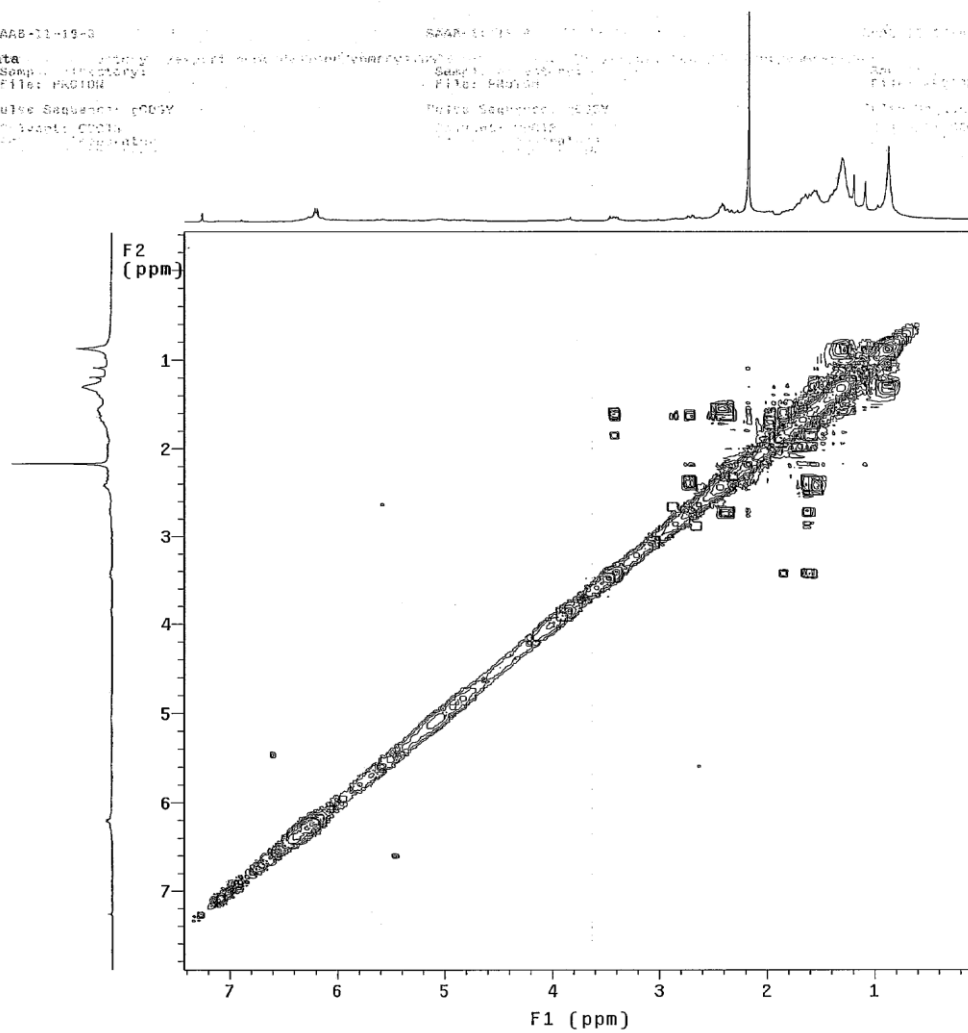
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13

SAAB-II-19-8

Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13



2D-COSY spectrum of compound 3

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
16 repetitions  
128 increments  
OBSERVE HI, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 42 min, 39 sec

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"

SAAB-II-19-8

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"

SAAB-II-19-8

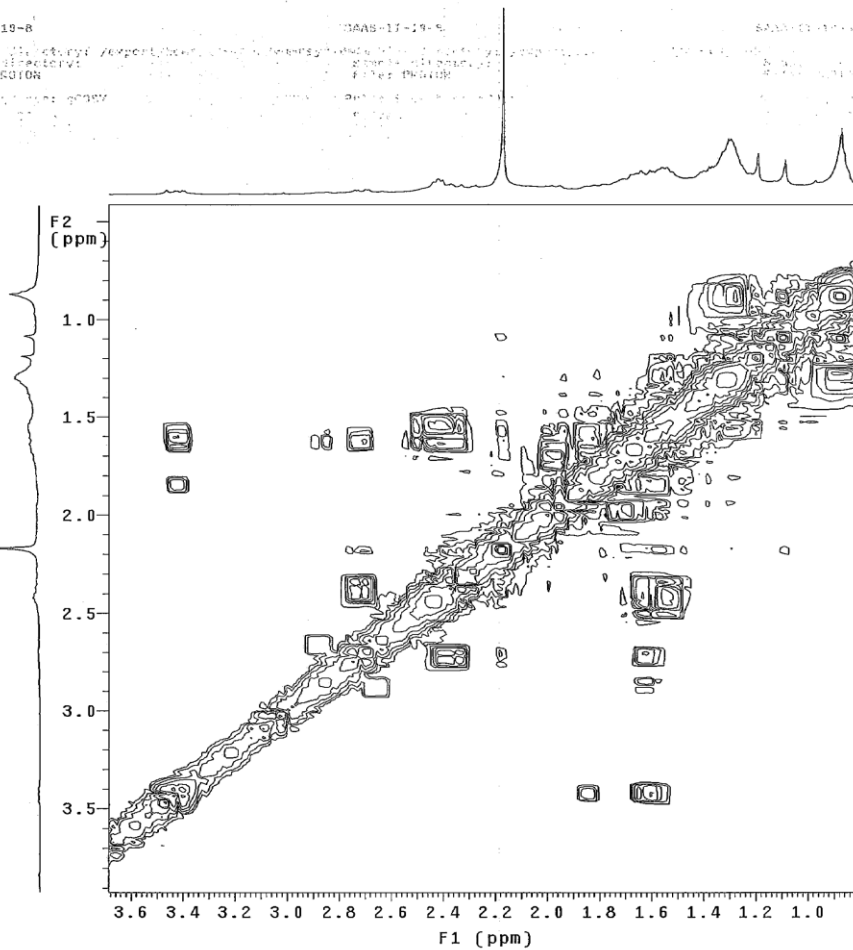
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory: /export/home/desmond/vnmrsys/data  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl3

Ambient temperature

Mercury-400BB "unknown"



Partial expansions of the 2D-COSY spectrum of compound 3

SAAB-19-B

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 3.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
30 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
FT size 2048 x 2048  
Total time 0 min, 0 sec

SAAB-19-B

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

SAAB-19-B

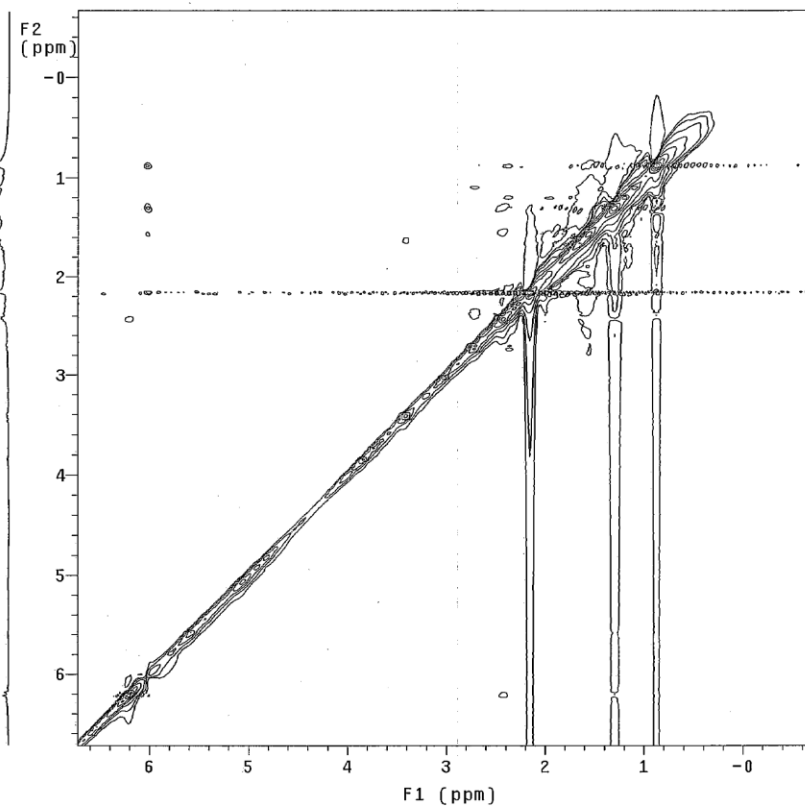
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

SAAB-19-B

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

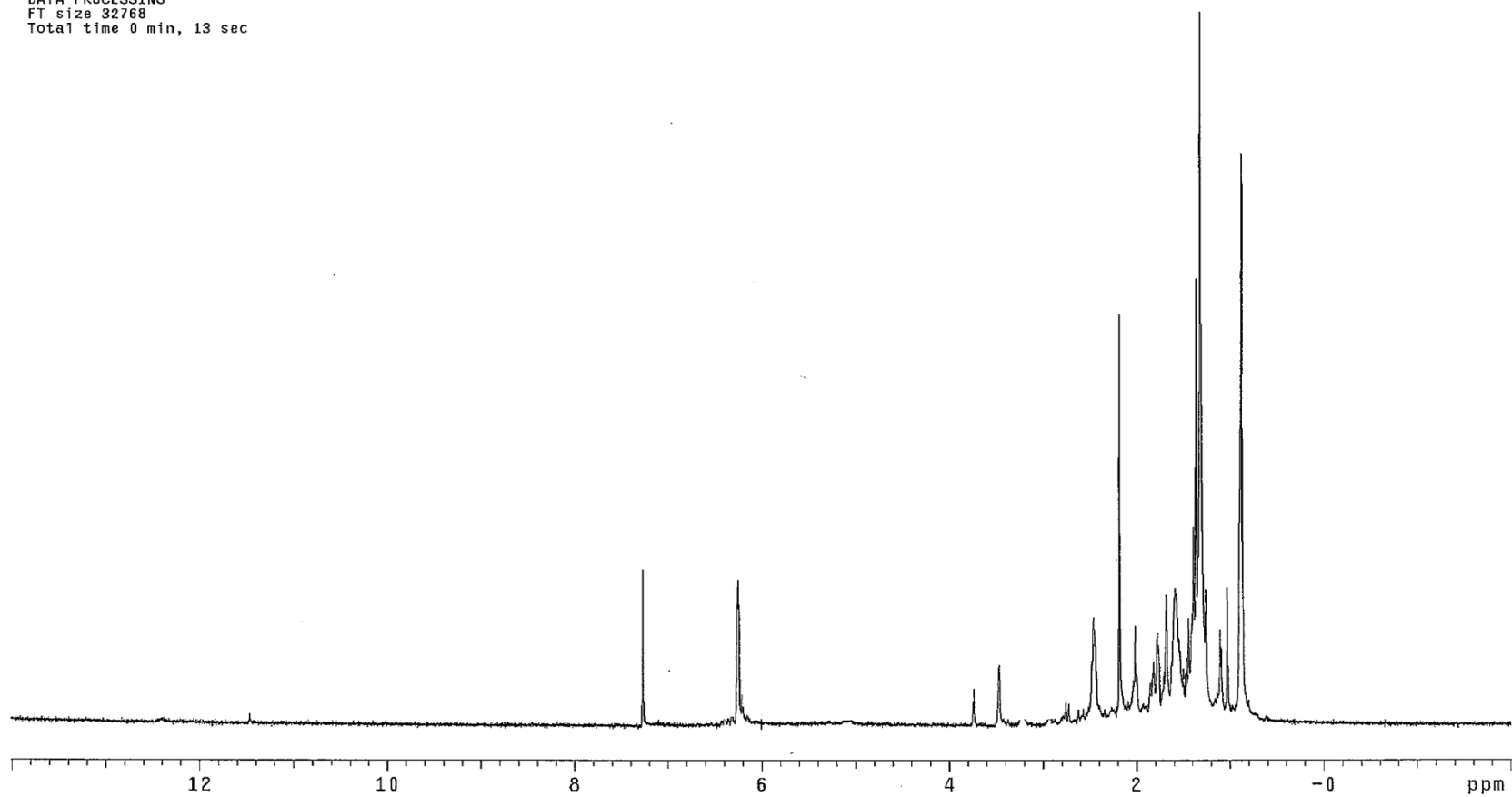


2D-ROESY spectrum of compound 3

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
4 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 13 sec



**Fig (12) <sup>1</sup>H-NMR spectrum of compound 4 in (CDCl<sub>3</sub>, 400 MHz)**

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

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
6000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 3 hr, 48 min, 56 sec

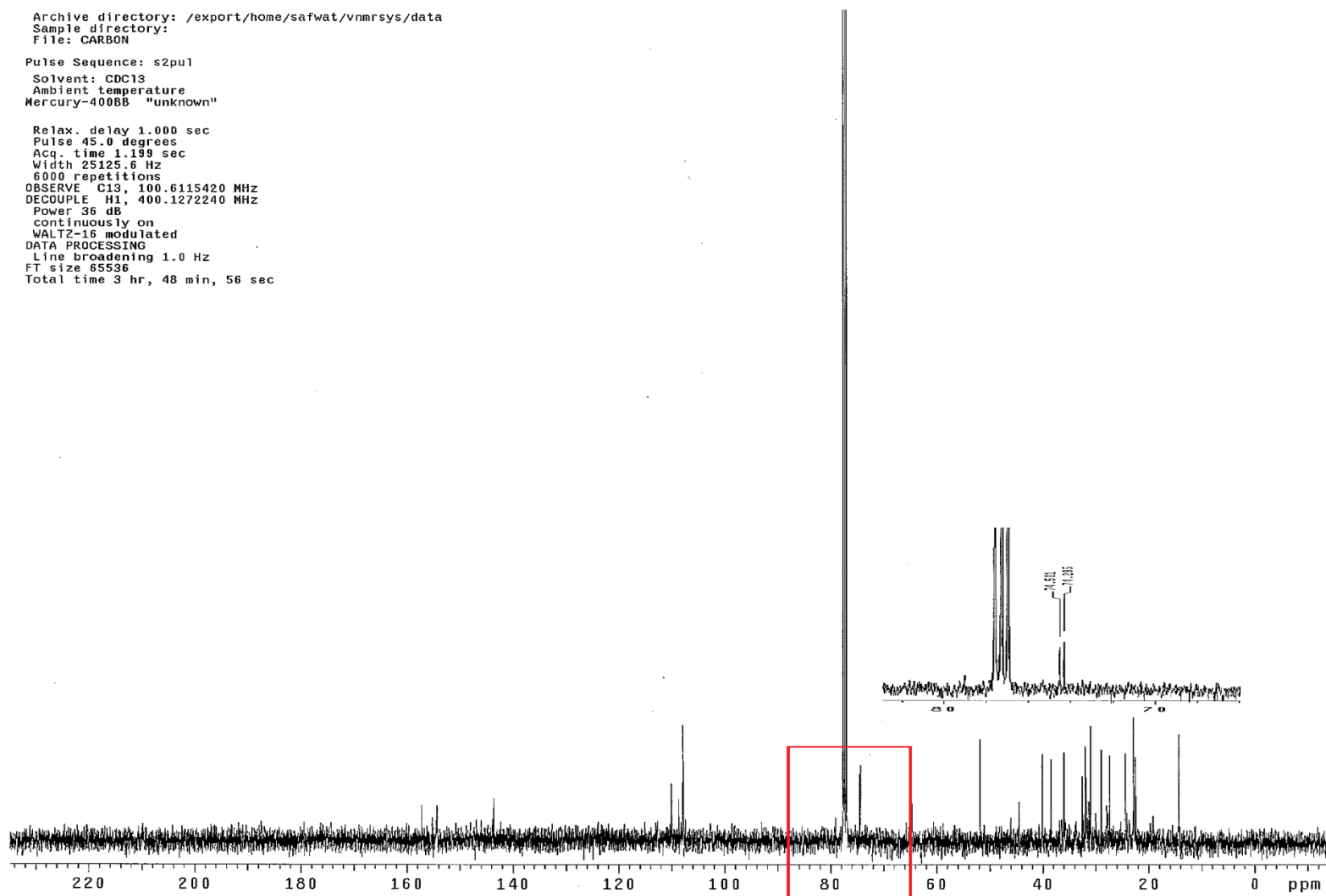


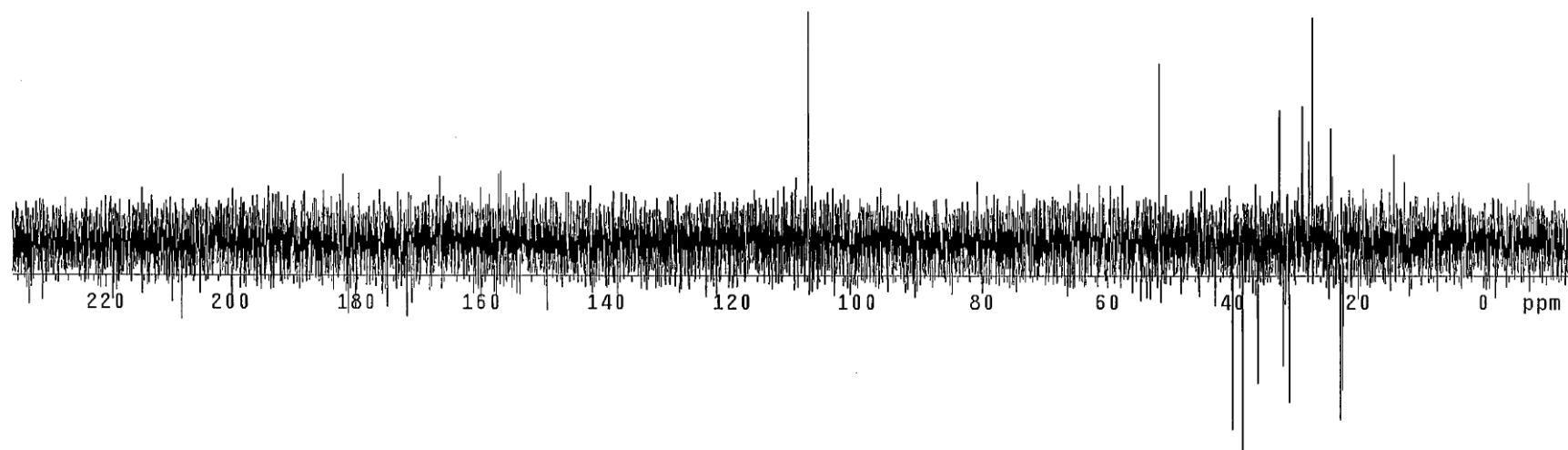
Fig (13)  $^{13}\text{C}$ -NMR spectrum of compound 4 in ( $\text{CDCl}_3$ , 100 MHz)



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

Pulse Sequence: DEPT  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 90.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
2000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 1 hr, 17 min, 13 sec



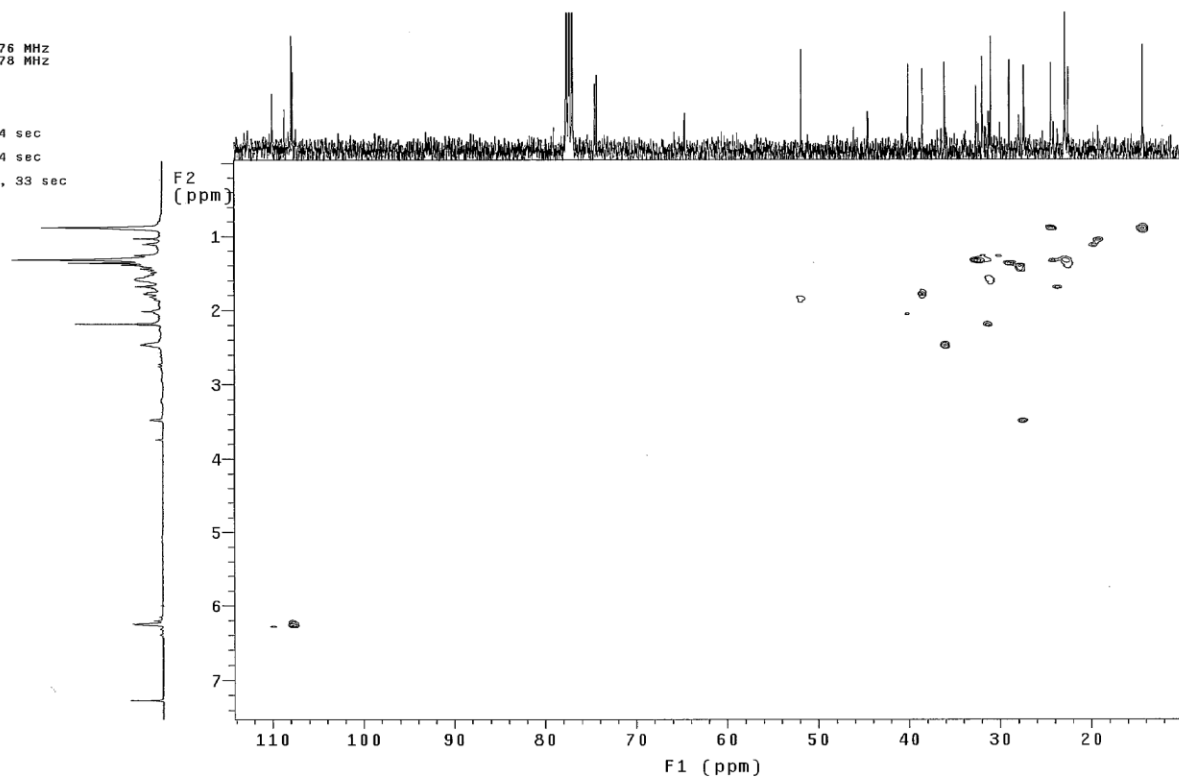
**Fig (14) DEPT spectrum of compound 4 in (CDCl<sub>3</sub>, 100 MHz)**

MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-40055 "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
24 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 10 min, 33 sec



2D-HMOC spectrum of compound 4

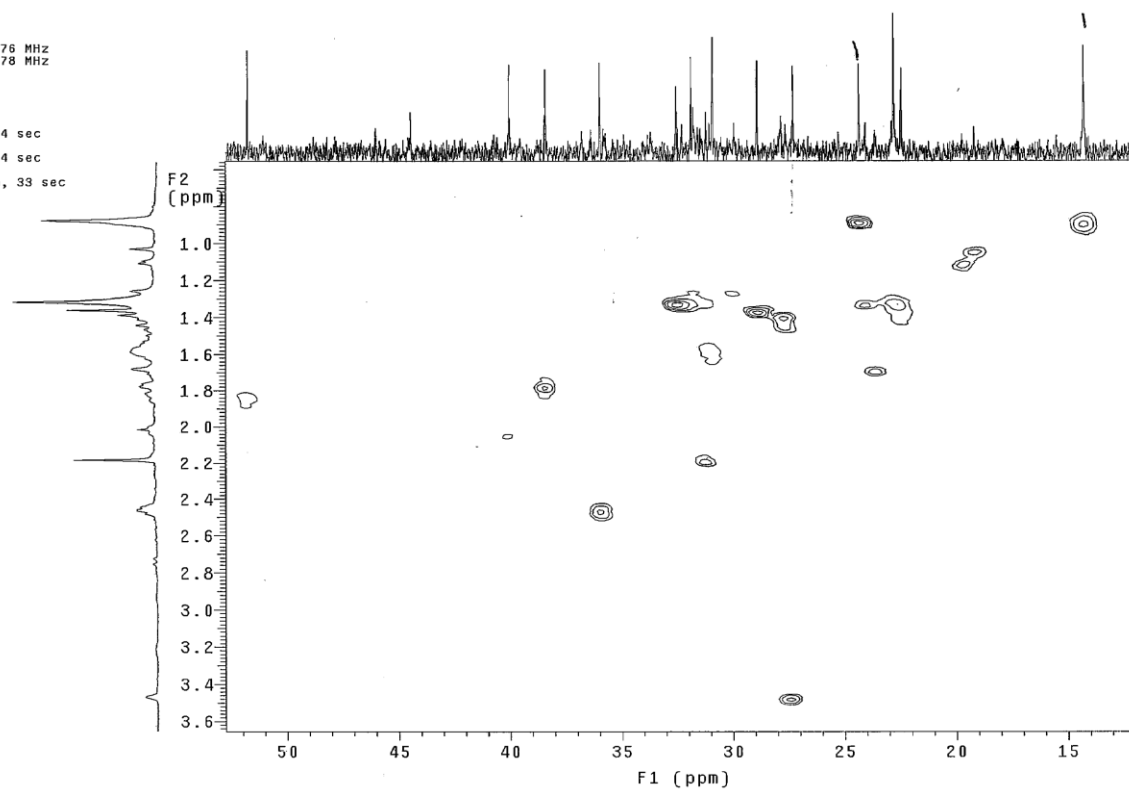
MRRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
24 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
F1 size 2048 x 2048  
Total time 2 hr, 10 min, 33 sec



Partial expansions of the 2D-HMPC spectrum of compound 4

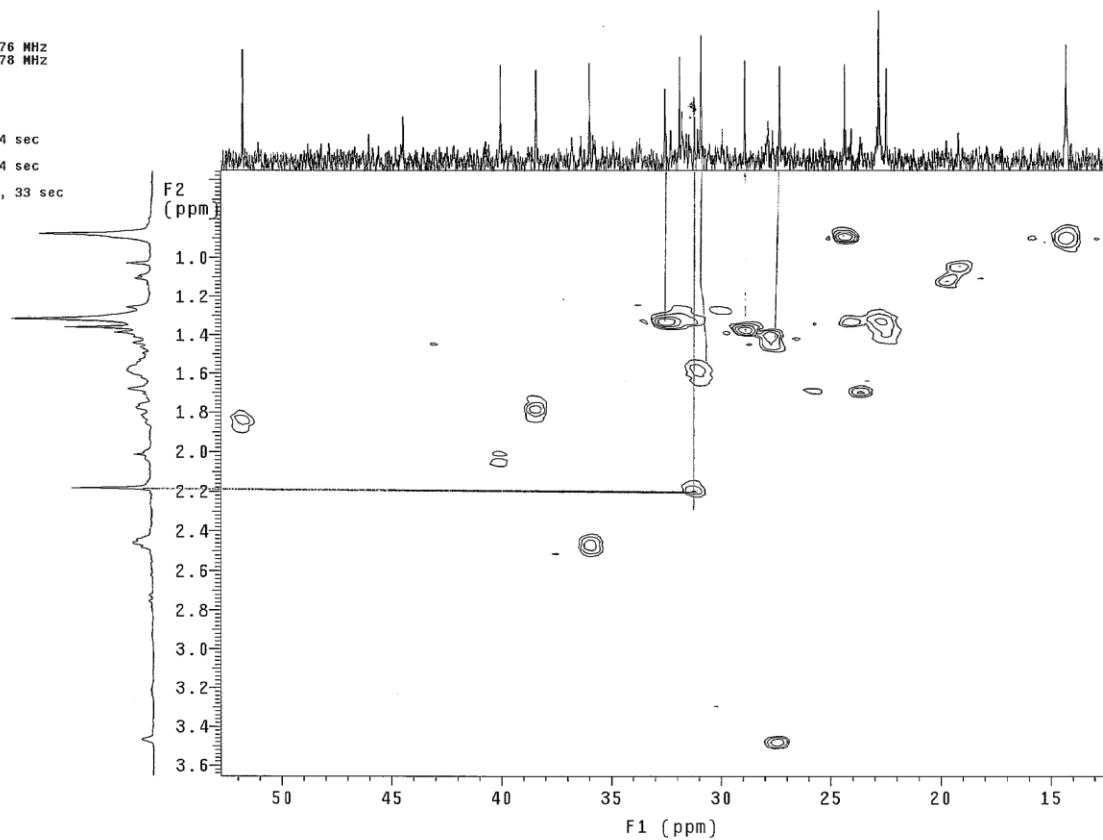
MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrsws/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDC13  
Ambient temperature  
Mercury-400SB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
24 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 10 min, 33 sec



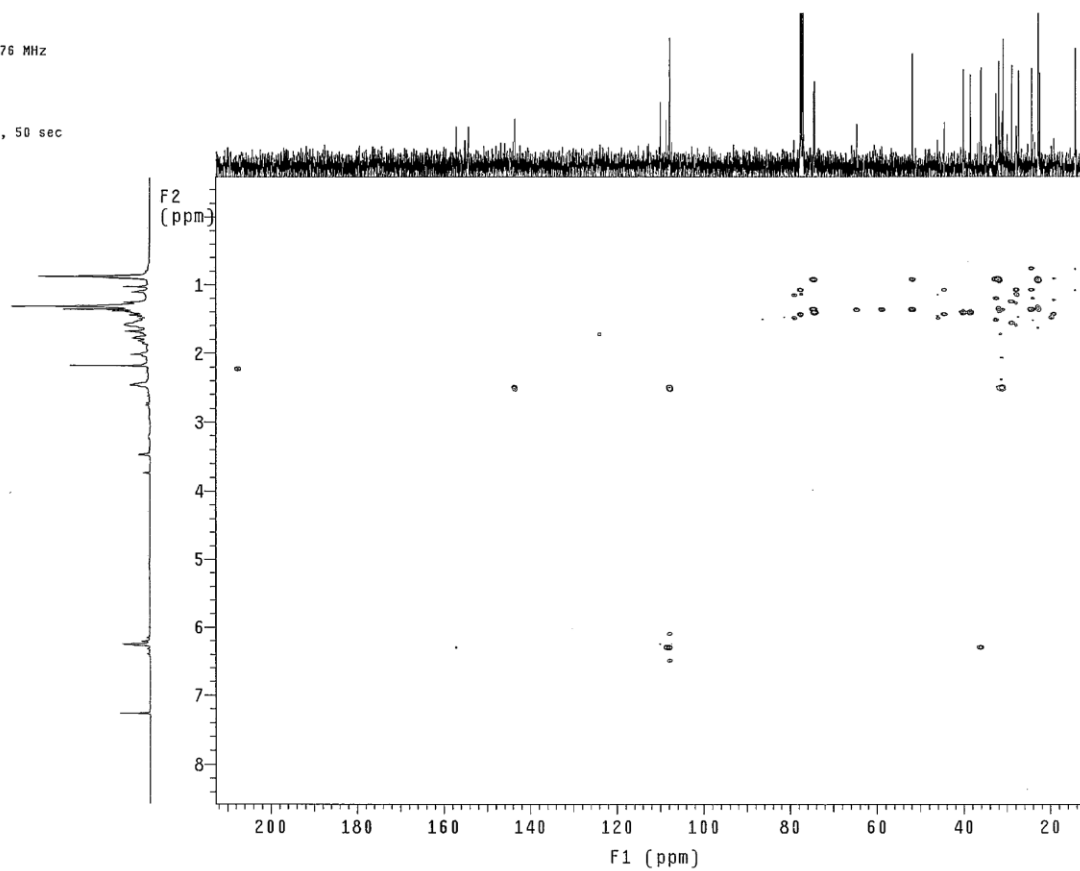
Partial expansions of the 2D-HMPC spectrum of compound 4

MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
24 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 29 min, 50 sec

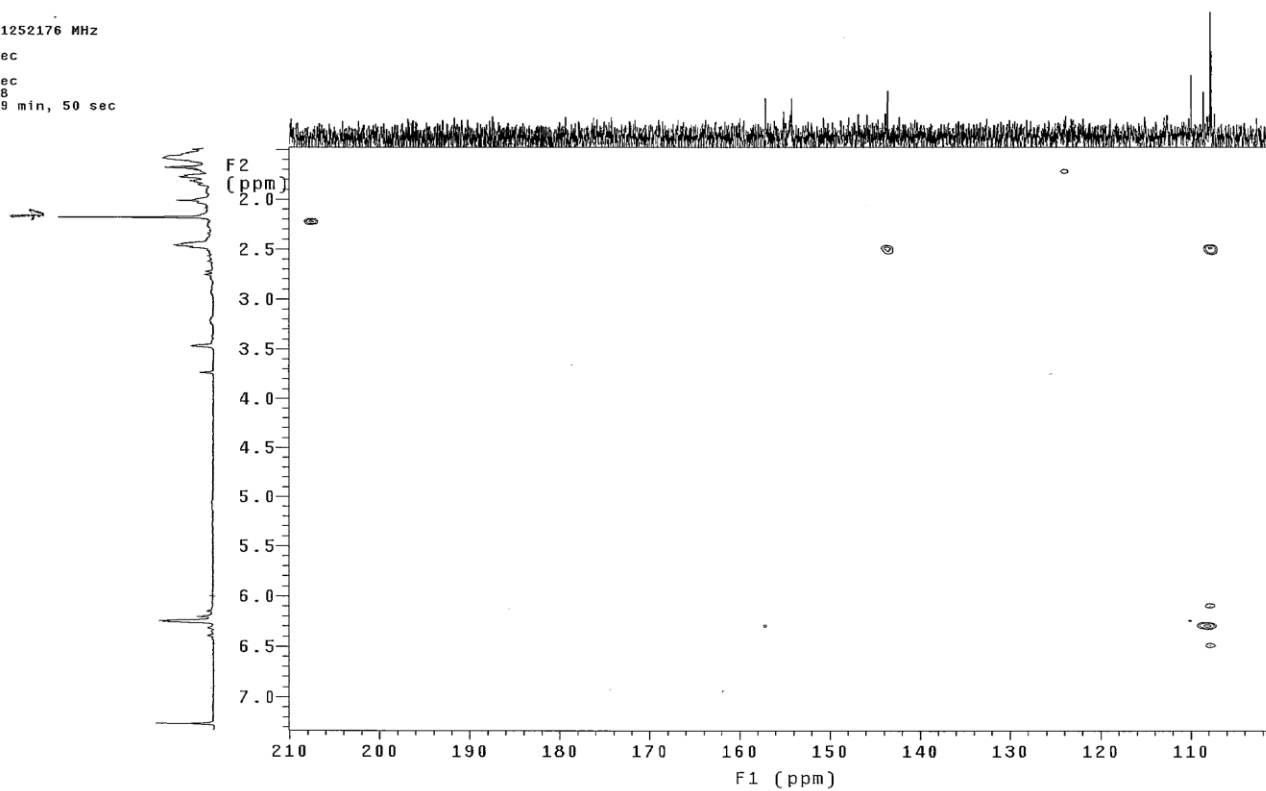
**2D-HMBC spectrum of compound 4**

MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.180 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
24 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 29 min, 50 sec



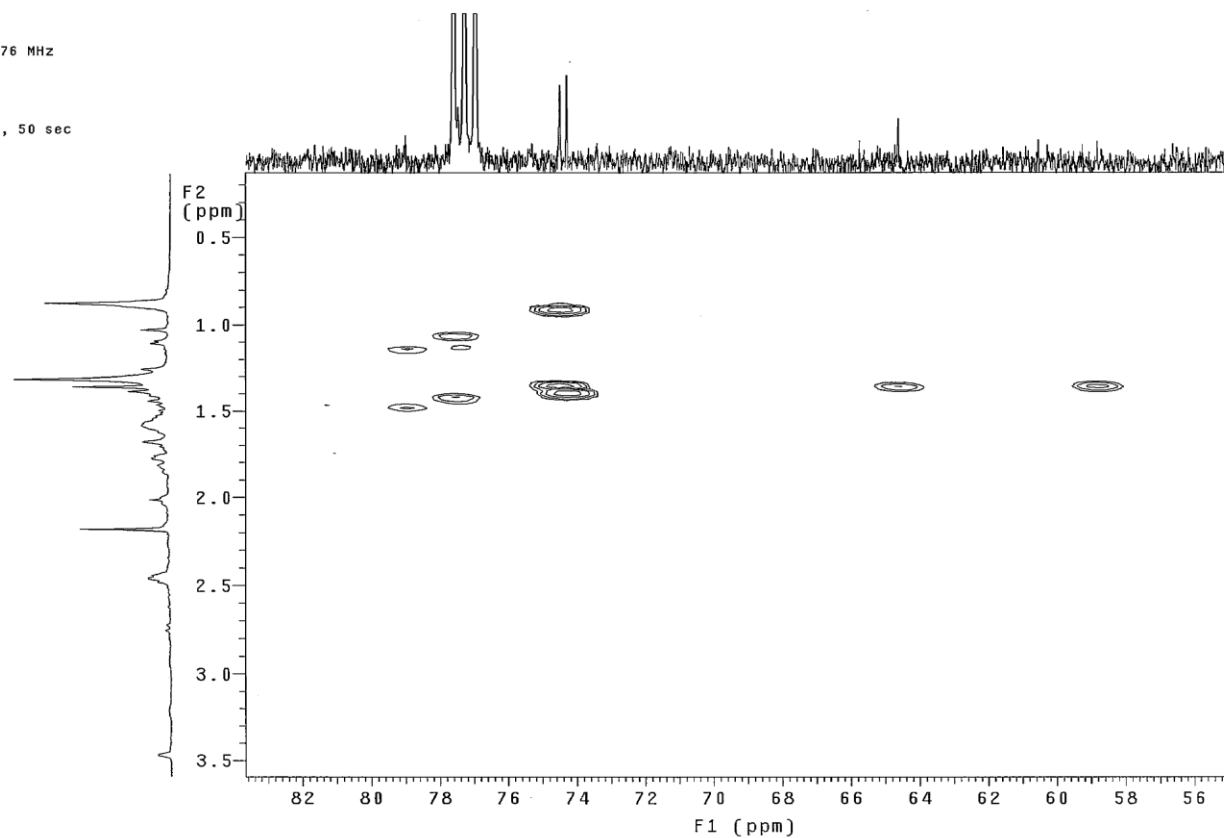
Partial expansions of the 2D-HMBC spectrum of compound 4

MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
24 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 29 min, 50 sec



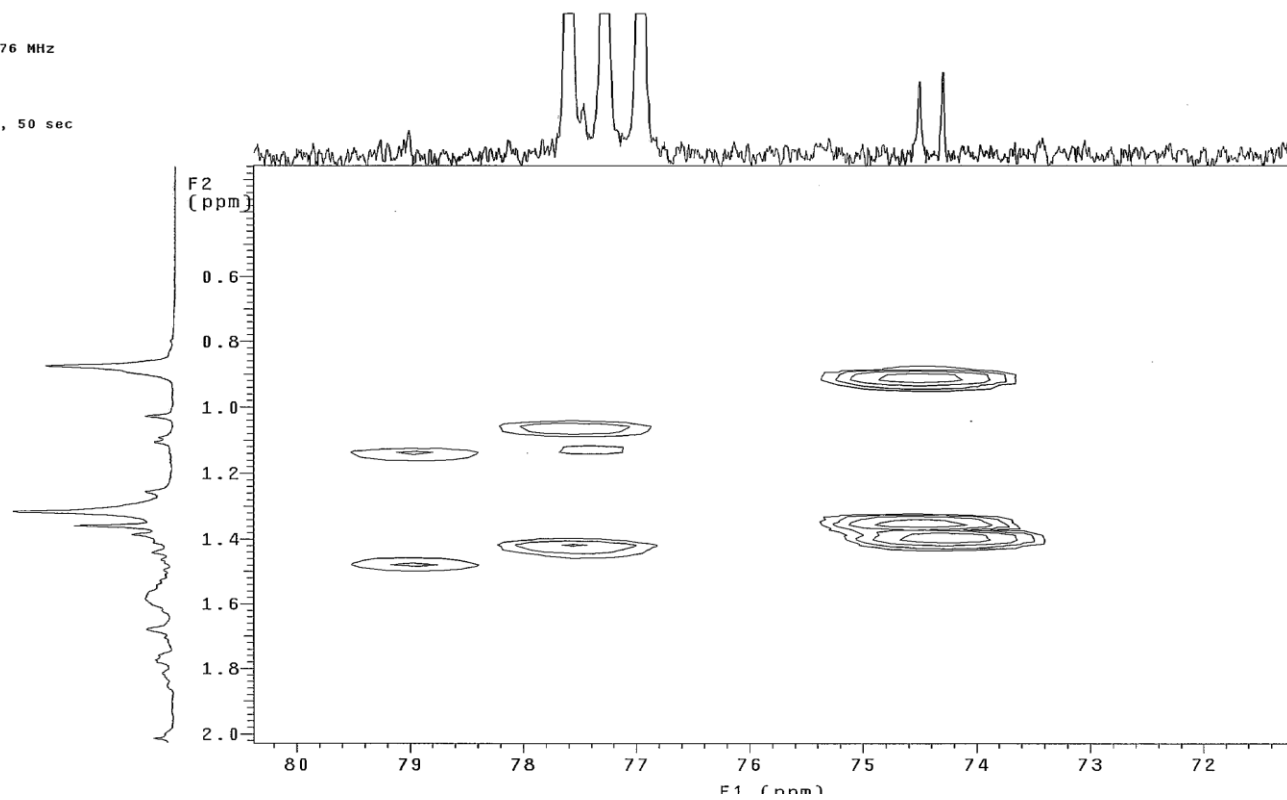
Partial expansions of the 2D-HMBC spectrum of compound 4

MRROSS-03-24-HPLC-10

Archive directory: /export/home/safwat/vnmrsws/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
24 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 3 hr, 29 min, 50 sec



Partial expansions of the 2D-HMBC spectrum of compound 4



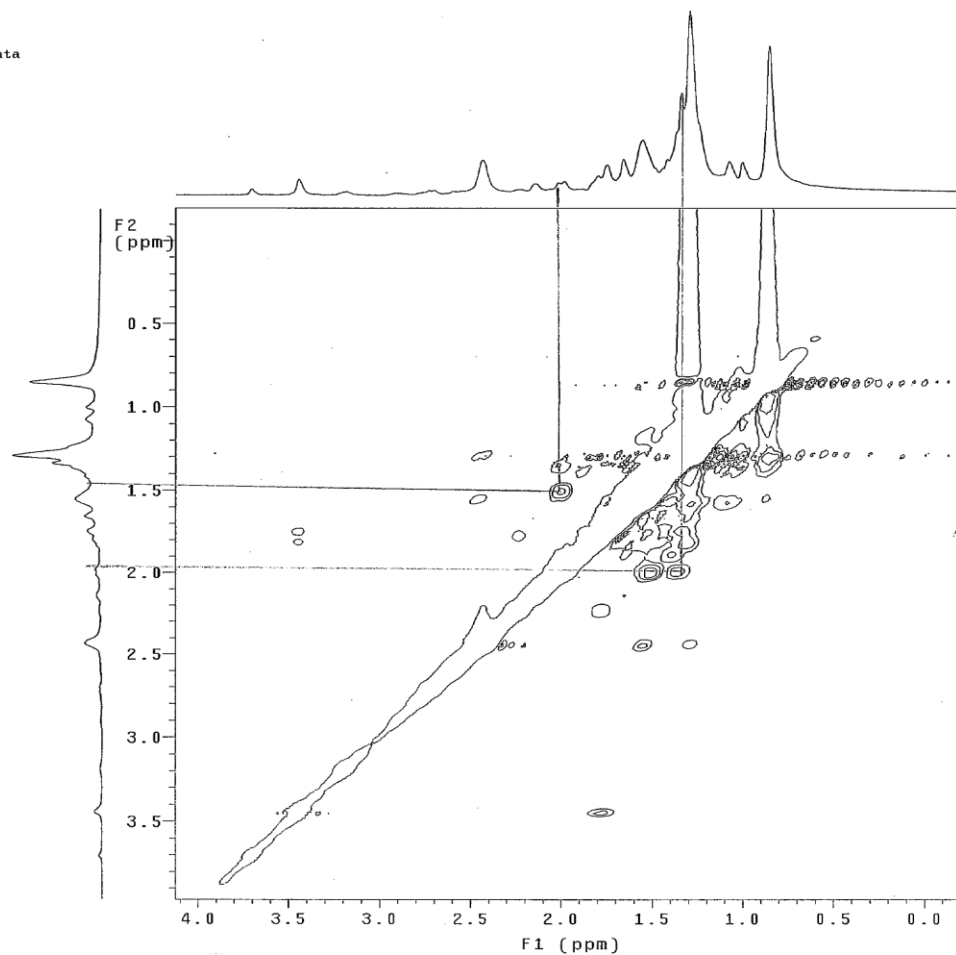


Mr0ss-24-10

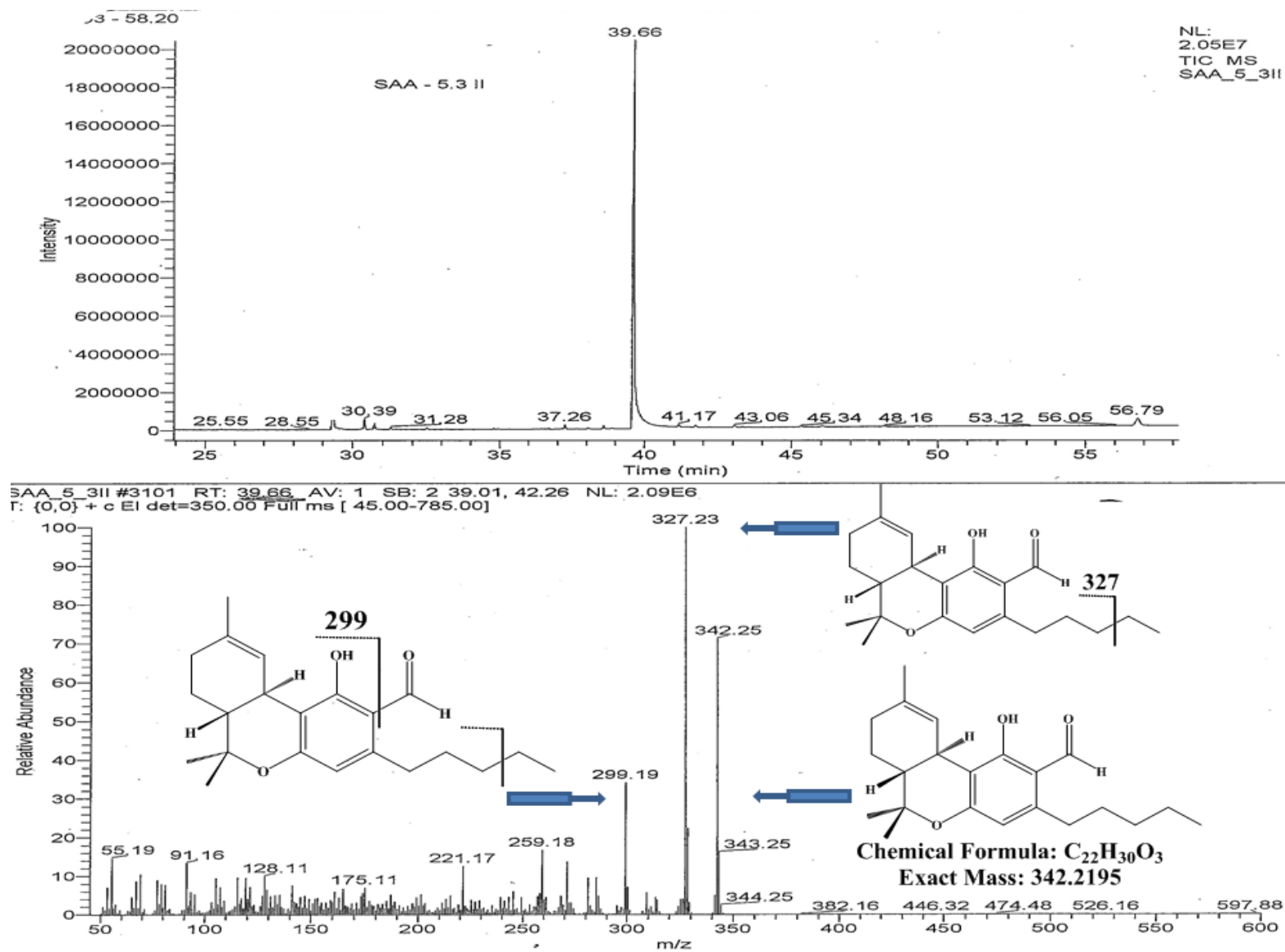
Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

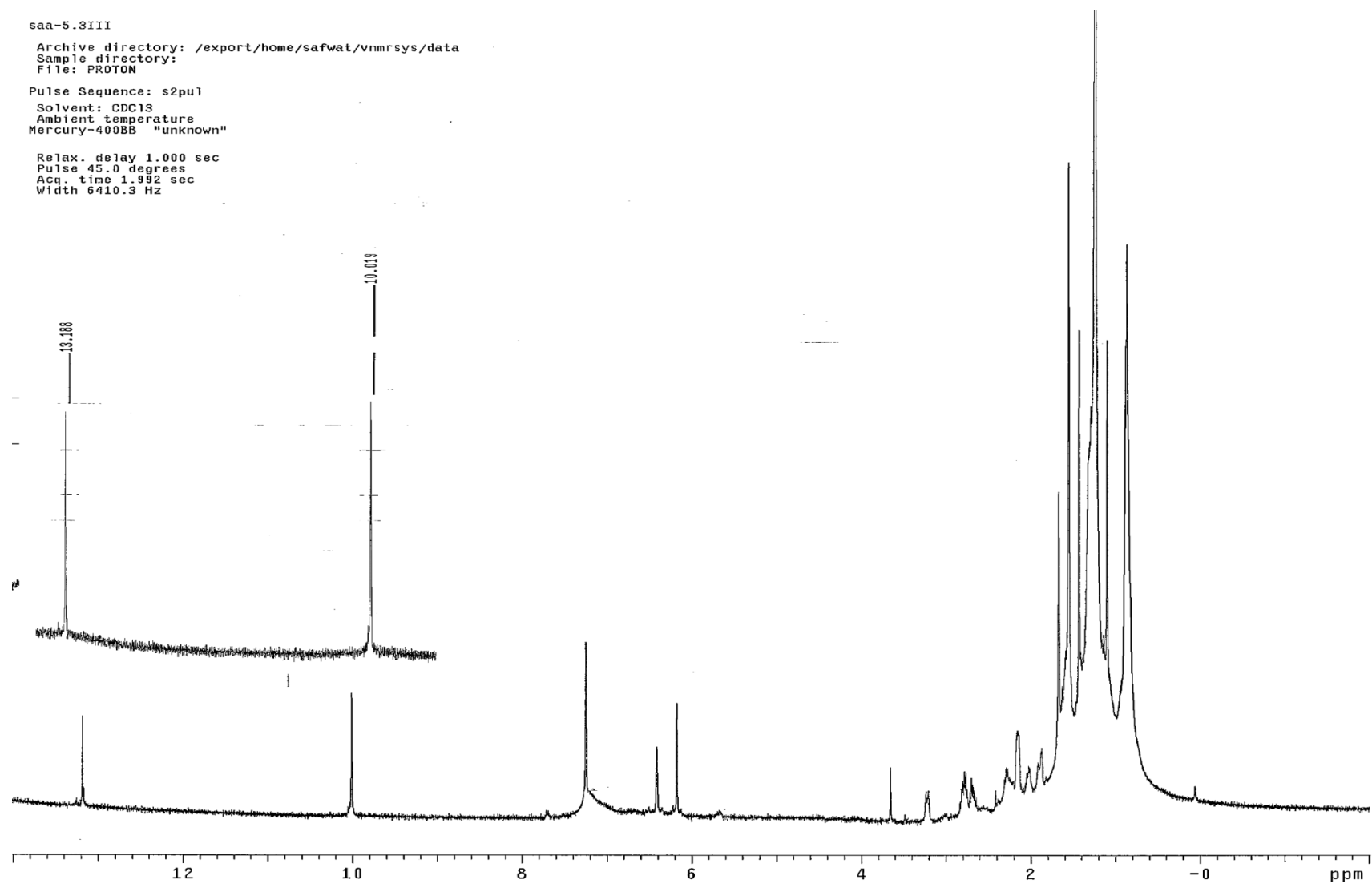
Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
26 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
FT size 2048 x 2048  
Total time 3 hr, 47 min, 41 sec



Partial expansions of the 2D-ROESY spectrum of compound 4



GCMS spectrum of compound 6



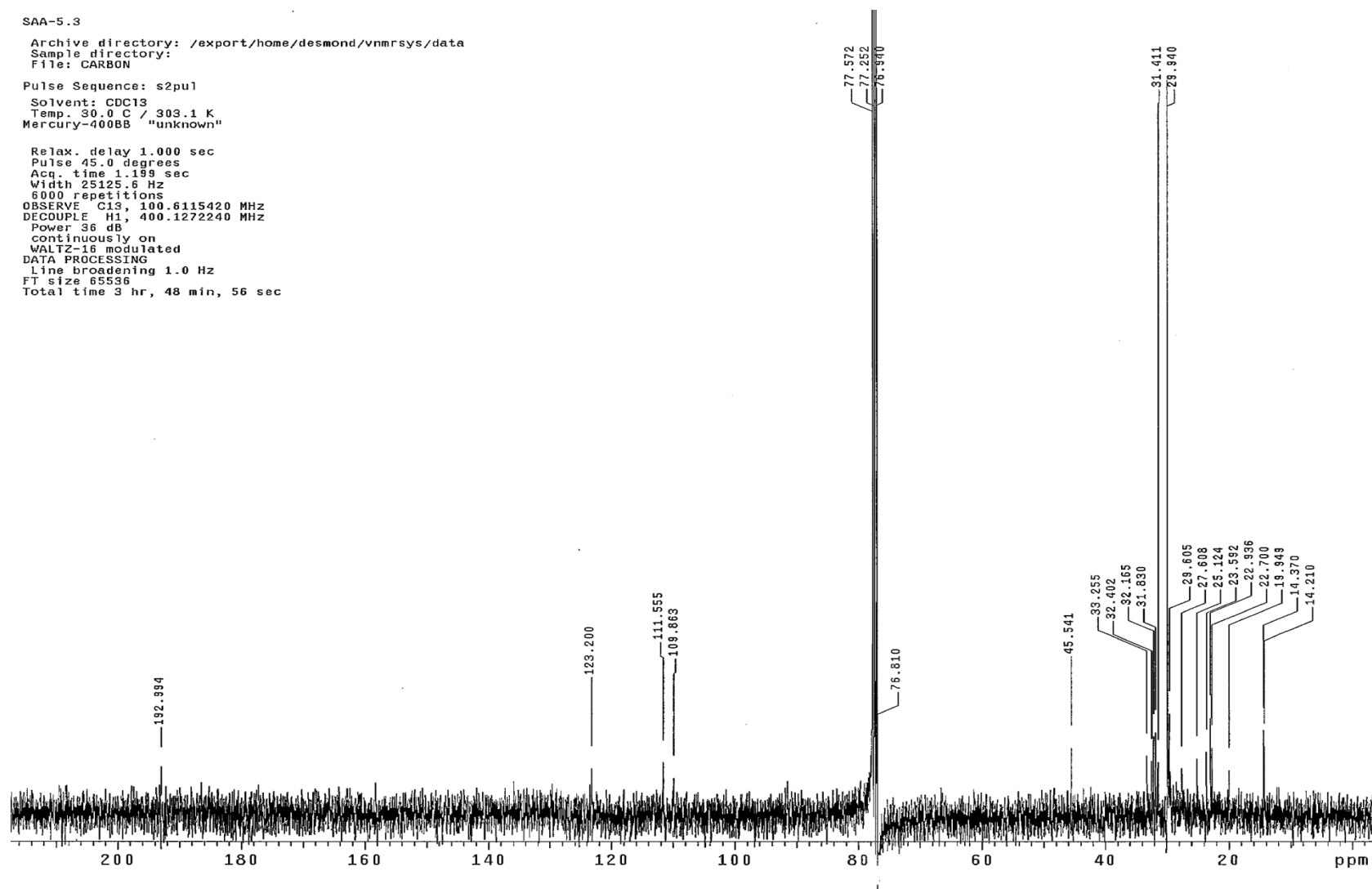
**$^1\text{H}$ -NMR spectrum of compound 5 in ( $\text{CDCl}_3$ , 400 MHz)**

SAA-5.3

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

Pulse Sequence: s2pu1  
Solvent: CDC13  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
8000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 3 hr, 48 min, 56 sec



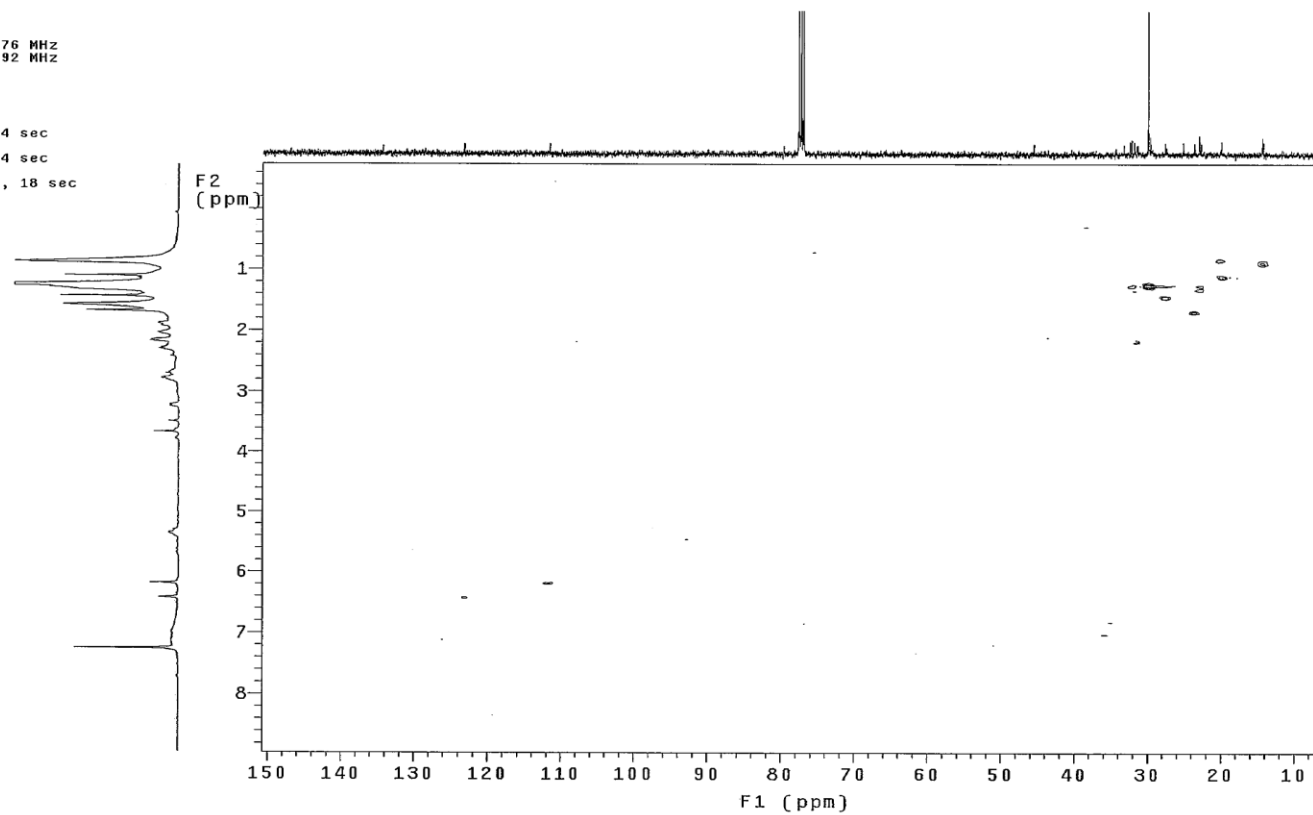
$^{13}\text{C}$ -NMR spectrum of compound 5 in ( $\text{CDCl}_3$ , 100 MHz)

SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



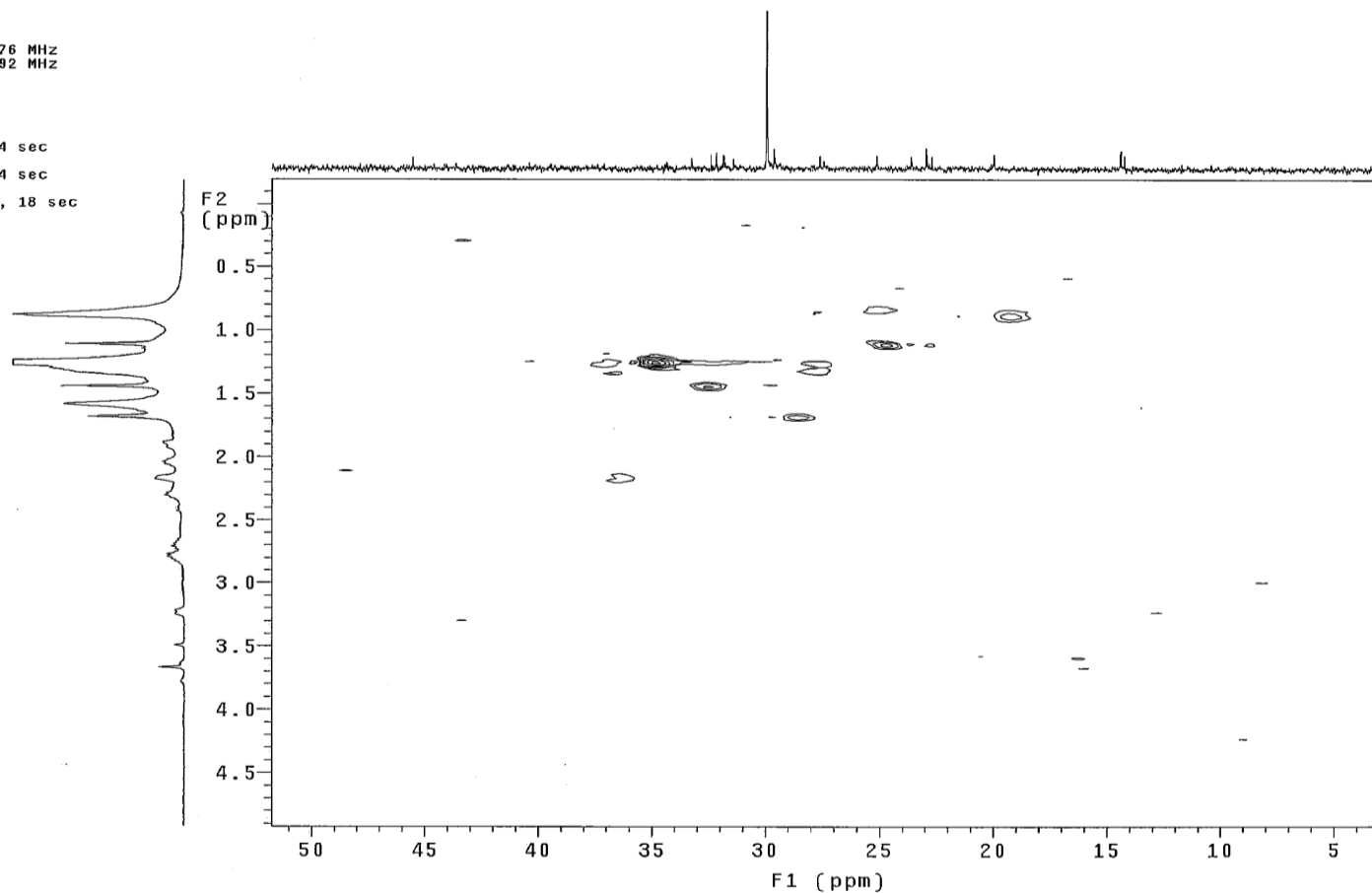
2D-HMPC spectrum of compound 5

SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

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

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



Partial expansions of the 2D-HMQC spectrum of compound 5

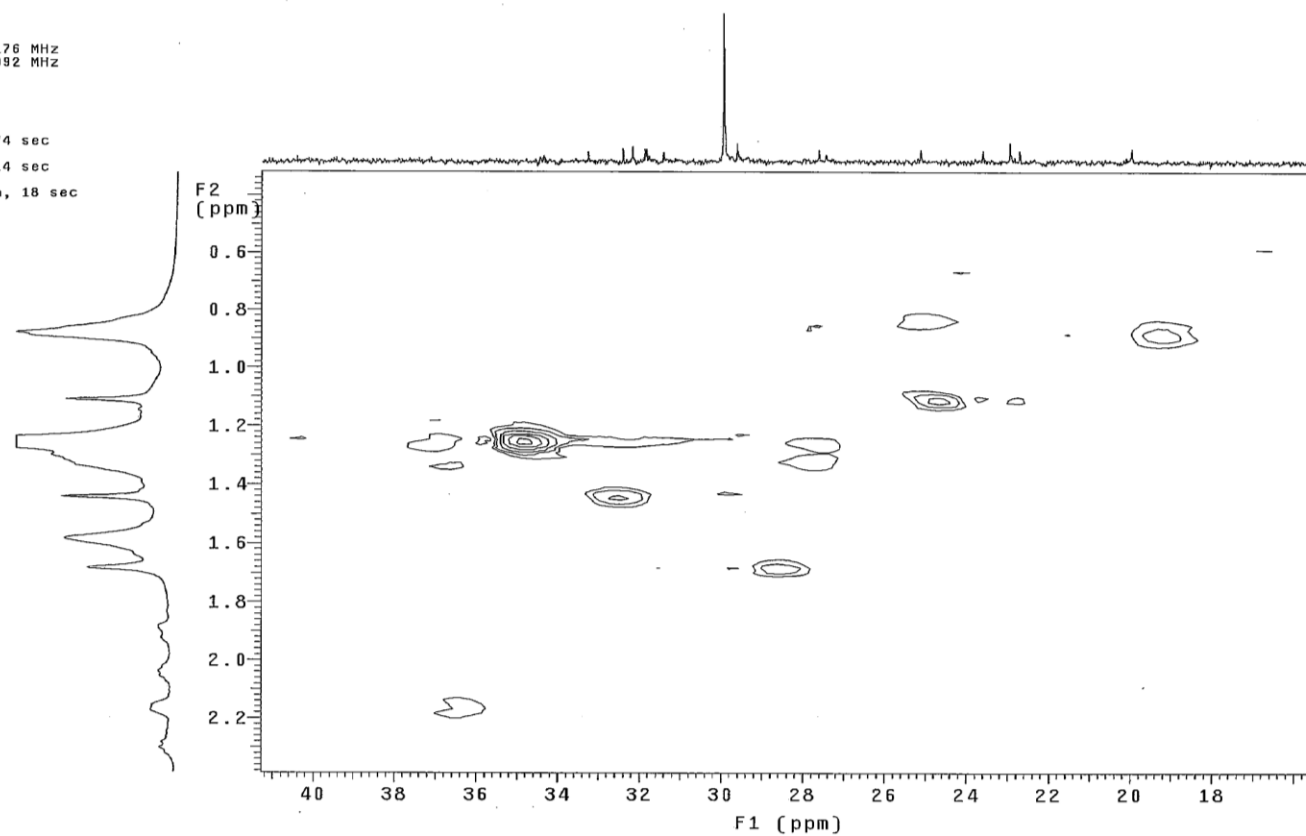
SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
On during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



Partial expansions of the 2D-HMQC spectrum of compound 5

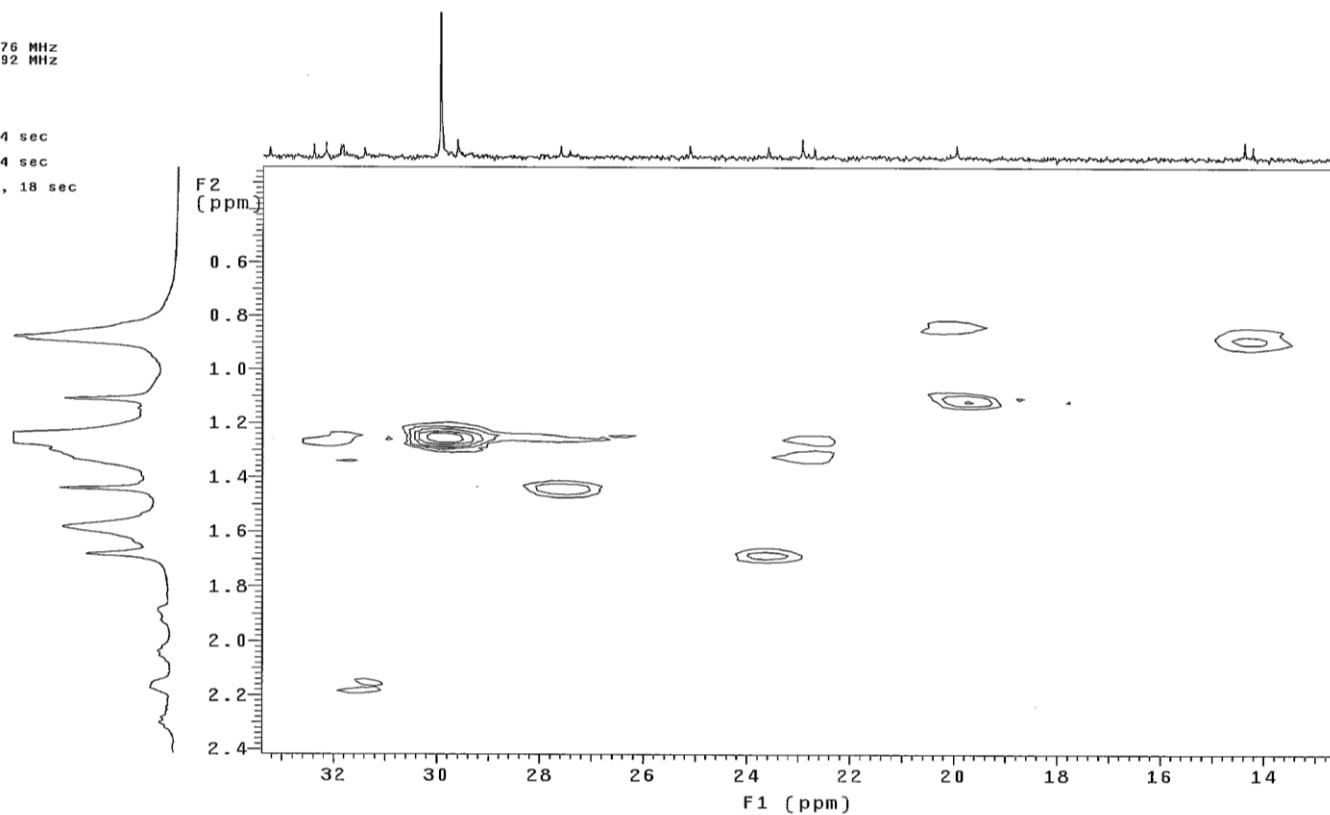


SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDCl3  
Temp: 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



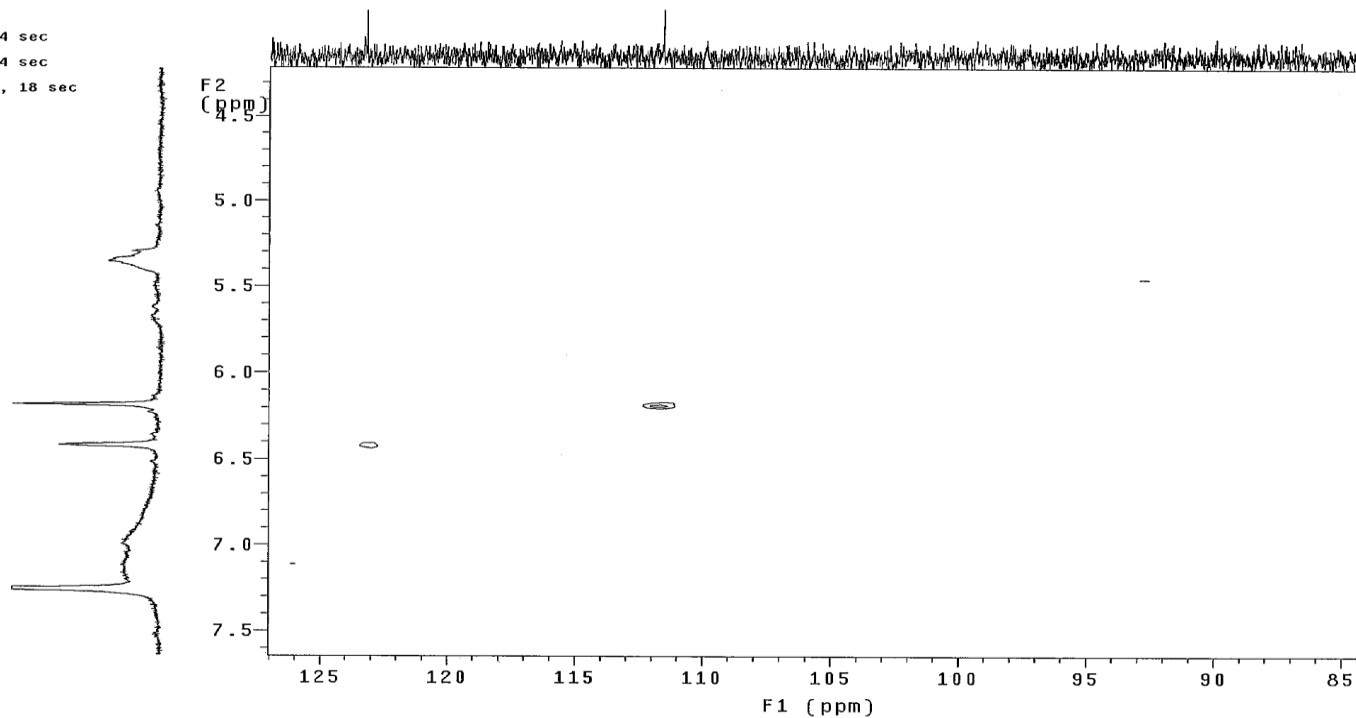
Partial expansions of the 2D-HMOC spectrum of compound 5

SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

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

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



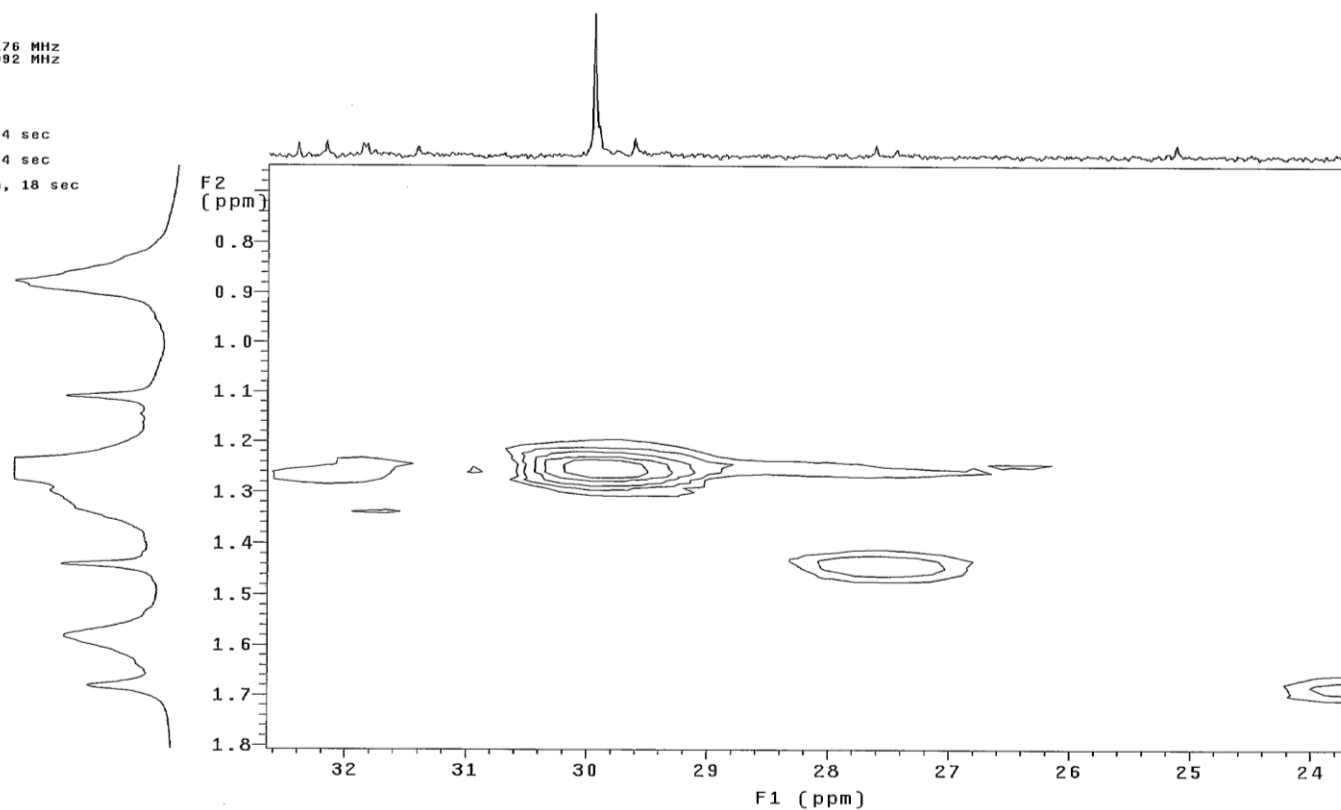
Partial expansions of the 2D-HMOC spectrum of compound 5

SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDCl3  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



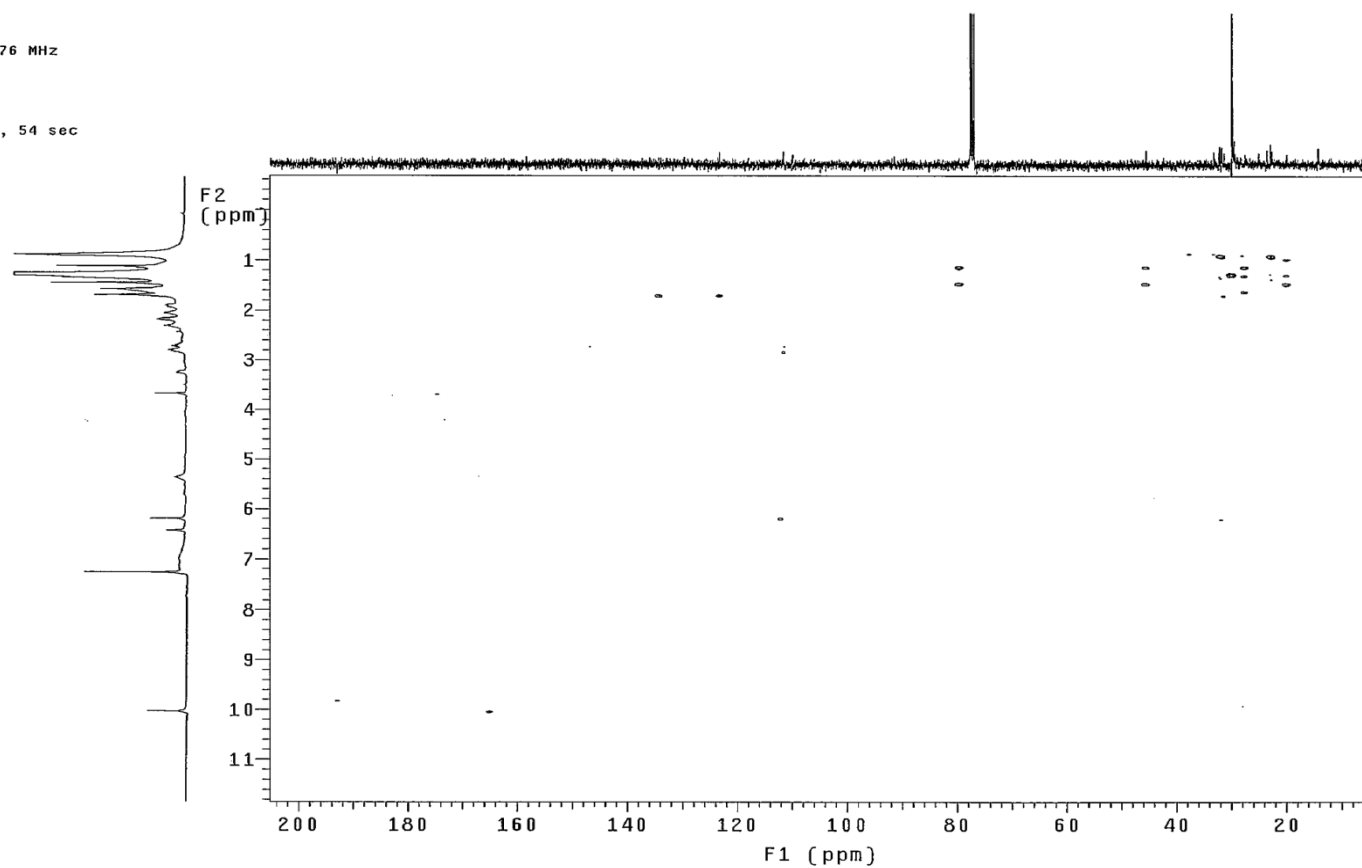
Partial expansions of the 2D-HMQC spectrum of compound 5

SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



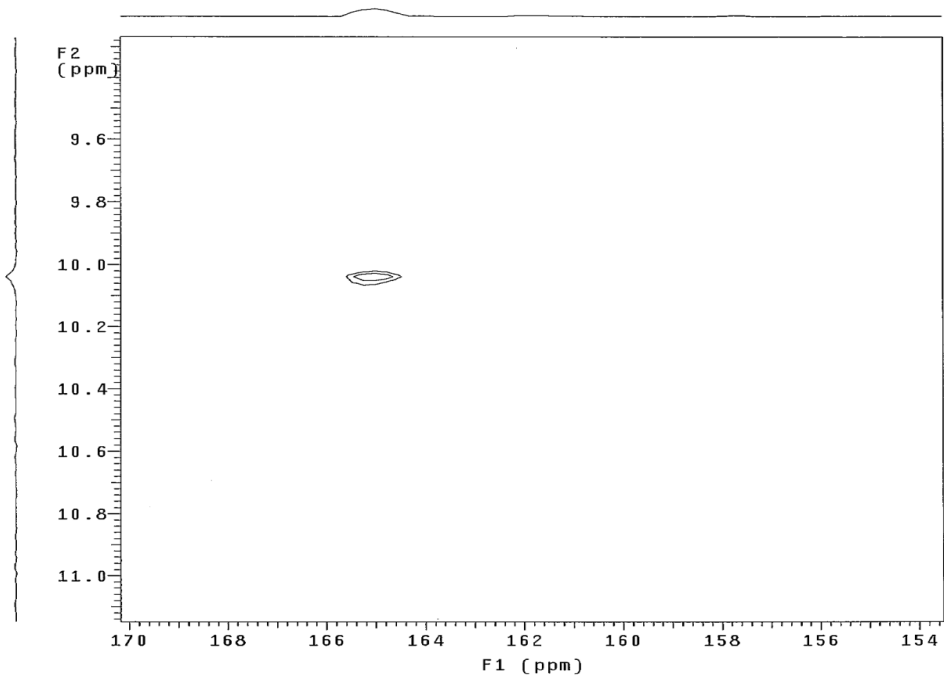
SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC

Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



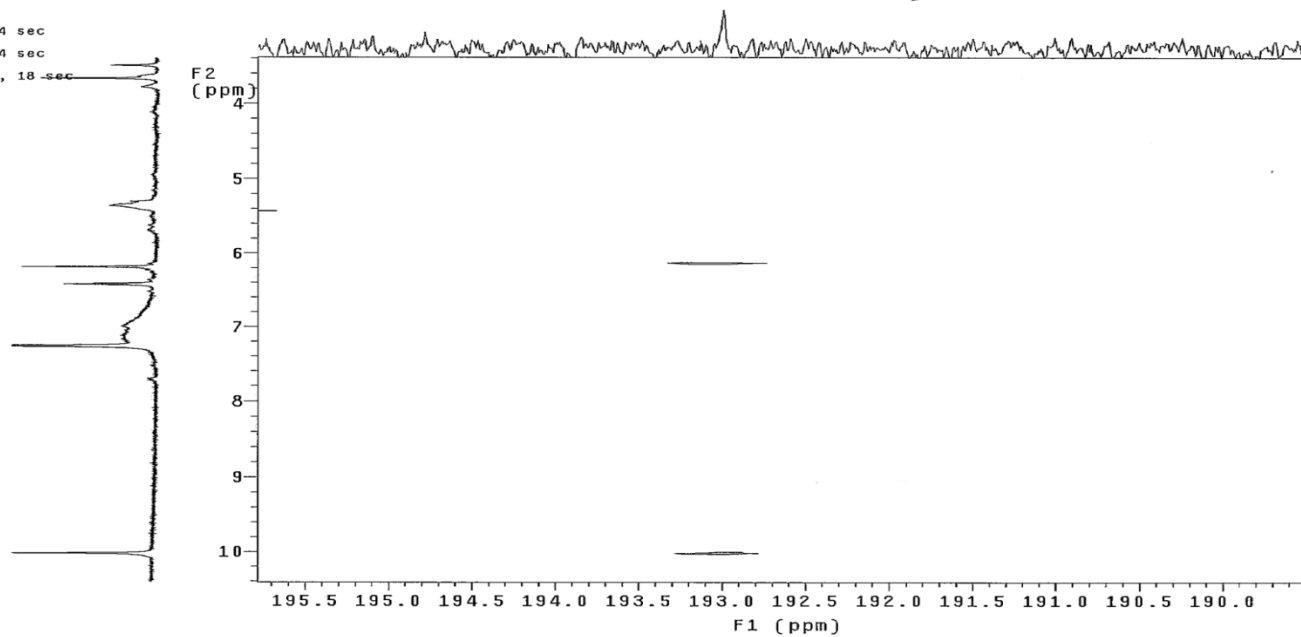
Partial expansions of the 2D-HMBC spectrum of compound 5

SAA-5.3III

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 25125.6 Hz  
40 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6226092 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 3 hr, 36 min, 18 sec



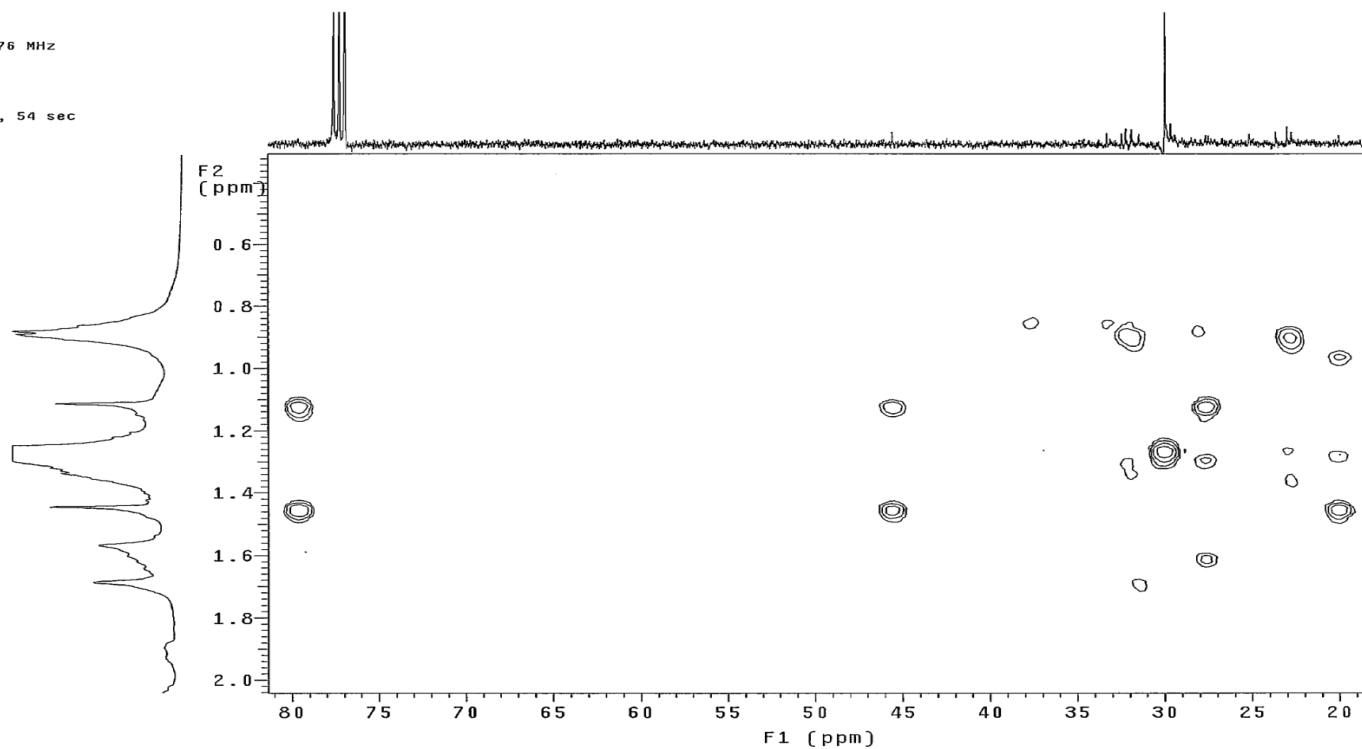
Partial expansions of the 2D-HMBC spectrum of compound 5

SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

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

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



Partial expansions of the 2D-HMBC spectrum of compound 5

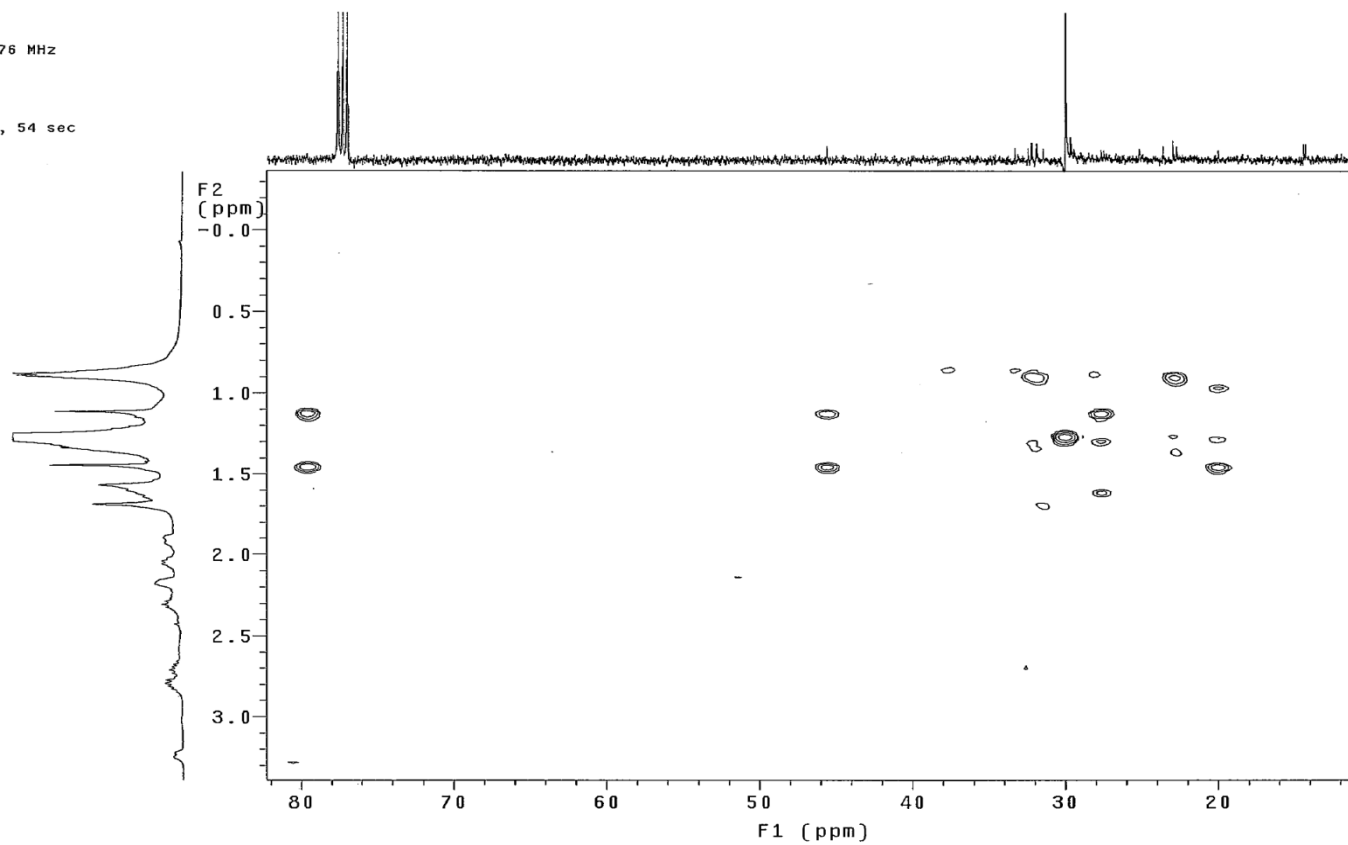
SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC

Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



Partial expansions of the 2D-HMBC spectrum of compound 5



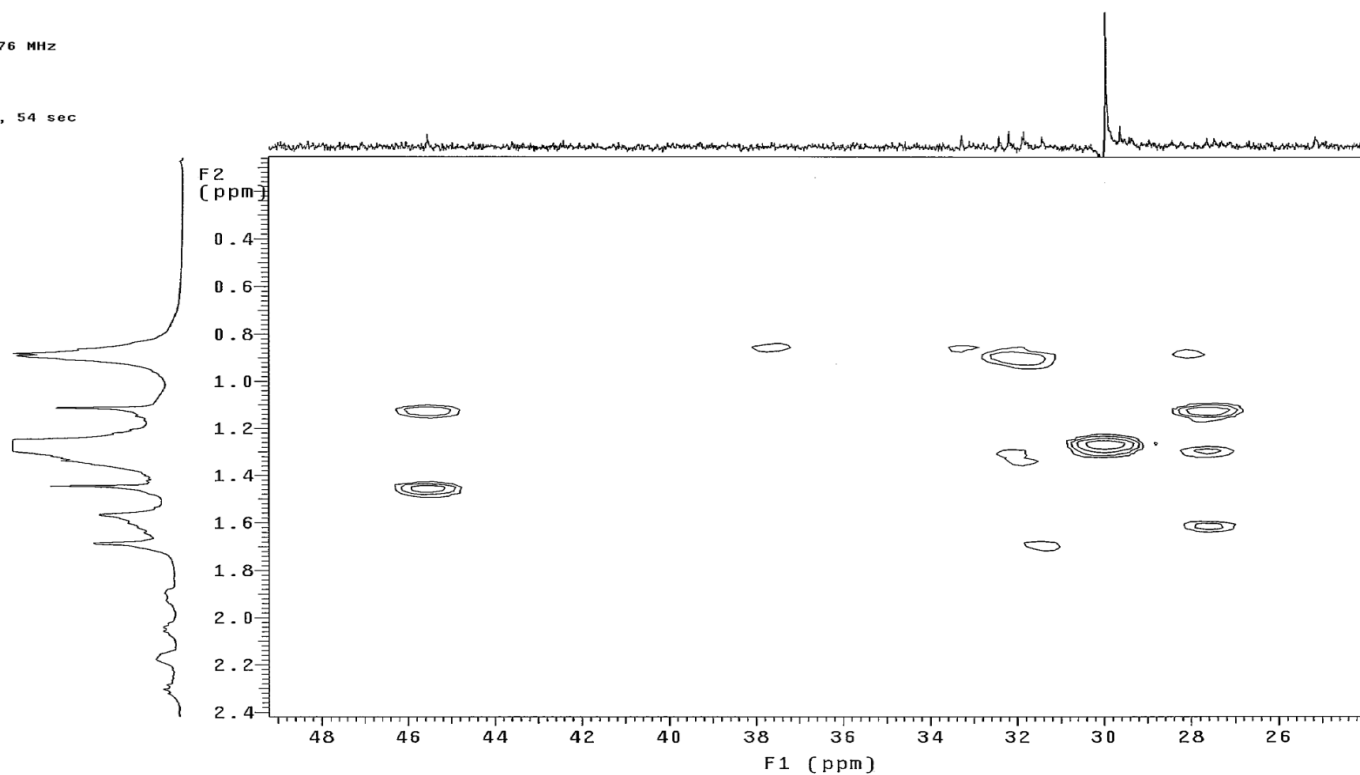
SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC

Solvent: CDCl<sub>3</sub>  
Temp. 30.0 C / 303.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec



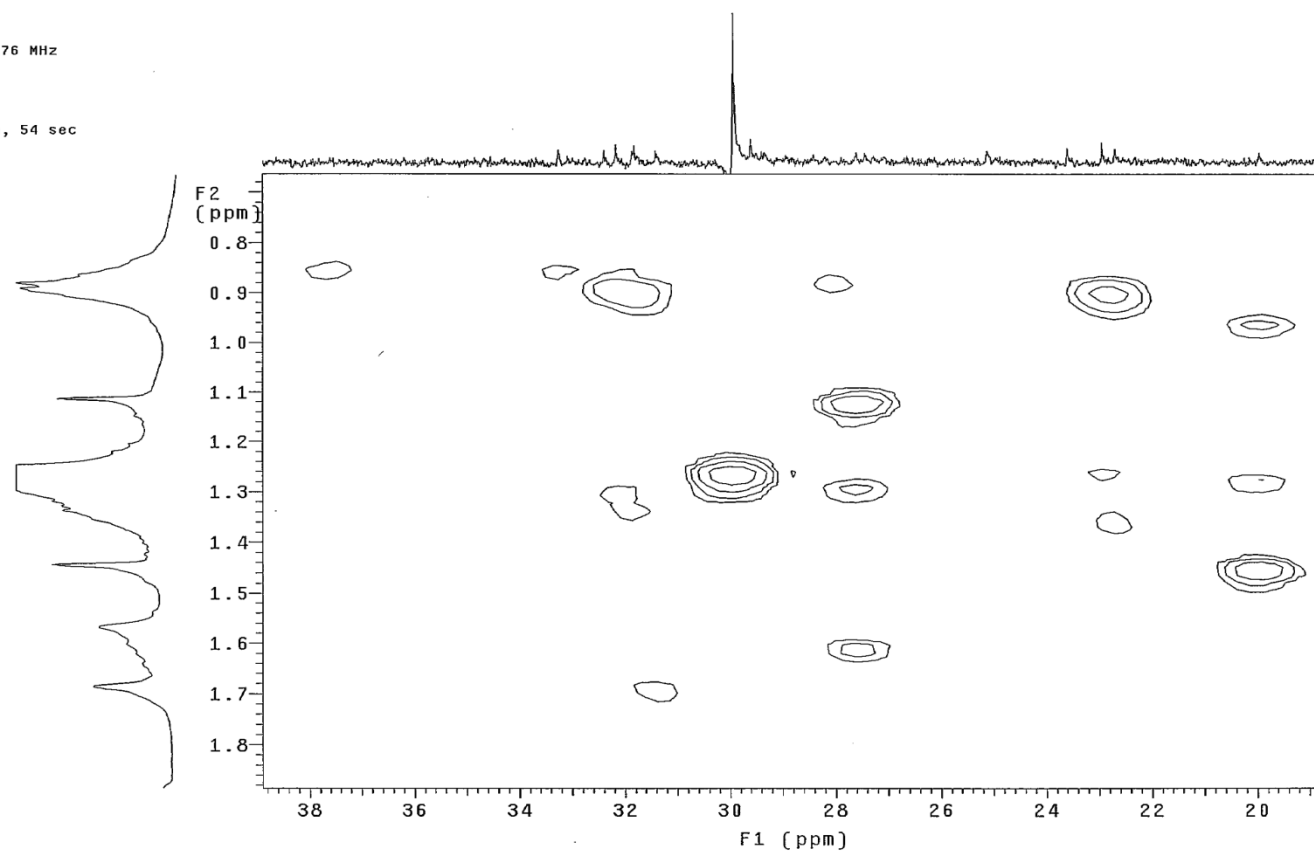
Partial expansions of the 2D-HMBC spectrum of compound 5

SAA-5.3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

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

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
32 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 4 hr, 37 min, 54 sec

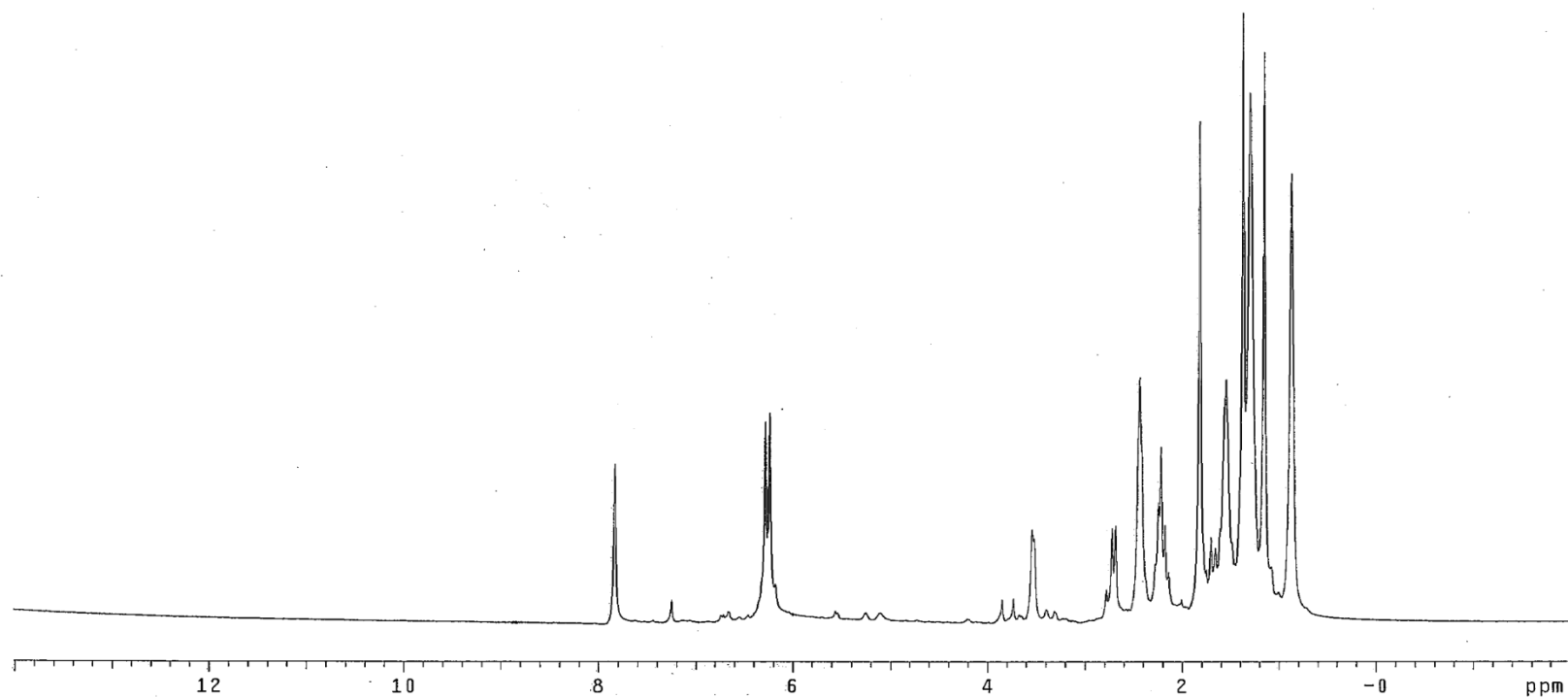


Partial expansions of the 2D-HMBC spectrum of compound 5

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
32 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
F1 size 32768  
Total time 1 min, 40 sec



**Fig (18)  $^1\text{H}$ -NMR spectrum of compound 6 in ( $\text{CDCl}_3$ , 400 MHz)**

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

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
320 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 1 hr, 16 min, 21 sec

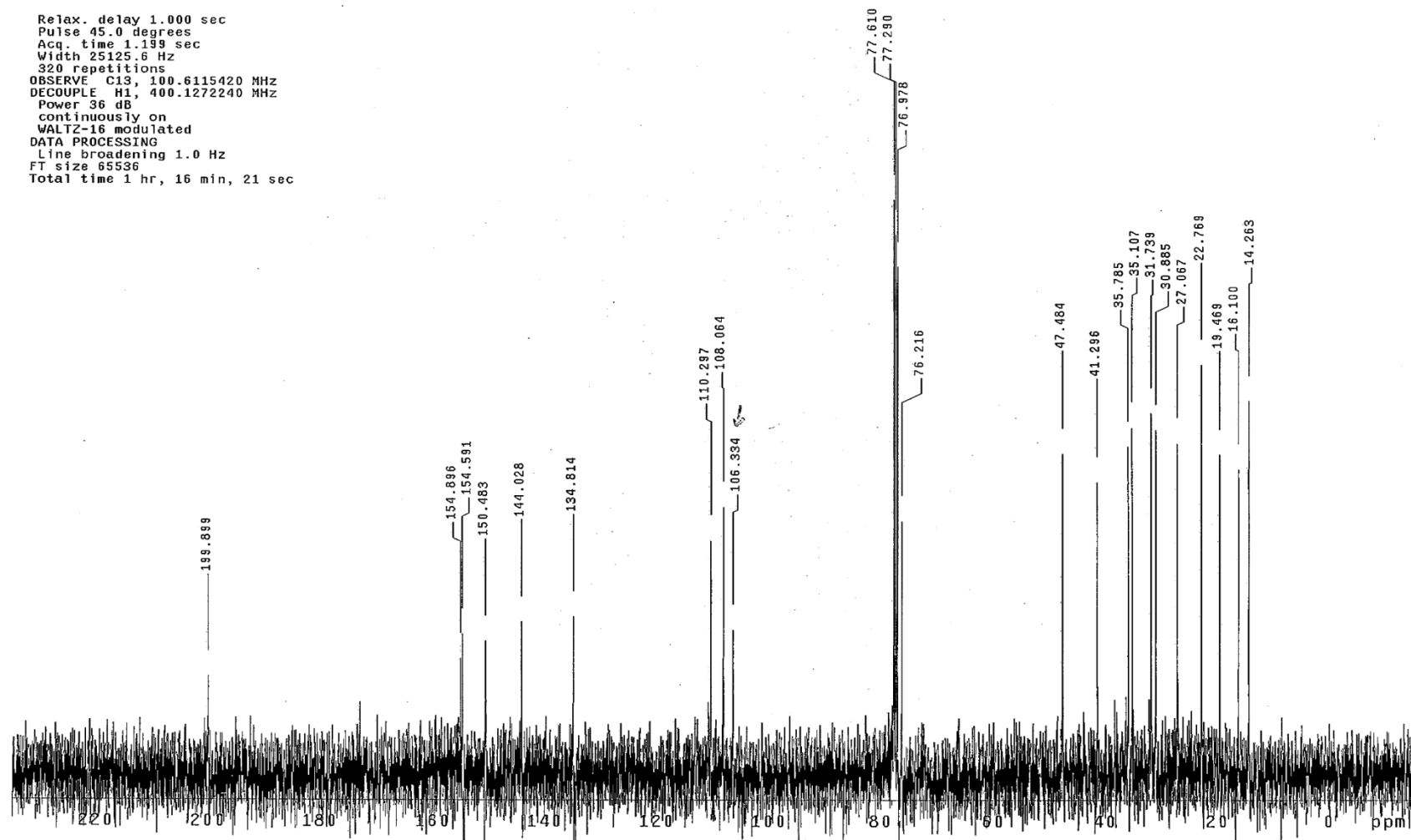


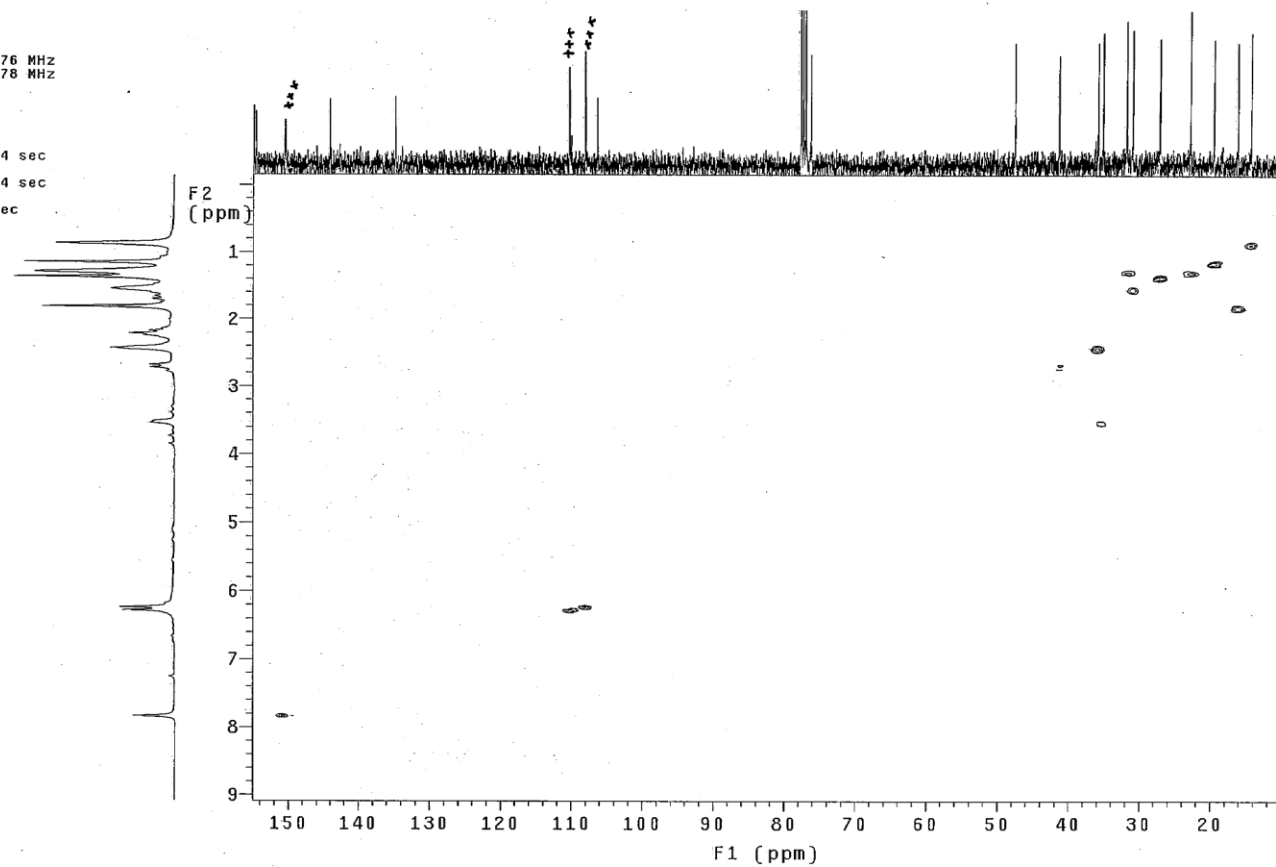
Fig (19)  $^{13}\text{C}$ -NMR spectrum of compound 6 in ( $\text{CDCl}_3$ , 100 MHz)

STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 23 min, 29 sec



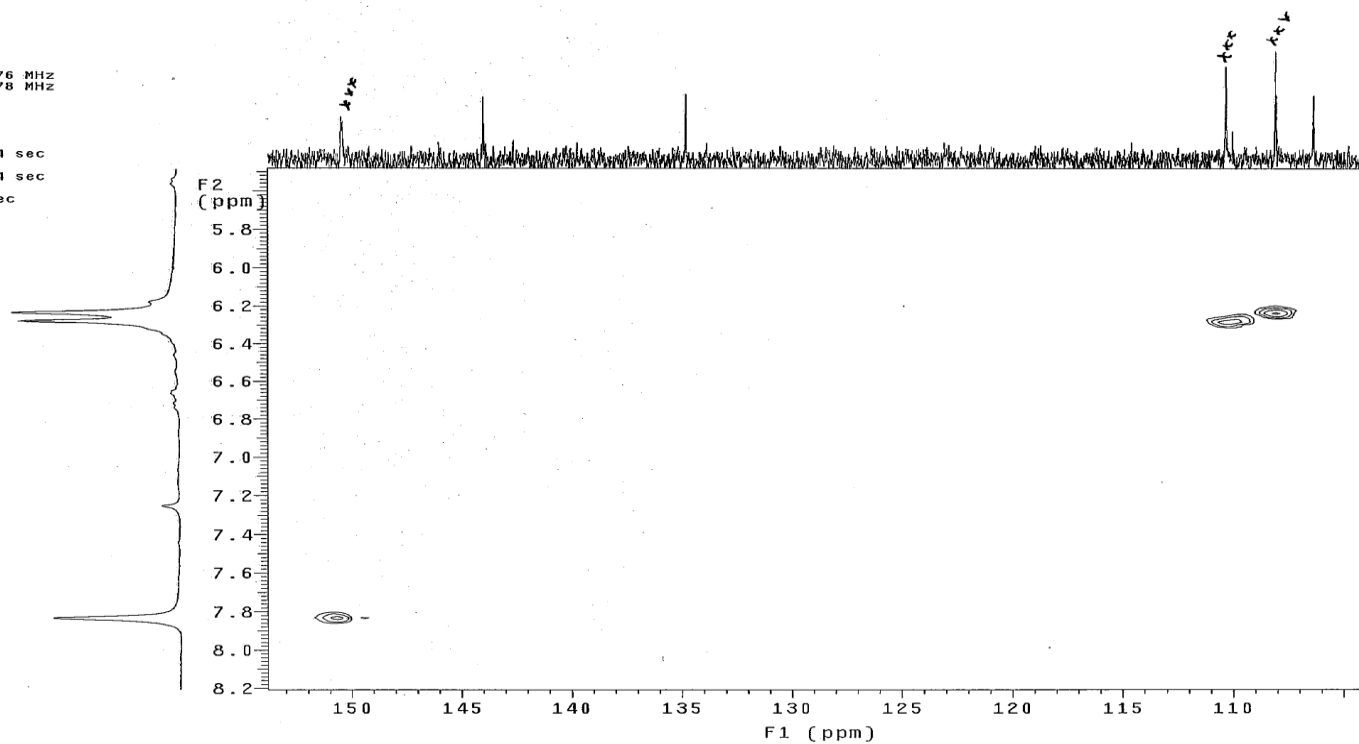
2D-HMPC spectrum of compound 6

## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
F1 size 2048 x 2048  
Total time 23 min, 29 sec



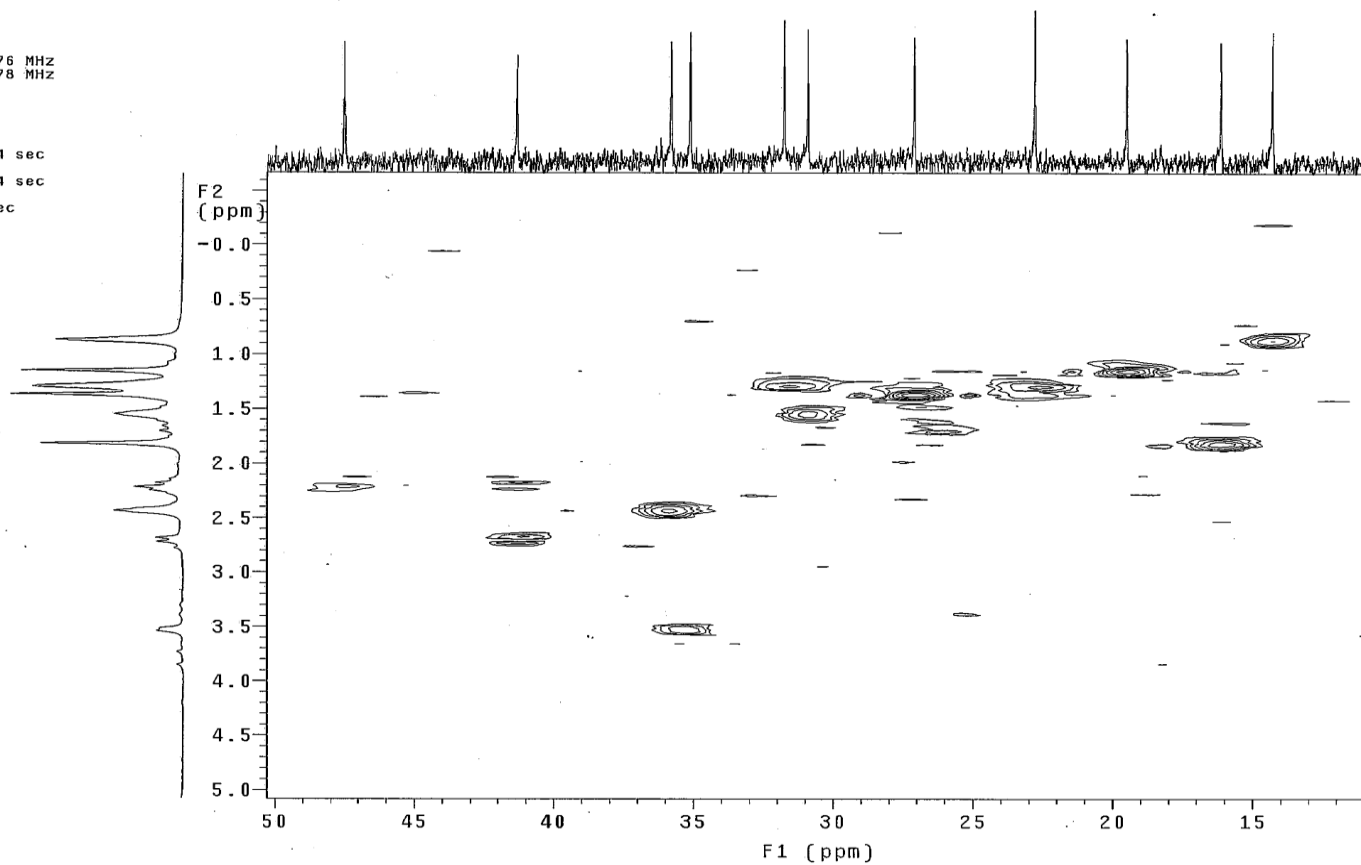
Partial expansions of the 2D-HMPC spectrum of compound 6

STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 23 min, 29 sec



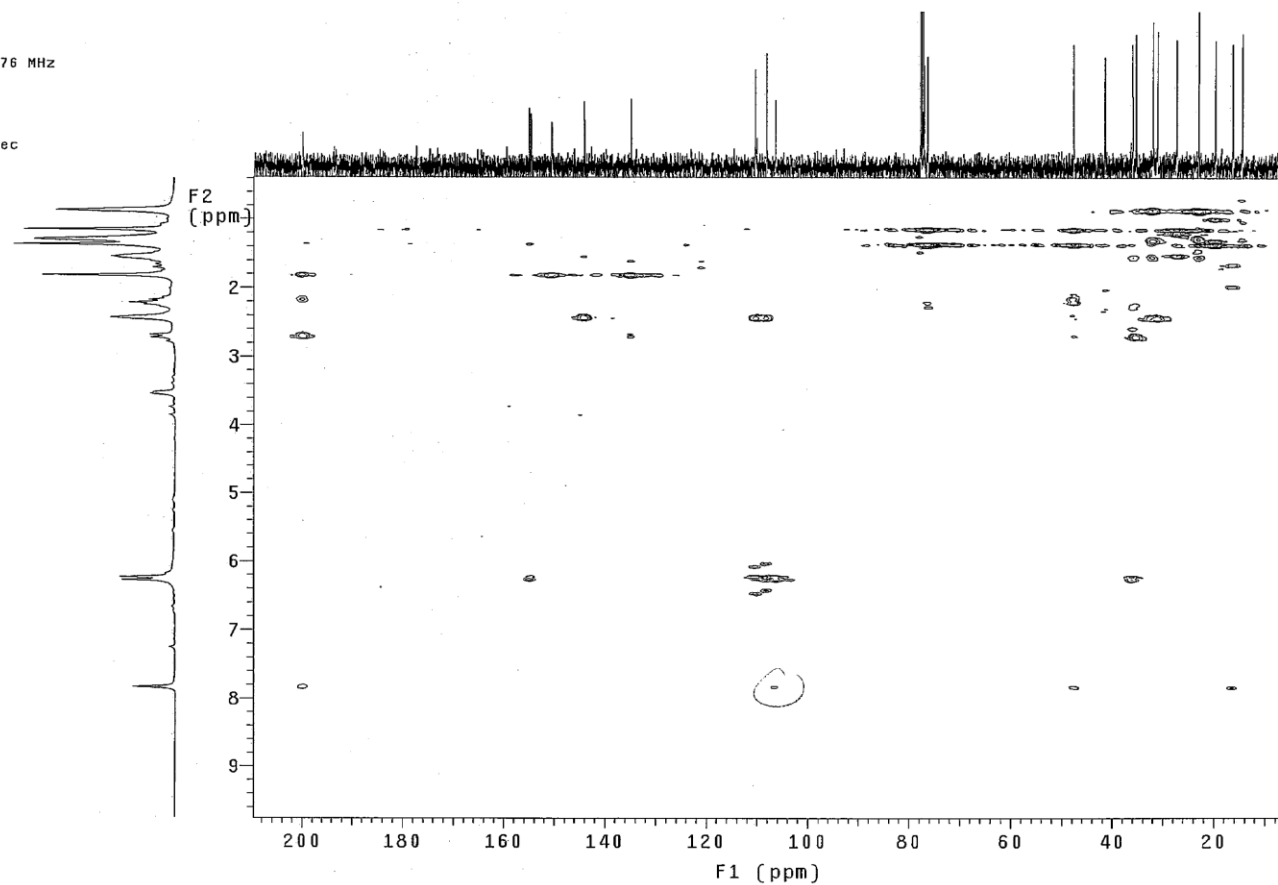
Partial expansions of the 2D-HMPC spectrum of compound 6

## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



2D-HMBC spectrum of compound 6

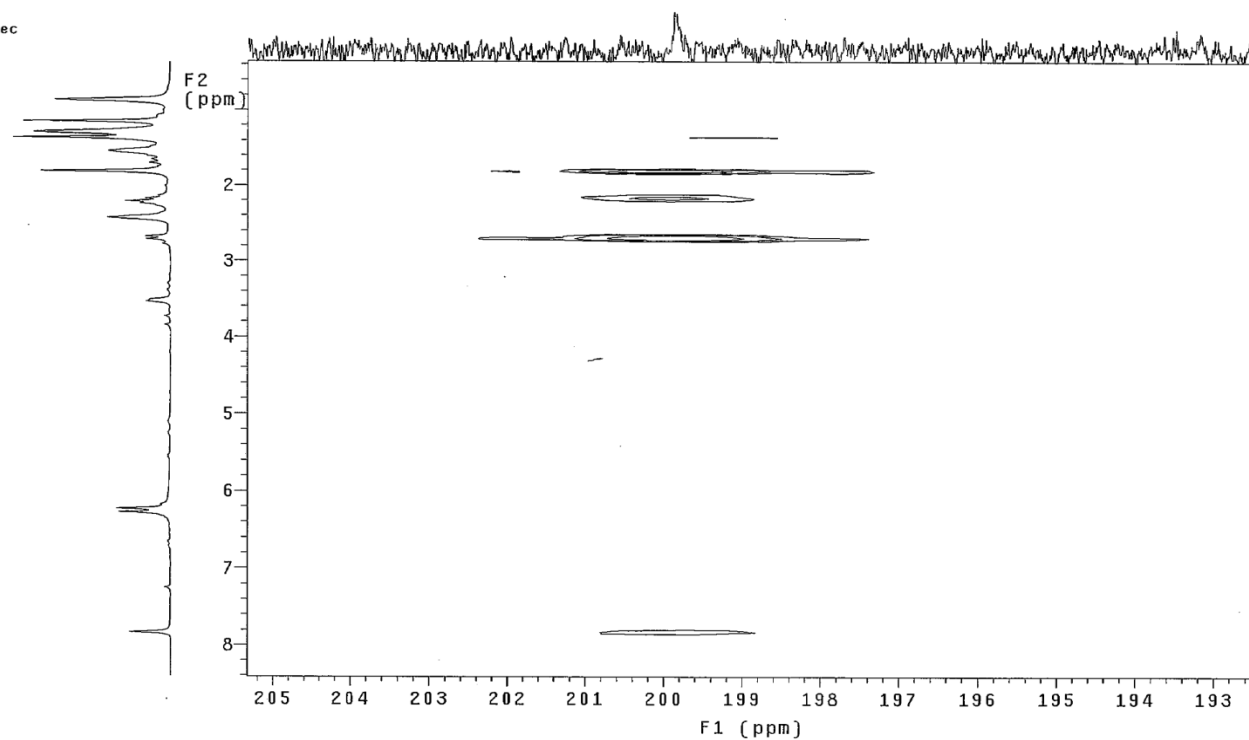


## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



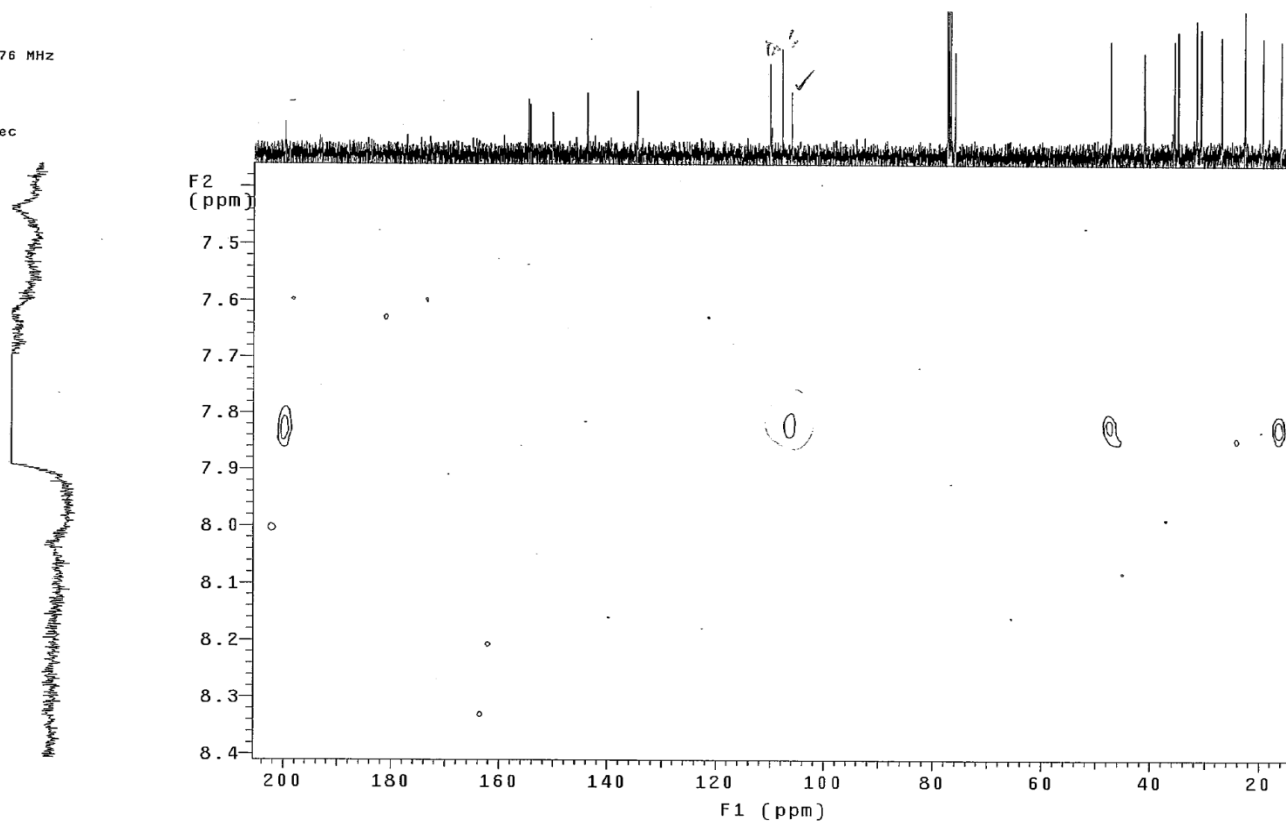
Partial expansions of the 2D-HMBC spectrum of compound 6

## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1: 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



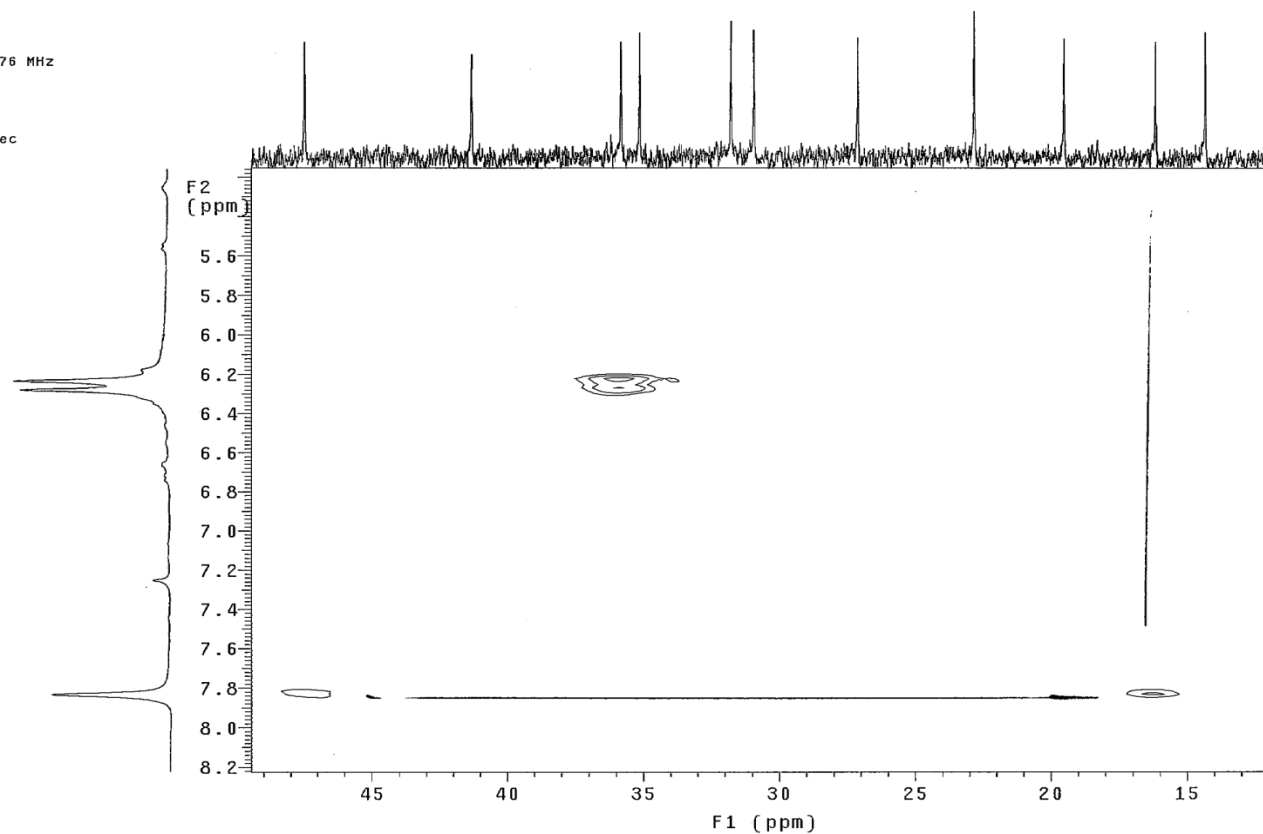
Partial expansions of the 2D-HMBC spectrum of compound 6

STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



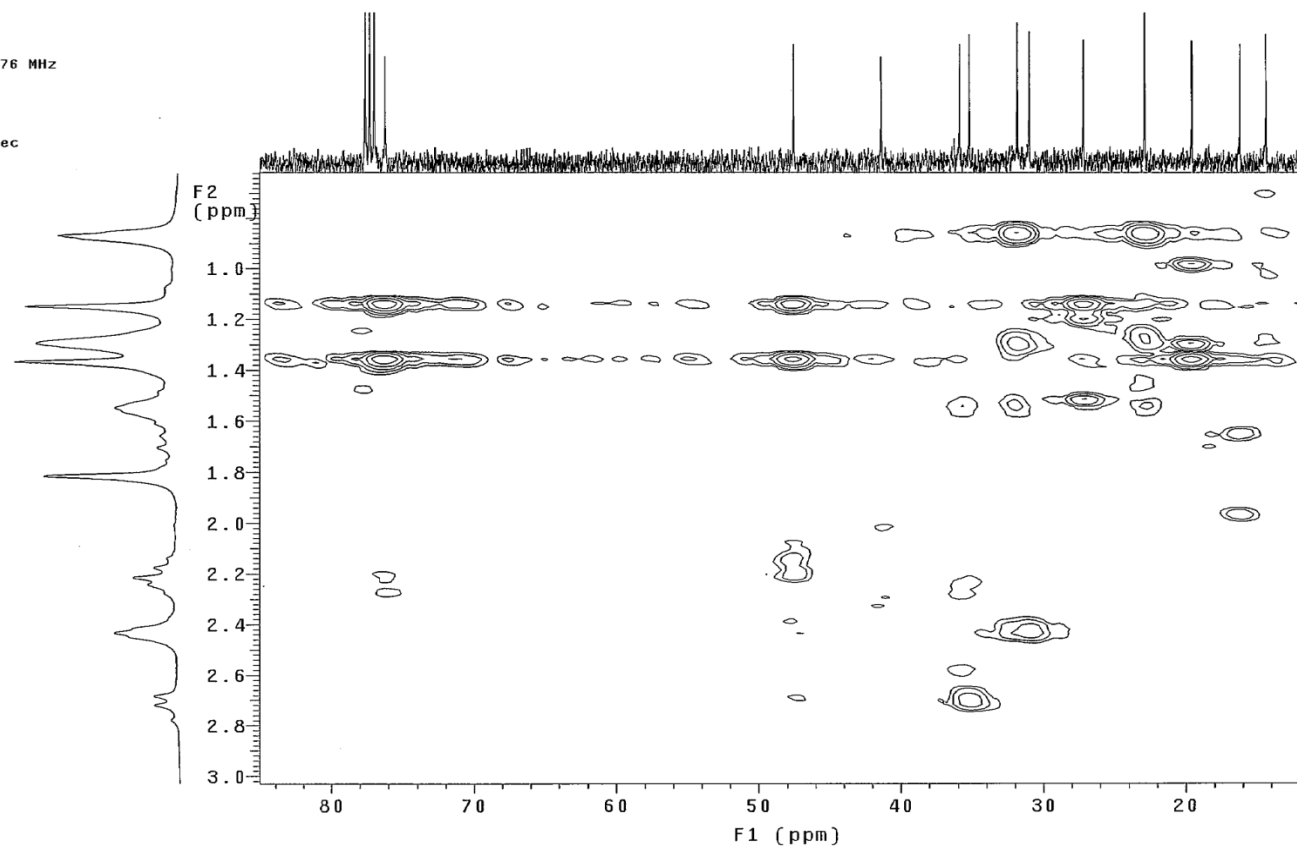
Partial expansions of the 2D-HMBC spectrum of compound 6

STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



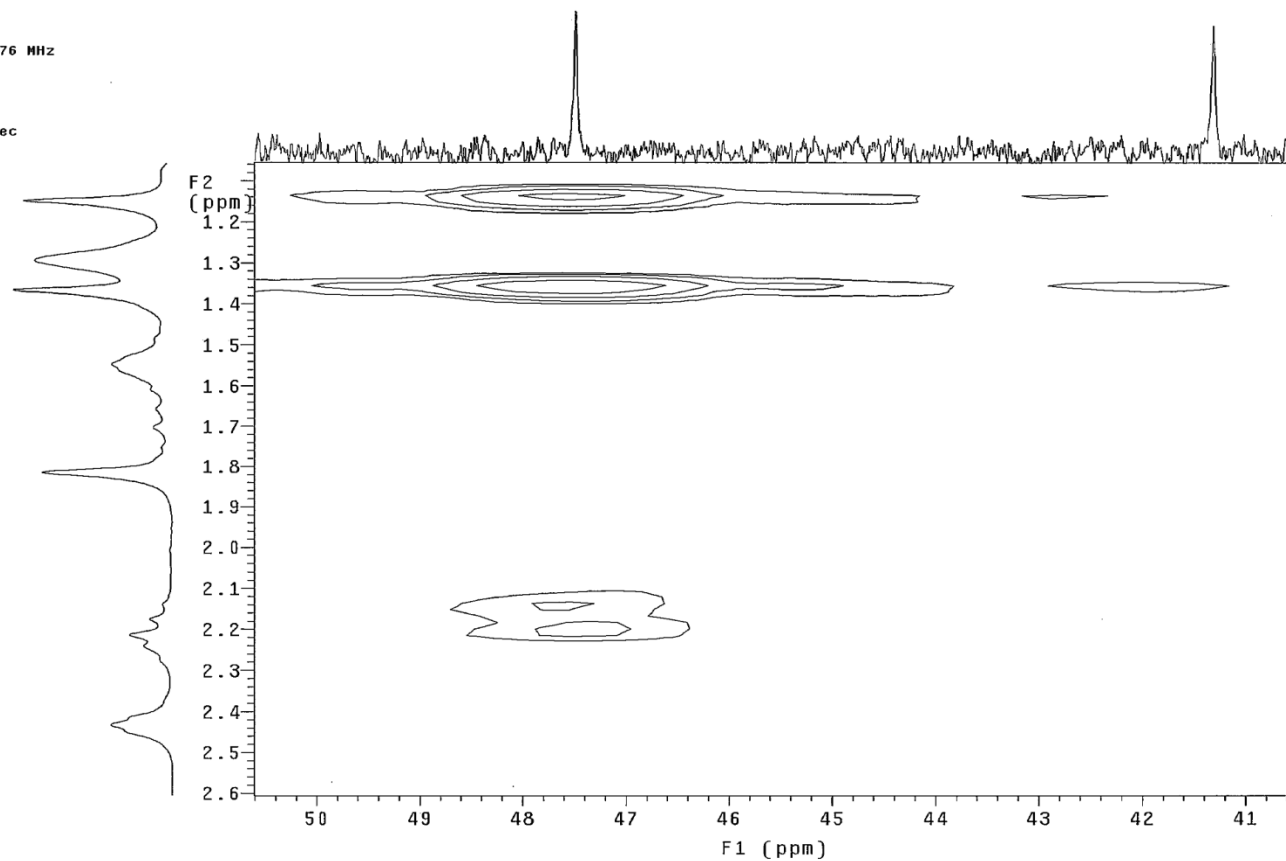
Partial expansions of the 2D-HMBC spectrum of compound 6

## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 37 min, 17 sec



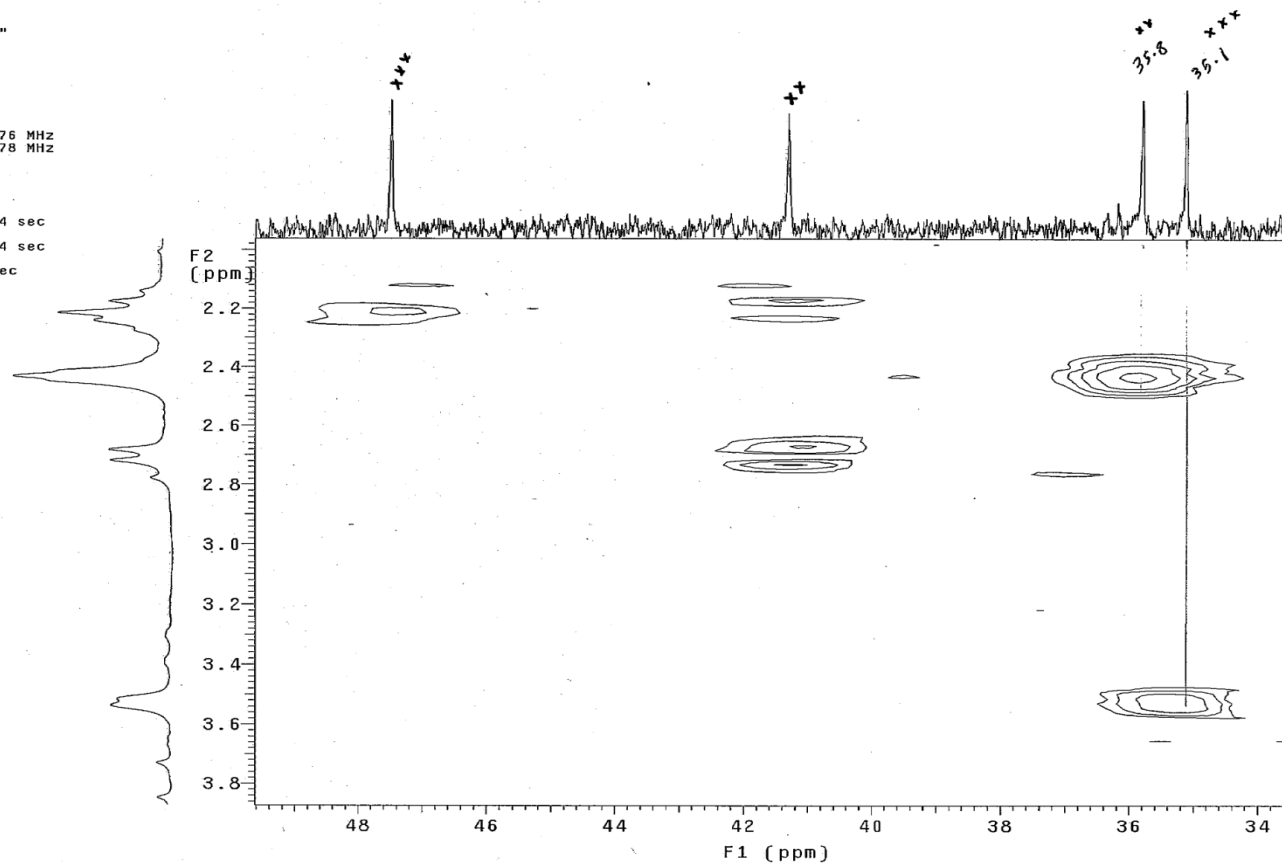
Partial expansions of the 2D-HMBC spectrum of compound 6

## STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 23 min, 29 sec



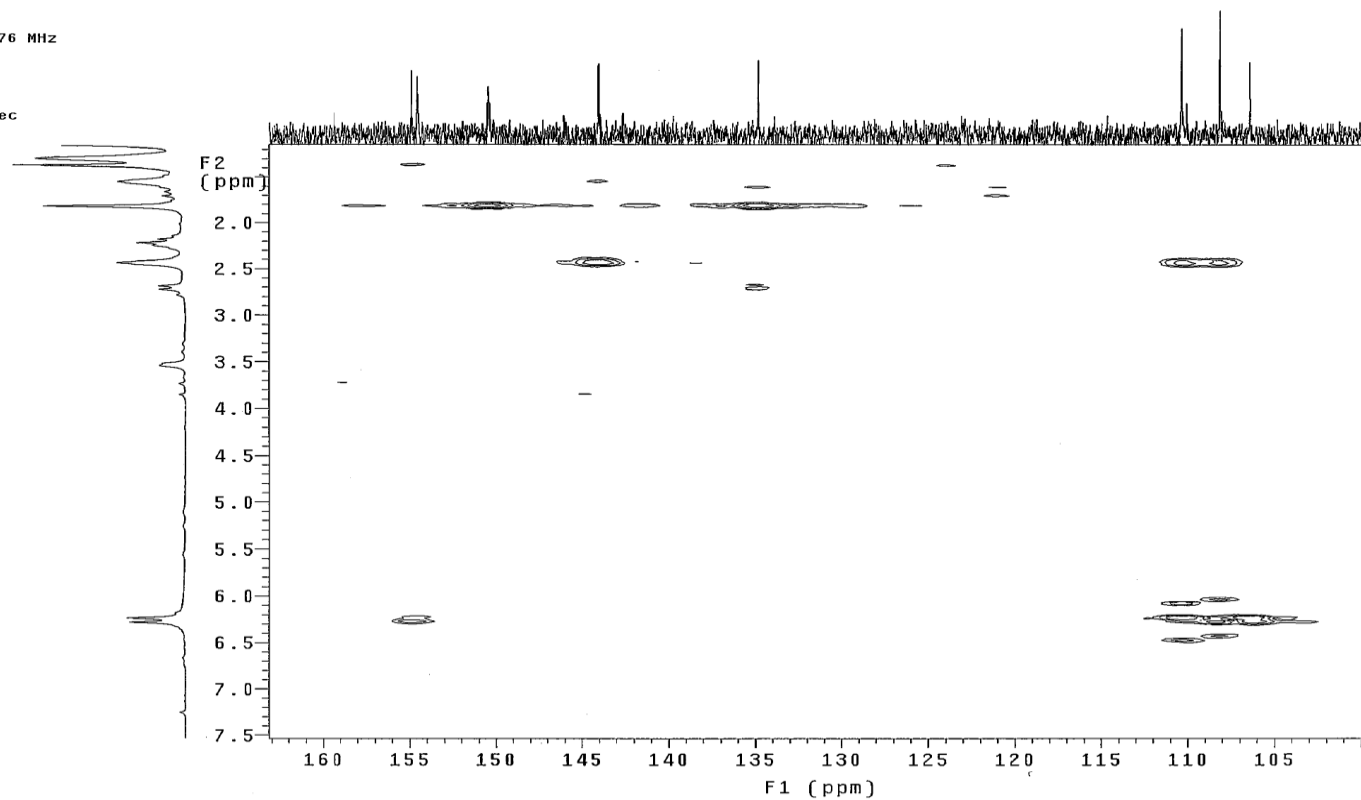
Partial expansions of the 2D-HMBC spectrum of compound 6

STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D width 24154.6 Hz  
4 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
F1 size 2048 x 2048  
Total time 37 min, 17 sec



Partial expansions of the 2D-HMBC spectrum of compound 6

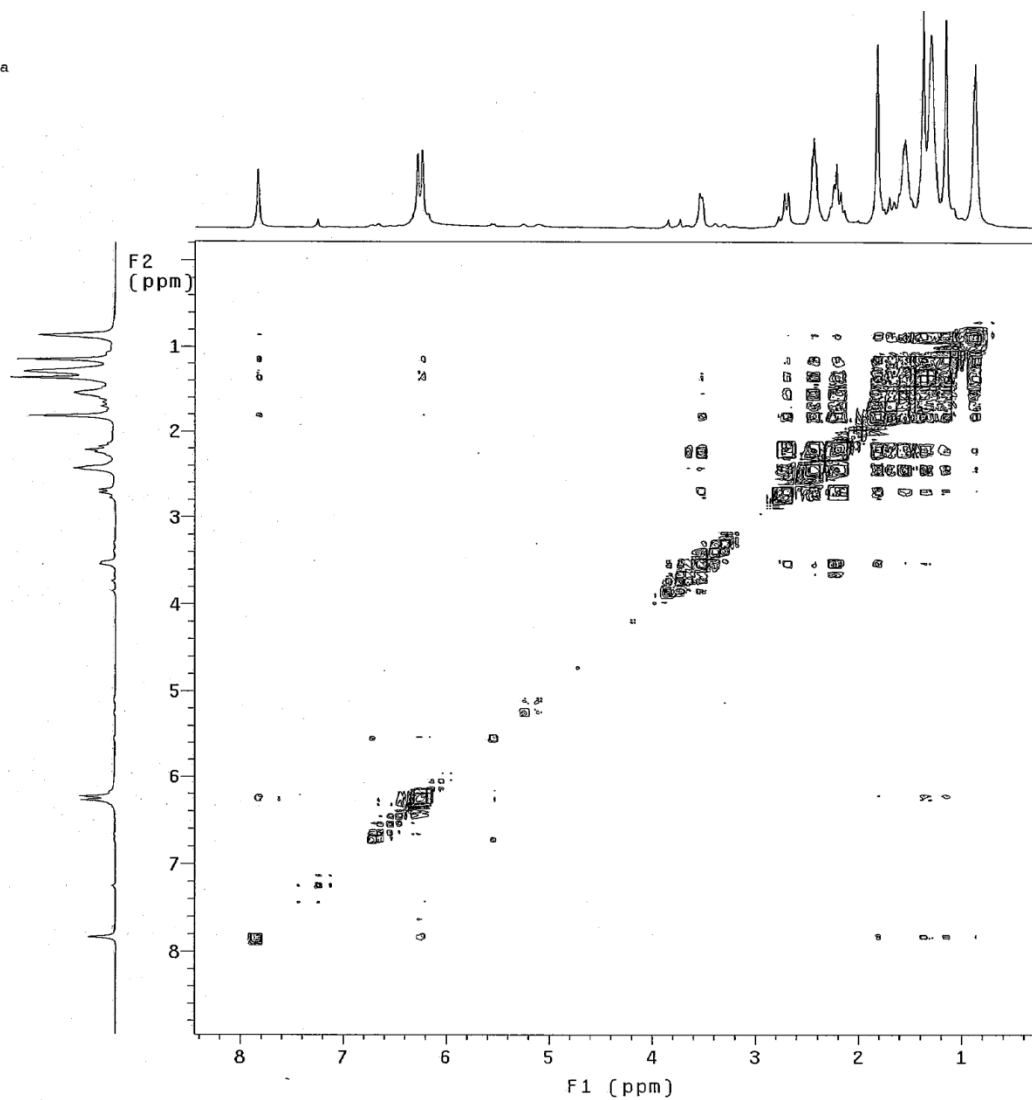
STANDARD 1H OBSERVE

Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
4 repetitions  
128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 11 min, 33 sec



2D-COSY spectrum of compound 6

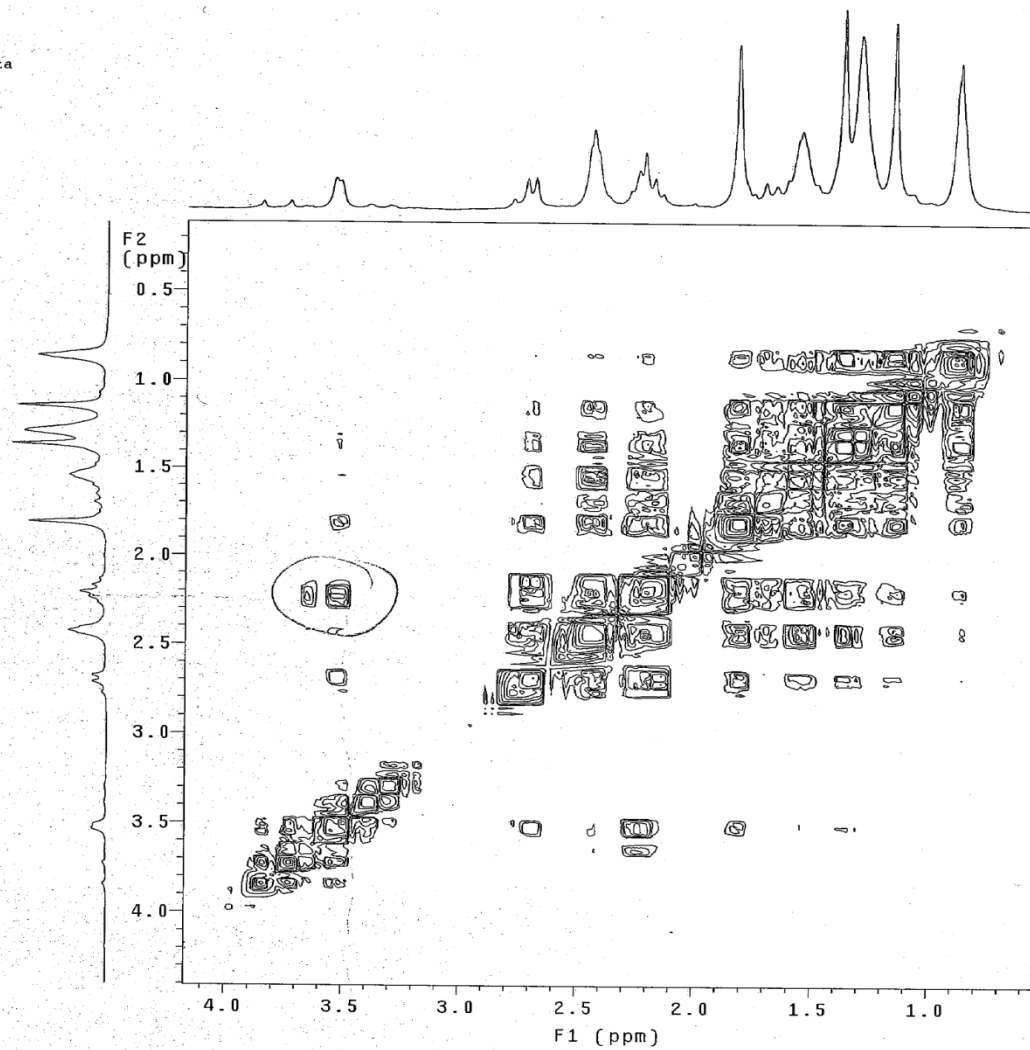


STANDARD 1H OBSERVE

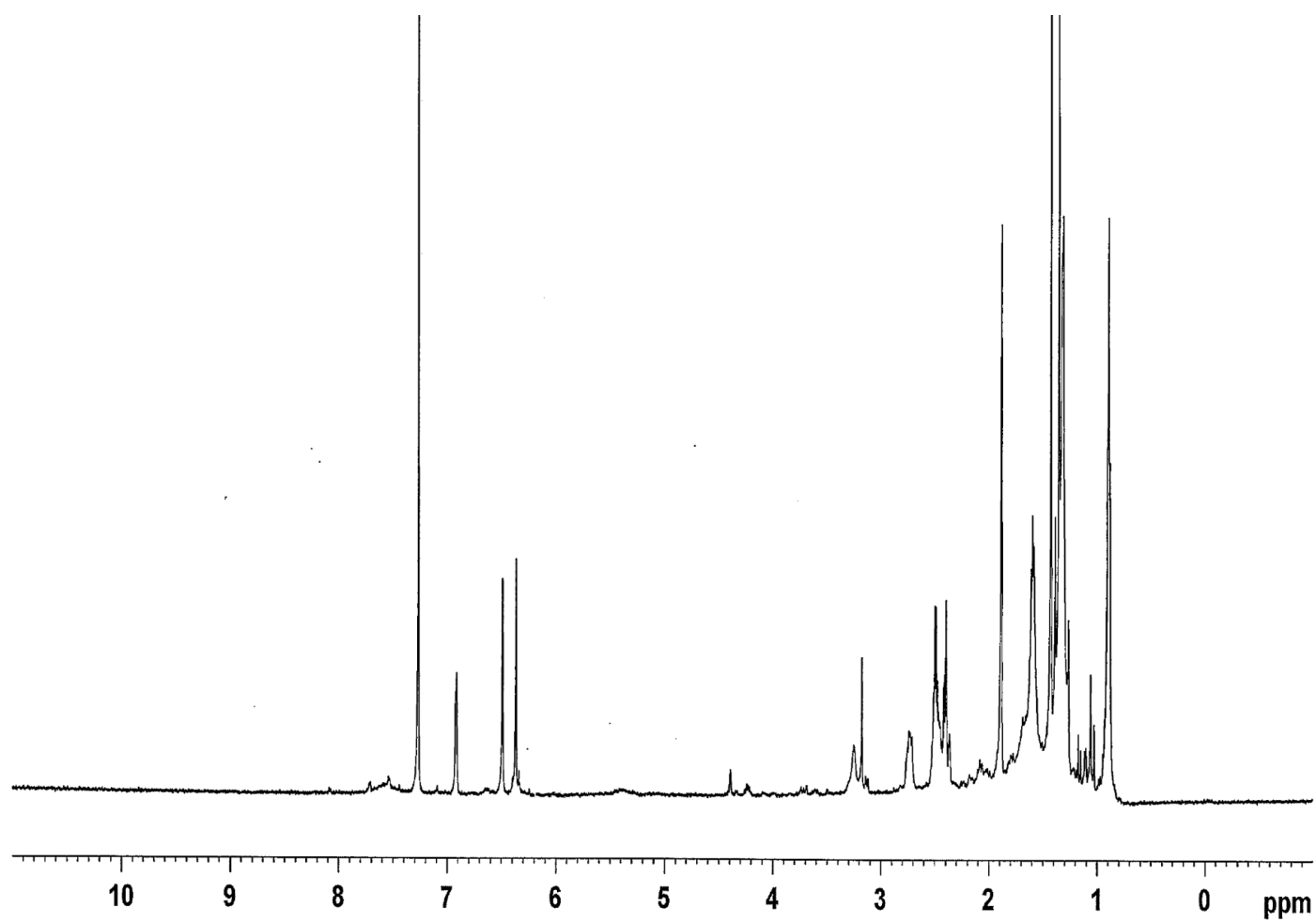
Archive directory: /export/home/safwat/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
4 repetitions  
128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 11 min, 33 sec



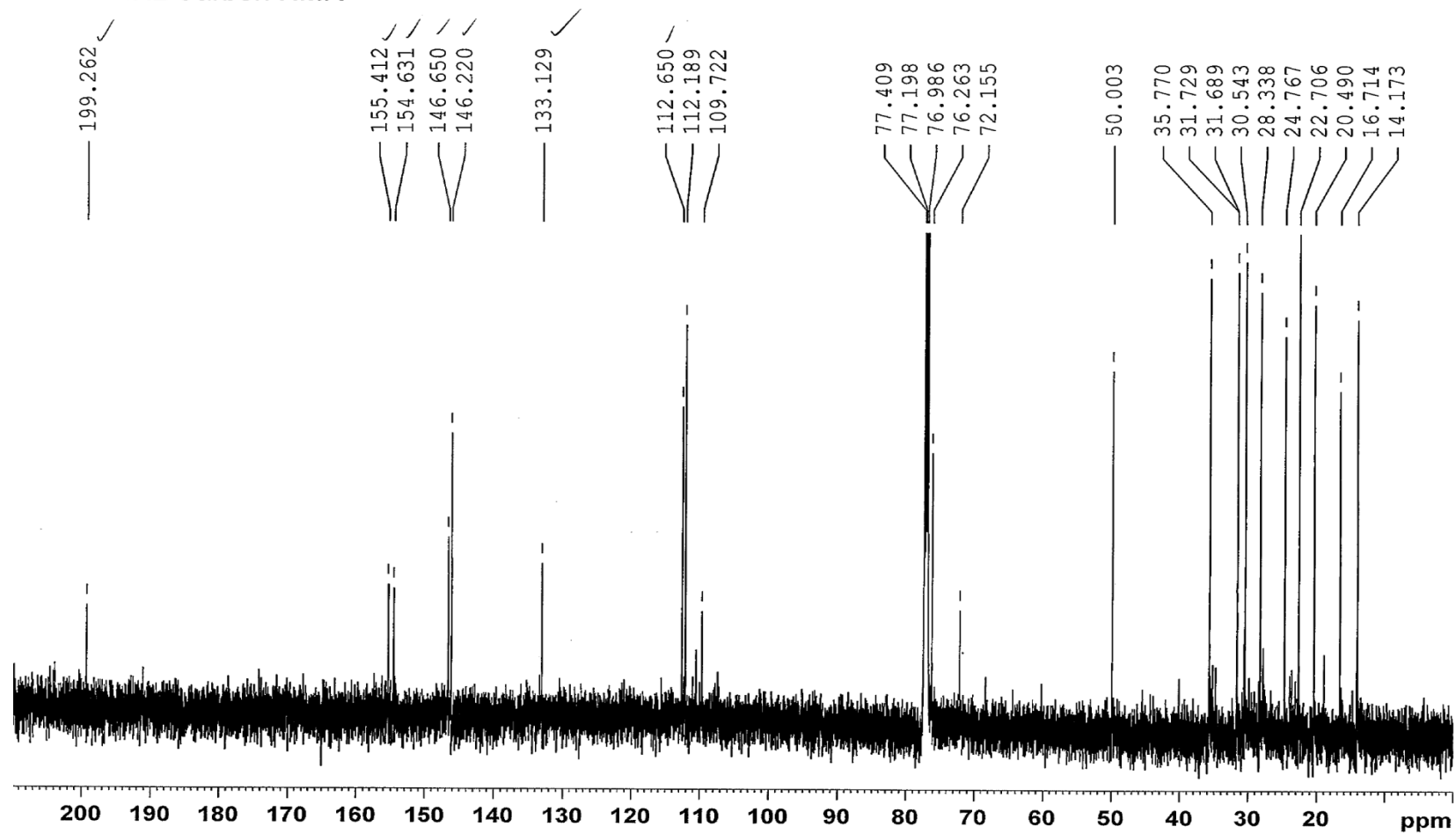
Partial expansions of the 2D-COSY spectrum of compound 6

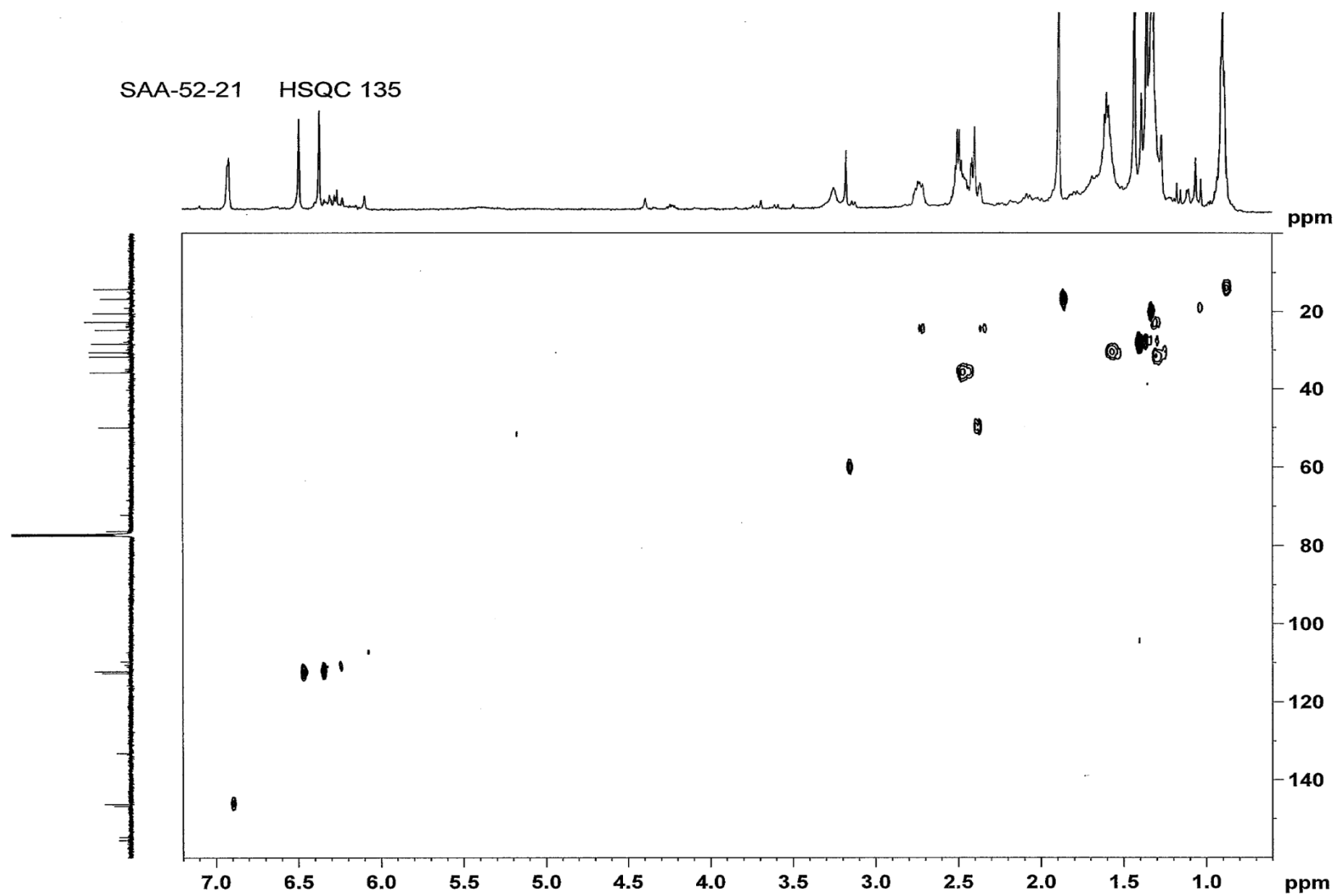


$^1\text{H-NMR}$  spectrum of compound 7 in ( $\text{CDCl}_3$ , 600 MHz)

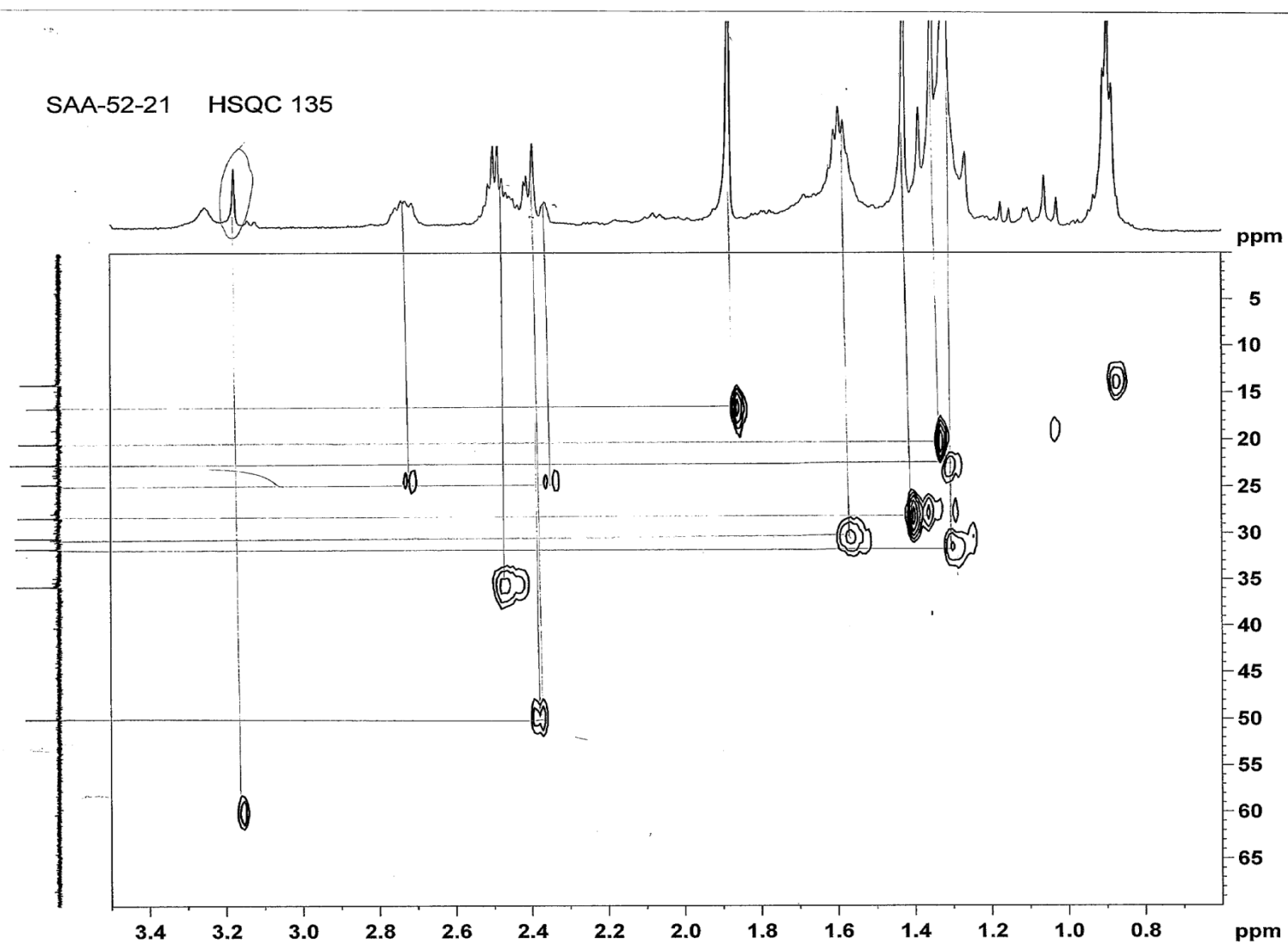
SAA-52-21

150 MHz Carbon NMR

 $^{13}\text{C}$ -NMR spectrum of compound 7 in ( $\text{CDCl}_3$ , 150 MHz)

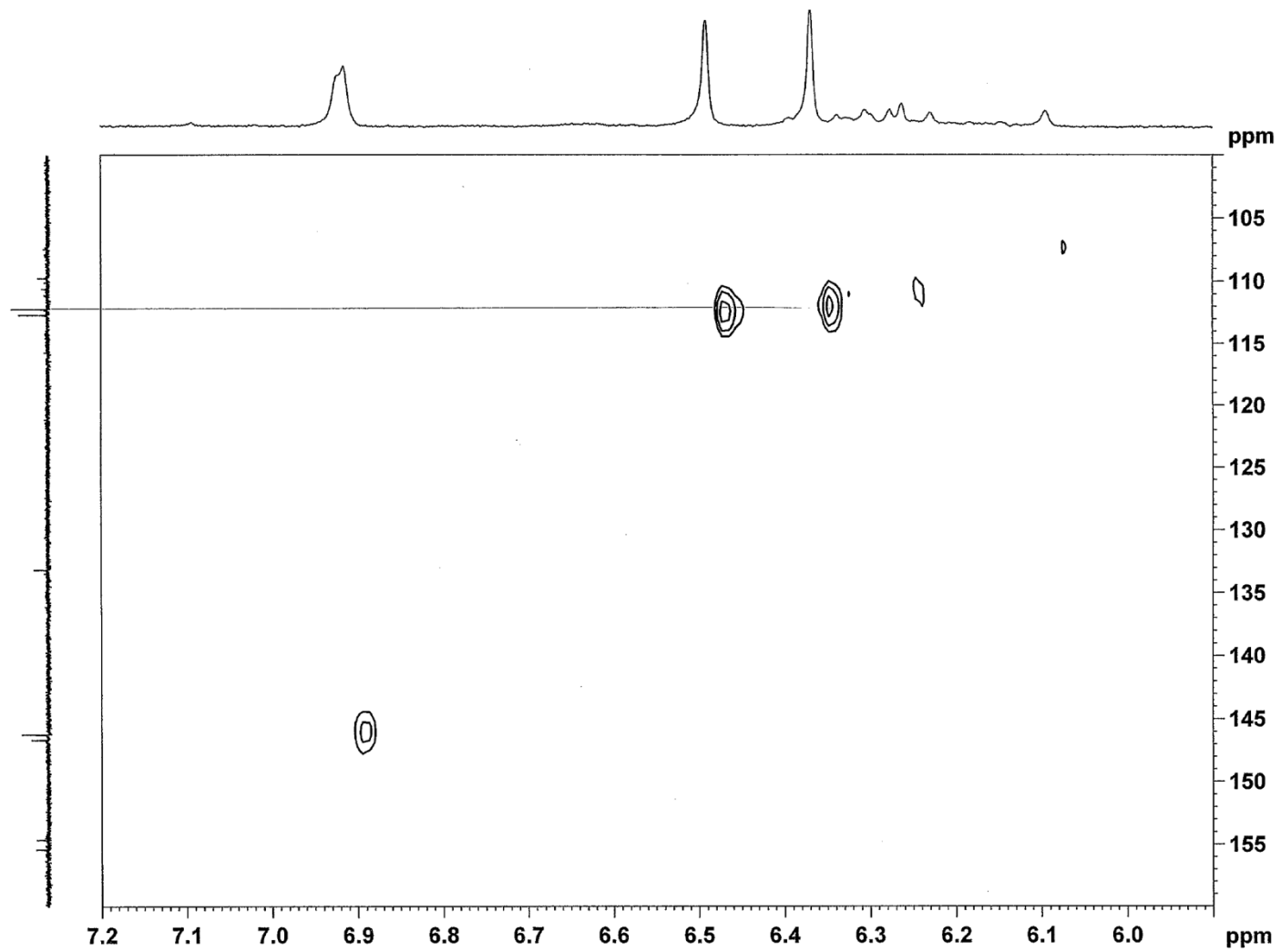


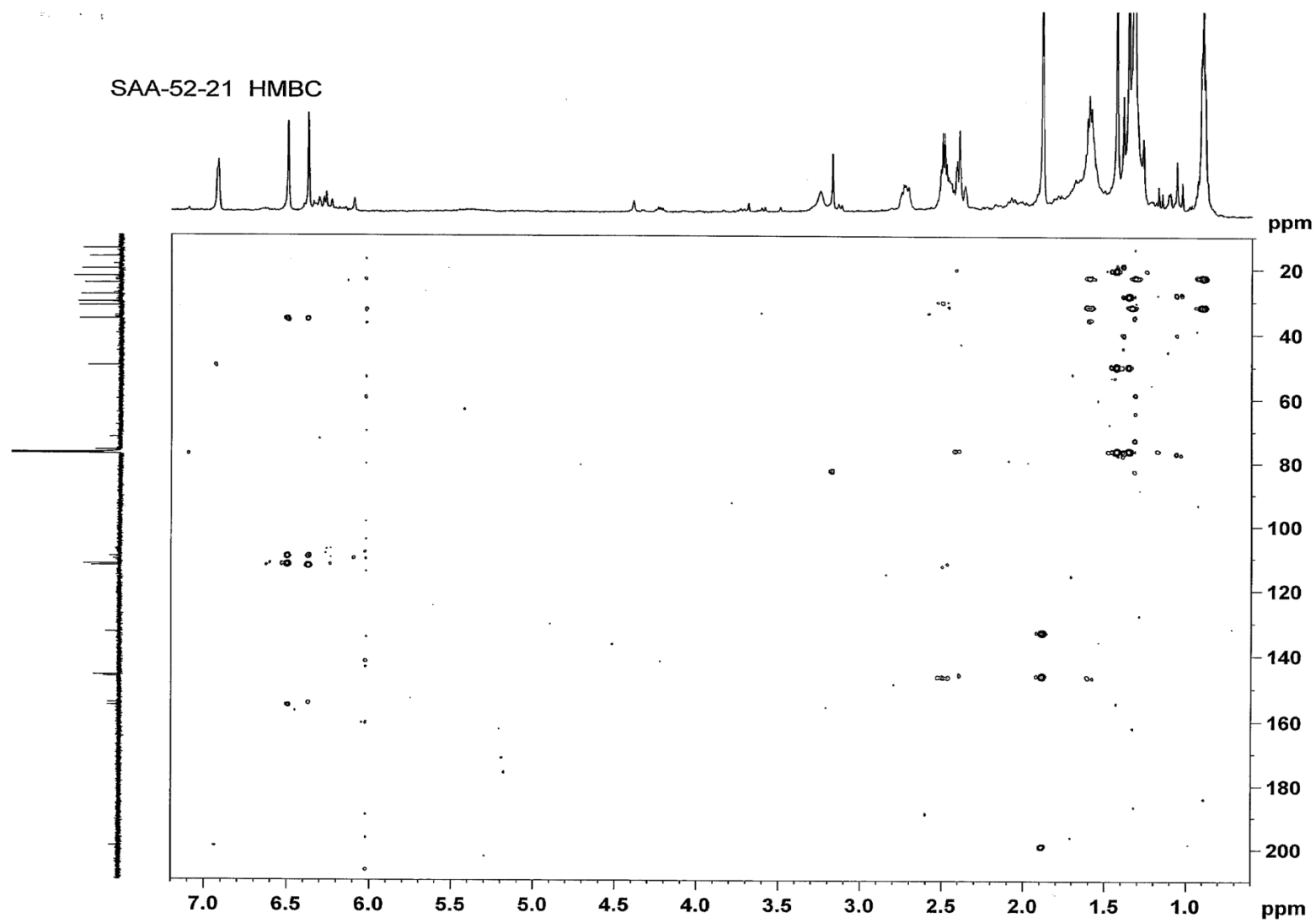
2D-HSQC spectrum of compound 7



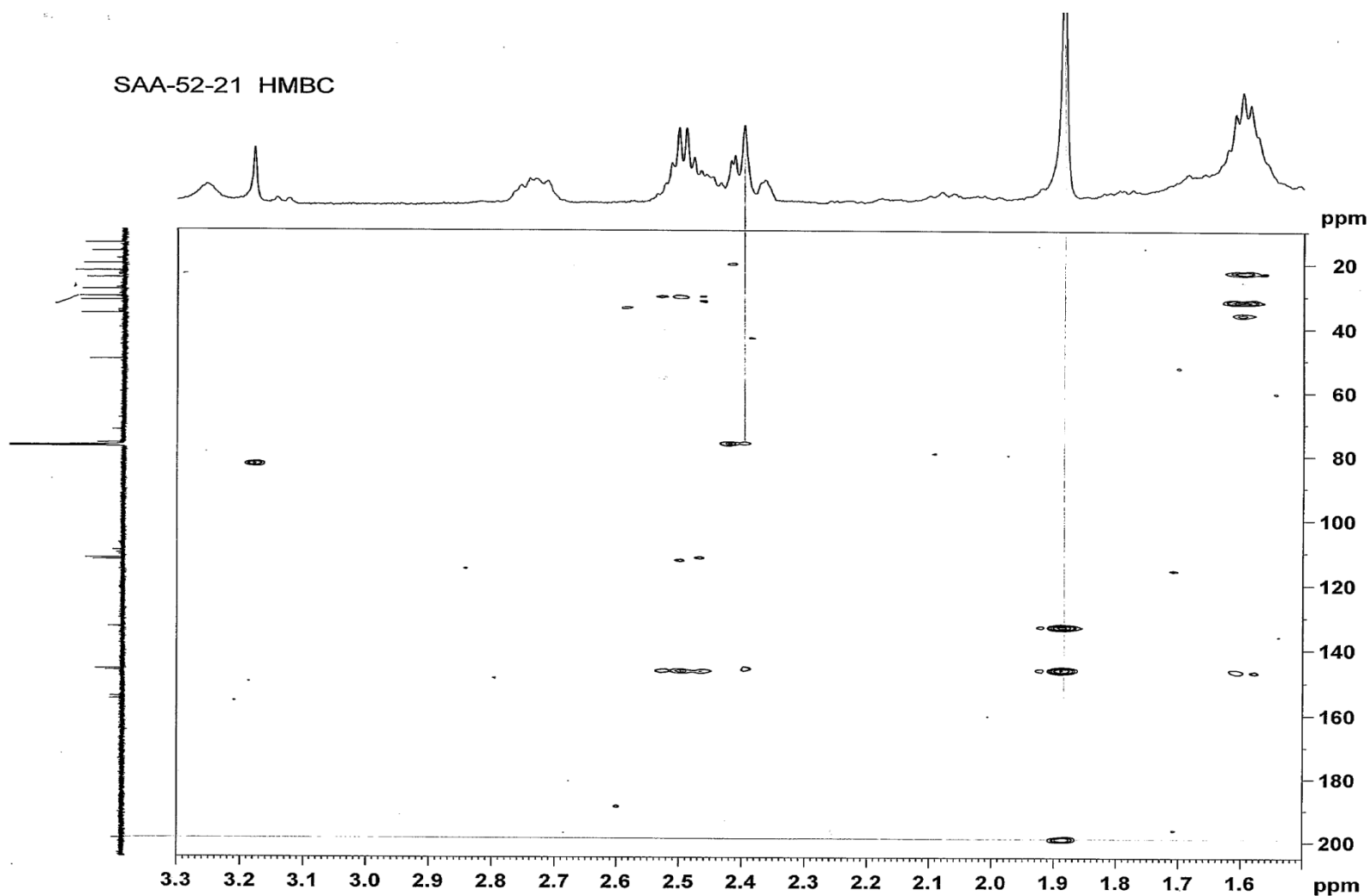
Partial expansions of the 2D-HSQC spectrum of compound 7

SAA-52-21 HSQC 135





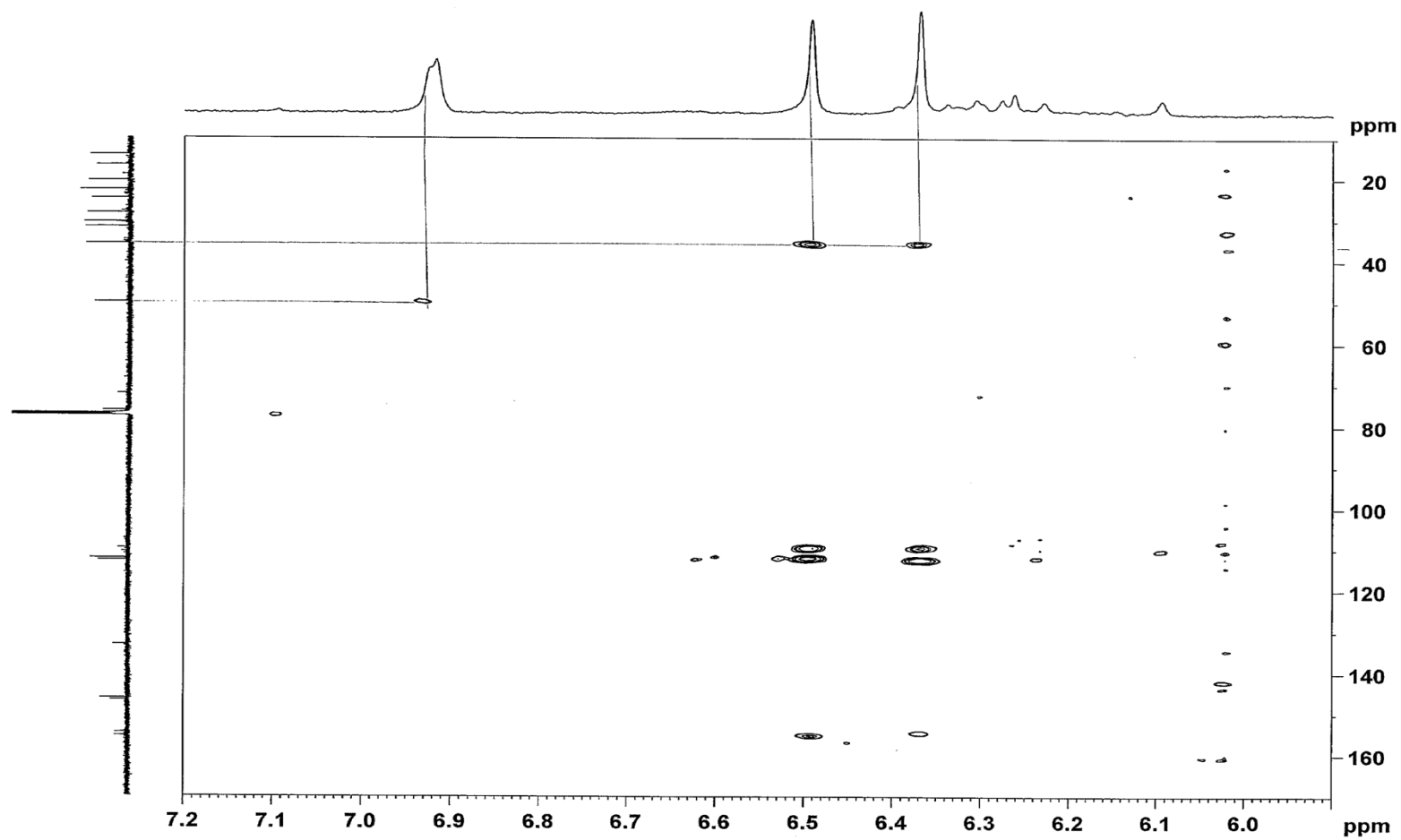
2D-HMBC spectrum of compound 7



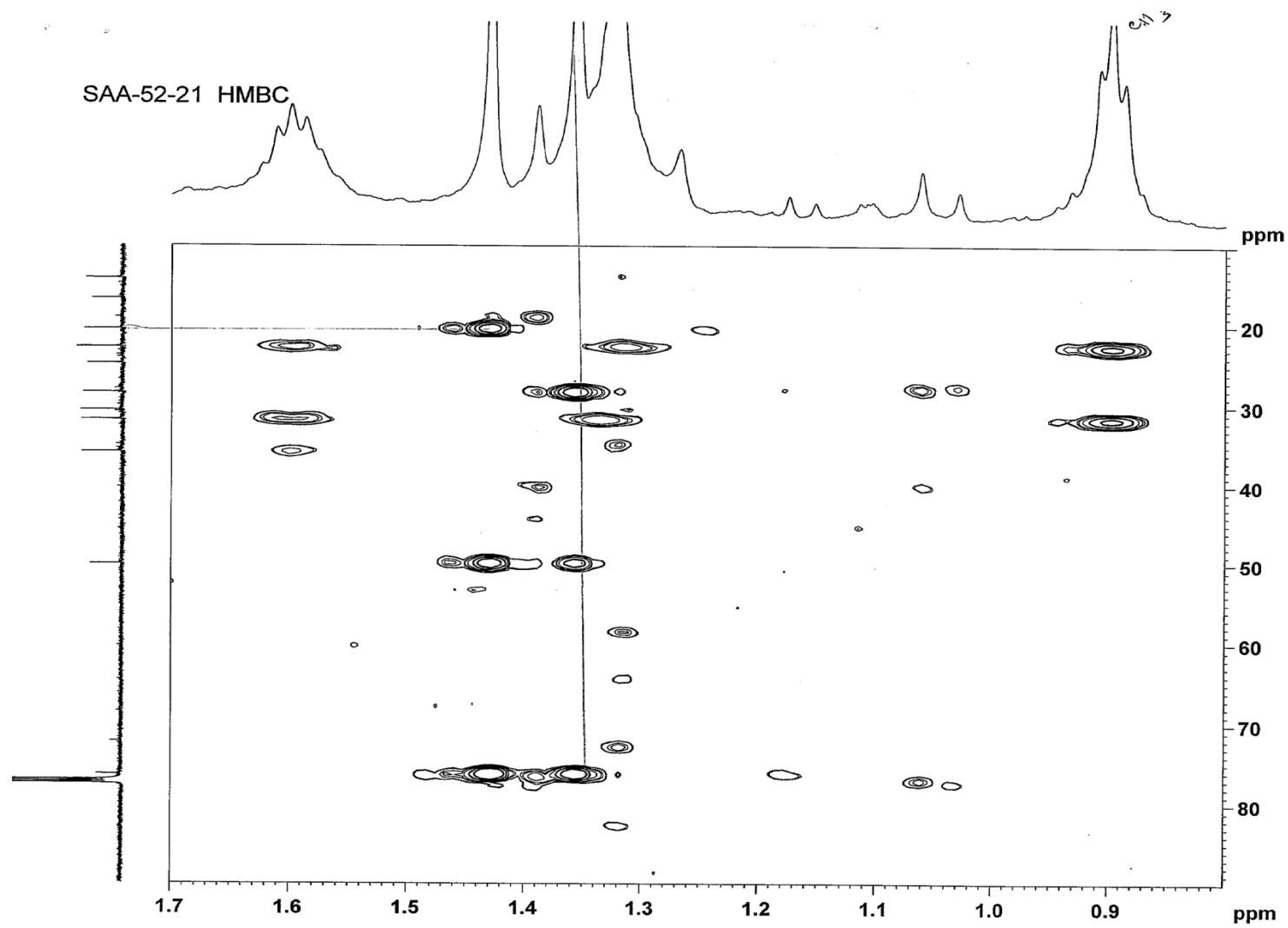
Partial expansions of the 2D- HMBC spectrum of compound 7

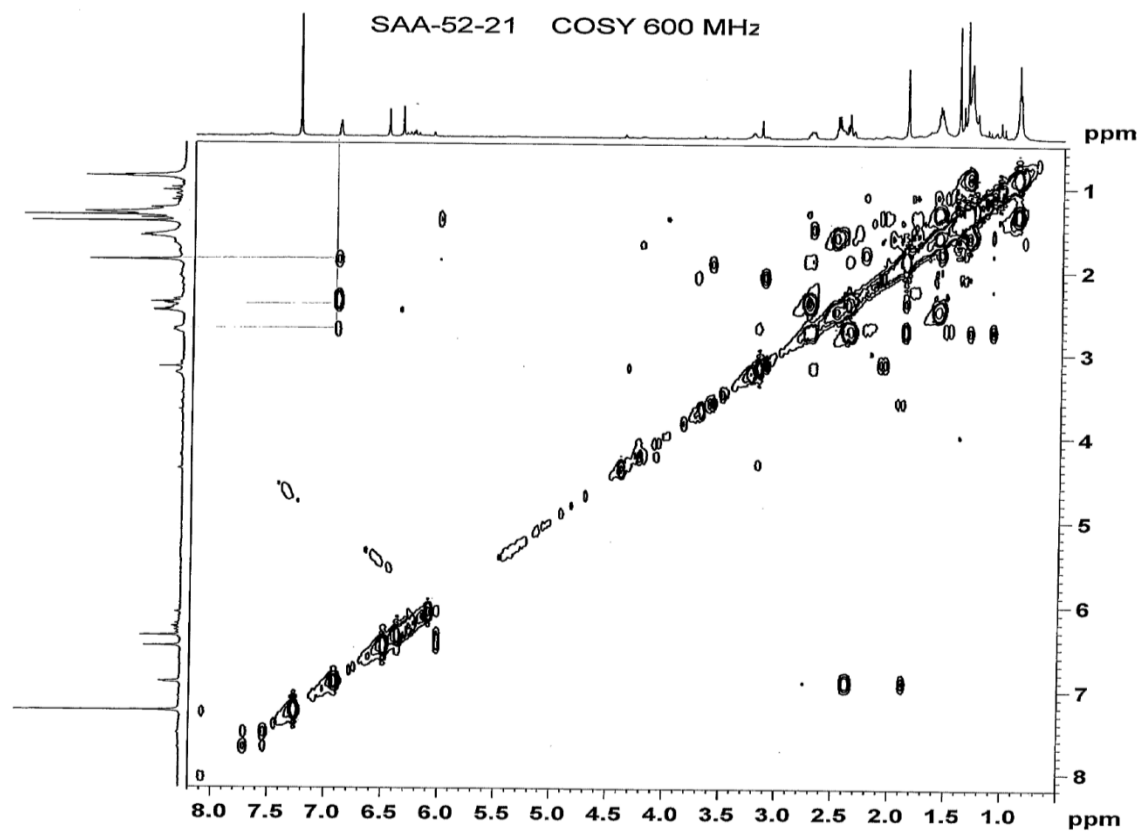


SAA-52-21 HMBC



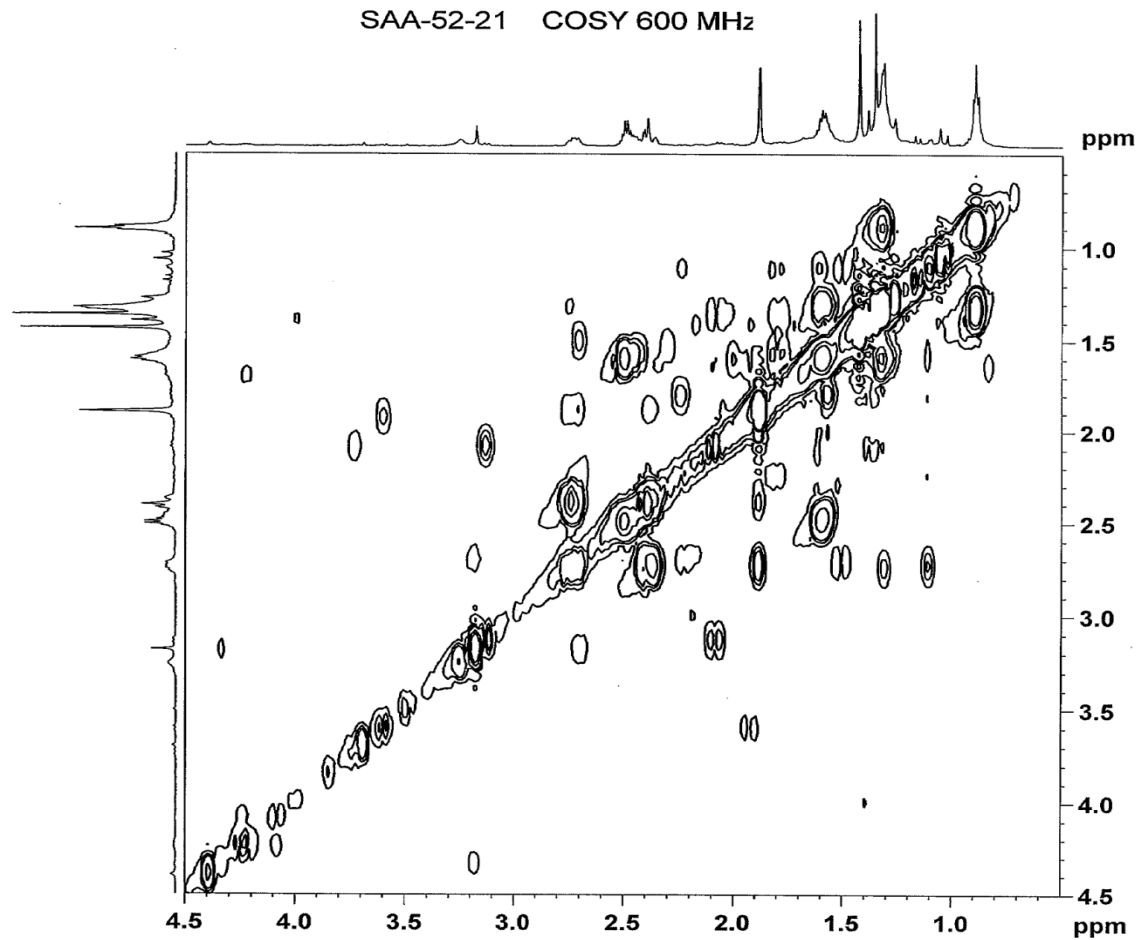
Partial expansions of the 2D- HMBC spectrum of compound 7



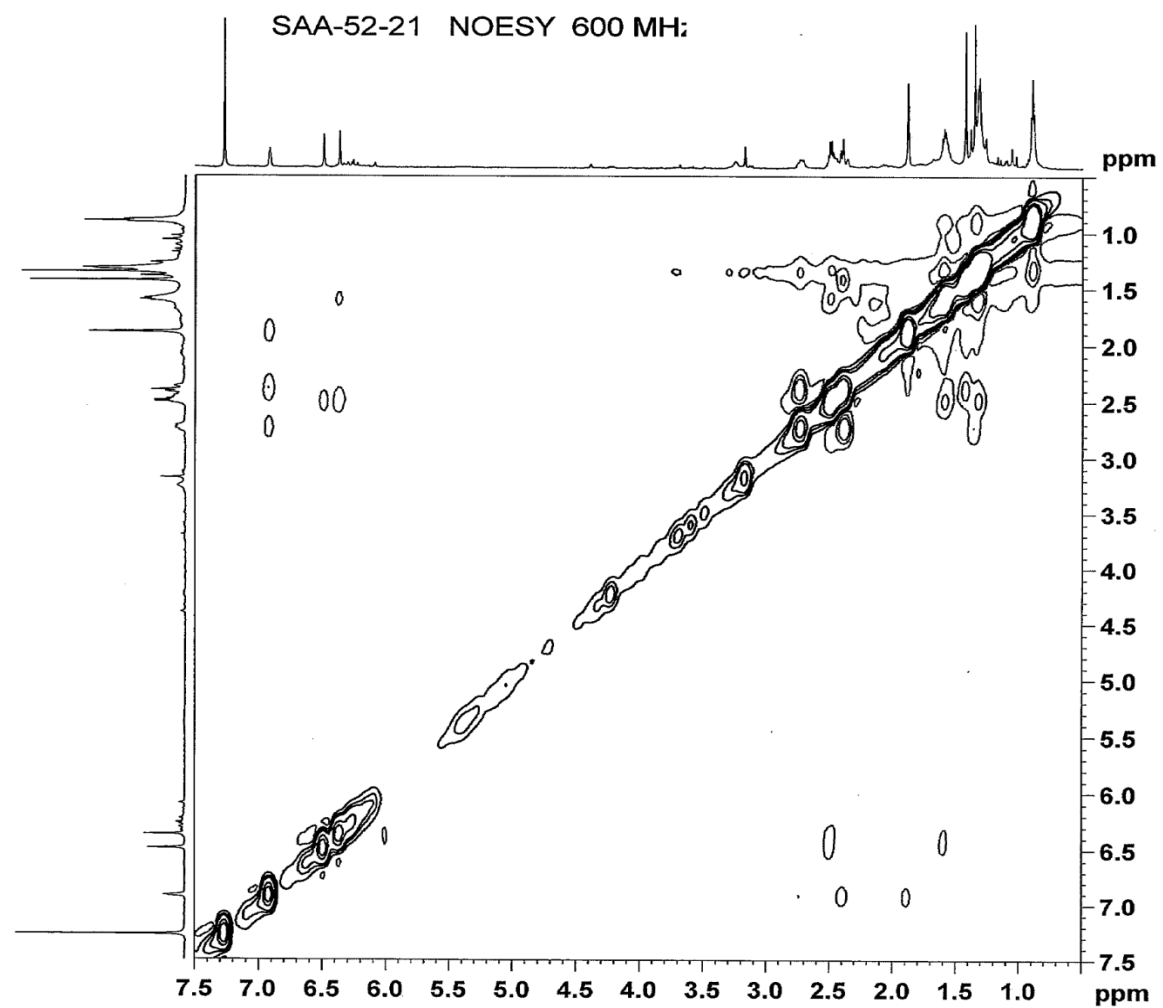


2D-COSY spectrum of compound 7

SAA-52-21 COSY 600 MHz

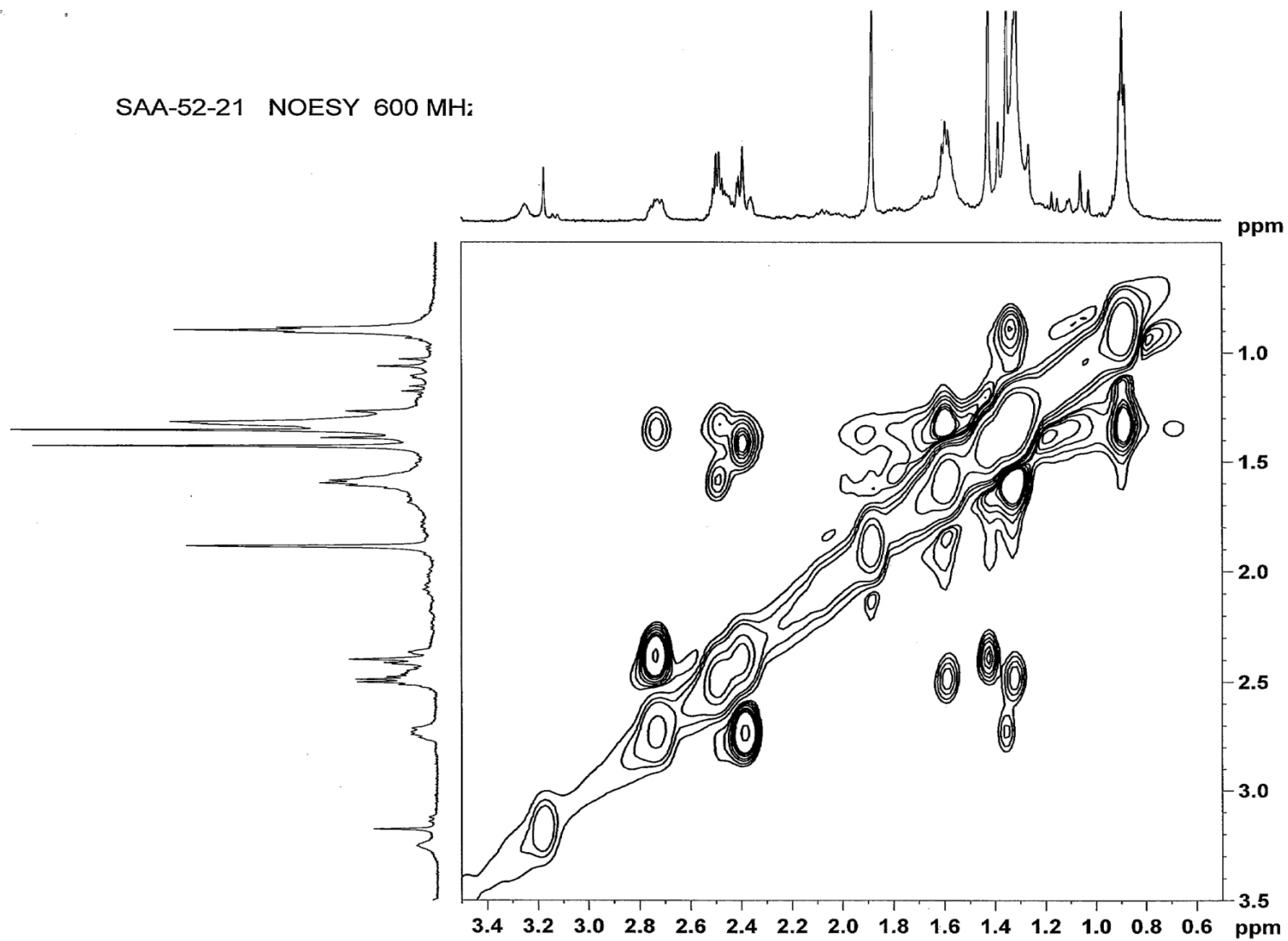


Partial expansions of the 2D-COSY spectrum of compound 7

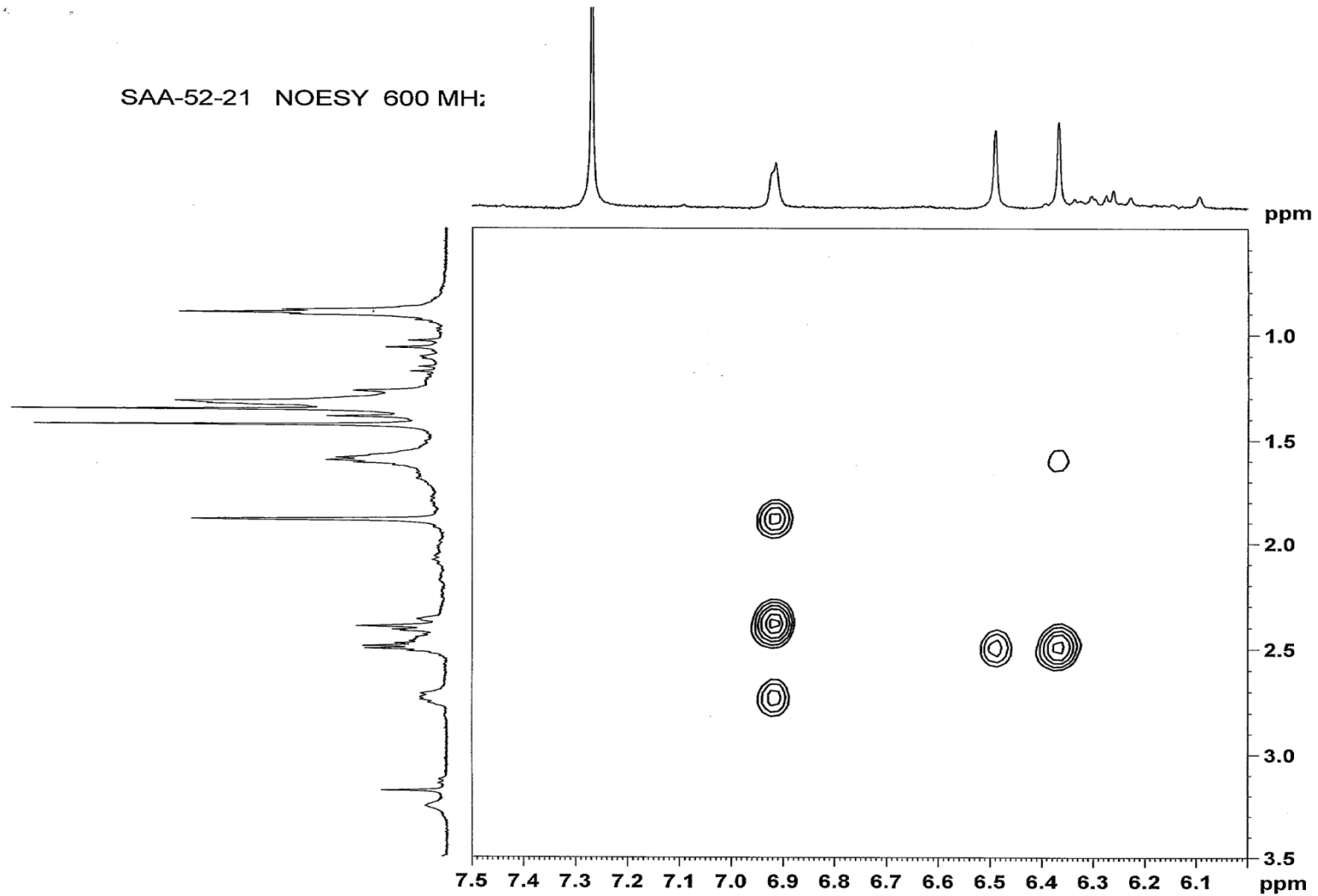


2D-NOESY spectrum of compound 7

SAA-52-21 NOESY 600 MH:



Partial expansions of the 2D-NOESY spectrum of compound 7



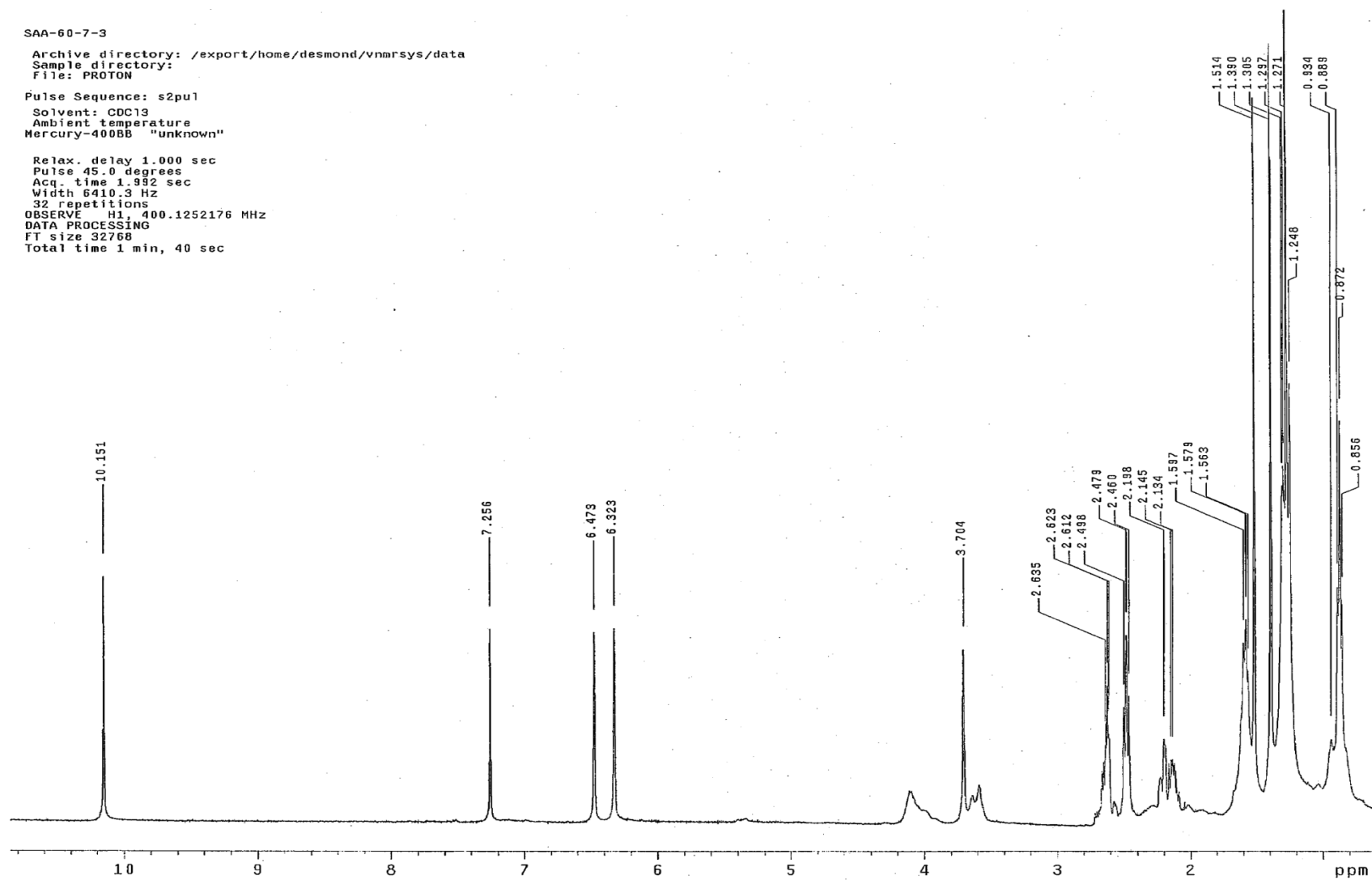
Partial expansions of the 2D-NOESY spectrum of compound 7

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
32 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
FT size 32768  
Total time 1 min, 40 sec



$^1\text{H-NMR}$  spectrum of compound 8 in ( $\text{CDCl}_3$ , 400 MHz)



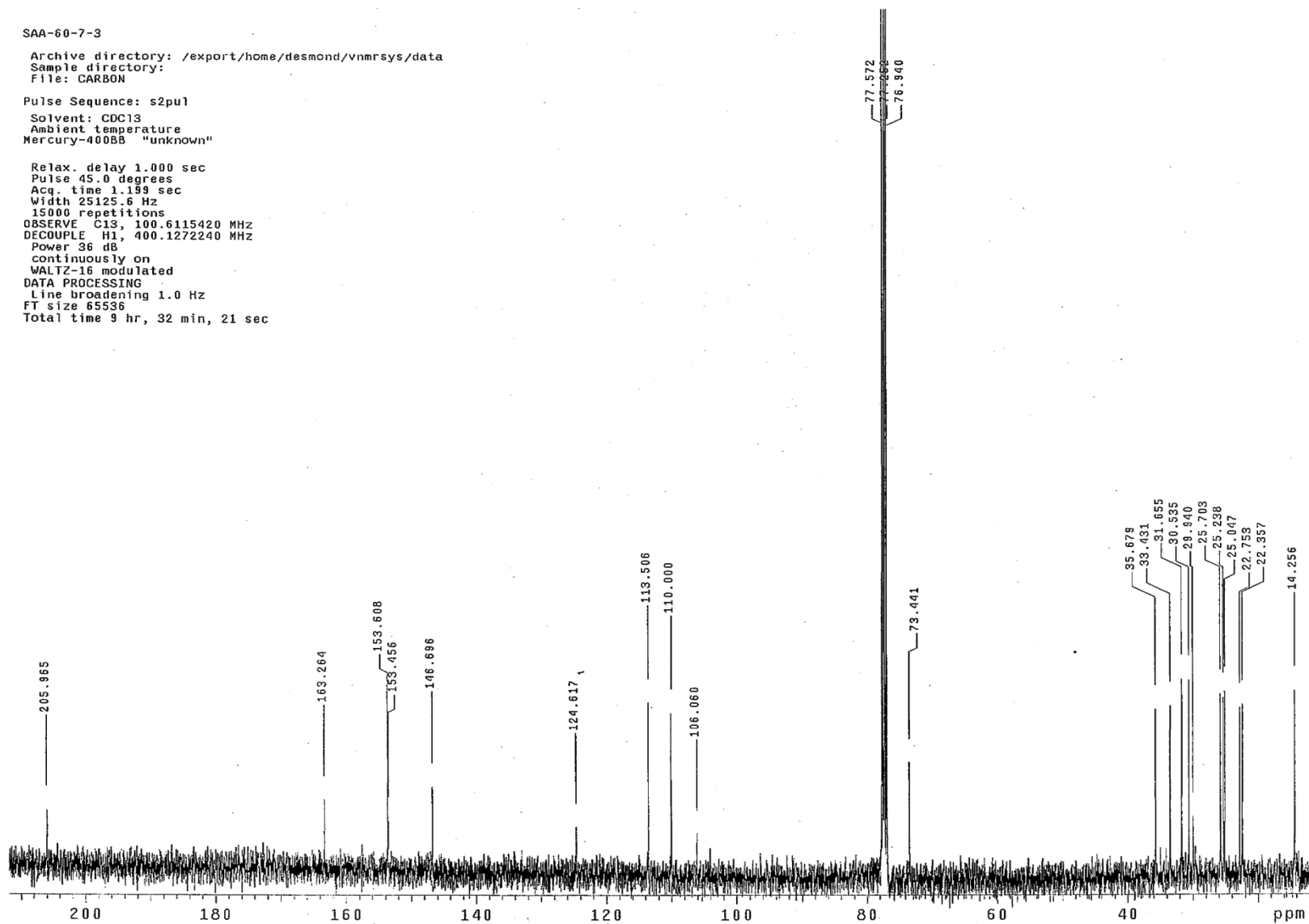
SAA-60-7-3

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

Pulse Sequence: s2pul

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
15000 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 9 hr, 32 min, 21 sec



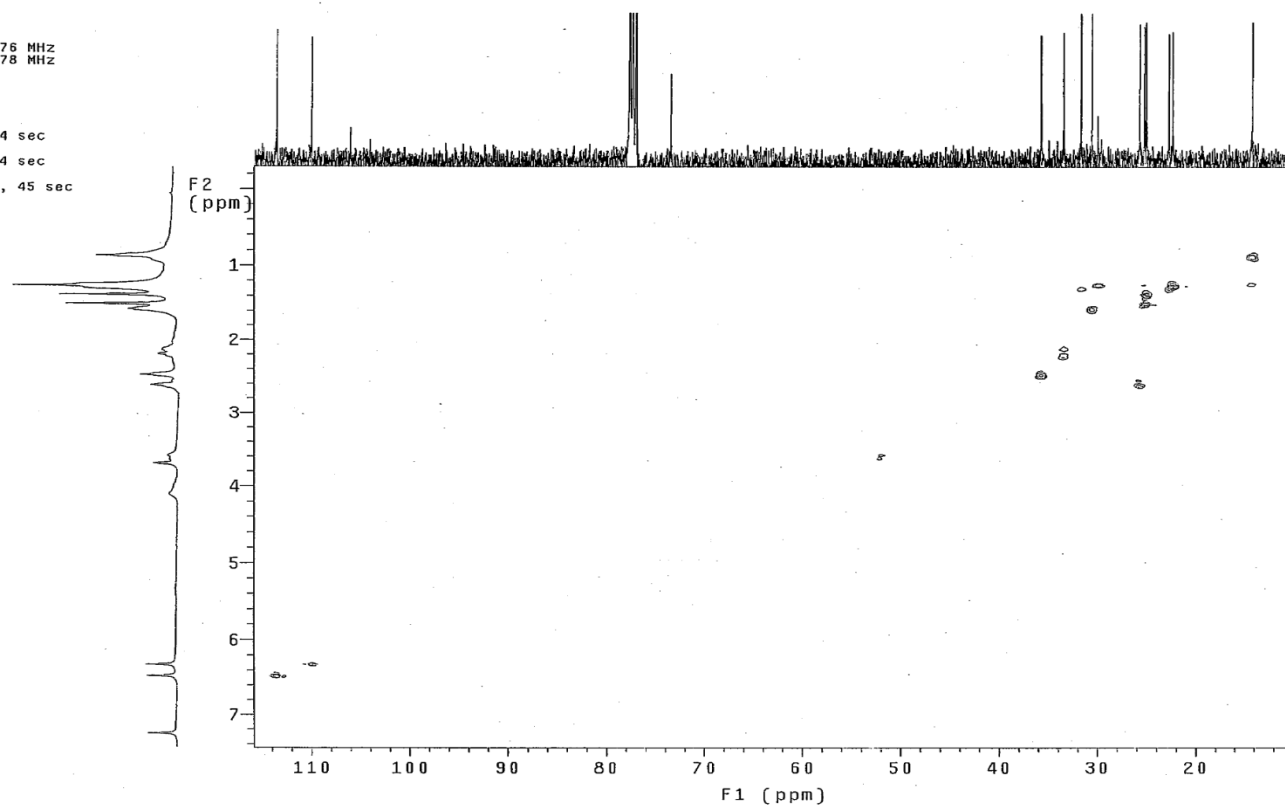
<sup>13</sup>C-NMR spectrum of compound 8 in (CDCl<sub>3</sub>, 100 MHz)

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-40088 "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



2D-HMQC spectrum of compound 8

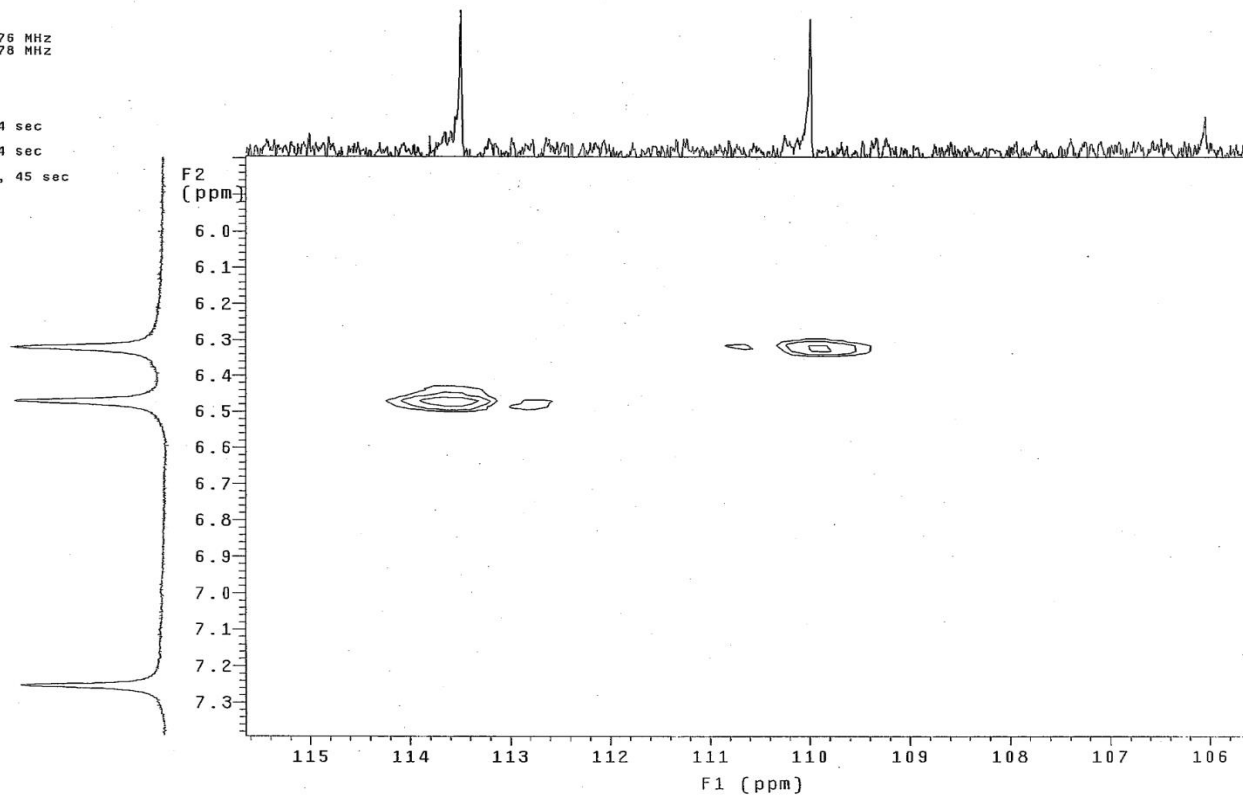
SAA-60-7-3

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



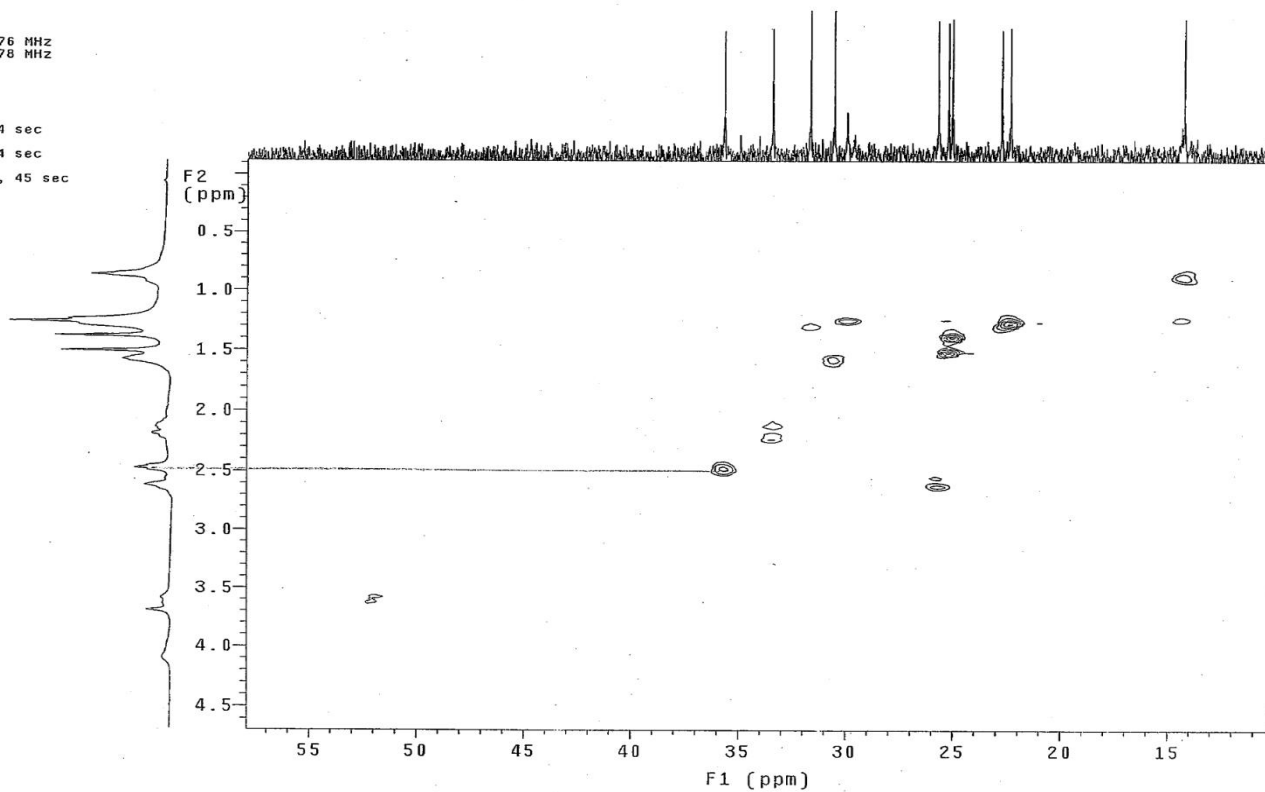
Partial expansions of the 2D-HMQC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



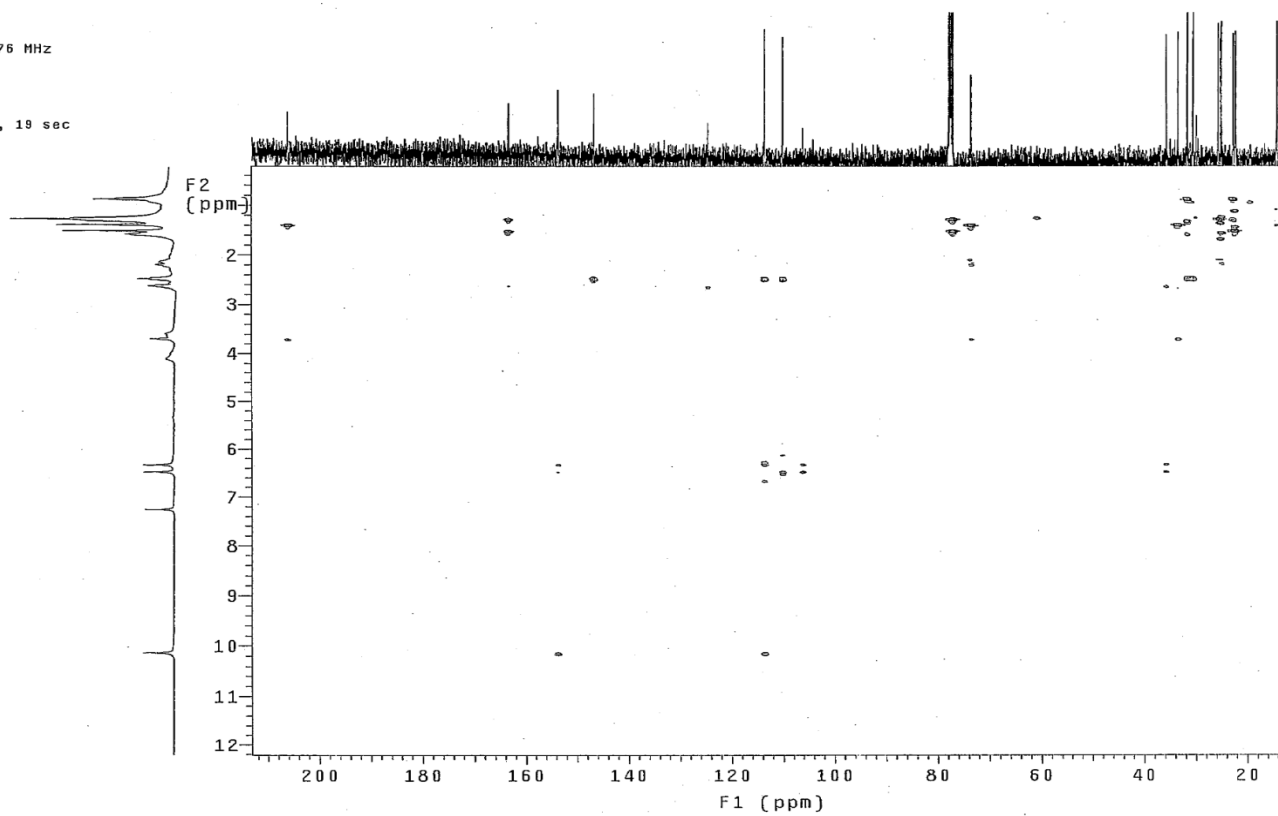
Partial expansions of the 2D-HMOC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



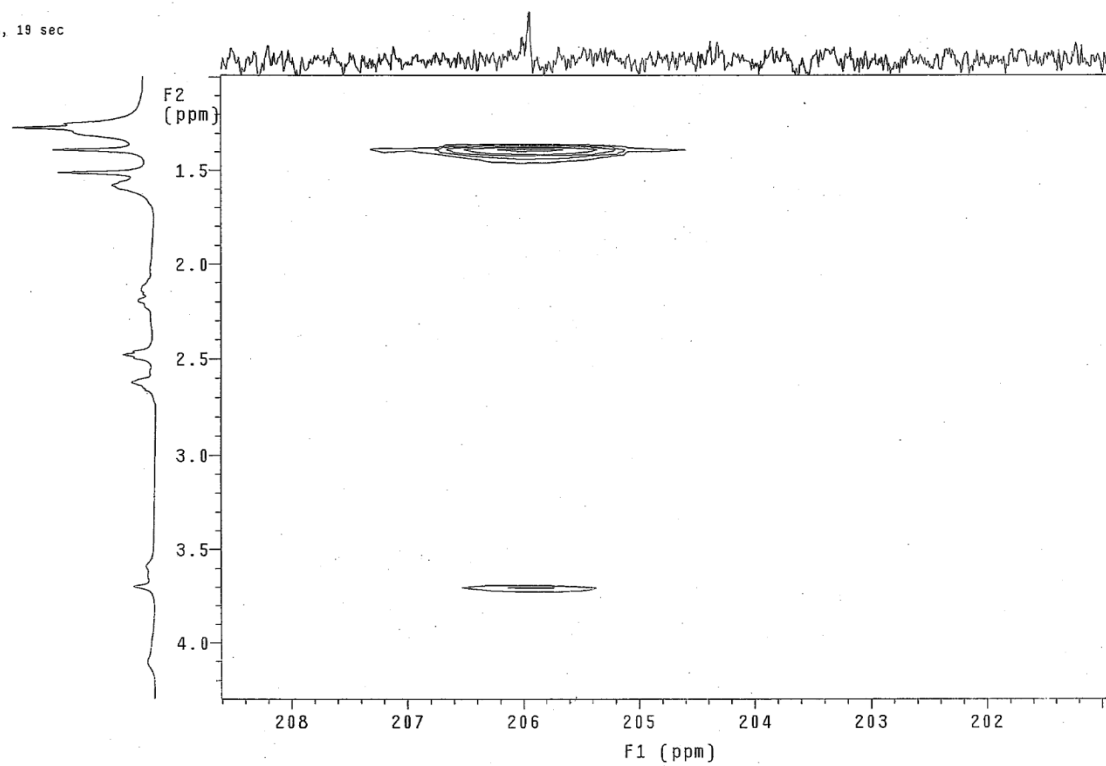
2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



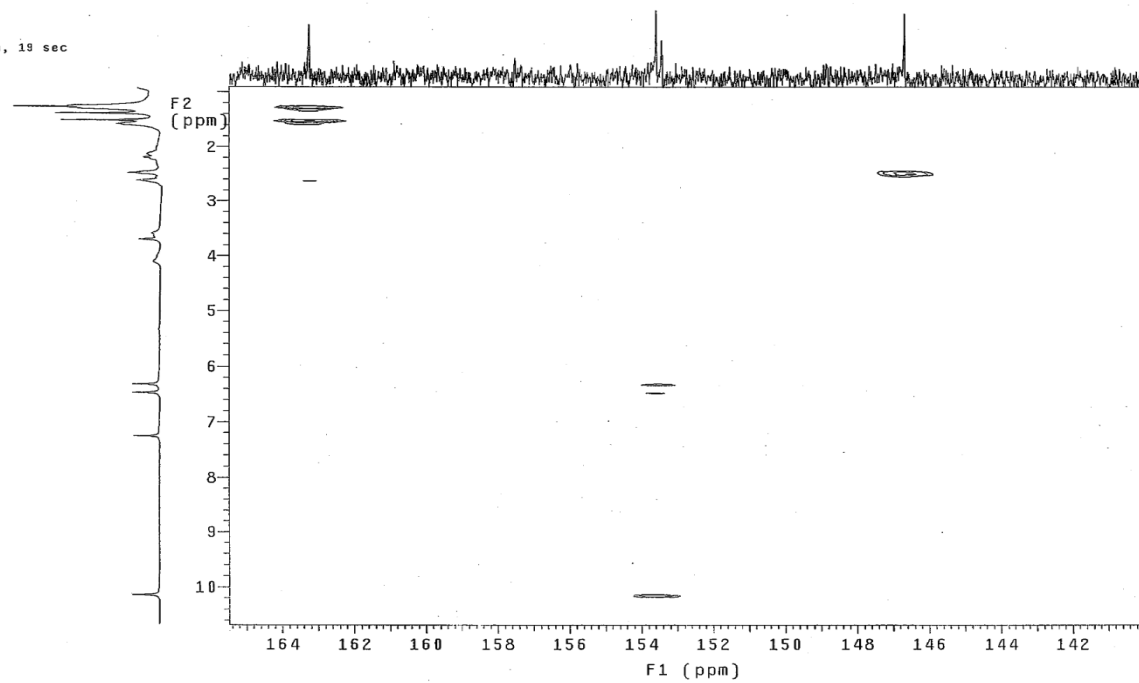
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.000 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



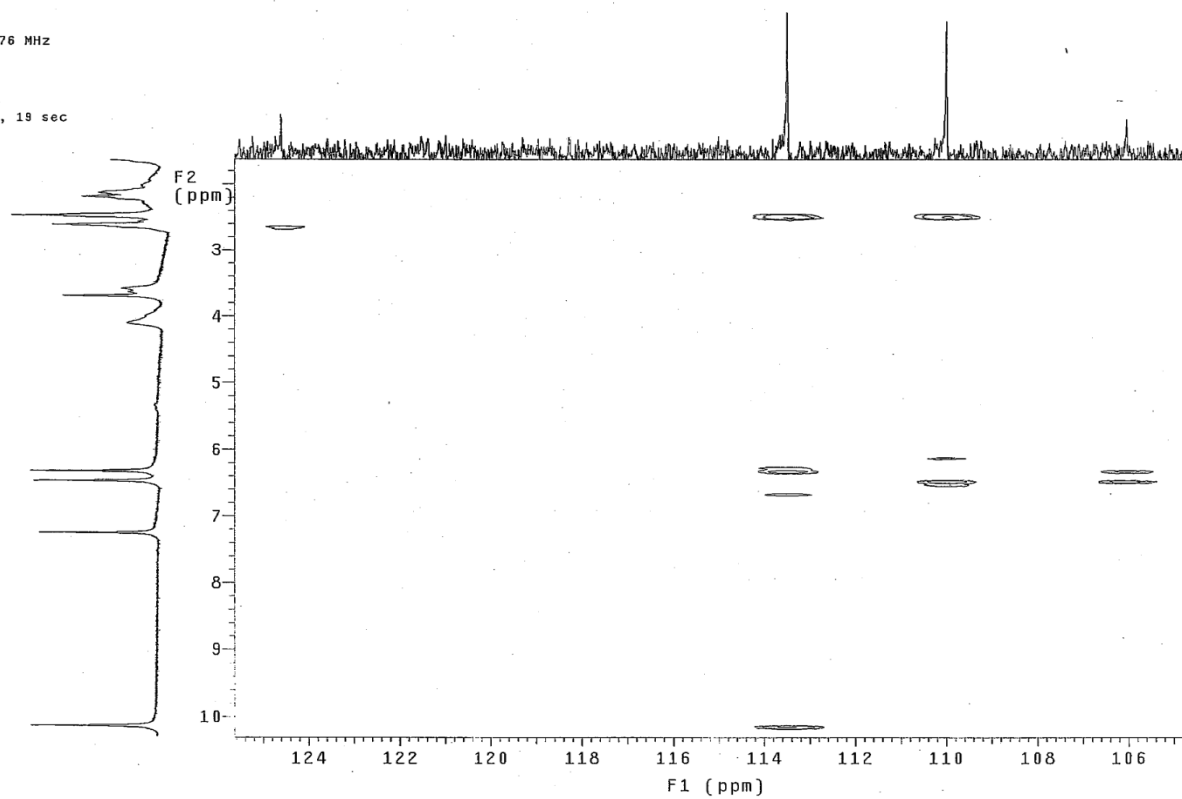
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmr/sys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 8410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



Partial expansions of the 2D-HMBC spectrum of compound 8

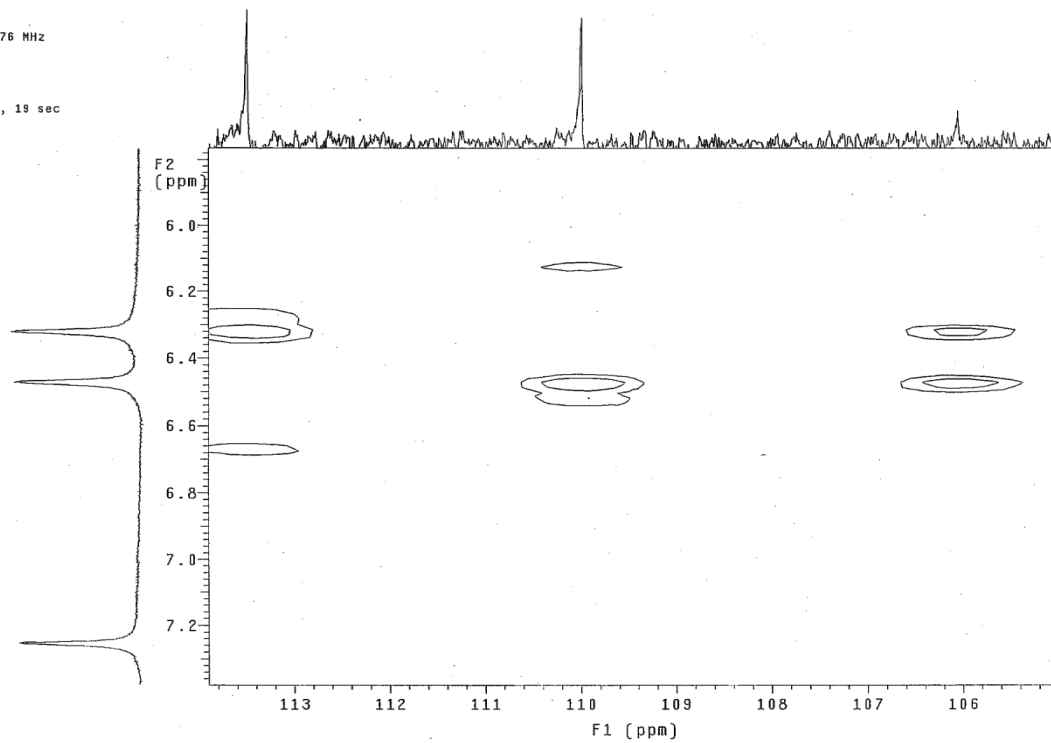


SAA-60-7-3

Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE F1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



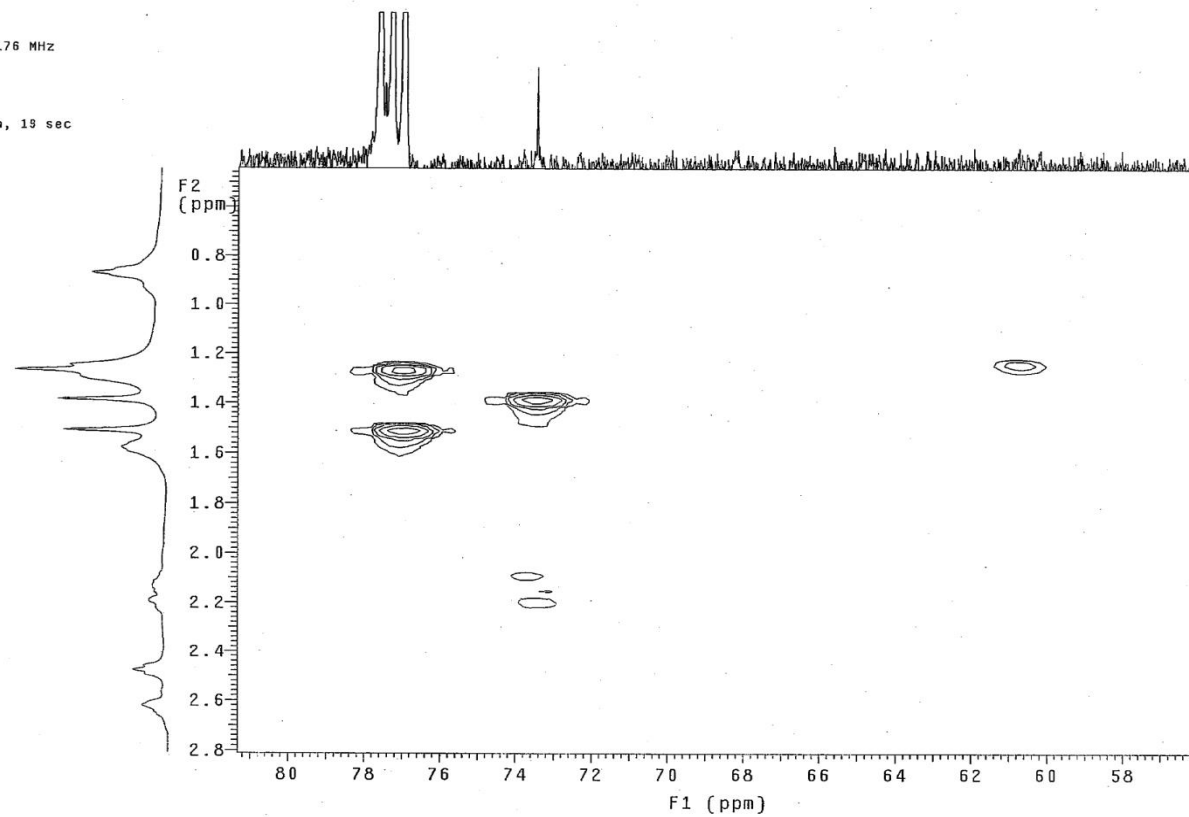
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



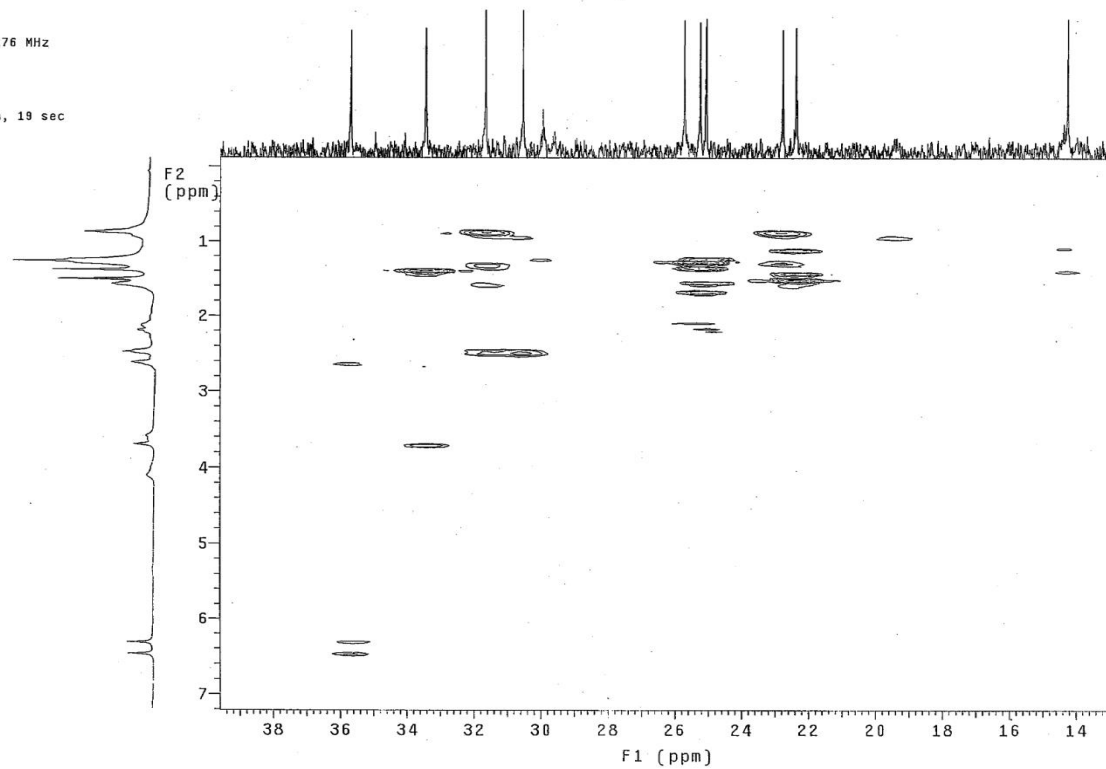
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



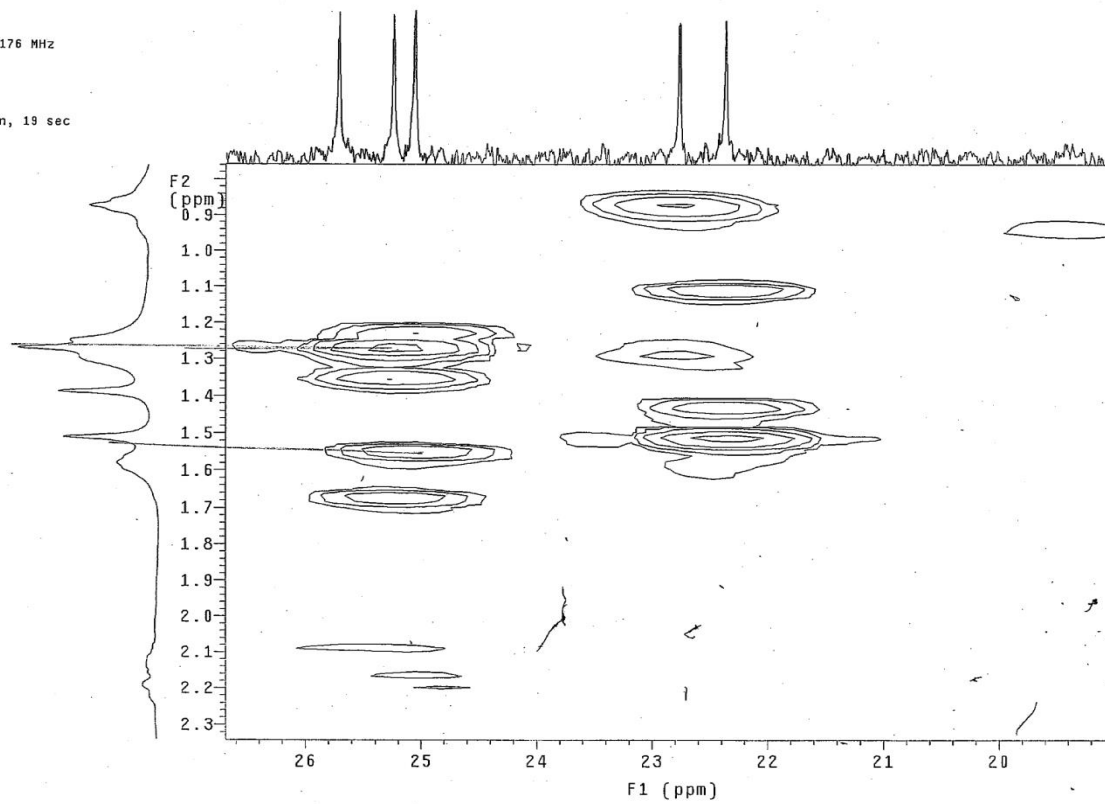
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



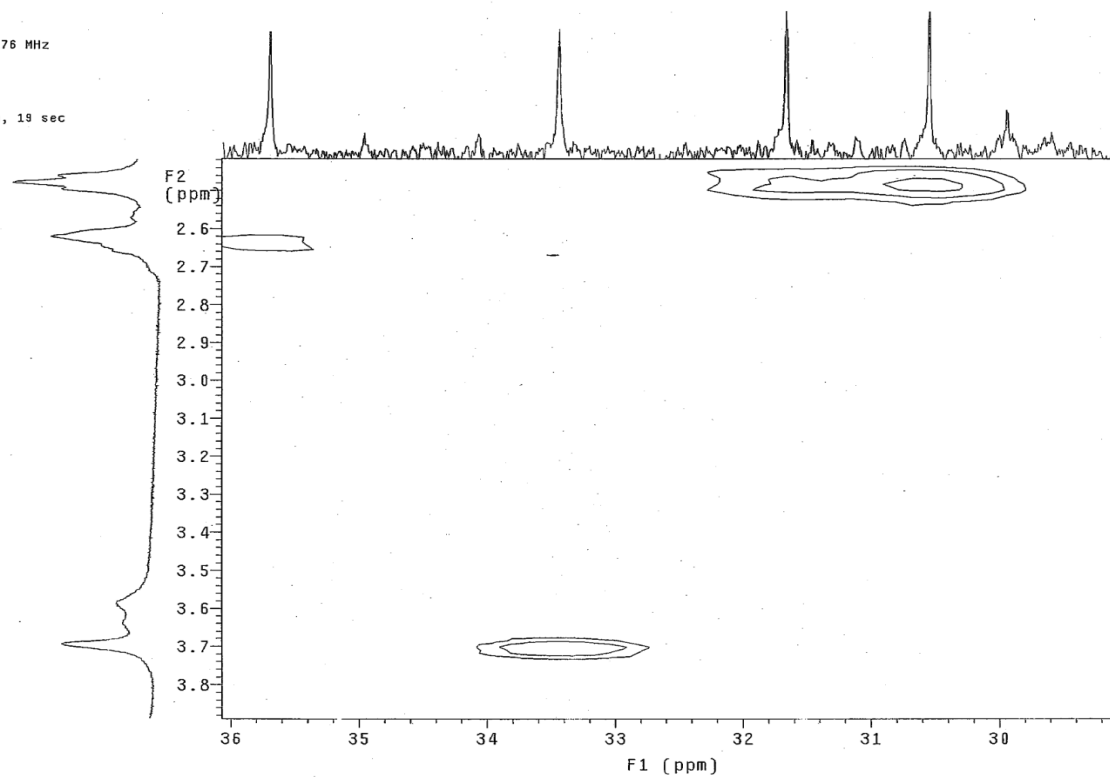
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



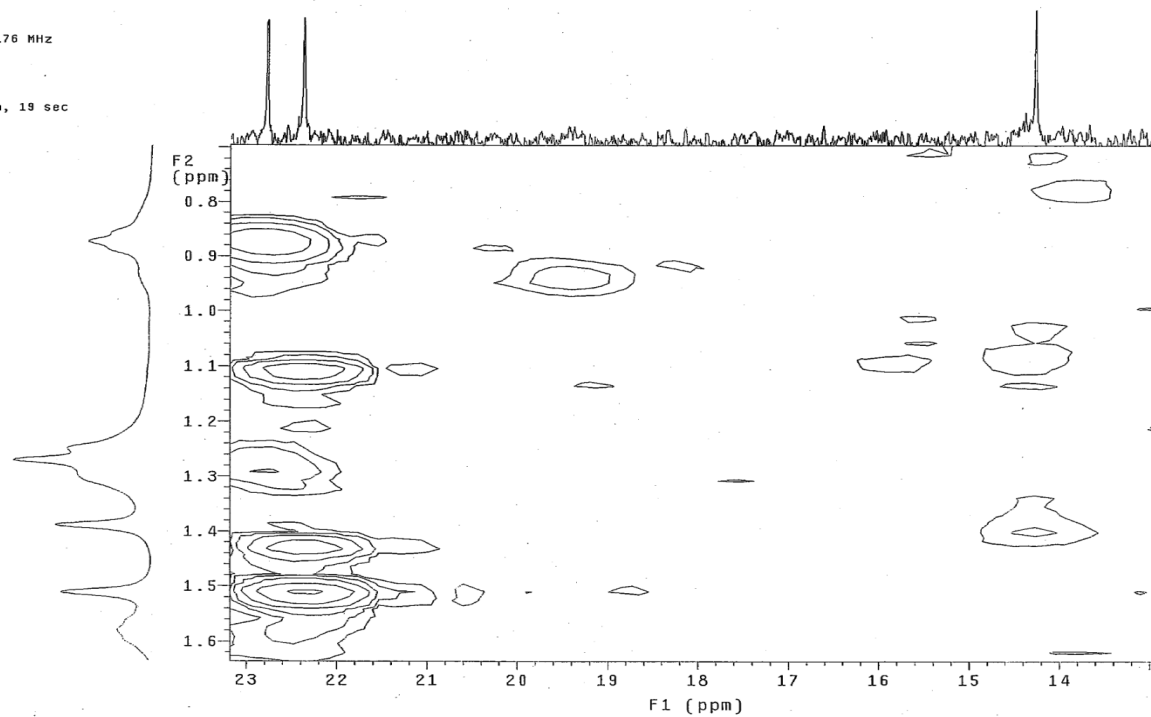
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
36 repetitions  
400 increments  
OBSERVE H1: 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 5 hr, 14 min, 19 sec



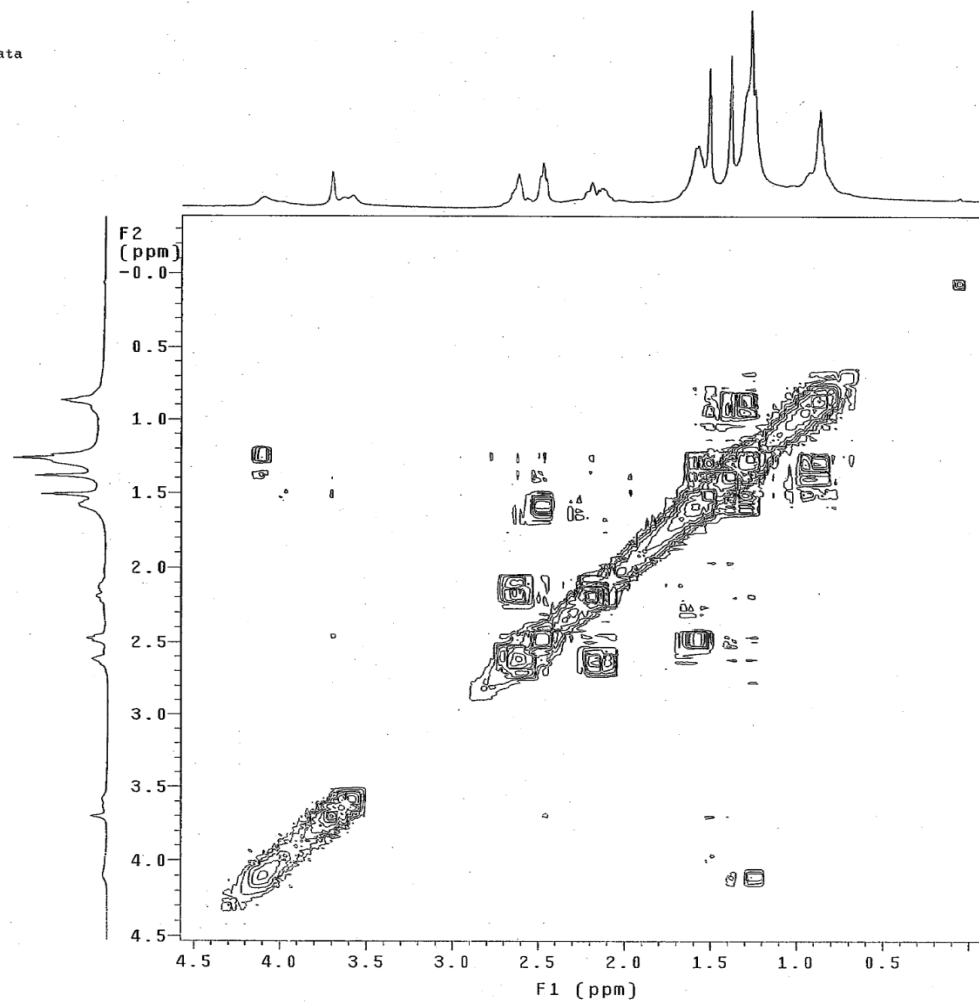
Partial expansions of the 2D-HMBC spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDC13  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
8 repetitions  
128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 21 min, 55 sec

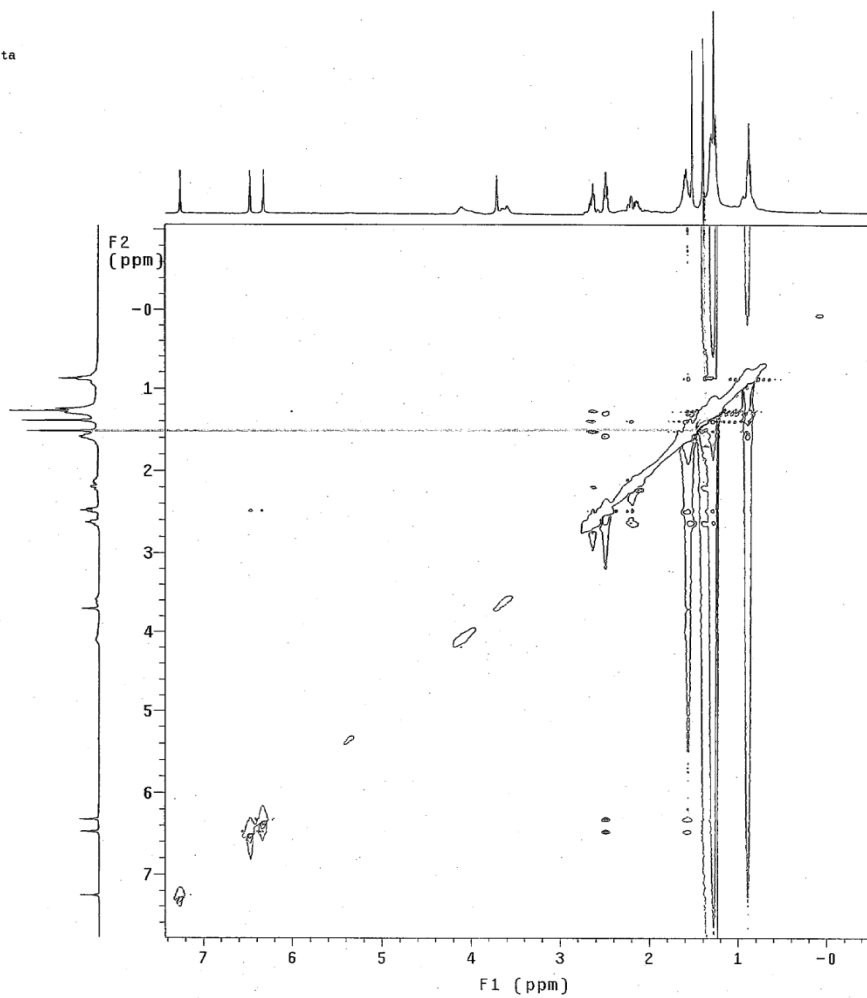


Partial expansions of the 2D-COSY spectrum of compound 8

SAA-60-7-3

Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: ROESY

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Mercury-4005B "unknown"Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.180 sec  
Width 8410.3 Hz  
2D Width 8410.3 Hz  
22 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
FT size 2048 x 2048  
Total time 0 min, 0 sec

2D-ROESY spectrum of compound 8

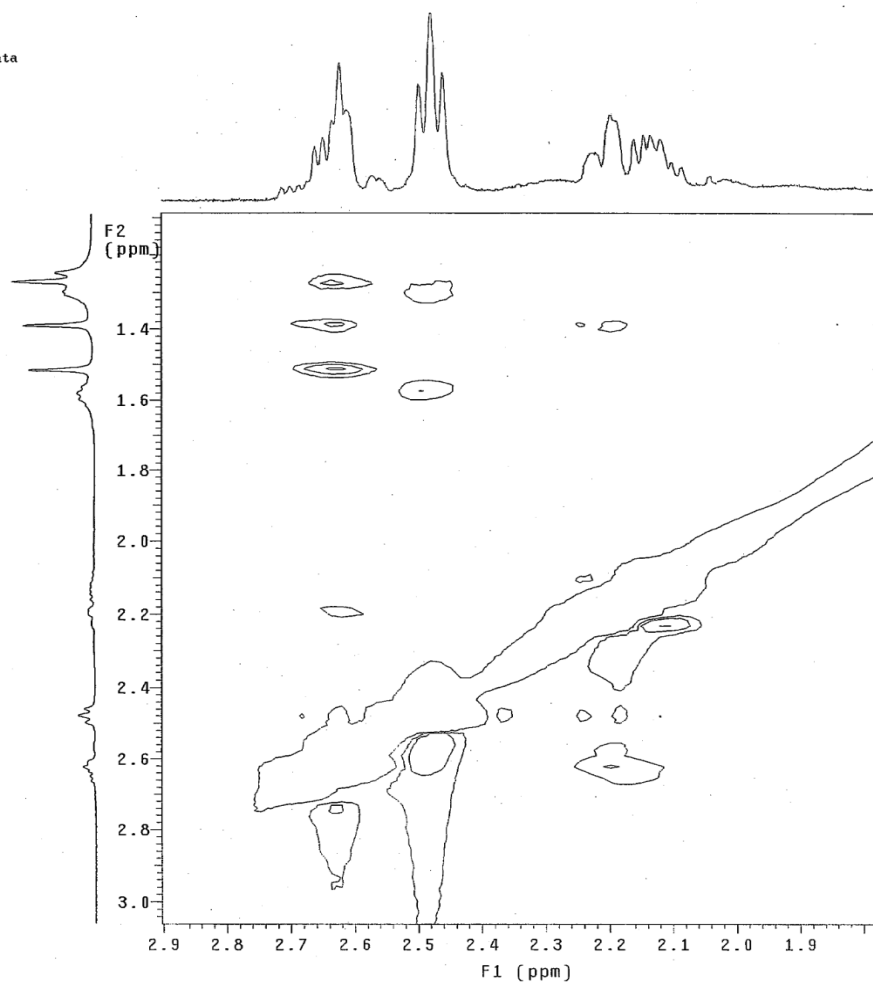


SAA-60-7-3

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

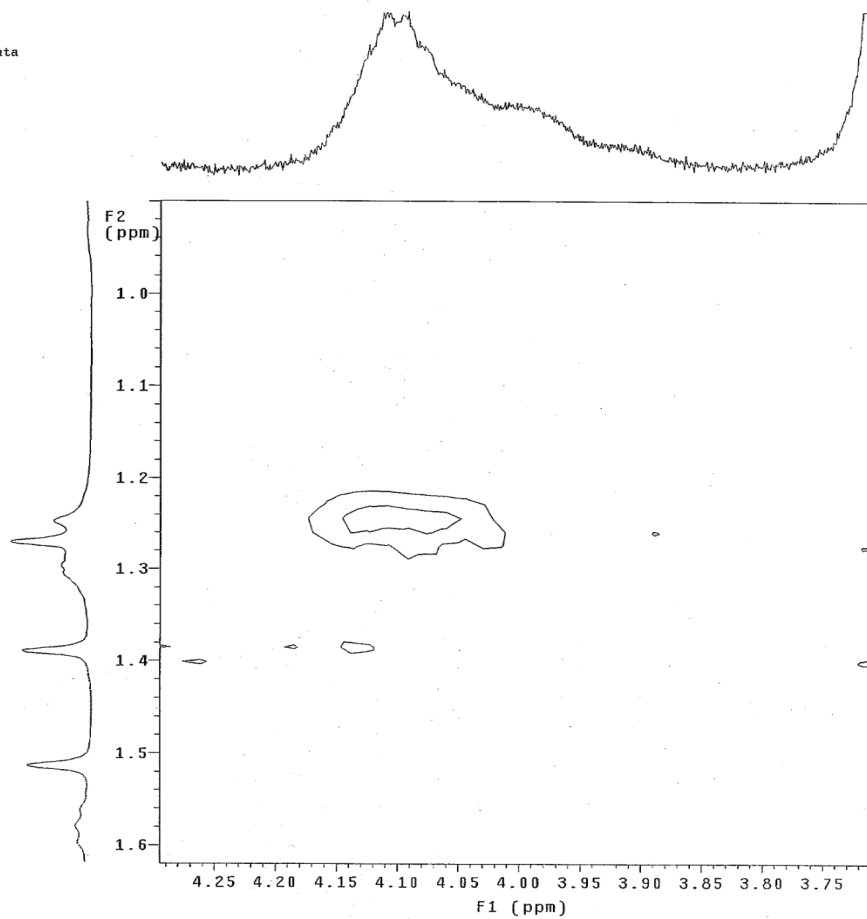
Pulse Sequence: ROESY  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
22 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
F1 size 2048 x 2048  
Total time 3 hr, 13 min, 31 sec



Partial expansions of the 2D-ROESY spectrum of compound 8

SAA-60-7-3  
Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON  
Pulse Sequence: ROESY  
Solvent: CDCl3  
Ambient temperature  
Mercury-400BB "unknown"  
Relax. delay 1.000 sec  
Mixing 0.800 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
22 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.029 sec  
FT size 2048 x 2048  
Total time 3 hr, 13 min, 31 sec



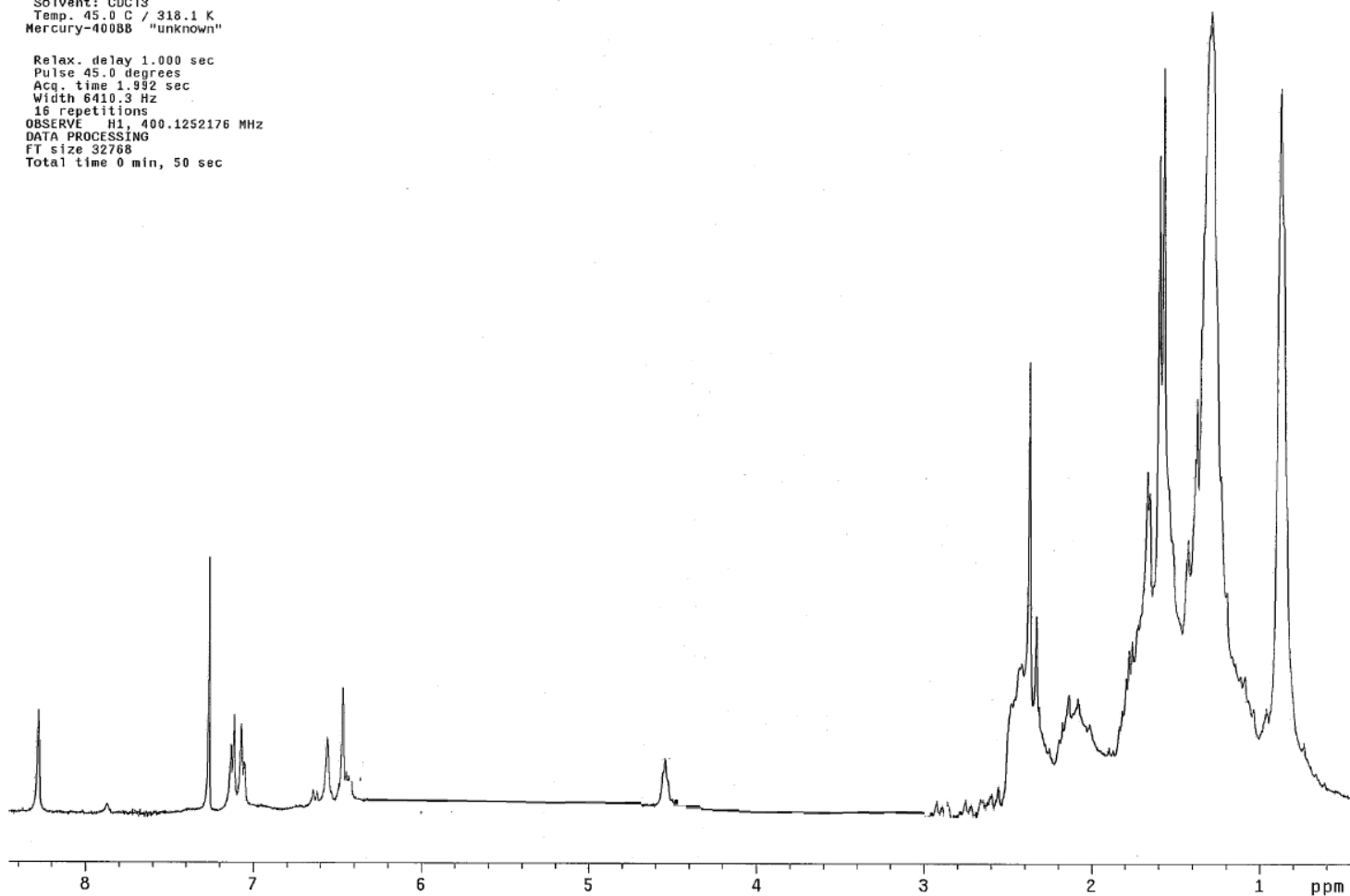
Partial expansions of the 2D-ROESY spectrum of compound 8

STANDARD 1H OBSERVE

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: s2pu1  
Solvent: CDCl3  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.992 sec  
Width 6410.3 Hz  
16 repetitions  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 50 sec



**<sup>1</sup>H-NMR spectrum of compound 9 in (CDCl<sub>3</sub>, 400 MHz)**

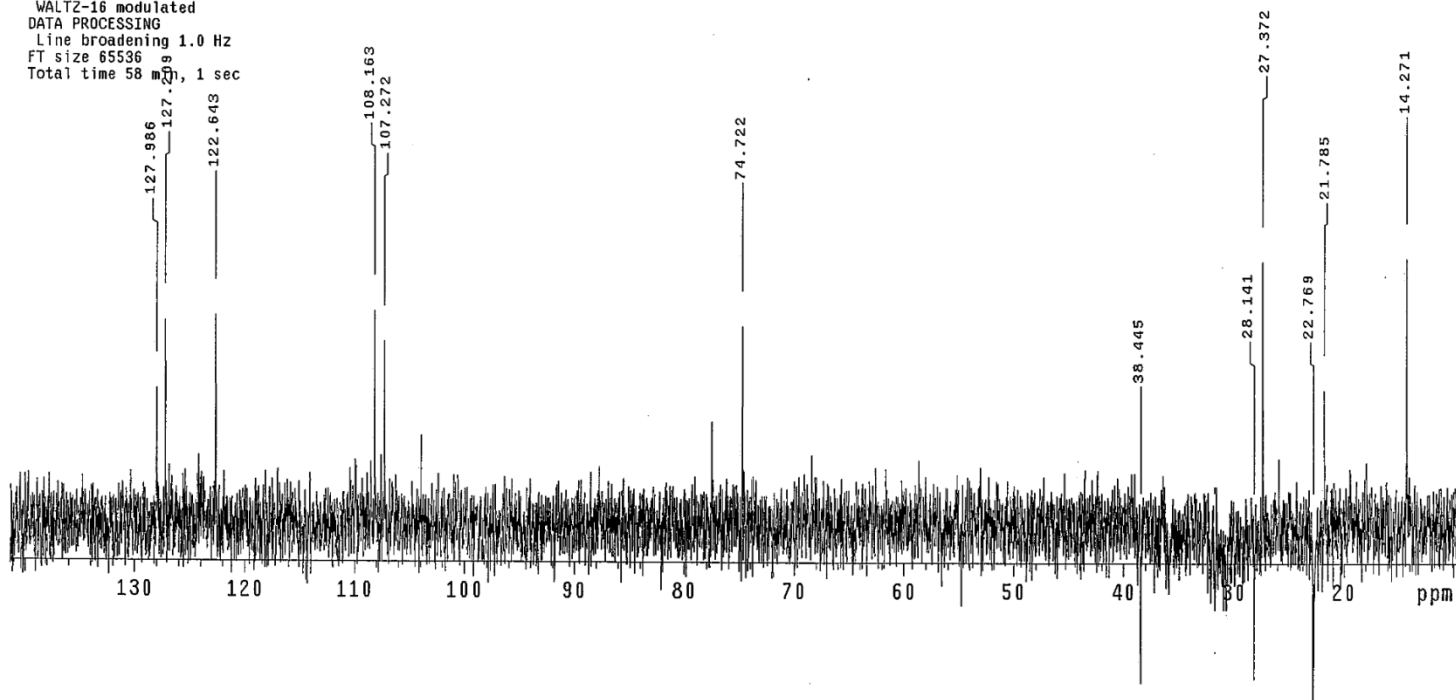
SAAB-II-3-6.2

Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: CARBON

Pulse Sequence: DEPT

Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Pulse 90.0 degrees  
Acq. time 1.199 sec  
Width 25125.6 Hz  
1088 repetitions  
OBSERVE C13, 100.6115420 MHz  
DECOUPLE H1, 400.1272240 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 58 min, 1 sec



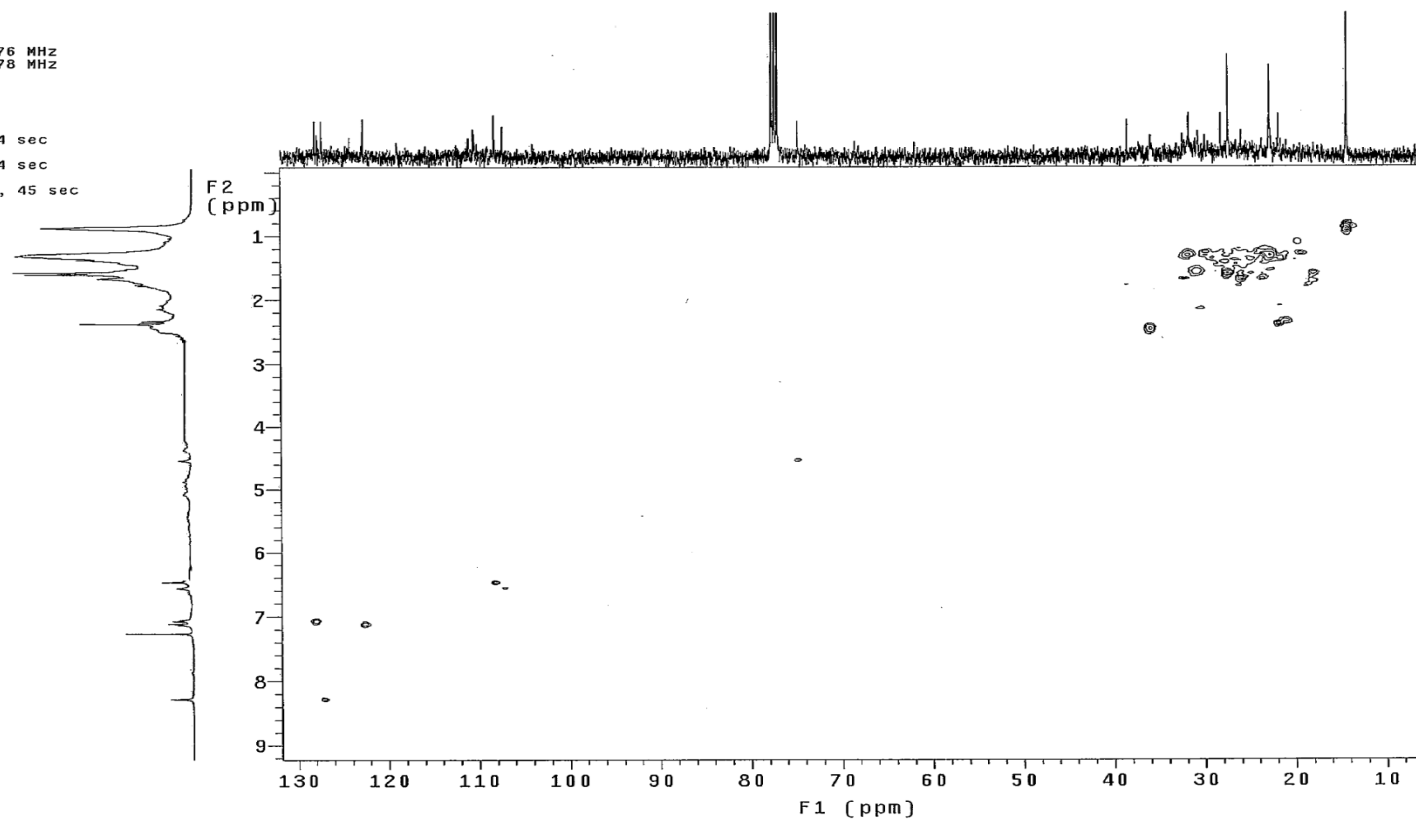
**DEPT spectrum of compound 9 in (CDCl<sub>3</sub>, 100 MHz)**

SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



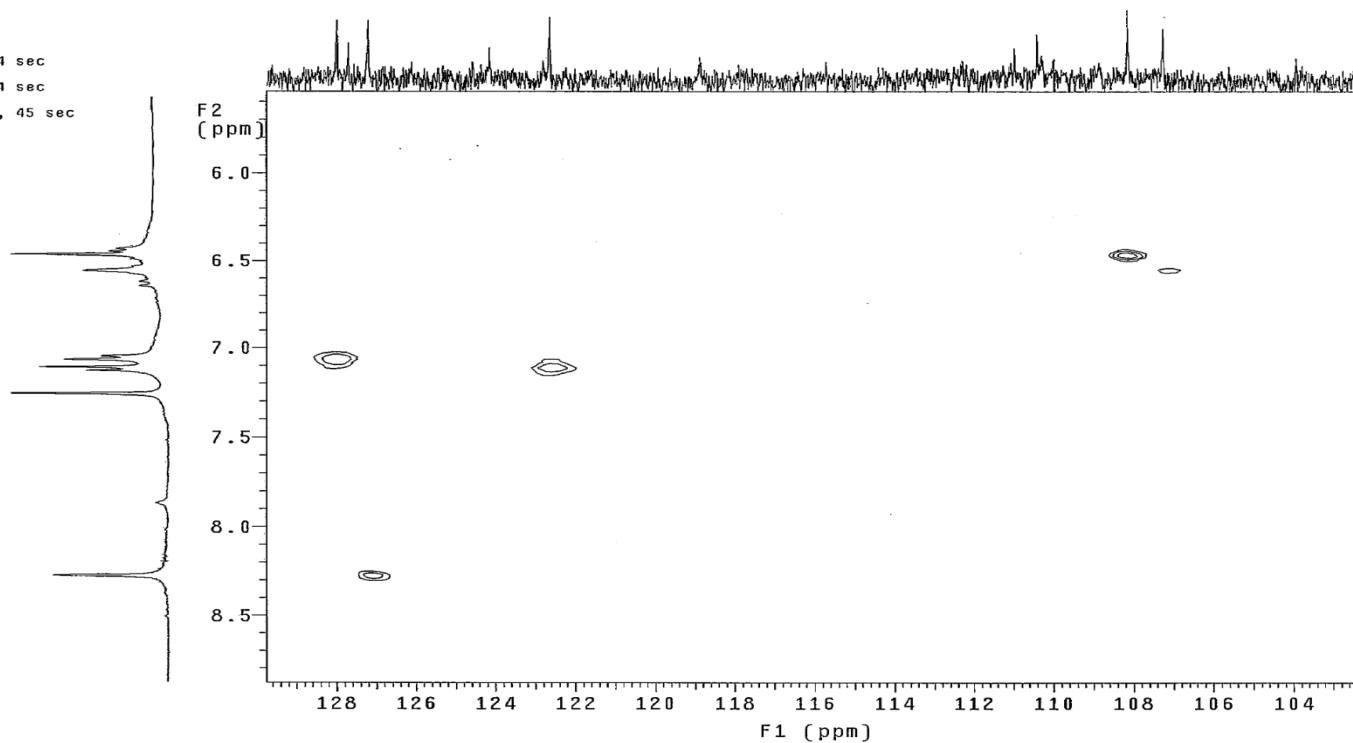
2D-HMQC spectrum of compound 9

SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC  
Solvent: CDCl3  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190876 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



Partial expansions of the 2D-HMQC spectrum of compound 9

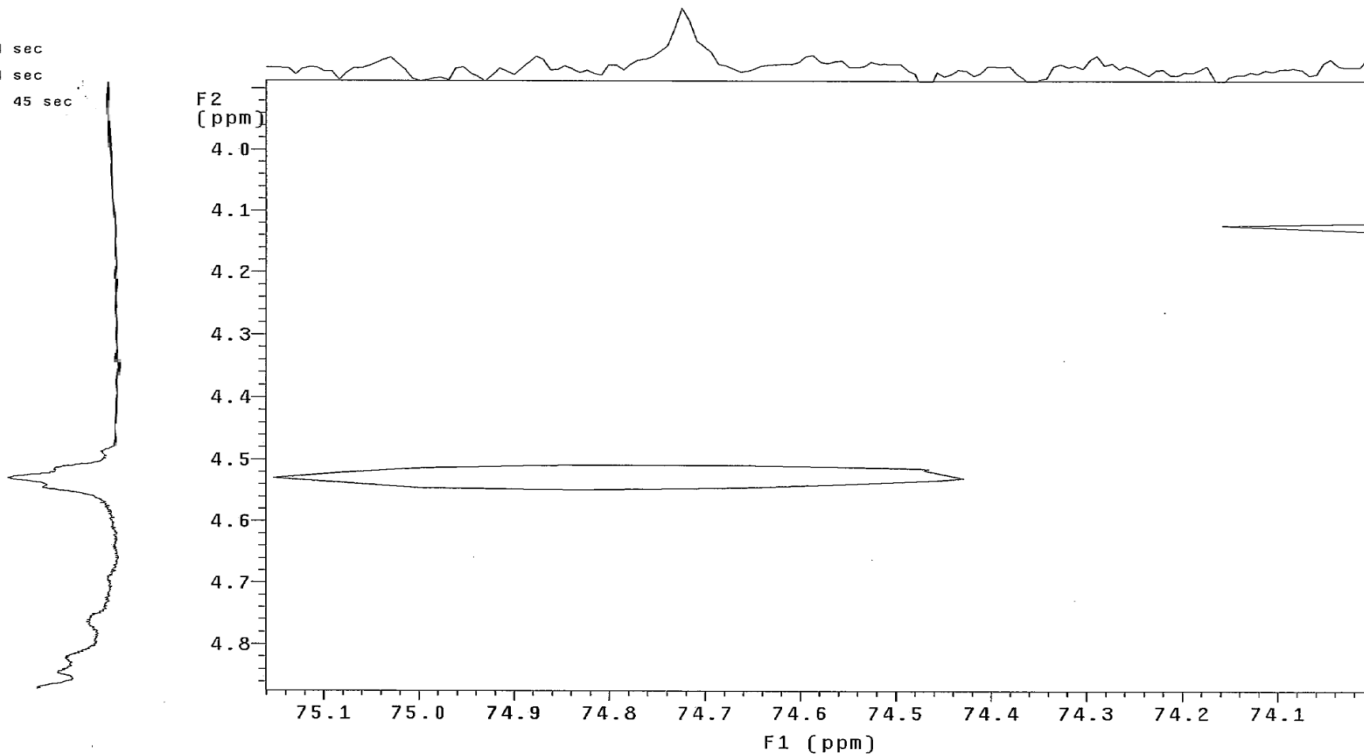
SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMQC

Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
F1 size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



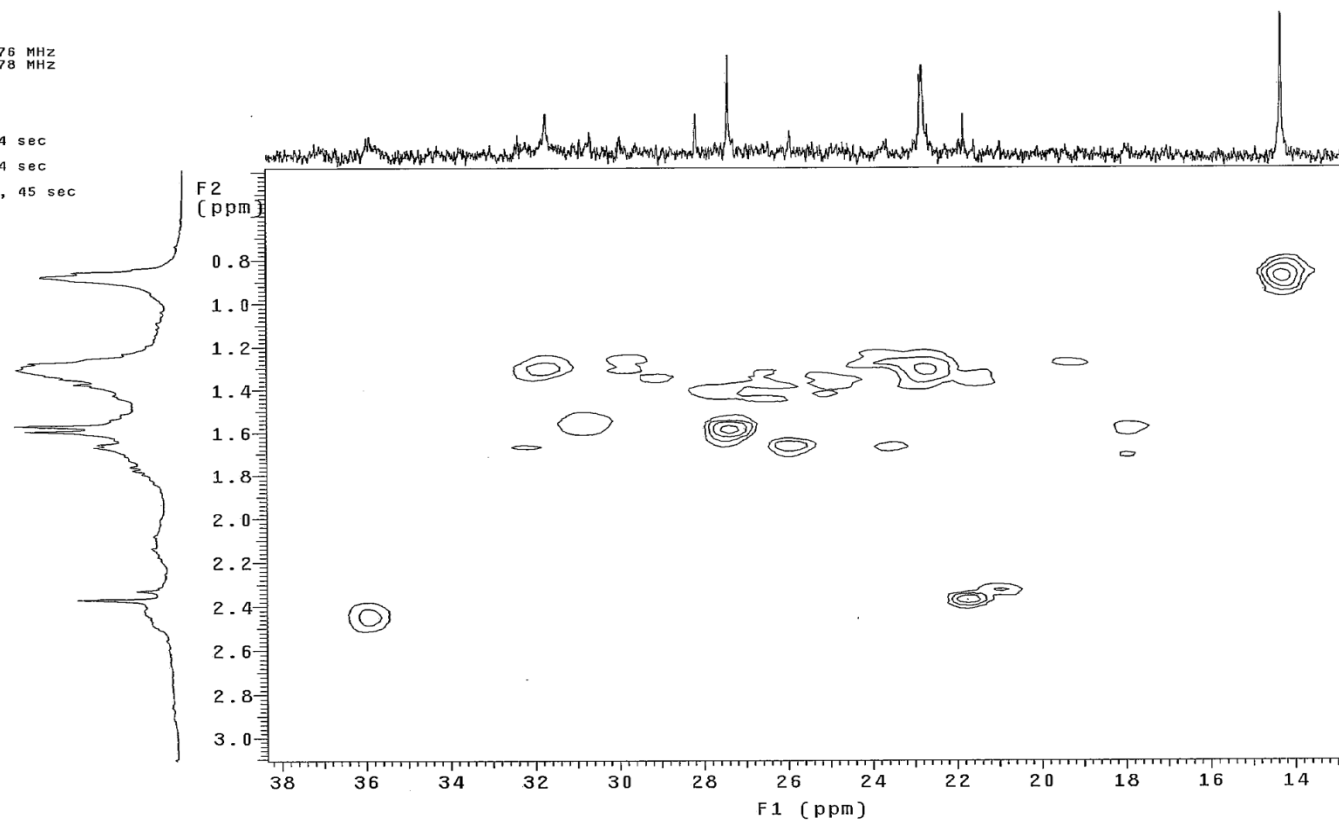
Partial expansions of the 2D-HMQC spectrum of compound 9

SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMOC  
Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 17094.0 Hz  
32 repetitions  
2 x 128 increments  
OBSERVE H1, 400.1252176 MHz  
DECOUPLE C13, 100.6190878 MHz  
Power 41 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.014 sec  
FT size 2048 x 2048  
Total time 2 hr, 52 min, 45 sec



Partial expansions of the 2D-HMOC spectrum of compound 9

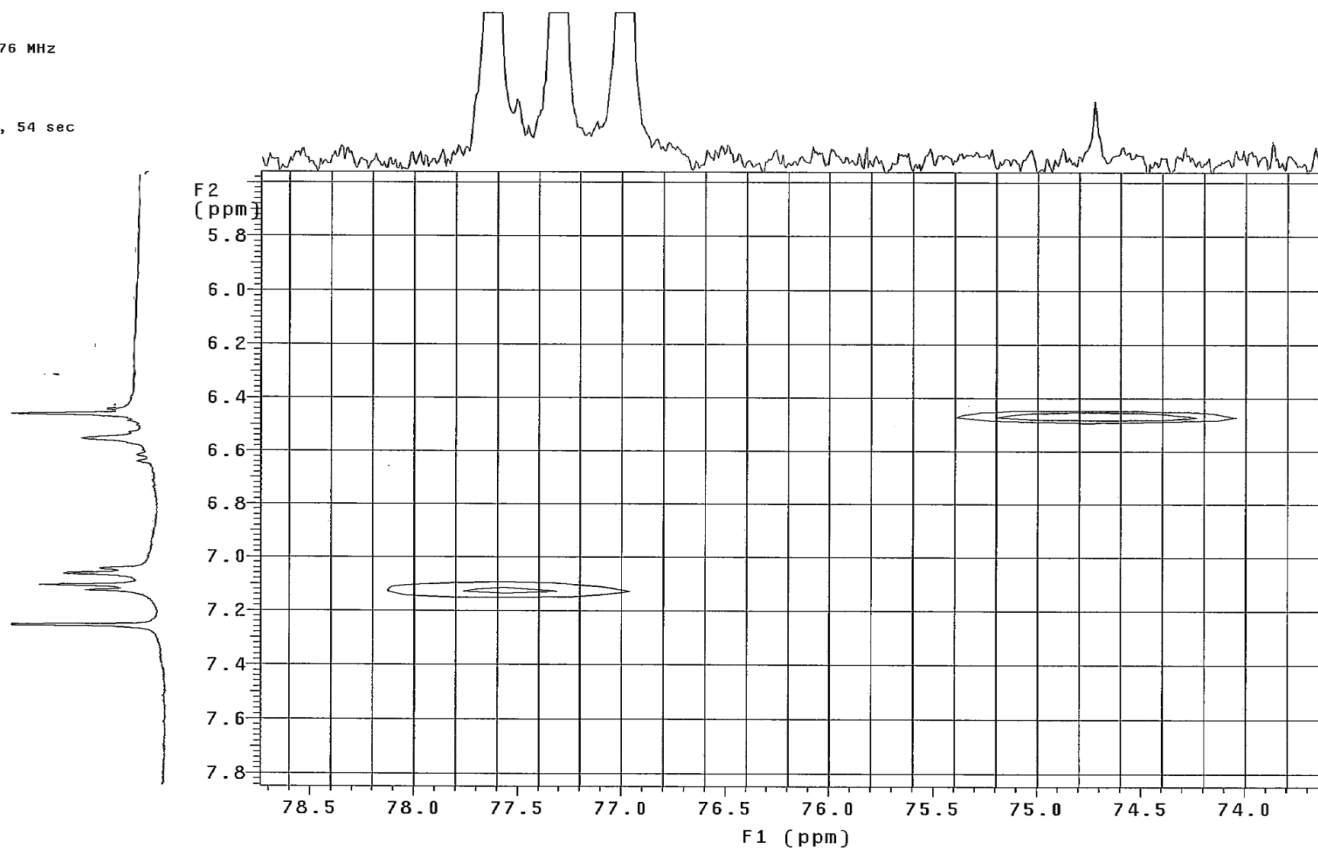


SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrSYS/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC  
Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
60 repetitions  
400 increments  
OBSERVE H1, 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 8 hr, 40 min, 54 sec



2D-HMBC spectrum of compound 9

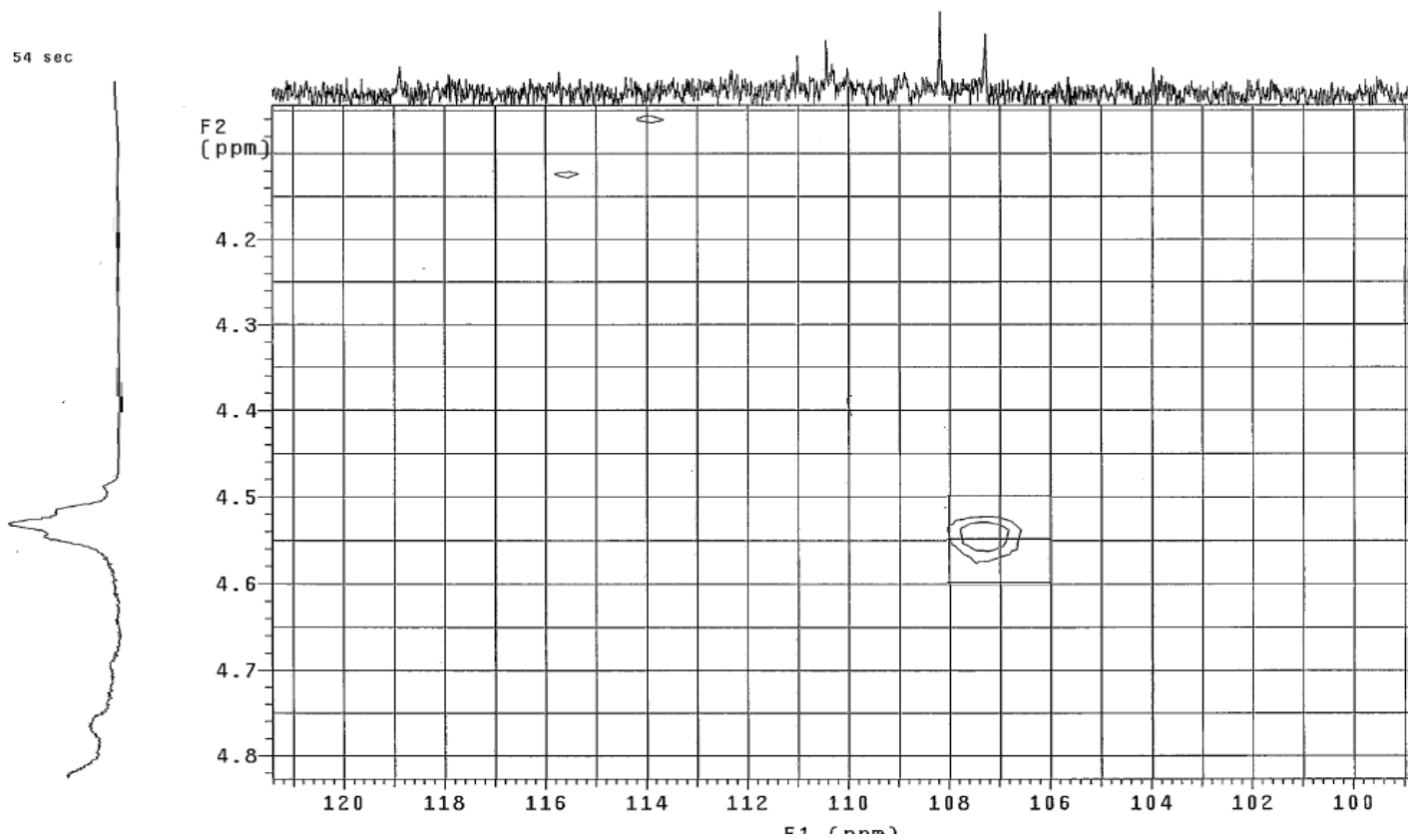
SAAB-II-6.5

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gHMBC

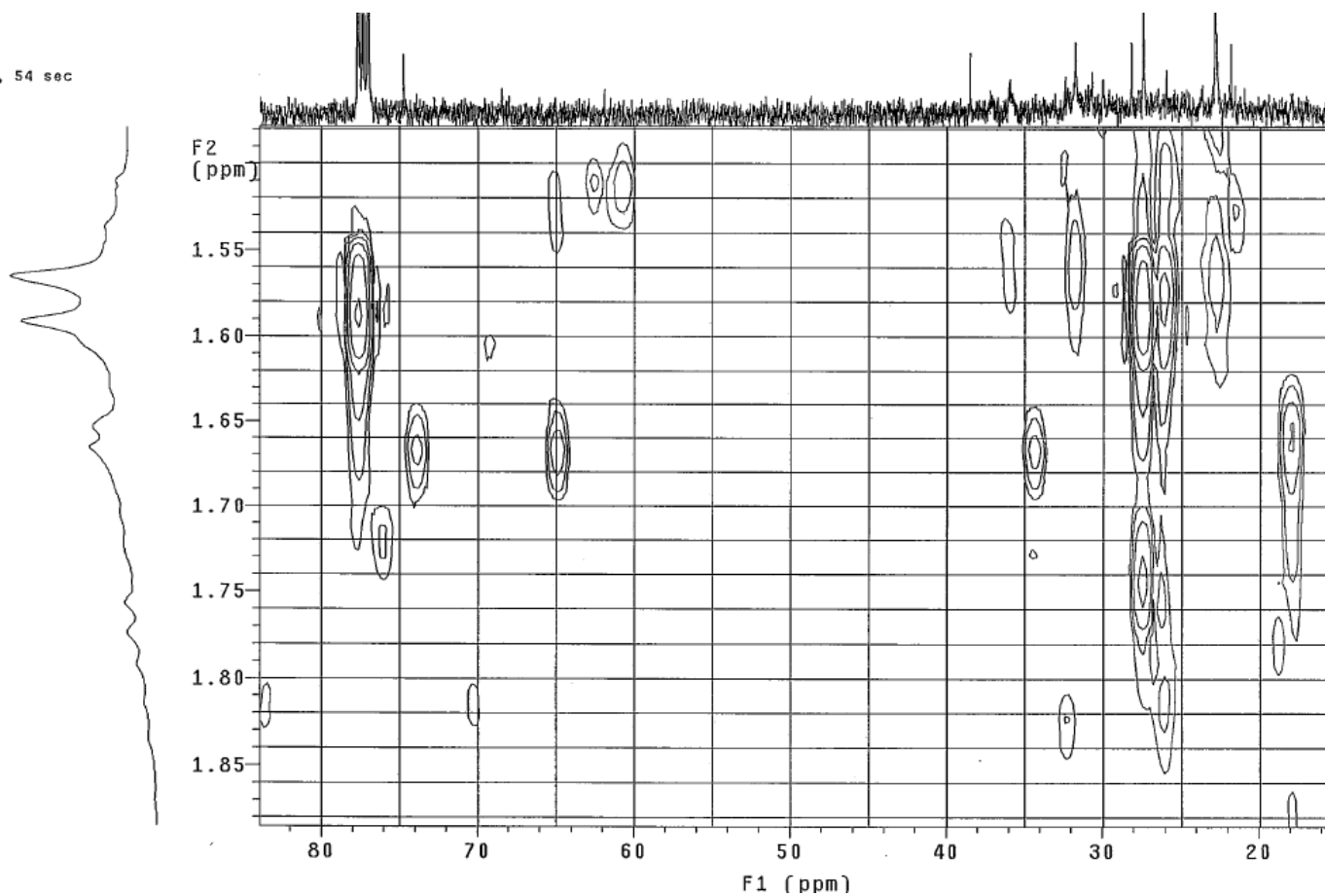
Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 24154.6 Hz  
60 repetitions  
400 increments  
OBSERVE H1 400.1252176 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 8 hr, 40 min, 54 sec



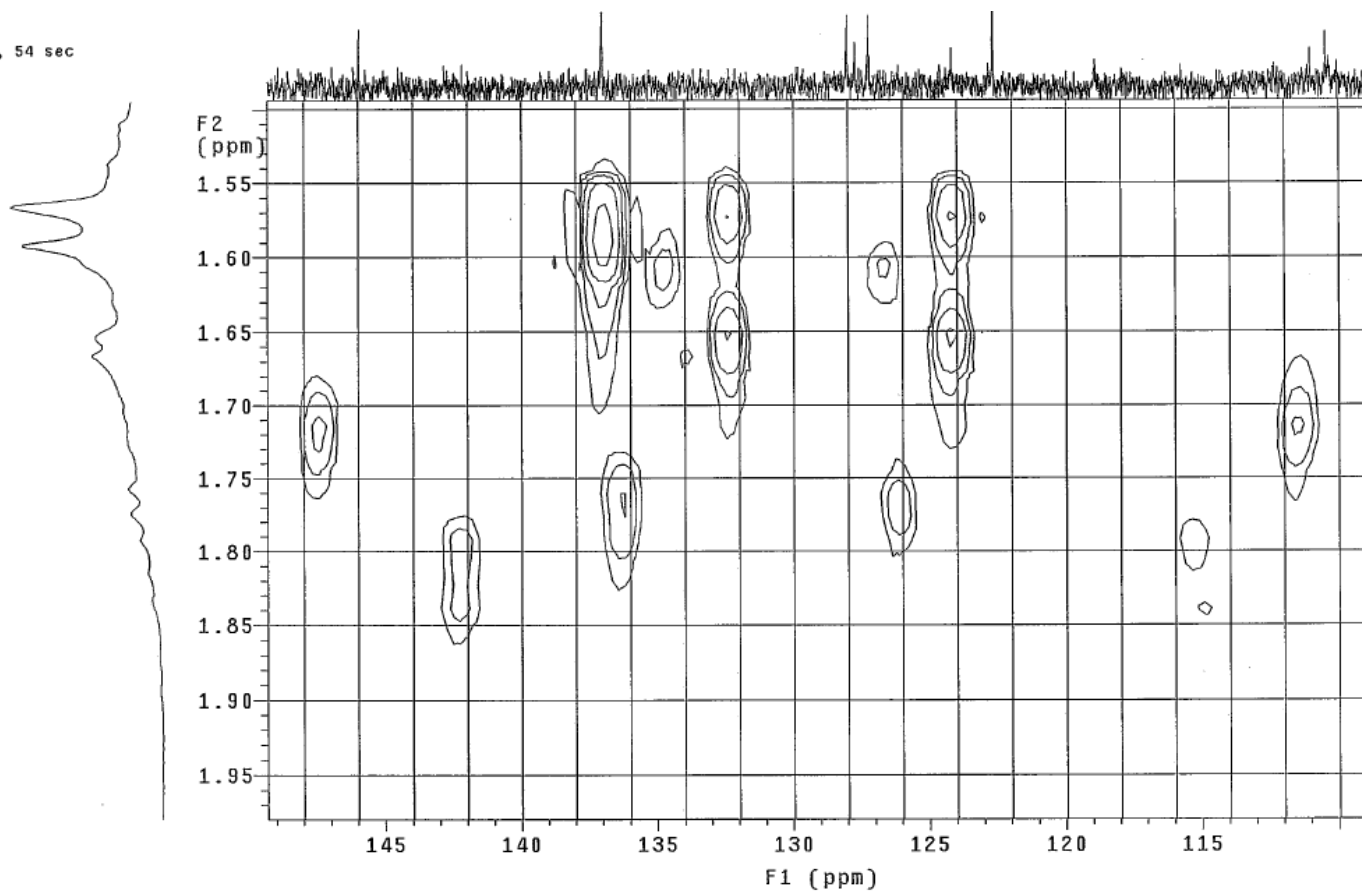
Partial expansions of the 2D-HMBC spectrum of compound 9

Sine bell 0.000 sec  
F1 DATA PROCESSING  
Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 8 hr, 40 min, 54 sec

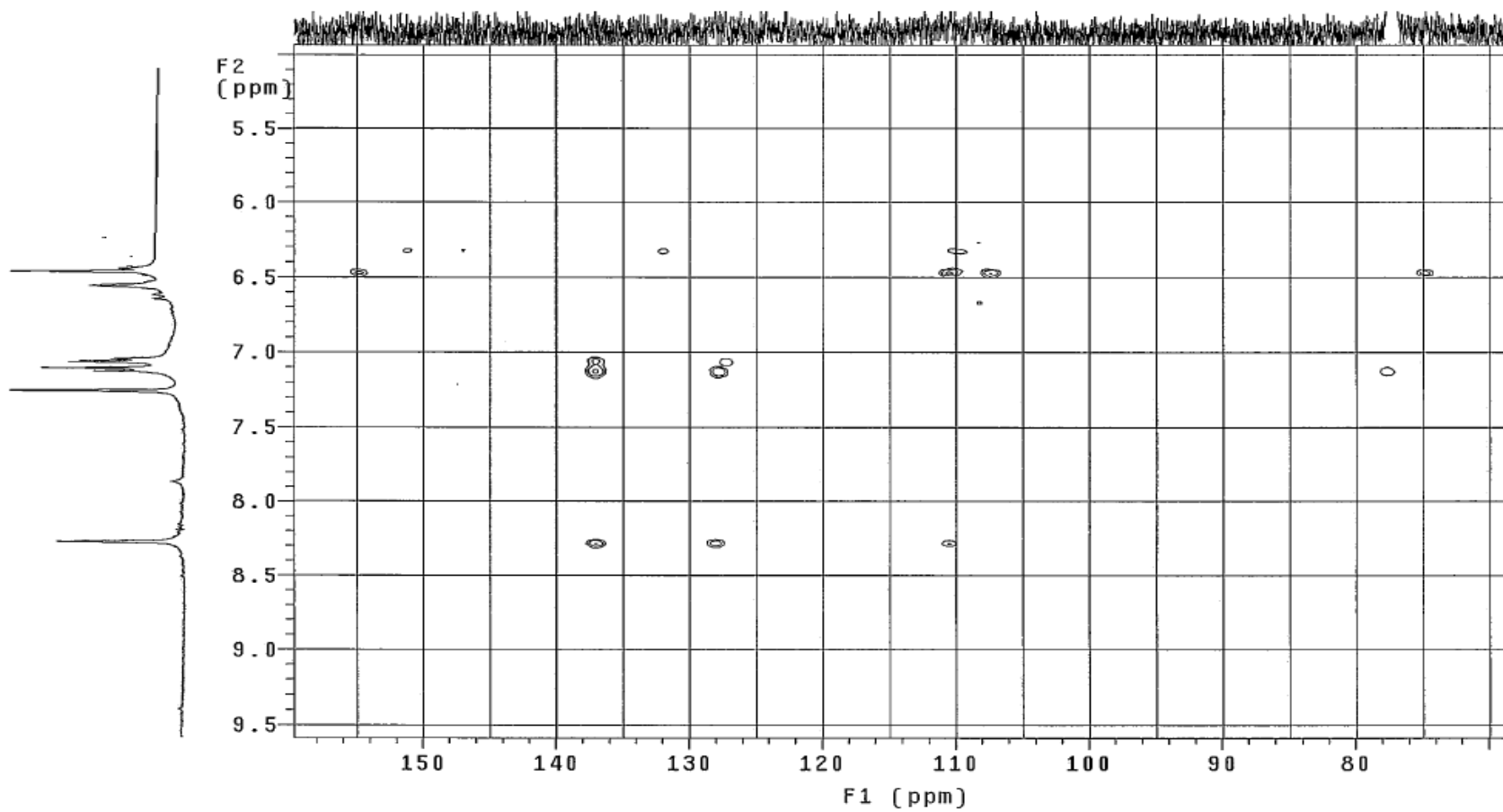


Partial expansions of the 2D-HMBC spectrum of compound 9

Sine bell 0.008 sec  
FT size 2048 x 2048  
Total time 8 hr, 40 min, 54 sec



Partial expansions of the 2D-HMBC spectrum of compound 9



Partial expansions of the 2D-HMBC spectrum of compound 9

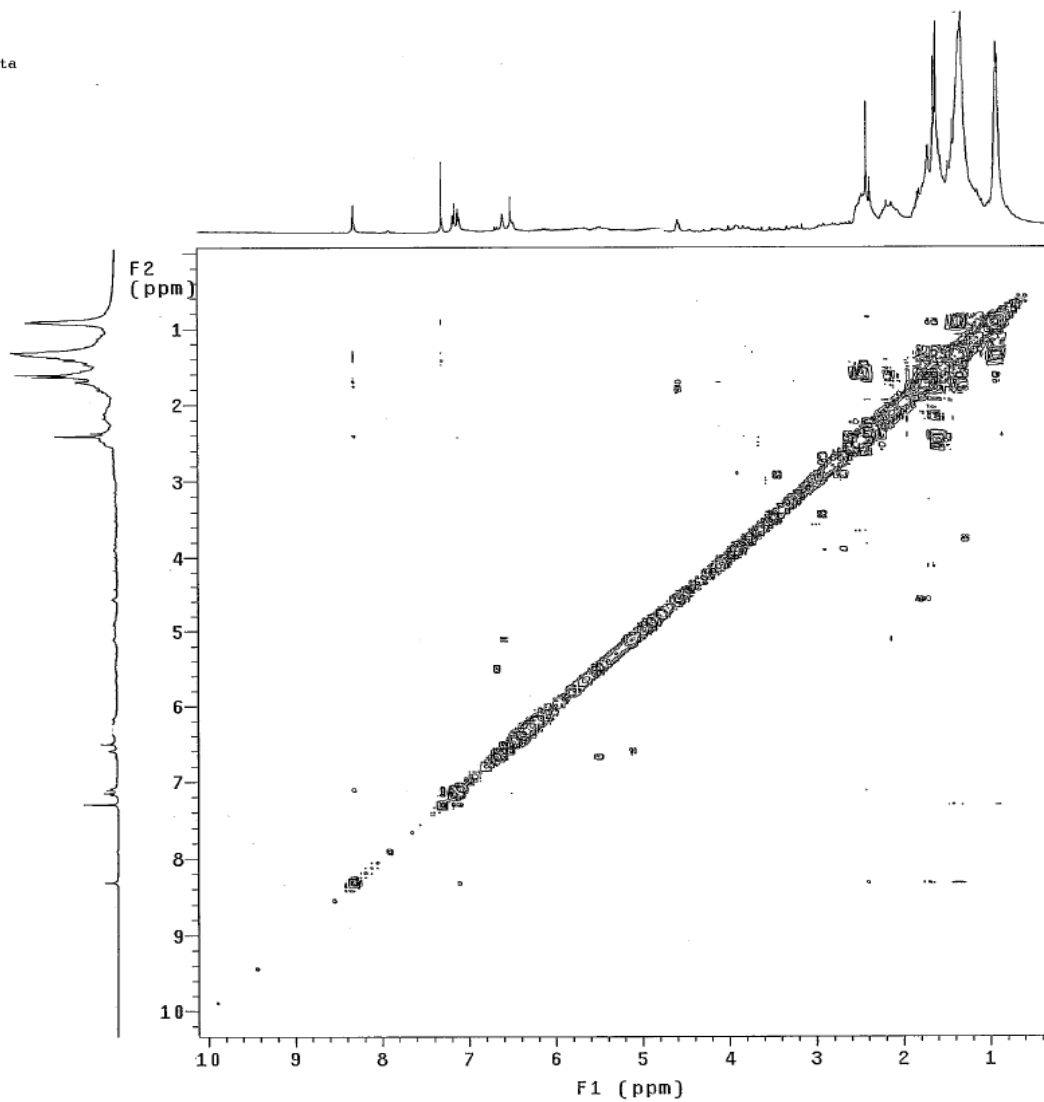
SAAB-II-3-6.2

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDC13  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
4 repetitions  
128 increments  
OBSERVE H1, 400.1252038 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 11 min, 33 sec



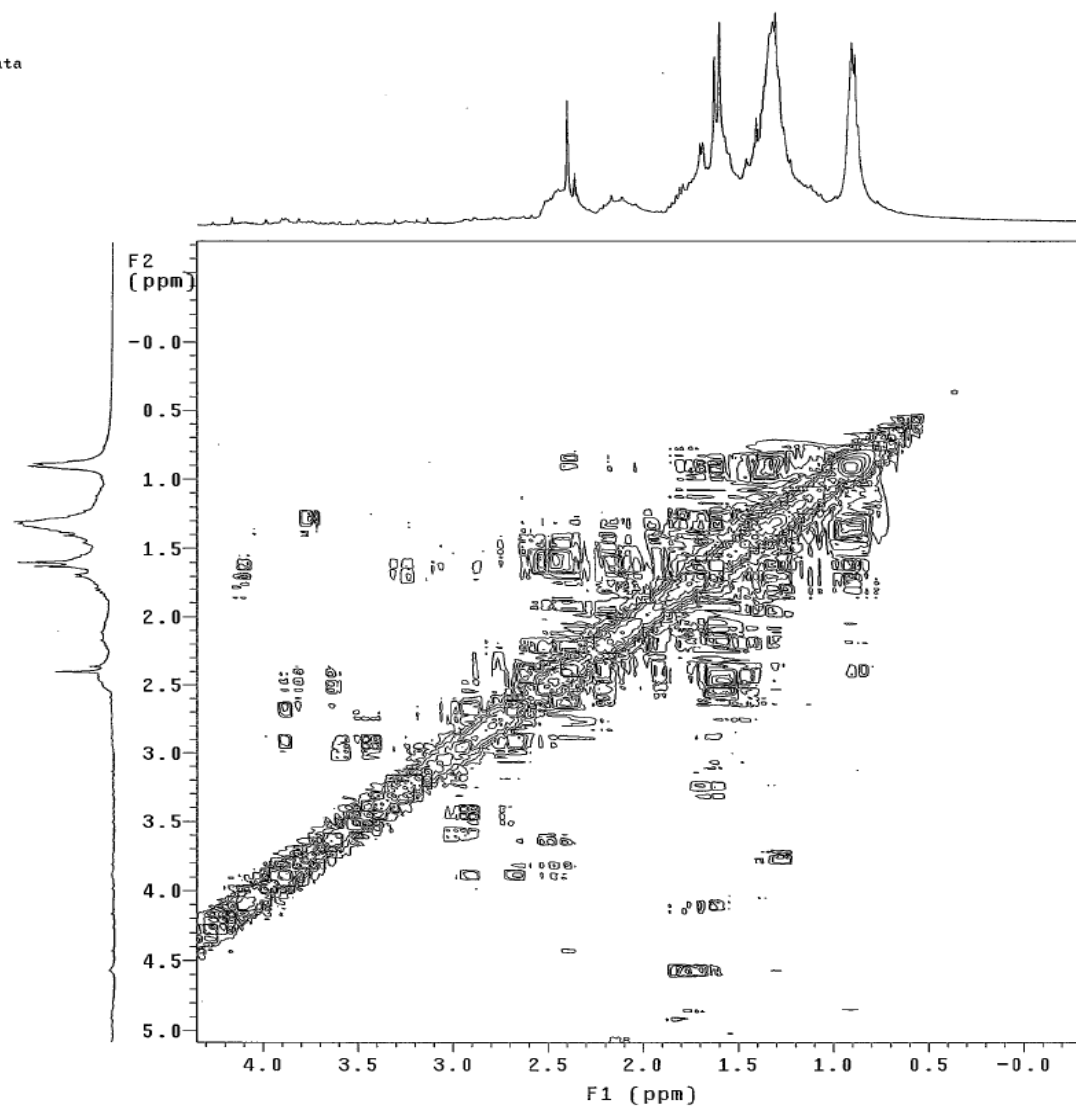
2D-COSY spectrum of compound 9

SAAB-II-3-6.2

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY  
Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
4 repetitions  
128 increments  
OBSERVE H1, 400.1252038 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 11 min, 33 sec



Partial expansions of the 2D-COSY spectrum of compound 9

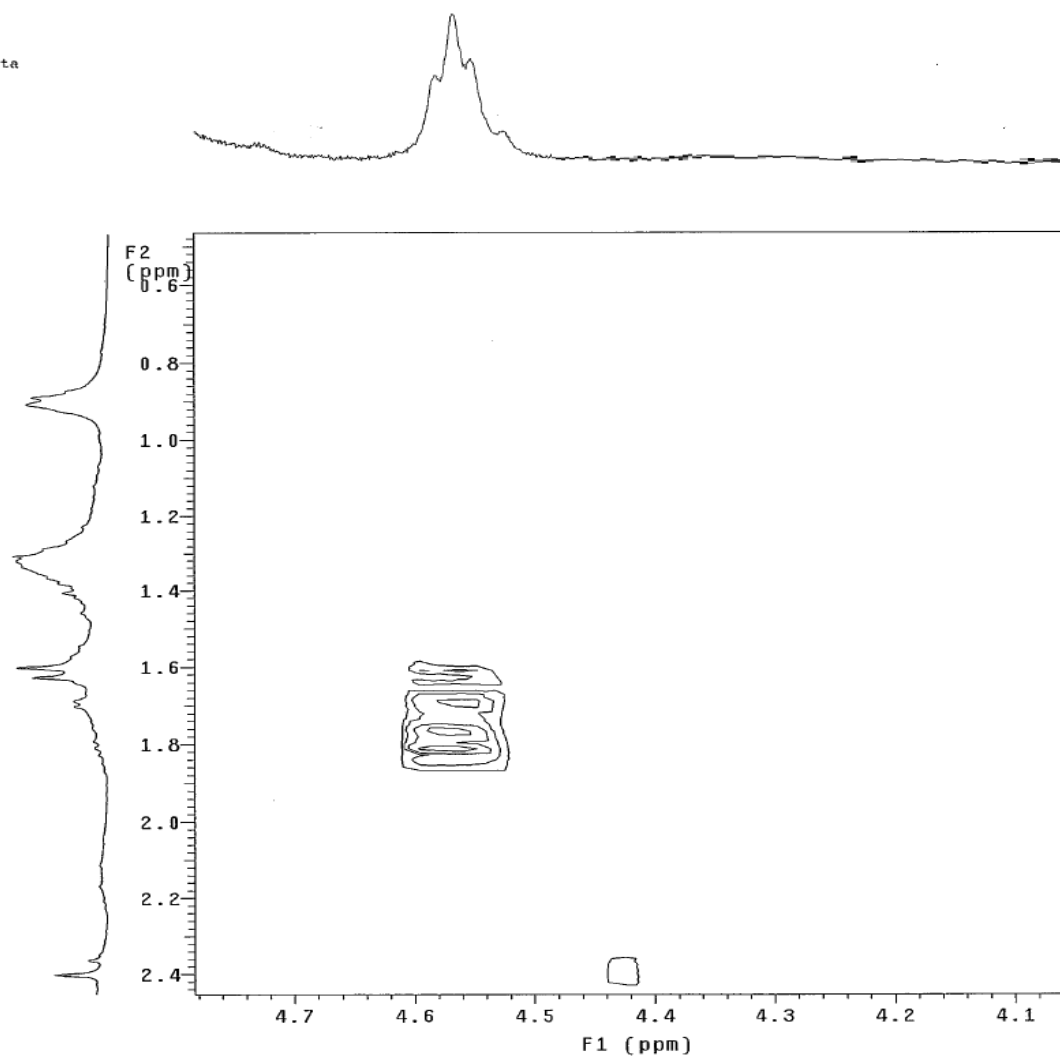
SAAB-II-3-6.2

Archive directory: /export/home/desmond/vnmrsys/data  
Sample directory:  
File: PROTON

Pulse Sequence: gCOSY

Solvent: CDCl<sub>3</sub>  
Temp. 45.0 C / 318.1 K  
Mercury-400BB "unknown"

Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
4 repetitions  
128 increments  
OBSERVE H1, 400.1252038 MHz  
DATA PROCESSING  
Sq. sine bell 0.080 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.020 sec  
FT size 2048 x 2048  
Total time 11 min, 33 sec



Partial expansions of the 2D-COSY spectrum of compound 9



