

# Supporting Information

**Copies of HR-ESIMS, and 1D and 2D NMR spectra of Compounds 1-14.**

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**Table S1:** Cytotoxic assay results for compounds against human cancer cells [**IC<sub>50</sub>** (μM)]

<b>Compound</b>	<b>HCT-8</b>	<b>HCT-8/T</b>	<b>A2780</b>	<b>A2780/T</b>	<b>MDA-MB-231</b>
<b>1</b>	>100	>100	>100	>100	>100
<b>3</b>	>100	>100	>100	>100	>100
<b>8</b>	>100	>100	>100	>100	>100
<b>10</b>	>100	>100	>100	>100	>100
<b>11</b>	70.14	64.14	62.04	82.17	>100
<b>14</b>	>100	>100	>100	>100	>100
<b>15</b>	>100	>100	>100	>100	>100
<b>16</b>	>100	>100	>100	>100	94.51
<b>20</b>	>100	>100	>100	>100	>100
<b>23</b>	68.84	>100	64.18	77.80	37.68
<b>25</b>	>100	>100	>100	>100	>100
<b>27</b>	>100	>100	>100	>100	>100
<b>Cisplatin</b>	15.43	21.98	8.54	9.26	6.25

**Table S2:** HIV-inhibitory bioassay results for tested compounds

<b>Compound</b>	<b>HIV-1 inhibitory rate (100μM)</b>	<b>HIV-1 inhibitory rate (20μM)</b>	<b>IC<sub>50</sub> (μM)</b>	<b>CC<sub>50</sub> (μM)</b>
<b>1</b>	53.74 ± 7.31	17.49 ± 6.93	NA	NT
<b>3</b>	15.15 ± 19.49	NA	NA	NT
<b>8</b>	NA	NA	NA	NT
<b>10</b>	40.69 ± 2.25	NA	NA	NT
<b>11</b>	98.56 ± 0.81	24.47 ± 5.04	NA	NT
<b>14</b>	59.33 ± 1.54	NA	NA	NT
<b>20</b>	31.83 ± 3.20	NA	NA	NT
<b>23</b>	97.57 ± 2.67	14.77 ± 5.91	NA	NT
<b>24</b>	31.83 ± 2.58	14.34 ± 3.92	NA	NT
<b>25</b>	99.44 ± 0.77	NA	NA	NT
<b>27</b>	23.86 ± 2.89	NA	NA	NT

NA = No activity ; NT = None tested

# HR-ESIMS for compound 1

## Mass Spectrum SmartFormula Report

### Analysis Info

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Sample Name SCSIO  
Comment

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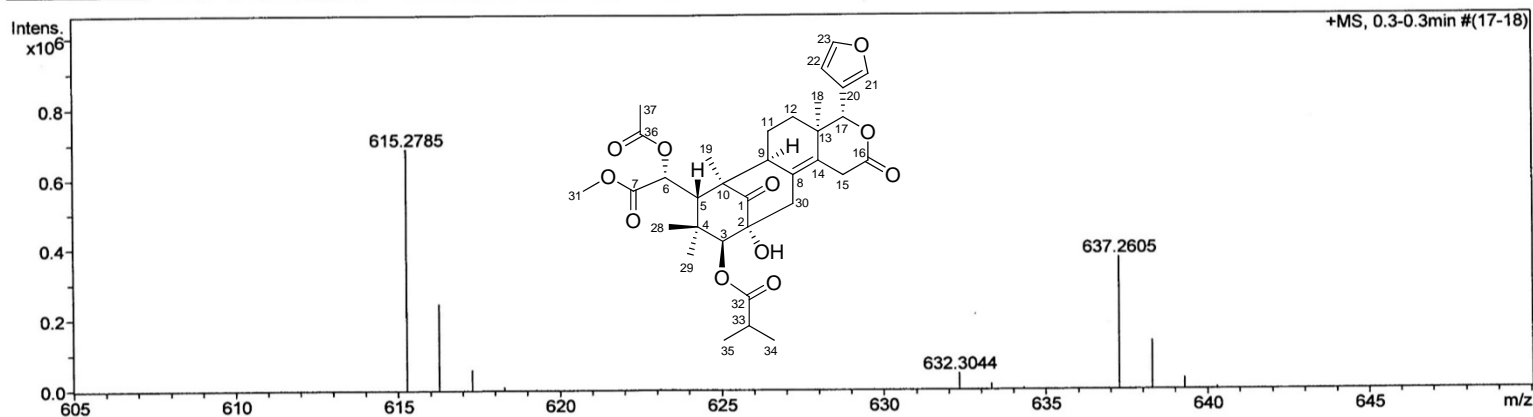
Operator SCSIO  
Instrument / Ser# maXis 29

### Acquisition Parameter

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Focus Not active  
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Scan End 2000 m/z

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Set End Plate Offset -500 V  
Set Collision Cell RF 2000.0 Vpp

Set Nebulizer 0.3 Bar  
Set Dry Heater 180 °C  
Set Dry Gas 4.0 l/min  
Set Divert Valve Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
615.2785	1	C <sub>33</sub> H <sub>43</sub> O <sub>11</sub>	100.00	615.2800	1.5	2.4	5.9	12.5	even	ok
637.2605	1	C <sub>33</sub> H <sub>42</sub> NaO <sub>11</sub>	100.00	637.2619	1.5	2.3	3.7	12.5	even	ok



# HR-ESIMS for compound 1

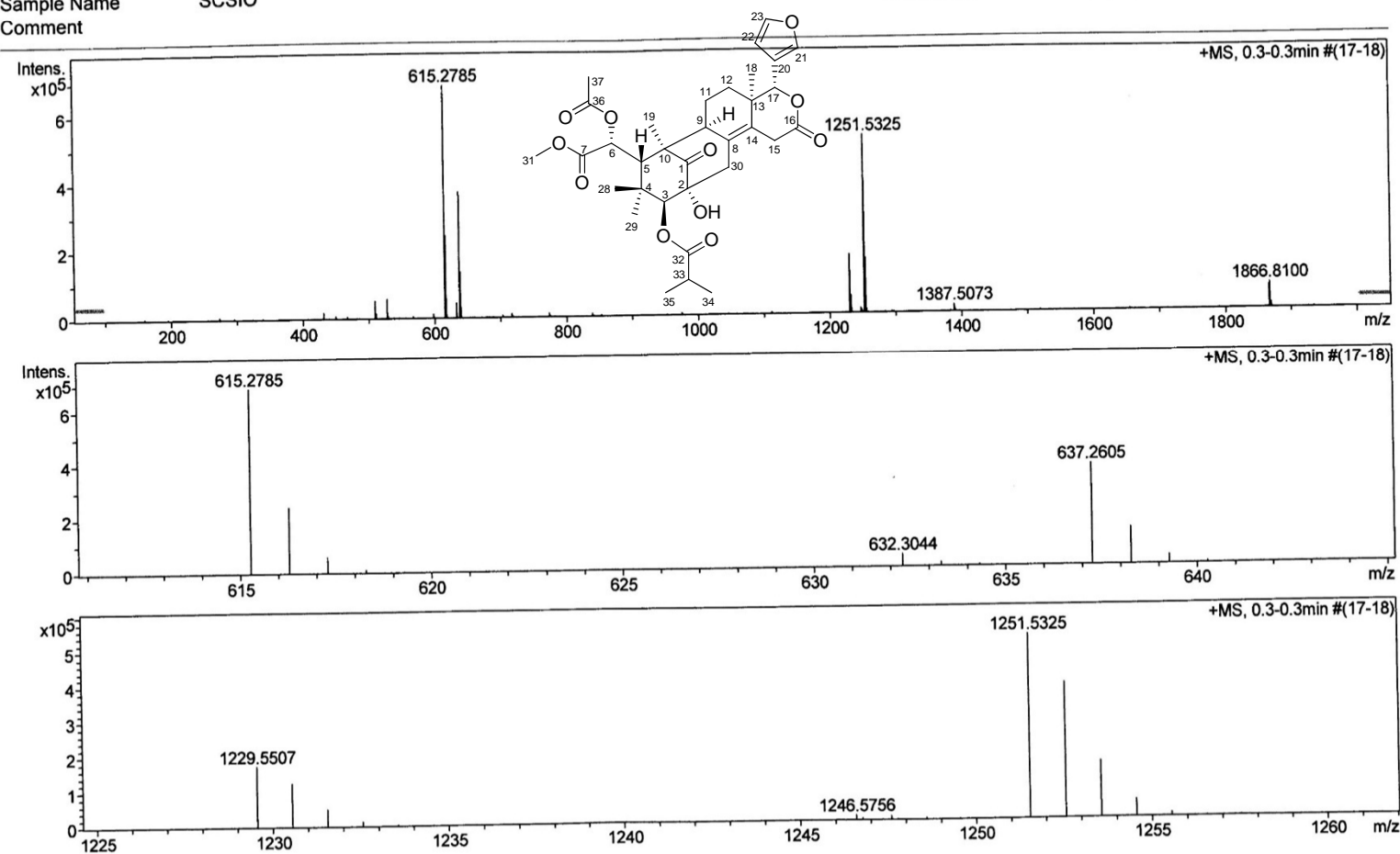
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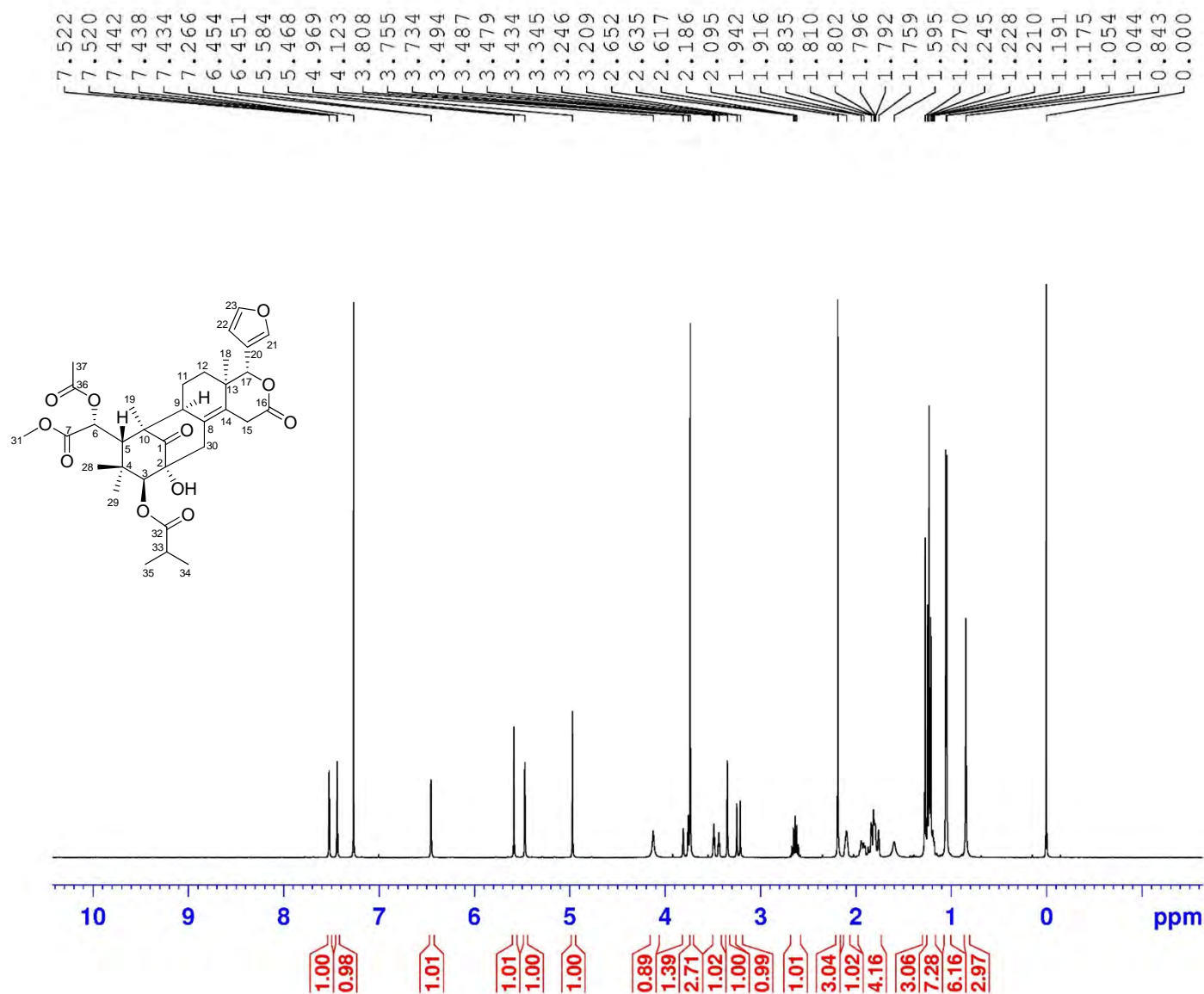
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Sample Name SCSIO  
Comment

Acquisition Date 4/27/2015 10:36:39 AM

Operator SCSIO  
Instrument maXis



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



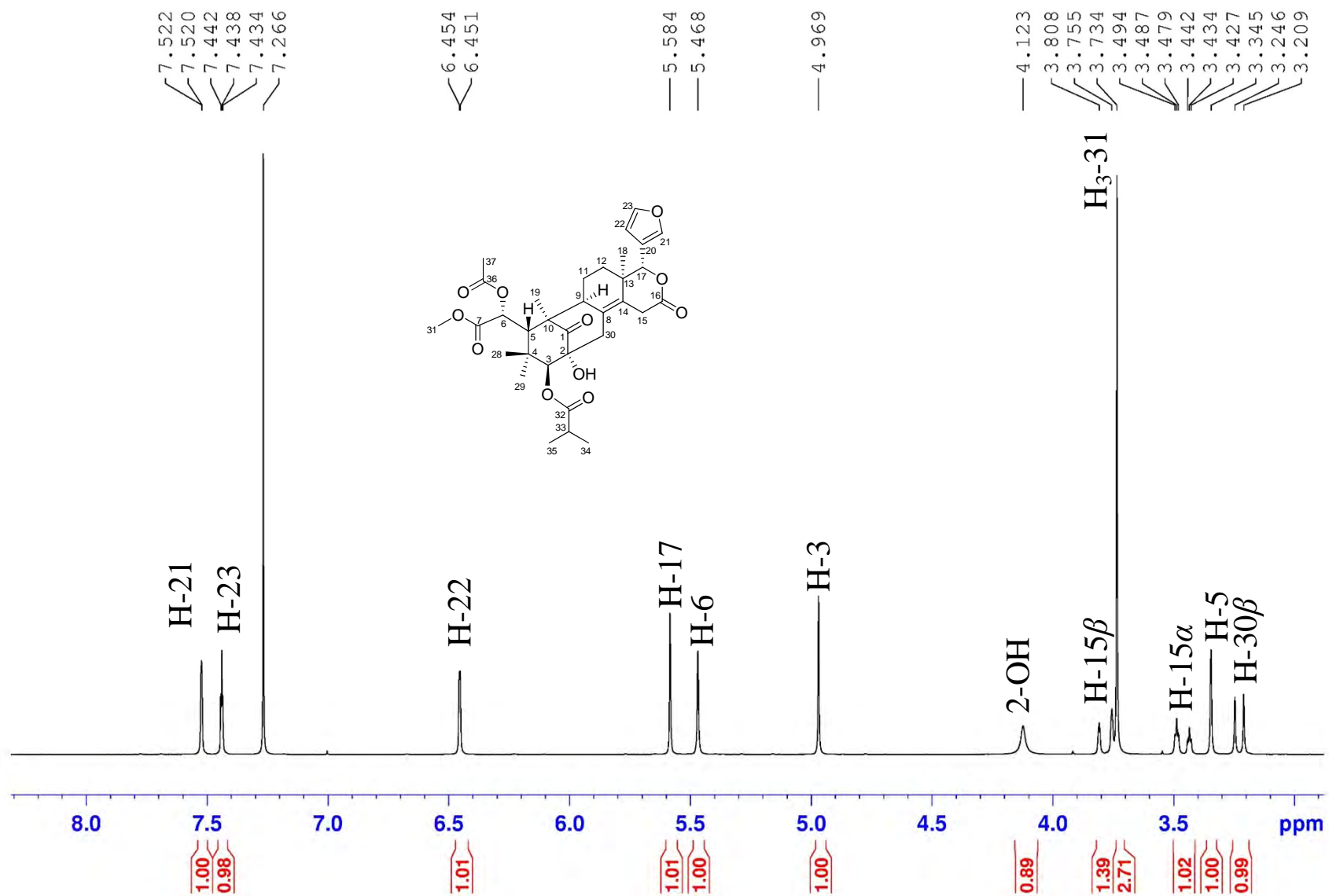
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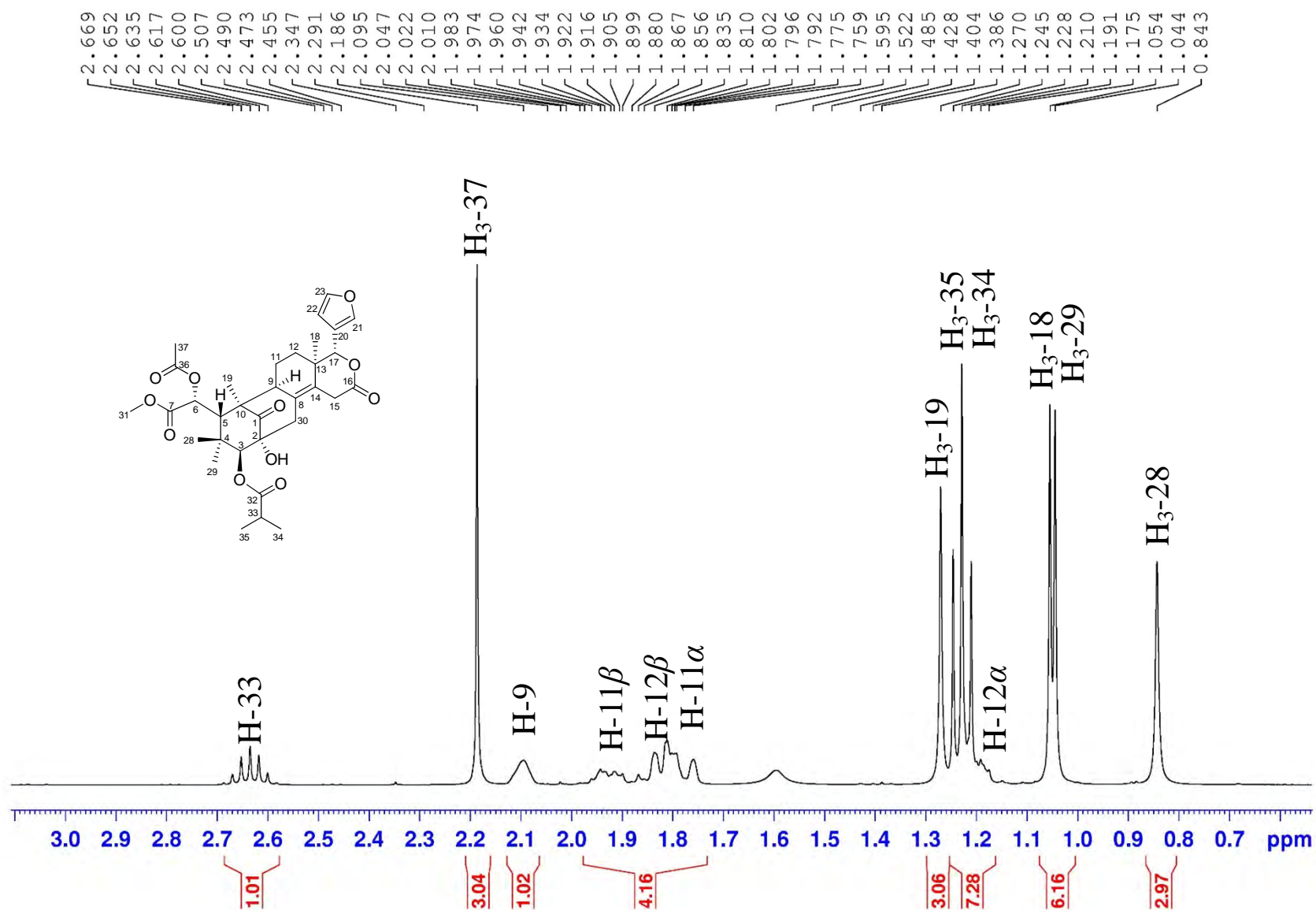
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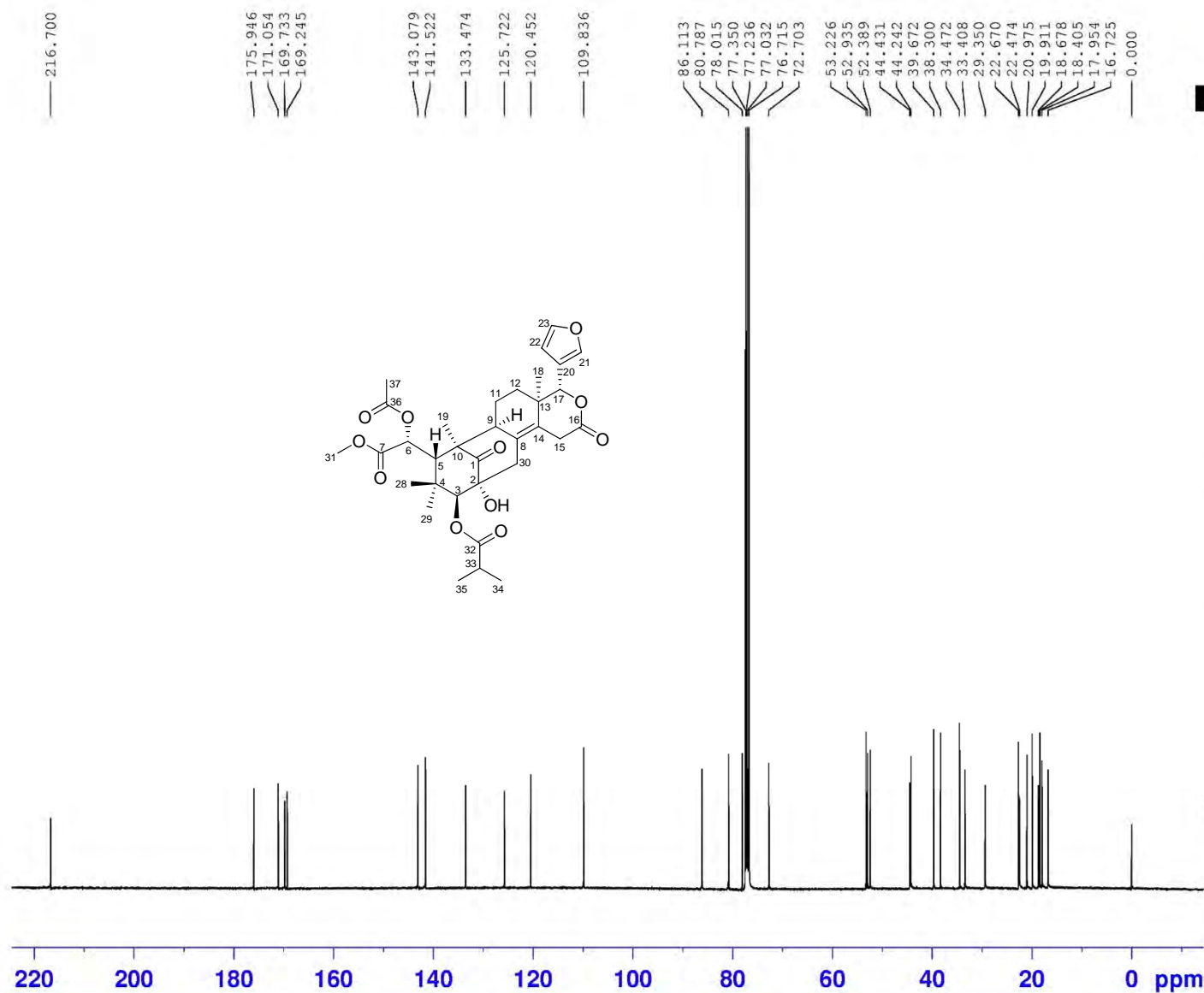
$^1\text{H}$  NMR (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



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



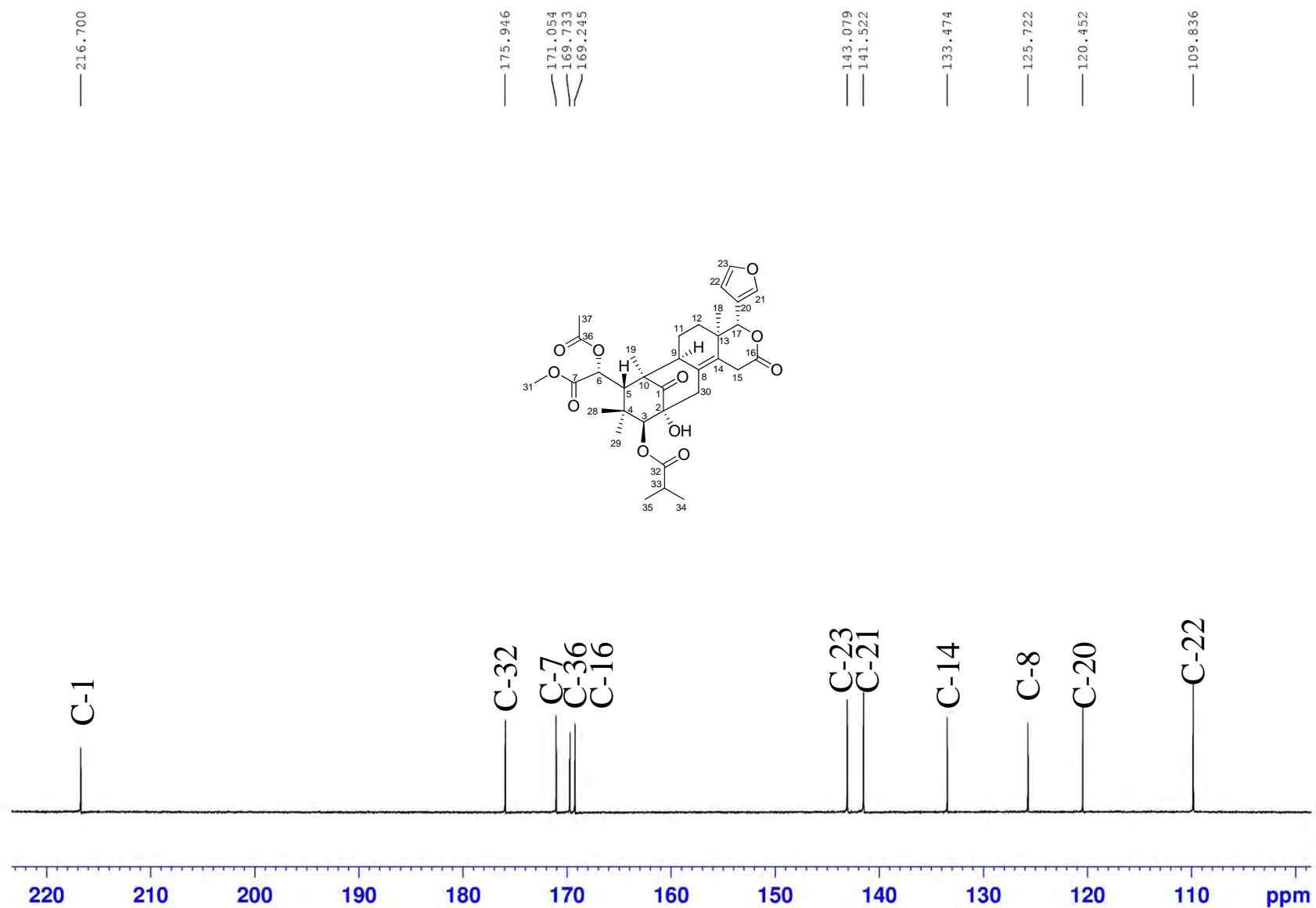
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



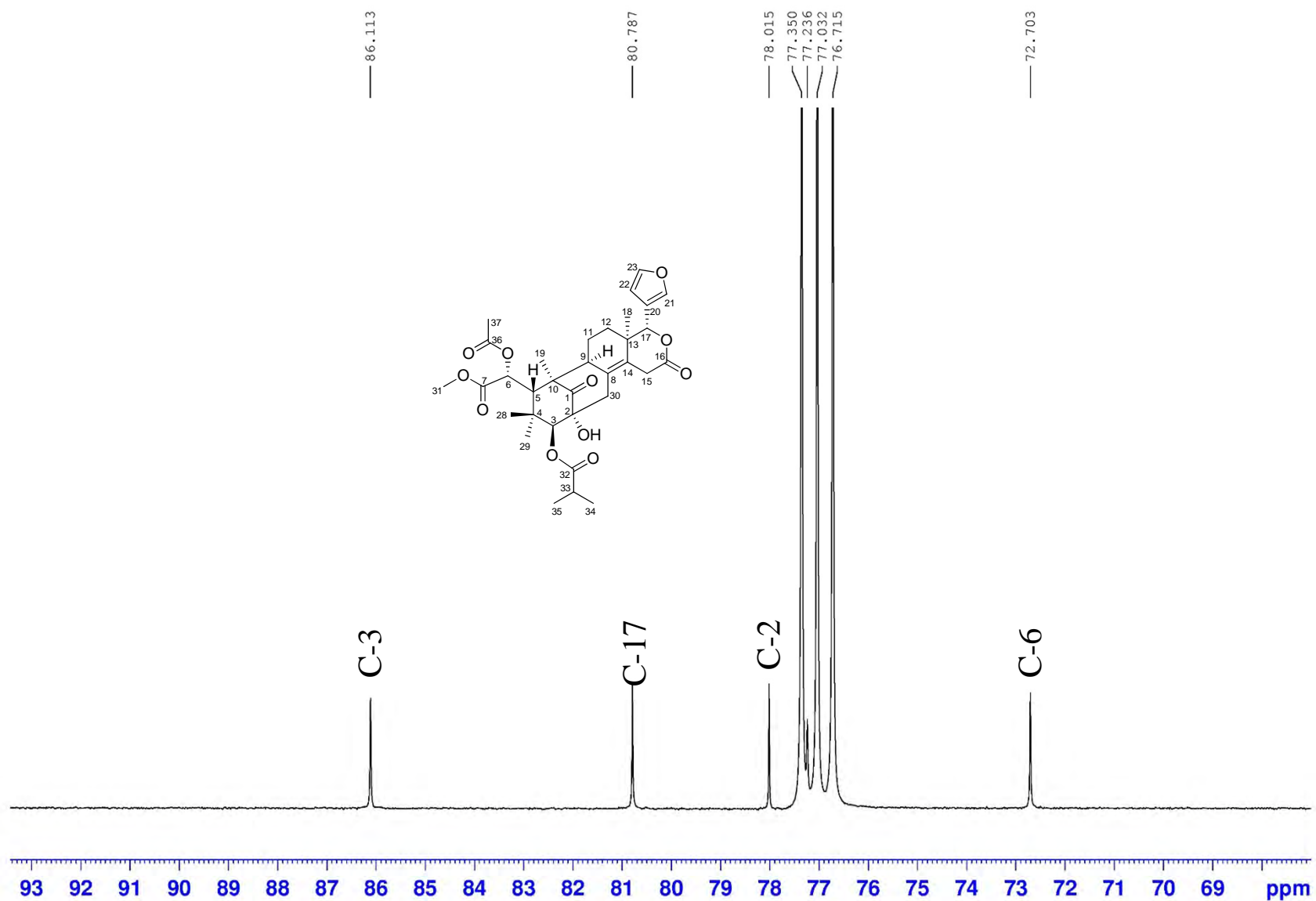
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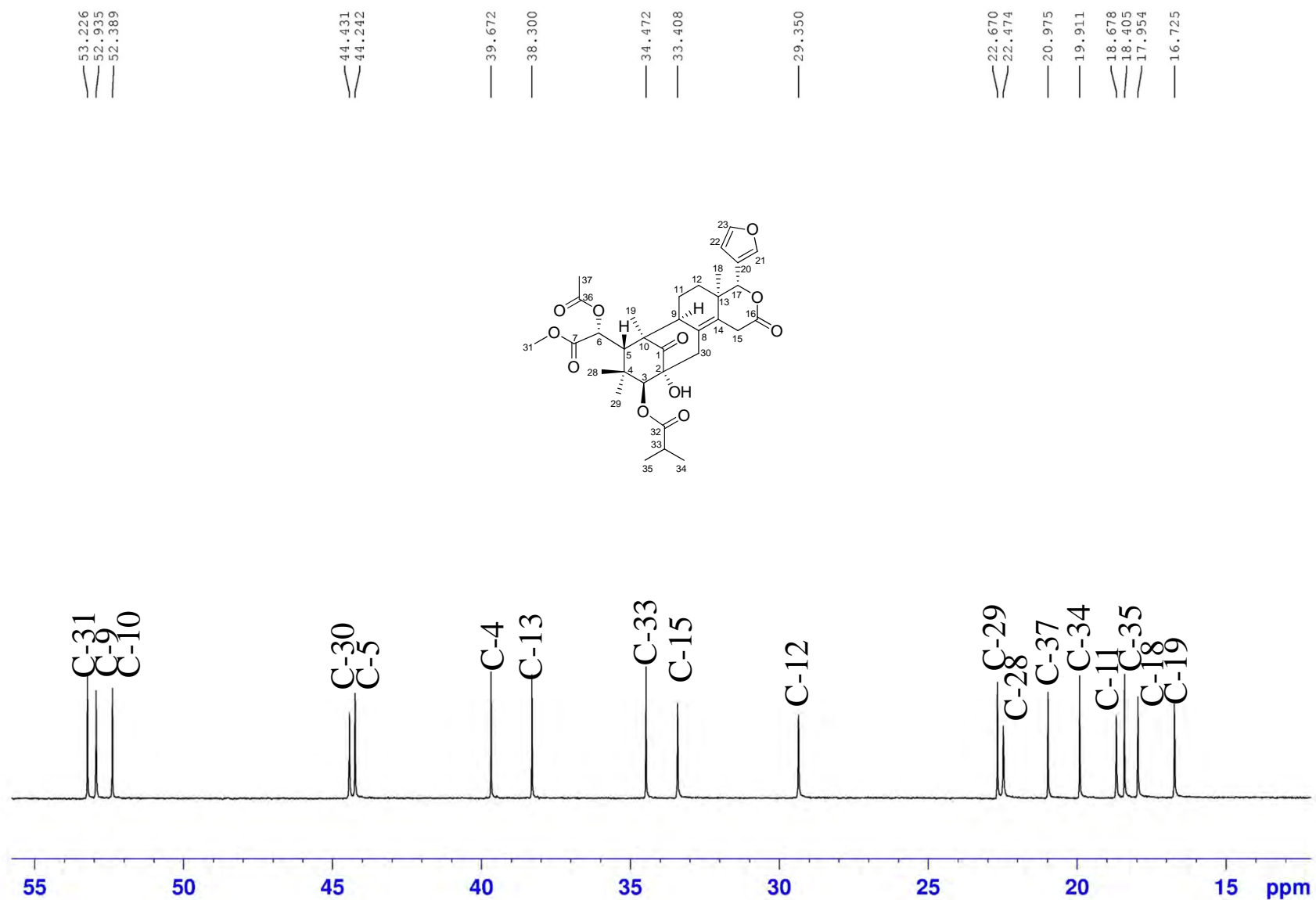
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



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

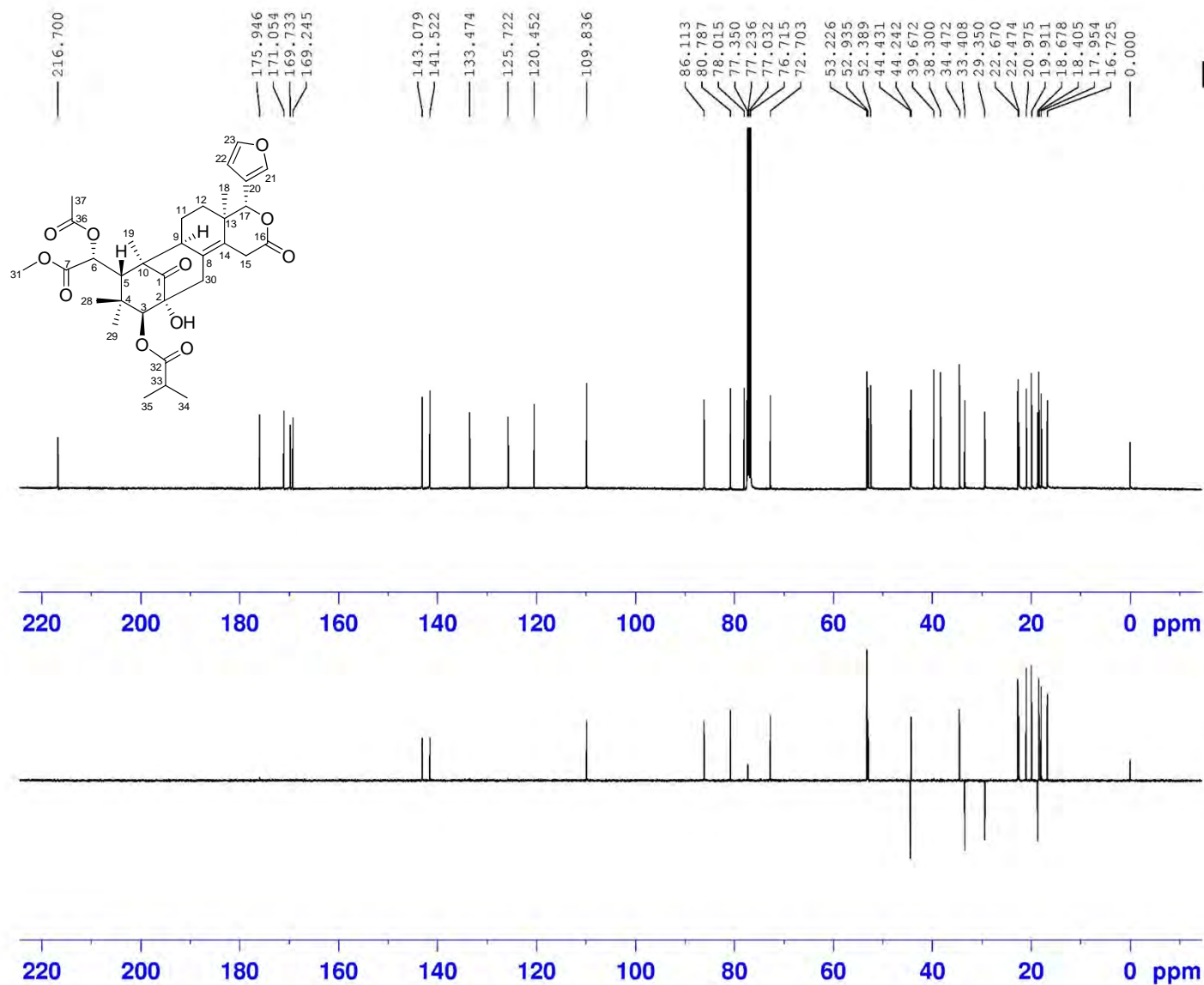


$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **1** in  $\text{CDCl}_3$





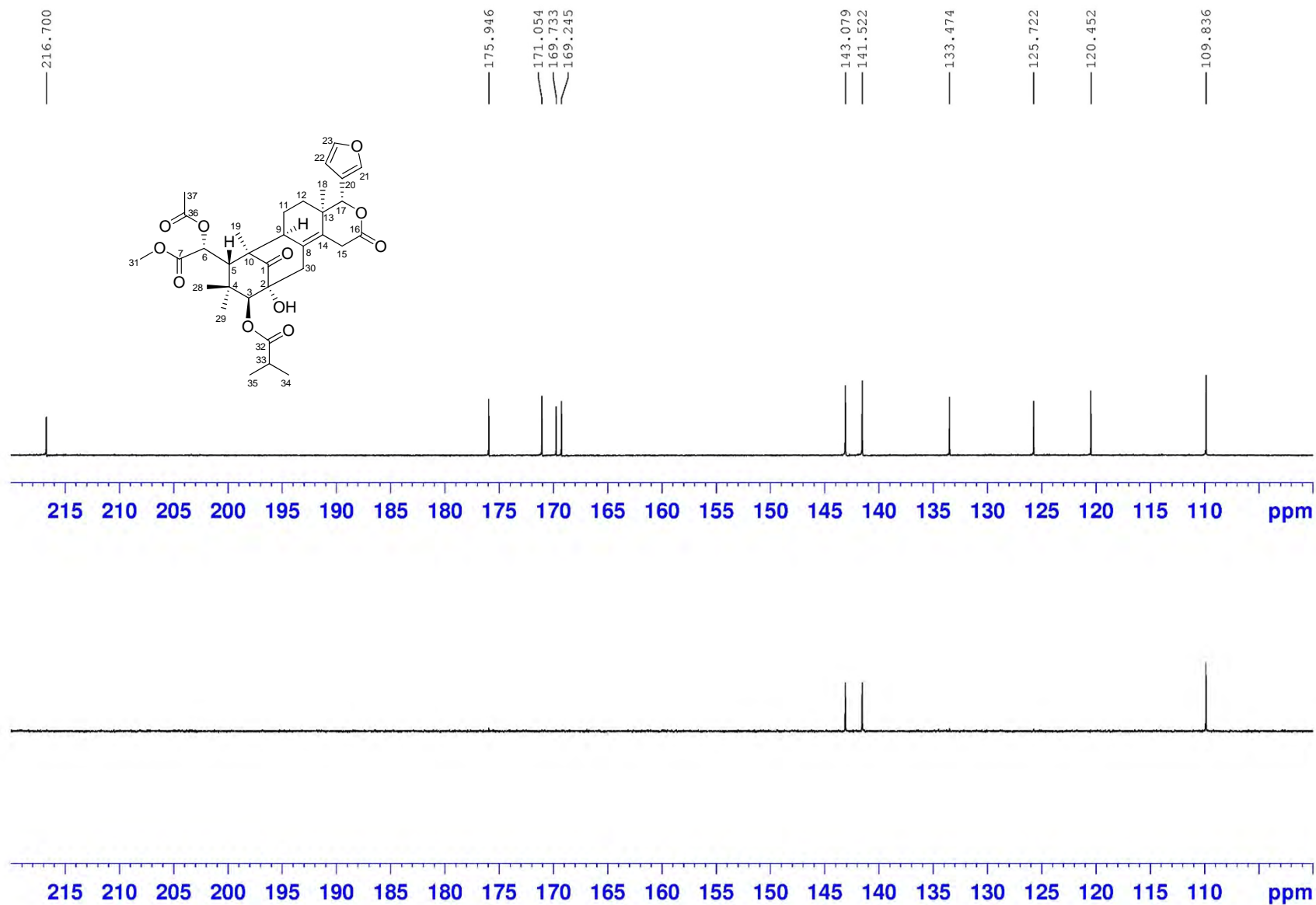
# DEPT135 (100 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

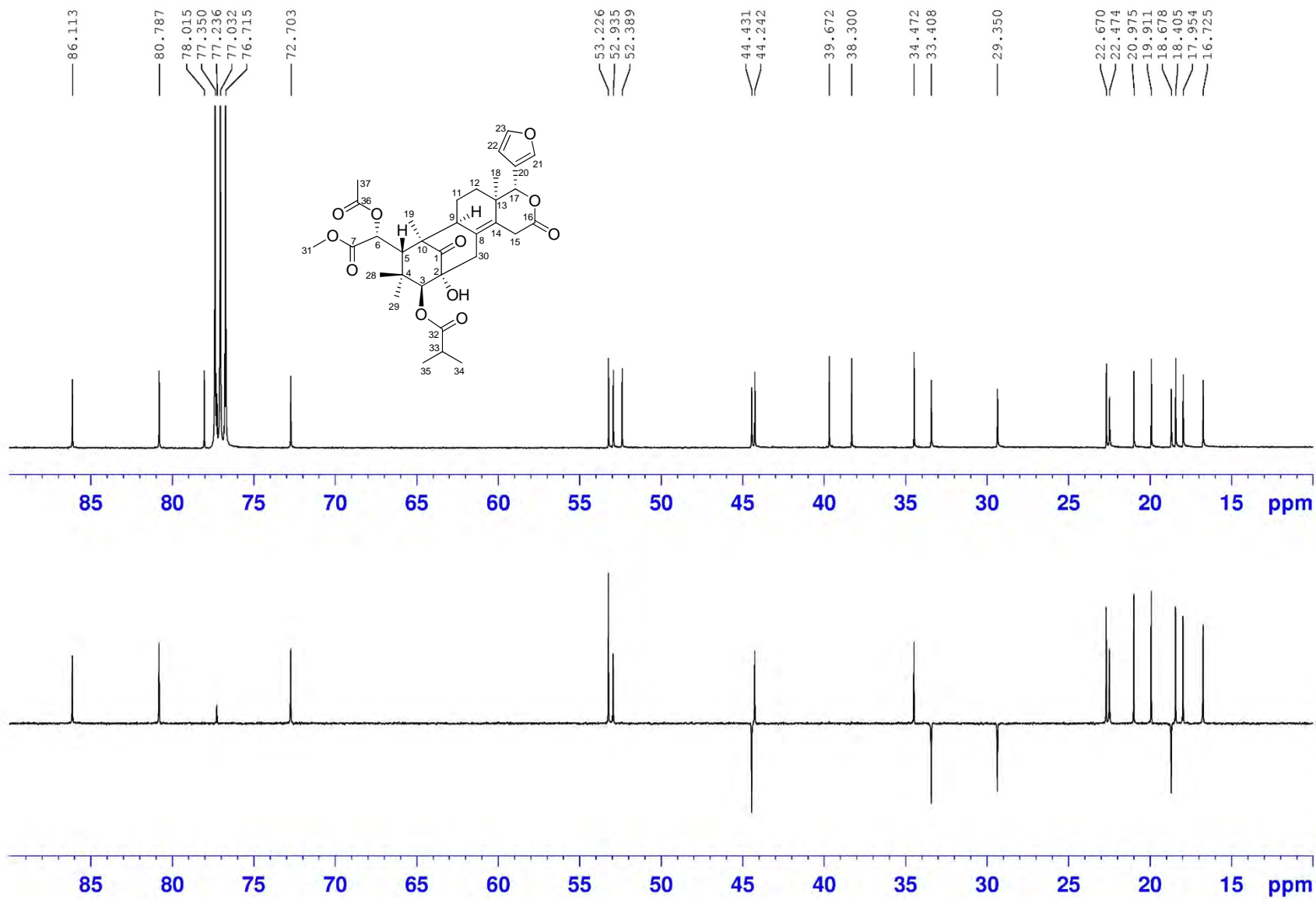


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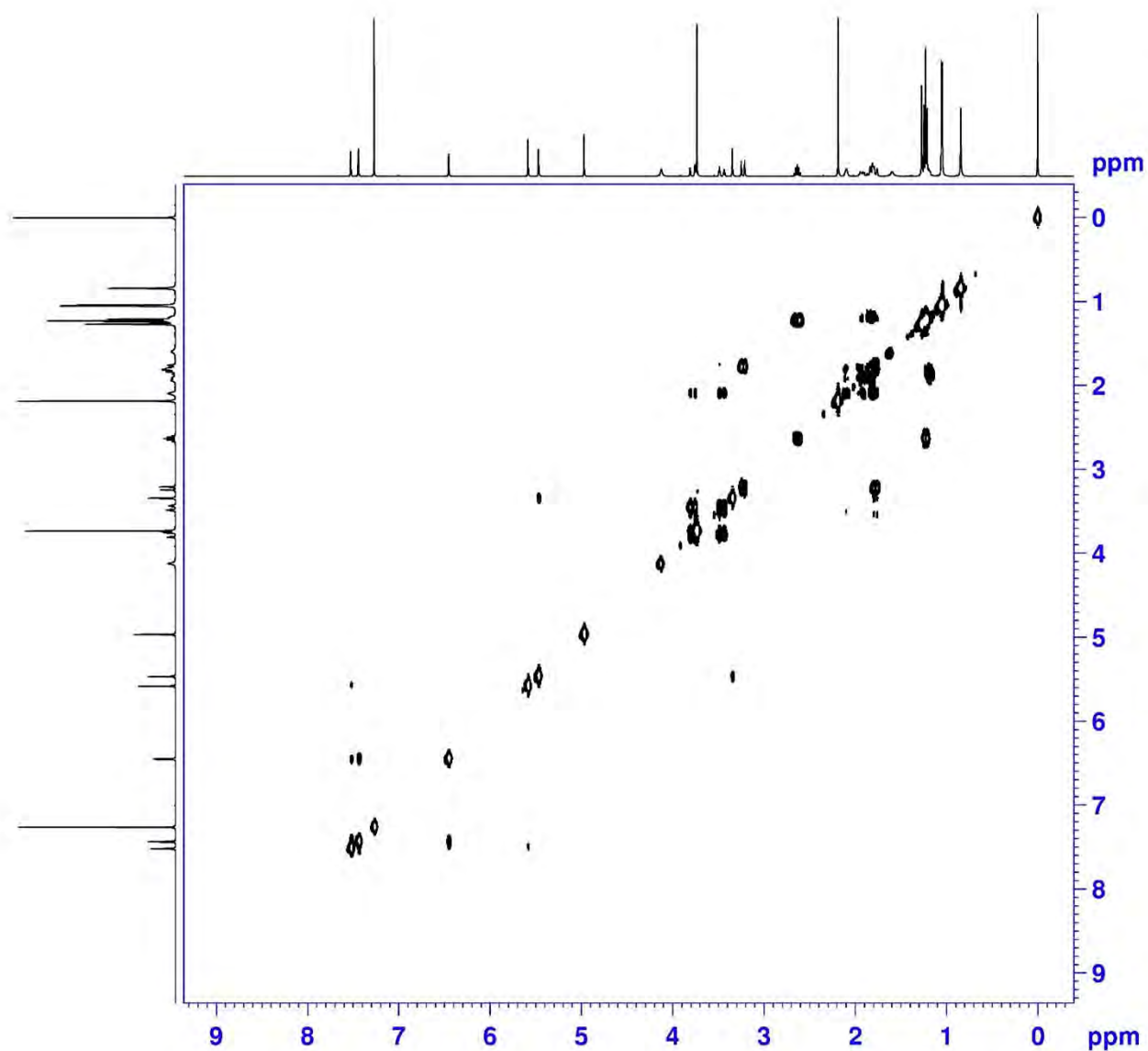
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# DEPT135 (100 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

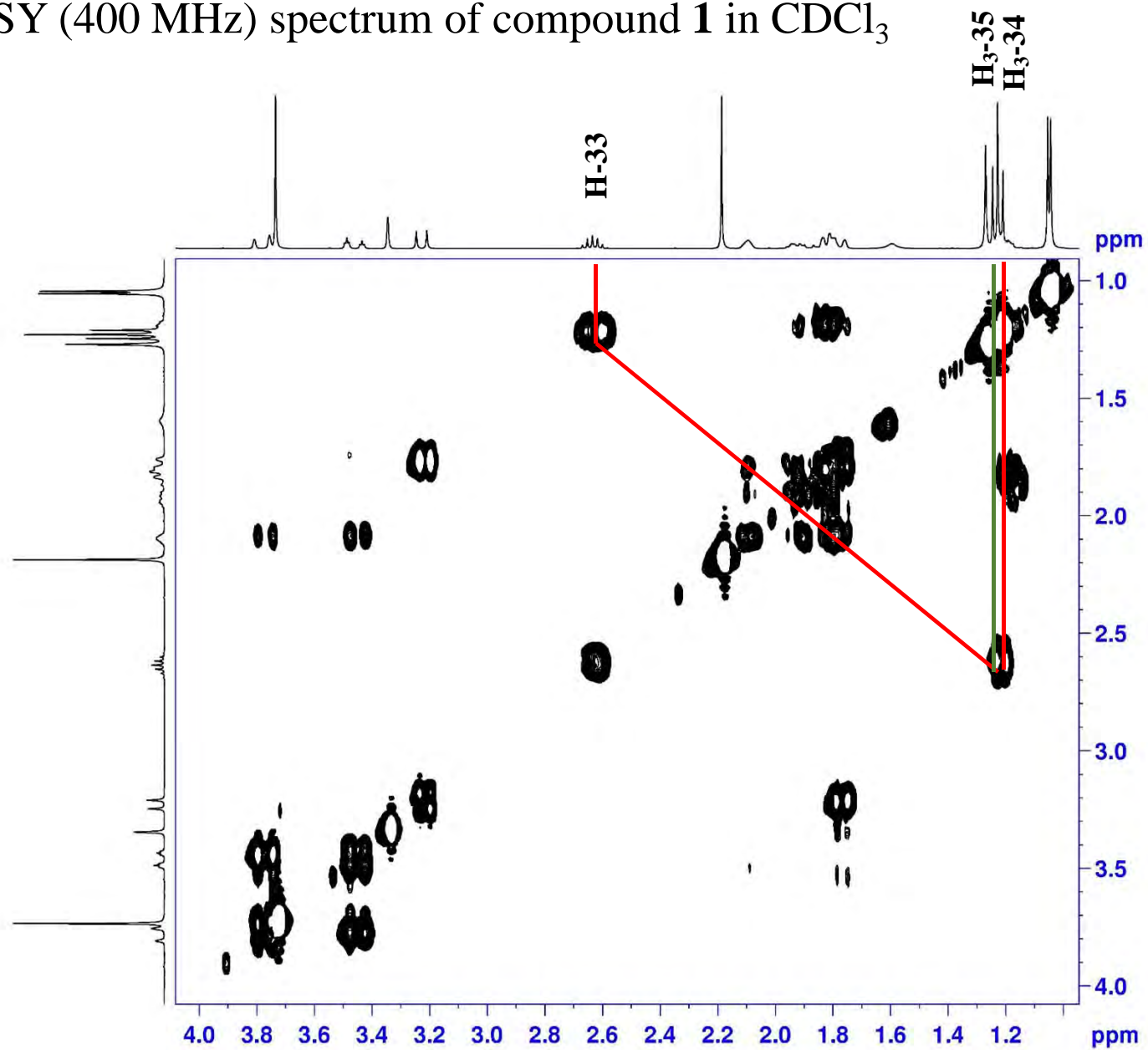
# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **1** in $\text{CDCl}_3$



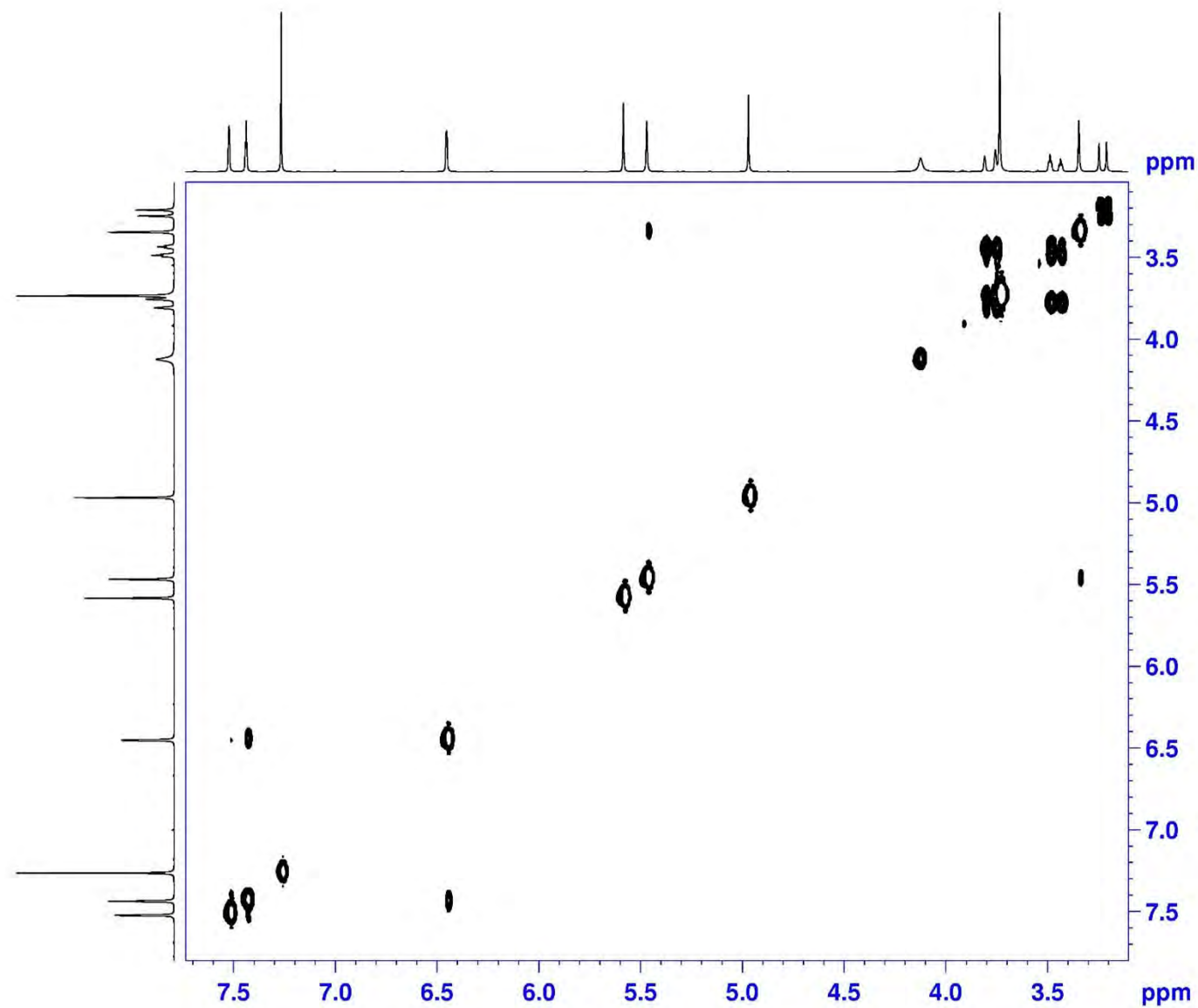
NAME lws-46  
EXPNO 4  
PROCNO 1  
Date\_ 20150221  
Time 19.08  
INSTRUM spect  
PROBHD 5 mm CPPBBO BB  
PULPROG cosygpppgf  
TD 2048  
SOLVENT  $\text{CDCl}_3$   
NS 32  
DS 8  
SWH 3906.250 Hz  
FIDRES 1.907349 Hz  
AQ 0.2621940 sec  
RG 171.57  
DW 128.000 usec  
DE 10.00 usec  
TE 297.0 K  
D0 0.00000300 sec  
D1 1.89678097 sec  
D11 0.03000000 sec  
D12 0.00002000 sec  
D13 0.00000400 sec  
D16 0.00020000 sec  
IN0 0.00025600 sec

===== CHANNEL f1 =====  
SFO1 400.1318006 MHz  
NUC1  $^1\text{H}$   
P0 12.00 usec  
P1 12.00 usec  
P17 2500.00 usec  
ND0 1  
TD 128  
SFO1 400.1318 MHz  
FIDRES 30.517578 Hz  
SW 9.762 ppm  
FnMODE QF  
SI 1024  
SF 400.1300098 MHz  
WDW QSINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40  
SI 1024  
MC2 QF  
SF 400.1300098 MHz  
WDW QSINE  
SSB 0

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

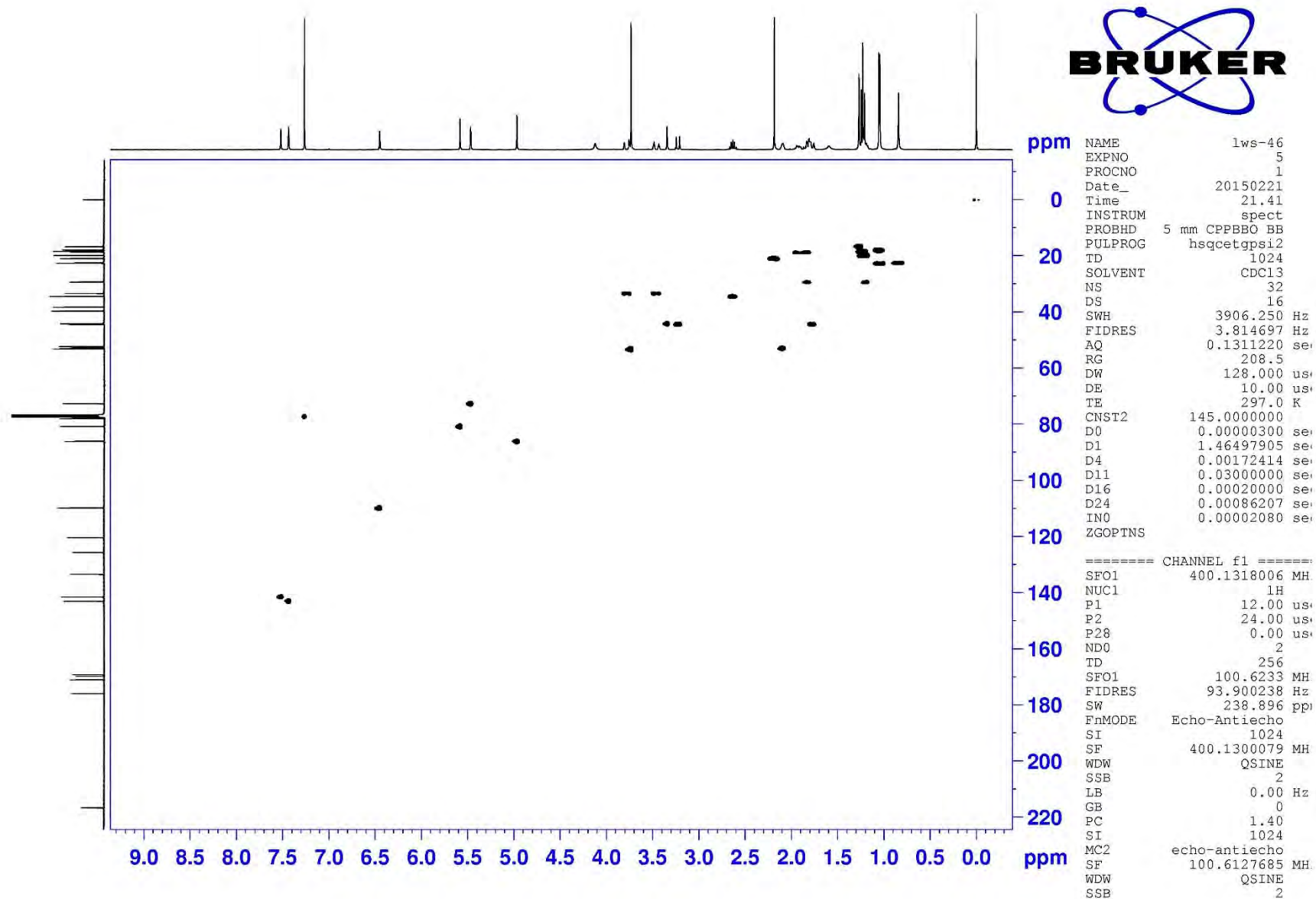


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$

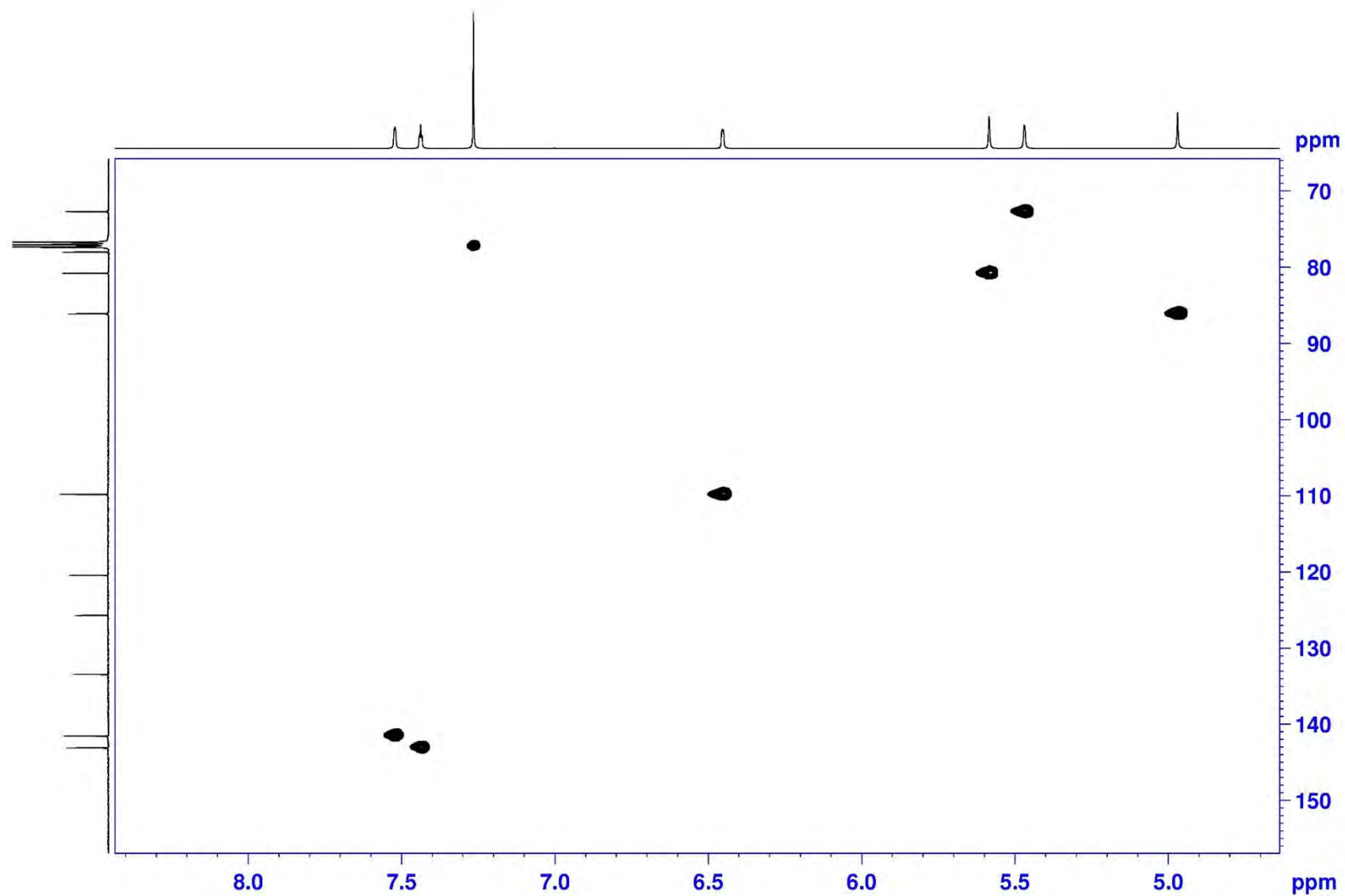




# HSQC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

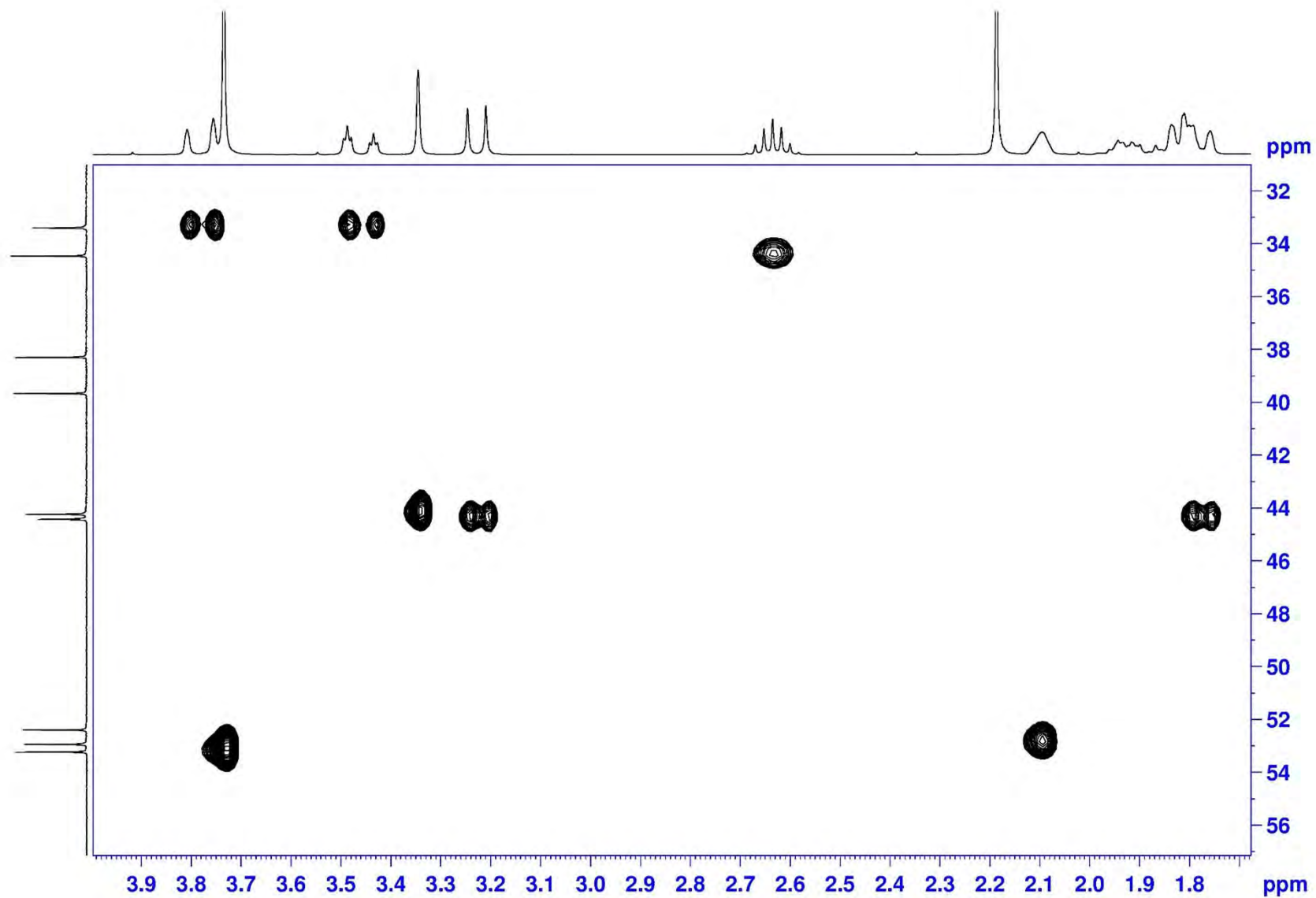


HSQC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

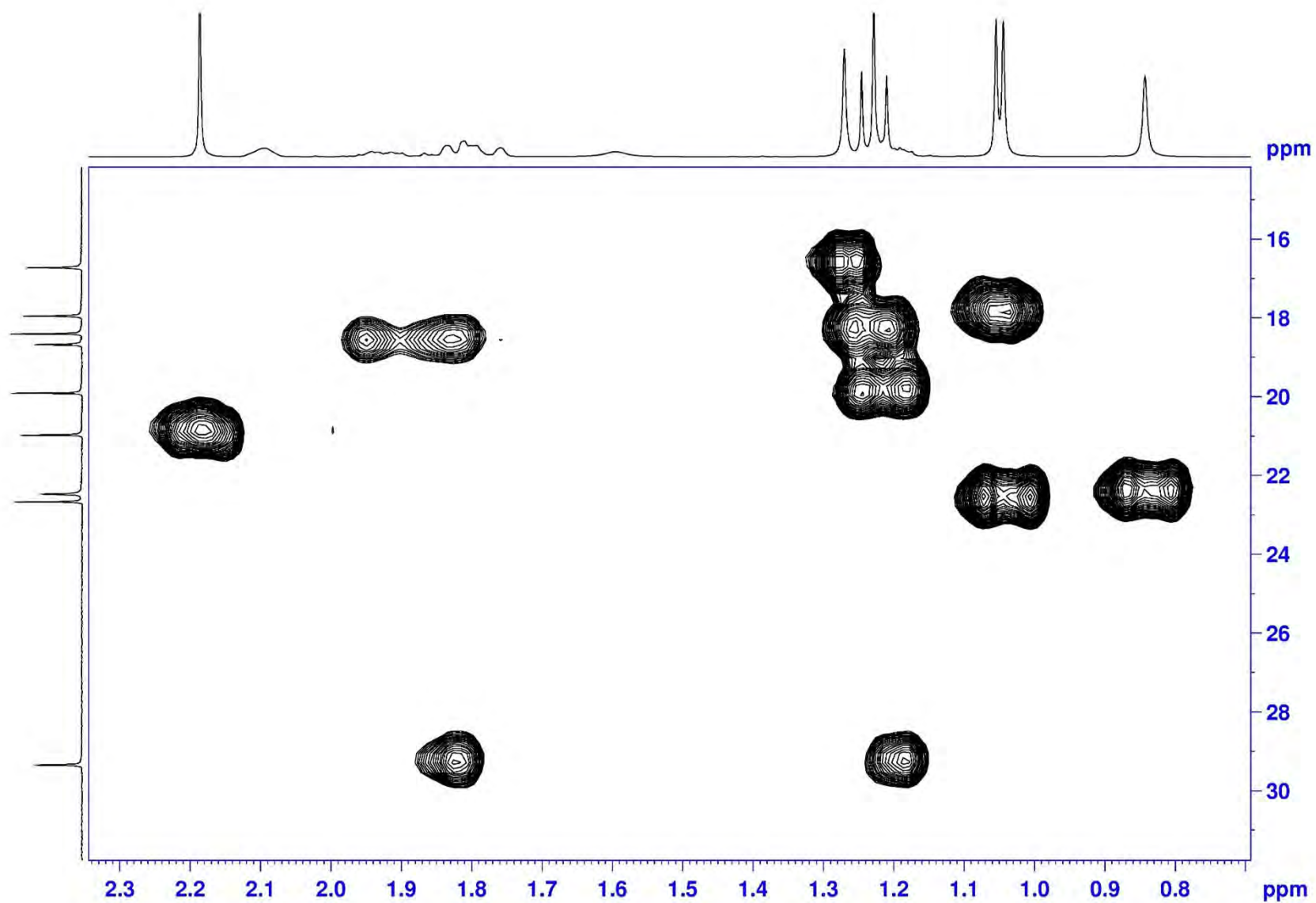




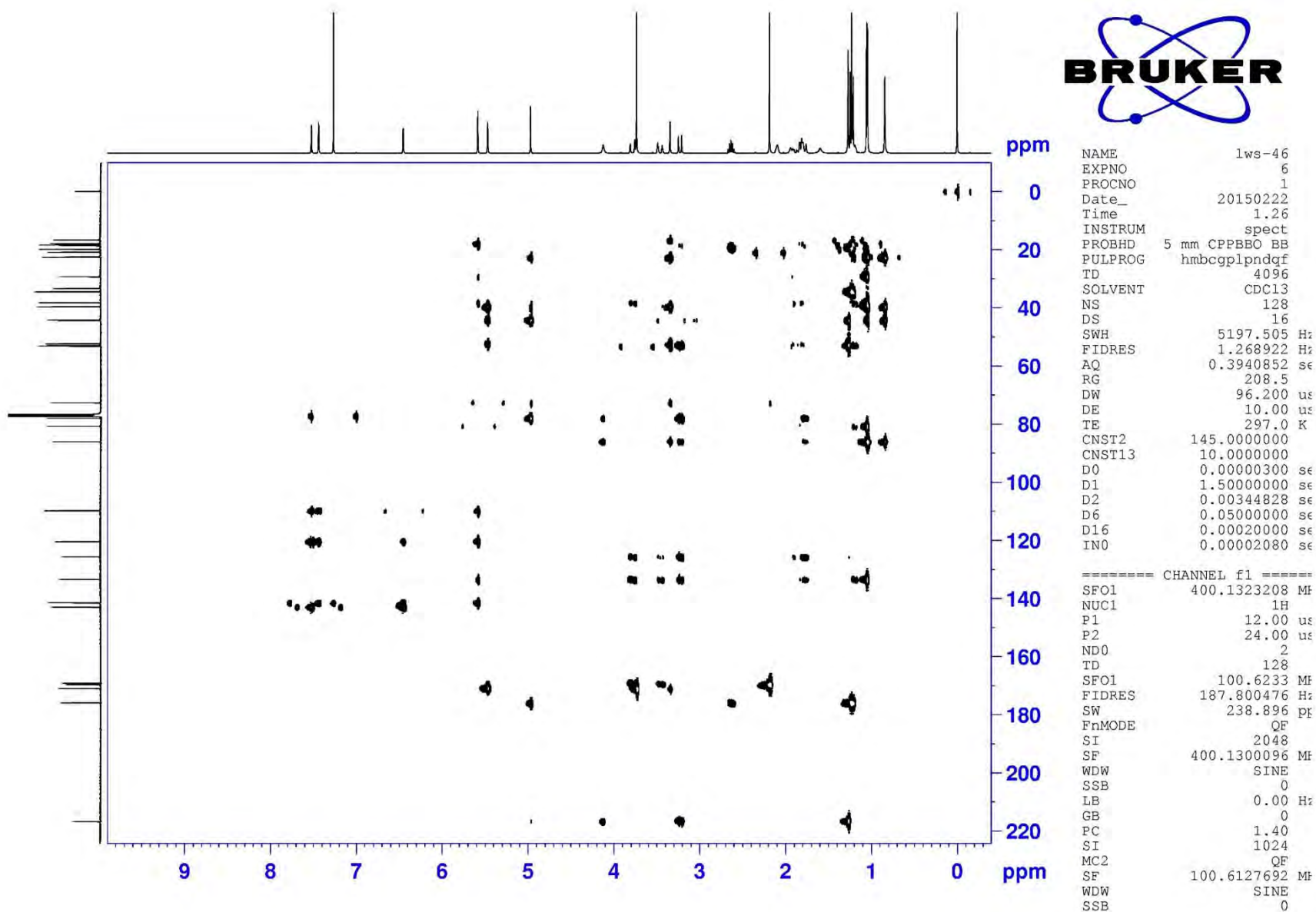
HSQC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



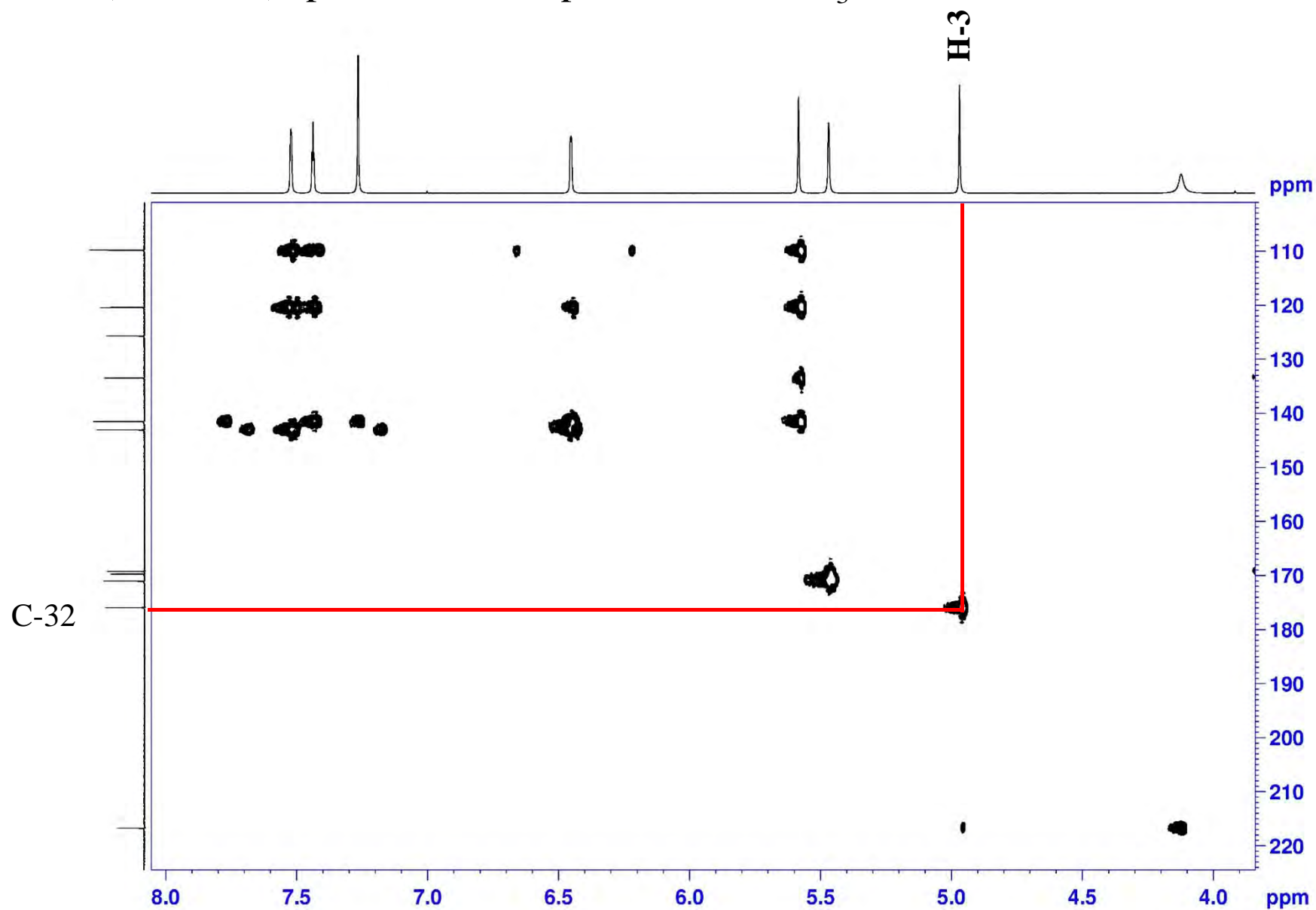
HSQC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



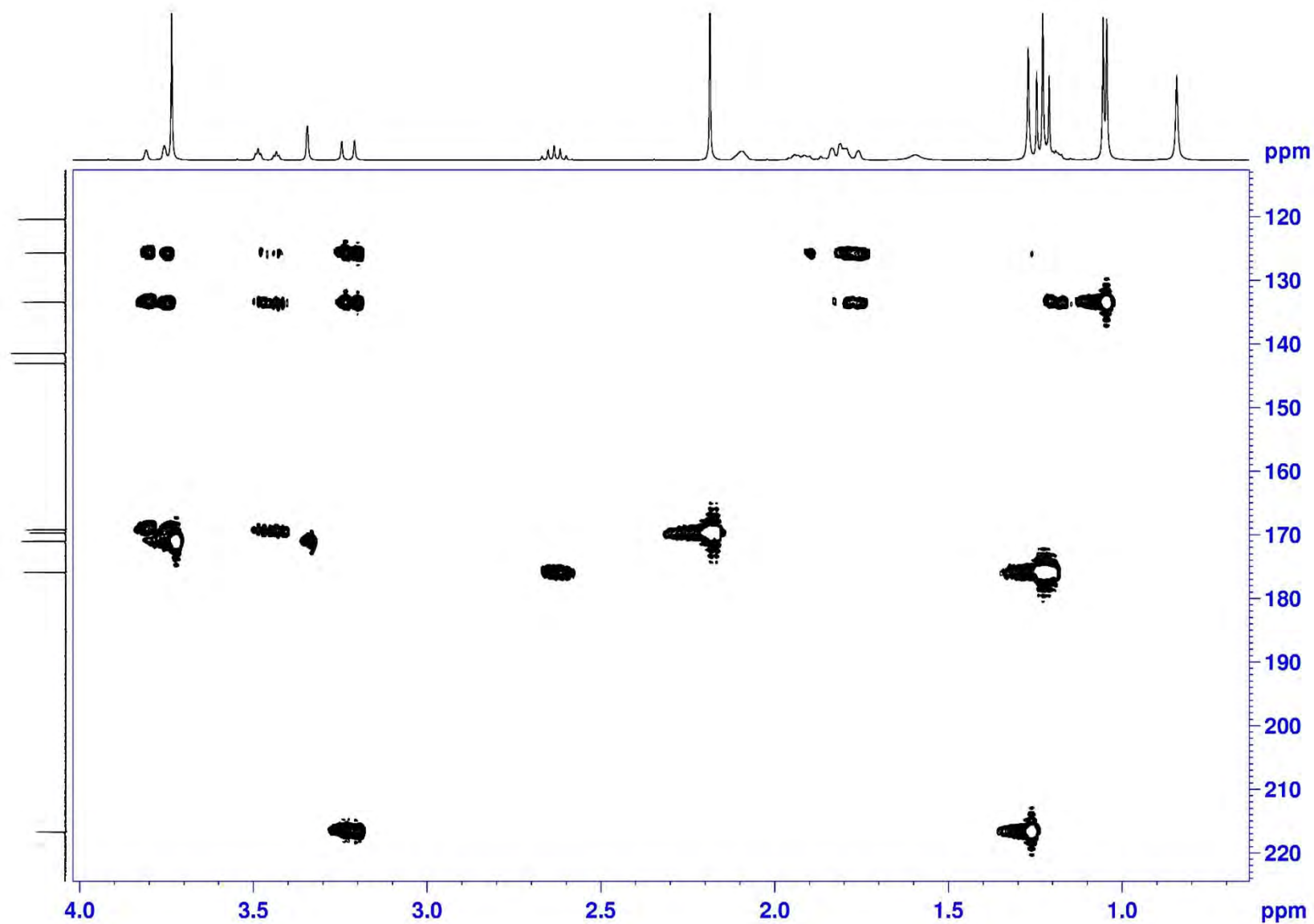
# HMBC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



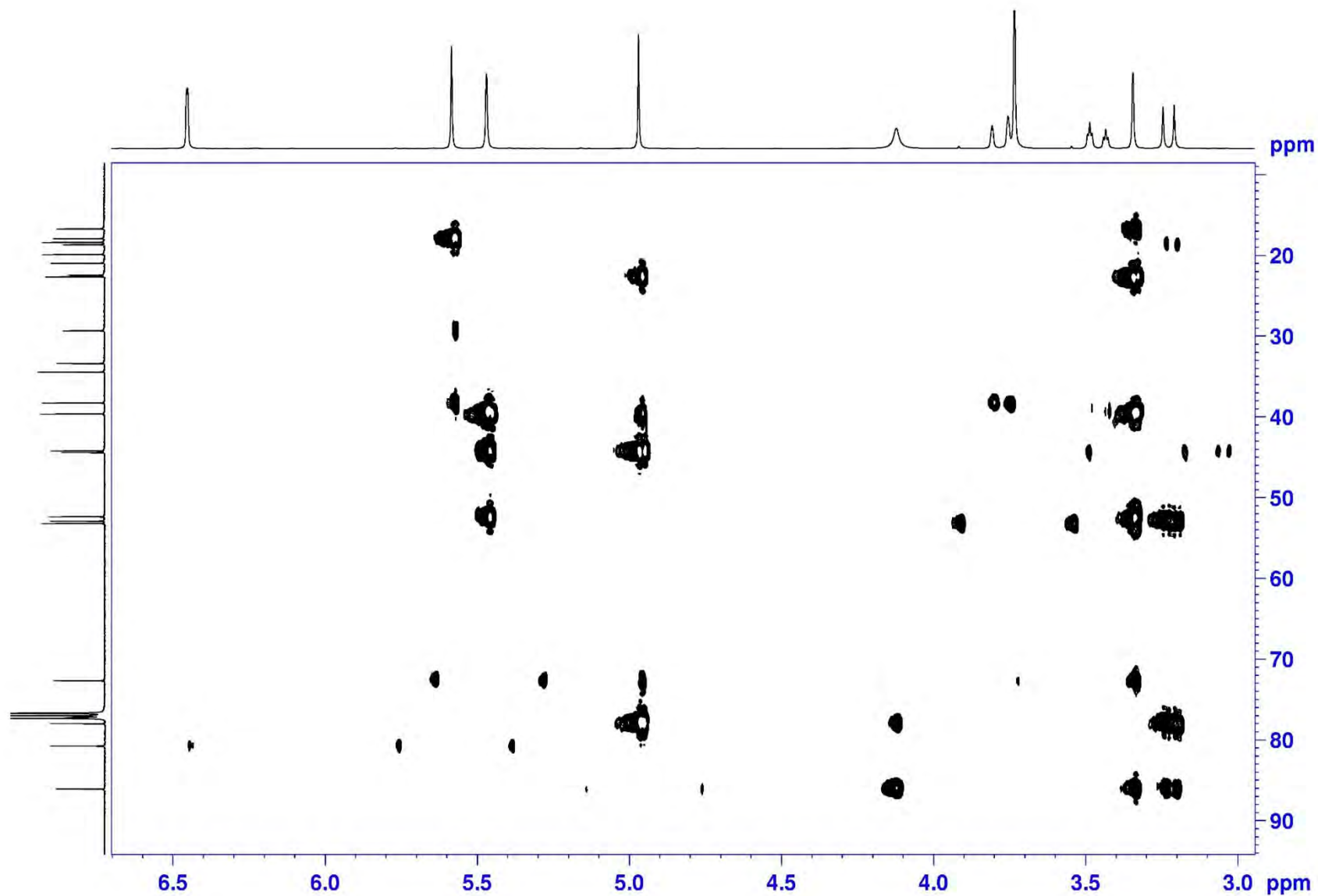
HMBC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



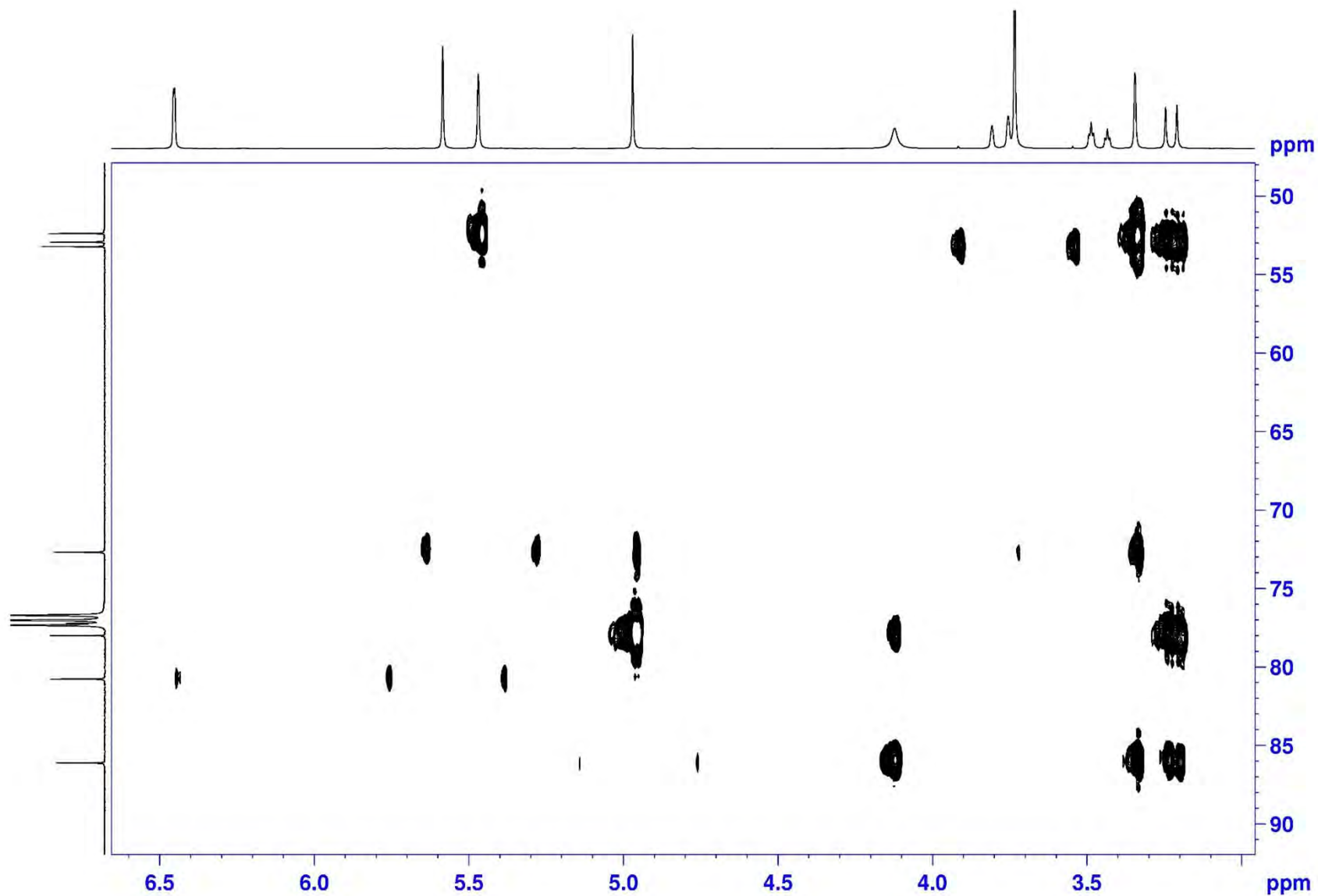
HMBC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

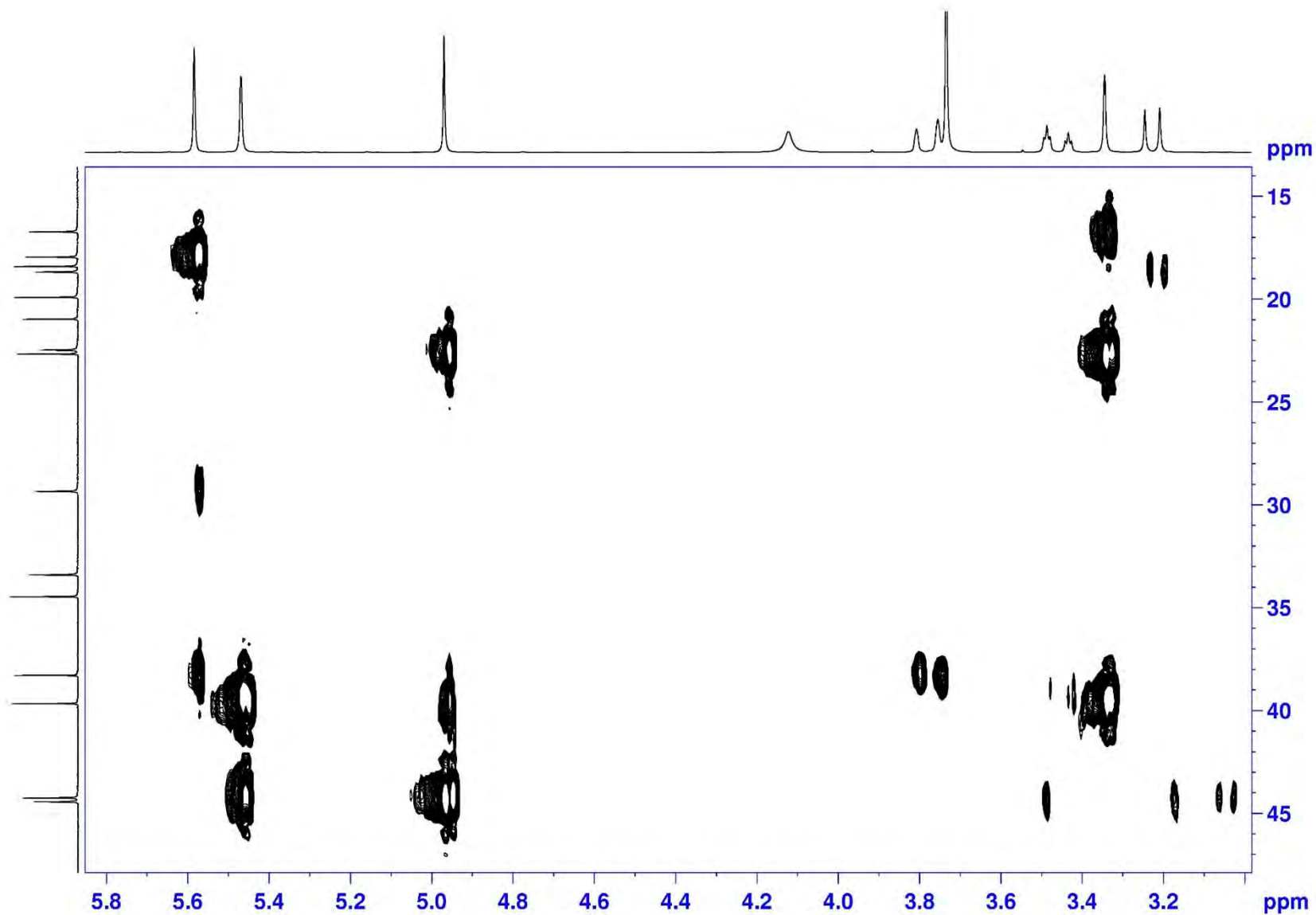


HMBC (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



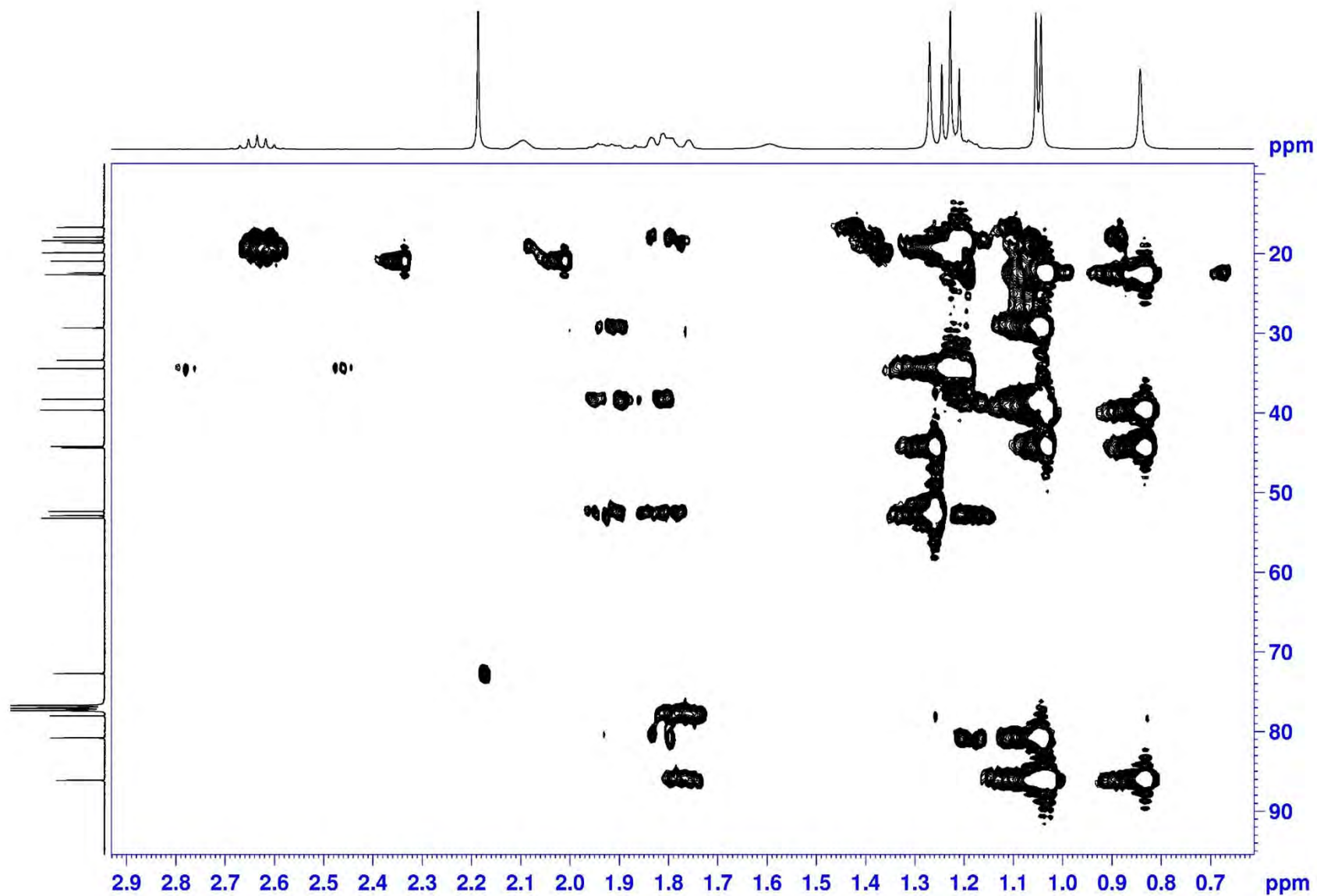


HMBC (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

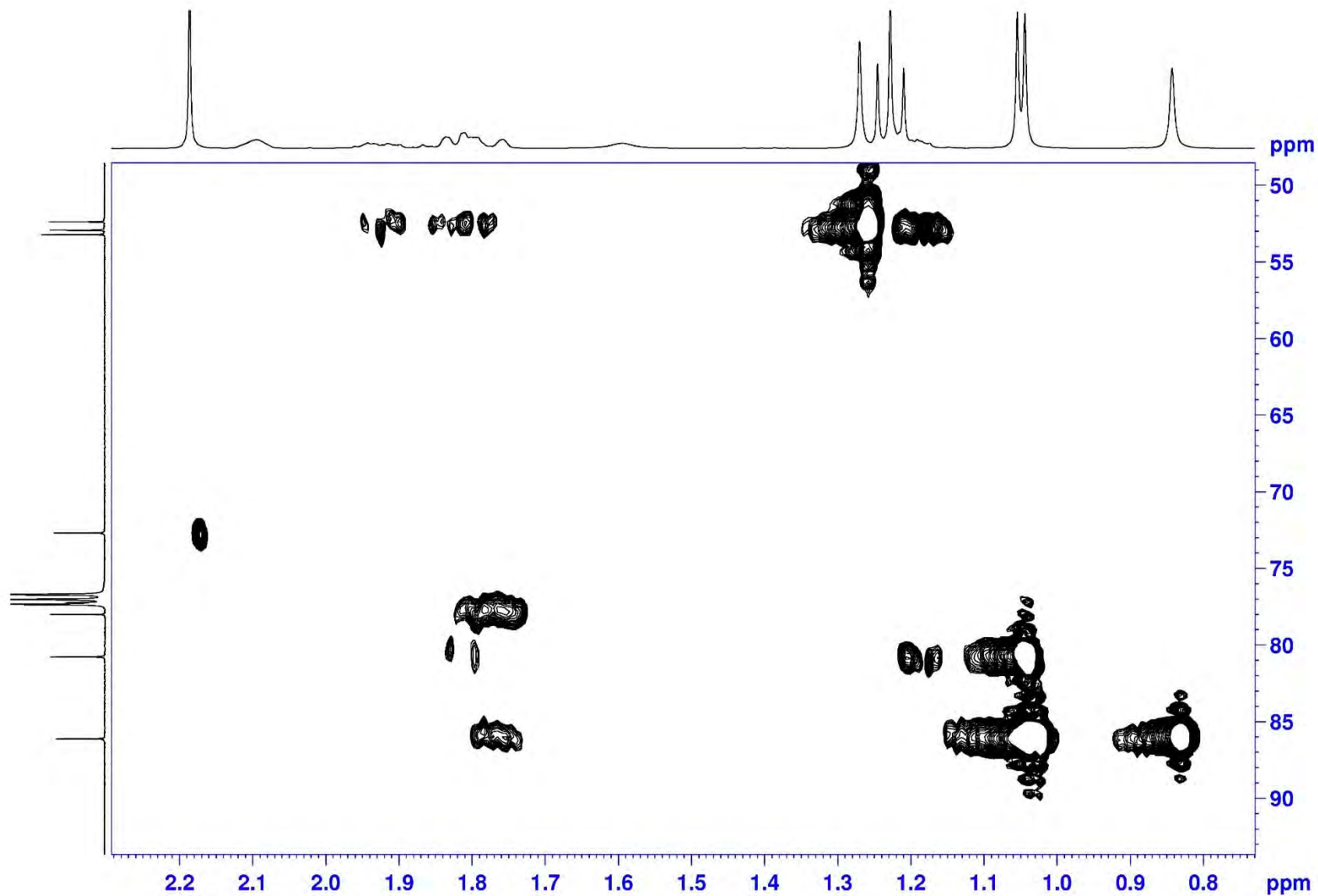




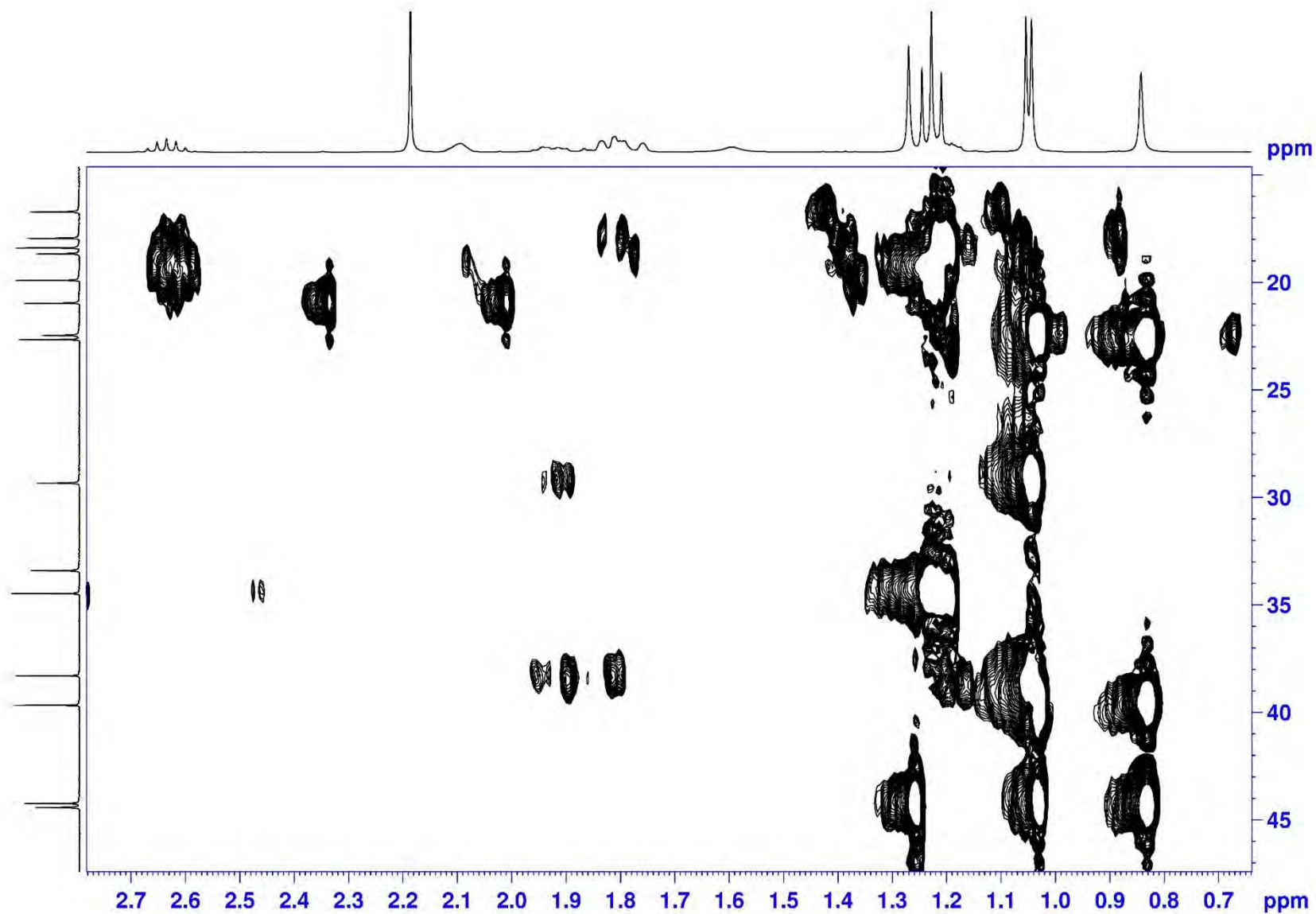
HMBC (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



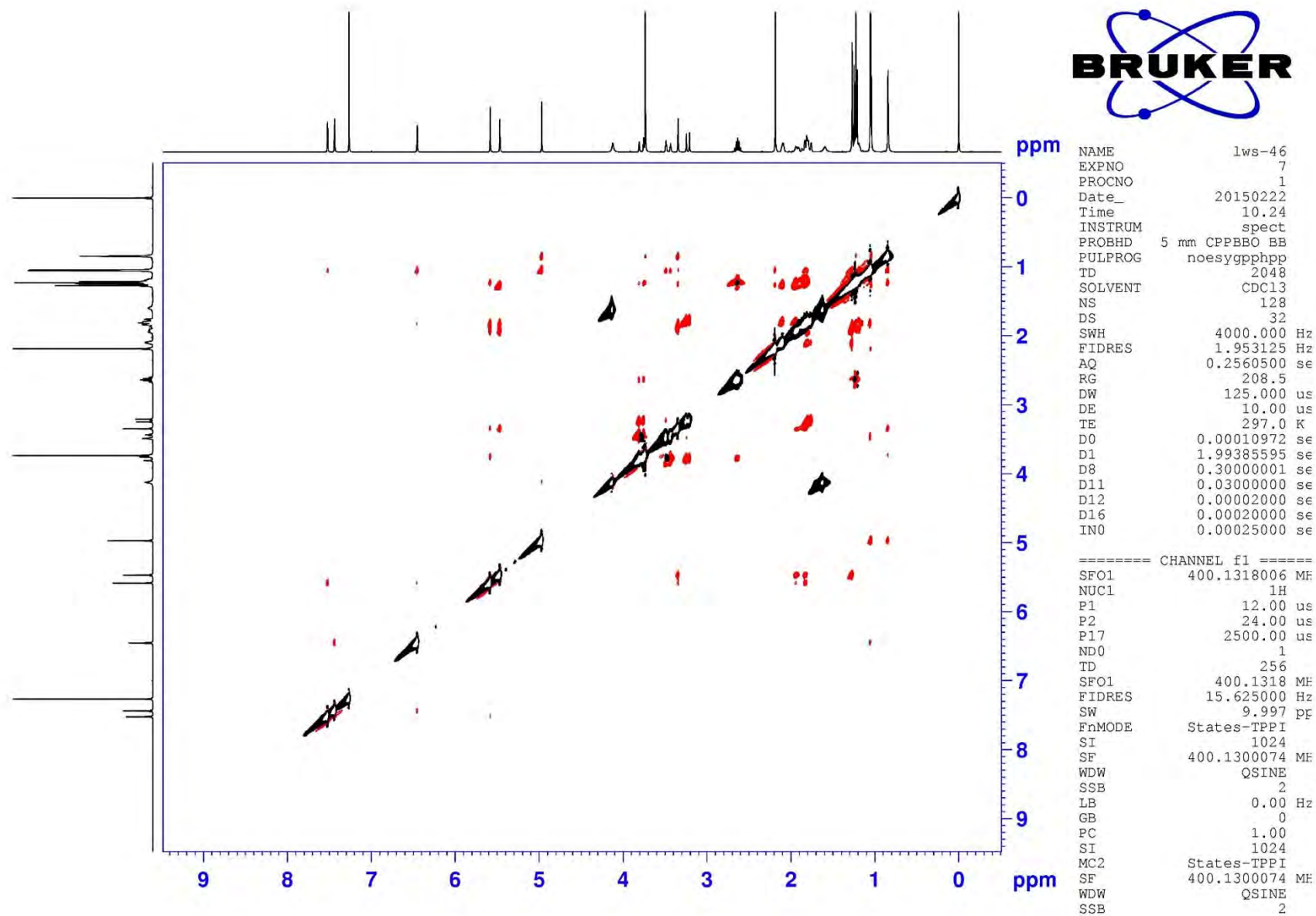
HMBC (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



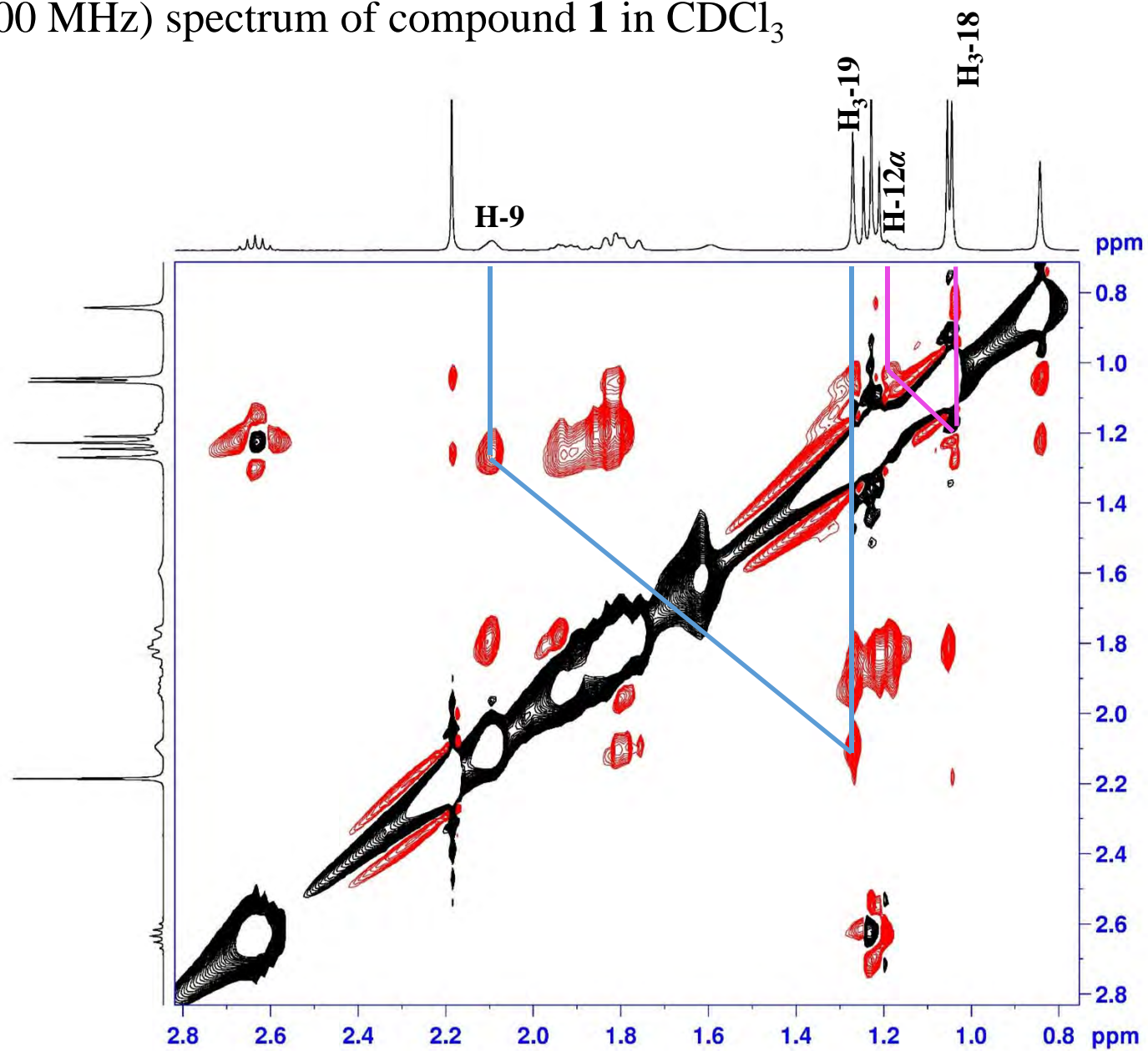
HMBC (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **1** in CDCl<sub>3</sub>

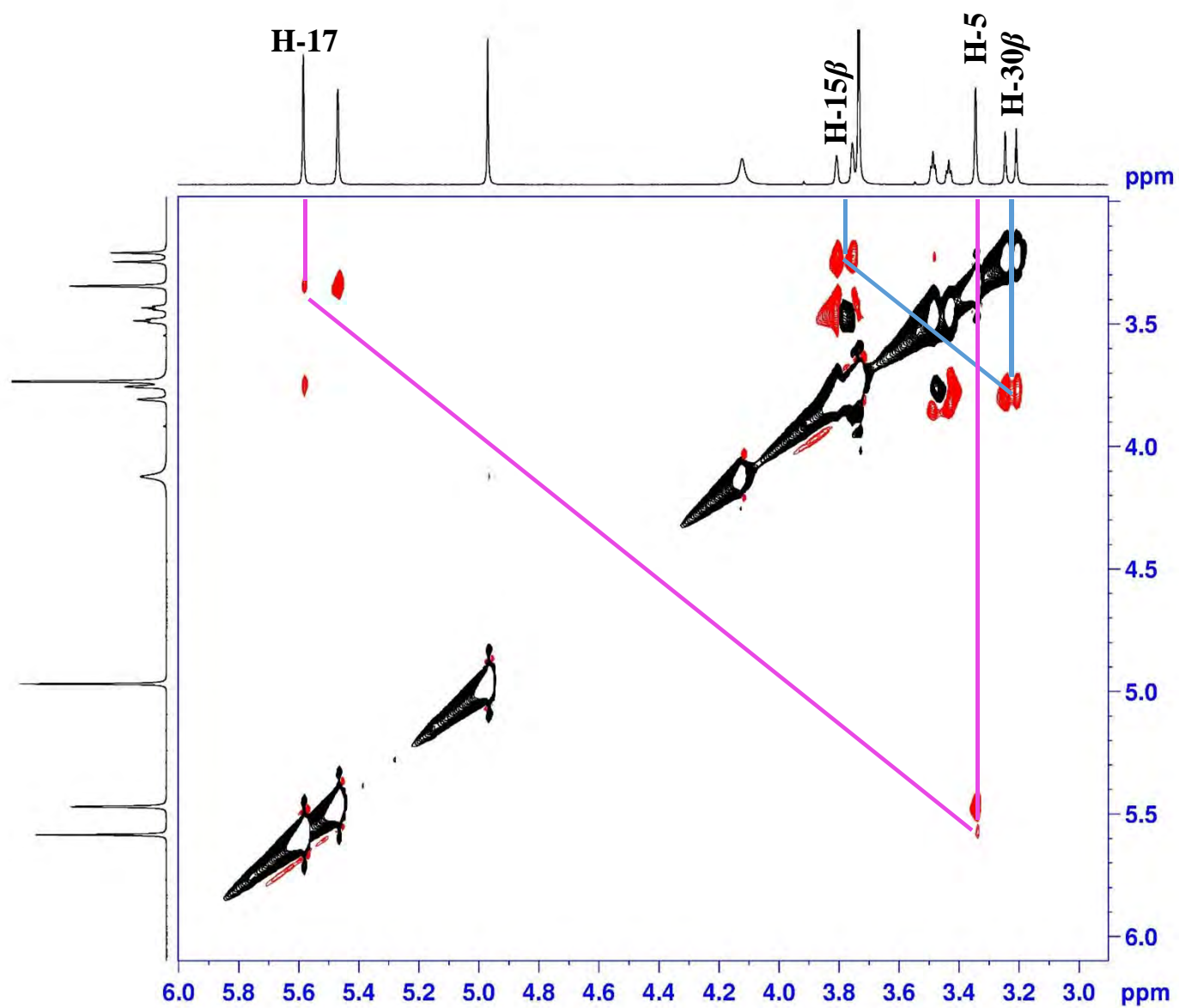


NOESY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$

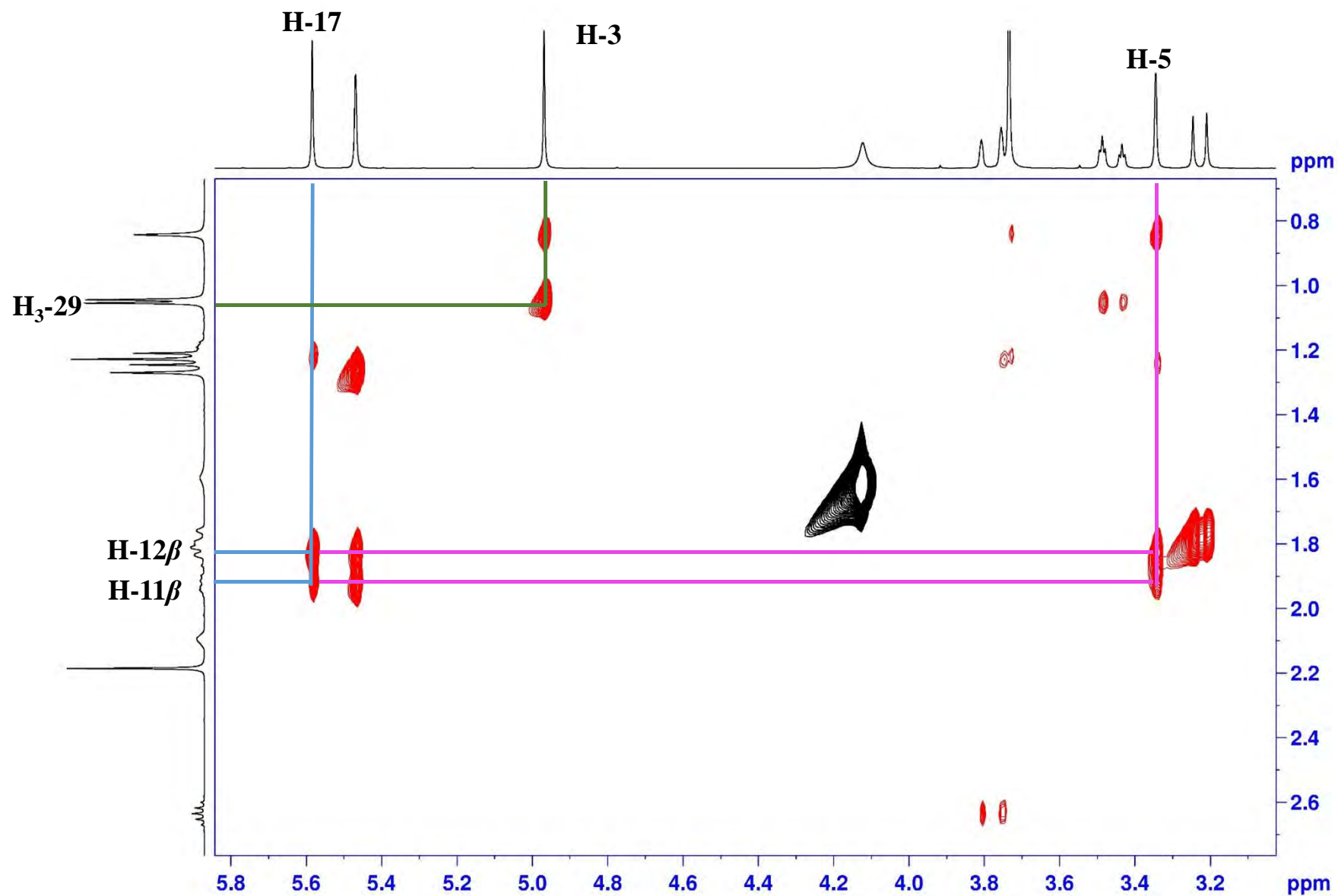




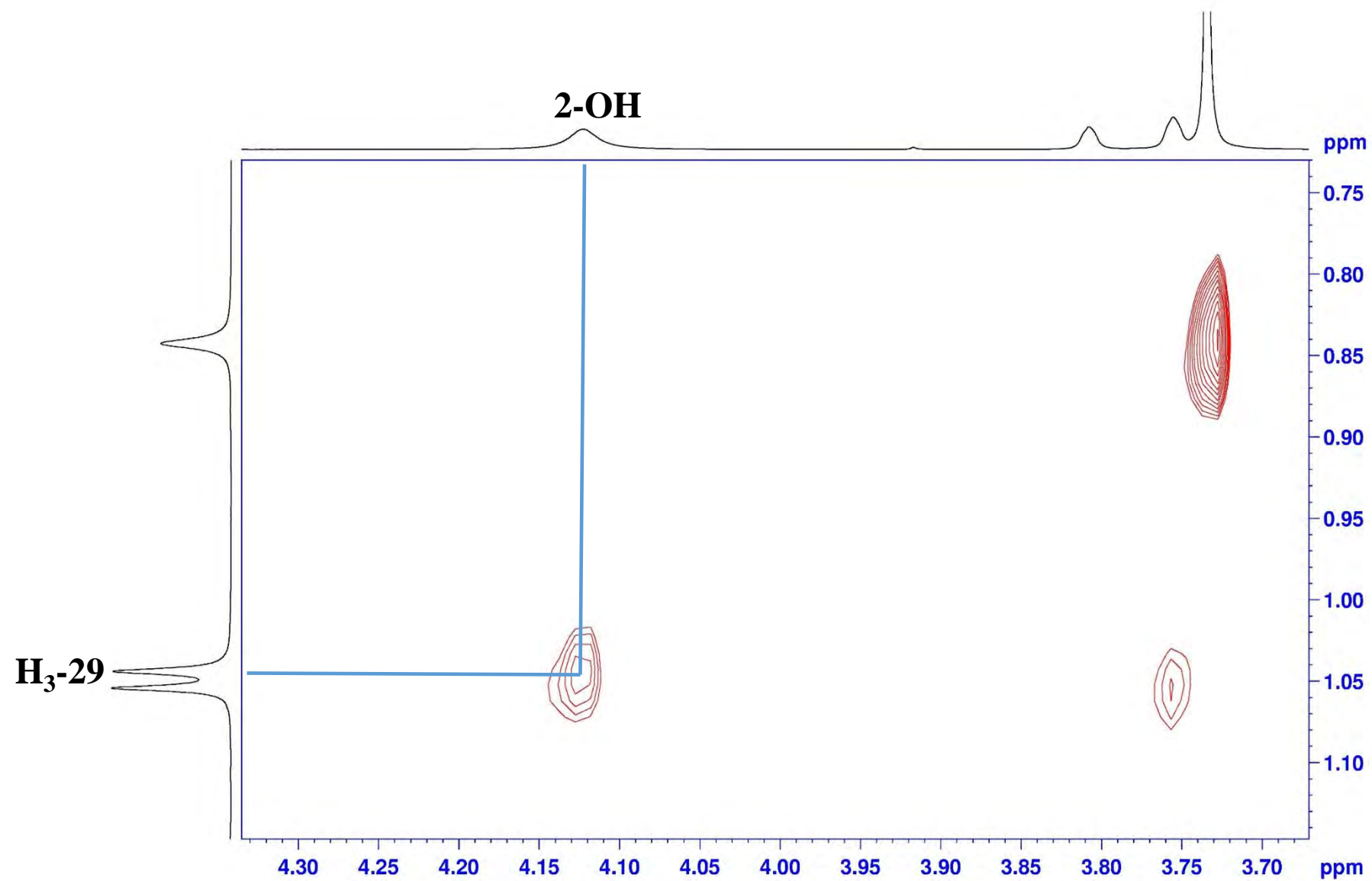
NOESY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$

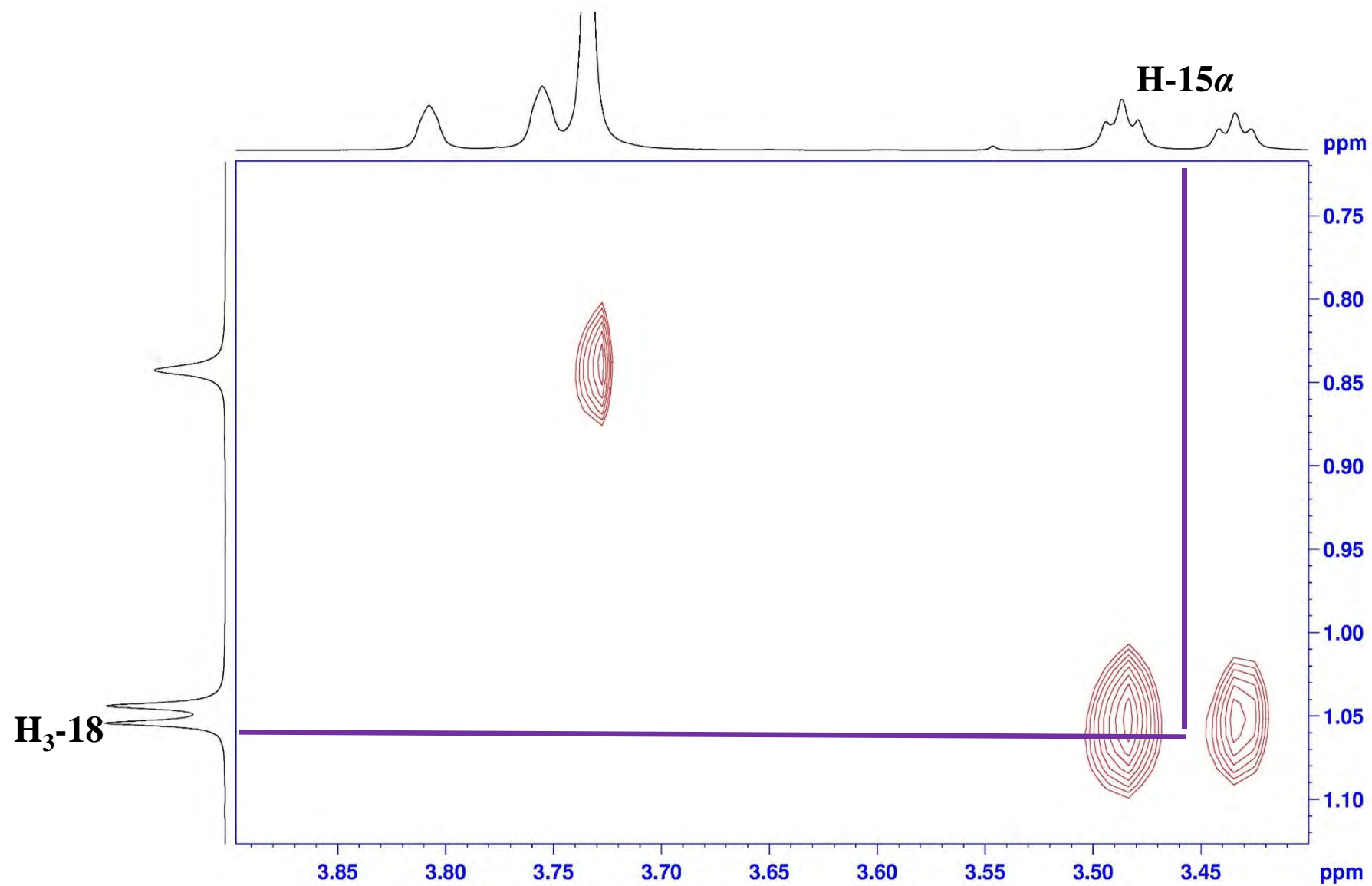


NOESY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$





NOESY (400 MHz) spectrum of compound **1** in  $\text{CDCl}_3$



# HRESIMS for compound 2

## Mass Spectrum SmartFormula Report

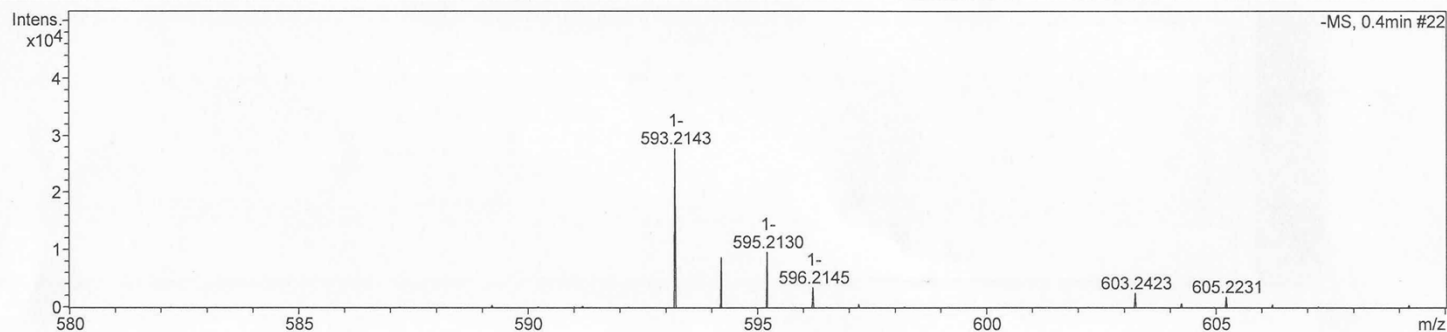
### Analysis Info

Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-26\_neg\_83\_01\_2736.d  
 Method LC\_Direct Infusion\_neg\_100-1000mz.m  
 Sample Name zhangjianzhi\_zjz-26\_neg  
 Comment

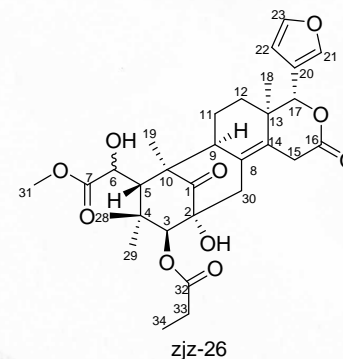
Acquisition Date 12/12/2016 3:15:13 PM  
 Operator SCSIO  
 Instrument maXis 255552.00029

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e <sup>-</sup> Conf	N-Rule
593.214270	1	C30H38ClO10	100.00	593.215899	2.7	1.6	21.1	11.5	even	ok
1151.459232	1	C60H76ClO20	100.00	1151.462396	-2.7	-3.2	23.0	22.5	even	ok



zhangjianzhi\_zjz-26\_neg\_83\_01\_2736.d

Bruker Compass DataAnalysis 4.1

printed: 12/12/2016 3:24:22 PM

by: SCSIO

Page 1 of 1

# HRESIMS for compound 2

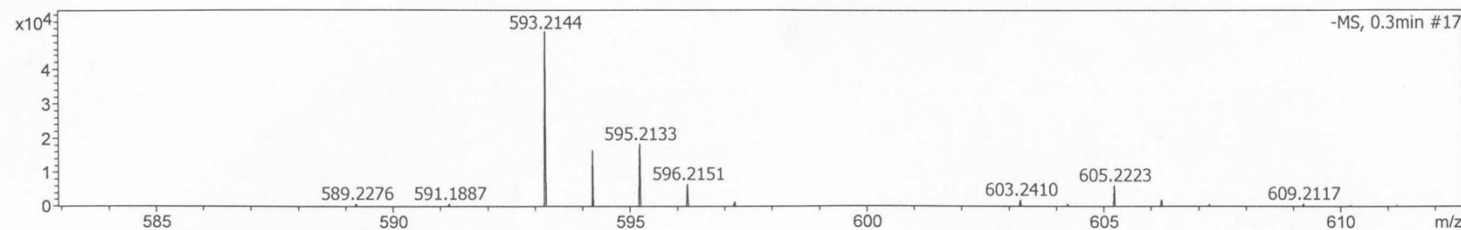
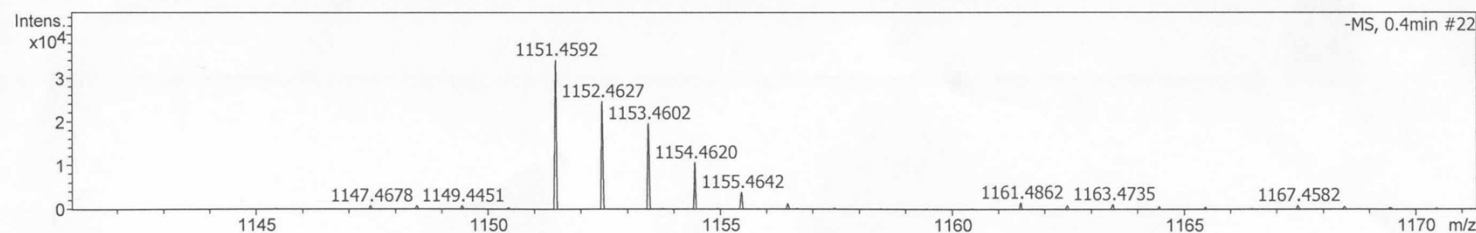
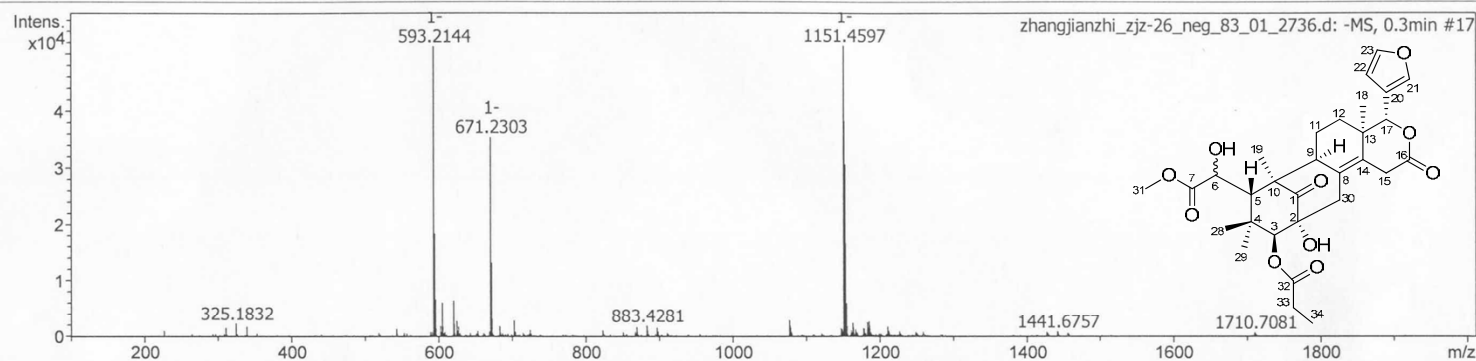
## Generic Display Report

### Analysis Info

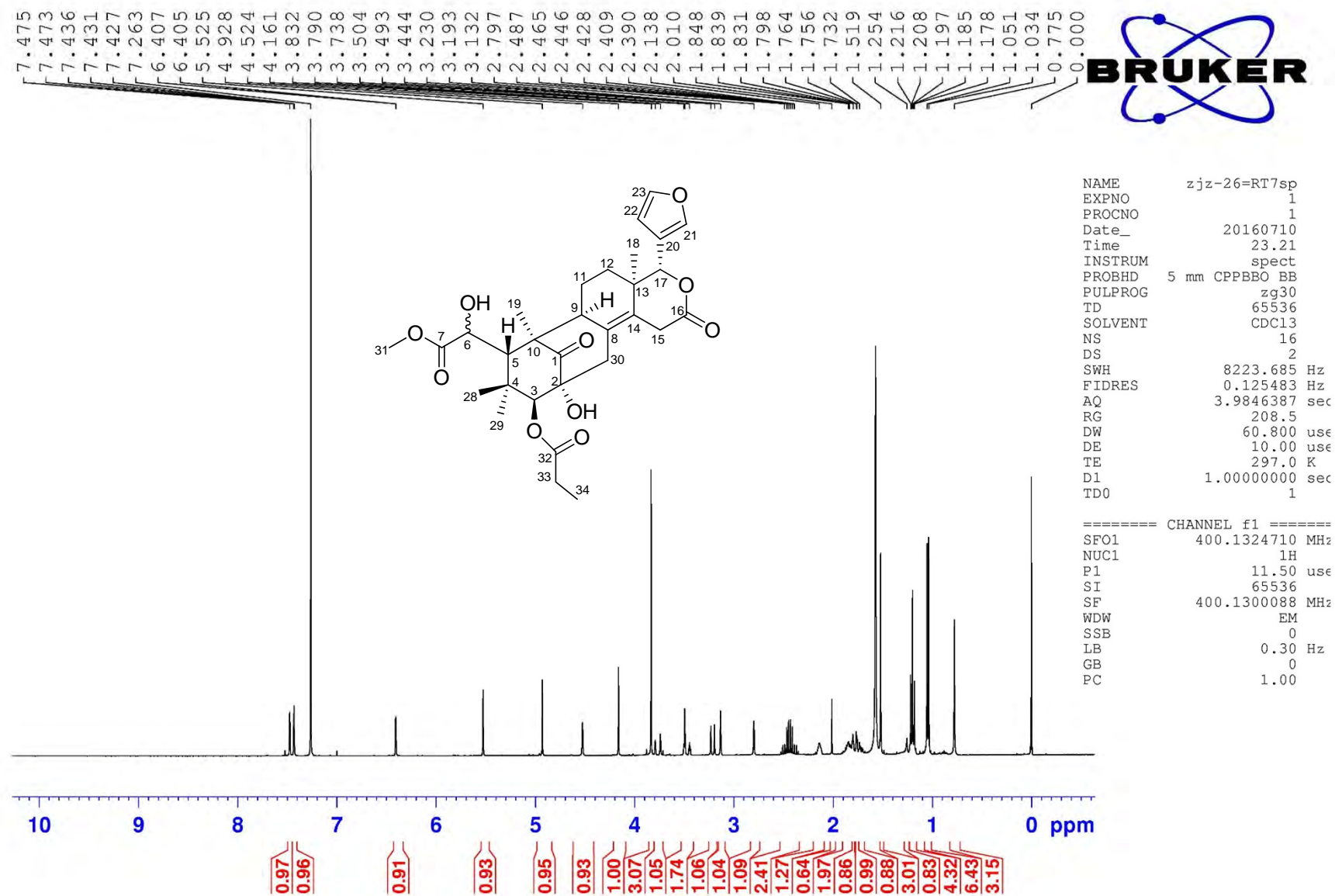
Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-26\_neg\_83\_01\_2736.d  
Method LC\_Direct Infusion\_neg\_100-1000mz.m  
Sample Name zhangjianzhi\_zjz-26\_neg  
Comment

Acquisition Date 12/12/2016 3:15:13 PM

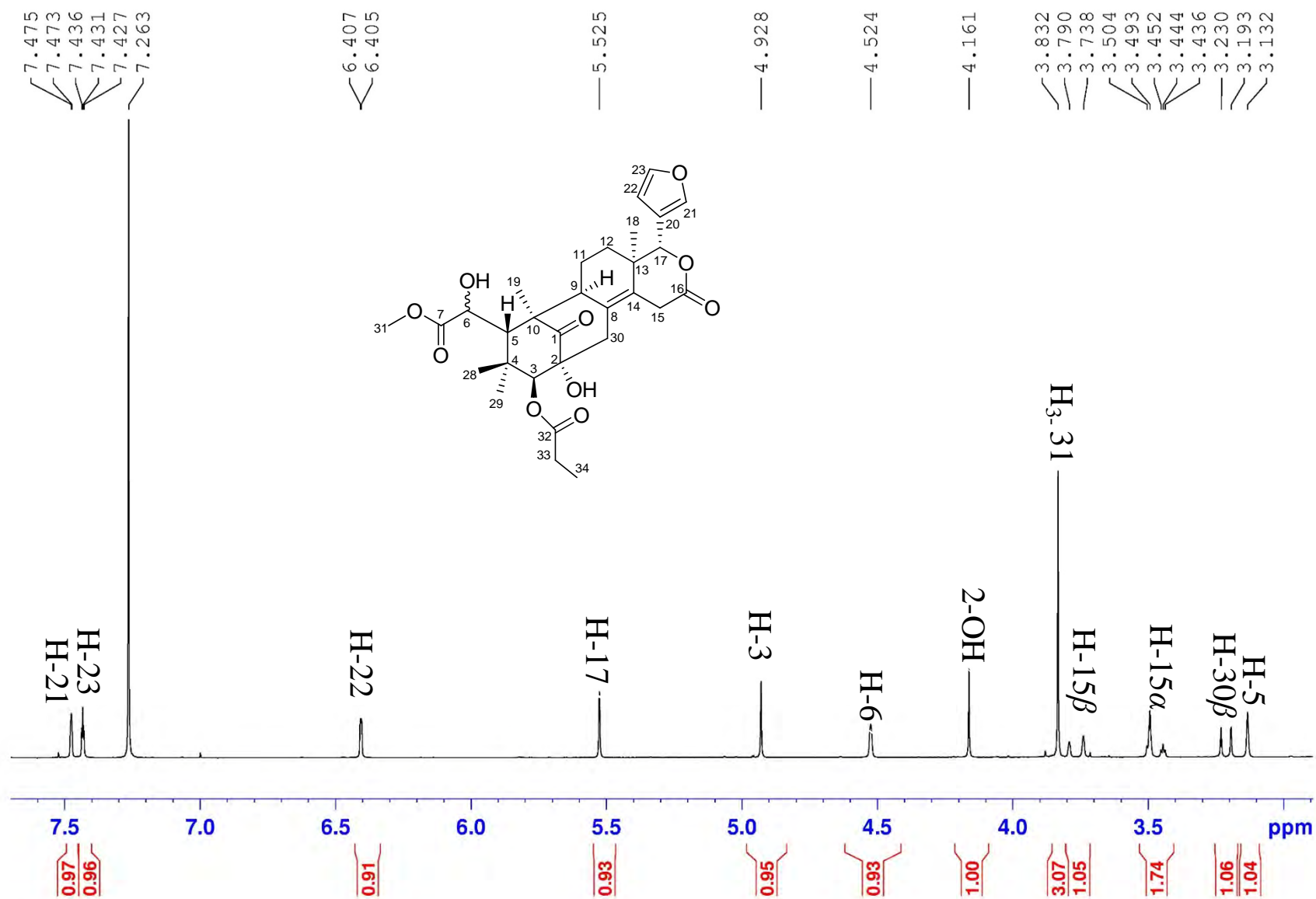
Operator SCSIO  
Instrument maXis



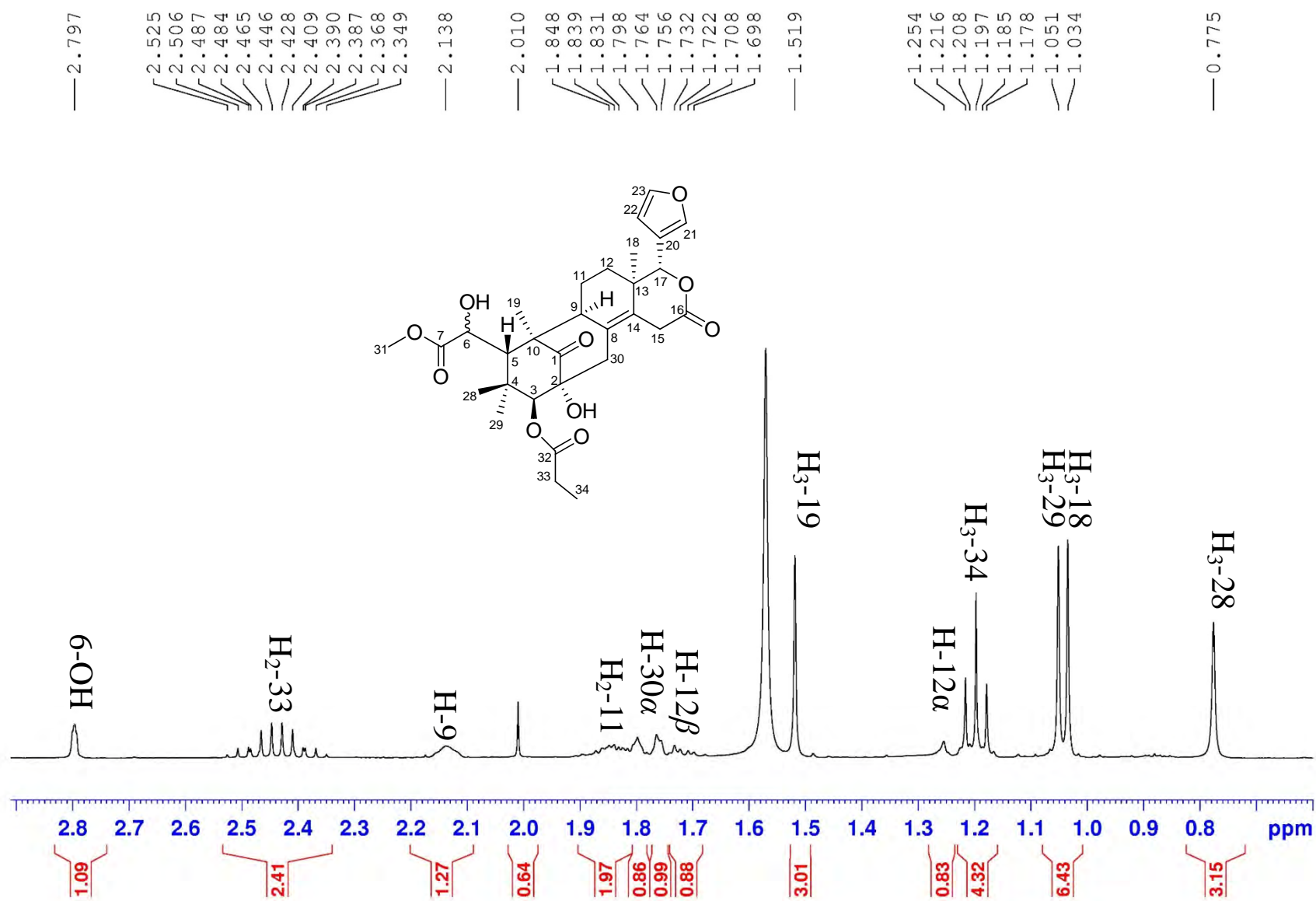
<sup>1</sup>H NMR (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



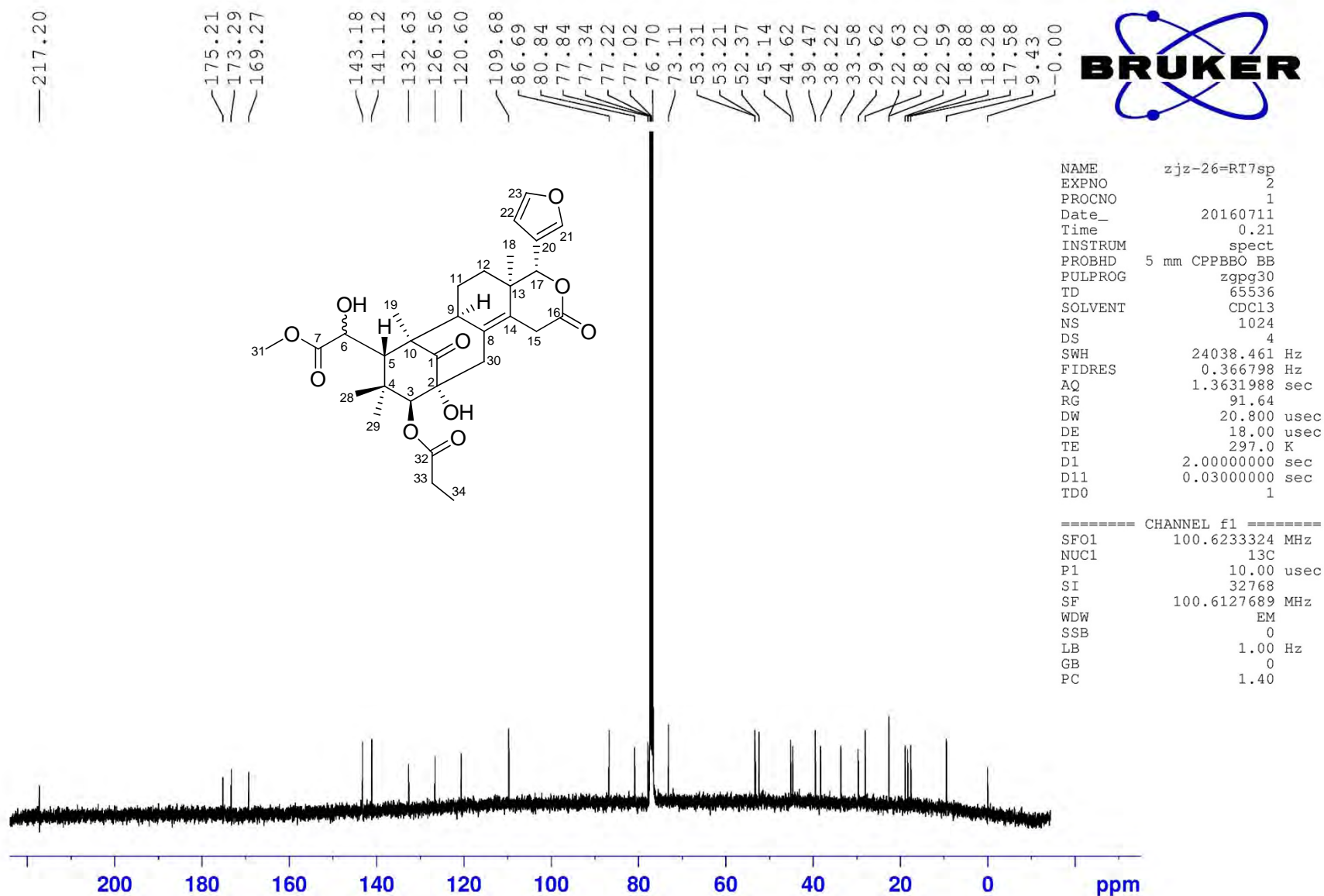
$^1\text{H}$  NMR (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$

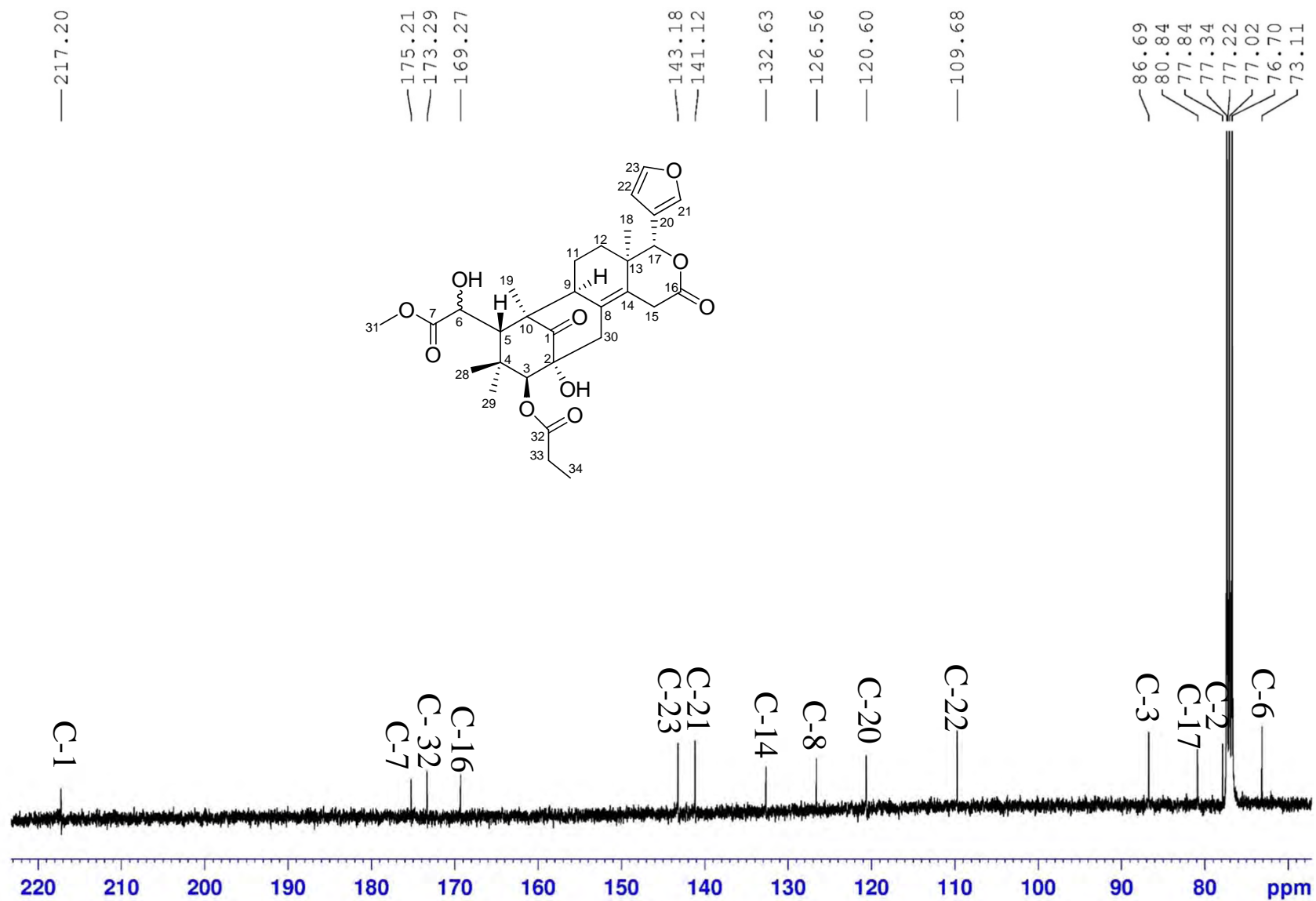


$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **2** in  $\text{CDCl}_3$





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





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

53.31  
53.21  
52.37

45.14  
44.62

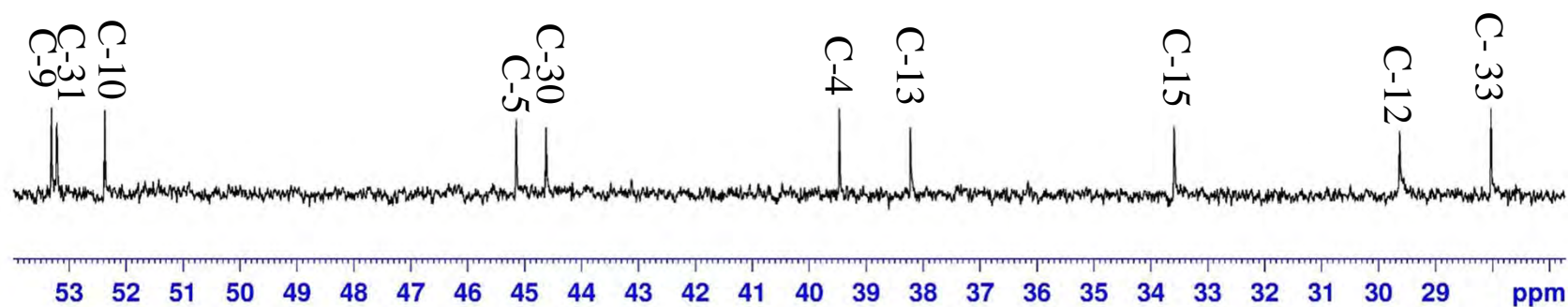
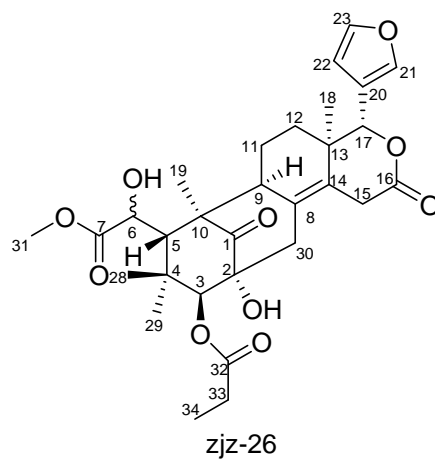
39.47

38.22

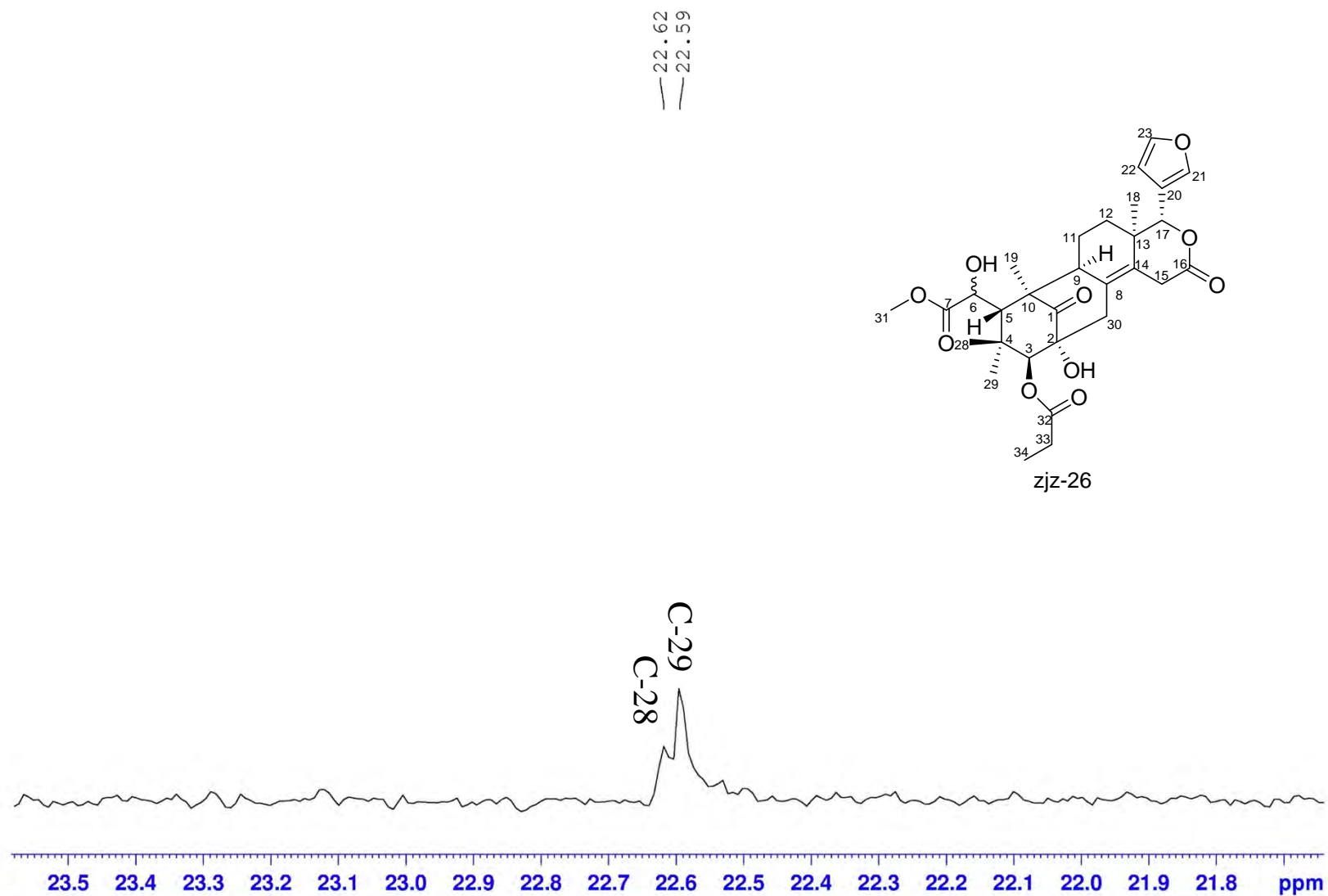
33.58

29.62

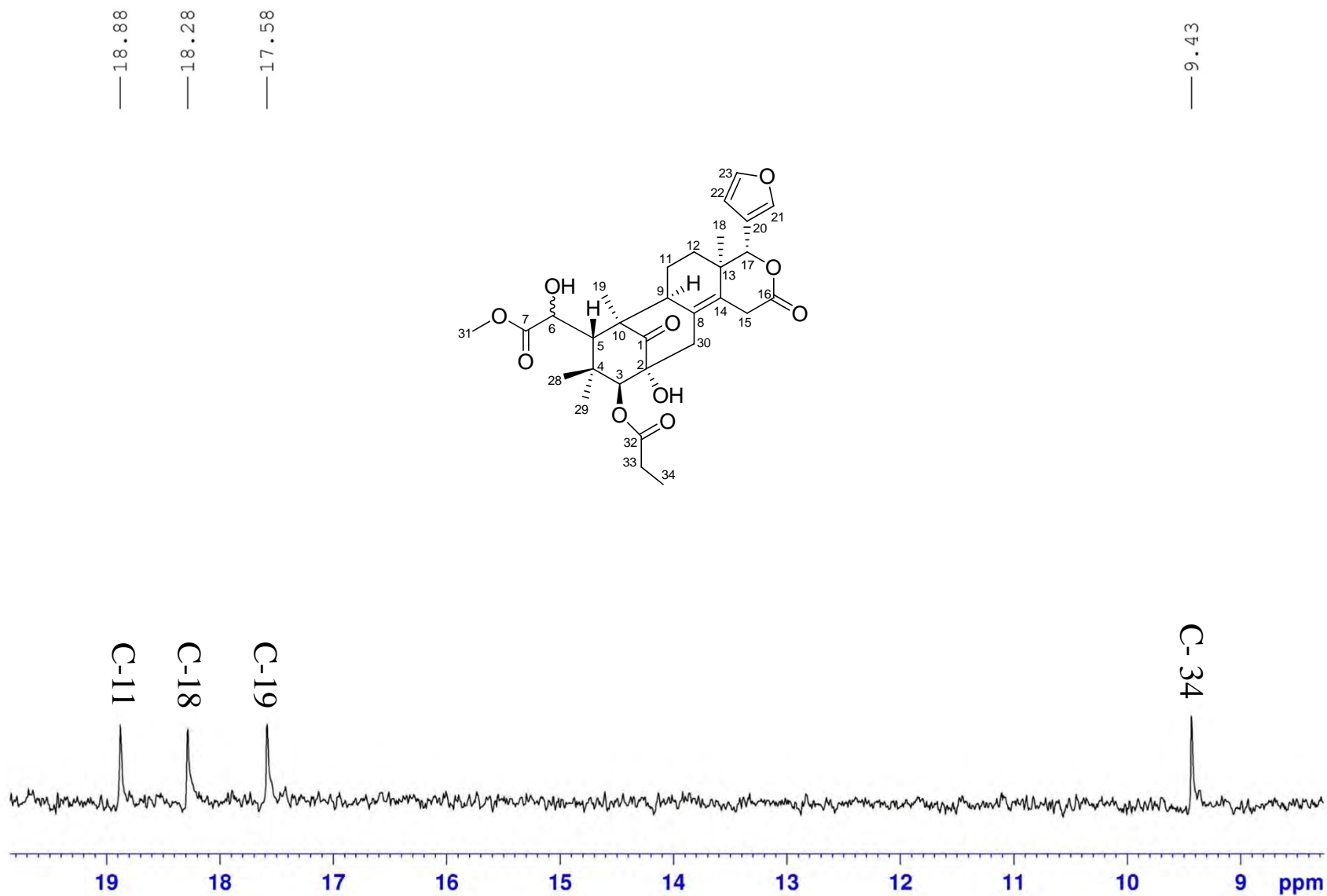
28.02



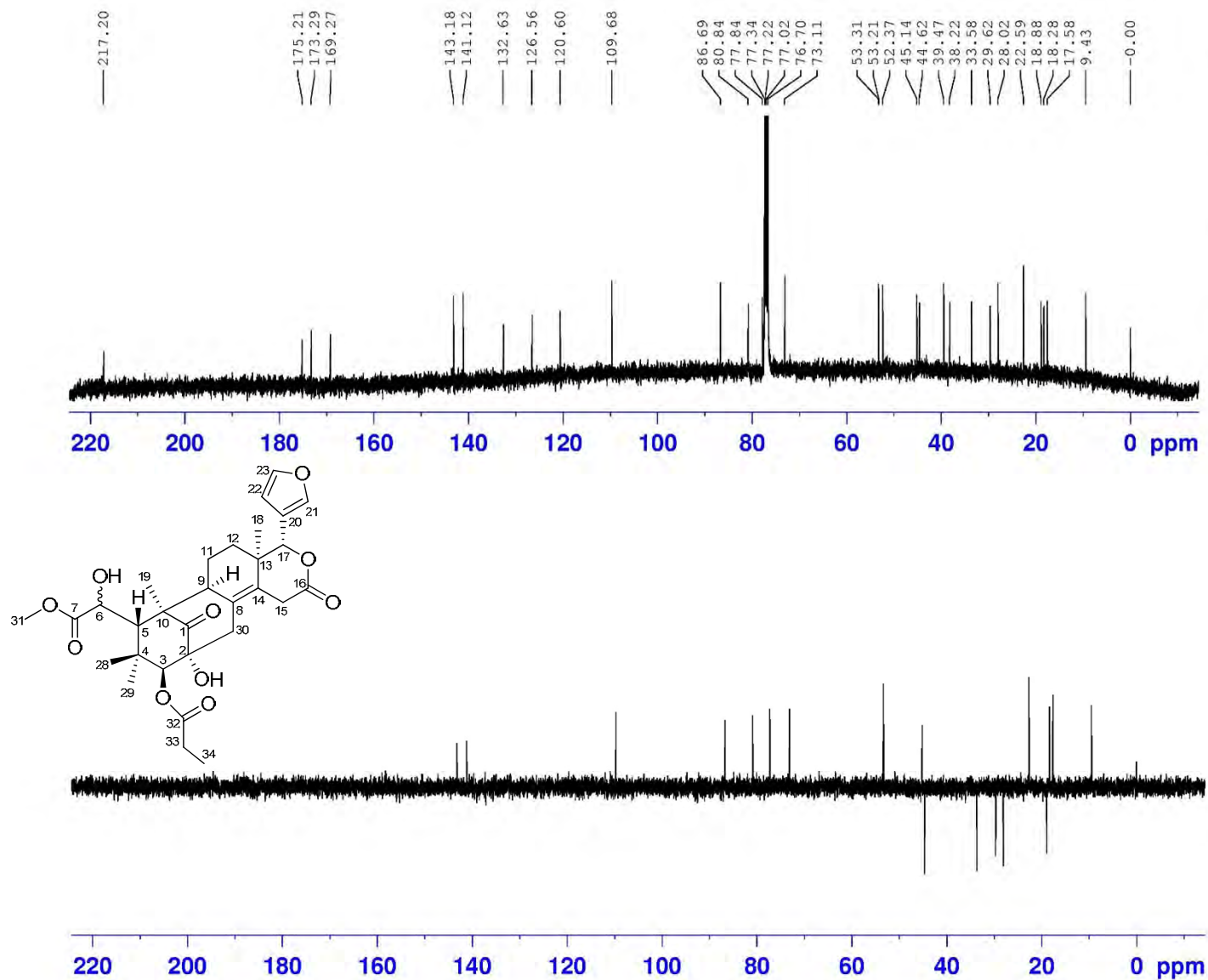
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



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



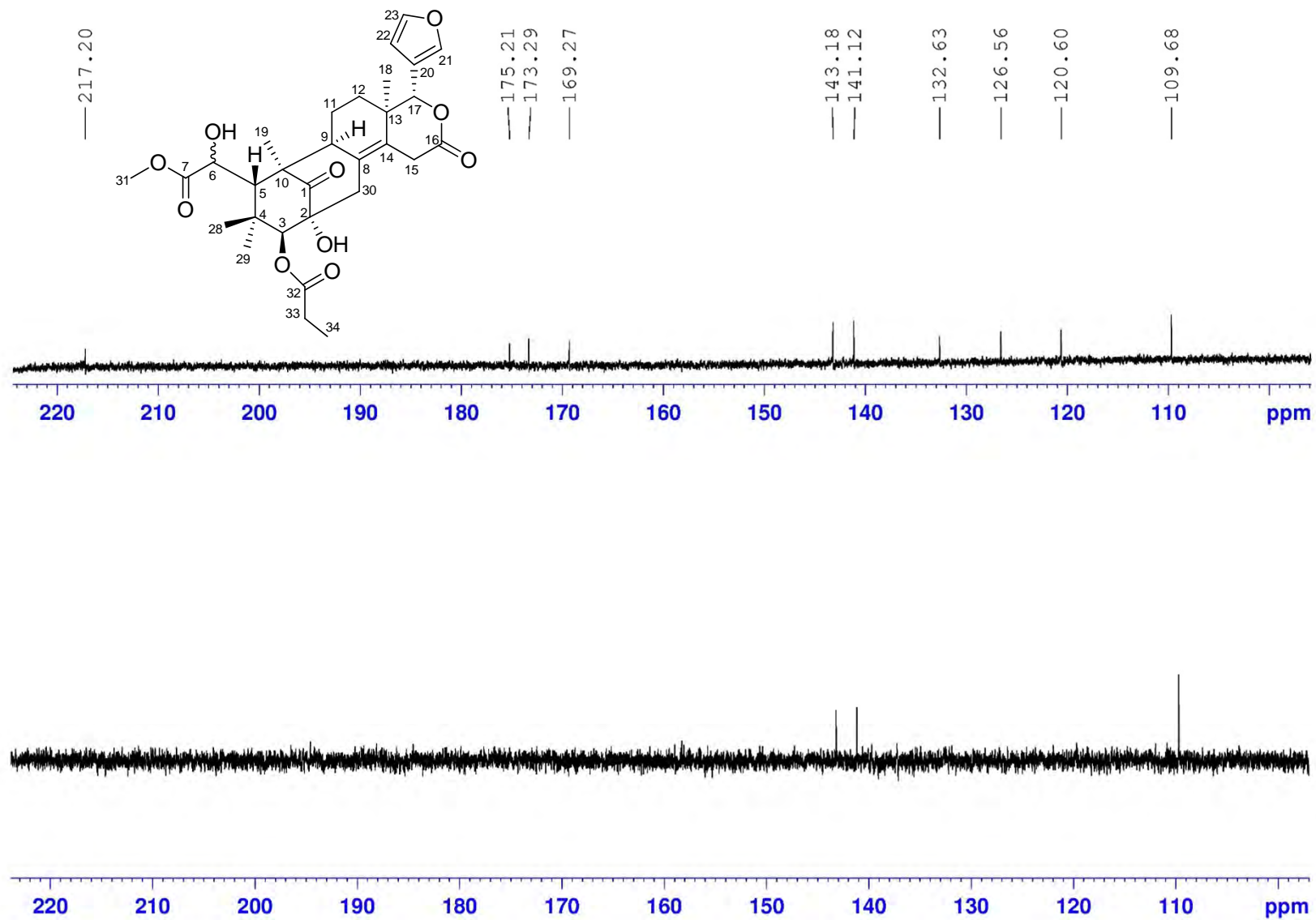
# DEPT135 (100 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



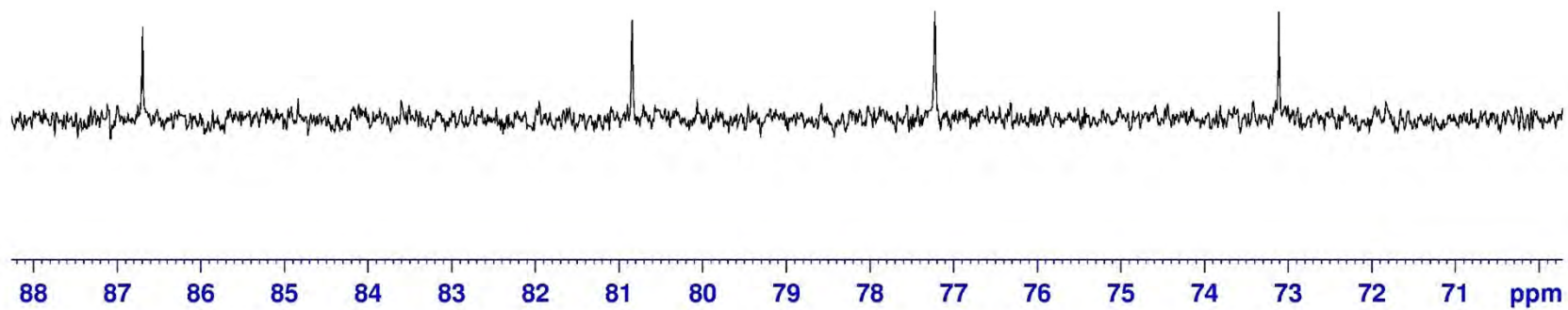
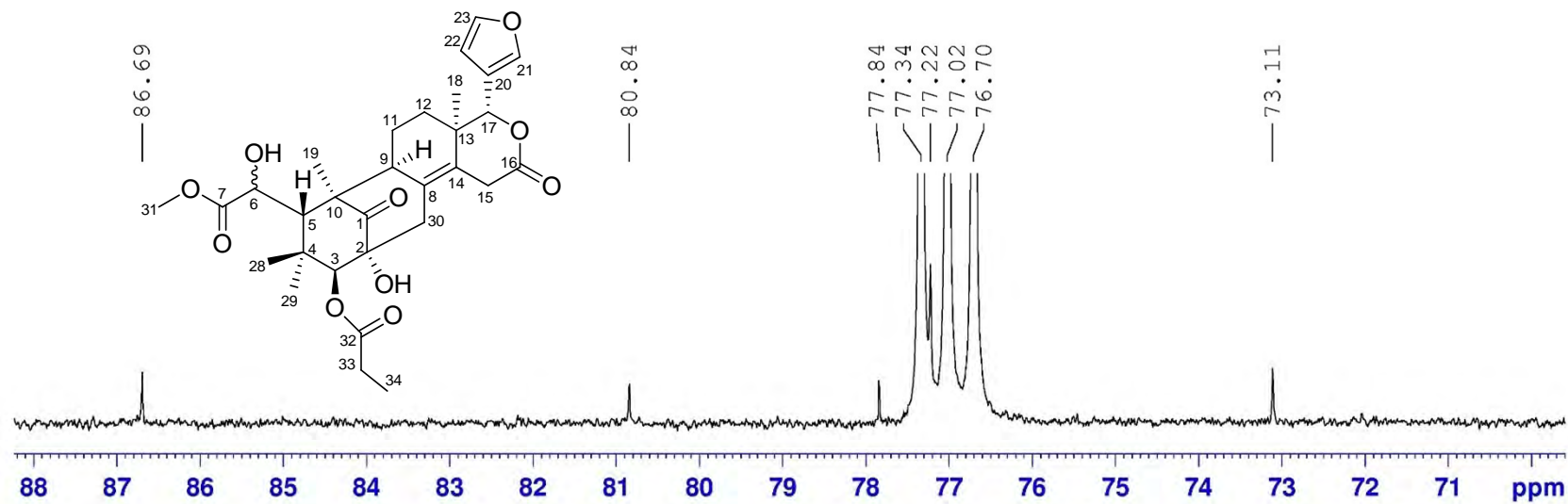
NAME zjz-26=RT7sp  
 EXPNO 3  
 PROCNO 1  
 Date\_ 20160711  
 Time 0.39  
 INSTRUM spect  
 PROBHD 5 mm CPMBO BB  
 PULPROG deptsp135  
 TD 65536  
 SOLVENT CDCl3  
 NS 300  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 130.26  
 DW 20.800 usec  
 DE 18.00 usec  
 TE 297.0 K  
 CNST2 145.0000000  
 D1 2.00000000 sec  
 D2 0.00344828 sec  
 D12 0.00002000 sec  
 TD0 1

===== CHANNEL f1 =====  
 SF01 100.6233324 MHz  
 NUC1 13C  
 P1 10.00 usec  
 P13 2000.00 usec  
 SI 32768  
 SF 100.6127689 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

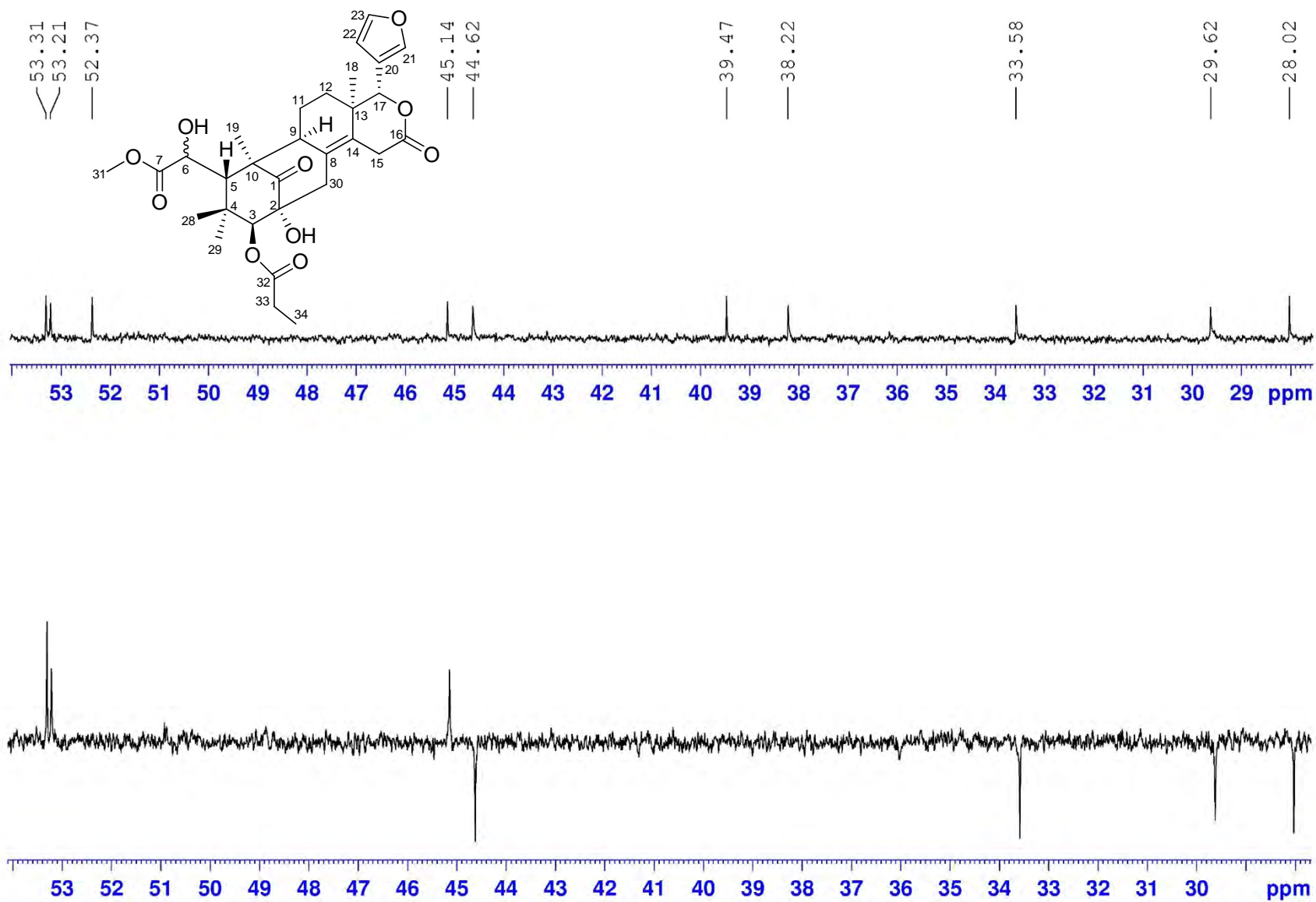
# DEPT135 (100 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **2** in  $\text{CDCl}_3$

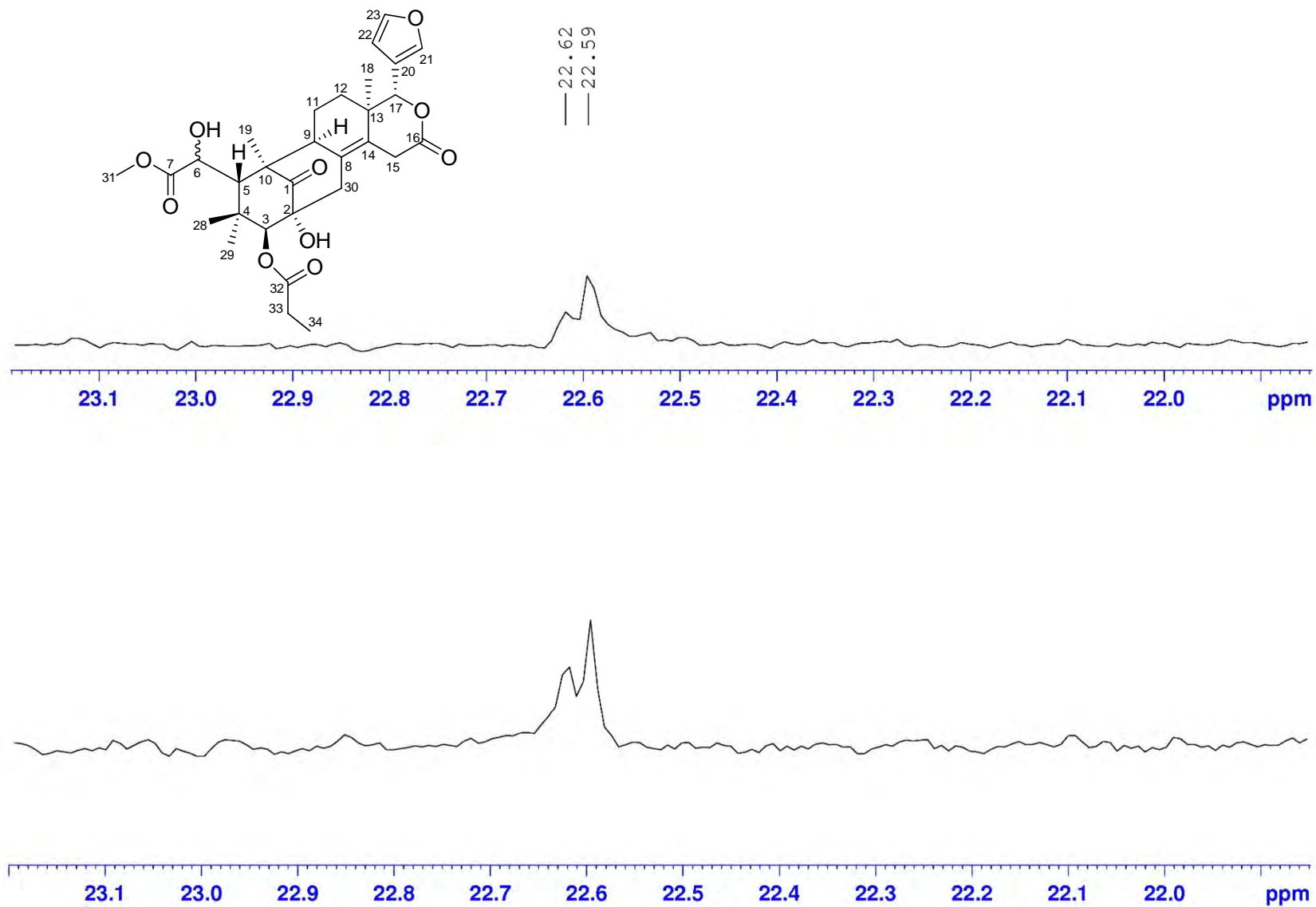


# DEPT135 (100 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



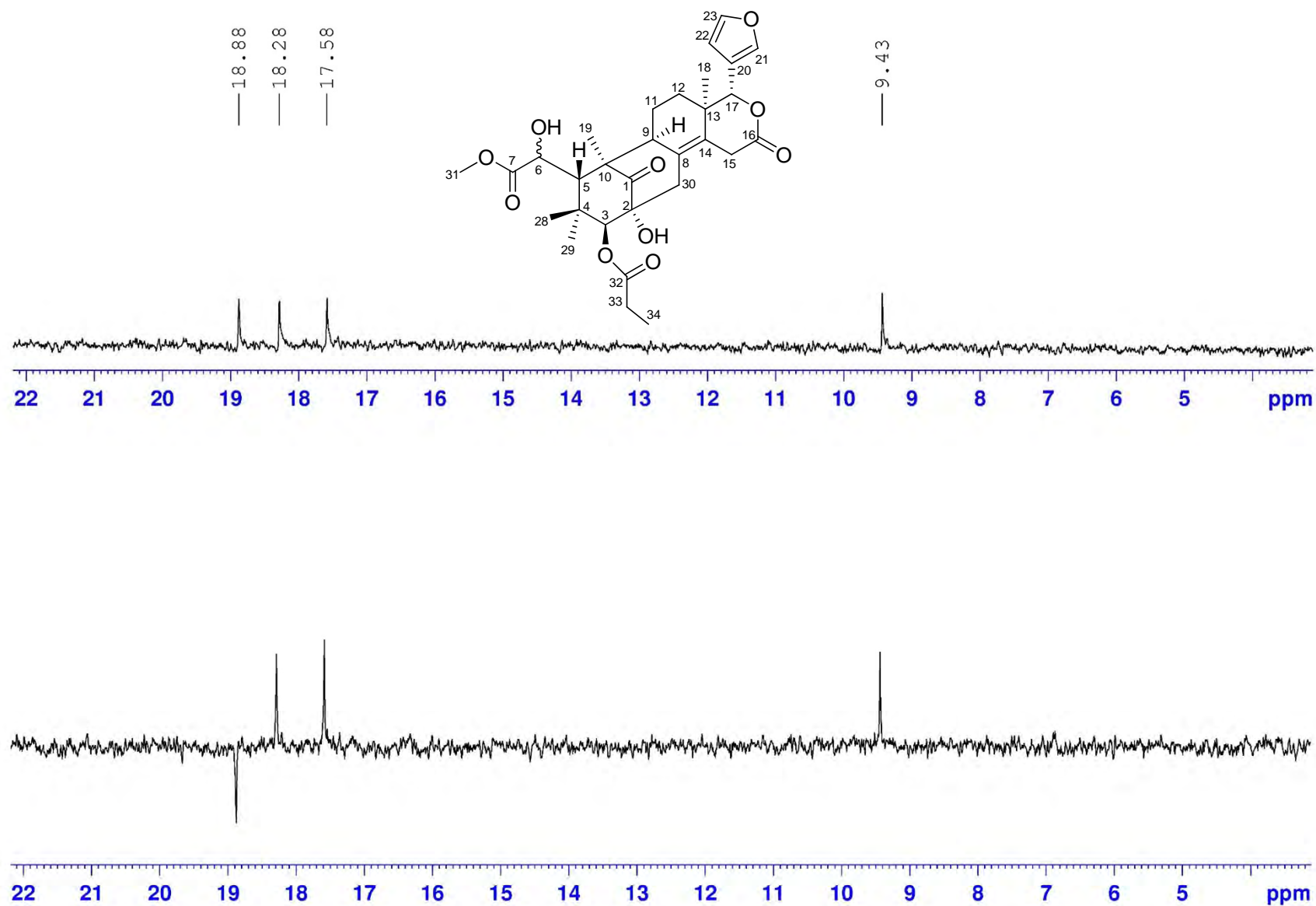


DEPT135 (100 MHz) spectrum of compound **2** in CDCl<sub>3</sub>

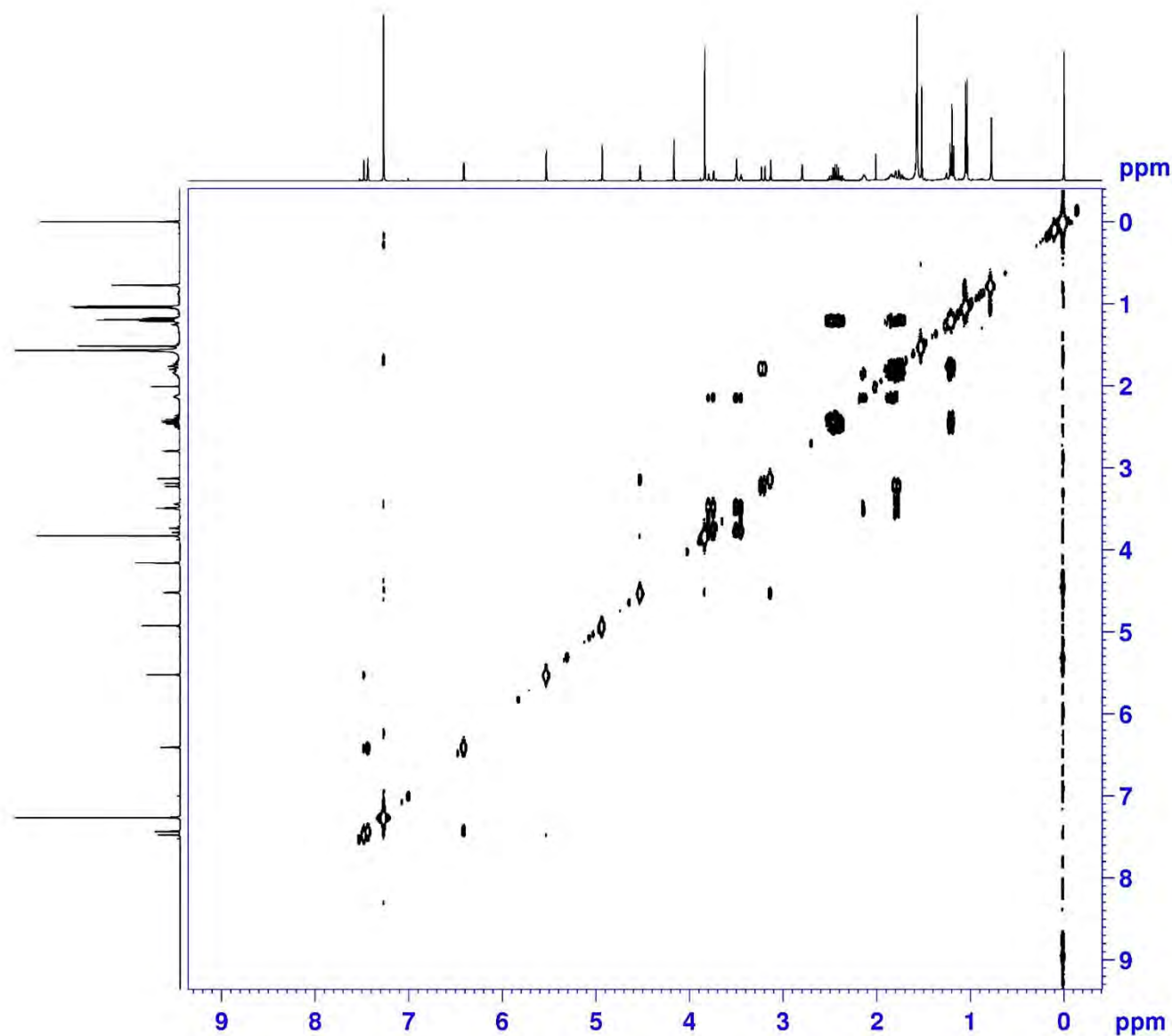




DEPT135 (100 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



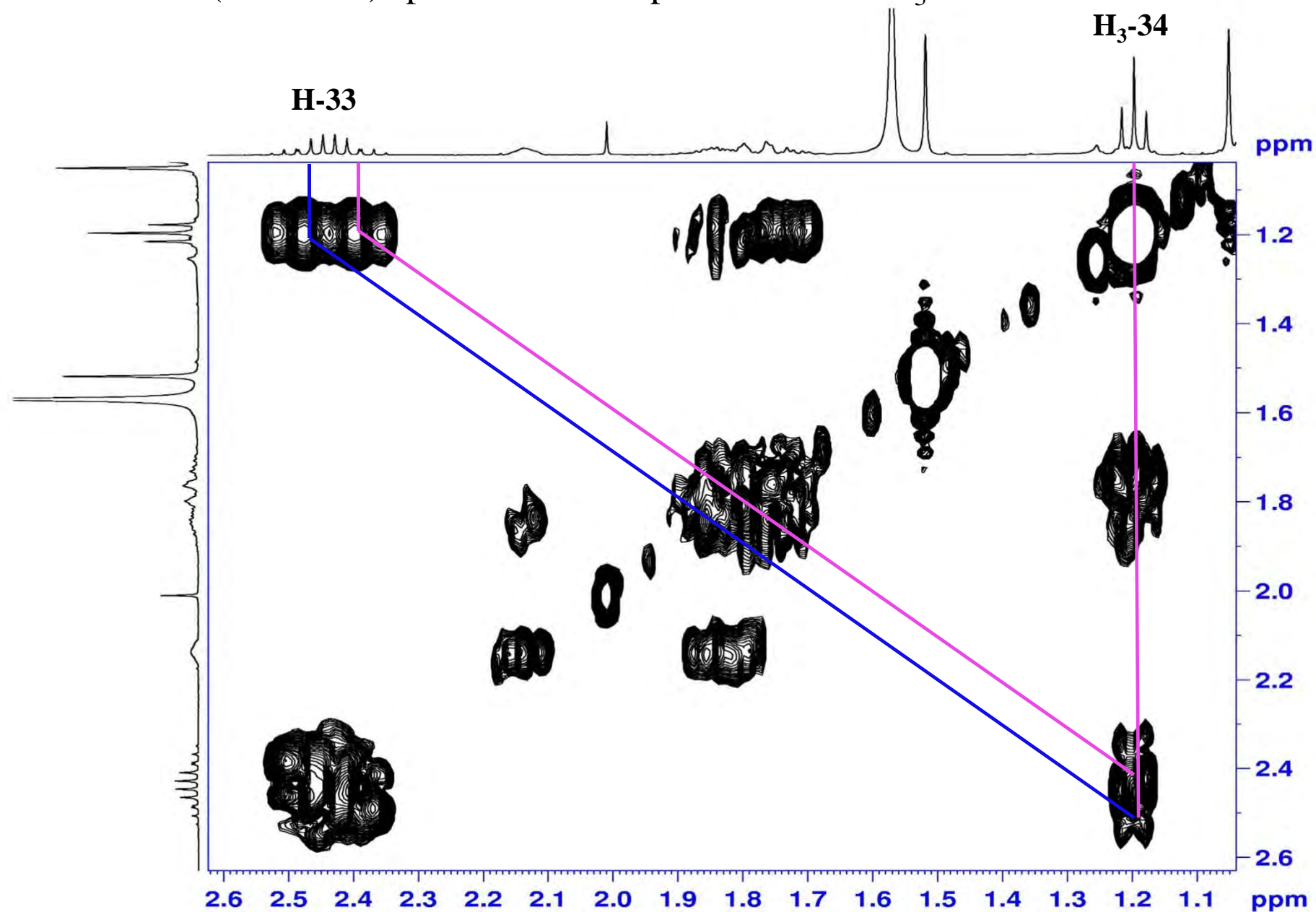
# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **2** in $\text{CDCl}_3$



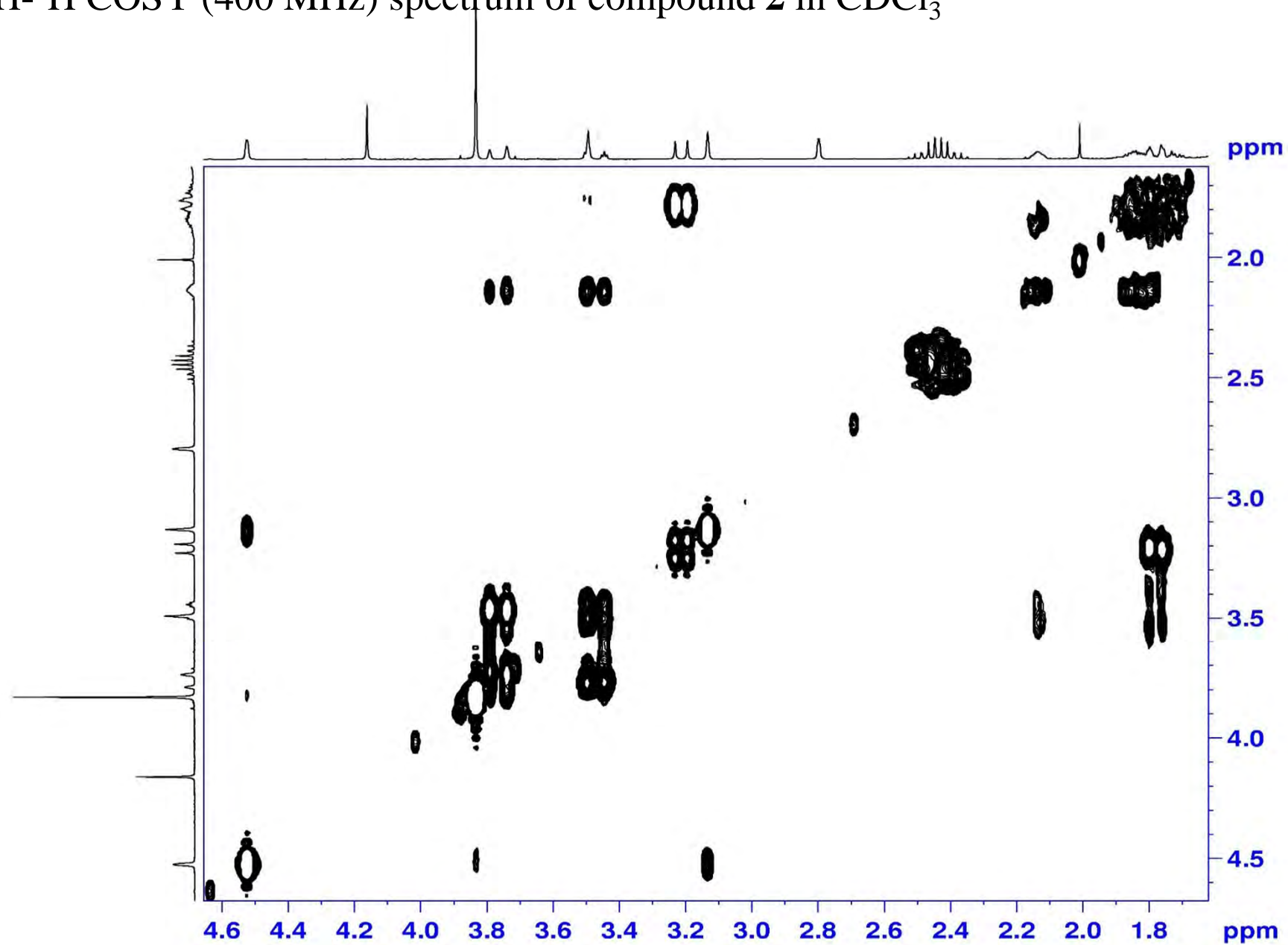
NAME zjz-26=RT7sp  
EXPNO 4  
PROCNO 1  
Date\_ 20160806  
Time 15.53  
INSTRUM spect  
PROBHD 5 mm CFPBBO BB  
PULPROG cosygpppqf  
TD 2048  
SOLVENT  $\text{CDCl}_3$   
NS 32  
DS 8  
SWH 3906.250 Hz  
FIDRES 1.907349 Hz  
AQ 0.2621940 sec  
RG 208.5  
DW 128.000 usec  
DE 10.00 usec  
TE 297.0 K  
D0 0.00000300 sec  
D1 1.89678097 sec  
D11 0.03000000 sec  
D12 0.00002000 sec  
D13 0.00000400 sec  
D16 0.00020000 sec  
IN0 0.00025600 sec

===== CHANNEL f1 =====  
SFO1 400.1318006 MHz  
NUC1  $^1\text{H}$   
P0 11.50 usec  
P1 11.50 usec  
P17 2500.00 usec  
ND0 1  
TD 128  
SFO1 400.1318 MHz  
FIDRES 30.517578 Hz  
SW 9.762 ppm  
FMODE QF  
SI 1024  
SF 400.1300076 MHz  
WDW QSINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40  
SI 1024  
MC2 QF  
SF 400.1300067 MHz  
WDW QSINE  
SSB 0  
LB 0.00 Hz  
GB 0

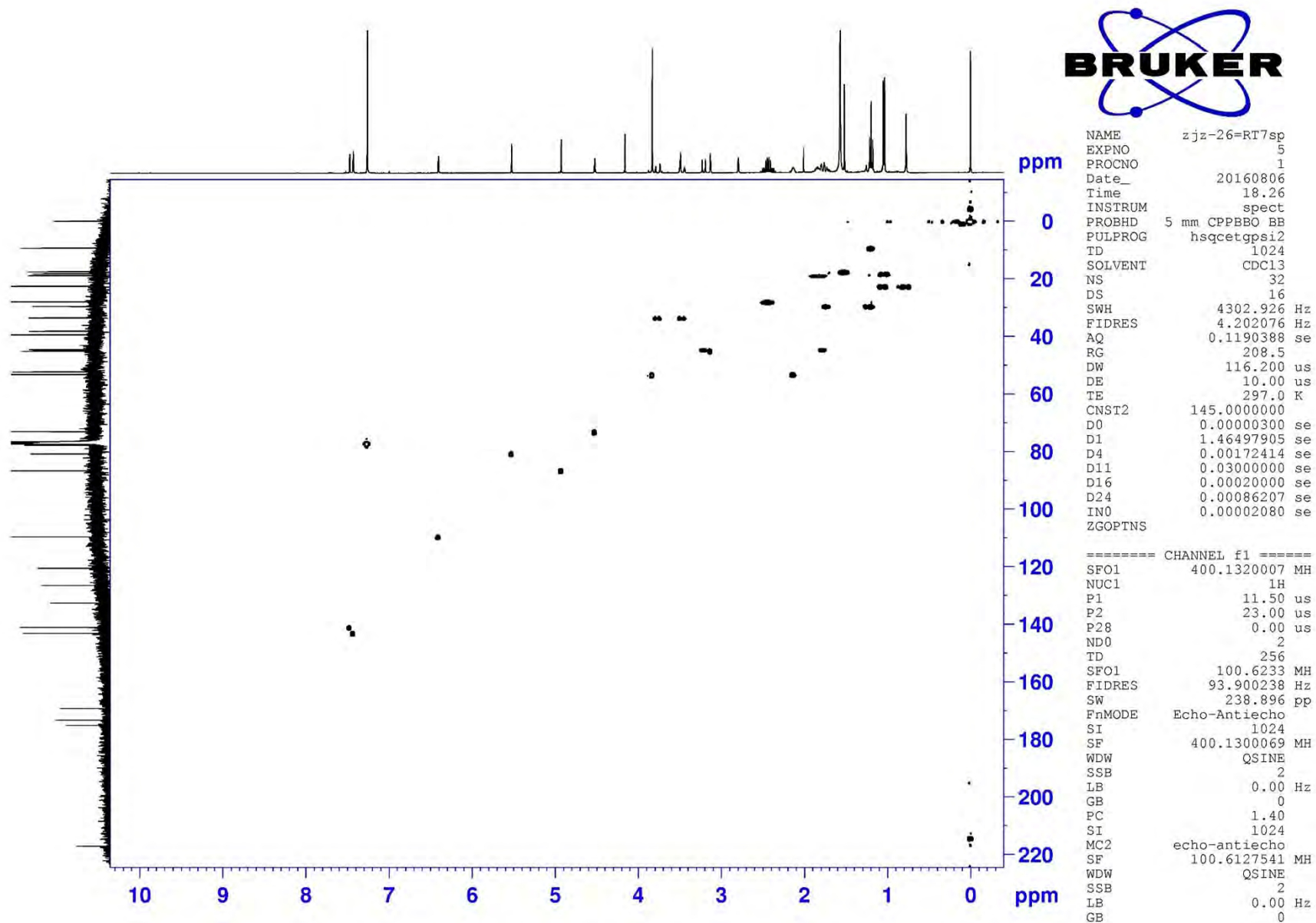
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$

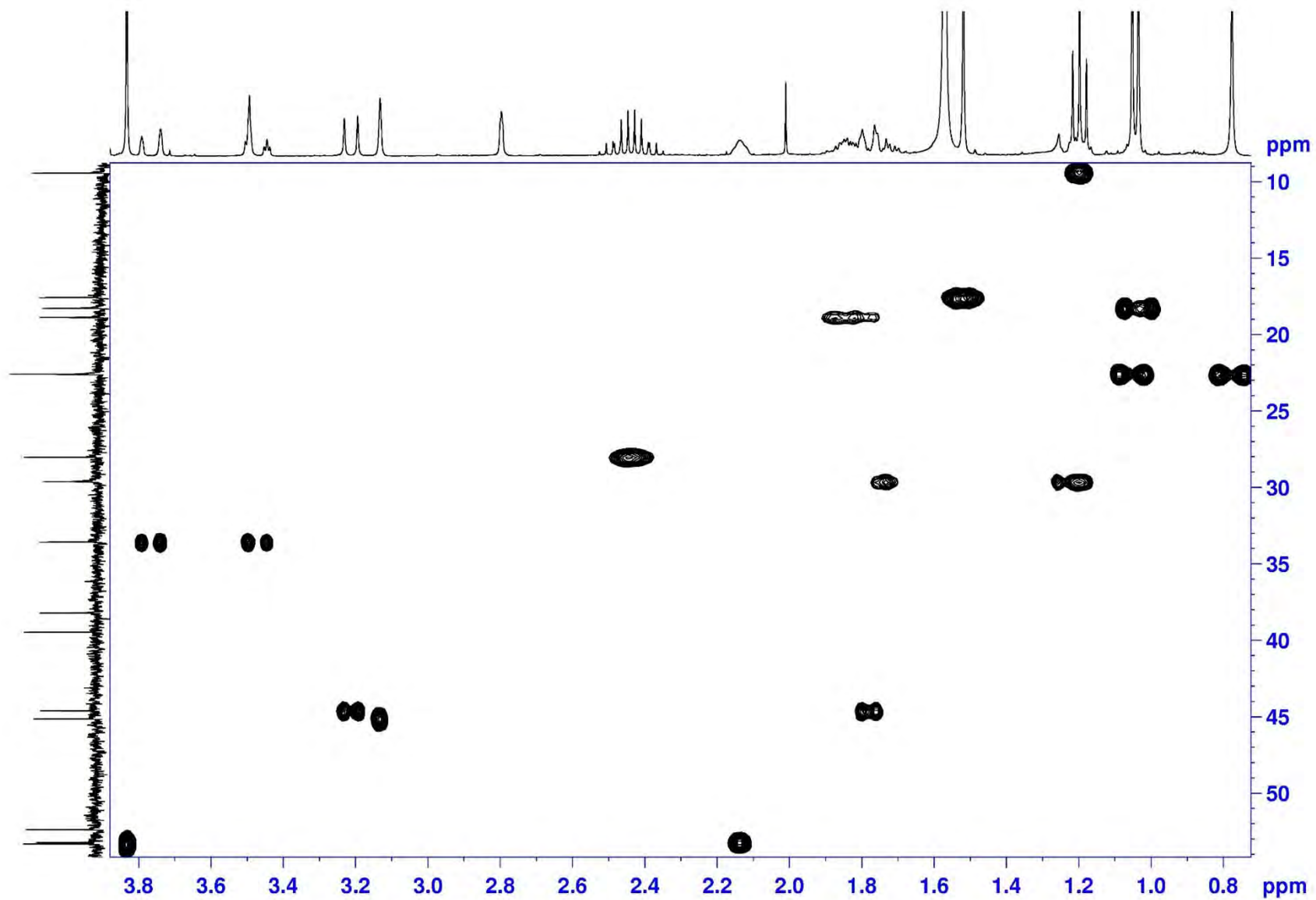


# HSQC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>

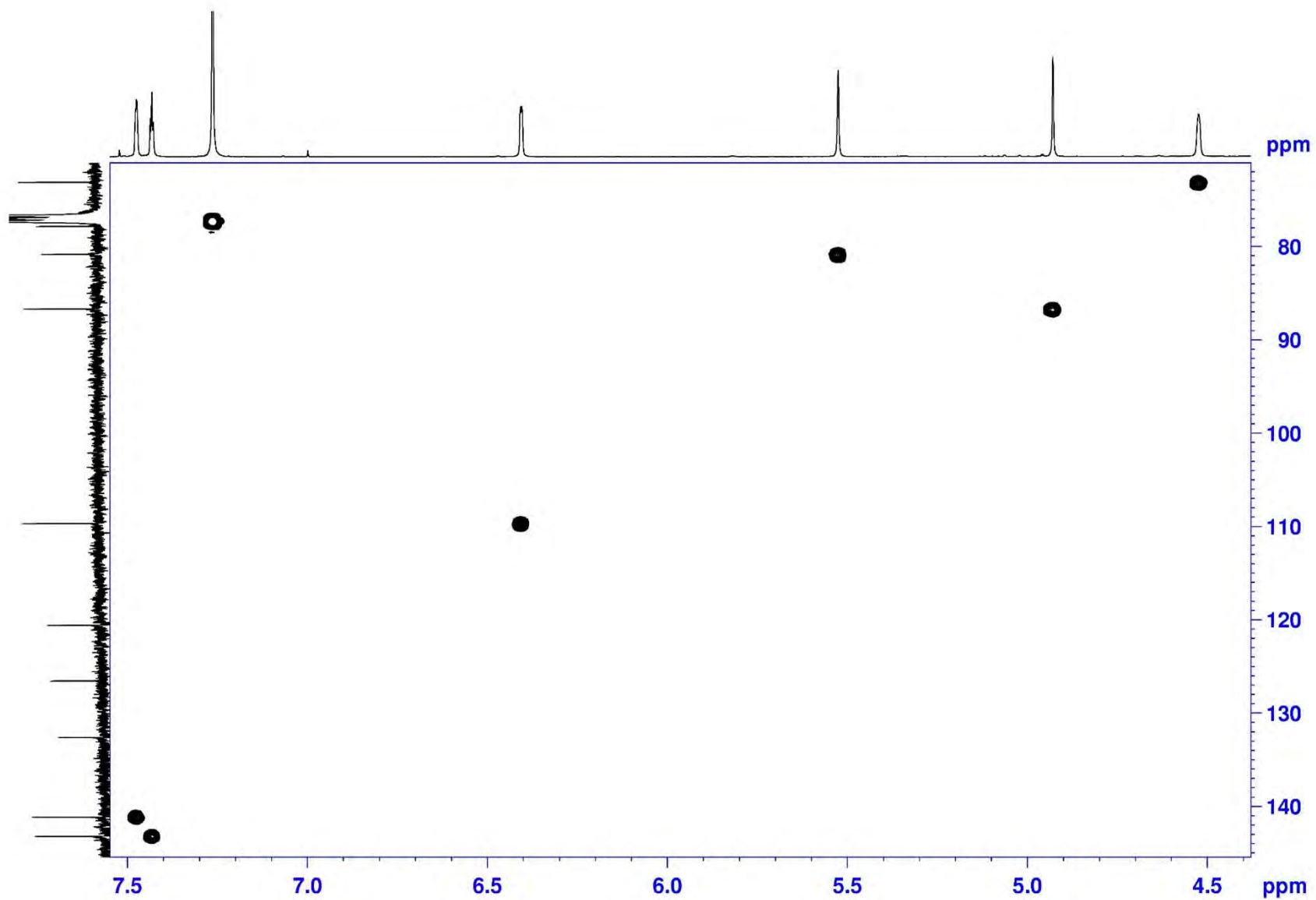




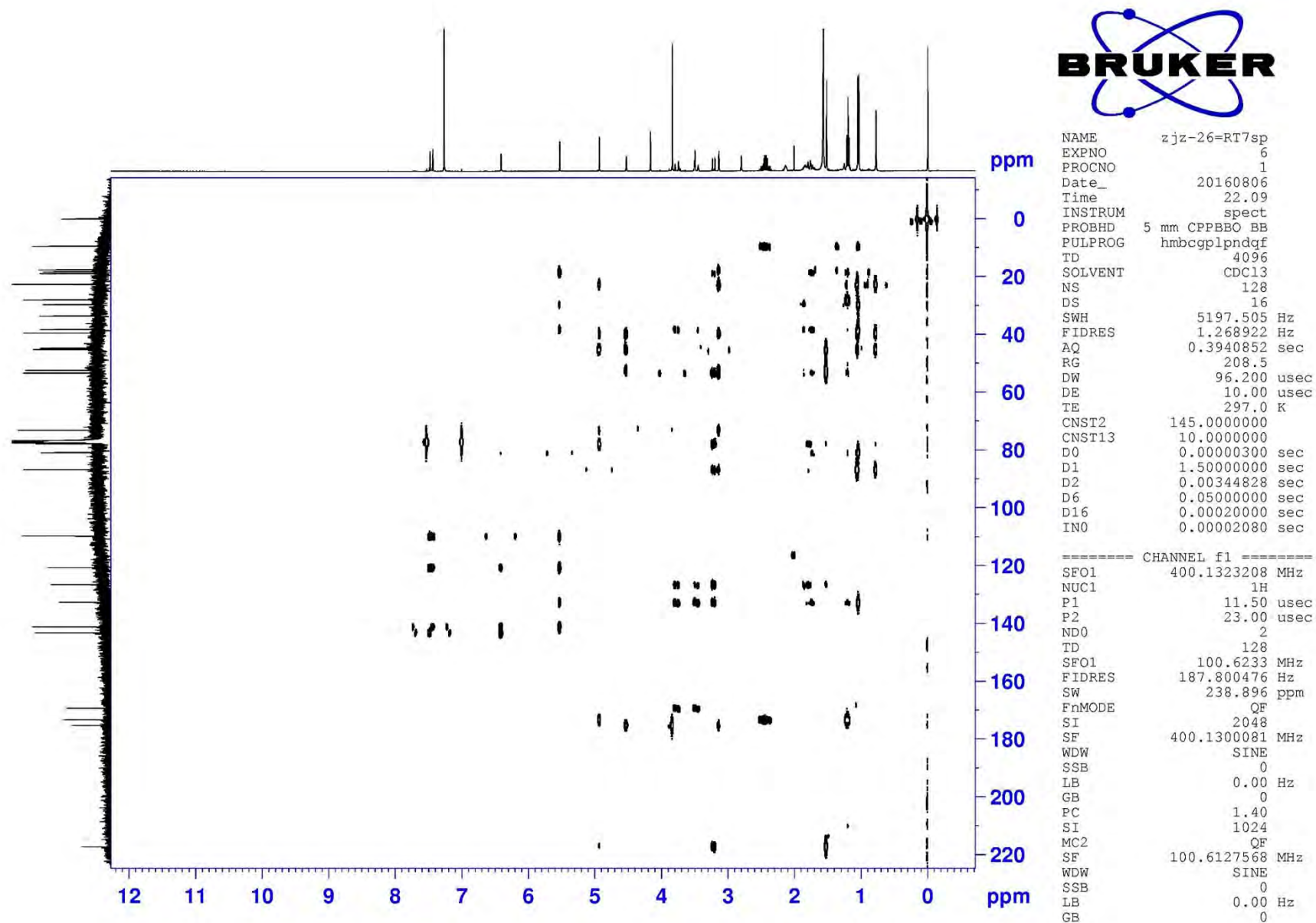
HSQC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



HSQC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>

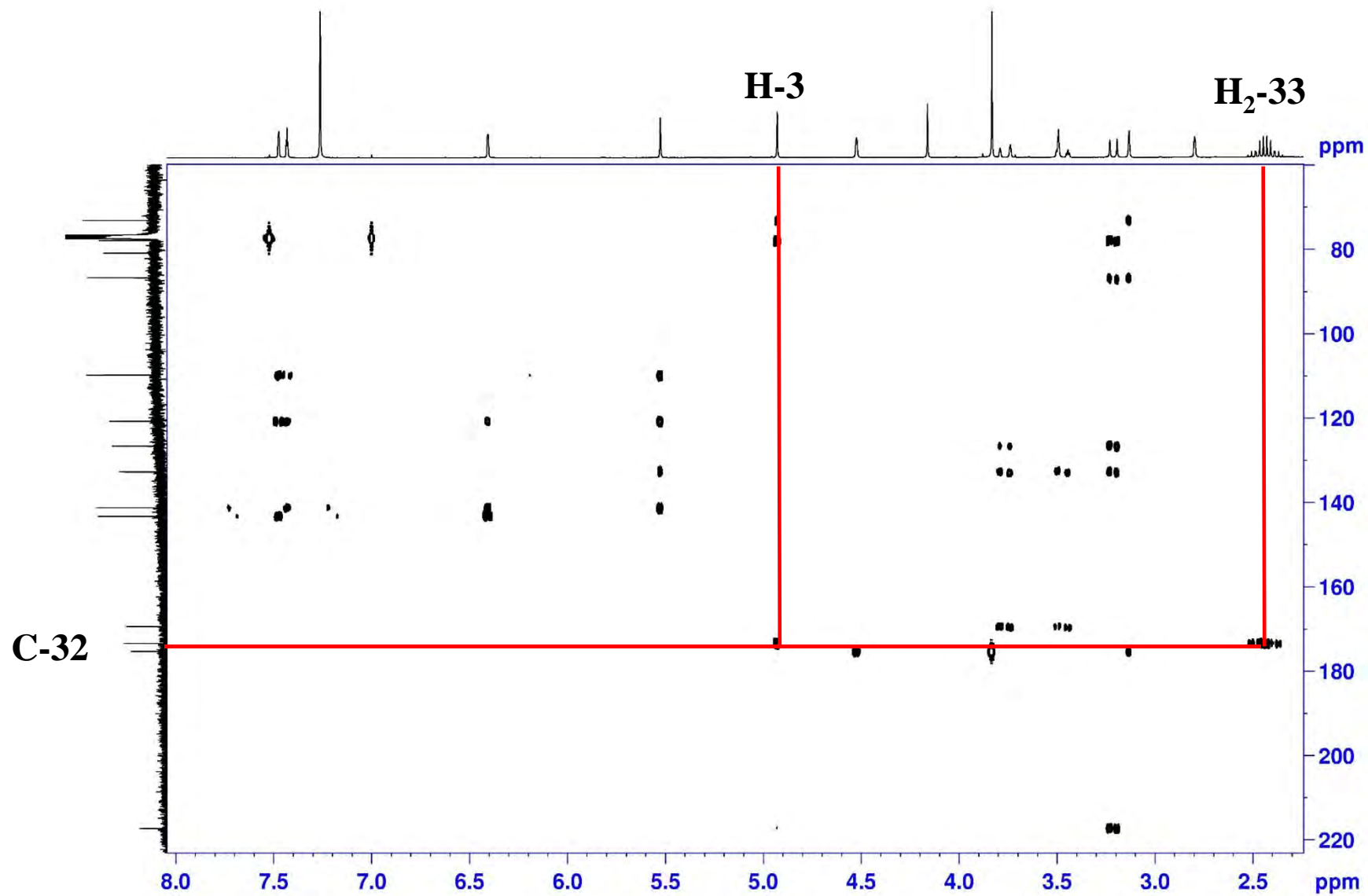


# HMBC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>

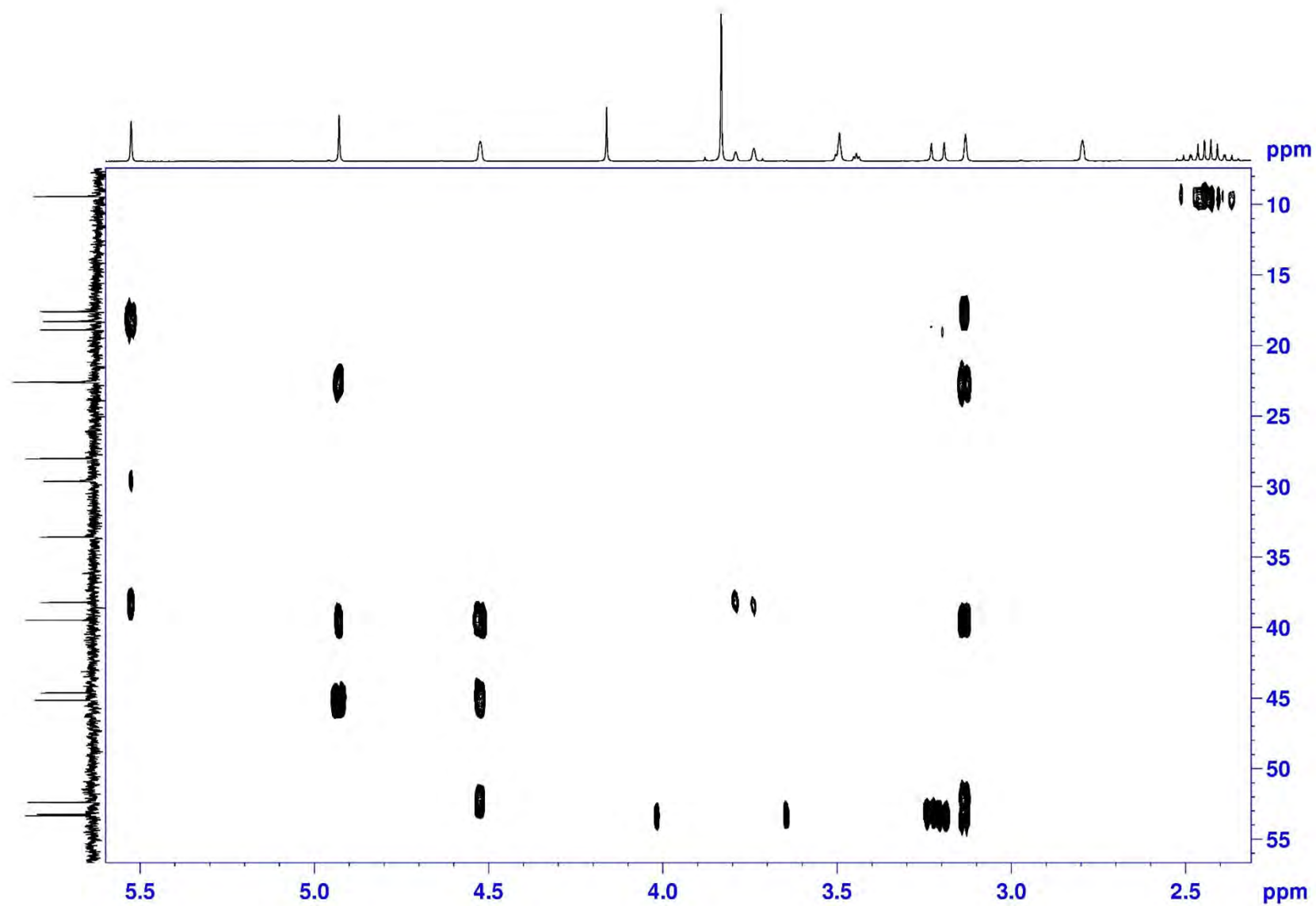




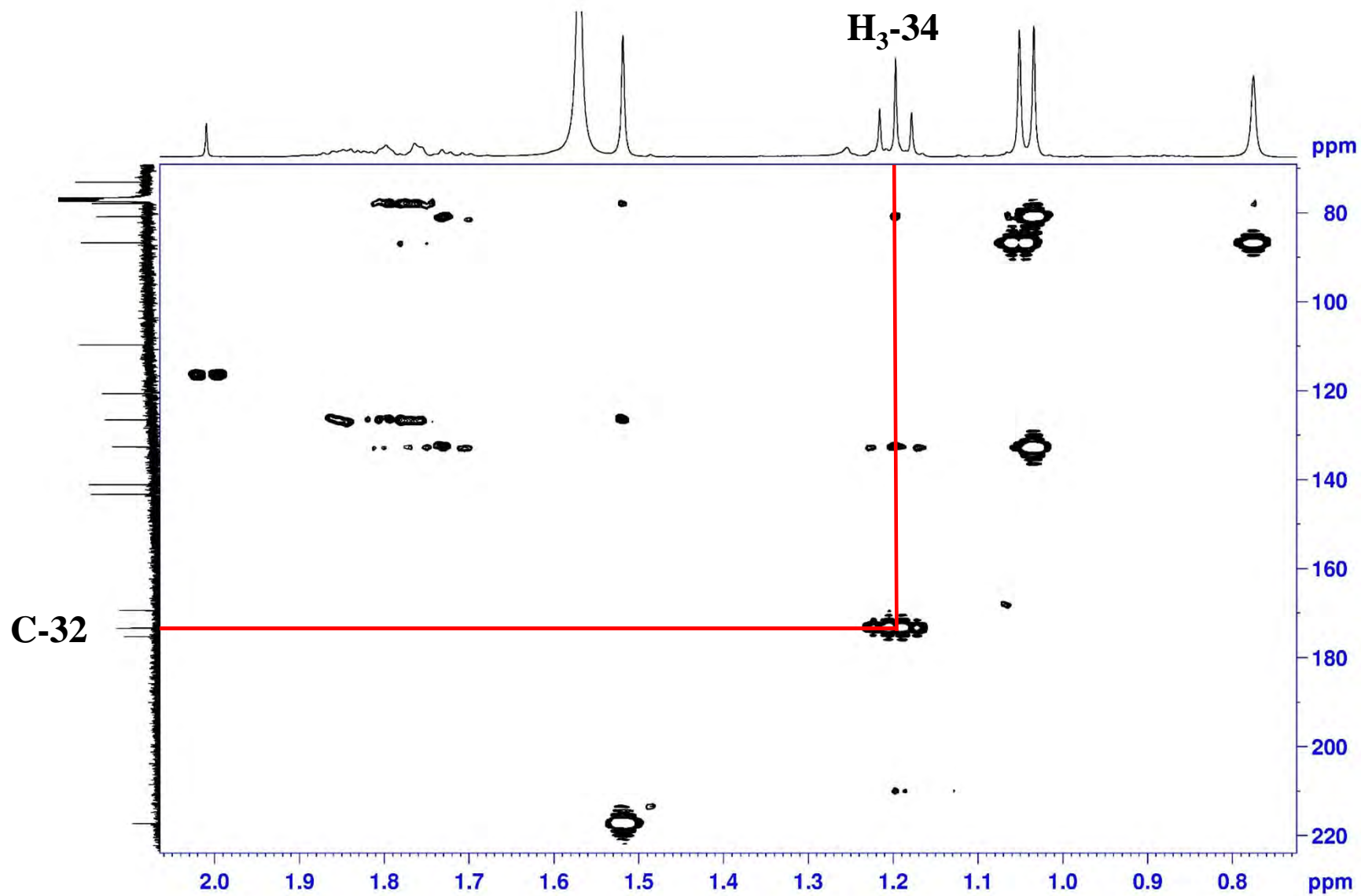
HMBC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



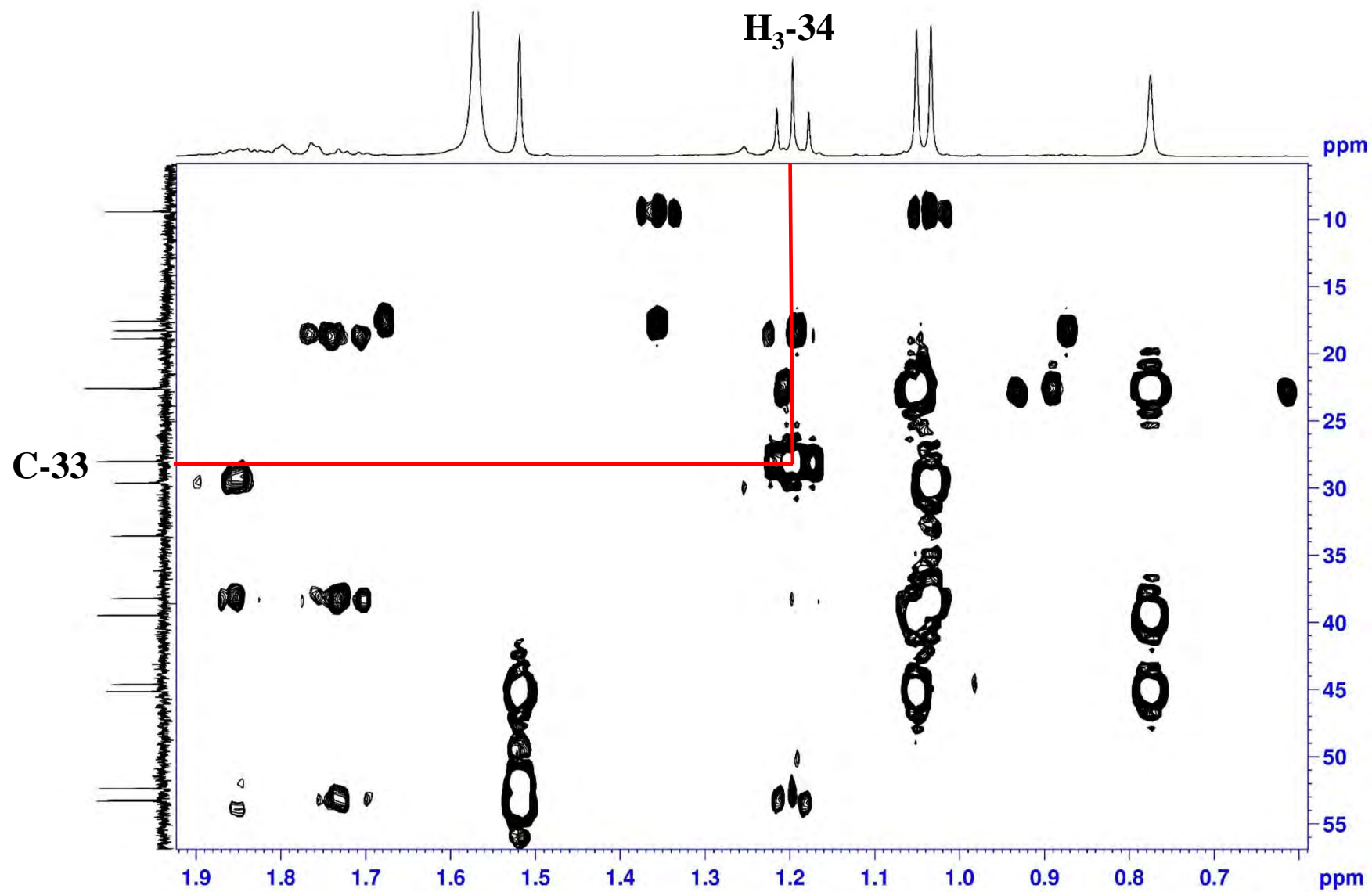
HMBC (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



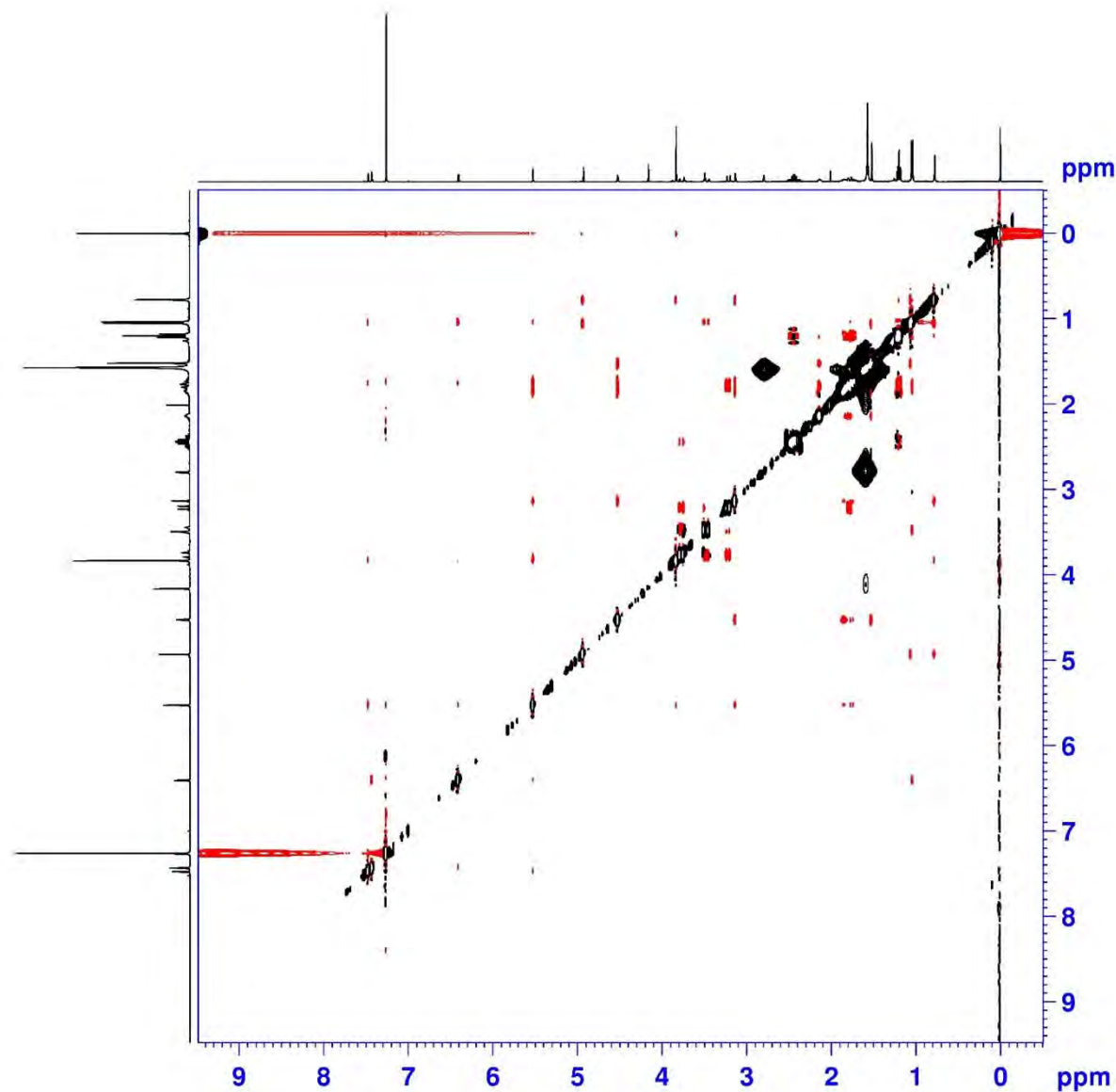
HMBC (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>



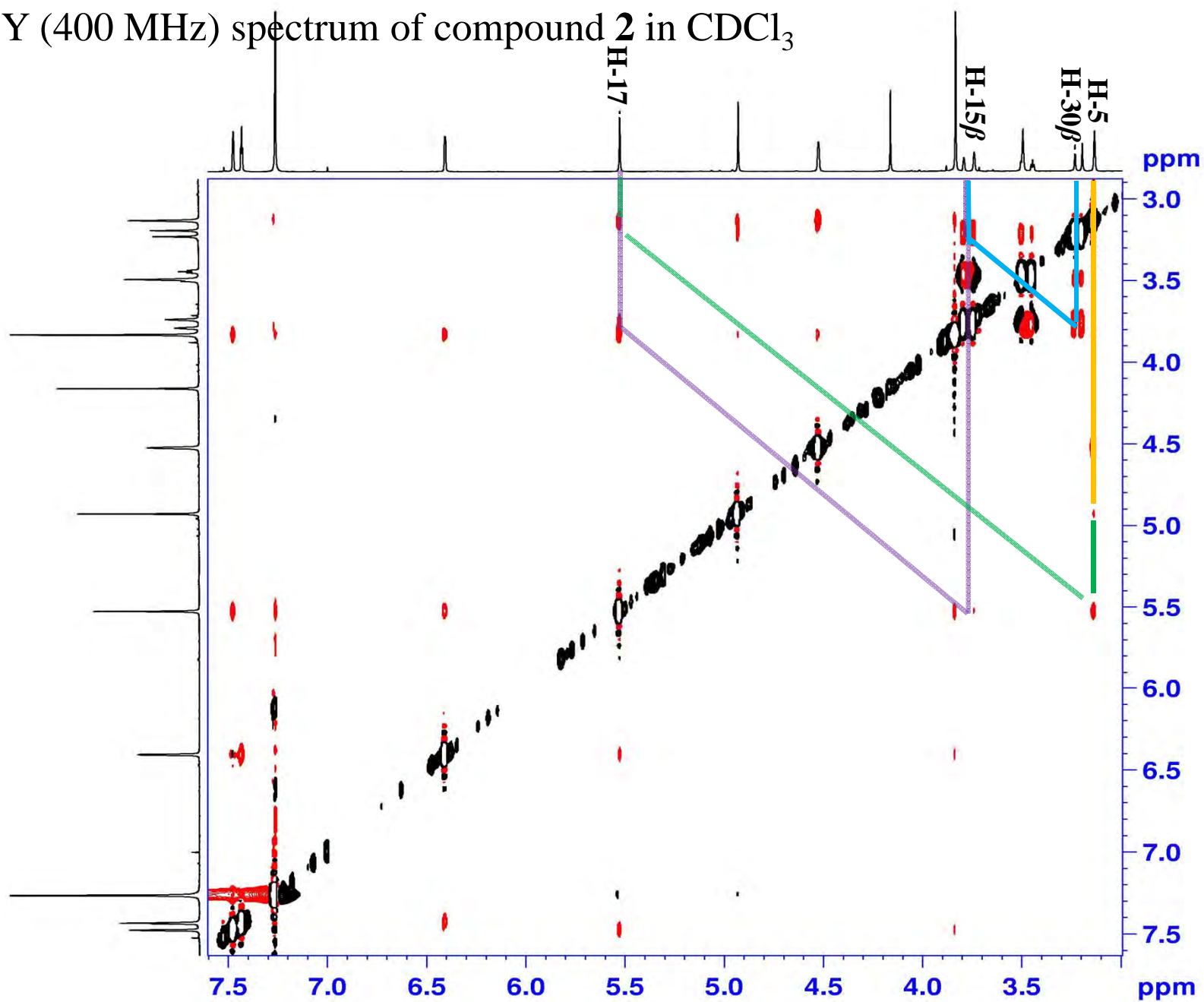
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EXPNO     7
PROCNO    1
Date_     20160807
Time      7.04
INSTRUM   spect
PROBHD    5 mm CFPBBO BB
PULPROG   noesygpphpp
TD         2048
SOLVENT   CDCl3
NS         64
DS         32
SWH        4000.000 Hz
FIDRES     1.953125 Hz
AQ         0.2560500 sec
RG         208.5
DW         125.000 usec
DE         10.00 usec
TE         297.0 K
D0         0.00011036 sec
D1         1.99385595 sec
D8         0.30000001 sec
D11        0.03000000 sec
D12        0.00002000 sec
D16        0.00020000 sec
IN0        0.00025000 sec
  
```

```

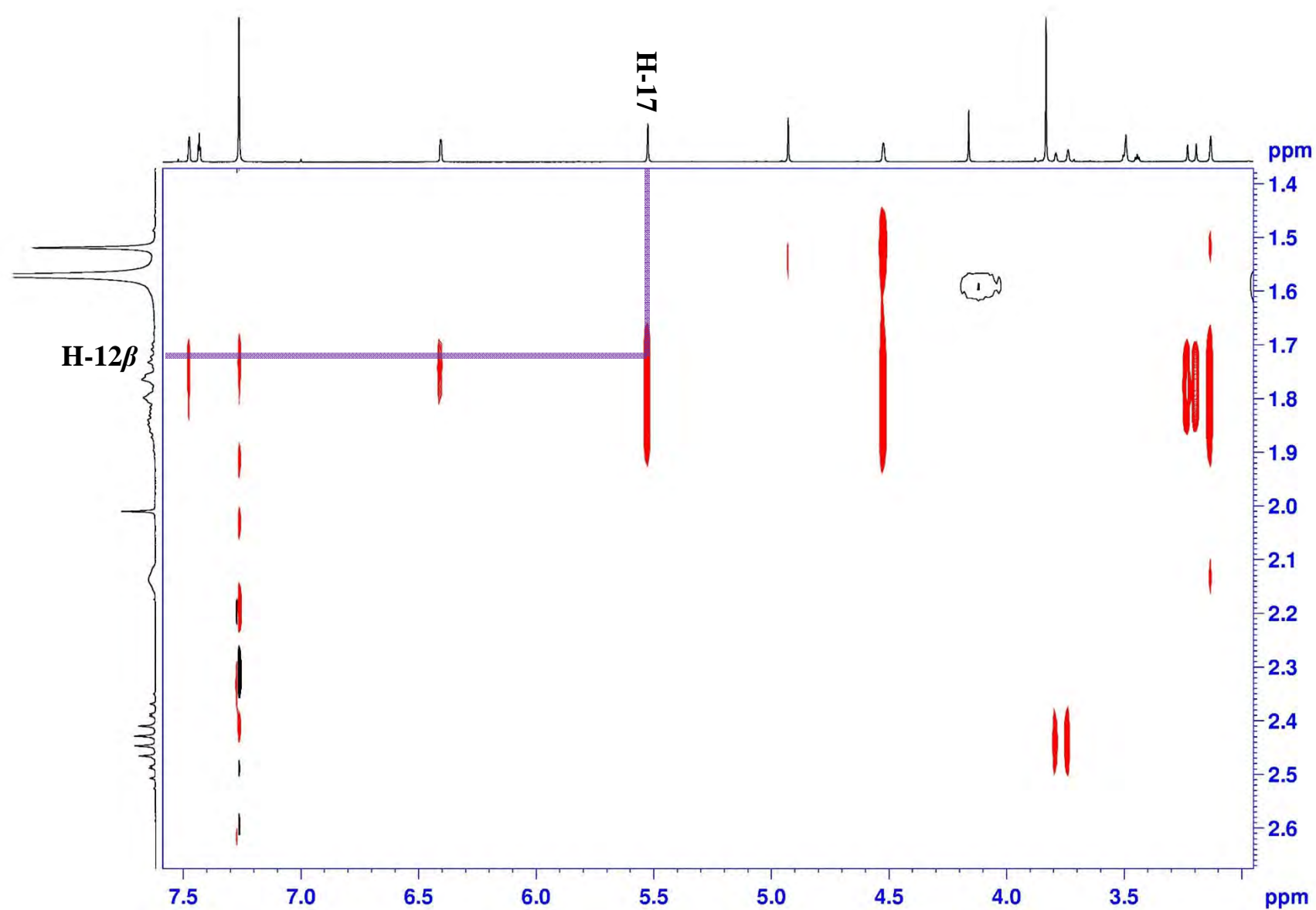
===== CHANNEL f1 =====
SFO1      400.1318006 MHz
NUC1       1H
P1         11.50 usec
P2         23.00 usec
P17        2500.00 usec
ND0         1
TD         256
SFO1      400.1318 MHz
FIDRES     15.625000 Hz
SW          9.997 ppm
FnMODE     States-TPPI
SI         1024
SF         400.1300064 MHz
WDW        QSINE
SSB         2
LB          0.00 Hz
GB          0
PC          1.00
SI         1024
MC2        States-TPPI
SF         400.1300079 MHz
WDW        QSINE
SSB         2
LB          0.00 Hz
GB          0
  
```

NOESY (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$

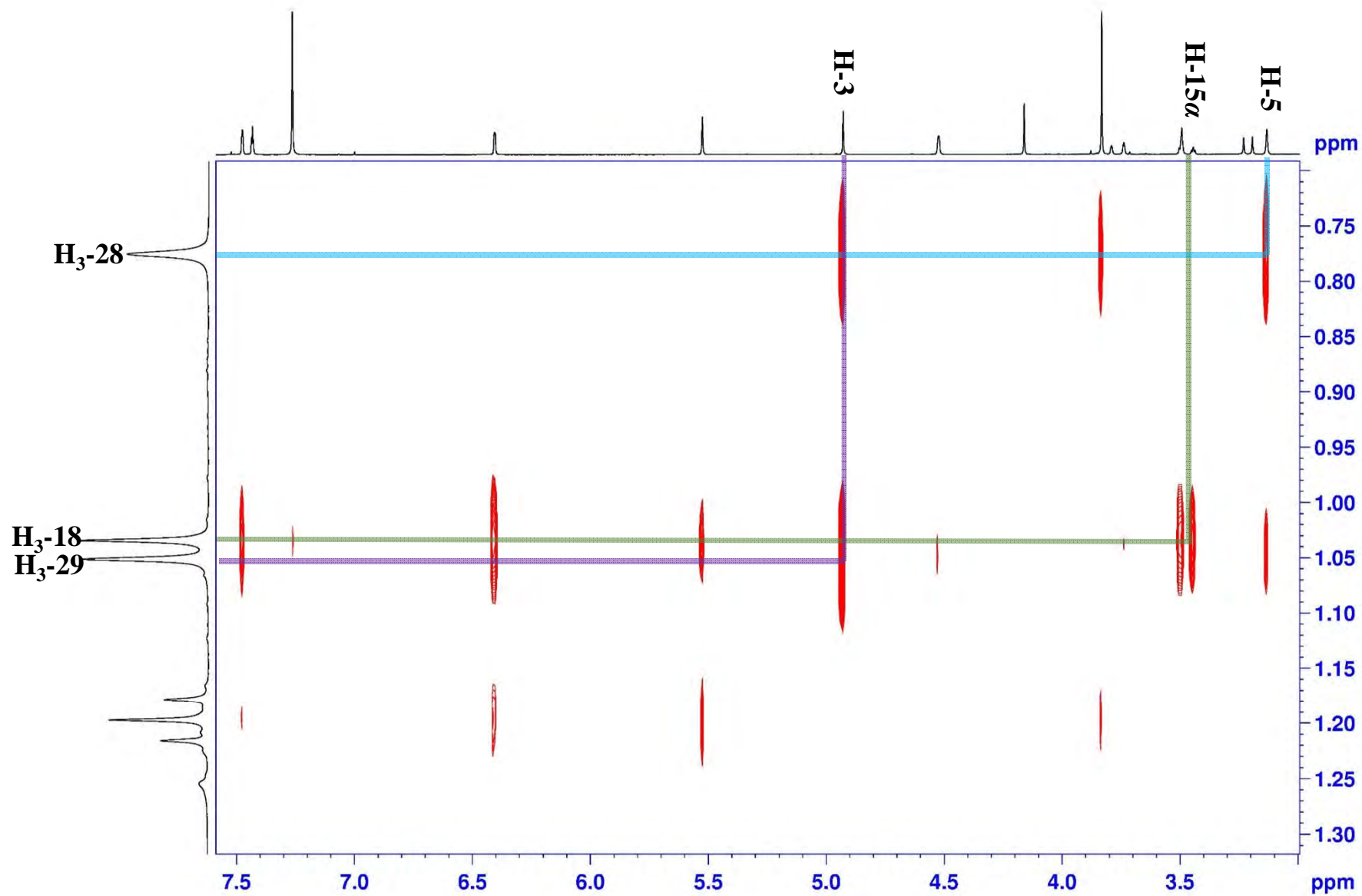




NOESY (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$

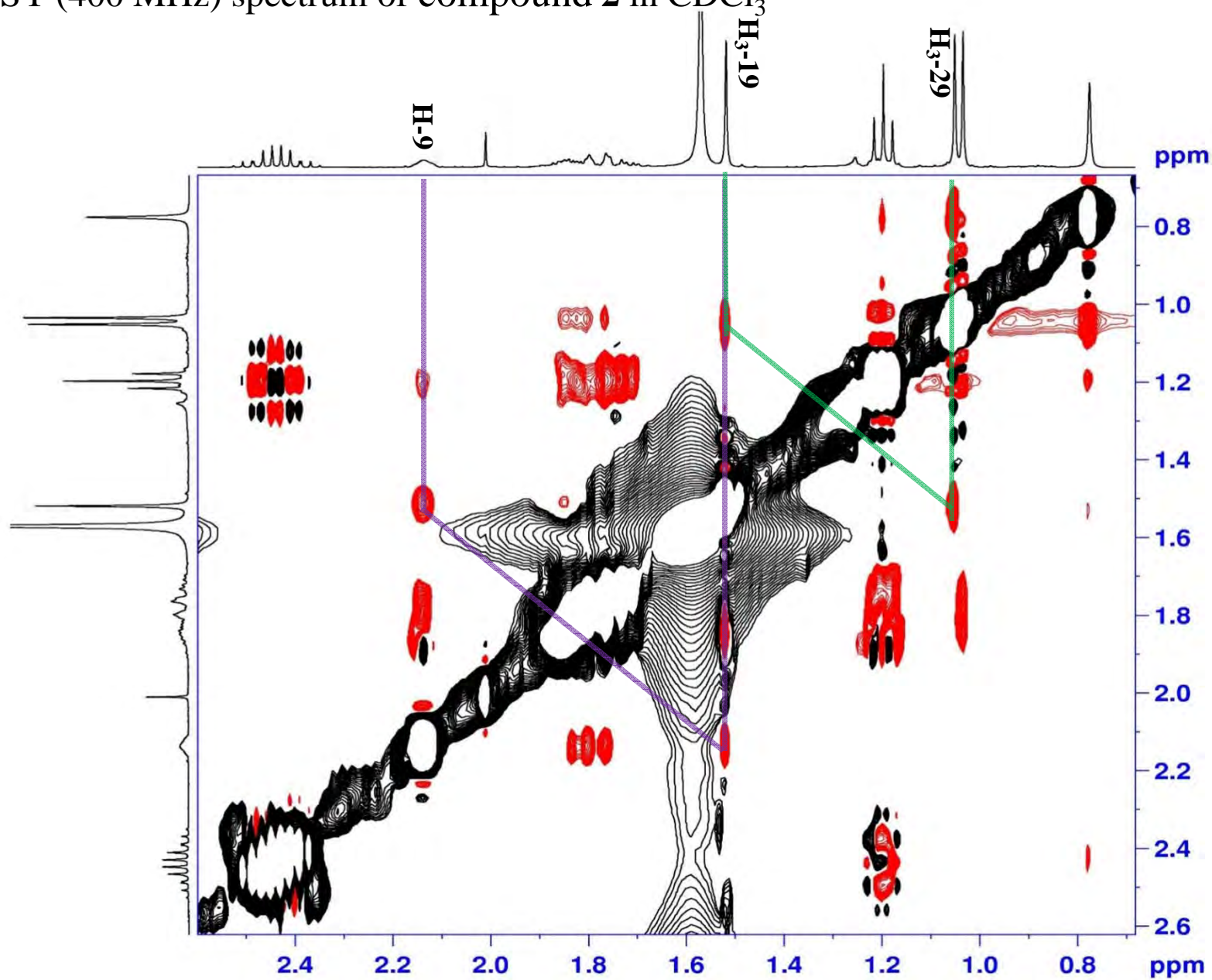


NOESY (400 MHz) spectrum of compound **2** in CDCl<sub>3</sub>





NOESY (400 MHz) spectrum of compound **2** in  $\text{CDCl}_3$



# HR-ESIMS for compound 3

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-63-1\_pos\_14\_01\_1861.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-63-1\_pos  
 Comment

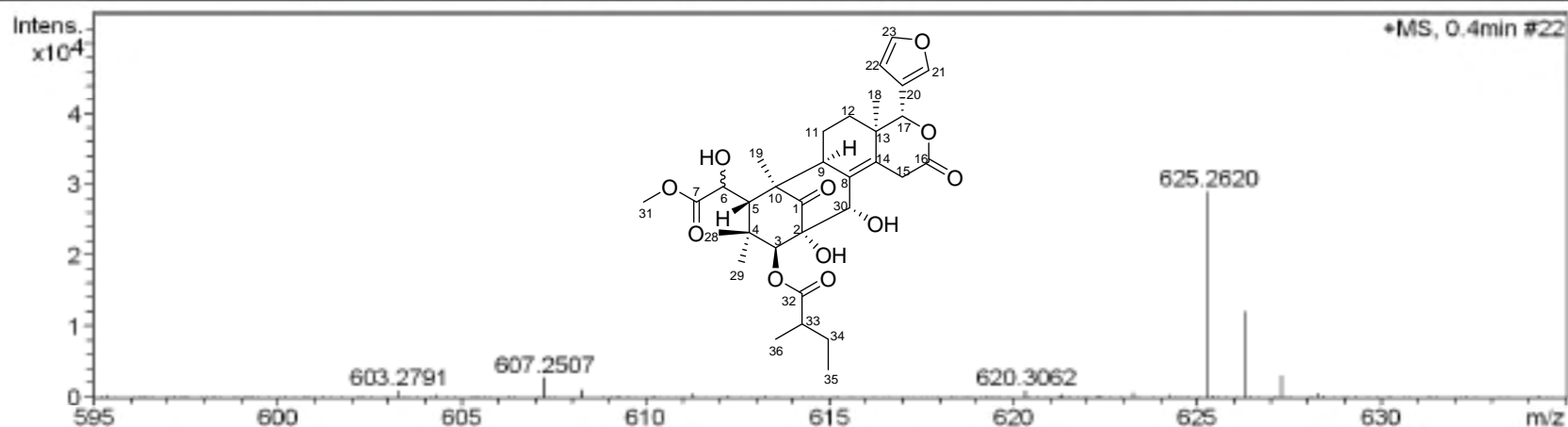
Acquisition Date 5/19/2016 9:01:46 AM  
 Operator SCSIO  
 Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type ESI  
 Focus Active  
 Scan Begin 100 m/z  
 Scan End 2000 m/z

Ion Polarity Positive  
 Set Capillary 4500 V  
 Set End Plate Offset -500 V  
 Set Collision Cell RF 800.0 Vpp

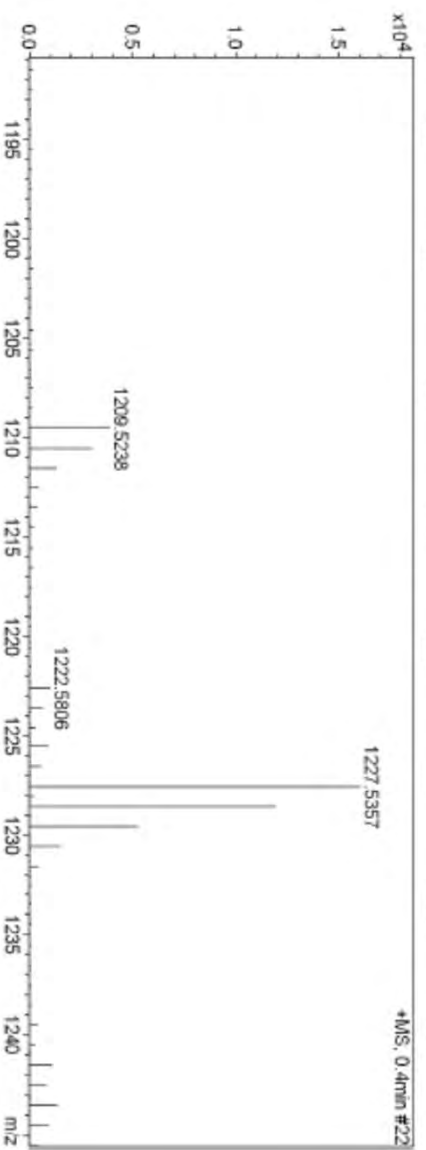
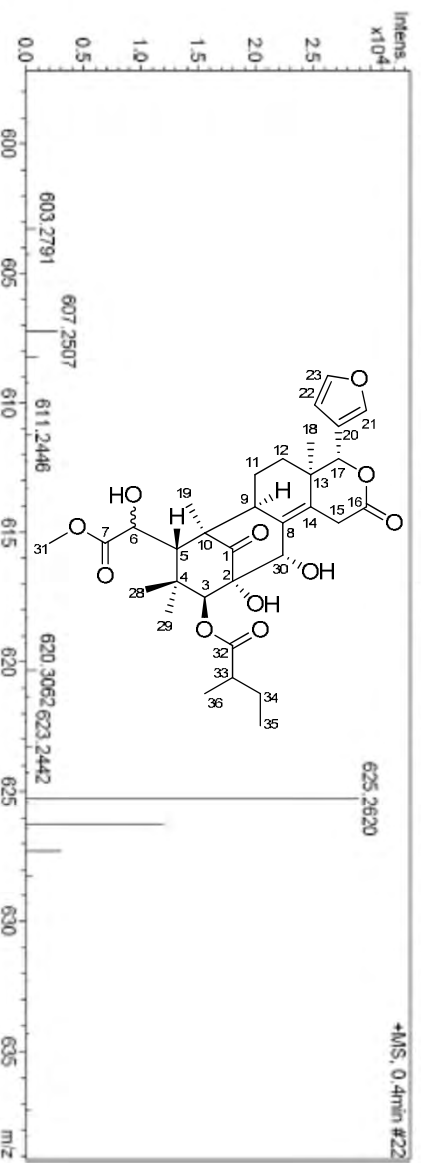
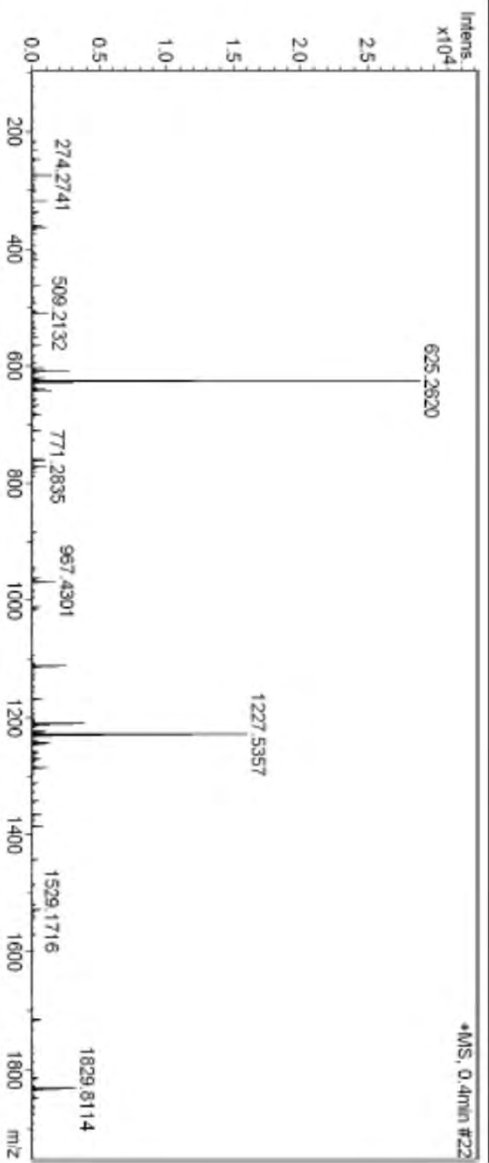
Set Nebulizer 0.4 Bar  
 Set Dry Heater 180 °C  
 Set Dry Gas 4.0 l/min  
 Set Divert Valve Waste



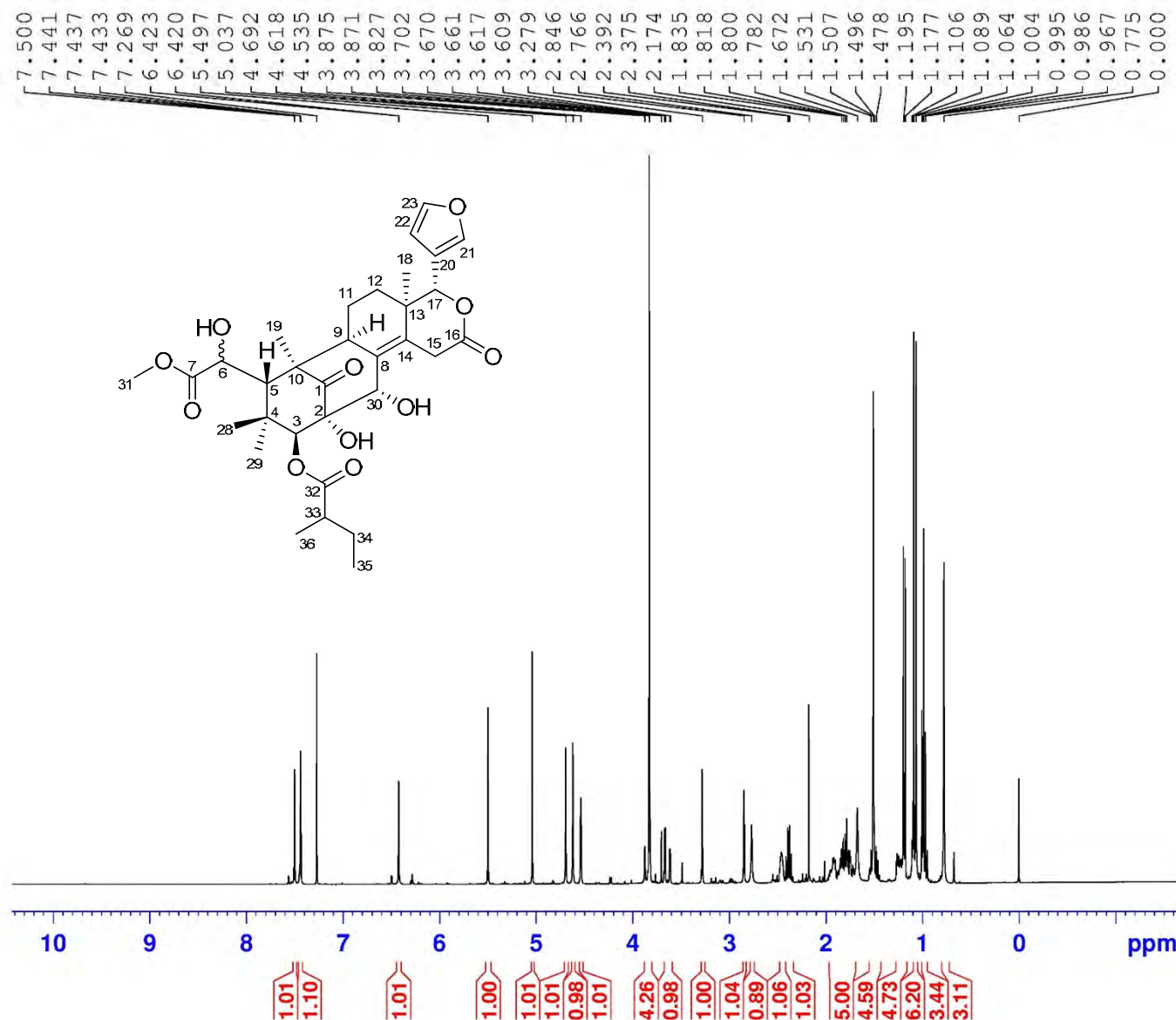
Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e <sup>-</sup> Conf	N-Rule
603.2791	1	C 32 H 43 O 11	100.00	603.2800	1.4	0.9	47.9	11.5	even	ok
625.2620	1	C 32 H 42 Na O 11	100.00	625.2619	-0.1	-0.1	31.7	11.5	even	ok
1227.5357	1	C 64 H 84 Na O 22	90.63	1227.5346	-0.9	-1.1	20.7	22.5	even	ok

## Generic Display Report

<b>Analysis Info</b>		Acquisition Date    5/19/2018 9:01:48 AM	
Analysis Name	D:\Data\MS\data\201805\liwanshan_lws-63-1_pos_14_01_1881.d	Operator	SCSIO
Method	LC_Direct Infusion_pos_100-1000mz.m	Instrument	maxis
Sample Name	liwanshan_lws-63-1_pos		
Comment			



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



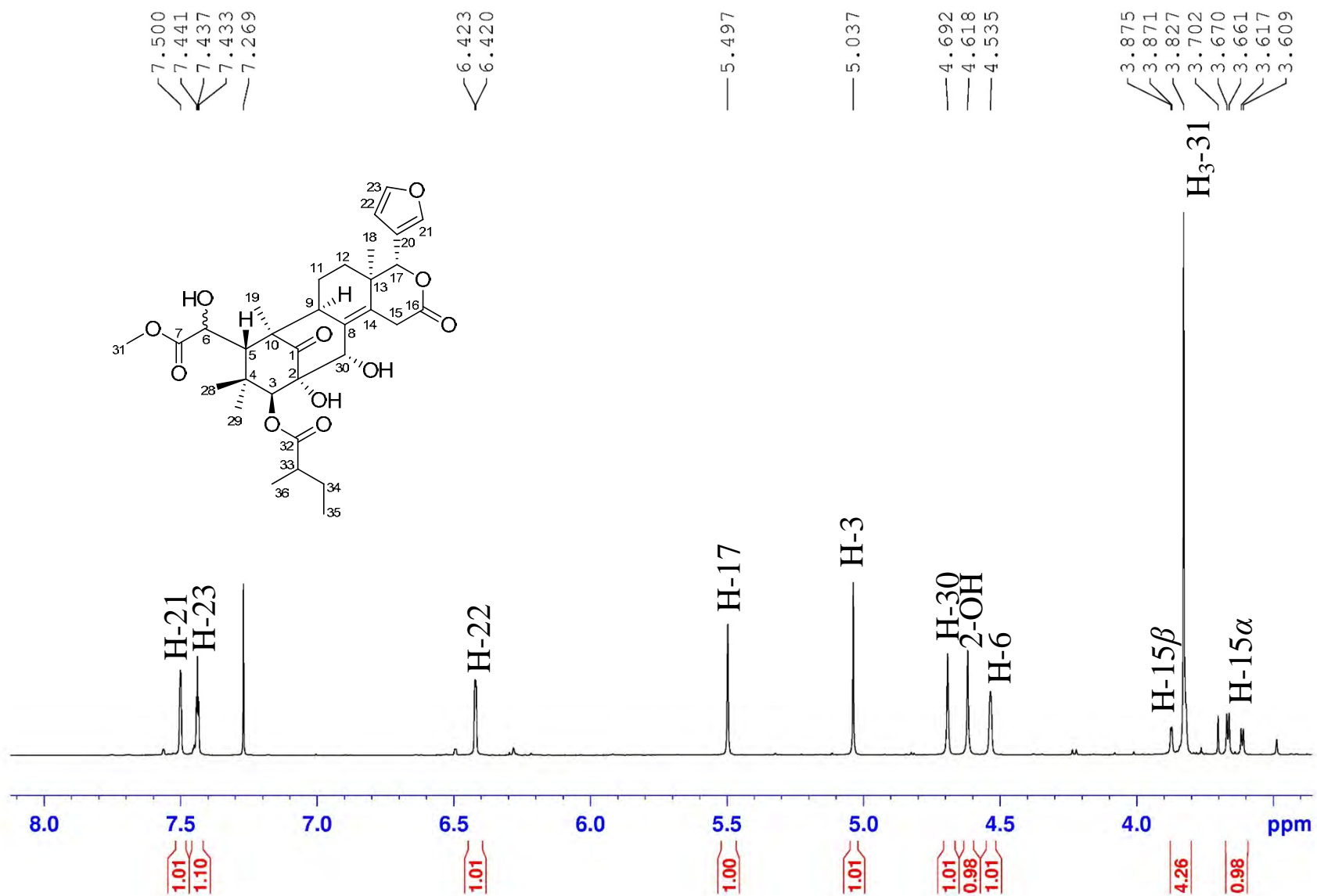
```

NAME      lws-63-1
EXPNO     1
PROCNO    1
Date_     20150708
Time      6.30
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 F
FIDRES    0.125483 F
AQ        3.9846387 s
RG        102.3
DW        60.800 u
DE        10.00 u
TE        297.0 K
D1        1.00000000 s
TD0       1
    
```

```

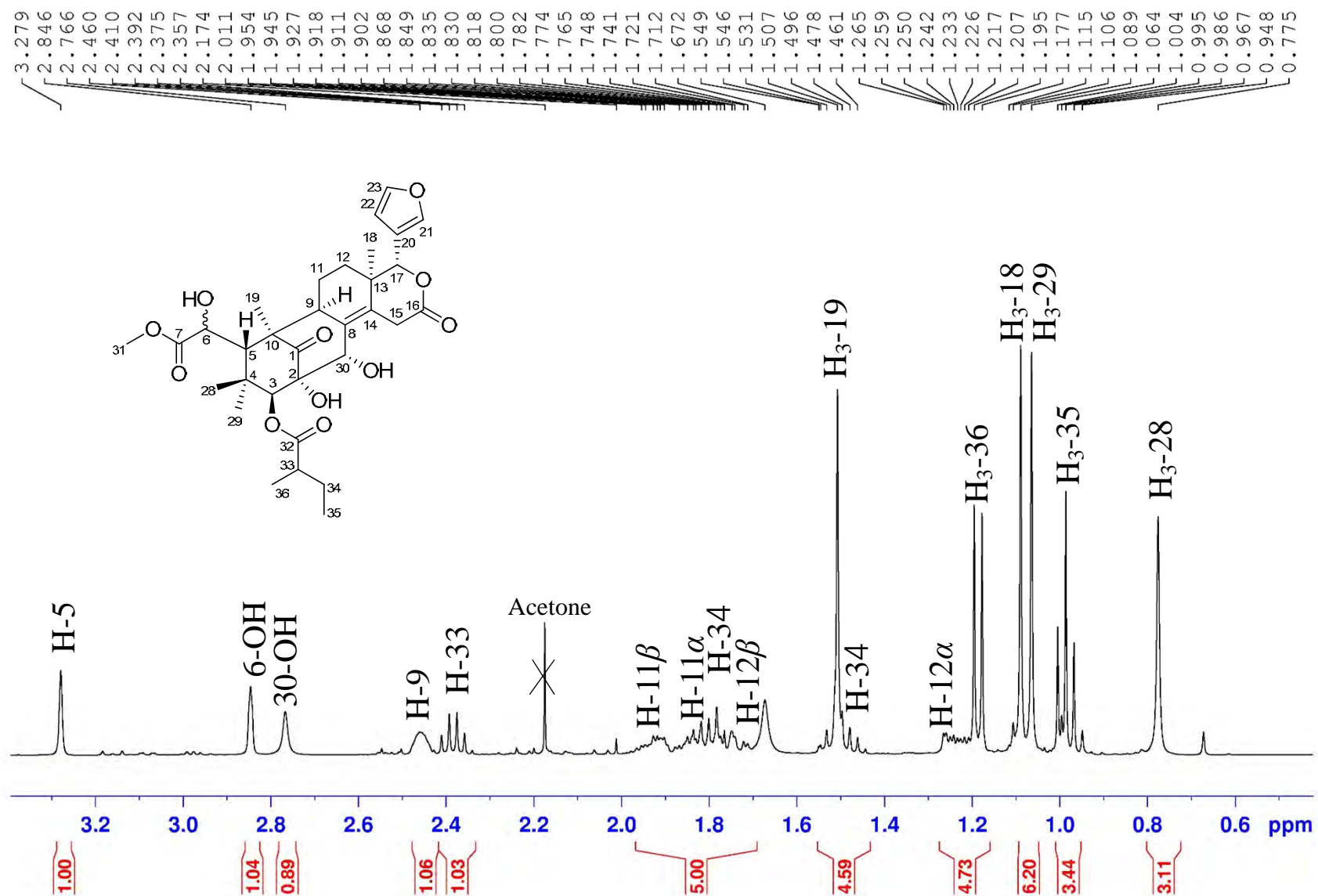
===== CHANNEL f1 =====
SFO1    400.1324710 M
NUC1     1H
P1       12.00 u
SI       65536
SF       400.1300058 M
WDW      EM
SSB      0
LB       0.30 F
GB       0
PC       1.00
    
```

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

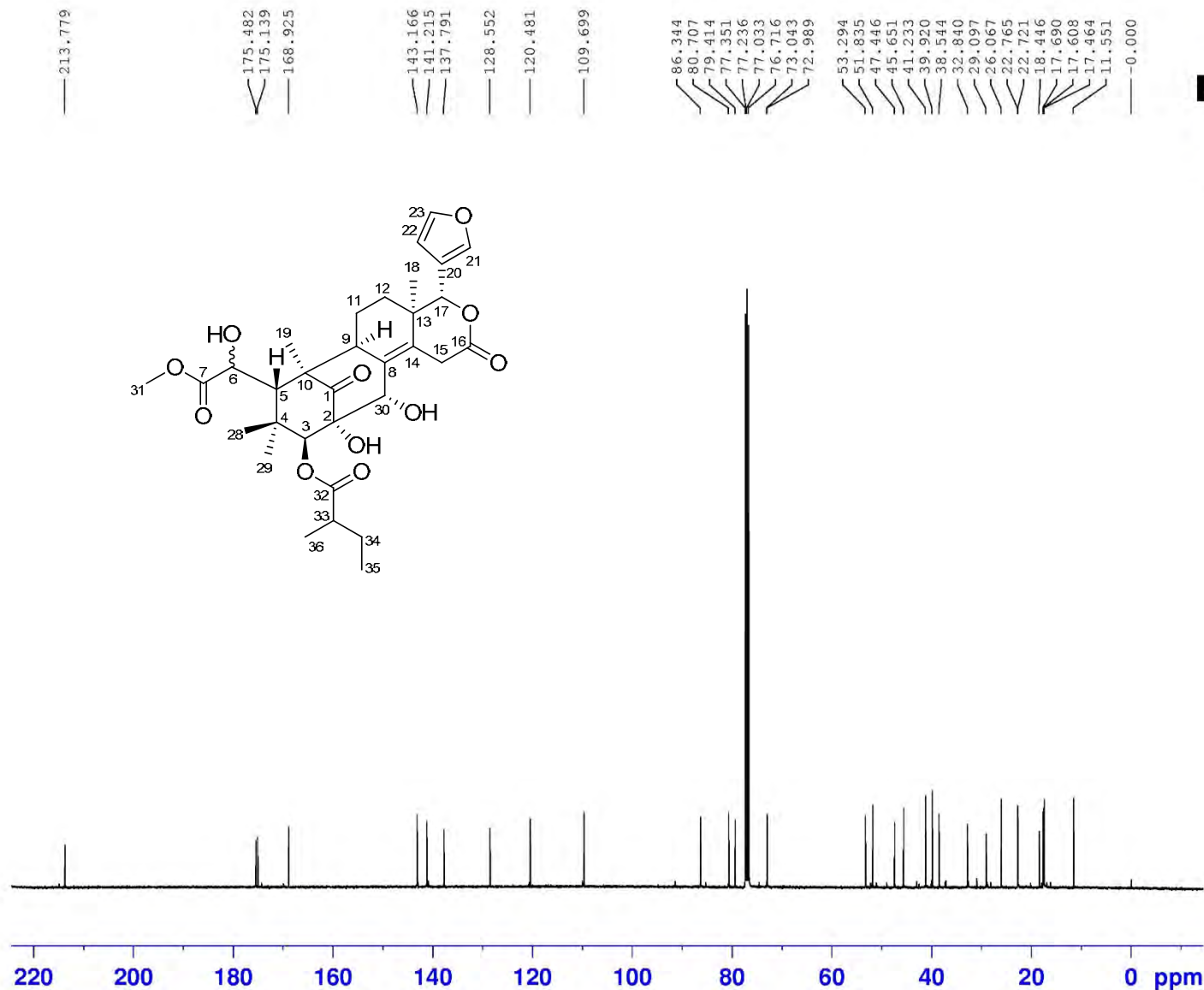




$^1\text{H}$  NMR (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



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



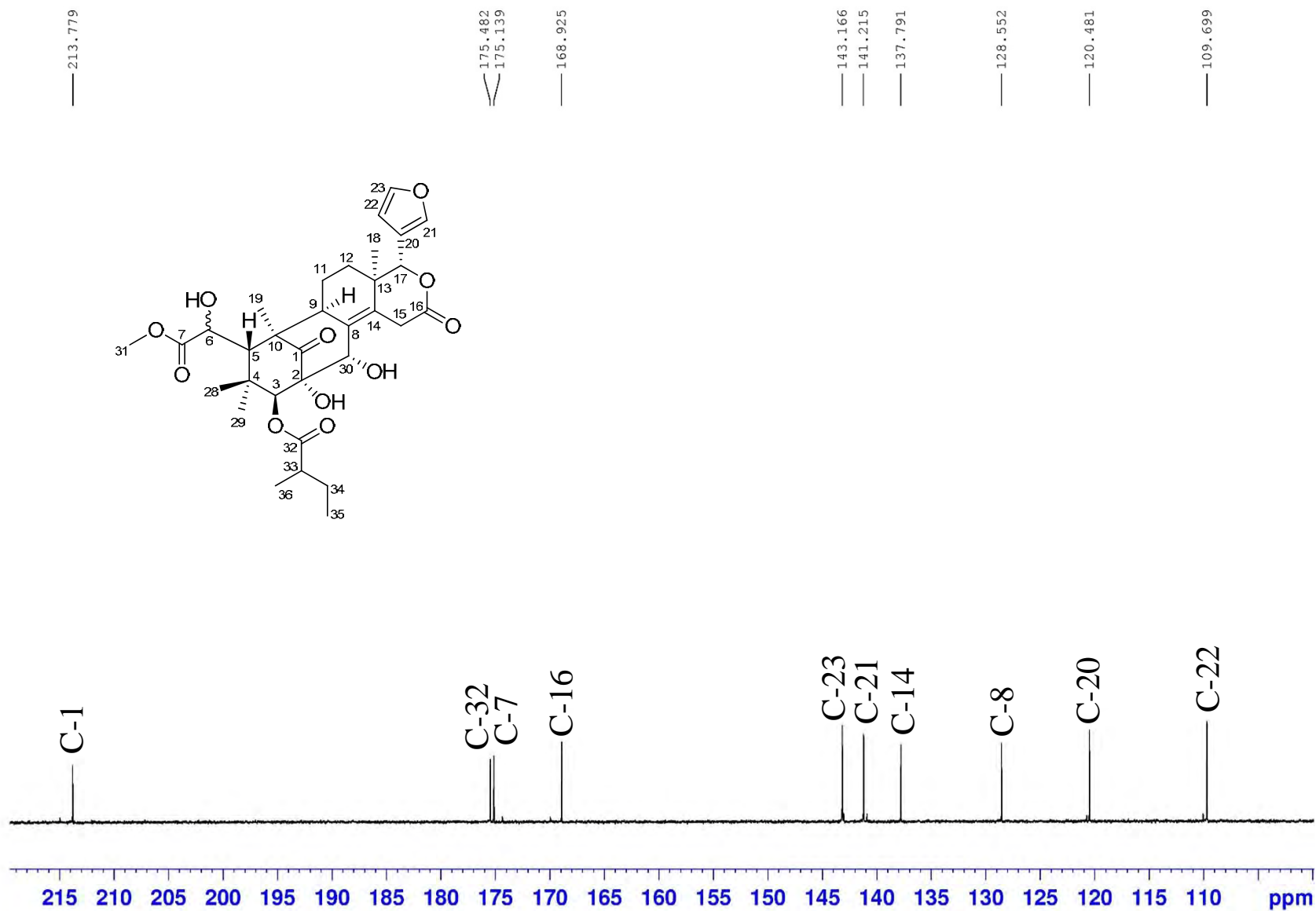
```

NAME          lws-63-1
EXPNO         2
PROCNO        1
Date_         20150708
Time          7.29
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 F
FIDRES        0.366798 F
AQ            1.3631988 s
RG            85.34
DW            20.800 u
DE            18.00 u
TE            297.0 K
D1            2.00000000 s
D11           0.03000000 s
TD0           1
  
```

```

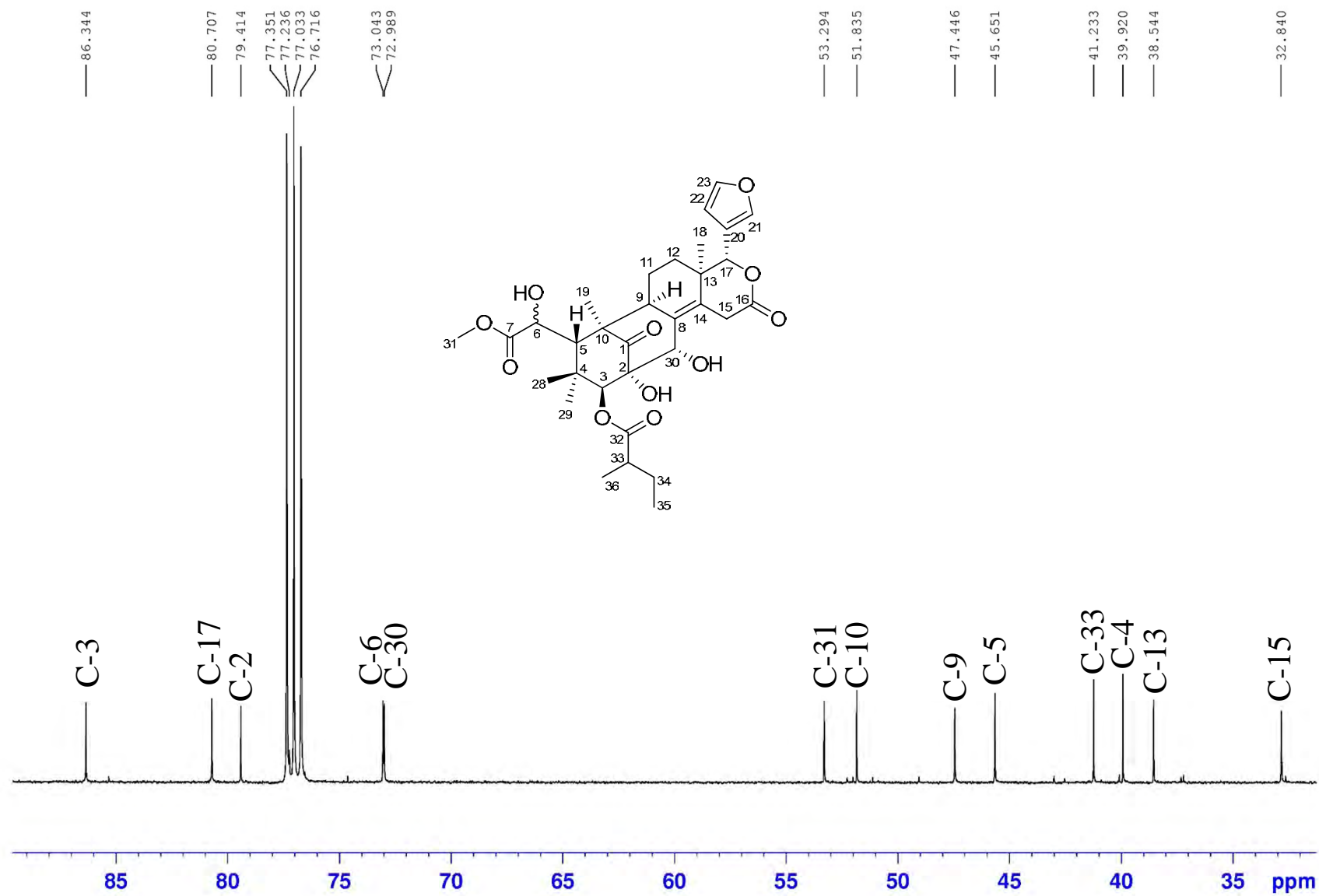
===== CHANNEL f1 =====
SFO1          100.6233324 M
NUC1          13C
P1            10.00 u
SI            32768
SF            100.6127689 M
WDW           EM
SSB           0
LB            1.00 F
GB            0
PC            1.40
  
```

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

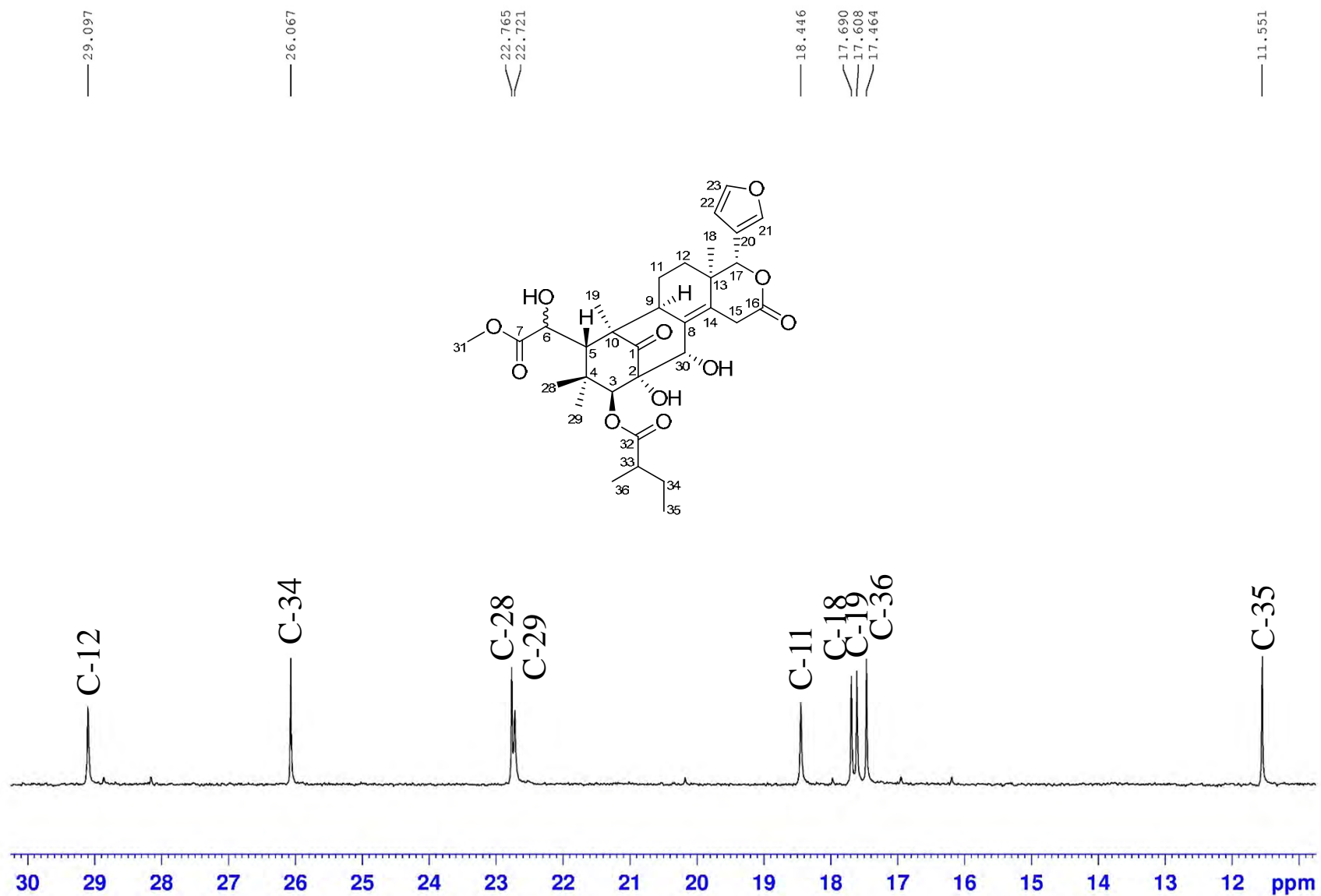




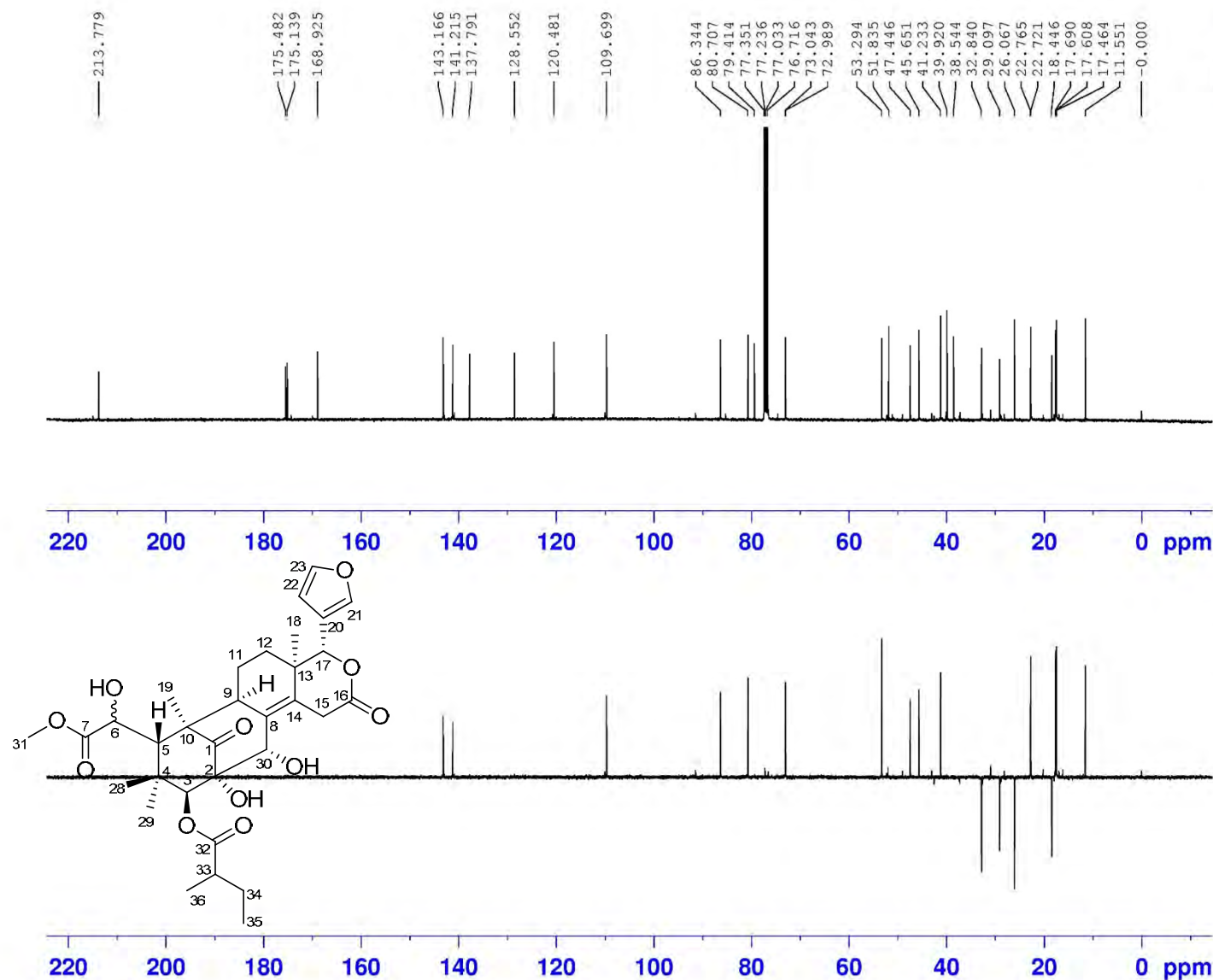
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



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



# DEPT135 (100 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



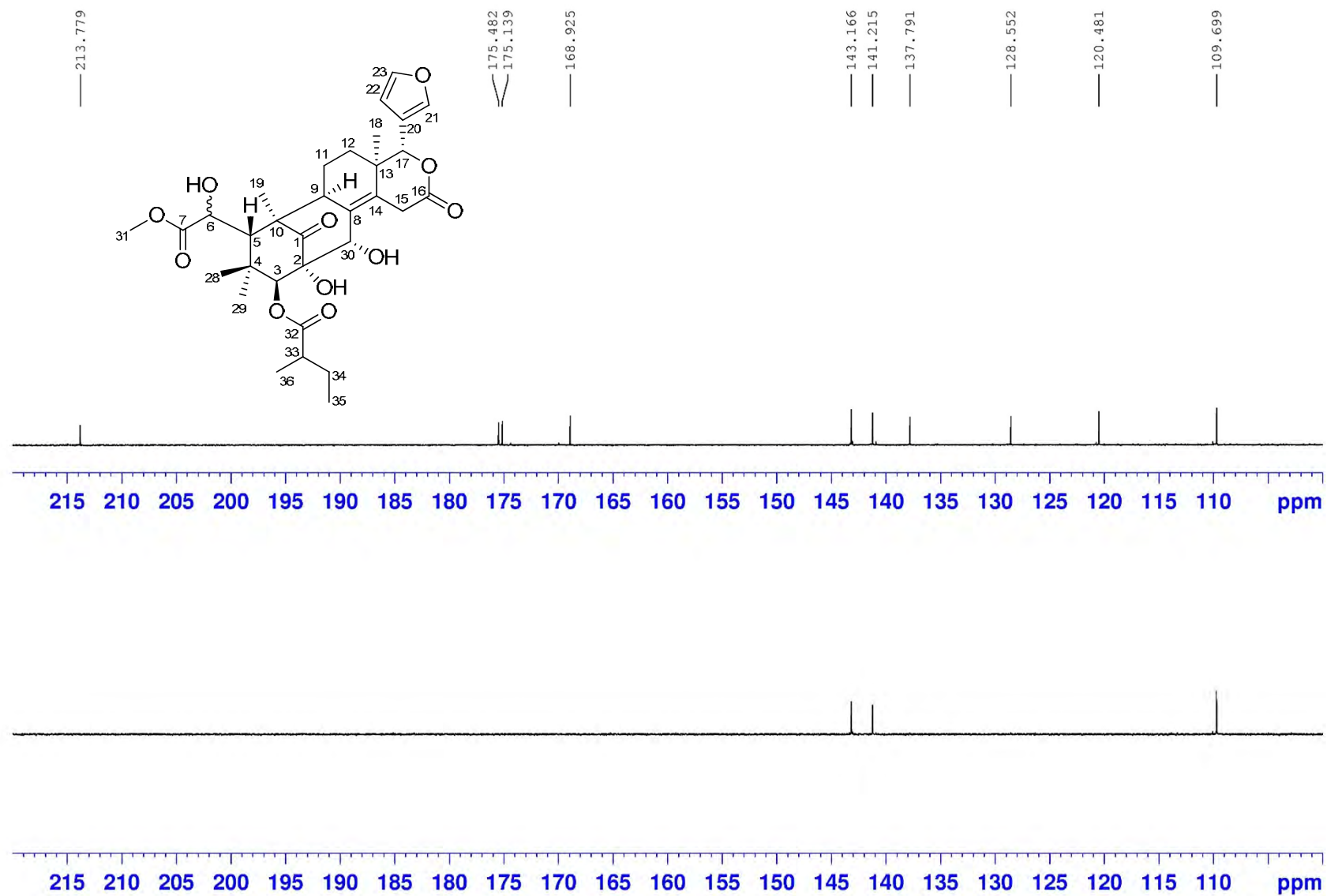
```

NAME          lws-63-1
EXPNO         3
PROCNO        1
Date_         20150708
Time          7.48
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       deptsp135
TD            65536
SOLVENT       CDCl3
NS            300
DS            4
SWH           24038.461 F
FIDRES        0.366798 F
AQ            1.3631988 s
RG            130.26
DW            20.800 u
DE            18.00 u
TE            297.0 F
CNST2         145.0000000
D1            2.0000000 s
D2            0.00344828 s
D12           0.00002000 s
TD0           1
    
```

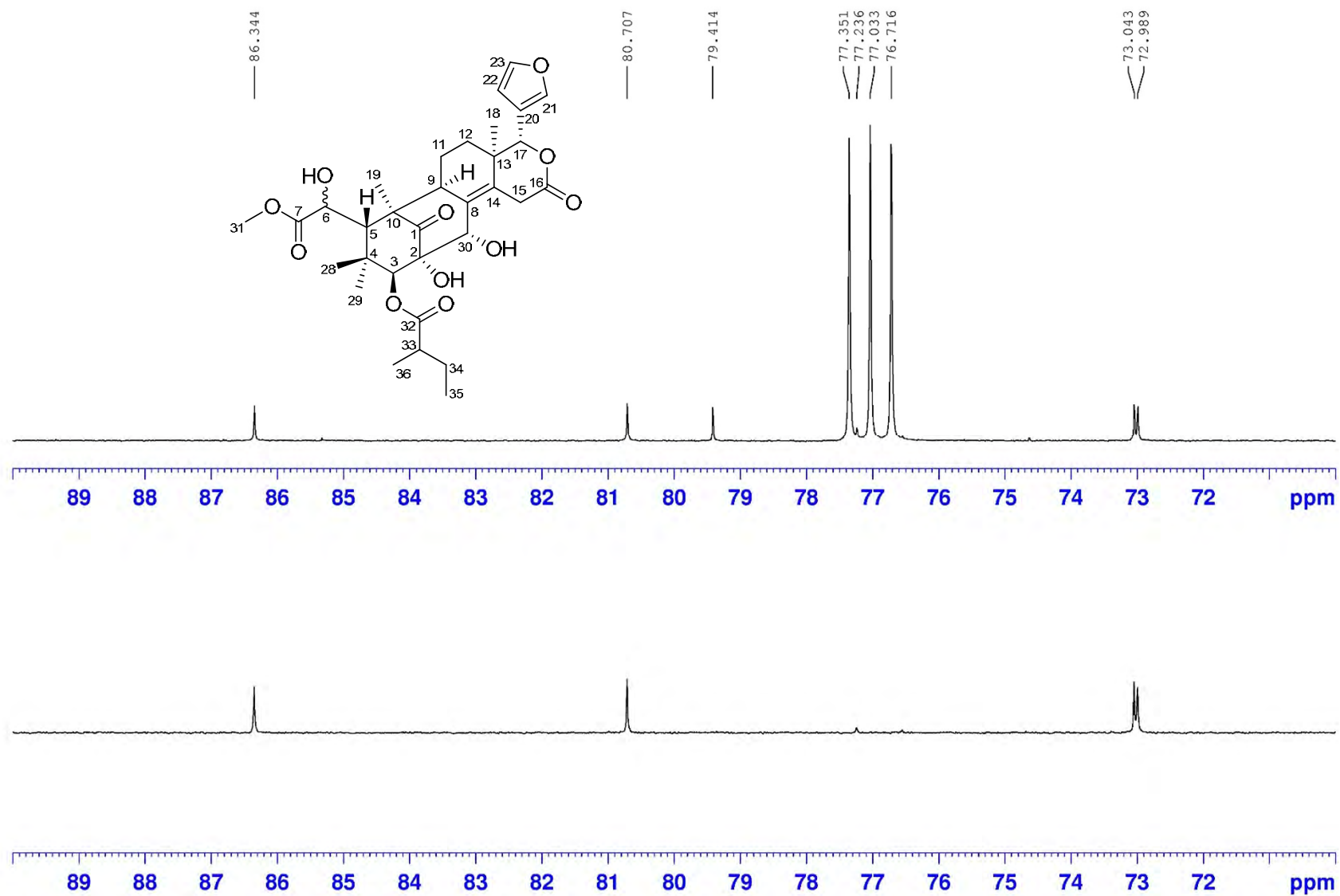
```

===== CHANNEL f1 =====
SFO1         100.623324 M
NUC1          13C
P1            10.00 u
P13           2000.00 u
SI            32768
SF            100.6127689 M
WDW           EM
SSB           0
LB            1.00 F
GB            0
PC            1.40
    
```

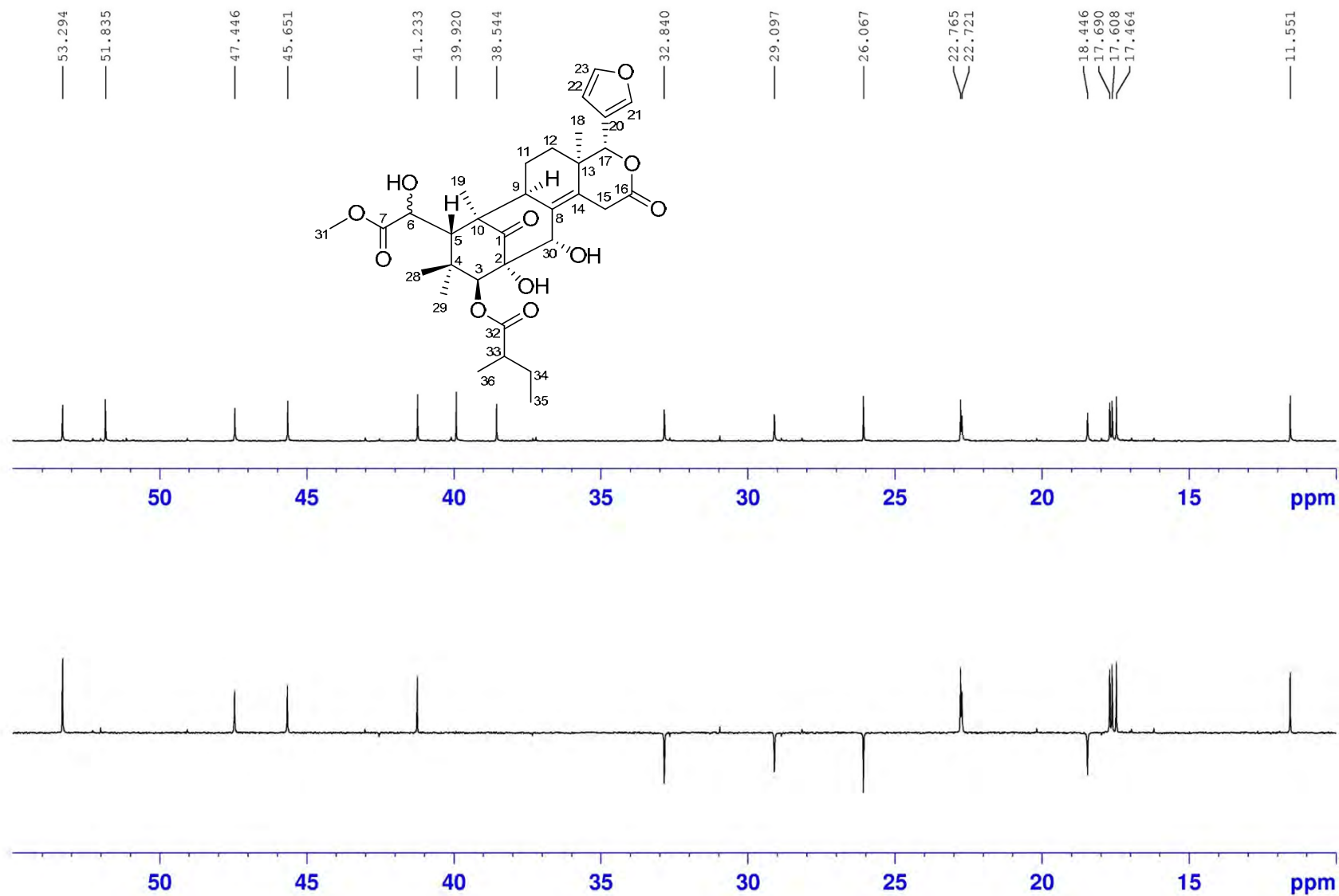
# DEPT135 (100 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



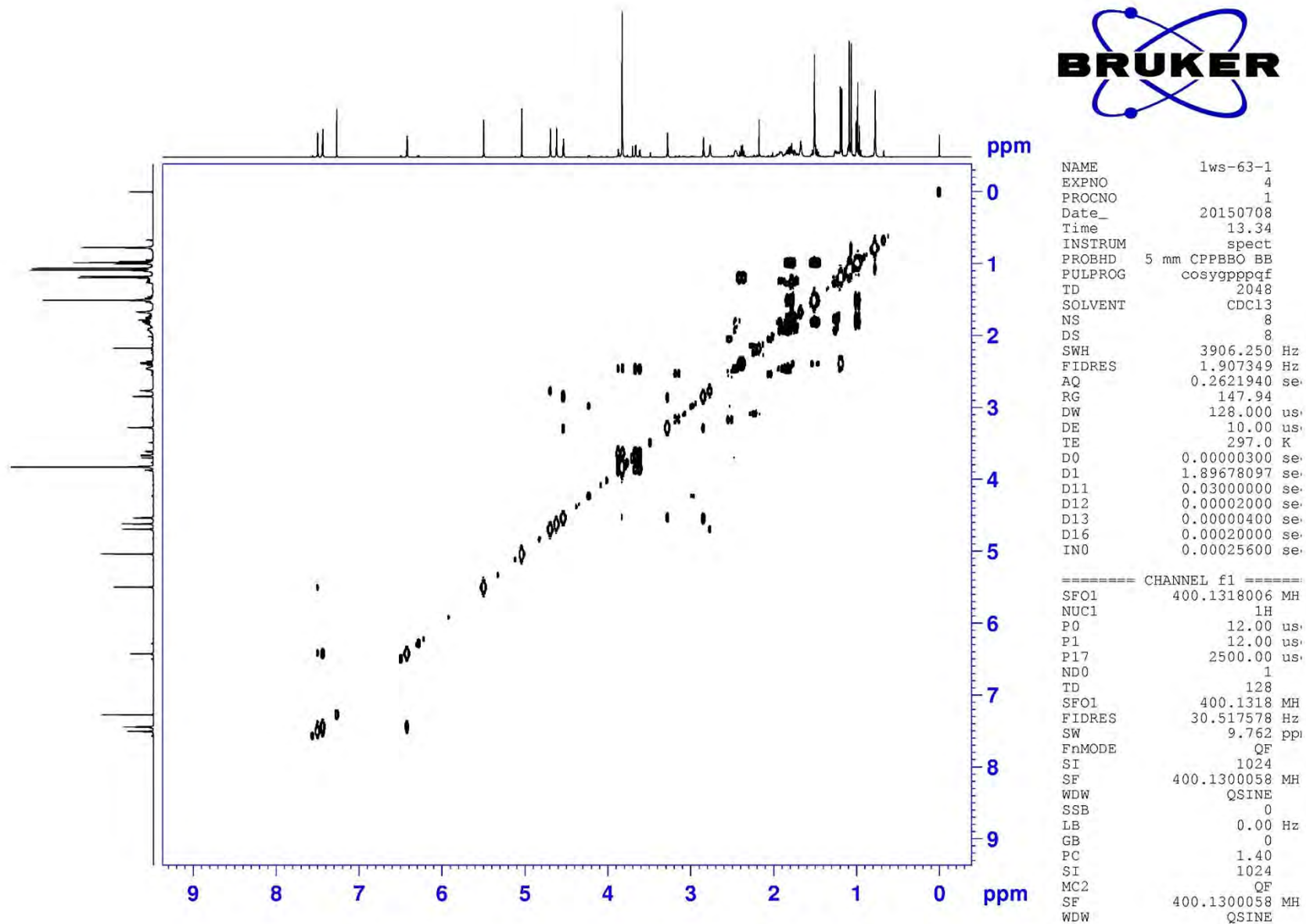
# DEPT135 (100 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



# DEPT135 (100 MHz) spectrum of compound **3** in CDCl<sub>3</sub>

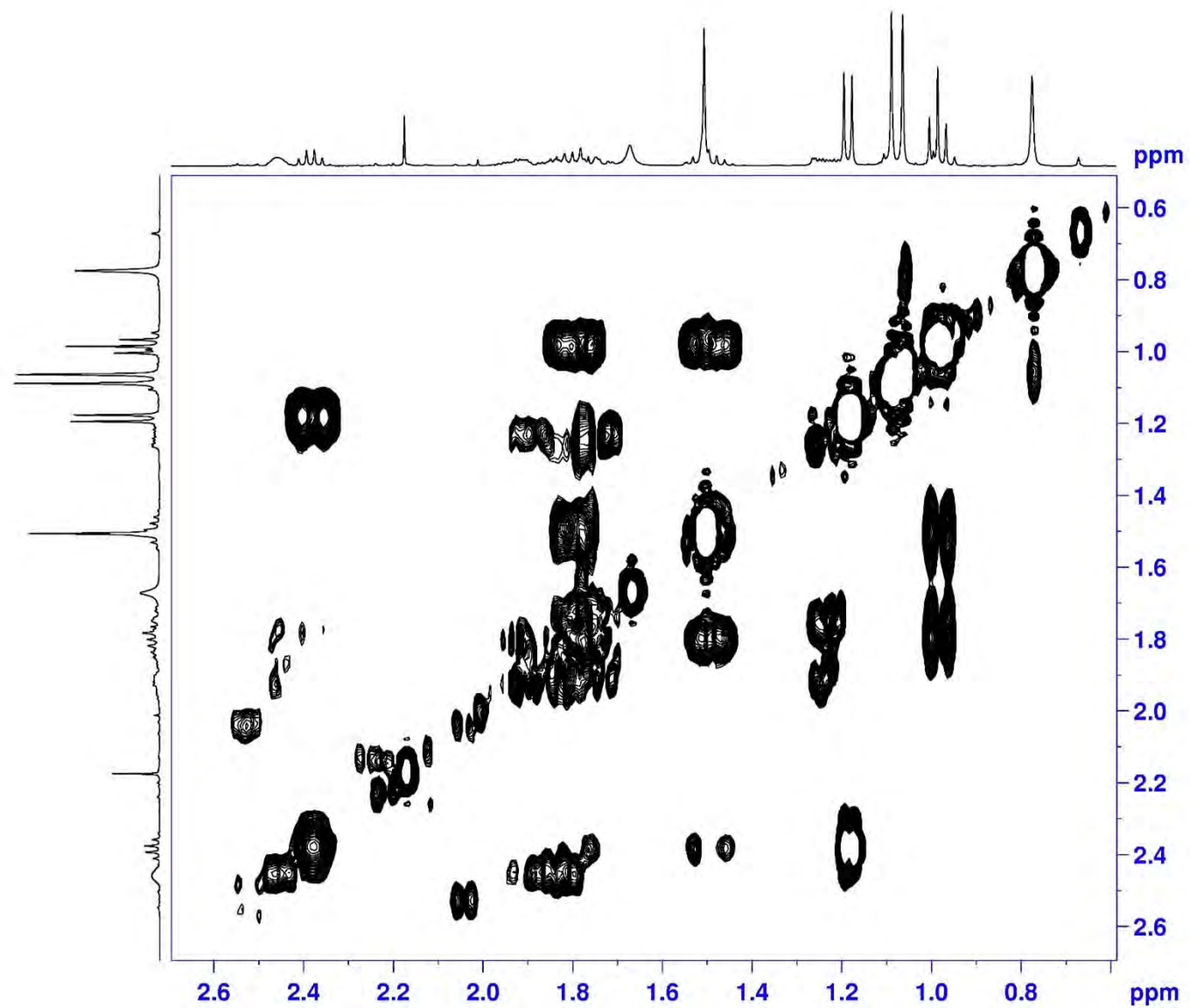


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



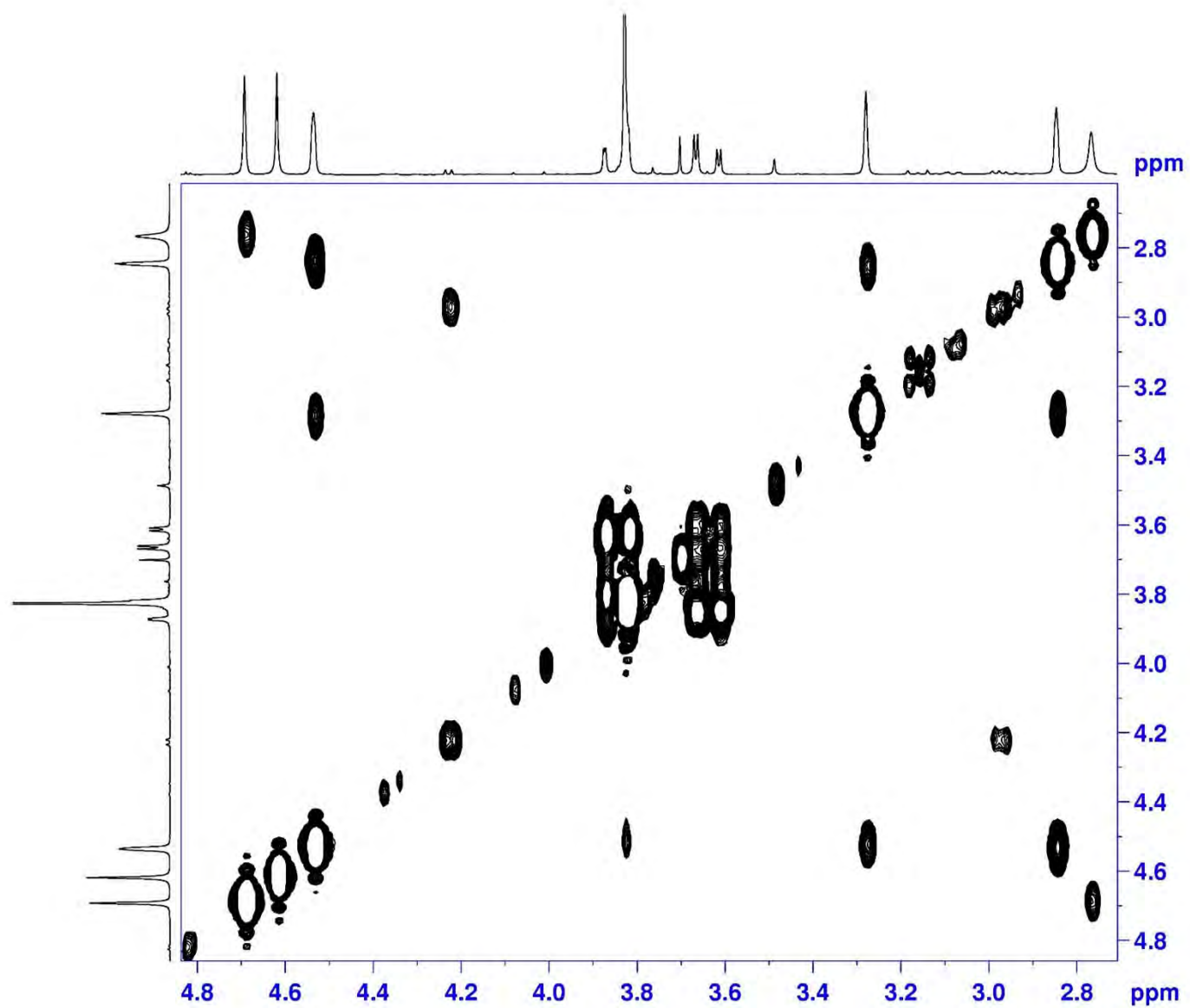


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$

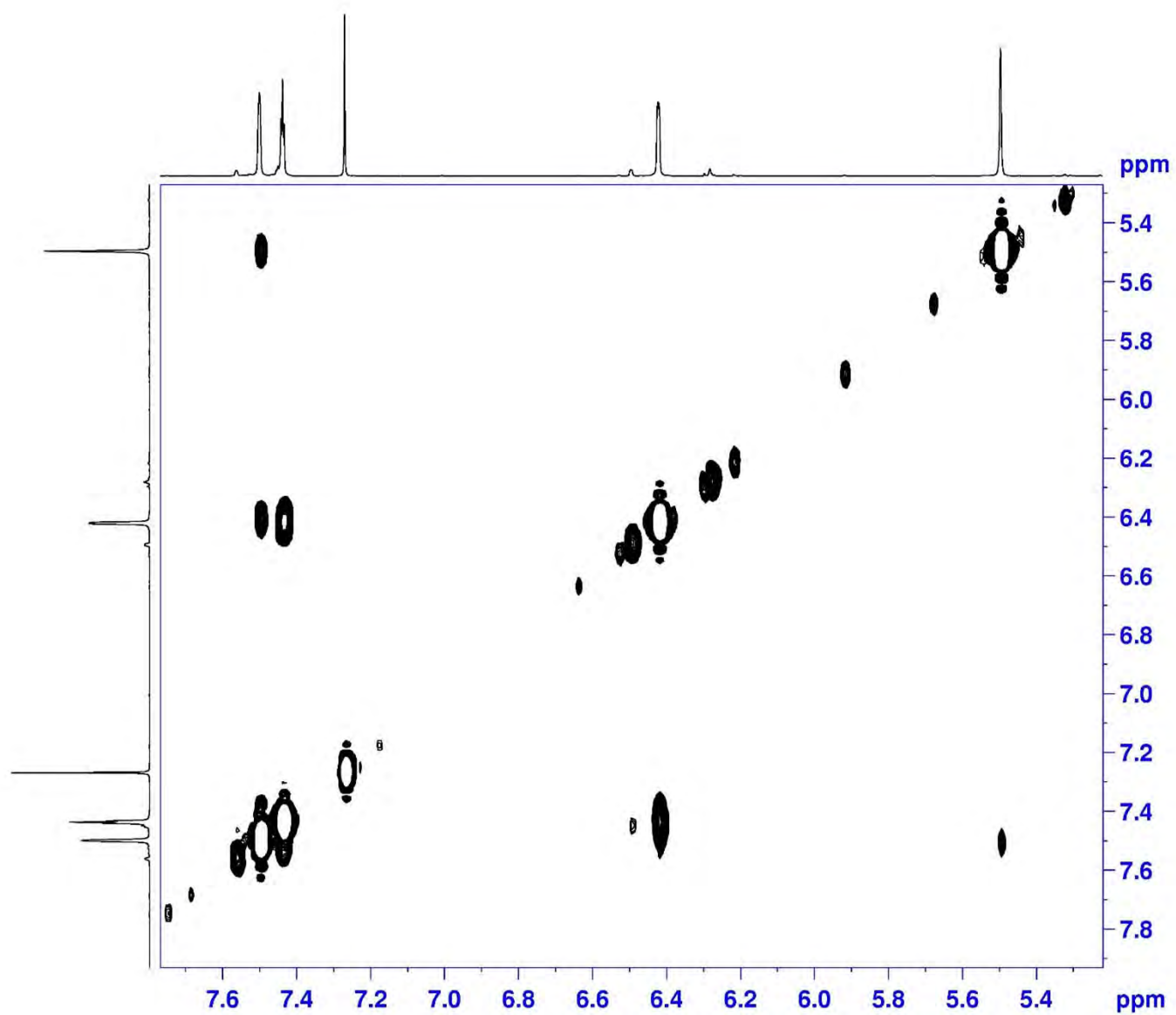




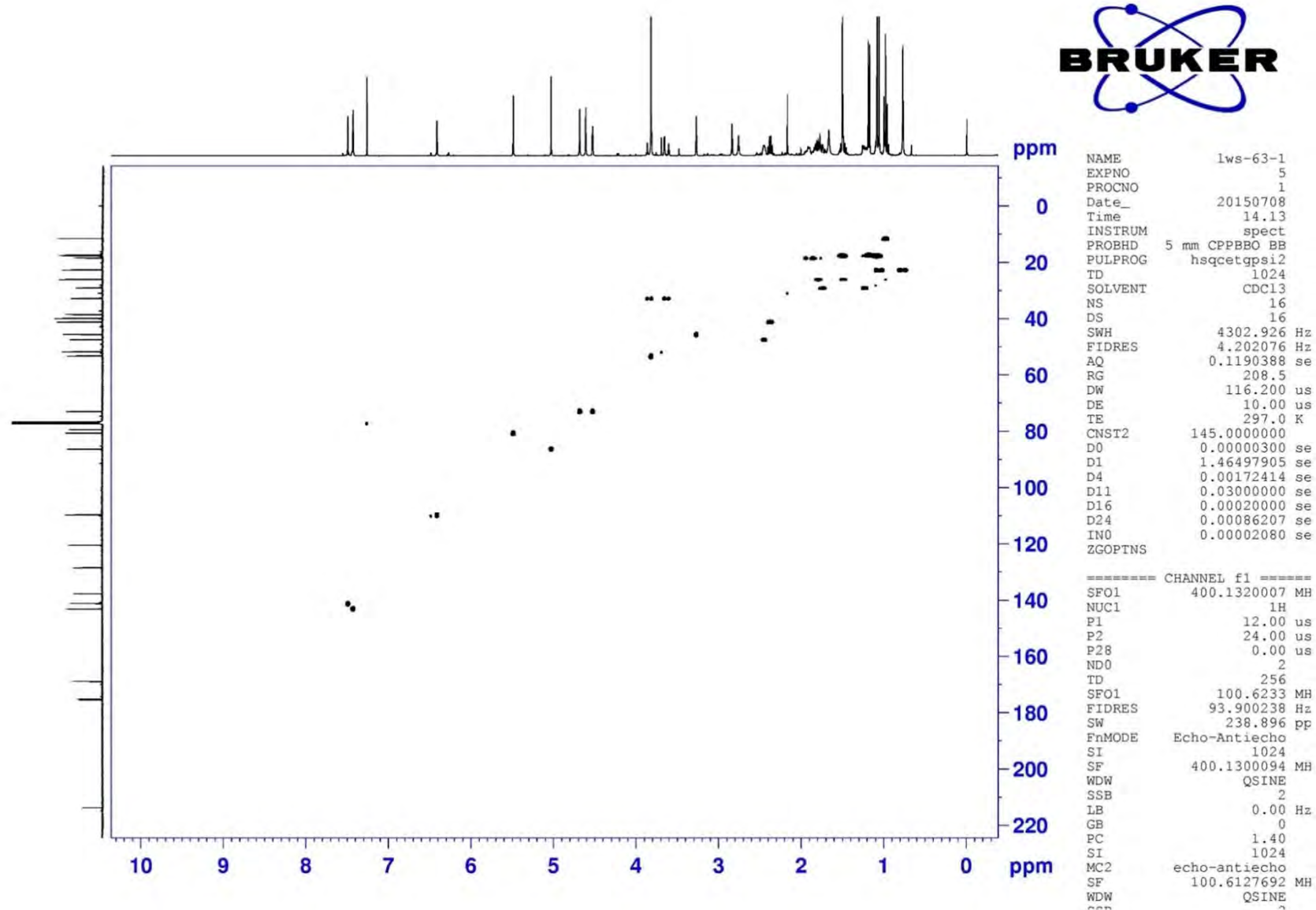
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



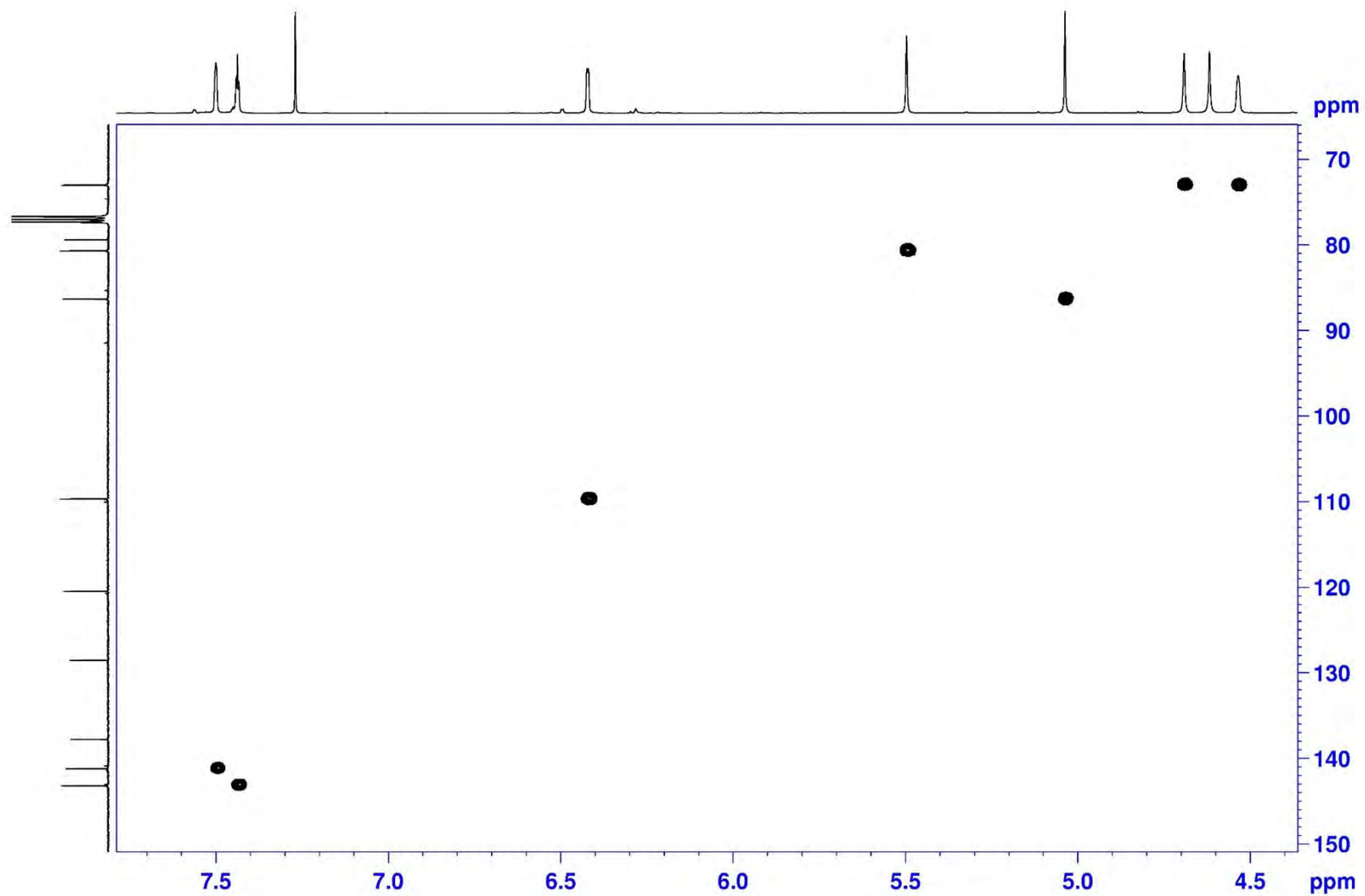
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



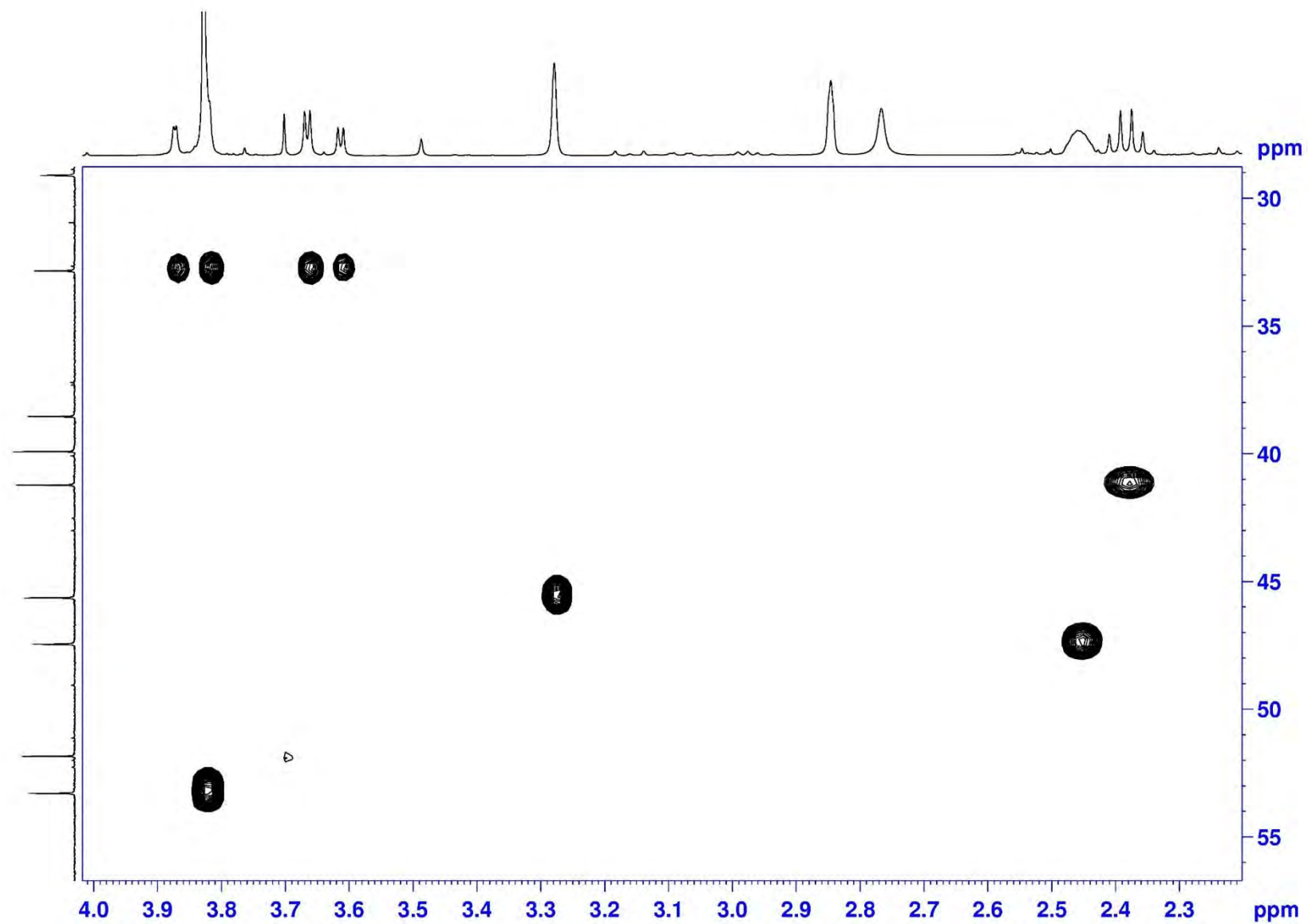
# HSQC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



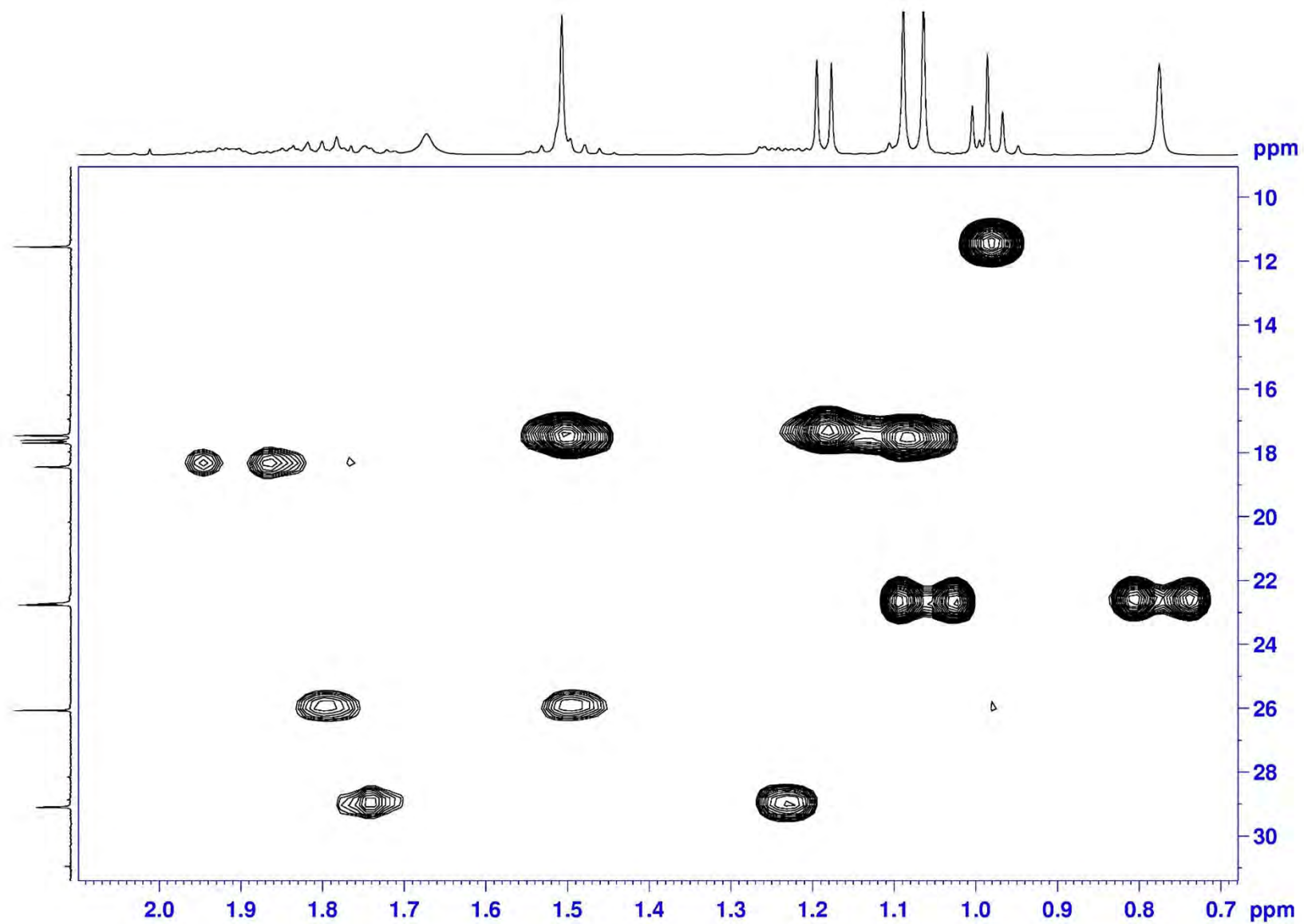
HSQC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



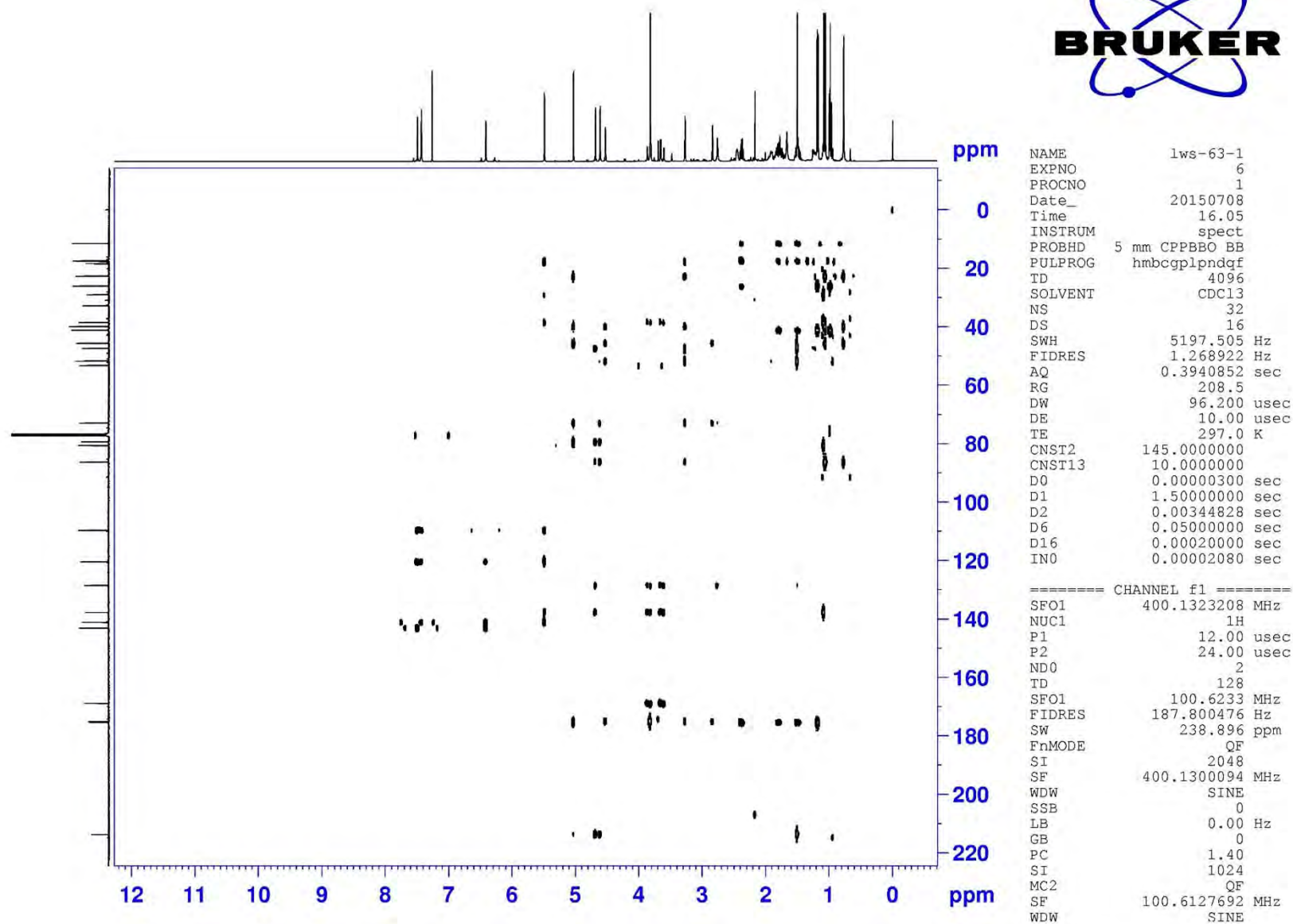
HSQC (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



HSQC (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$

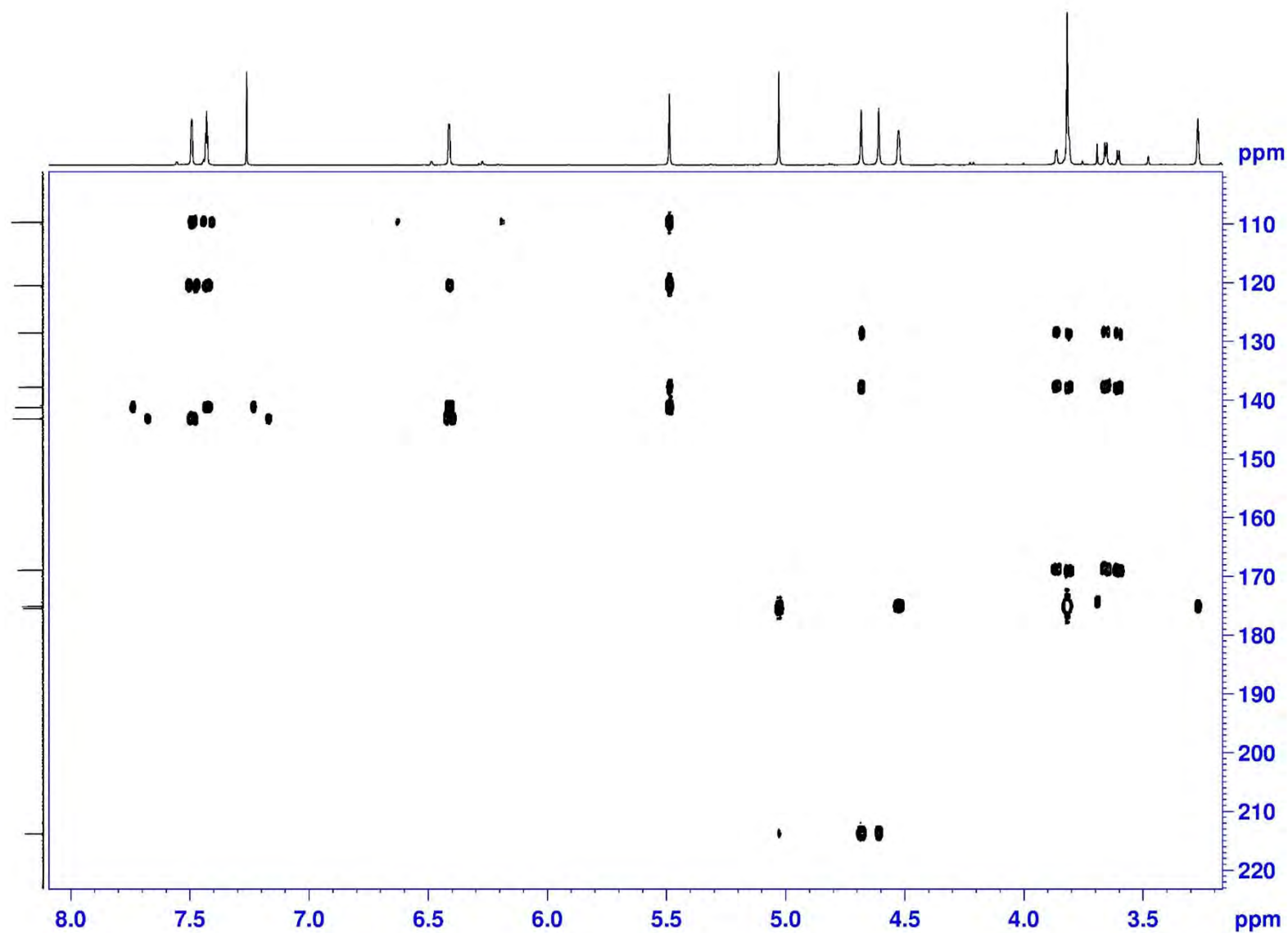


# HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>

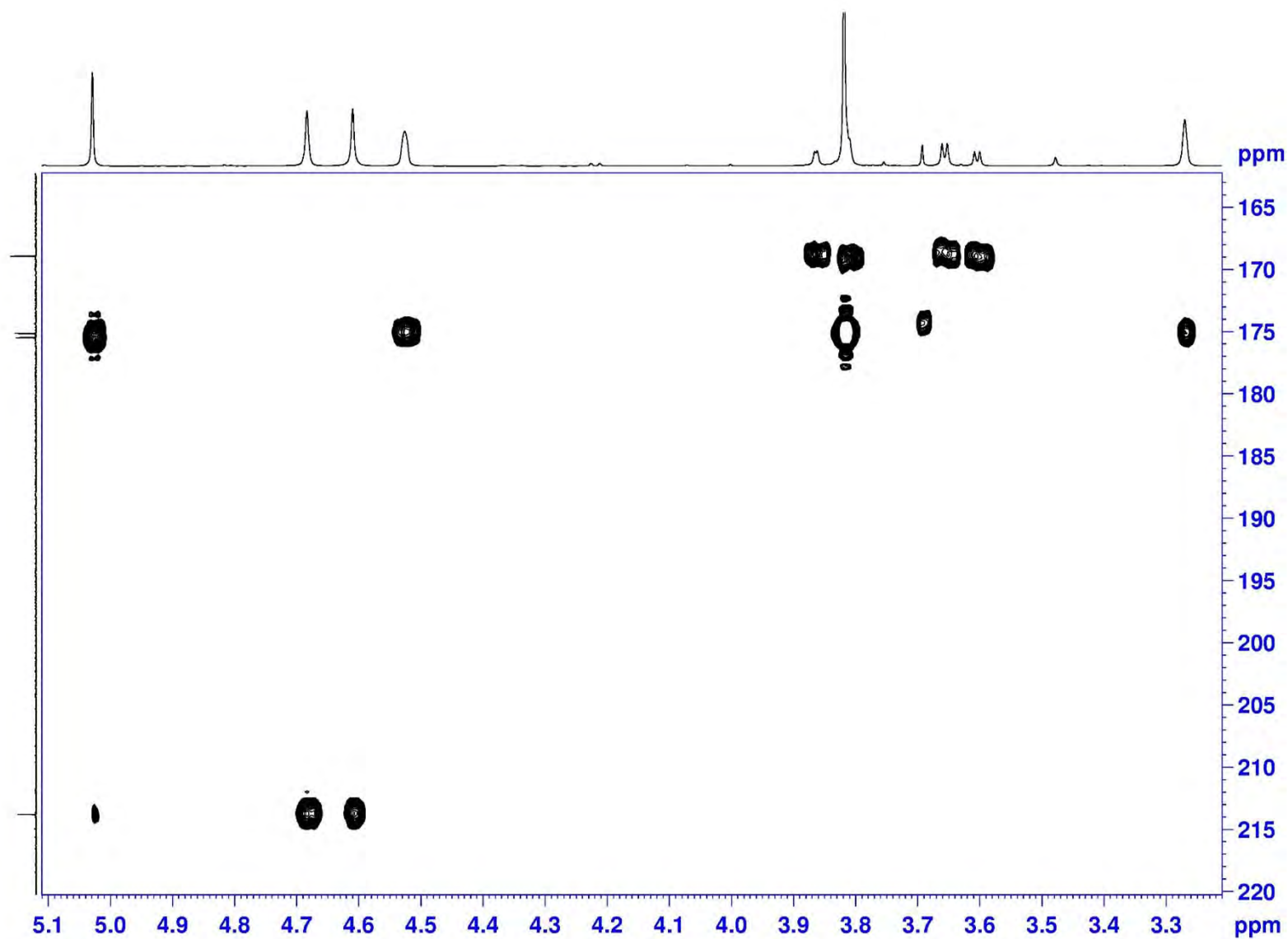




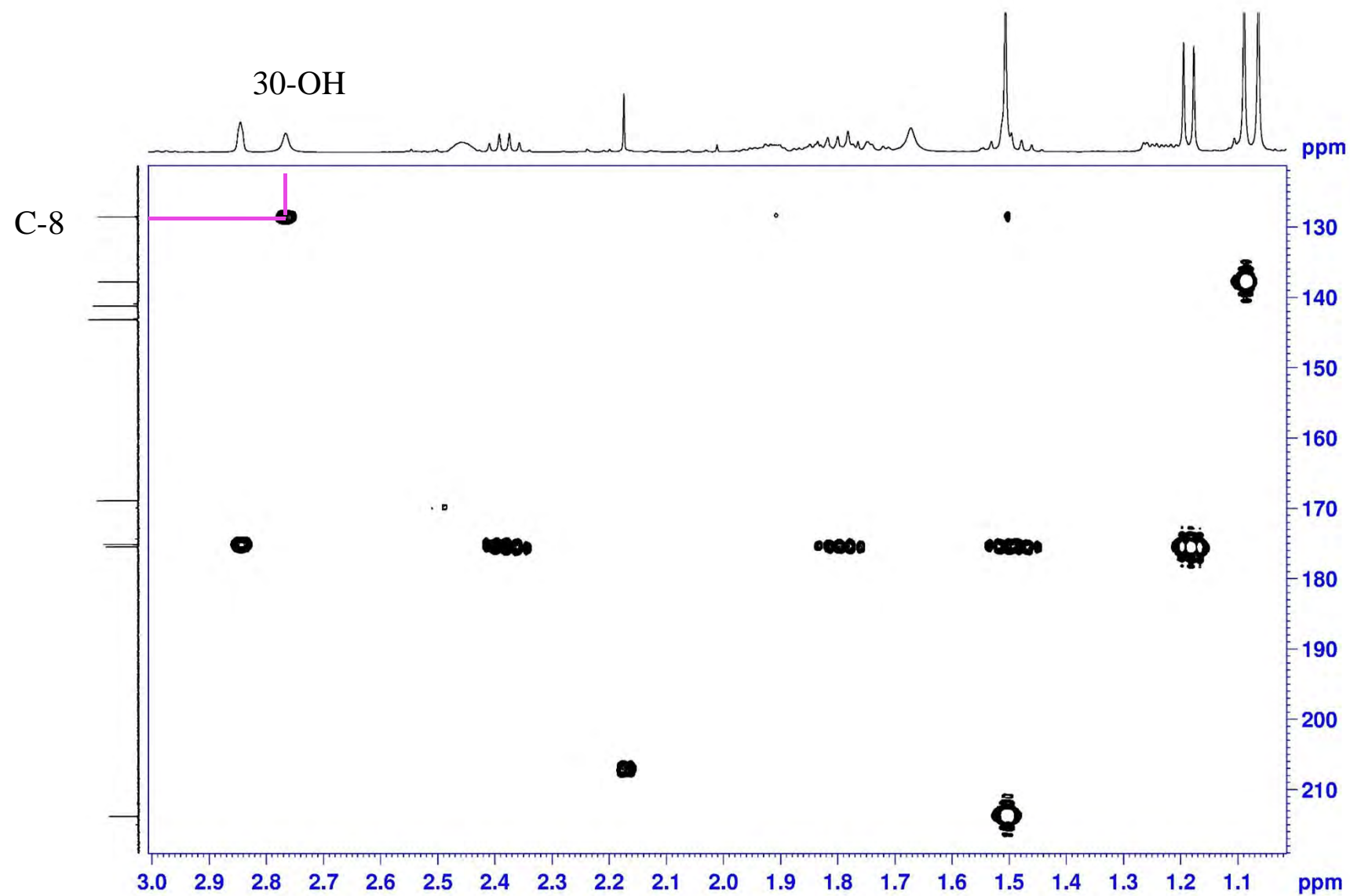
HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



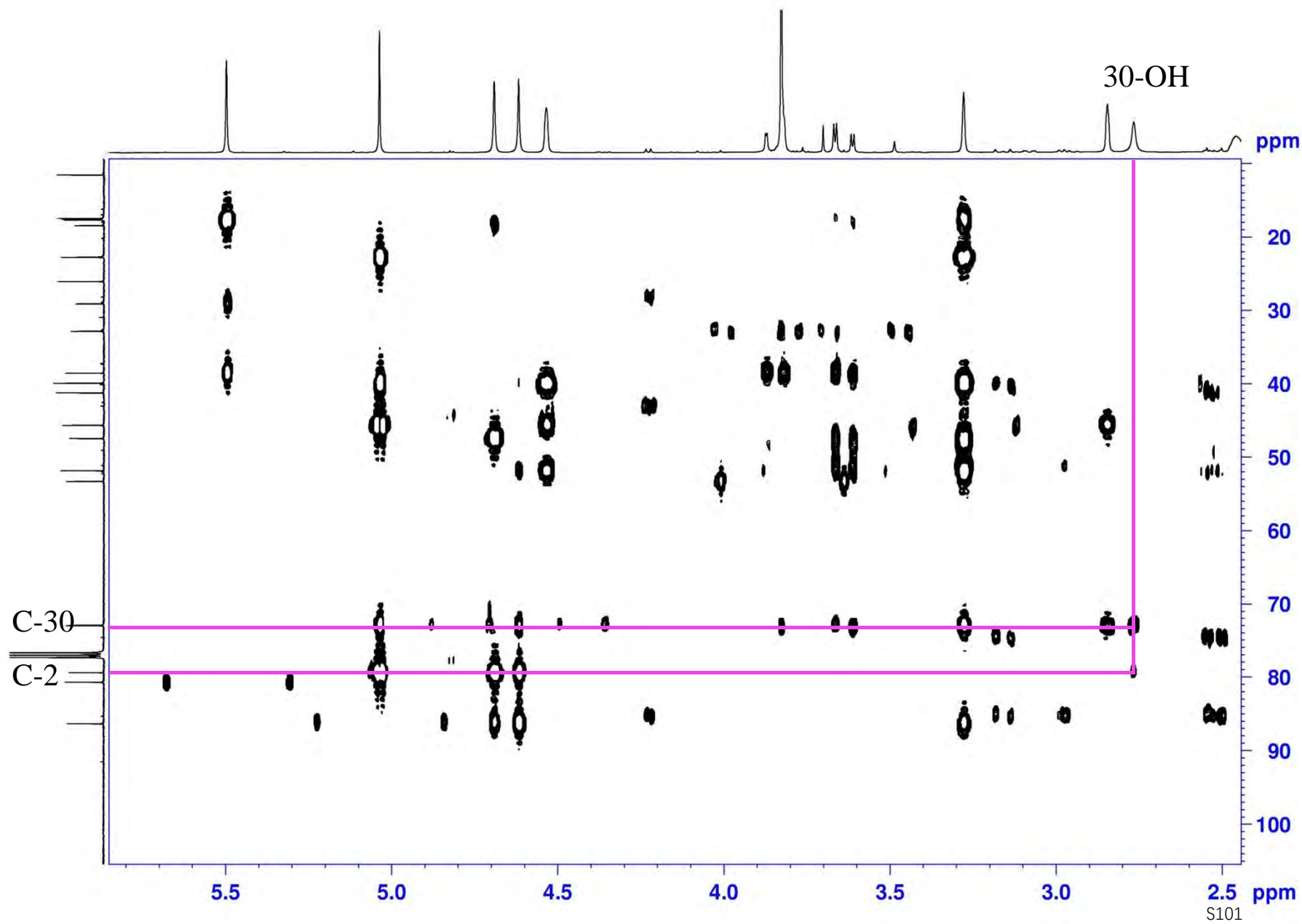
HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



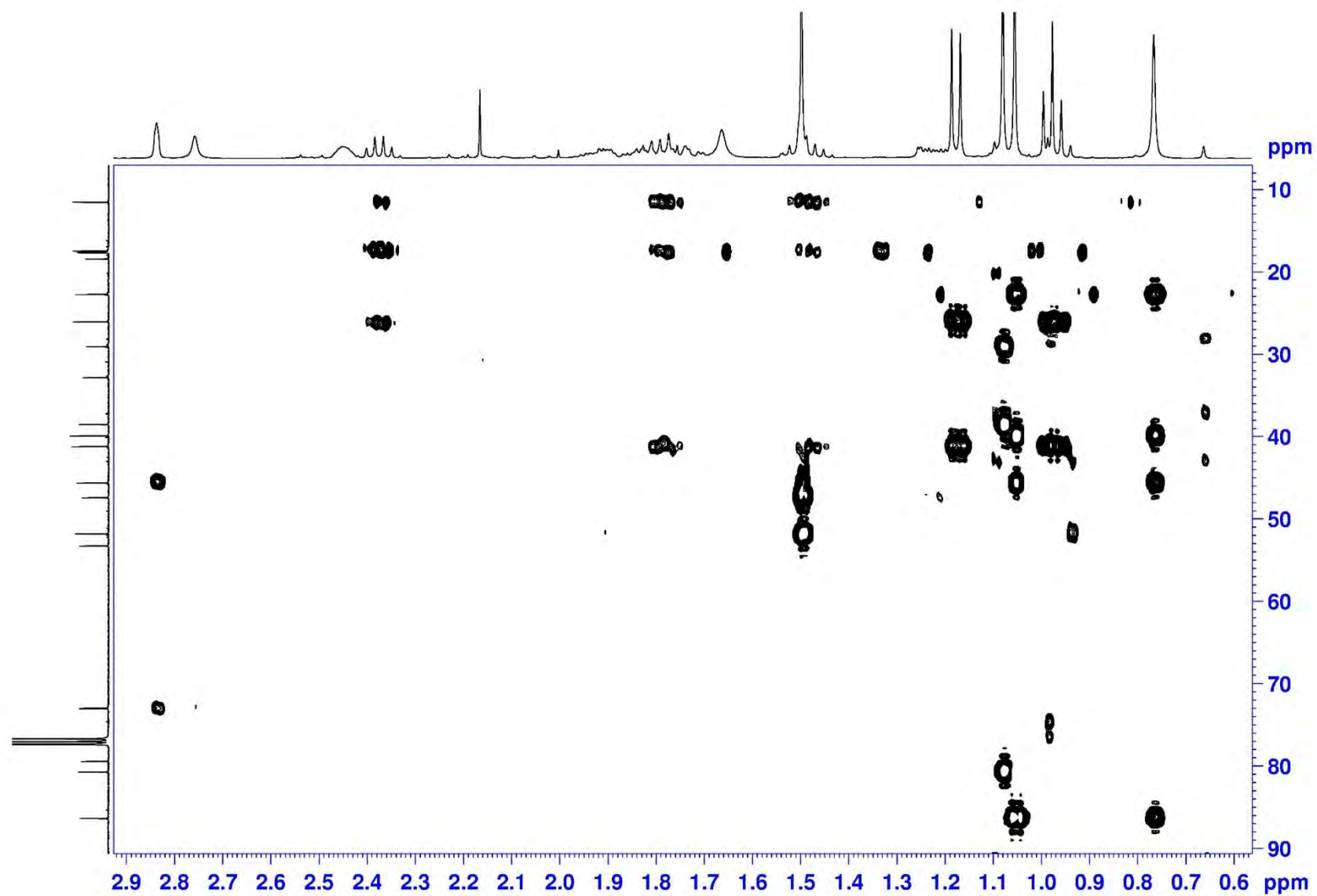
HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



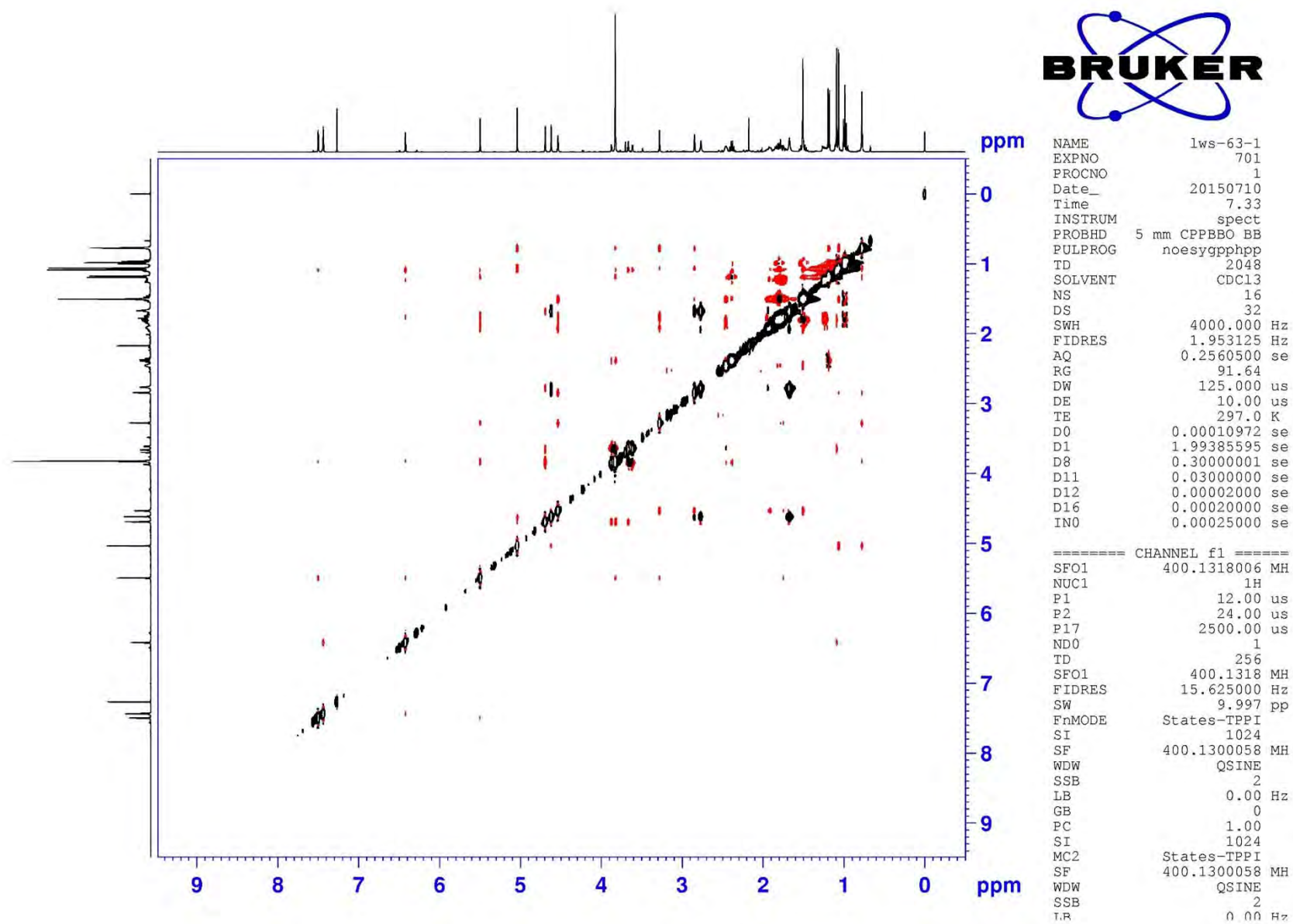
HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>

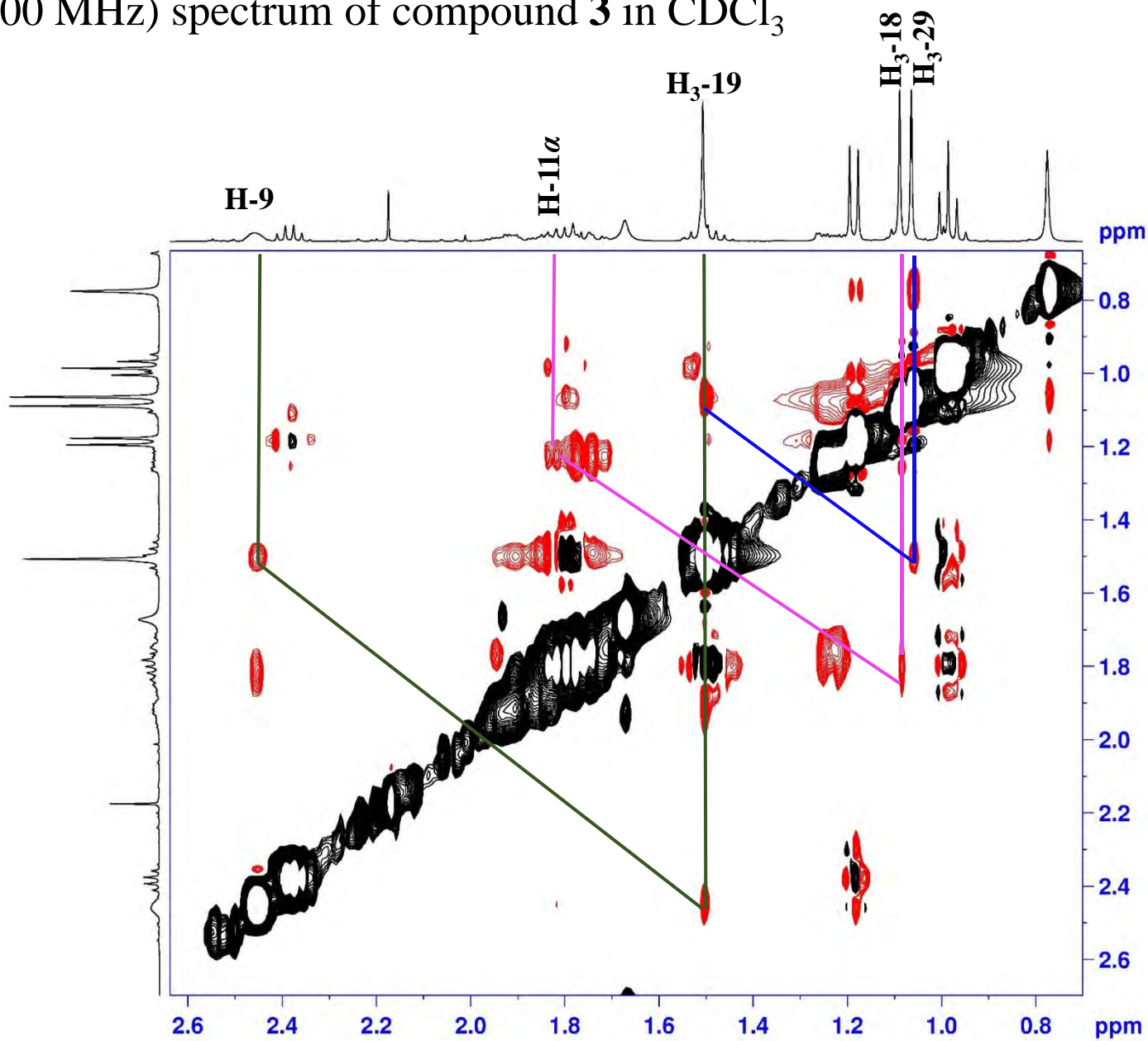


# NOESY (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



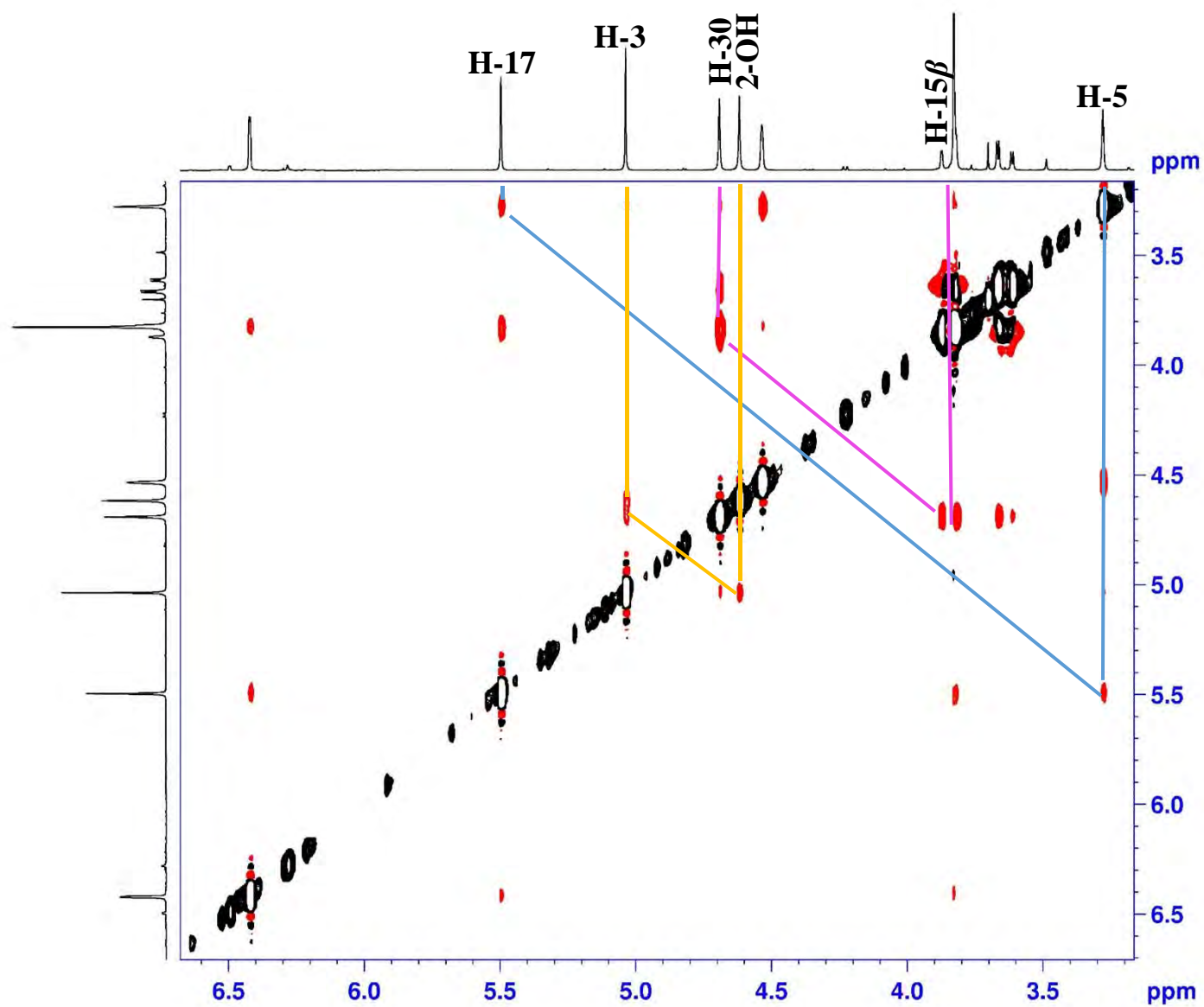


NOESY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$

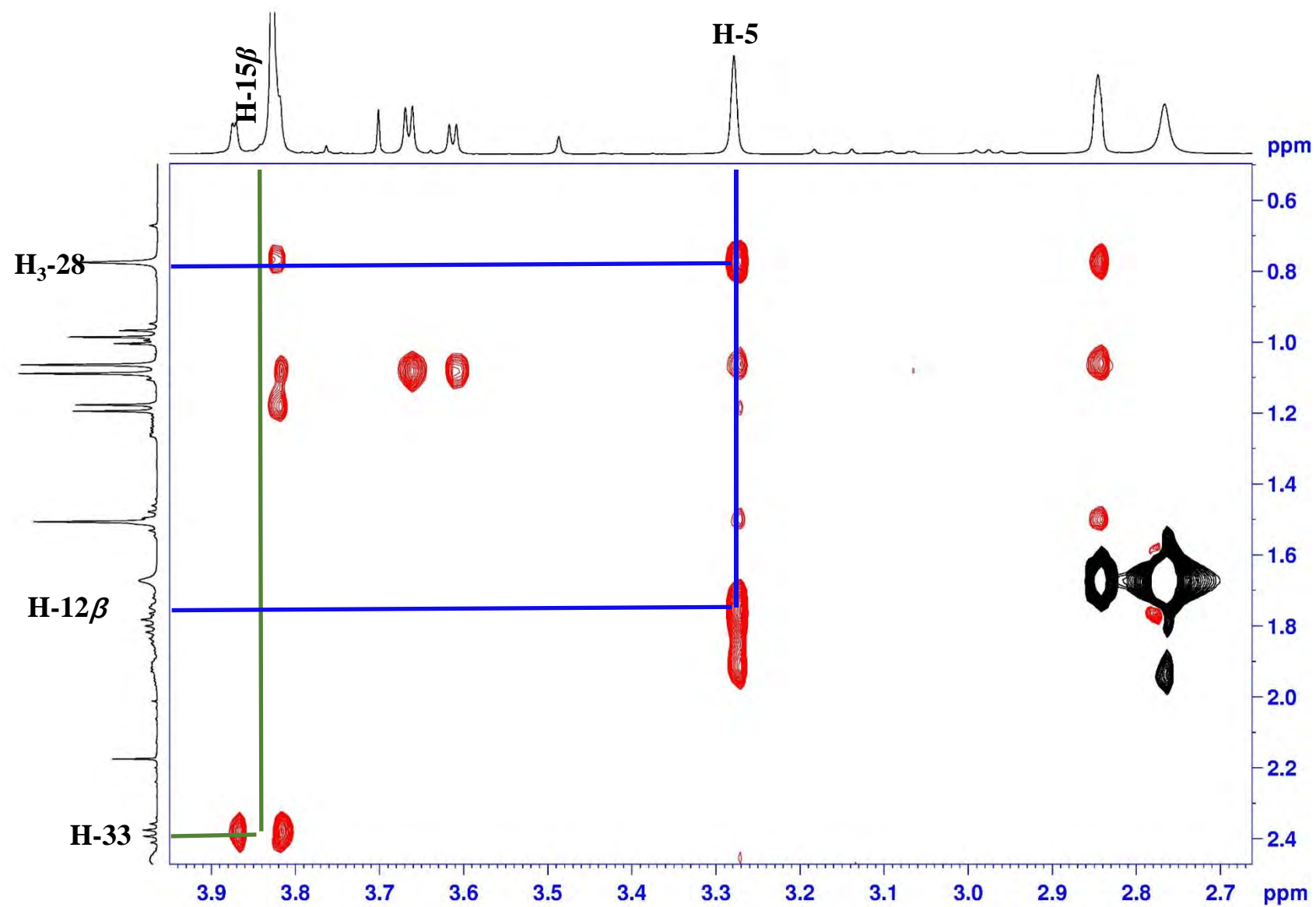




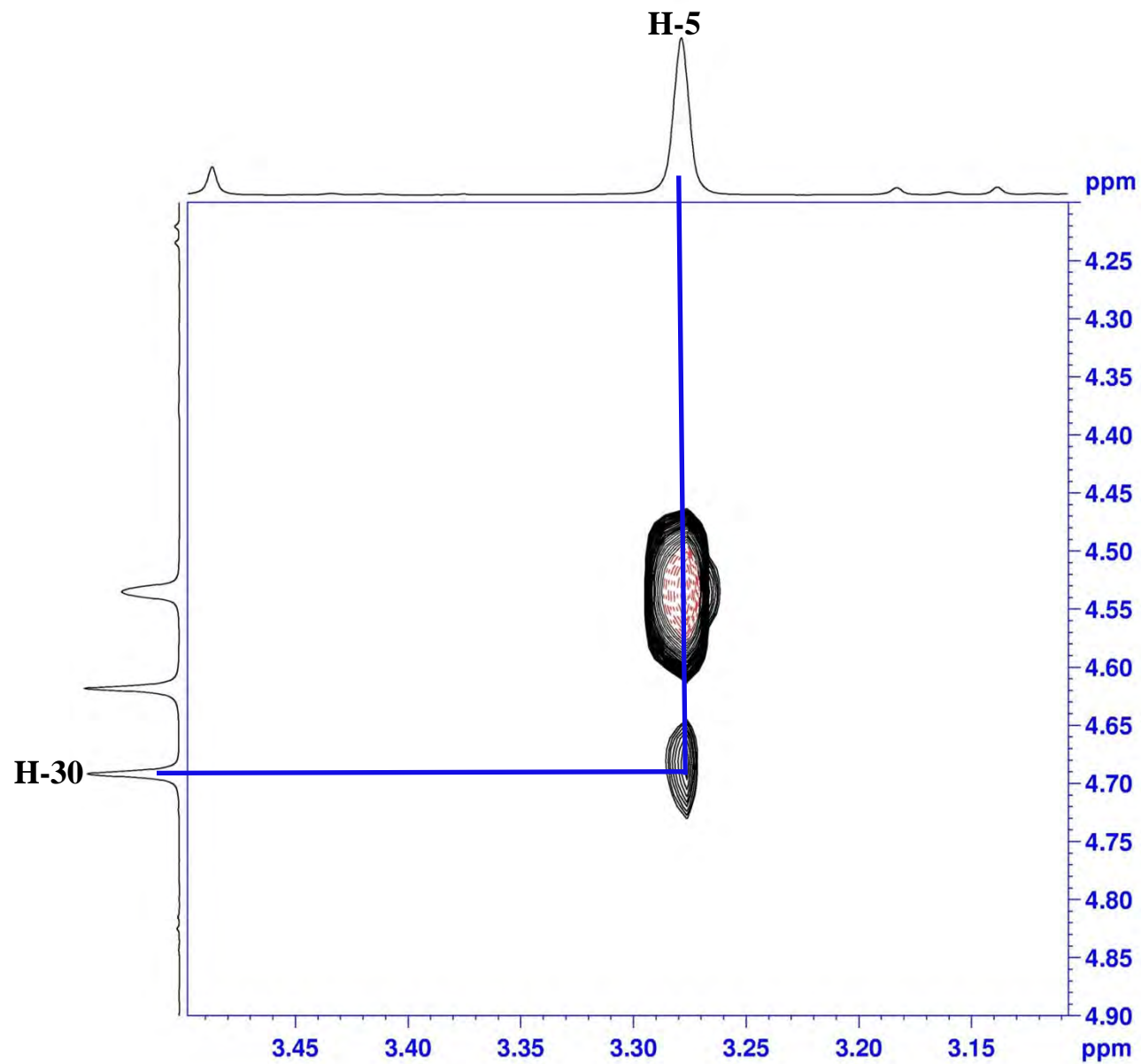
NOESY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



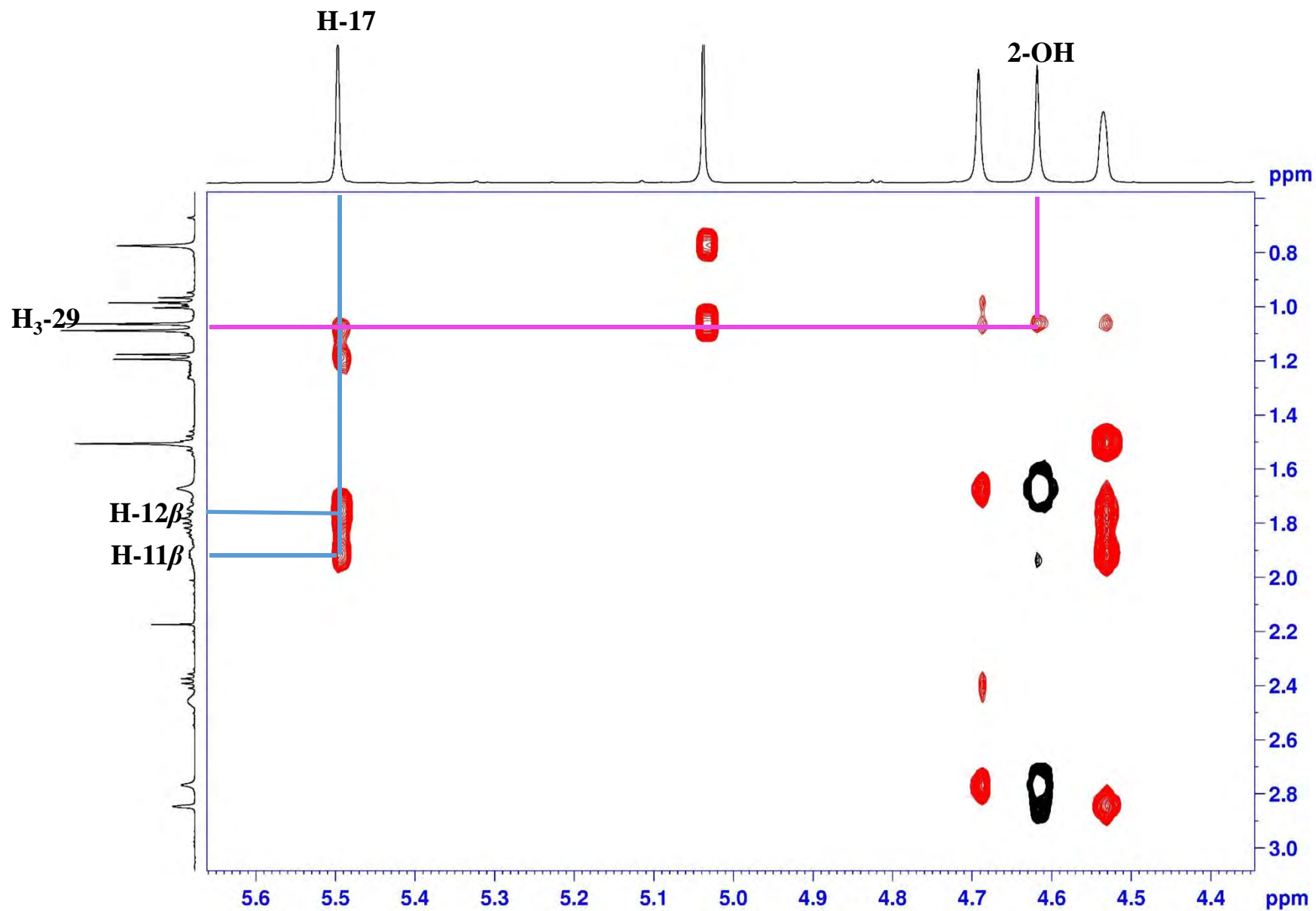
NOESY (400 MHz) spectrum of compound **3** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **3** in  $\text{CDCl}_3$



# HR-ESIMS for compound 4

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-91\_pos\_11\_01\_1858.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-91\_pos  
 Comment

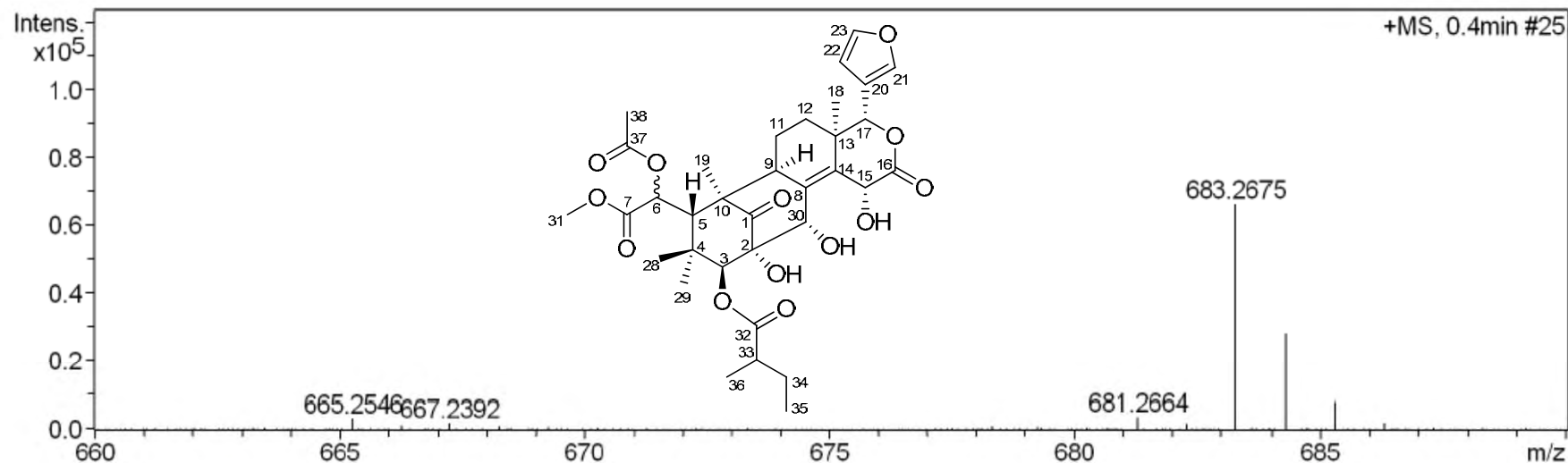
Acquisition Date 5/19/2016 8:51:32 AM

Operator SCSIO

Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste

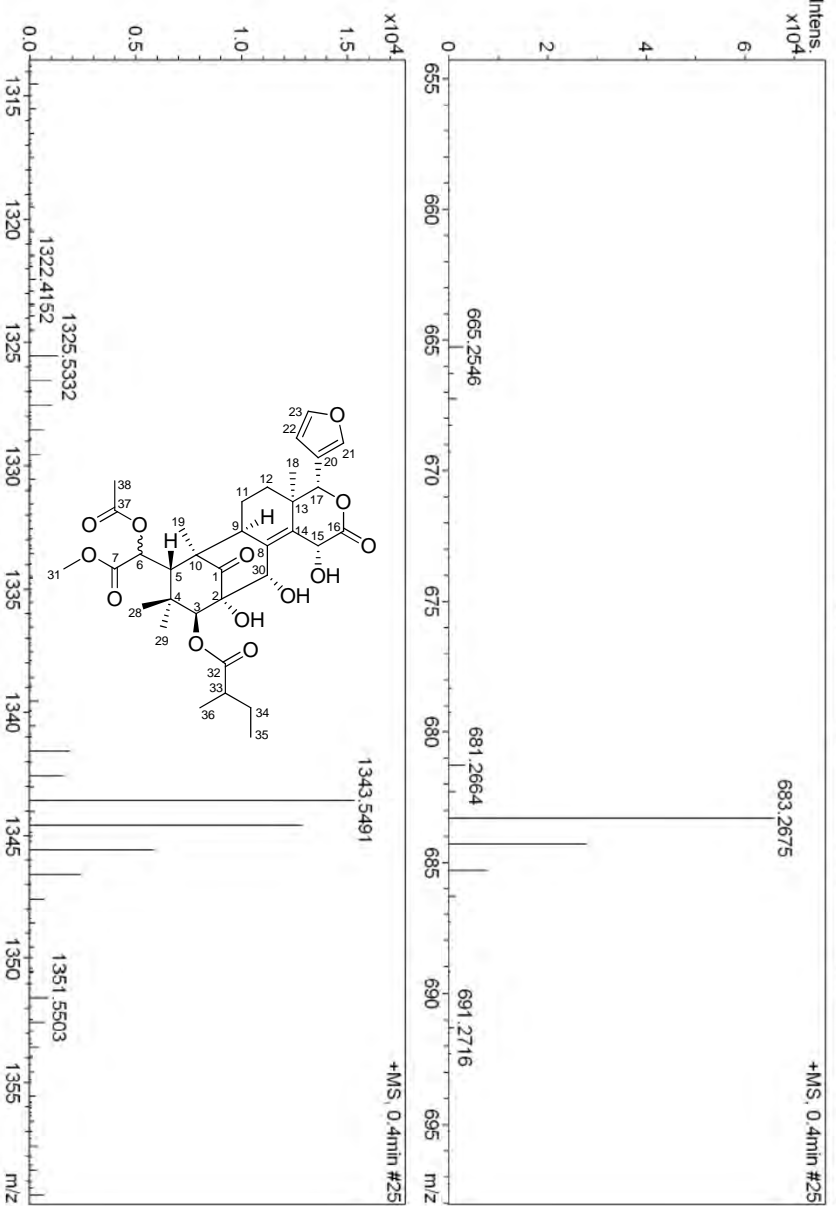
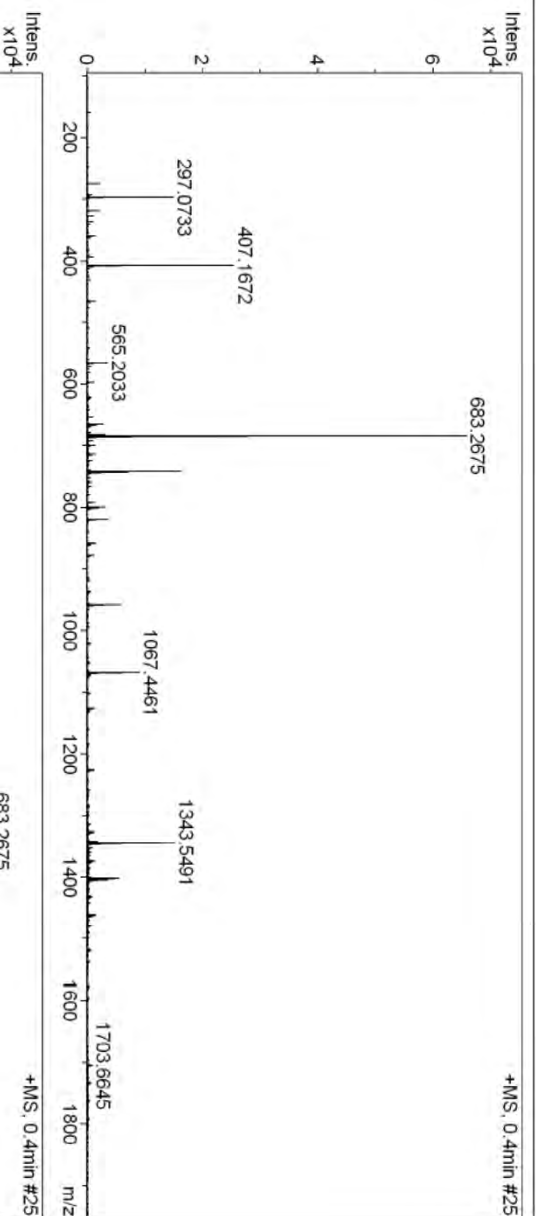


Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e <sup>-</sup> Conf	N-Rule
683.2675	1	C 34 H 44 Na O 13	100.00	683.2674	-0.1	-0.1	24.3	12.5	even	ok
1343.5491	1	C 68 H 88 Na O 26	8.87	1343.5456	-2.6	-3.5	48.9	24.5	even	ok

## Generic Display Report

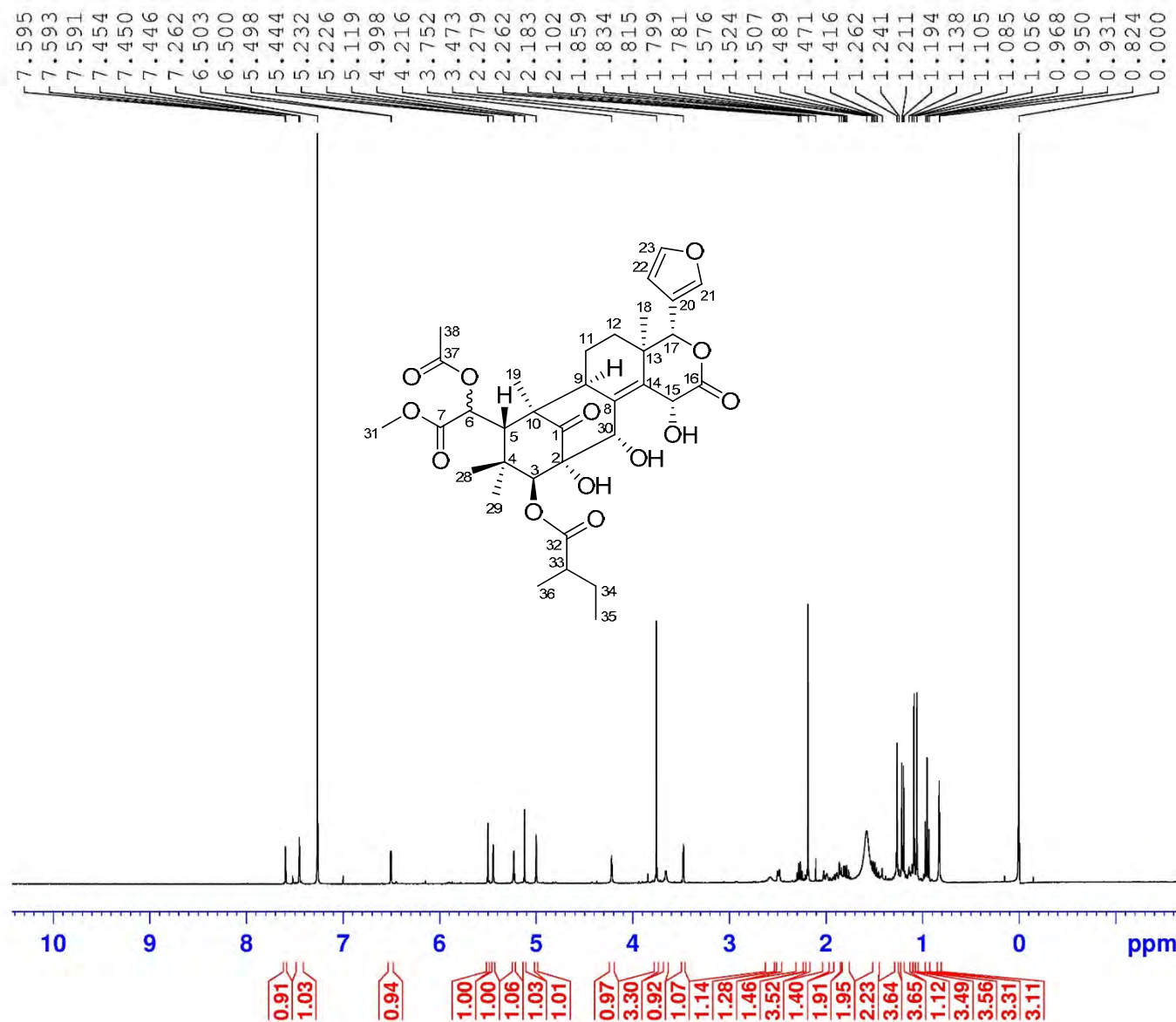
### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-91\_pos\_11\_01\_1858.d Acquisition Date 5/19/2016 8:51:32 AM  
Method LC\_Direct Infusion\_pos\_100-1000mz.m Operator SCSIO  
Sample Name liwanshan\_lws-91\_pos Instrument maxis  
Comment





$^1\text{H}$  NMR (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



```

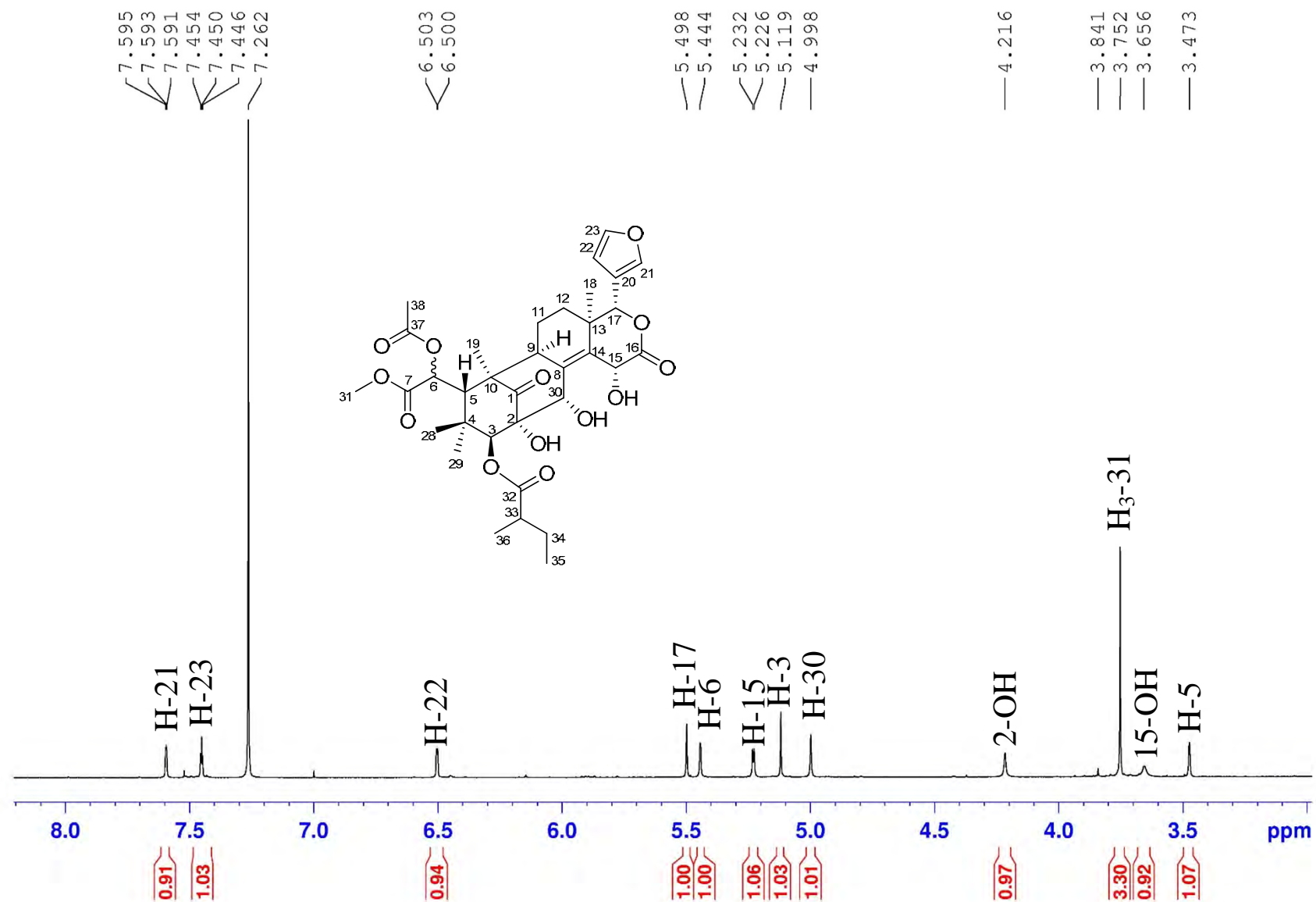
NAME          lws-93
EXPNO          1
PROCNO         1
Date_          20160204
Time           16.55
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        zg30
TD             65536
SOLVENT        CDCl3
NS             16
DS             2
SWH            8223.685 F
FIDRES         0.125483 F
AQ            3.9846387 s
RG            208.5
DW            60.800 u
DE            10.00 u
TE            297.0 K
D1            1.00000000 s
TD0           1
  
```

```

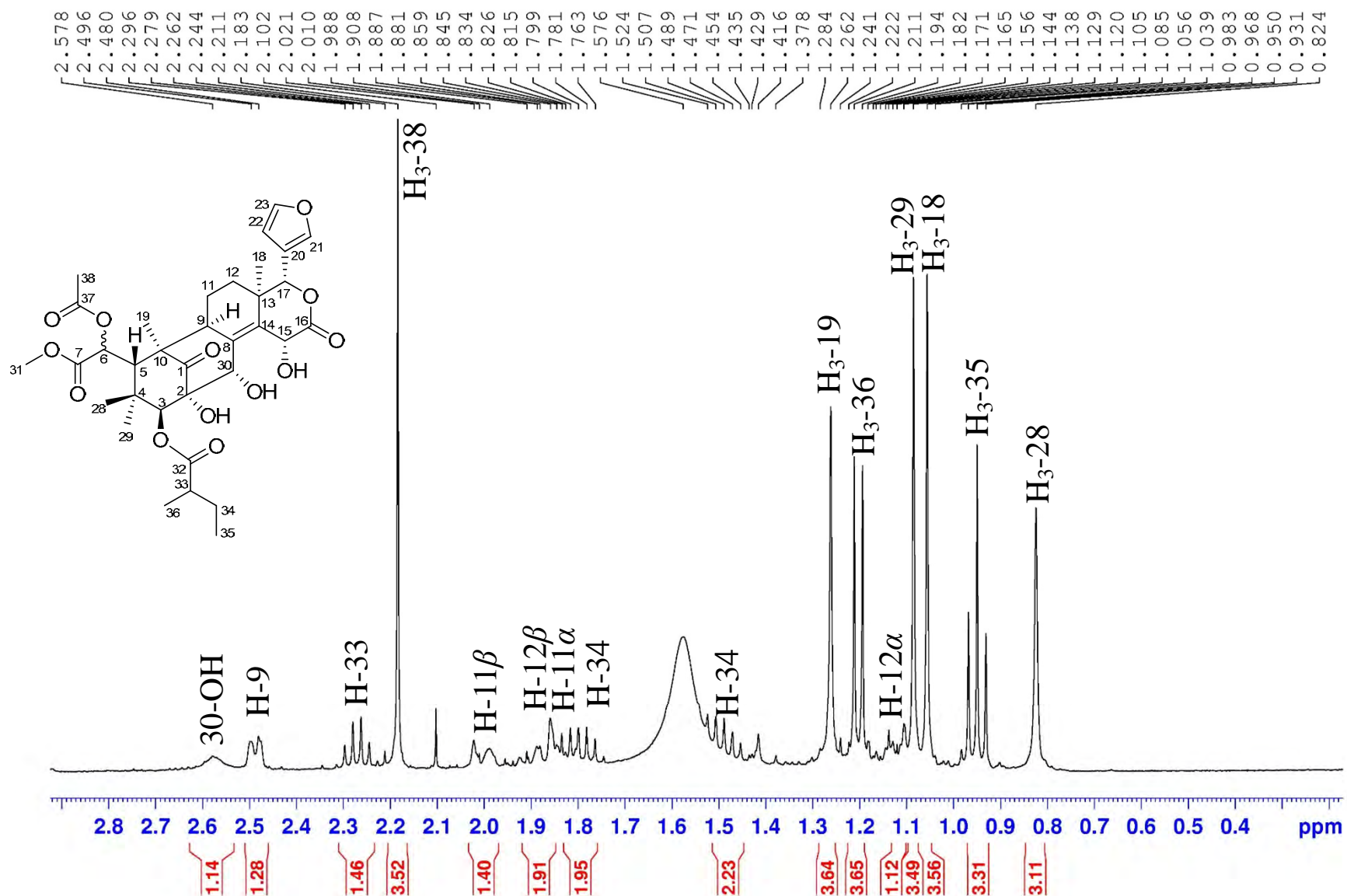
===== CHANNEL f1 =====
SFO1          400.1324710 M
NUC1           1H
P1            11.50 u
SI            65536
SF            400.1300090 M
WDW            EM
SSB            0
LB            0.30 F
GB            0
PC            1.00
  
```



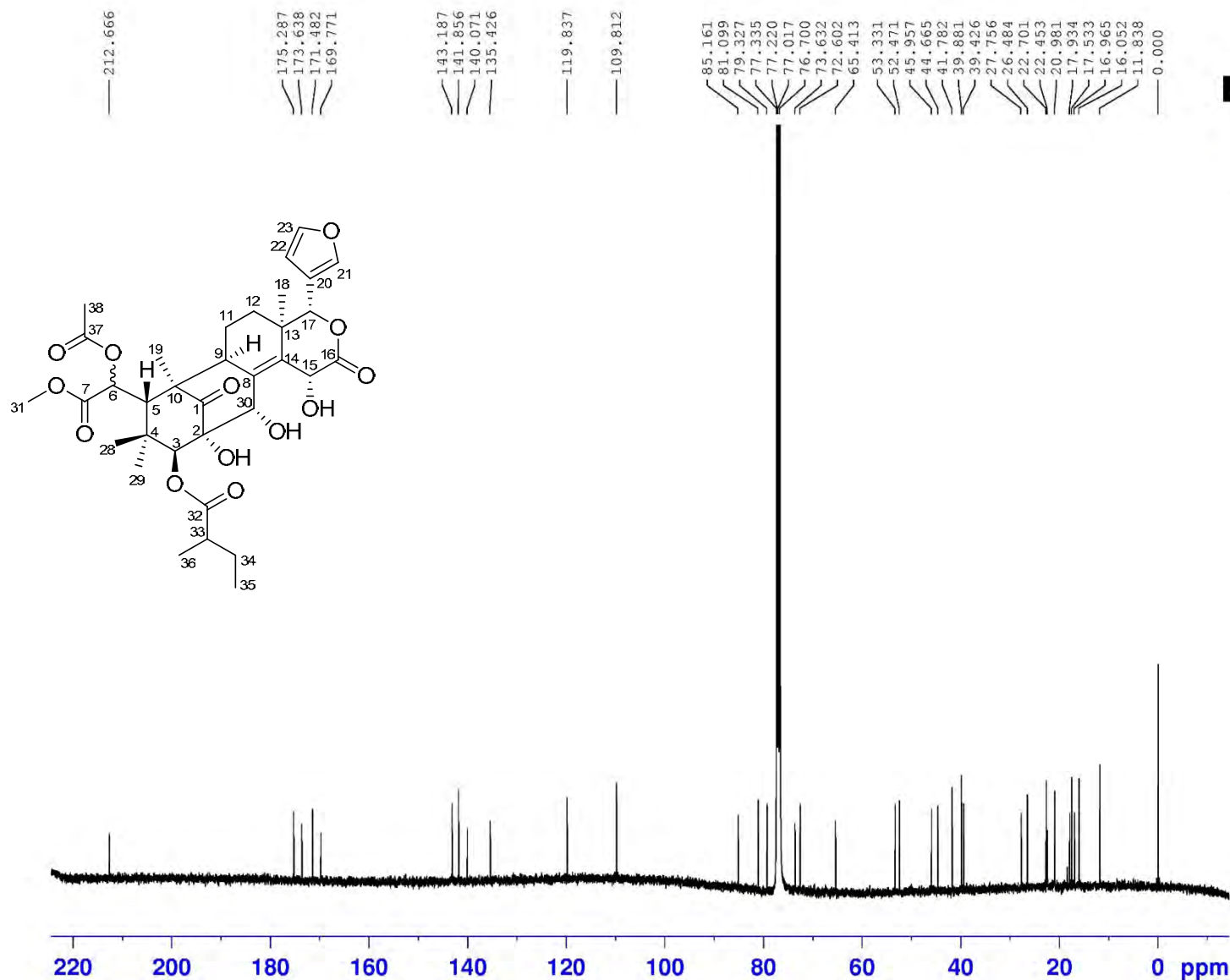
$^1\text{H}$  NMR (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



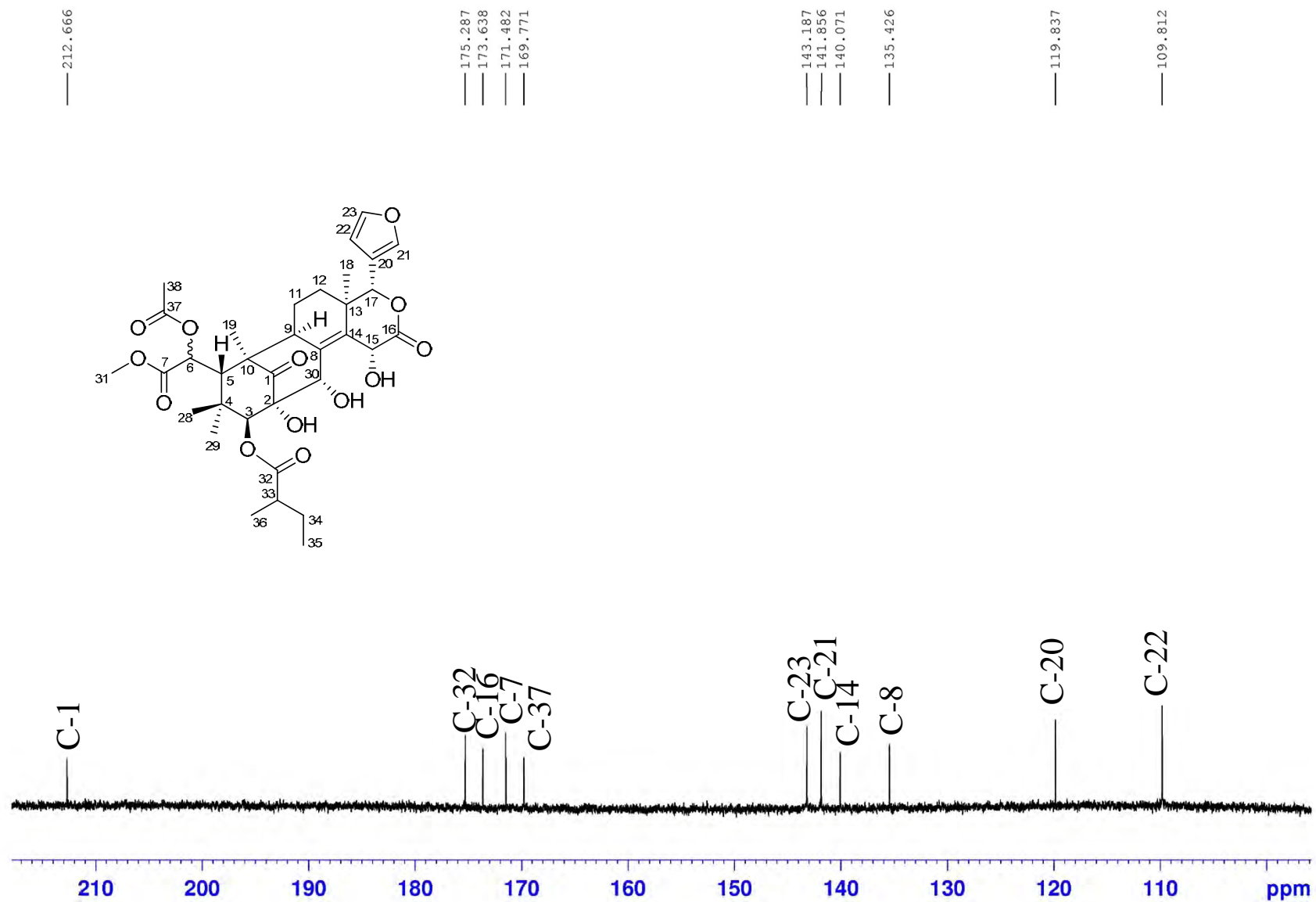
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



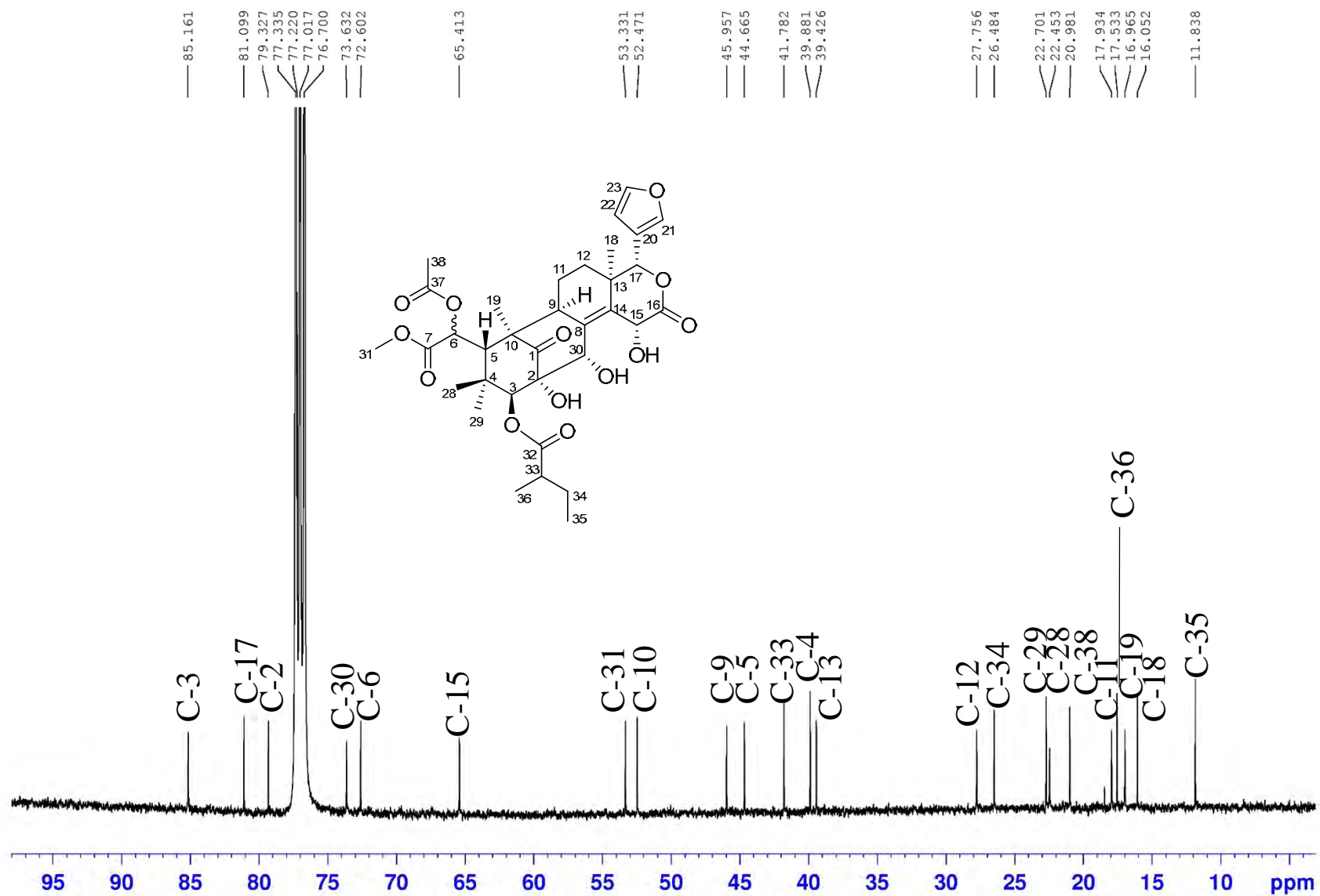
NAME lws-93  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20160205  
 Time 12.24  
 INSTRUM spect  
 PROBHD 5 mm CPPBBO BB  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT  $\text{CDCl}_3$   
 NS 20000  
 DS 4  
 SWH 24038.461 F  
 FIDRES 0.366798 F  
 AQ 1.3631988 s  
 RG 102.3  
 DW 20.800  $\mu$   
 DE 18.00  $\mu$   
 TE 297.0 K  
 D1 2.00000000 s  
 D11 0.03000000 s  
 TD0 1

===== CHANNEL f1 =====  
 SFO1 100.623324 M  
 NUC1  $^{13}\text{C}$   
 P1 10.00  $\mu$   
 SI 32768  
 SF 100.6127692 M  
 WDW EM  
 SSB 0  
 LB 1.00 F  
 GB 0  
 PC 1.40

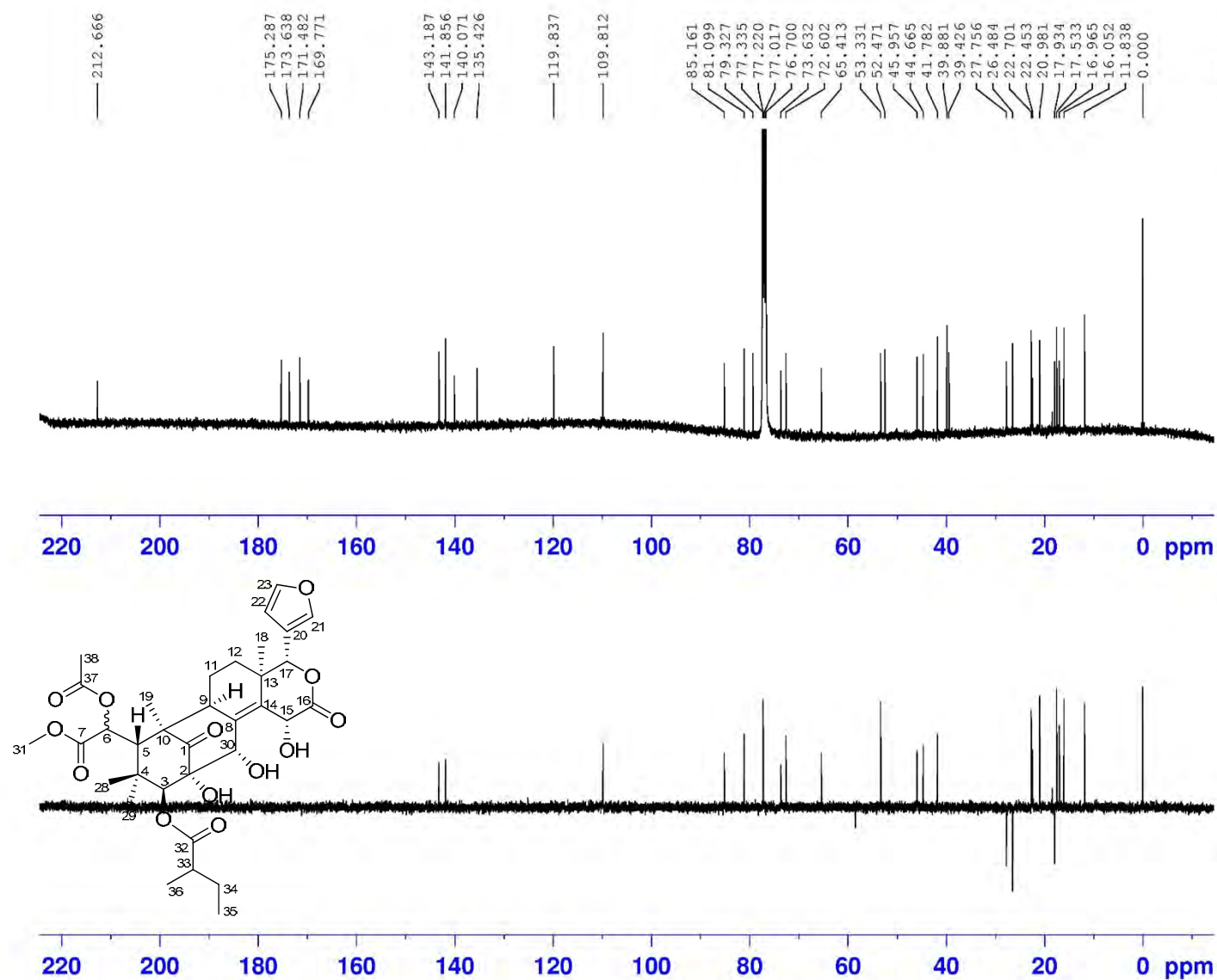
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



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



# DEPT135 (100 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



```

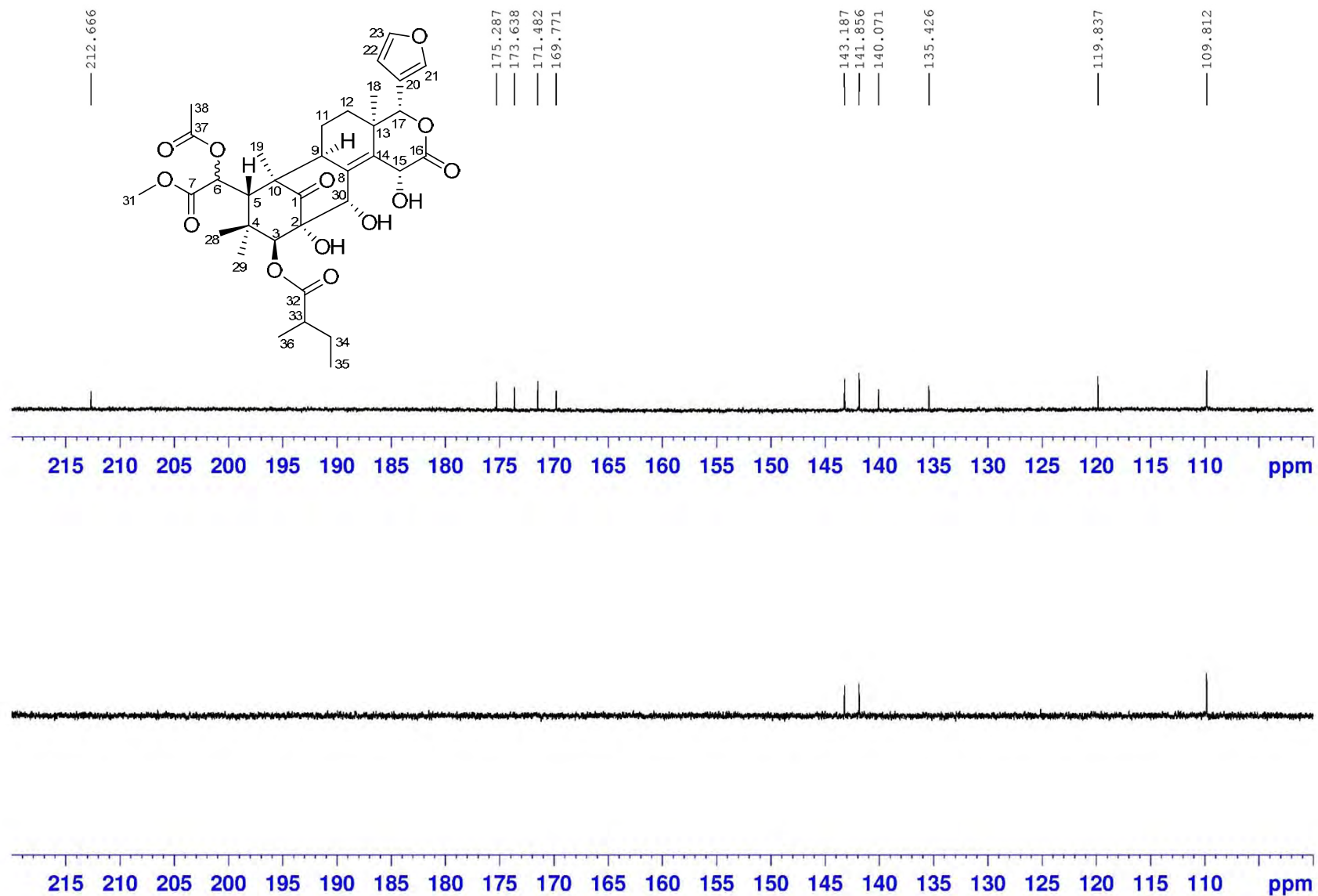
NAME          lws-93
EXPNO          3
PROCNO         1
Date_          20160205
Time           17.09
INSTRUM        spect
PROBHD         5 mm CPMBO BB
PULPROG        deptspl35
TD             65536
SOLVENT        CDCl3
NS             5000
DS             4
SWH            24038.461 F
FIDRES         0.366798 F
AQ            1.3631988 s
RG             130.26
DW            20.800 s
DE            18.00 s
TE            297.0 F
CNST2          145.0000000
D1            2.00000000 s
D2            0.00344828 s
D12           0.00002000 s
TD0           1
  
```

```

===== CHANNEL f1 =====
SFO1          100.6233324 M
NUC1           13C
P1            10.00 s
P13           2000.00 s
SI            32768
SF            100.6127690 M
WDW            EM
SSB            0
LB            1.00 F
GB            0
PC            1.40
  
```

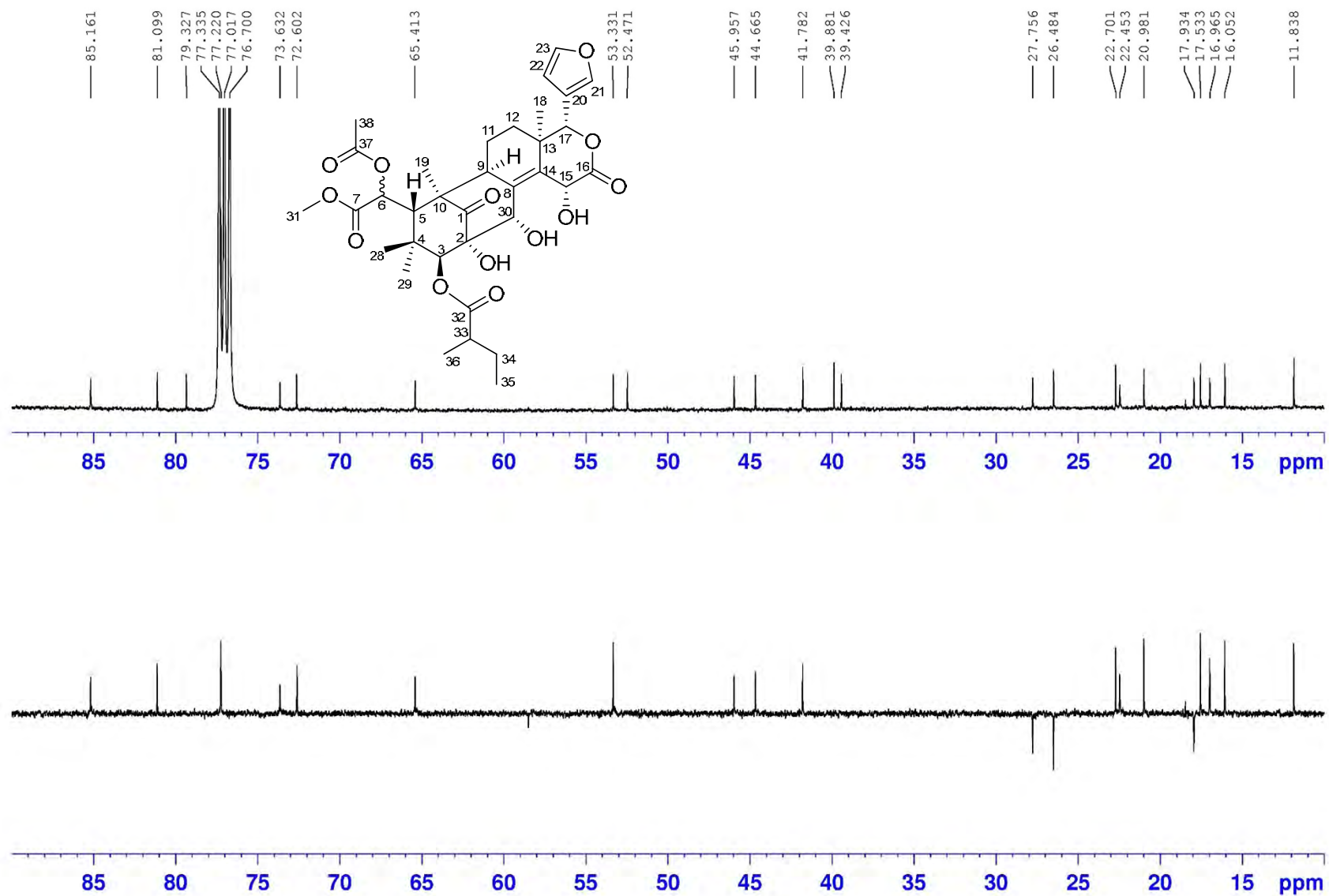


# DEPT135 (100 MHz) spectrum of compound **4** in CDCl<sub>3</sub>

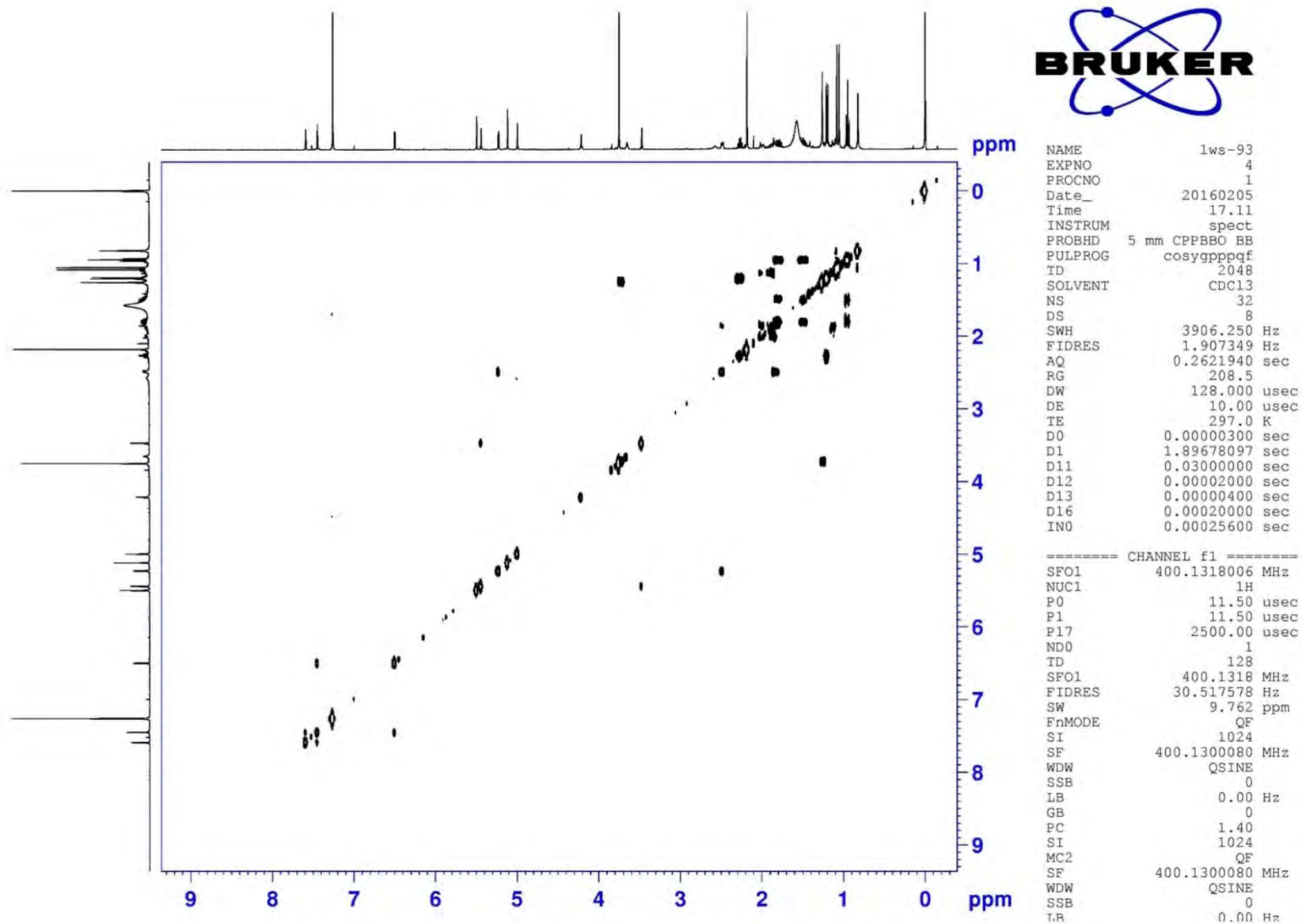




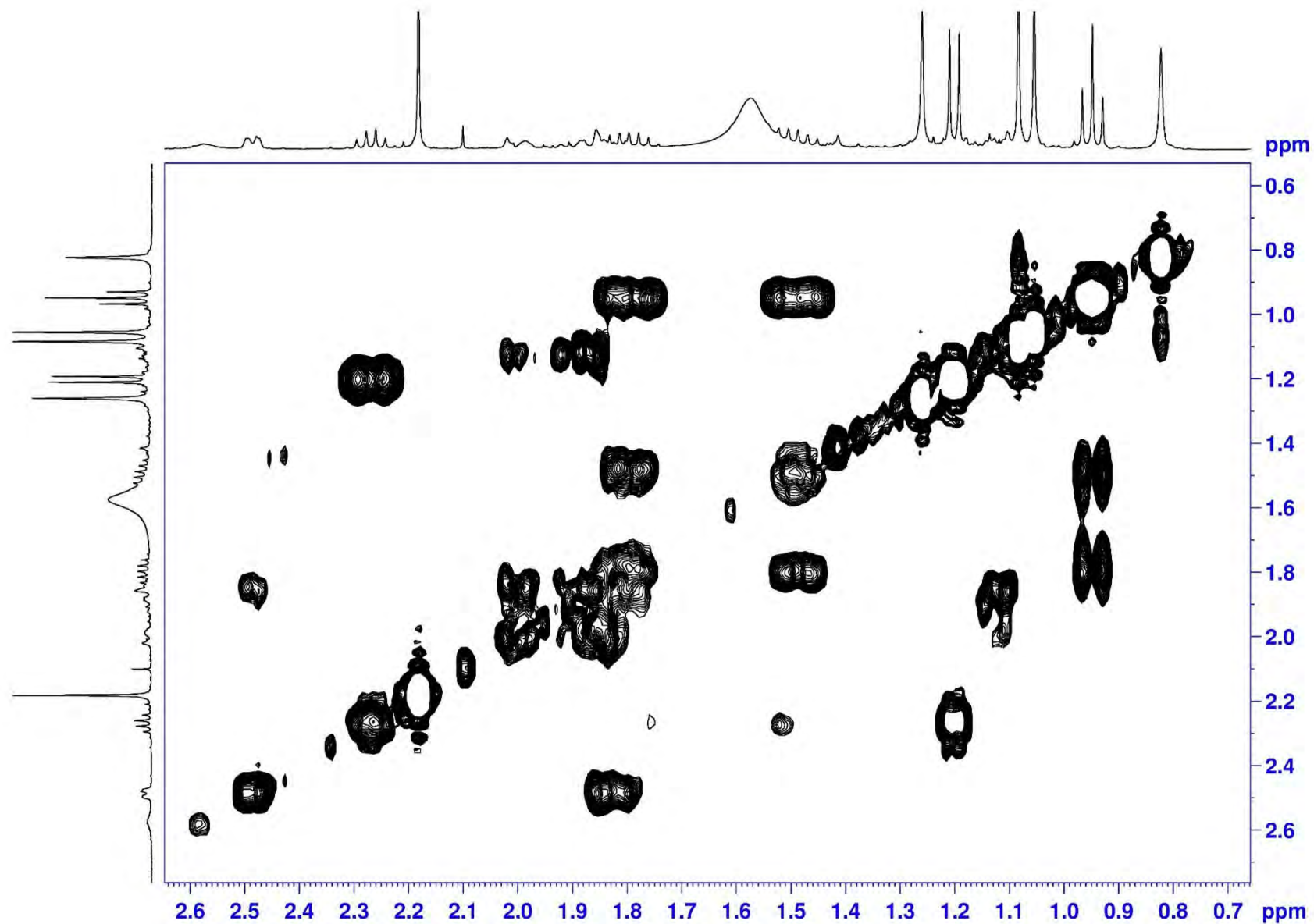
DEPT135 (100 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



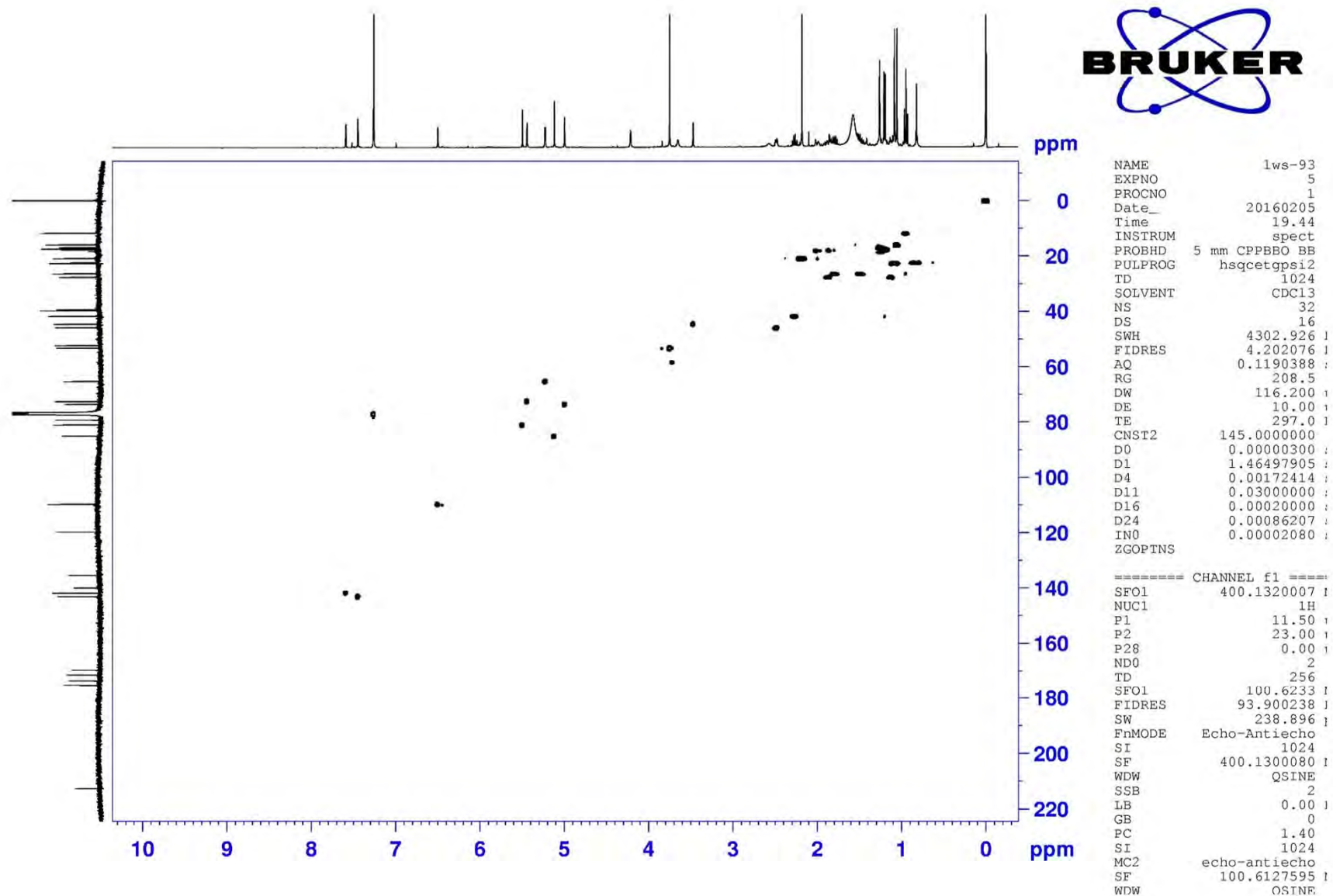
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



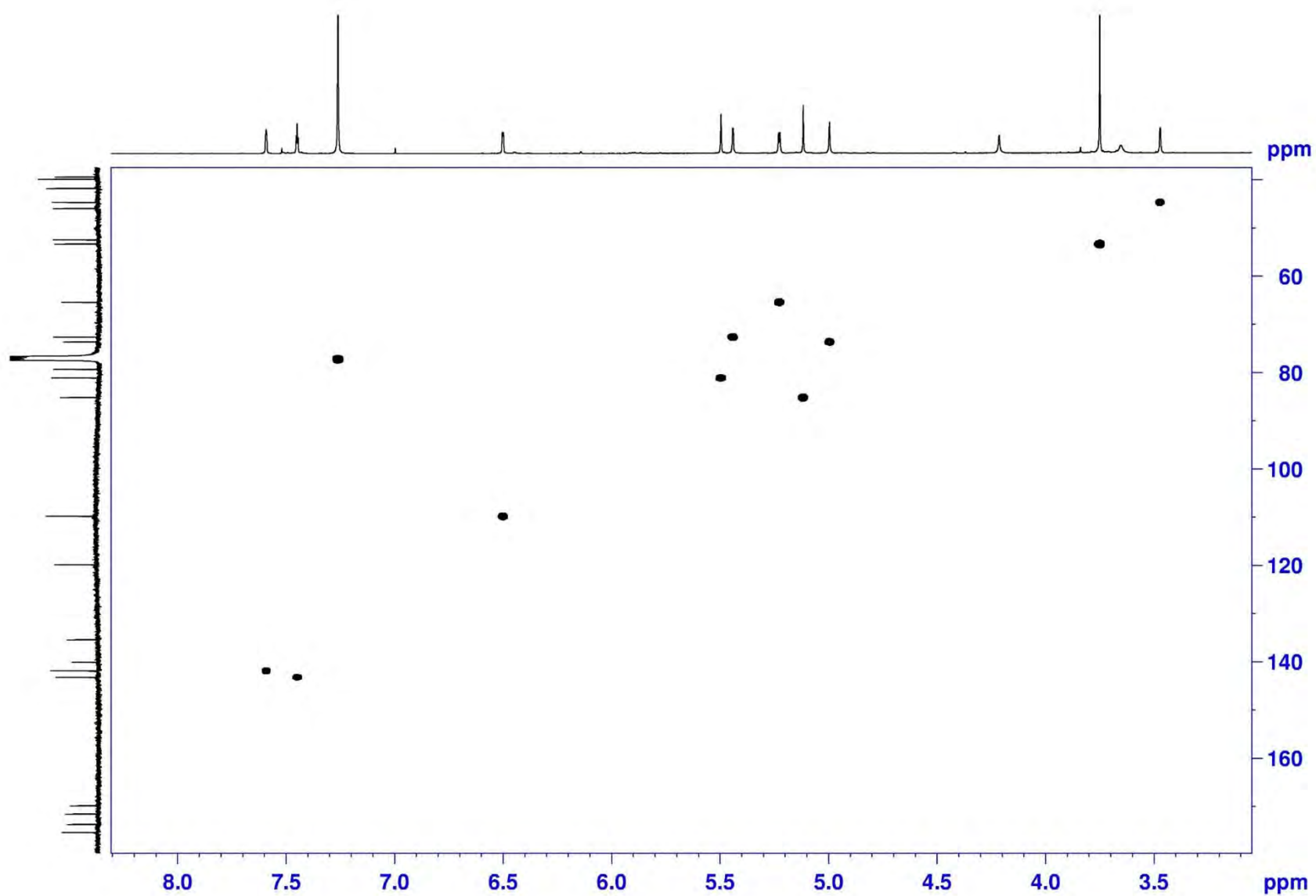
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



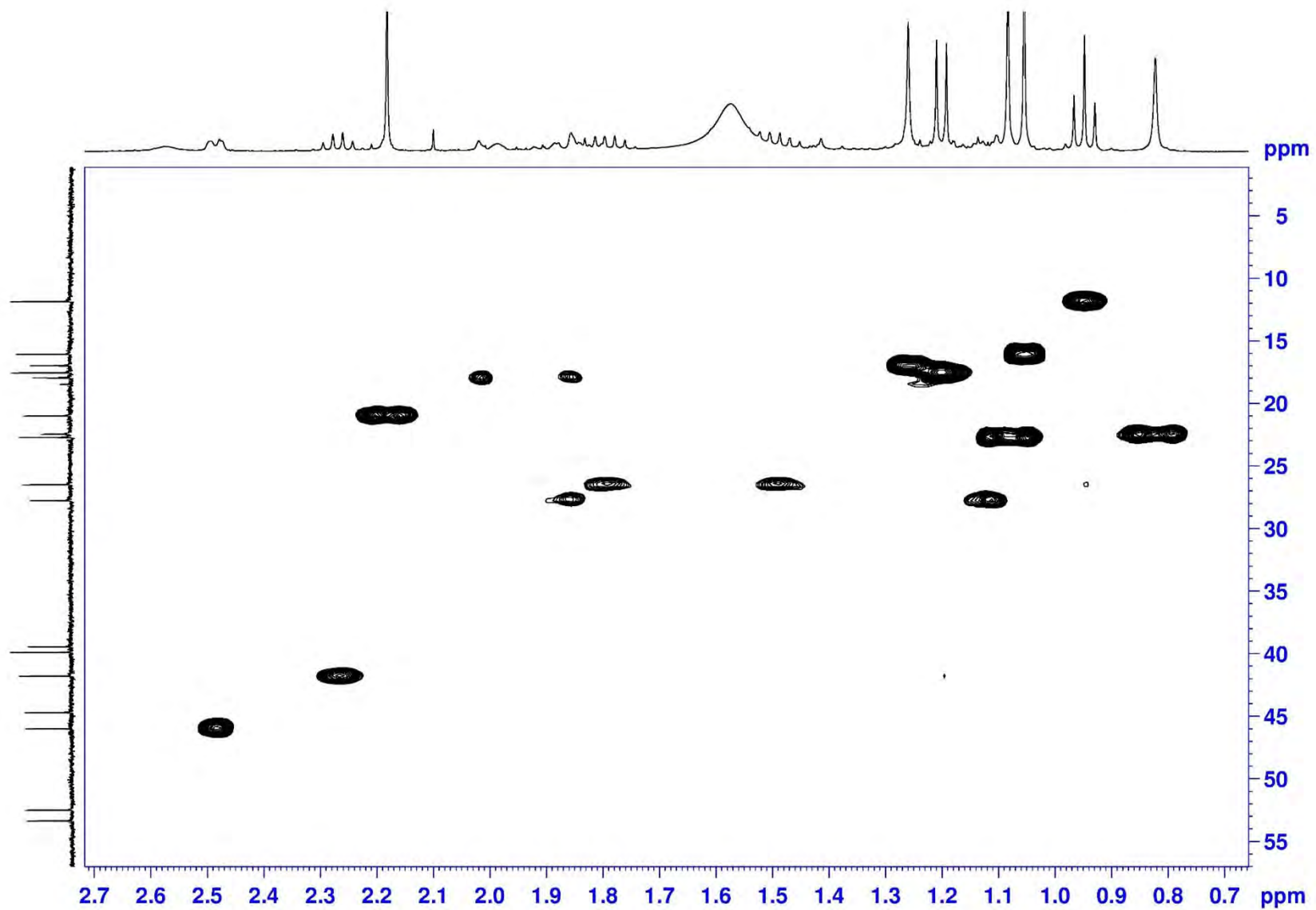
# HSQC (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



HSQC (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$

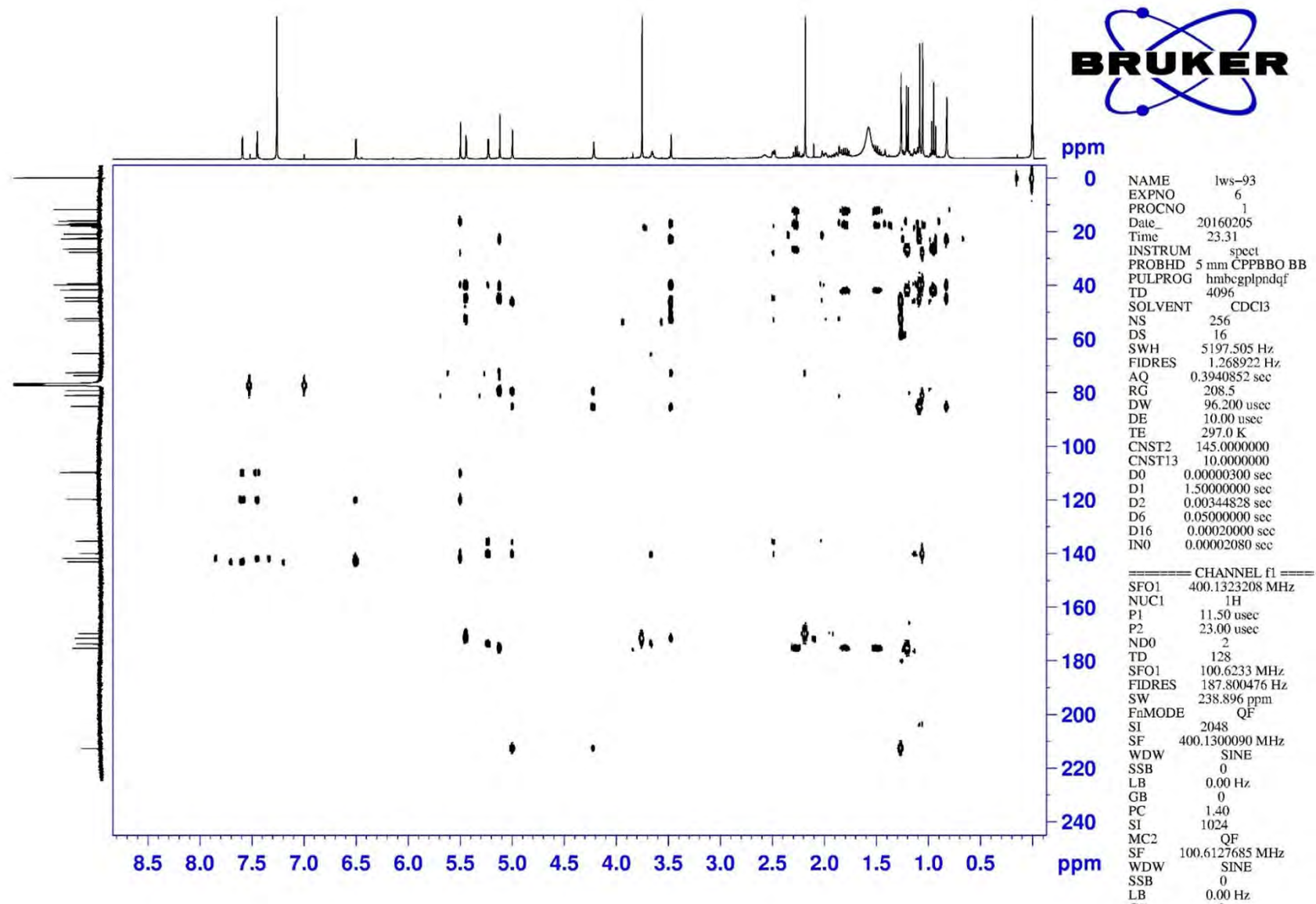


HSQC (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



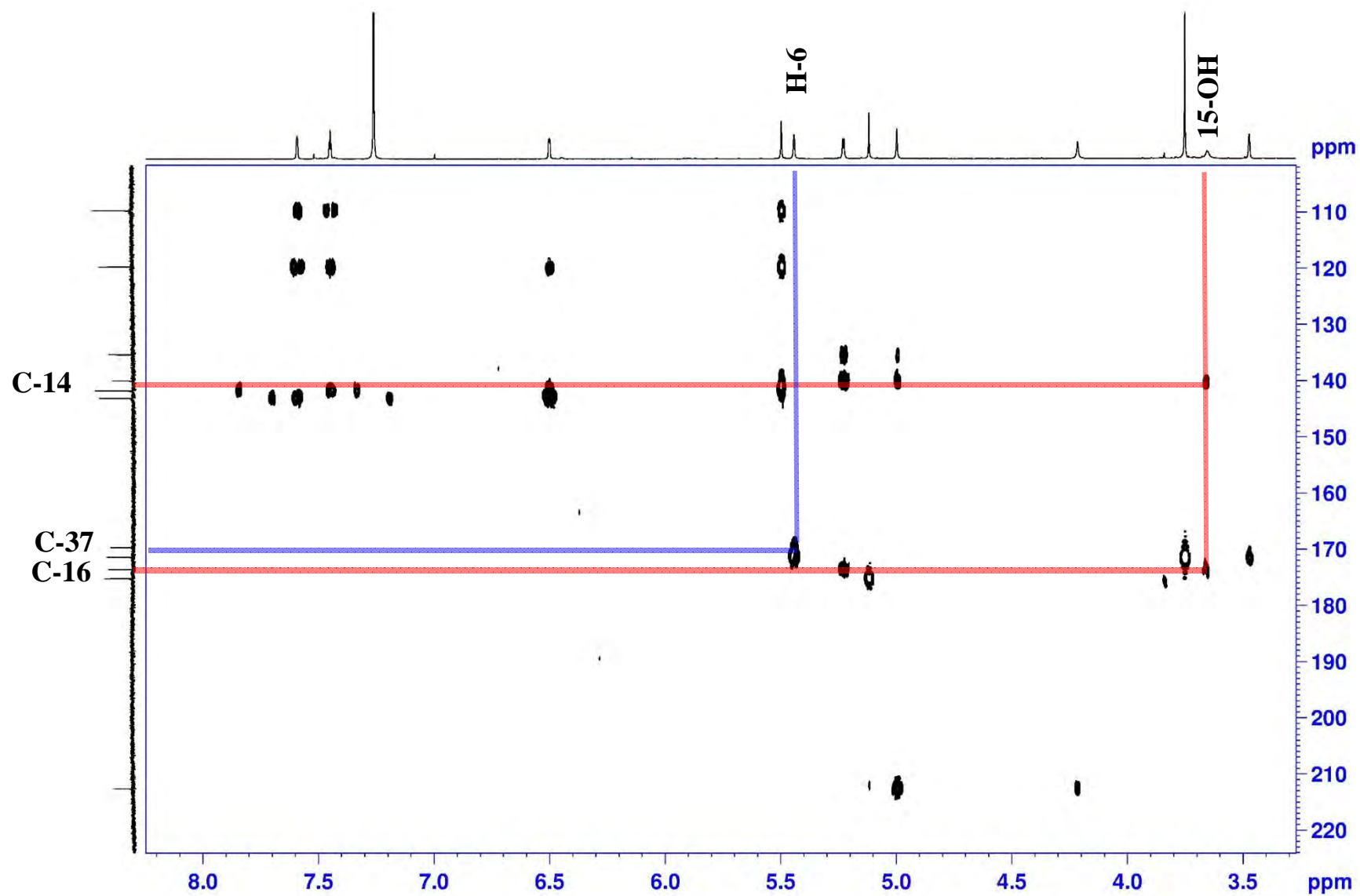


# HMBC (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>

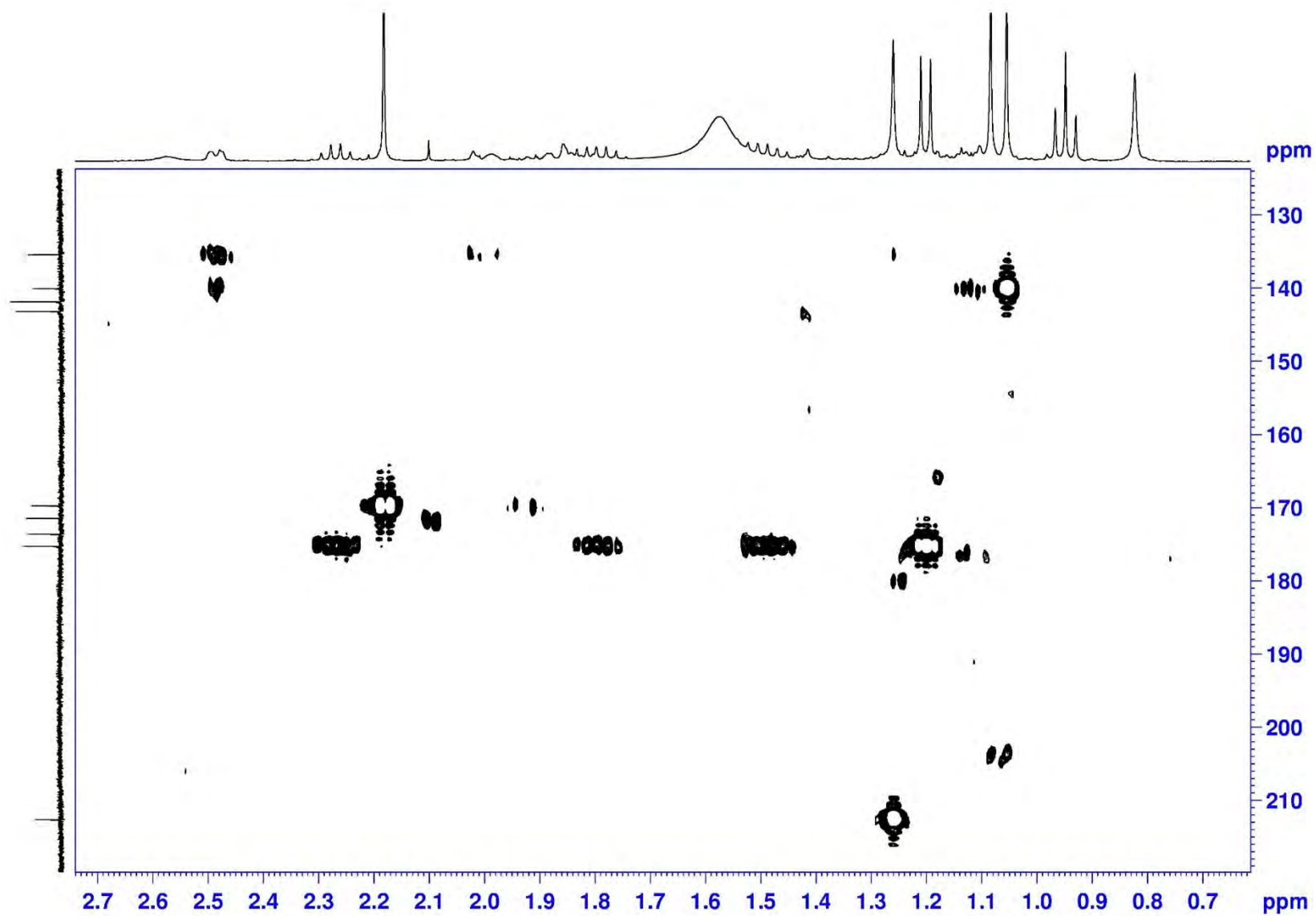




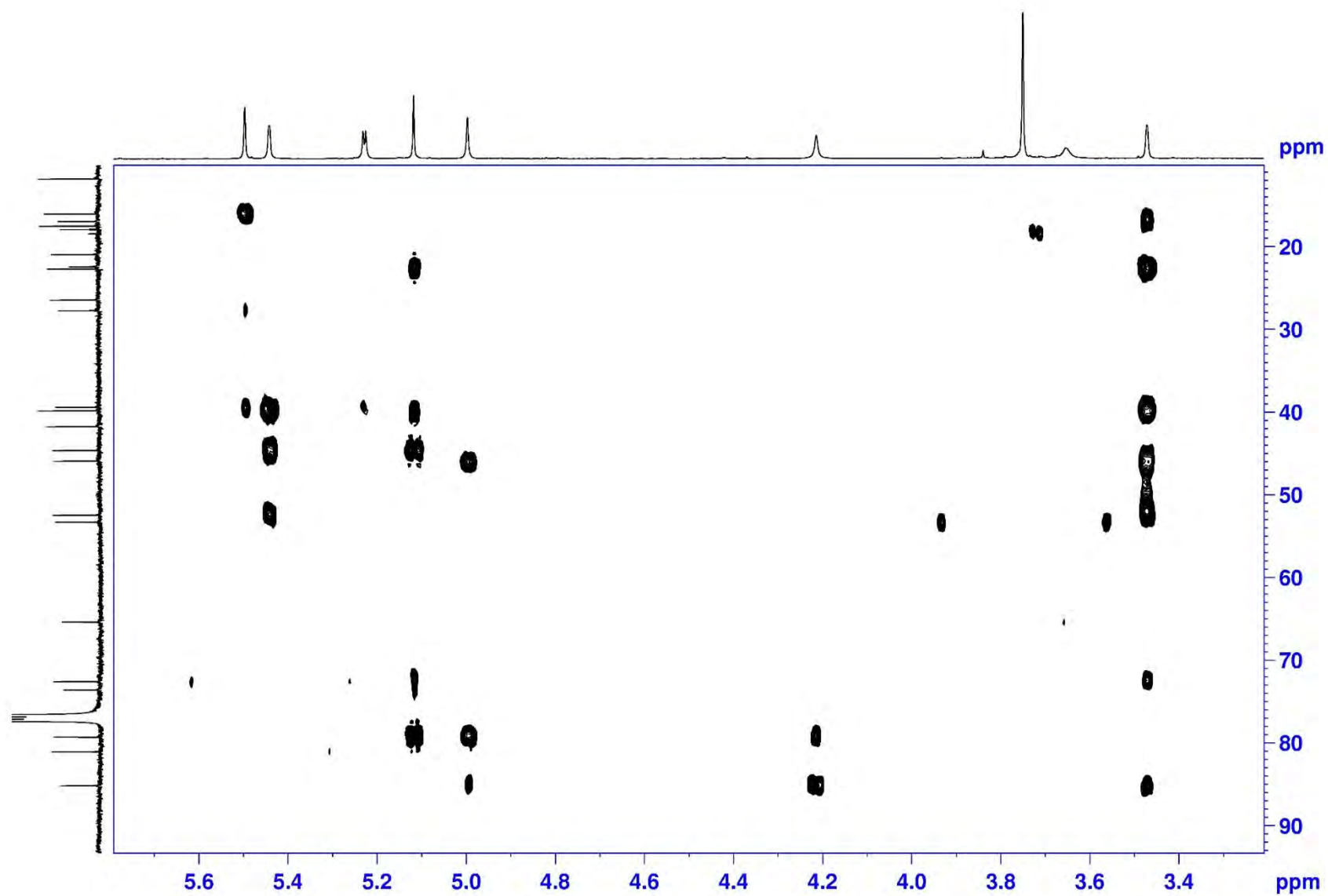
HMBC (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



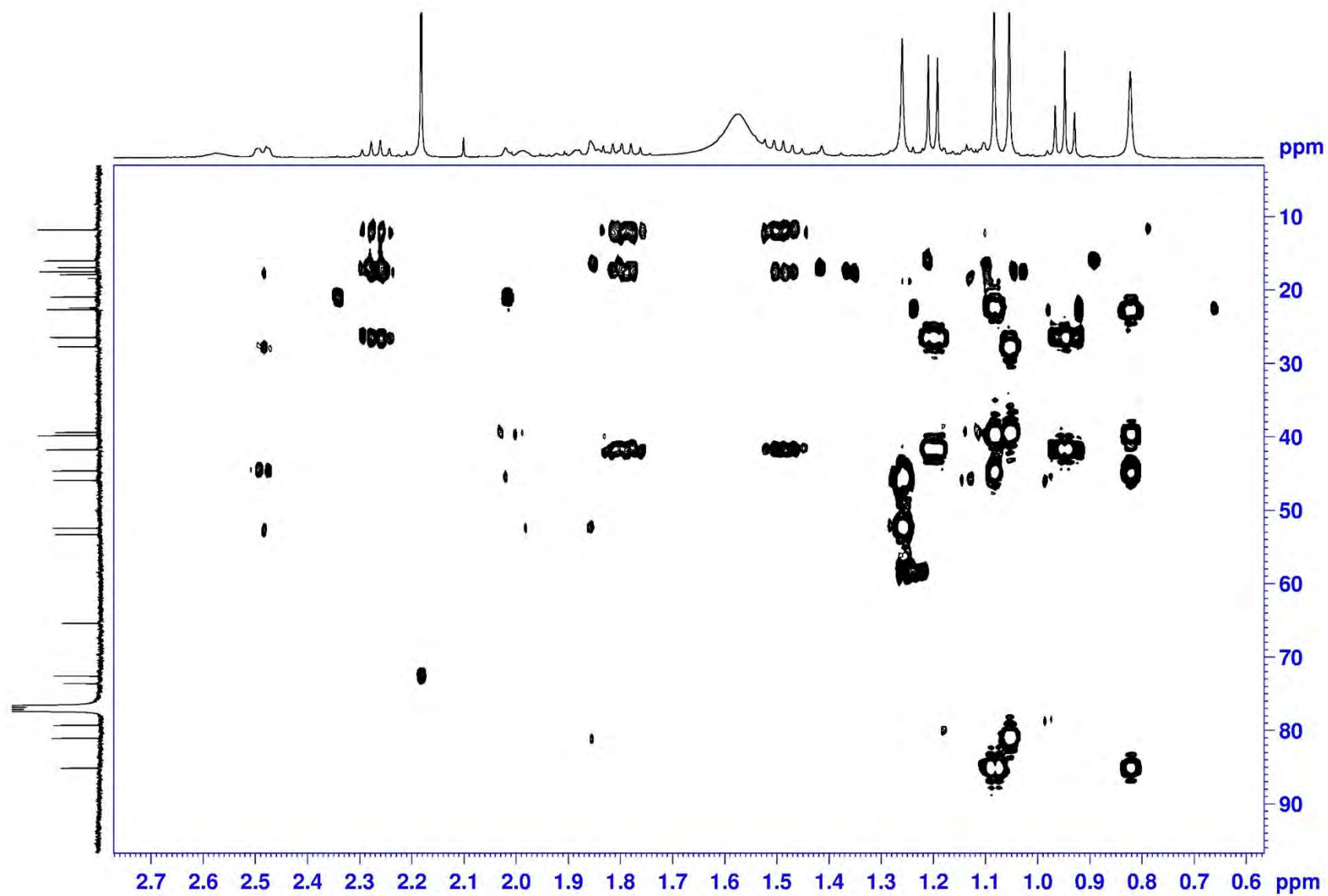
HMBC (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



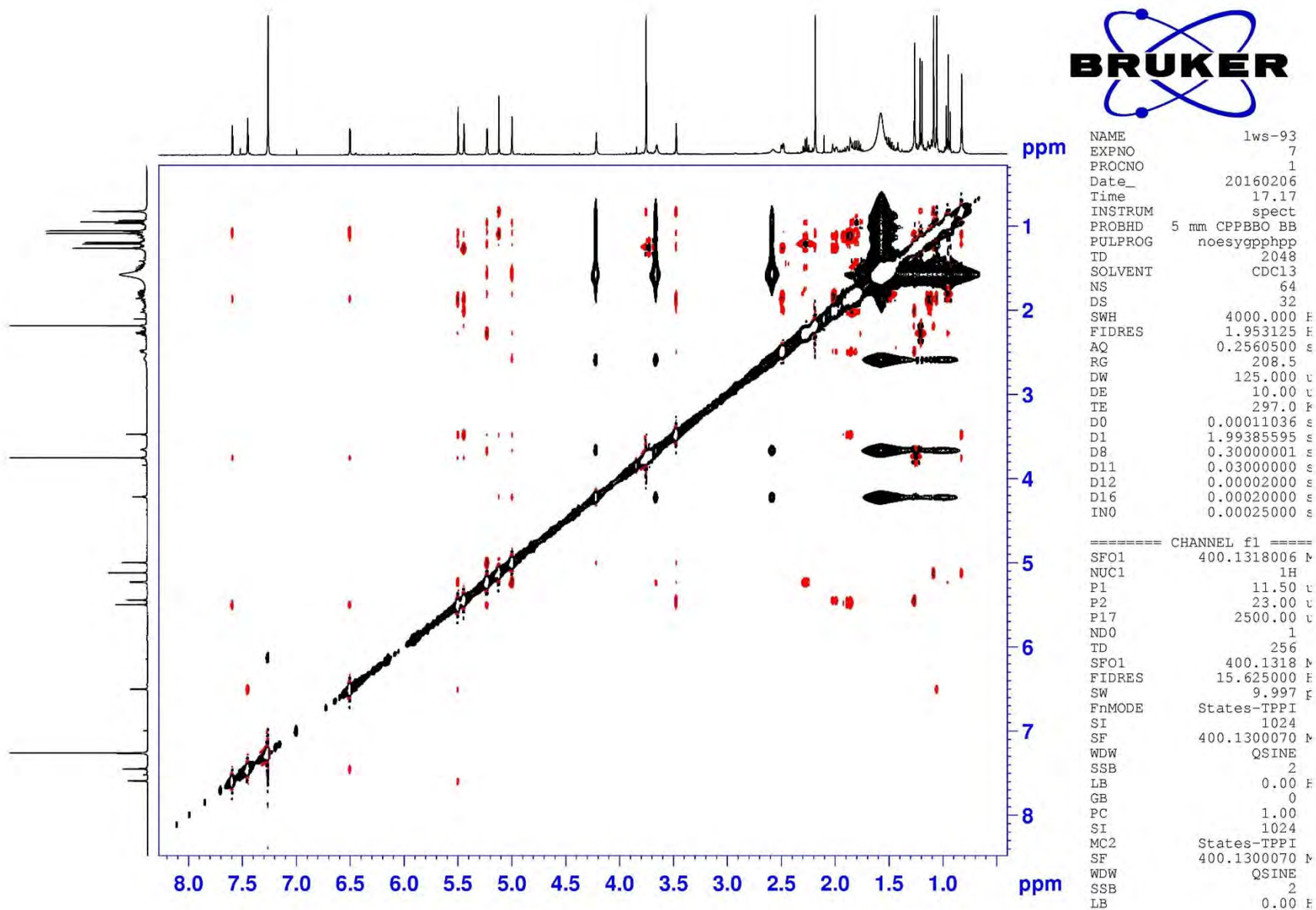
HMBC (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$

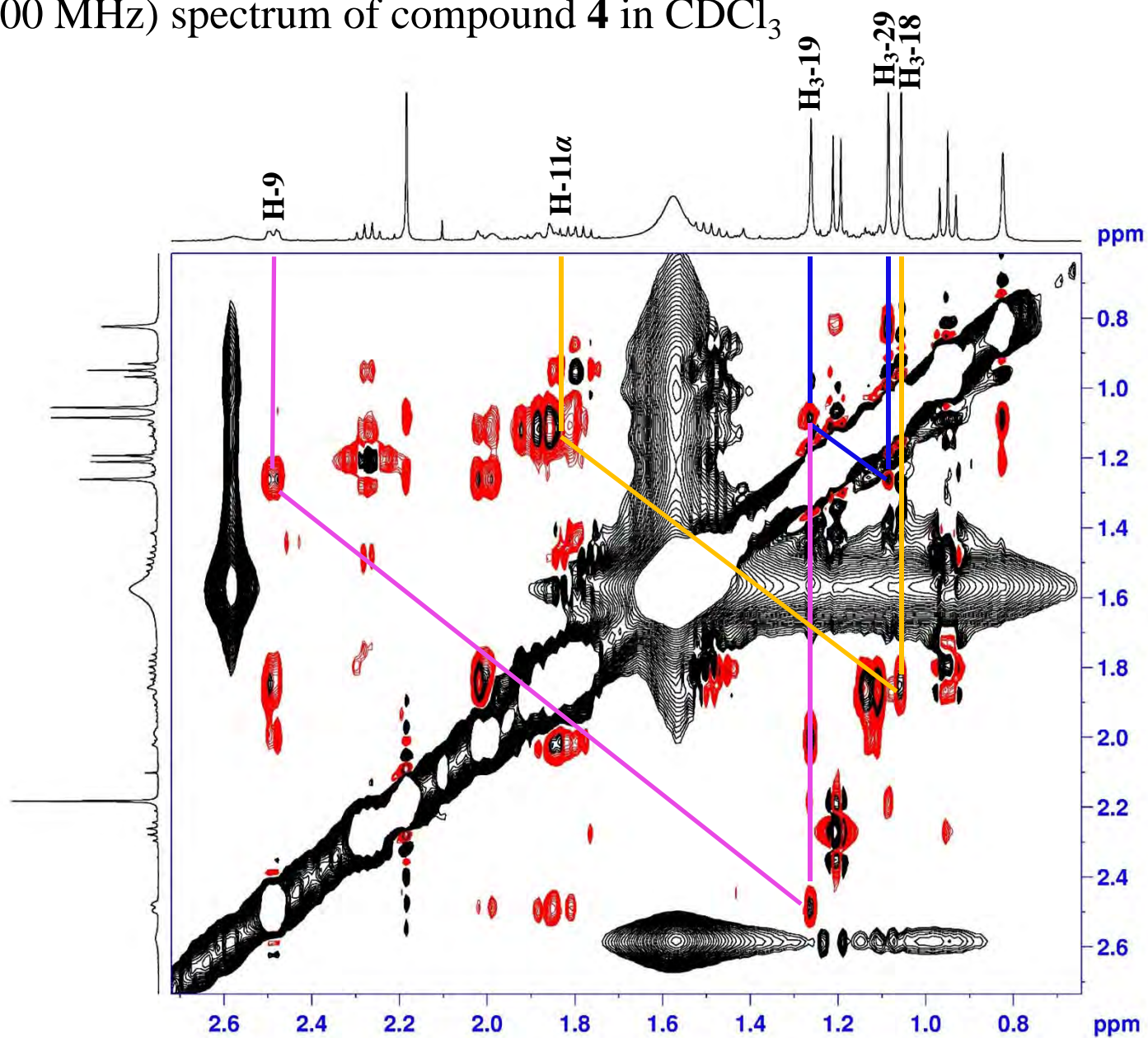


# NOESY (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>

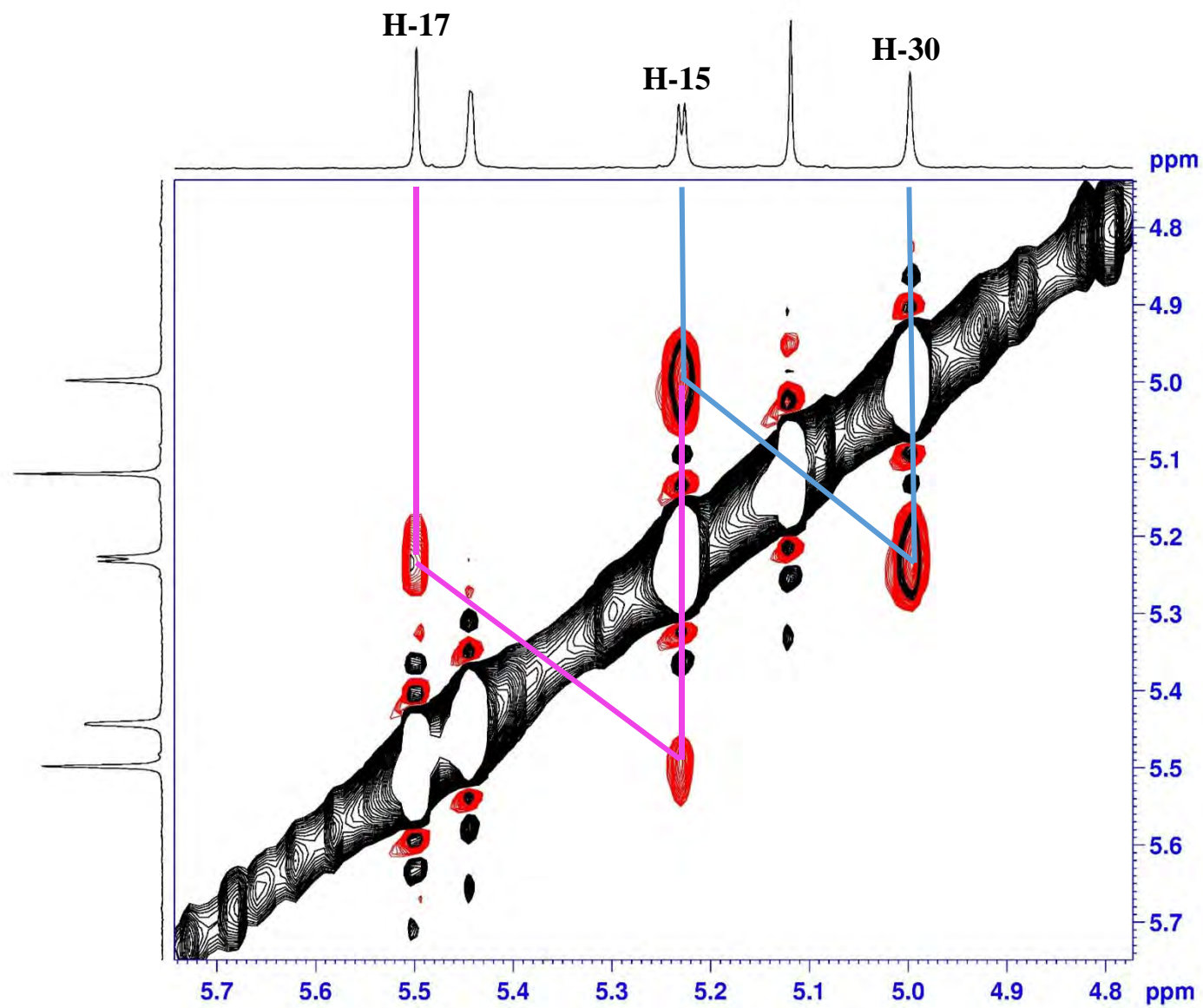




NOESY (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$

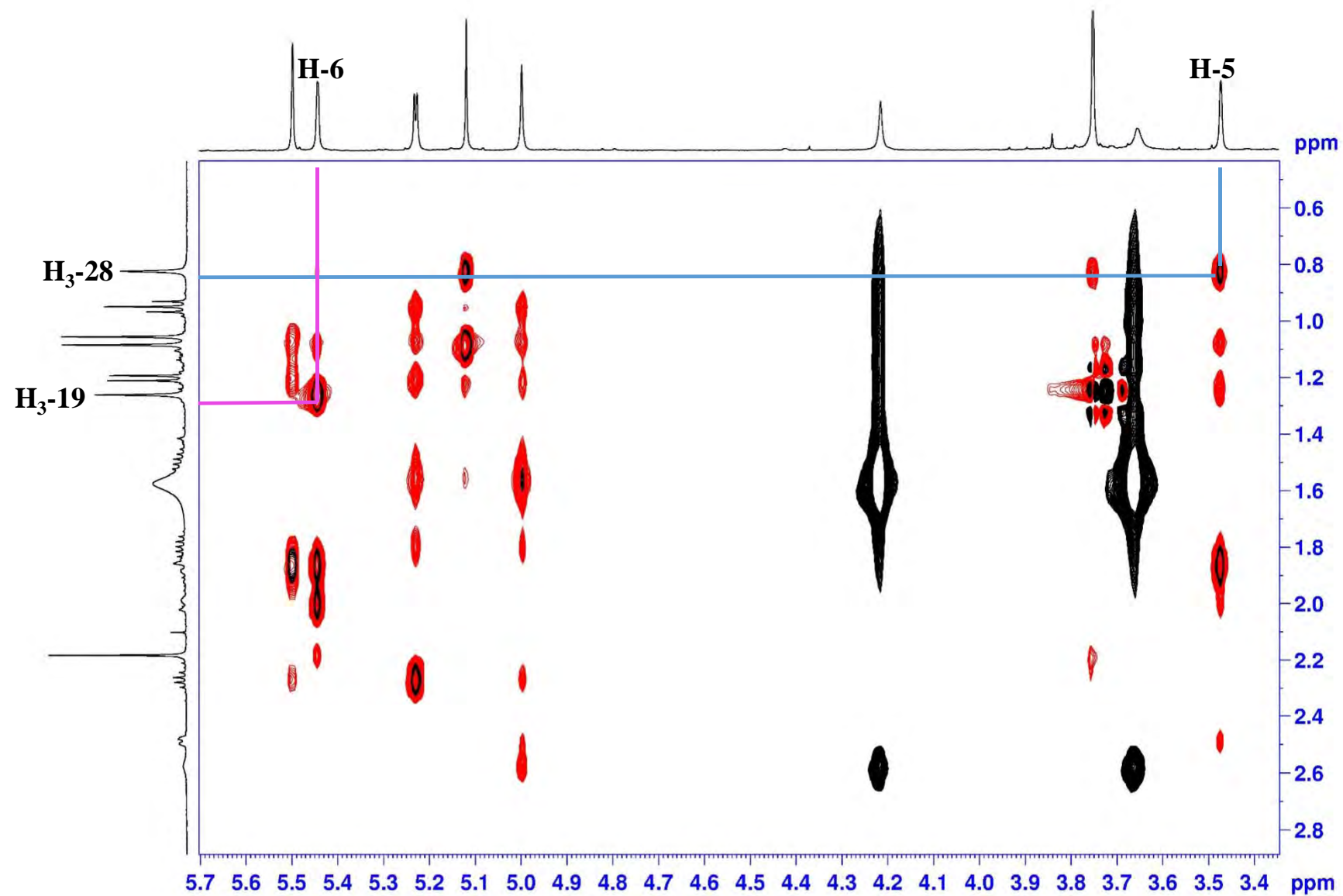


NOESY (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$

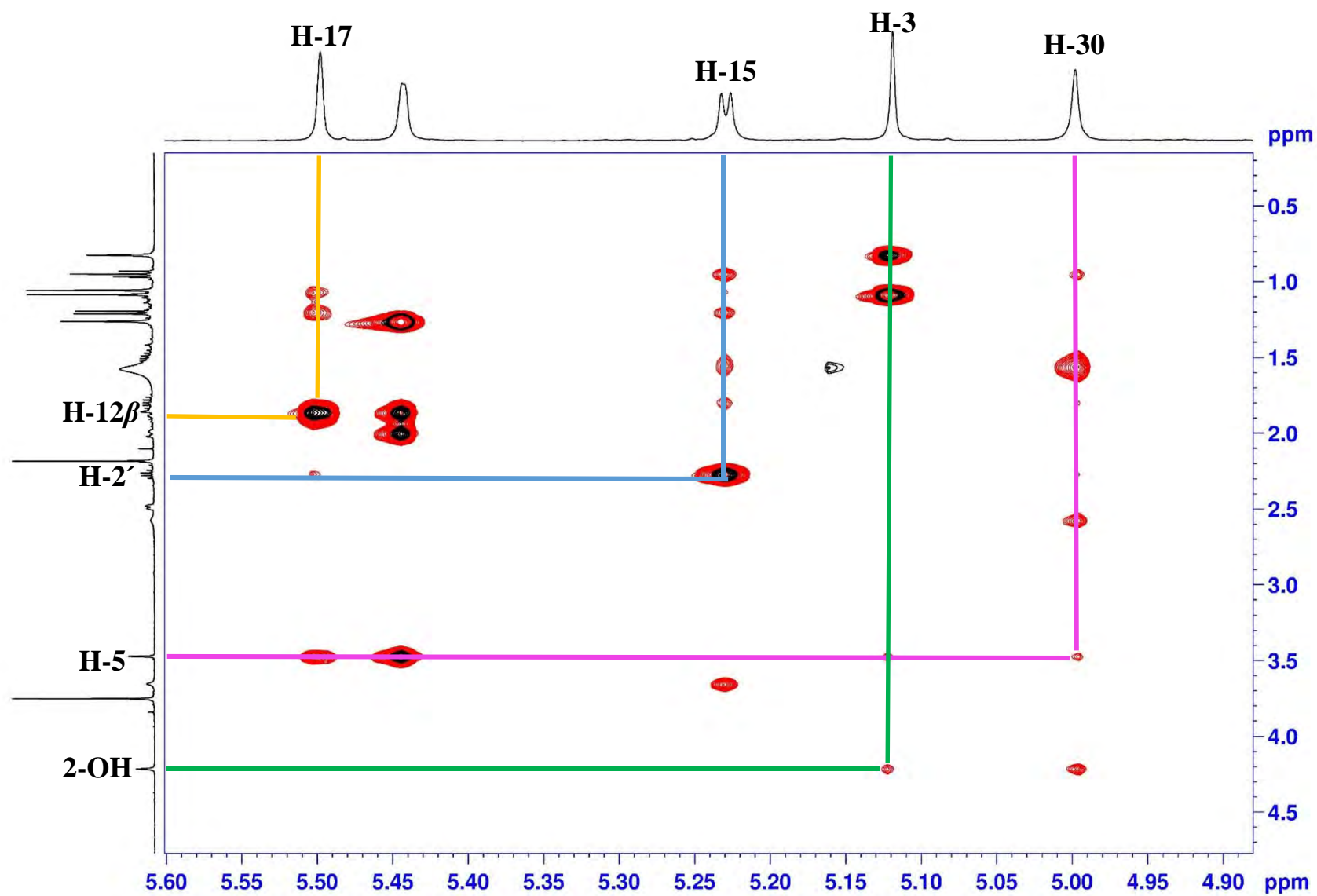




NOESY (400 MHz) spectrum of compound **4** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **4** in CDCl<sub>3</sub>



# HR-ESIMS for compound 5

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-65\_pos\_15\_01\_1862.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-65\_pos  
 Comment

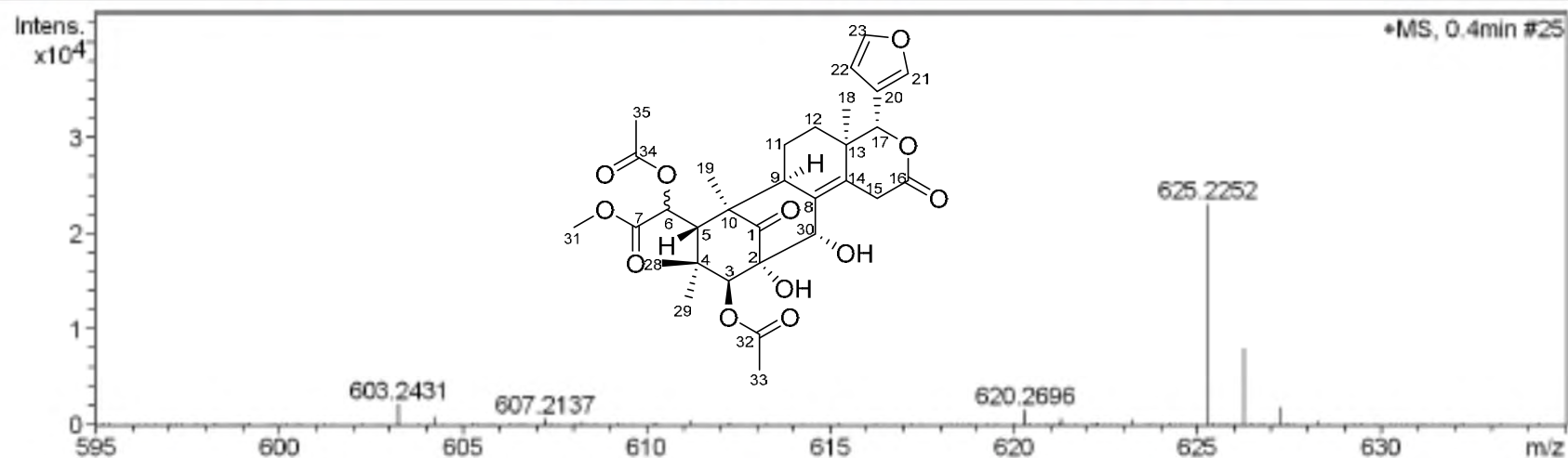
Acquisition Date 5/19/2016 9:05:13 AM

Operator SCSIO

Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste

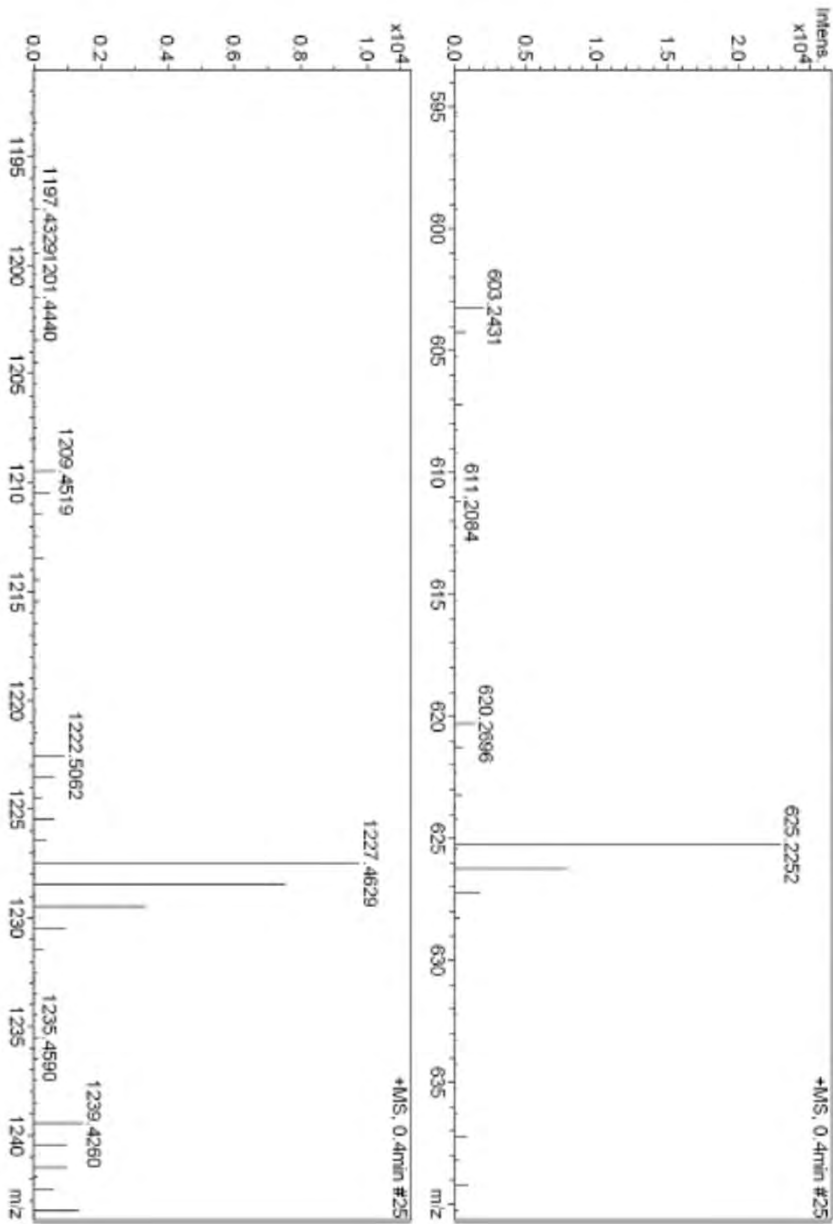
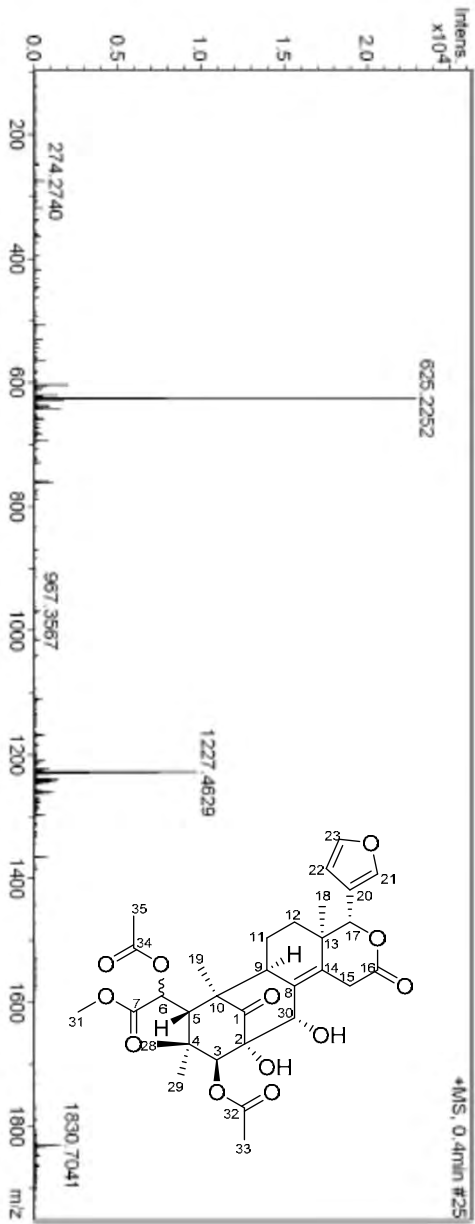


Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
603.2431	1	C 31 H 39 O 12	100.00	603.2436	0.9	0.5	18.0	12.5	even	ok
625.2252	1	C 31 H 38 Na O 12	100.00	625.2255	0.5	0.3	2.4	12.5	even	ok
1227.4629	1	C 62 H 76 Na O 24	90.59	1227.4619	-0.8	-1.0	45.6	24.5	even	ok

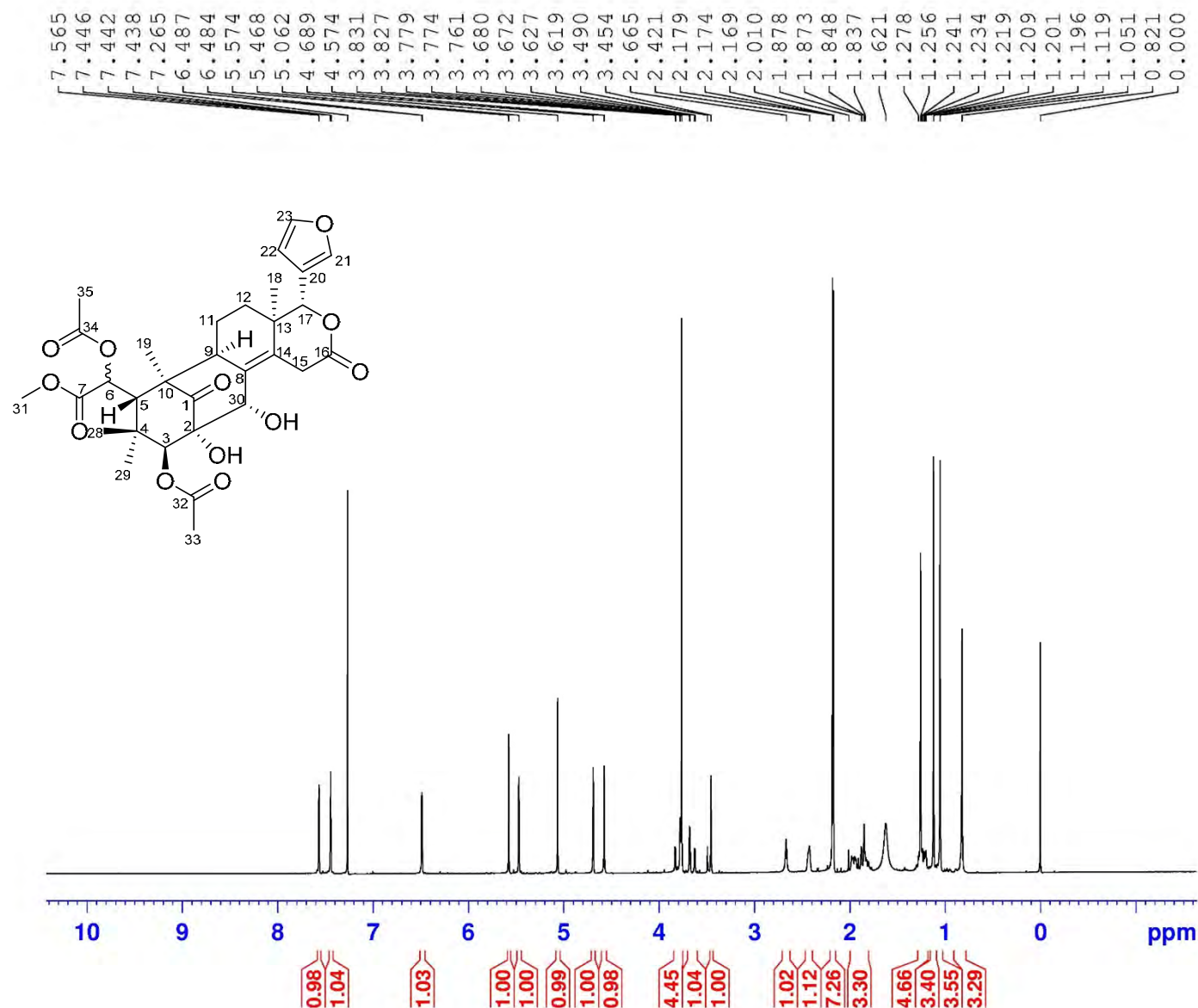
HR-ESIMS for compound 5

Generic Display Report

Analysis Info		Acquisition Date 5/19/2016 9:06:13 AM	
Analysis Name	D:\Data\MS\data\201605\liwanshan_lws-65_pos_15_01_1862.d	Operator	SCSIO
Method	LC_Direct Infusion_pos_100-1000mz.m	Instrument	maxis
Sample Name	liwanshan_lws-65_pos		
Comment			



# <sup>1</sup>H NMR (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



```

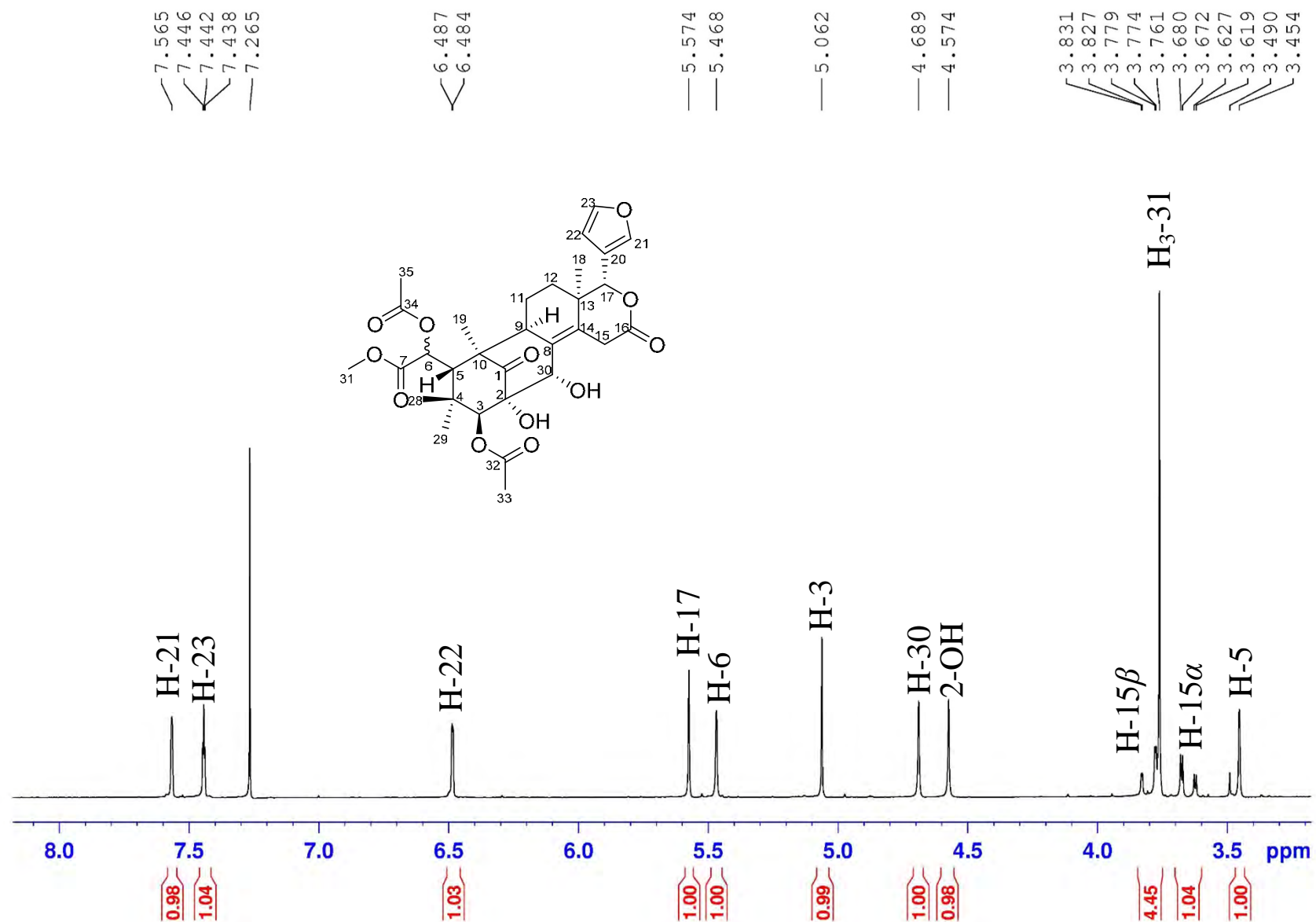
NAME          lws-65
EXPNO          1
PROCNO         1
Date_          20150708
Time           9.14
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        zg30
TD             65536
SOLVENT        CDCl3
NS             16
DS             2
SWH            8223.685 F
FIDRES         0.125483 F
AQ            3.9846387 s
RG            208.5
DW            60.800 u
DE            10.00 u
TE            297.0 K
D1            1.00000000 s
TD0           1
  
```

```

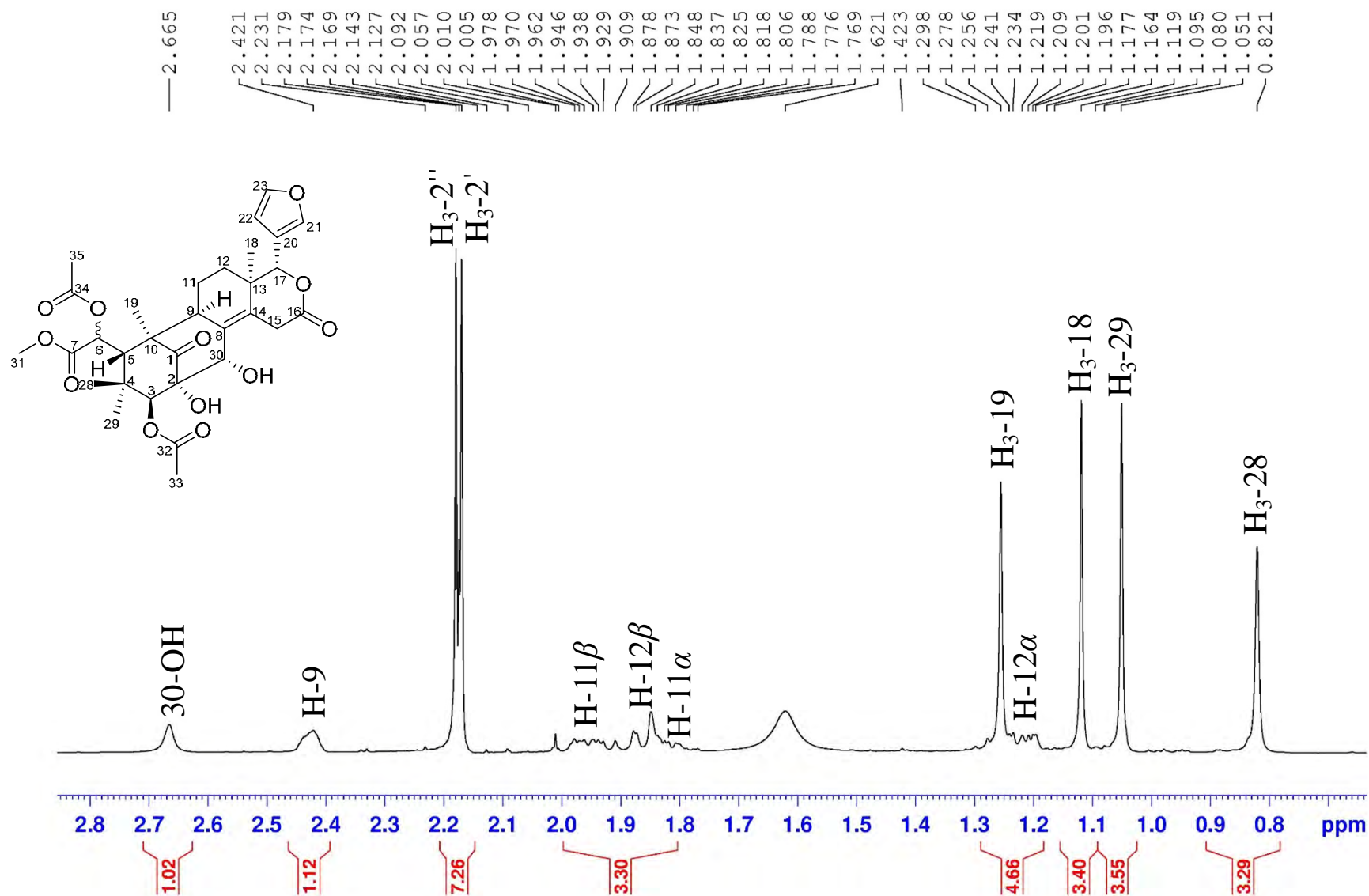
===== CHANNEL f1 =====
SFO1          400.1324710 M
NUC1           1H
P1            12.00 u
SI            65536
SF            400.1300075 M
WDW            EM
SSB            0
LB            0.30 F
GB            0
PC            1.00
  
```



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

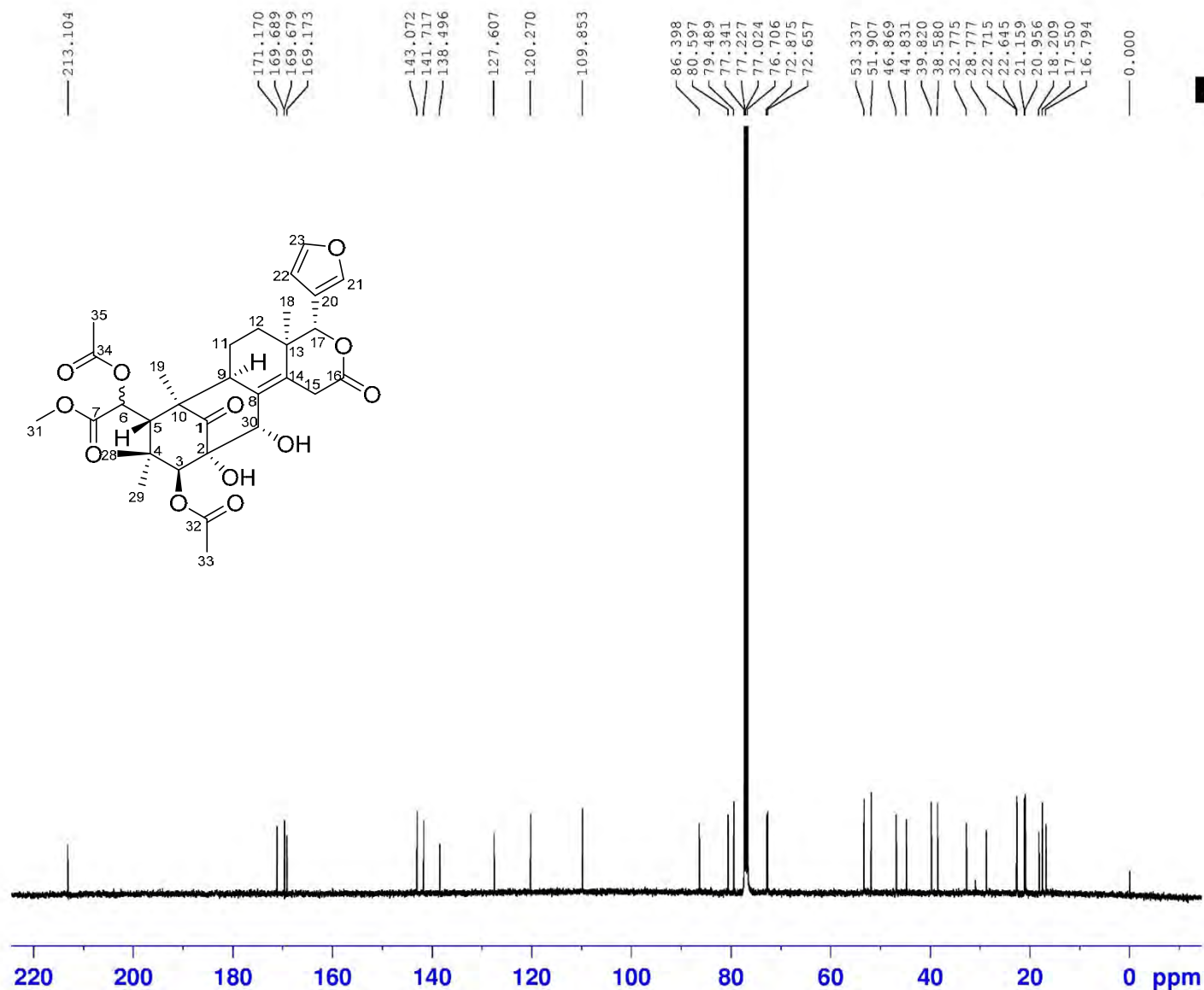


$^1\text{H}$  NMR (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$





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



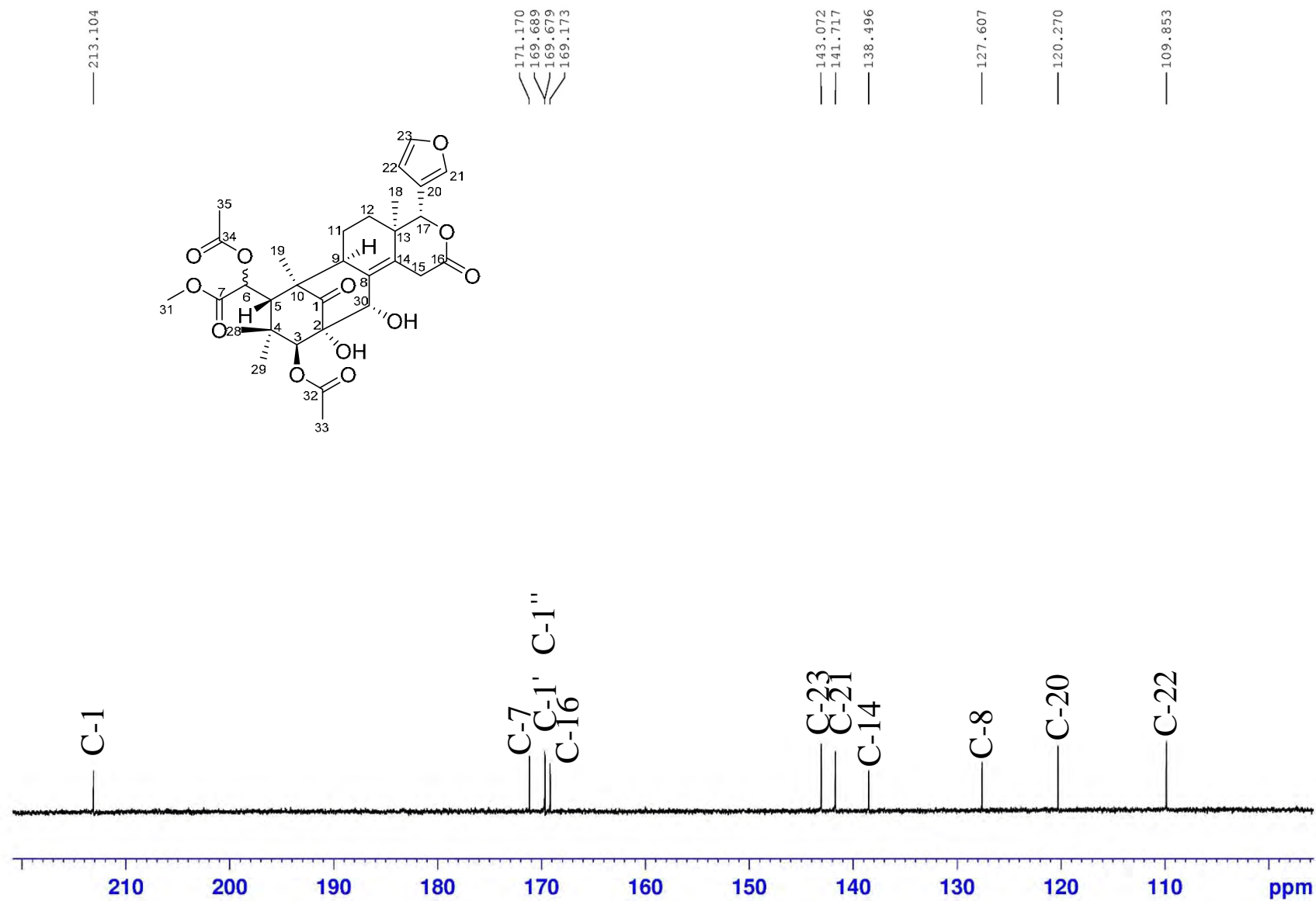
```

NAME                lws-65
EXPNO                2
PROCNO              1
Date_               20150708
Time                10.14
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                    4
SWH                 24038.461 F
FIDRES              0.366798 F
AQ                  1.3631988 s
RG                   117.37
DW                  20.800 u
DE                   18.00 u
TE                   297.0 K
D1                   2.00000000 s
D11                  0.03000000 s
TD0                  1
  
```

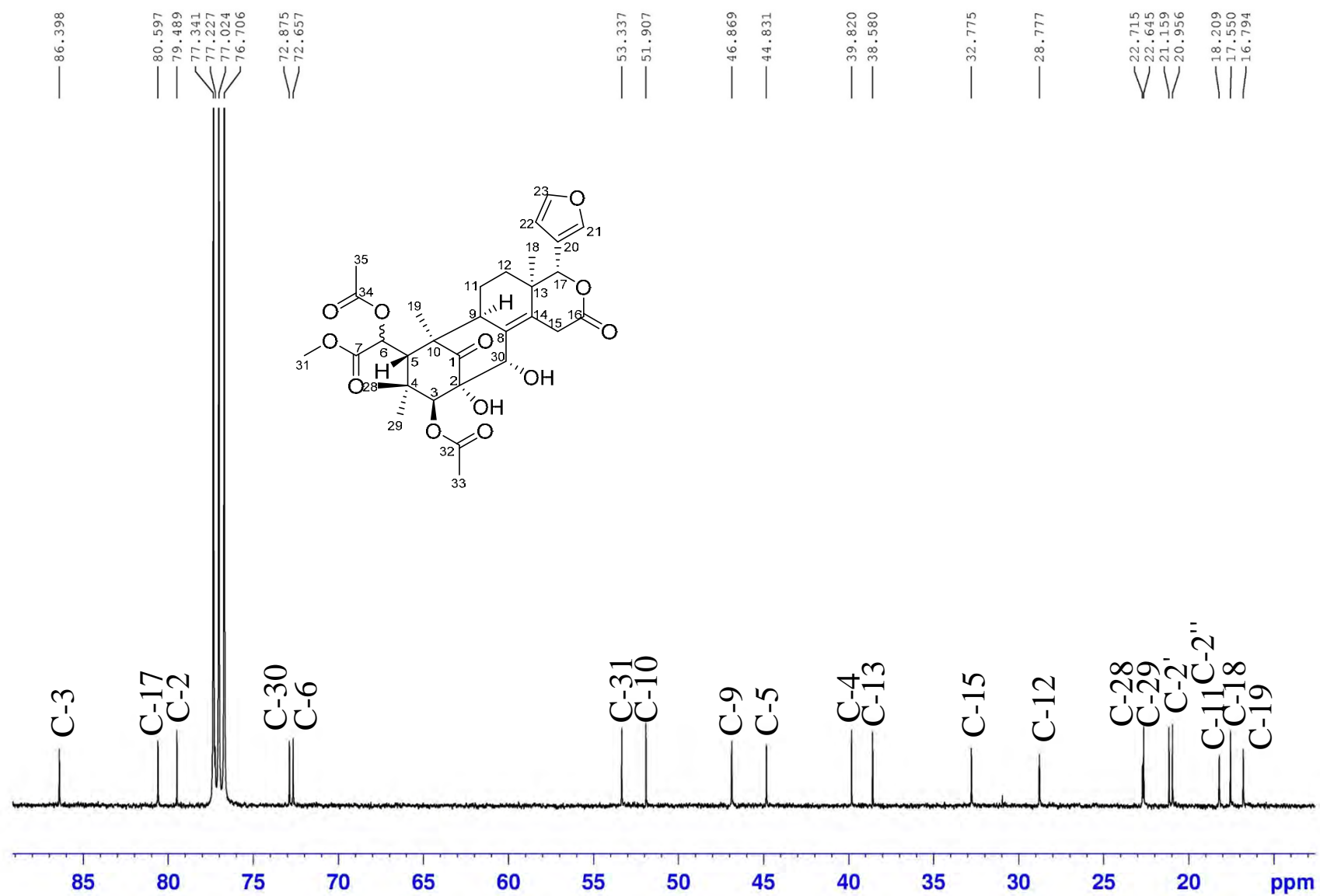
```

===== CHANNEL f1 =====
SFO1                100.6233324 M
NUC1                 13C
P1                   10.00 u
SI                   32768
SF                  100.6127689 M
WDW                  EM
SSB                   0
LB                   1.00 F
GB                    0
PC                   1.40
  
```

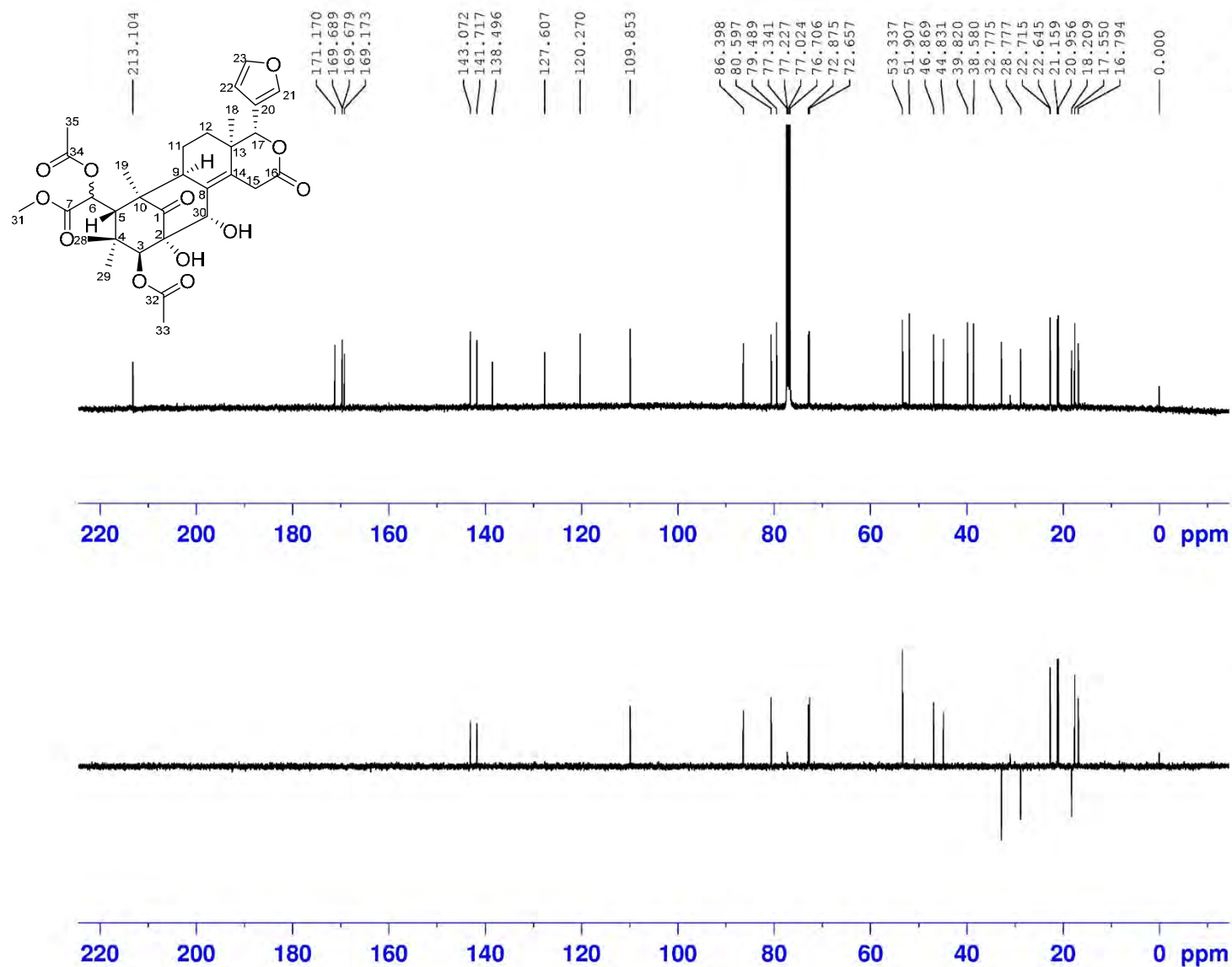
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



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



# DEPT135 (100 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



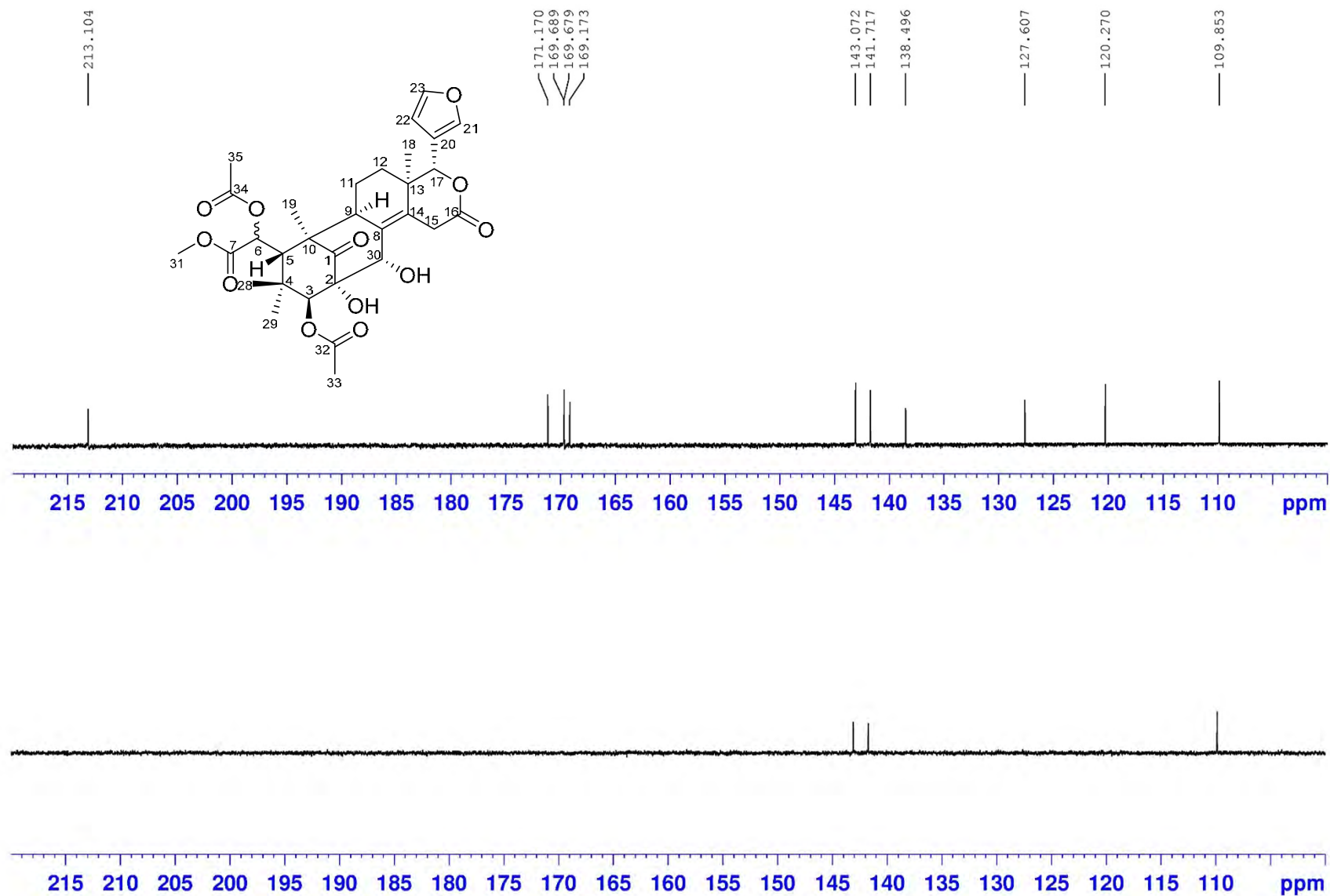
```

NAME          lws-65
EXPNO         3
PROCNO        1
Date_         20150708
Time          10.32
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       deptsp135
TD            65536
SOLVENT       CDCl3
NS            300
DS            4
SWH           24038.461 F
FIDRES        0.366798 F
AQ            1.3631988 s
RG            117.37
DW            20.800 s
DE            18.00 s
TE            297.0 F
CNST2         145.0000000
D1            2.0000000 s
D2            0.00344828 s
D12           0.00002000 s
TD0           1
  
```

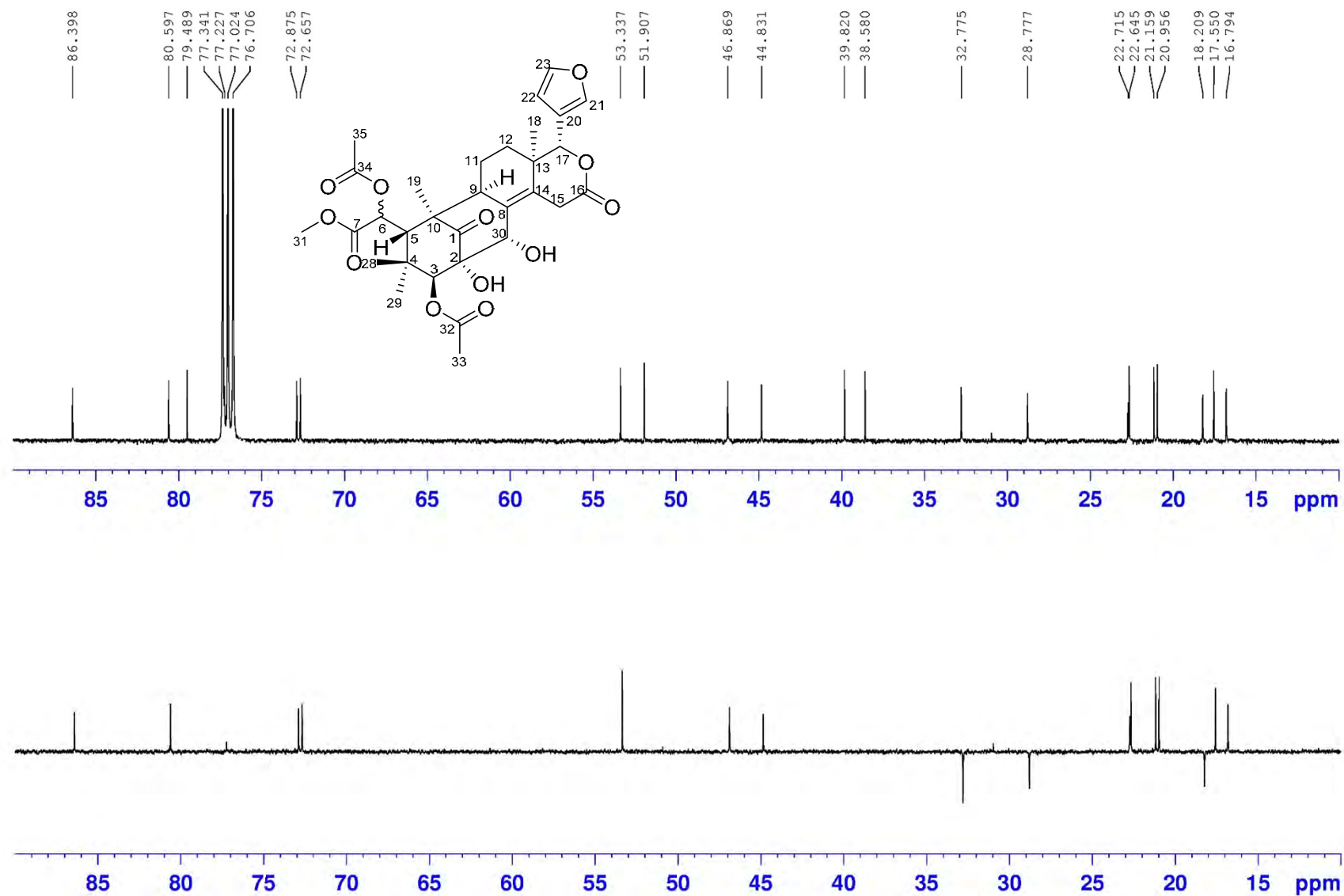
```

===== CHANNEL f1 =====
SFO1         100.6233324 M
NUC1          13C
P1            10.00 s
P13           2000.00 s
SI            32768
SF            100.6127690 M
WDW           EM
SSB           0
LB            1.00 F
GB            0
PC            1.40
  
```

# DEPT135 (100 MHz) spectrum of compound **5** in CDCl<sub>3</sub>

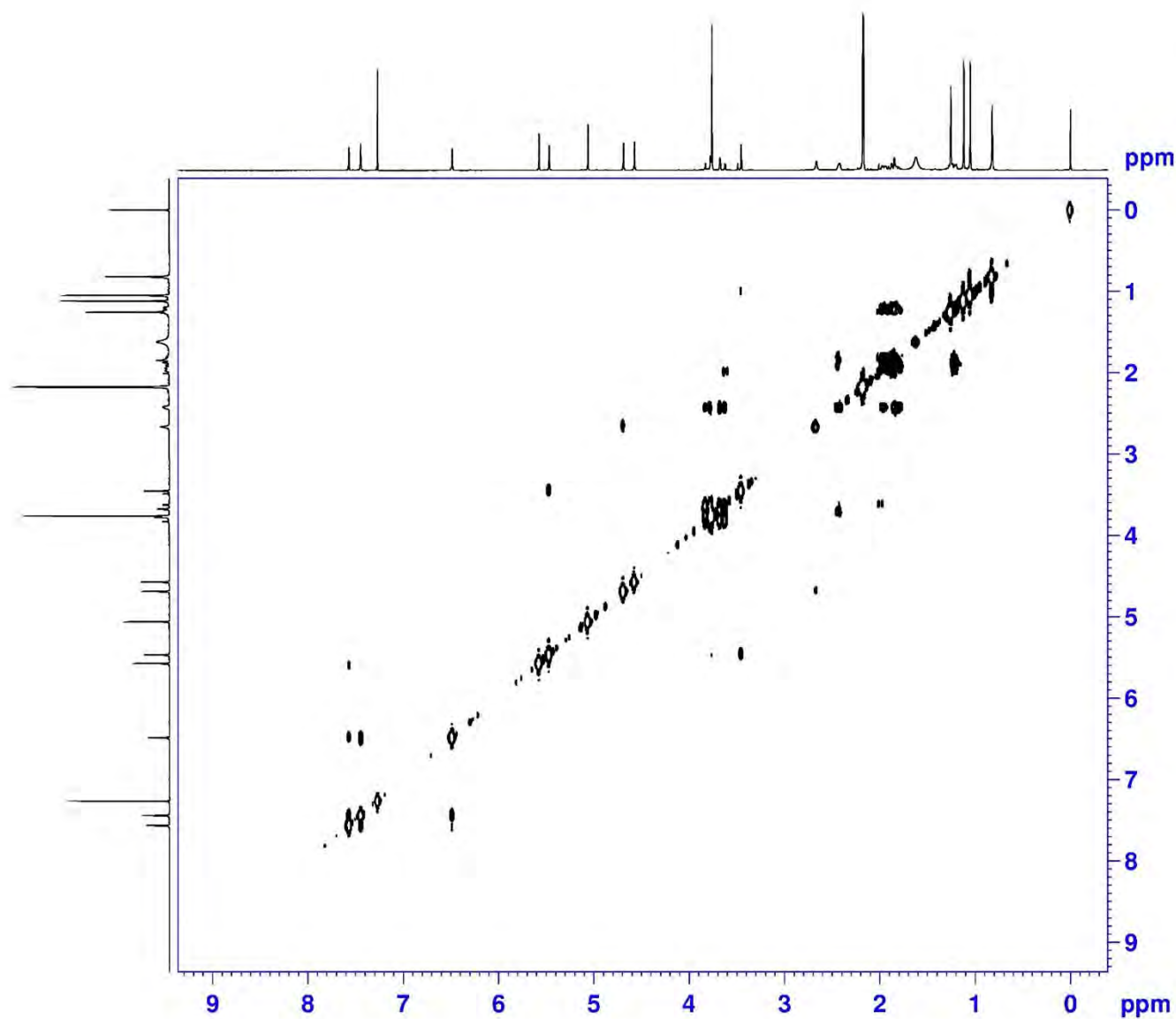


# DEPT135 (100 MHz) spectrum of compound **5** in CDCl<sub>3</sub>





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



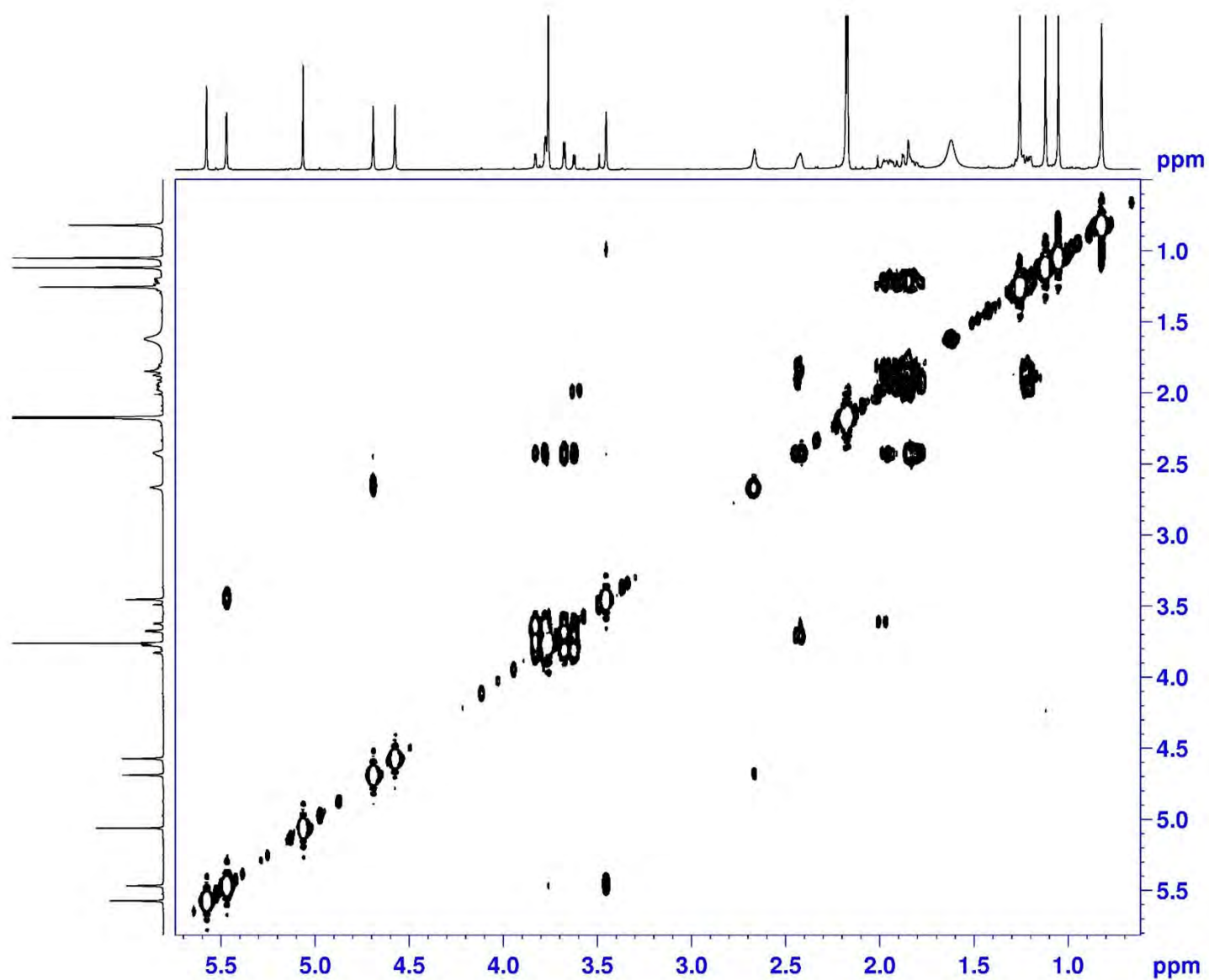
```

NAME          lws-65
EXPNO         4
PROCNO        1
Date_         20150709
Time          3.58
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       cosygpppgf
TD            2048
SOLVENT       CDCl3
NS            8
DS            8
SWH           3906.250 Hz
FIDRES        1.907349 Hz
AQ            0.2621940 se
RG            208.5
DW            128.000 us
DE            10.00 us
TE            297.0 K
D0            0.00000300 se
D1            1.89678097 se
D11           0.03000000 se
D12           0.00002000 se
D13           0.00000400 se
D16           0.00020000 se
IN0           0.00025600 se
  
```

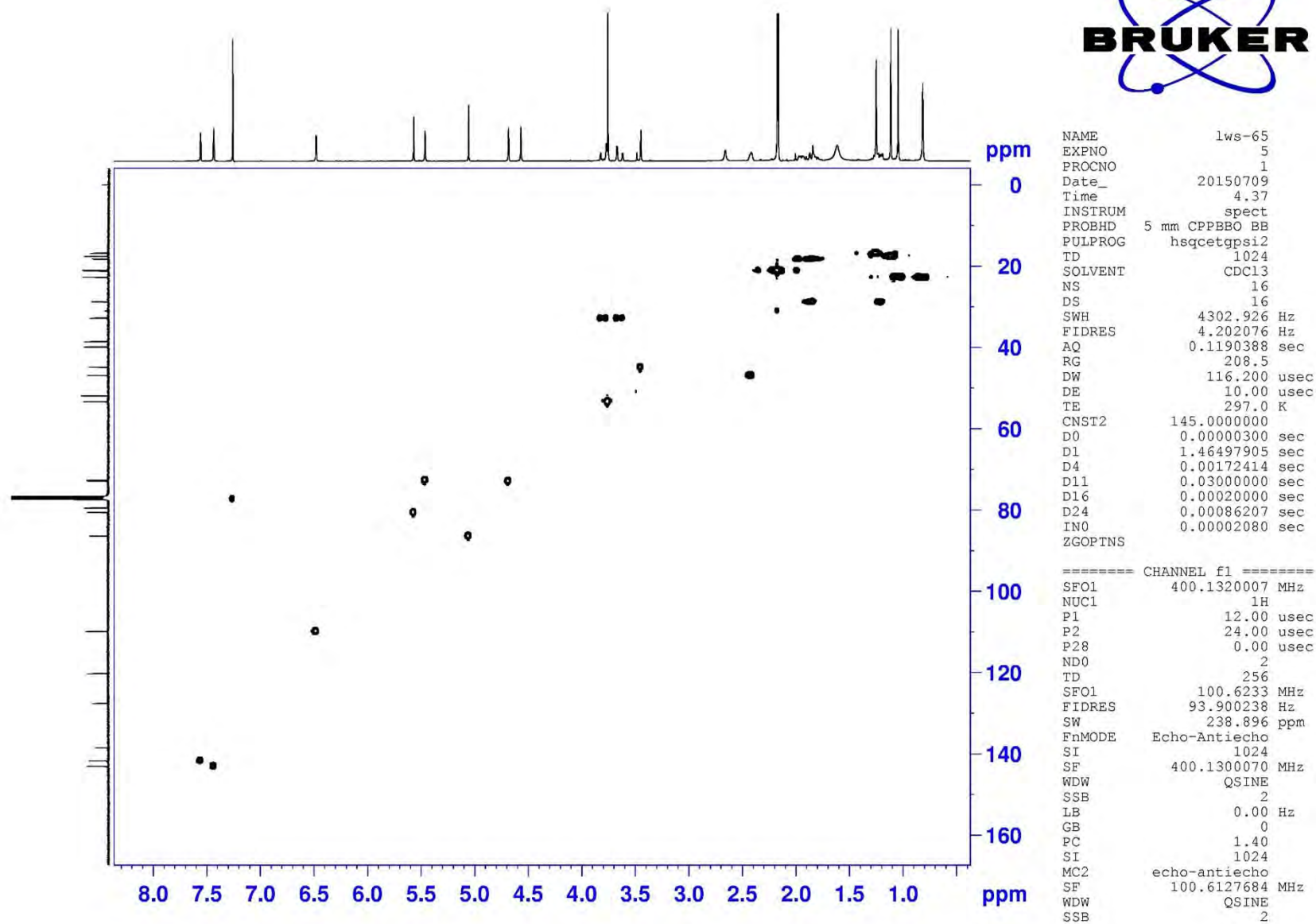
```

===== CHANNEL f1 =====
SFO1         400.1318006 MH
NUC1          1H
P0            12.00 us
P1            12.00 us
P17           2500.00 us
ND0           1
TD            128
SFO1         400.1318 MH
FIDRES        30.517578 Hz
SW            9.762 pp
FnMODE        QF
SI            1024
SF            400.1300065 MH
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            400.1300065 MH
WDW           QSINE
SSB           0
  
```

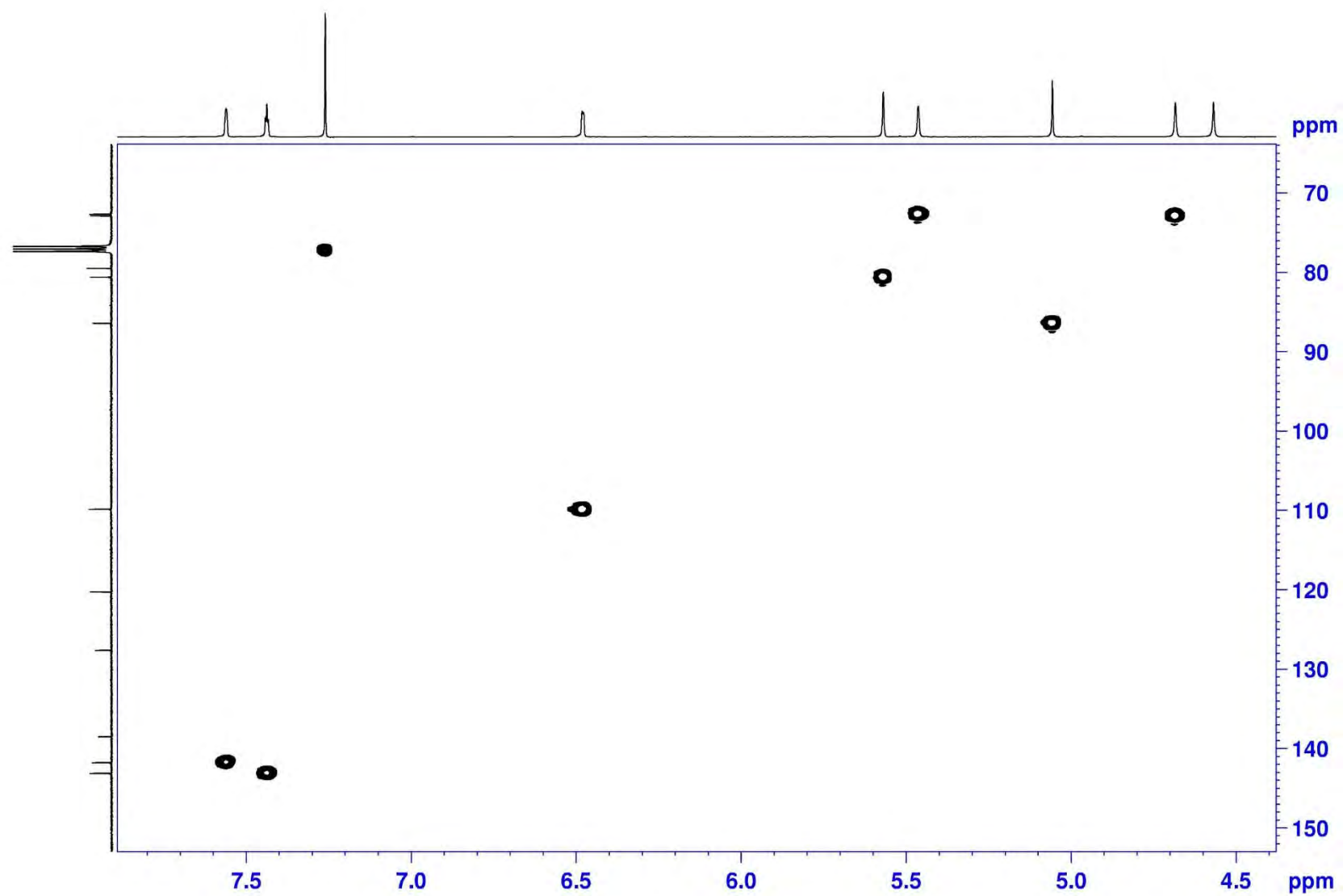
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



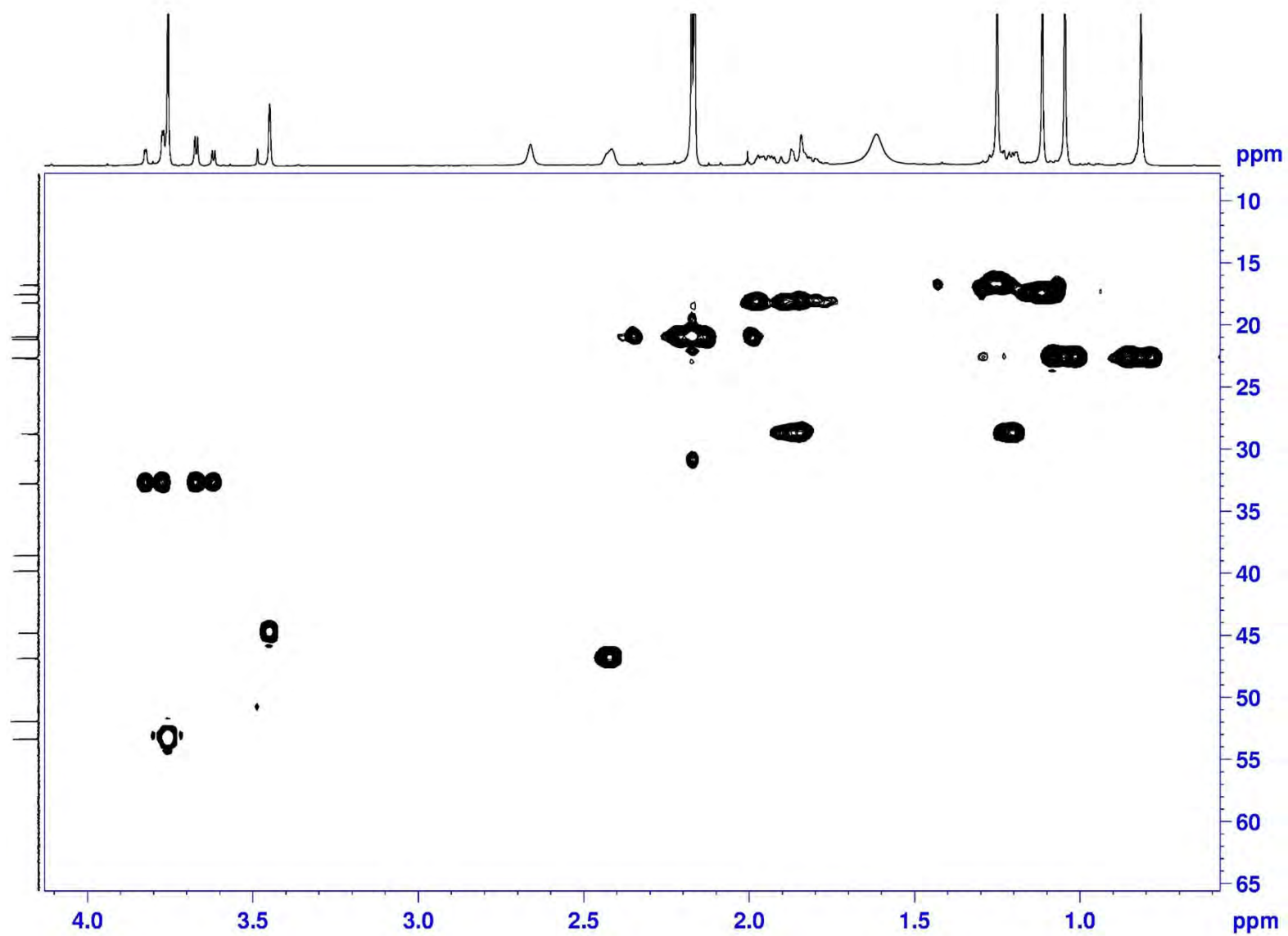
# HSQC (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



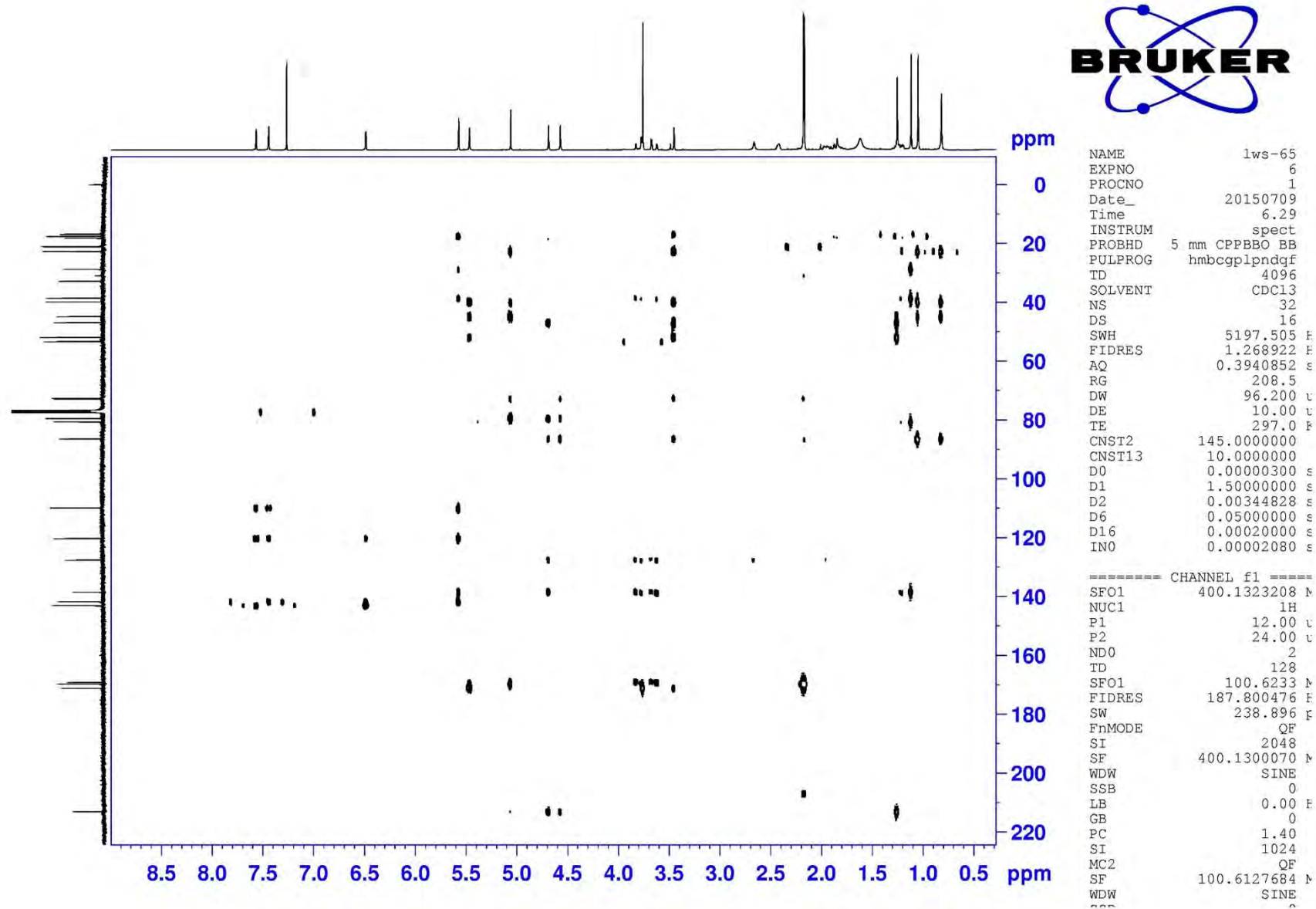
HSQC (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



HSQC (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>

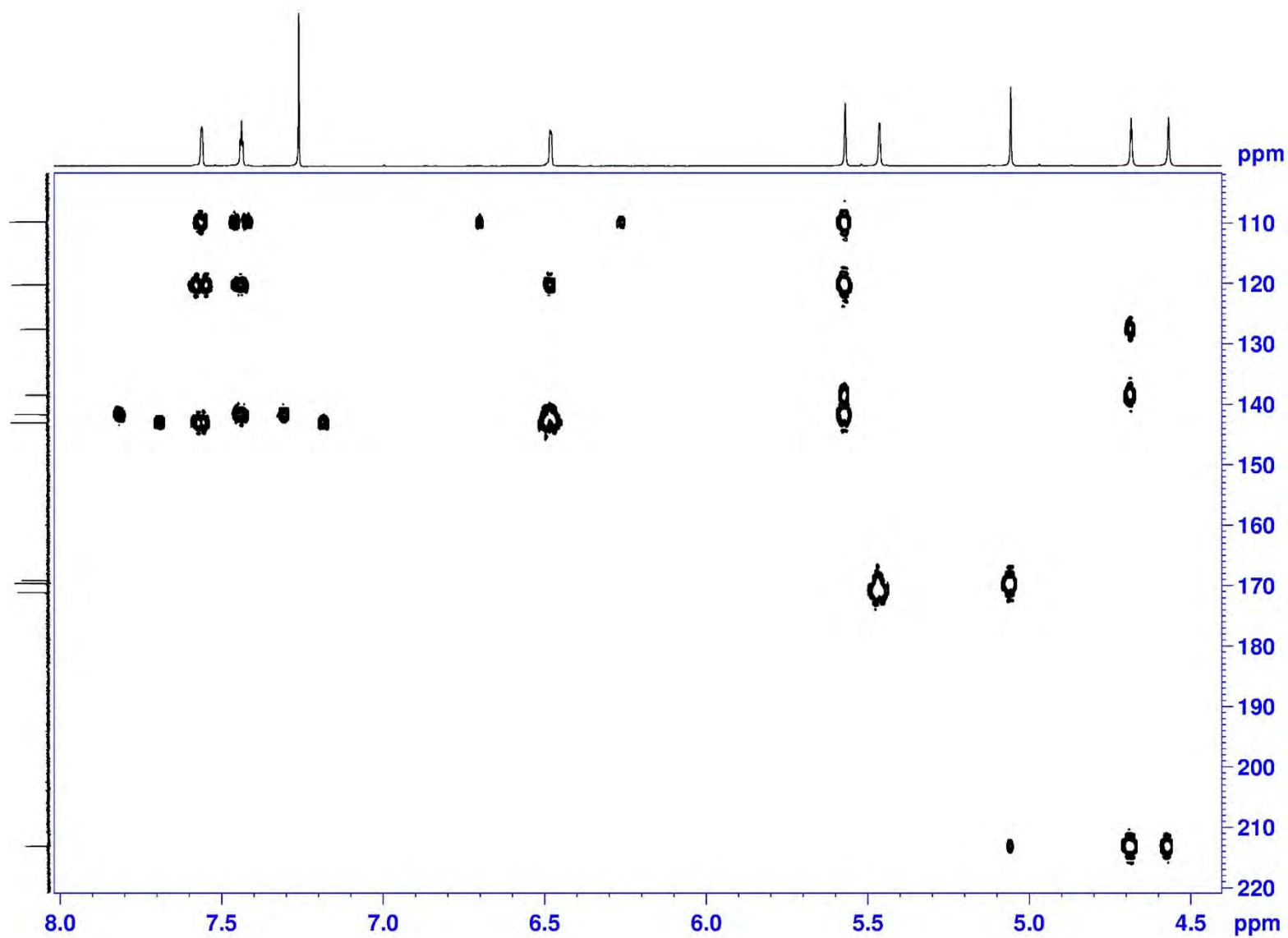


# HMBC (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>

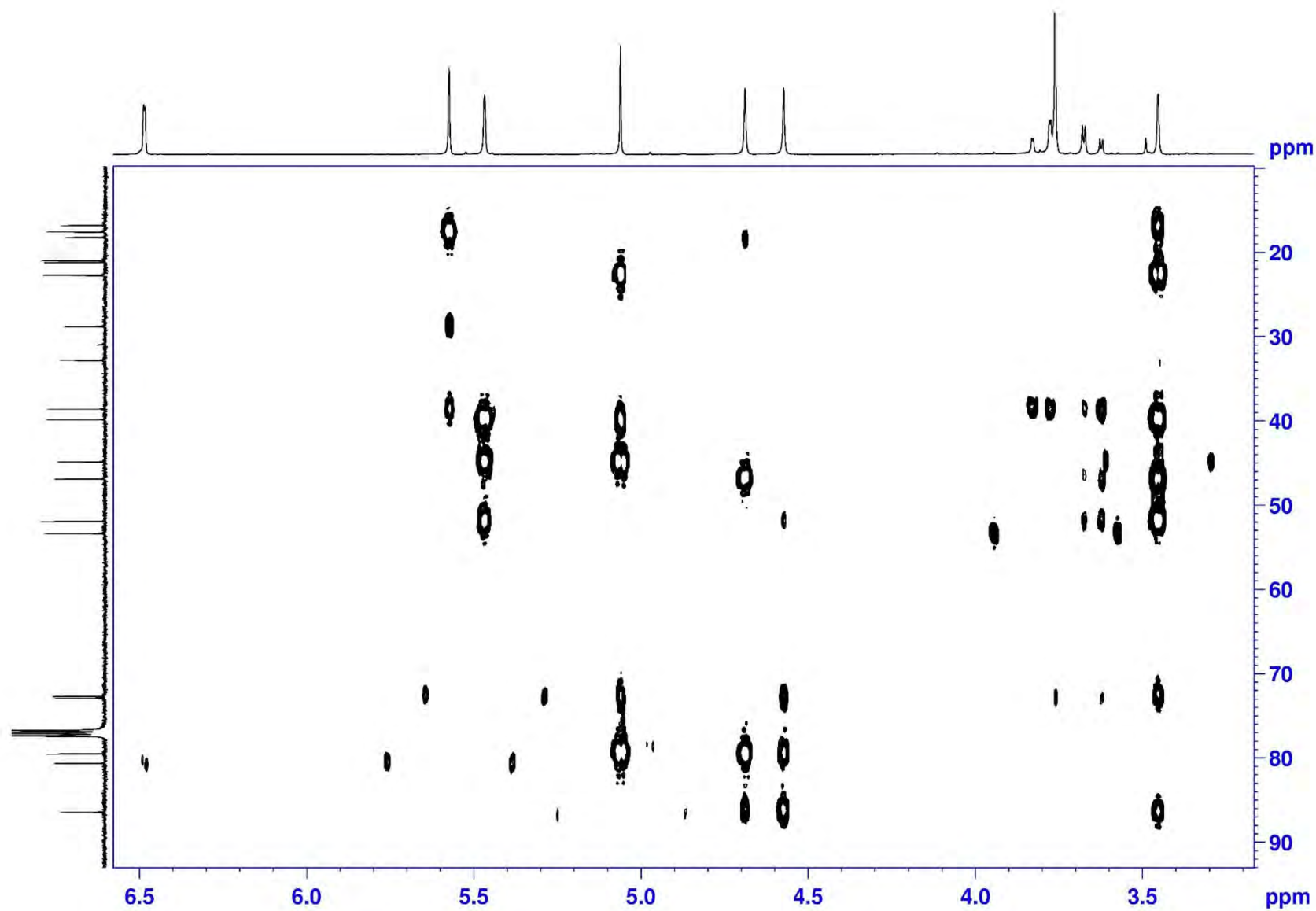




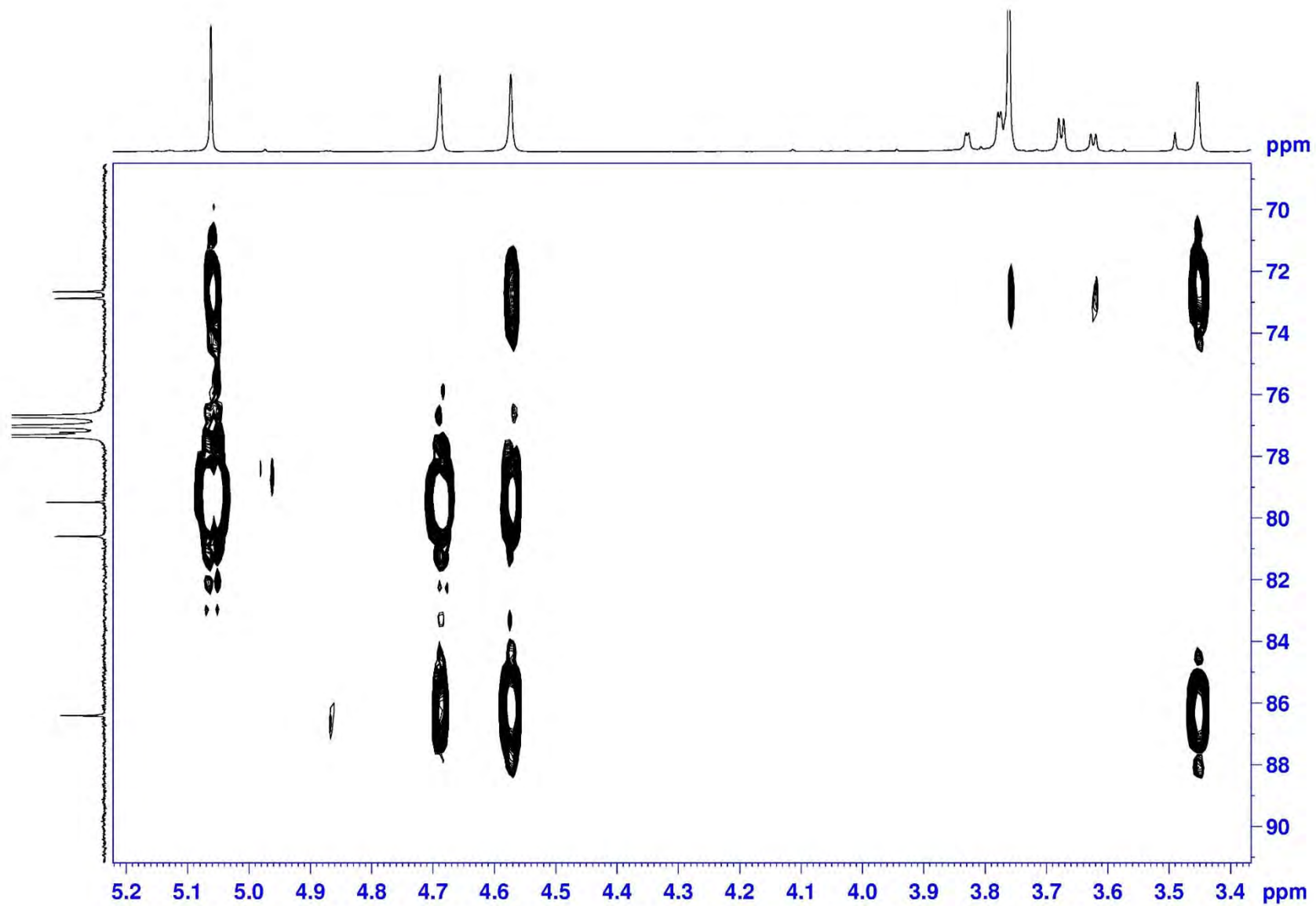
HMBC (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



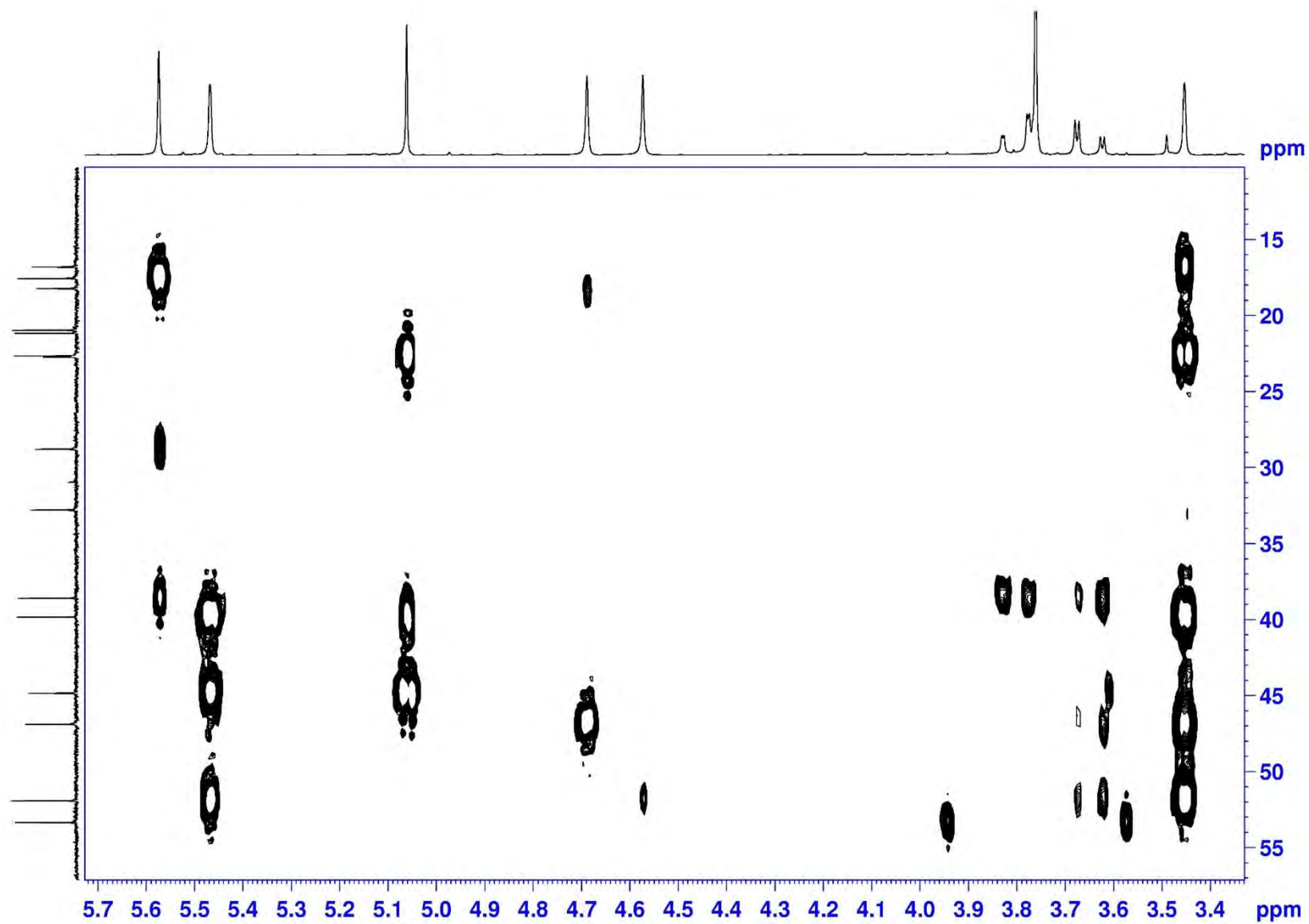
HMBC (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



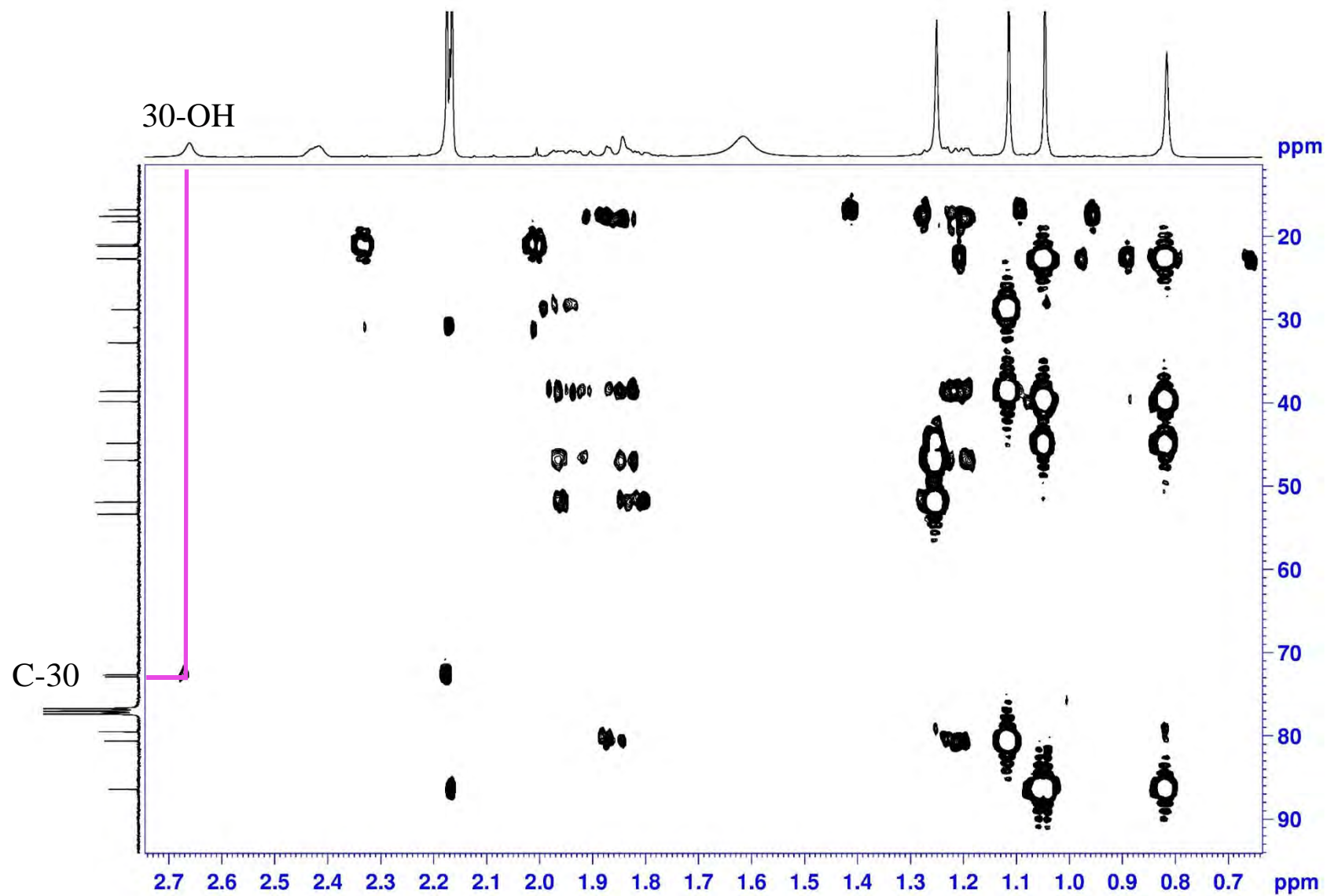
HMBC (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



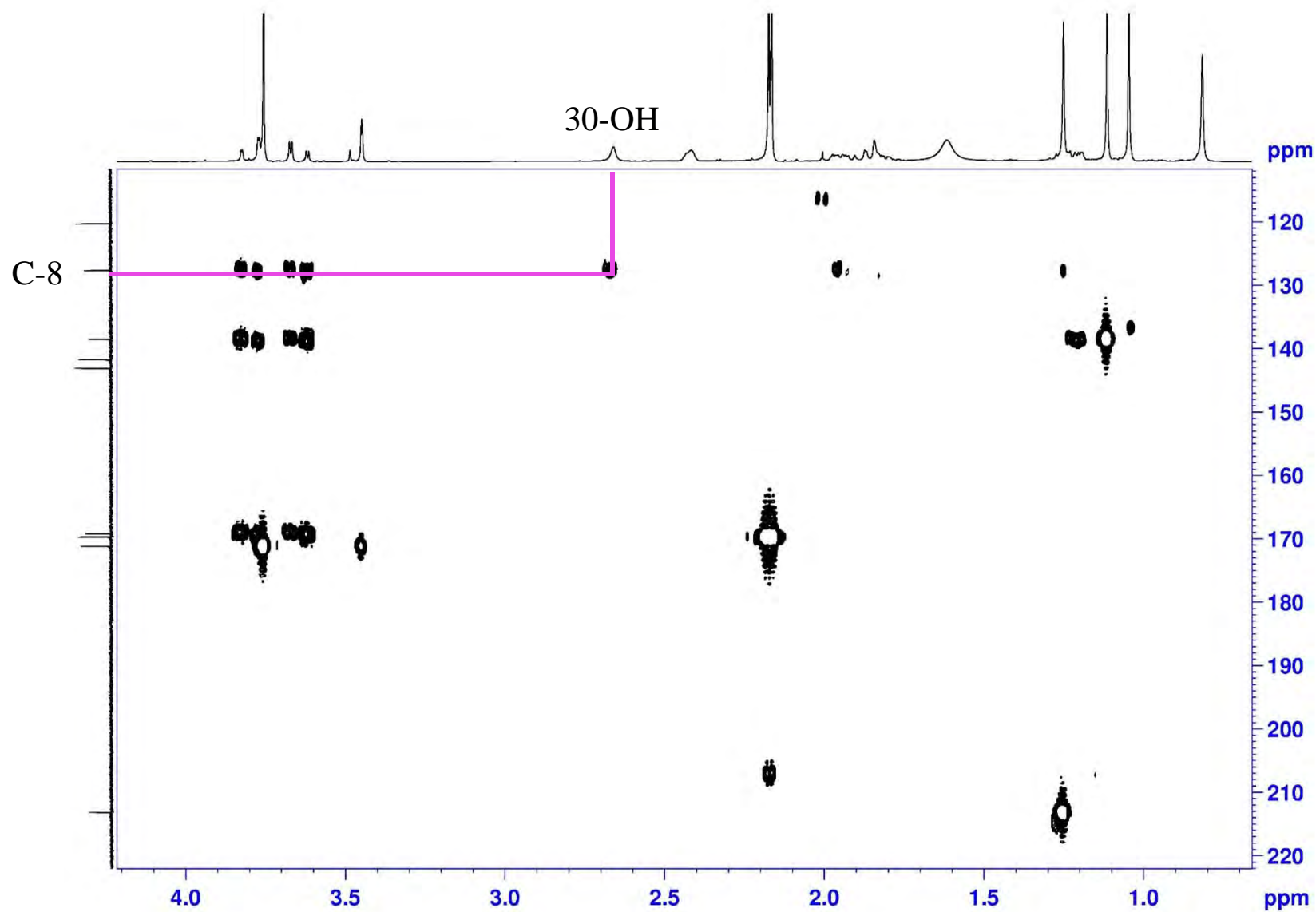
HMBC (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>

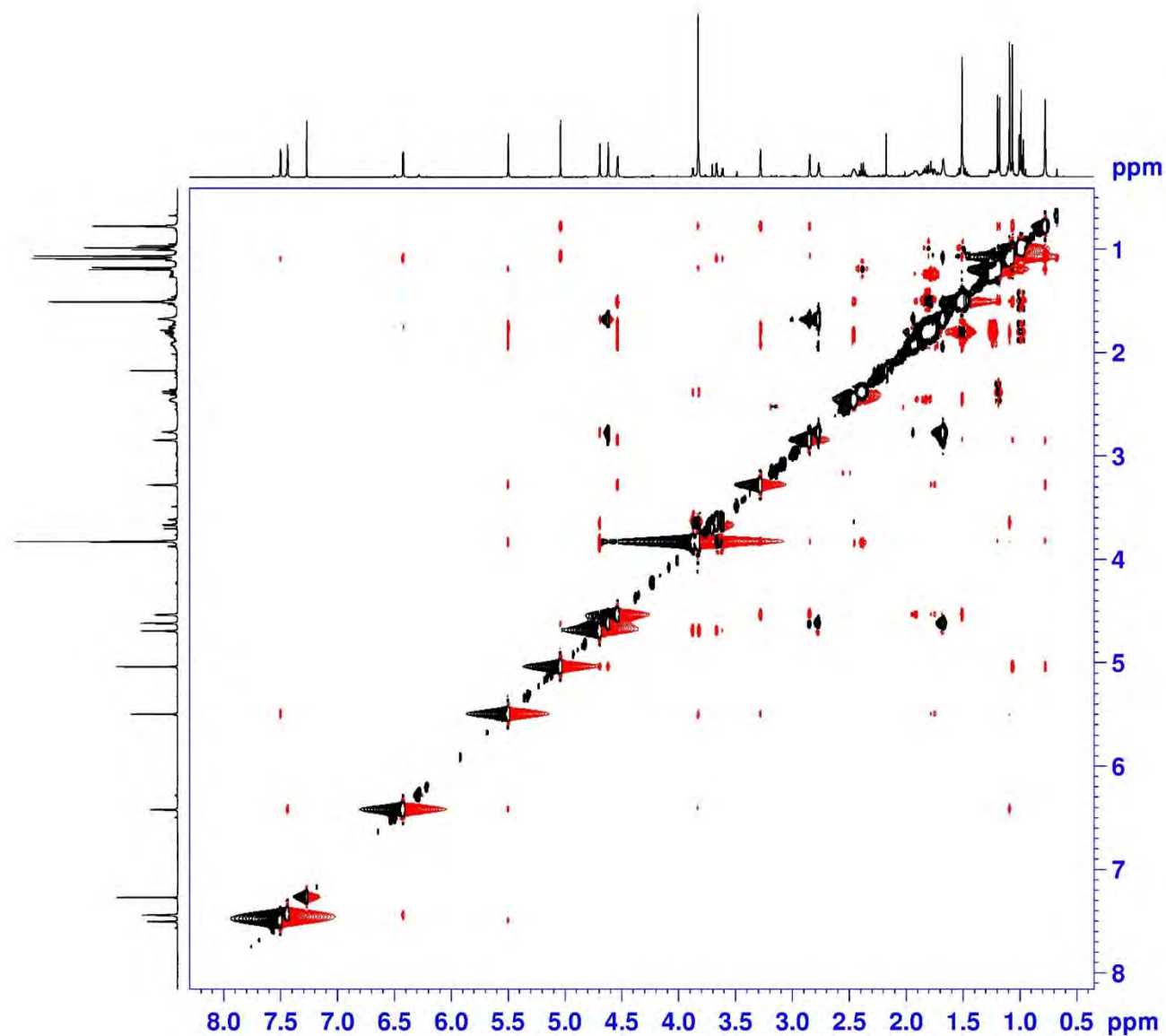


HMBC (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$





# NOESY (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



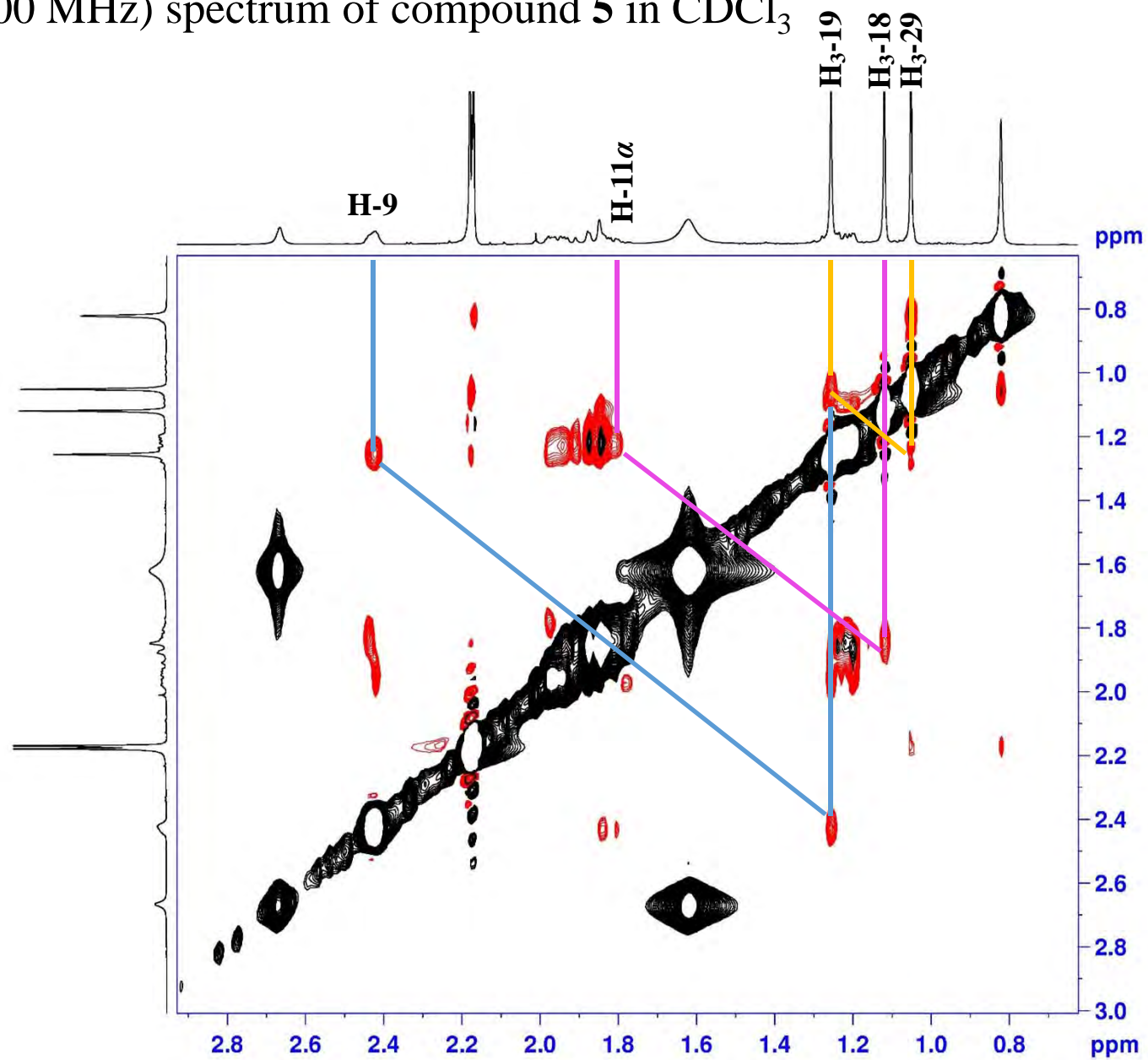
```

NAME          lws-63-1
EXPNO          701
PROCNO         1
Date_         20150710
Time           7.33
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        noesygpphph
TD             2048
SOLVENT        CDCl3
NS             16
DS             32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ             0.2560500 s
RG             91.64
DW             125.000 us
DE             10.00 us
TE             297.0 K
D0             0.00010972 s
D1             1.99385595 s
D8             0.30000001 s
D11            0.03000000 s
D12            0.00002000 s
D16            0.00020000 s
IN0            0.00025000 s
  
```

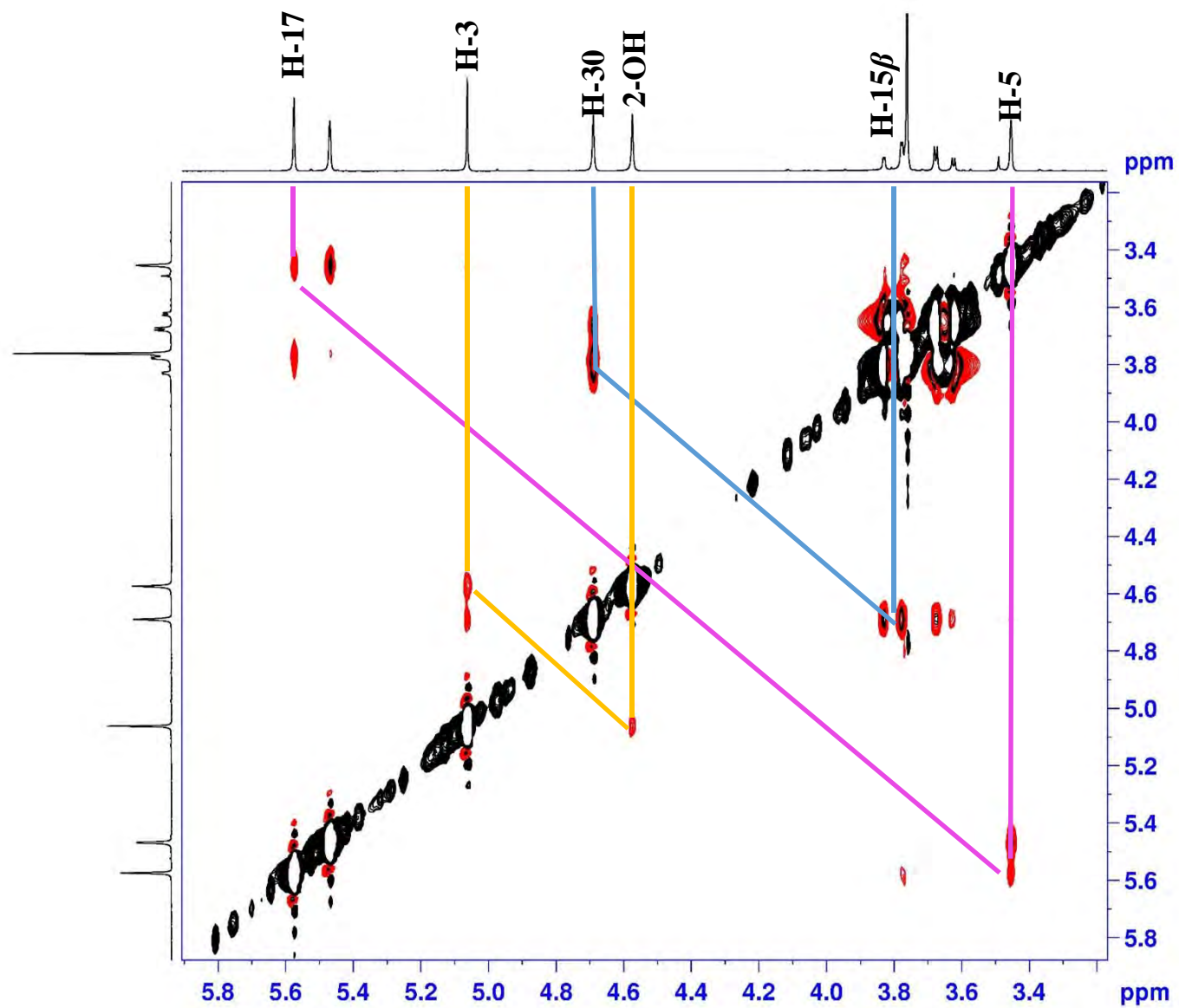
```

===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1           1H
P1             12.00 us
P2             24.00 us
P17            2500.00 us
ND0            1
TD             256
SFO1          400.1318 MHz
FIDRES         15.625000 Hz
SW             9.997 MHz
FnMODE         States-TPPI
SI             1024
SF            400.1300058 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF            400.1300058 MHz
WDW            QSINE
  
```

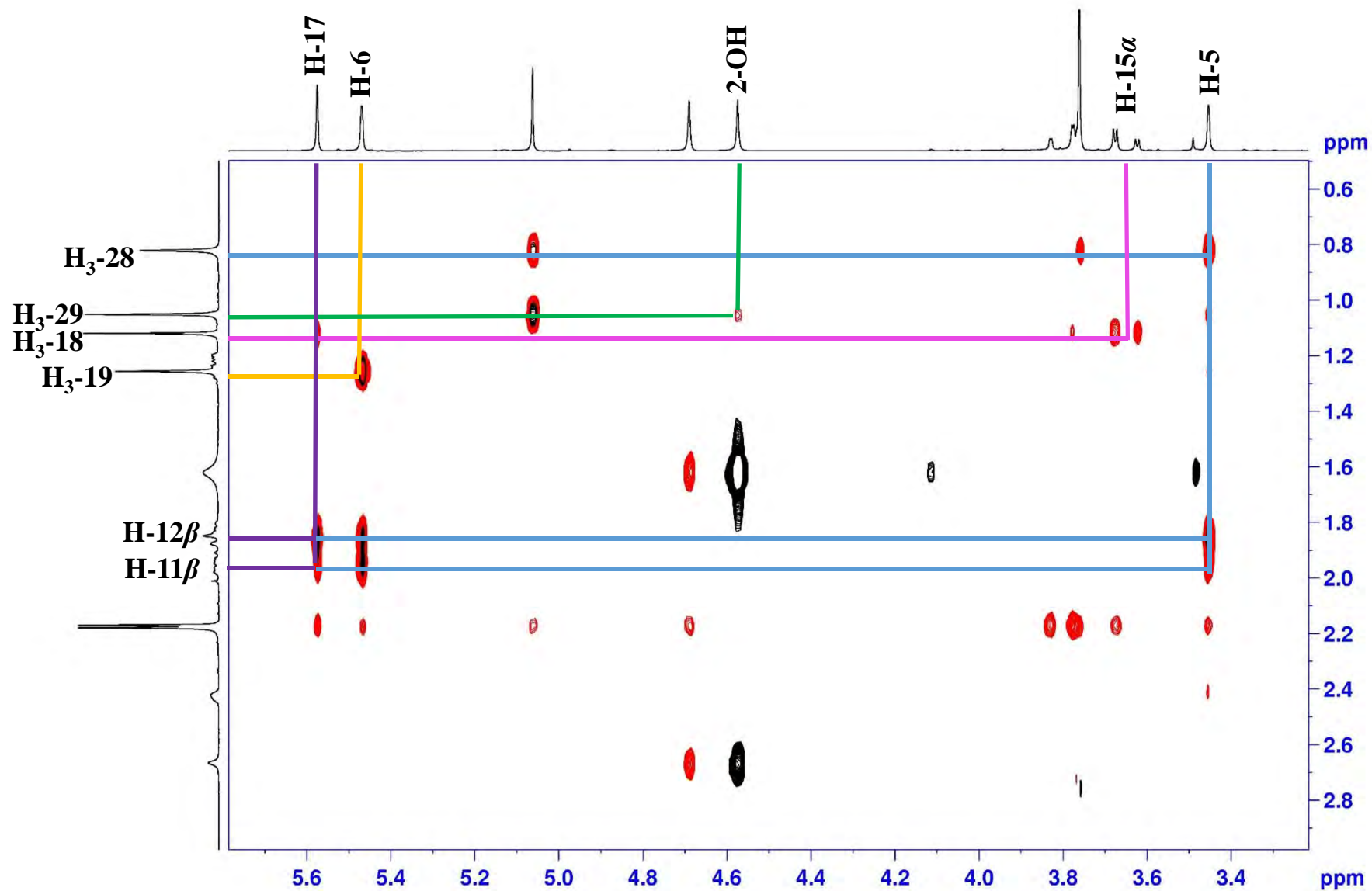
NOESY (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



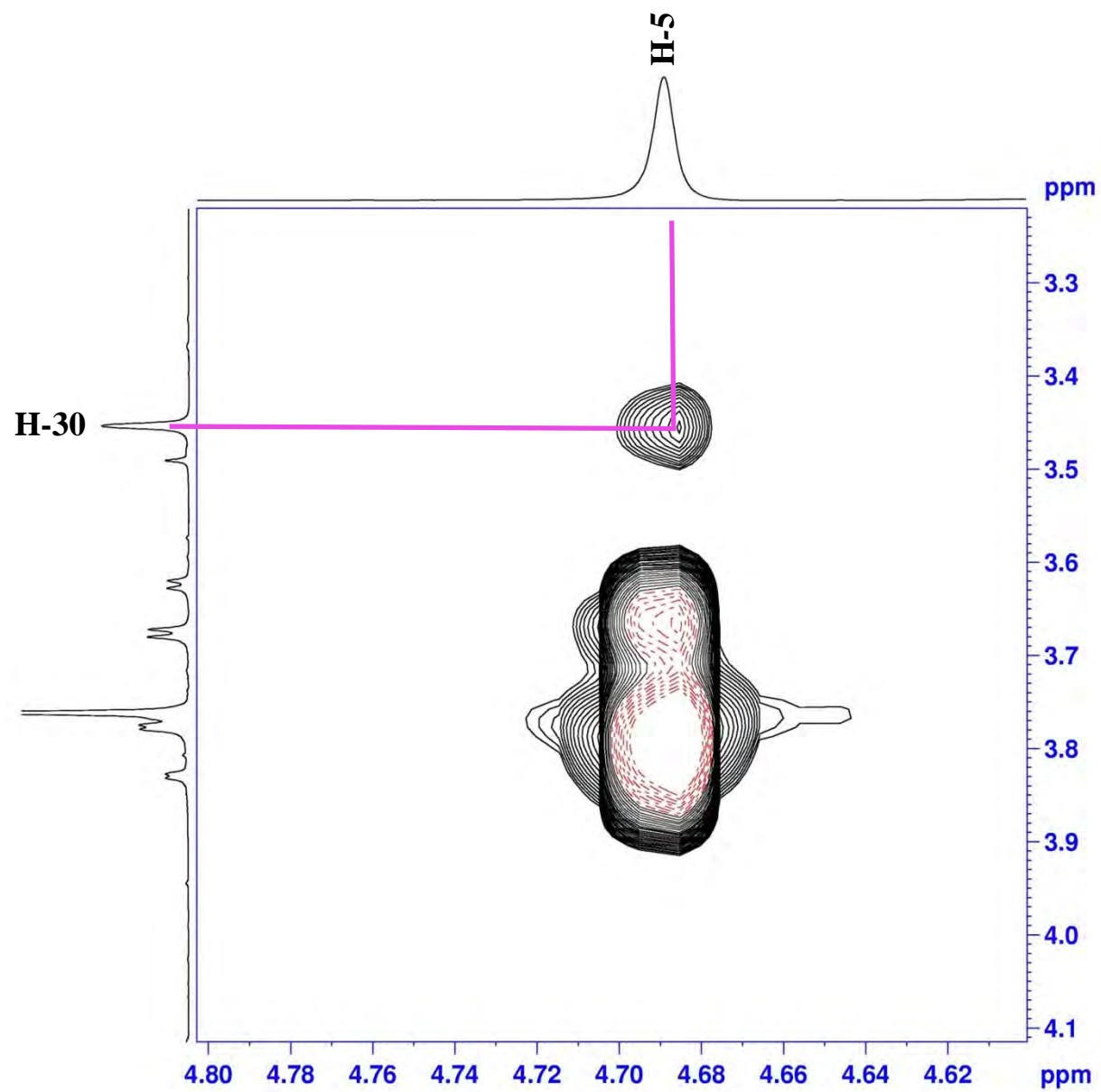
NOESY (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **5** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **5** in  $\text{CDCl}_3$



# HR-ESIMS for compound 6

## Mass Spectrum SmartFormula Report

### Analysis Info

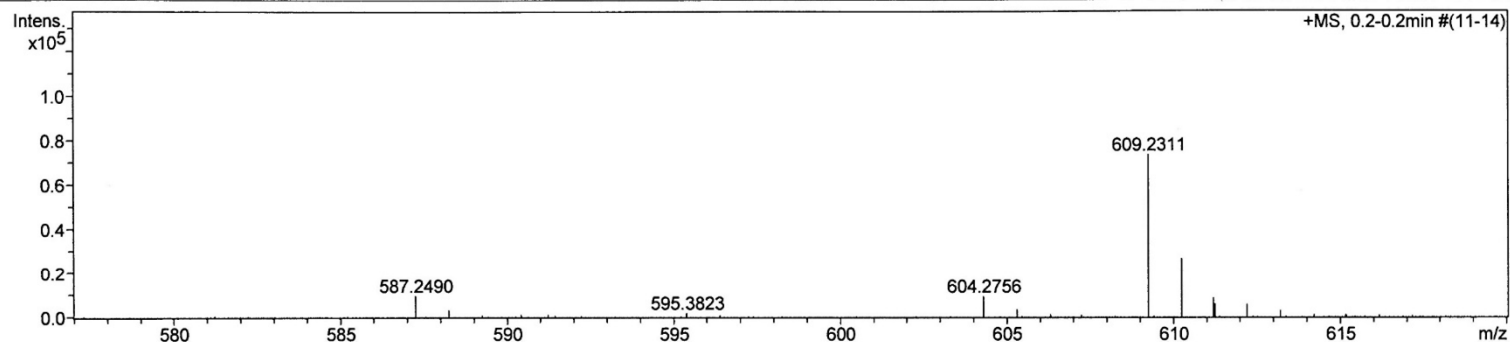
Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-80-1\_pos\_23\_01\_833.d  
Method LC\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name liwanshan\_lws-80-1\_pos  
Comment

Acquisition Date 11/18/2015 10:07:03 PM

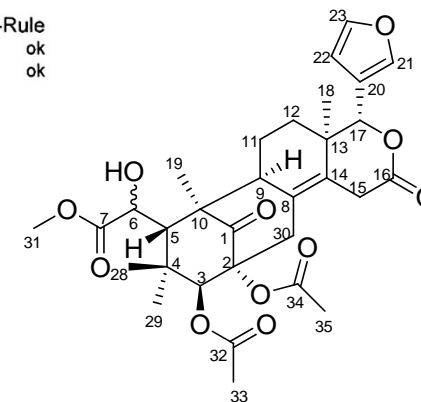
Operator SCSIO  
Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
587.2490	1	C 31 H 39 O 11	100.00	587.2487	-0.6	-0.4	4.3	12.5	even	ok
609.2311	1	C 31 H 38 Na O 11	100.00	609.2306	-0.7	-0.4	11.0	12.5	even	ok





# HR-ESIMS for compound 6

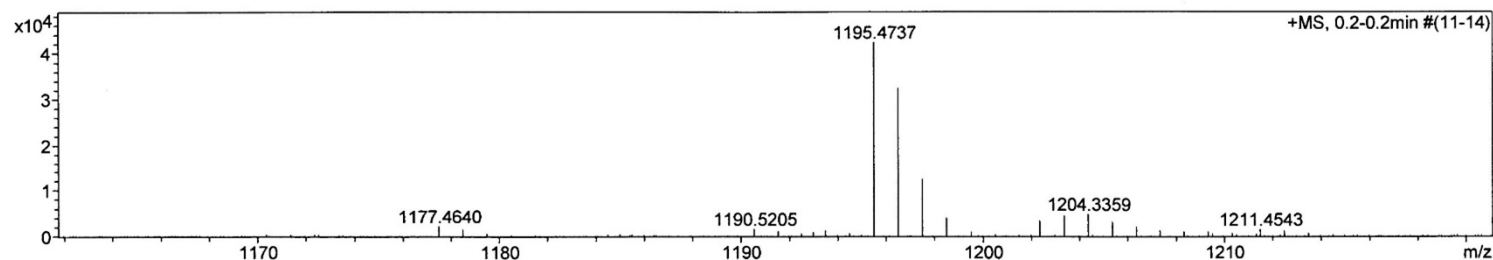
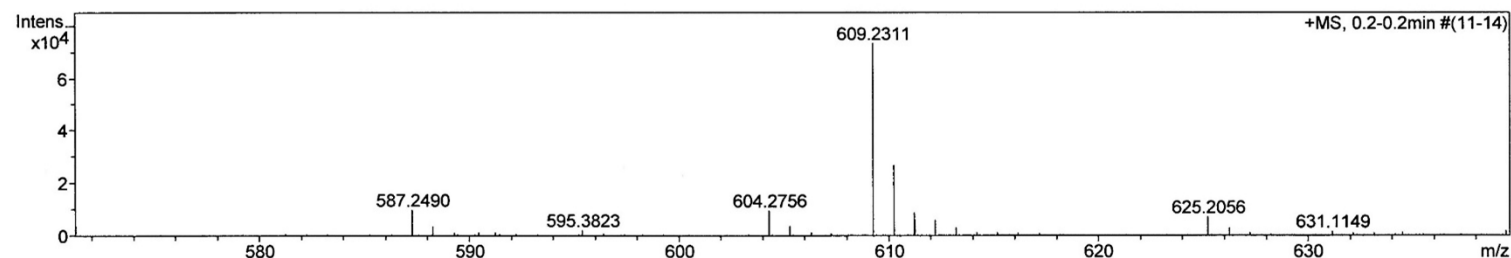
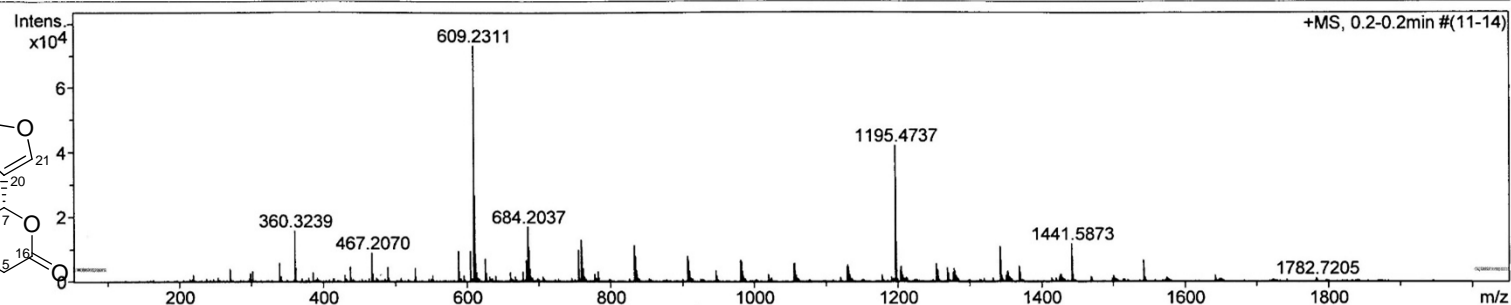
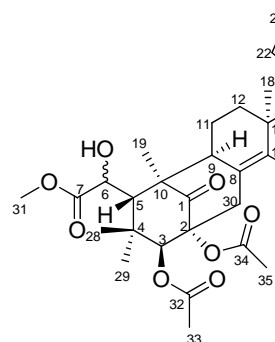
## Generic Display Report

### Analysis Info

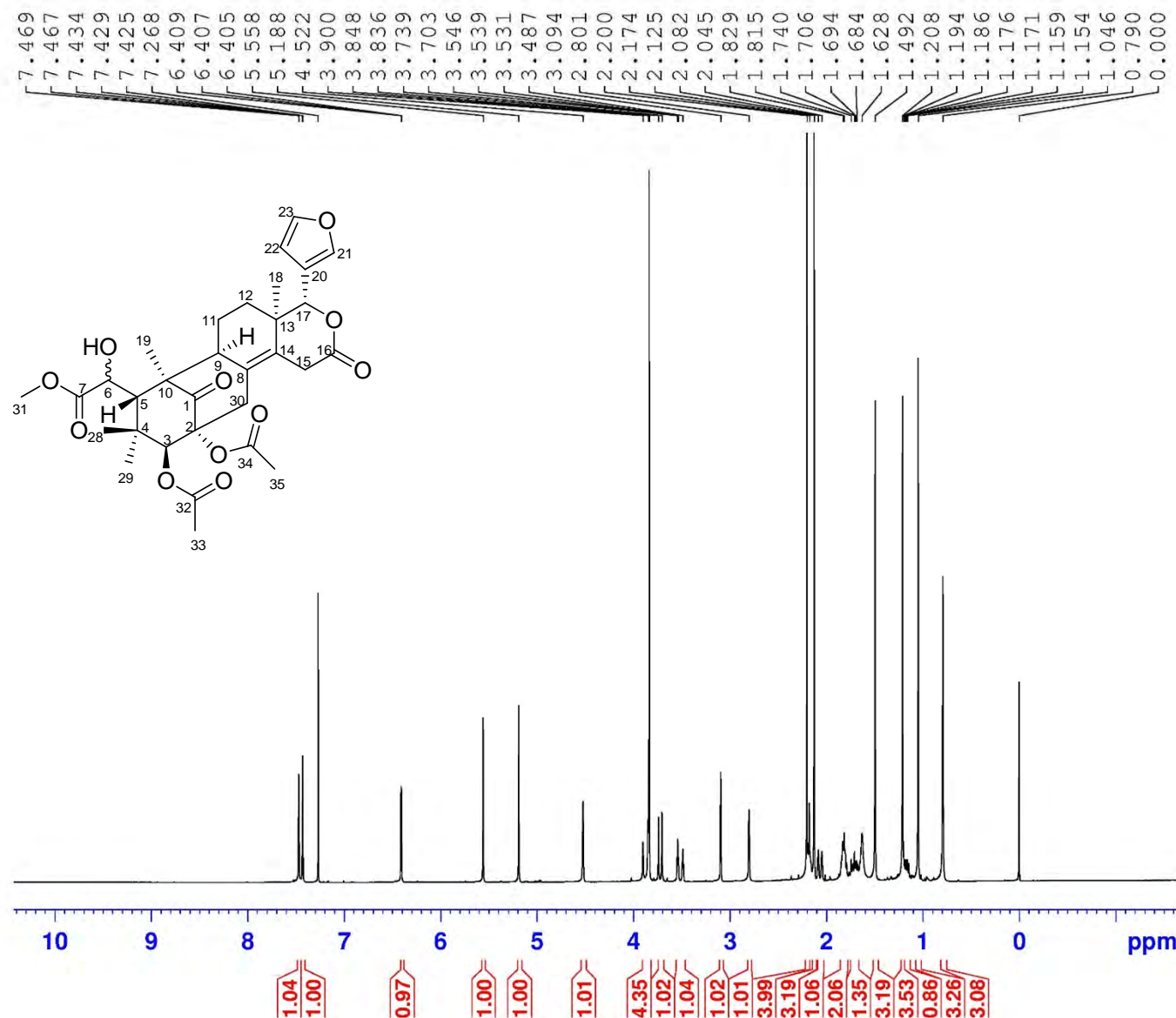
Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-80-1\_pos\_23\_01\_833.d  
Method LC\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name liwanshan\_lws-80-1\_pos  
Comment

Acquisition Date 11/18/2015 10:07:03 PM

Operator SCSIO  
Instrument maXis



# <sup>1</sup>H NMR (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



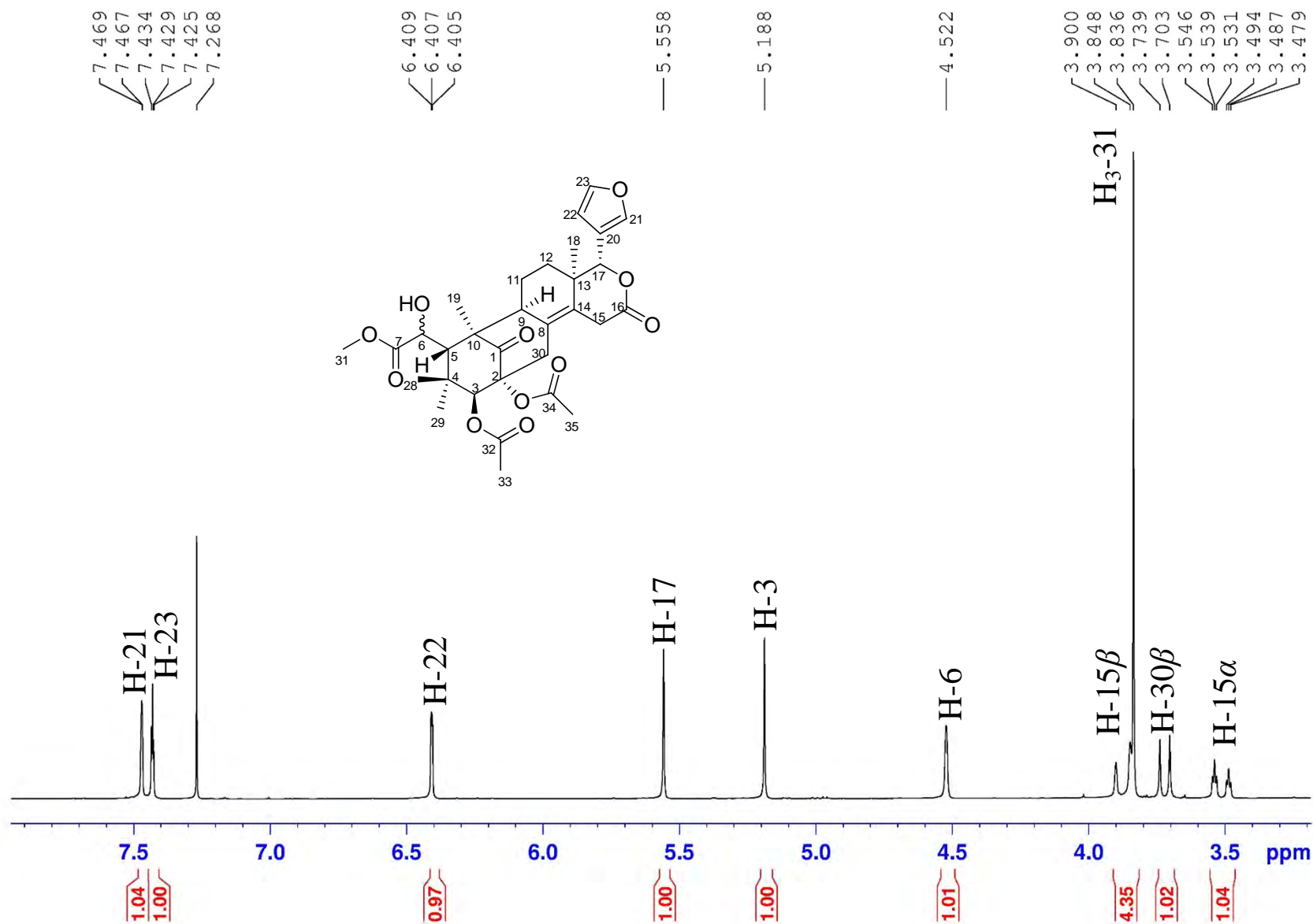
```

NAME          lws-80-1
EXPNO          1
PROCNO         1
Date_          20151003
Time           18.35
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        zg30
TD             65536
SOLVENT        CDCl3
NS             16
DS             2
SWH            8223.685 F
FIDRES         0.125483 F
AQ            3.9846387 s
RG            130.26
DW            60.800 u
DE            10.00 u
TE            297.0 K
D1            1.00000000 s
TD0            1
    
```

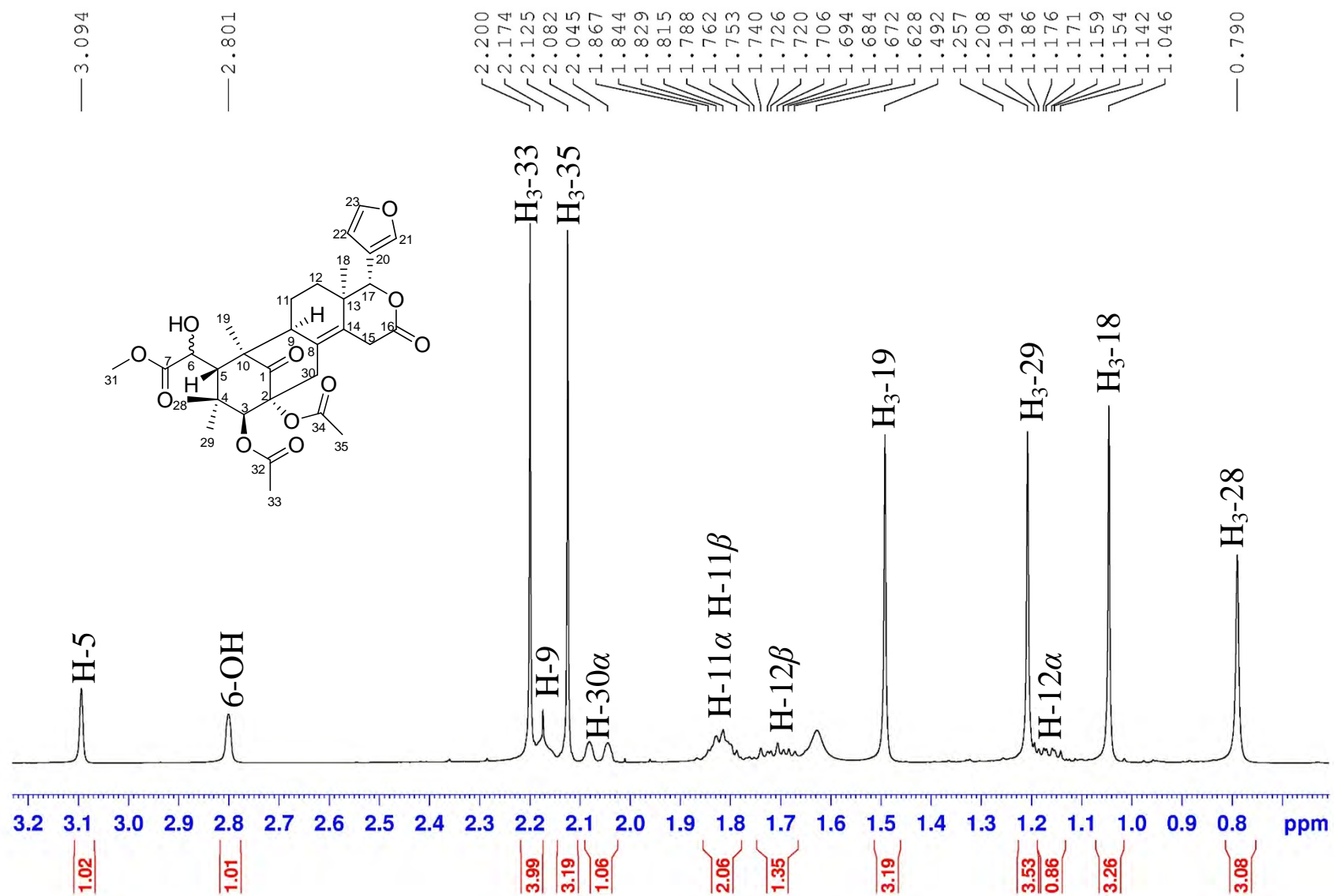
```

===== CHANNEL f1 =====
SFO1          400.1324710 M
NUC1           1H
P1            12.00 u
SI            65536
SF            400.1300067 M
WDW            EM
SSB            0
LB            0.30 F
GB            0
PC            1.00
    
```

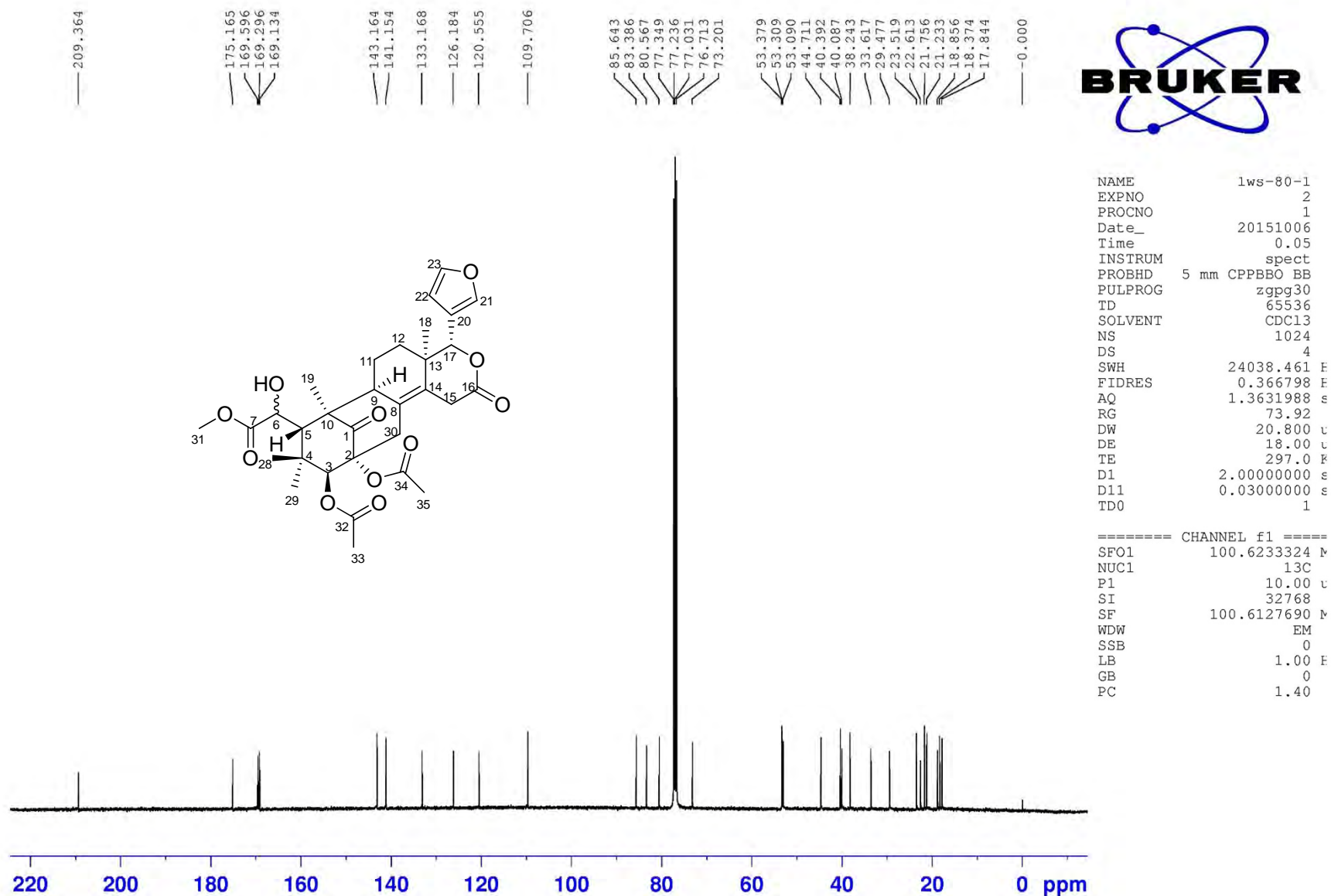
$^1\text{H}$  NMR (400 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



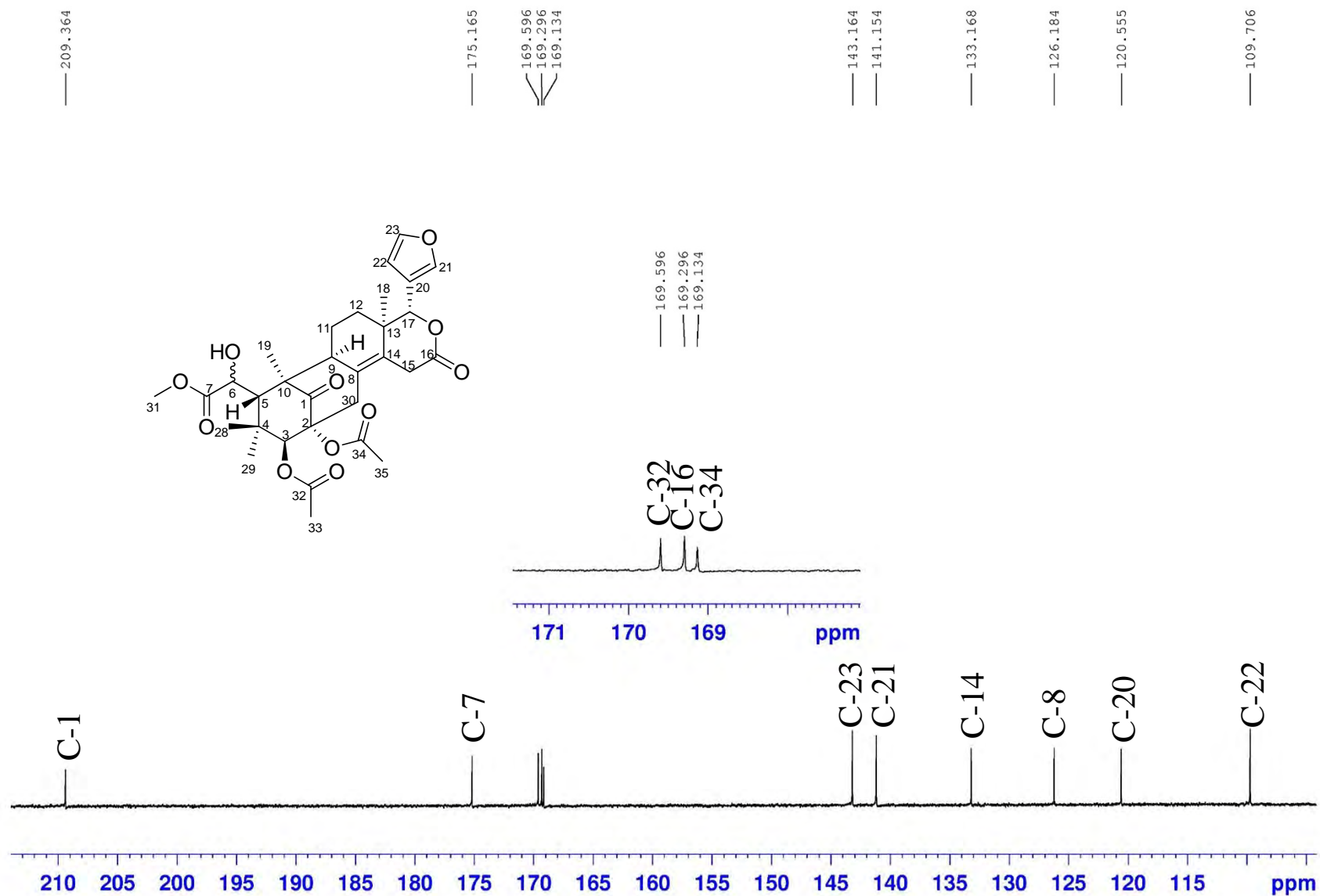
$^1\text{H}$  NMR (400 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



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

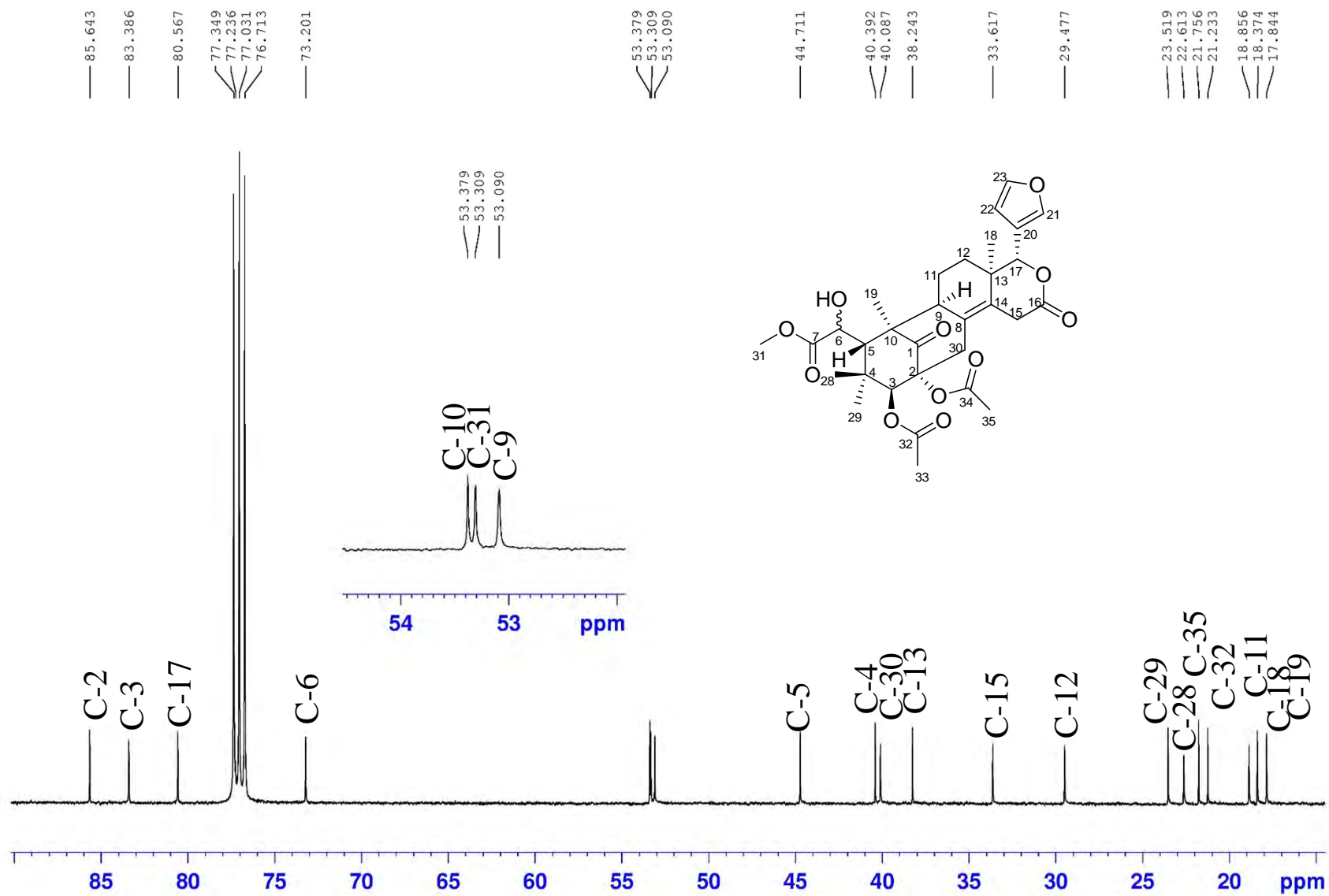


$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **6** in  $\text{CDCl}_3$

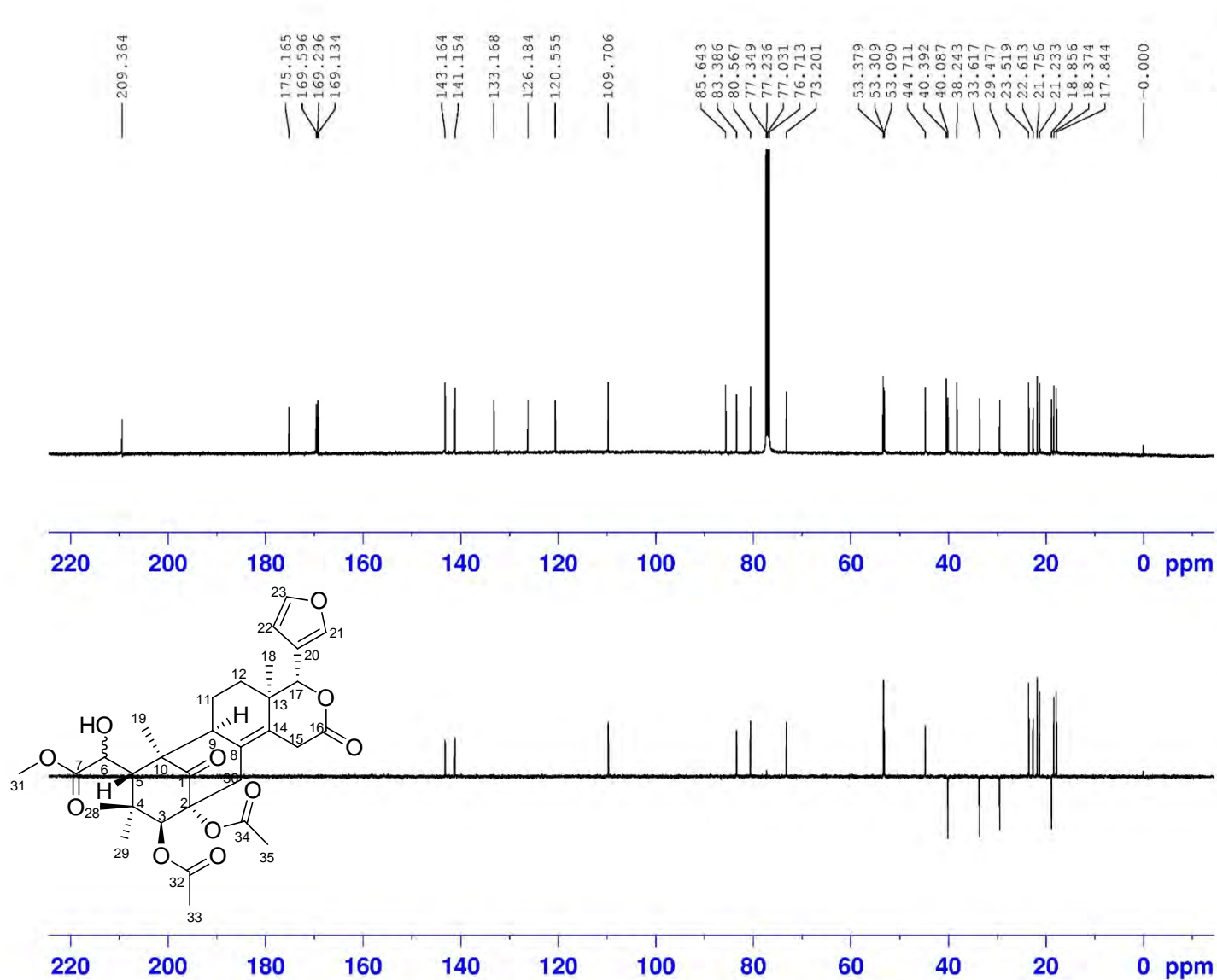




$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



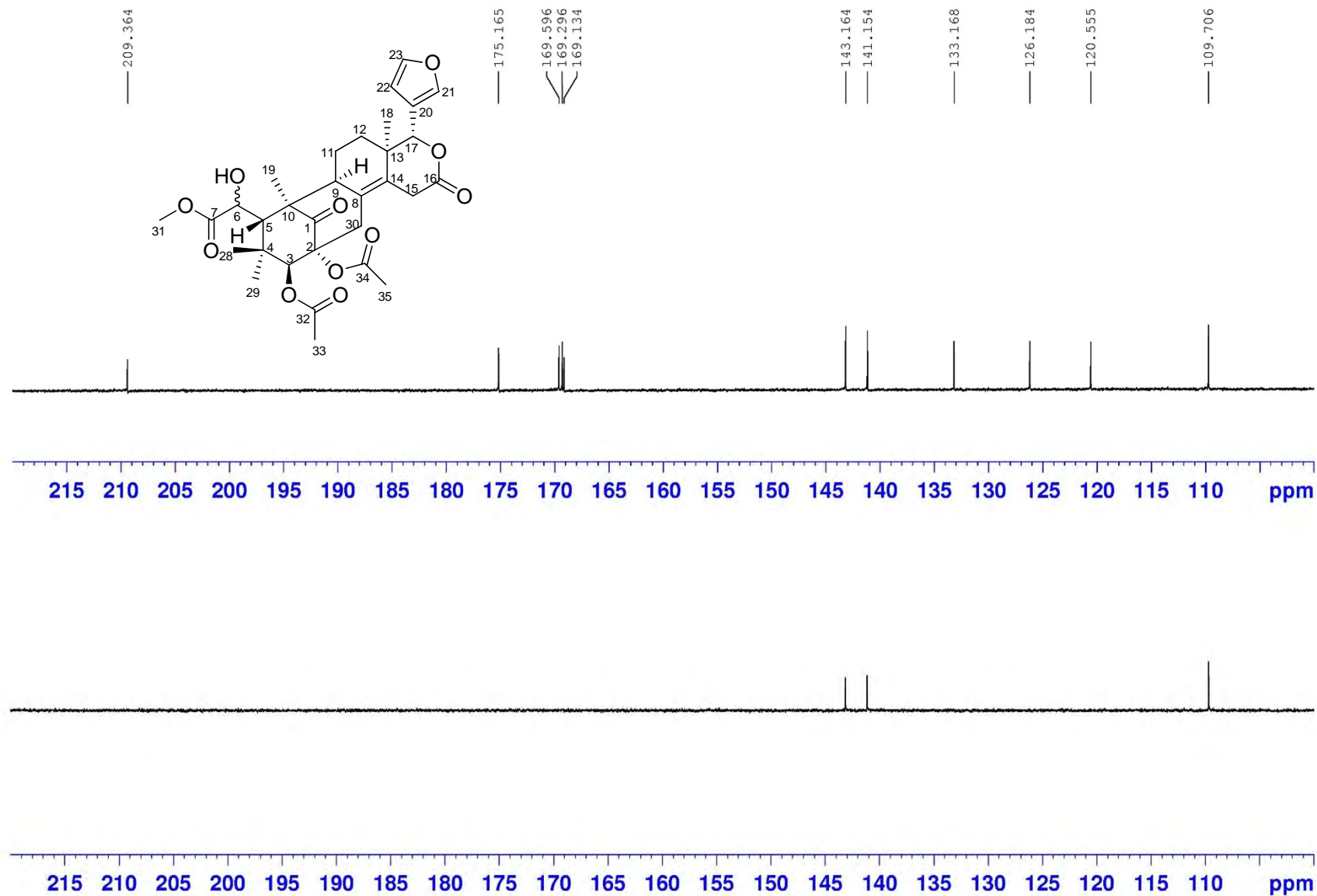
```

NAME          lws-80-1
EXPNO         3
PROCNO        1
Date_         20151006
Time          0.23
INSTRUM       spect
PROBHD        5 mm CPBBO BB
PULPROG       deptsp135
TD            65536
SOLVENT       CDCl3
NS            300
DS            4
SWH           24038.461 F
FIDRES        0.366798 F
AQ            1.3631988 s
RG            130.26
DW            20.800 s
DE            18.00 s
TE            297.0 F
CNST2         145.0000000
D1            2.00000000 s
D2            0.00344828 s
D12           0.00002000 s
TD0           1
    
```

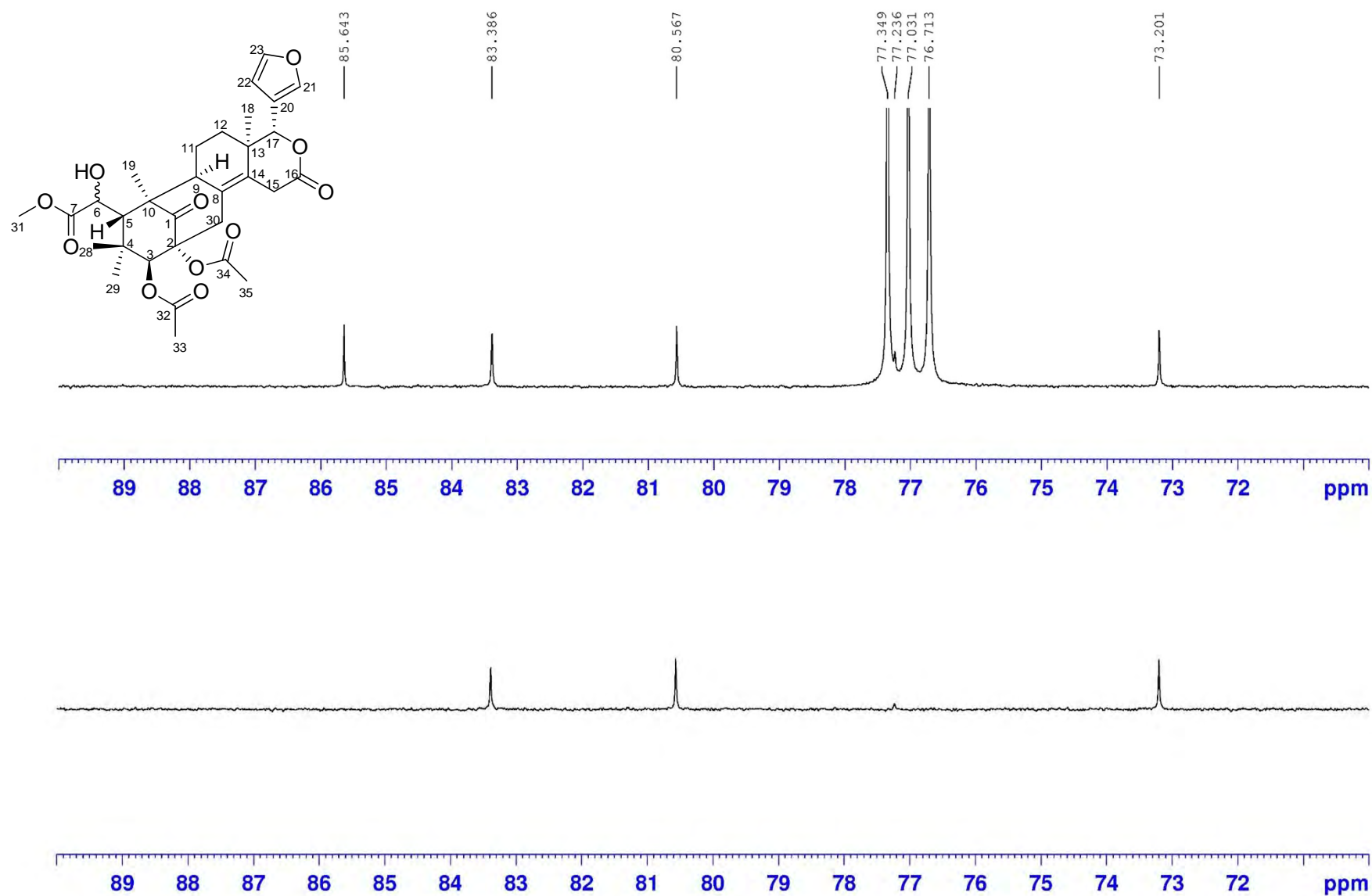
```

===== CHANNEL f1 =====
SFO1          100.6233324 M
NUC1          13C
P1            10.00 s
P13           2000.00 s
SI            32768
SF            100.6127690 M
WDW           EM
SSB           0
LB            1.00 F
GB            0
PC            1.40
    
```

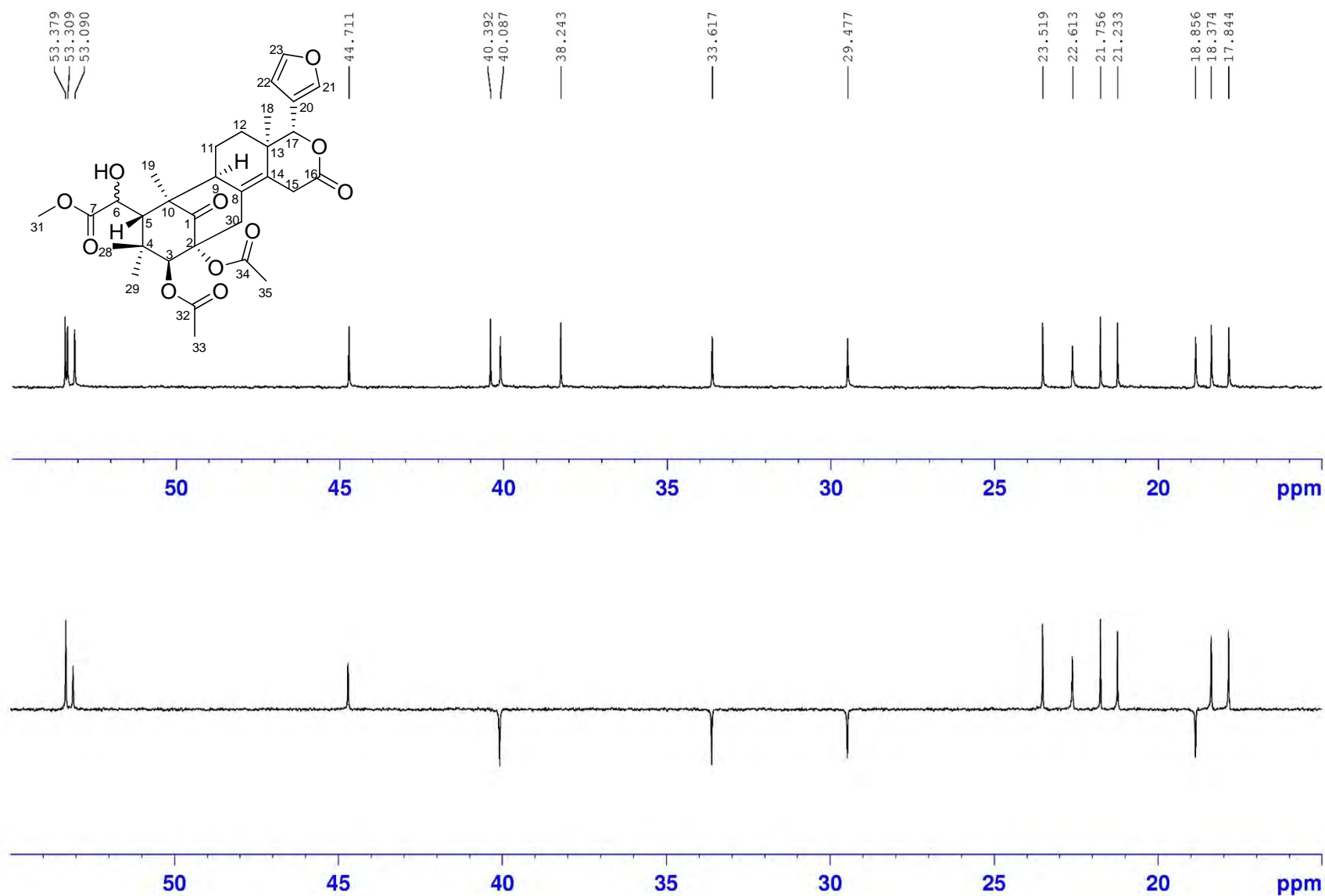
# DEPT135 (100 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



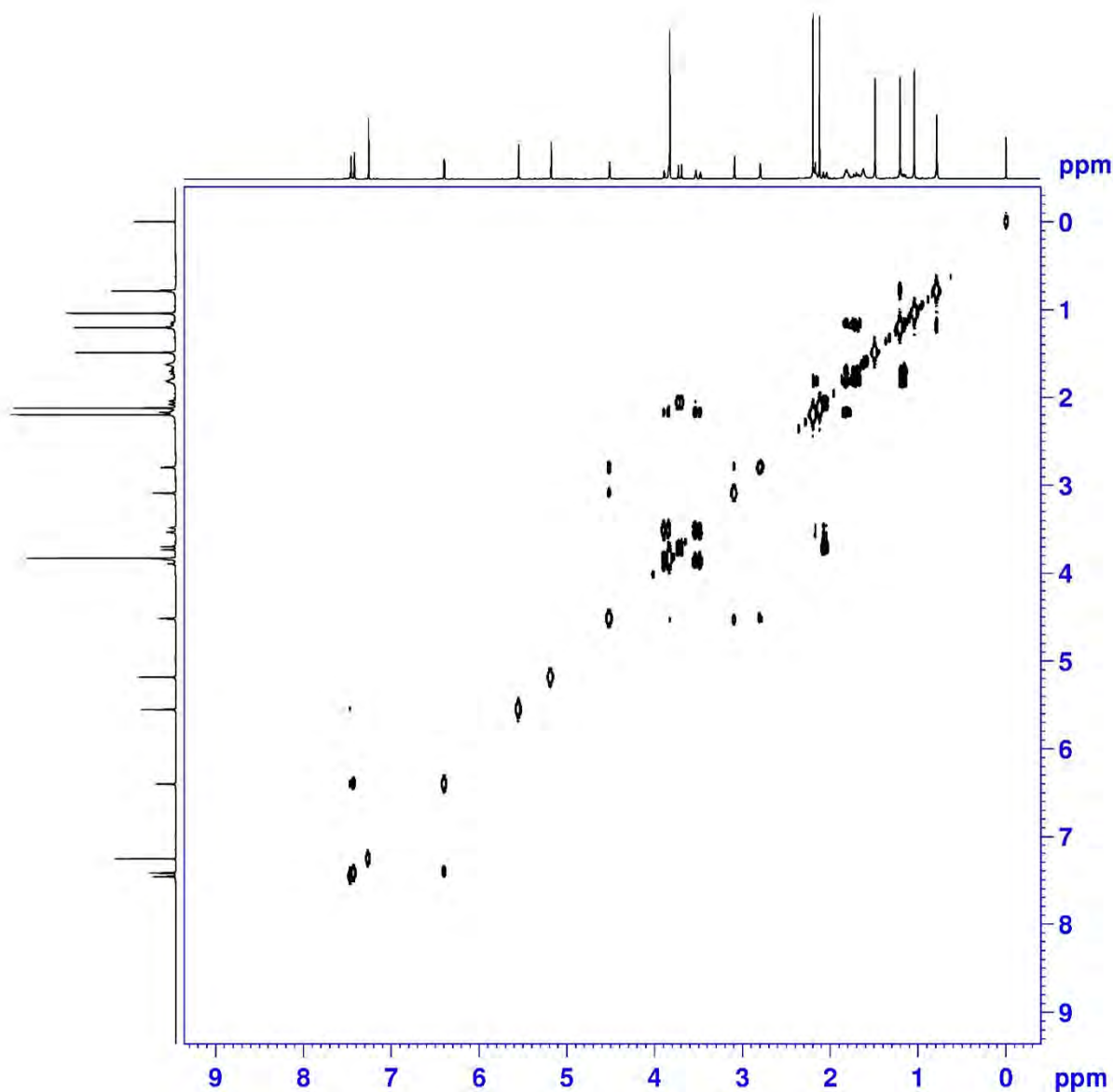
# DEPT135 (100 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



# DEPT135 (100 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **6** in $\text{CDCl}_3$

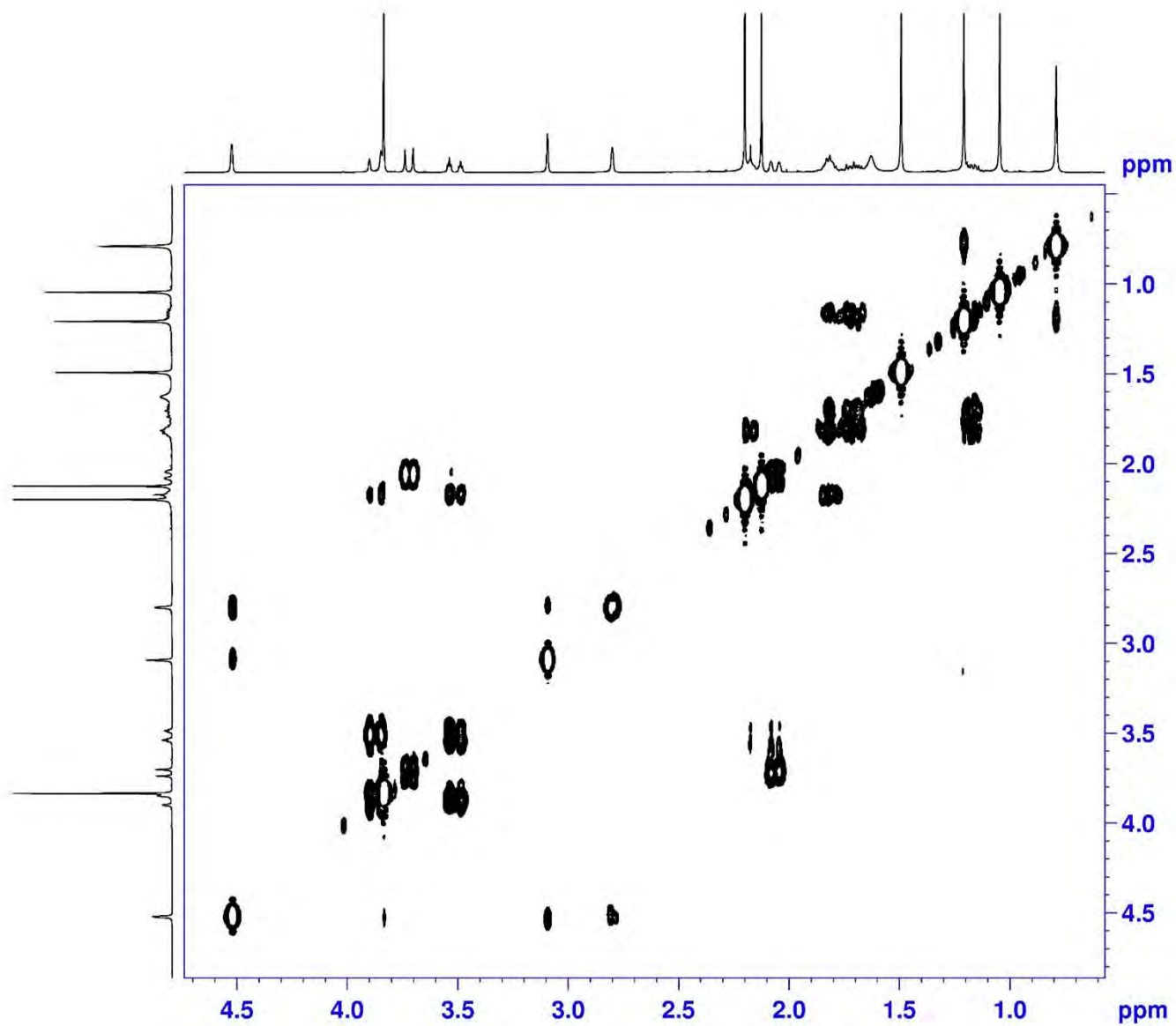


```
NAME          lws-80-1
EXPNO          104
PROCNO         1
Date_          20151027
Time           3.29
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        cosygpppqf
TD             2048
SOLVENT        CDCl3
NS              8
DS              8
SWH            3906.250 Hz
FIDRES         1.907349 Hz
AQ             0.2621940 sec
RG             208.5
DW            128.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00000300 sec
D1             1.89678097 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00025600 sec
```

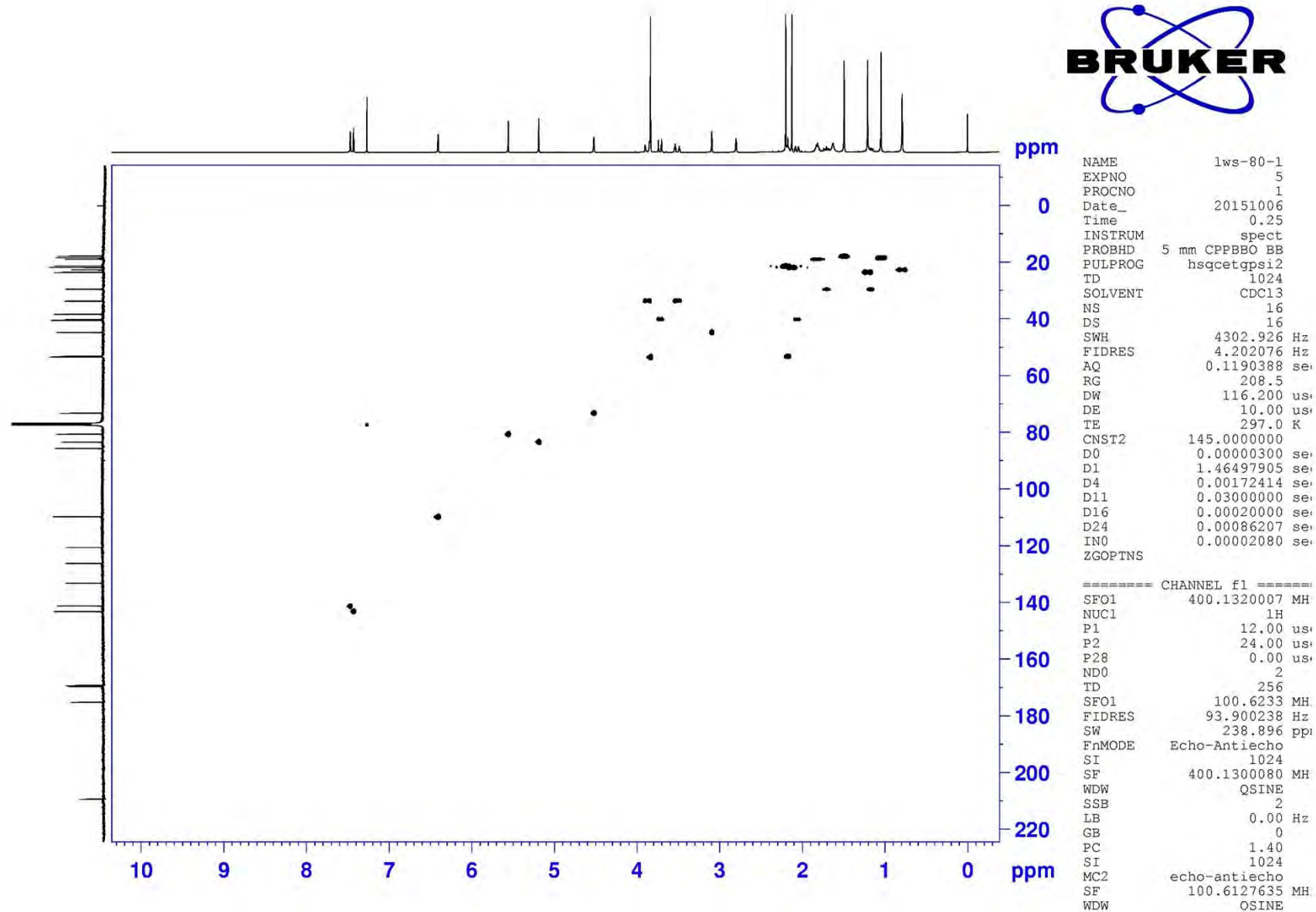
```
===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1           1H
P0             12.00 usec
P1             12.00 usec
P17           2500.00 usec
ND0            1
TD             128
SFO1          400.1318 MHz
FIDRES         30.517578 Hz
SW             9.762 ppm
FnmODE         QF
SI             1024
SF            400.1300098 MHz
WDW            QSINE
SSB            0
LB             0.00 Hz
GB            0
PC             1.40
SI             1024
MC2            QF
SF            400.1300098 MHz
WDW            QSINE
---
```



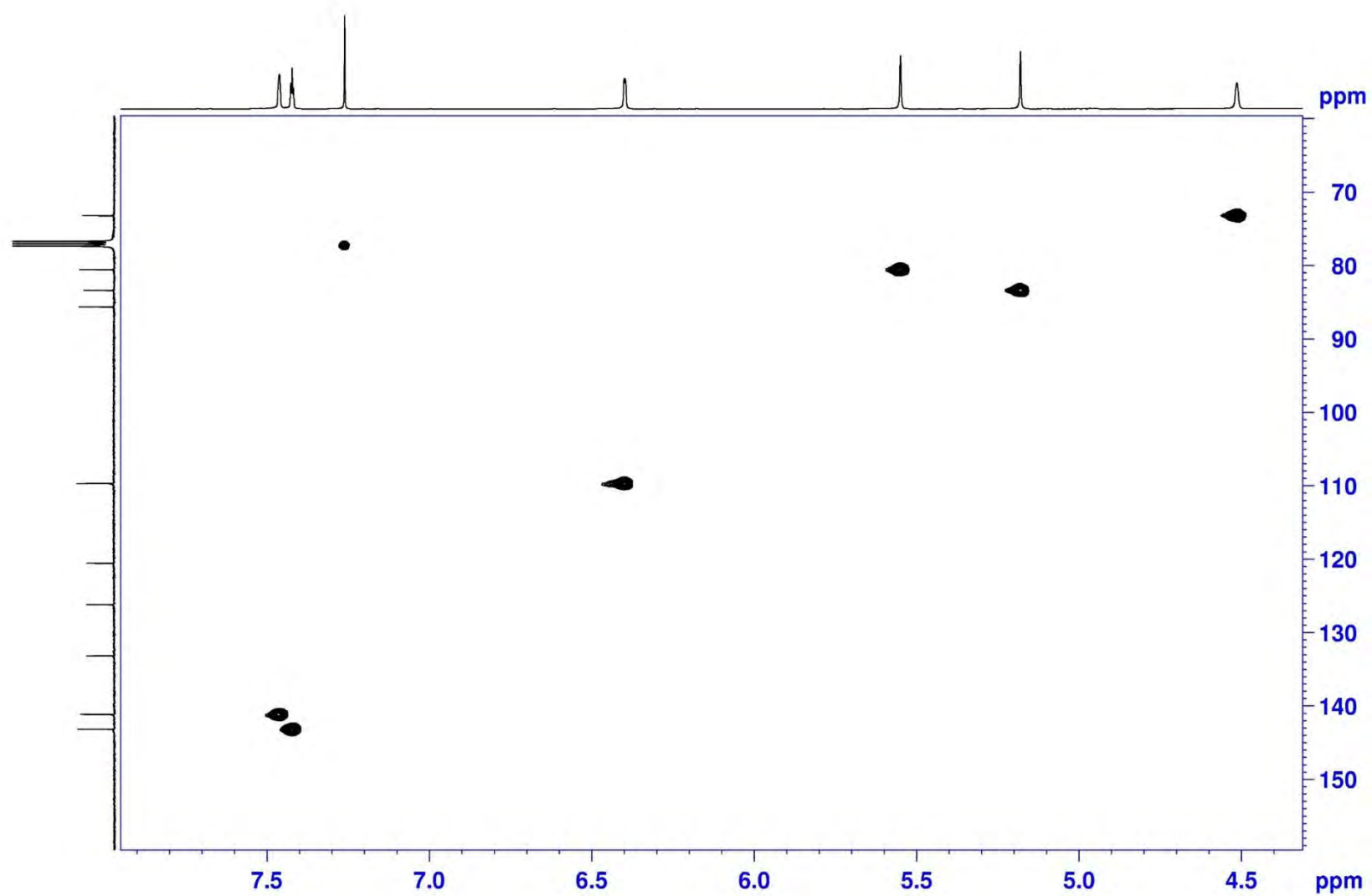
<sup>1</sup>H-<sup>1</sup>H COSY (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



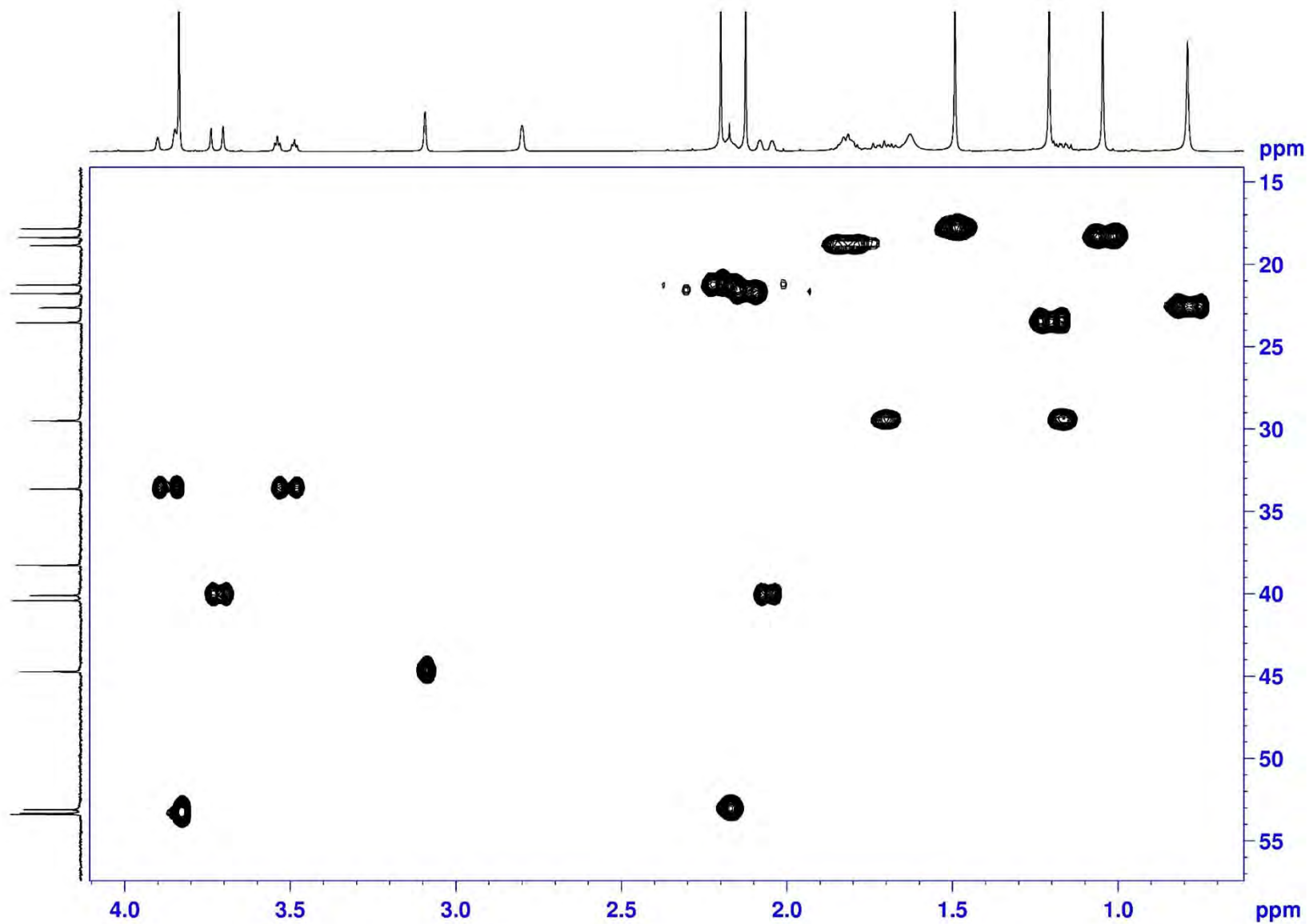
# HSQC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



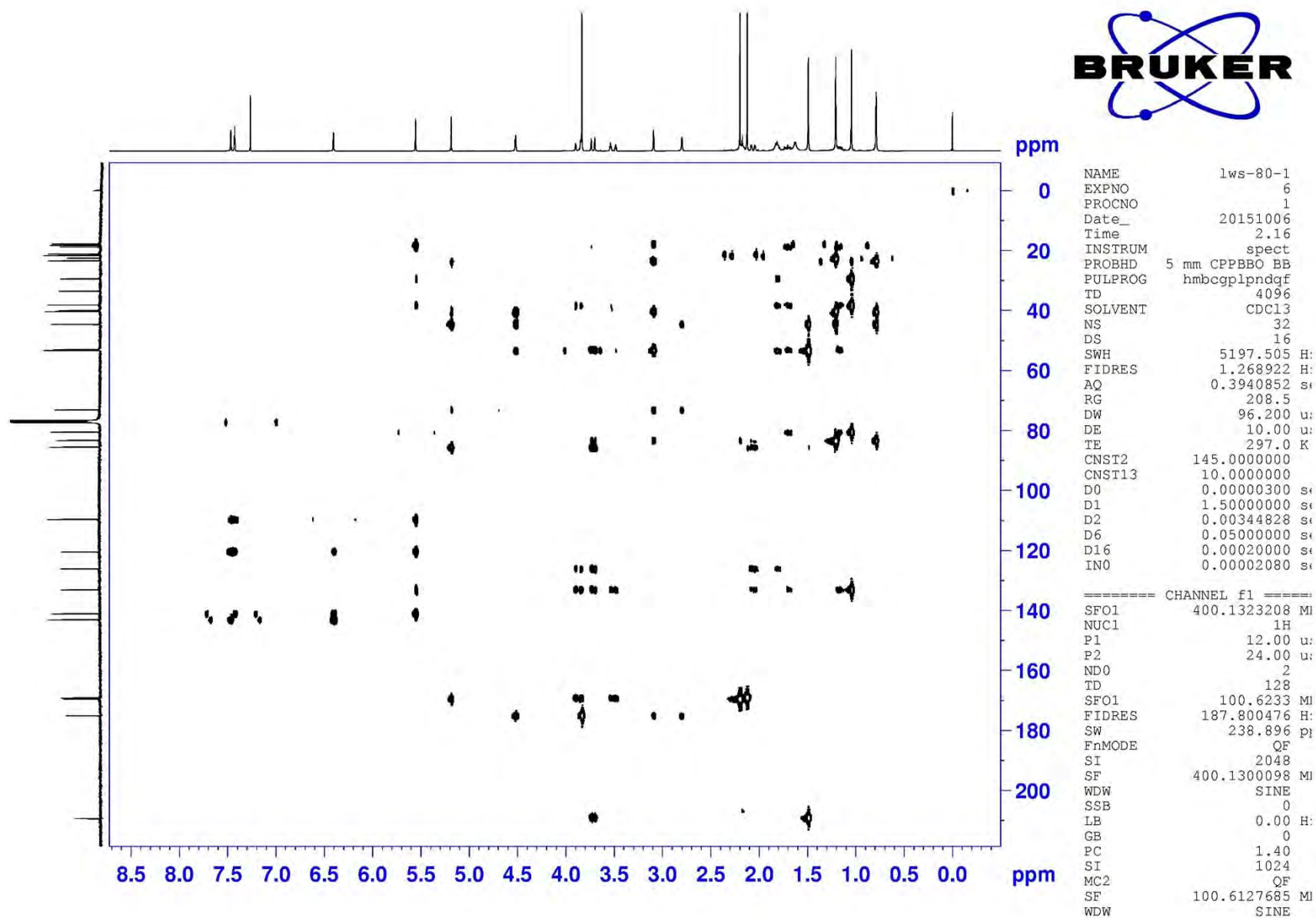
HSQC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



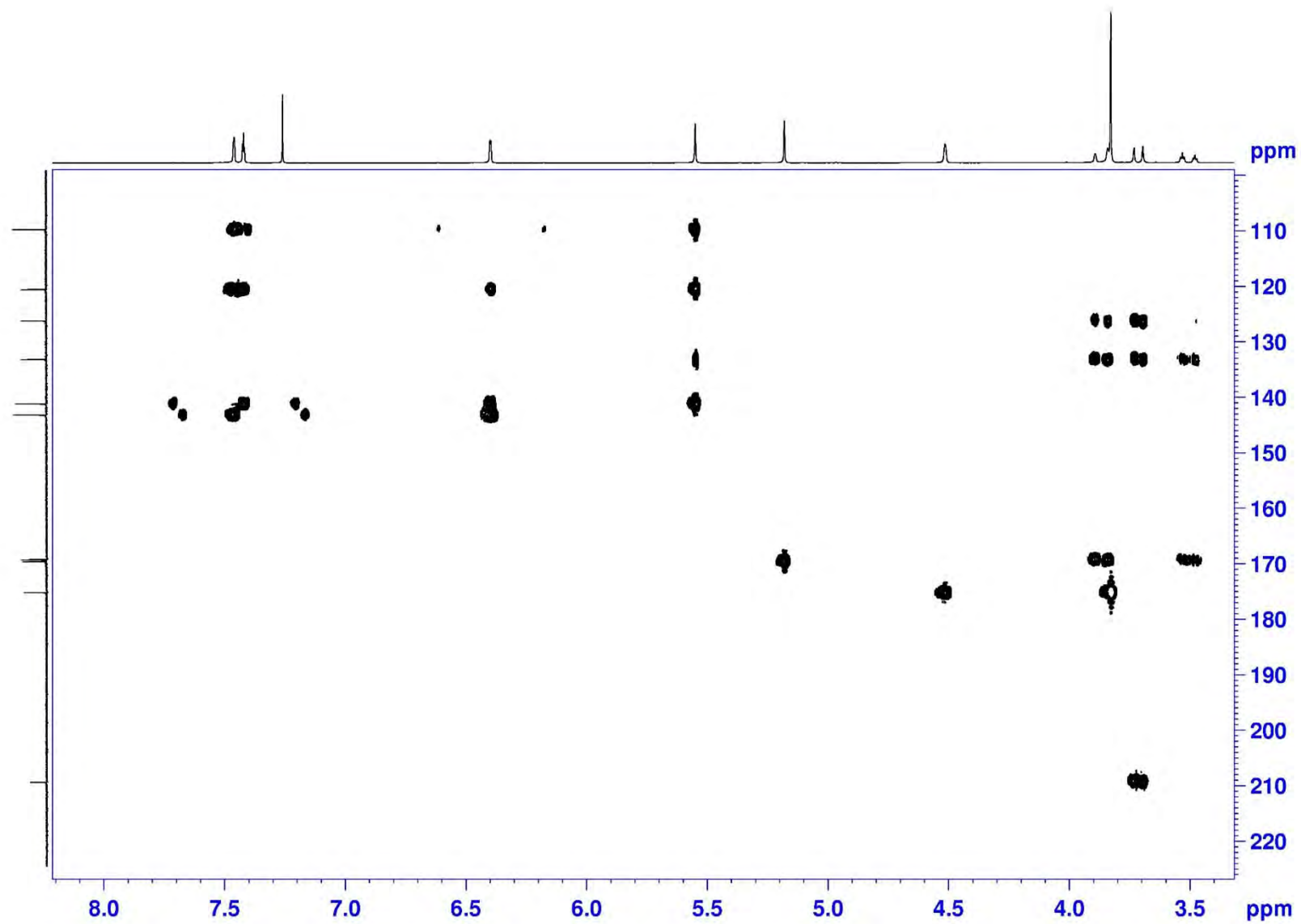
HSQC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



# HMBC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>

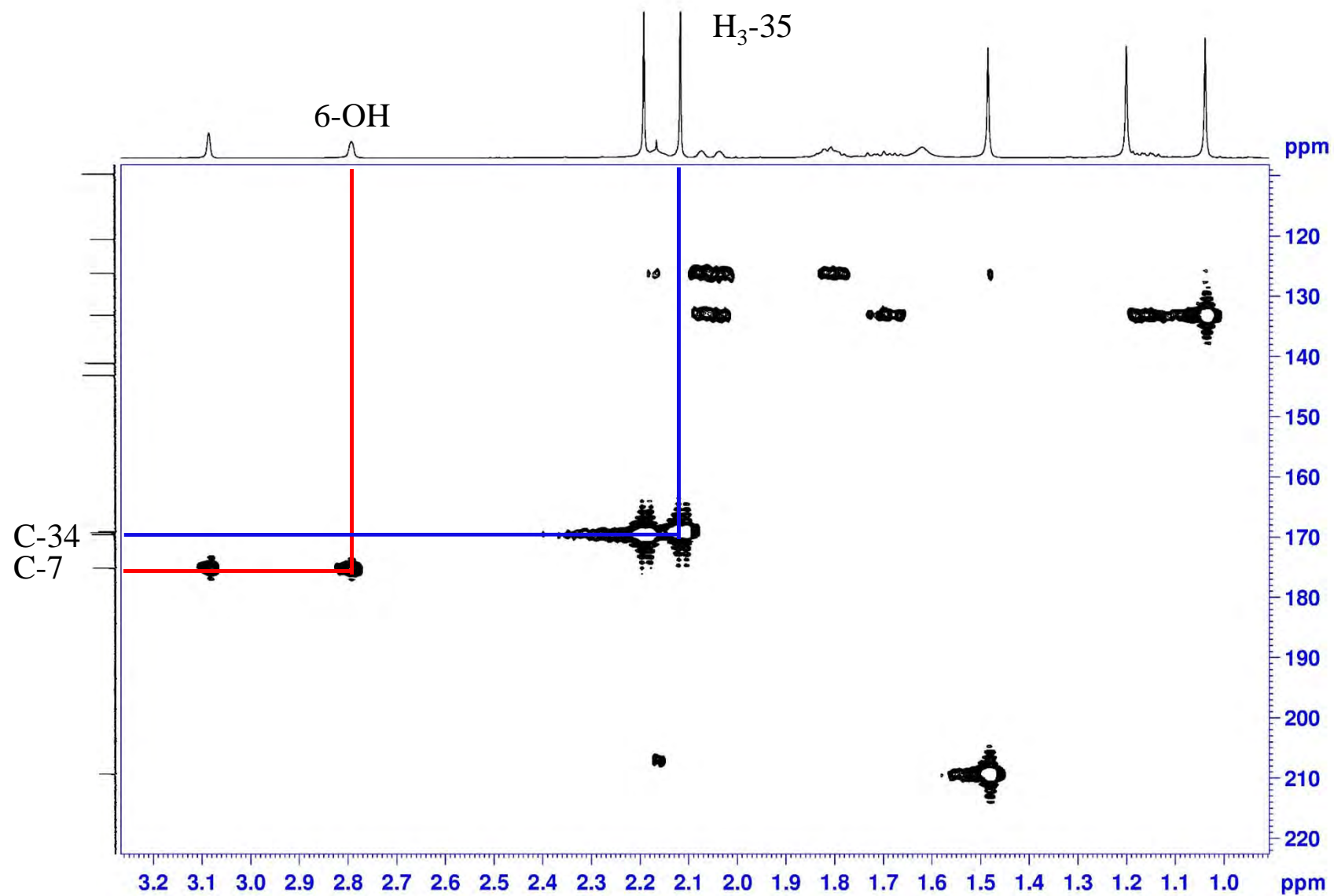


HMBC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>

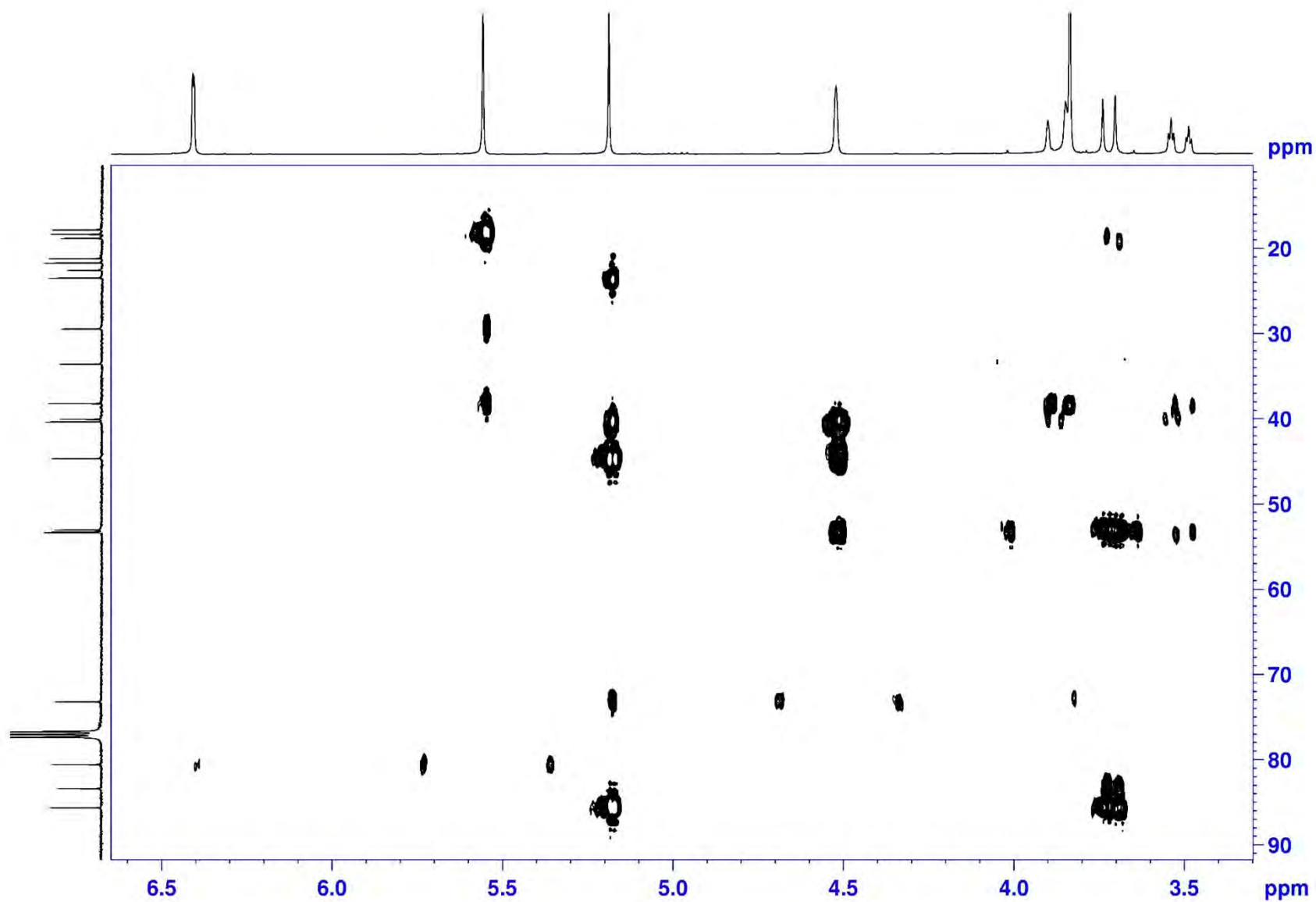




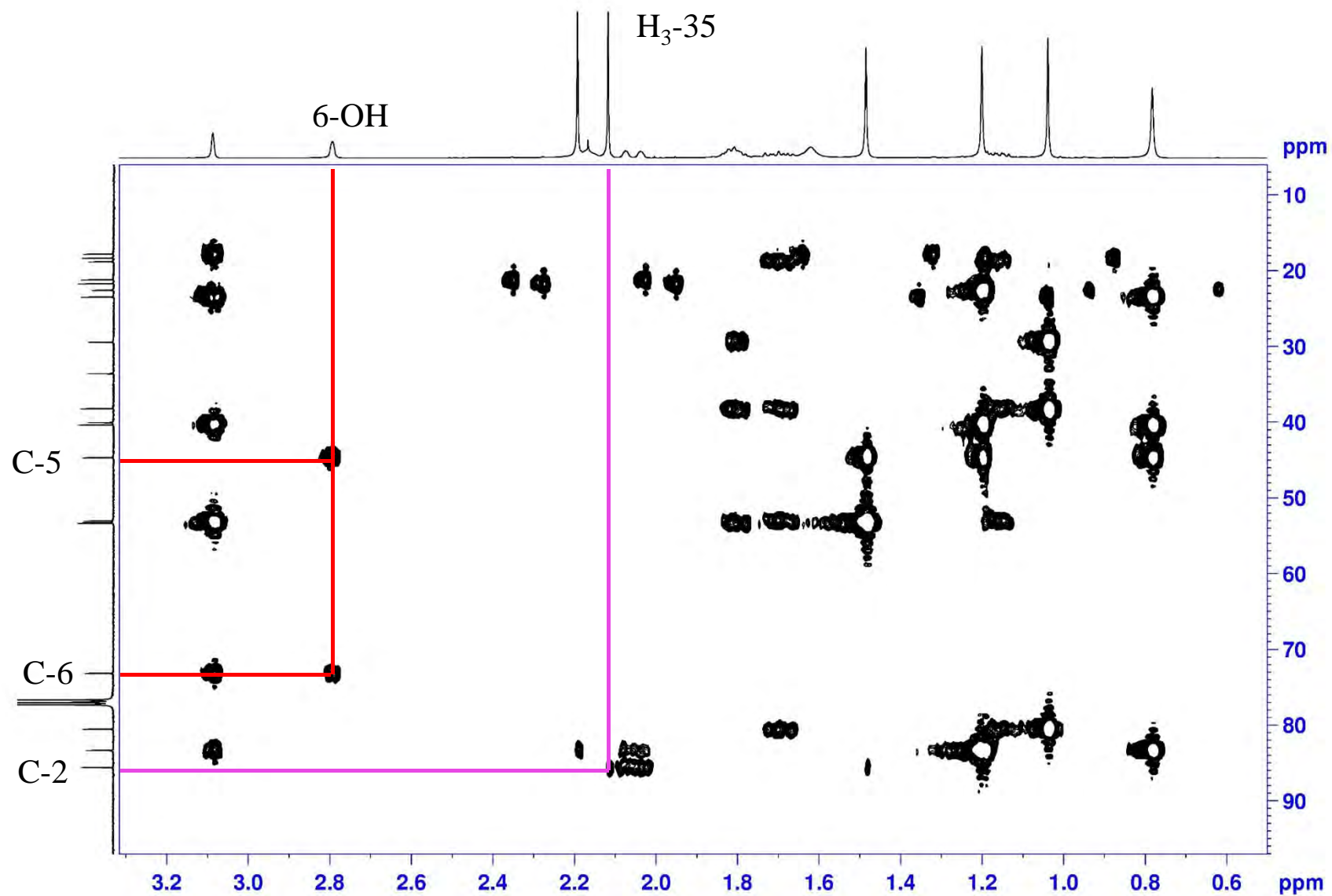
HMBC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



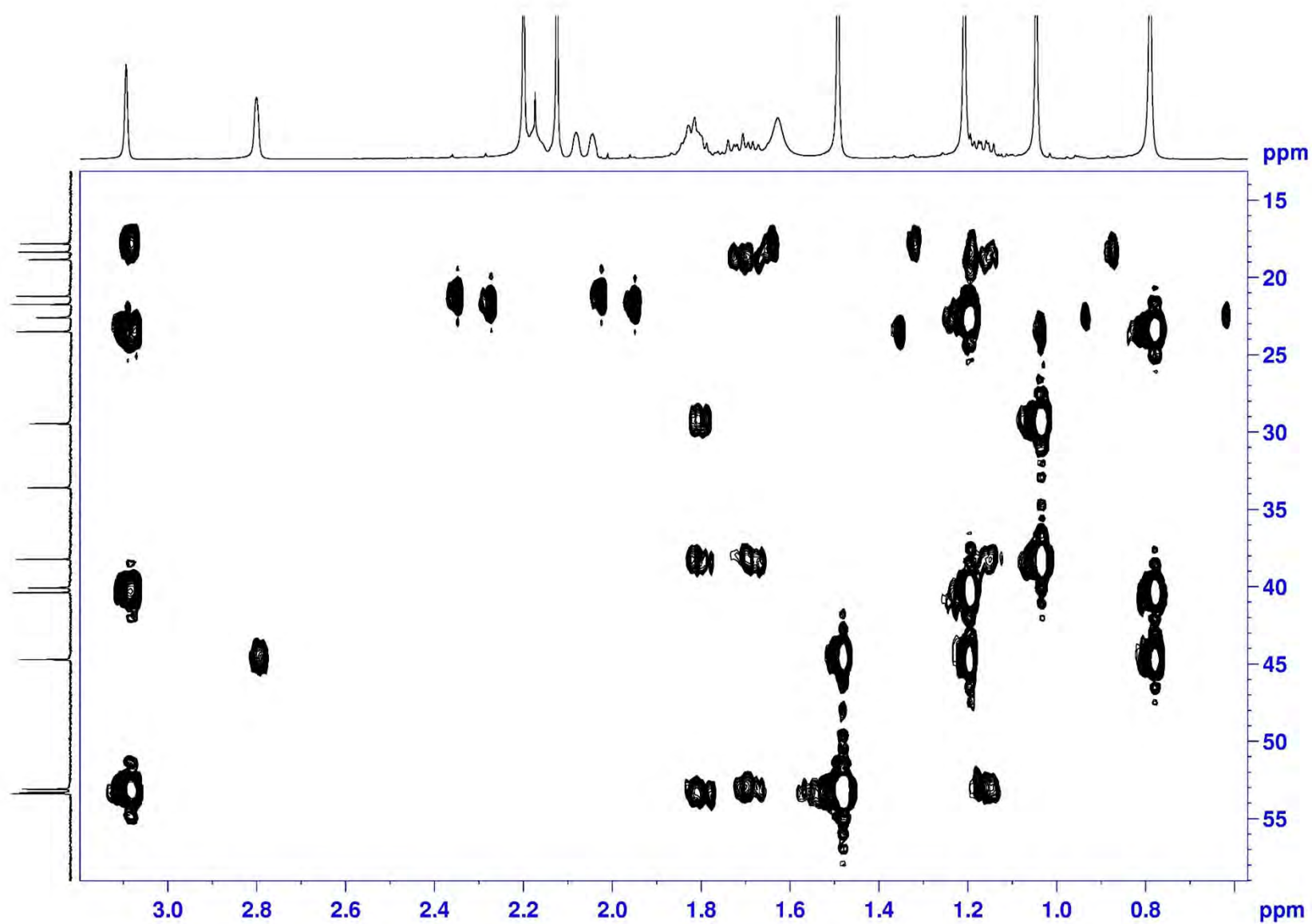
HMBC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



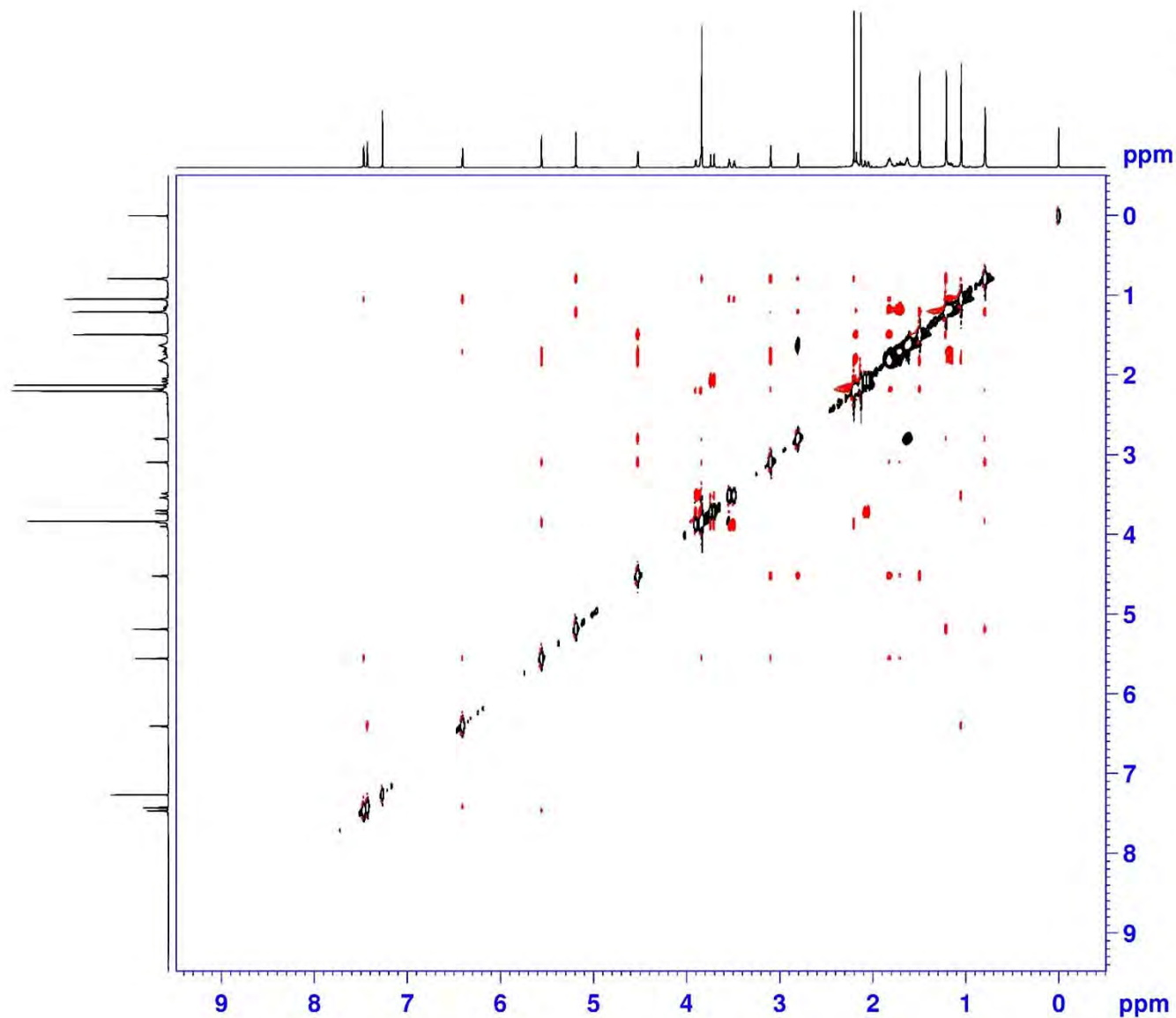
HMBC (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>



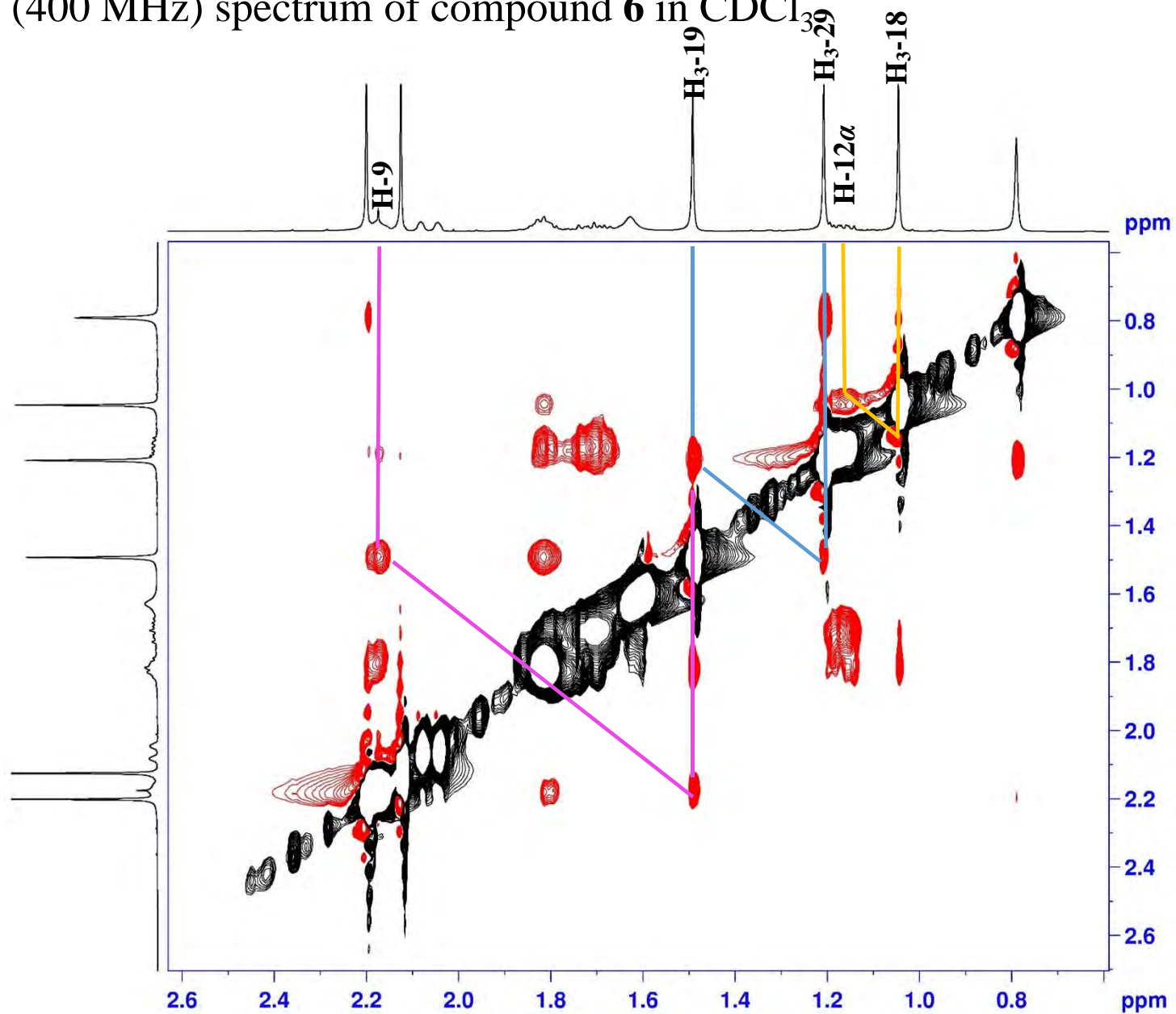
```

NAME          lws-80-1
EXPNO         107
PROCNO        1
Date_         20151027
Time          4.10
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpphph
TD            2048
SOLVENT       CDCl3
NS            32
DS            32
SWH           4000.000 H:
FIDRES        1.953125 H:
AQ            0.2560500 S:
RG            208.5
DW            125.000 u:
DE            10.00 u:
TE            297.0 K
D0            0.00010972 S:
D1            1.99385595 S:
D8            0.30000001 S:
D11           0.03000000 S:
D12           0.00002000 S:
D16           0.00020000 S:
IN0           0.00025000 S:
  
```

```

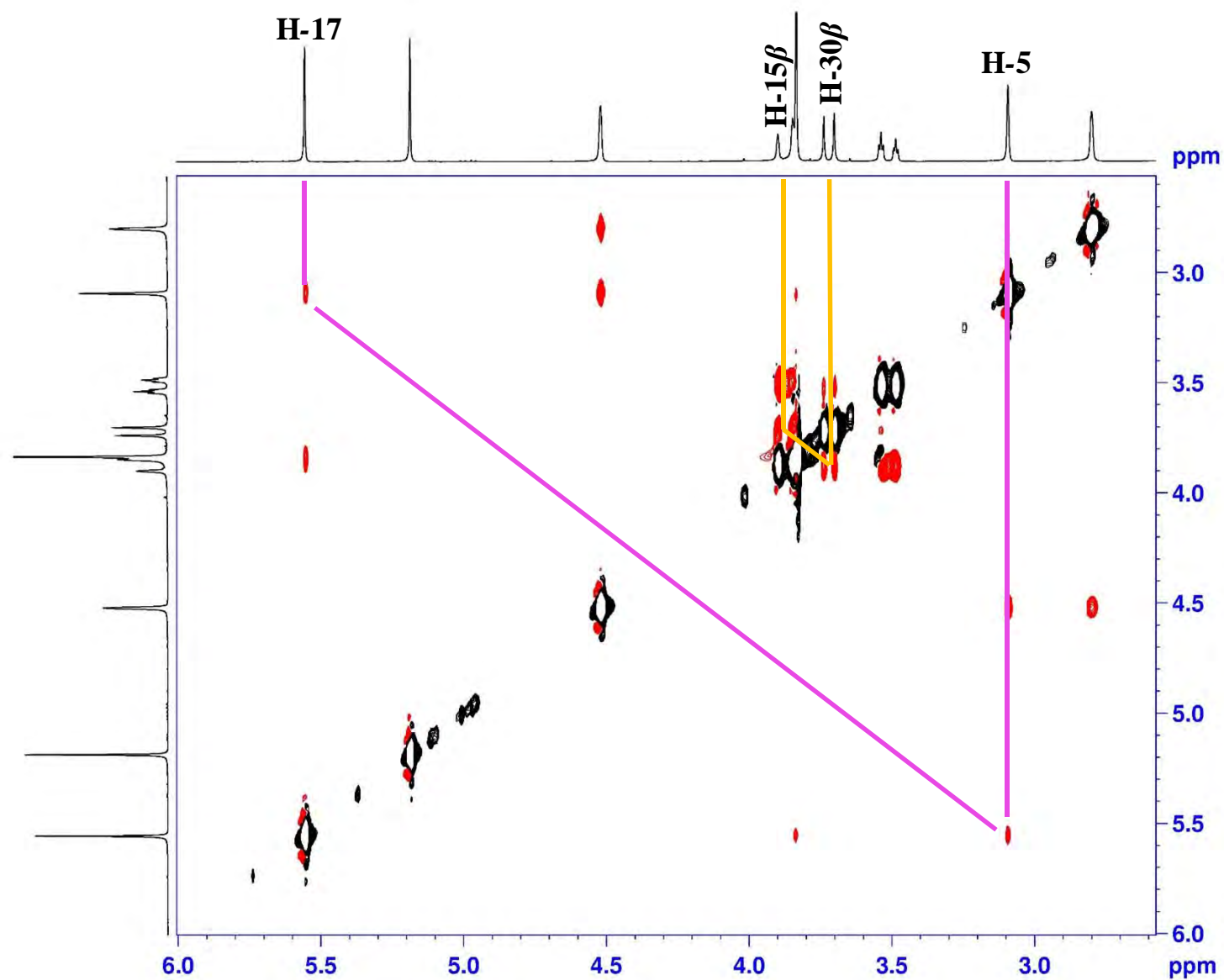
===== CHANNEL f1 =====
SFO1         400.1318006 MHz
NUC1          1H
P1            12.00 u:
P2            24.00 u:
P17           2500.00 u:
ND0           1
TD            256
SFO1         400.1318 MHz
FIDRES        15.625000 H:
SW            9.997 MHz
FnMODE        States-TPPI
SI            1024
SF            400.1300067 MHz
WDW           QSINE
SSB           2
LB            0.00 H:
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            400.1300067 MHz
WDW           QSINE
  
```

NOESY (400 MHz) spectrum of compound **6** in CDCl<sub>3</sub>

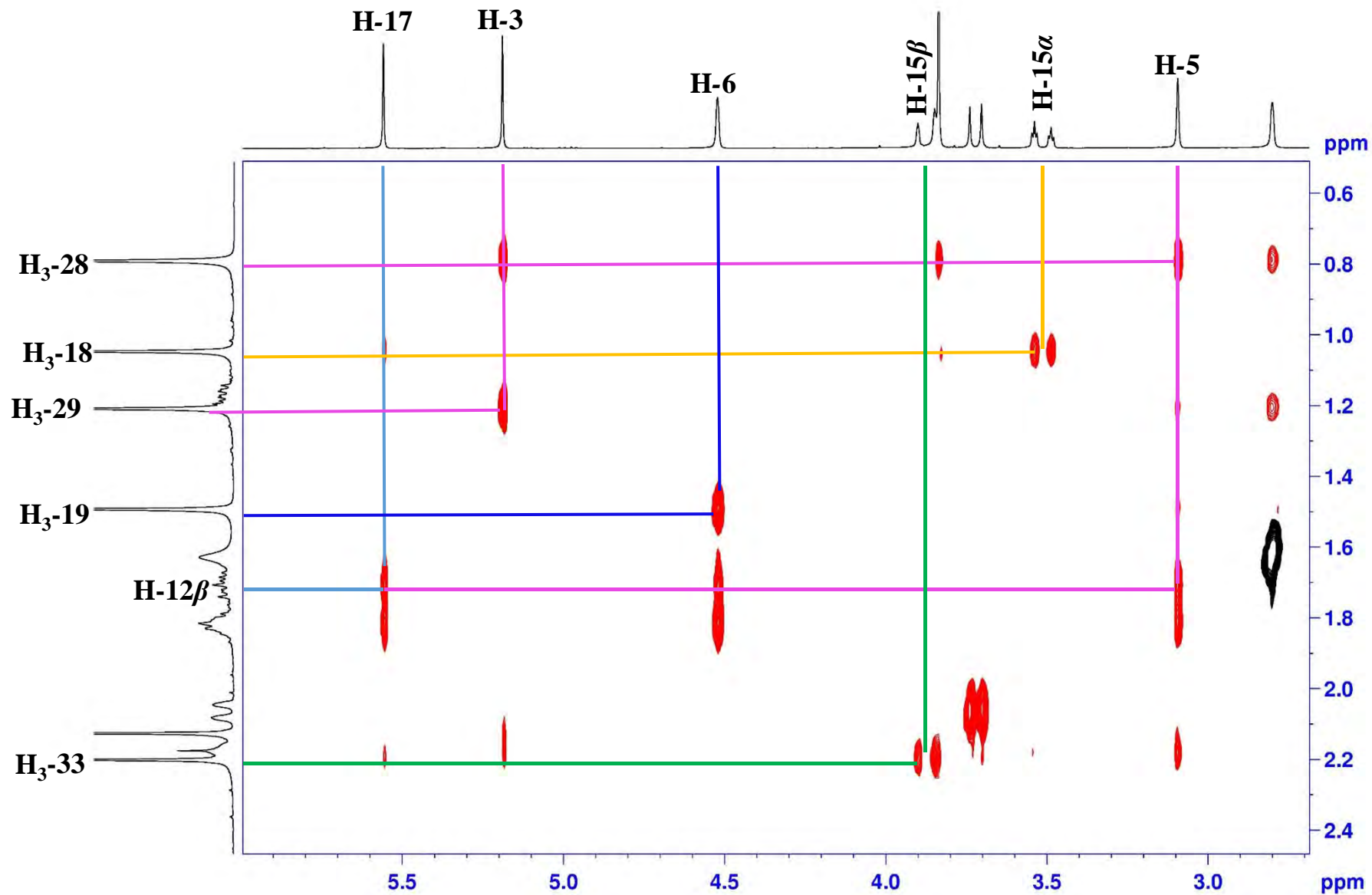




NOESY (400 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **6** in  $\text{CDCl}_3$



# HRESIMS for compound 7

## Mass Spectrum SmartFormula Report

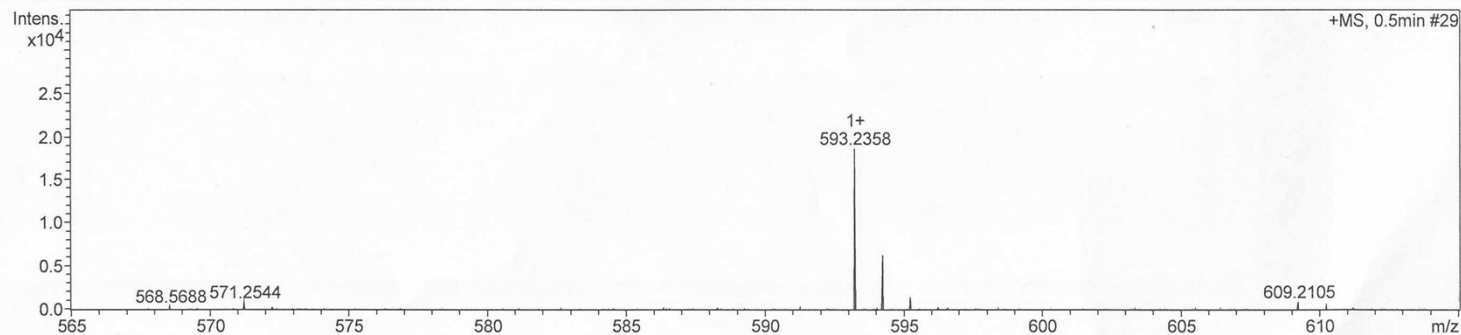
### Analysis Info

Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-34\_pos\_83\_01\_2759.d  
 Method LC MS\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name zhangjianzhi\_zjz-34\_pos  
 Comment

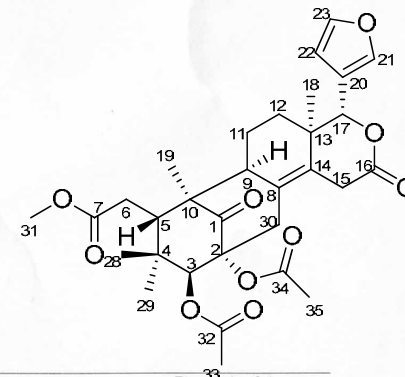
Acquisition Date 12/14/2016 2:51:51 PM  
 Operator SCSIO  
 Instrument maXis 255552.00029

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
571.254411	1	C31H39O10	100.00	571.253774	-1.1	-0.6	47.1	12.5	even	ok
593.235801	1	C31H38NaO10	100.00	593.235718	-0.1	-0.1	3.4	12.5	even	ok
1163.482868	1	C62H76NaO20	100.00	1163.482216	-0.6	-0.7	35.0	24.5	even	ok



# HRESIMS for compound 7

## Generic Display Report

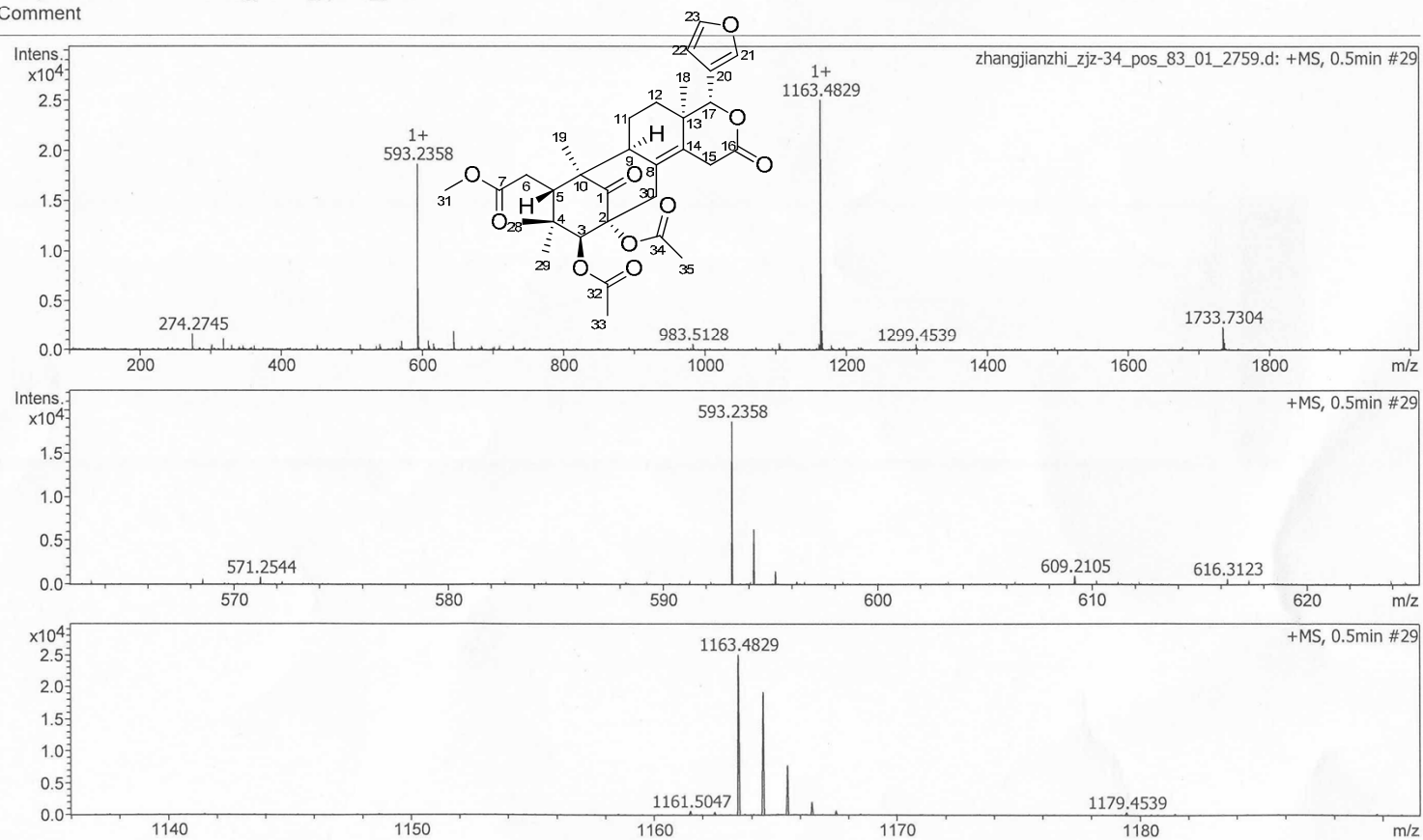
### Analysis Info

Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-34\_pos\_83\_01\_2759.d  
Method LC\_MS\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name zhangjianzhi\_zjz-34\_pos  
Comment

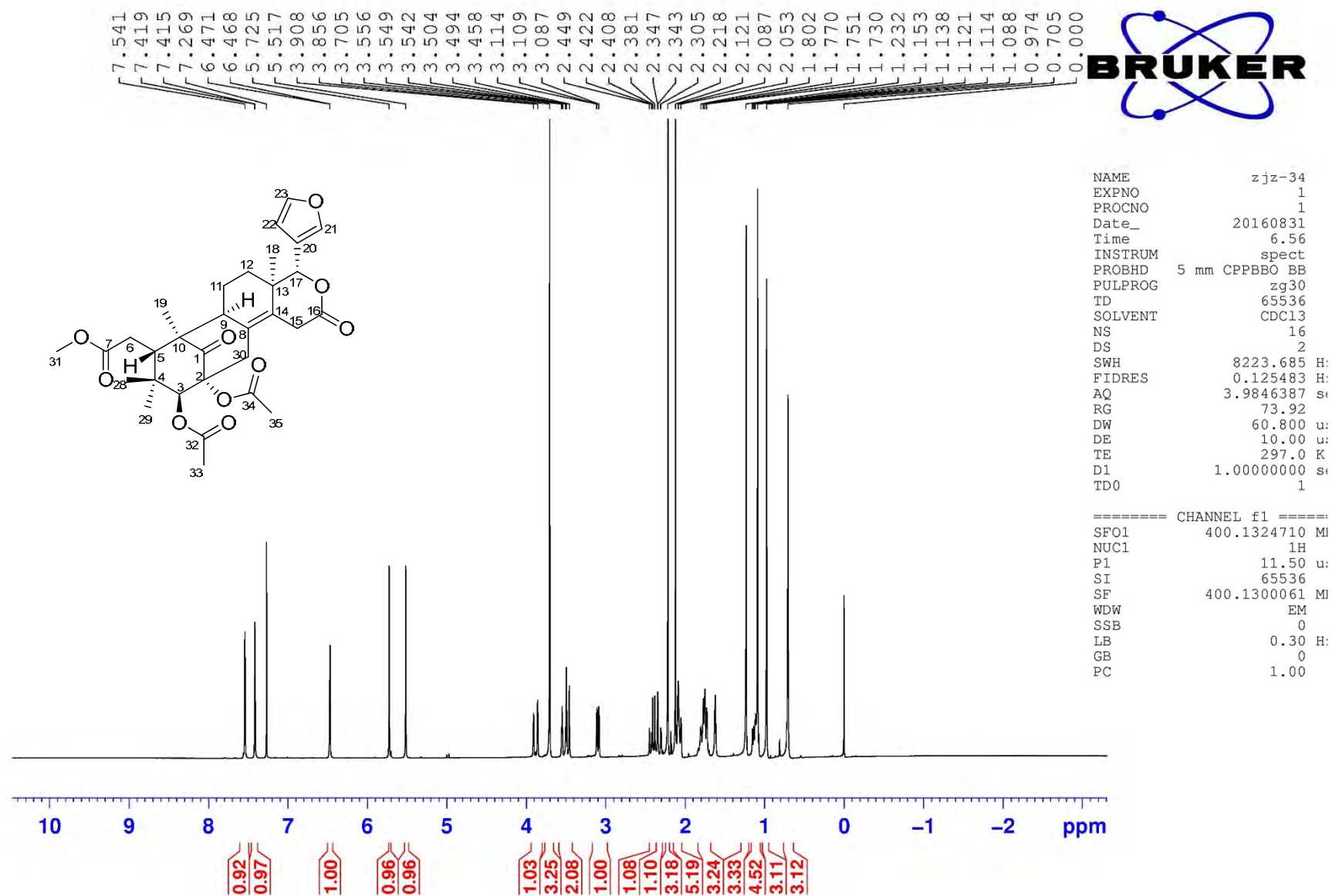
Acquisition Date 12/14/2016 2:51:51 PM

Operator SCSIO

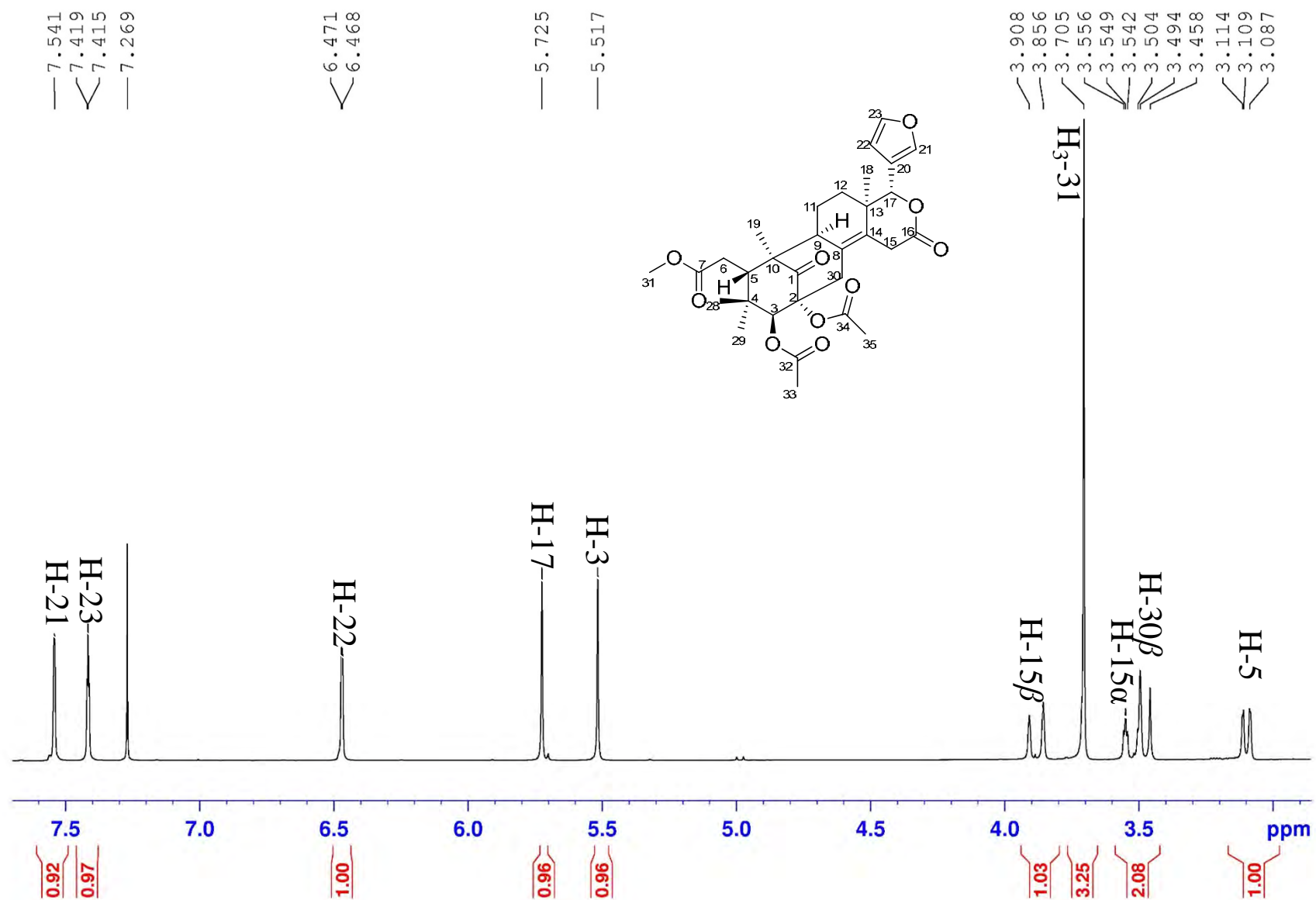
Instrument maXis



$^1\text{H}$  NMR (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$

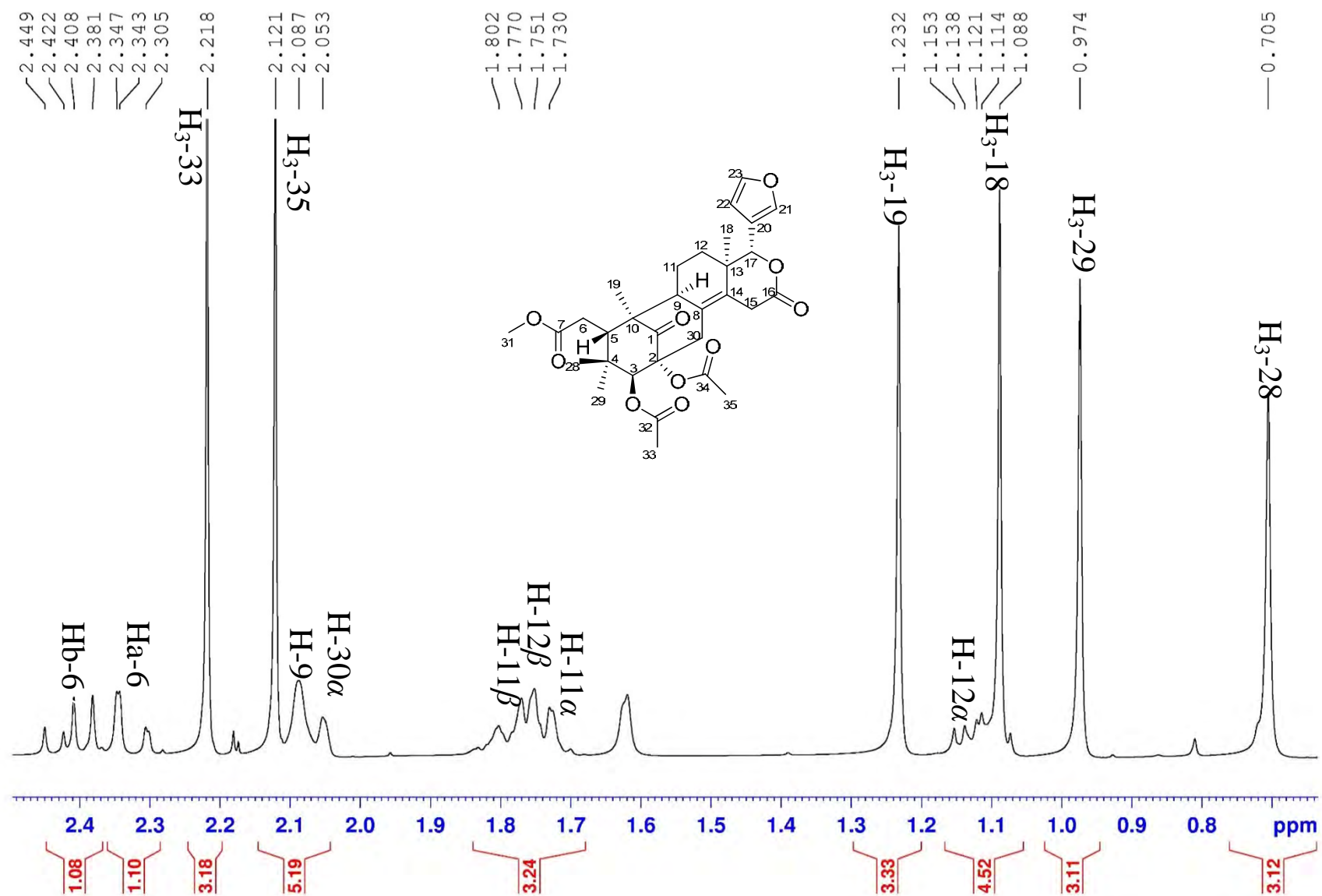


$^1\text{H}$  NMR (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$

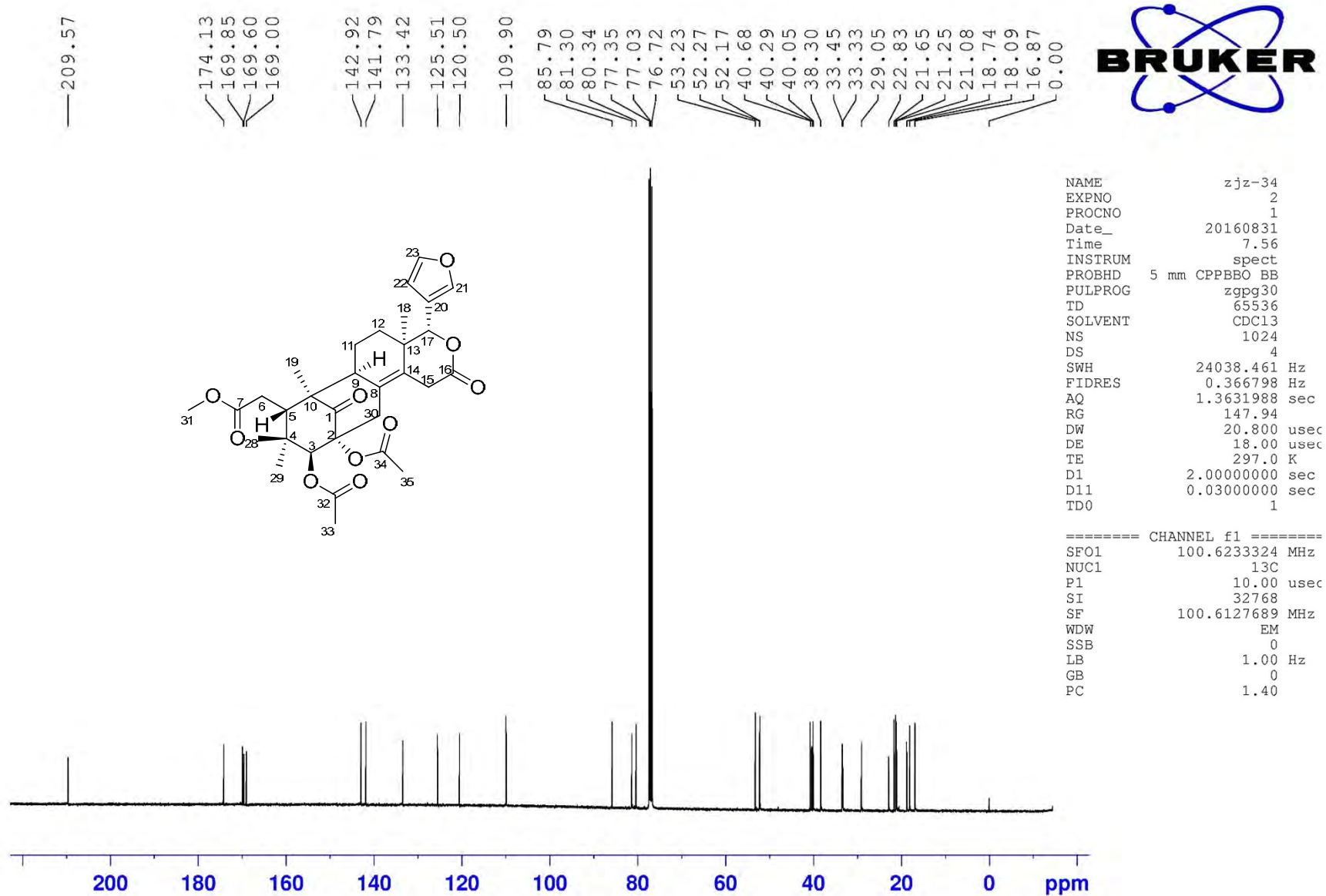




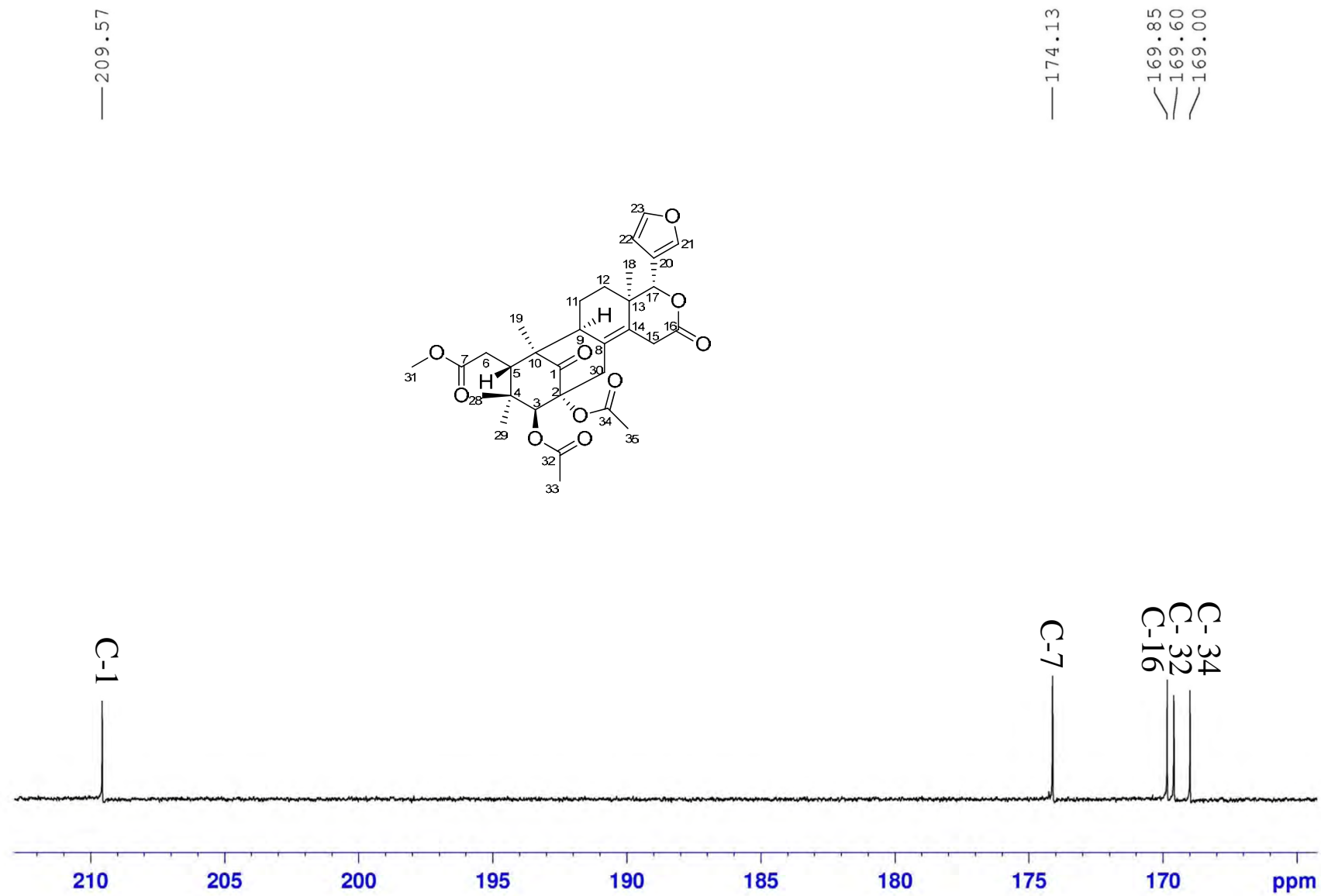
$^1\text{H}$  NMR (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



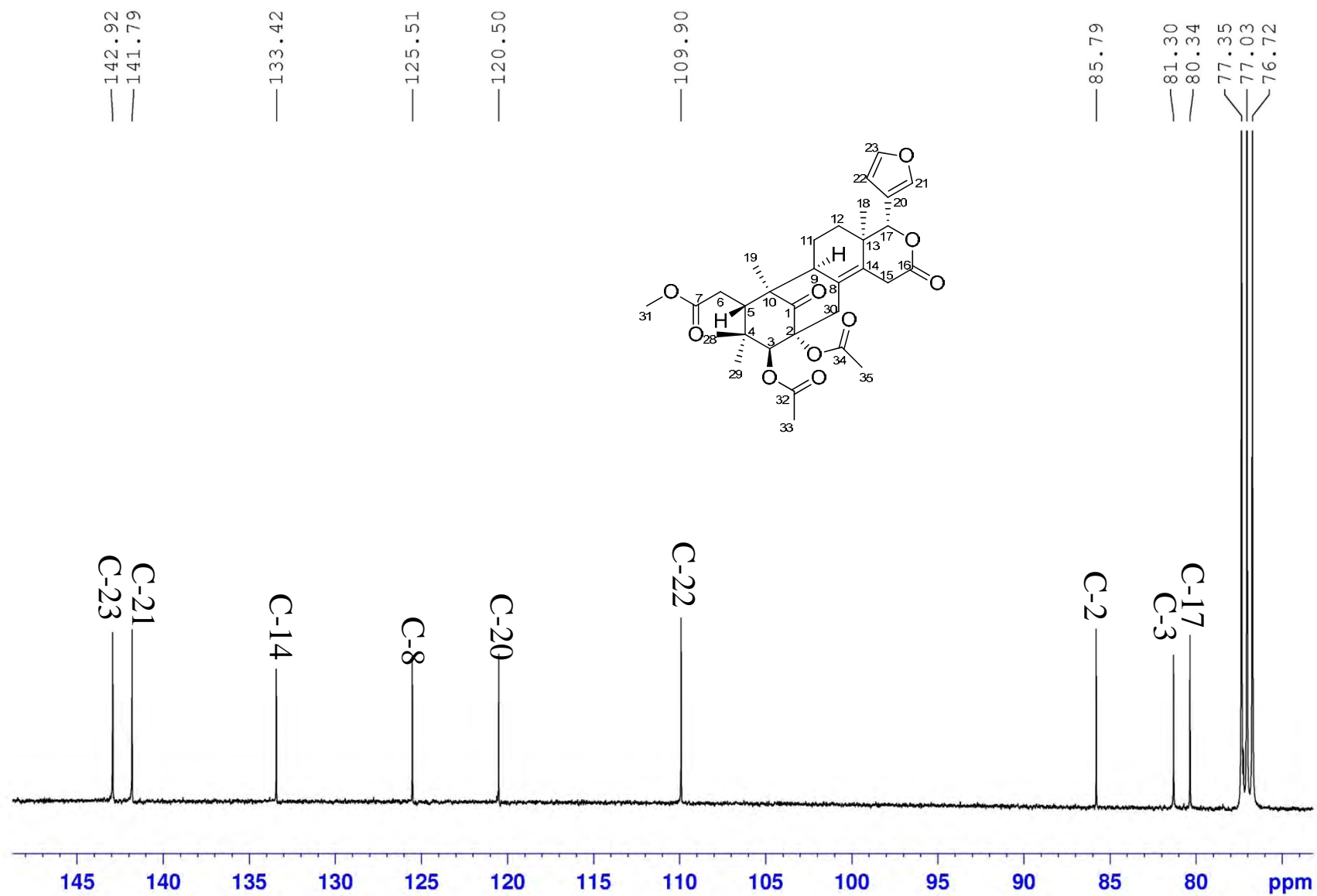
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



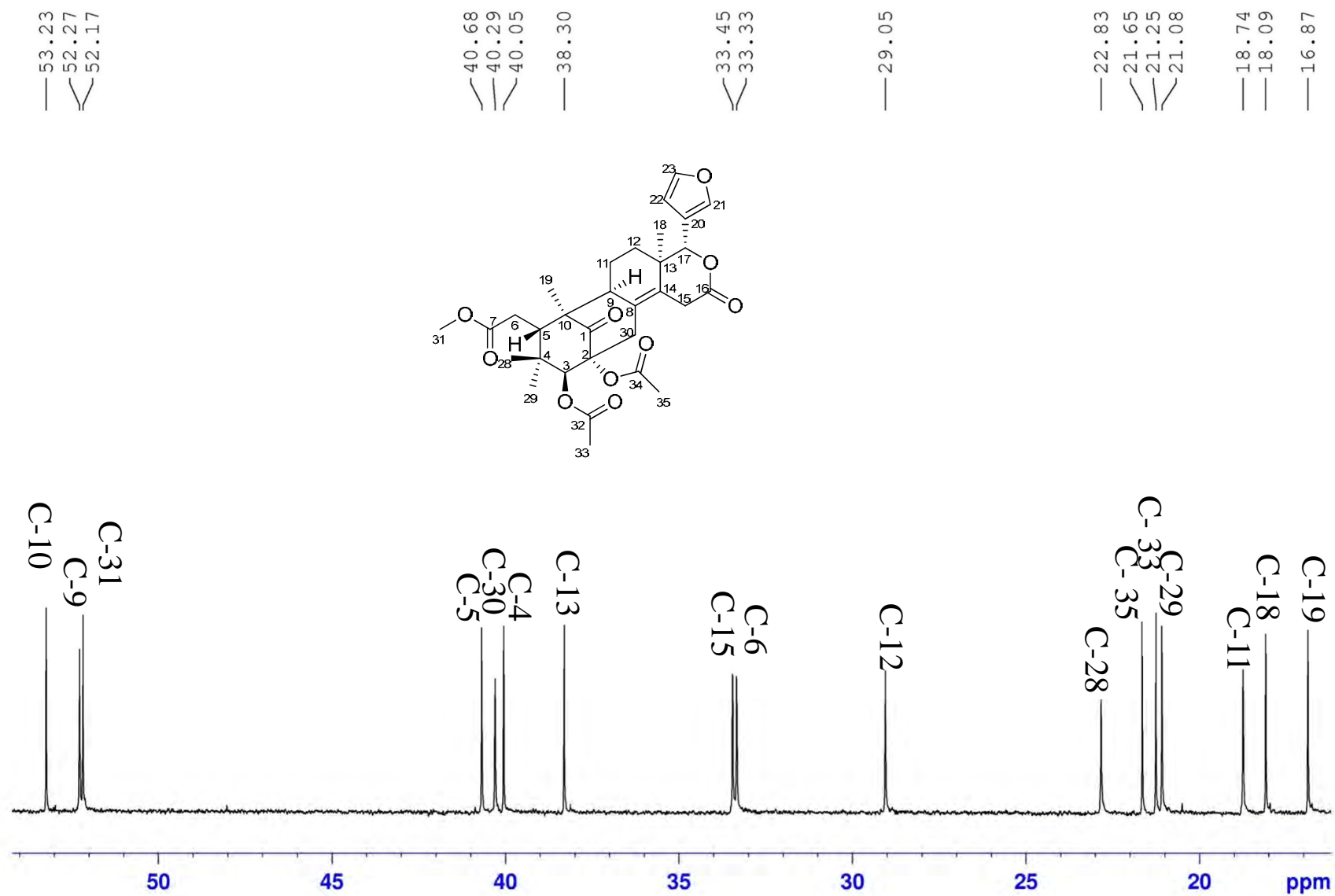
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



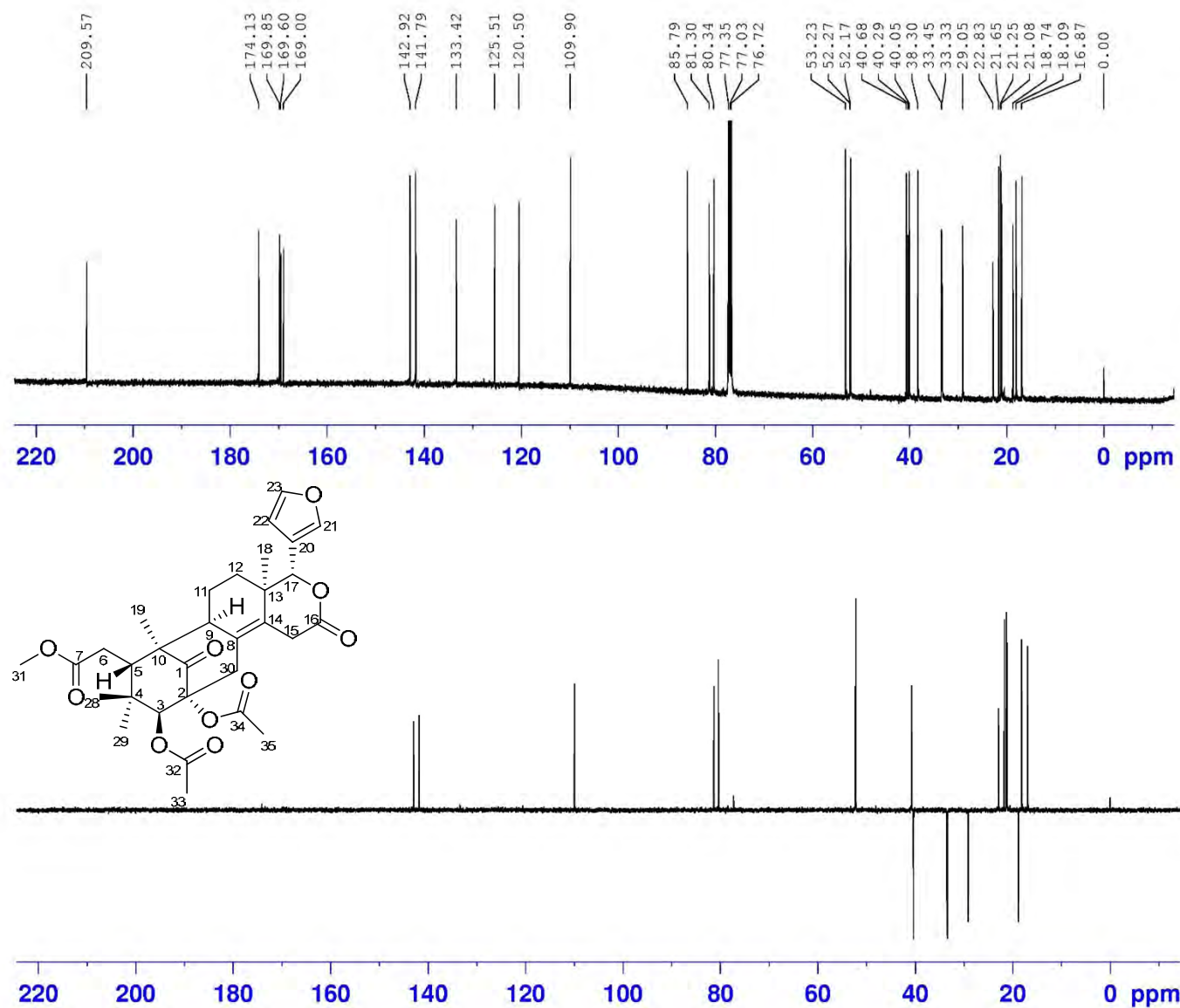
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **7** in CDCl<sub>3</sub>

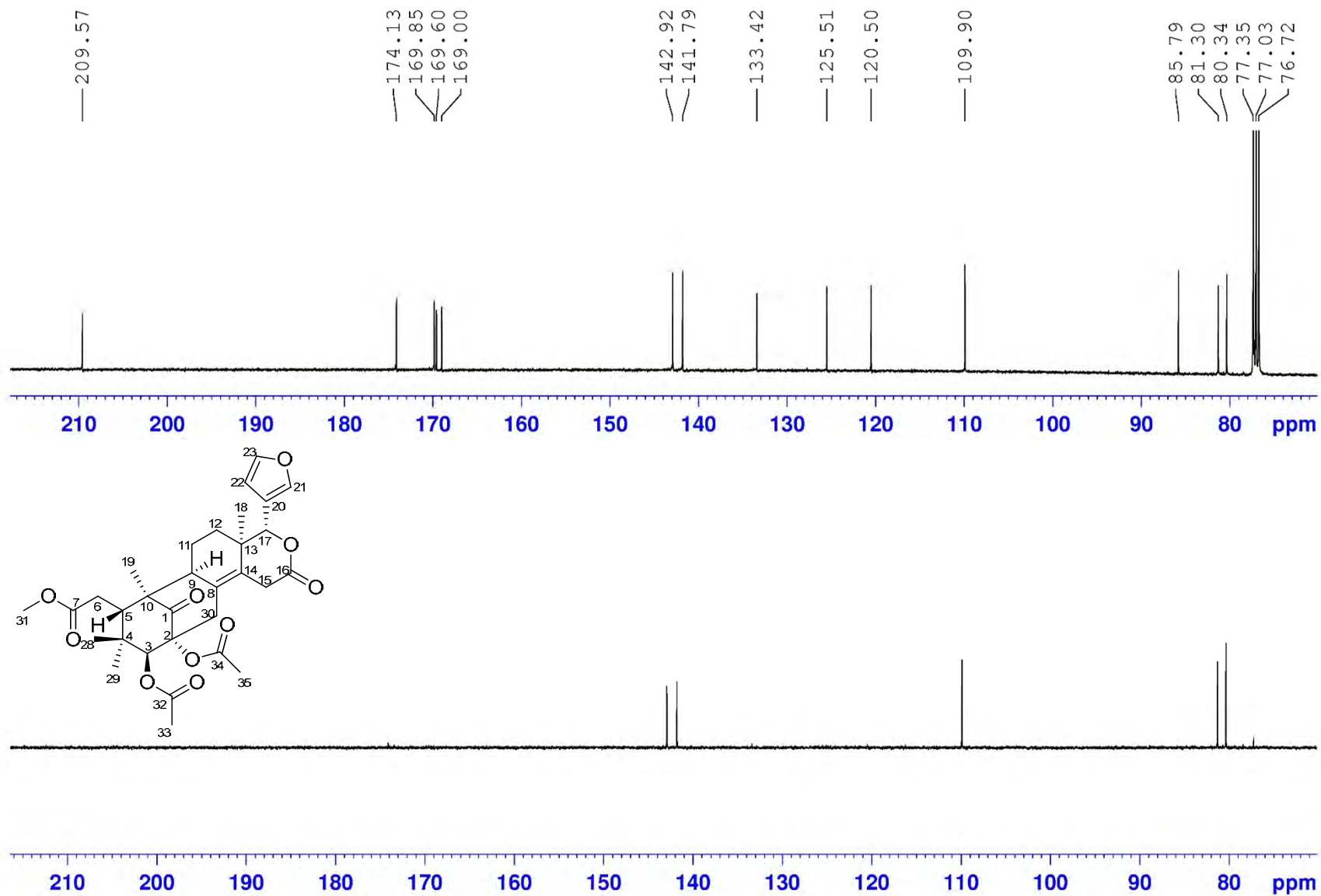


NAME zjz-34  
 EXPNO 3  
 PROCNO 1  
 Date\_ 20160831  
 Time 8.14  
 INSTRUM spect  
 PROBHD 5 mm CPBBO BB  
 PULPROG deptsp135  
 TD 65536  
 SOLVENT CDCl3  
 NS 300  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 91.64  
 DW 20.800 usec  
 DE 18.00 usec  
 TE 297.0 K  
 CNST2 145.0000000  
 D1 2.00000000 sec  
 D2 0.00344828 sec  
 D12 0.00002000 sec  
 TD0 1

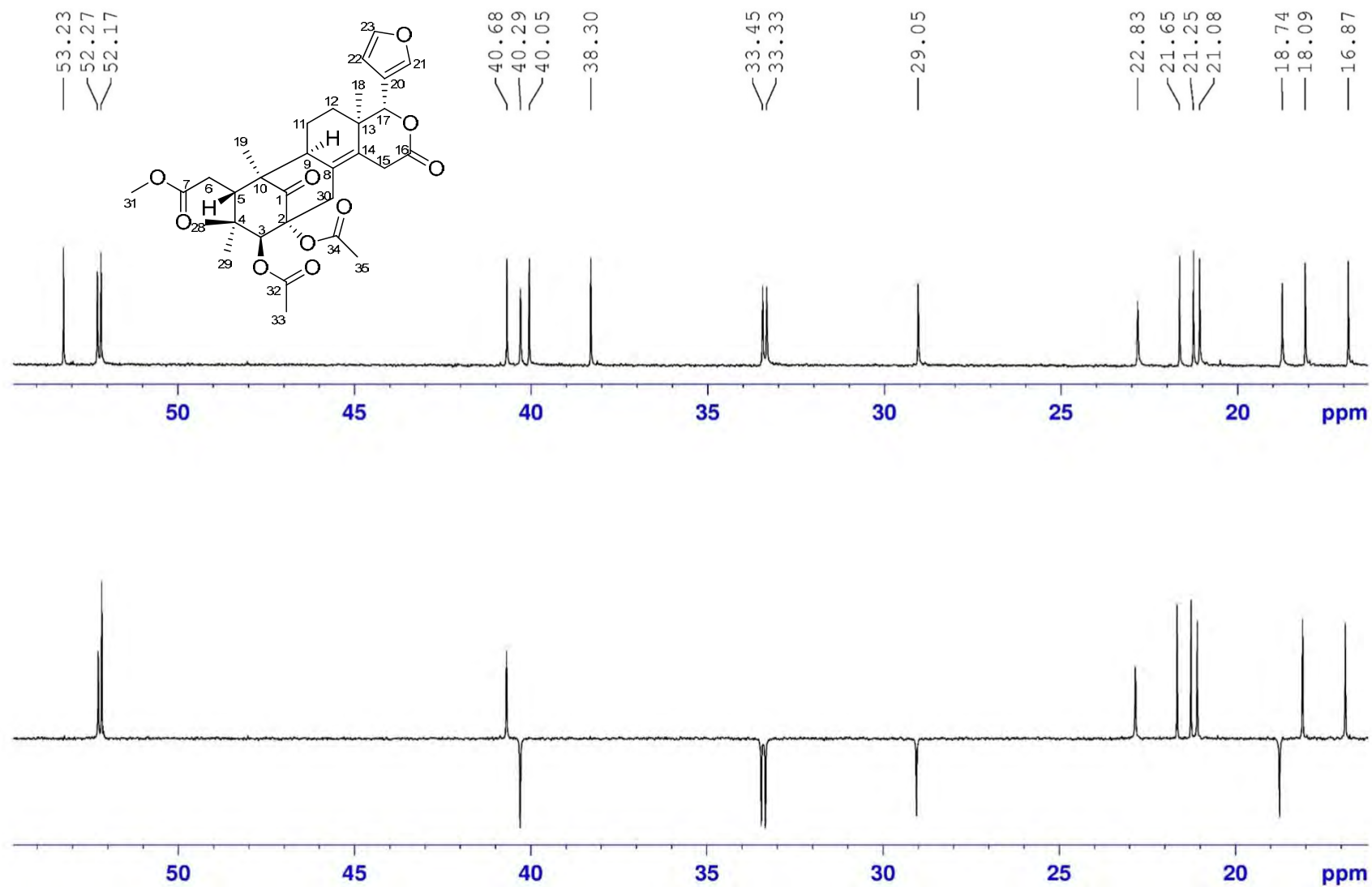
===== CHANNEL f1 =====  
 SF01 100.6233324 MHz  
 NUC1 13C  
 P1 10.00 usec  
 P13 2000.00 usec  
 SI 32768  
 SF 100.6127689 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



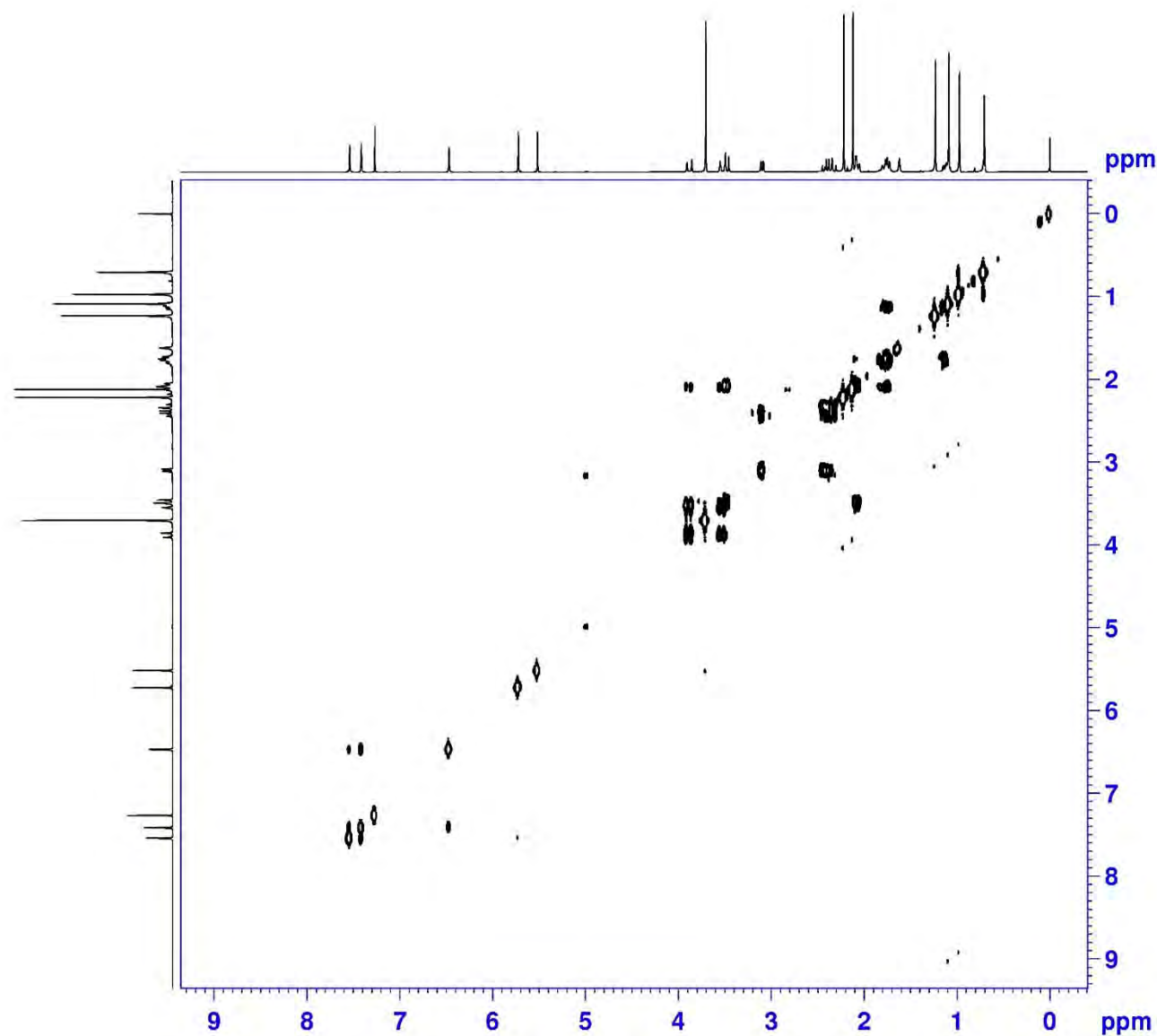
# DEPT135 (100 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **7** in $\text{CDCl}_3$



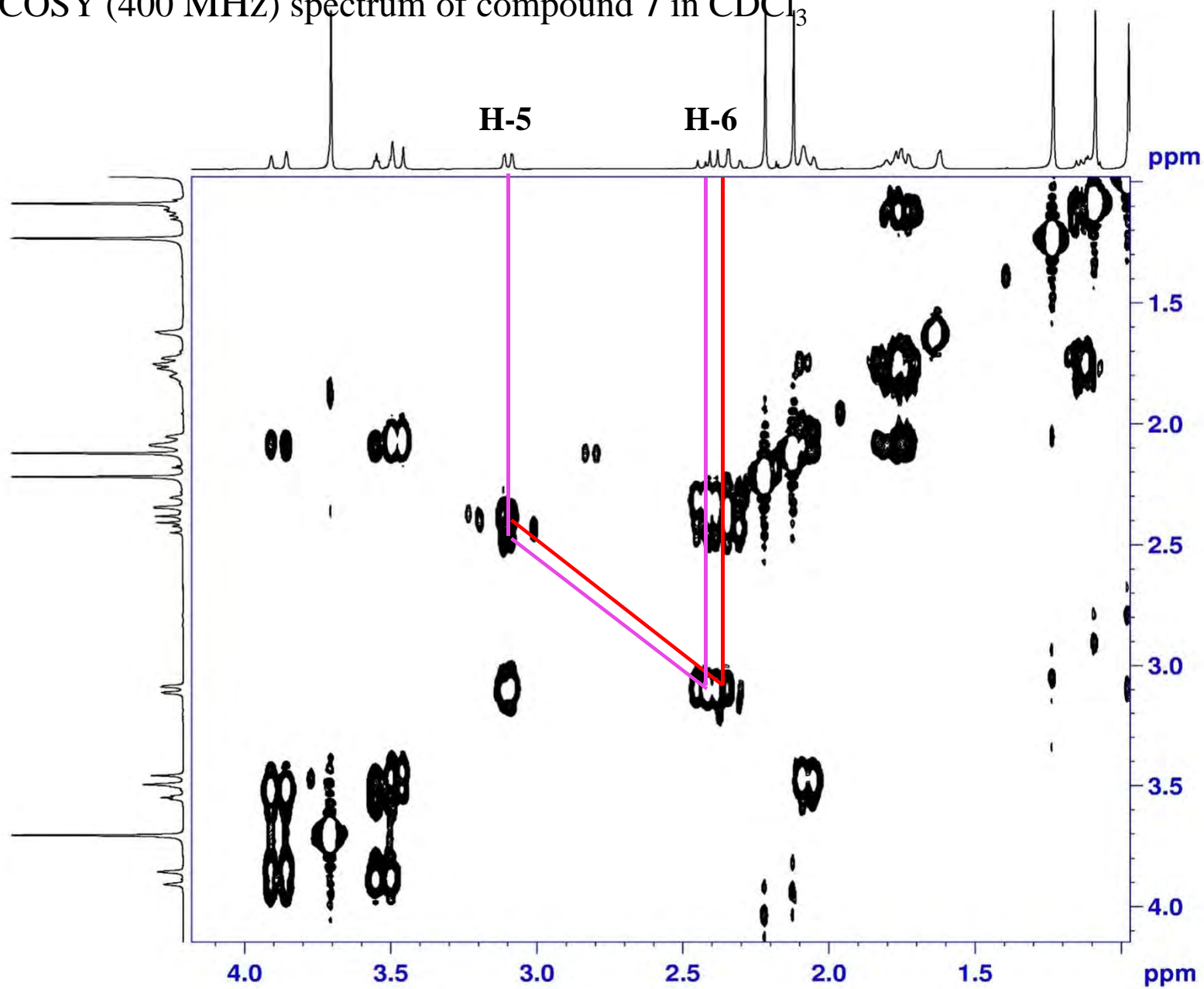
```

NAME           zjz-34
EXPNO          4
PROCNO         1
Date_          20160913
Time           22.31
INSTRUM        spect
PROBHD         5 mm CFPBBO BB
PULPROG        cosygpppqf
TD             2048
SOLVENT        CDCl3
NS             8
DS             8
SWH            3906.250 Hz
FIDRES         1.907349 Hz
AQ             0.2621940 sec
RG             171.57
DW             128.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00000300 sec
D1             1.89678097 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00025600 sec
    
```

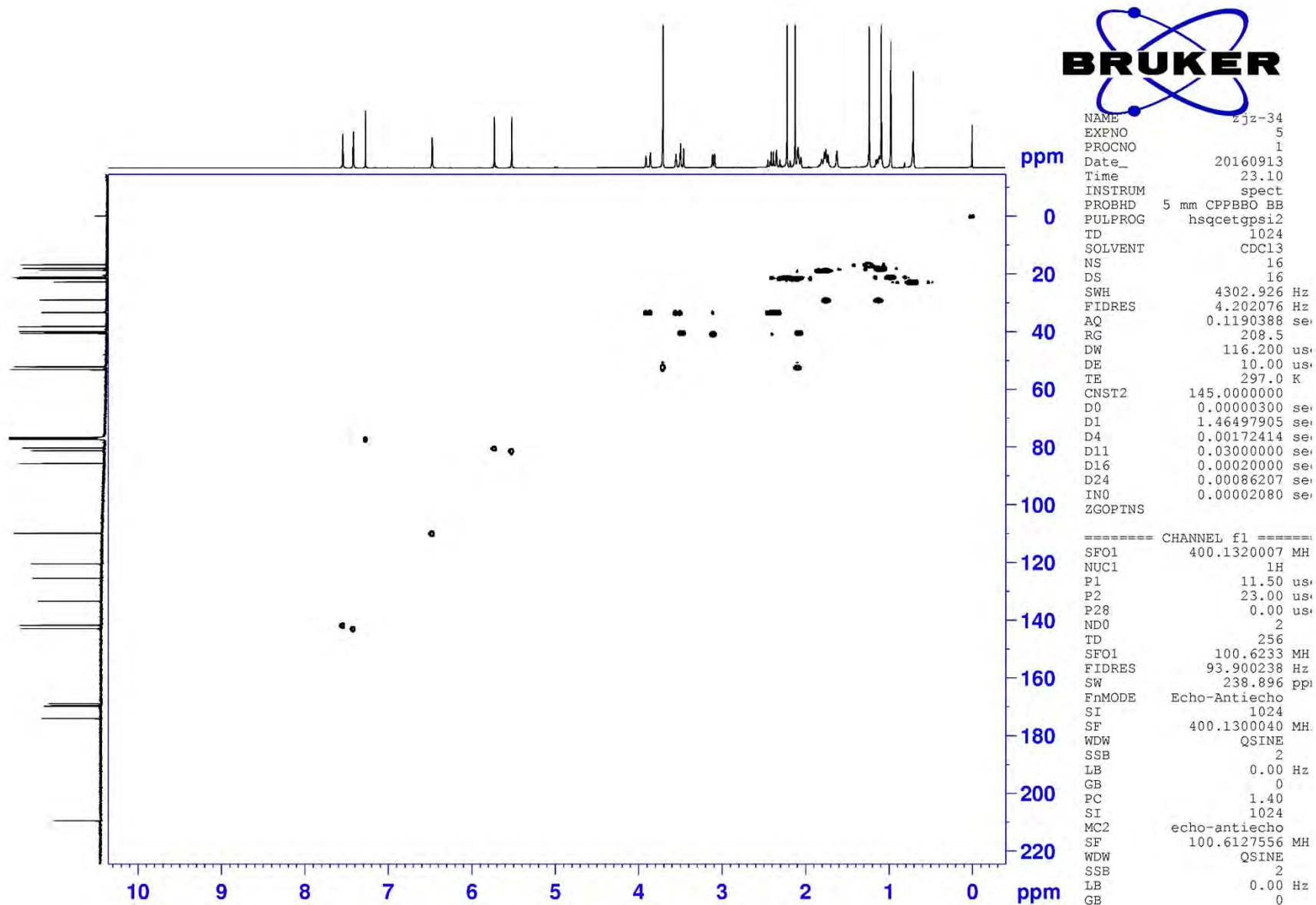
```

===== CHANNEL f1 =====
SFO1         400.1318006 MHz
NUC1          1H
P0            11.50 usec
P1            11.50 usec
P17           2500.00 usec
ND0           1
TD            128
SFO1         400.1318 MHz
FIDRES        30.517578 Hz
SW             9.762 ppm
FnMODE        QF
SI            1024
SF            400.1300040 MHz
WDW           QSINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40
SI            1024
MC2           QF
SF            400.1300051 MHz
WDW           QSINE
SSB            0
LB             0.00 Hz
GB             0
    
```

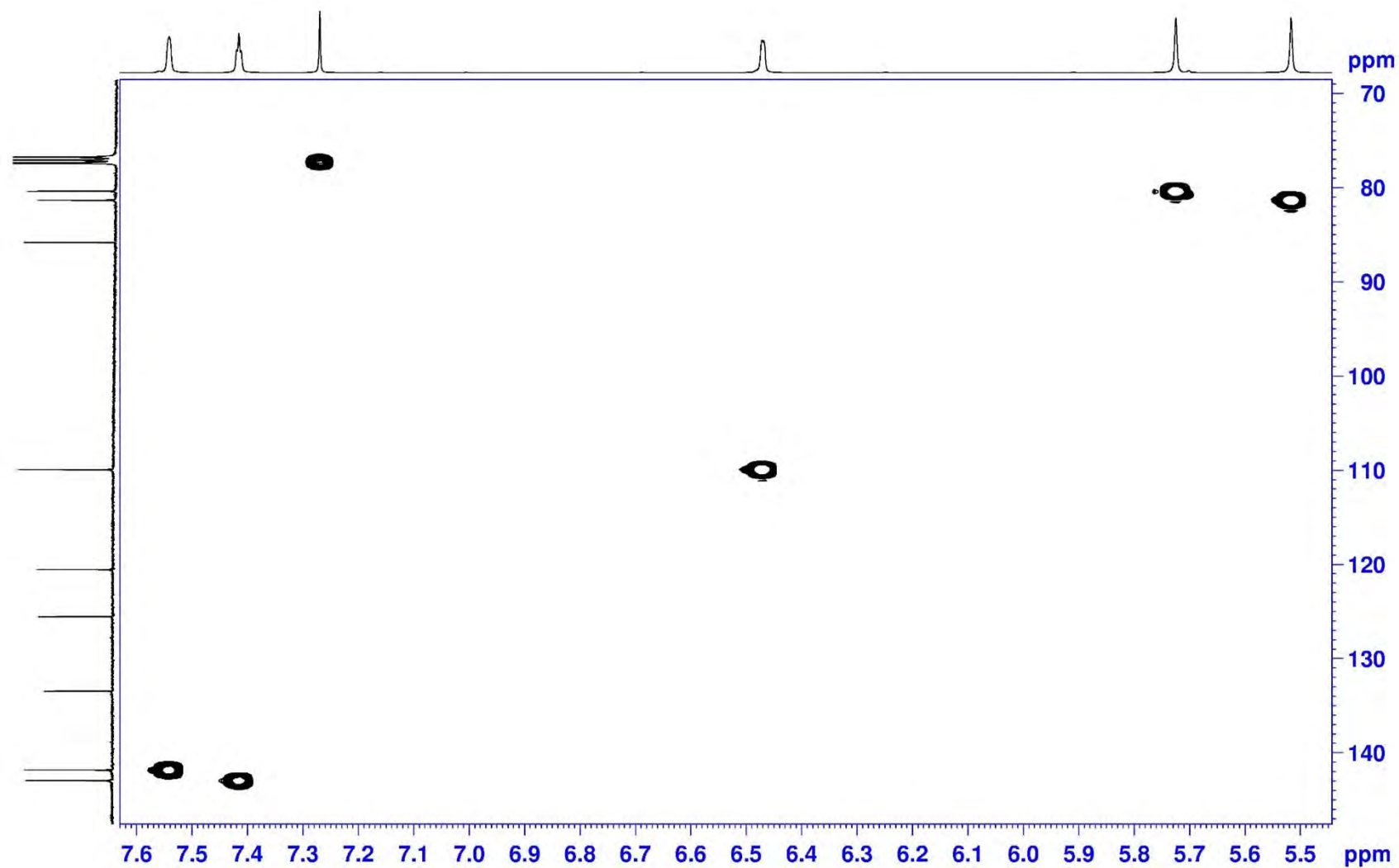
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



# HSQC (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>

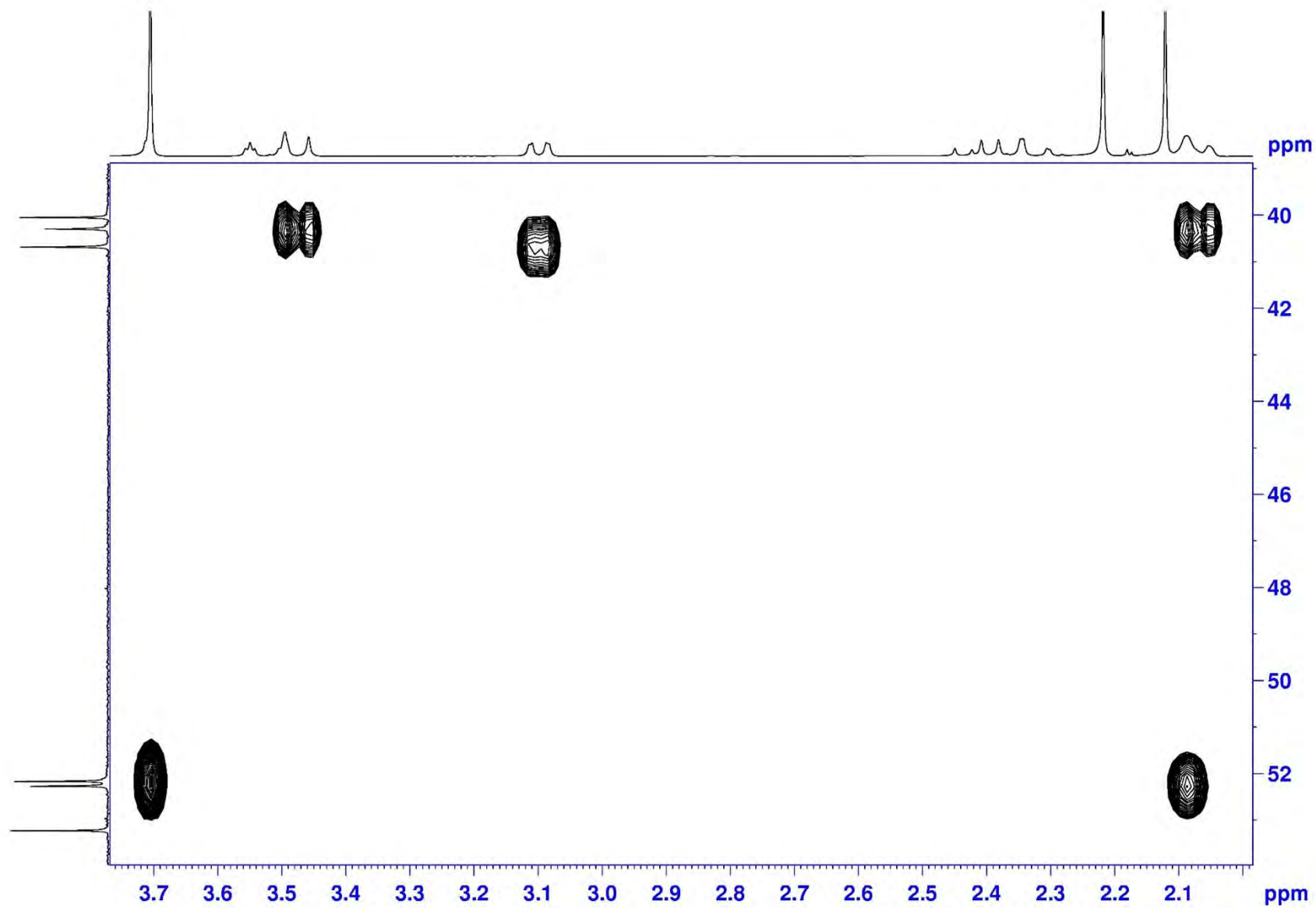


HSQC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



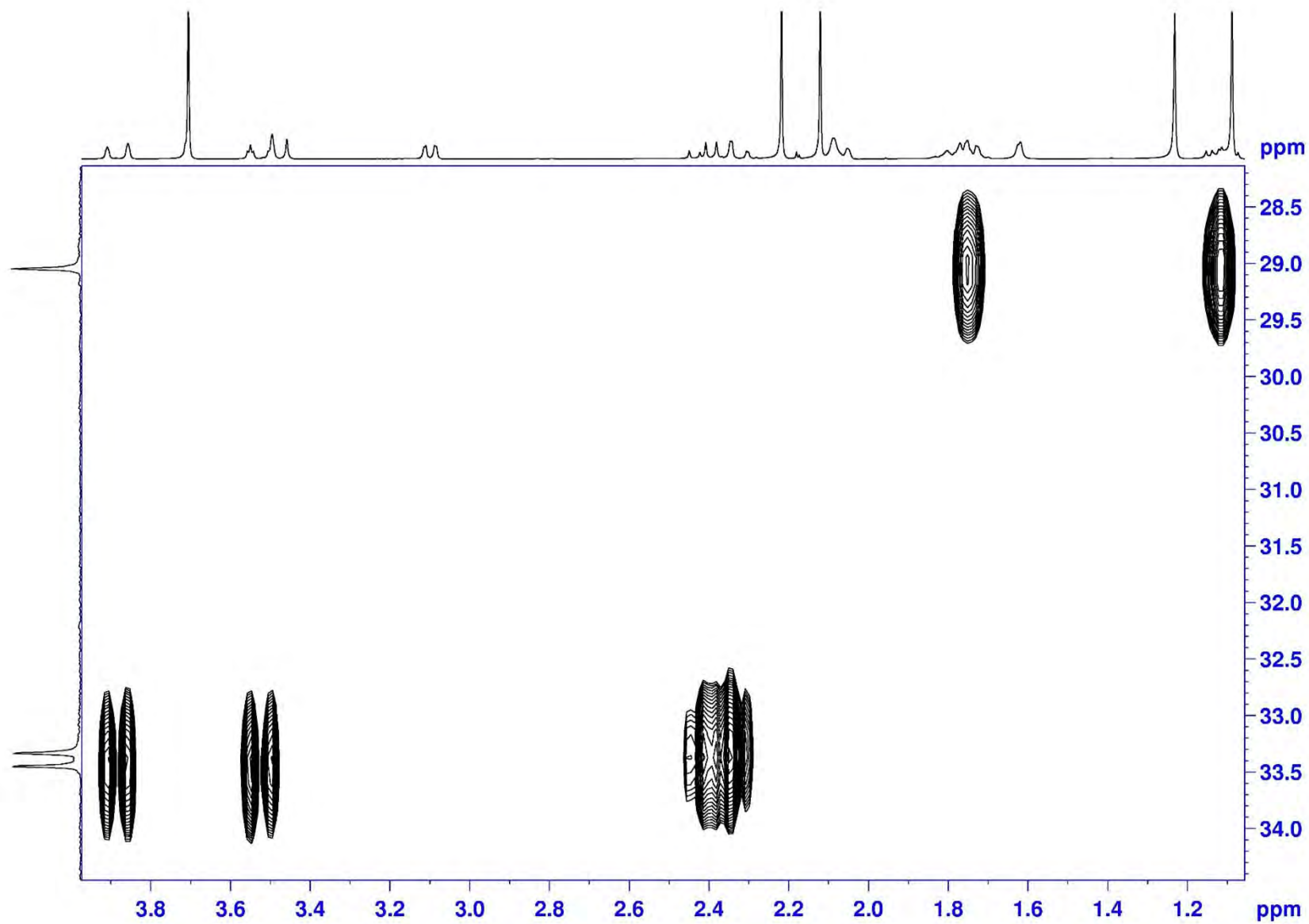


HSQC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$

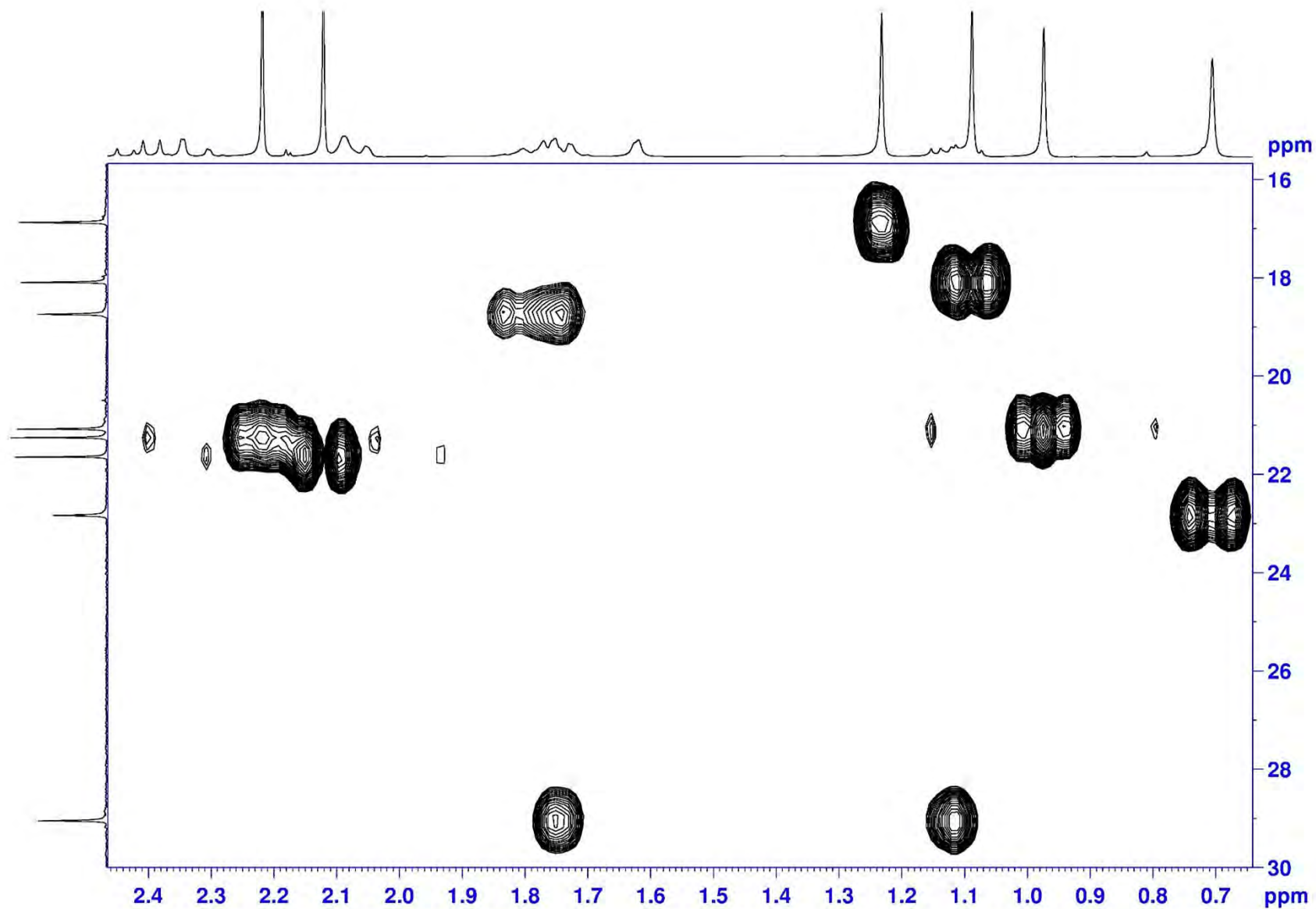




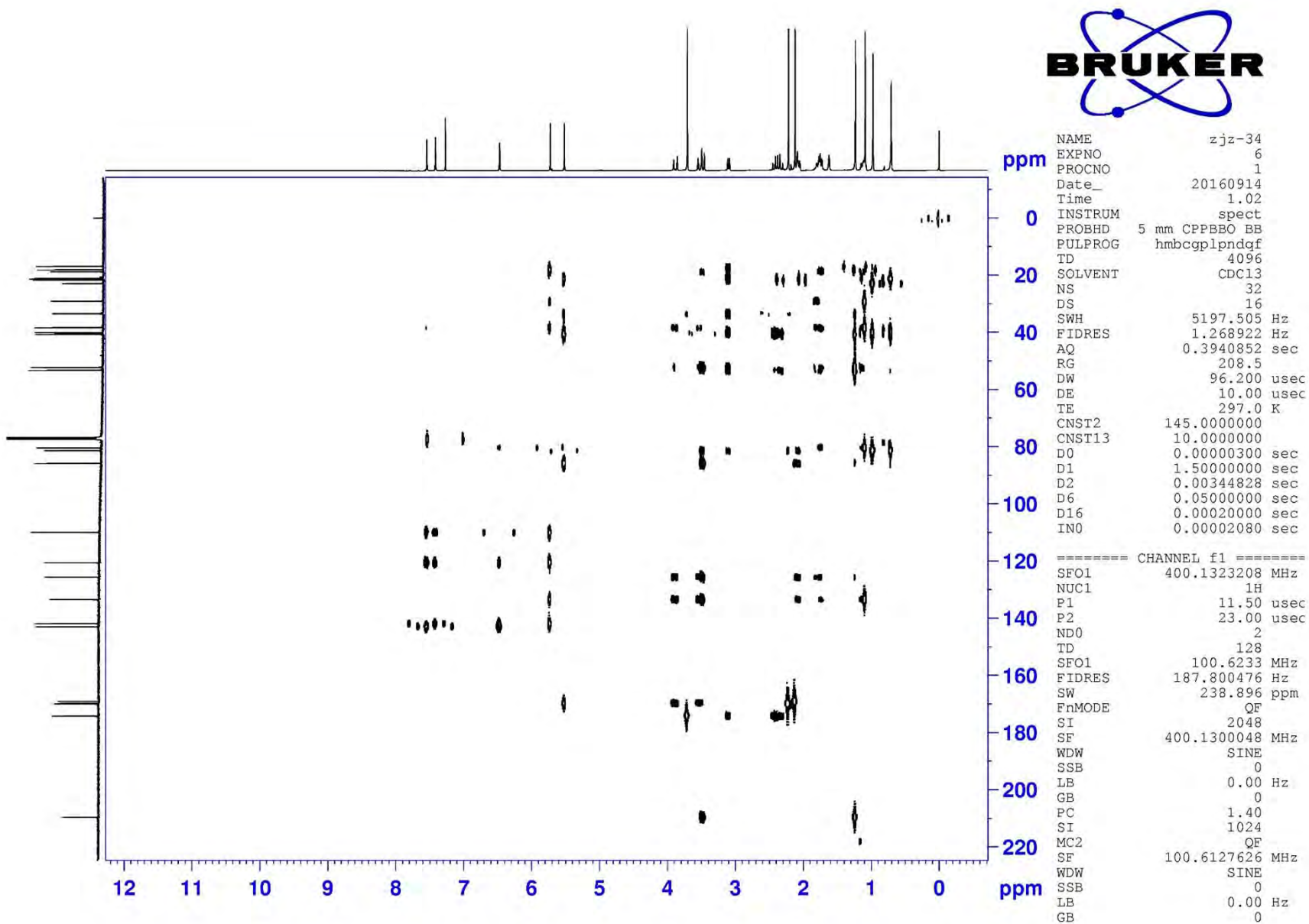
HSQC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



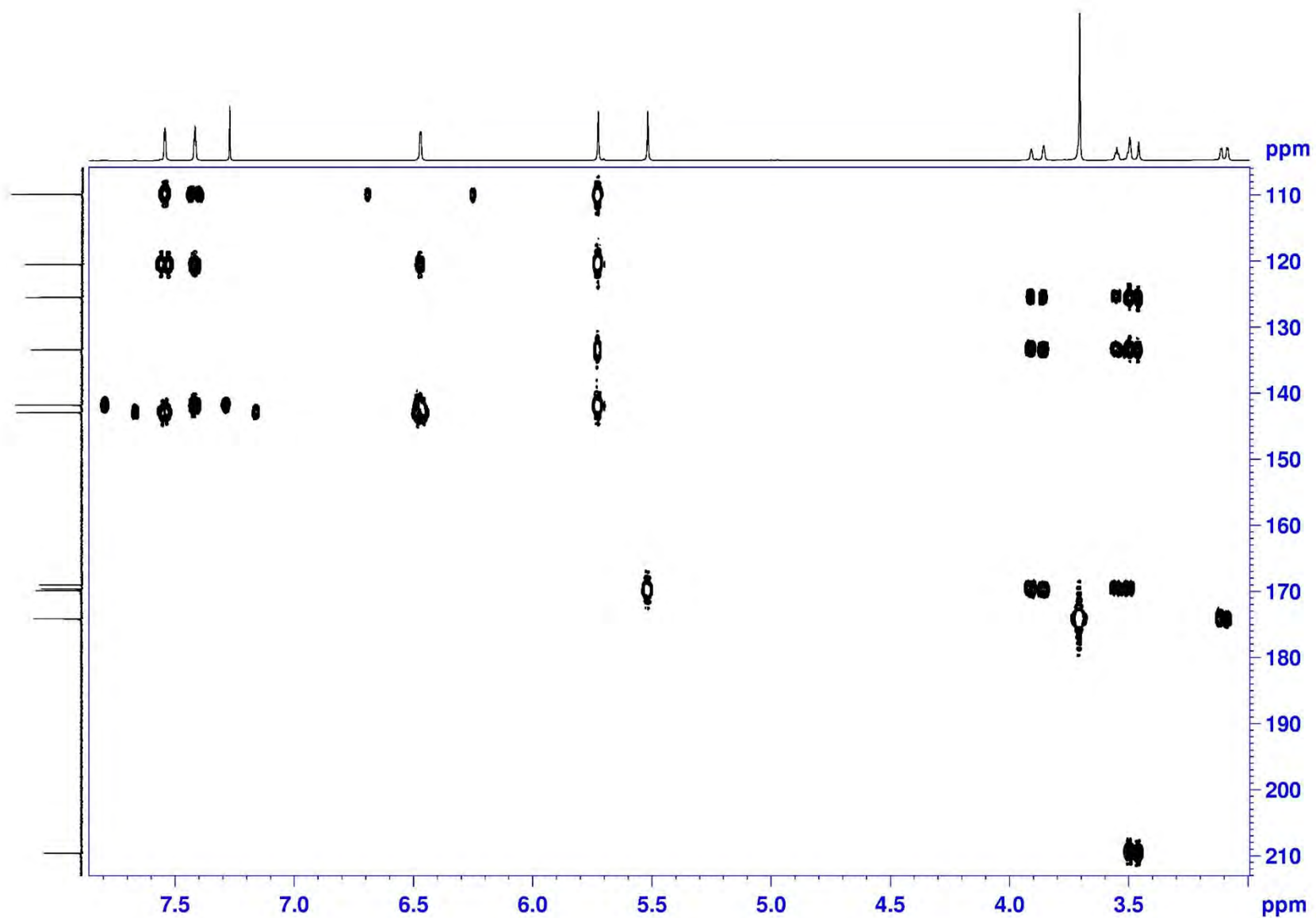
HSQC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



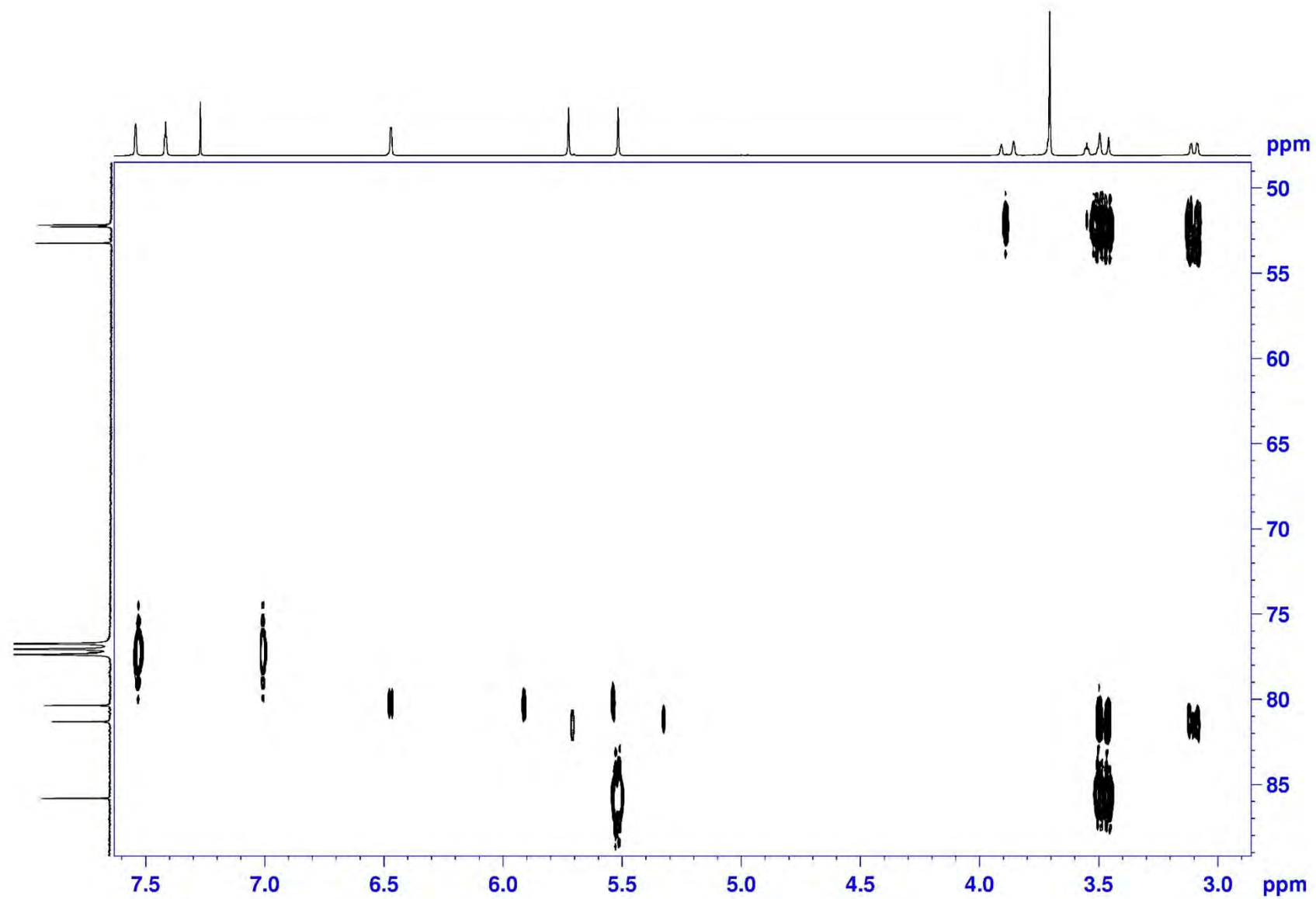
# HMBC (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



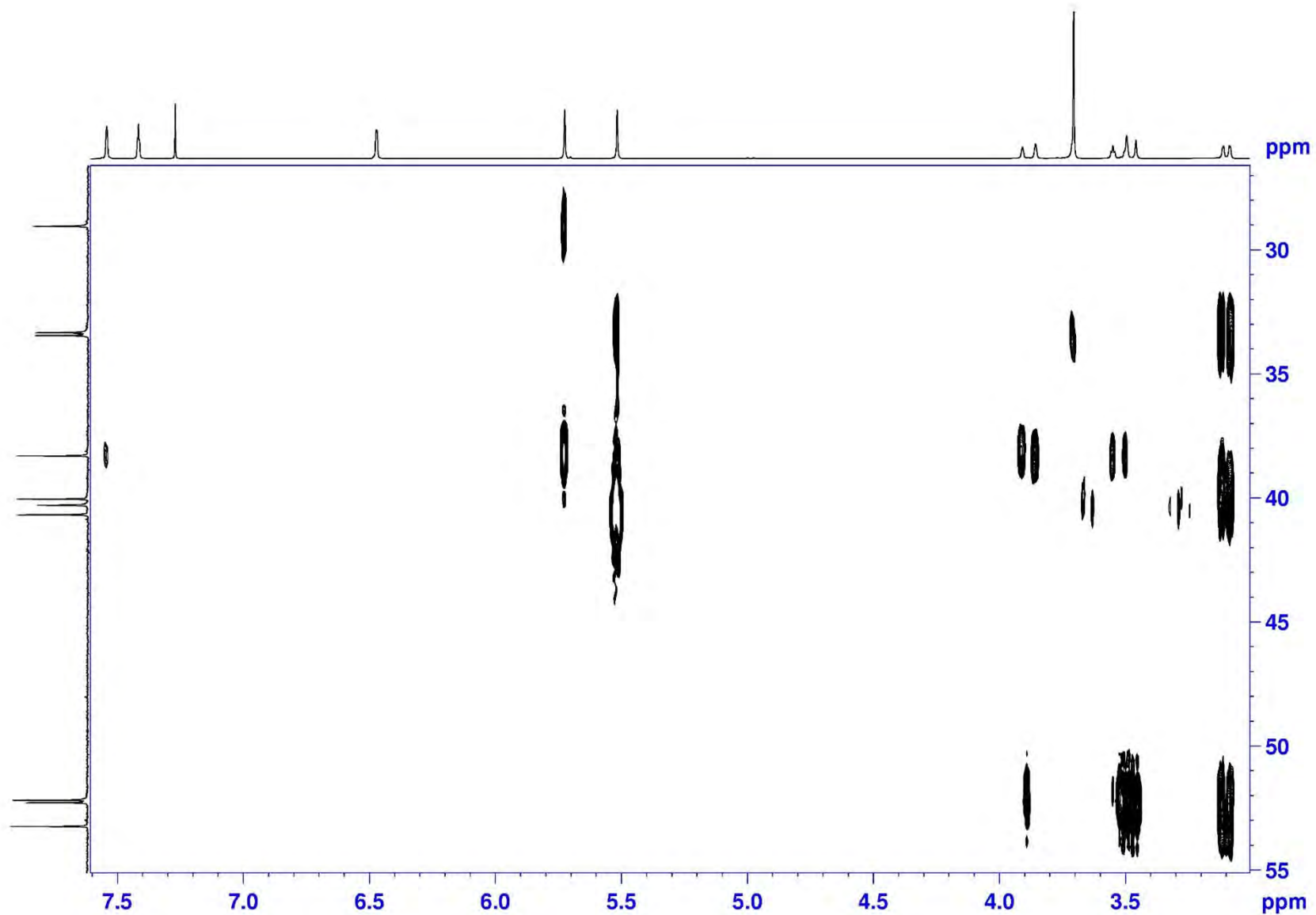
HMBC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>

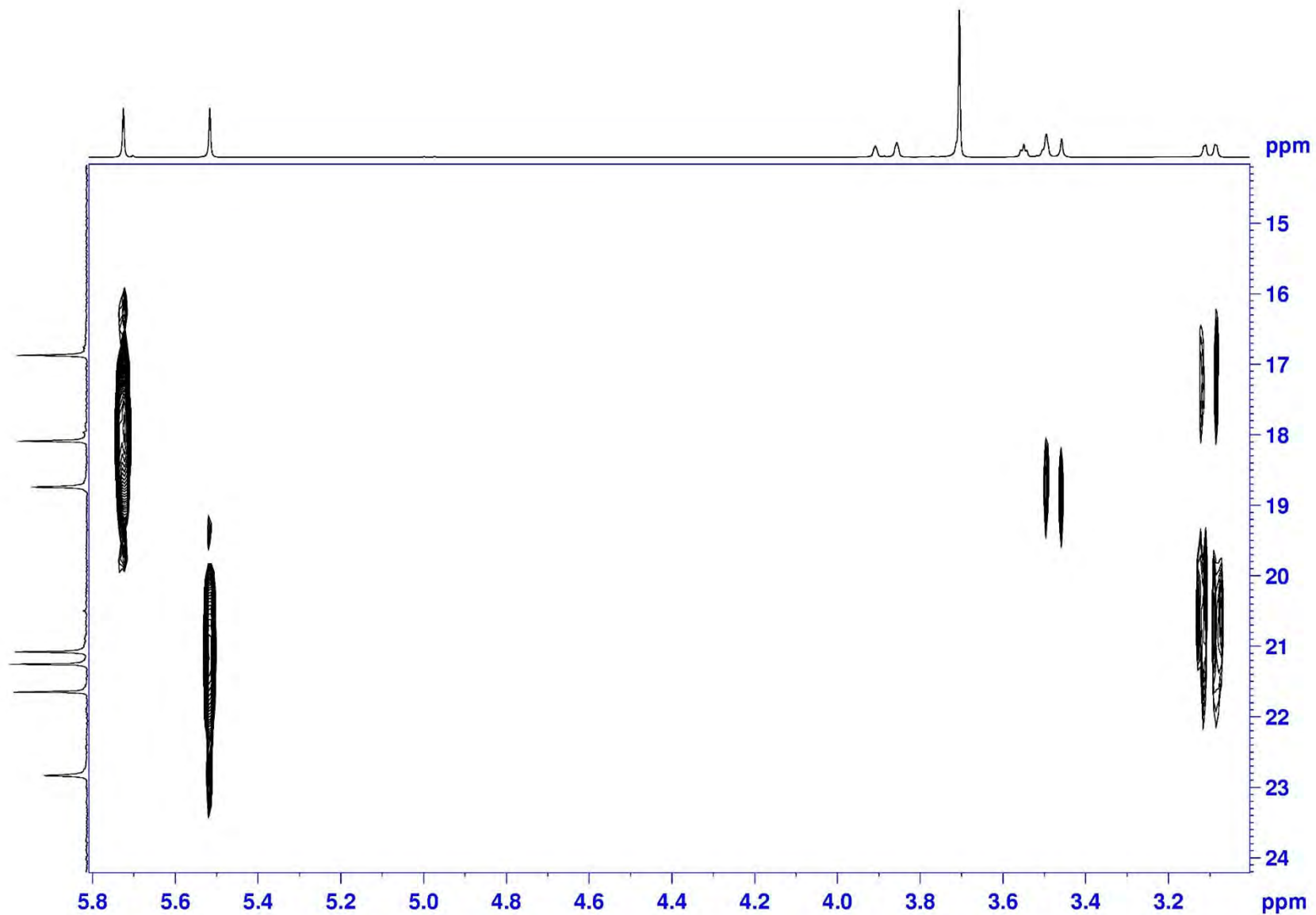


HMBC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$

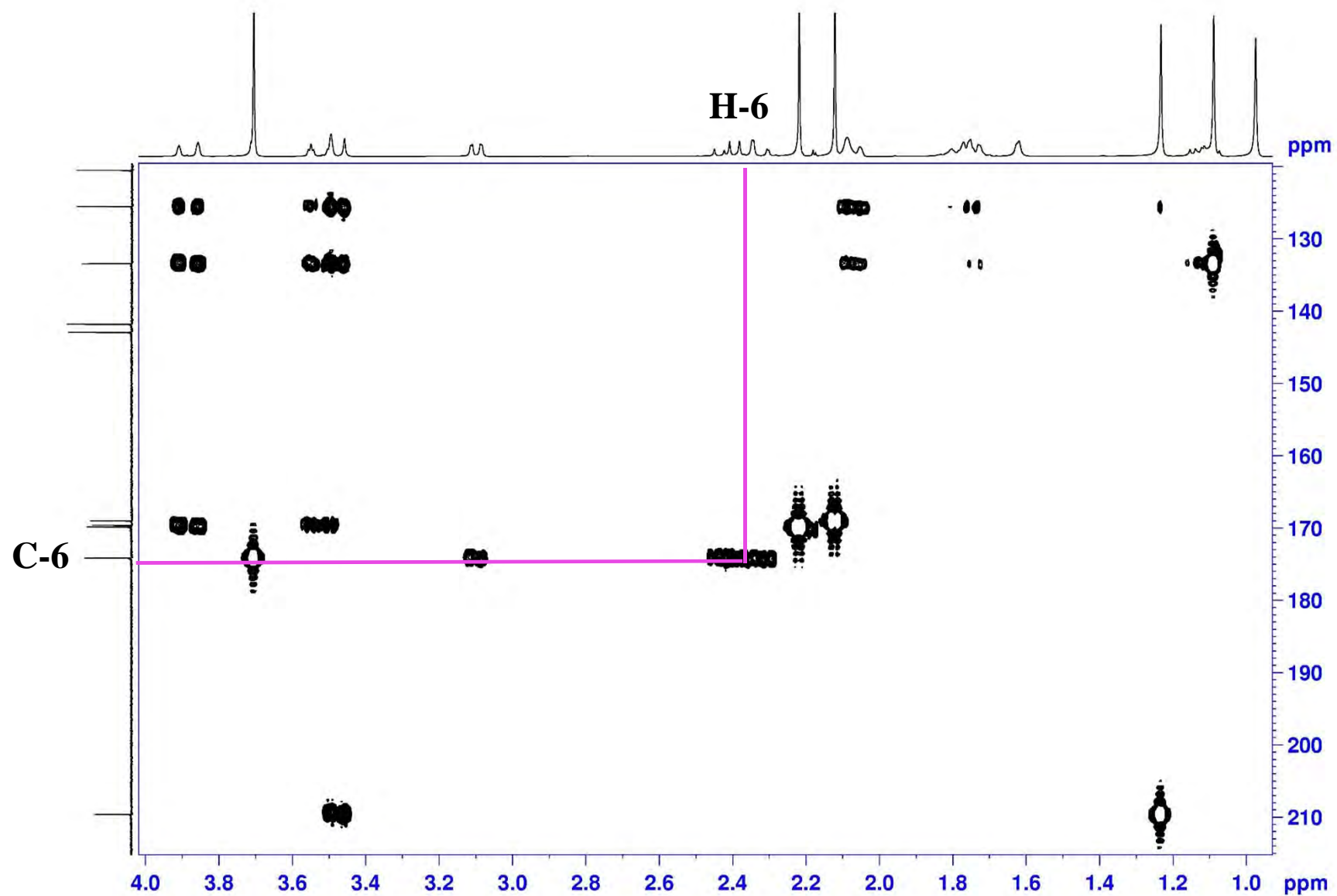




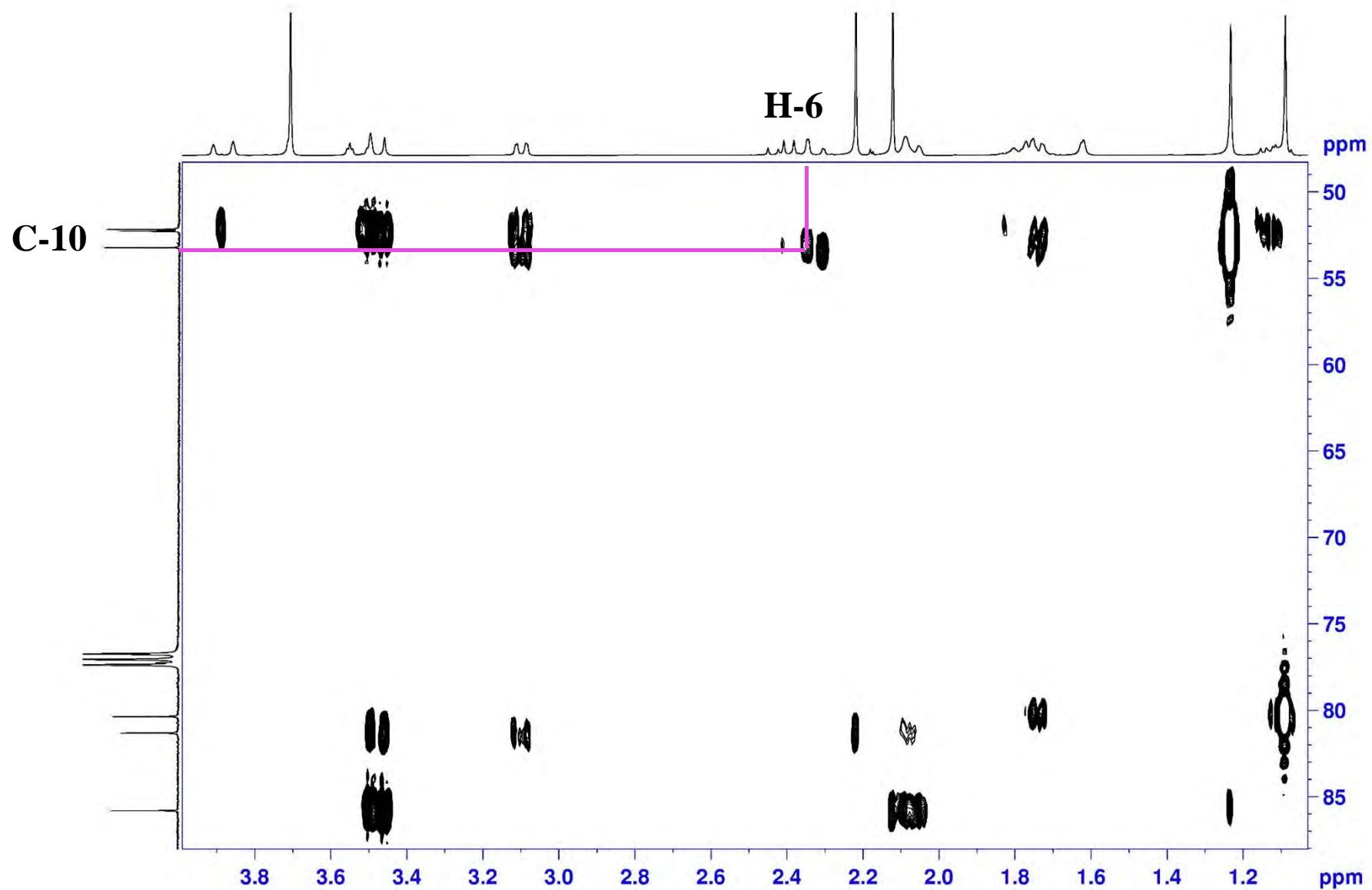
HMBC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



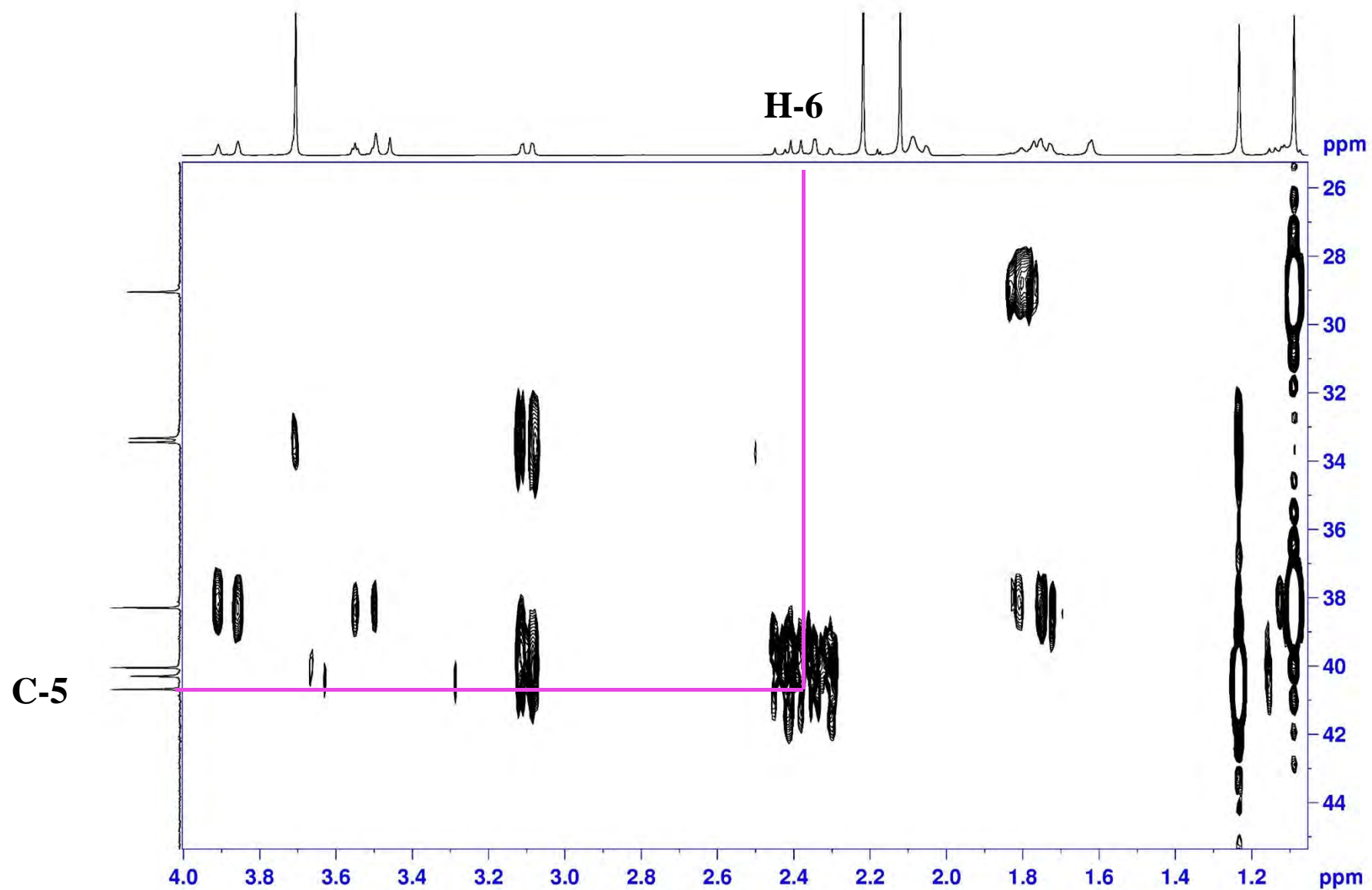
HMBC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



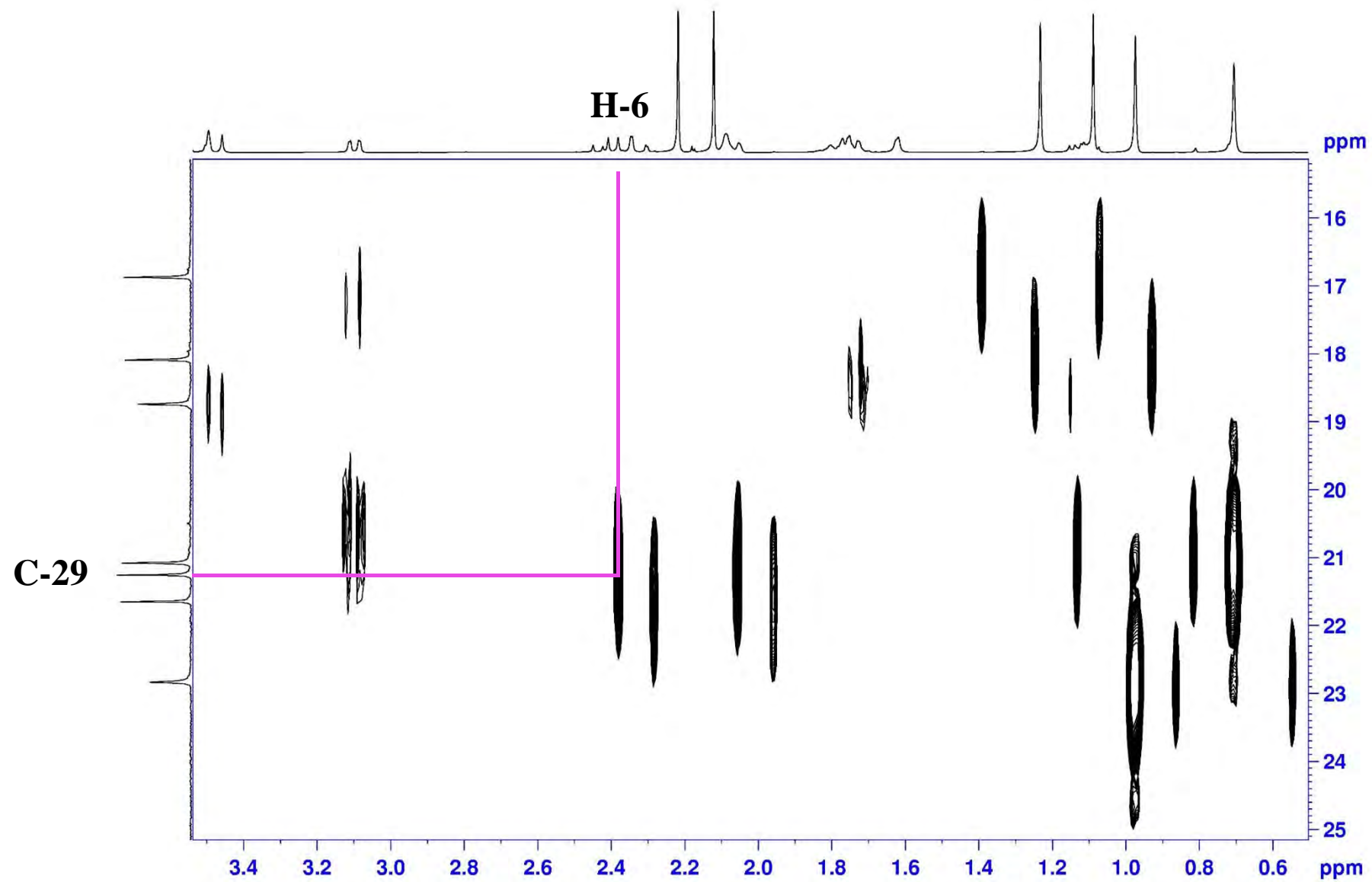
HMBC (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



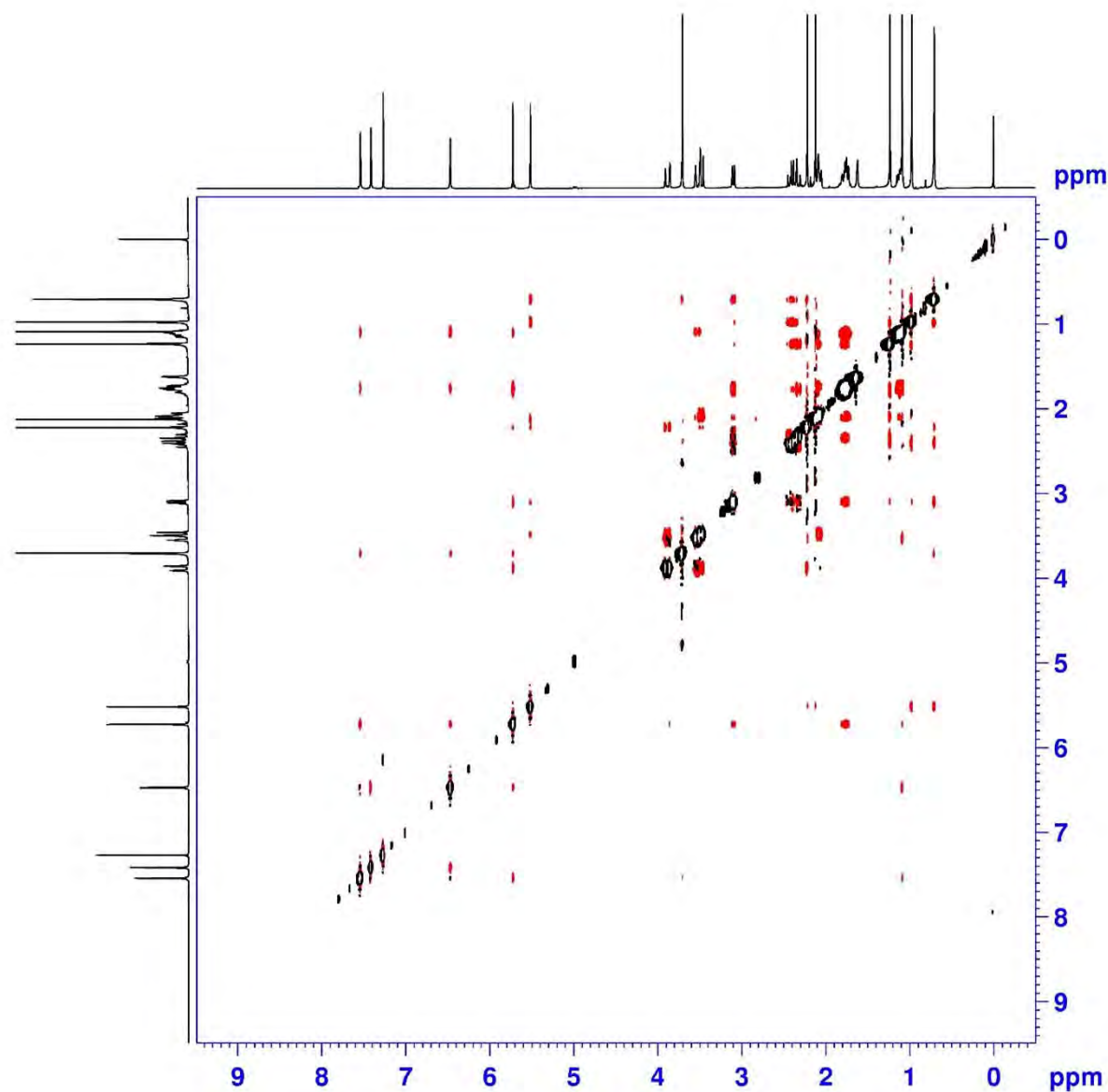
HMBC (400 MHz) spectrum of compound **7** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



# NOESY (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



```

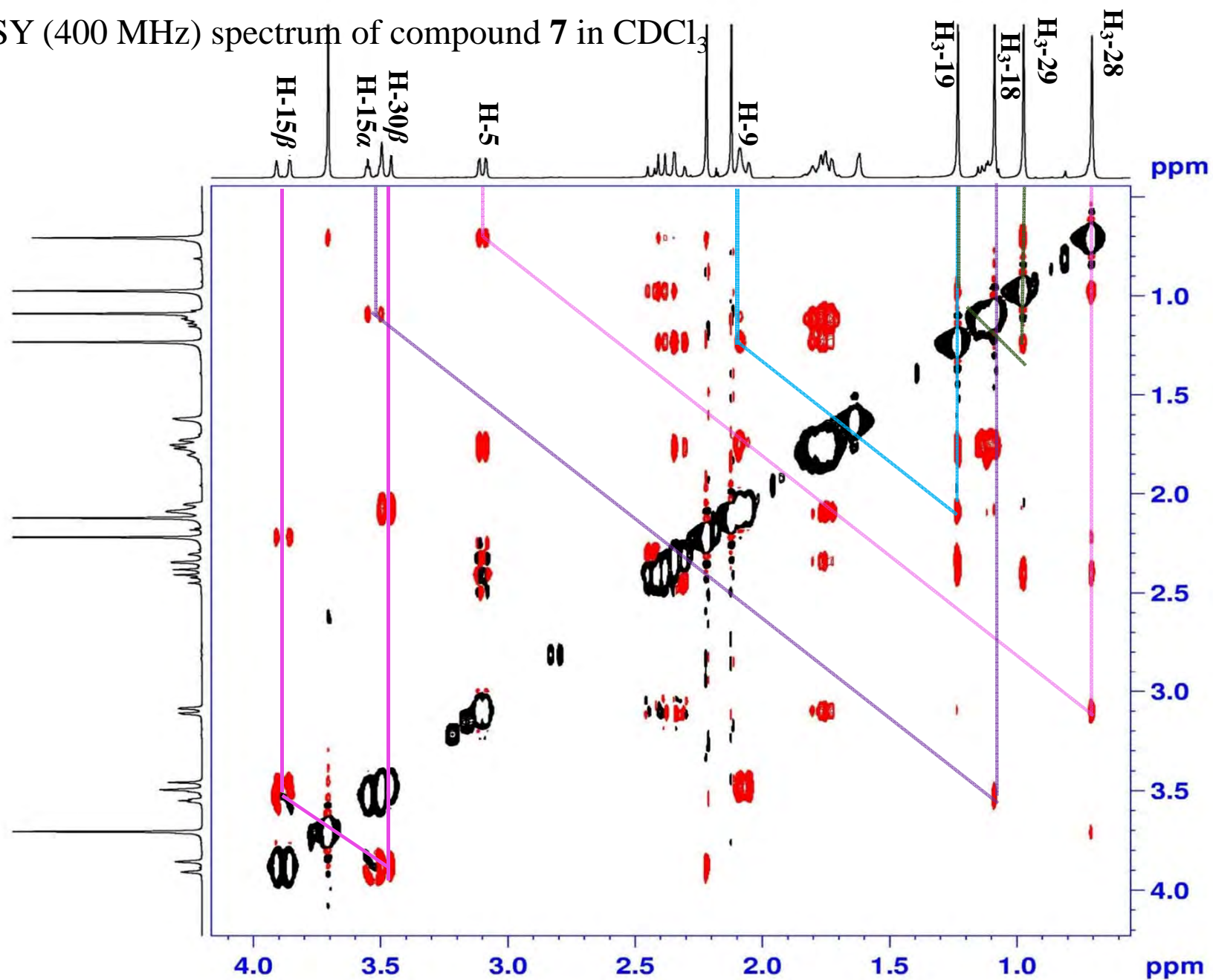
NAME          zjz-34
EXPNO          7
PROCNO         1
Date_          20160914
Time           3.17
INSTRUM        spect
PROBHD         5 mm CFPBBO BB
PULPROG        noesygpph
TD             2048
SOLVENT        CDCl3
NS             16
DS             32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ             0.2560500 sec
RG             102.3
DW            125.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00011036 sec
D1             1.99385595 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00025000 sec
  
```

```

===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1           1H
P1             11.50 usec
P2             23.00 usec
P17            2500.00 usec
ND0            1
TD             256
SFO1          400.1318 MHz
FIDRES         15.625000 Hz
SW             9.997 ppm
FnMODE         States-TPPI
SI             1024
SF            400.1300044 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF            400.1300044 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
  
```

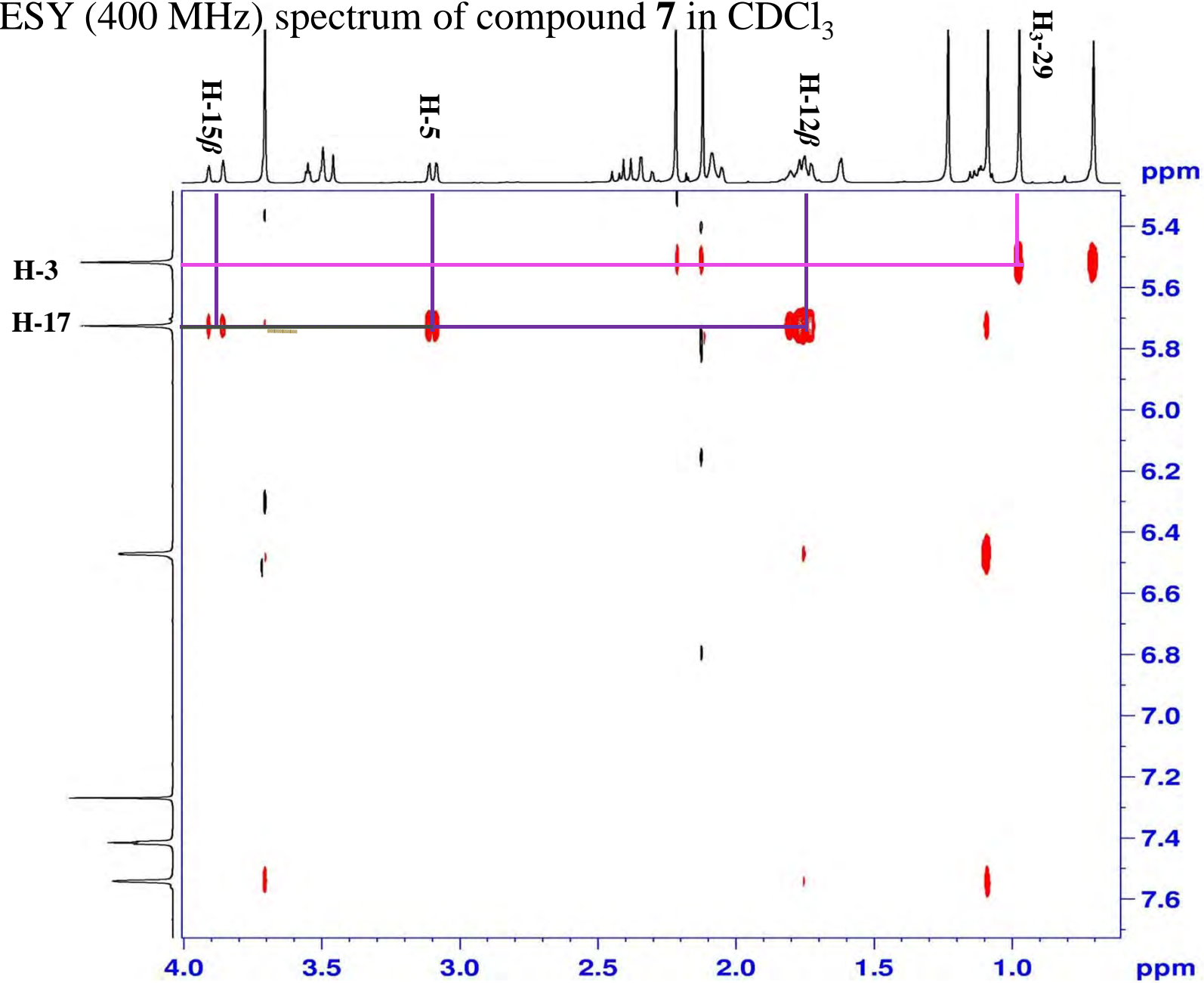


NOESY (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>

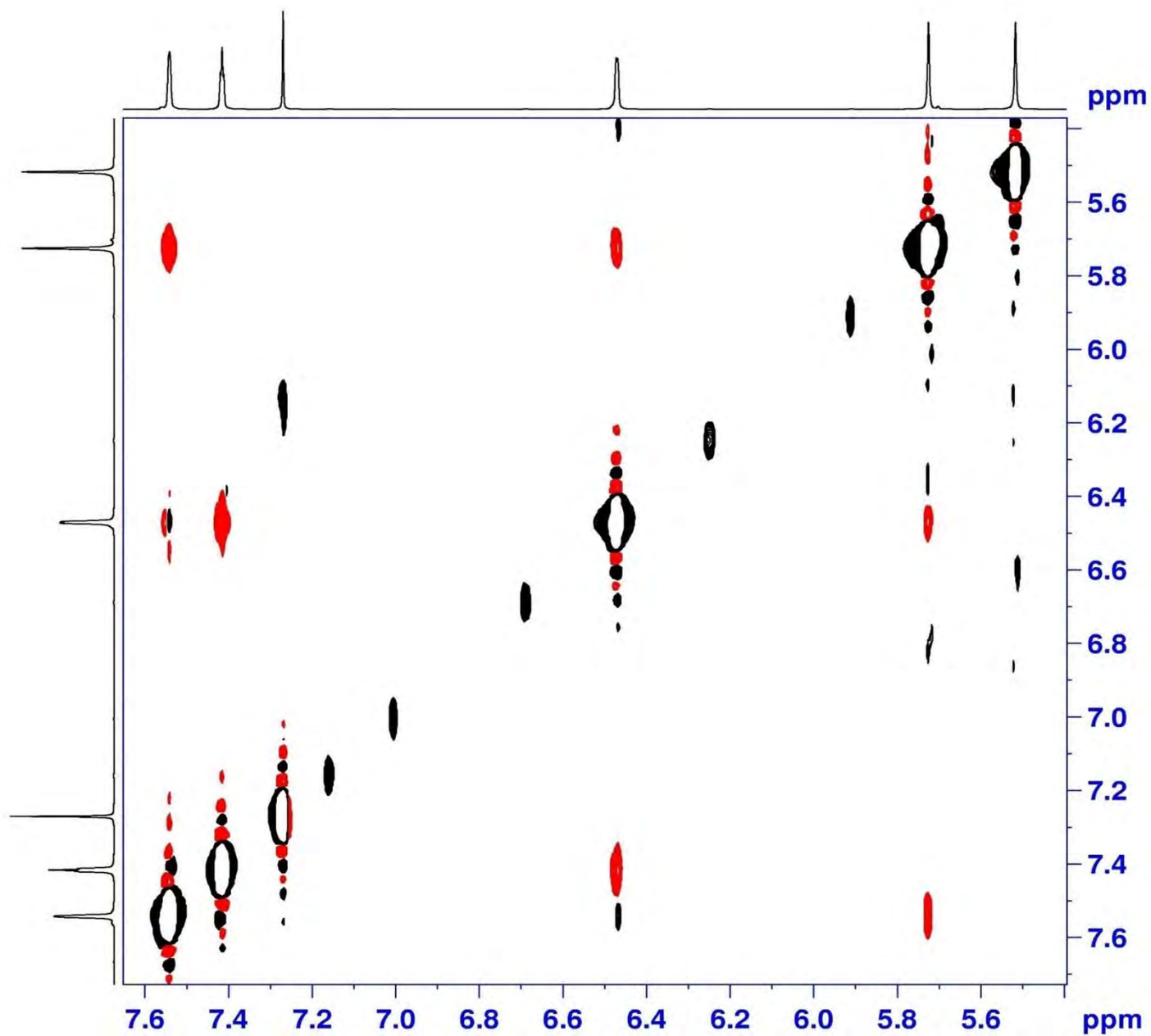




NOESY (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **7** in CDCl<sub>3</sub>



# HR-ESIMS for compound 8

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-58\_pos\_12\_01\_1859.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-58\_pos  
 Comment

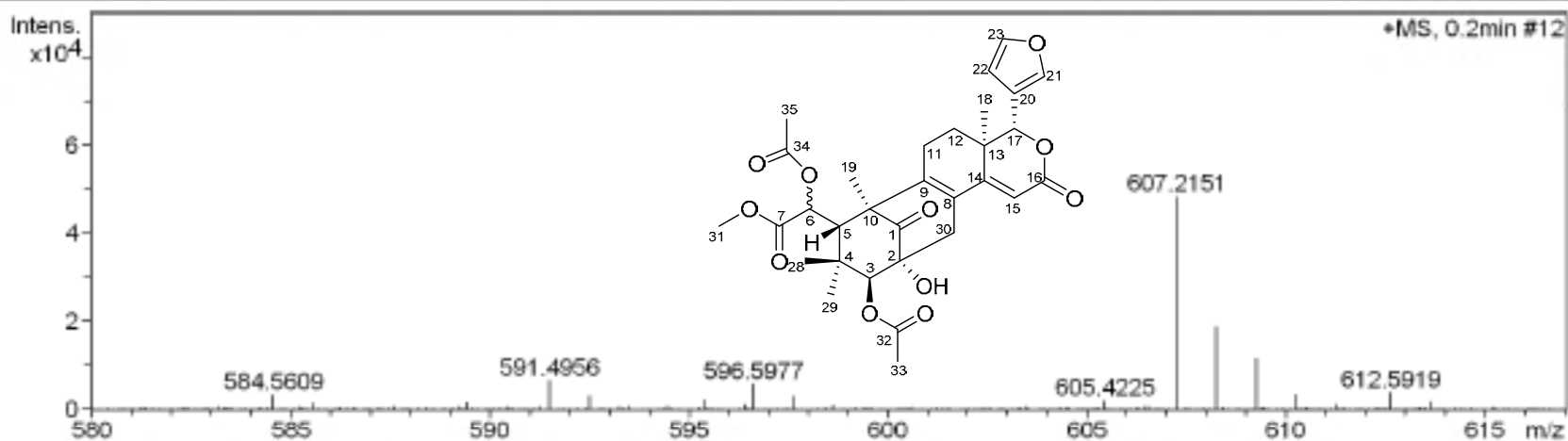
Acquisition Date 5/19/2016 8:54:55 AM

Operator SCSIO

Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



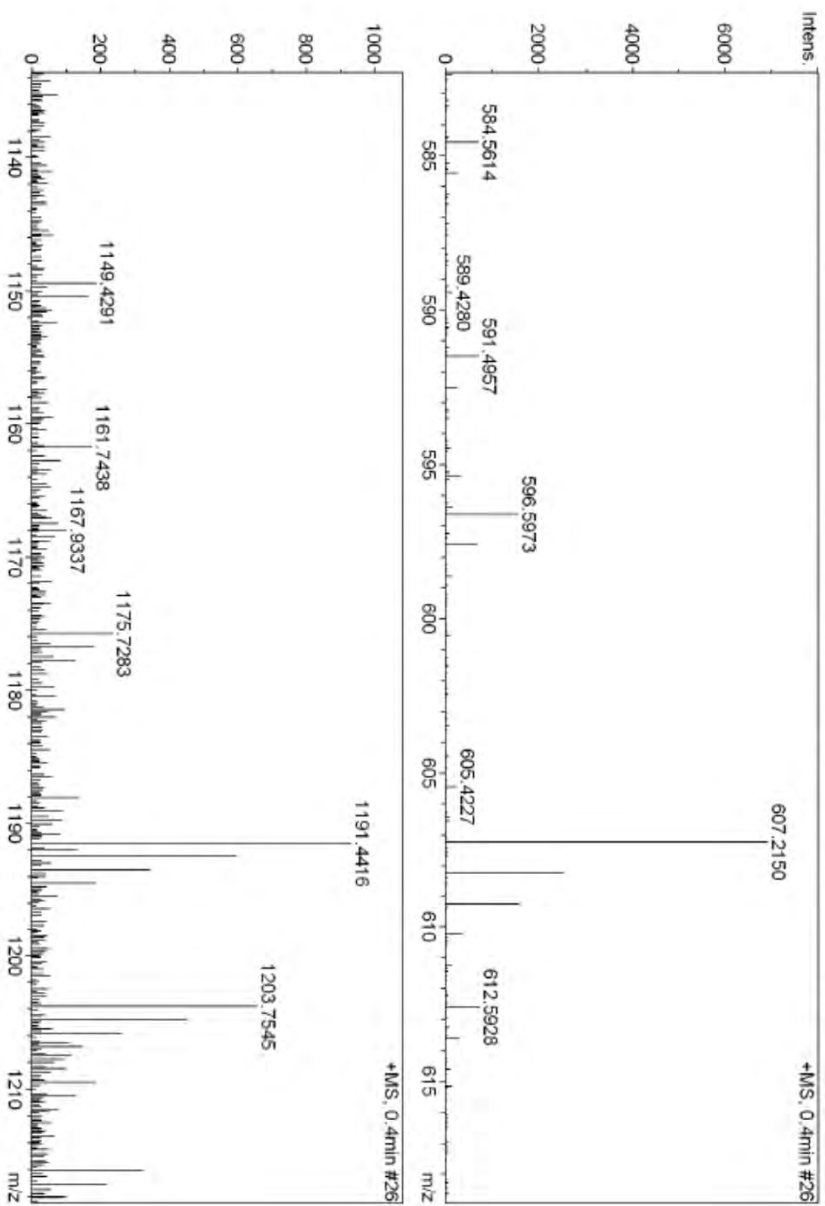
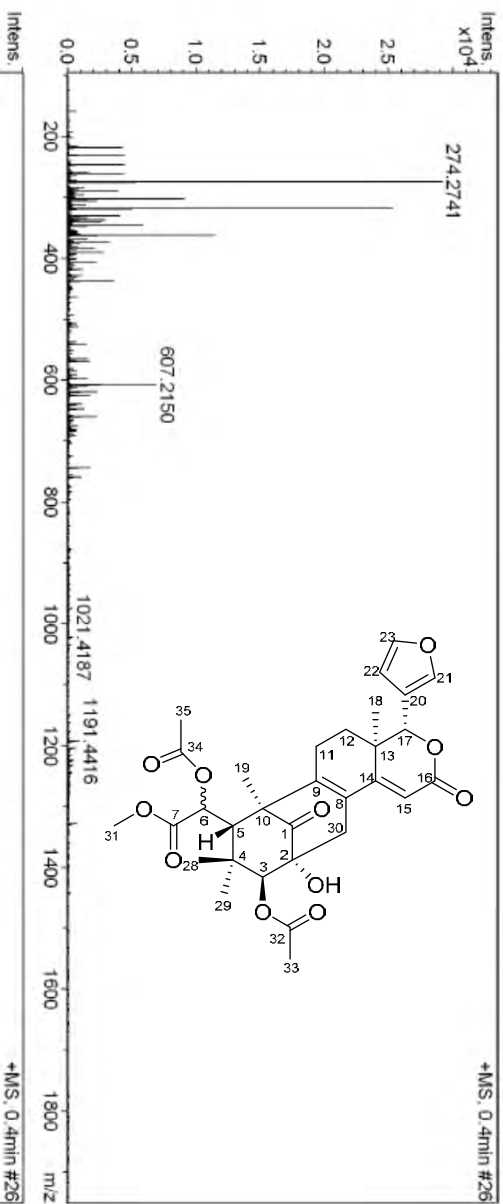
Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e <sup>-</sup> Conf	N-Rule
585.2295	1	C 31 H 37 O 11	7.04	585.2330	6.0	3.5	128.8	13.5	even	ok
607.2151	1	C 31 H 36 Na O 11	100.00	607.2150	-0.3	-0.2	85.5	13.5	even	ok
1191.4416	1	C 62 H 72 Na O 22	100.00	1191.4407	-0.7	-0.8	50.9	26.5	even	ok

# HR-ESIMS for compound 8

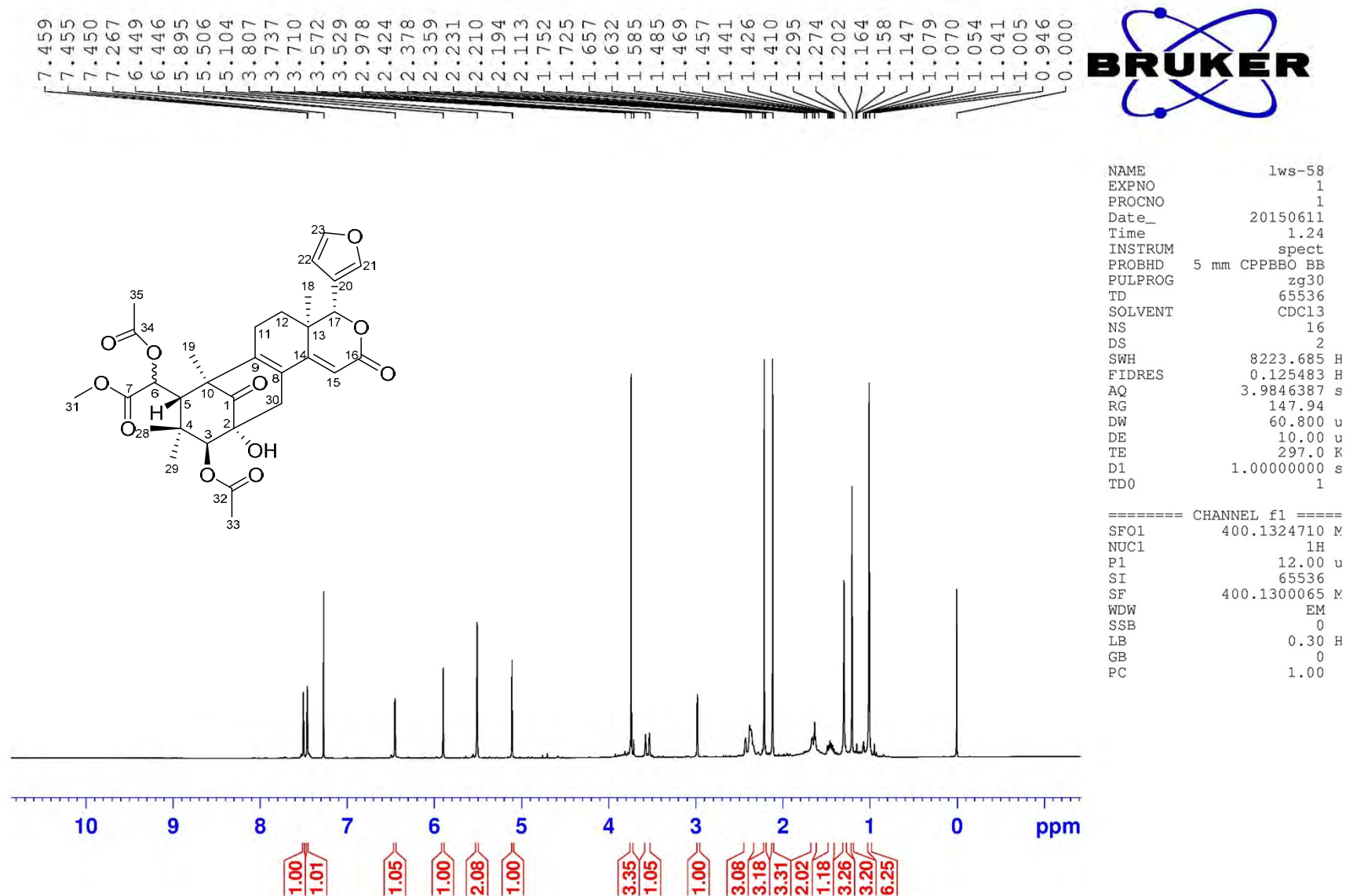
## Generic Display Report

### Analysis Info

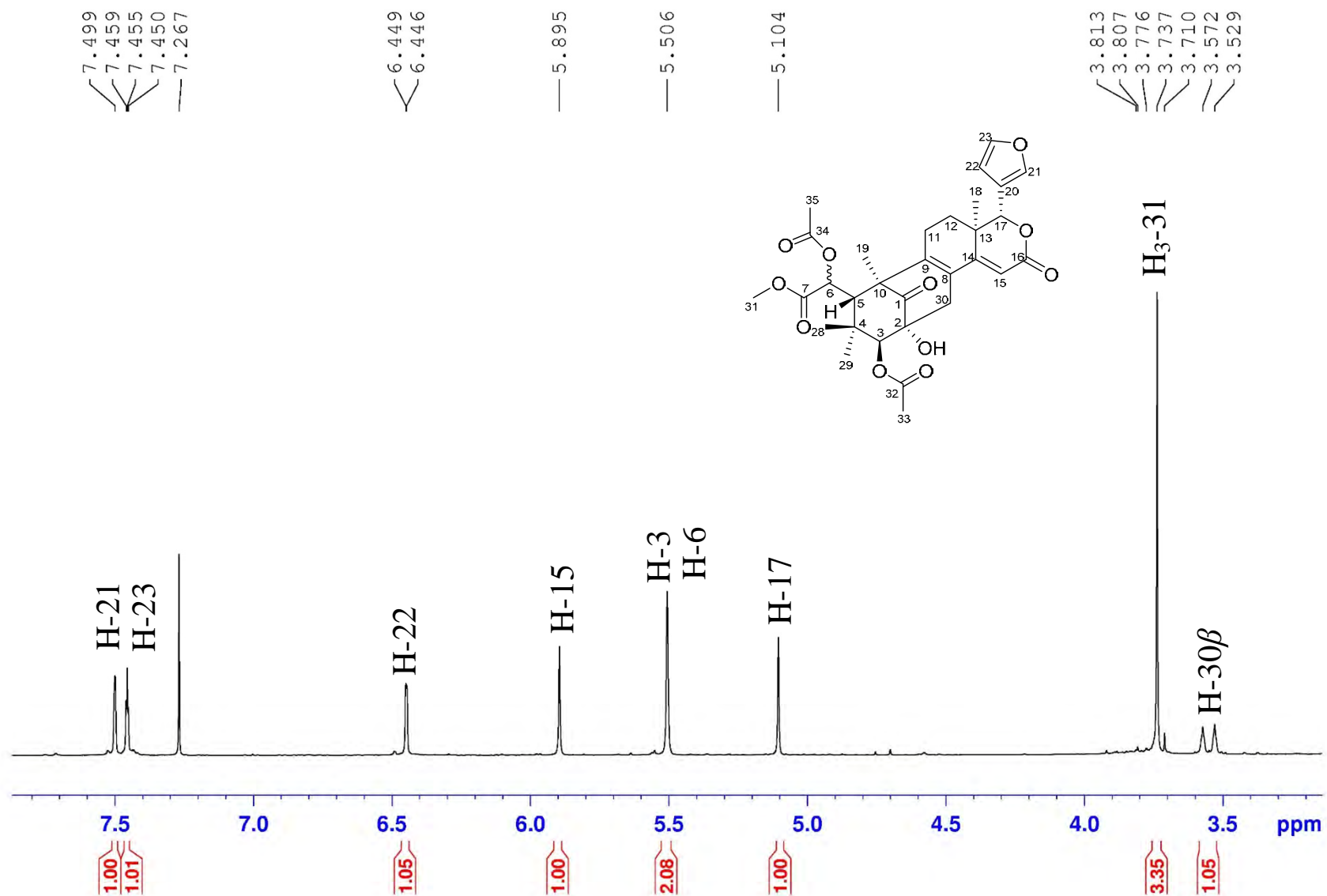
Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-58\_pos\_12\_01\_1859.d Acquisition Date 5/19/2016 8:54:55 AM  
Method LC\_Direct Infusion\_pos\_100-1000mz.m Operator SCSIO  
Sample Name liwanshan\_lws-58\_pos Instrument maxIs  
Comment



# <sup>1</sup>H NMR (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>

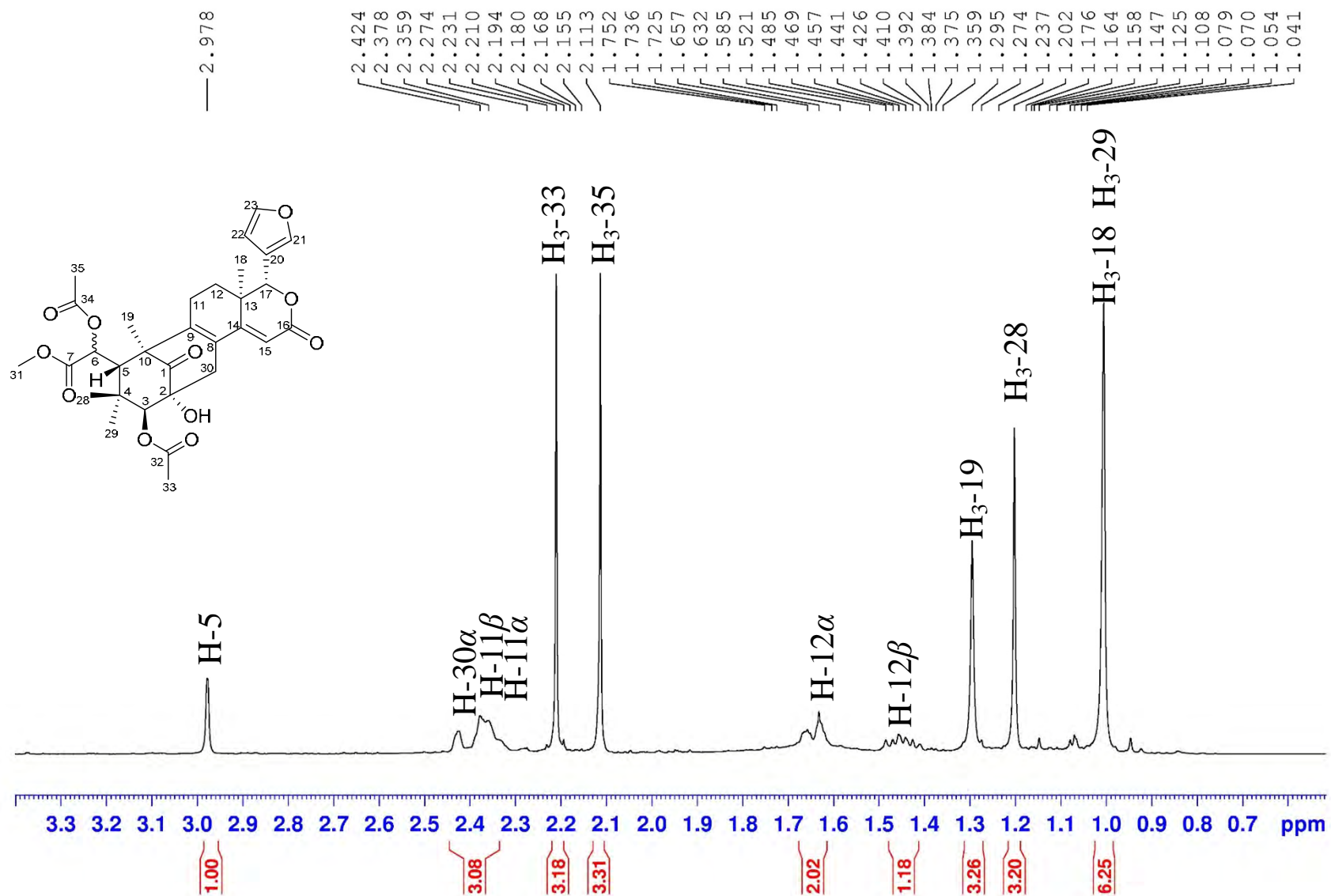


$^1\text{H}$  NMR (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$

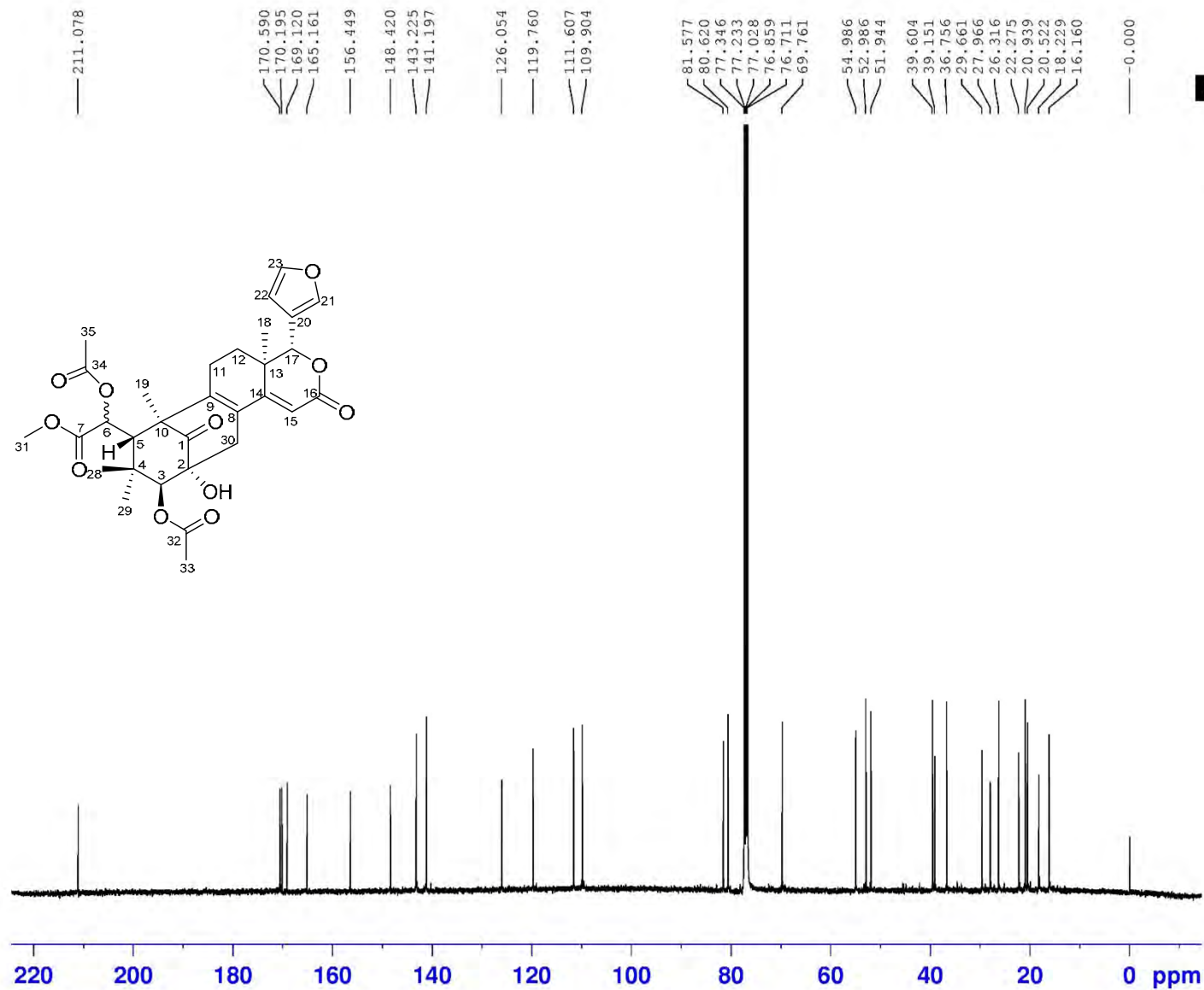




$^1\text{H}$  NMR (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



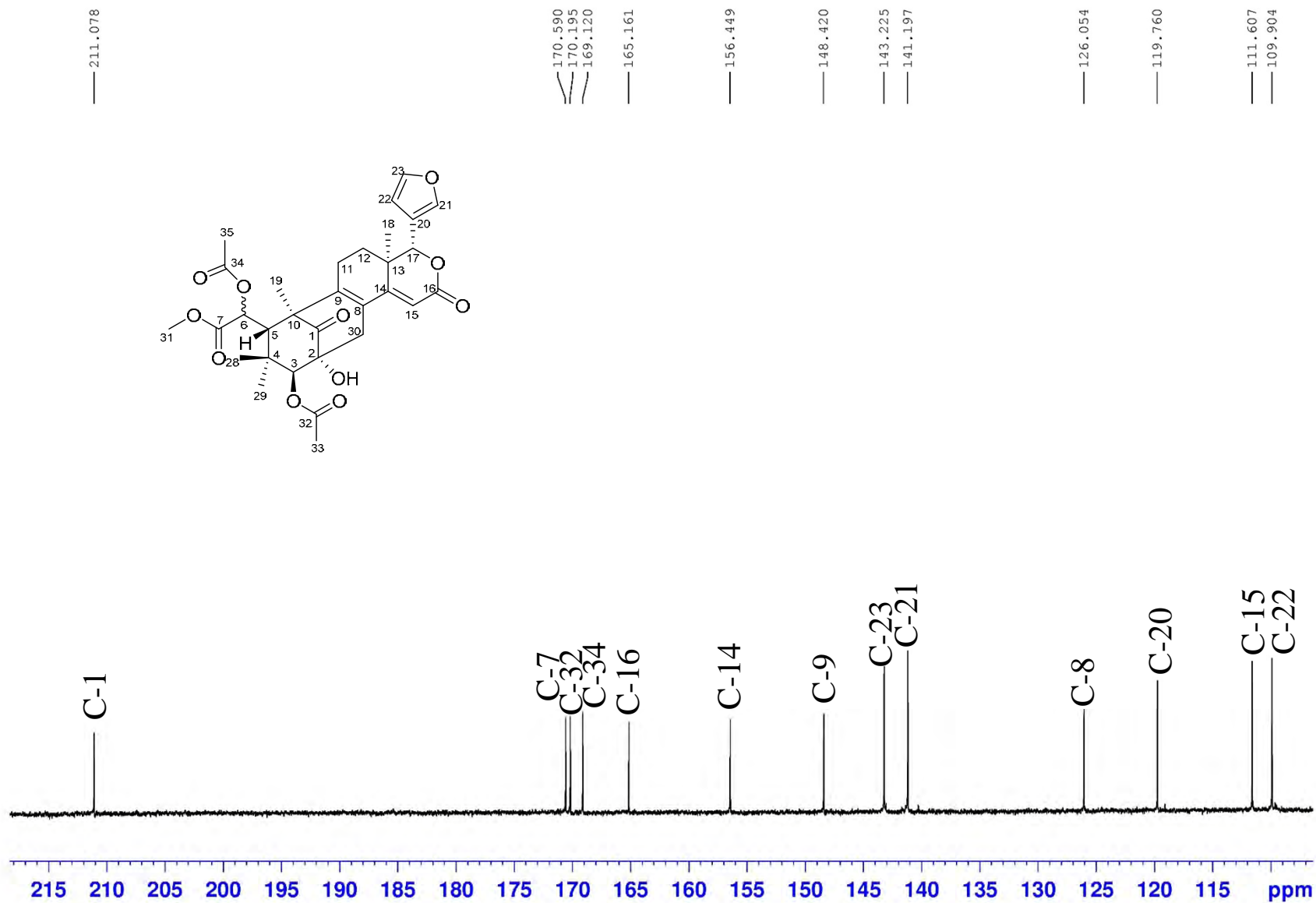
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



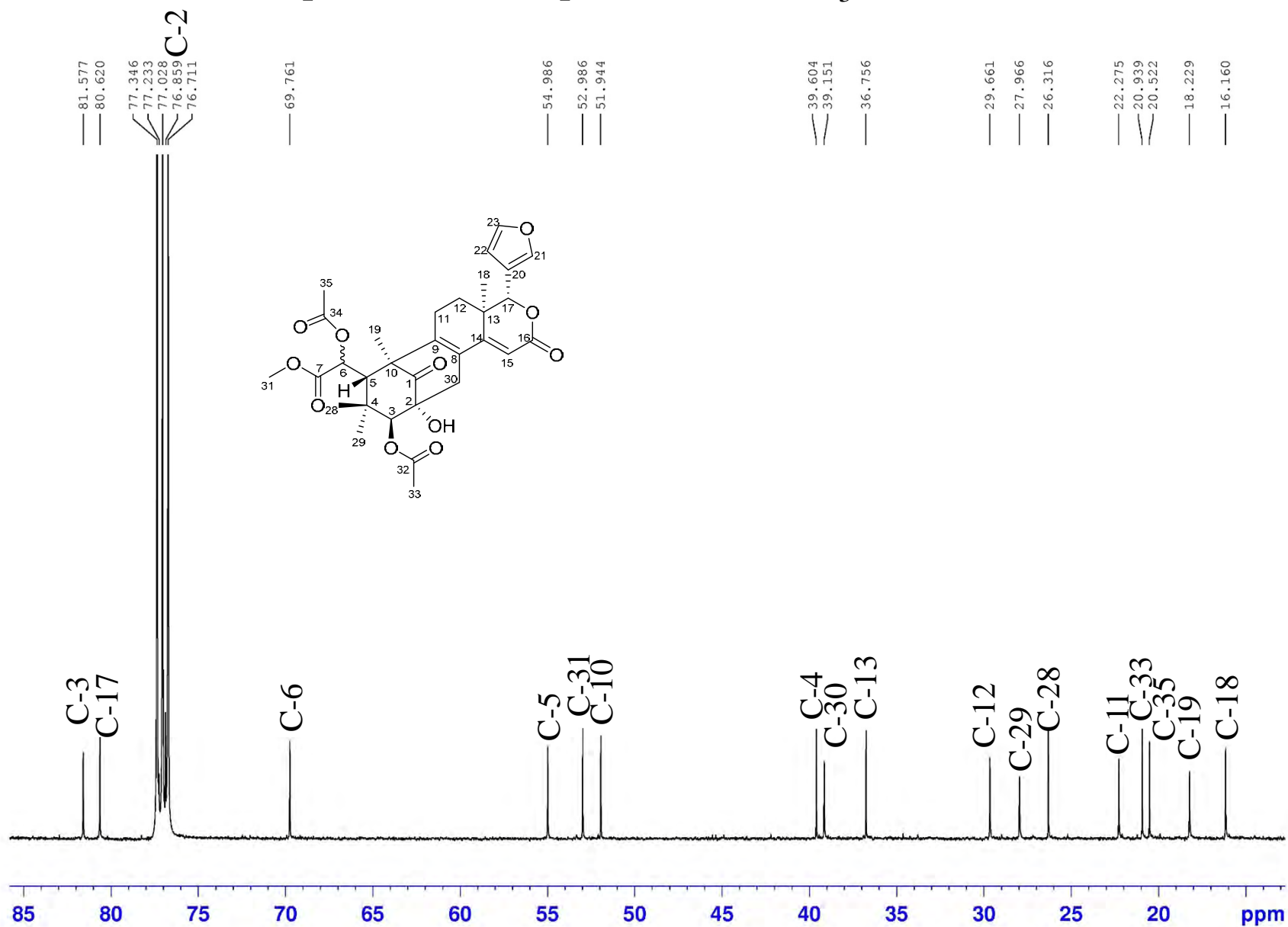
NAME lws-58  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20150611  
 Time 17.57  
 INSTRUM spect  
 PROBHD 5 mm CPPBBO BB  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT  $\text{CDCl}_3$   
 NS 3000  
 DS 4  
 SWH 24038.461 F  
 FIDRES 0.366798 F  
 AQ 1.3631988 s  
 RG 102.3  
 DW 20.800  $\mu$   
 DE 18.00  $\mu$   
 TE 297.0 K  
 D1 2.00000000 s  
 D11 0.03000000 s  
 TD0 1

===== CHANNEL f1 =====  
 SFO1 100.6233324 M  
 NUC1  $^{13}\text{C}$   
 P1 10.00  $\mu$   
 SI 32768  
 SF 100.6127690 M  
 WDW EM  
 SSB 0  
 LB 1.00 F  
 GB 0  
 PC 1.40

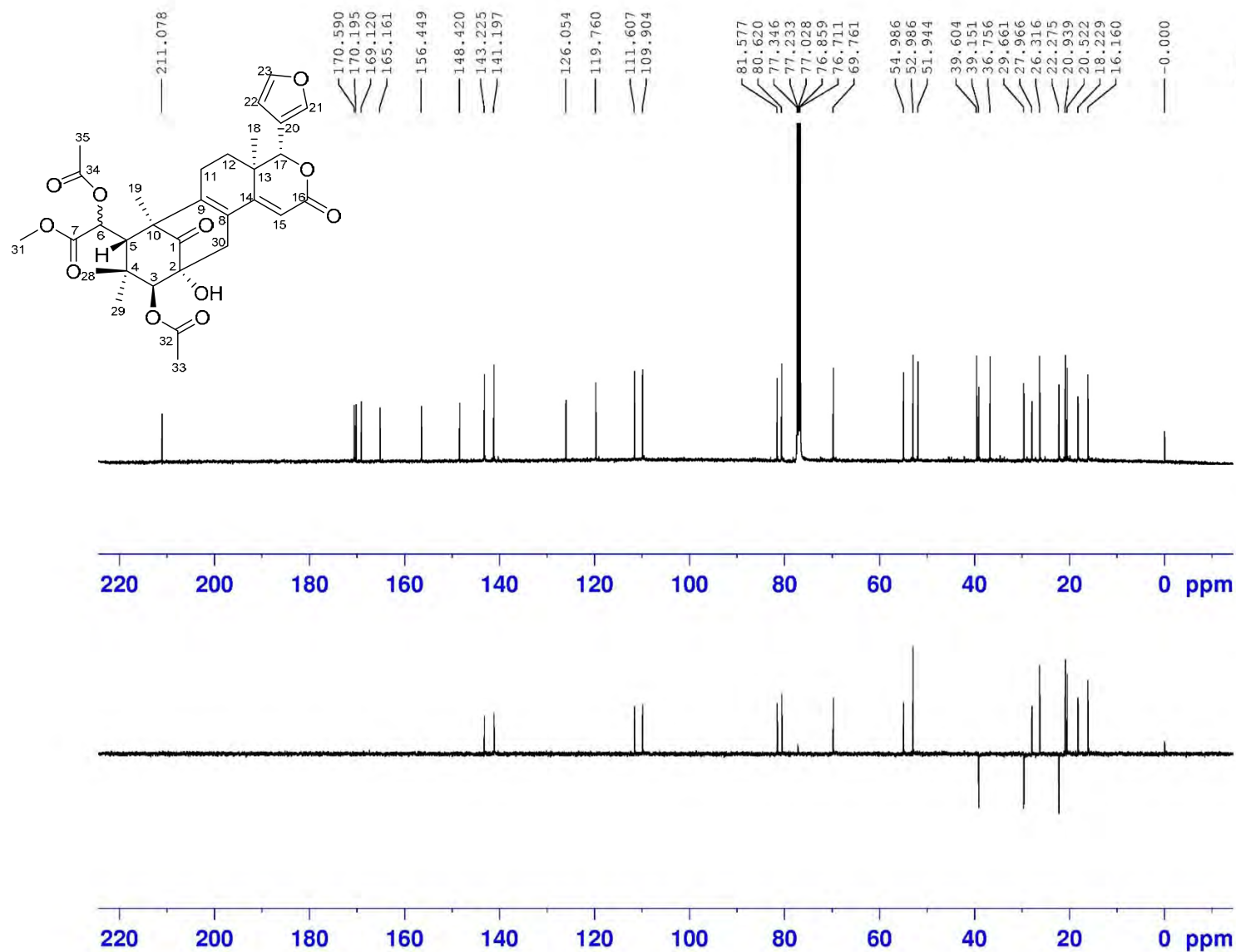
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



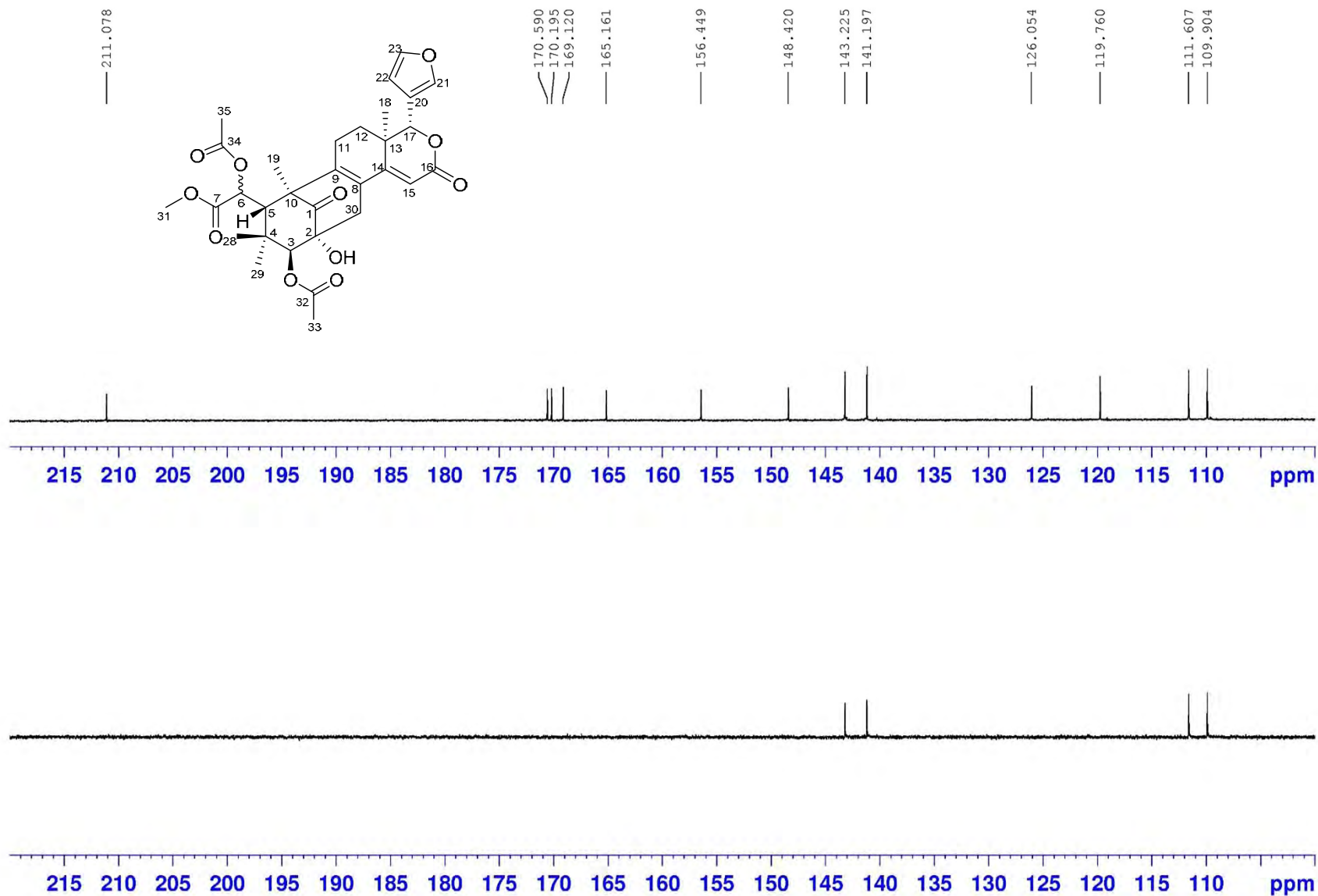
```

NAME          lws-58
EXPNO          103
PROCNO         1
Date_          20150613
Time           17.26
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        deptspl35
TD             65536
SOLVENT        CDCl3
NS             300
DS             4
SWH            24038.461 F
FIDRES         0.366798 F
AQ             1.3631988 s
RG             91.64
DW             20.800 s
DE             18.00 s
TE             297.0 F
CNST2          145.0000000
D1             2.00000000 s
D2             0.00344828 s
D12            0.00002000 s
TD0            1
    
```

```

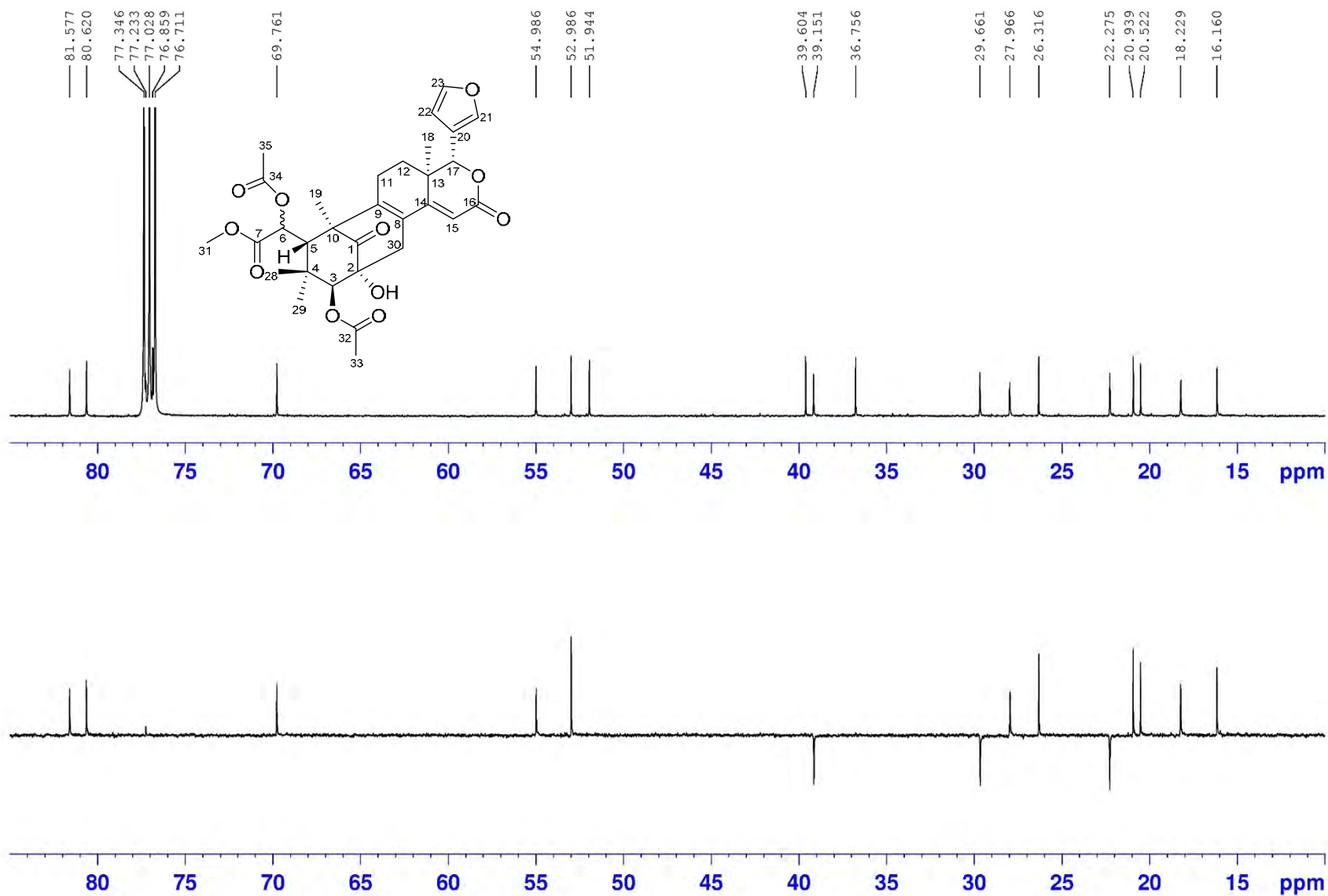
===== CHANNEL f1 =====
SFO1          100.6233324 M
NUC1           13C
P1             10.00 s
P13            2000.00 s
SI             32768
SF             100.6127690 M
WDW            EM
SSB            0
LB             1.00 F
GB             0
PC             1.40
    
```

# DEPT135 (100 MHz) spectrum of compound **8** in CDCl<sub>3</sub>

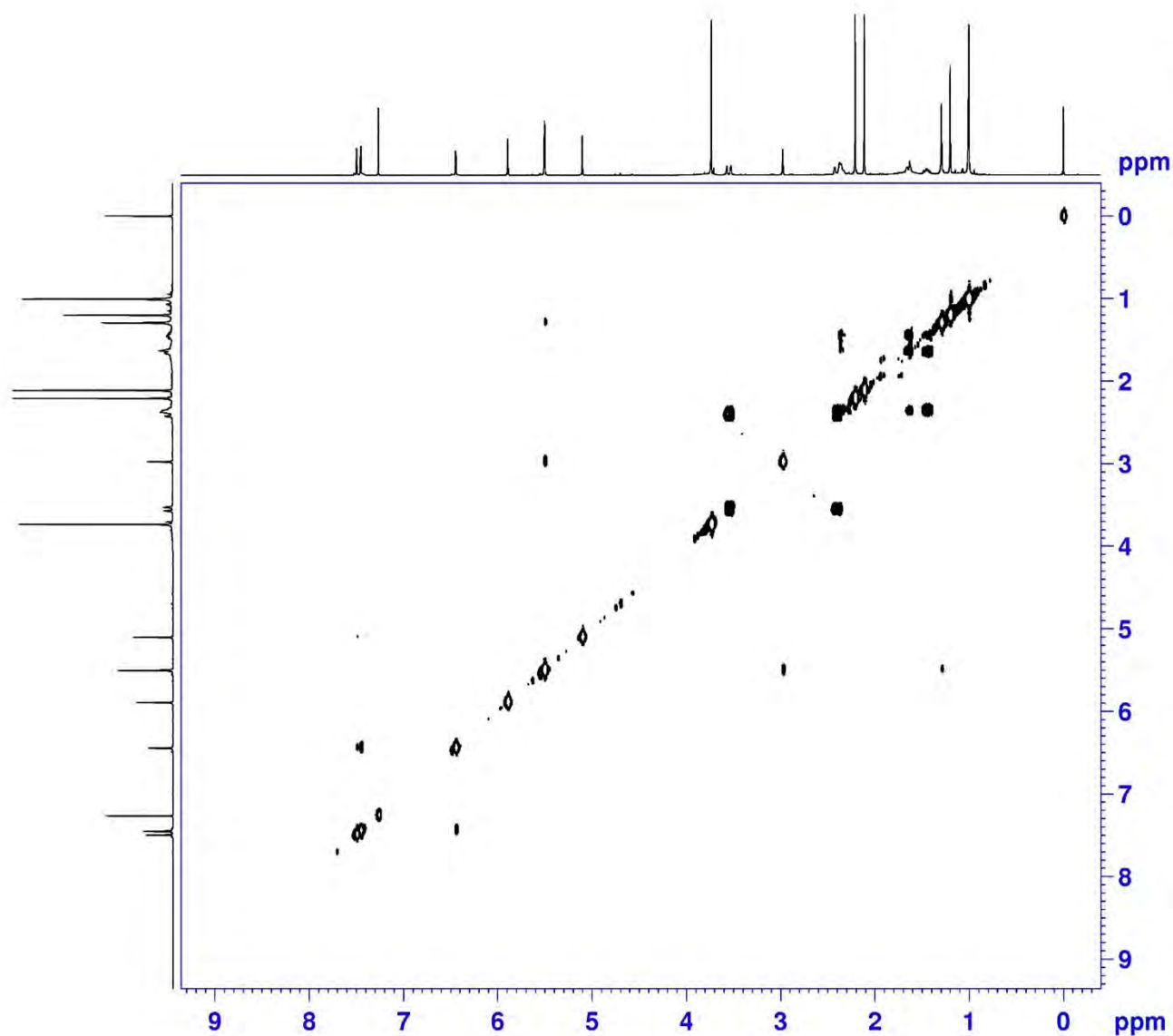




DEPT135 (100 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



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



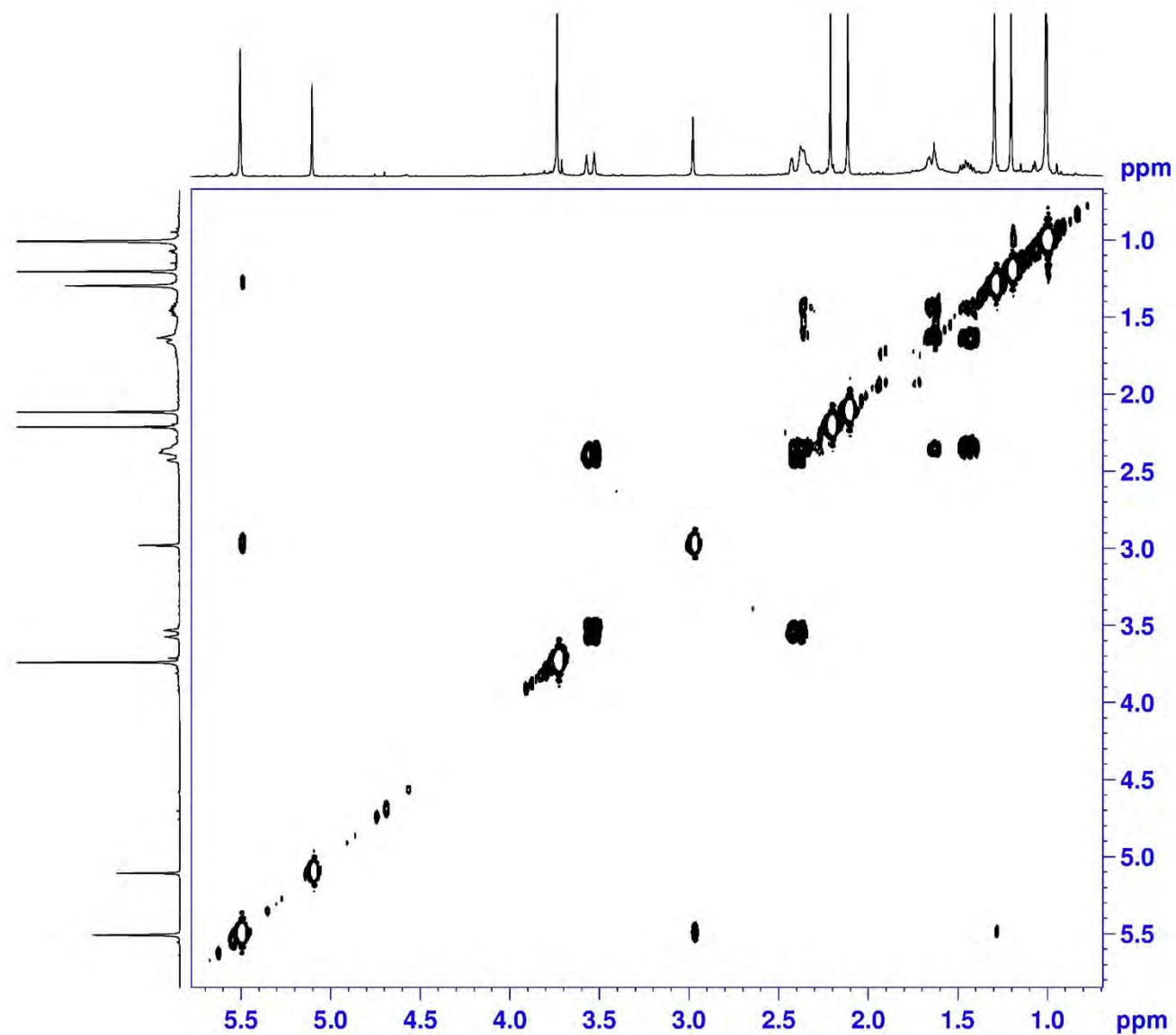
```

NAME                lws-58
EXPNO                4
PROCNO              1
Date_               20150611
Time                18.37
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             cosygpppgf
TD                  2048
SOLVENT             CDCl3
NS                   16
DS                   8
SWH                 3906.250 Hz
FIDRES              1.907349 Hz
AQ                  0.2621940 s
RG                   208.5
DW                  128.000 us
DE                   10.00 us
TE                   297.0 K
D0                   0.00000300 s
D1                   1.89678097 s
D11                  0.03000000 s
D12                  0.00002000 s
D13                  0.00000400 s
D16                  0.00020000 s
IN0                  0.00025600 s
  
```

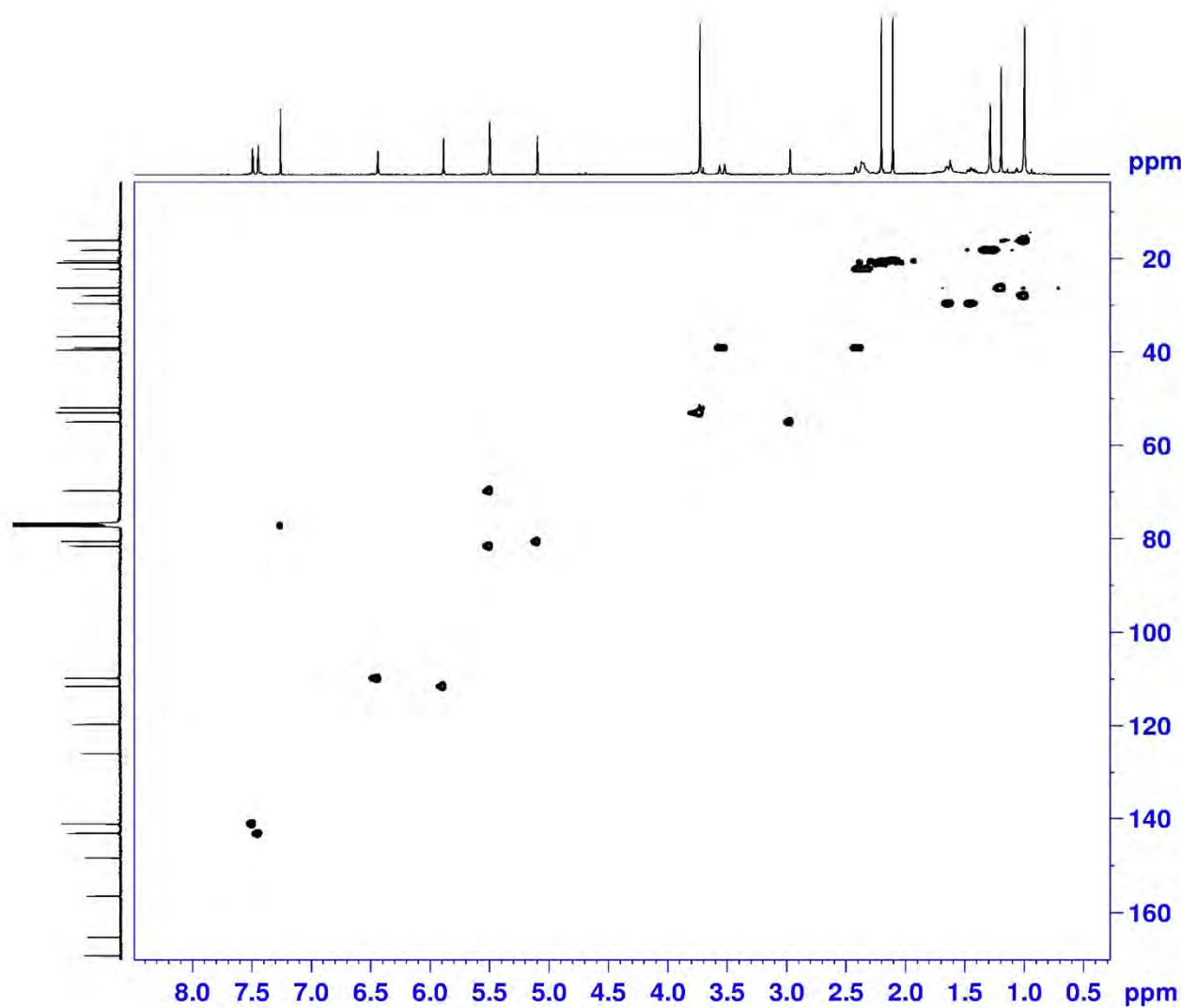
```

===== CHANNEL f1 =====
SFO1                400.1318006 MHz
NUC1                 1H
P0                   12.00 us
P1                   12.00 us
P17                  2500.00 us
ND0                   1
TD                   128
SFO1                400.1318 MHz
FIDRES              30.517578 Hz
SW                   9.762 MHz
FnMODE              QF
SI                   1024
SF                   400.1300098 MHz
WDW                  QSINE
SSB                   0
LB                   0.00 Hz
GB                   0
PC                   1.40
SI                   1024
MC2                  QF
SF                   400.1300098 MHz
WDW                  QSINE
SSB                   0
  
```

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



# HSQC (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



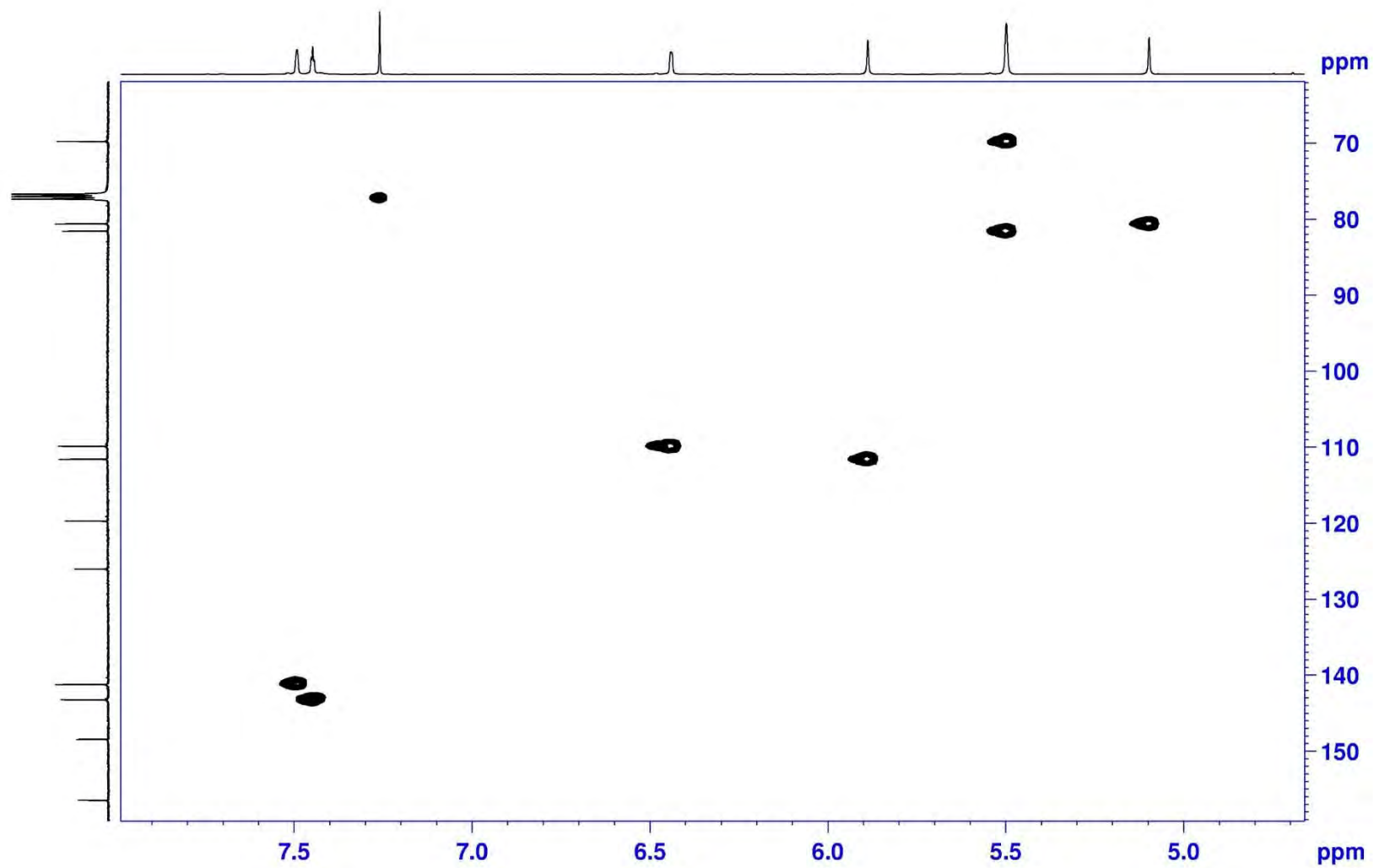
```

NAME                lws-58
EXPNO                5
PROCNO               1
Date_                20150611
Time                19.54
INSTRUM              spect
PROBHD               5 mm CPBBO BB
PULPROG              hsqcetgpsi2
TD                   1024
SOLVENT              CDCl3
NS                   16
DS                   16
SWH                  4302.926 Hz
FIDRES               4.202076 Hz
AQ                   0.1190388 sec
RG                   208.5
DW                   116.200 usec
DE                   10.00 usec
TE                   297.0 K
CNST2                145.0000000
D0                   0.00000300 sec
D1                   1.46497905 sec
D4                   0.00172414 sec
D11                  0.03000000 sec
D16                  0.00020000 sec
D24                  0.00086207 sec
IN0                  0.00002080 sec
ZGPTNS
  
```

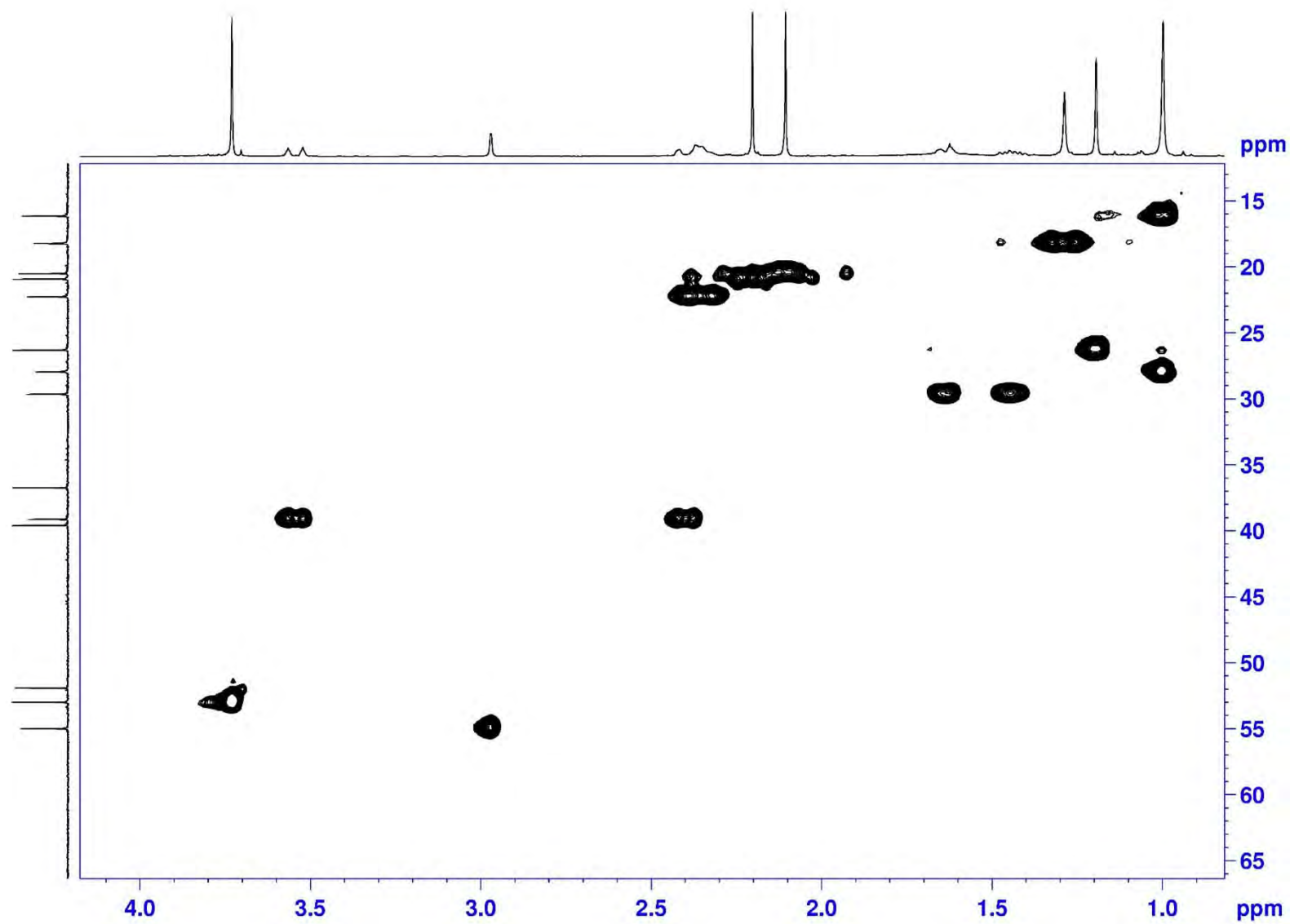
```

===== CHANNEL f1 =====
SFO1                400.1320007 MHz
NUC1                 1H
P1                   12.00 usec
P2                   24.00 usec
P28                  0.00 usec
ND0                  2
TD                   256
SFO1                100.6233 MHz
FIDRES               93.900238 Hz
SW                   238.896 ppm
FnMODE              Echo-Antiecho
SI                   1024
SF                   400.1300079 MHz
WDW                  QSINE
SSB                  2
LB                   0.00 Hz
GB                   0
PC                   1.40
SI                   1024
MC2                  echo-antiecho
SF                   100.6127685 MHz
WDW                  QSINE
  
```

HSQC (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$

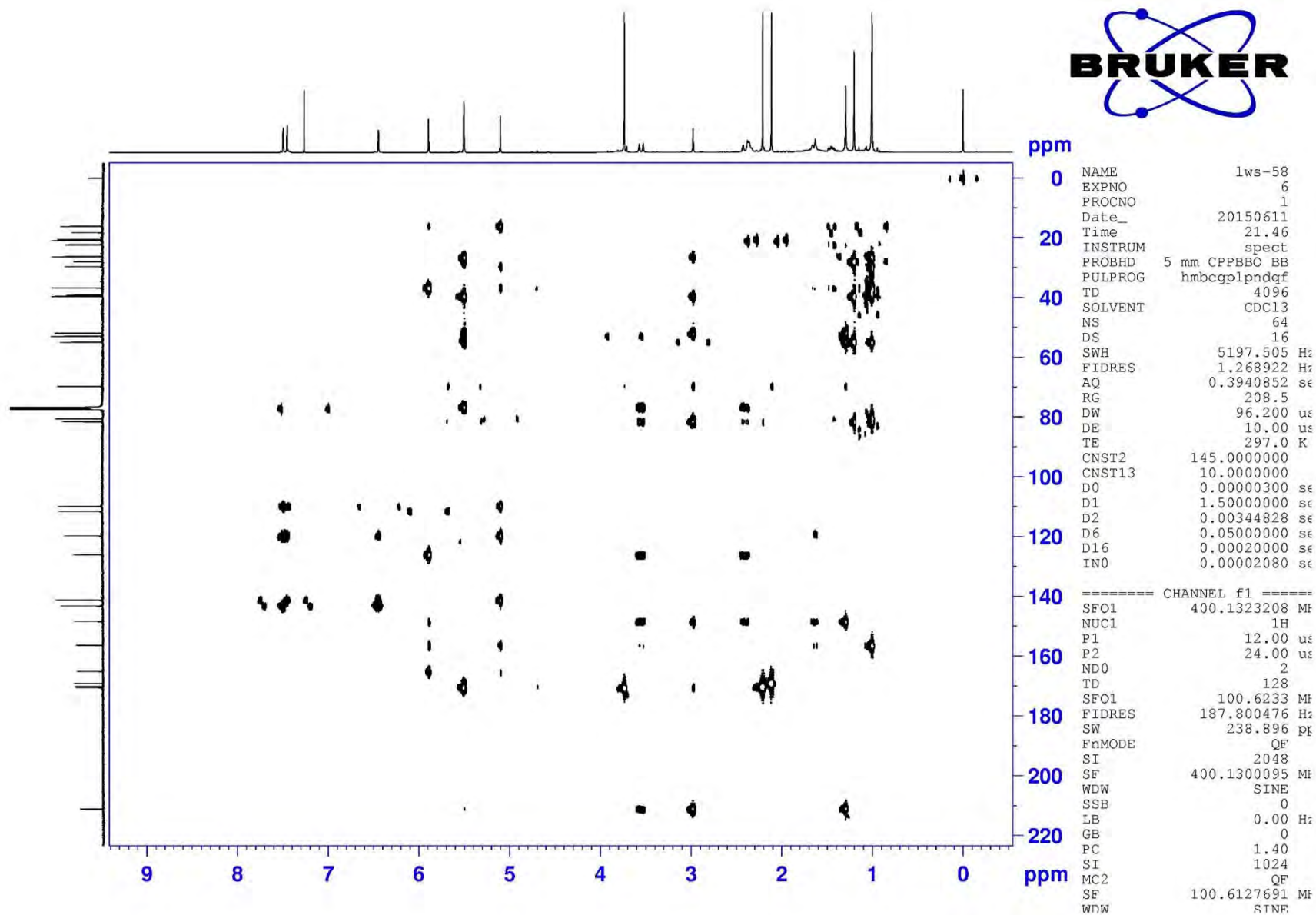


HSQC (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$

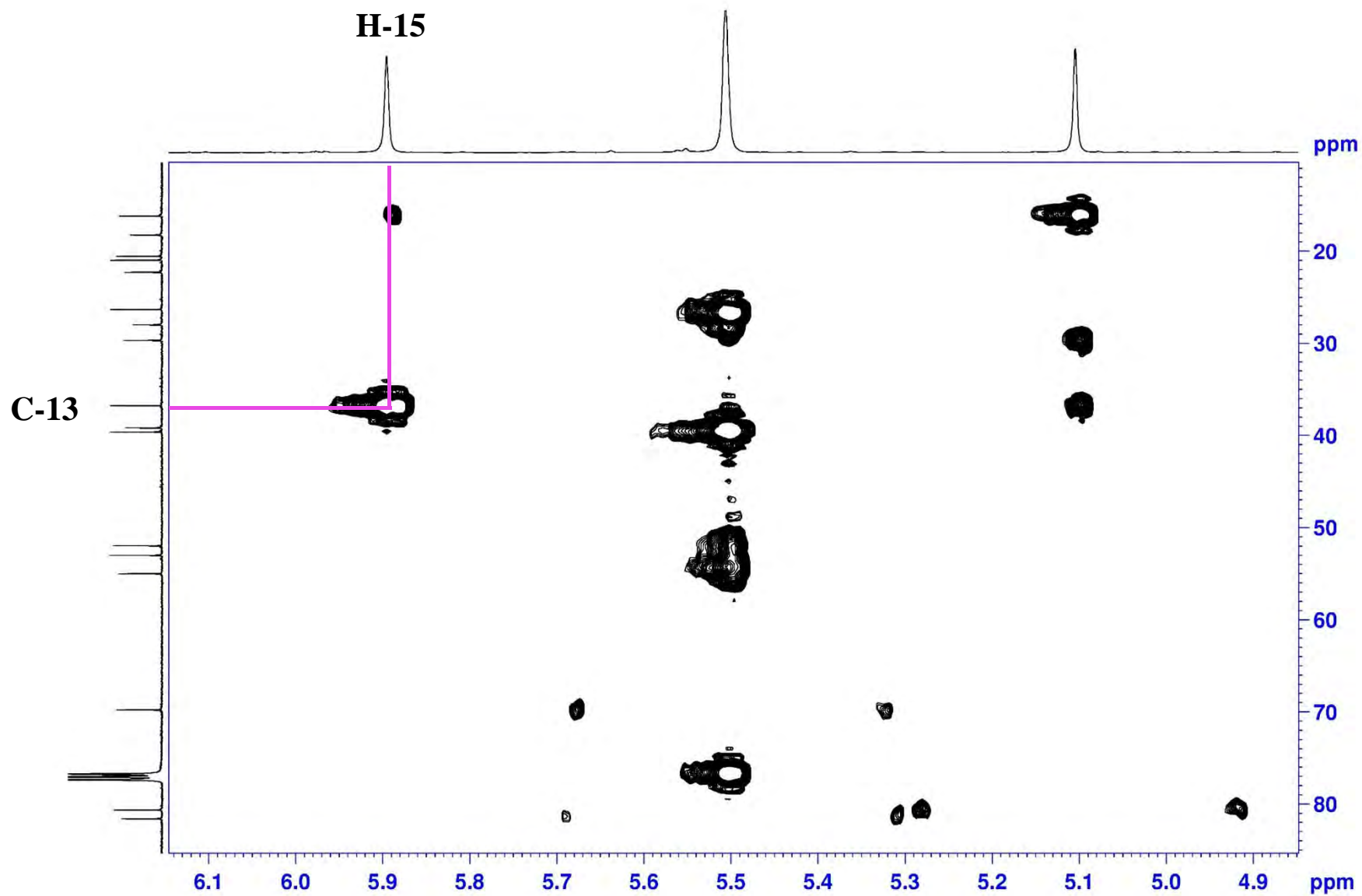




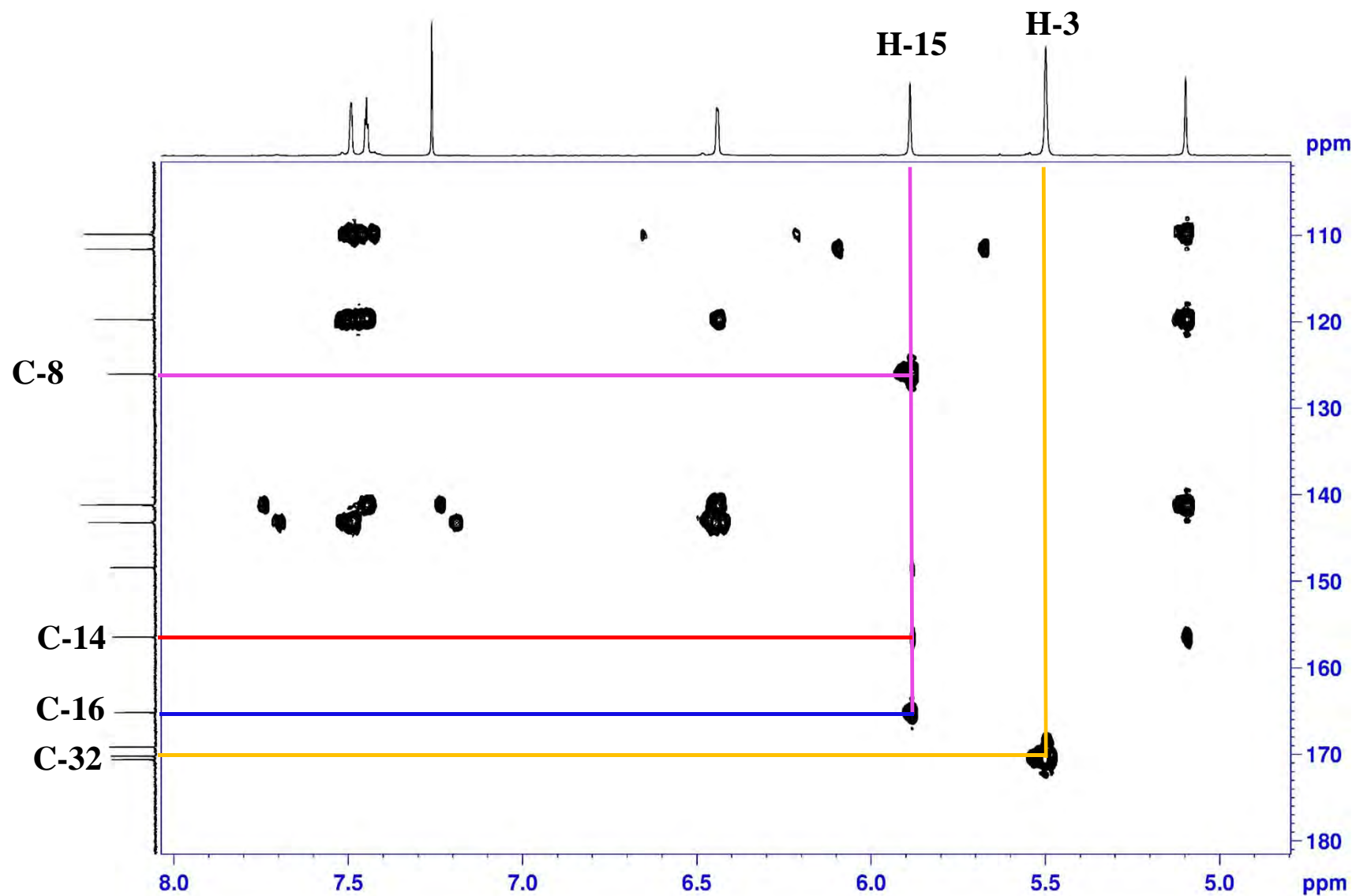
# HMBC (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



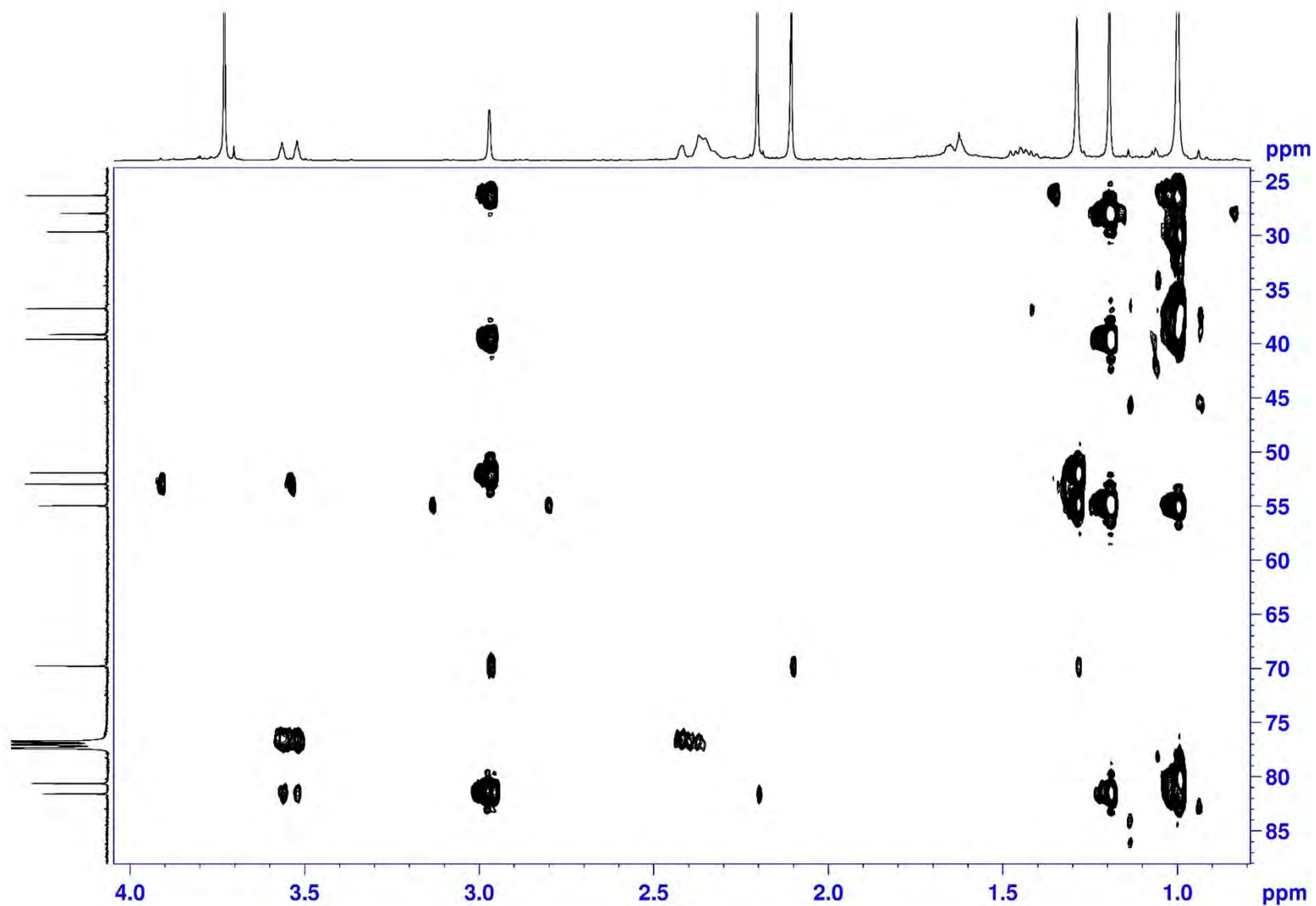
HMBC (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



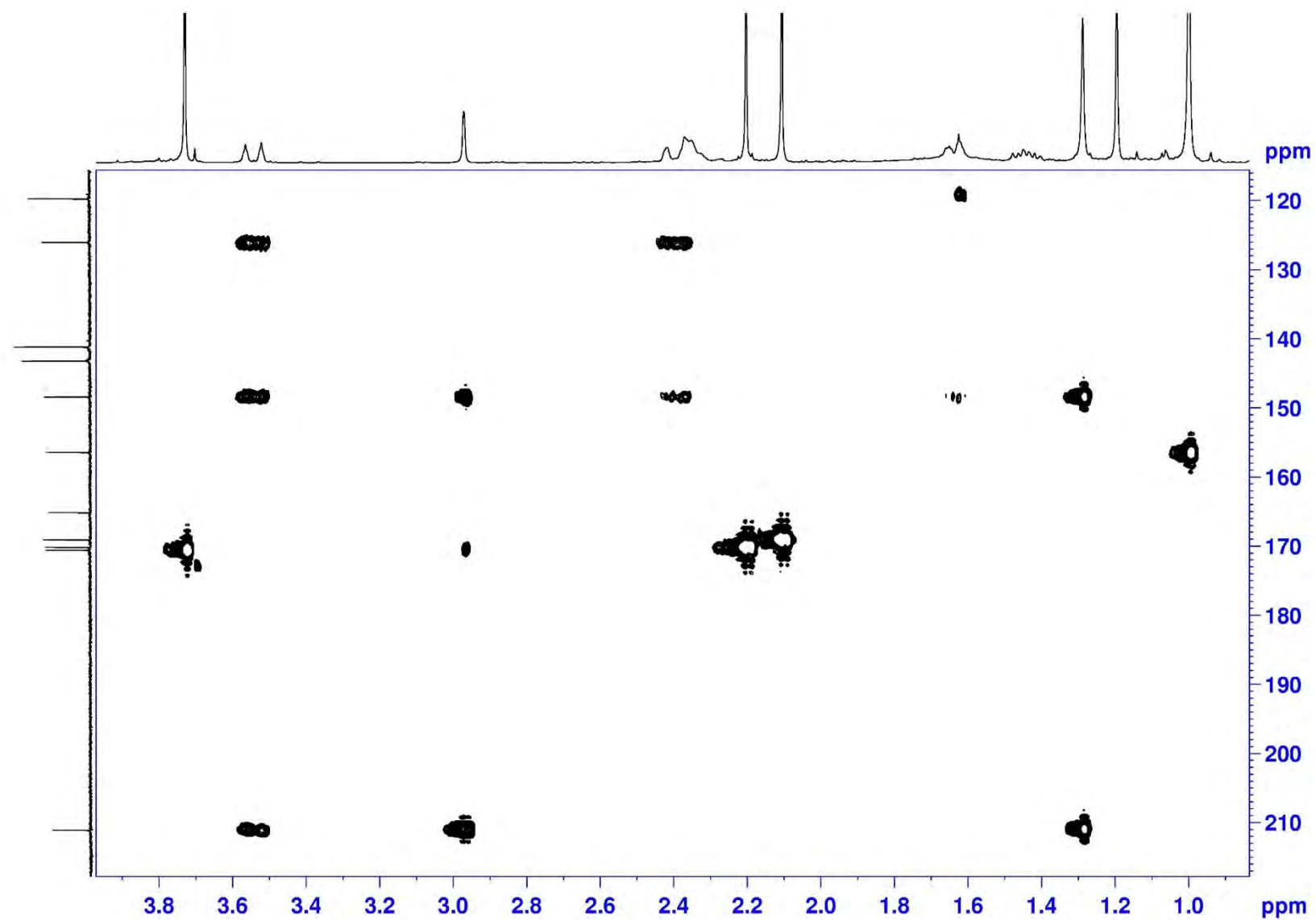
HMBC (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



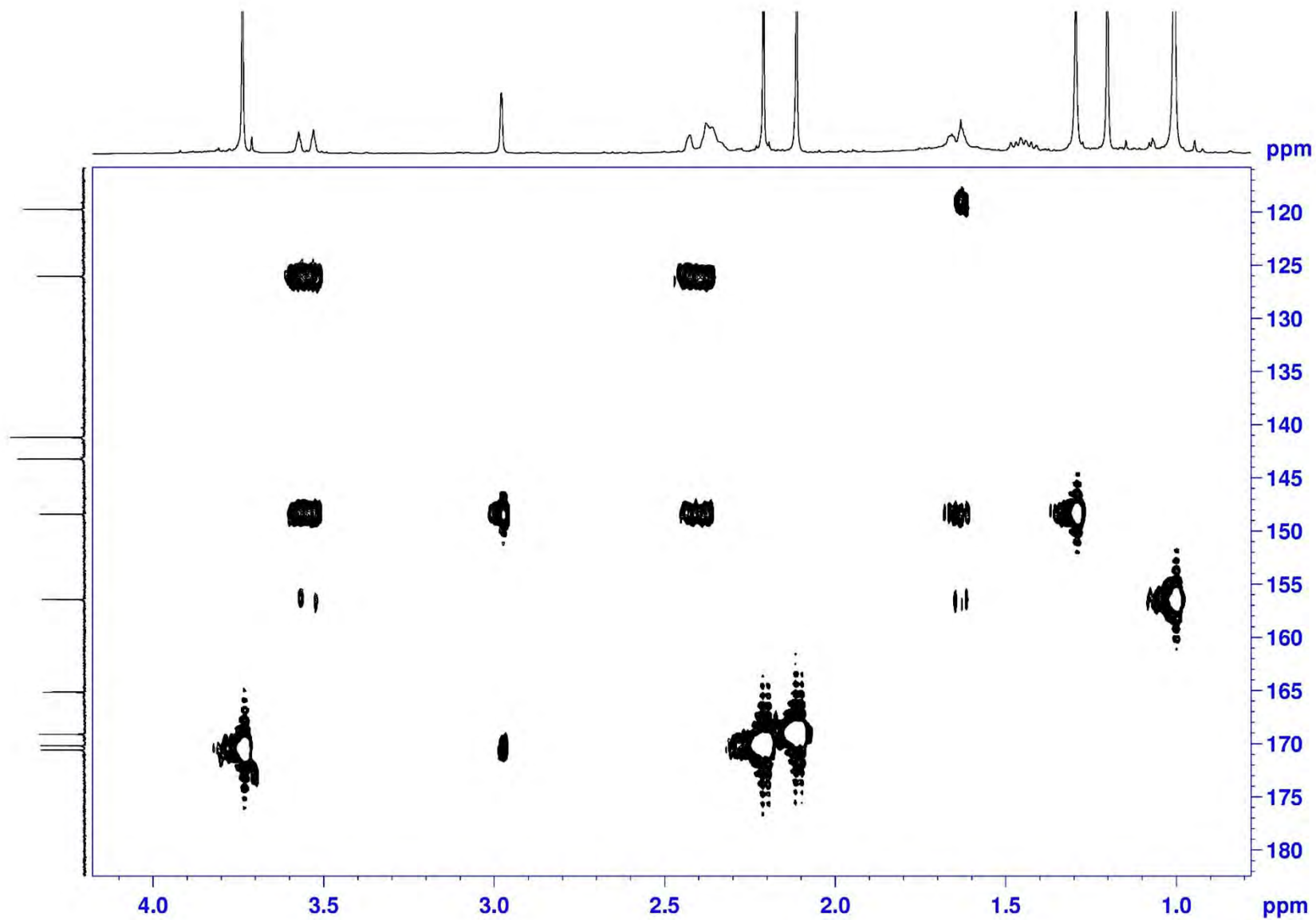
HMBC (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$

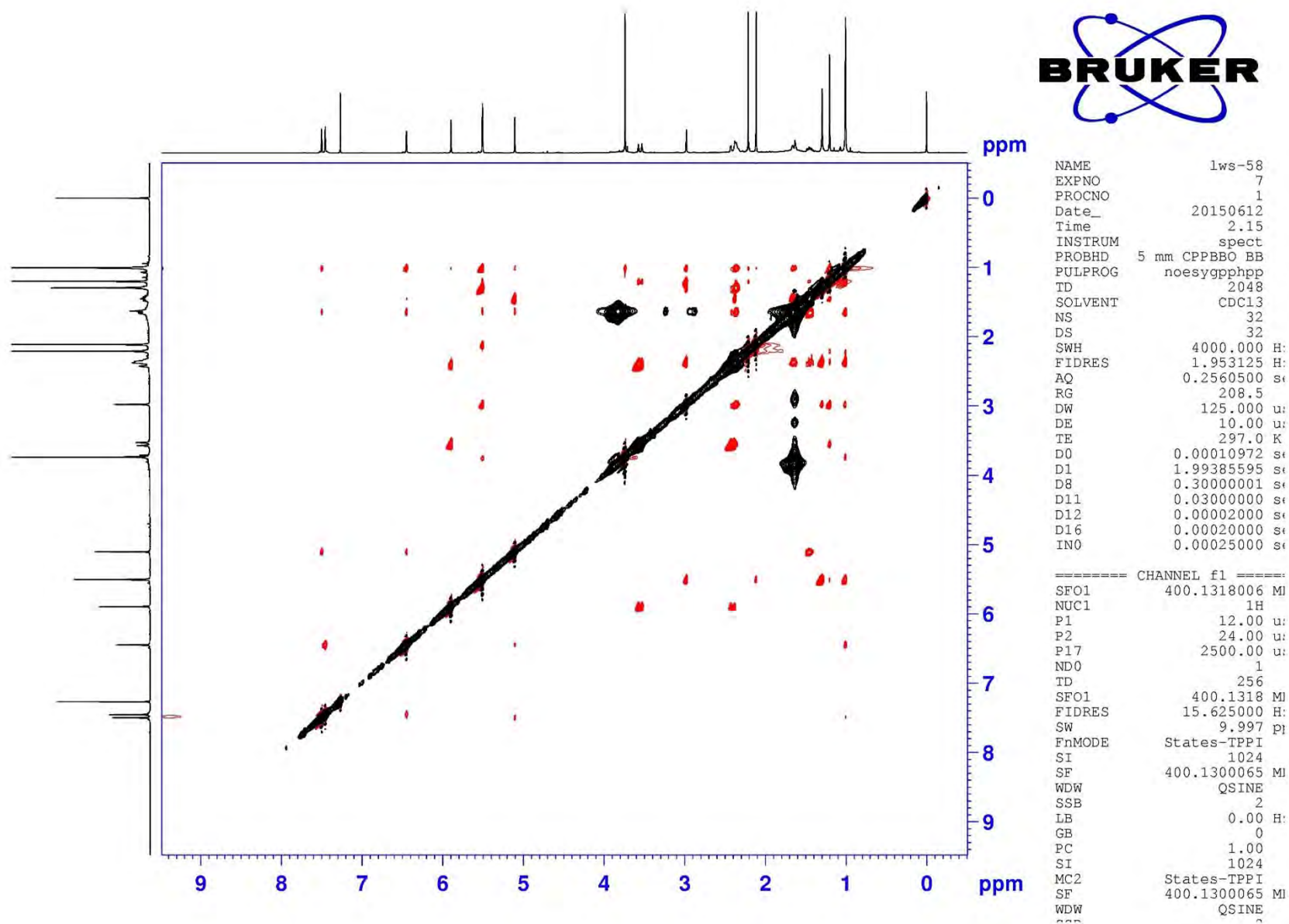


HMBC (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>

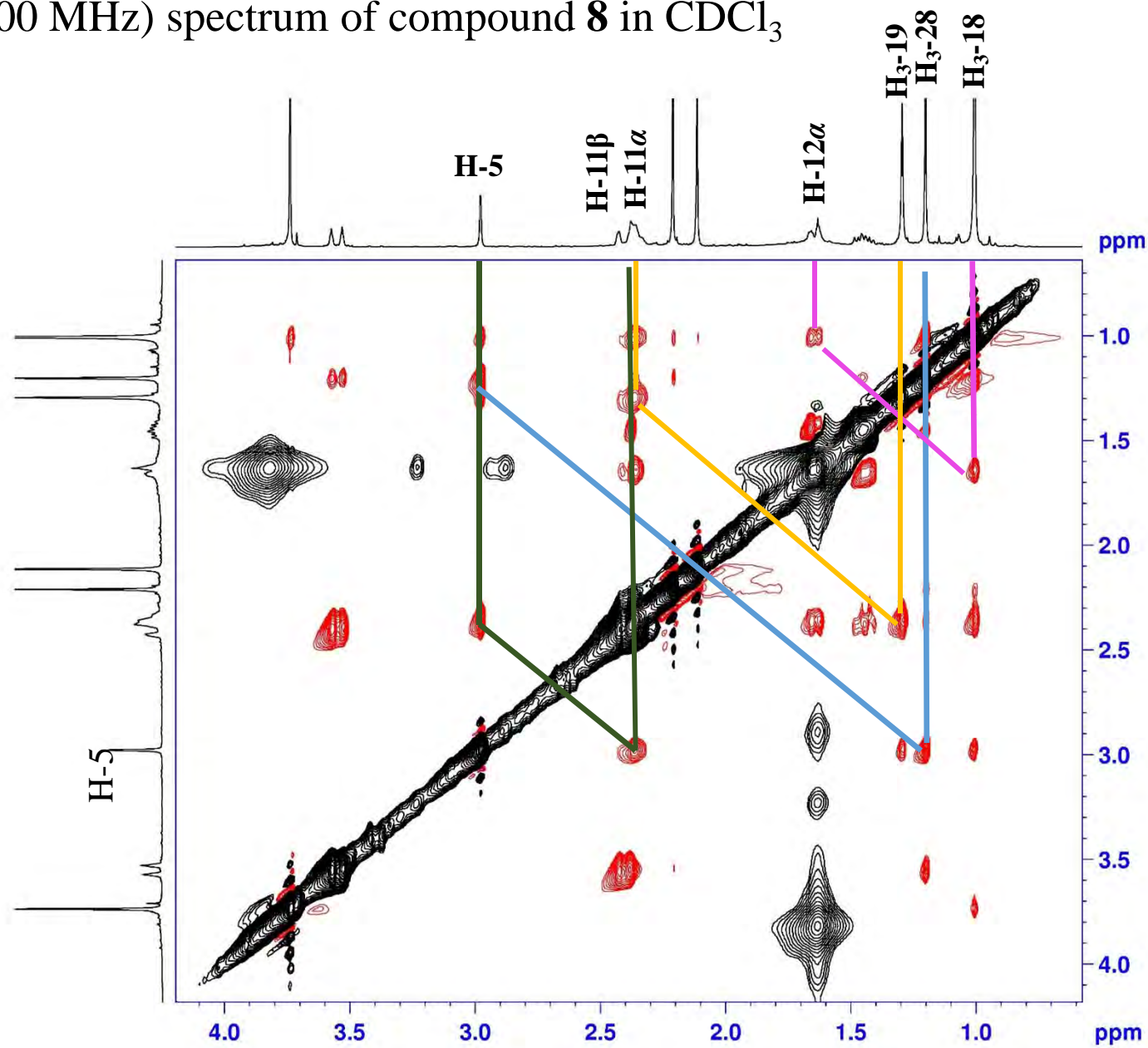




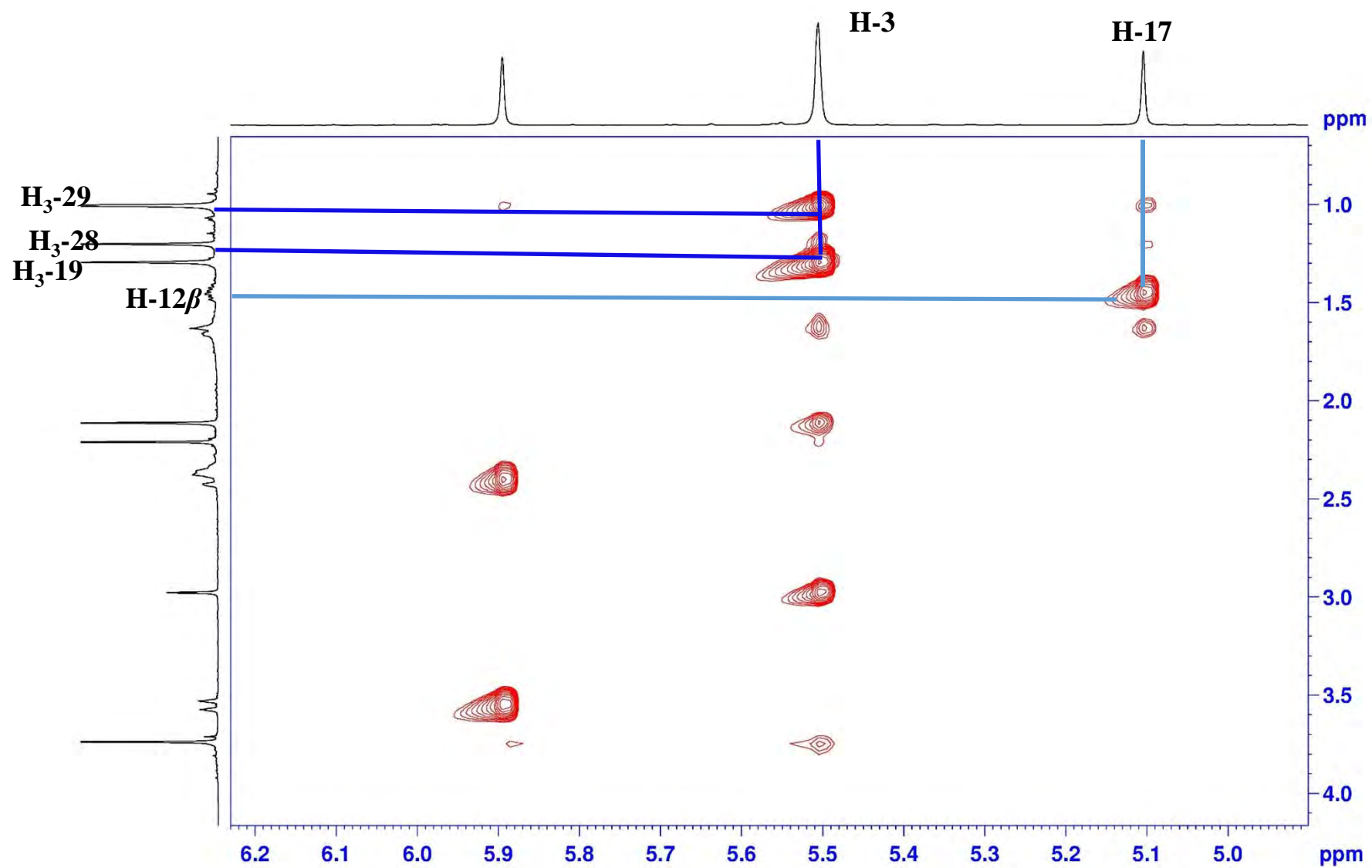
# NOESY (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **8** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **8** in CDCl<sub>3</sub>



# HR-ESIMS for compound 9

## Mass Spectrum SmartFormula Report

### Analysis Info

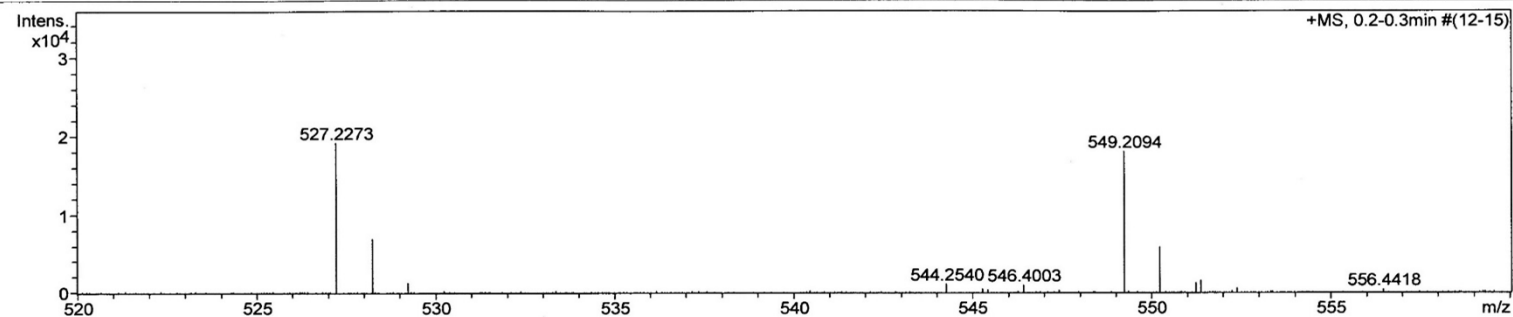
Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-86\_pos\_24\_01\_834.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-86\_pos  
 Comment

Acquisition Date 11/18/2015 10:10:28 PM

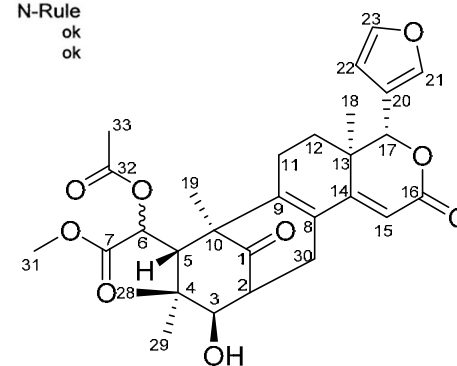
Operator SCSIO  
 Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
527.2273	1	C <sub>29</sub> H <sub>35</sub> O <sub>9</sub>	100.00	527.2276	0.5	0.2	23.5	12.5	even	ok
549.2094	1	C <sub>29</sub> H <sub>34</sub> NaO <sub>9</sub>	100.00	549.2095	0.1	0.1	4.3	12.5	even	ok



# HR-ESIMS for compound 9

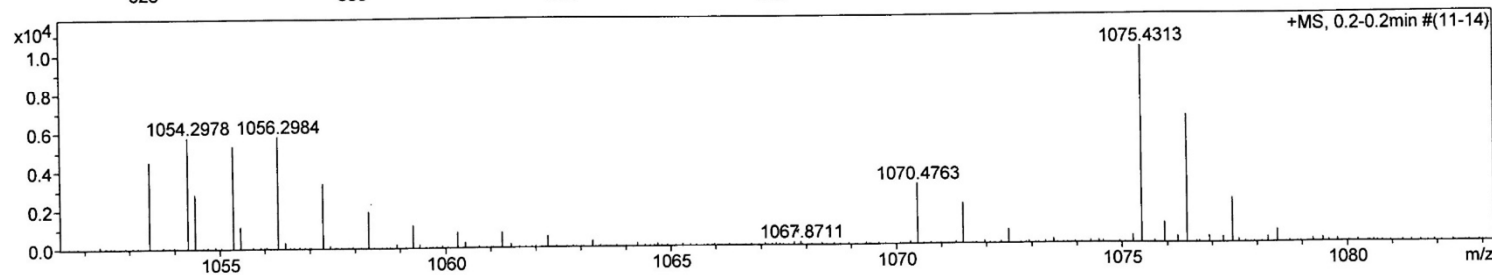
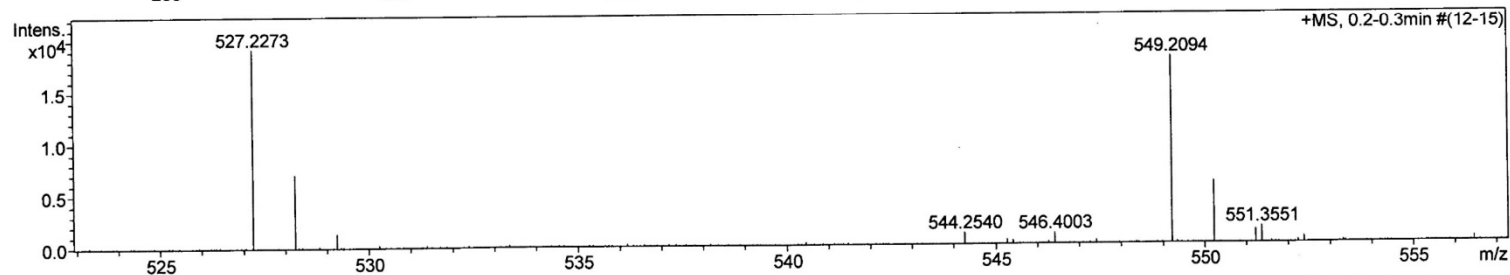
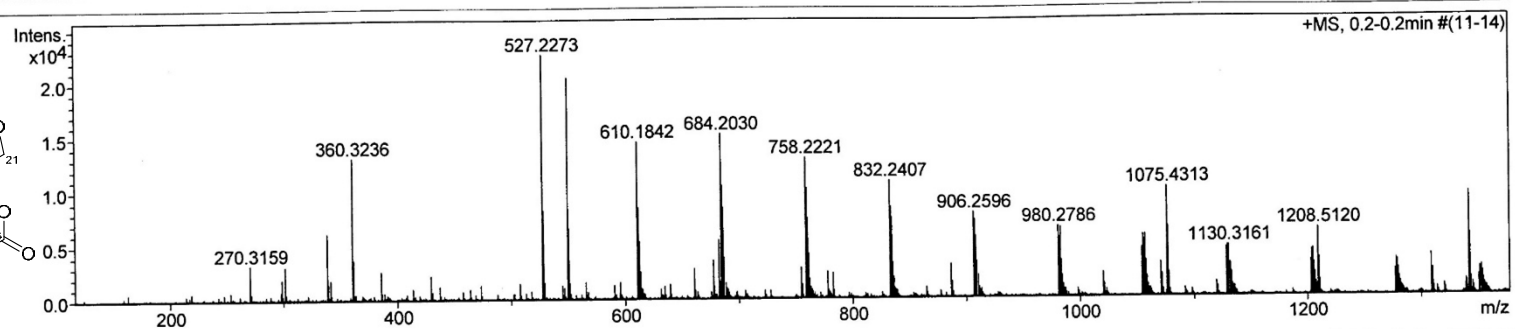
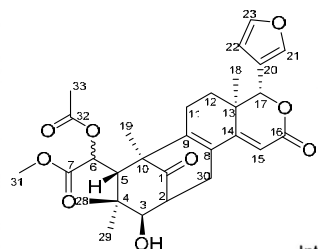
## Generic Display Report

### Analysis Info

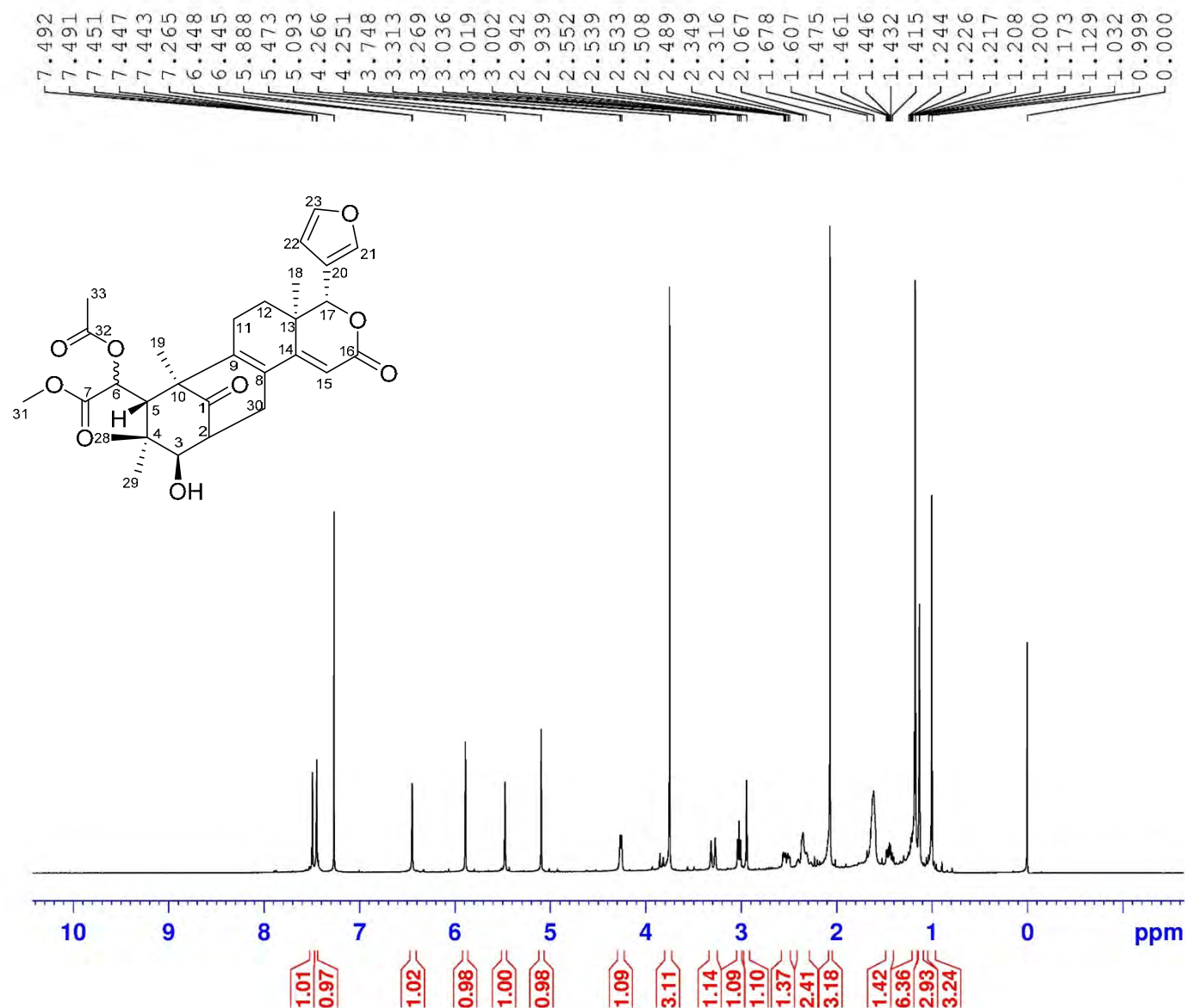
Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-86\_pos\_24\_01\_834.d  
Method LC\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name liwanshan\_lws-86\_pos  
Comment

Acquisition Date 11/18/2015 10:10:28 PM

Operator SCSIO  
Instrument maXis



# $^1\text{H}$ NMR (400 MHz) spectrum of compound **9** in $\text{CDCl}_3$



```

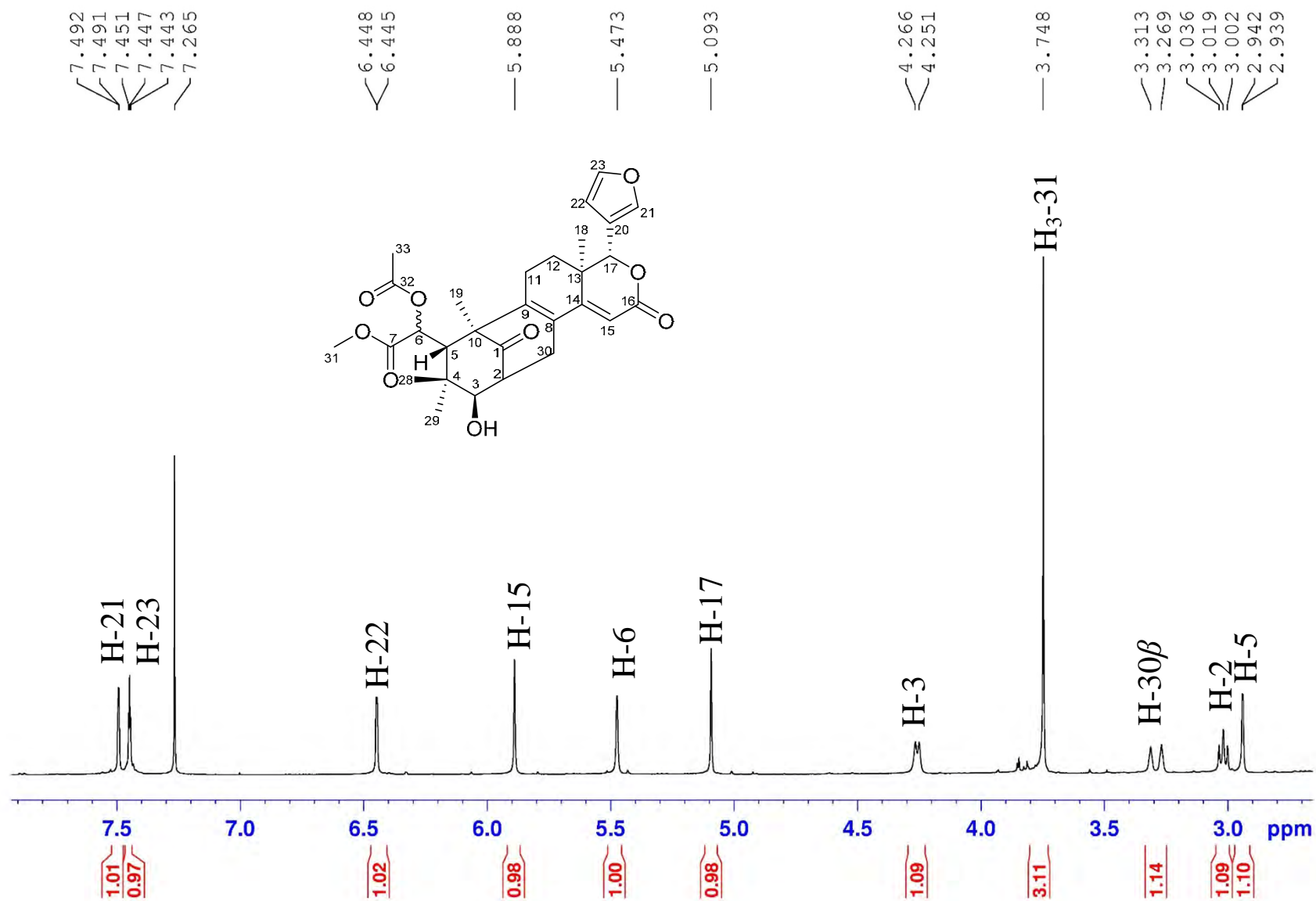
NAME          lws-86
EXPNO          1
PROCNO         1
Date_          20151105
Time           14.12
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        zg30
TD             65536
SOLVENT        CDCl3
NS             16
DS             2
SWH            8223.685 F
FIDRES         0.125483 F
AQ            3.9846387 s
RG            208.5
DW            60.800 u
DE            10.00 u
TE            297.0 K
D1            1.00000000 s
TD0            1
    
```

```

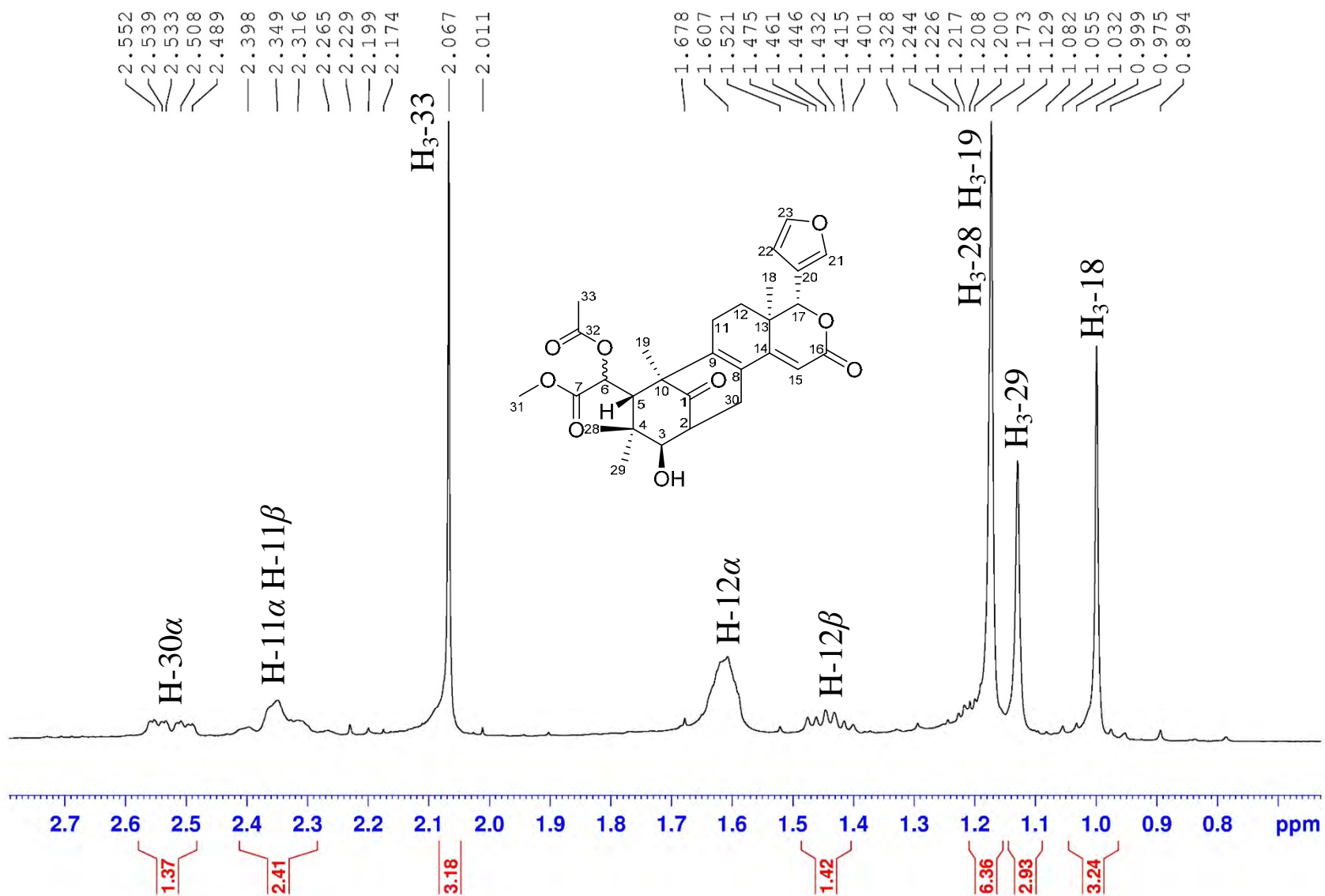
===== CHANNEL f1 =====
SFO1          400.1324710 M
NUC1           1H
P1            12.00 u
SI            65536
SF            400.1300075 M
WDW            EM
SSB            0
LB            0.30 F
GB            0
PC            1.00
    
```



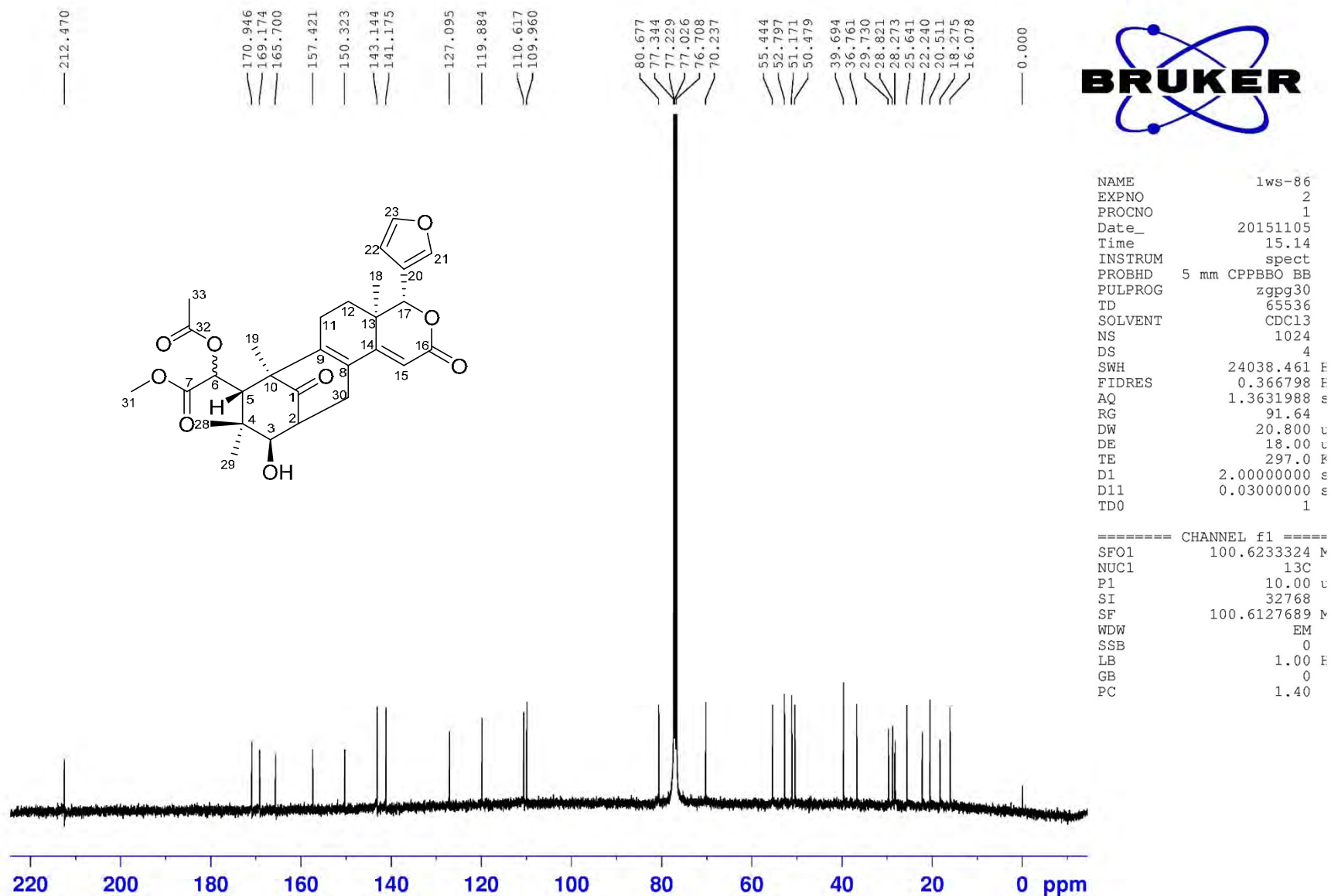
$^1\text{H}$  NMR (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



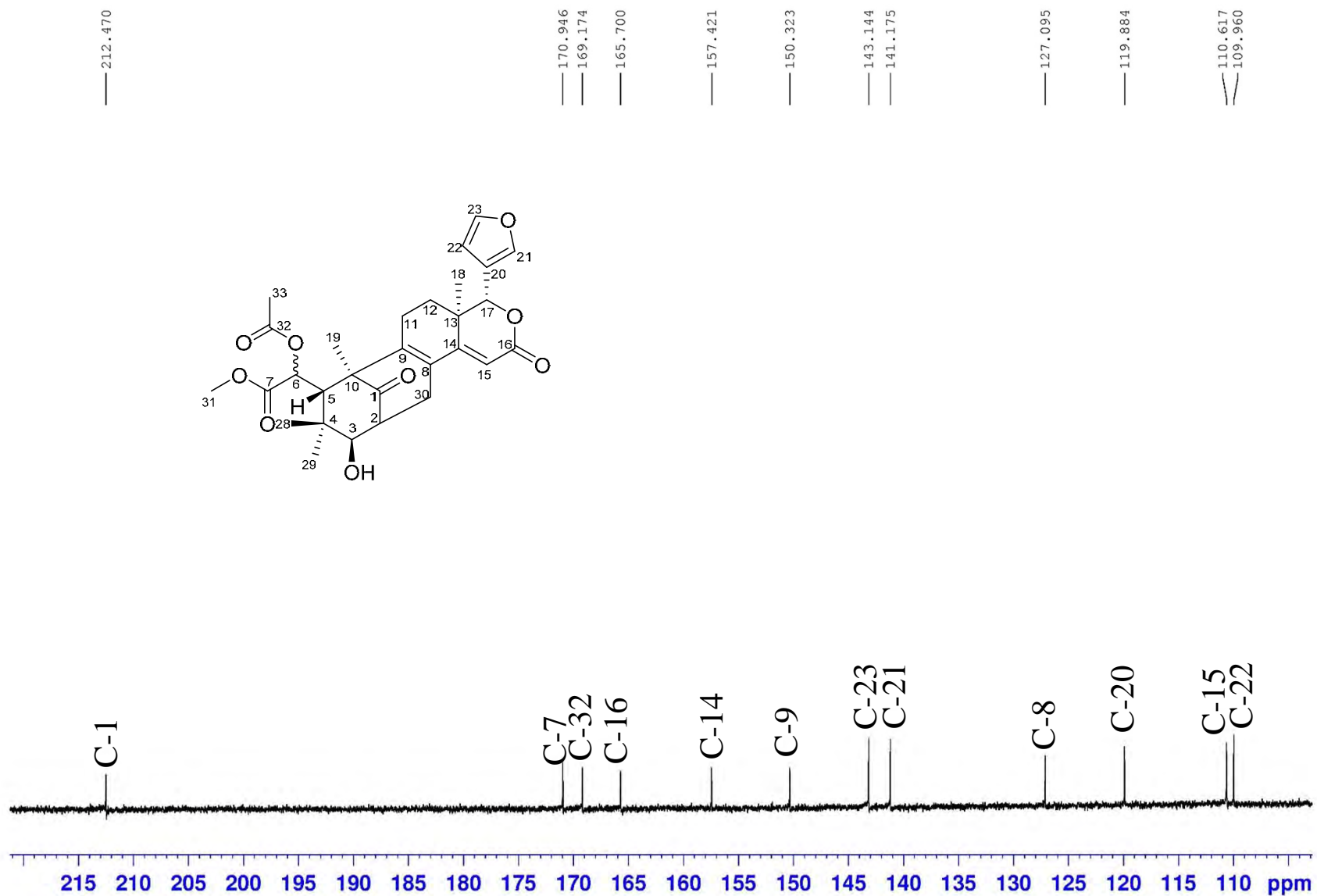
$^1\text{H}$  NMR (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



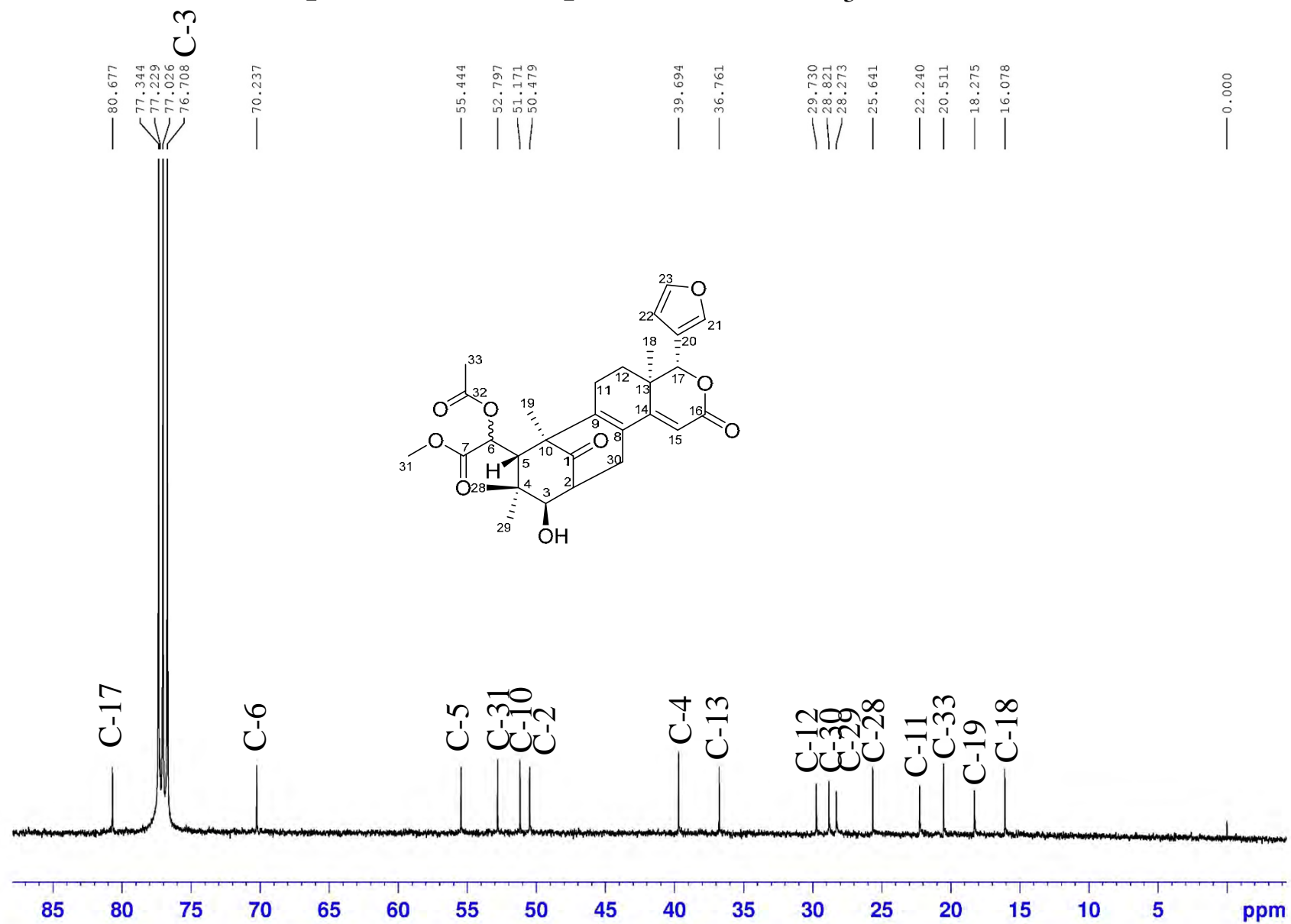
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



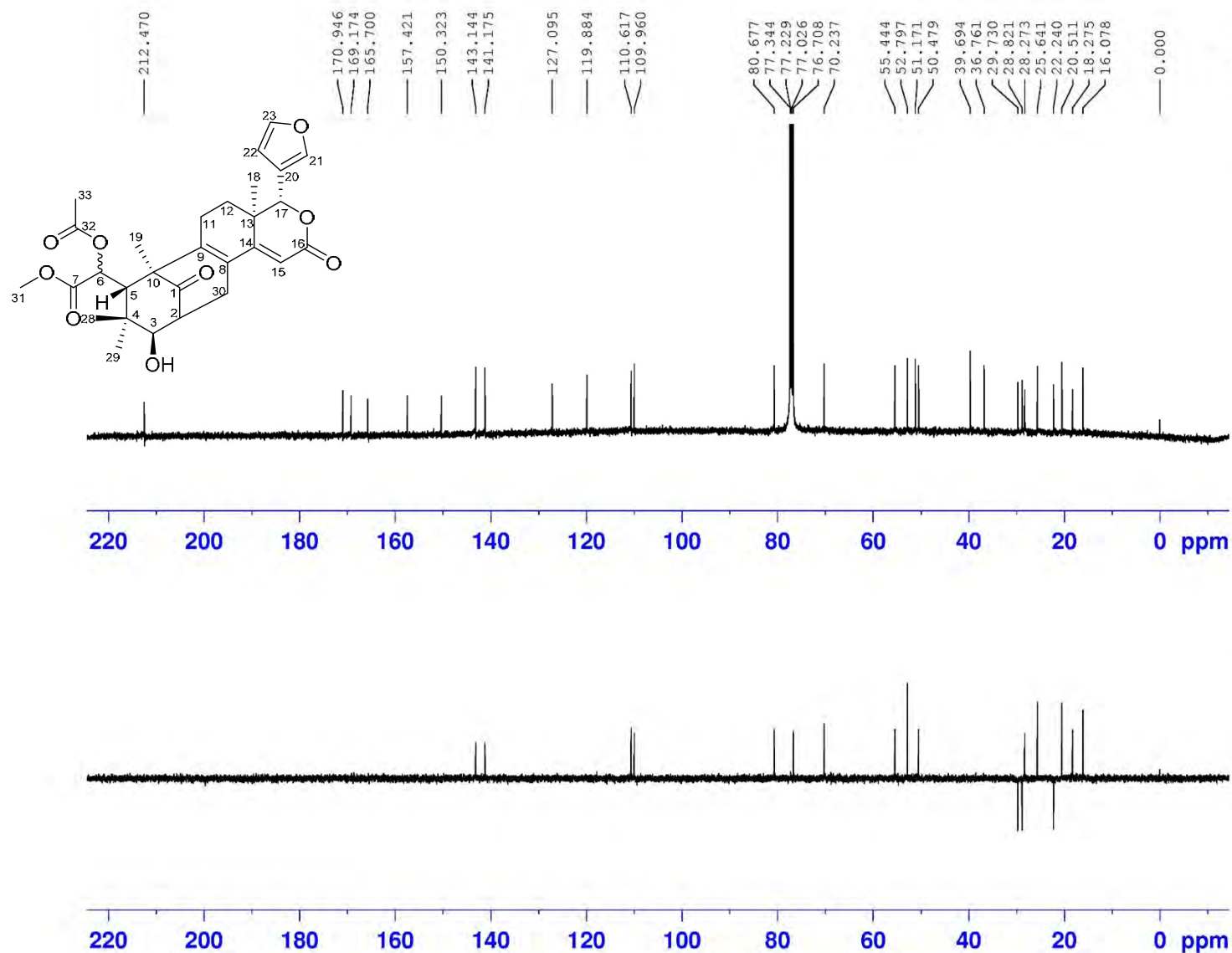
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



```

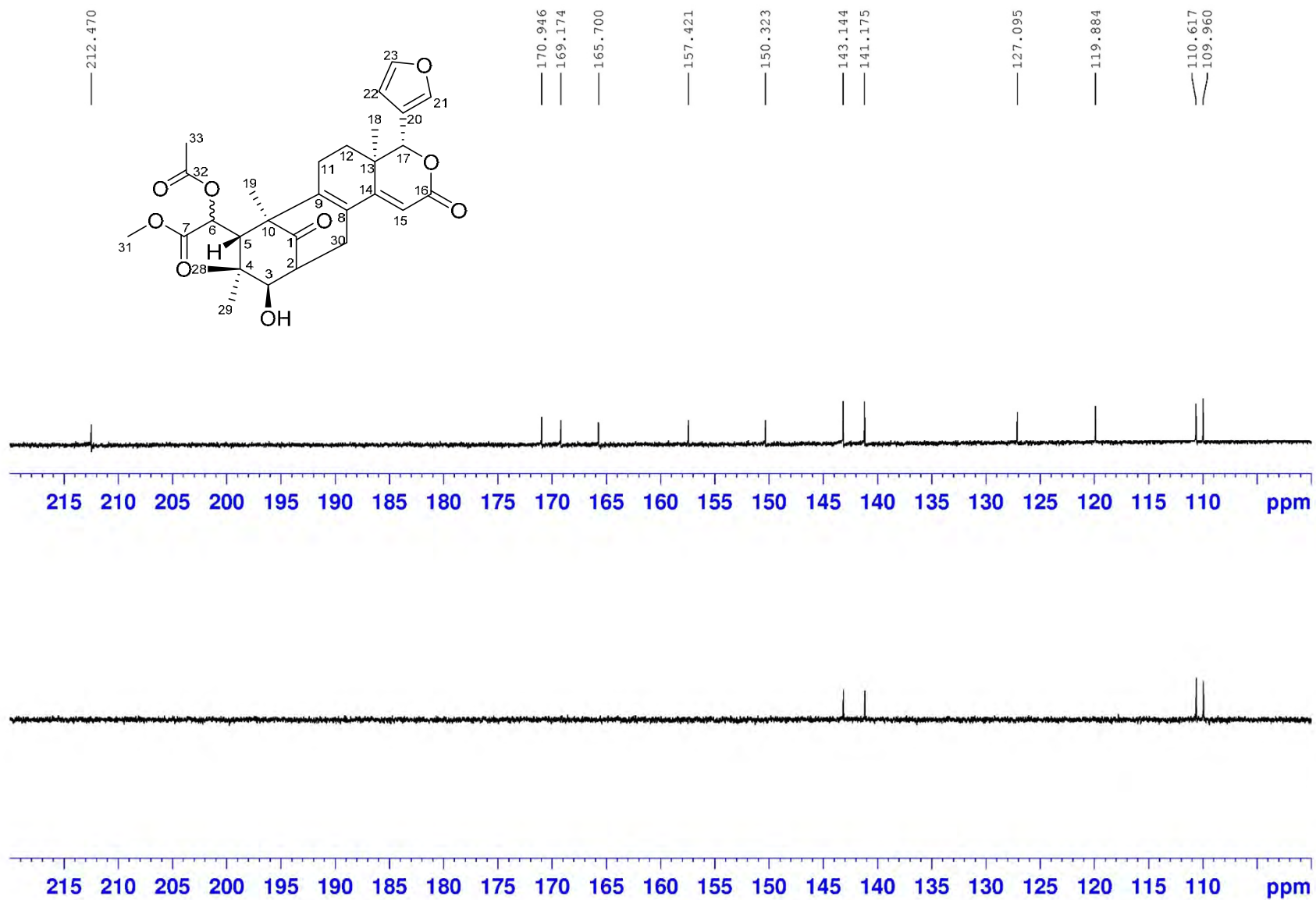
NAME                lws-86
EXPNO                3
PROCNO              1
Date_                20151105
Time                 15.32
INSTRUM              spect
PROBHD               5 mm CPBBO BB
PULPROG              deptspl35
TD                   65536
SOLVENT              CDCl3
NS                   300
DS                    4
SWH                  24038.461 F
FIDRES               0.366798 F
AQ                   1.3631988 s
RG                   130.26
DW                   20.800 u
DE                   18.00 u
TE                   297.0 F
CNST2                145.0000000
D1                   2.00000000 s
D2                   0.00344828 s
D12                  0.00002000 s
TD0                  1
  
```

```

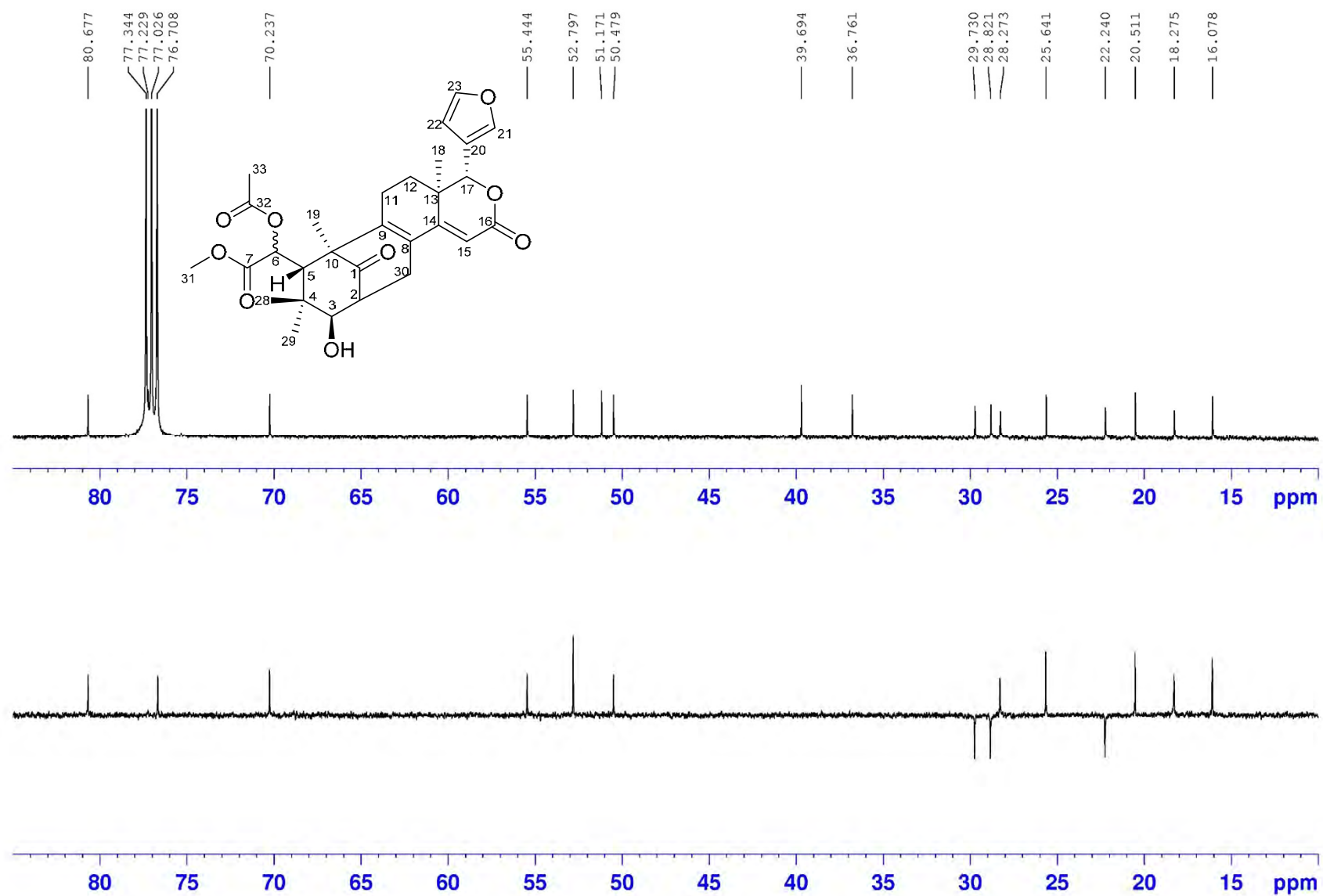
===== CHANNEL f1 =====
SFO1                100.6233324 M
NUC1                 13C
P1                   10.00 u
P13                  2000.00 u
SI                   32768
SF                   100.6127689 M
WDW                  EM
SSB                   0
LB                   1.00 F
GB                   0
PC                   1.40
  
```



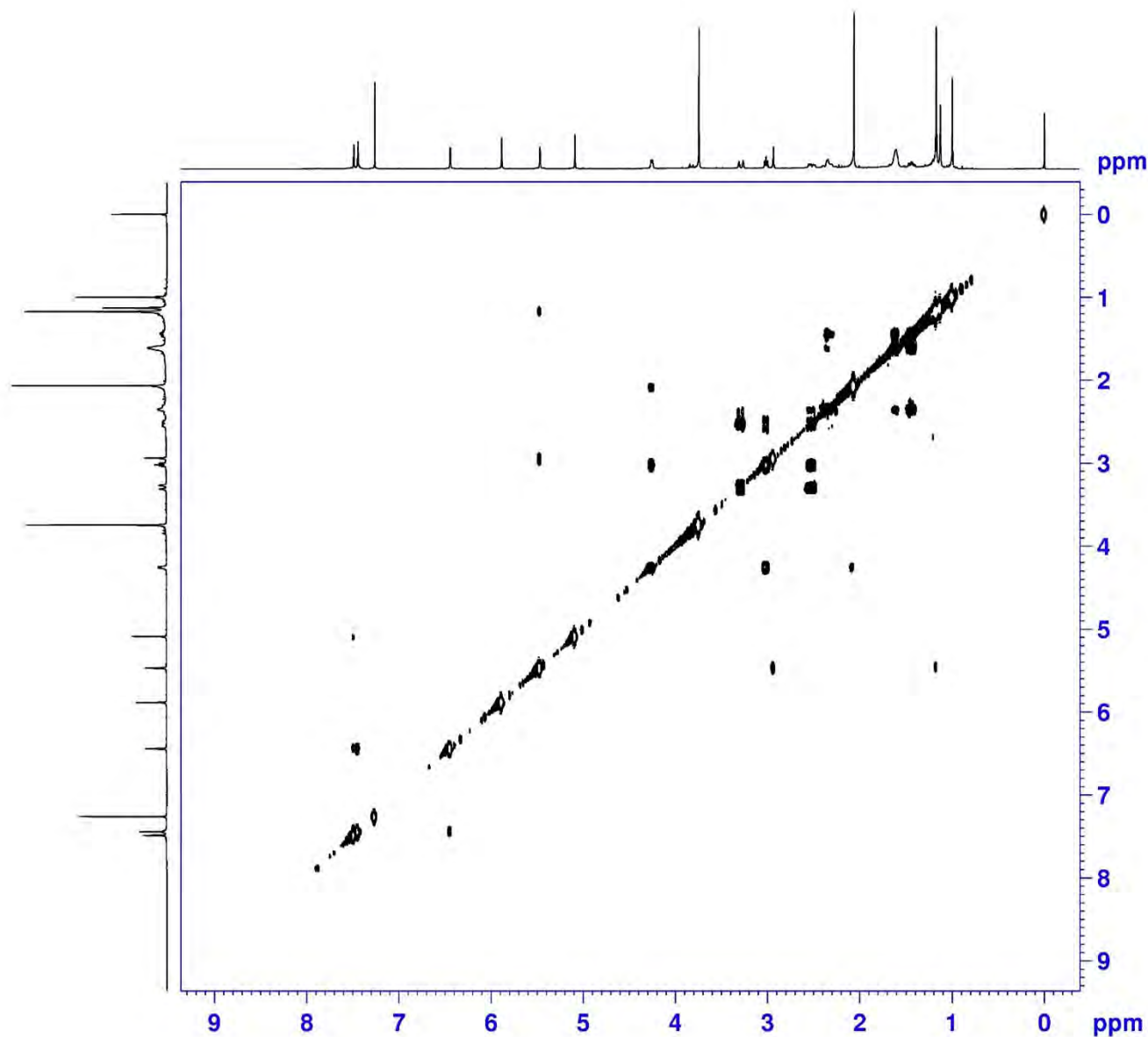
# DEPT135 (100 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



# DEPT135 (100 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



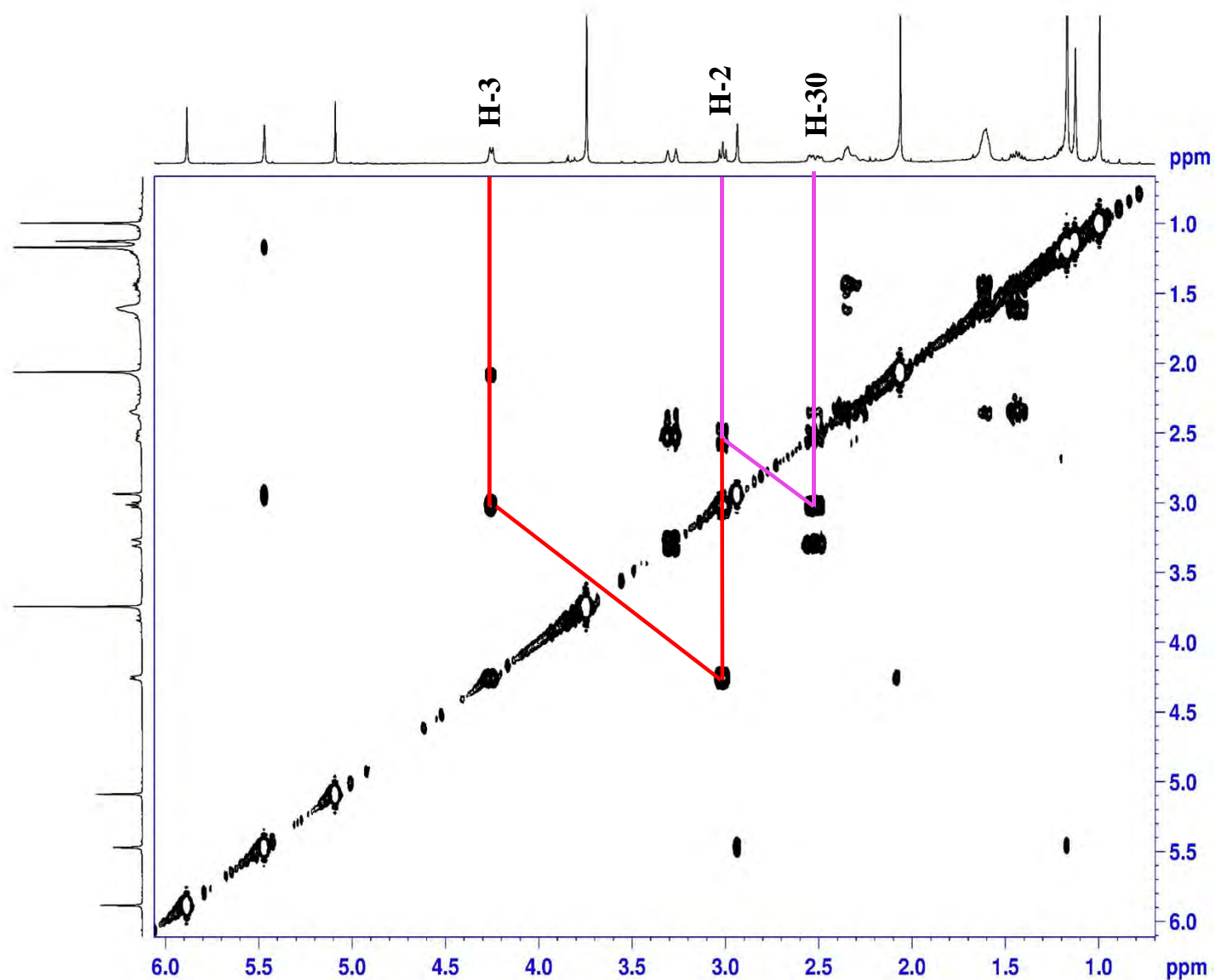
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



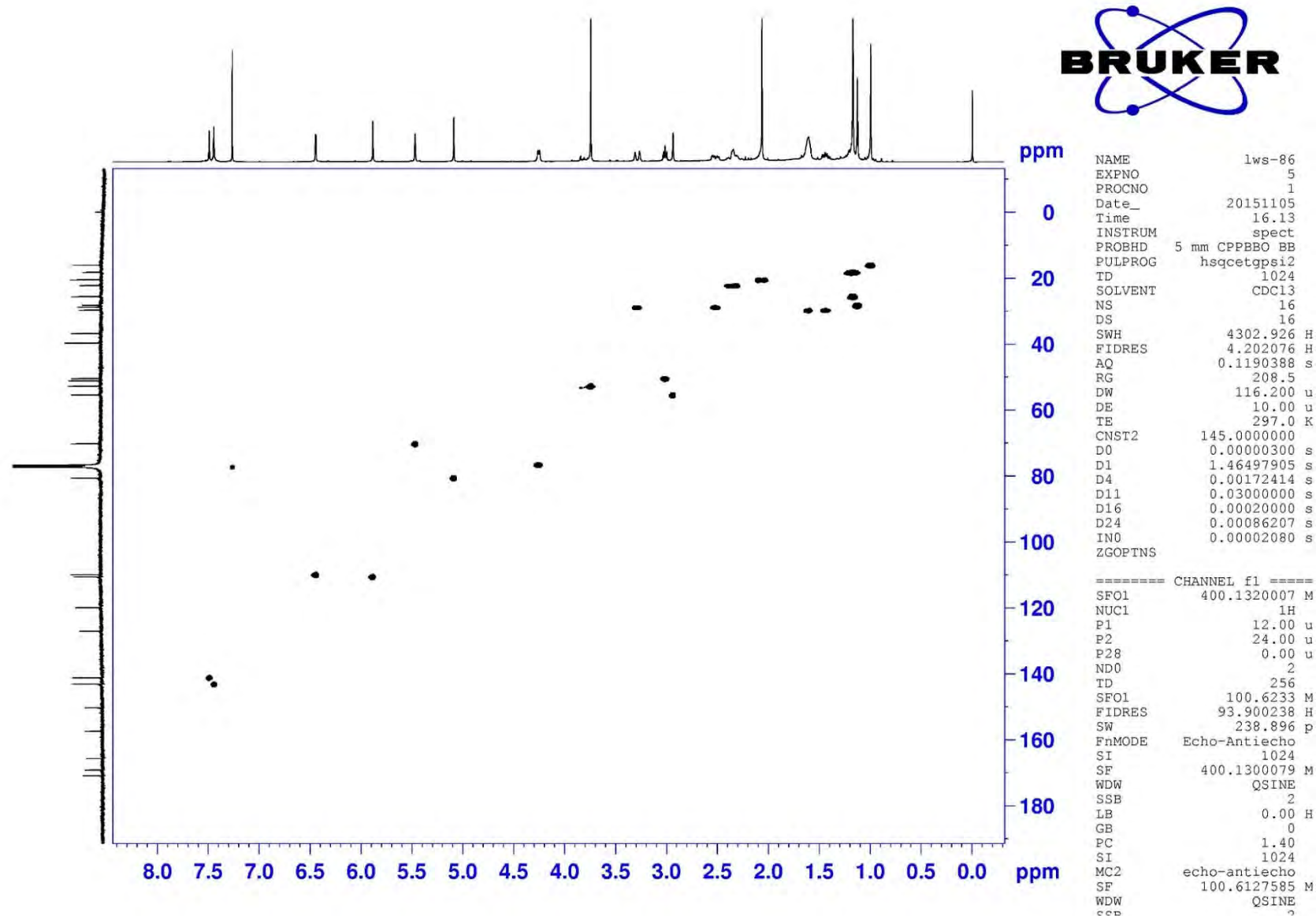
```
NAME          lws-86
EXPNO          4
PROCNO         1
Date_         20151105
Time          15.33
INSTRUM        spect
PROBHD         5 mm CFPBBO BB
PULPROG        cosygpppqf
TD             2048
SOLVENT        CDCl3
NS              8
DS              8
SWH            3906.250 Hz
FIDRES         1.907349 Hz
AQ             0.2621940 sec
RG             208.5
DW             128.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00000300 sec
D1             1.89678097 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00025600 sec
```

```
===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1           1H
P0             12.00 usec
P1             12.00 usec
P17            2500.00 usec
ND0            1
TD             128
SFO1          400.1318 MHz
FIDRES         30.517578 Hz
SW             9.762 ppm
FnMODE         QF
SI             1024
SF            400.1300070 MHz
WDW            QSINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40
SI             1024
MC2            QF
SF            400.1300070 MHz
```

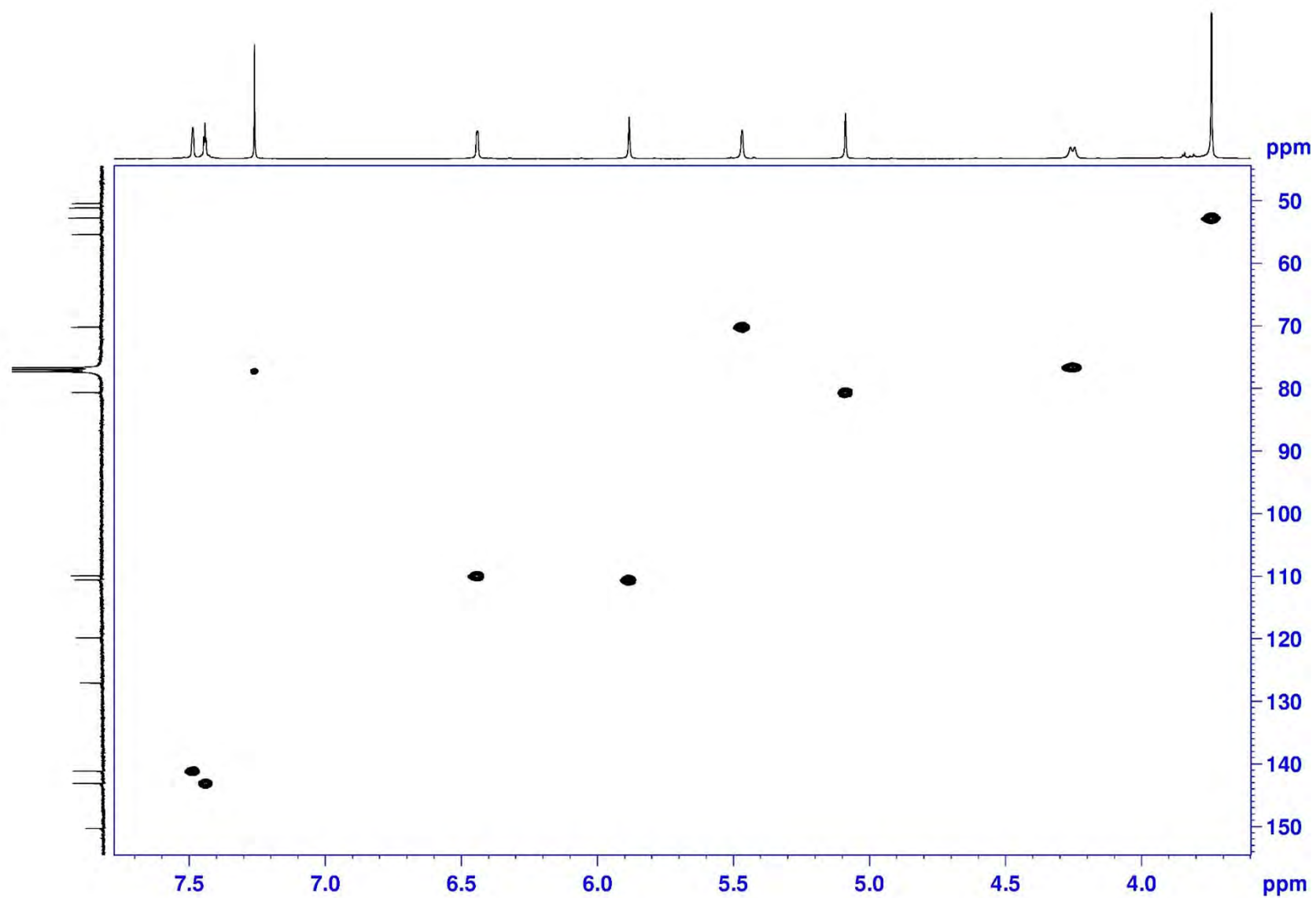
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



# HSQC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>

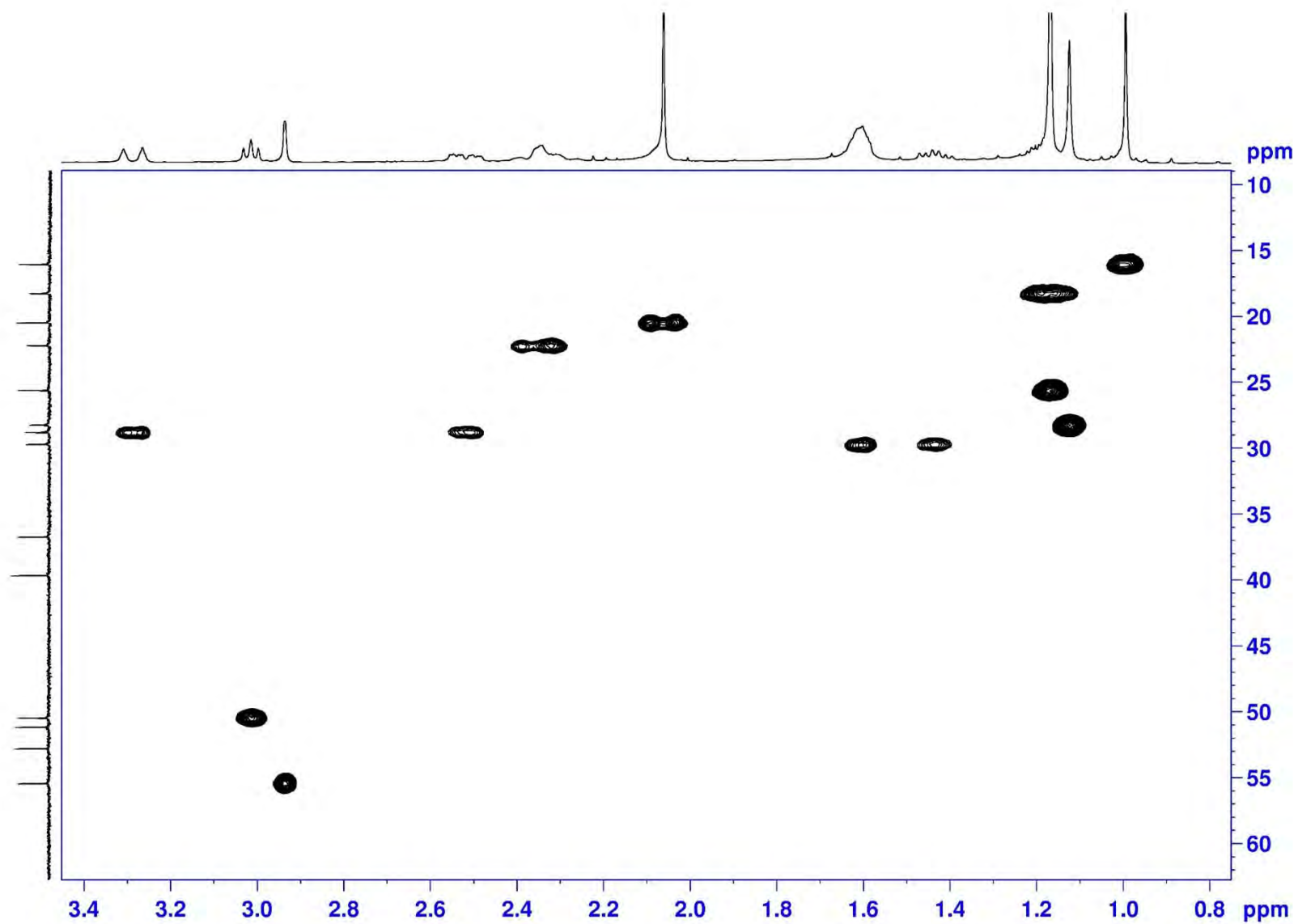


HSQC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



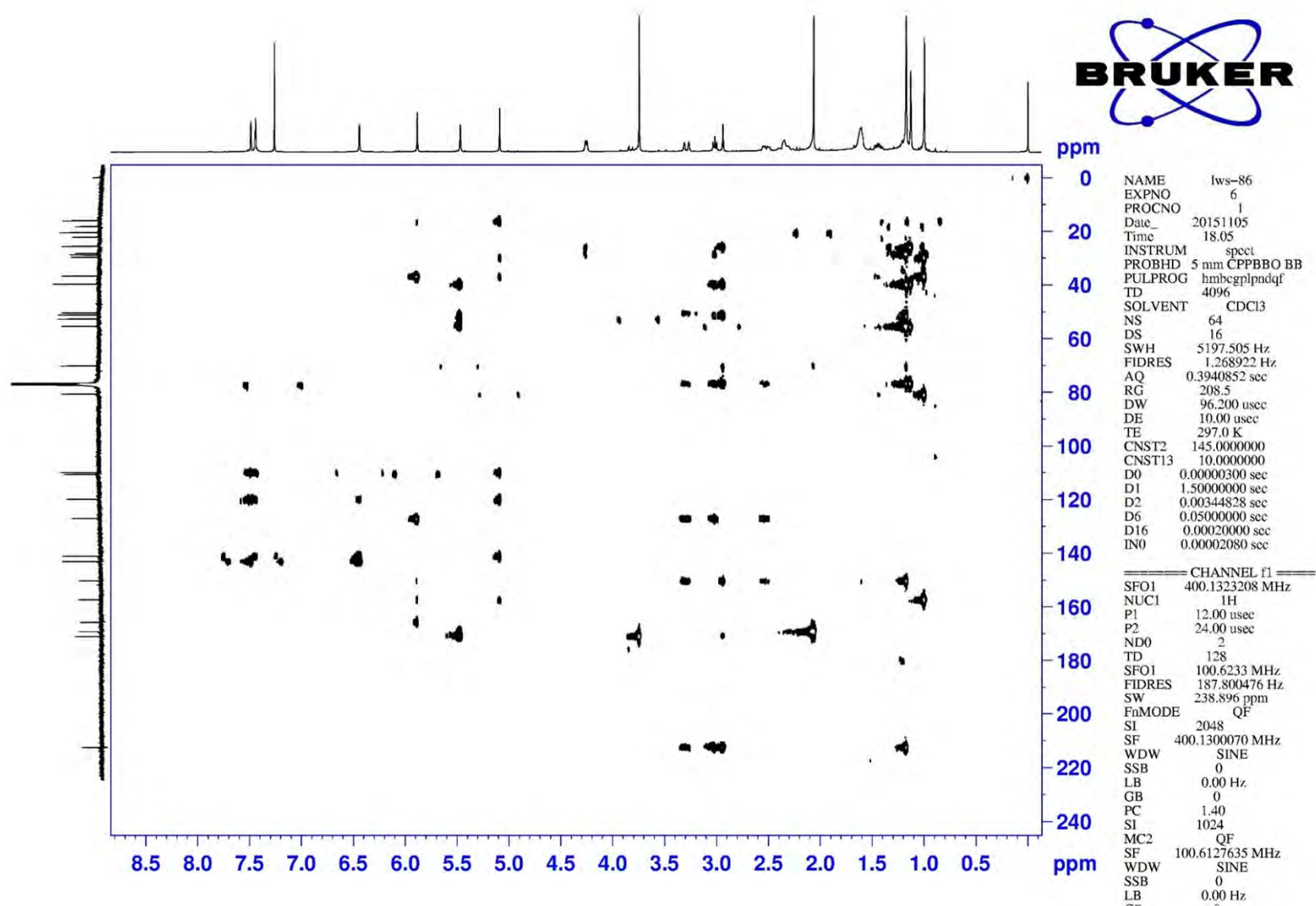


HSQC (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$

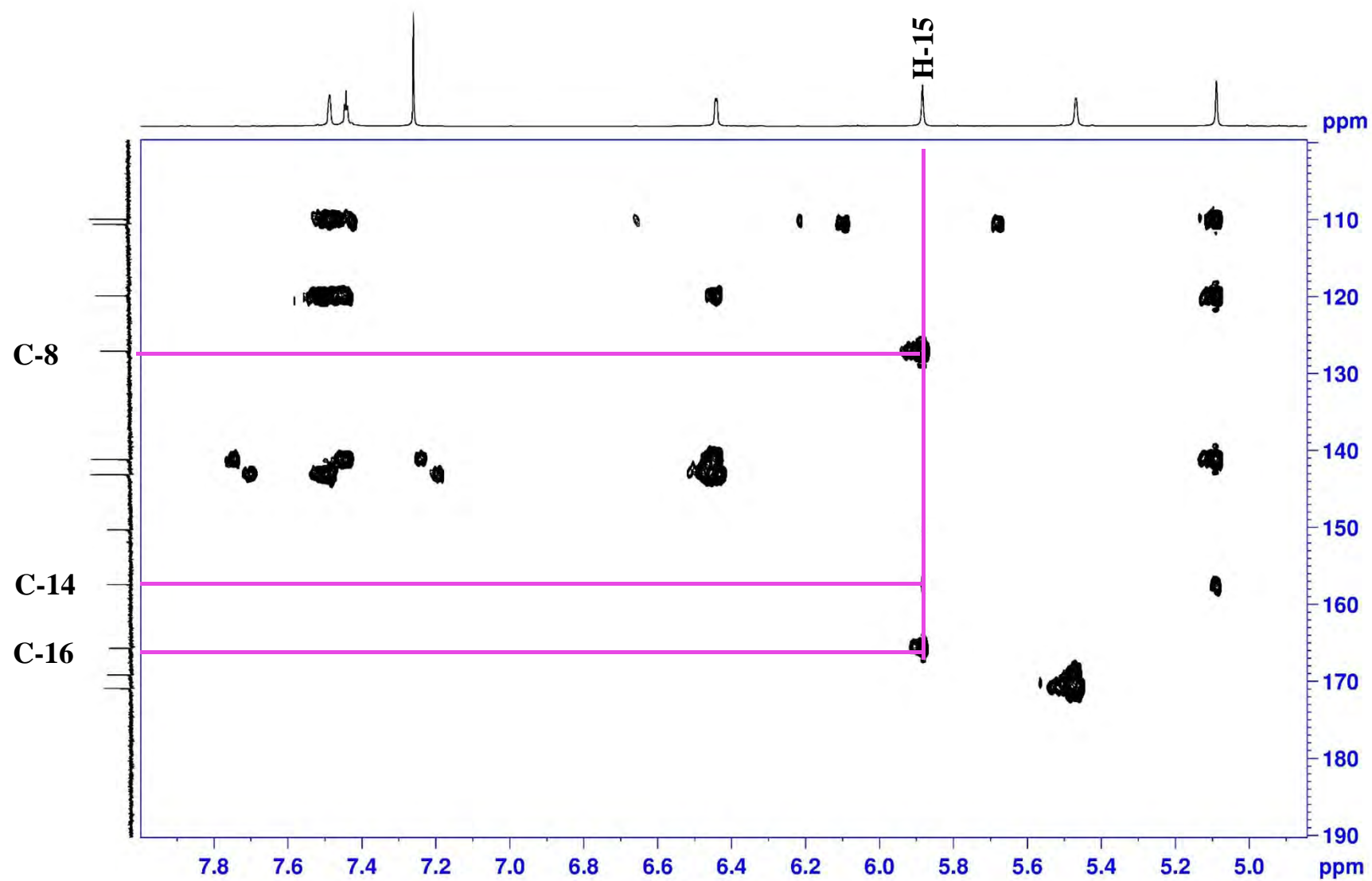




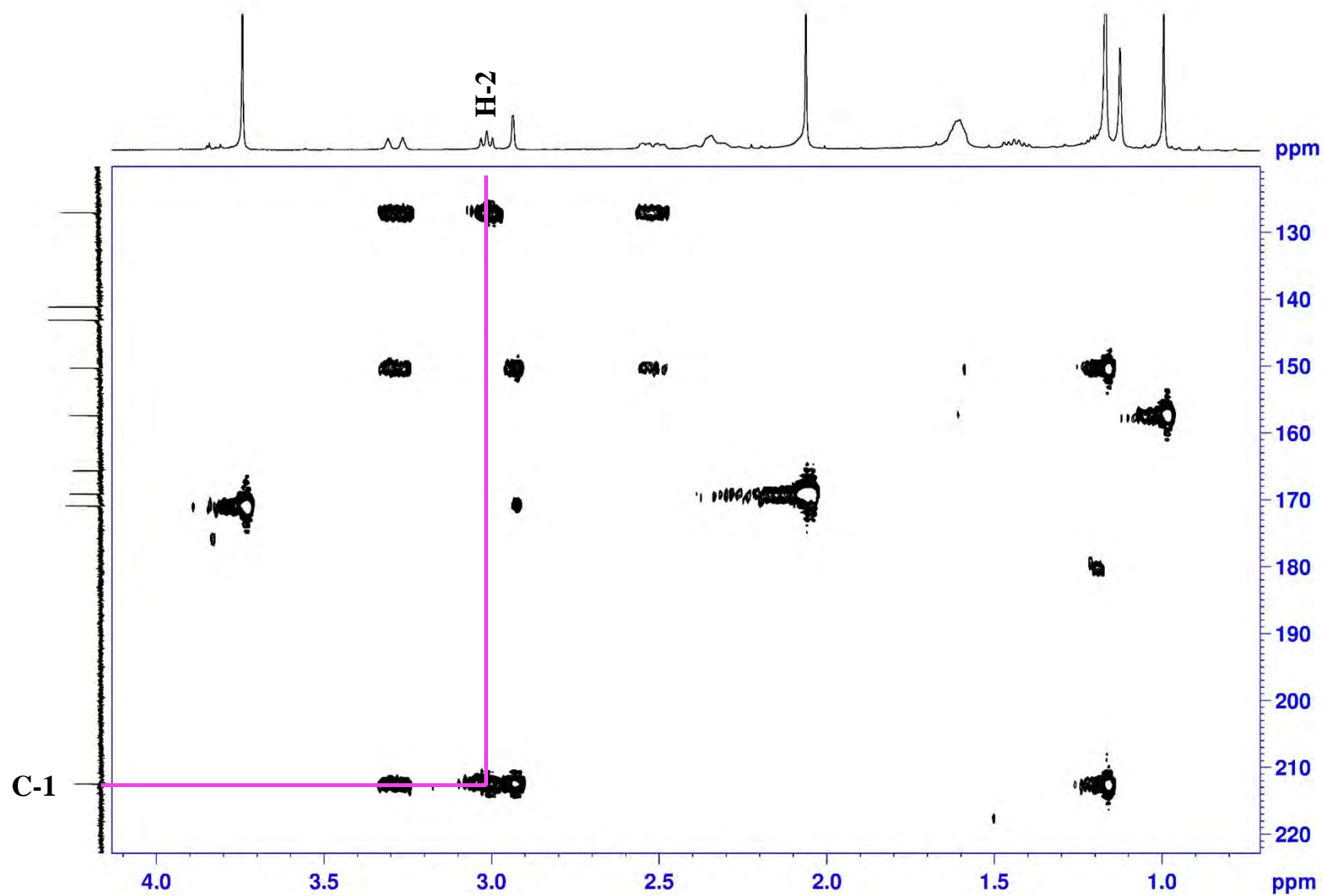
# HMBC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



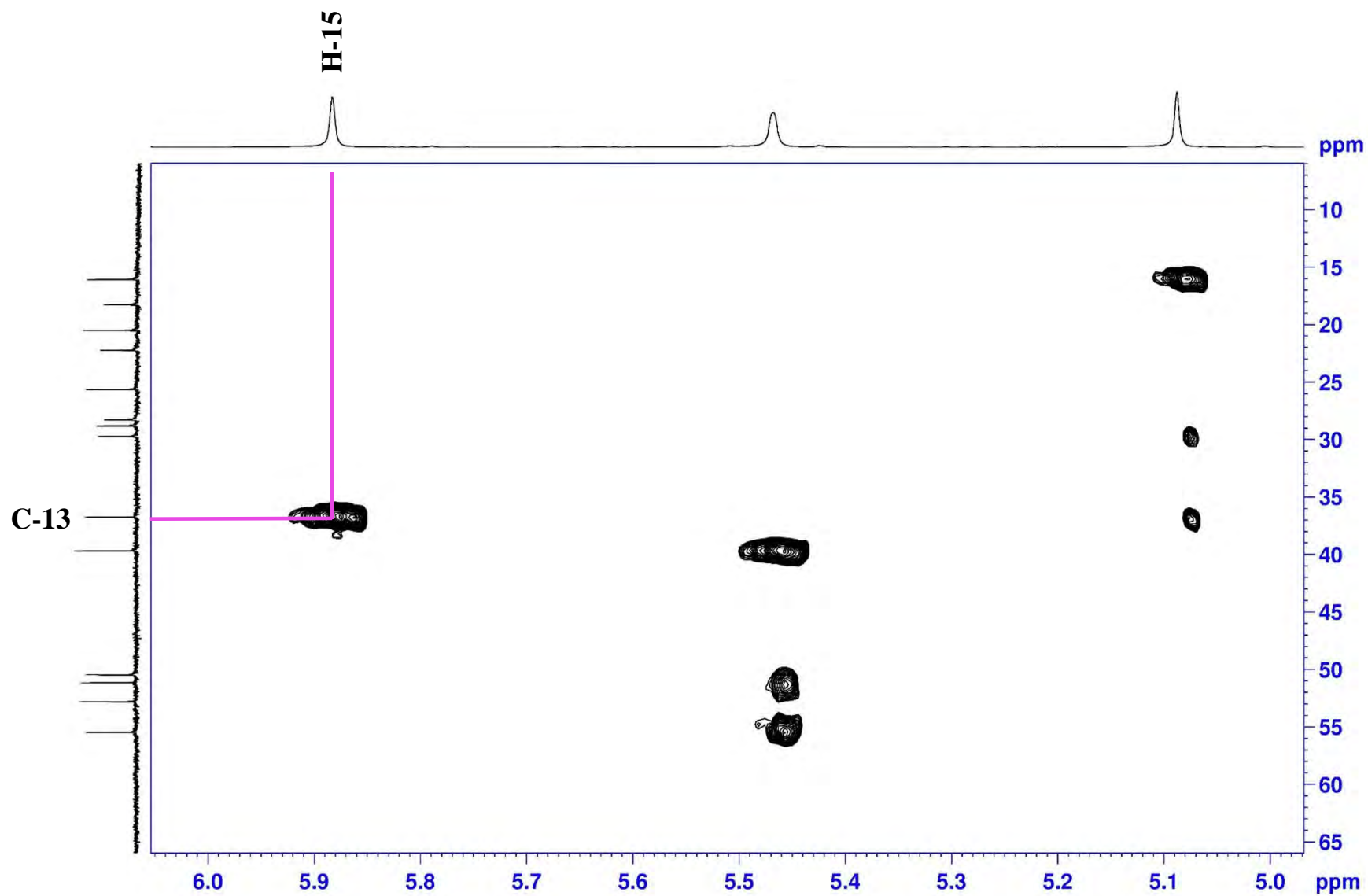
HMBC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



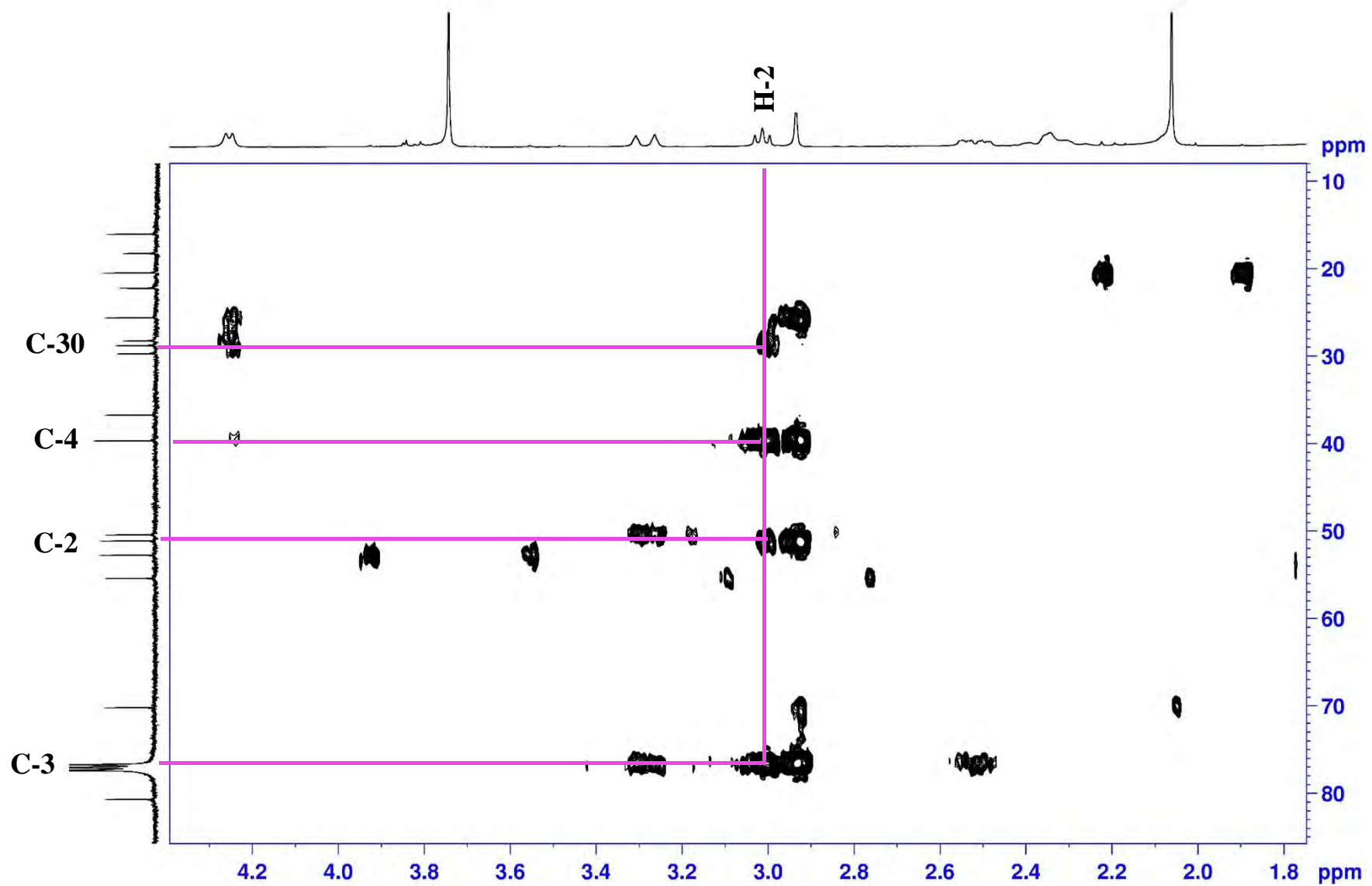
HMBC (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



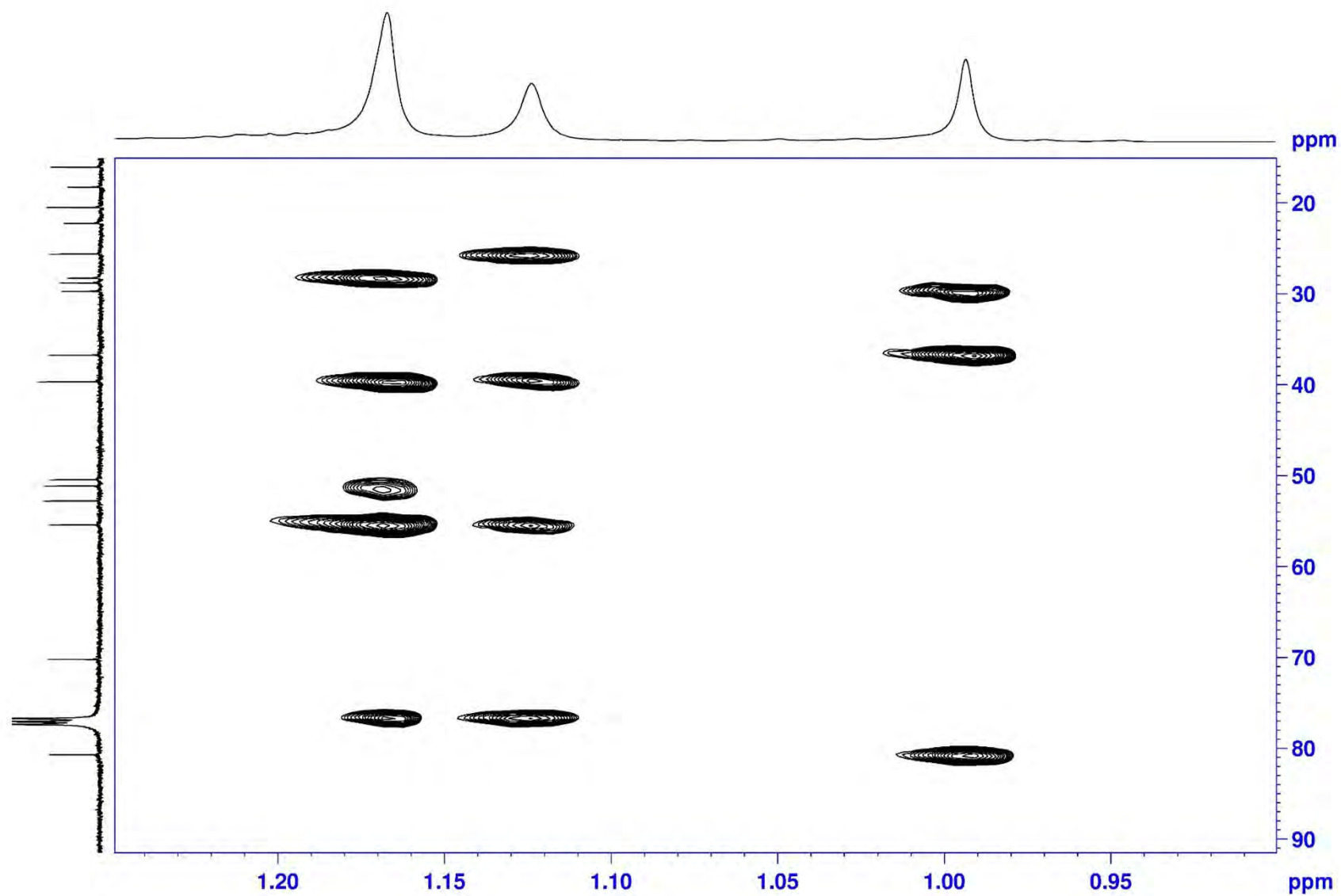
HMBC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>

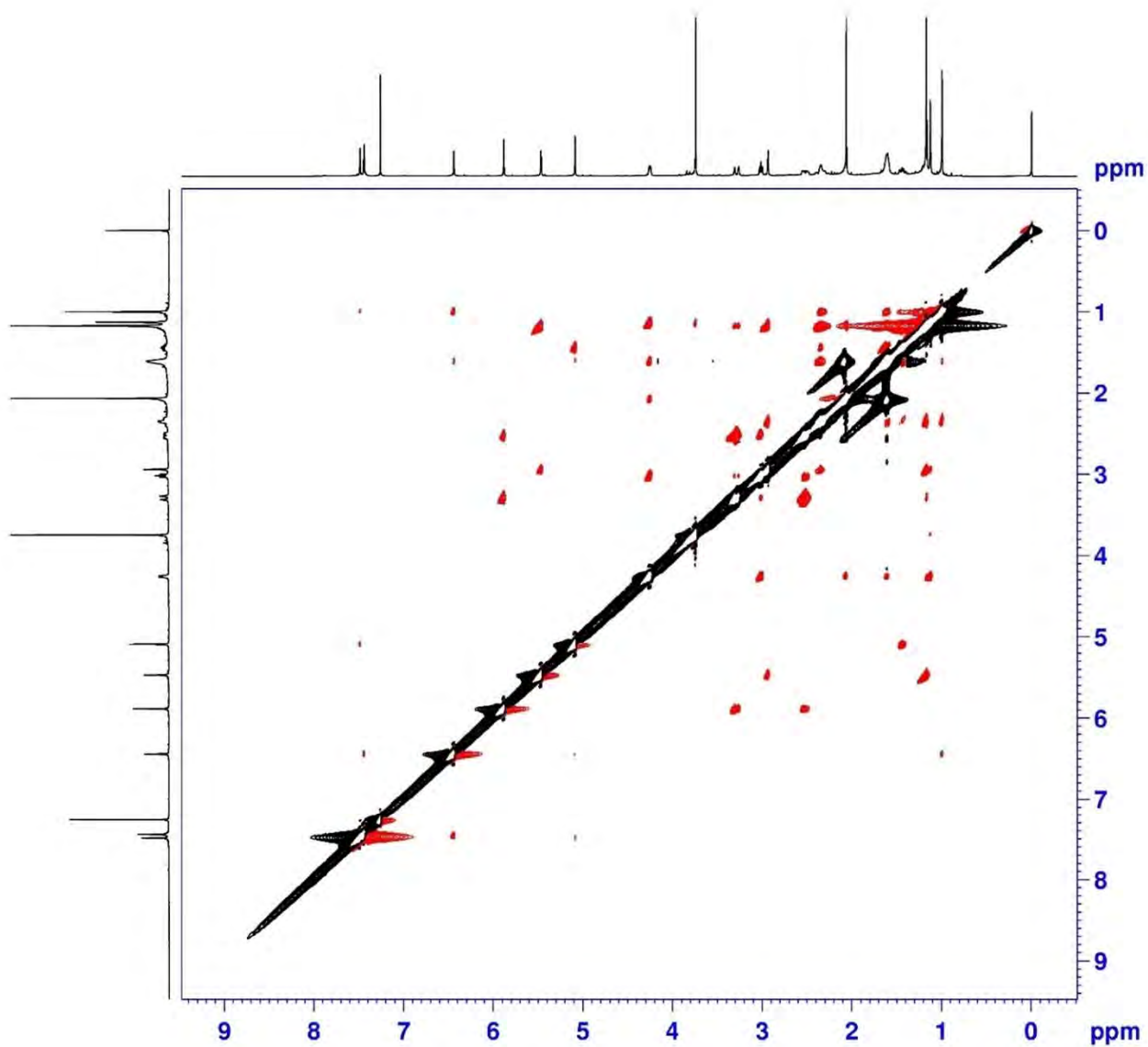


HMBC (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$





# NOESY (400 MHz) spectrum of compound **9** in CDCl<sub>3</sub>



```

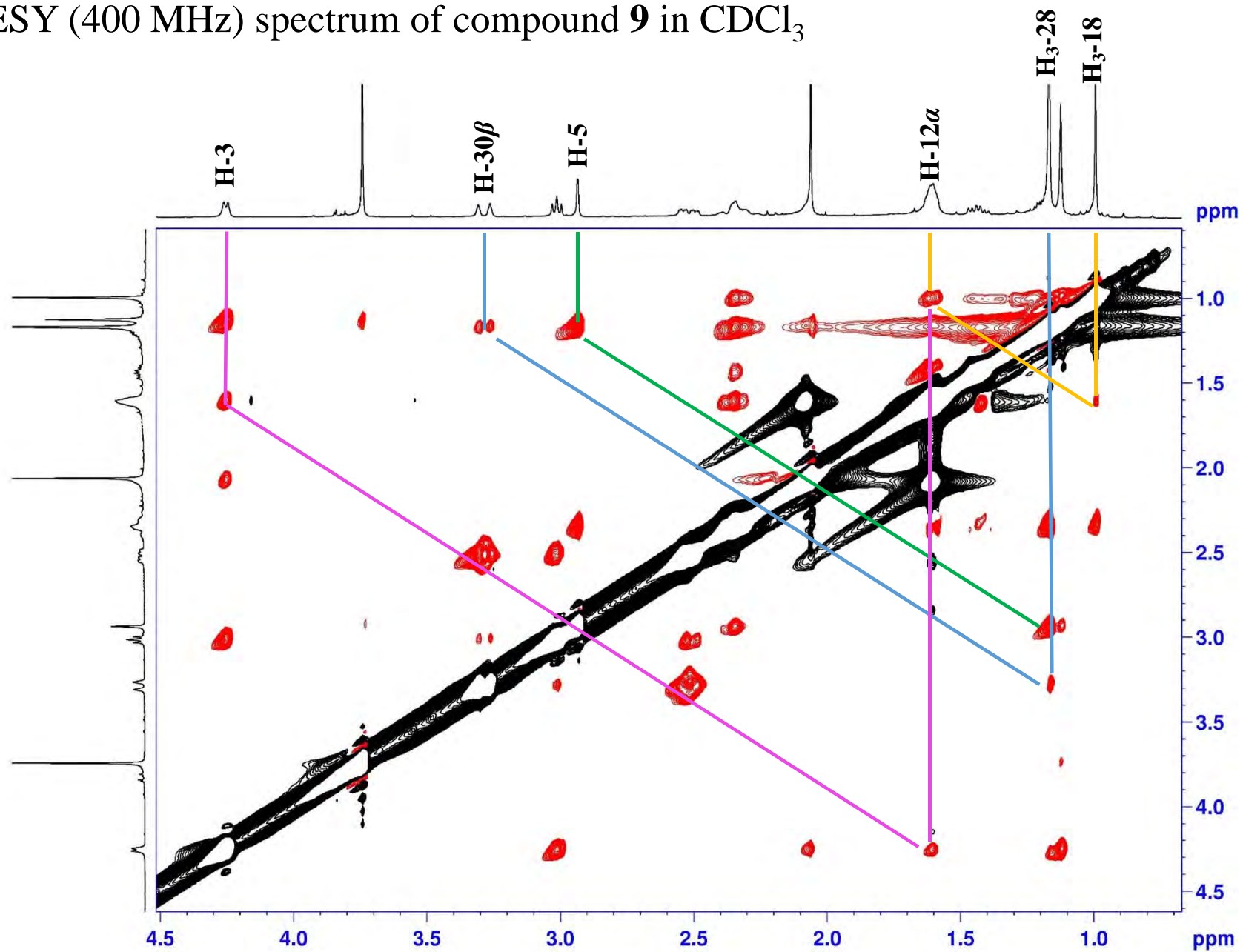
NAME          lws-86
EXPNO          7
PROCNO         1
Date_         20151105
Time_         22.33
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        noesyggpphpp
TD             2048
SOLVENT        CDCl3
NS             16
DS             32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ             0.2560500 sec
RG             208.5
DW             125.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00010972 sec
D1             1.99385595 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00025000 sec
  
```

```

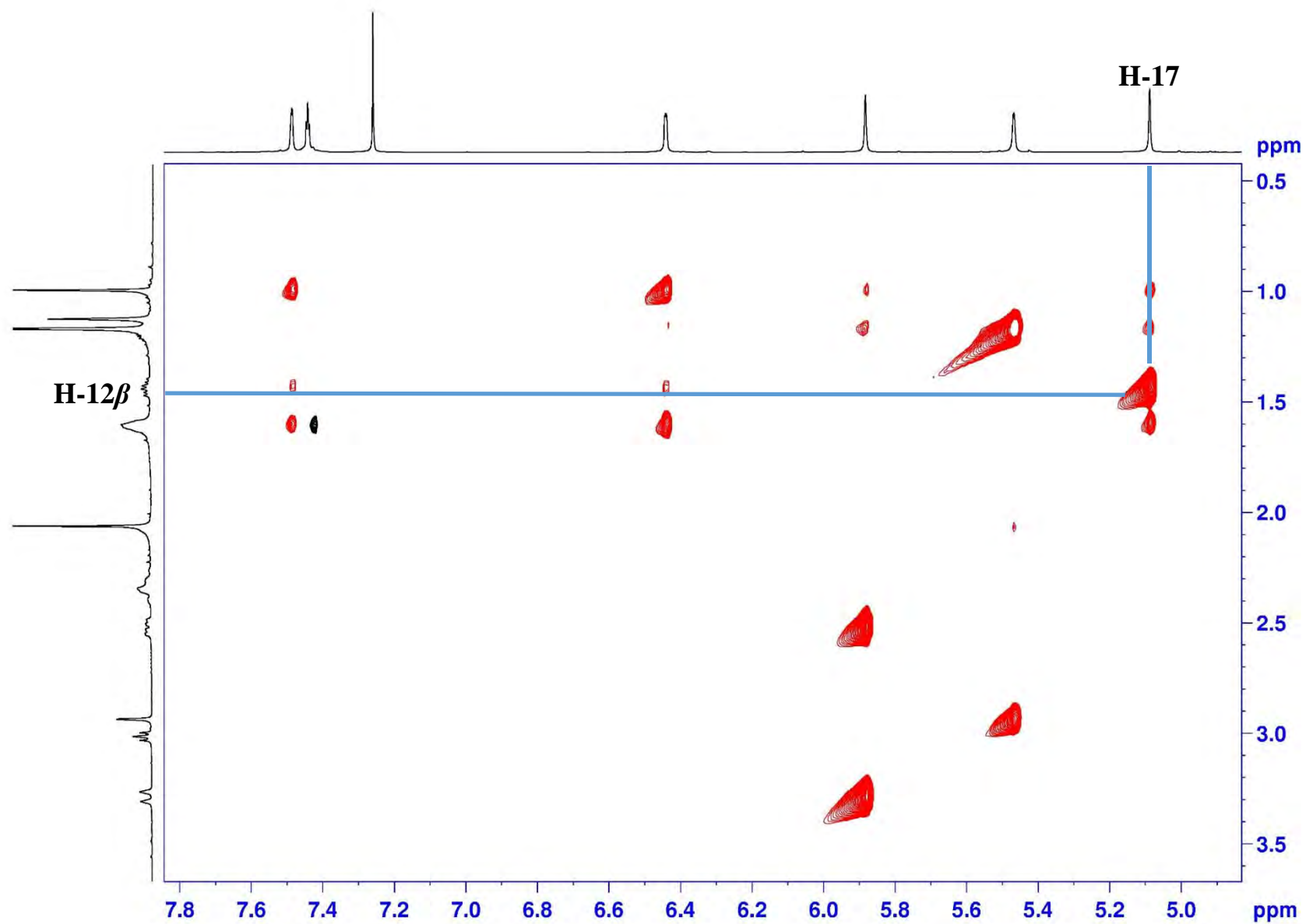
===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1           1H
P1             12.00 usec
P2             24.00 usec
P17            2500.00 usec
ND0            1
TD             256
SFO1          400.1318 MHz
FIDRES         15.625000 Hz
SW             9.997 ppm
FnMODE         States-TPPI
SI             1024
SF             400.1300098 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF             400.1300098 MHz
WDW            QSINE
  
```



NOESY (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **9** in  $\text{CDCl}_3$



# HRESIMS for compound **10**

## Elemental Composition Report

### Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

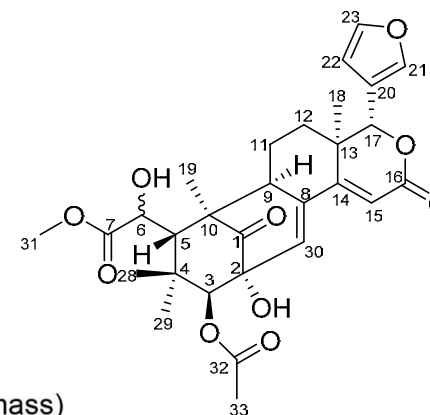
151 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-200 O: 0-100

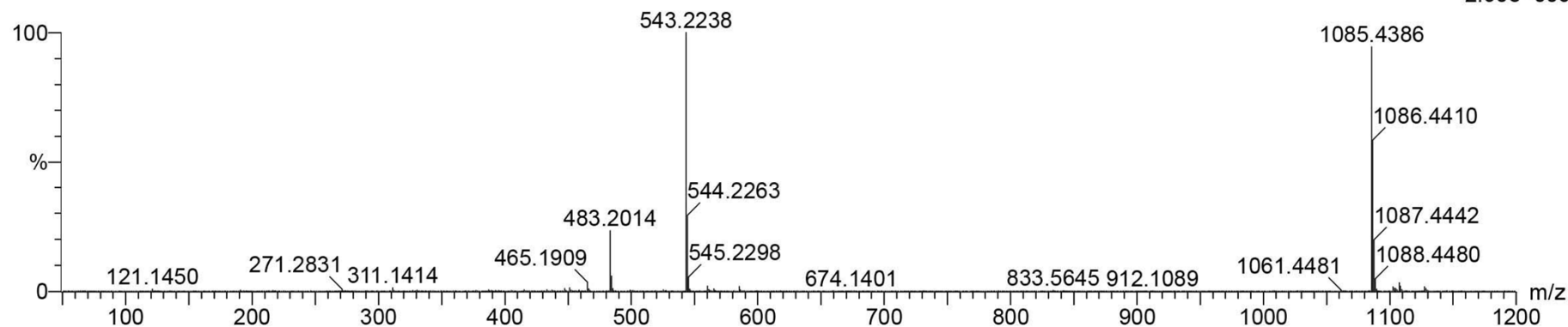
2j2

20161212-27 144 (1.167) Cm (143:146)



Page 1

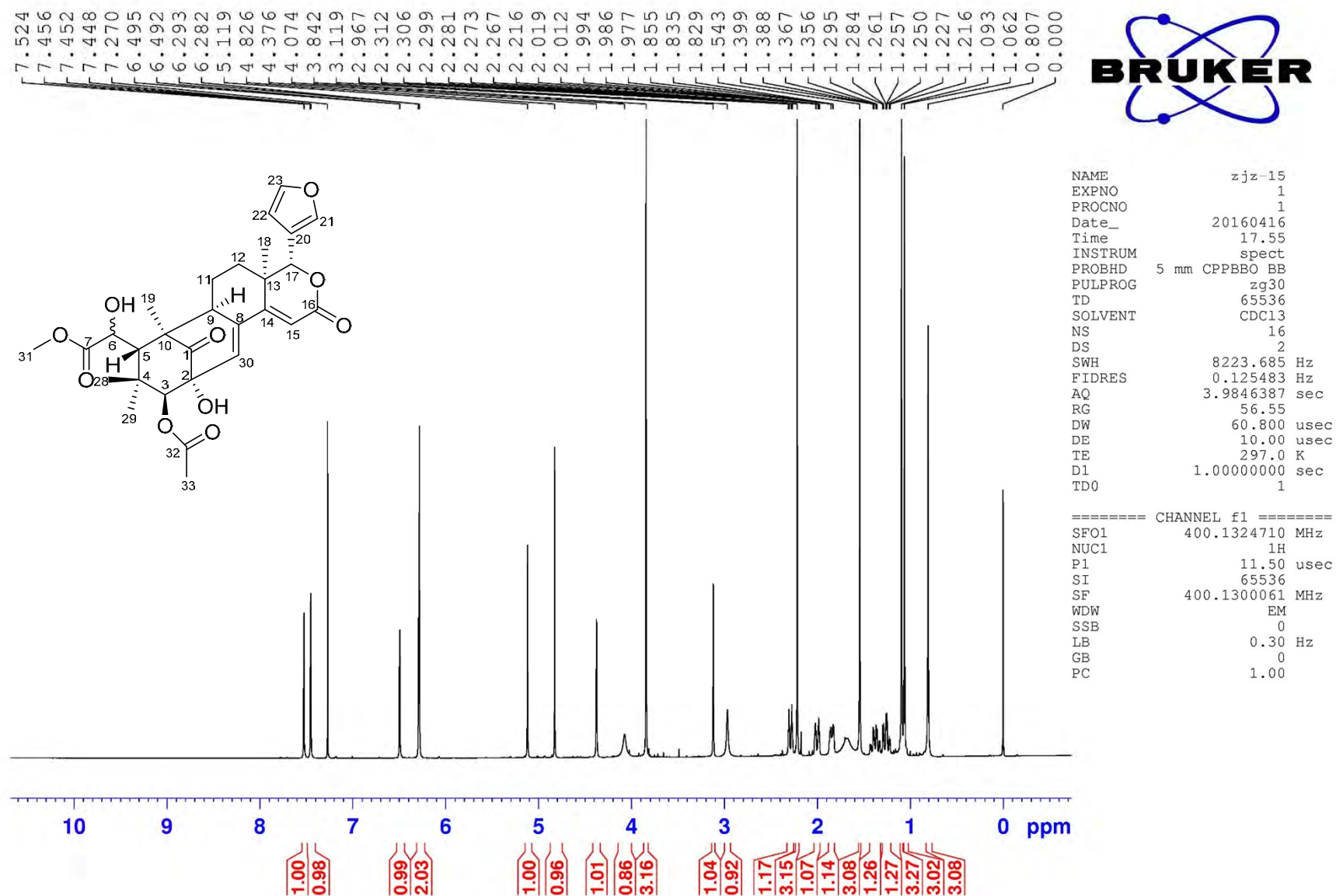
1: TOF MS ES+  
2.90e+006



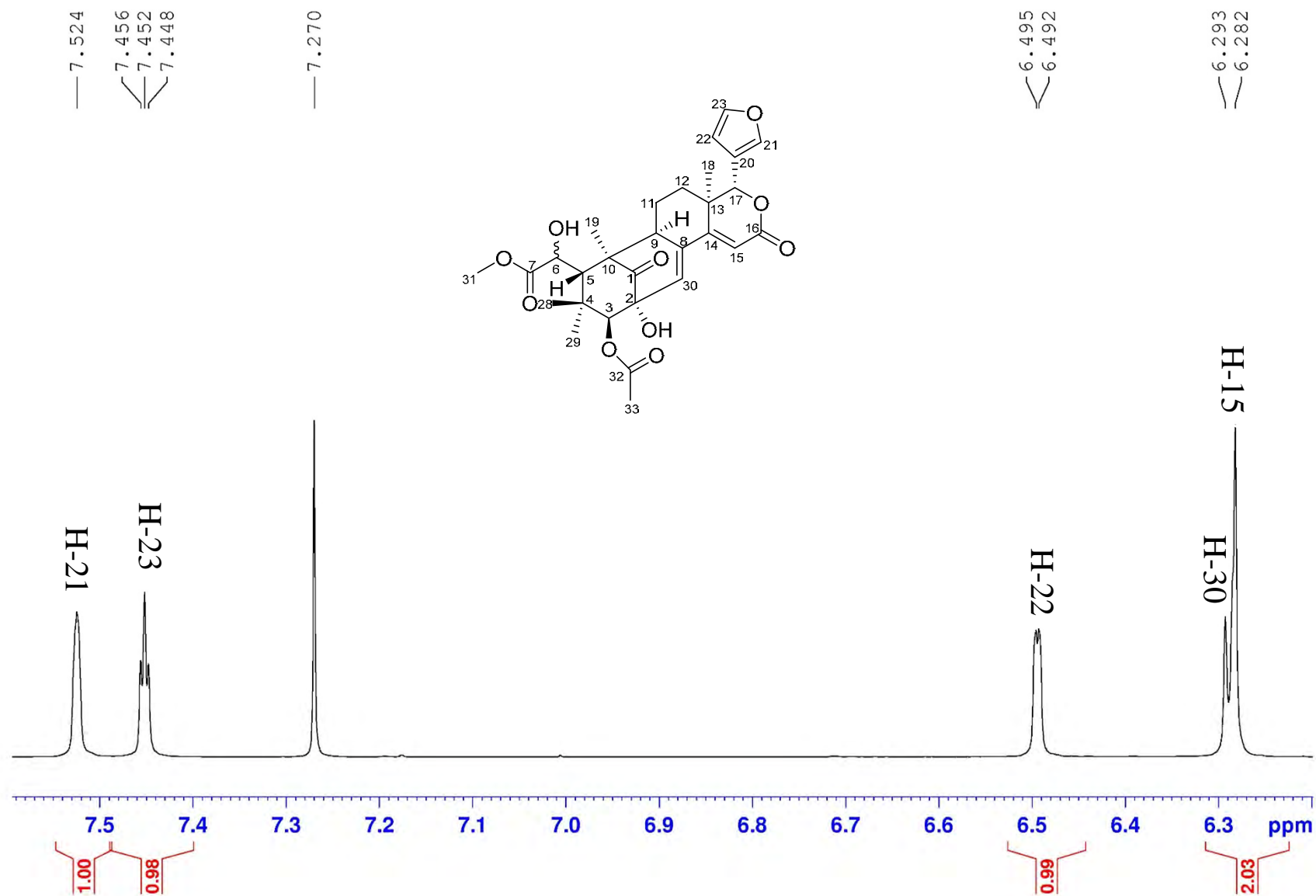
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
543.2238	543.2230	0.8	1.5	12.5	34.7	n/a	n/a	C <sub>29</sub> H <sub>35</sub> O <sub>10</sub>

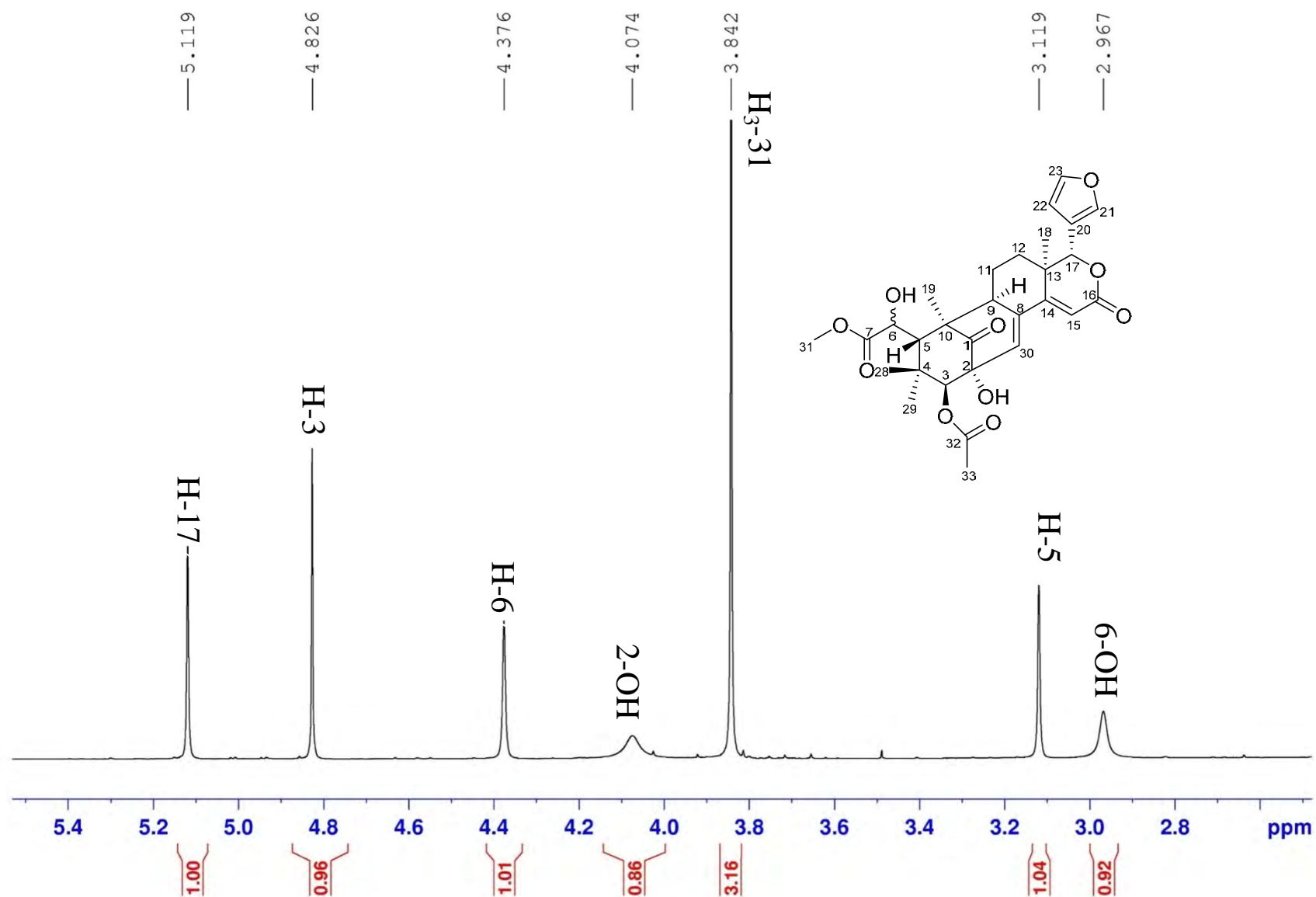
$^1\text{H}$  NMR (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$

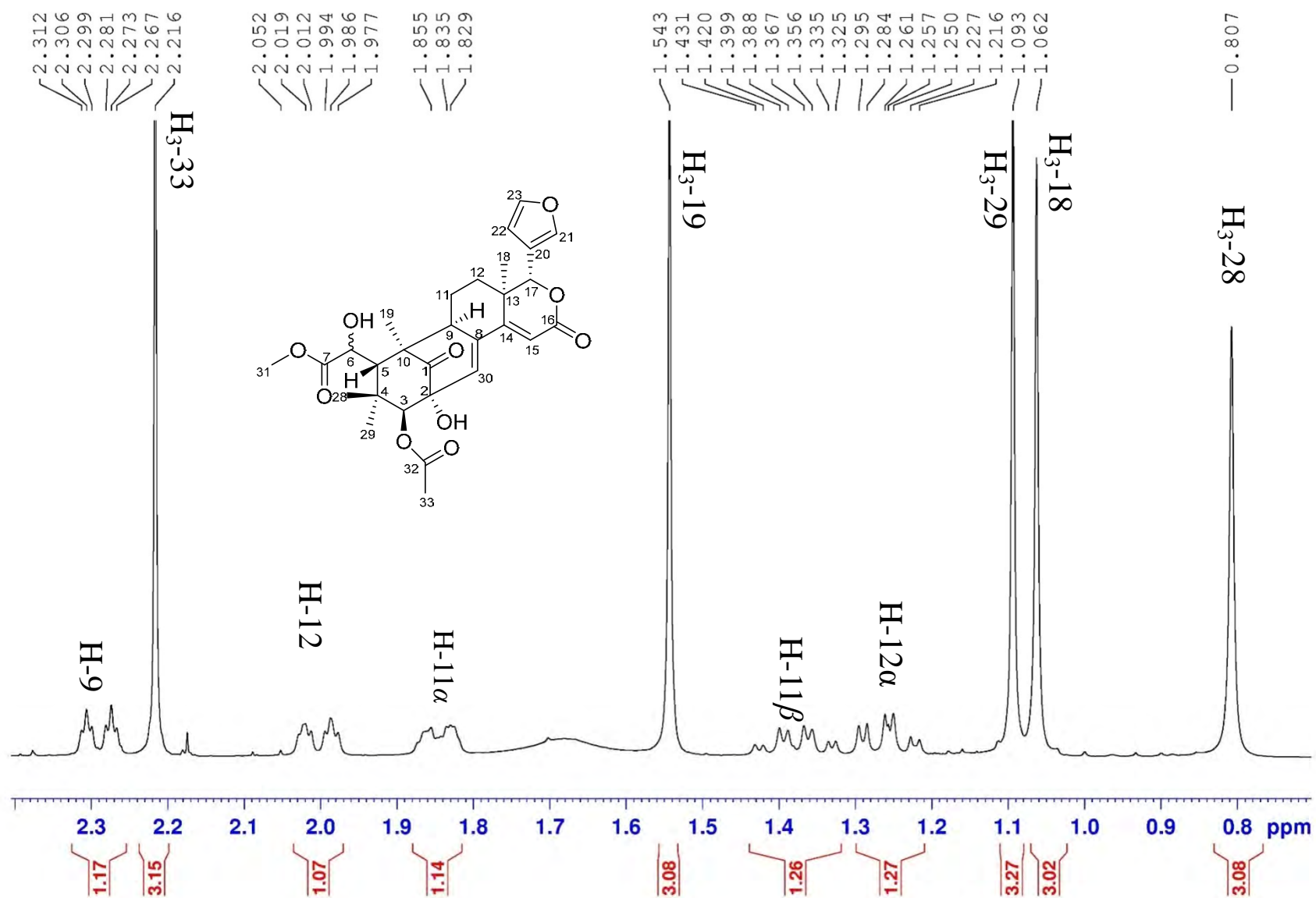


$^1\text{H}$  NMR (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$

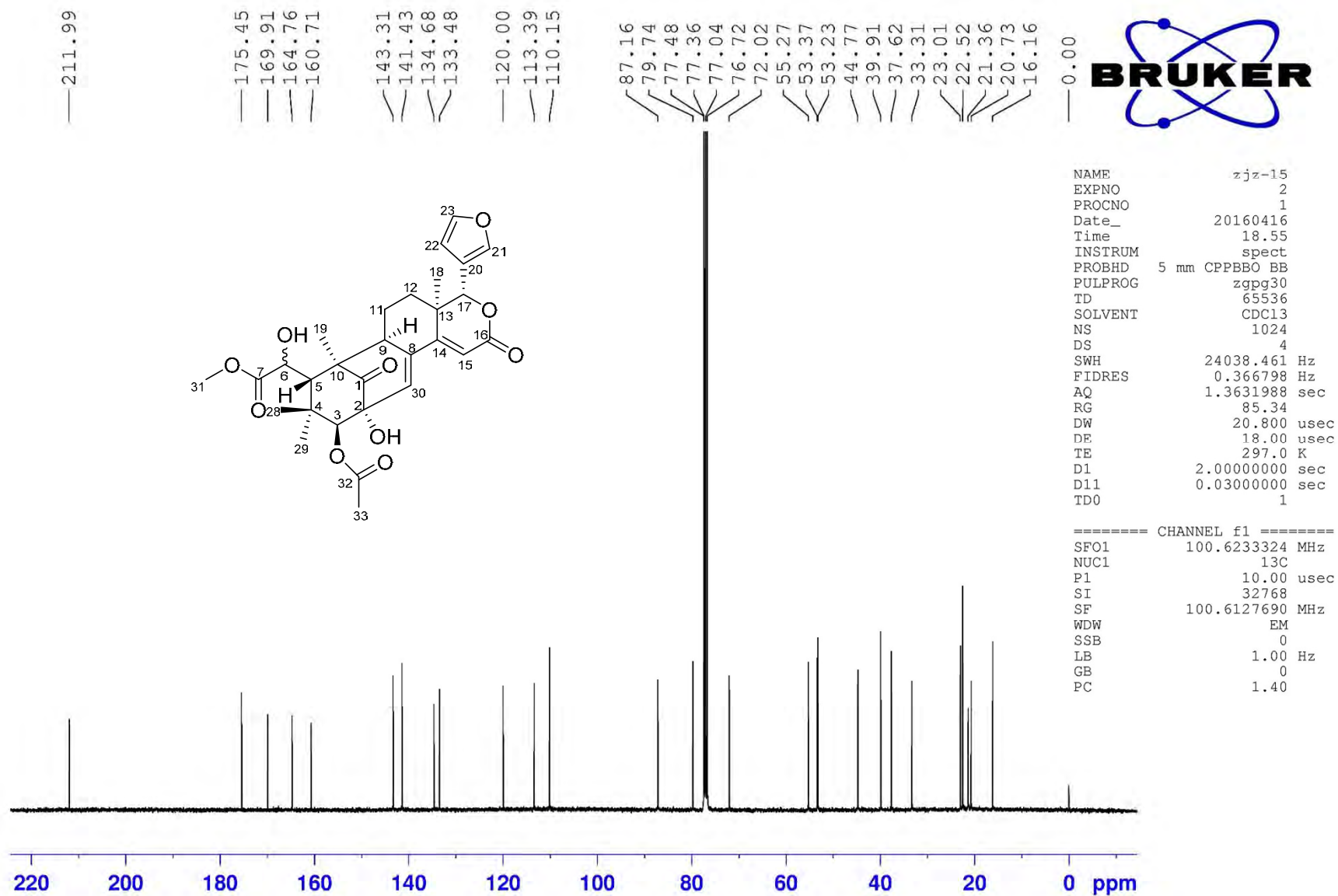




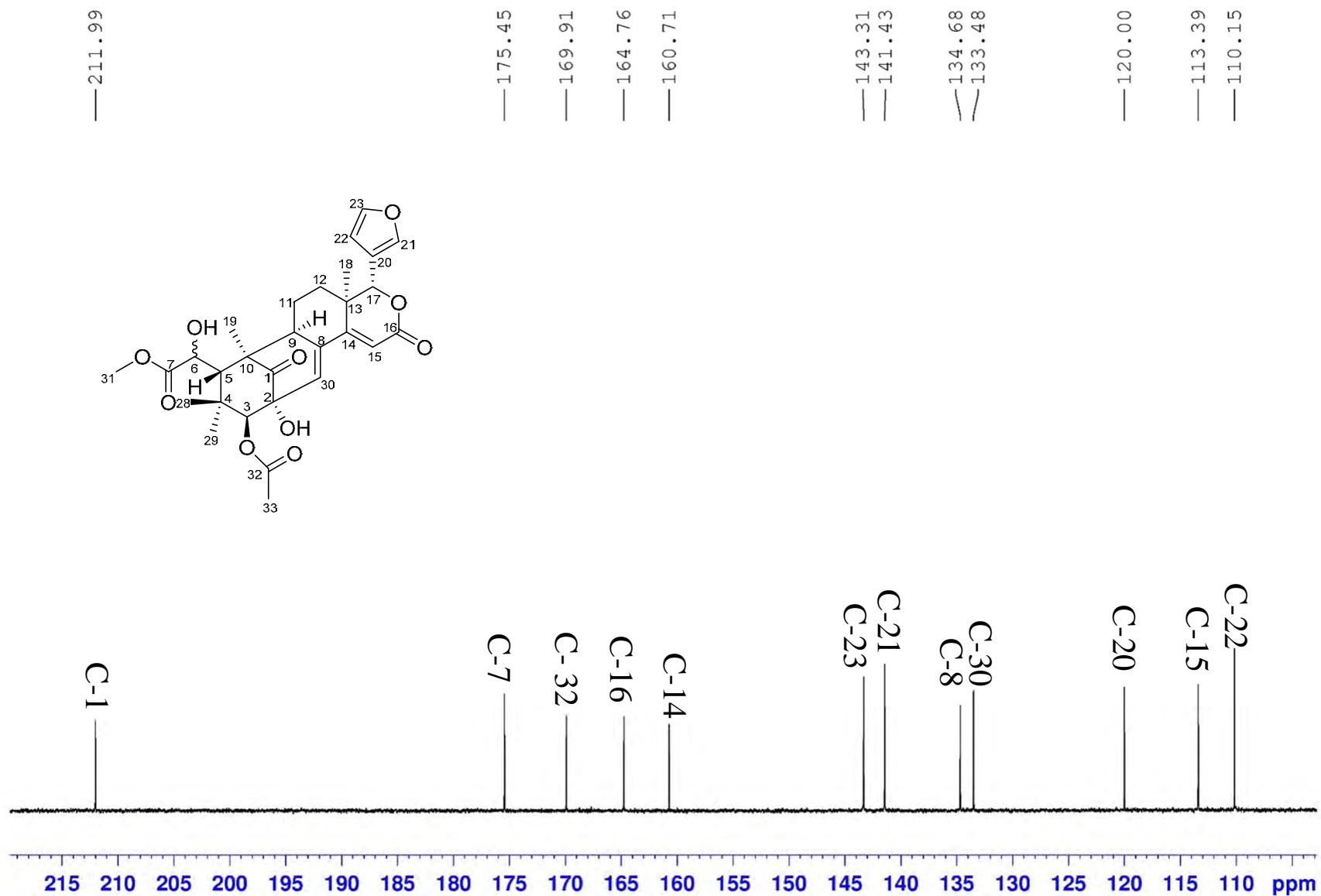
$^1\text{H}$  NMR (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



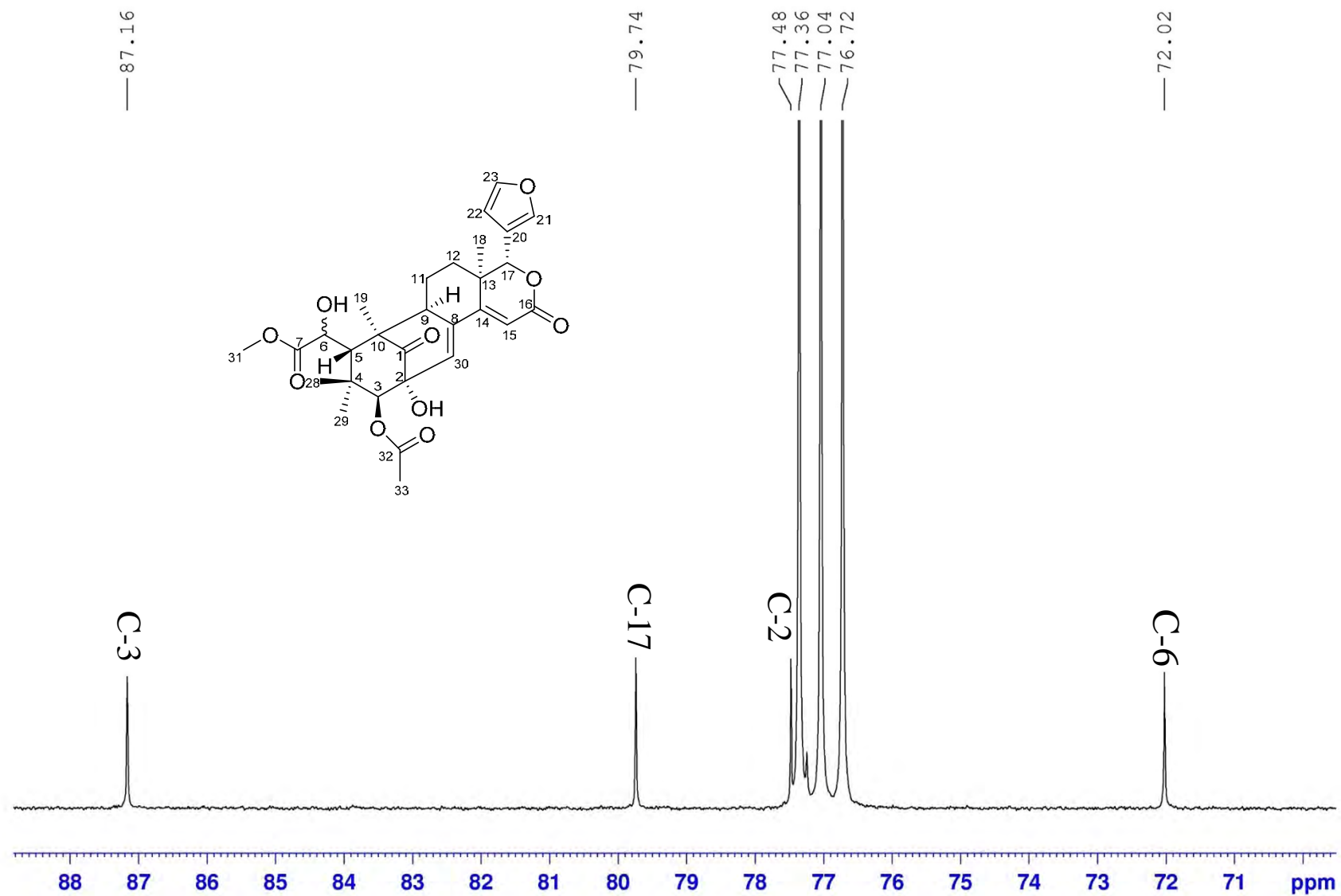
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



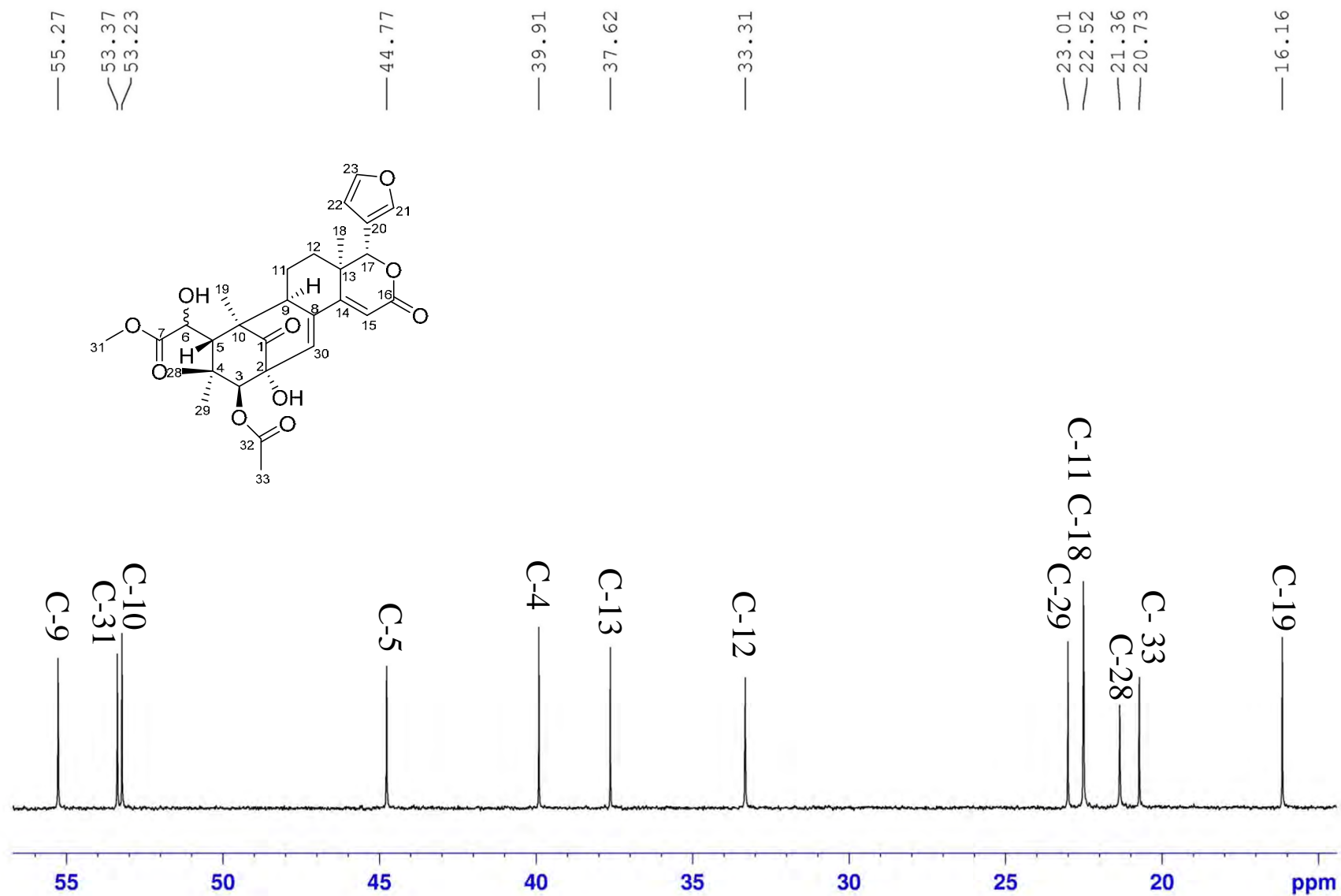
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



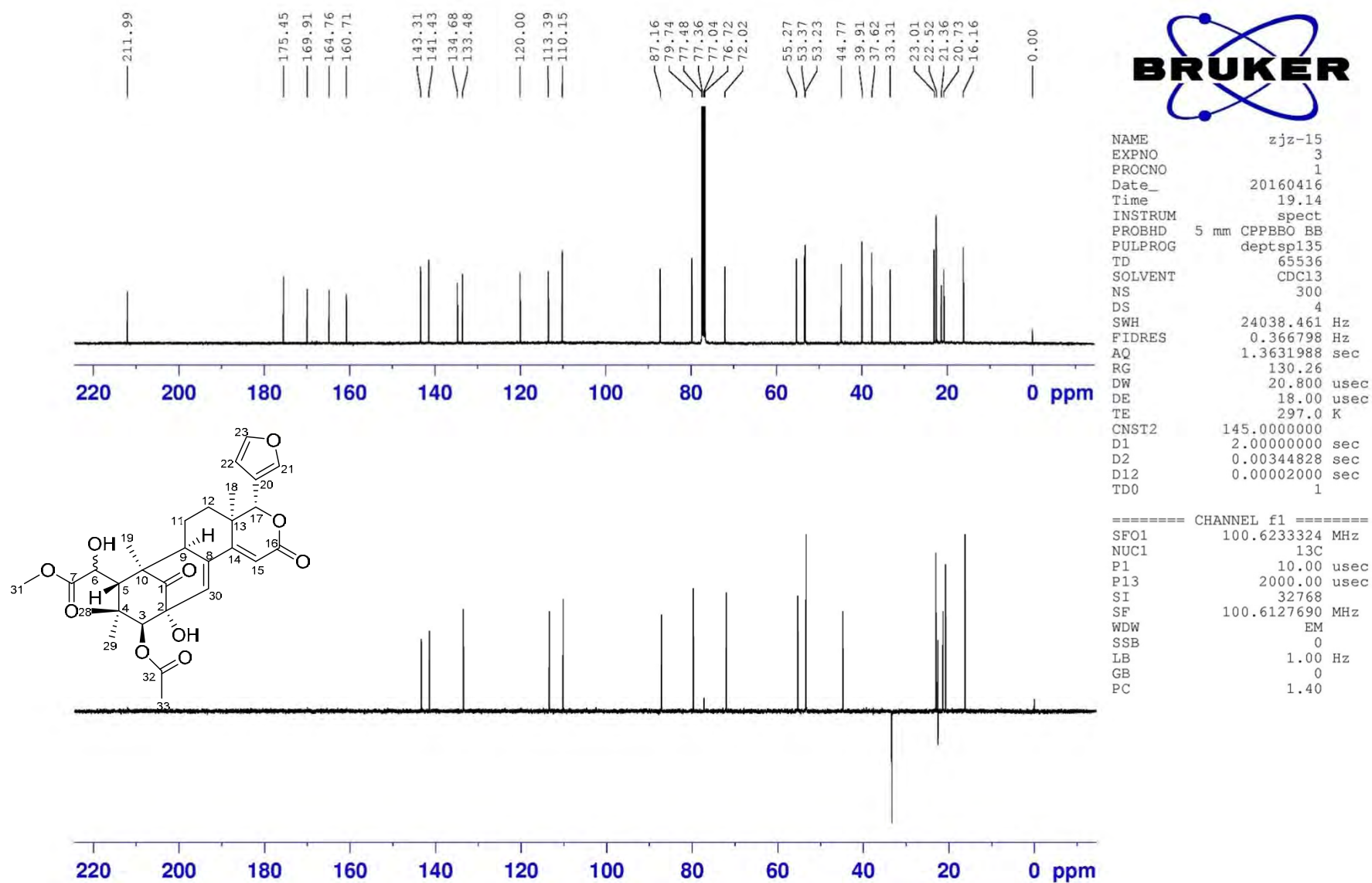
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



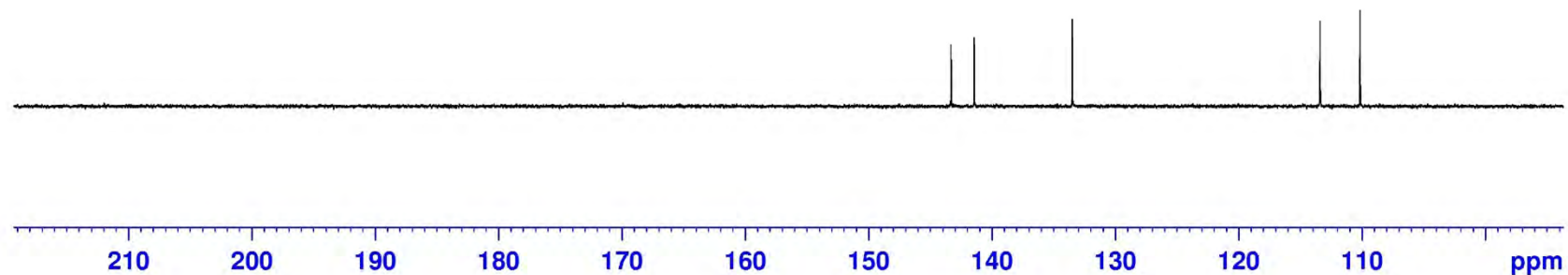
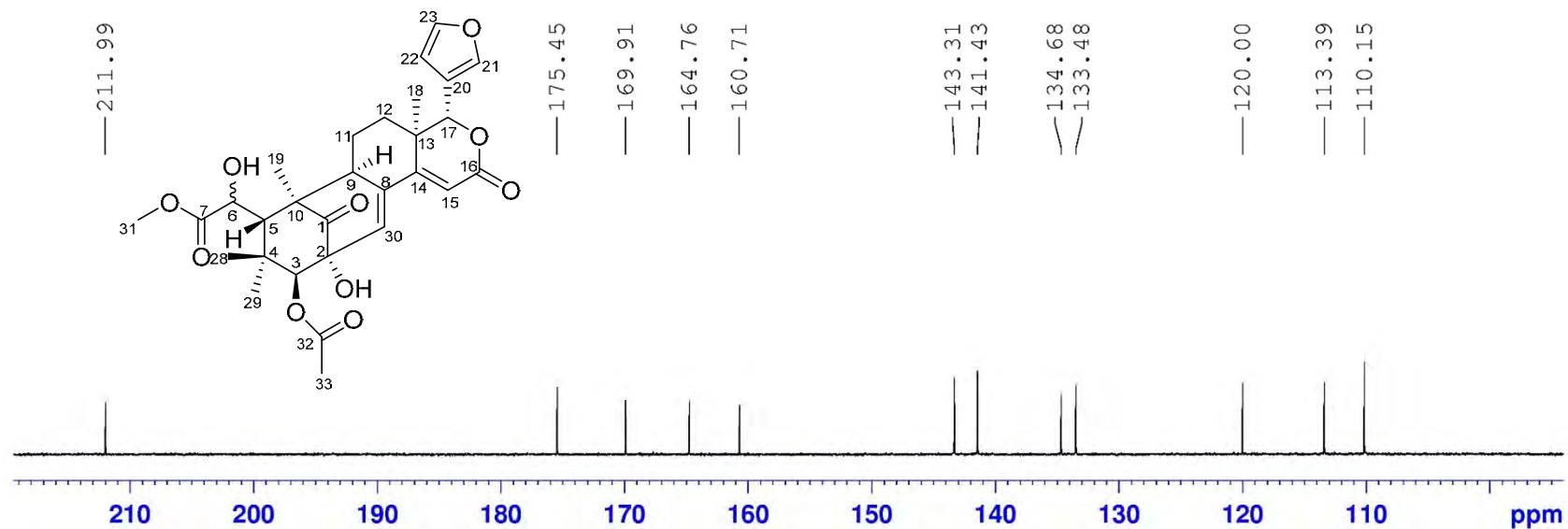
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **10** in CDCl<sub>3</sub>

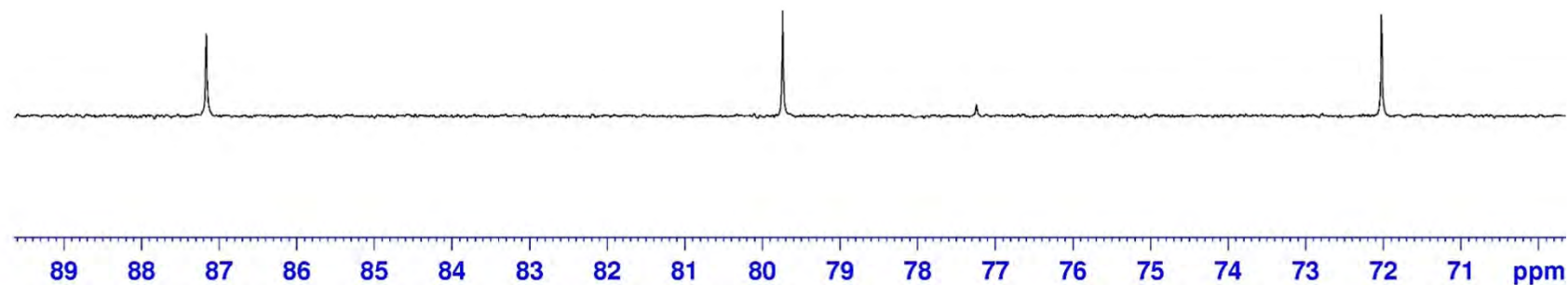
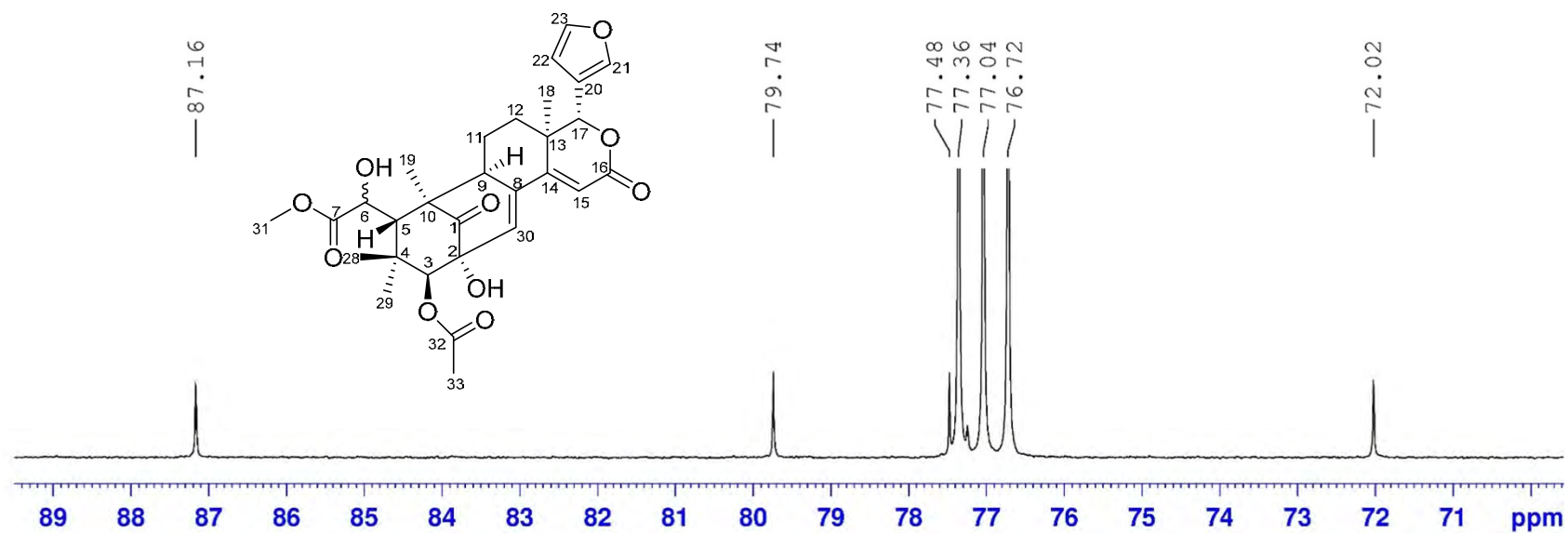


# DEPT135 (100 MHz) spectrum of compound **10** in CDCl<sub>3</sub>

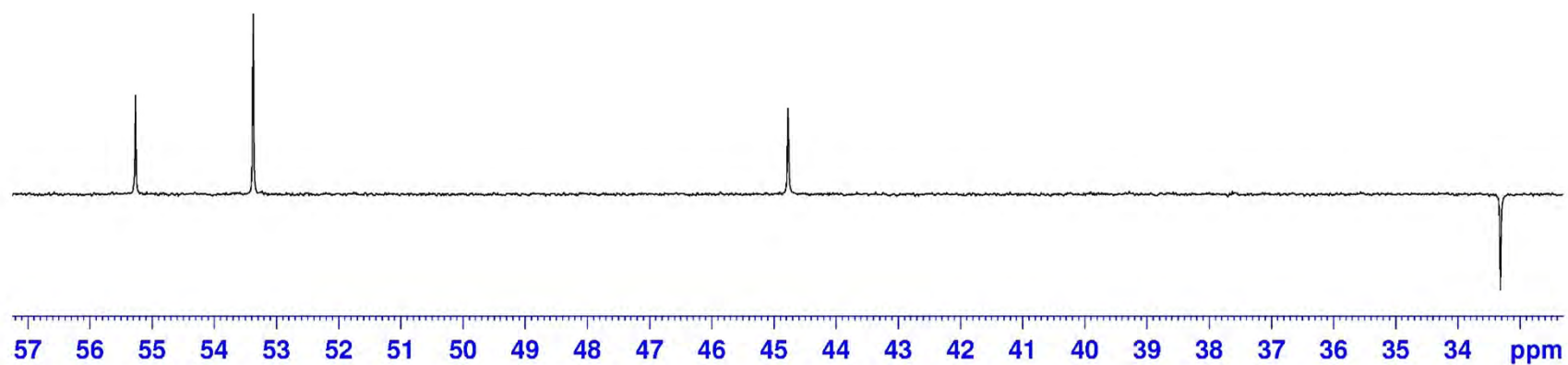
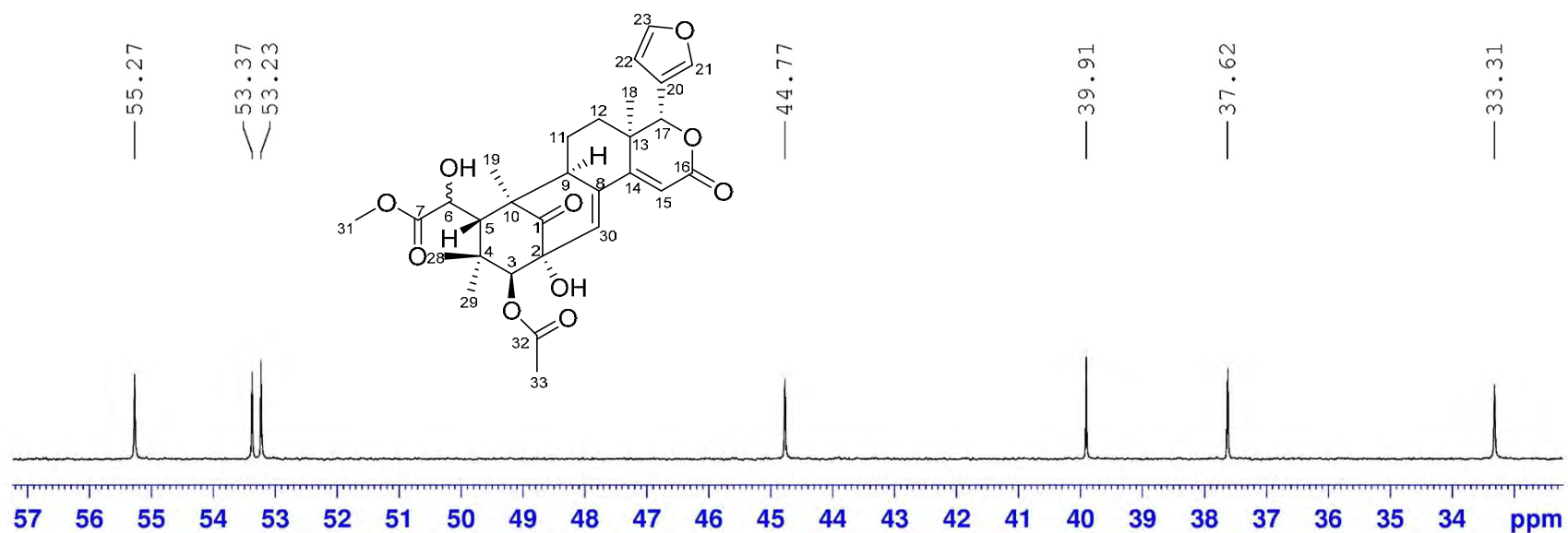




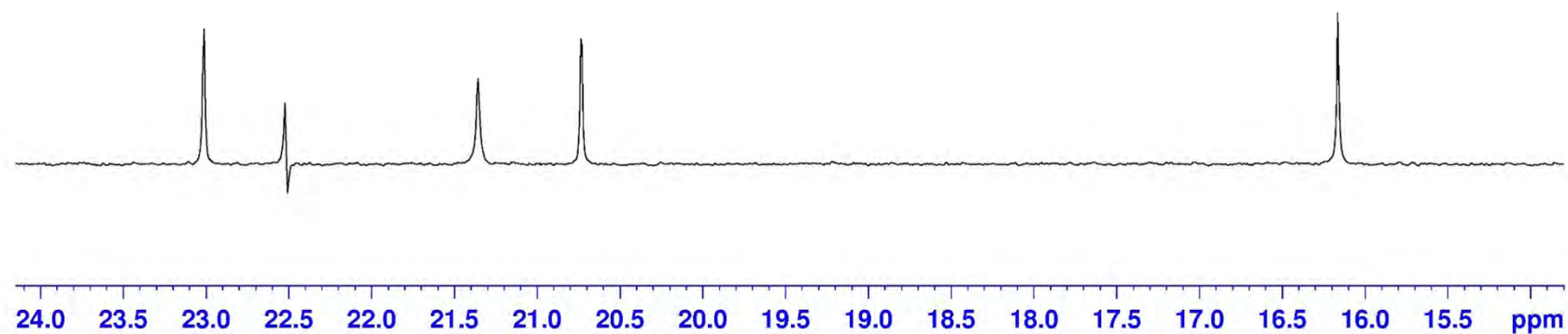
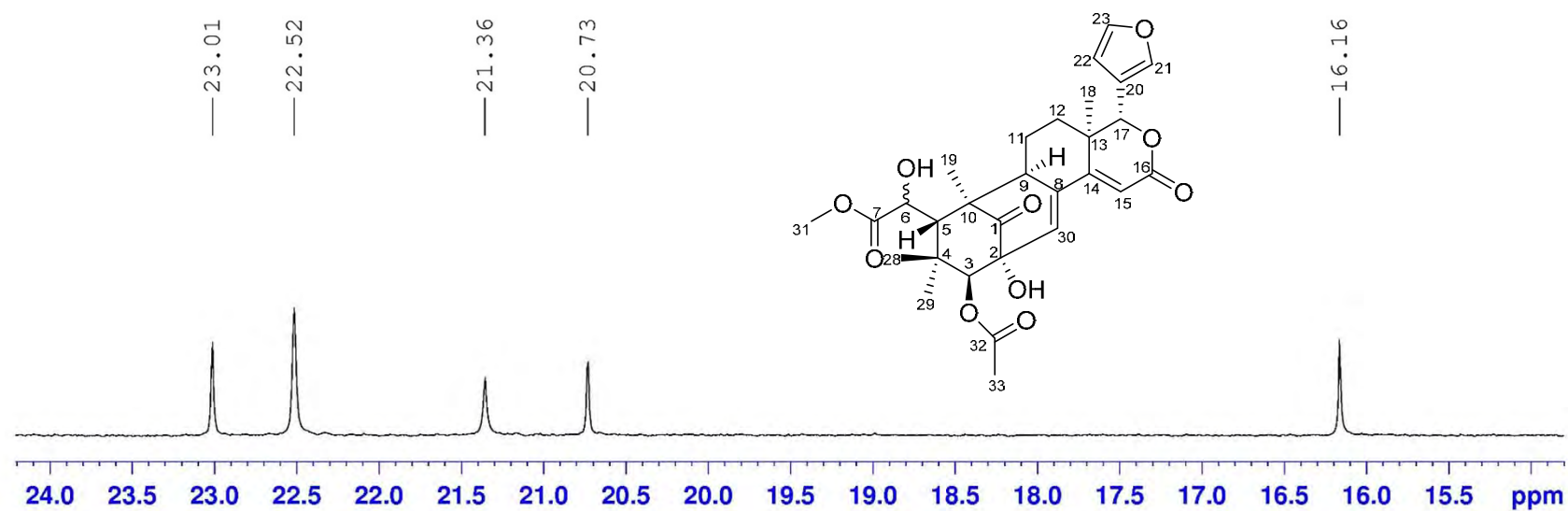
DEPT135 (100 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



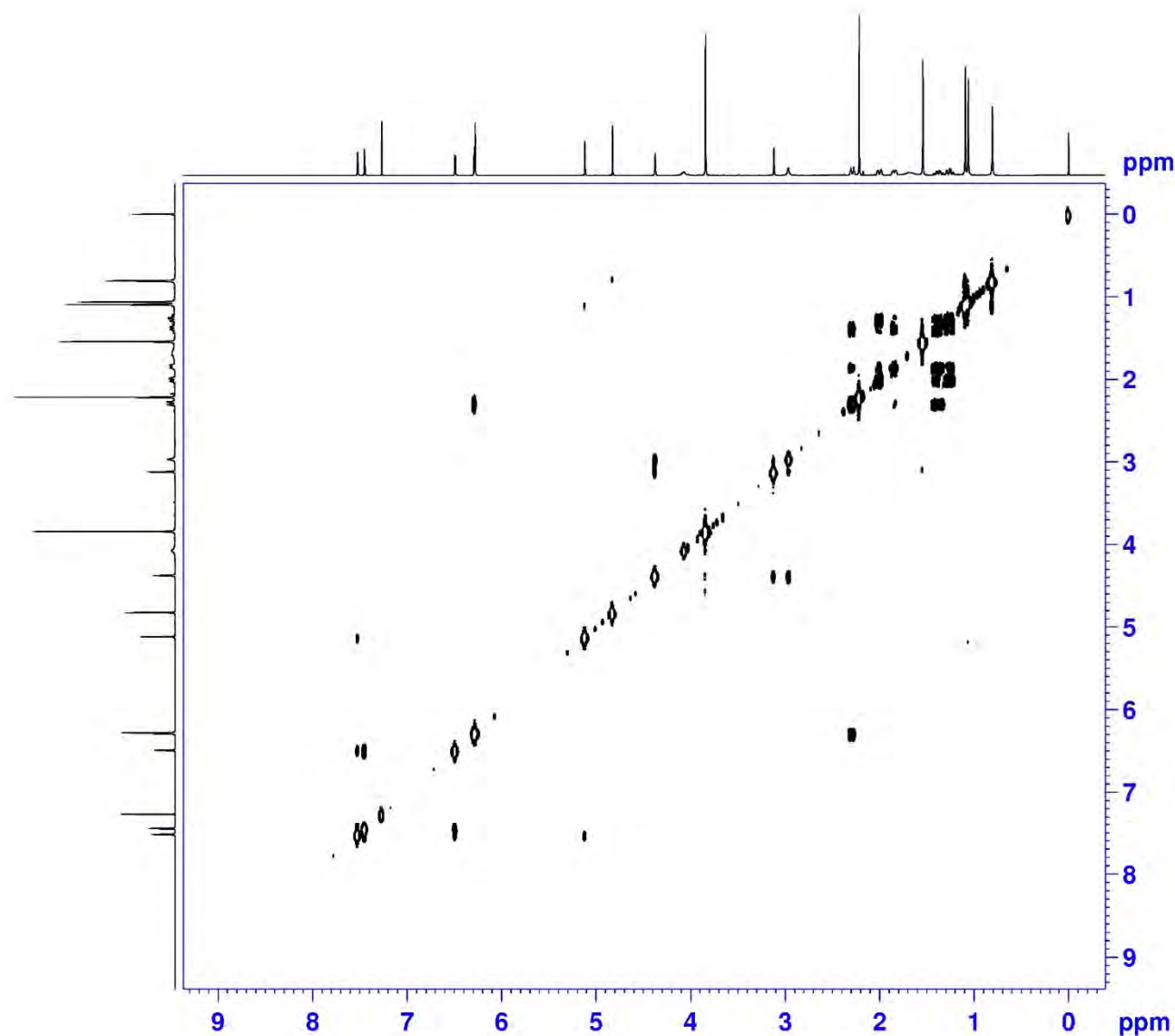
DEPT135 (100 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



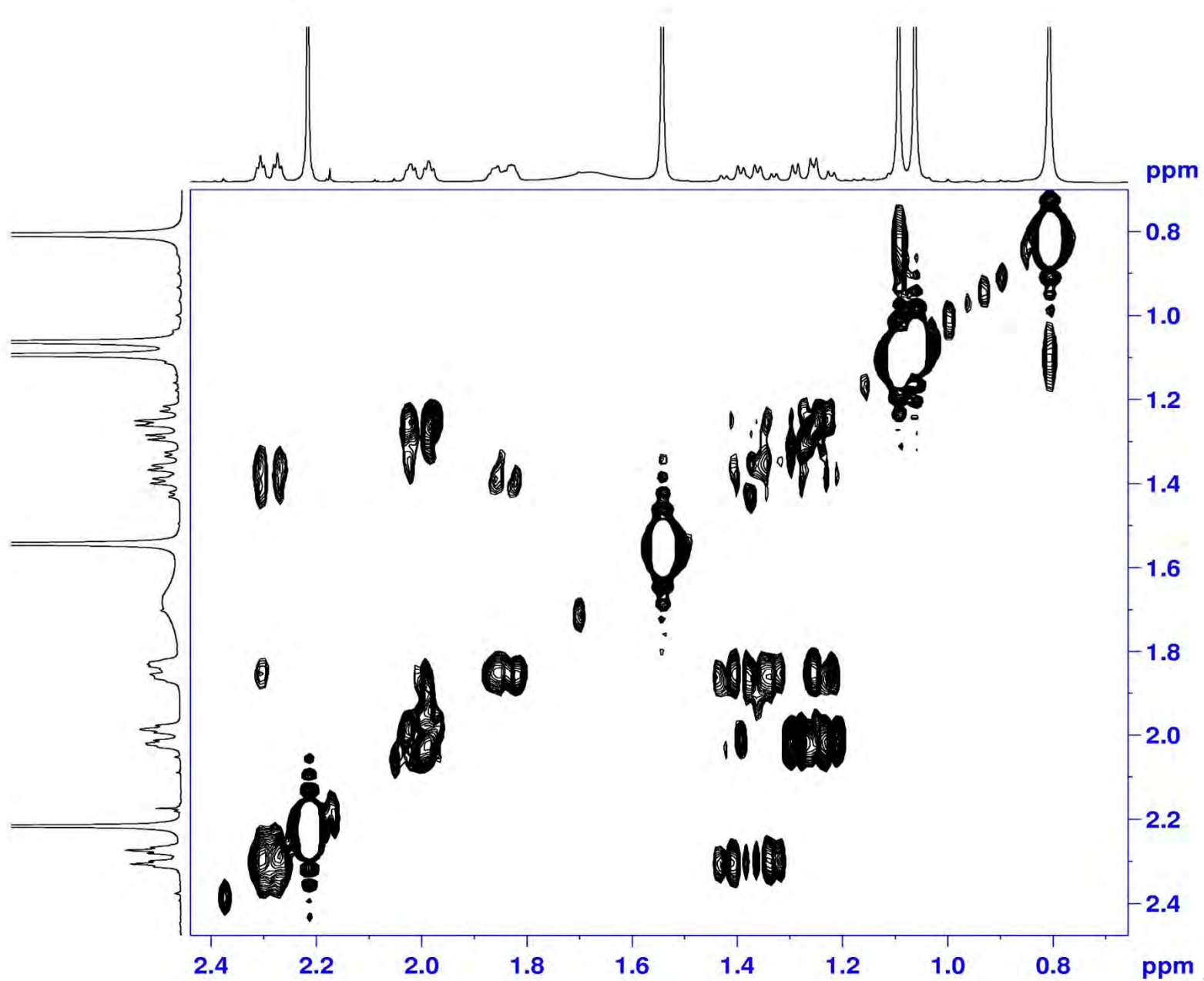
# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **10** in $\text{CDCl}_3$



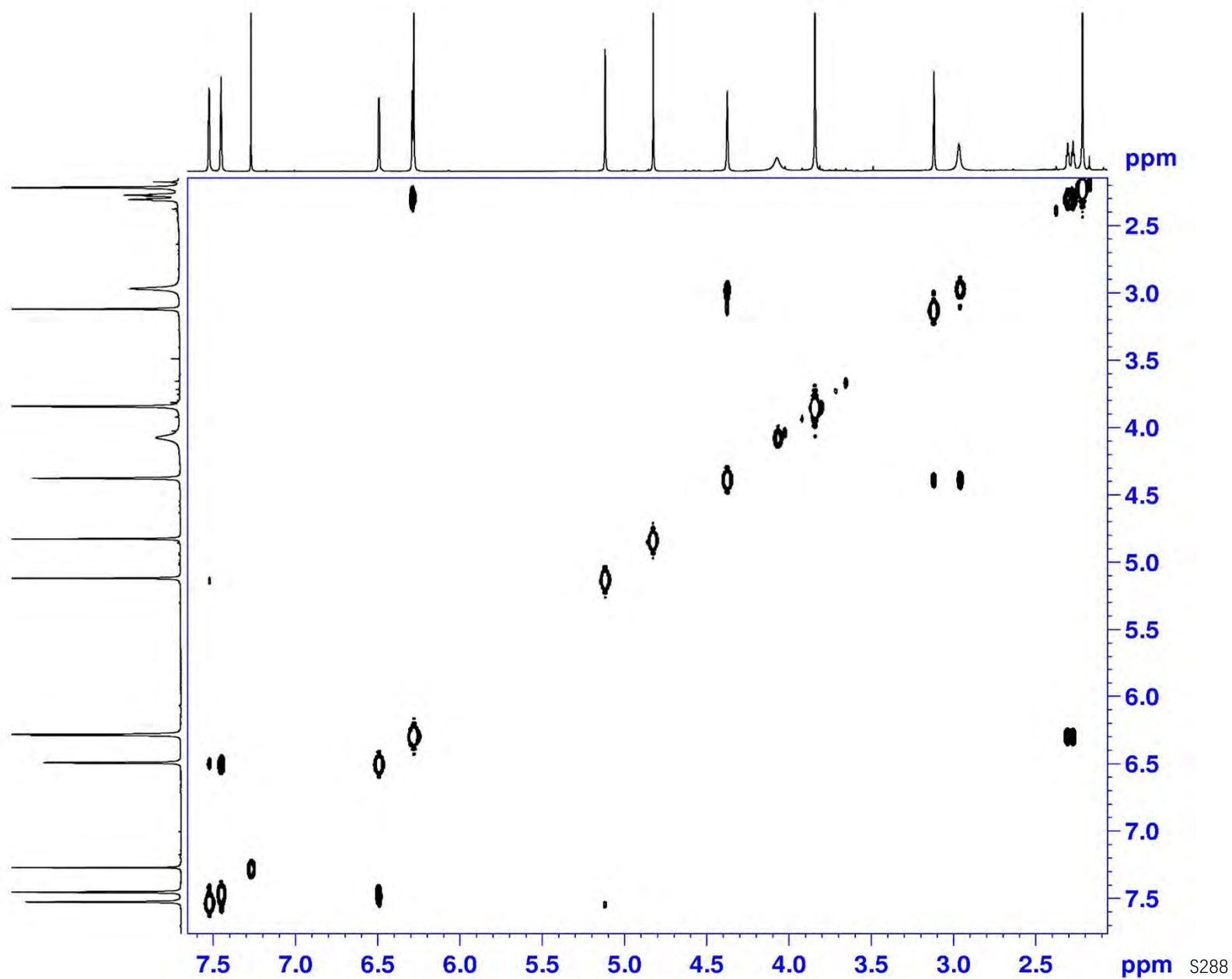
```
NAME                zjz-15
EXPNO                4
PROCNO               1
Date_                20160517
Time                9.25
INSTRUM              spect
PROBHD               5 mm CPPBBO BB
PULPROG              cosygpppqf
TD                   2048
SOLVENT              CDCl3
NS                   8
DS                   8
SWH                  3906.250 Hz
FIDRES               1.907349 Hz
AQ                   0.2621940 sec
RG                   208.5
DW                   128.000 usec
DE                   10.00 usec
TE                   297.0 K
D0                   0.00000300 sec
D1                   1.89678097 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00025600 sec
```

```
===== CHANNEL f1 =====
SFO1                  400.1318006 MHz
NUC1                   1H
P0                     11.50 usec
P1                     11.50 usec
P17                    2500.00 usec
ND0                     1
TD                     128
SFO1                  400.1318 MHz
FIDRES                30.517578 Hz
SW                     9.762 ppm
FnMODE                 QF
SI                     1024
SF                    400.1300061 MHz
WDW                    QSINE
SSB                     0
LB                     0.00 Hz
GB                     0
PC                     1.40
SI                     1024
MC2                    QF
SF                    400.1300005 MHz
WDW                    QSINE
SSB                     0
LB                     0.00 Hz
GB                     0
```

$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$

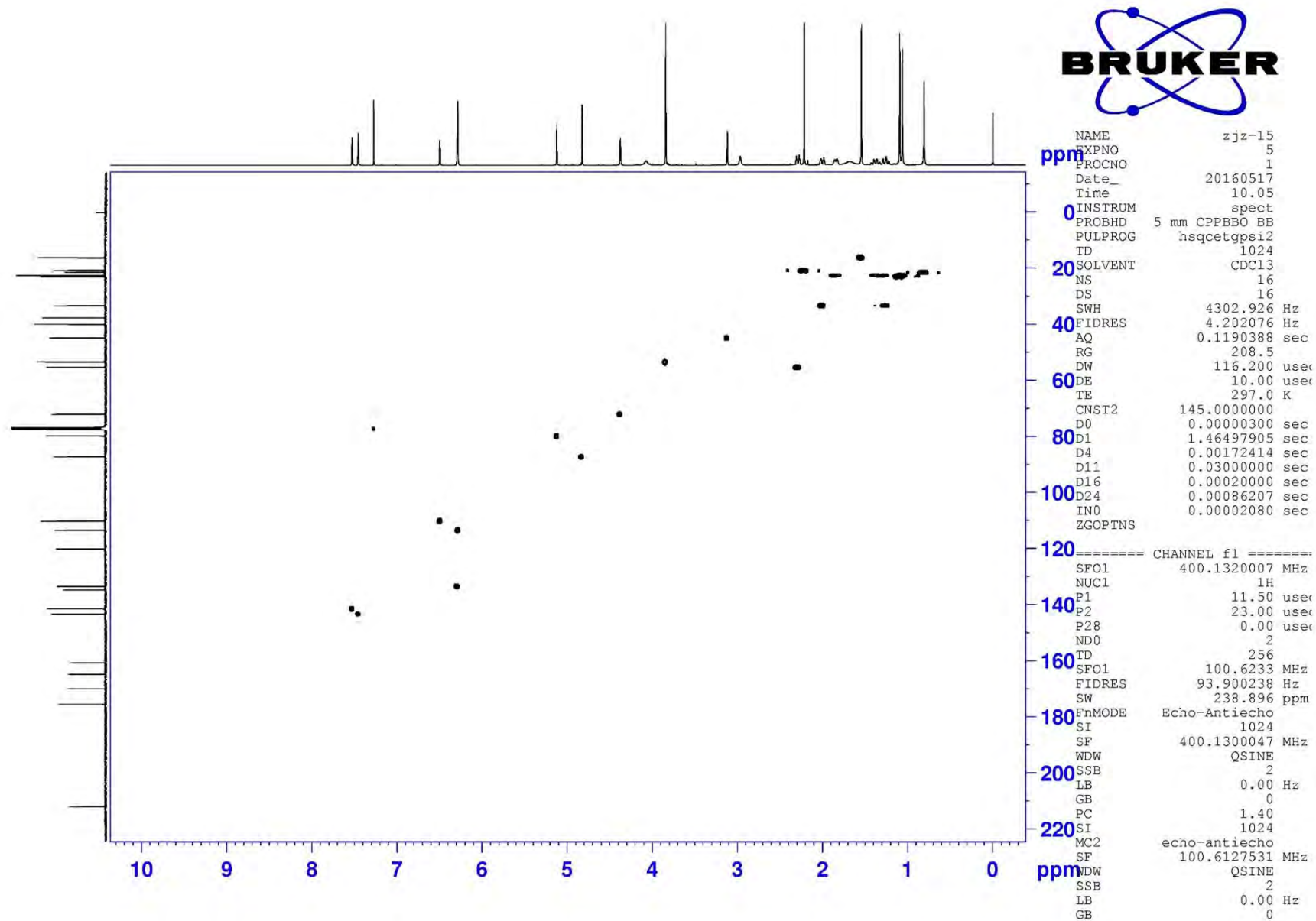


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



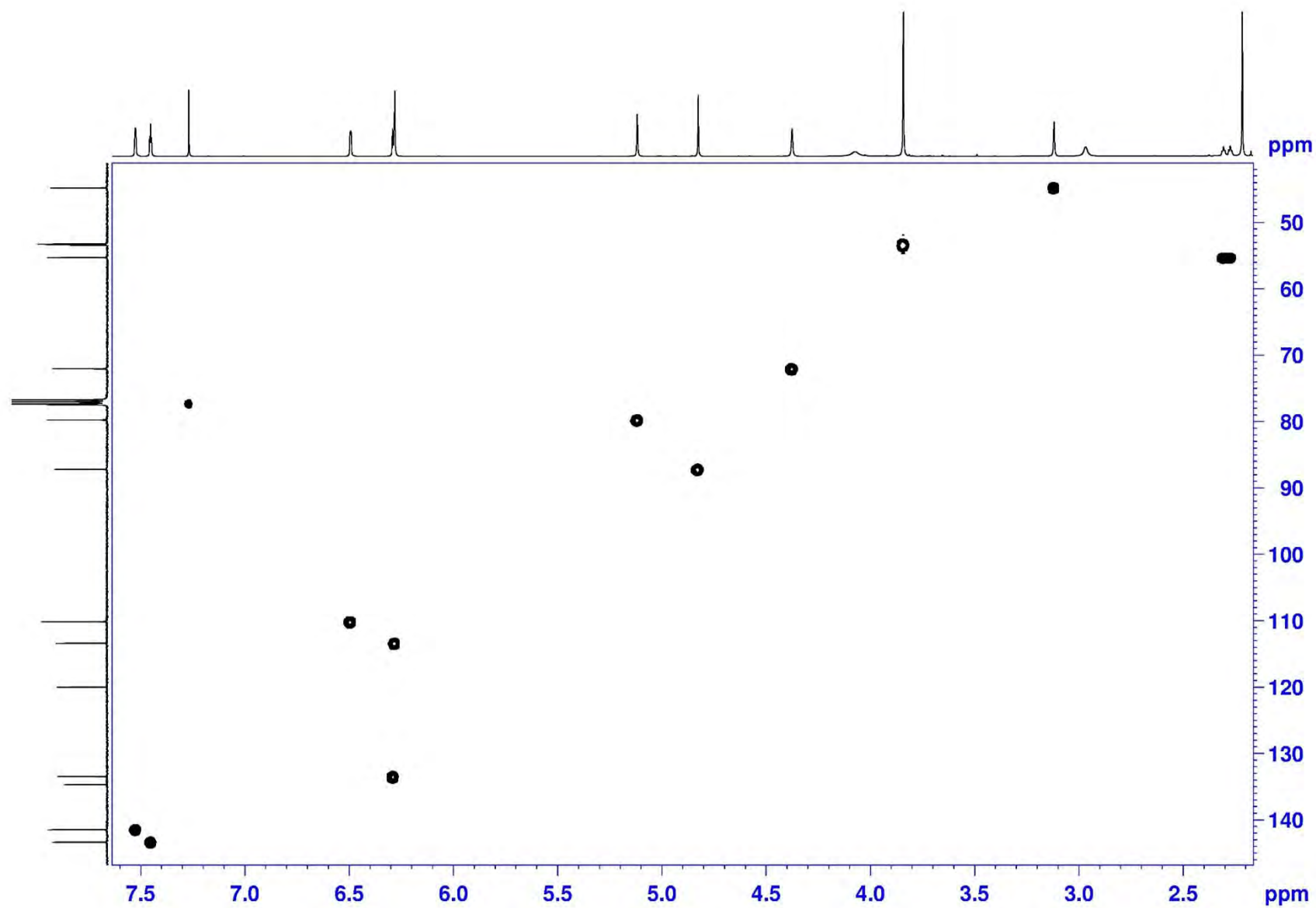


# HSQC (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>

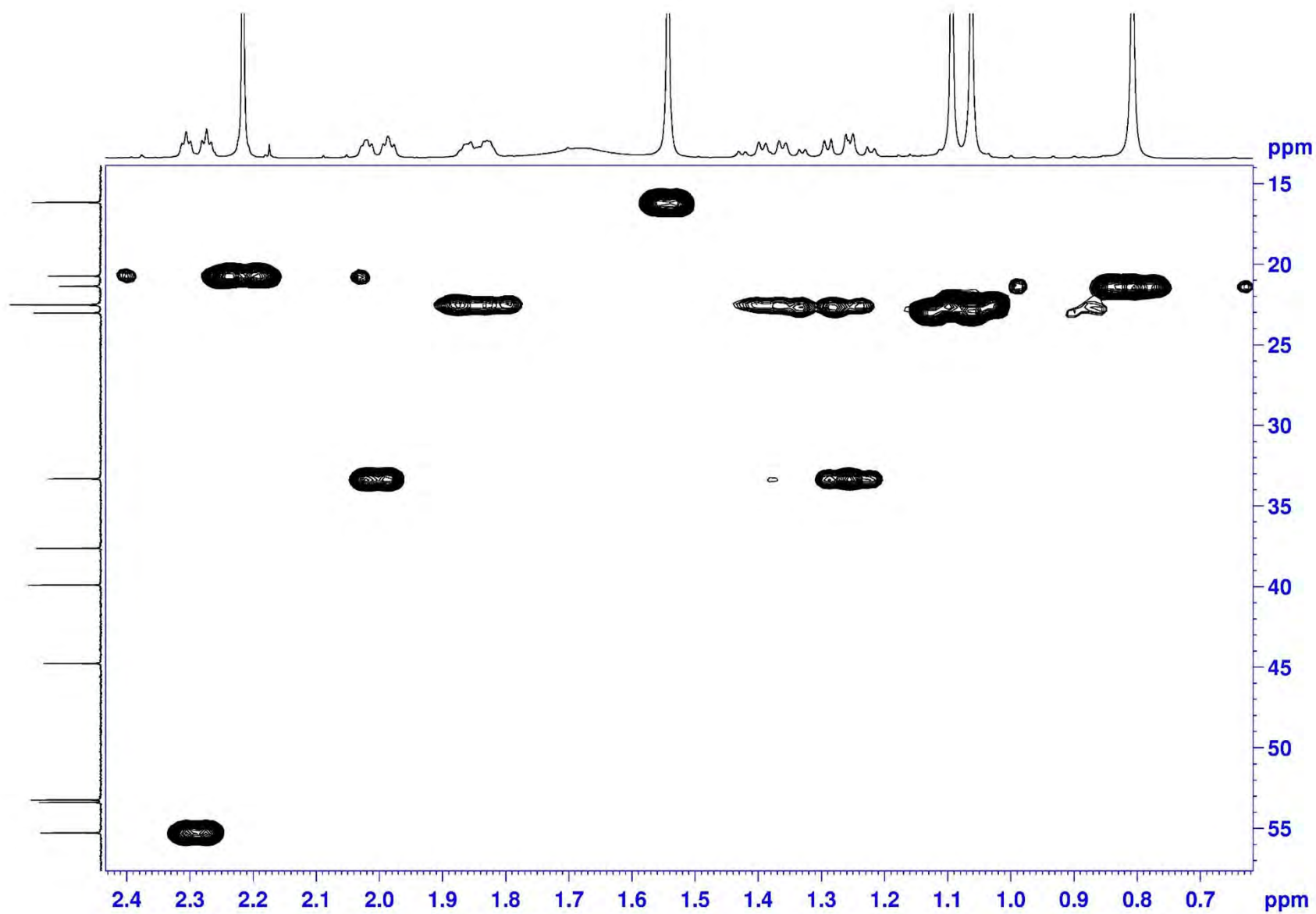




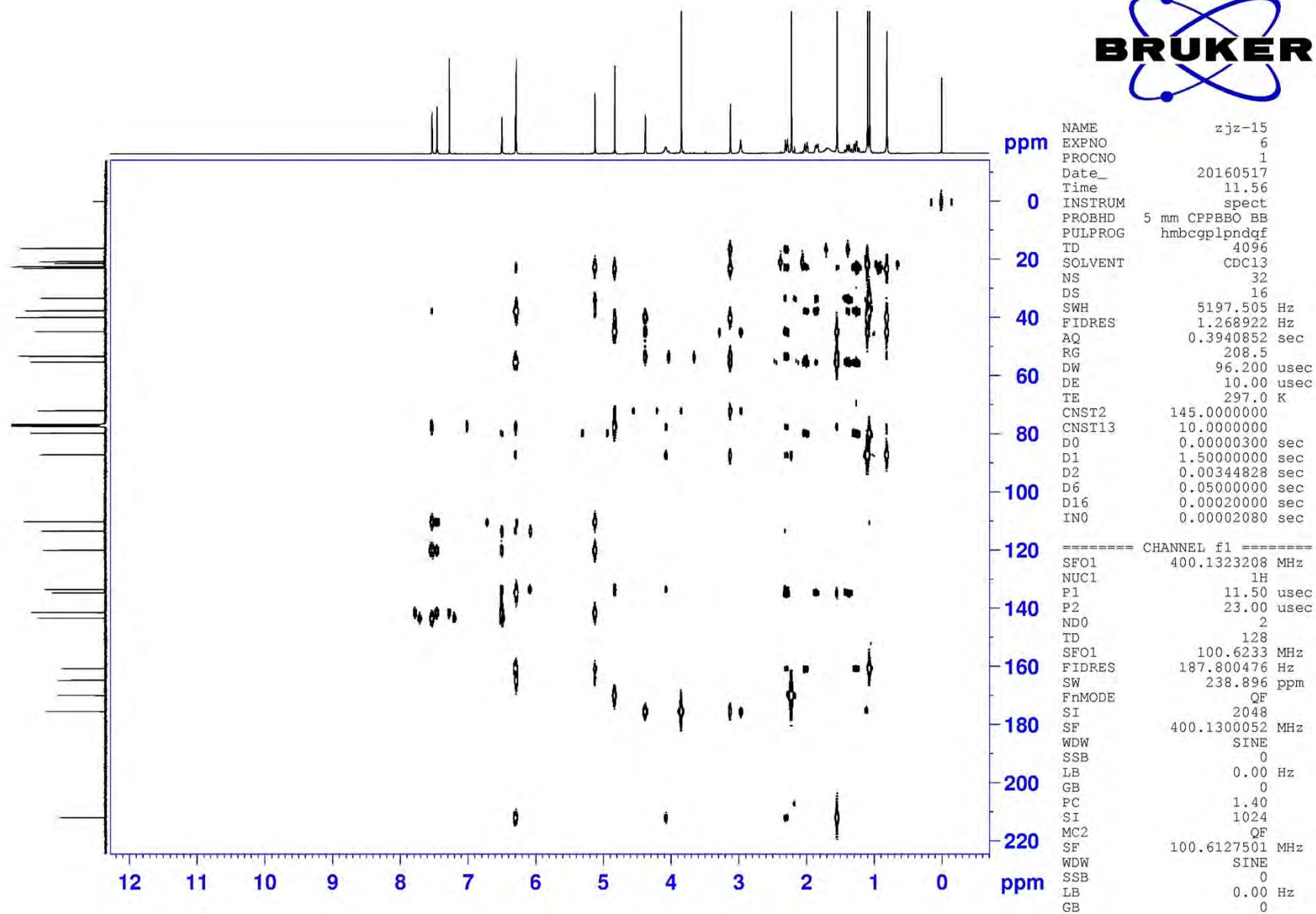
HSQC (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



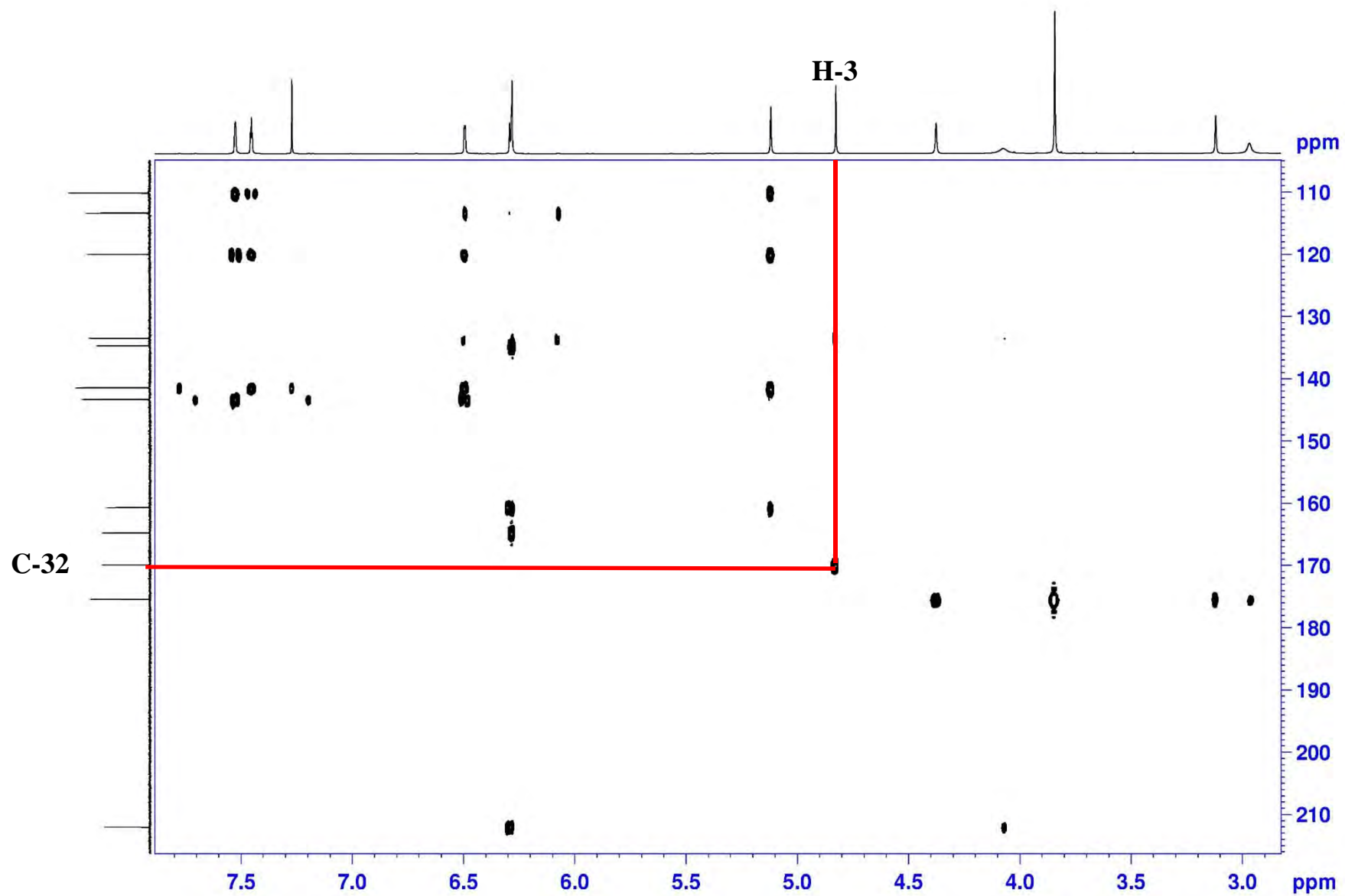
HSQC (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



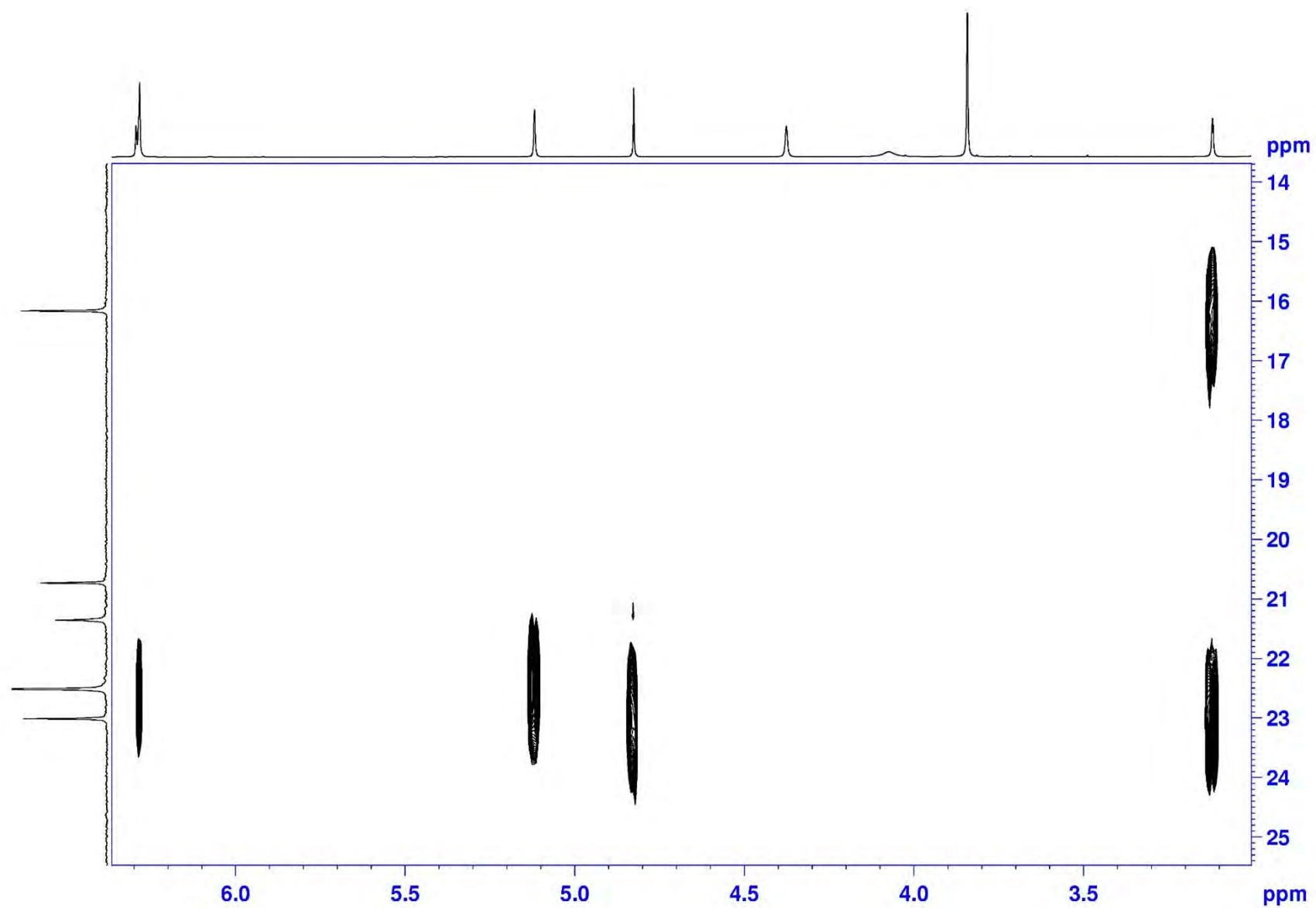
# HMBC (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



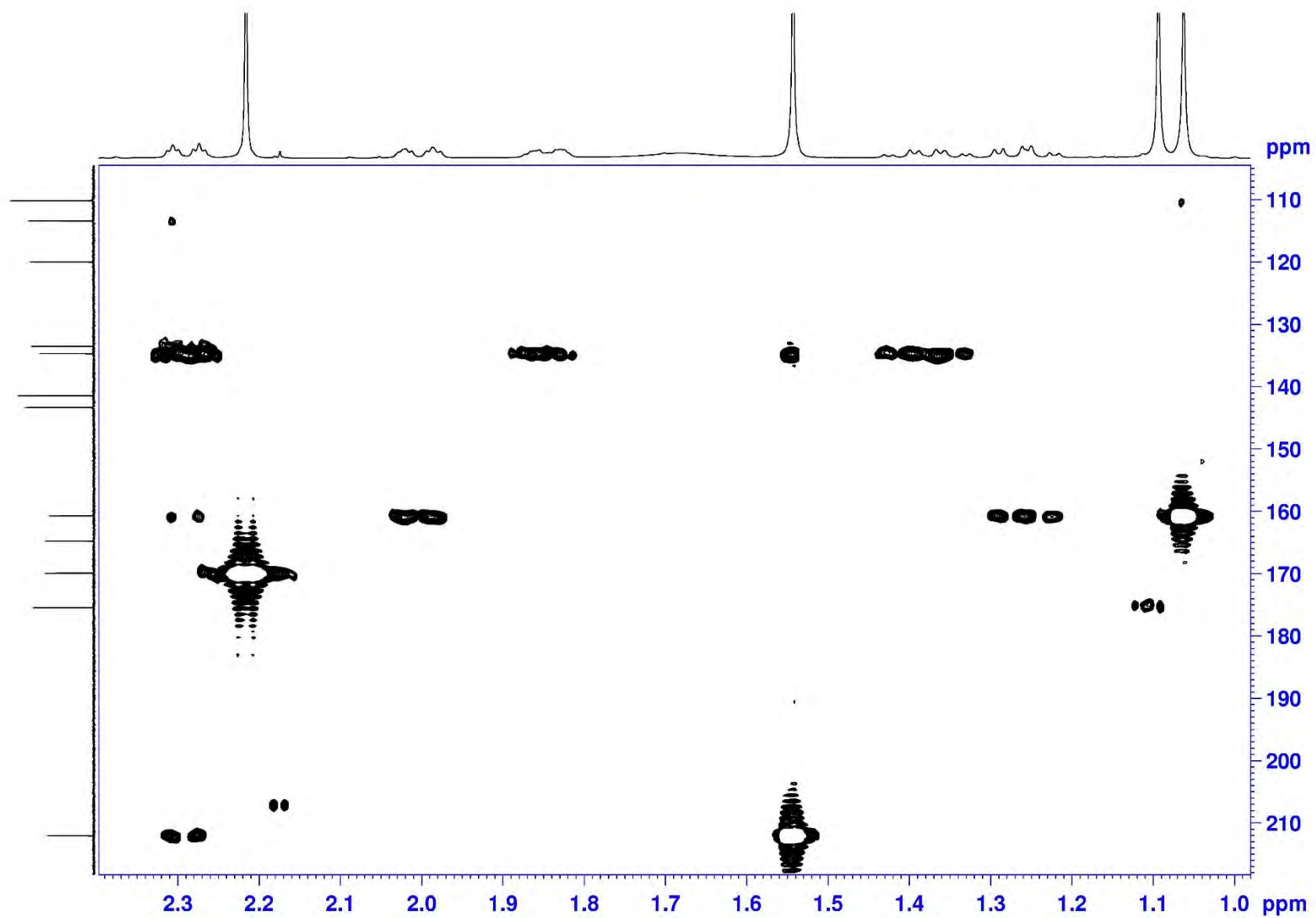
HMBC (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



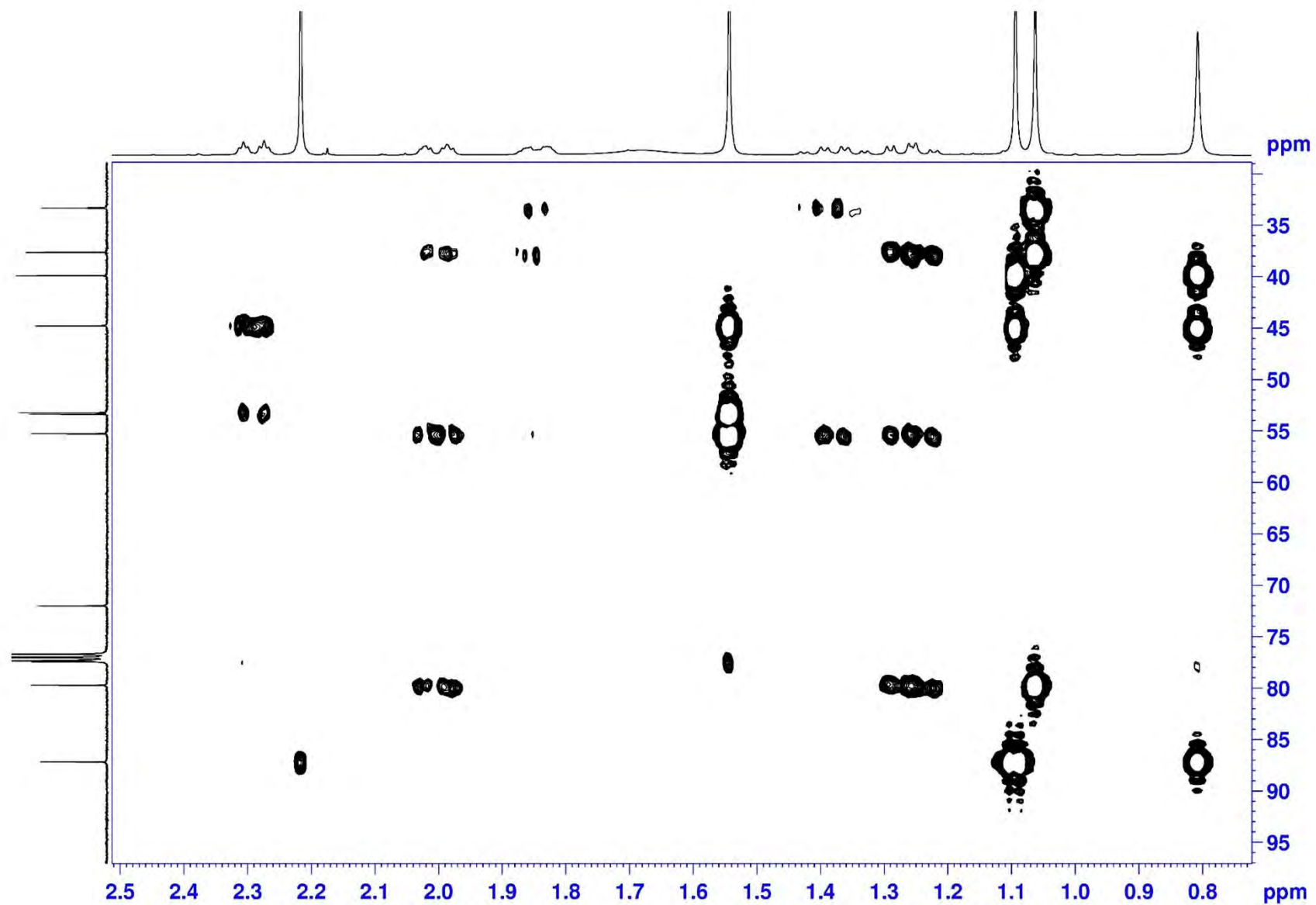
HMBC (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$

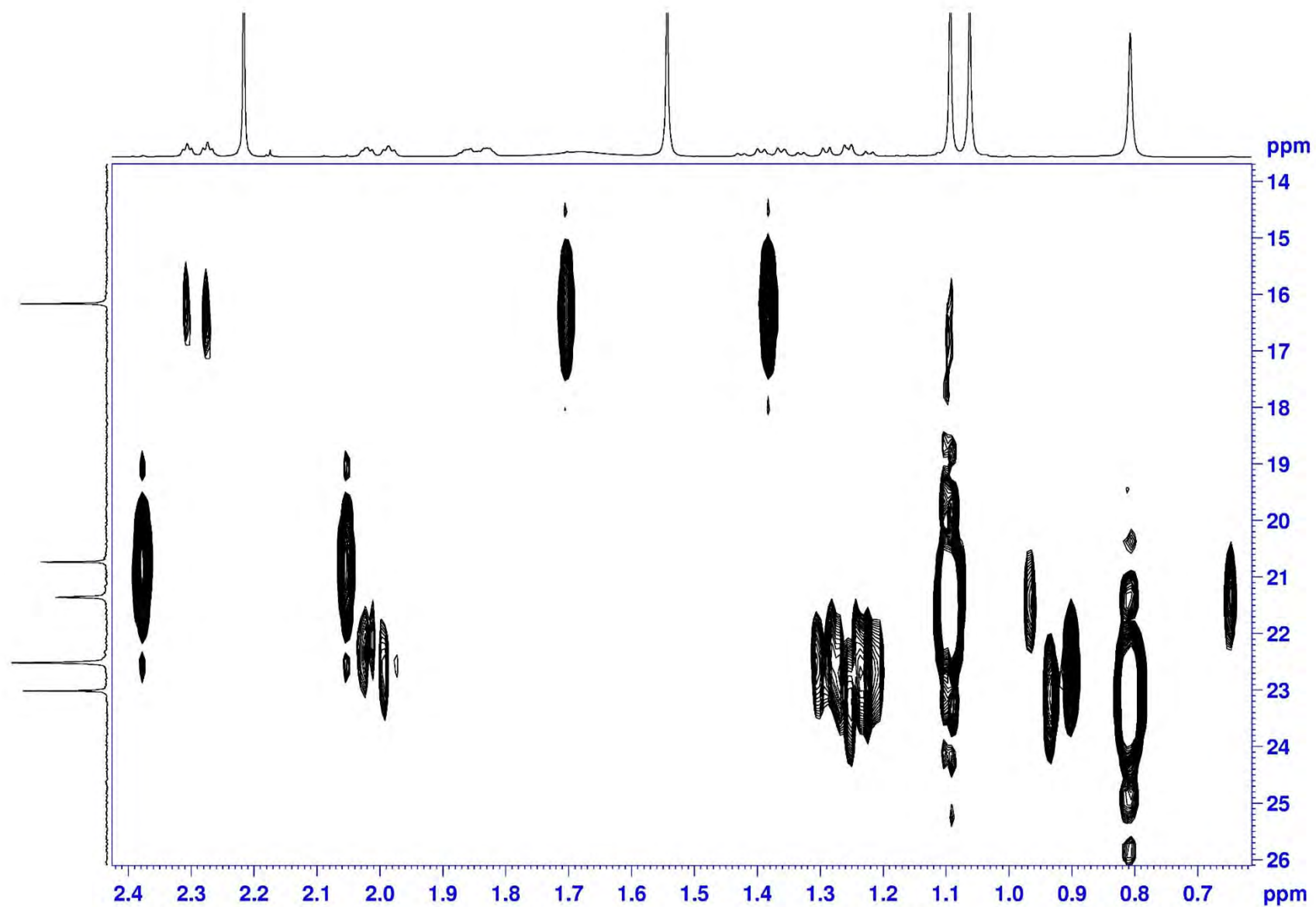


HMBC (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$

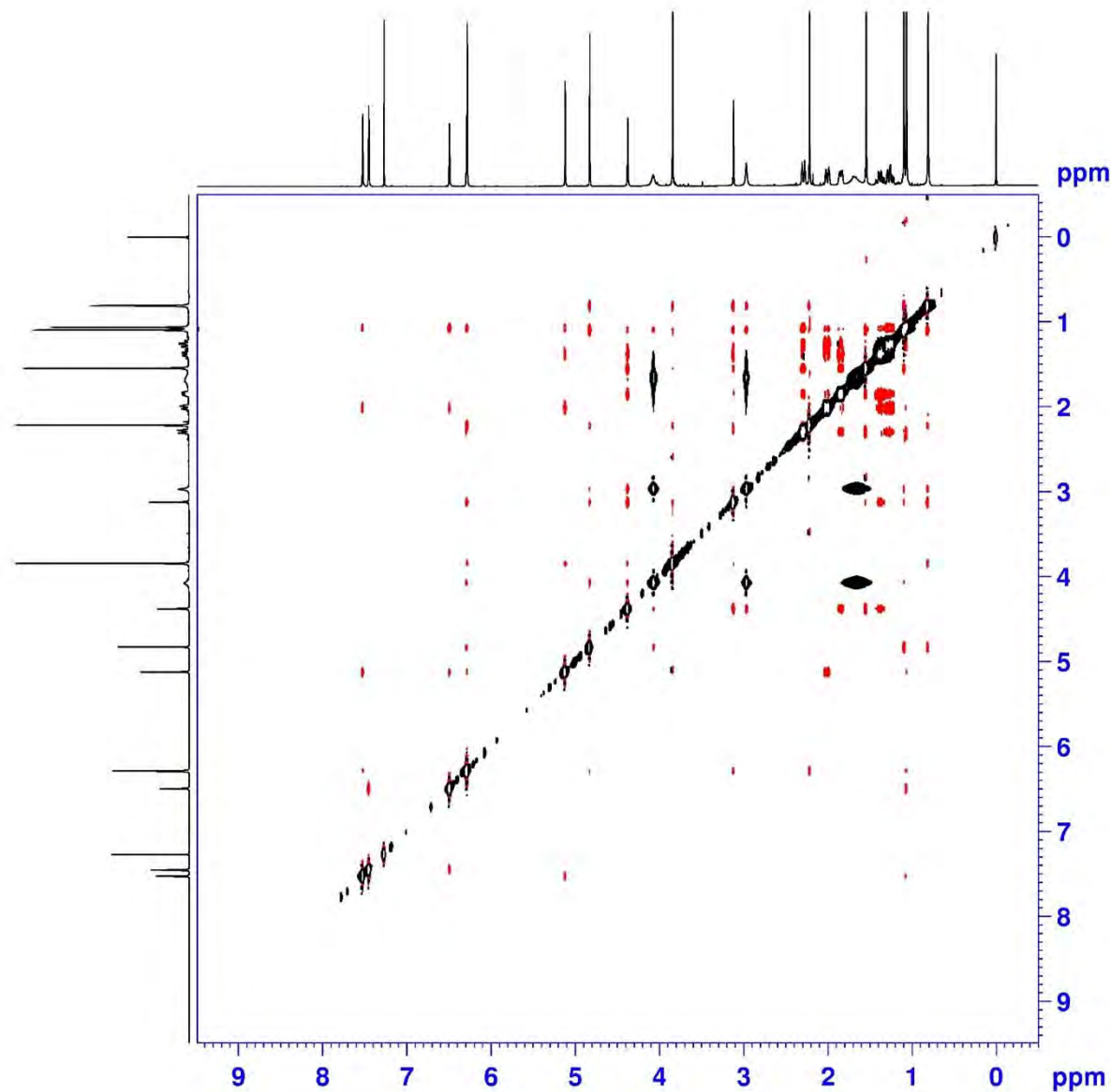




HMBC (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



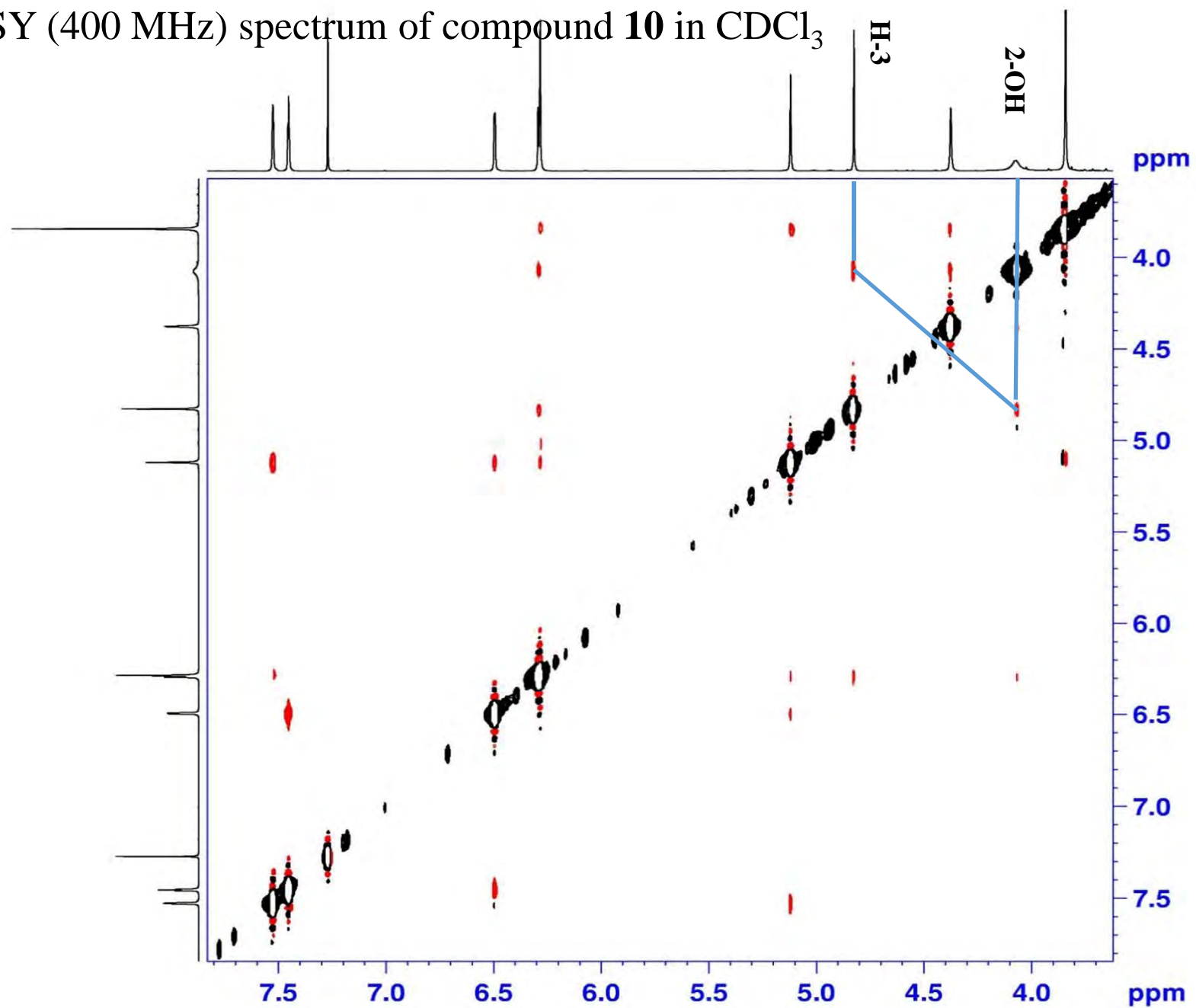
```

NAME          zjz-15
EXPNO         7
PROCNO        1
Date_         20160517
Time          14.12
INSTRUM       spect
PROBHD        5 mm CFPBBO BB
PULPROG       noesygpph
TD            2048
SOLVENT       CDCl3
NS            16
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

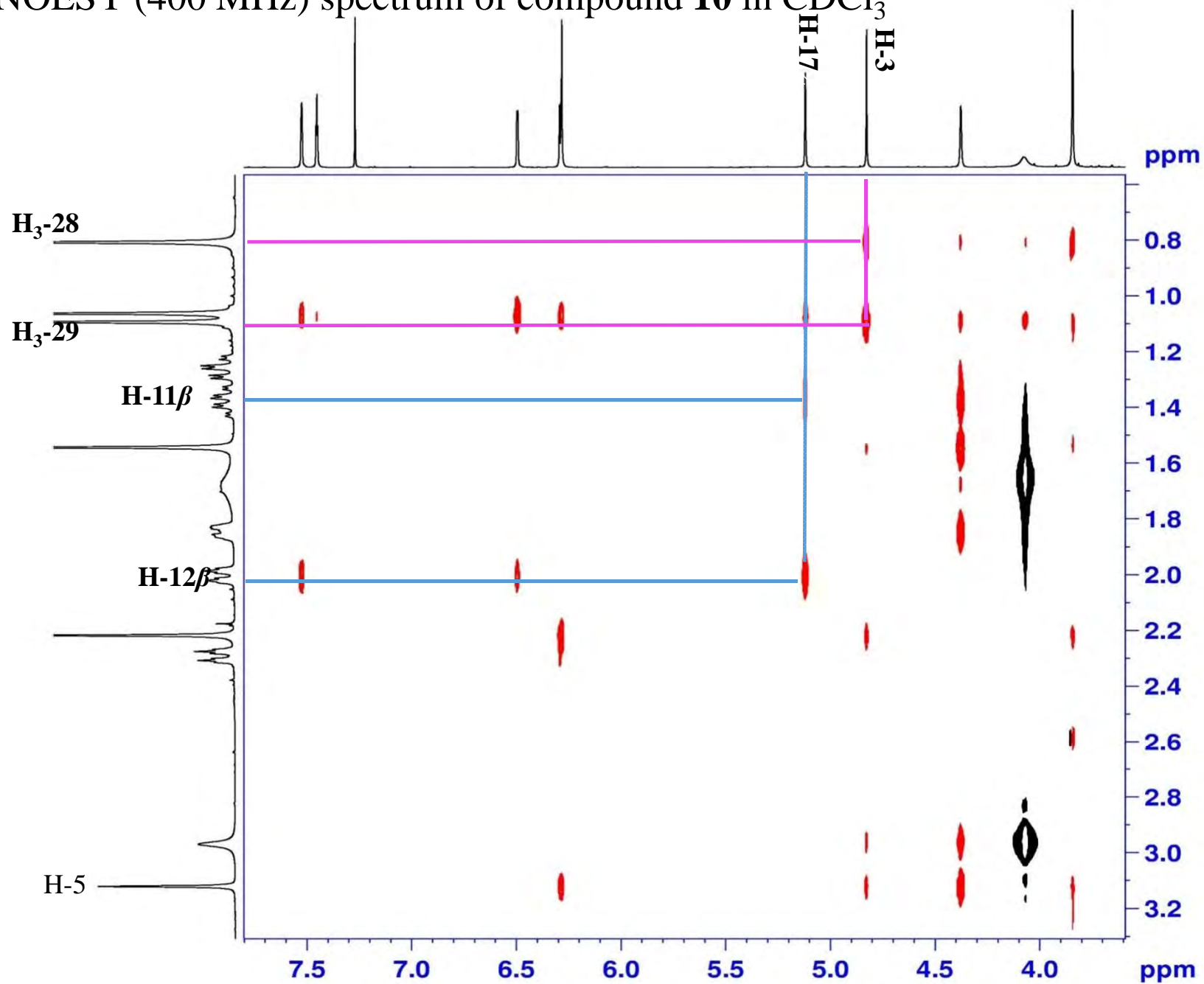
```

===== CHANNEL f1 =====
SFO1         400.1318006 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
P17           2500.00 usec
ND0           1
TD            256
SFO1         400.1318 MHz
FIDRES        15.625000 Hz
SW            9.997 ppm
FnMODE        States-TPPI
SI            1024
SF            400.1300049 MHz
WDW           QSINE
SSB            2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            400.1300039 MHz
WDW           QSINE
SSB            2
LB            0.00 Hz
GB            0
    
```

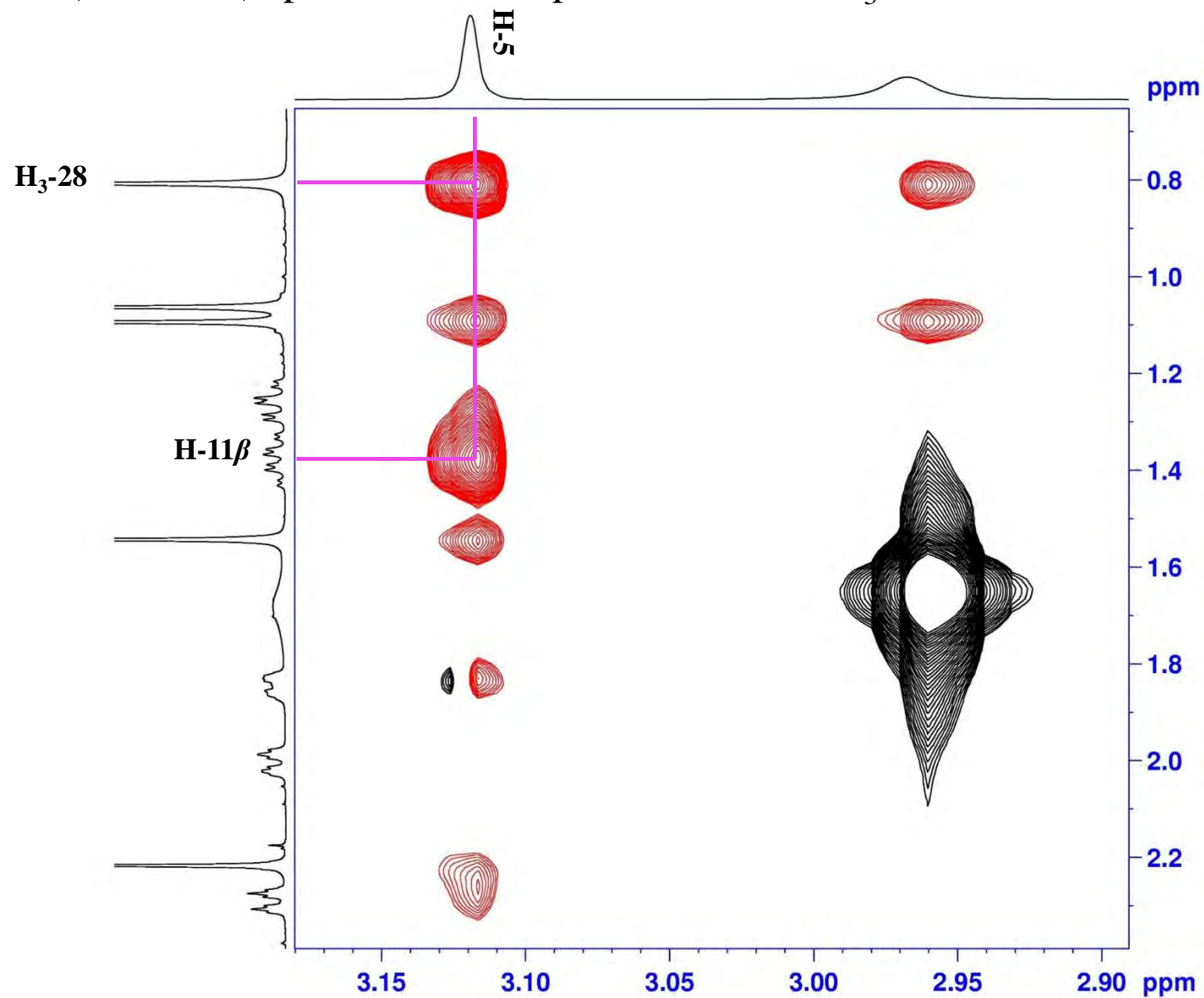
NOESY (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **10** in CDCl<sub>3</sub>

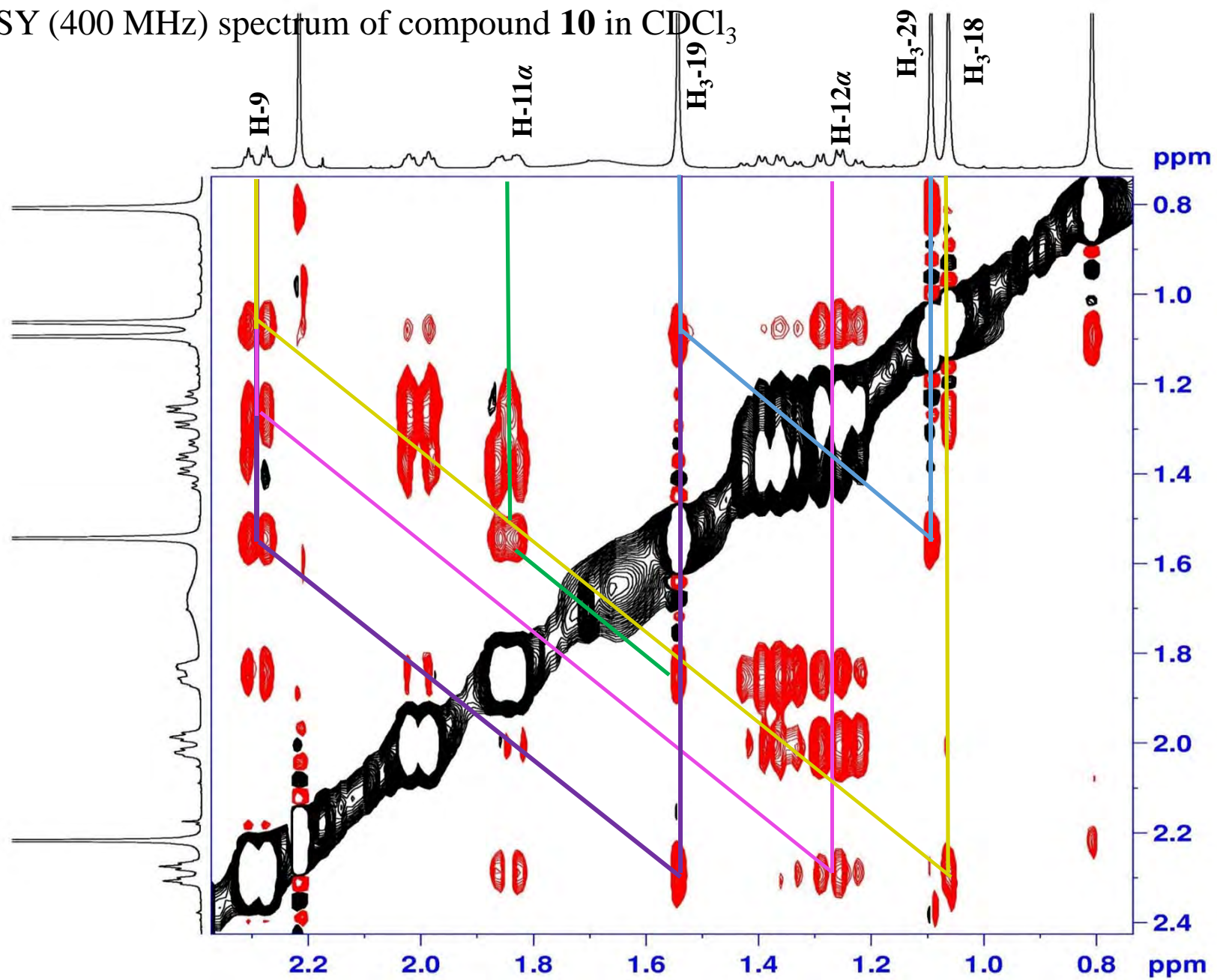


NOESY (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$





NOESY (400 MHz) spectrum of compound **10** in  $\text{CDCl}_3$



# HRESIMS for compound 11

## Mass Spectrum SmartFormula Report

### Analysis Info

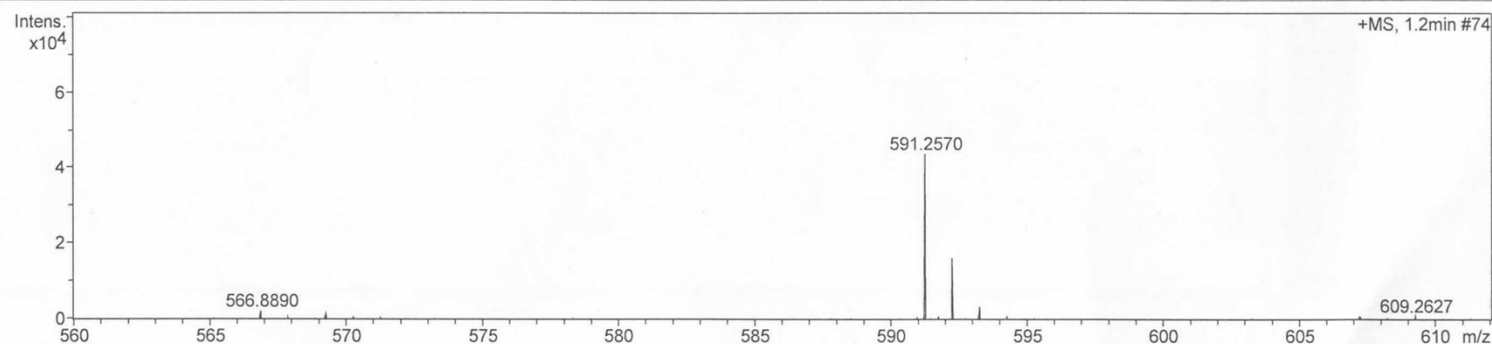
Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-31\_pos\_82\_01\_2758.d  
Method LC MS\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name zhangjianzhi\_zjz-31\_pos  
Comment

Acquisition Date 12/14/2016 2:48:30 PM

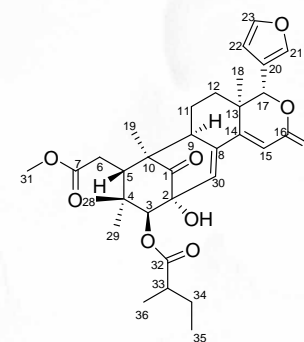
Operator SCSIO  
Instrument maXis 255552.00029

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
569.274225	1	C32H41O9	100.00	569.274509	-0.5	-0.3	42.9	12.5	even	ok
591.256980	1	C32H40NaO9	100.00	591.256454	0.9	0.5	7.3	12.5	even	ok
1159.523732	1	C64H80NaO18	100.00	1159.523686	0.0	0.0	48.1	24.5	even	ok





# HRESIMS for compound 11

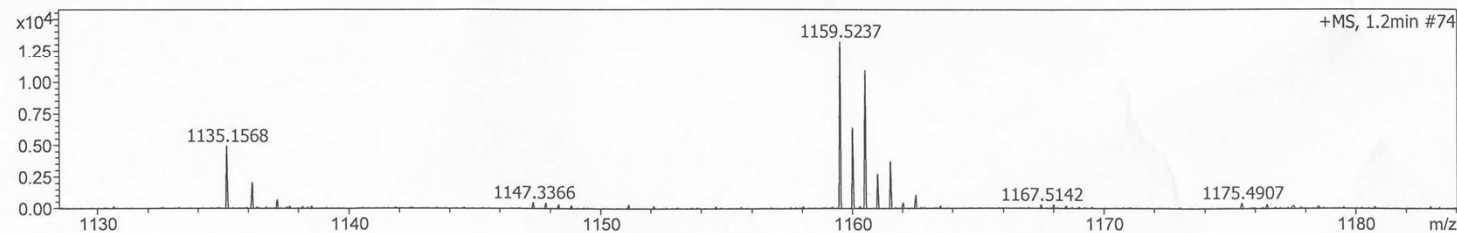
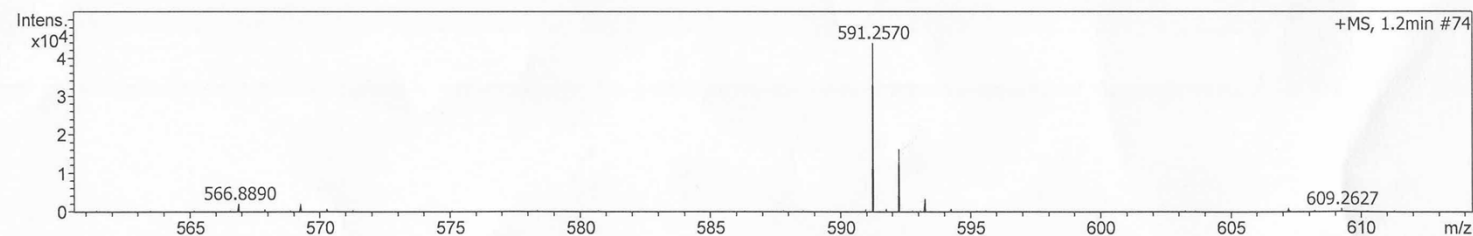
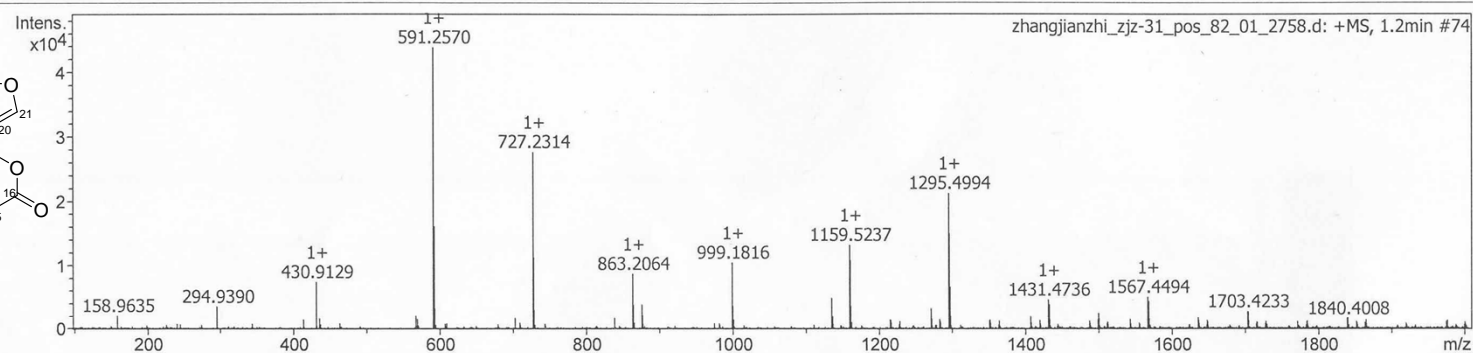
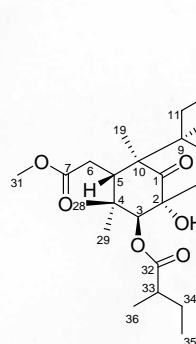
## Generic Display Report

### Analysis Info

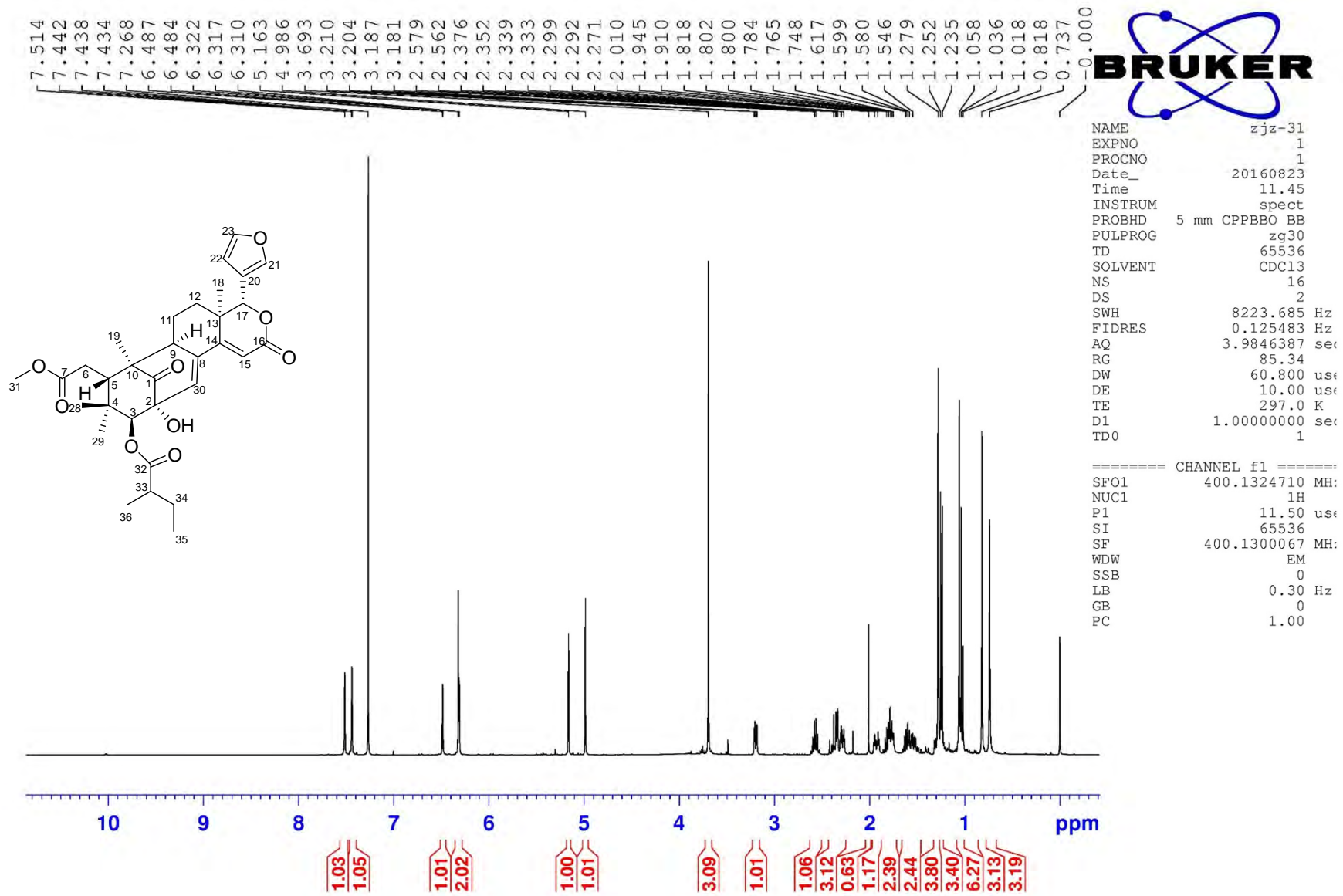
Analysis Name D:\Data\MS\data\201612\zhangjianzhi\_zjz-31\_pos\_82\_01\_2758.d  
Method LC\_MS\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name zhangjianzhi\_zjz-31\_pos  
Comment

Acquisition Date 12/14/2016 2:48:30 PM

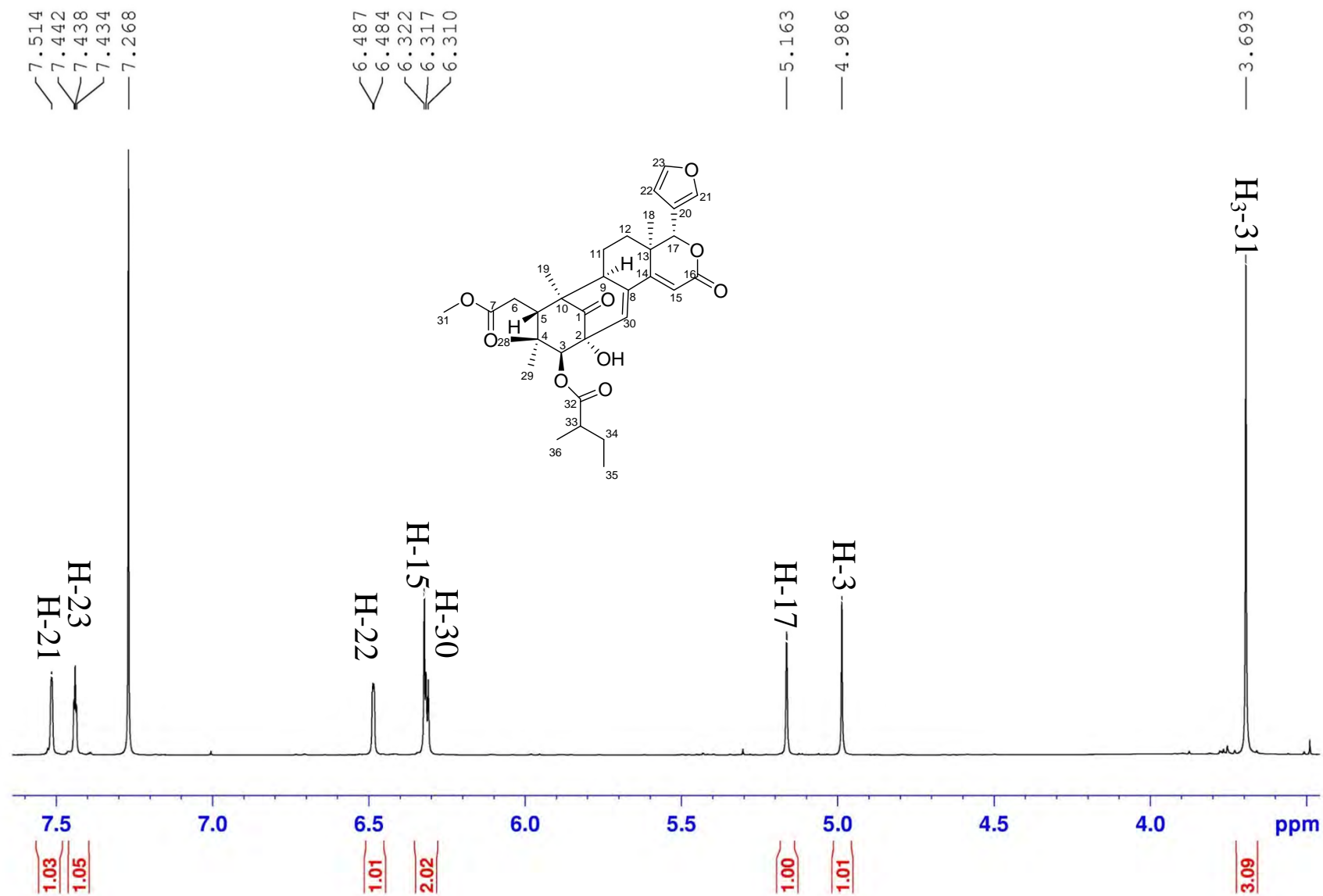
Operator SCSIO  
Instrument maXis



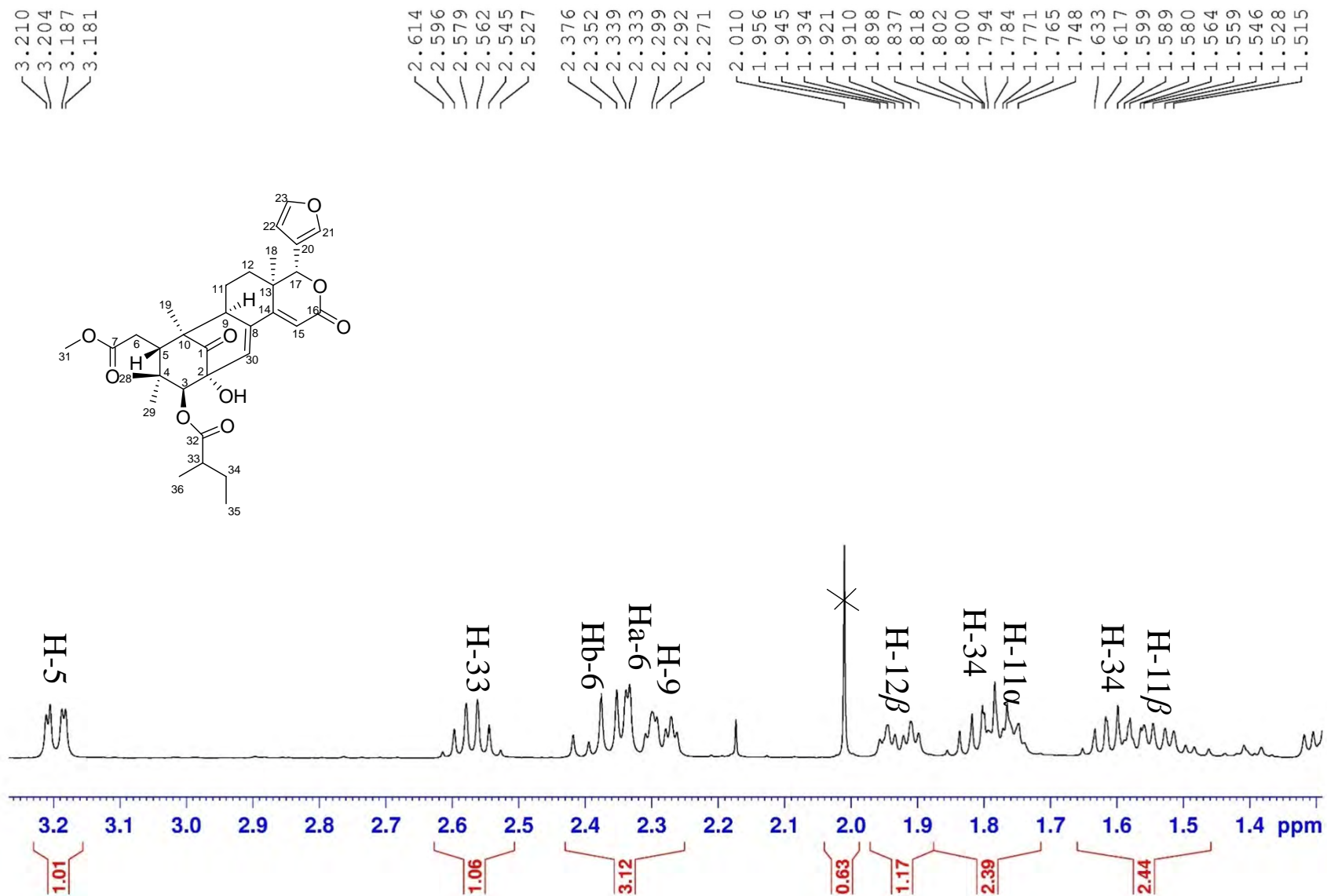
<sup>1</sup>H NMR (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



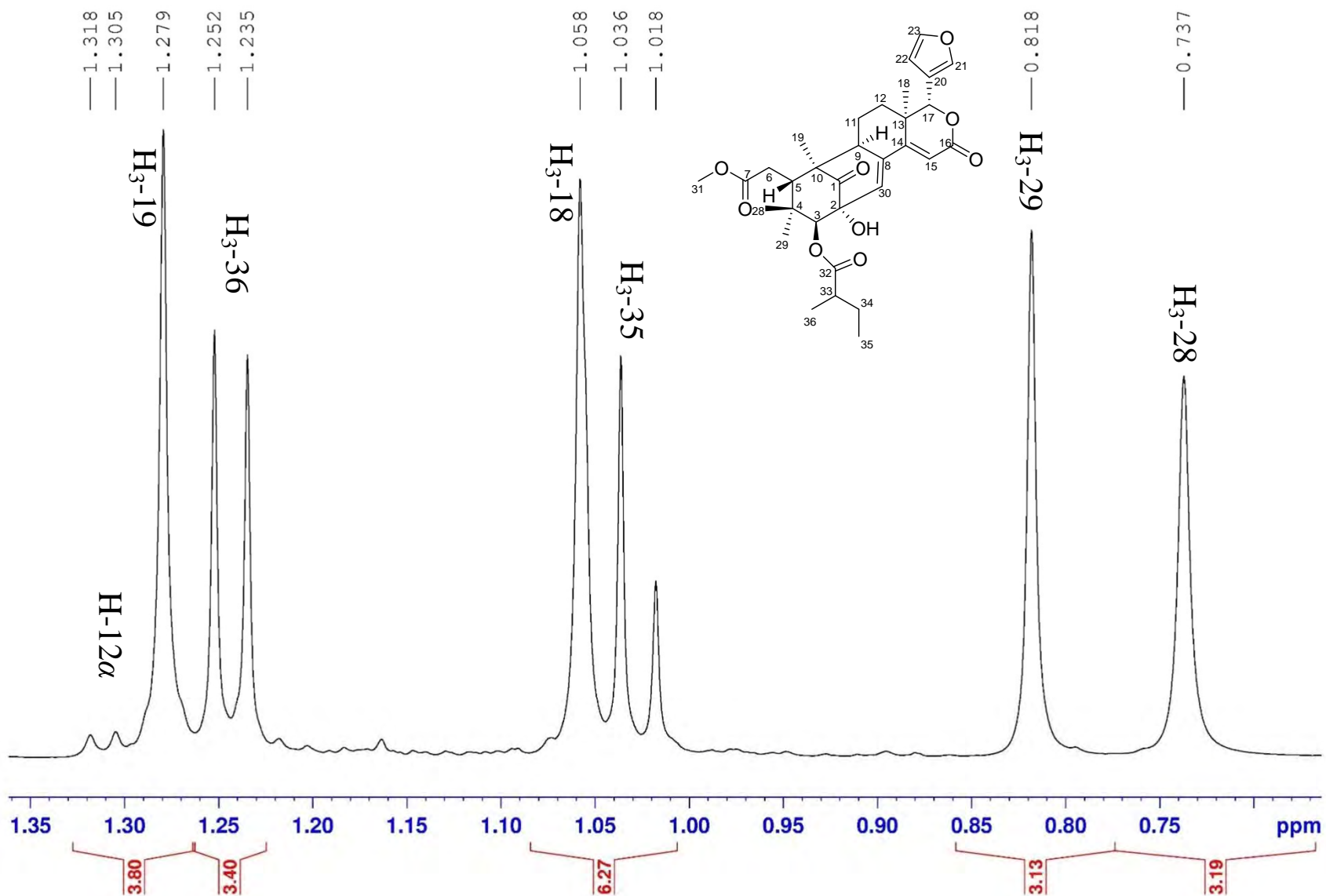
$^1\text{H}$  NMR (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



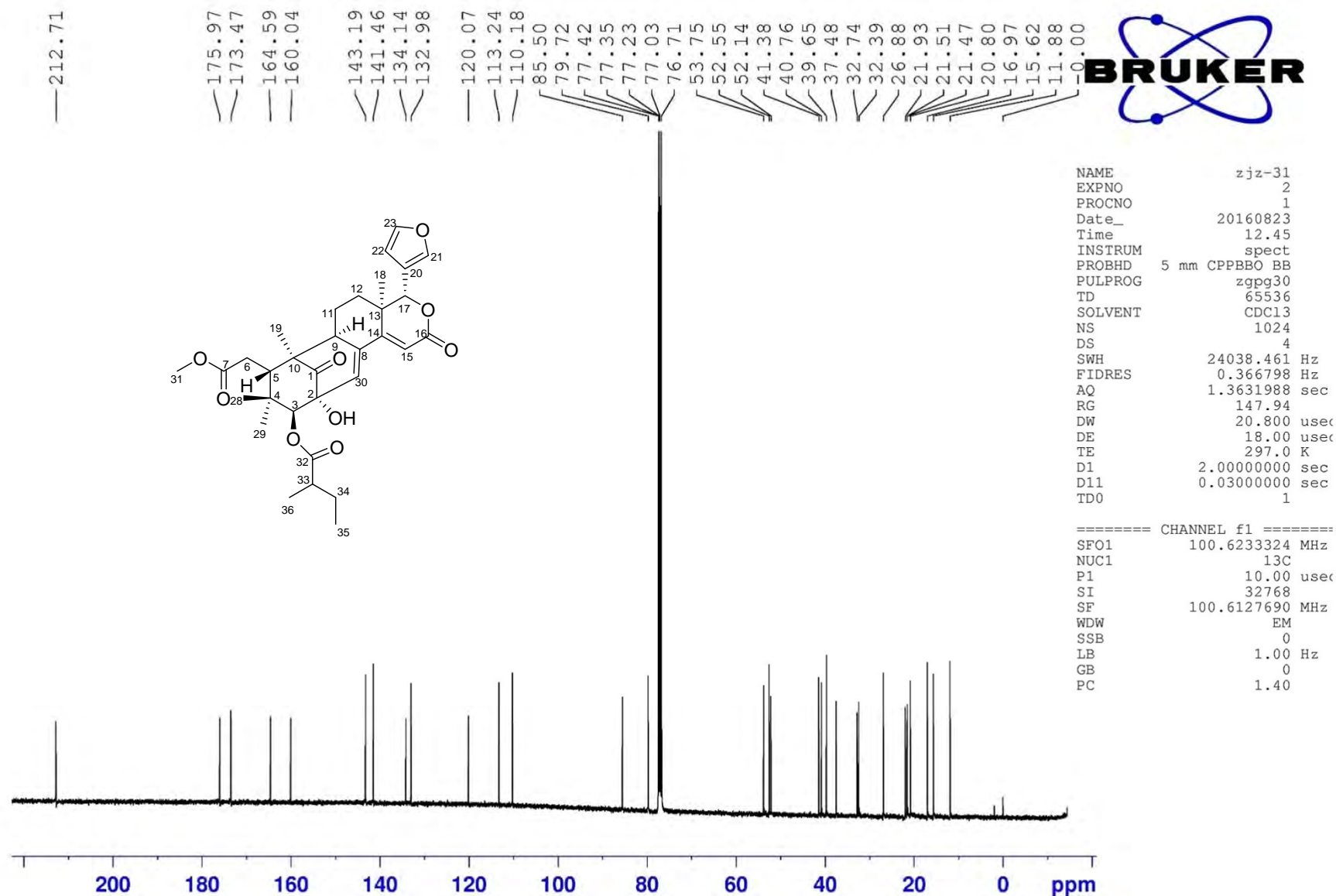
$^1\text{H}$  NMR (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

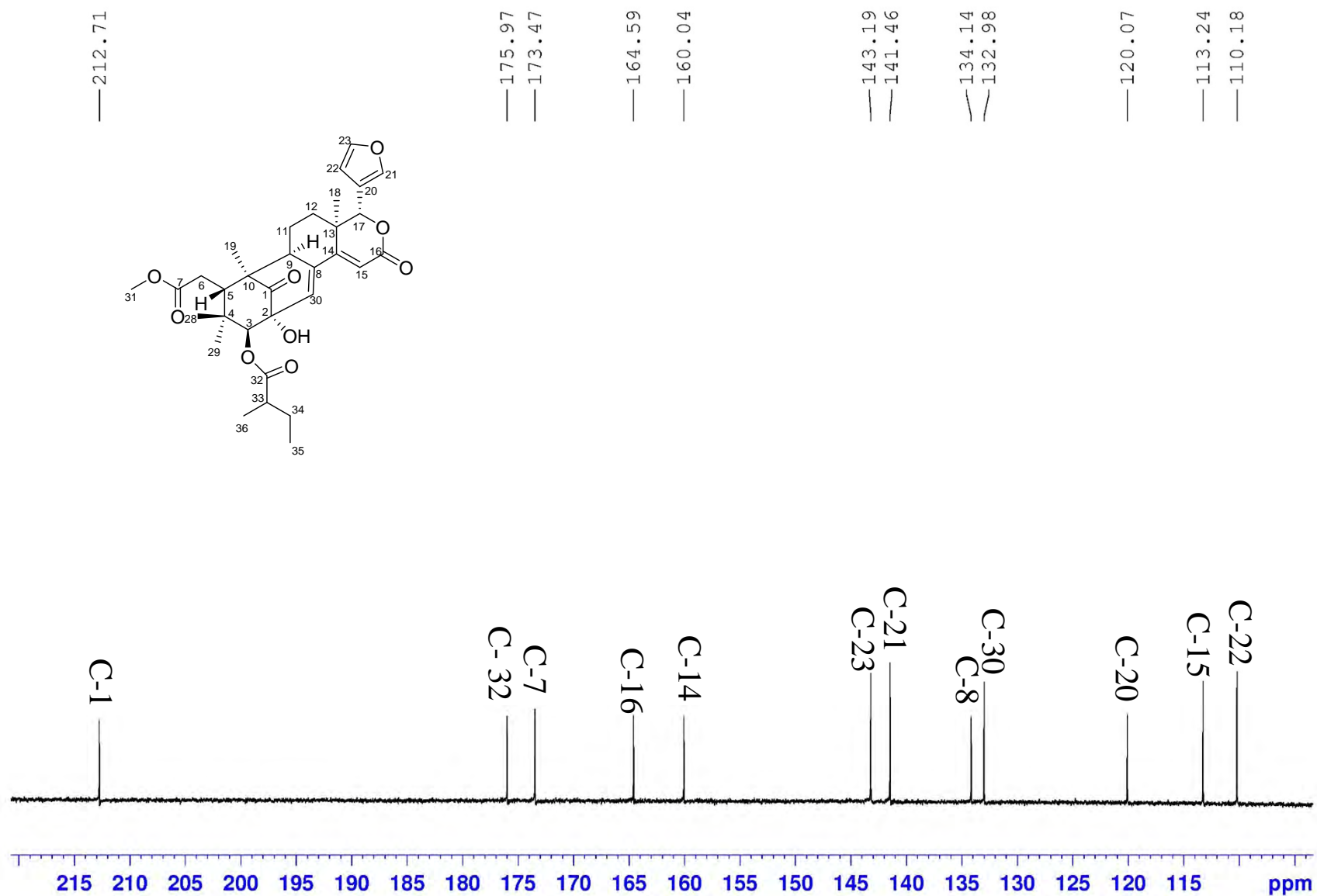


$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

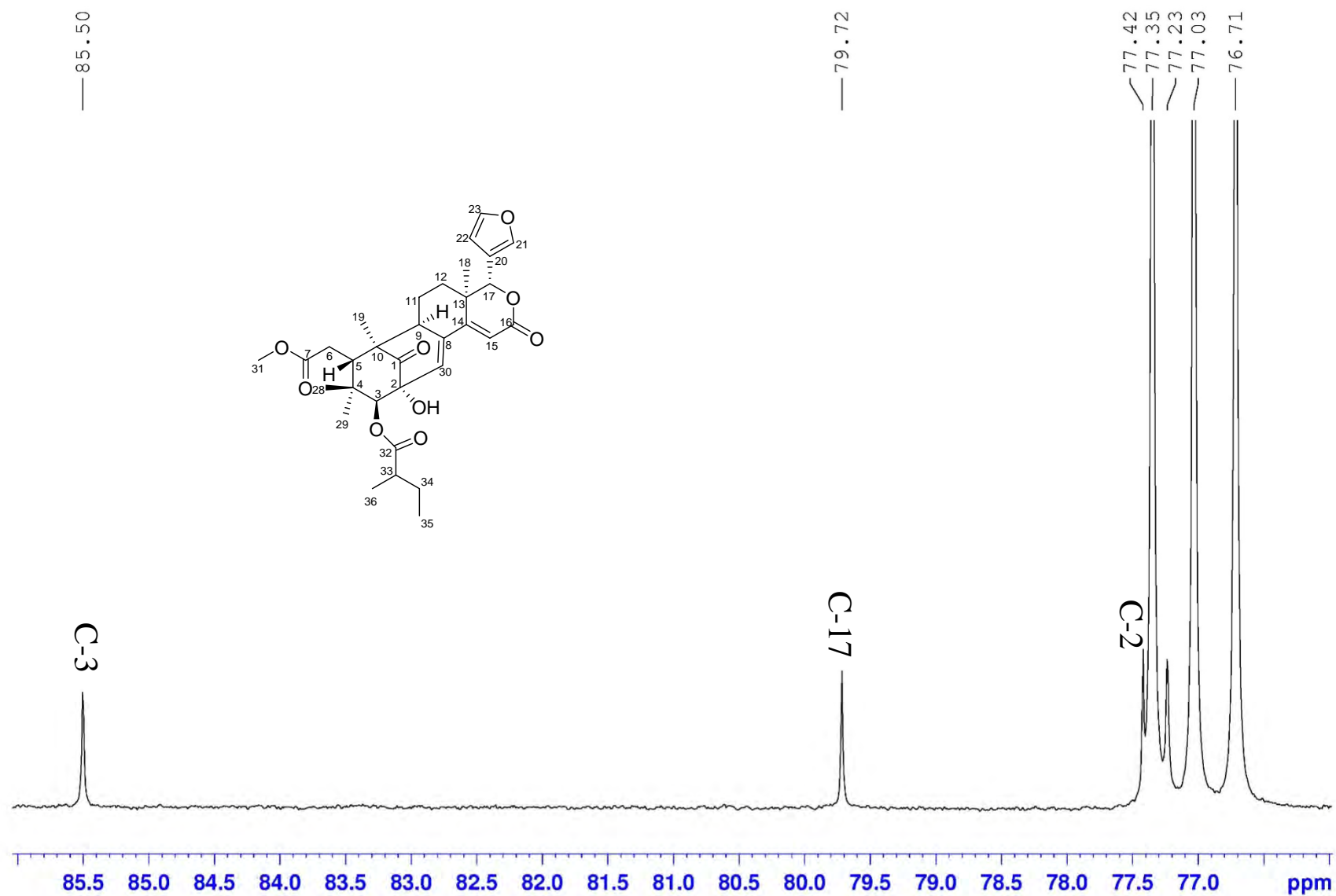




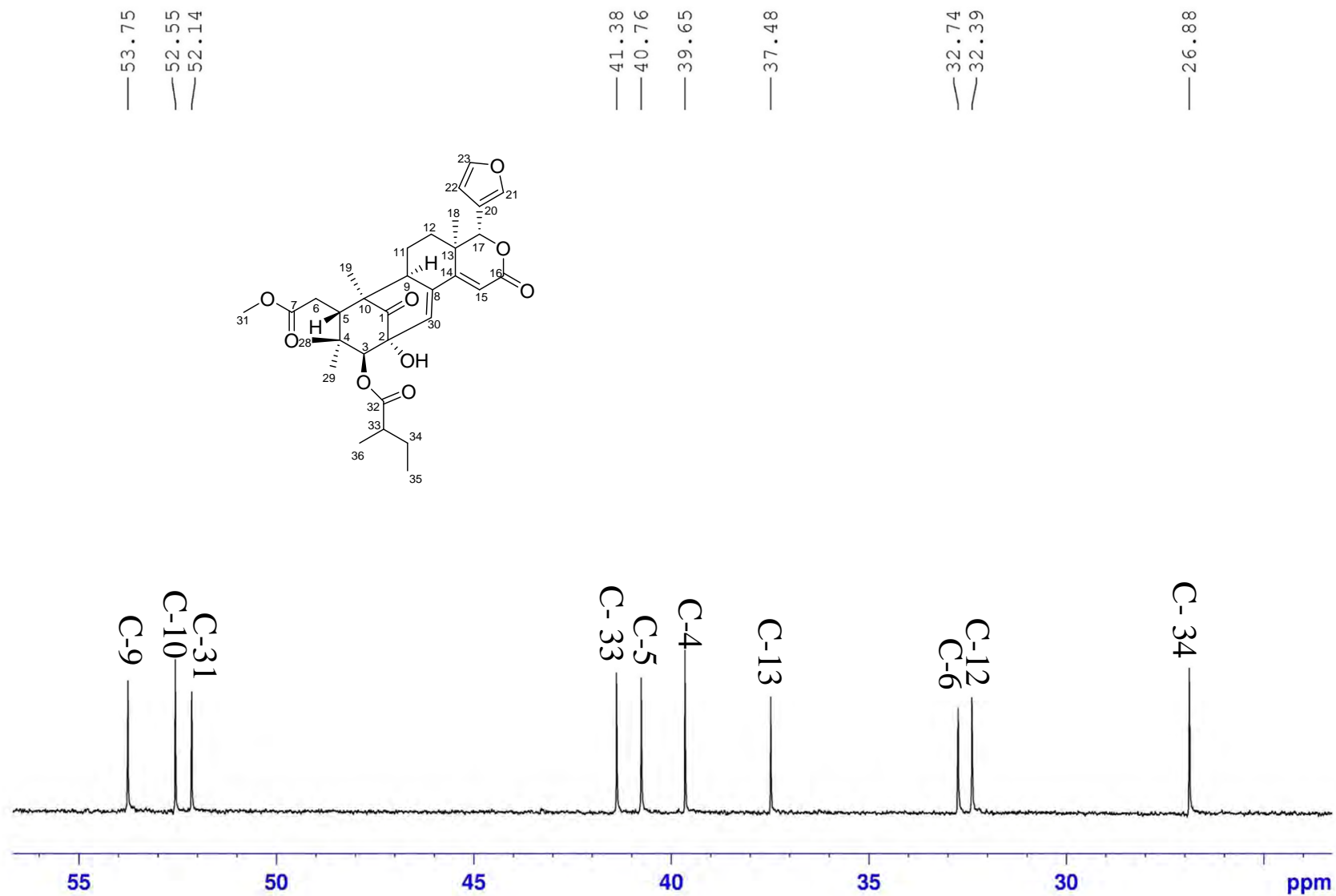
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



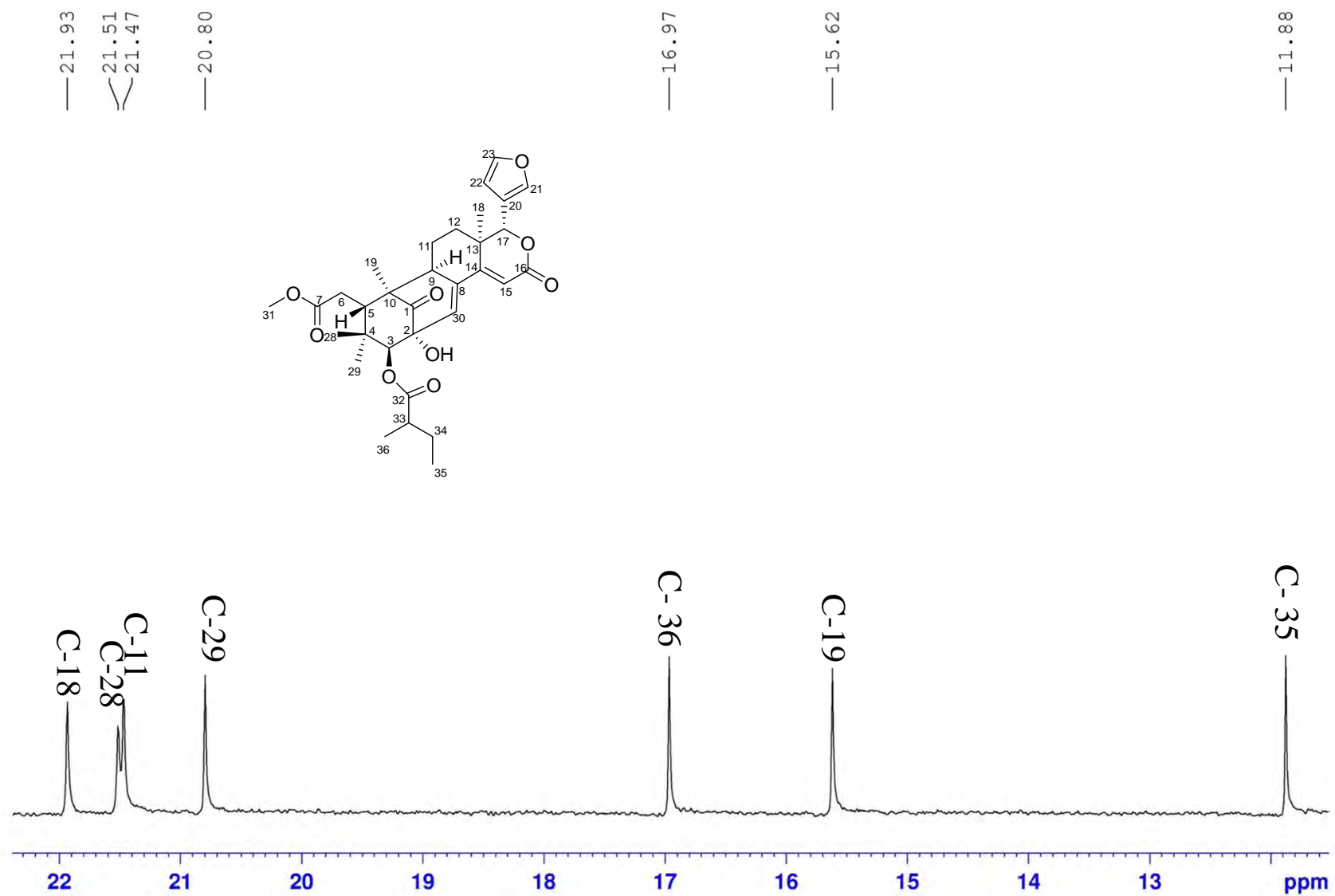
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



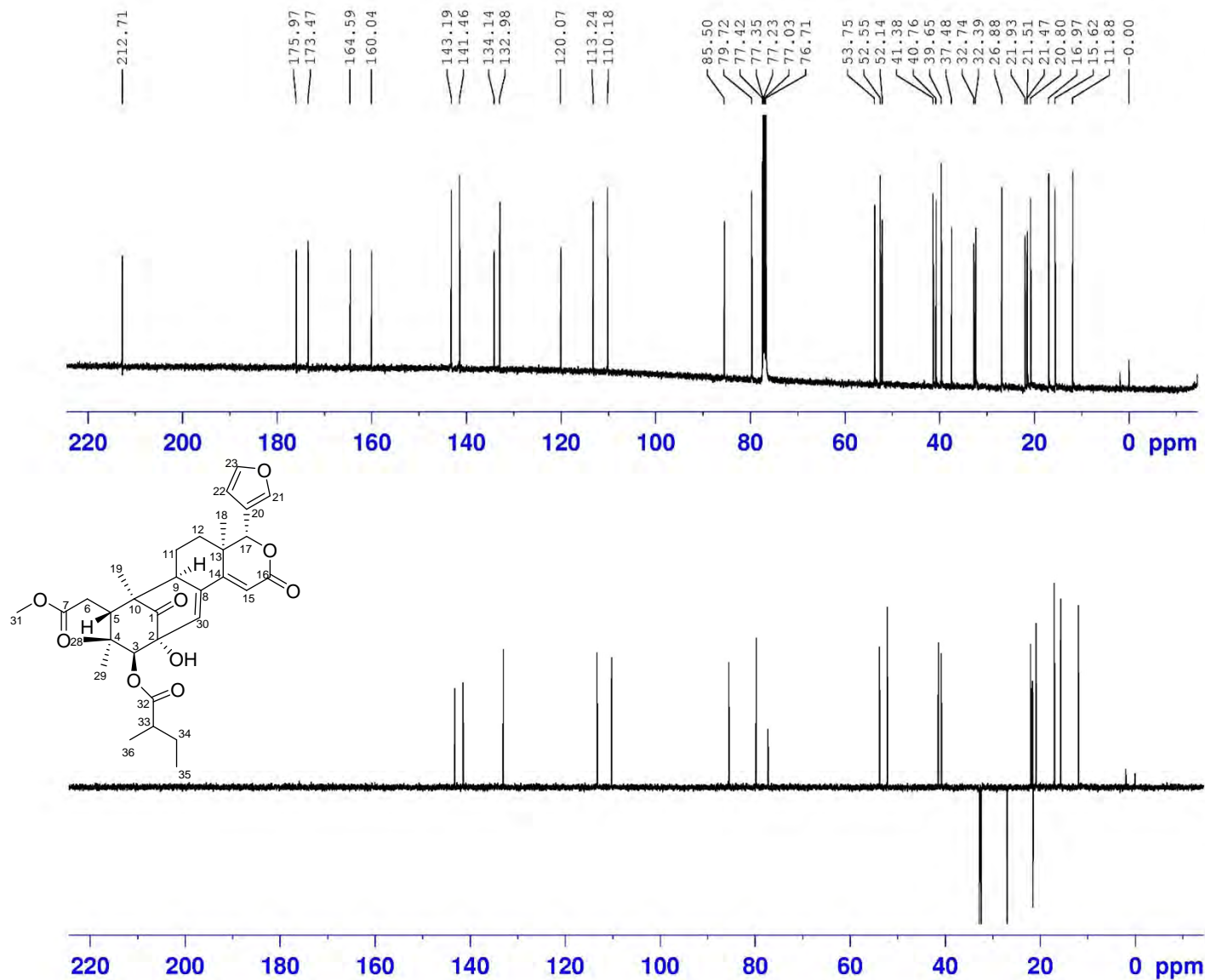
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



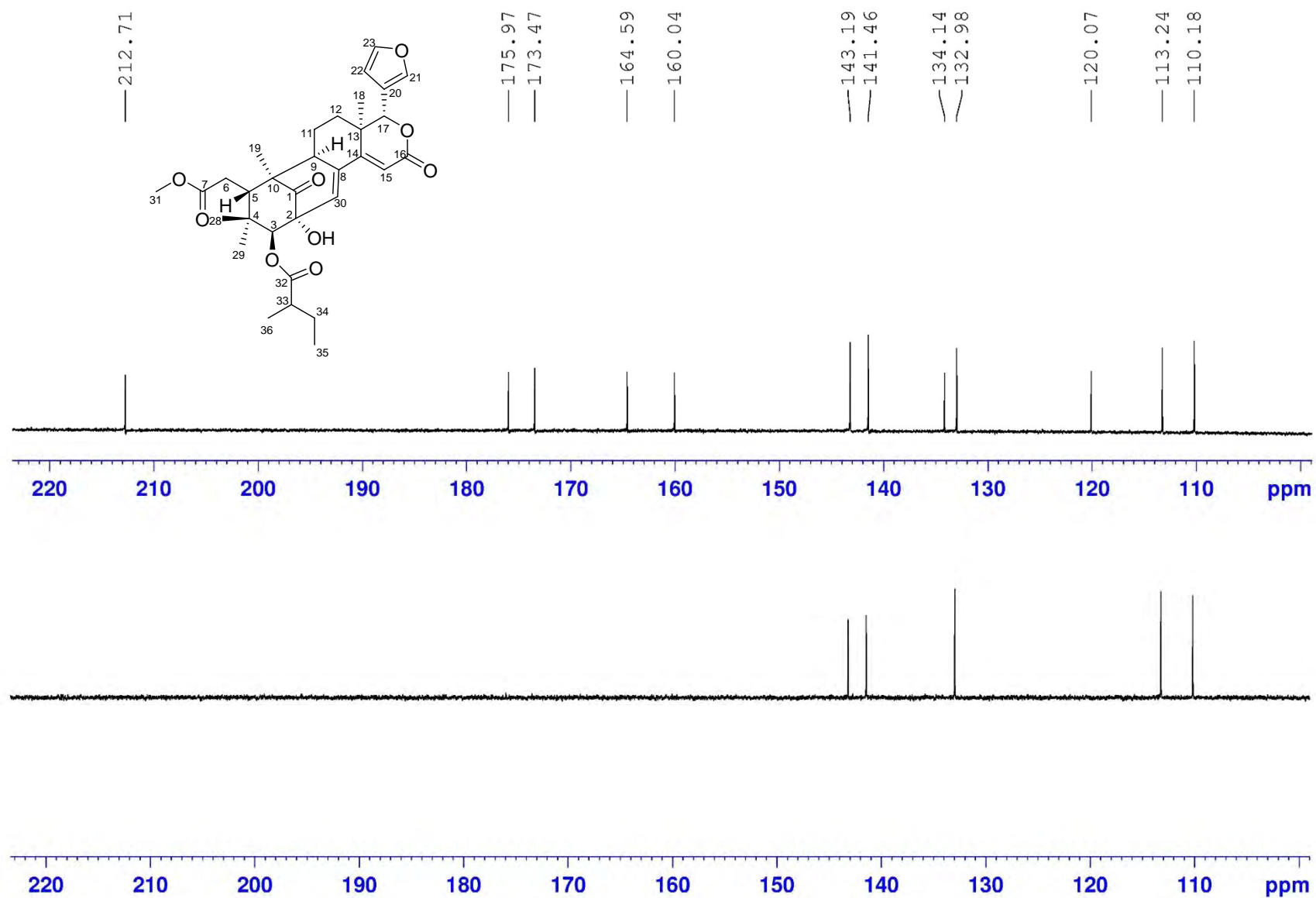
# DEPT135 (100 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



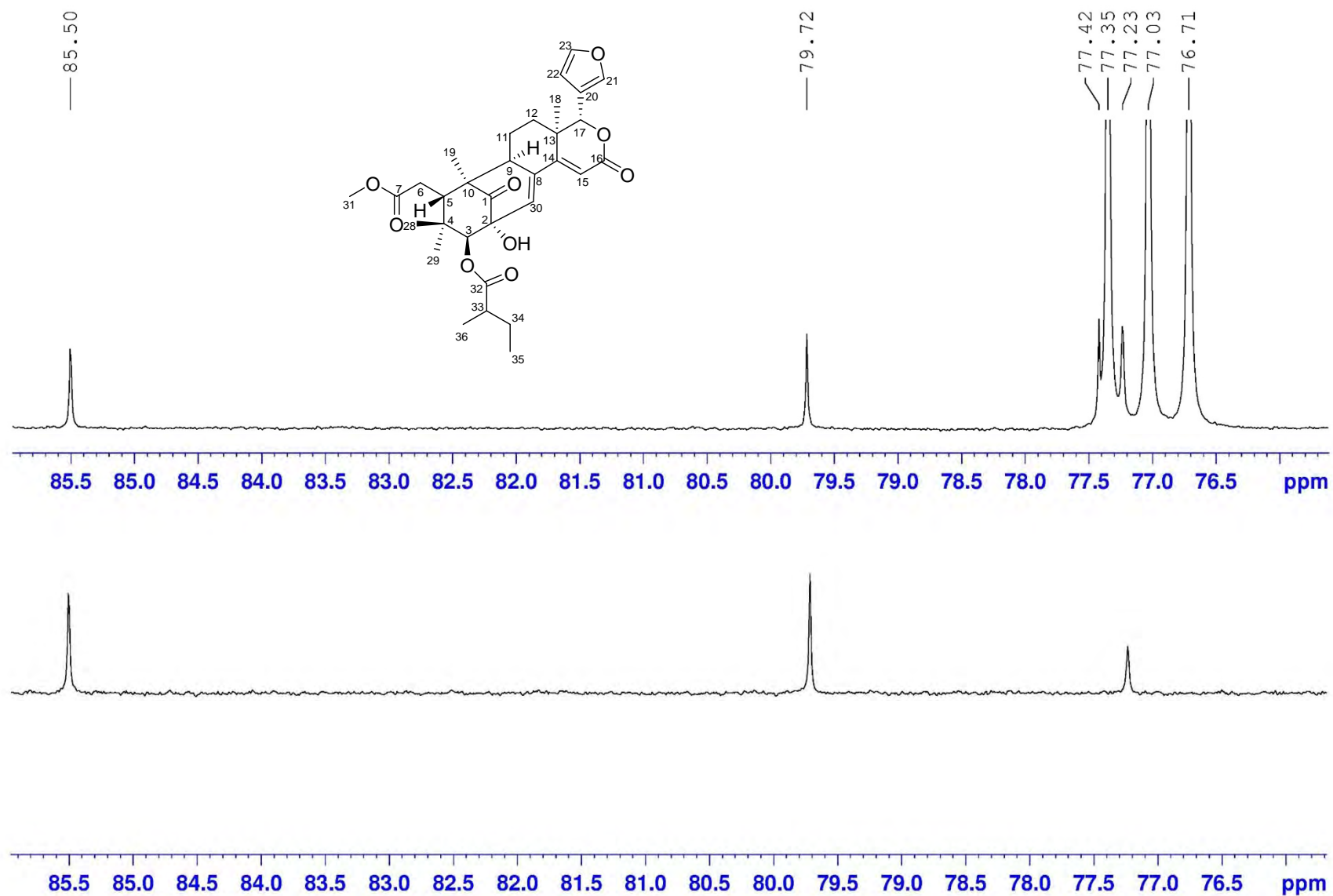
NAME zjz-31  
 EXPNO 3  
 PROCNO 1  
 Date\_ 20160823  
 Time 13.03  
 INSTRUM spect  
 PROBHD 5 mm CPMBBO BB  
 PULPROG deptsp135  
 TD 65536  
 SOLVENT CDCl3  
 NS 300  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 130.26  
 DW 20.800 usec  
 DE 18.00 usec  
 TE 297.0 K  
 CNST2 145.0000000  
 D1 2.00000000 sec  
 D2 0.00344828 sec  
 D12 0.00002000 sec  
 TD0 1

===== CHANNEL f1 =====  
 SF01 100.6233324 MHz  
 NUC1 13C  
 P1 10.00 usec  
 P13 2000.00 usec  
 SI 32768  
 SF 100.6127690 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

DEPT135 (100 MHz) spectrum of compound **11** in CDCl<sub>3</sub>

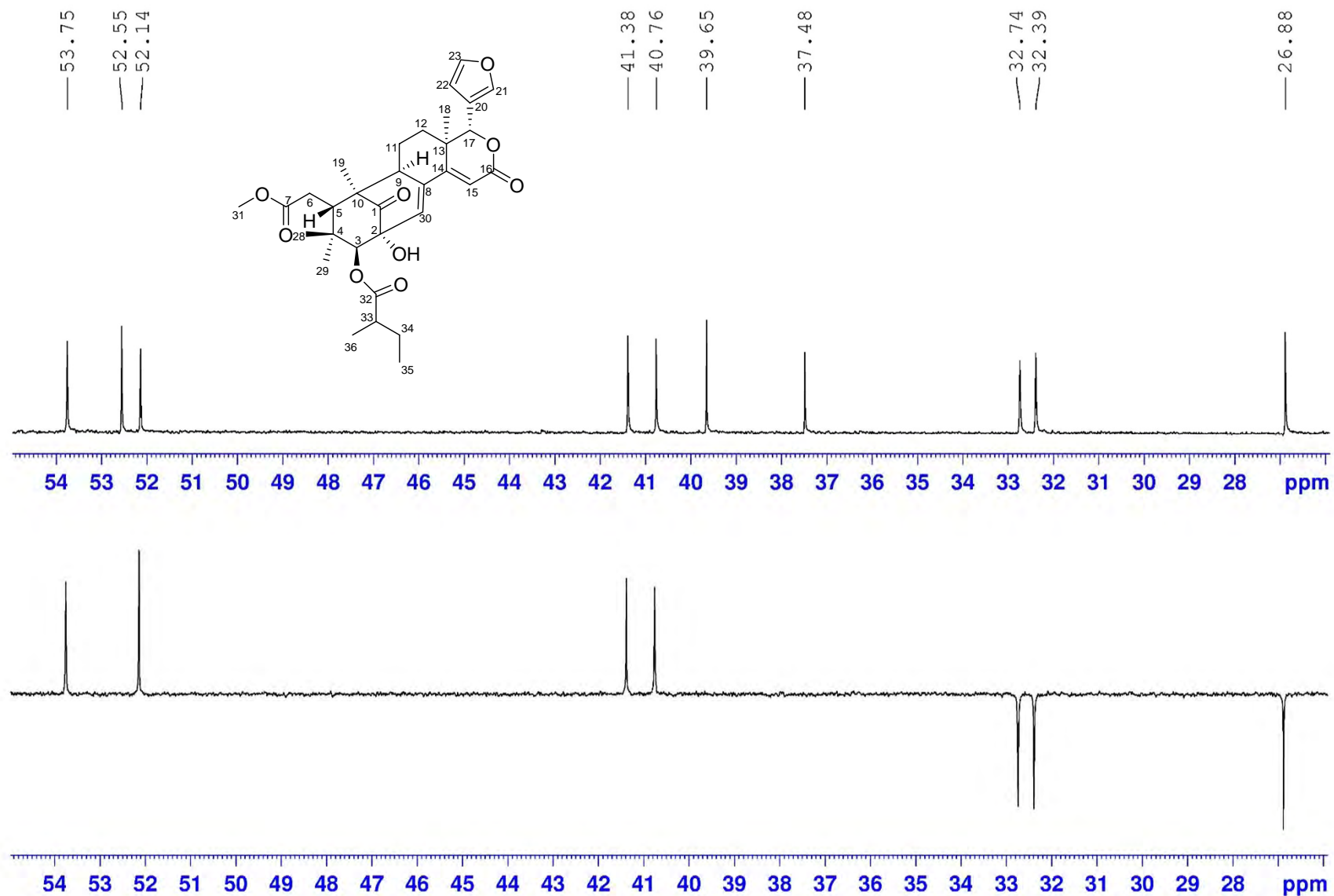


DEPT135 (100 MHz) spectrum of compound **11** in CDCl<sub>3</sub>

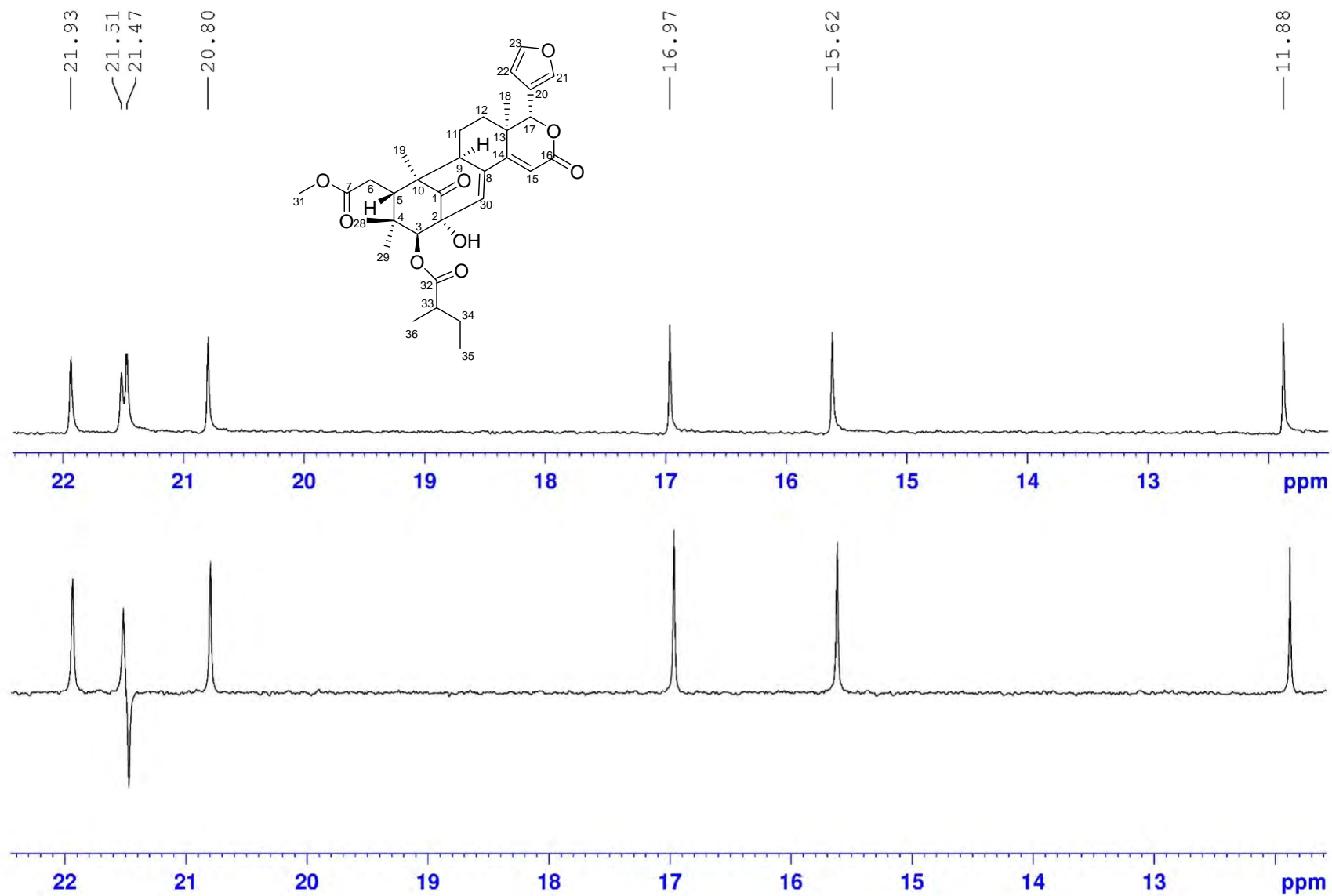




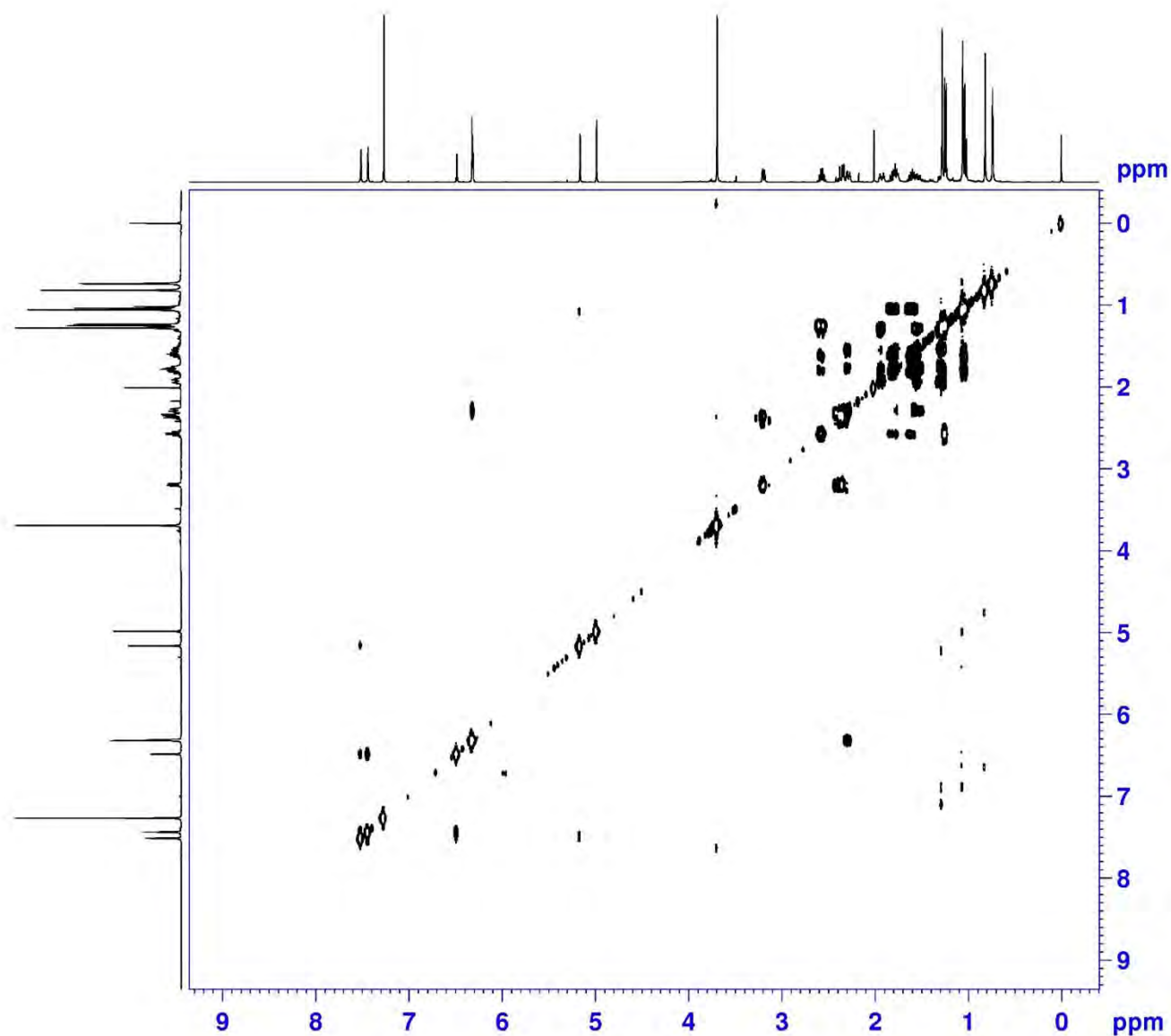
DEPT135 (100 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



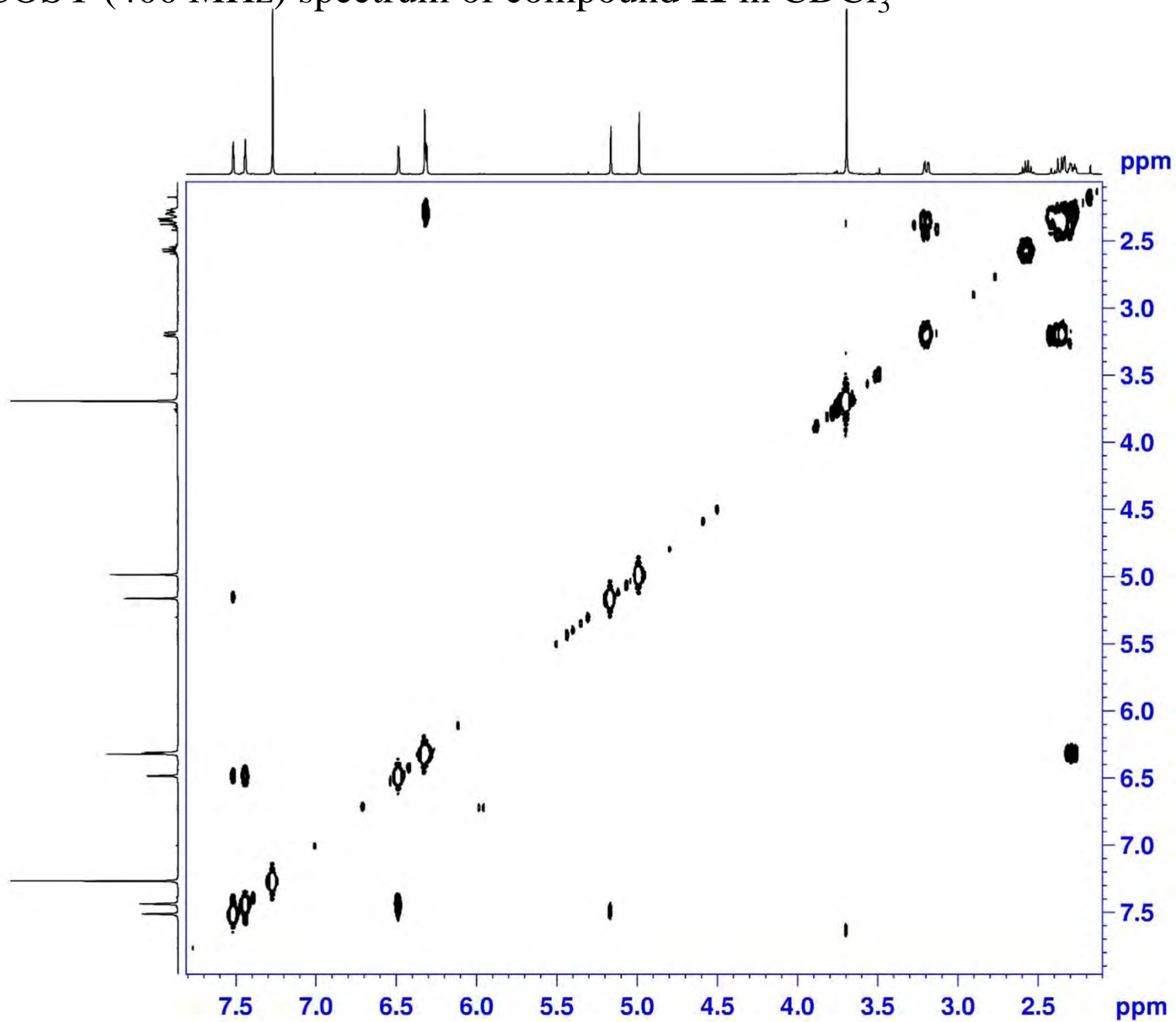
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



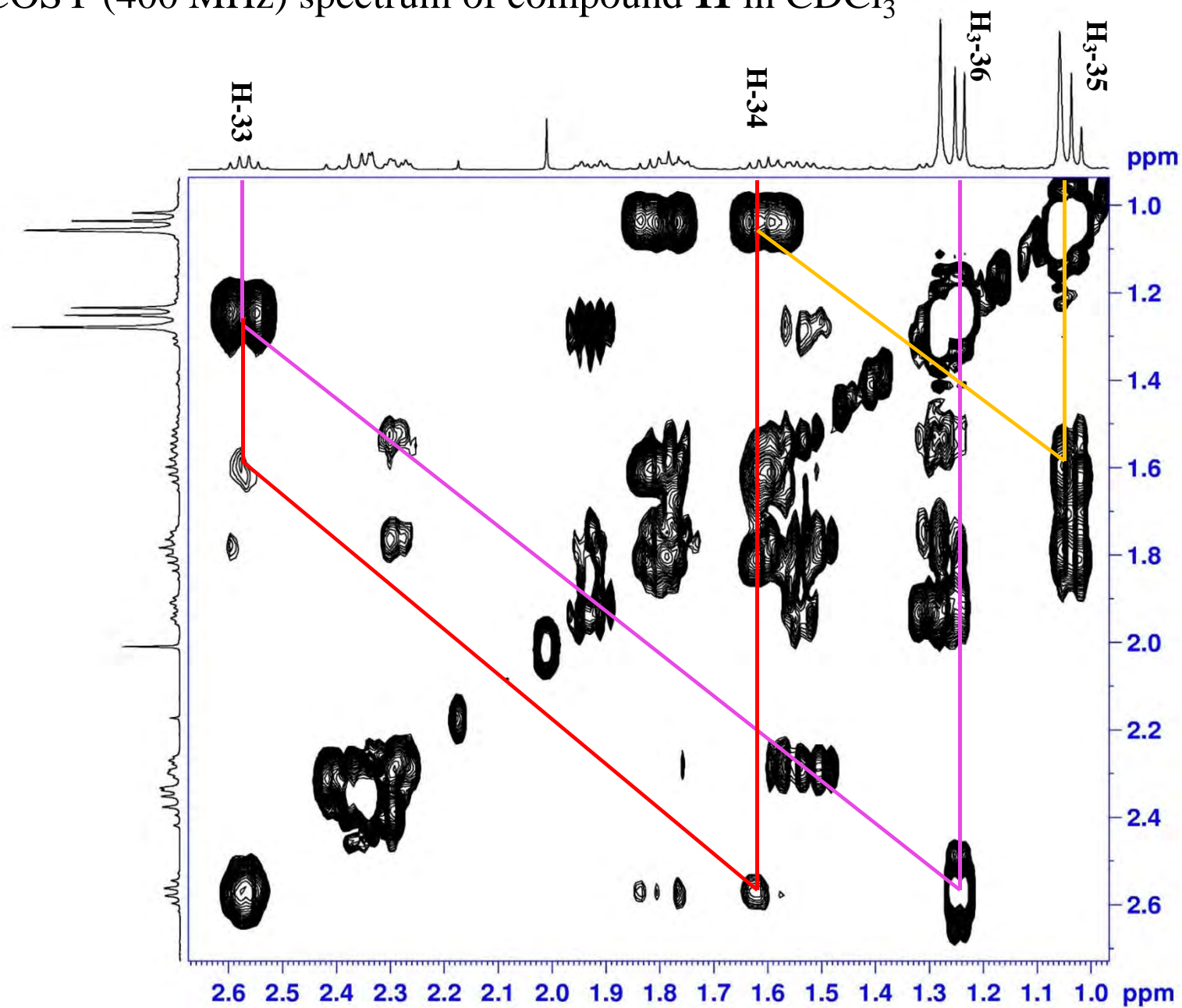
NAME zjz-31  
EXPNO 4  
PROCNO 1  
Date\_ 20160824  
Time 16.31  
INSTRUM spect  
PROBHD 5 mm CFPBBO BB  
PULPROG cosygpppqf  
TD 2048  
SOLVENT  $\text{CDCl}_3$   
NS 8  
DS 8  
SWH 3906.250 Hz  
FIDRES 1.907349 Hz  
AQ 0.2621940 sec  
RG 208.5  
DW 128.000 usec  
DE 10.00 usec  
TE 297.0 K  
D0 0.00000300 sec  
D1 1.89678097 sec  
D11 0.03000000 sec  
D12 0.00002000 sec  
D13 0.00000400 sec  
D16 0.00020000 sec  
IN0 0.00025600 sec

===== CHANNEL f1 =====  
SFO1 400.1318006 MHz  
NUC1  $^1\text{H}$   
P0 11.50 usec  
P1 11.50 usec  
P17 2500.00 usec  
ND0 1  
TD 128  
SFO1 400.1318 MHz  
FIDRES 30.517578 Hz  
SW 9.762 ppm  
FnMODE QF  
SI 1024  
SF 400.1300049 MHz  
WDW QSINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40  
SI 1024  
MC2 QF  
SF 400.1300044 MHz  
WDW QSINE  
SSB 0  
LB 0.00 Hz  
GB 0

$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

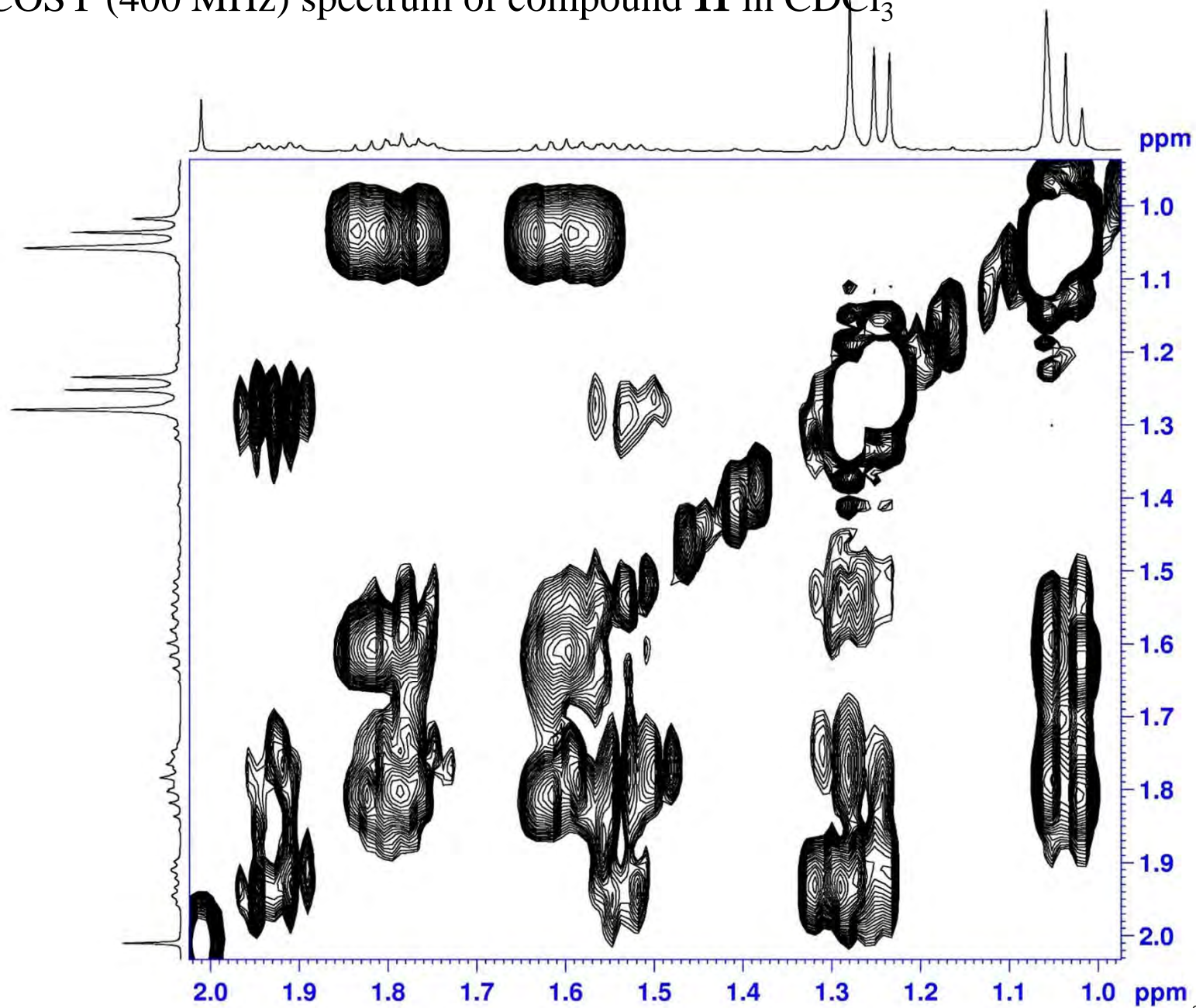


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

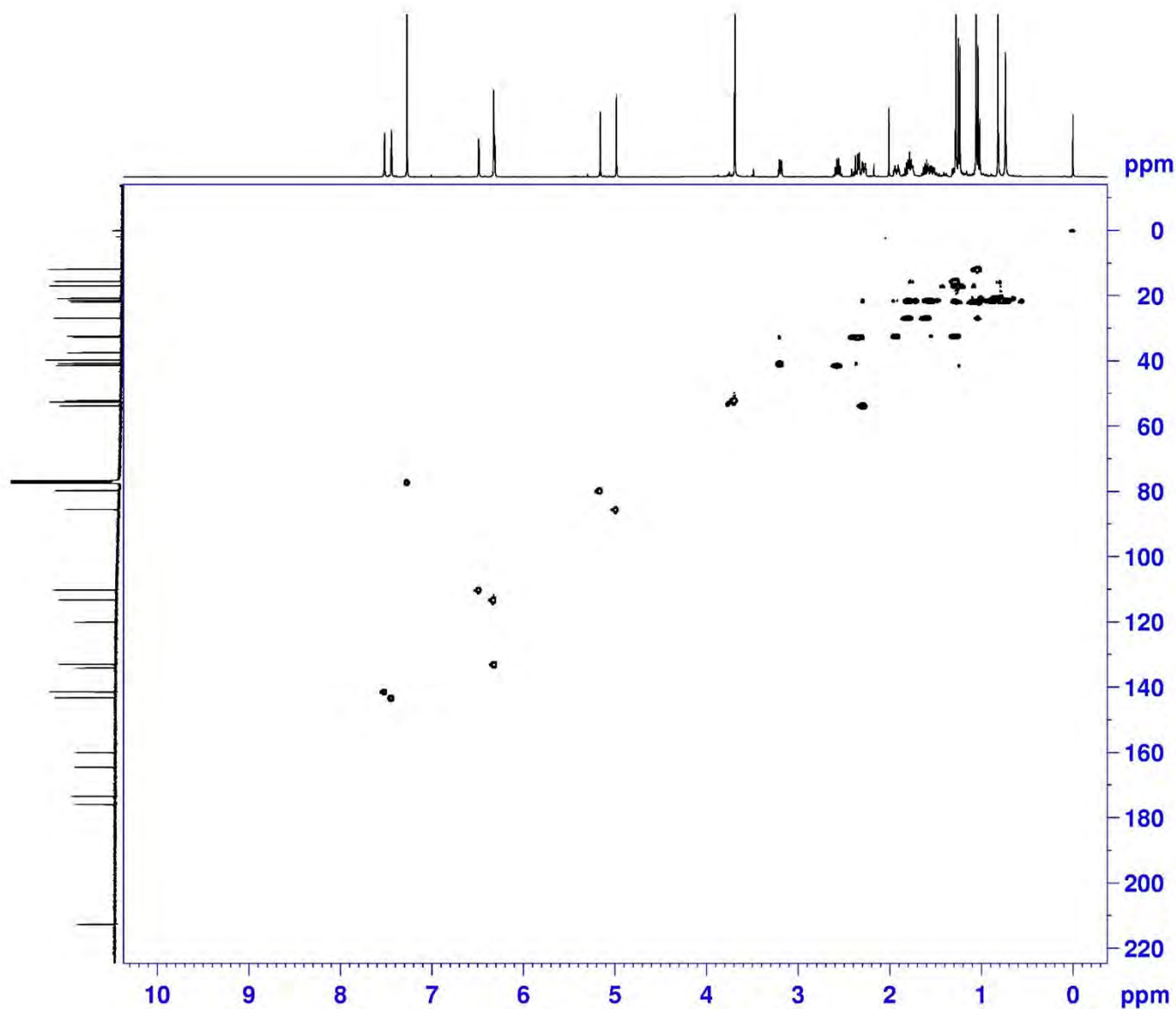




$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



# HSQC (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



```

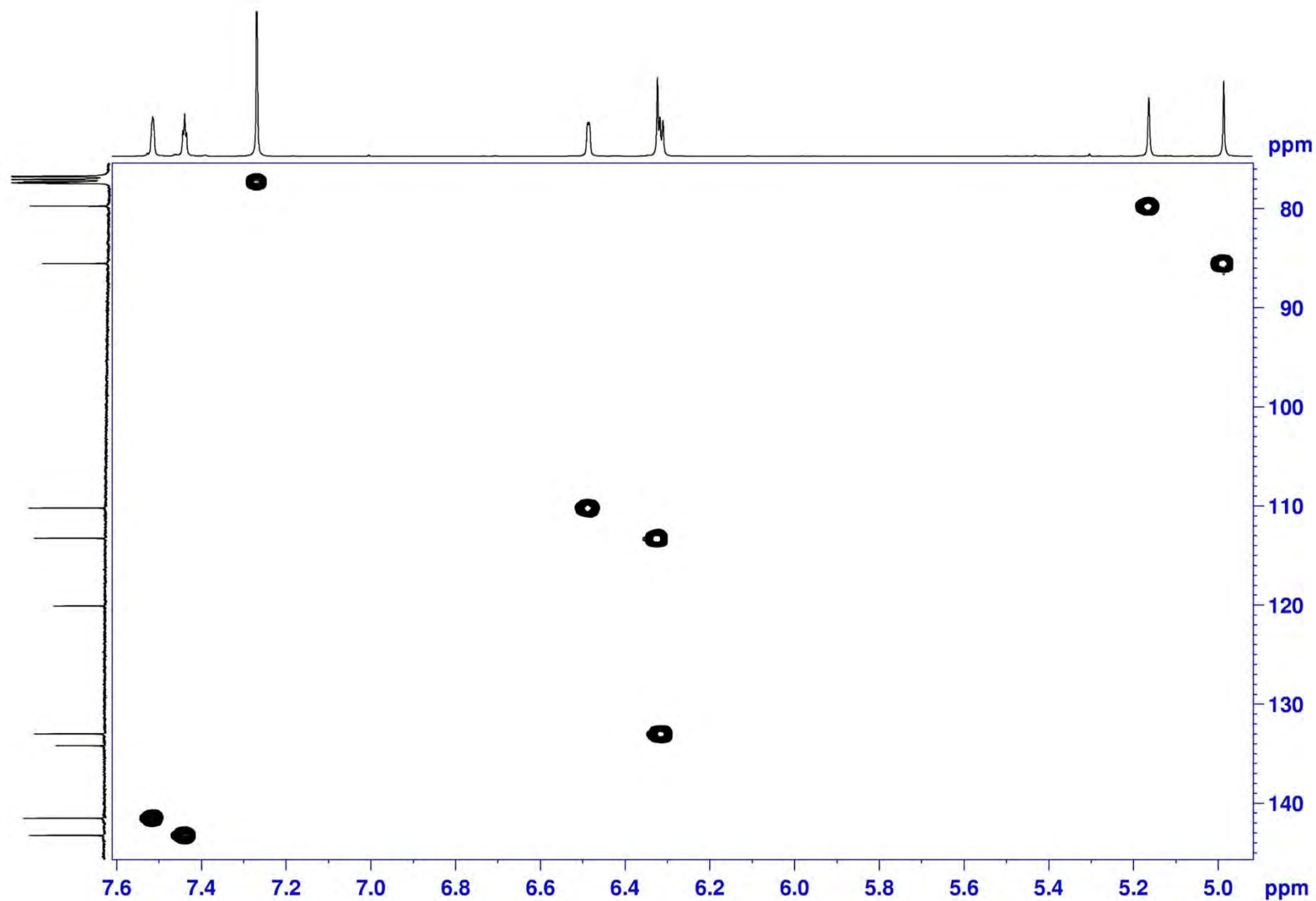
NAME          zjz-31
EXPNO          5
PROCNO         1
Date_         20160824
Time          17.11
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS             16
DS             16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 sec
RG            208.5
DW            116.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
D0            0.00000300 sec
D1            1.46497905 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00002080 sec
ZGPTNS
  
```

```

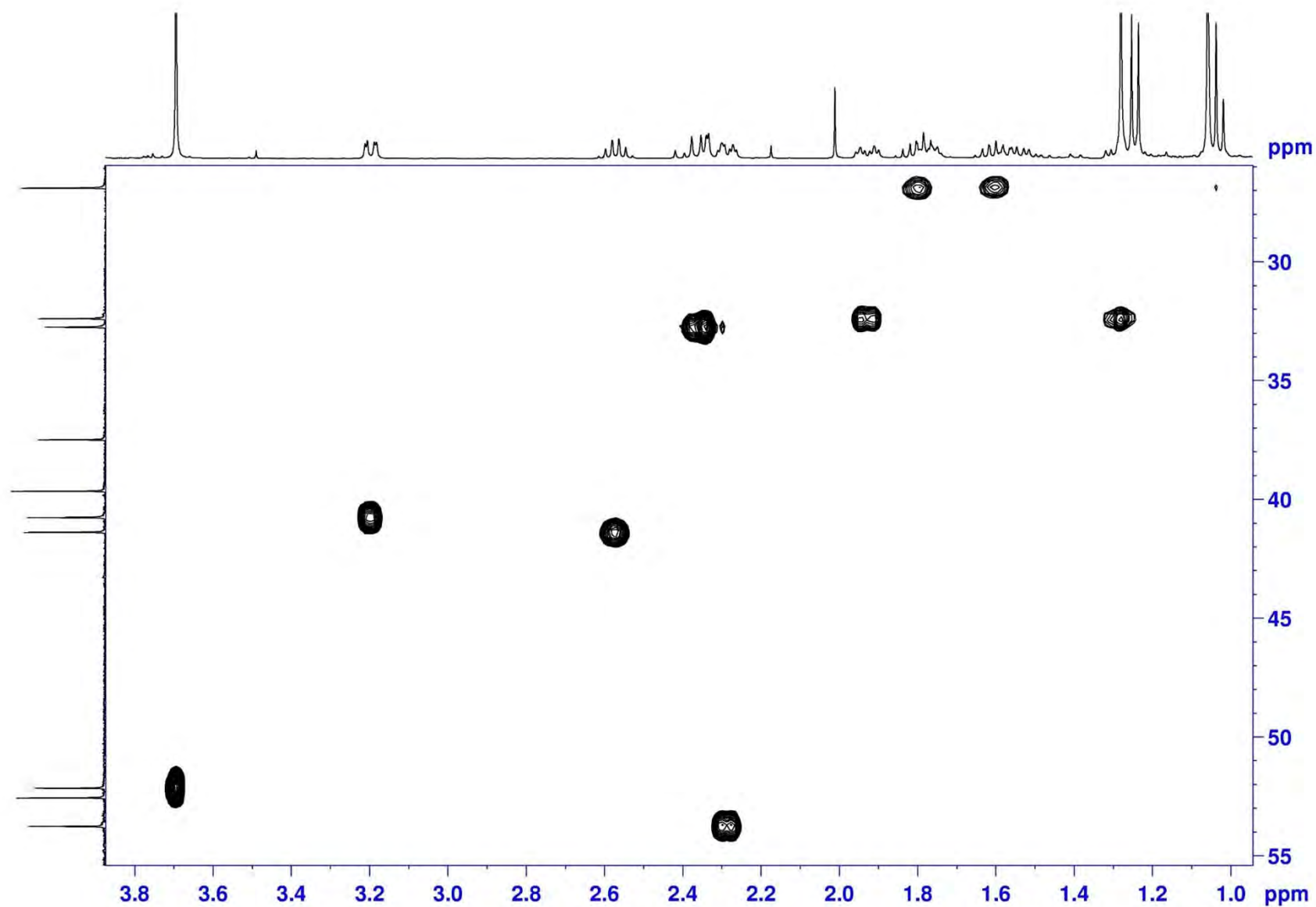
===== CHANNEL f1 =====
SF01         400.1320007 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
P28           0.00 usec
ND0           2
TD            256
SF01         100.6233 MHz
FIDRES        93.900238 Hz
SW            238.896 ppm
FnMODE       Echo-Antiecho
SI            1024
SF            400.1300041 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2          echo-antiecho
SF            100.6127544 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
  
```



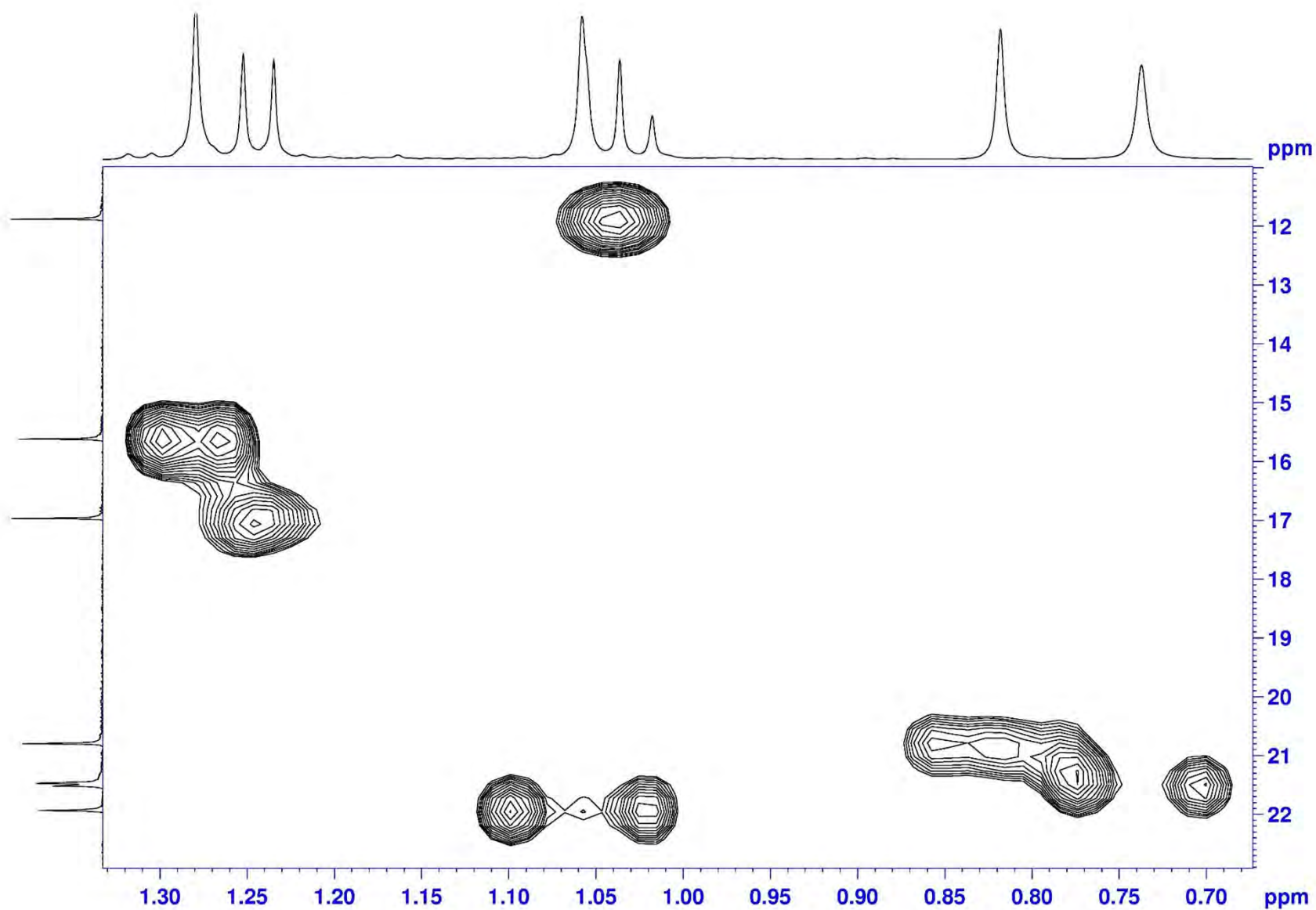
HSQC (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



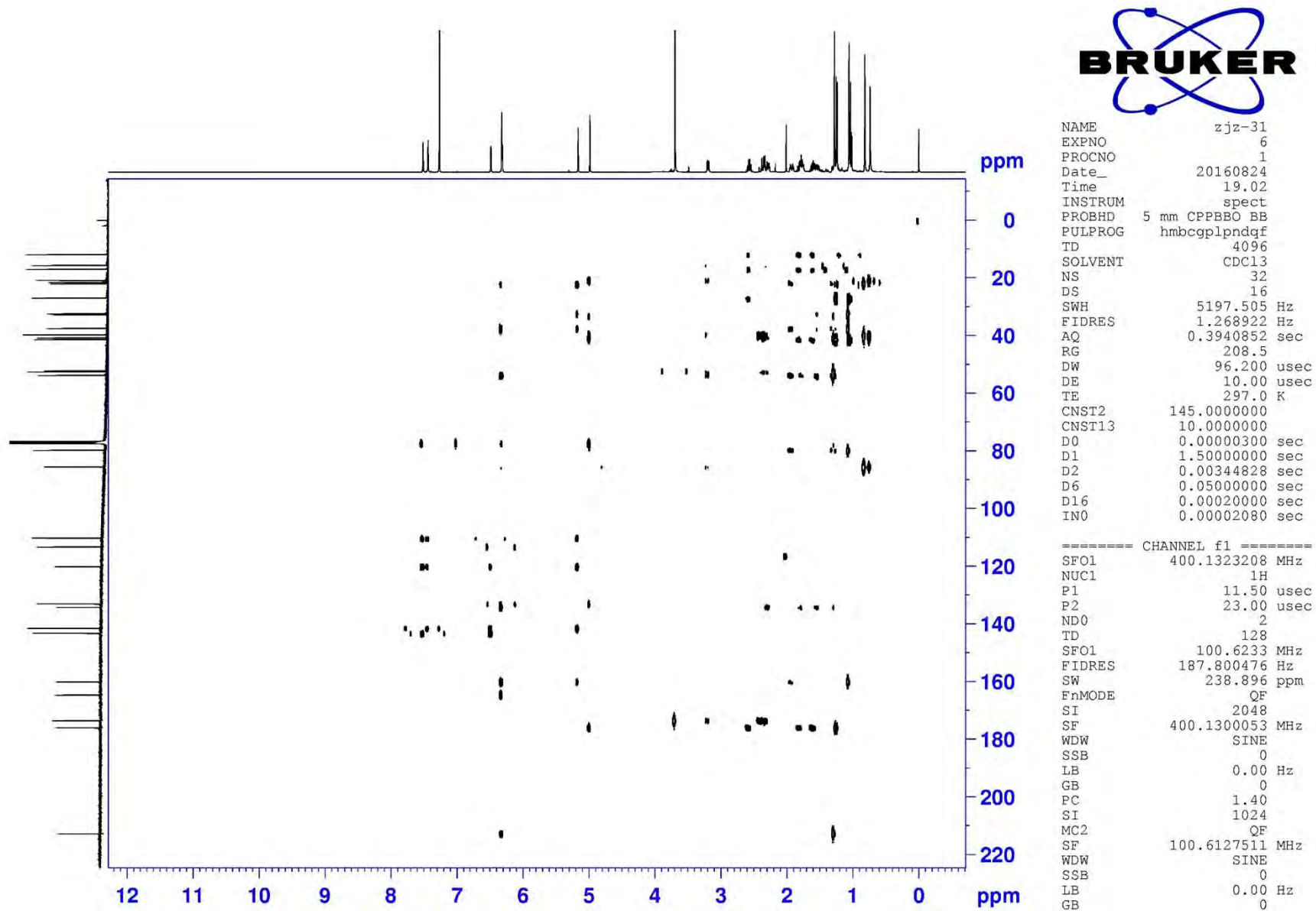
HSQC (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



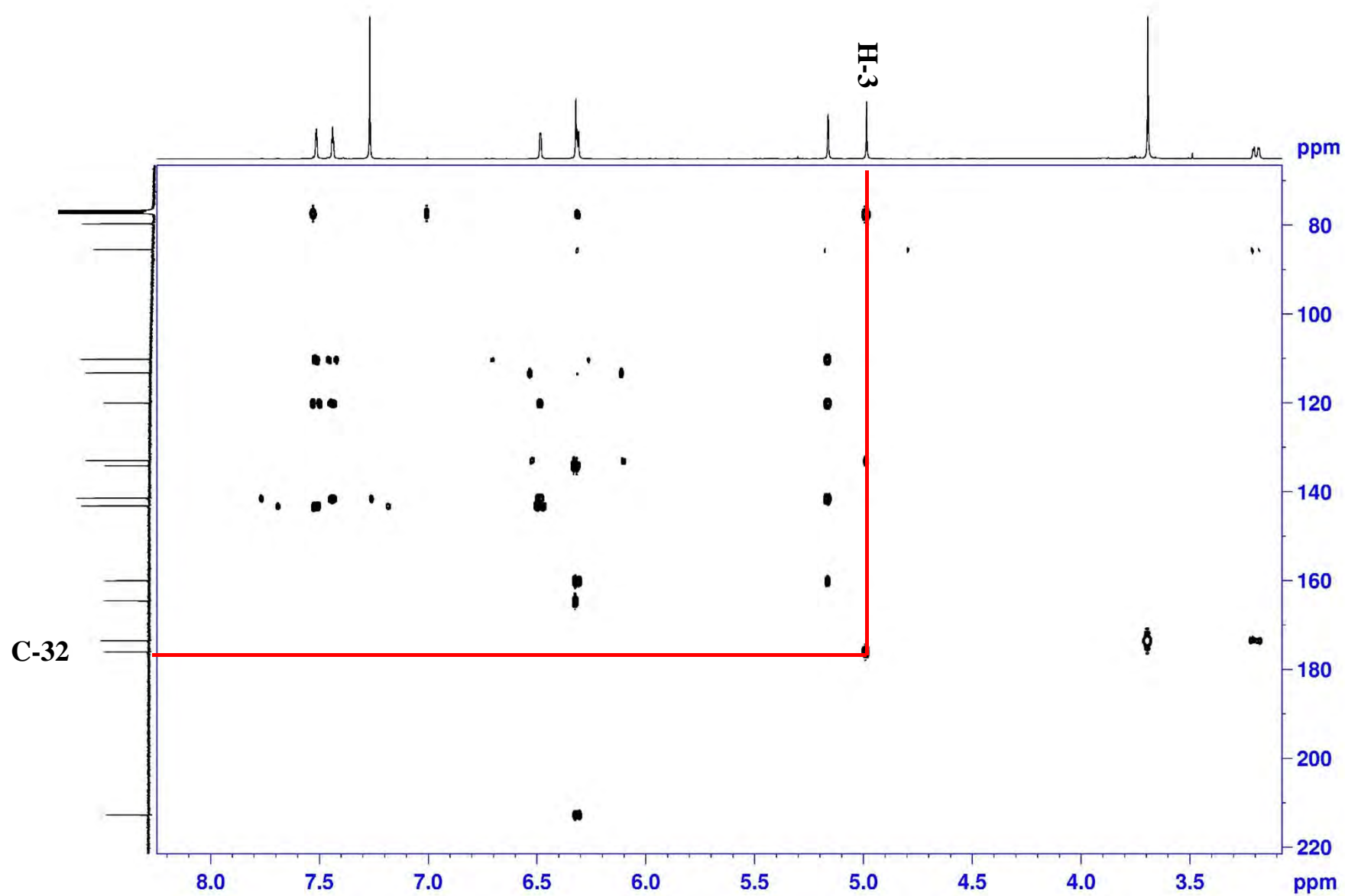
HSQC (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



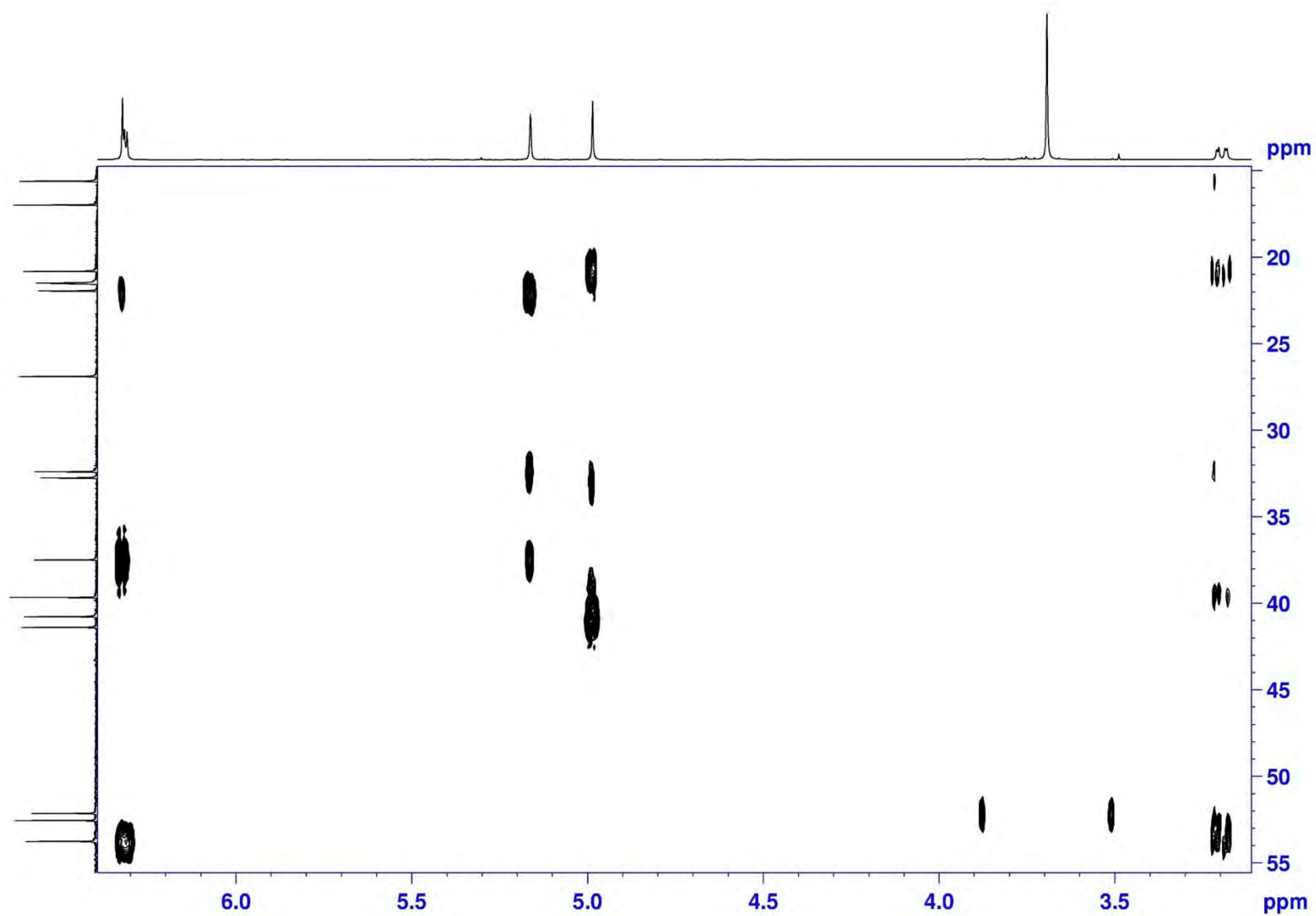
# HMBC (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

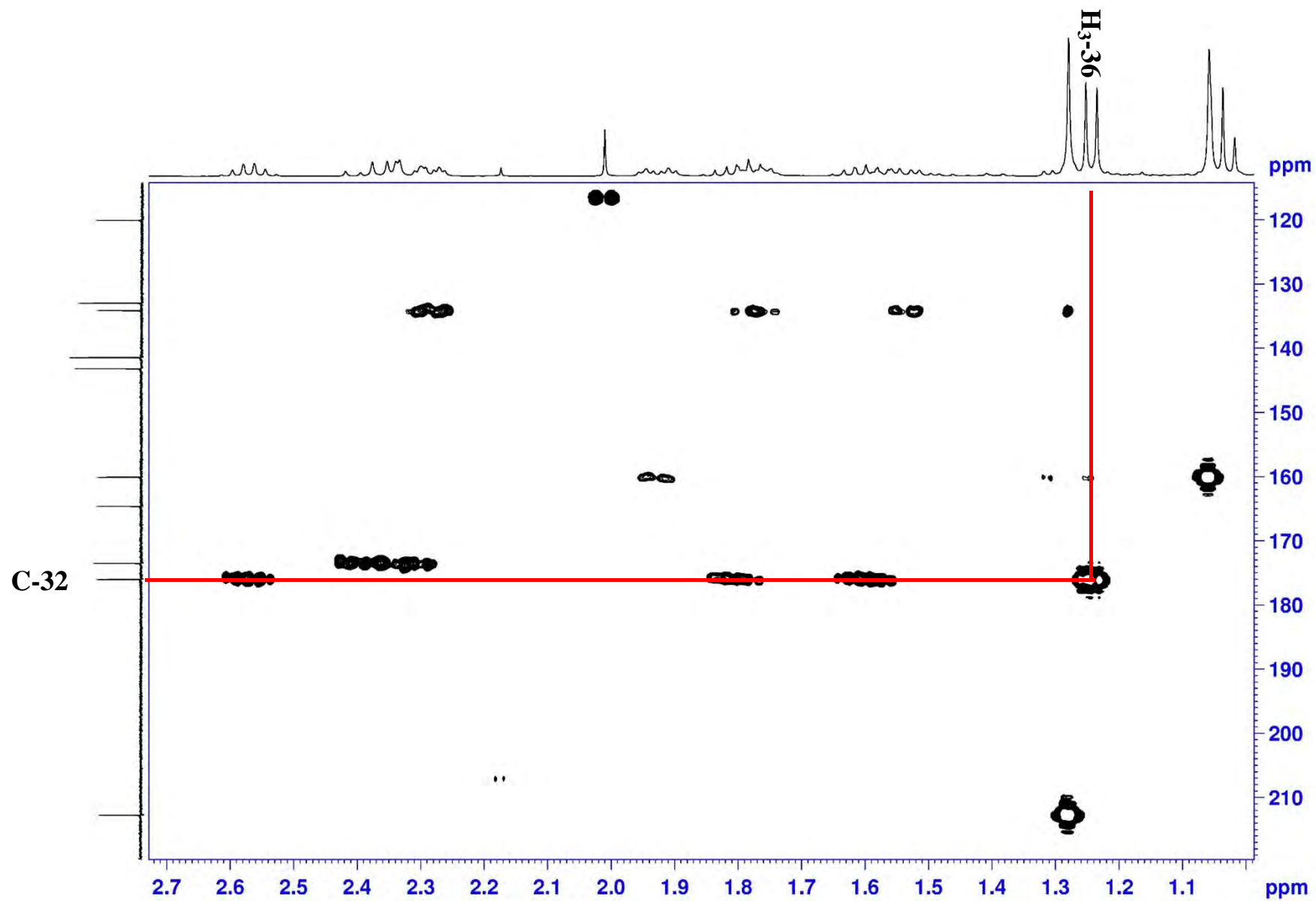


HMBC (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$

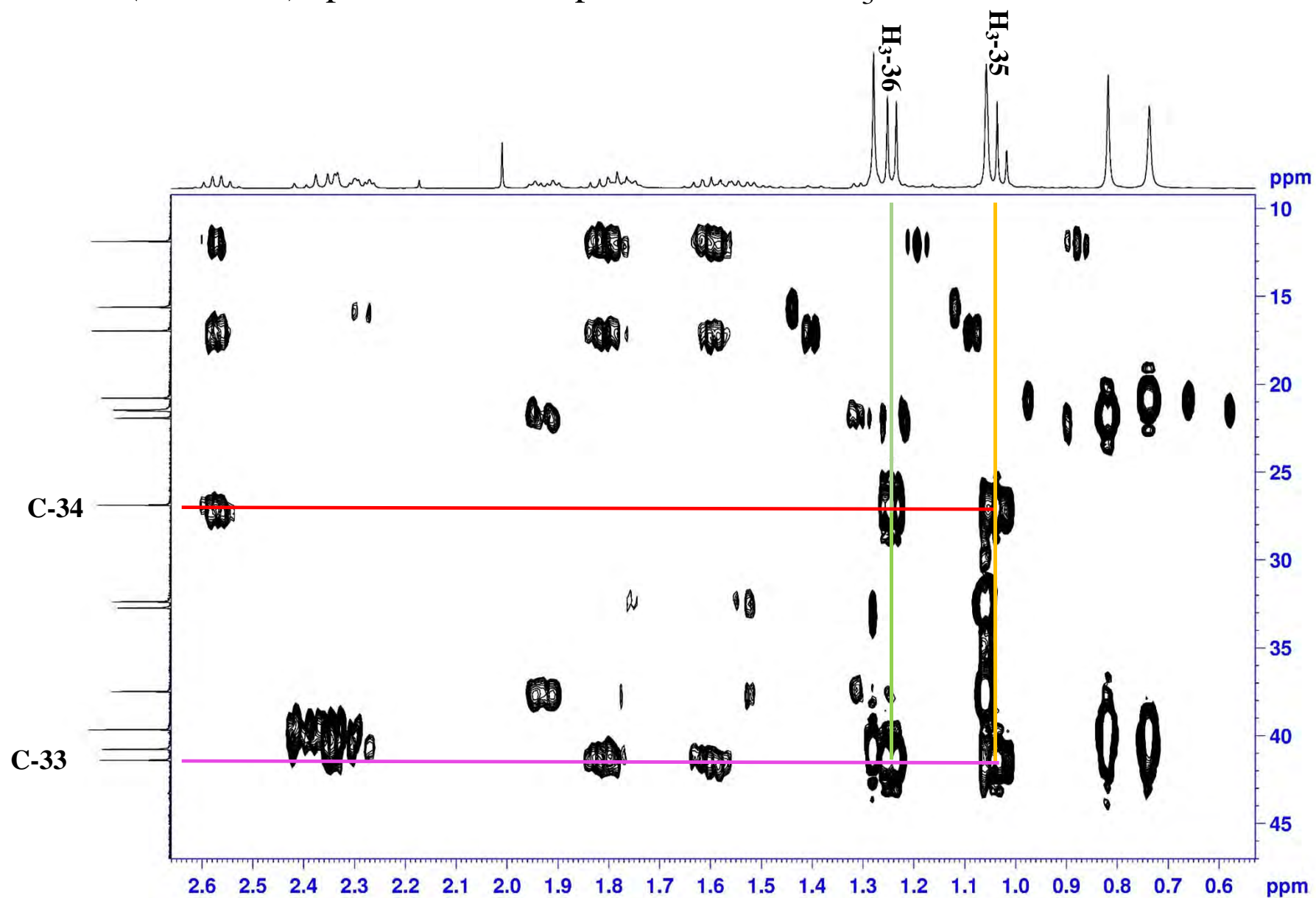




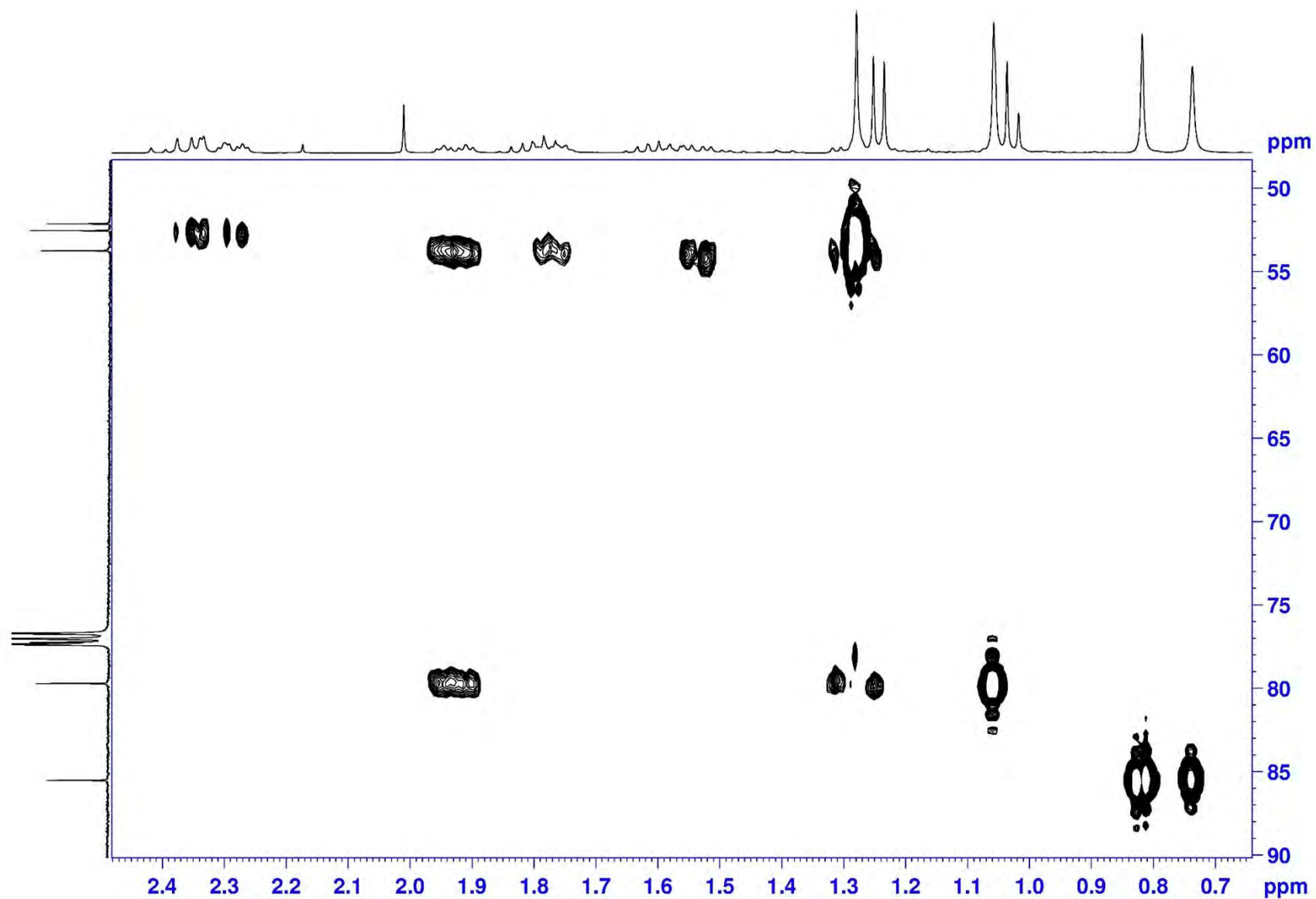
HMBC (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



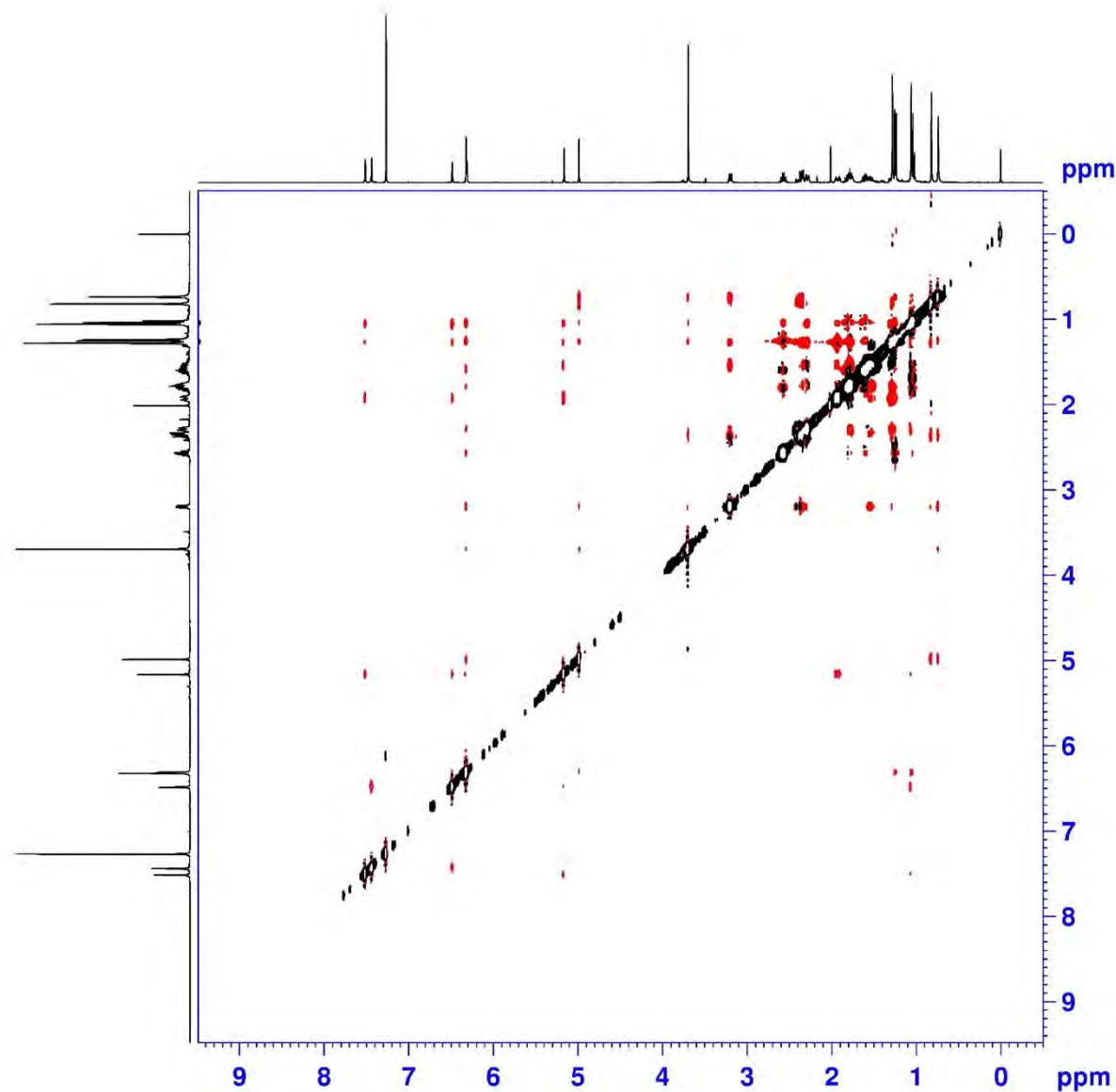
HMBC (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **11** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



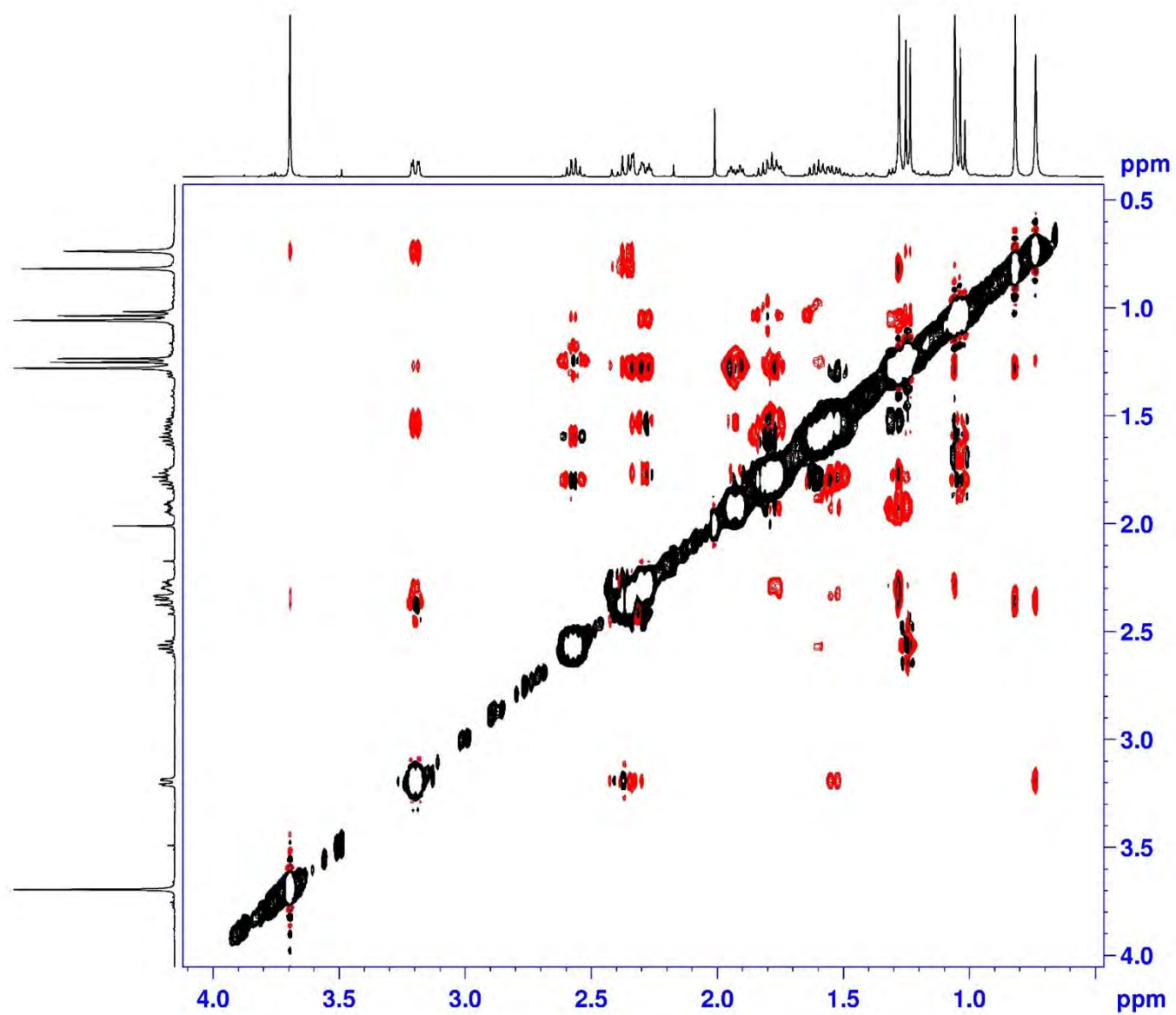
```

NAME                zjz-31
EXPNO                7
PROCNO              1
Date_               20160824
Time                21.18
INSTRUM             spect
PROBHD              5 mm CFPBBO BB
PULPROG             noesygpphpg
TD                  2048
SOLVENT             CDC13
NS                   16
DS                   32
SWH                 4000.000 Hz
FIDRES              1.953125 Hz
AQ                  0.2560500 sec
RG                   208.5
DW                  125.000 usec
DE                   10.00 usec
TE                   297.0 K
D0                  0.00011036 sec
D1                  1.99385595 sec
D8                  0.30000001 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D16                 0.00020000 sec
IN0                 0.00025000 sec
  
```

```

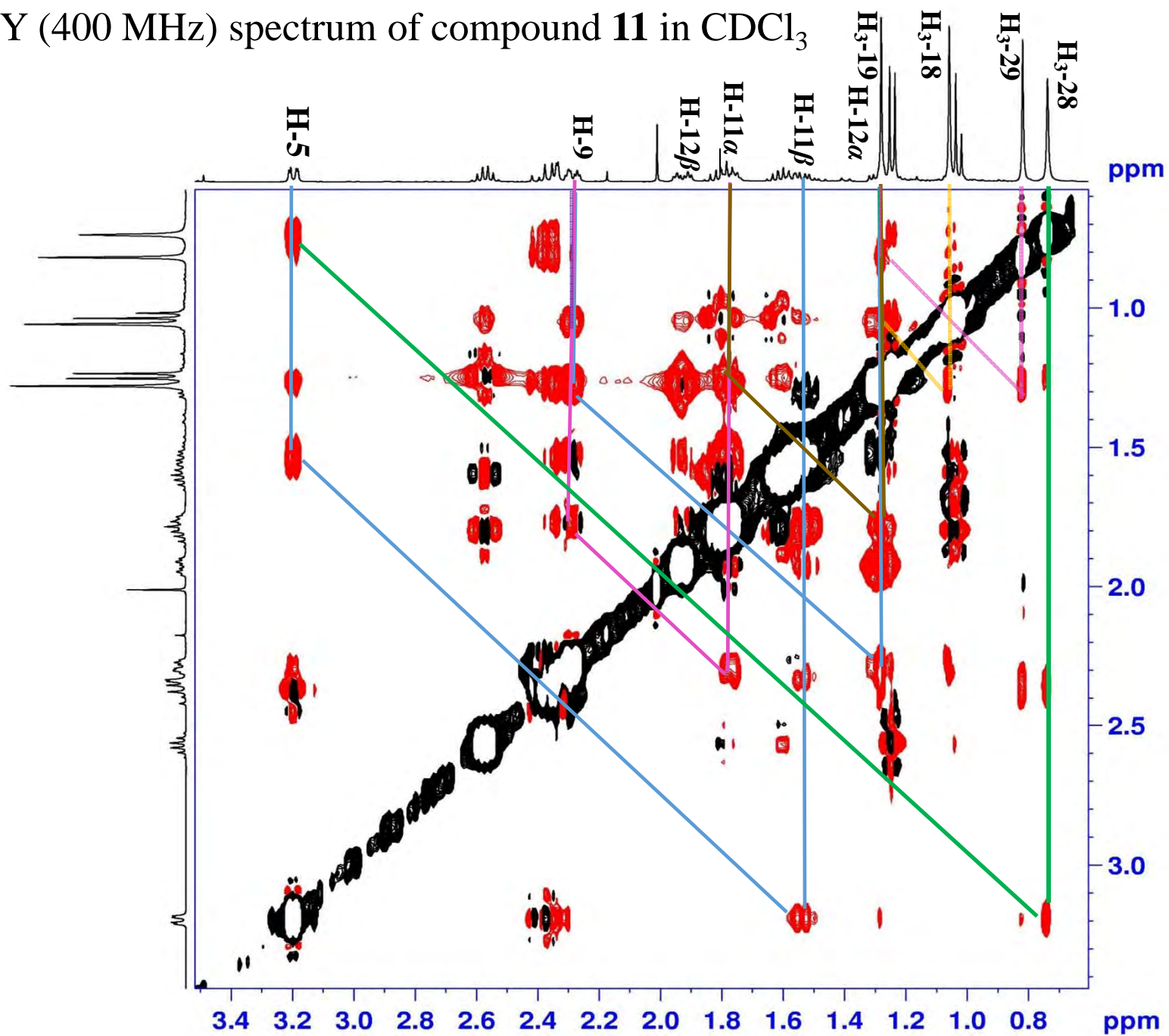
===== CHANNEL f1 =====
SFO1                400.1318006 MHz
NUC1                 1H
P1                   11.50 usec
P2                   23.00 usec
P17                  2500.00 usec
ND0                  1
TD                   256
SFO1                400.1318 MHz
FIDRES              15.625000 Hz
SW                   9.997 ppm
FnMODE              States-TPPI
SI                   1024
SF                  400.1300046 MHz
WDW                  QSINE
SSB                  2
LB                   0.00 Hz
GB                   0
PC                   1.00
SI                   1024
MC2                  States-TPPI
SF                  400.1300069 MHz
WDW                  QSINE
SSB                  2
LB                   0.00 Hz
GB                   0
  
```

NOESY (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>

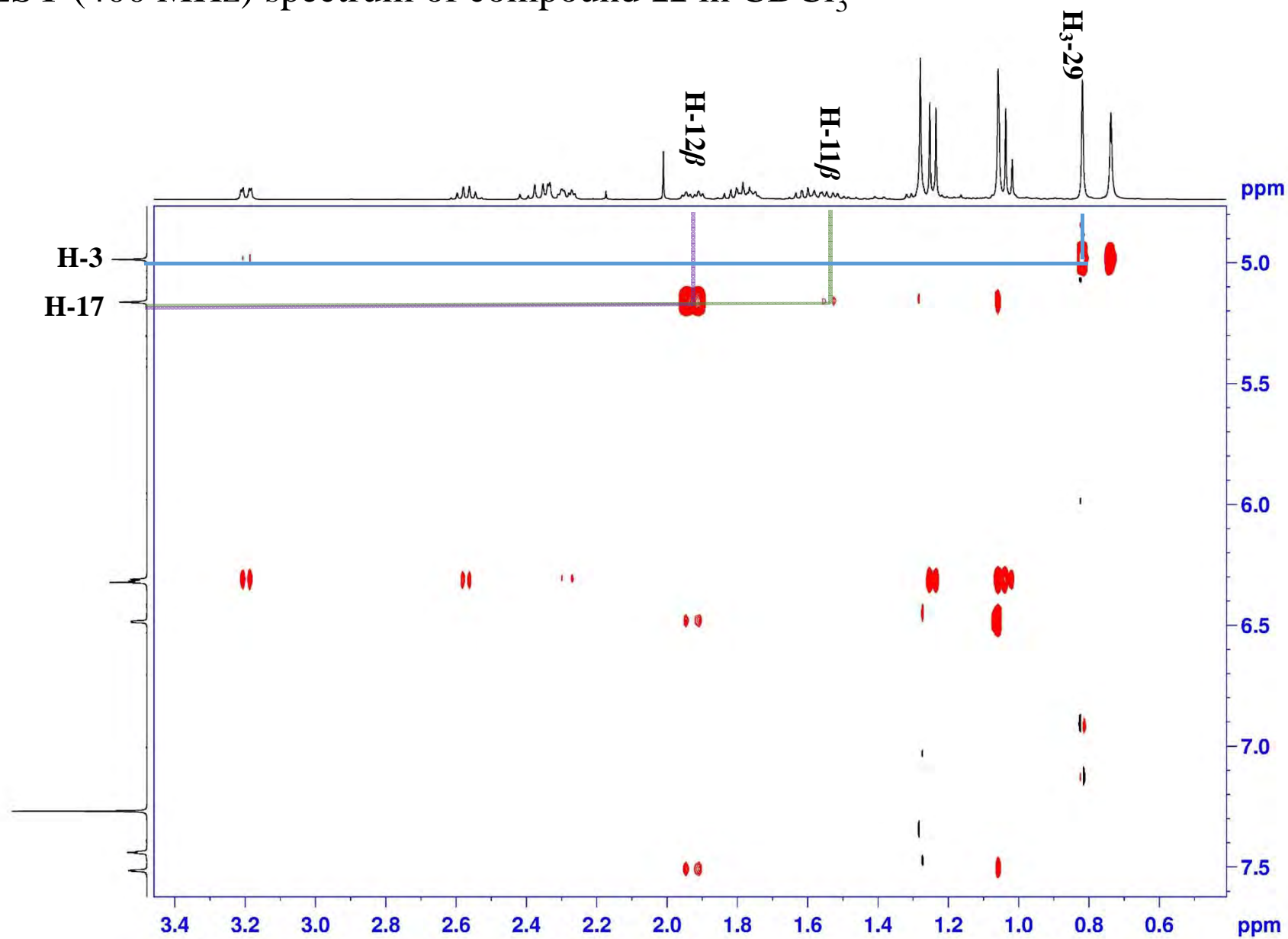




NOESY (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **11** in CDCl<sub>3</sub>





# HR-ESIMS for compound 12

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_lws-67\_pos\_16\_01\_1863.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-67\_pos  
 Comment

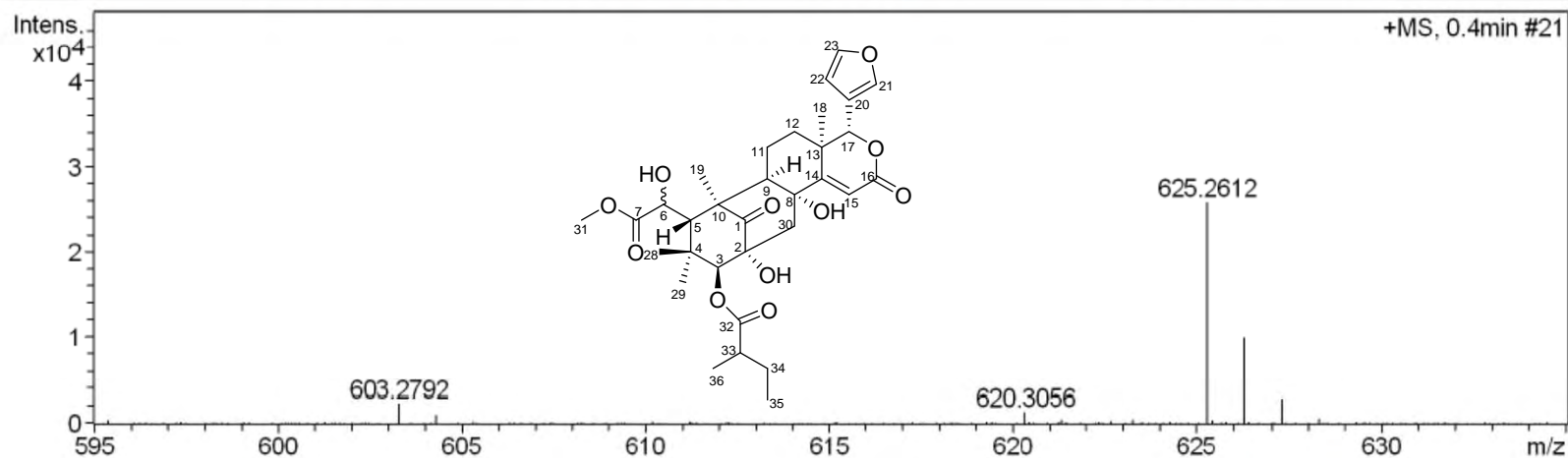
Acquisition Date 5/19/2016 9:08:38 AM

Operator SCSIO

Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



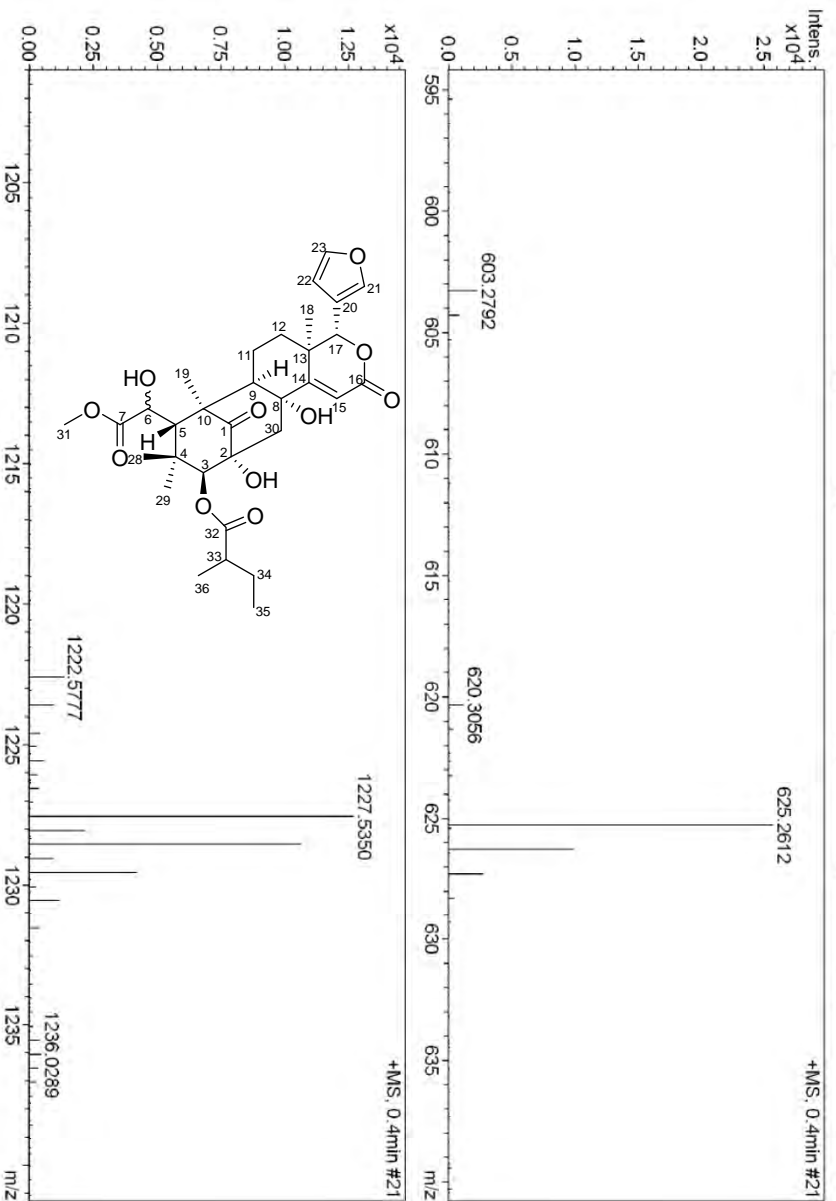
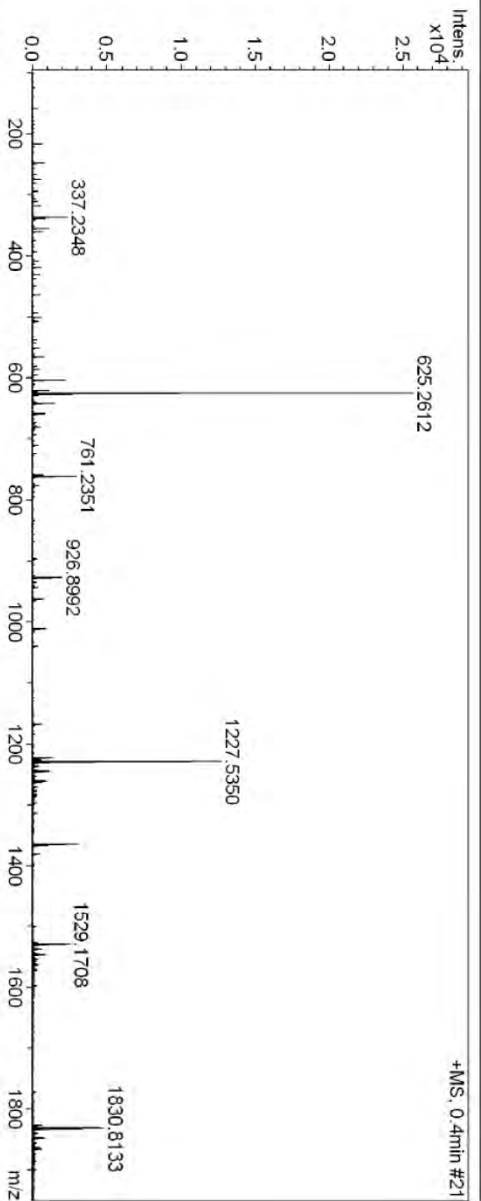
Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
603.2792	1	C 32 H 43 O 11	100.00	603.2800	1.3	0.8	8.8	11.5	even	ok
625.2612	1	C 32 H 42 Na O 11	100.00	625.2619	1.1	0.7	18.4	11.5	even	ok
1227.5350	1	C 64 H 84 Na O 22	100.00	1227.5346	-0.3	-0.3	59.9	22.5	even	ok

## Generic Display Report

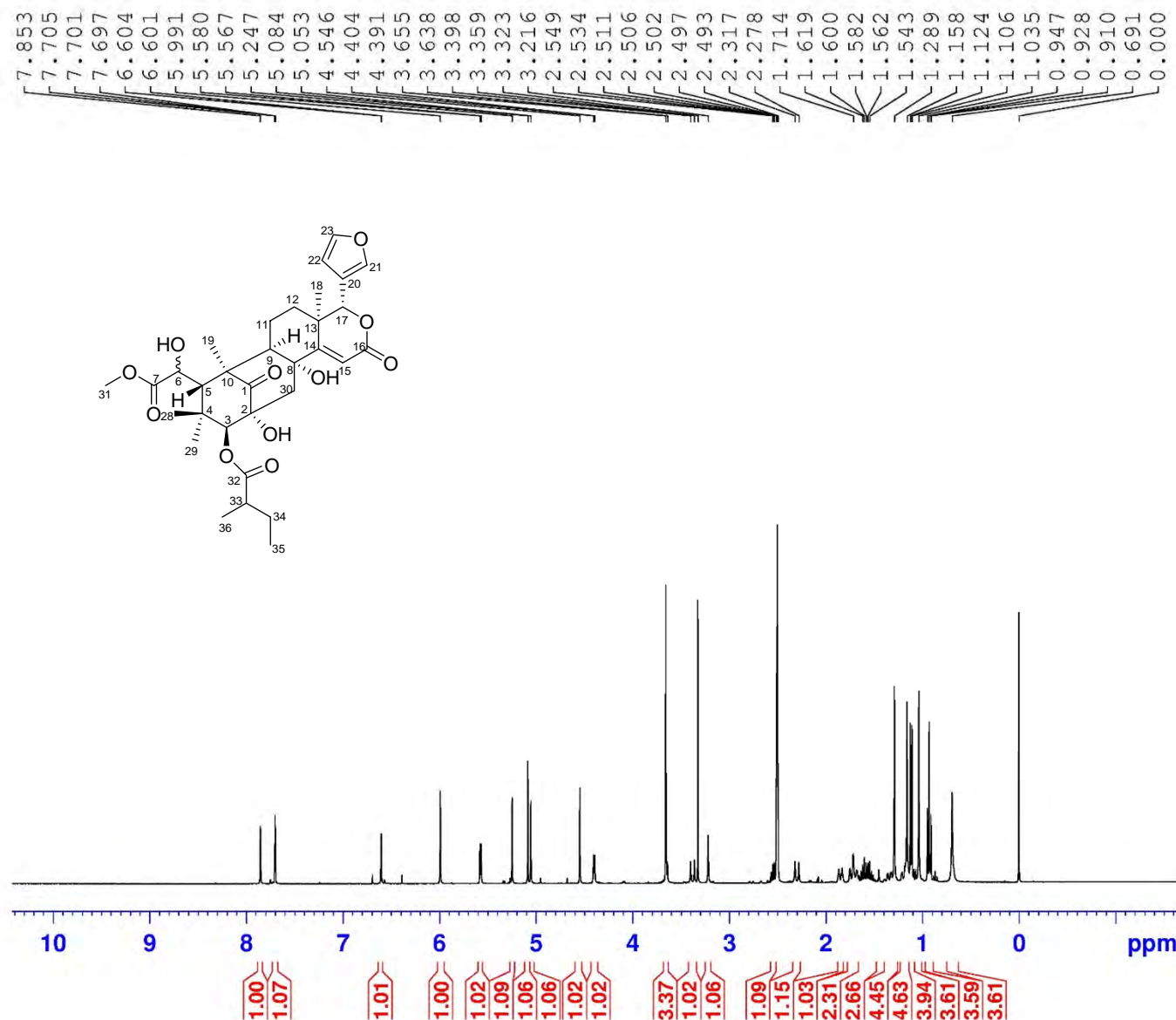
S338

### Analysis Info

Analysis Name	D:\Data\MS\data\201605\liwanshan_lws-67_pos_16_01_1863.d	Acquisition Date	5/19/2016 9:08:38 AM
Method	LC_Direct Infusion_pos_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_lws-67_pos	Instrument	maxis
Comment			



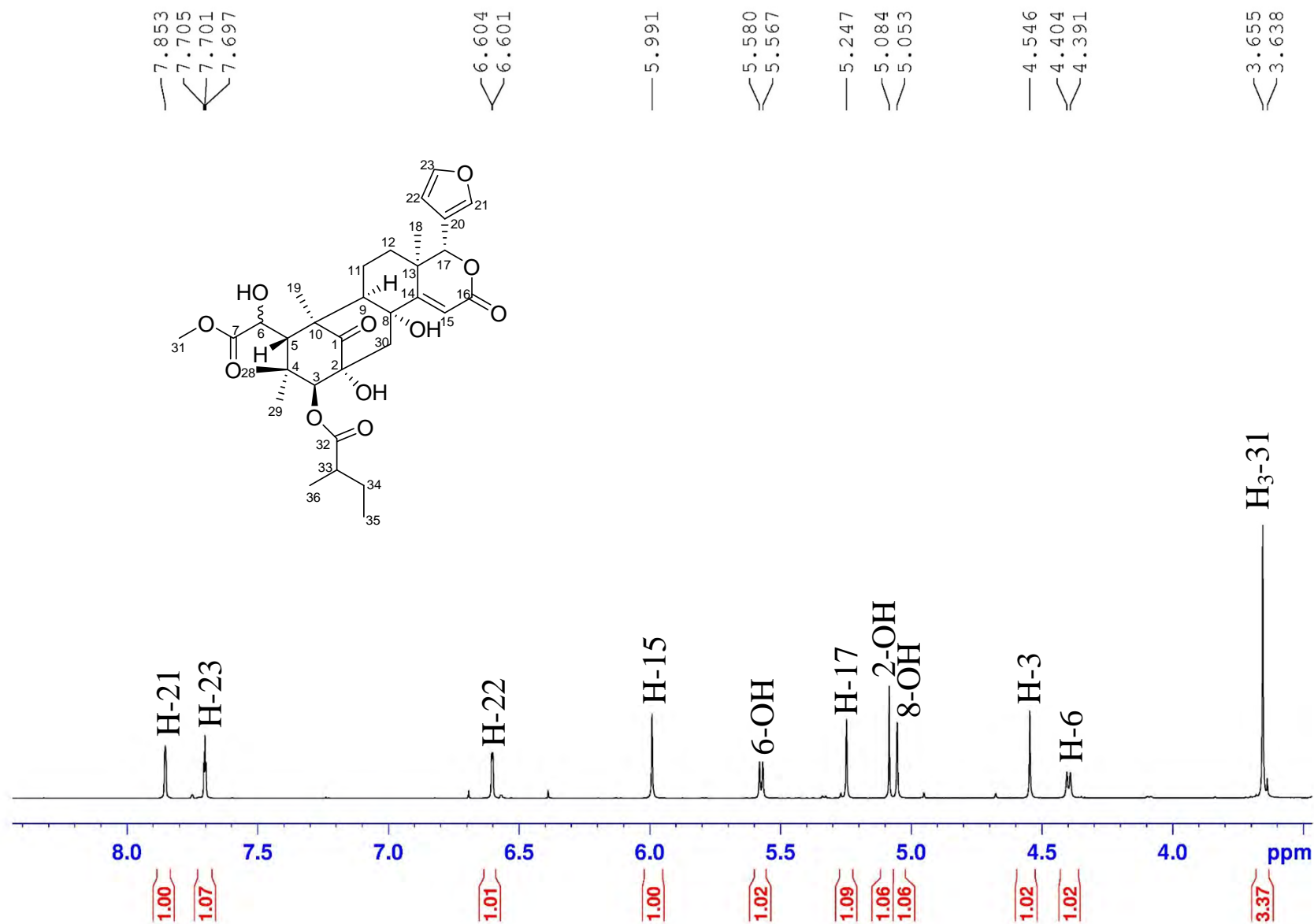
# $^1\text{H}$ NMR (400 MHz) spectrum of compound **12** in DMSO- $d_6$



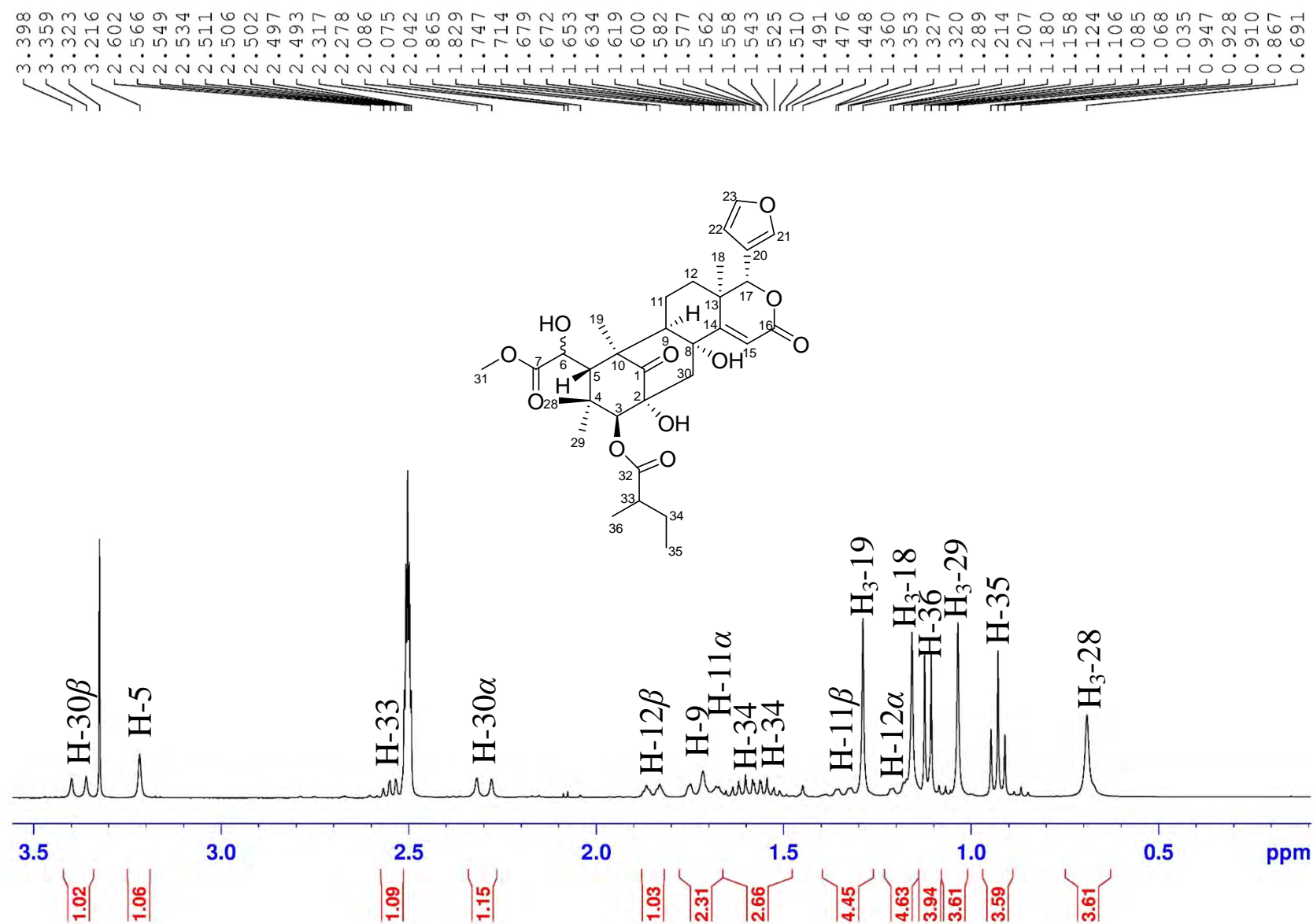
NAME lws-67-DMSO  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20160627  
 Time 17.22  
 INSTRUM spect  
 PROBHD 5 mm CPPBBO BB  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 16  
 DS 2  
 SWH 8223.685 F  
 FIDRES 0.125483 F  
 AQ 3.9846387 s  
 RG 208.5  
 DW 60.800 u  
 DE 10.00 u  
 TE 297.0 K  
 D1 1.00000000 s  
 TD0 1

===== CHANNEL f1 =====  
 SFO1 400.1324710 M  
 NUC1  $^1\text{H}$   
 P1 11.50 u  
 SI 65536  
 SF 400.1300030 M  
 WDW EM  
 SSB 0  
 LB 0.30 F  
 GB 0  
 PC 1.00

$^1\text{H}$  NMR (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

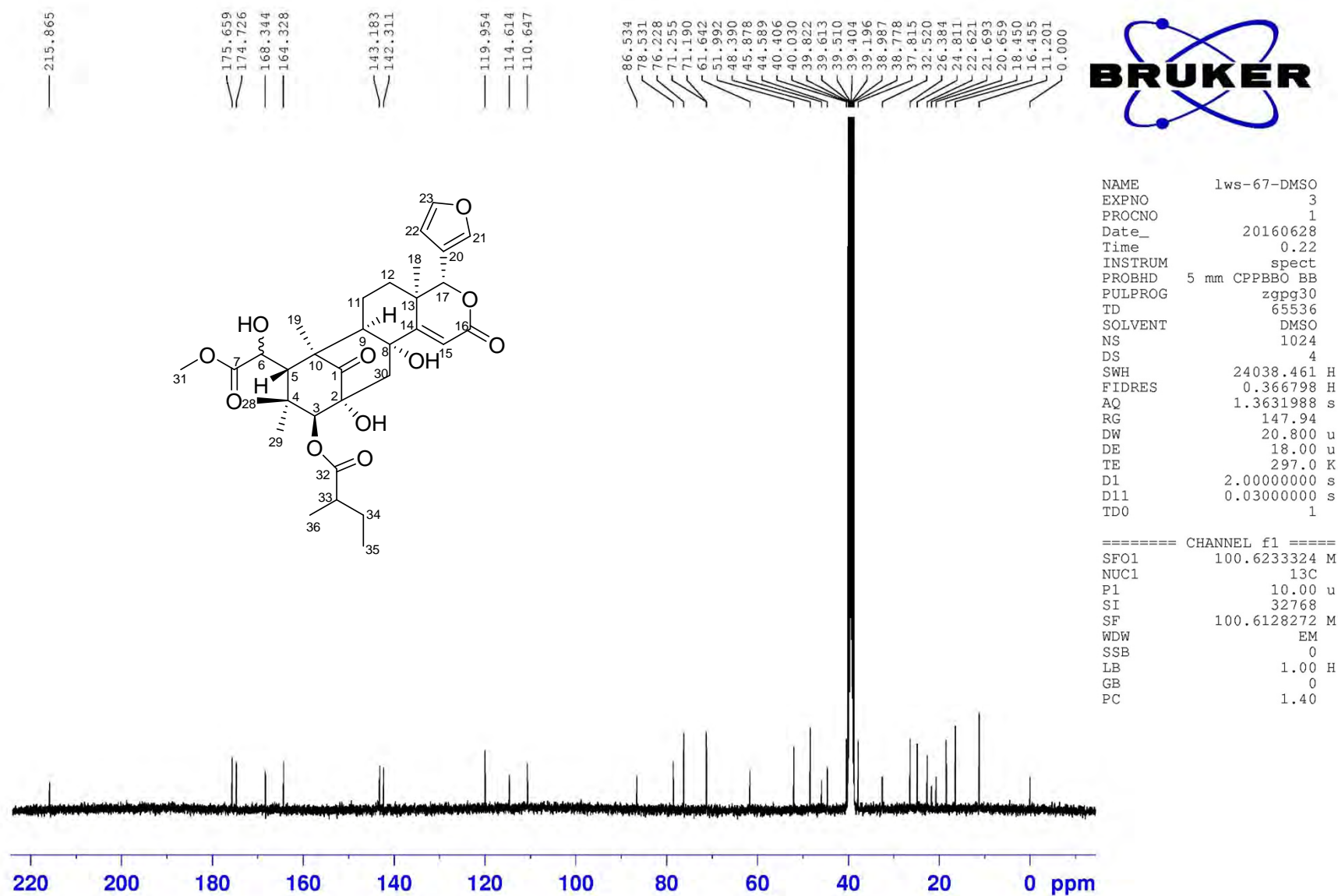


$^1\text{H}$  NMR (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

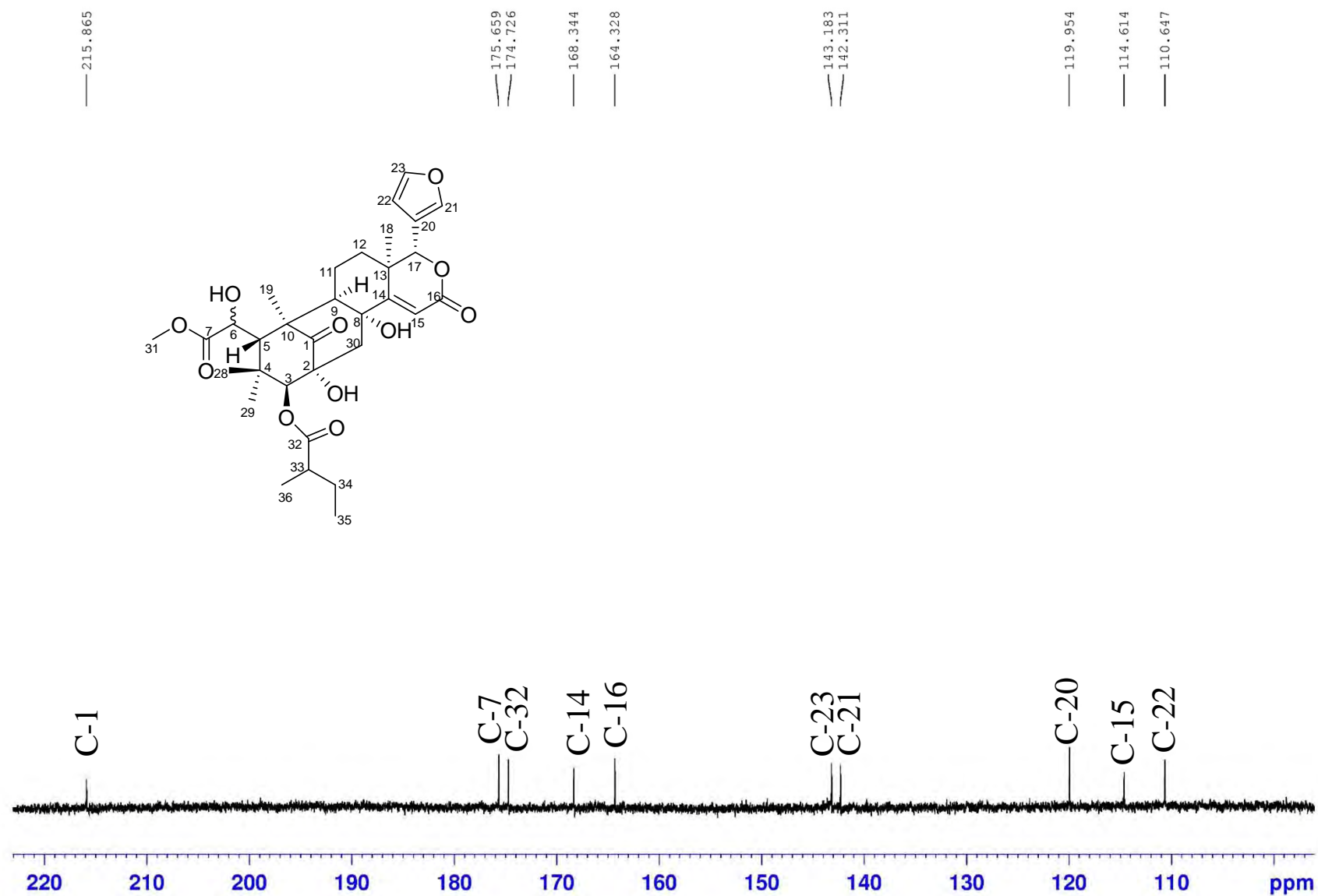




# $^{13}\text{C}$ NMR (100 MHz) spectrum of compound **12** in $\text{DMSO-}d_6$

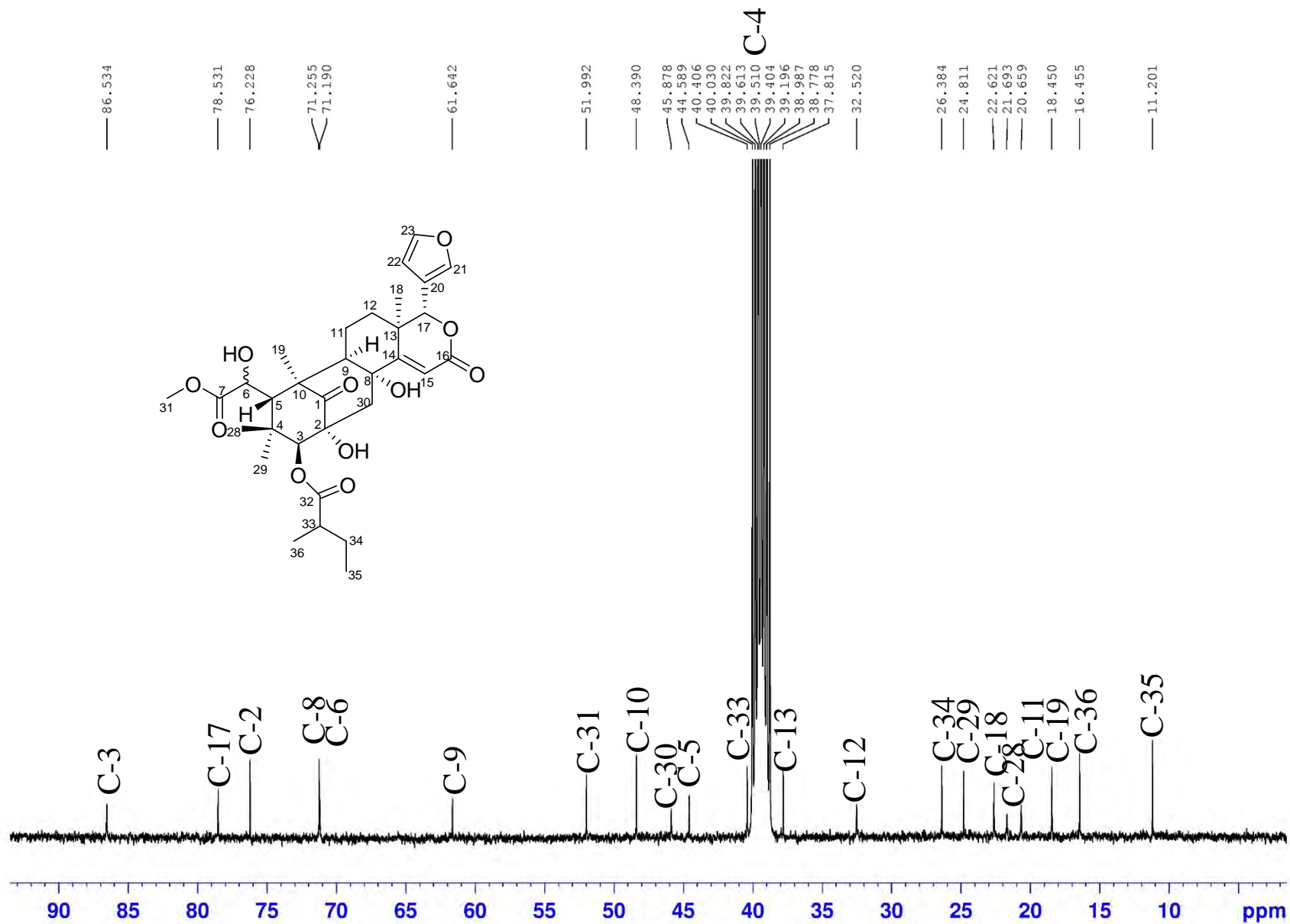


$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **12** in  $\text{DMSO-}d_6$

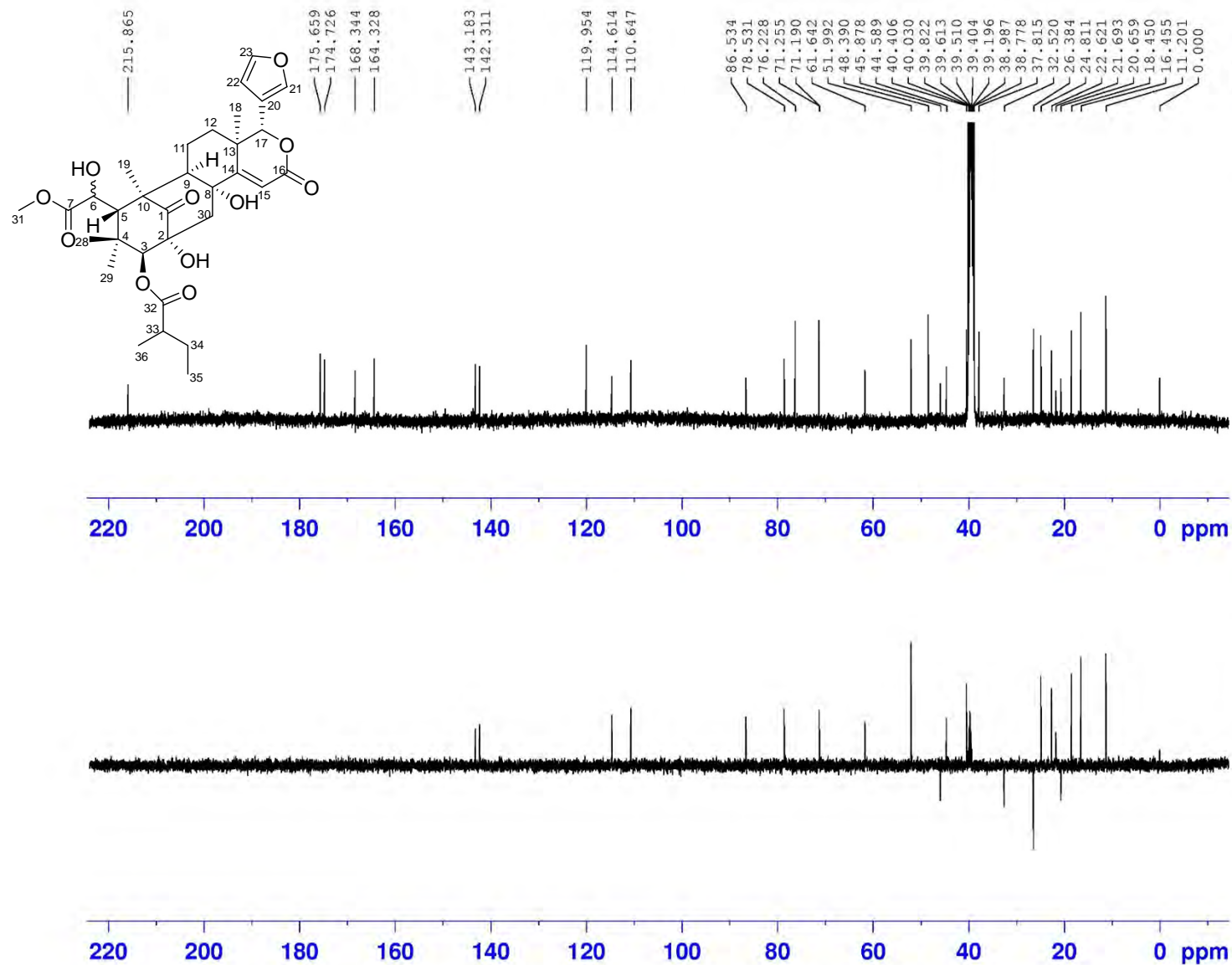




$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **12** in  $\text{DMSO-}d_6$



# DEPT135 (100 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



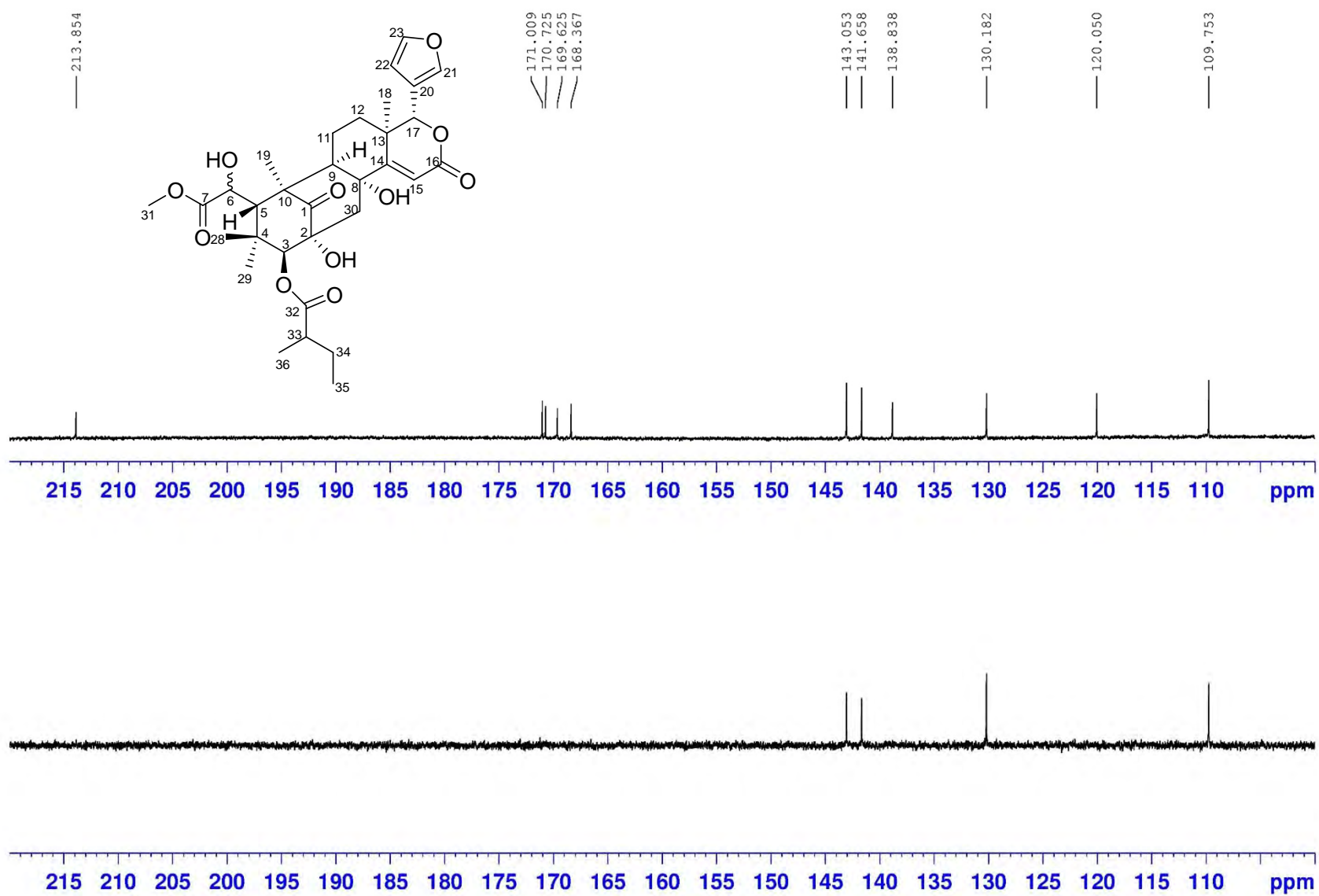
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NAME      lws-67-DMSO
EXPNO      4
PROCNO     1
Date_      20160628
Time       0.40
INSTRUM    spect
PROBHD     5 mm CPPBBO BB
PULPROG    deptspl35
TD         65536
SOLVENT    DMSO
NS         300
DS         4
SWH        24038.461 F
FIDRES     0.366798 F
AQ         1.3631988 s
RG         130.26
DW         20.800 u
DE         18.00 u
TE         297.0 F
CNST2      145.0000000
D1         2.0000000 s
D2         0.00344828 s
D12        0.00002000 s
TD0        1
    
```

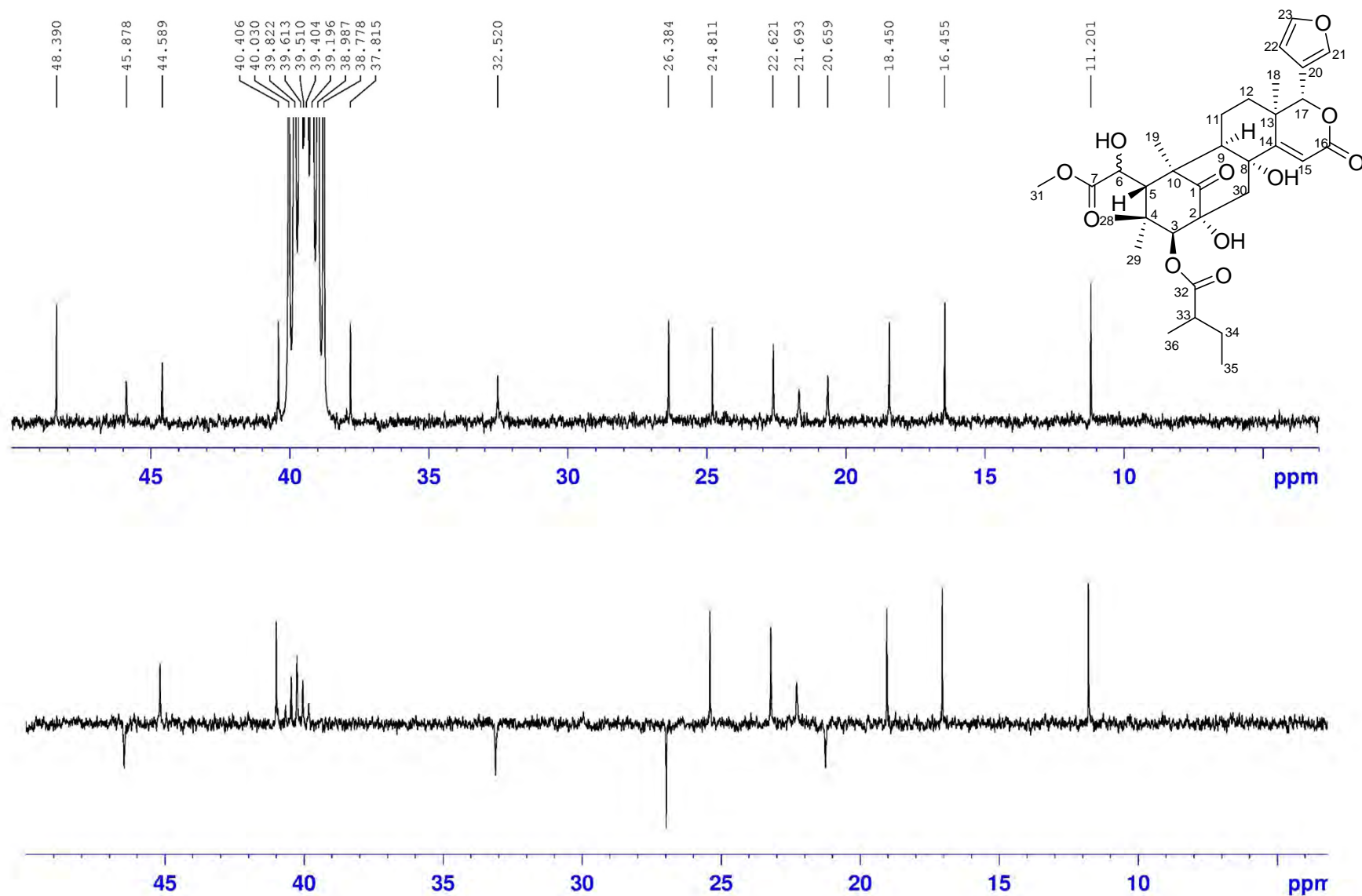
```

===== CHANNEL f1 =====
SFO1      100.6233324 M
NUC1       13C
P1         10.00 u
P13        2000.00 u
SI         32768
SF         100.6128273 M
WDW        EM
SSB        0
LB         1.00 F
GB         0
PC         1.40
    
```

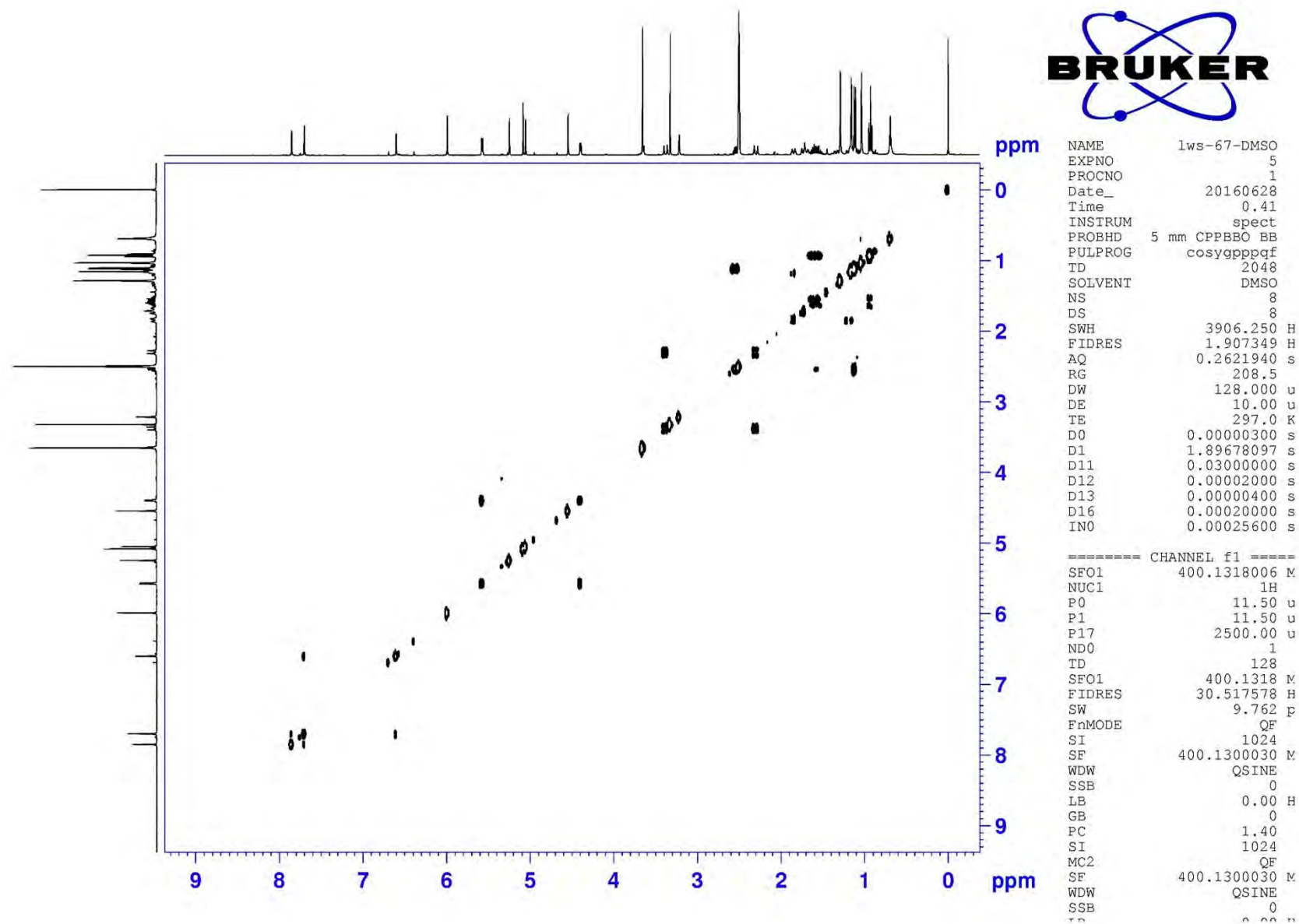
# DEPT135 (100 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



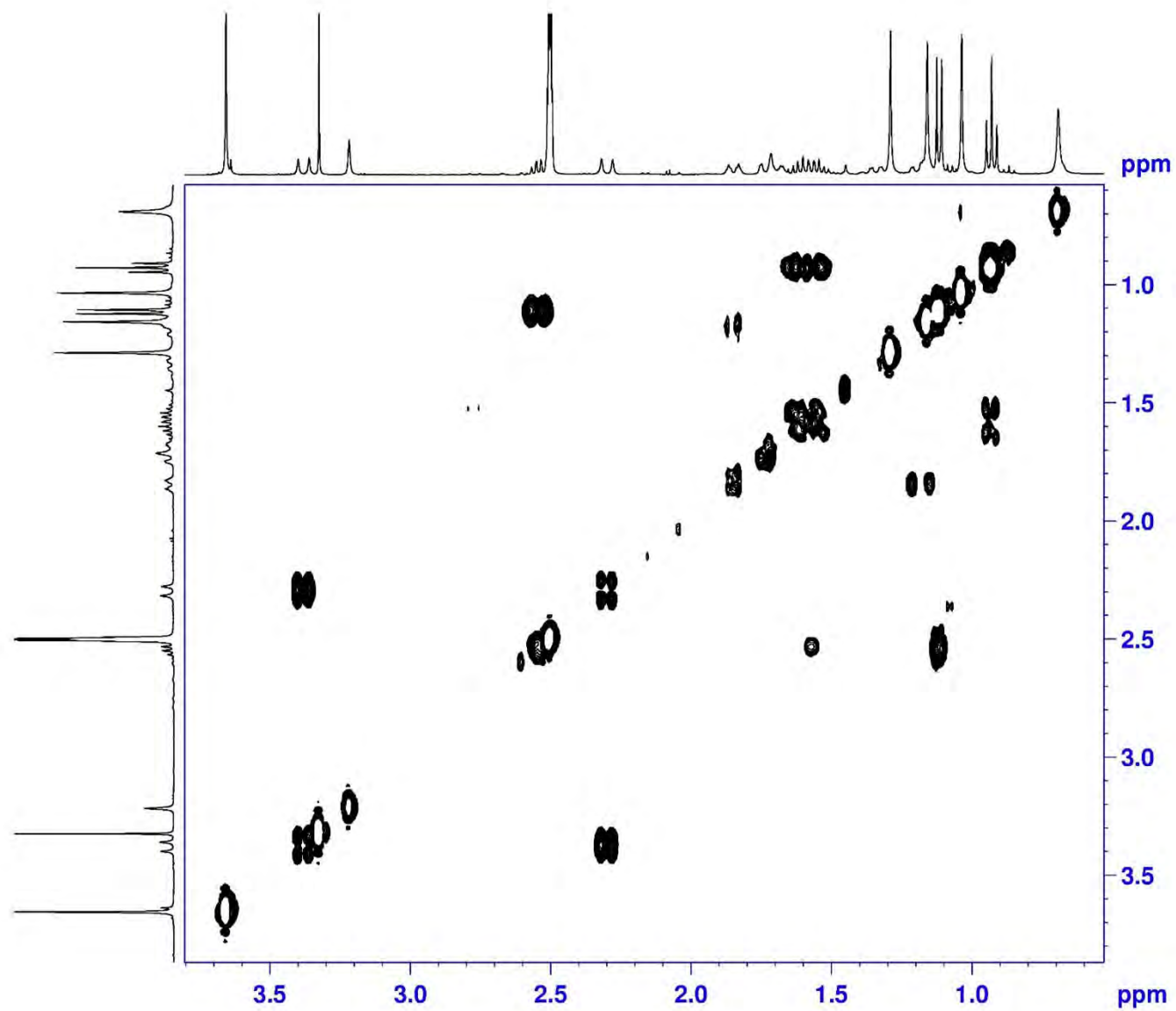
# DEPT135 (100 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



# $^1\text{H}$ - $^1\text{H}$ COSY (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

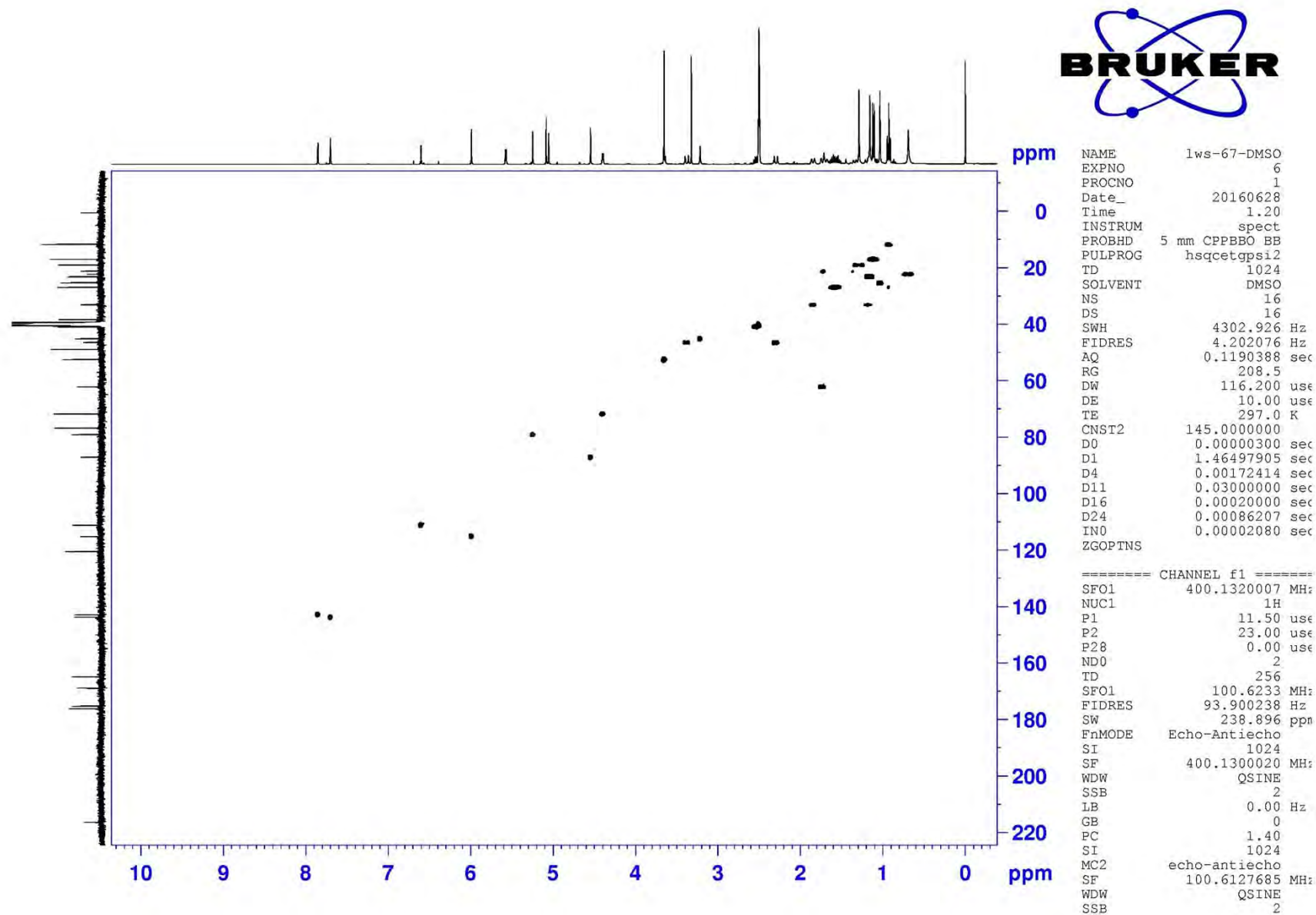


$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **12** in DMSO- $d_6$

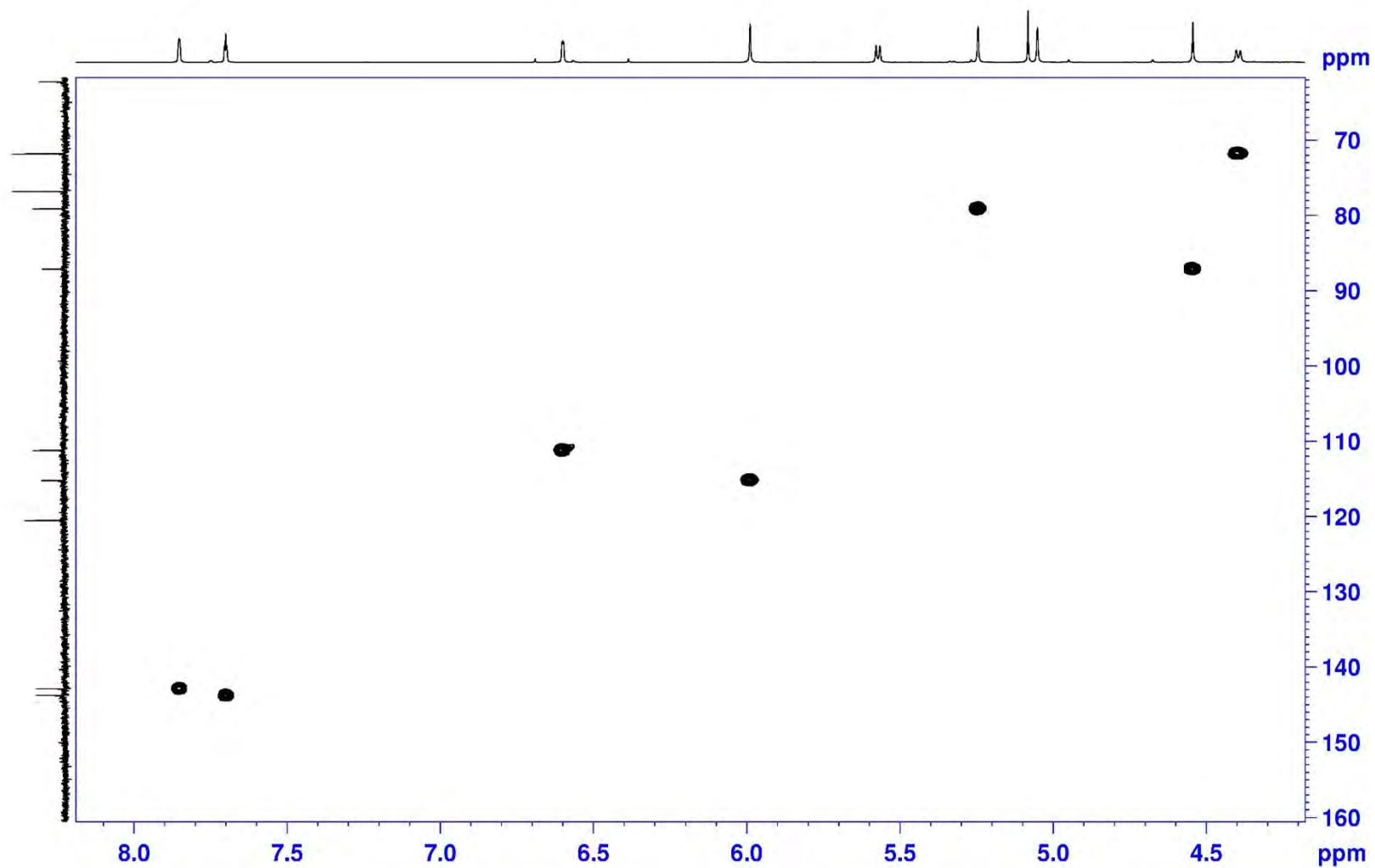




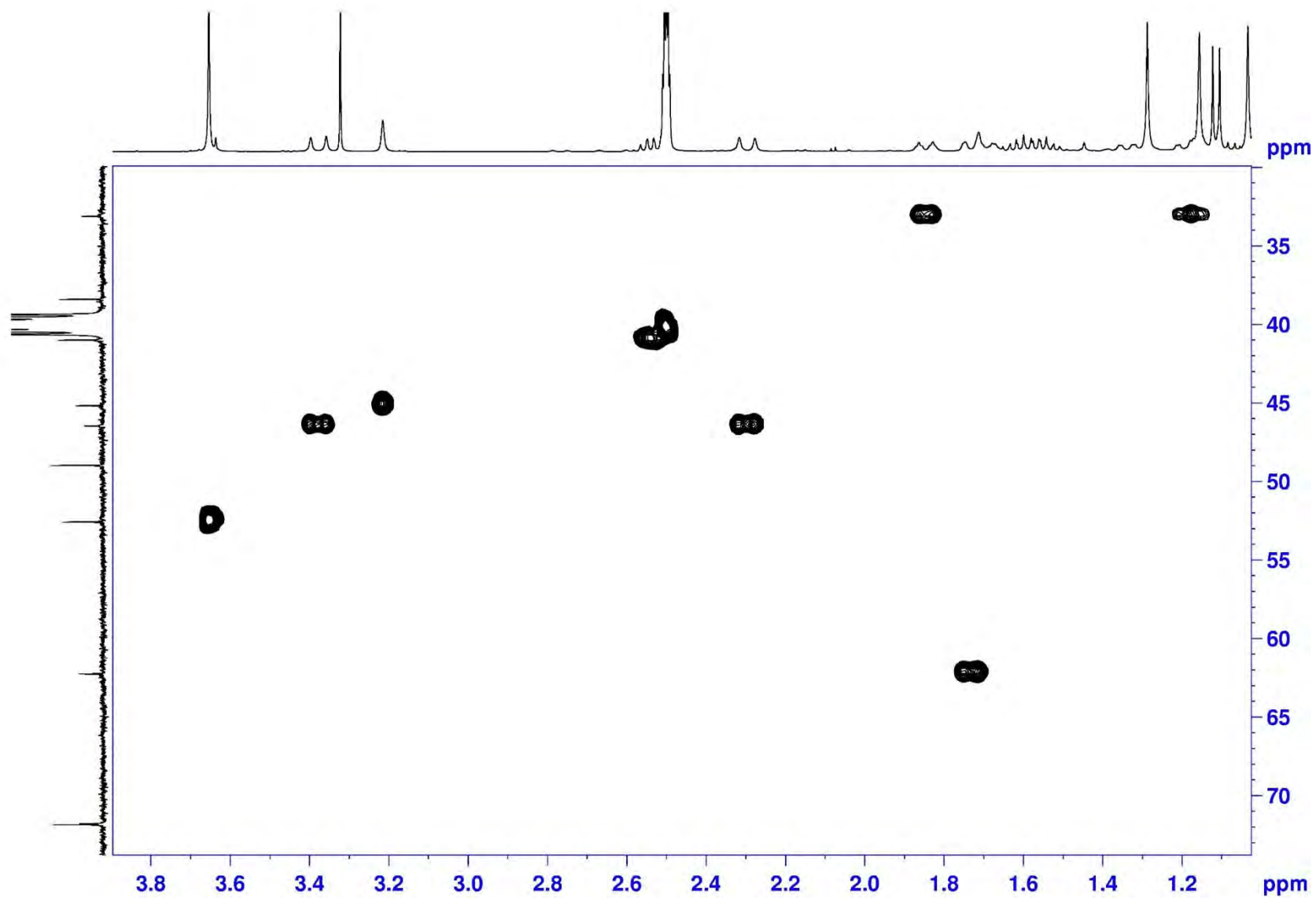
# HSQC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



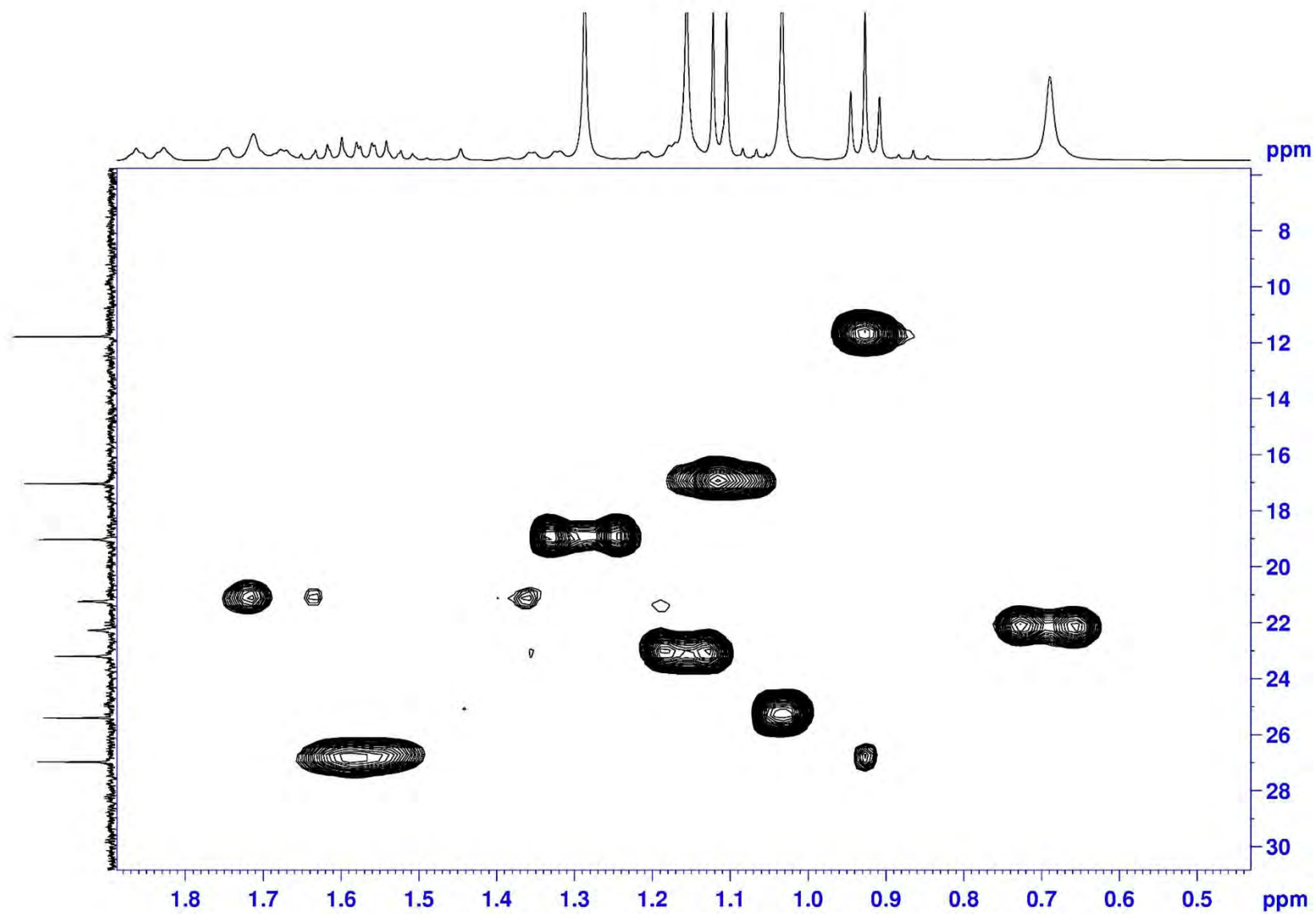
HSQC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



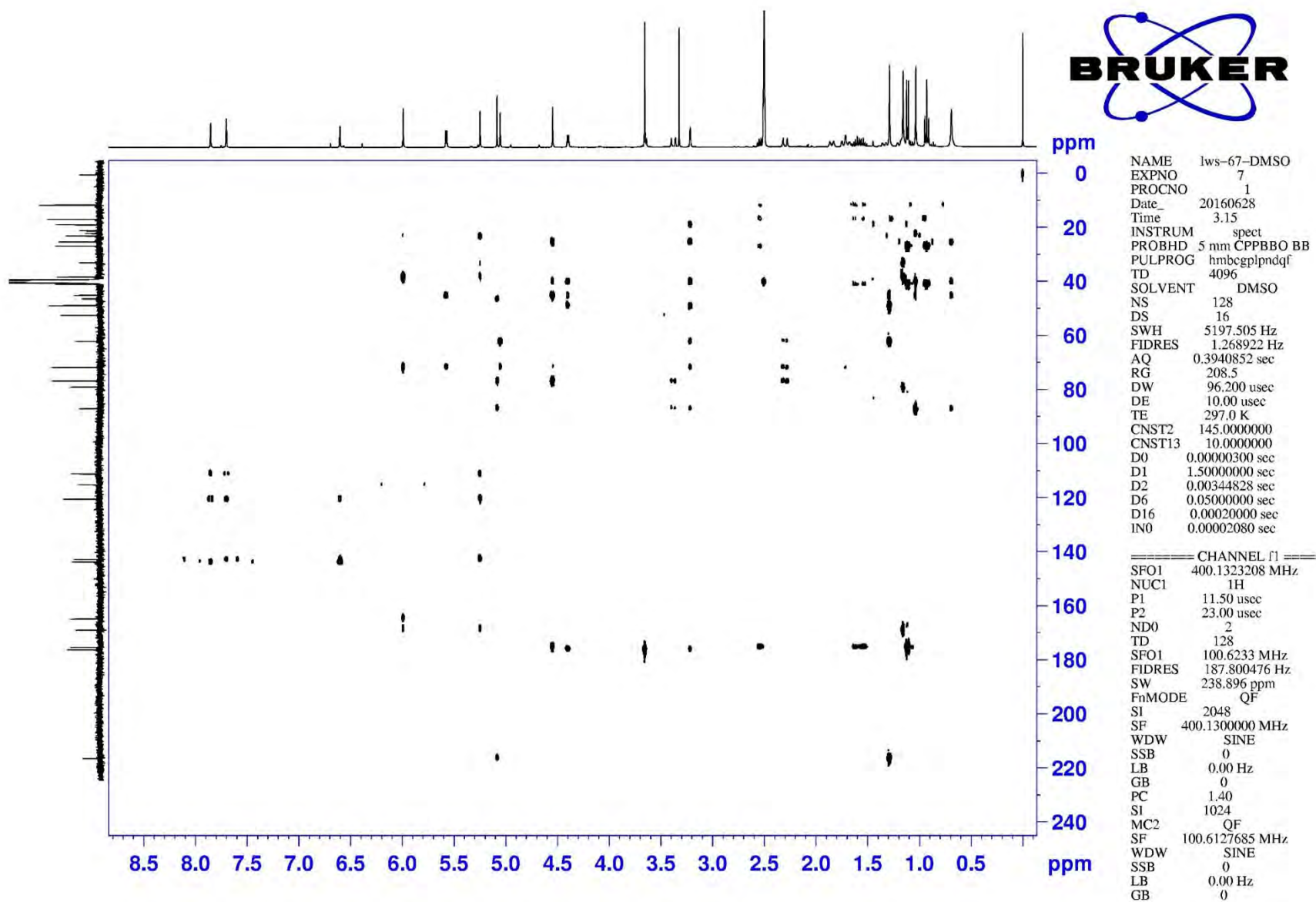
HSQC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



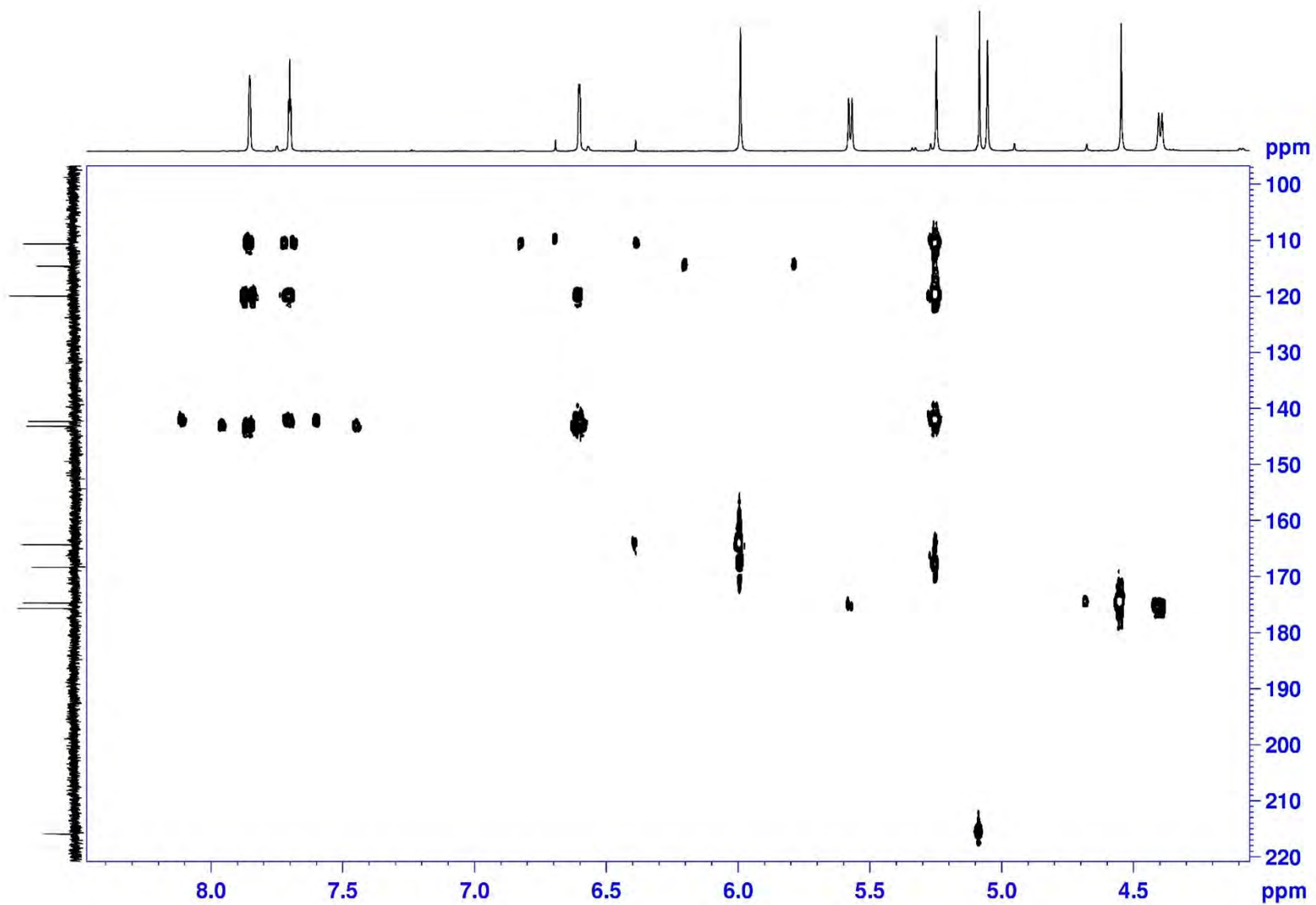
HSQC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



# HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

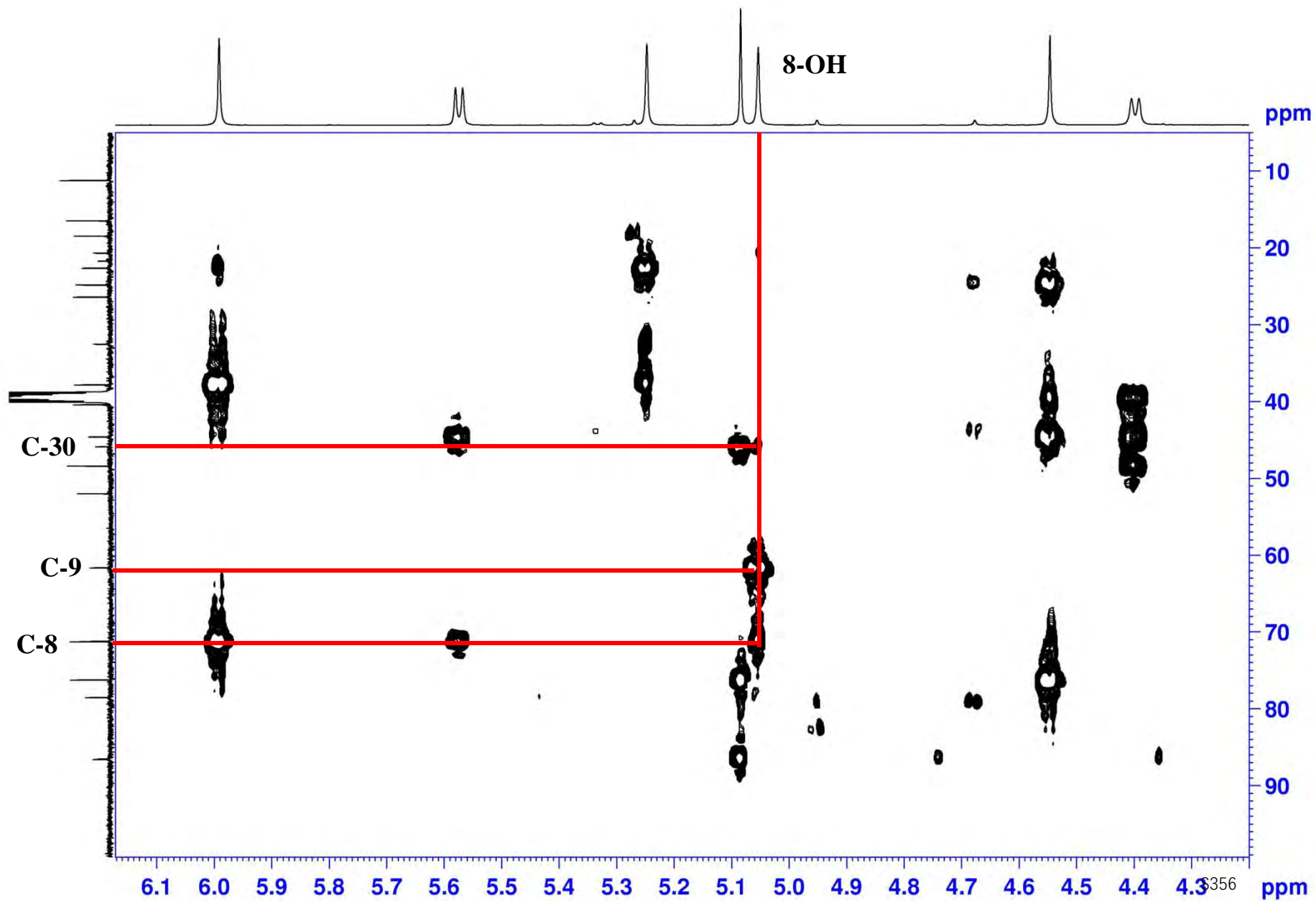


HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

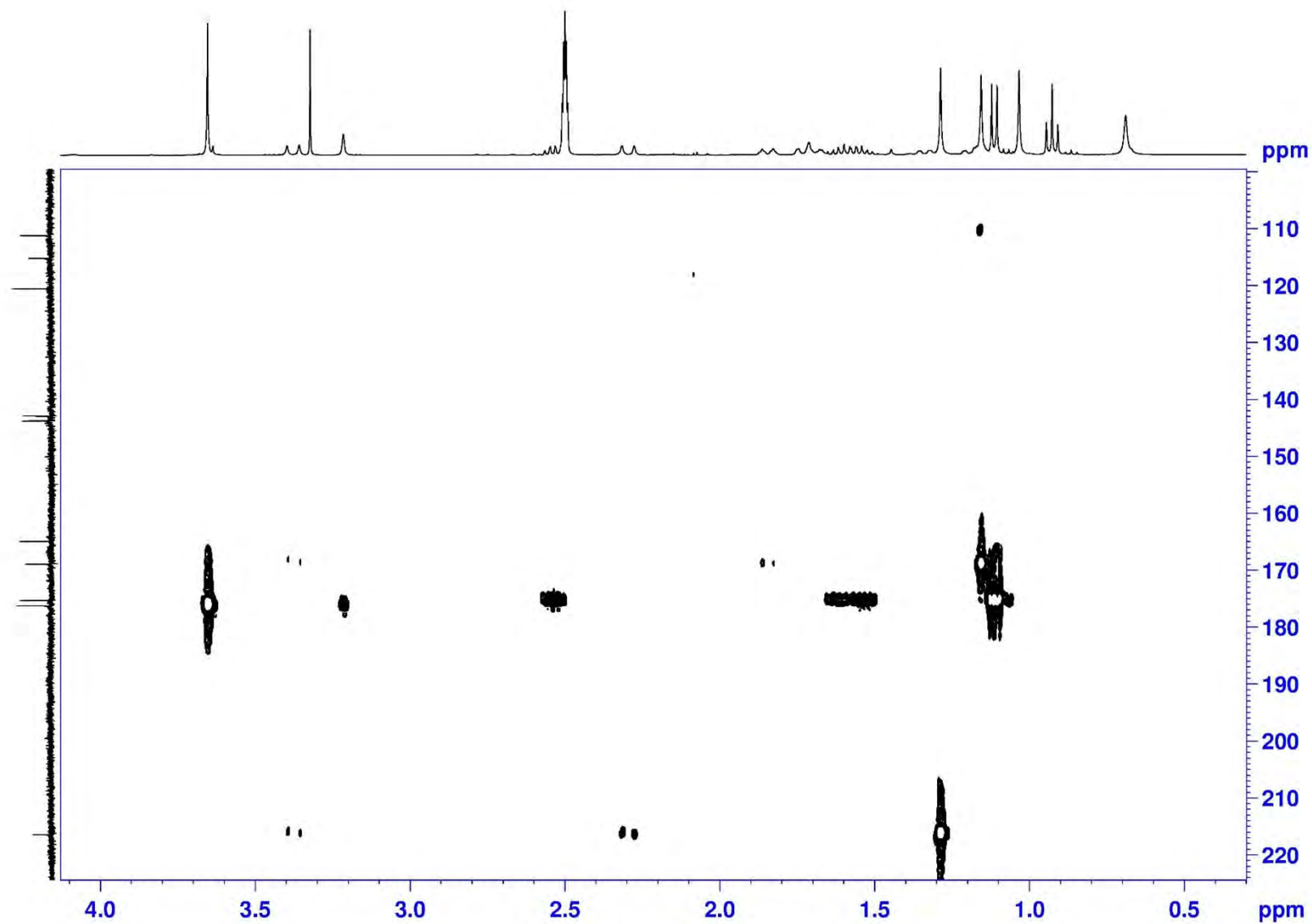




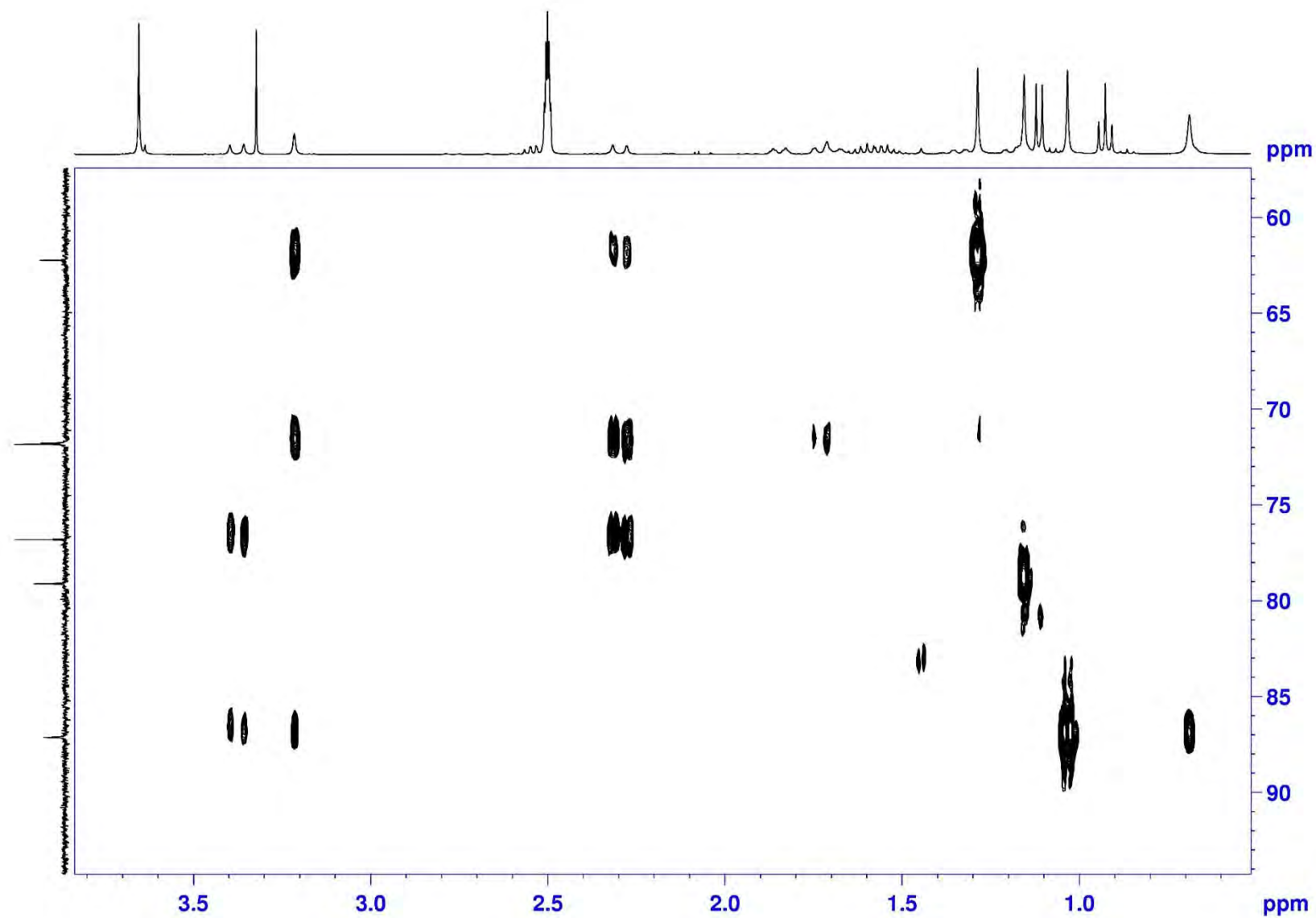
HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



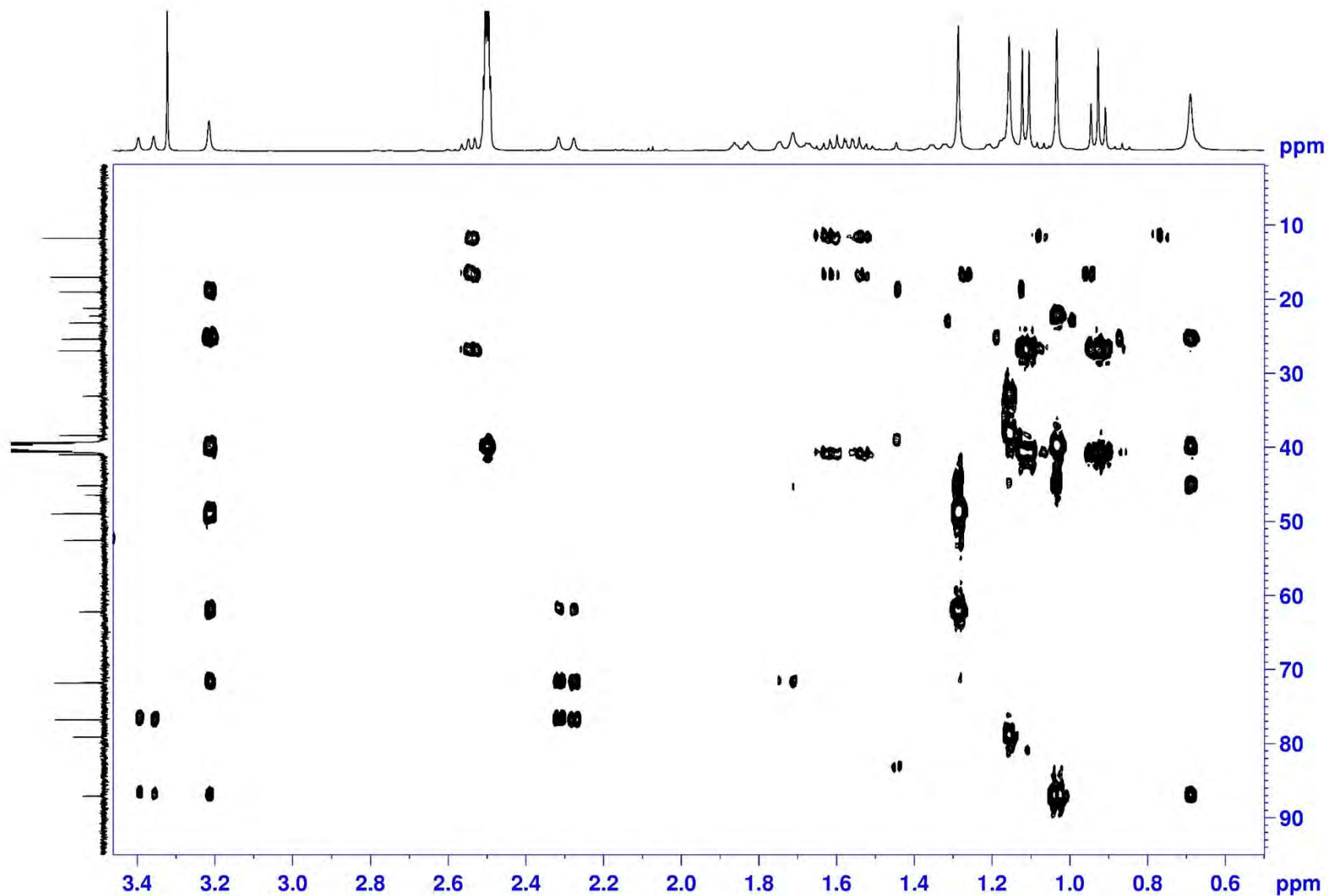
HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



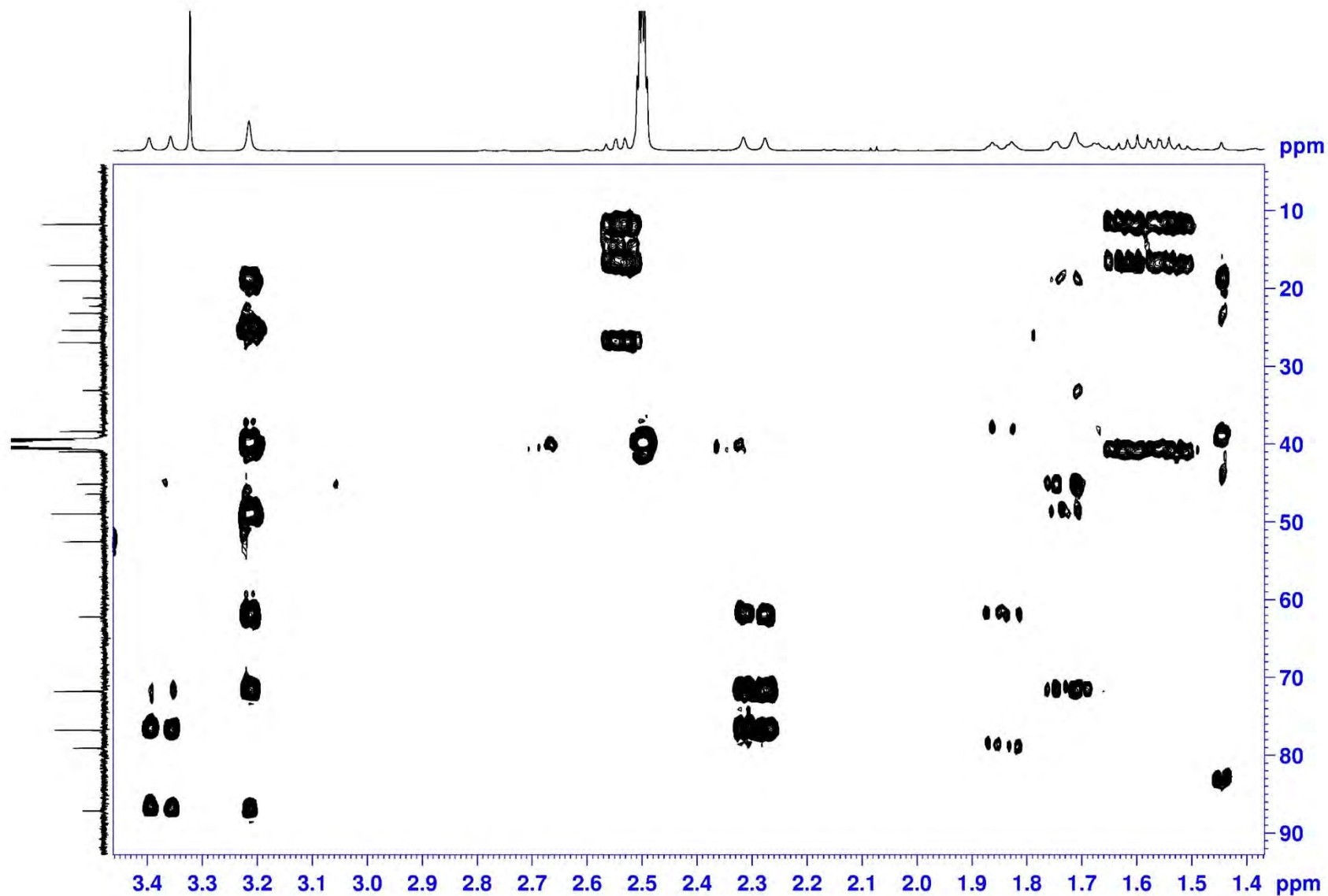
HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



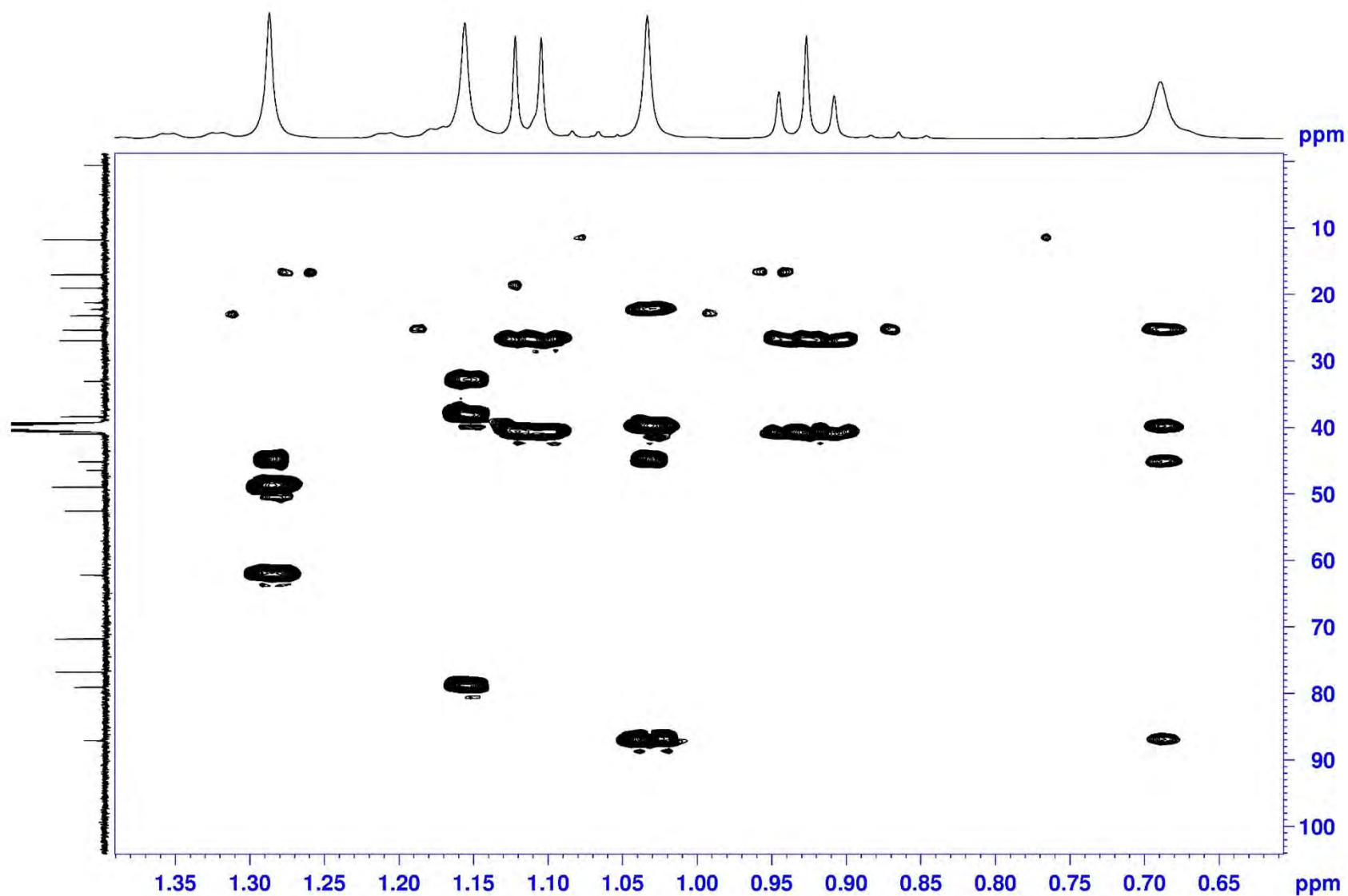
HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

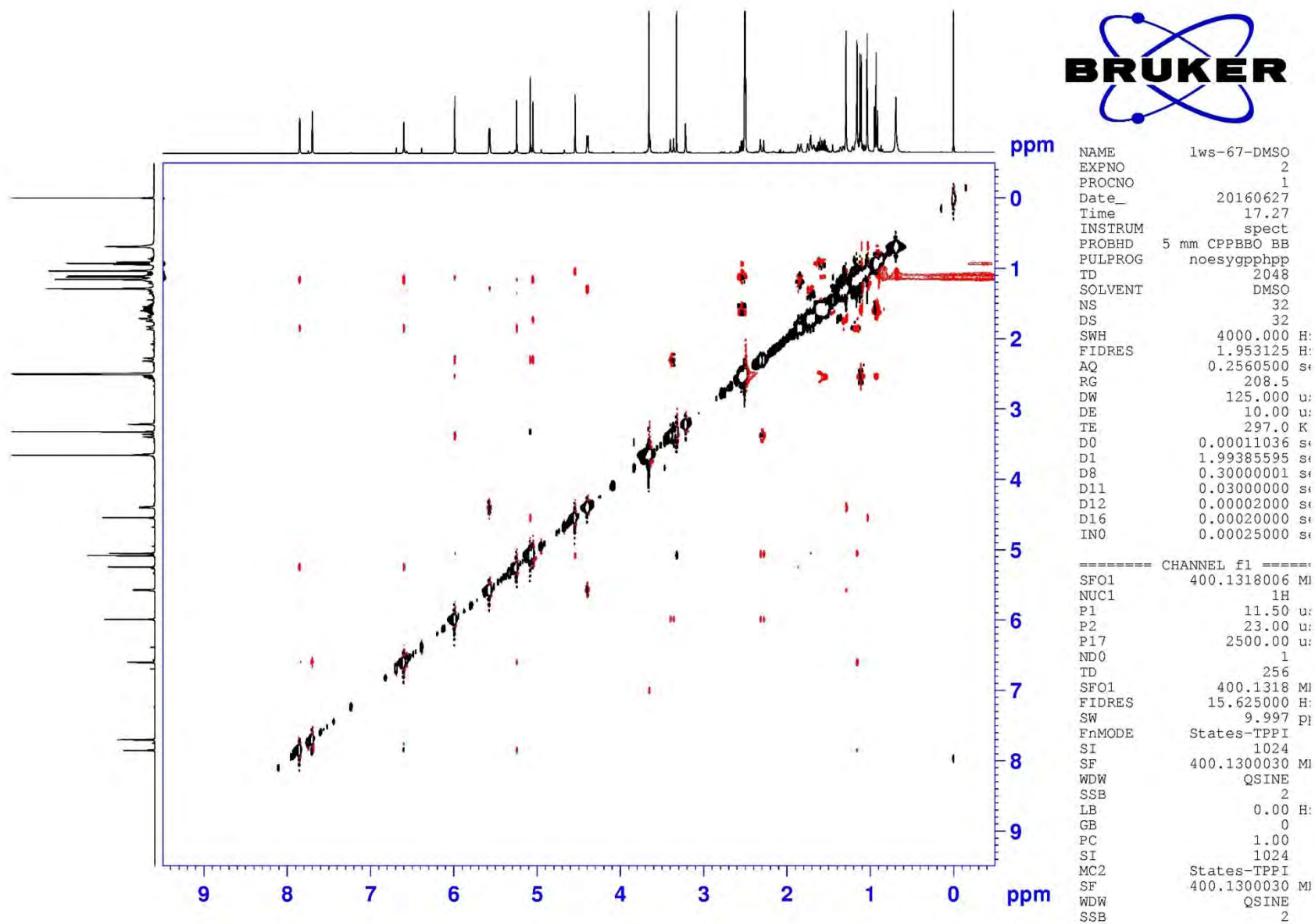


HMBC (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>

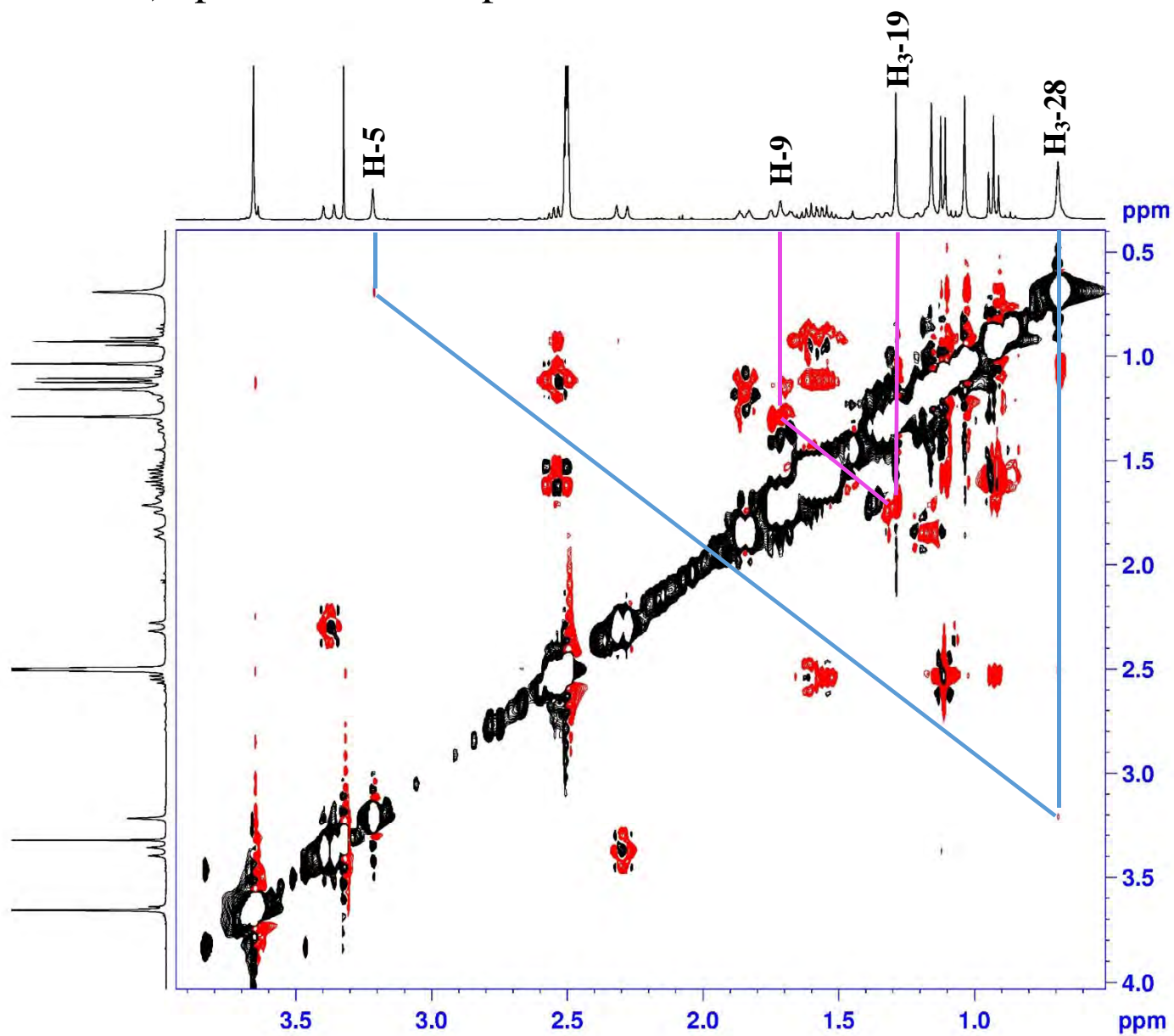




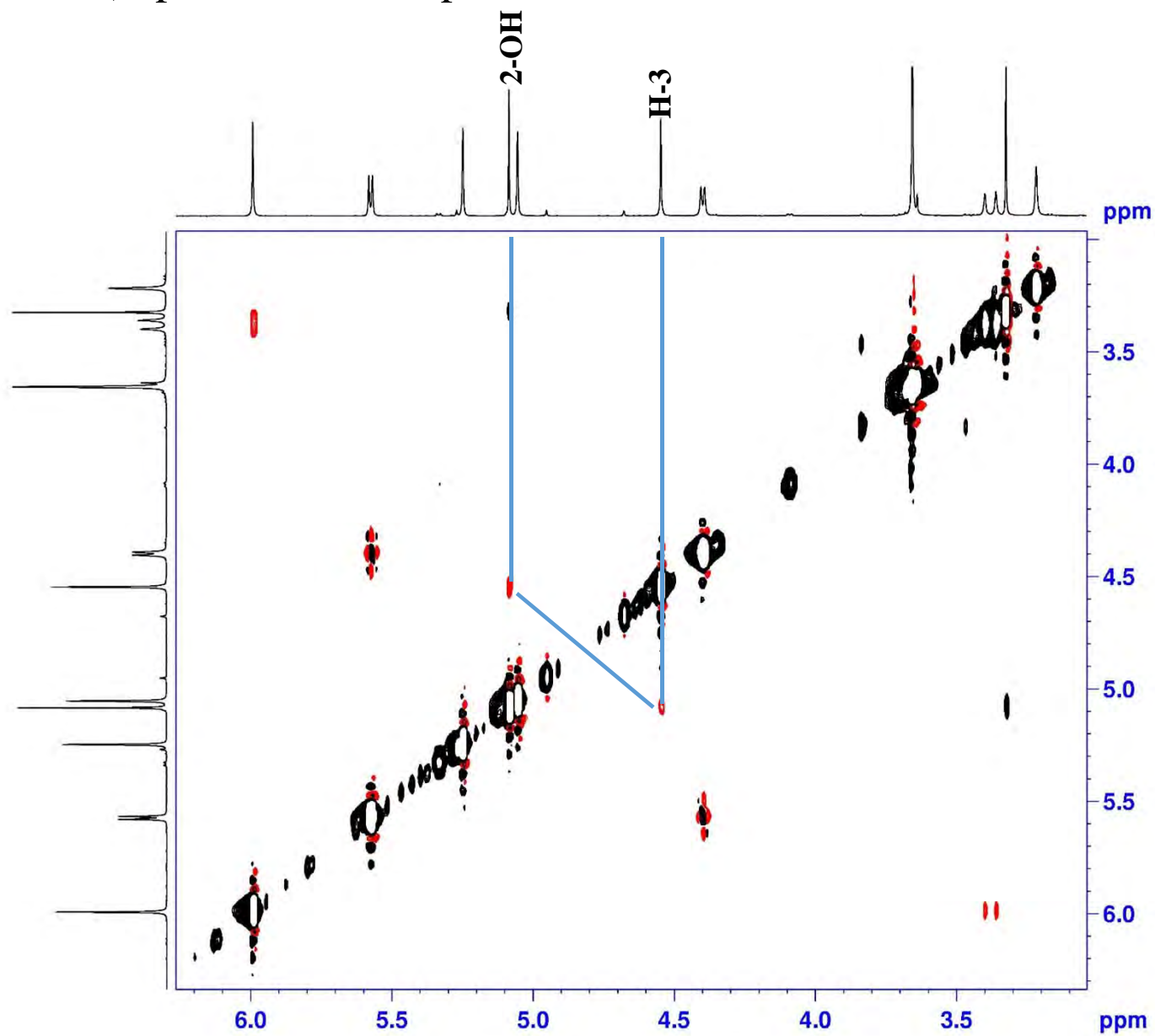
# NOESY (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



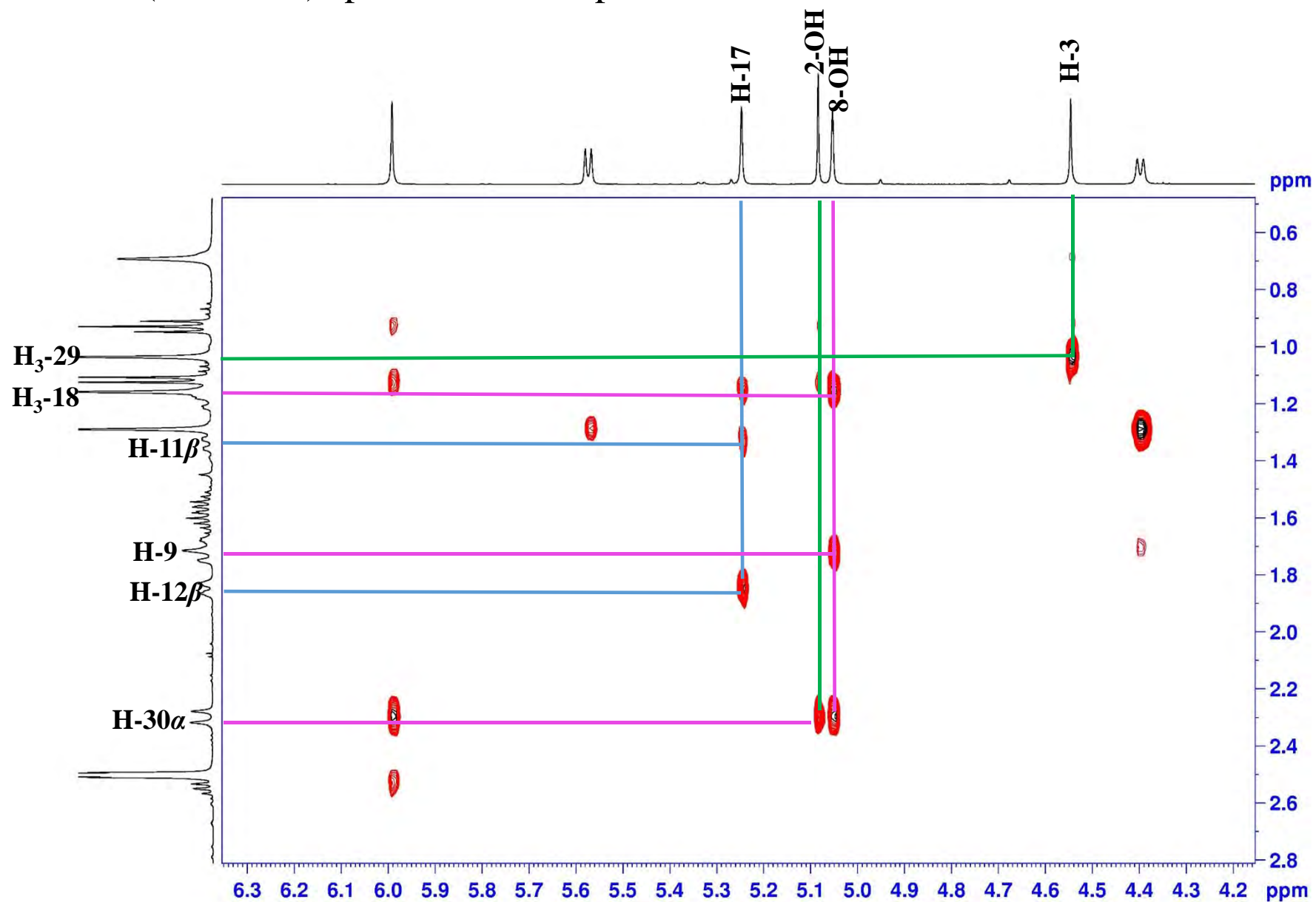
NOESY (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



NOESY (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



NOESY (400 MHz) spectrum of compound **12** in DMSO-*d*<sub>6</sub>



# HR-ESIMS for compound 13

## Mass Spectrum SmartFormula Report

### Analysis Info

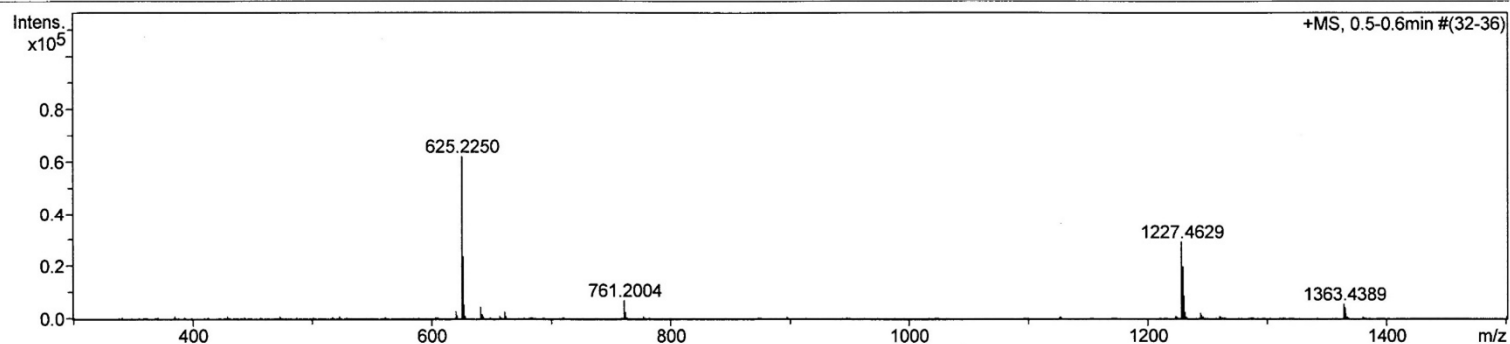
Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-74\_pos\_22\_01\_832.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_lws-74\_pos  
 Comment

Acquisition Date 11/18/2015 10:03:37 PM

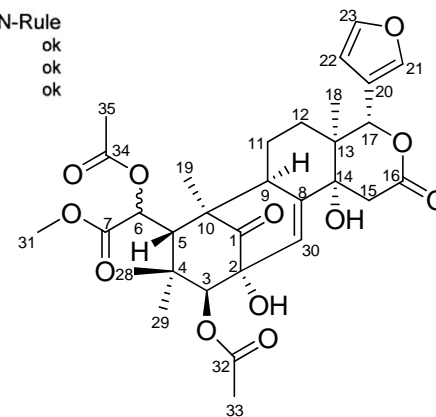
Operator SCSIO  
 Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
603.2433	1	C 31 H 39 O 12	100.00	603.2436	0.5	0.3	46.5	12.5	even	ok
625.2250	1	C 31 H 38 Na O 12	100.00	625.2255	0.9	0.5	20.2	12.5	even	ok
1227.4629	1	C 62 H 76 Na O 24	100.00	1227.4619	-0.9	-1.1	8.0	24.5	even	ok



# HR-ESIMS for compound 13

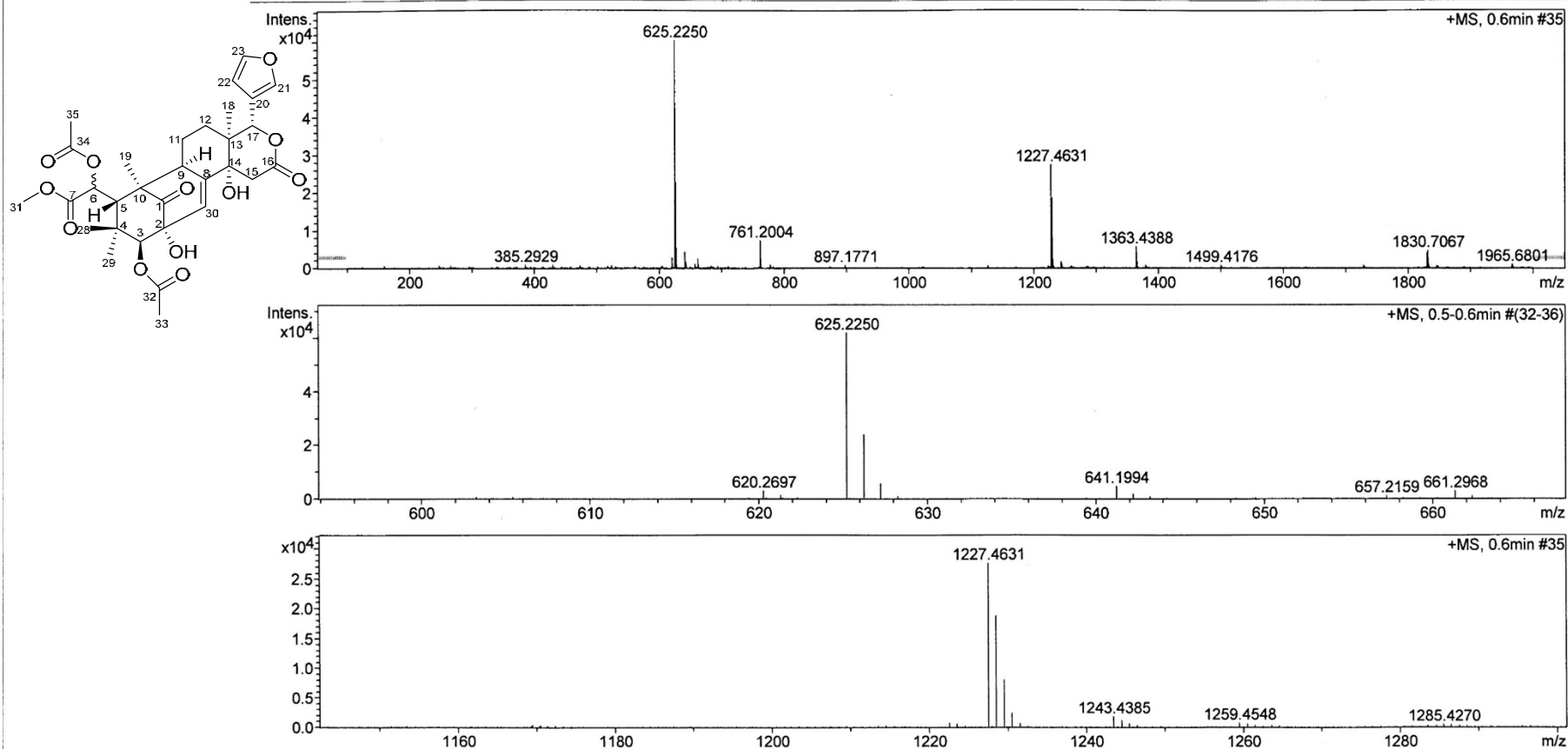
## Generic Display Report

### Analysis Info

Analysis Name D:\Data\MS\data\201511\liwanshan\_lws-74\_pos\_22\_01\_832.d  
Method LC\_Direct Infusion\_pos\_100-1000mz.m  
Sample Name liwanshan\_lws-74\_pos  
Comment

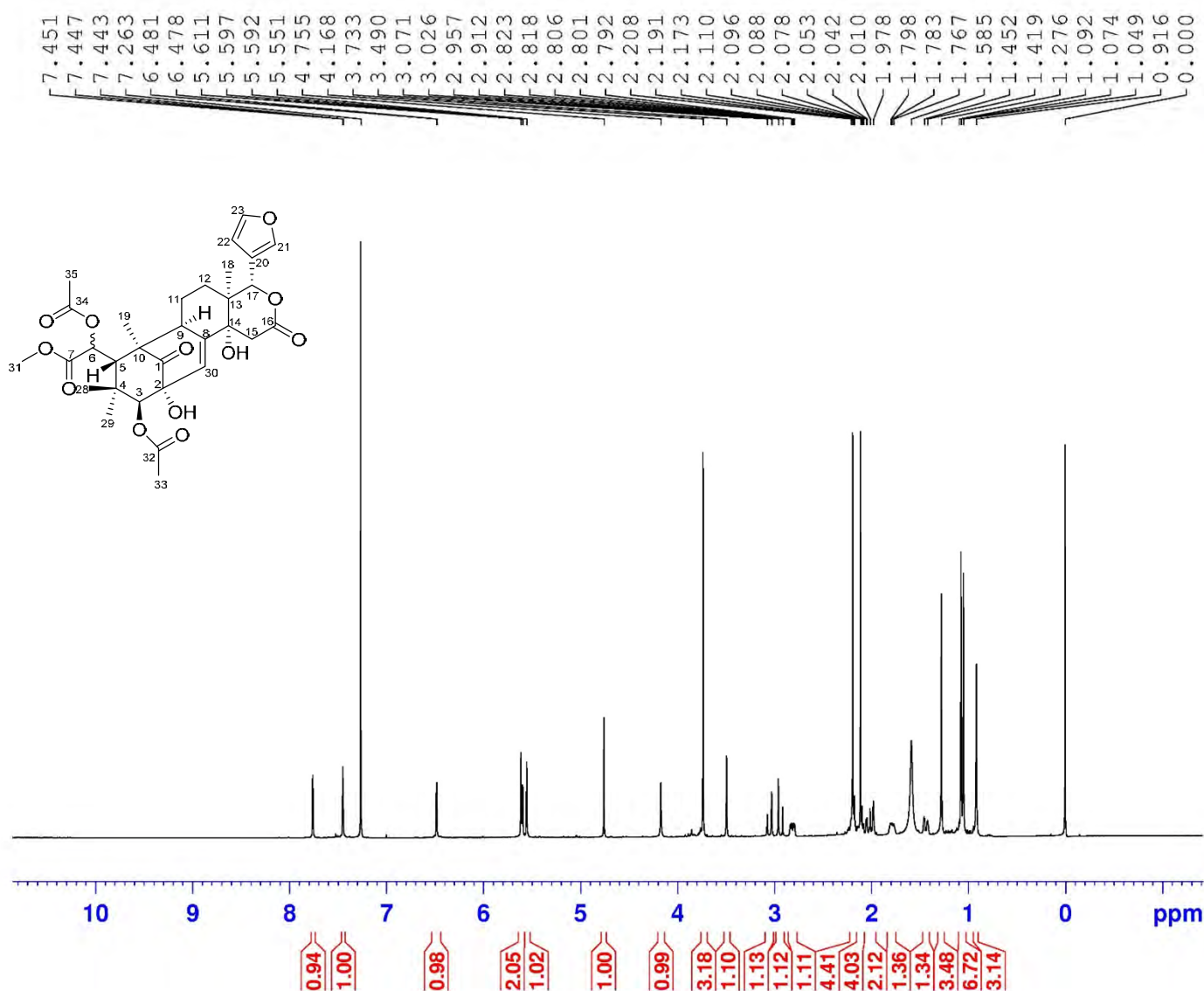
Acquisition Date 11/18/2015 10:03:37 PM

Operator SCSIO  
Instrument maXis





# <sup>1</sup>H NMR (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>

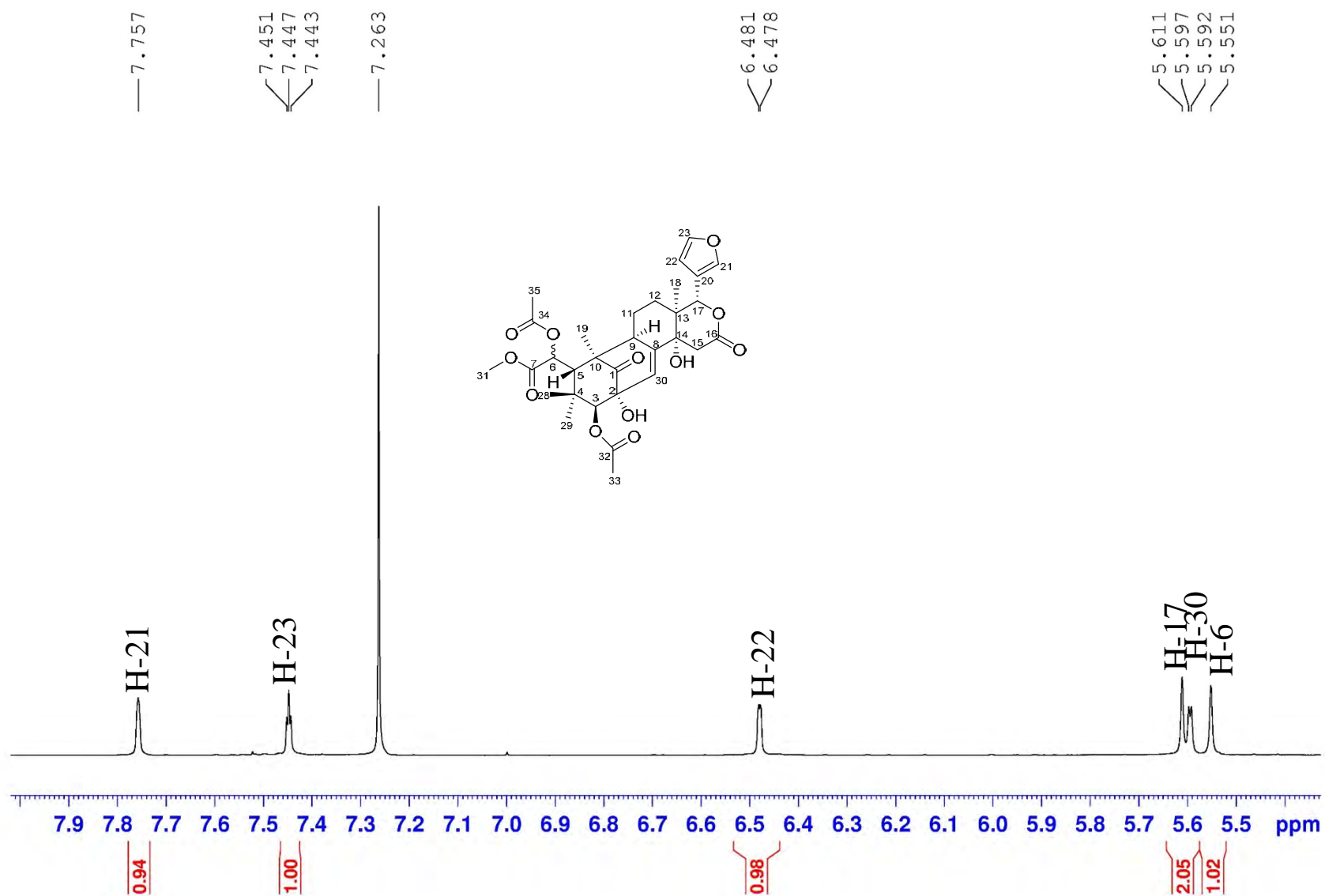


```

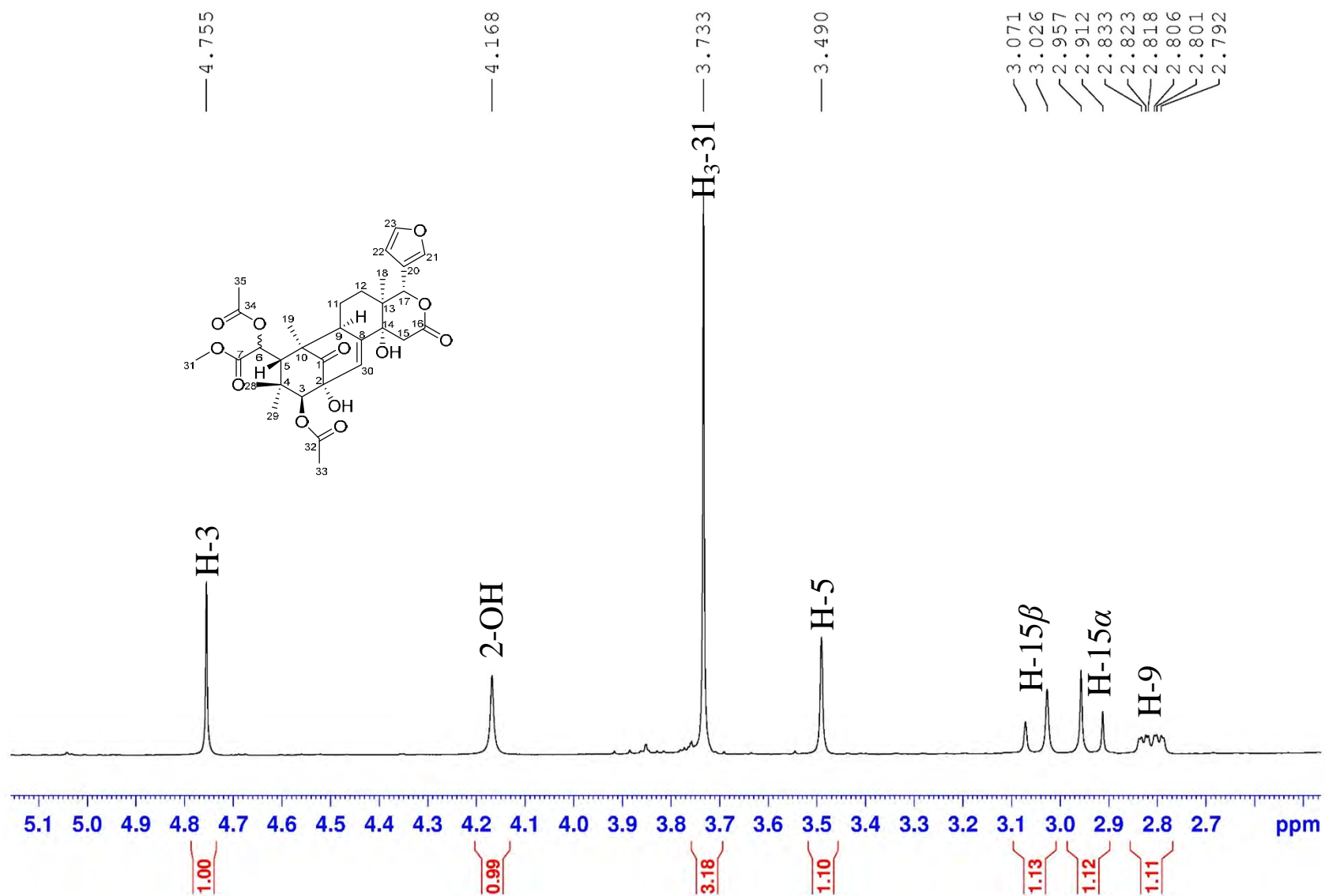
NAME                lws-74
EXPNO                1
PROCNO              1
Date_               20150906
Time                16.25
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             zg30
TD                  65536
SOLVENT             CDCl3
NS                   32
DS                   2
SWH                  8223.685
FIDRES              0.125483
AQ                  3.9846387
RG                   208.5
DW                   60.800
DE                   10.00
TE                   297.0
D1                   1.00000000
TD0                  1

===== CHANNEL f1 =====
SF01                400.1324710
NUC1                 1H
P1                   12.00
SI                   65536
SF                   400.1300087
WDW                  EM
SSB                   0
LB                   0.30
GB                   0
PC                    1.00
    
```

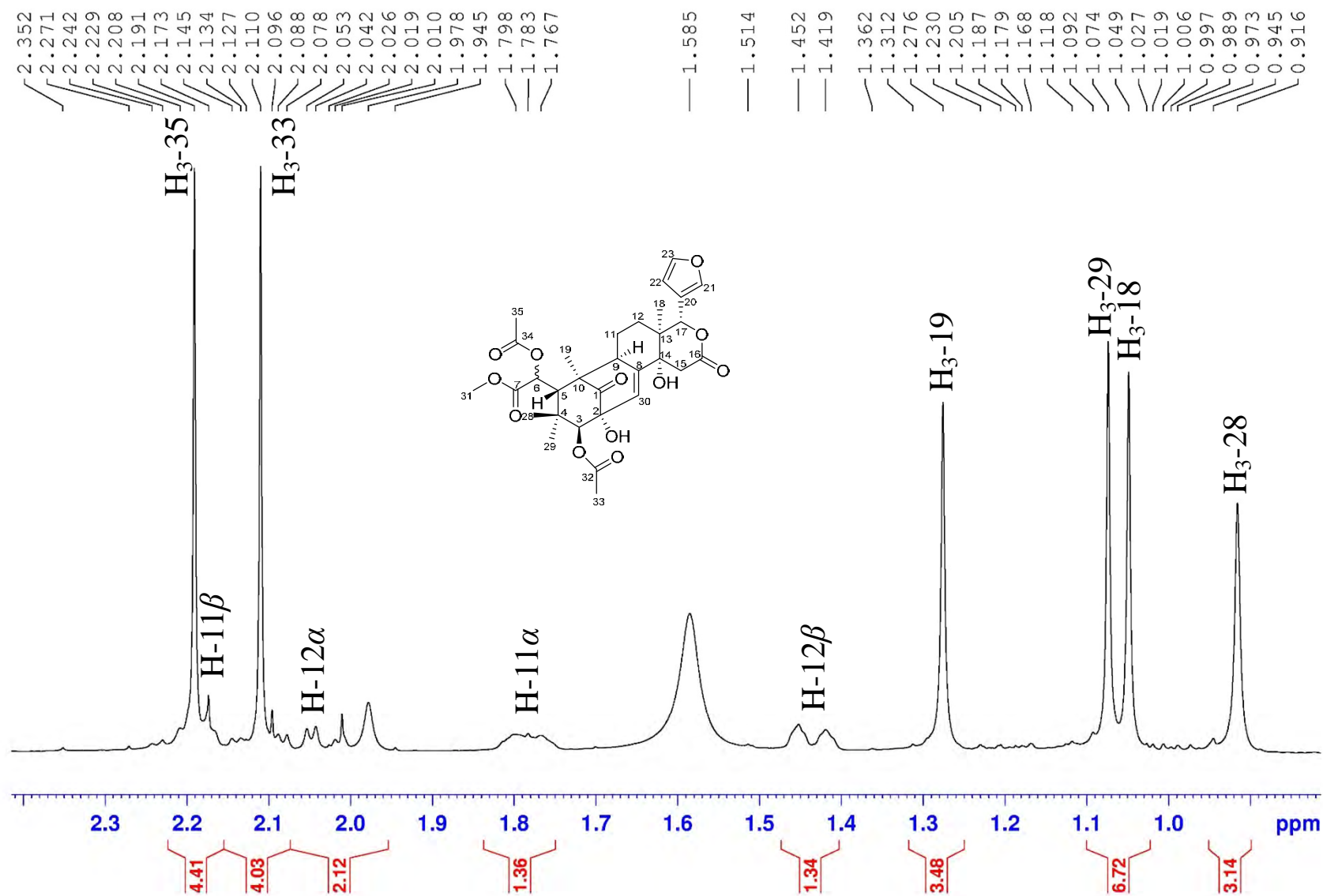
$^1\text{H}$  NMR (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



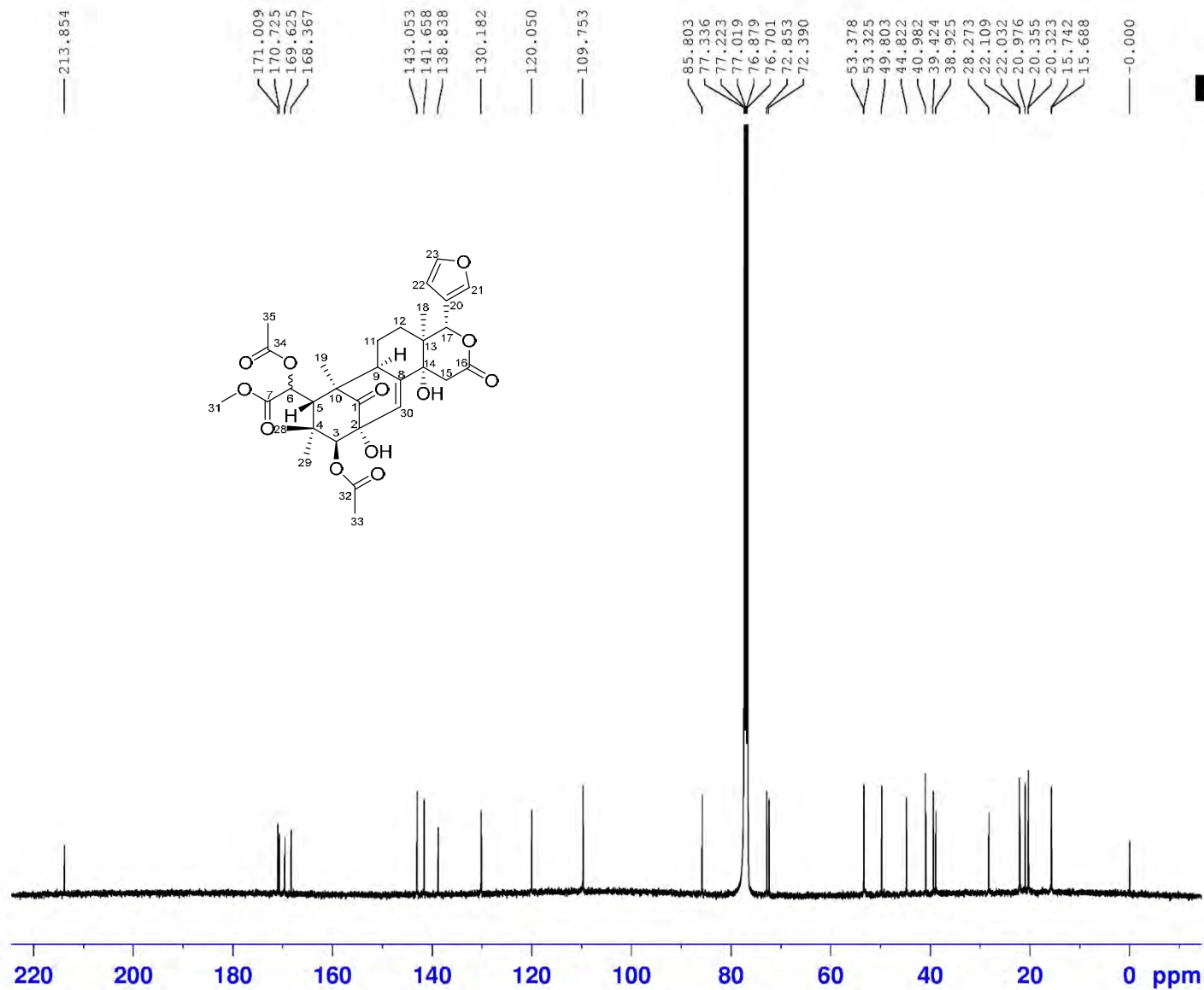
$^1\text{H}$  NMR (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



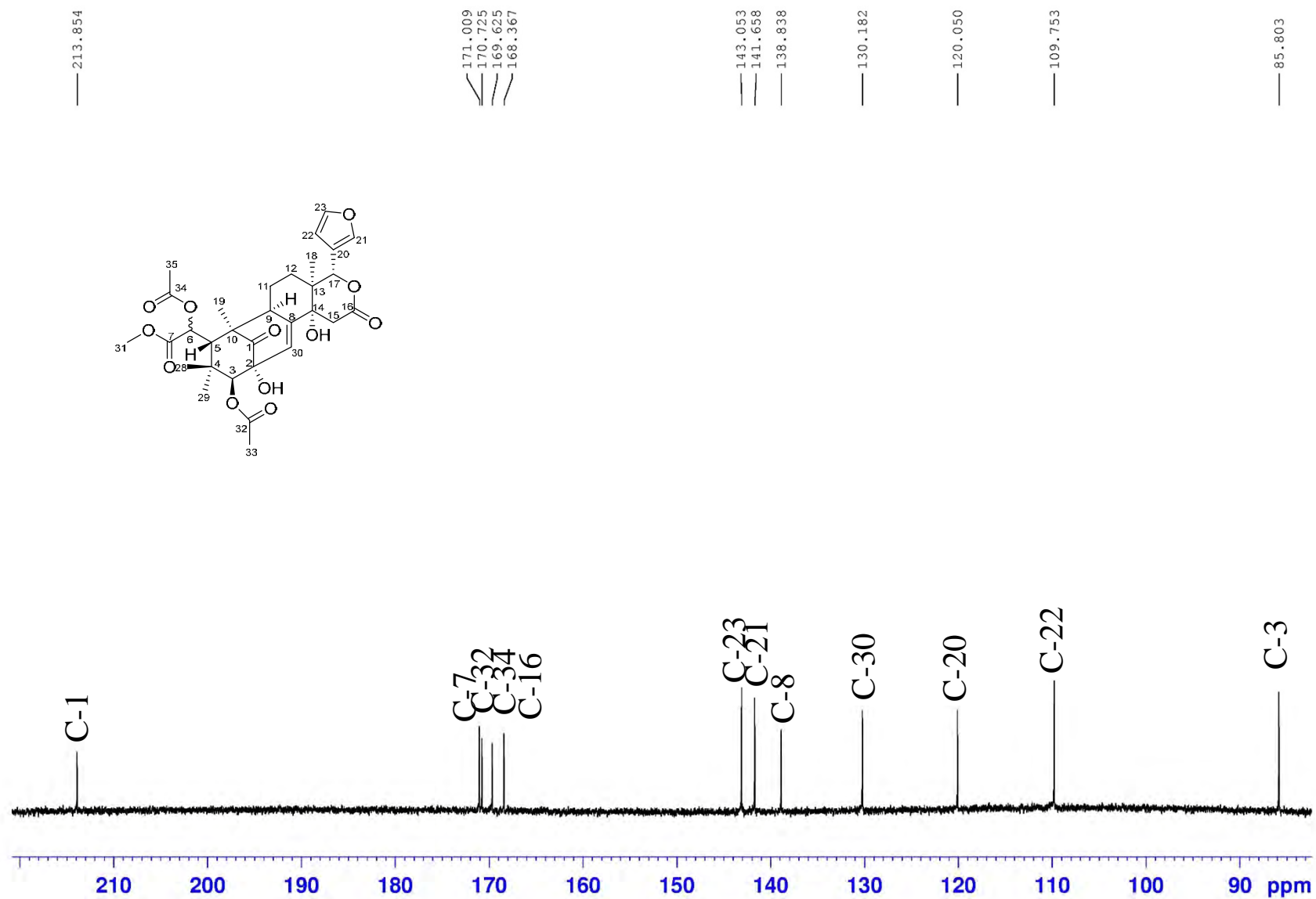
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



NAME lws-74  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20150907  
 Time 19.08  
 INSTRUM spect  
 PROBHD 5 mm CPPBBO BB  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT  $\text{CDCl}_3$   
 NS 10000  
 DS 4  
 SWH 24038.461 F  
 FIDRES 0.366798 F  
 AQ 1.3631988 s  
 RG 147.94  
 DW 20.800 u  
 DE 18.00 u  
 TE 297.0 K  
 D1 2.00000000 s  
 D11 0.03000000 s  
 TD0 1

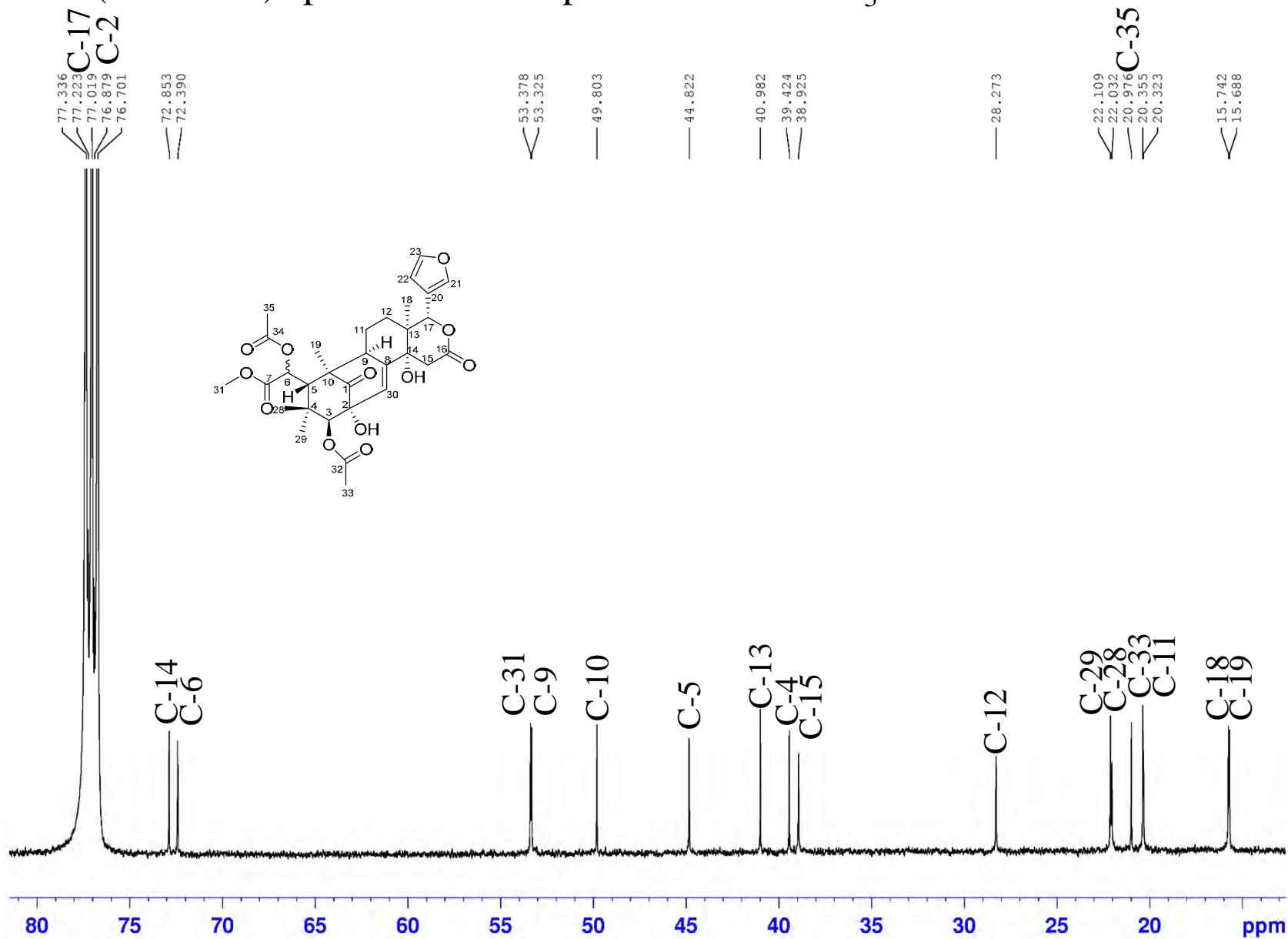
===== CHANNEL f1 =====  
 SFO1 100.6233324 M  
 NUC1  $^{13}\text{C}$   
 P1 10.00 u  
 SI 32768  
 SF 100.6127692 M  
 WDW EM  
 SSB 0  
 LB 1.00 F  
 GB 0  
 PC 1.40

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

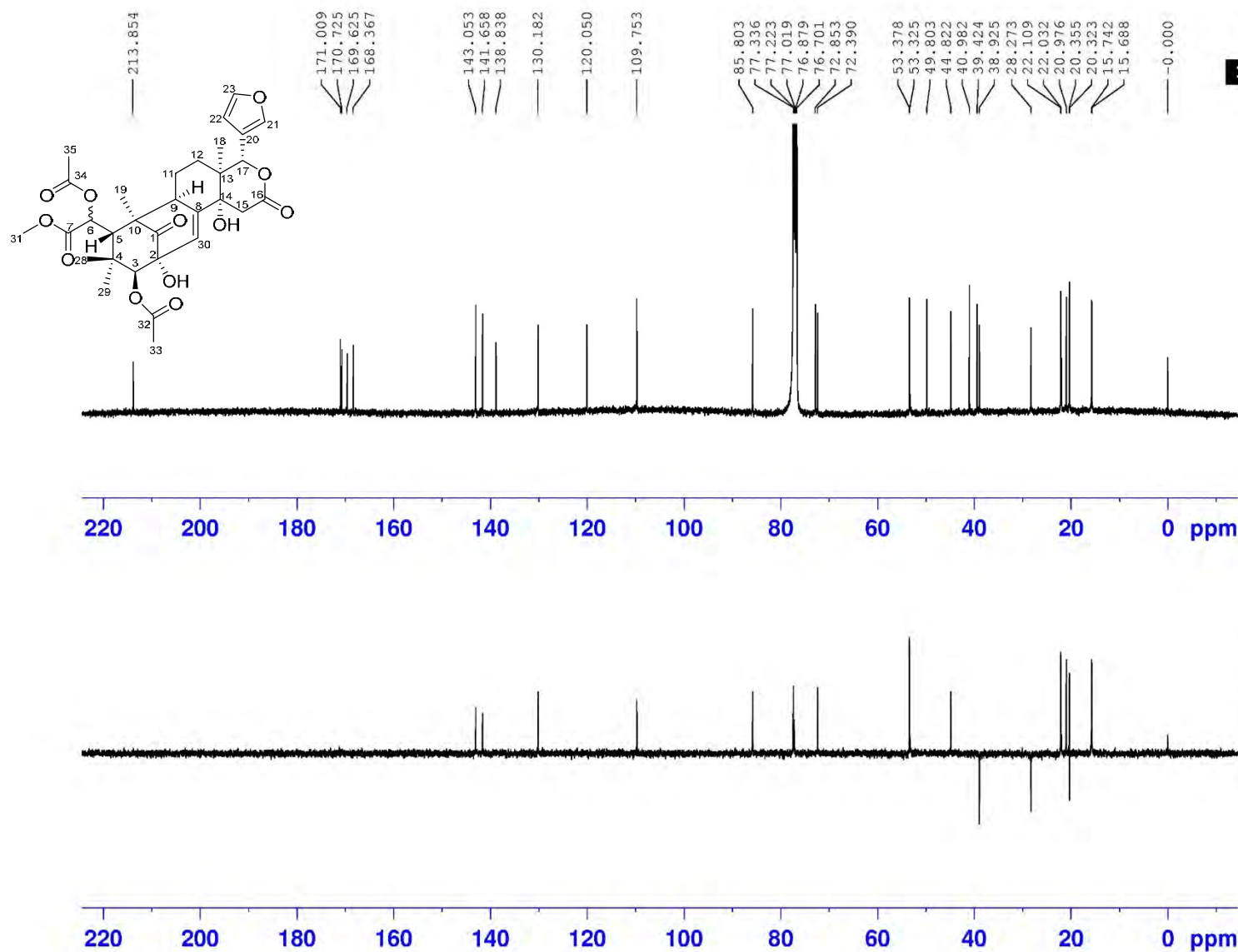




$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



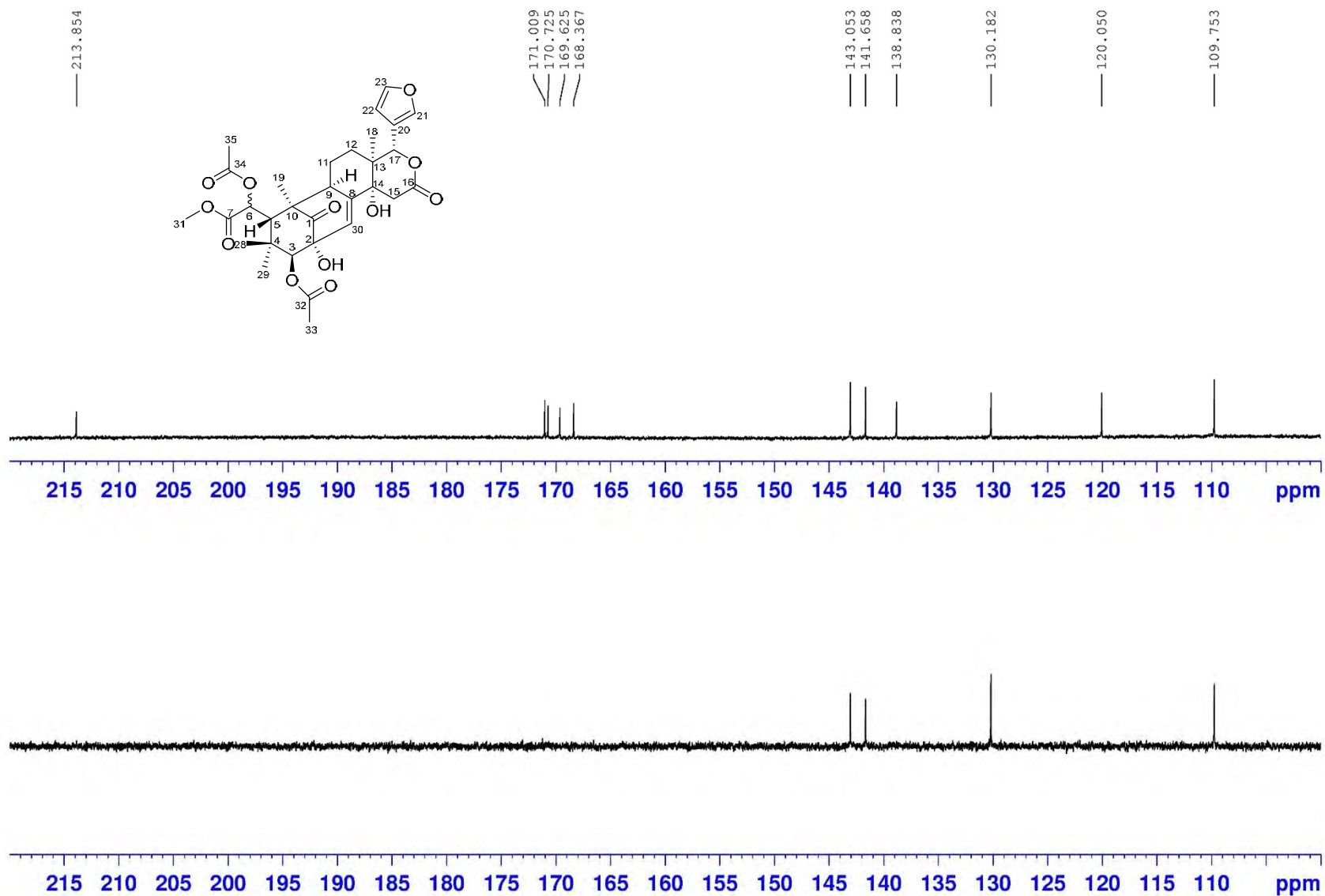
```

NAME          lws-74
EXPNO          3
PROCNO         1
Date_          20150907
Time           21.31
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        deptspl35
TD             65536
SOLVENT        CDCl3
NS             2500
DS             4
SWH            24038.461
FIDRES         0.366798
AQ            1.3631988
RG            102.3
DW            20.800
DE            18.00
TE            297.0
CNST2          145.0000000
D1             2.00000000
D2             0.00344828
D12            0.00002000
TD0            1
  
```

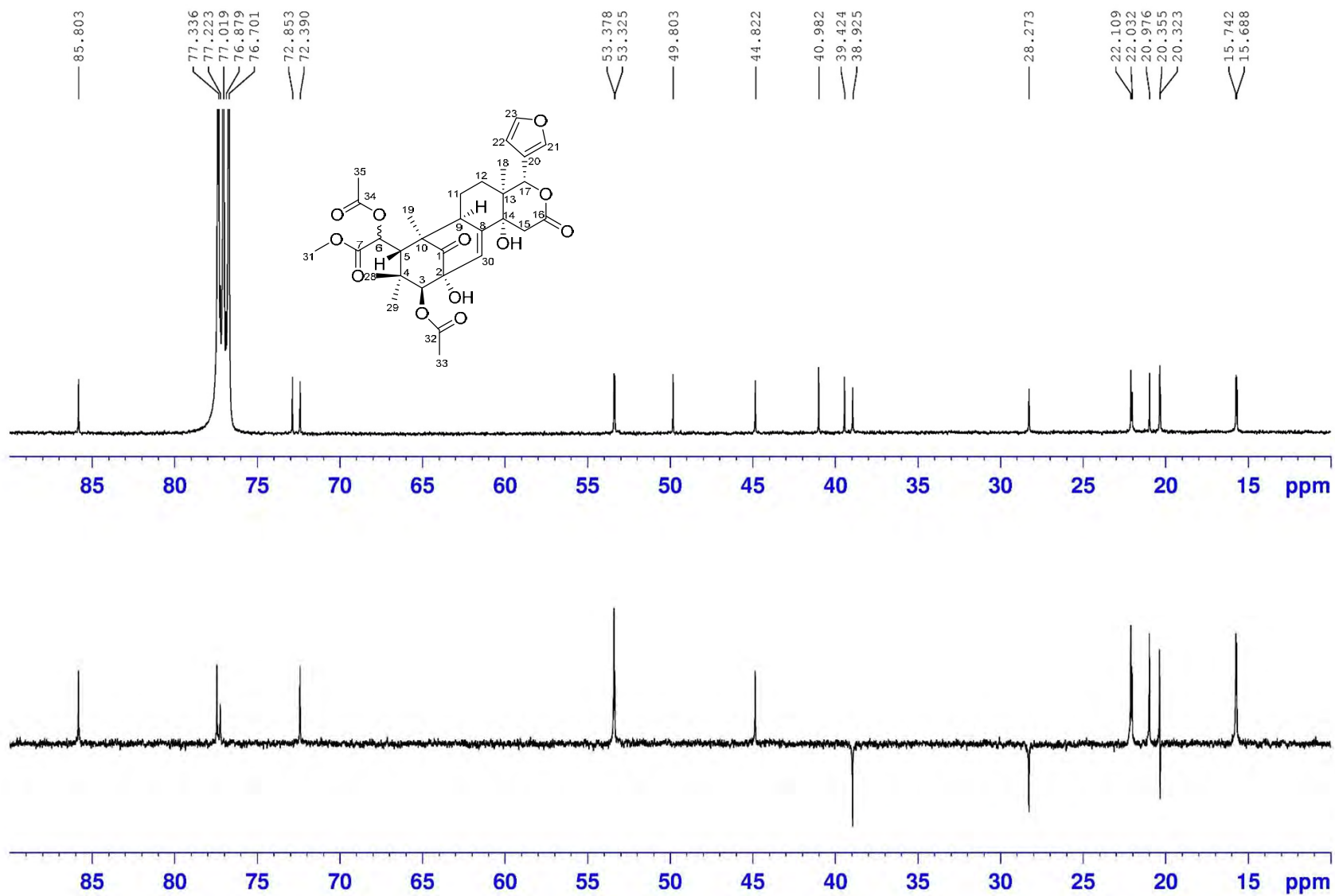
```

===== CHANNEL f1 =====
SFO1          100.6233324
NUC1           13C
P1             10.00
P13            2000.00
SI            32768
SF            100.6127694
WDW            EM
SSB            0
LB             1.00
GB             0
PC             1.40
  
```

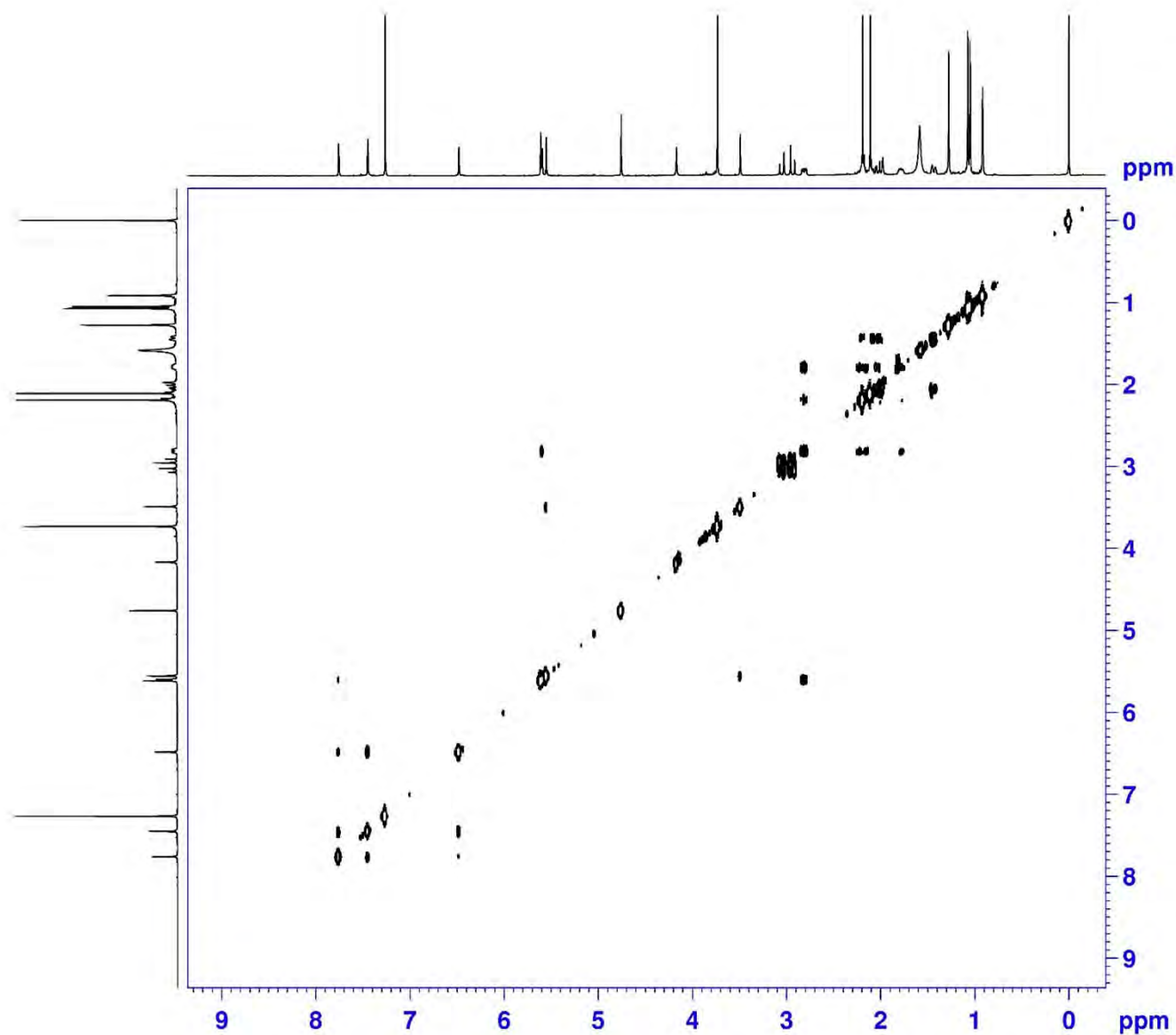
# DEPT135 (100 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



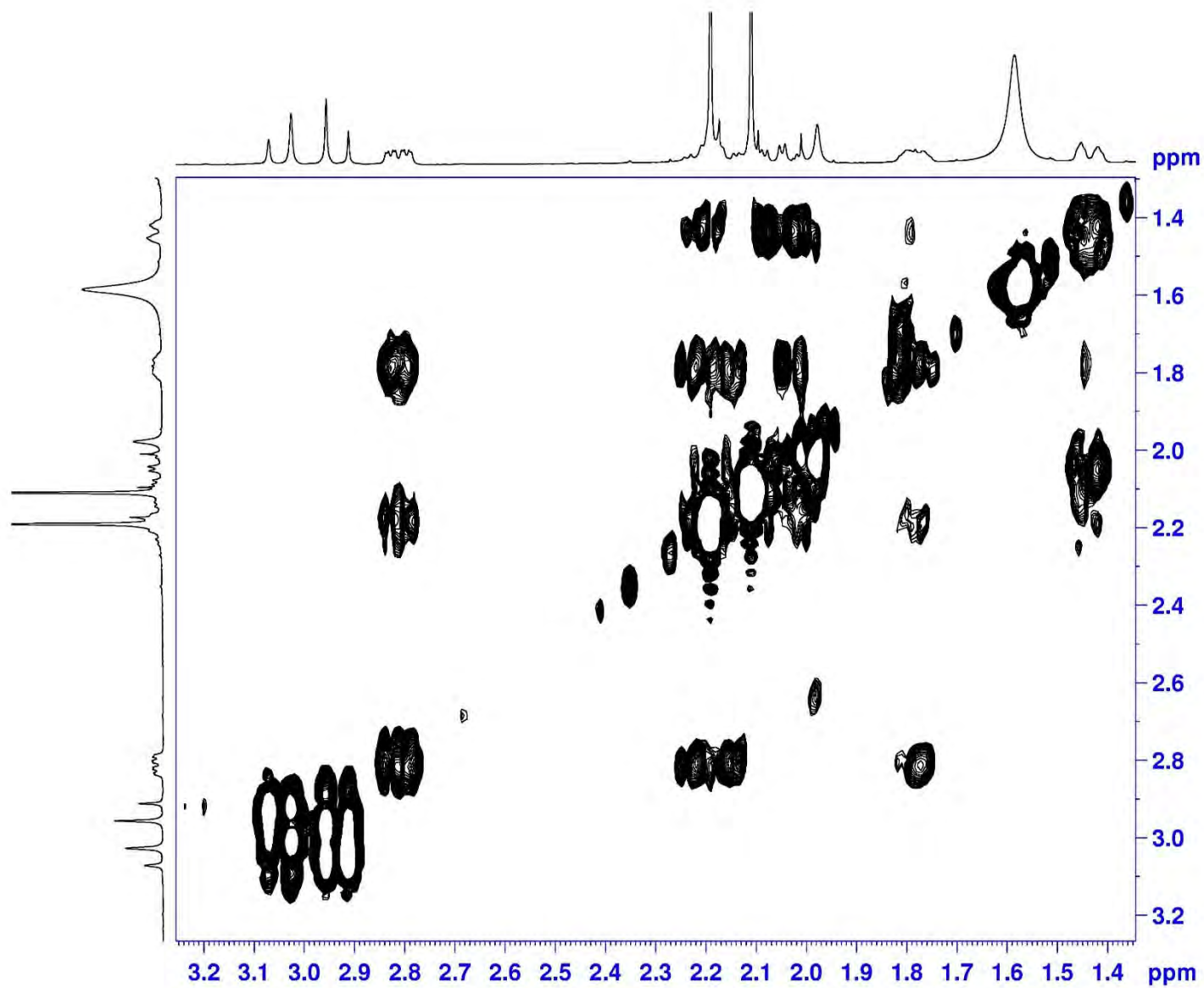
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



NAME lws-74  
EXPNO 104  
PROCNO 1  
Date\_ 20150917  
Time 17.36  
INSTRUM spect  
PROBHD 5 mm CFPBBO BB  
PULPROG cosygpppqf  
TD 2048  
SOLVENT  $\text{CDCl}_3$   
NS 16  
DS 8  
SWH 3906.250 H  
FIDRES 1.907349 H  
AQ 0.2621940 s  
RG 208.5  
DW 128.000 u  
DE 10.00 u  
TE 297.0 K  
D0 0.00000300 s  
D1 1.89678097 s  
D11 0.03000000 s  
D12 0.00002000 s  
D13 0.00000400 s  
D16 0.00020000 s  
IN0 0.00025600 s

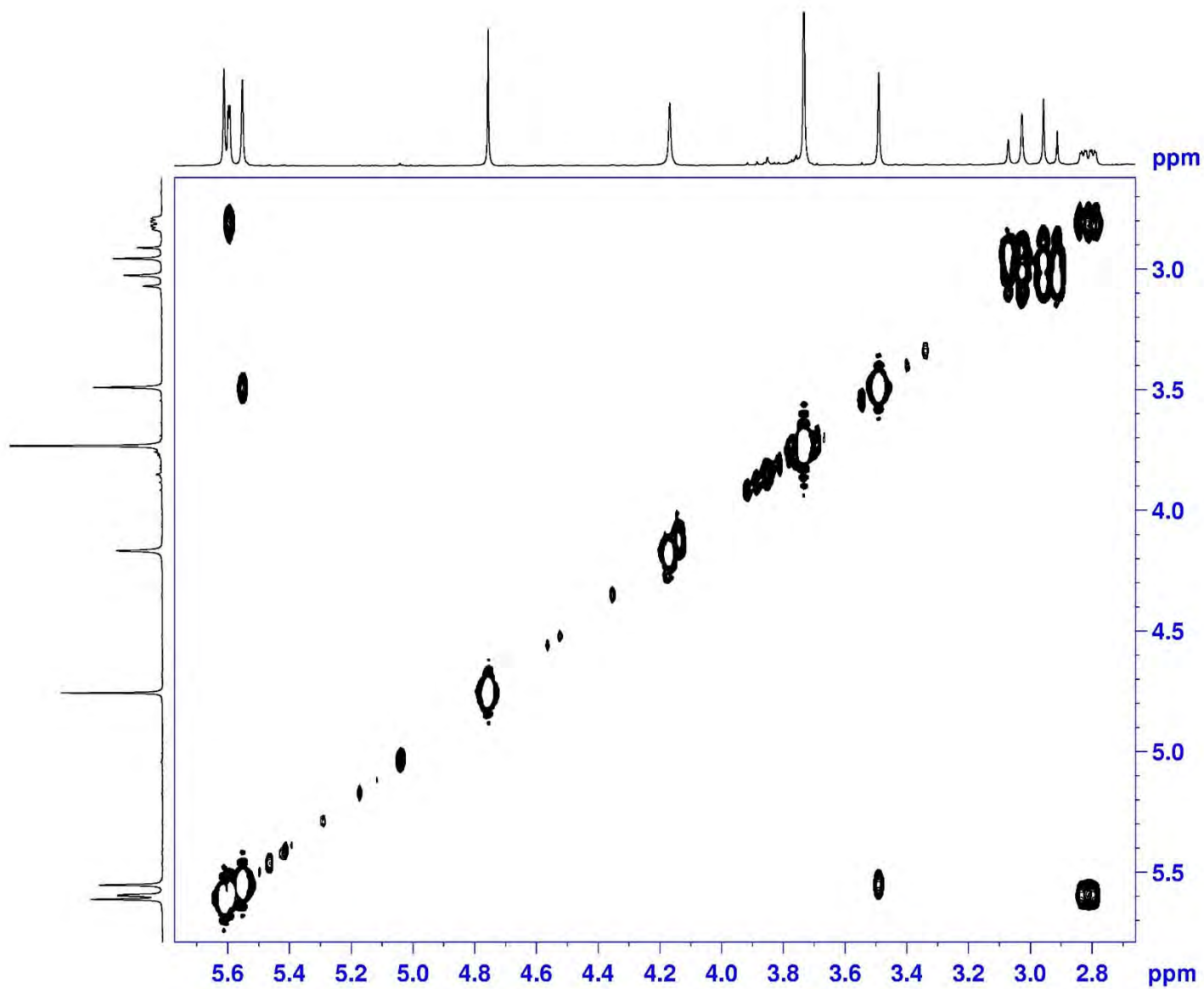
===== CHANNEL f1 =====  
SFO1 400.1318006 M  
NUC1  $^1\text{H}$   
P0 12.00 u  
P1 12.00 u  
P17 2500.00 u  
ND0 1  
TD 128  
SFO1 400.1318 M  
FIDRES 30.517578 H  
SW 9.762 p  
FrMODE QF  
SI 1024  
SF 400.1300075 M  
WDW QSINE  
SSB 0  
LB 0.00 H  
GB 0  
PC 1.40  
SI 1024  
MC2 QF  
SF 400.1300075 M  
WDW QSINE  
SSB 0  
-- --

$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$

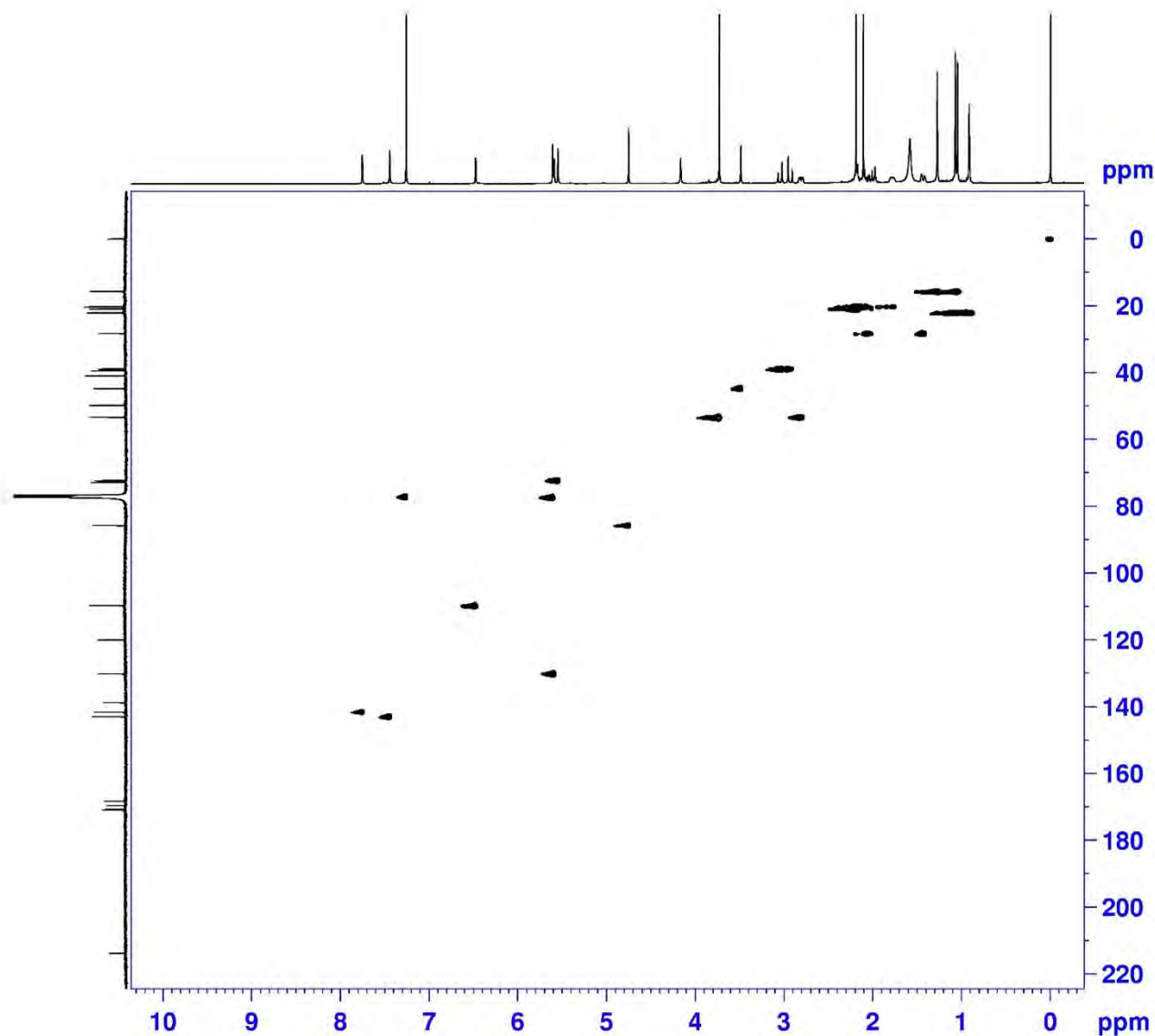




$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



# HSQC (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



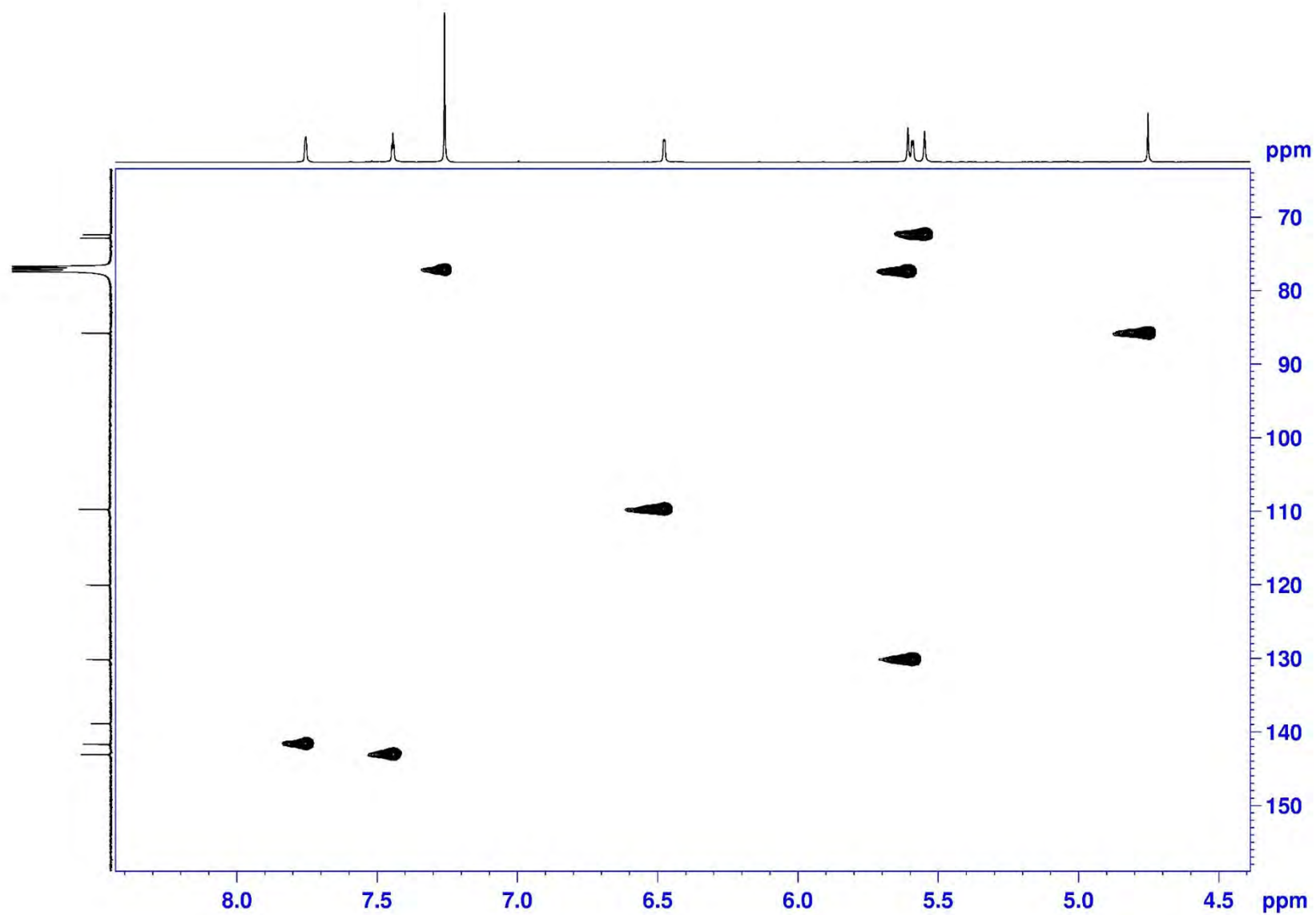
```

NAME                lws-74
EXPNO                5
PROCNO               1
Date_                20150907
Time                21.33
INSTRUM              spect
PROBHD               5 mm CPPBBO BB
PULPROG              hsqcetgpsi2
TD                   1024
SOLVENT              CDCl3
NS                   32
DS                   16
SWH                  4302.926 Hz
FIDRES               4.202076 Hz
AQ                   0.1190388 sec
RG                   208.5
DW                   116.200 usec
DE                   10.00 usec
TE                   297.0 K
CNST2                145.0000000
D0                   0.00000300 sec
D1                   1.46497905 sec
D4                   0.00172414 sec
D11                  0.03000000 sec
D16                  0.00020000 sec
D24                  0.00086207 sec
IN0                  0.00002080 sec
ZGPTNS
  
```

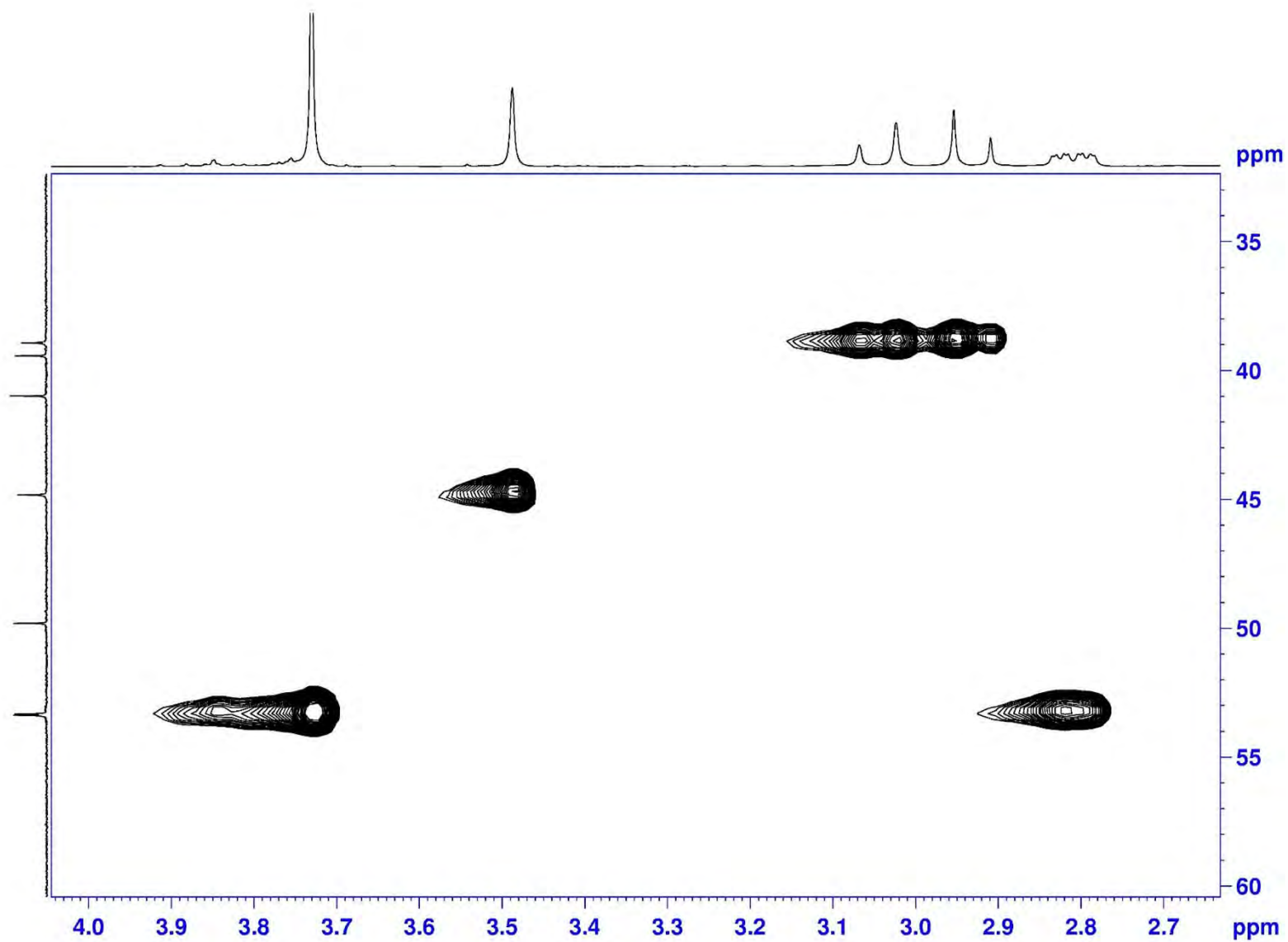
```

===== CHANNEL f1 =====
SFO1                400.1320007 MHz
NUC1                 1H
P1                   12.00 usec
P2                   24.00 usec
P28                  0.00 usec
ND0                  2
TD                   256
SFO1                100.6233 MHz
FIDRES               93.900238 Hz
SW                   238.896 ppm
FnMODE              Echo-Antiecho
SI                   1024
SF                   400.1300079 MHz
WDW                  QSINE
SSB                  2
LB                   0.00 Hz
GB                   0
PC                   1.40
SI                   1024
MC2                 echo-antiecho
SF                   100.6127685 MHz
WDW                  QSINE
  
```

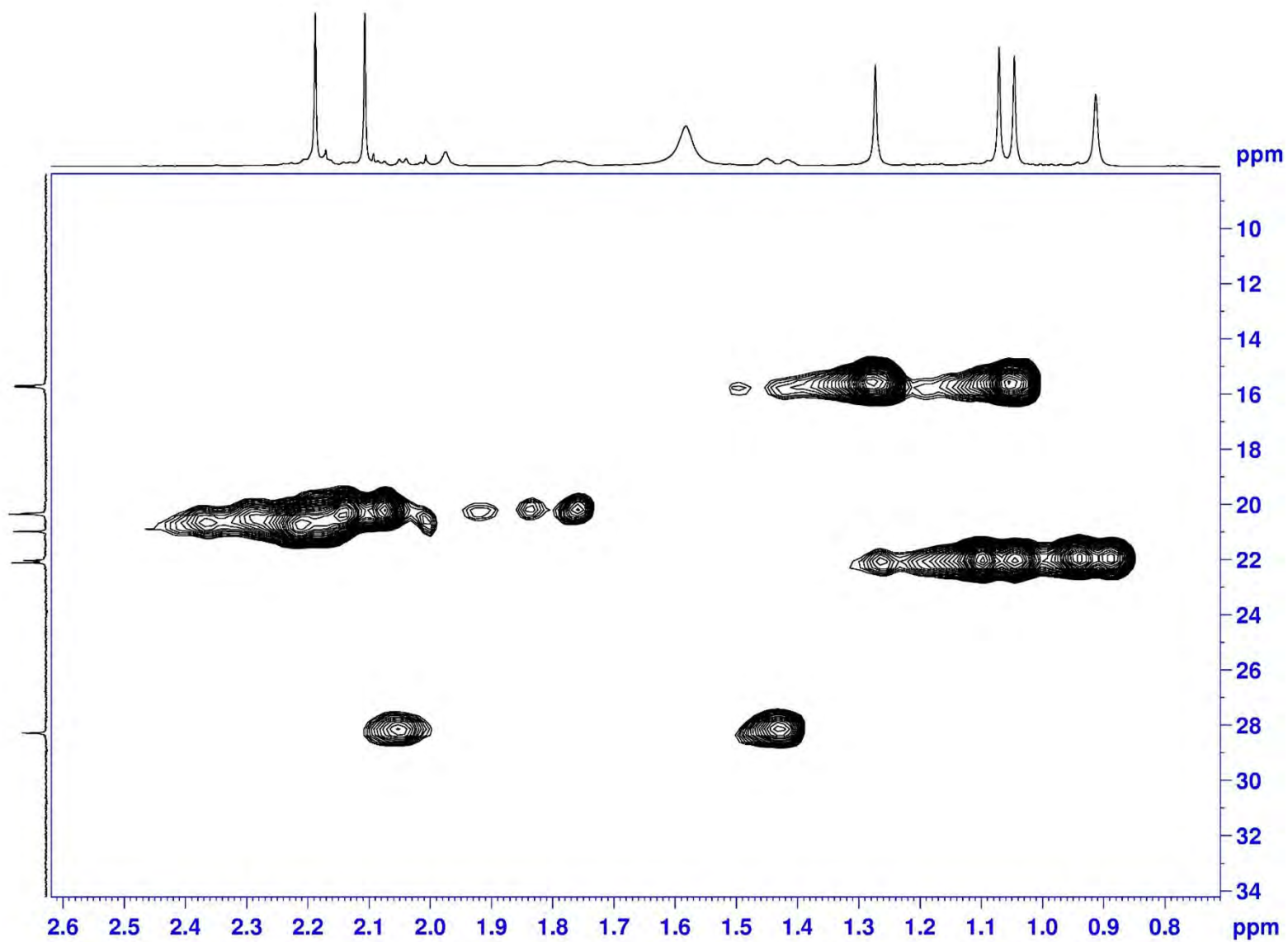
HSQC (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



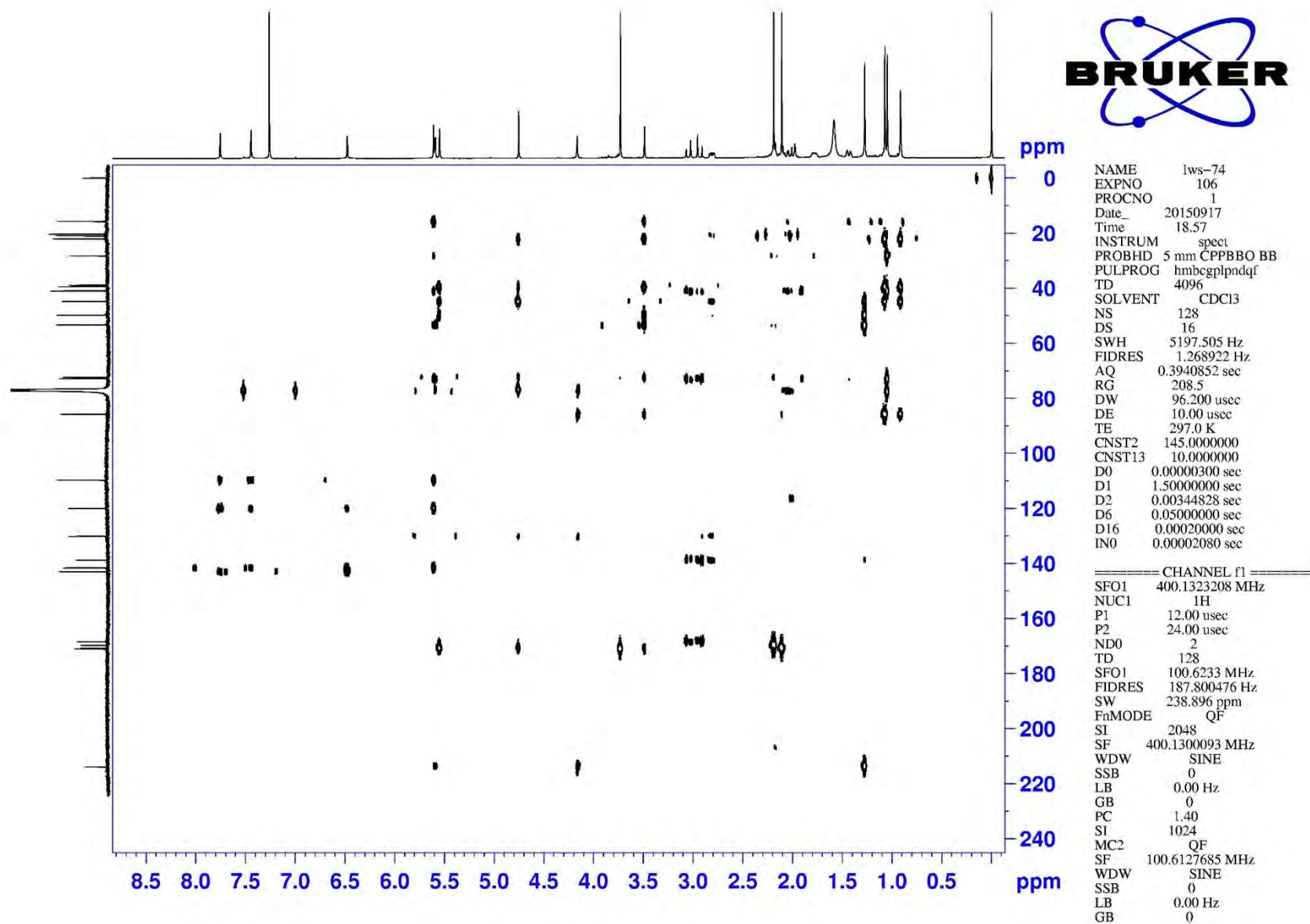
HSQC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



HSQC (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>

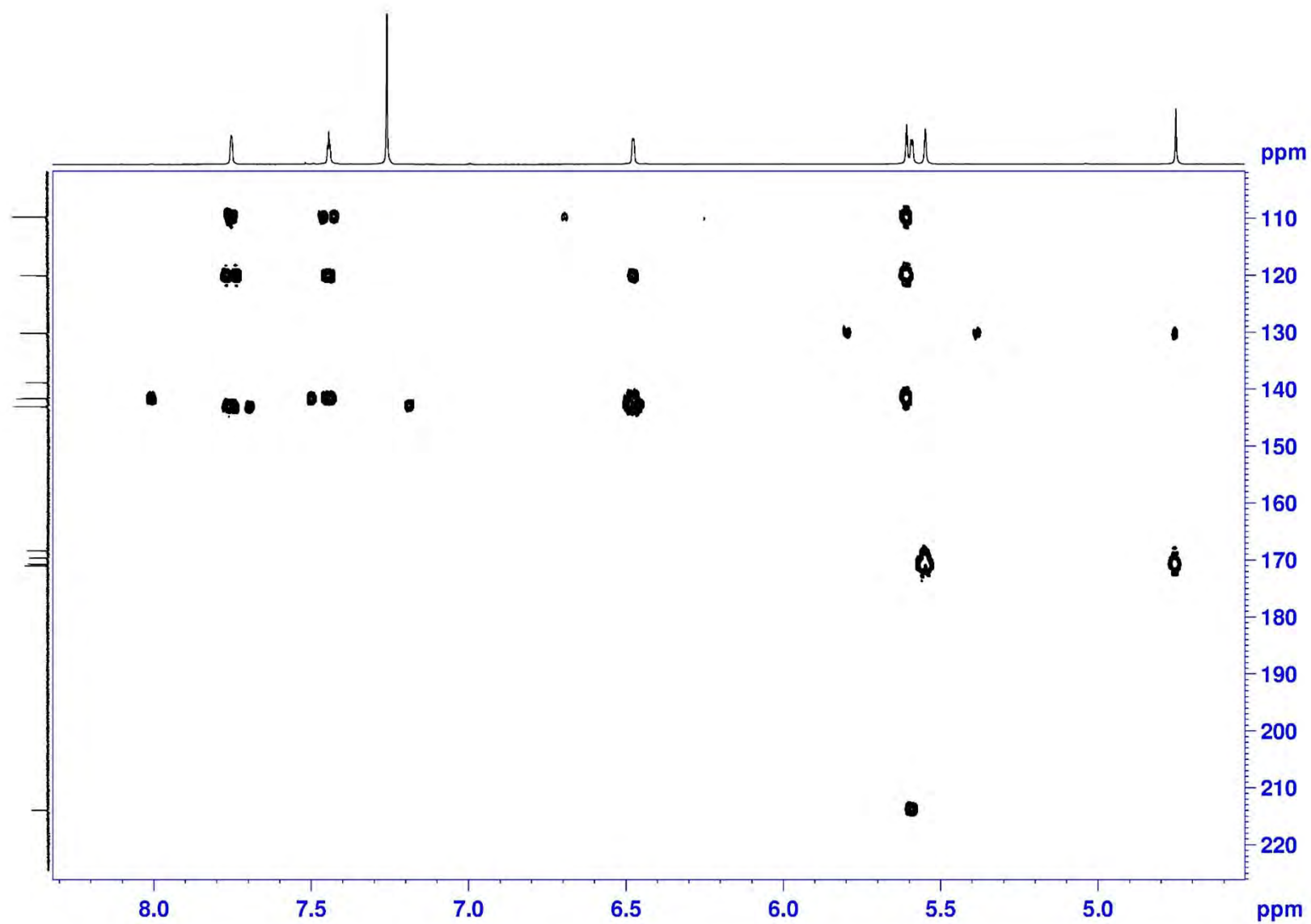


# HMBC (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>

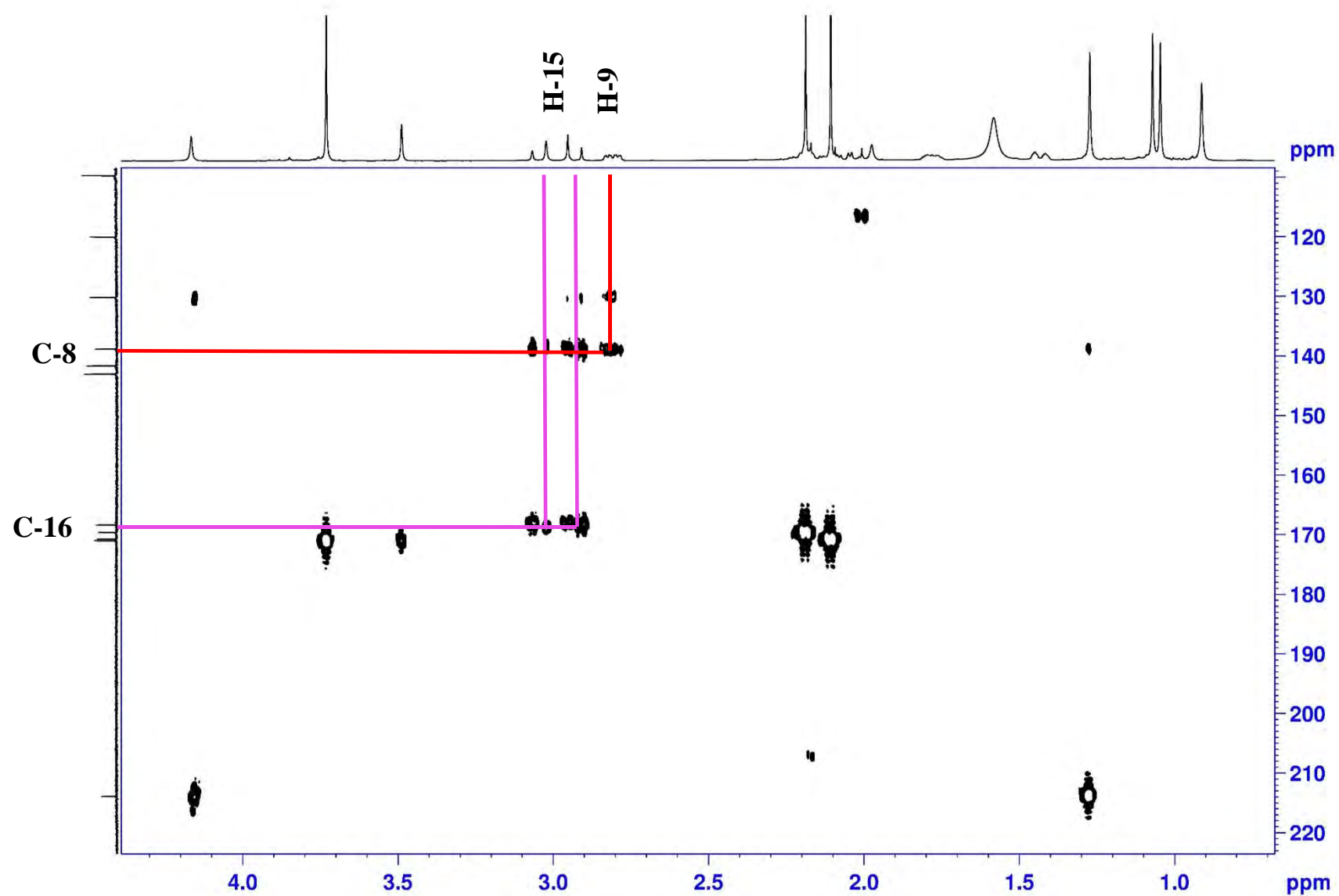




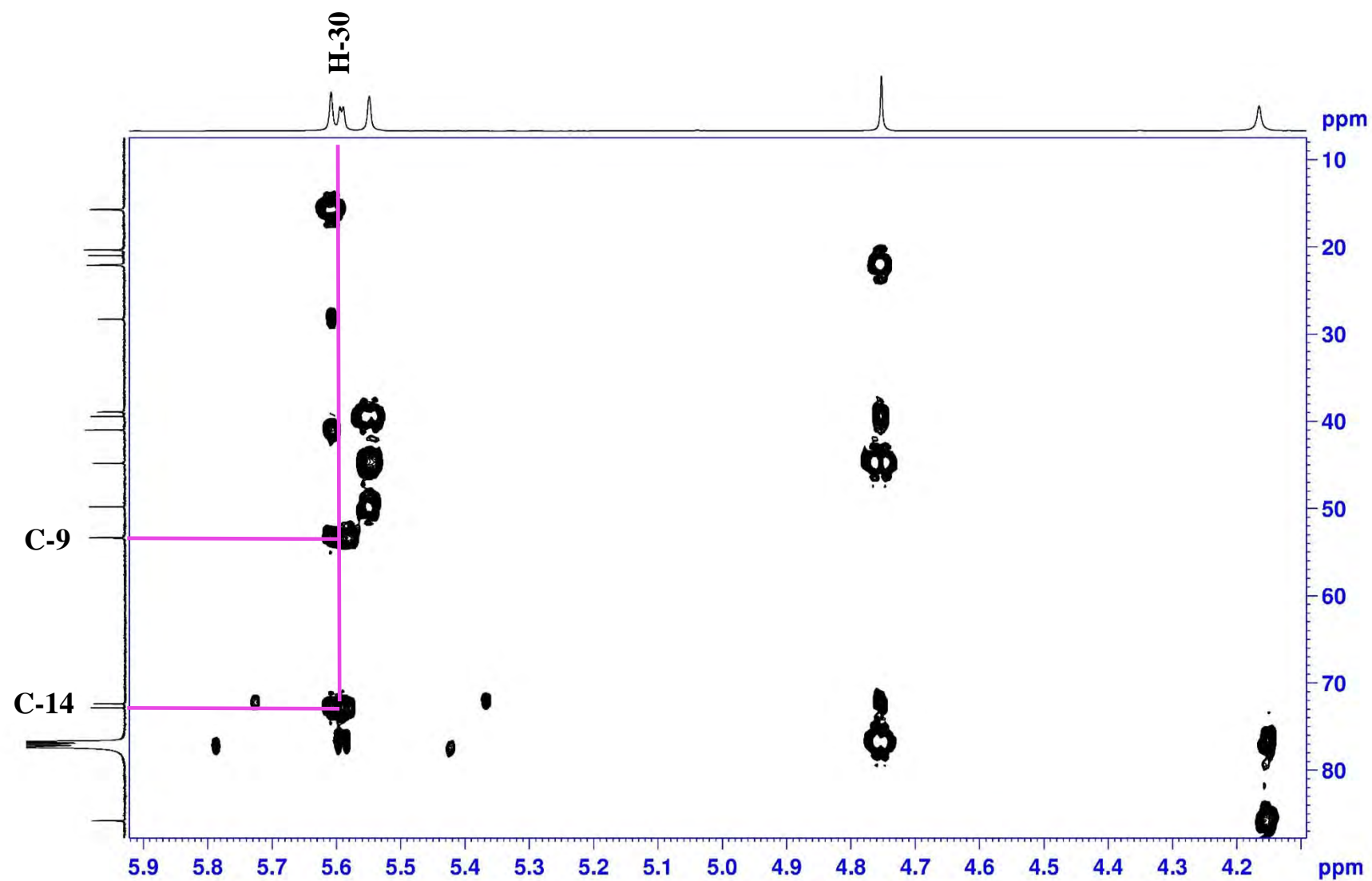
HMBC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



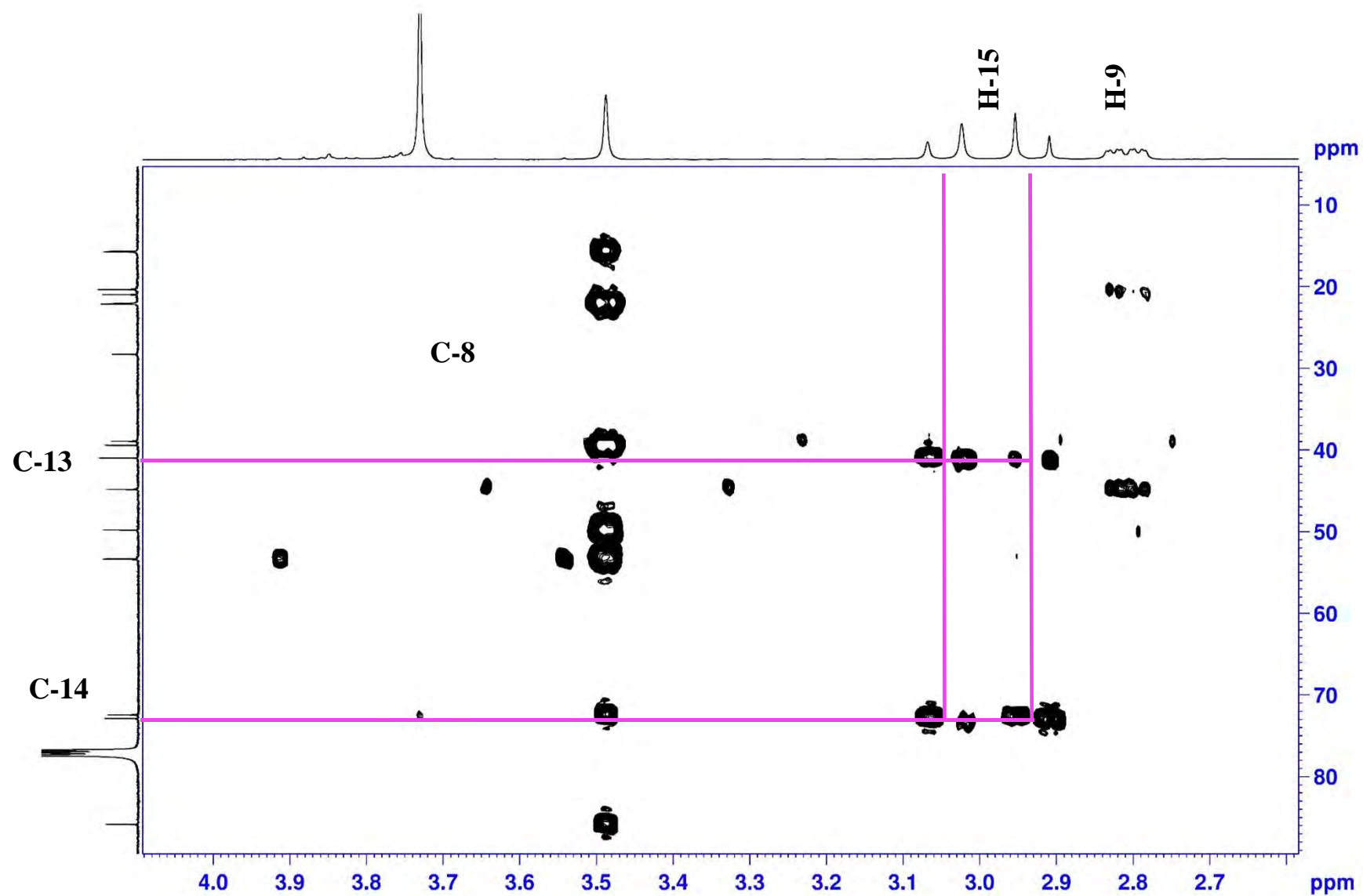
HMBC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



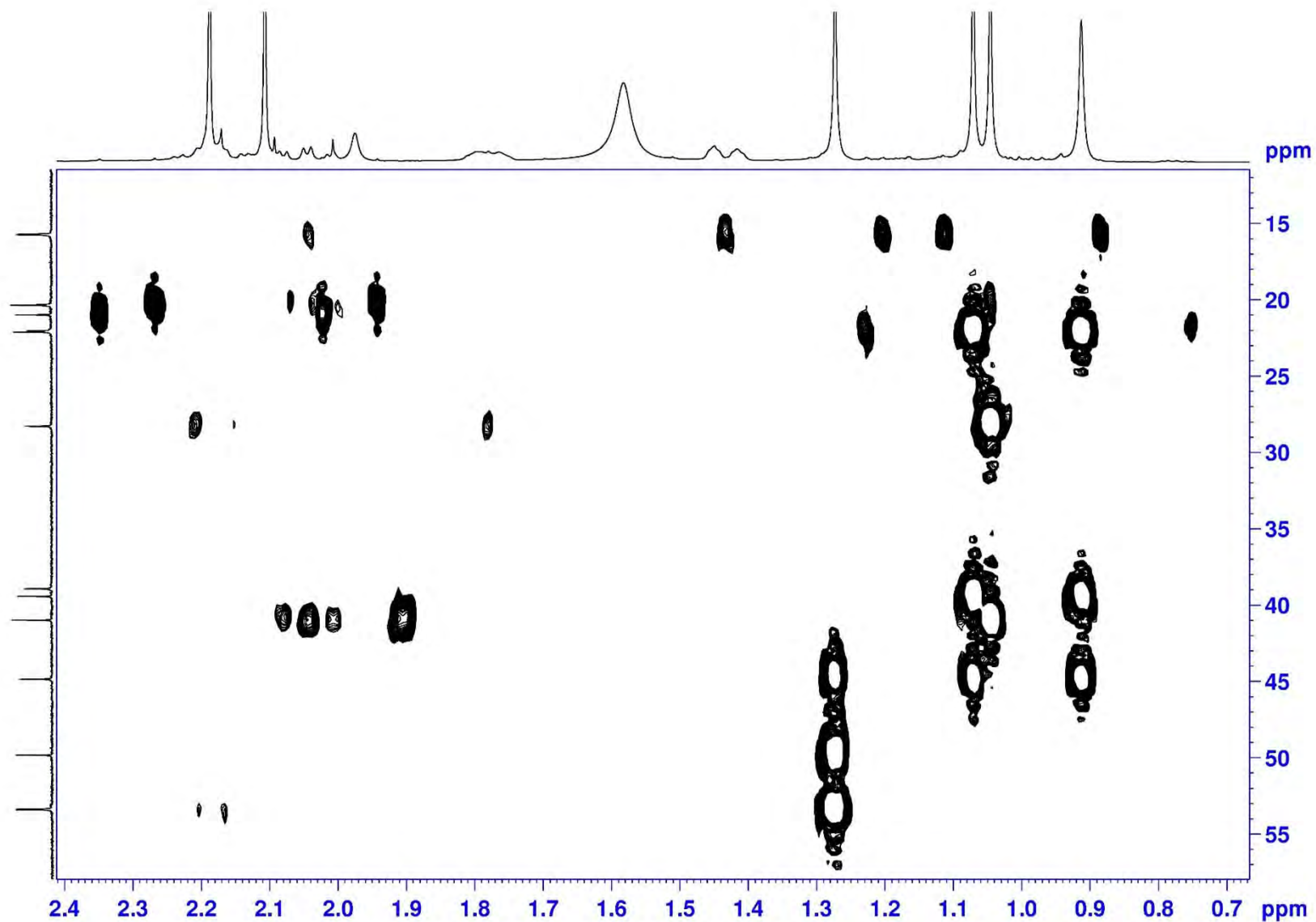
HMBC (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



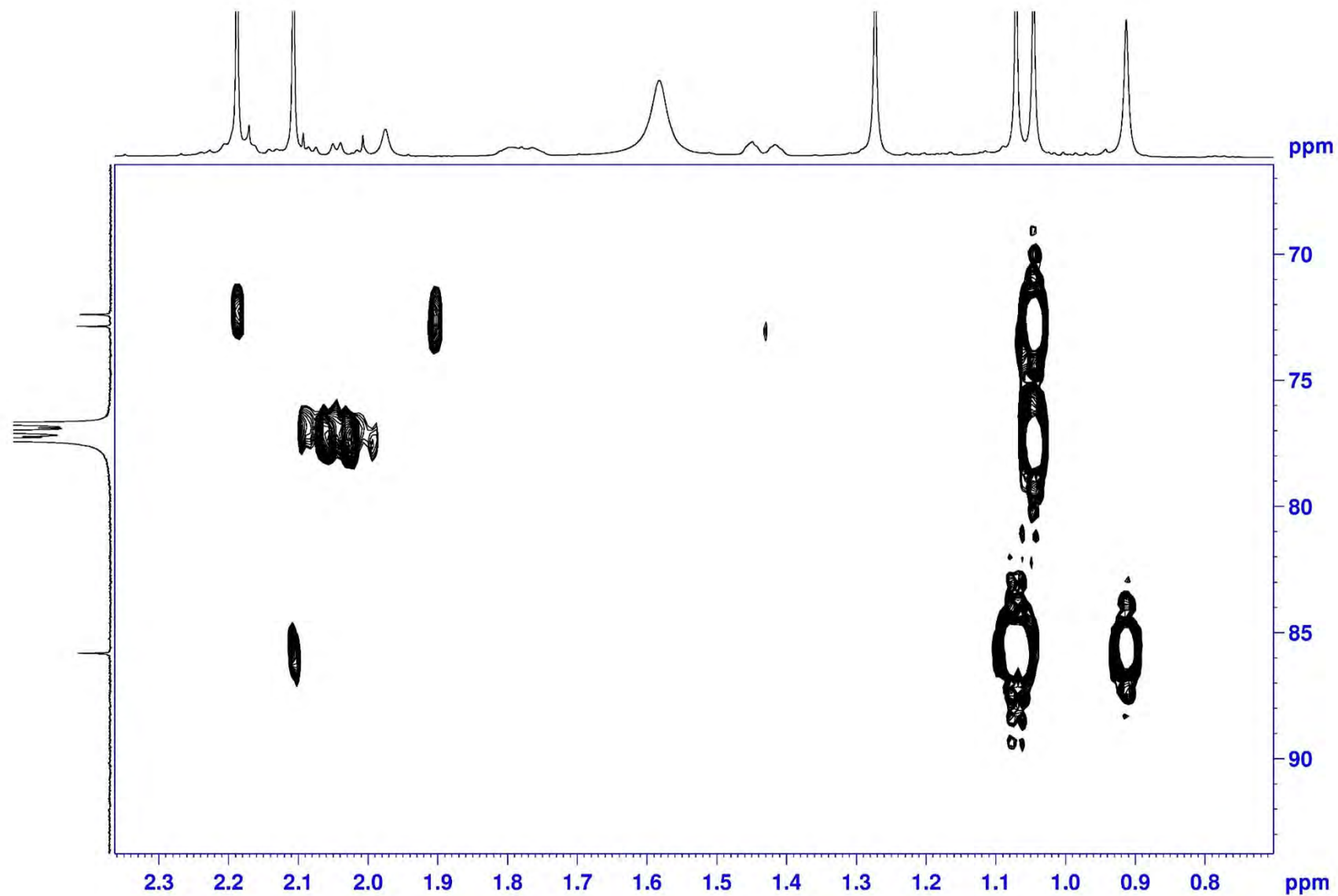
HMBC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$



HMBC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$

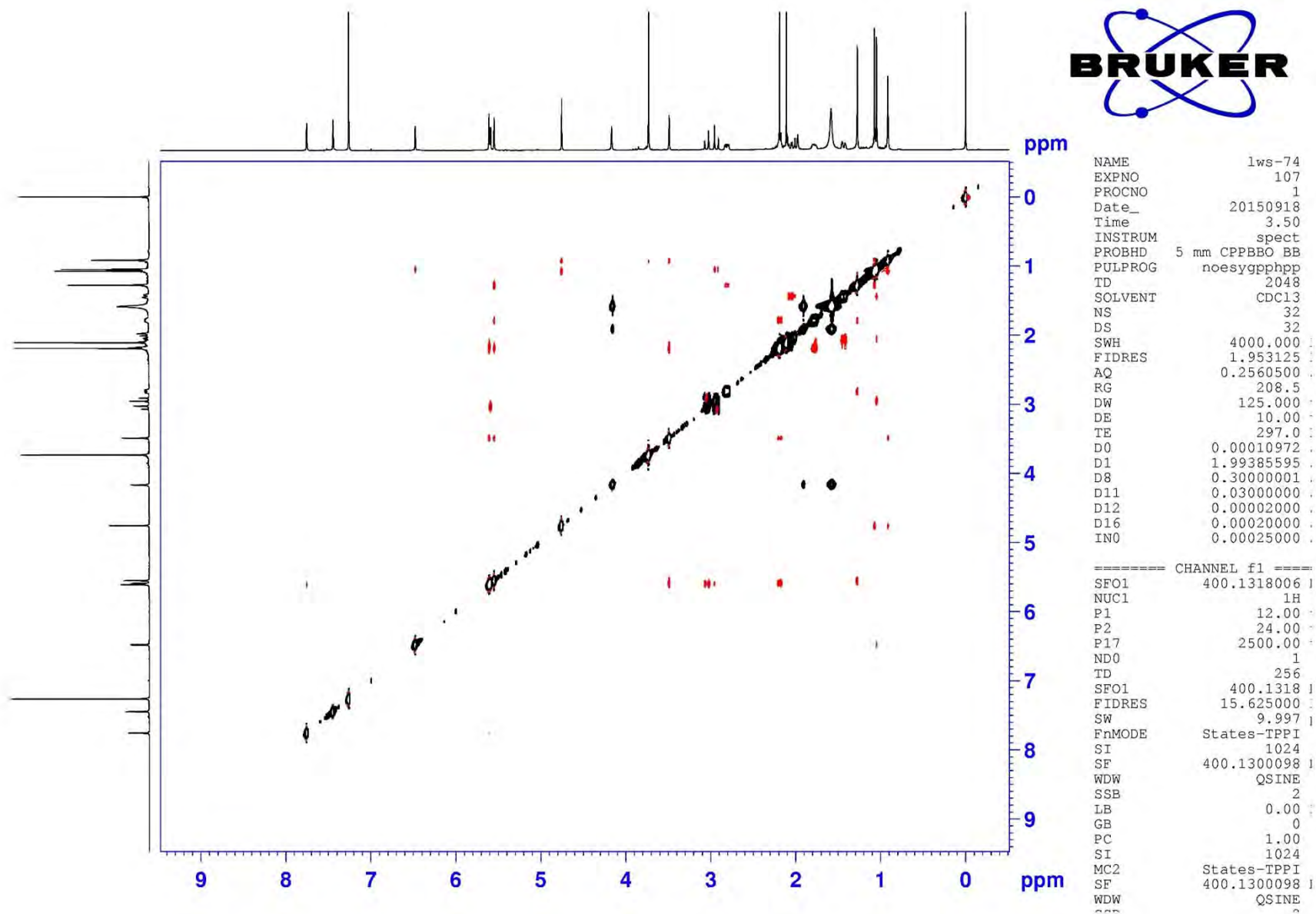


HMBC (400 MHz) spectrum of compound **13** in  $\text{CDCl}_3$

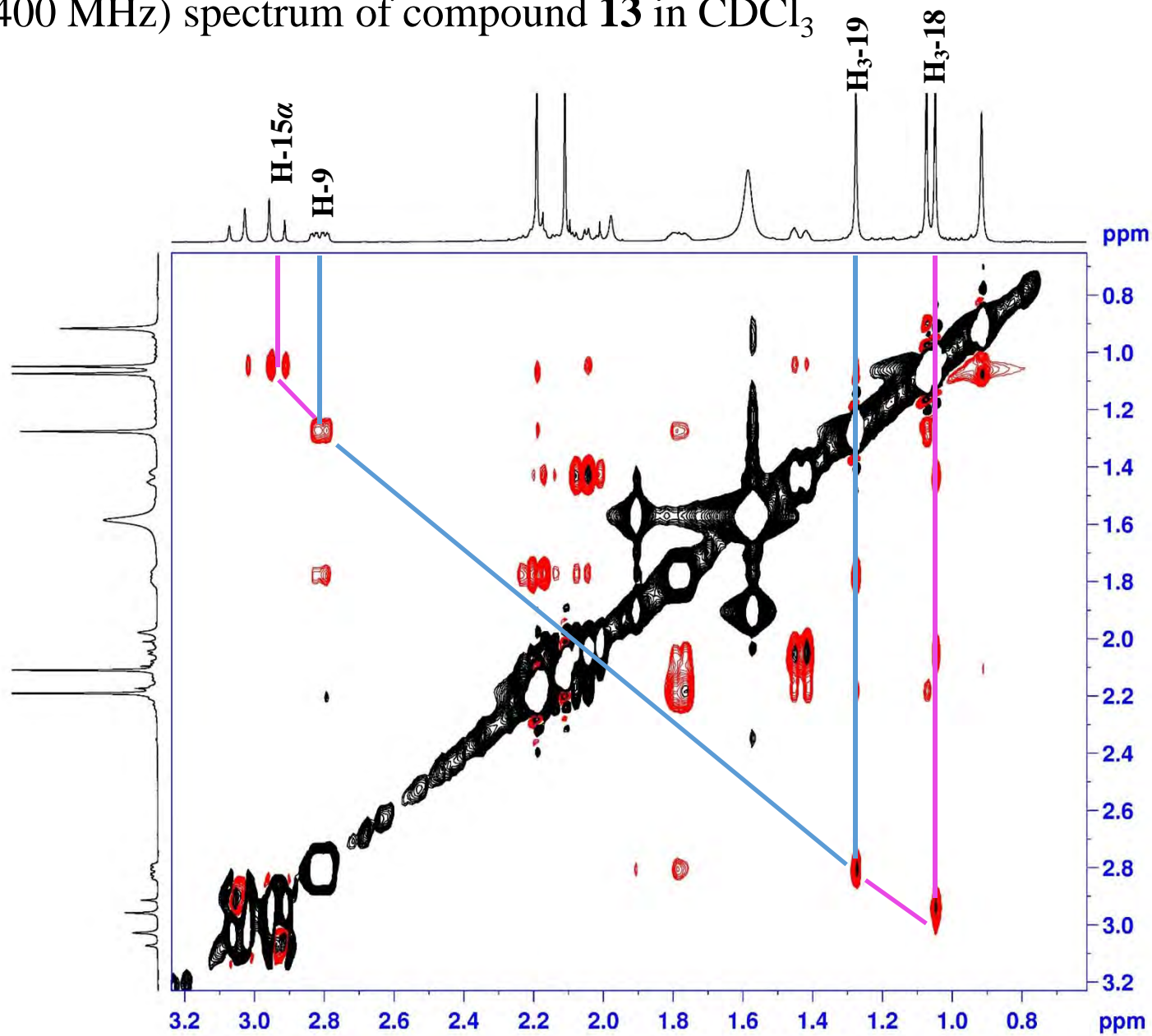




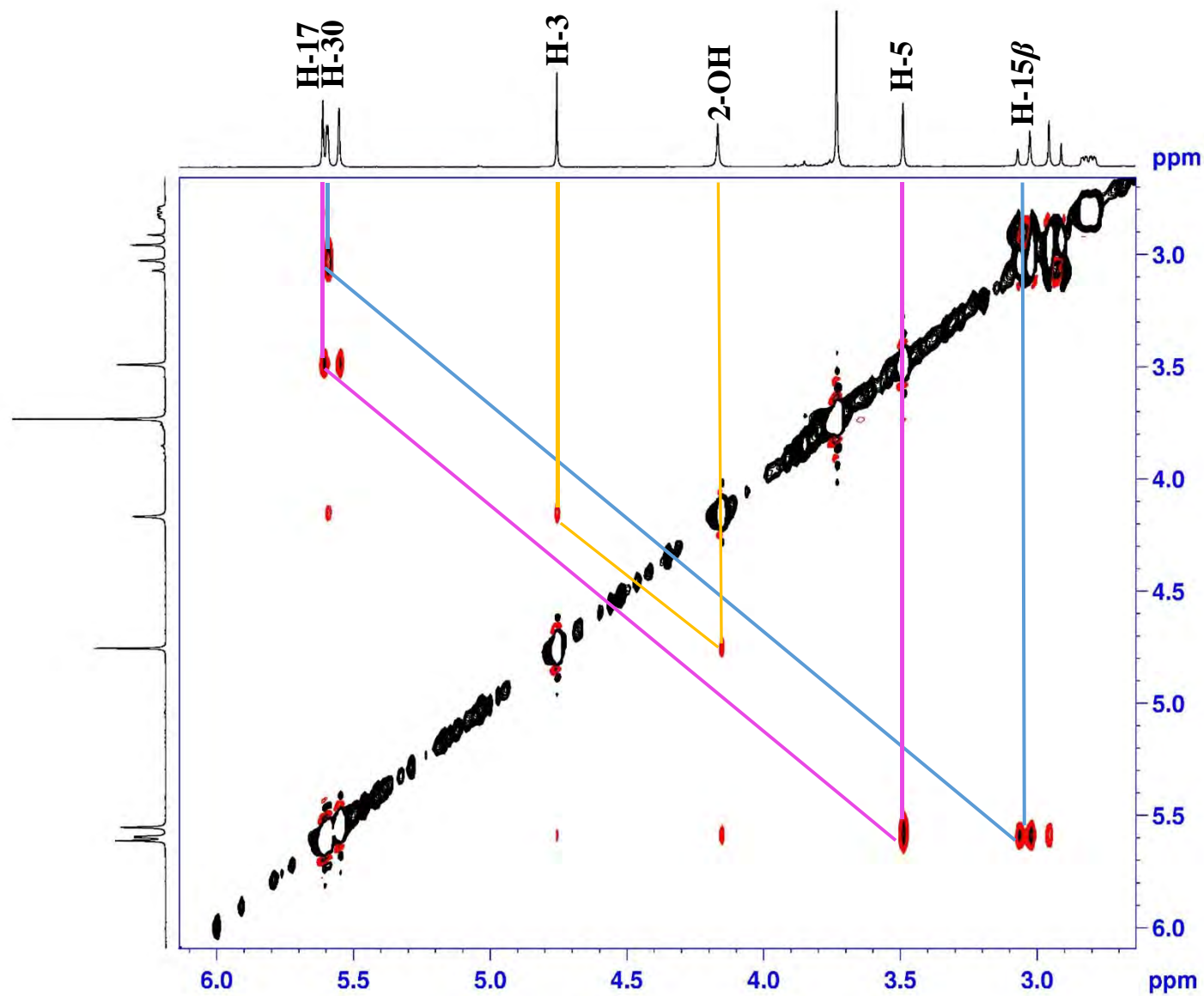
# NOESY (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



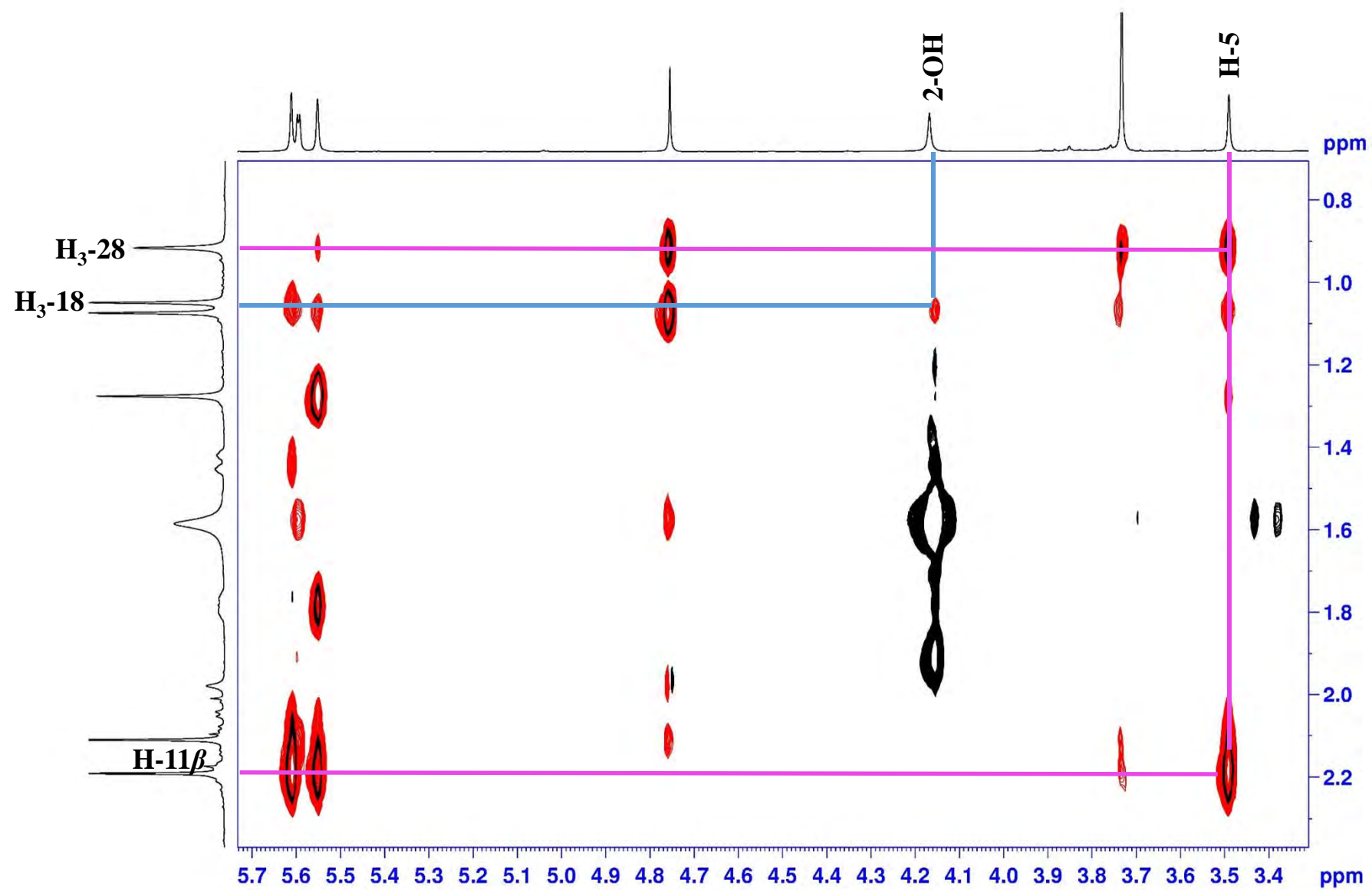
NOESY (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



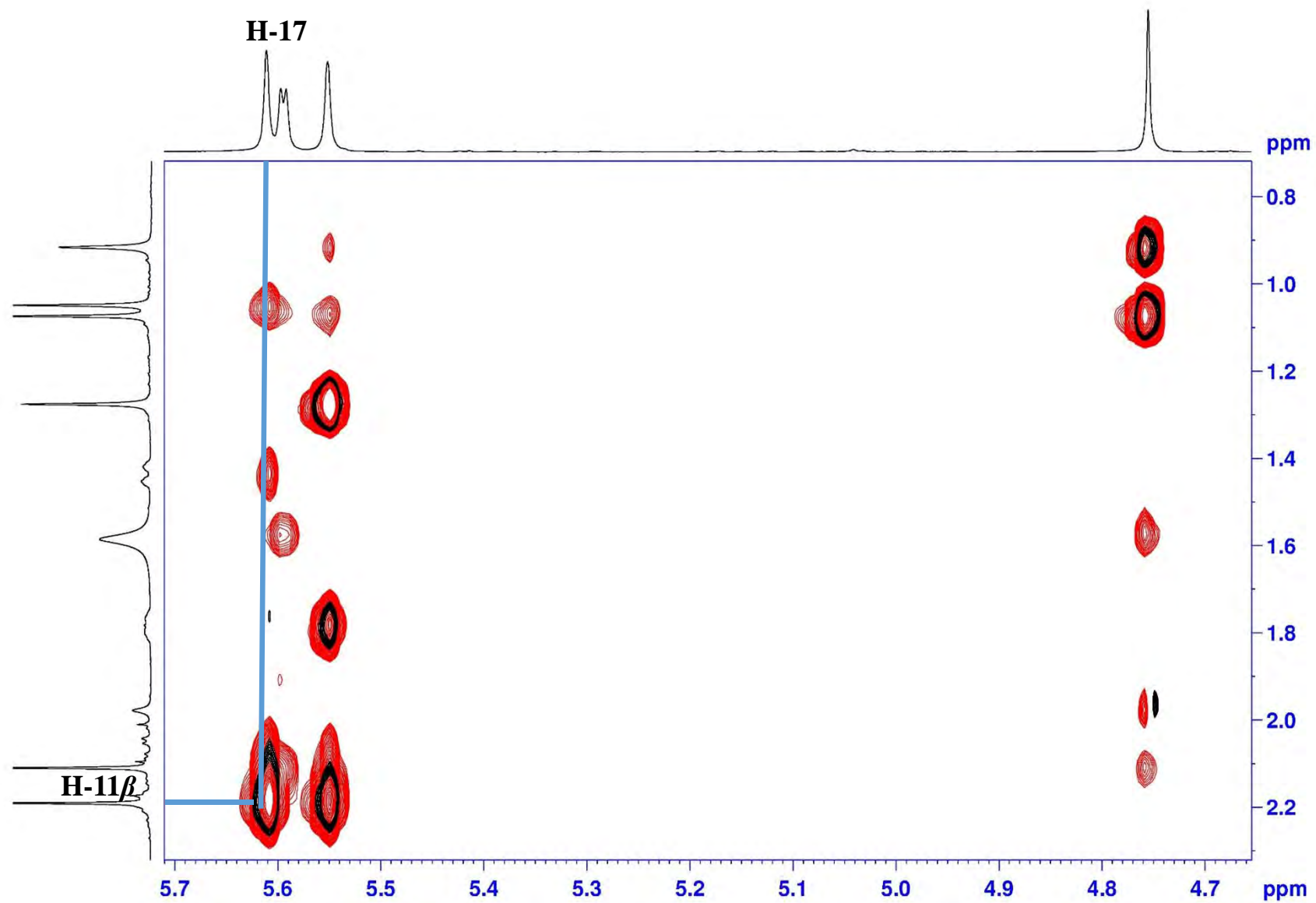
NOESY (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>



NOESY (400 MHz) spectrum of compound **13** in CDCl<sub>3</sub>





# HR-ESIMS for compound 14

## Mass Spectrum SmartFormula Report

### Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan\_dgy-6\_pos\_2\_01\_1850.d  
 Method LC\_Direct Infusion\_pos\_100-1000mz.m  
 Sample Name liwanshan\_dgy-6\_pos  
 Comment

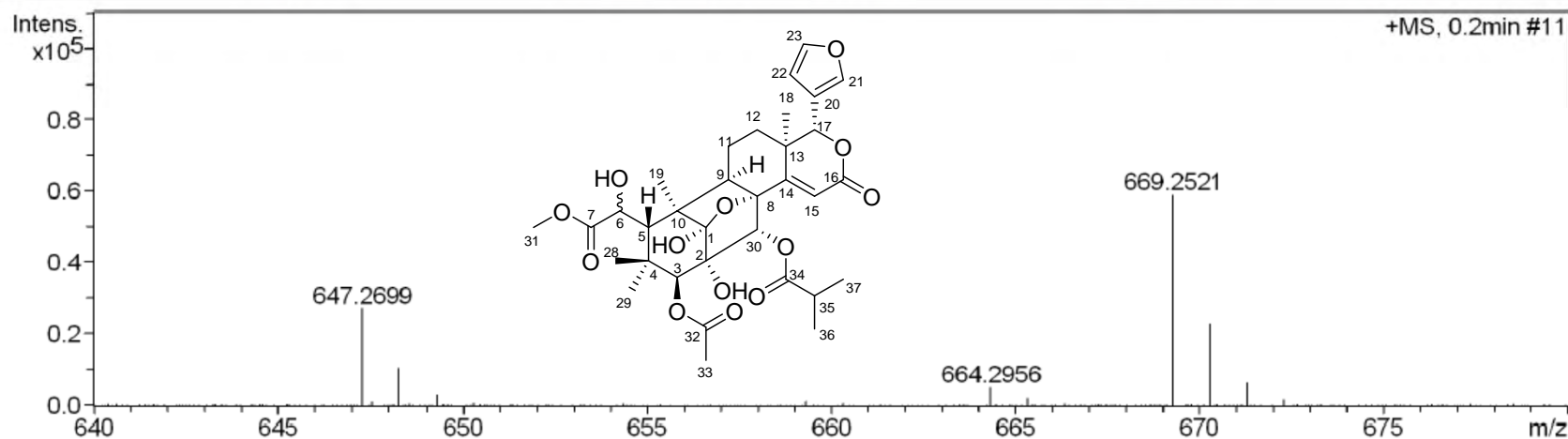
Acquisition Date 5/19/2016 8:24:06 AM

Operator SCSIO

Instrument / Ser# maXis 29

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2000 m/z	Set Collision Cell RF	800.0 Vpp	Set Divert Valve	Waste



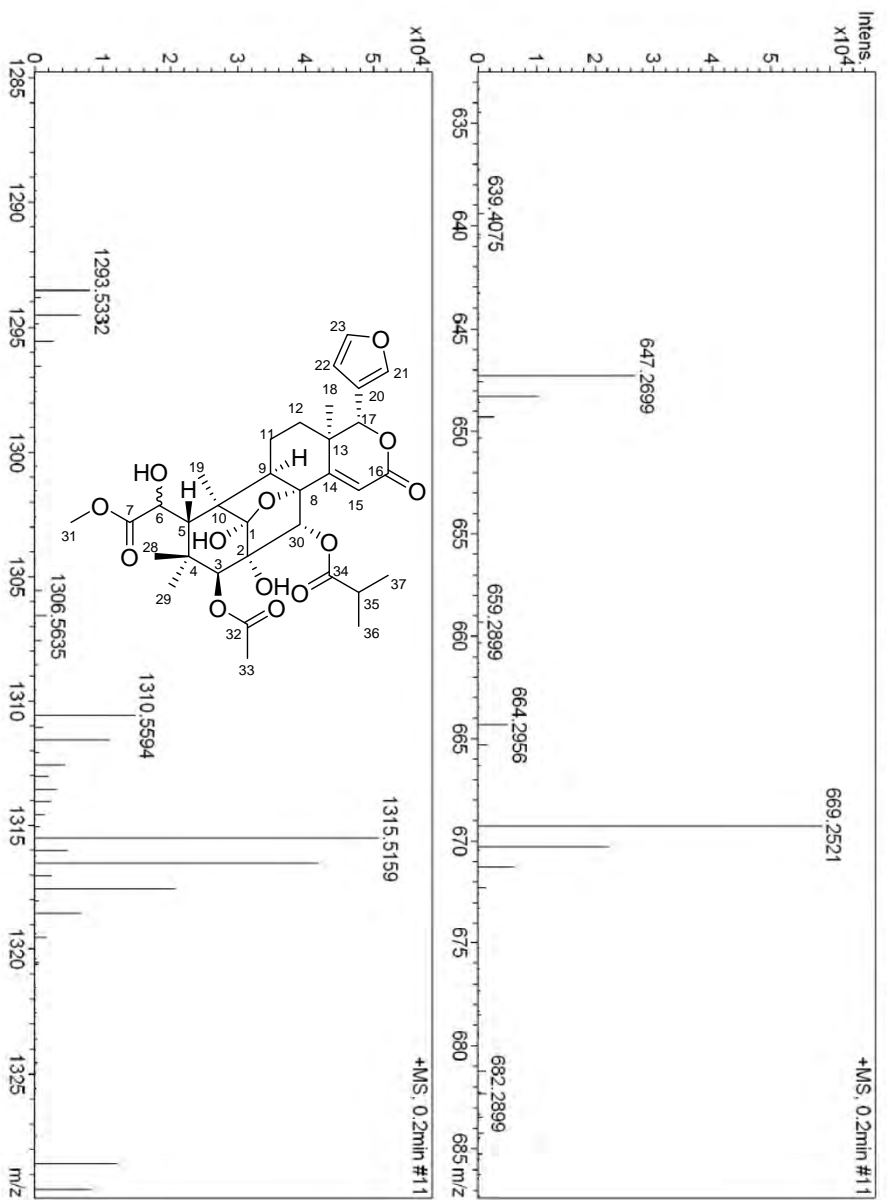
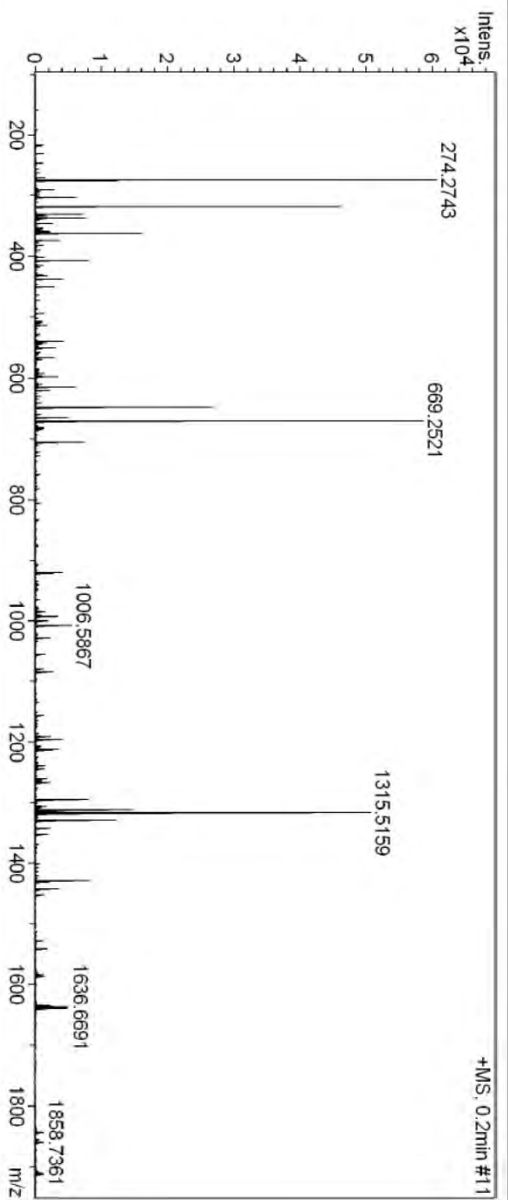
Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e <sup>-</sup> Conf	N-Rule
647.2699	1	C 33 H 43 O 13	100.00	647.2698	-0.1	-0.0	9.3	12.5	even	ok
669.2521	1	C 33 H 42 Na O 13	100.00	669.2518	-0.5	-0.3	10.7	12.5	even	ok
1315.5159	1	C 66 H 84 Na O 26	44.81	1315.5143	-1.2	-1.5	58.3	24.5	even	ok



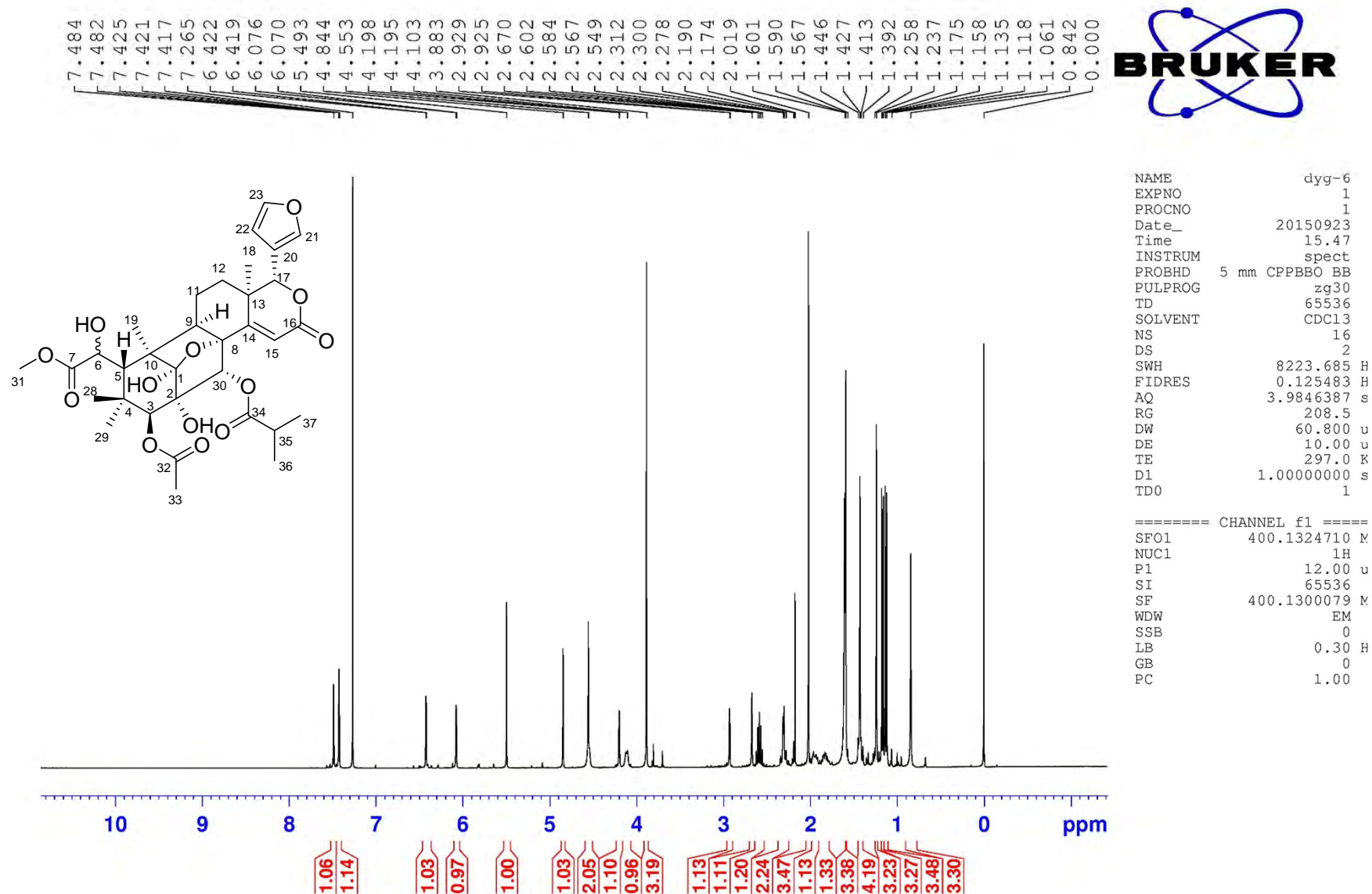
## Generic Display Report

### Analysis Info

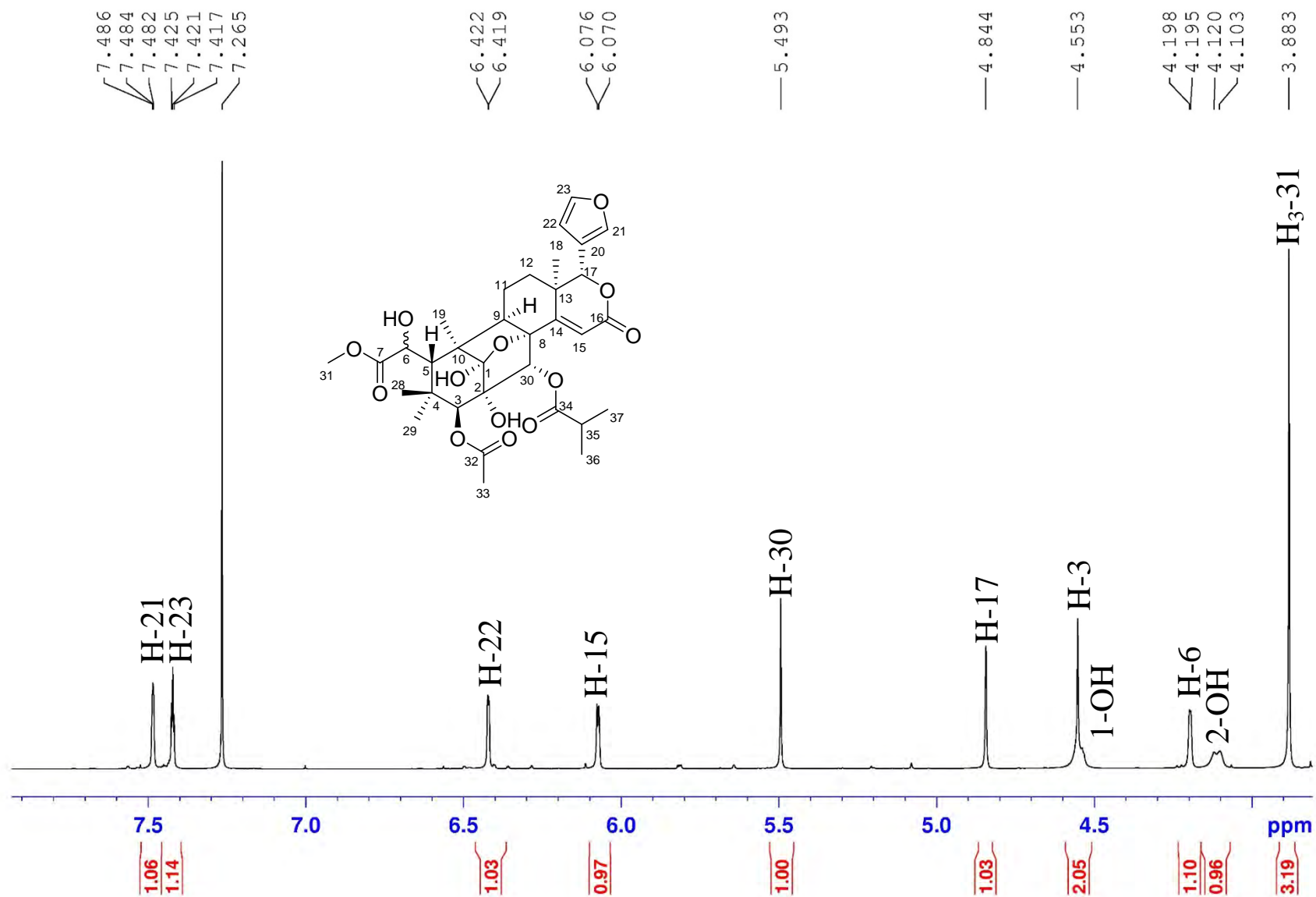
Analysis Name	D:\Data\MS\data\201605\liwanshan_dgy-6_pos_2_01_1850.d	Acquisition Date	5/19/2016 8:24:06 AM
Method	LC_Direct Infusion_pos_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_dgy-6_pos	Instrument	maxis
Comment			



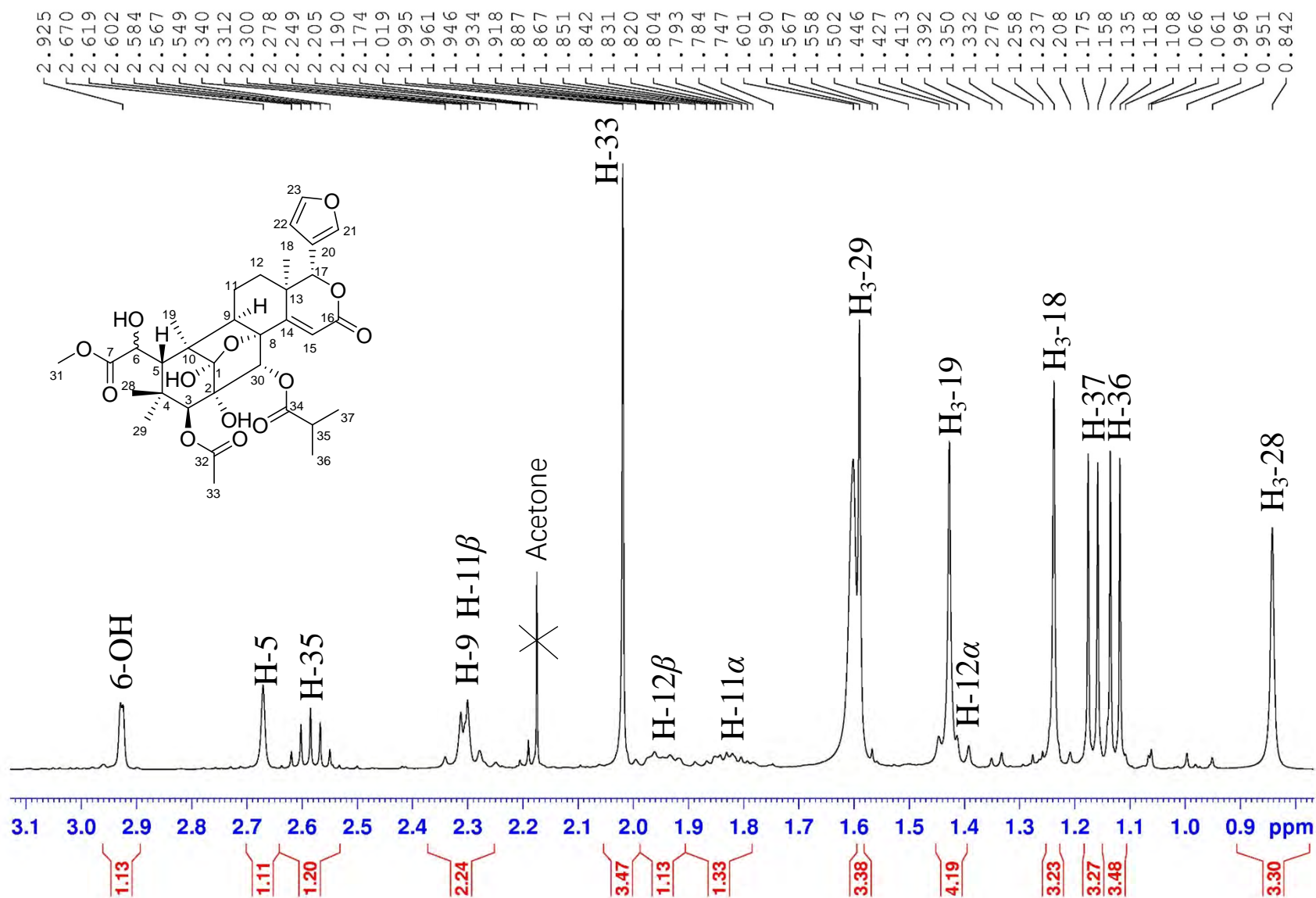
$^1\text{H}$  NMR (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



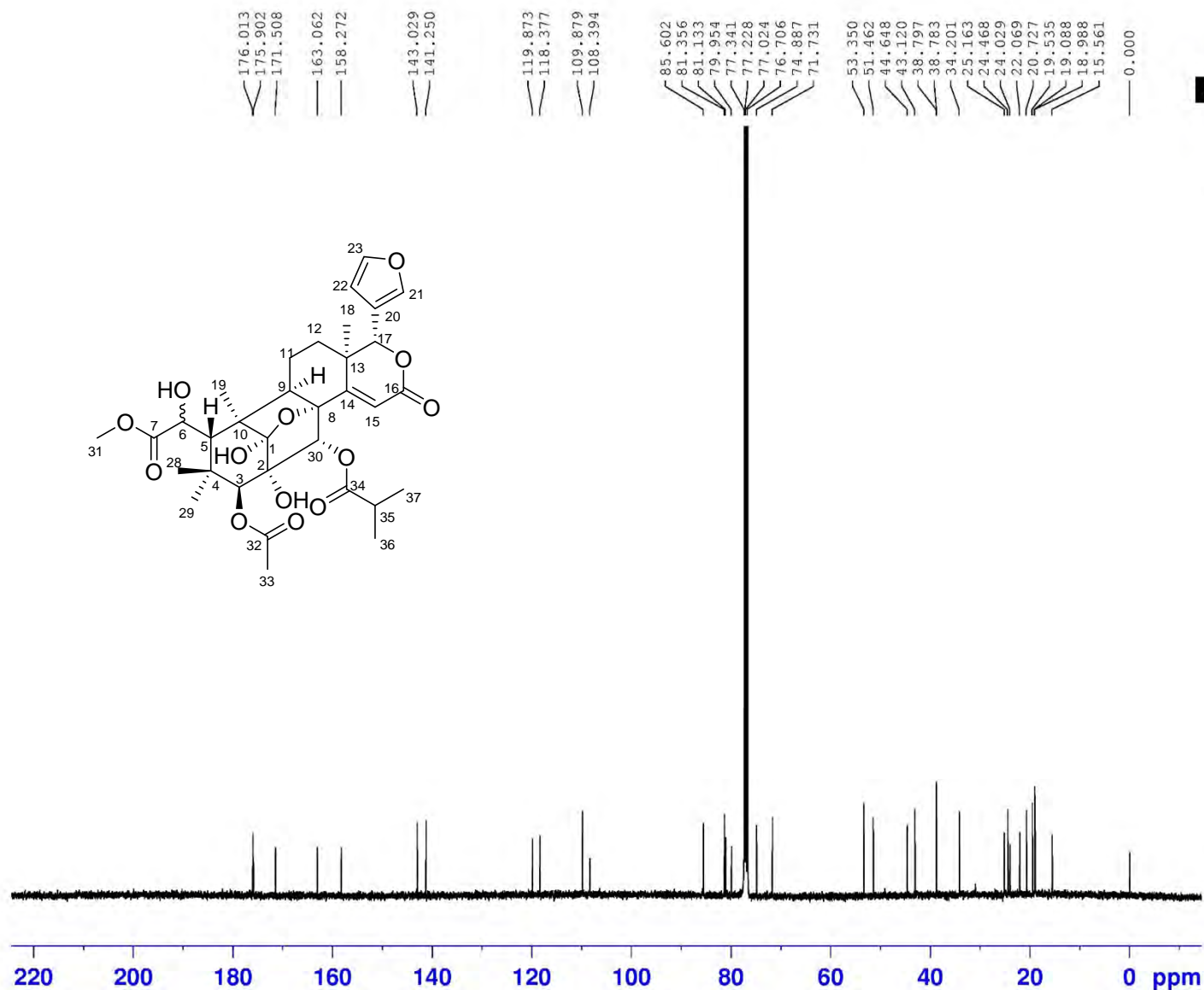
$^1\text{H}$  NMR (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



$^1\text{H}$  NMR (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



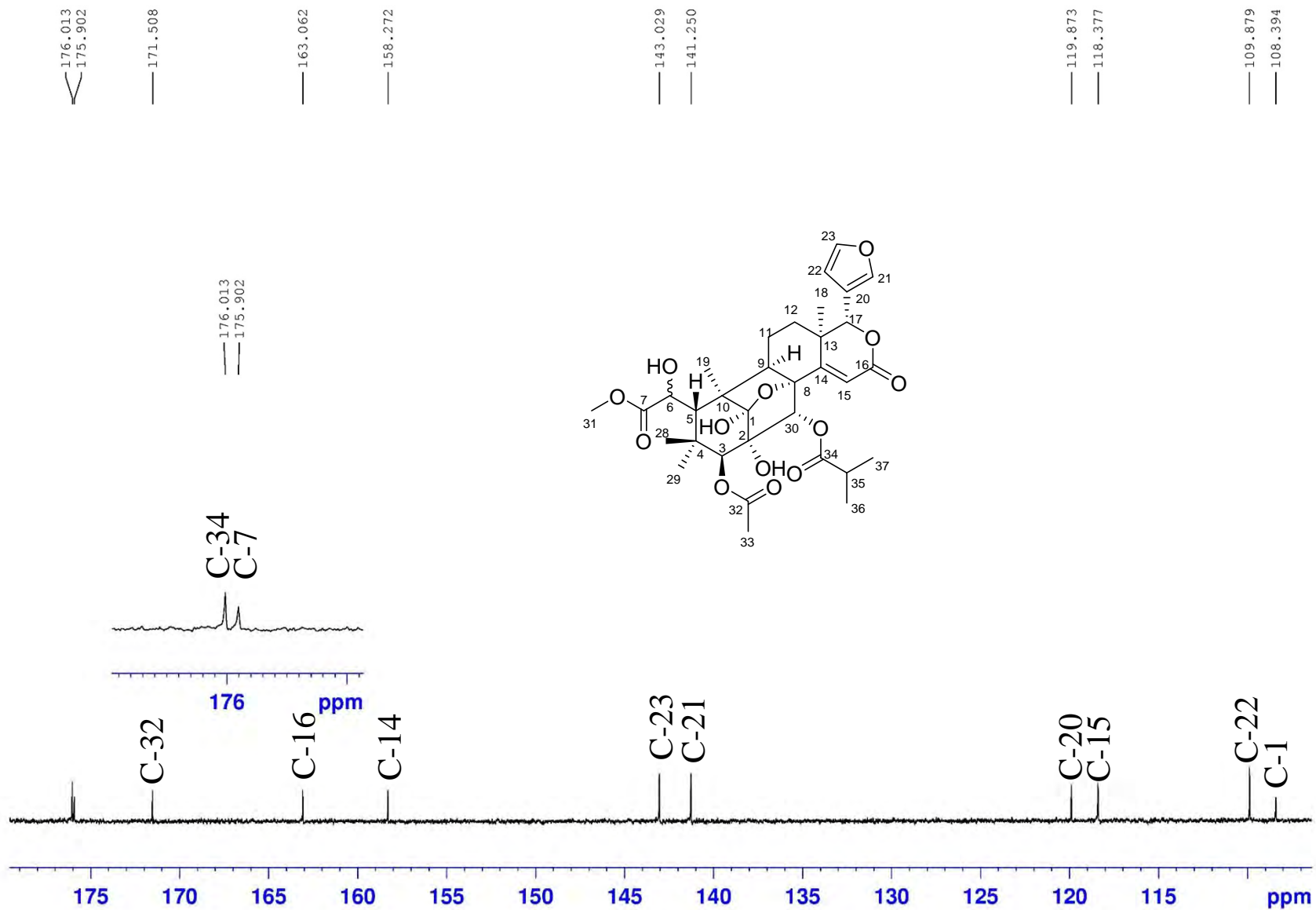
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



NAME dyg-6  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20150923  
 Time 16.47  
 INSTRUM spect  
 PROBHD 5 mm CPPBBO BB  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT  $\text{CDCl}_3$   
 NS 1024  
 DS 4  
 SWH 24038.461 F  
 FIDRES 0.366798 F  
 AQ 1.3631988 s  
 RG 102.3  
 DW 20.800  $\mu$   
 DE 18.00  $\mu$   
 TE 297.0 K  
 D1 2.00000000 s  
 D11 0.03000000 s  
 TD0 1

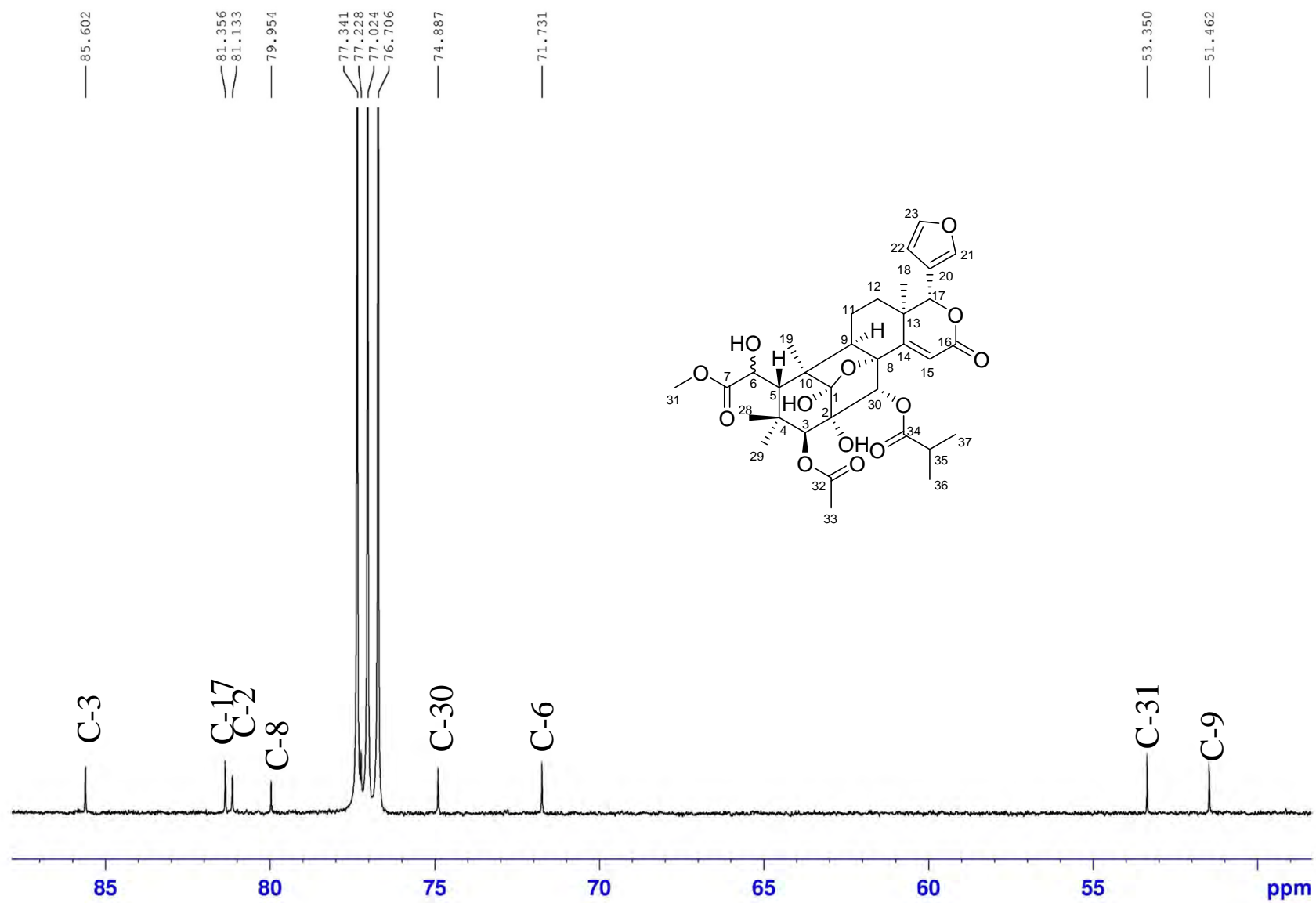
===== CHANNEL f1 =====  
 SFO1 100.6233324 M  
 NUC1  $^{13}\text{C}$   
 P1 10.00  $\mu$   
 SI 32768  
 SF 100.6127690 M  
 WDW EM  
 SSB 0  
 LB 1.00 F  
 GB 0  
 PC 1.40

$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **14** in  $\text{CDCl}_3$

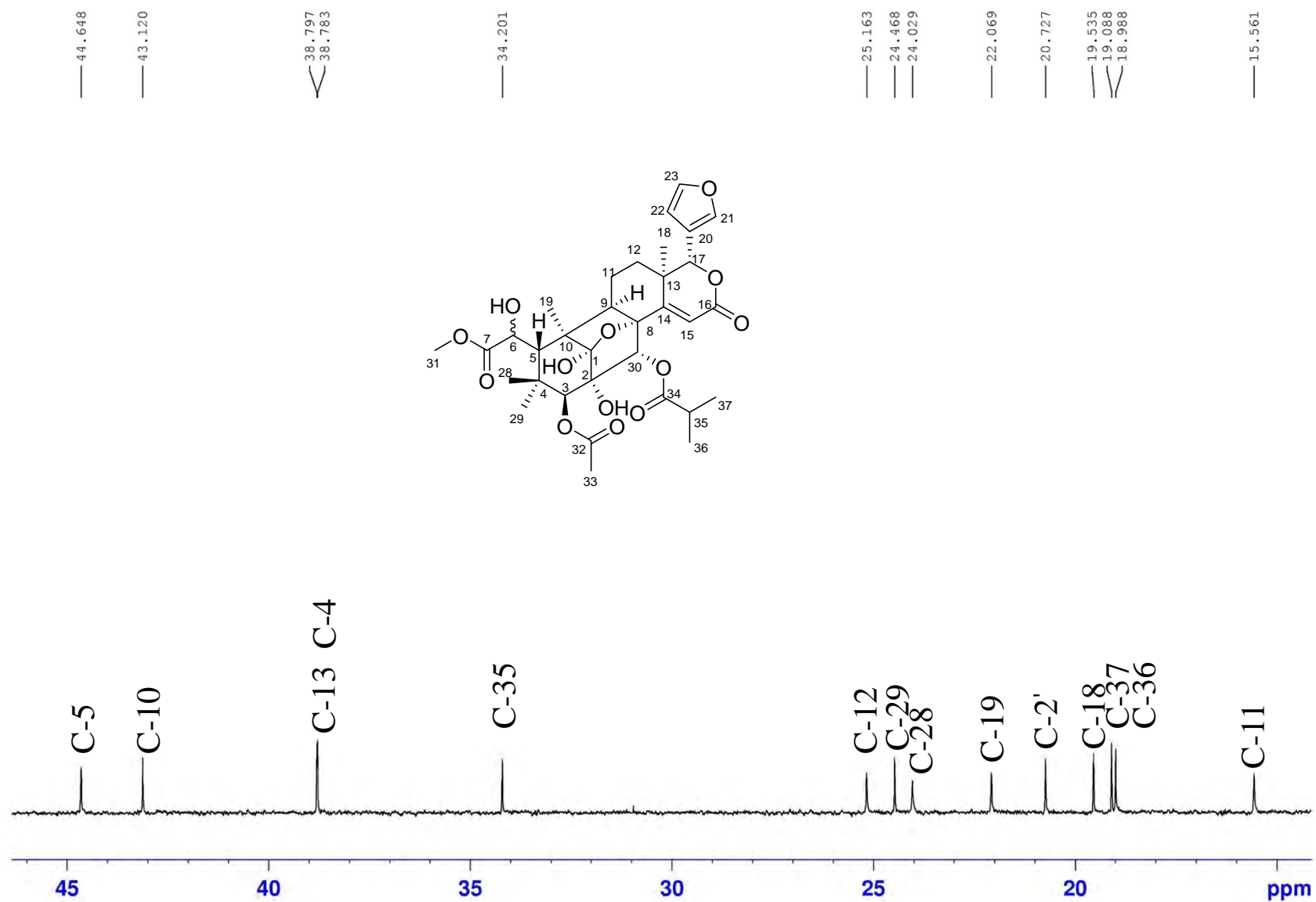




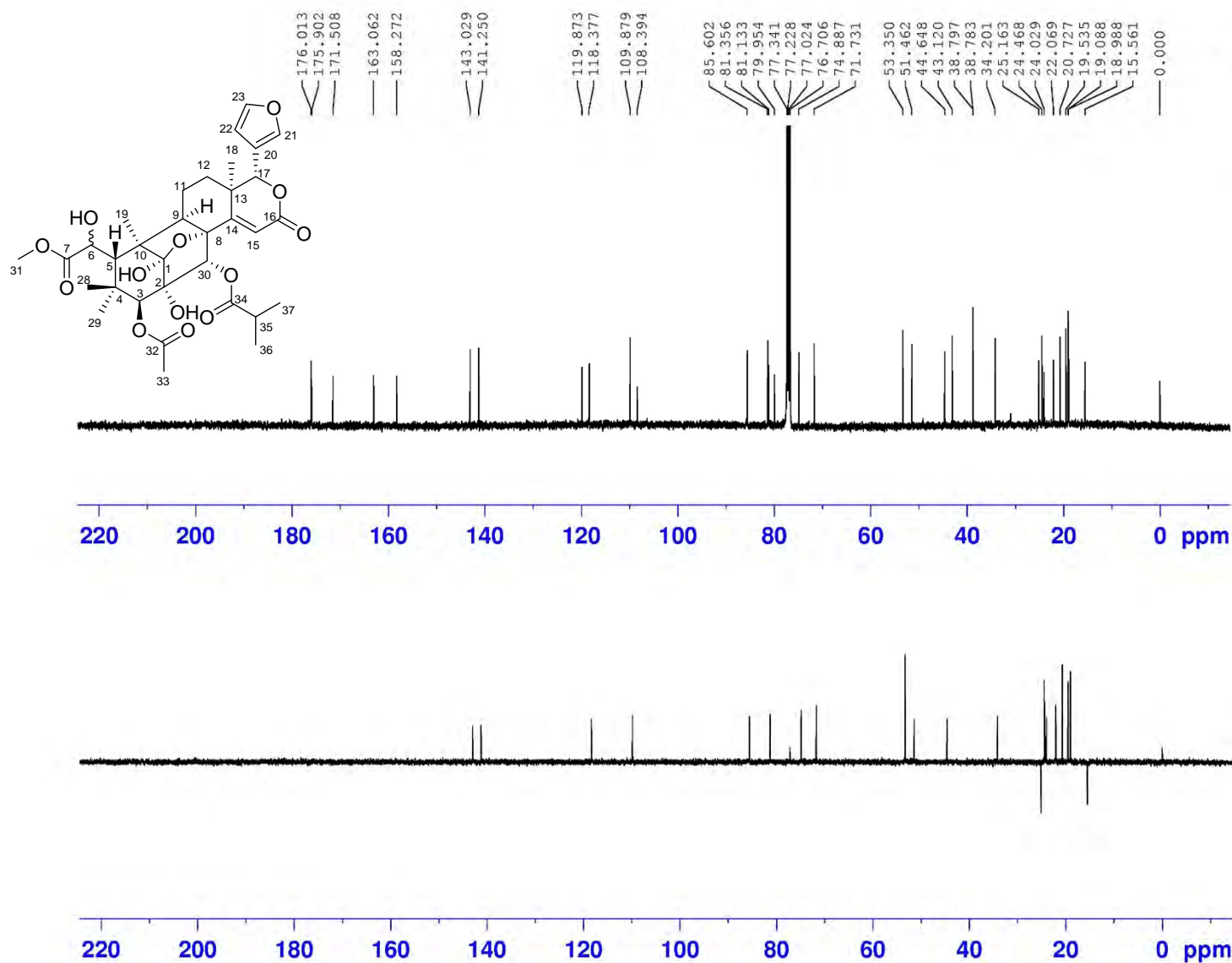
$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



$^{13}\text{C}$  NMR (100 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



# DEPT135 (100 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



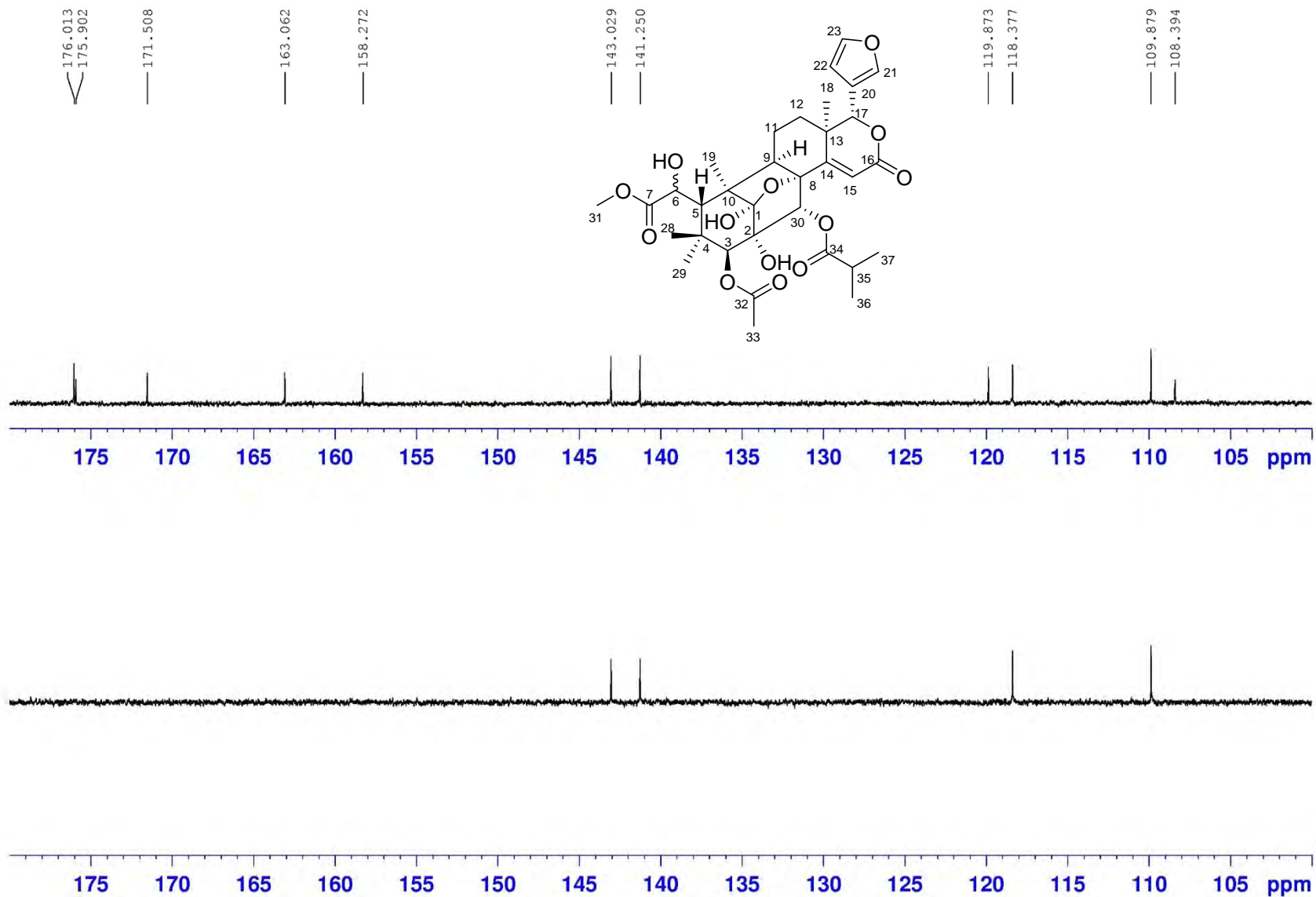
```

NAME          dyg-6
EXPNO         103
PROCNO        1
Date_         20151027
Time          10.25
INSTRUM       spect
PROBHD        5 mm CPMBO BB
PULPROG       deptspl35
TD            65536
SOLVENT       CDCl3
NS            300
DS            4
SWH           24038.461 F
FIDRES        0.366798 F
AQ            1.3631988 s
RG            130.26
DW            20.800 s
DE            18.00 s
TE            297.0 F
CNST2         145.0000000
D1            2.0000000 s
D2            0.00344828 s
D12           0.00002000 s
TD0           1
    
```

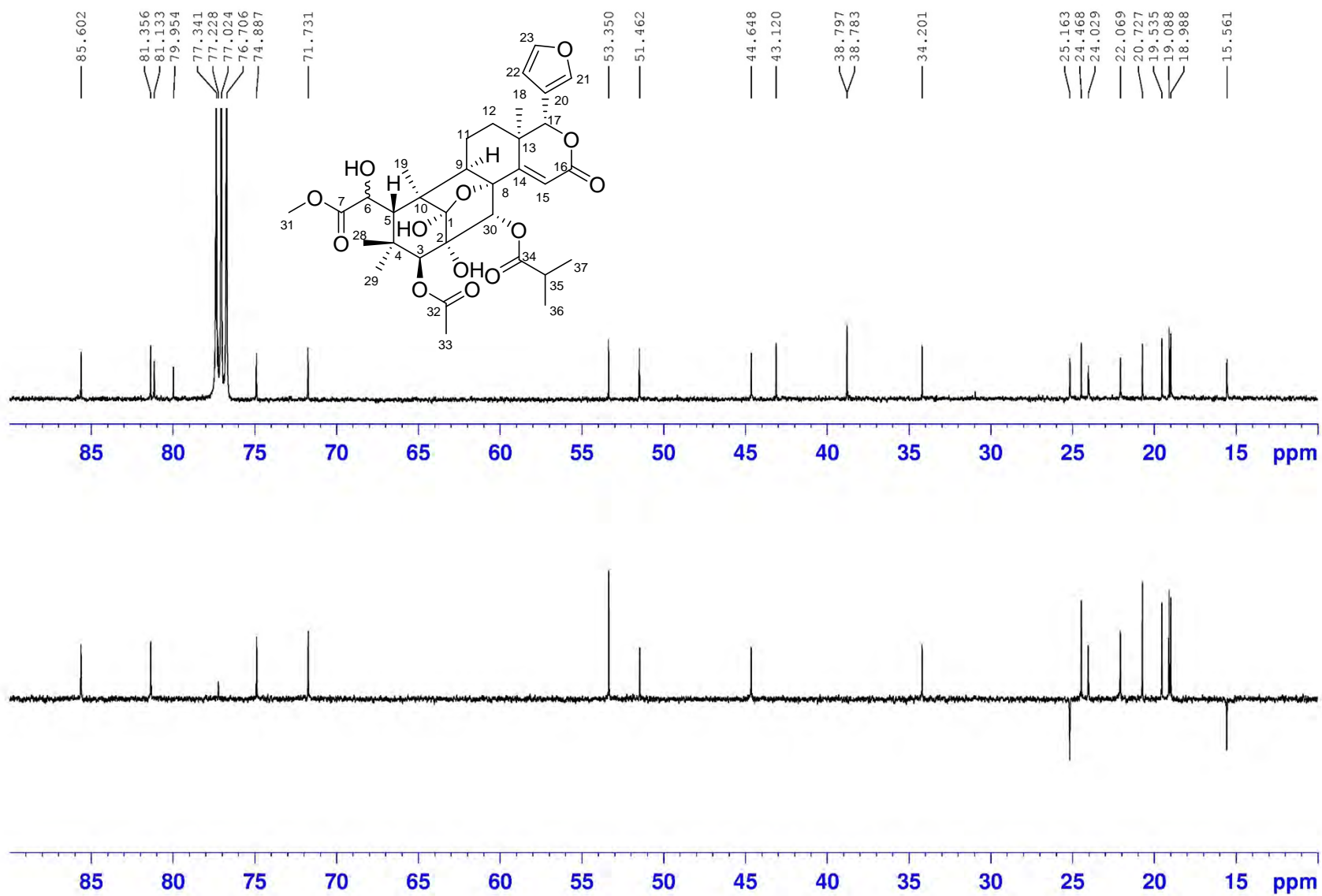
```

===== CHANNEL f1 =====
SFO1         100.6233324 M
NUC1          13C
P1            10.00 s
P13           2000.00 s
SI            32768
SF            100.6127689 M
WDW           EM
SSB           0
LB            1.00 F
GB            0
PC            1.40
    
```

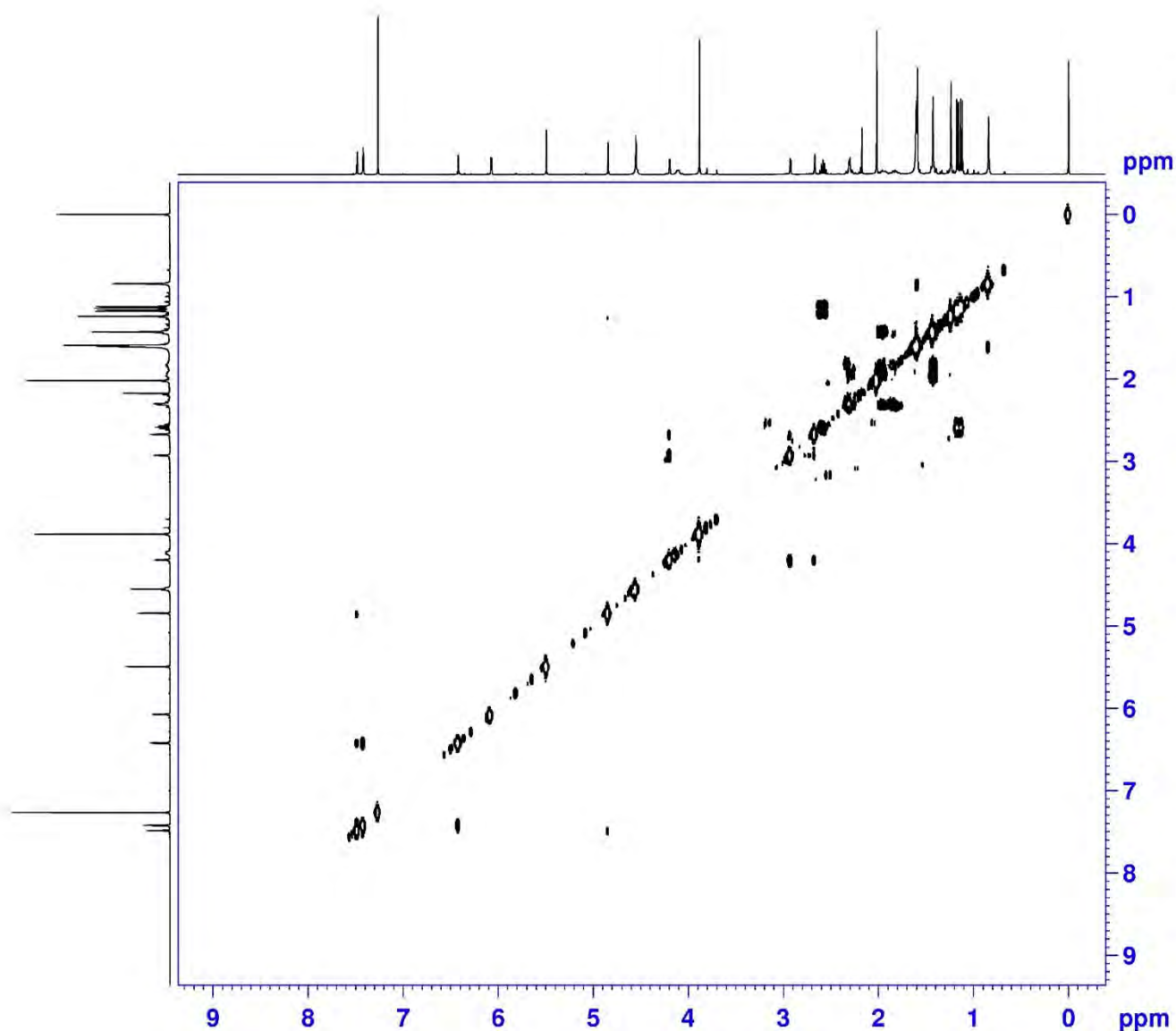
# DEPT135 (100 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



DEPT135 (100 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$

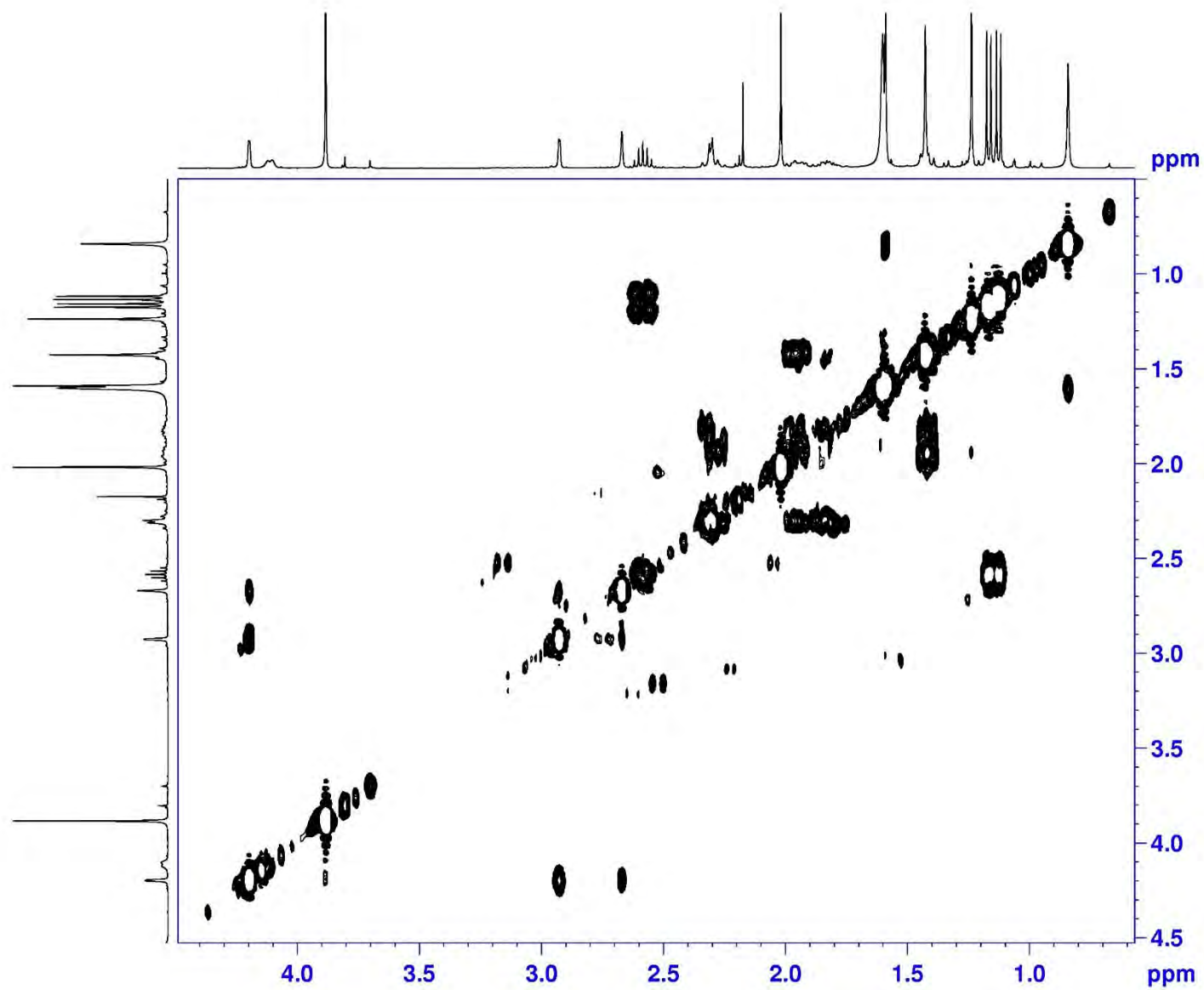


```
NAME          dyg-6
EXPNO          104
PROCNO         1
Date_          20151027
Time           10.27
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        cosygpppgf
TD             2048
SOLVENT        CDCl3
NS              8
DS              8
SWH            3906.250 H:
FIDRES         1.907349 H:
AQ             0.2621940 s:
RG             208.5
DW             128.000 u:
DE             10.00 u:
TE             297.0 K
D0             0.00000300 s:
D1             1.89678097 s:
D11            0.03000000 s:
D12            0.00002000 s:
D13            0.00000400 s:
D16            0.00020000 s:
IN0            0.00025600 s:
```

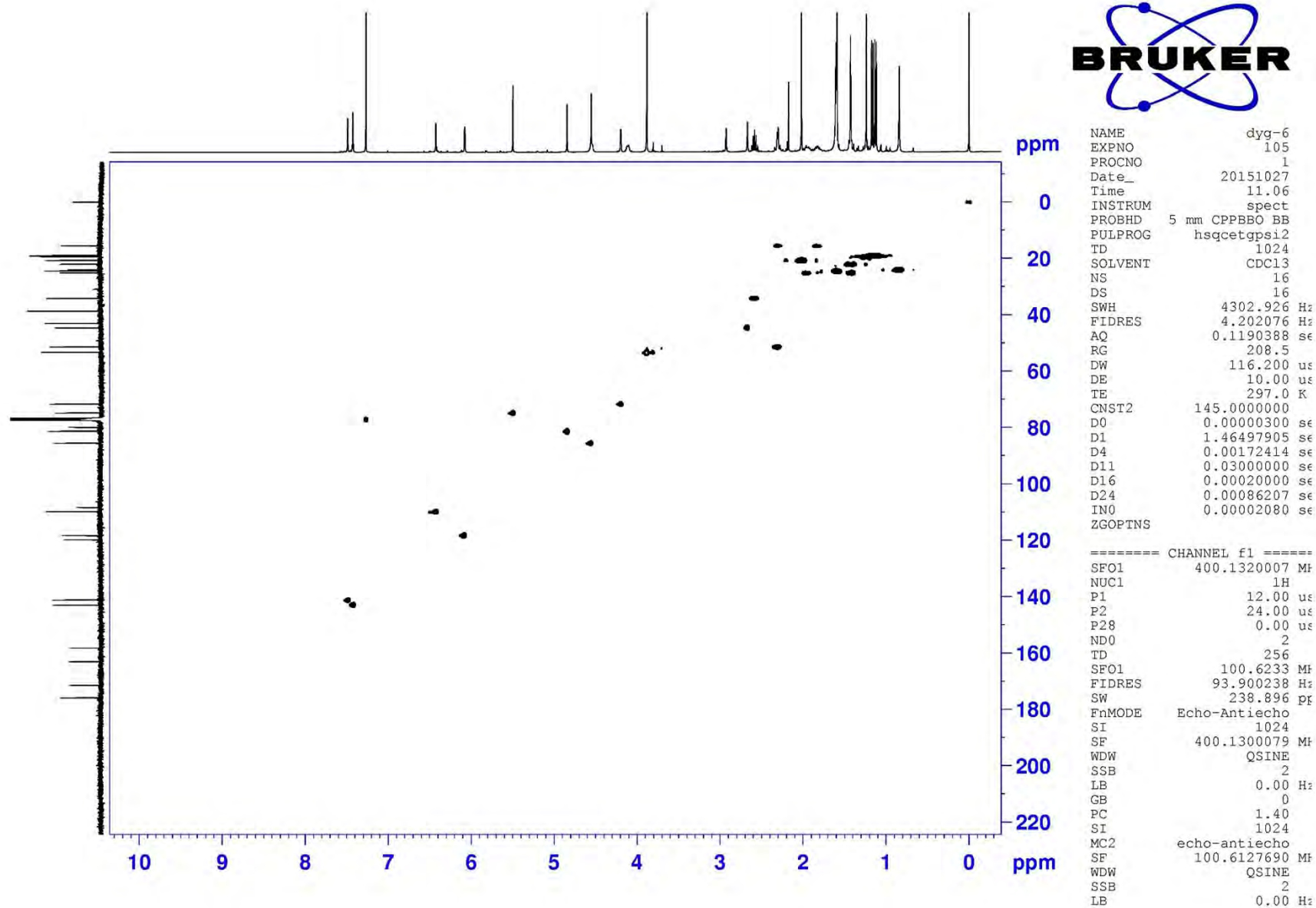
```
===== CHANNEL f1 =====
SFO1          400.1318006 MI
NUC1           1H
P0             12.00 u:
P1             12.00 u:
P17            2500.00 u:
ND0            1
TD             128
SFO1          400.1318 MI
FIDRES         30.517578 H:
SW             9.762 p:
FnMODE         QF
SI             1024
SF            400.1300065 MI
WDW            QSINE
SSB            0
LB             0.00 H:
GB             0
PC             1.40
SI             1024
MC2            QF
SF            400.1300065 MI
WDW            QSINE
SSB            0
```



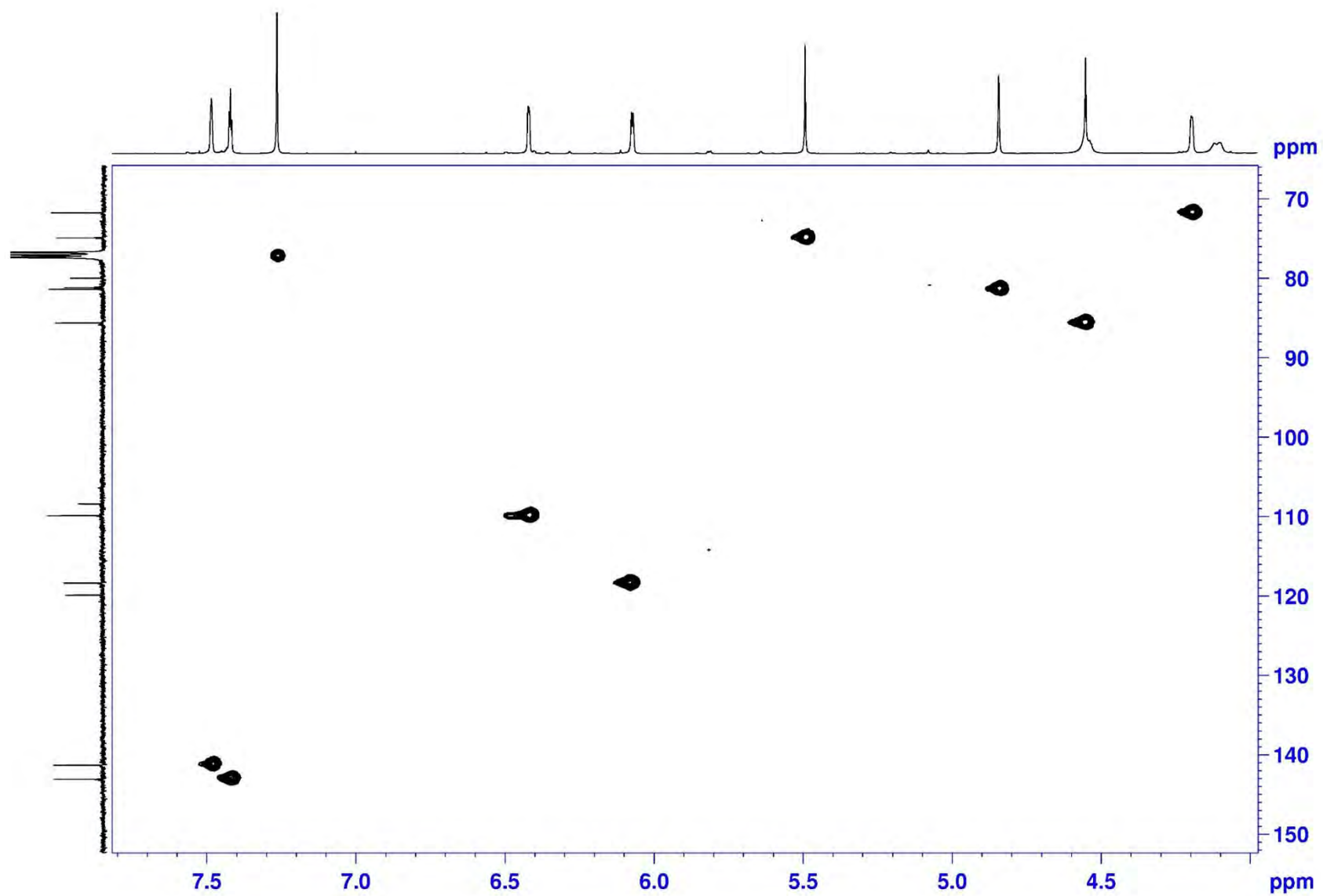
$^1\text{H}$ - $^1\text{H}$  COSY (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



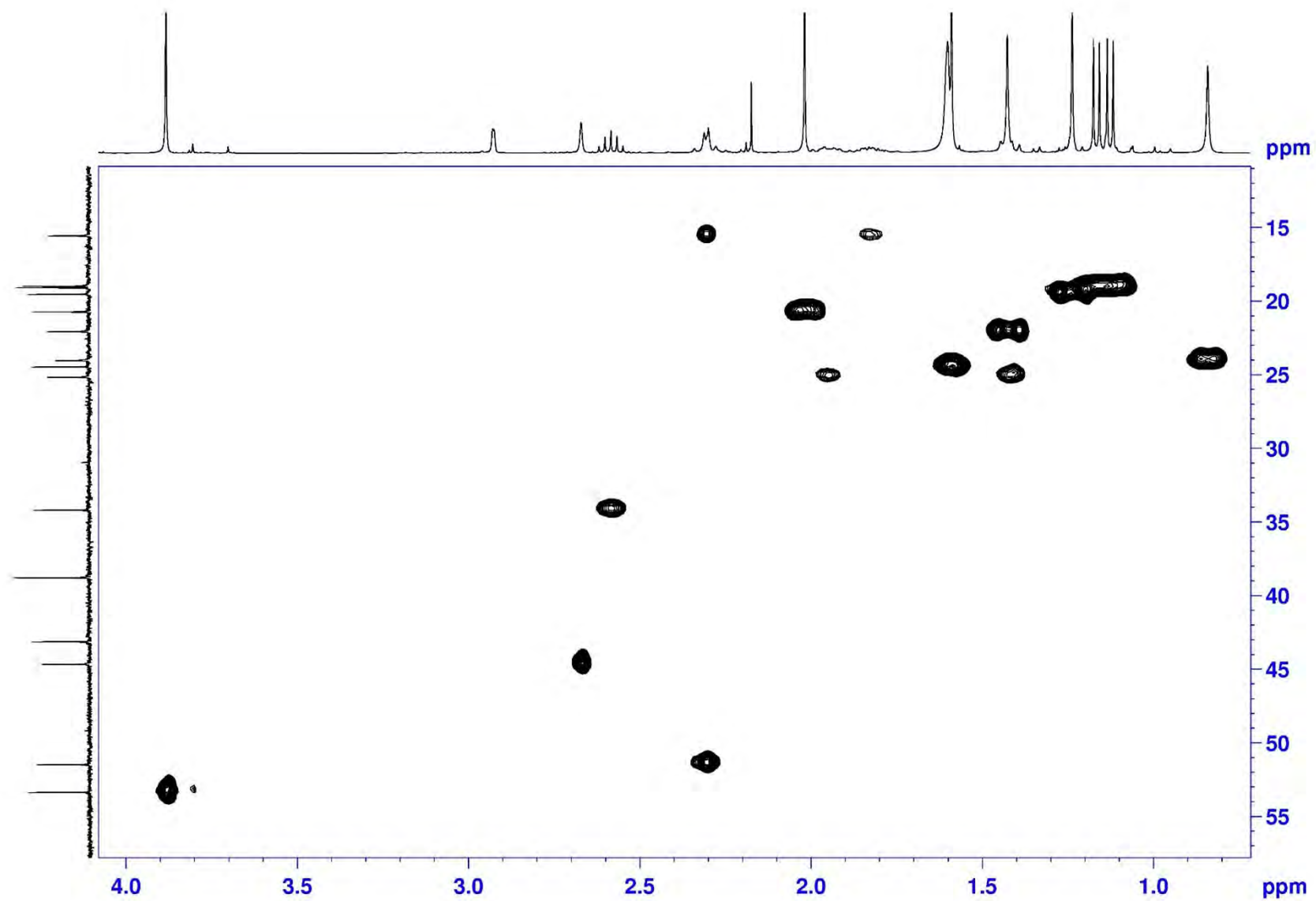
# HSQC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



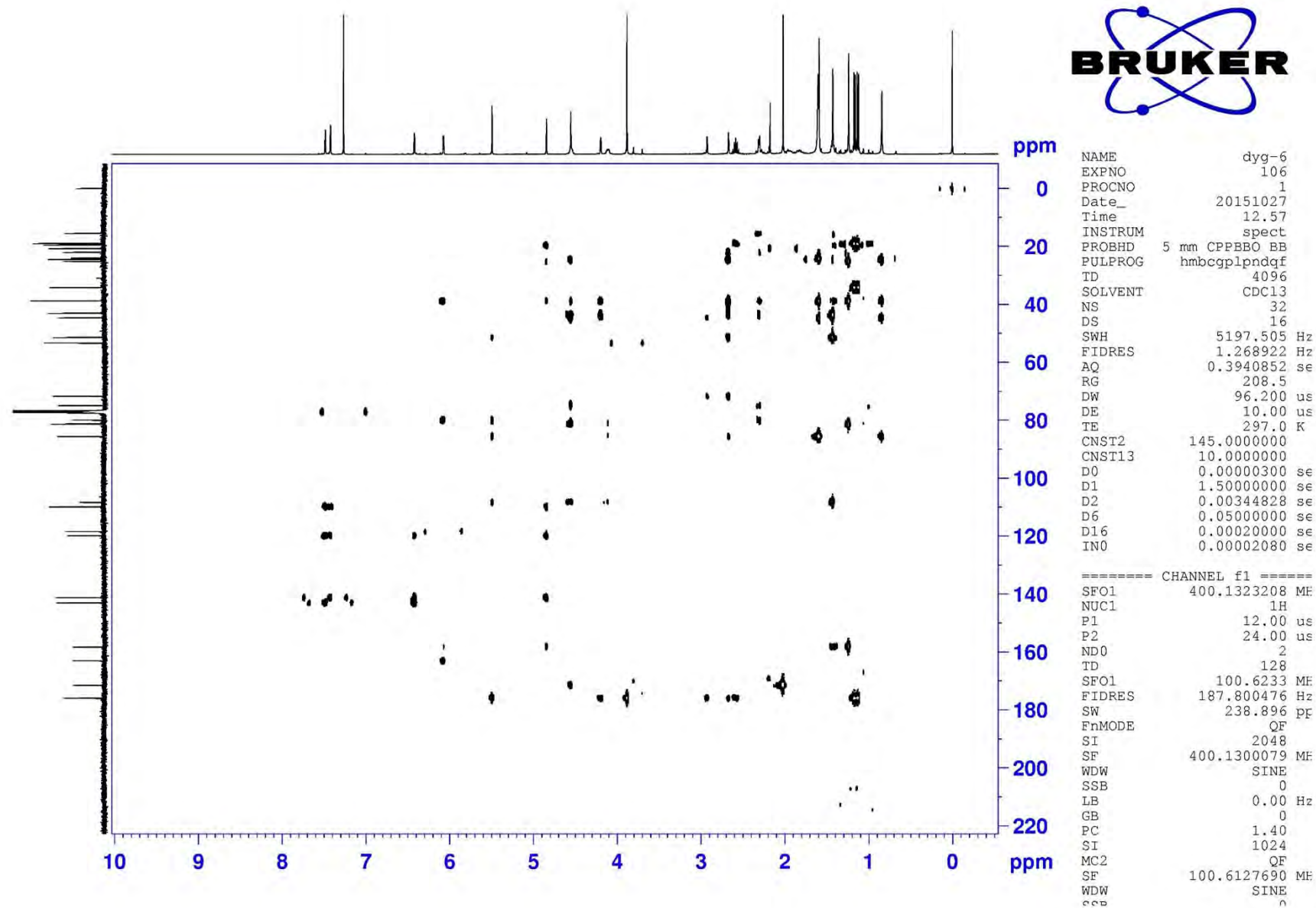
HSQC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



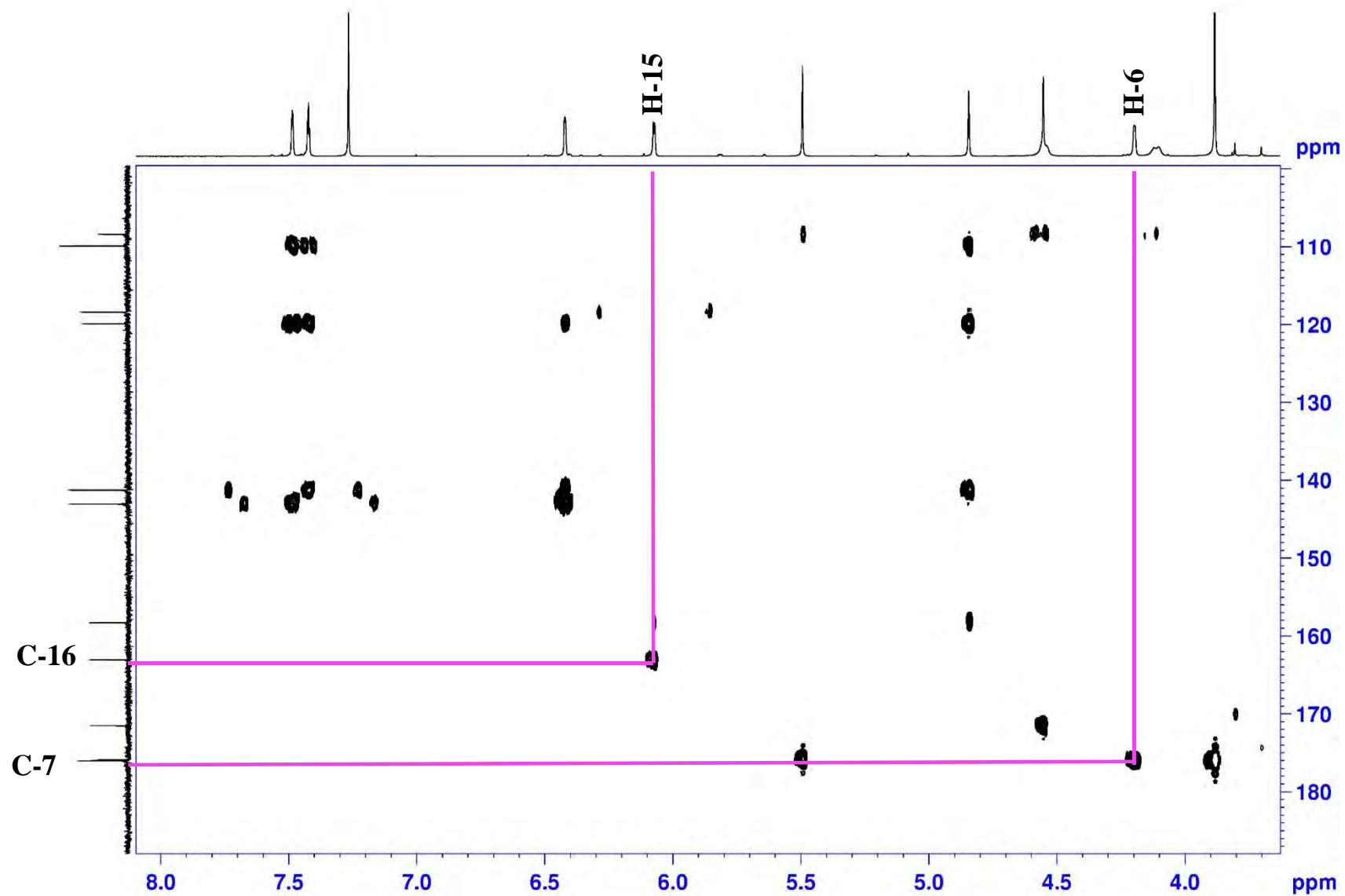
HSQC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



# HMBC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>

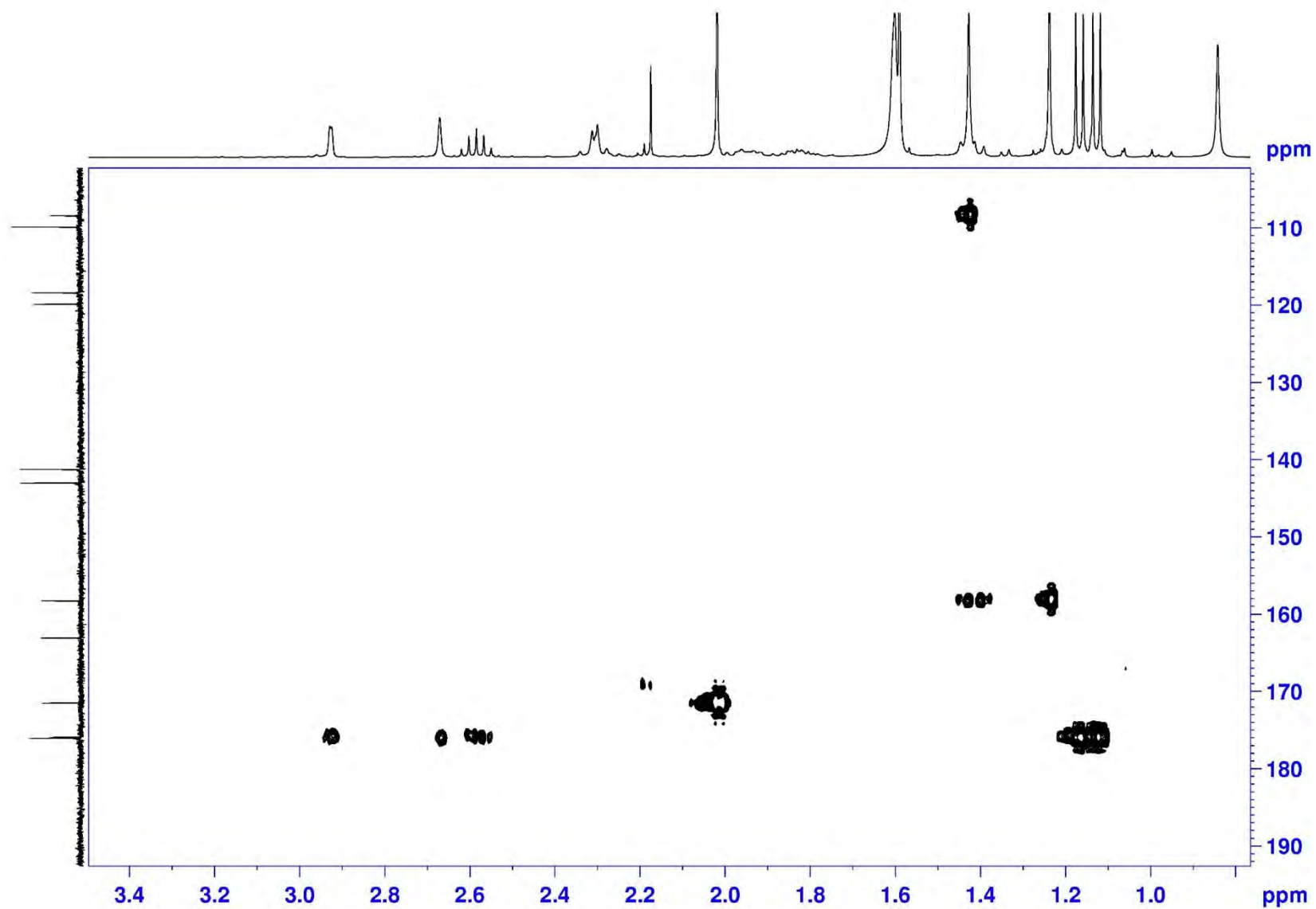


HMBC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>

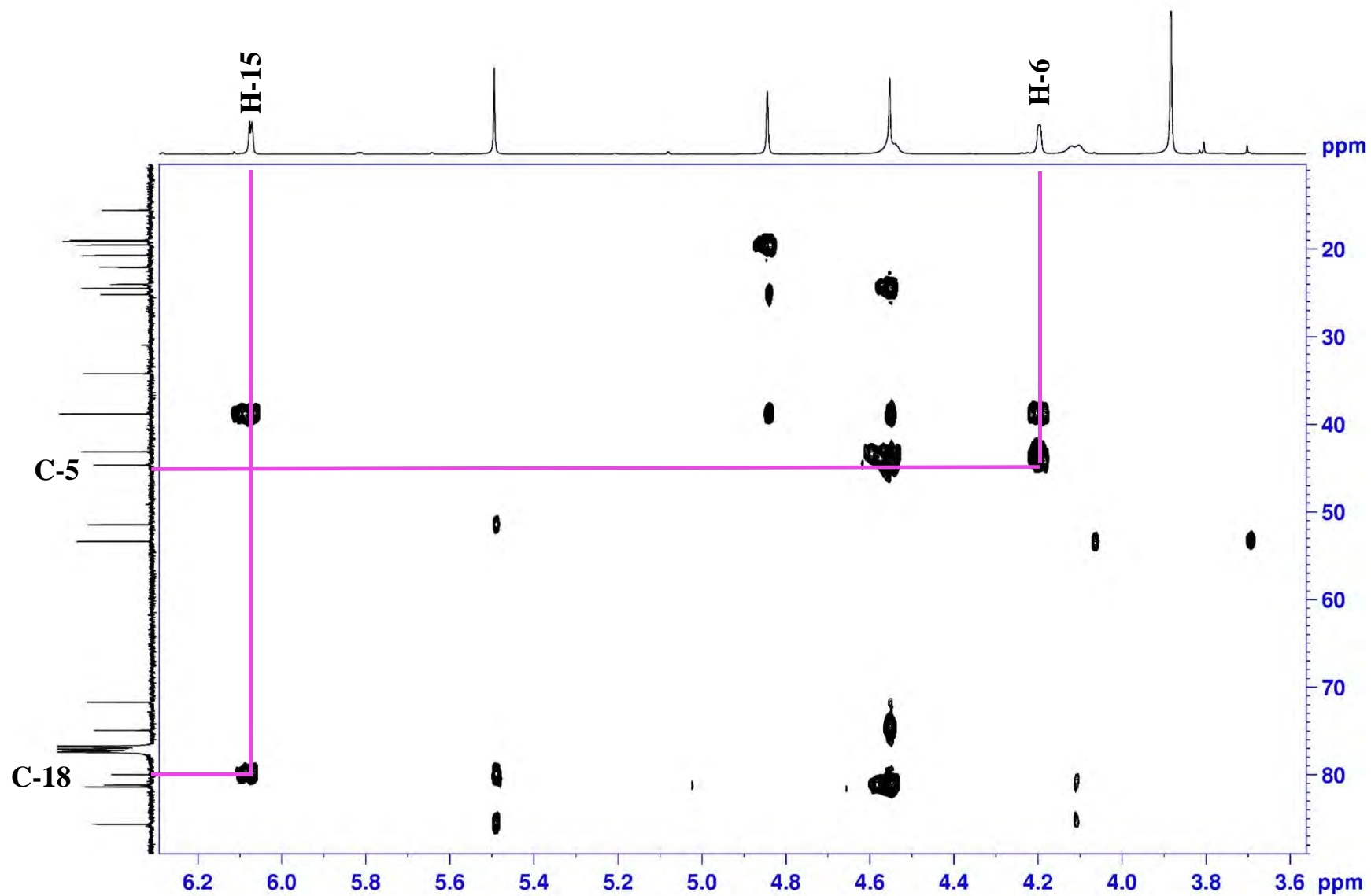




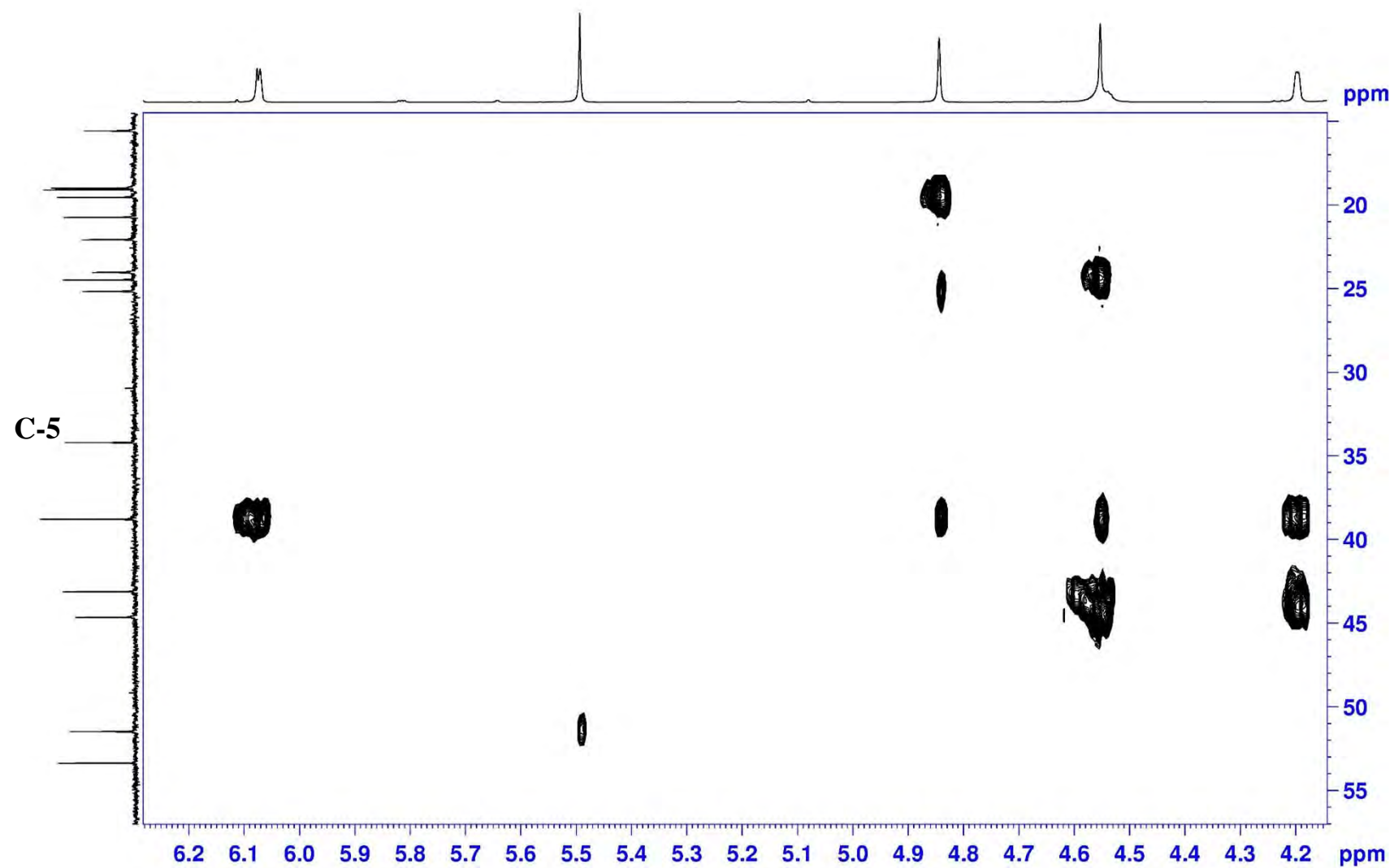
HMBC (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



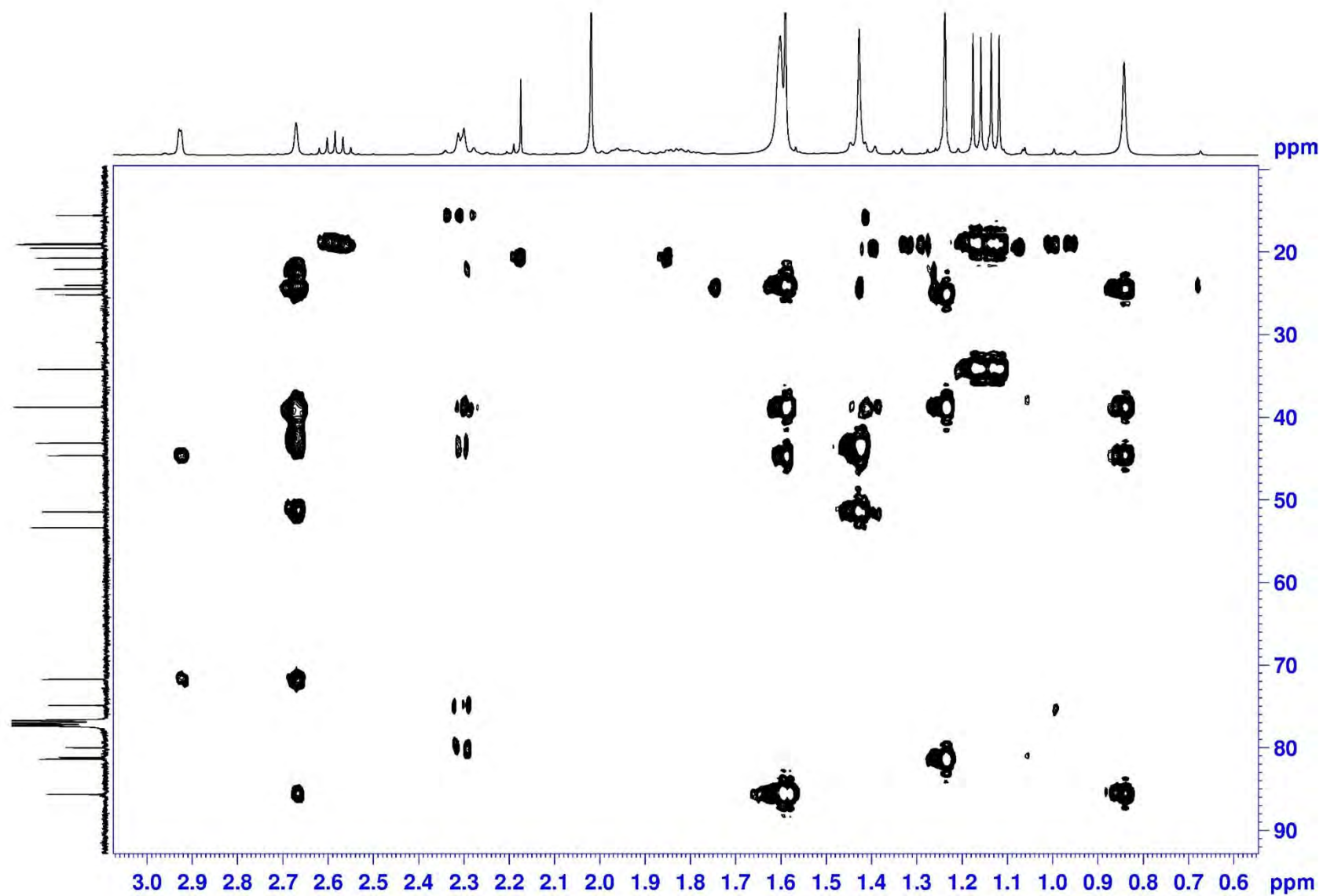
HMBC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



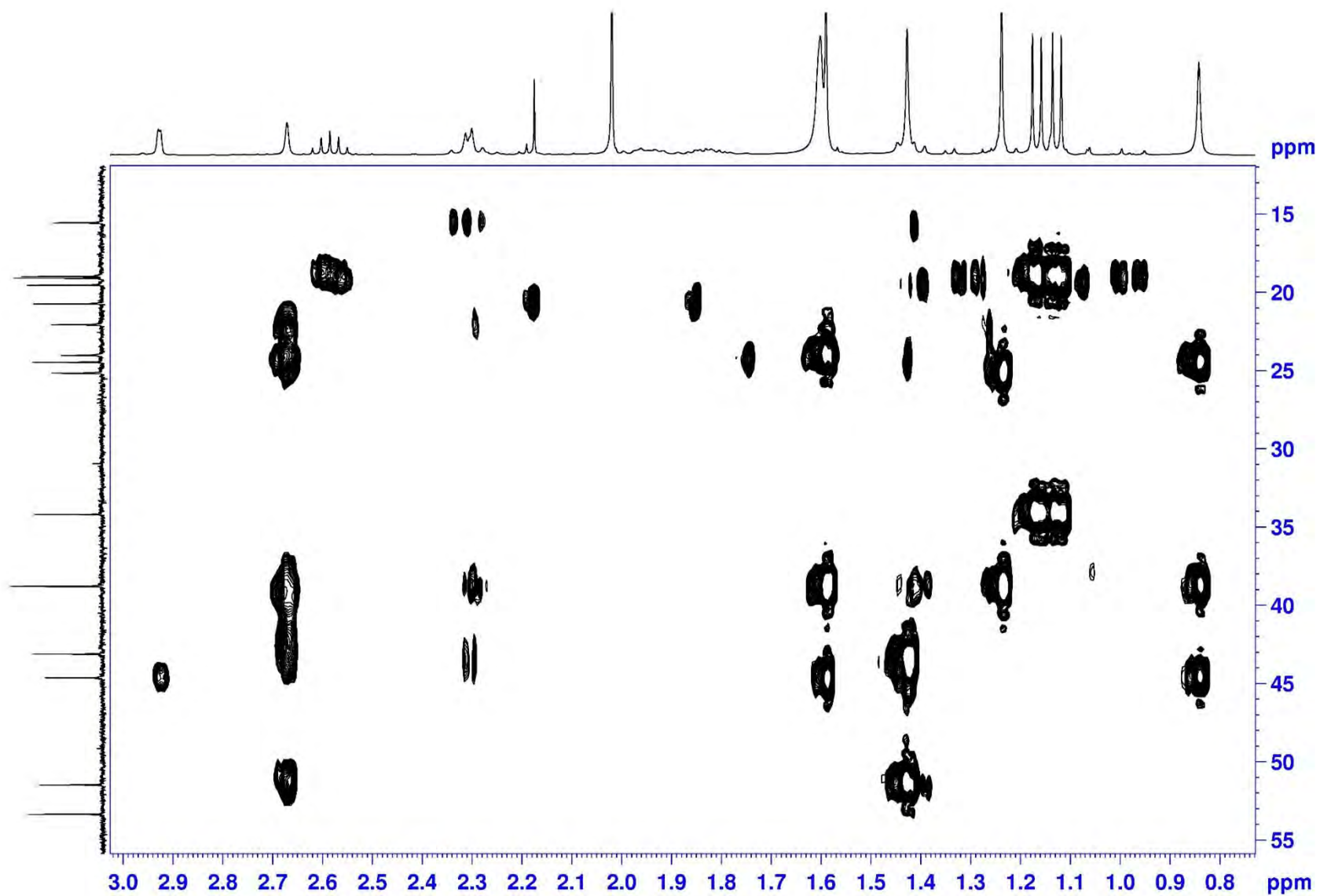
HMBC (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



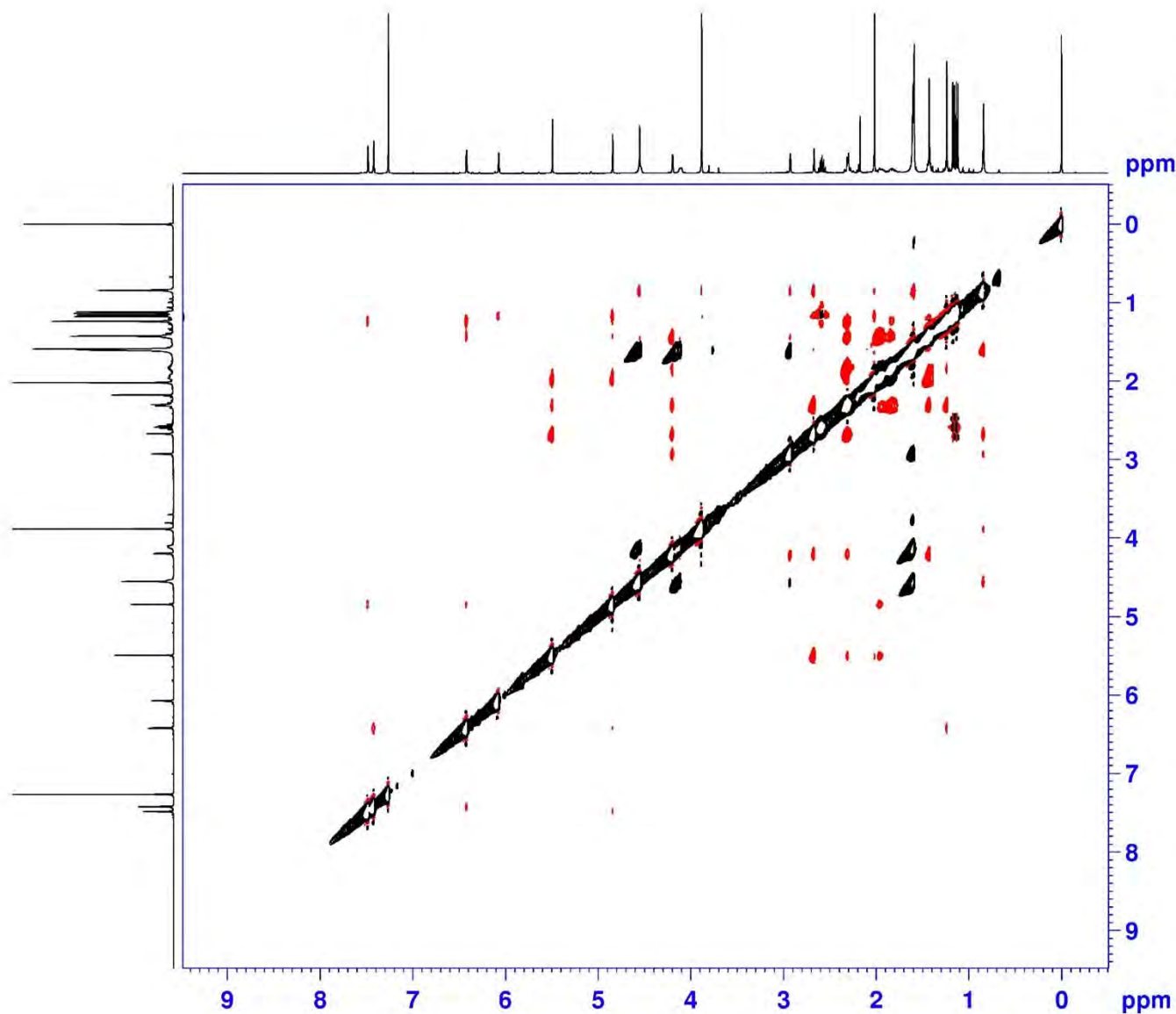
HMBC (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



HMBC (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



# NOESY (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>



```

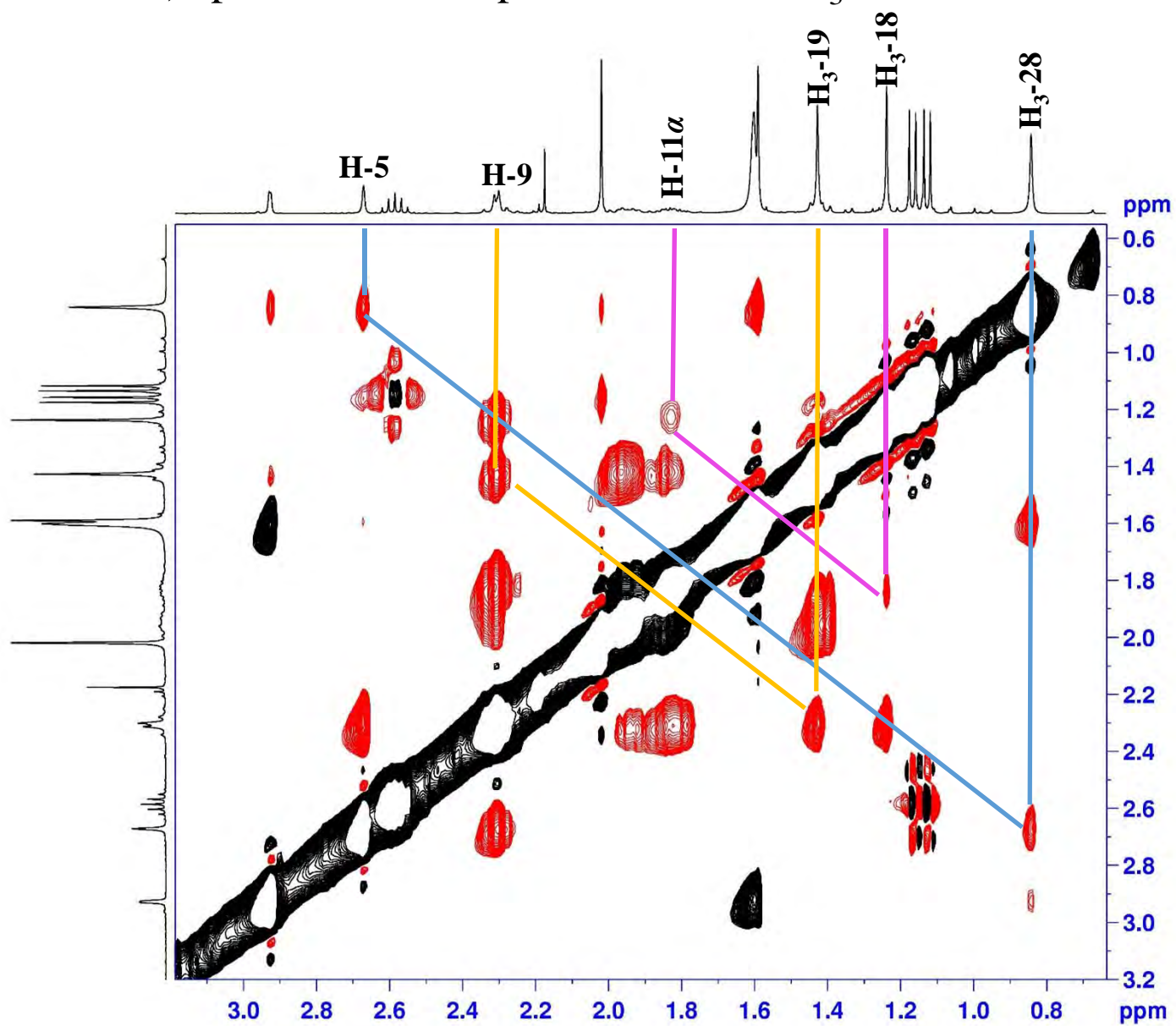
NAME          dyg-6
EXPNO         107
PROCNO        1
Date_         20151027
Time          15.13
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpphph
TD            2048
SOLVENT       CDCl3
NS            16
DS            32
SWH           4000.000 H
FIDRES        1.953125 H
AQ            0.2560500 s
RG            208.5
DW            125.000 u
DE            10.00 u
TE            297.0 K
D0            0.00010972 s
D1            1.99385595 s
D8            0.30000001 s
D11           0.03000000 s
D12           0.00002000 s
D16           0.00020000 s
IN0           0.00025000 s
    
```

```

===== CHANNEL f1 =====
SFO1      400.1318006 M
NUC1       1H
P1         12.00 u
P2         24.00 u
P17        2500.00 u
ND0         1
TD          167
SFO1      400.1318 M
FIDRES     23.952095 H
SW          9.997 p
FnMODE     States-TPPI
SI          1024
SF         400.1300070 M
WDW         QSINE
SSB          2
LB           0.00 H
GB           0
PC           1.00
SI          1024
MC2        States-TPPI
SF         400.1300070 M
WDW         QSINE
SSB          2
    
```



NOESY (400 MHz) spectrum of compound **14** in  $\text{CDCl}_3$



NOESY (400 MHz) spectrum of compound **14** in CDCl<sub>3</sub>

