

Supporting Information-3:

Scaffold diversity-oriented synthesis of limonoid dimers: discovery of an axially chiral agent with *in vivo* anti-breast cancer activity

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Copies of HRESIMS, UV, IR, 1D and 2D NMR spectra of compounds 3, 3', 4, and 4'.

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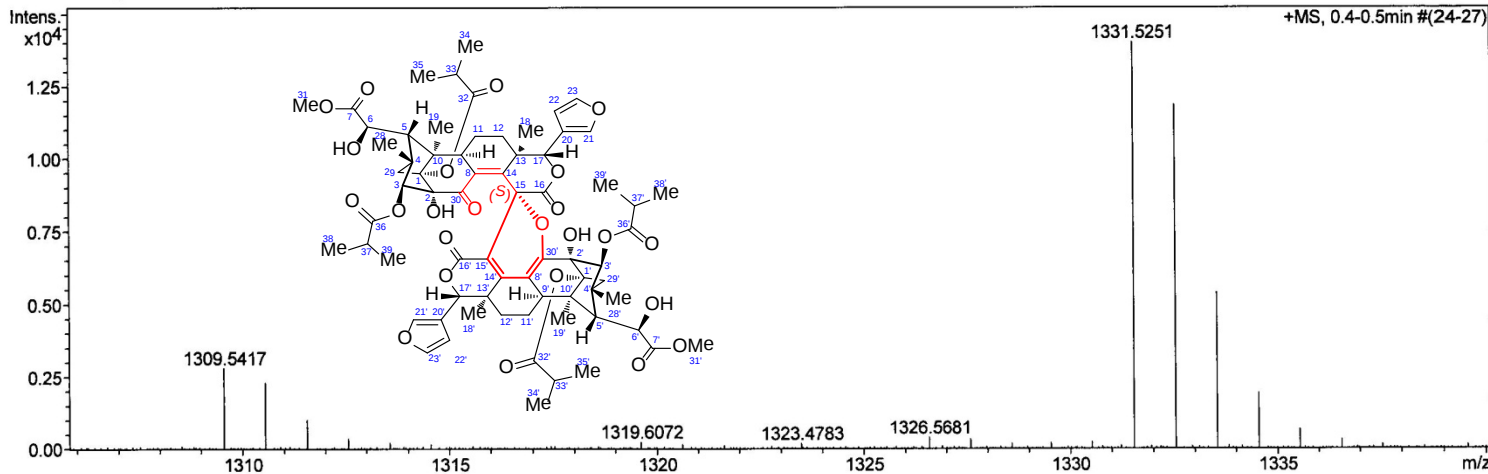
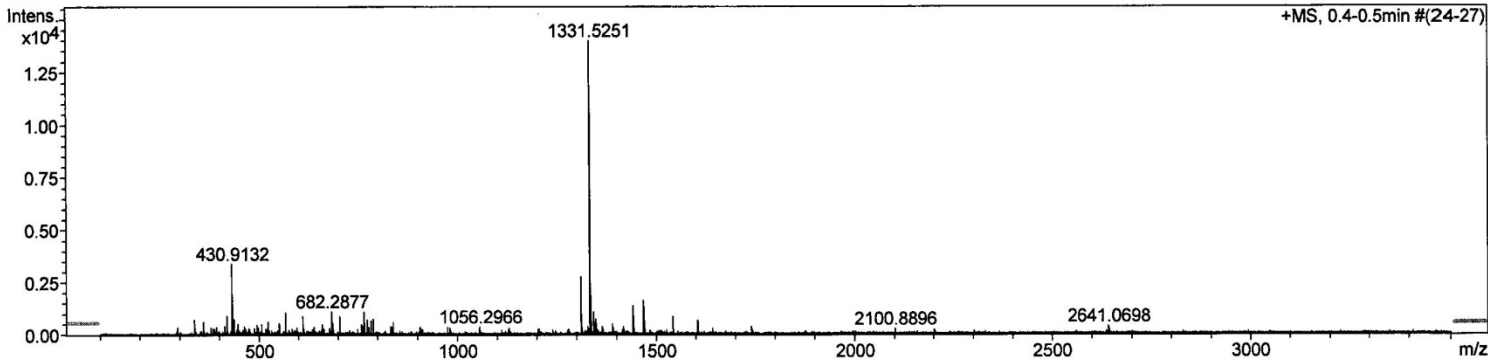
HR-ESIMS for compound 3

Generic Display Report

Analysis Info

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Comment

Acquisition Date 9/7/2015 11:45:55 PM
Operator SCSIO
Instrument maXis



HR-ESIMS for compound 3

Mass Spectrum SmartFormula Report

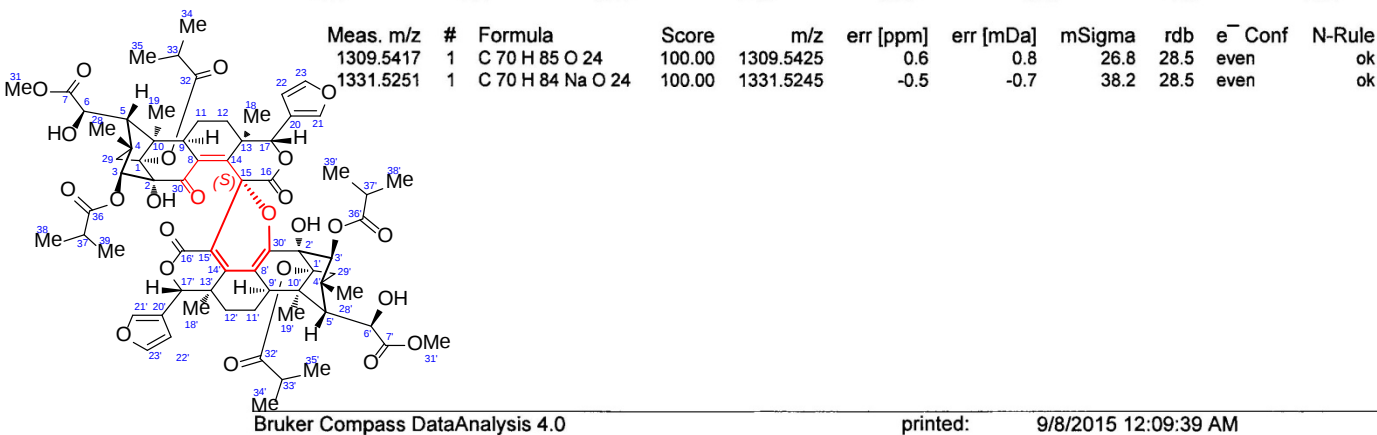
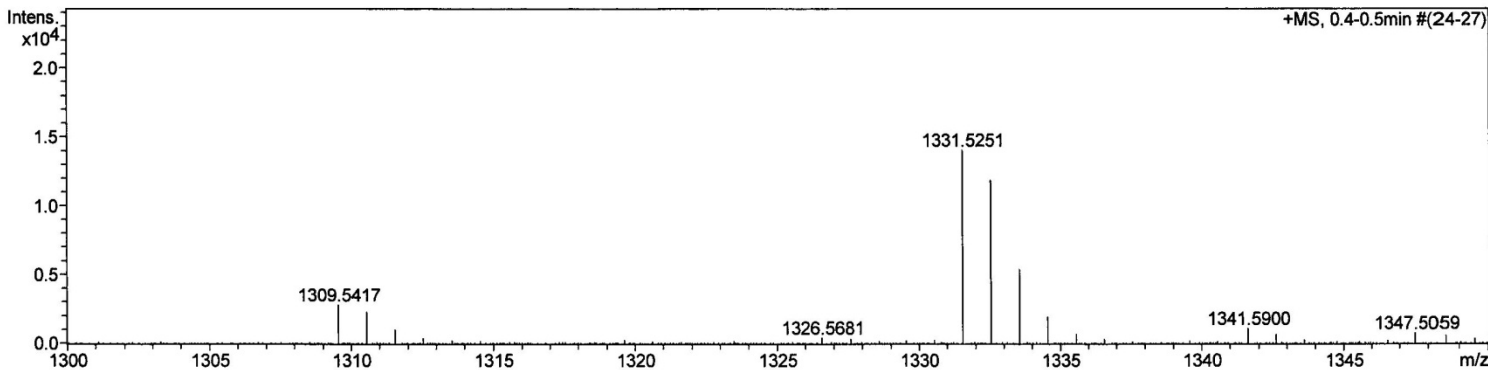
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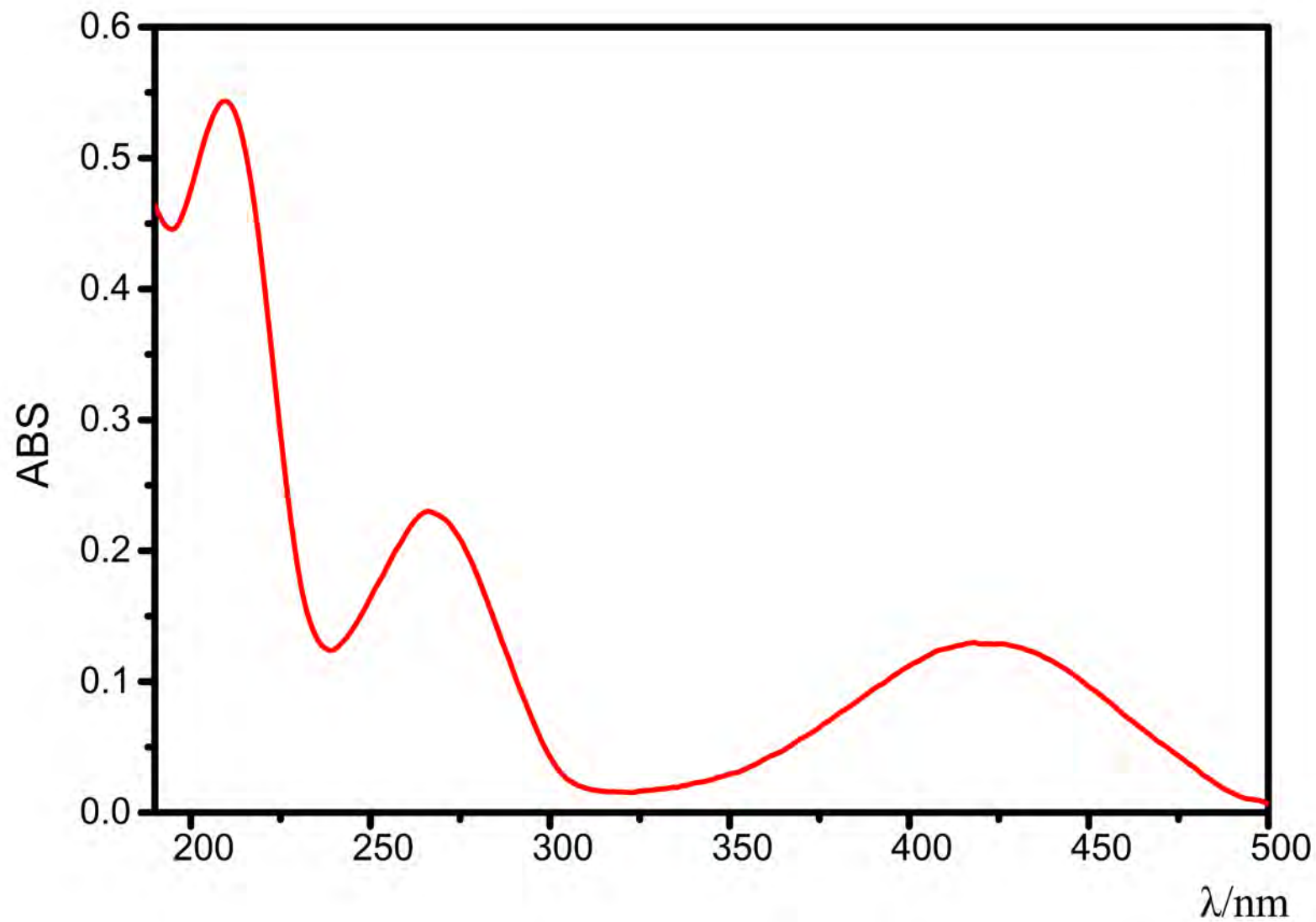
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Instrument / Ser# maXis 29

Acquisition Parameter

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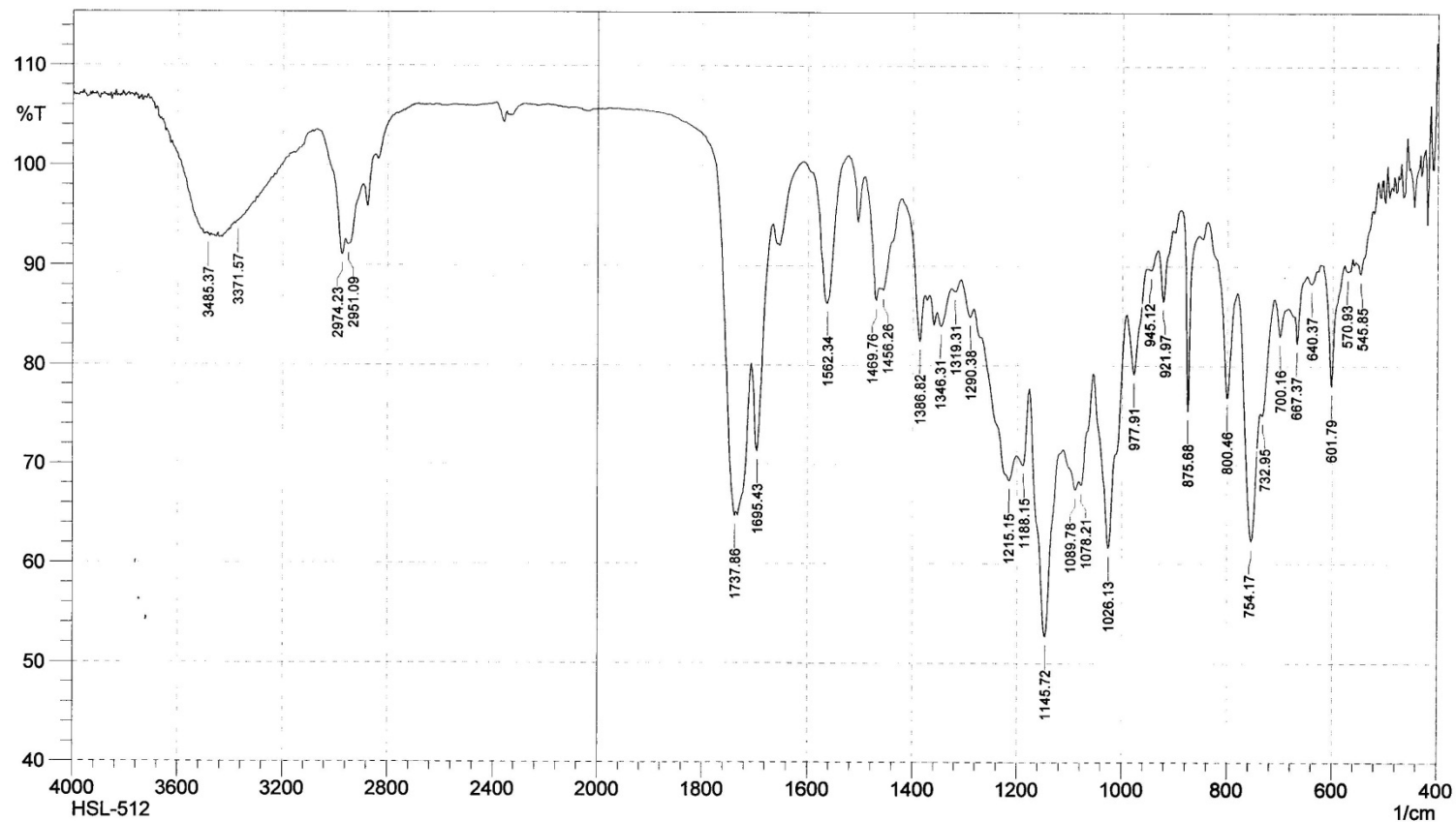


UV spectrum for compound **3** in CH₃CN



IR spectrum for compound 3

SHIMADZU

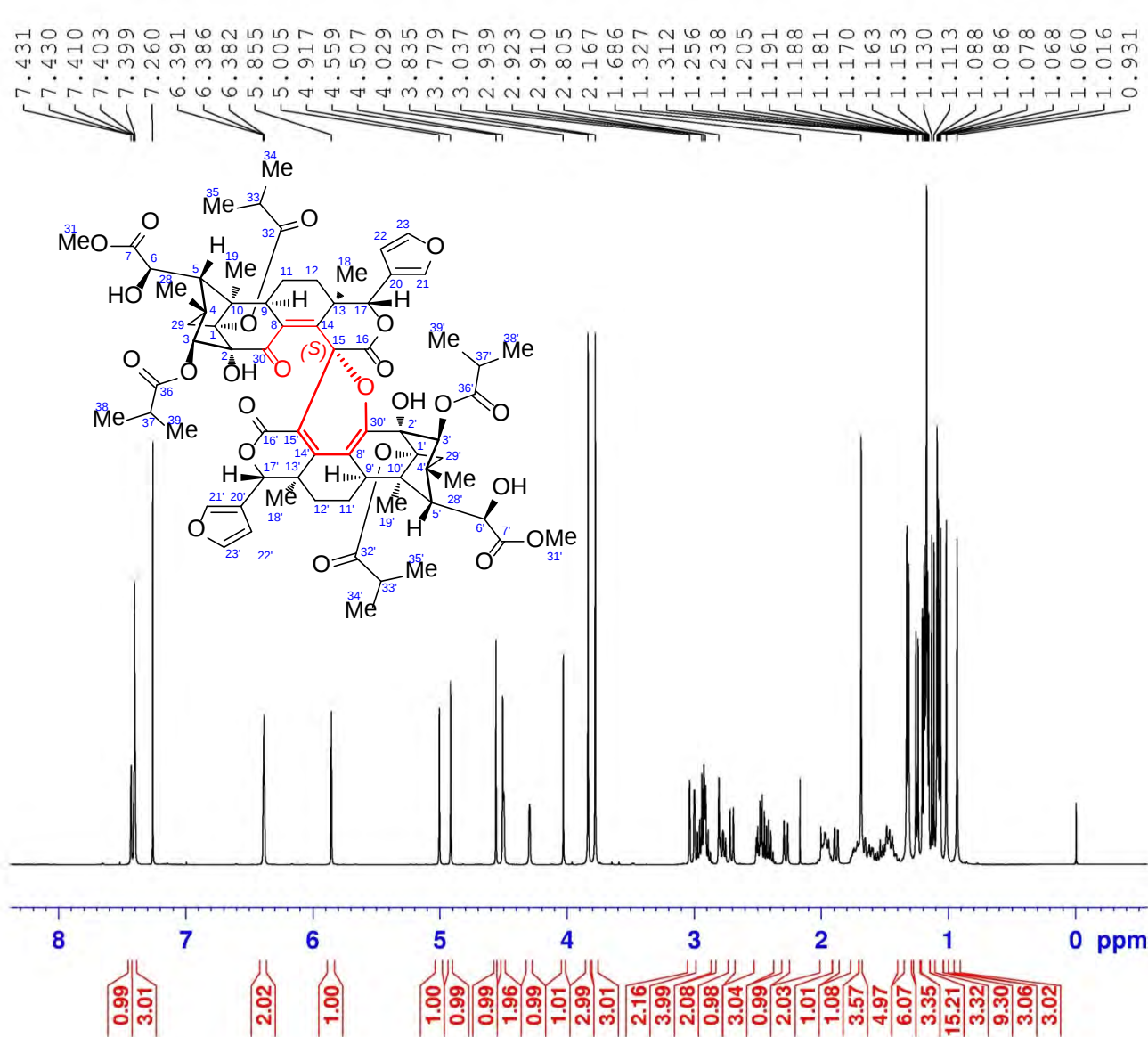


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^1H NMR (400 MHz) spectrum of compound **3** in CDCl_3

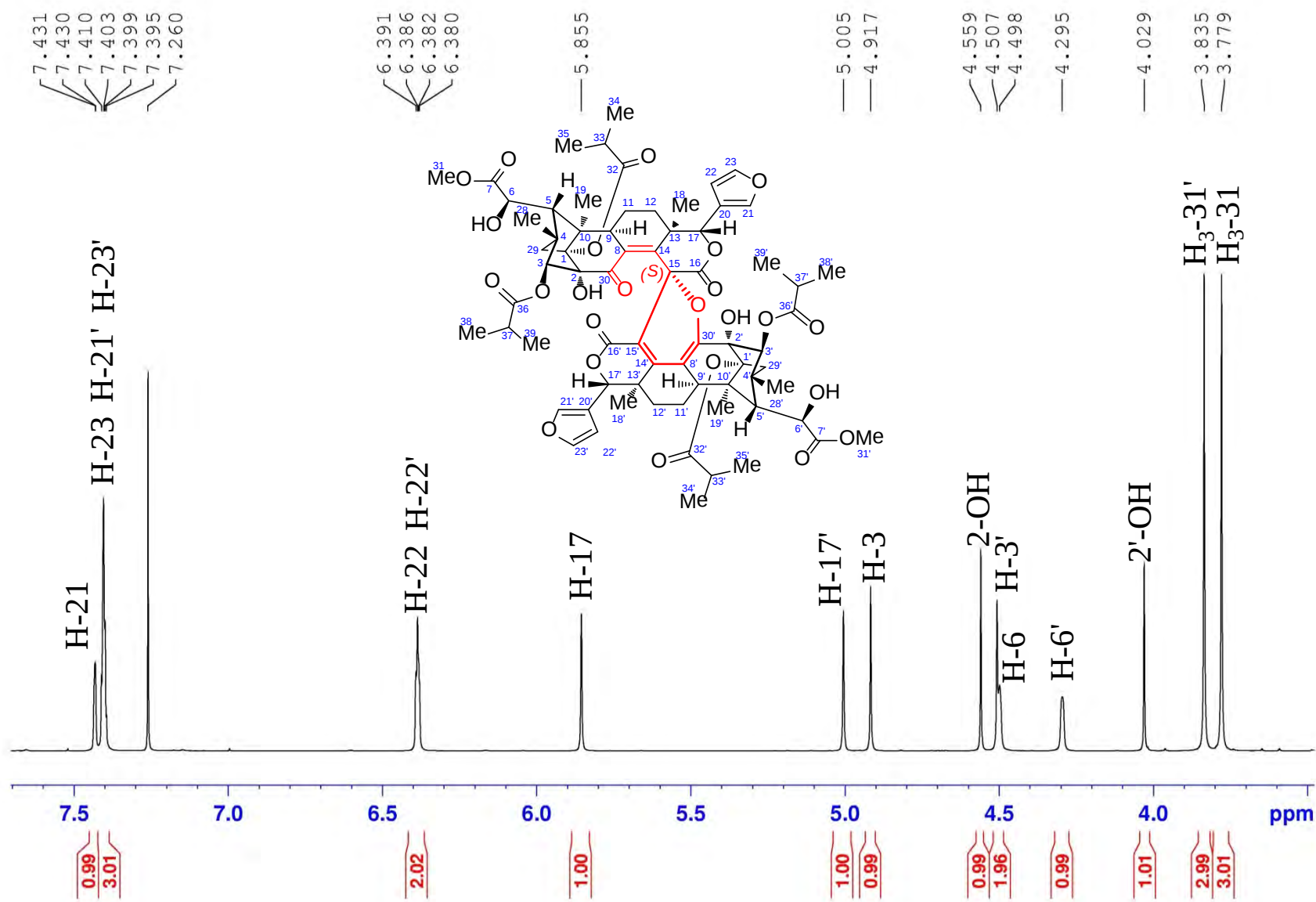


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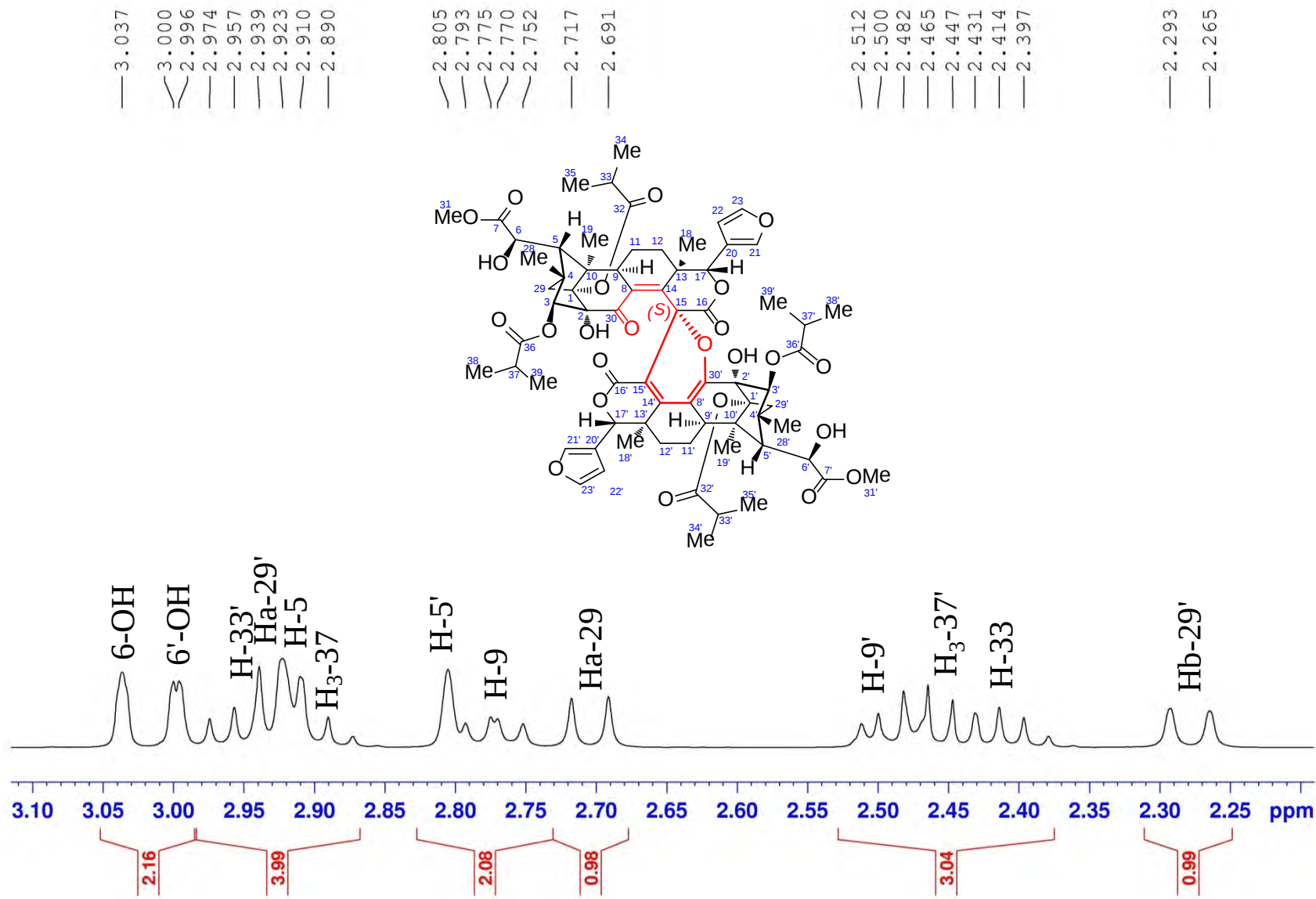
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EXPNO     1
PROCNO    1
Date_     20150812
Time      13.24
INSTRUM   spect
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PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        73.92
DW        60.800 usec
DE        10.00 usec
TE        297.0 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1      1H
P1        12.00 usec
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SF        400.1300095 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
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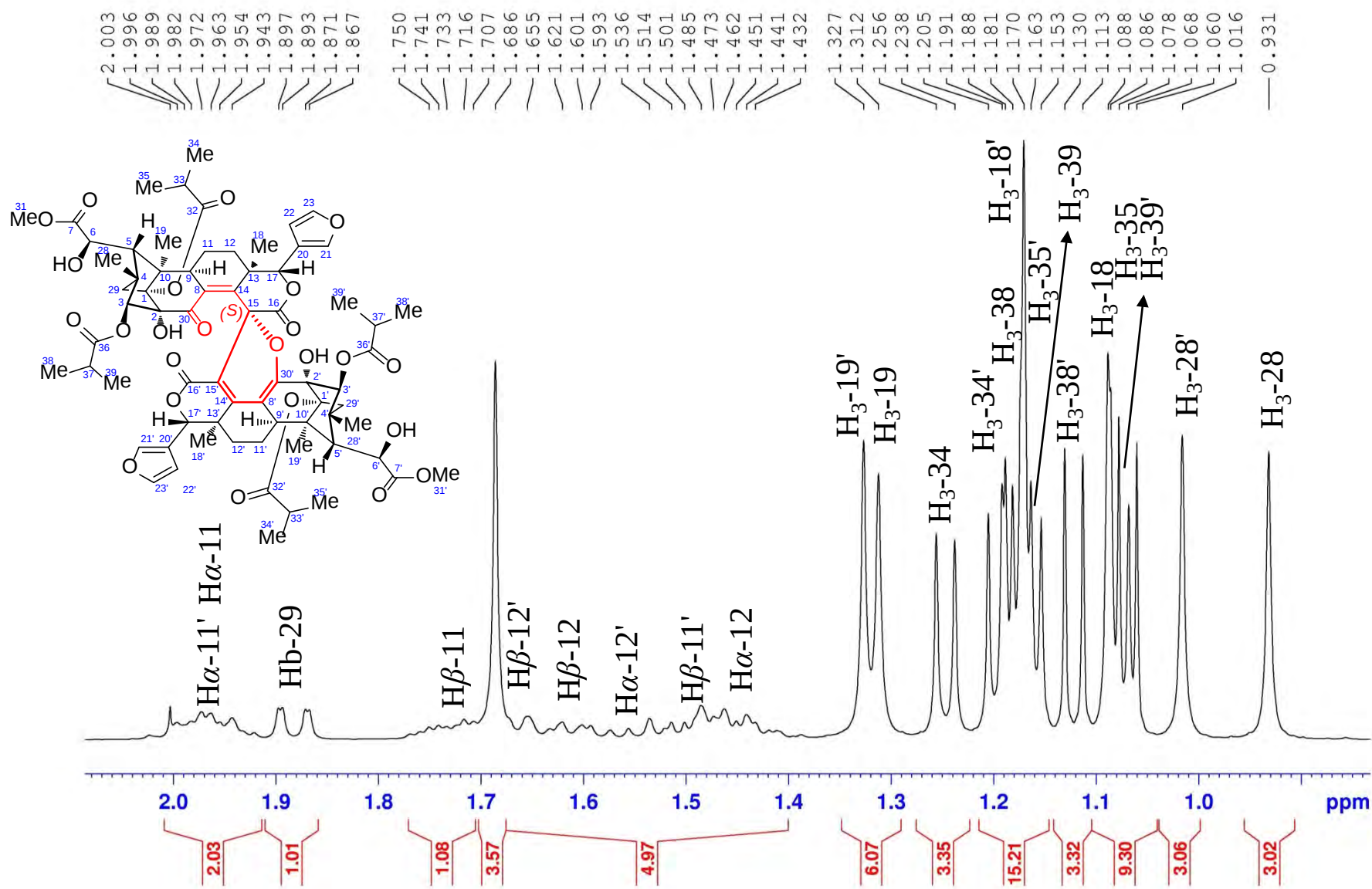
¹H NMR (400 MHz) spectrum of compound **3** in CDCl₃



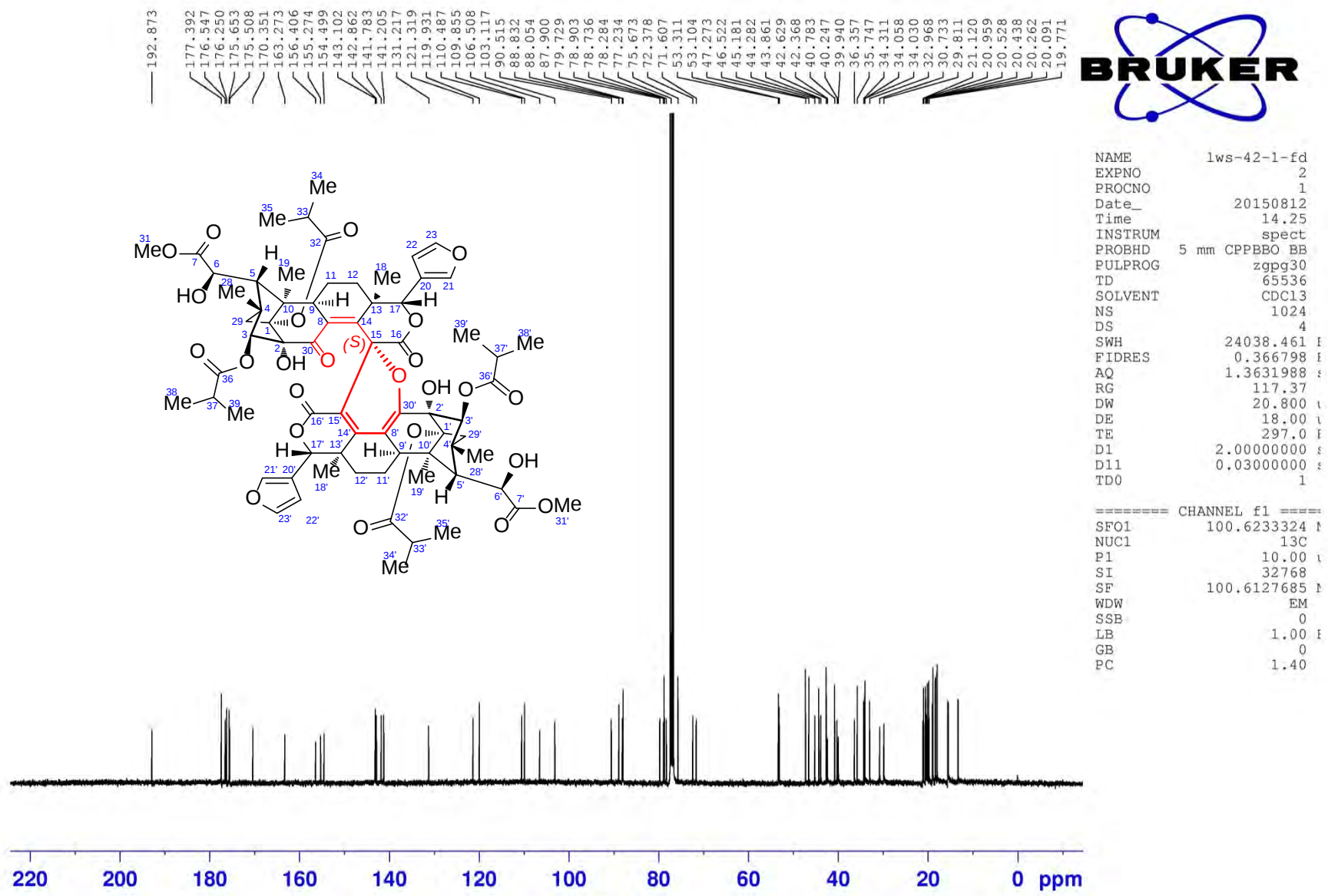
¹H NMR (400 MHz) spectrum of compound **3** in CDCl₃



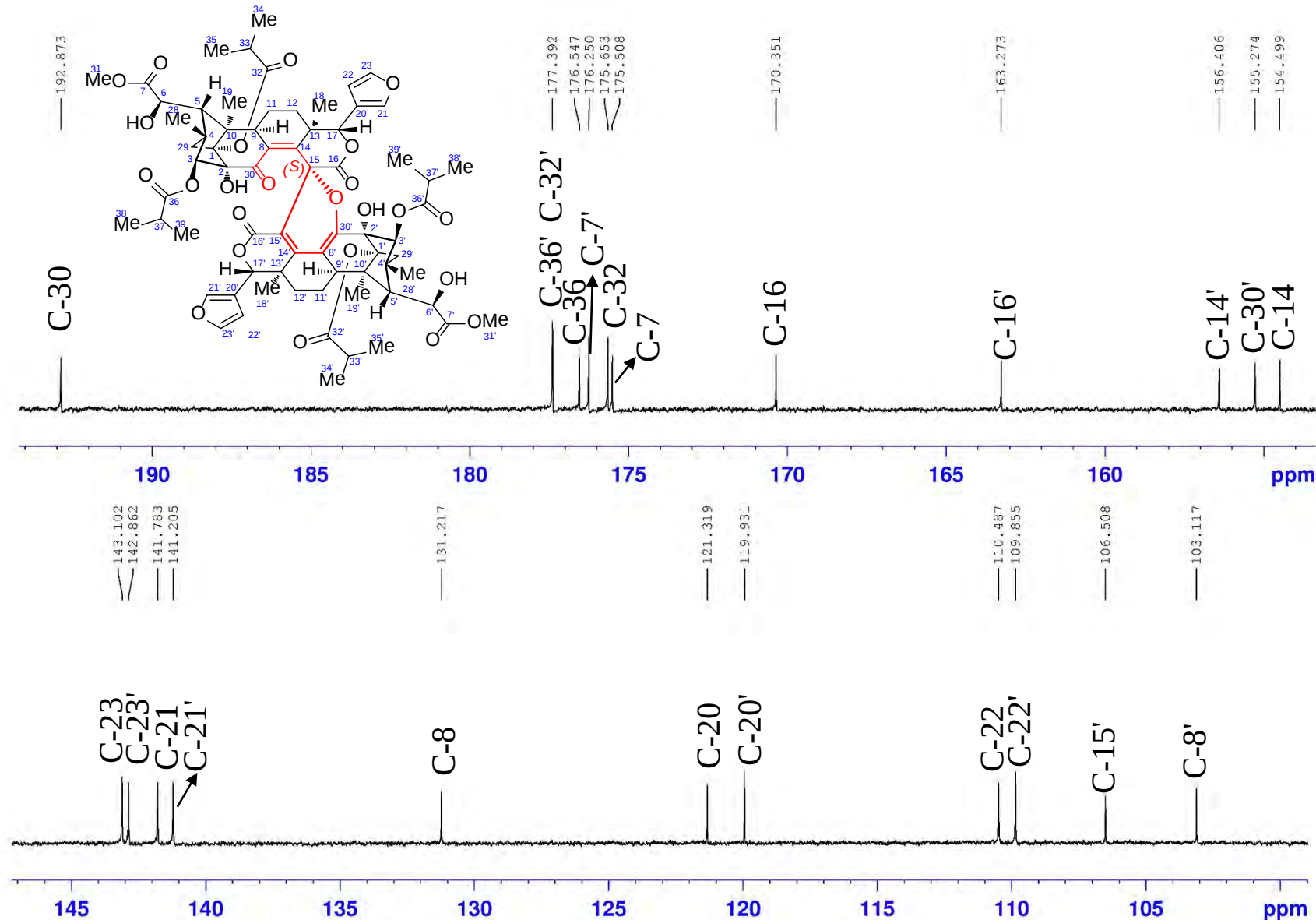
¹H NMR (400 MHz) spectrum of compound **3** in CDCl₃



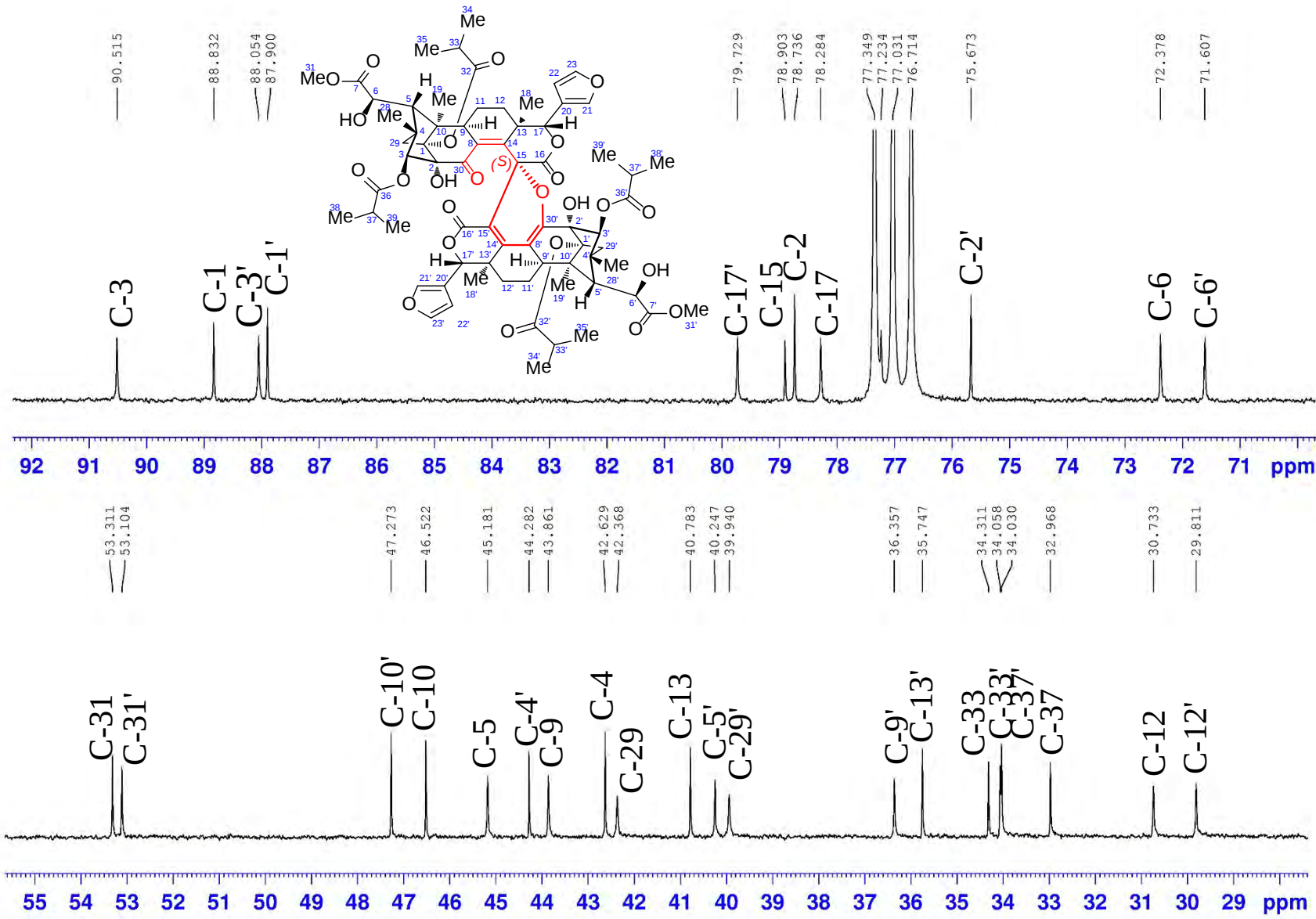
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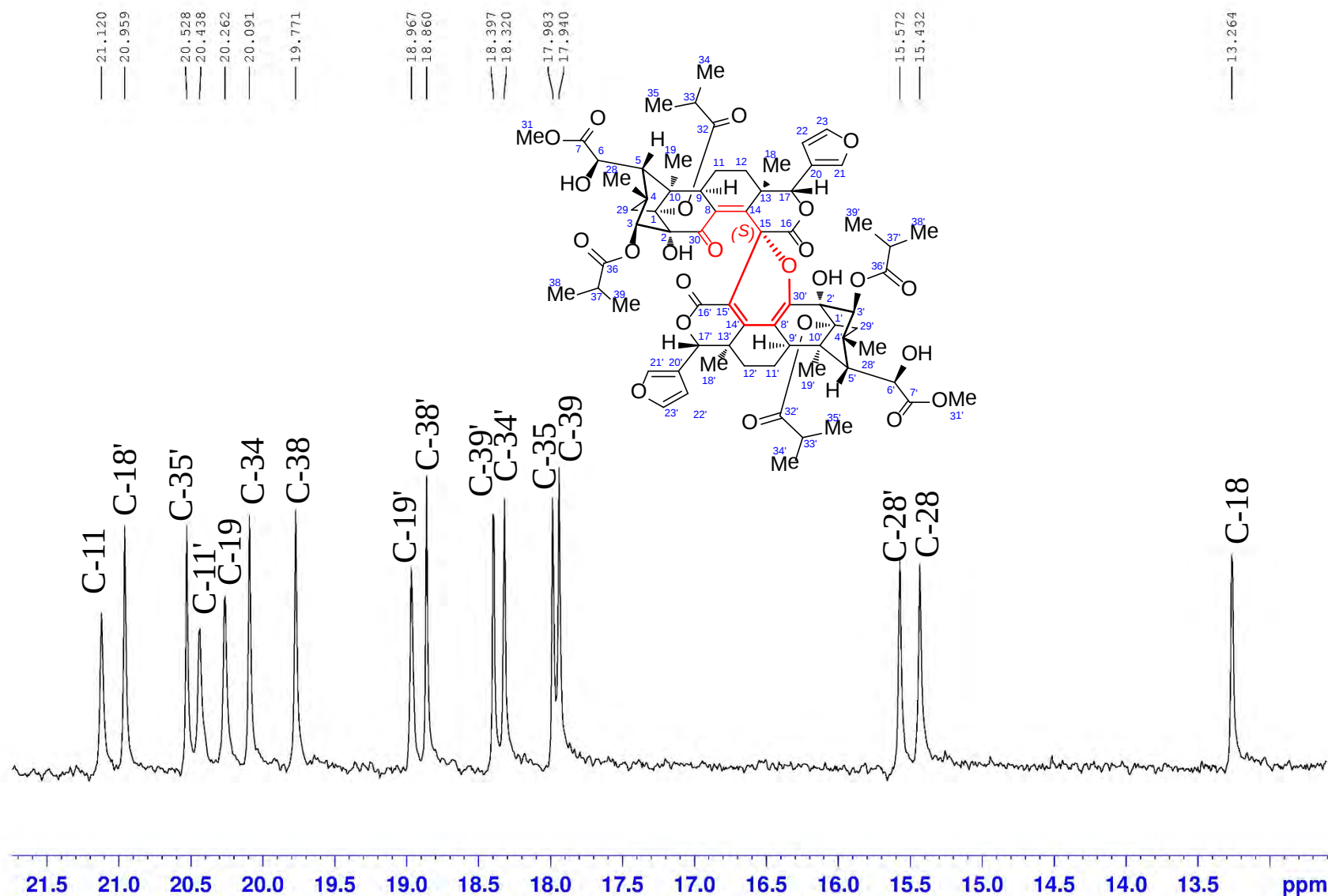
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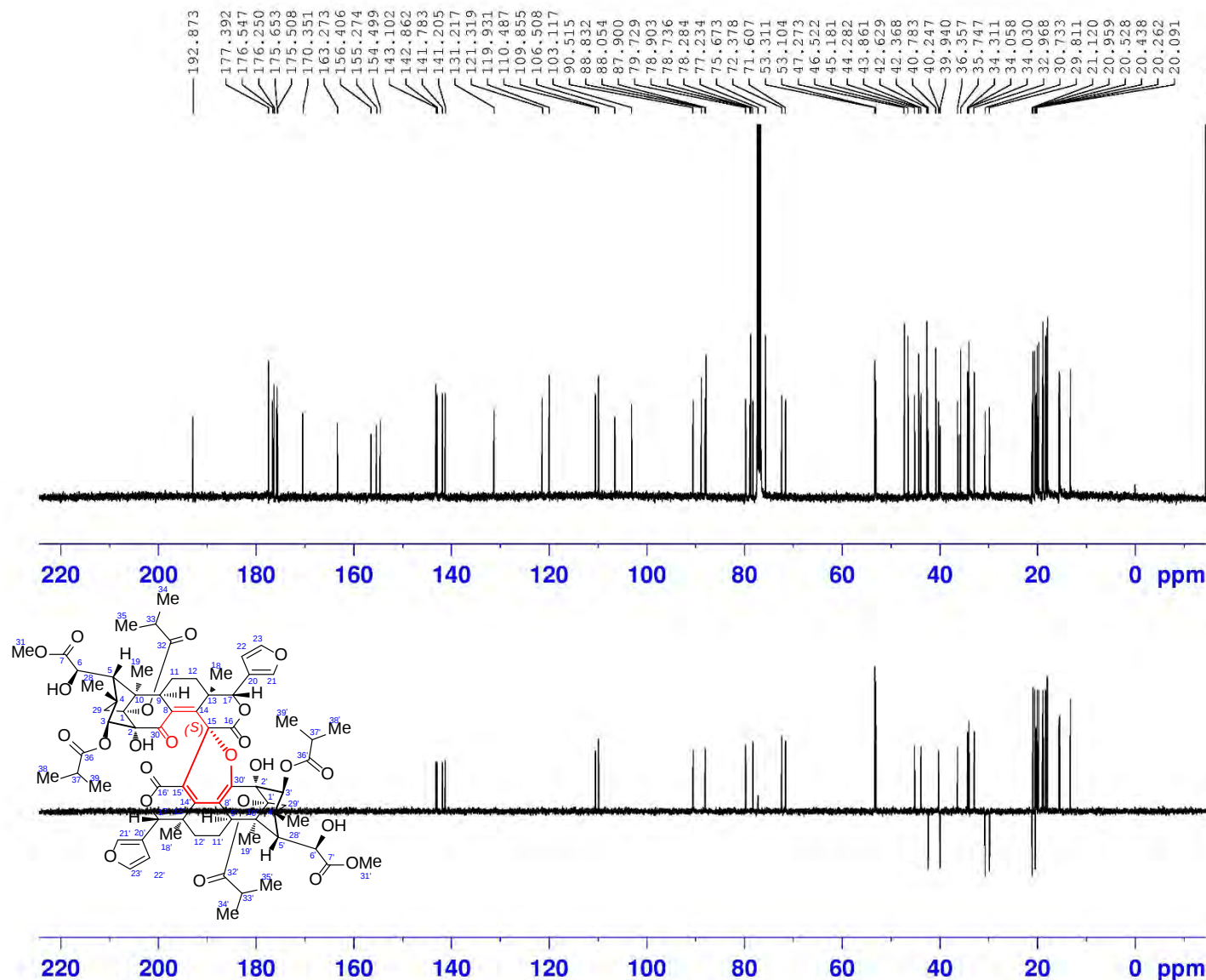
¹³C NMR (100 MHz) spectrum of compound **3** in CDCl₃



¹³C NMR (100 MHz) spectrum of compound **3** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



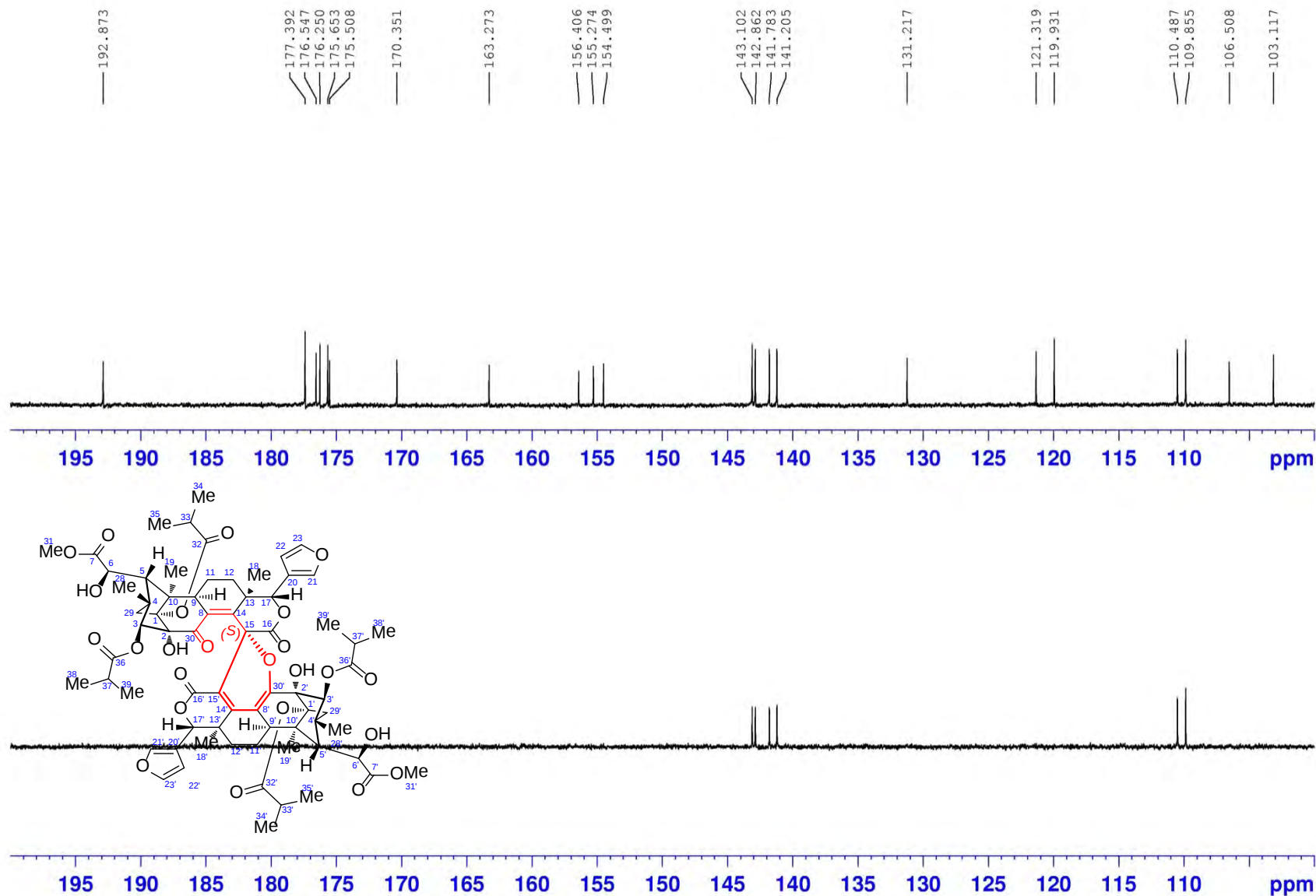
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PROCNO         1
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PULPROG        deptsp135
TD             65536
SOLVENT        CDC13
NS             300
DS             4
SWH            24038.461
FIDRES         0.366798
AQ             1.3631988
RG             91.64
DW             20.800
DE             18.00
TE             297.0
CNST2          145.0000000
D1             2.00000000
D2             0.00344828
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TD0            1
  
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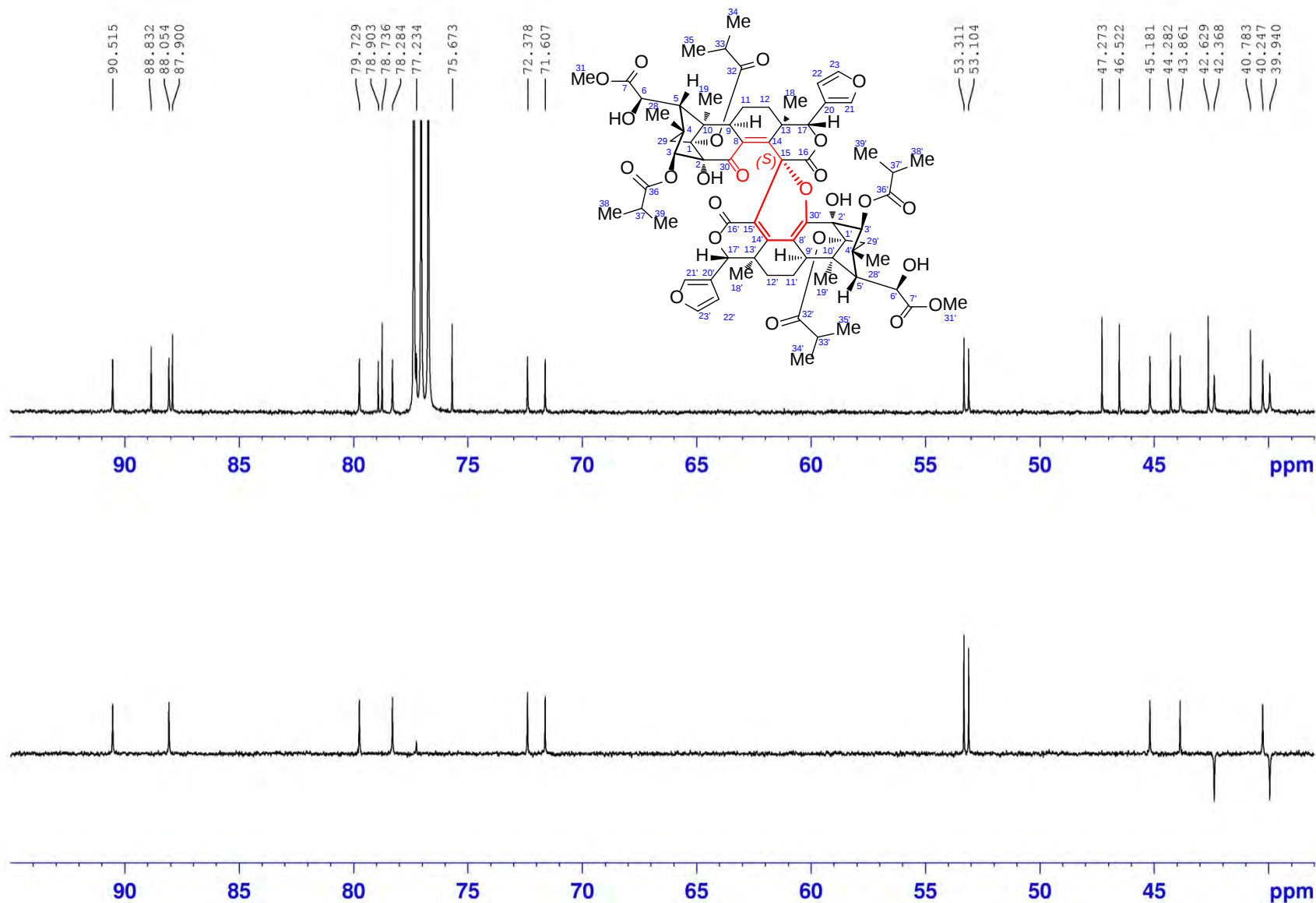
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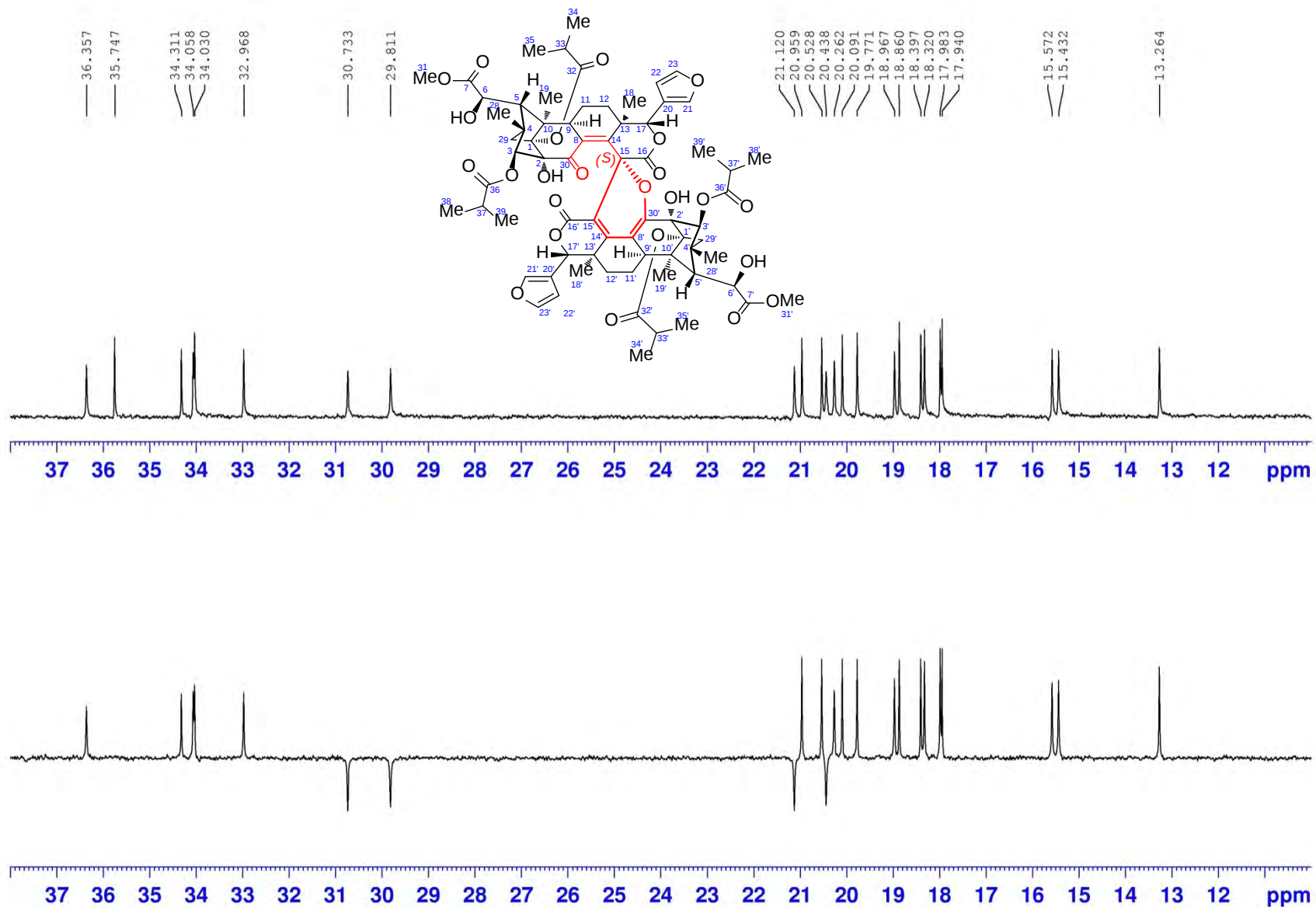
DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



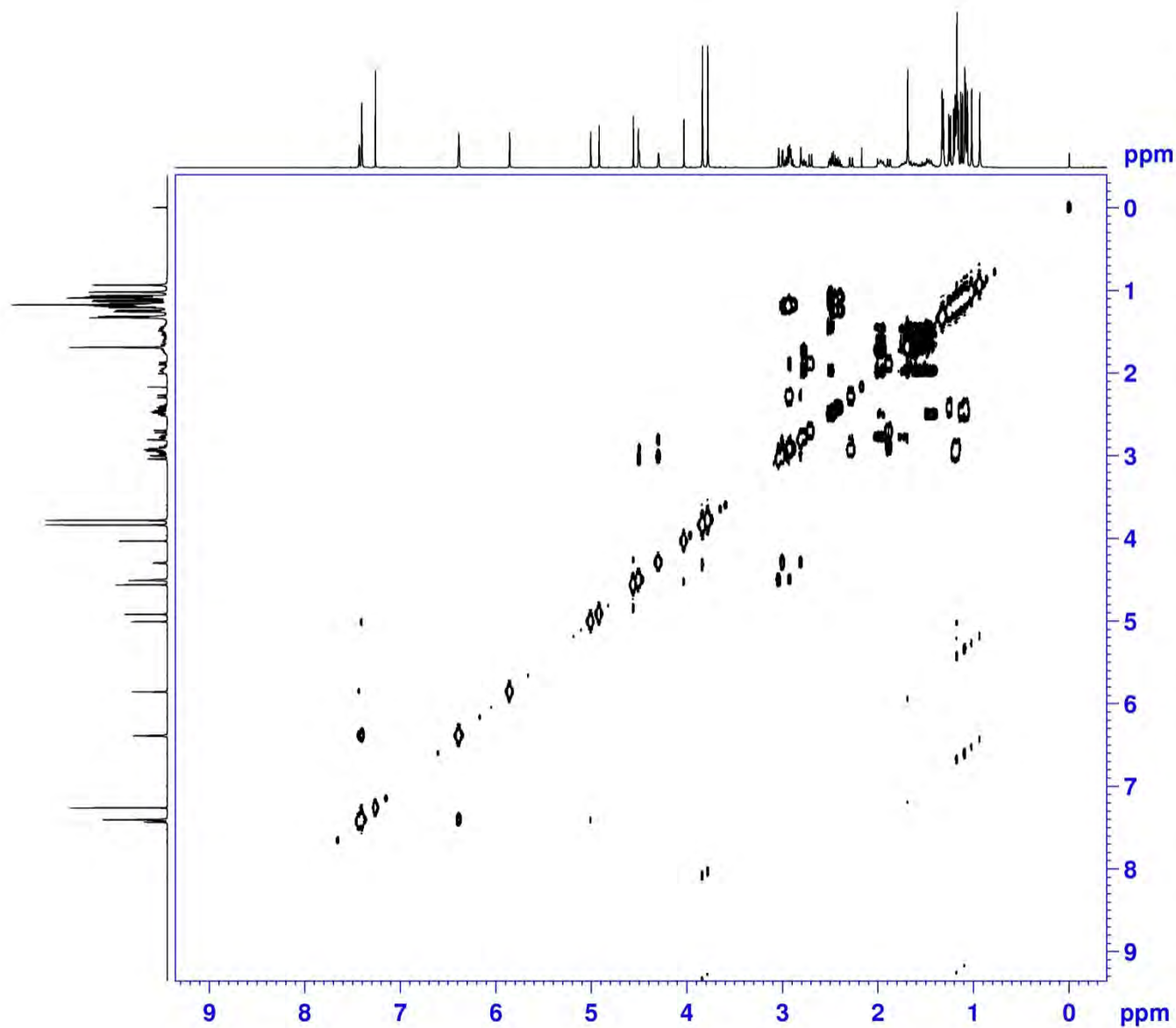
DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



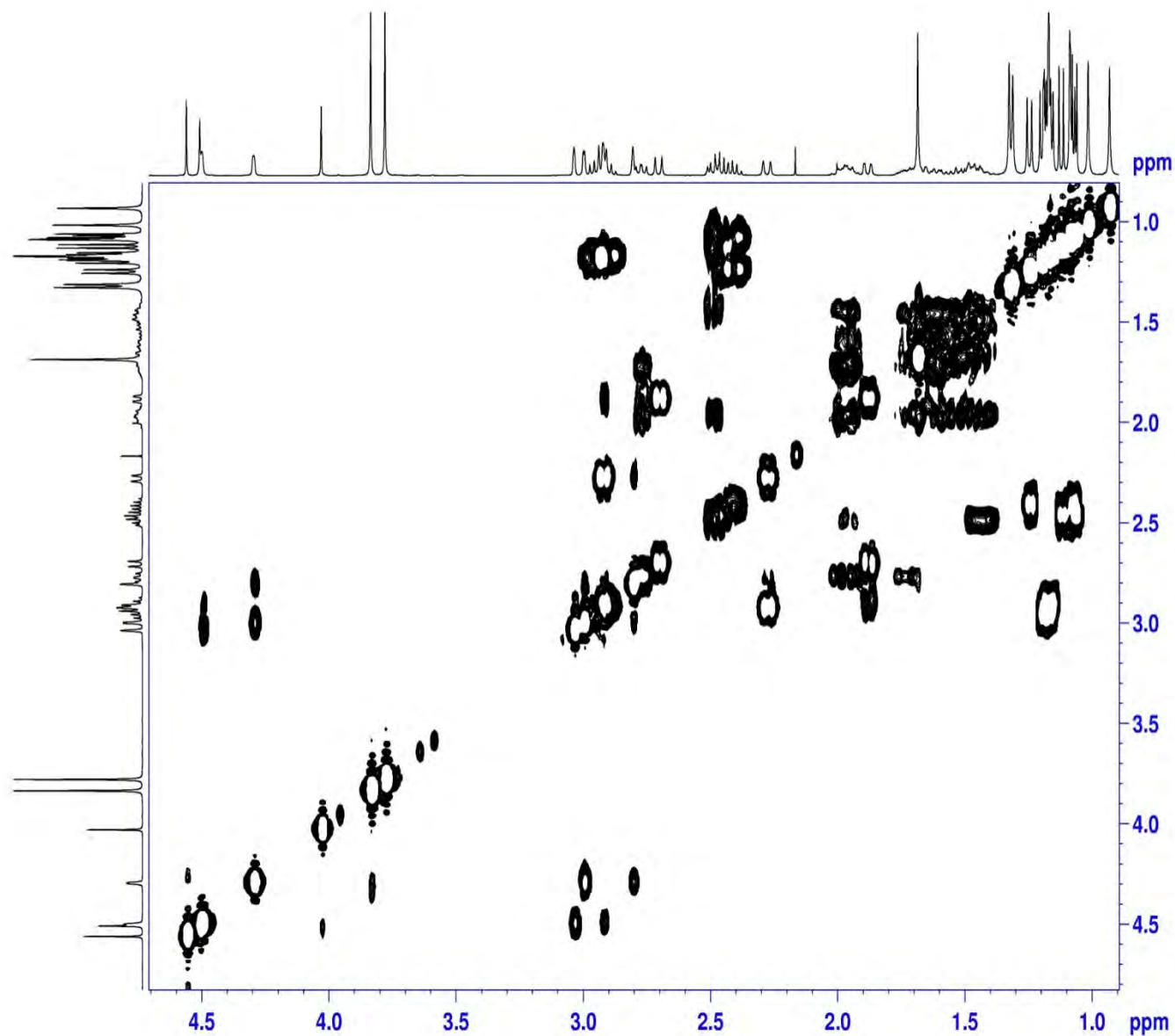
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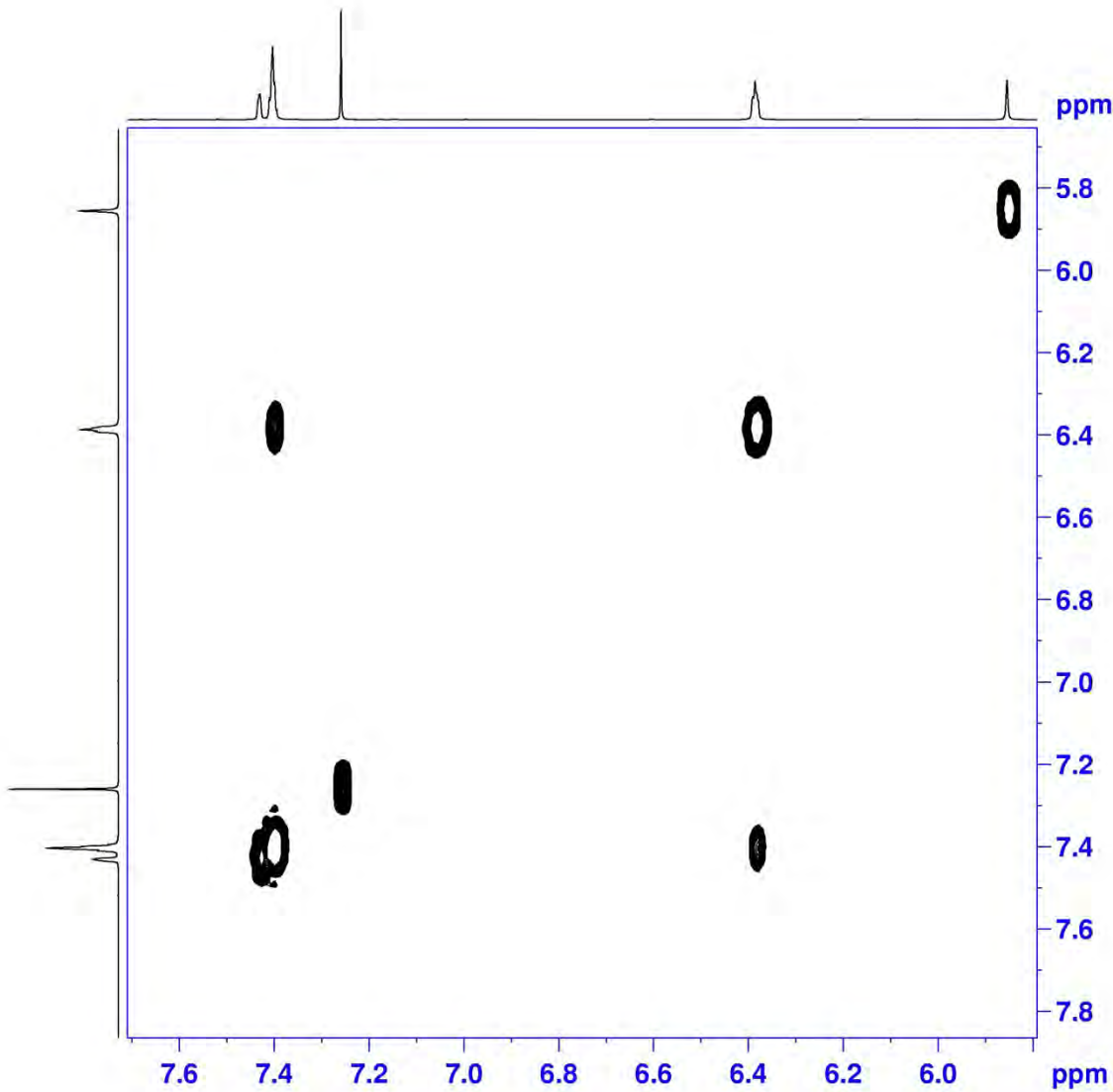
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TD             2048
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DS              8
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FIDRES         1.907349 Hz
AQ             0.2621940 sec
RG             147.94
DW             128.000 usec
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 =====
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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
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SF            400.1300098 MHz
WDW            QSINE
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^1H - ^1H COSY (400 MHz) spectrum of compound **3** in CDCl_3



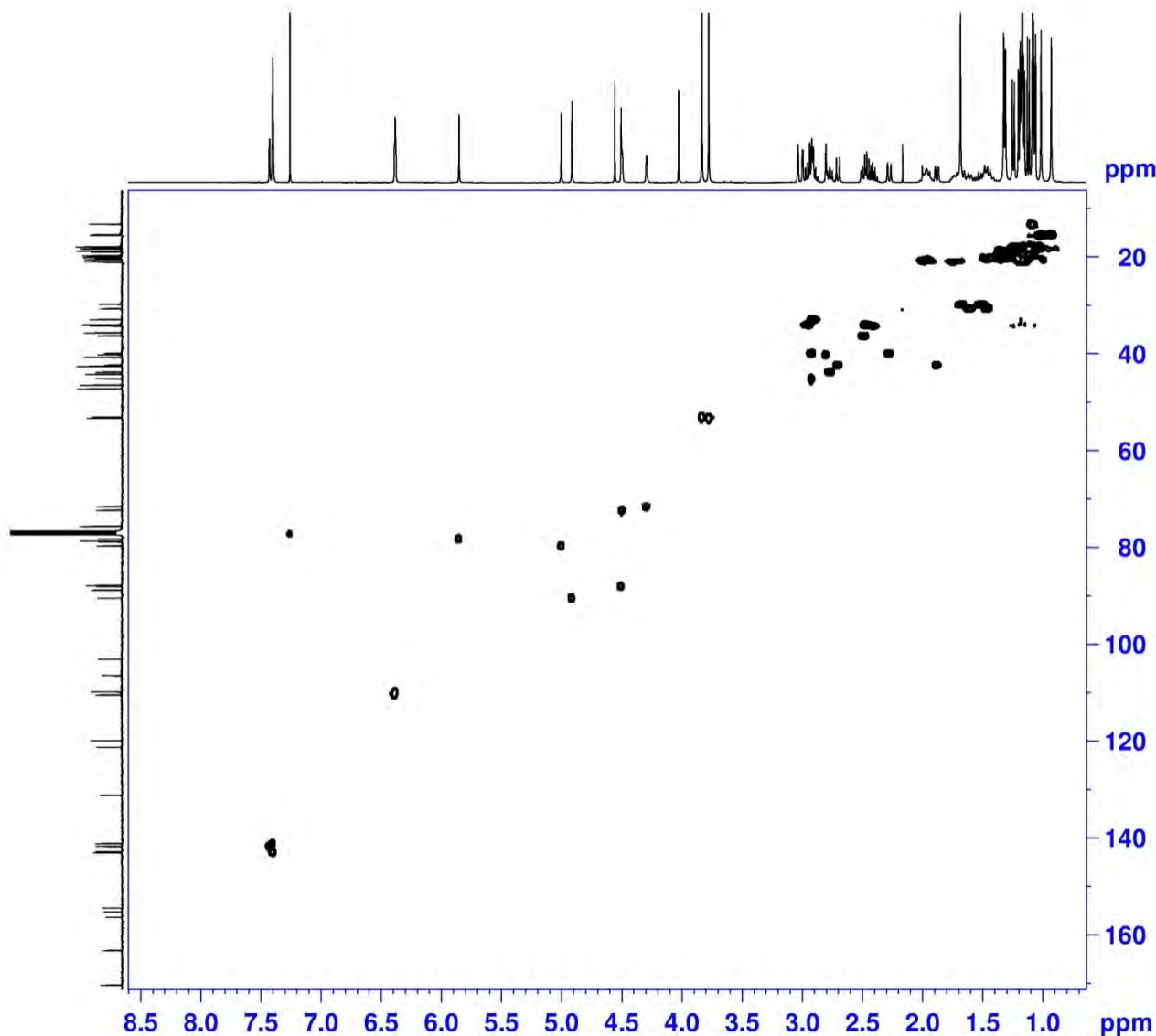
¹H-¹H COSY (400 MHz) spectrum of compound **3** in CDCl₃



```
NAME      lws-42-1-fd
EXPNO      4
PROCNO     1
Date_      20150812
Time       14.45
INSTRUM    spect
PROBHD     5 mm CPMBO BB
PULPROG    cosygpppqf
TD         2048
SOLVENT    CDCl3
NS         8
DS         8
SWH        3906.250 Hz
FIDRES     1.907349 Hz
AQ         0.2621940 sec
RG         147.94
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
FhMODE     QF
SI         1024
SF         400.1300098 MHz
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SSB        0
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GB         0
PC         1.40
SI         1024
MC2        QF
SF         400.1300098 MHz
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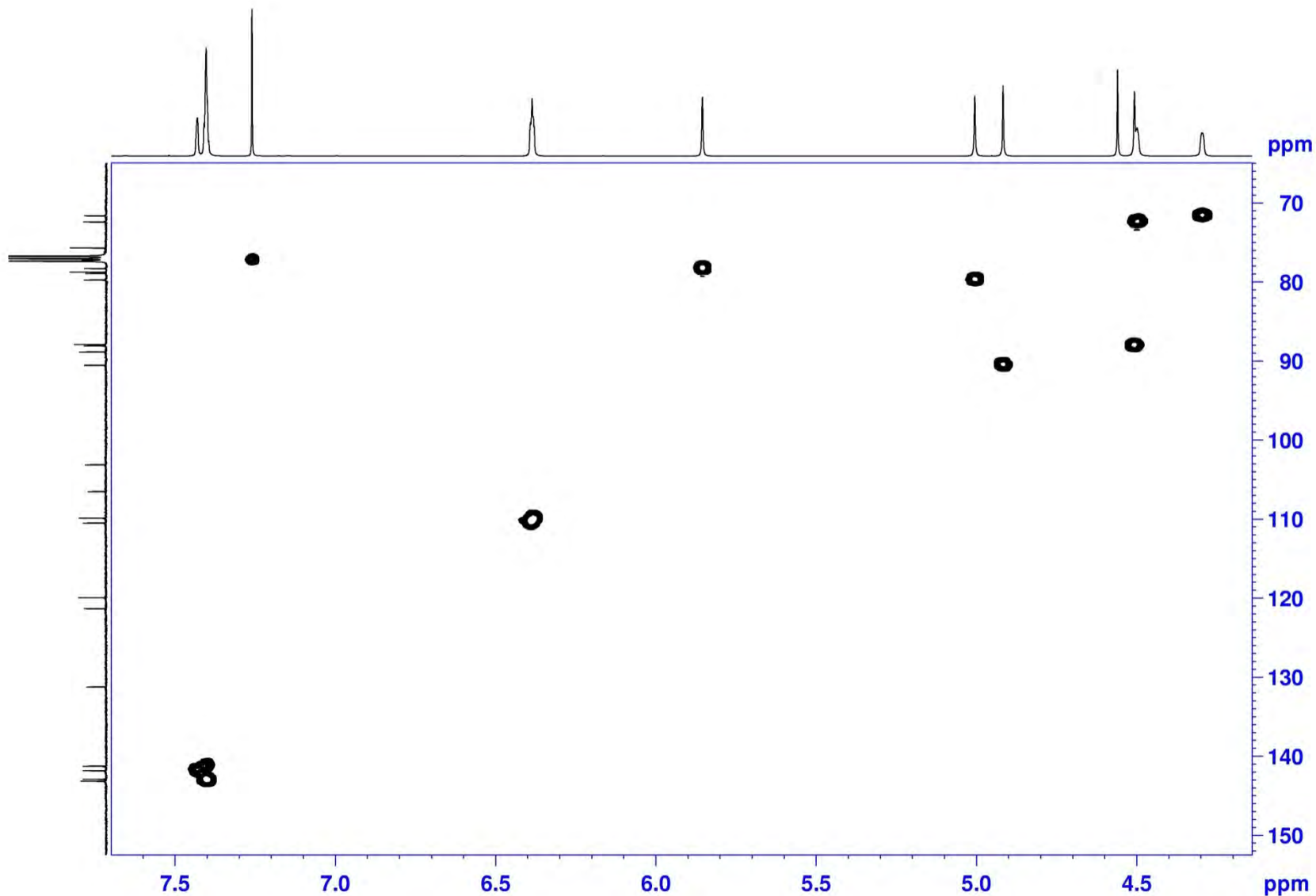
HSQC (400 MHz) spectrum of compound **3** in CDCl₃



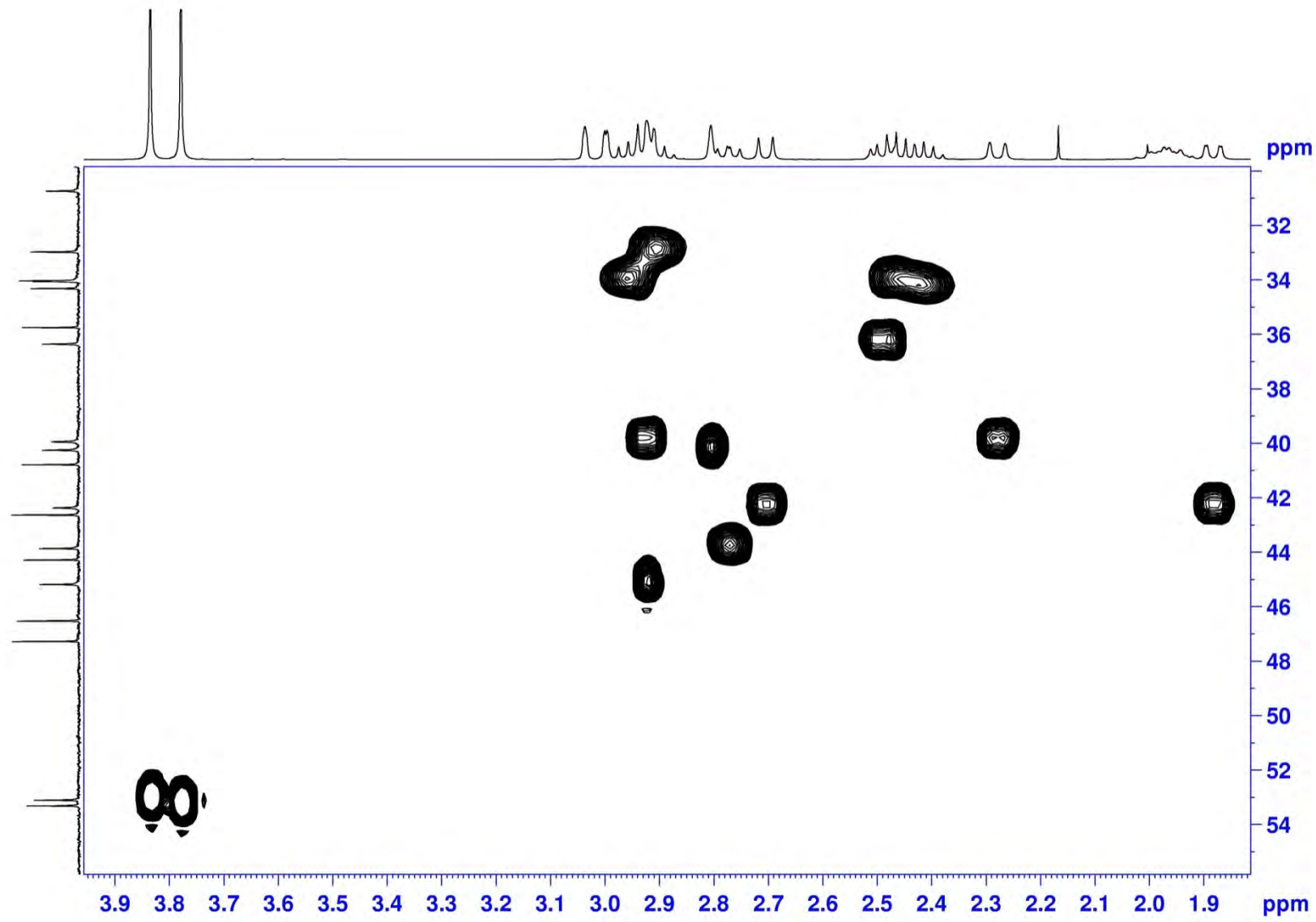
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Time           15.24
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PULPROG        hsqcetgpsi2
TD             1024
SOLVENT        CDCl3
NS             16
DS             16
SWH            4302.926 Hz
FIDRES         4.202076 Hz
AQ             0.1190388 sec
RG             208.5
DW             116.200 usec
DE             10.00 usec
TE             297.0 K
CNST2          145.0000000
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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

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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
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GB            0
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SI            1024
MC2           echo-antiecho
SF            100.6127685 MHz
WDW           QSINE
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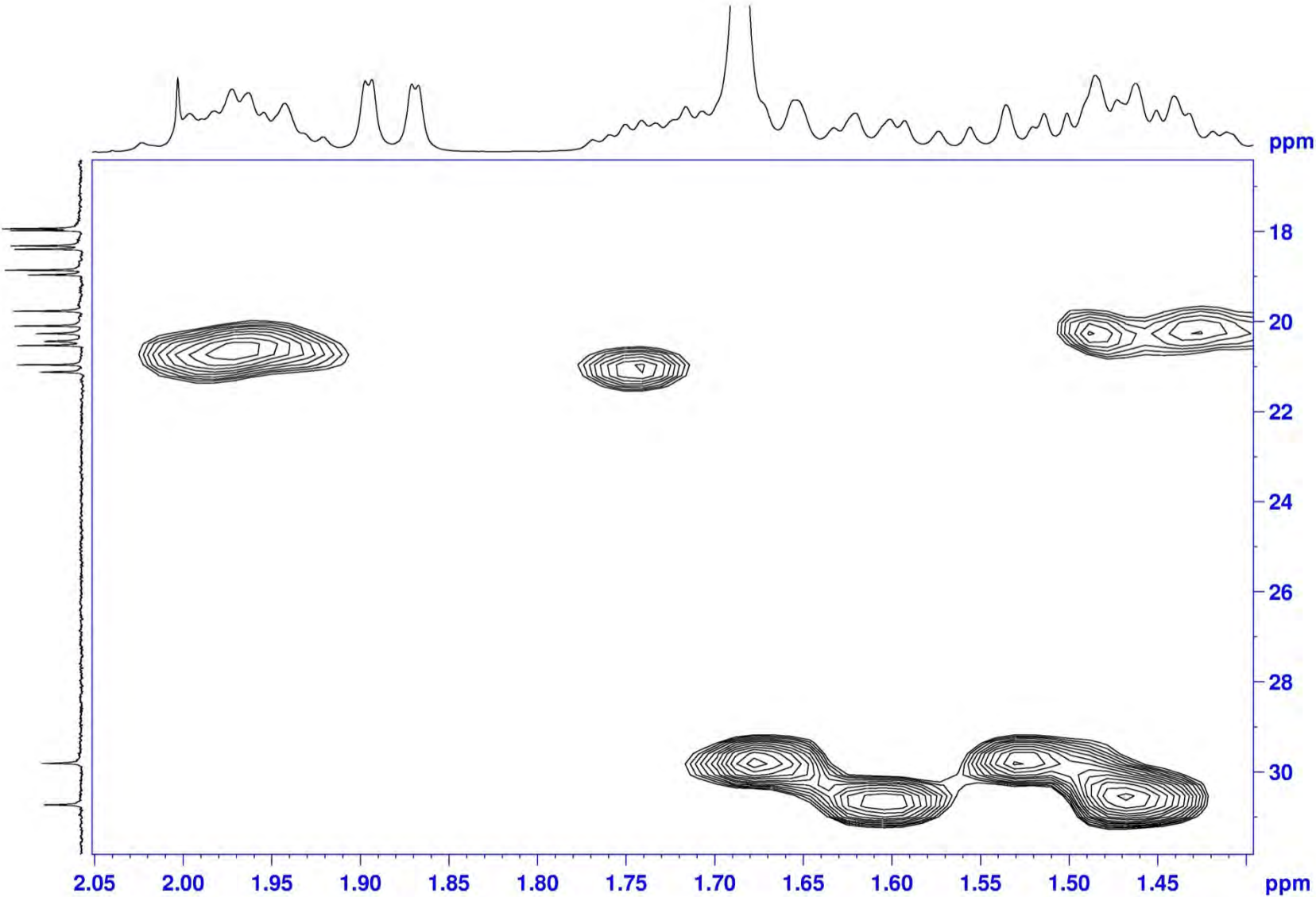
HSQC (400 MHz) spectrum of compound **3** in CDCl₃



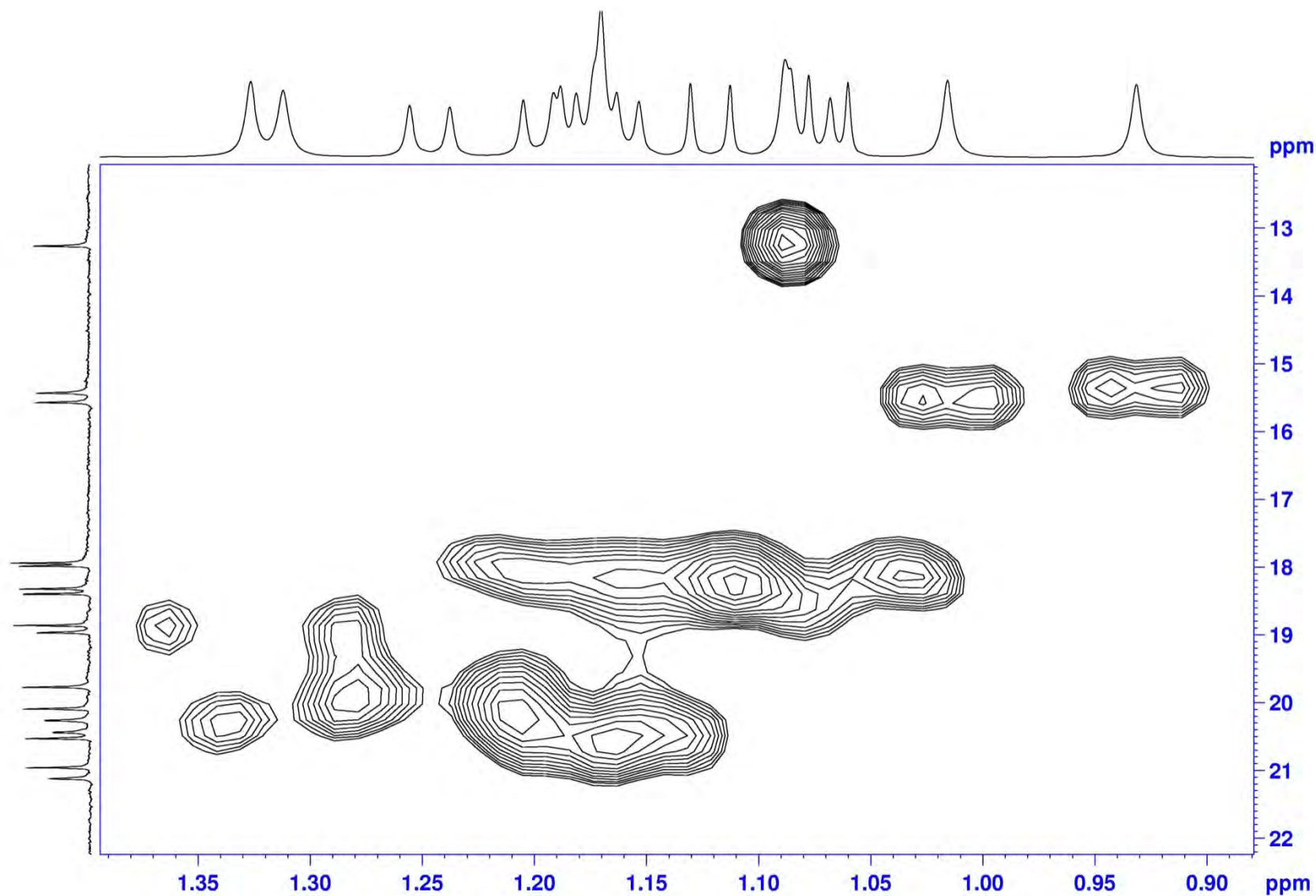
HSQC (400 MHz) spectrum of compound **3** in CDCl₃



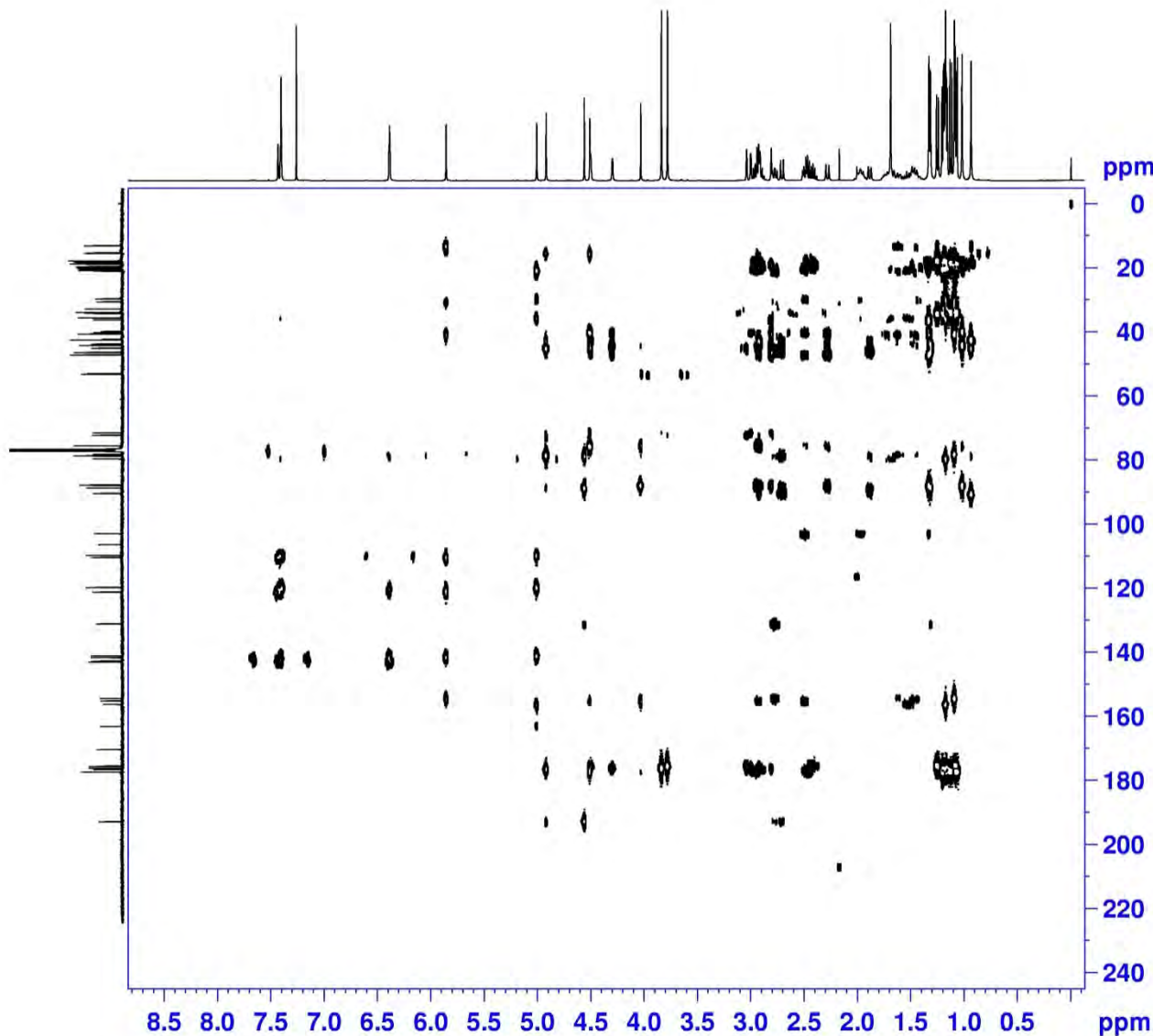
HSQC (400 MHz) spectrum of compound **3** in CDCl₃



HSQC (400 MHz) spectrum of compound **3** in CDCl₃



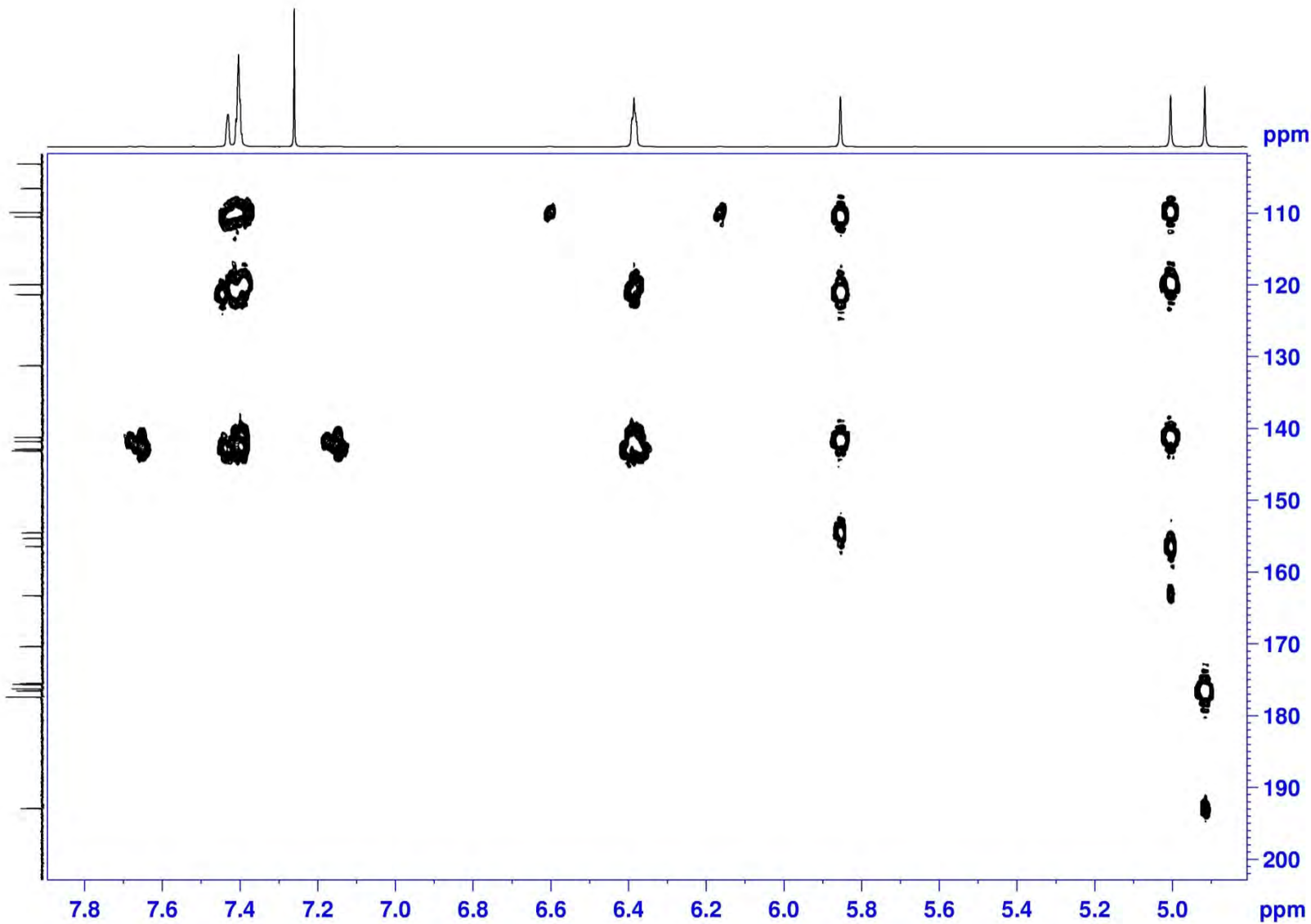
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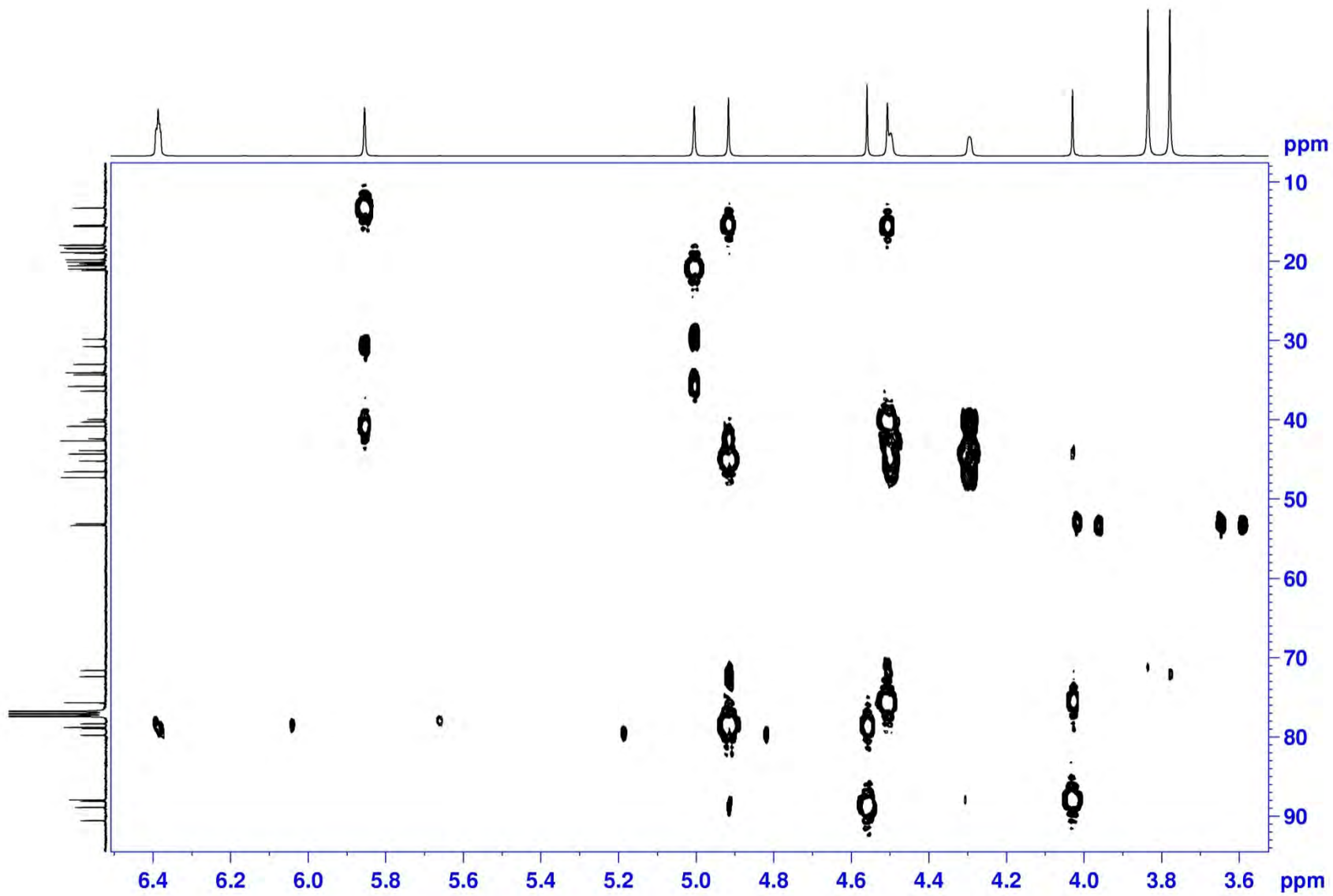
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PROCNO 1
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PULPROG hmbcgp1pndqf
TD 4096
SOLVENT CDCl3
NS 32
DS 16
SWH 5197.505 Hz
FIDRES 1.268922 Hz
AQ 0.3940852 sec
RG 208.5
DW 96.200 usec
DE 10.00 usec
TE 297.0 K
CNST2 145.0000000
CNST13 10.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.05000000 sec
D16 0.00020000 sec
IN0 0.00002080 sec

===== CHANNEL f1 =====
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NUC1 1H
P1 12.00 usec
P2 24.00 usec
ND0 2
TD 128
SFO1 100.6233 MHz
FIDRES 187.800476 Hz
SW 238.896 ppm
FnMODE QF
SI 2048
SF 400.1300088 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
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MC2 QF
SF 100.6127685 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

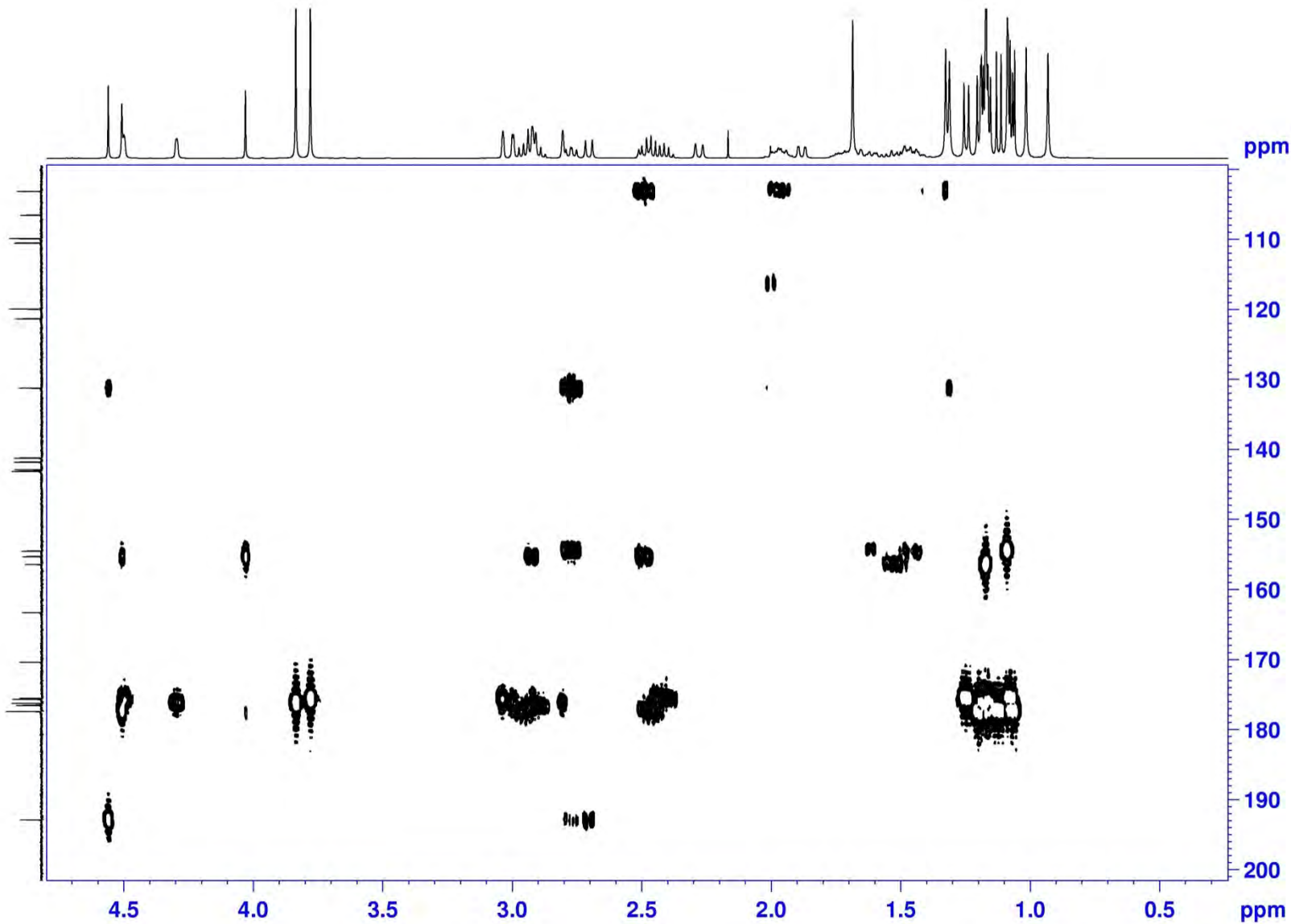
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



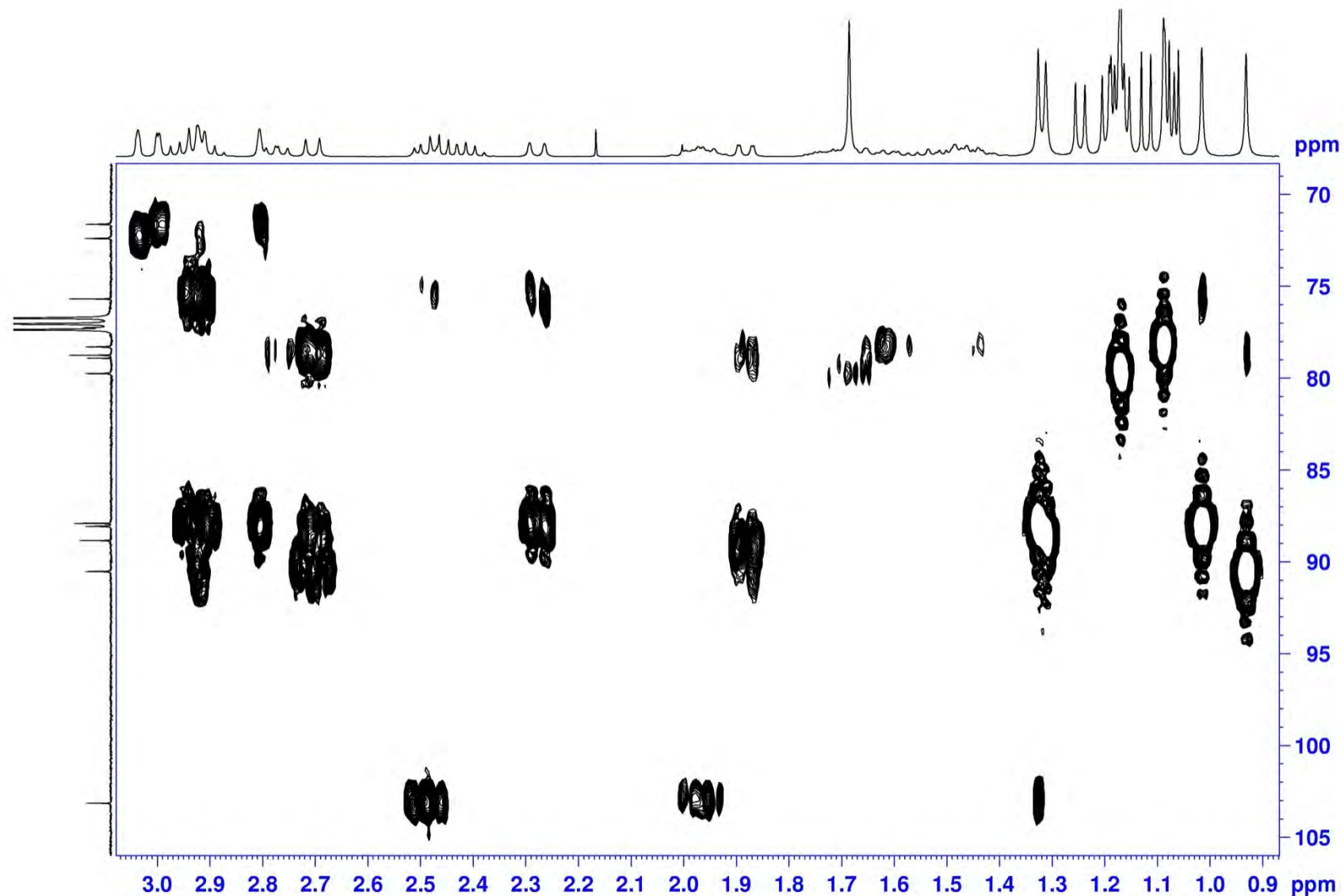
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



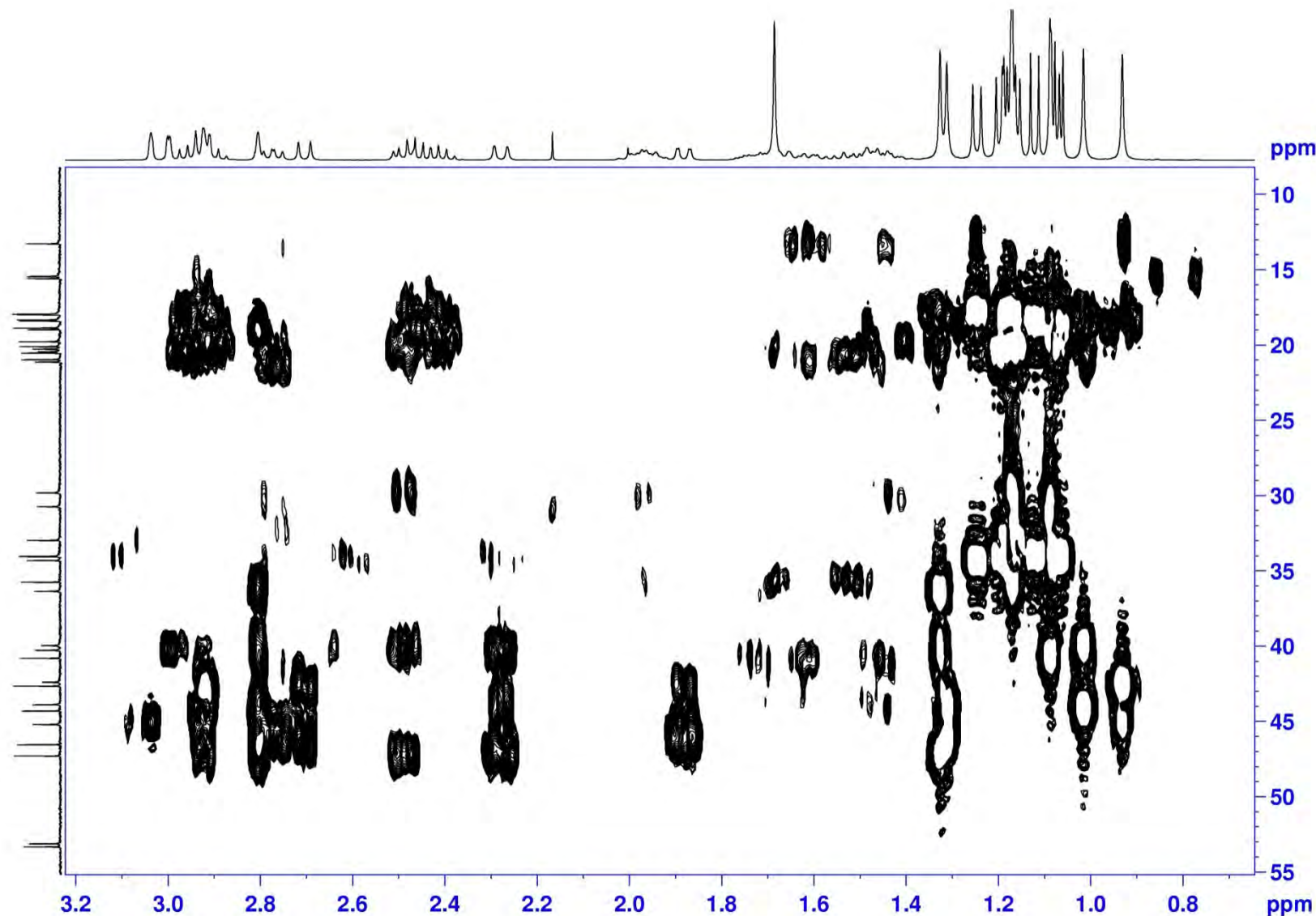
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



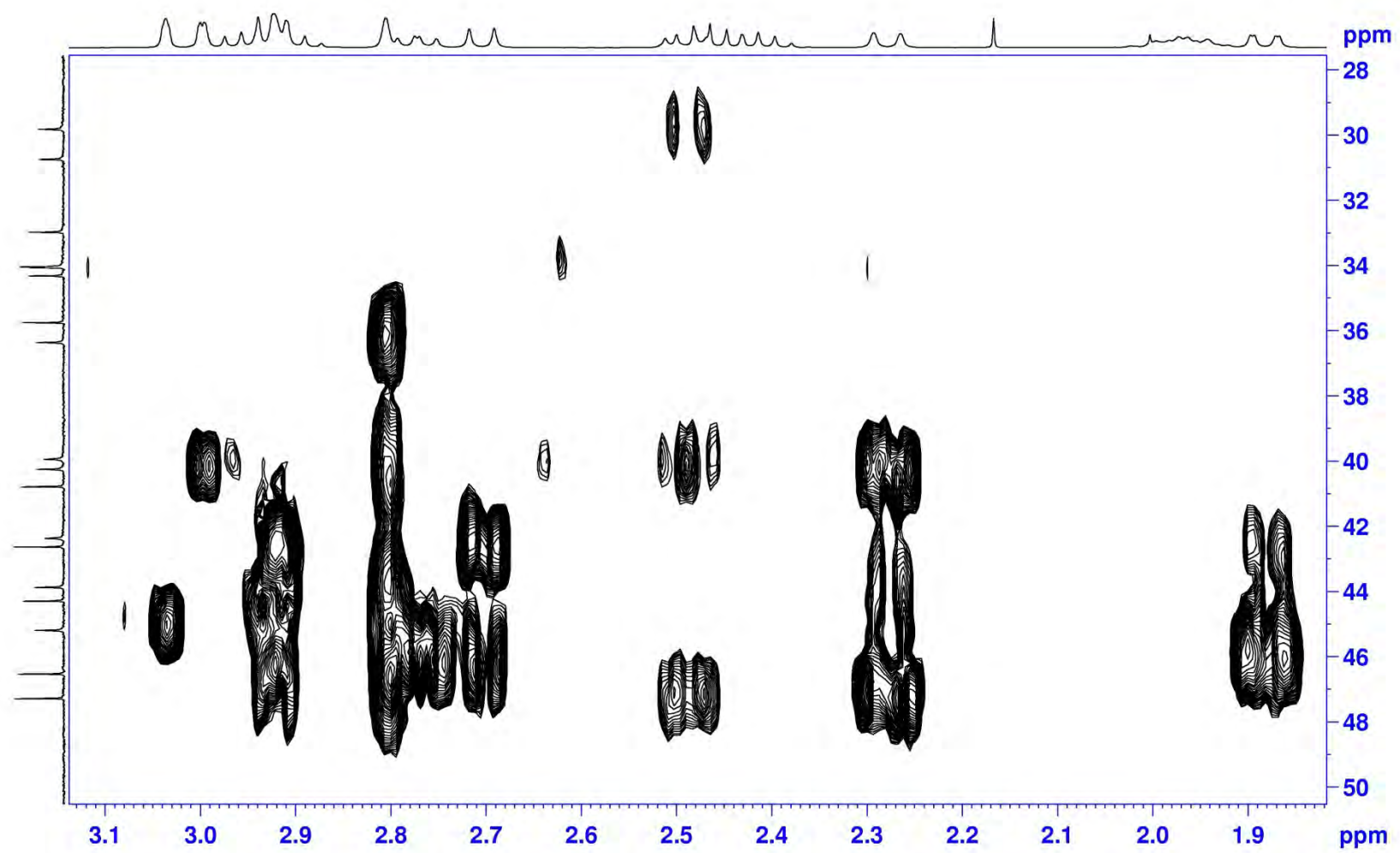
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



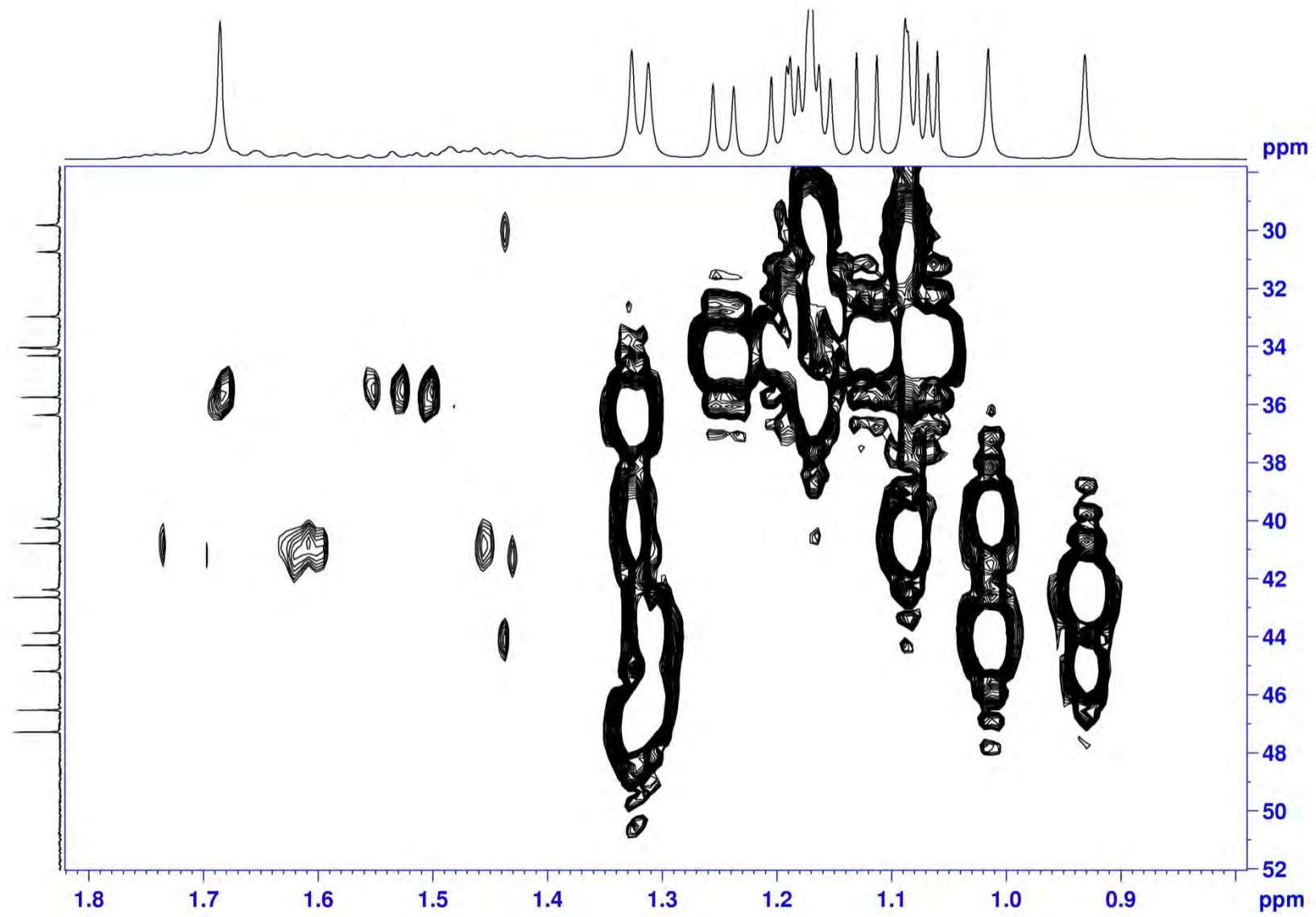
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



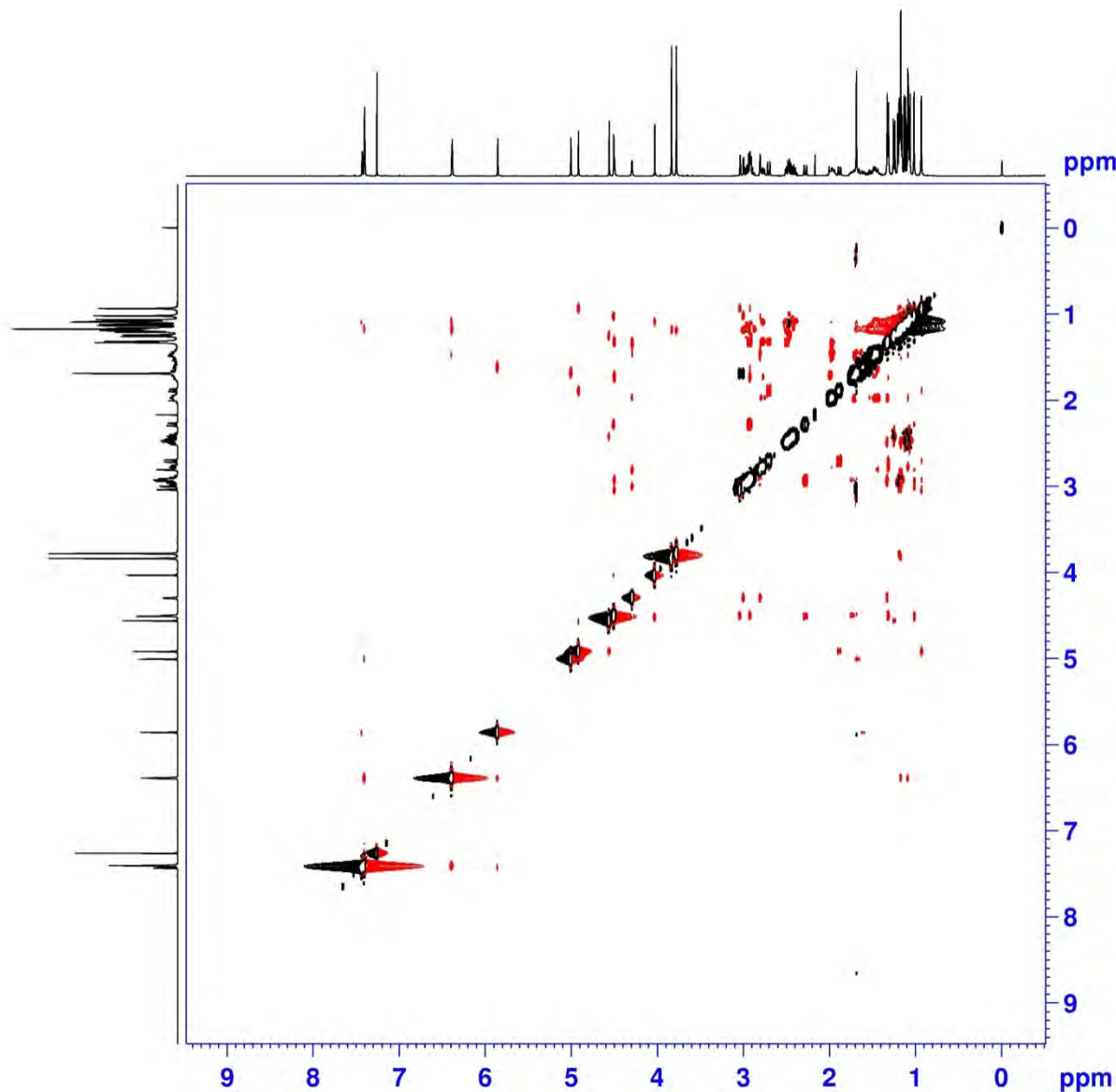
HMBC (400 MHz) spectrum of compound **3** in CDCl₃



HMBC (400 MHz) spectrum of compound **3** in CDCl₃



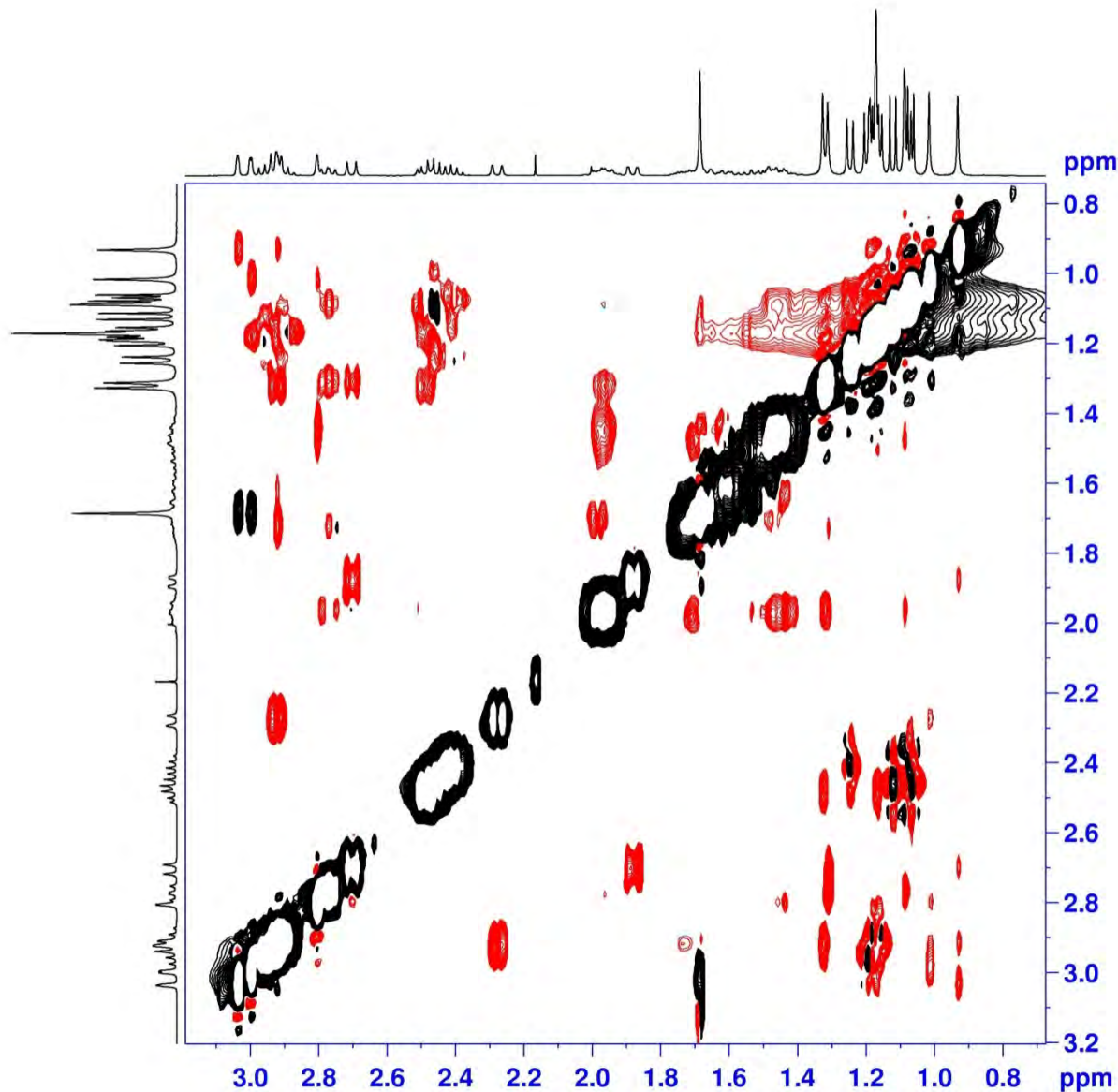
NOESY (400 MHz) spectrum of compound **3** in CDCl₃



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EXPNO 7
PROCNO 1
Date_ 20150812
Time 19.31
INSTRUM spect
PROBHD 5 mm CPBBO BB
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 16
DS 32
SWH 4000.000 Hz
FIDRES 1.953125 Hz
AQ 0.2560500 sec
RG 117.37
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 400.1300094 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPPI
SF 400.1300094 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

NOESY (400 MHz) spectrum of compound **3** in CDCl₃



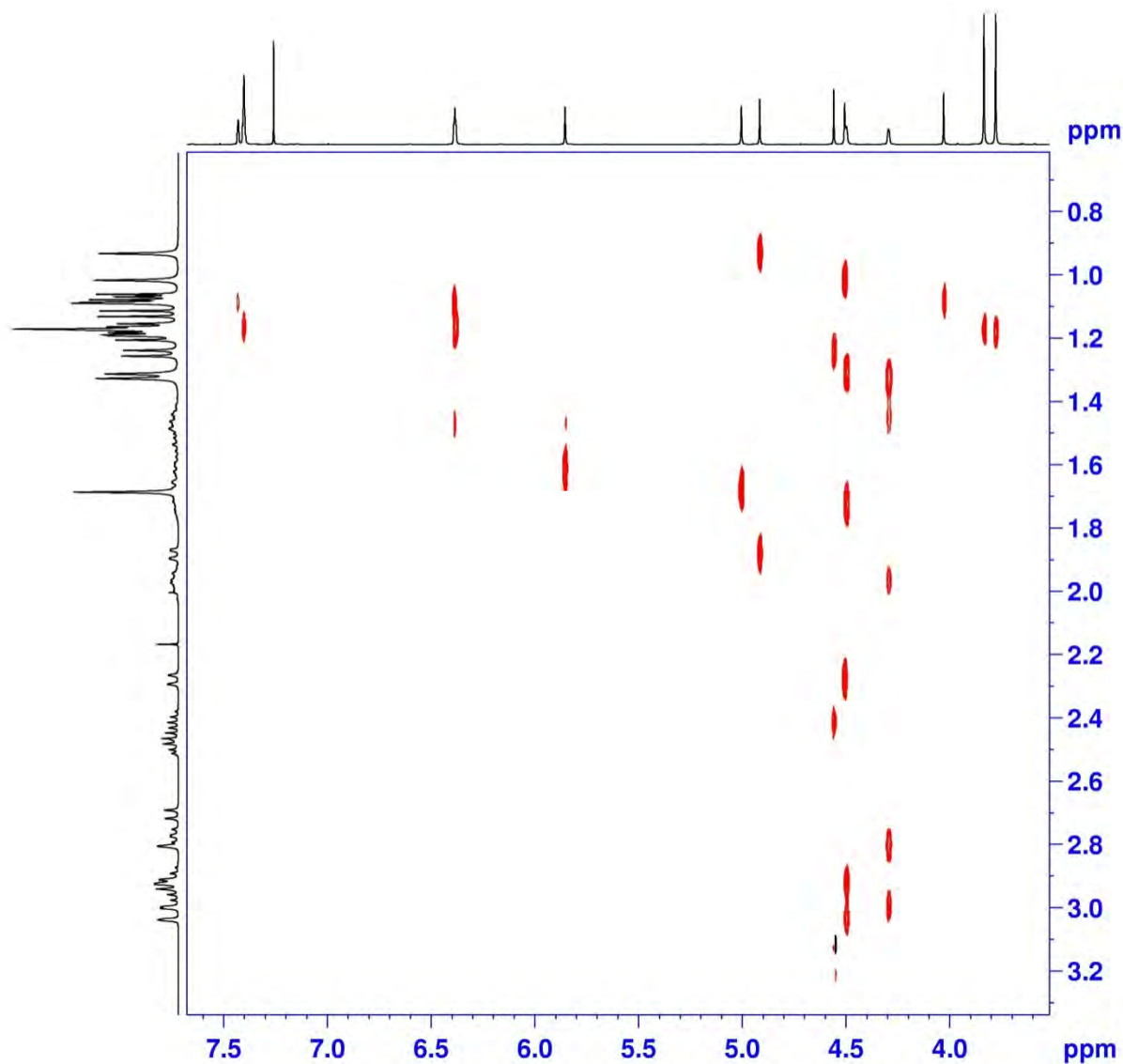
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EXPNO          7
PROCNO         1
Date_         20150812
Time          19.31
INSTRUM       spect
PROBHD        5 mm CXPBBO BB
PULPROG       noesygpphph
TD            2048
SOLVENT       CDCl3
NS            16
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            117.37
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            400.1300094 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF            400.1300094 MHz
WDW            QSINE
SSB            2
    
```

NOESY (400 MHz) spectrum of compound **3** in CDCl₃



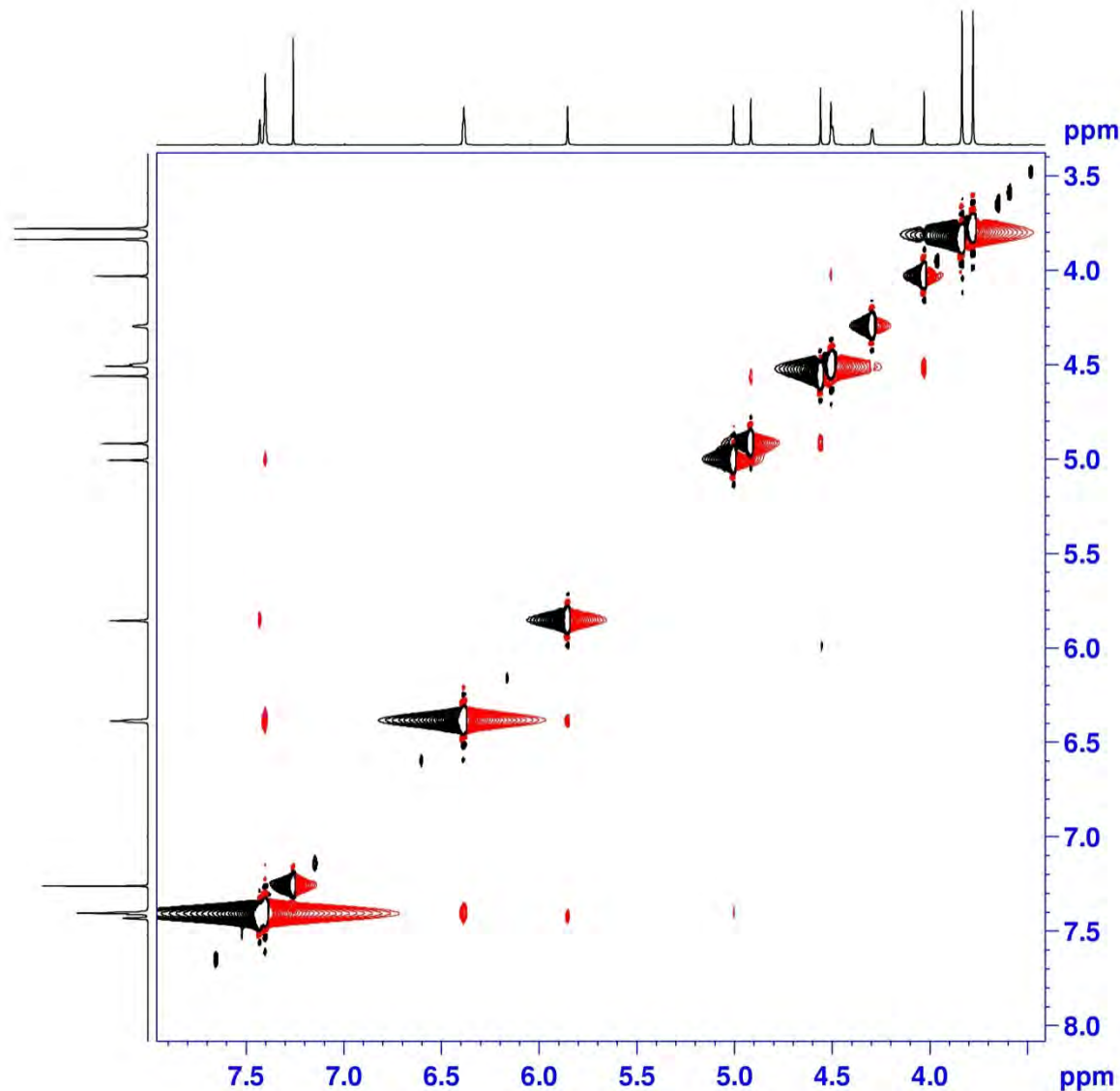
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NAME          lws-42-1-fd
EXPNO          7
PROCNO         1
Date_          20150812
Time           19.31
INSTRUM        spect
PROBHD         5 mm CFPBBO BB
PULPROG        noesygpphph
TD             2048
SOLVENT        CDCl3
NS              16
DS              32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ             0.2560500 sec
RG             117.37
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            400.1300094 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF            400.1300094 MHz
WDW            QSINE
    
```

NOESY (400 MHz) spectrum of compound 3 in CDCl₃



NAME lws-42-1-fd
EXPNO 7
PROCNO 1
Date_ 20150812
Time 19.31
INSTRUM spect
PROBHD 5 mm CFPBBO BB
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 16
DS 32
SWH 4000.000 Hz
FIDRES 1.953125 Hz
AQ 0.2560500 sec
RG 117.37
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 400.1300094 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPPI
SF 400.1300094 MHz
WDW QSINE
--

HR-ESIMS for compound 3'

Generic Display Report

Analysis Info

Acquisition Date 7/15/2016 5:07:57 PM

Analysis Name

D:\Data\MS\data\201607\liwanshan_37-1_pos_22_01_2191.d

Method

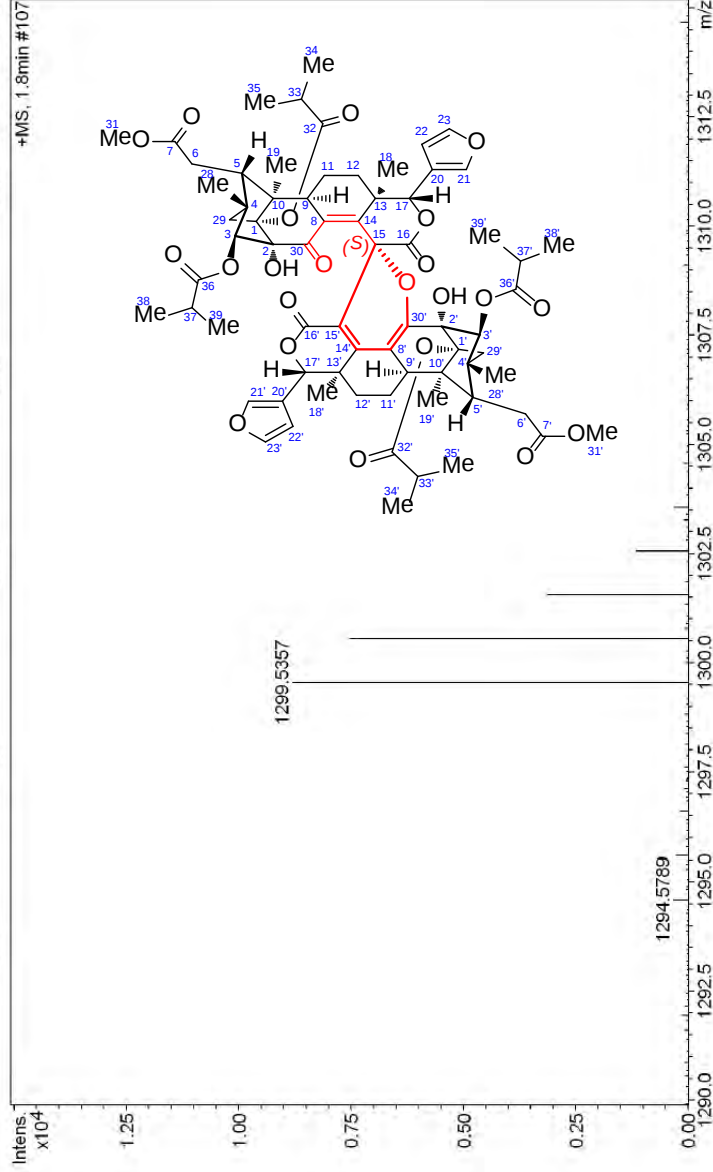
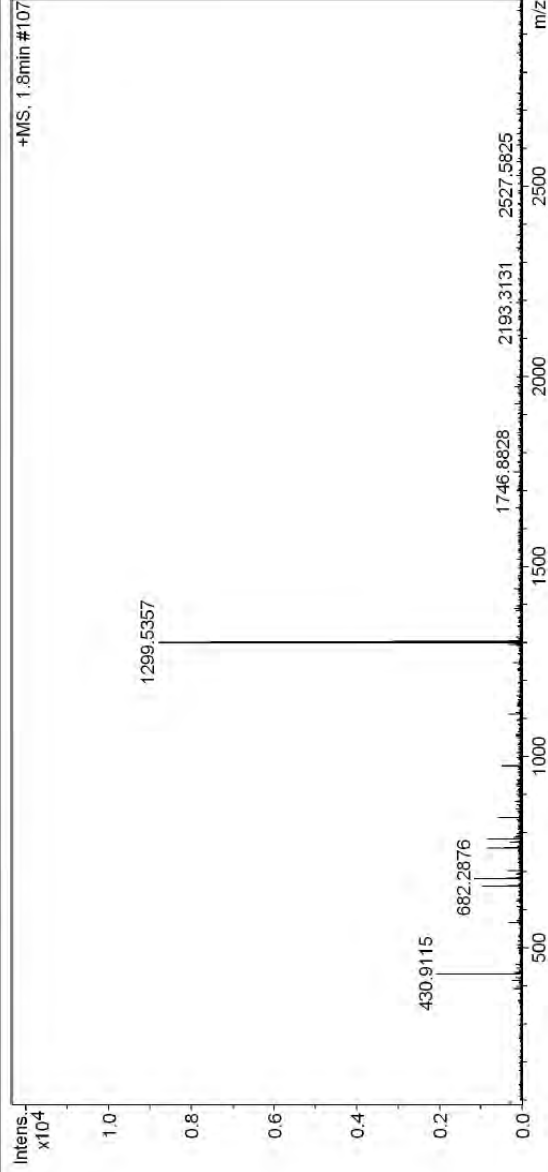
LC_Direct Infusion_pos_100-3000mz.m

Sample Name

liwanshan_37-1_pos

Comment

Operator SCSIO
Instrument maxis



HR-ESIMS for compound 3'

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\MS\data\201607\liwanshan_37-1_pos_22_01_2191.d
Method LC_Direct Infusion_pos_100-3000mz.m
Sample Name liwanshan_37-1_pos
Comment

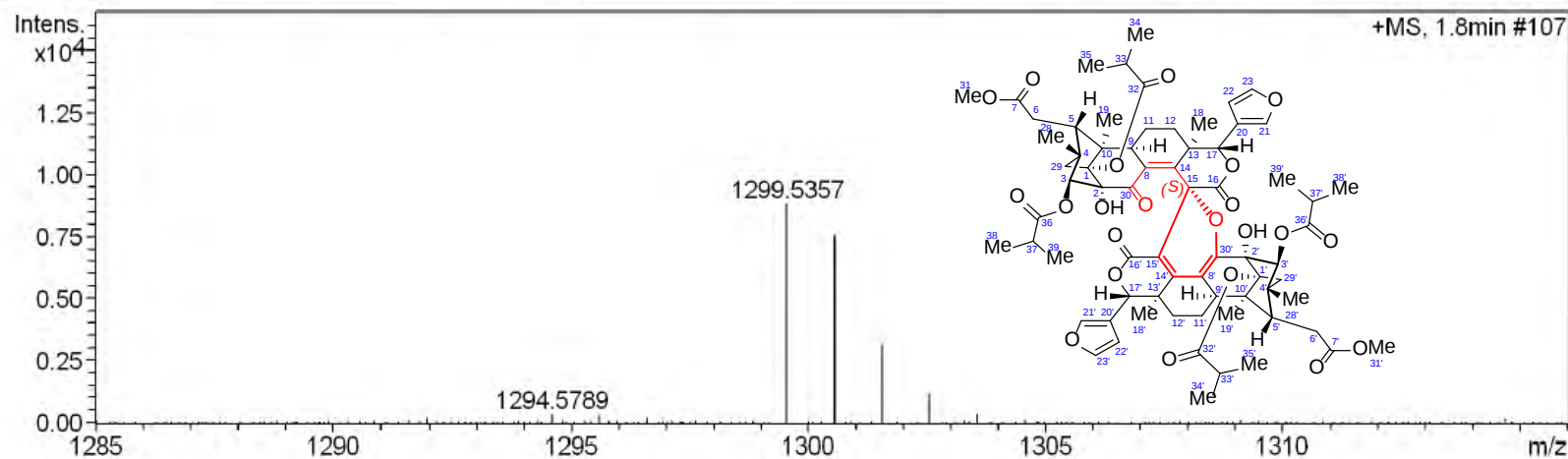
Acquisition Date 7/15/2016 5:07:57 PM

Operator SCSIO

Instrument / Ser# maXis 29

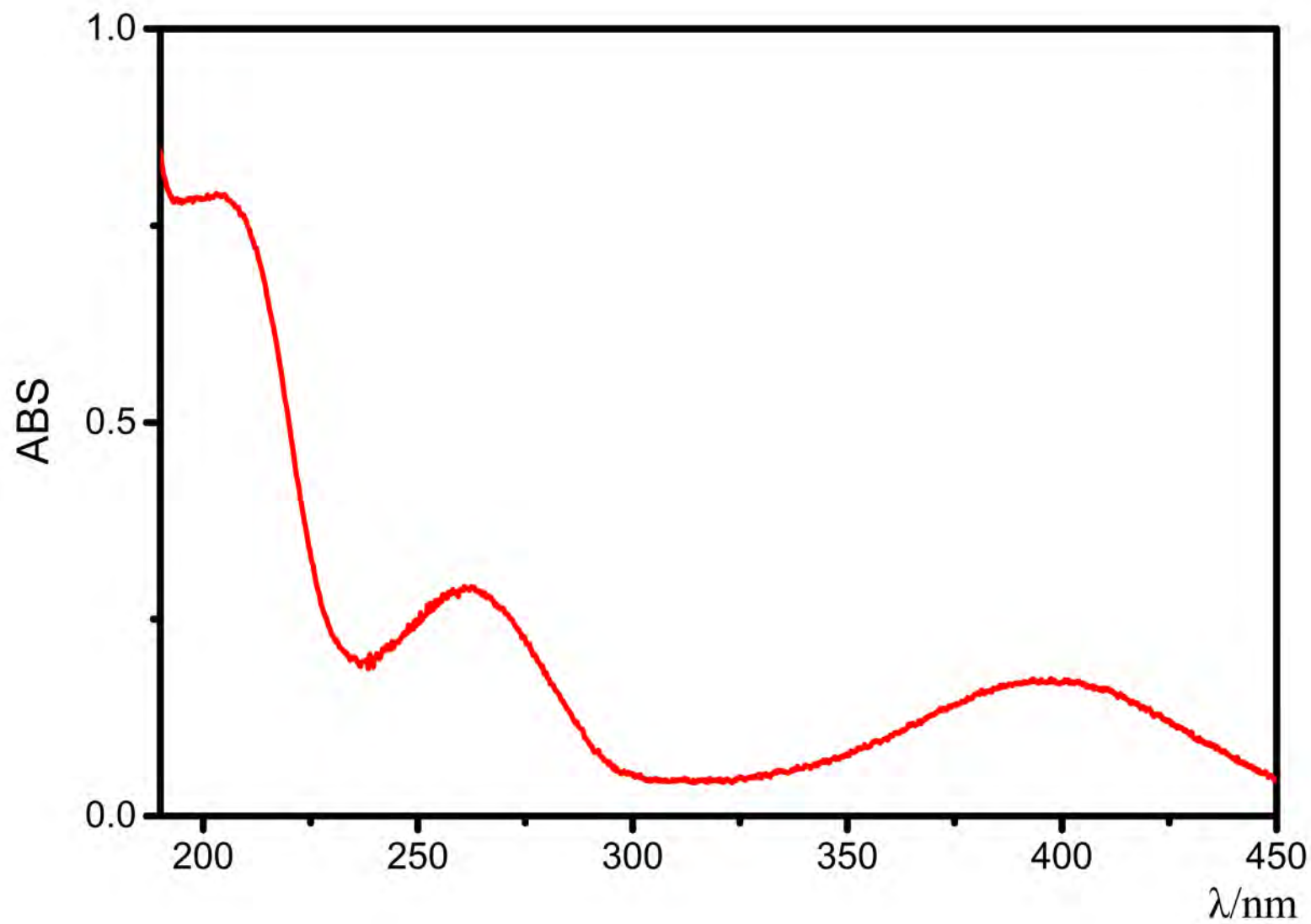
Acquisition Parameter

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	3000 m/z	Set Collision Cell RF	1800.0 Vpp	Set Divert Valve	Waste



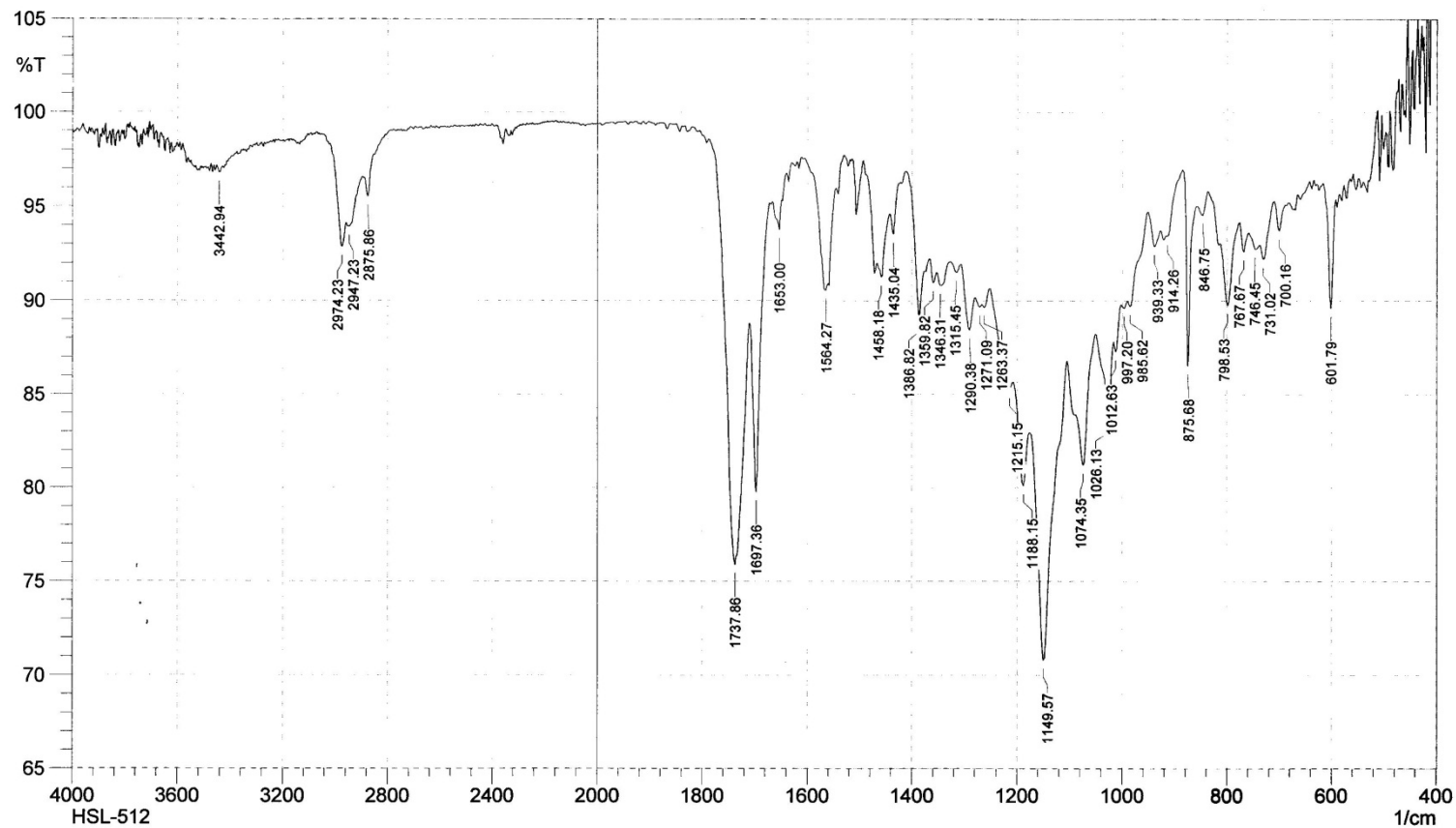
Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e ⁻ Conf	N-Rule
1299.5357	1	C 70 H 84 Na O 22	100.00	1299.5346	-0.8	-1.1	38.4	28.5	even	ok

UV spectrum for compound **3'** in CH₃CN



IR spectrum for compound 3'

SHIMADZU

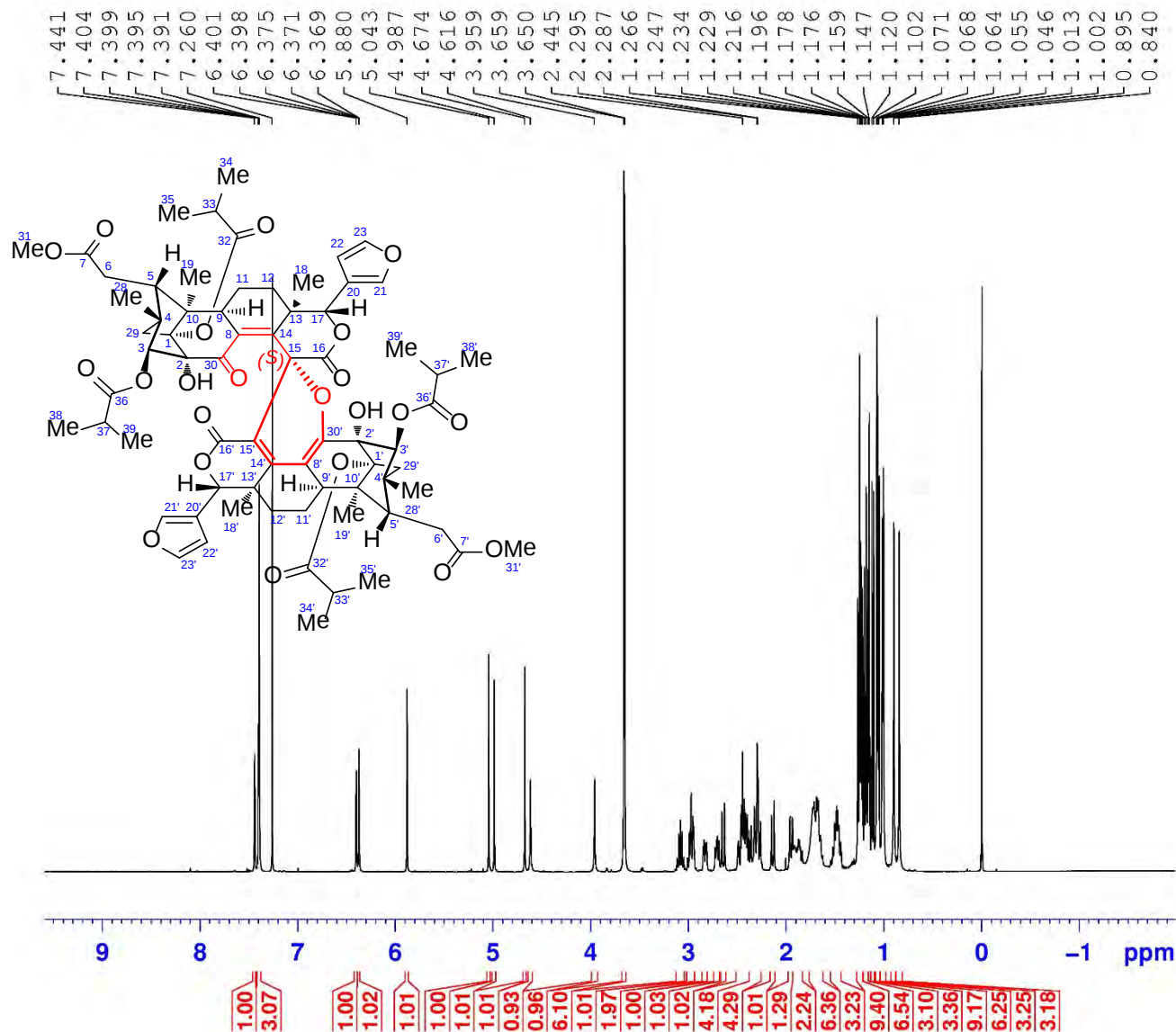


Sample name: 37-1(7)2 (3')
F:\liwanshan\20180226\37-
1(7)2.smf

Scan number: 25
Resolution: 4 [1/cm]

Data: 2018-2-26 16:41:39
Operator: SCS10

^1H NMR (400 MHz) spectrum of compound 3' in CDCl_3



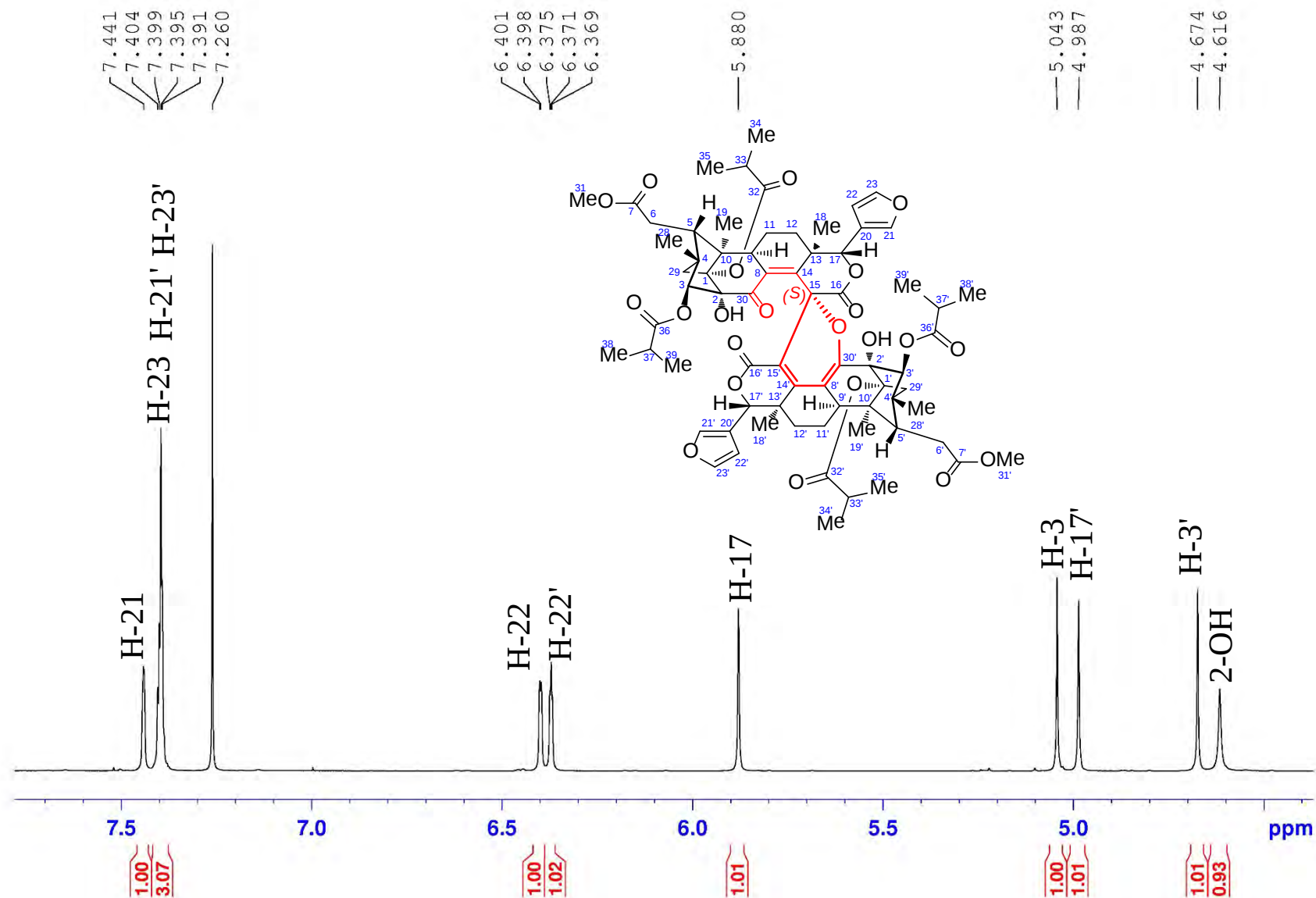
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NAME      lws-37-1 (ddq)
EXPNO     1
PROCNO    1
Date_     20160523
Time      20.04
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685
FIDRES     0.125483
AQ         3.9846387
RG         117.37
DW         60.800
DE         10.00
TE         297.0
D1         1.00000000
TD0        1
  
```

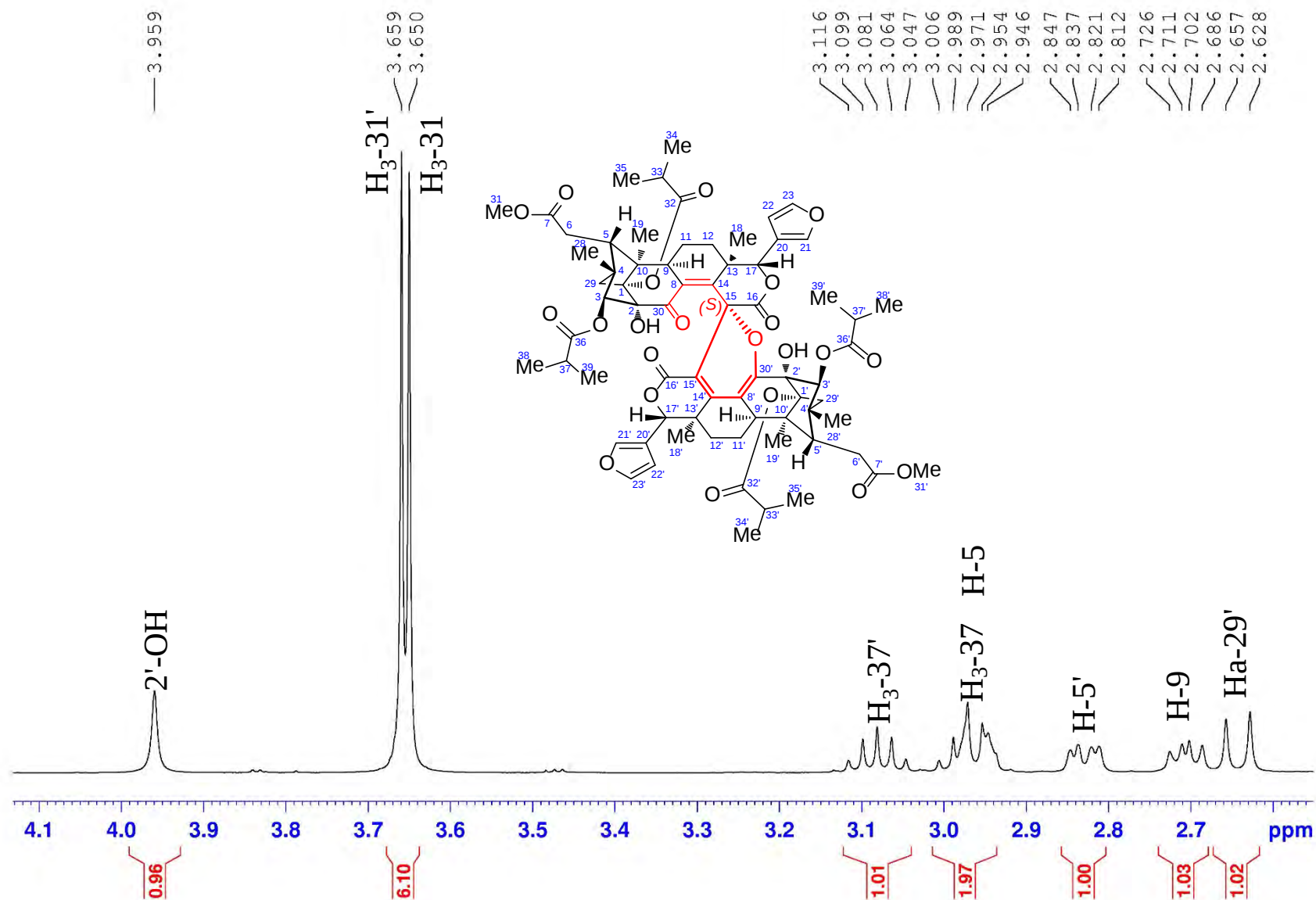
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===== CHANNEL f1 =====
SFO1      400.1324710
NUC1      1H
P1        11.50
SI        65536
SF        400.1300100
WDW       EM
SSB       0
LB        0.30
GB        0
PC        1.00
  
```

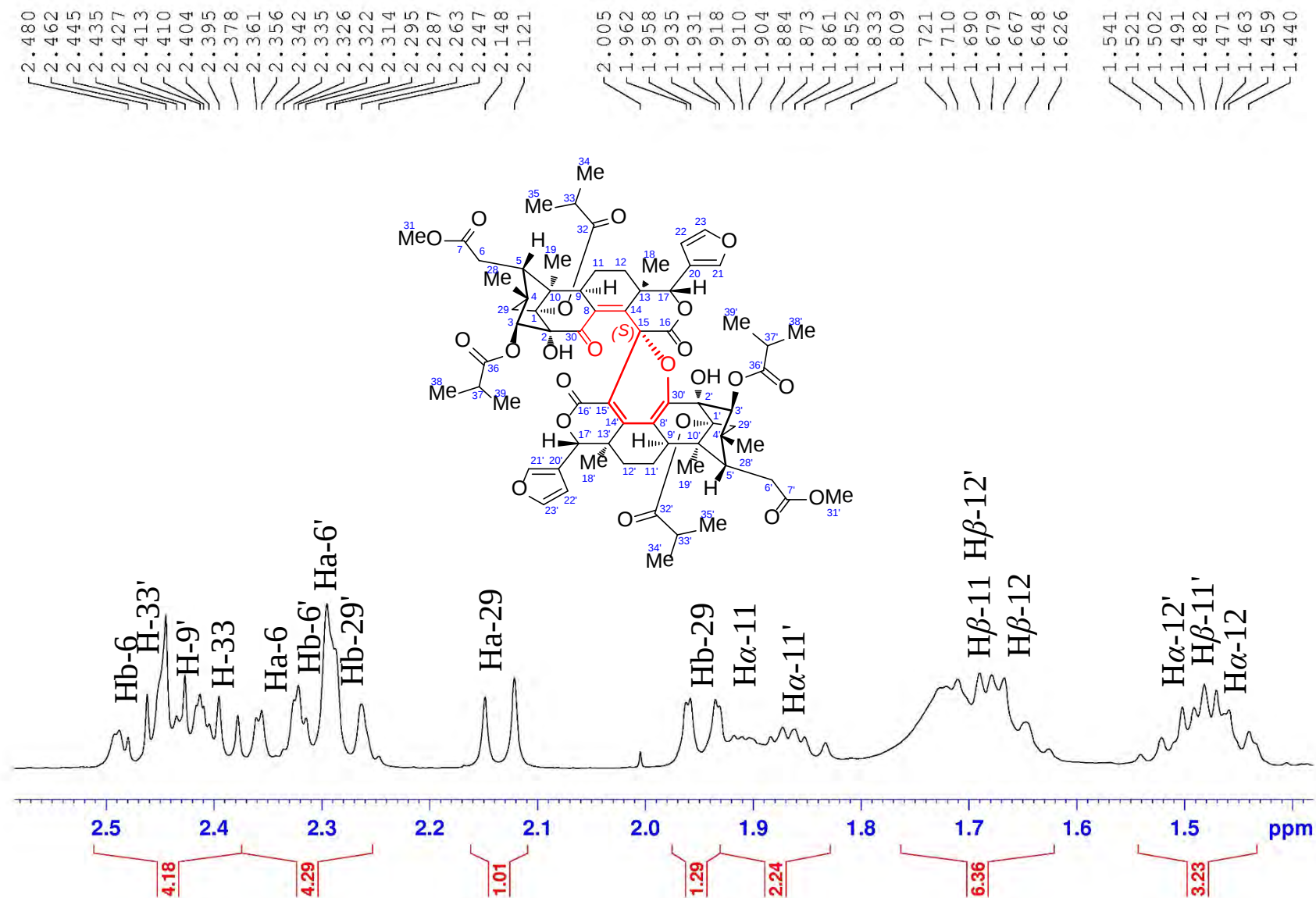
^1H NMR (400 MHz) spectrum of compound **3'** in CDCl_3



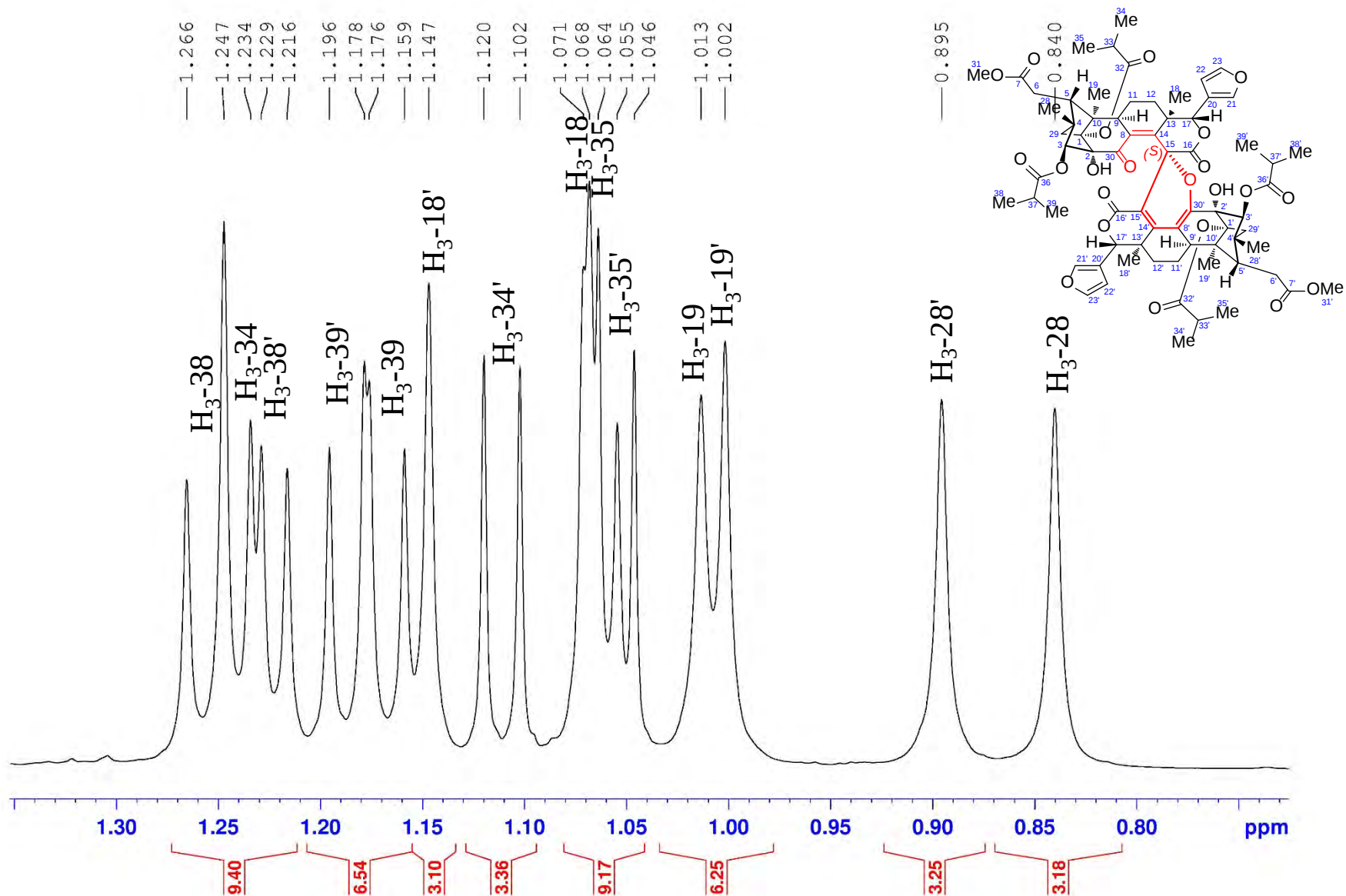
^1H NMR (400 MHz) spectrum of compound **3'** in CDCl_3



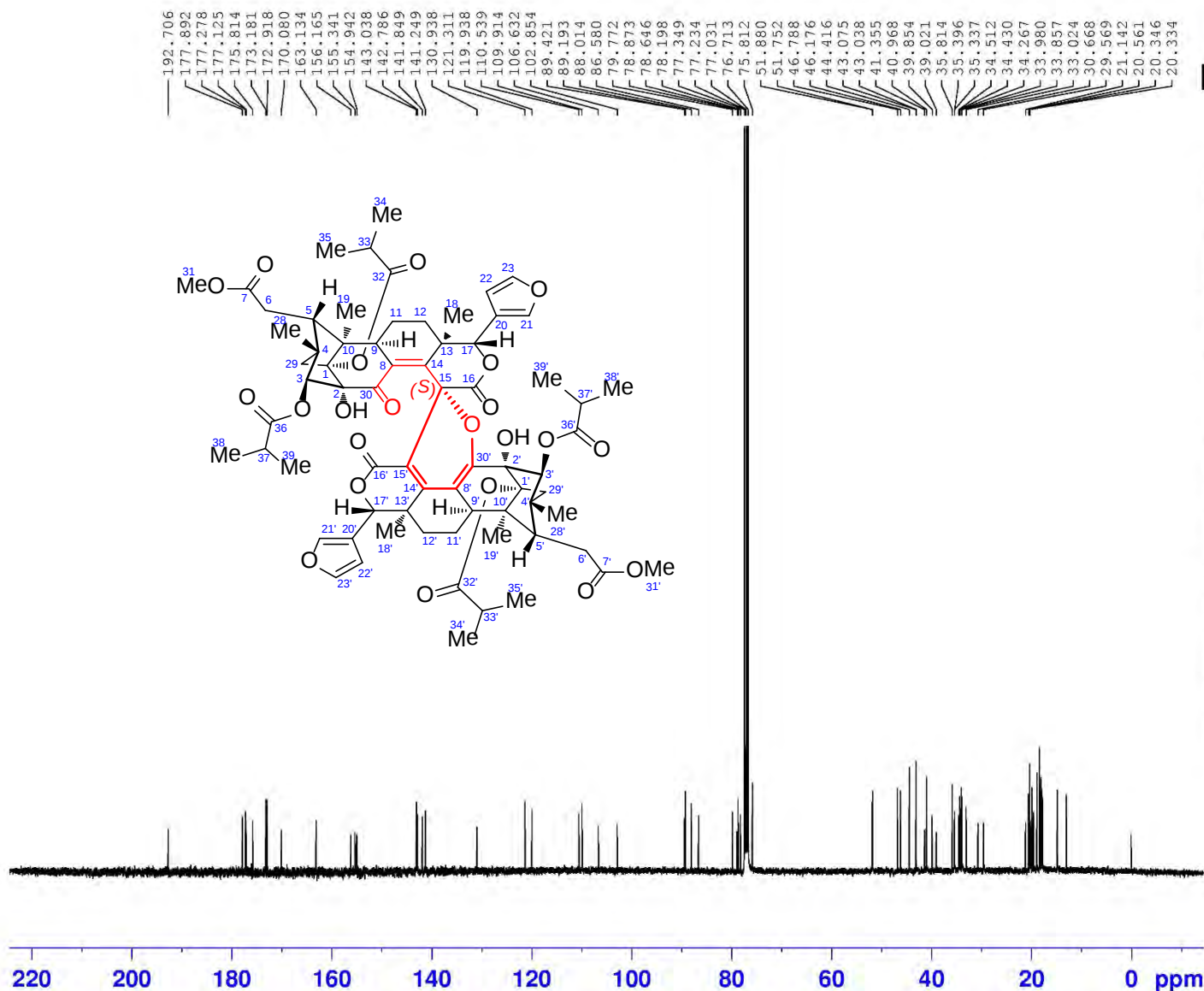
^1H NMR (400 MHz) spectrum of compound **3'** in CDCl_3



^1H NMR (400 MHz) spectrum of compound **3'** in CDCl_3



^{13}C NMR (100 MHz) spectrum of compound **3'** in CDCl_3



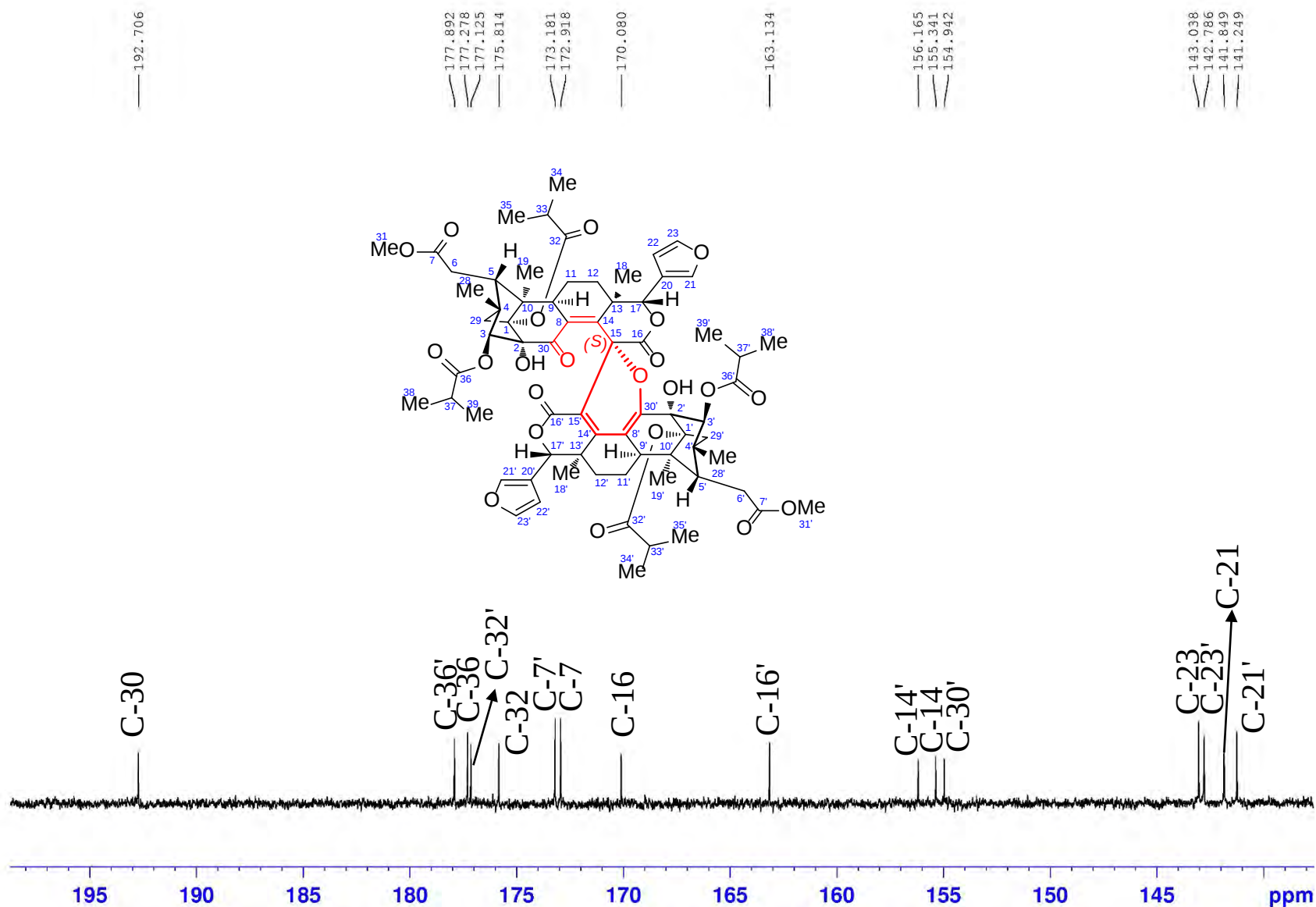
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NAME      lws-37-1(ddq)
EXPNO     2
PROCNO    1
Date_     20160523
Time      21.05
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        24038.461 F
FIDRES     0.366798 F
AQ         1.3631988 s
RG         91.64
DW         20.800 s
DE         18.00 s
TE         297.0 F
D1         2.00000000 s
D11        0.03000000 s
TD0        1
  
```

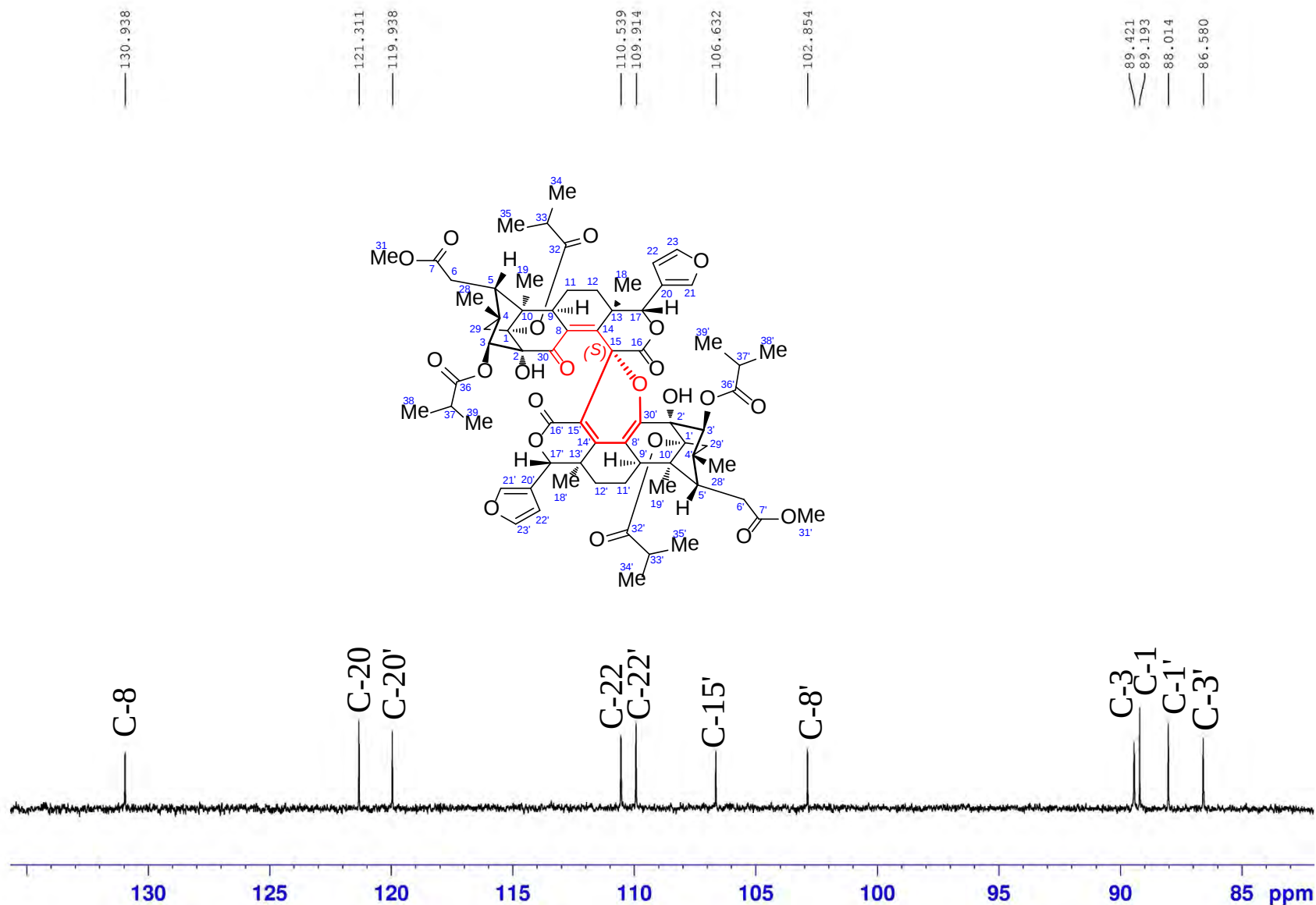
```

===== CHANNEL f1 =====
SF01      100.6233324 F
NUC1       13C
P1         10.00 s
SI         32768
SF         100.6127684 F
WDW        EM
SSB        0
LB         1.00 F
GB         0
PC         1.40
  
```

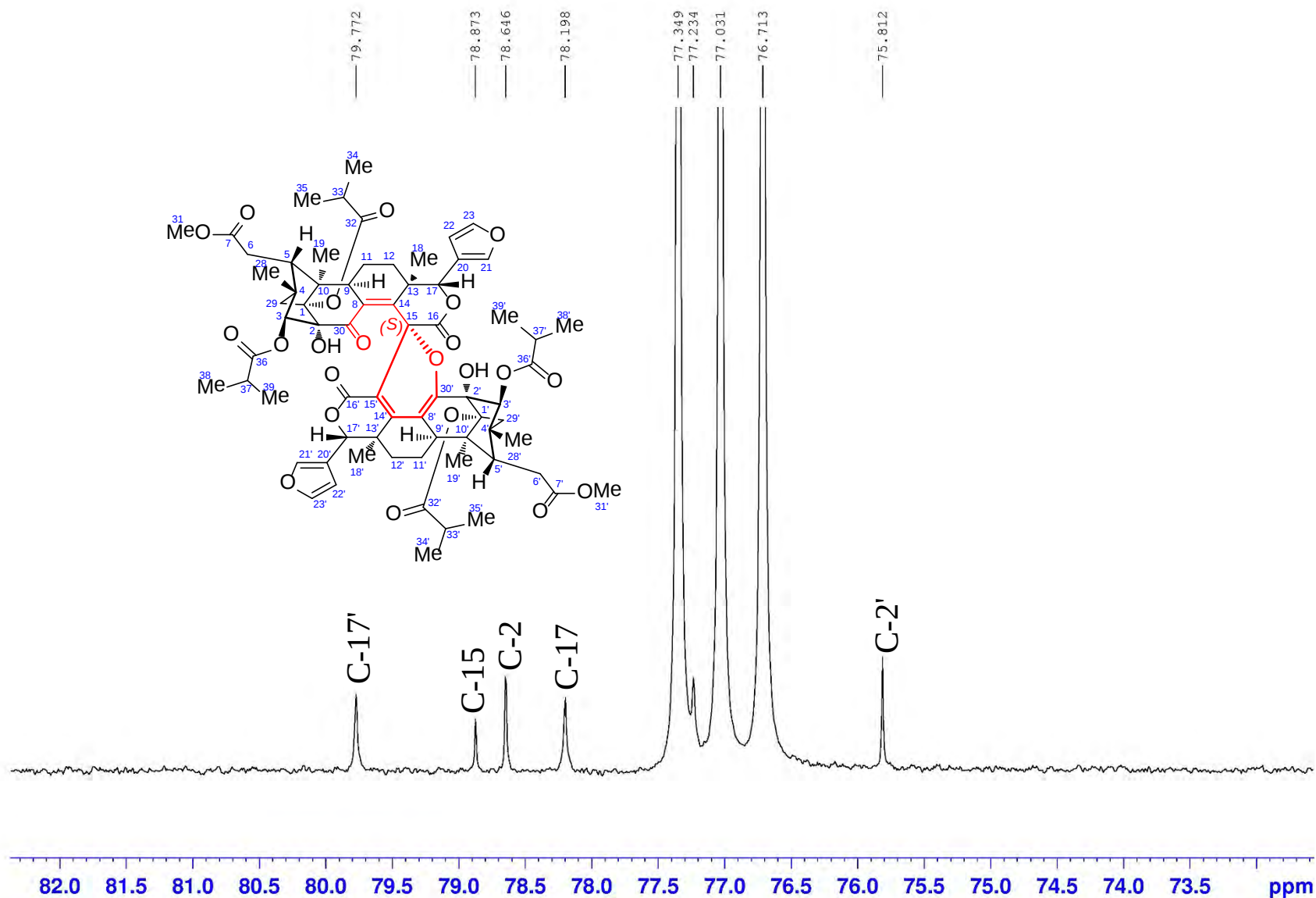
^{13}C NMR (100 MHz) spectrum of compound **3'** in CDCl_3



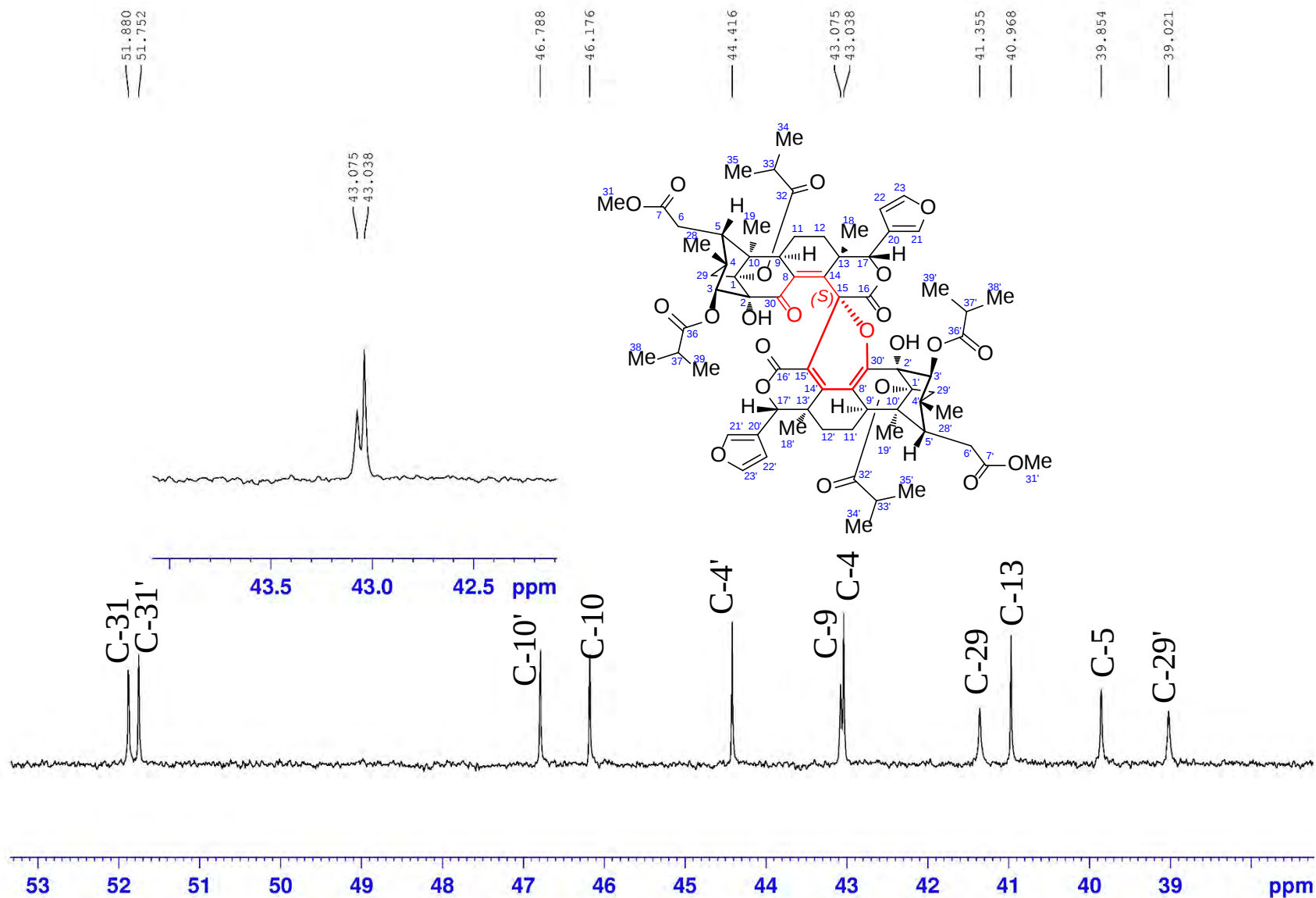
^{13}C NMR (100 MHz) spectrum of compound **3'** in CDCl_3



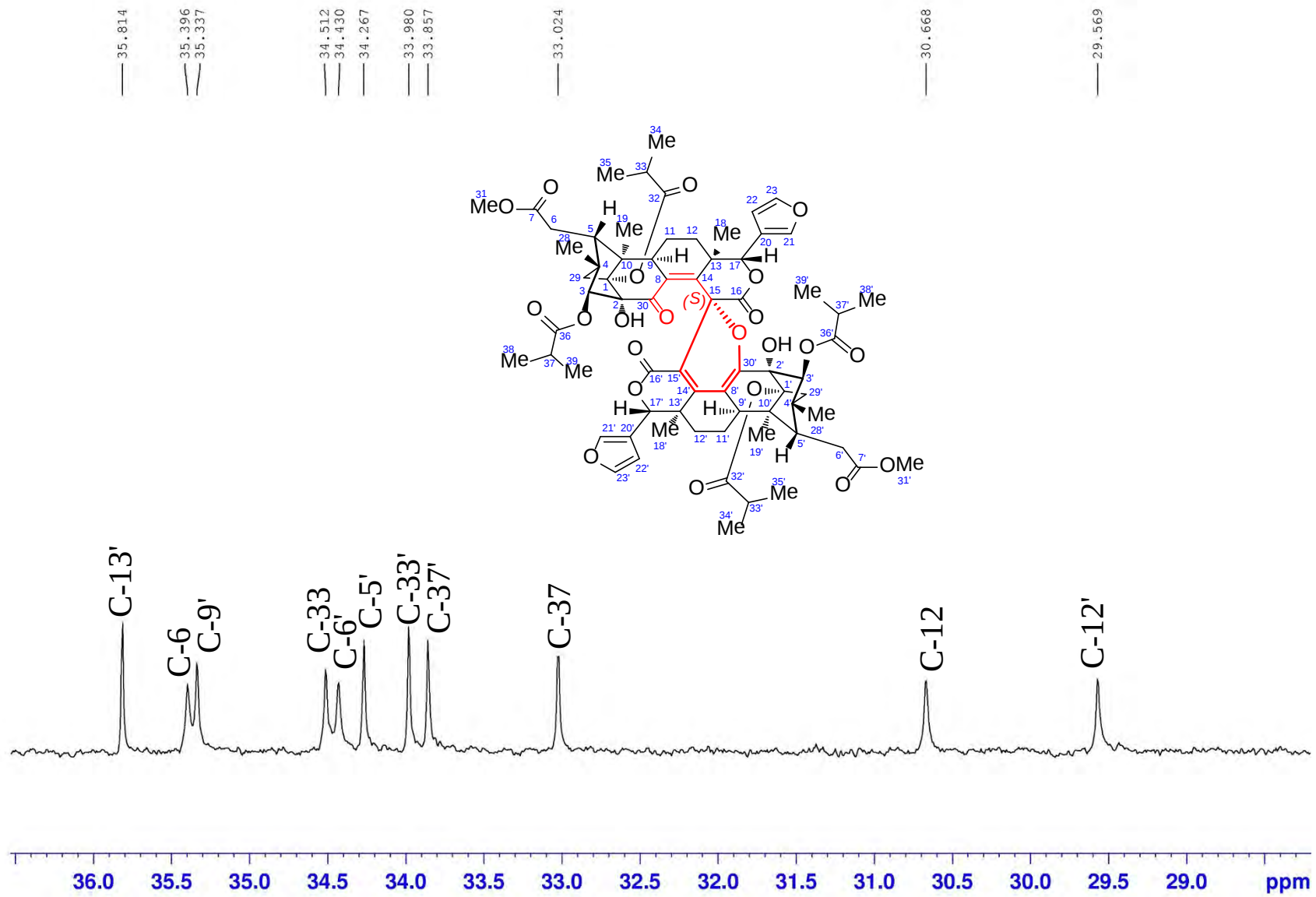
^{13}C NMR (100 MHz) spectrum of compound **3'** in CDCl_3



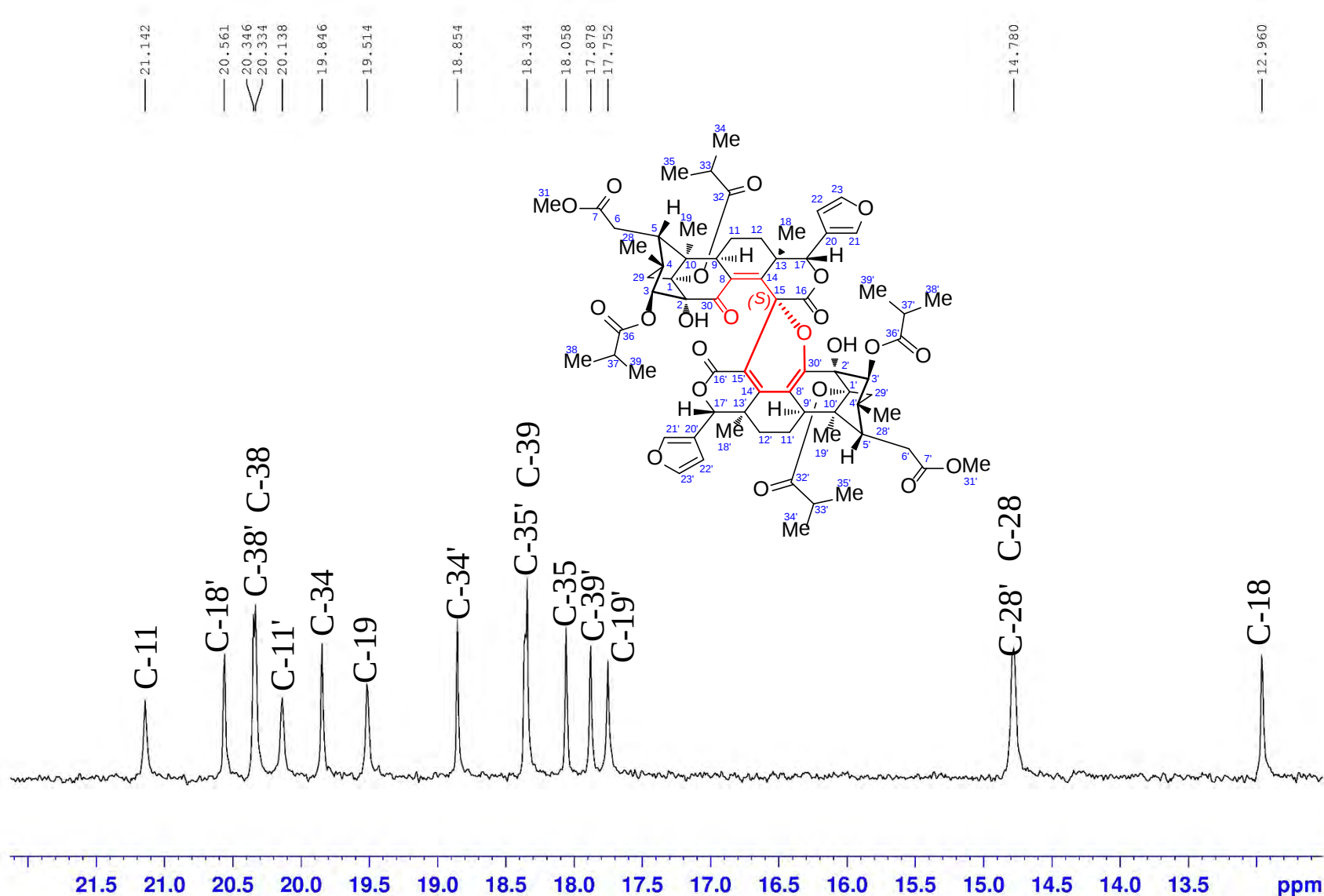
^{13}C NMR (100 MHz) spectrum of compound **3'** in CDCl_3



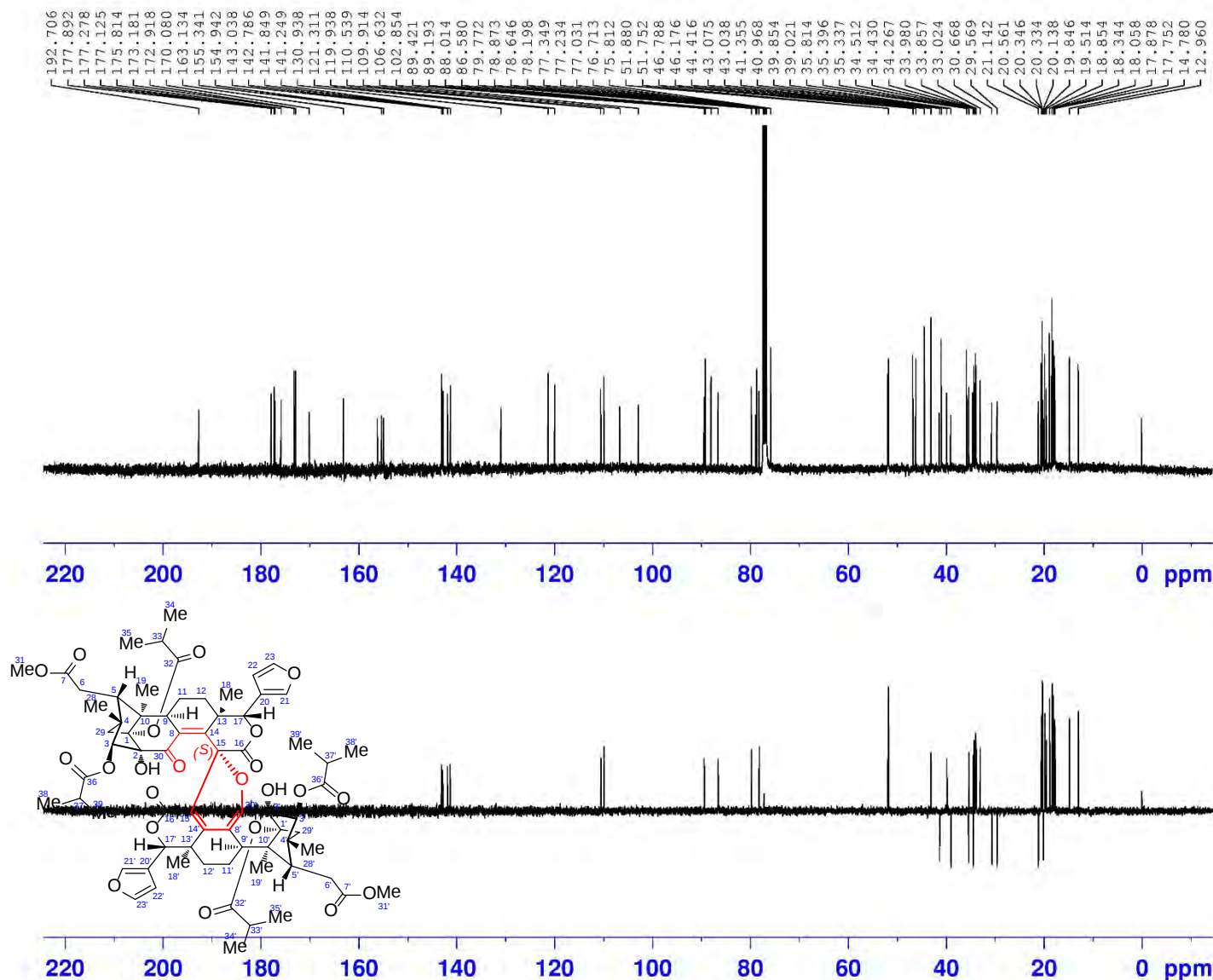
¹³C NMR (100 MHz) spectrum of compound **3'** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



DEPT135 (100 MHz) spectrum of compound 3' in CDCl₃



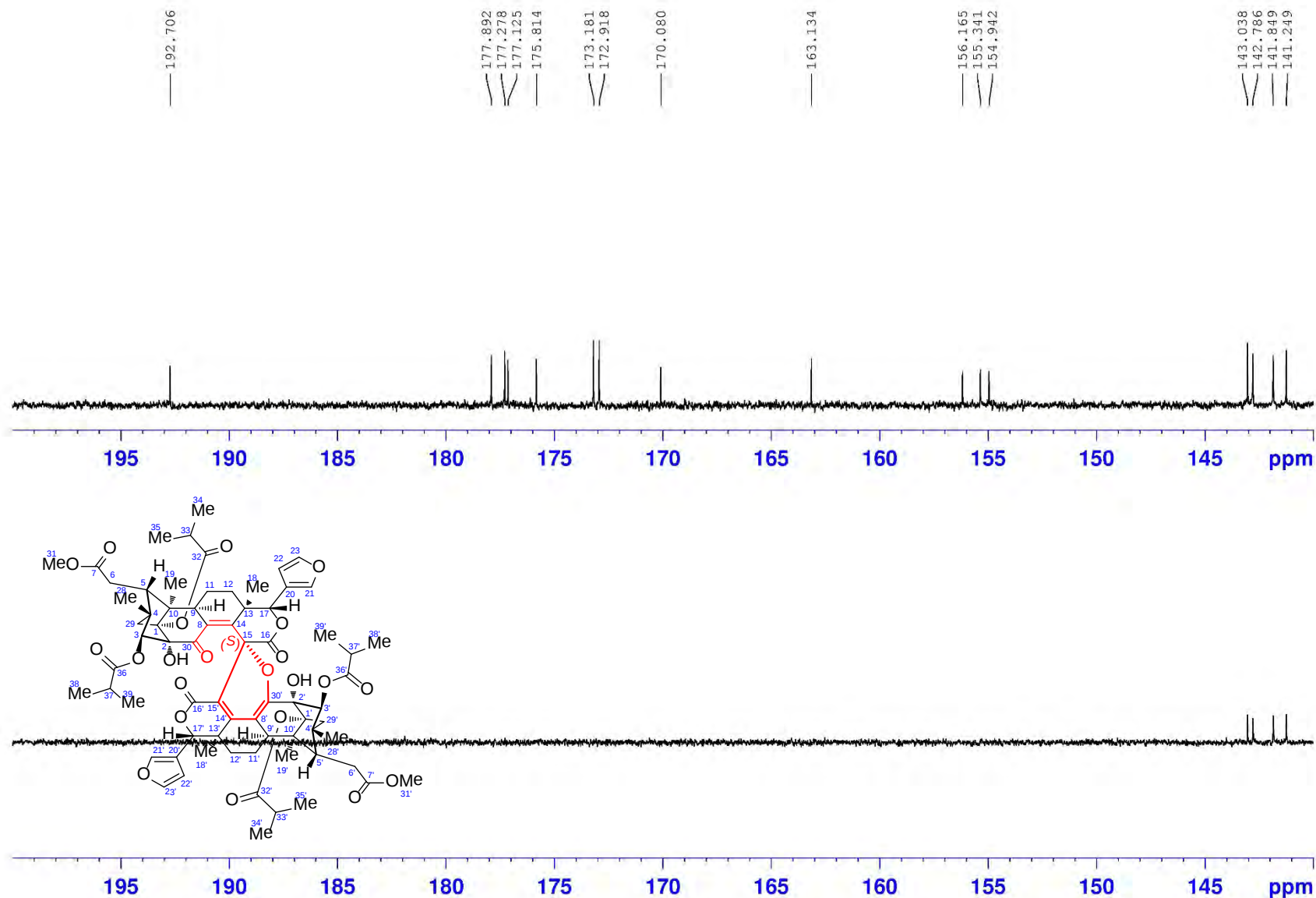
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NAME      lws-37-1 (ddq)
EXPNO     3
PROCNO    1
Date_     20160523
Time      21.23
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   deptsp135
TD         65536
SOLVENT   CDC13
NS         300
DS         4
SWH        24038.461
FIDRES     0.366798
AQ         1.3631988
RG         130.26
DW         20.800
DE         18.00
TE         297.0
CNST2     145.0000000
D1         2.00000000
D2         0.00344828
D12        0.00002000
TD0        1
    
```

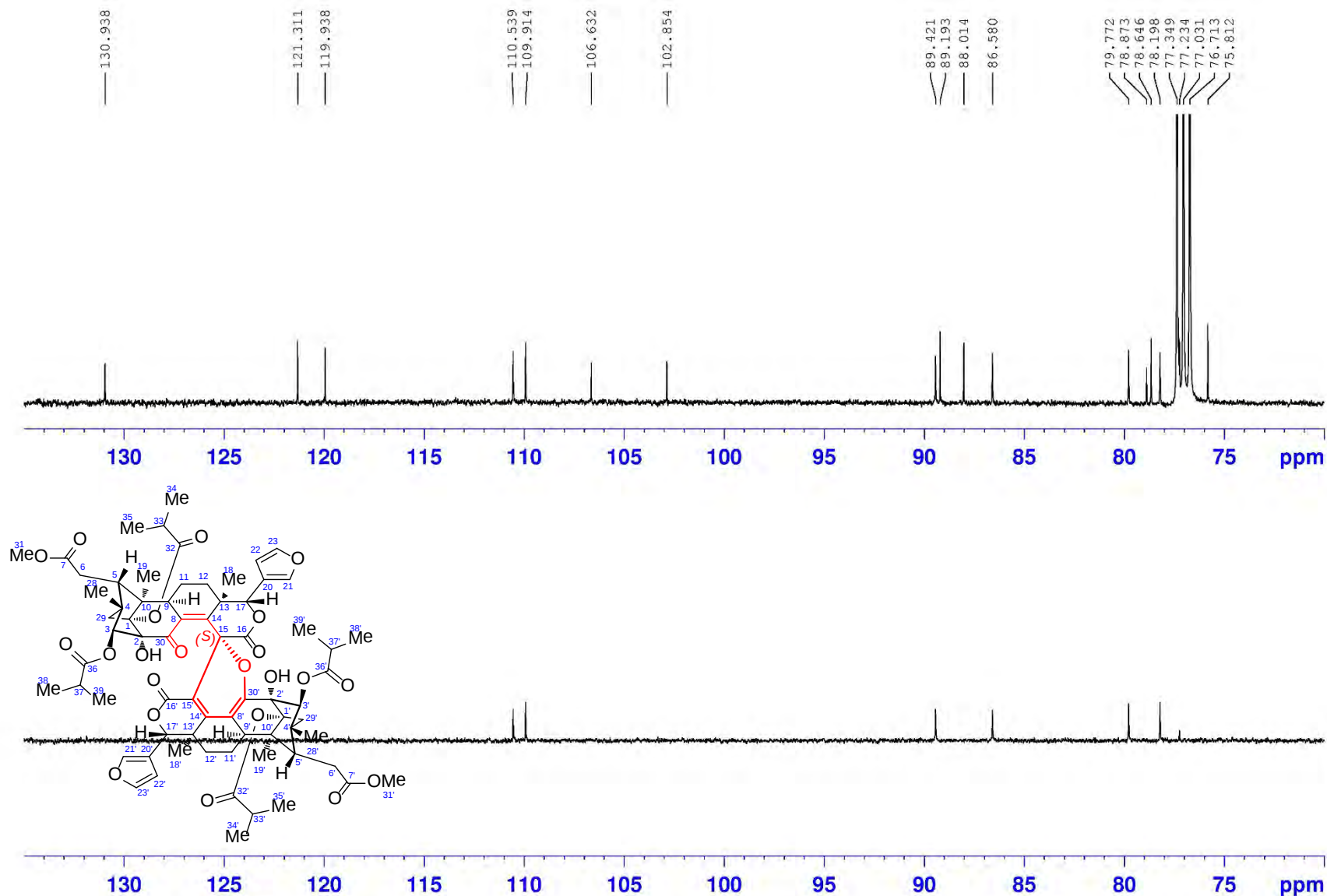
```

===== CHANNEL f1 =====
SFO1      100.6233324
NUC1       13C
P1         10.00
P13        2000.00
SI         32768
SF         100.6127685
WDW         EM
SSB         0
LB          1.00
GB          0
PC          1.40
    
```

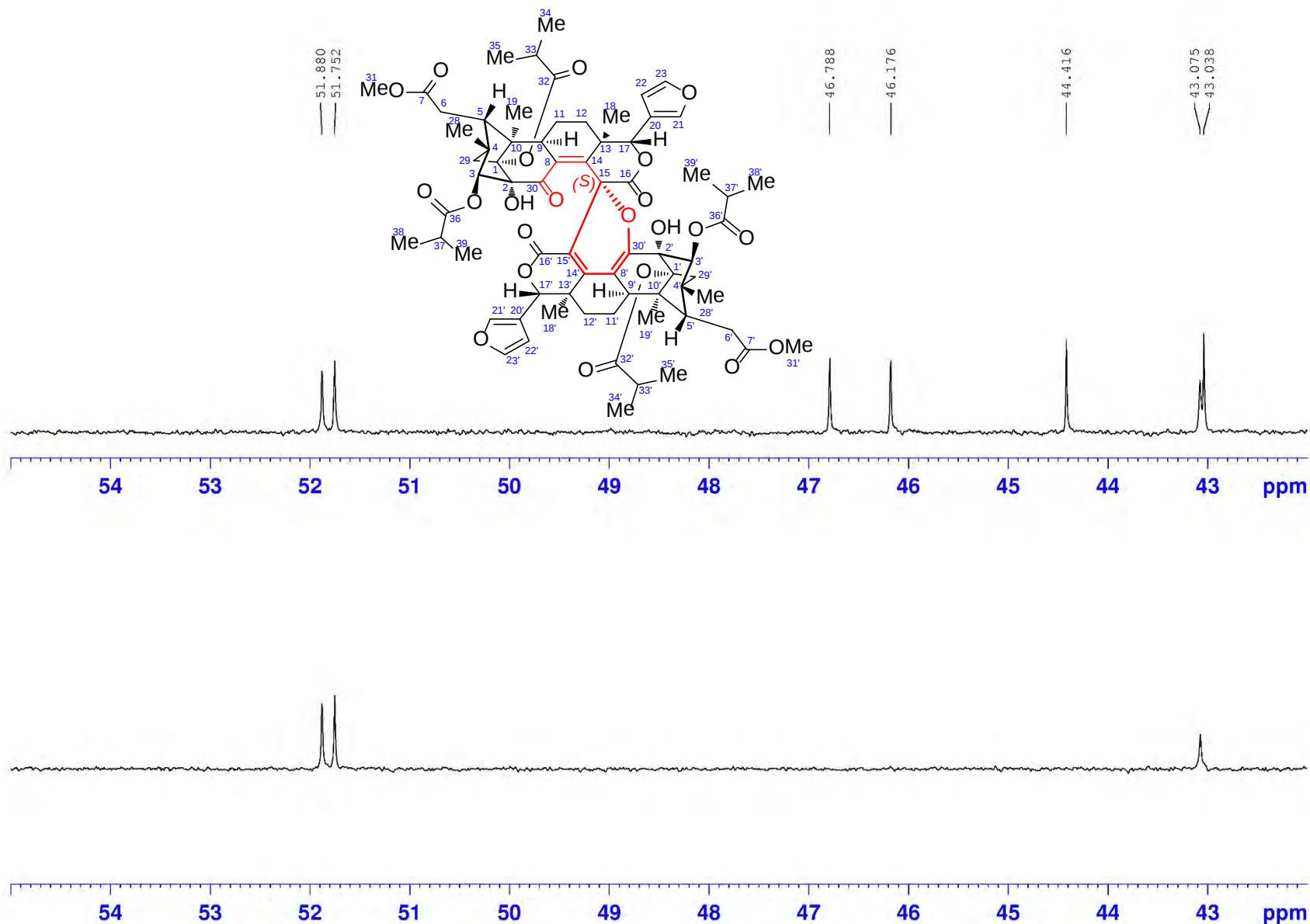
DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



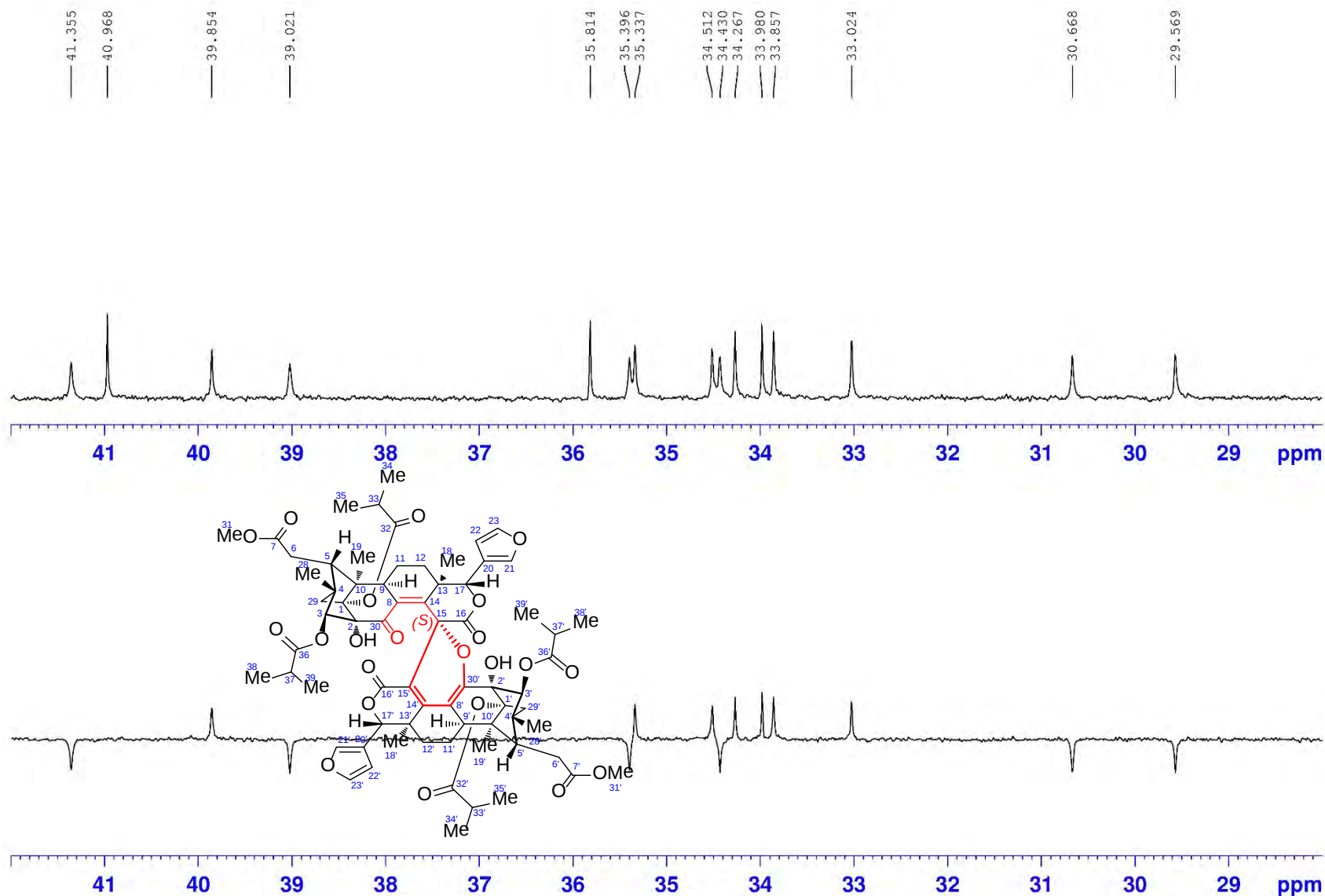
DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



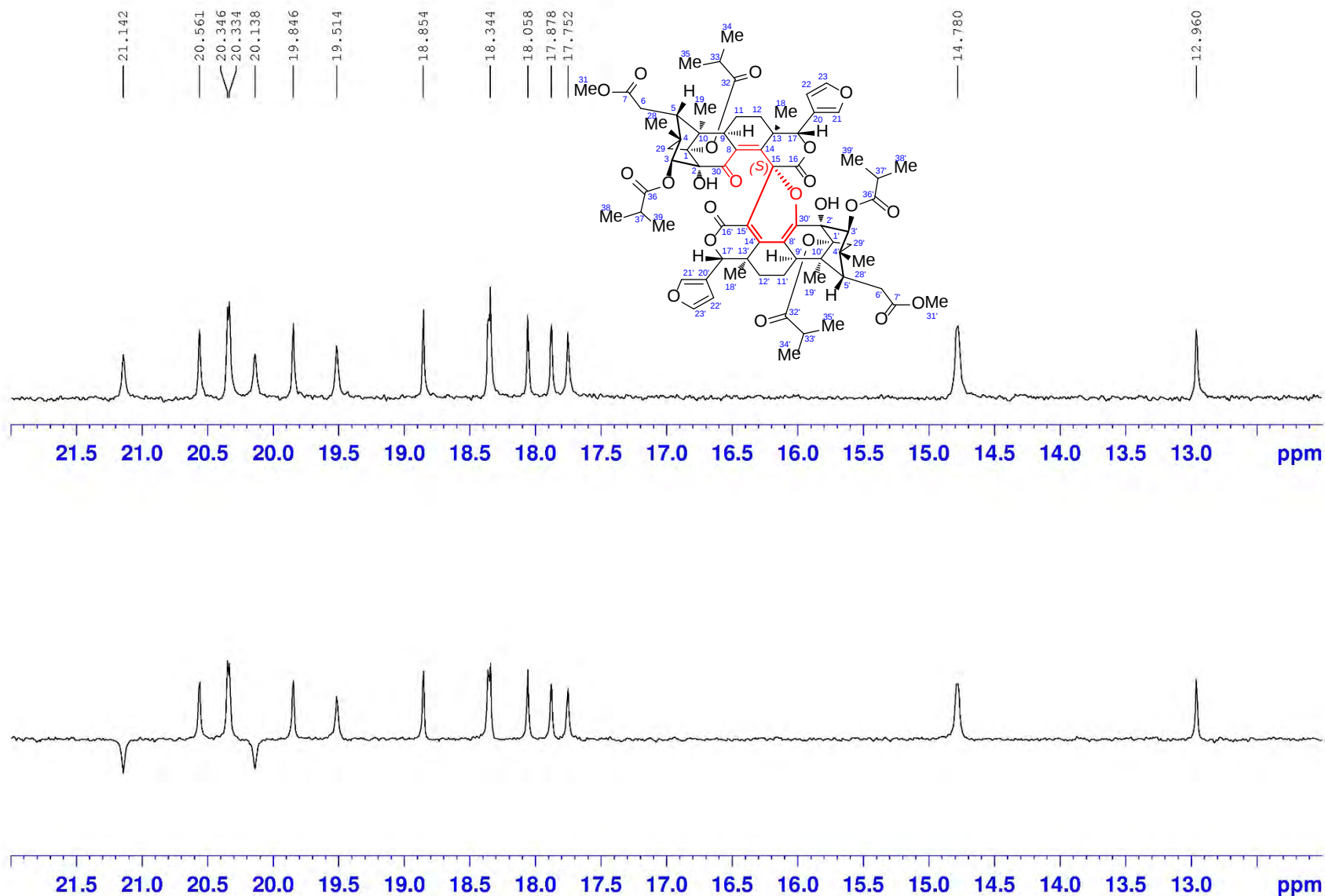
DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



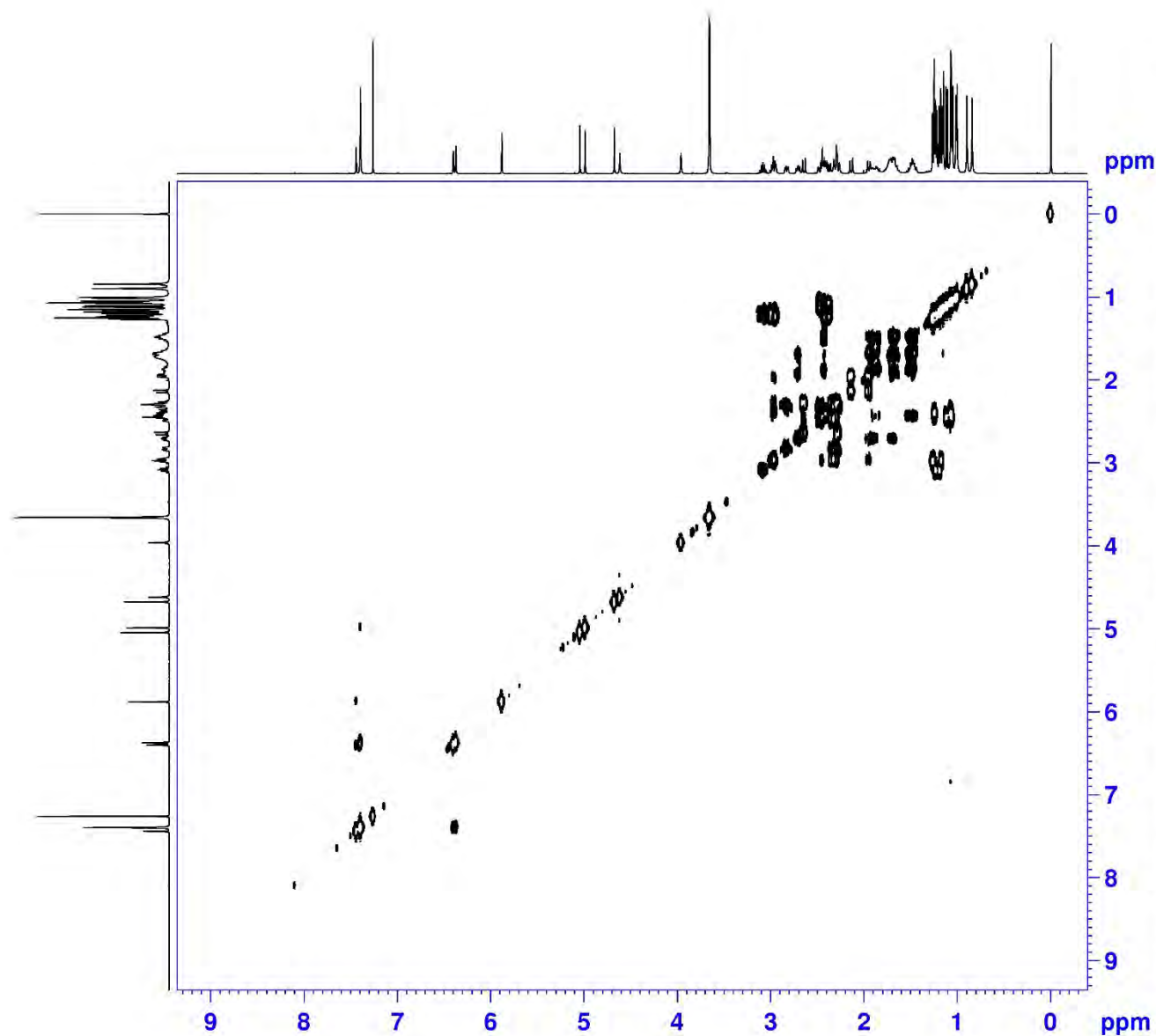
DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **3'** in CDCl₃



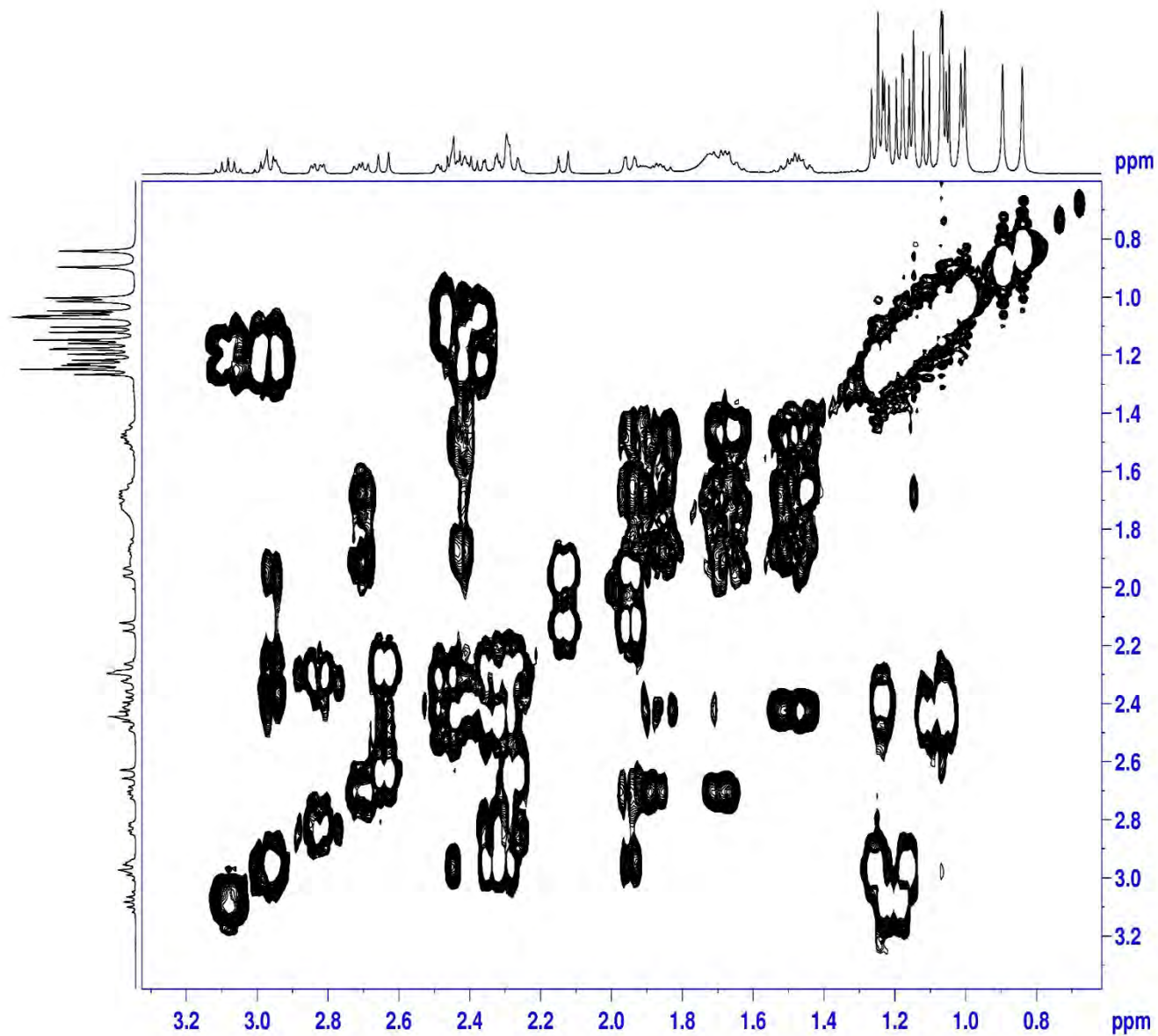
^1H - ^1H COSY (400 MHz) spectrum of compound **3'** in CDCl_3



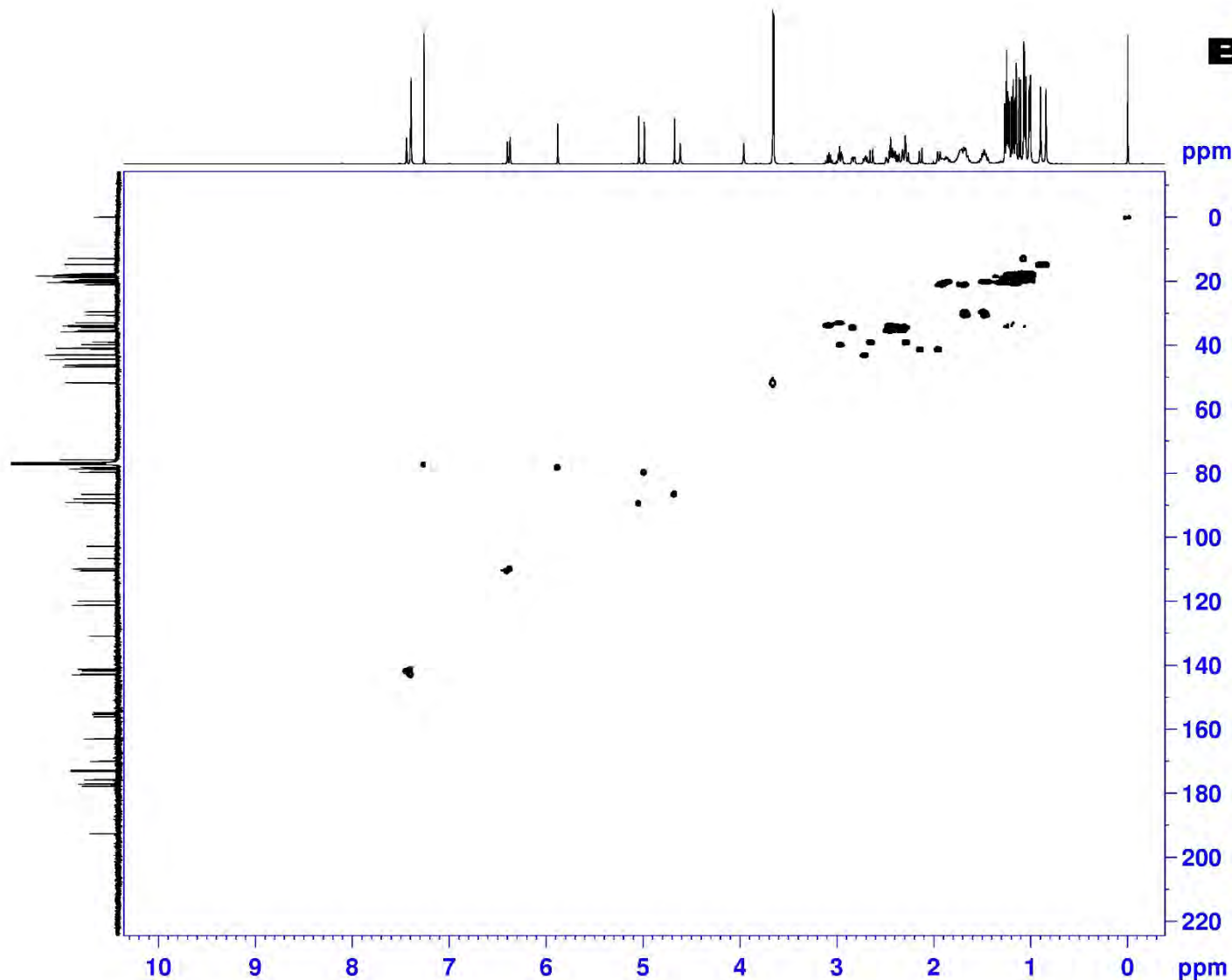
NAME lws-37-1 (ddq)
EXPNO 5
PROCNO 1
Date_ 20160524
Time 3.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG cosygpppqf
TD 2048
SOLVENT CDCl_3
NS 8
DS 8
SWH 3906.250 Hz
FIDRES 1.907349 Hz
AQ 0.2621940 sec
RG 208.5
DW 128.000 usec
DE 10.00 usec
TE 297.0 K
D0 0.00000300 sec
D1 1.89678097 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00025600 sec

===== CHANNEL f1 =====
SFO1 400.1318006 MHz
NUC1 ^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 400.1300098 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 400.1300098 MHz
WDW QSINE

^1H - ^1H COSY (400 MHz) spectrum of compound **3'** in CDCl_3



HSQC (400 MHz) spectrum of compound **3'** in CDCl₃

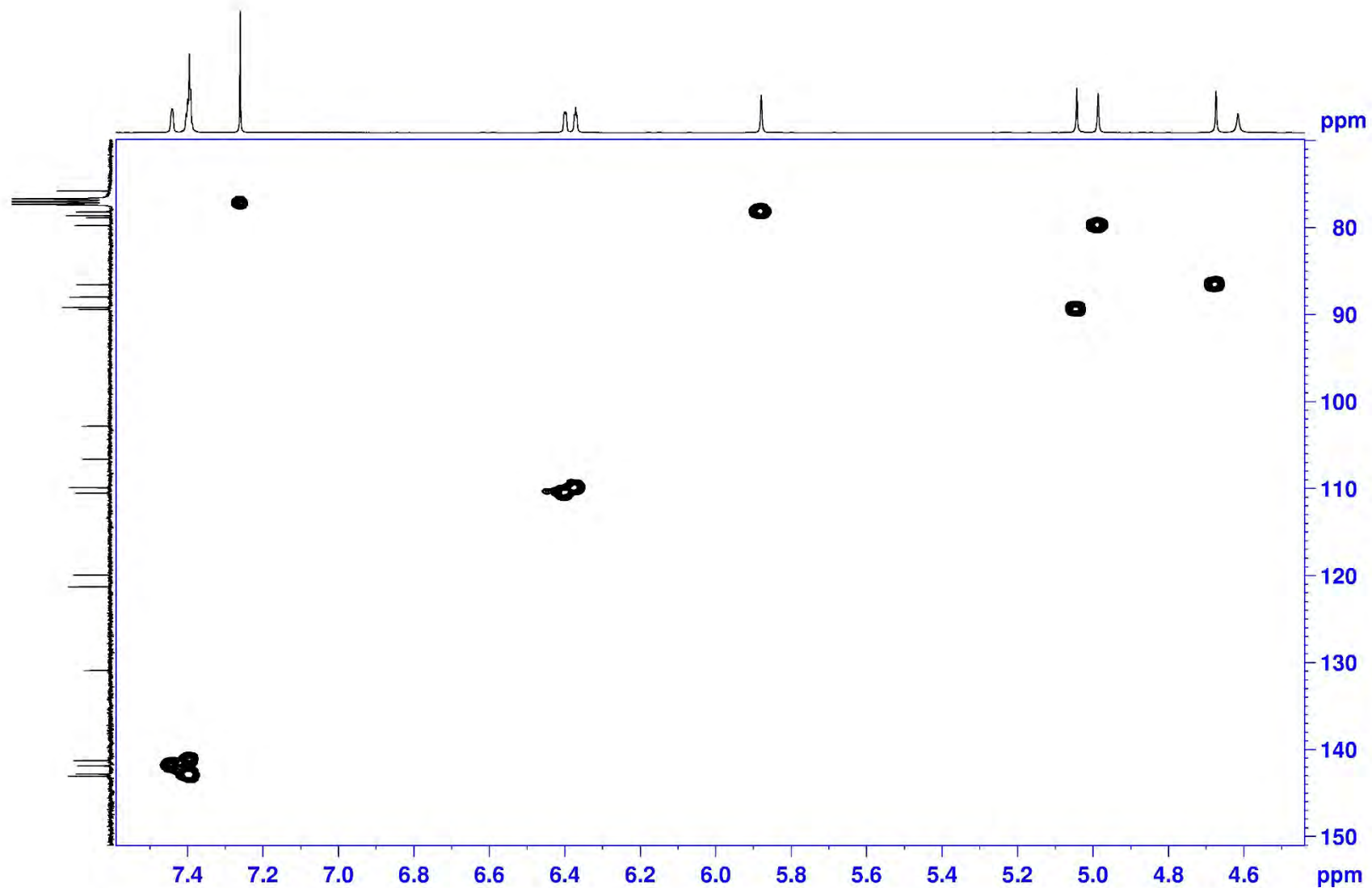


ppm

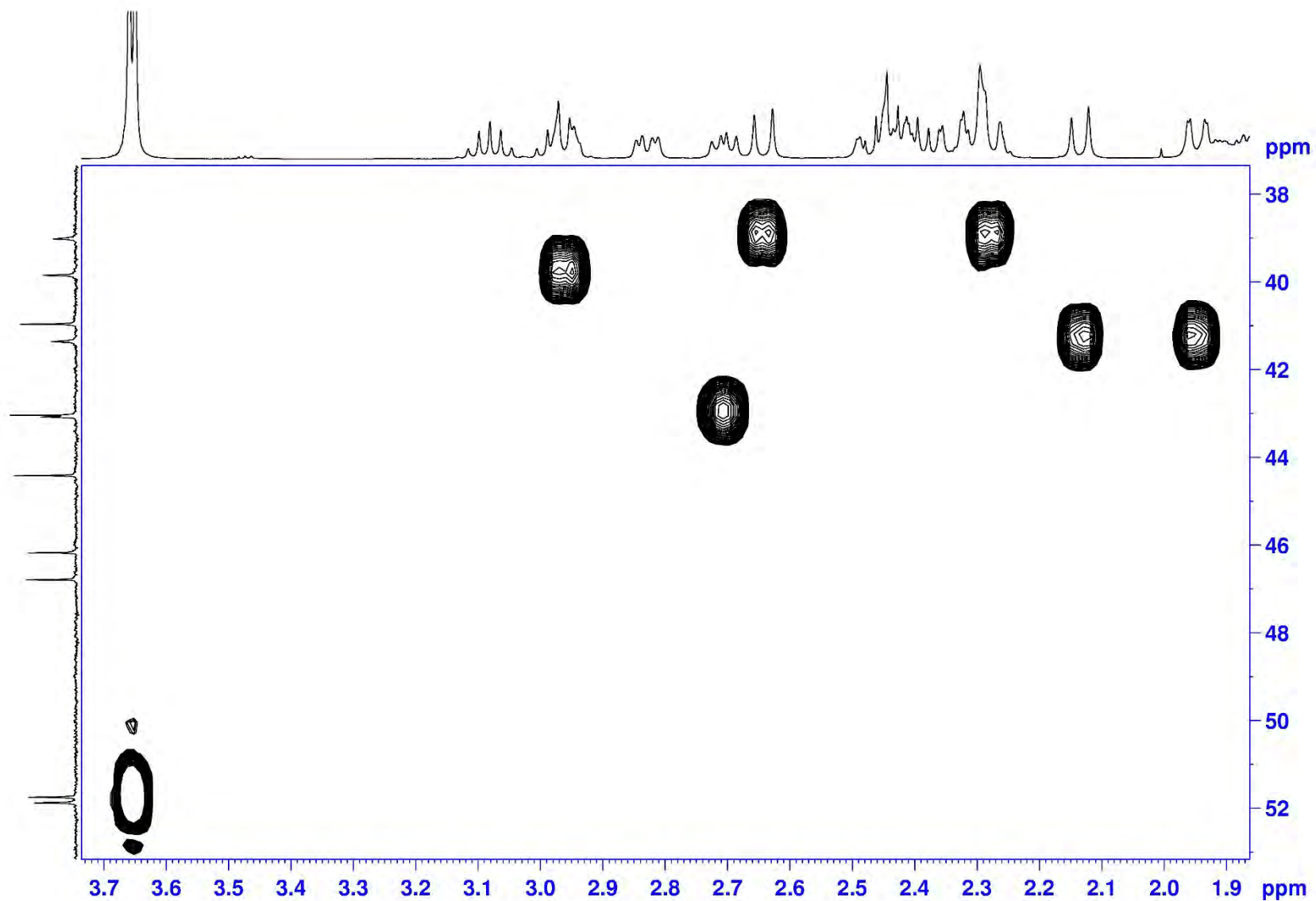
NAME	lws-37-1(ddq)
EXPNO	7
PROCNO	1
Date_	20160524
Time	8.30
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	hsqcetgpsi2
TD	1024
SOLVENT	CDCl3
NS	16
DS	16
SWH	4302.926
FIDRES	4.202076
AQ	0.1190388
RG	208.5
DW	116.200
DE	10.00
TE	297.0
CNST2	145.0000000
D0	0.00000300
D1	1.46497905
D4	0.00172414
D11	0.03000000
D16	0.00020000
D24	0.00086207
IN0	0.00002080
ZGPTNS	
===== CHANNEL f1 =====	
SFO1	400.1320007
NUC1	1H
P1	11.50
P2	23.00
P28	0.00
ND0	2
TD	256
SFO1	100.6233
FIDRES	93.900238
SW	238.896
FnMODE	Echo-Antiecho
SI	1024
SF	400.1300079
WDW	QSINE
SSB	2
LB	0.00
GB	0
PC	1.40
SI	1024
MC2	echo-antiecho
SF	100.6127685
WDW	QSINE
---	---

ppm

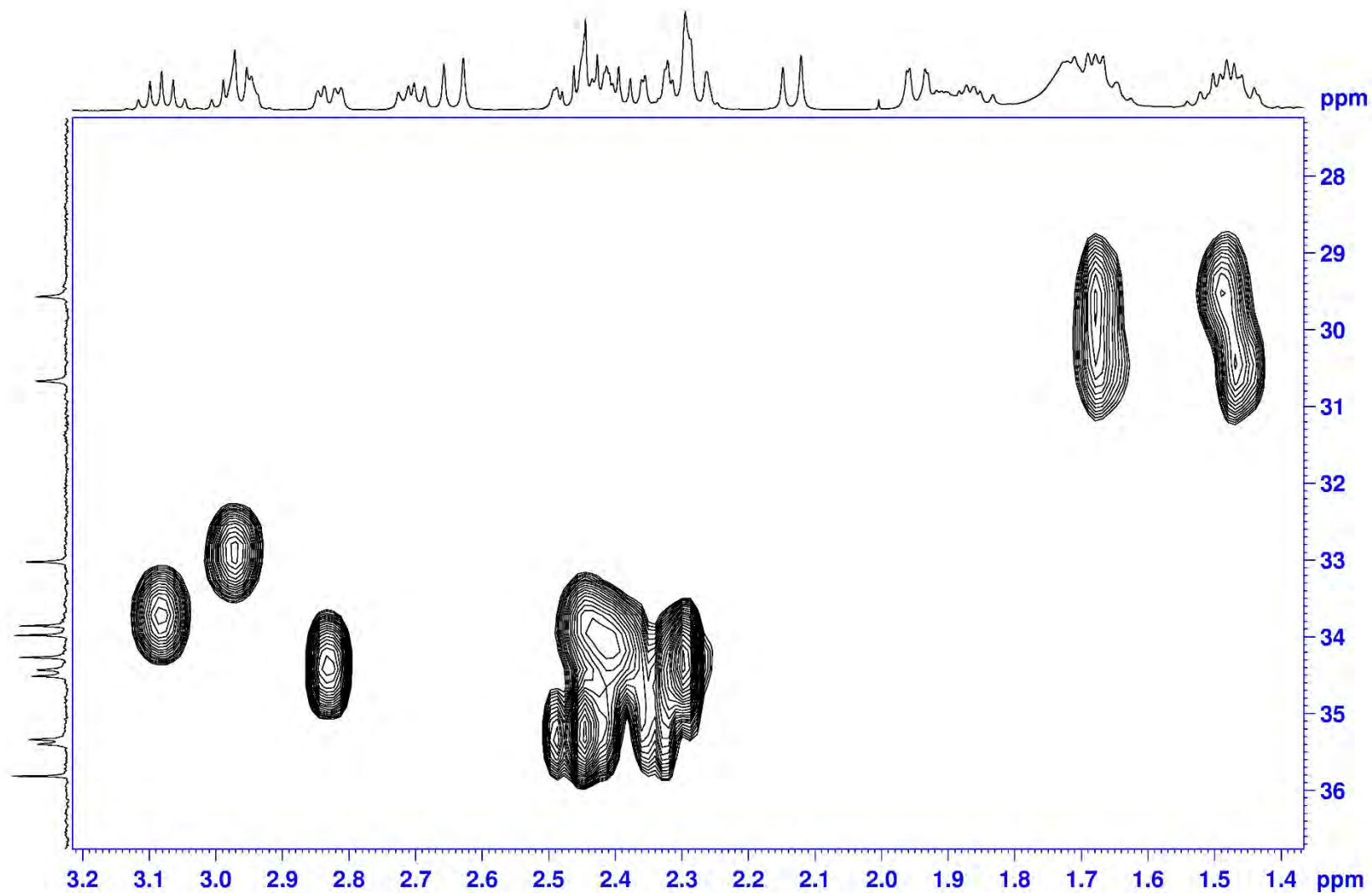
HSQC (400 MHz) spectrum of compound **3'** in CDCl₃



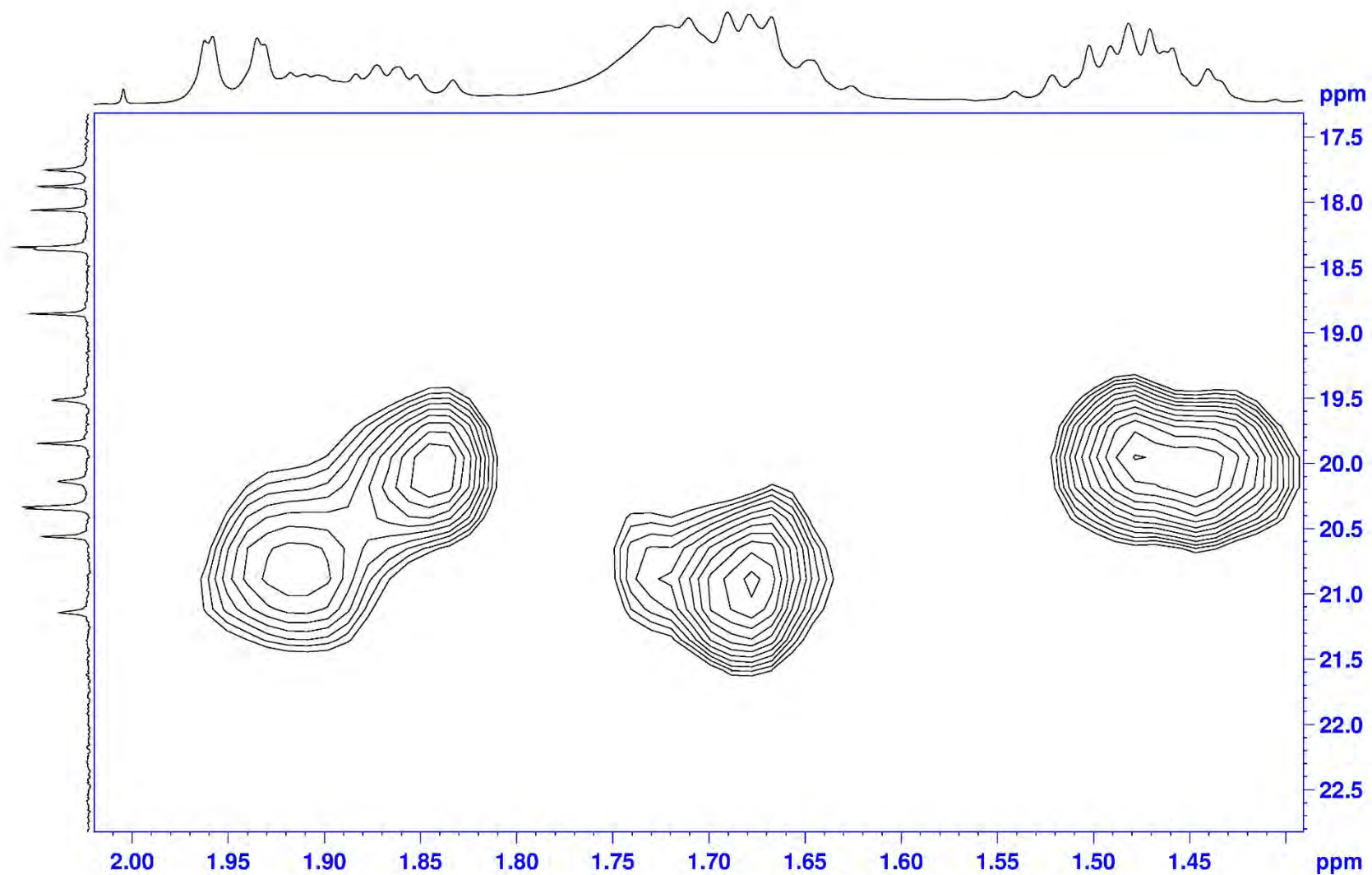
HSQC (400 MHz) spectrum of compound **3'** in CDCl₃



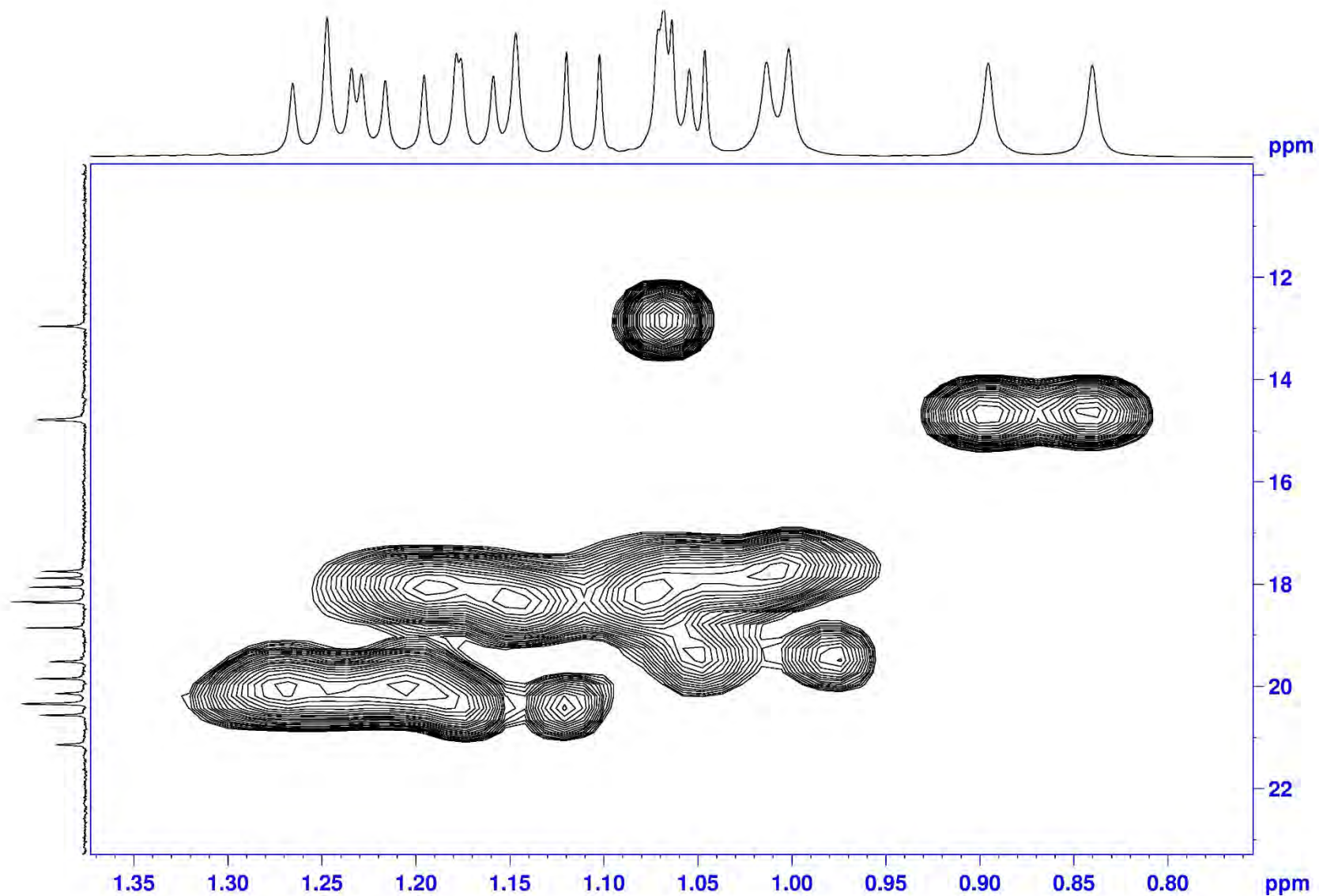
HSQC (400 MHz) spectrum of compound **3'** in CDCl₃



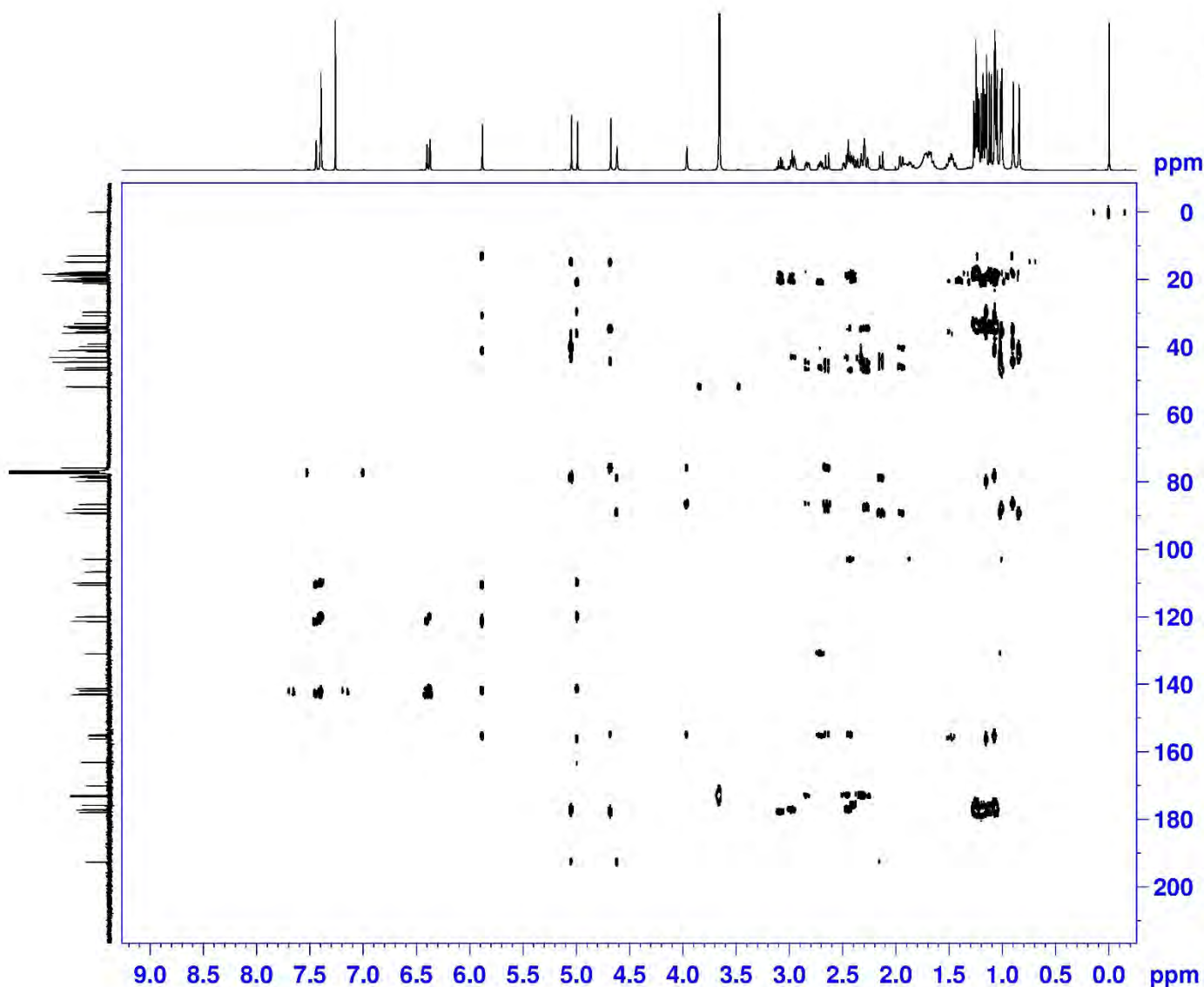
HSQC (400 MHz) spectrum of compound **3'** in CDCl₃



HSQC (400 MHz) spectrum of compound **3'** in CDCl₃



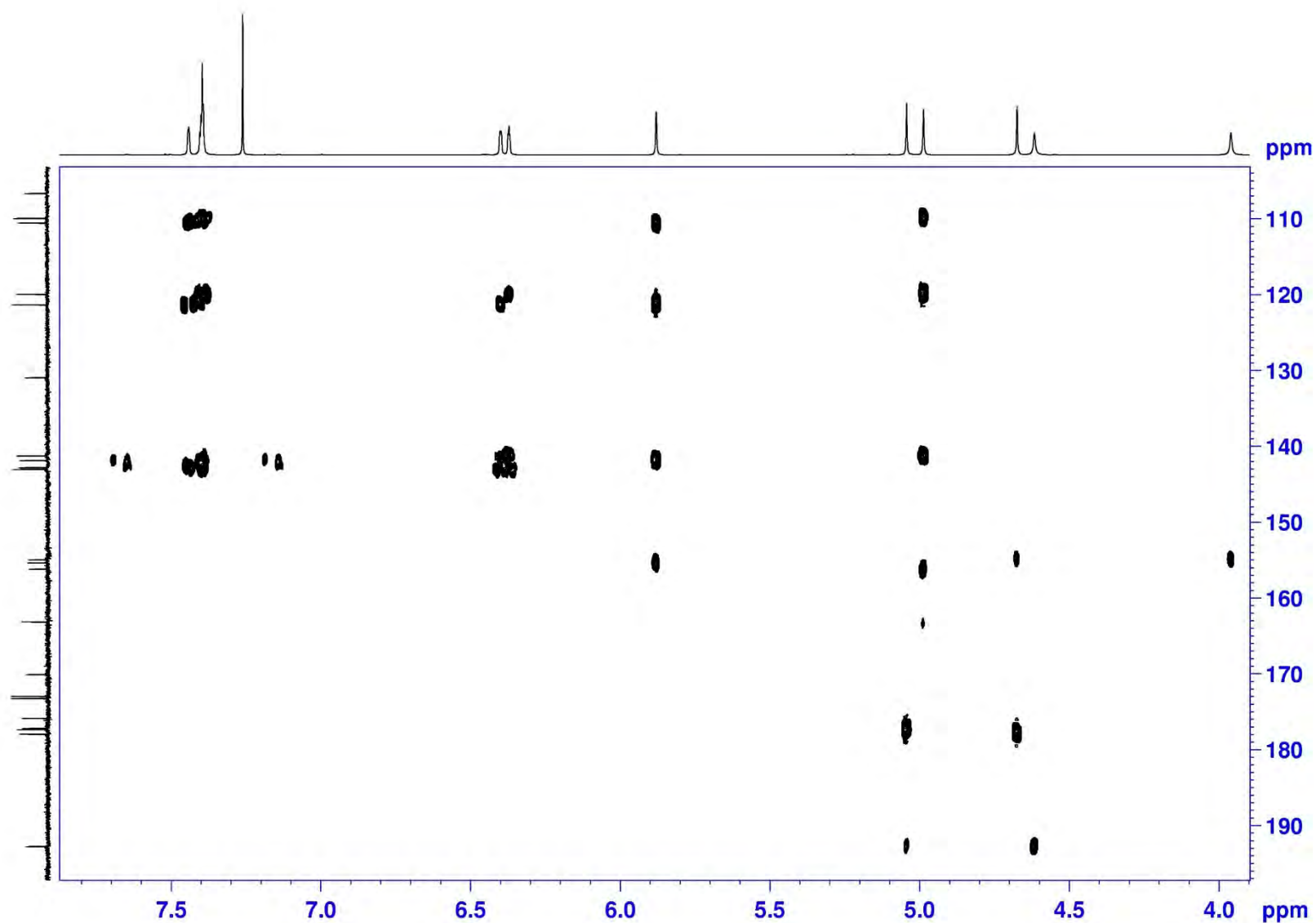
HMBC (400 MHz) spectrum of compound 3' in CDCl₃



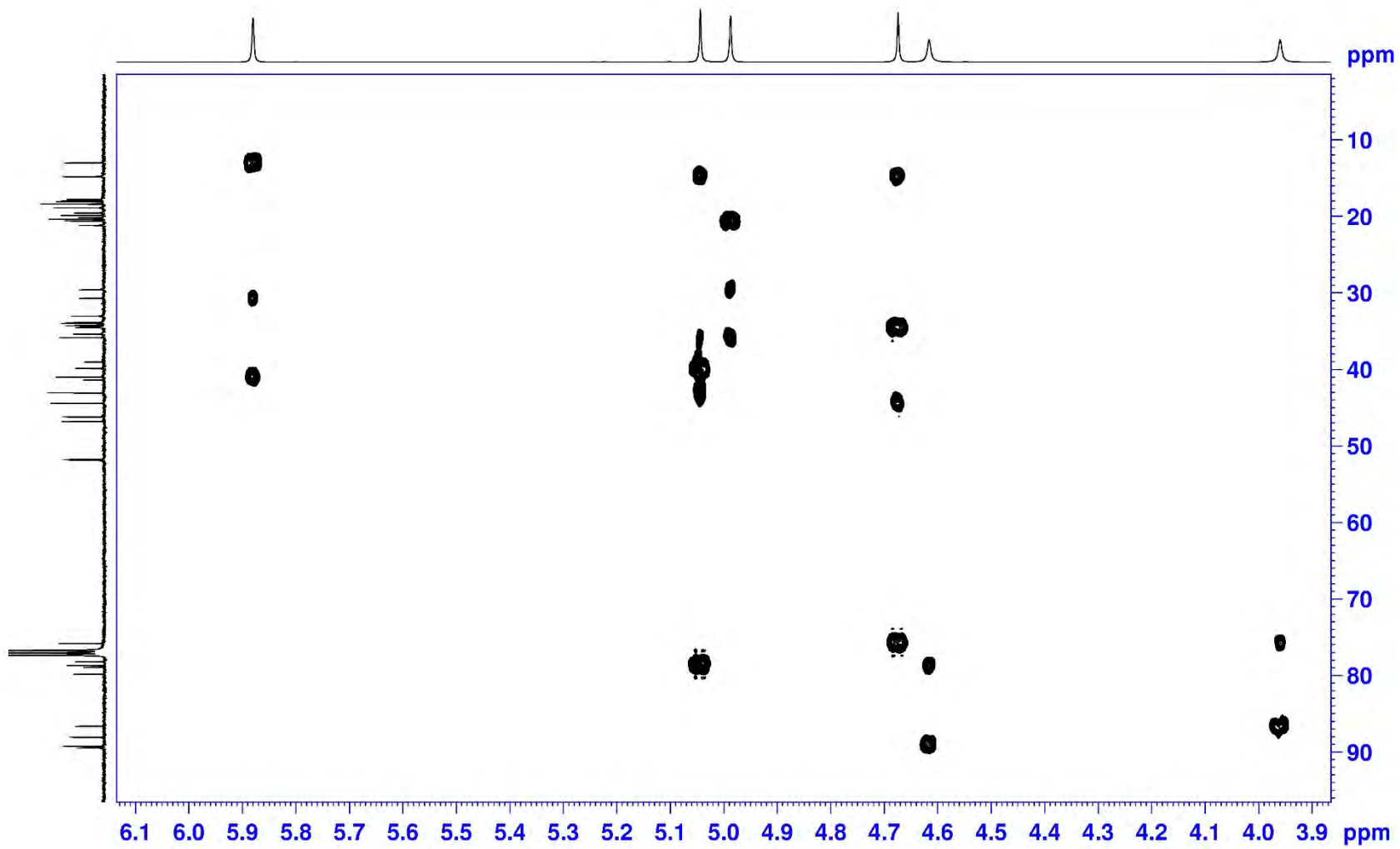
NAME lws-37-1(ddq)
 EXPNO 6
 PROCNO 1
 Date_ 20160524
 Time 4.03
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG hmbcplndqf
 TD 4096
 SOLVENT CDCl3
 NS 64
 DS 16
 SWH 5197.505 Hz
 FIDRES 1.268922 Hz
 AQ 0.3940852 sec
 RG 208.5
 DW 96.200 usec
 DE 10.00 usec
 TE 297.0 K
 CNST2 145.0000000
 CNST13 10.0000000
 D0 0.00000300 sec
 D1 1.50000000 sec
 D2 0.00344828 sec
 D6 0.05000000 sec
 D16 0.00020000 sec
 IN0 0.00002080 sec

===== CHANNEL f1 =====
 SFO1 400.1323208 MHz
 NUC1 1H
 P1 11.50 usec
 P2 23.00 usec
 ND0 2
 TD 128
 SFO1 100.6233 MHz
 FIDRES 187.800476 Hz
 SW 238.896 ppm
 FMODE QF
 SI 2048
 SF 400.1300090 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40
 SI 1024
 MC2 QF
 SF 100.6127685 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz

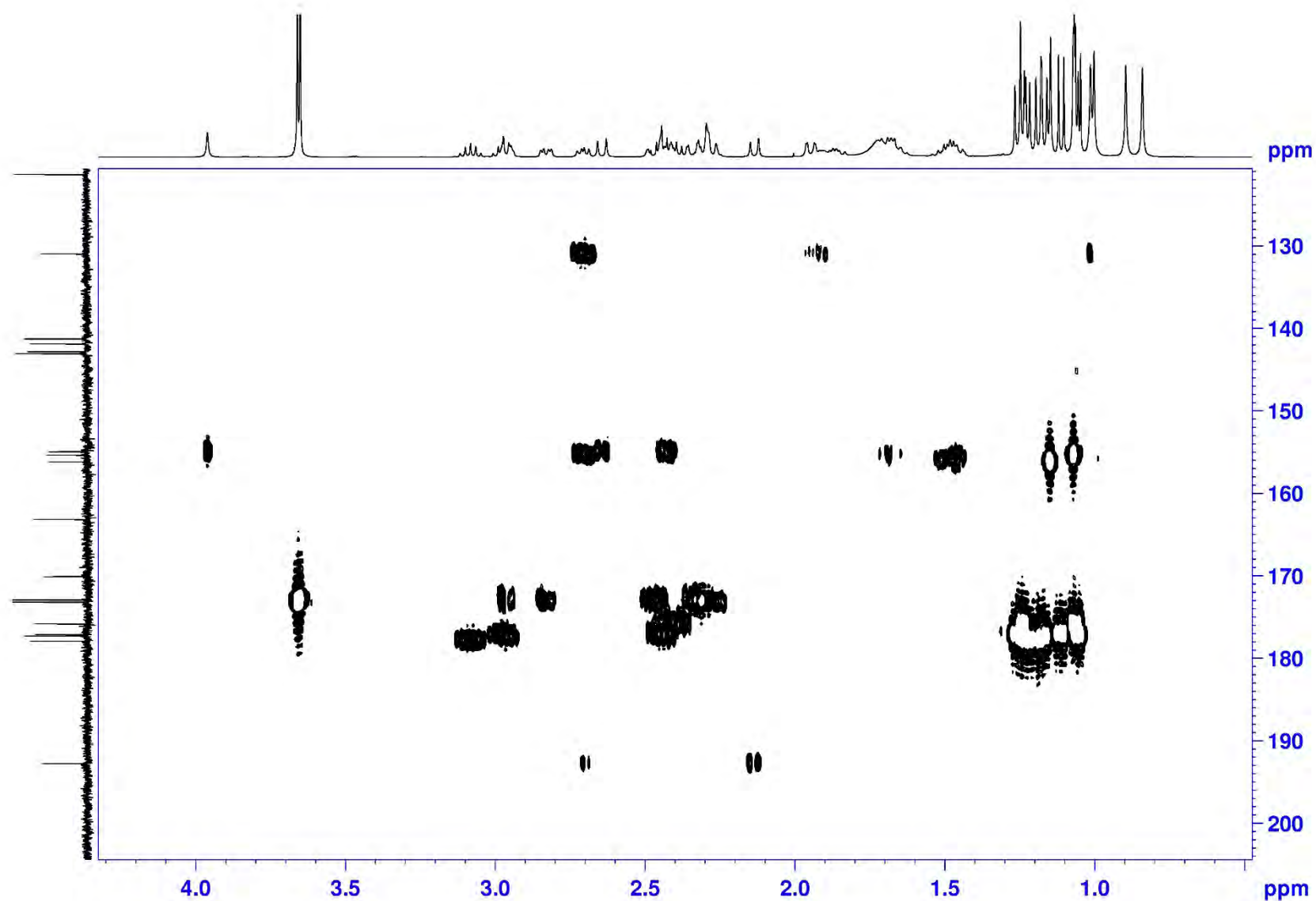
HMBC (400 MHz) spectrum of compound 3' in CDCl₃



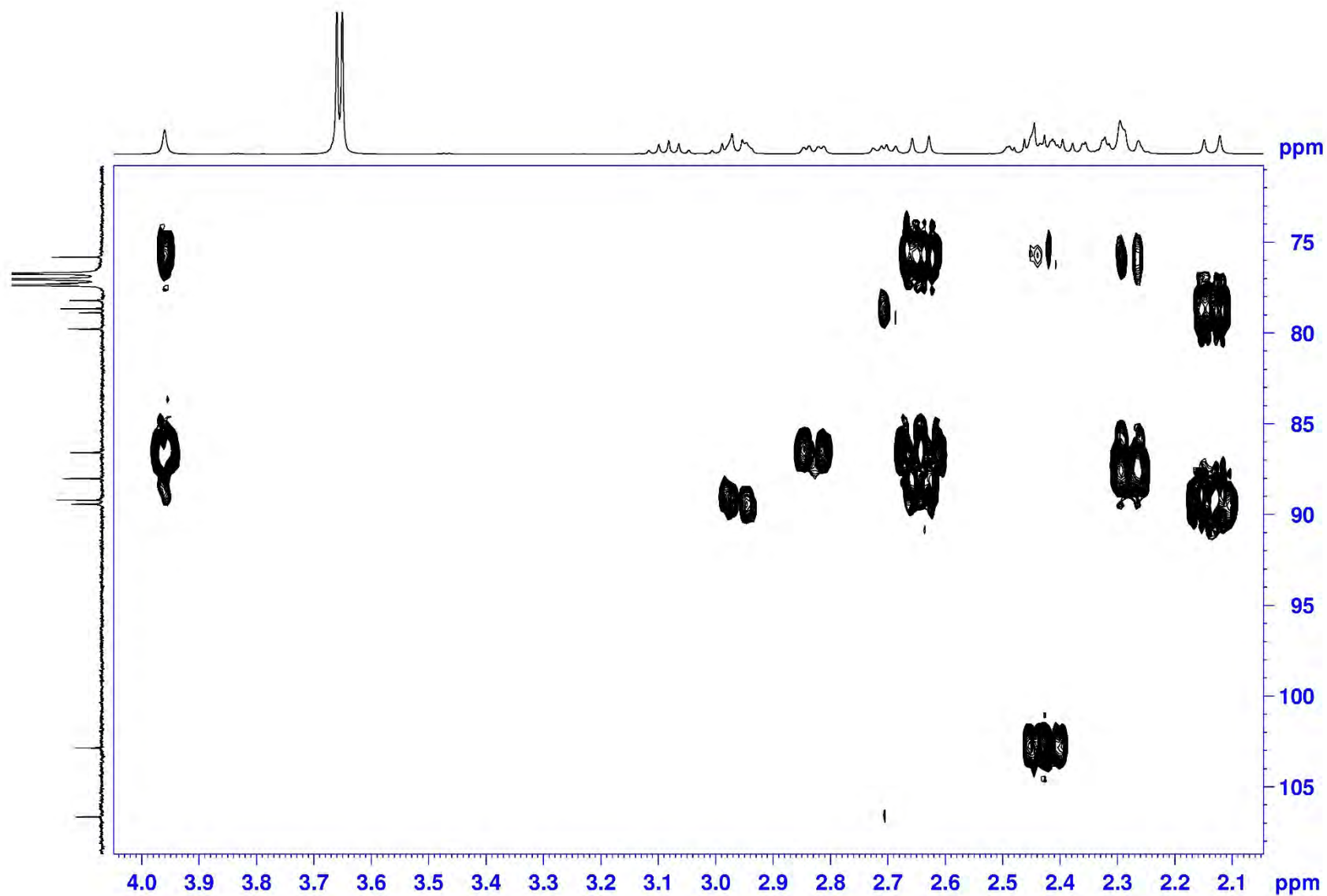
HMBC (400 MHz) spectrum of compound **3'** in CDCl₃



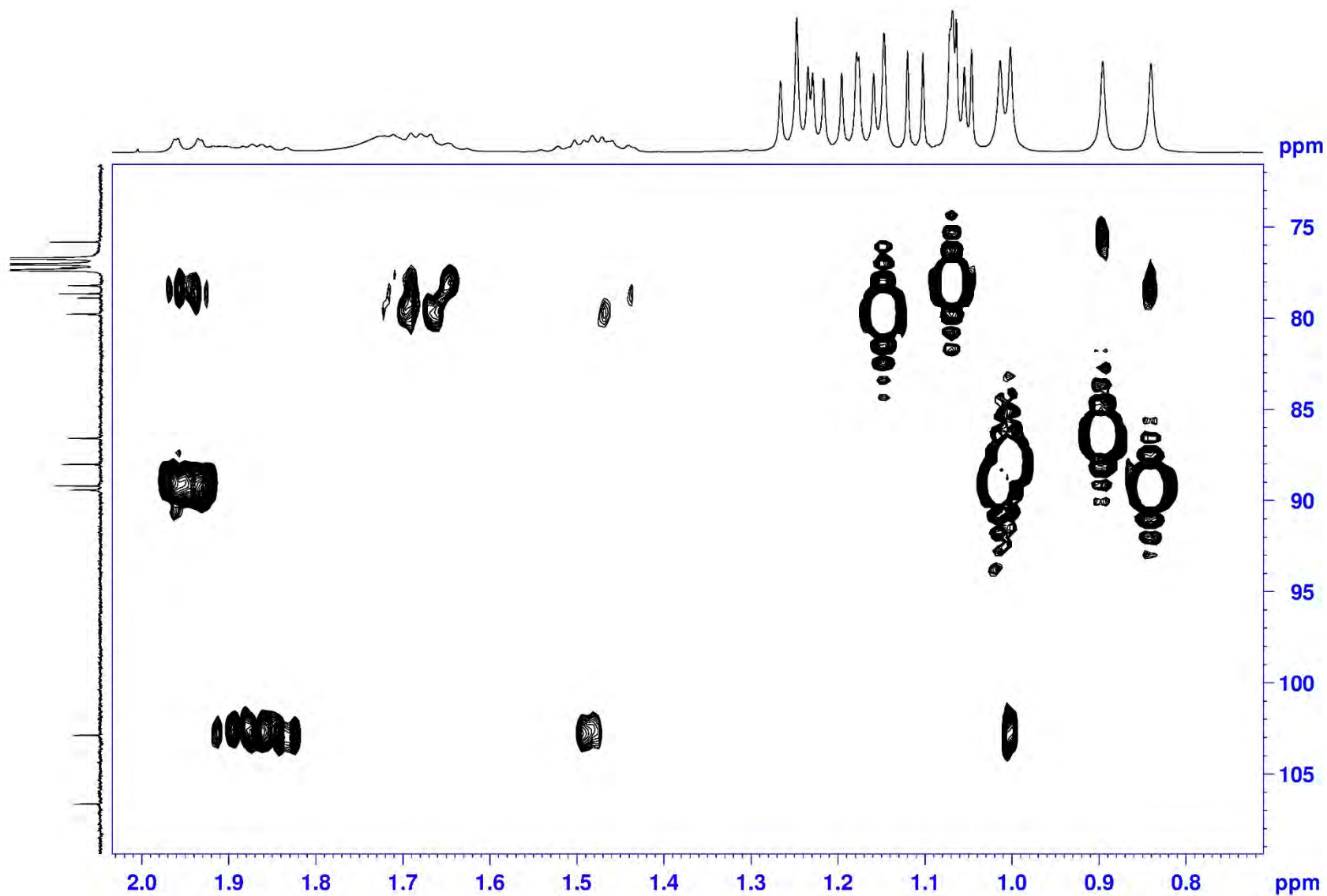
HMBC (400 MHz) spectrum of compound **3'** in CDCl₃



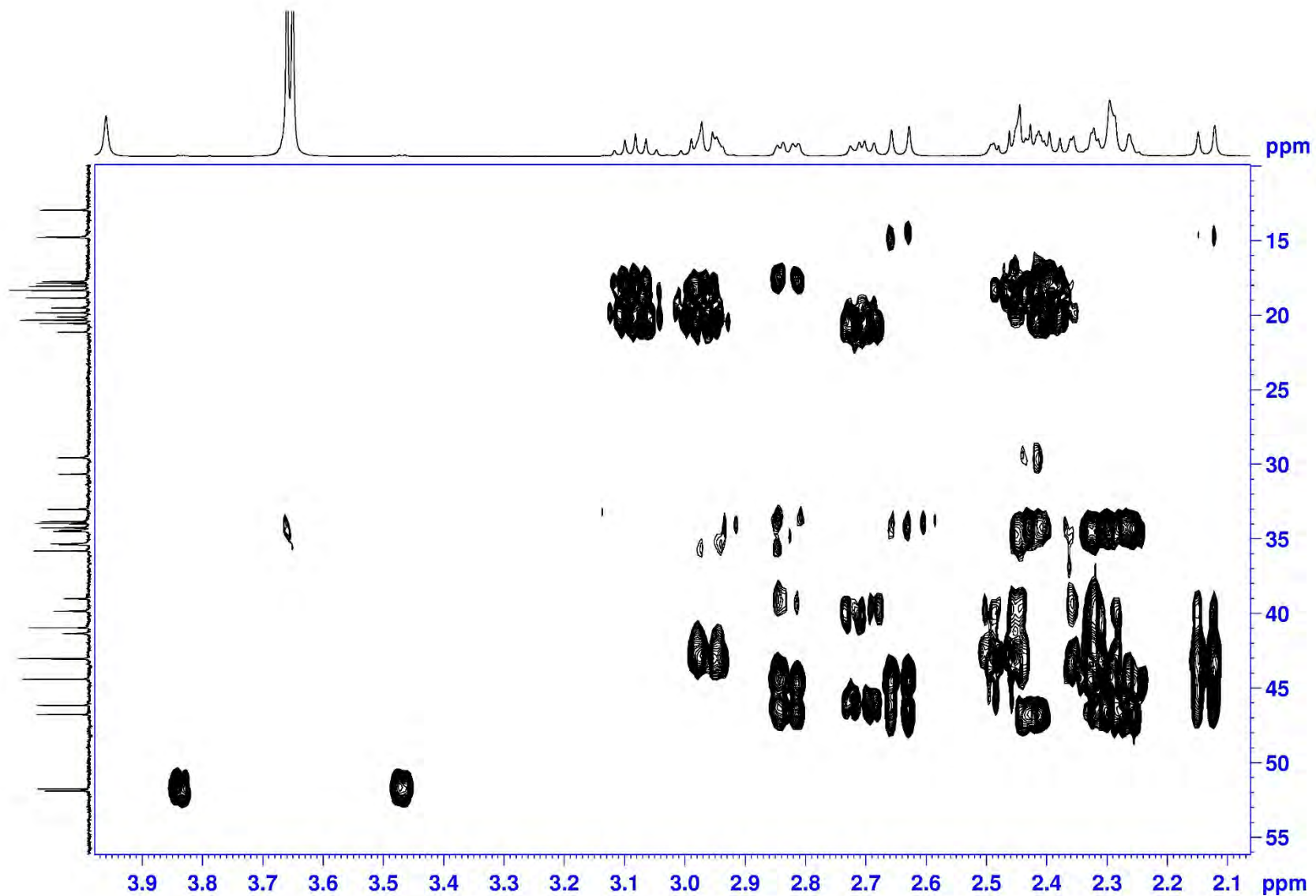
HMBC (400 MHz) spectrum of compound **3'** in CDCl₃



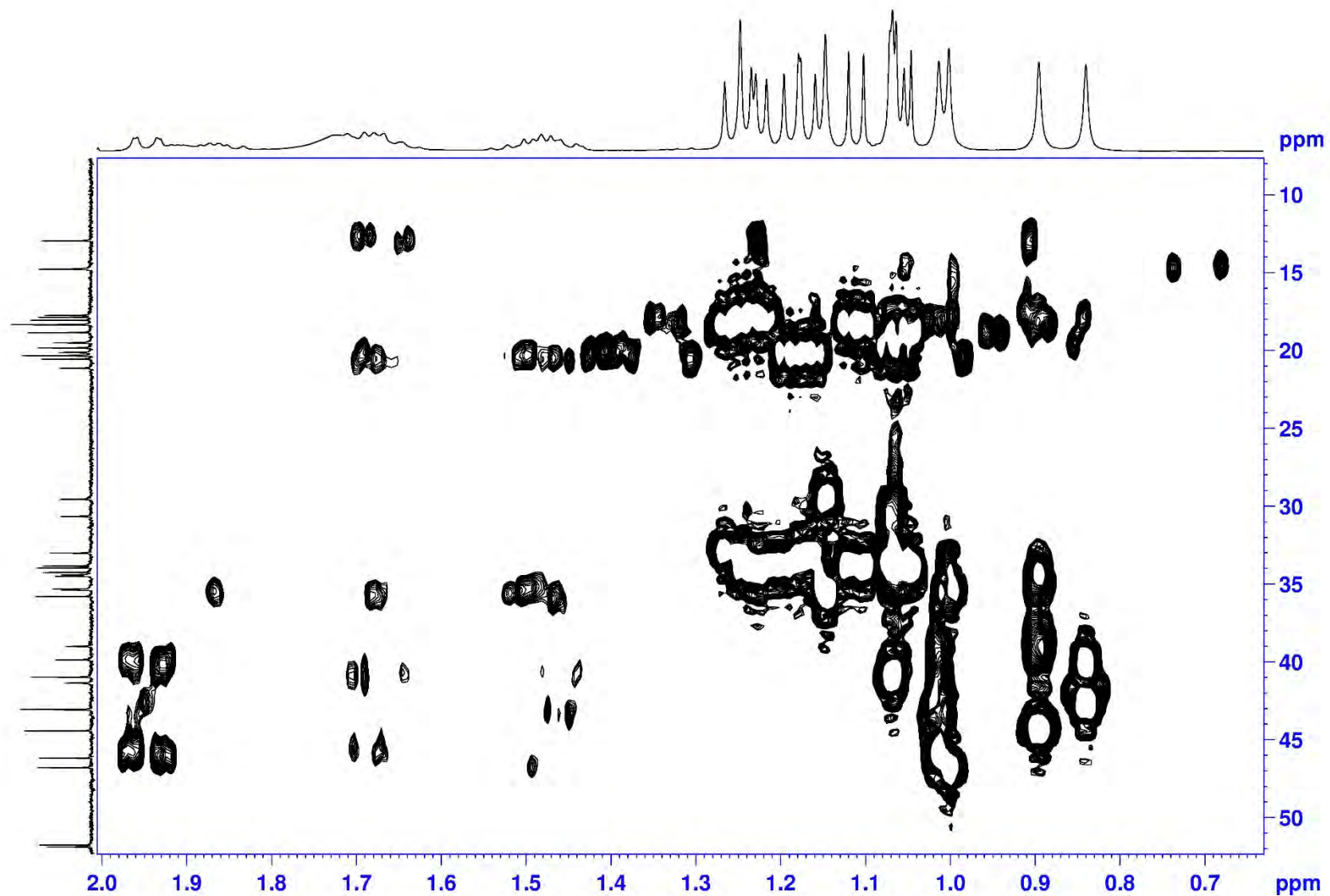
HMBC (400 MHz) spectrum of compound 3' in CDCl₃



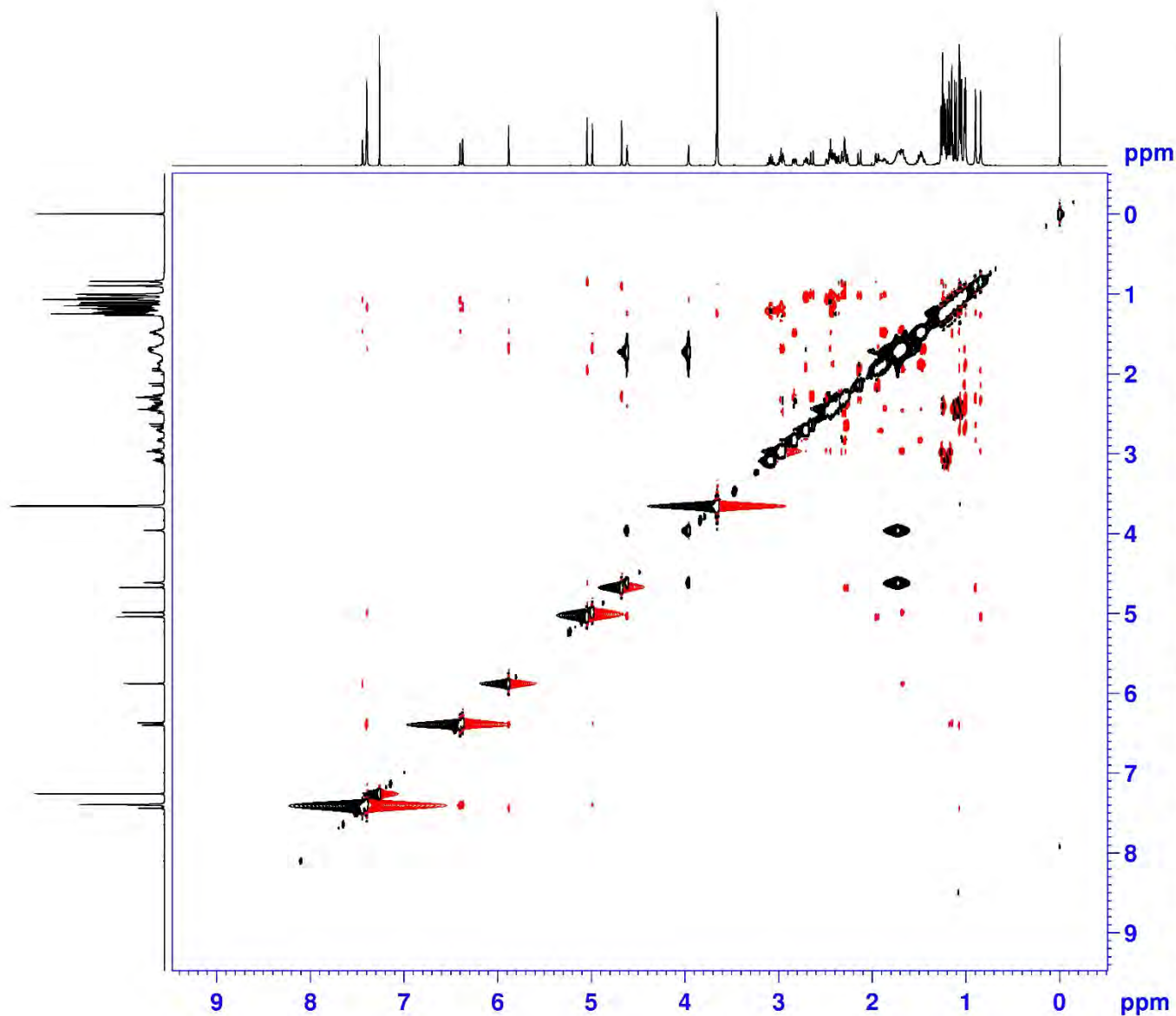
HMBC (400 MHz) spectrum of compound **3'** in CDCl₃



HMBC (400 MHz) spectrum of compound 3' in CDCl₃



NOESY (400 MHz) spectrum of compound **3'** in CDCl₃



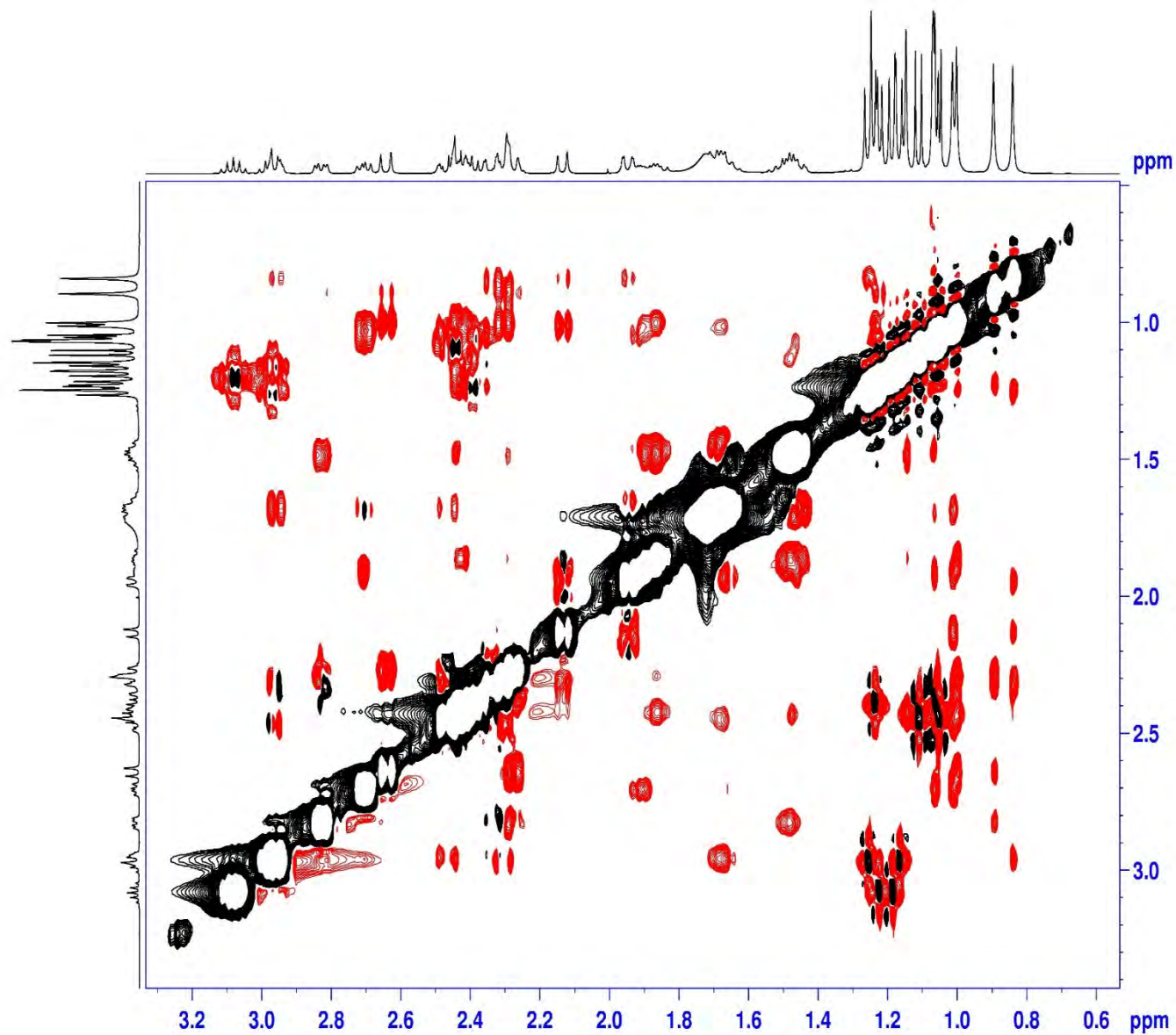
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NAME      lws-37-1 (ddq)
EXPNO     4
PROCNO    1
Date_     20160523
Time      21.26
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   noesygpphph
TD        2048
SOLVENT   CDCl3
NS        32
DS        32
SWH       4000.000 Hz
FIDRES    1.953125 Hz
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
    
```

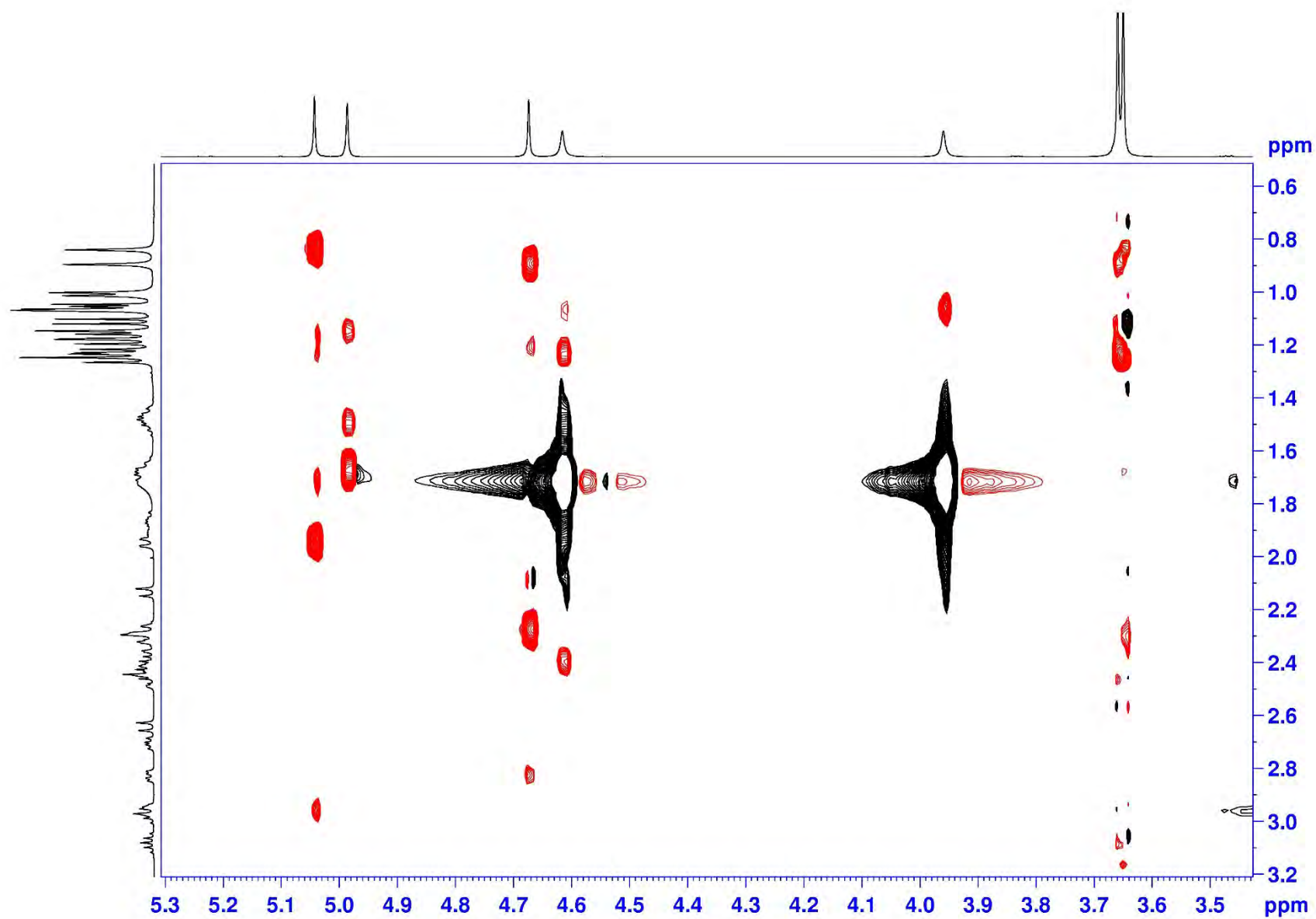
```

===== 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       400.1300098 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
SSB      2
    
```

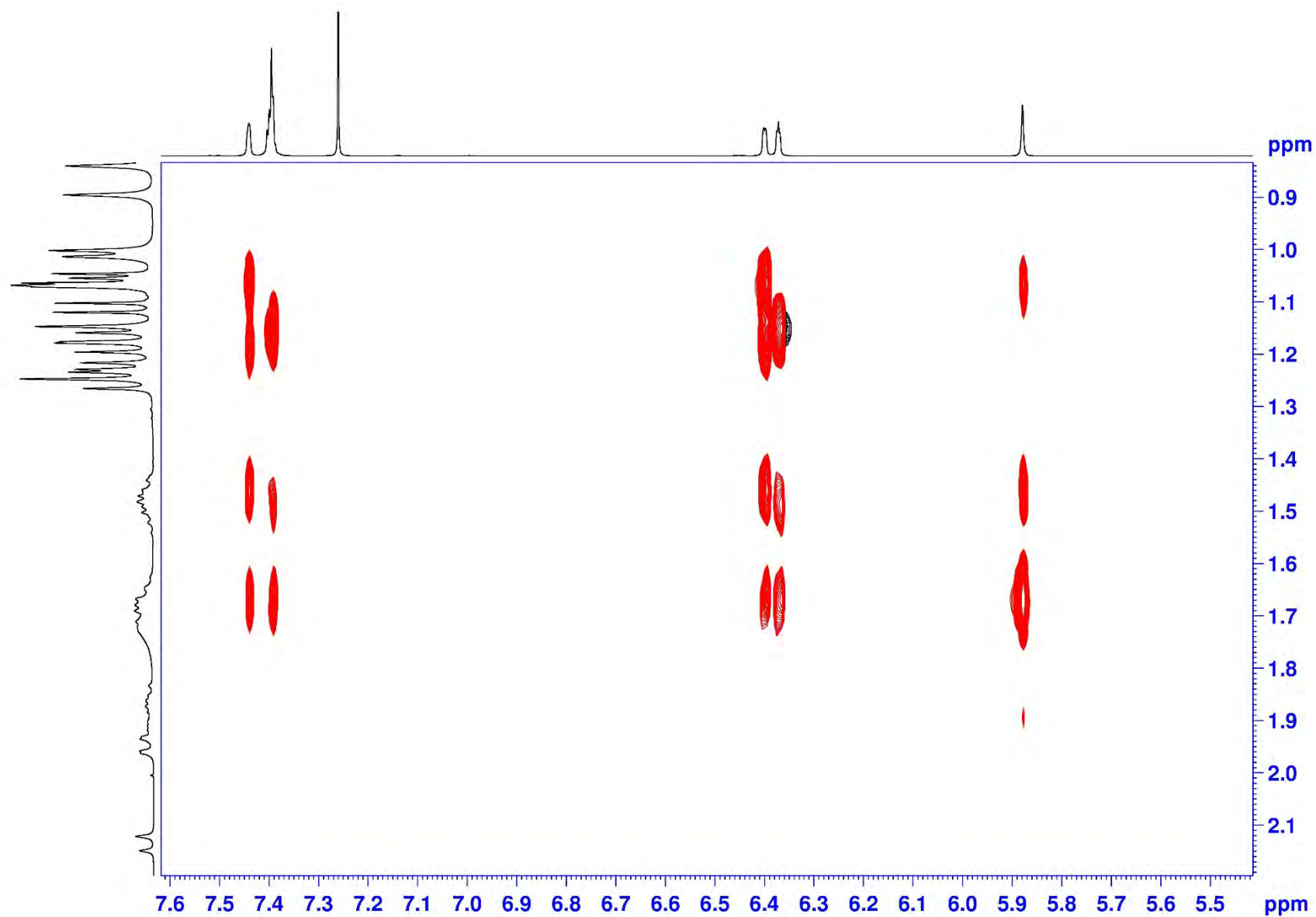
NOESY (400 MHz) spectrum of compound **3'** in CDCl₃



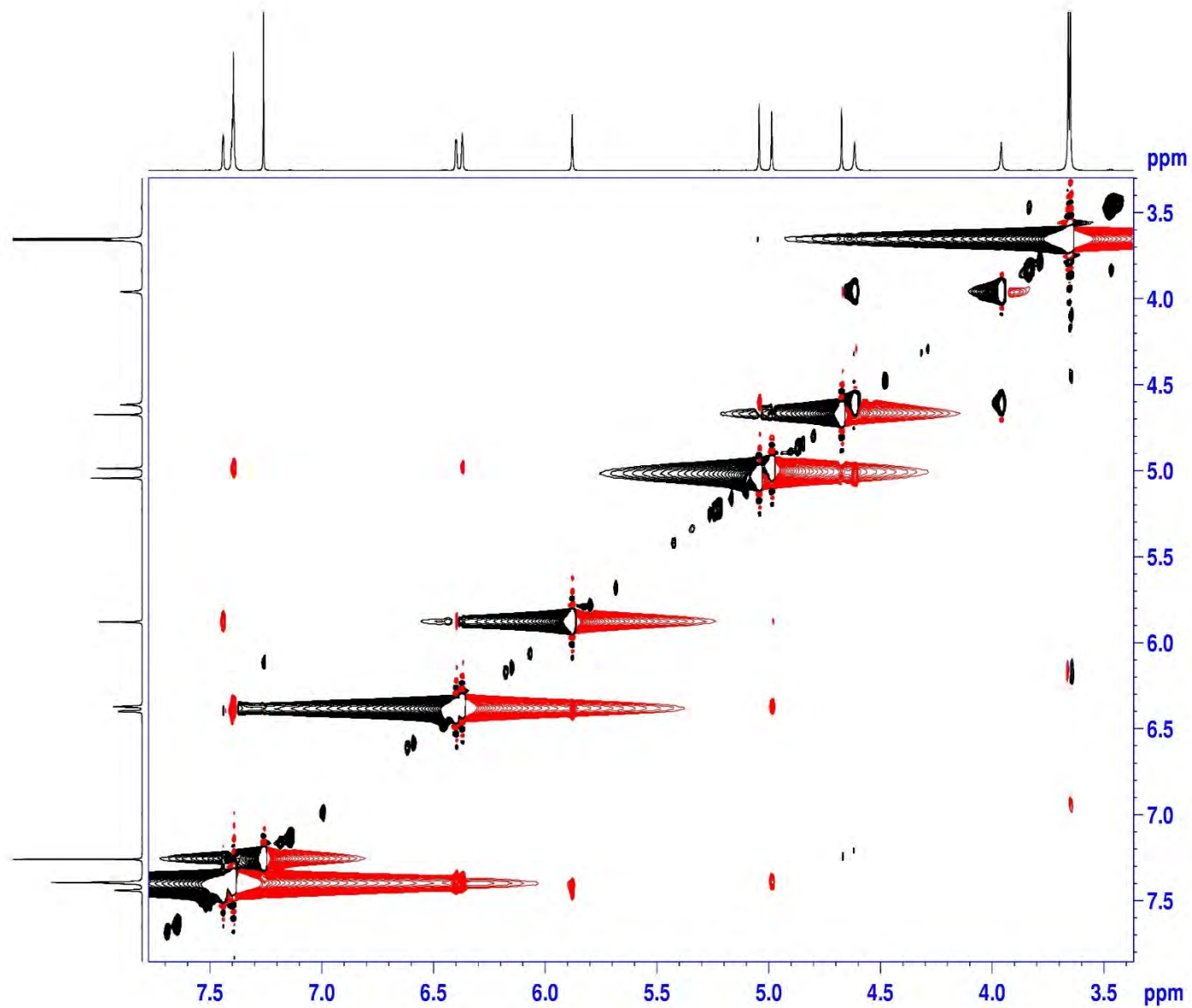
NOESY (400 MHz) spectrum of compound **3'** in CDCl₃



NOESY (400 MHz) spectrum of compound **3'** in CDCl₃



NOESY (400 MHz) spectrum of compound **3'** in CDCl₃



HR-ESIMS for compound 4

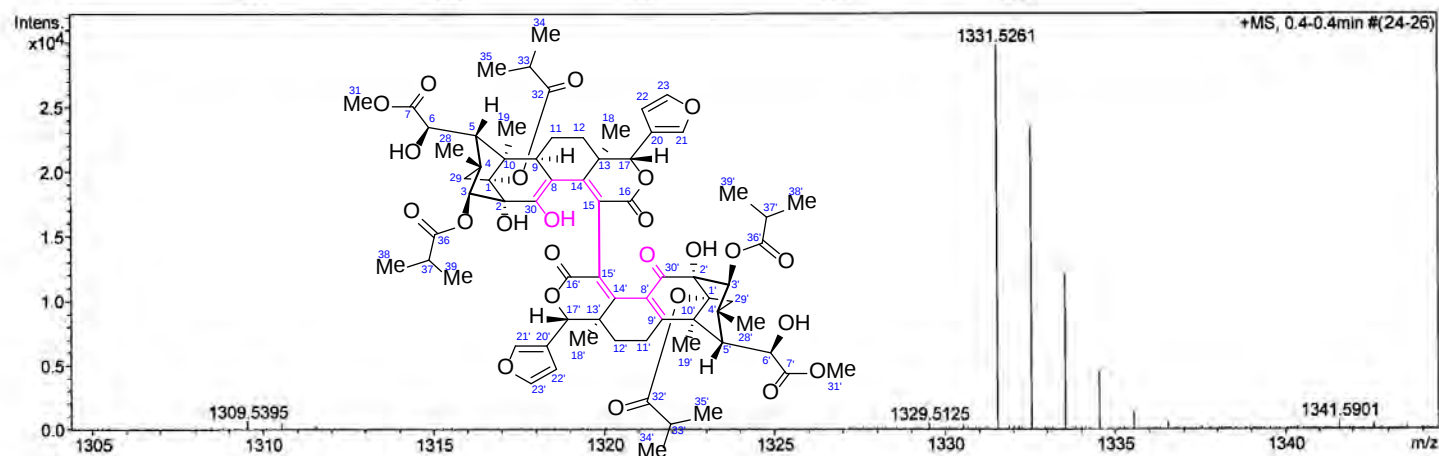
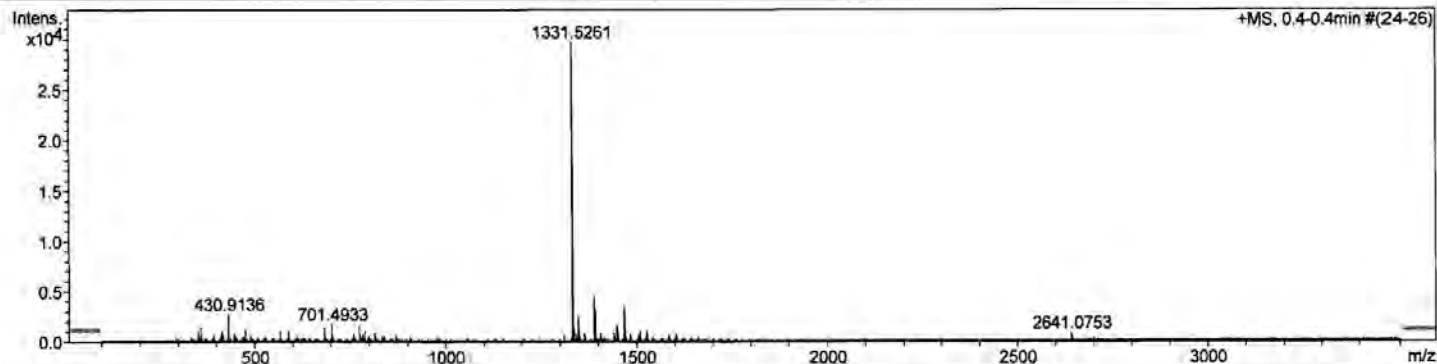
Generic Display Report

Analysis Info

Analysis Name D:\Data\MS\data\201509\liwanshan_lws-42-6_pos_7_01_350.d
Method LC_Direct Infusion_pos_100-3000mz.m
Sample Name liwanshan_lws-42-6_pos
Comment

Acquisition Date 9/8/2015 12:03:01 AM

Operator SCSIO
Instrument maXis



HR-ESIMS for compound 4

Mass Spectrum SmartFormula Report

Analysis Info

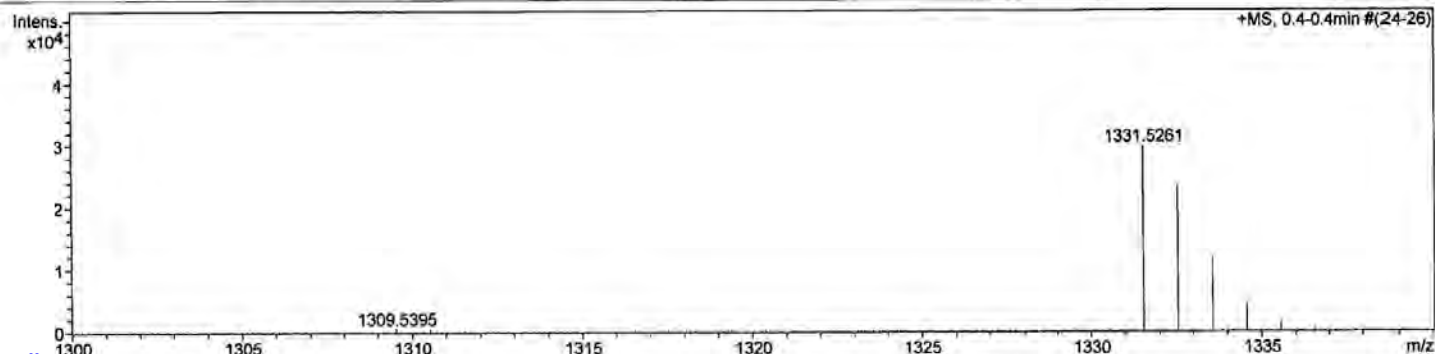
Analysis Name D:\Data\MS\data\201509\liwanshan_lws-42-6_pos_7_01_350.d
 Method LC_Direct Infusion_pos_100-3000mz.m
 Sample Name liwanshan_lws-42-6_pos
 Comment

Acquisition Date 9/8/2015 12:03:01 AM

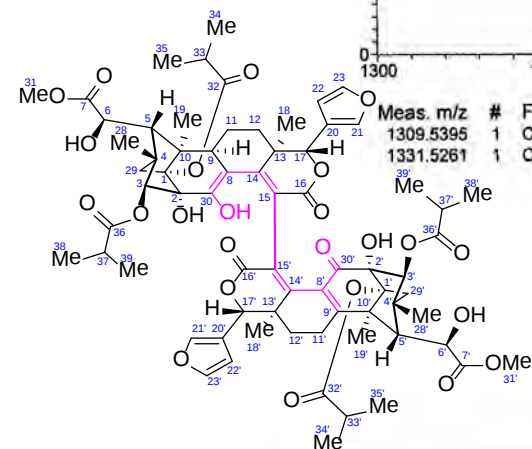
Operator SCSIO
 Instrument / Ser# maXis 29

Acquisition Parameter

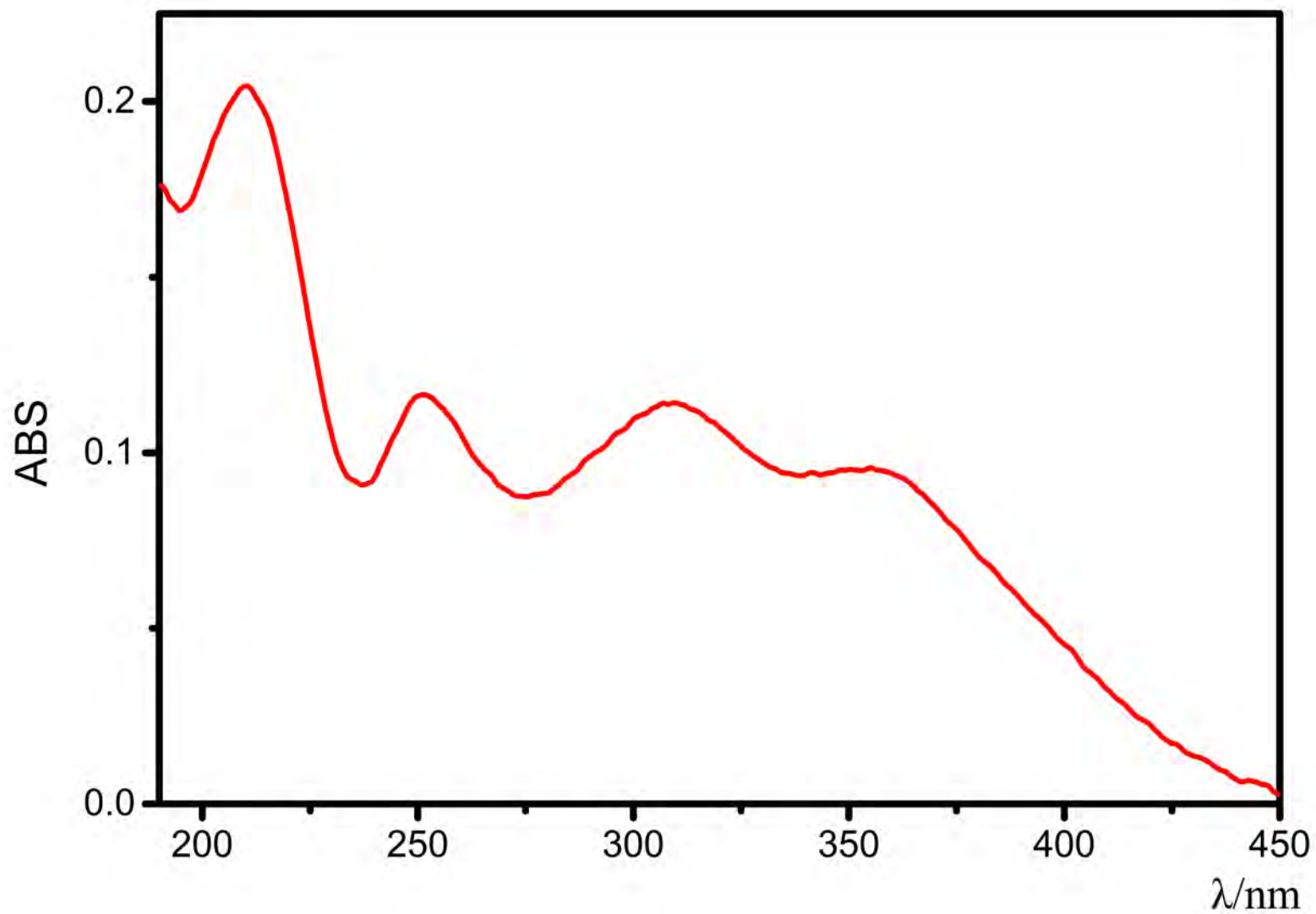
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3500 m/z	Set Collision Cell RF	1800.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e ⁻ Conf	N-Rule
1309.5395	1	C 70 H 85 O 24	17.36	1309.5425	2.3	3.0	78.7	28.5	even	ok
1331.5261	1	C 70 H 84 Na O 24	49.45	1331.5245	-1.2	-1.6	31.7	28.5	even	ok

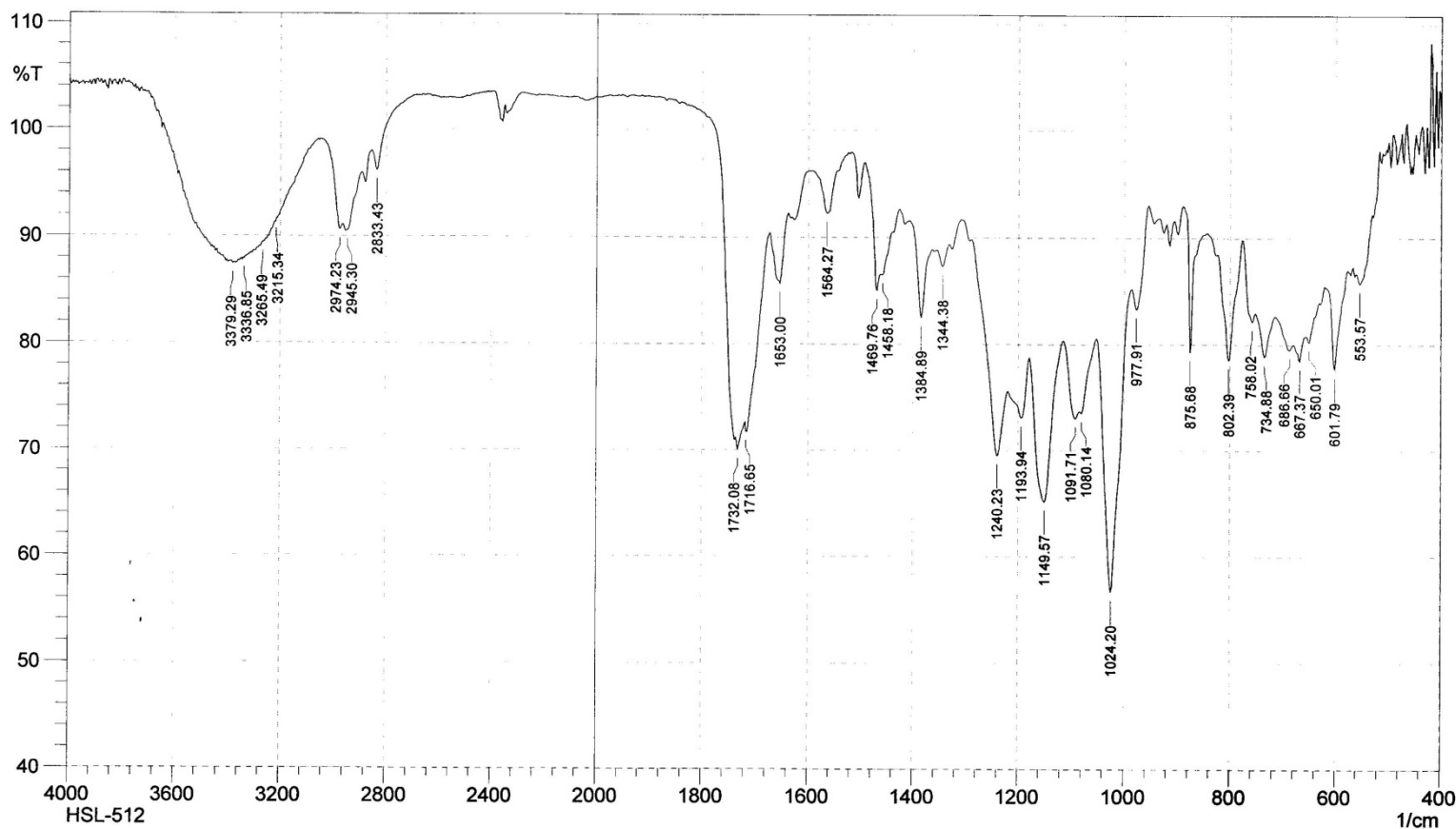


UV spectrum for compound **4** in CH₃CN



IR spectrum for compound 4

SHIMADZU

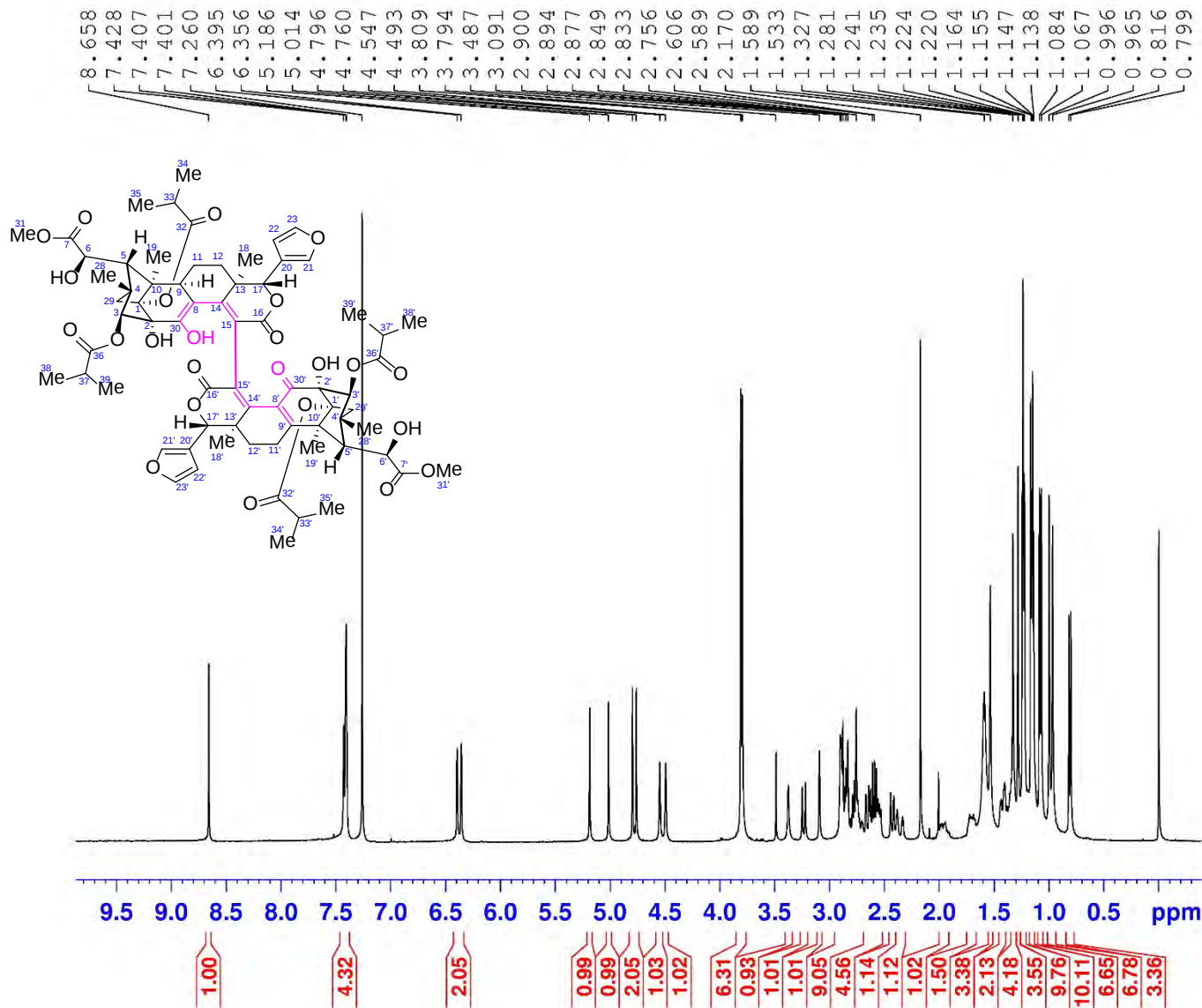


Sample name: 42-61 (4)
F:\liwanshan\20180226\42-
61.smf

Scan number: 25
Resolution: 4 [1/cm]

Data: 2018-2-26 15:02:48
Operator: SCSIO

^1H NMR (400 MHz) spectrum of compound **4** in CDCl_3



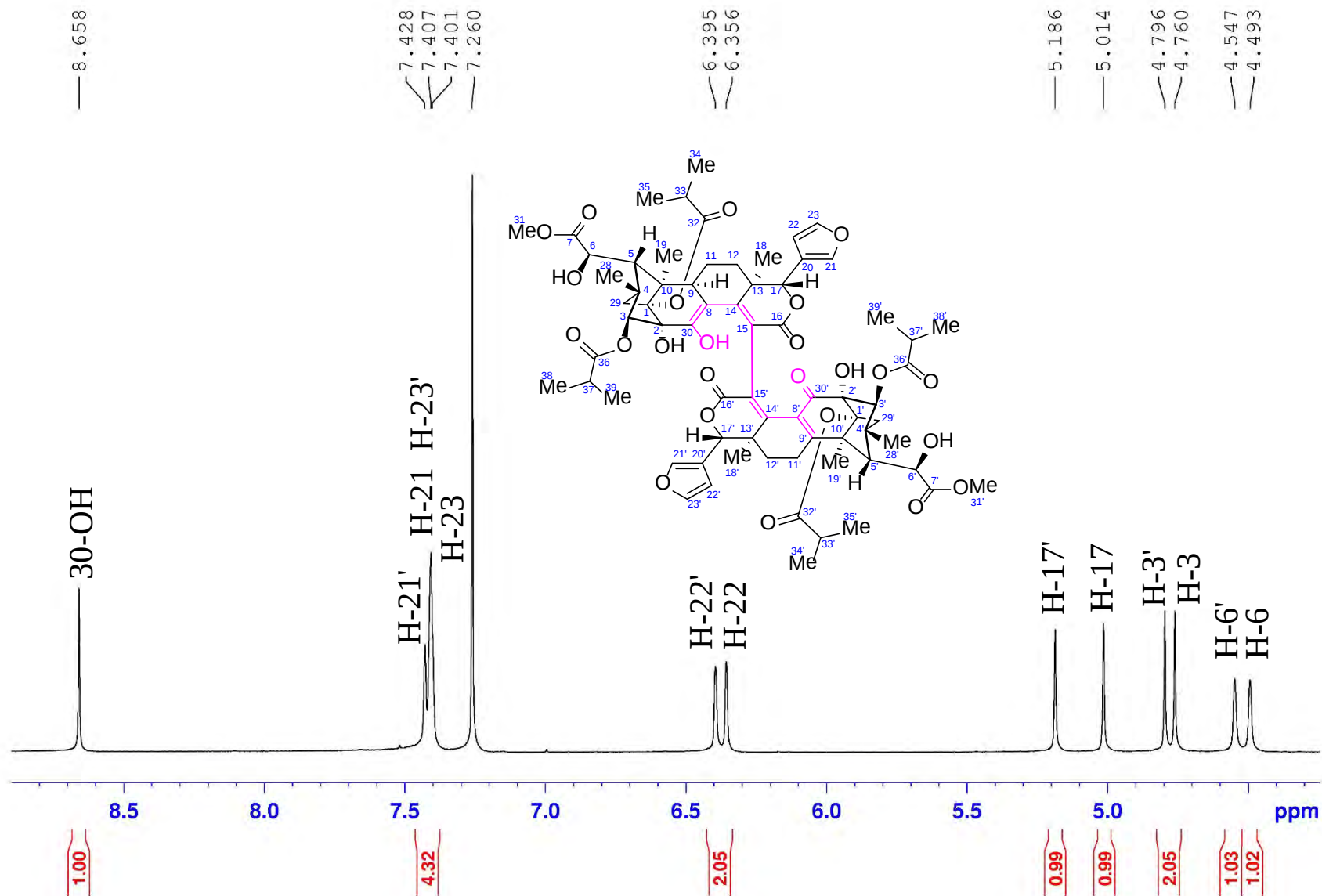
```

NAME      lws-42-6-fd
EXPNO     1
PROCNO    1
Date_     20150815
Time      15.50
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         171.57
DW         60.800 usec
DE         10.00 usec
TE         297.0 K
D1         1.00000000 sec
TD0        1
    
```

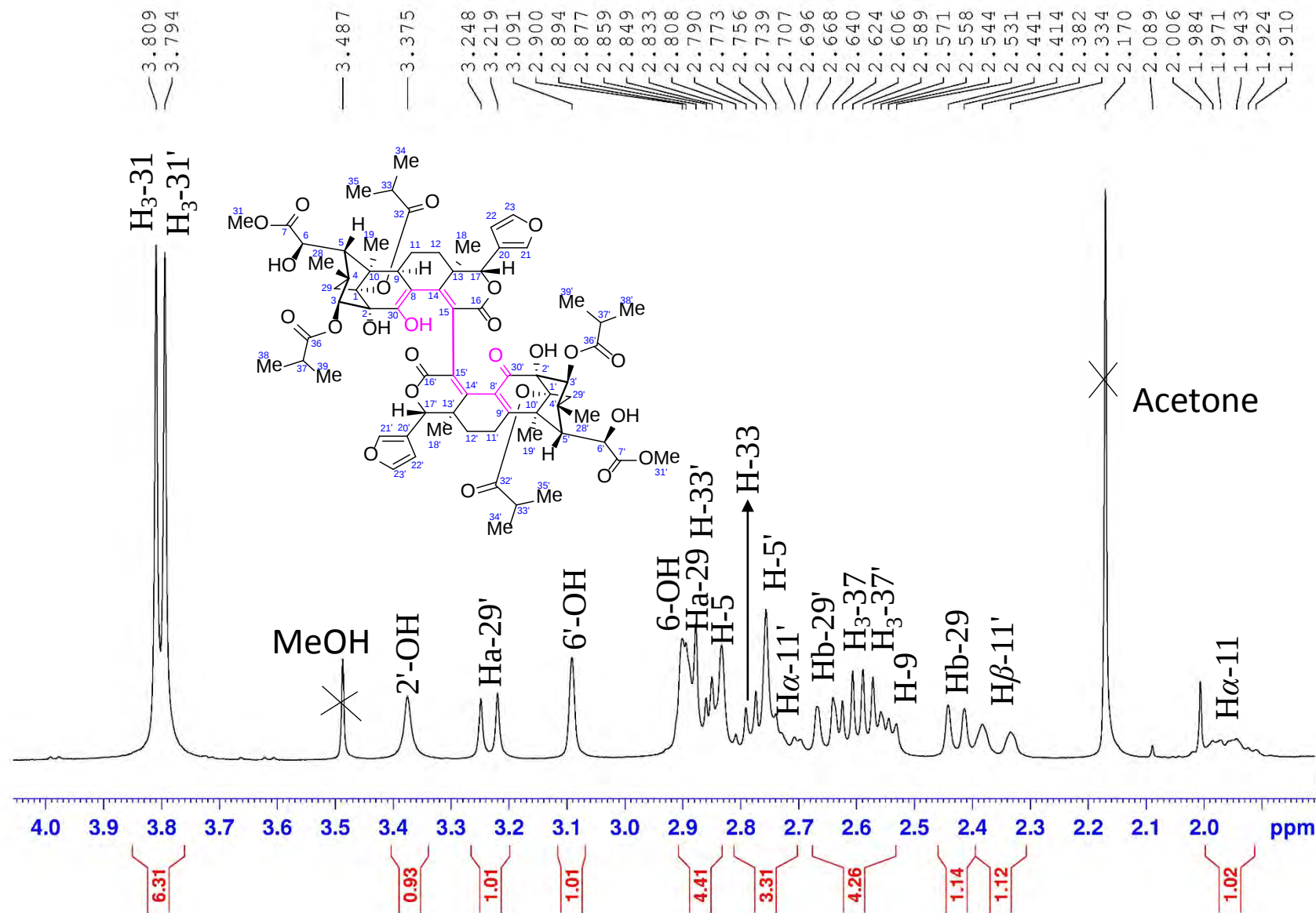
```

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1      1H
P1        12.00 usec
SI        65536
SF        400.1300095 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

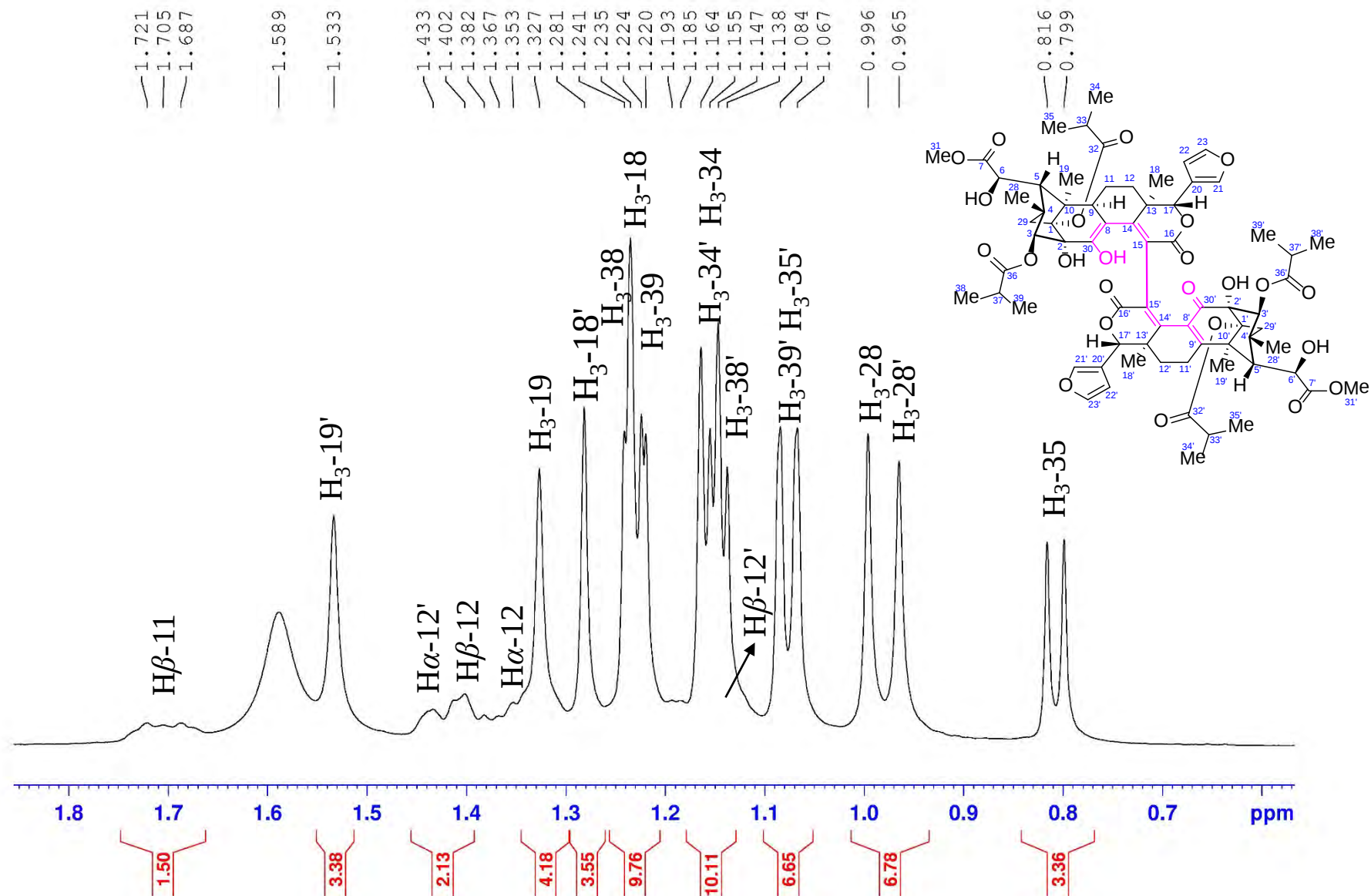
^1H NMR (400 MHz) spectrum of compound **4** in CDCl_3



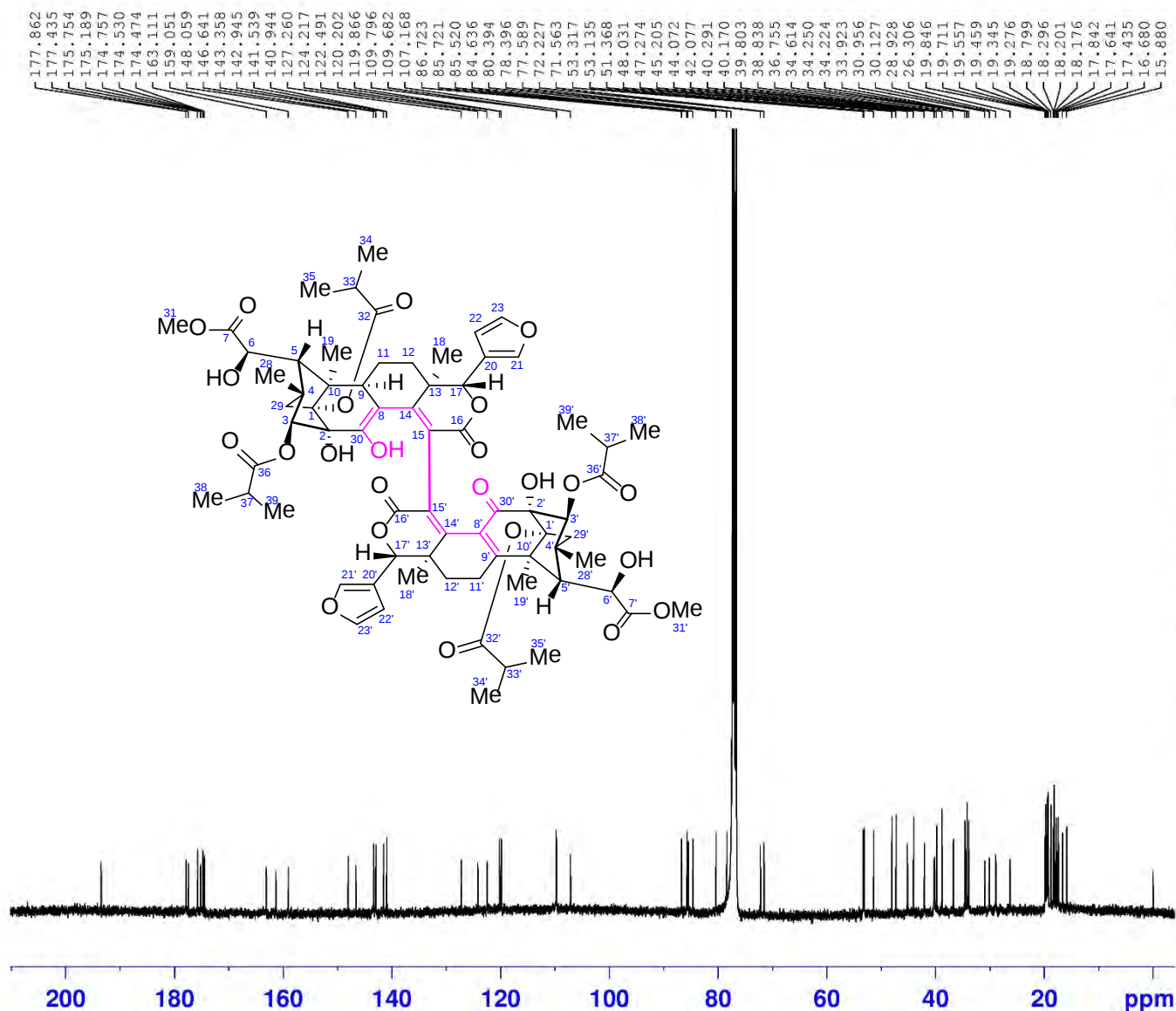
¹H NMR (400 MHz) spectrum of compound **4** in CDCl₃



^1H NMR (400 MHz) spectrum of compound **4** in CDCl_3



^{13}C NMR (100 MHz) spectrum of compound **4** in CDCl_3



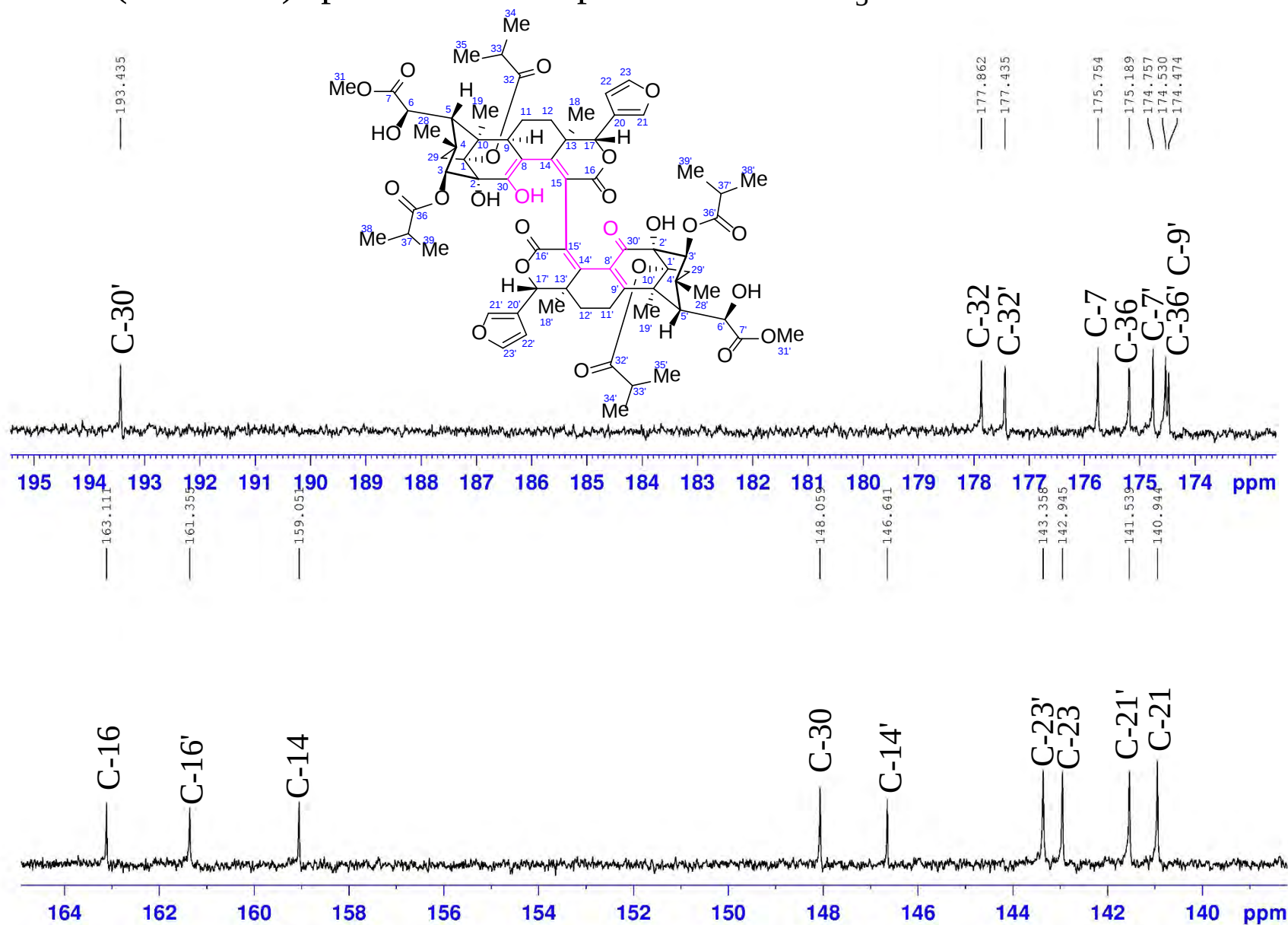
```

NAME      lws-42-6-fd
EXPNO     2
PROCNO    1
Date_     20150815
Time      21.54
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        6000
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        147.94
DW        20.800 usec
DE        18.00 usec
TE        297.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

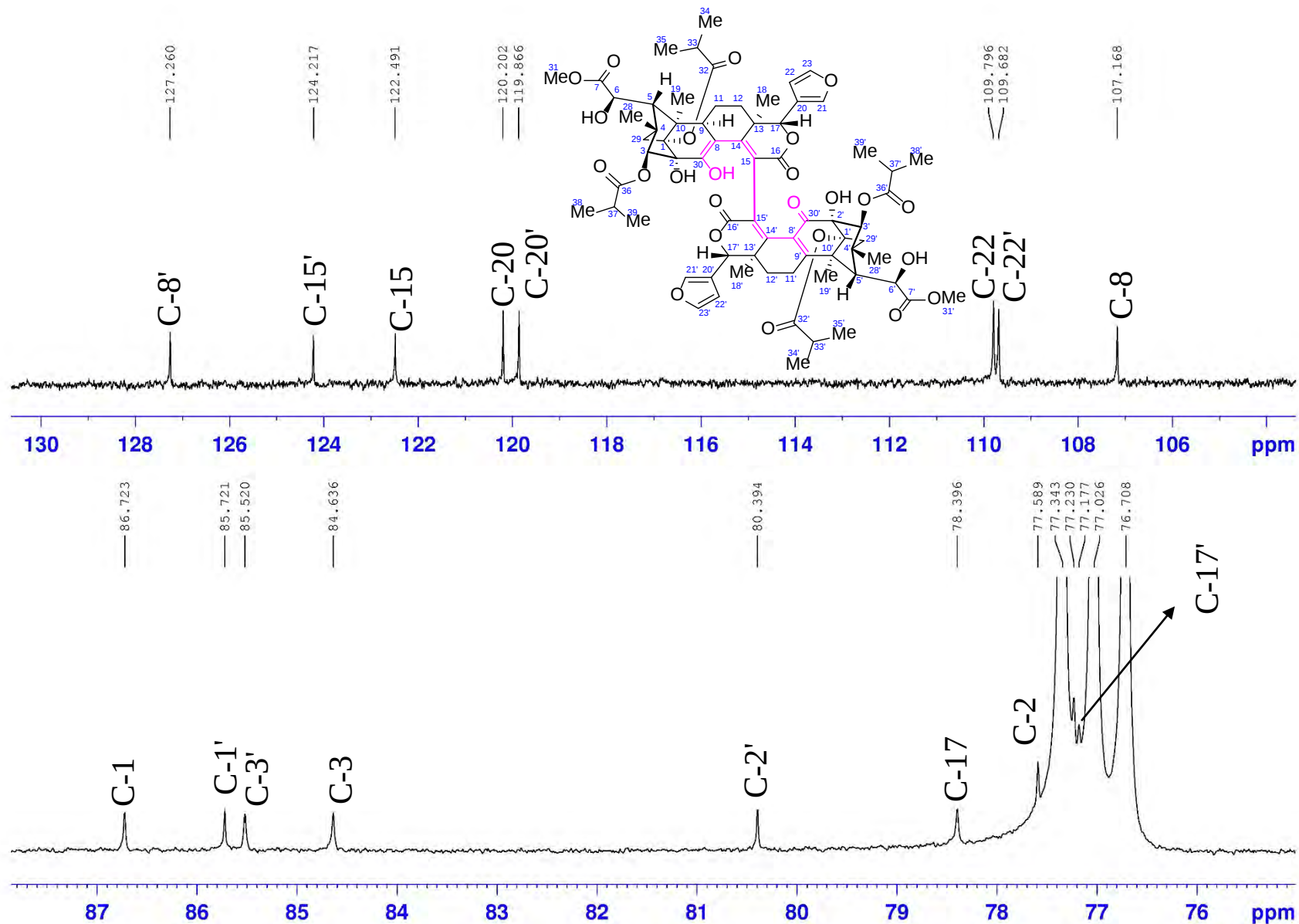
```

===== CHANNEL f1 =====
SF01    100.6233324 MHz
NUC1     13C
P1       10.00 usec
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

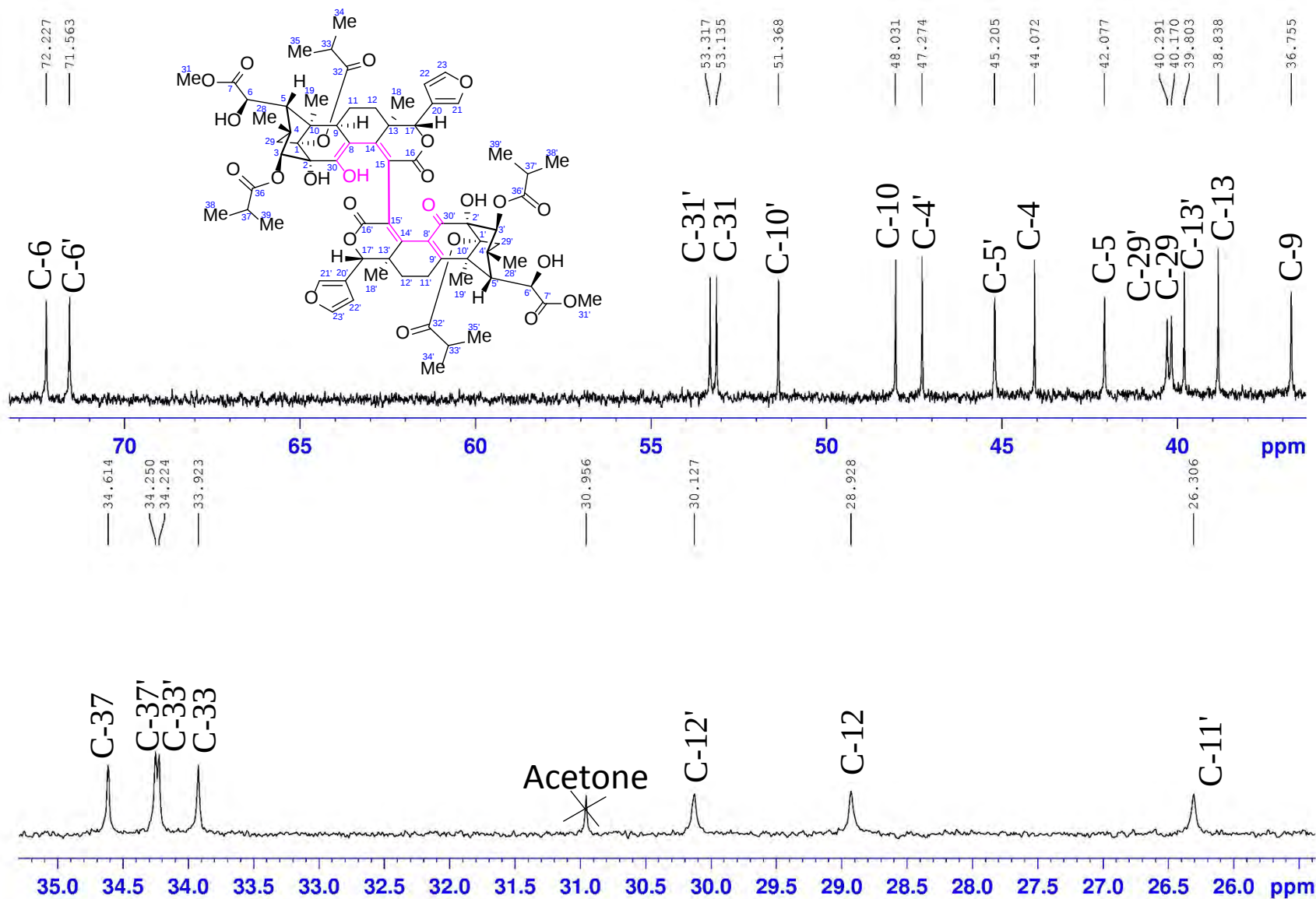
^{13}C NMR (100 MHz) spectrum of compound **4** in CDCl_3



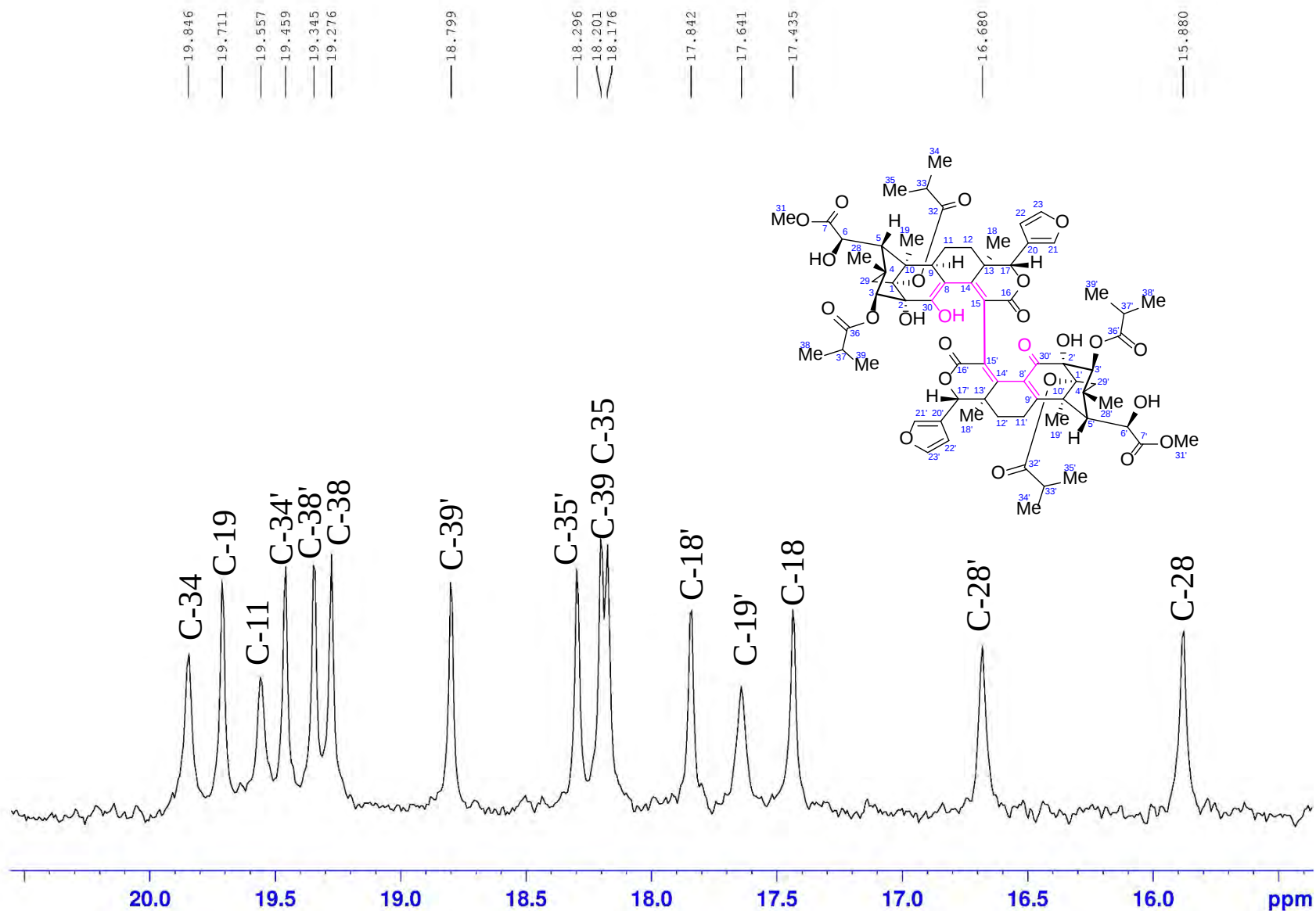
^{13}C NMR (100 MHz) spectrum of compound **4** in CDCl_3



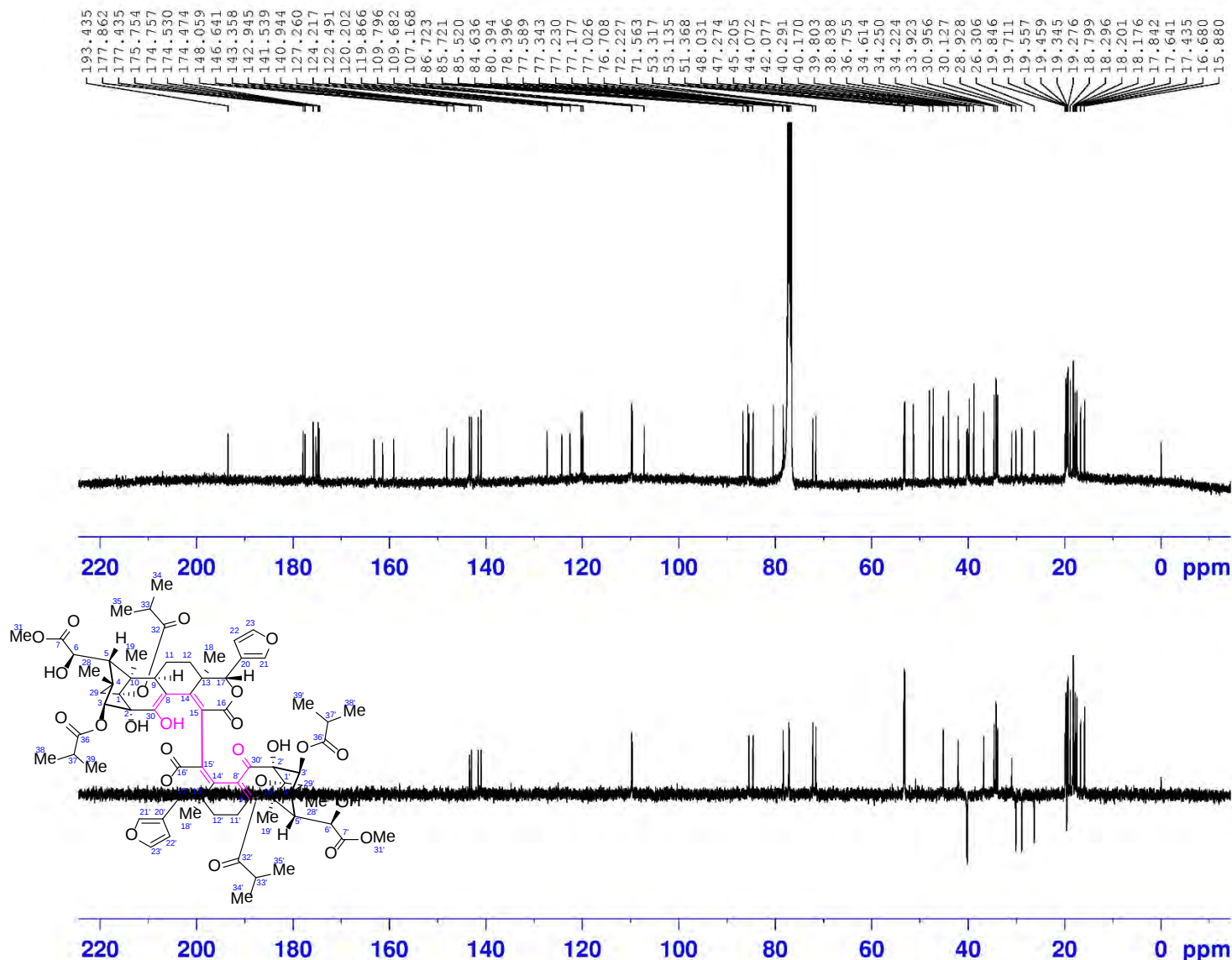
^{13}C NMR (100 MHz) spectrum of compound **4** in CDCl_3



^{13}C NMR (100 MHz) spectrum of compound **4** in CDCl_3



DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



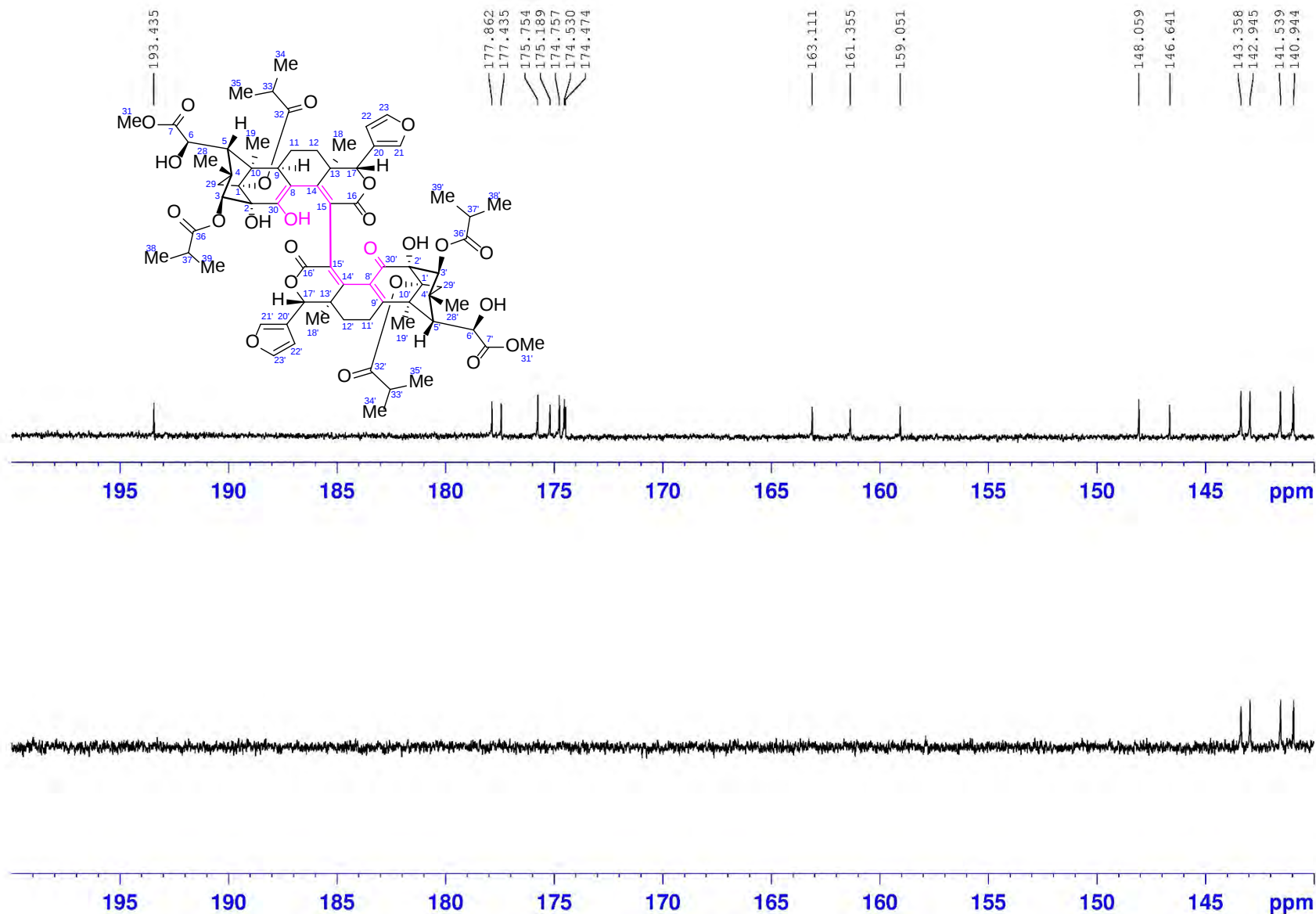
```

NAME          lws-42-6-fd
EXPNO          3
PROCNO         1
Date_          20150815
Time           23.21
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        deptsp135
TD             65536
SOLVENT        CDCl3
NS             1500
DS             4
SWH            24038.461
FIDRES         0.366798
AQ             1.3631988
RG             130.26
DW             20.800
DE             18.00
TE             297.0
CNST2          145.0000000
D1             2.00000000
D2             0.00344828
D12            0.00002000
TD0            1
    
```

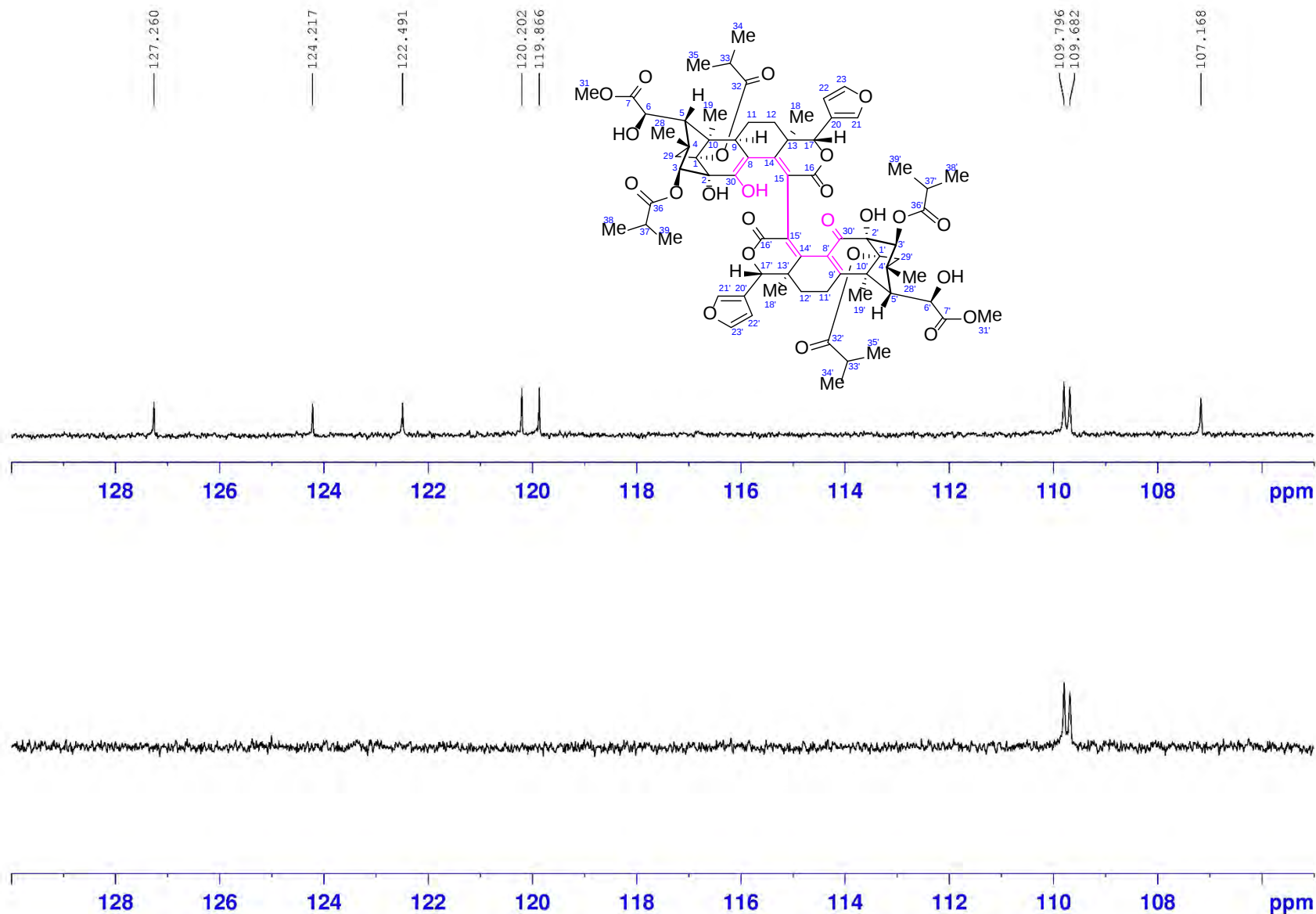
```

===== CHANNEL f1 =====
SFO1          100.6233324
NUC1           13C
P1             10.00
P13            2000.00
SI             32768
SF            100.6127685
WDW            EM
SSB            0
LB             1.00
GB             0
PC             1.40
    
```

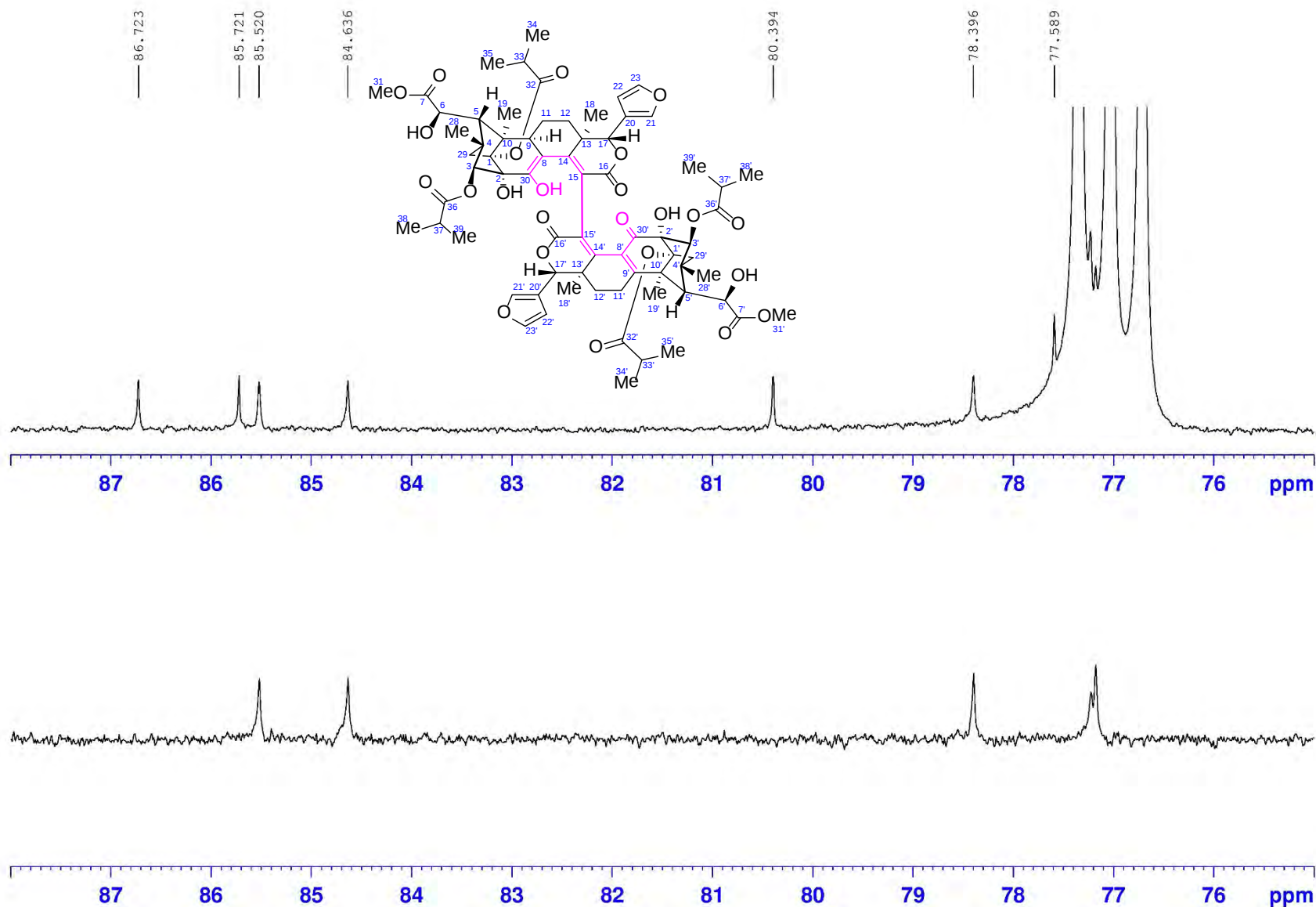
DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



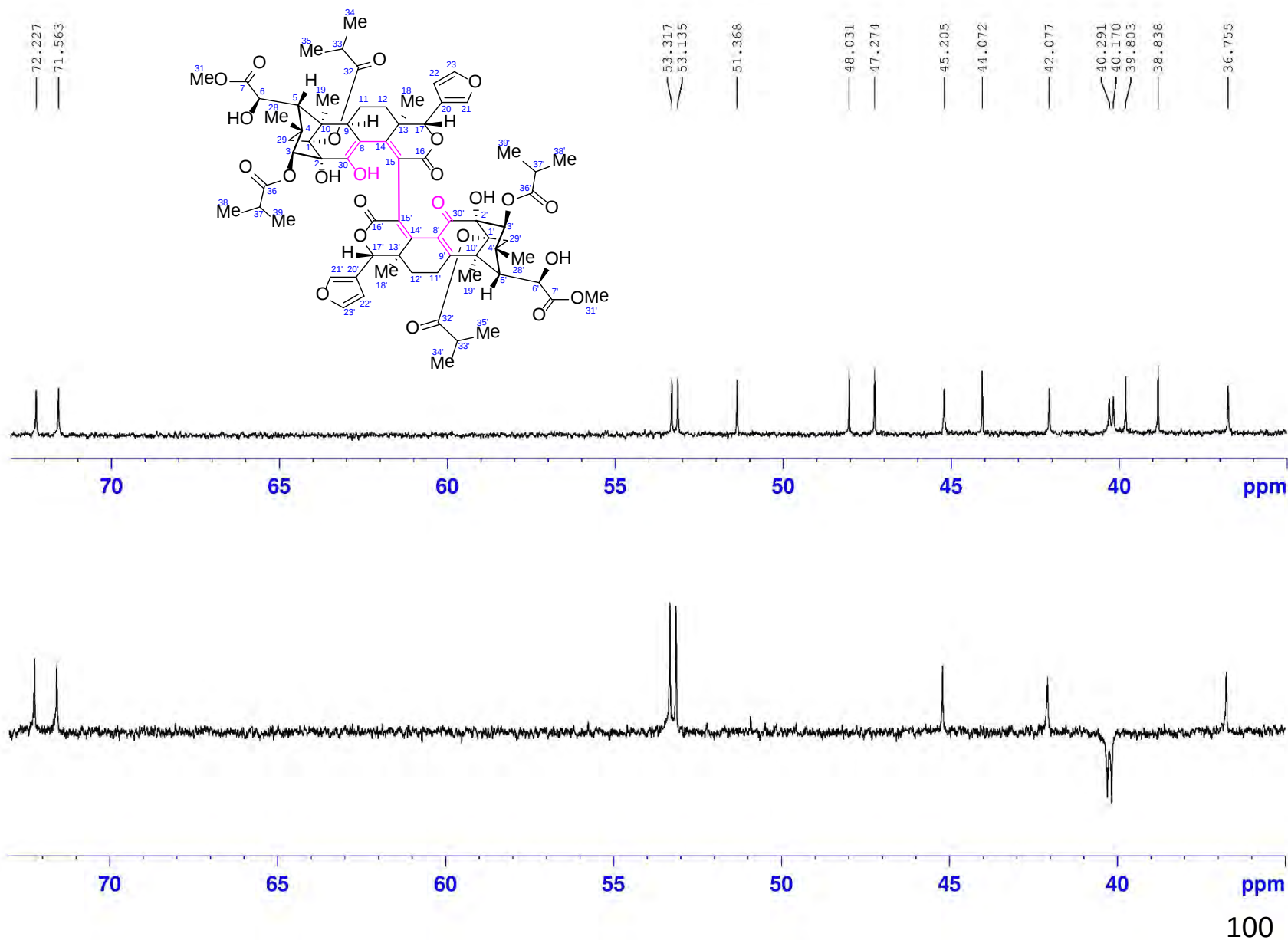
DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



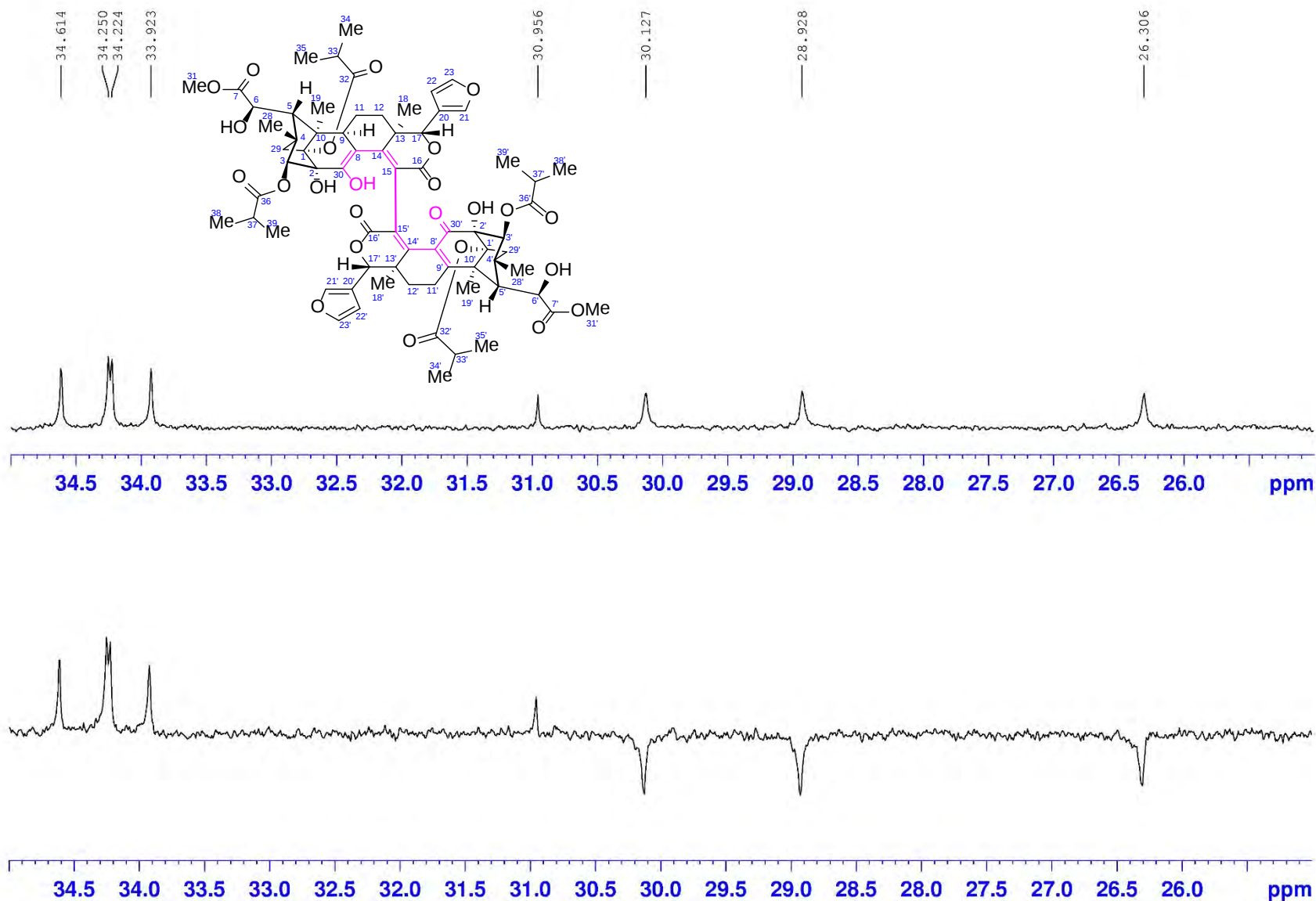
DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



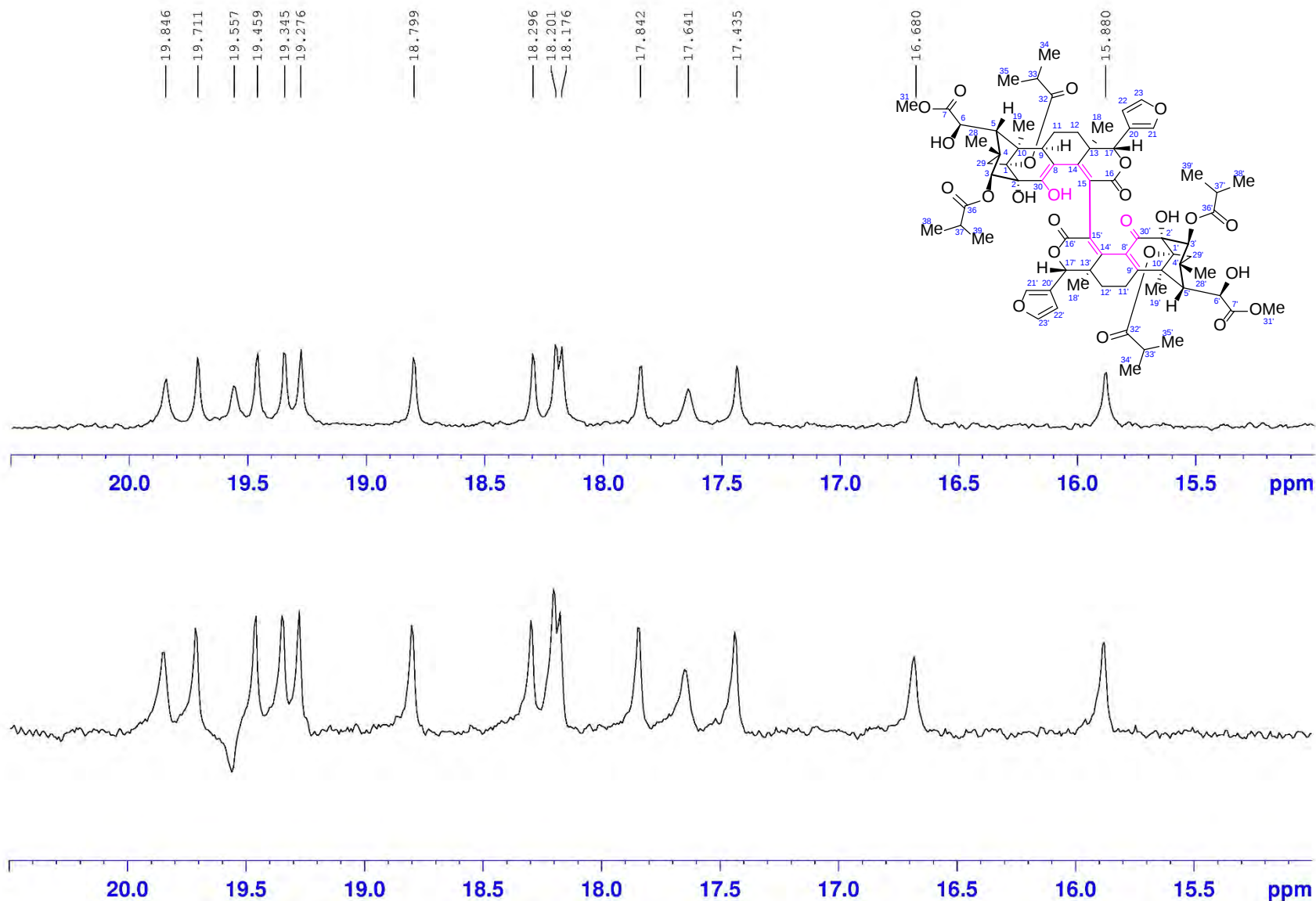
DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



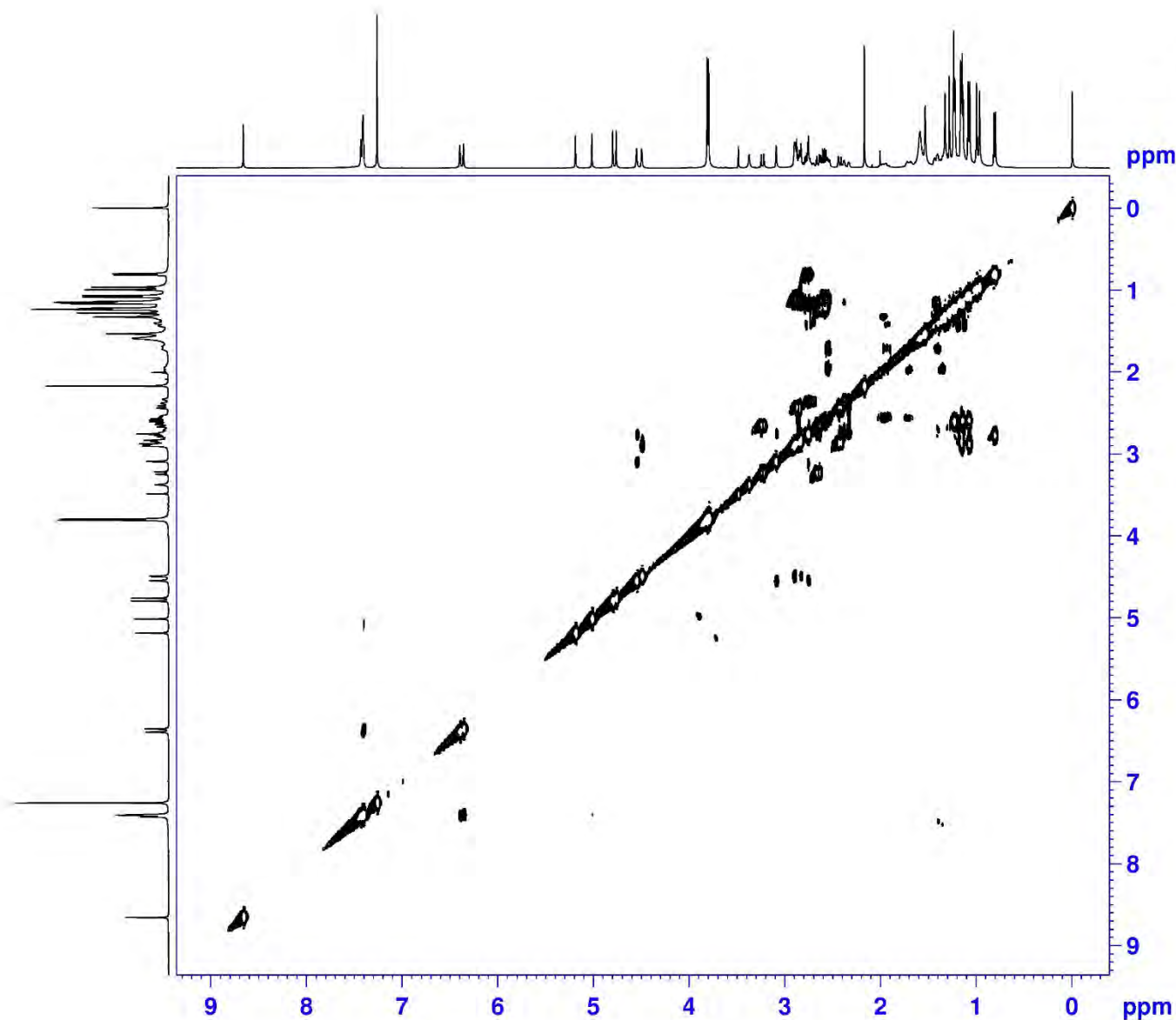
DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



^1H - ^1H COSY (400 MHz) spectrum of compound **4** in CDCl_3



```
NAME          lws-42-6-fd
EXPNO          4
PROCNO         1
Date_          20150815
Time           23.22
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        cosygpppqf
TD             2048
SOLVENT        CDCl3
NS             16
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 =====
SF01          400.1318006 MH
NUC1           1H
P0            12.00 us
P1            12.00 us
P17           2500.00 us
ND0            1
TD            128
SF01          400.1318 MH
FIDRES        30.517578 Hz
SW            9.762 pf
FnMODE        QF
SI            1024
SF            400.1300096 MH
WDW           QSINE
SSB            0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            400.1300096 MH
wrtw          QSTNR
```

^1H - ^1H COSY (400 MHz) spectrum of compound **4** in CDCl_3

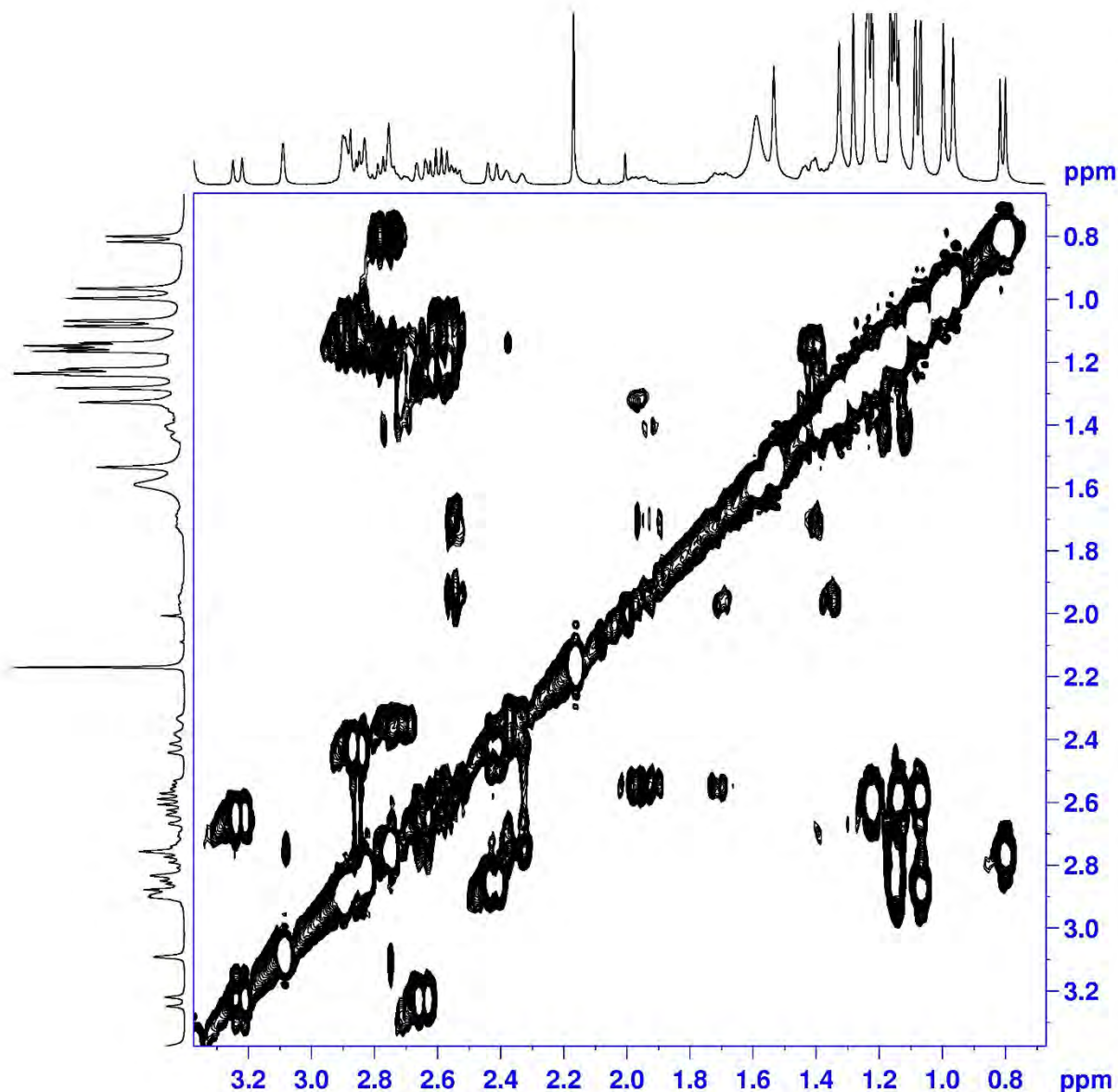


```

NAME      lws-42-6-fd
EXPNO     4
PROCNO    1
Date_     20150815
Time      23.22
INSTRUM   spect
PROBHD    5 mm CPMAS BB
PULPROG   cosygpppqf
TD         2048
SOLVENT   CDCl3
NS         16
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
SSB         0
LB         0.00 Hz
GB         0
  
```

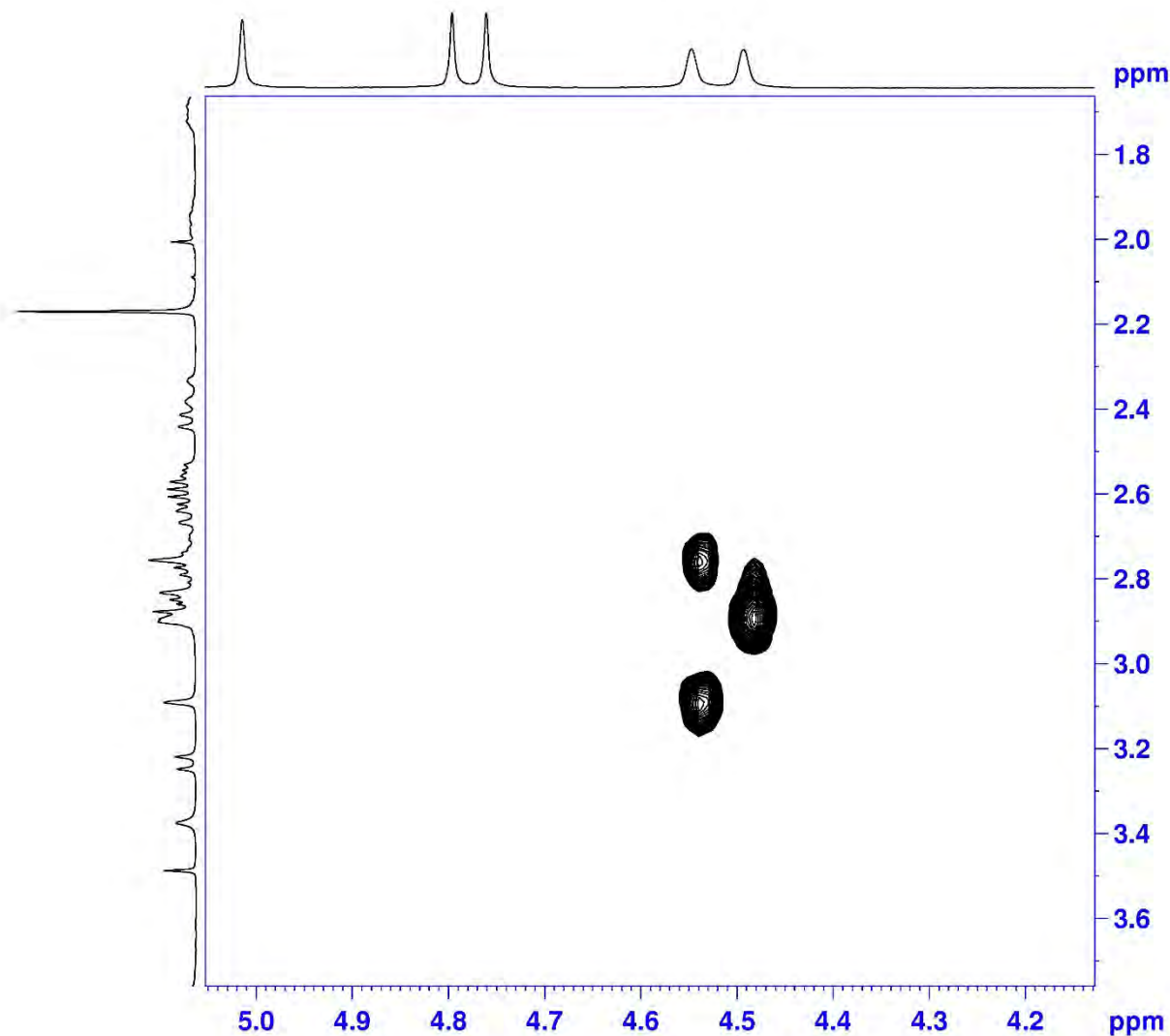


^1H - ^1H COSY (400 MHz) spectrum of compound **4** in CDCl_3

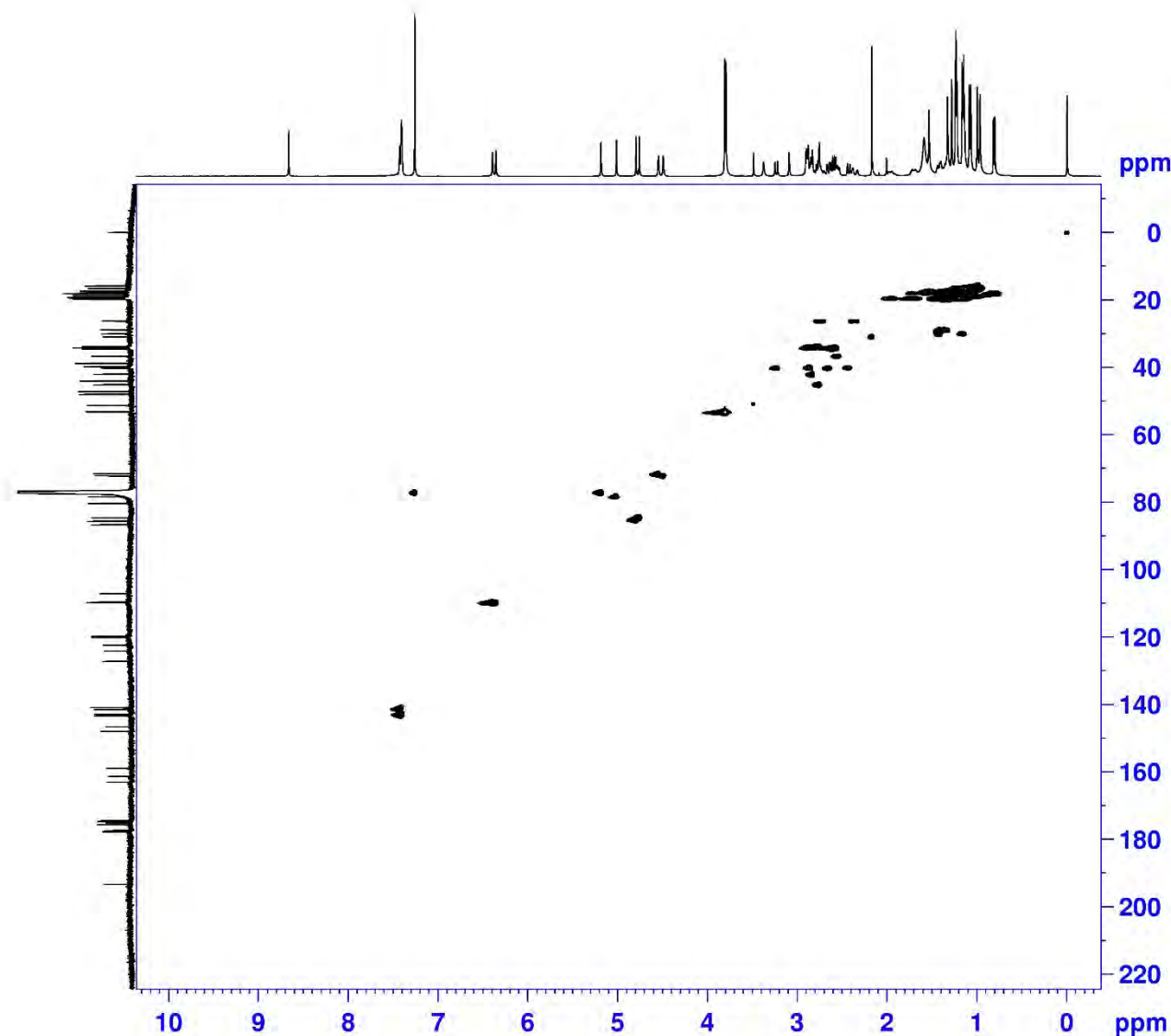


NAME lws-42-6-fd
EXPNO 4
PROCNO 1
Date_ 20150815
Time 23.22
INSTRUM spect
PROBHD 5 mm CPMBO BB
PULPROG cosygpppqf
TD 2048
SOLVENT CDCl_3
NS 16
DS 8
SWH 3906.250 Hz
FIDRES 1.907349 Hz
AQ 0.2621940 sec
RG 208.5
DW 128.000 usec
DE 10.00 usec
TE 297.0 K
D0 0.00000300 sec
D1 1.89678097 sec
D11 0.03000000 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
FMODE 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



HSQC (400 MHz) spectrum of compound **4** in CDCl₃

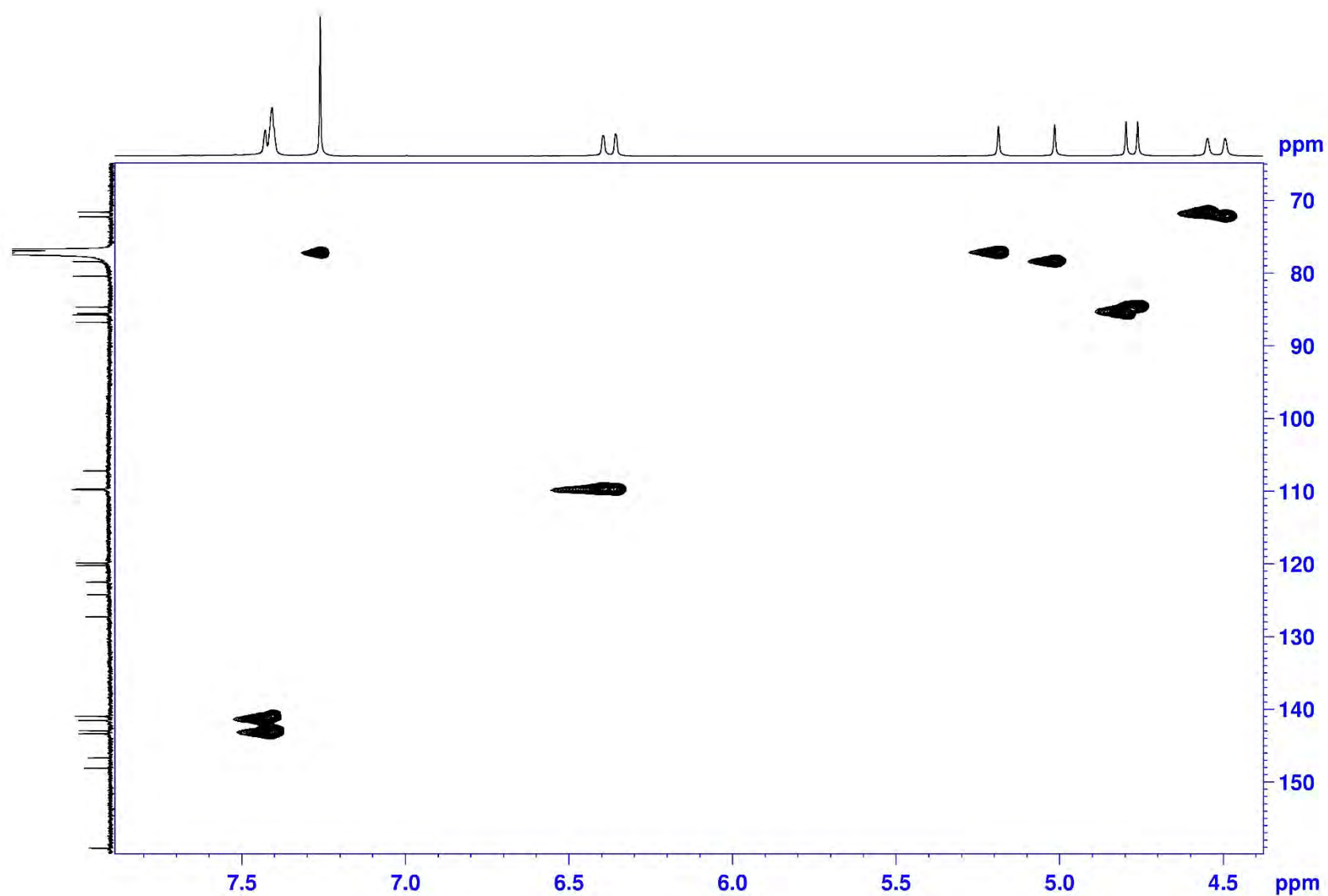


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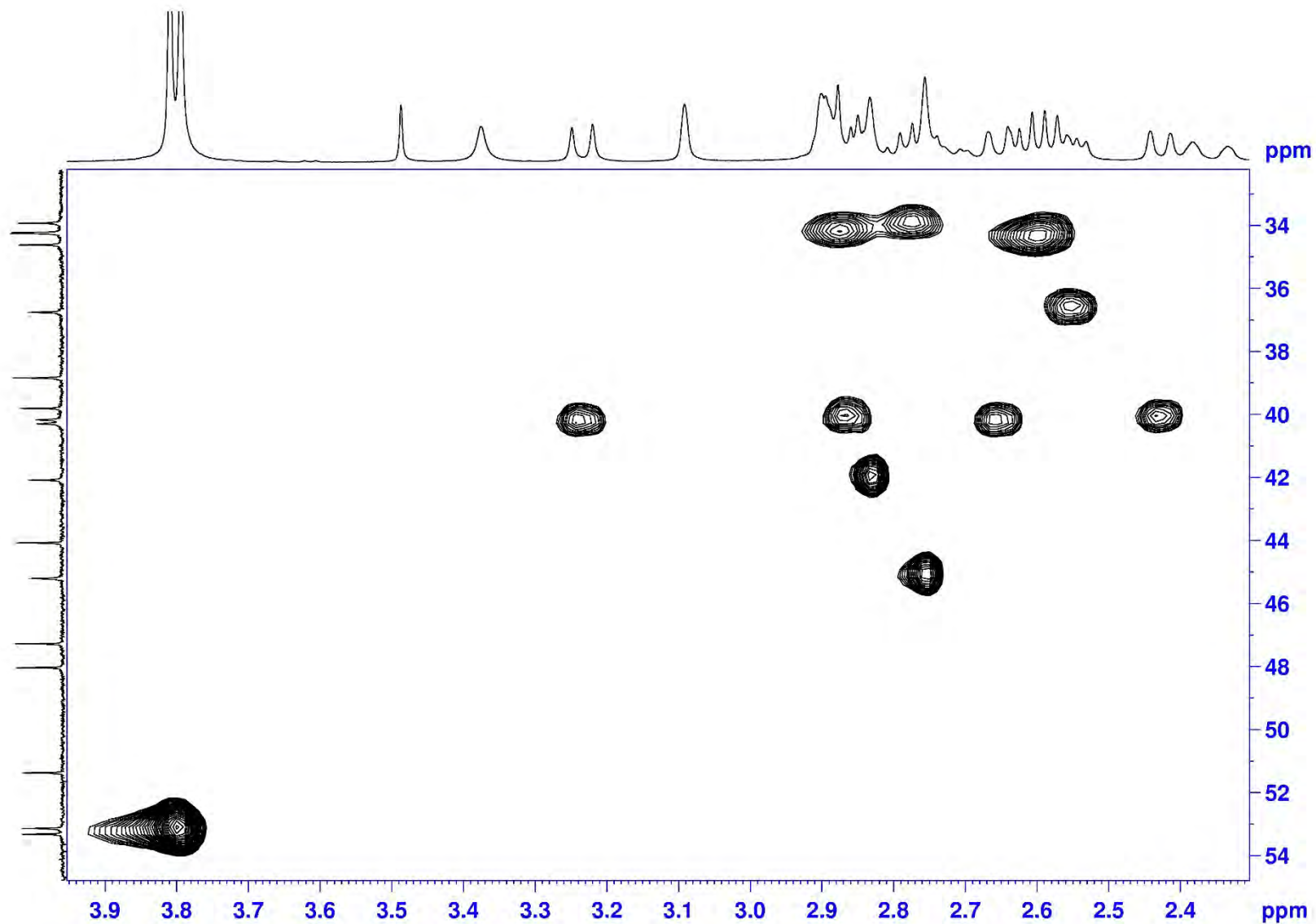
NAME          lws-42-6-fd
EXPNO          5
PROCNO         1
Date_          20150816
Time           0.39
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        hsqcetgpsi2
TD             1024
SOLVENT        CDCl3
NS              32
DS              16
SWH            4302.926 Hz
FIDRES         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 =====
SF01           400.1320007 MHz
NUC1            1H
P1              12.00 usec
P2              24.00 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             400.1300079 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
SSB              ?
    
```

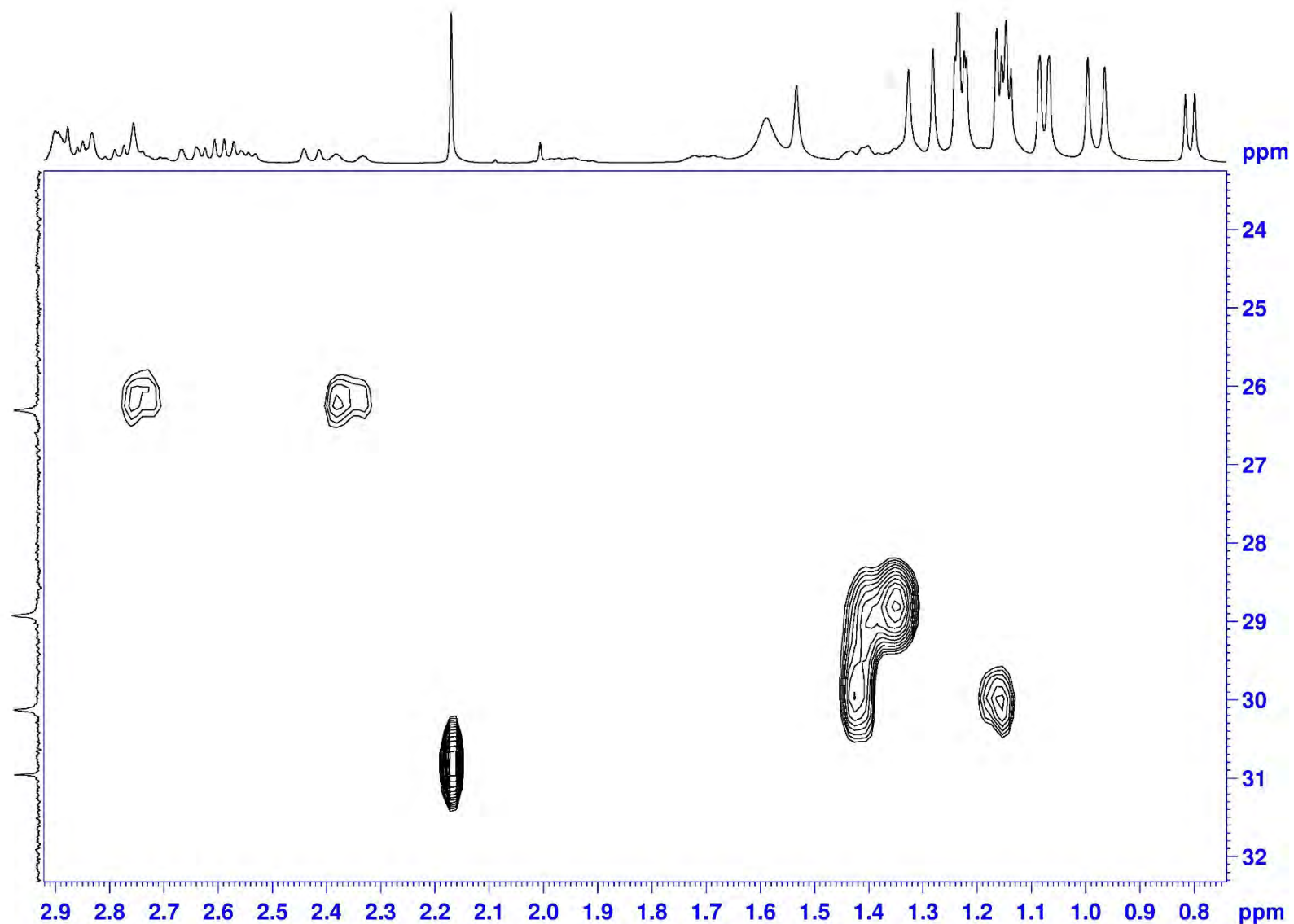
HSQC (400 MHz) spectrum of compound **4** in CDCl₃



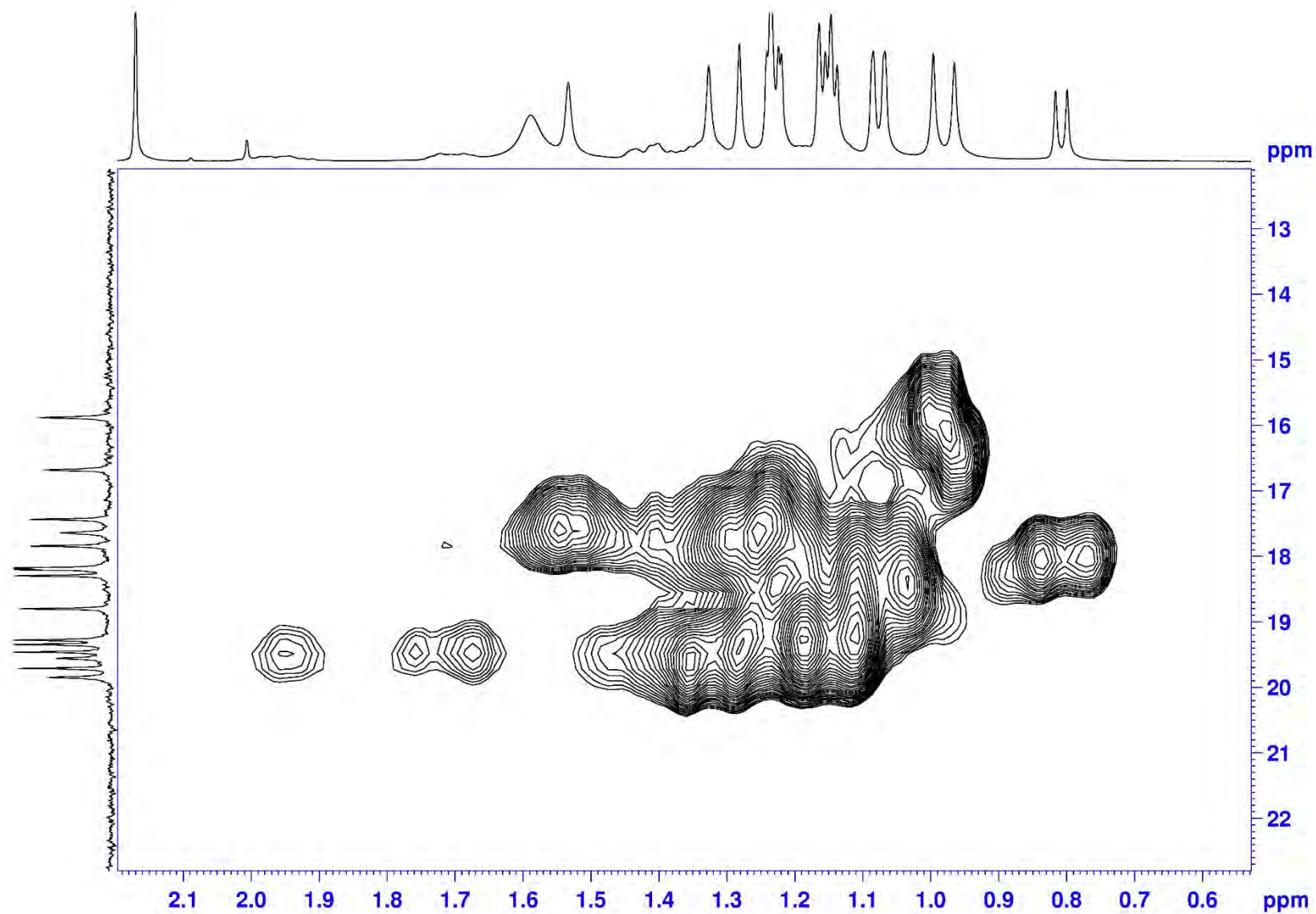
HSQC (400 MHz) spectrum of compound **4** in CDCl₃



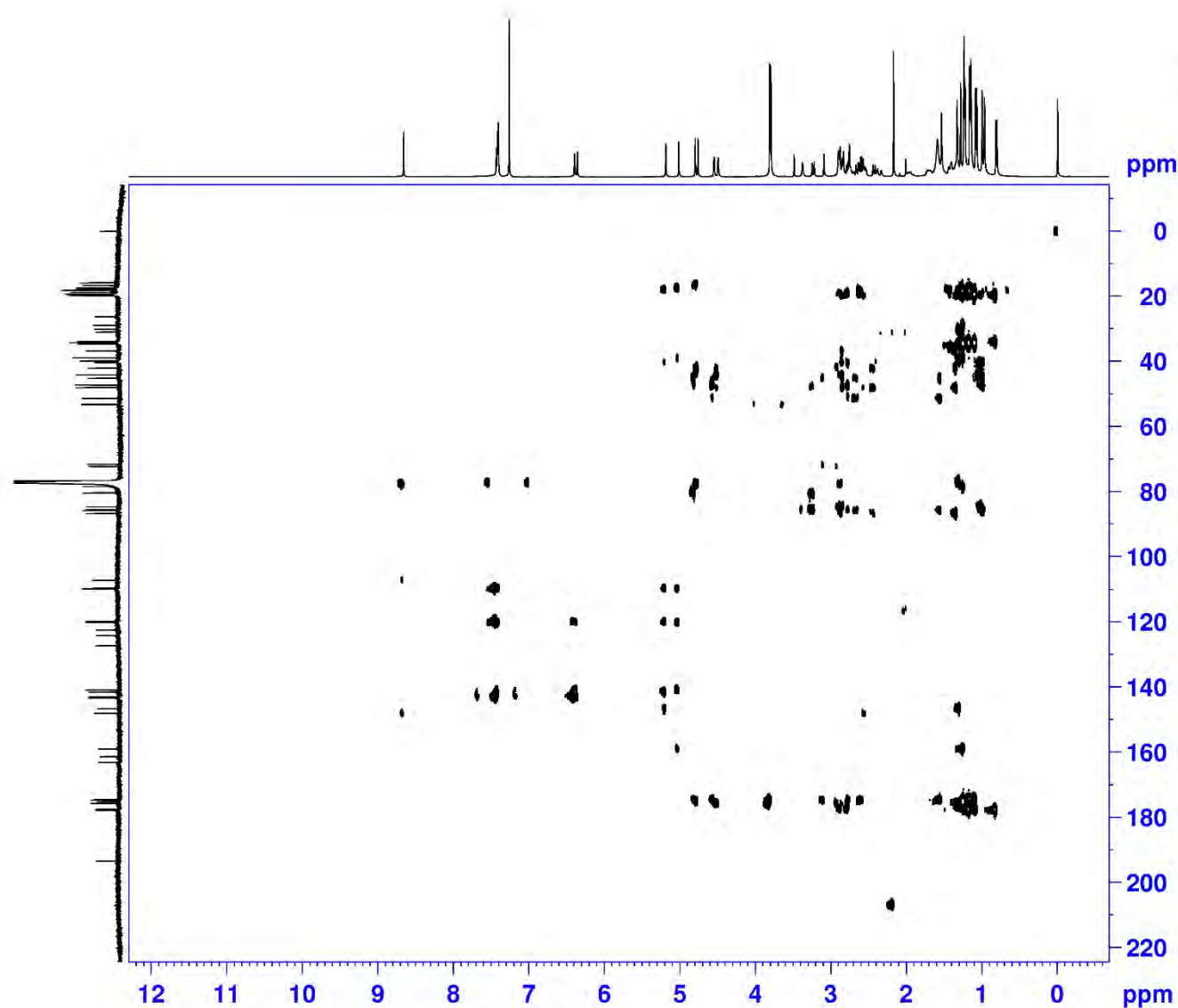
HSQC (400 MHz) spectrum of compound **4** in CDCl₃



HSQC (400 MHz) spectrum of compound **4** in CDCl₃



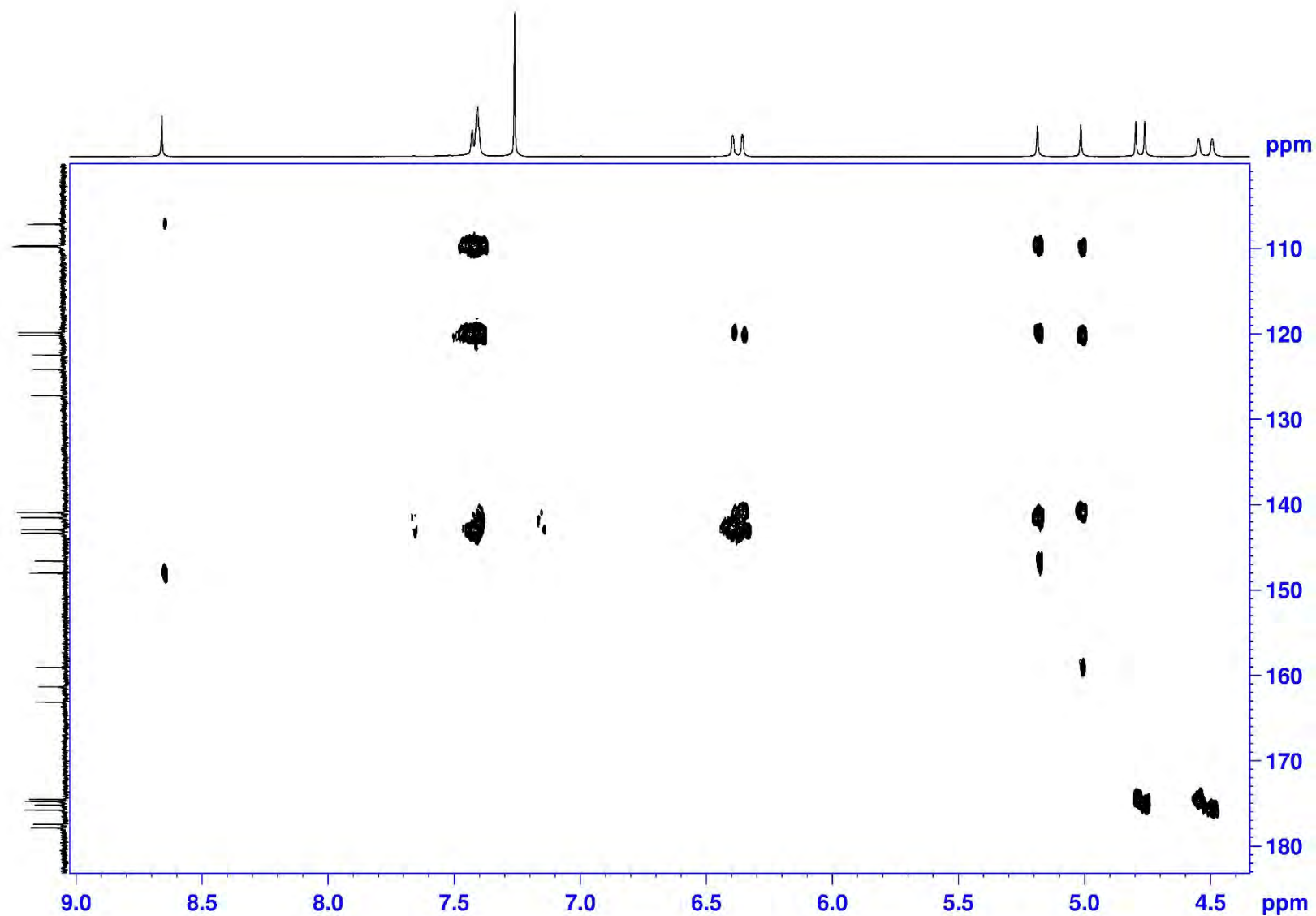
HMBC (400 MHz) spectrum of compound **4** in CDCl₃



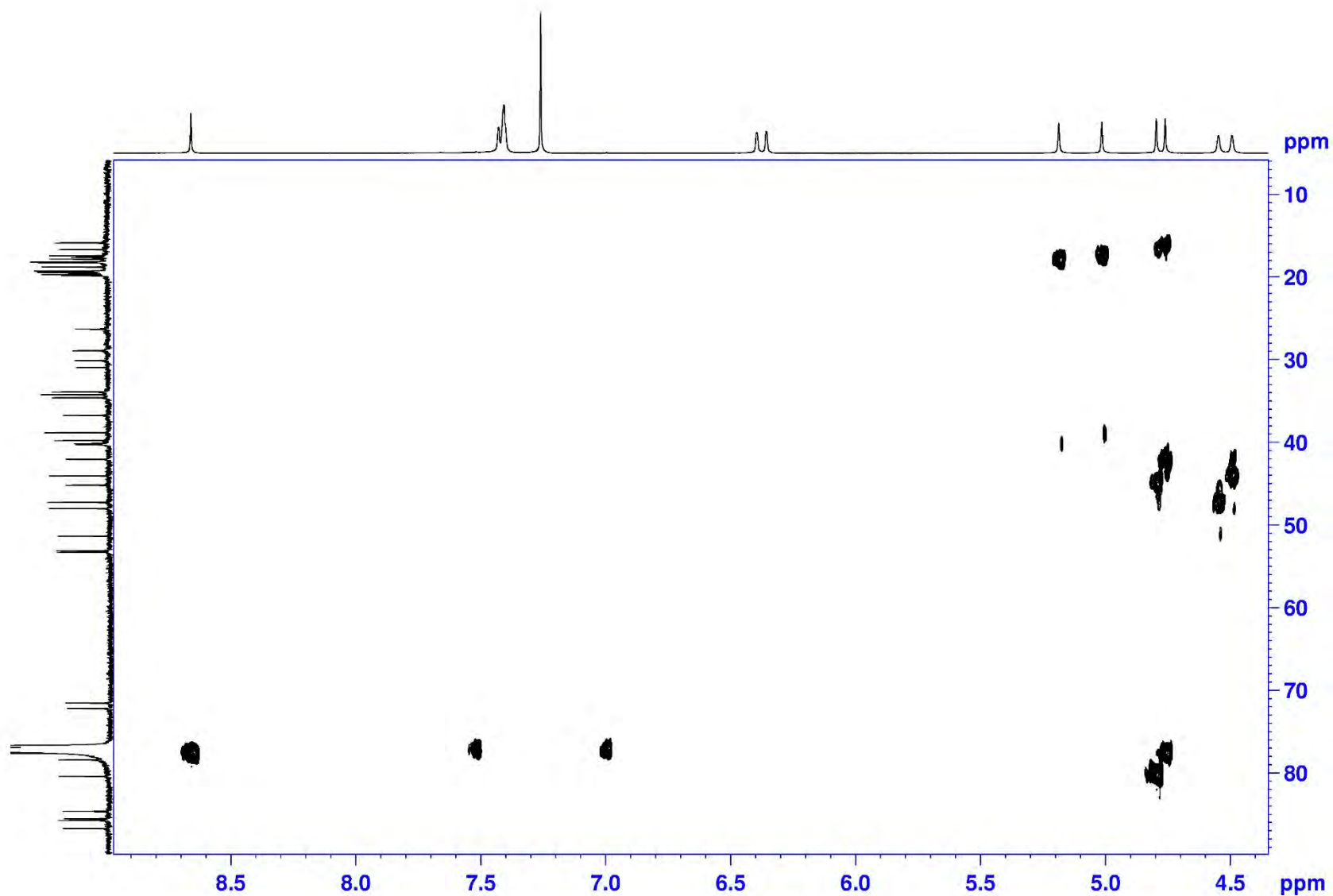
NAME lws-42-6-fd
 EXPNO 6
 PROCNO 1
 Date_ 20150816
 Time 4.21
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG hmbcgp1pndqf
 TD 4096
 SOLVENT CDCl3
 NS 64
 DS 16
 SWH 5197.505 Hz
 FIDRES 1.268922 Hz
 AQ 0.3940852 sec
 RG 208.5
 DW 96.200 usec
 DE 10.00 usec
 TE 297.0 K
 CNST2 145.0000000
 CNST13 10.0000000
 D0 0.00000300 sec
 D1 1.50000000 sec
 D2 0.00344828 sec
 D6 0.05000000 sec
 D16 0.00020000 sec
 IN0 0.00002080 sec

===== CHANNEL f1 =====
 SFO1 400.1323208 MHz
 NUC1 1H
 P1 12.00 usec
 P2 24.00 usec
 ND0 2
 TD 128
 SFO1 100.6233 MHz
 FIDRES 187.800476 Hz
 SW 238.896 ppm
 FMODE QF
 SI 2048
 SF 400.1300000 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40
 SI 1024
 MC2 QF
 SF 100.6127685 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0

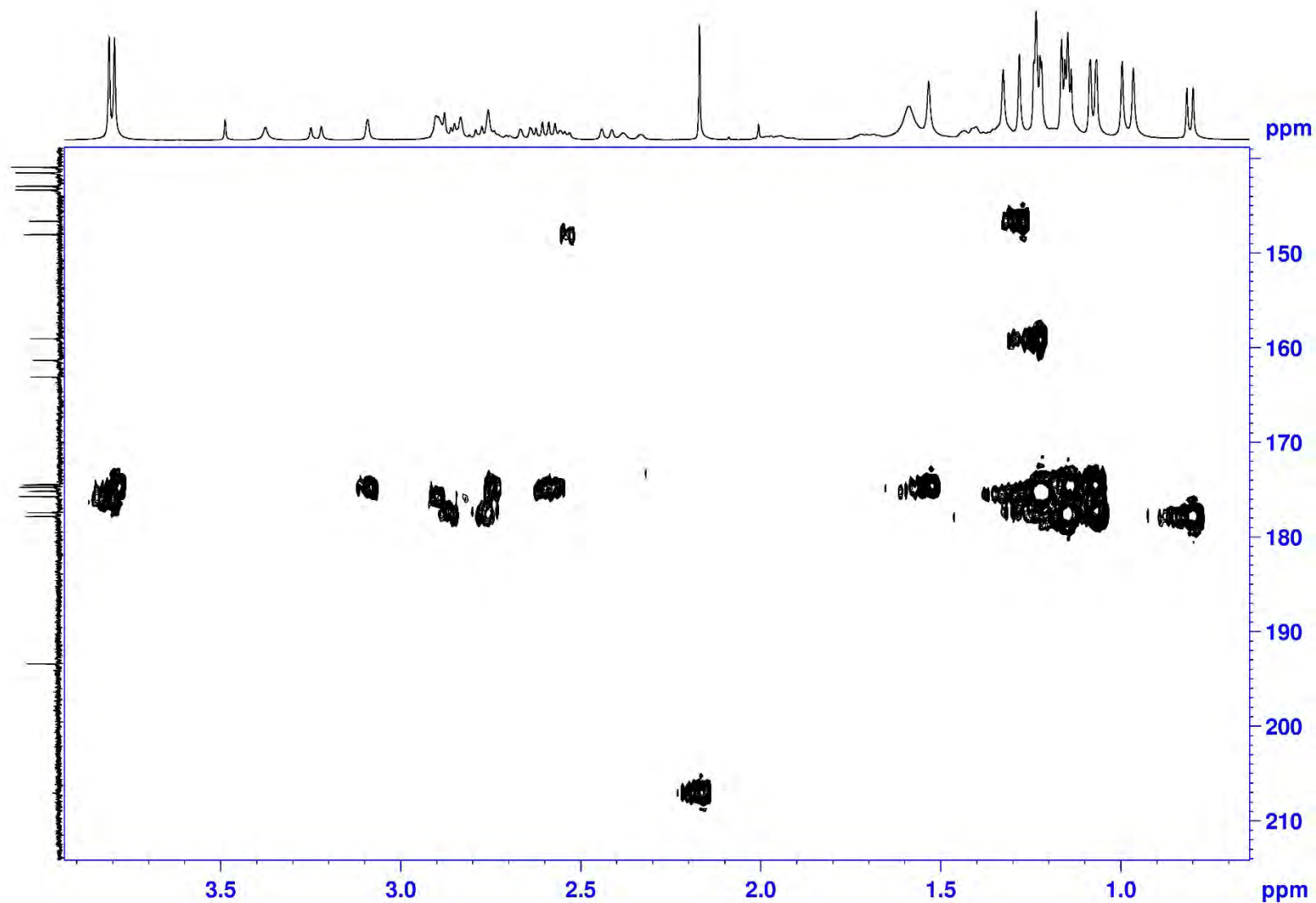
HMBC (400 MHz) spectrum of compound **4** in CDCl₃



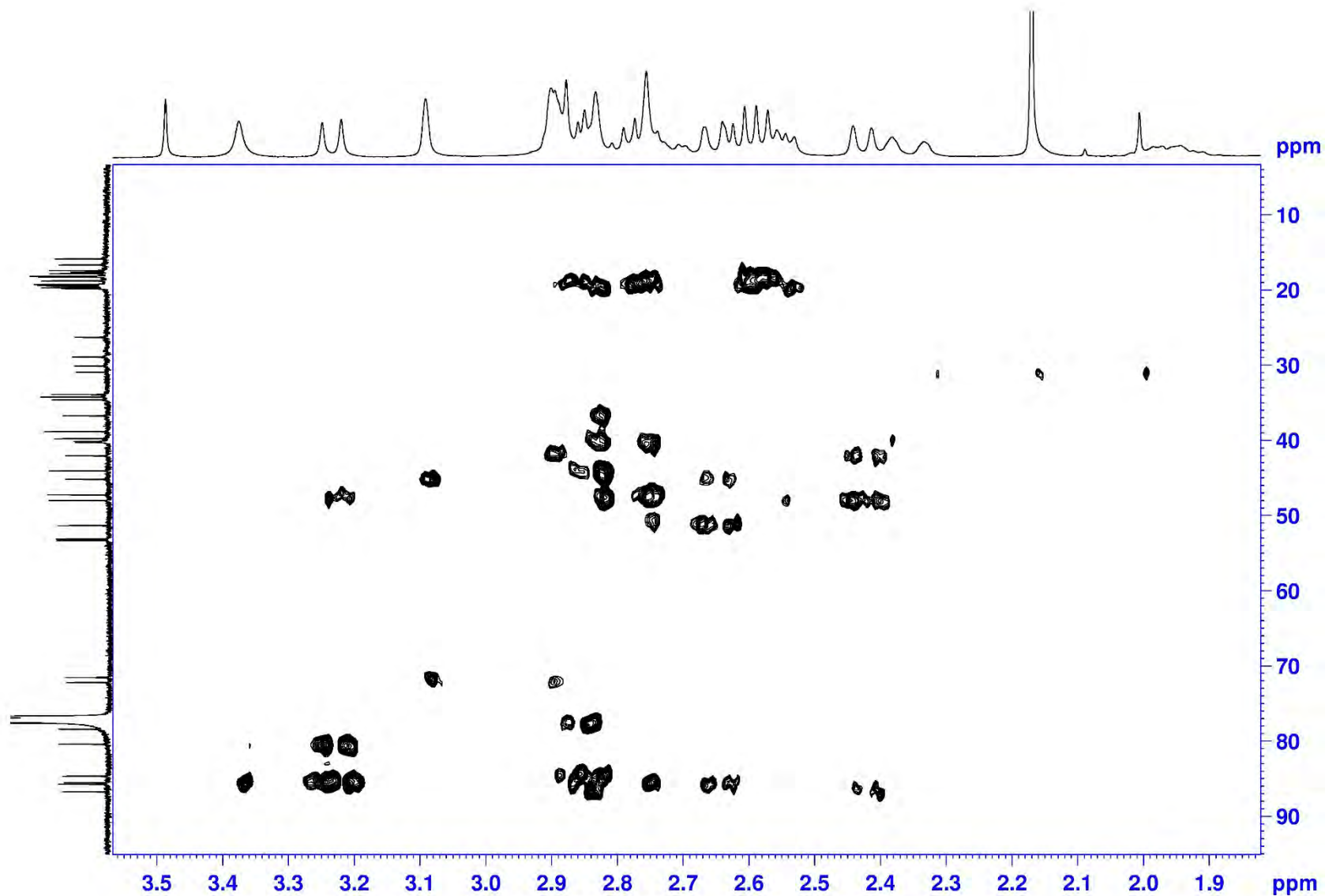
HMBC (400 MHz) spectrum of compound **4** in CDCl₃



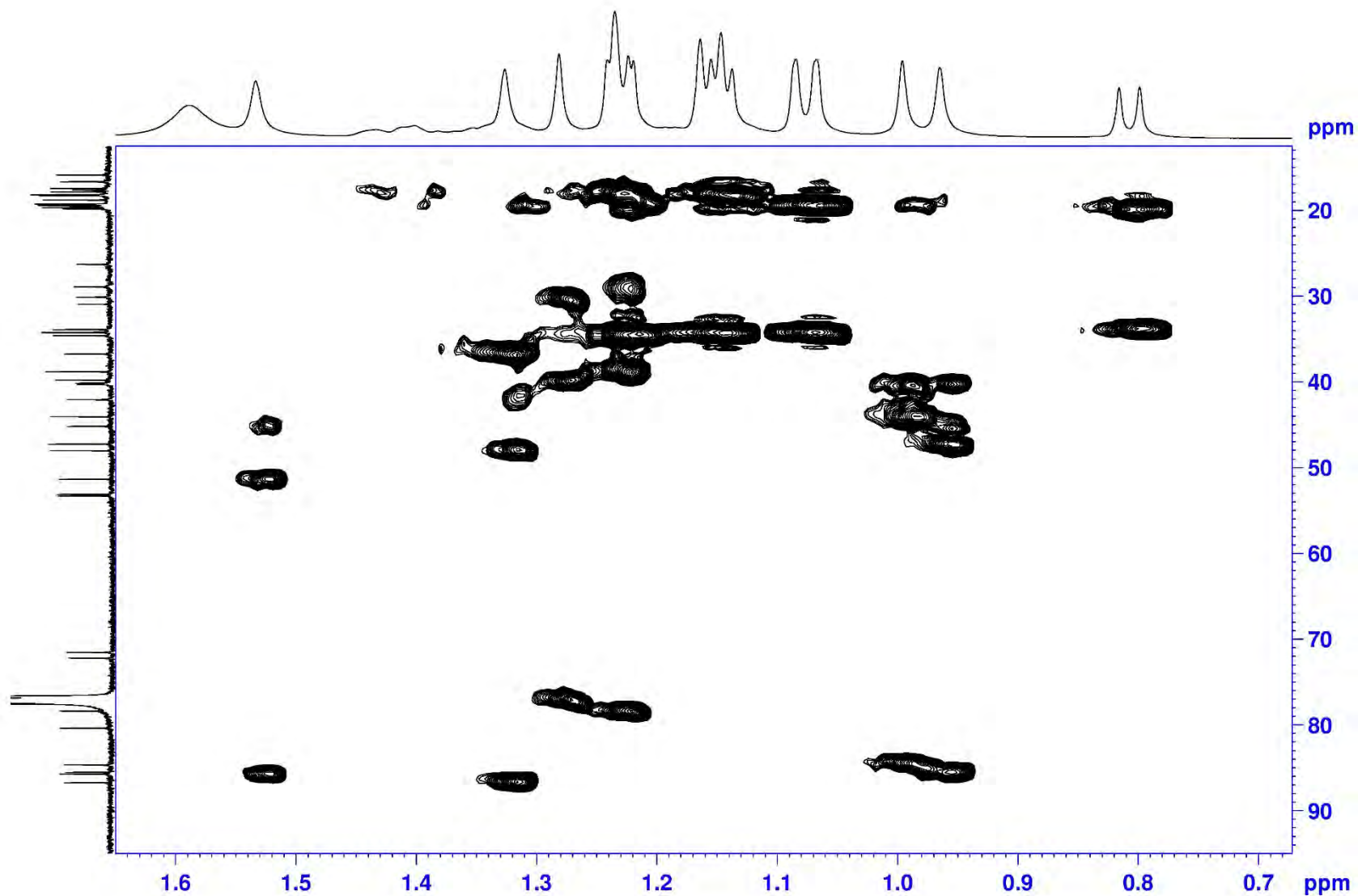
HMBC (400 MHz) spectrum of compound **4** in CDCl₃



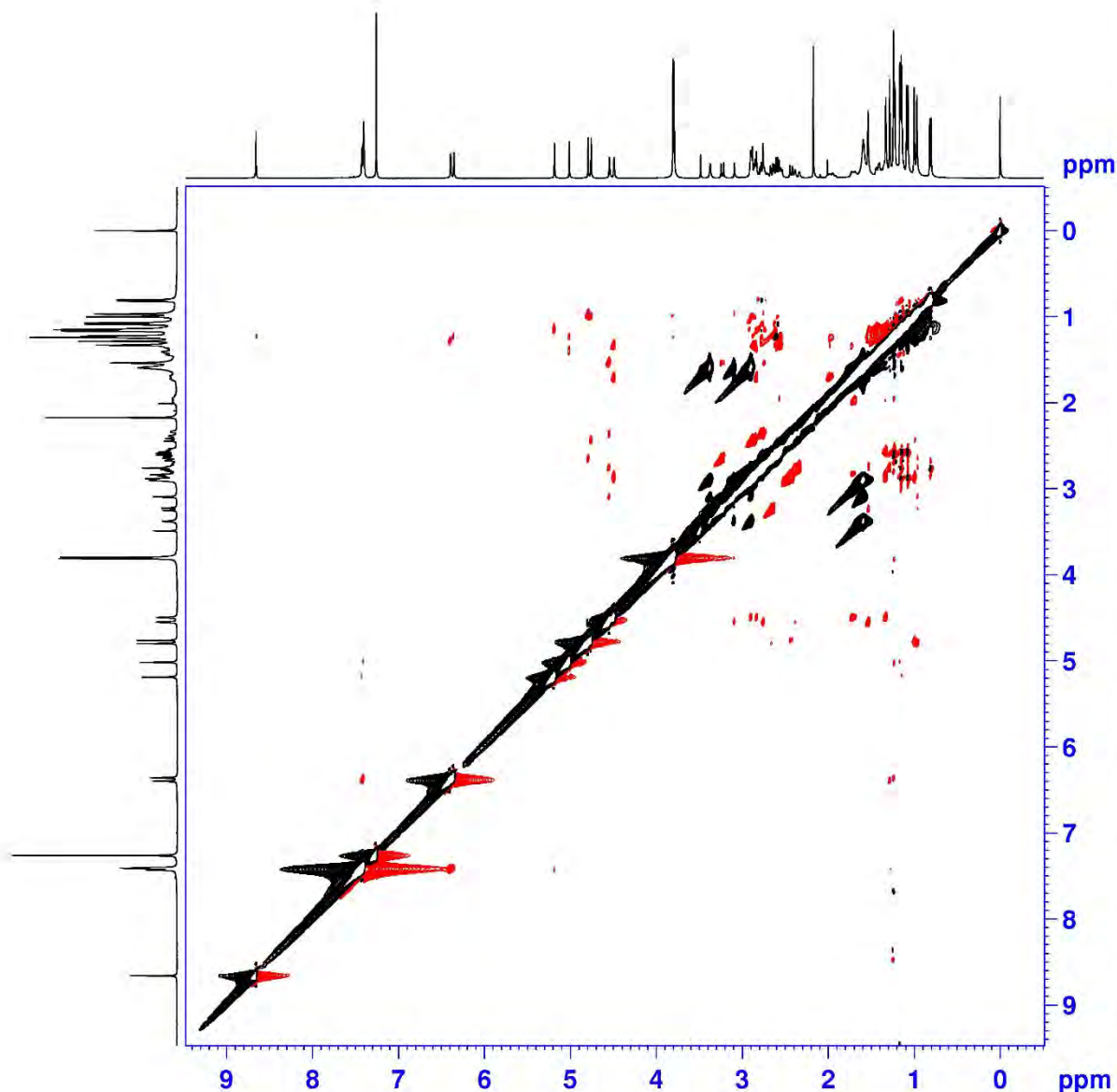
HMBC (400 MHz) spectrum of compound **4** in CDCl_3



HMBC (400 MHz) spectrum of compound **4** in CDCl₃



NOESY (400 MHz) spectrum of compound **4** in CDCl₃



```

NAME          lws-42-6-fd
EXPNO         7
PROCNO        1
Date_         20150816
Time          8.50
INSTRUM       spect
PROBHD        5 mm CPBBO BB
PULPROG       noesygpphpp
TD            2048
SOLVENT       CDCl3
NS            32
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
SSB            2
LB             0.00 Hz
GB             0
    
```

NOESY (400 MHz) spectrum of compound **4** in CDCl₃

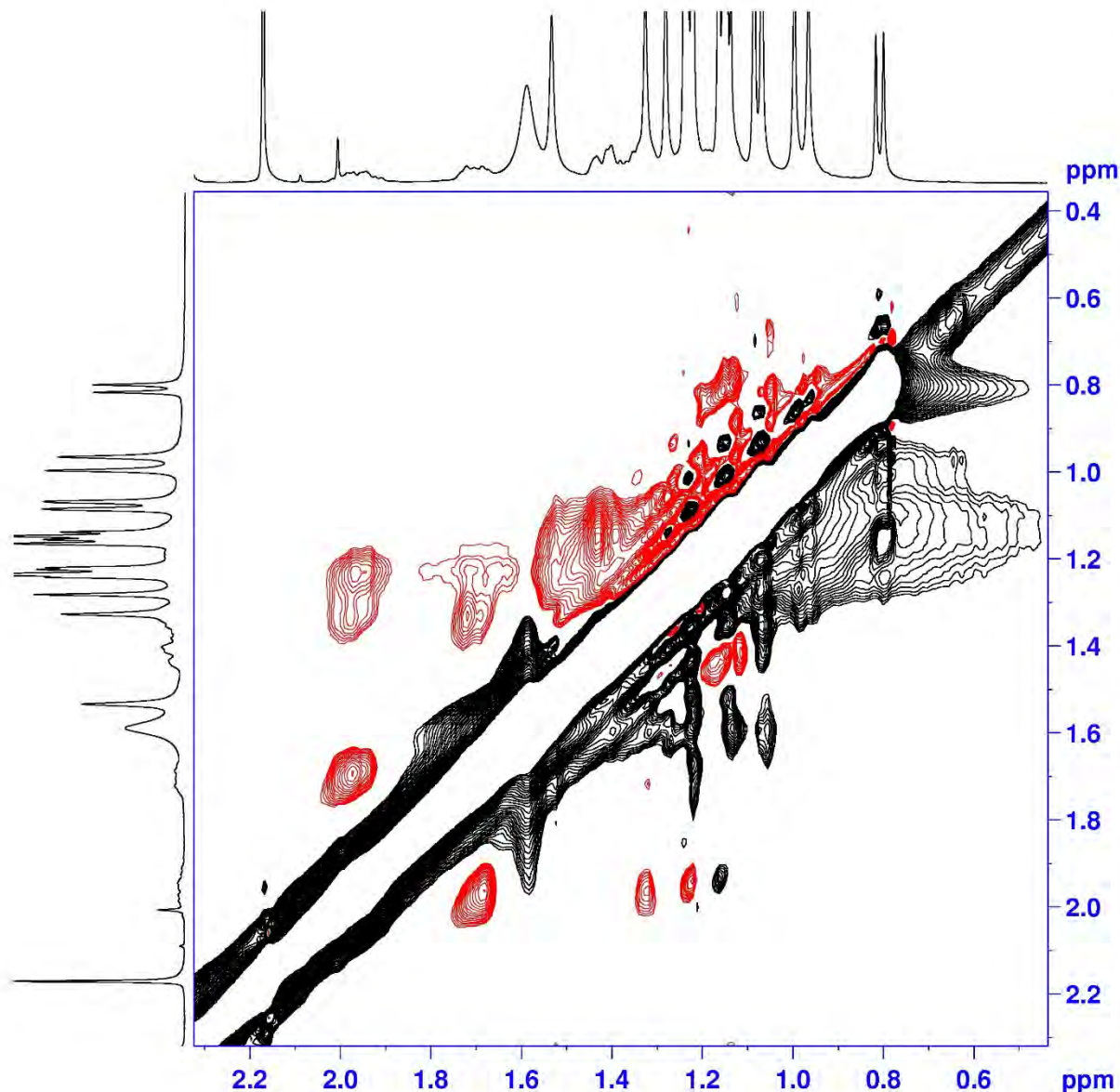


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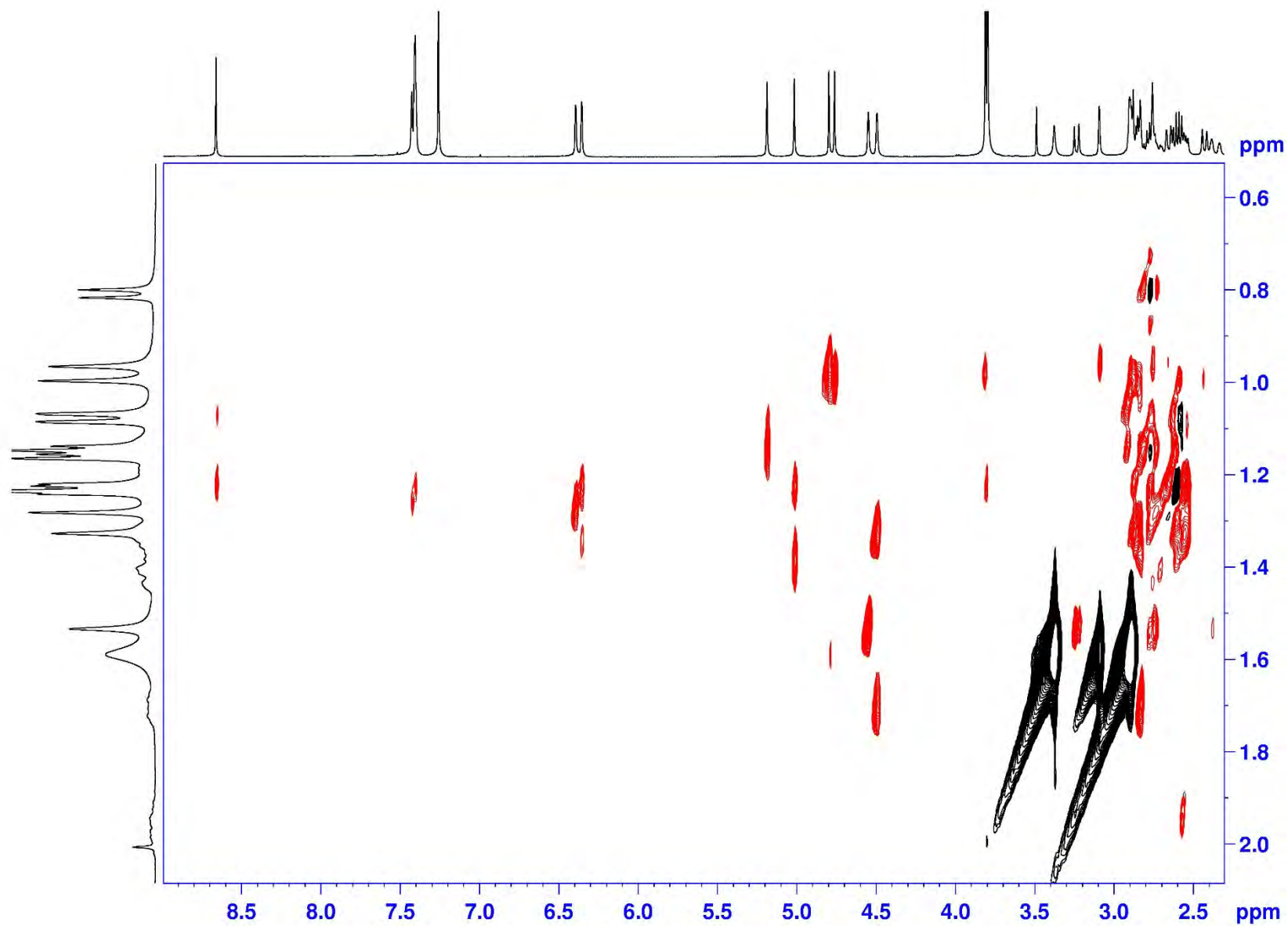
NAME      lws-42-6-fd
EXPNO     7
PROCNO    1
Date_     20150816
Time      8.50
INSTRUM   spect
PROBHD    5 mm CPBBO BB
PULPROG   noesygpphpp
TD        2048
SOLVENT   CDCl3
NS        32
DS        32
SWH       4000.000 Hz
FIDRES    1.953125 Hz
AQ        0.2560500 sec
RG        208.5
DW        125.000 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
  
```

```

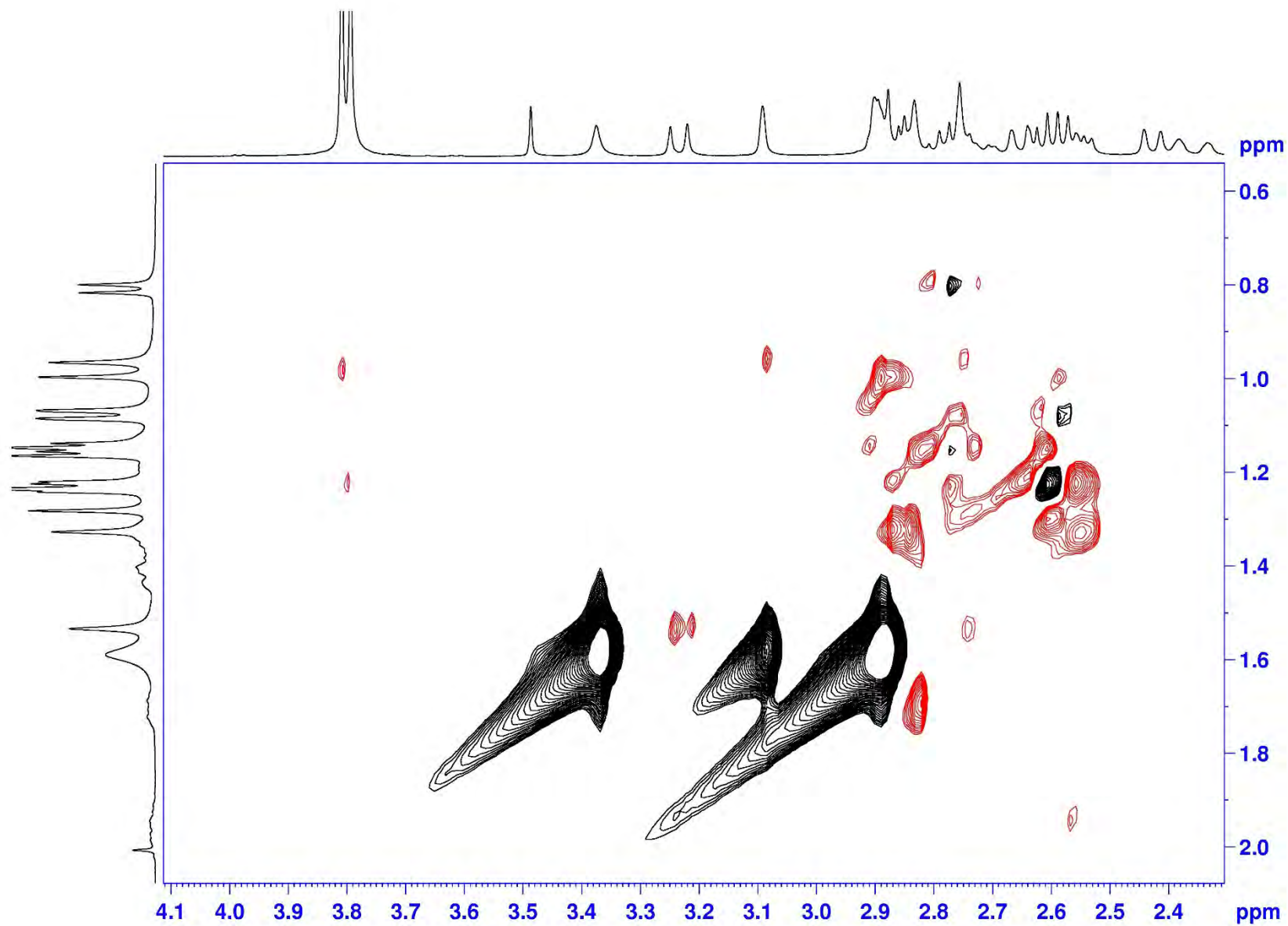
===== CHANNEL f1 =====
SFO1    400.1318006 MHz
NUC1     1H
P1       12.00 usec
P2       24.00 usec
P17      2500.00 usec
ND0       1
TD       256
SFO1    400.1318 MHz
FIDRES   15.625000 Hz
SW        9.997 ppm
FnMODE   States-TPPI
SI       1024
SF       400.1300098 MHz
WDW      QSINE
SSB       2
LB  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



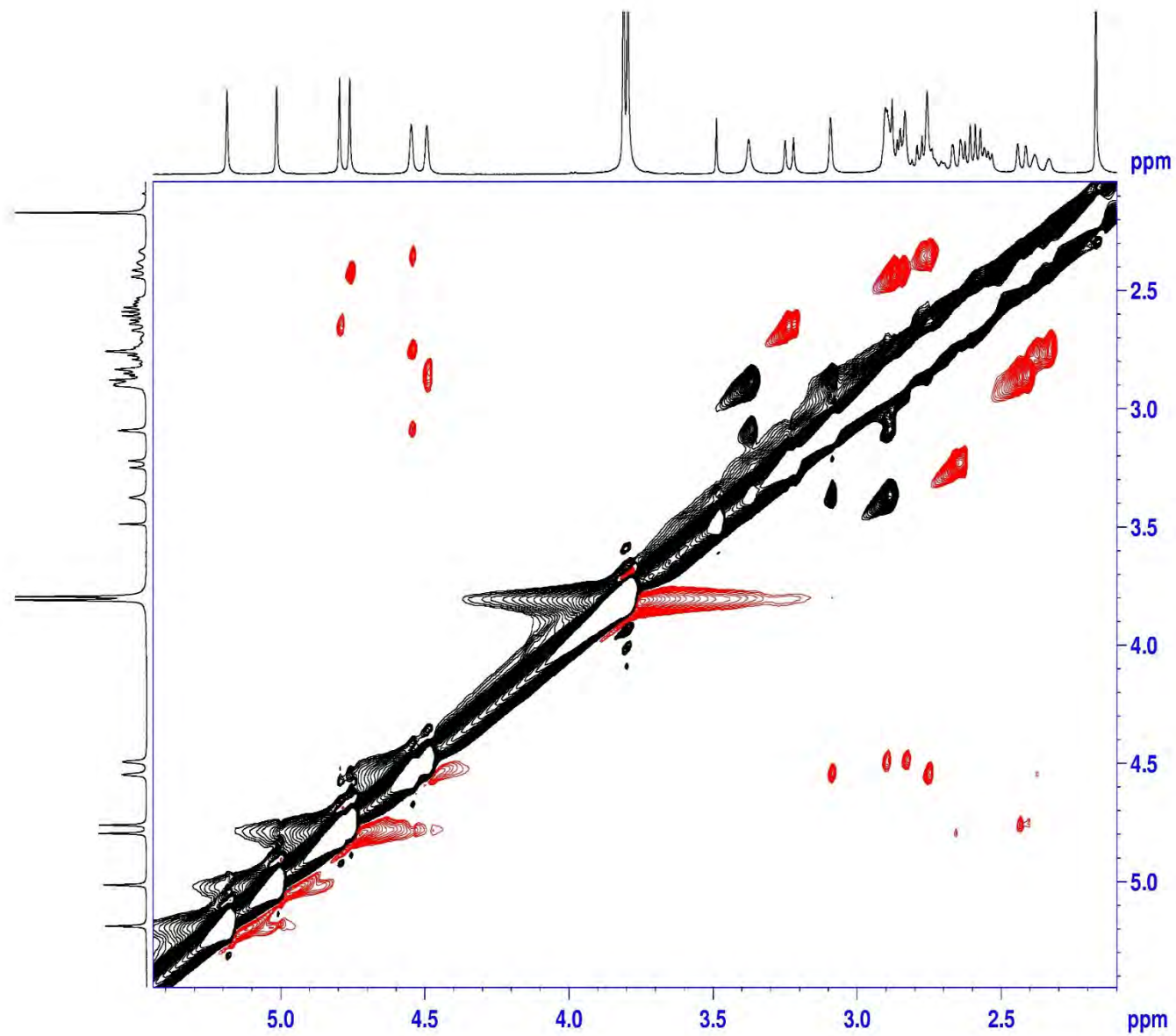
NOESY (400 MHz) spectrum of compound **4** in CDCl₃



NOESY (400 MHz) spectrum of compound **4** in CDCl₃



NOESY (400 MHz) spectrum of compound **4** in CDCl₃

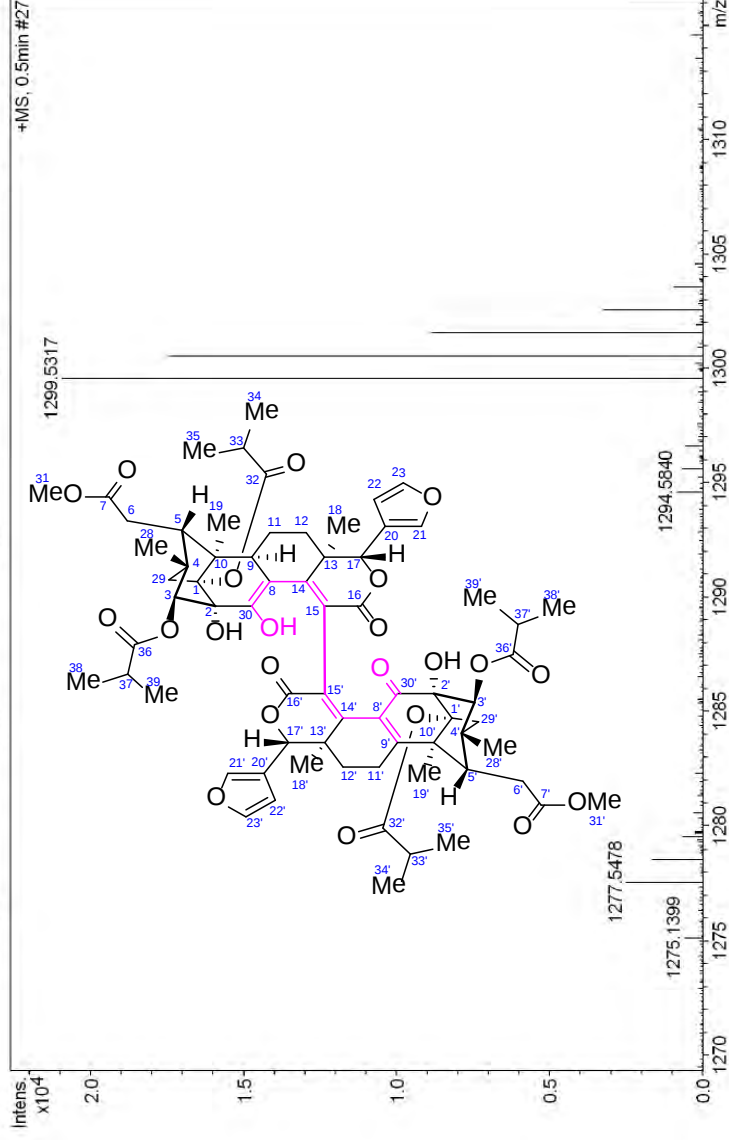
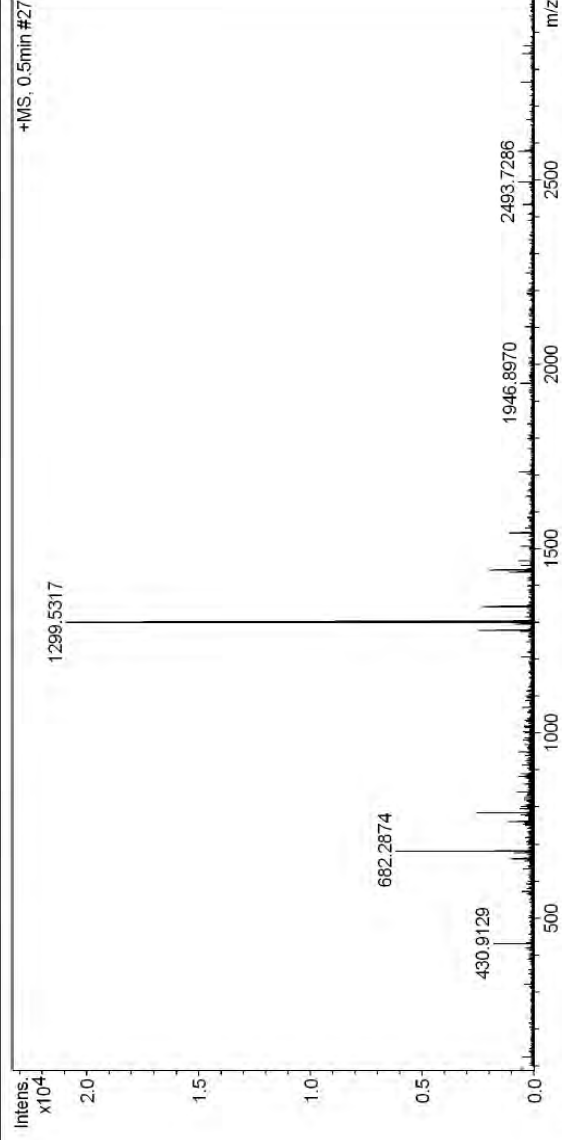


HR-ESIMS for compound 4'

Generic Display Report

Analysis Info

Analysis Name: D:\Data\MS\data\201607\liwanshan_37-6_pos_24_01_2183.d
 Method: LC_Direct Infusion_pos_100-3000mz.m
 Sample Name: liwanshan_37-6_pos
 Comment:
 Acquisition Date: 7/15/2016 5:14:49 PM
 Operator: SCSIO
 Instrument: maXis

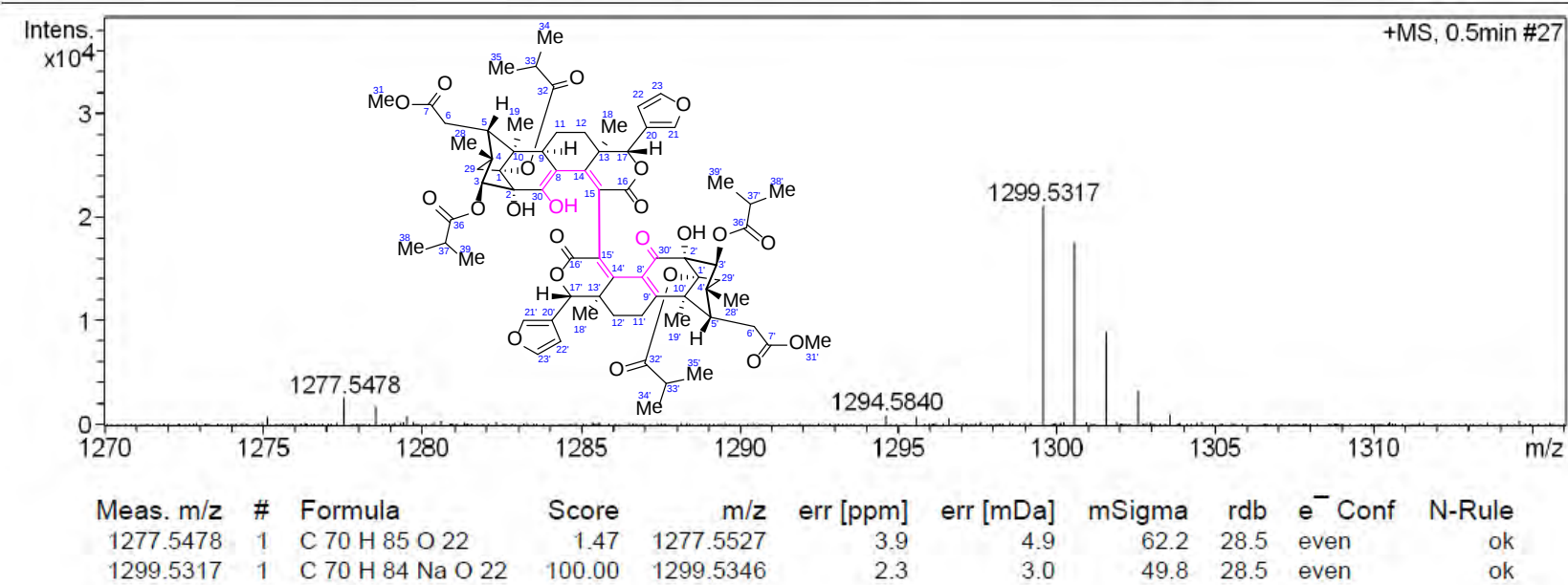


HR-ESIMS for compound 4'

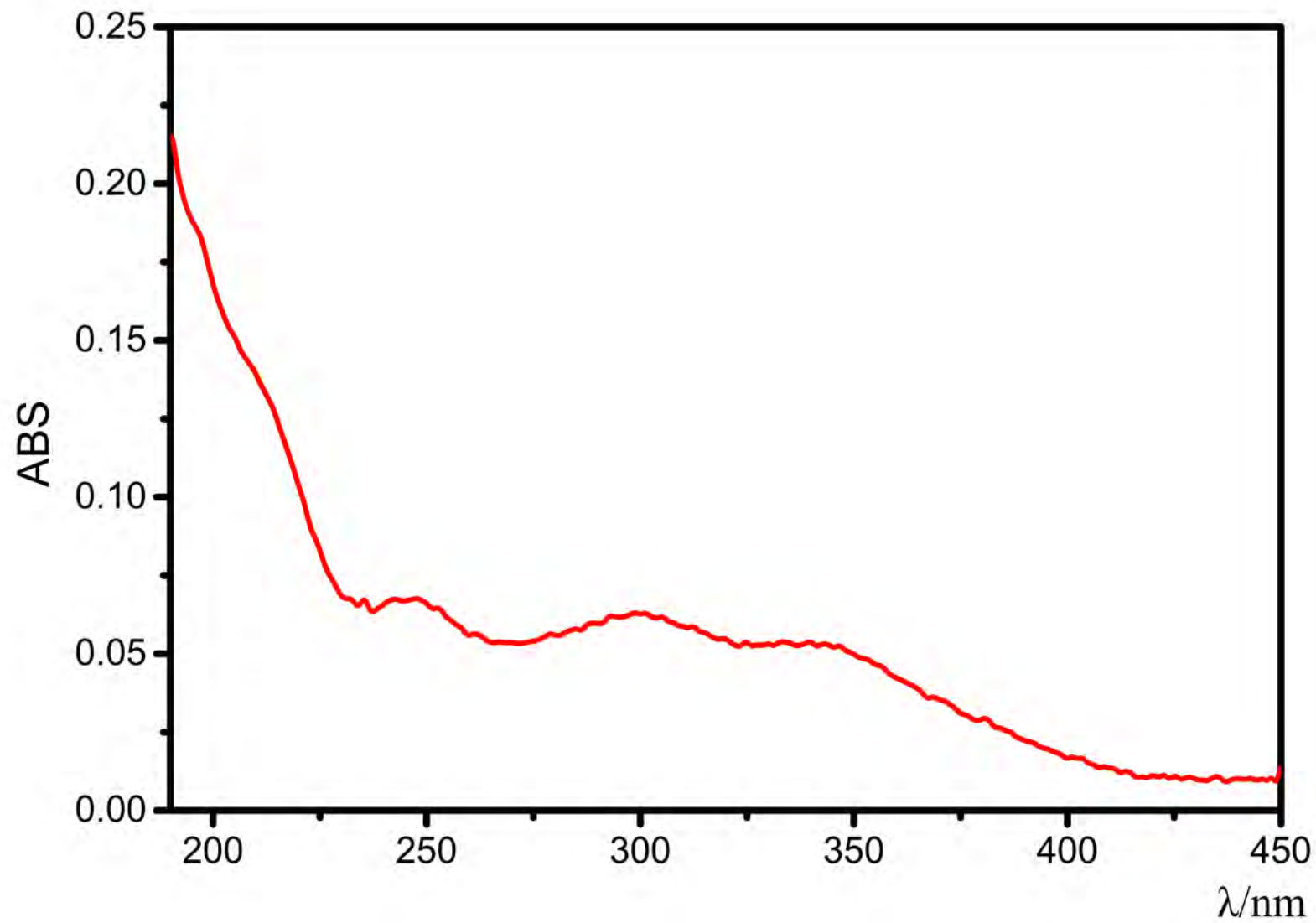
Mass Spectrum SmartFormula Report

Analysis Info		Acquisition Date		7/15/2016 5:14:49 PM	
Analysis Name	D:\Data\MS\data\201607\liwanshan_37-6_pos_24_01_2193.d		Operator	SCSIO	
Method	LC_Direct Infusion_pos_100-3000mz.m		Instrument / Ser#	maXis	29
Sample Name	liwanshan_37-6_pos				
Comment					

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	3000 m/z	Set Collision Cell RF	1800.0 Vpp	Set Divert Valve	Waste

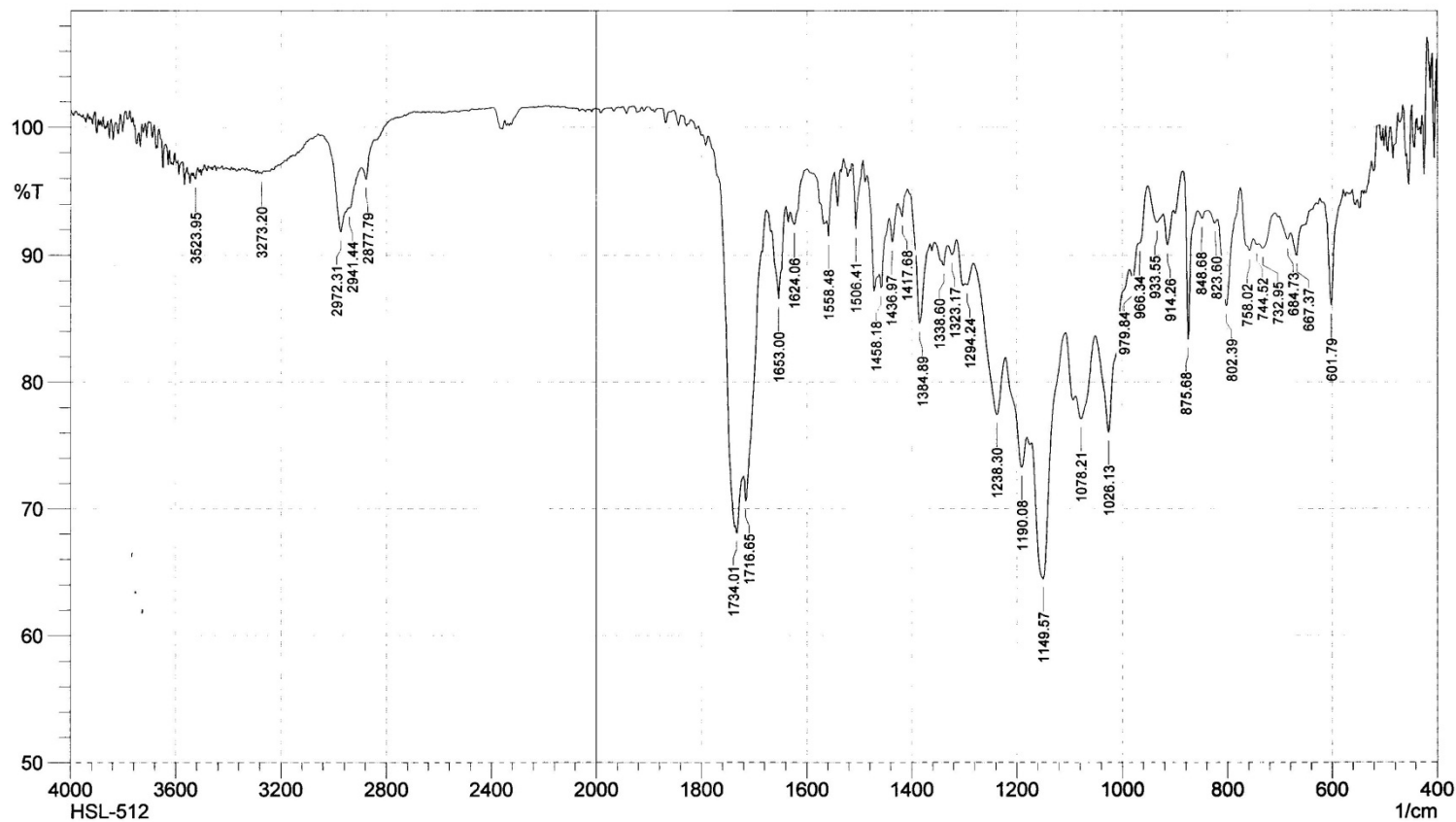


UV spectrum for compound **4'** in CH₃CN



IR spectrum for compound 4'

SHIMADZU

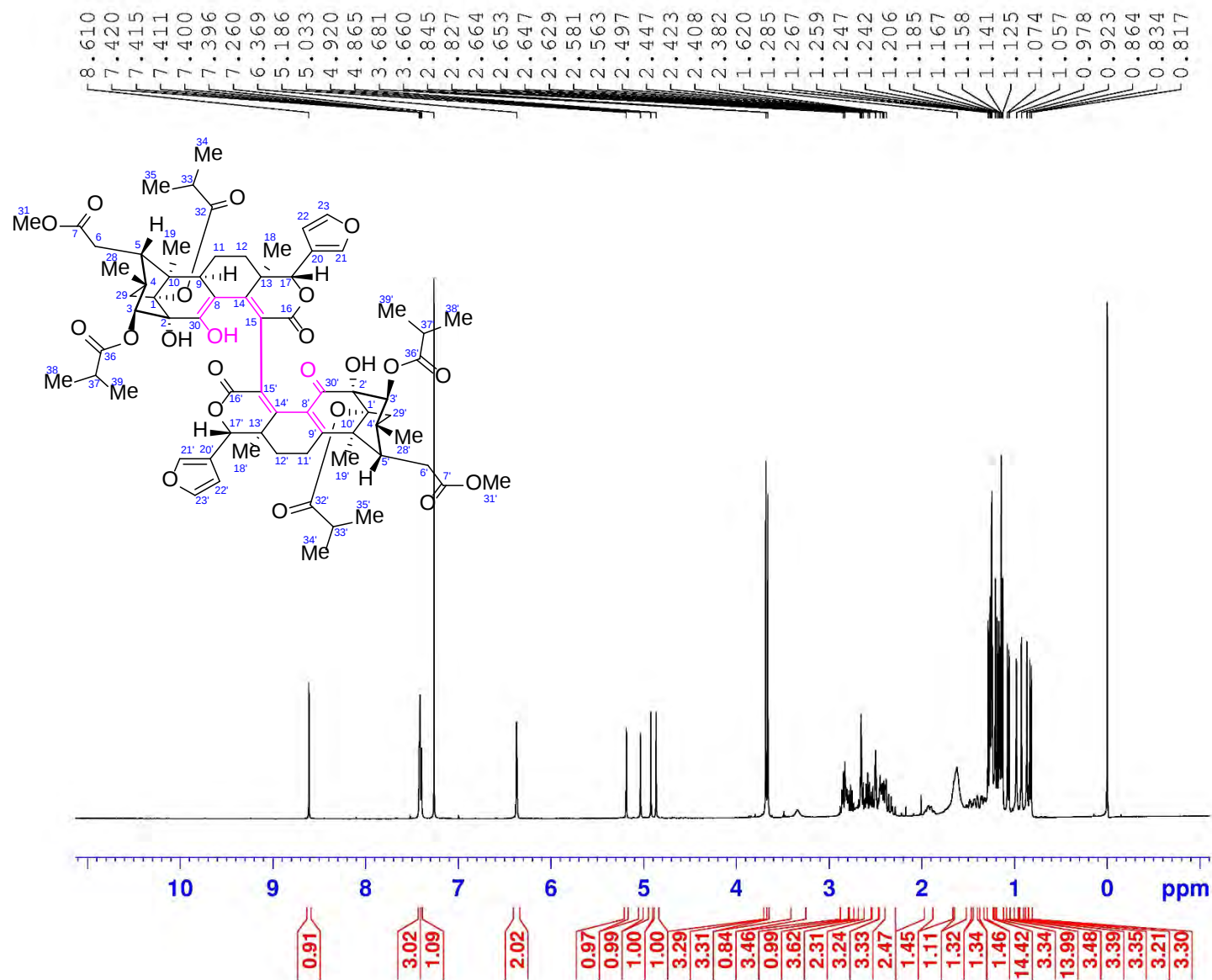


Sample name: 37-6(2)1 (4')
F:\liwanshan\20180226\37-
6(2)1.smf

Scan number: 25
Resolution: 4 [1/cm]

Date: 2018-2-26 15:20:00
Operator: SCSIO

^1H NMR (400 MHz) spectrum of compound **4'** in CDCl_3



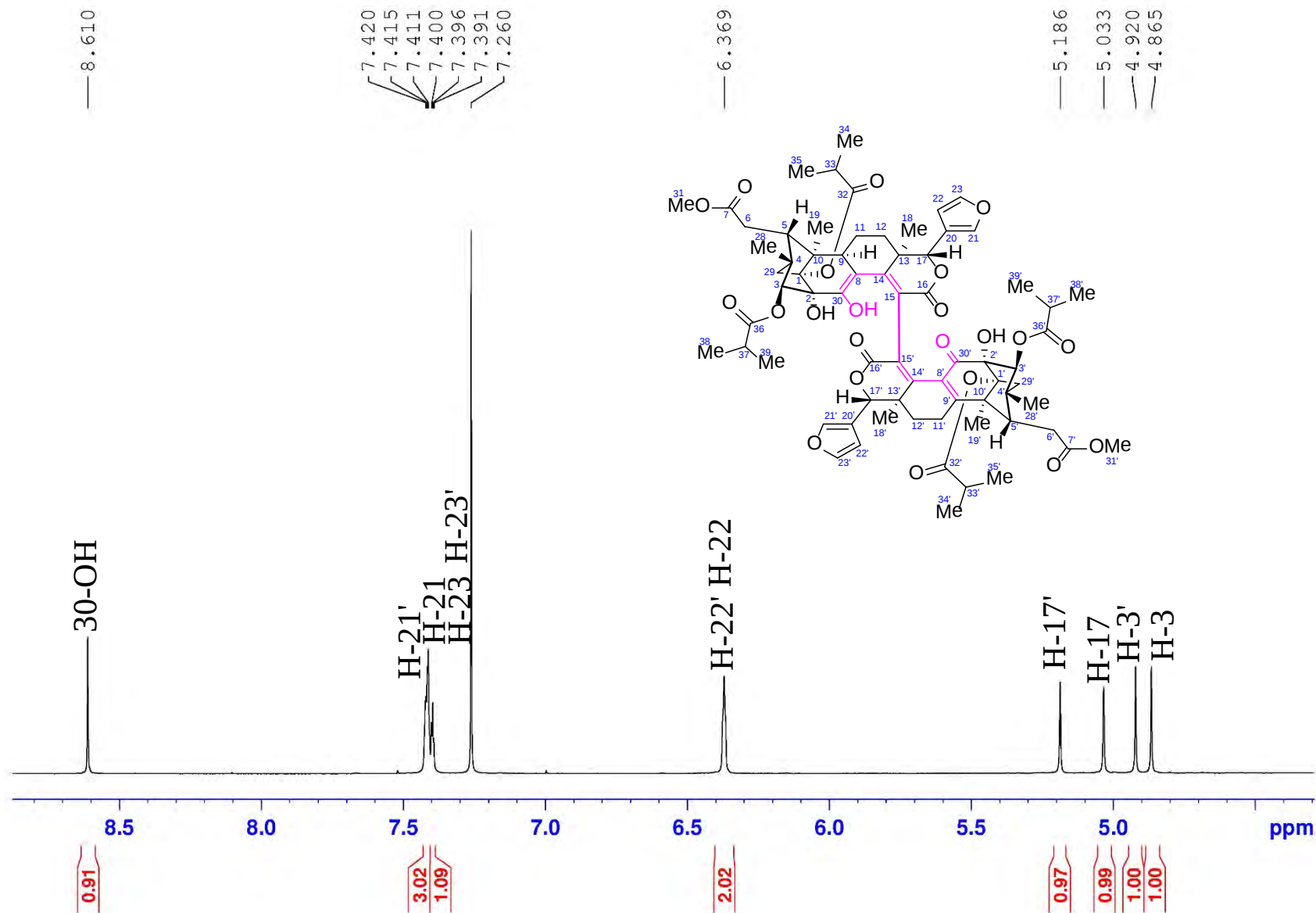
```

NAME          lws-37-6
EXPNO          1
PROCNO         1
Date_          20160613
Time           0.13
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
    
```

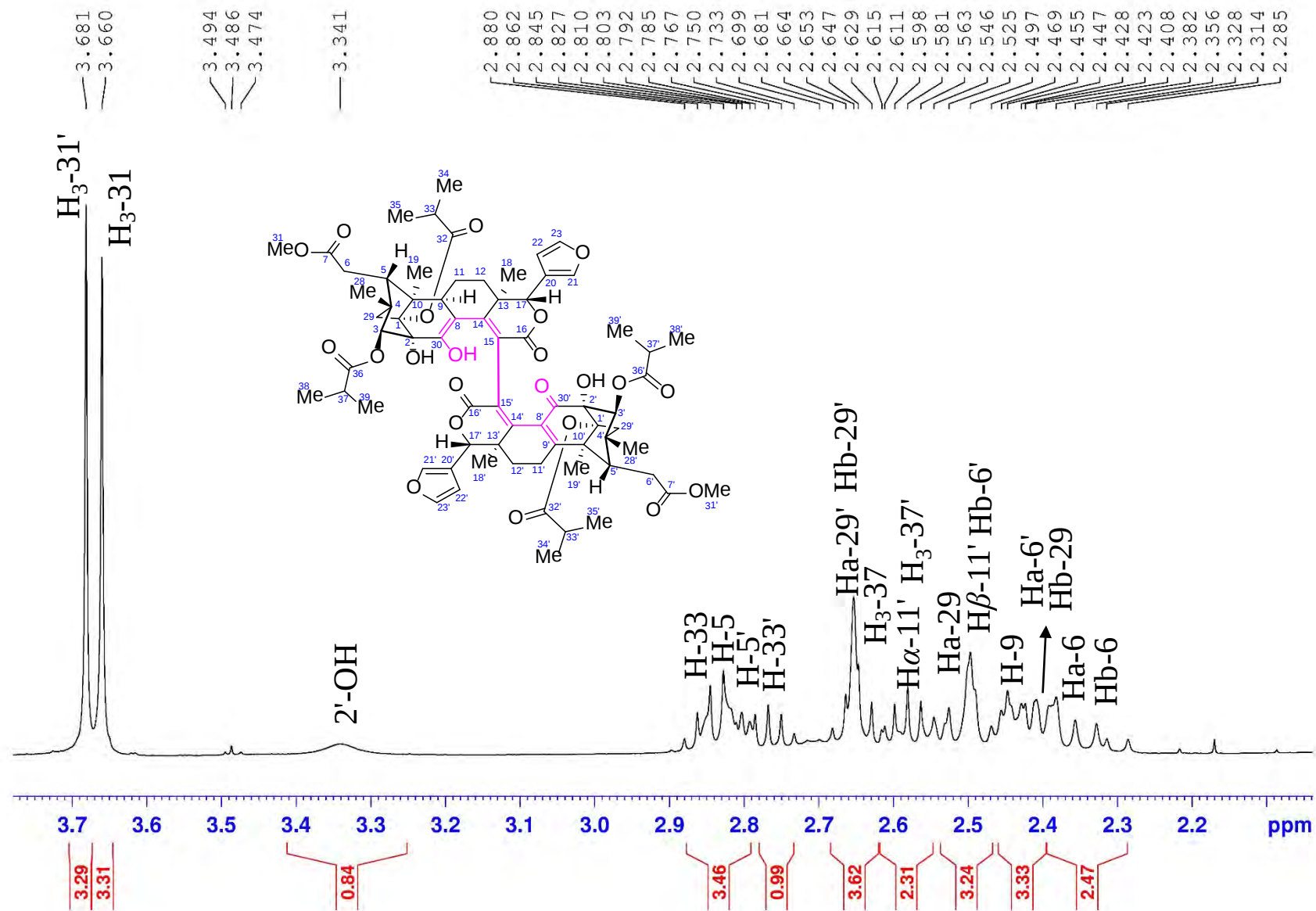
```

===== CHANNEL f1 =====
SFO1          400.1324710 M
NUC1           1H
P1            11.50 u
SI            65536
SF            400.1300101 M
WDW            EM
SSB            0
LB            0.30 F
GB            0
PC            1.00
    
```

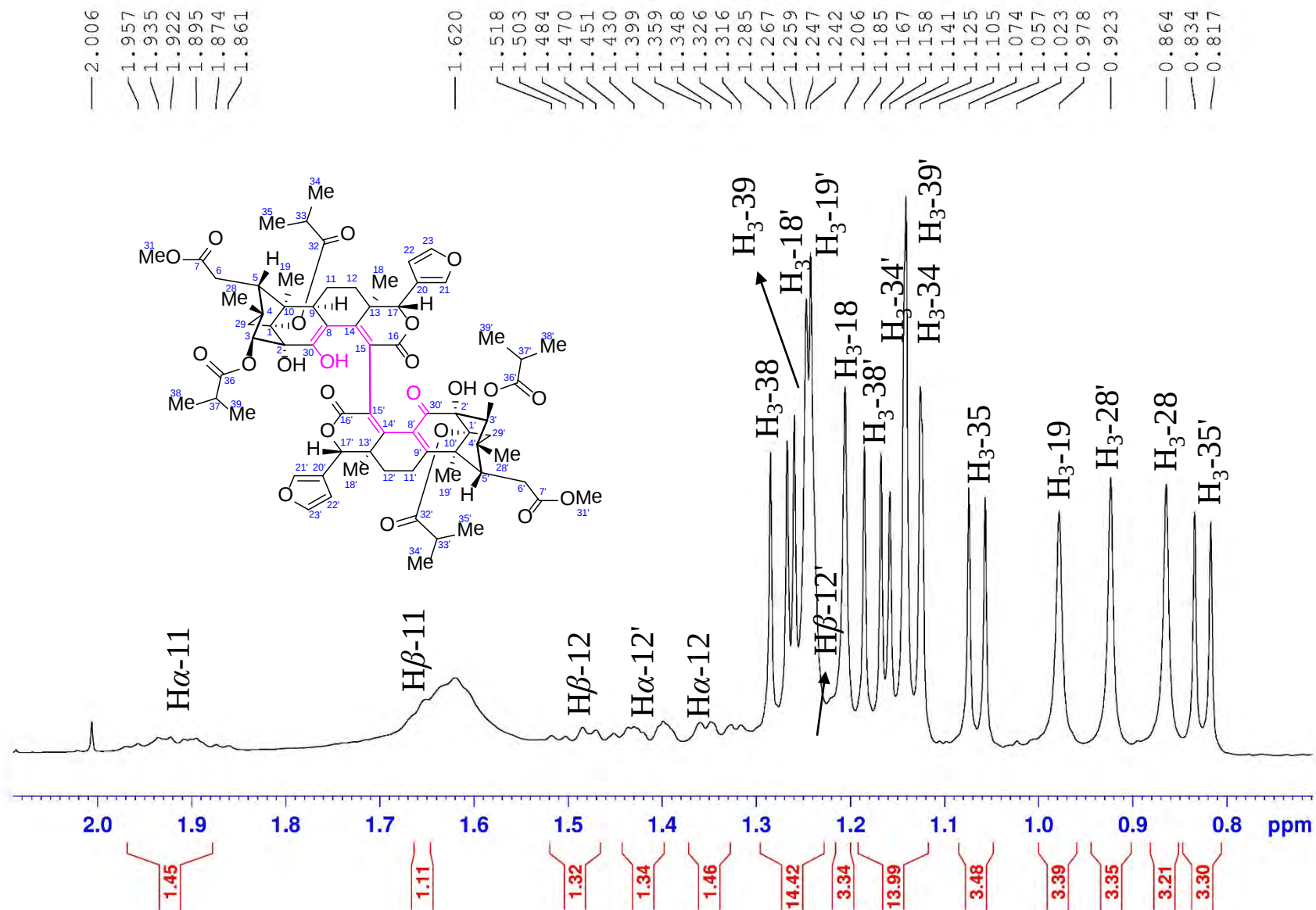
^1H NMR (400 MHz) spectrum of compound **4'** in CDCl_3



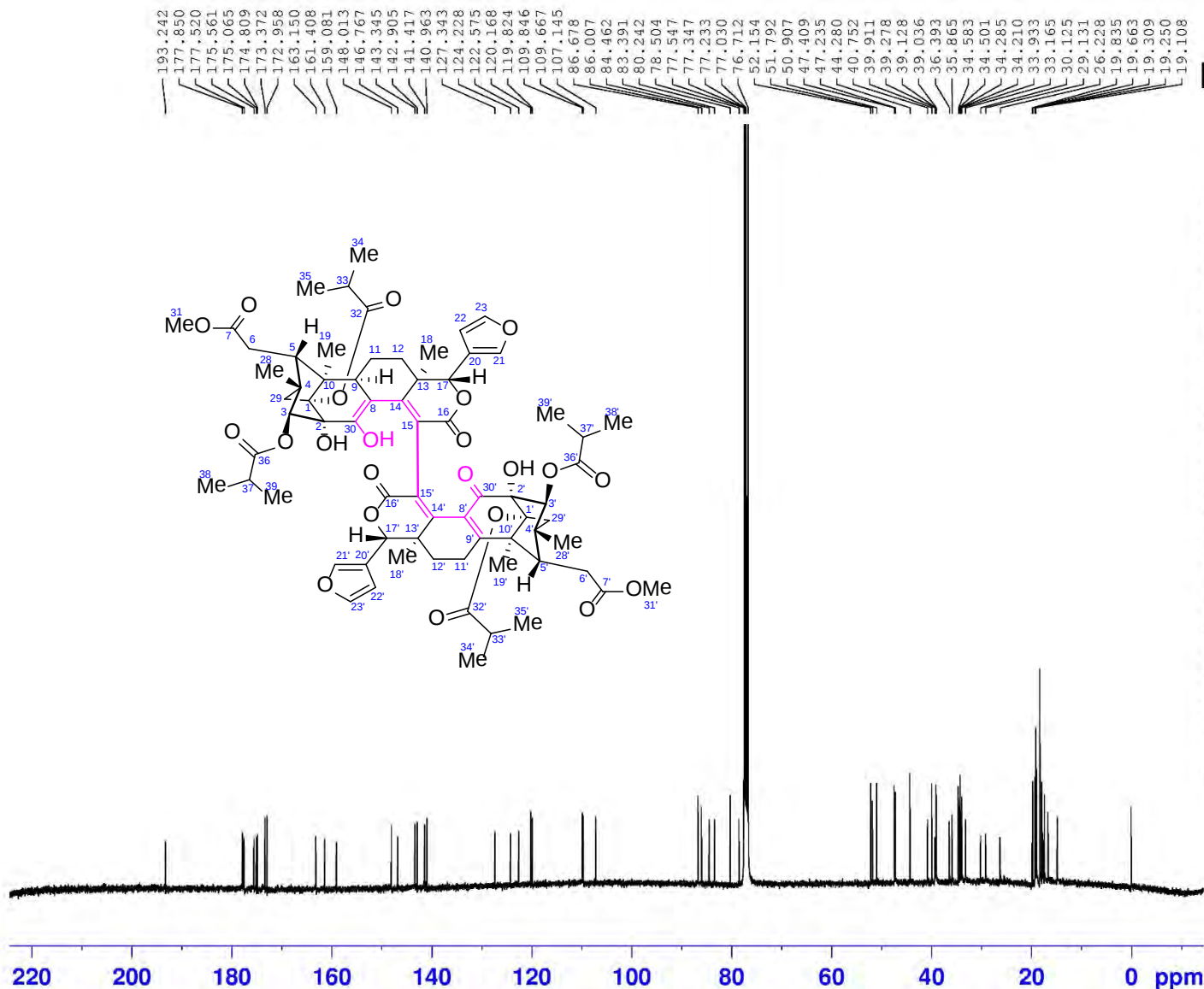
^1H NMR (400 MHz) spectrum of compound **4'** in CDCl_3



^1H NMR (400 MHz) spectrum of compound **4'** in CDCl_3



^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



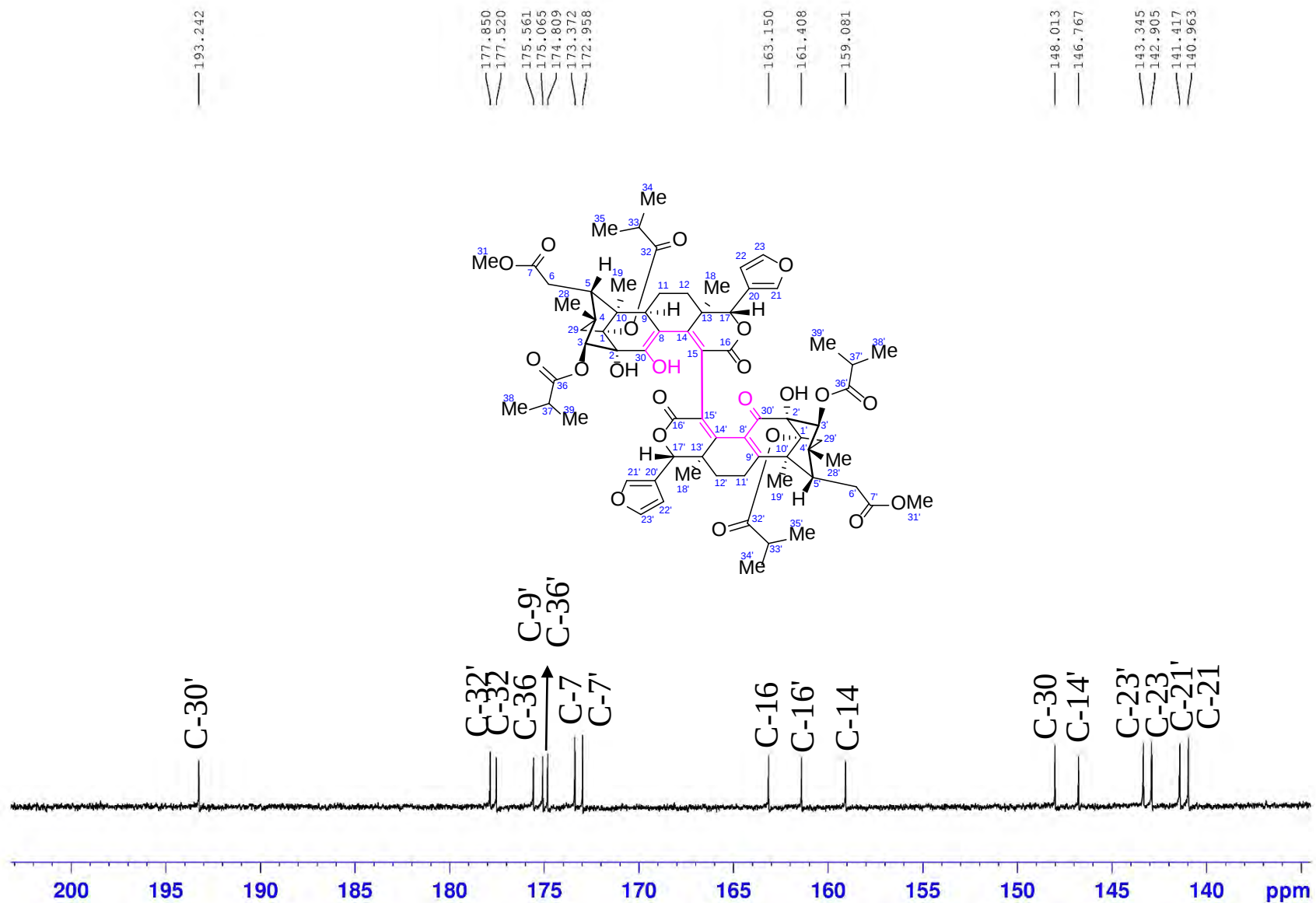
```

NAME          lws-37-6
EXPNO          2
PROCNO         1
Date_         20160613
Time           6.01
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             6000
DS             4
SWH            24038.461 H
FIDRES         0.366798 H
AQ             1.3631988 s
RG             102.3
DW             20.800 u
DE             18.00 u
TE             297.0 K
D1             2.00000000 s
D11            0.03000000 s
TD0            1
    
```

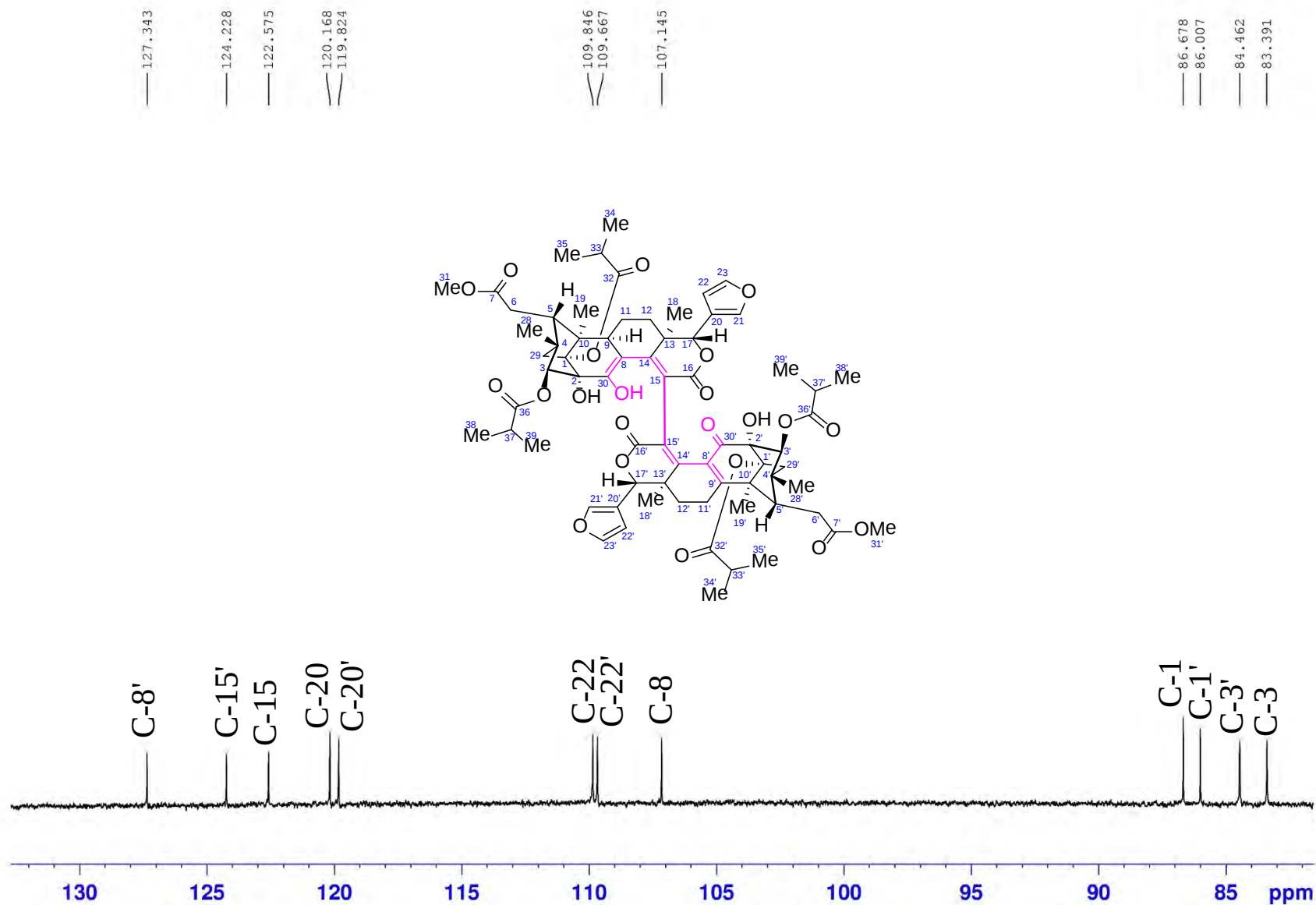
```

===== CHANNEL f1 =====
SFO1          100.6233324 M
NUC1           13C
P1             10.00 u
SI             32768
SF            100.6127682 M
WDW            EM
SSB            0
LB             1.00 H
GB             0
PC             1.40
    
```

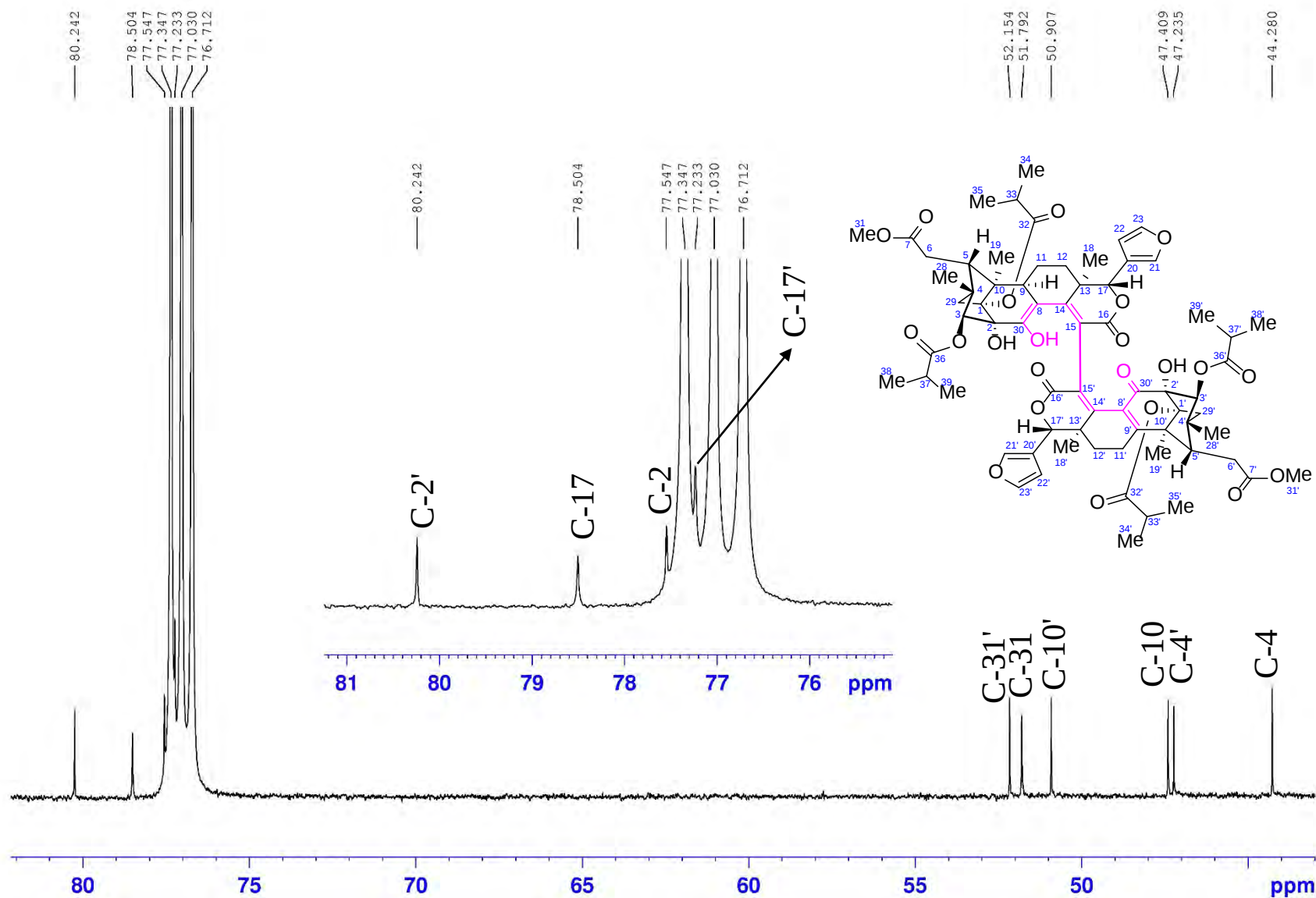
^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



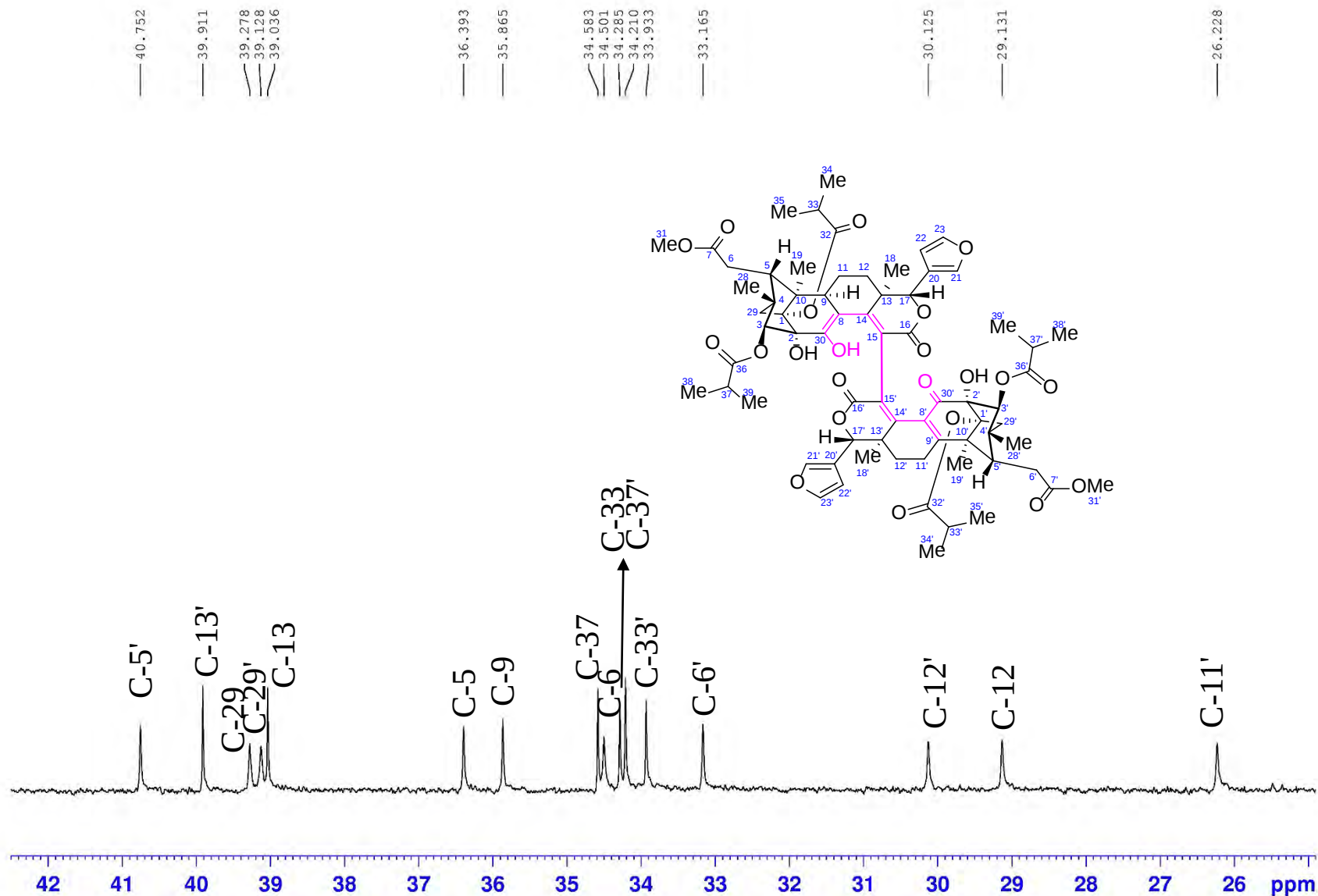
^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



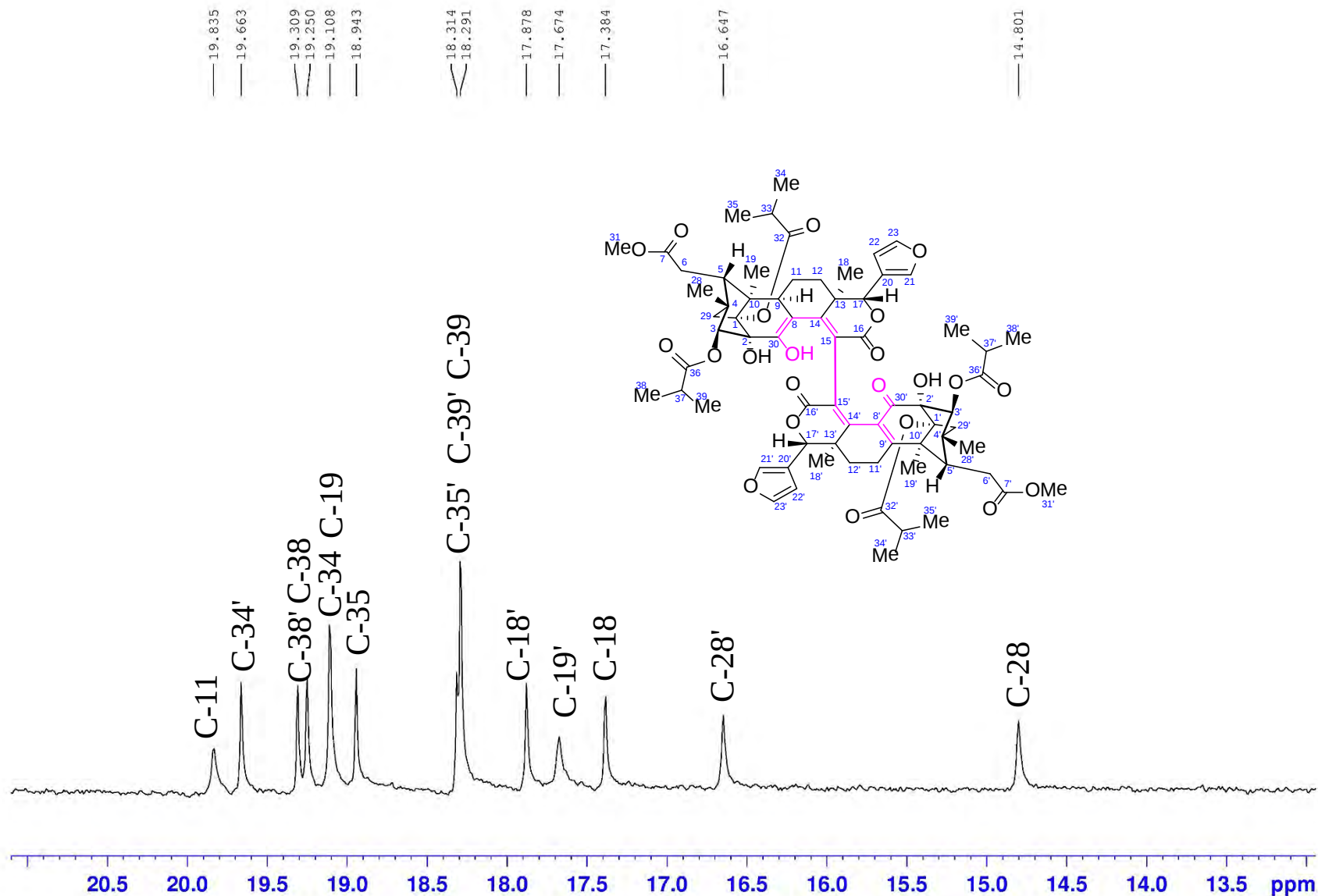
^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



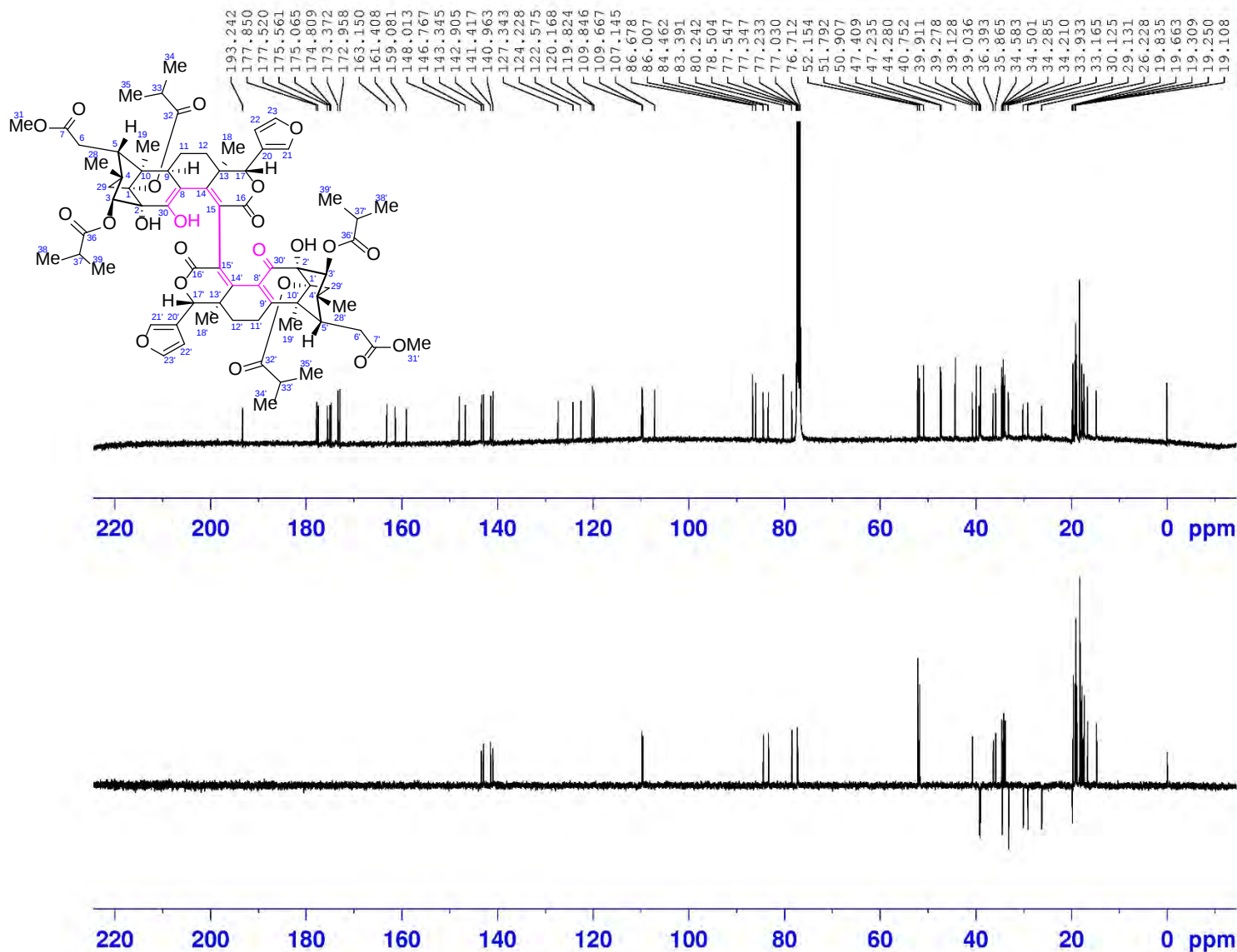
^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



^{13}C NMR (100 MHz) spectrum of compound **4'** in CDCl_3



DEPT135 (100 MHz) spectrum of compound 4' in CDCl₃



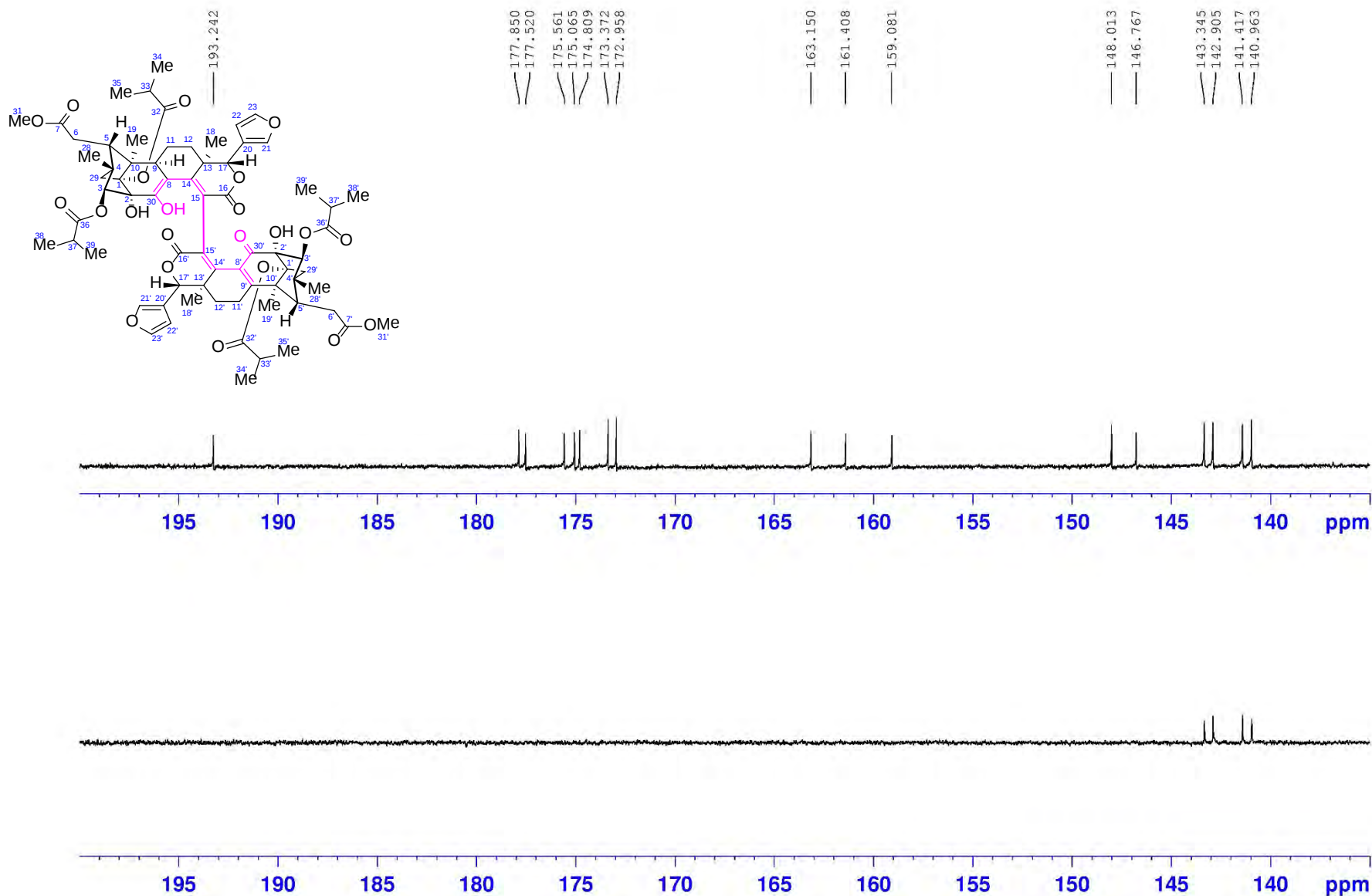
```

NAME          lws-37-6
EXPNO          3
PROCNO         1
Date_          20160613
Time           7.28
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        deptsp135
TD             65536
SOLVENT        CDCl3
NS             1500
DS             4
SWH            24038.461
FIDRES         0.366798
AQ             1.3631988
RG             130.26
DW             20.800
DE             18.00
TE             297.0
CNST2          145.0000000
D1             2.000000000
D2             0.00344828
D12            0.00002000
TD0            1
    
```

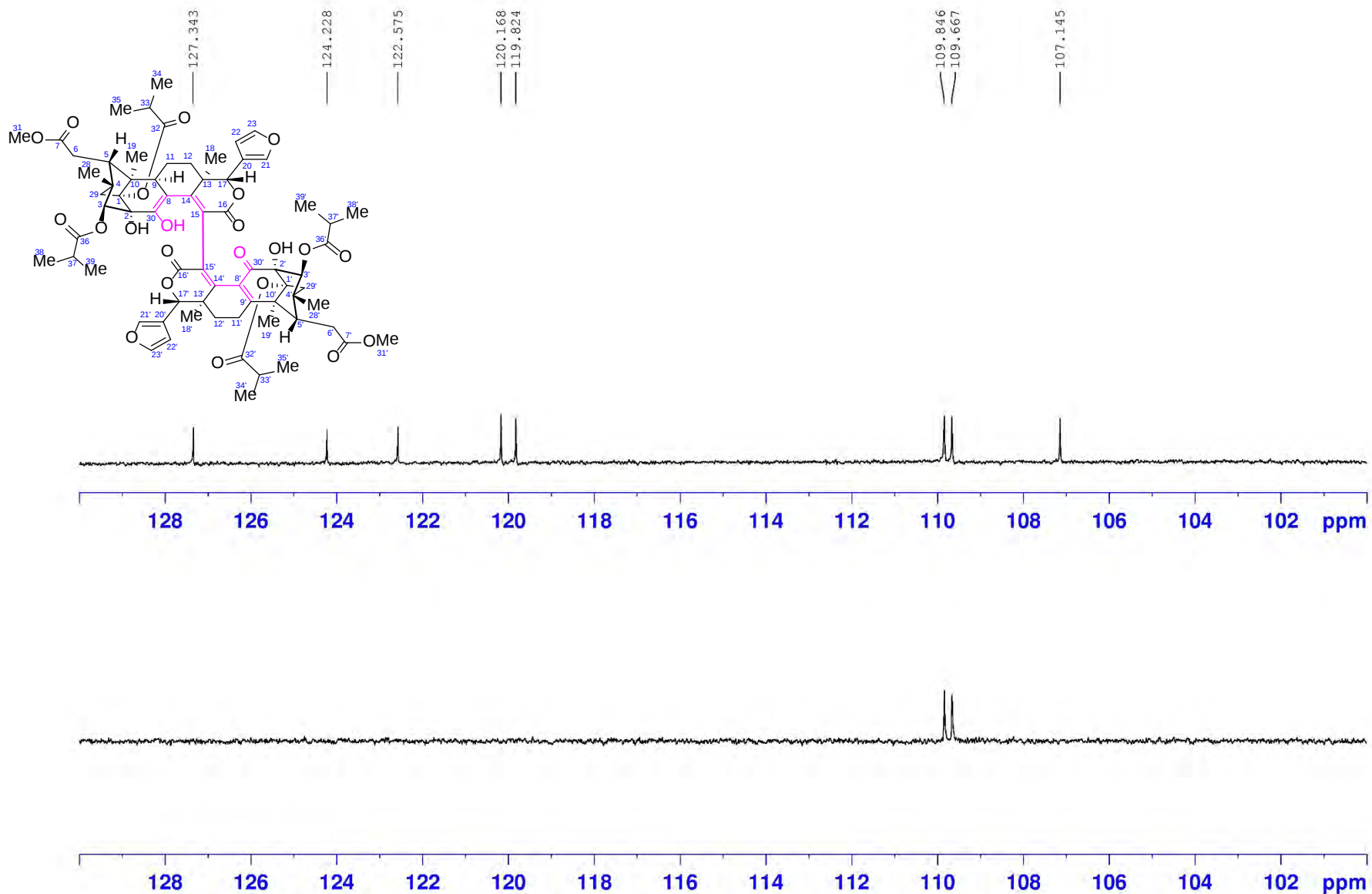
```

===== CHANNEL f1 =====
SFO1          100.6233324
NUC1           13C
P1            10.00
P13           2000.00
SI            32768
SF            100.6127685
WDW            EM
SSB            0
LB            1.00
GB            0
PC            1.40
    
```

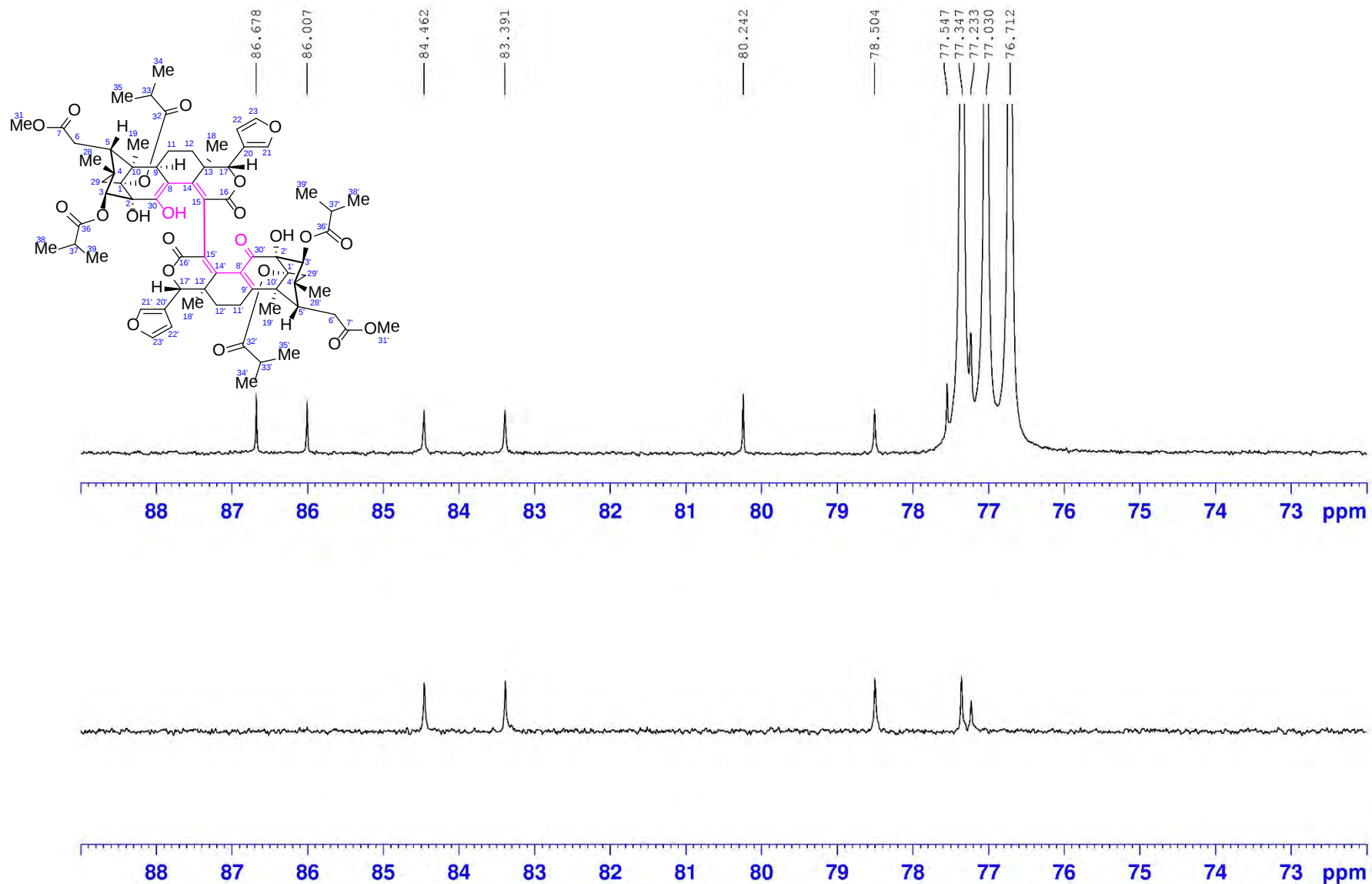
DEPT135 (100 MHz) spectrum of compound **4'** in CDCl₃



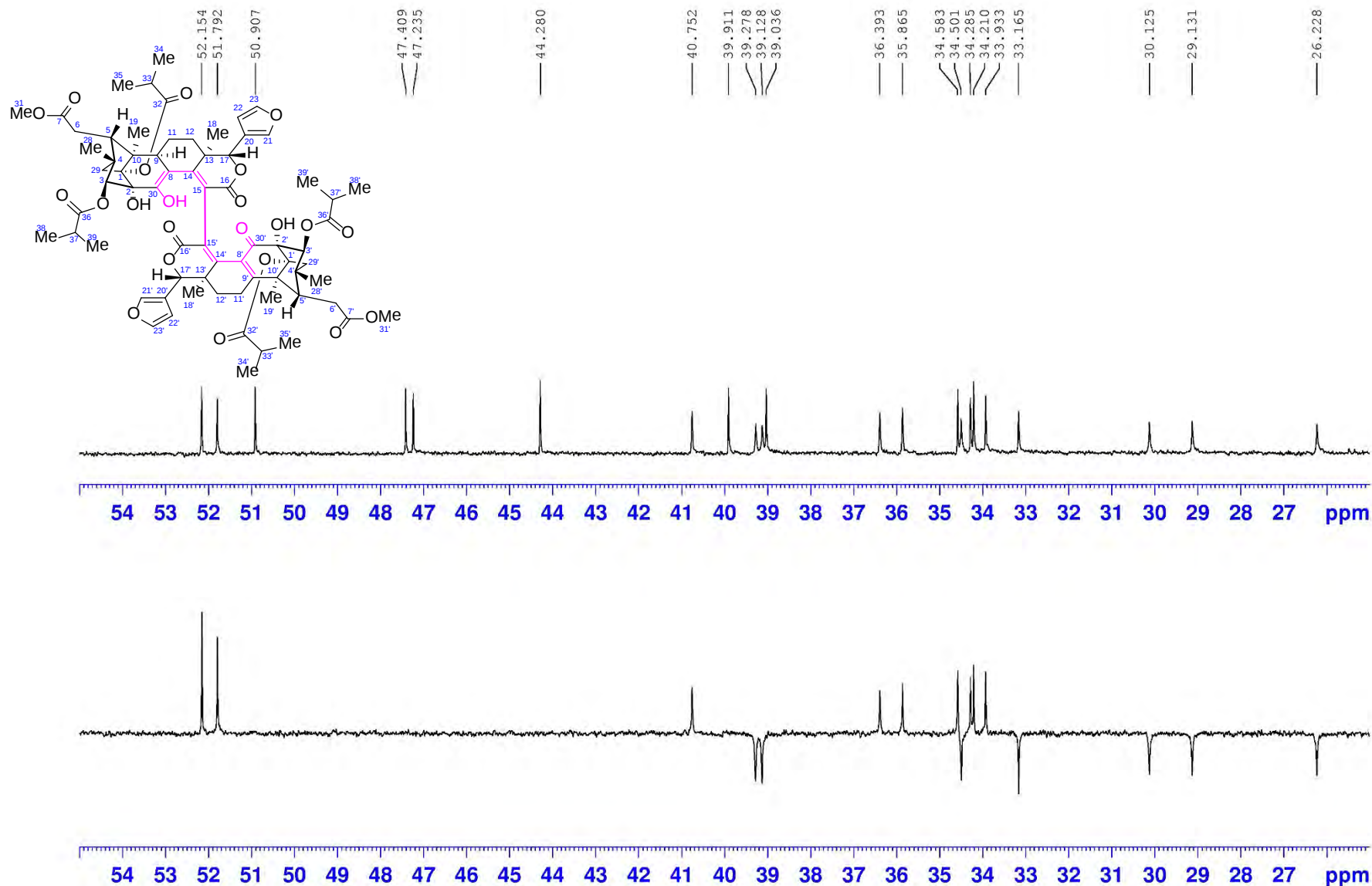
DEPT135 (100 MHz) spectrum of compound **4'** in CDCl₃



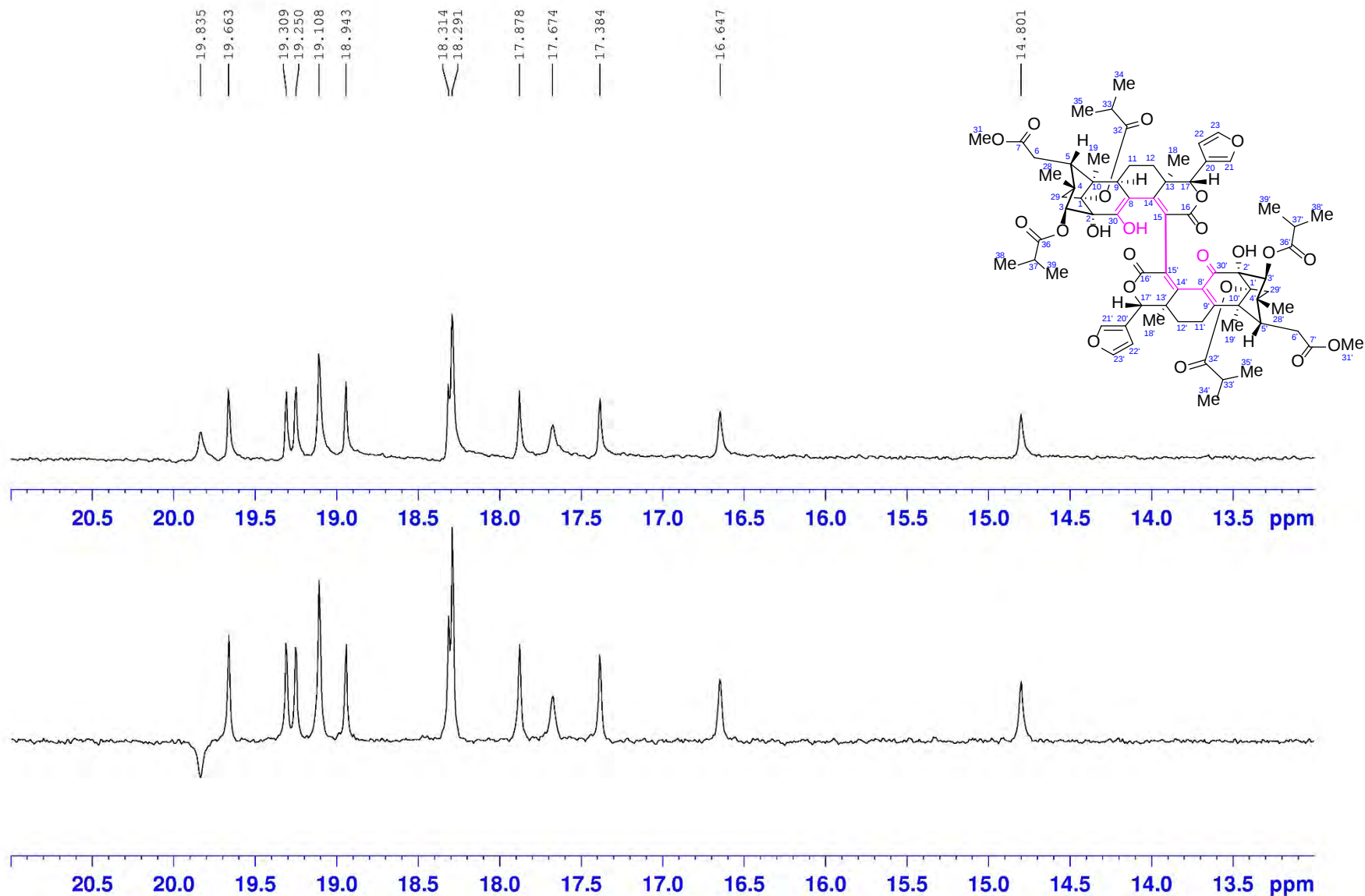
DEPT135 (100 MHz) spectrum of compound **4'** in CDCl₃



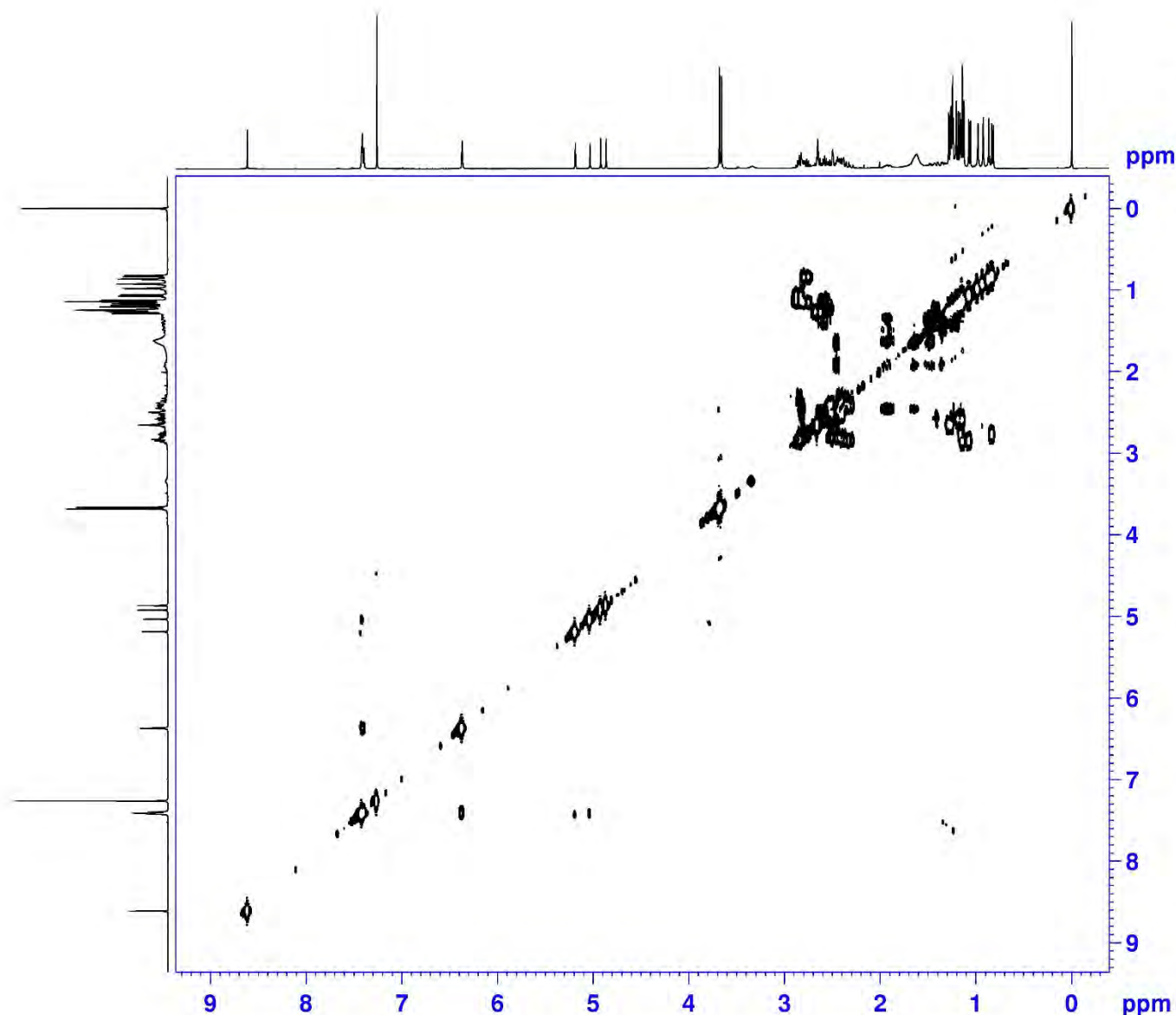
DEPT135 (100 MHz) spectrum of compound **4'** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **4'** in CDCl₃



^1H - ^1H COSY (400 MHz) spectrum of compound **4'** in CDCl_3



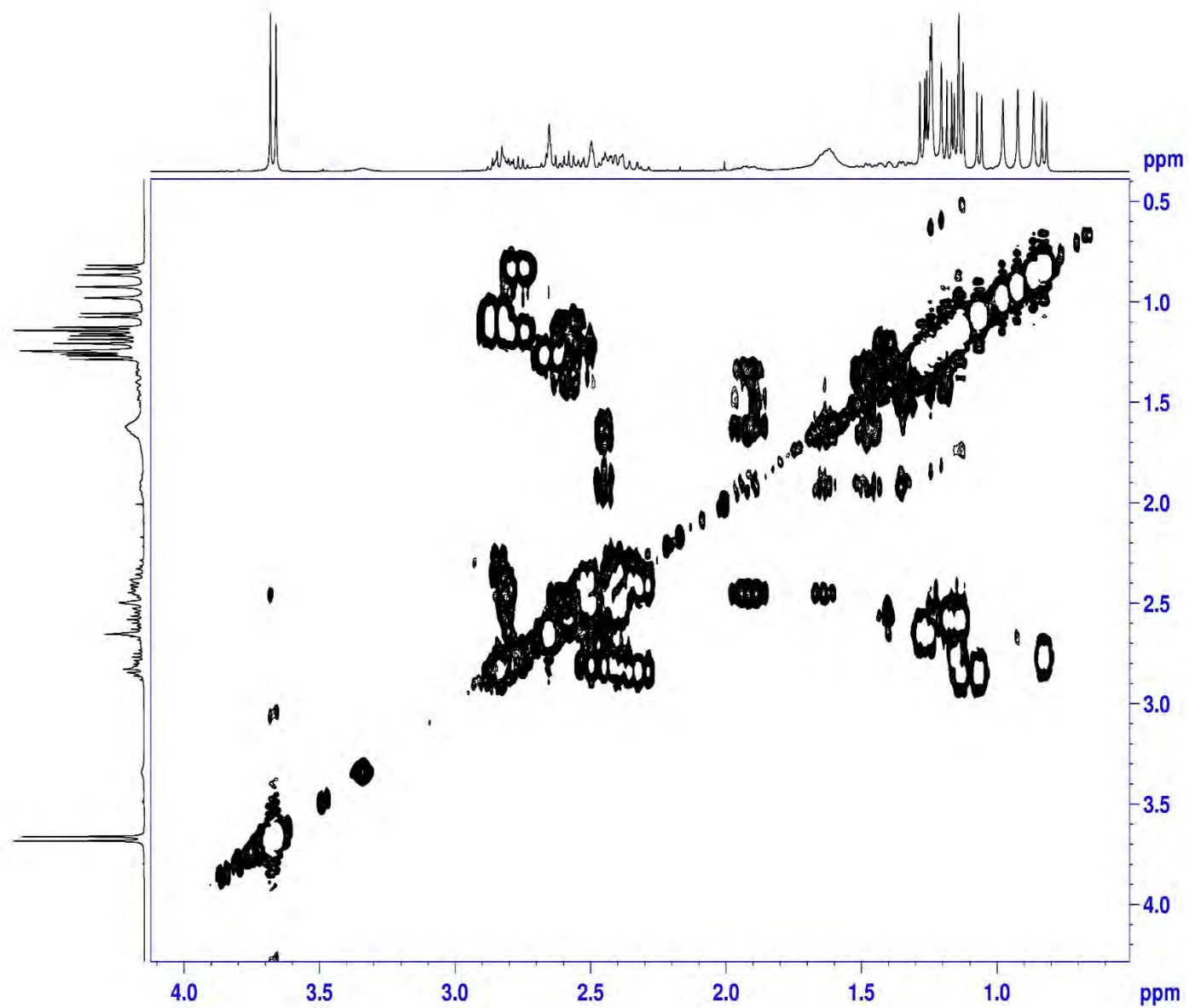
```

NAME                lws-37-6
EXPNO                4
PROCNO              1
Date_               20160613
Time                13.28
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             cosygpppqf
TD                  2048
SOLVENT             CDCl3
NS                   32
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
  
```

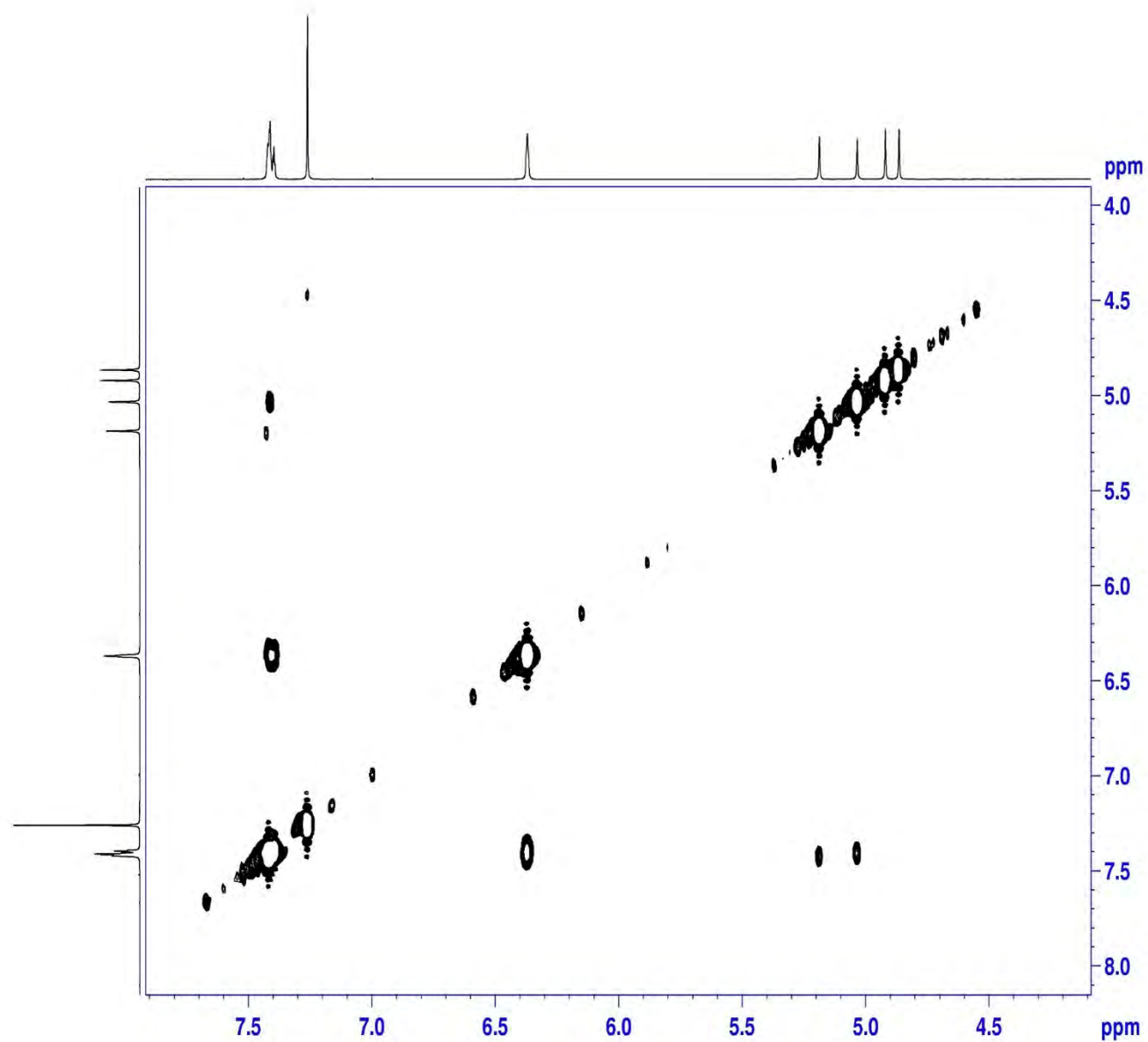
```

===== CHANNEL f1 =====
SFO1                400.1318006 M
NUC1                 1H
P0                   11.50 u
P1                   11.50 u
P17                  2500.00 u
ND0                   1
TD                   128
SFO1                400.1318 M
FIDRES              30.517578 H
SW                   9.762 p
FnMODE              QF
SI                   1024
SF                  400.1300085 M
WDW                  QSINE
SSB                   0
LB                   0.00 H
GB                   0
PC                   1.40
SI                   1024
MC2                  QF
SF                  400.1300085 M
WDW                  QSINE
  
```

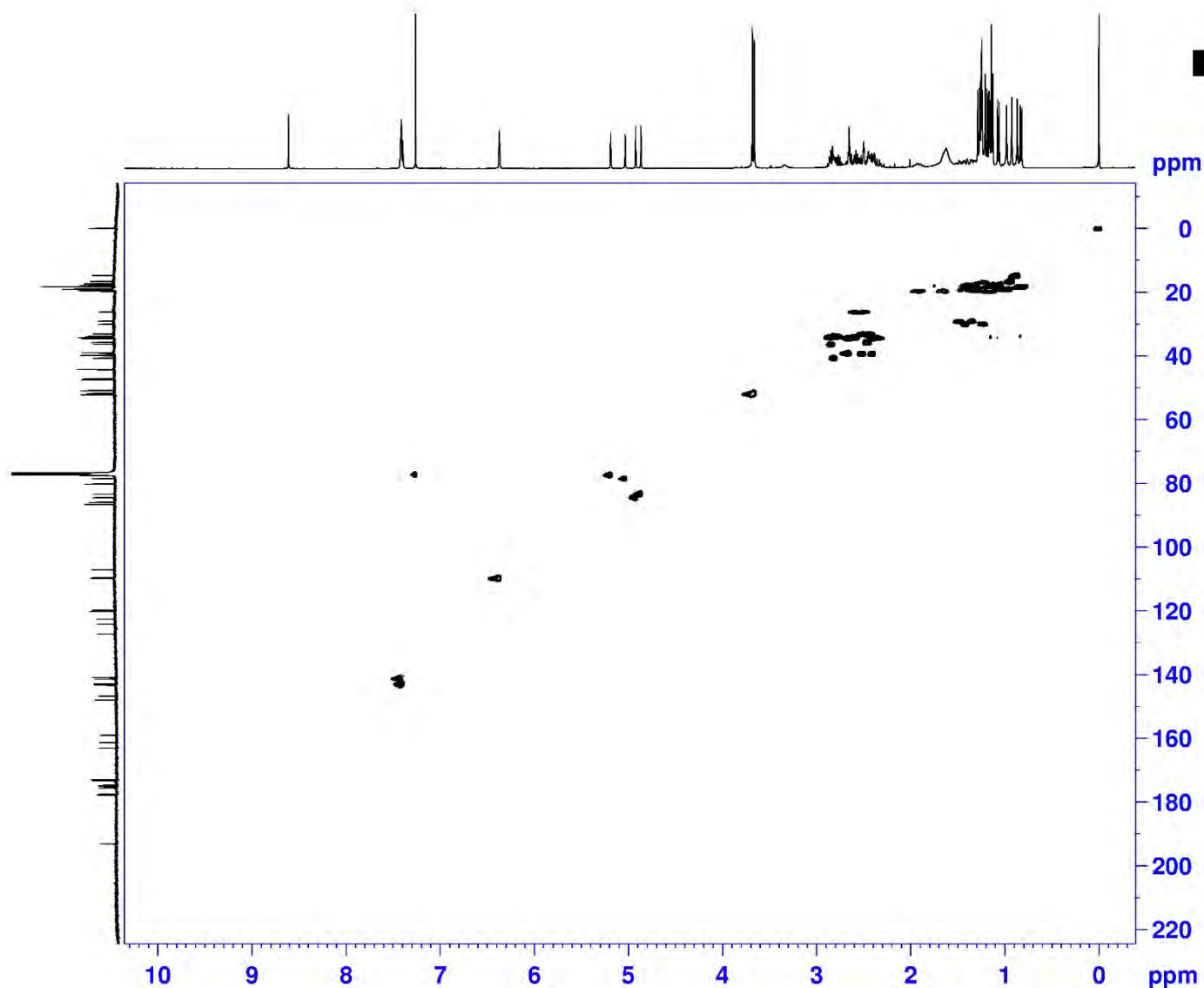
^1H - ^1H COSY (400 MHz) spectrum of compound **4'** in CDCl_3



^1H - ^1H COSY (400 MHz) spectrum of compound **4'** in CDCl_3



HSQC (400 MHz) spectrum of compound 4' in CDCl₃

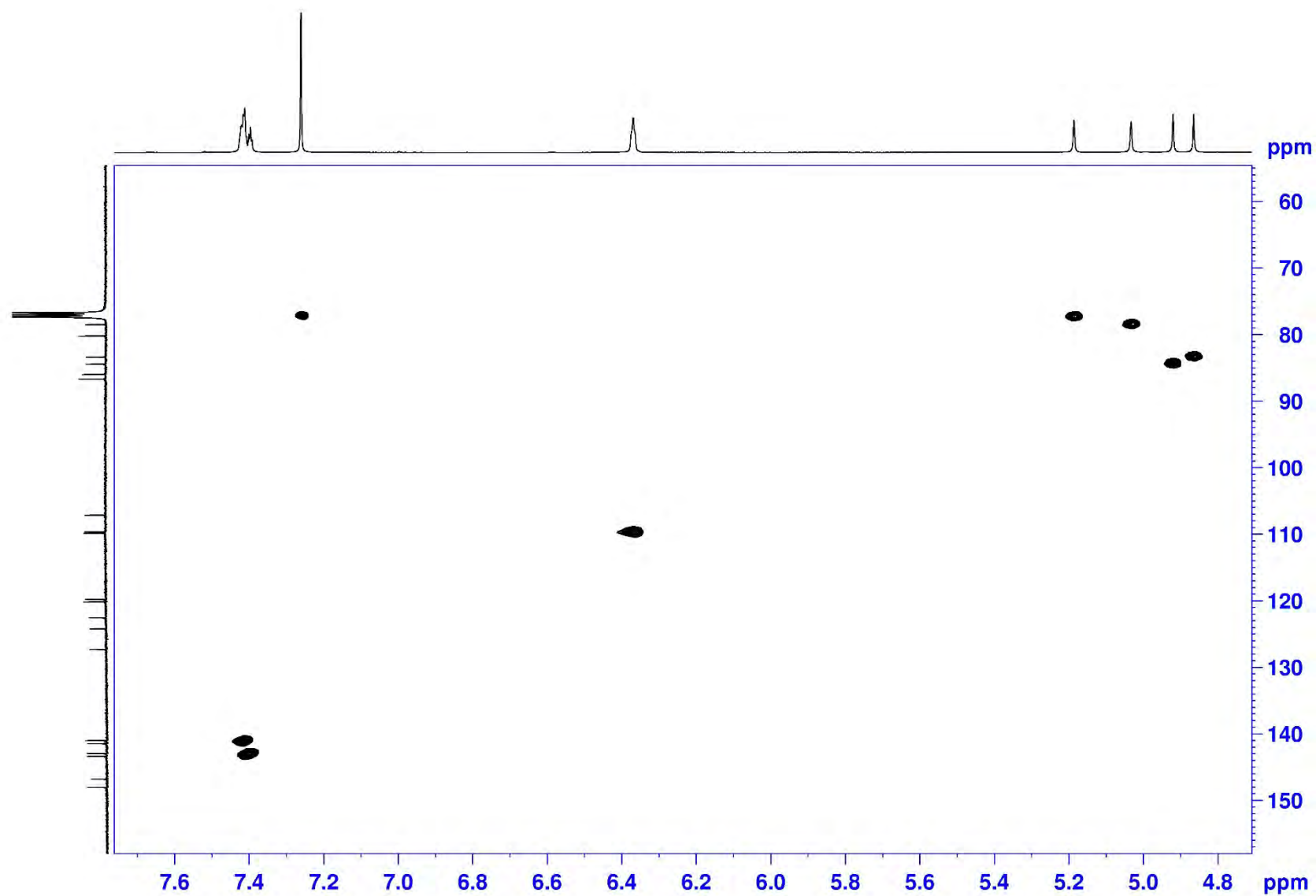


```

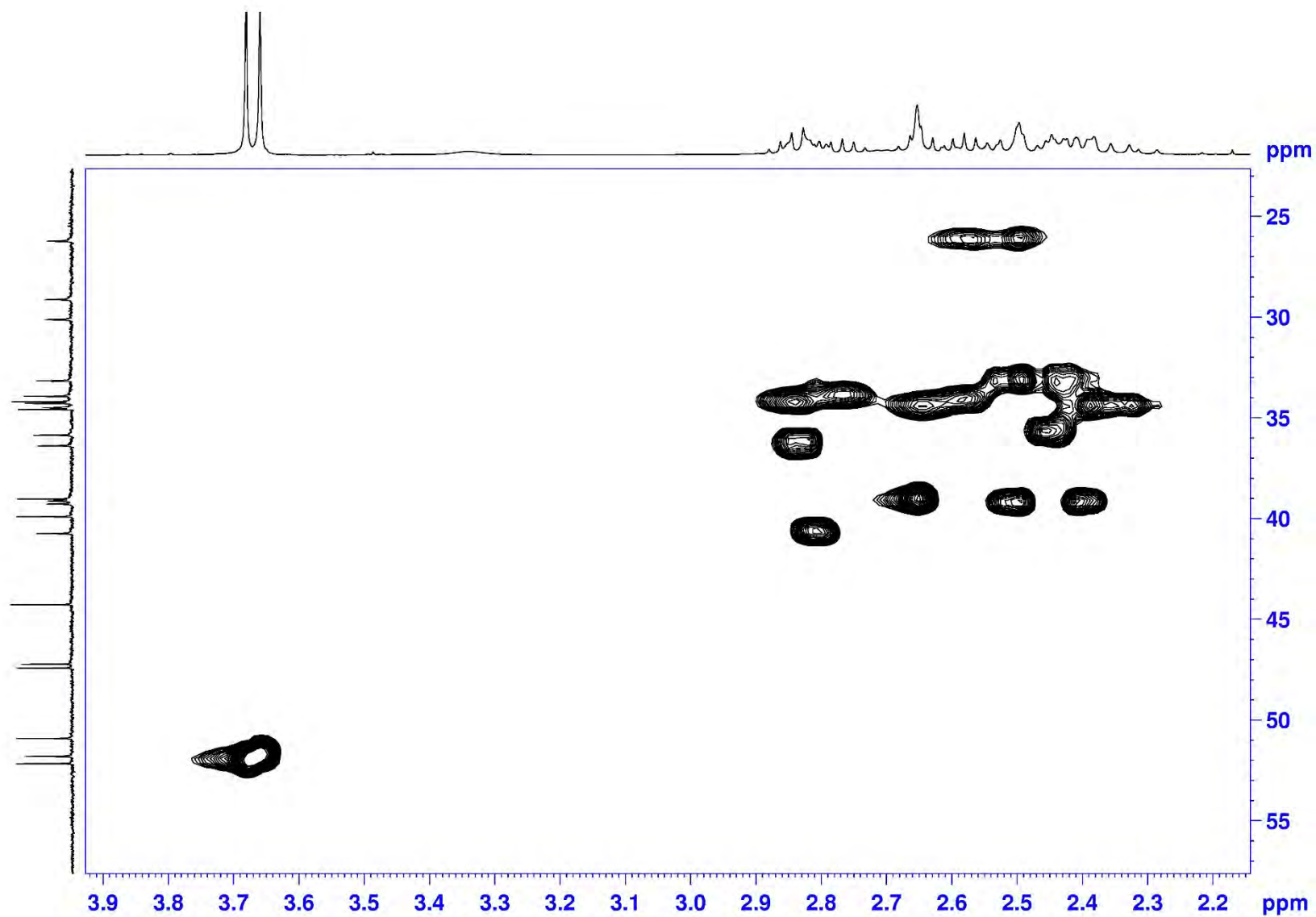
NAME                lws-37-6
EXPNO                5
PROCNO              1
Date_               20160613
Time                16.00
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             hsqcetgpsi2
TD                  1024
SOLVENT             CDCl3
NS                   32
DS                   16
SWH                 4302.926 H
FIDRES              4.202076 H
AQ                  0.1190388 s
RG                   208.5
DW                  116.200 u
DE                   10.00 u
TE                   297.0 K
CNST2               145.0000000
D0                   0.00000300 s
D1                   1.46497905 s
D4                   0.00172414 s
D11                  0.03000000 s
D16                  0.00020000 s
D24                  0.00086207 s
IN0                  0.00002080 s
ZGOPTNS

===== CHANNEL f1 =====
SF01                 400.1320007 M
NUC1                  1H
P1                    11.50 u
P2                    23.00 u
P28                   0.00 u
ND0                   2
TD                   256
SF01                 100.6233 M
FIDRES              93.900238 H
SW                   238.896 p
FnMODE              Echo-Antiecho
SI                   1024
SF                   400.1300079 M
WDW                  QSINE
SSB                   2
LB                   0.00 H
GB                   0
PC                   1.40
SI                   1024
MC2                  echo-antiecho
SF                   100.6127685 M
WDW                  QSINE
SSB                   2
    
```

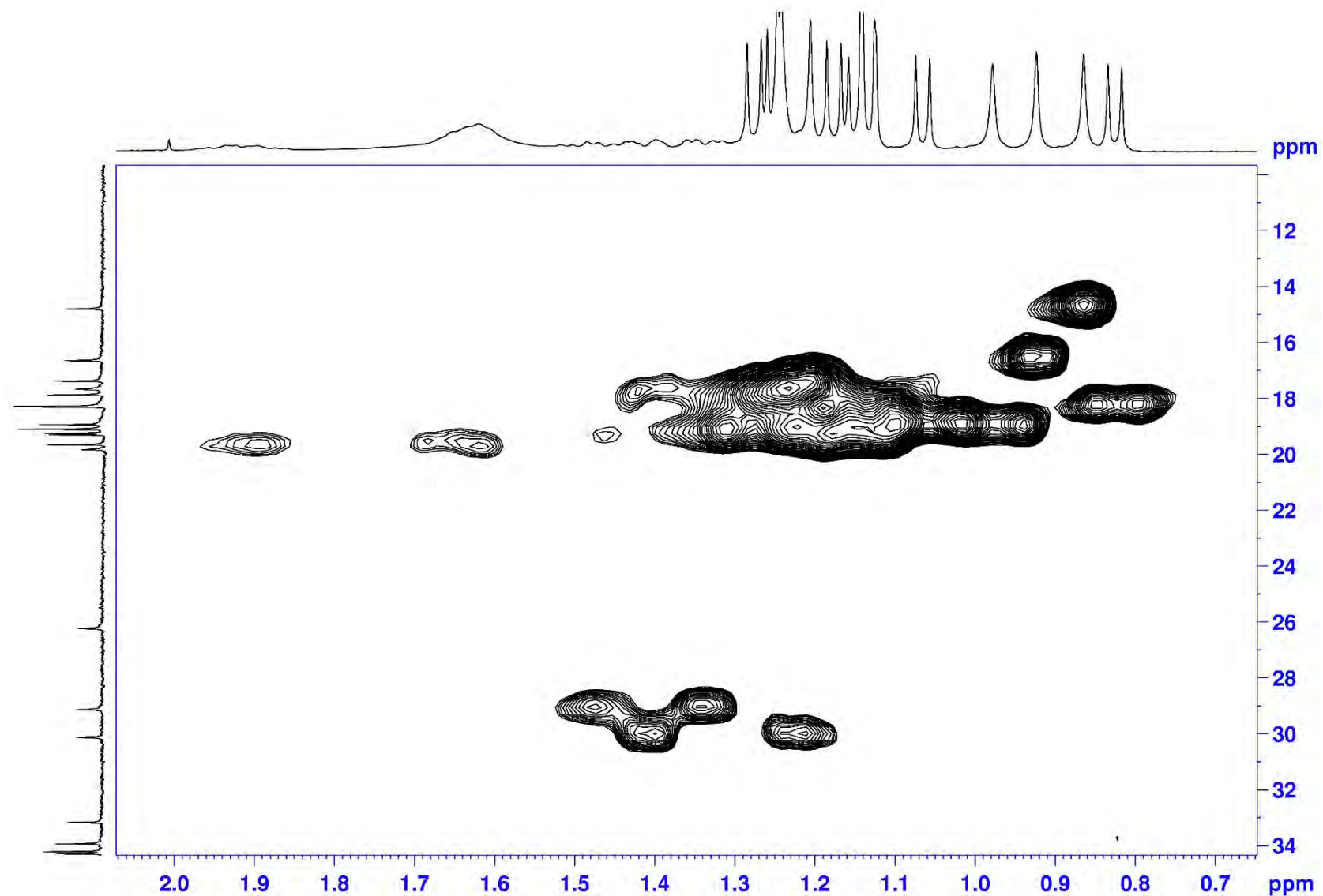
HSQC (400 MHz) spectrum of compound **4'** in CDCl₃



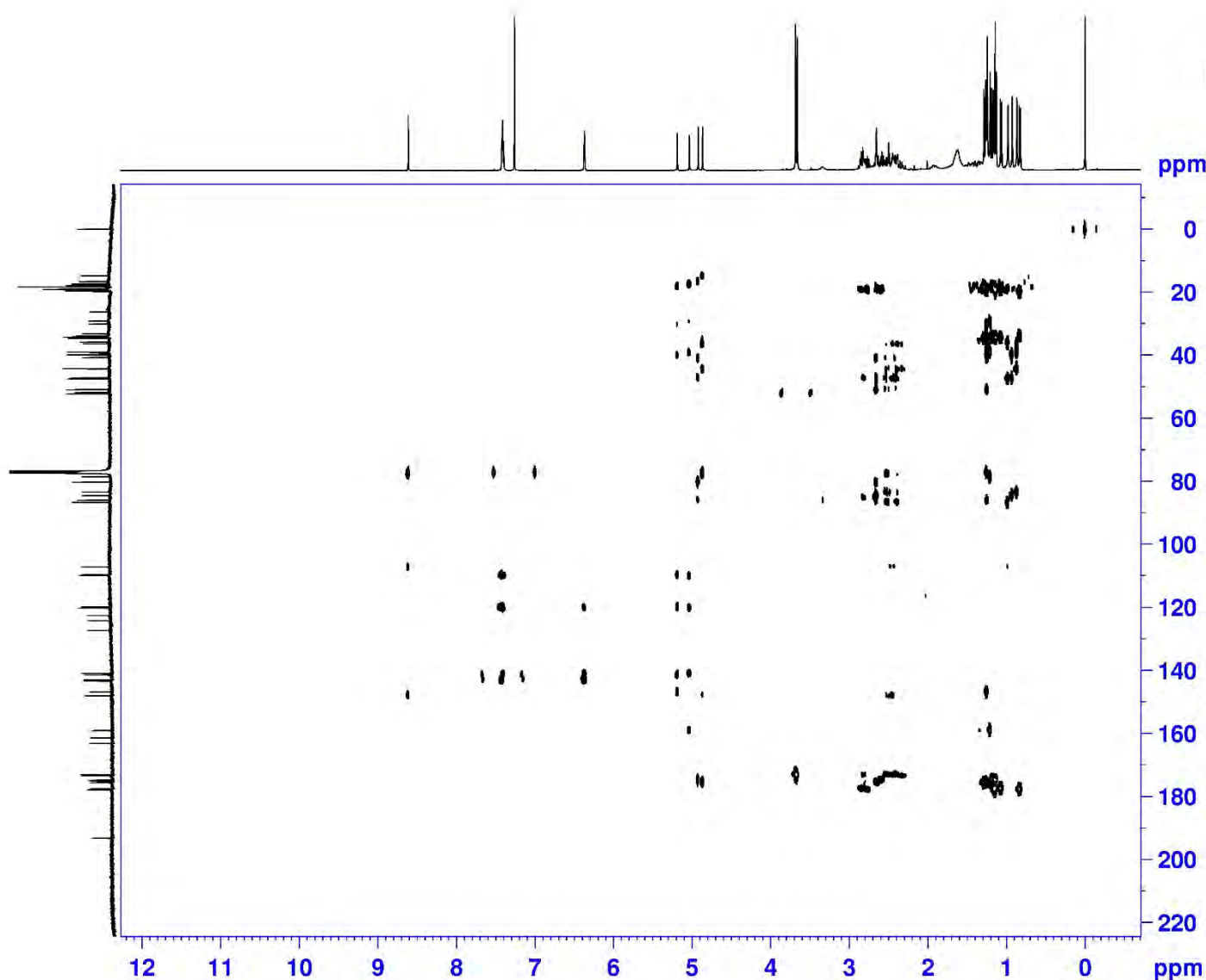
HSQC (400 MHz) spectrum of compound **4'** in CDCl₃



HSQC (400 MHz) spectrum of compound **4'** in CDCl₃



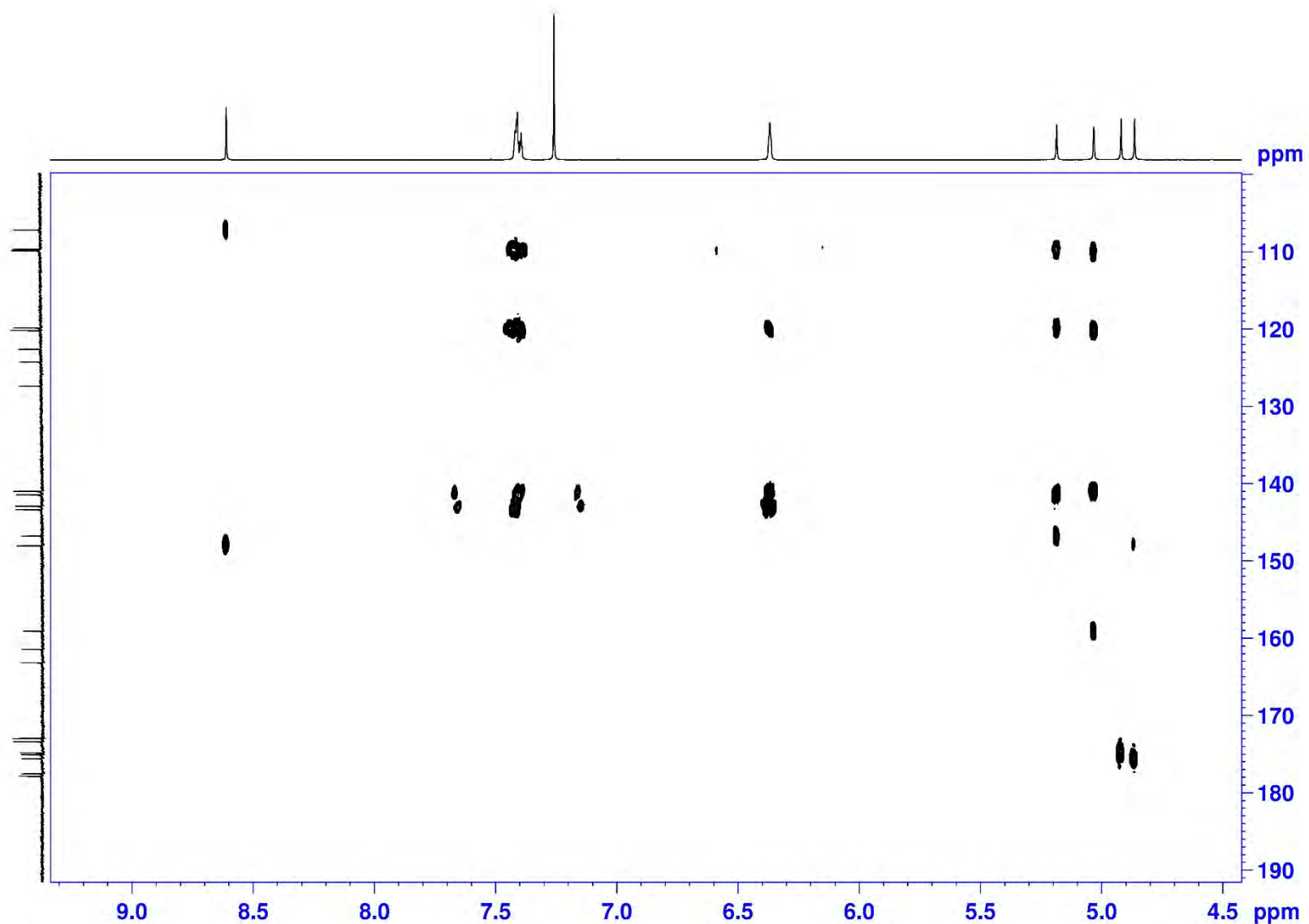
HMBC (400 MHz) spectrum of compound 4' in CDCl₃



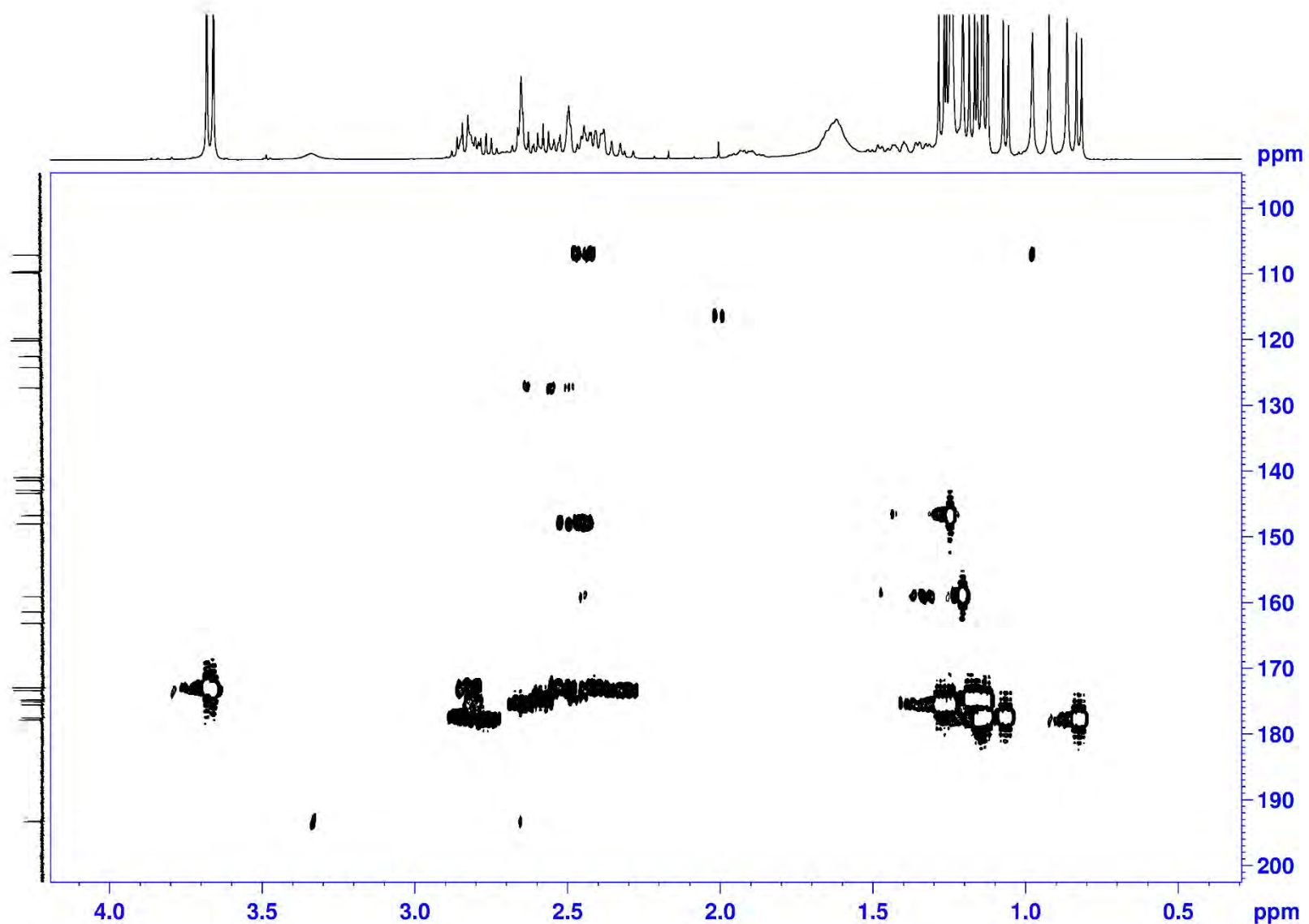
NAME lws-37-6
 EXPNO 6
 PROCNO 1
 Date_ 20160613
 Time 19.48
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG hmbcgp1pndqf
 TD 4096
 SOLVENT CDCl3
 NS 256
 DS 16
 SWH 5197.505 Hz
 FIDRES 1.268922 Hz
 AQ 0.3940852 sec
 RG 208.5
 DW 96.200 usec
 DE 10.00 usec
 TE 297.0 K
 CNST2 145.0000000
 CNST13 10.0000000
 D0 0.00000300 sec
 D1 1.50000000 sec
 D2 0.00344828 sec
 D6 0.05000000 sec
 D16 0.00020000 sec
 IN0 0.00002080 sec

===== CHANNEL f1 =====
 SFO1 400.1323208 MHz
 NUC1 1H
 P1 11.50 usec
 P2 23.00 usec
 ND0 2
 TD 128
 SFO1 100.6233 MHz
 FIDRES 187.800476 Hz
 SW 238.896 ppm
 FnmODE QF
 S1 2048
 SF 400.1300096 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40
 SI 1024
 MC2 QF
 SF 100.6127685 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz

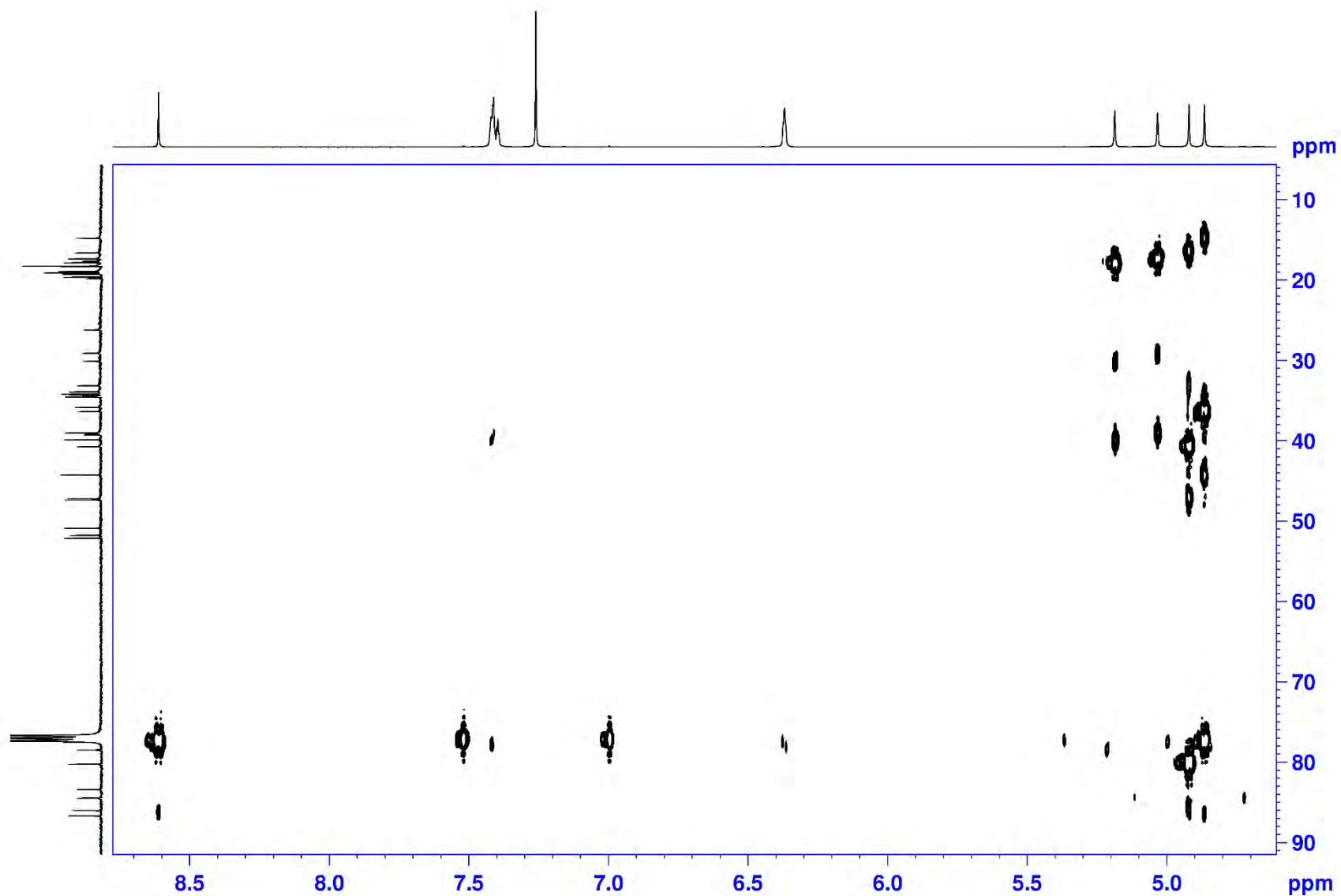
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



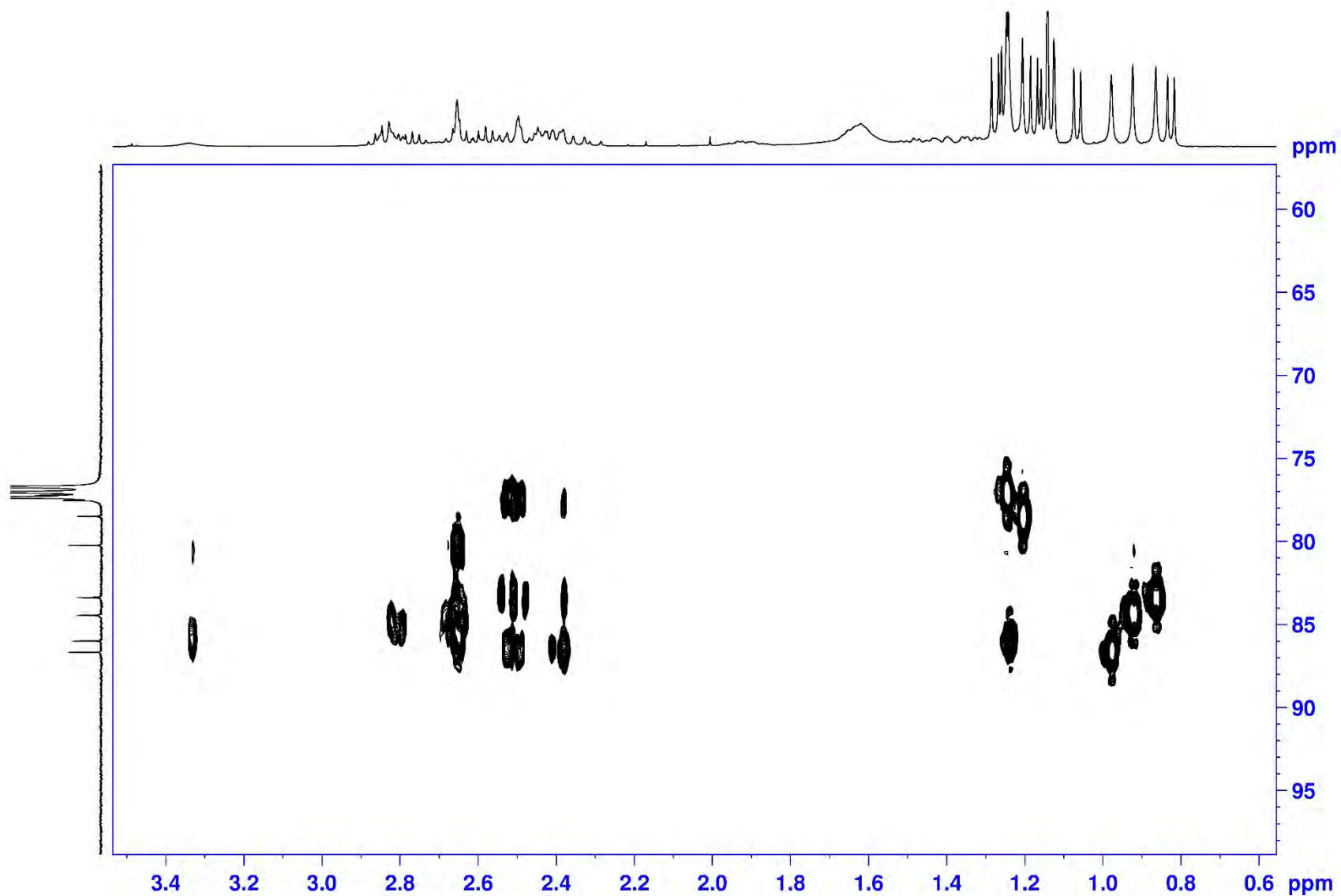
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



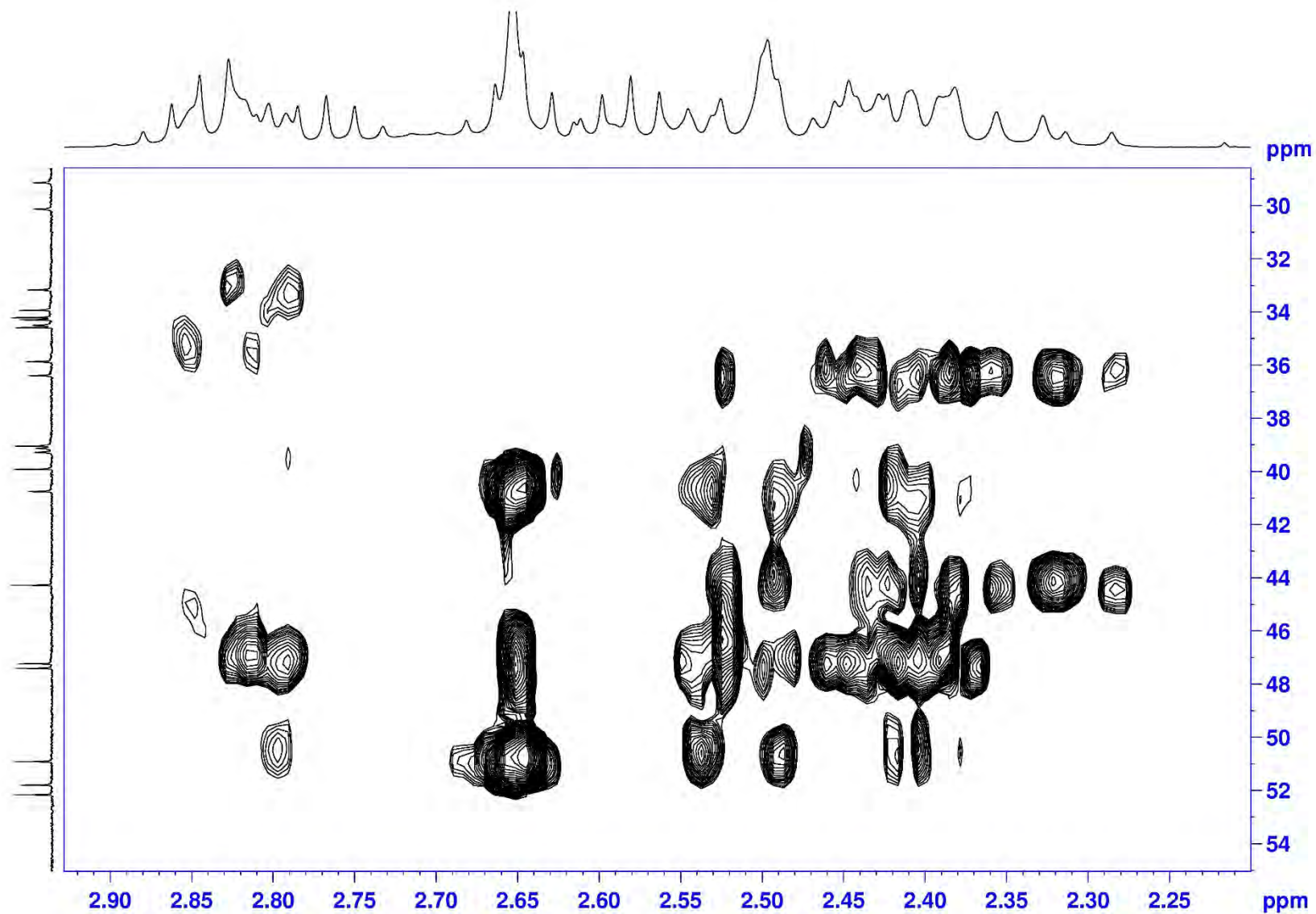
HMBC (400 MHz) spectrum of compound **4'** in CDCl_3



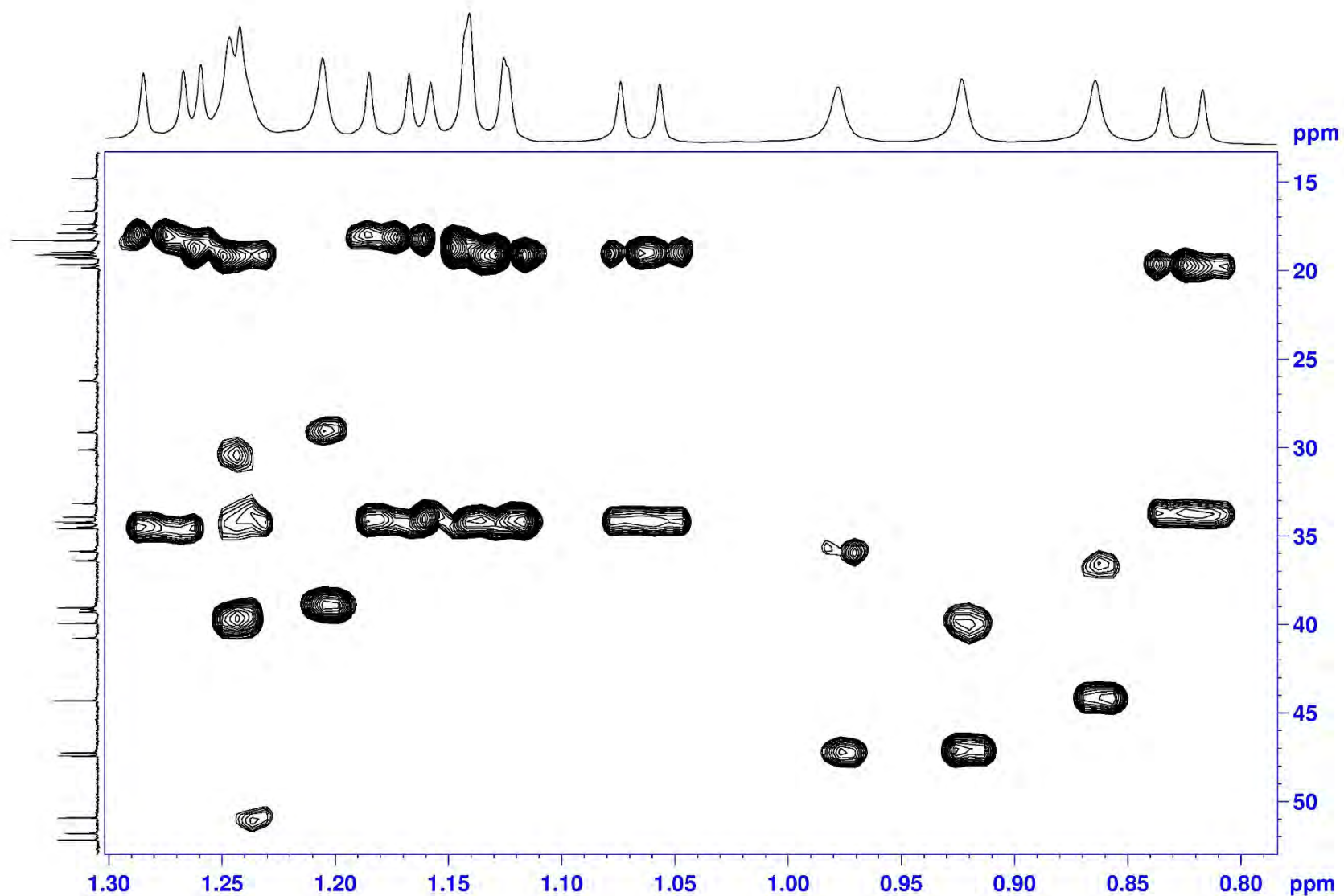
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



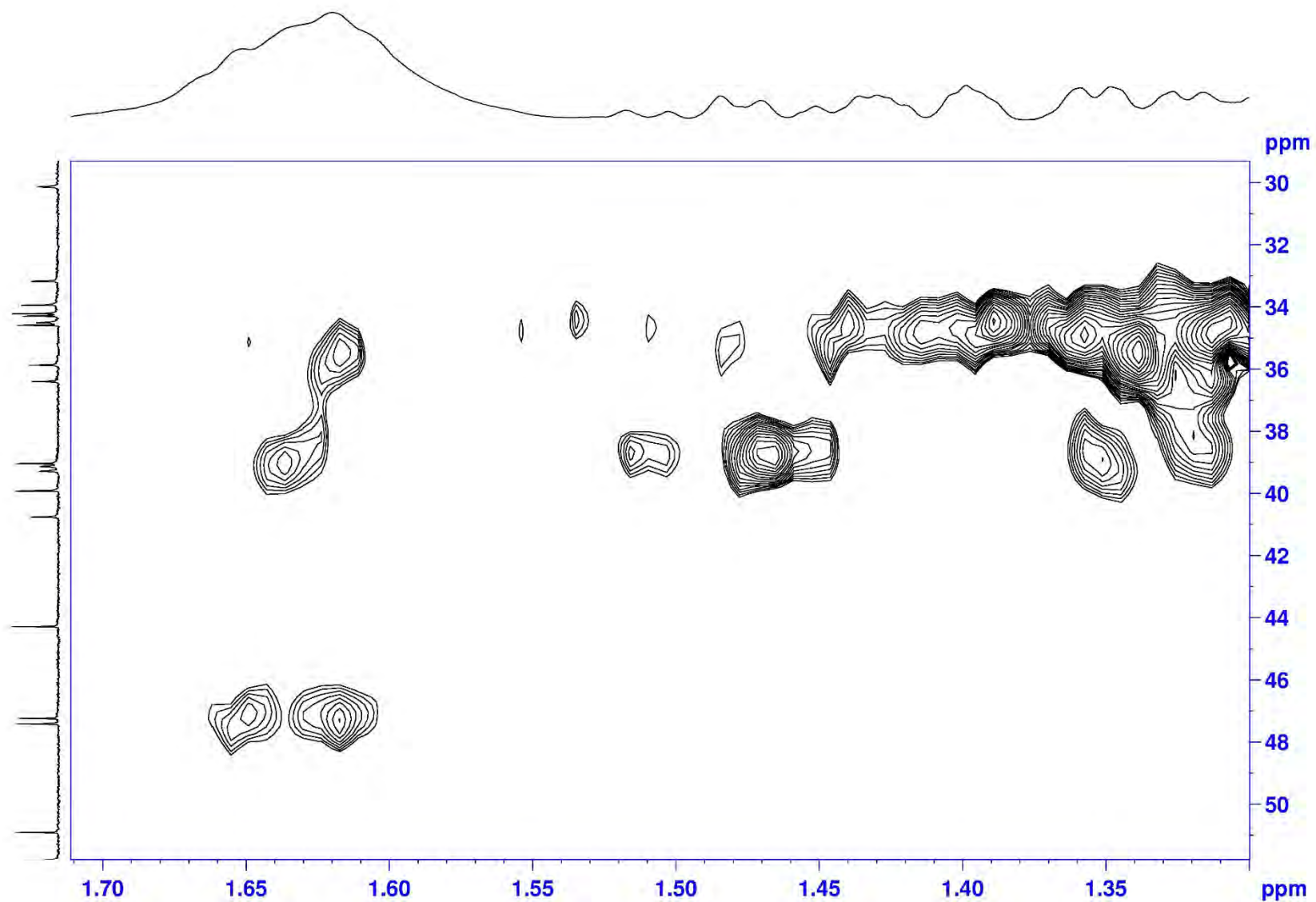
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



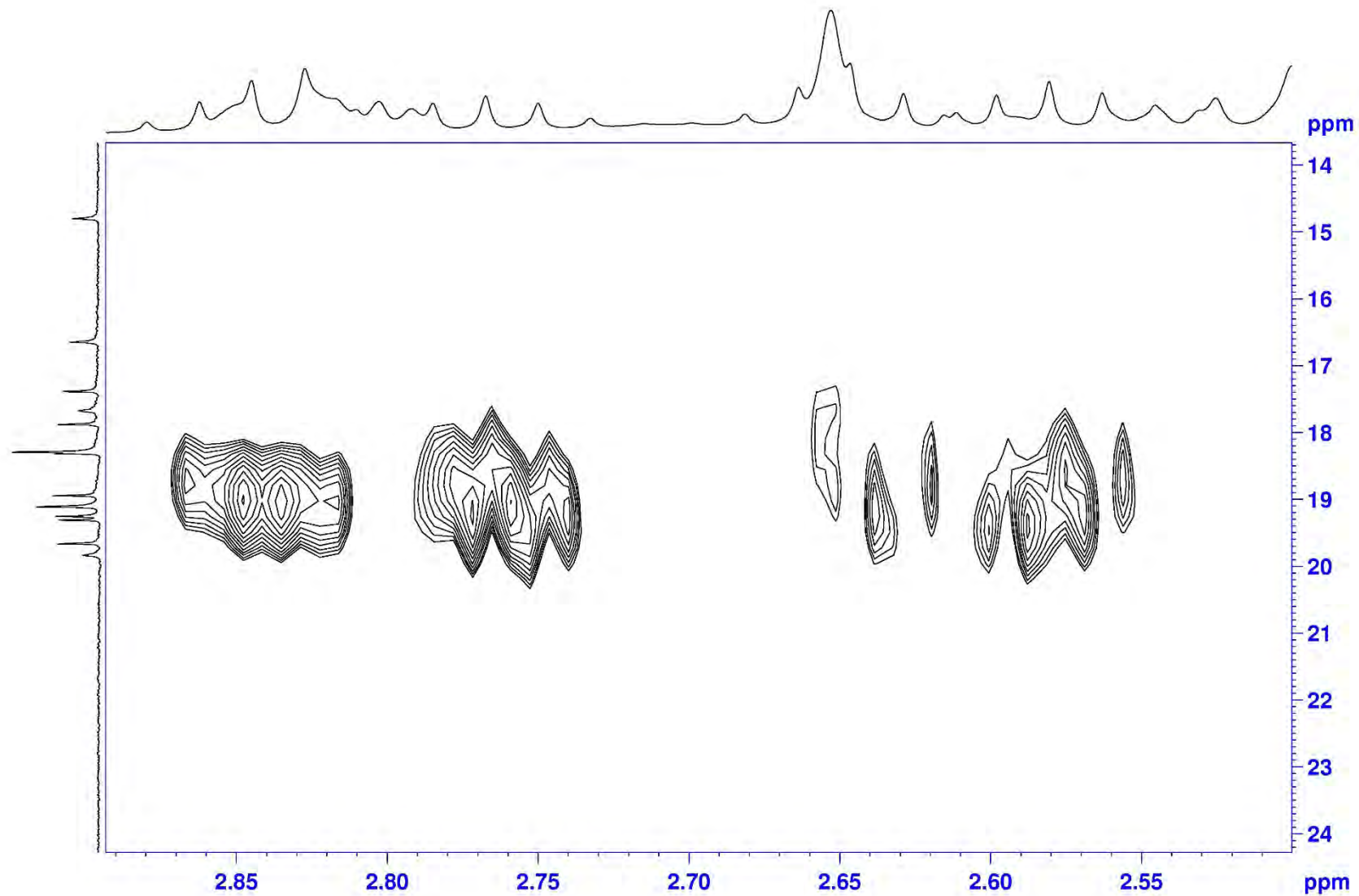
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



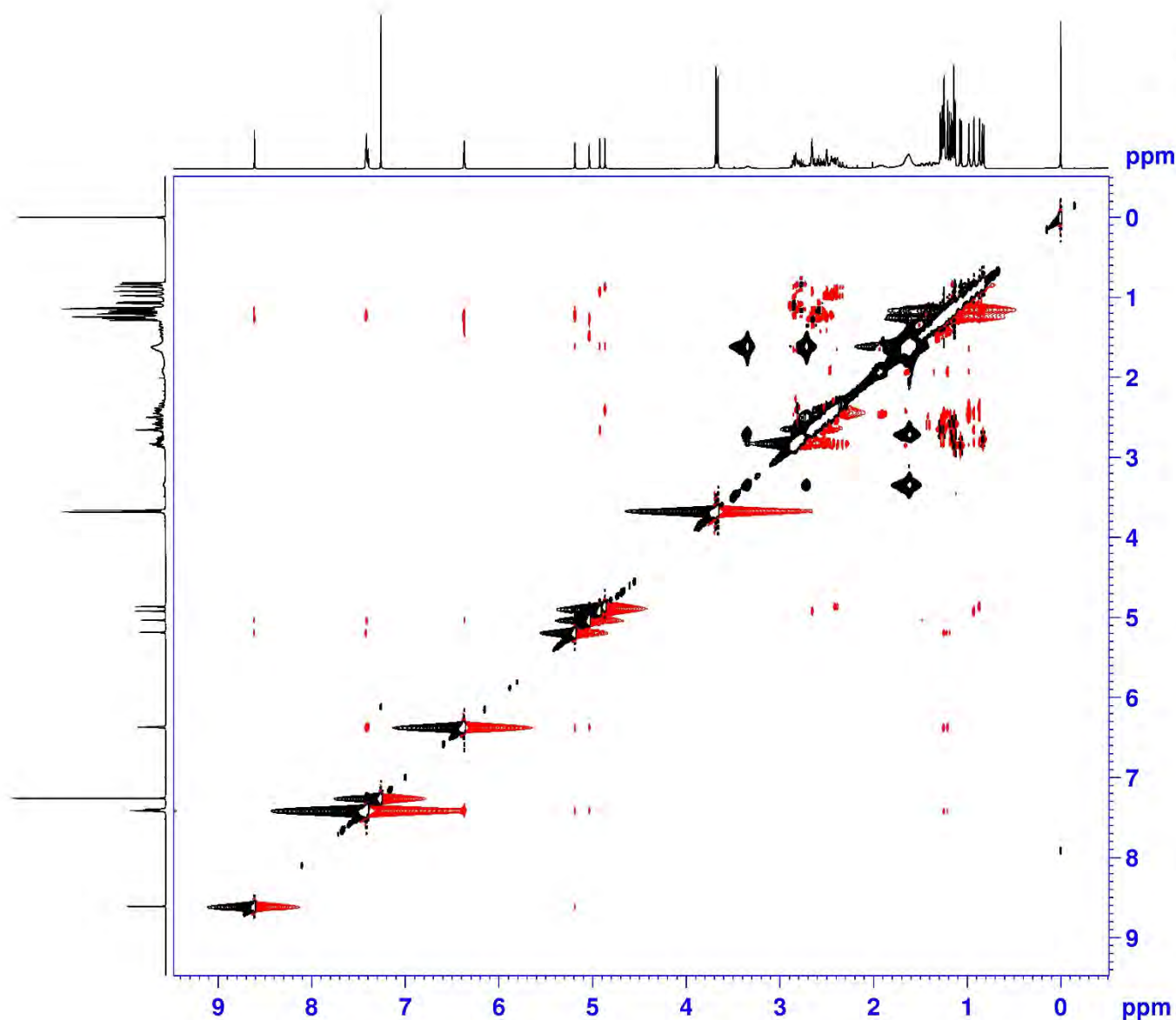
HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



HMBC (400 MHz) spectrum of compound **4'** in CDCl₃



NOESY (400 MHz) spectrum of compound 4' in CDCl₃



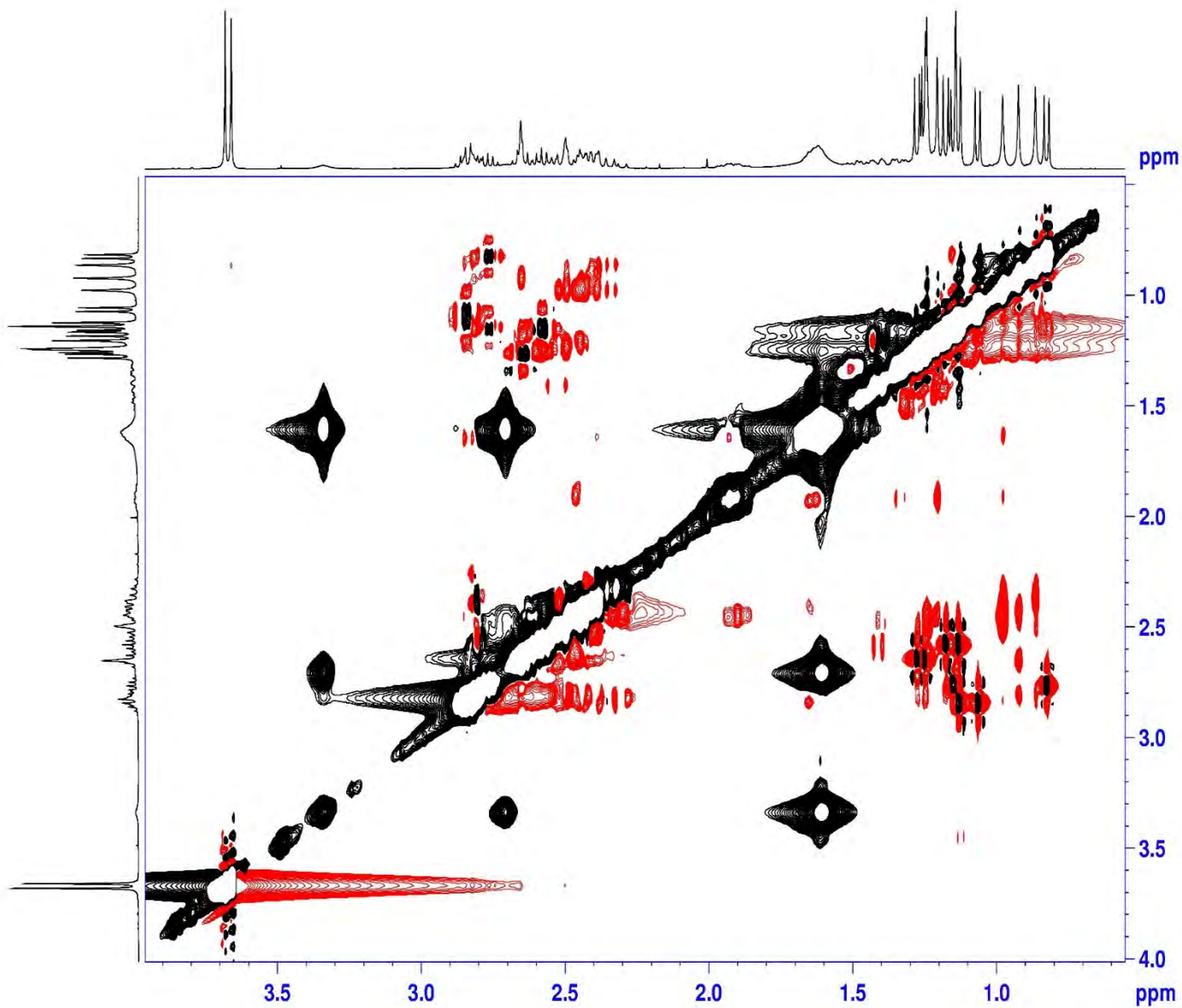
```

NAME          lws-37-6
EXPNO          7
PROCNO         1
Date_          20160613
Time           7.31
INSTRUM        spect
PROBHD         5 mm CPPBBO BB
PULPROG        noesygpphph
TD             2048
SOLVENT        CDC13
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             0.00011036 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
    
```

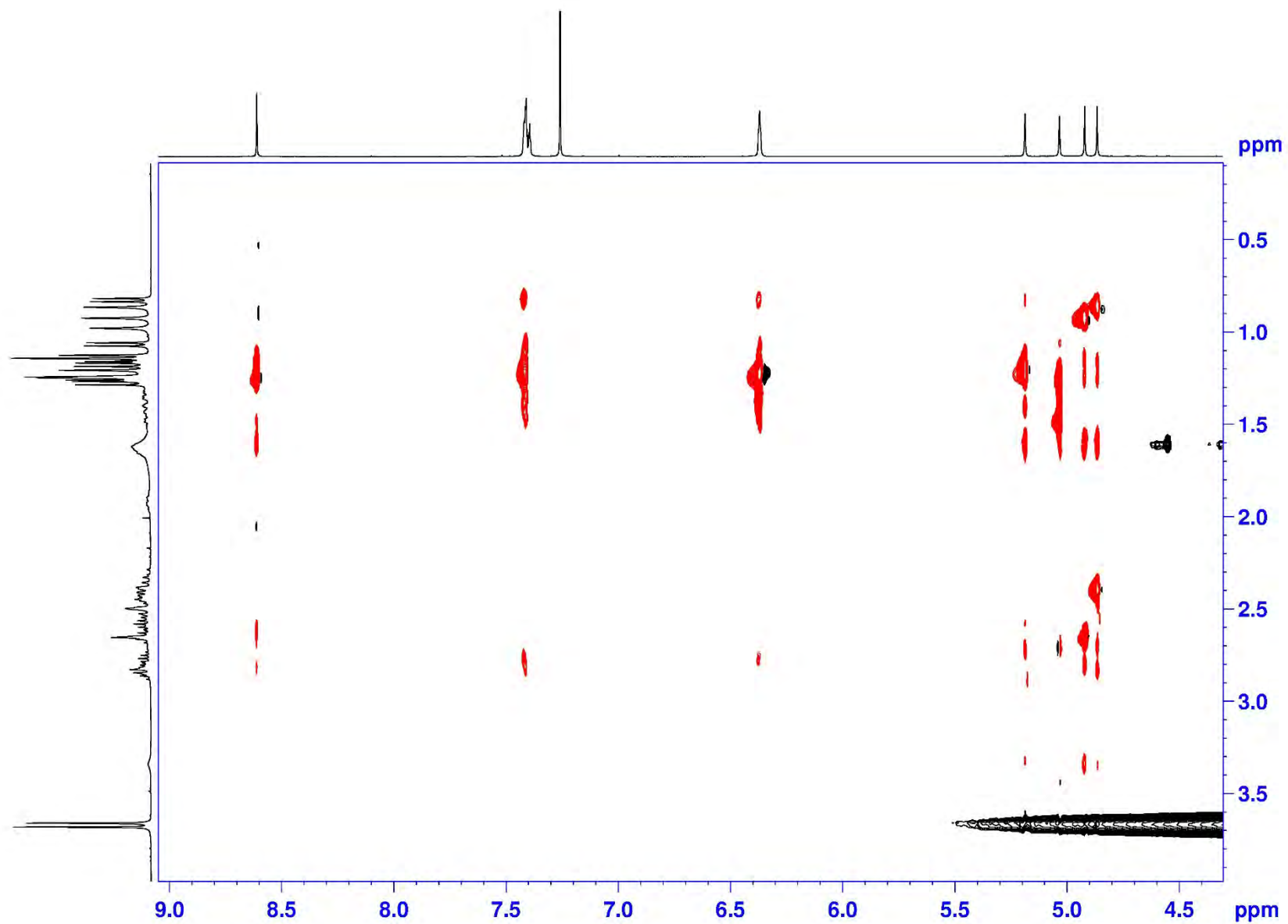
```

===== CHANNEL f1 =====
SFO1          400.1318006 M
NUC1           1H
P1             11.50 u
P2             23.00 u
P17            2500.00 u
ND0            1
TD             256
SFO1          400.1318 M
FIDRES         15.625000 H
SW             9.997 F
FnMODE         States-TPPI
SI             1024
SF            400.1300098 M
WDW            QSINE
SSB            2
LB             0.00 H
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF            400.1300098 M
    
```

NOESY (400 MHz) spectrum of compound **4'** in CDCl_3



NOESY (400 MHz) spectrum of compound **4'** in CDCl₃



NOESY (400 MHz) spectrum of compound **4'** in CDCl₃

