

## Supplementary Material

### NMR and X-ray characterization of steroids with furospirostane side chains

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Rafael Moreno-Esparza\* and Martín A. Iglesias-Arteaga\*

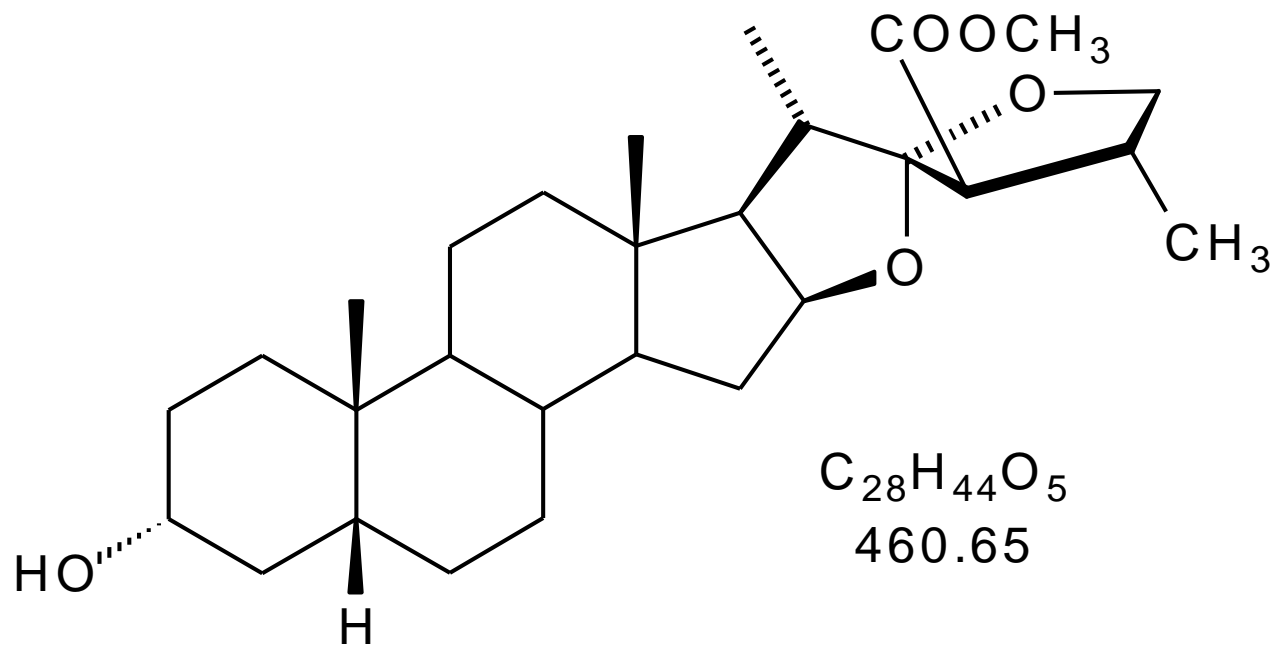
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*E-mails: [moresp@servidor.unam.mx](mailto:moresp@servidor.unam.mx), [martin.iglesias@servidor.unam.mx](mailto:martin.iglesias@servidor.unam.mx)*

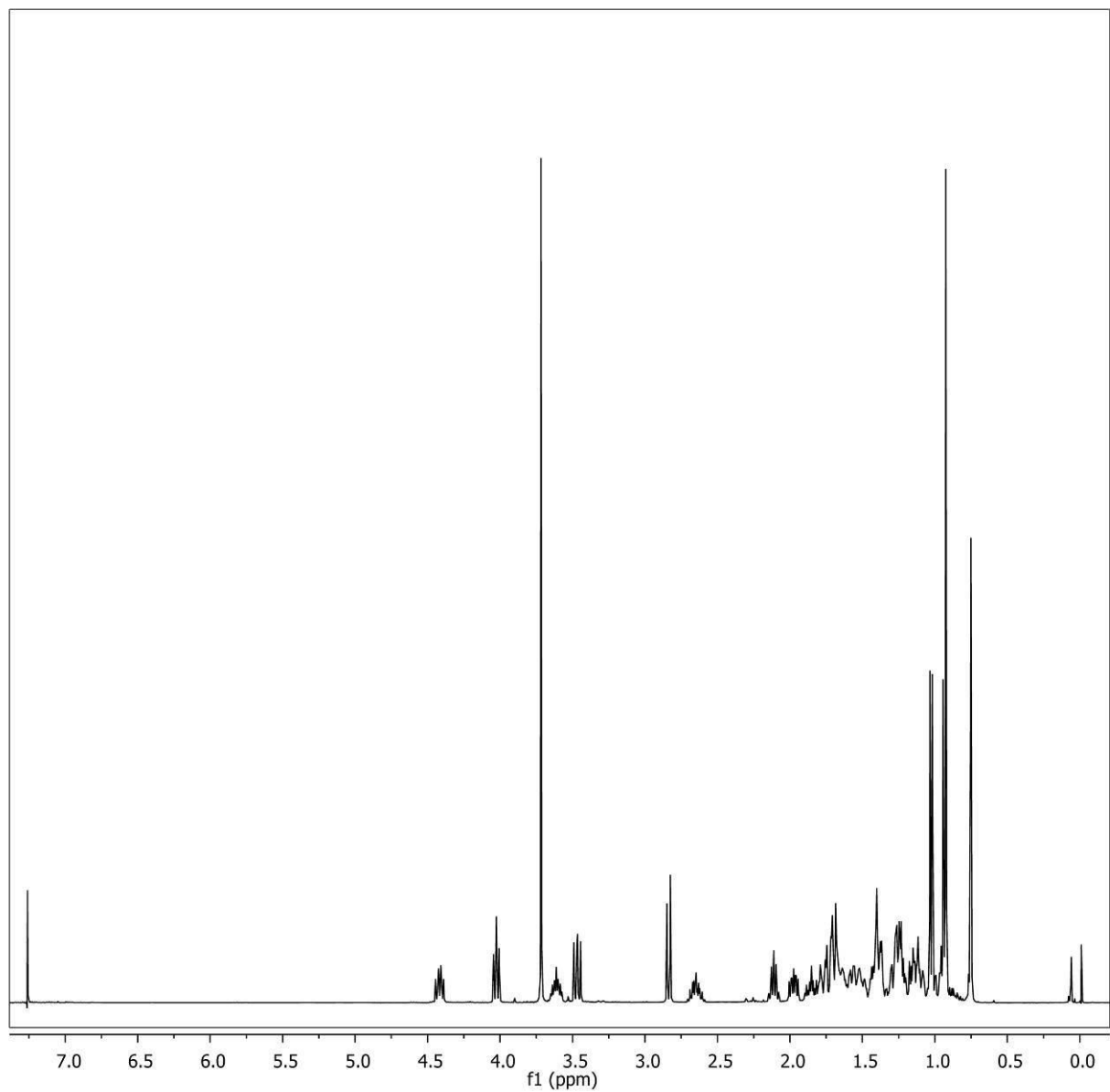
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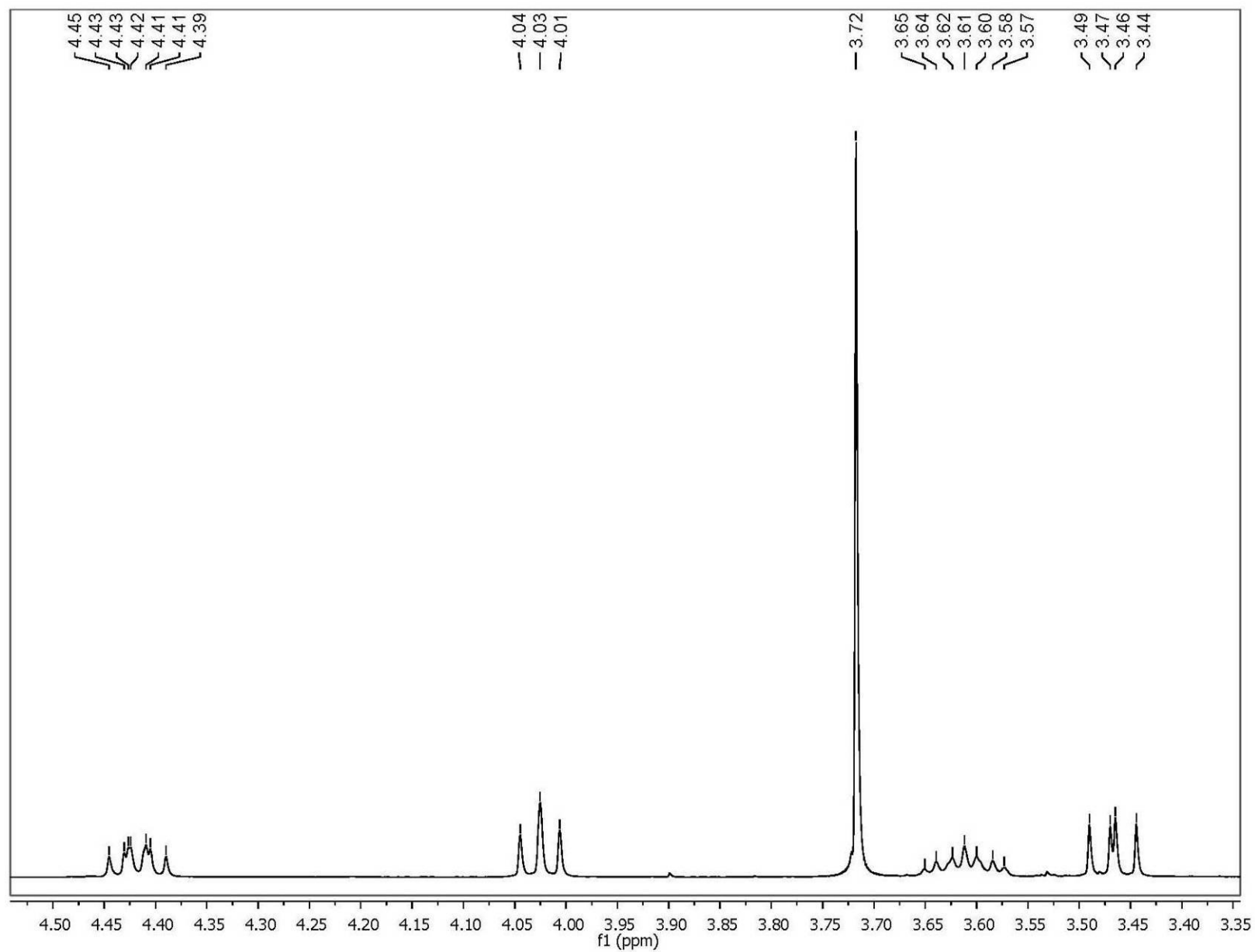
1.1 (2 <i>S</i> , 23 <i>R</i> , 24 <i>R</i> )-16 $\beta$ , 22:22, 25-diepoxi-23-carboxymethyl-24-methyl-26,27-dinor-5 $\beta$ -cholestan-3 $\alpha$ -ol ( <b>10</b> )	S1
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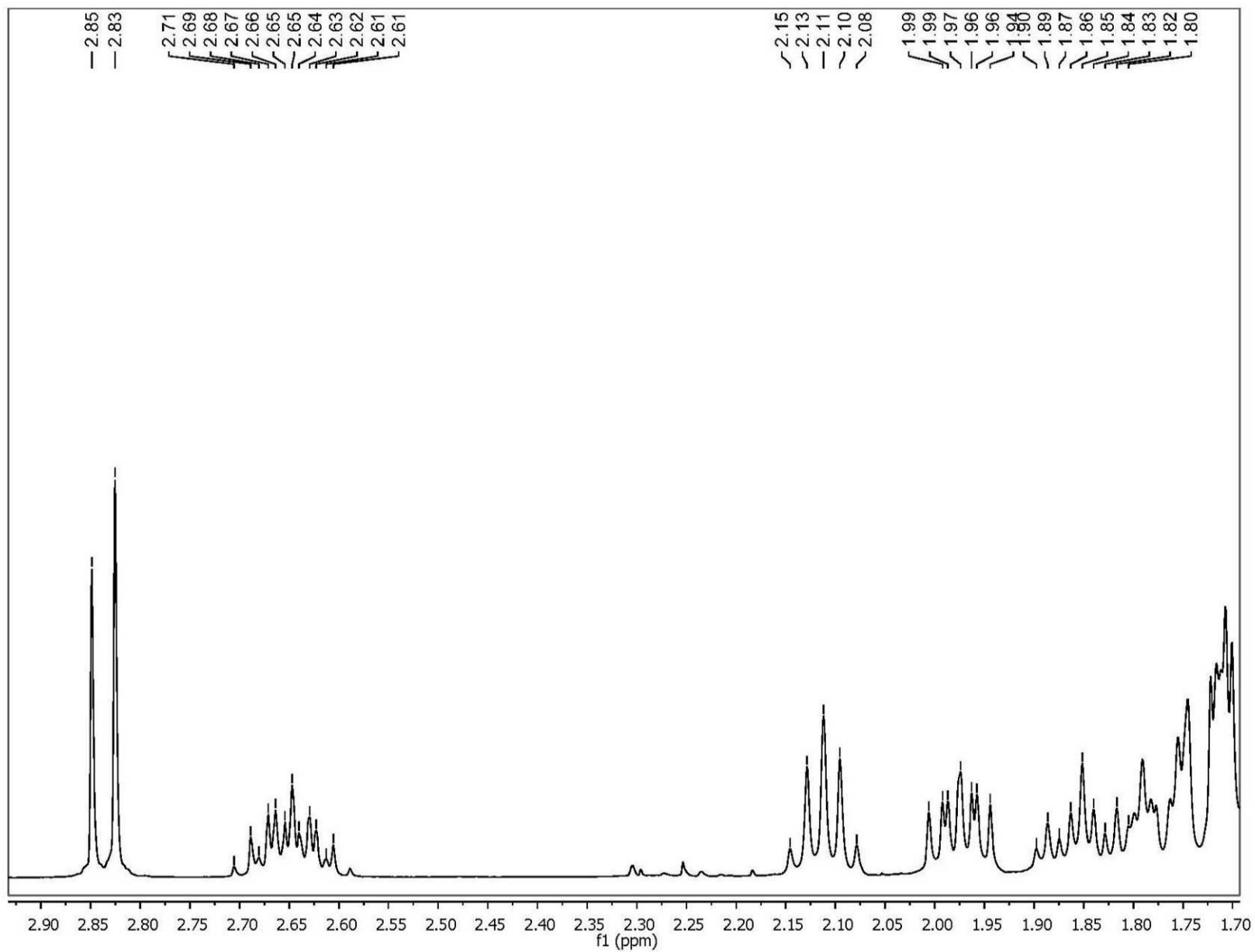


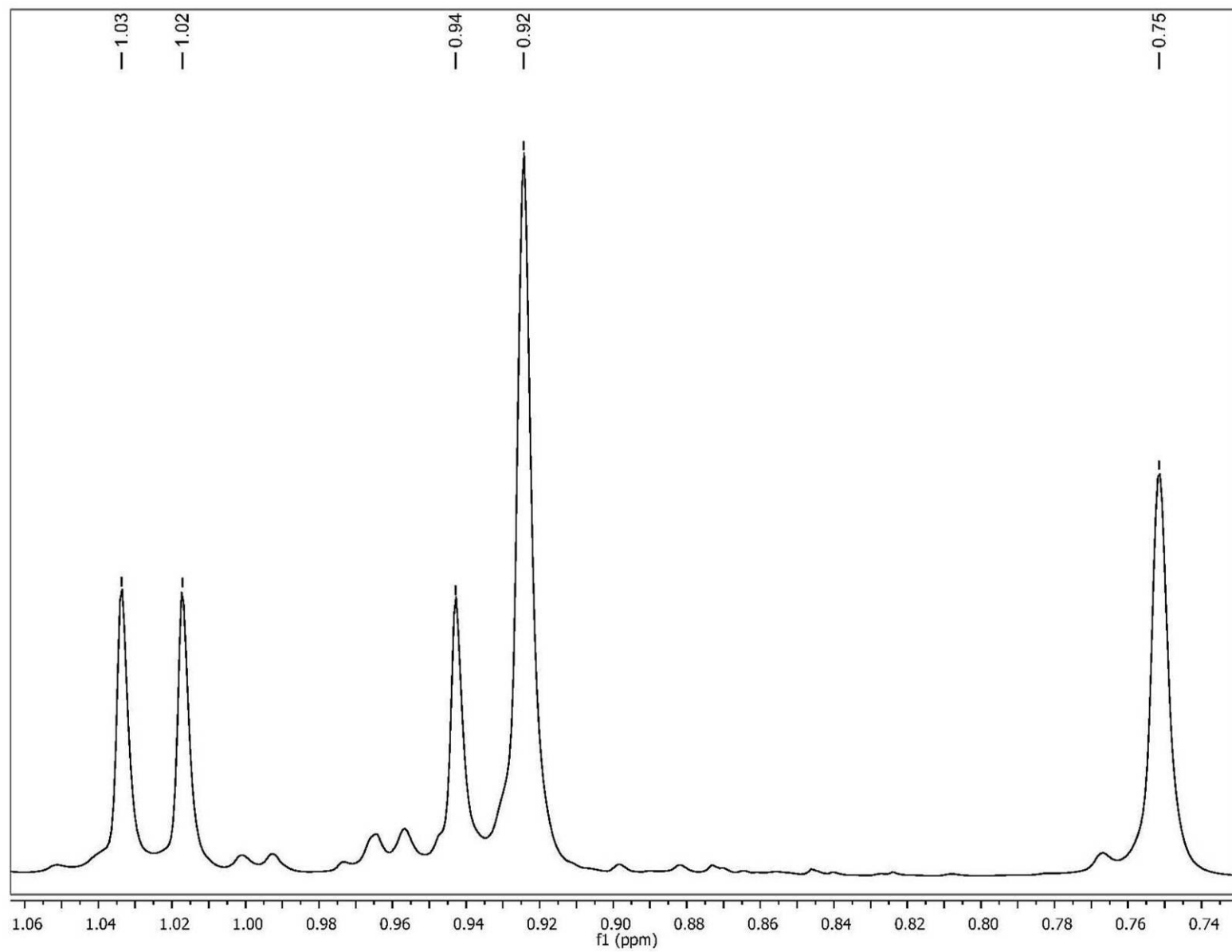
**(22*S*, 23*R*, 24*R*)-16 $\beta$ , 22:22, 25-diepoxi-23-carboxymethyl-24-methyl-26,27-dinor-5 $\beta$ -cholestan-3 $\alpha$ -ol (10)**

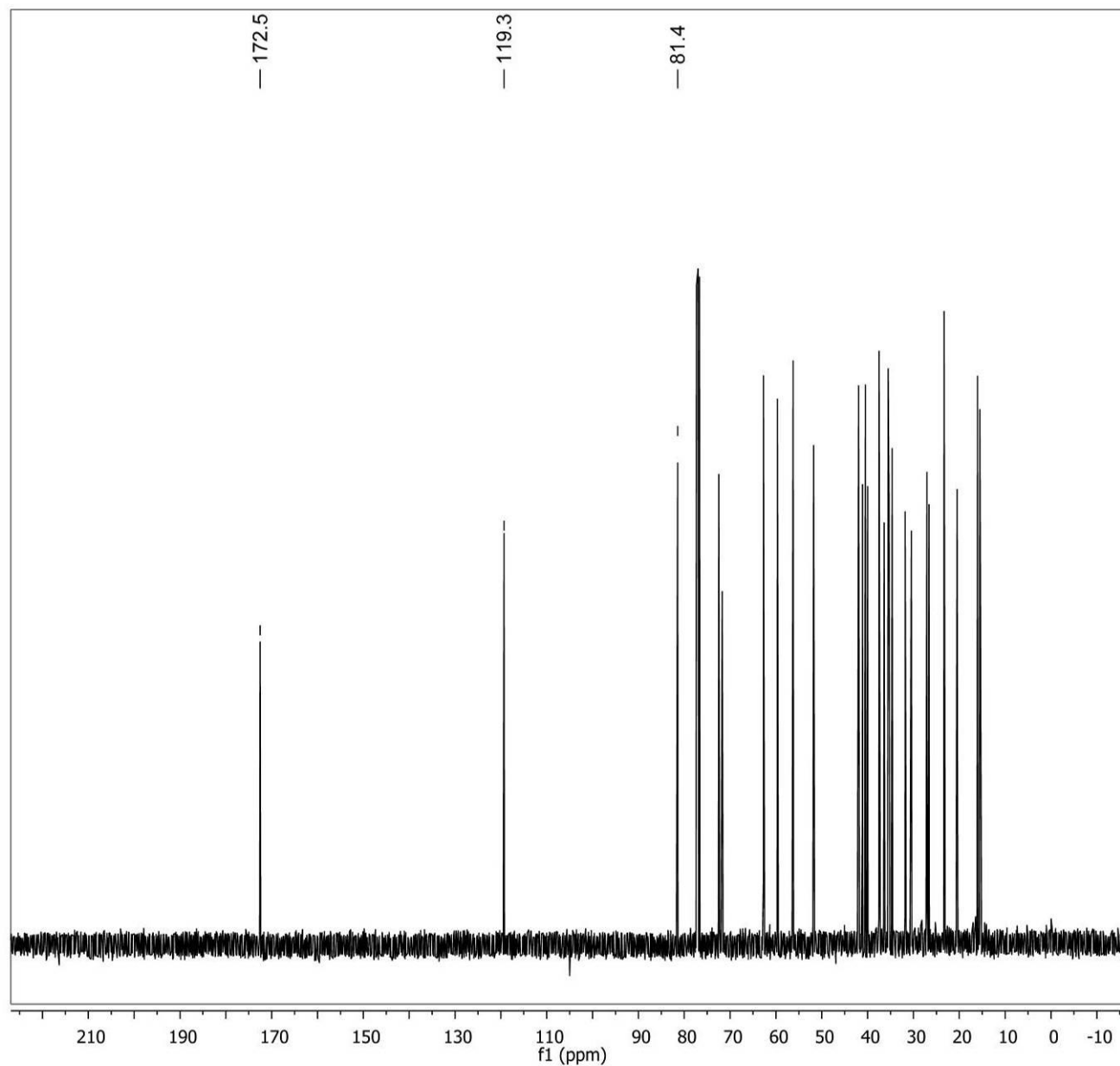


Parámetro	Valor
1 Comment	11398
	USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnms
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	s2pul
8 Number of Scans	16
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	2.0486
13 Acquisition Date	2010-04-10T0 1:32:12
14 Modification Date	2010-04-10T0 8:49:16
15 Spectrometer Frequency	399.72
16 Spectral Width	3787.9
17 Lowest Frequency	-467.1
18 Nucleus	1H
19 Acquired Size	7760
20 Spectral Size	16384

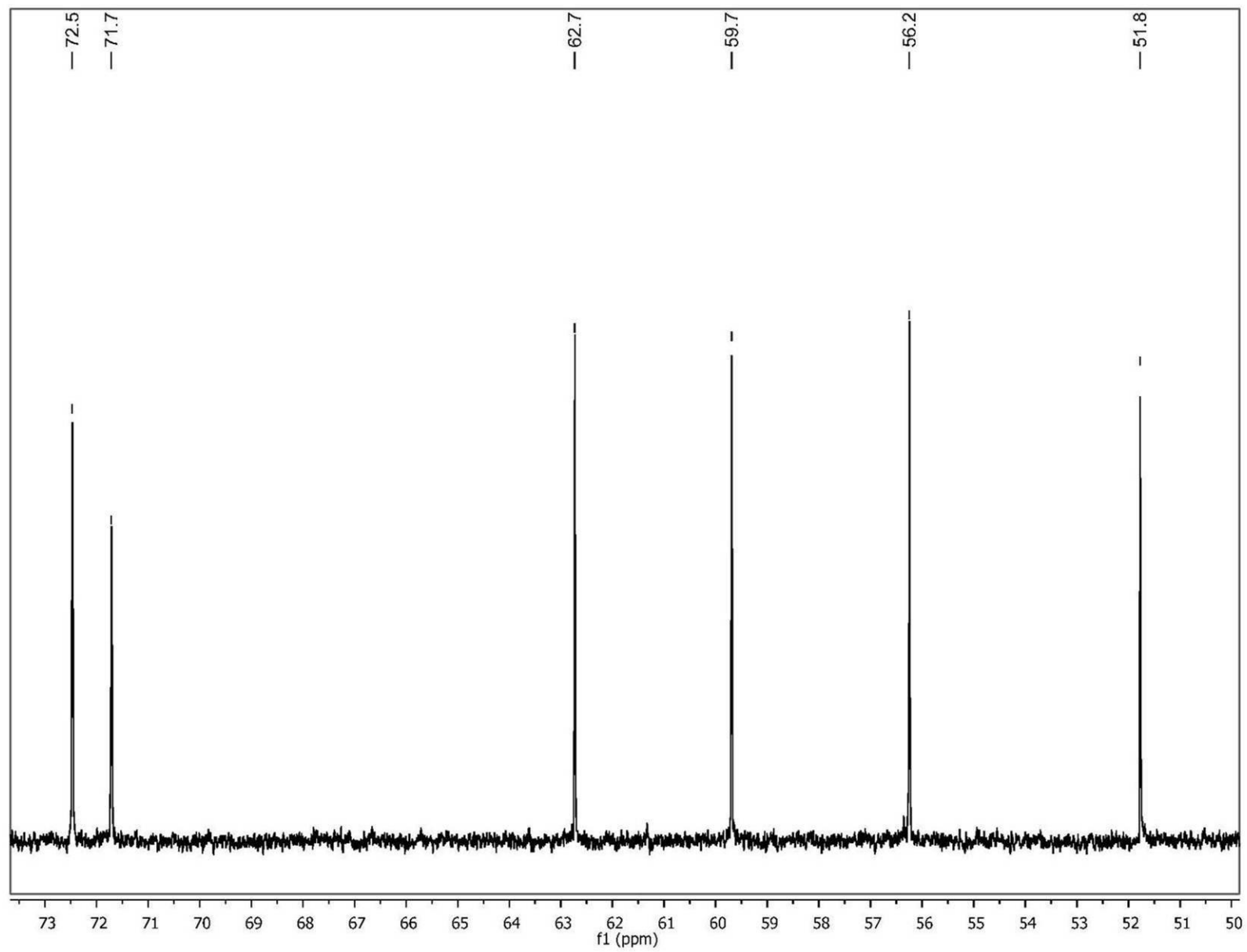




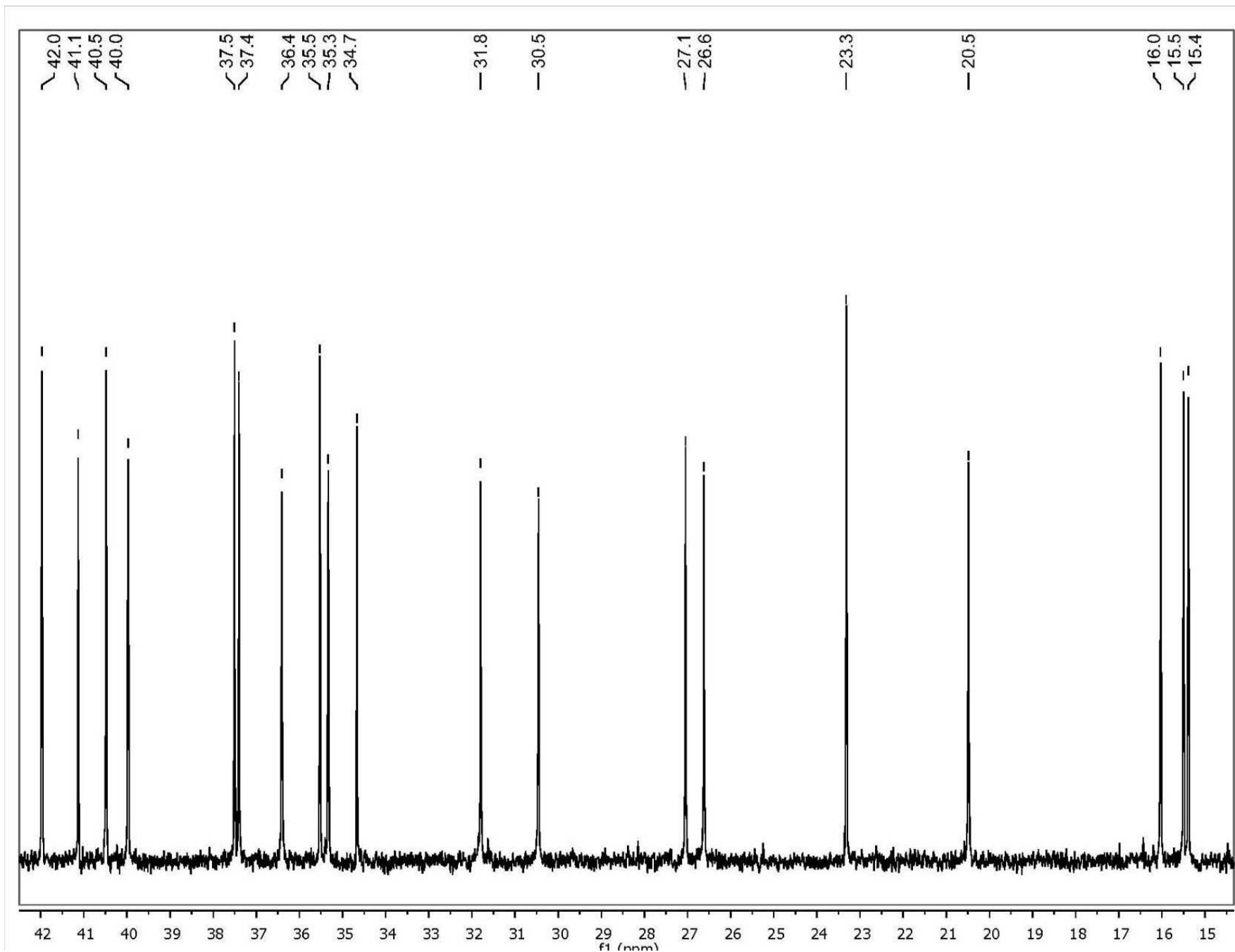




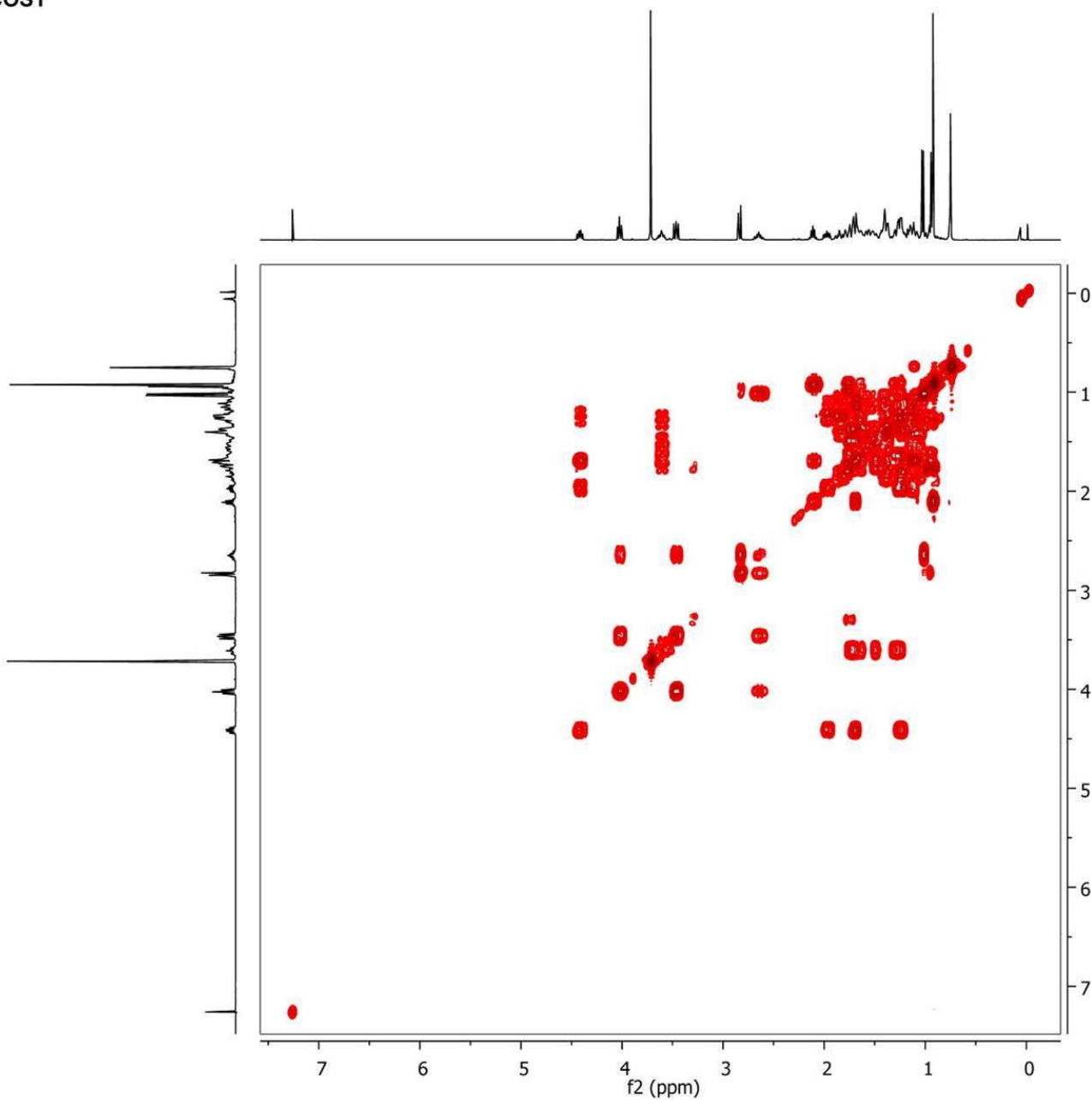
Parámetro	Valor
1 Comment	11398
	USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmr5
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	s2pul
8 Experiment	1D
9 Number of Scans	2000
10 Receiver Gain	30
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	1.3005
14 Acquisition Date	2010-04-09T 20:33:12
15 Modification Date	2010-04-10T 08:49:26
16 Spectrometer Frequency	100.52
17 Spectral Width	24509.8
18 Lowest Frequency	-1699.6
19 Nucleus	13C
20 Acquired Size	31875
21 Spectral Size	65536





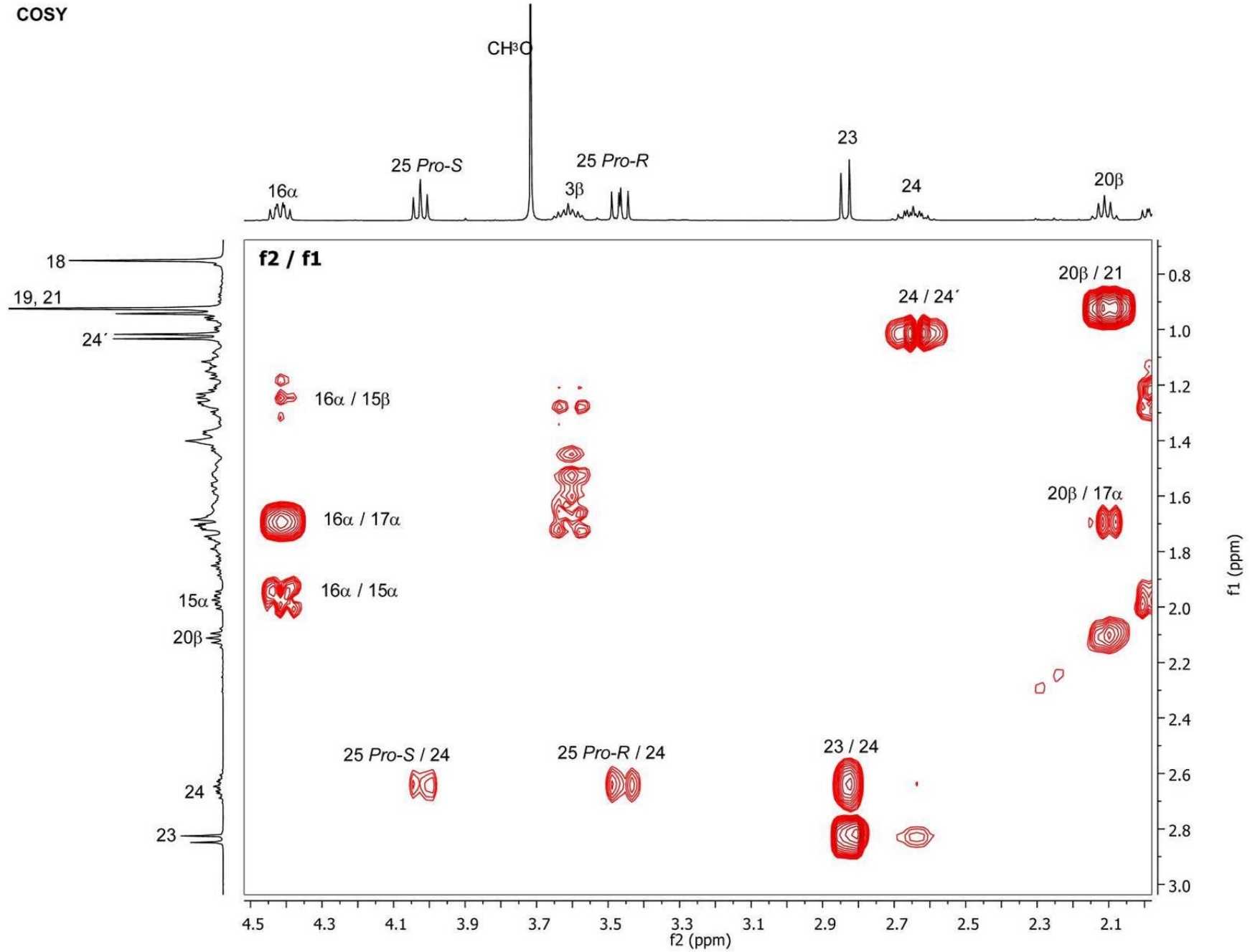


## COSY

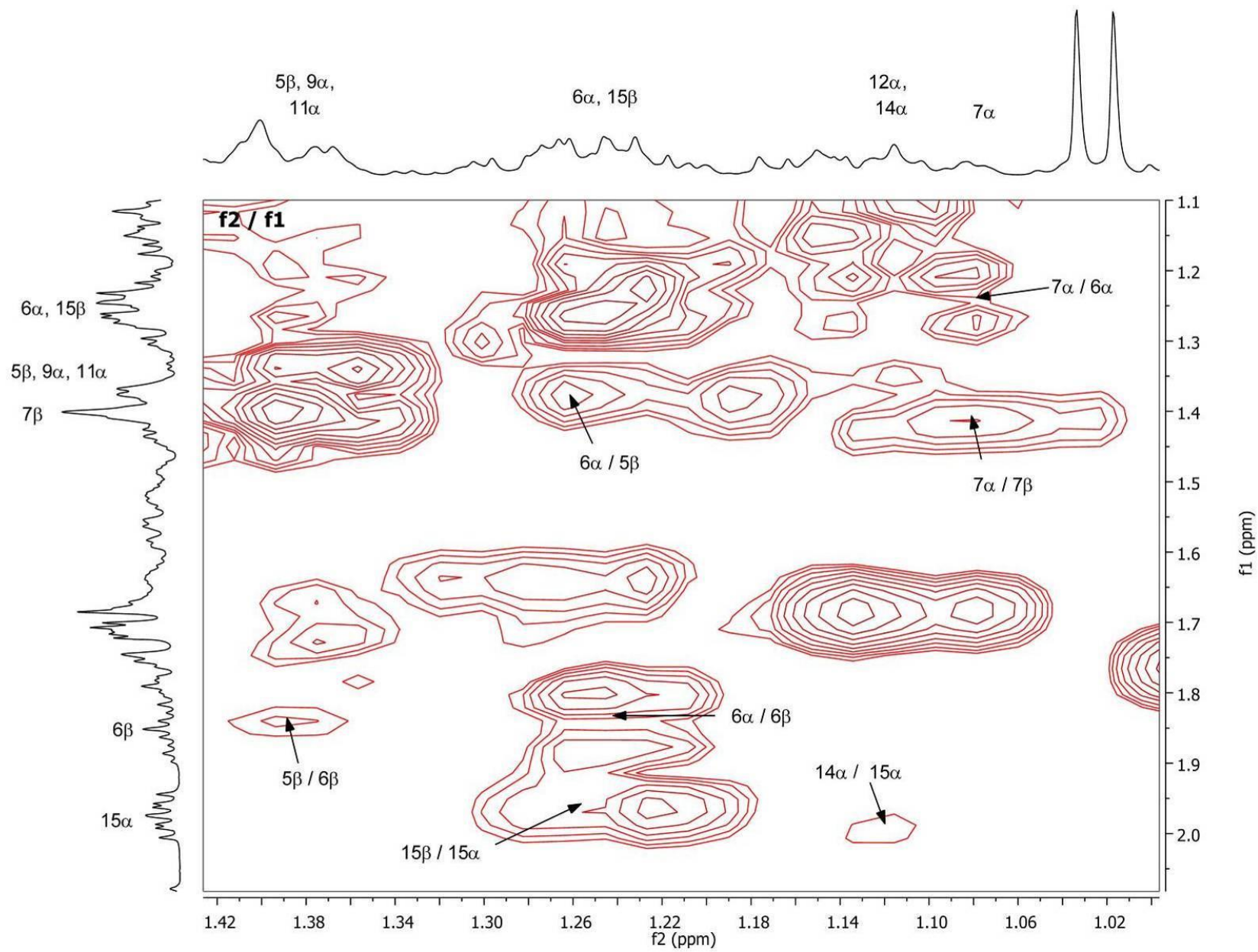


Parámetro	Valor (f2, f1)
1 Comment	11398
	USAI-UNAM
	9/ ABRIL/ 2010
	Dr. Martin
	Iglesias /
	Mariana
	Macias
	FES-30H/
	CDCI3
	1H 400 MHz
	400-MR
	NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	vnmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gCOSY
8 Number of Scans	4
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1352
13 Acquisition Date	2010-04-10T02:50:00
14 Modification Date	2010-04-10T08:49:38
15 Spectrometer Frequency	(399.72, 399.72)
16 Spectral Width	(3787.9, 3787.9)
17 Lowest Frequency	(-465.8, -465.4)
18 Nucleus	(1H, 1H)
19 Acquired Size	(512, 256)
20 Spectral Size	(512, 512)

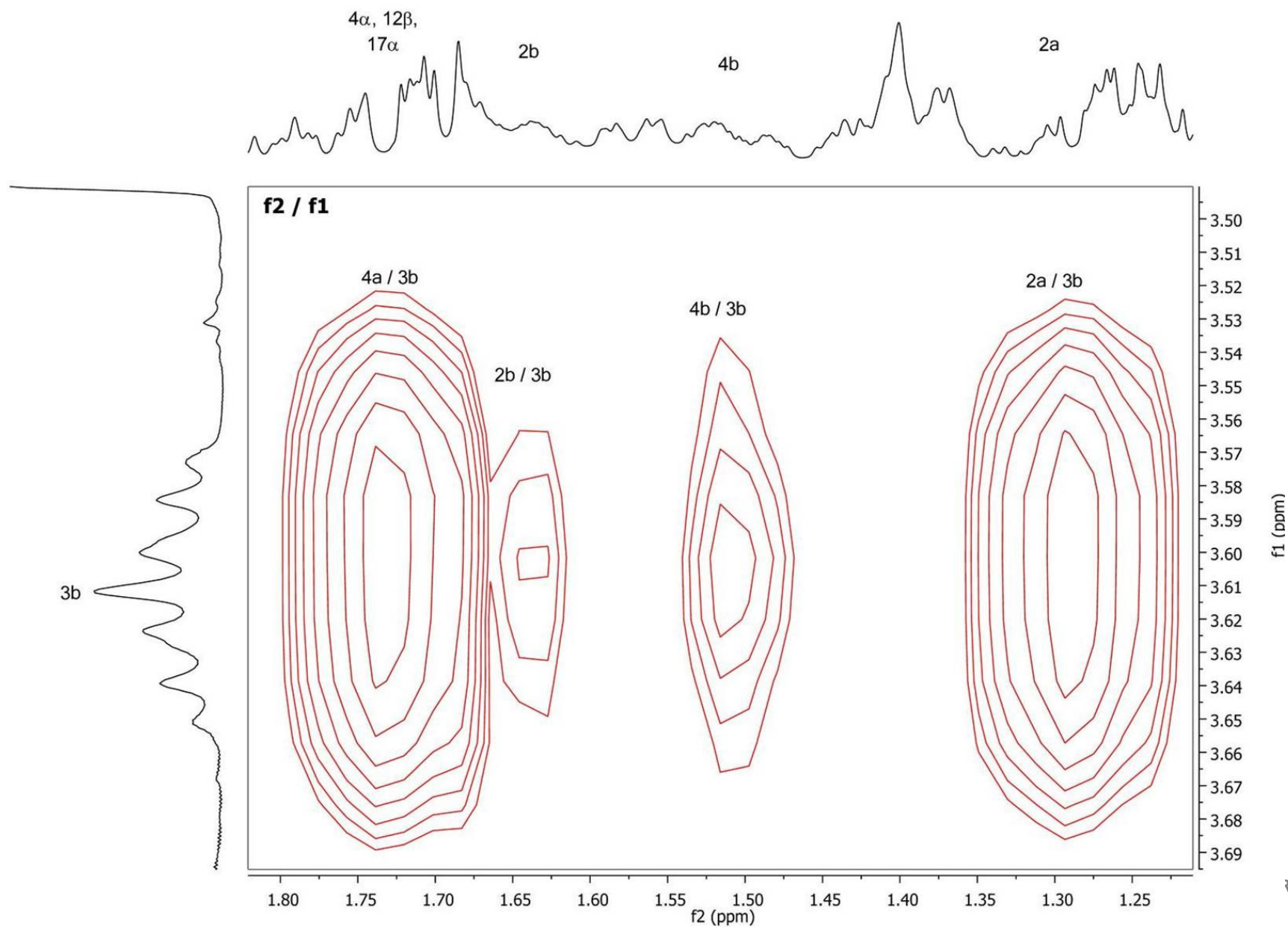
COSY



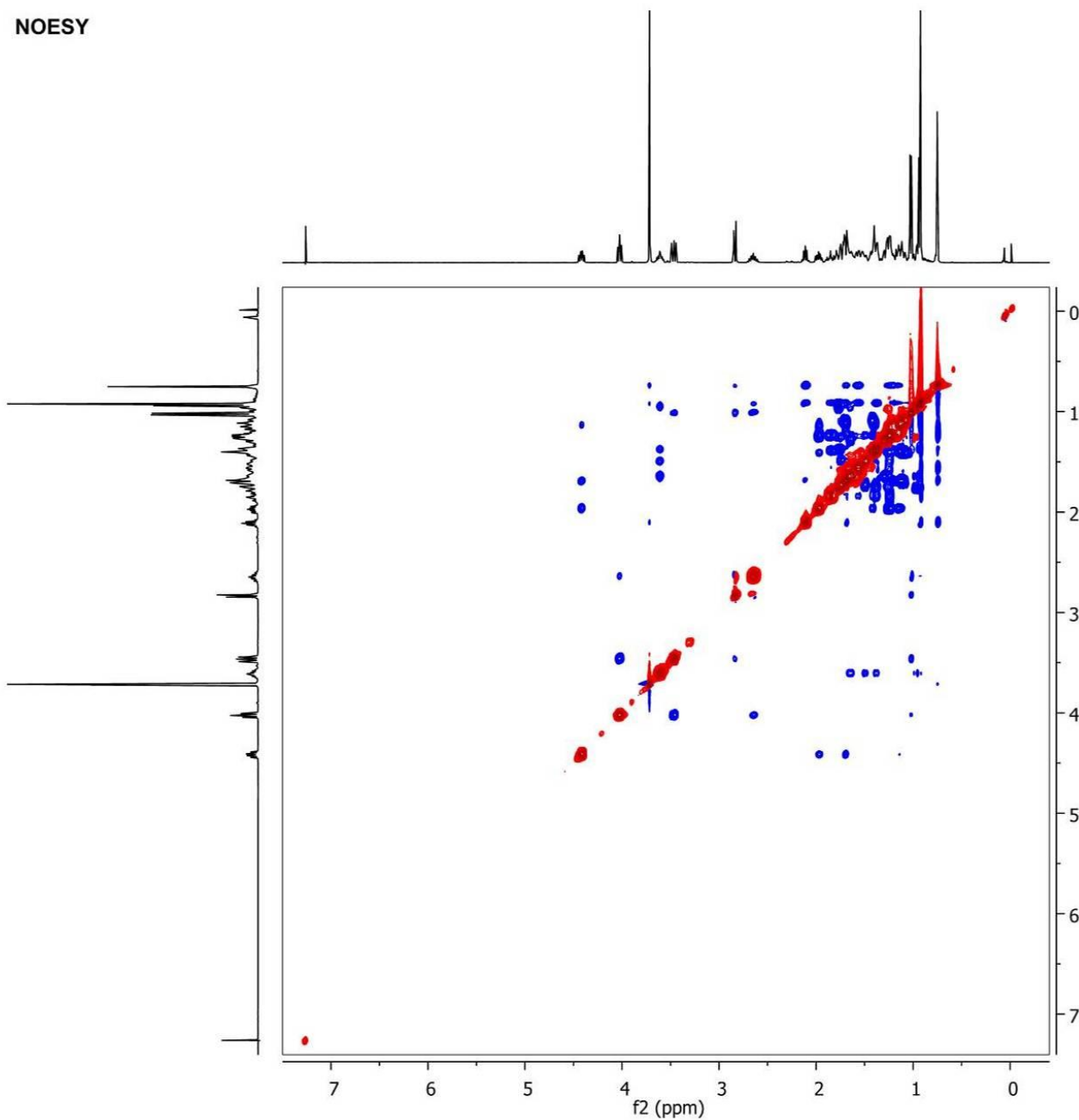
COSY



COSY

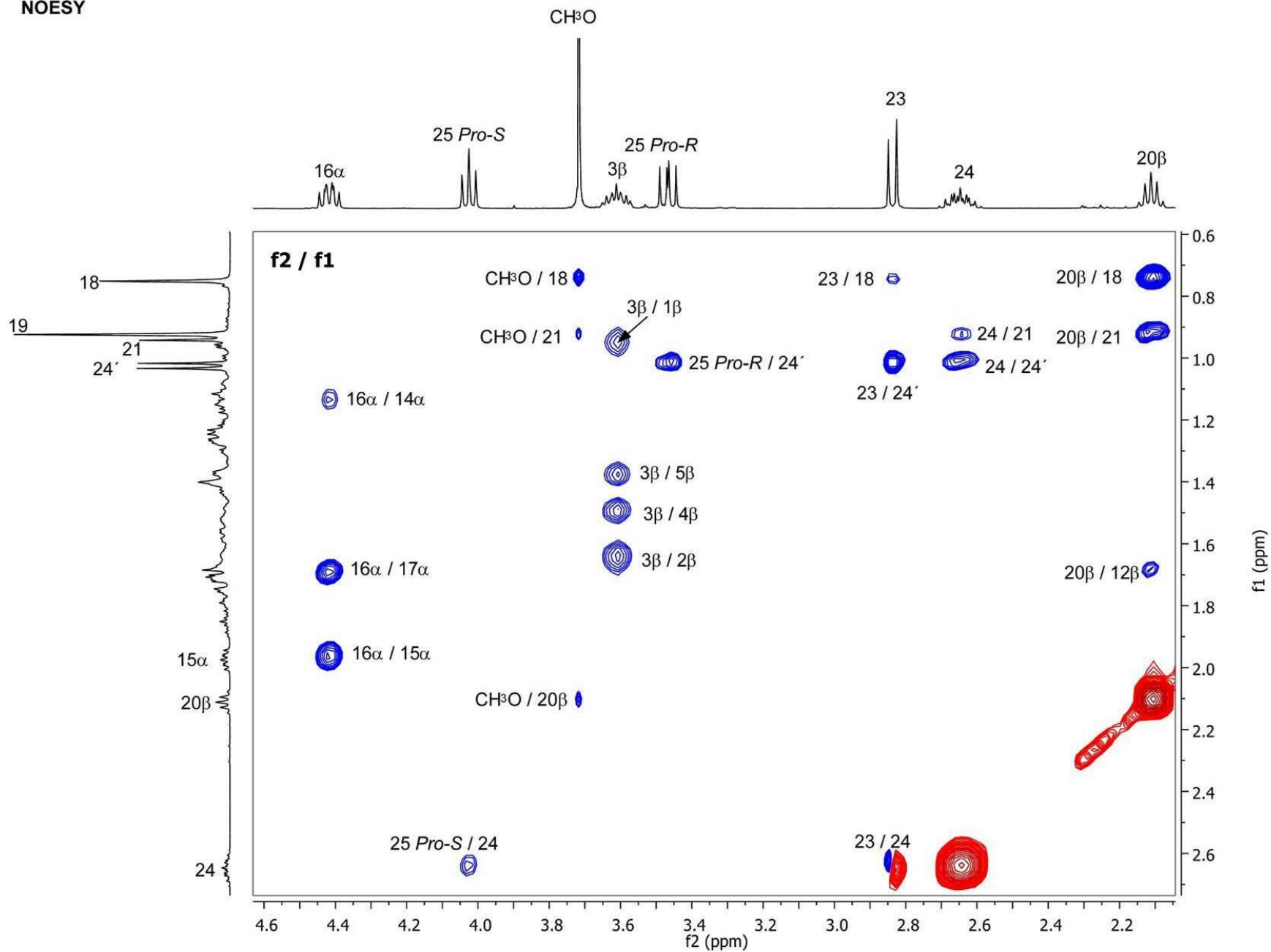


## NOESY

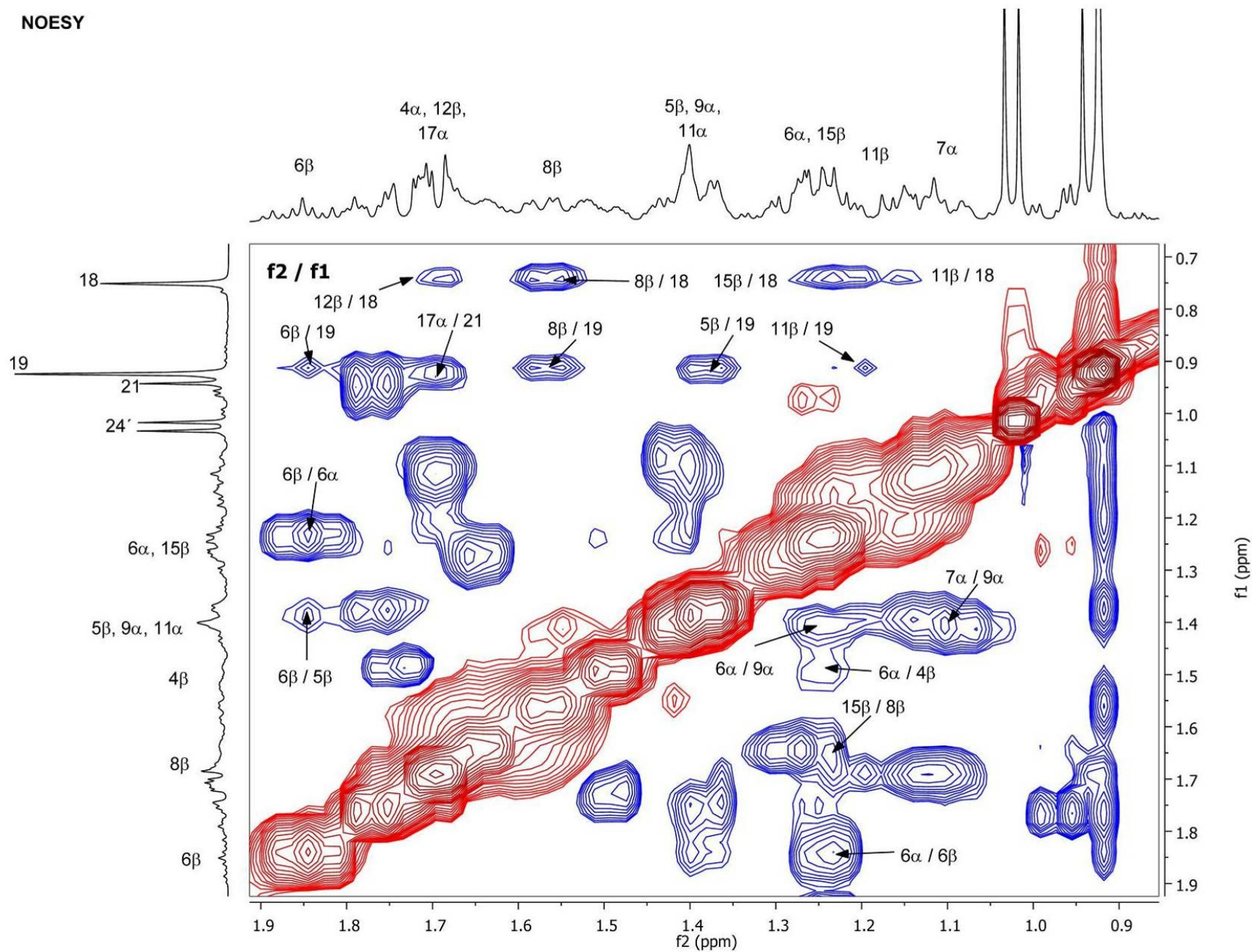


Parámetro	Valor (f2, f1)
1 Comment	11398
	USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	NOESY
8 Number of Scans	8
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1352
13 Acquisition Date	2010-04-10T0 3:10:36
14 Modification Date	2010-04-10T0 8:49:54
15 Spectrometer Frequency	(399.72, 399.72)
16 Spectral Width	(3787.9, 3787.9)
17 Lowest Frequency	(-463.4, -465.4)
18 Nucleus	(1H, 1H)
19 Acquired Size	(512, 256)
20 Spectral Size	(512, 512)

NOESY

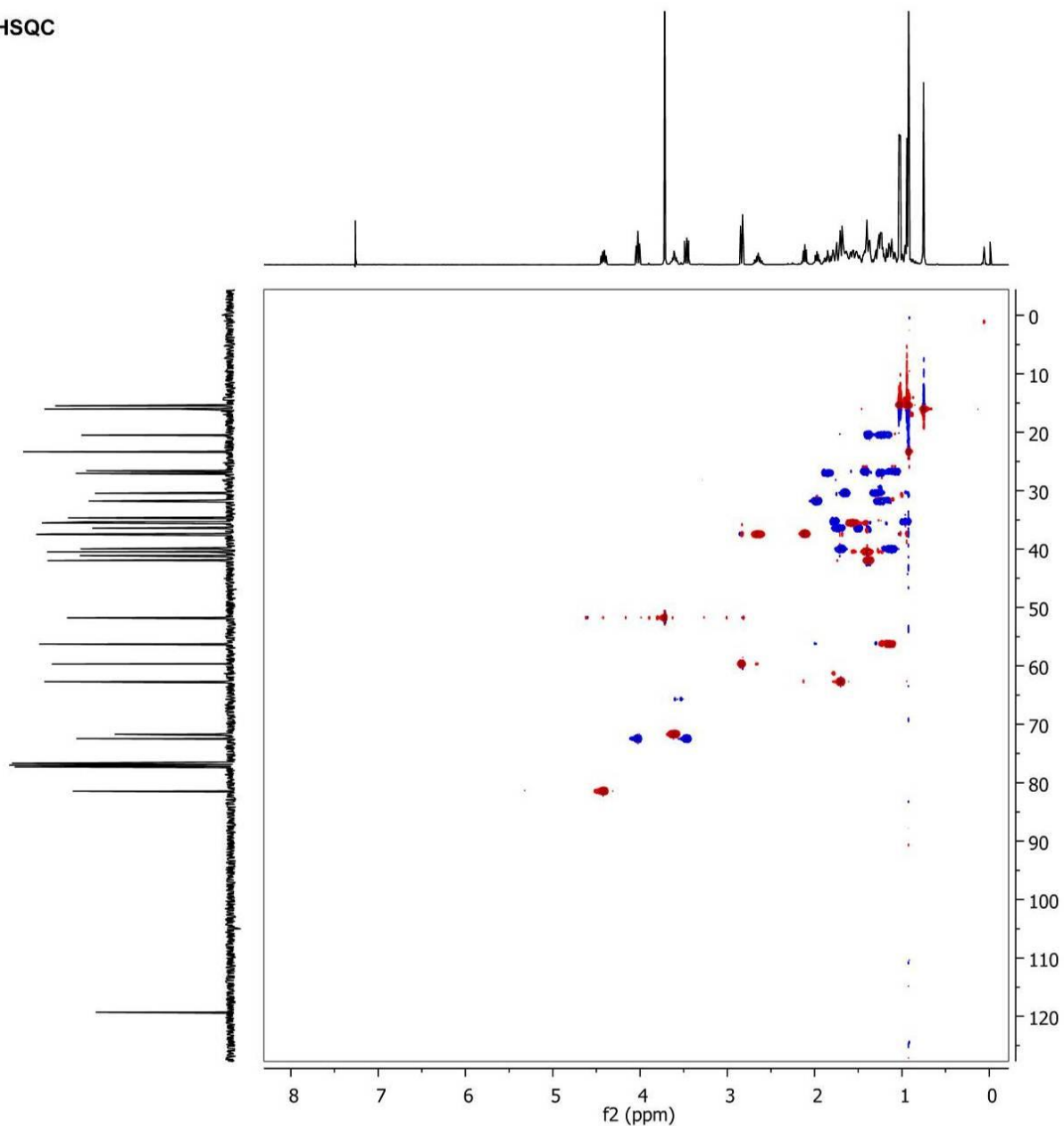


NOESY



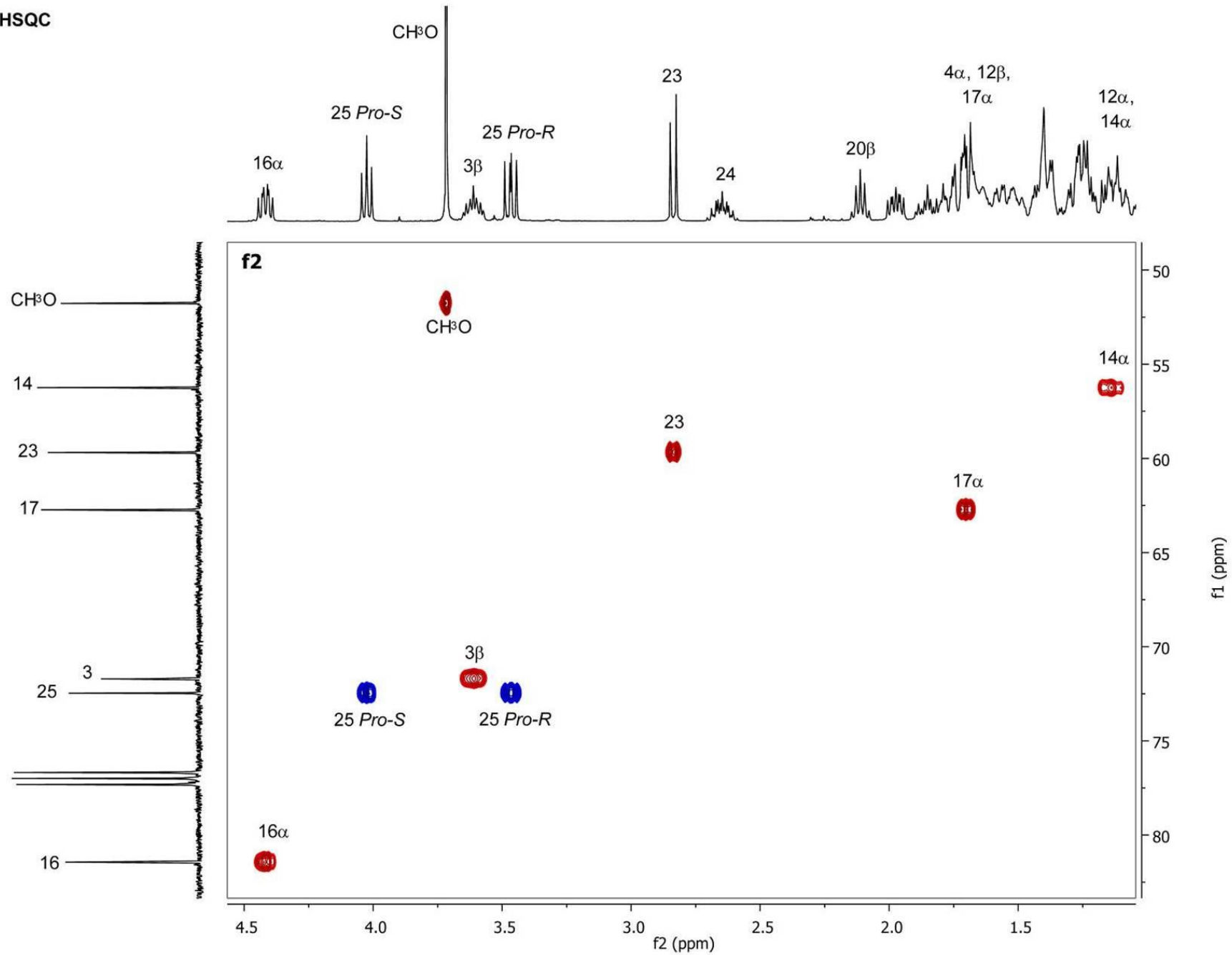


## HSQC

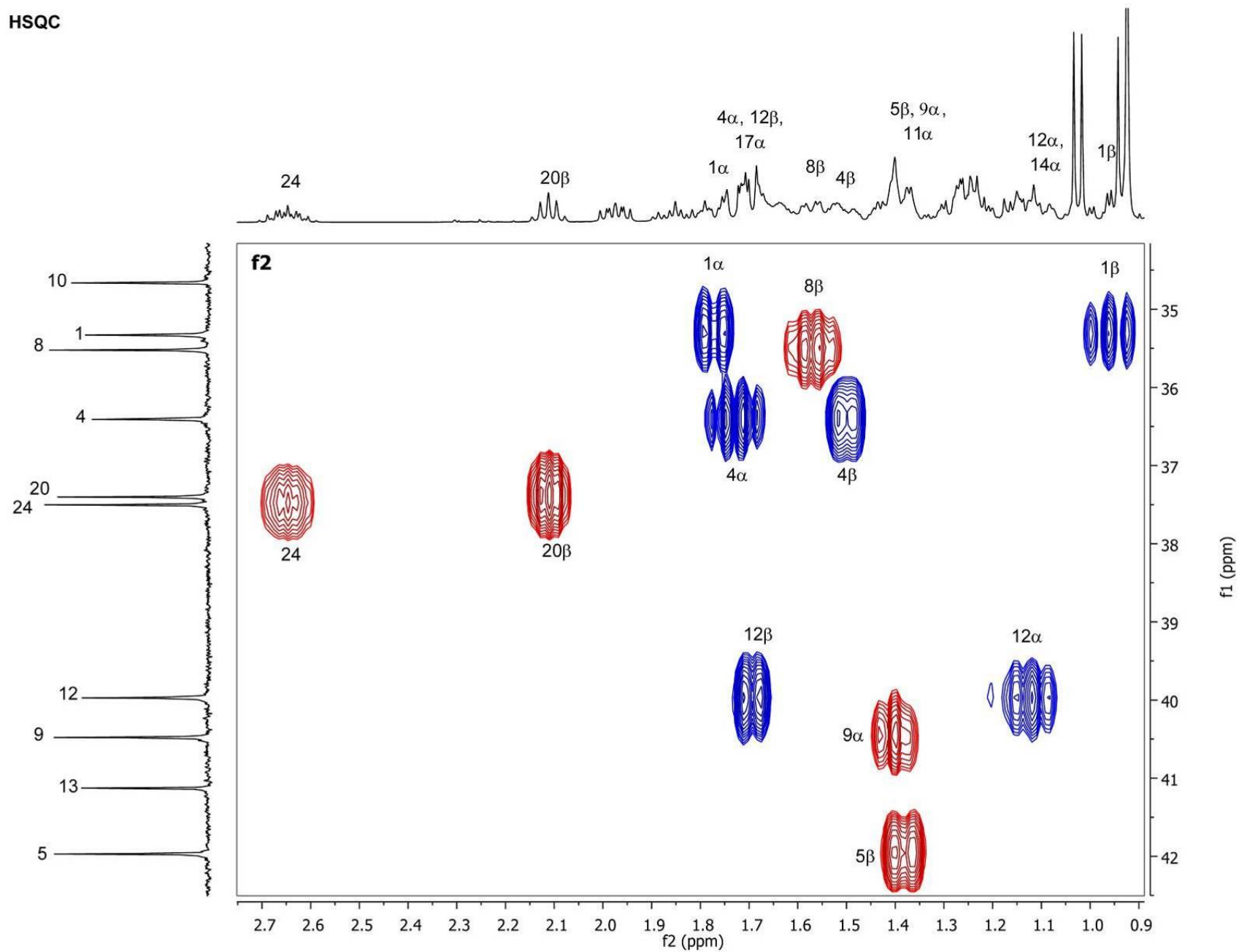


Parámetro	Valor (f2, f1)
1 Comment	11398 USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	vnmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHSQC
8 Experiment	2D-HSQC- EDITED
9 Number of Scans	8
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1993
14 Acquisition Date	2010-04-10T0 0:05:59
15 Modification Date	2010-04-10T0 8:50:08
16 Spectrometer Frequency	(399.72, 100.52)
17 Spectral Width	(3787.9, 17086.7)
18 Lowest Frequency	(-466.2, -1005.2)
19 Nucleus	(1H, 13C)

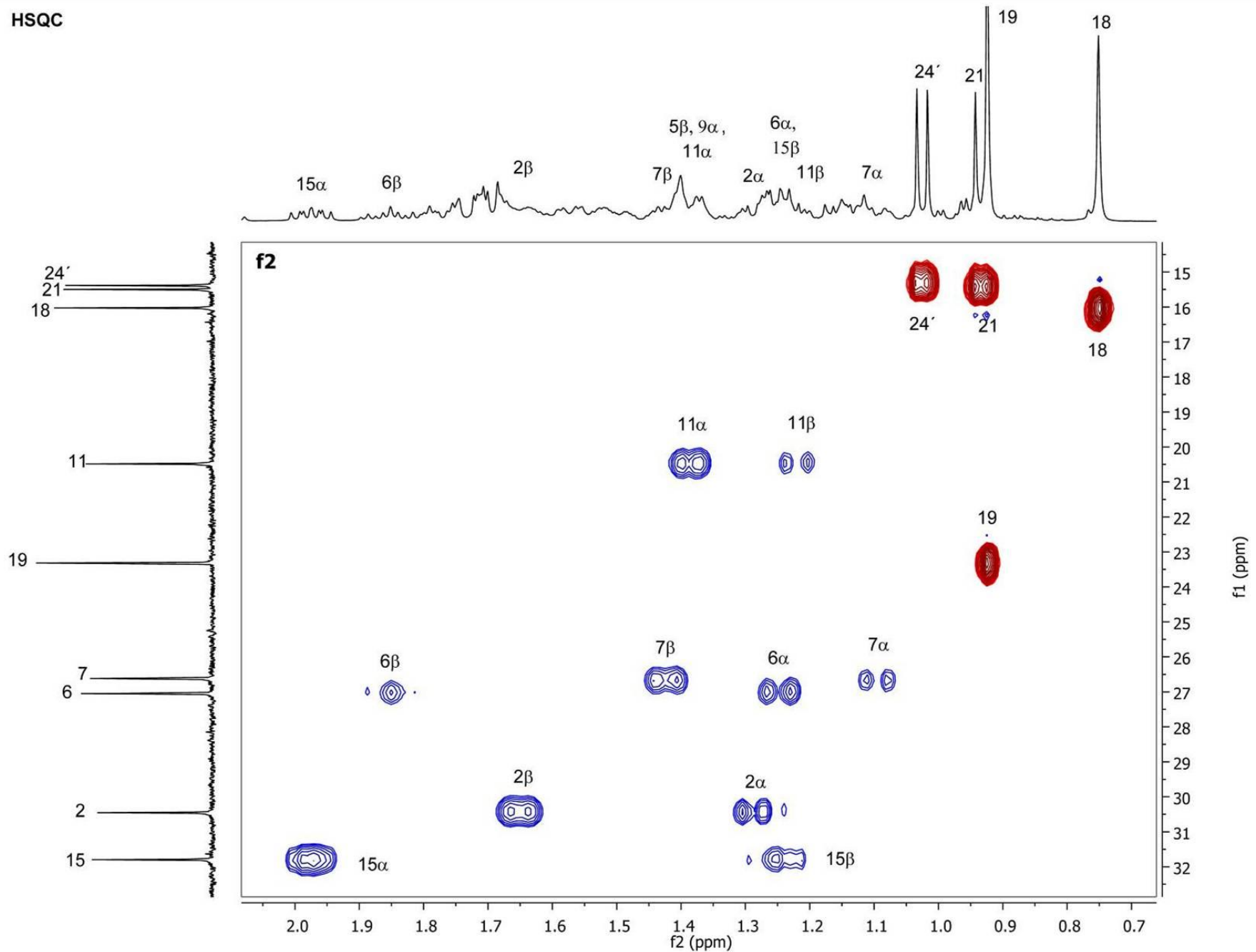
HSQC



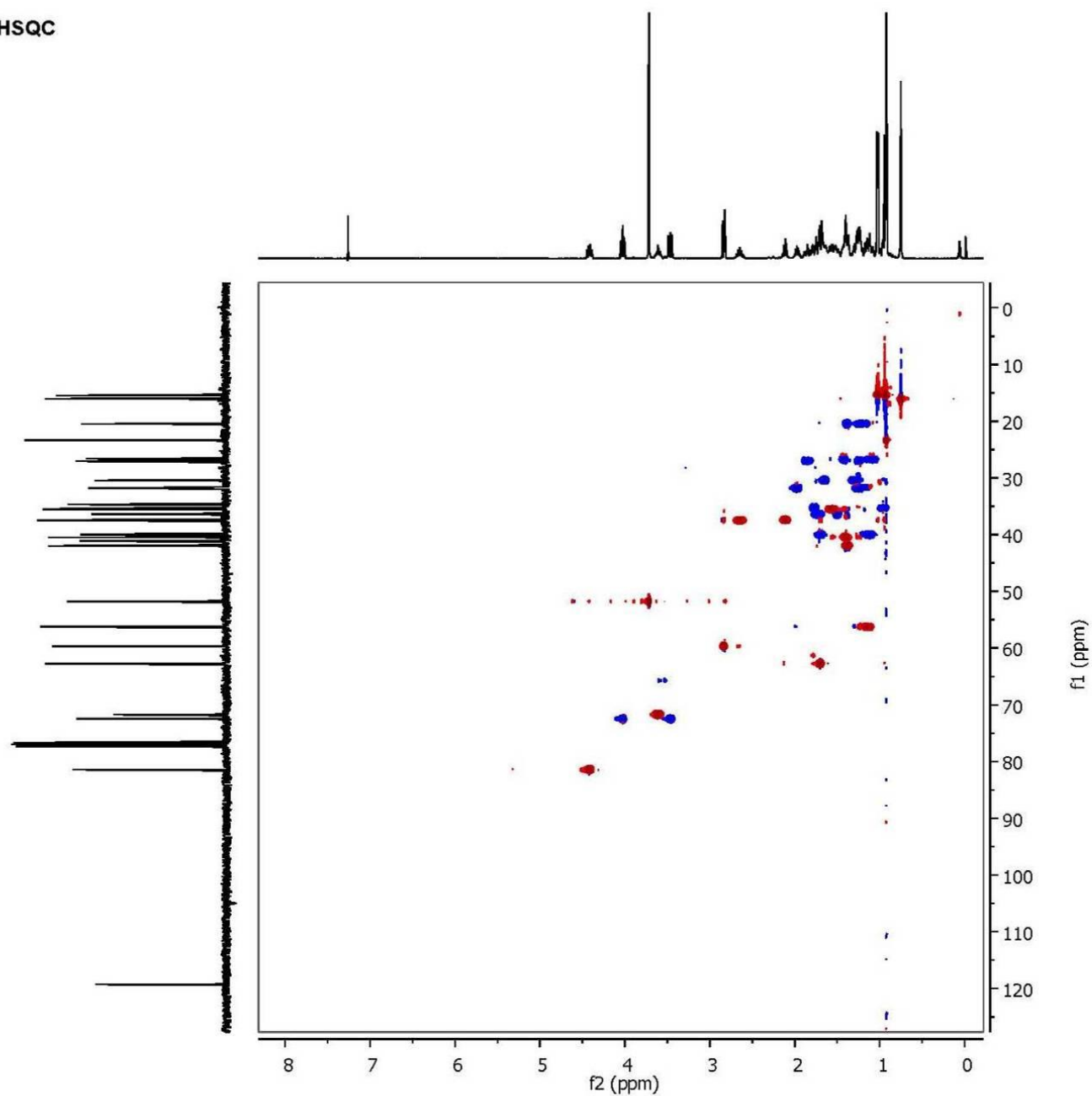
HSQC



HSQC

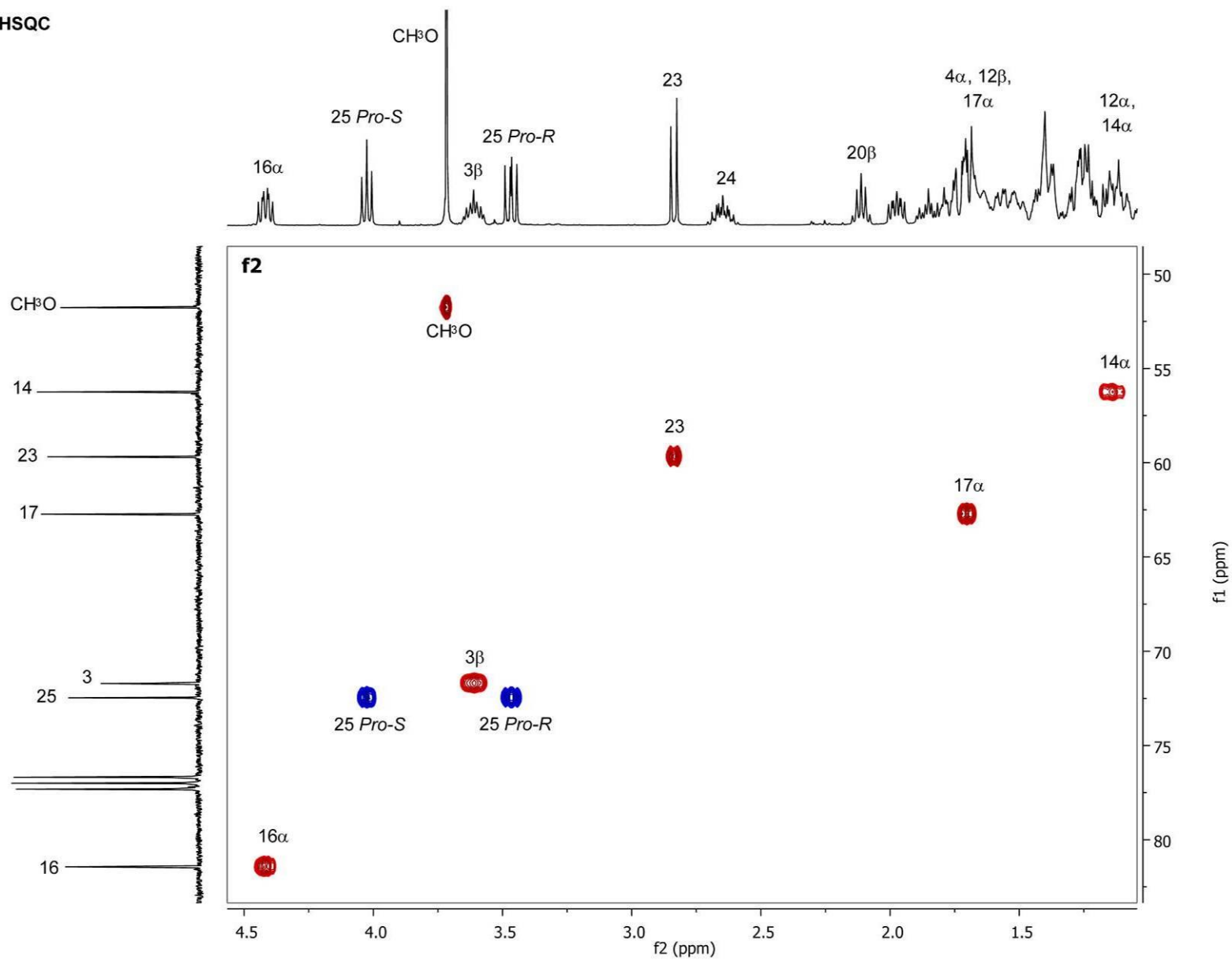


## HSQC

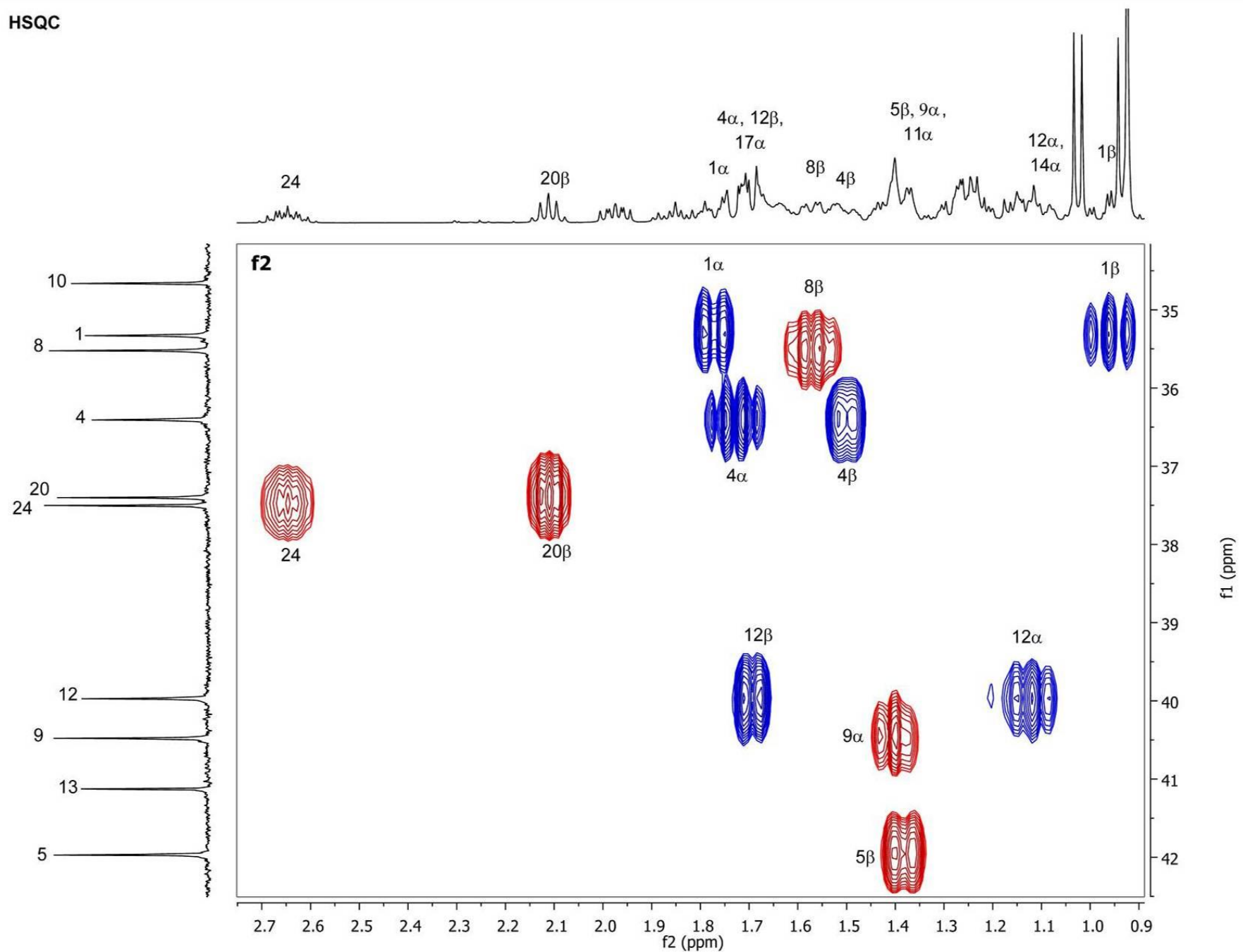


Parámetro	Valor (f2, f1)
1 Comment	11398
	USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	vnmr1
5 Solvent	cdd3
6 Temperature	26.0
7 Pulse Sequence	gHSQC
8 Experiment	2D-HSQC- EDITED
9 Number of Scans	8
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1993
14 Acquisition Date	2010-04-10T0 0:05:59
15 Modification Date	2010-04-10T0 8:50:08
16 Spectrometer Frequency	(399.72, 100.52)
17 Spectral Width	(3787.9, 17086.7)
18 Lowest Frequency	(-466.2, -1005.2)
19 Nucleus	(1H, 13C)

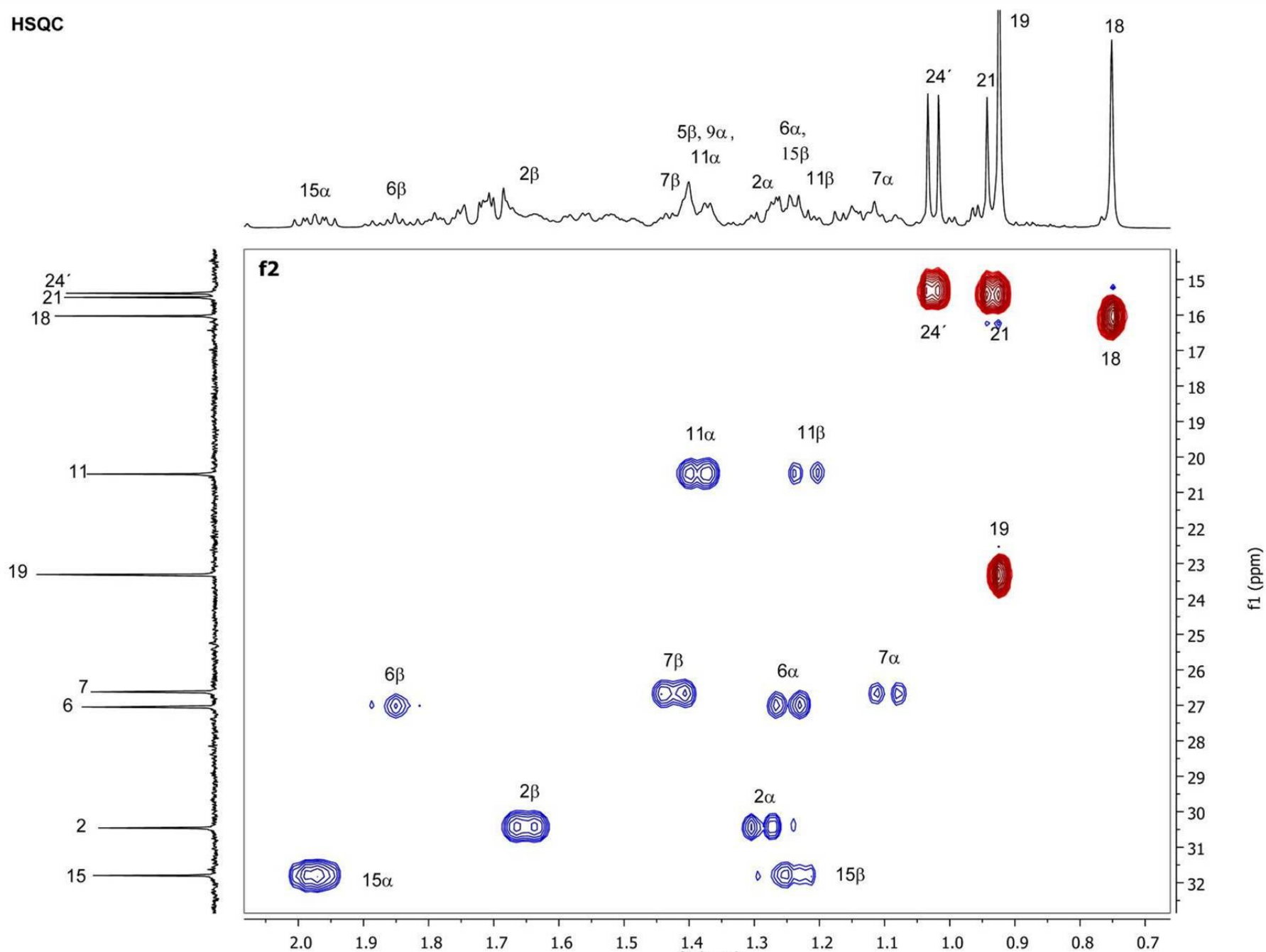
HSQC



HSQC

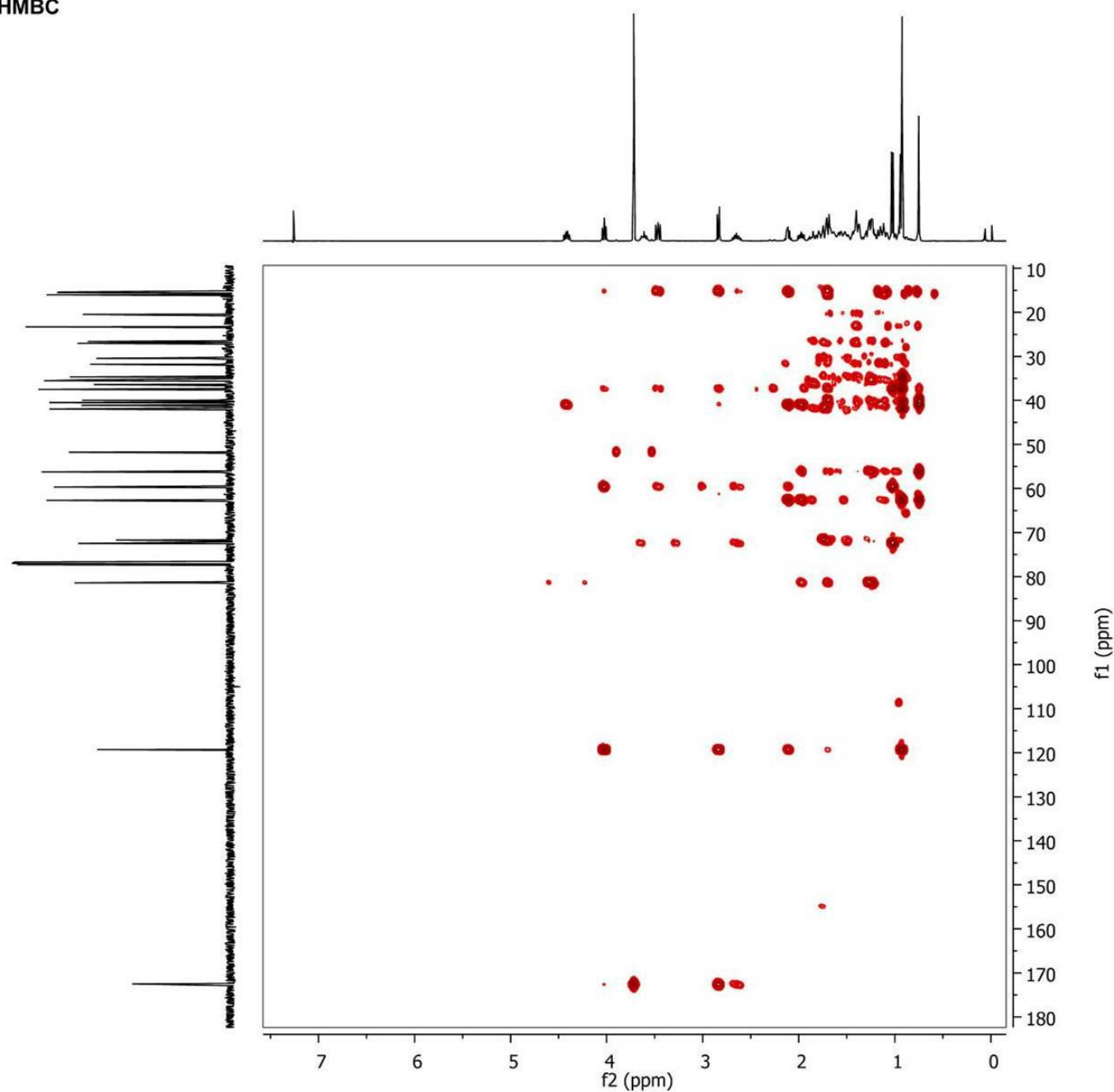


HSQC





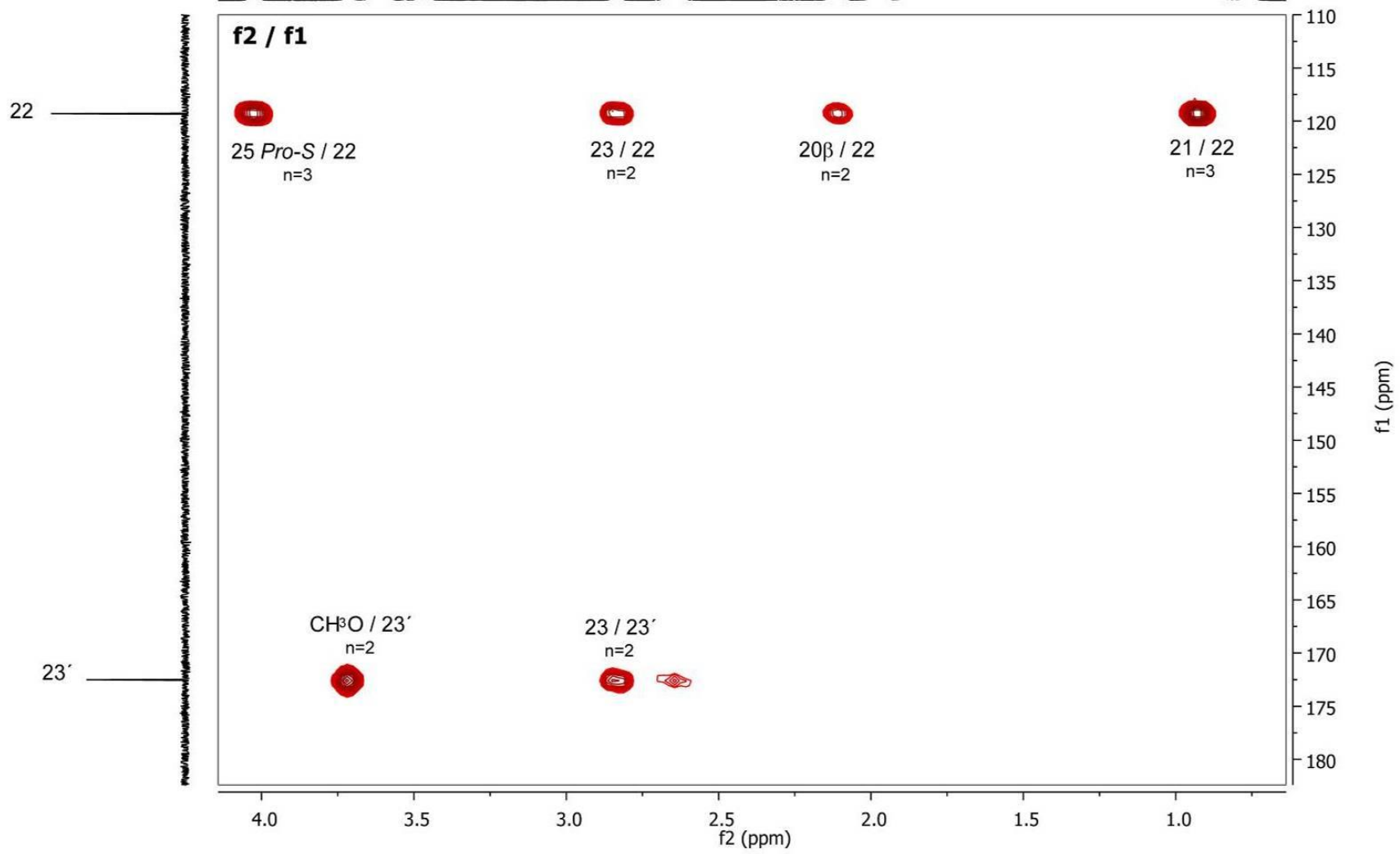
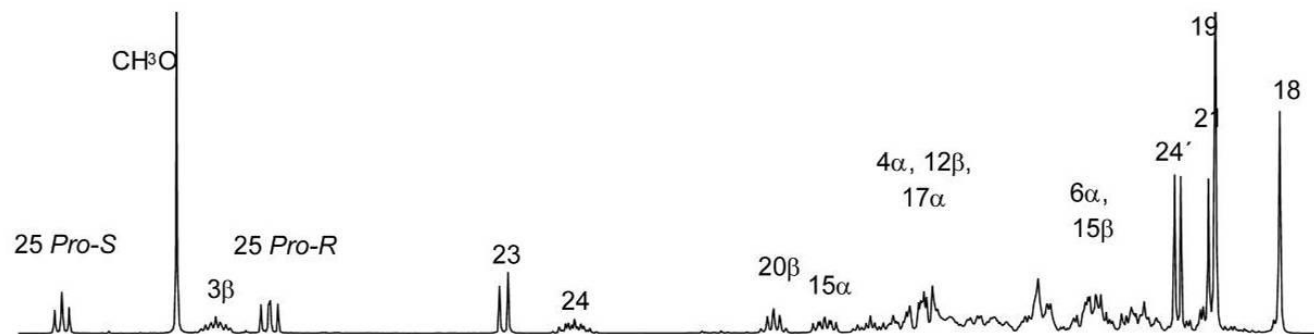
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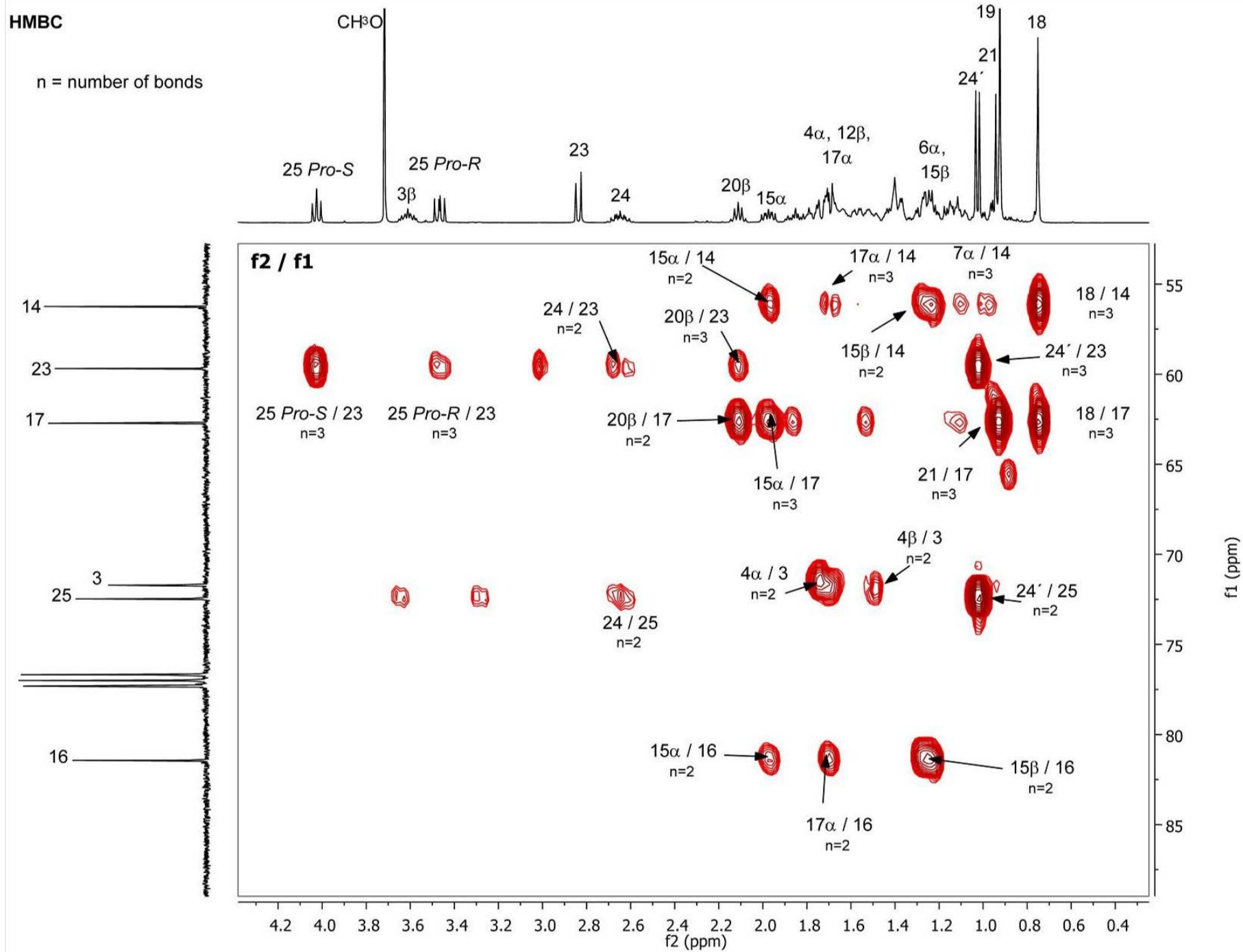


Parámetro	Valor (f2, f1)
1 Comment	11398
	USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHMBC
8 Number of Scans	16
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1280
13 Acquisition Date	2010-04-10T06:32:15
14 Modification Date	2010-04-10T08:50:22
15 Spectrometer Frequency	(399.72, 100.52)
16 Spectral Width	(3787.9, 24125.5)
17 Lowest Frequency	(-462.6, -1488.4)
18 Nucleus	(1H, 13C)
19 Acquired Size	(485, 400)
20 Spectral Size	(512, 512)

HMBC

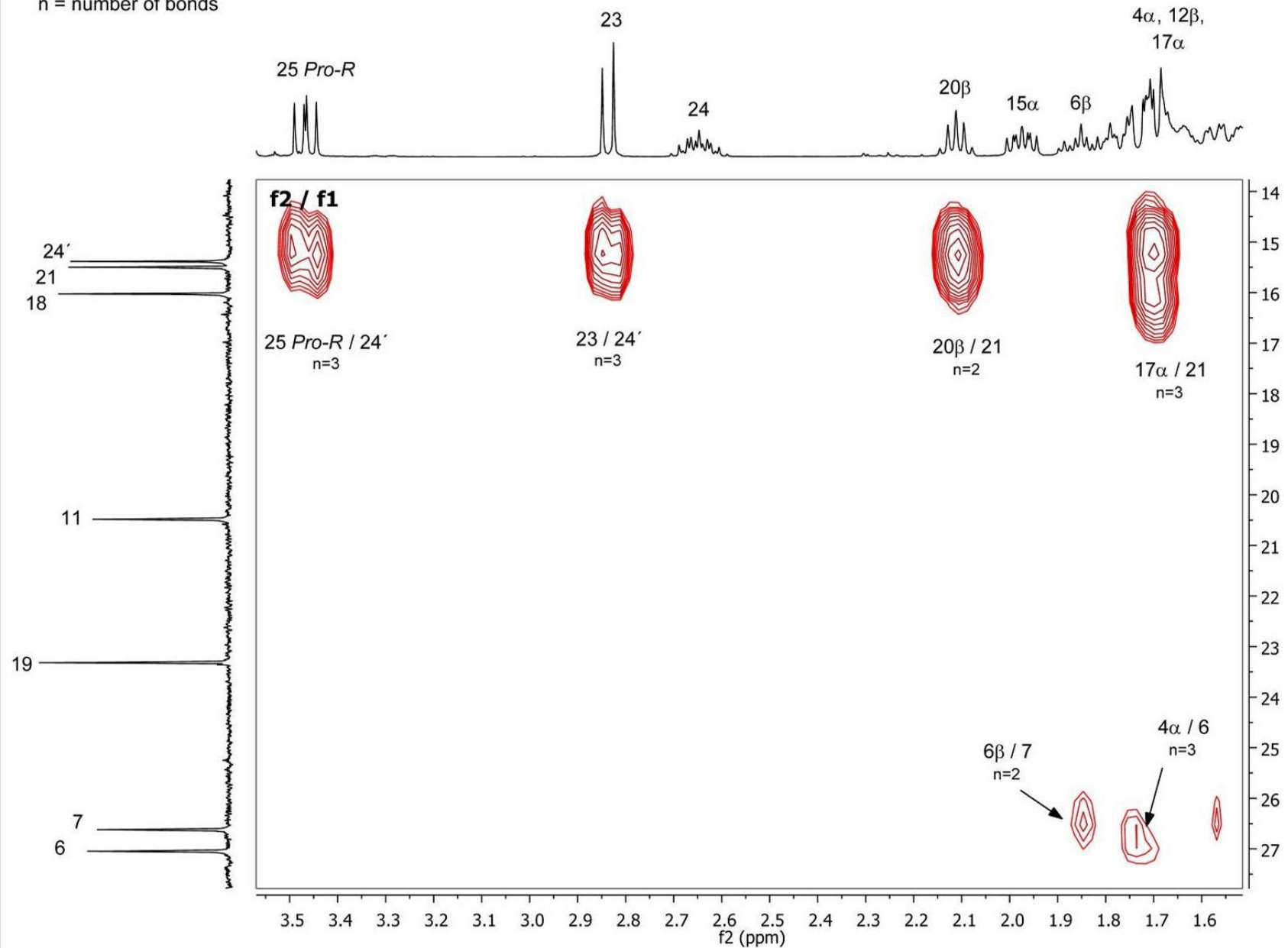
n = number of bonds





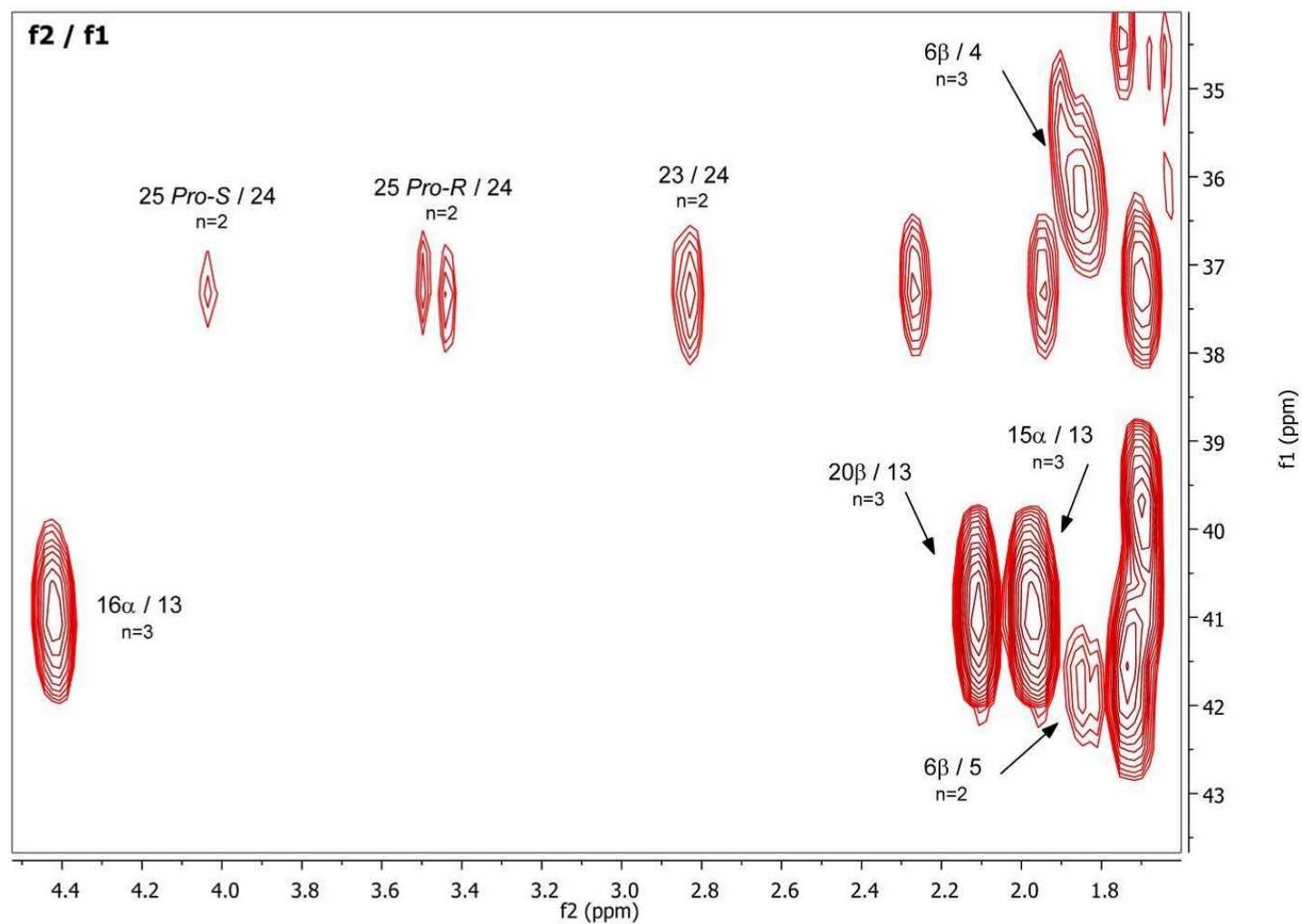
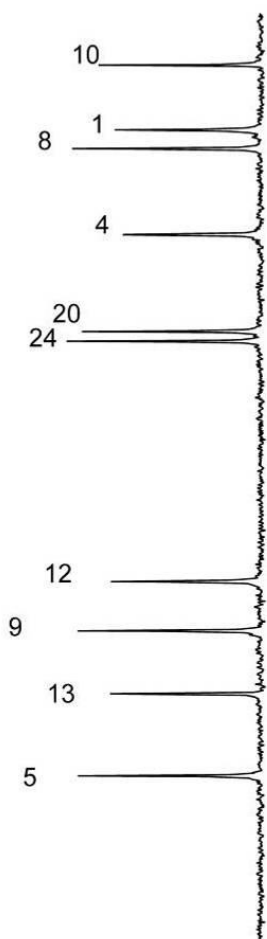
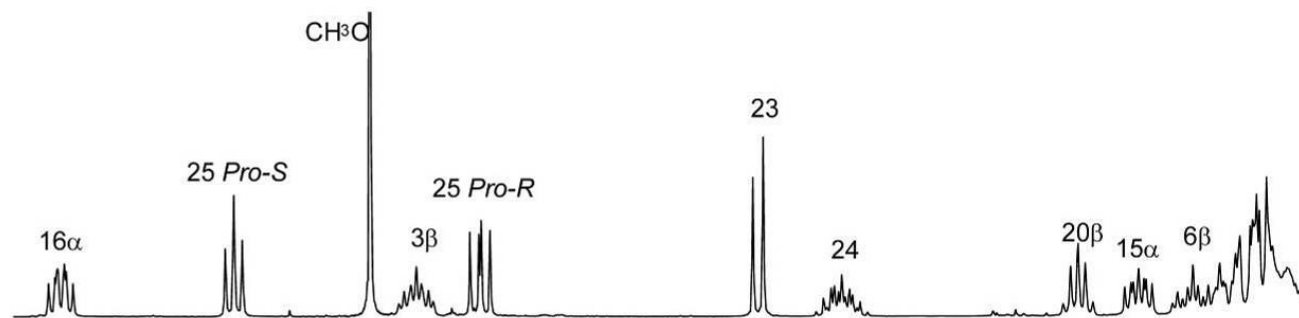
HMBC

n = number of bonds



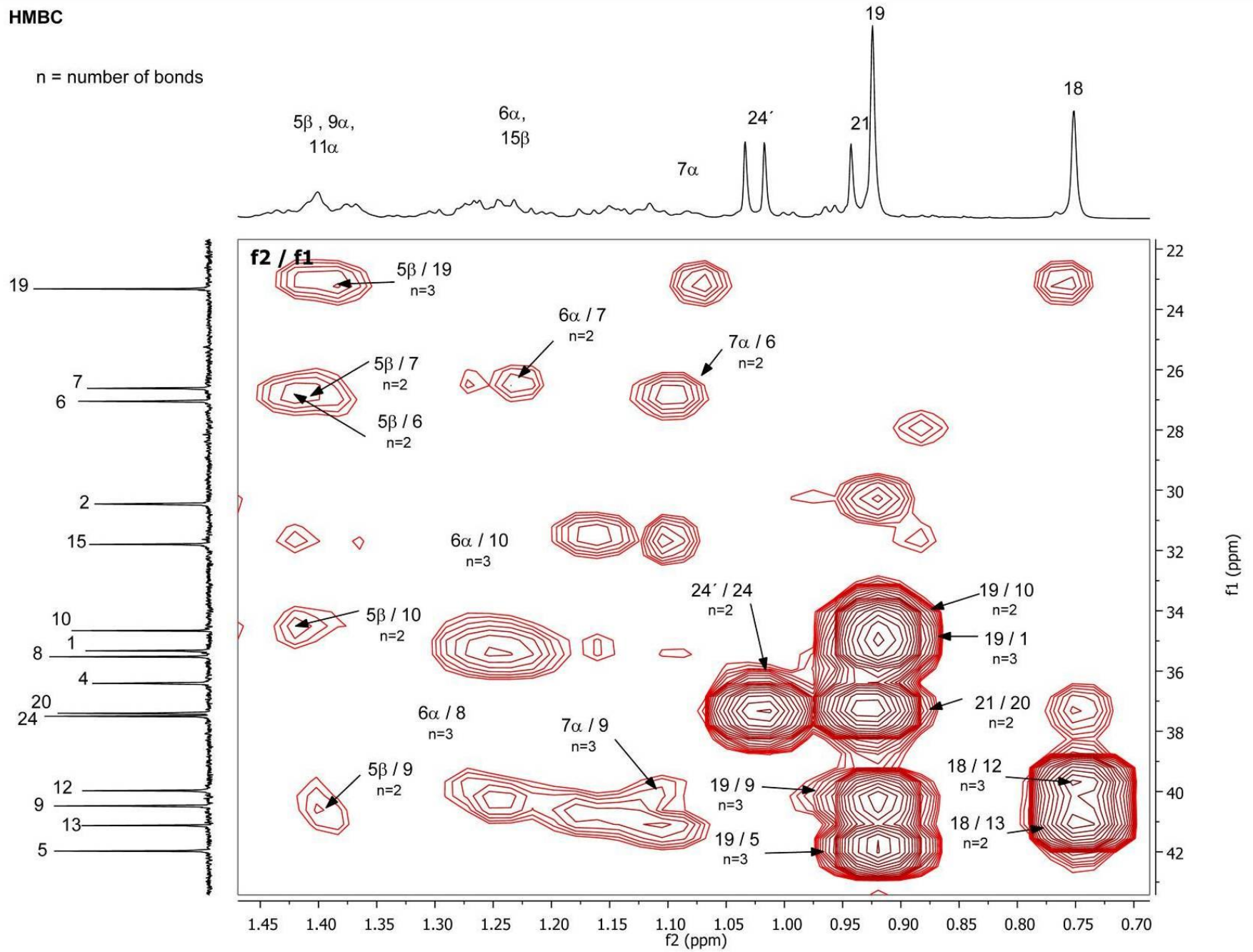
HMBC

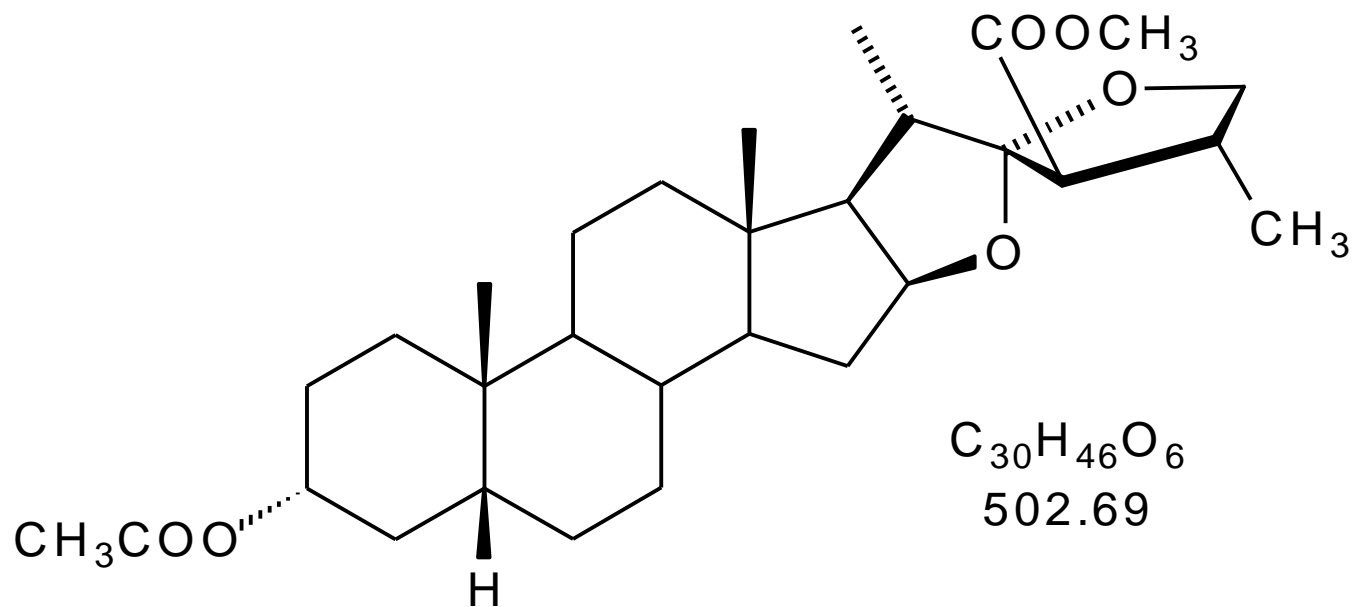
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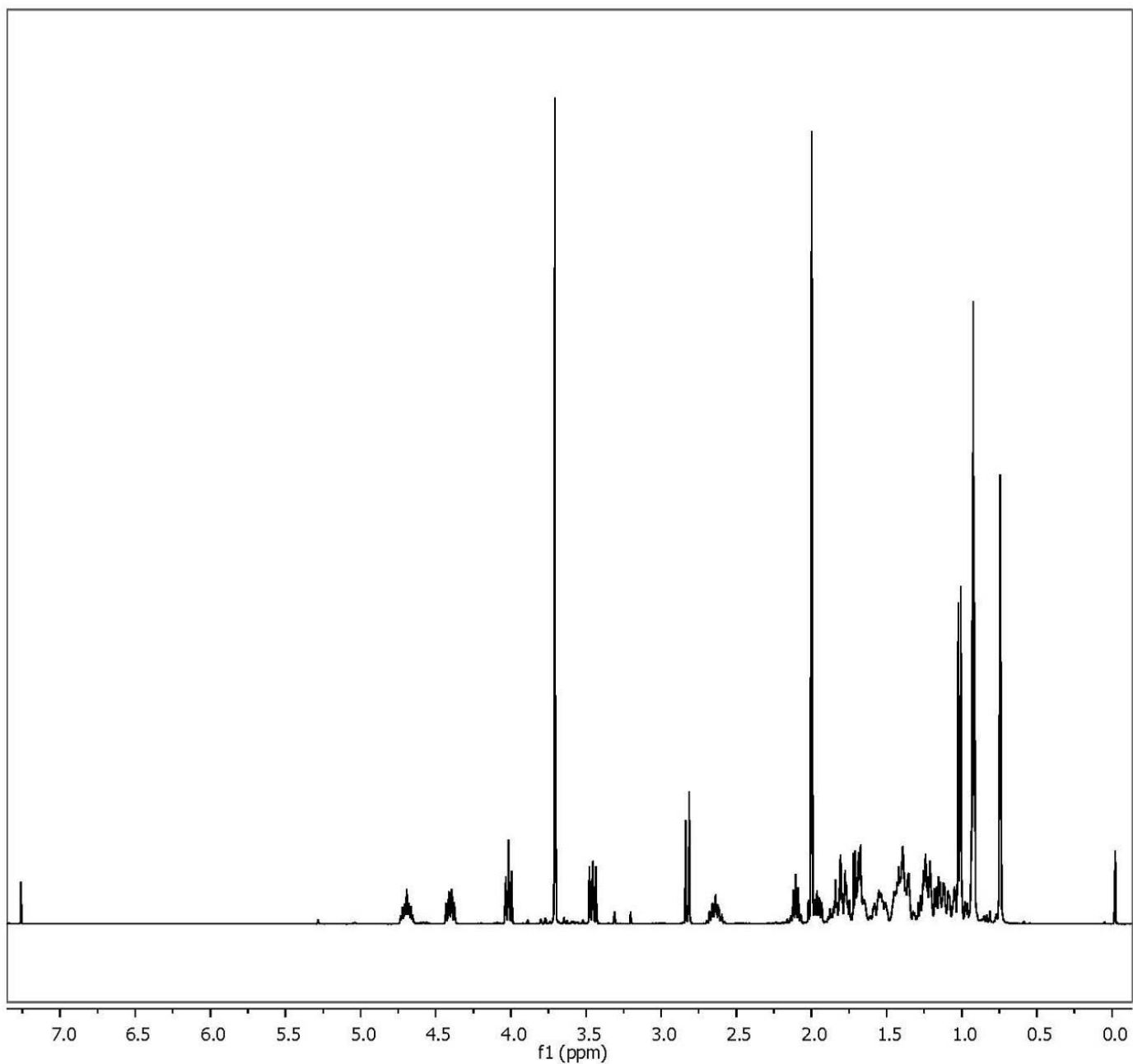
HMBC

n = number of bonds



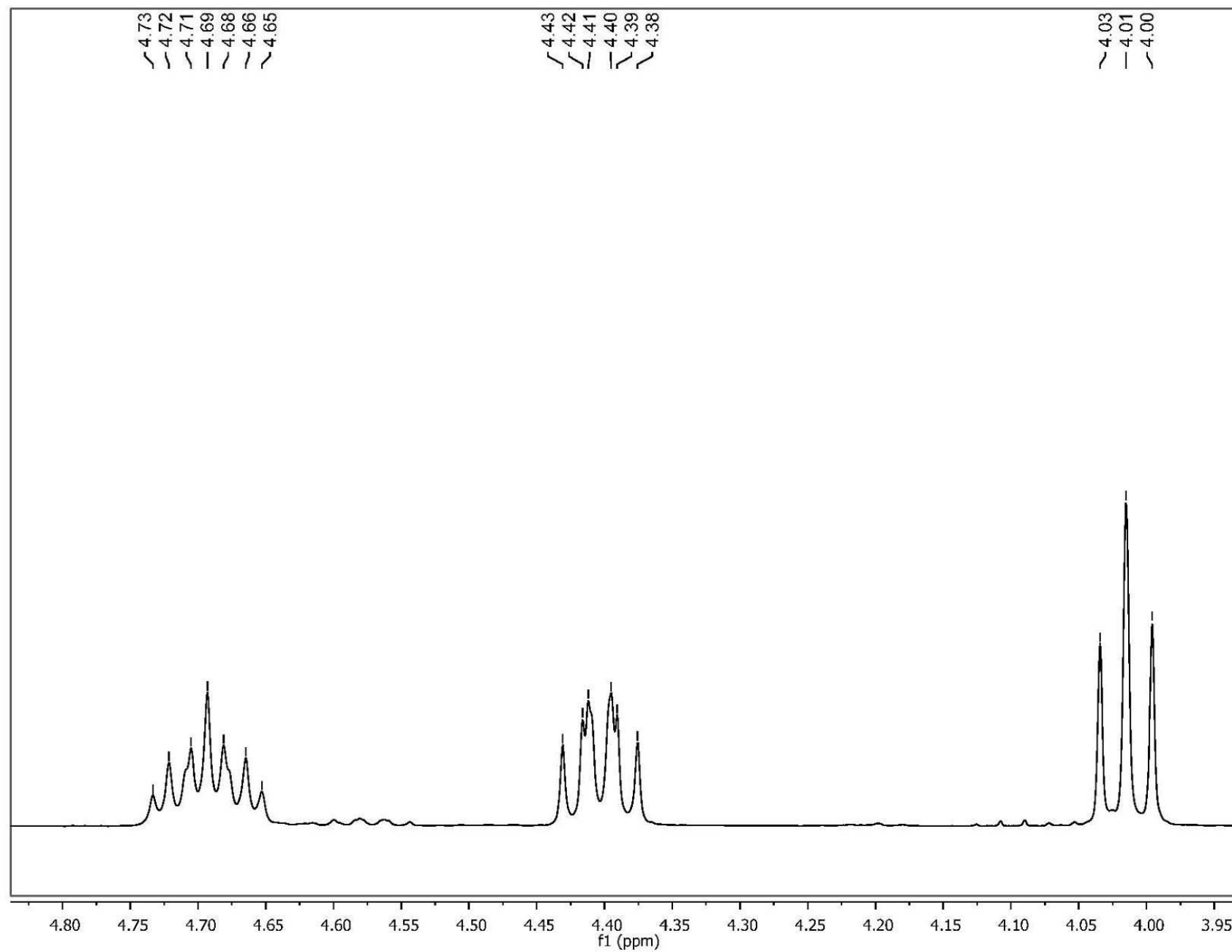


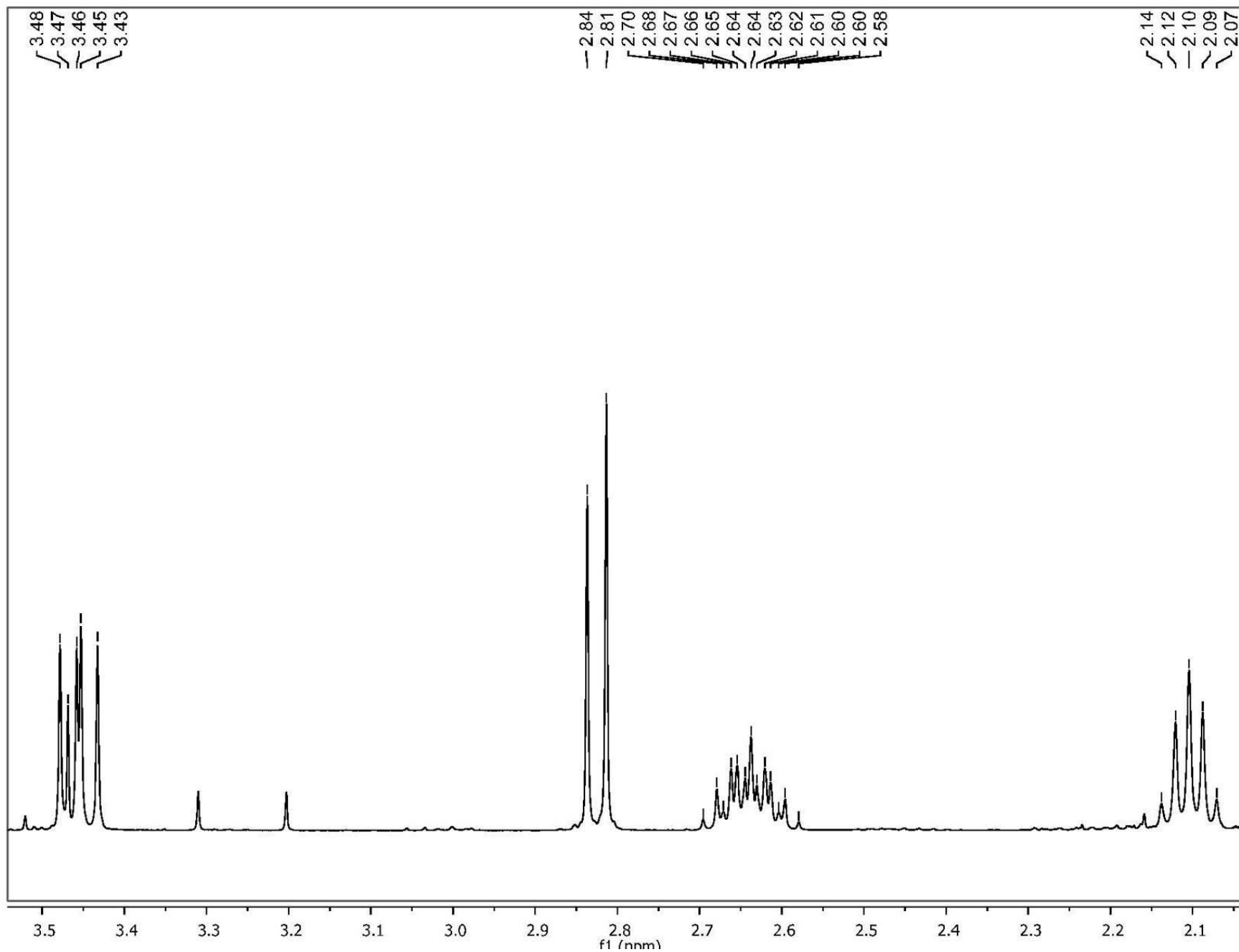
(22*S*, 23*R*, 24*R*)-16 $\beta$ , 22:22, 25-diepoxi-23-carboxymethyl-24-methyl-26,27-dinor-5b-cholestan-3 $\alpha$ -ol acetate (11)

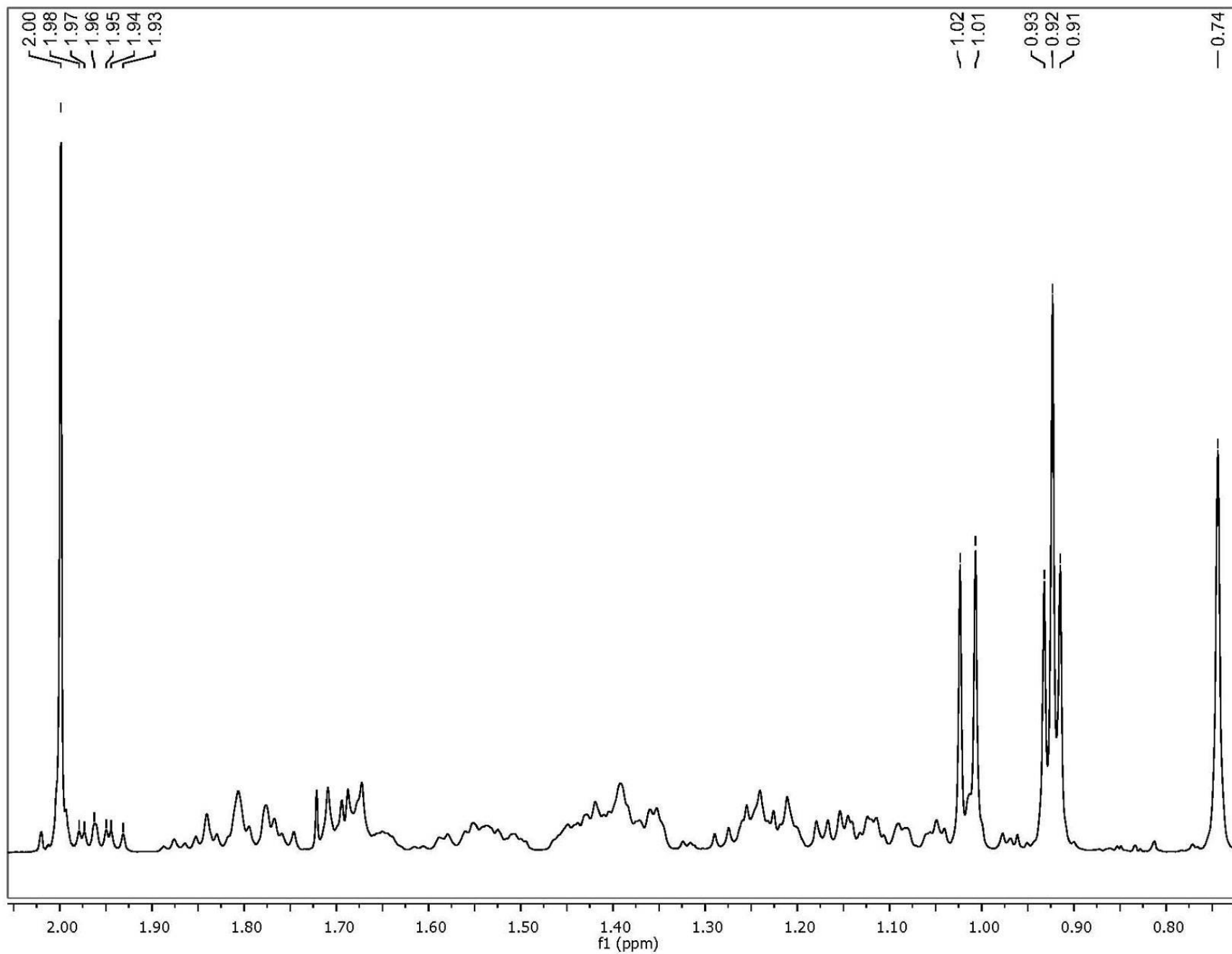


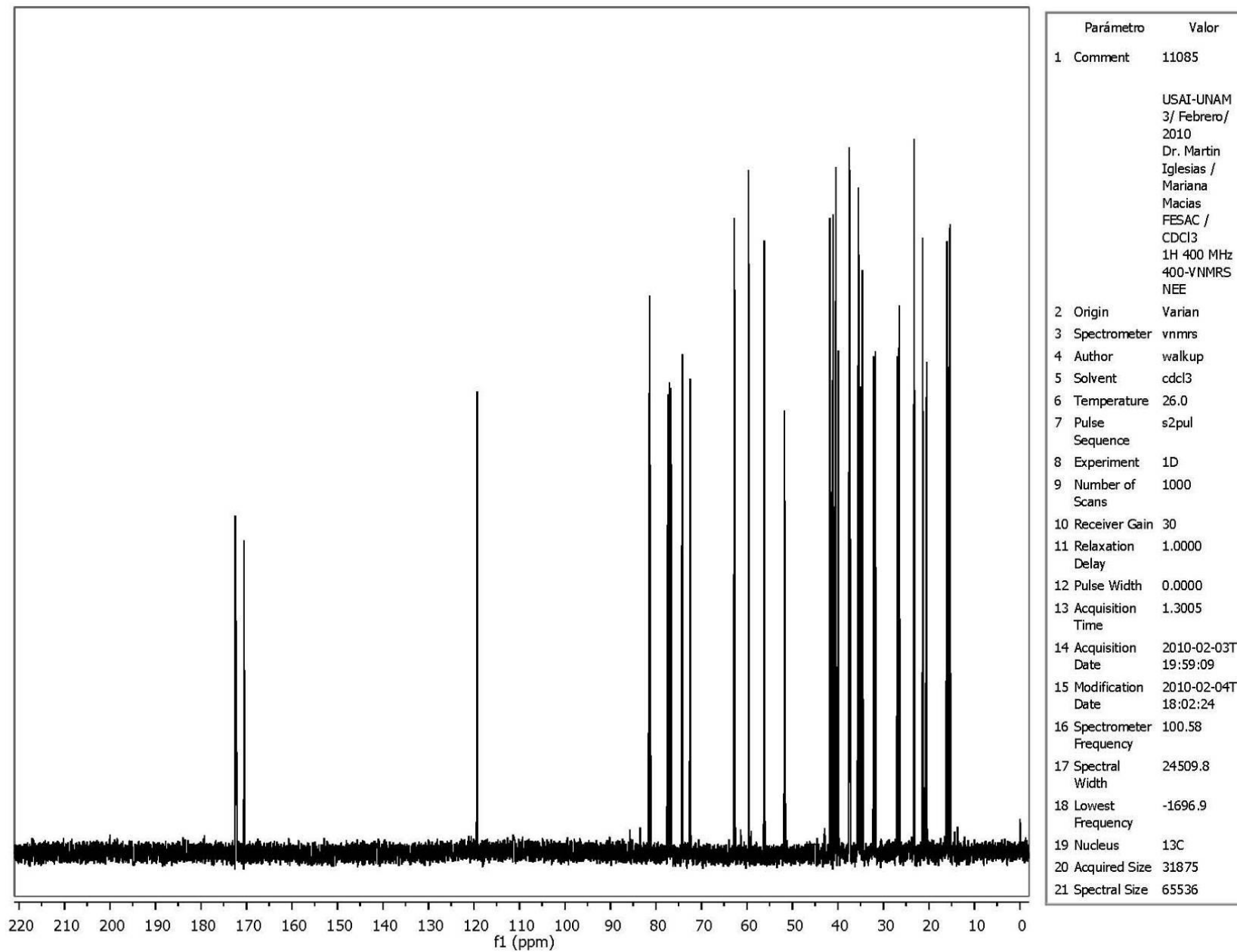
Parámetro	Valor
1 Comment	11085
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2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdd3
6 Temperature	26.0
7 Pulse Sequence	s2pul
8 Experiment	1D
9 Number of Scans	16
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	2.0488
14 Acquisition Date	2010-02-03 T19:58:10
15 Modification Date	2010-02-04 T18:02:12
16 Spectrometer Frequency	399.95
17 Spectral Width	3906.2
18 Lowest Frequency	-502.9
19 Nucleus	1H
20 Acquired	8003

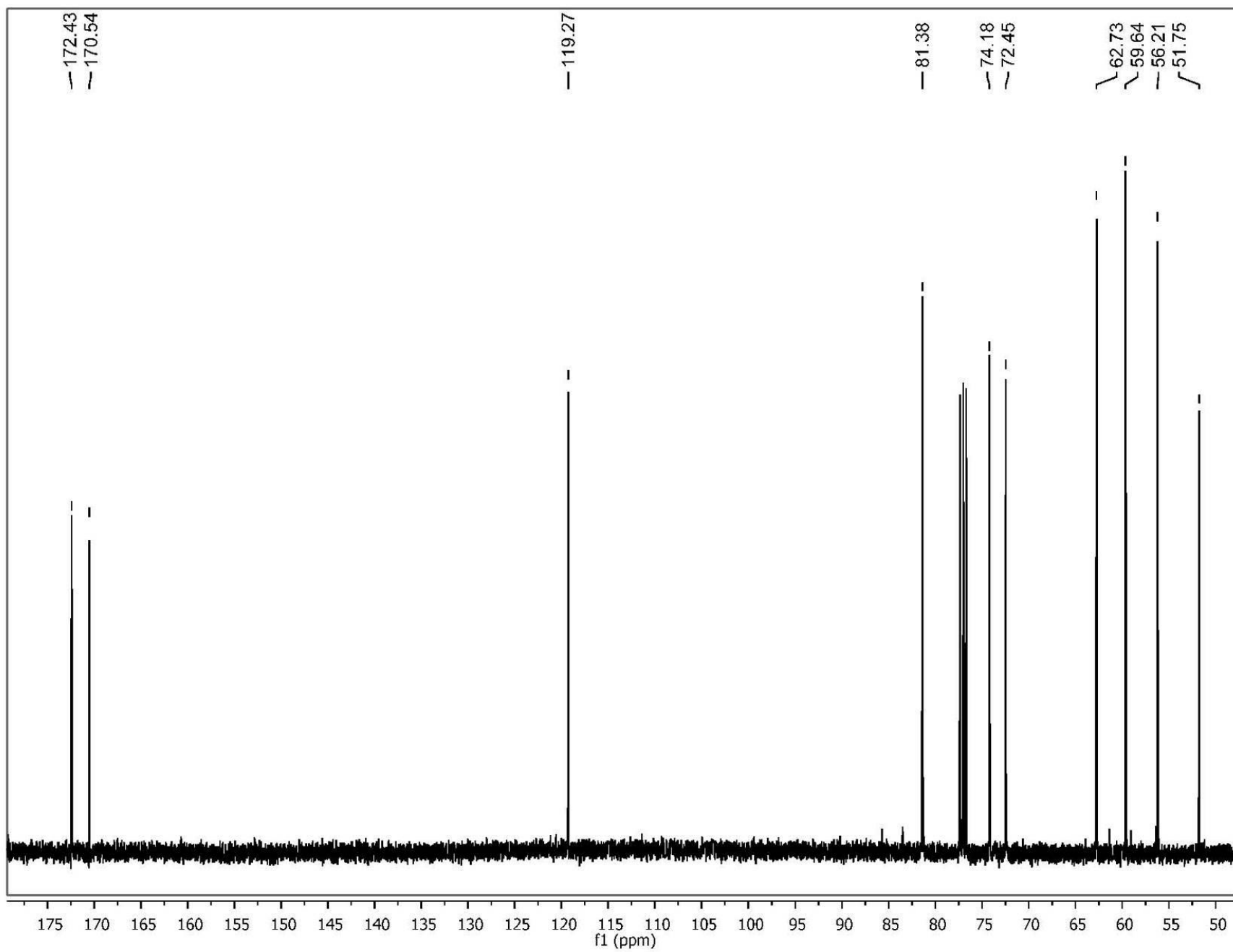


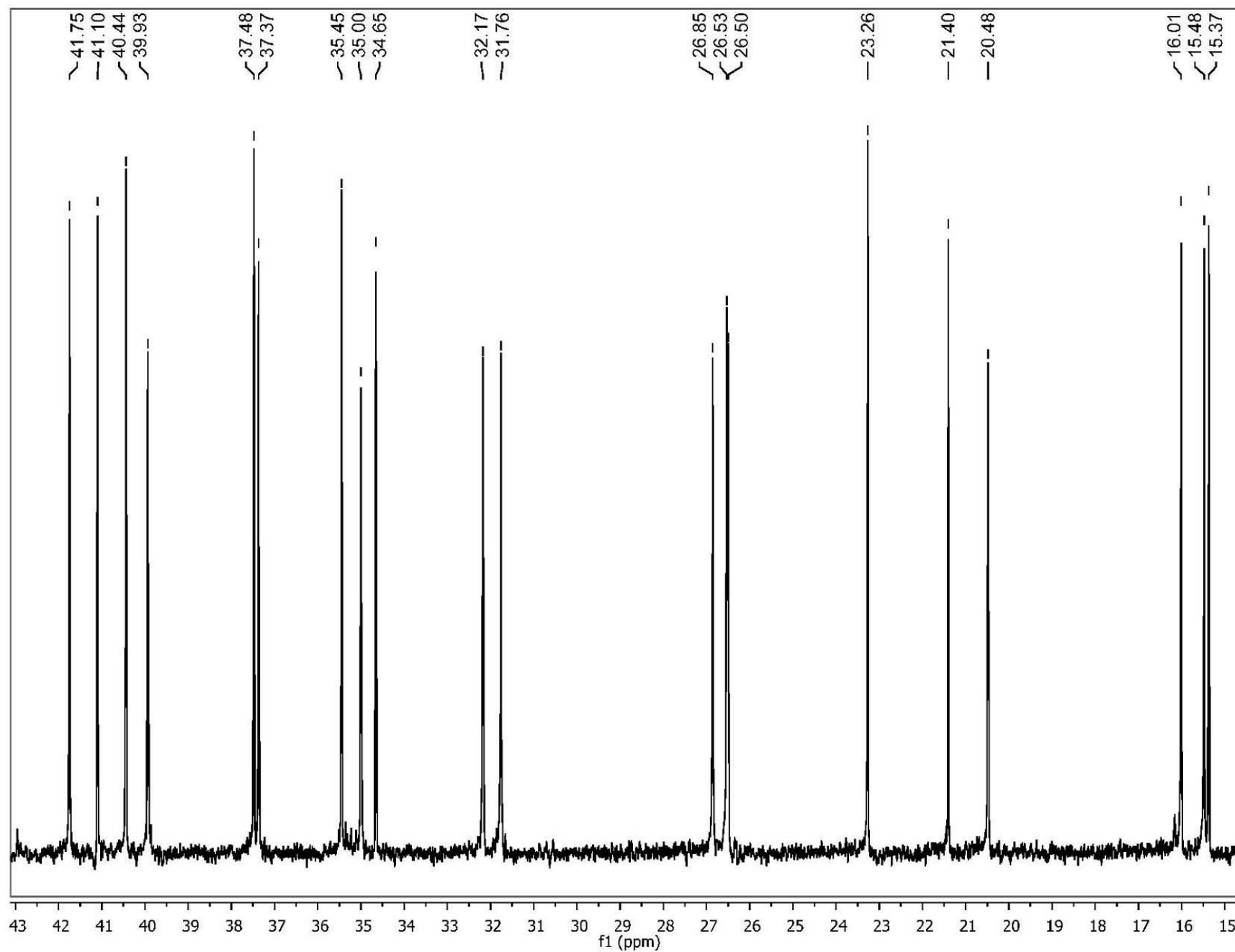




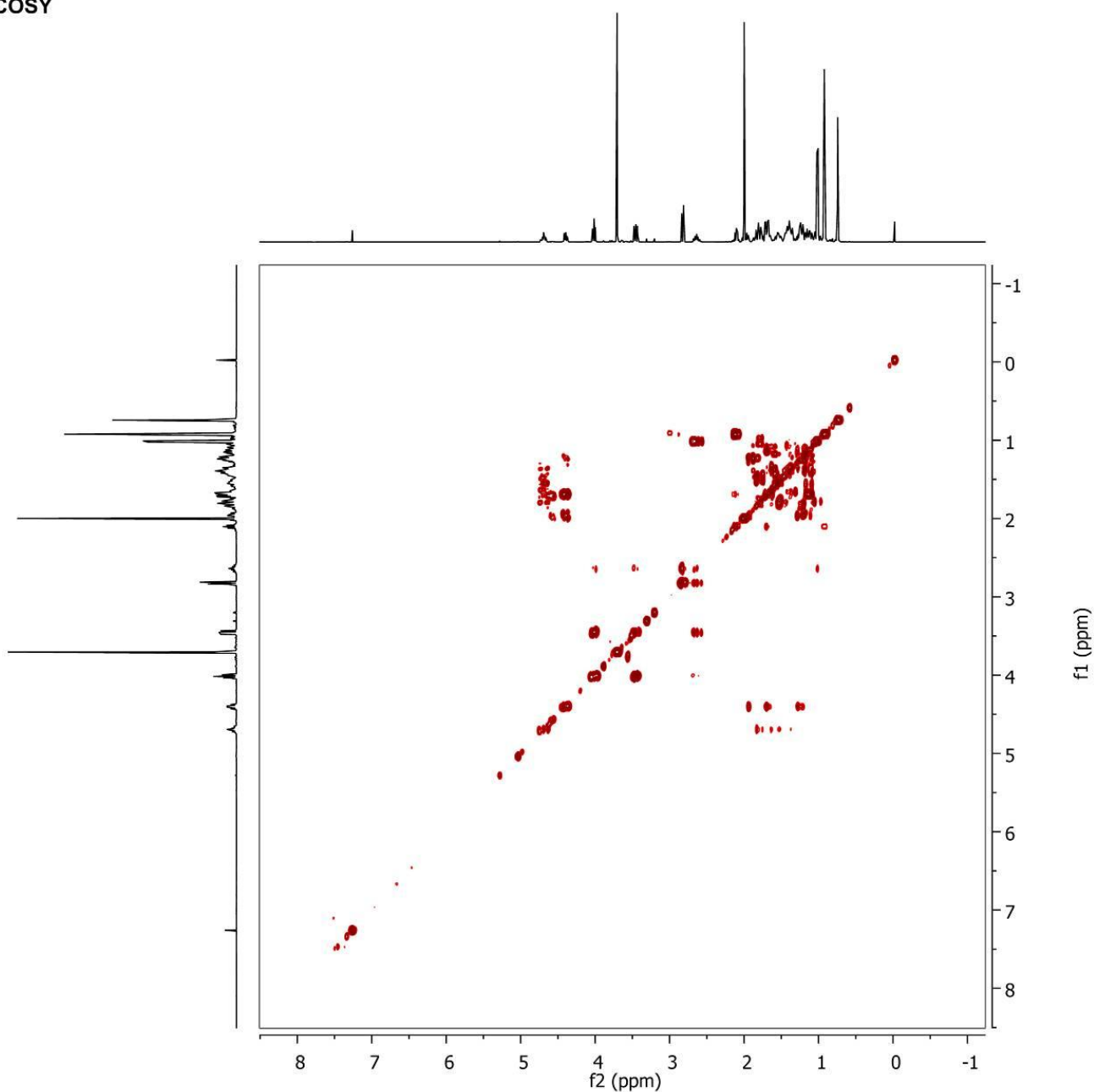




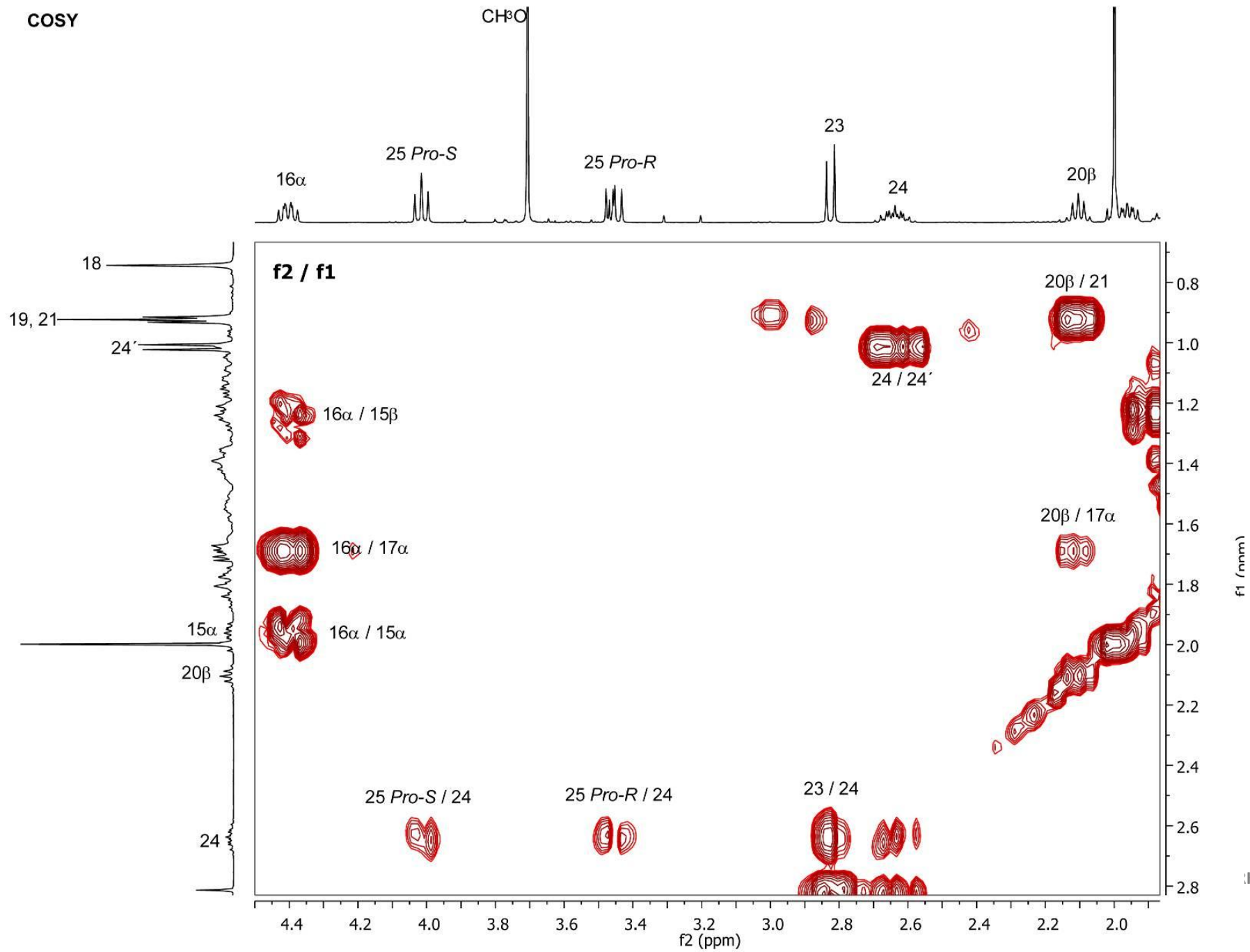




## COSY

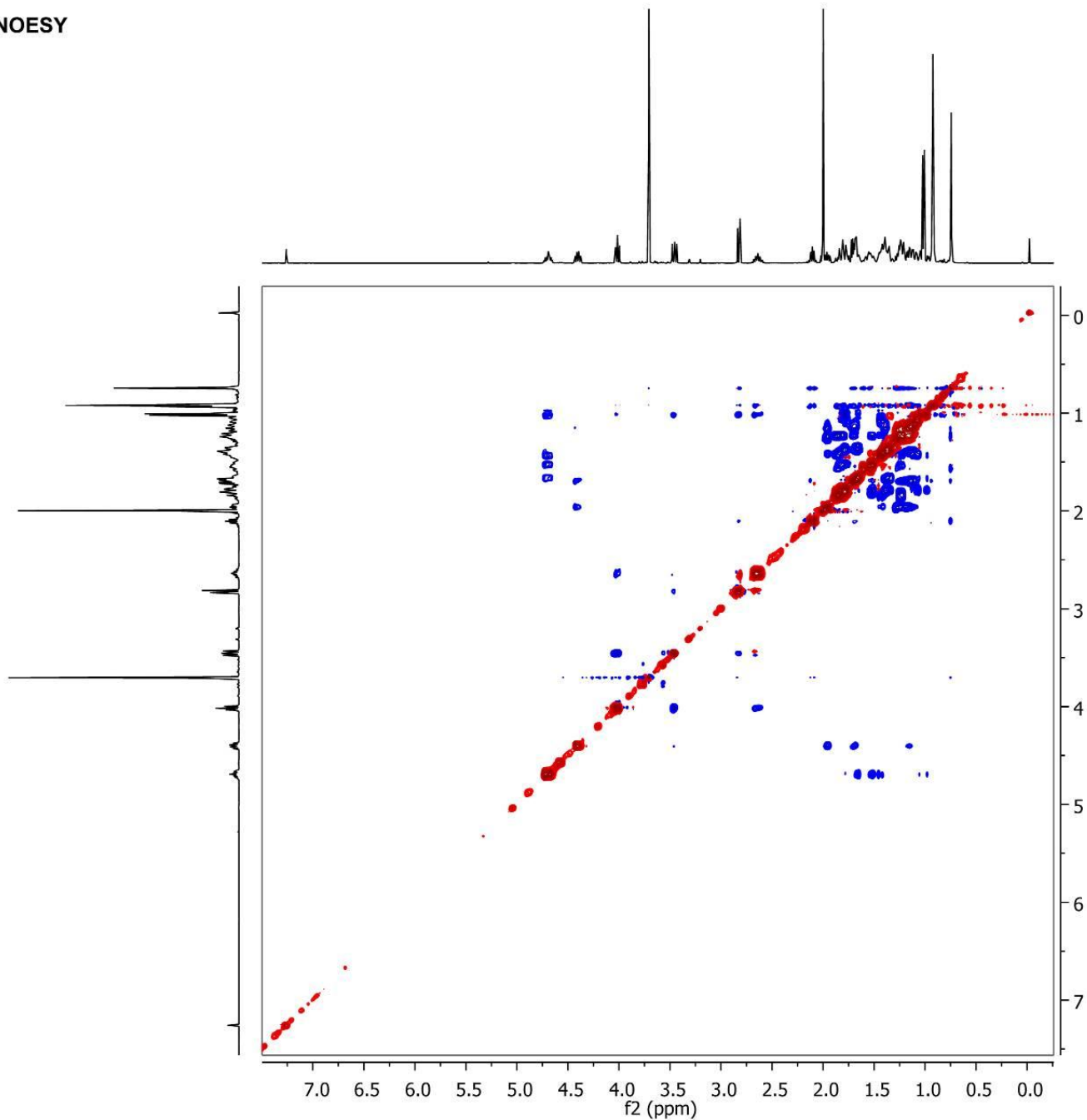


Parámetro	Valor (f2, f1)
1 Comment	11085
	USAI-UNAM 3/ Febrero/ 2010 Dr. Martin Iglesias / Mariana Macias FESAC / CDCI3 1H 400 MHz 400-VNMRS NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	vnmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gCOSY
8 Experiment	2D-COSY
9 Number of Scans	4
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1311
14 Acquisition Date	2010-02-03T23:27:39
15 Modification Date	2010-02-04T18:03:10
16 Spectrometer Frequency	(399.95, 399.95)
17 Spectral Width	(3906.2, 3906.2)
18 Lowest Frequency	(-503.4, -503.0)
19 Nucleus	(1H, 1H)
20 Acquired Size	(512, 256)
21 Spectral Size	(512, 512)



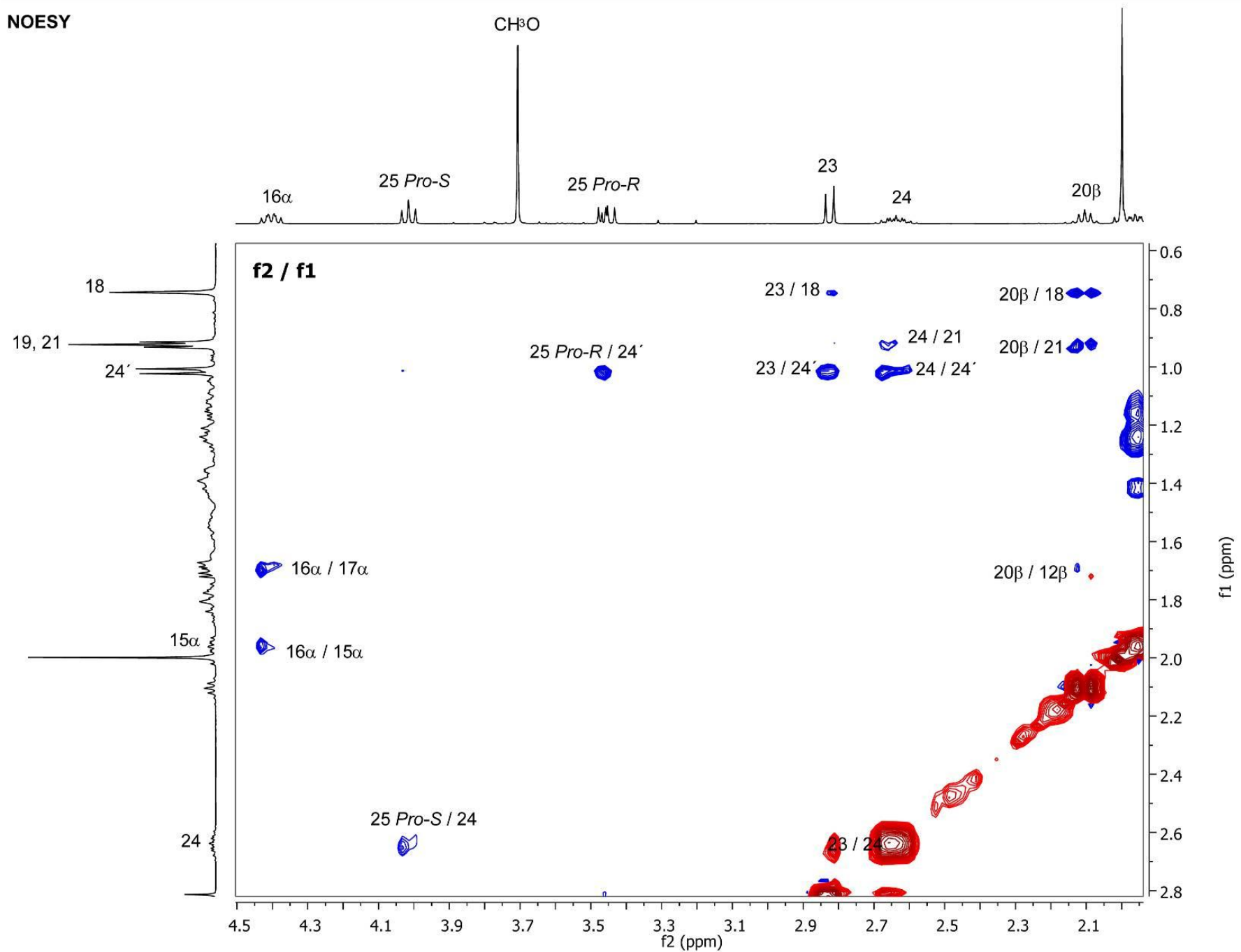


NOESY

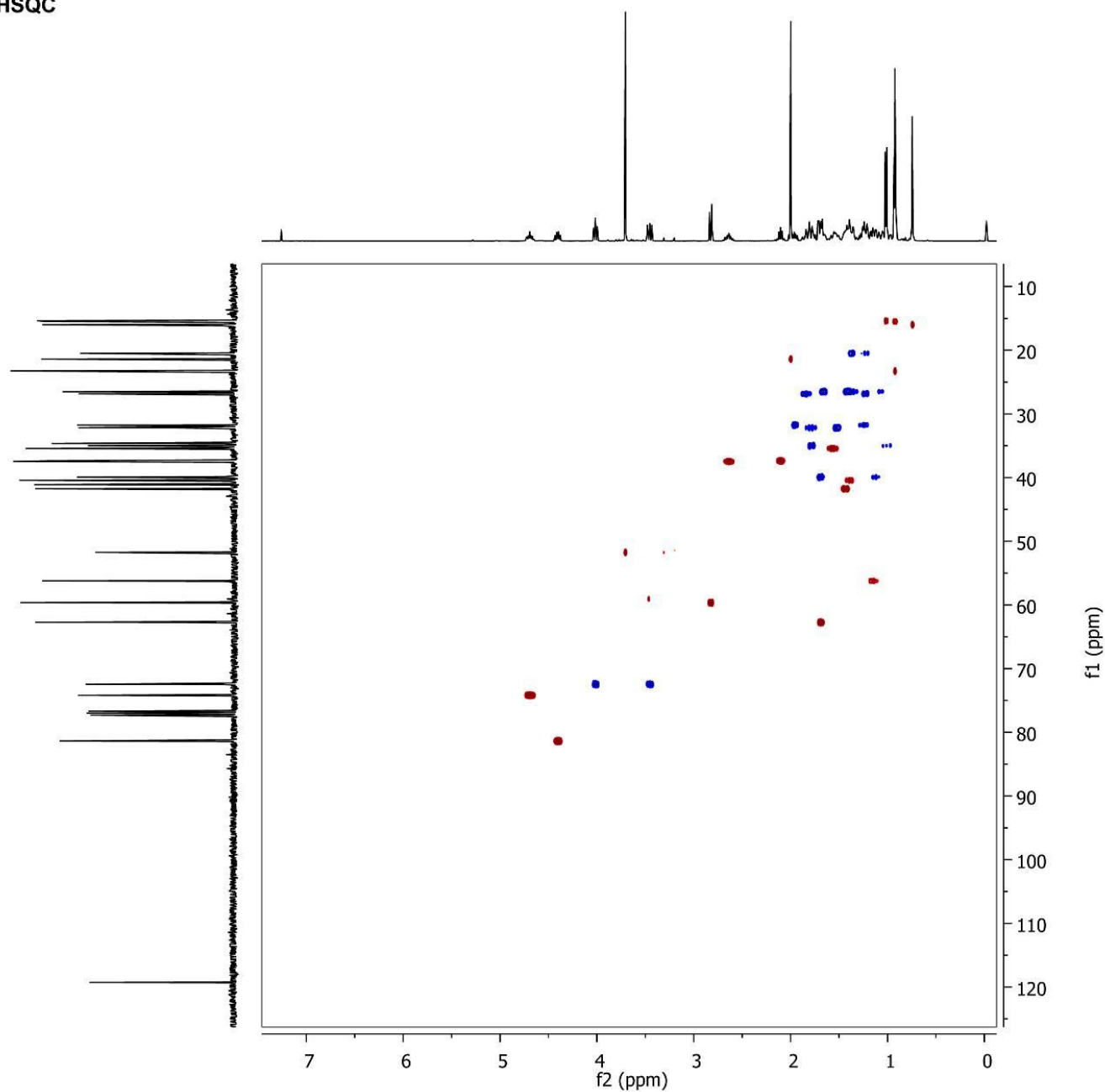


Parámetro	Valor (f2, f1)
1 Comment	11085
	USAI-UNAM 3/ Febrero/ 2010 Dr. Martin Iglesias / Mariana Macias FESAC / CDCl3 1H 400 MHz 400-VNMR5 NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	NOESY
8 Experiment	2D-NOESY
9 Number of Scans	8
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1311
14 Acquisition Date	2010-02-03T2 3:48:09
15 Modification Date	2010-02-04T1 8:03:24
16 Spectrometer Frequency	(399.95, 399.95)
17 Spectral Width	(3906.2, 3906.2)
18 Lowest Frequency	(-500.6, -502.6)
19 Nucleus	(1H, 1H)
20 Acquired Size	(512, 256)
21 Spectral Size	(512, 512)

NOESY

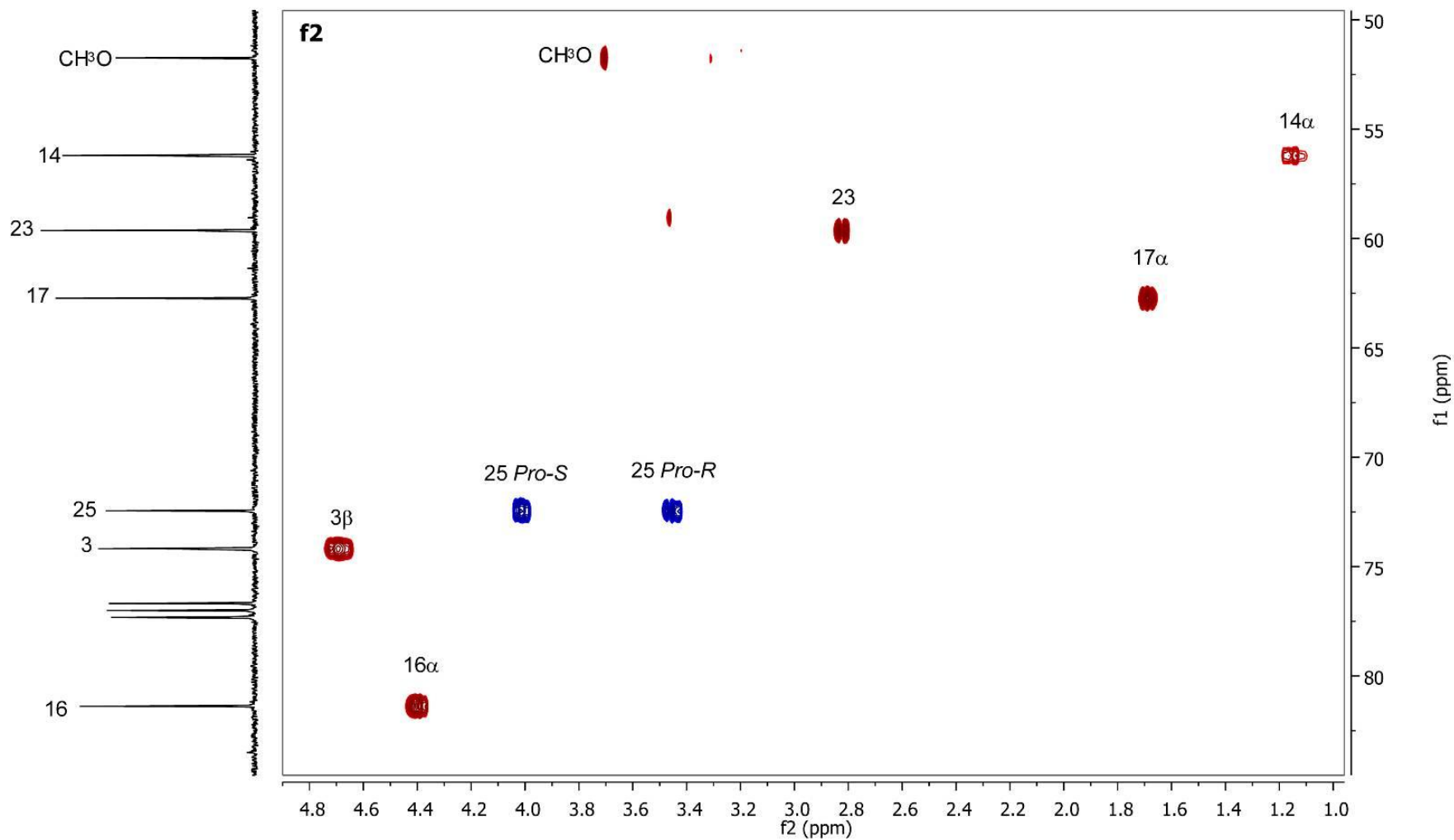
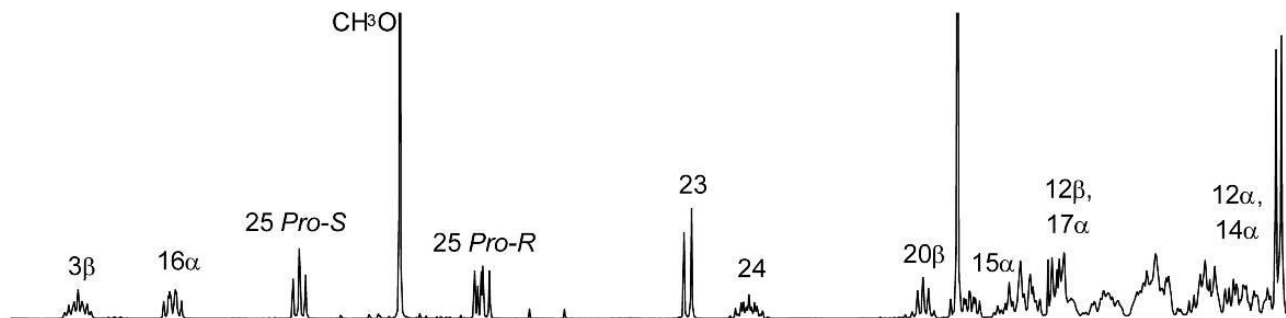


## HSQC

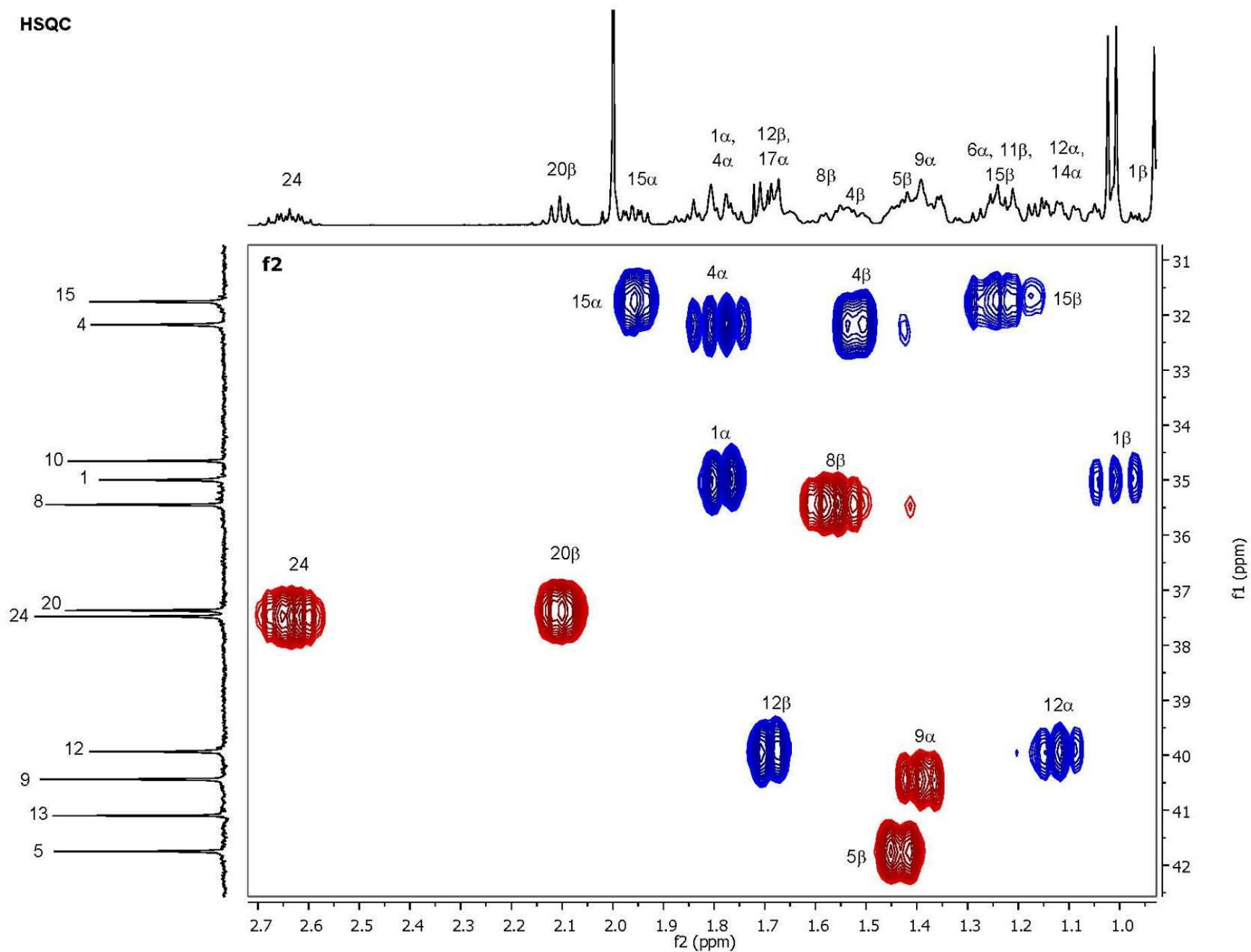


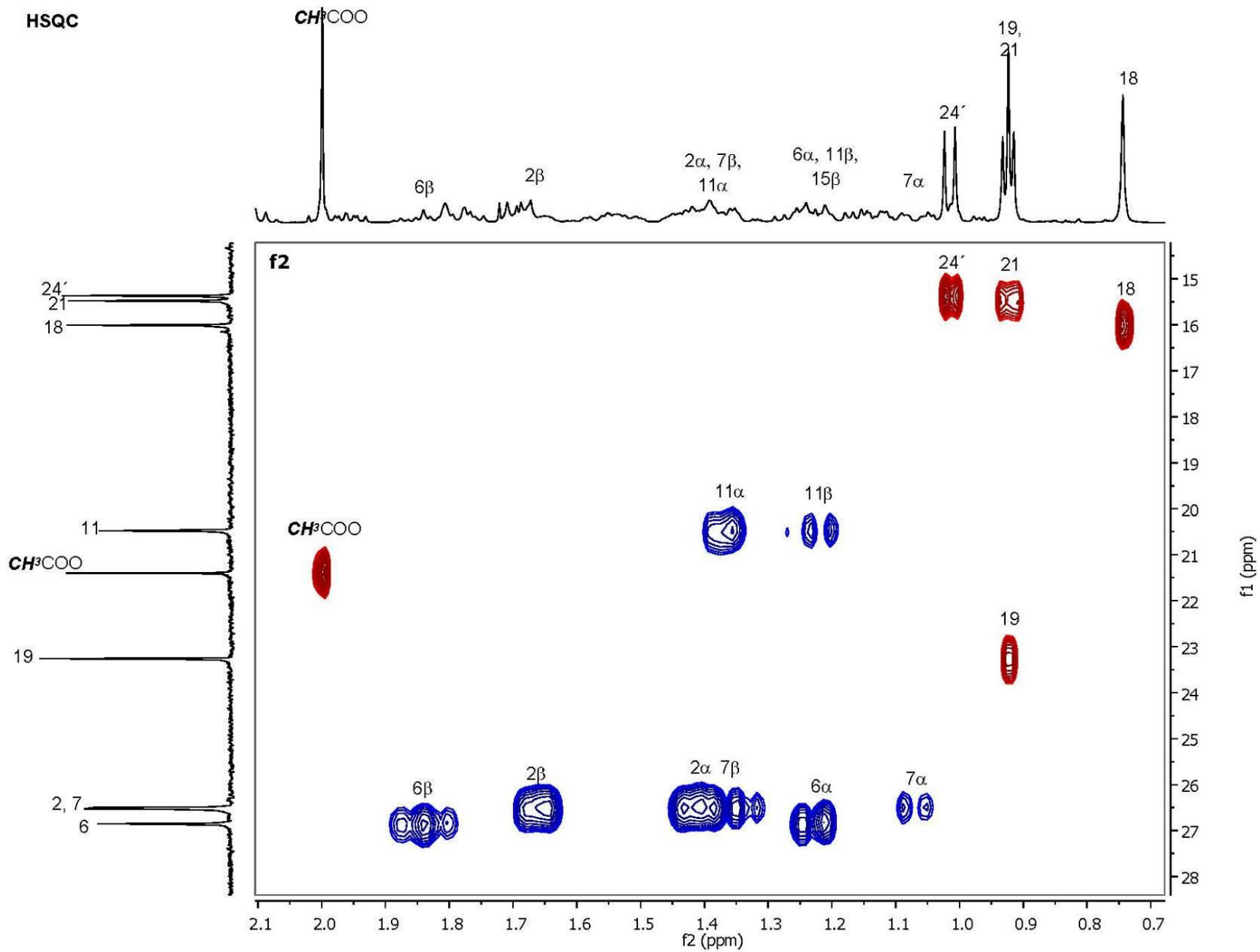
Parámetro	Valor (f2, f1)
1 Comment	11085
	USAI-UNAM 3/ Febrero/ 2010 Dr. Martin Iglesias / Mariana Macias FESAC / CDCI3 1H 400 MHz 400-VNMRS NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	vnmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHSQC
8 Experiment	2D-HSQC- EDITED
9 Number of Scans	8
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1994
14 Acquisition Date	2010-02-03T20: :37:35
15 Modification Date	2010-02-04T18: :02:44
16 Spectrometer Frequency	(399.95, 100.58)
17 Spectral Width	(3906.2, 17097.7)
18 Lowest Frequency	(-503.0, -1008.5)
19 Nucleus	(1H, 13C)
20 Acquired Size	(779, 256)

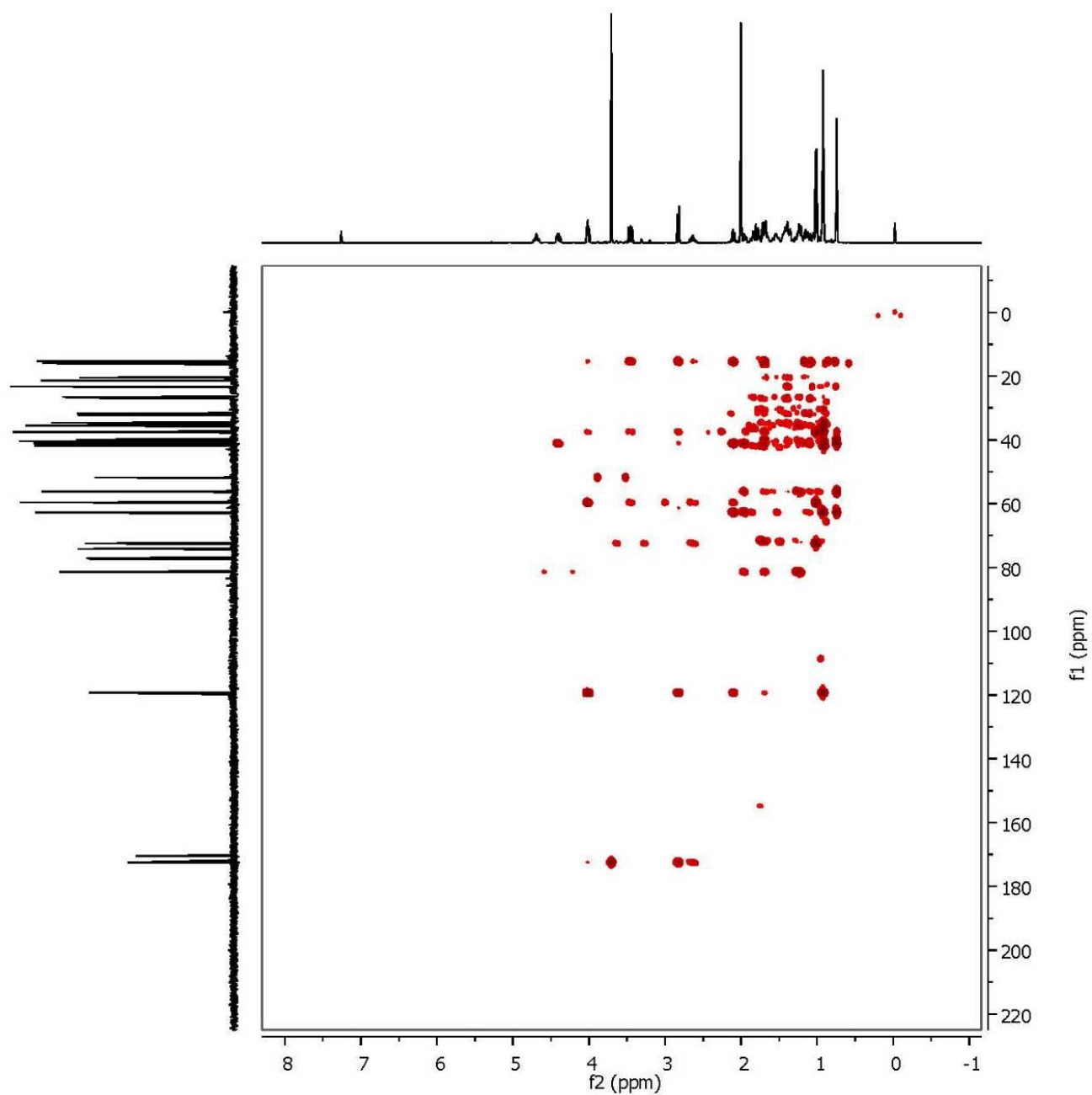
HSQC



HSQC



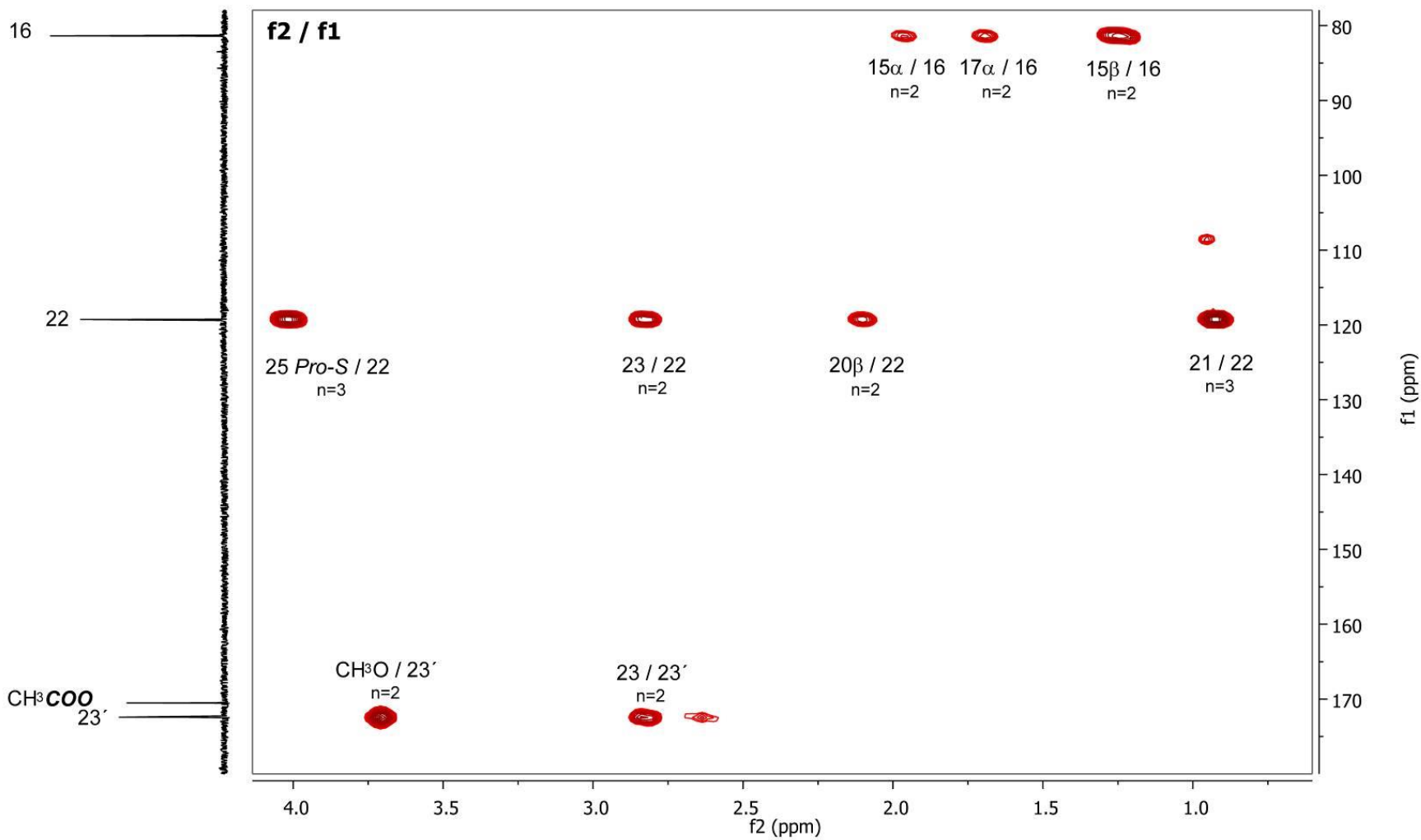
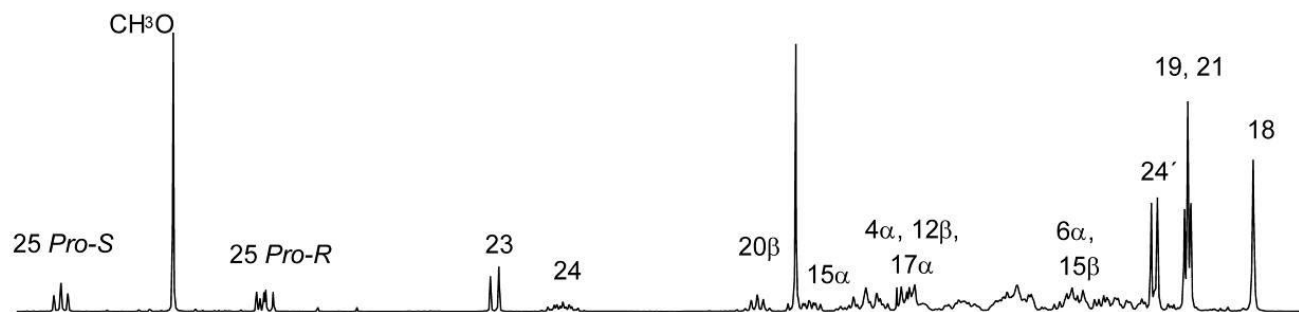




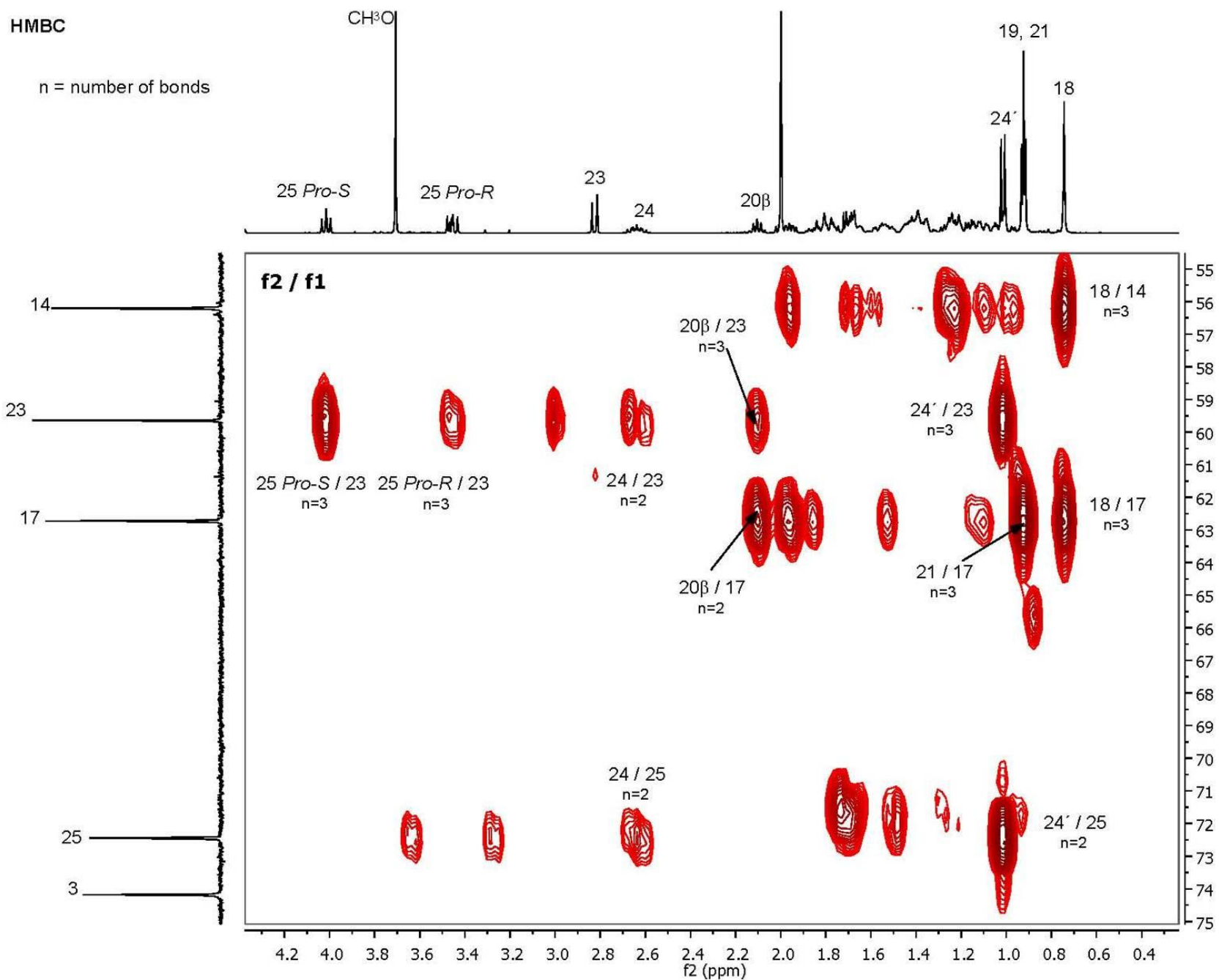
Parámetro	Valor (f2, f1)
1 Comment	11398  USAI-UNAM 9/ ABRIL/ 2010 Dr. Martin Iglesias / Mariana Macias FES-30H/ CDCl3 1H 400 MHz 400-MR NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHMBC
8 Experiment	2D-HMBC
9 Number of Scans	16
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1280
14 Acquisition Date	2010-04-10T01:32:15
15 Modification Date	2010-04-10T08:50:22
16 Spectrometer Frequency	(399.72, 100.52)
17 Spectral Width	(3787.9, 24125.5)
18 Lowest Frequency	(-470.2, -1512.3)
19 Nucleus	(1H, 13C)
20 Acquired Size	(485, 400)
21 Spectral Size	(512, 512)

HMBC

n = number of bonds

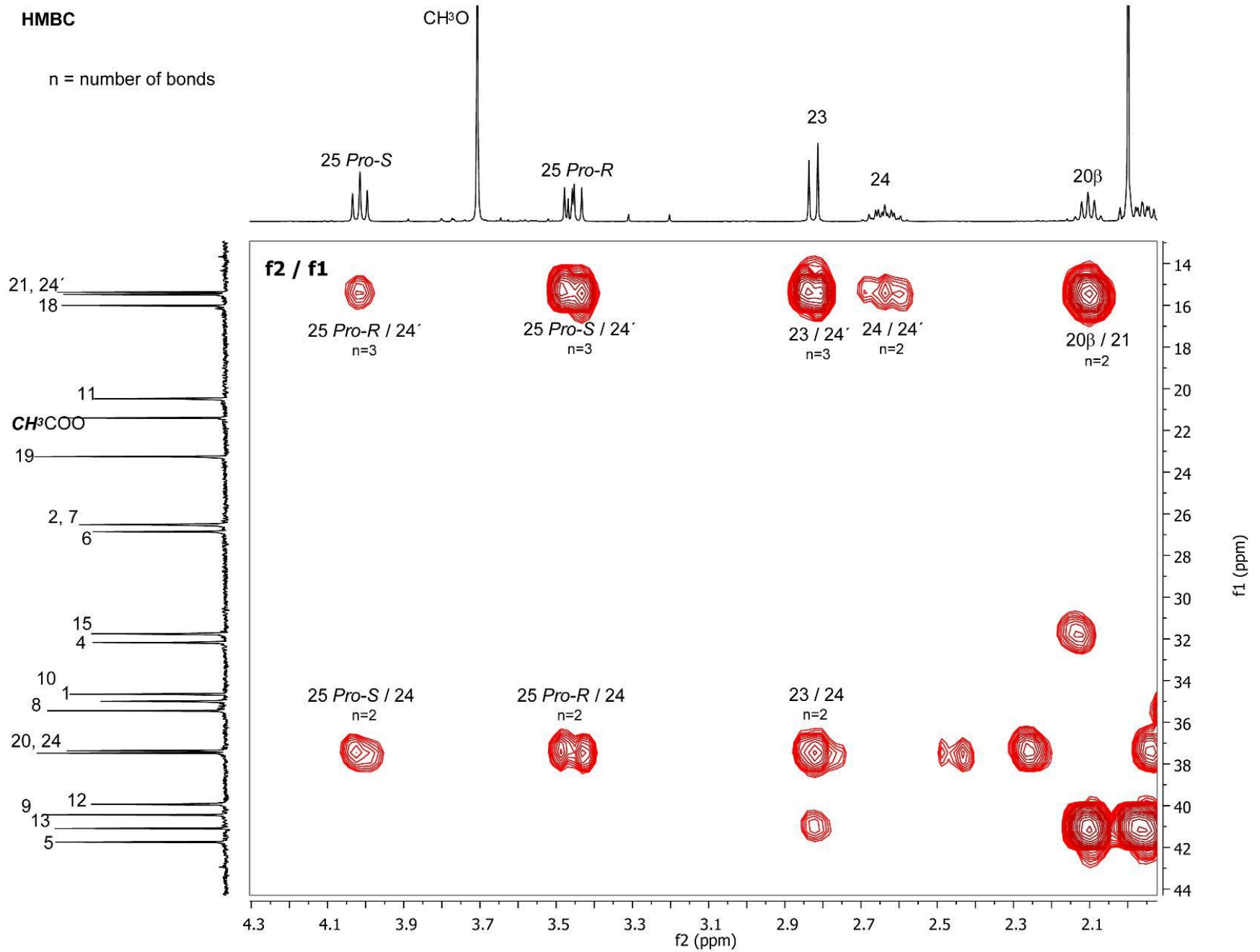






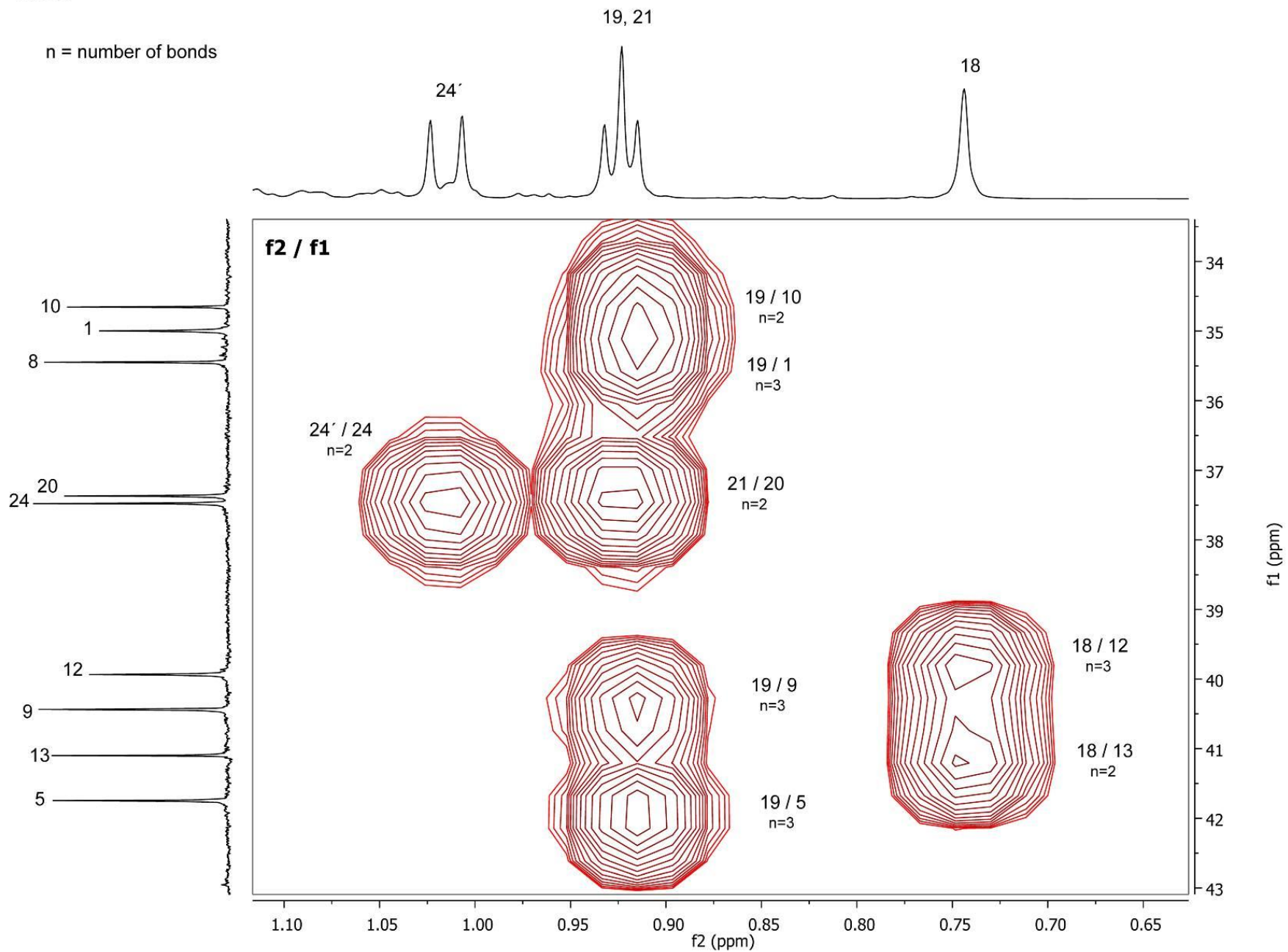
HMBC

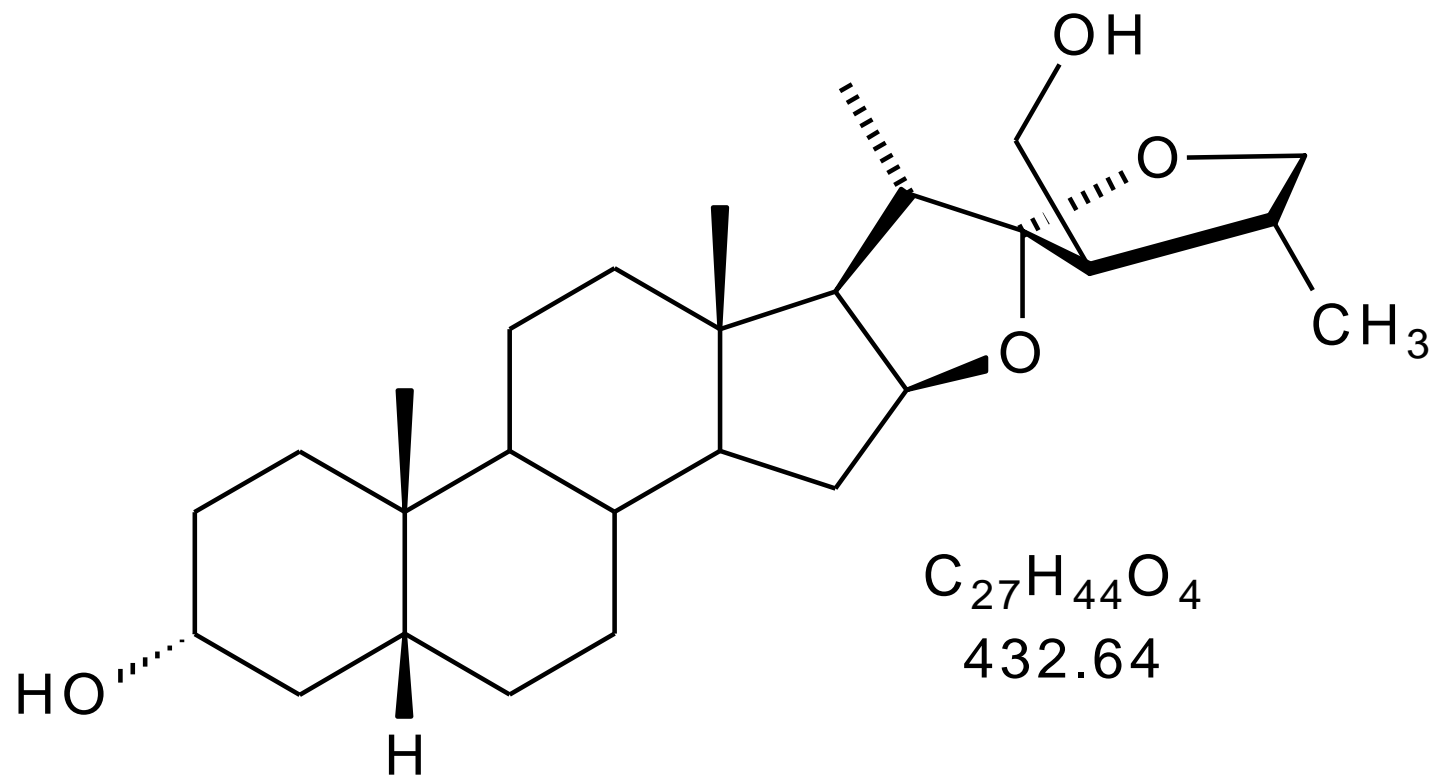
n = number of bonds



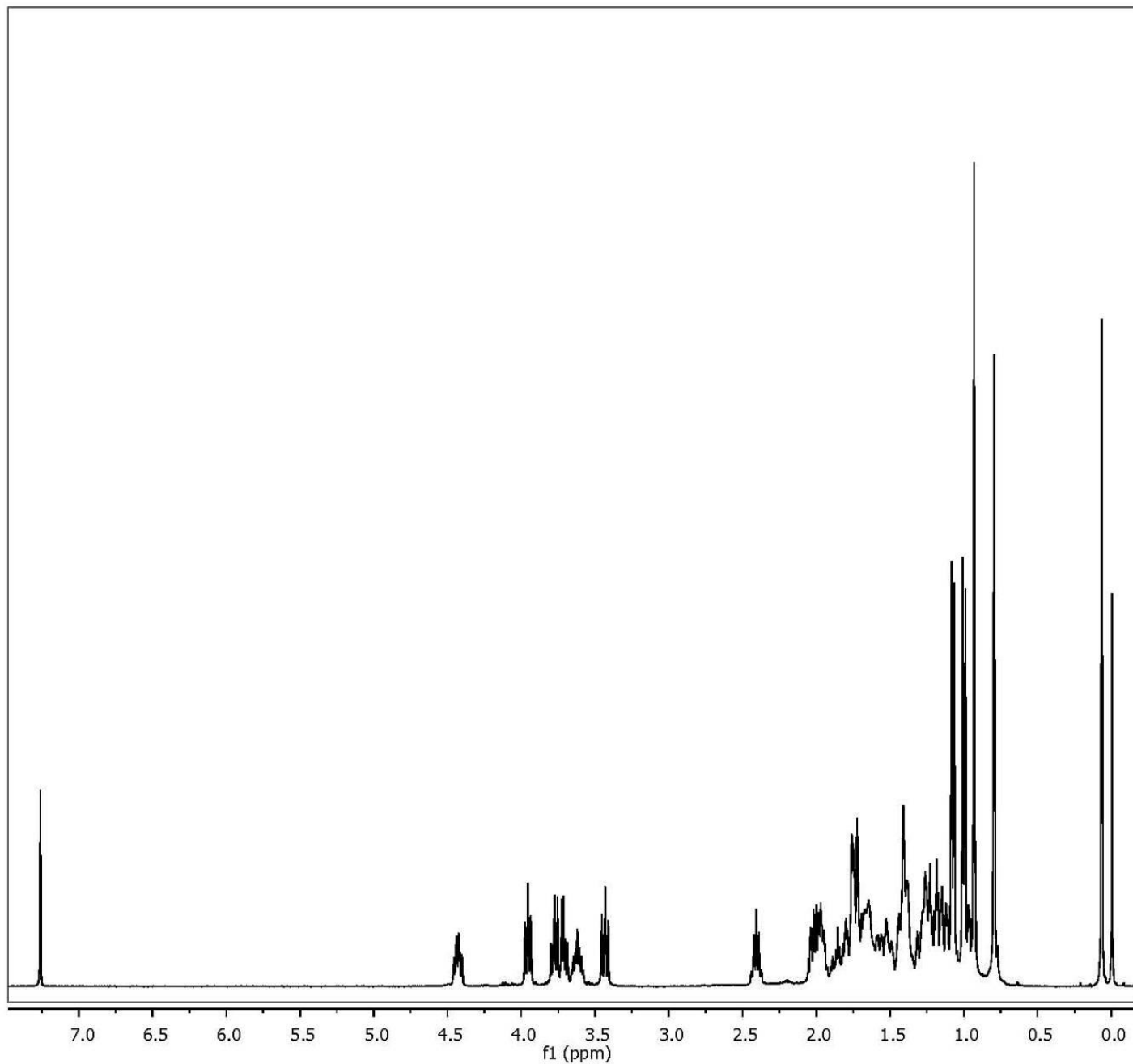
HMBC

n = number of bonds

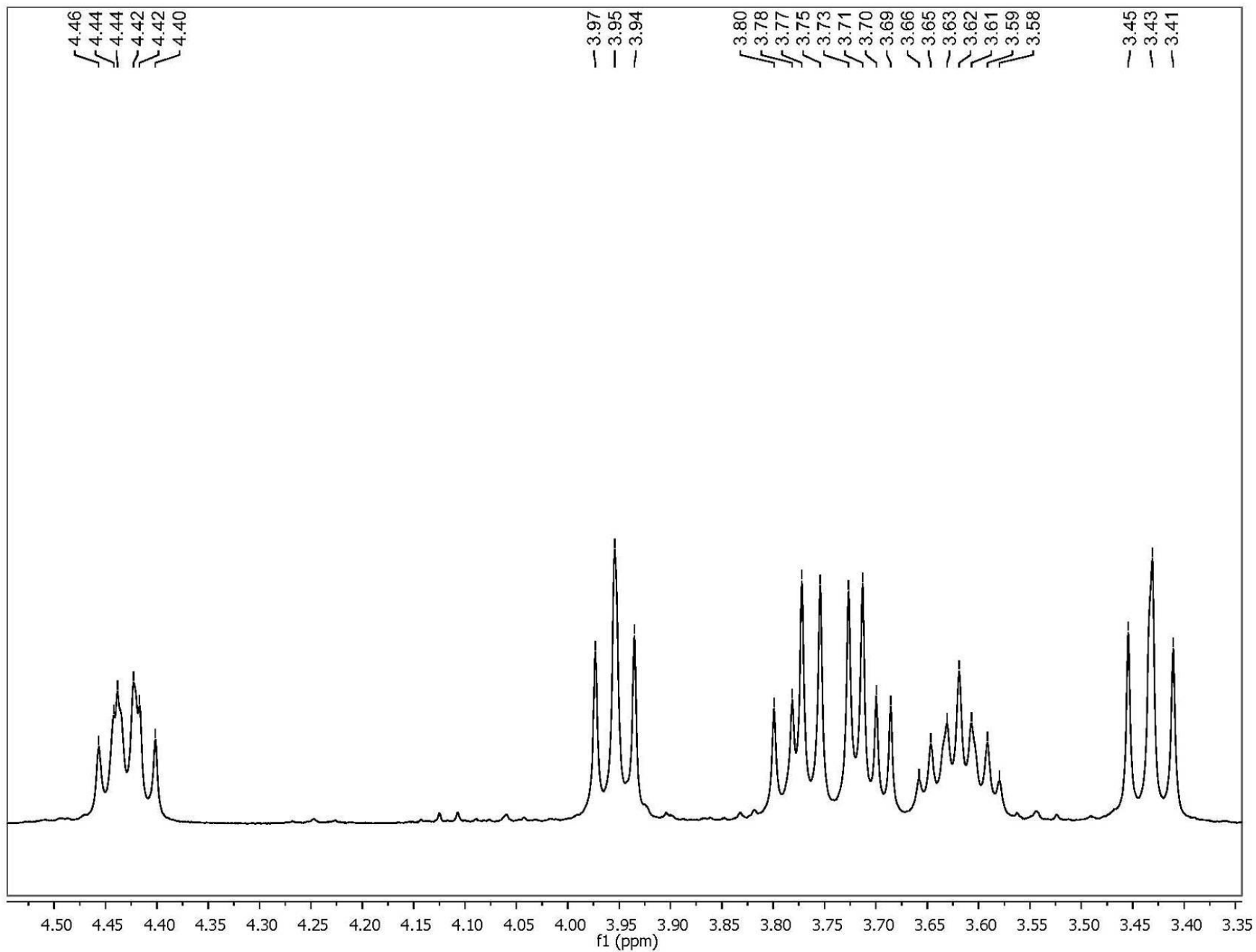


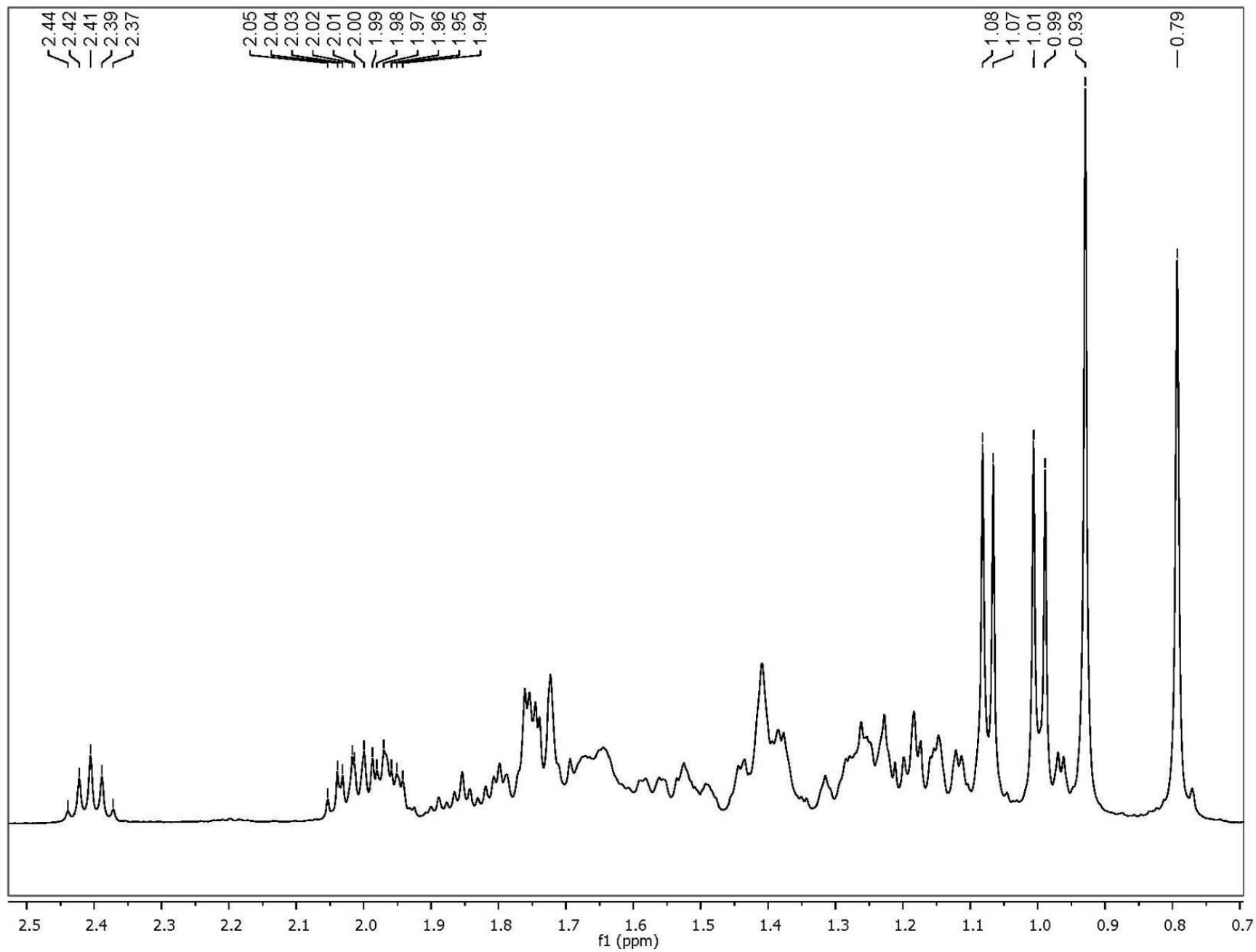


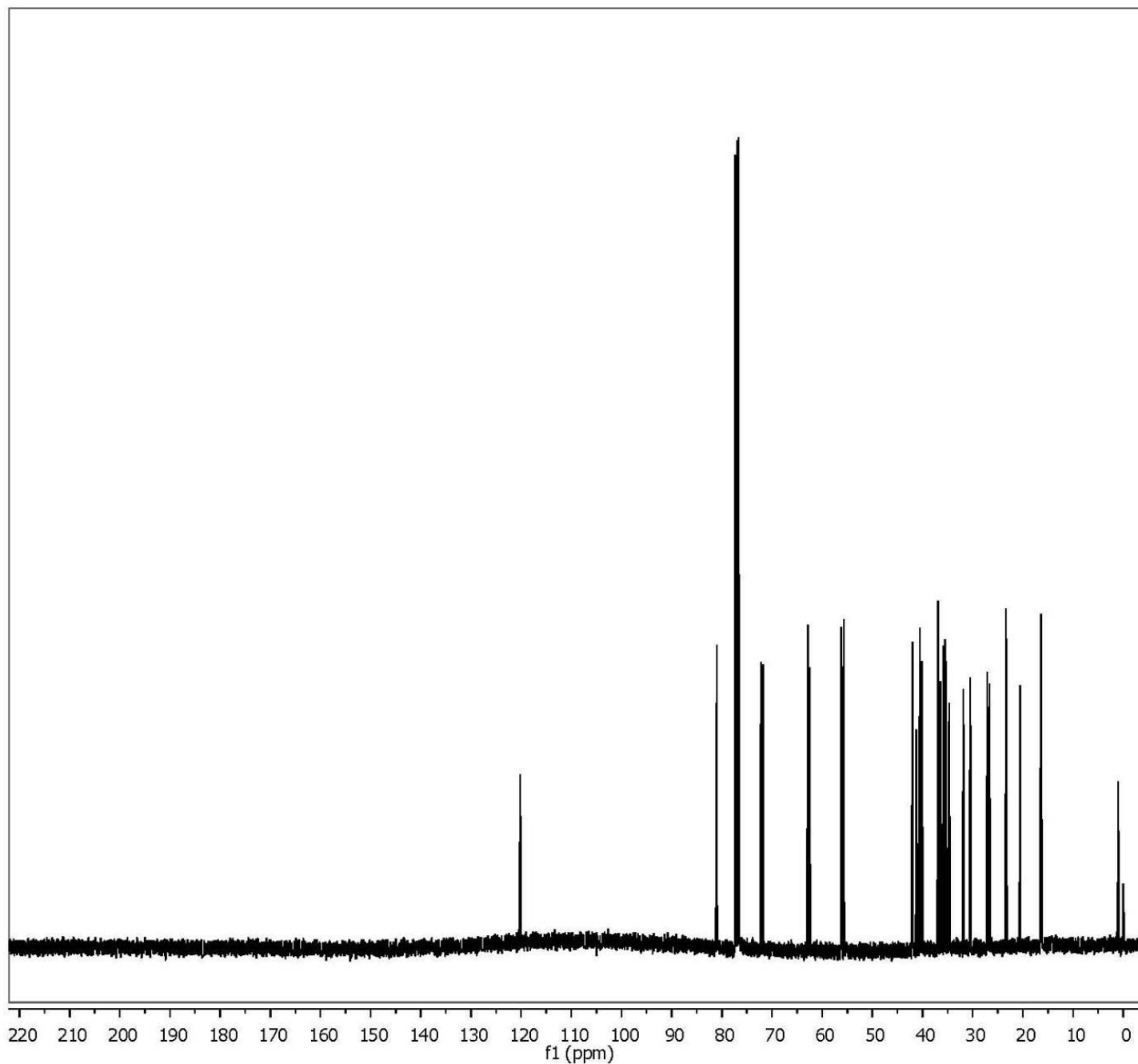
(22*S*, 23*S*, 24*R*)-16 $\beta$ ,22:22,25-diepoxi-23-hydroxymethyl-24-methyl-26,27-dinor-5 $\beta$ -cholestan-3 $\alpha$ -ol (12)



Parámetro	Valor
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCl3 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	s2pul
8 Experiment	1D
9 Number of Scans	16
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	3.5001
14 Acquisition Date	2009-11-17T 17:56:44
15 Modification Date	2009-11-18T 17:05:16
16 Spectrometer Frequency	399.95
17 Spectral Width	3787.9
18 Lowest Frequency	-471.1
19 Nucleus	1H
20 Acquired Size	13258
21 Spectral Size	32768

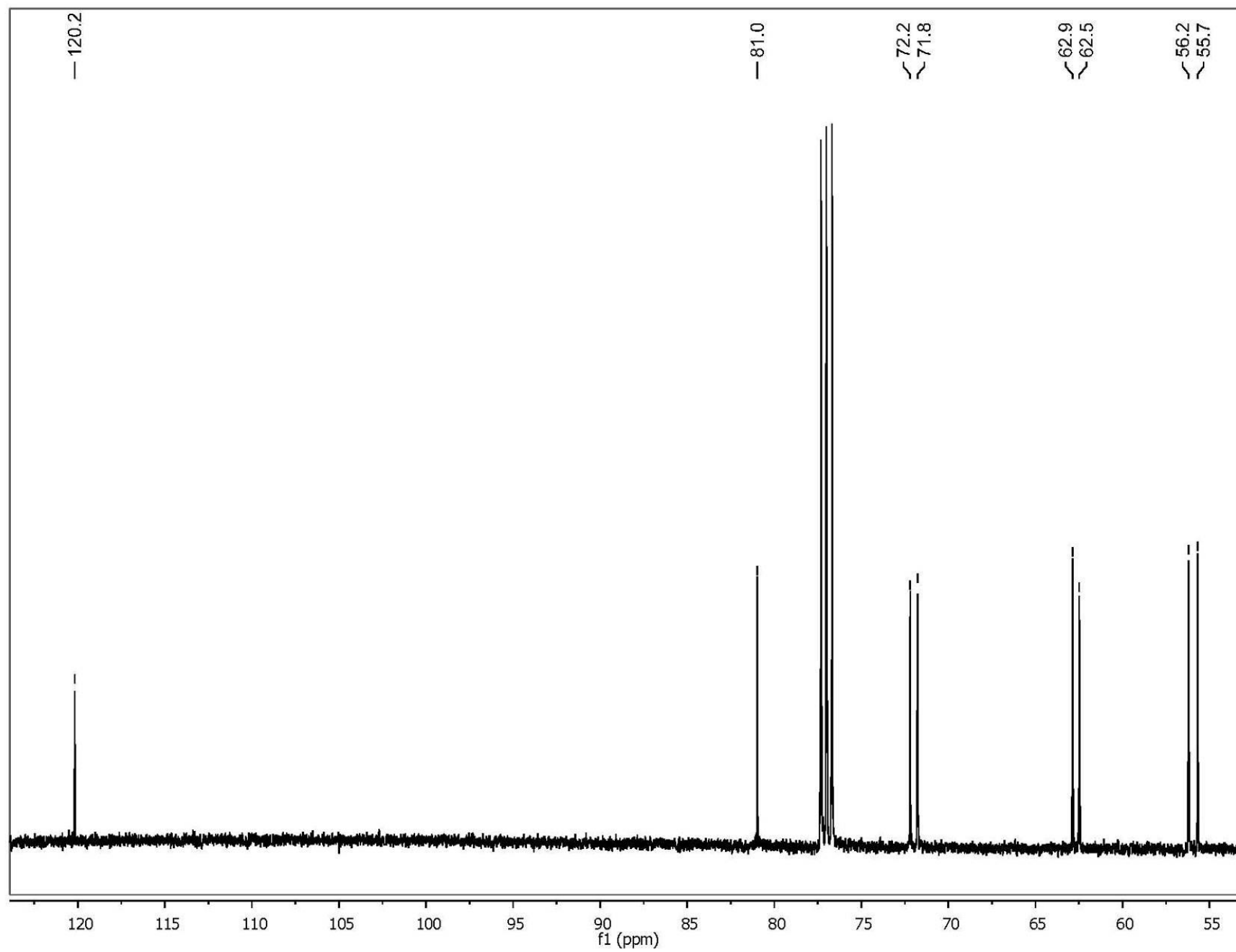


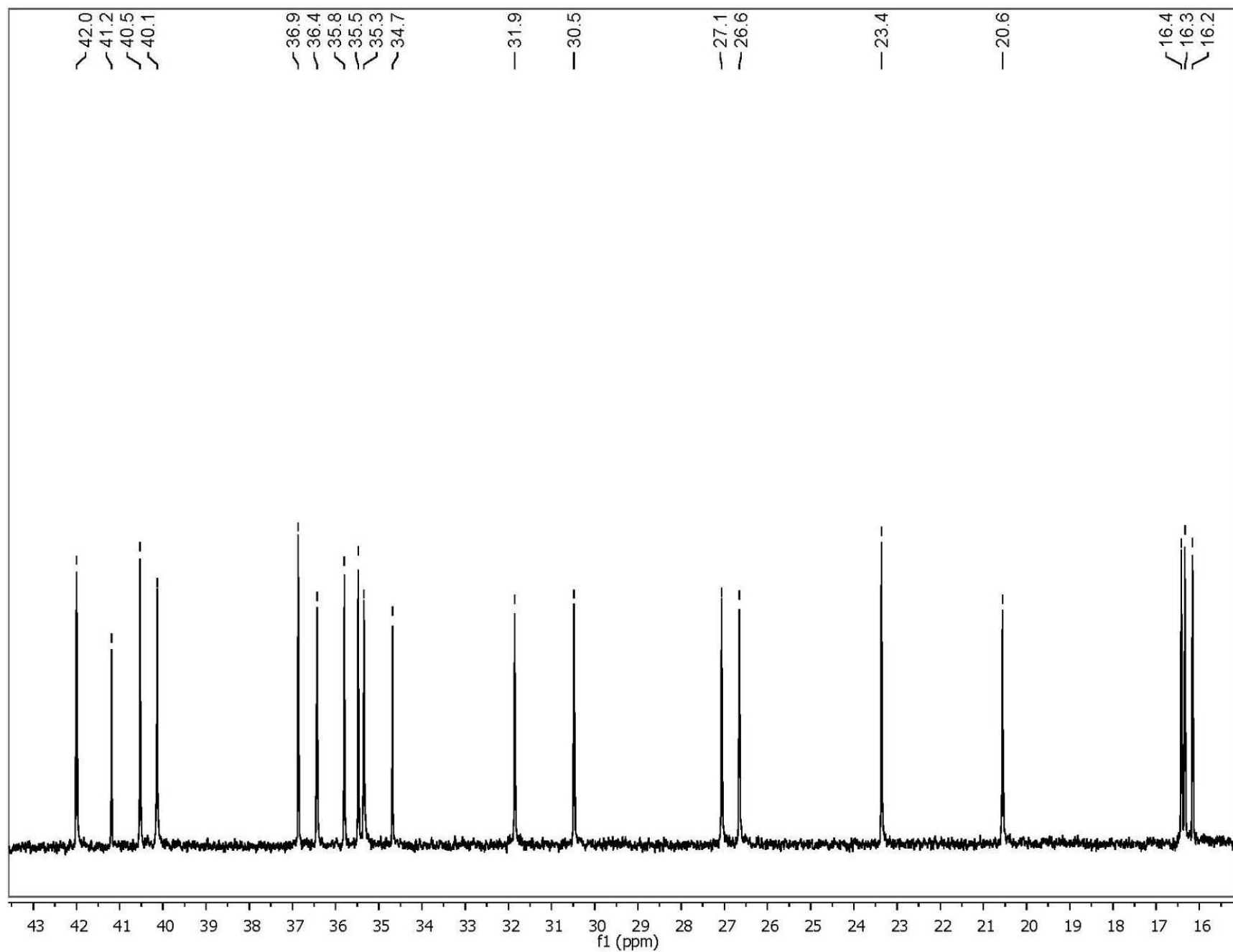




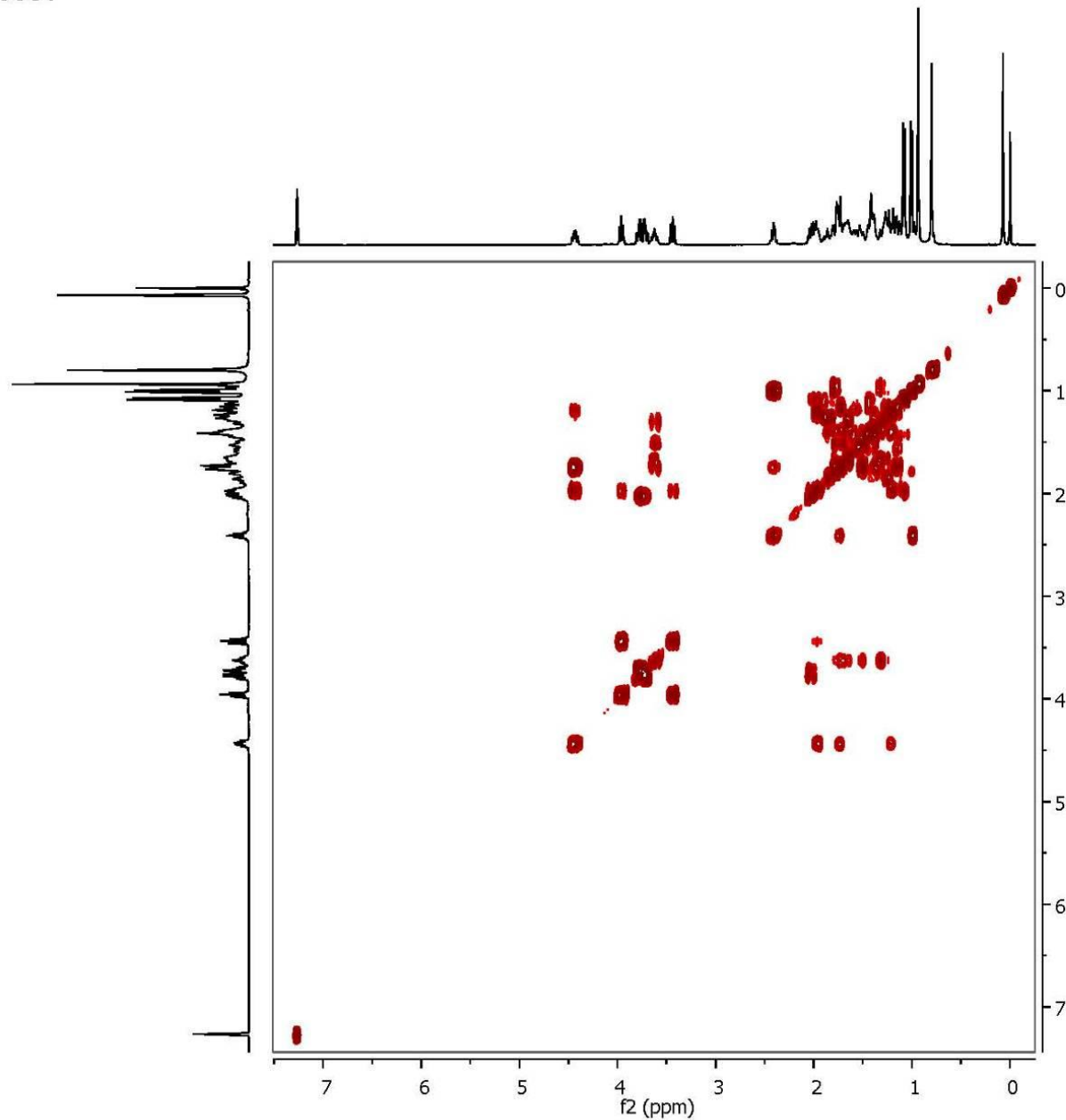
Parámetro	Valor
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCl <sub>3</sub> 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	vnmr5
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	s2pul
8 Experiment	1D
9 Number of Scans	10000
10 Receiver Gain	30
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	1.3005
14 Acquisition Date	2009-11-18 T02:19:40
15 Modification Date	2009-11-18 T17:07:16
16 Spectrometer Frequency	100.58
17 Spectral Width	24509.8
18 Lowest Frequency	-1694.1
19 Nucleus	13C
20 Acquired Size	31875
21 Spectral Size	65536





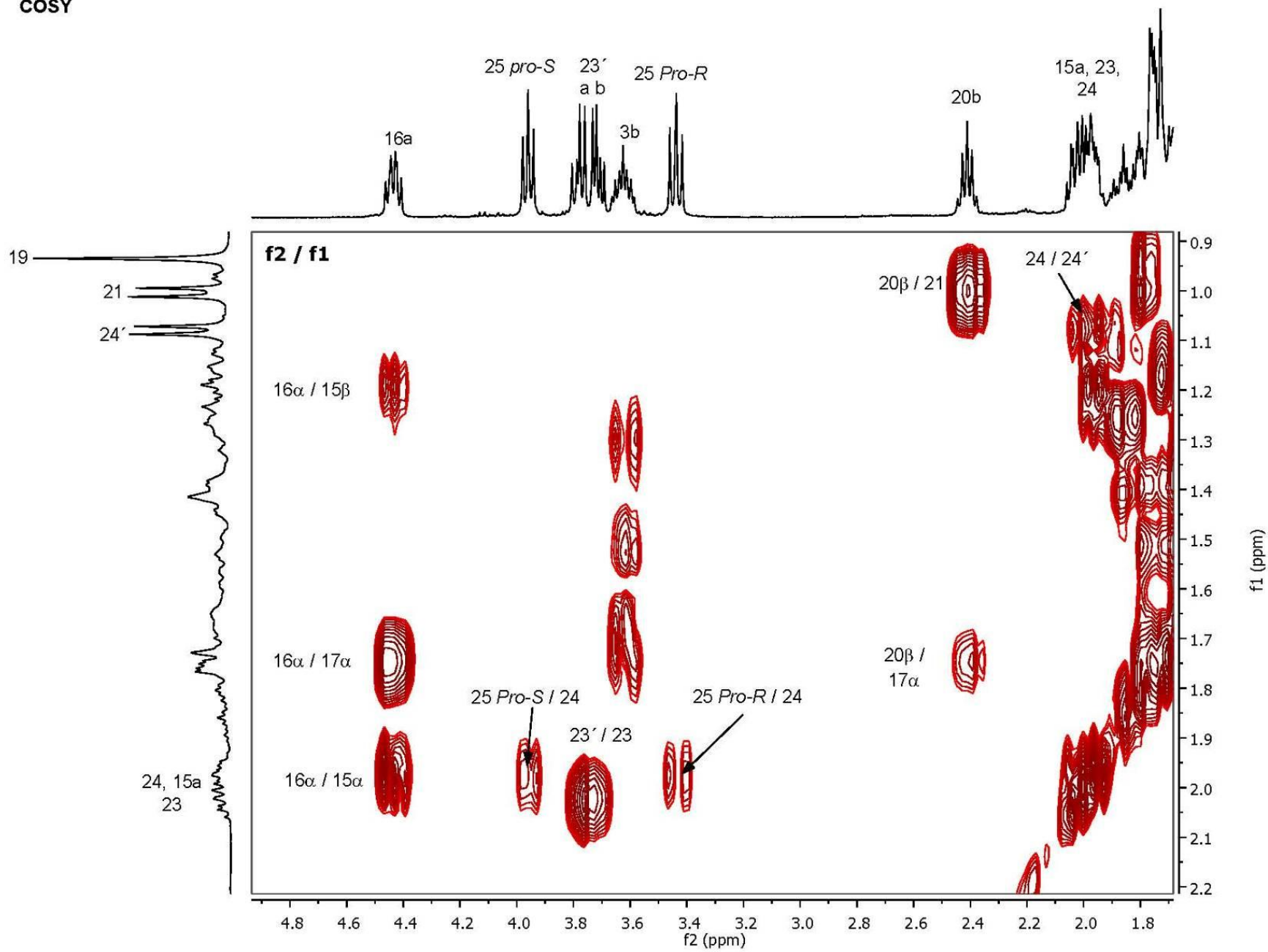


## COSY

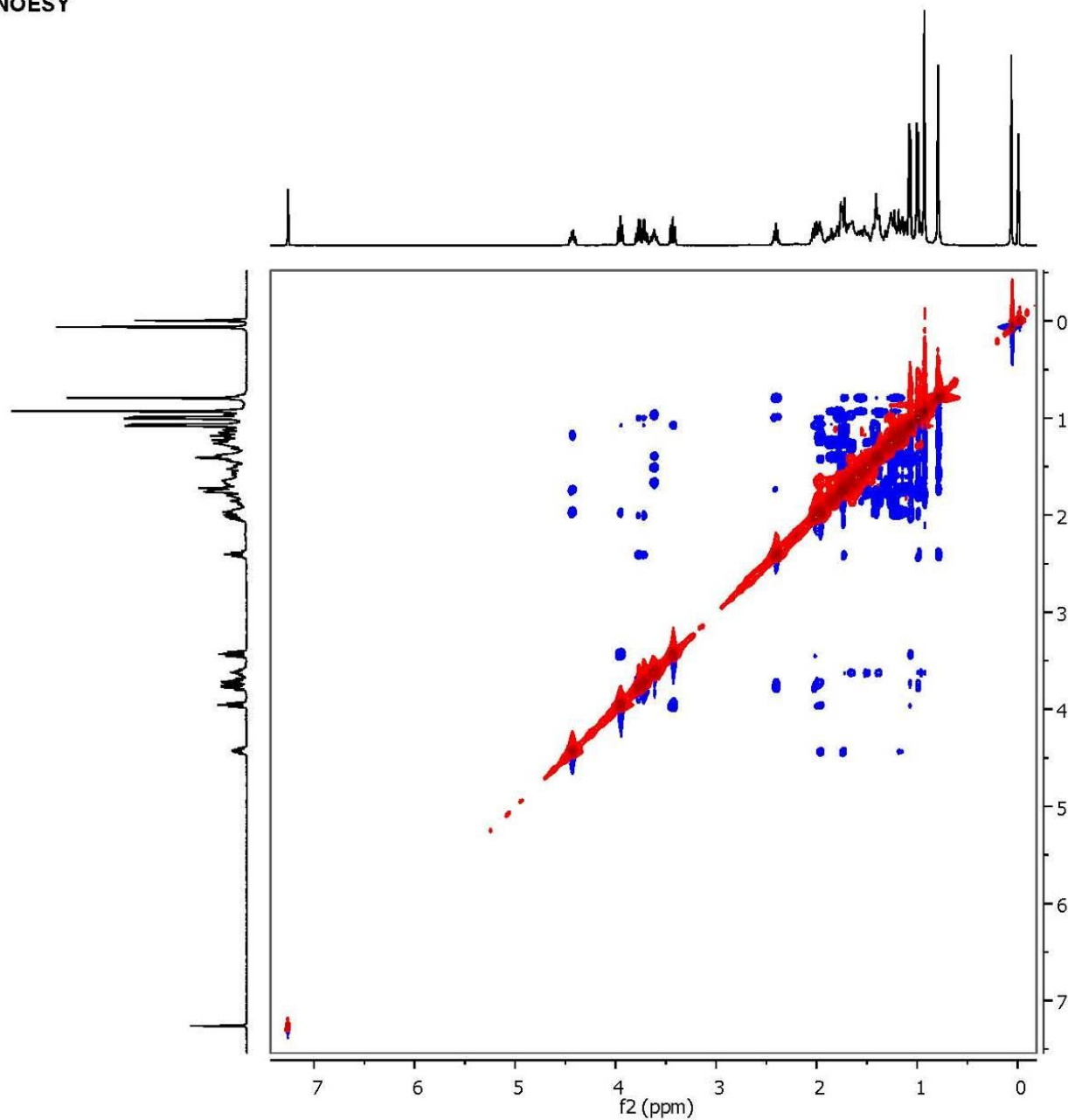


Parámetro	Valor (f2, f1)
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCI3 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	nmrs
4 Author	nmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gCOSY
8 Number of Scans	8
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1352
13 Acquisition Date	2009-11-17T2 3:58:09
14 Modification Date	2009-11-18T1 7:05:32
15 Spectrometer Frequency	(399.95, 399.95)
16 Spectral Width	(3787.9, 3787.9)
17 Lowest Frequency	(-466.7, -463.9)
18 Nucleus	(1H, 1H)
19 Acquired Size	(512, 200)
20 Spectral Size	(512, 512)

COSY

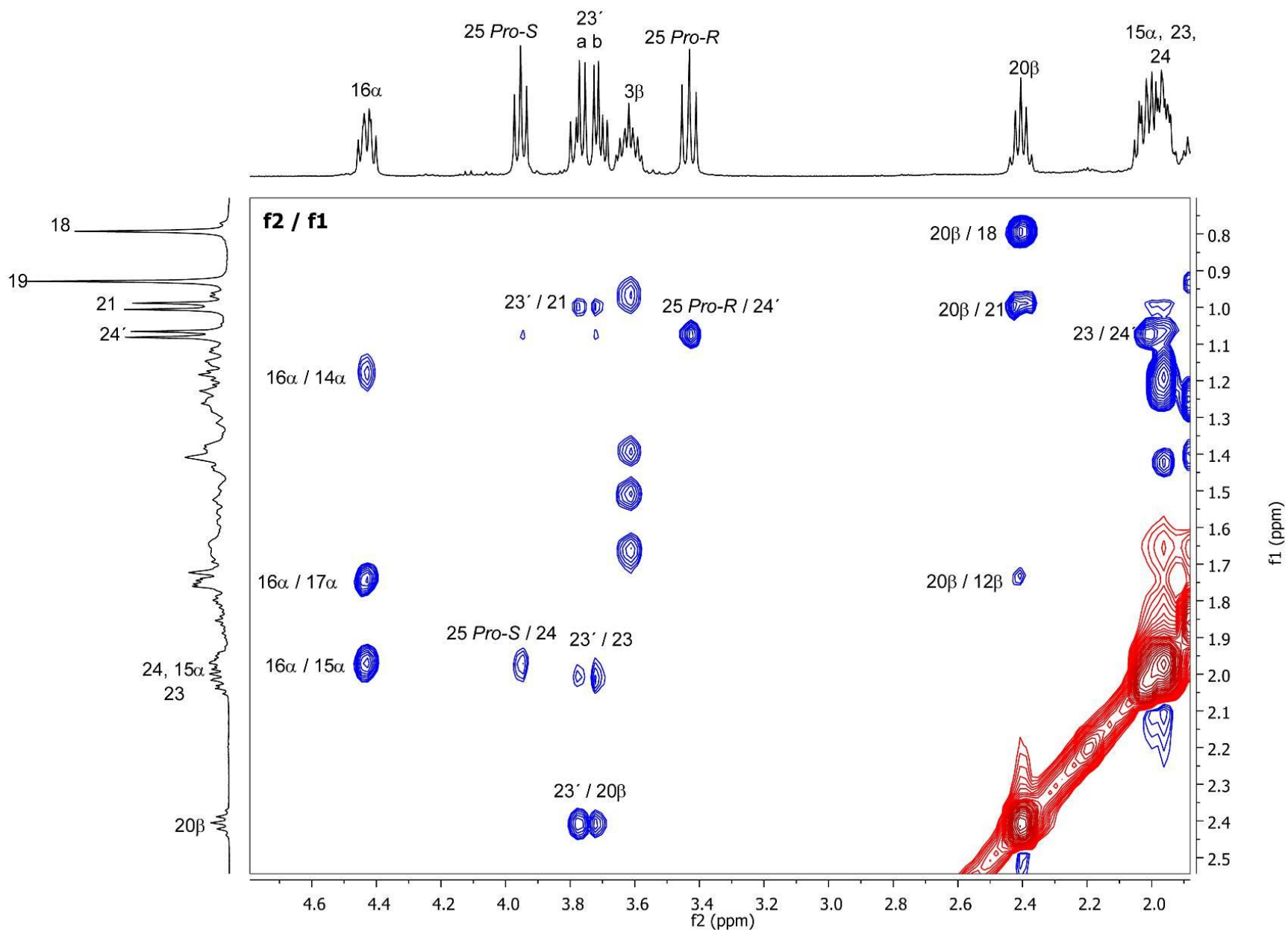


## NOESY

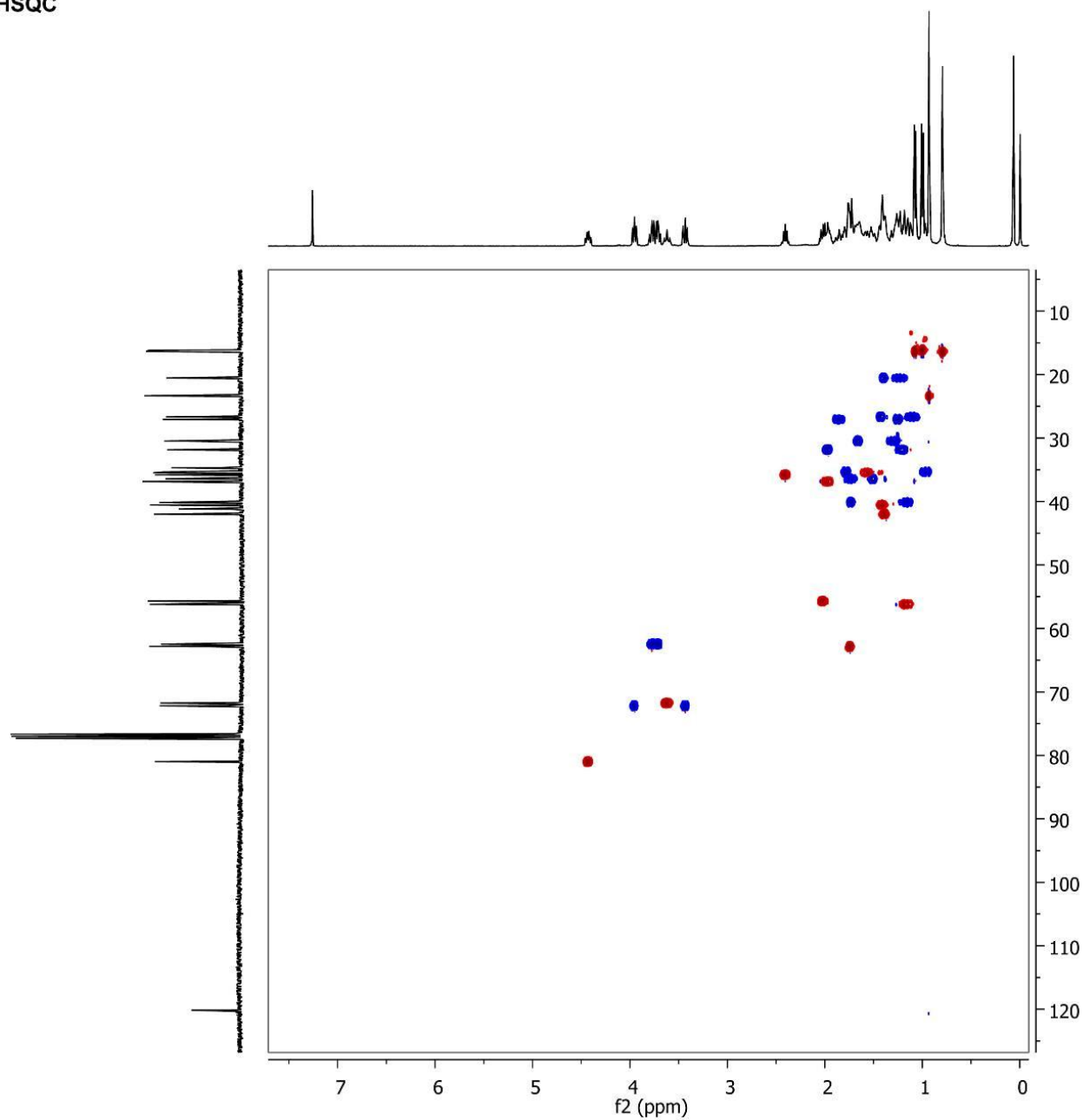


Parámetro	Valor (f2, f1)
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCl3 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	nmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	NOESY
8 Number of Scans	16
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1352
13 Acquisition Date	2009-11-18T0 0:29:56
14 Modification Date	2009-11-18T1 7:05:46
15 Spectrometer Frequency	(399.95, 399.95)
16 Spectral Width	(3787.9, 3787.9)
17 Lowest Frequency	(-467.9, -464.3)
18 Nucleus	(1H, 1H)
19 Acquired Size	(512, 200)
20 Spectral Size	(512, 512)

NOESY

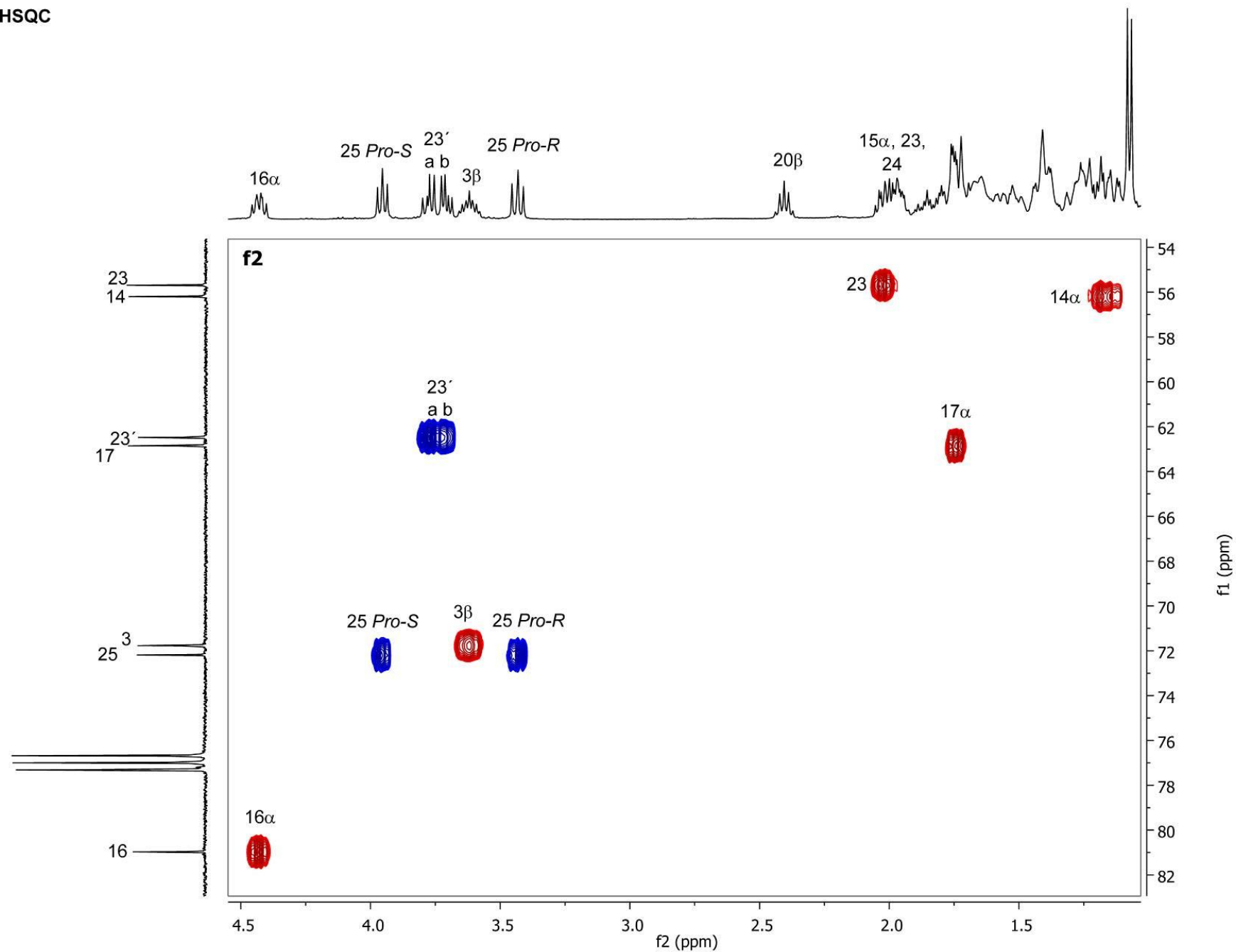


## HSQC



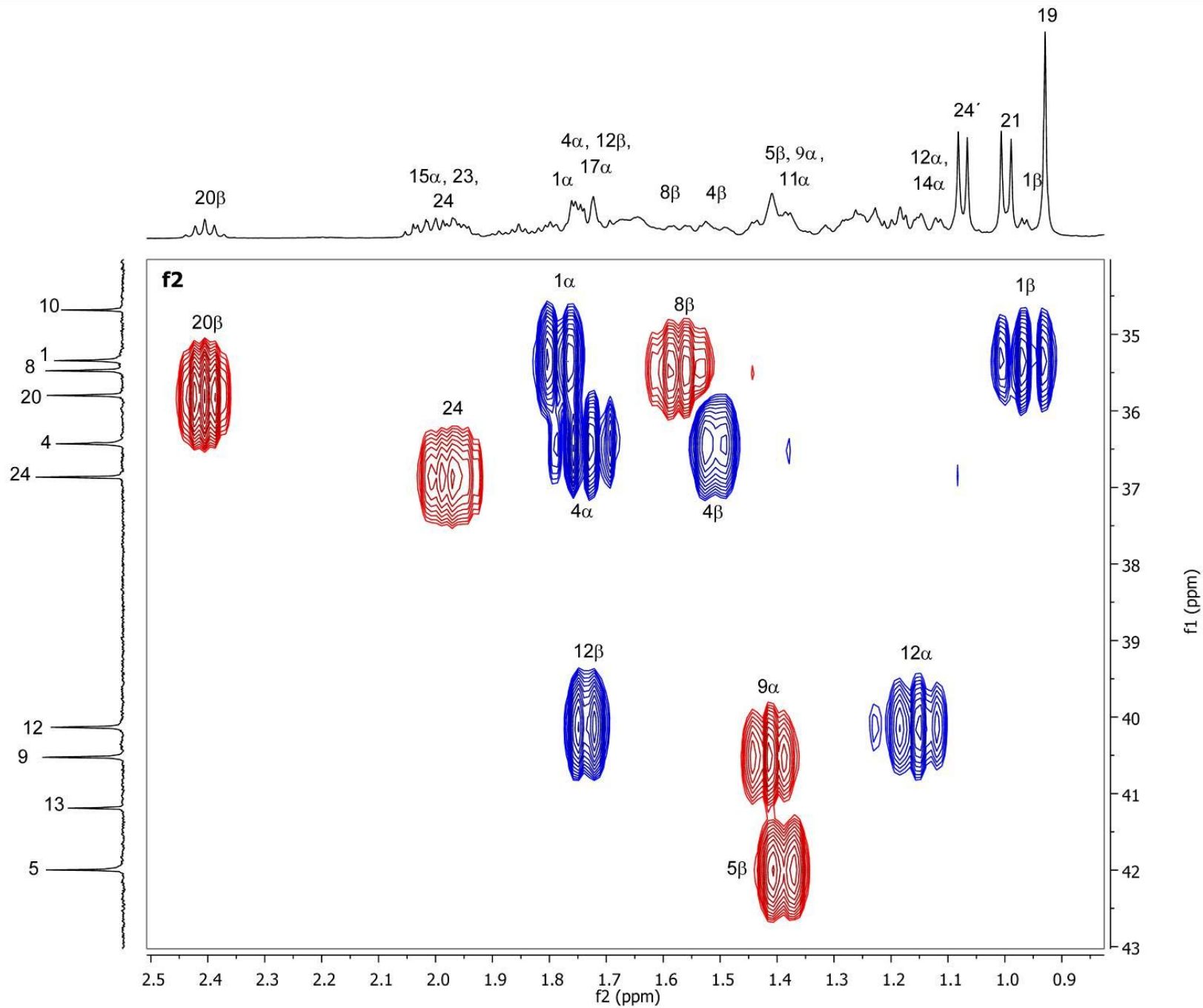
Parámetro	Valor (f2, f1)
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCl3 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	vnmr5
4 Author	vnmr1
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHSQC
8 Experiment	2D-HSQC- EDITED
9 Number of Scans	16
10 Receiver Gain	20
11 Relaxation Delay	1.0000
12 Pulse Width	0.0000
13 Acquisition Time	0.1993
14 Acquisition Date	2009-11-17T2 1:18:28
15 Modification Date	2009-11-18T1 7:06:06
16 Spectrometer Frequency	(399.95, 100.58)
17 Spectral Width	(3787.9, 17097.7)
18 Lowest Frequency	(-469.5, -1002.5)
19 Nucleus	(1H, 13C)
20 Acquired Size	(755, 200)
21 Spectral Size	(1024, 1024)

HSQC

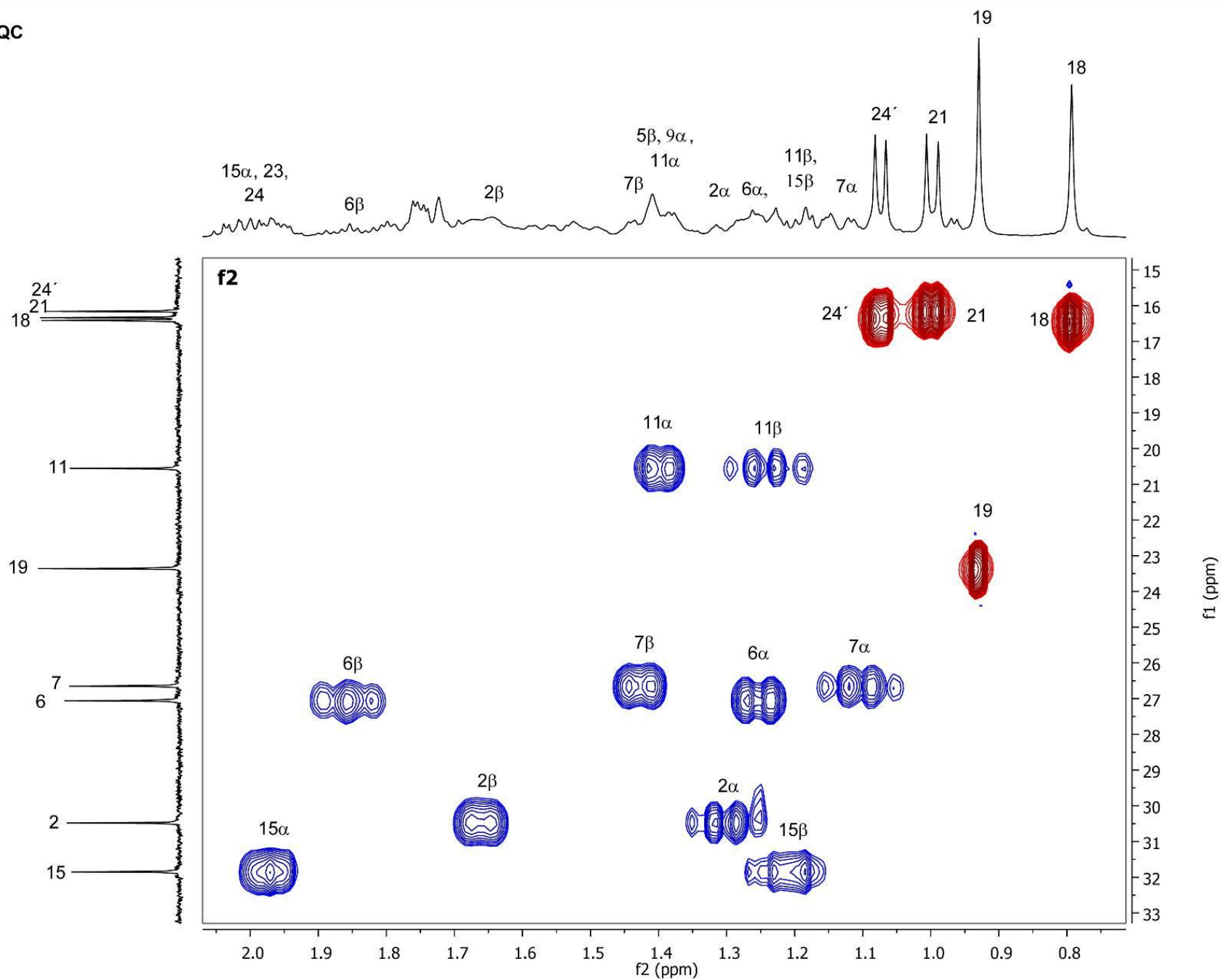




HSQC

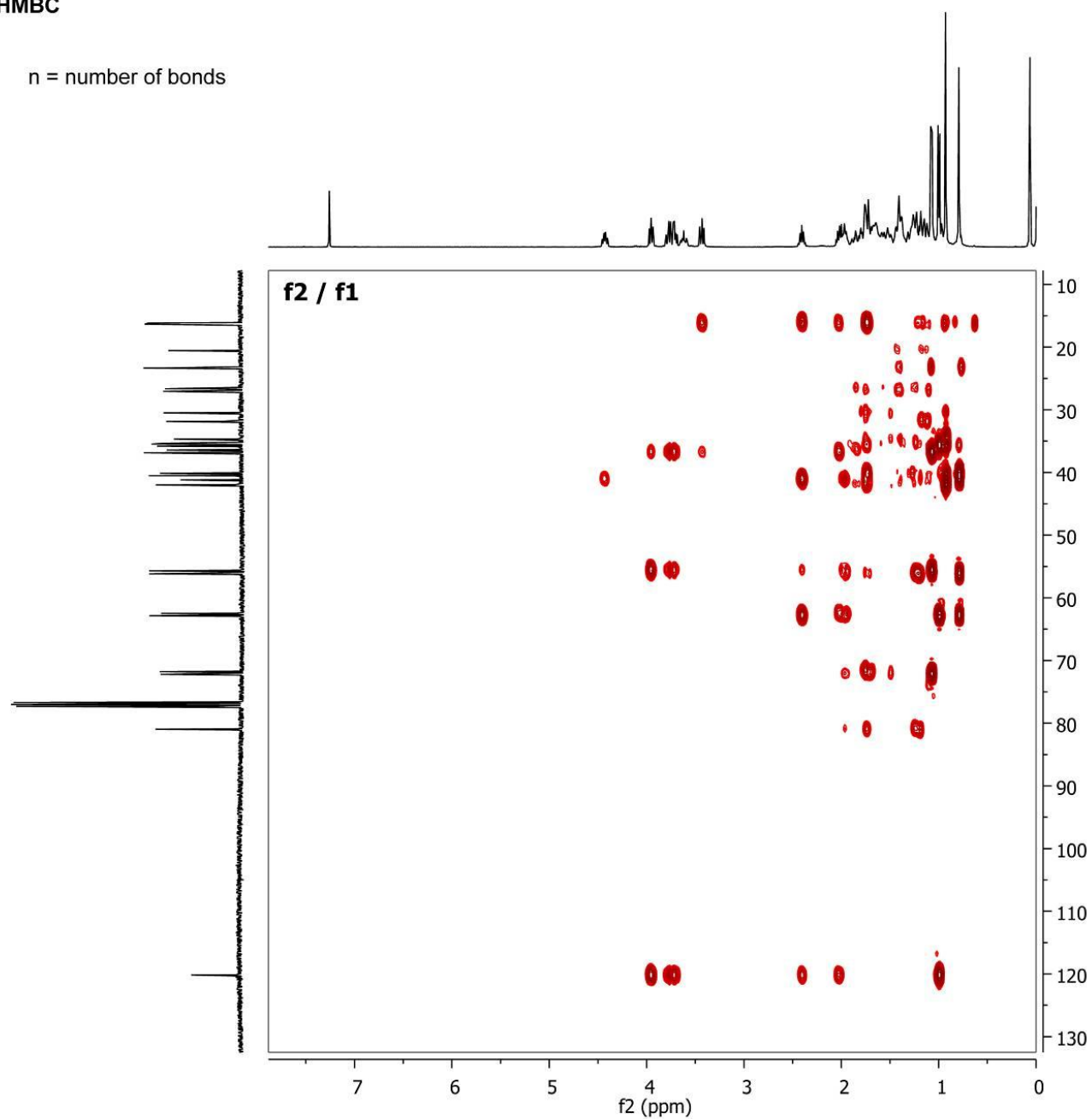


HSQC



## HMBC

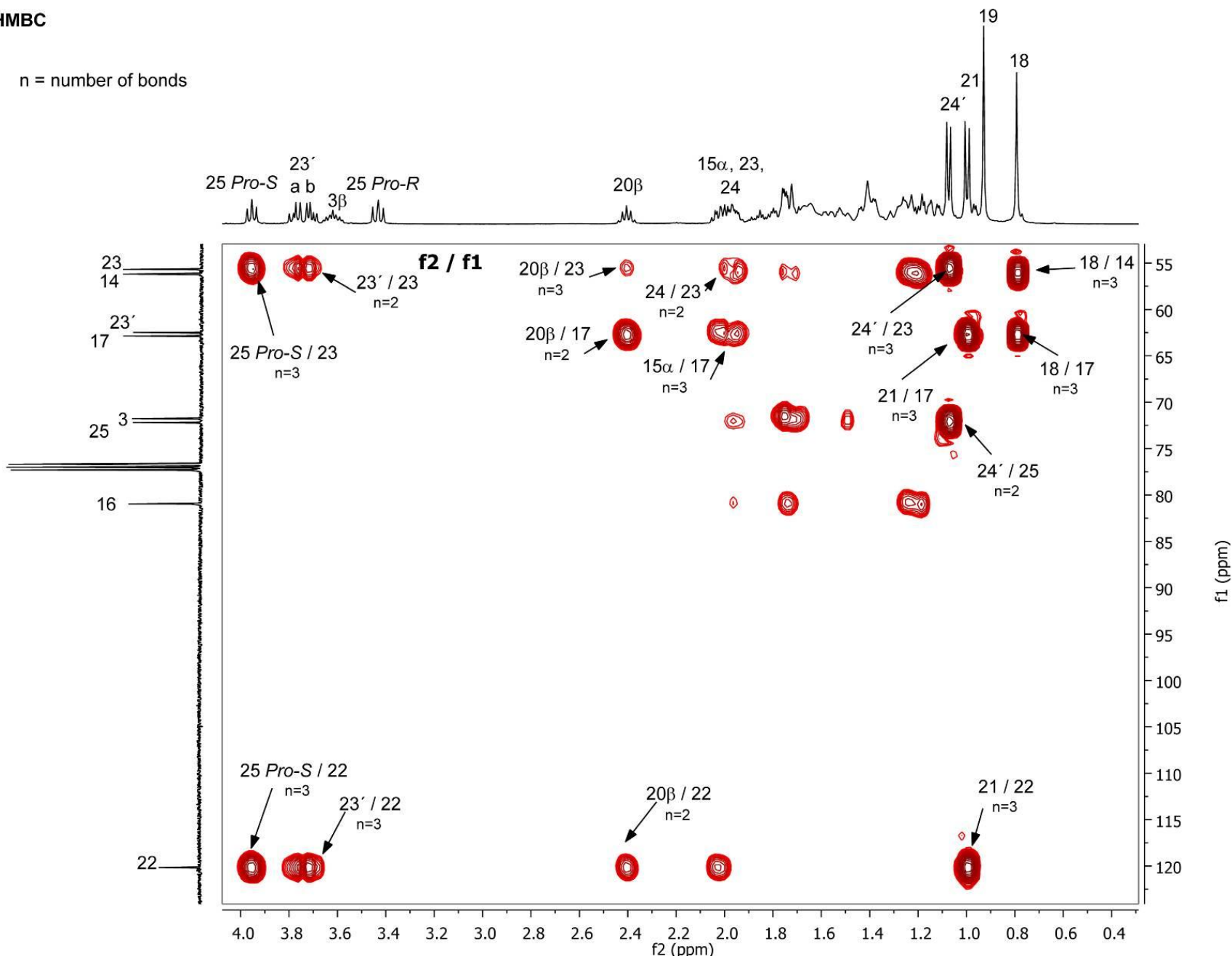
n = number of bonds



Parámetro	Valor (f2, f1)
1 Comment	10886
	USAI-UNAM Dr. Martin Iglesias/ Mariana Macias FES-R/ CDCl3 1H, 400 MHz 400-S NEE
2 Origin	Varian
3 Spectrometer	vnmrs
4 Author	walkup
5 Solvent	cdcl3
6 Temperature	26.0
7 Pulse Sequence	gHMBC
8 Number of Scans	32
9 Receiver Gain	20
10 Relaxation Delay	1.0000
11 Pulse Width	0.0000
12 Acquisition Time	0.1280
13 Acquisition Date	2009-11-18T05:32:41
14 Modification Date	2009-11-18T17:06:22
15 Spectrometer Frequency	(399.95, 100.58)
16 Spectral Width	(3787.9, 24132.7)
17 Lowest Frequency	(-467.1, -1486.5)
18 Nucleus	(1H, 13C)
19 Acquired Size	(485, 256)
20 Spectral Size	(512, 512)

HMBC

n = number of bonds



## HMBC

n = number of bonds

