Supporting Information

Copies of HR-ESIMS of **1–10**, **4s/r**, **6s/r**, and **7s/r**; 1D and 2D NMR spectra of **1–10**; and ^{1}H NMR spectra of **4s/r**, **6s/r**, and **7s/r**.

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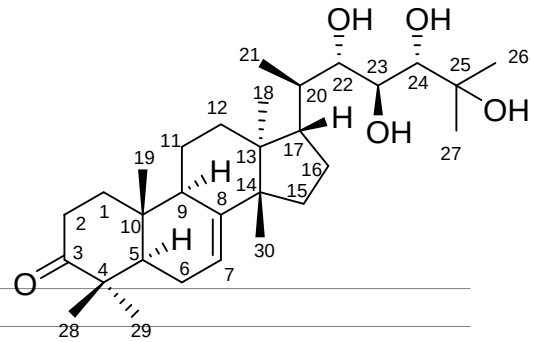
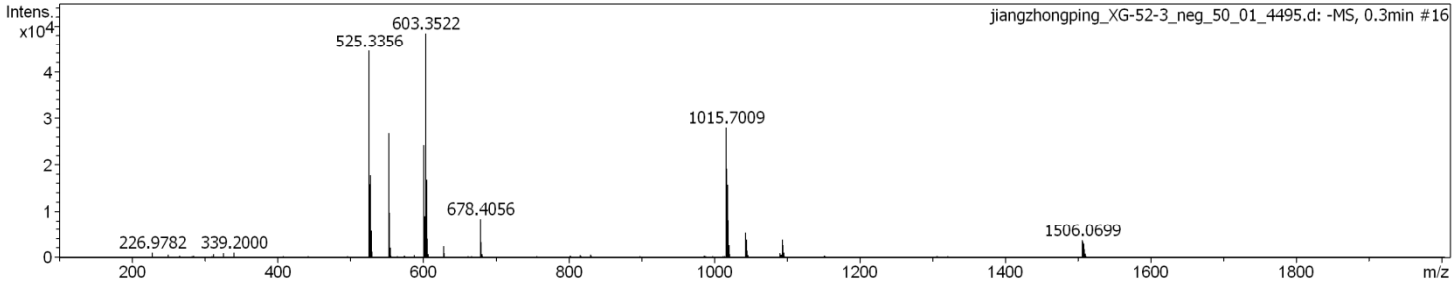
HR-ESIMS for compound **1**

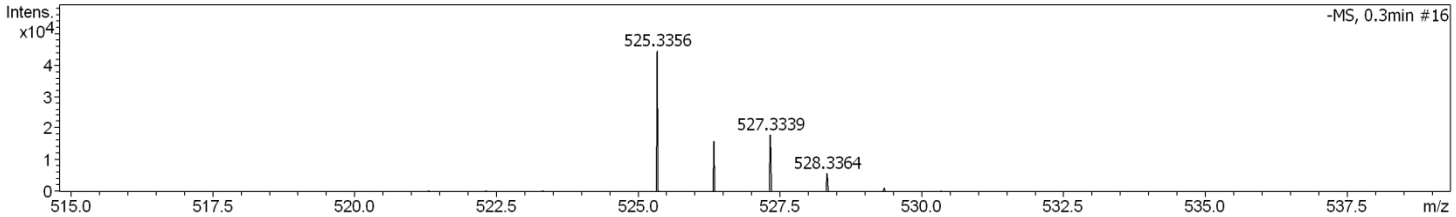


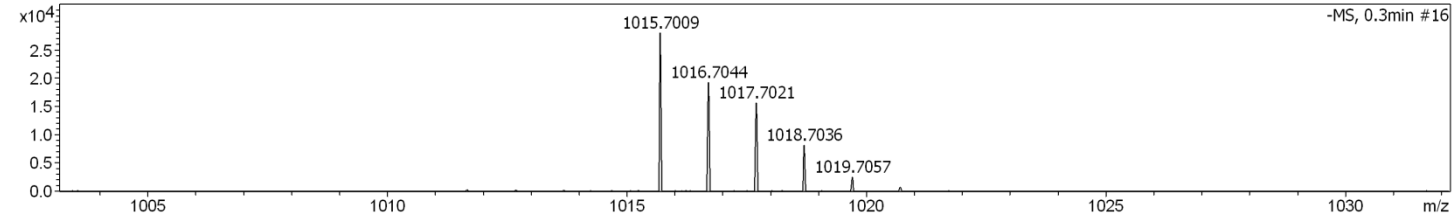
Generic Display Report

Analysis Info

Analysis Name	D:\Data\MS\data\201804\jiangzhongping_XG-52-3_neg_50_01_4495.d	Acquisition Date	4/3/2018 10:52:52 AM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_XG-52-3_neg	Instrument	maXis
Comment			







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HR-ESIMS for compound **1**

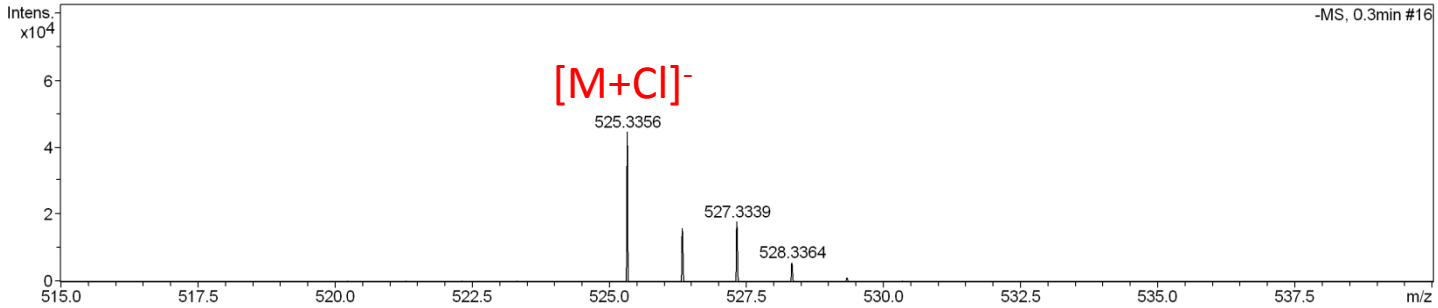
Mass Spectrum SmartFormula Report

Analysis Info

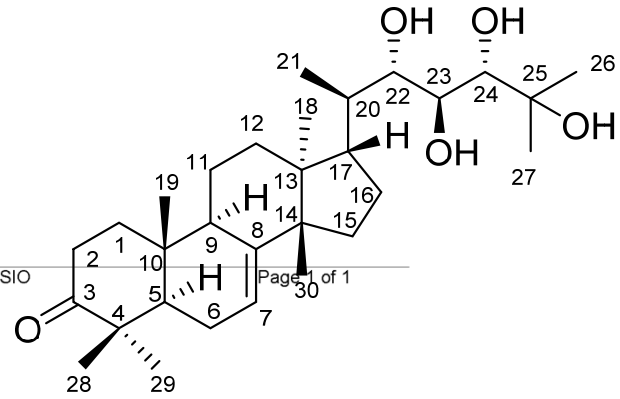
Analysis Name	D:\Data\MS\data\201804\jiangzhongping_XG-52-3_neg_50_01_4495.d	Acquisition Date	4/3/2018 10:52:52 AM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_XG-52-3_neg	Instrument	maXis 255552.00029
Comment			

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⁻ Conf	N-Rule
525.3356	1	C30H50ClO5	100.00	525.3352	-0.8	-0.4	14.0	5.5	even	ok
1015.7009	1	C60H100ClO10	100.00	1015.7011	0.1	0.1	10.4	10.5	even	ok



jiangzhongping_XG-52-3_neg_50_01_4495.d

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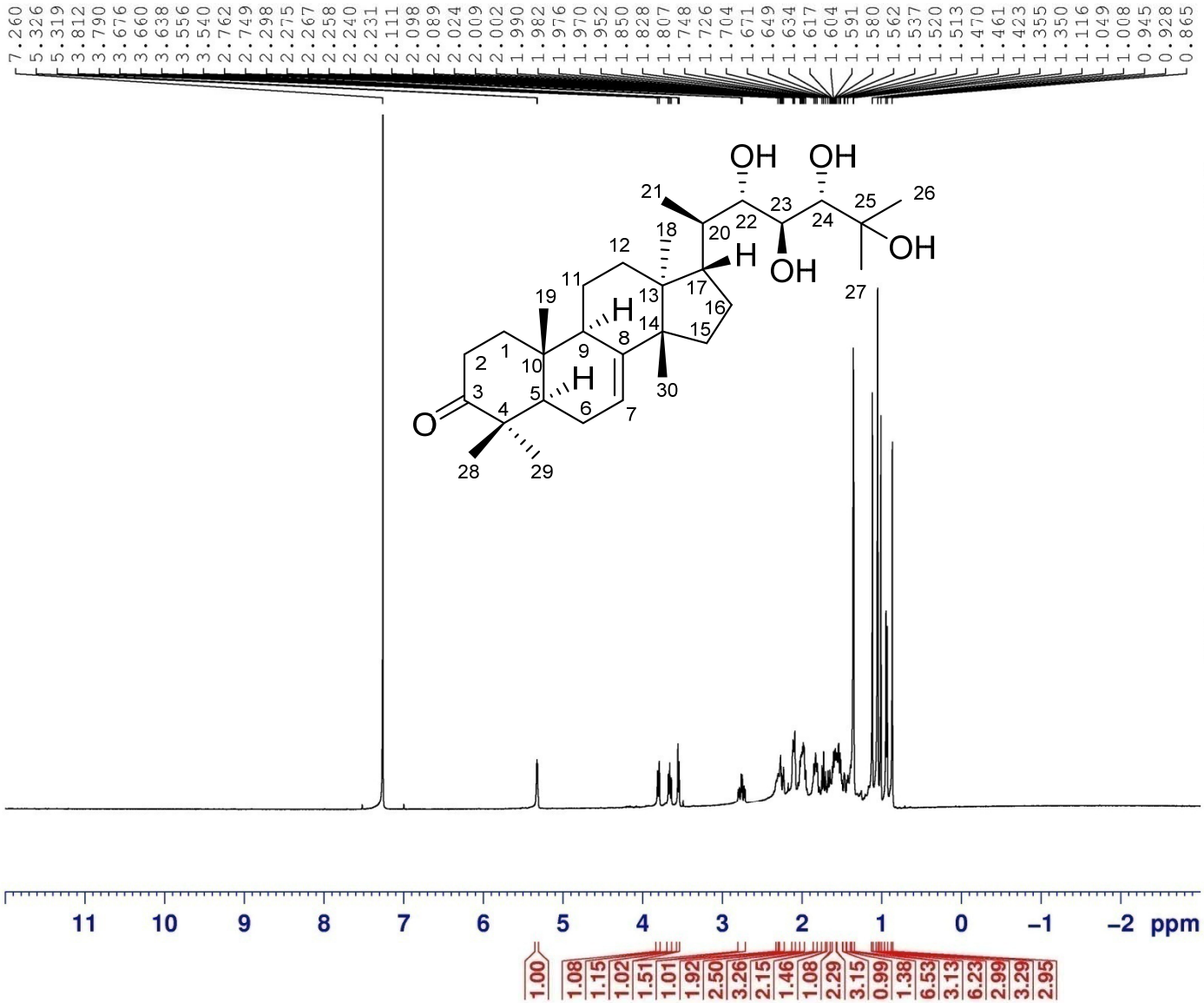
^{1}H NMR (400 MHz) spectrum of compound **1** in $CDCl_3$




```
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EXPNO                 1
PROCNO                1
Date_          20171226
Time              16.56
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               208.5
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               11.50 usec
SI               65536
SF         400.1300096 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```
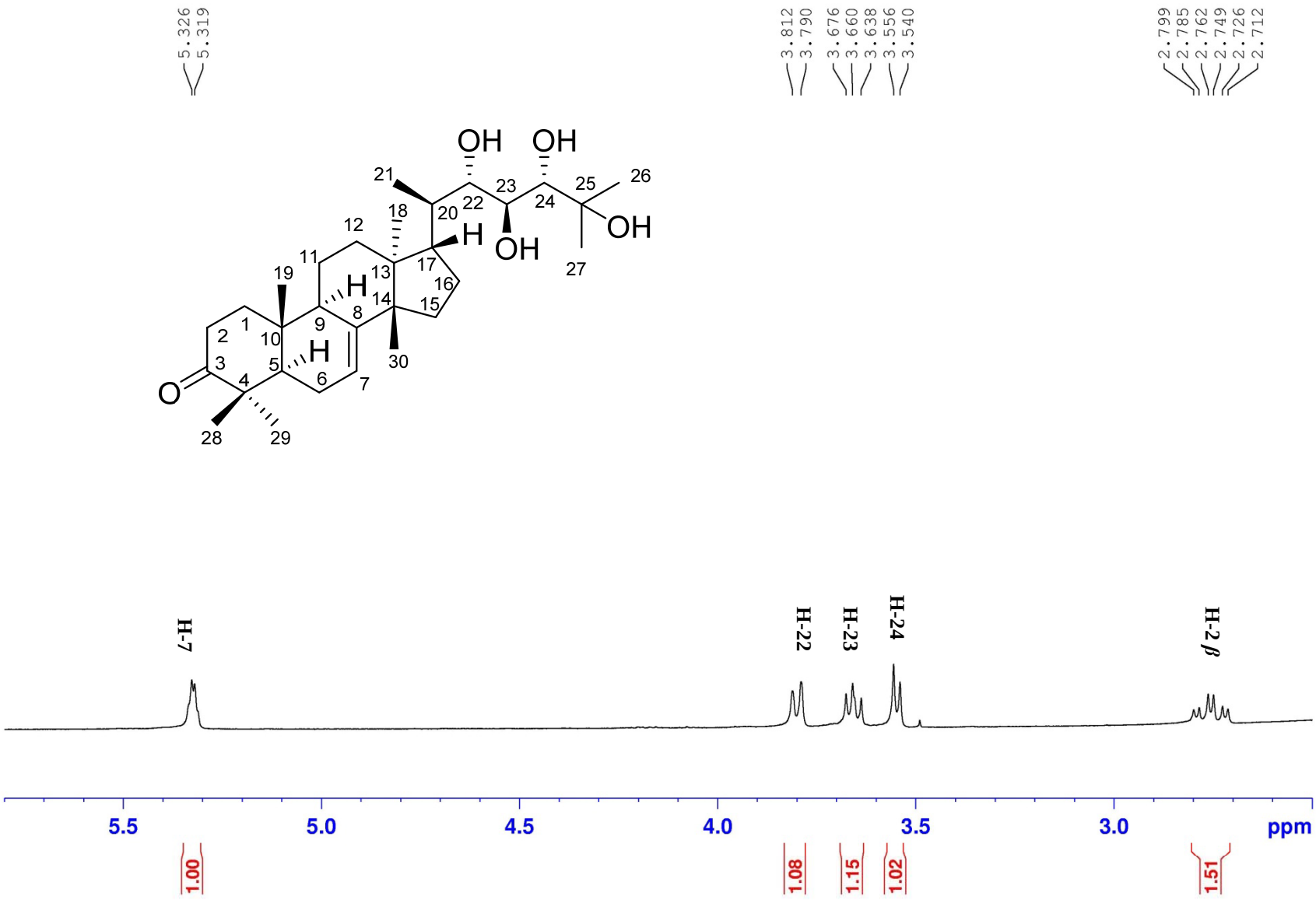
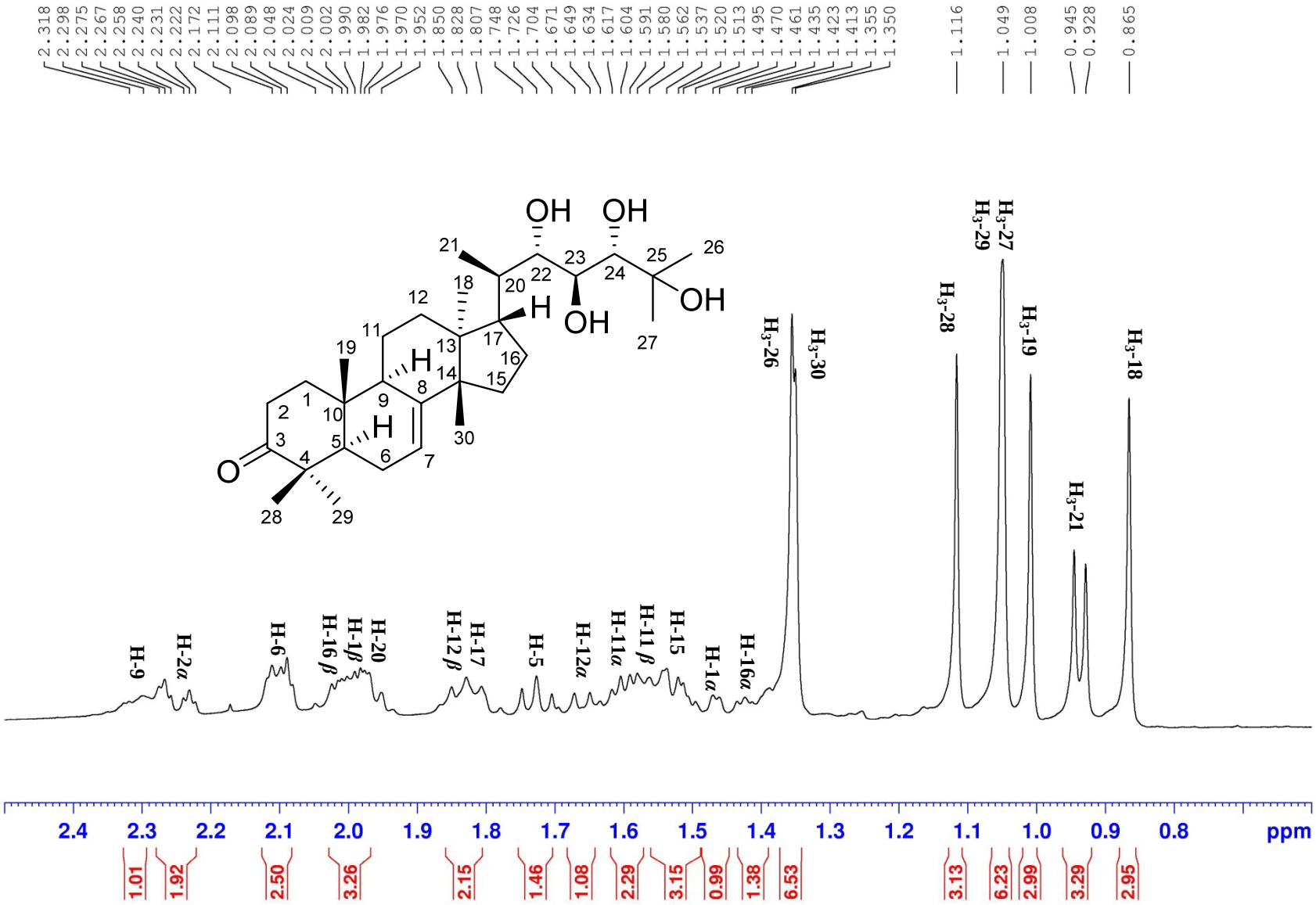
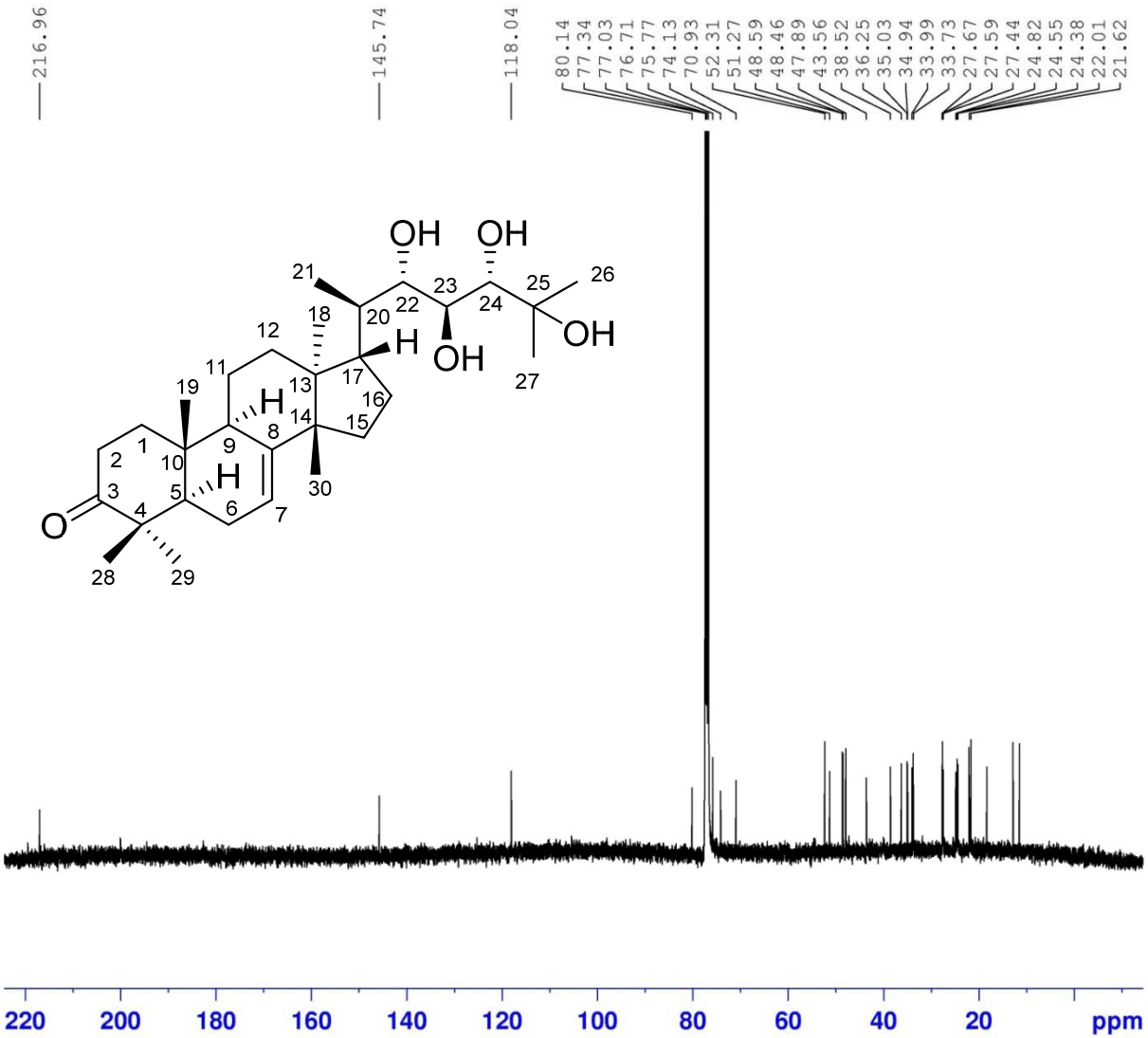

^{1}H NMR (400 MHz) spectrum of compound **1** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **1** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **1** in $CDCl_3$

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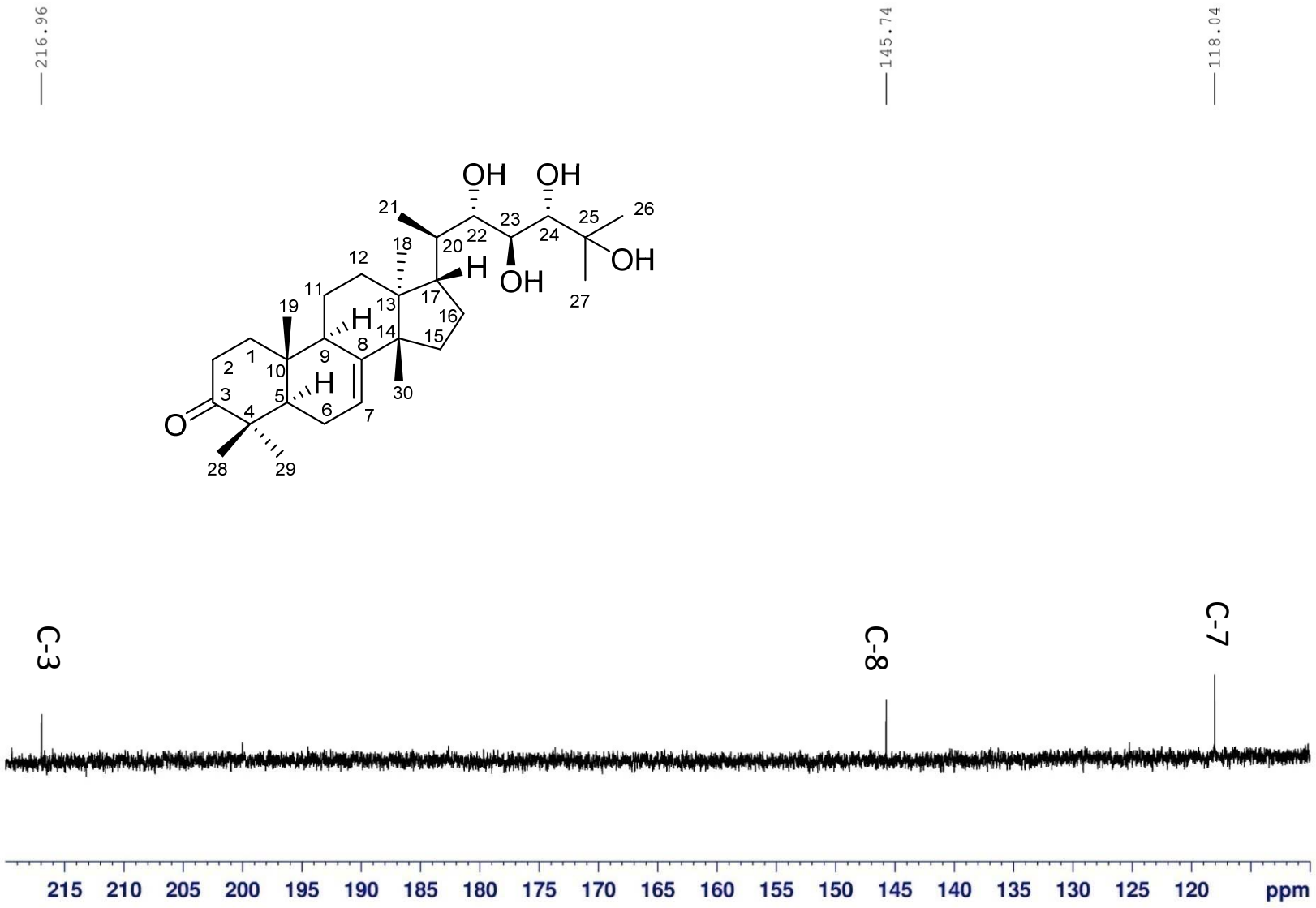
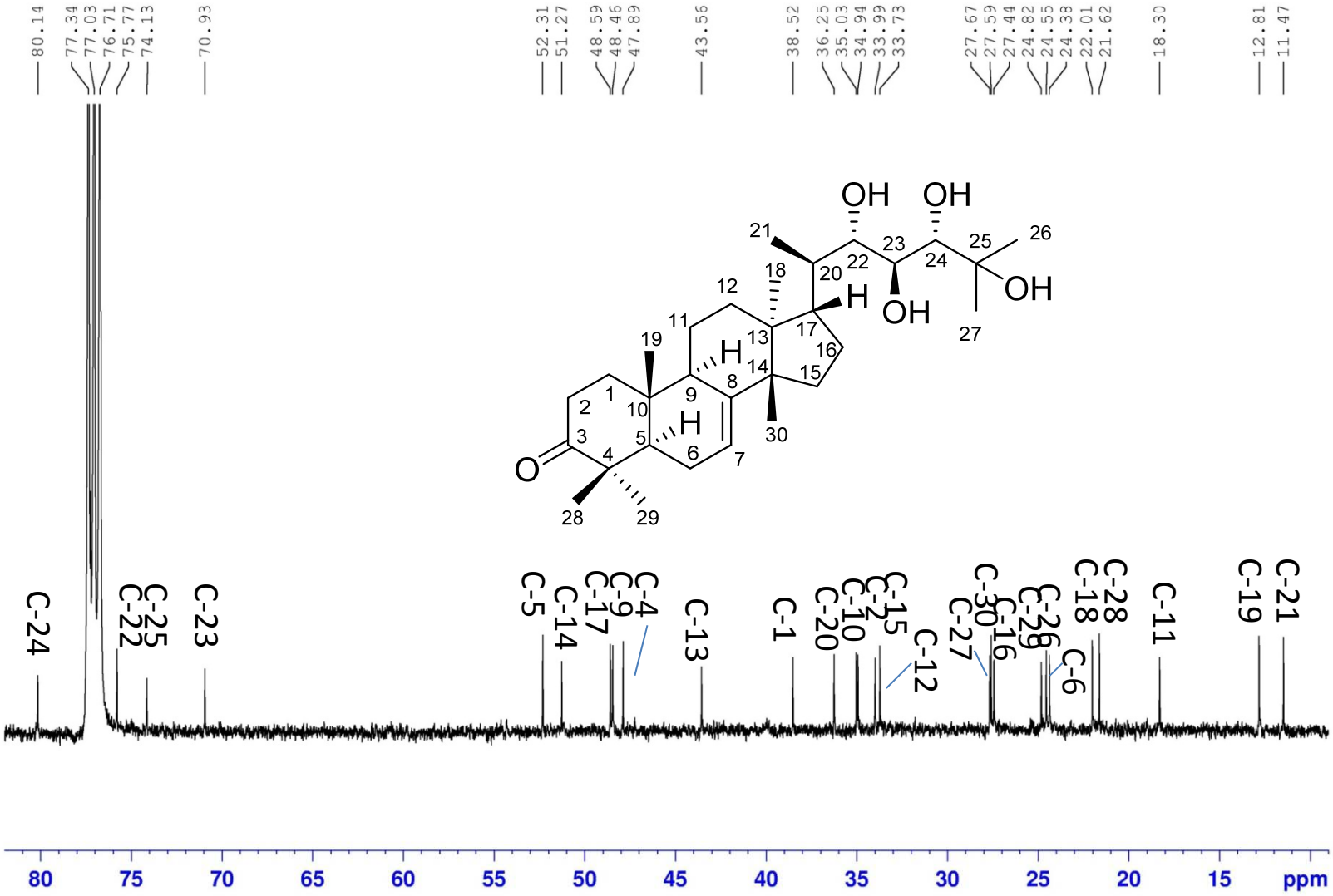
EXPNO                 2
PROCNO                1
Date_          20171226
Time              17.56
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                85.34
DW               20.800 usec
DE               18.00 usec
TE               297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        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
```

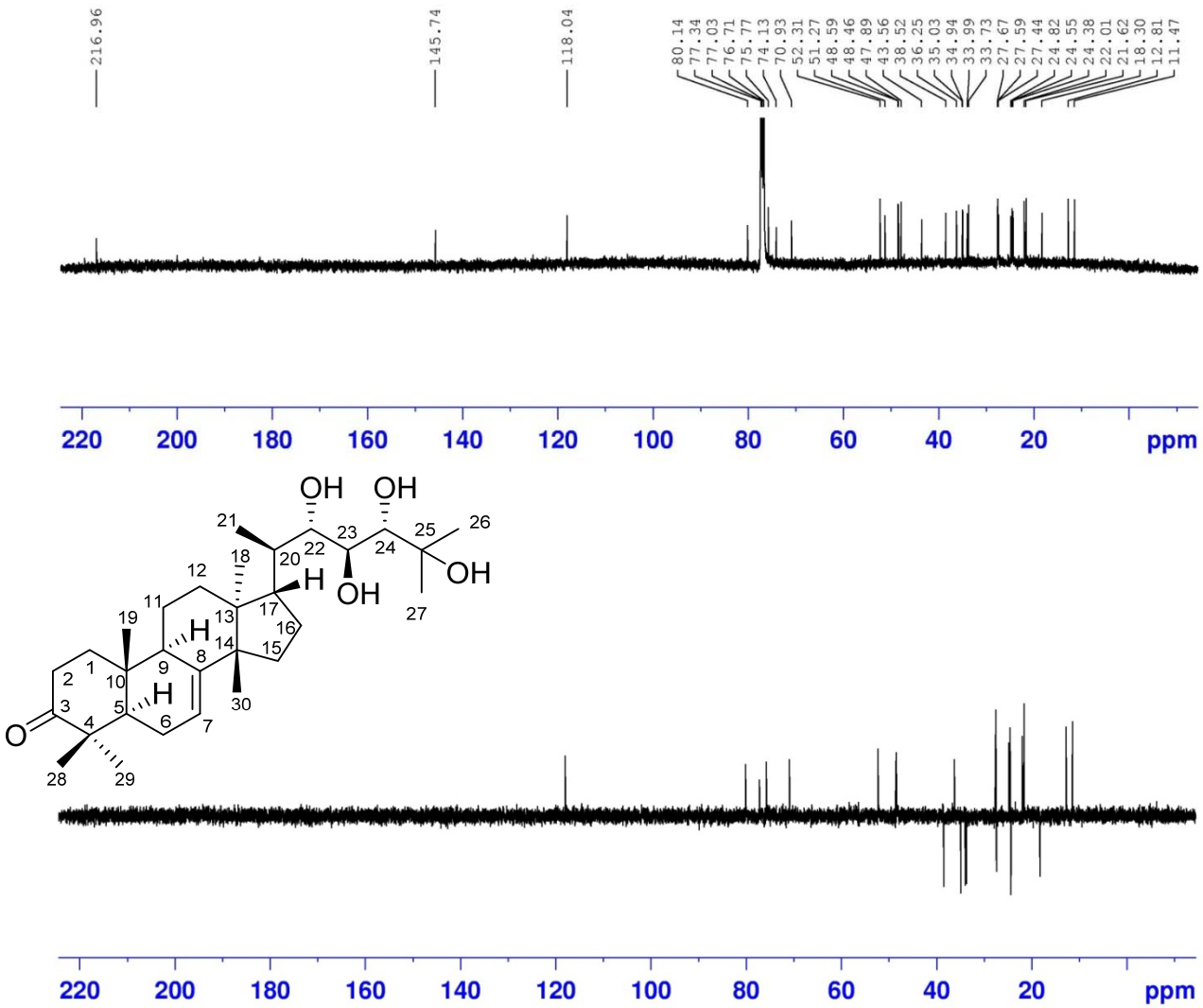
^{13}C NMR (100 MHz) spectrum of compound **1** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **1** in $CDCl_3$



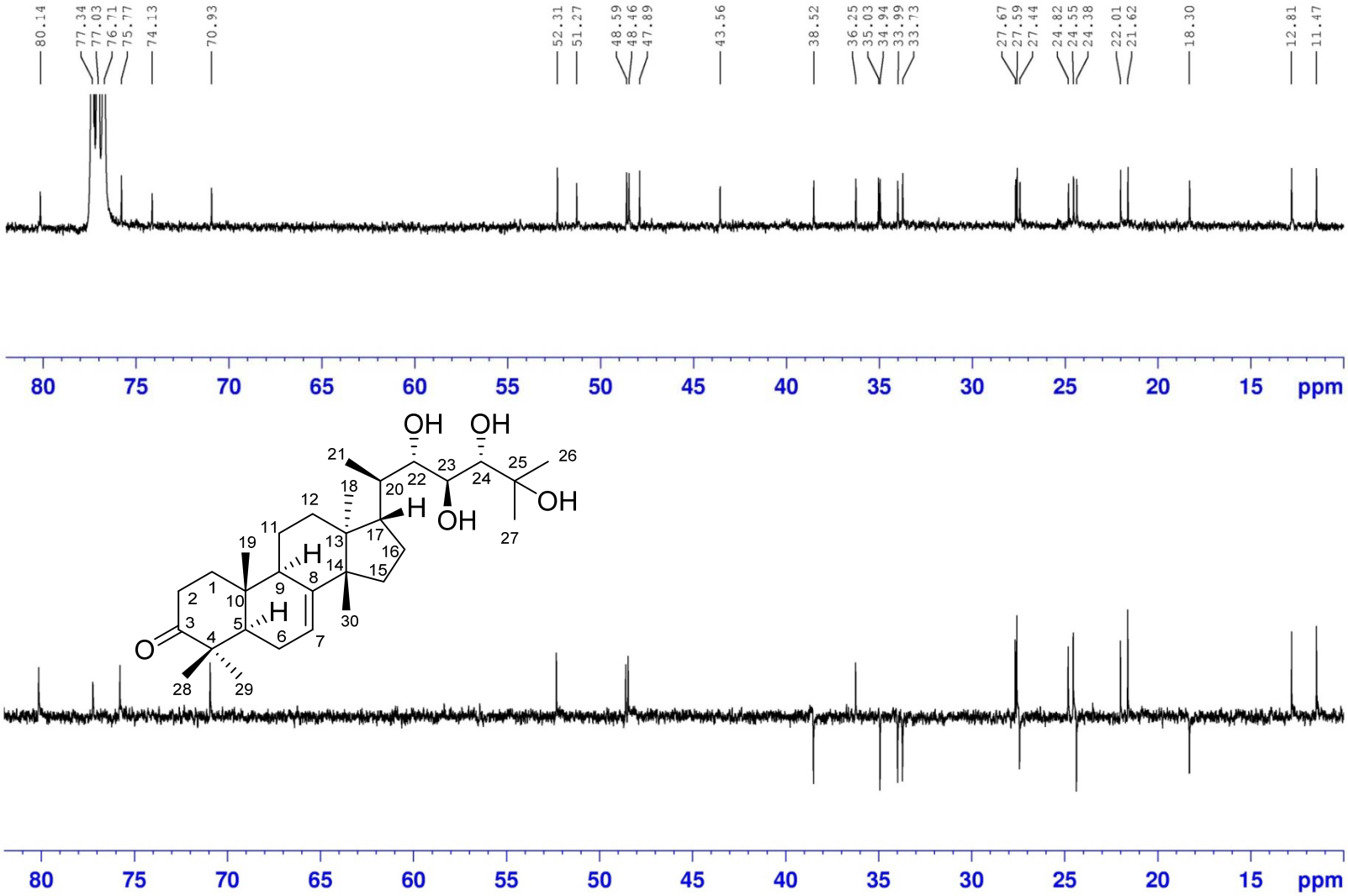
DEPT135 (100 MHz) spectrum of compound **1** in $CDCl_3$



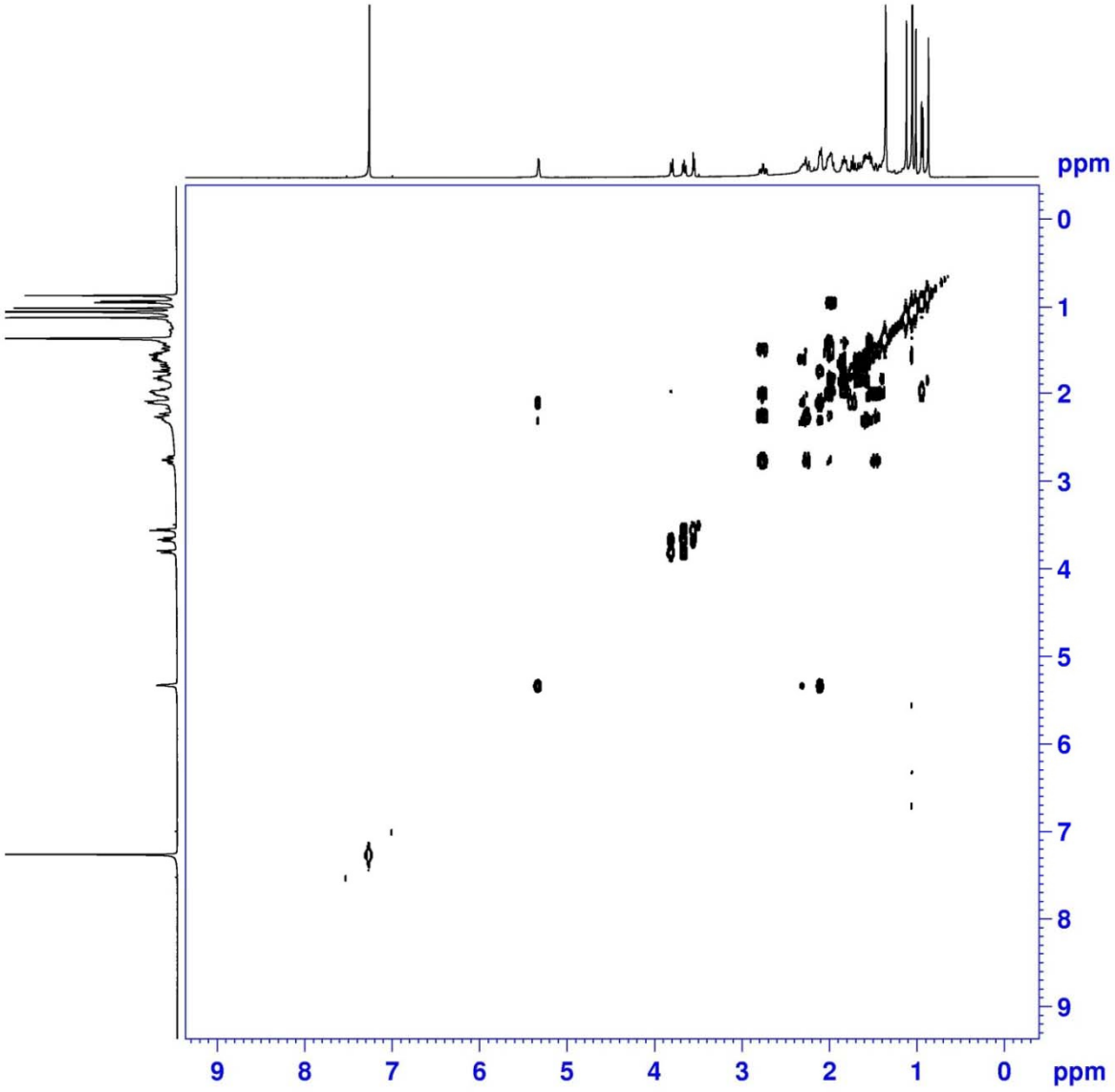

```
NAME            XG-52-3
EXPNO                 3
PROCNO                1
Date_          20171226
Time              18.14
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG       deptsp135
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                85.34
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 ========
SFO1        100.6233324 MHz
NUC1                13C
P1                10.00 usec
P13             2000.00 usec
SI                32768
SF          100.6127685 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

DEPT135 (100 MHz) spectrum of compound **1** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **1** in $CDCl_3$




```
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PROCNO                1
Date_          20180205
Time              21.34
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          400.1300069 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
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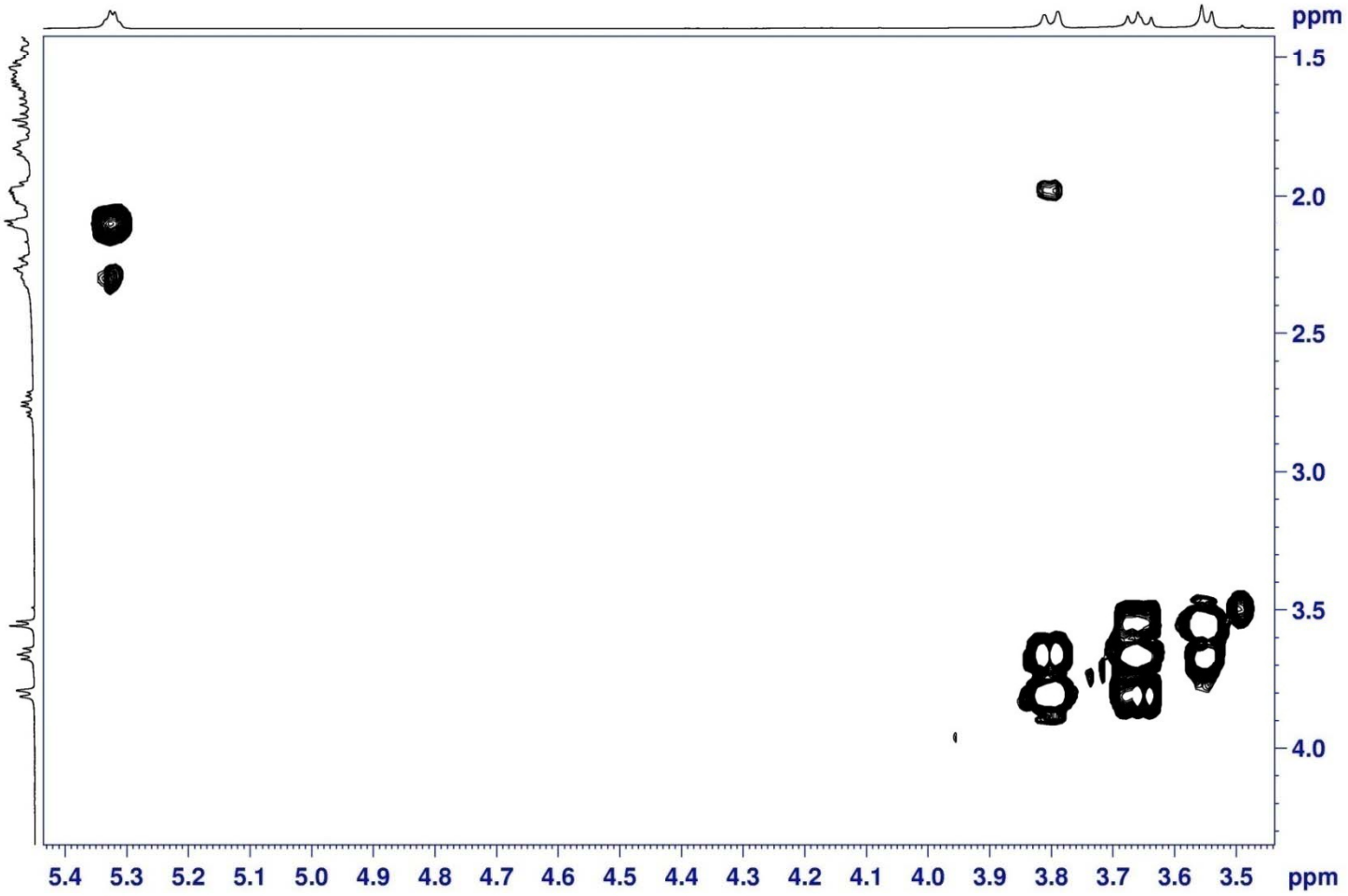
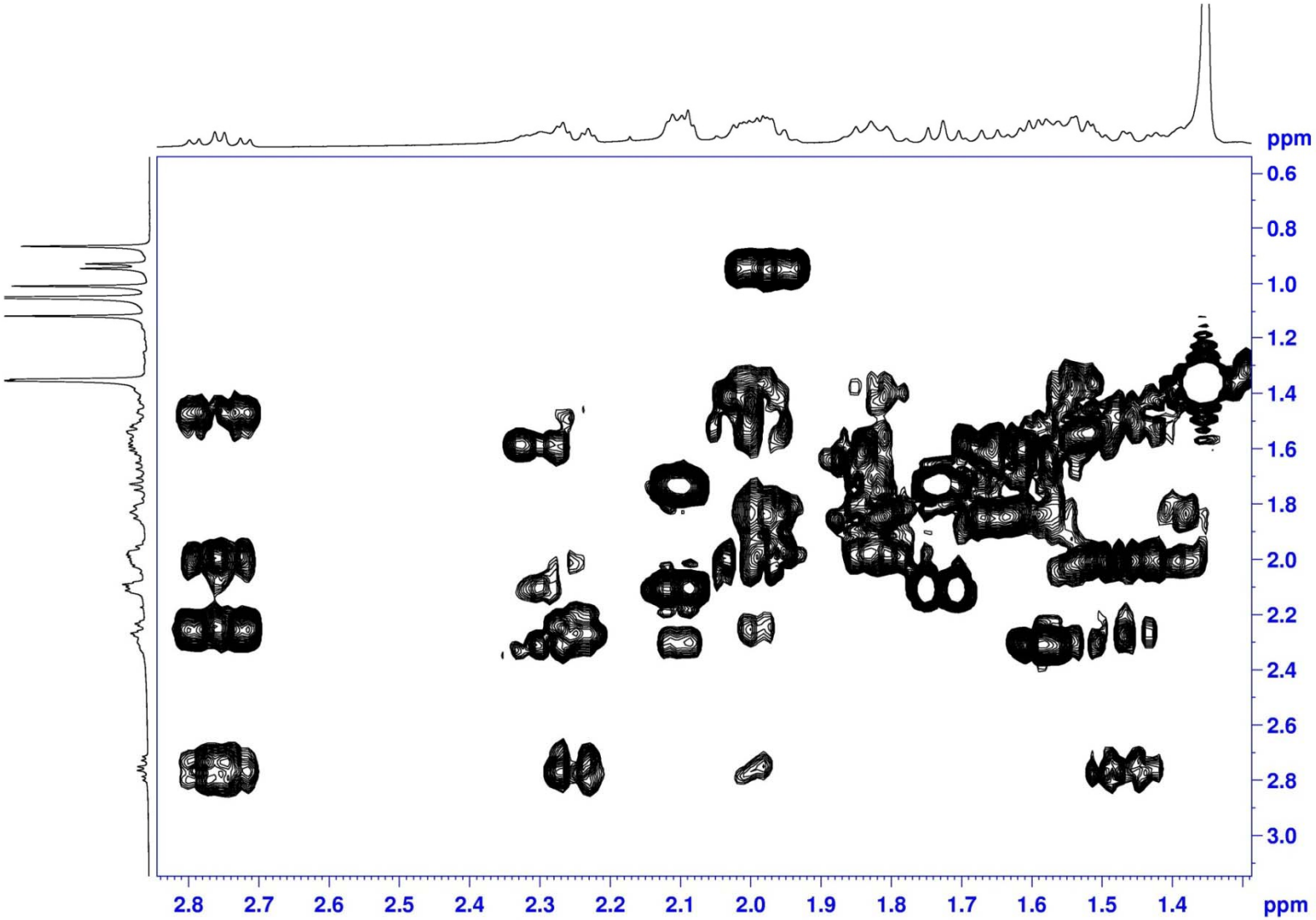
```

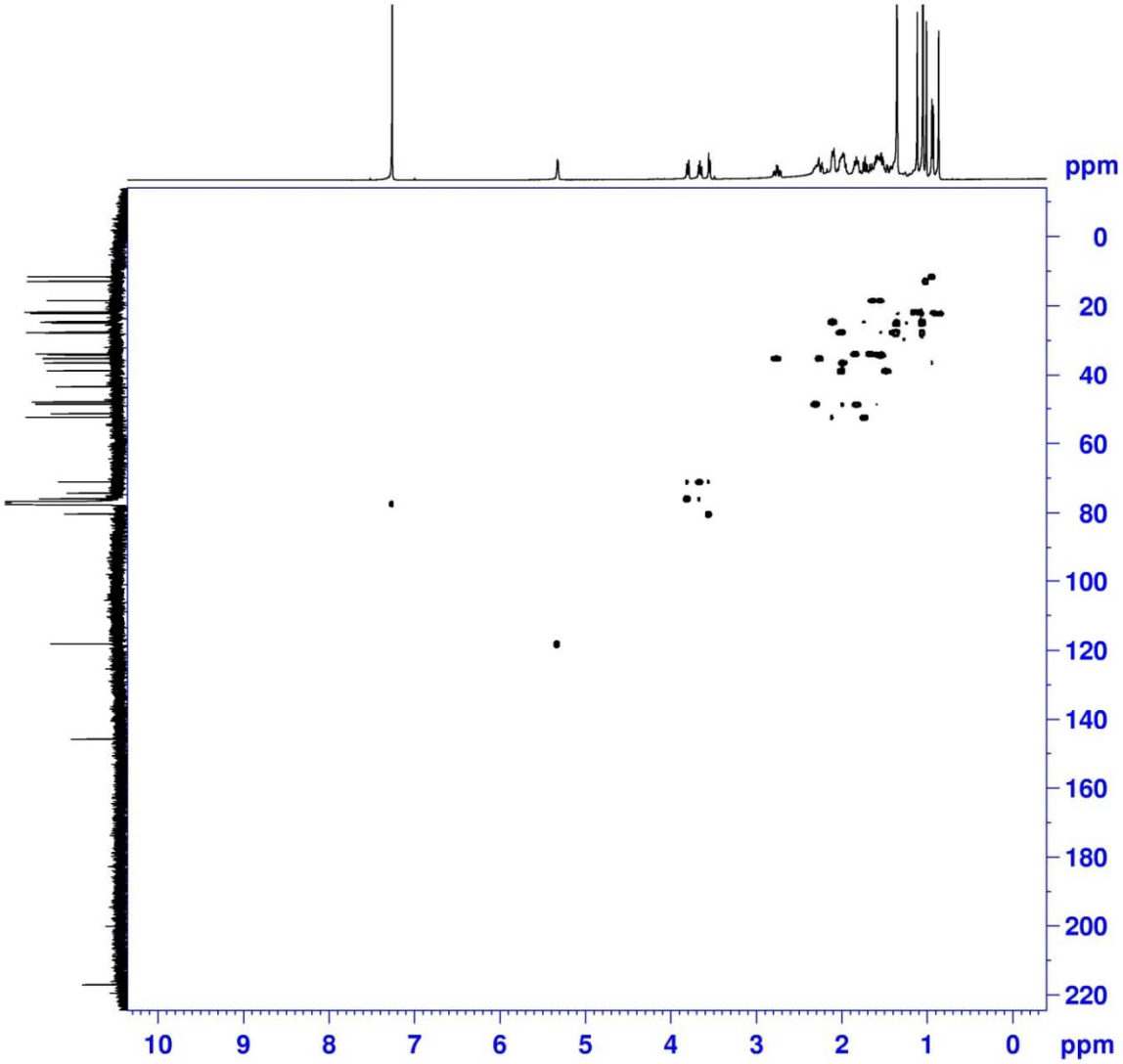
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **1** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **1** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **1** in $CDCl_3$




```
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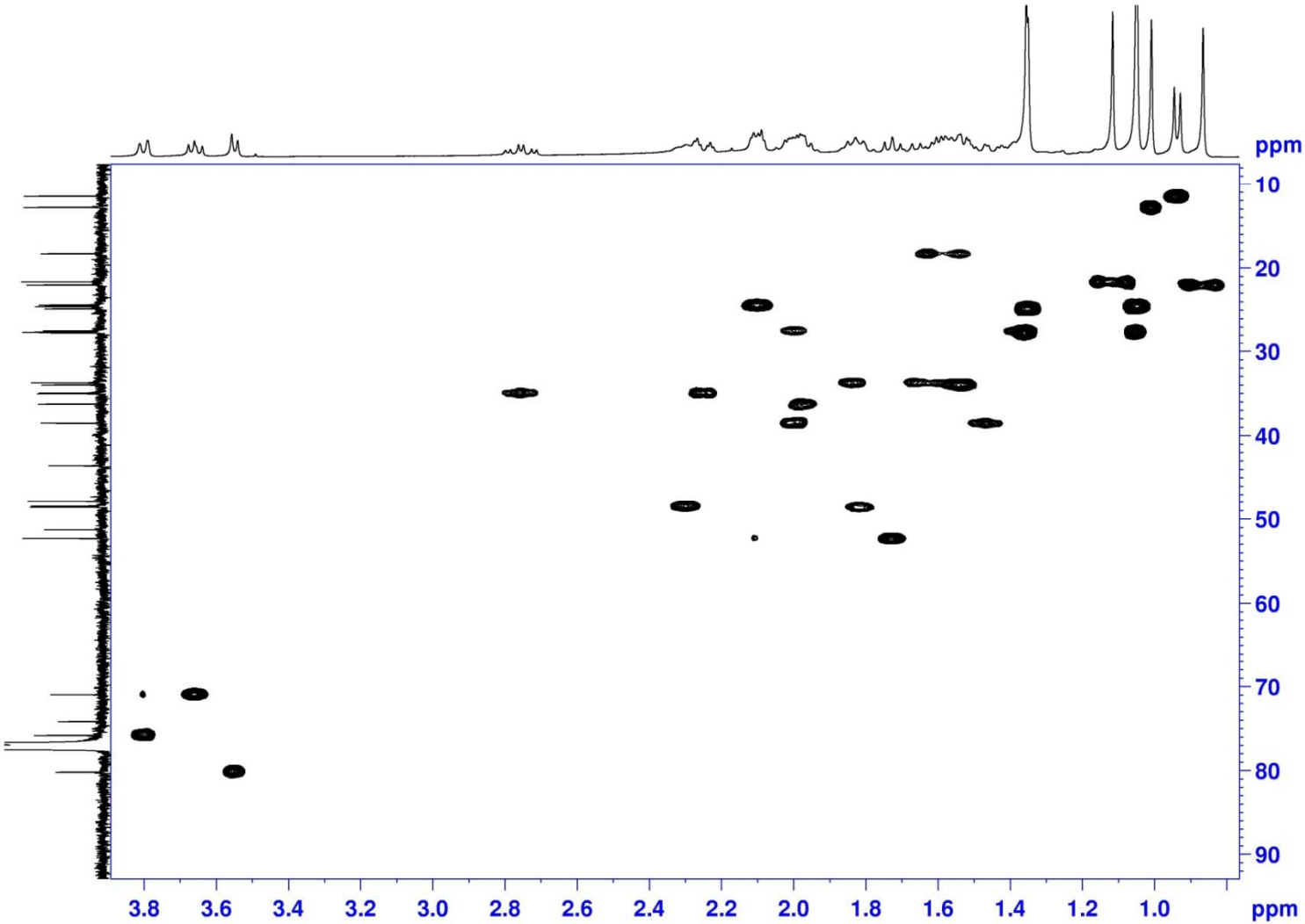
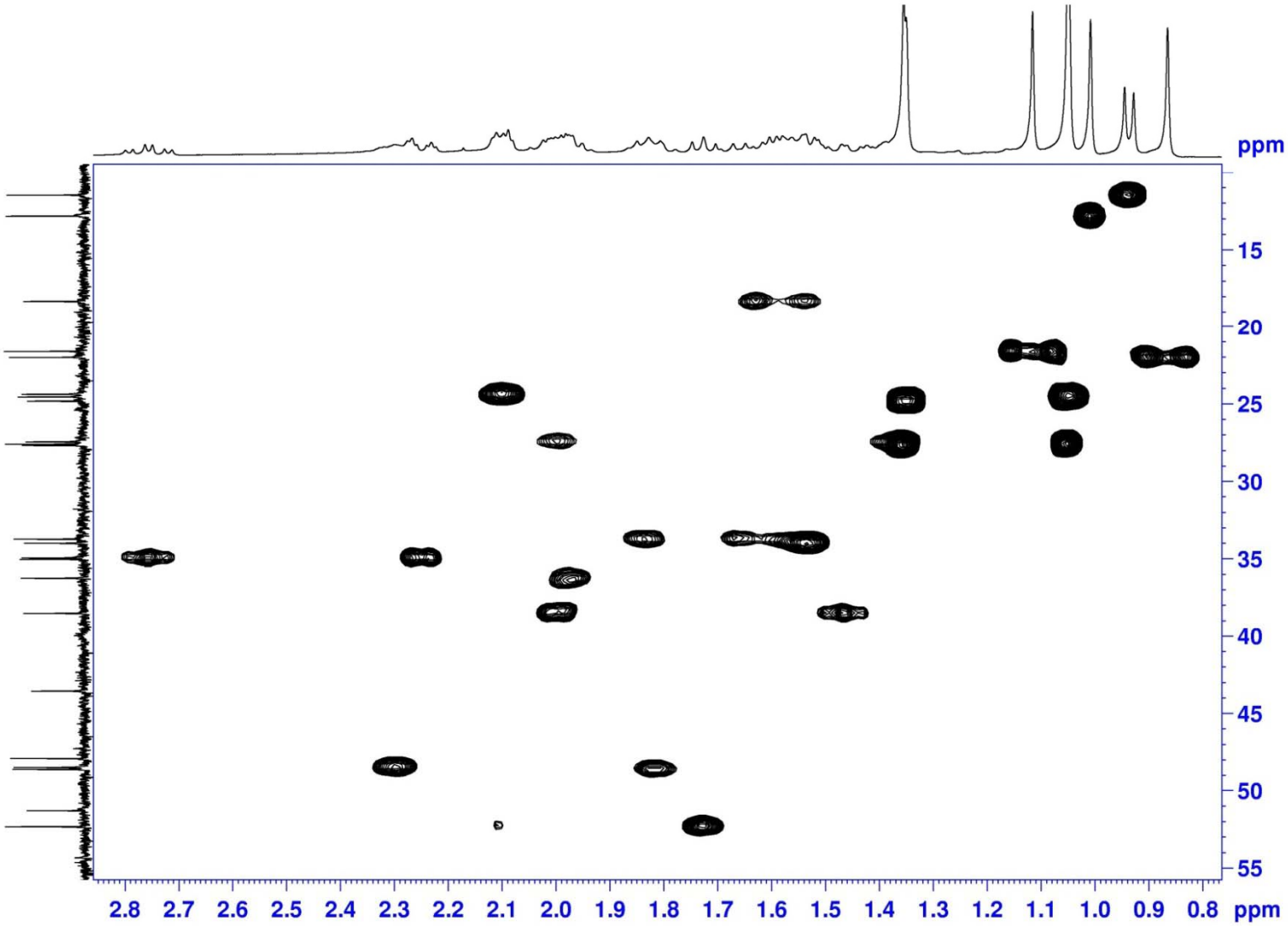
EXPNO                 5
PROCNO                1
Date_          20180205
Time              22.14
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG     hsqcetgpsi2
TD                 1024
SOLVENT           CDCl3
NS                   16
DS                   16
SWH            4302.926 Hz
FIDRES         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
ZGOPTNS

======== CHANNEL f1 ========
SFO1        400.1320007 MHz
NUC1                 1H
P1                11.50 usec
P2                23.00 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          400.1300070 MHz
WDW               QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2       echo-antiecho
SF          100.6127585 MHz
WDW               QSINE
SSB                   2
LB                 0.00 Hz
```

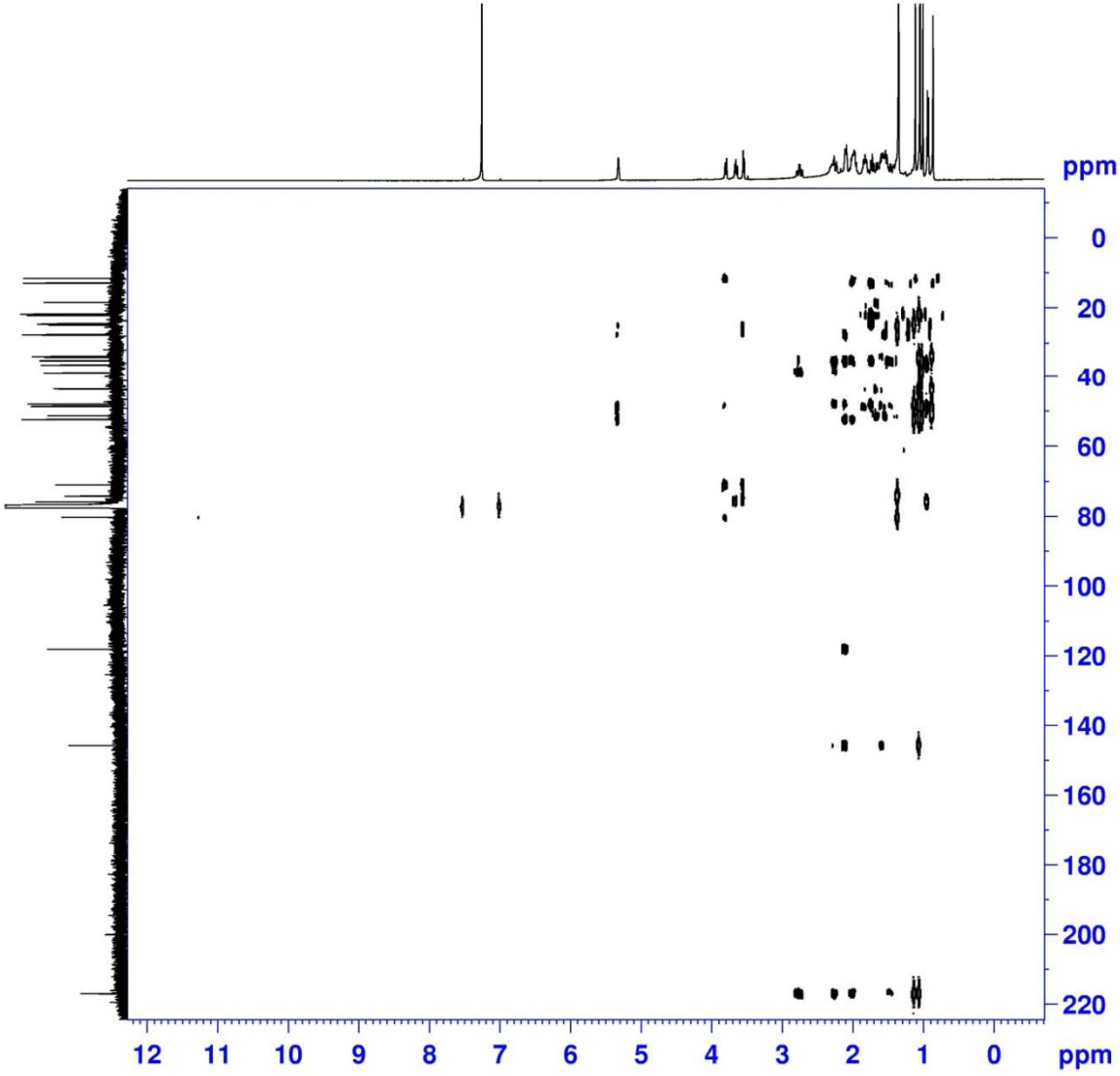
HSQC (400 MHz) spectrum of compound **1** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **1** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$

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PROCNO                1
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PROBHD   5 mm CPPBBO BB
PULPROG    hmbcgplpndqf
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 ========
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
SI                 2048
SF           400.1300070 MHz
WDW                SINE
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2                  QF
SF           100.6127630 MHz
WDW                SINE
SSB                   0
```
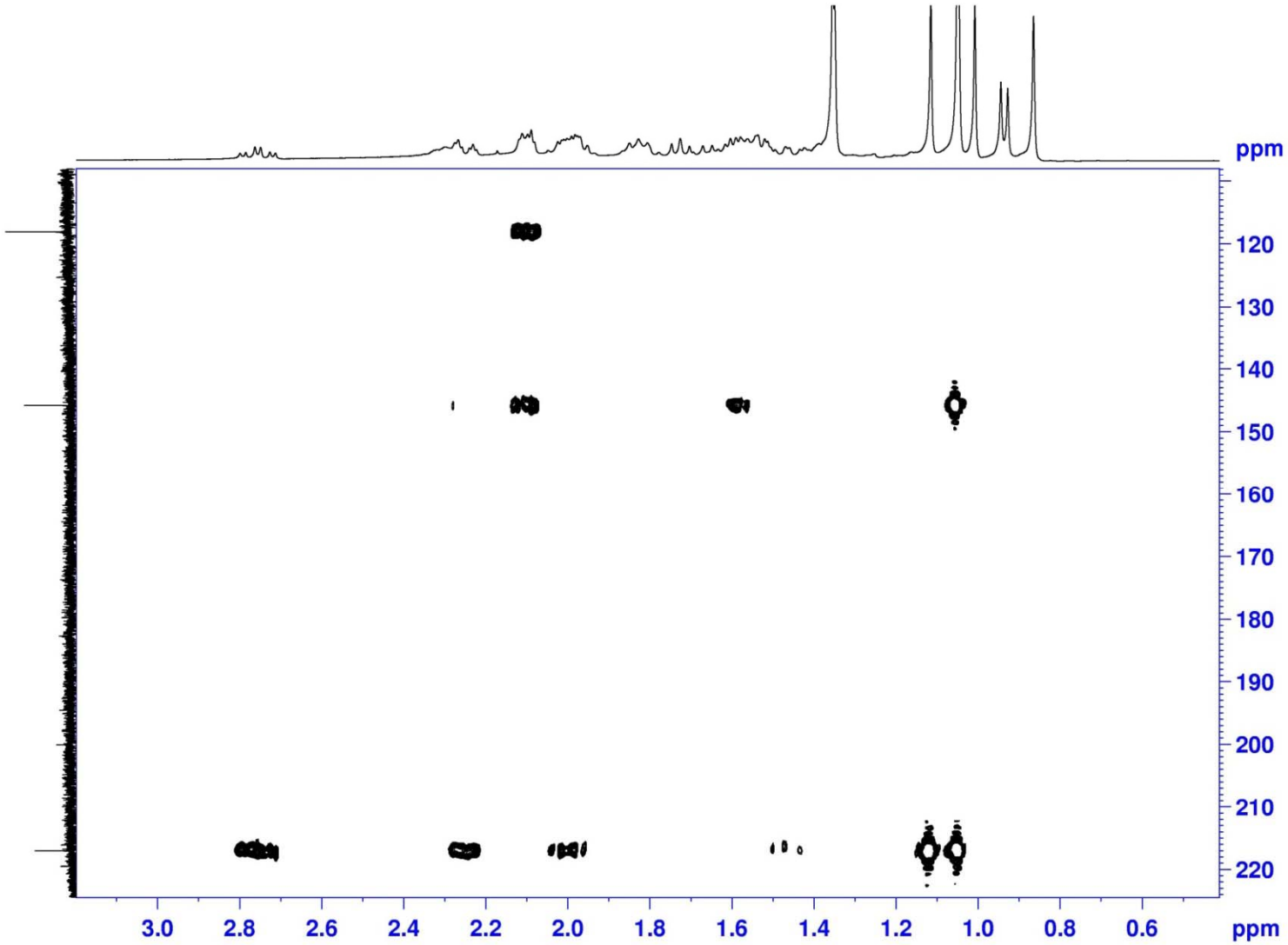
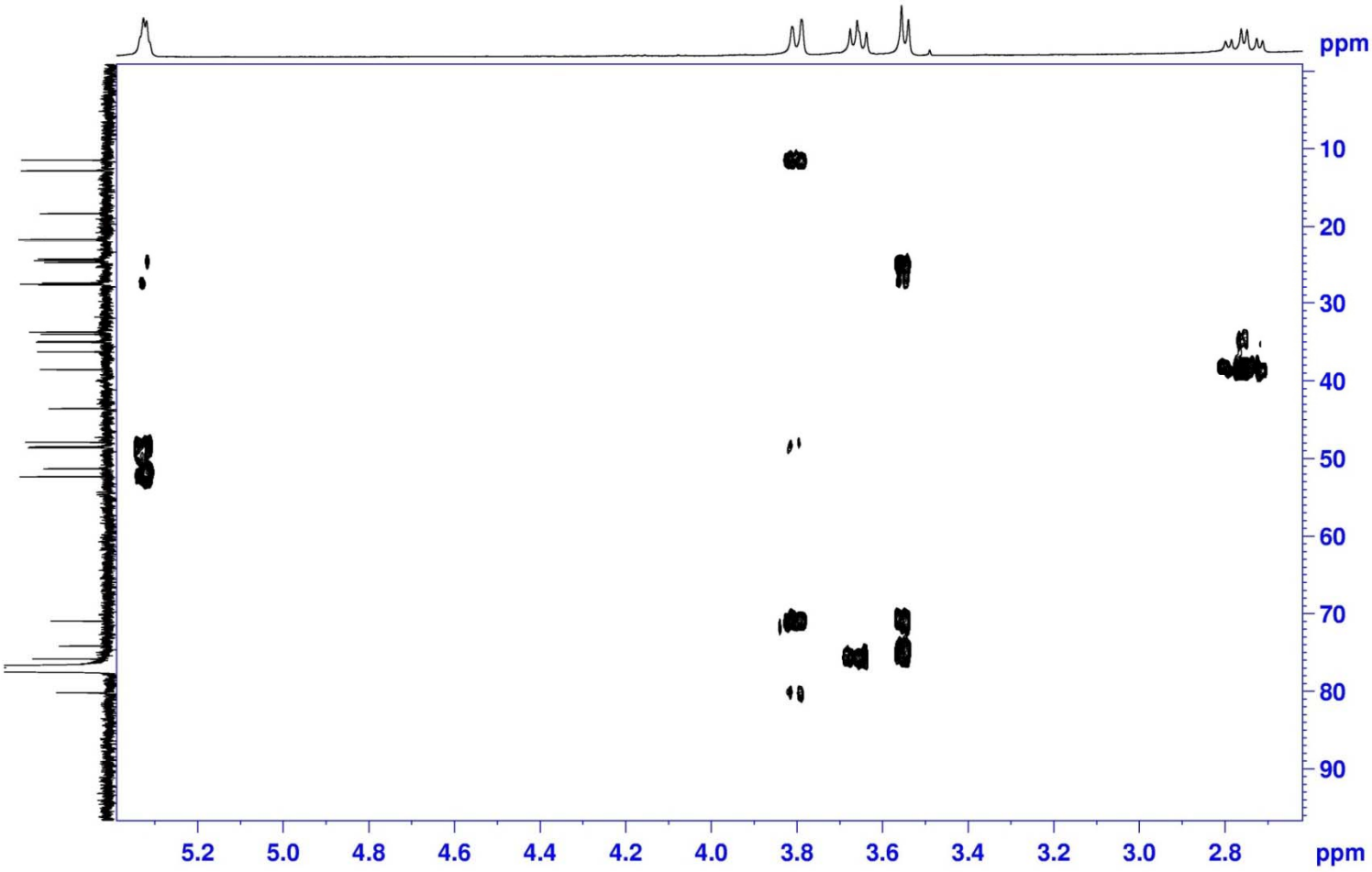
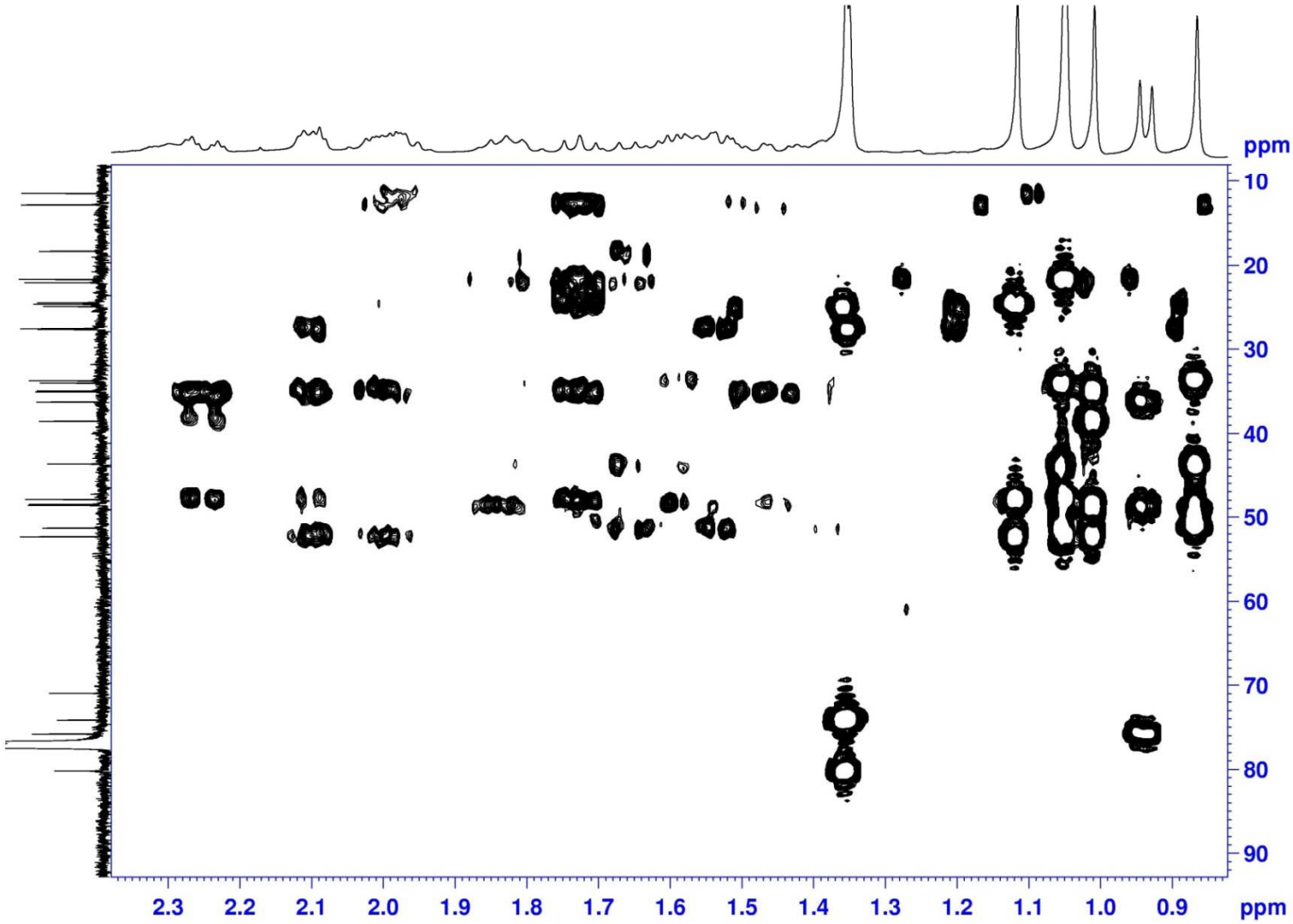

HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$



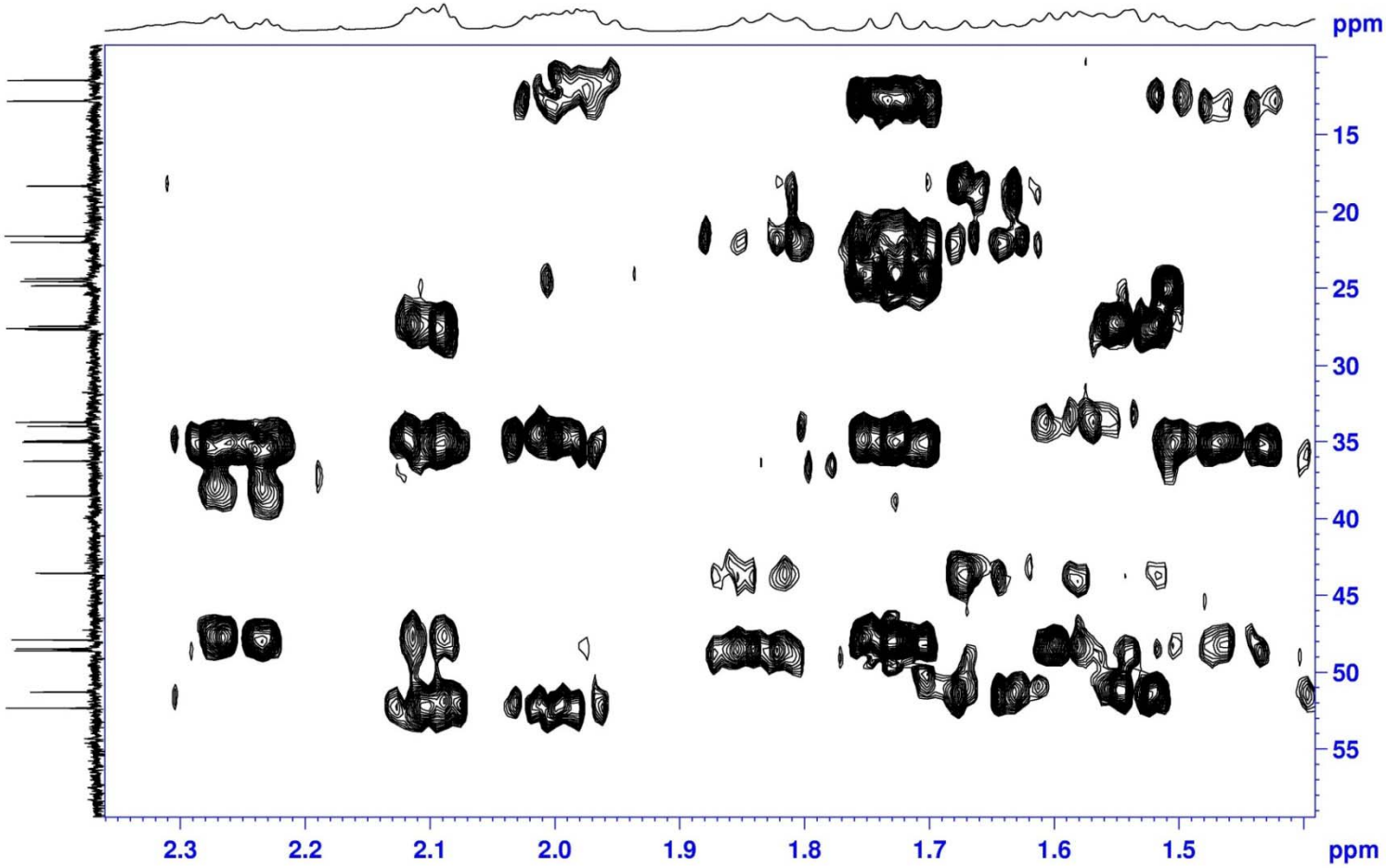
HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$



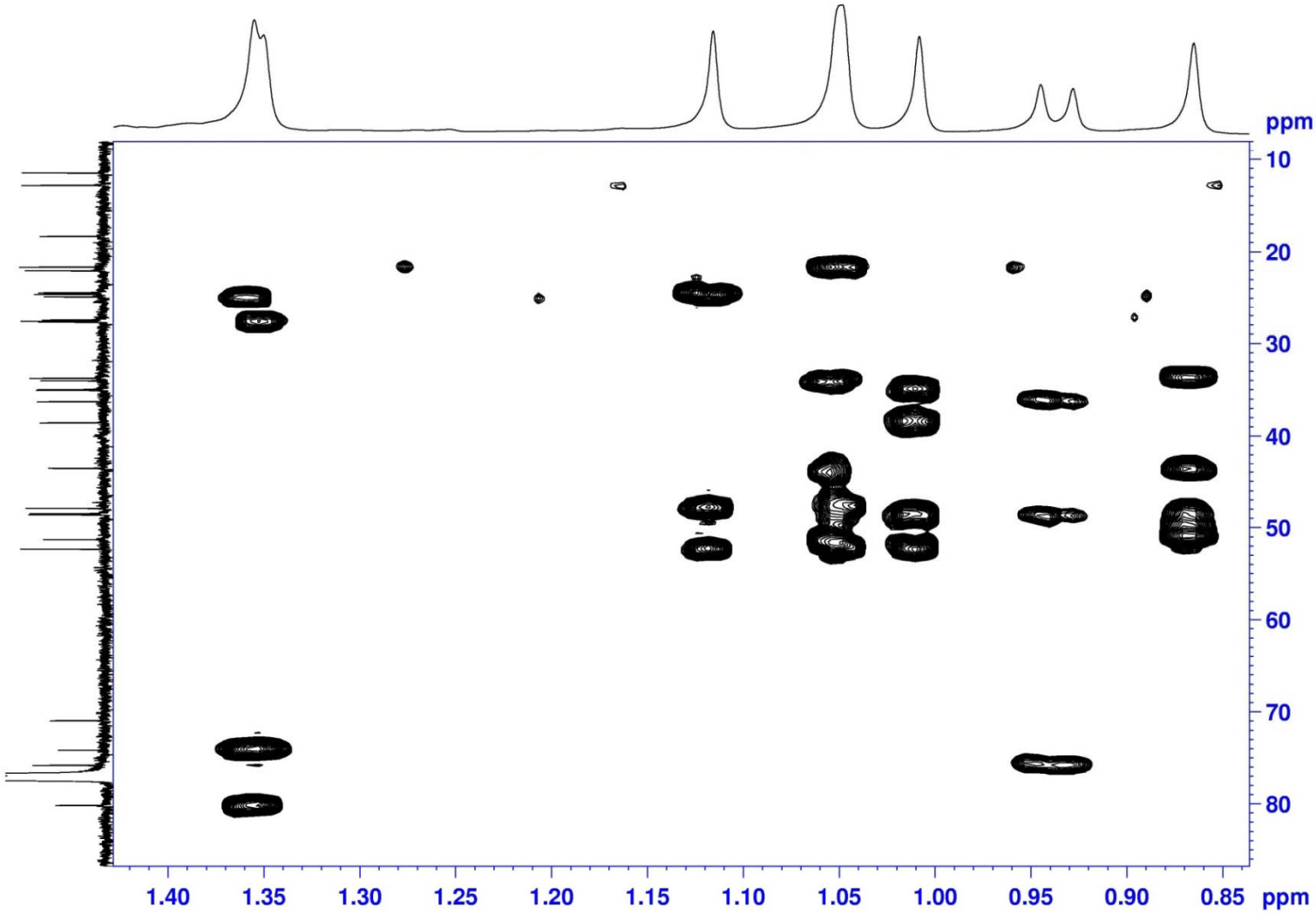
HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$



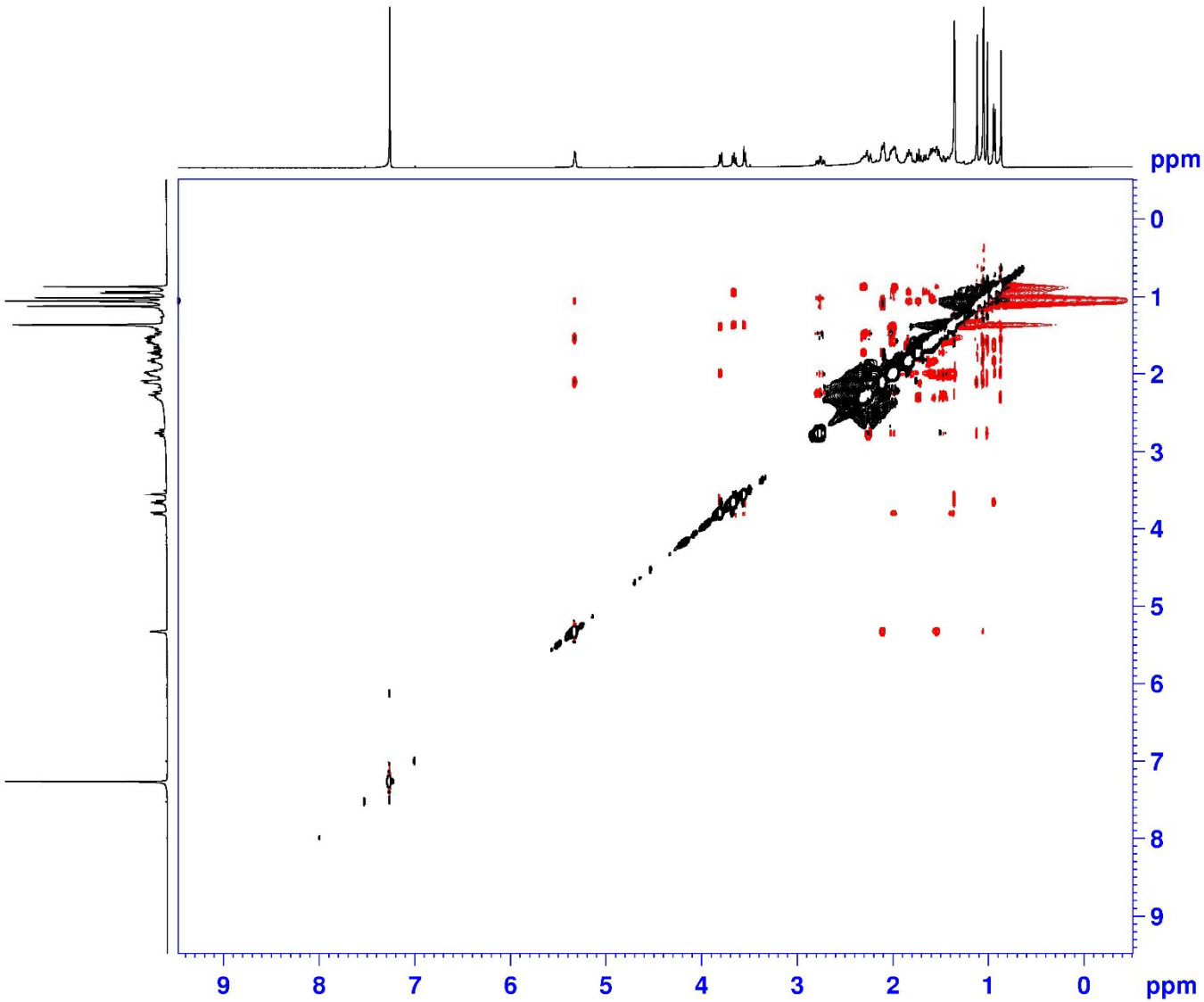
HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **1** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **1** in $CDCl_3$




```
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EXPNO                 7
PROCNO                1
Date_          20180206
Time               2.21
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG     noesygpphpp
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            0.00011036 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 p
FnMODE      States-TPPI
SI                 1024
SF          400.1300072 M
WDW               QSINE
SSB                   2
LB                 0.00 H
GB                    0
PC                 1.00
SI                 1024
MC2         States-TPPI
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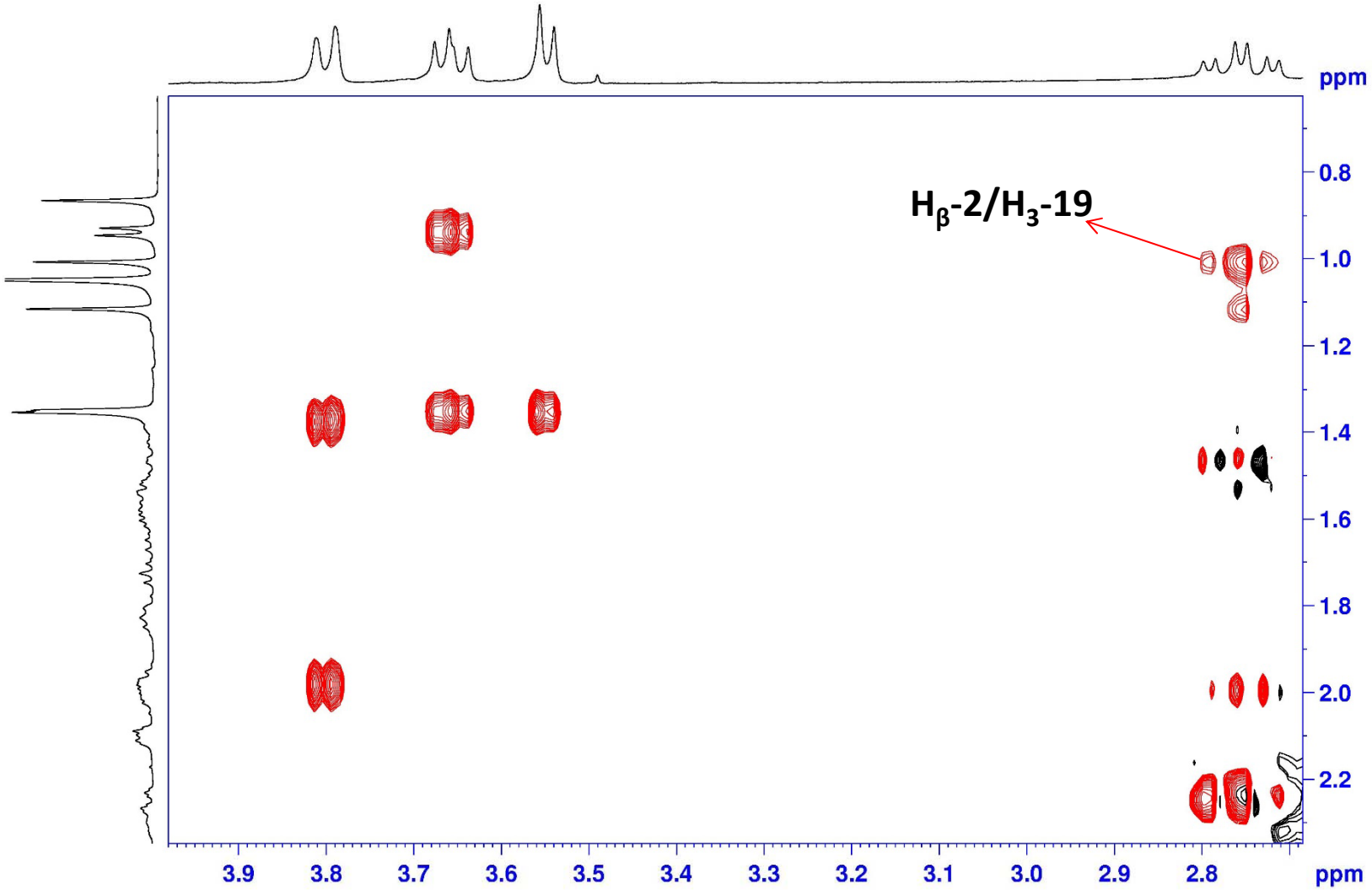
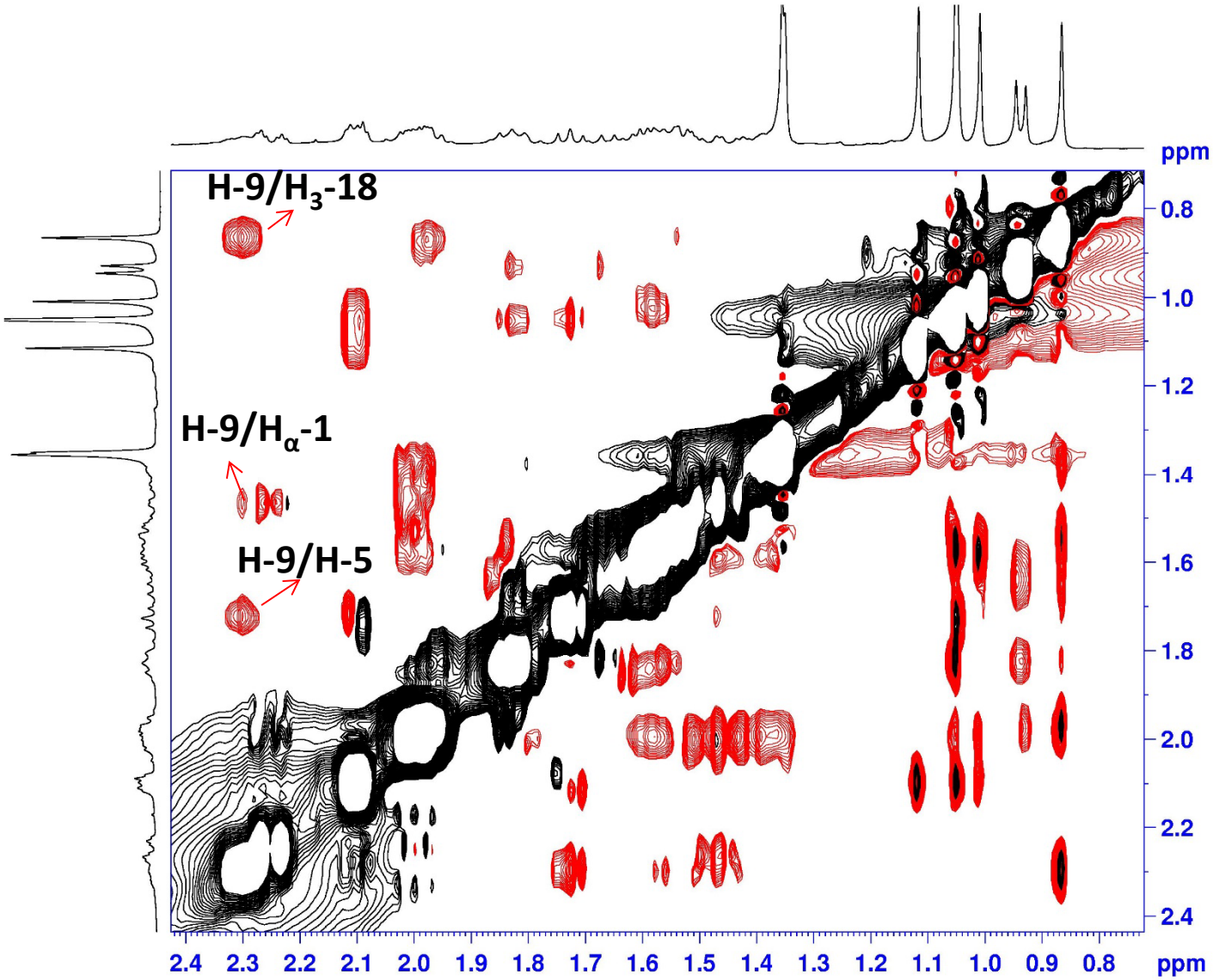
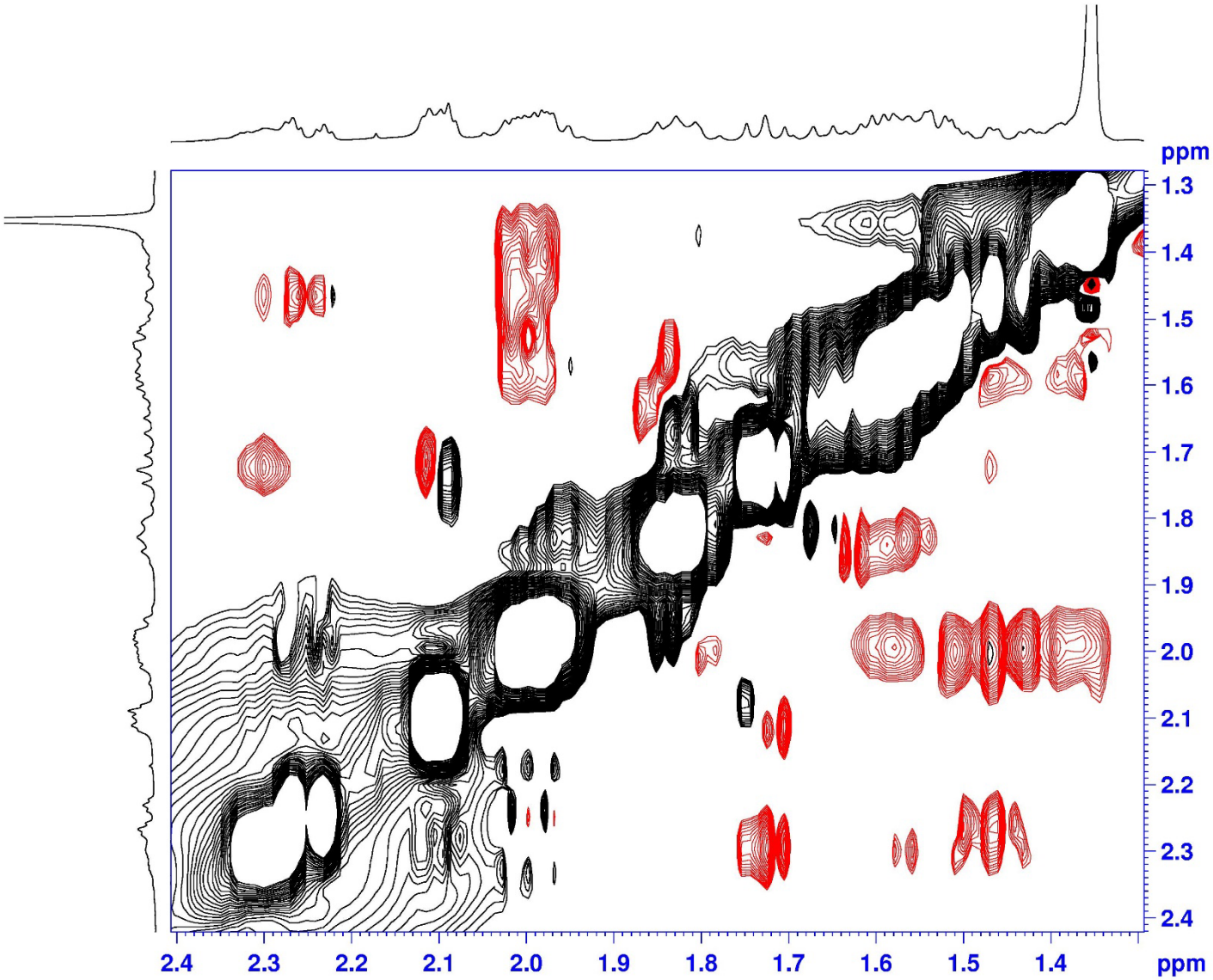
NOESY (400 MHz) spectrum of compound **1** in $CDCl_3$

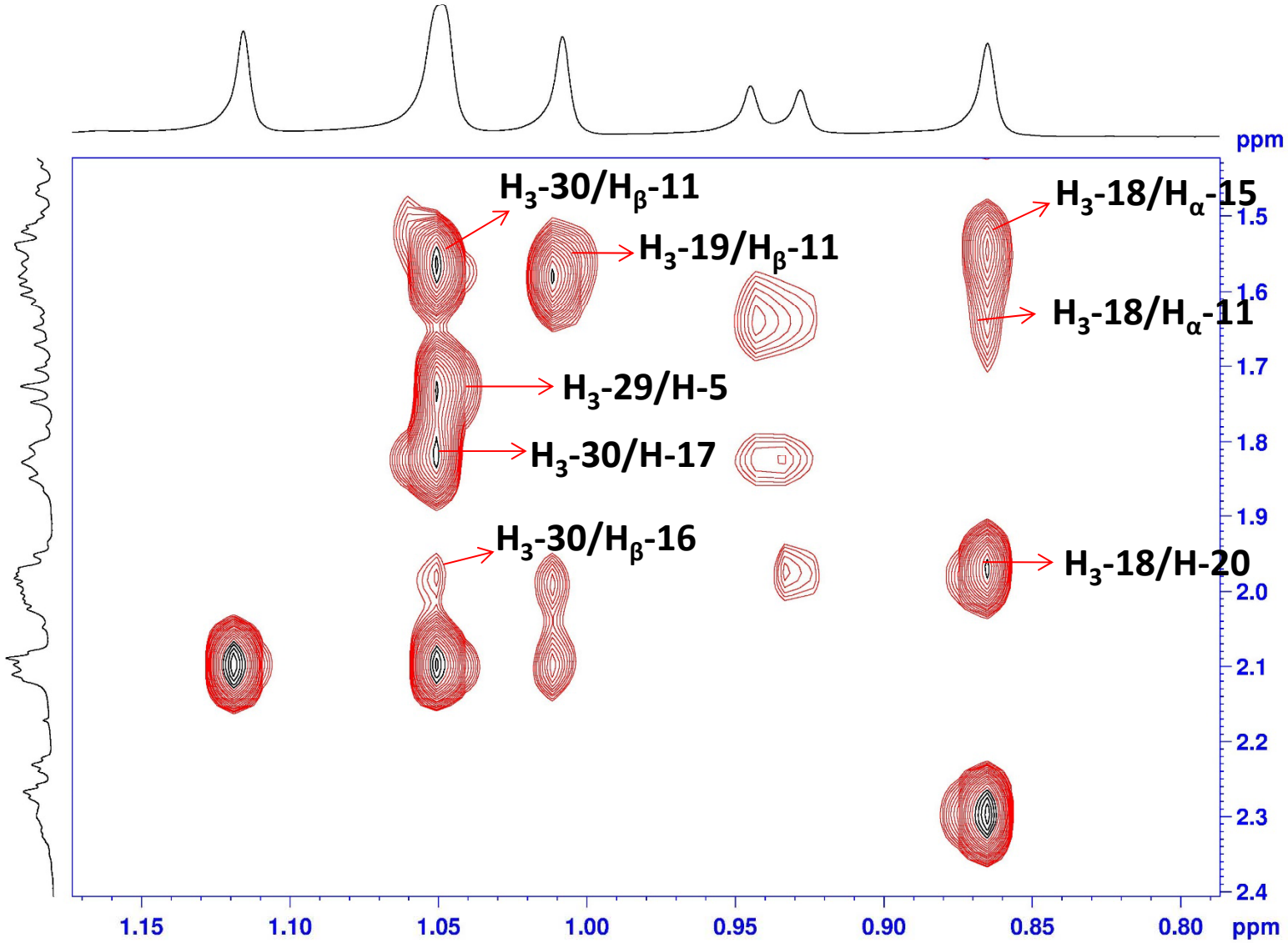


NOESY (400 MHz) spectrum of compound **1** in $CDCl_3$

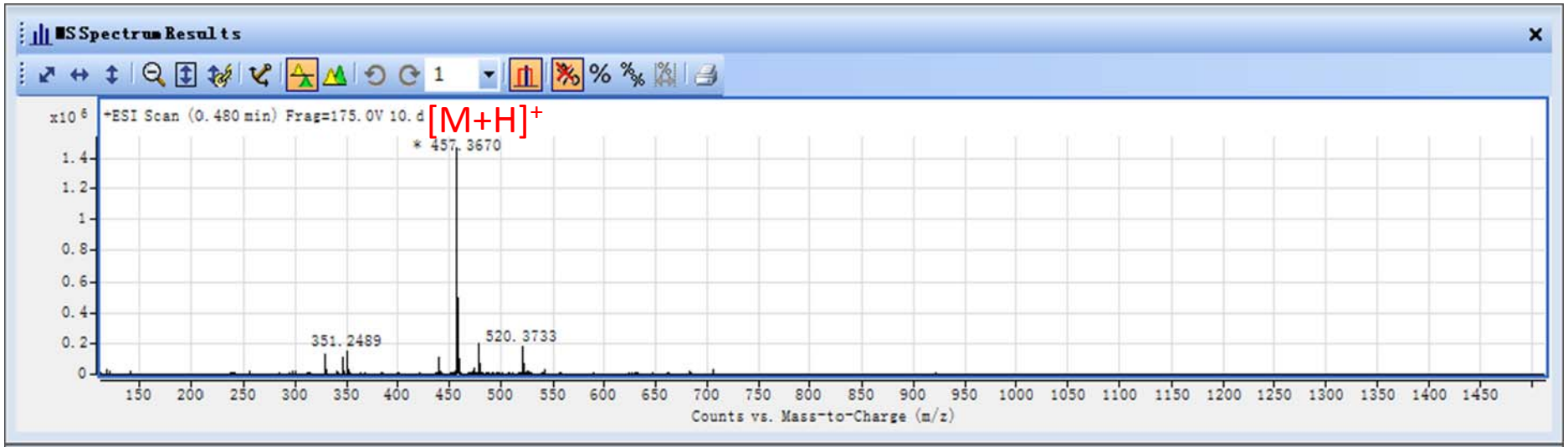


NOESY (400 MHz) spectrum of compound **1** in $CDCl_3$




H3-30/Hβ-11
H3-19/Hβ-11
H3-29/H-5
H3-30/H-17
H3-30/Hβ-16
H3-18/Hα-15
H3-18/Hα-11
H3-18/H-20
ppm
1.5
1.6
1.7
1.8
1.9
2.0
2.1
2.2
2.3
2.4
1.15
1.10
1.05
1.00
0.95
0.90
0.85
0.80
ppm

HR-ESIMS for compound **2**

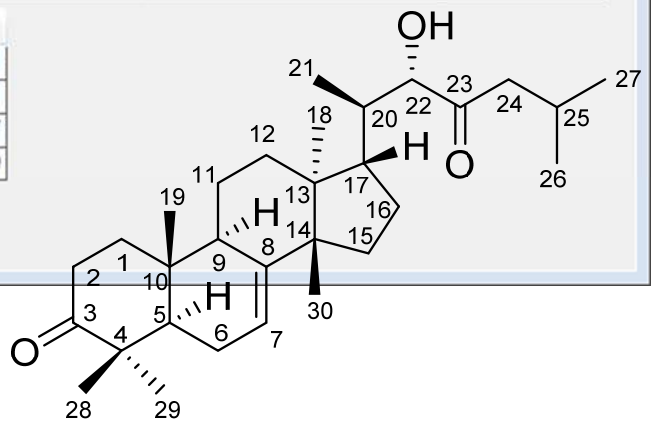


MS Formula Results: + Scan (0.480 min)

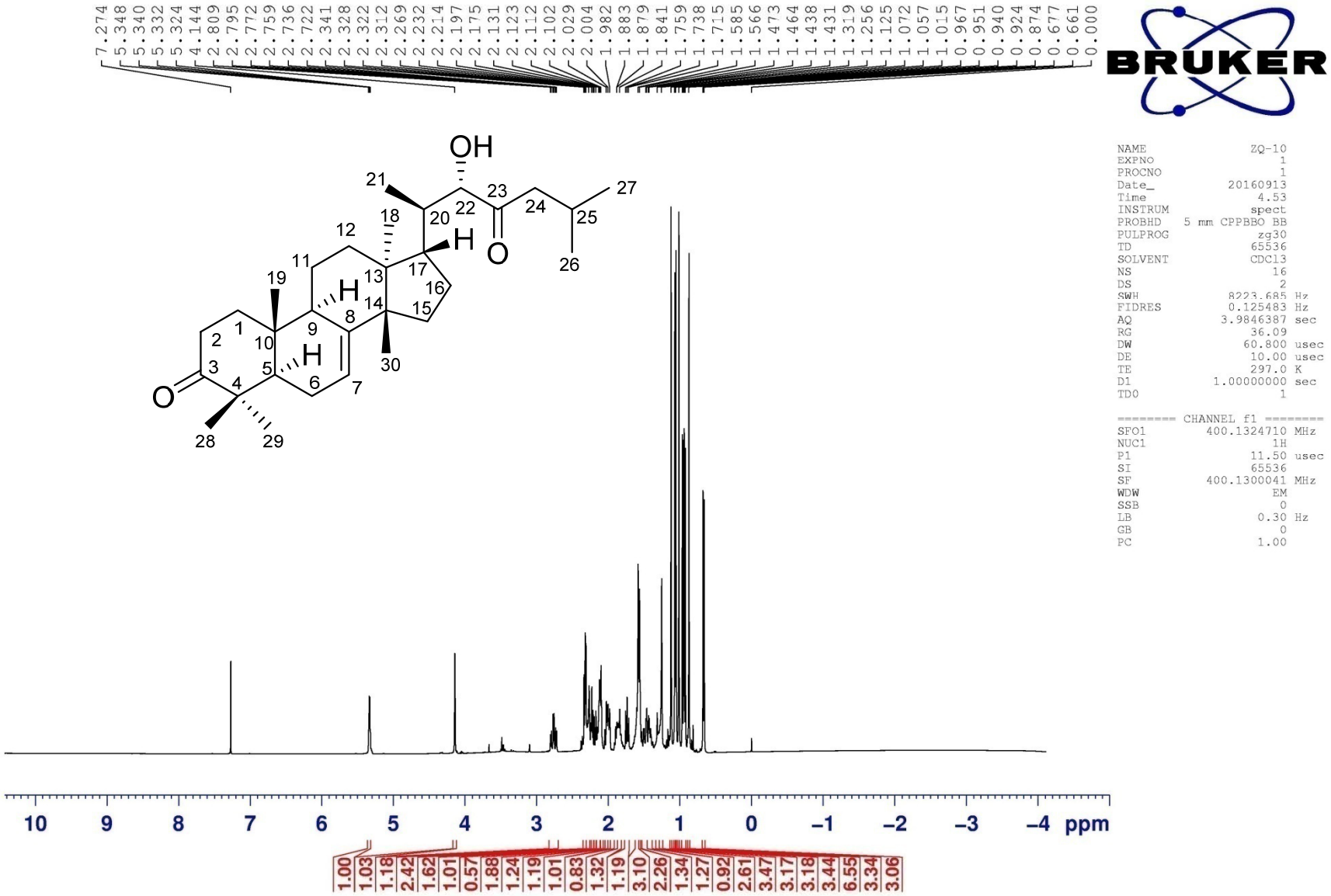
m/z	Ion	Formula	Abundance
457.367	(M+H)+	C30 H49 O3	1494171.9

Best	Formula (M)	Ion Formula	Score	Cross Score	Calc m/z	Diff (ppm)	Mass Match	Abund Match	Spacing Match
☑	C30 H48 O3	C30 H49 O3	95.4		457.3676	1.47	97.86	99.01	86.12

Isotope	Abund Sum%	Calc Abund Sum%	m/z	Calc m/z	Diff (ppm)
1	70.36	71.53	457.367	457.3676	1.31
2	24.32	23.69	458.3686	458.371	5.21
3	4.57	4.24	459.3715	459.3742	5.87
4	0.75	0.54	460.3715	460.3772	12.39



^{1}H NMR (400 MHz) spectrum of compound **2** in $CDCl_3$

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NAME                ZQ-10
EXPNO                   1
PROCNO                  1
Date_            20160913
Time                 4.53
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                  36.09
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                 11.50 usec
SI                 65536
SF            400.1300041 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```
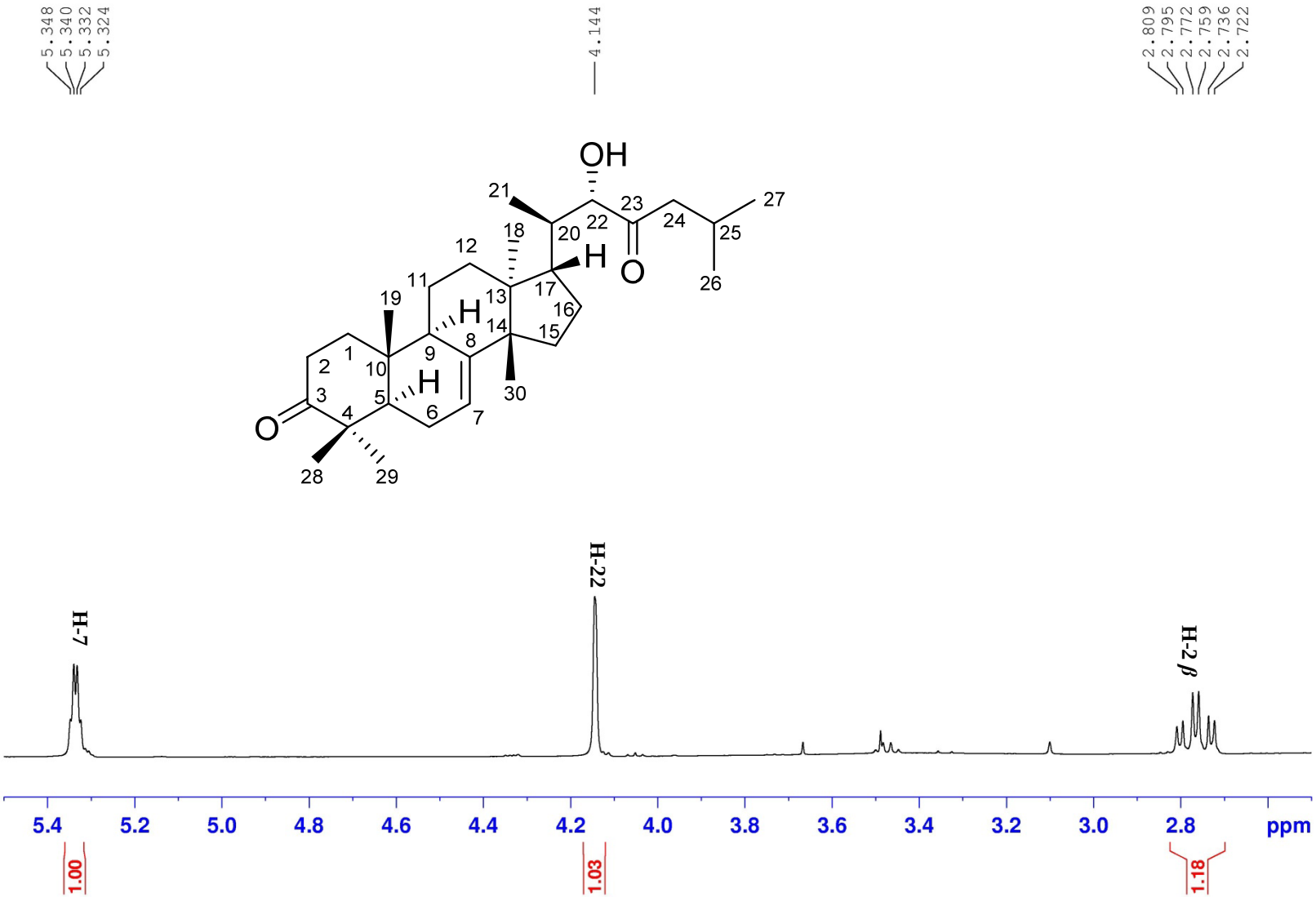
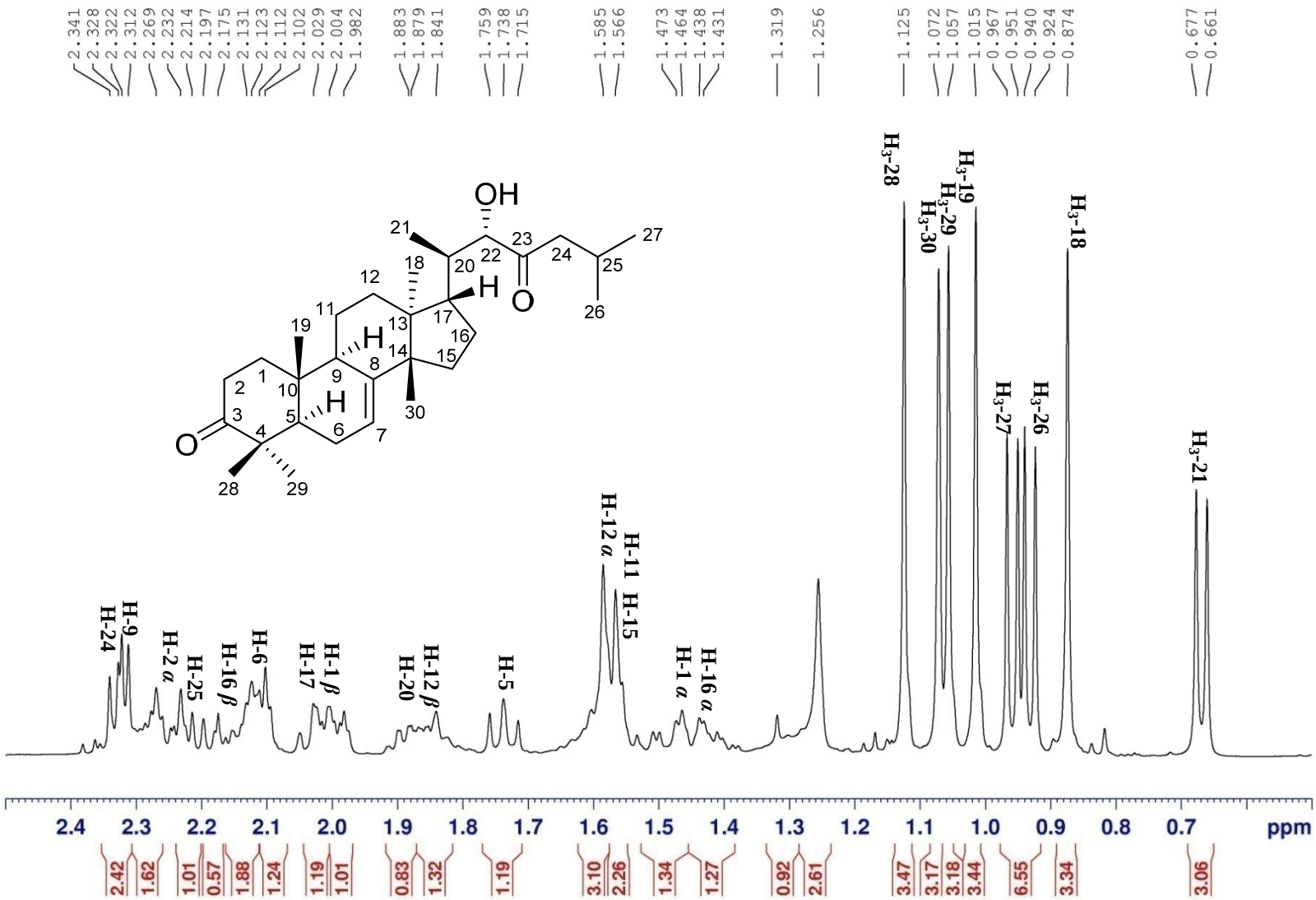
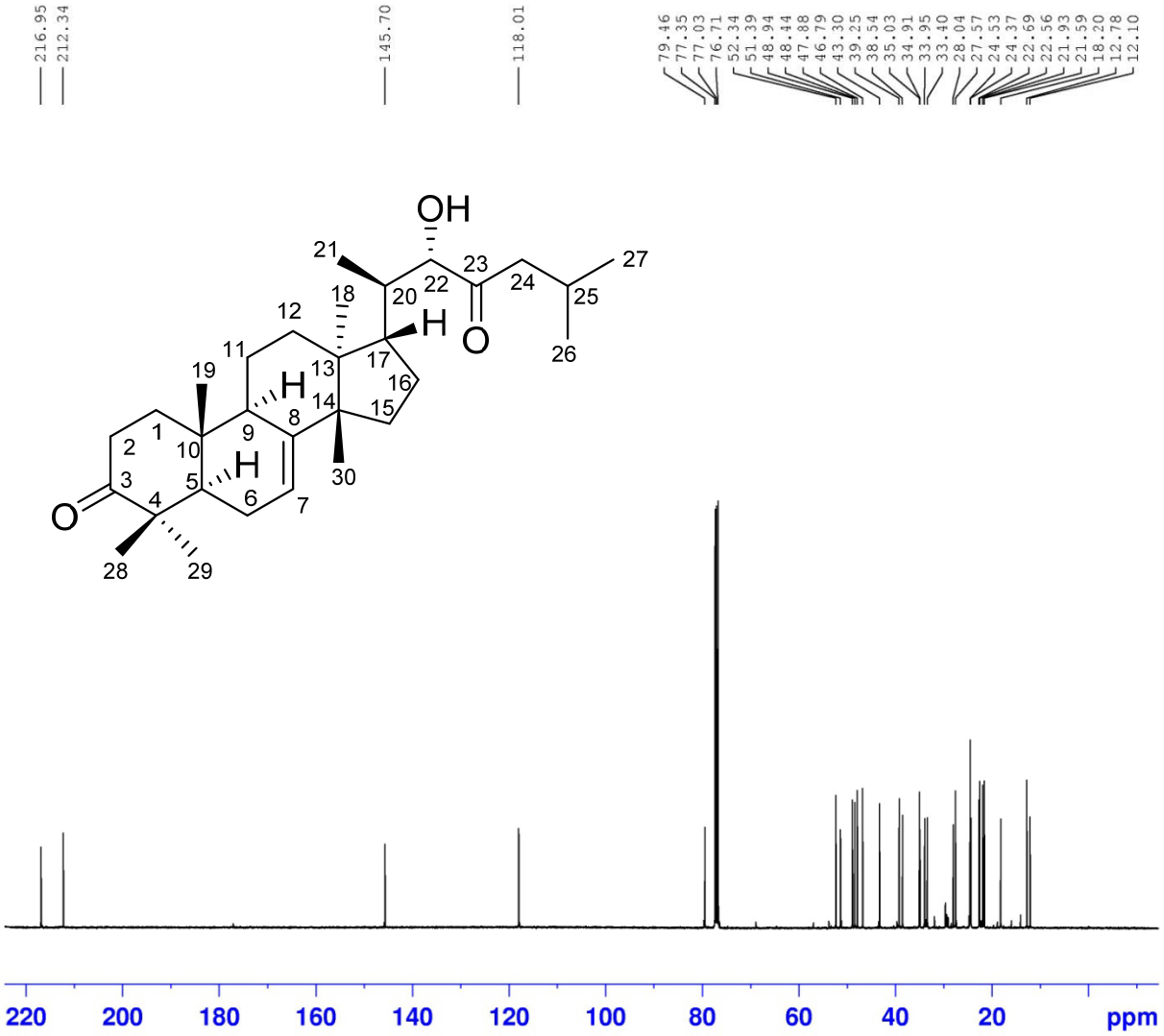

^{1}H NMR (400 MHz) spectrum of compound **2** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **2** in $CDCl_3$

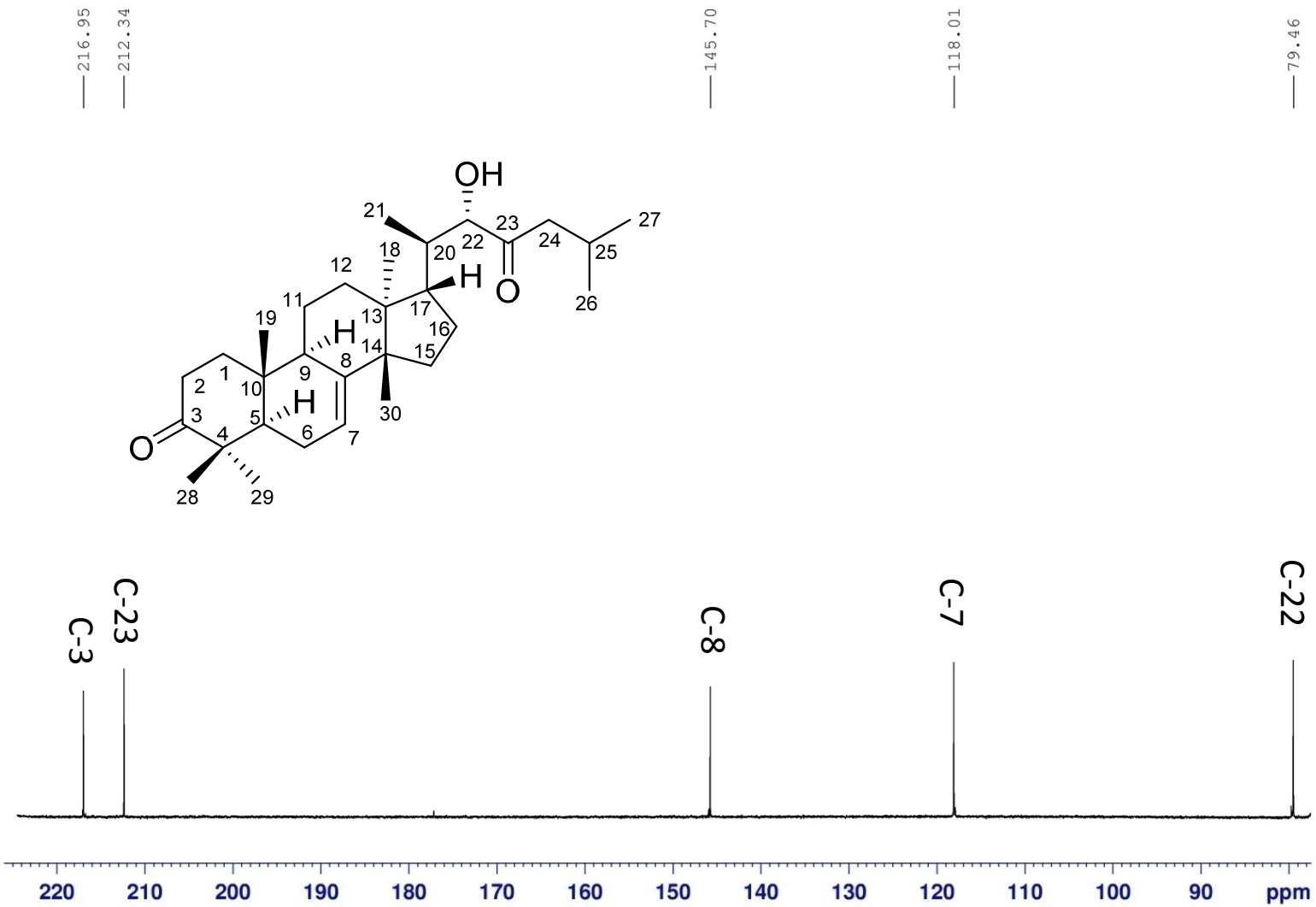


^{13}C NMR (100 MHz) spectrum of compound **2** in $CDCl_3$

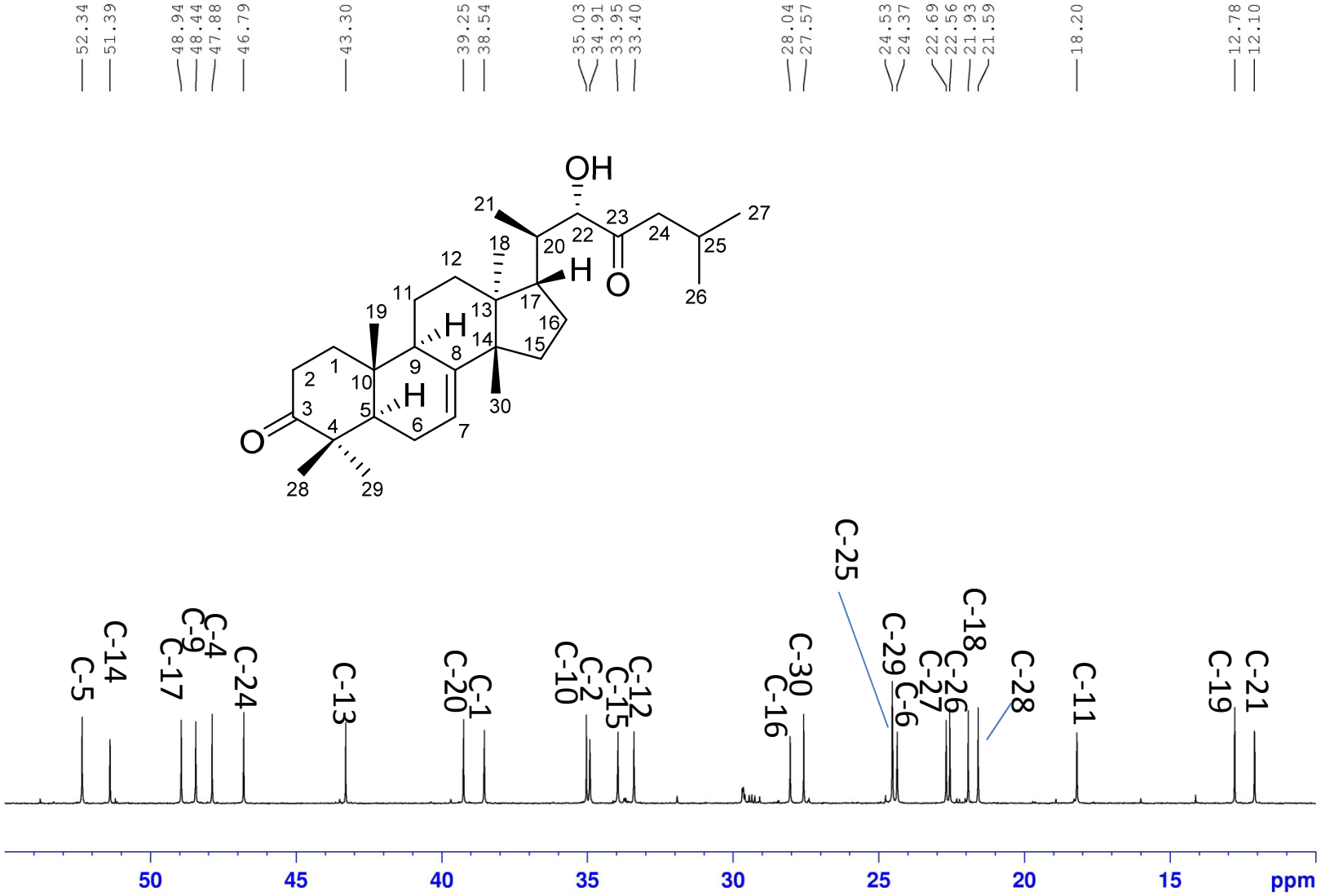



```
NAME            ZQ-10-1
EXPNO                 2
PROCNO                1
Date_          20160913
Time               5.53
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG               73.92
DW               20.800 usec
DE               18.00 usec
TE               297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6233324 MHz
NUC1                 13C
P1               10.00 usec
SI               32768
SF          100.6127698 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

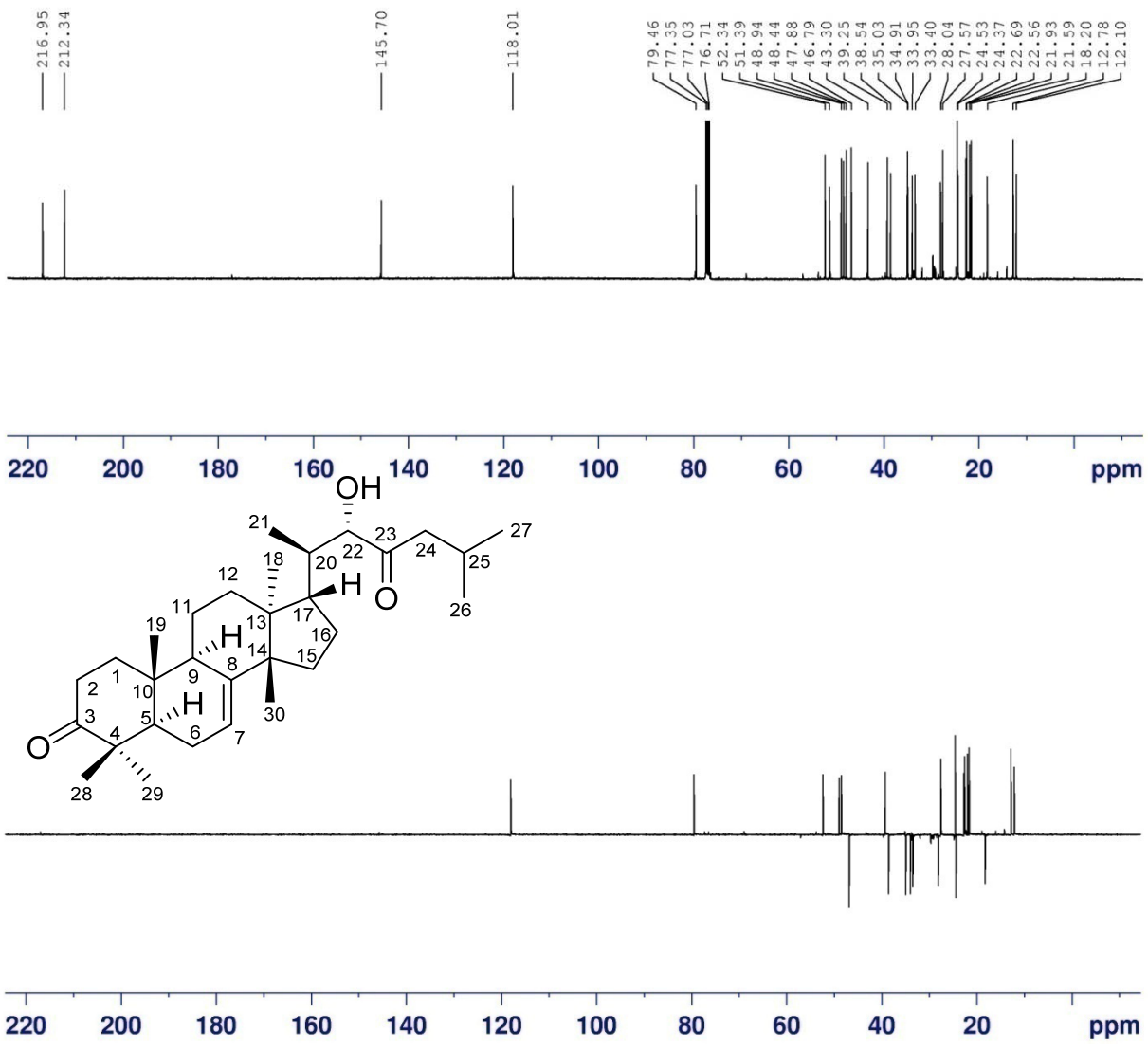
^{13}C NMR (100 MHz) spectrum of compound **2** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **2** in $CDCl_3$

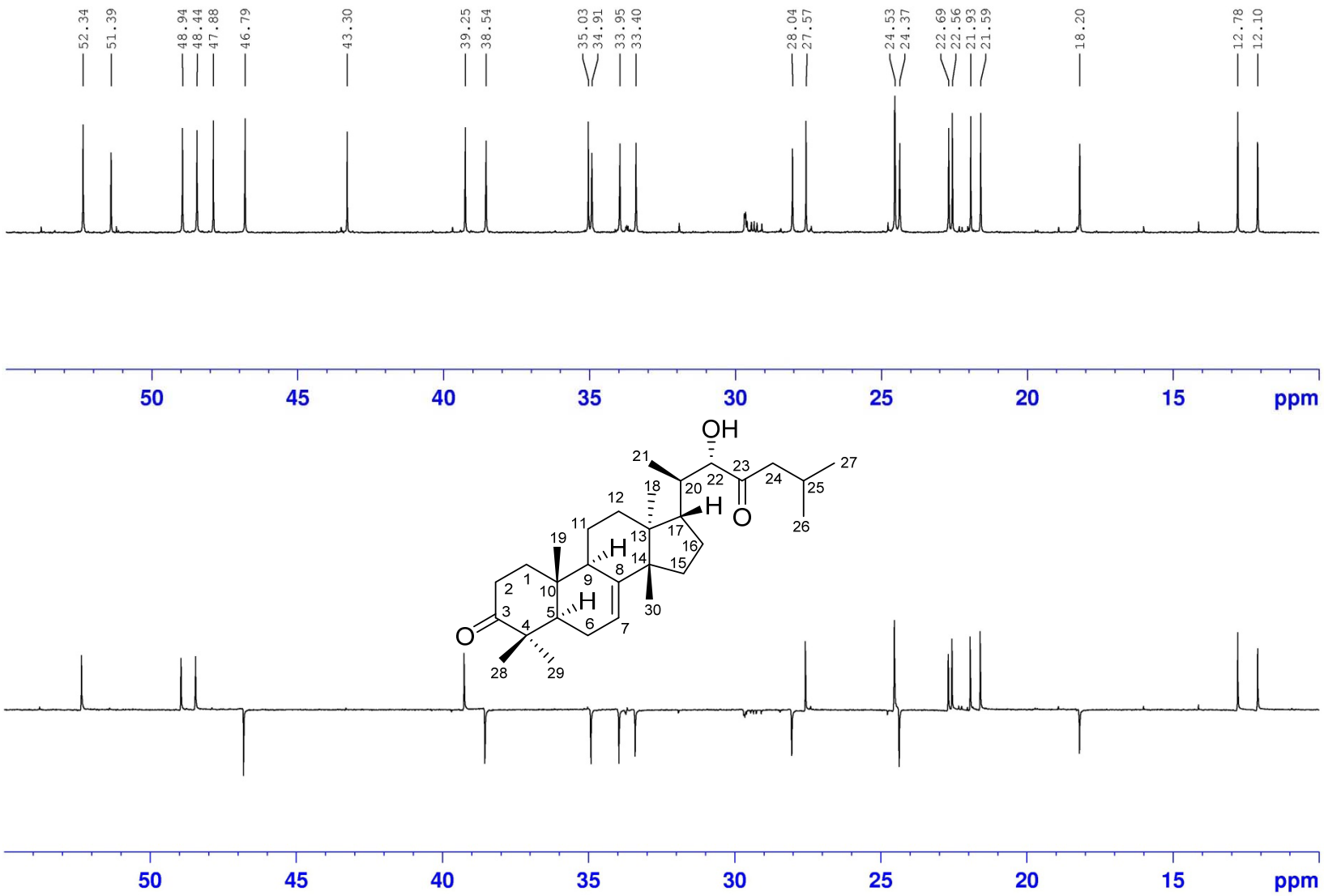


DEPT135 (100 MHz) spectrum of compound **2** in $CDCl_3$

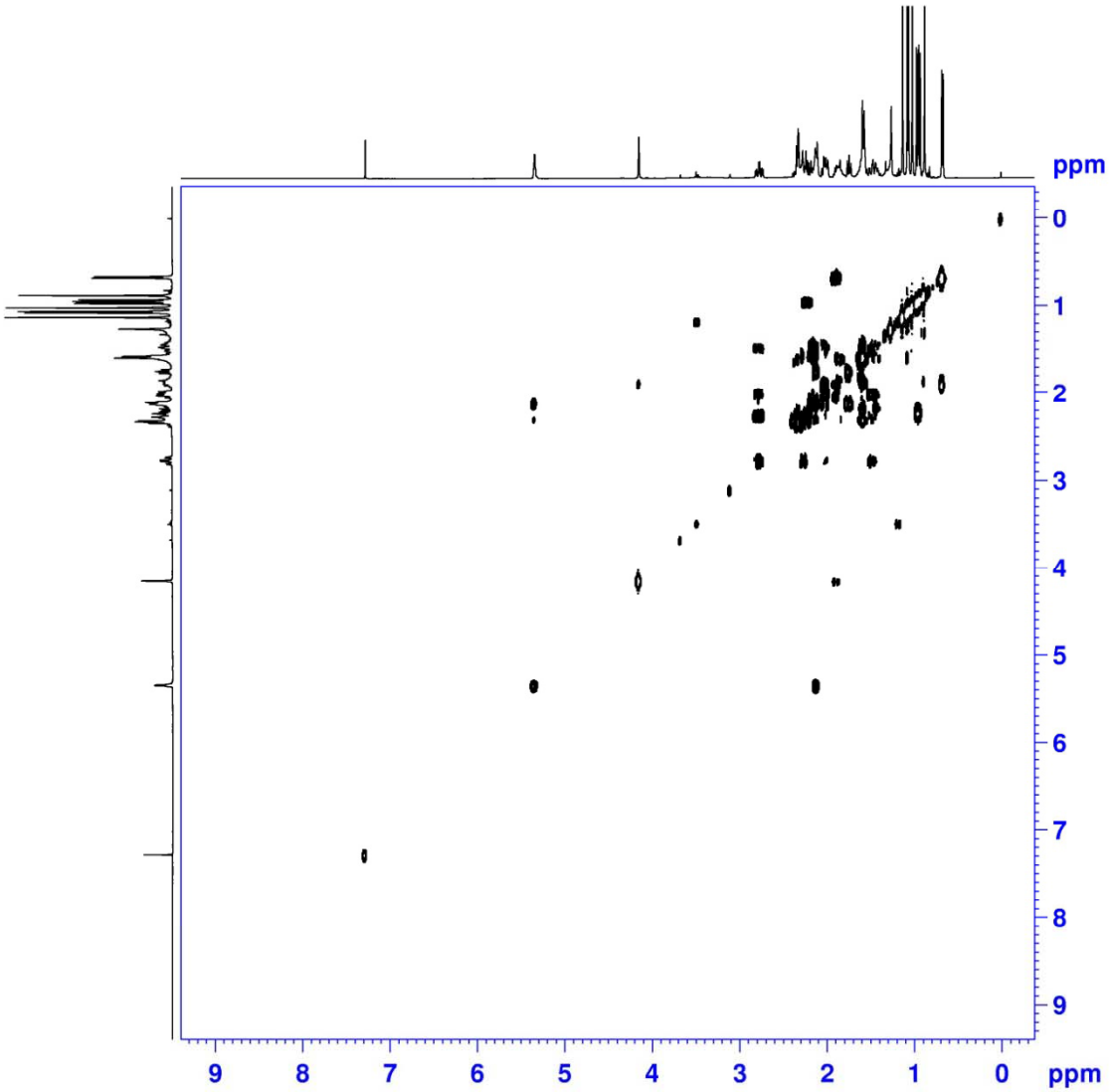



```
NAME            ZQ-10-1
EXPNO                 3
PROCNO                1
Date_          20160913
Time               6.11
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG       deptsp135
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH           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 ========
SFO1        100.6233324 MHz
NUC1                13C
P1                10.00 usec
P13             2000.00 usec
SI                32768
SF          100.6127698 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

DEPT135 (100 MHz) spectrum of compound **2** in $CDCl_3$

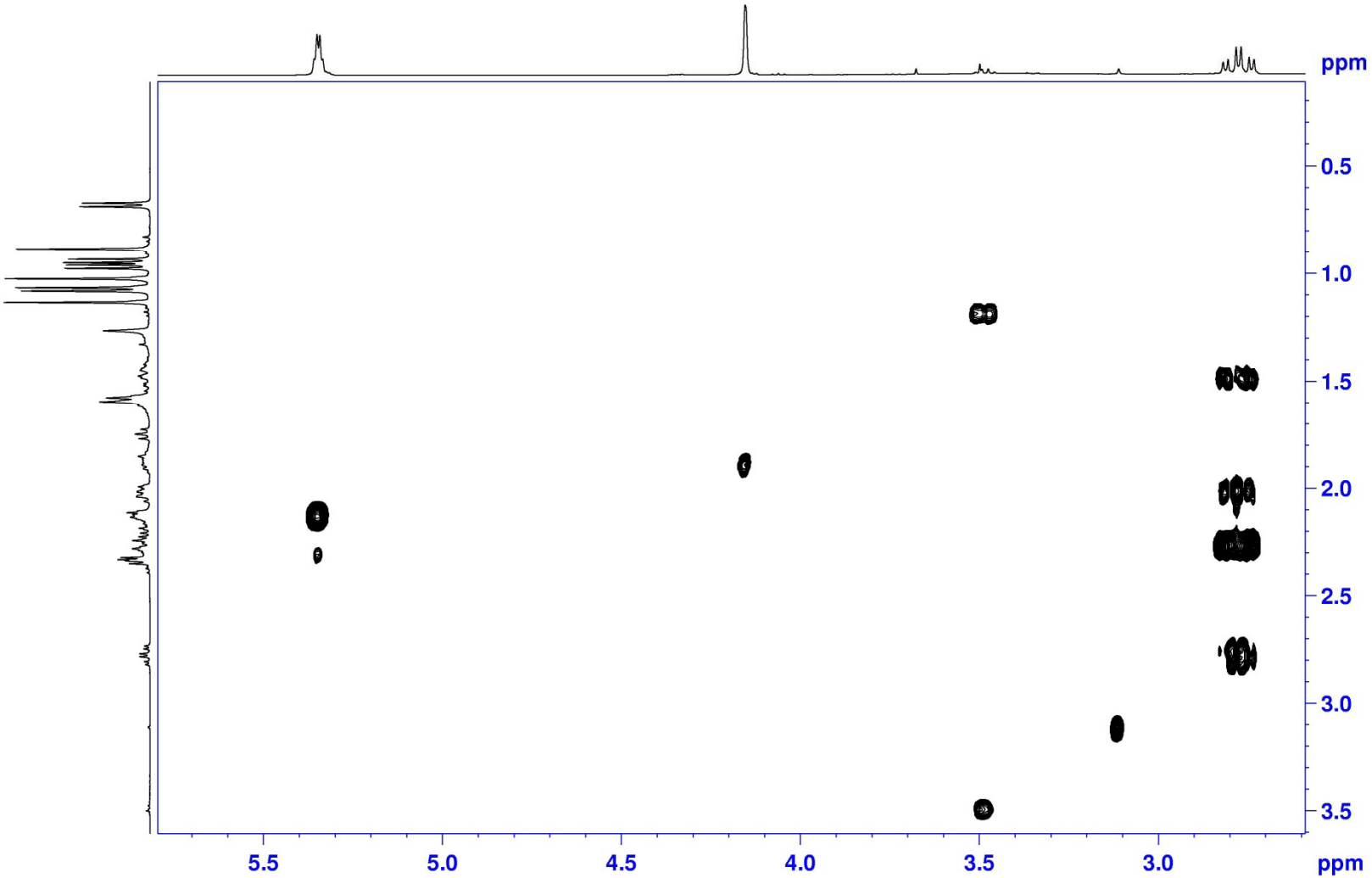


^{1}H-^{1}H COSY (400 MHz) spectrum of compound **2** in $CDCl_3$

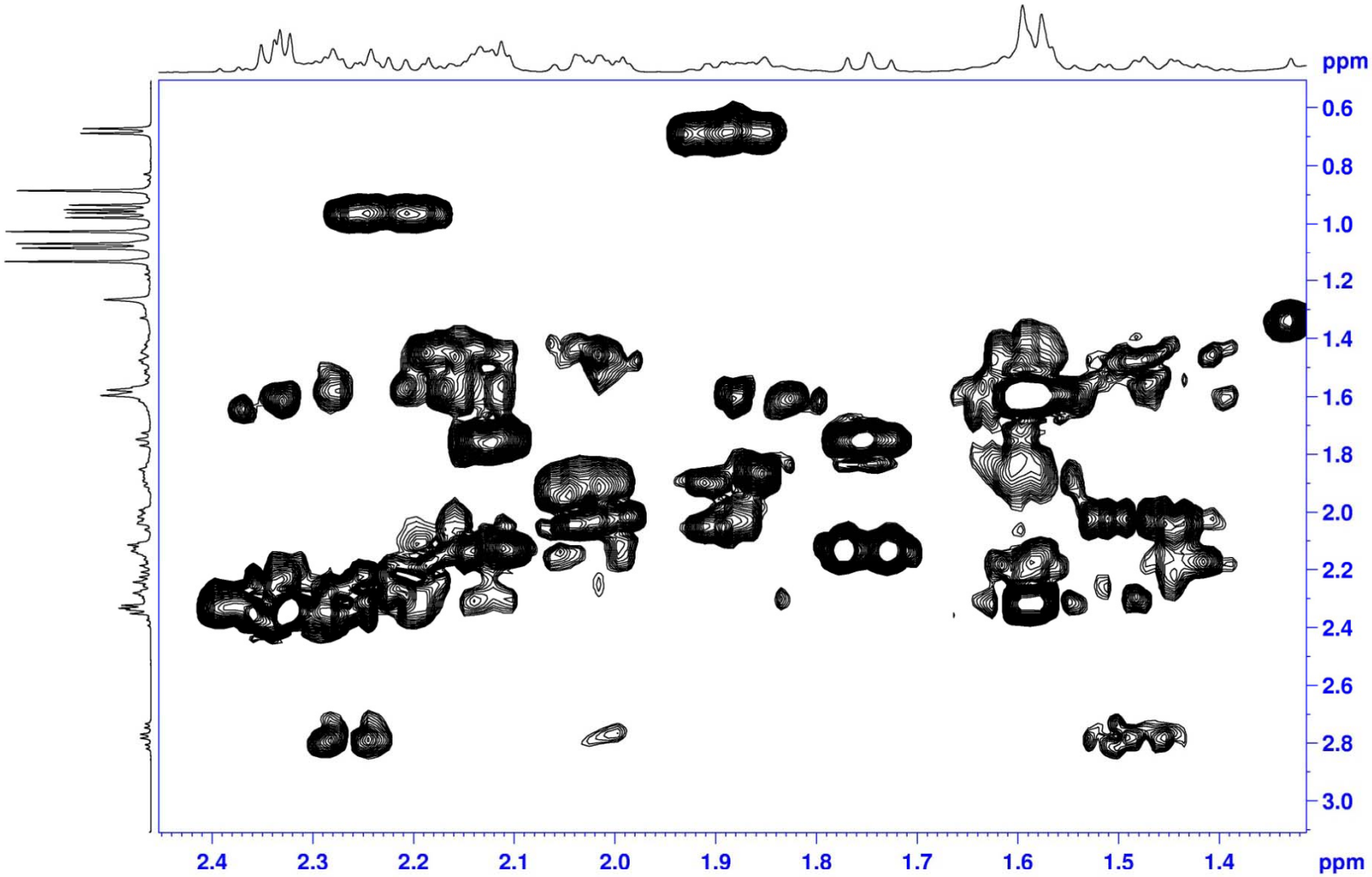



```
NAME            ZQ-10-1
EXPNO                 4
PROCNO                1
Date_          20160929
Time              12.09
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG      cosygpppqf
TD                 2048
SOLVENT           CDCl3
NS                    8
DS                    8
SWH            3906.250 Hz
FIDRES         1.907349 Hz
AQ            0.2621940 sec
RG              36.09
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          400.1299966 MHz
WDW               QSINE
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2                  QF
SF          400.1299937 MHz
WDW               OSINE
```

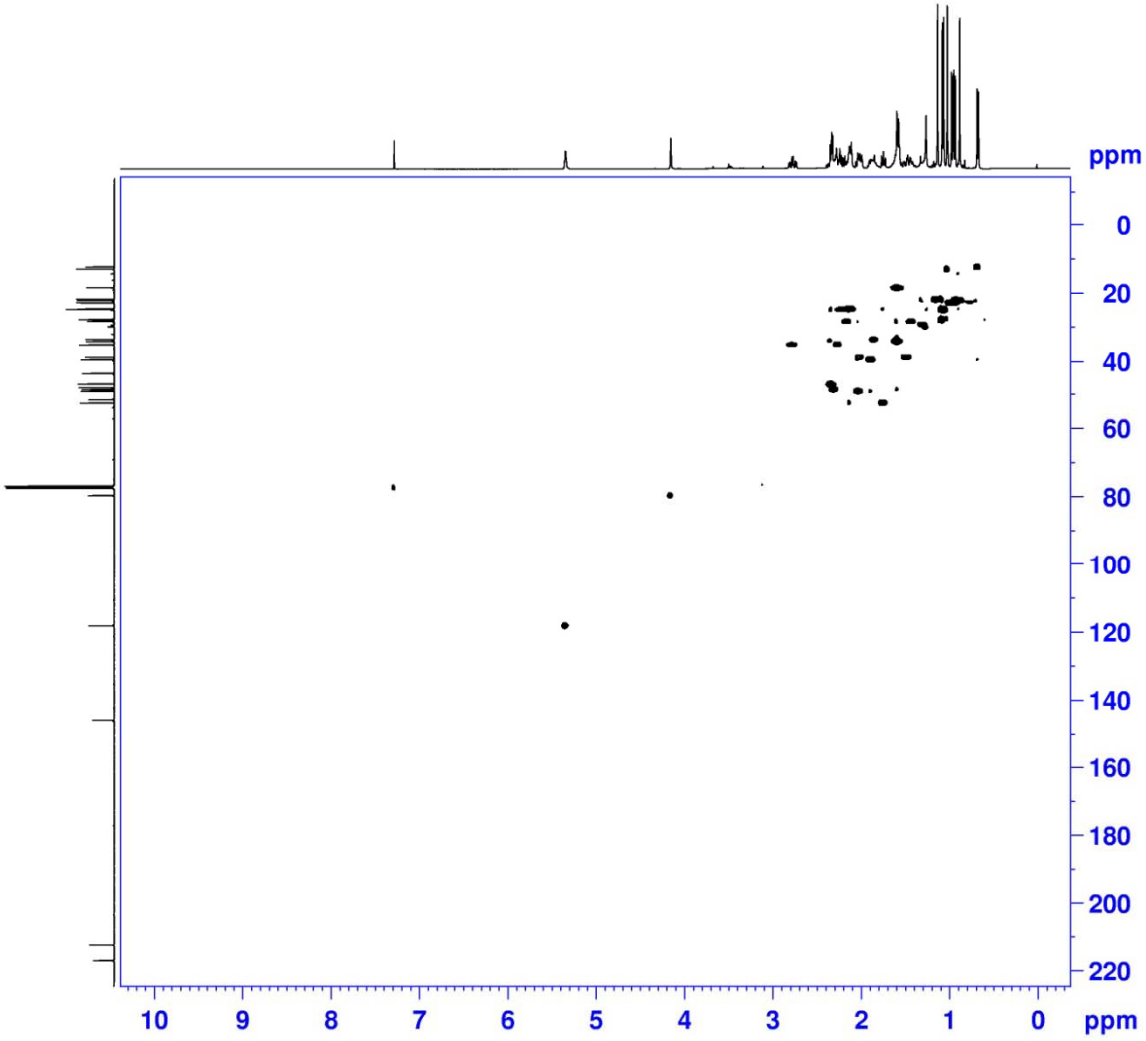
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **2** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **2** in $CDCl_3$

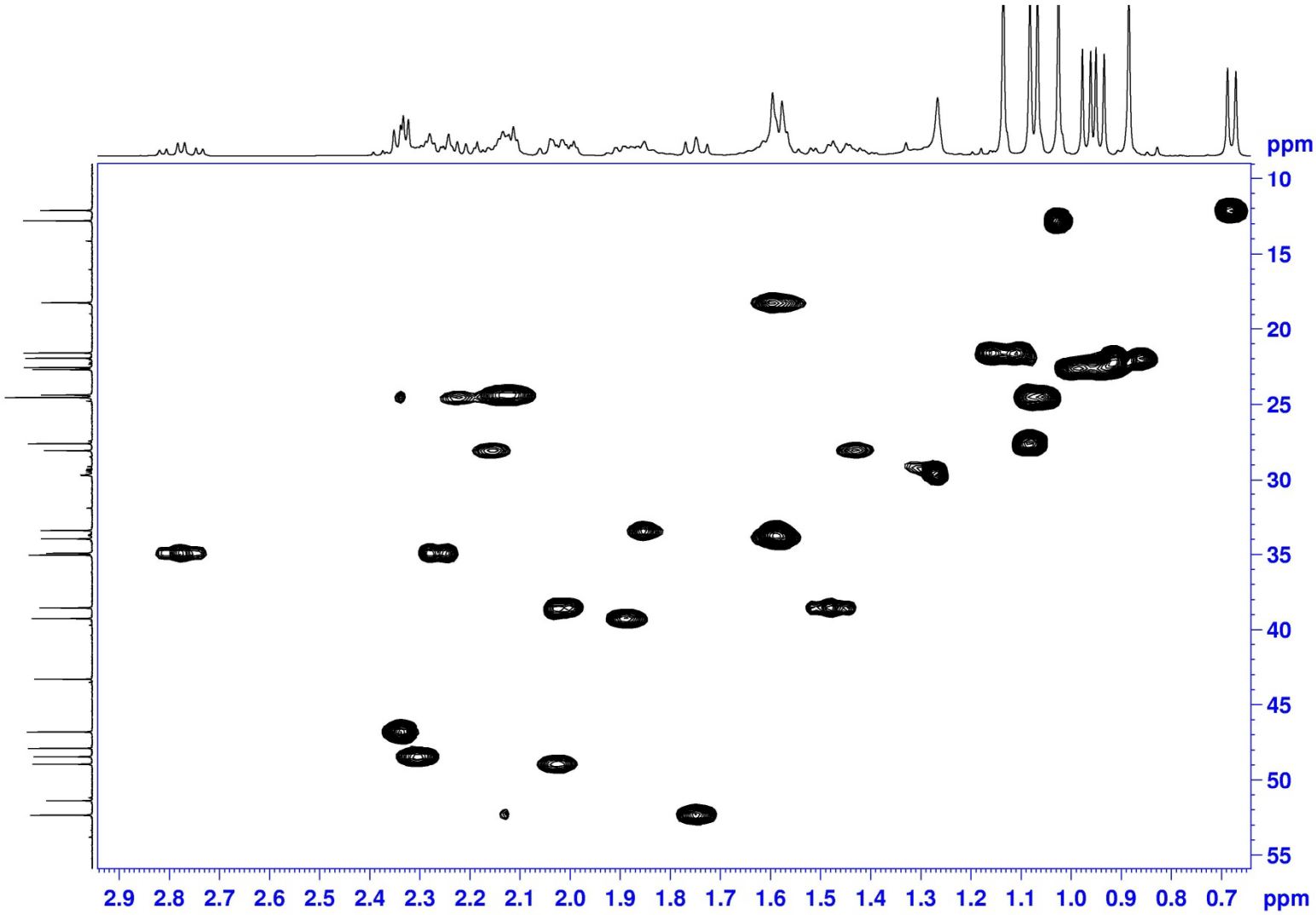


HSQC (400 MHz) spectrum of compound **2** in $CDCl_3$

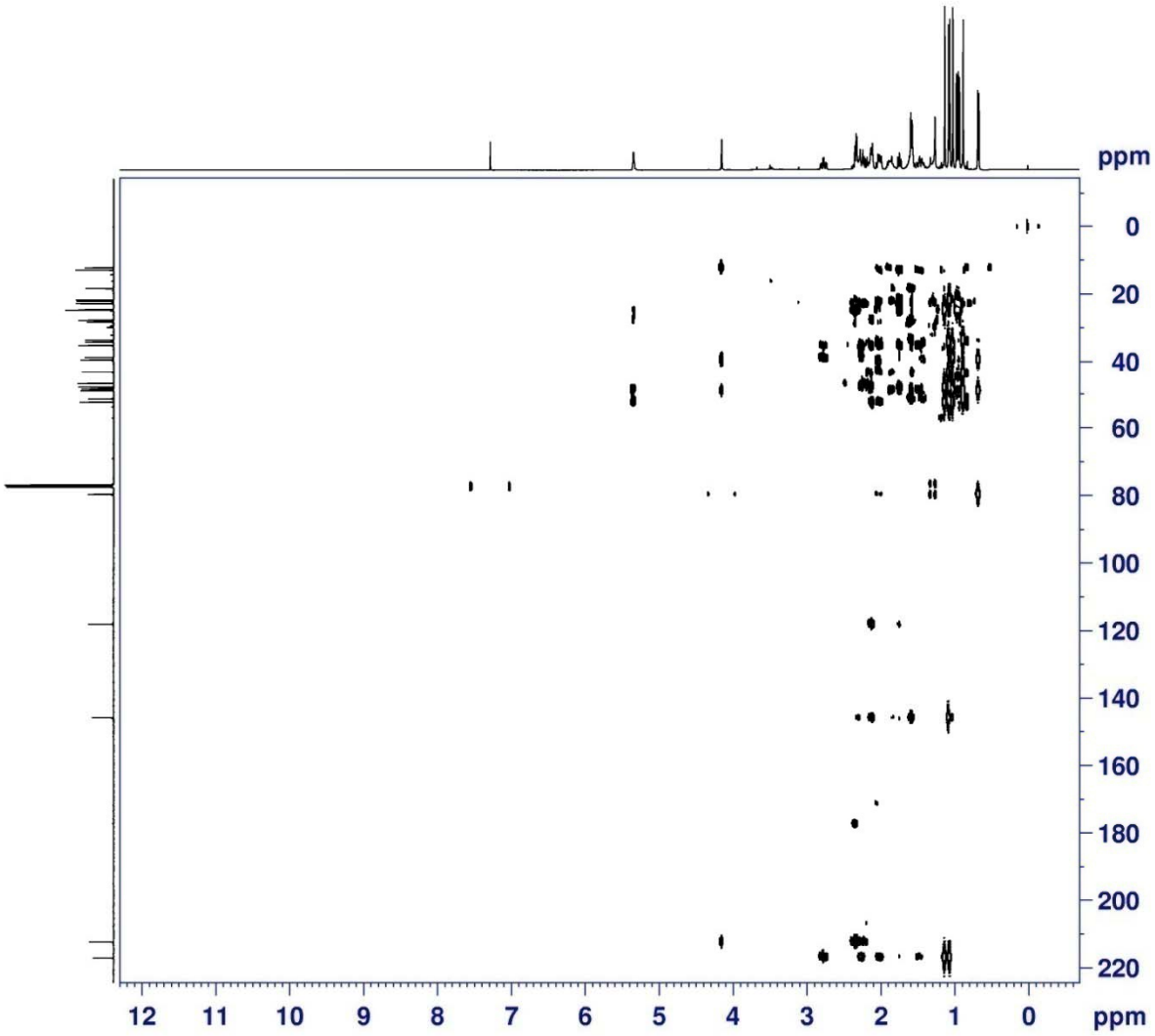



```
NAME              ZQ-10-1
EXPNO                   5
PROCNO                  1
Date_            20160929
Time                12.48
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      hsqcetgpsi2
TD                   1024
SOLVENT             CDCl3
NS                     16
DS                     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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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           400.1299963 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2        echo-antiecho
SF           100.6127561 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
```

HSQC (400 MHz) spectrum of compound **2** in $CDCl_3$

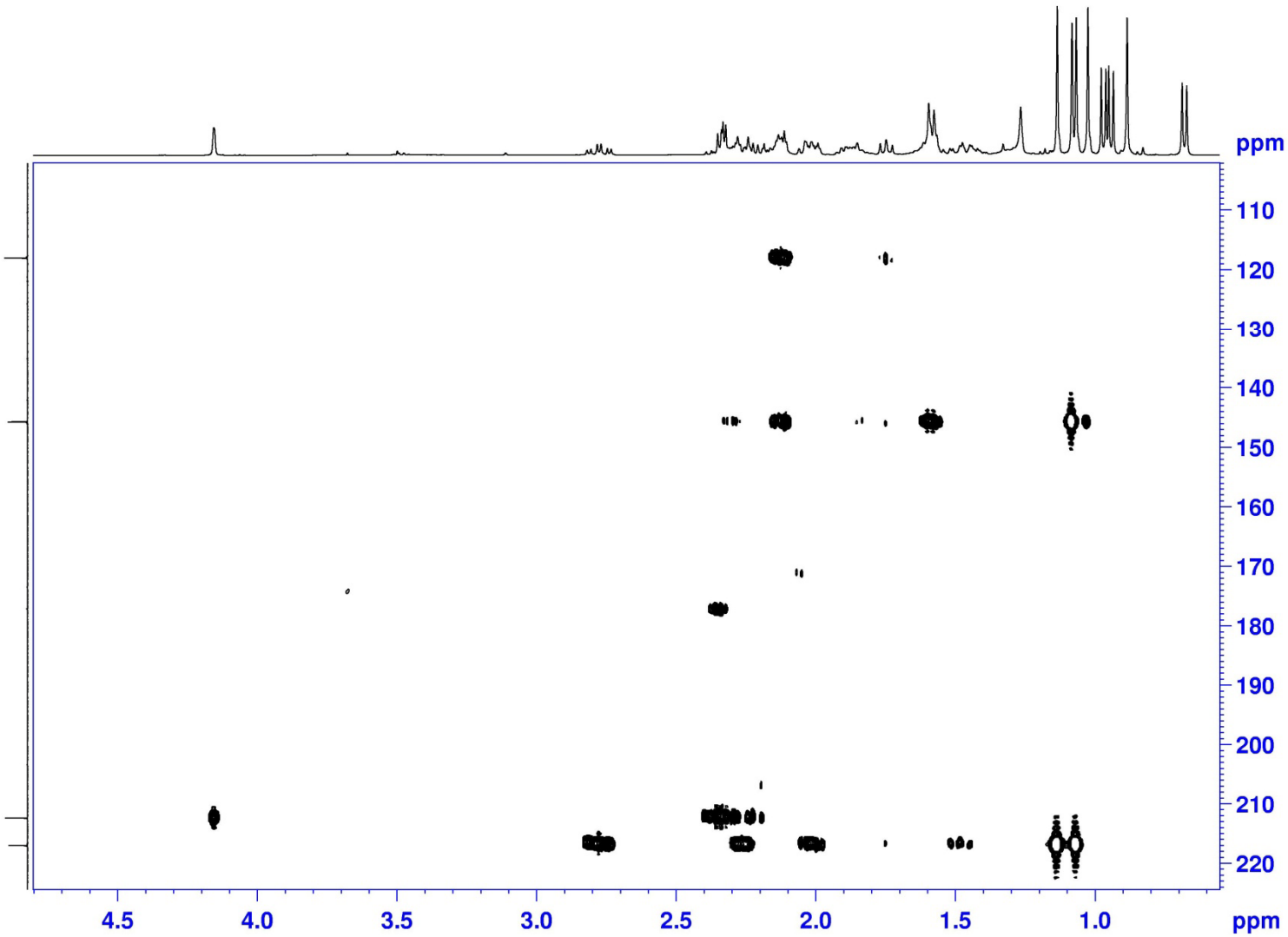


HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$

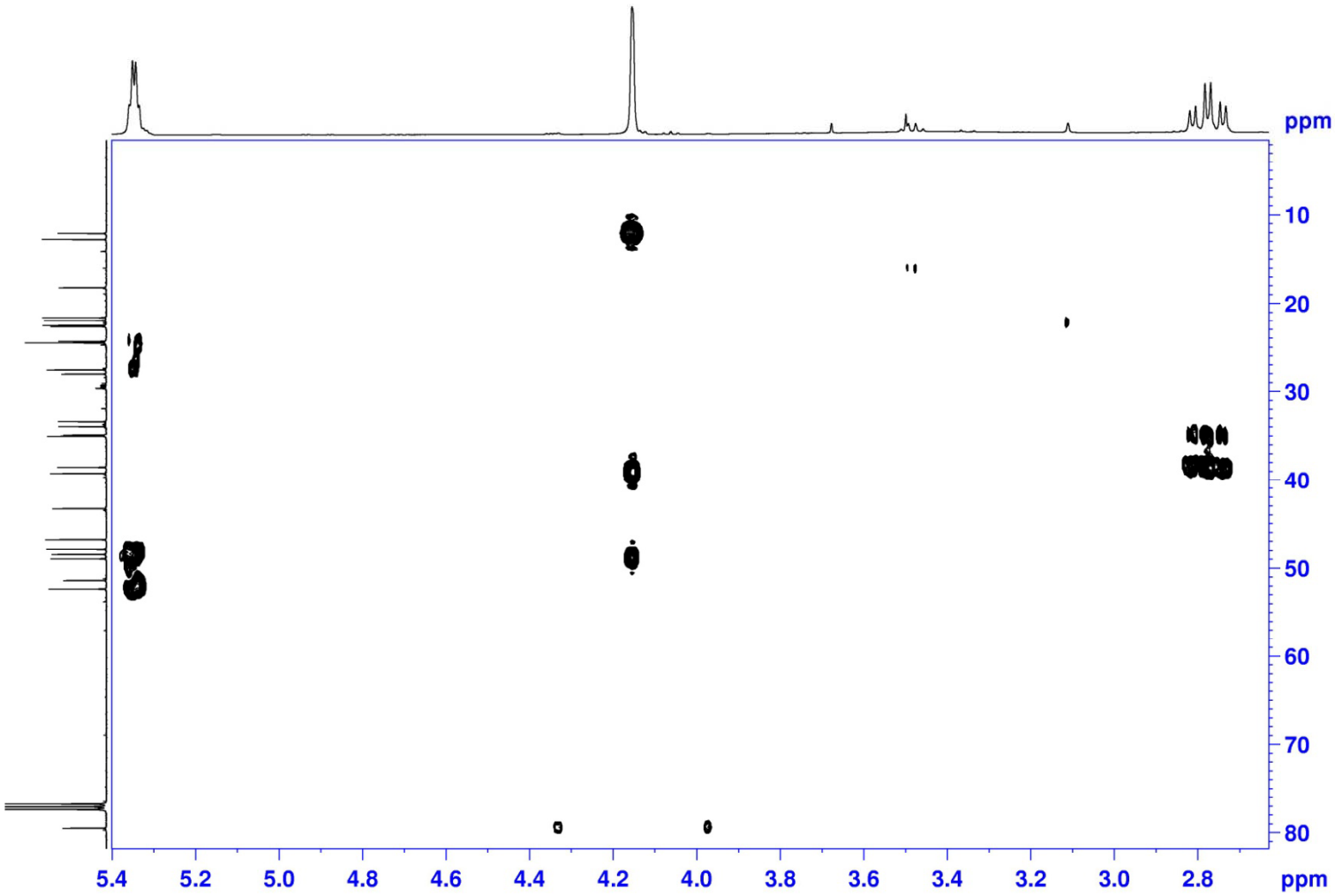



```
NAME                ZQ-10-1
EXPNO                     6
PROCNO                    1
Date_              20160929
Time                  14.40
INSTRUM               spect
PROBHD     5 mm CPPBBO BB
PULPROG      hmbcgplpndqf
TD                     4096
SOLVENT              CDCl3
NS                        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 ========
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
SI                     2048
SF              400.1299977 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40
SI                     1024
MC2                      QF
SF              100.6127709 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
```

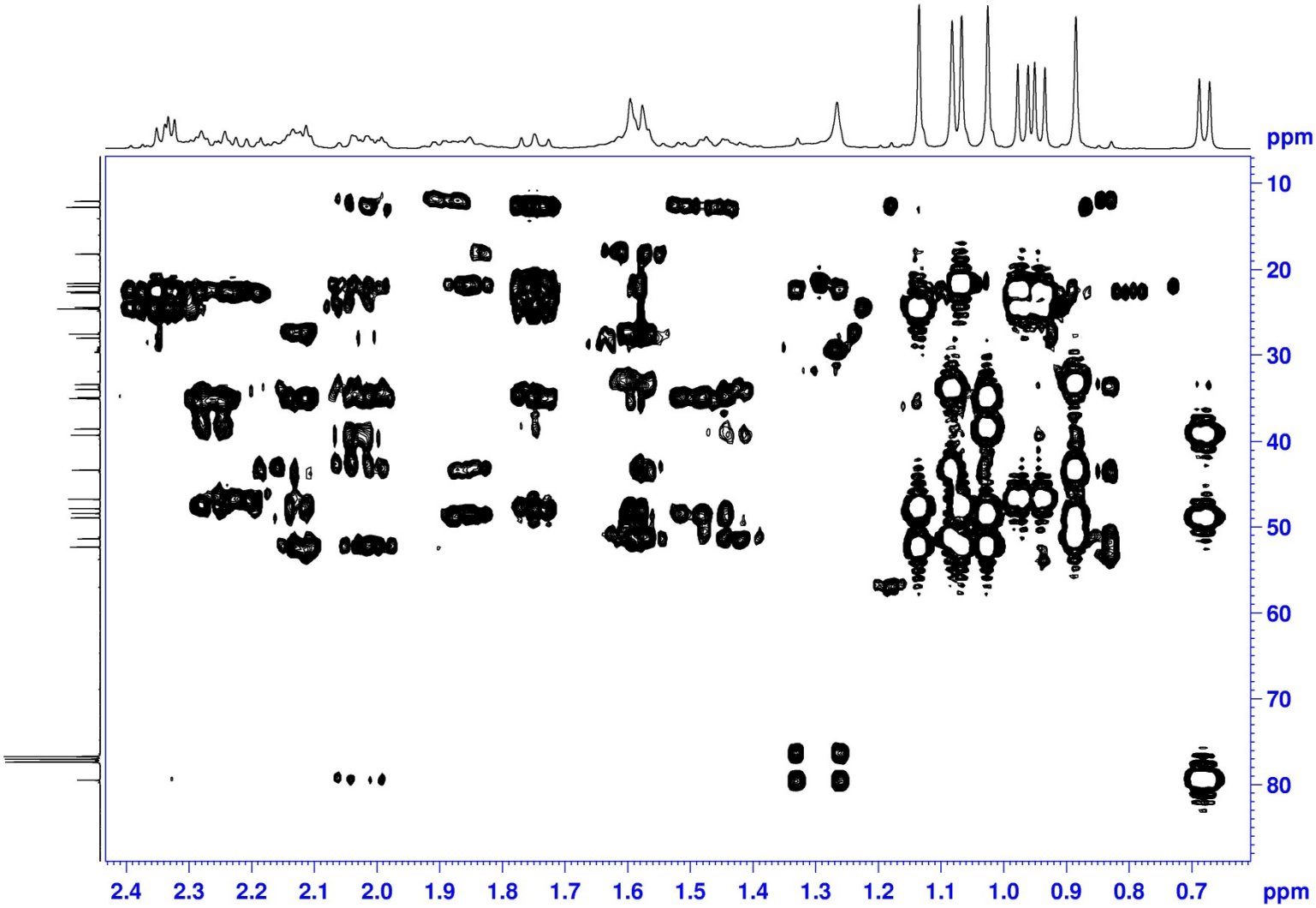
HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$



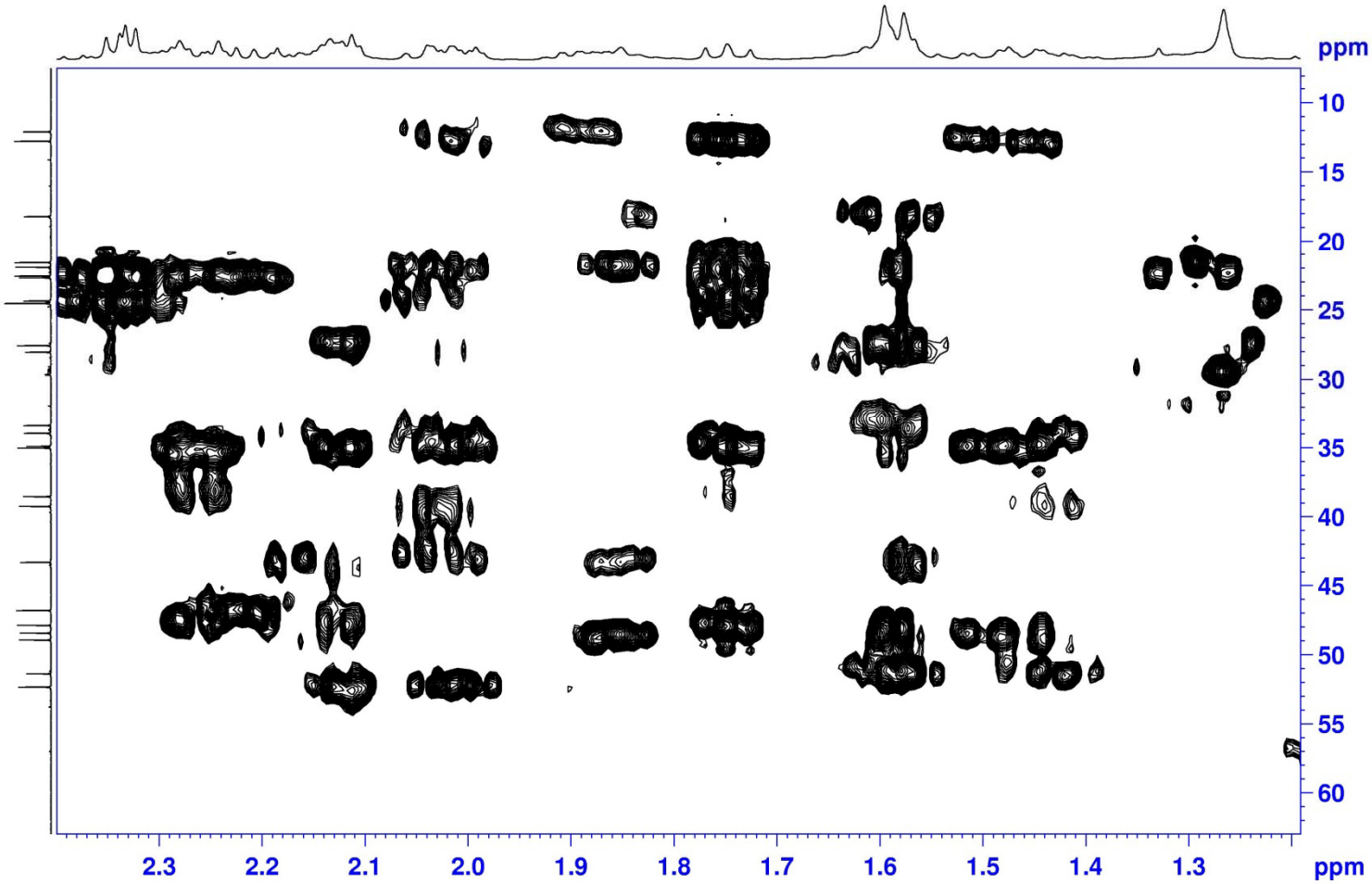
HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$



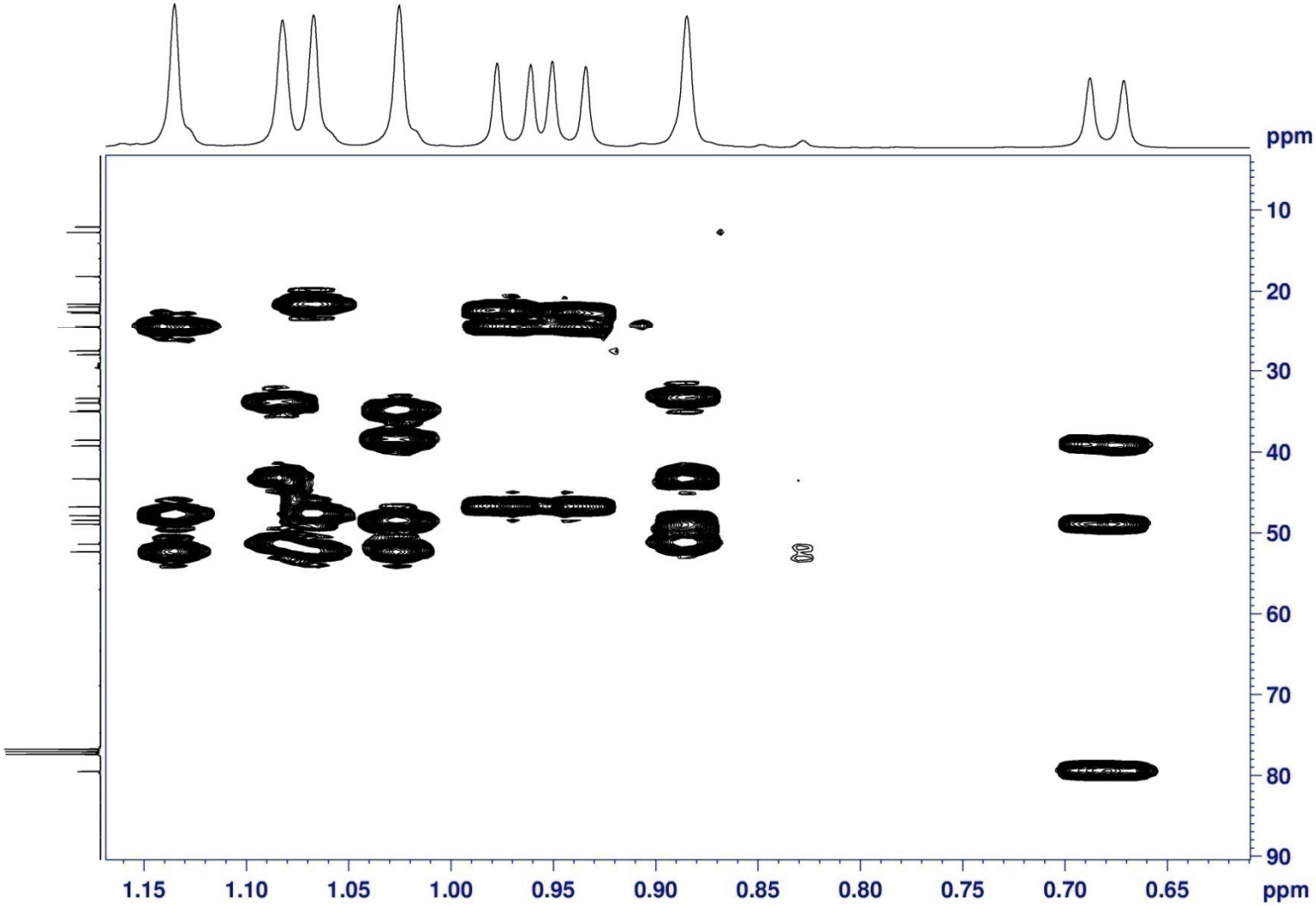
HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$



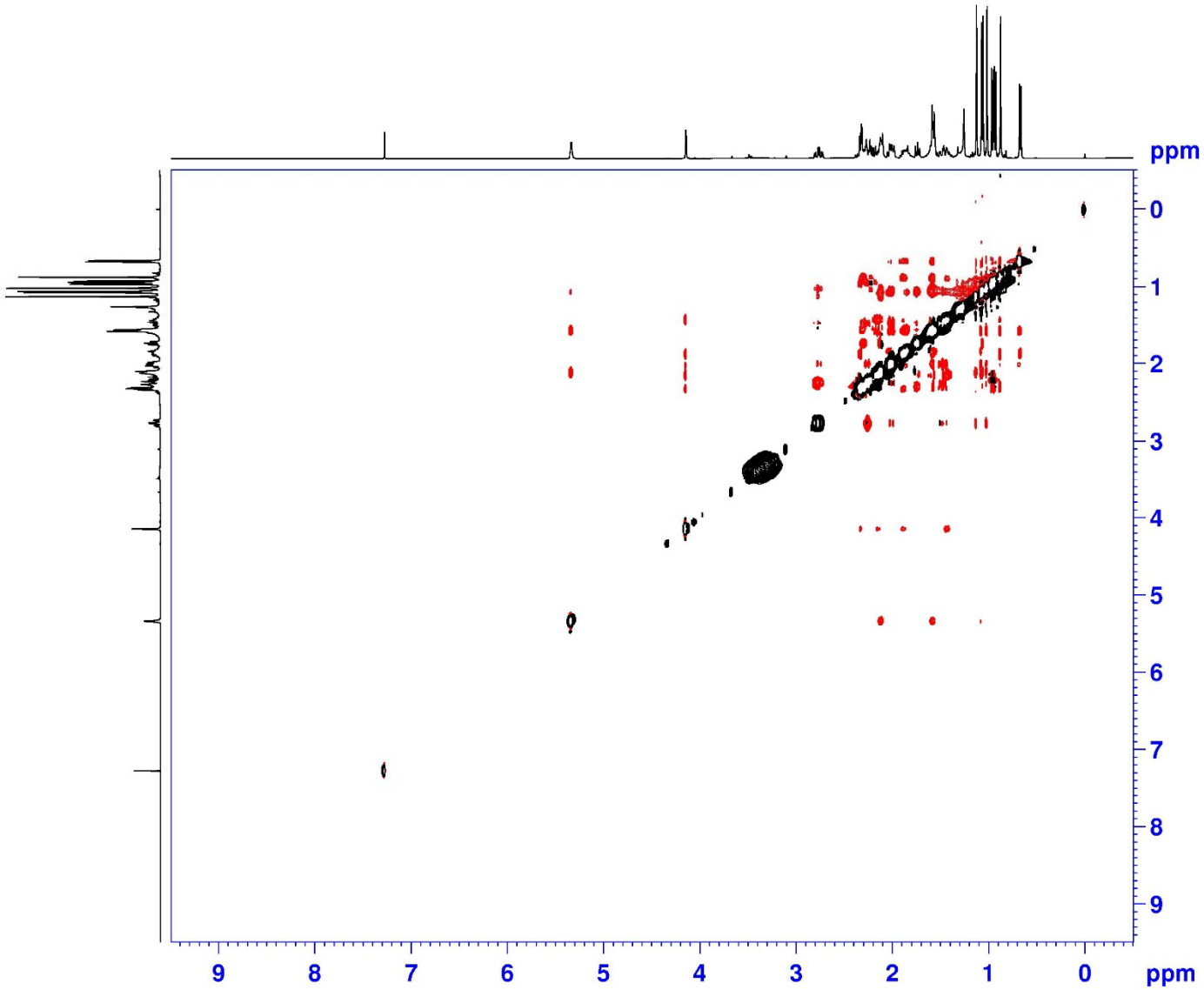
HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$



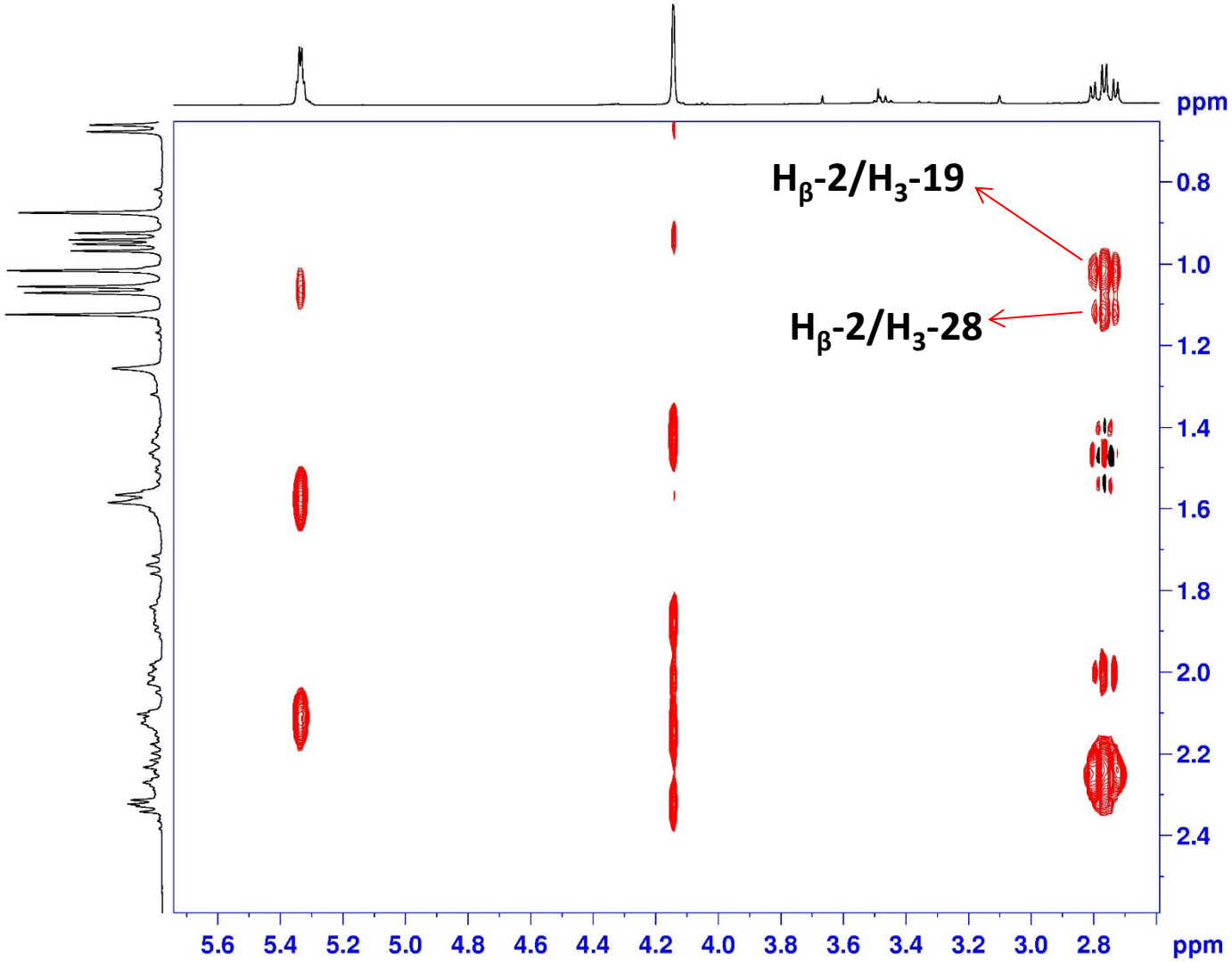
HMBC (400 MHz) spectrum of compound **2** in $CDCl_3$



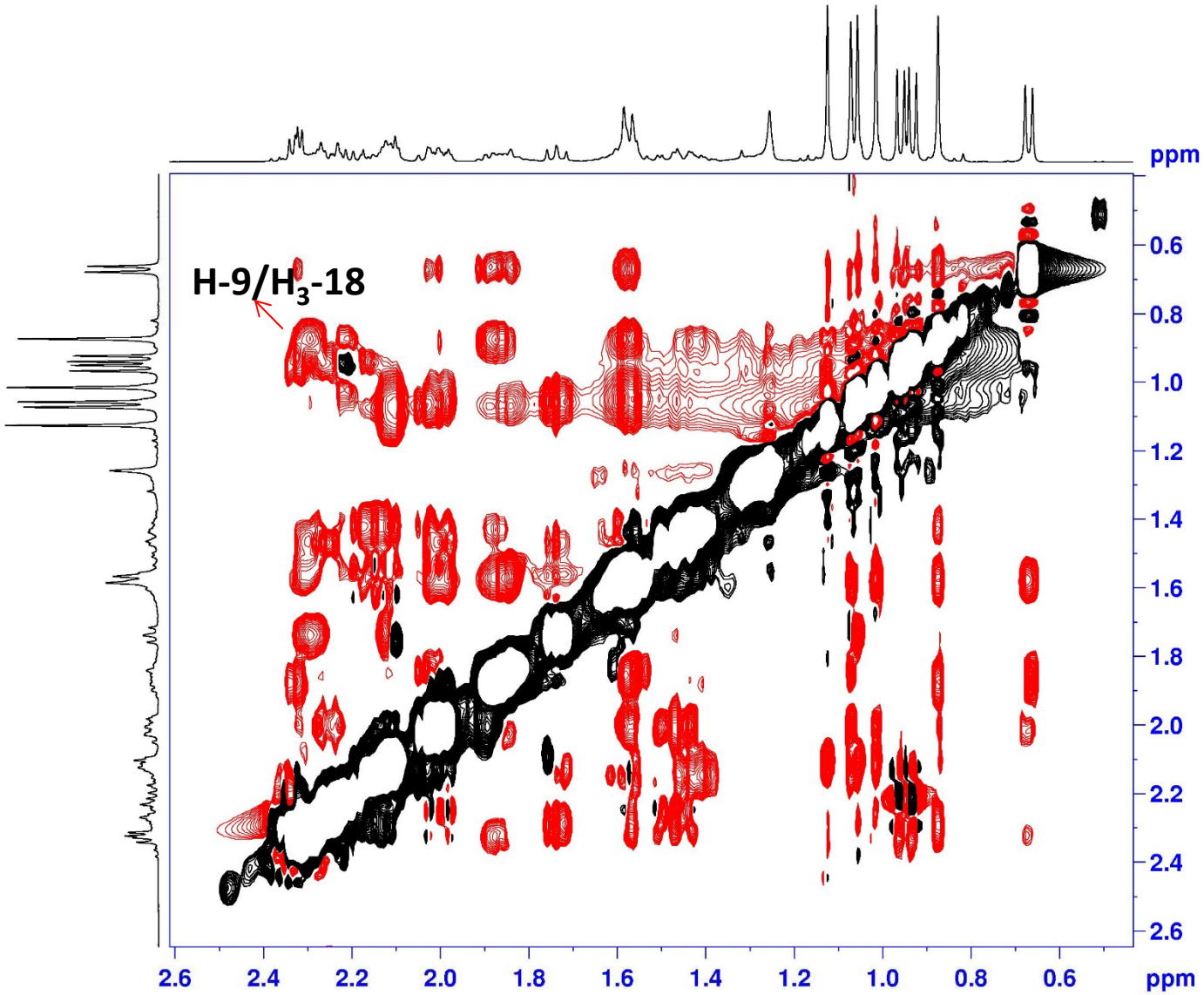
NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



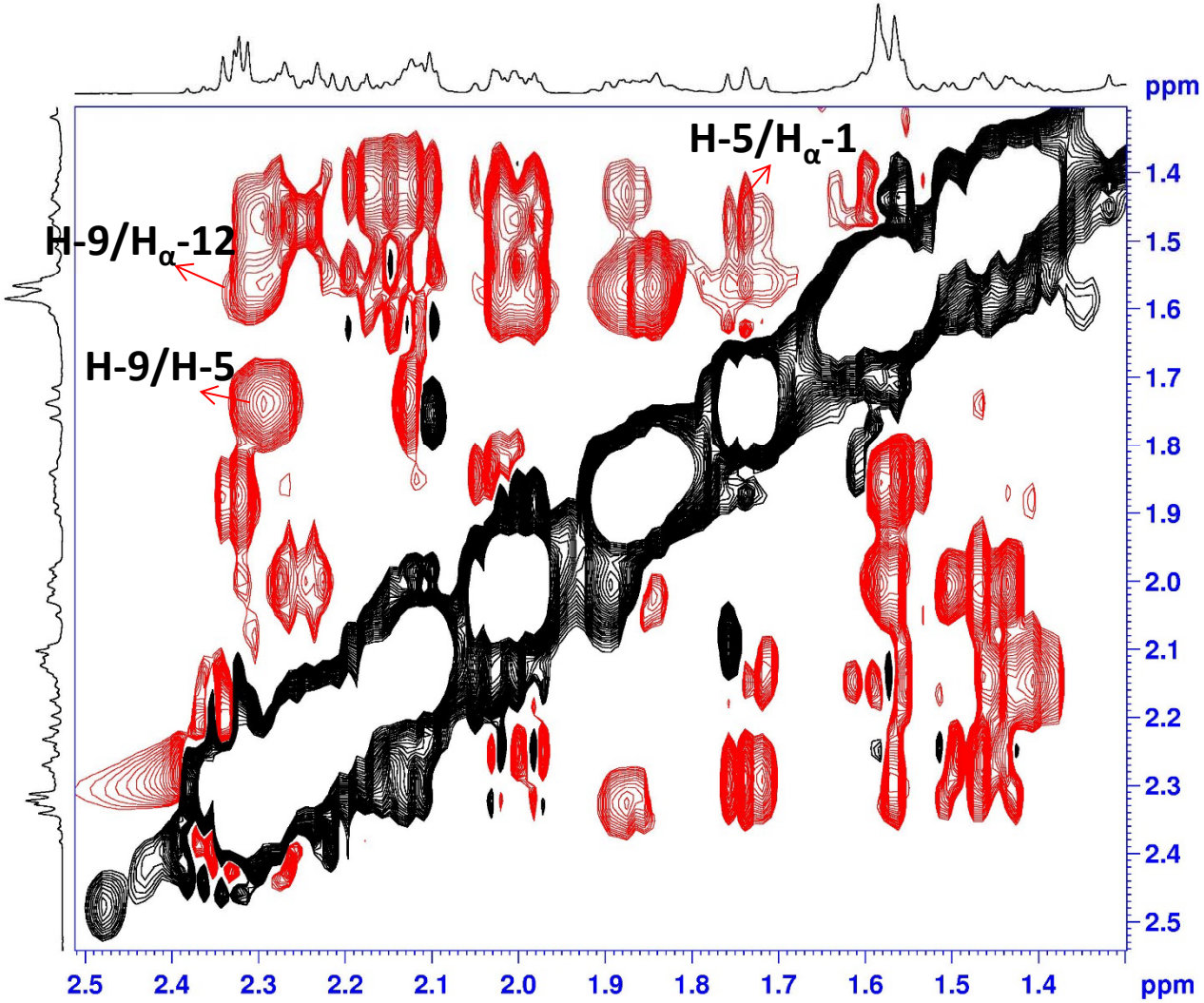
NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



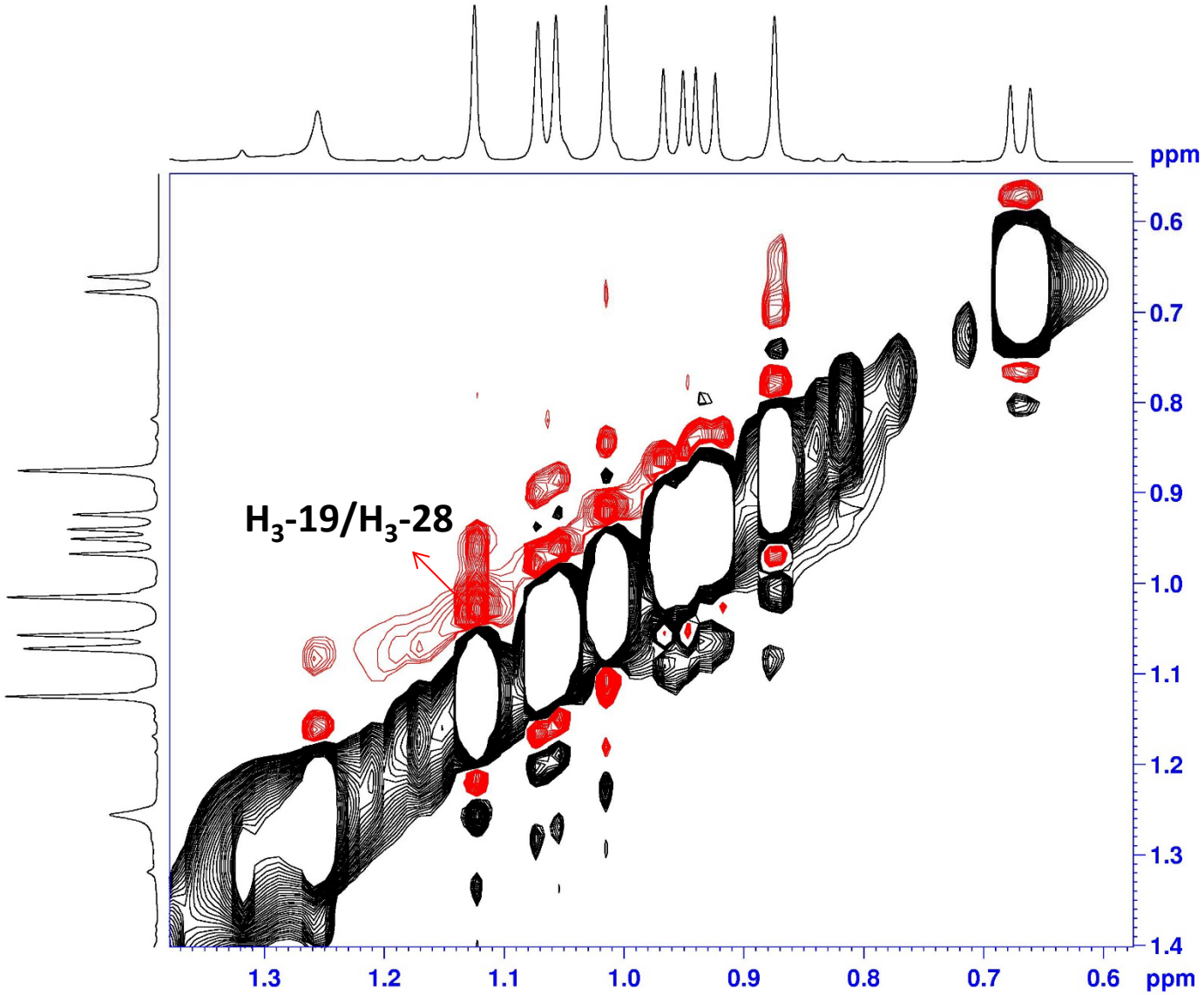
NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



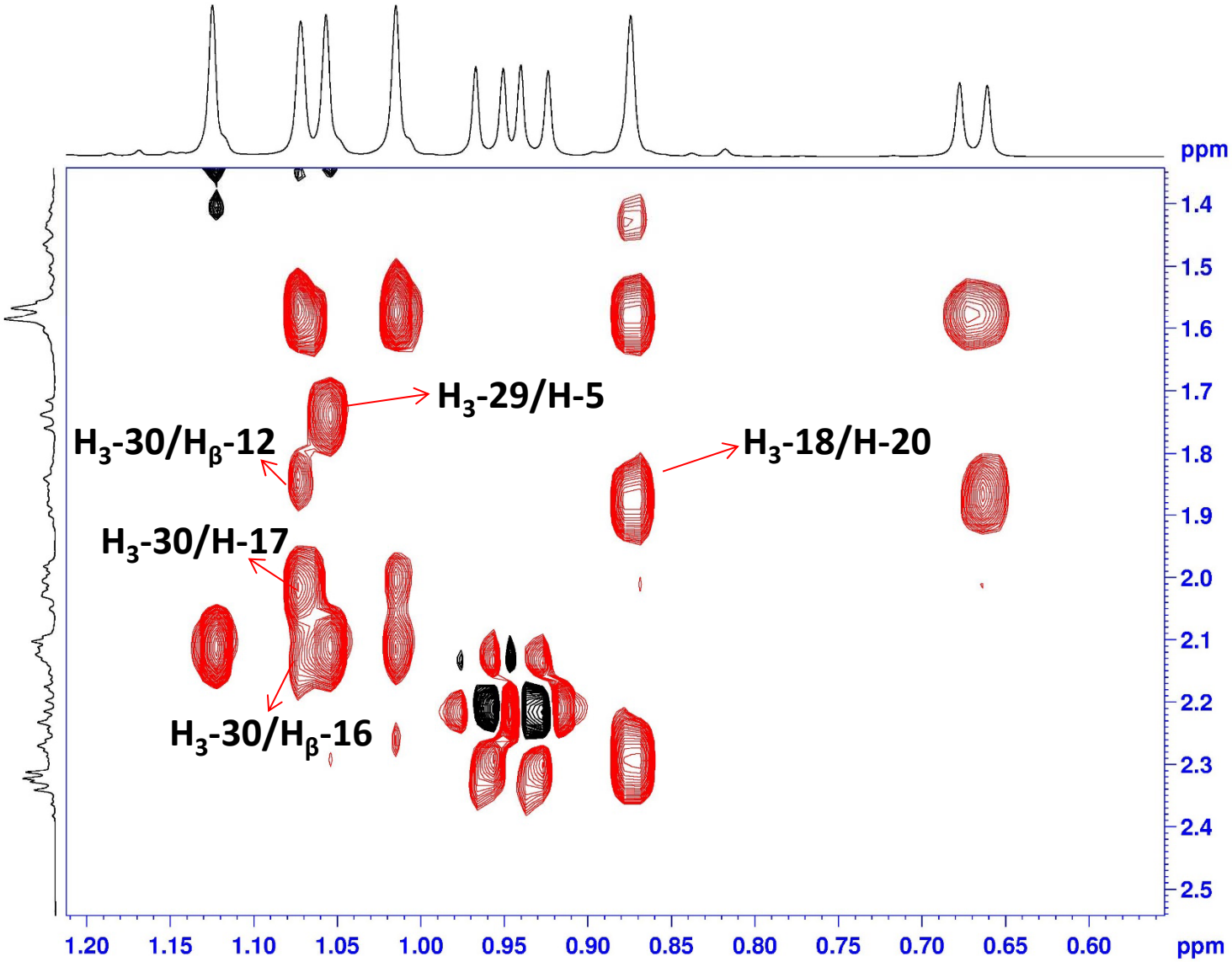
NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



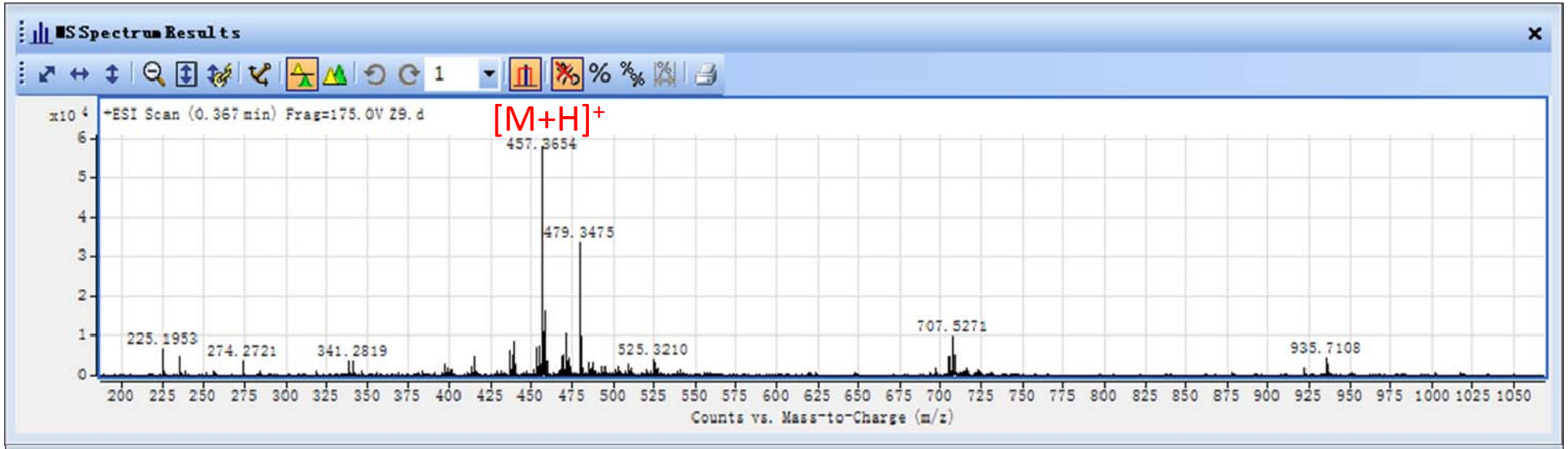
NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **2** in $CDCl_3$



HR-ESIMS for compound **3**

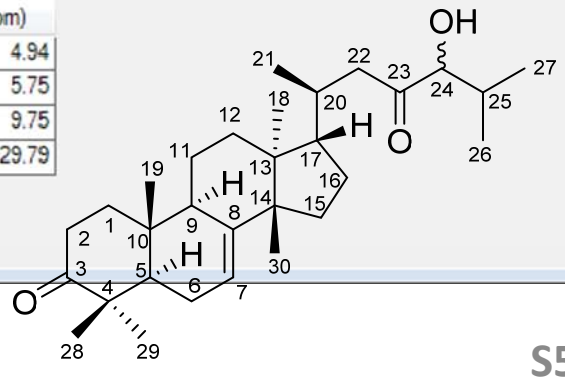


MS Formula Results: + Scan (0.367 min)

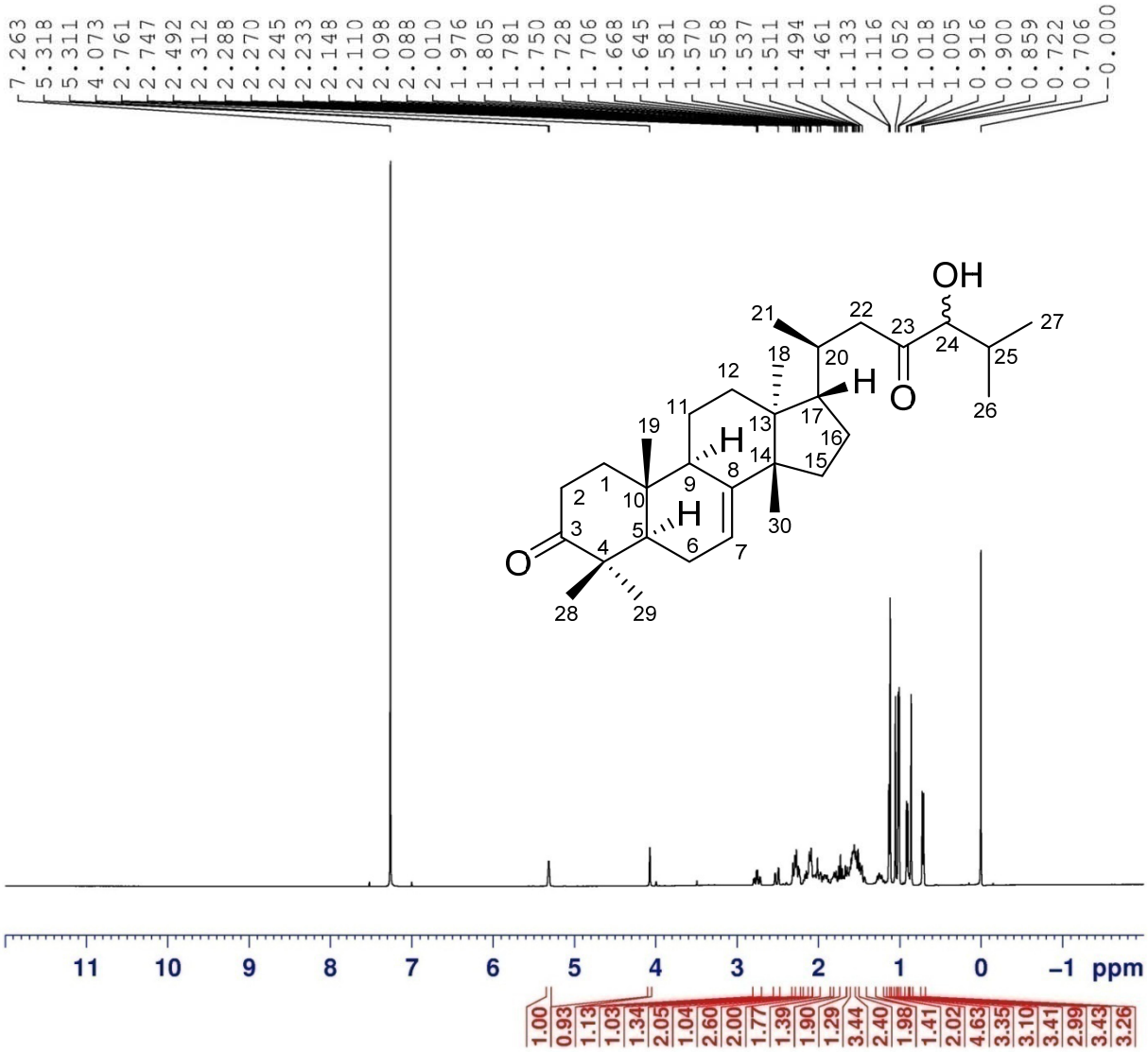
m/z	Ion	Formula	Abundance
457.3654	(M+H)+	C30 H49 O3	59344.7

Best	Formula (M)	Ion Formula	Score	Cross Score	Calc m/z	Diff (ppm)	Mass Match	Abund Match	Spacing Match
✓	C30 H48 O3	C30 H49 O3	86.45		457.3676	4.97	78.06	92.06	96.48

Isotope	Abund Sum%	Calc Abund Sum%	m/z	Calc m/z	Diff (ppm)
1	73.47	71.53	457.3654	457.3676	4.94
2	21.53	23.69	458.3684	458.371	5.75
3	4.41	4.24	459.3697	459.3742	9.75
4	0.59	0.54	460.3634	460.3772	29.79



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

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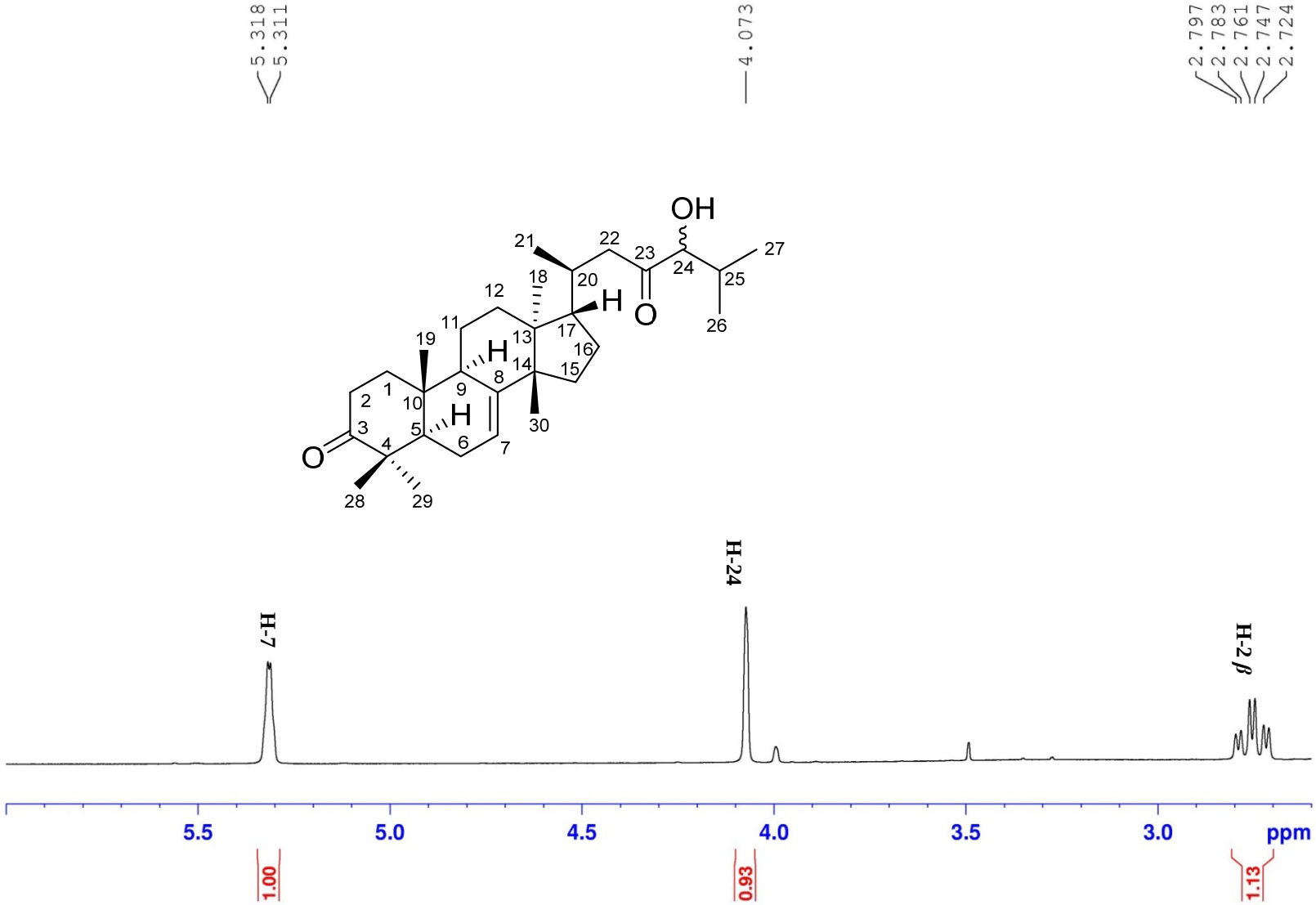
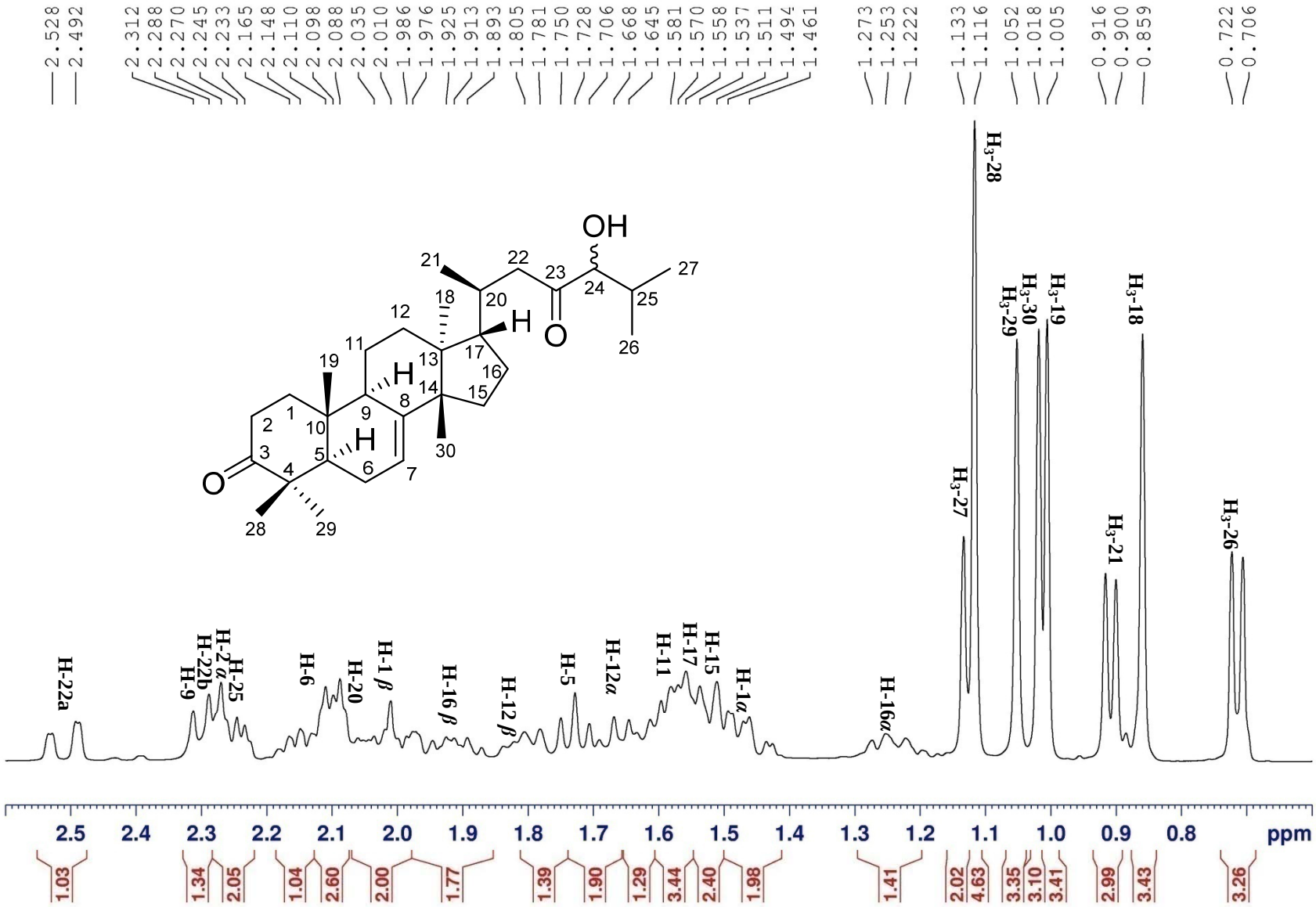
NAME                zq-9
EXPNO                  1
PROCNO                 1
Date_           20161202
Time                9.56
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                 208.5
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                 11.50 usec
SI                 65536
SF          400.1300083 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

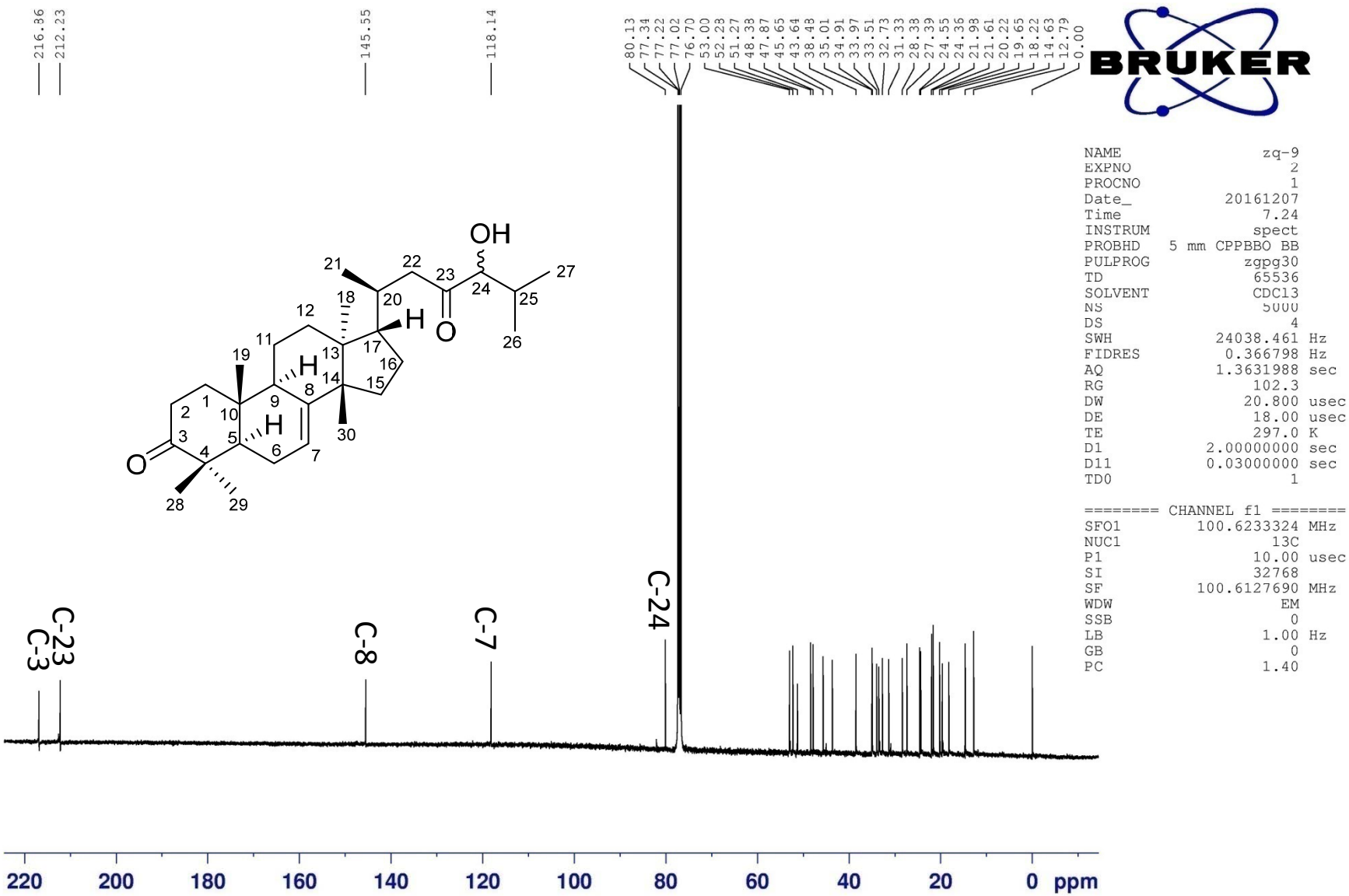
^{1}H NMR (400 MHz) spectrum of compound **3** in $CDCl_3$



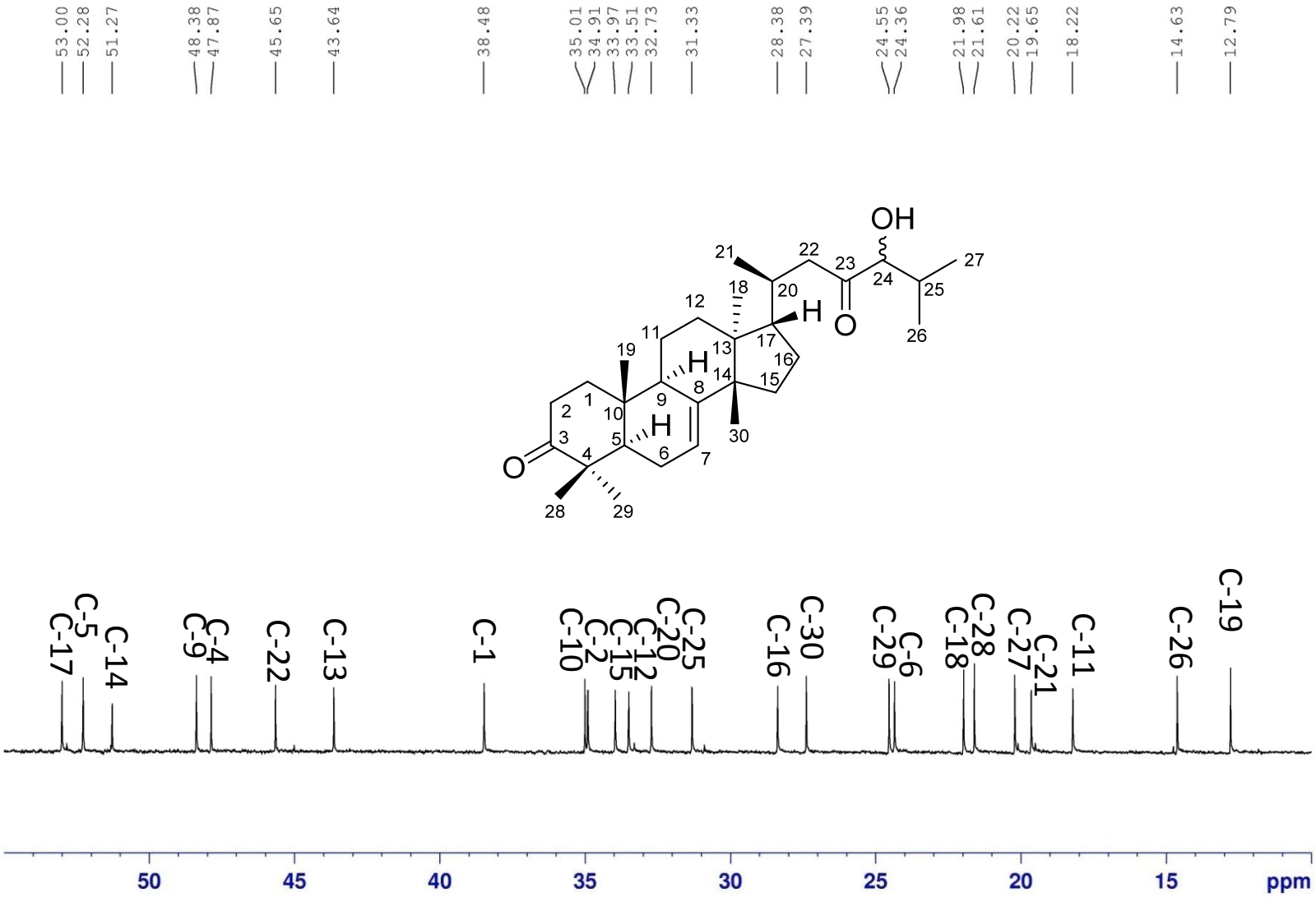
^{1}H NMR (400 MHz) spectrum of compound **3** in $CDCl_3$



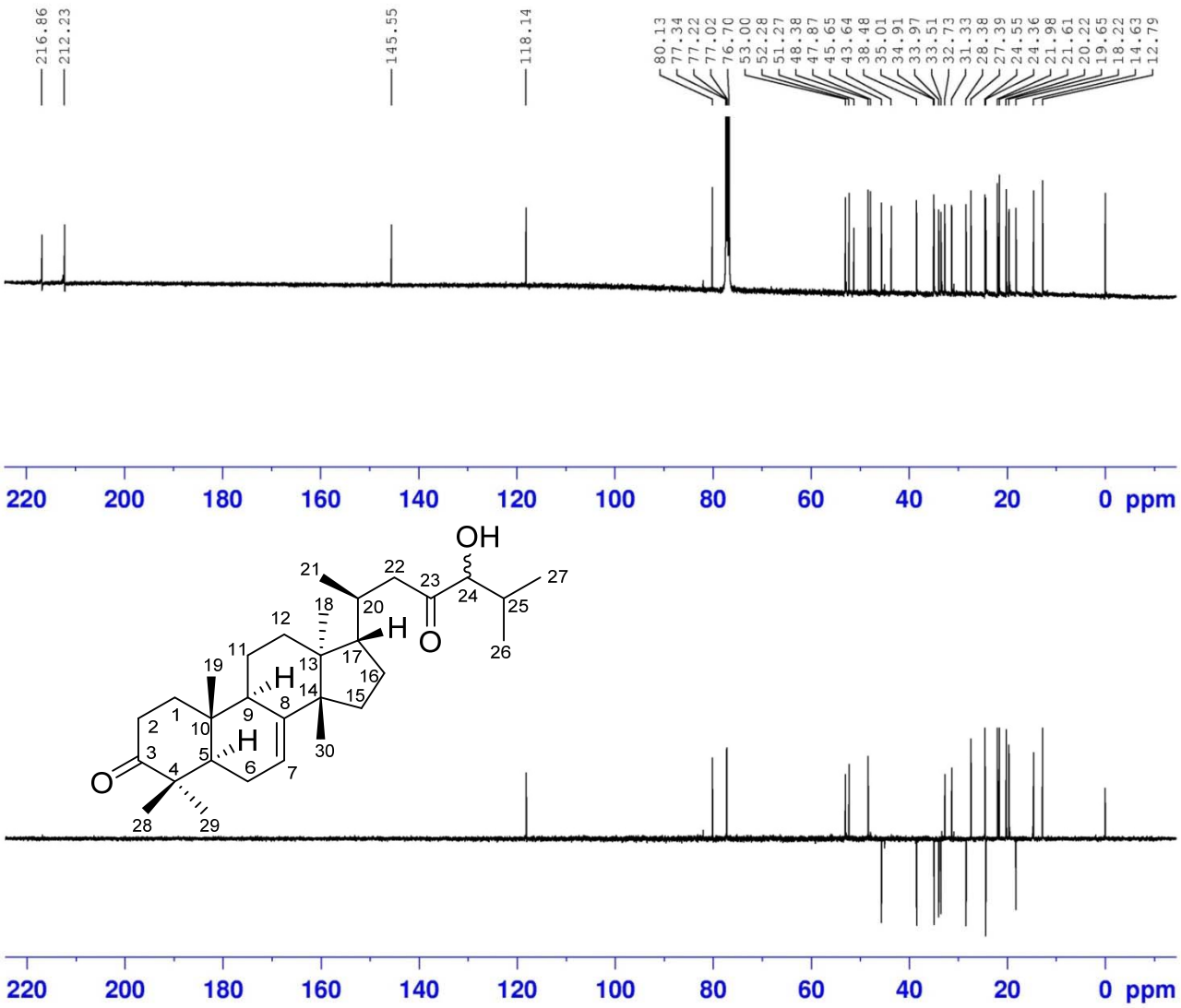
^{13}C NMR (100 MHz) spectrum of compound **3** in $CDCl_3$



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



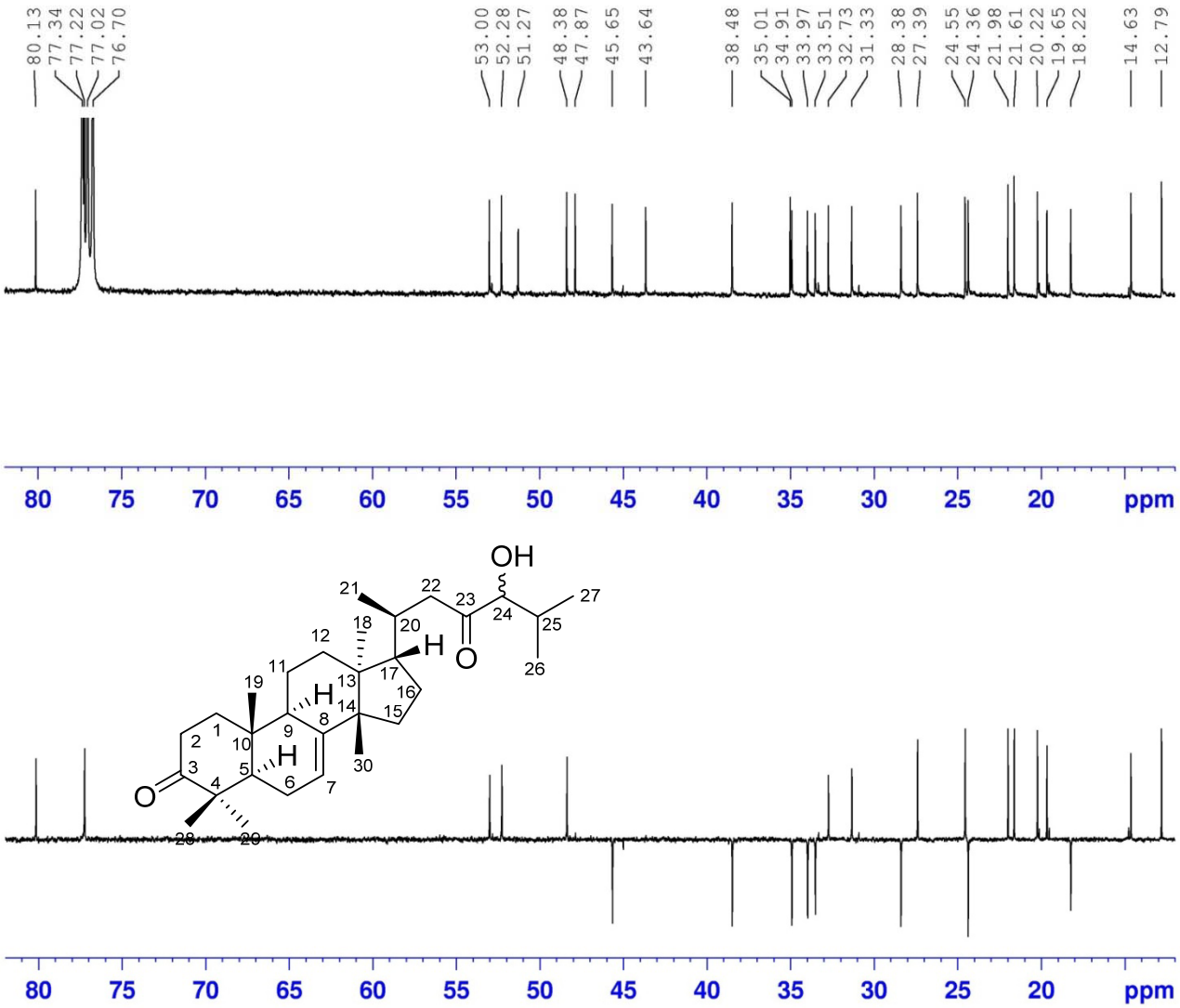
DEPT135 (100 MHz) spectrum of compound **3** in $CDCl_3$




```
NAME                zq-9
EXPNO                  3
PROCNO                 1
Date_           20161207
Time                8.51
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG       deptsp135
TD                65536
SOLVENT           CDCl3
NS                 1500
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 ========
SFO1         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
```

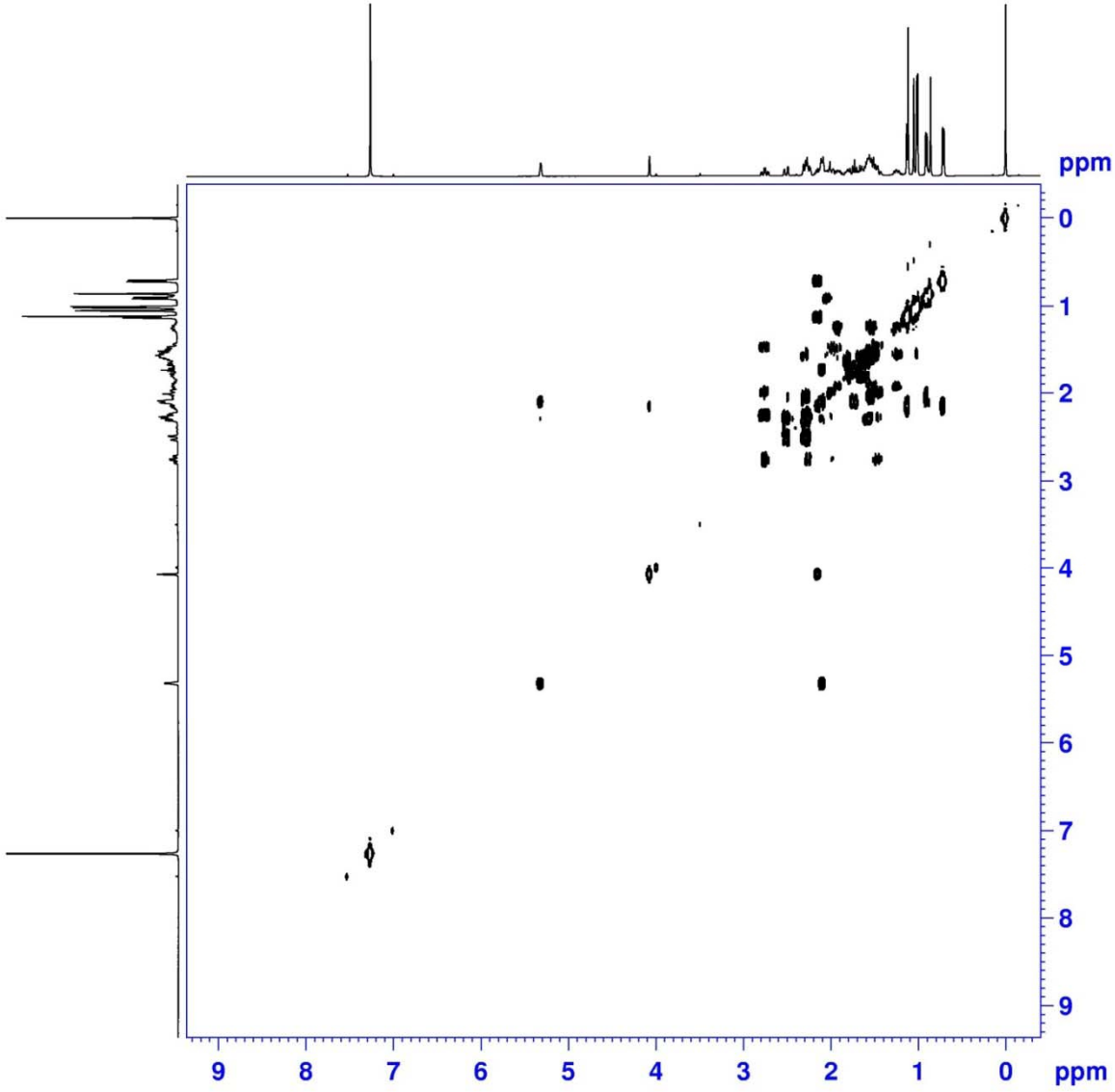
DEPT135 (100 MHz) spectrum of compound **3** in $CDCl_3$




```
NAME                zq-9
EXPNO                  3
PROCNO                 1
Date_           20161207
Time                8.51
INSTRUM            spect
PROBHD    5 mm CPPBBO BB
PULPROG        deptsp135
TD                 65536
SOLVENT            CDCl3
NS                  1500
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 ========
SFO1         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
```

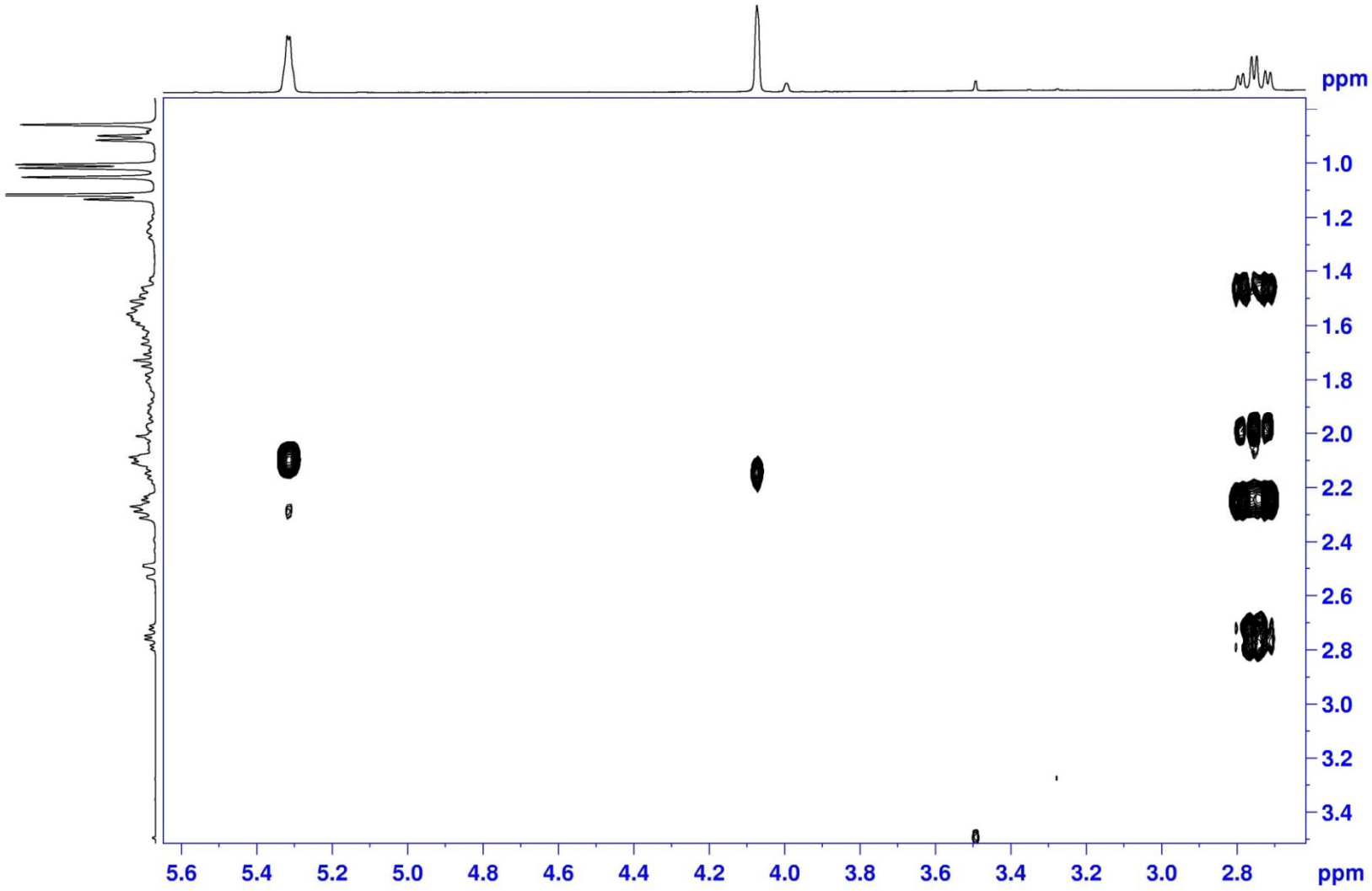
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **3** in $CDCl_3$



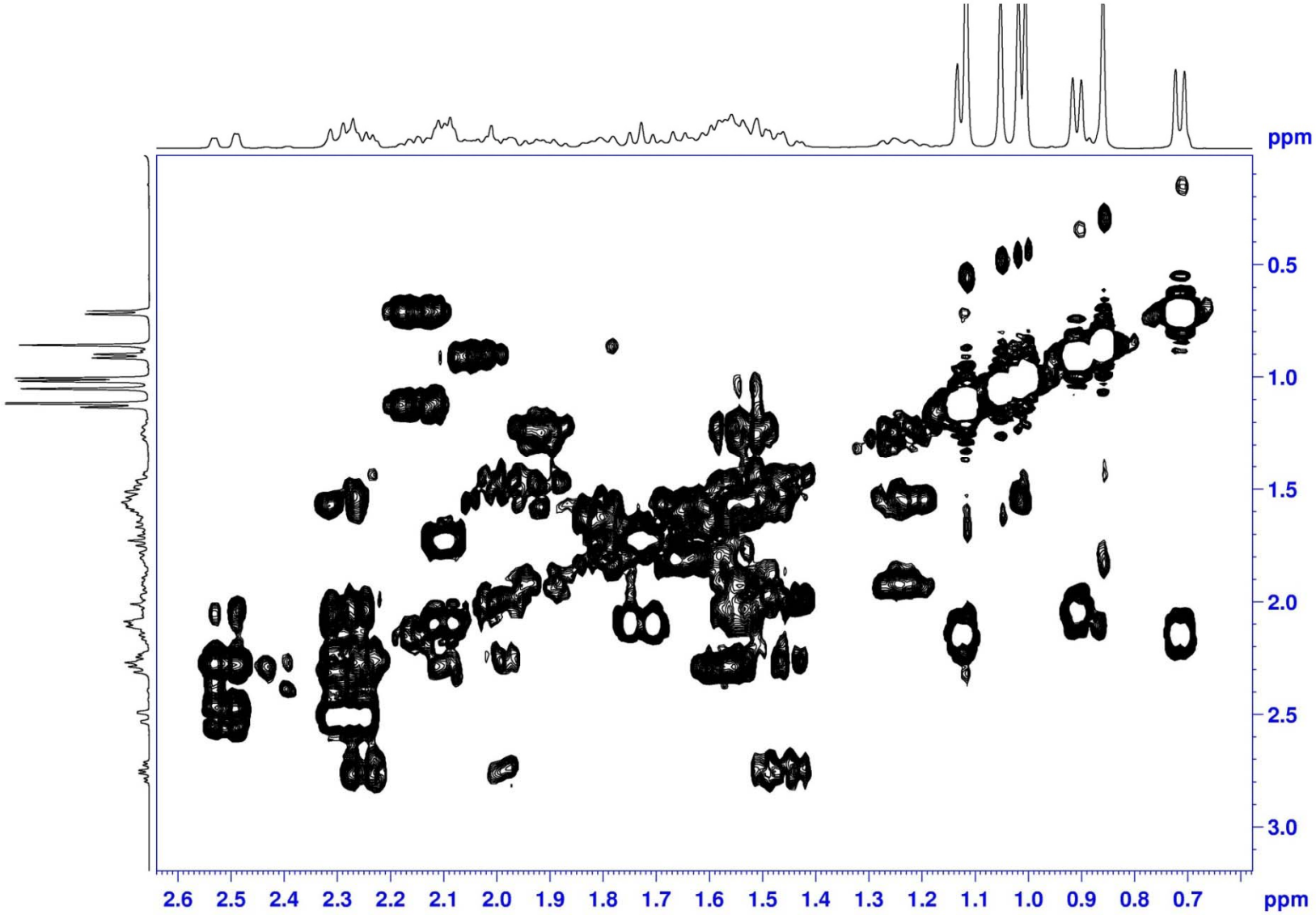

```
NAME              ZQ-9-1
EXPNO                  4
PROCNO                 1
Date_           20161208
Time               15.26
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG      cosygpppqf
TD                  2048
SOLVENT            CDCl3
NS                    32
DS                     8
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                 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          400.1300064 MHz
WDW               QSINE
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2                  QF
SF          400.1300061 MHz
WDW               QSINE
SSB                   0
LB                 0.00 Hz
```

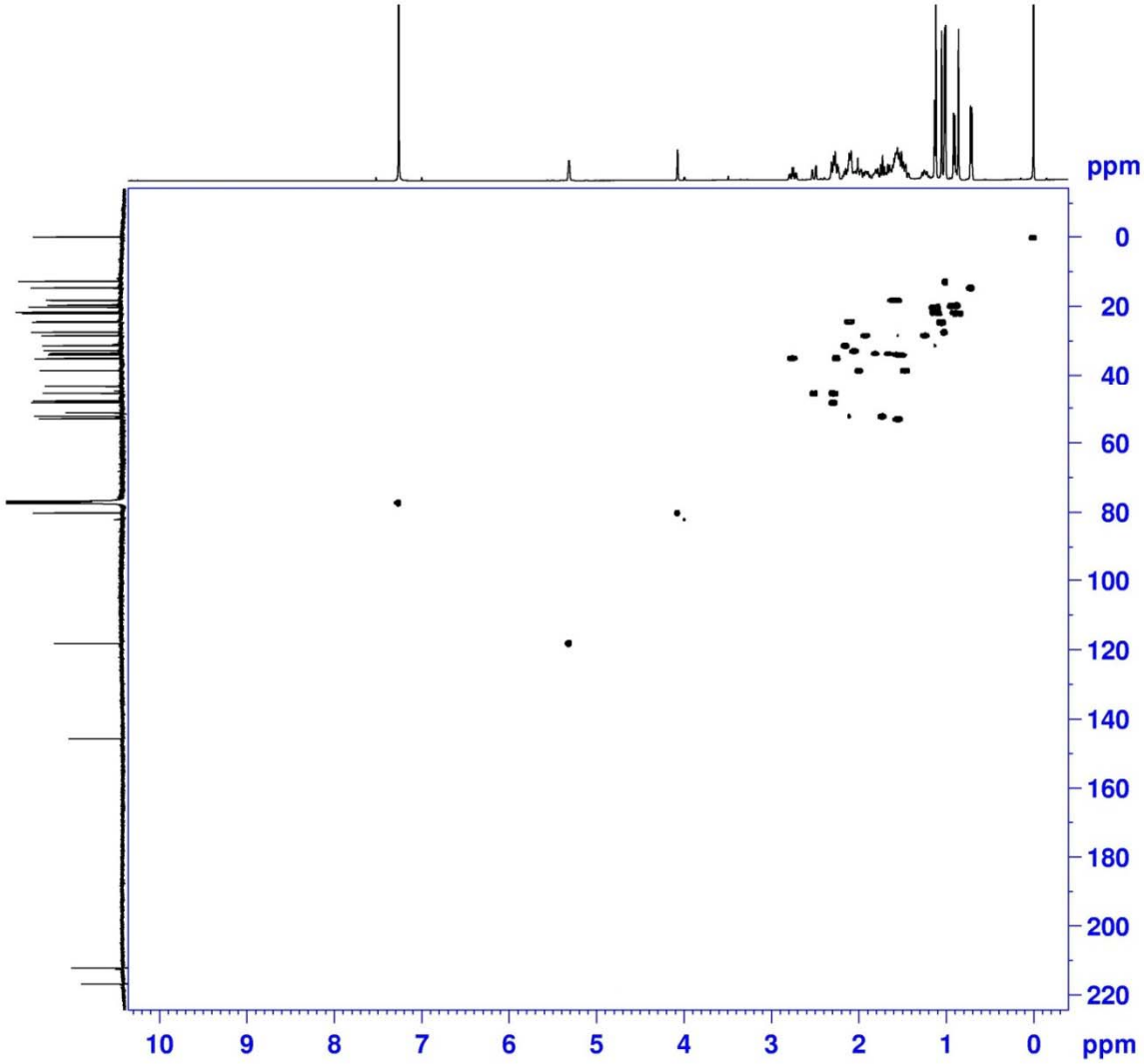
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **3** in $CDCl_3$



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



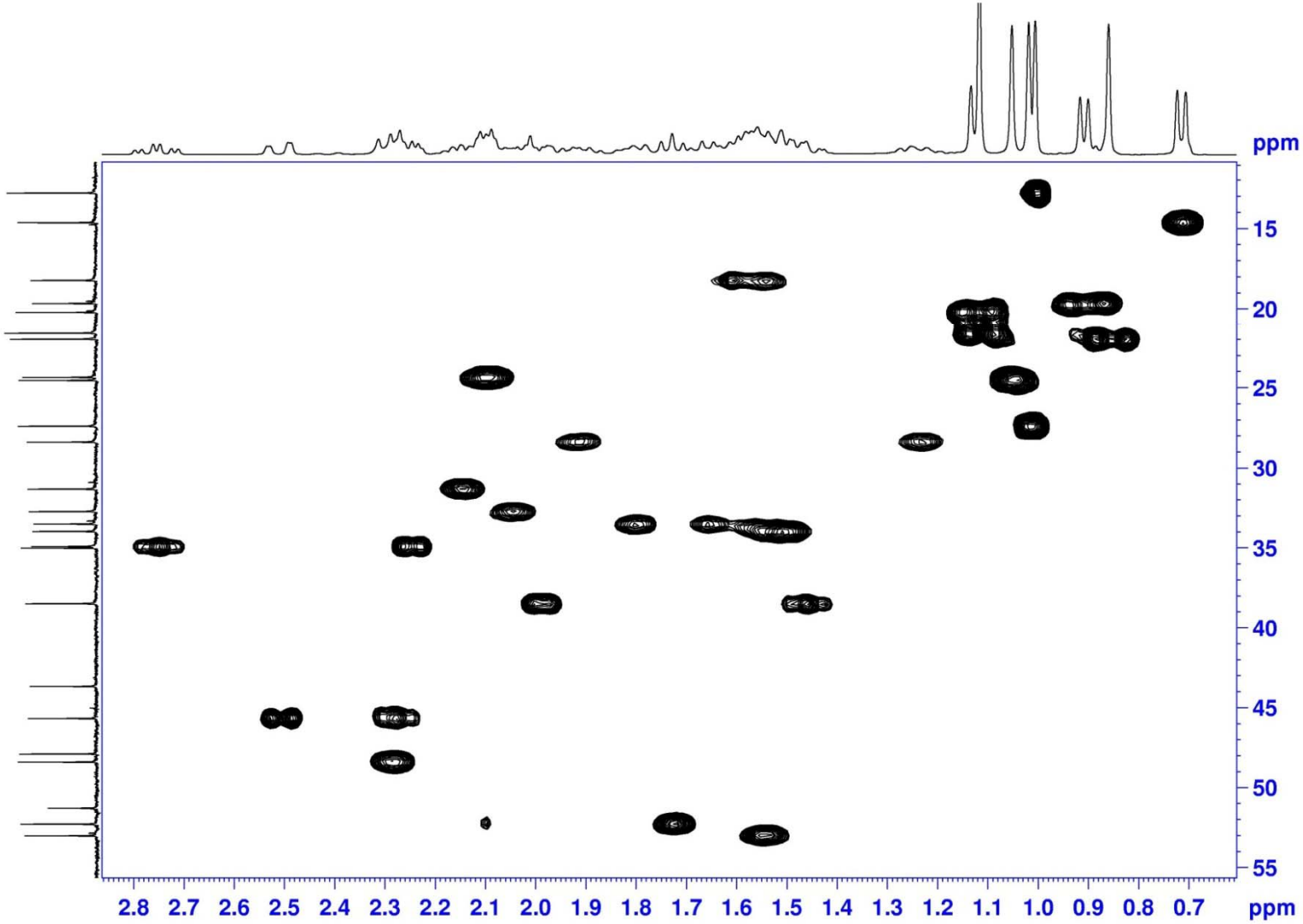
HSQC (400 MHz) spectrum of compound **3** in $CDCl_3$



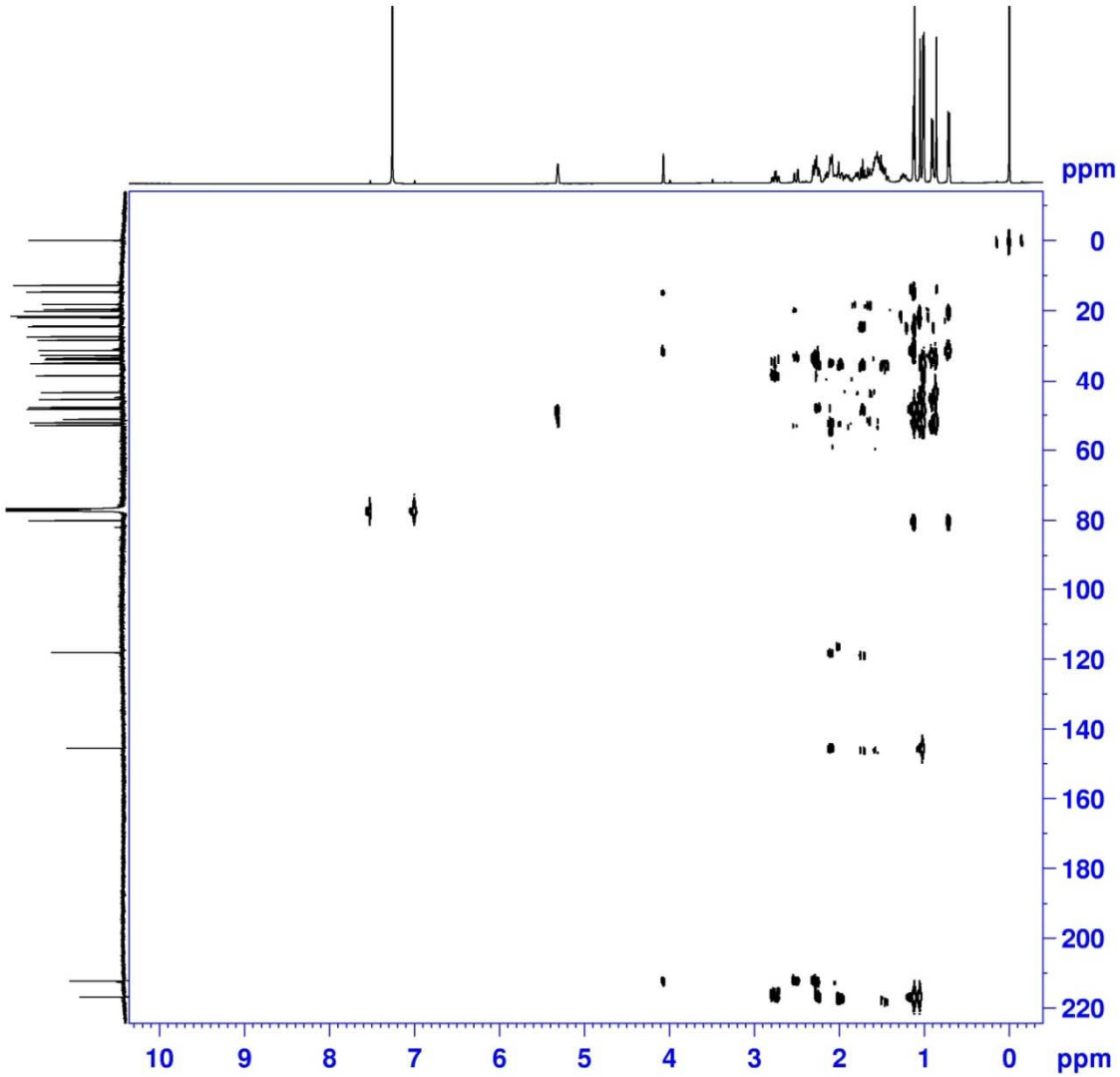

```
NAME                ZQ-9-1
EXPNO                  105
PROCNO                   1
Date_             20161208
Time                 17.58
INSTRUM              spect
PROBHD    5 mm CPPBBO BB
PULPROG      hsqcetgpsi2
TD                    1024
SOLVENT              CDCl3
NS                      32
DS                      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
ZGOPTNS

======== CHANNEL f1 ========
SFO1          400.1320007 MHz
NUC1                   1H
P1                   11.50 usec
P2                   23.00 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            400.1300083 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
PC                    1.40
SI                    1024
MC2        echo-antiecho
SF            100.6127571 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
```

HSQC (400 MHz) spectrum of compound **3** in $CDCl_3$



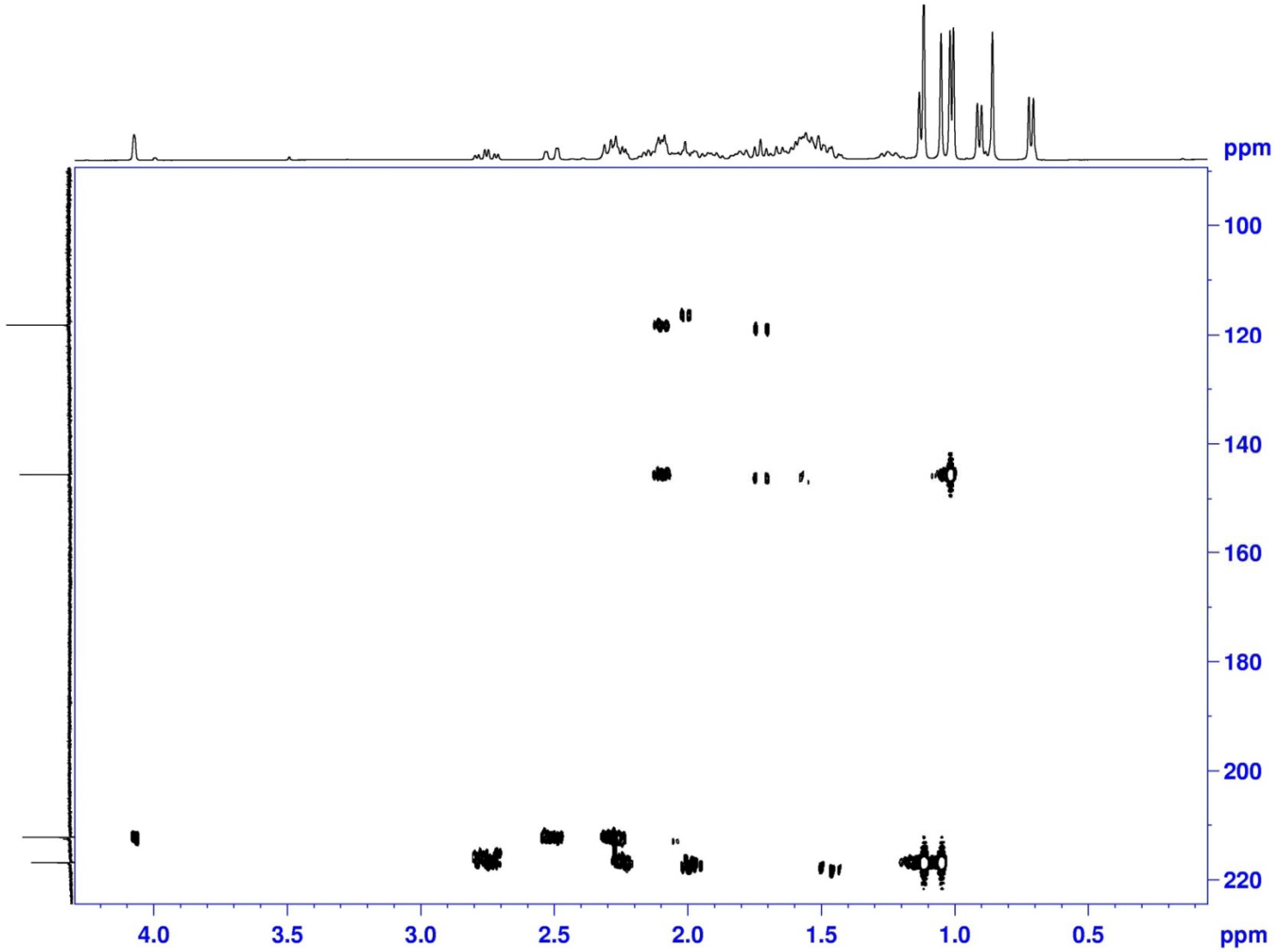
HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



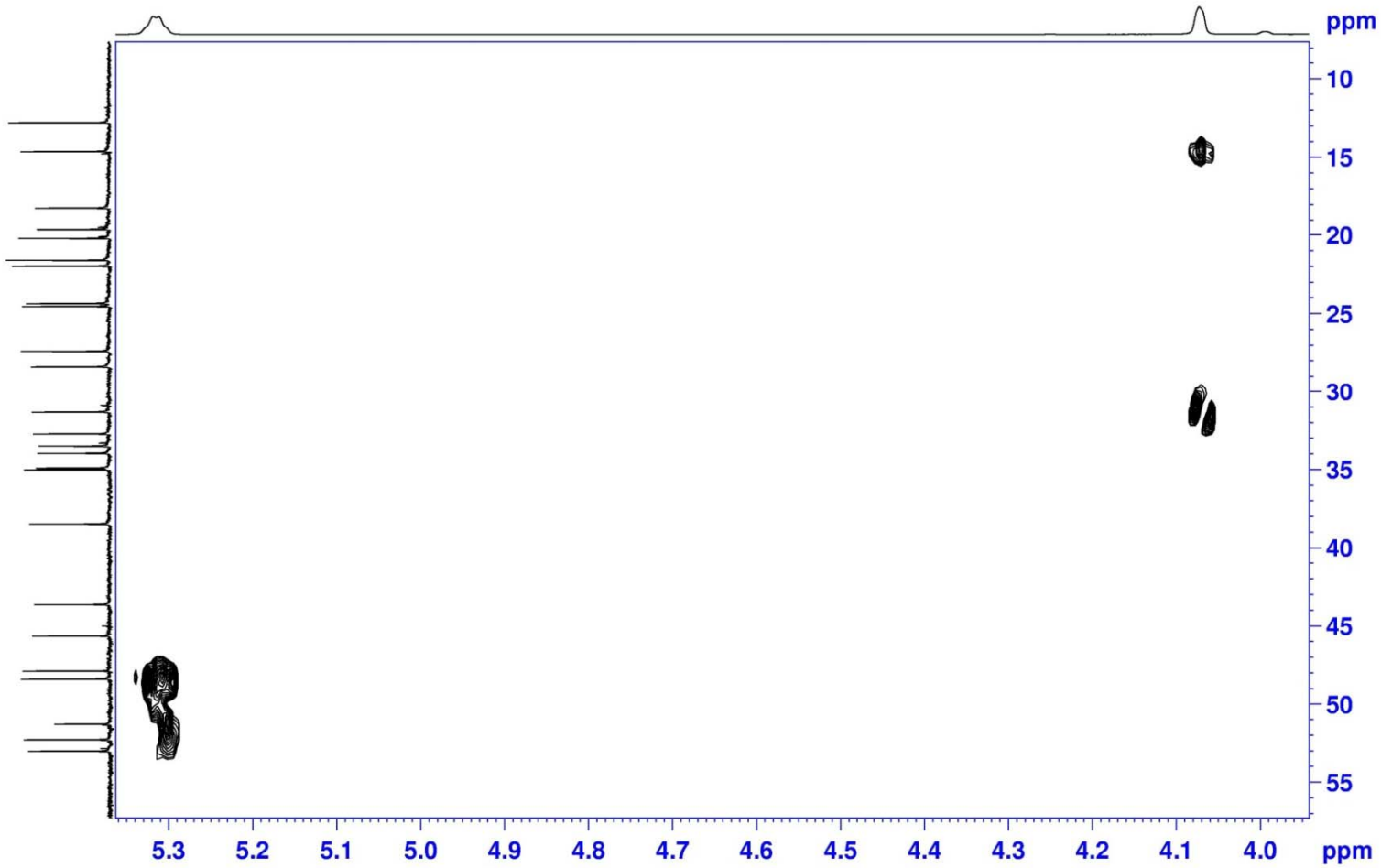

```
NAME              ZQ-9-1
EXPNO                106
PROCNO                 1
Date_           20161209
Time                4.26
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG    hmbcgplpndqf
TD                  4096
SOLVENT            CDCl3
NS                   128
DS                    16
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                   125
SFO1           100.6233 MHz
FIDRES        192.307693 Hz
SW               238.896 ppm
FnMODE                QF
SI                  2048
SF           400.1300083 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           100.6127571 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
```

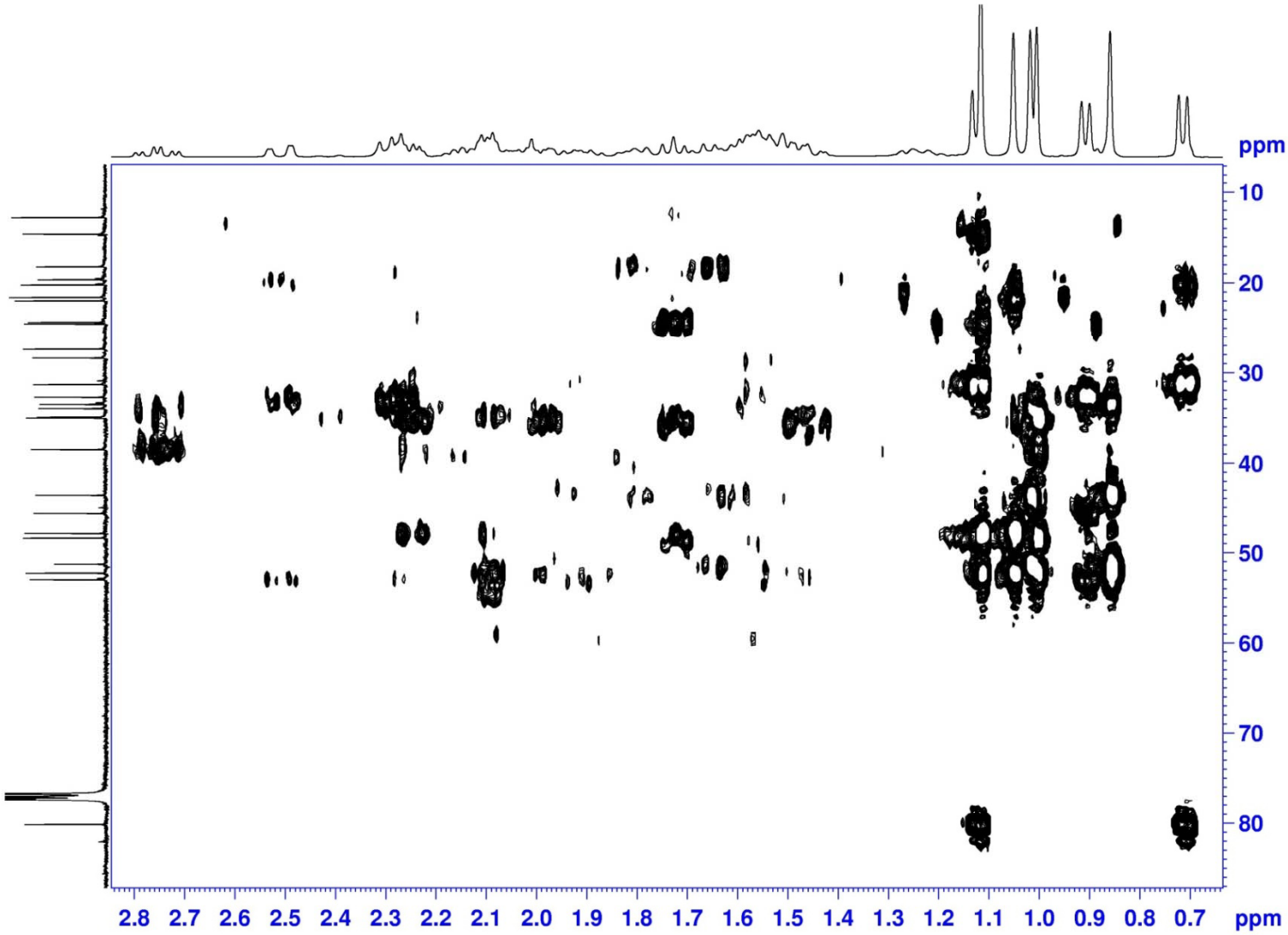
HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



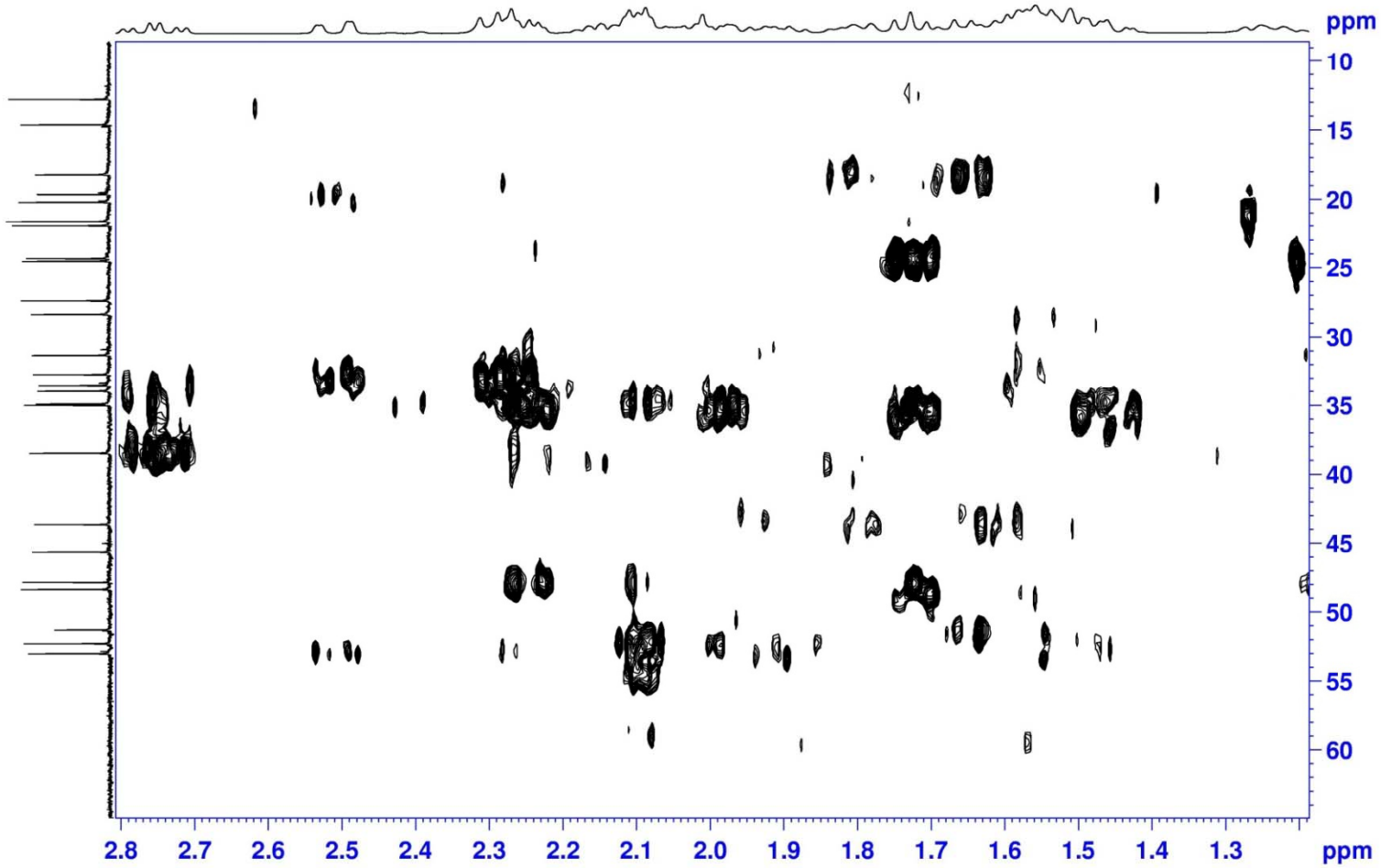
HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



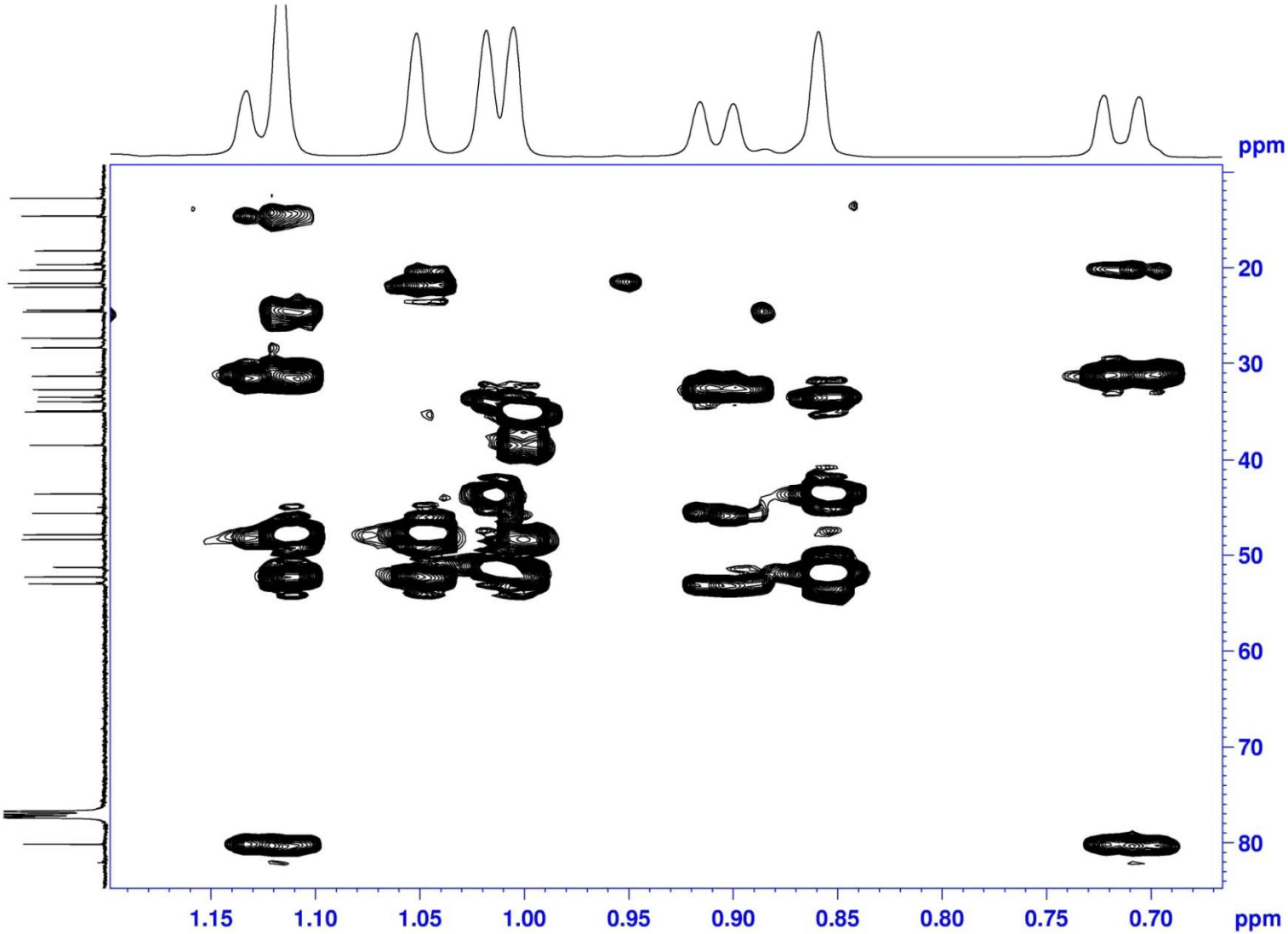
HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



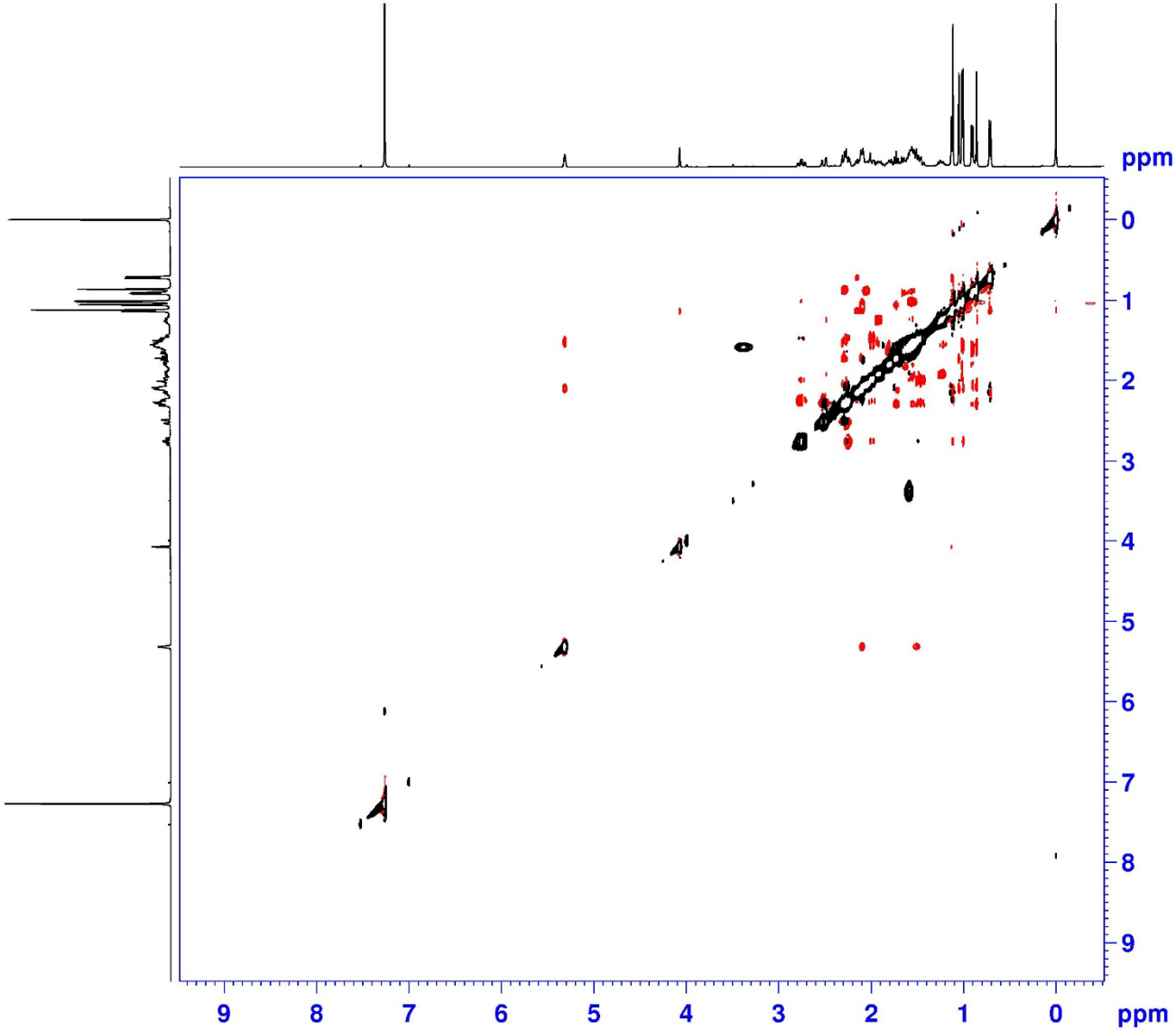
HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **3** in $CDCl_3$



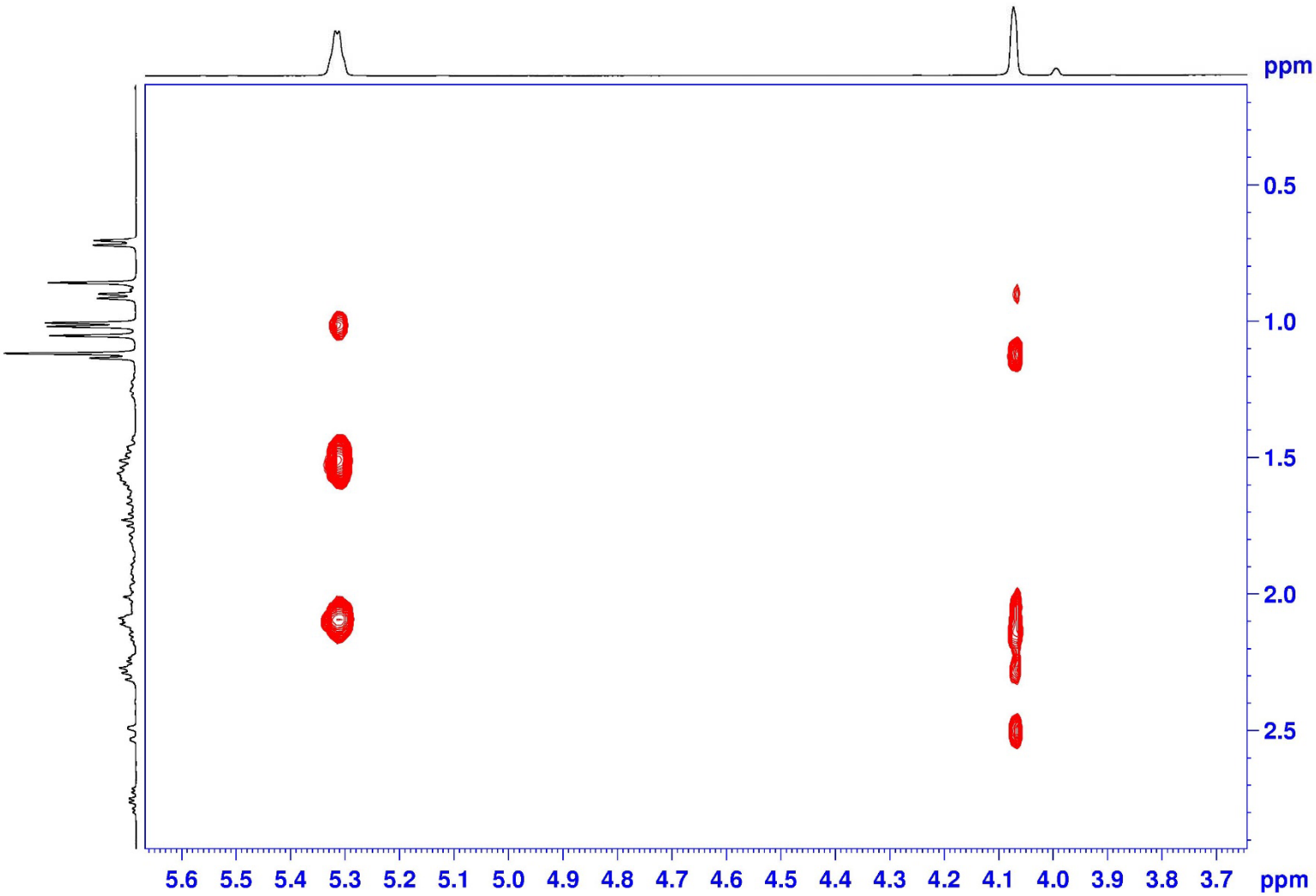
NOESY (400 MHz) spectrum of compound **3** in $CDCl_3$



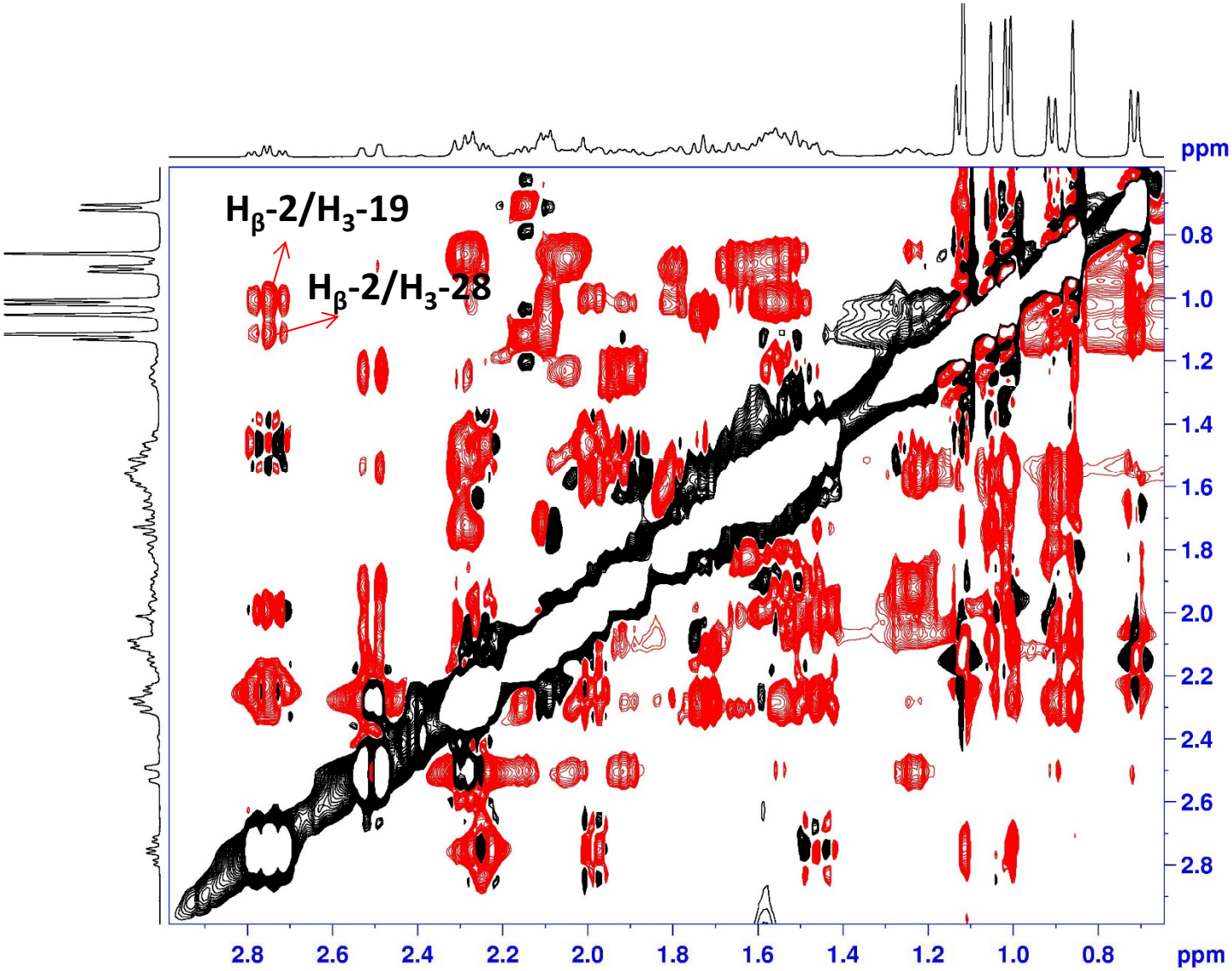

```
NAME                zq-9
EXPNO                  7
PROCNO                 1
Date_           20161208
Time               22.26
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG     noesygpphpp
TD                  2048
SOLVENT             CDC13
NS                     32
DS                     32
SWH             4000.000 Hz
FIDRES          1.953125 Hz
AQ             0.2560500 sec
RG                 208.5
DW               125.000 use
DE                 10.00 use
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 use
P2                 23.00 use
P17              2500.00 use
ND0                    1
TD                   256
SFO1            400.1318 MHz
FIDRES          15.625000 Hz
SW                  9.997 ppm
FnMODE       States-TPPI
SI                  1024
SF           400.1300083 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00
SI                  1024
MC2          States-TPPI
SF           400.1300083 MHz
WDW                 QSINE
```

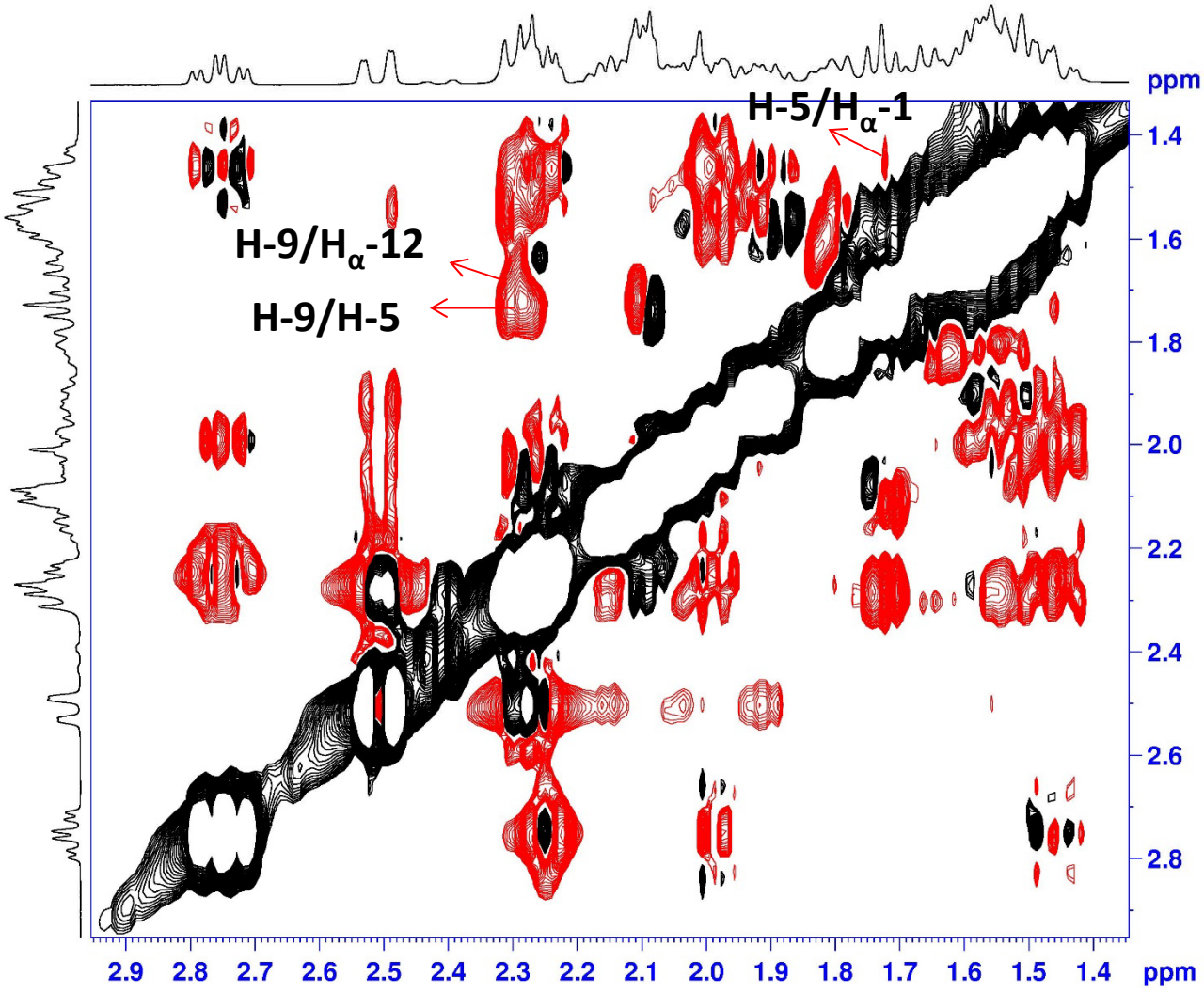
NOESY (400 MHz) spectrum of compound **3** in $CDCl_3$



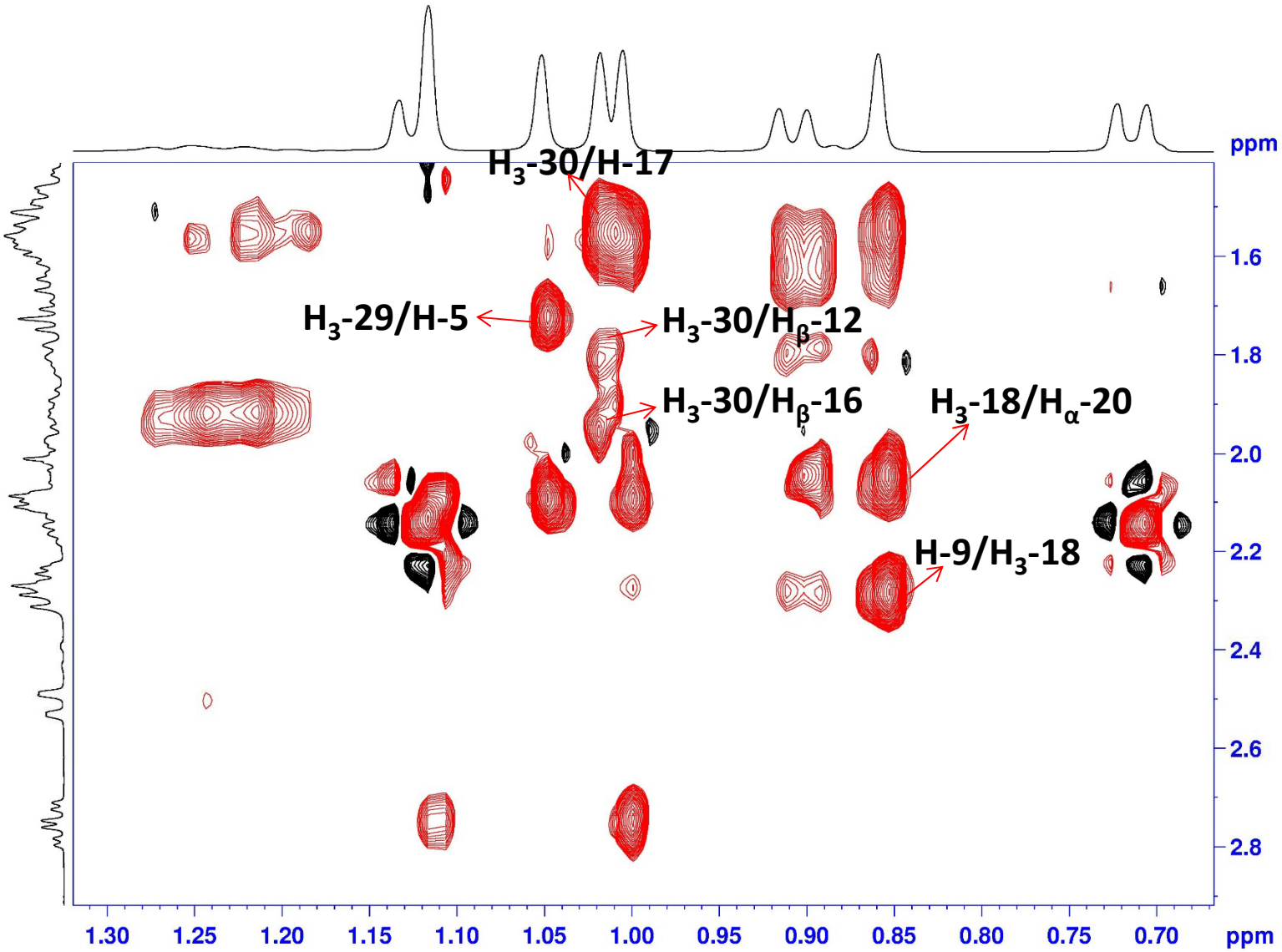
NOESY (400 MHz) spectrum of compound **3** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **3** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **3** in $CDCl_3$

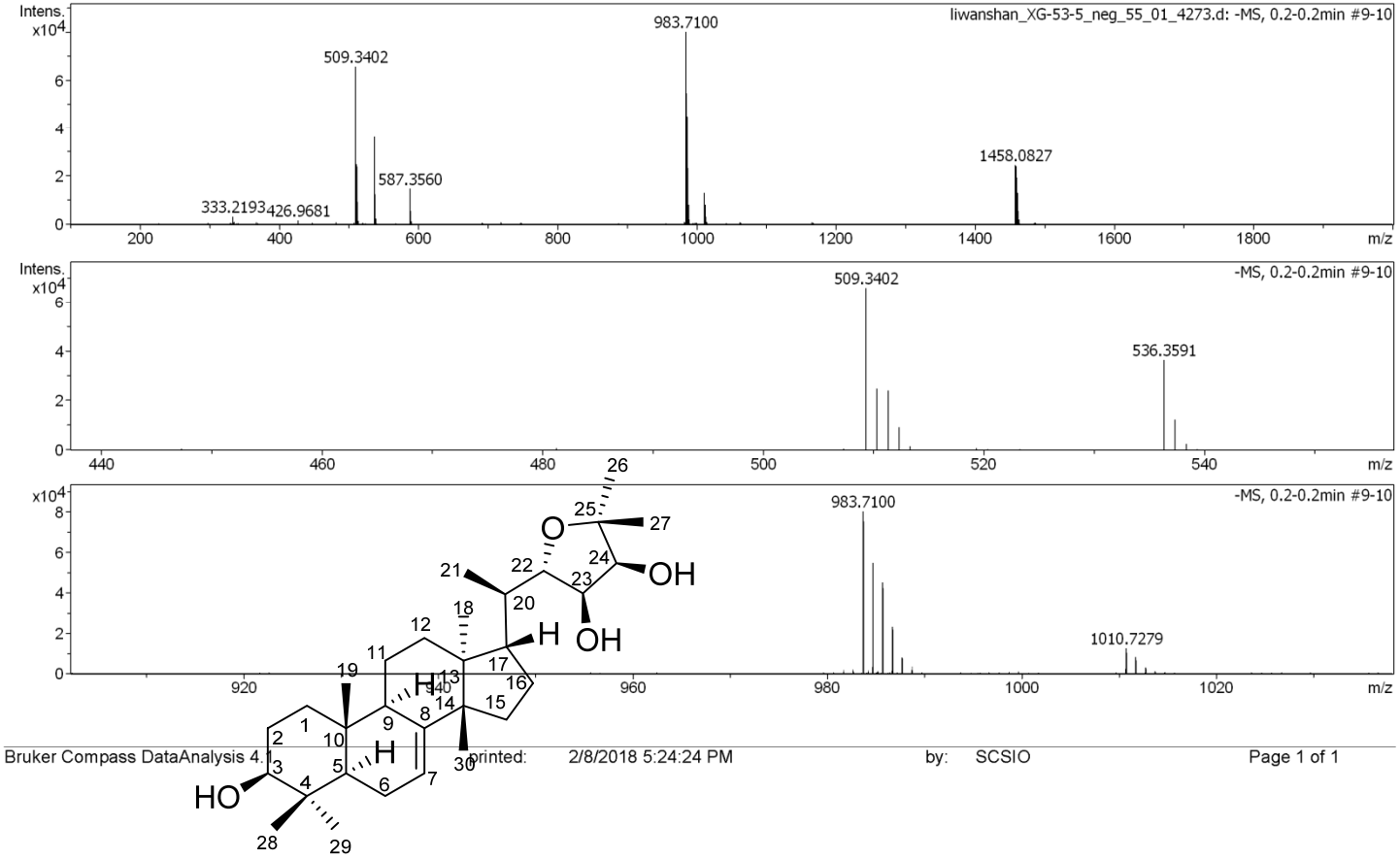


HR-ESIMS for compound **4**

Generic Display Report

Analysis Info

Analysis Name	D:\Data\MS\data\201802\liwanshan_XG-53-5_neg_55_01_4273.d	Acquisition Date	2/8/2018 5:09:54 PM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_XG-53-5_neg	Instrument	maXis
Comment			



Bruker Compass DataAnalysis 4.1 printed: 2/8/2018 5:24:24 PM by: SCSIO Page 1 of 1

HR-ESIMS for compound **4**

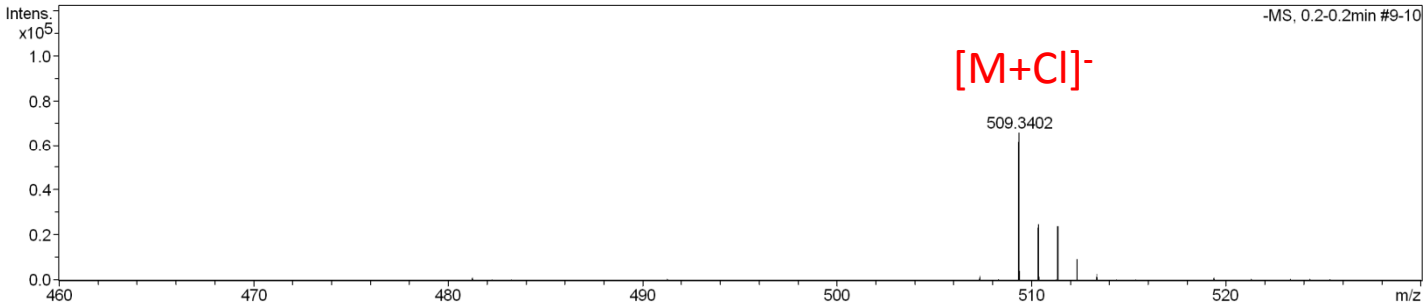
Mass Spectrum SmartFormula Report

Analysis Info

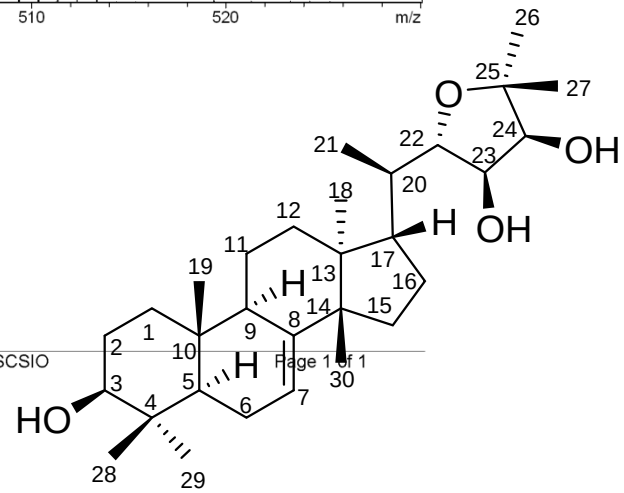
Analysis Name	D:\Data\MS\data\201802\liwanshan_XG-53-5_neg_55_01_4273.d	Acquisition Date	2/8/2018 5:09:54 PM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_XG-53-5_neg	Instrument	maXis 255552.00029
Comment			

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



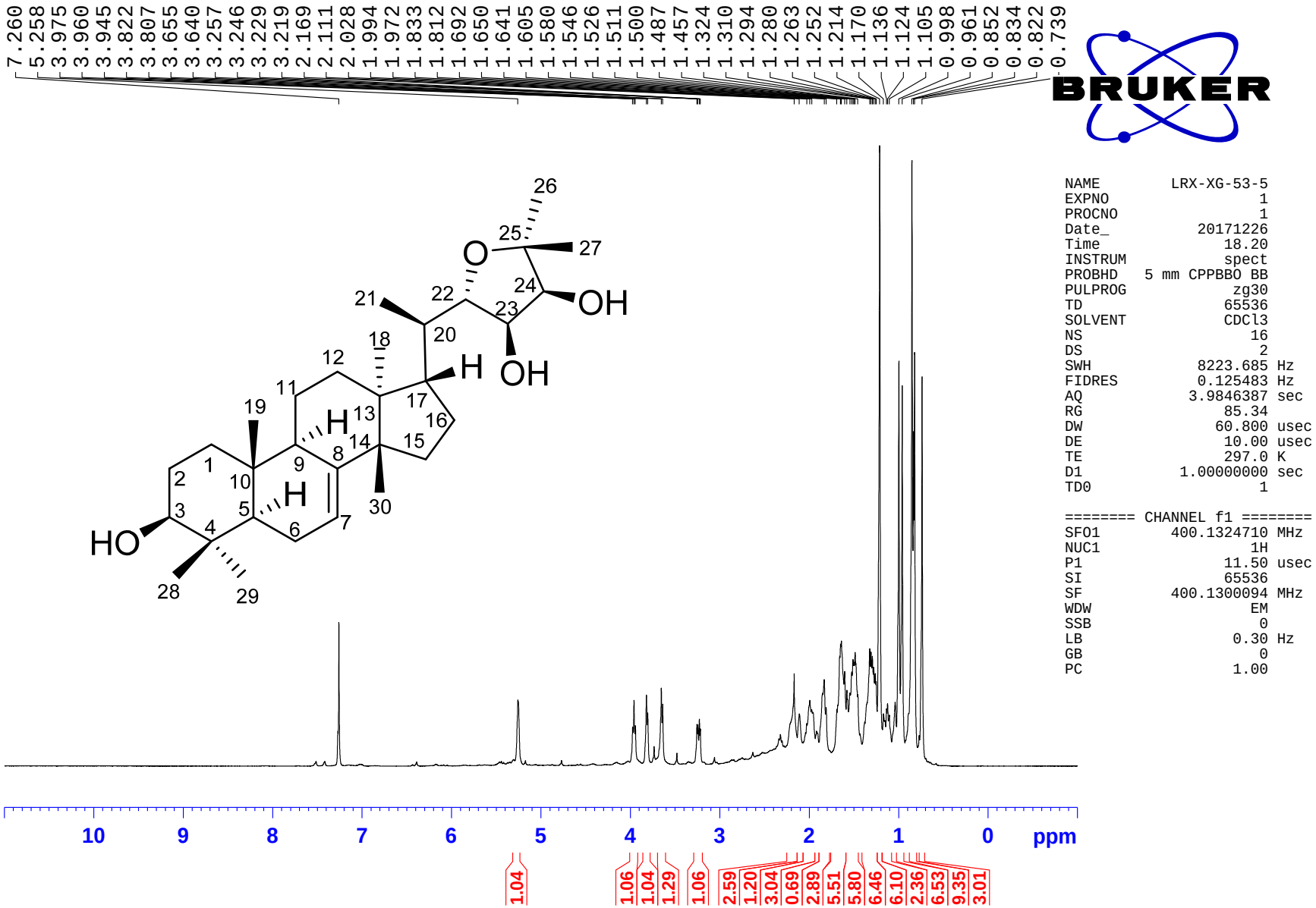
Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
509.3402	1	C30H50ClO4	100.00	509.3403	-0.2	-0.1	25.2	5.5	even	ok
983.7100	1	C60H100ClO8	72.88	983.7112	-1.3	-1.2	12.6	10.5	even	ok



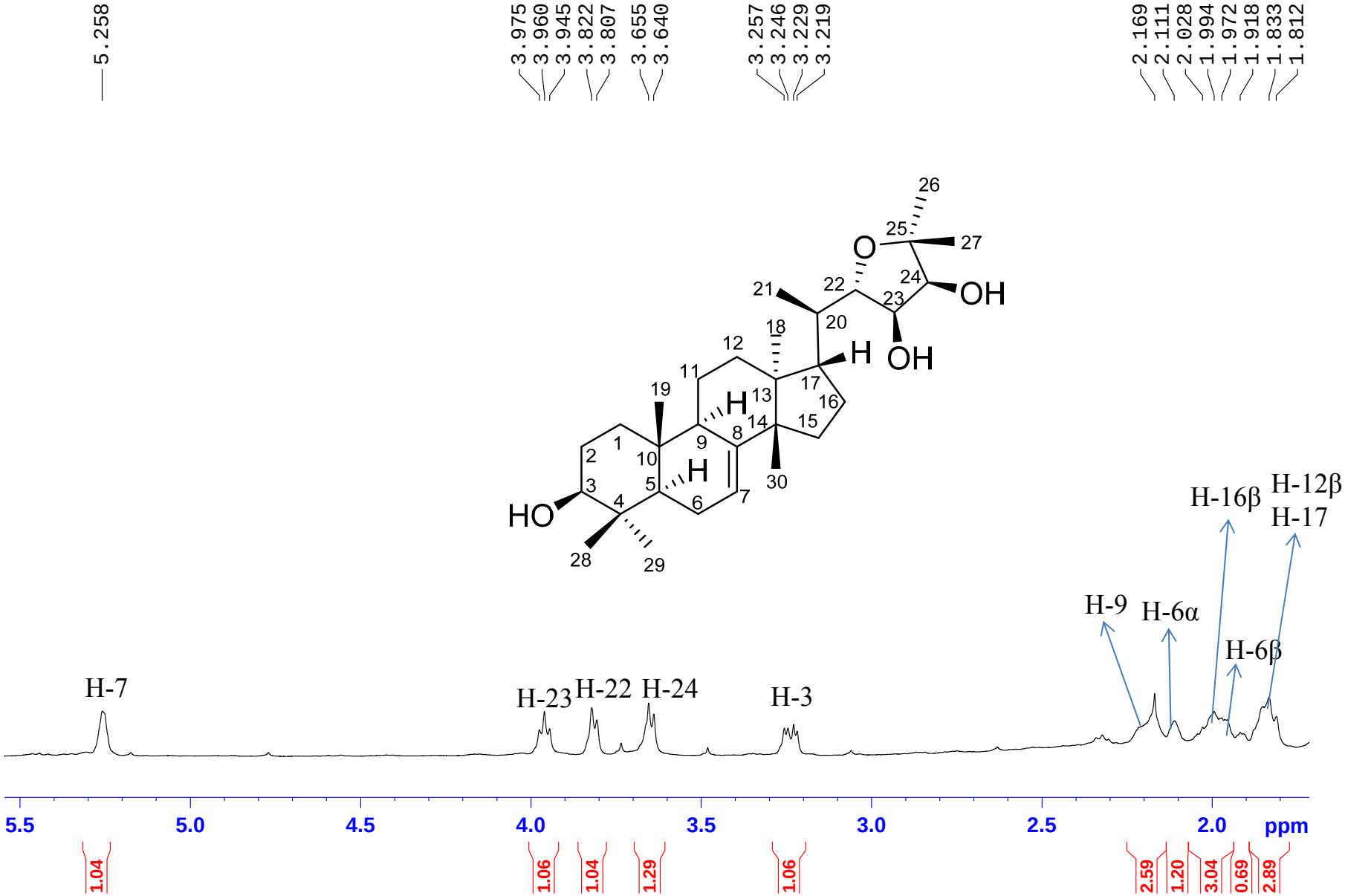
liwanshan_XG-53-5_neg_55_01_4273.d

Bruker Compass DataAnalysis 4.1 printed: 2/8/2018 5:23:39 PM by: SCSIO Page 1 of 1

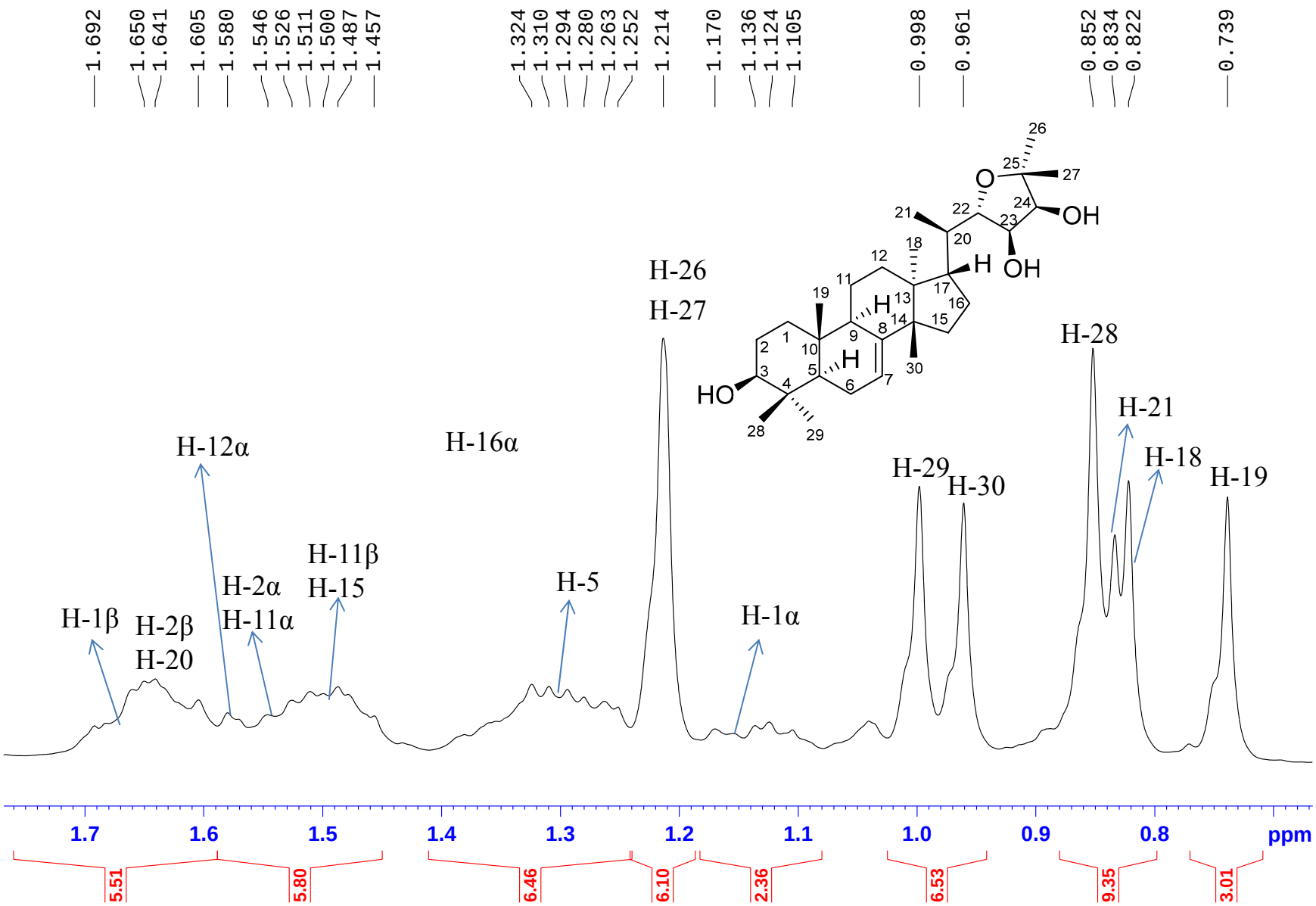
^{1}H NMR (400 MHz) spectrum of compound **4** in $CDCl_3$



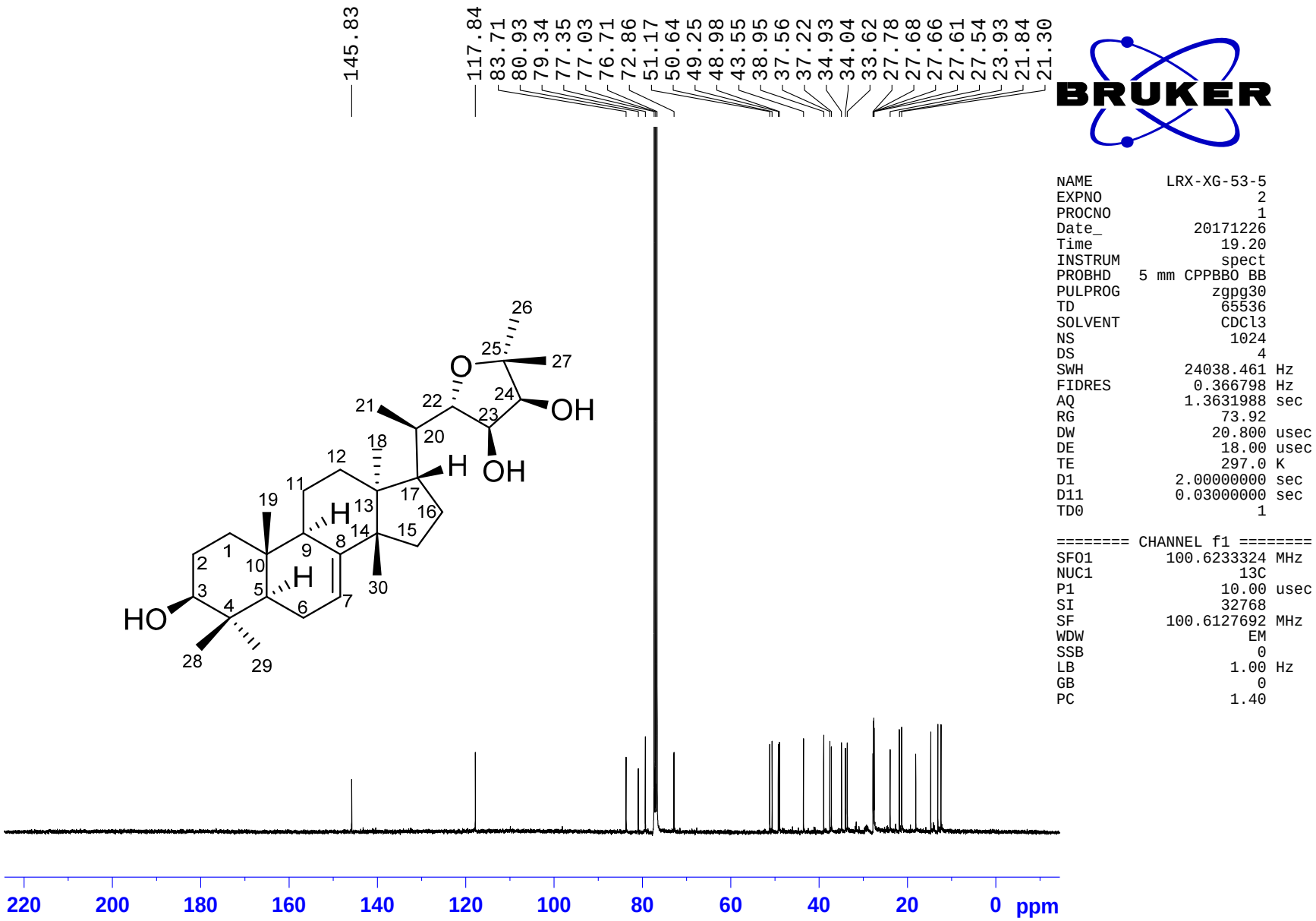
^{1}H NMR (400 MHz) spectrum of compound **4** in $CDCl_3$



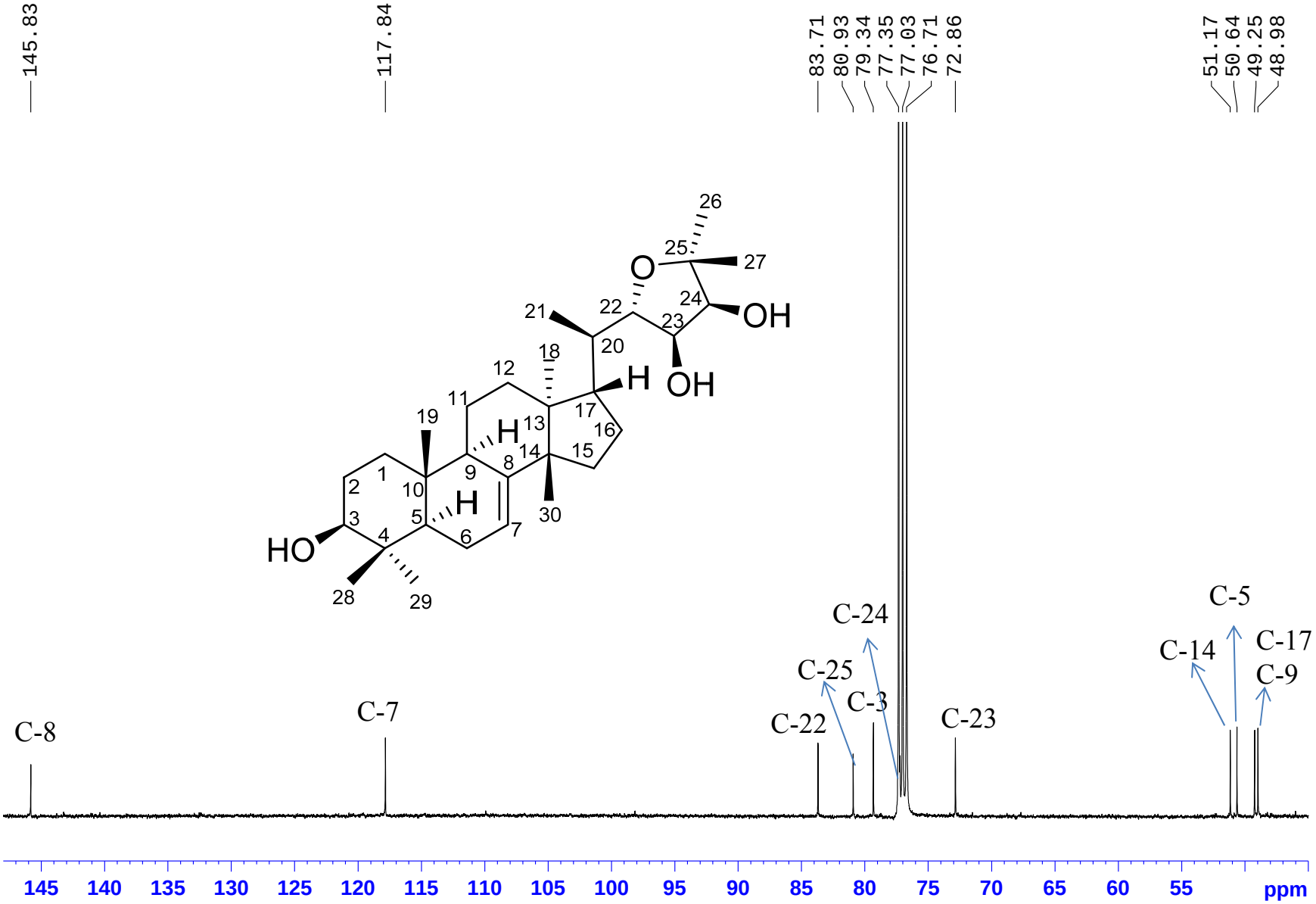
^{1}H NMR (400 MHz) spectrum of compound **4** in $CDCl_3$



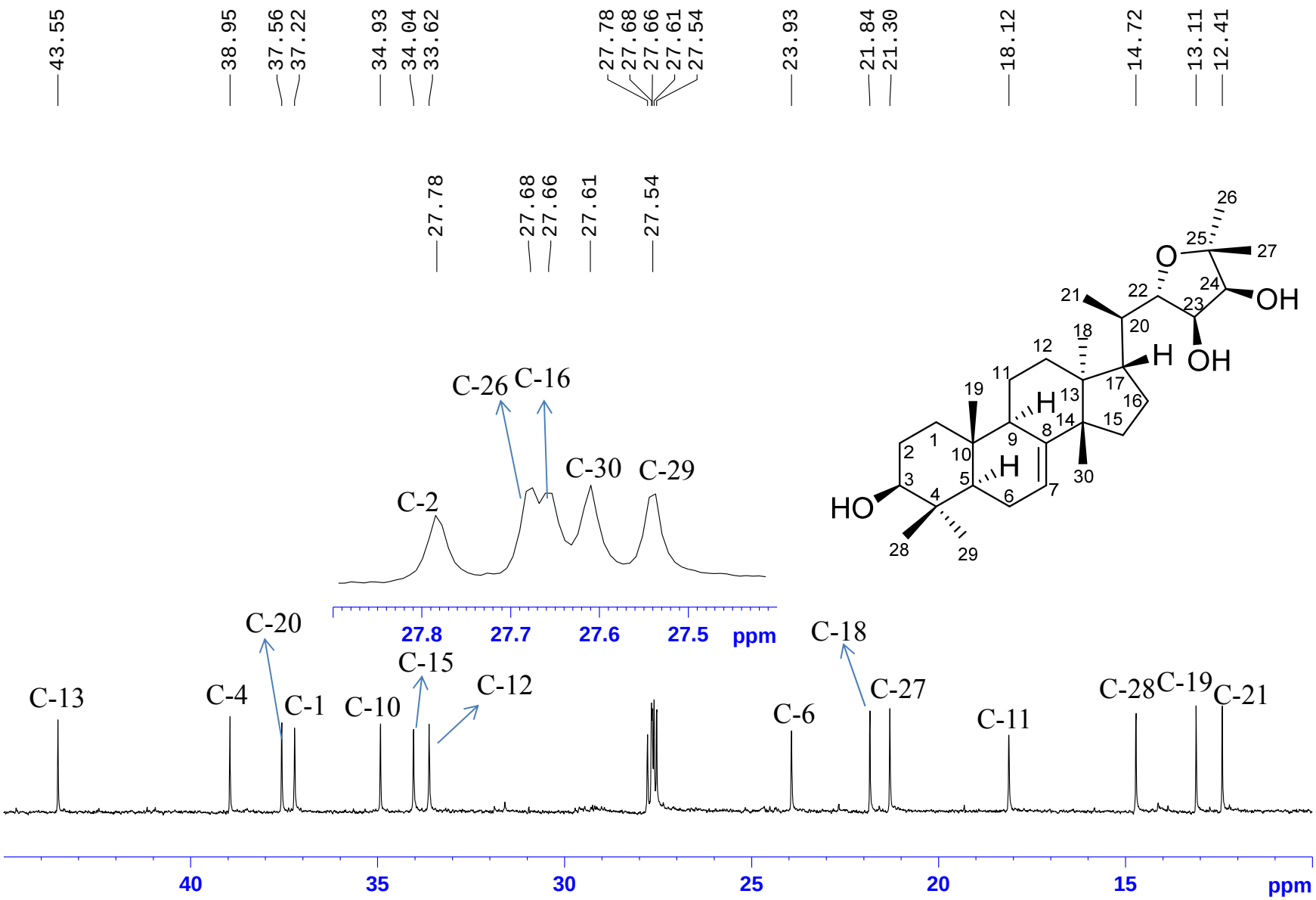
^{13}C NMR (100 MHz) spectrum of compound **4** in $CDCl_3$



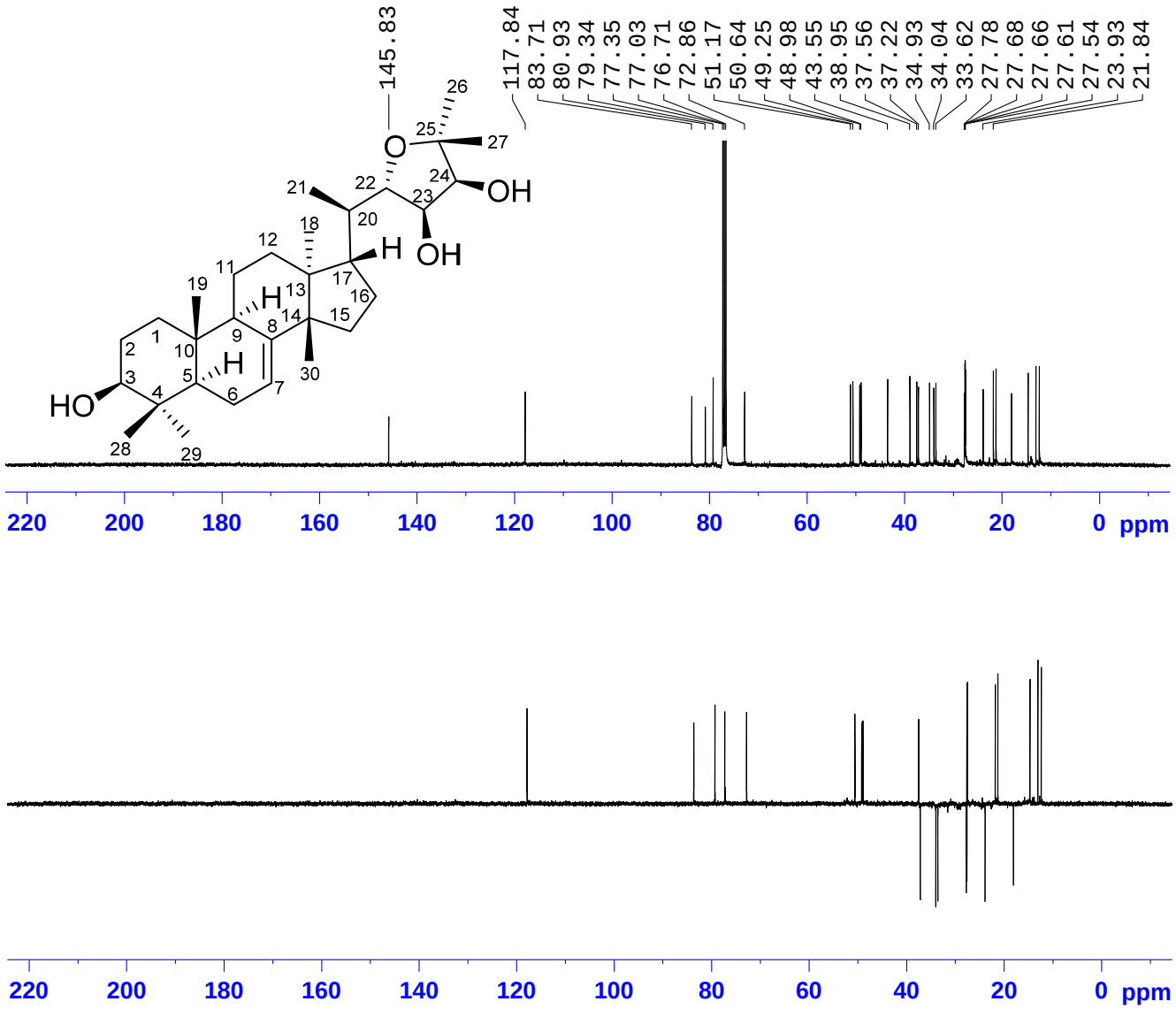
^{13}C NMR (100 MHz) spectrum of compound **4** in $CDCl_3$



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



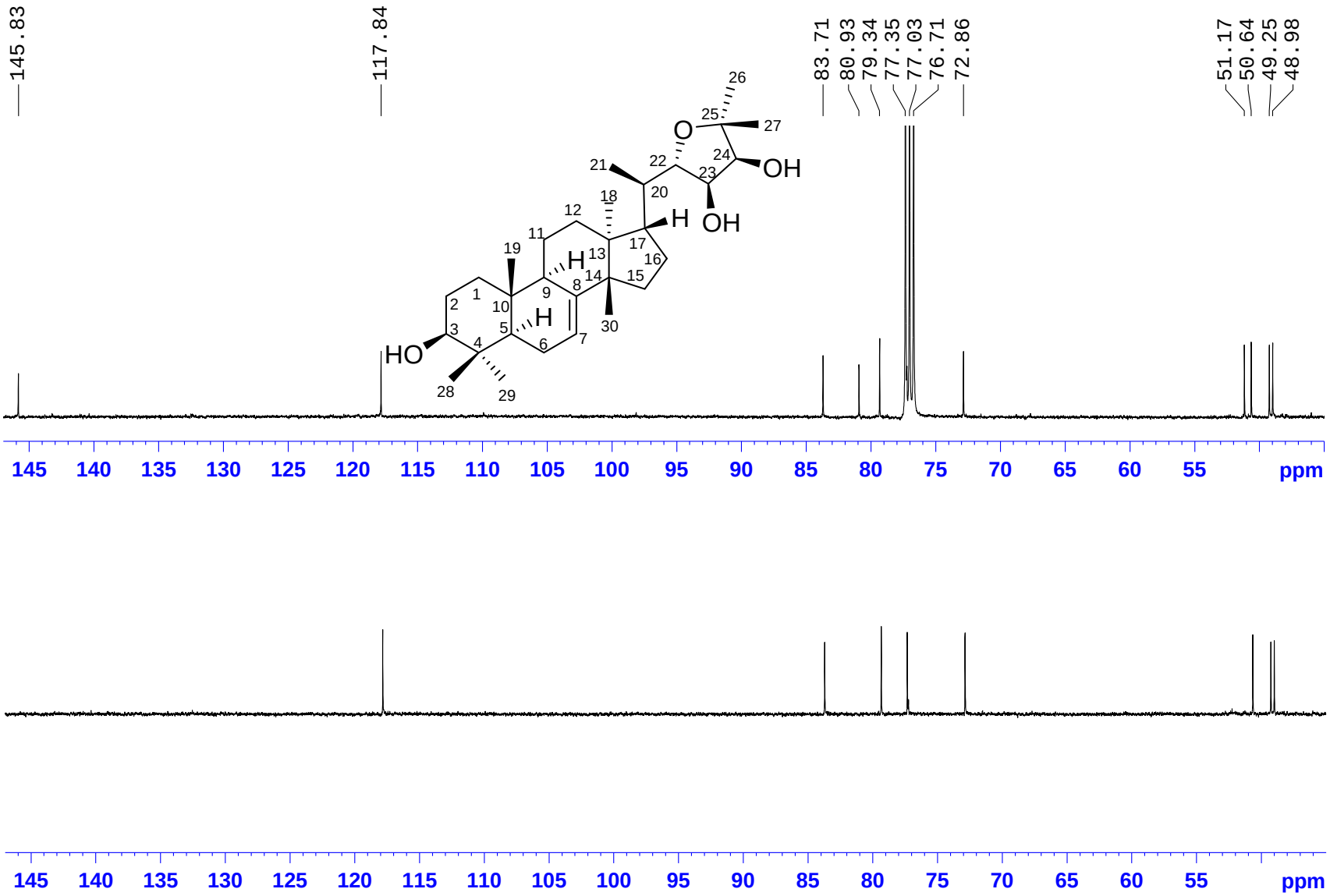
DEPT 135 spectrum of compound **4** in $CDCl_3$



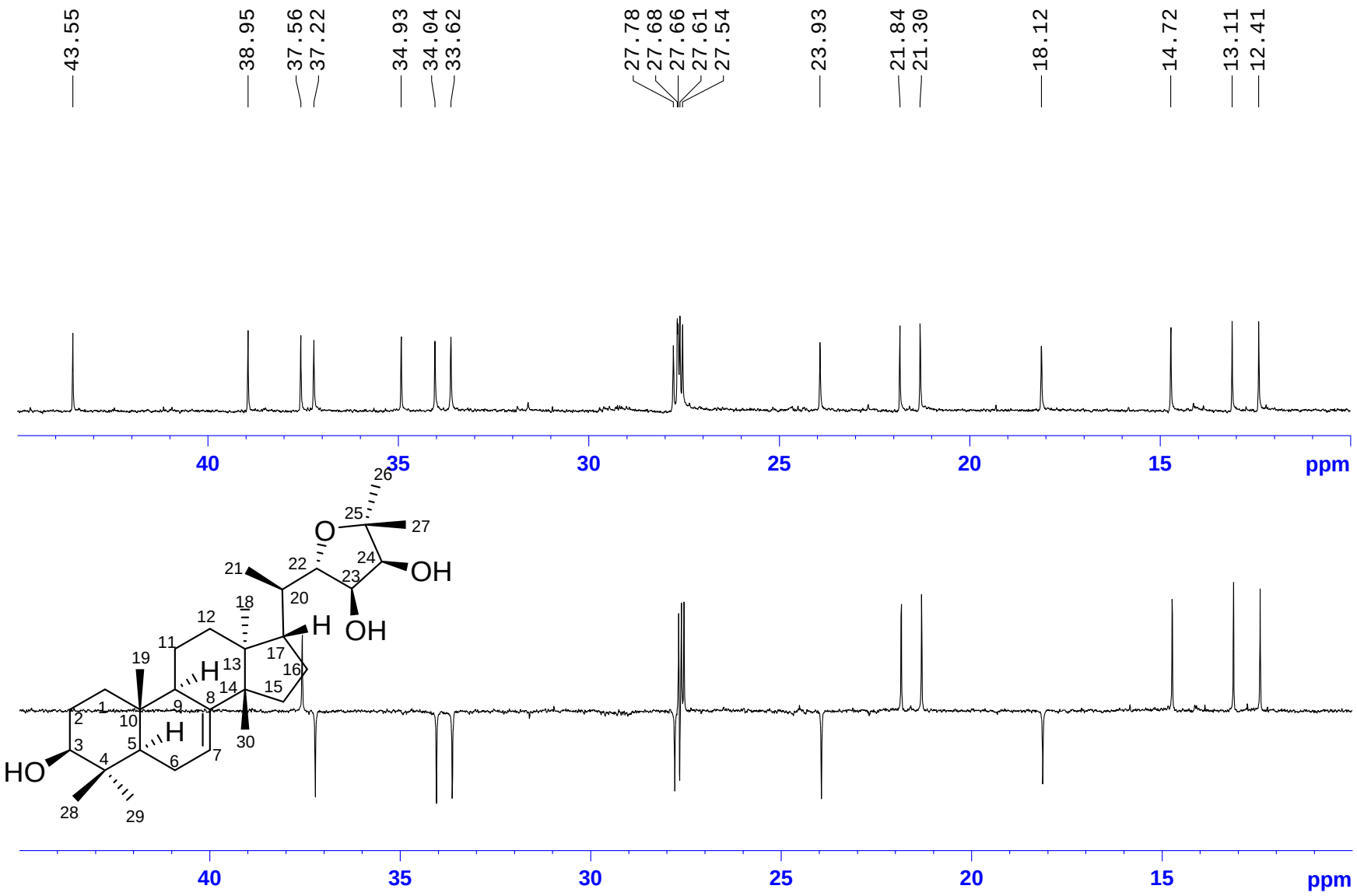

```
NAME          LRX-XG-53-5
EXPNO                   2
PROCNO                  1
Date_            20171226
Time               19.20
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                 73.92
DW                20.800 usec
DE                 18.00 usec
TE                 297.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6233324 MHz
NUC1                 13C
P1                 10.00 usec
SI                 32768
SF           100.6127692 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

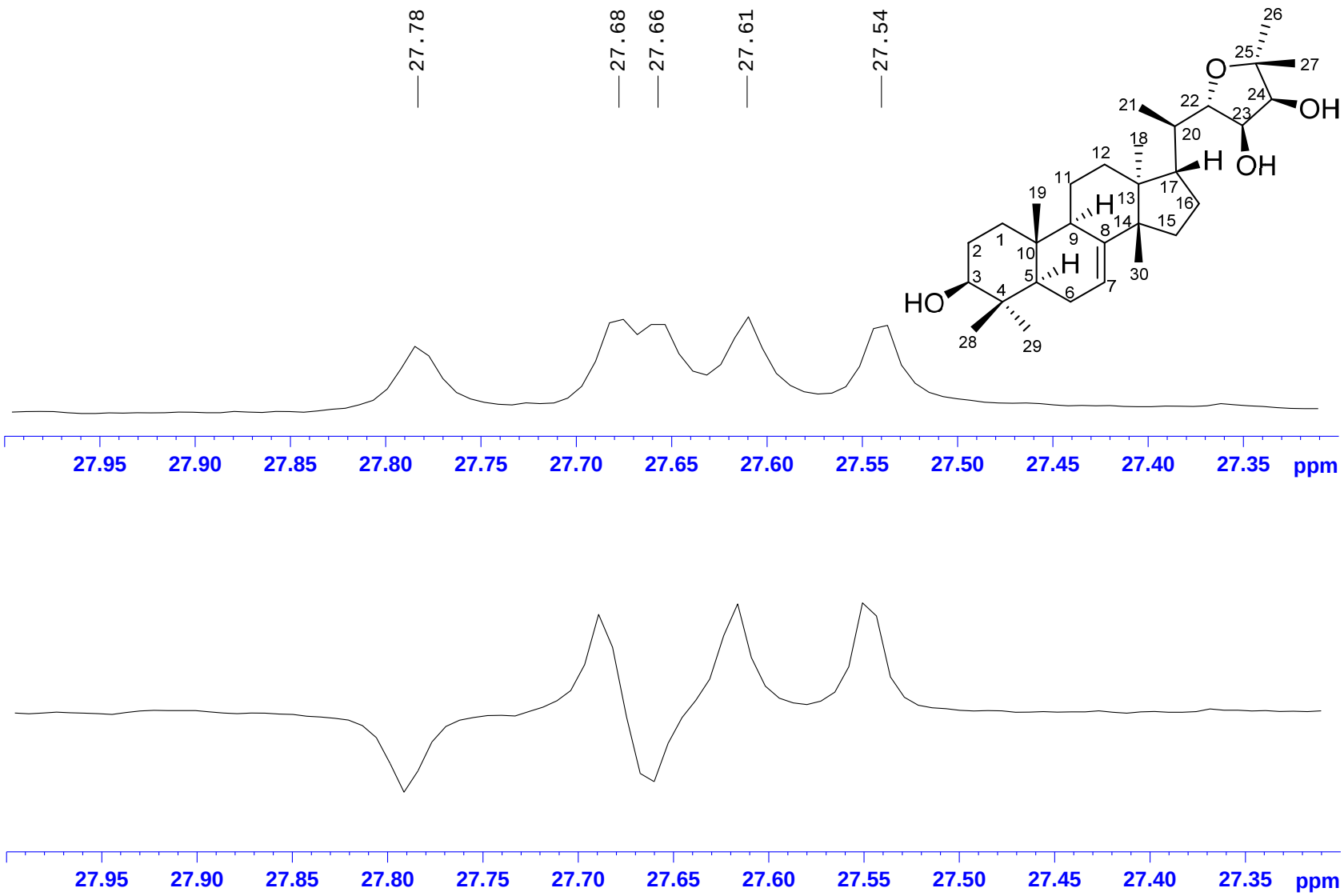
DEPT 135 spectrum of compound **4** in $CDCl_3$



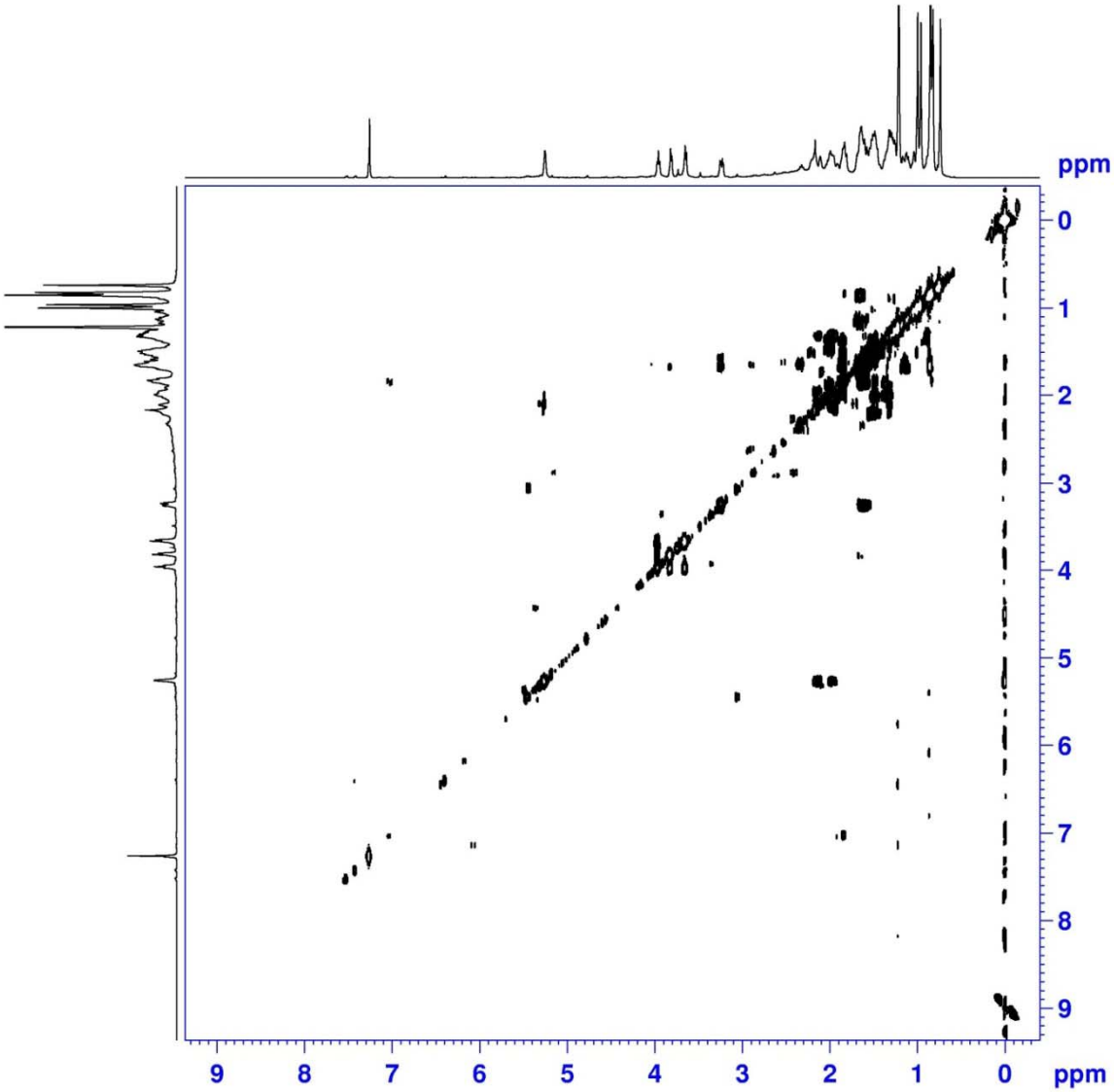
DEPT 135 spectrum of compound **4** in $CDCl_3$



DEPT 135 spectrum of compound **4** in $CDCl_3$



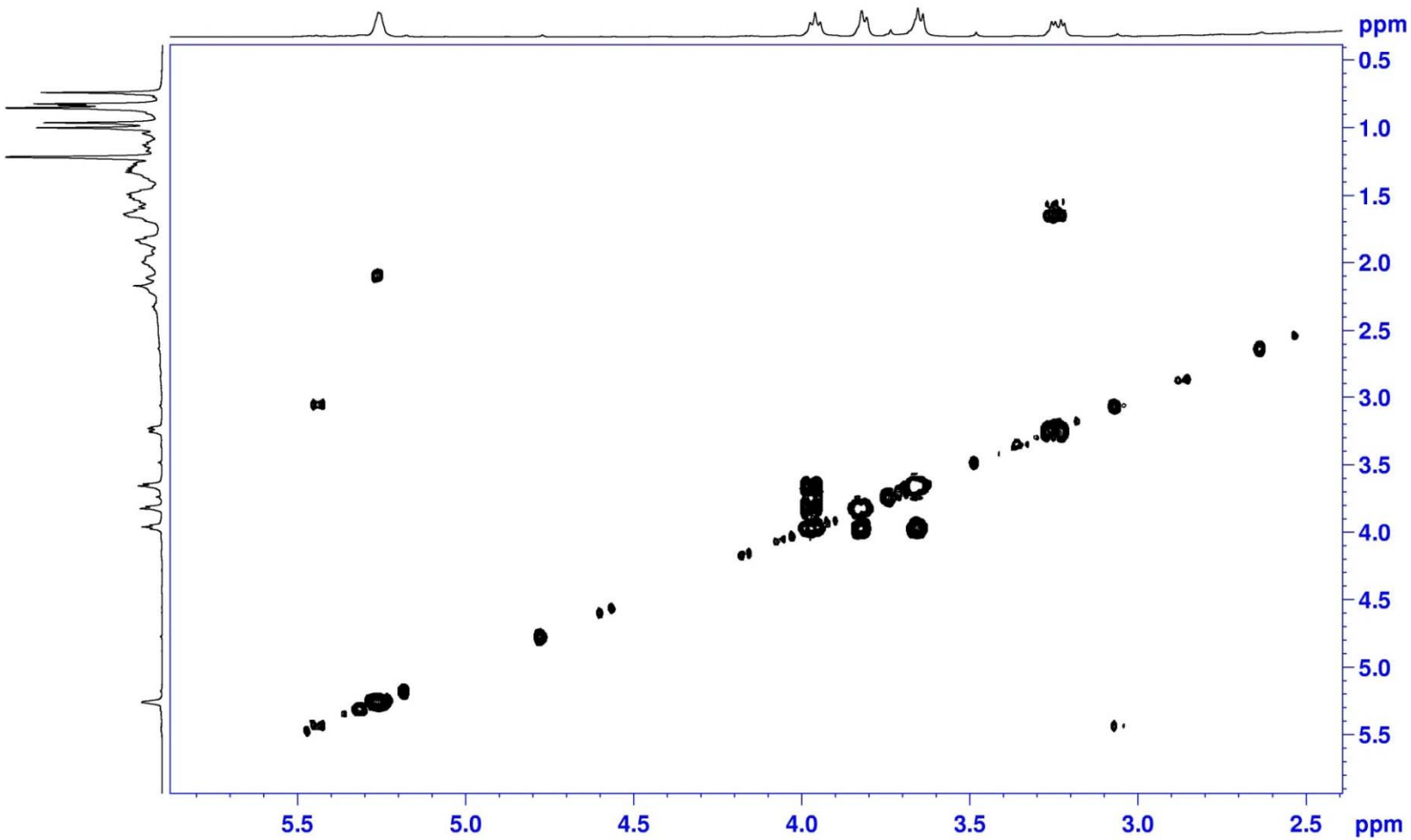
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **4** in $CDCl_3$



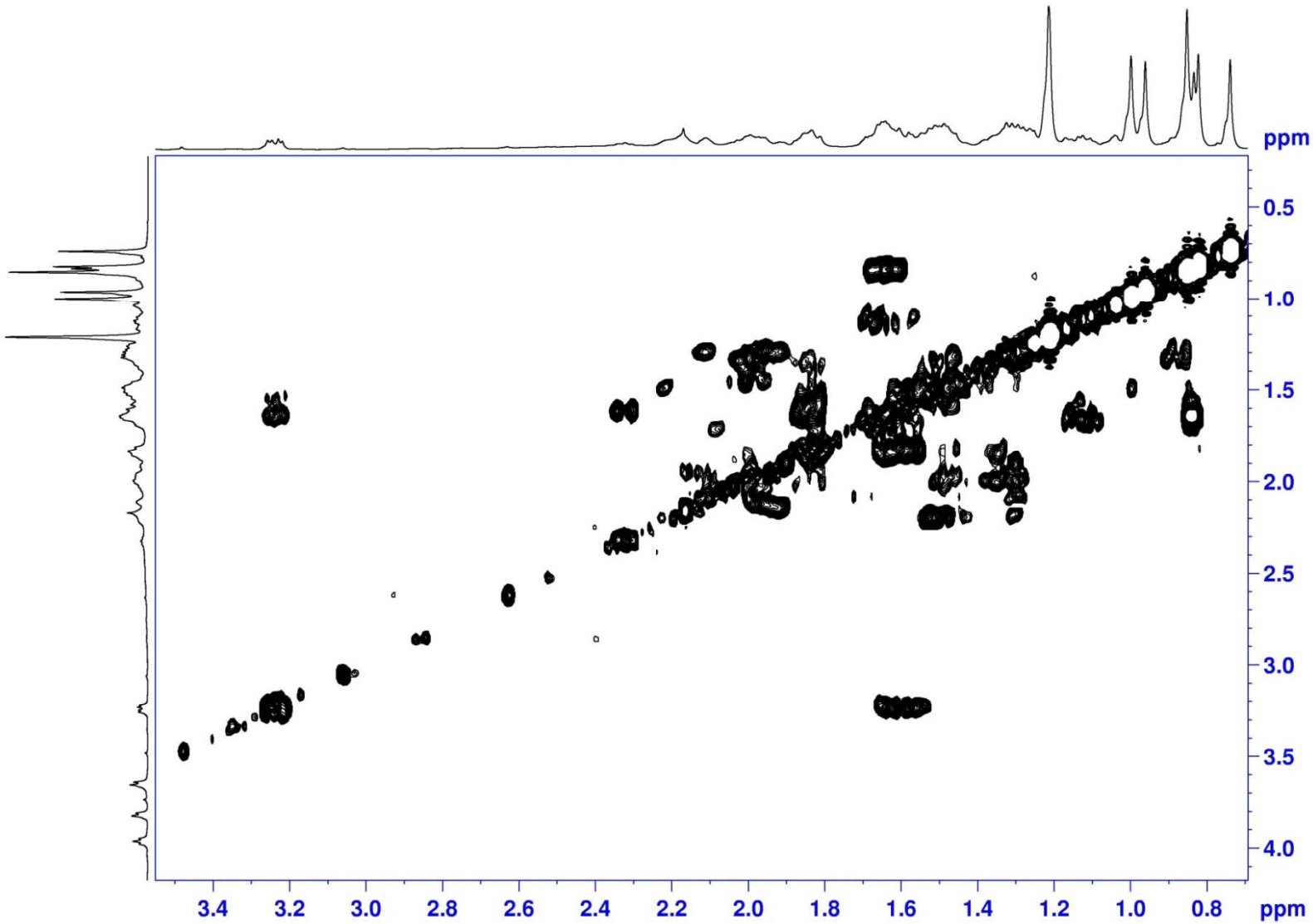

```
NAME          LRX-XG-53-5
EXPNO                   4
PROCNO                  1
Date_            20180112
Time                22.45
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG       cosygpppqf
TD                   2048
SOLVENT             CDCl3
NS                      8
DS                      8
SWH              3906.250 Hz
FIDRES           1.907349 Hz
AQ              0.2621940 sec
RG                  91.64
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            400.1300068 MHz
WDW                  QSINE
SSB                     0
LB                    0.00 Hz
GB                      0
PC                    1.40
SI                    1024
MC2                     QF
SF            400.1300075 MHz
WDW                  QSINE
SSB                     0
LB                    0.00 Hz
GB                      0
```

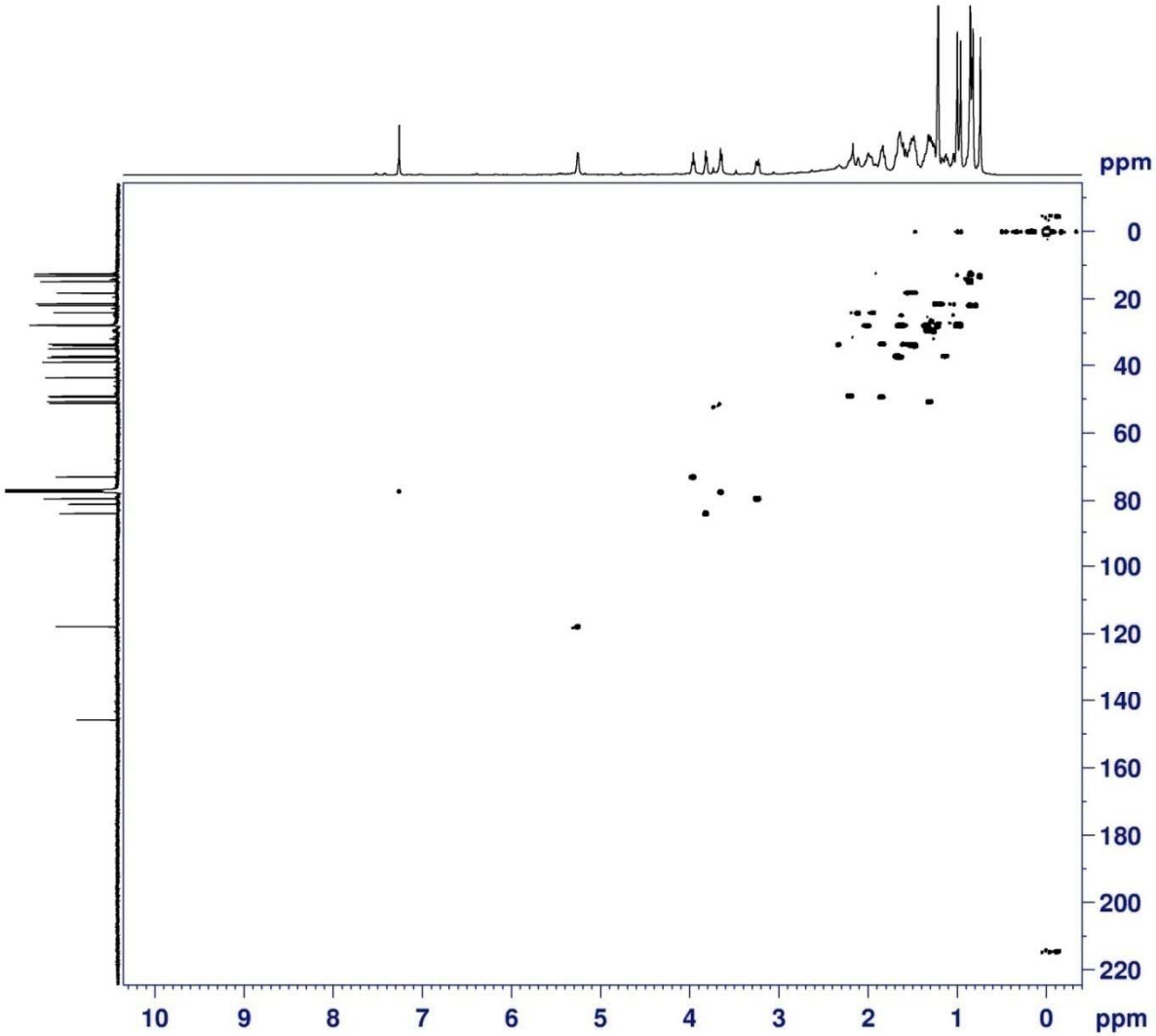
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **4** in $CDCl_3$



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



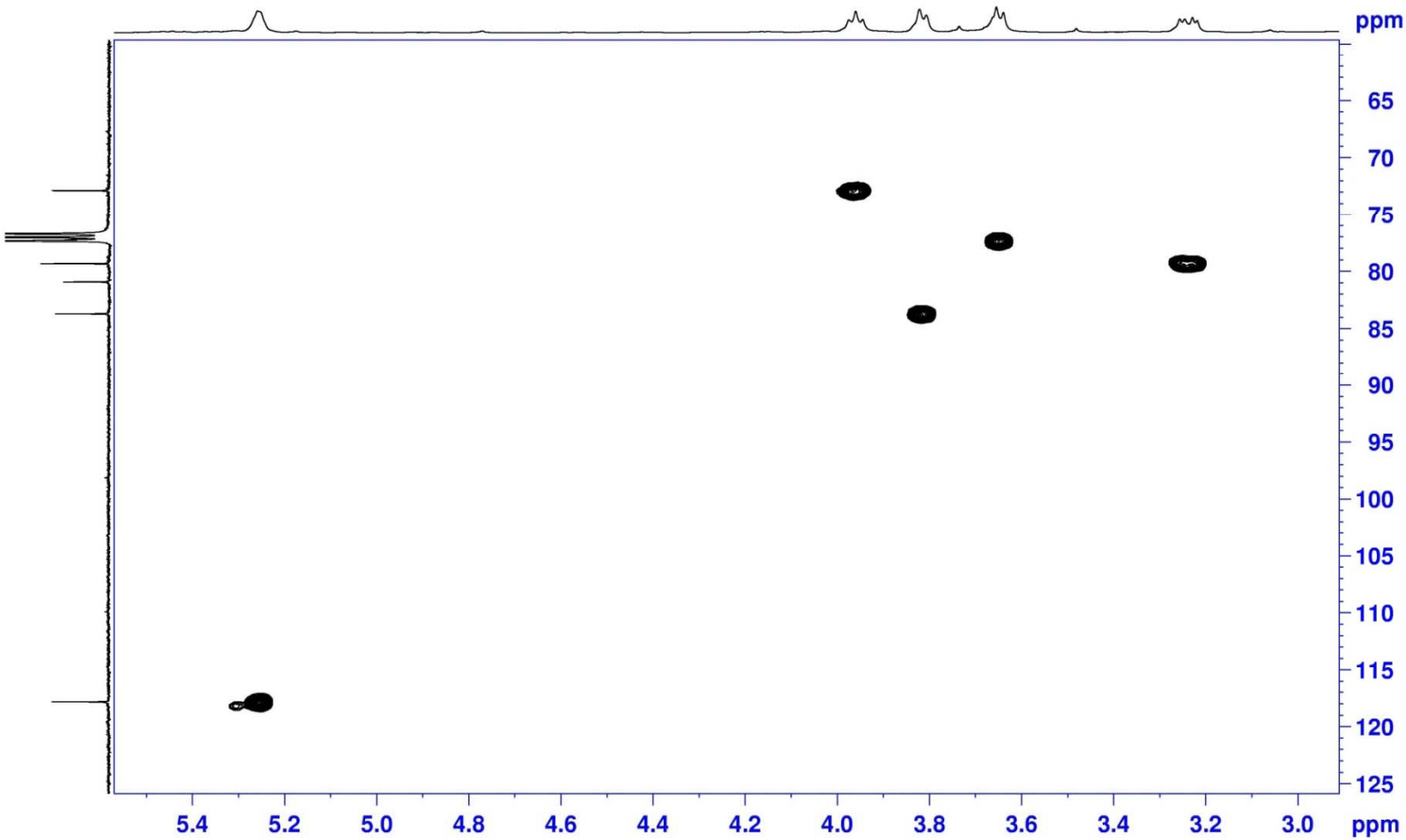
HSQC (400 MHz) spectrum of compound **4** in $CDCl_3$



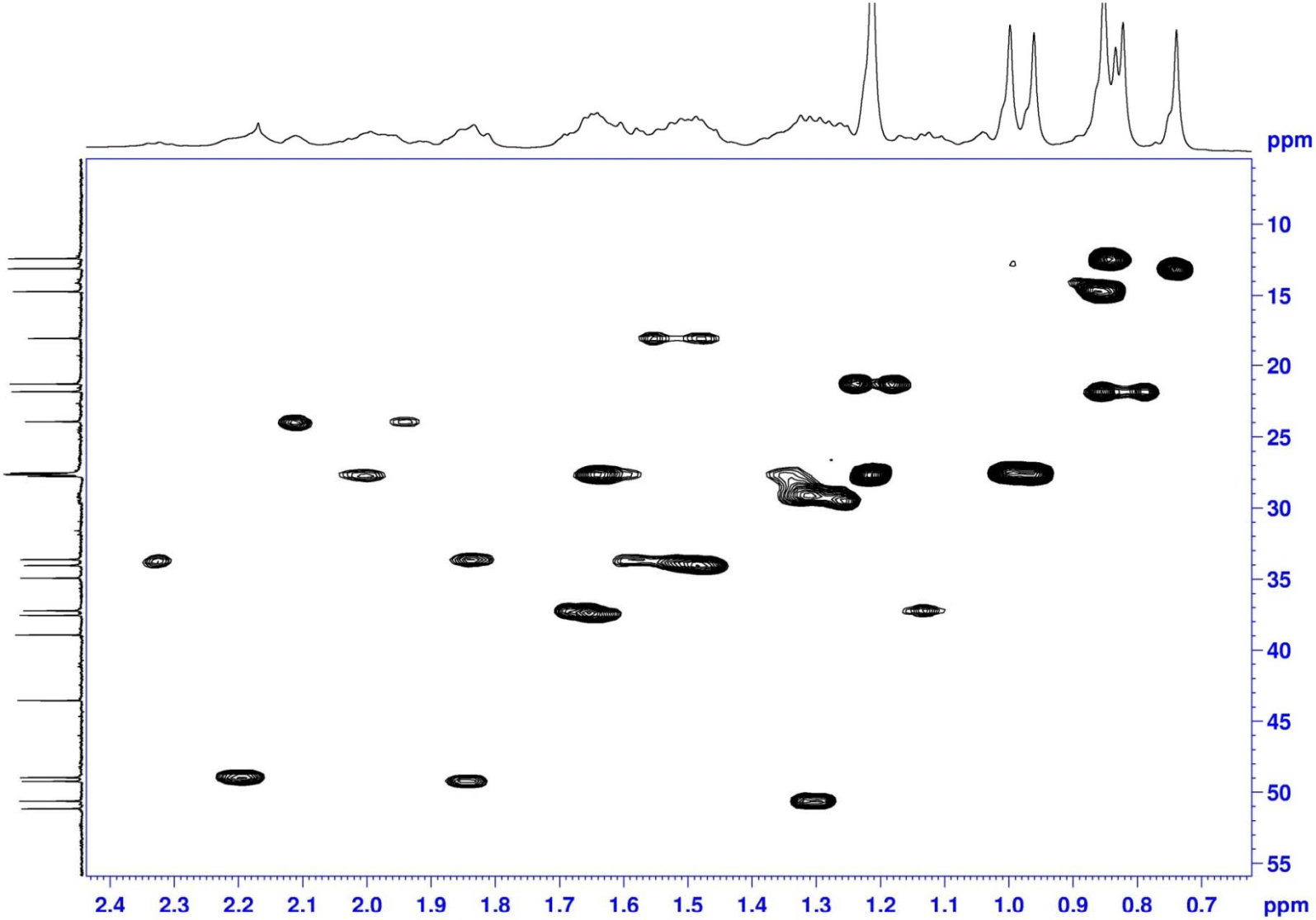

```
NAME          LRX-XG-53-5
EXPNO                   5
PROCNO                  1
Date_            20180112
Time                23.24
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      hsqcetgpsi2
TD                   1024
SOLVENT             CDCl3
NS                     16
DS                     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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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           400.1300095 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2        echo-antiecho
SF           100.6127551 MHz
WDW                 QSINE
SSB                    2
```

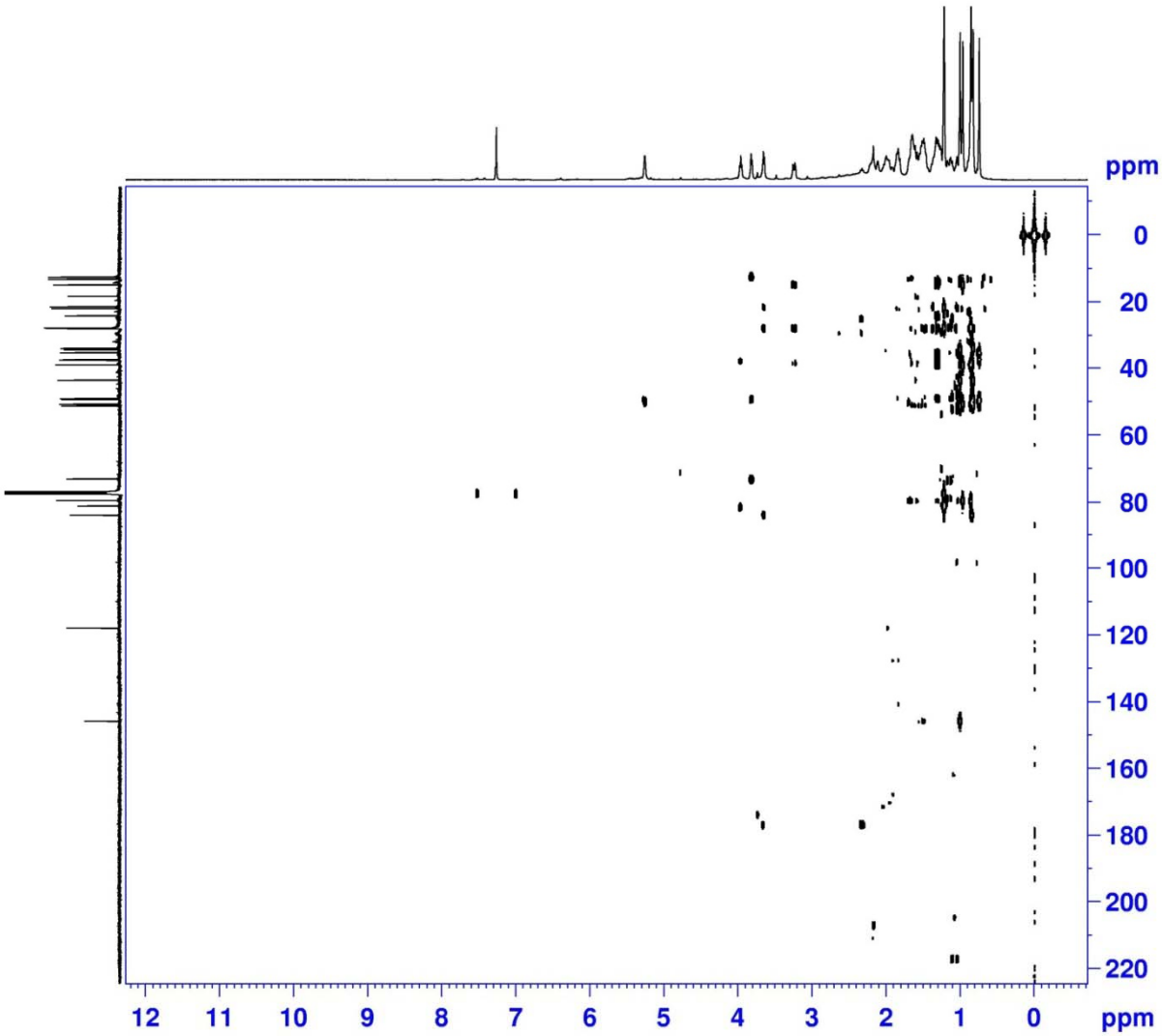
HSQC (400 MHz) spectrum of compound **4** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **4** in $CDCl_3$



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

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NAME          LRX-XG-53-5
EXPNO                   6
PROCNO                  1
Date_            20180113
Time                 1.16
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG    hmbcgplpndqf
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 ========
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
SI                  2048
SF           400.1300105 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           100.6127526 MHz
WDW                 SINE
SSB                    0
```
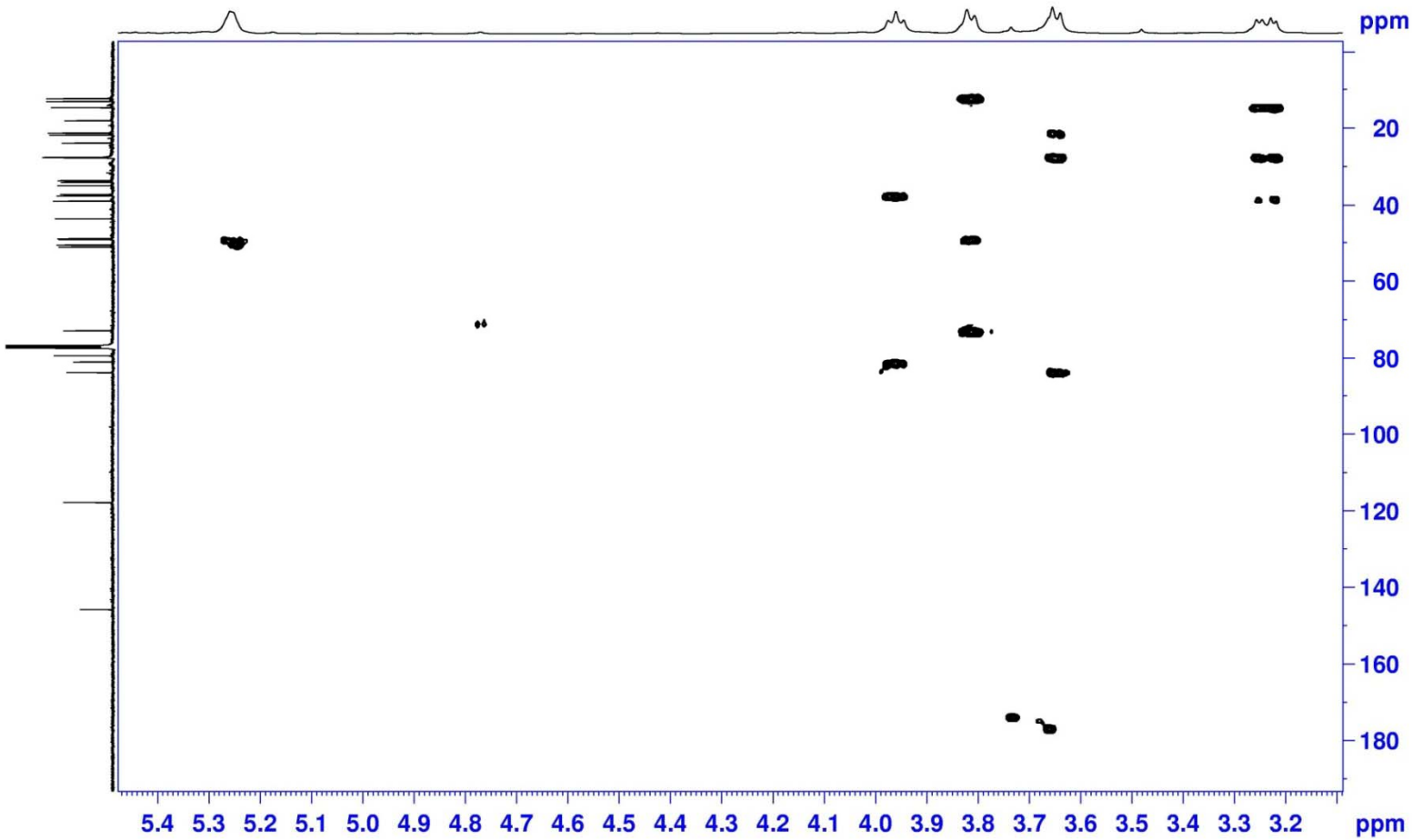
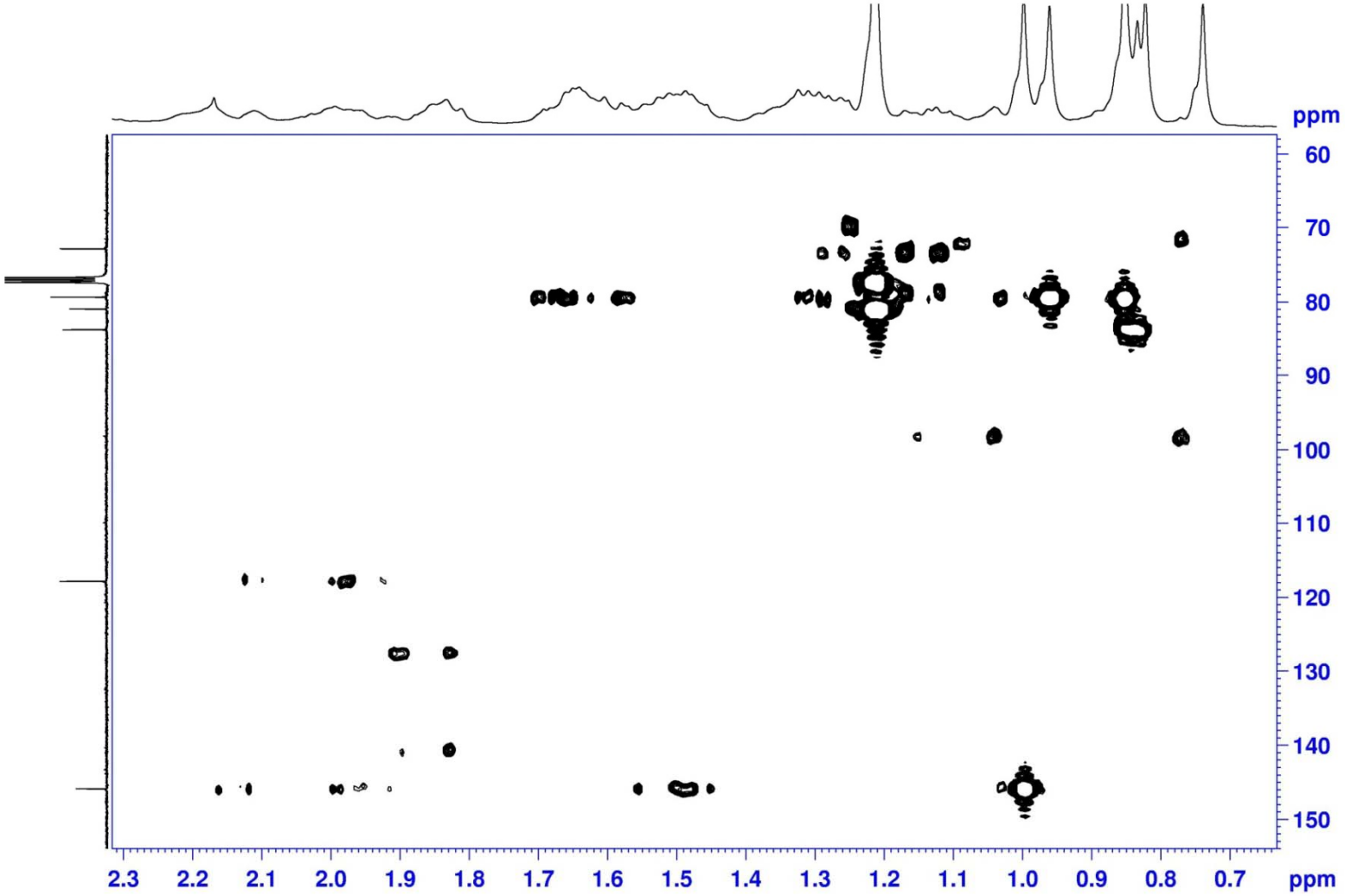
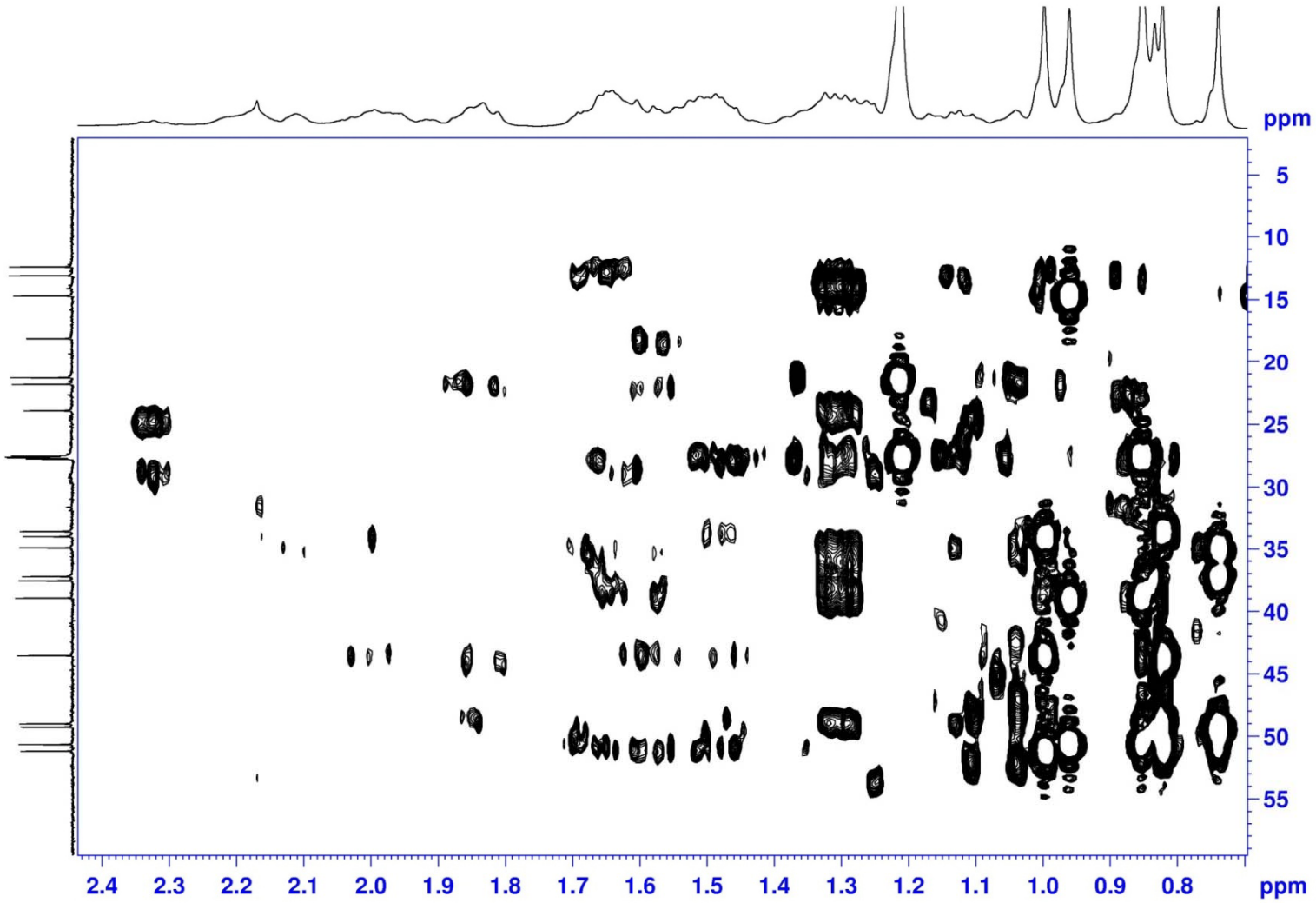

HMBC (400 MHz) spectrum of compound **4** in $CDCl_3$



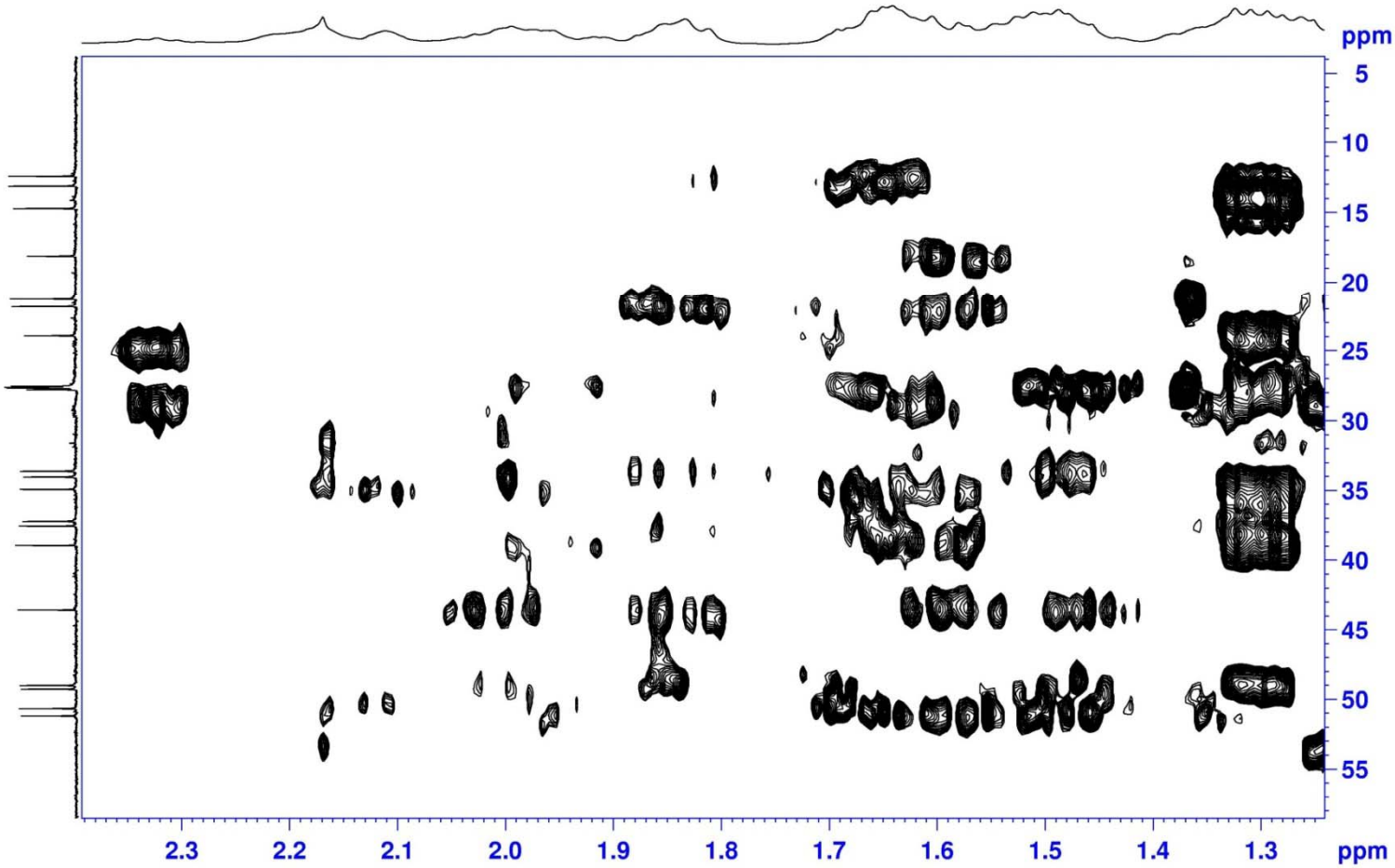
HMBC (400 MHz) spectrum of compound **4** in $CDCl_3$



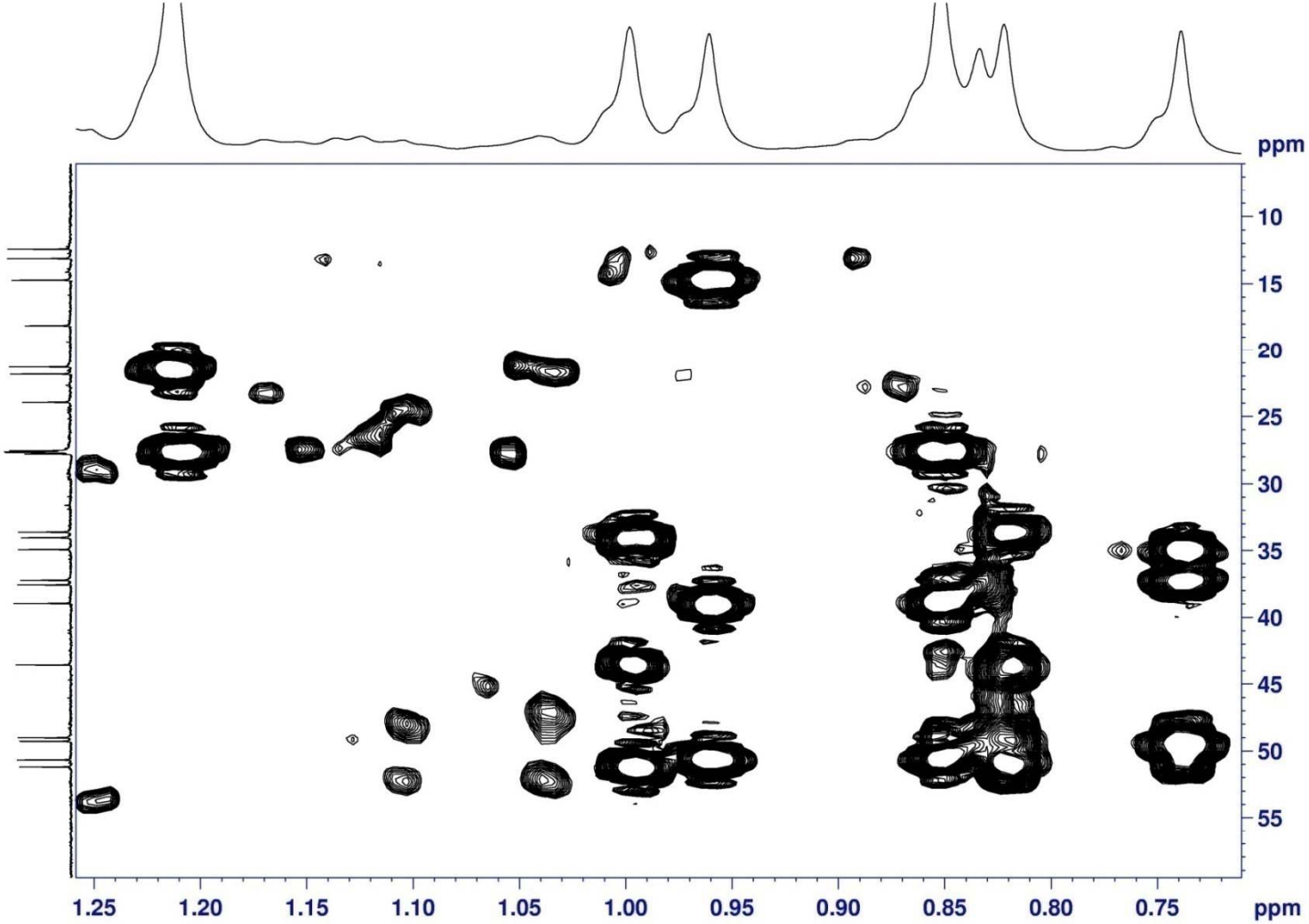
HMBC (400 MHz) spectrum of compound **4** in $CDCl_3$



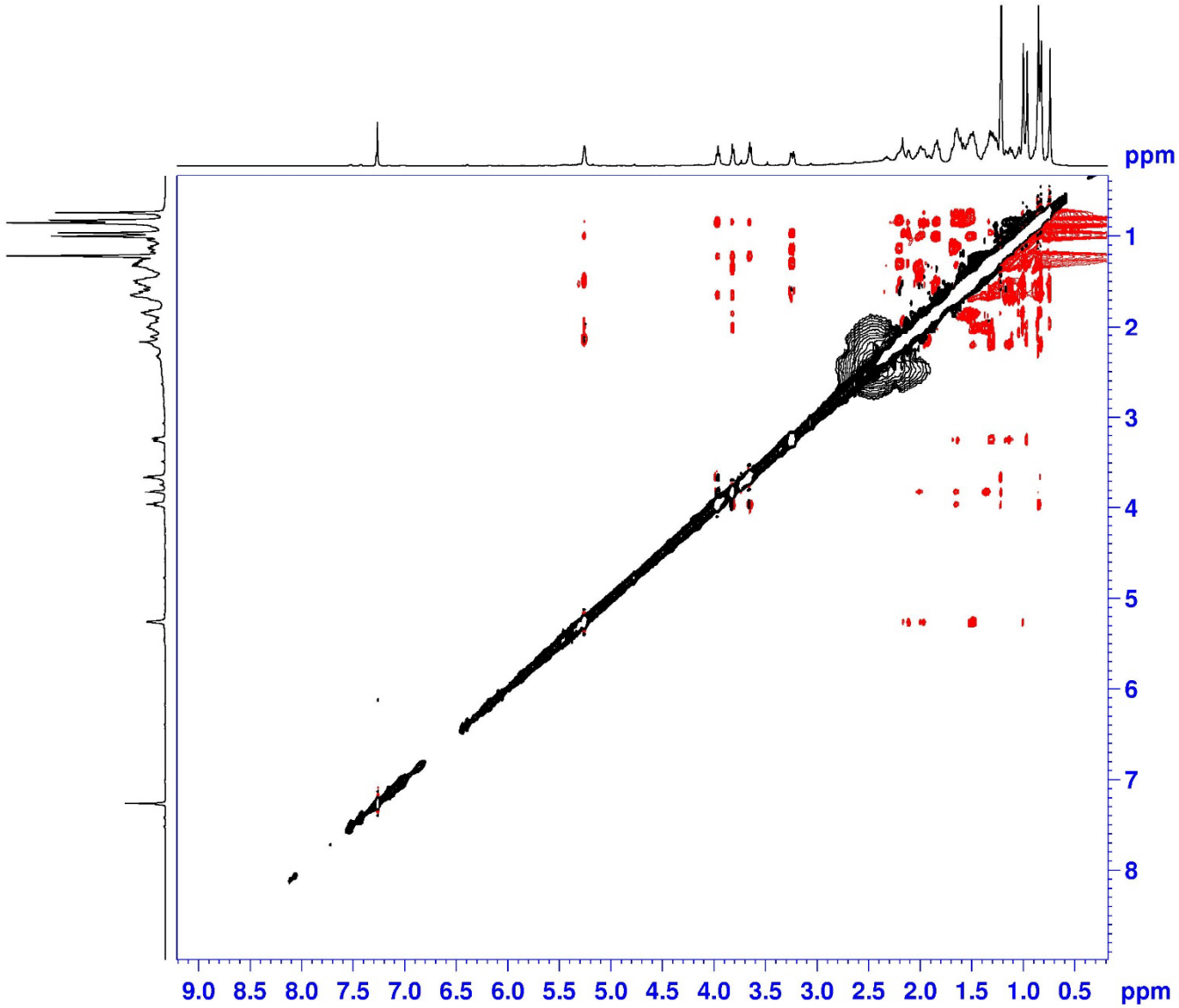
HMBC (400 MHz) spectrum of compound **4** in $CDCl_3$



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

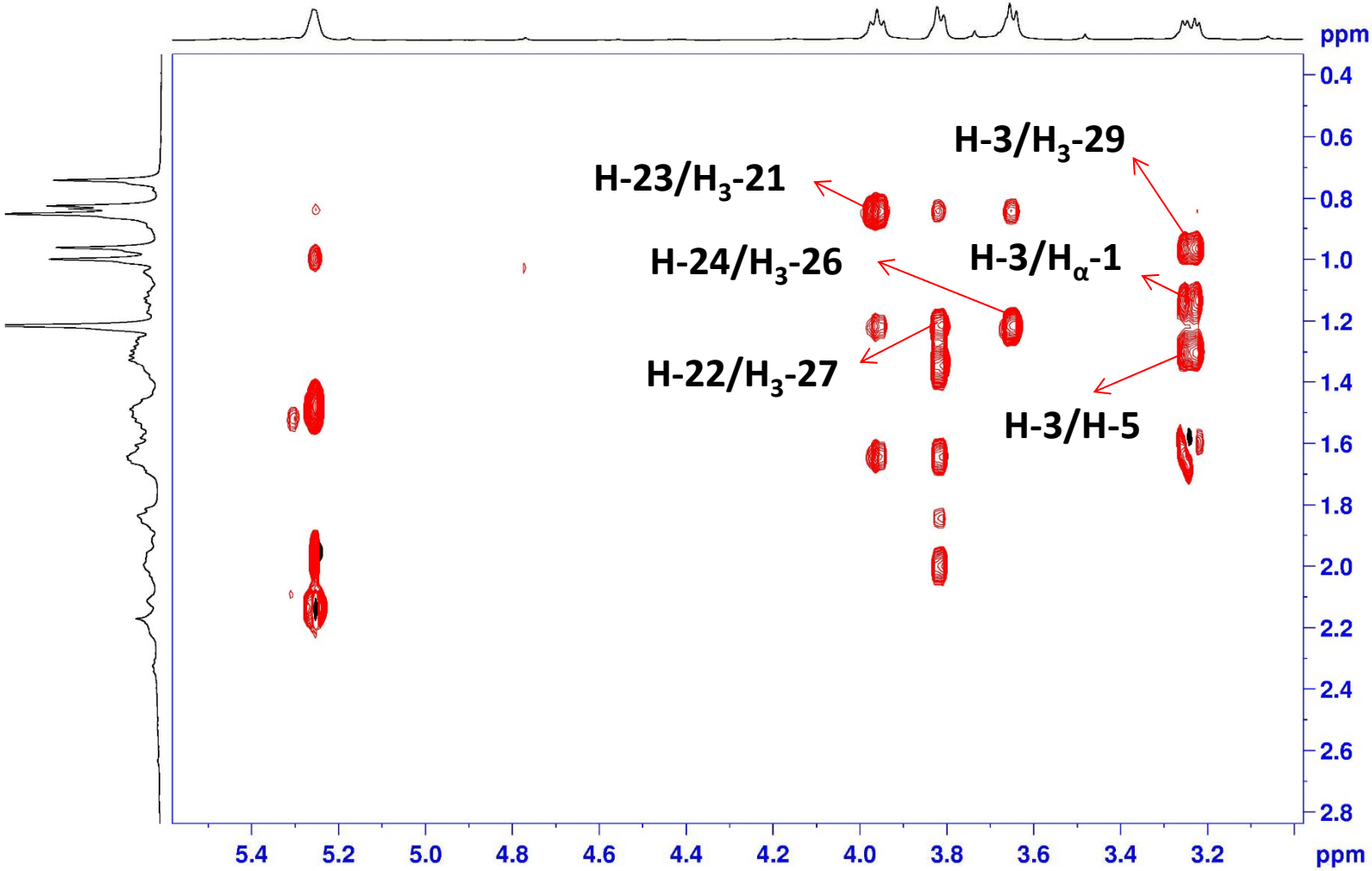


NOESY (400 MHz) spectrum of compound **4** in $CDCl_3$

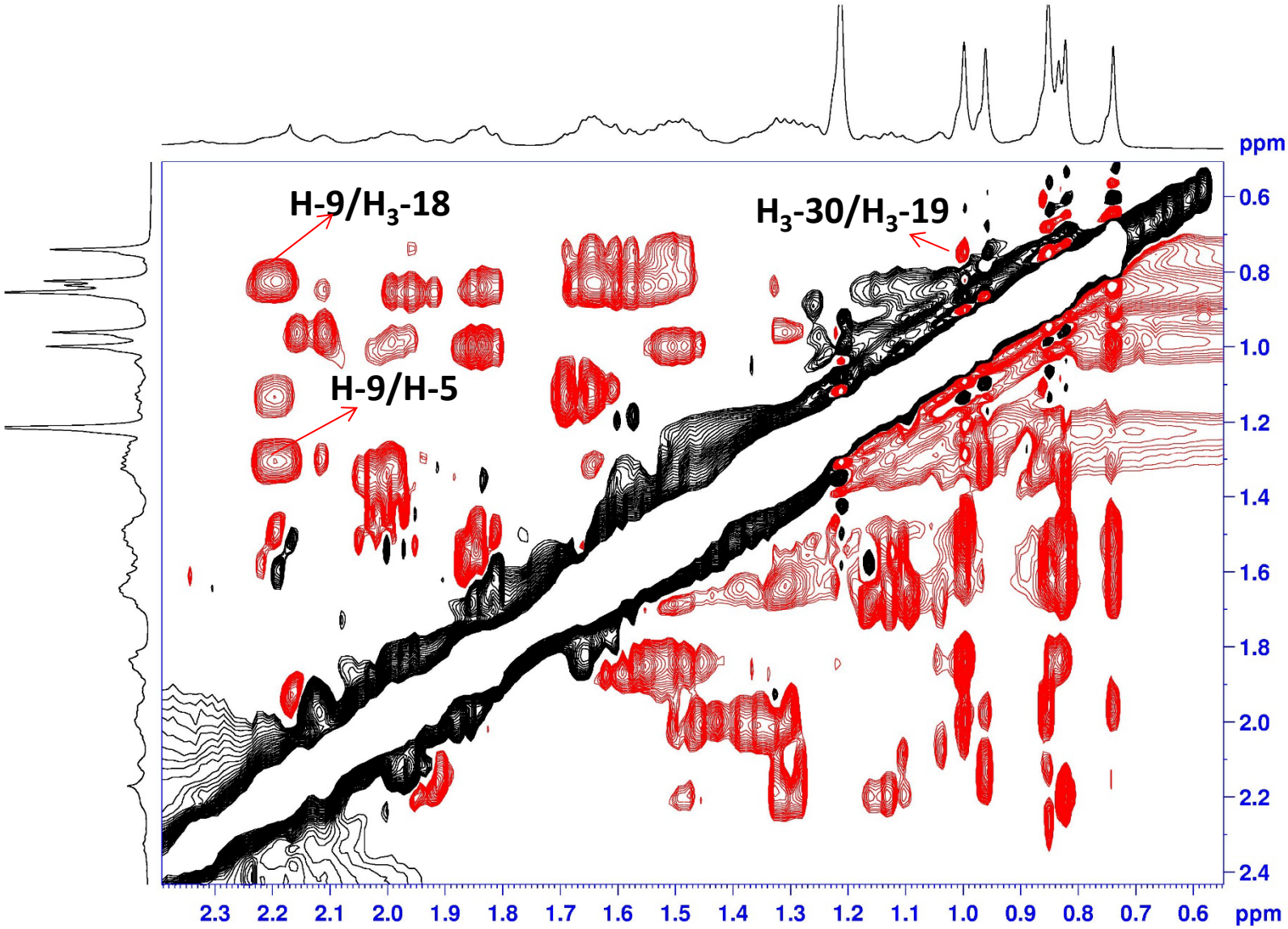



```
NAME          LRX-XG-53-5
EXPNO                   7
PROCNO                  1
Date_            20180113
Time                 3.31
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                     16
DS                     32
SWH             4000.000 Hz
FIDRES          1.953125 Hz
AQ             0.2560500 se
RG                 91.64
DW                125.000 us
DE                 10.00 us
TE                 297.0 K
D0             0.00011036 se
D1             1.99385595 se
D8             0.30000001 se
D11            0.03000000 se
D12            0.00002000 se
D16            0.00020000 se
IN0            0.00025000 se

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

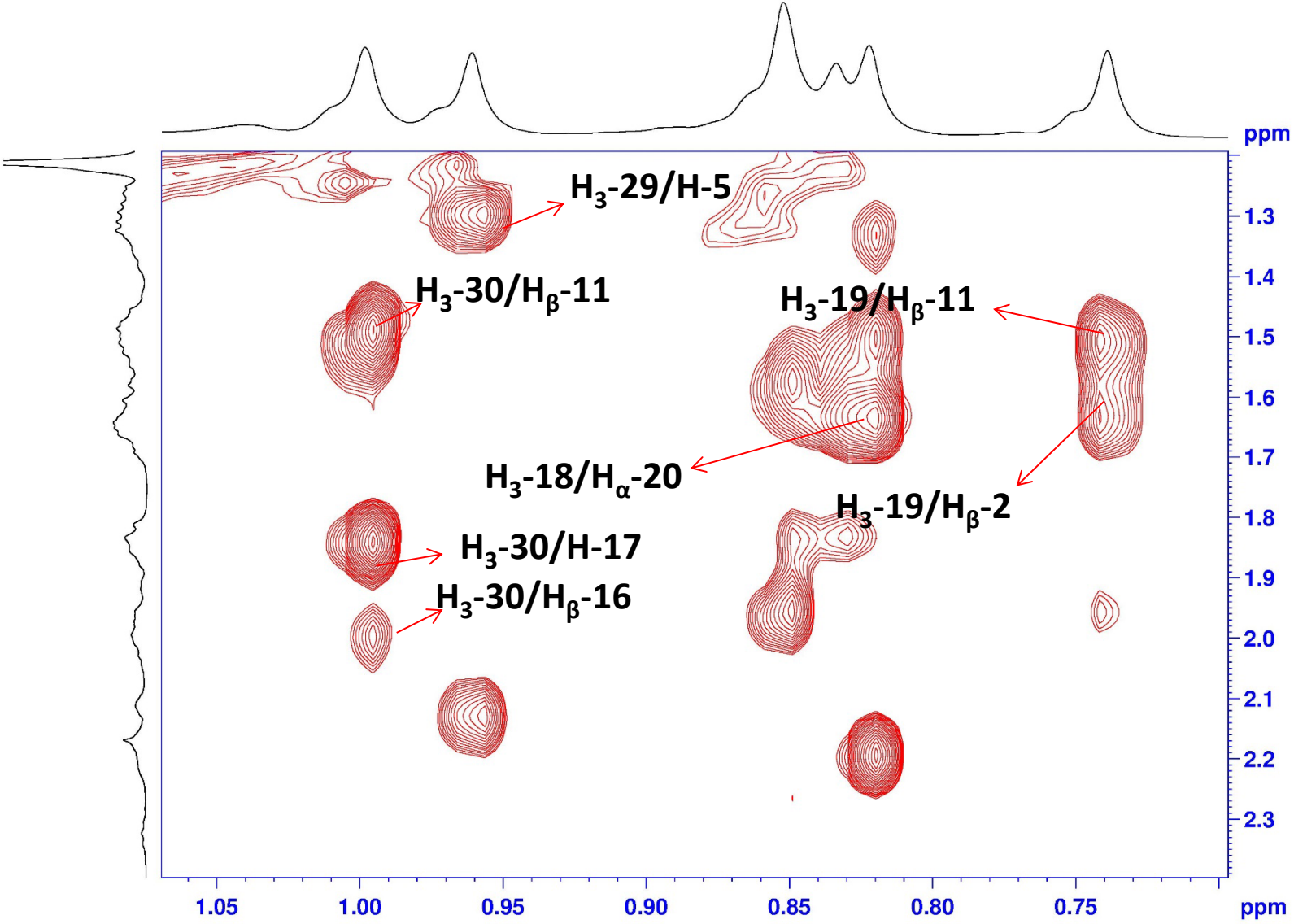
NOESY (400 MHz) spectrum of compound **4** in $CDCl_3$



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



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

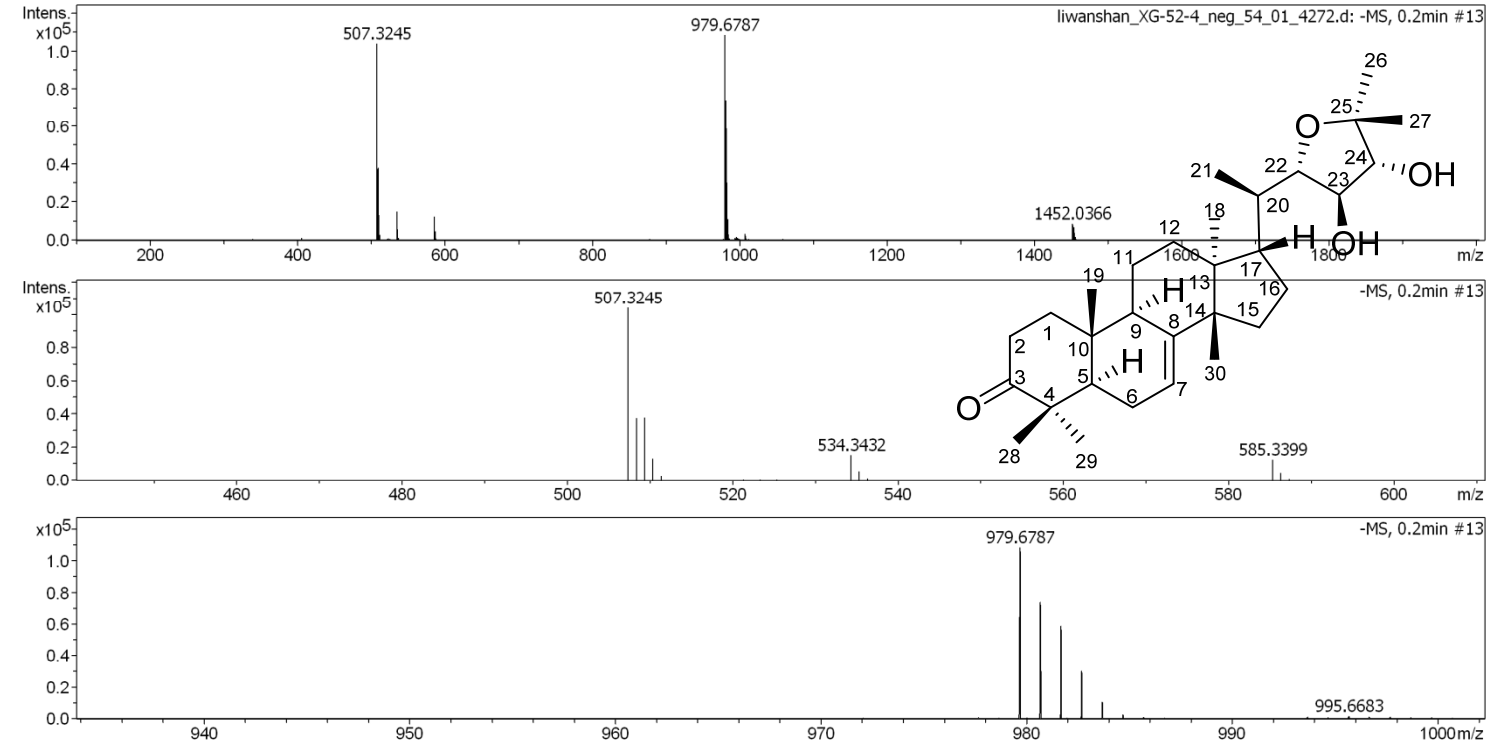


HR-ESIMS for compound **5**

Generic Display Report

Analysis Info

Analysis Name	D:\Data\MS\data\201802\liwanshan_XG-52-4_neg_54_01_4272.d	Acquisition Date	2/8/2018 5:06:23 PM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_XG-52-4_neg	Instrument	maXis
Comment			



Bruker Compass DataAnalysis 4.1 printed: 2/8/2018 5:21:51 PM by: SCSIO Page 1 of 1

HR-ESIMS for compound **5**

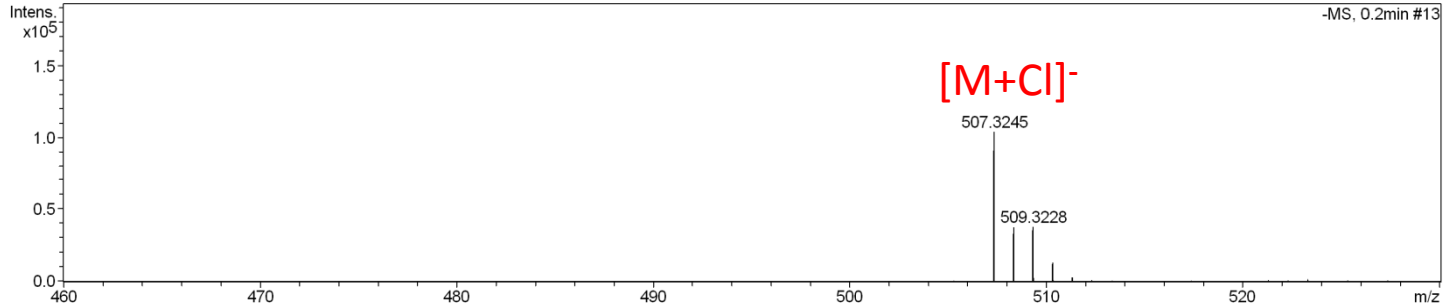
Mass Spectrum SmartFormula Report

Analysis Info

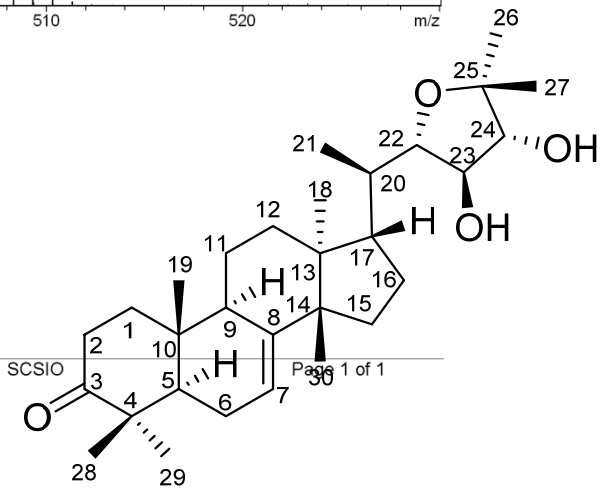
Analysis Name	D:\Data\MS\data\201802\liwanshan_XG-52-4_neg_54_01_4272.d	Acquisition Date	2/8/2018 5:06:23 PM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	liwanshan_XG-52-4_neg	Instrument	maXis 255552.00029
Comment			

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



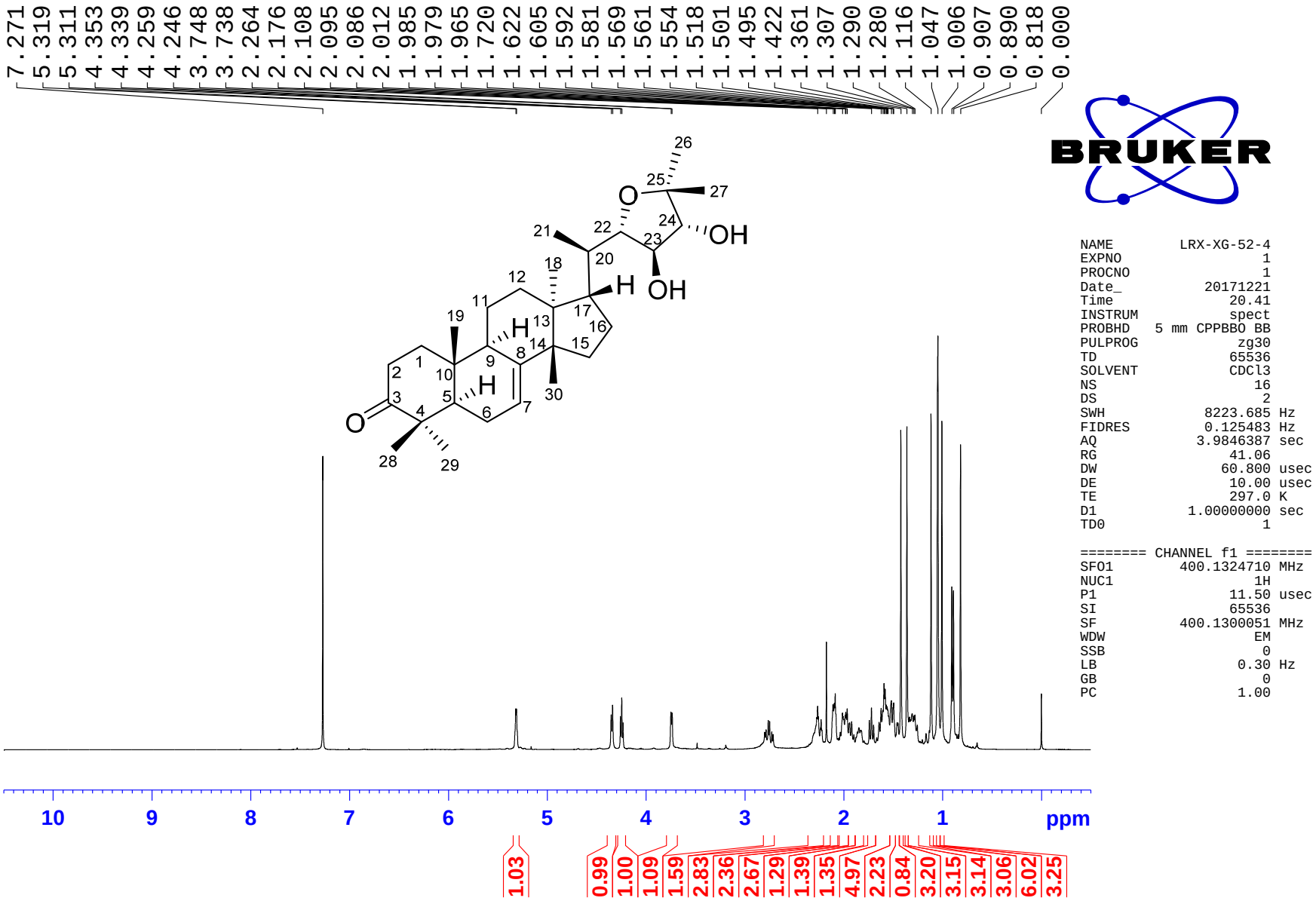
Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
507.3245	1	C30H48ClO4	100.00	507.3247	-0.3	-0.2	15.8	6.5	even	ok
979.6787	1	C60H96ClO8	70.91	979.6799	-1.2	-1.2	10.7	12.5	even	ok



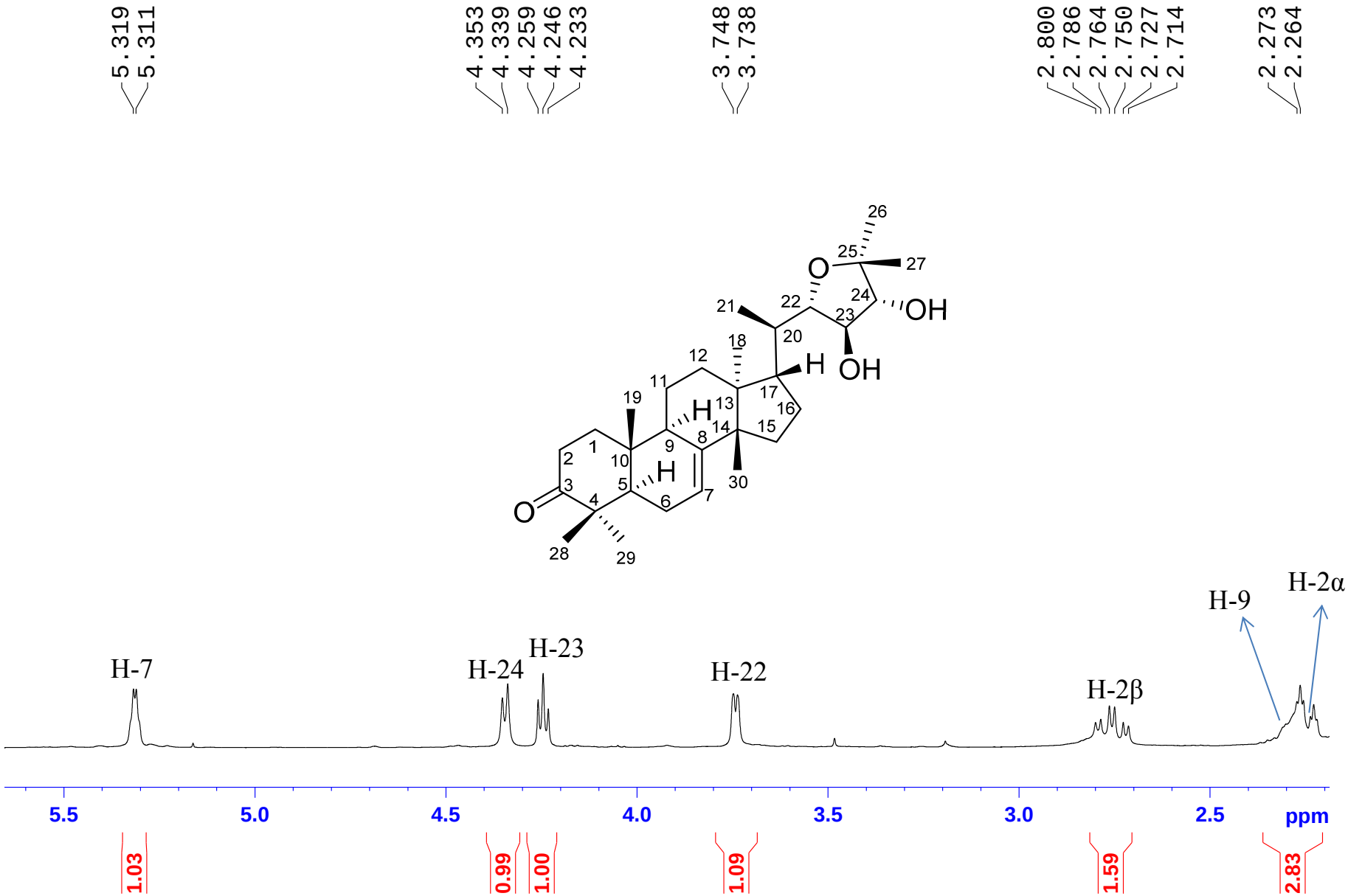
liwanshan_XG-52-4_neg_54_01_4272.d

Bruker Compass DataAnalysis 4.1 printed: 2/8/2018 5:21:18 PM by: SCSIO Page 1 of 1

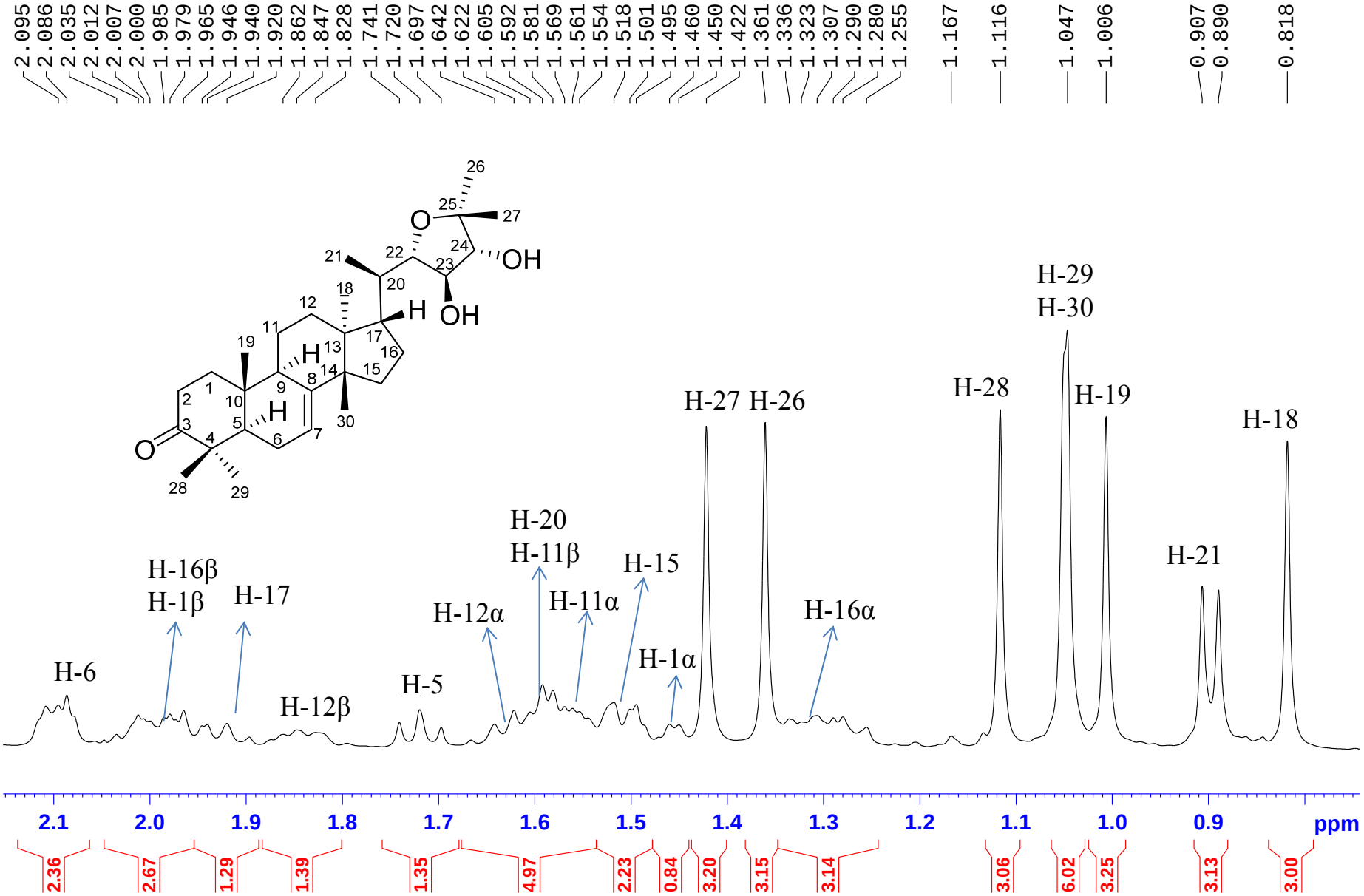
^{1}H NMR (400 MHz) spectrum of compound **5** in $CDCl_3$



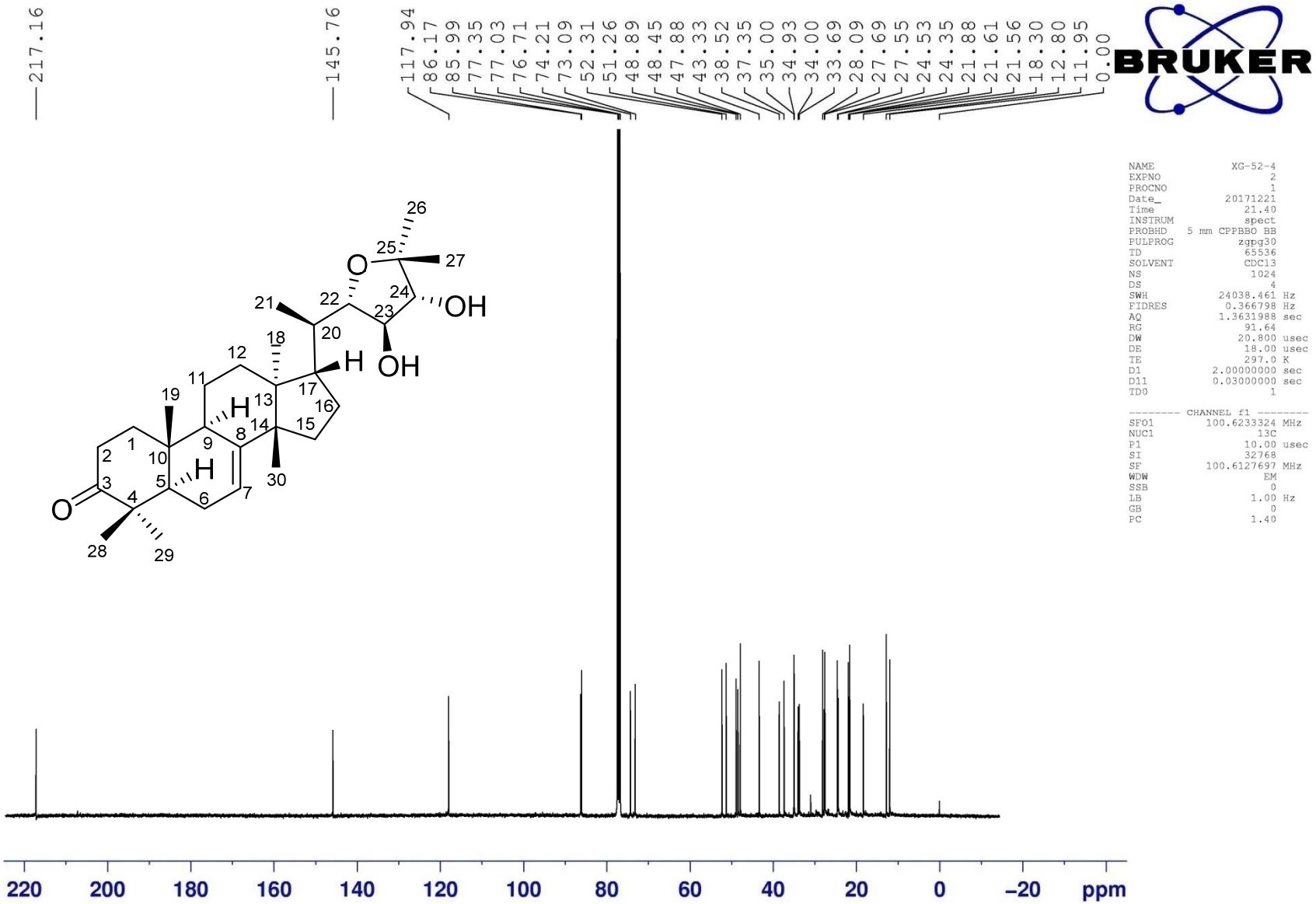
^{1}H NMR (400 MHz) spectrum of compound **5** in $CDCl_3$



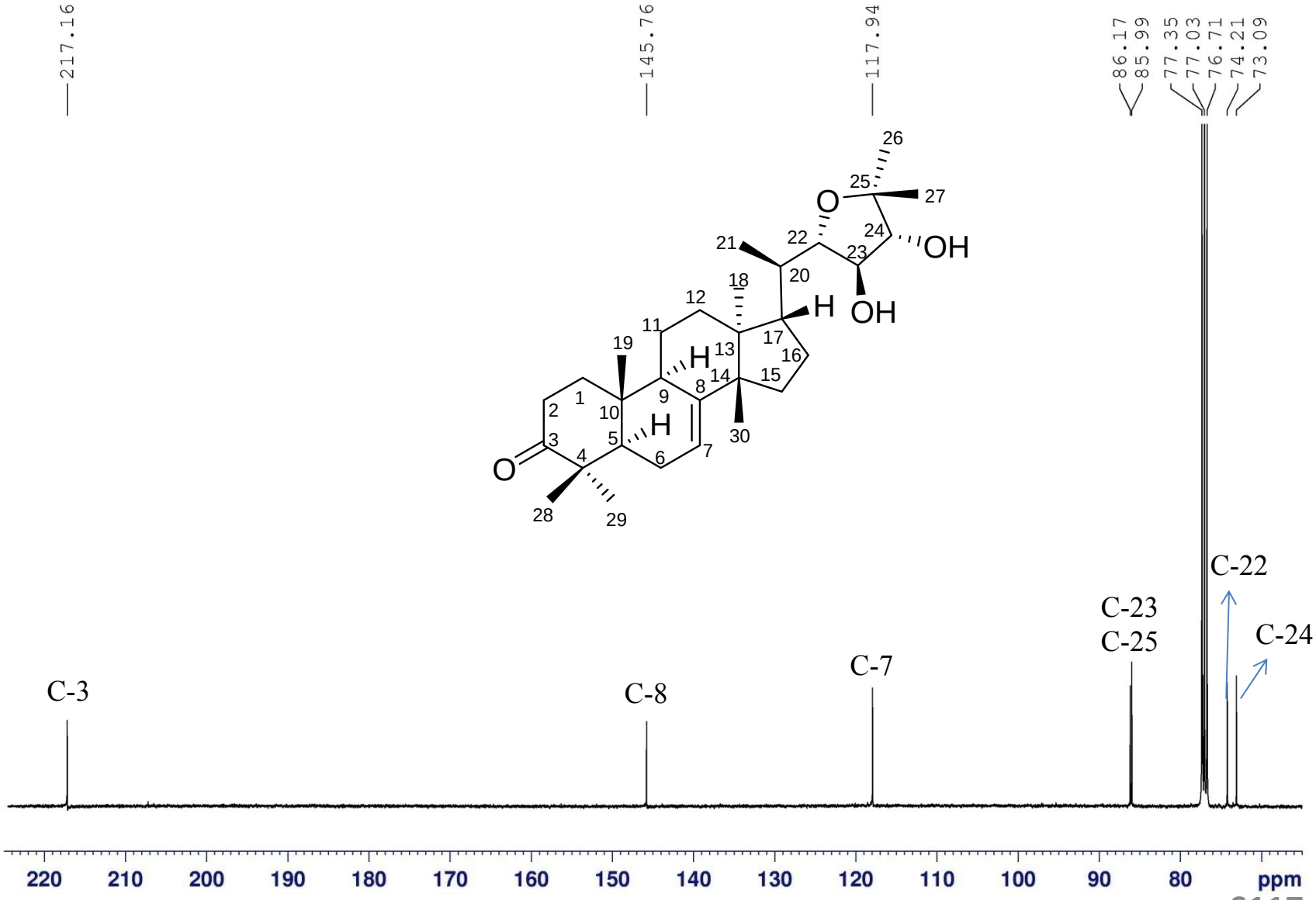
^{1}H NMR (400 MHz) spectrum of compound **5** in $CDCl_3$



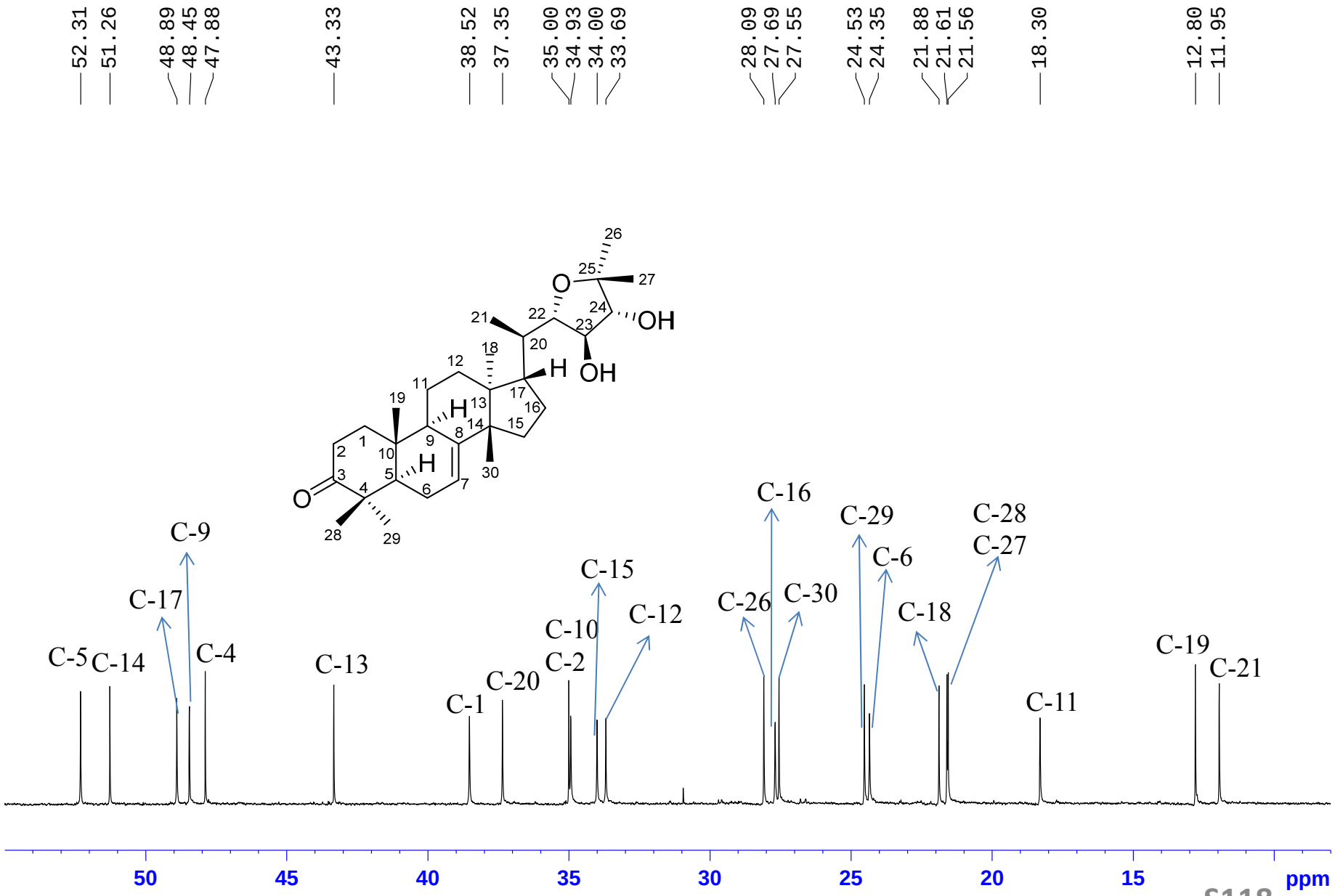
^{13}C NMR (100 MHz) spectrum of compound **5** in $CDCl_3$



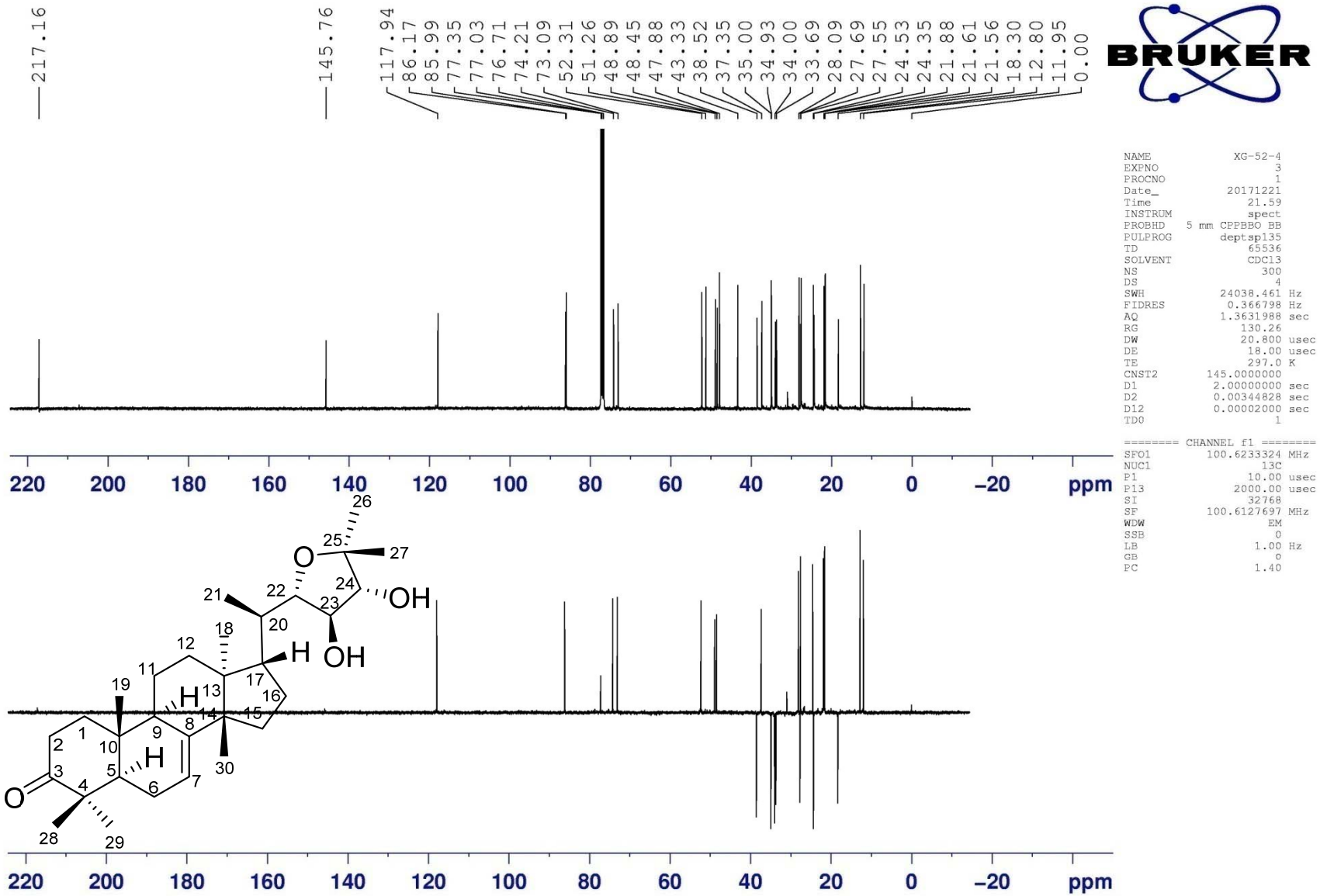
^{13}C NMR (100 MHz) spectrum of compound **5** in $CDCl_3$



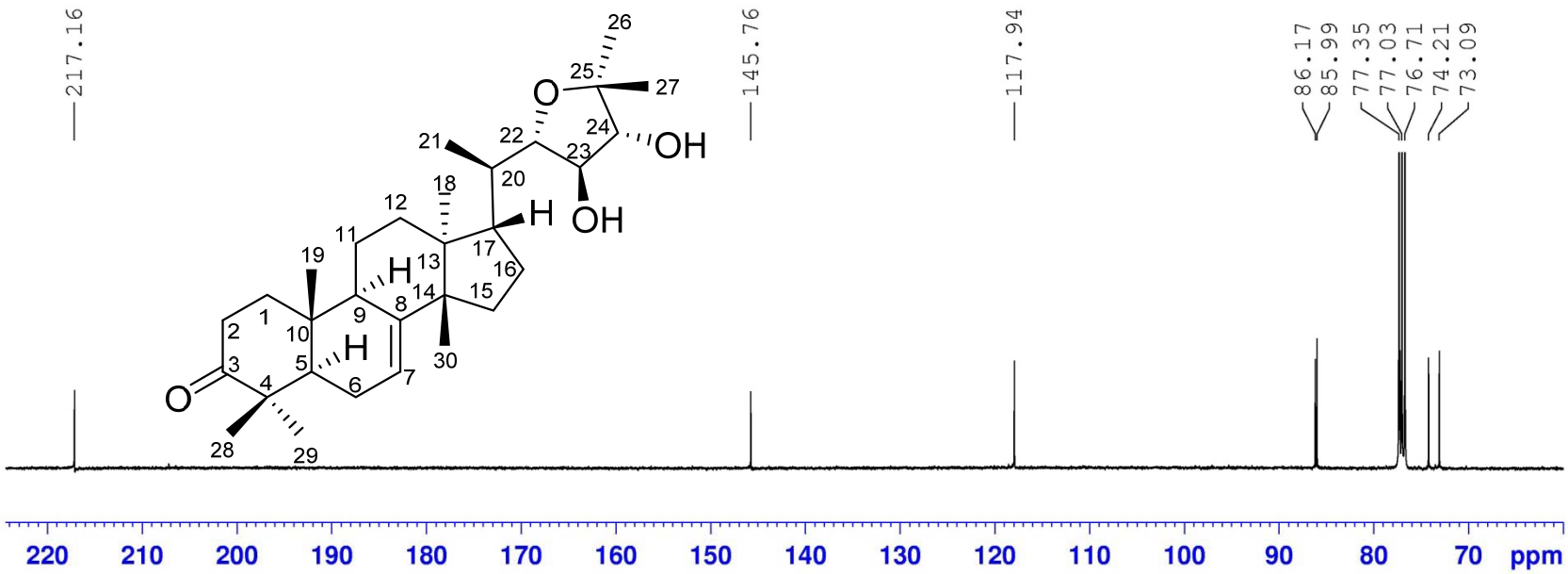
^{13}C NMR (100 MHz) spectrum of compound **5** in $CDCl_3$

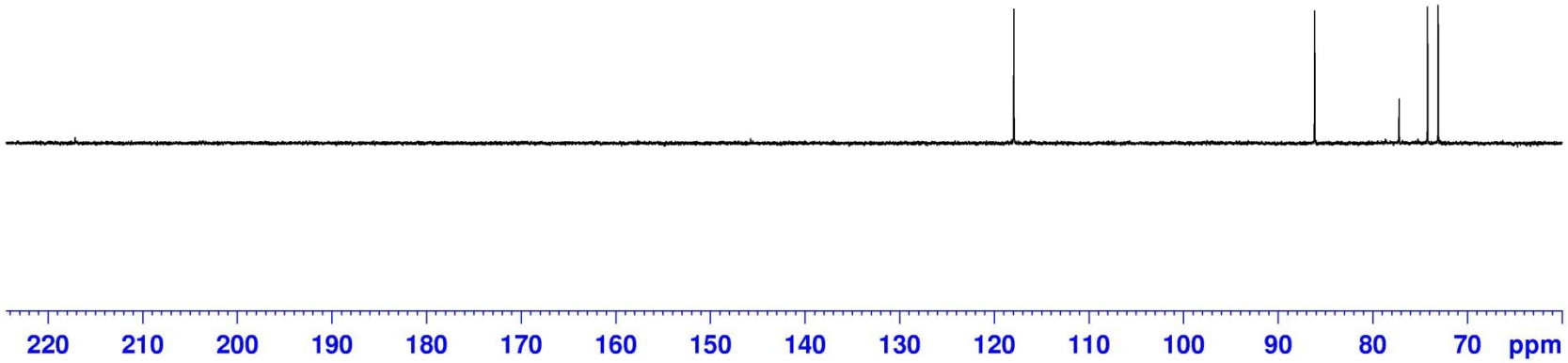


DEPT135 (100 MHz) spectrum of compound **5** in $CDCl_3$

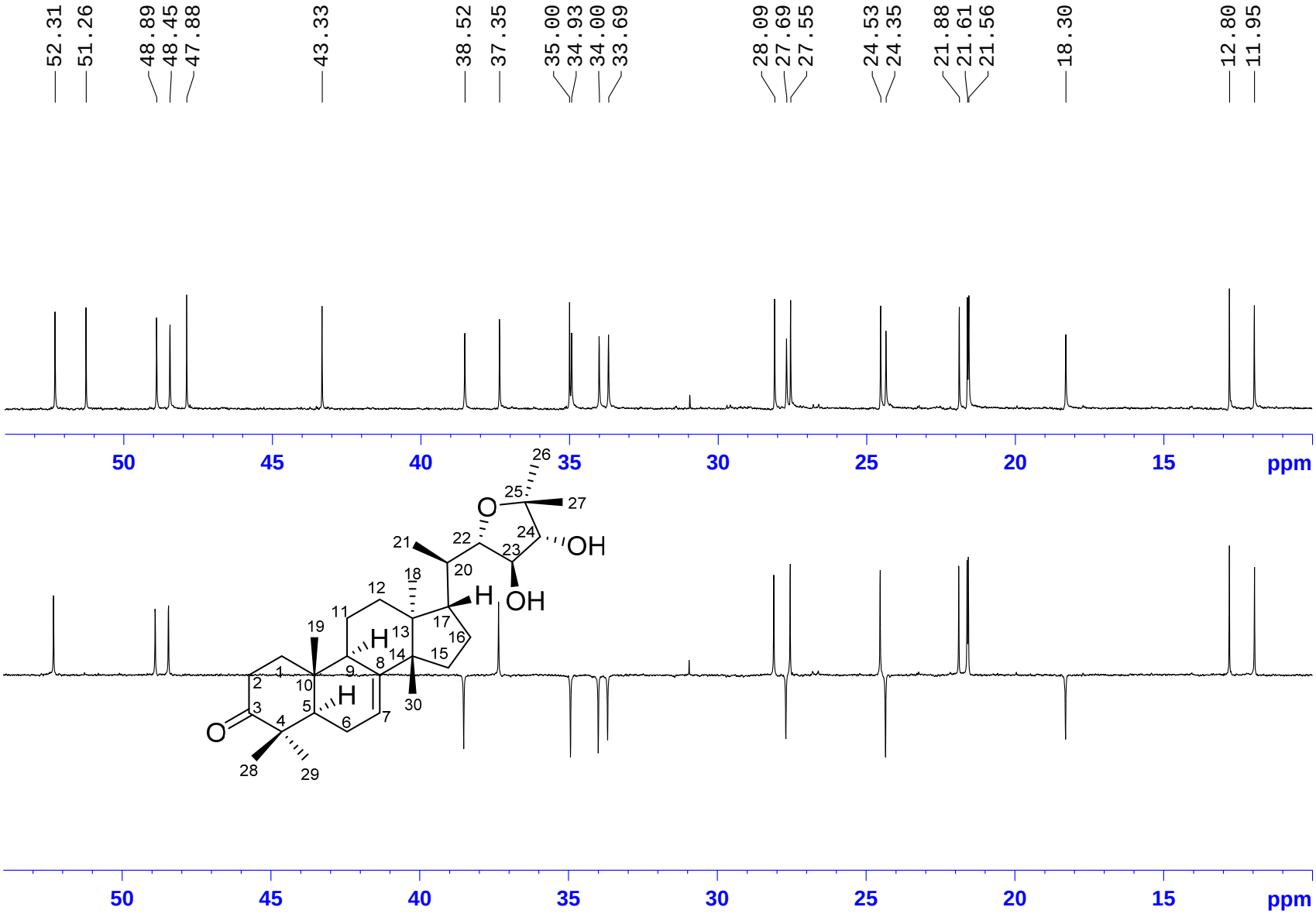


DEPT135 (100 MHz) spectrum of compound **5** in $CDCl_3$

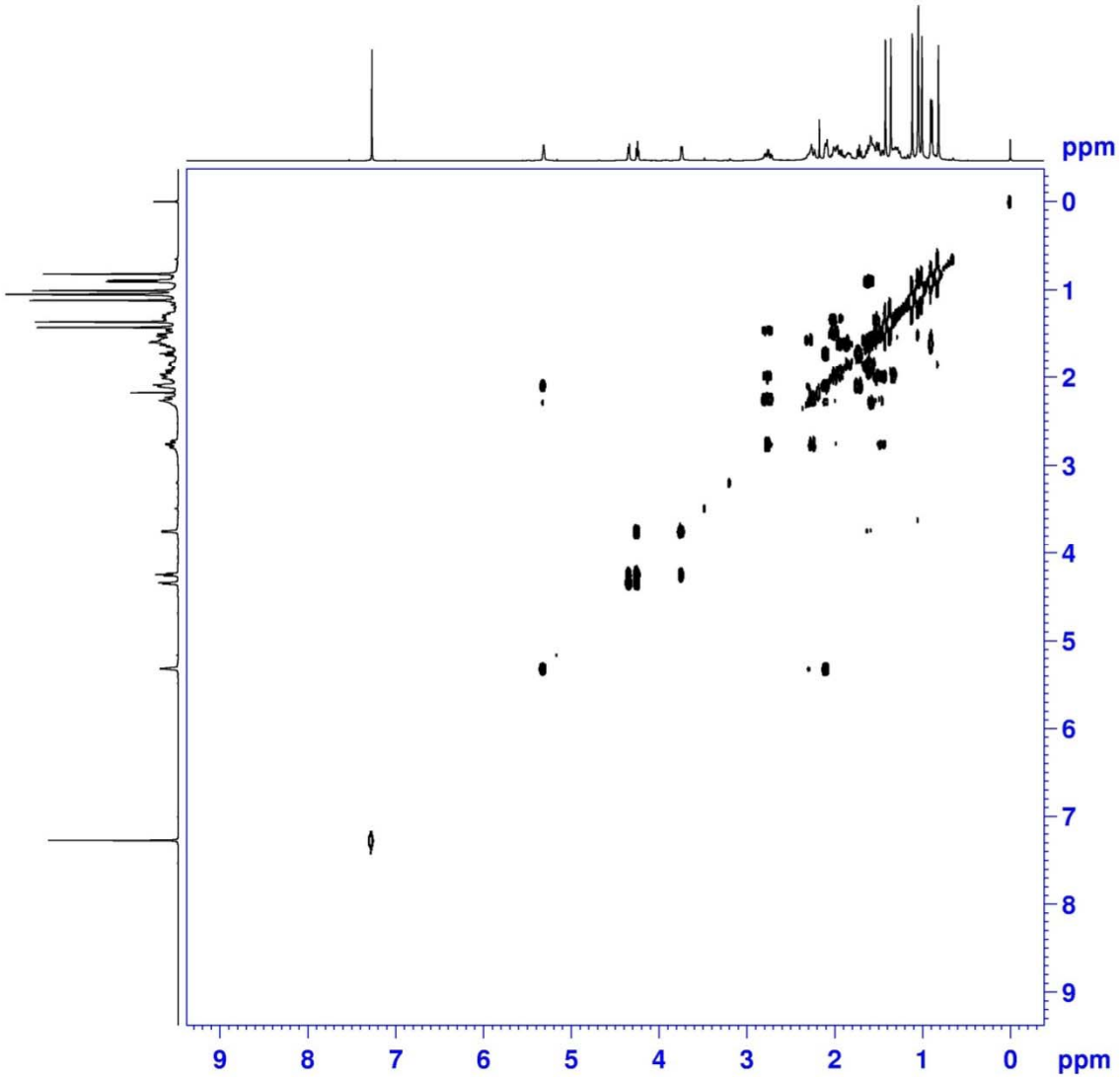




DEPT135 (100 MHz) spectrum of compound **5** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5** in $CDCl_3$

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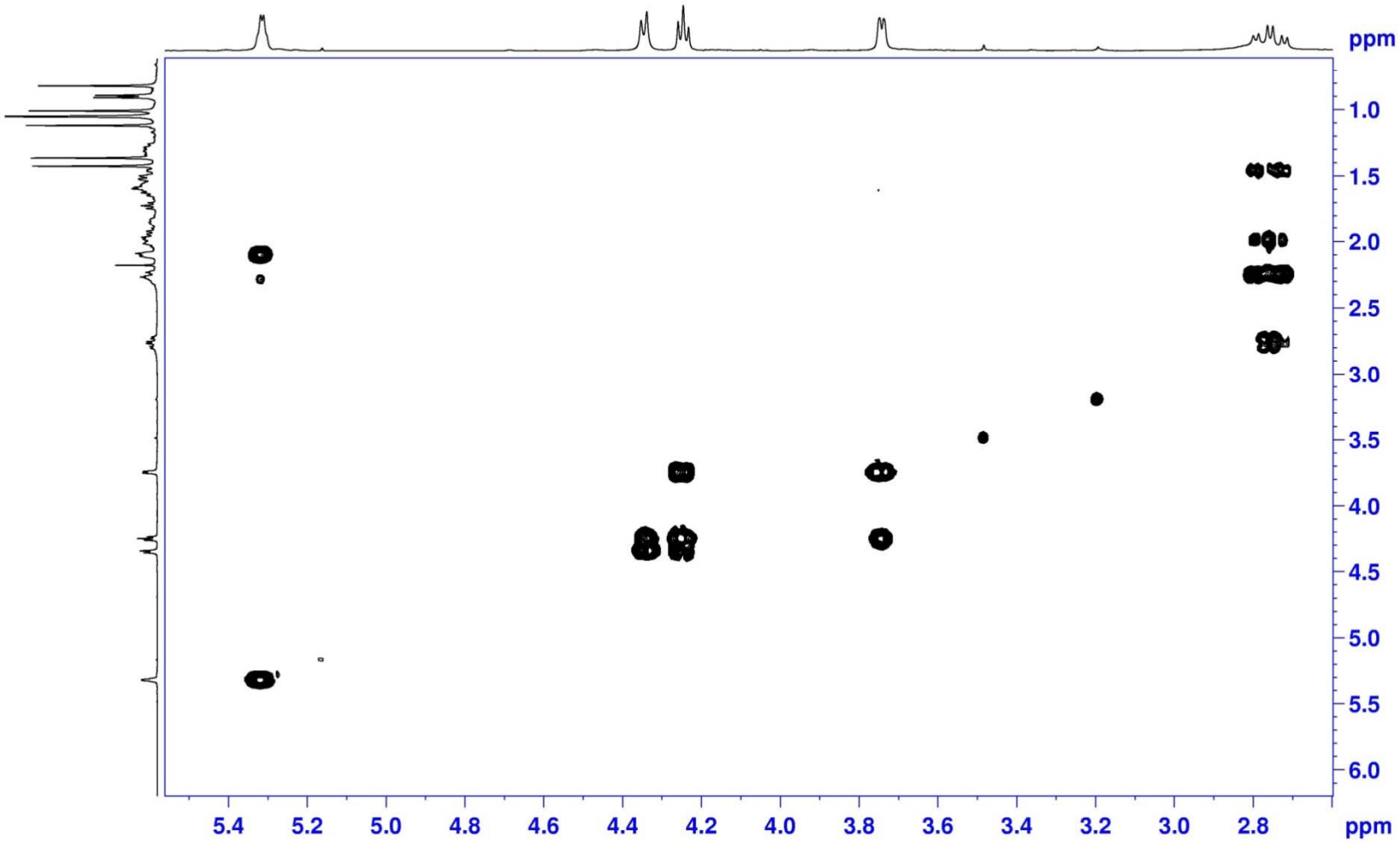
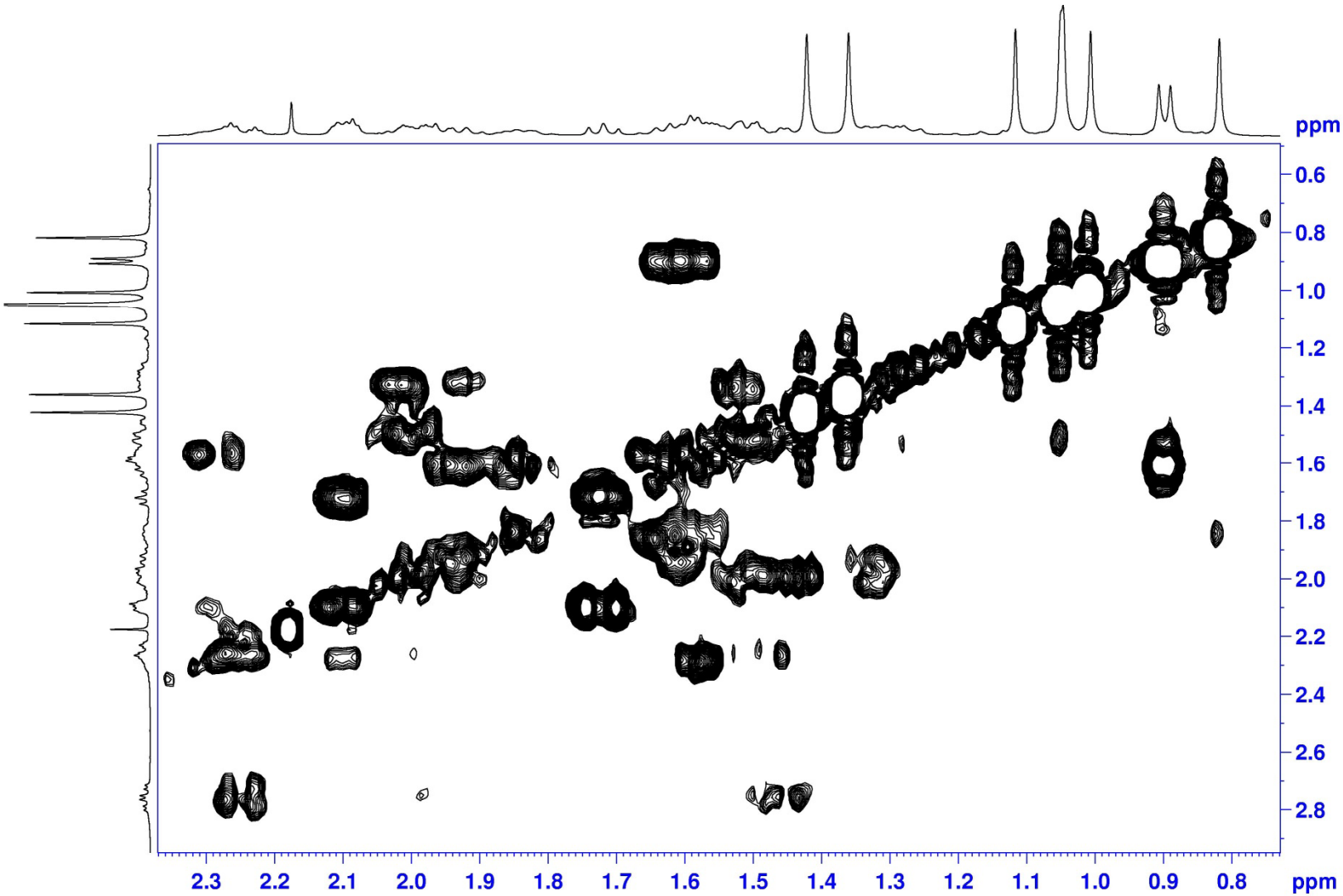
NAME                XG-52-4
EXPNO                     4
PROCNO                    1
Date_              20171226
Time                  19.41
INSTRUM               spect
PROBHD     5 mm CPPBBO BB
PULPROG        cosygpppqf
TD                     2048
SOLVENT               CDCl3
NS                        8
DS                        8
SWH                3906.250 Hz
FIDRES             1.907349 Hz
AQ                0.2621940 sec
RG                    65.23
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              400.1300009 MHz
WDW                    QSINE
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40
SI                     1024
MC2                      QF
SF              400.1300009 MHz
WDW                    QSINE
SSB                       0
LB                     0.00 Hz
GB                        0
```

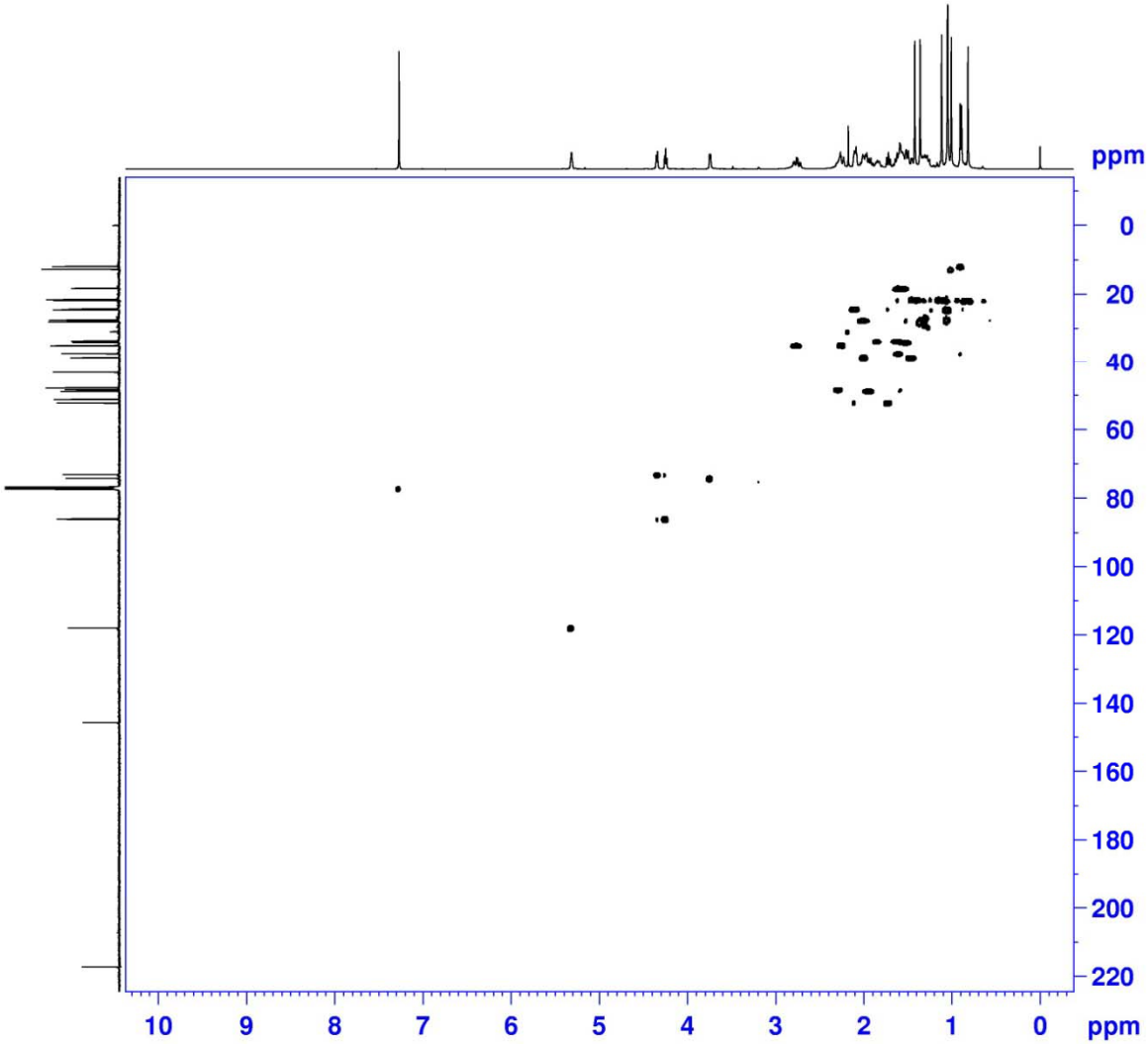
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **5** in $CDCl_3$

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NAME              XG-52-4
EXPNO                   5
PROCNO                  1
Date_            20171226
Time                20.21
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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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           400.1300014 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2        echo-antiecho
SF           100.6127577 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
```
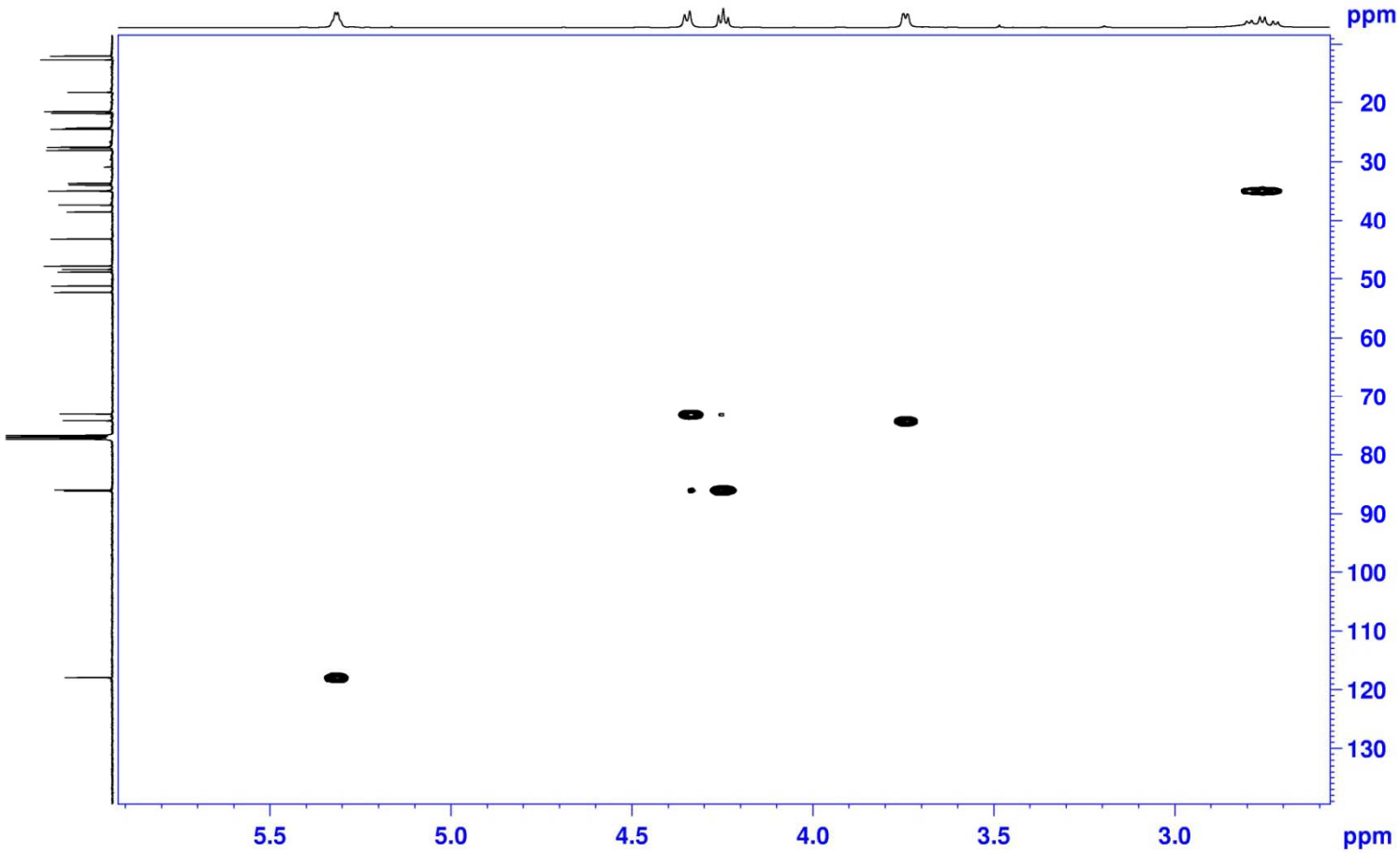
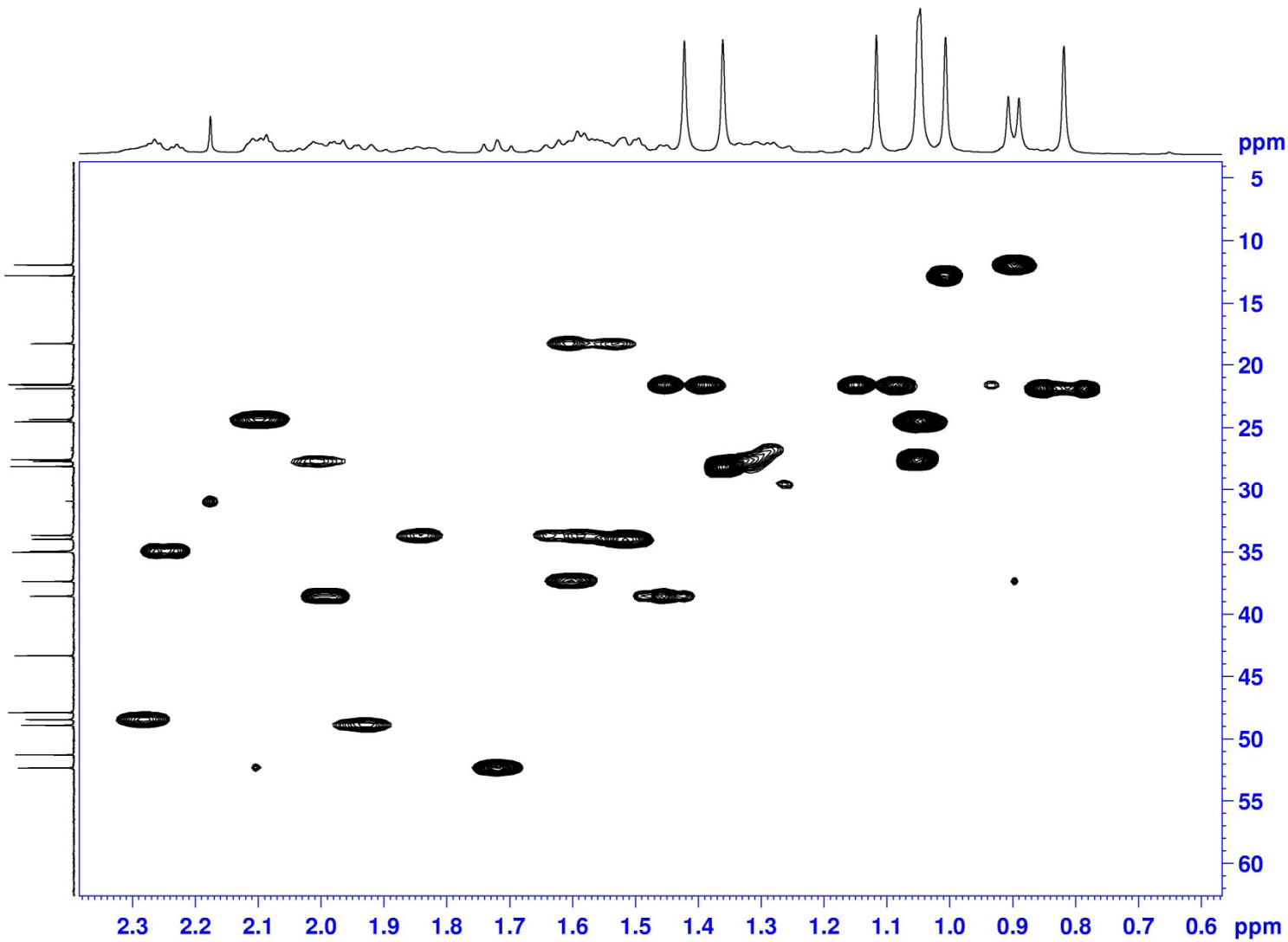
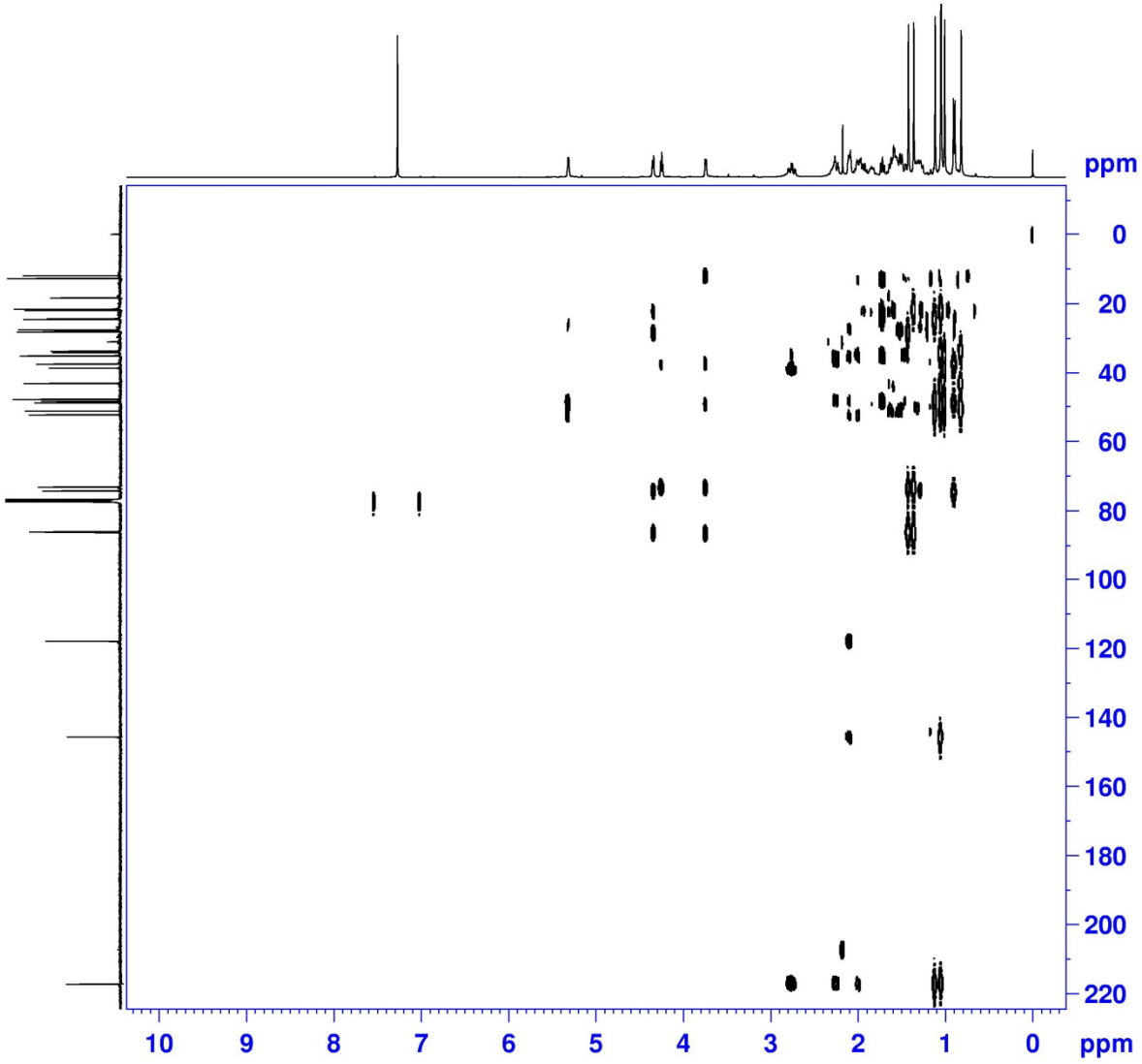

HSQC (400 MHz) spectrum of compound **5** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **5** in $CDCl_3$

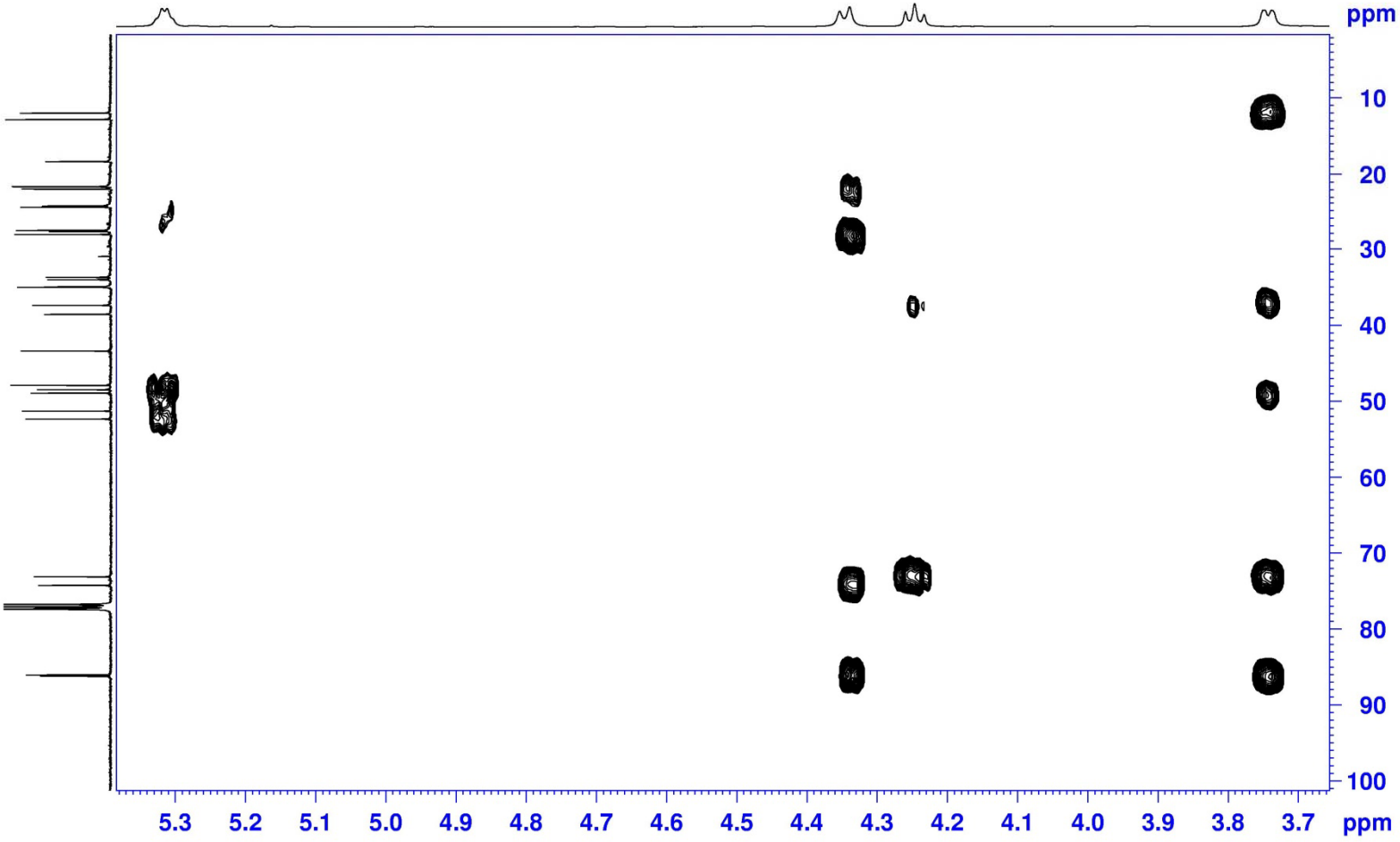


HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$

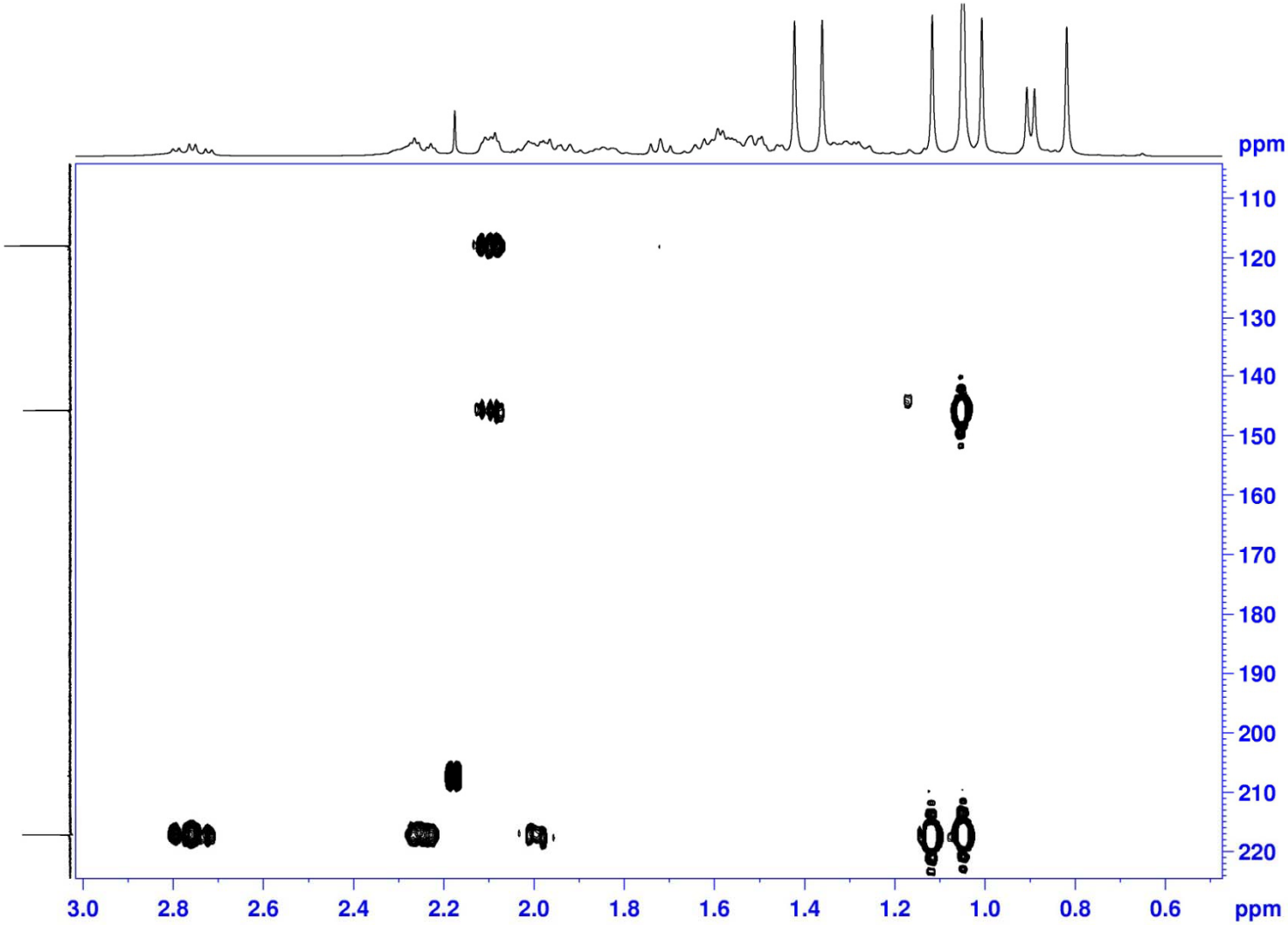



```
NAME                XG-52-4
EXPNO                     6
PROCNO                    1
Date_              20171226
Time                  22.12
INSTRUM               spect
PROBHD     5 mm CPPBBO BB
PULPROG      hmbcgplpndqf
TD                     4096
SOLVENT              CDCl3
NS                       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 ========
SFO1            400.1323208 MHz
NUC1                     1H
P1                    11.50 usec
P2                    23.00 usec
ND0                       2
TD                       61
SFO1                100.6233 MHz
FIDRES            394.073151 Hz
SW                   238.896 ppm
FnMODE                   QF
SI                     2048
SF              400.1300015 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40
SI                     1024
MC2                      QF
SF              100.6127590 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
```

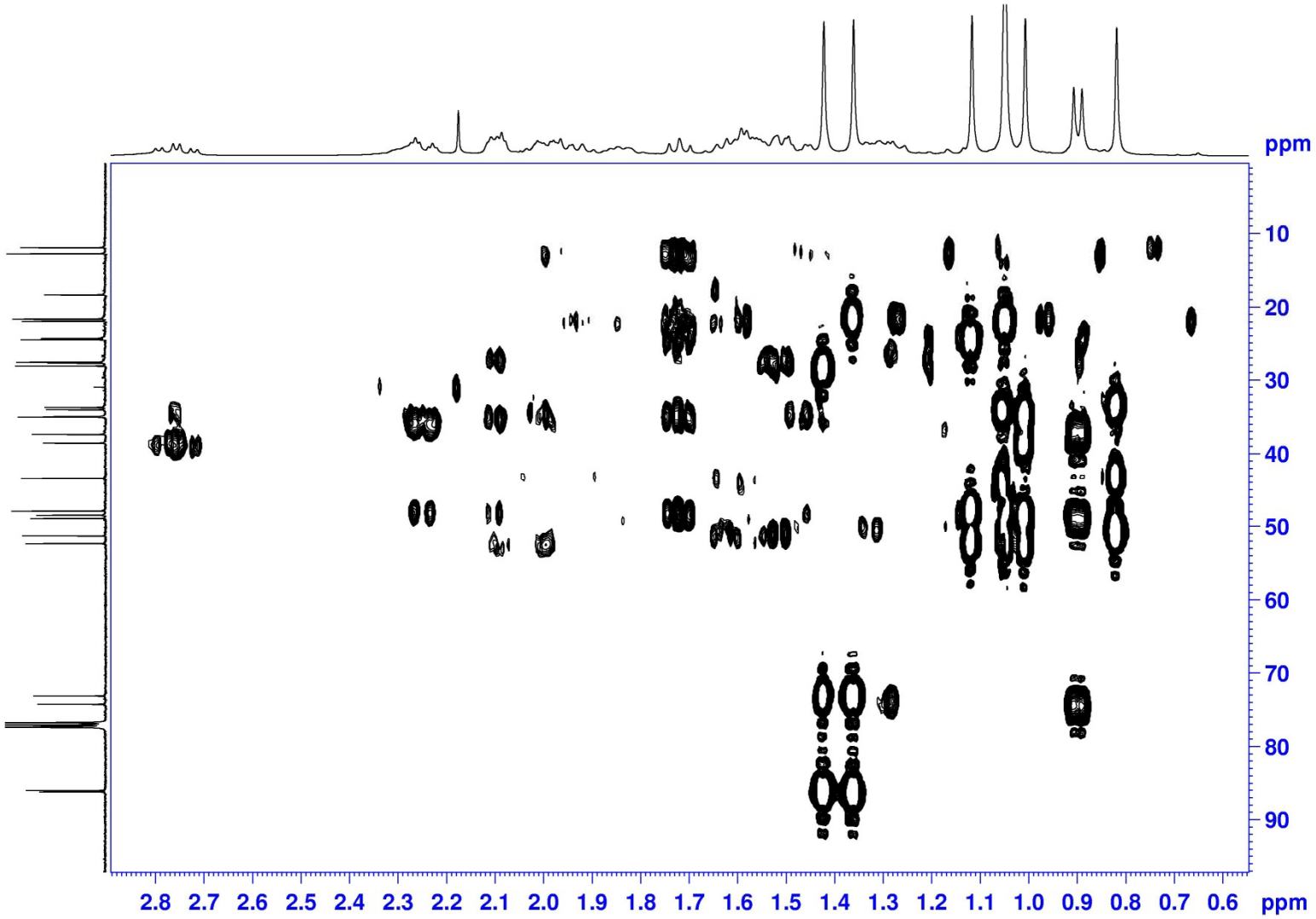
HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$



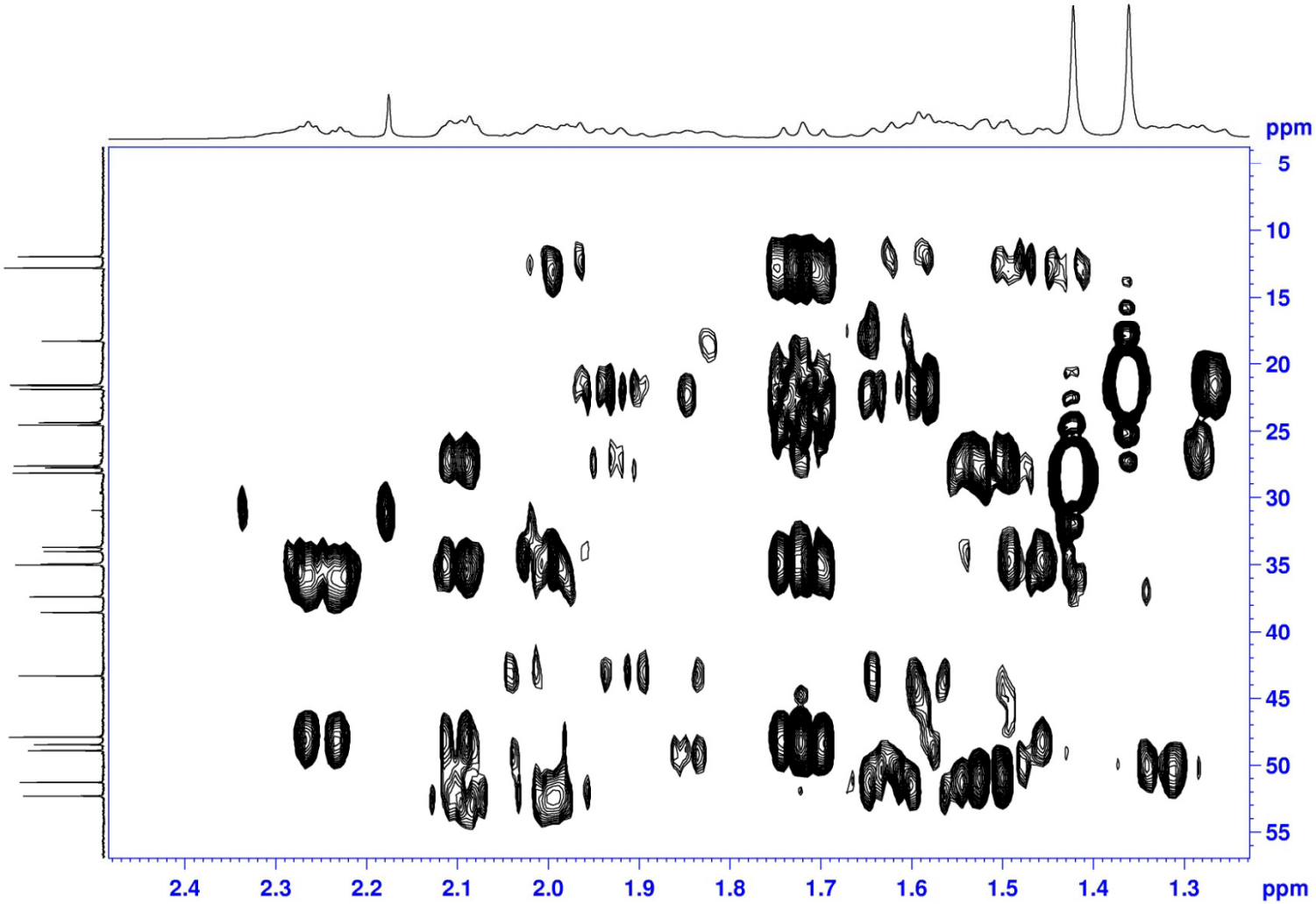
HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$



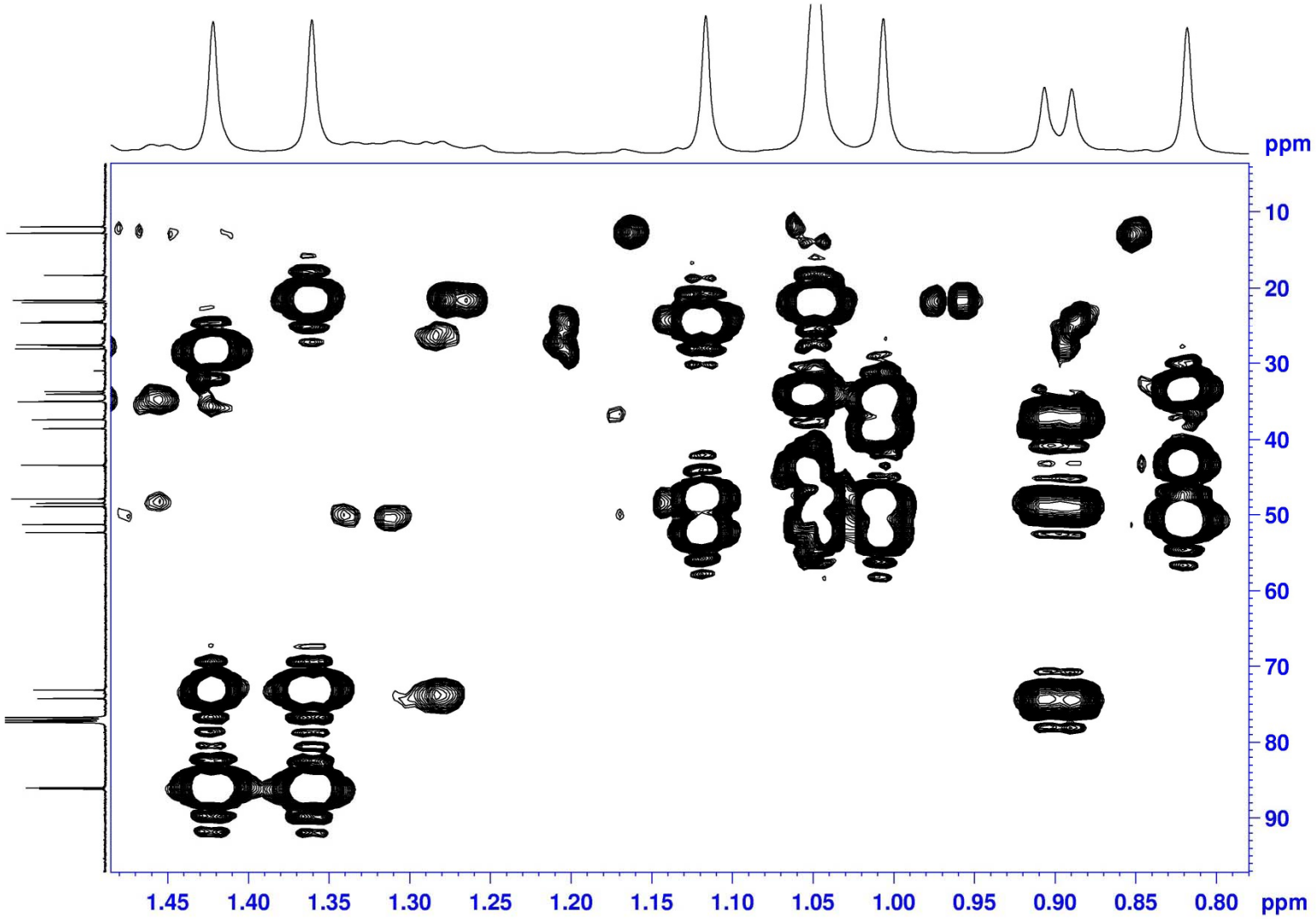
HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$



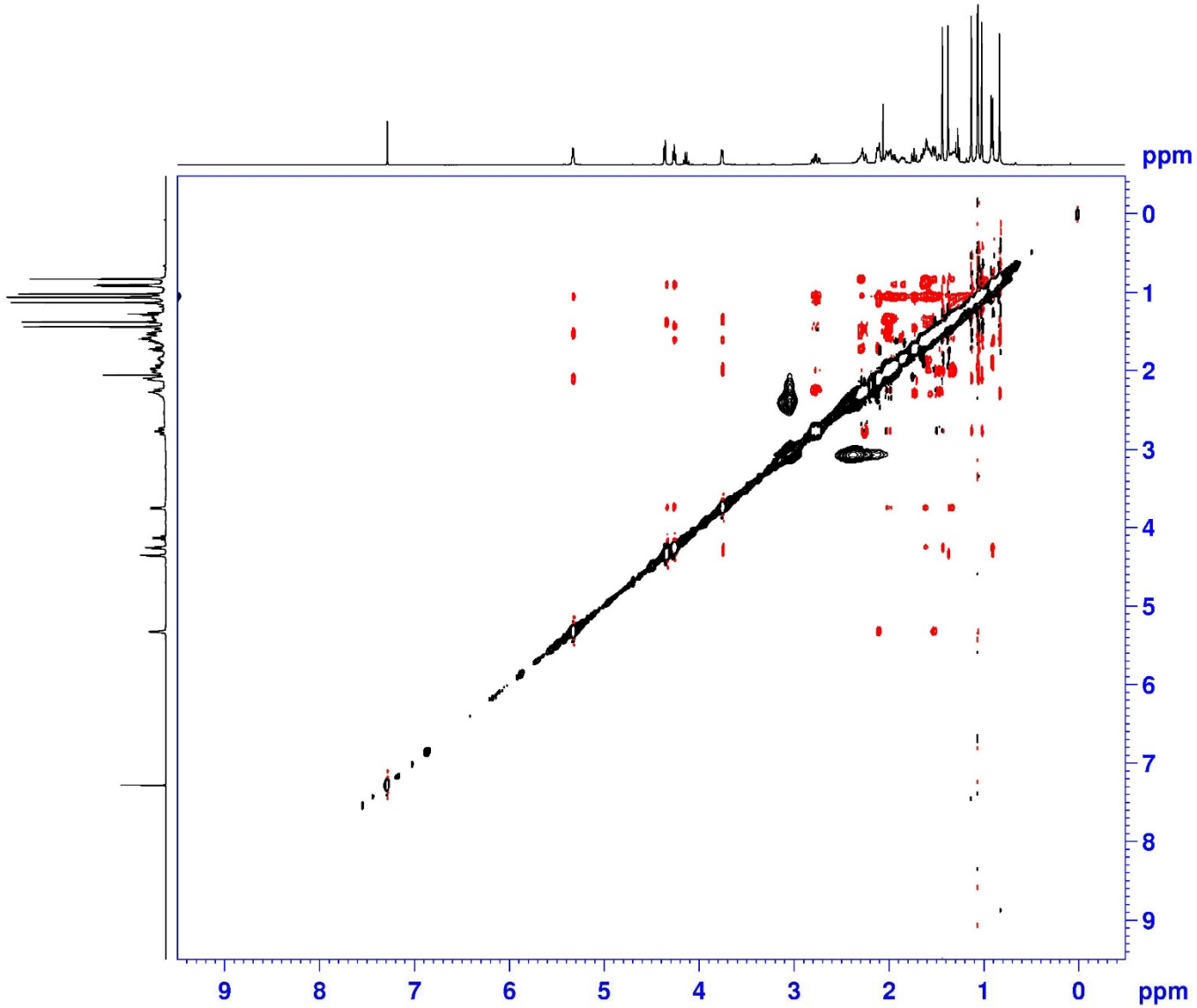
HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **5** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **5** in $CDCl_3$




```
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EXPNO                  7
PROCNO                 1
Date_           20171228
Time                5.30
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG     noesygpphpp
TD                  2048
SOLVENT            CDCl3
NS                     16
DS                     32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ            0.2560500 sec
RG                65.23
DW               125.000 use
DE                 10.00 use
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 use
P2                 23.00 use
P17             2500.00 use
ND0                    1
TD                   256
SFO1           400.1318 MHz
FIDRES         15.625000 Hz
SW                 9.997 ppm
FnMODE       States-TPPI
SI                  1024
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00
SI                  1024
MC2          States-TPPI
SF           400.1300000 MHz
WDW                QSINE
SSB                    2
```
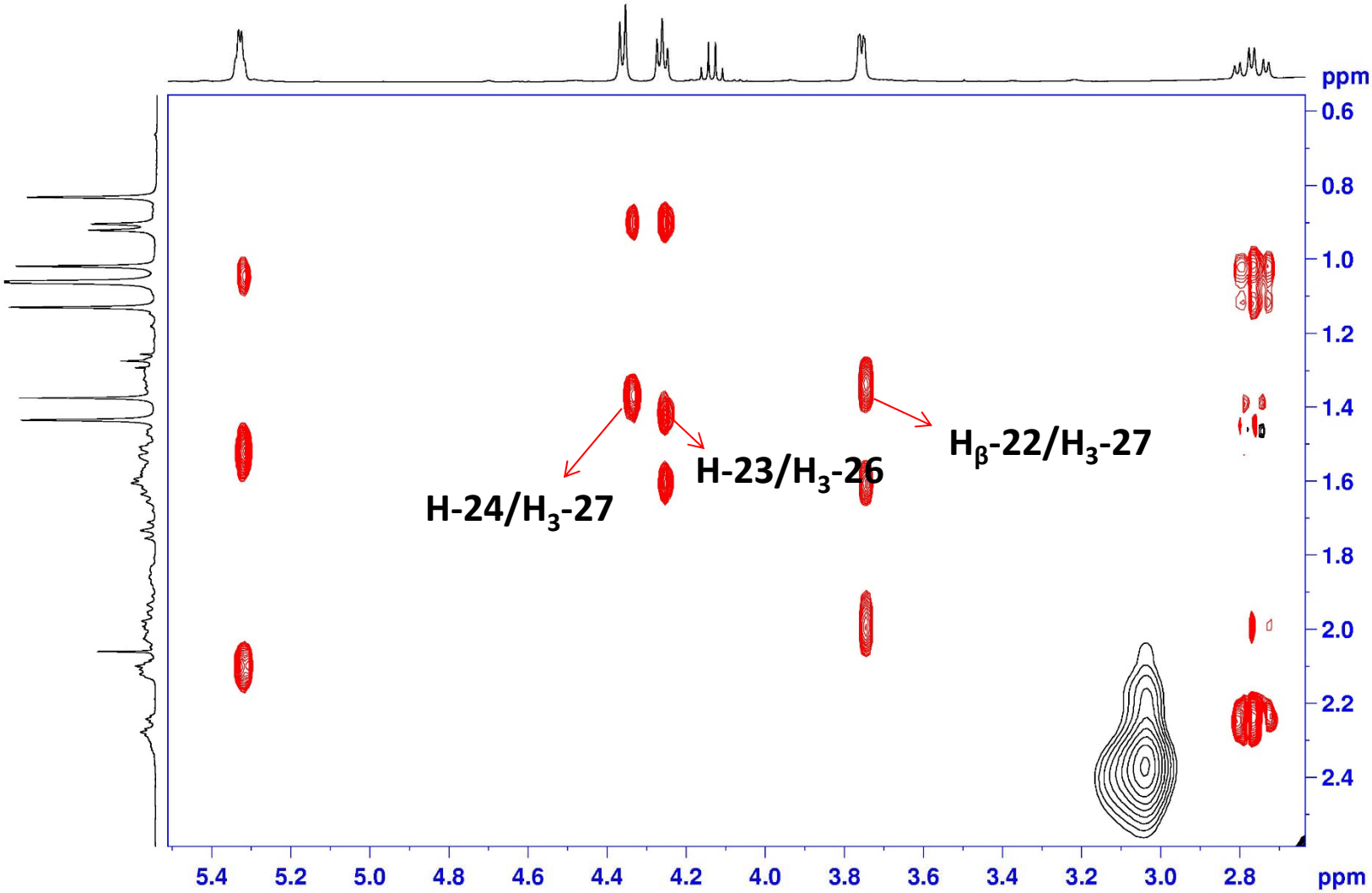
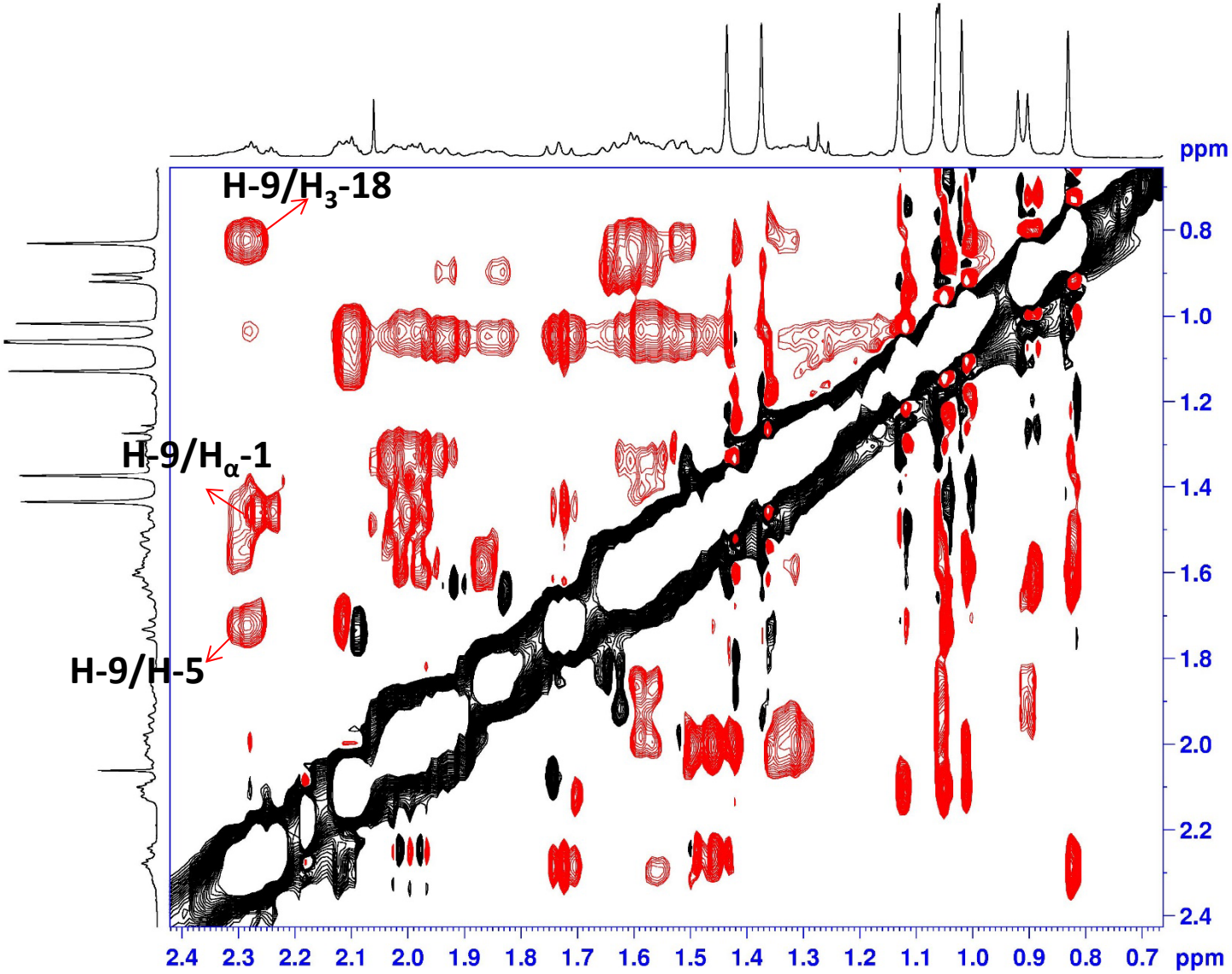
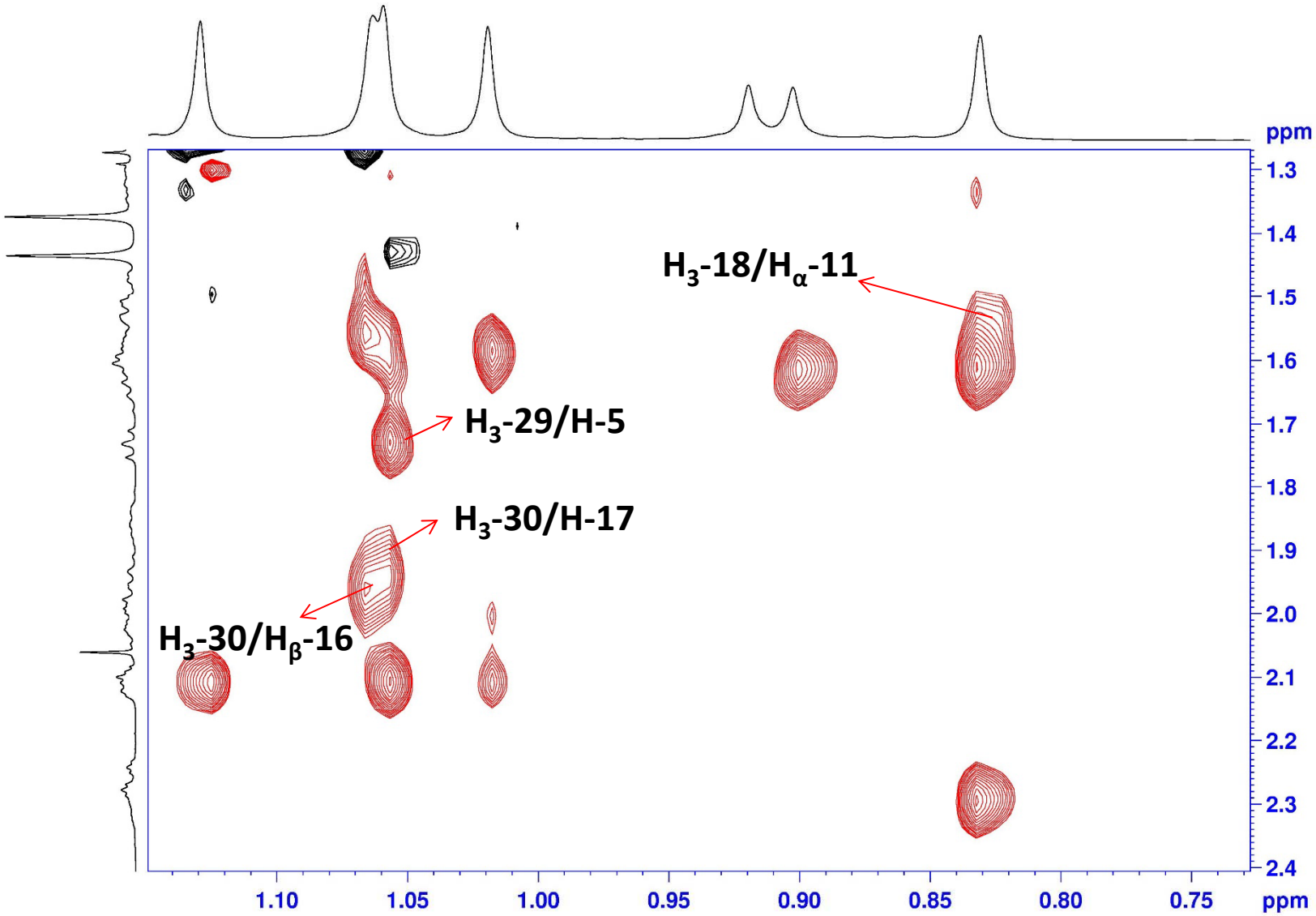

NOESY (400 MHz) spectrum of compound **5** in $CDCl_3$



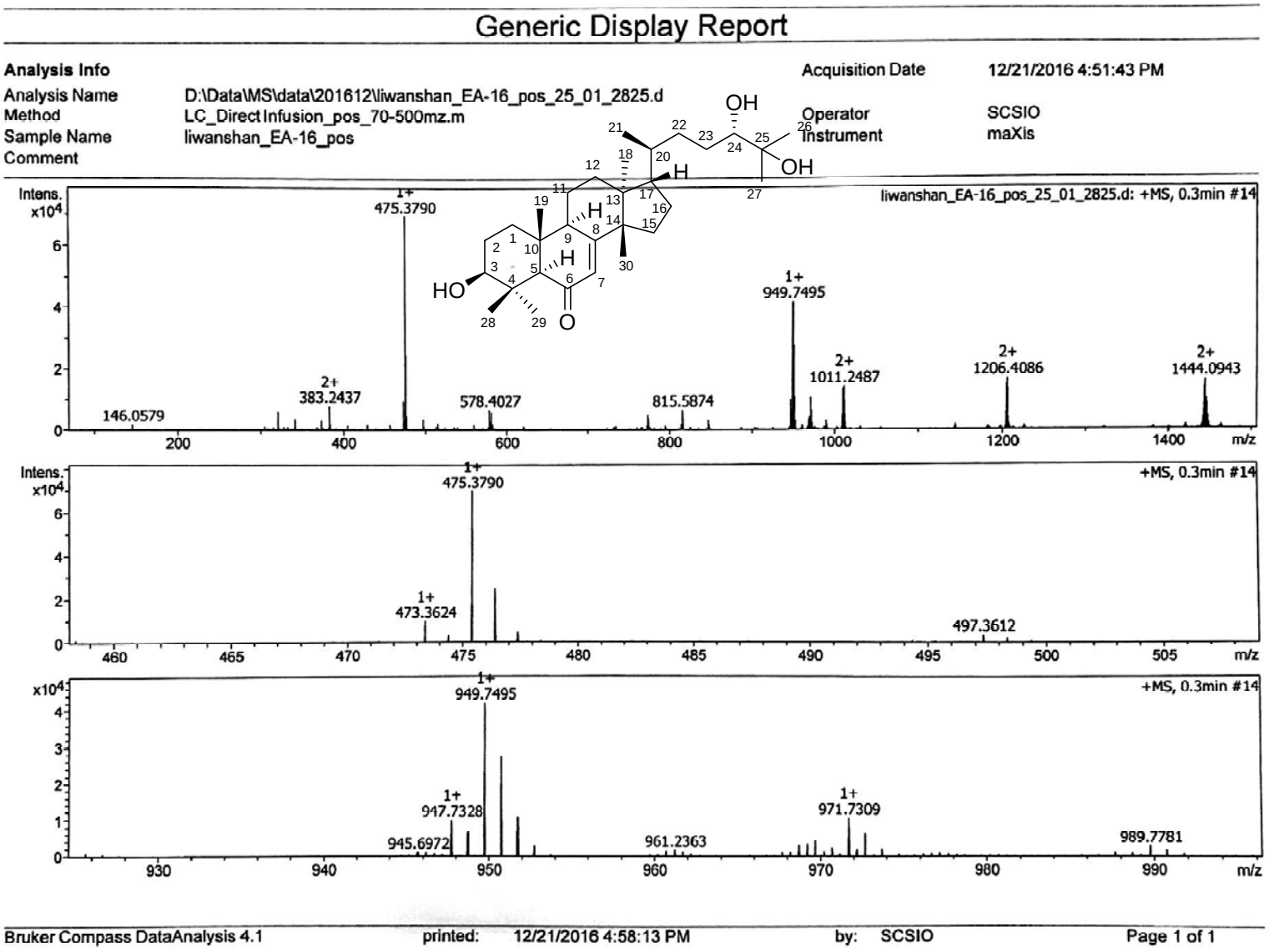
NOESY (400 MHz) spectrum of compound **5** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **5** in $CDCl_3$



HR-ESIMS for compound **6**



HR-ESIMS for compound **6**

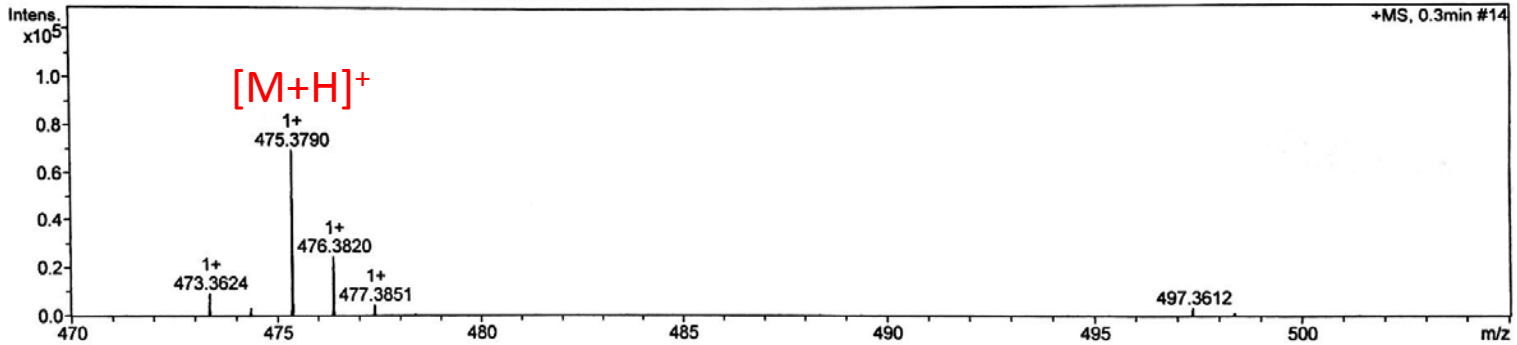
Mass Spectrum SmartFormula Report

Analysis Info

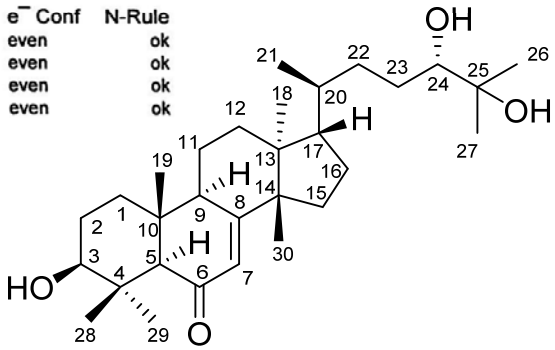
Analysis Name	D:\Data\MS\data\201612\liwanshan_EA-16_pos_25_01_2825.d	Acquisition Date	12/21/2016 4:51:43 PM
Method	LC_Direct Infusion_pos_70-500mz.m	Operator	SCSIO
Sample Name	liwanshan_EA-16_pos	Instrument	maXis 255552.00029
Comment			

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	70 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



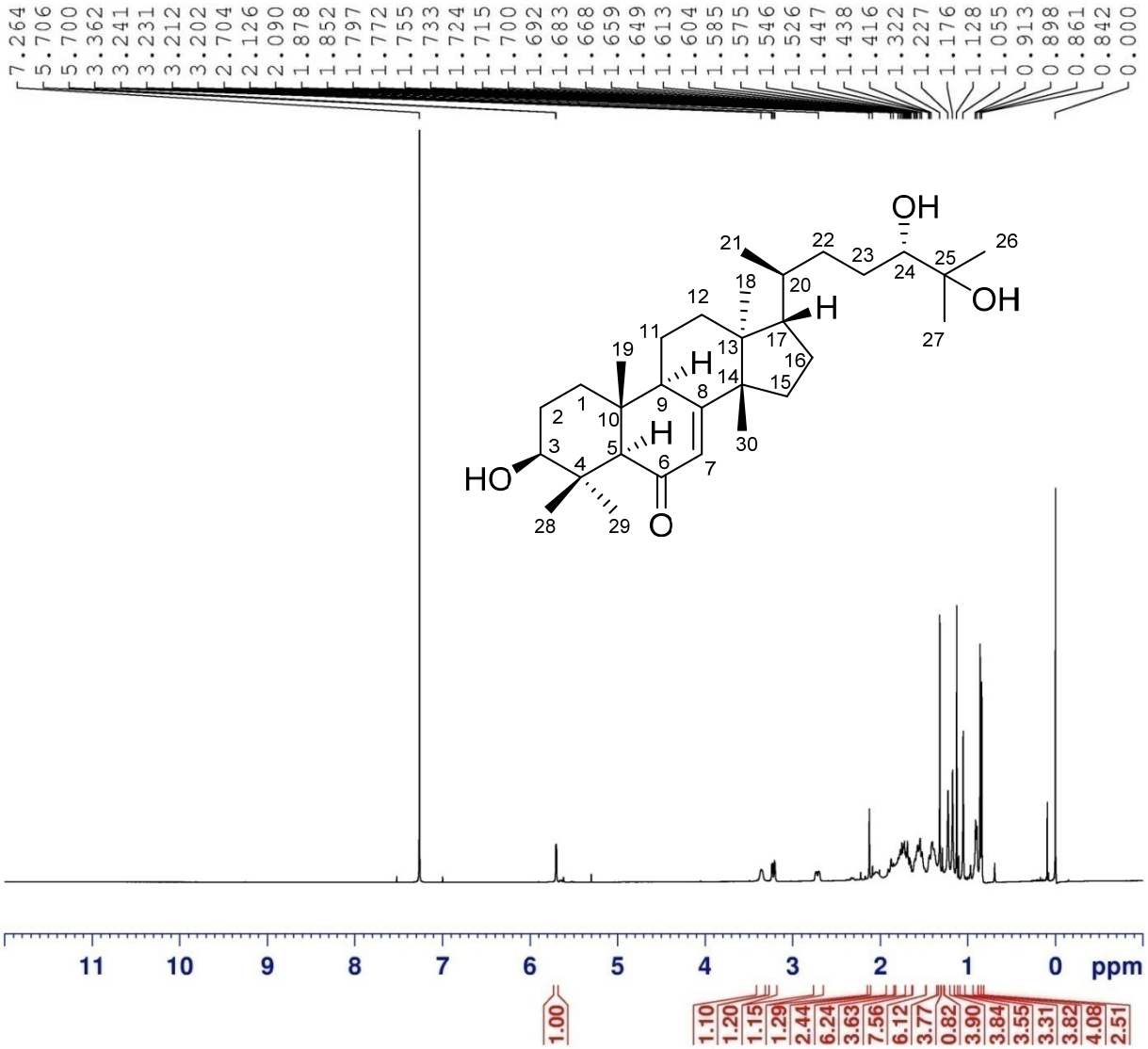
Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
475.379013	1	C30H51O4	100.00	475.378187	-1.7	-0.8	10.7	5.5	even	ok
497.361200	1	C30H50NaO4	100.00	497.360131	2.2	1.1	29.1	5.5	even	ok
949.749455	1	C60H101O8	100.00	949.749097	-0.4	-0.4	12.2	10.5	even	ok
971.730887	1	C60H100NaO8	100.00	971.731041	-0.2	-0.2	30.2	10.5	even	ok



liwanshan_EA-16_pos_25_01_2825.d

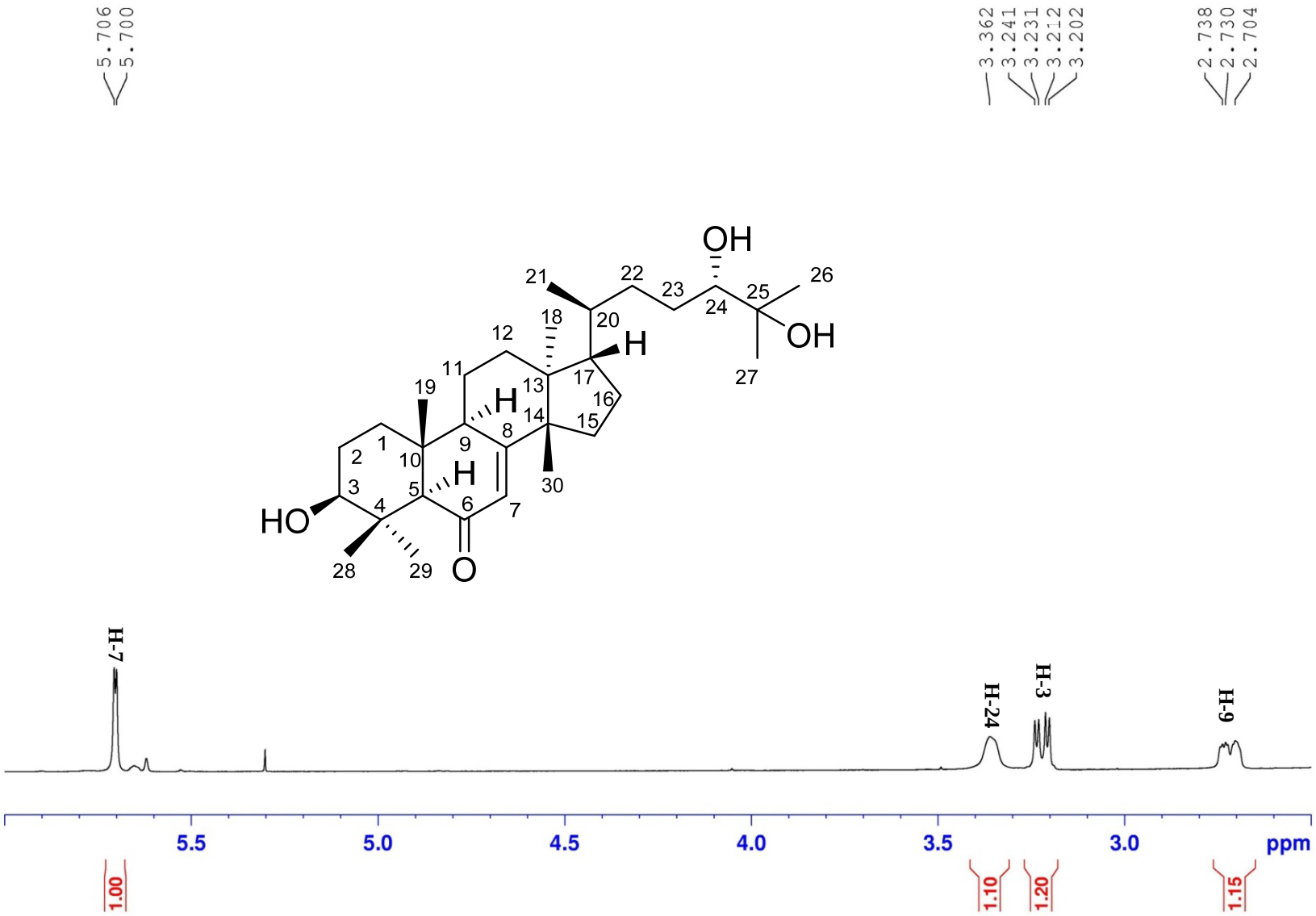
Bruker Compass DataAnalysis 4.1 printed: 12/21/2016 4:57:45 PM by: SCSIO Page 1 of 1

^{1}H NMR (400 MHz) spectrum of compound **6** in $CDCl_3$

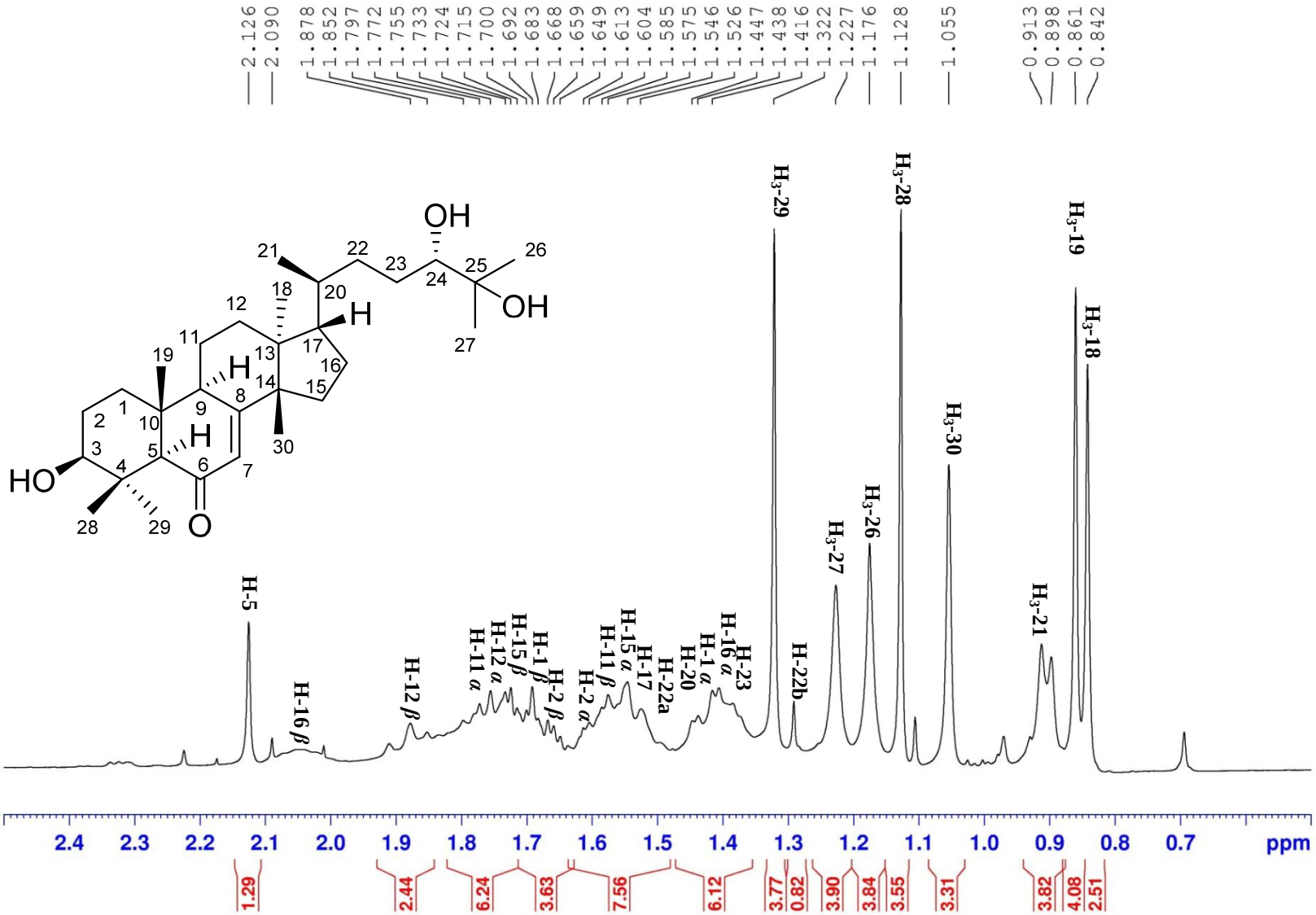



```
NAME                EA-16
EXPNO                   1
PROCNO                  1
Date_            20160726
Time                12.48
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                  102.3
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                 11.50 usec
SI                 65536
SF           400.1300079 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

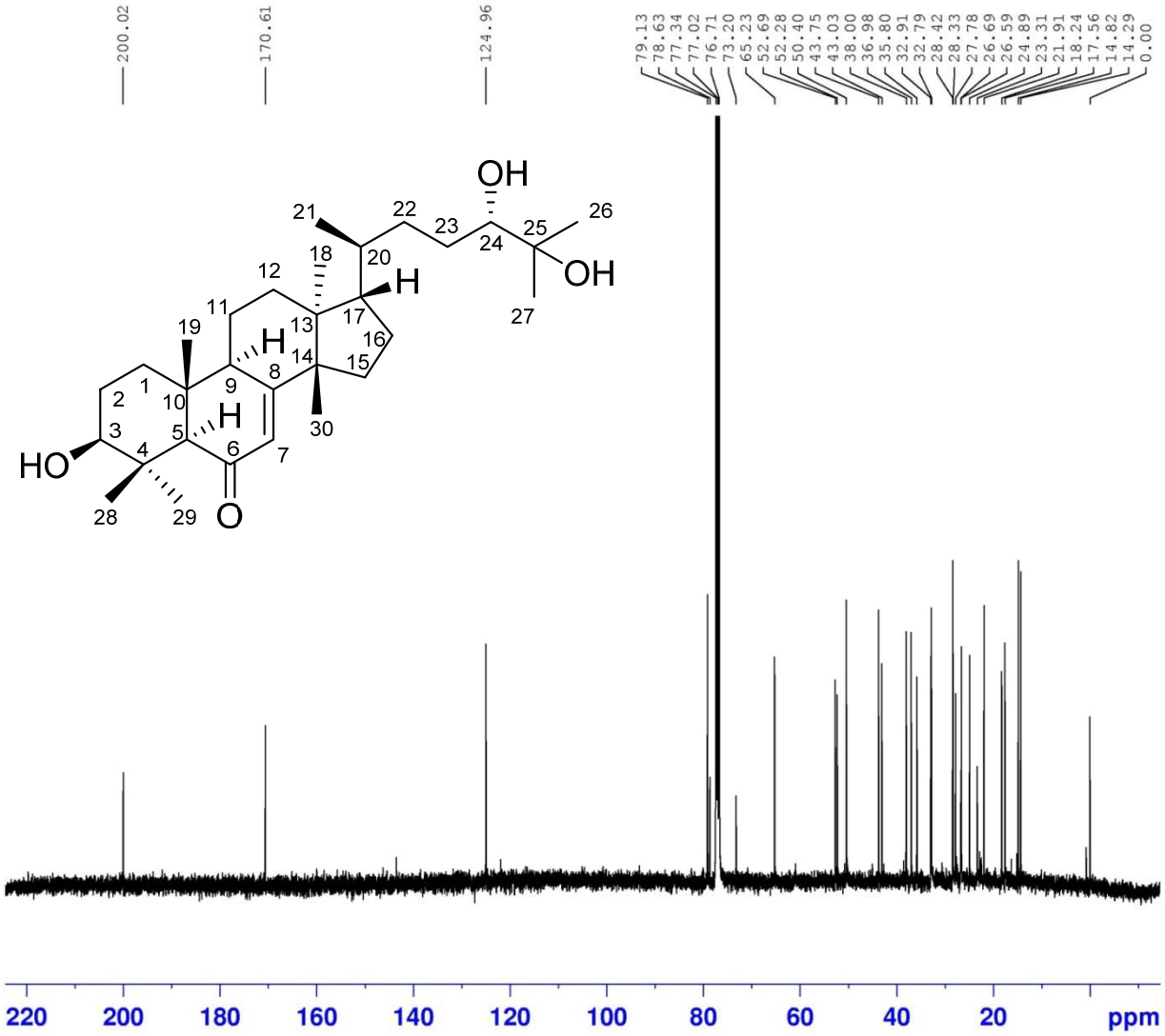
^{1}H NMR (400 MHz) spectrum of compound **6** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **6** in $CDCl_3$

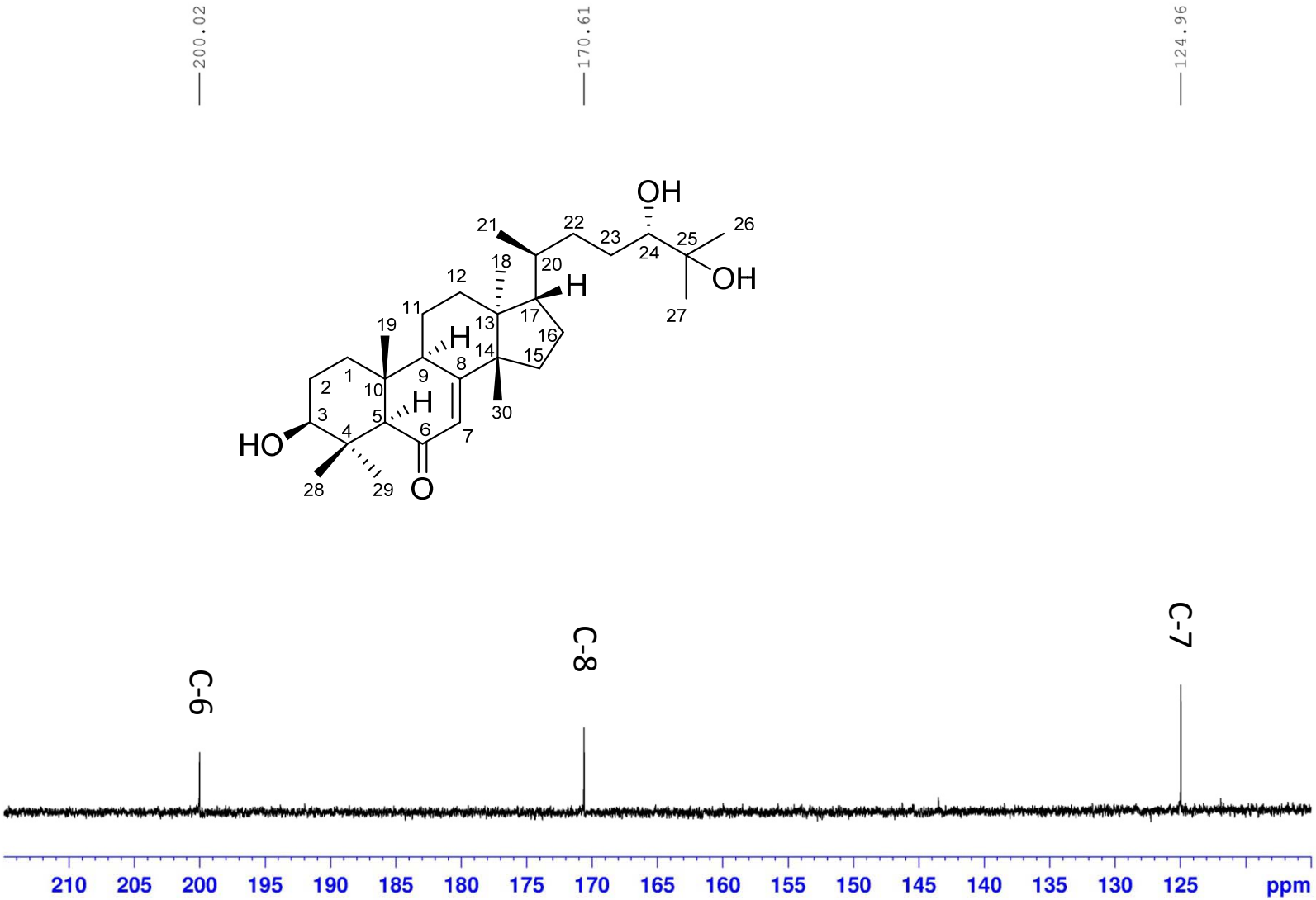


^{13}C NMR (100 MHz) spectrum of compound **6** in $CDCl_3$

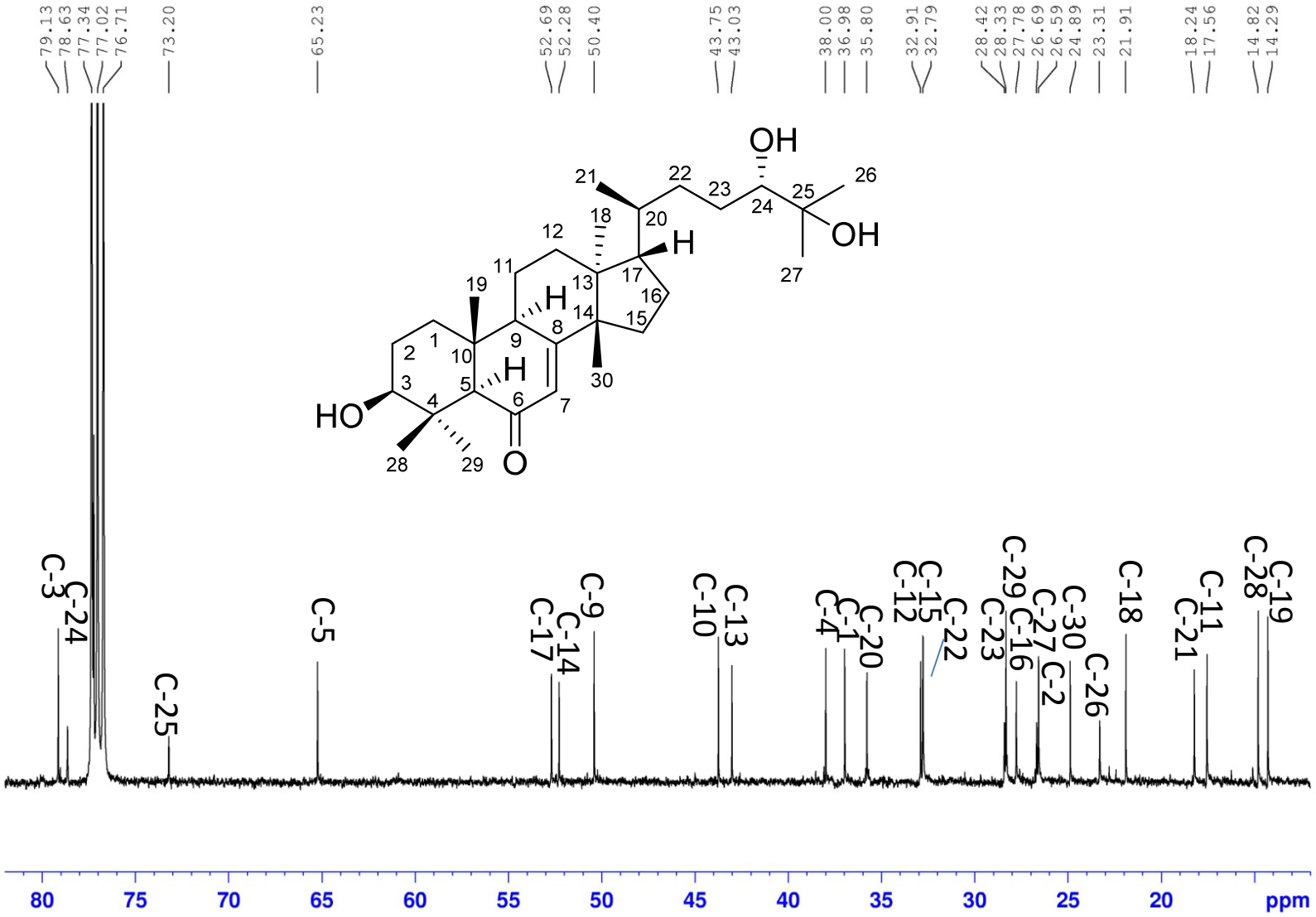



```
NAME                EA-16
EXPNO                 102
PROCNO                  1
Date_            20160726
Time                23.34
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                 102.3
DW                20.800 usec
DE                18.00 usec
TE                297.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

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

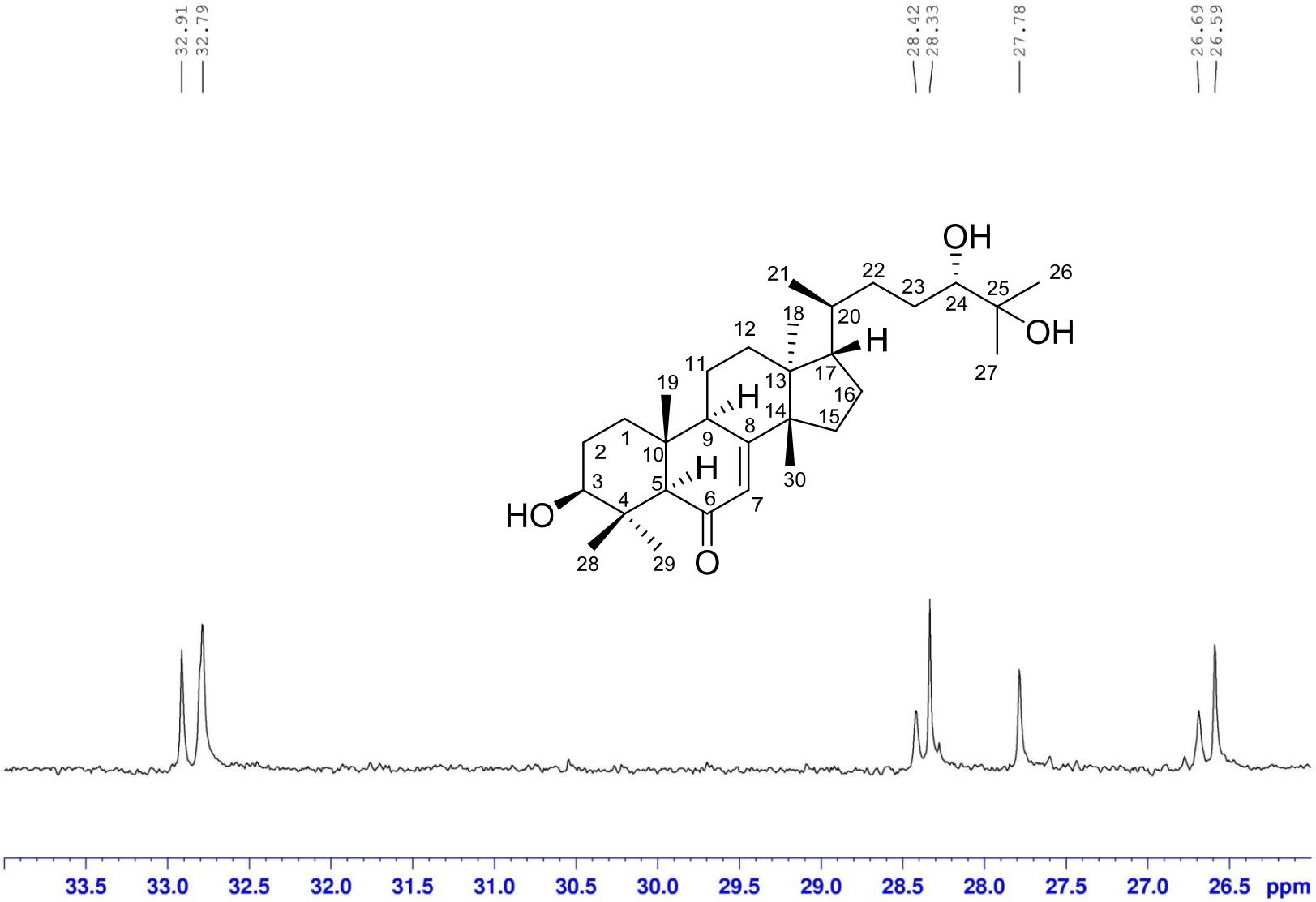
^{13}C NMR (100 MHz) spectrum of compound **6** in $CDCl_3$



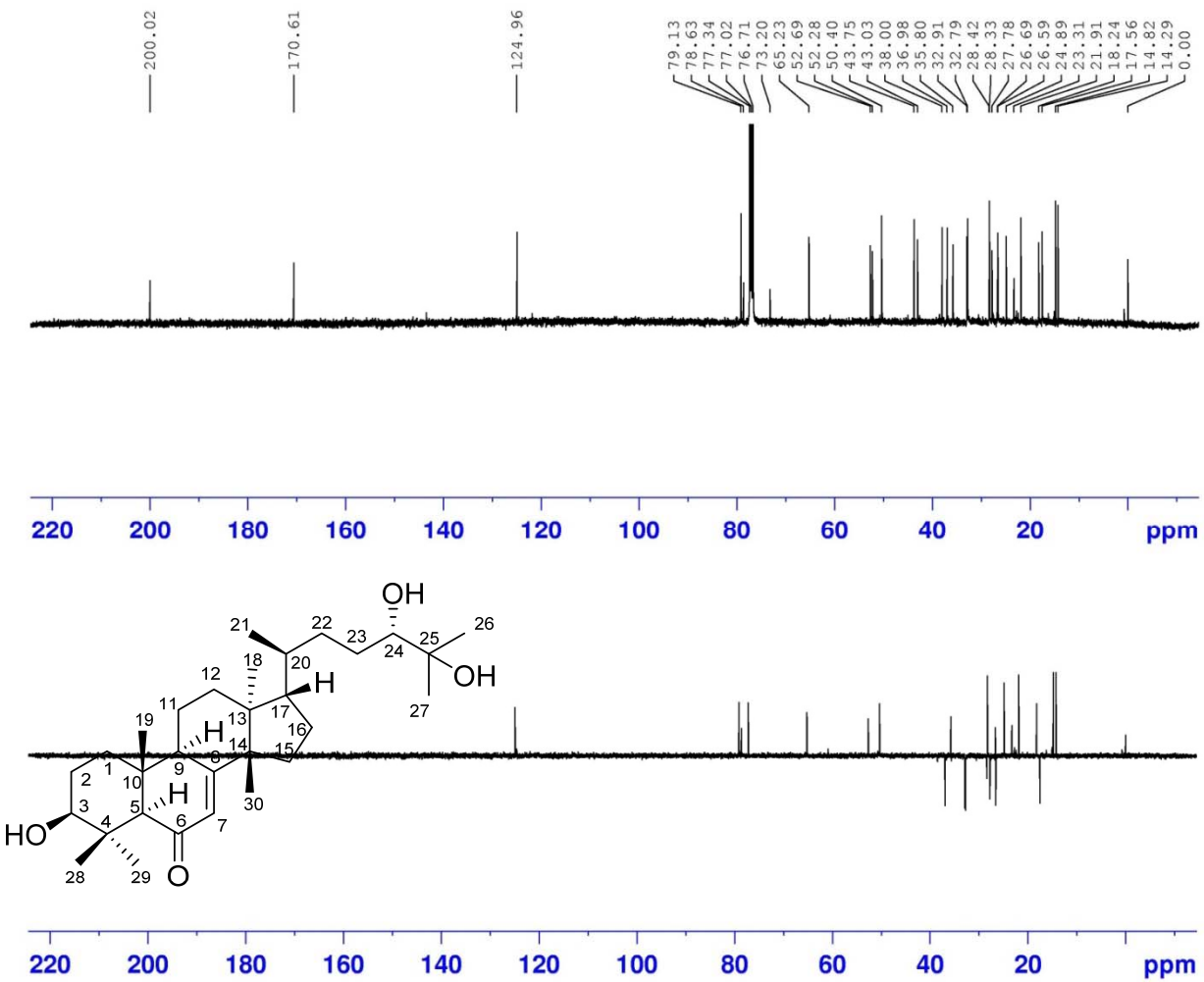
^{13}C NMR (100 MHz) spectrum of compound **6** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **6** in $CDCl_3$

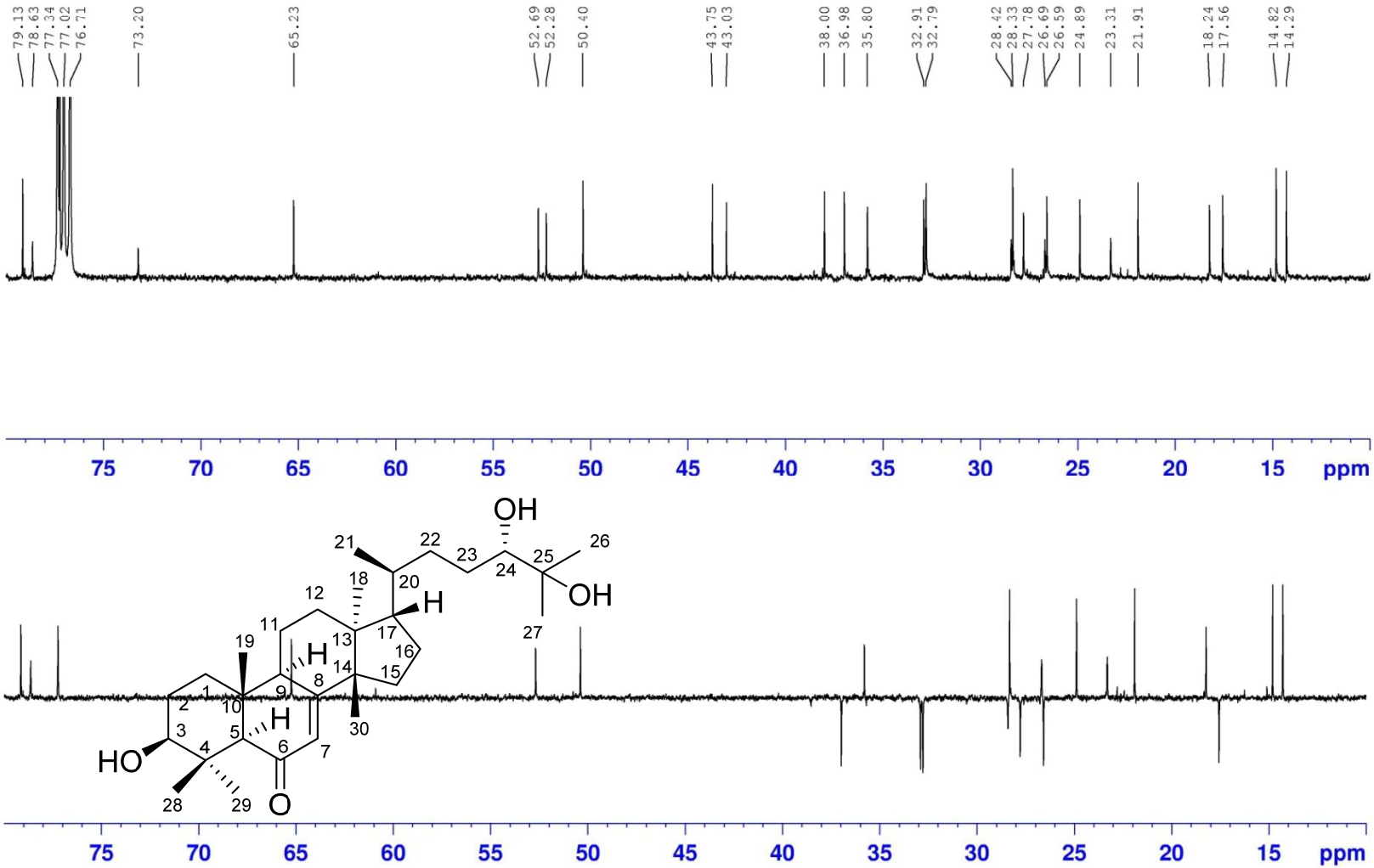


DEPT135 (100 MHz) spectrum of compound **6** in $CDCl_3$

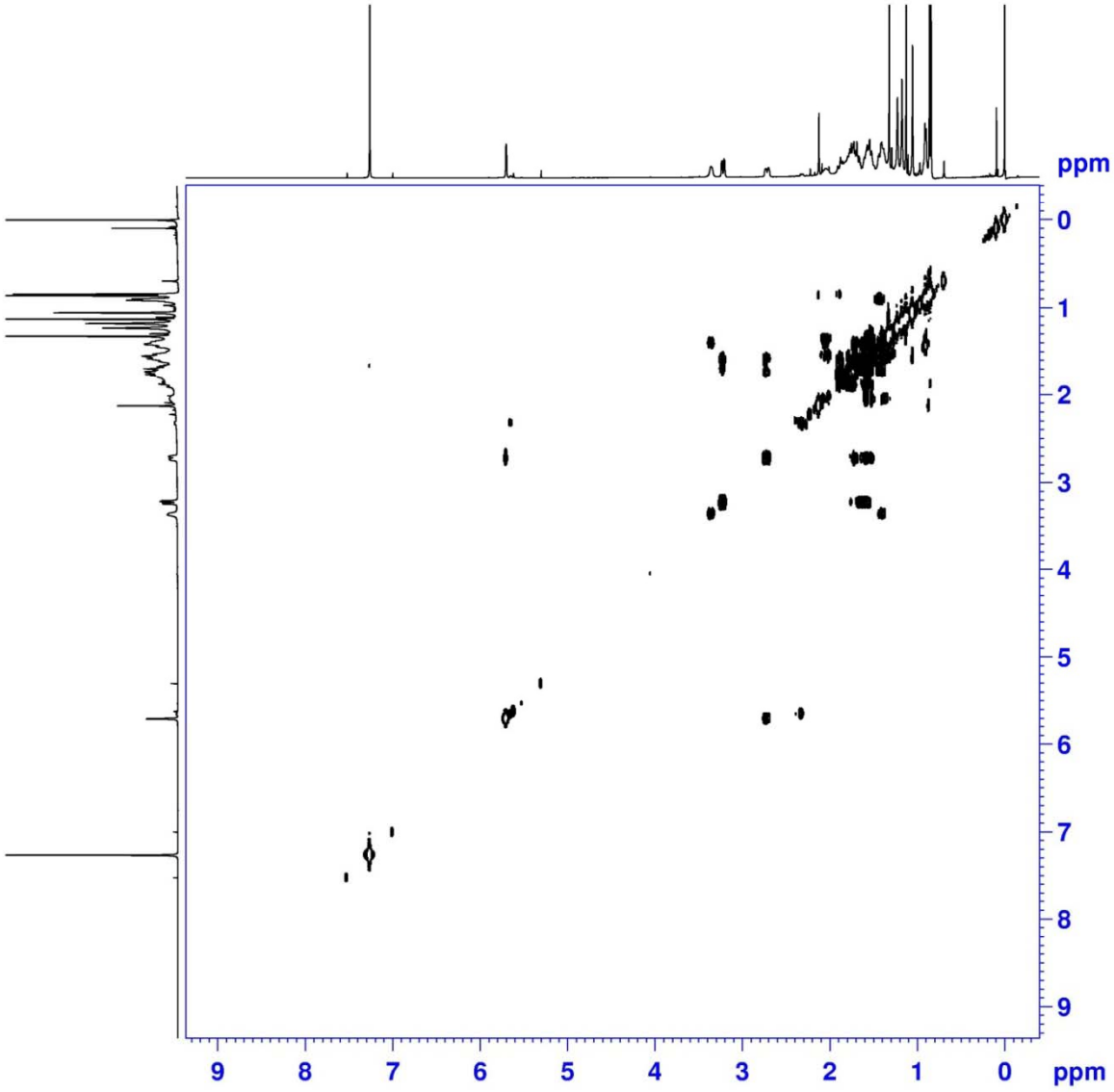



```
NAME                EA-16
EXPNO                 103
PROCNO                  1
Date_            20160726
Time                23.52
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG        deptsp135
TD                  65536
SOLVENT             CDCl3
NS                    300
DS                      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 ========
SFO1         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 **6** in $CDCl_3$

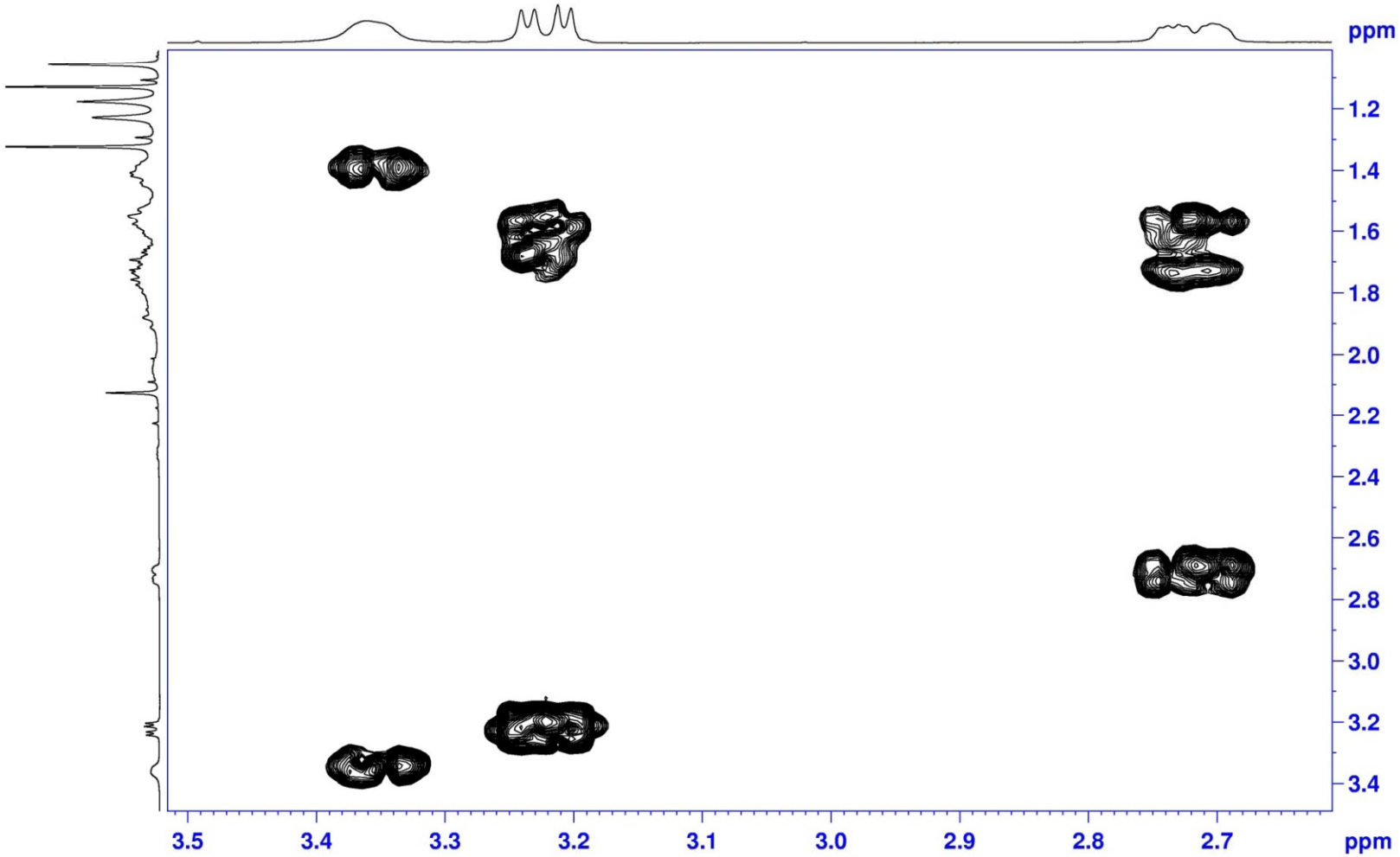


^{1}H-^{1}H COSY (400 MHz) spectrum of compound **6** in $CDCl_3$

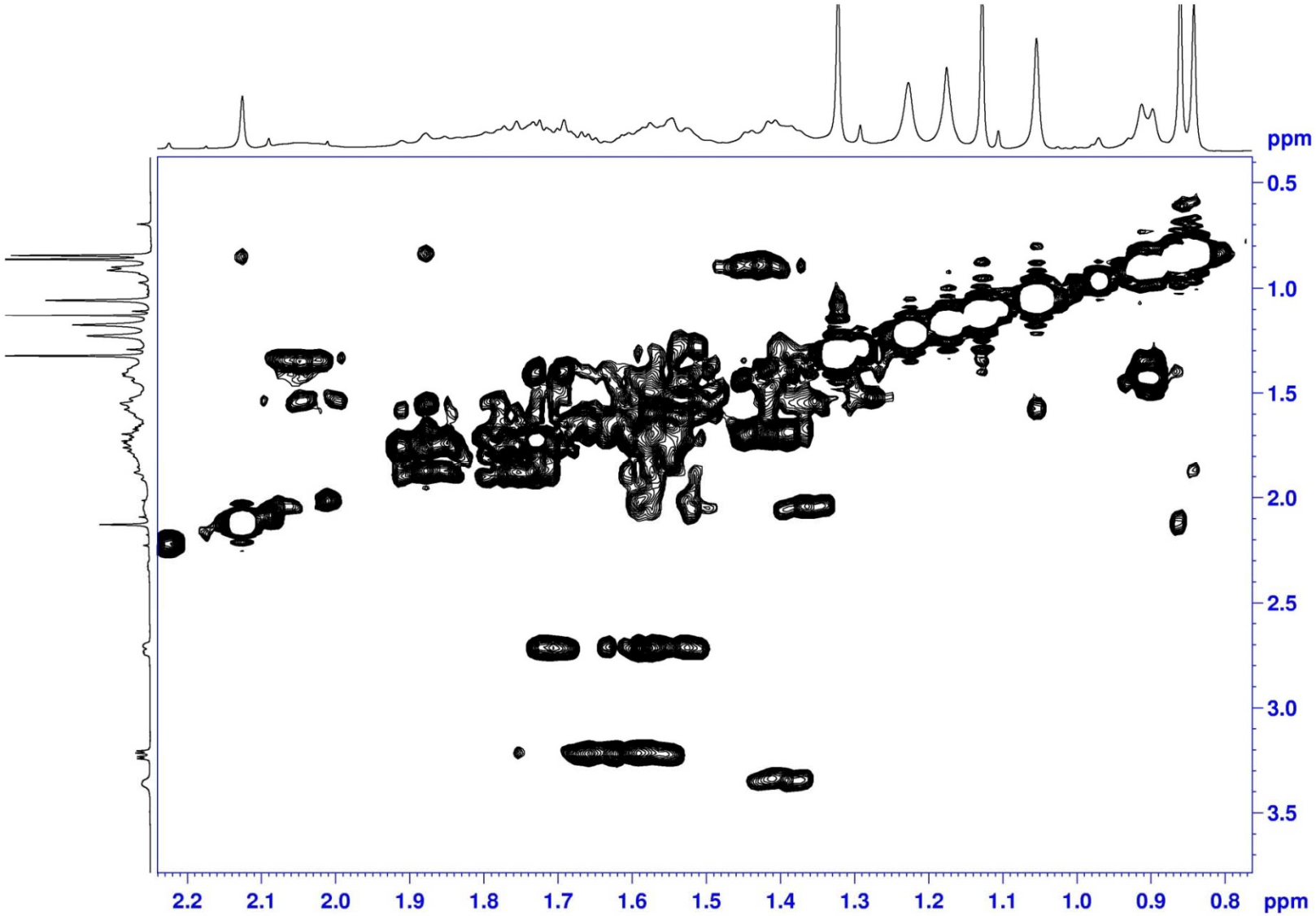



```
NAME                EA-16
EXPNO                   4
PROCNO                  1
Date_            20160806
Time                 5.58
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG       cosygpppqf
TD                   2048
SOLVENT             CDCl3
NS                      8
DS                      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            400.1300062 MHz
WDW                 QSINE
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
SI                   1024
MC2                     QF
SF            400.1300086 MHz
WDW                 QSINE
SSB                     0
```

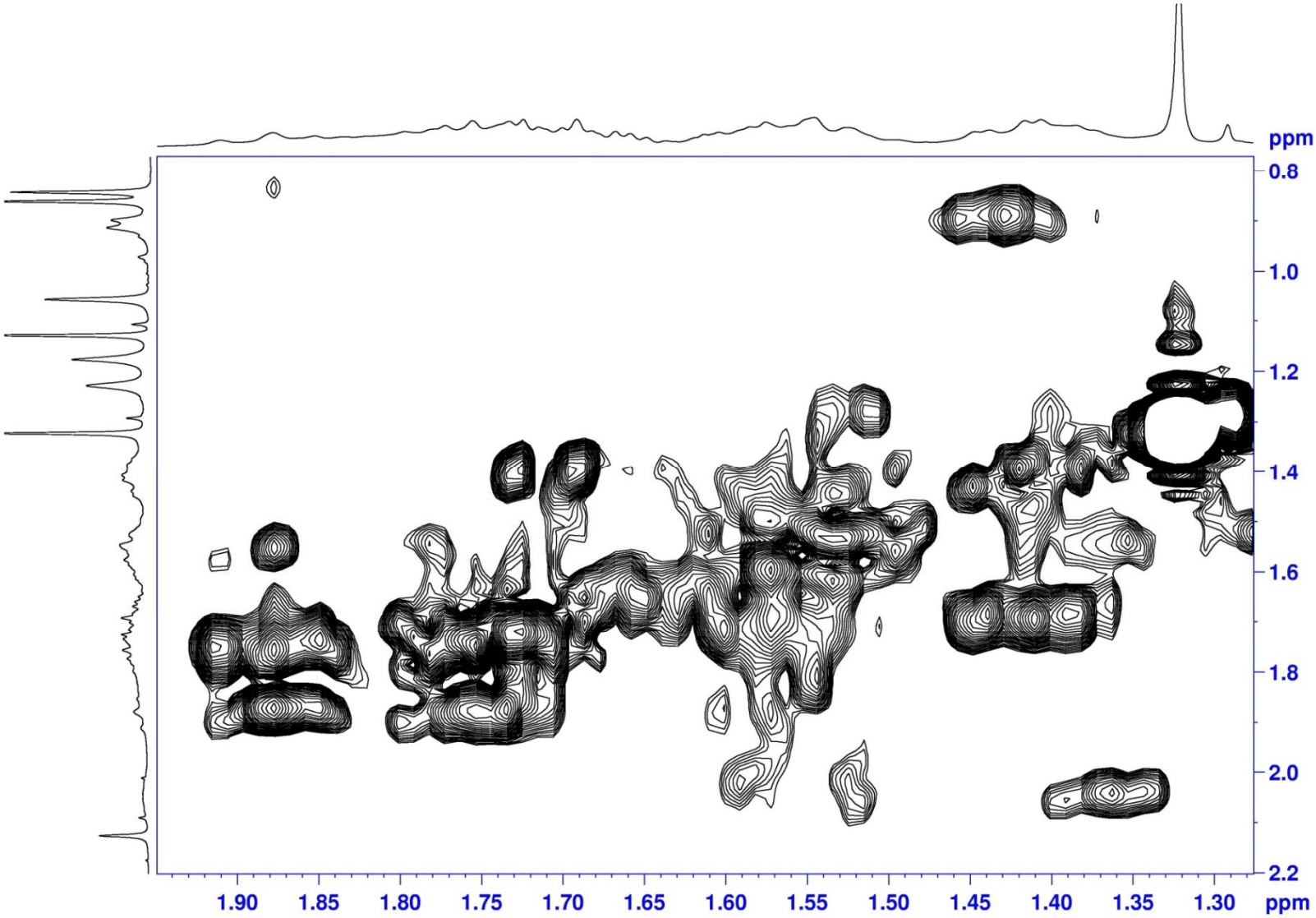
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **6** in $CDCl_3$



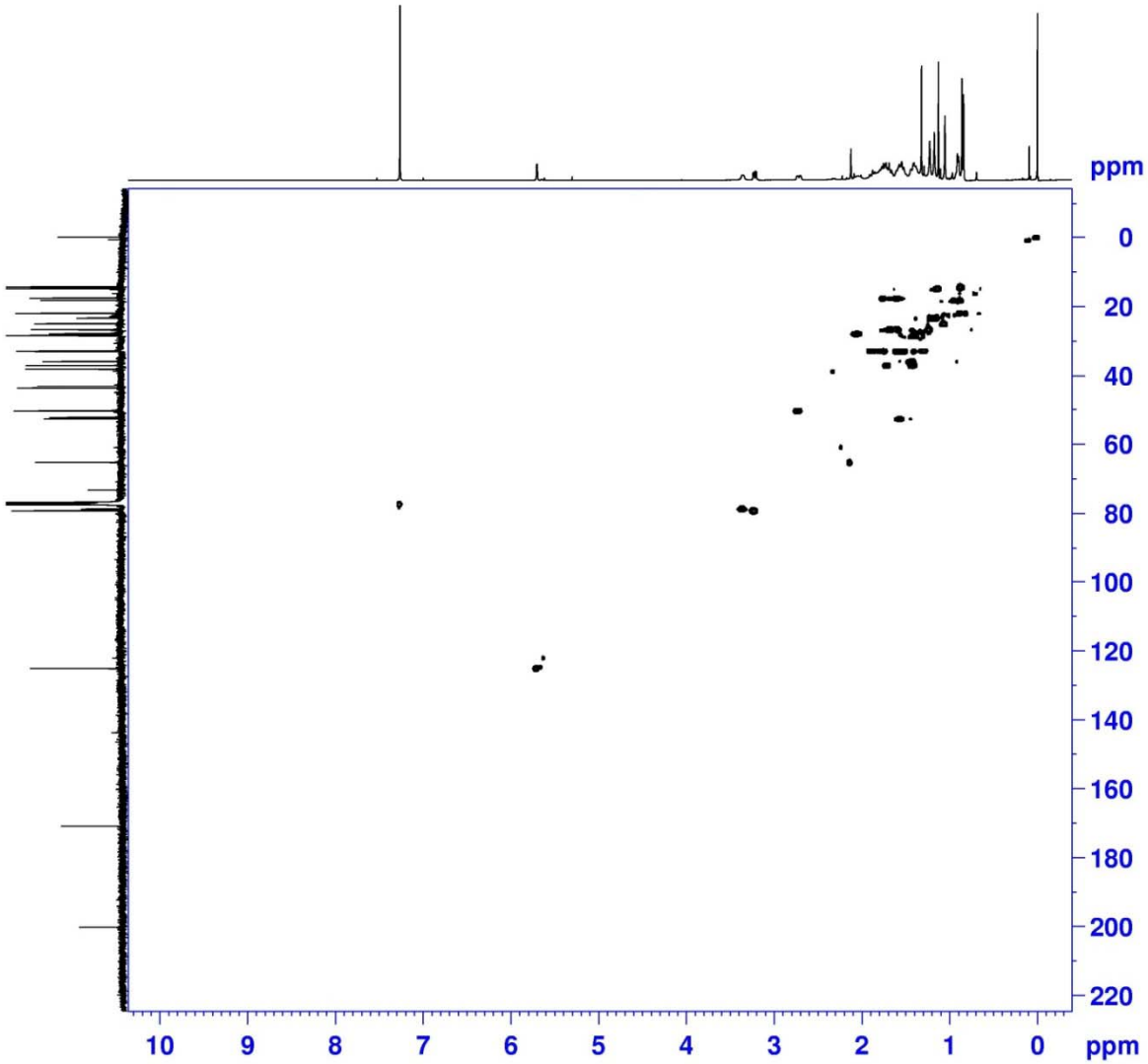
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **6** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **6** in $CDCl_3$

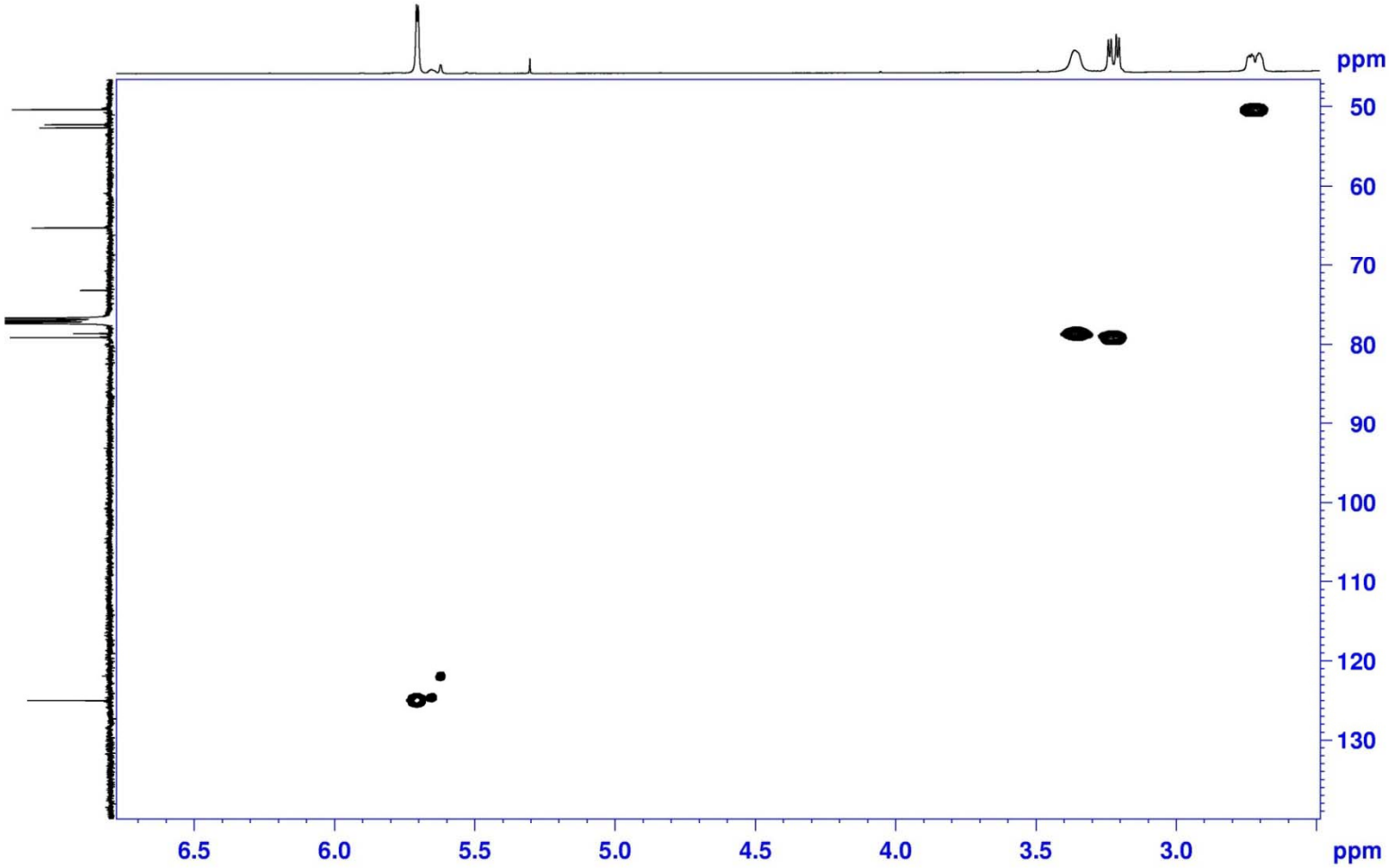


HSQC (400 MHz) spectrum of compound **6** in $CDCl_3$

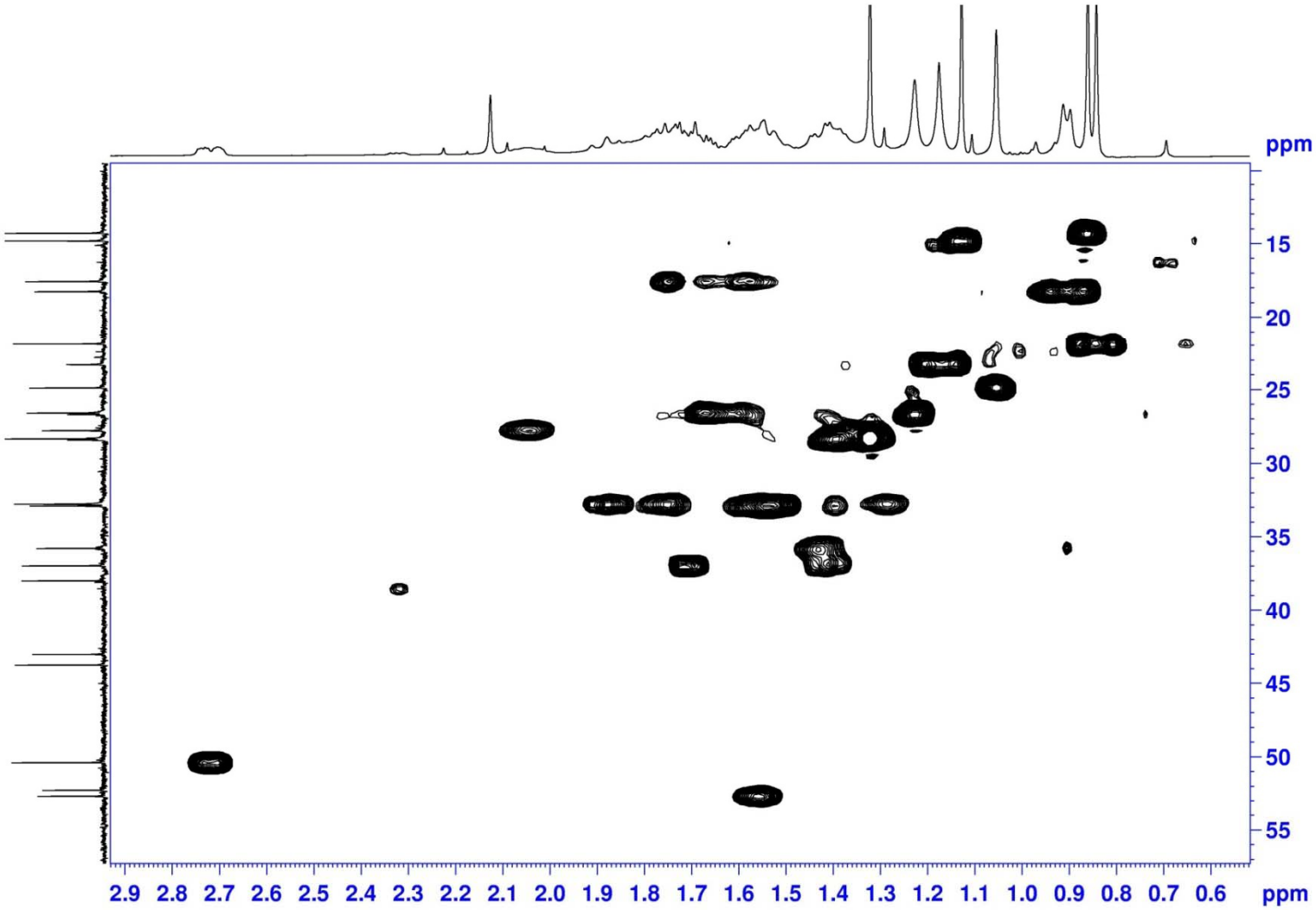



```
NAME                EA-16
EXPNO                   5
PROCNO                  1
Date_            20160806
Time                 6.37
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG     hsqcetgpsi2
TD                   1024
SOLVENT             CDCl3
NS                     16
DS                     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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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           400.1300059 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                   1024
MC2        echo-antiecho
SF           100.6127568 MHz
WDW                 QSINE
SSB                    2
```

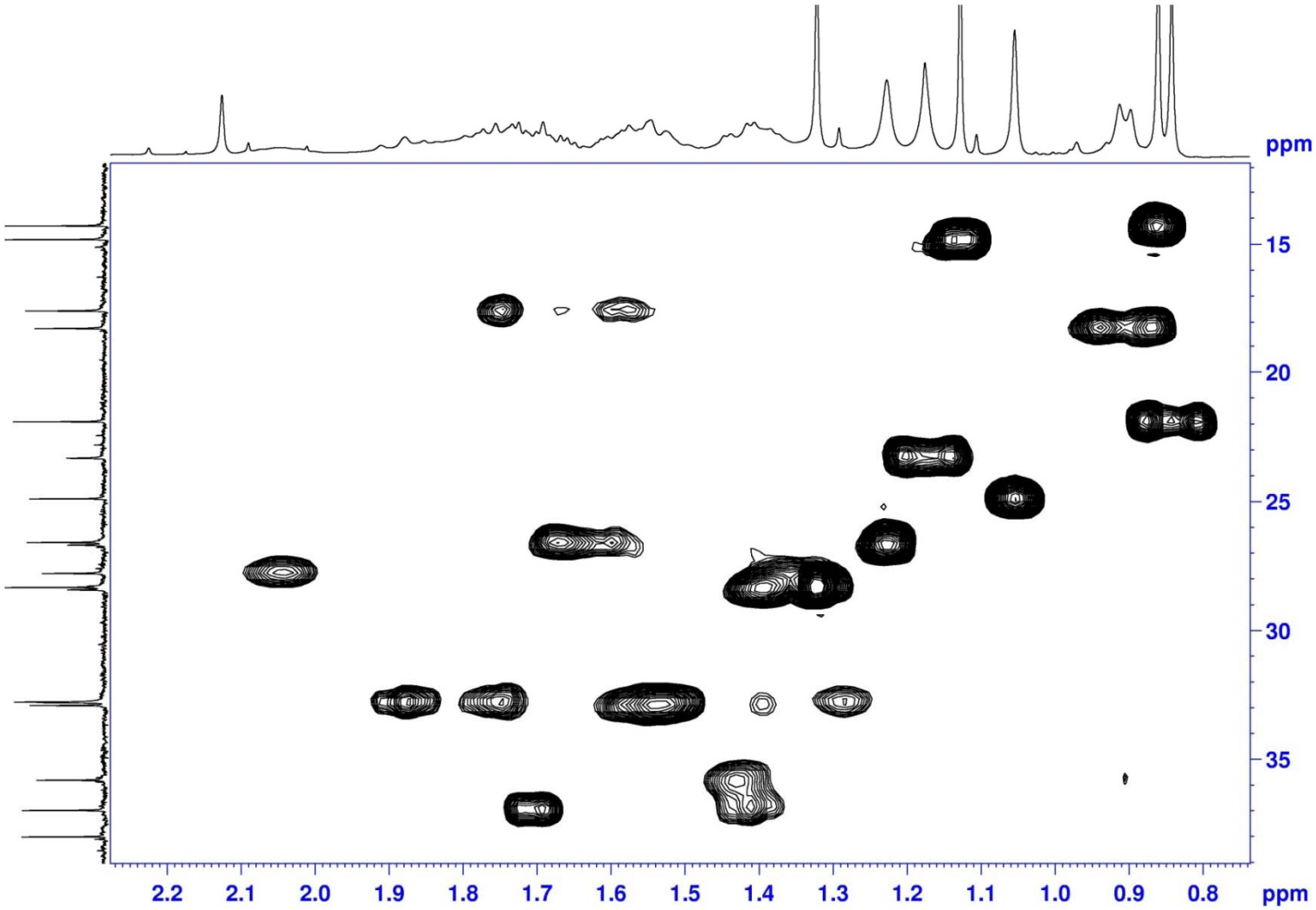
HSQC (400 MHz) spectrum of compound **6** in $CDCl_3$



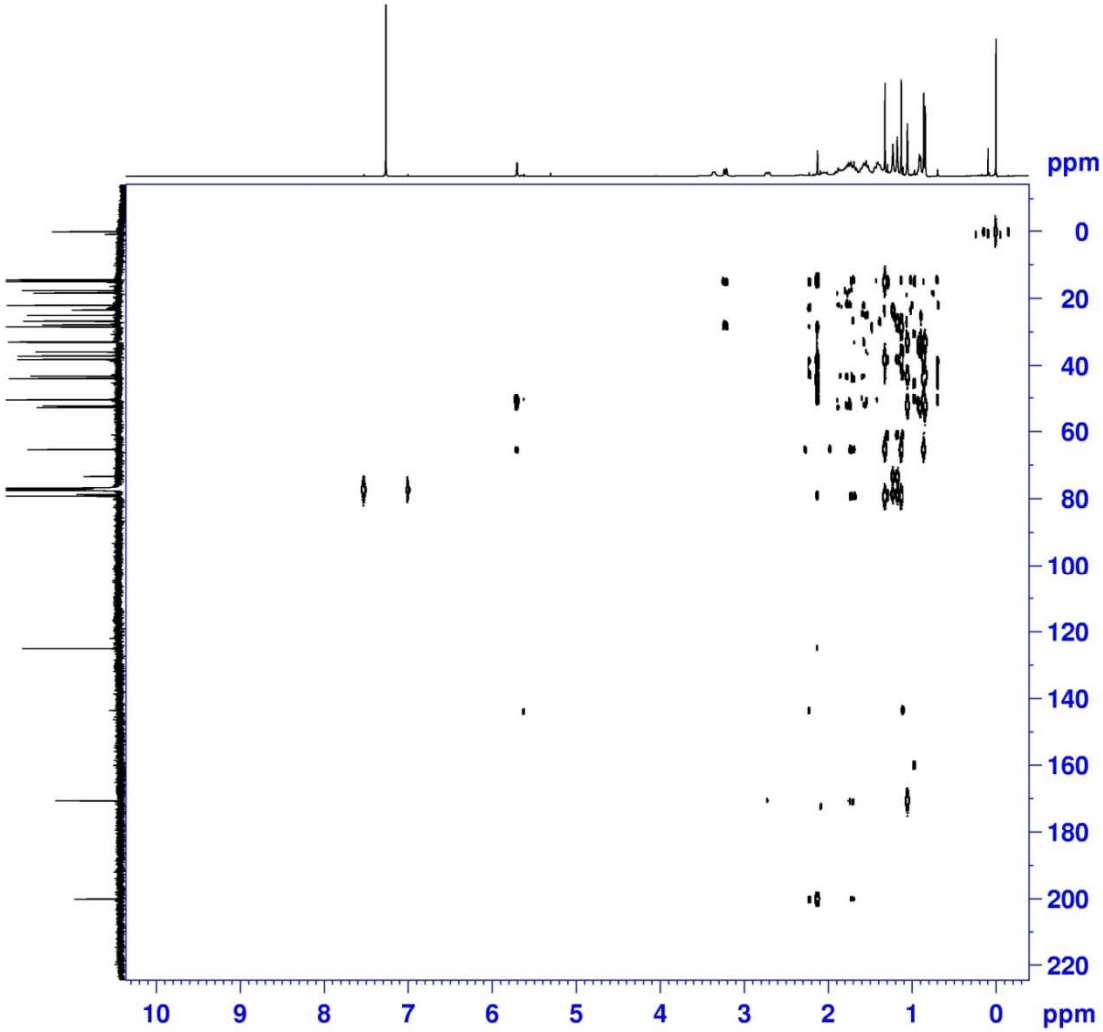
HSQC (400 MHz) spectrum of compound **6** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **6** in $CDCl_3$

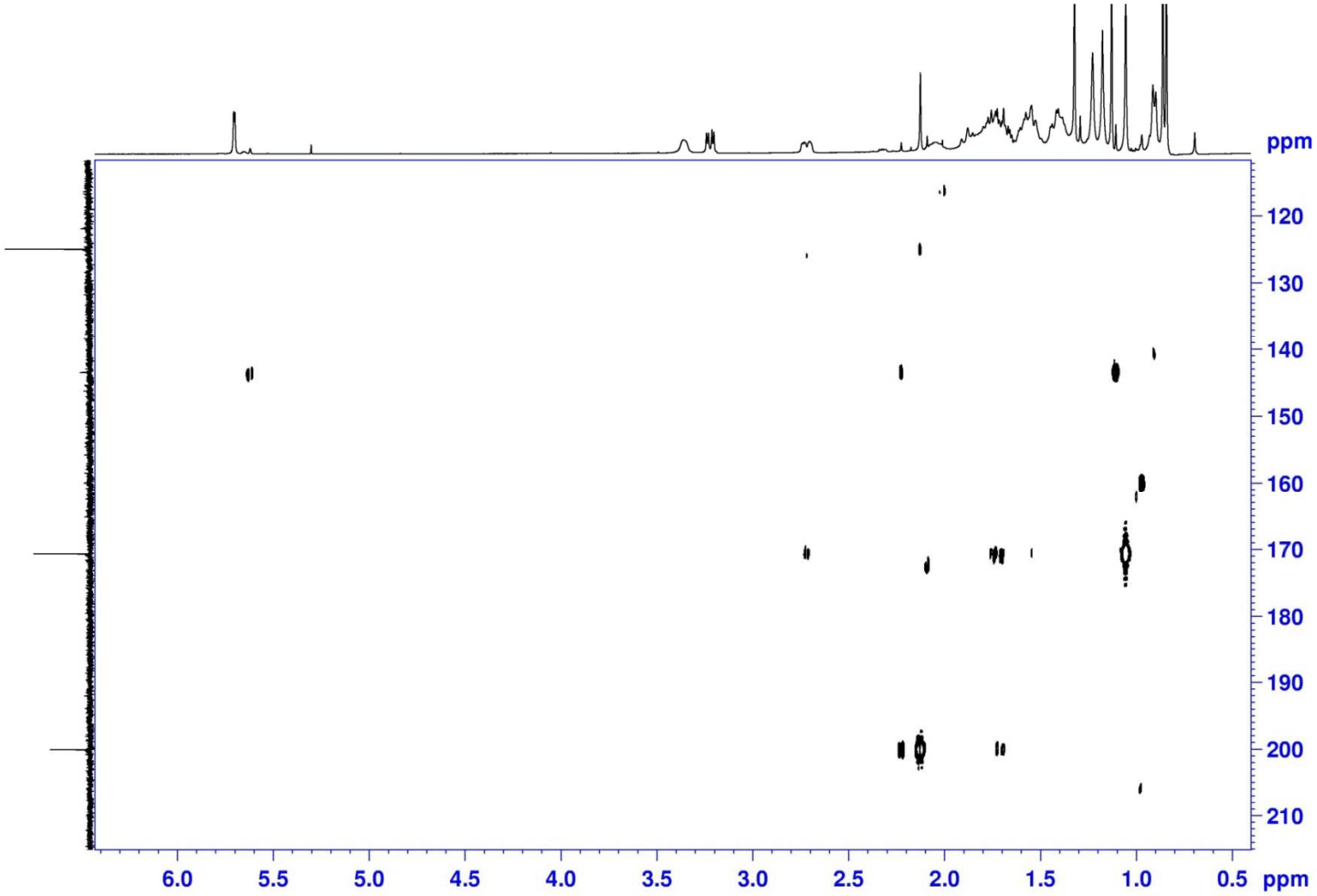


HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$

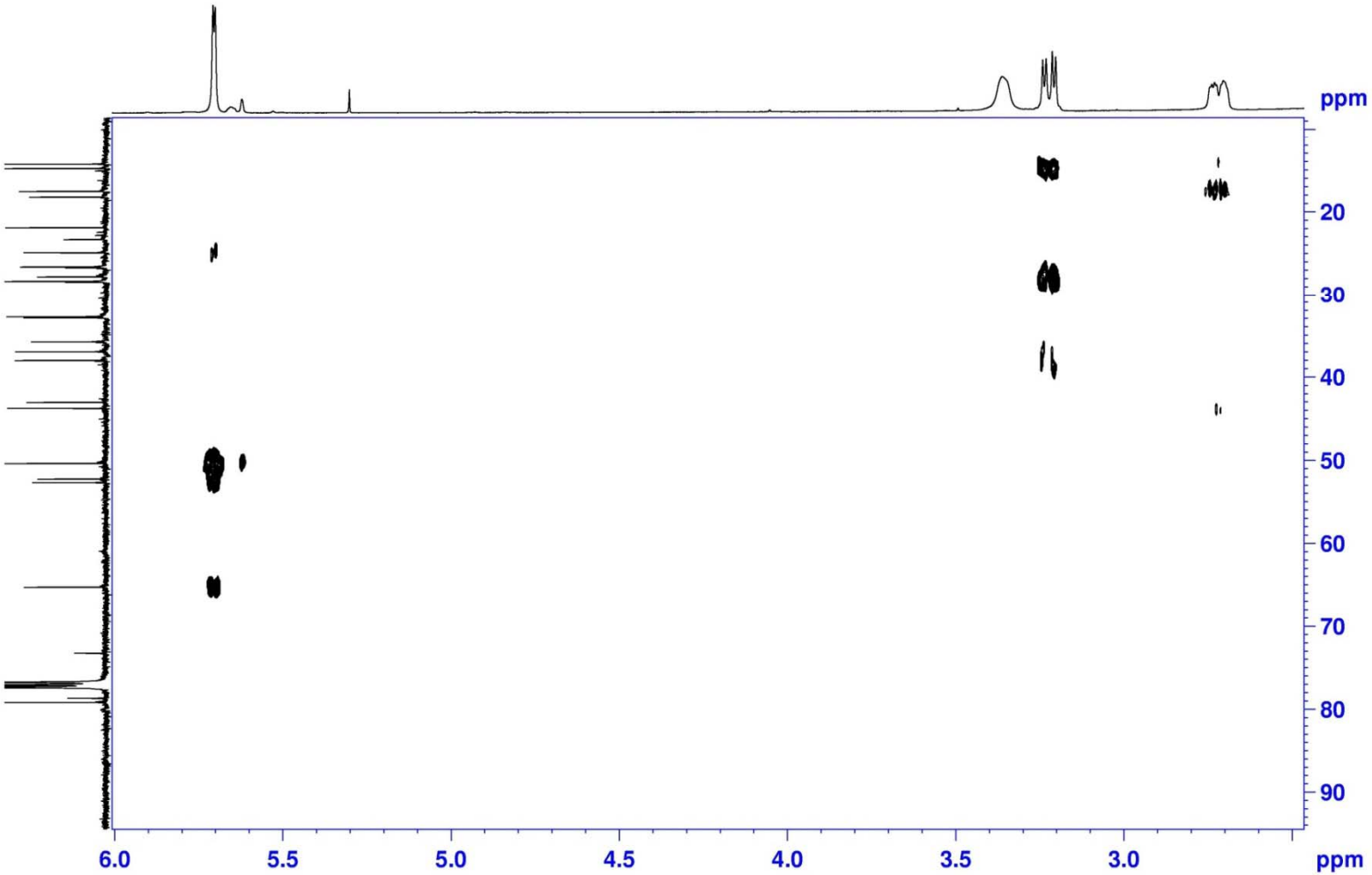



```
EXPNO                 6
PROCNO                1
Date_          20160806
Time               8.28
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG   hmbcgplpndqf
TD                 4096
SOLVENT           CDCl3
NS                   32
DS                   16
SWH            5197.505 Hz
FIDRES         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 ========
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
SI                 2048
SF          400.1300066 MHz
WDW                SINE
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2                  QF
SF          100.6127622 MHz
WDW                SINE
SSB                   0
LB                 0.00 Hz
GB                    0
```

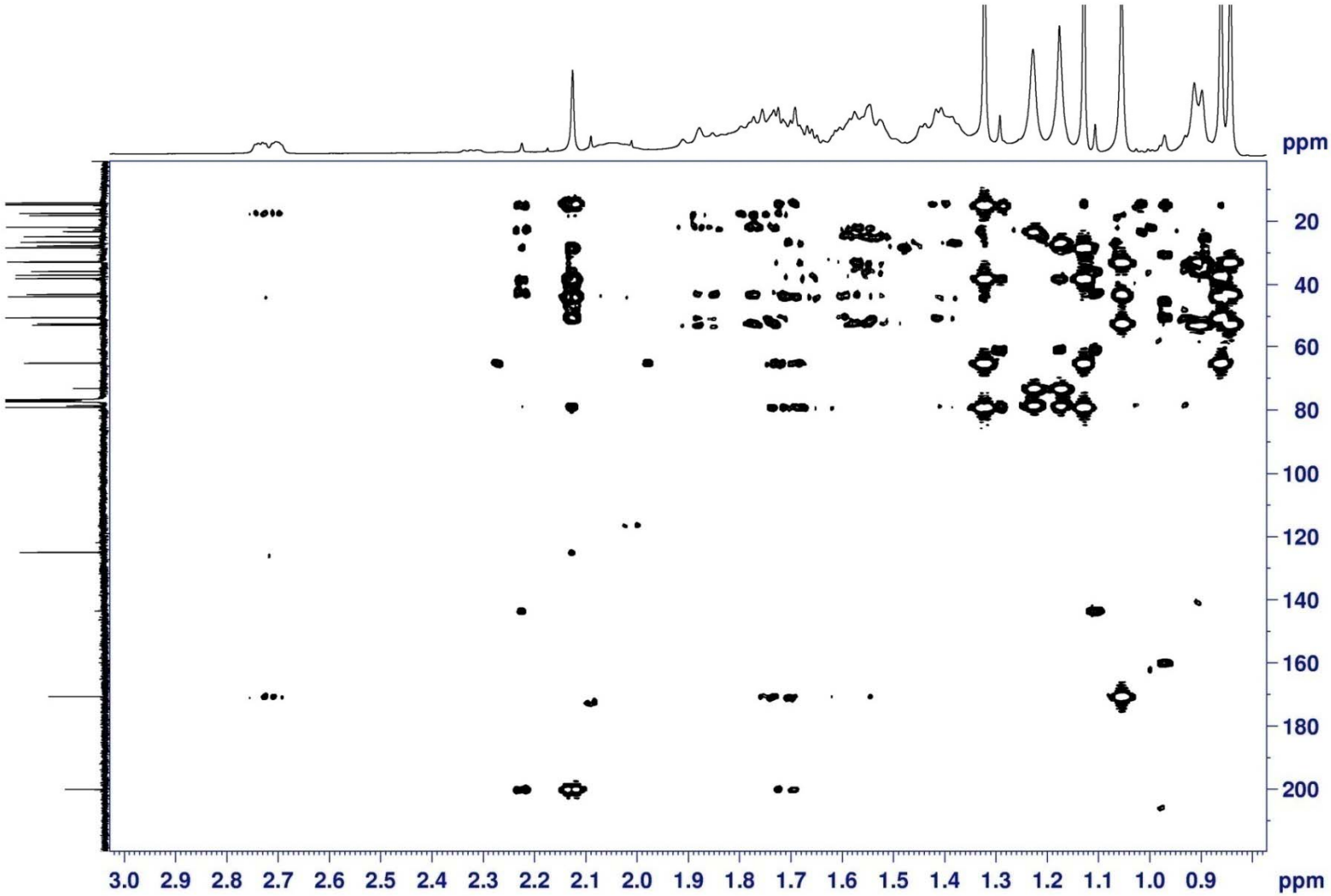
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



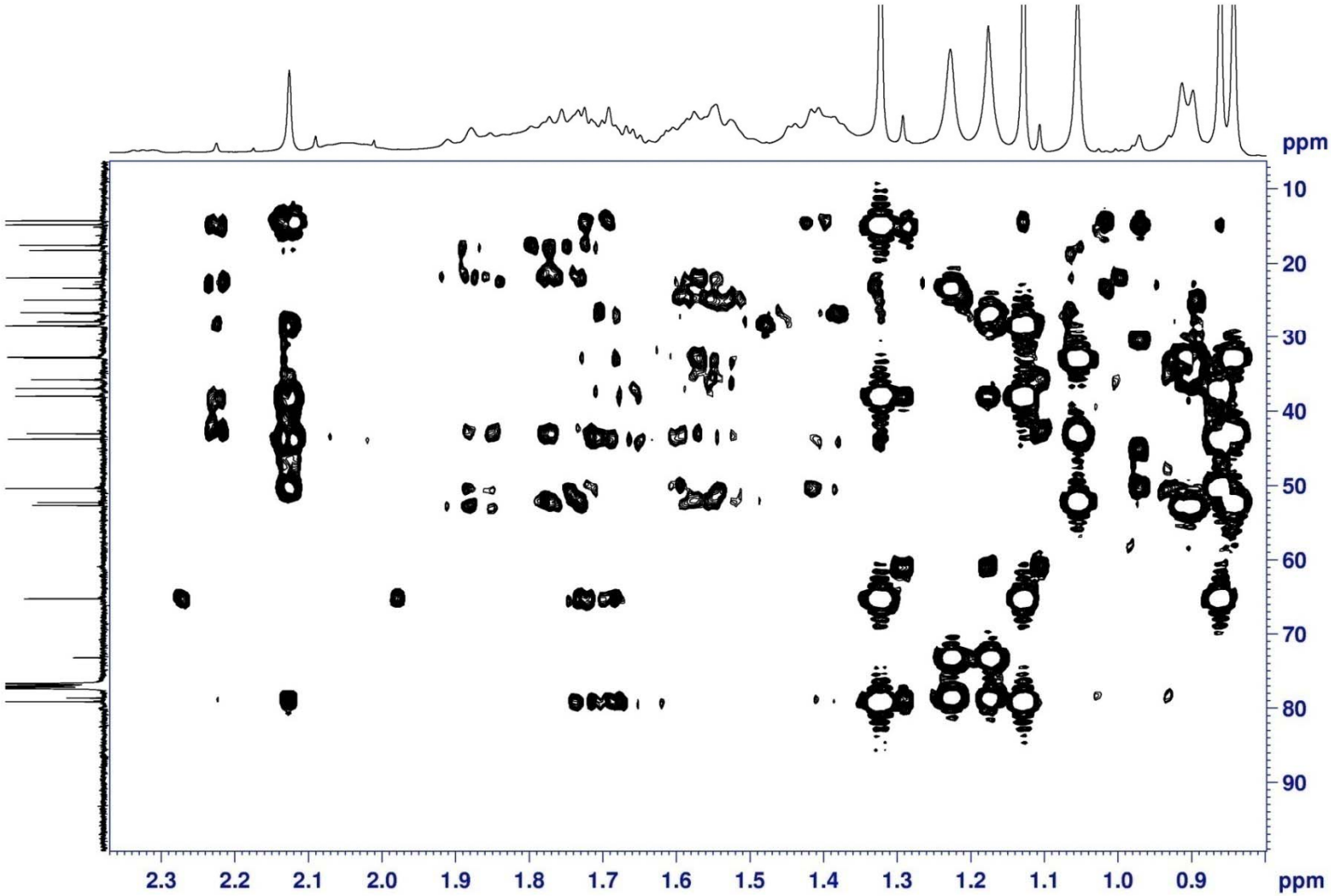
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



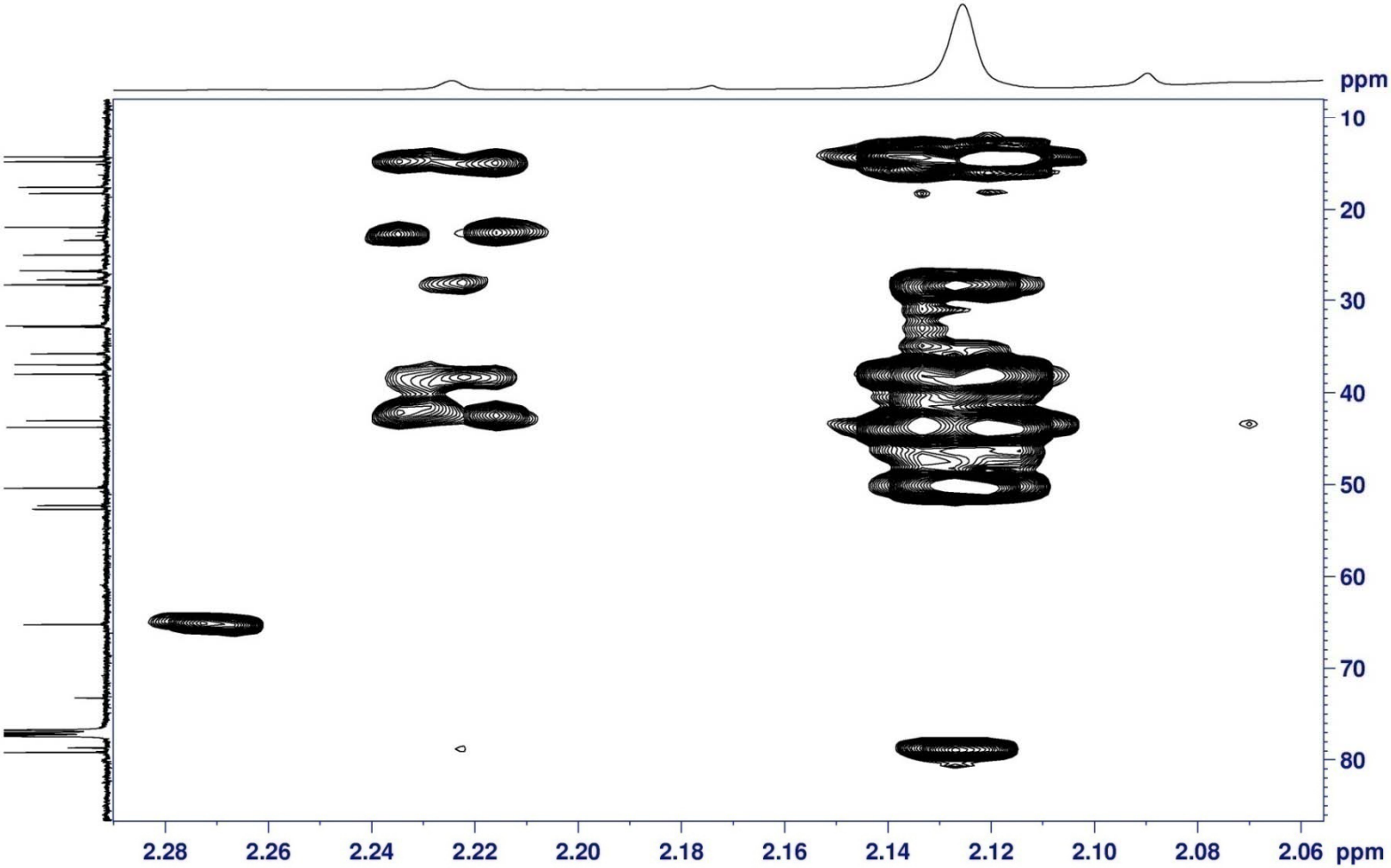
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



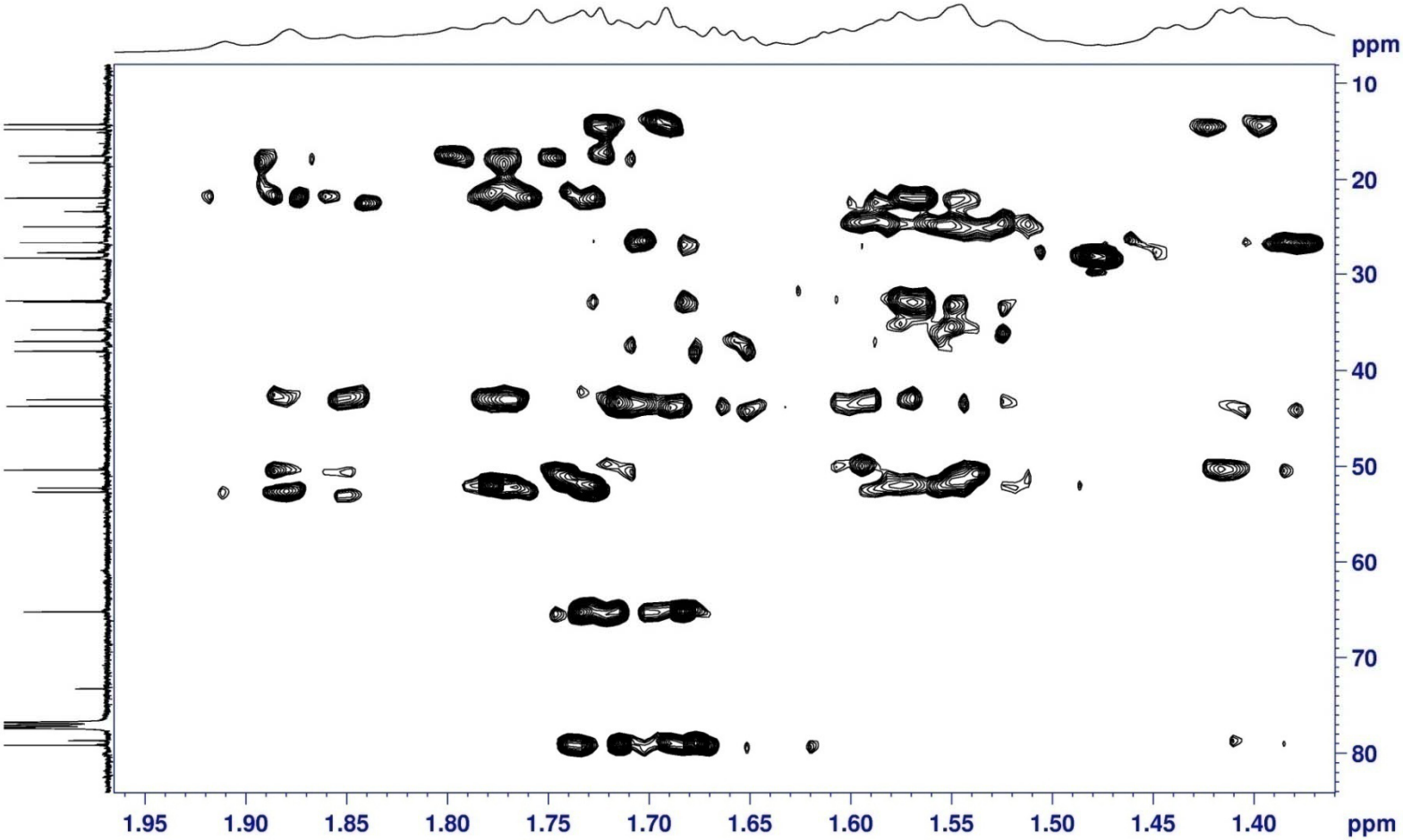
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



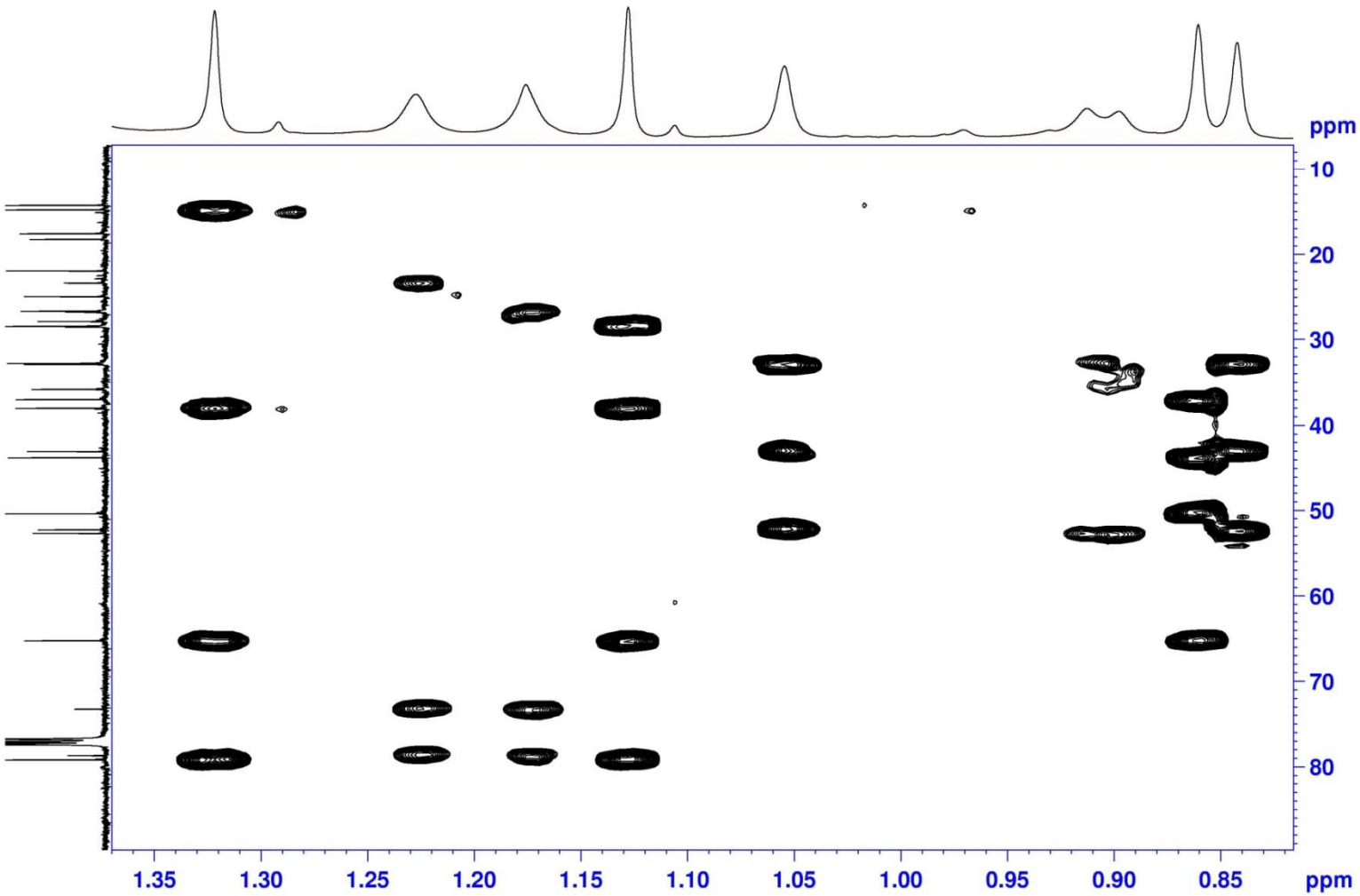
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



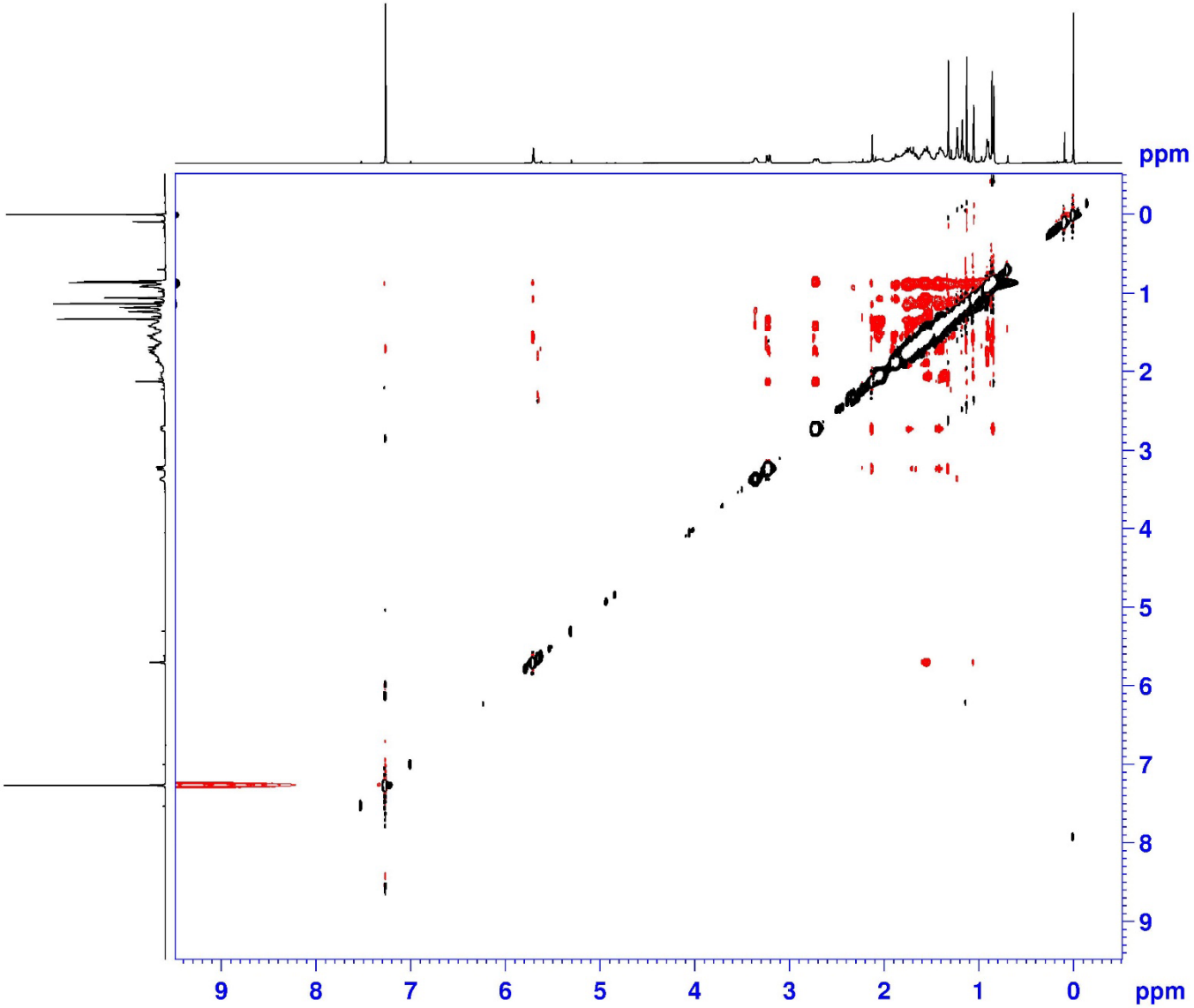
HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **6** in $CDCl_3$

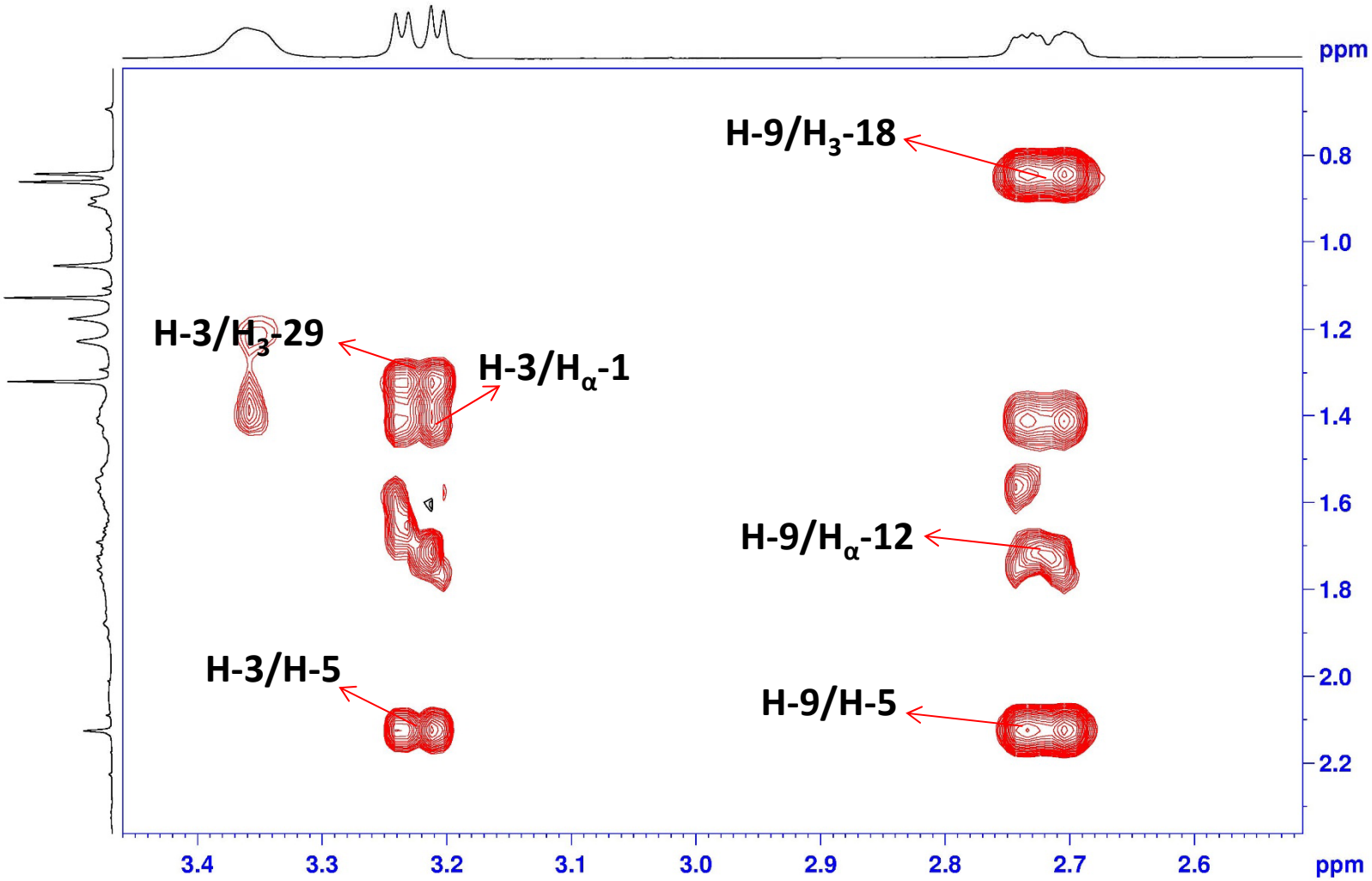


NOESY (400 MHz) spectrum of compound **6** in $CDCl_3$

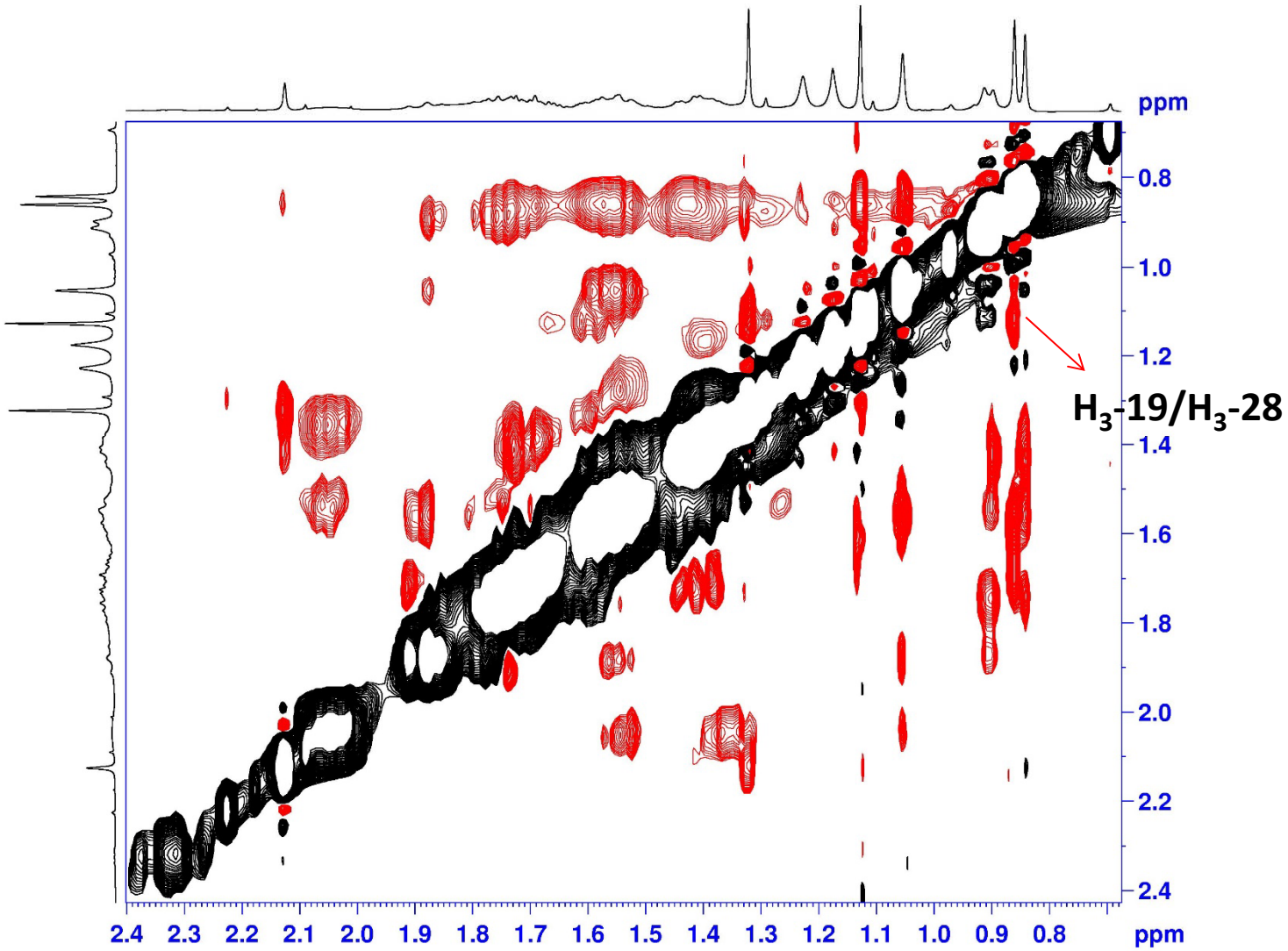



```
NAME                EA-16
EXPNO                   7
PROCNO                  1
Date_            20160806
Time                10.44
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                     16
DS                     32
SWH             4000.000 Hz
FIDRES          1.953125 Hz
AQ             0.2560500 se
RG                 208.5
DW               125.000 us
DE                 10.00 us
TE                 297.0 K
D0            0.00011036 se
D1            1.99385595 se
D8            0.30000001 se
D11           0.03000000 se
D12           0.00002000 se
D16           0.00020000 se
IN0           0.00025000 se

======== CHANNEL f1 ======
SFO1         400.1318006 MH
NUC1                  1H
P1                 11.50 us
P2                 23.00 us
P17              2500.00 us
ND0                     1
TD                    256
SFO1            400.1318 MH
FIDRES         15.625000 Hz
SW                 9.997 pp
FnMODE       States-TPPI
SI                   1024
SF           400.1300058 MH
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00
SI                   1024
MC2          States-TPPI
SF           400.1300069 MH
WDW                 OSINE
```

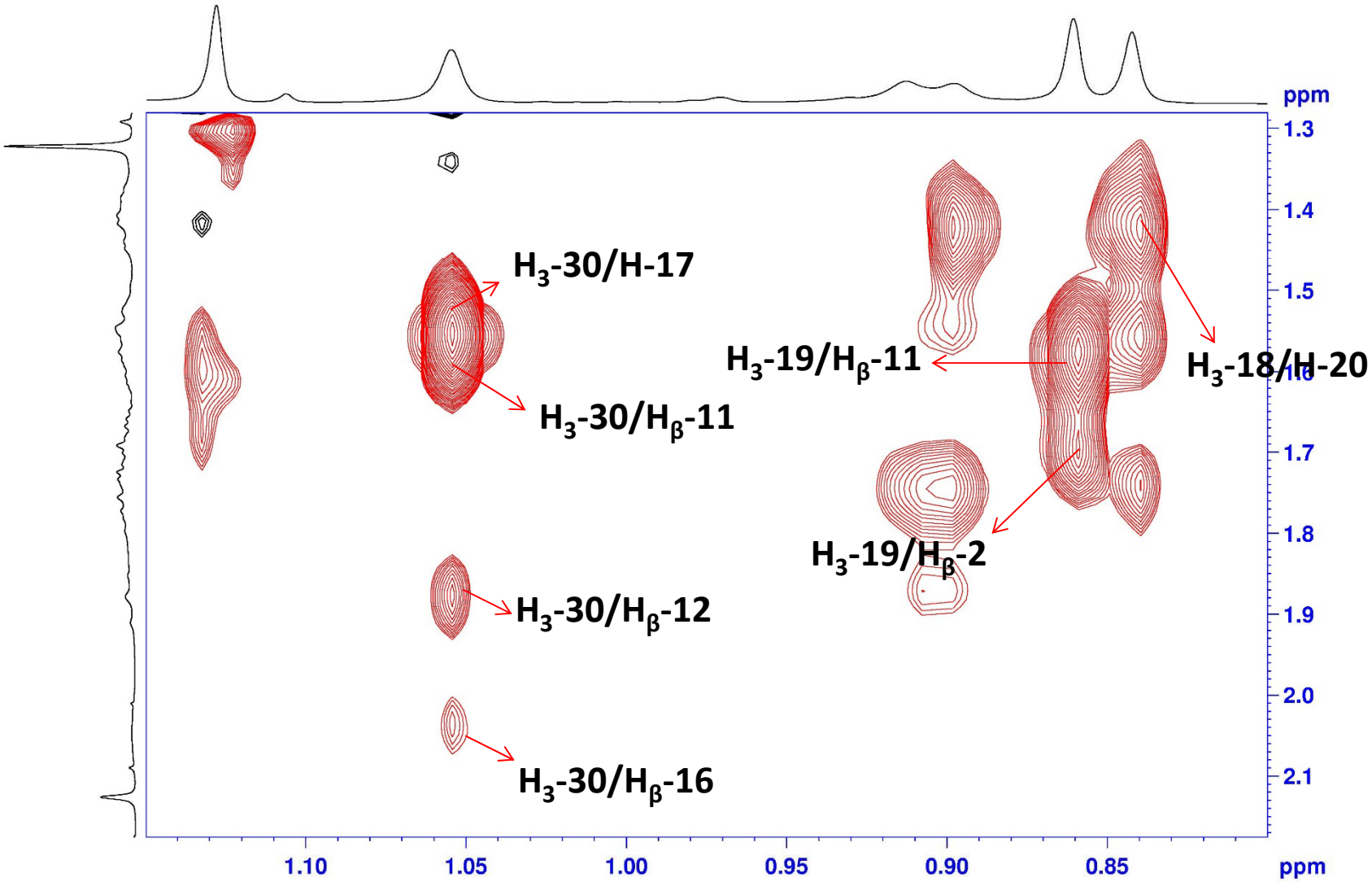
NOESY (400 MHz) spectrum of compound **6** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **6** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **6** in $CDCl_3$

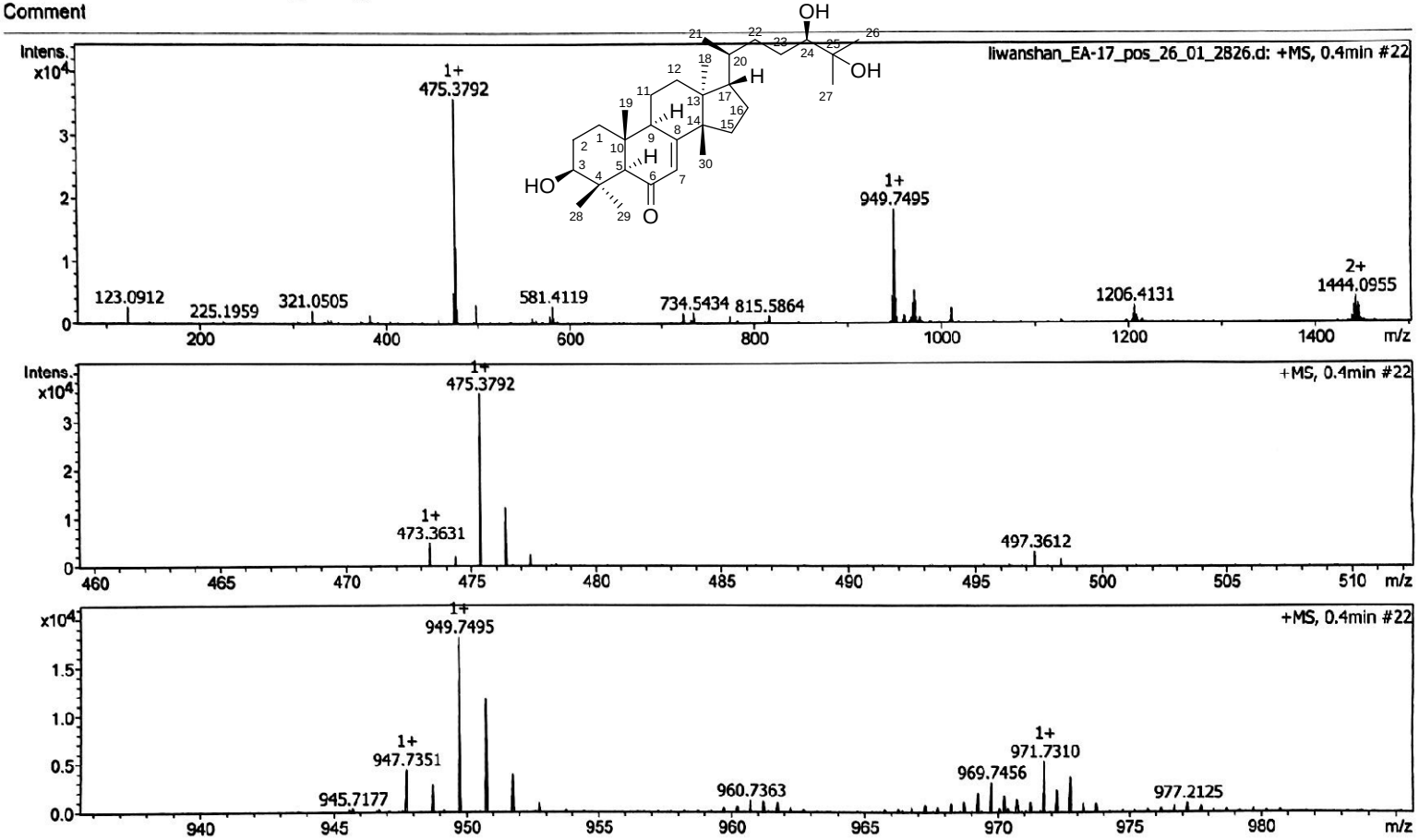


HR-ESIMS for compound **7**

Generic Display Report

Analysis Info

Analysis Name	D:\Data\MS\data\201612\liwanshan_EA-17_pos_26_01_2826.d	Acquisition Date	12/21/2016 4:55:09 PM
Method	LC_Direct Infusion_pos_70-500mz.m	Operator	SCSIO
Sample Name	liwanshan_EA-17_pos	Instrument	maXis
Comment			



HR-ESIMS for compound **7**

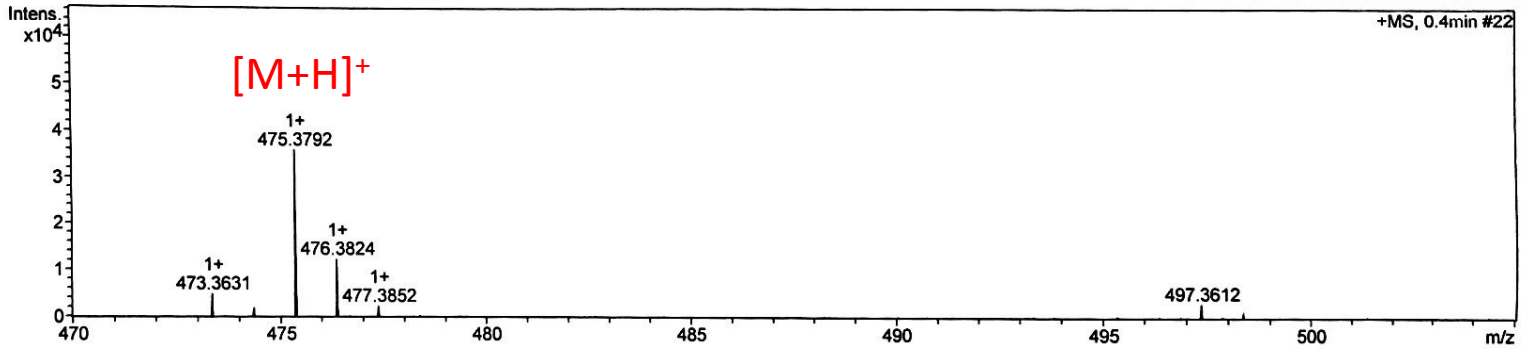
Mass Spectrum SmartFormula Report

Analysis Info

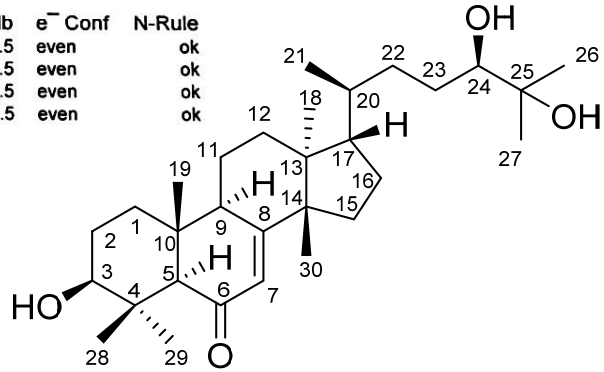
Analysis Name	D:\Data\MS\data\201612\liwanshan_EA-17_pos_26_01_2826.d	Acquisition Date	12/21/2016 4:55:09 PM
Method	LC_Direct Infusion_pos_70-500mz.m	Operator	SCSIO
Sample Name	liwanshan_EA-17_pos	Instrument	maXis 255552.00029
Comment			

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	70 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



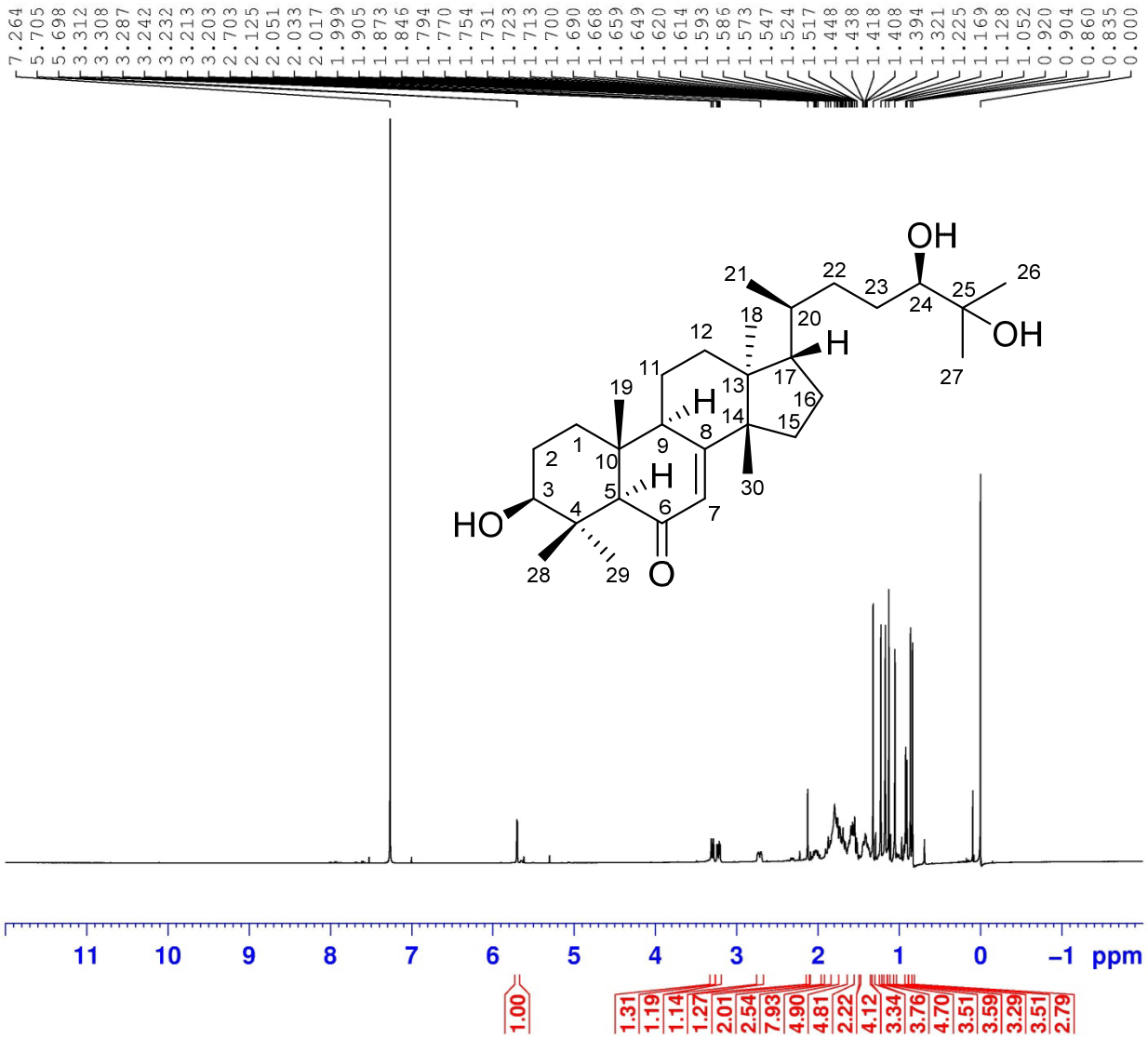
Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
475.379226	1	C30H51O4	100.00	475.378187	2.2	1.0	6.3	5.5	even	ok
497.361232	1	C30H50NaO4	100.00	497.360131	-2.2	-1.1	20.8	5.5	even	ok
949.749486	1	C60H101O8	100.00	949.749097	0.4	0.4	9.2	10.5	even	ok
971.730951	1	C60H100NaO8	100.00	971.731041	-0.1	-0.1	25.7	10.5	even	ok



liwanshan_EA-17_pos_26_01_2826.d

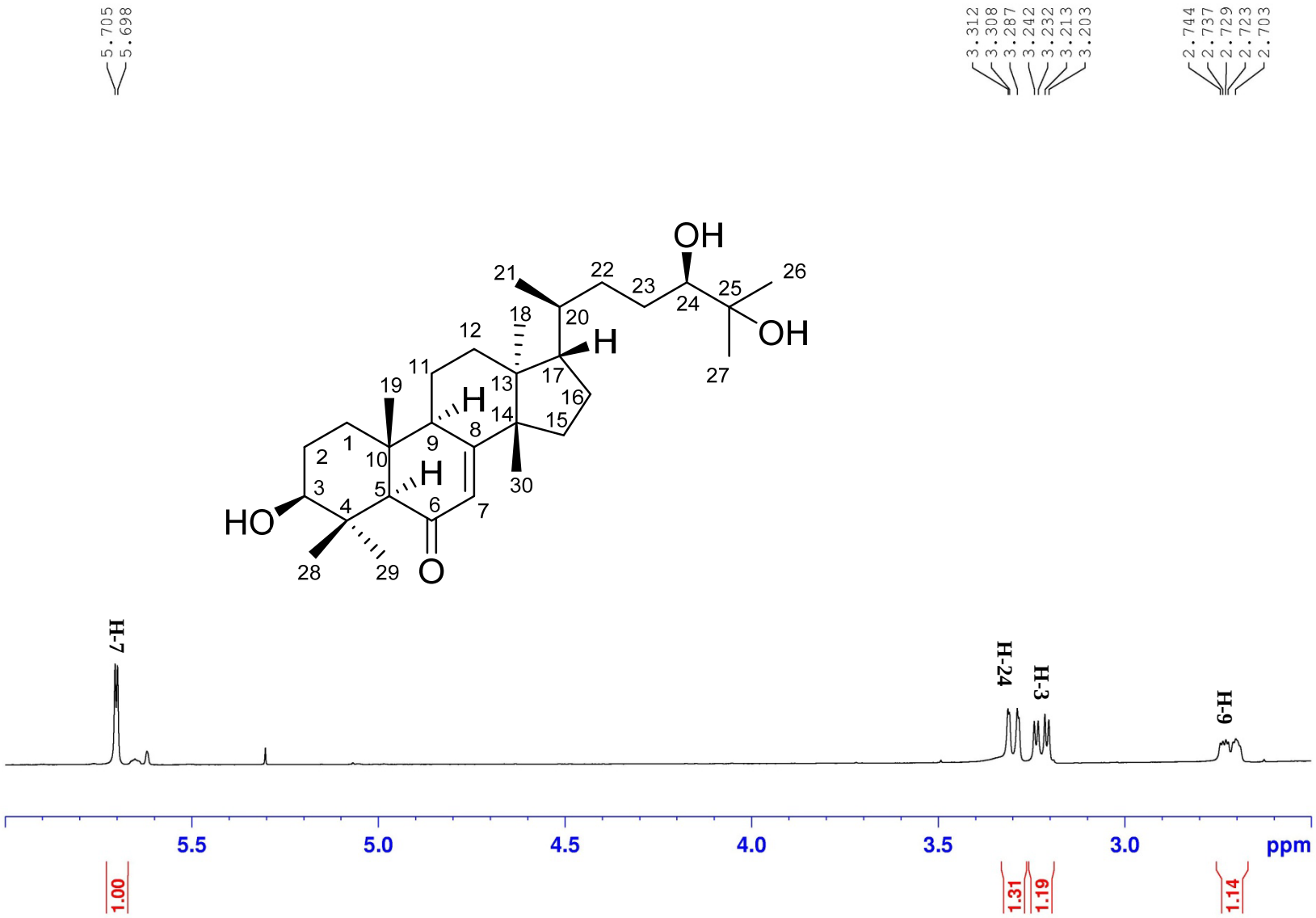
Bruker Compass DataAnalysis 4.1 printed: 12/21/2016 4:59:28 PM by: SCSIO Page 1 of 1

^{1}H NMR (400 MHz) spectrum of compound **7** in $CDCl_3$

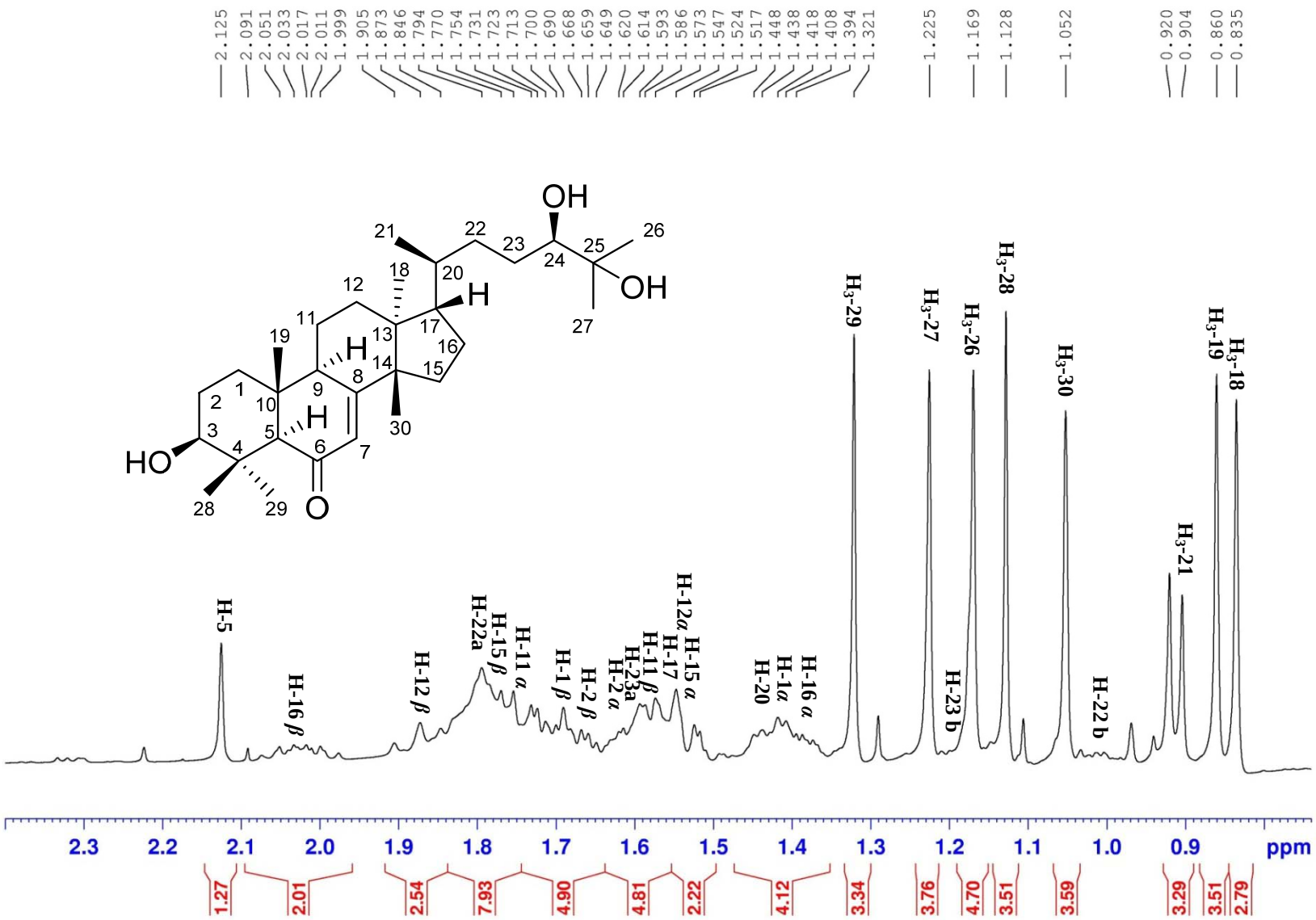



```
NAME              EA-17
EXPNO                 1
PROCNO                1
Date_          20160726
Time              12.53
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9846387 sec
RG               147.94
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                11.50 usec
SI                65536
SF          400.1300079 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

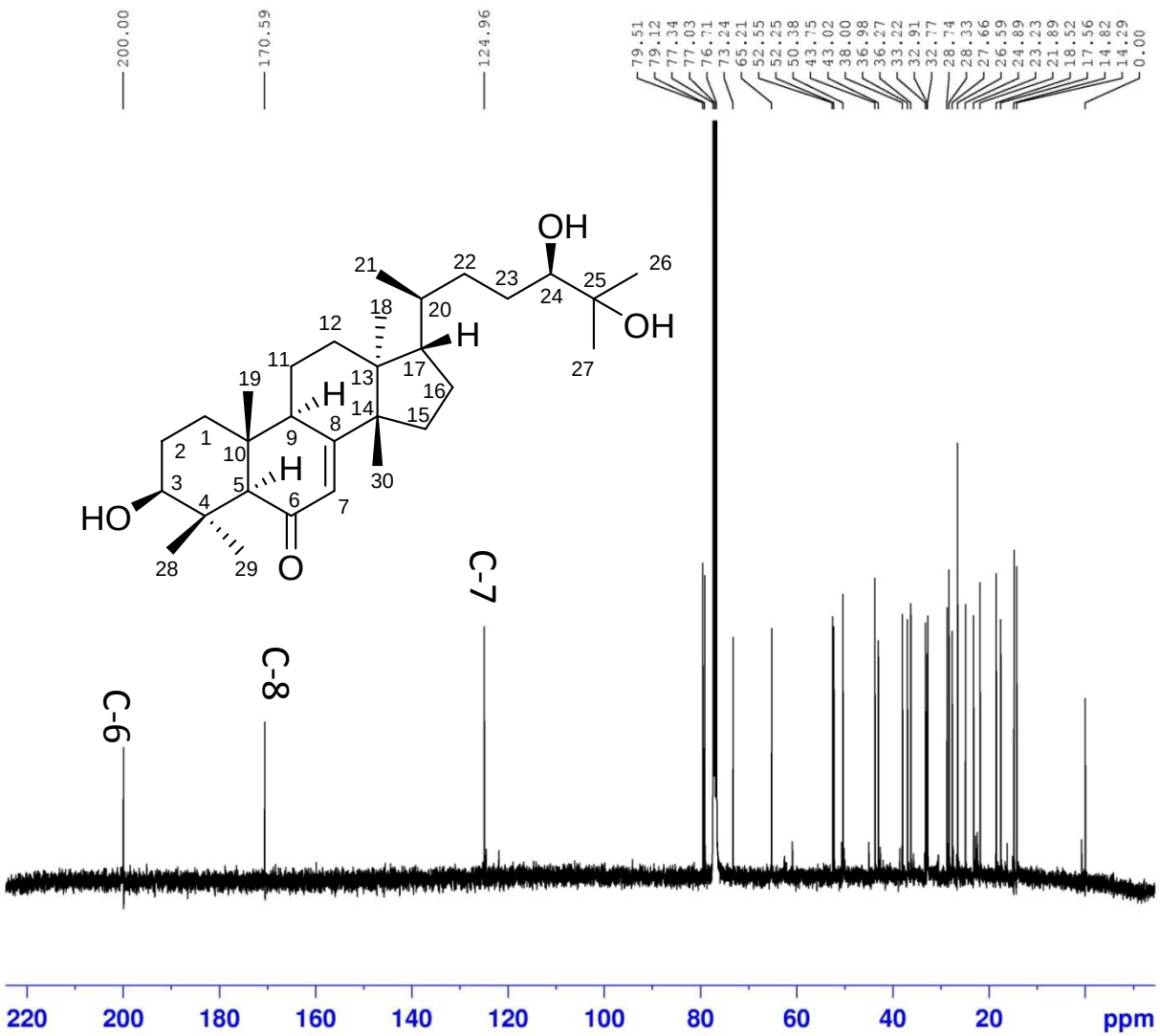
^{1}H NMR (400 MHz) spectrum of compound **7** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **7** in $CDCl_3$

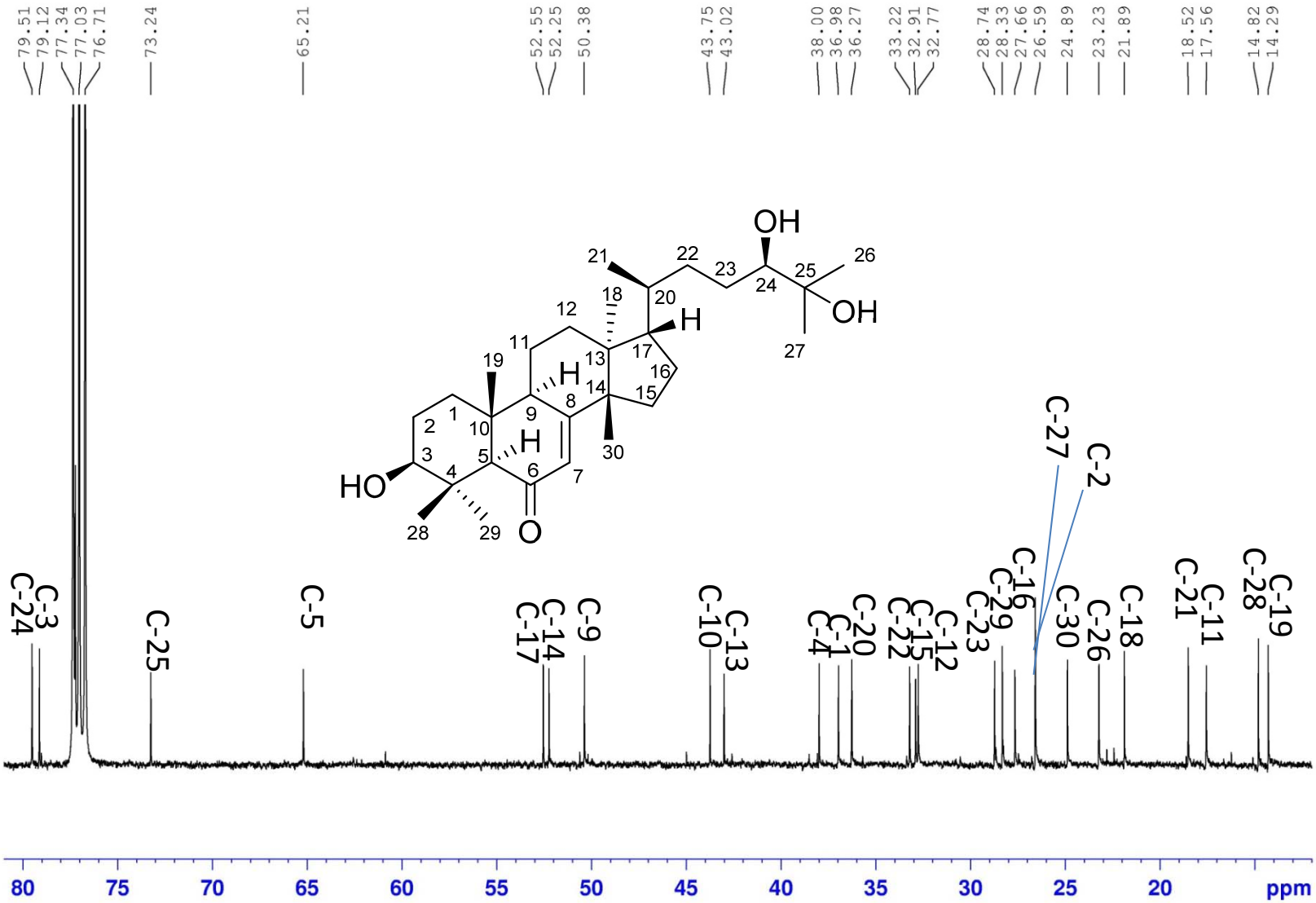


^{13}C NMR (100 MHz) spectrum of compound **7** in $CDCl_3$

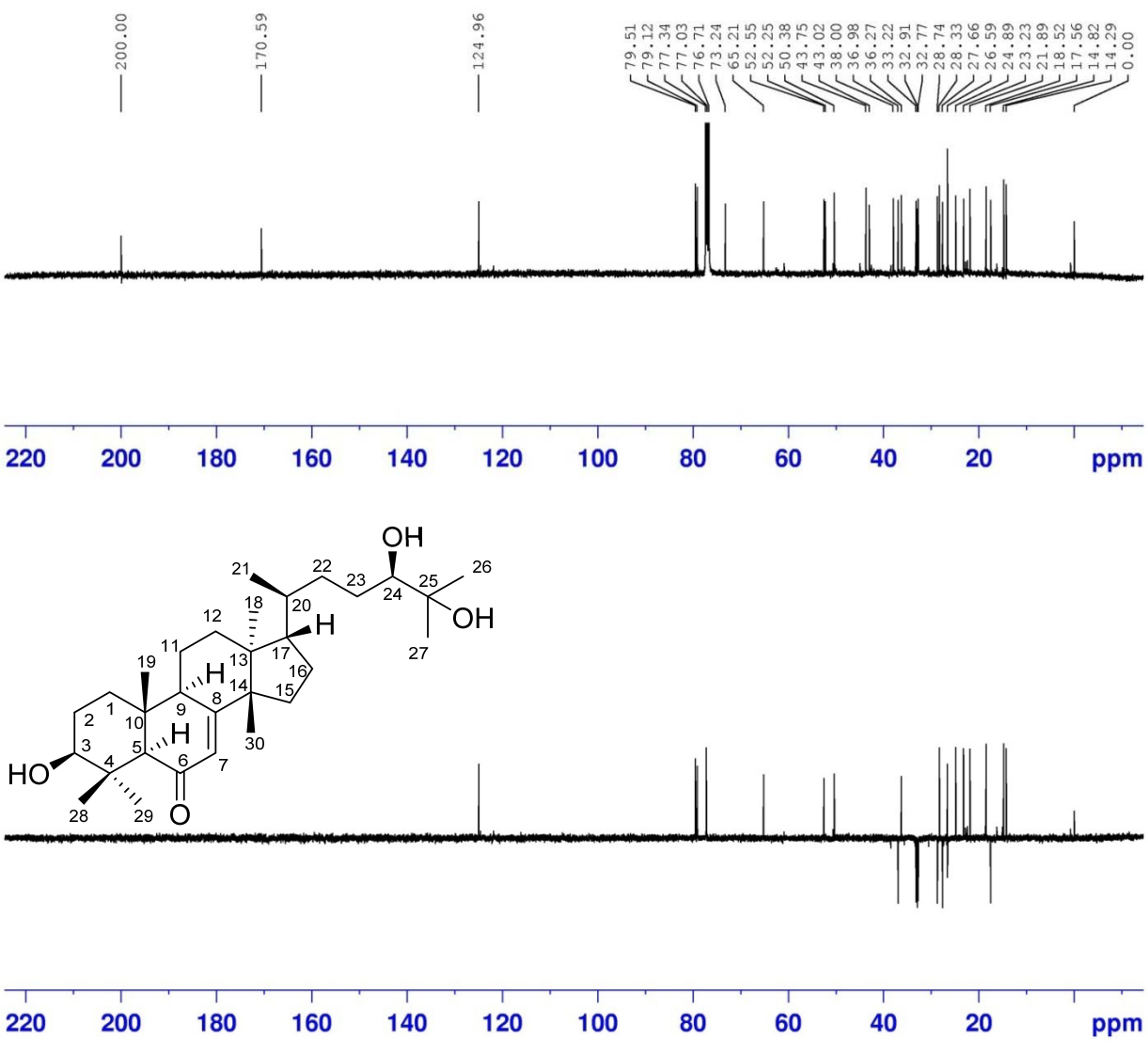



```
NAME              EA-17
EXPNO               102
PROCNO                1
Date_          20160727
Time               0.54
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                102.3
DW               20.800 usec
DE               18.00 usec
TE               297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6233324 MHz
NUC1                13C
P1                10.00 usec
SI                32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{13}C NMR (100 MHz) spectrum of compound **7** in $CDCl_3$

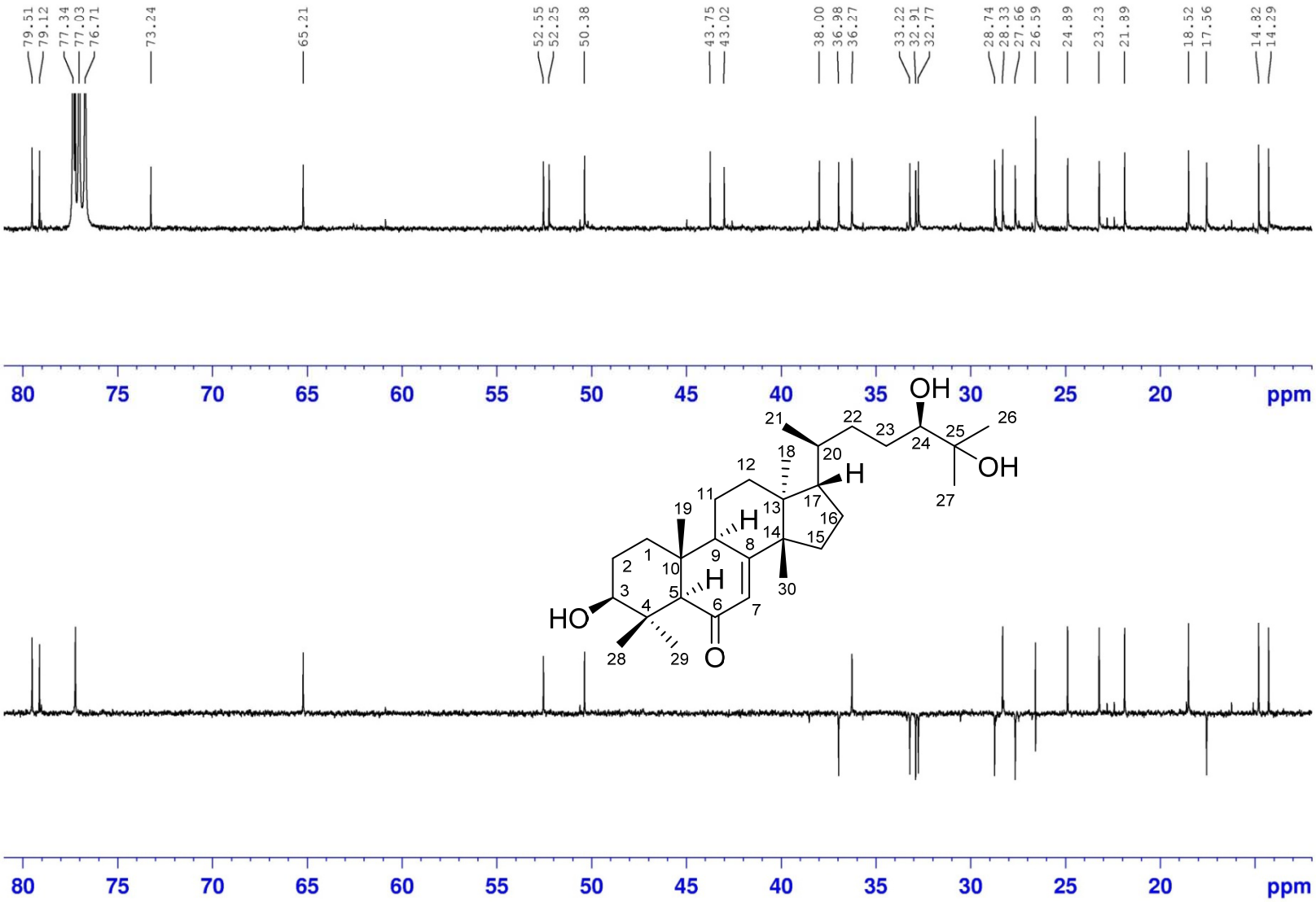


DEPT135 (100 MHz) spectrum of compound **7** in $CDCl_3$

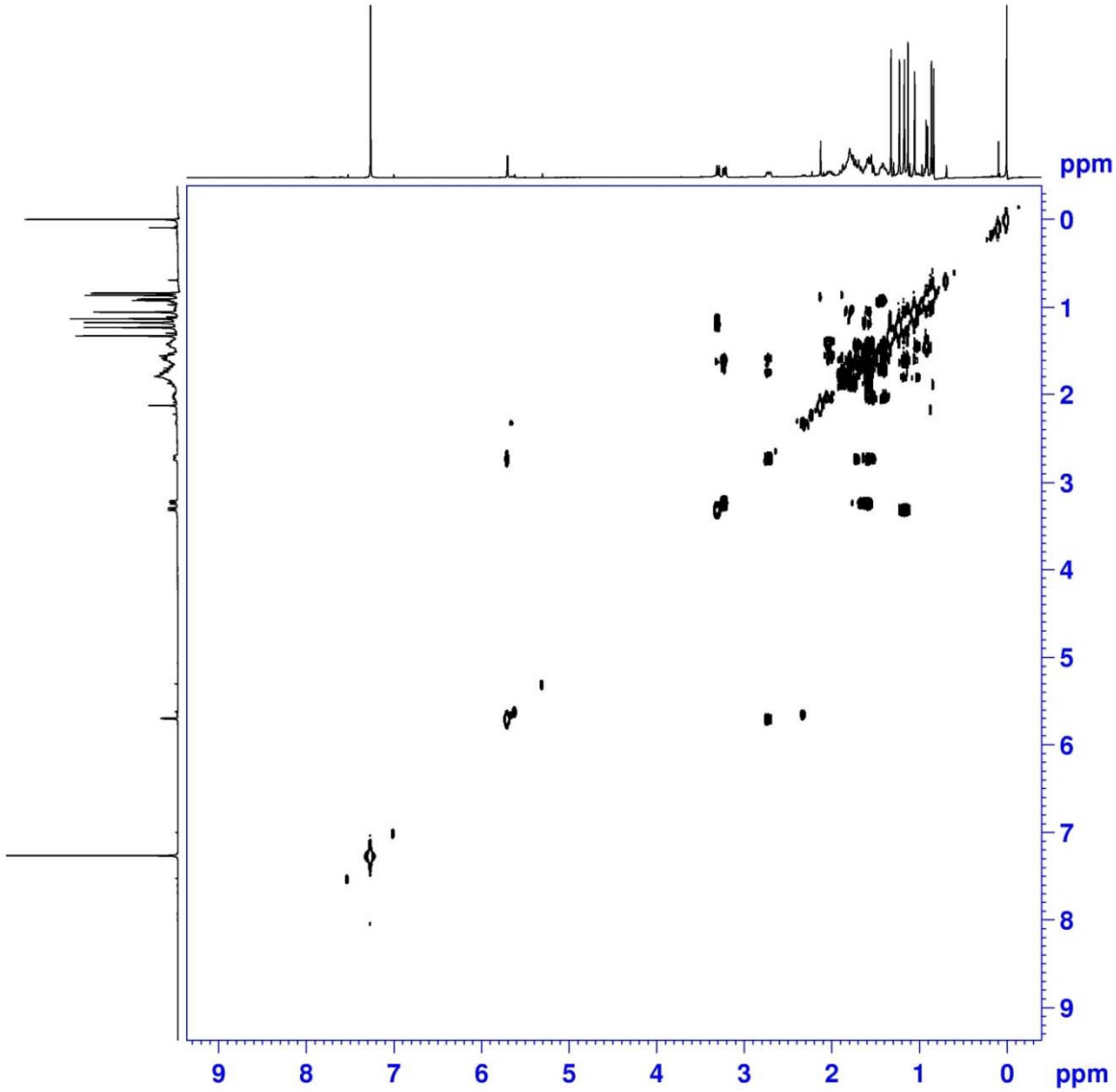



```
NAME                EA-17
EXPNO                 103
PROCNO                  1
Date_            20160727
Time                 1.12
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG        deptsp135
TD                  65536
SOLVENT             CDCl3
NS                    300
DS                      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 ========
SFO1         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 **7** in $CDCl_3$

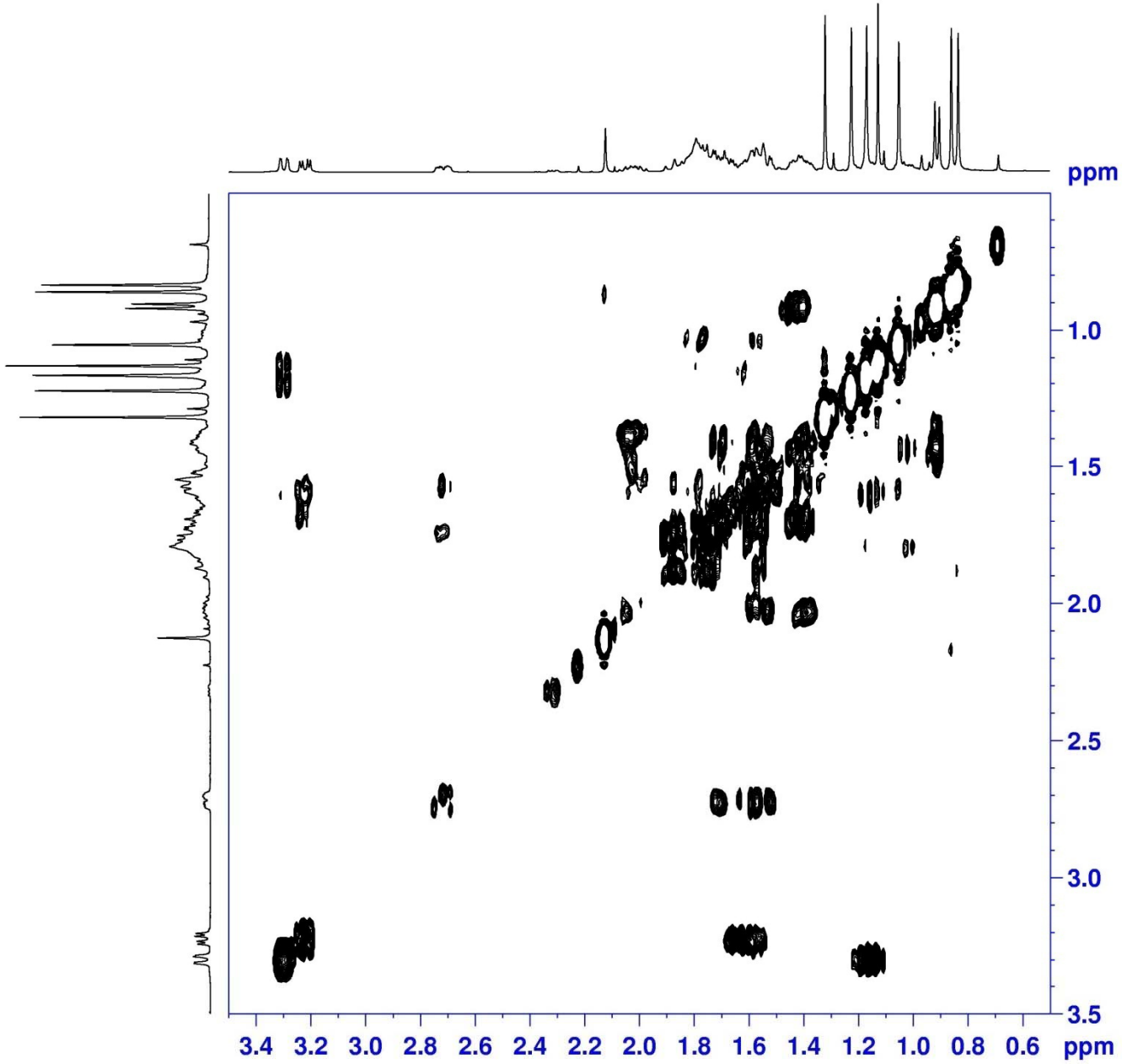


^{1}H-^{1}H COSY (400 MHz) spectrum of compound **7** in $CDCl_3$

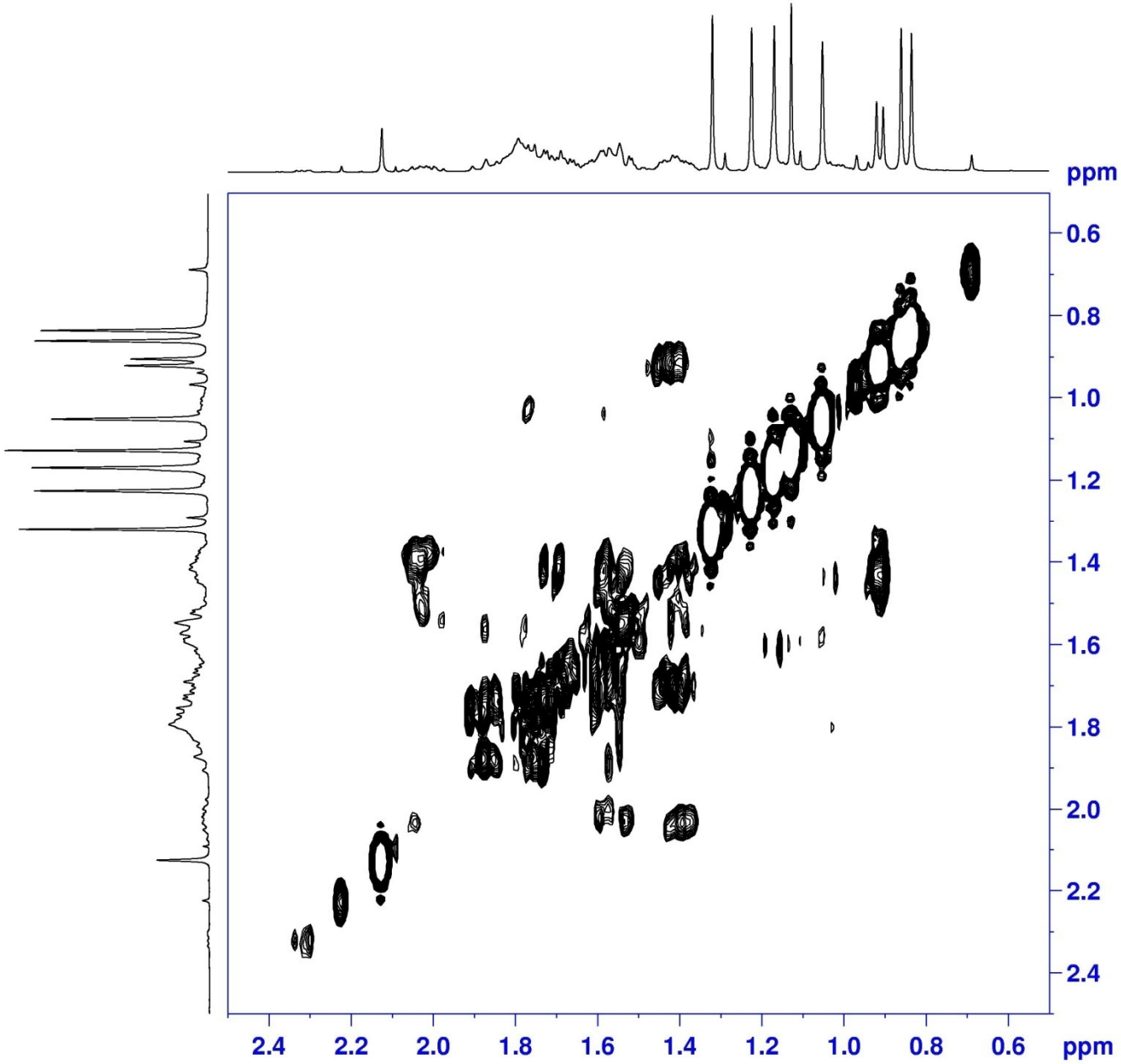



```
NAME                EA-17
EXPNO                 104
PROCNO                  1
Date_            20160805
Time                22.10
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG       cosygpppqf
TD                   2048
SOLVENT             CDC13
NS                      8
DS                      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            400.1300058 MHz
WDW                 QSINE
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
SI                   1024
MC2                    QF
SF            400.1300047 MHz
WDW                 QSINE
SSB                     0
```

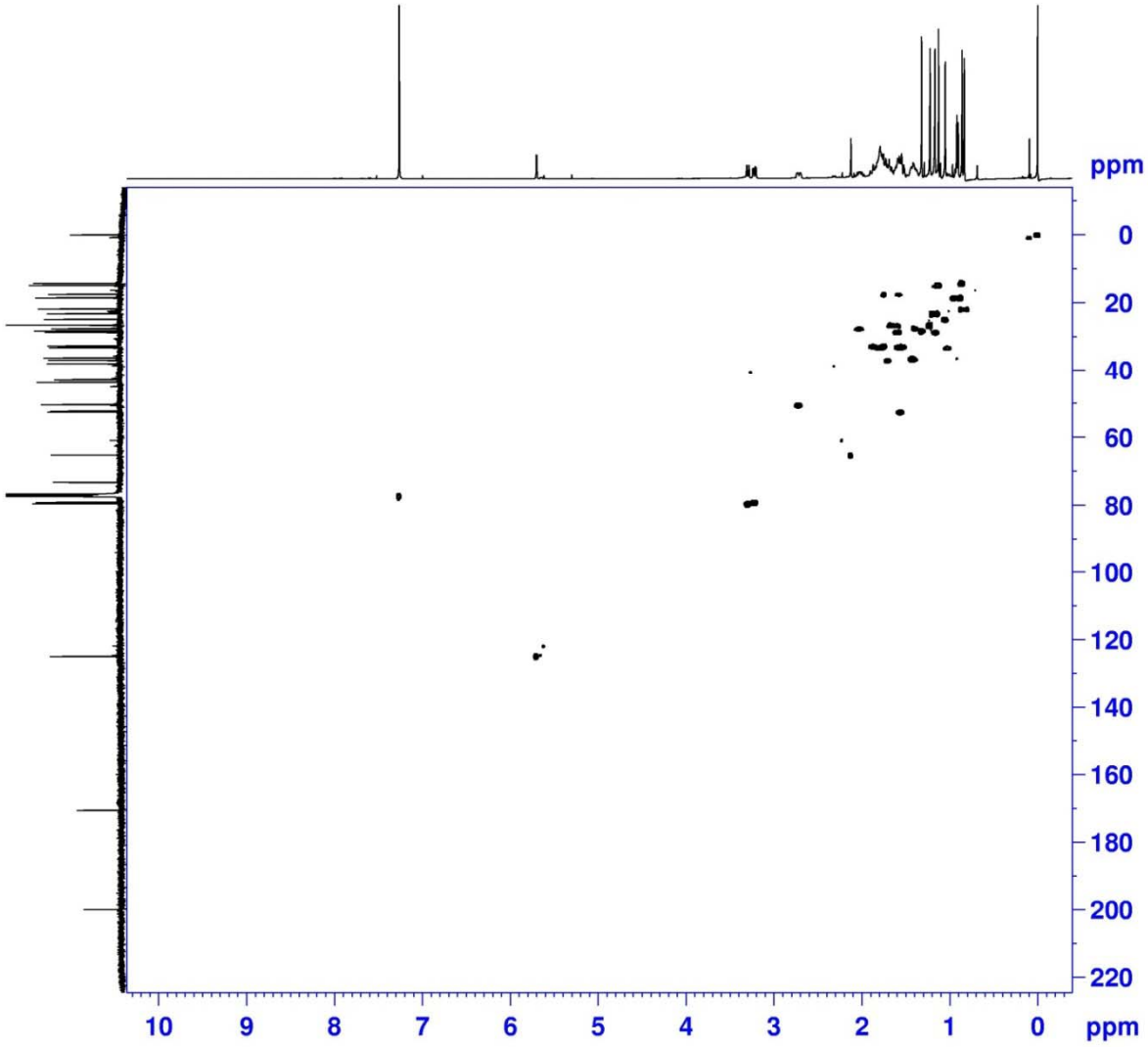
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **7** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **7** in $CDCl_3$

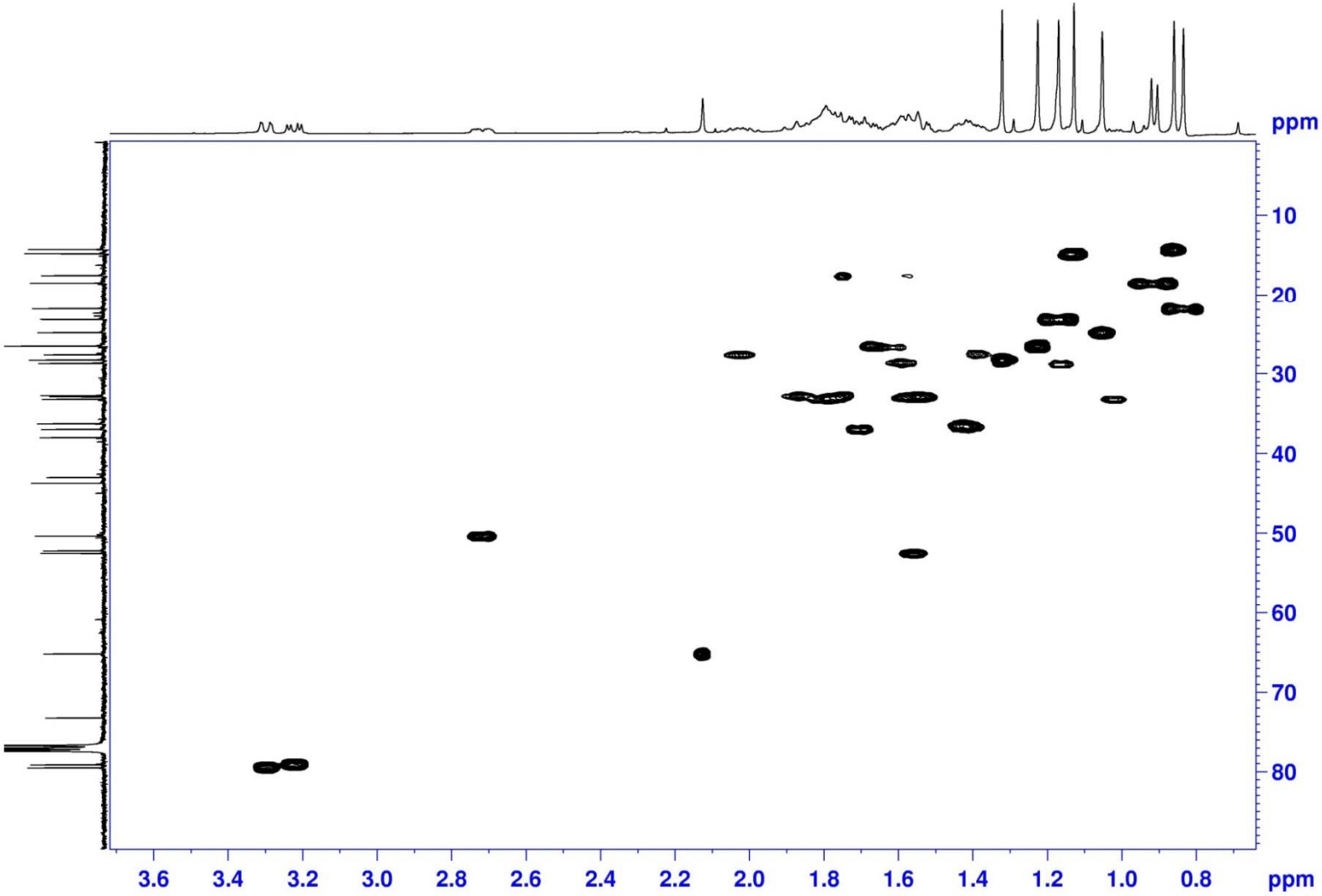


HSQC (400 MHz) spectrum of compound **7** in $CDCl_3$

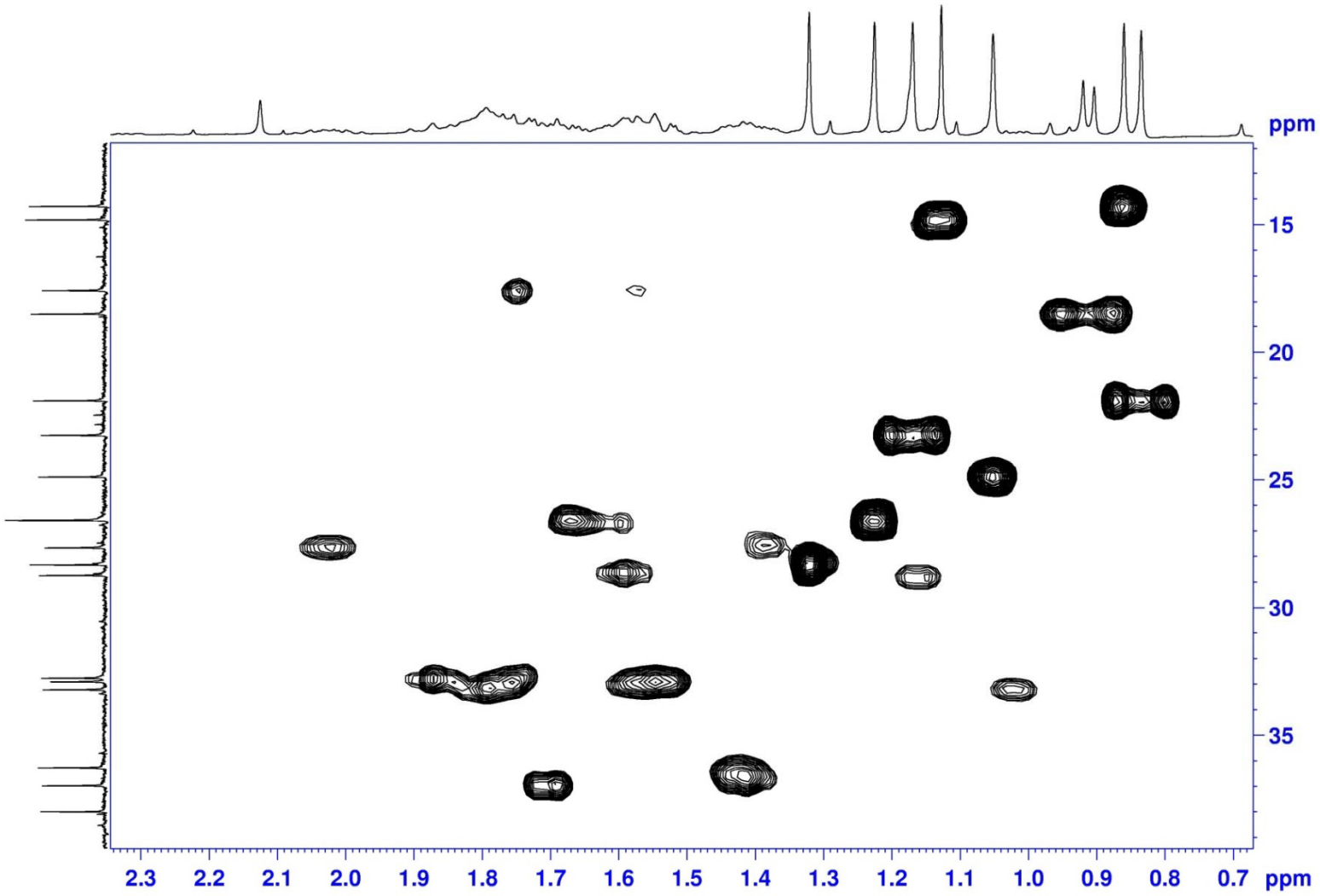



```
NAME                EA-17
EXPNO                 105
PROCNO                  1
Date_            20160805
Time                22.50
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      hsqcetgpsi2
TD                   1024
SOLVENT             CDCl3
NS                     16
DS                     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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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           400.1300061 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2        echo-antiecho
SF           100.6127554 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
```

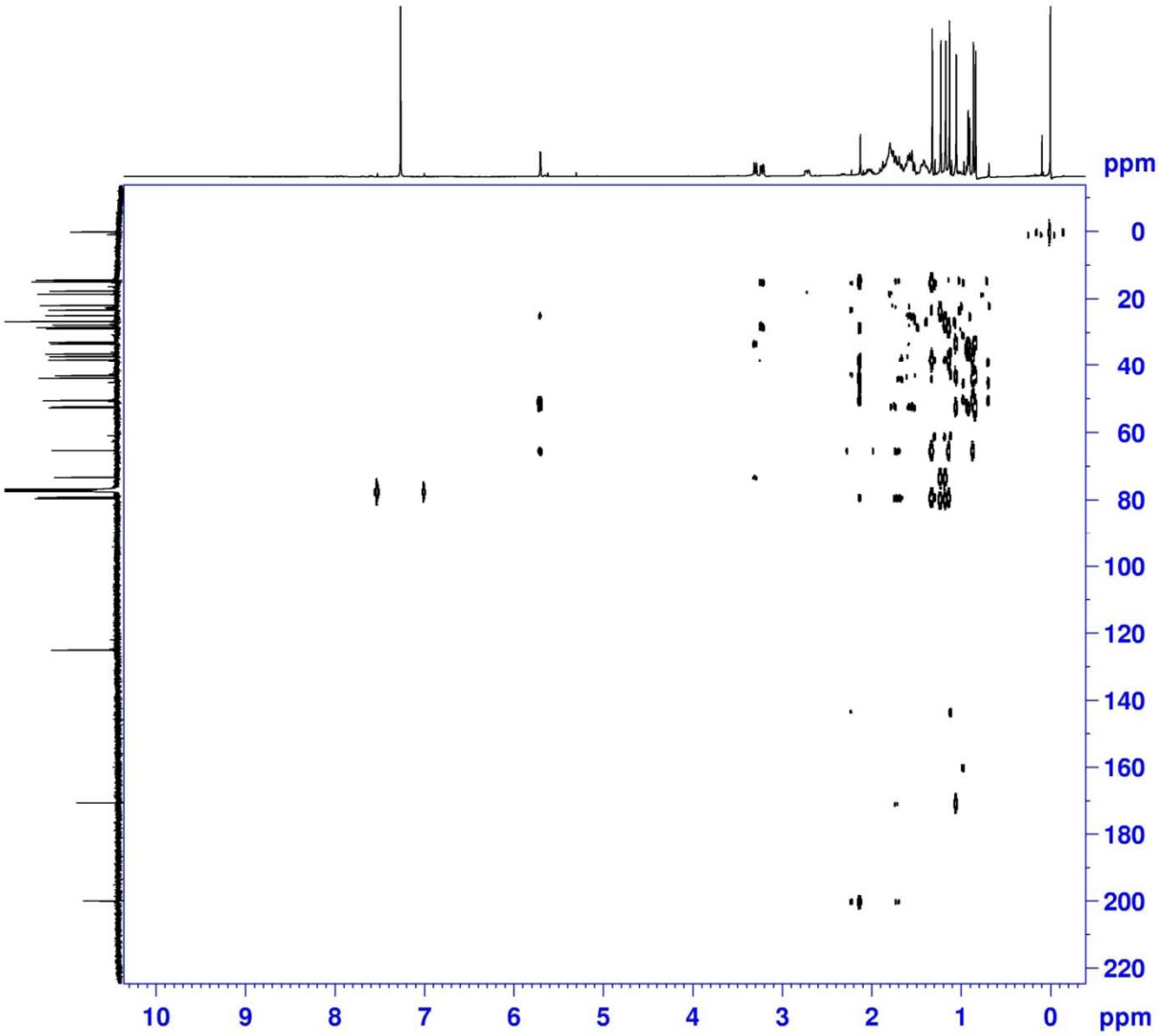
HSQC (400 MHz) spectrum of compound **7** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **7** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$




```
NAME                EA-17
EXPNO                 106
PROCNO                  1
Date_            20160806
Time                 0.41
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG     hmbcgplpndqf
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 ========
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
SI                  2048
SF           400.1300075 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           100.6127394 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
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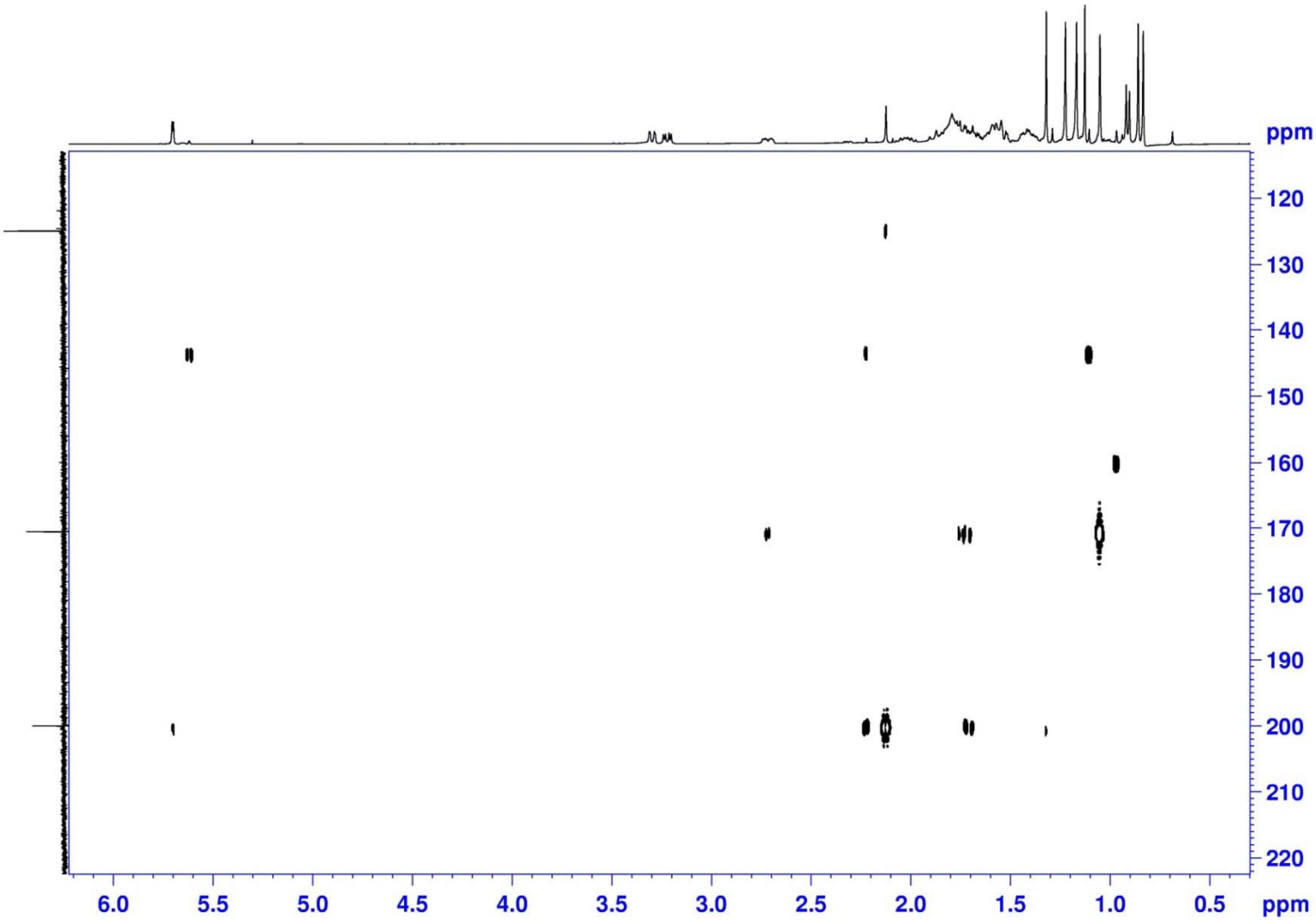
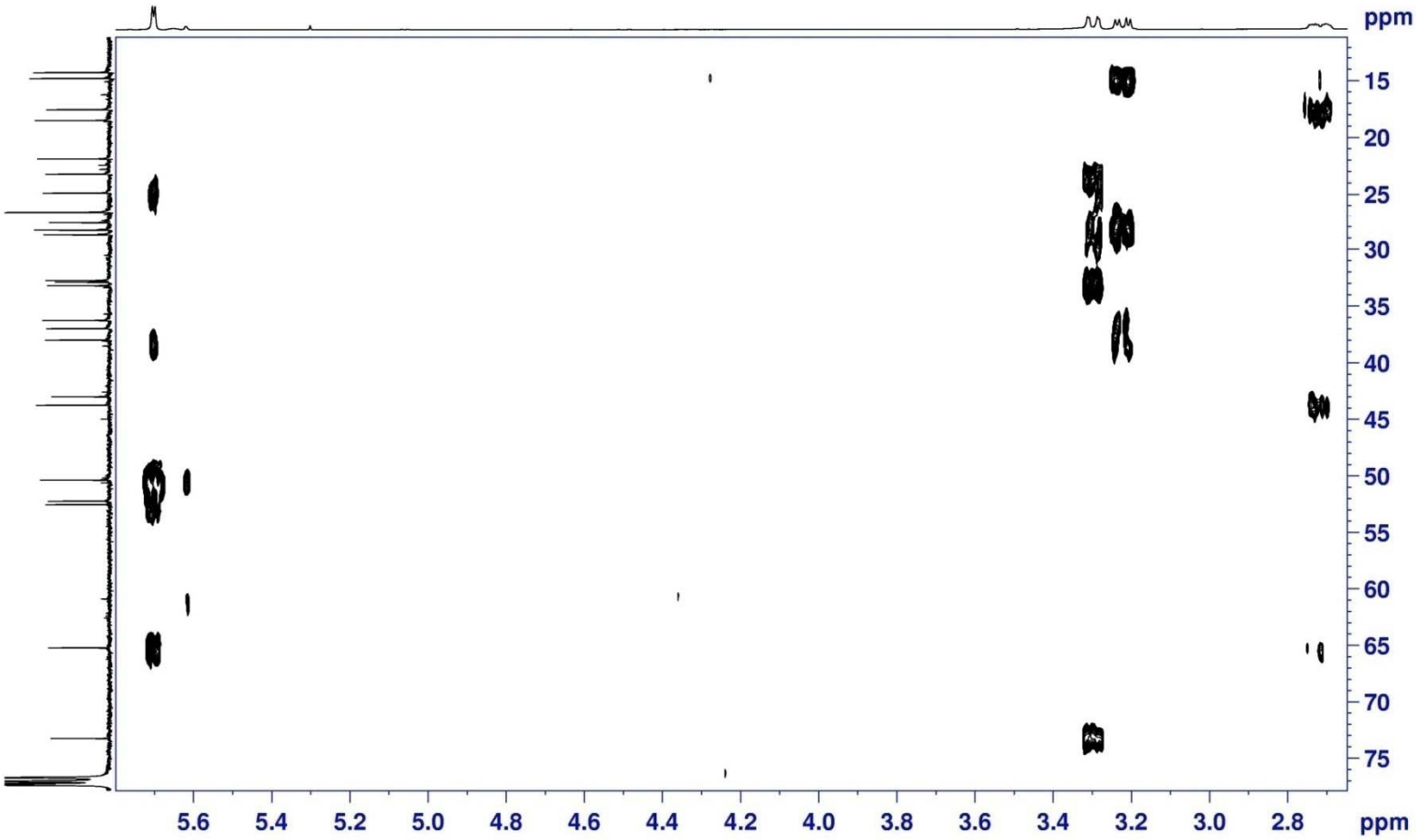
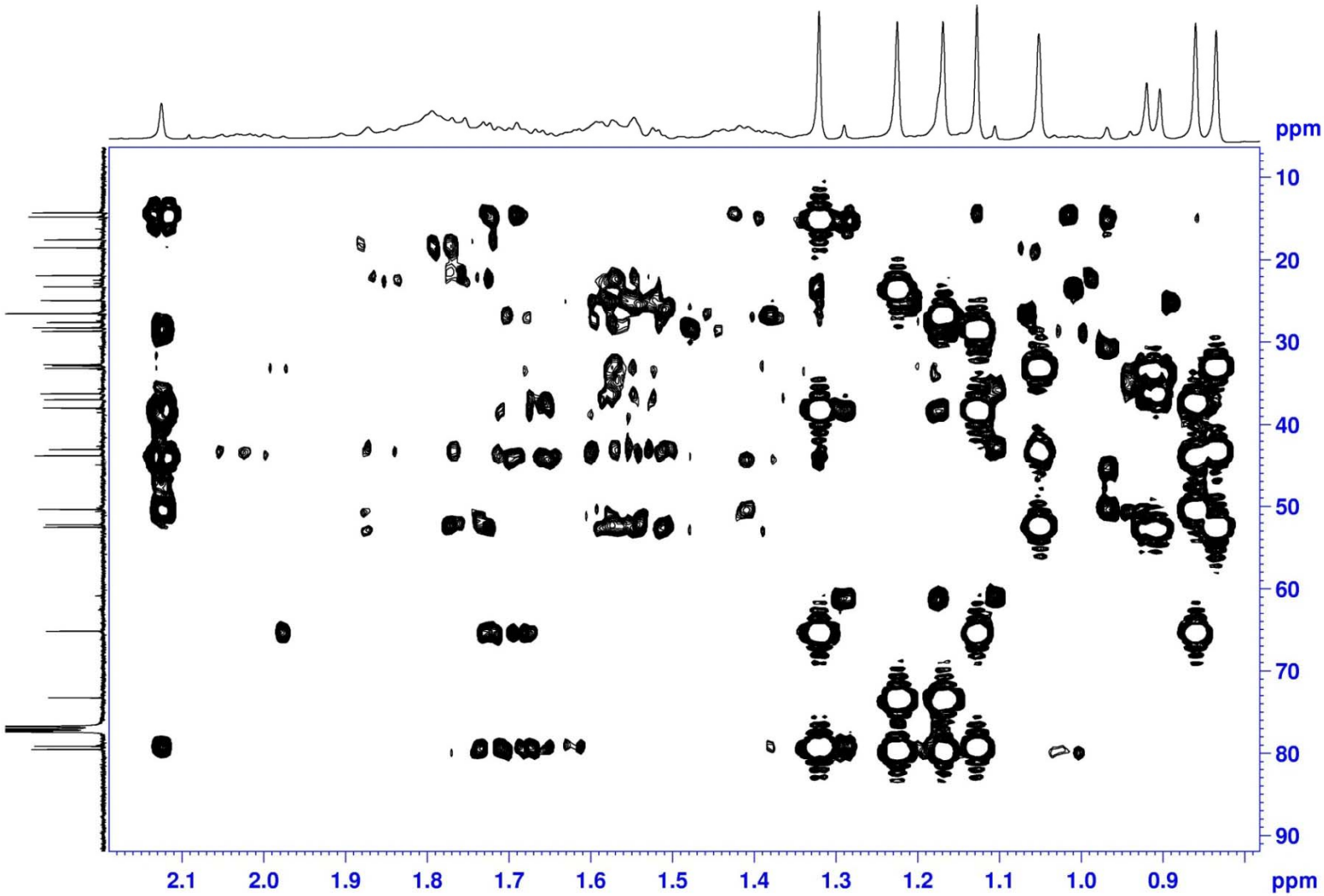
HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$



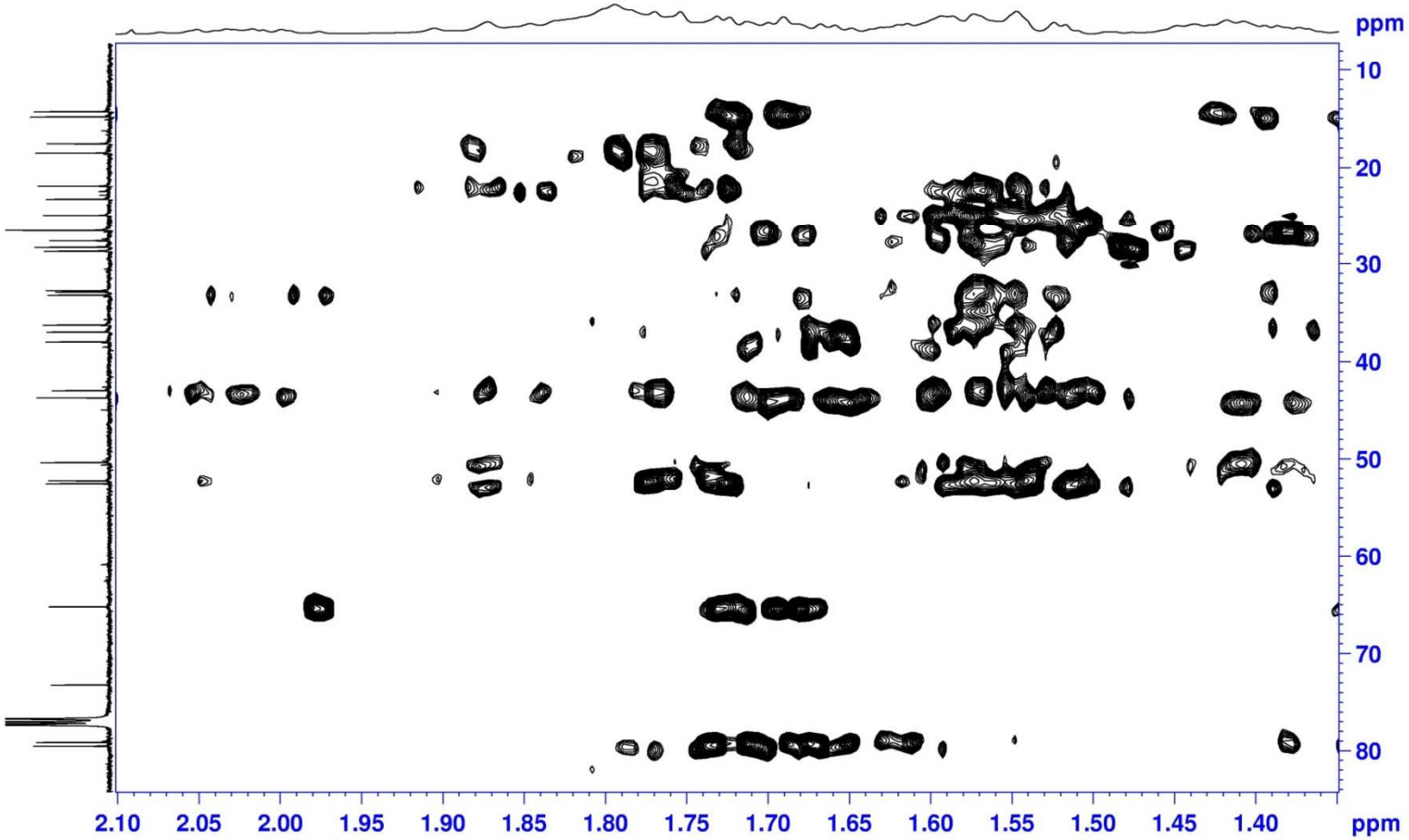
HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$



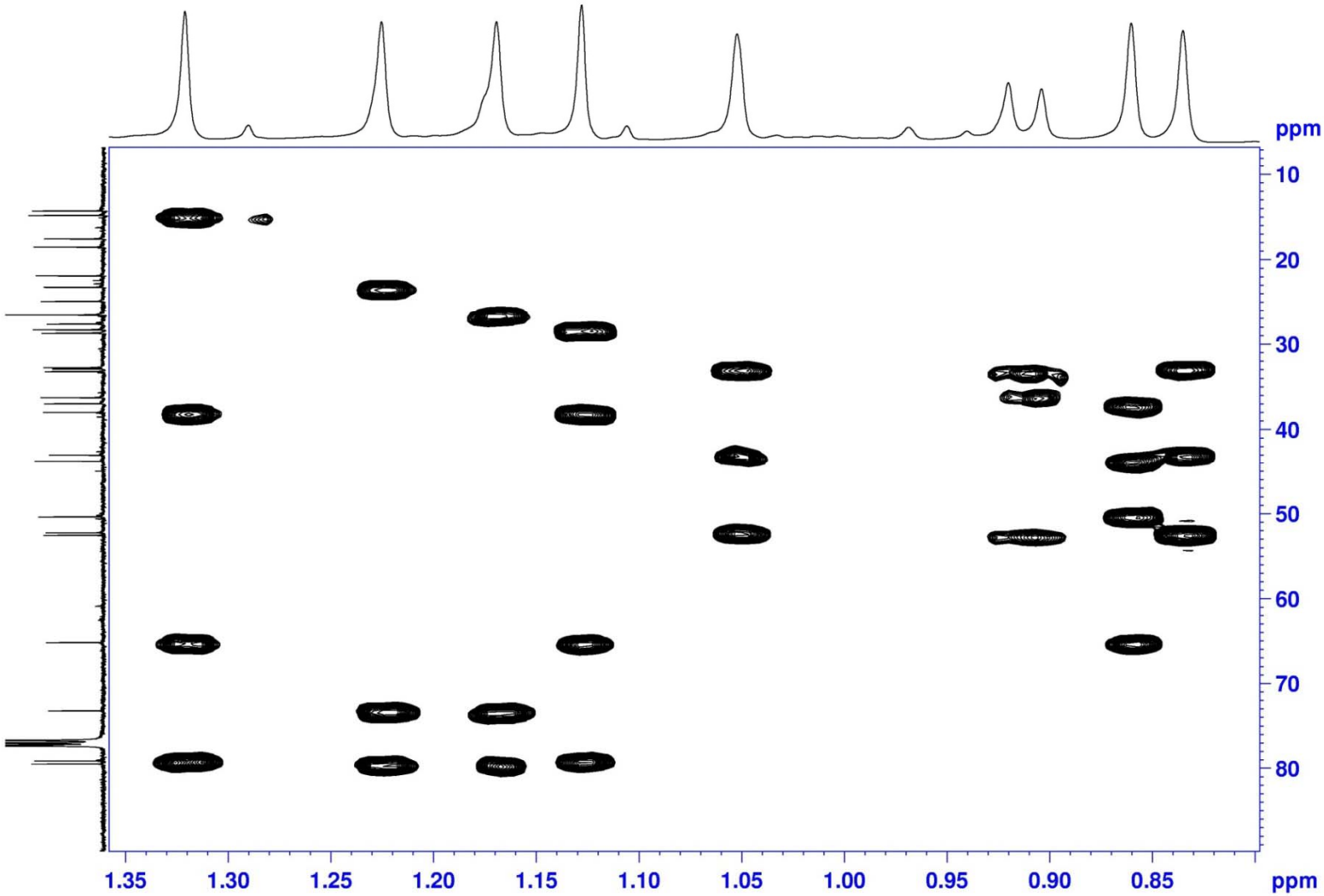
HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$



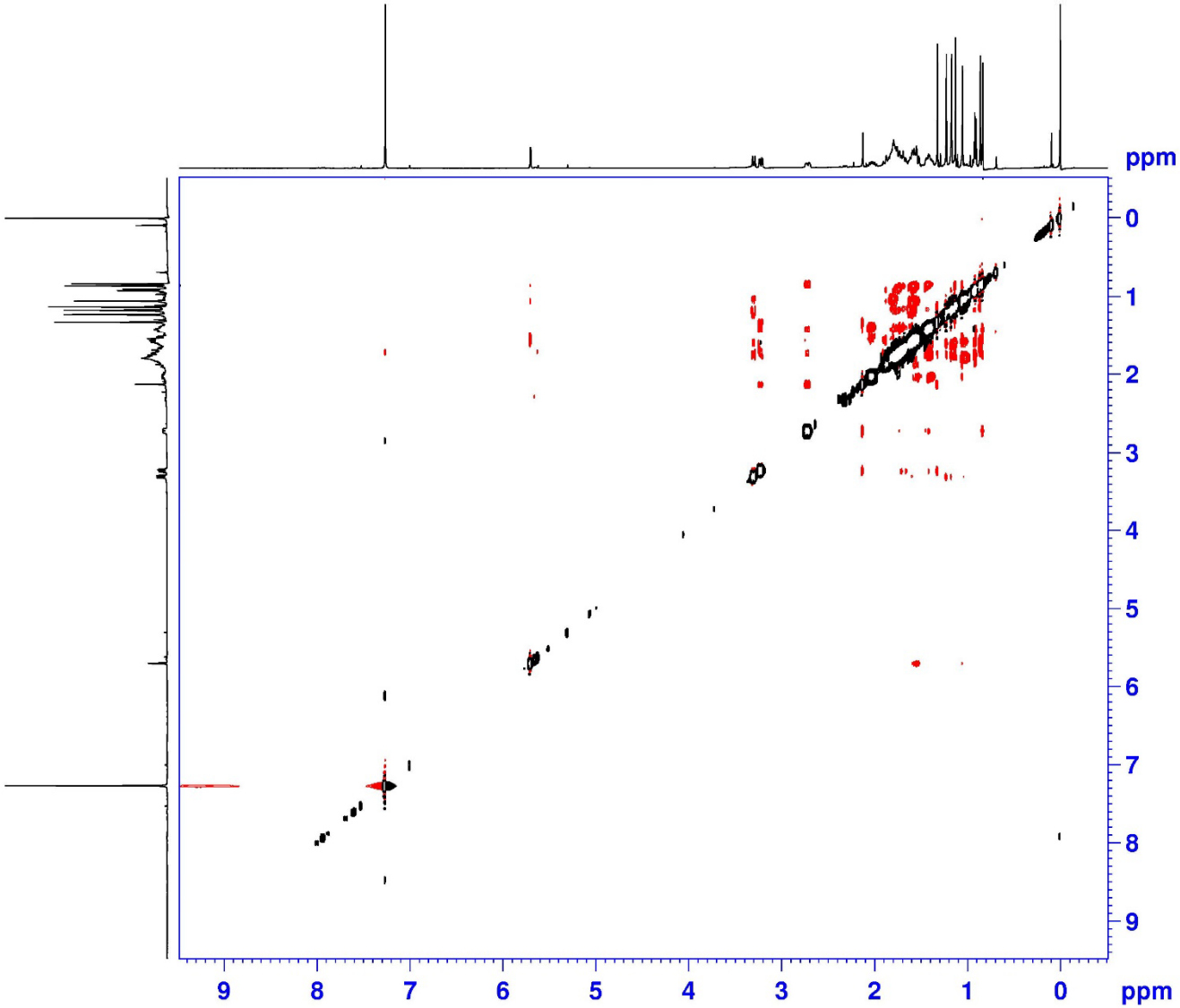
HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **7** in $CDCl_3$



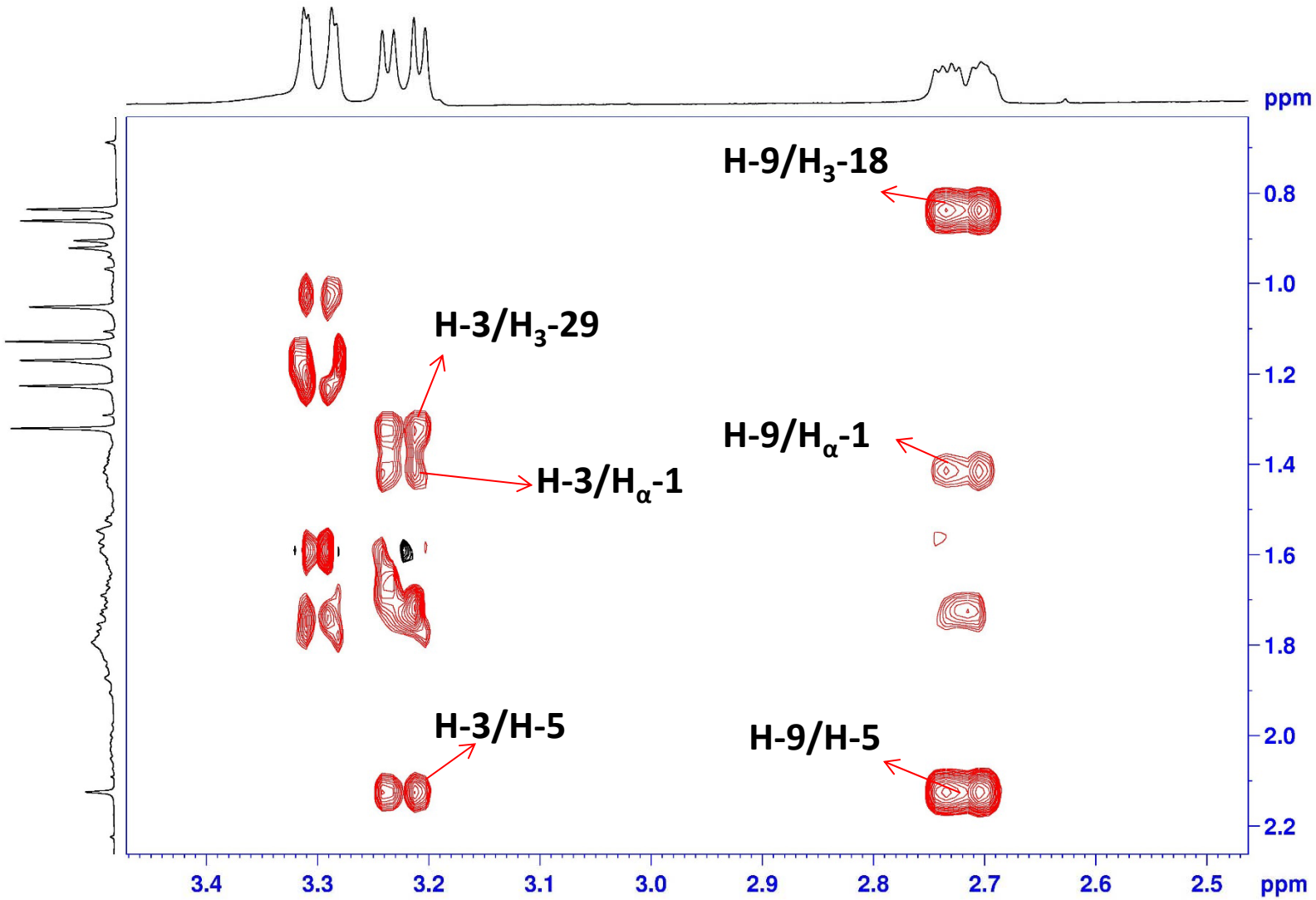
NOESY (400 MHz) spectrum of compound **7** in $CDCl_3$



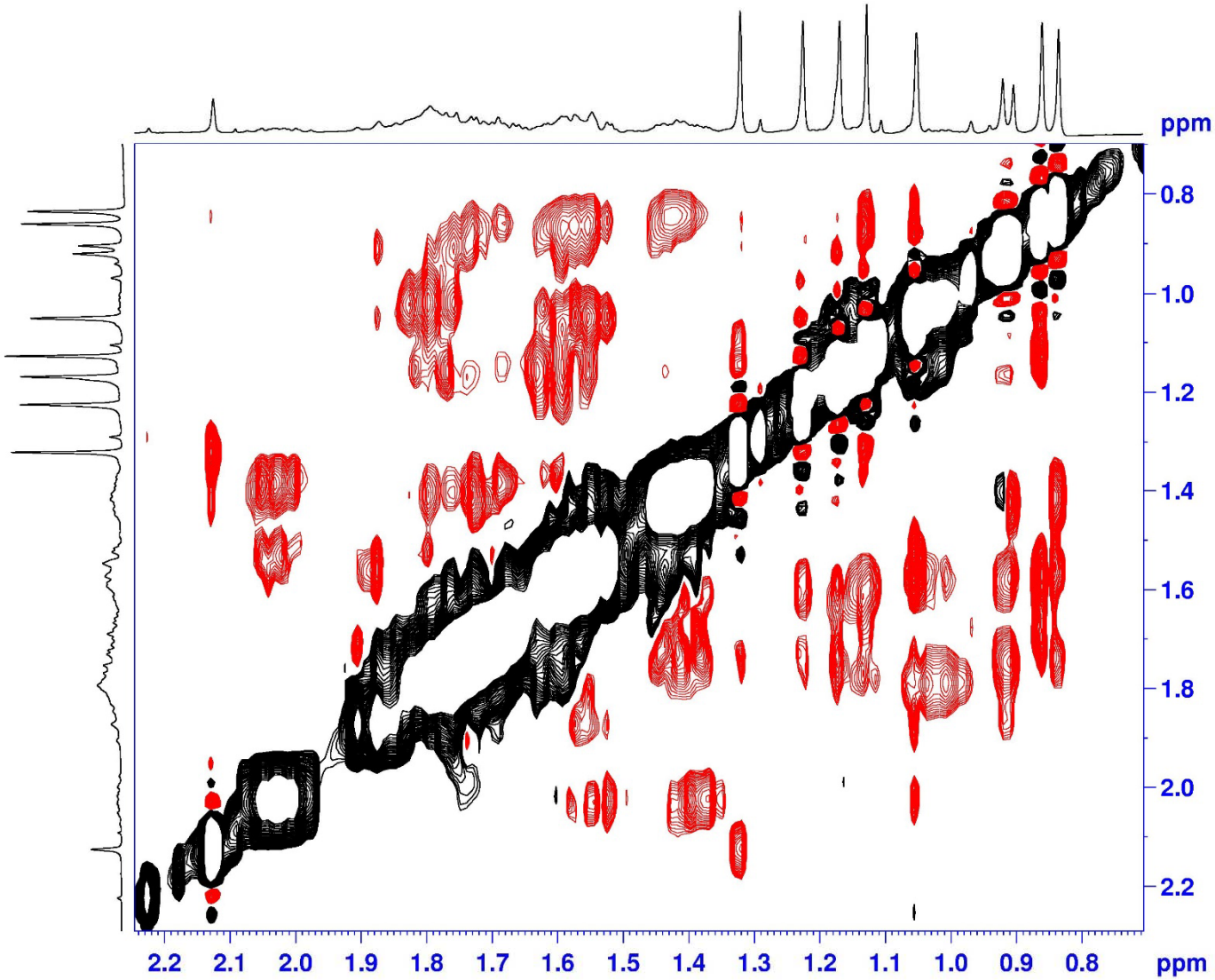

```
NAME                EA 17
EXPNO                 107
PROCNO                  1
Date_            20160806
Time                 2.56
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDC13
NS                     16
DS                     32
SWH             4000.000 Hz
FIDRES          1.953125 Hz
AQ             0.2560500 sec
RG                 208.5
DW               125.000 use
DE                 10.00 use
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 use
P2                 23.00 use
P17              2500.00 use
ND0                    1
TD                   256
SFO1            400.1318 MHz
FIDRES         15.625000 Hz
SW                 9.997 ppm
FnMODE       States-TPPI
SI                  1024
SF           400.1300055 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00
SI                  1024
MC2          States-TPPI
SF           400.1300064 MHz
WDW                 QSINE
```

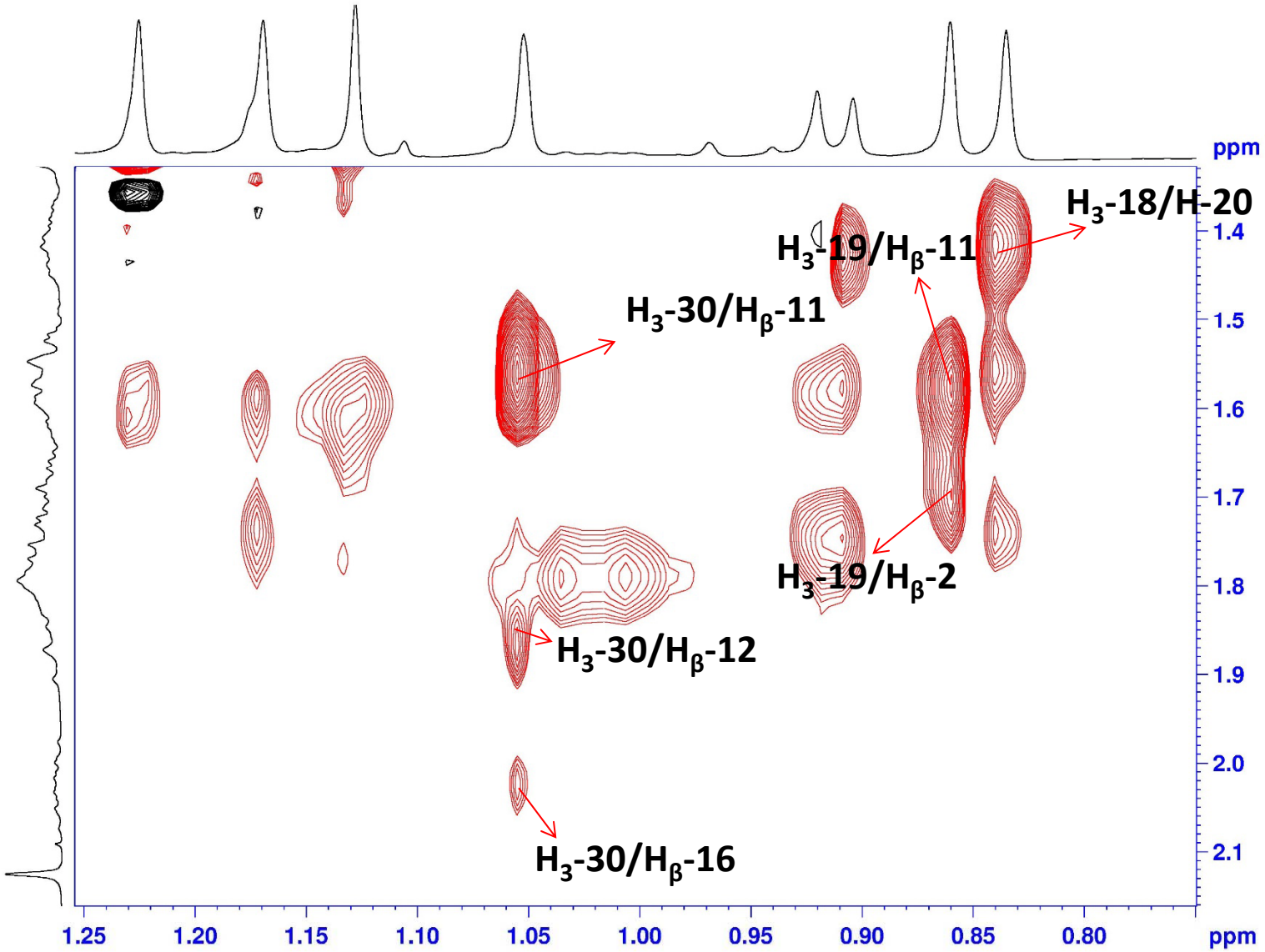
NOESY (400 MHz) spectrum of compound **7** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **7** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **7** in $CDCl_3$



HR-ESIMS for compound **8**

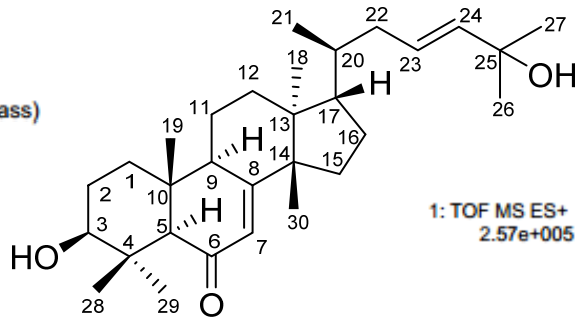
Elemental Composition Report **Page 1**

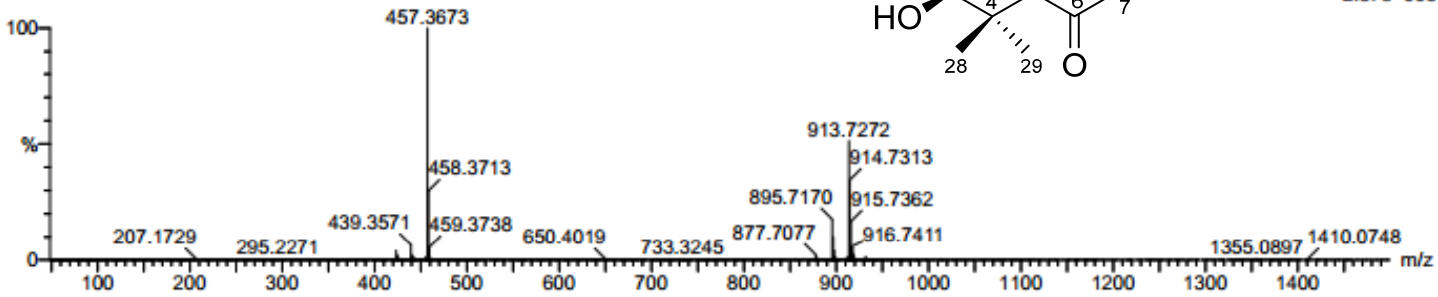
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
360 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-50 H: 0-100 N: 0-1 O: 0-20 Na: 0-1

EA3-44-3-1
201807308 258 (2.075)

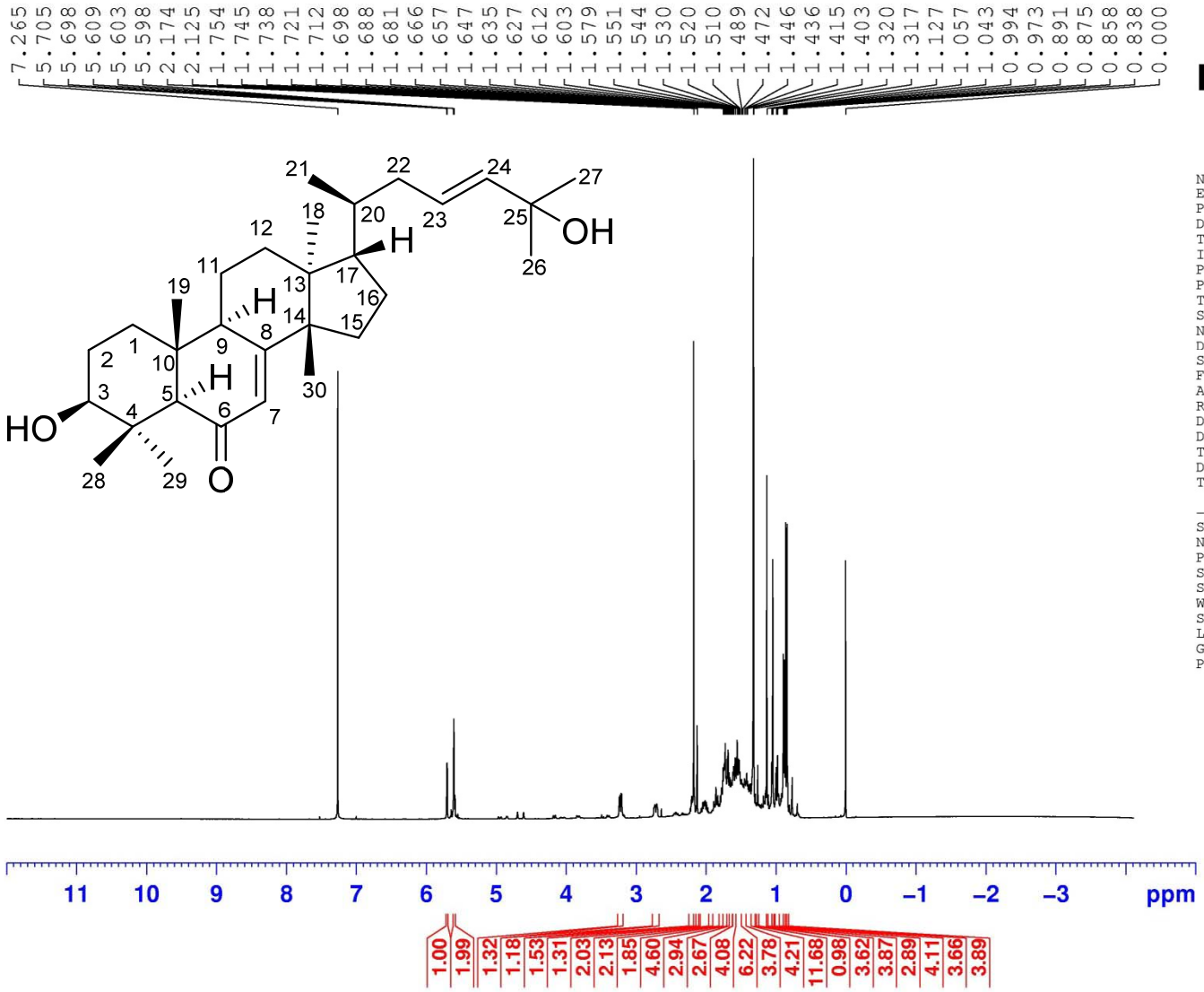
$[M+H]^+$





Minimum:				-1.5				
Maximum:		5.0	5.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
457.3673	457.3682	-0.9	-2.0	6.5	415.9	2.086	12.41	C30 H49 O3
	457.3658	1.5	3.3	3.5	414.0	0.133	87.59	C28 H50 O3 Na

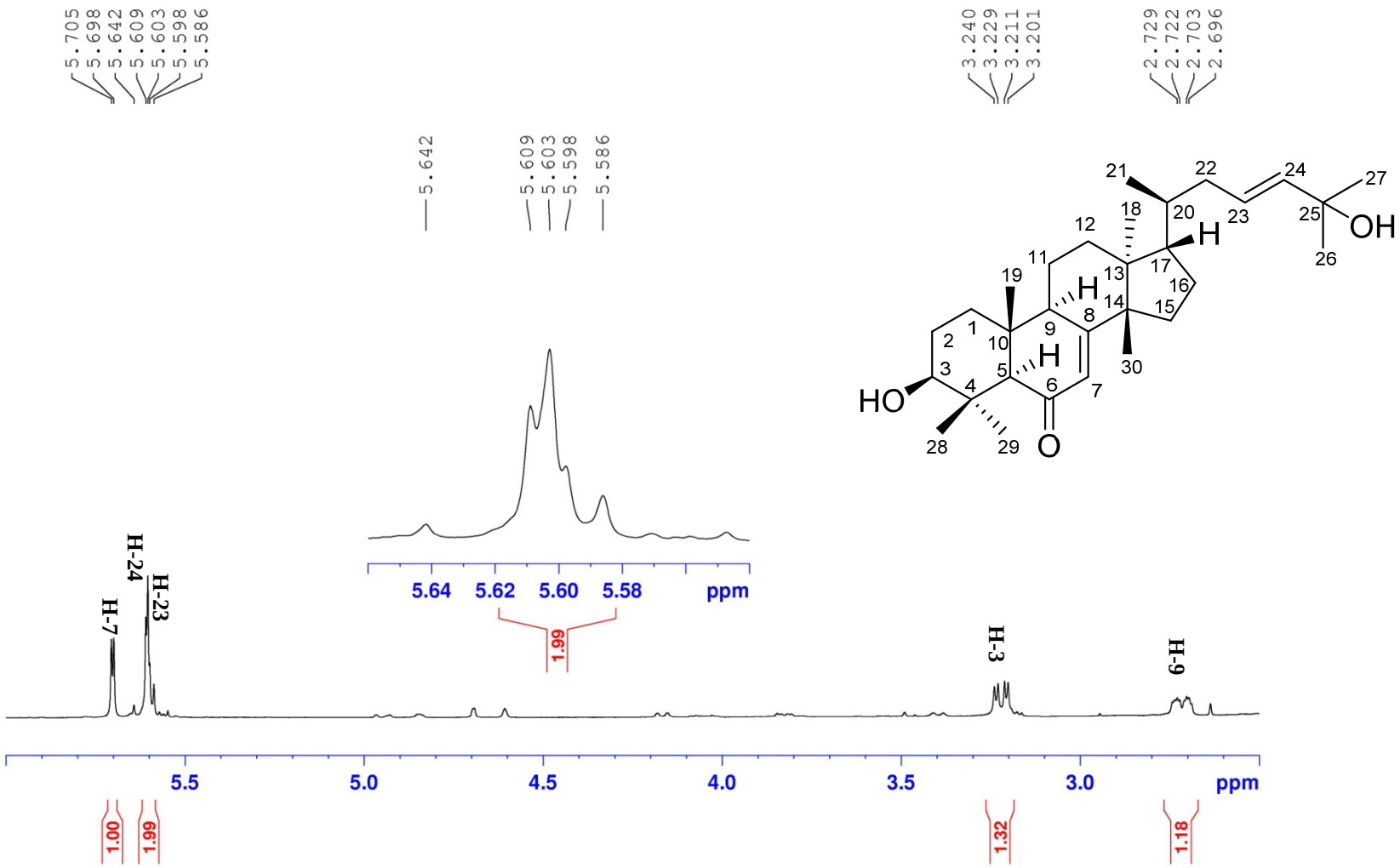
^{1}H NMR (400 MHz) spectrum of compound **8** in $CDCl_3$



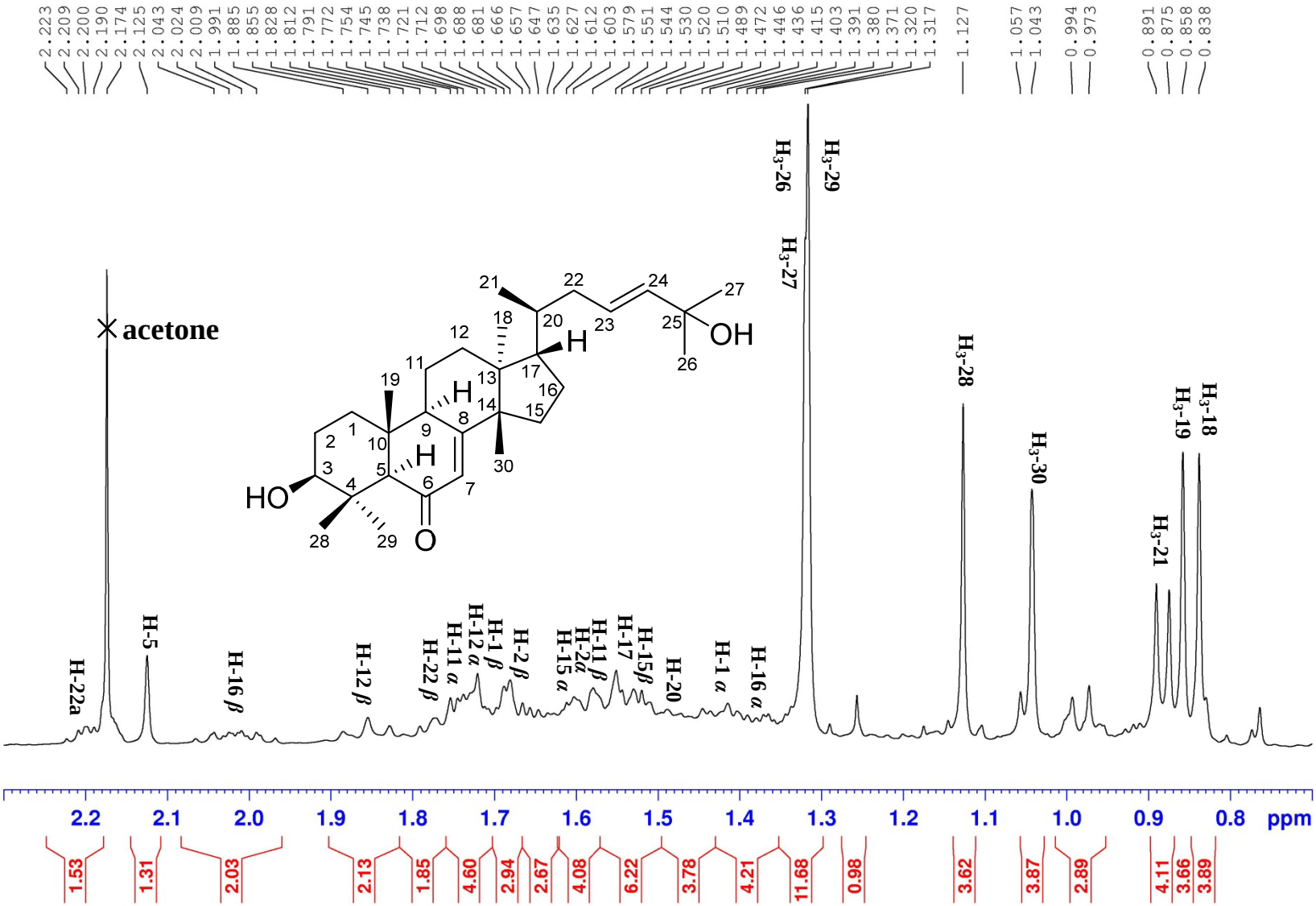

```
NAME          EA3-44-3-1
EXPNO                  1
PROCNO                 1
Date_           20171106
Time               22.27
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                147.94
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                 11.50 usec
SI                 65536
SF           400.1300076 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

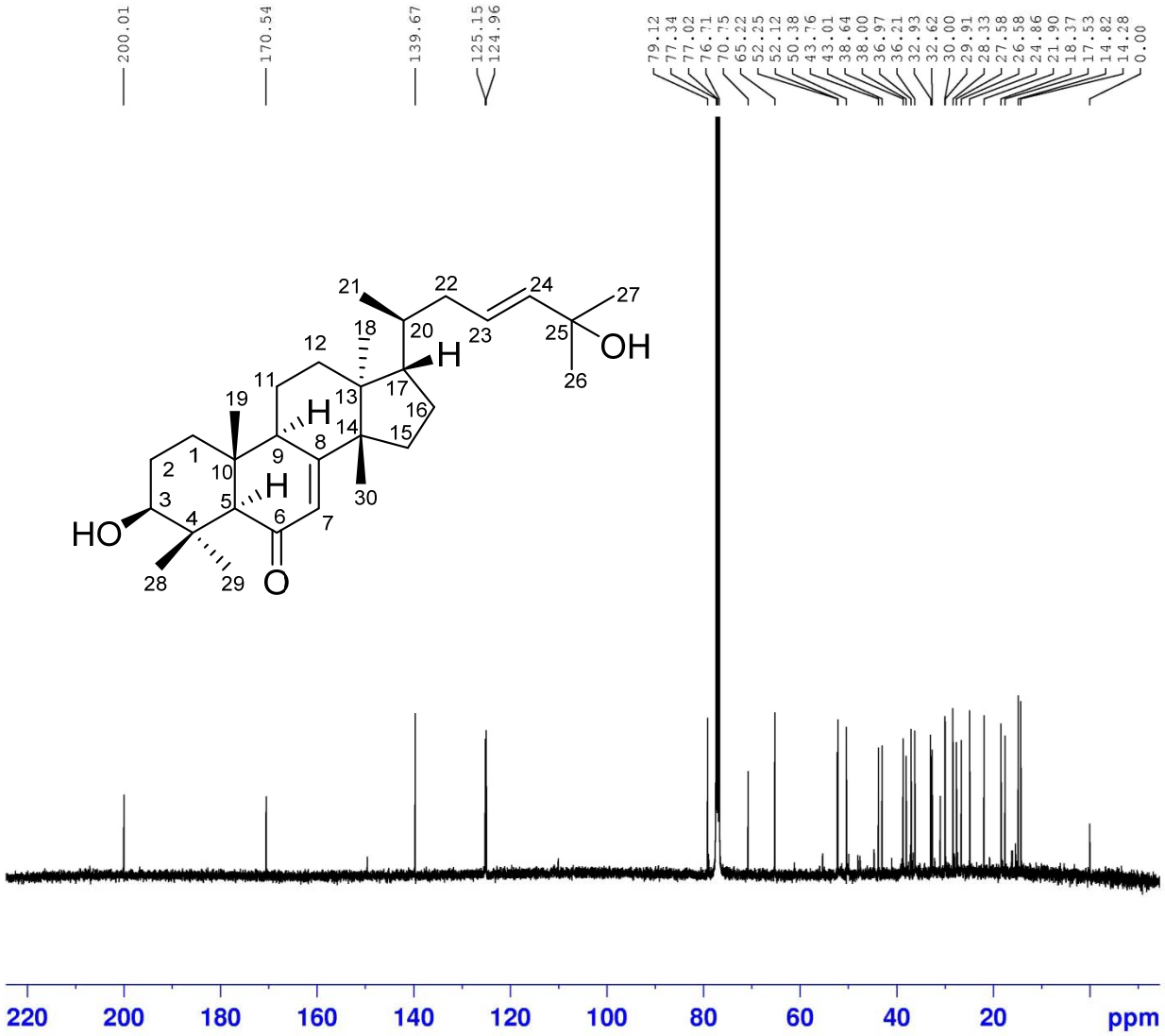
^{1}H NMR (400 MHz) spectrum of compound **8** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **8** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **8** in $CDCl_3$

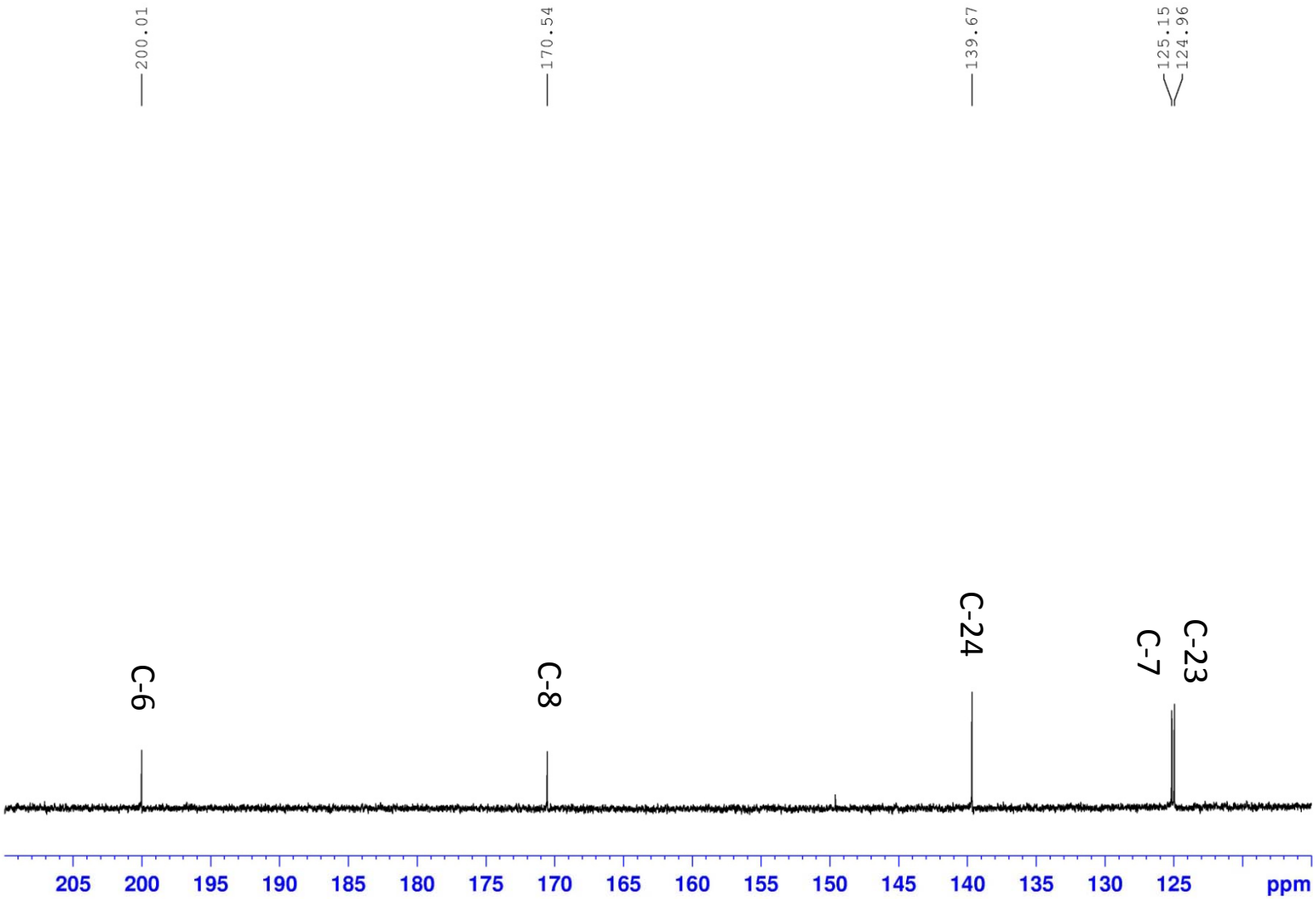


BRUKER

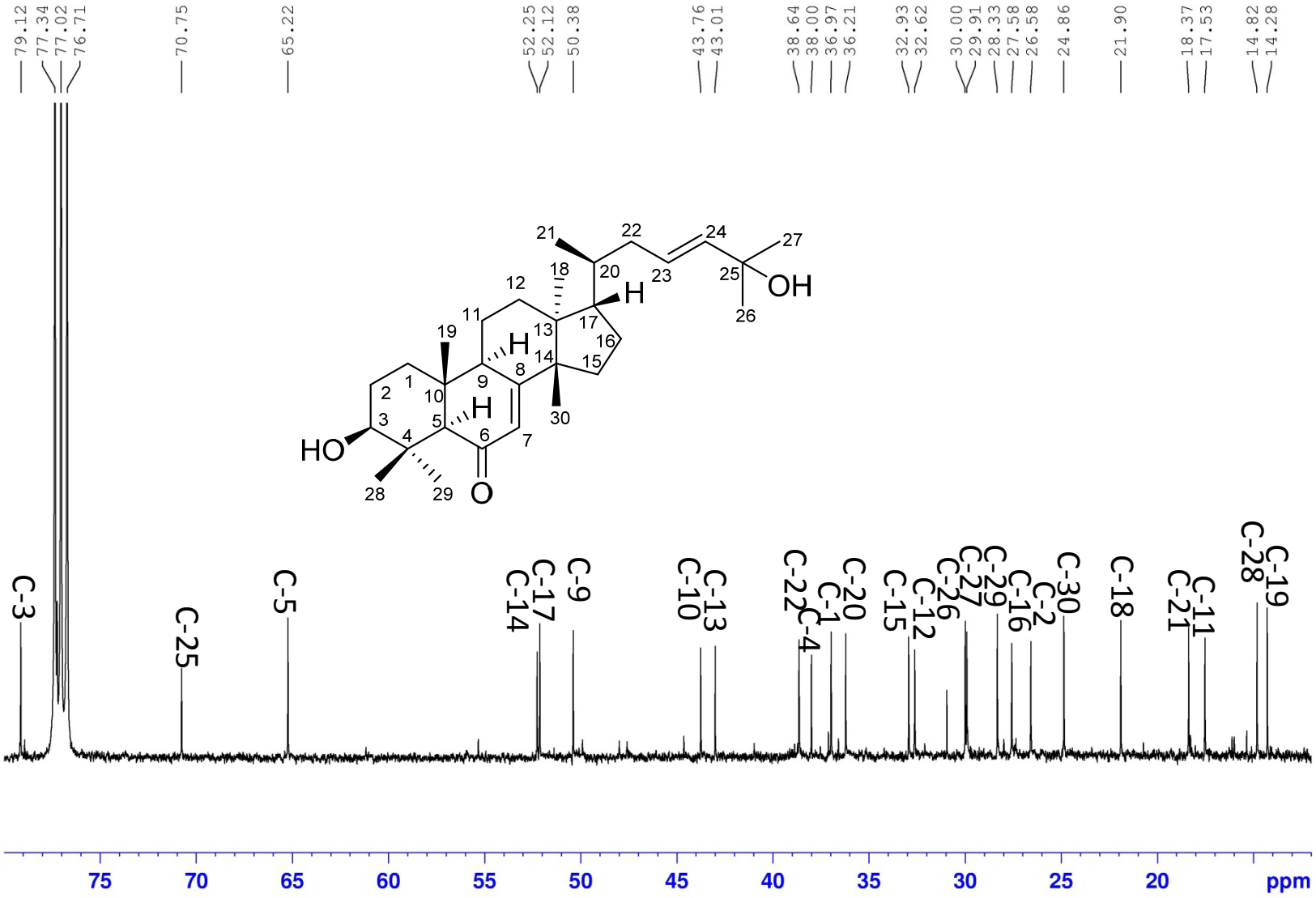
```
NAME          EA3-44-3-1
EXPNO                  2
PROCNO                 1
Date_           20171107
Time                2.49
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                117.37
DW                20.800 usec
DE                 18.00 usec
TE                 297.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

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

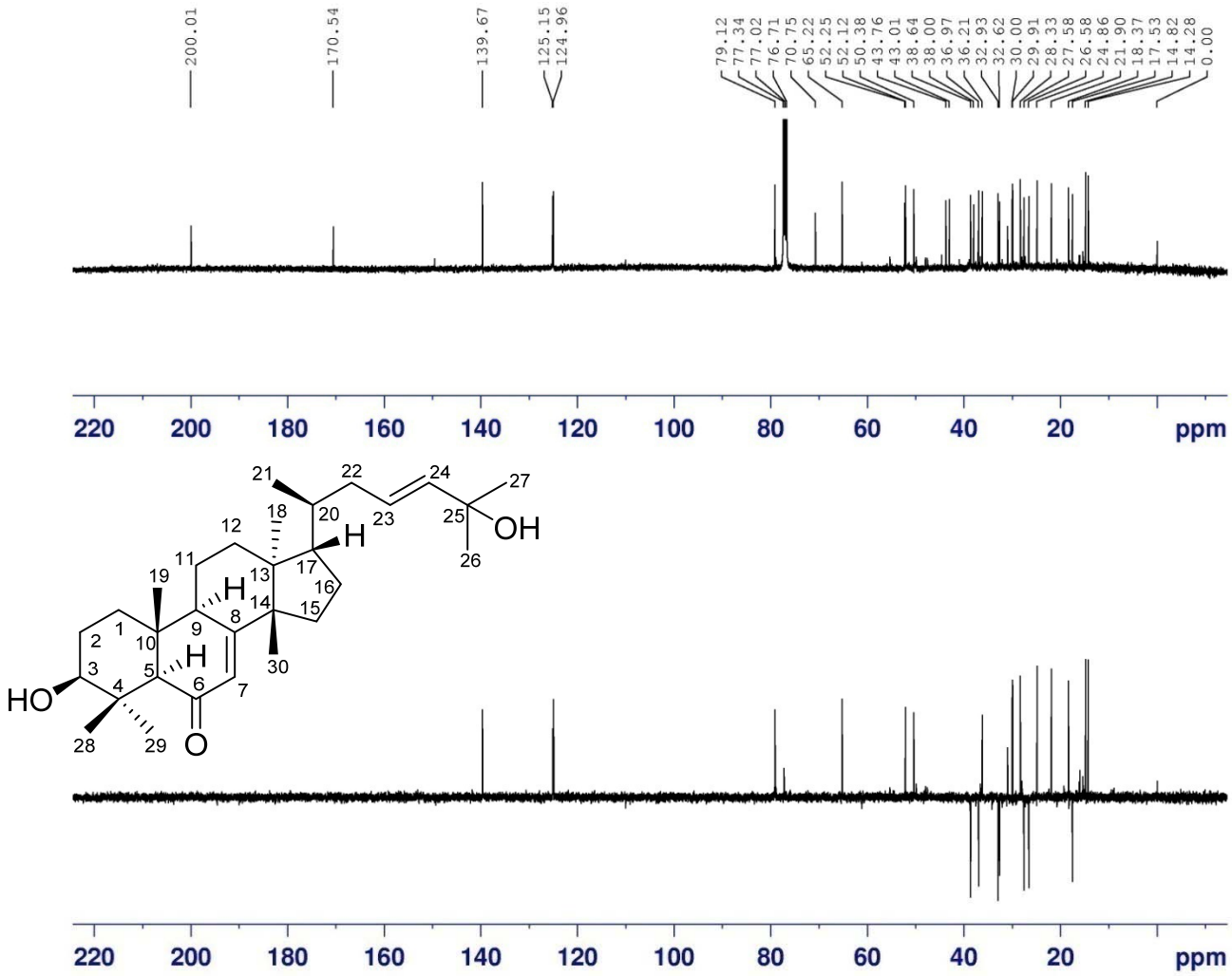
^{13}C NMR (100 MHz) spectrum of compound **8** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **8** in $CDCl_3$



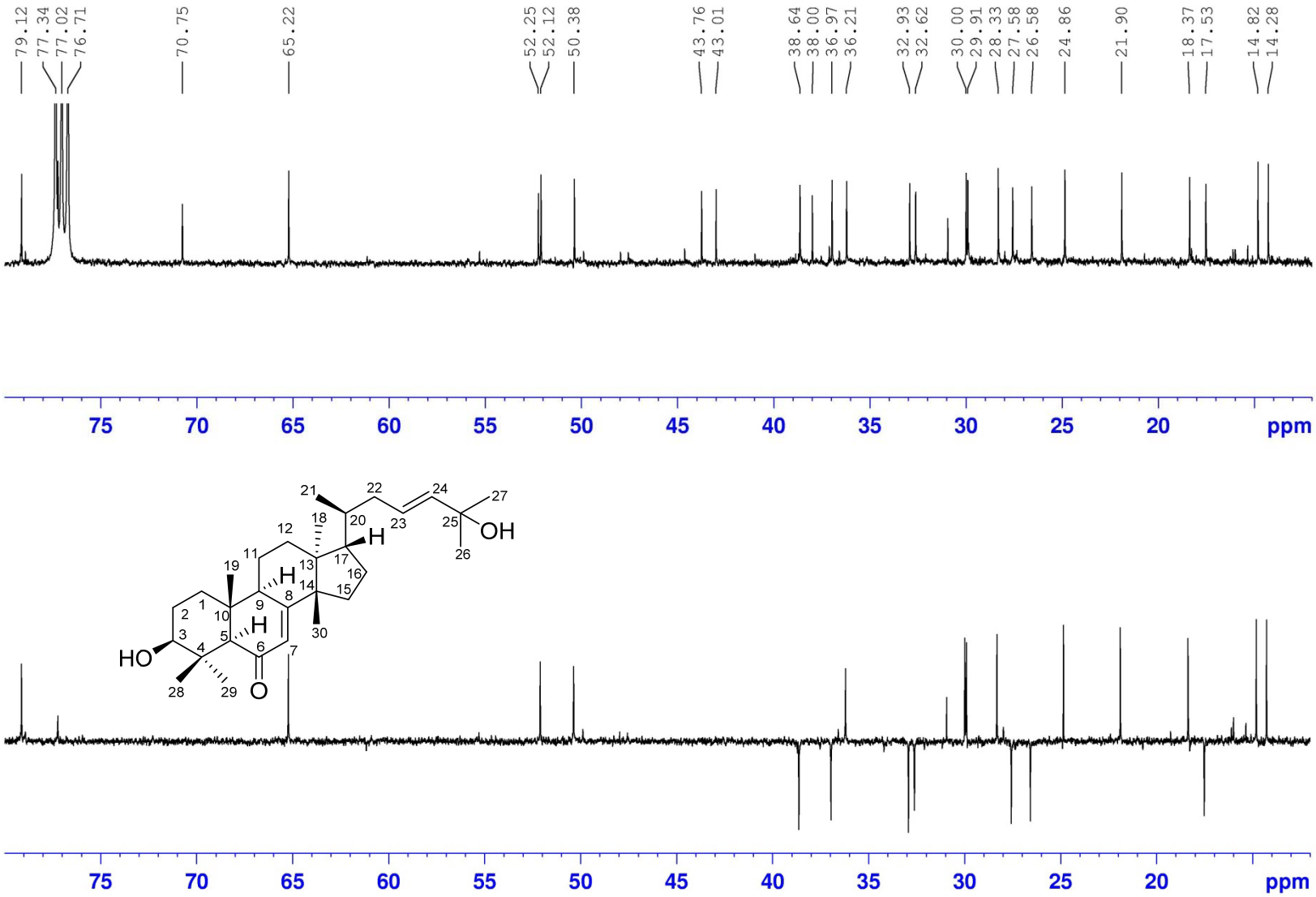
DEPT135 (100 MHz) spectrum of compound **8** in $CDCl_3$



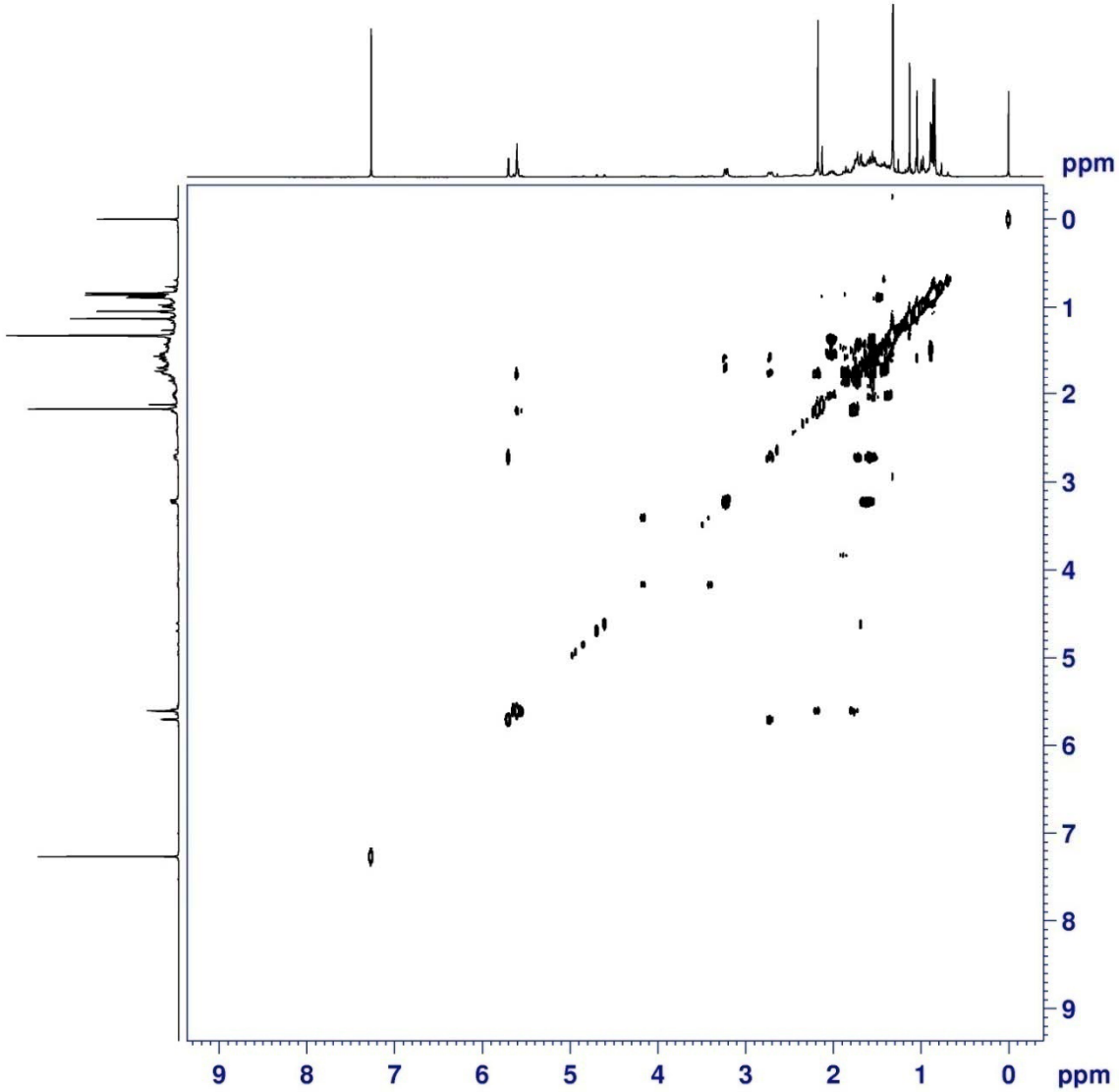

```
NAME          EA3-44-3-1
EXPNO                  3
PROCNO                 1
Date_           20171107
Time                3.07
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG        deptsp135
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     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 ========
SFO1        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 **8** in $CDCl_3$



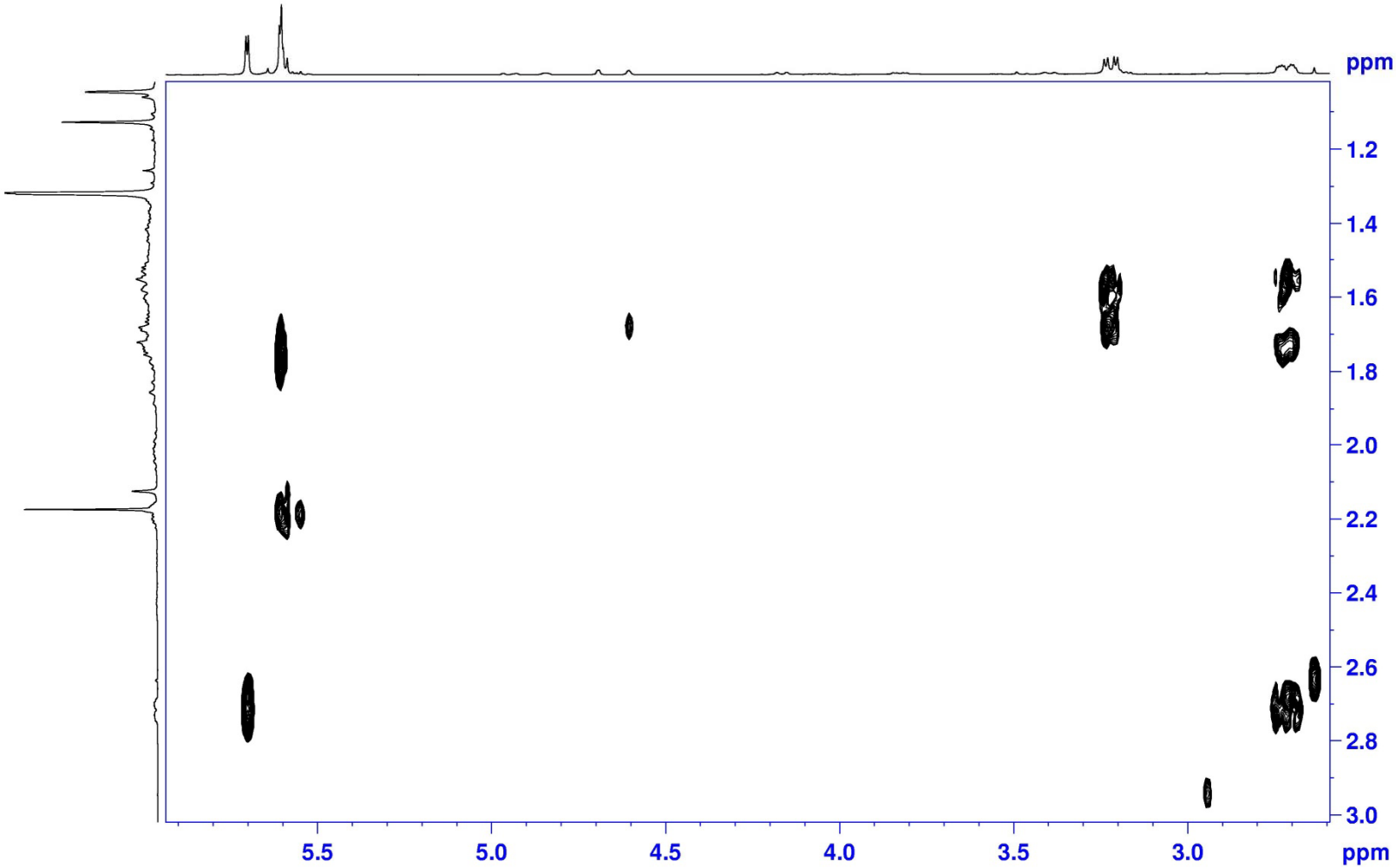
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **8** in $CDCl_3$



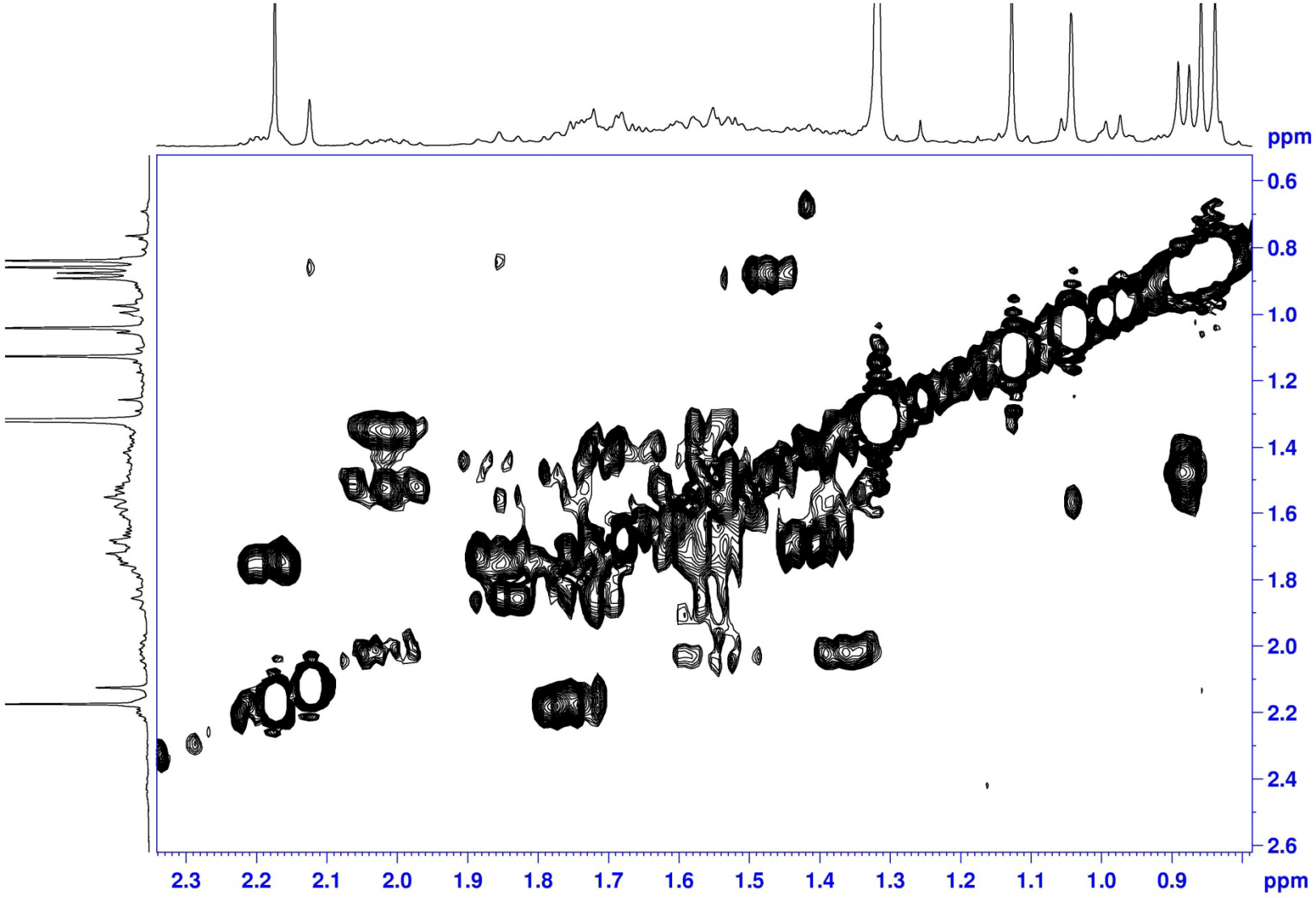

```
NAME          EA3-44-3-1
EXPNO                  4
PROCNO                 1
Date_           20171109
Time               16.41
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG      cosygpppqf
TD                  2048
SOLVENT            CDCl3
NS                     8
DS                     8
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          400.1300062 MHz
WDW               QSINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                 1024
MC2                  QF
SF          400.1300068 MHz
WDW               QSINE
SSB                    0
LB                  0.00 Hz
```

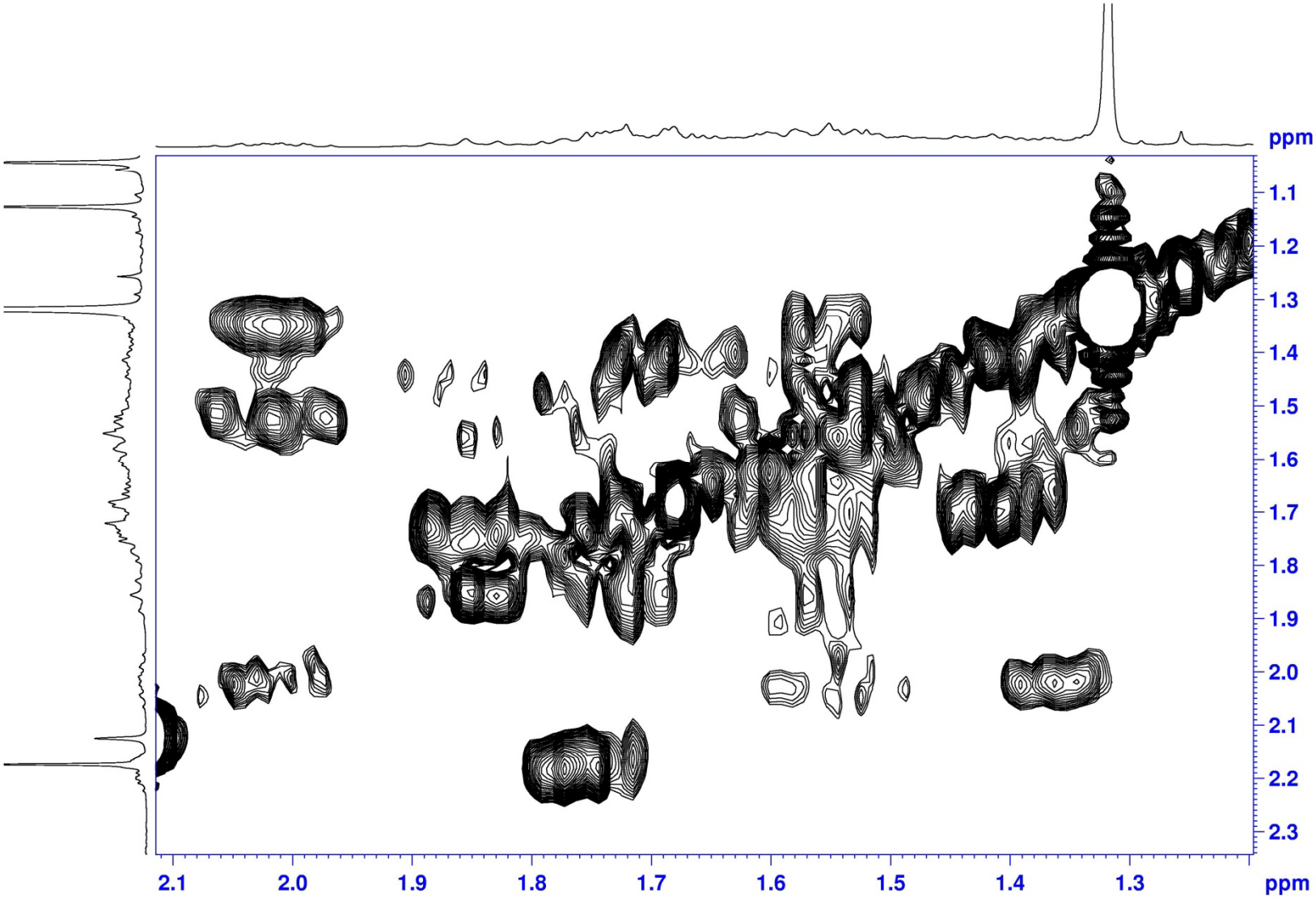
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **8** in $CDCl_3$



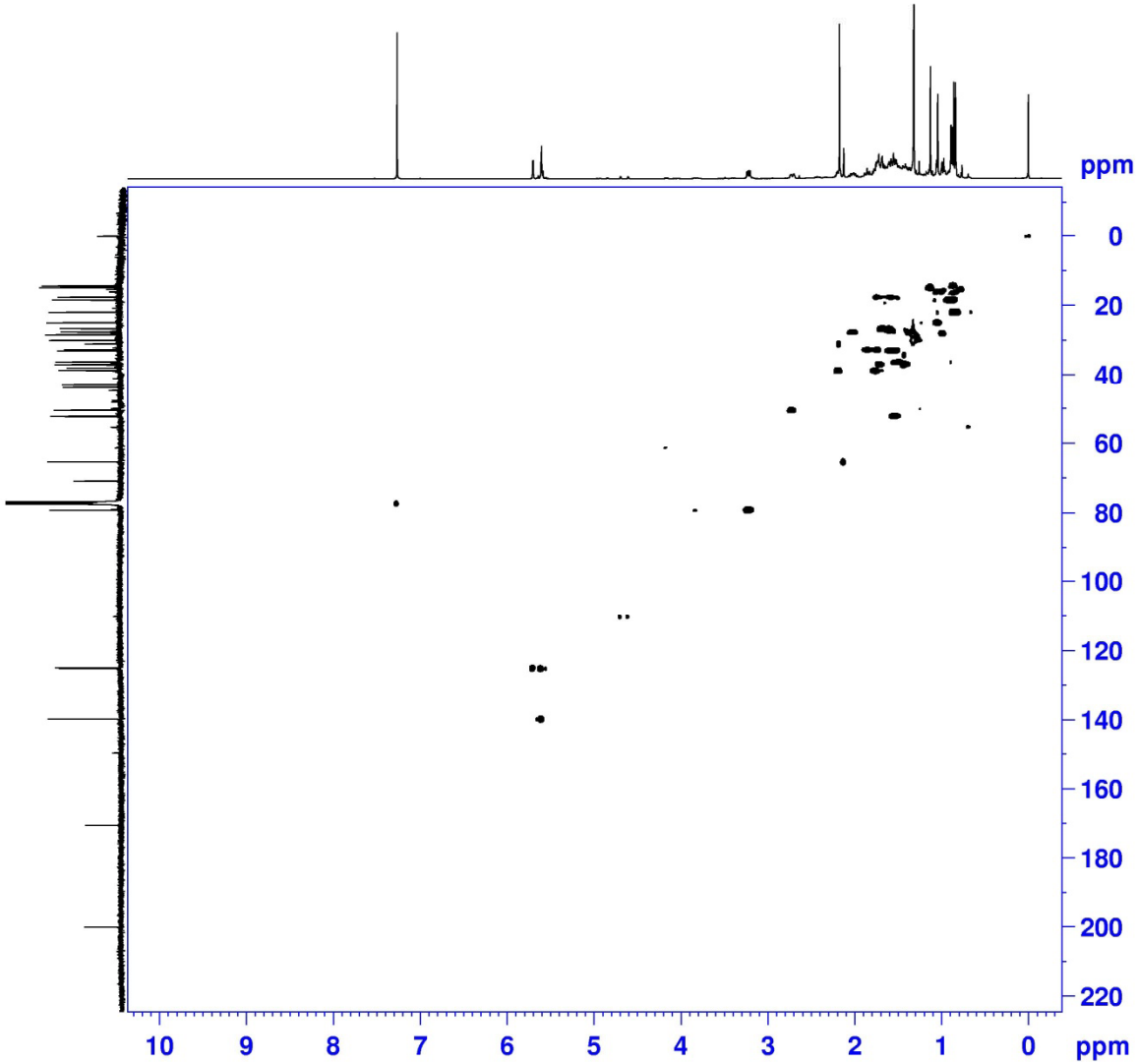
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **8** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **8** in $CDCl_3$



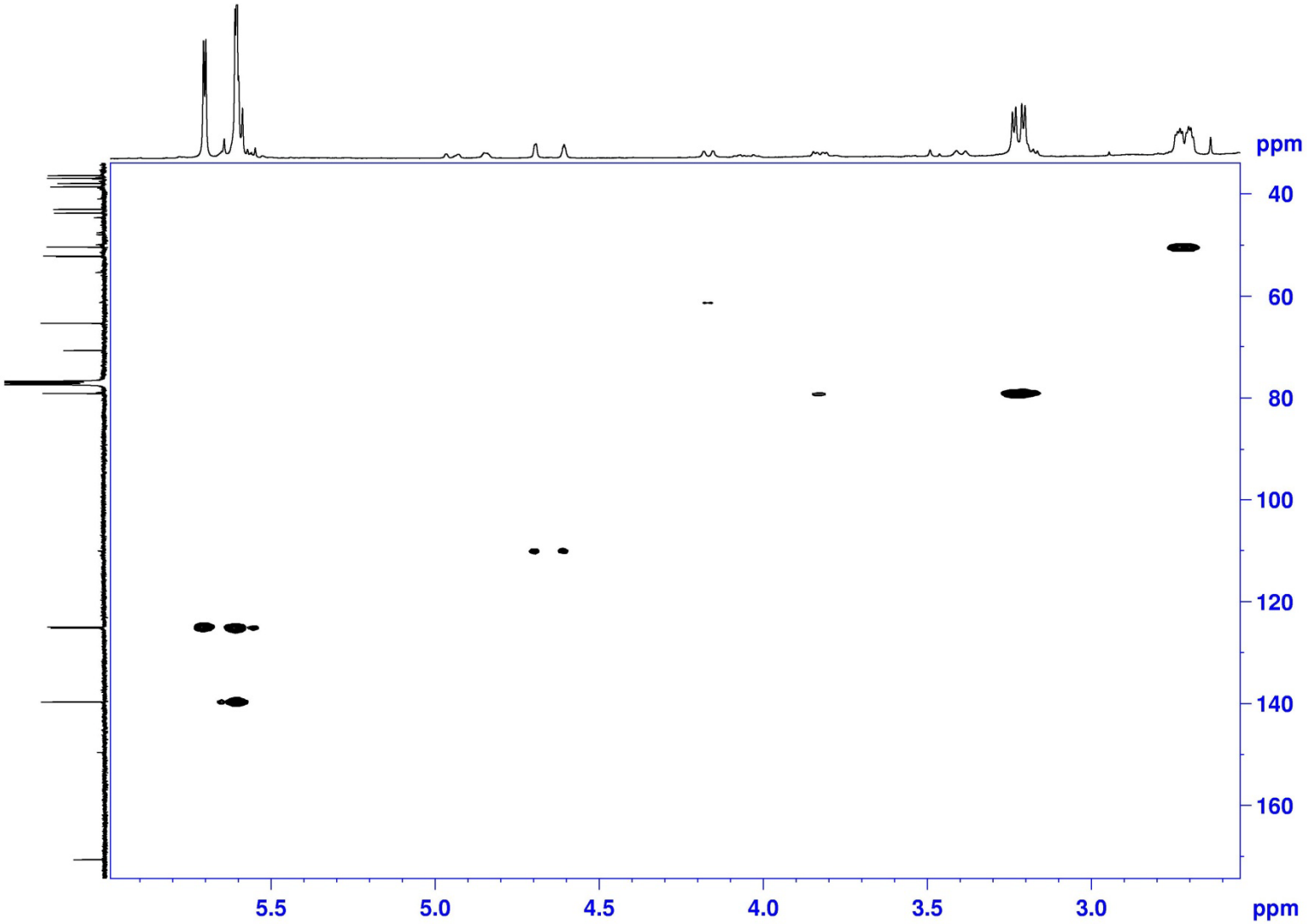
HSQC (400 MHz) spectrum of compound **8** in $CDCl_3$



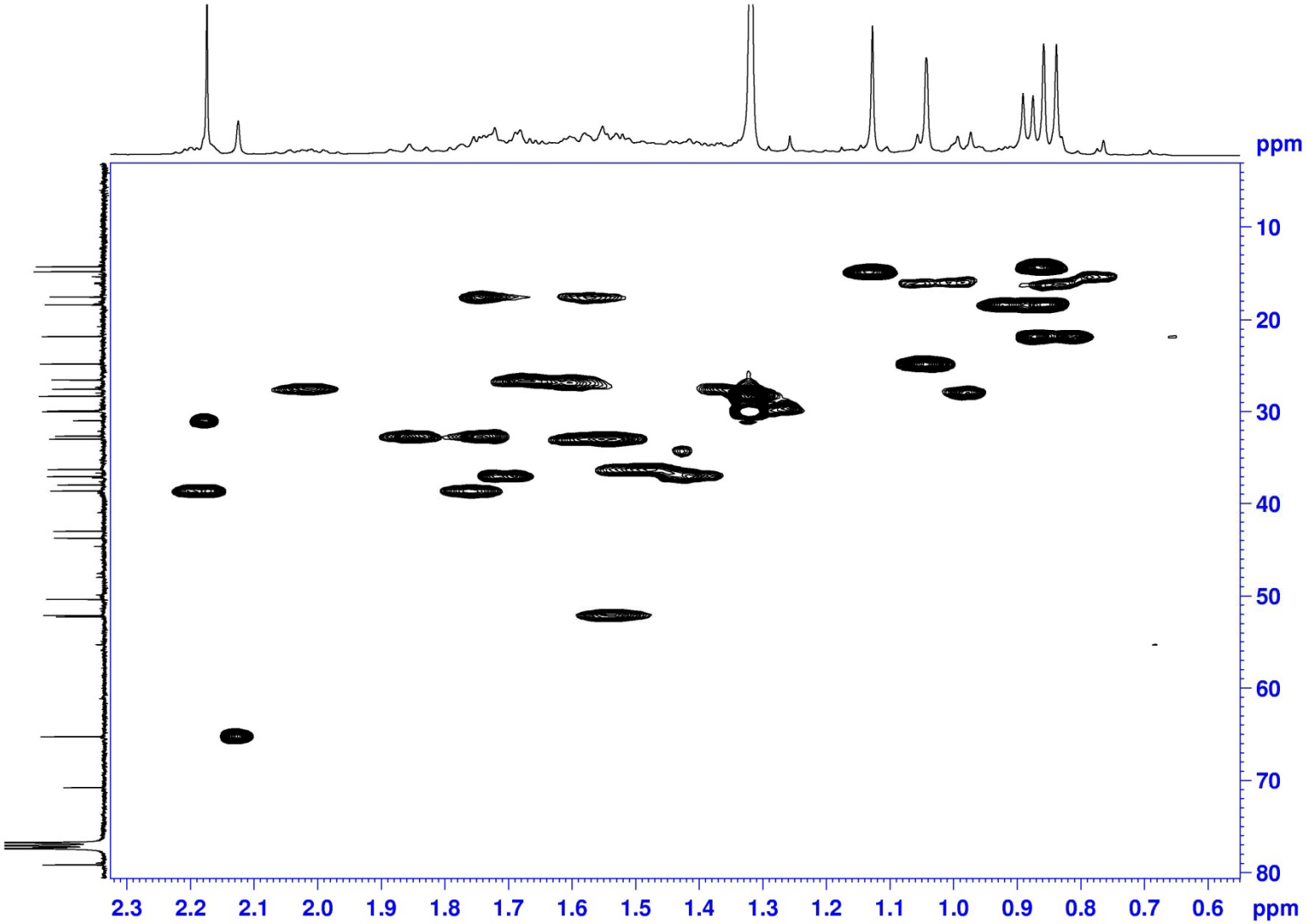

```
NAME          EA3-44-3-1
EXPNO                  5
PROCNO                 1
Date_           20171109
Time               17.20
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG     hsqcetgpsi2
TD                  1024
SOLVENT            CDCl3
NS                    16
DS                    16
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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                11.50 usec
P2                23.00 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           400.1300033 MHz
WDW               QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.40
SI                 1024
MC2        echo-antiecho
SF           100.6127549 MHz
WDW               QSINE
```

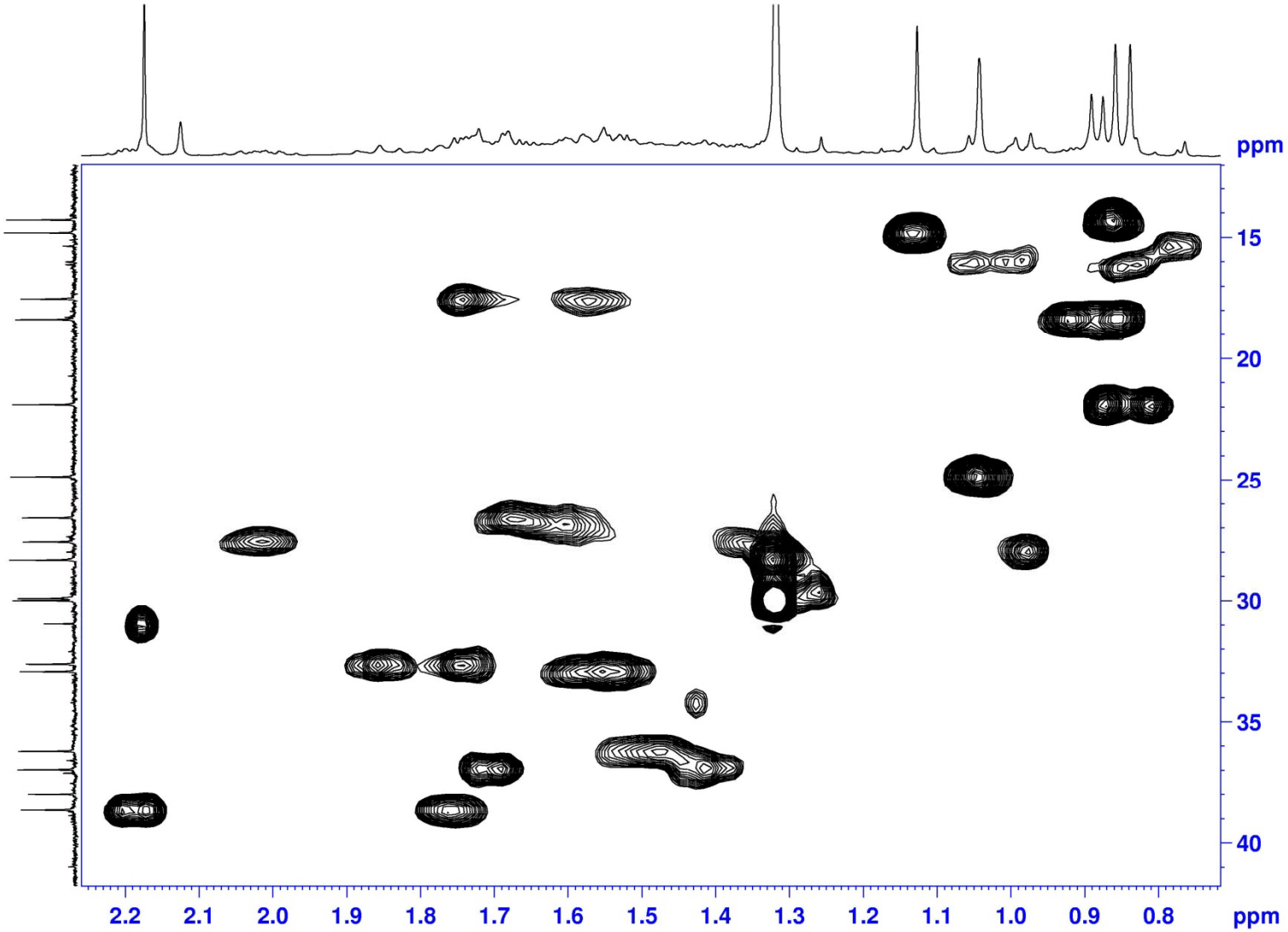
HSQC (400 MHz) spectrum of compound **8** in $CDCl_3$



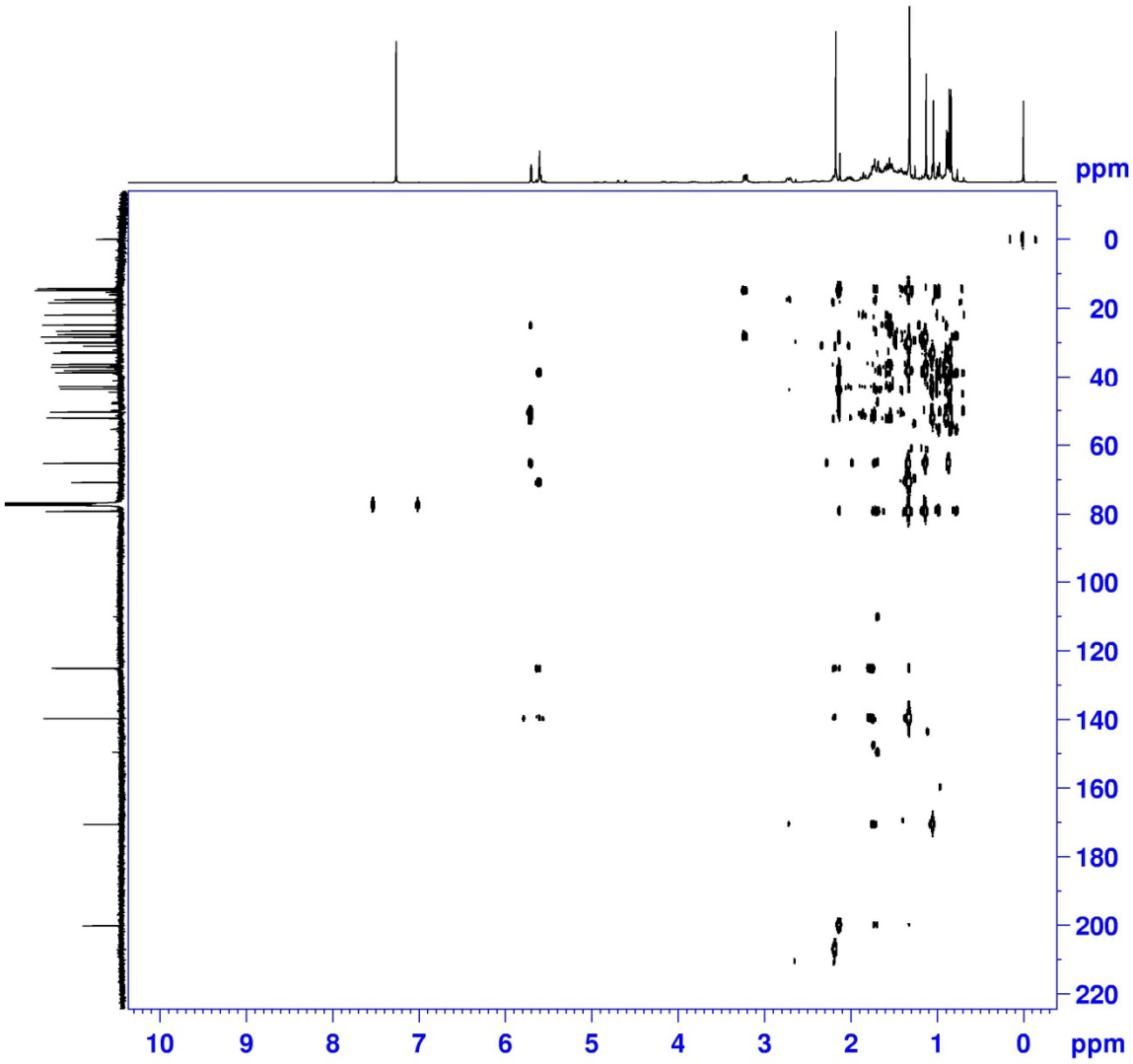
HSQC (400 MHz) spectrum of compound **8** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **8** in $CDCl_3$



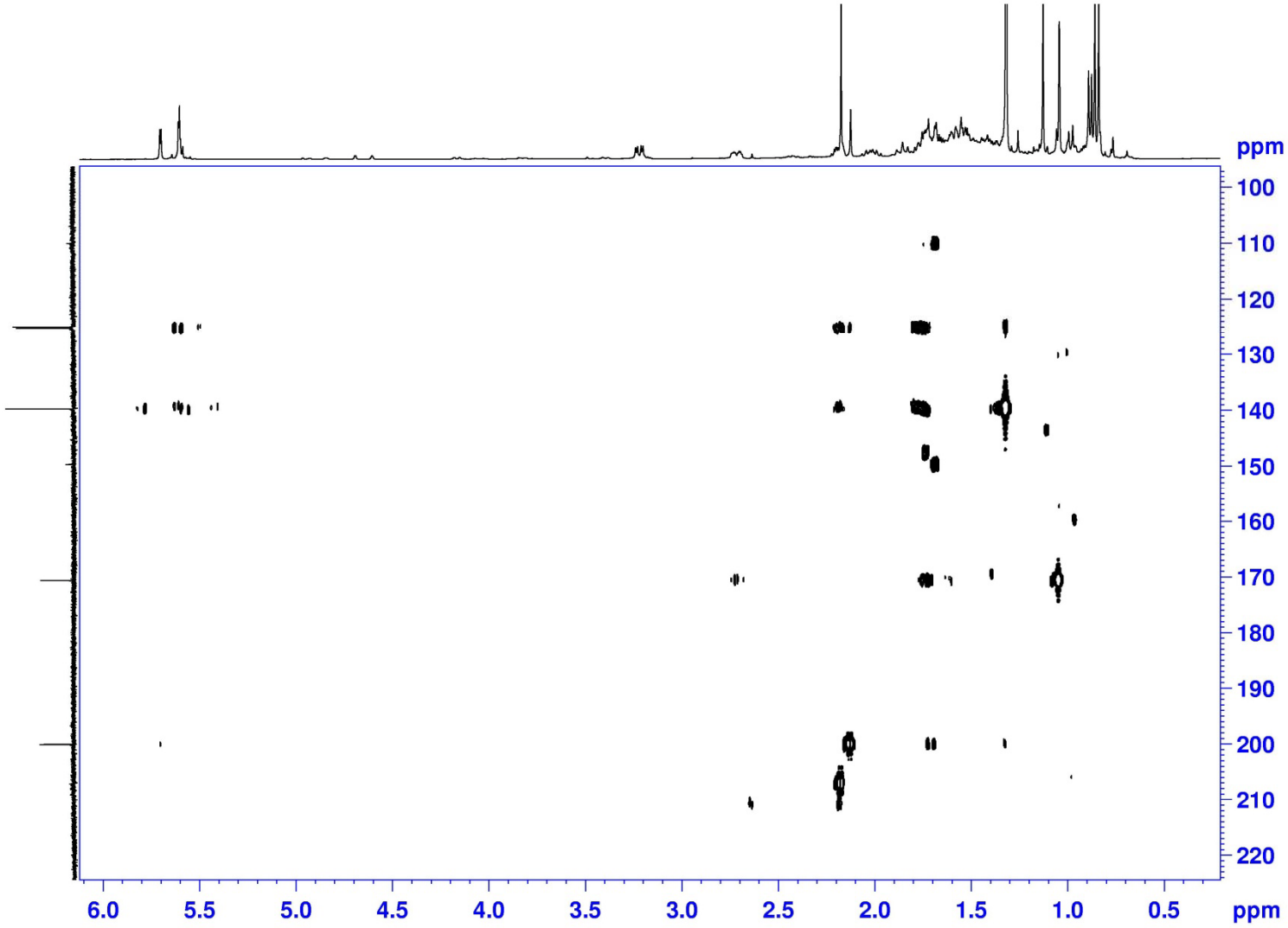
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



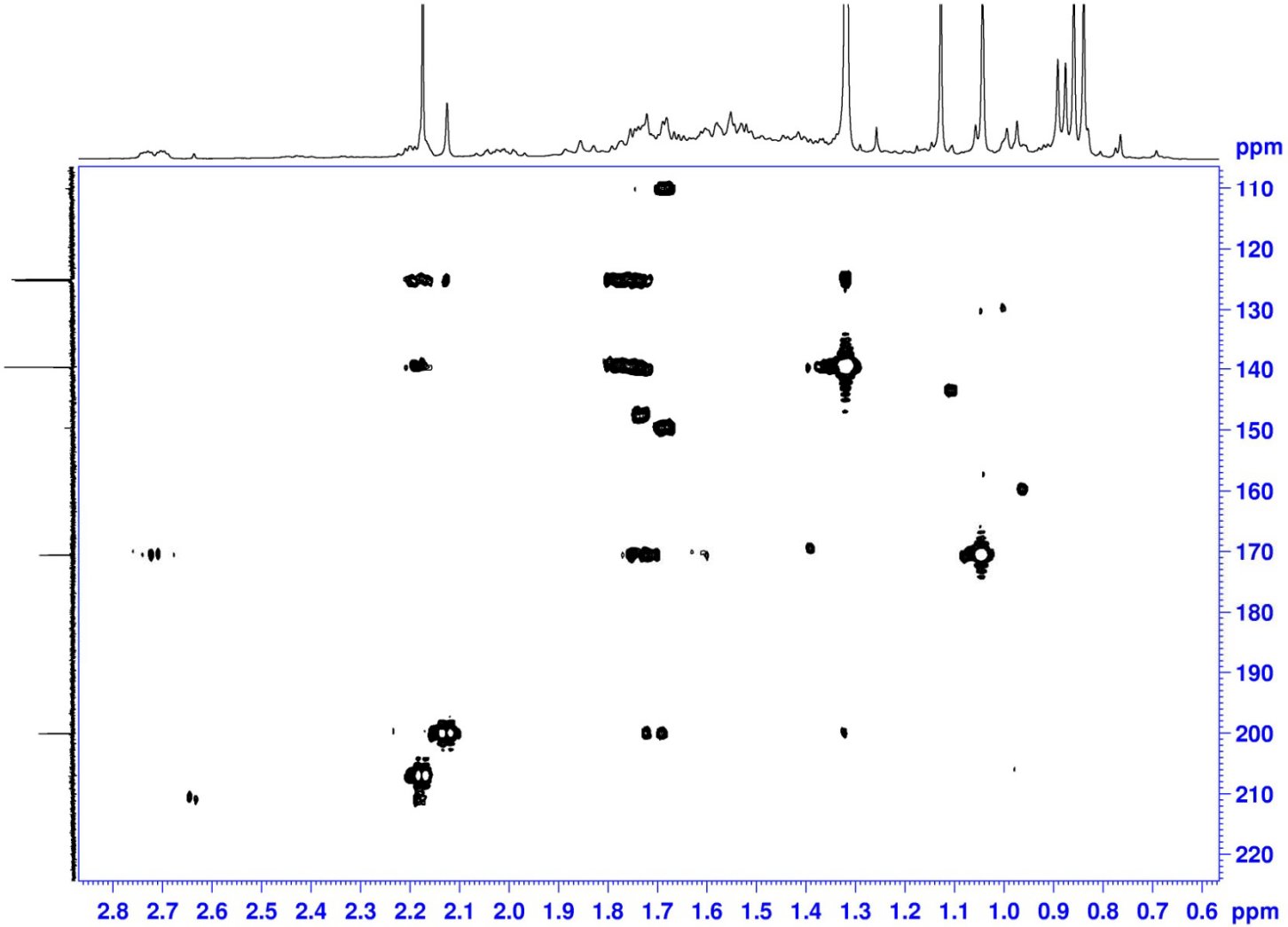

```
NAME          EA3-44-3-1
EXPNO                  6
PROCNO                 1
Date_           20171109
Time               19.11
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG    hmbcgplpndqf
TD                  4096
SOLVENT            CDCl3
NS                    32
DS                    16
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 ========
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
SI                  2048
SF           400.1300051 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           100.6127717 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
```

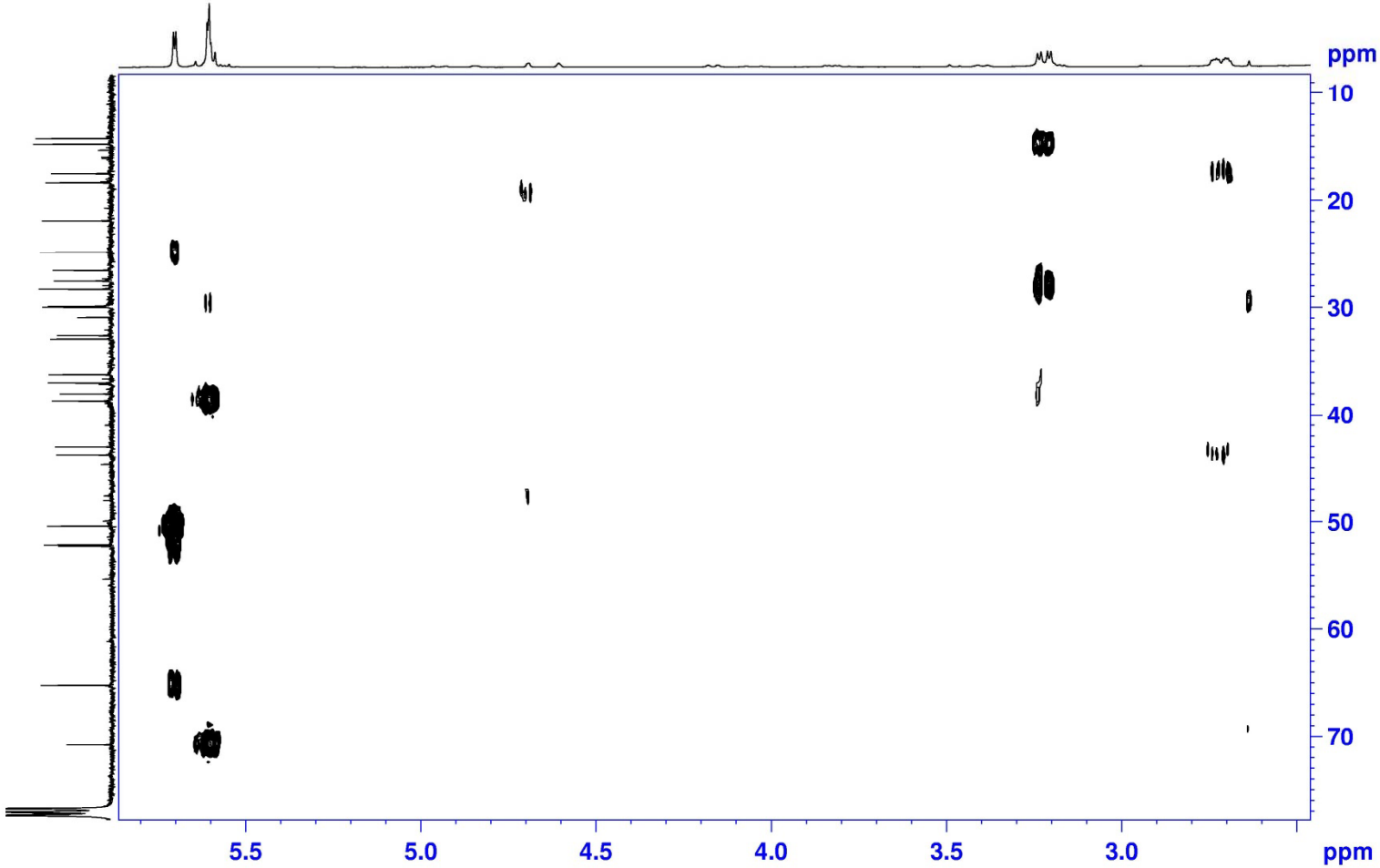
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



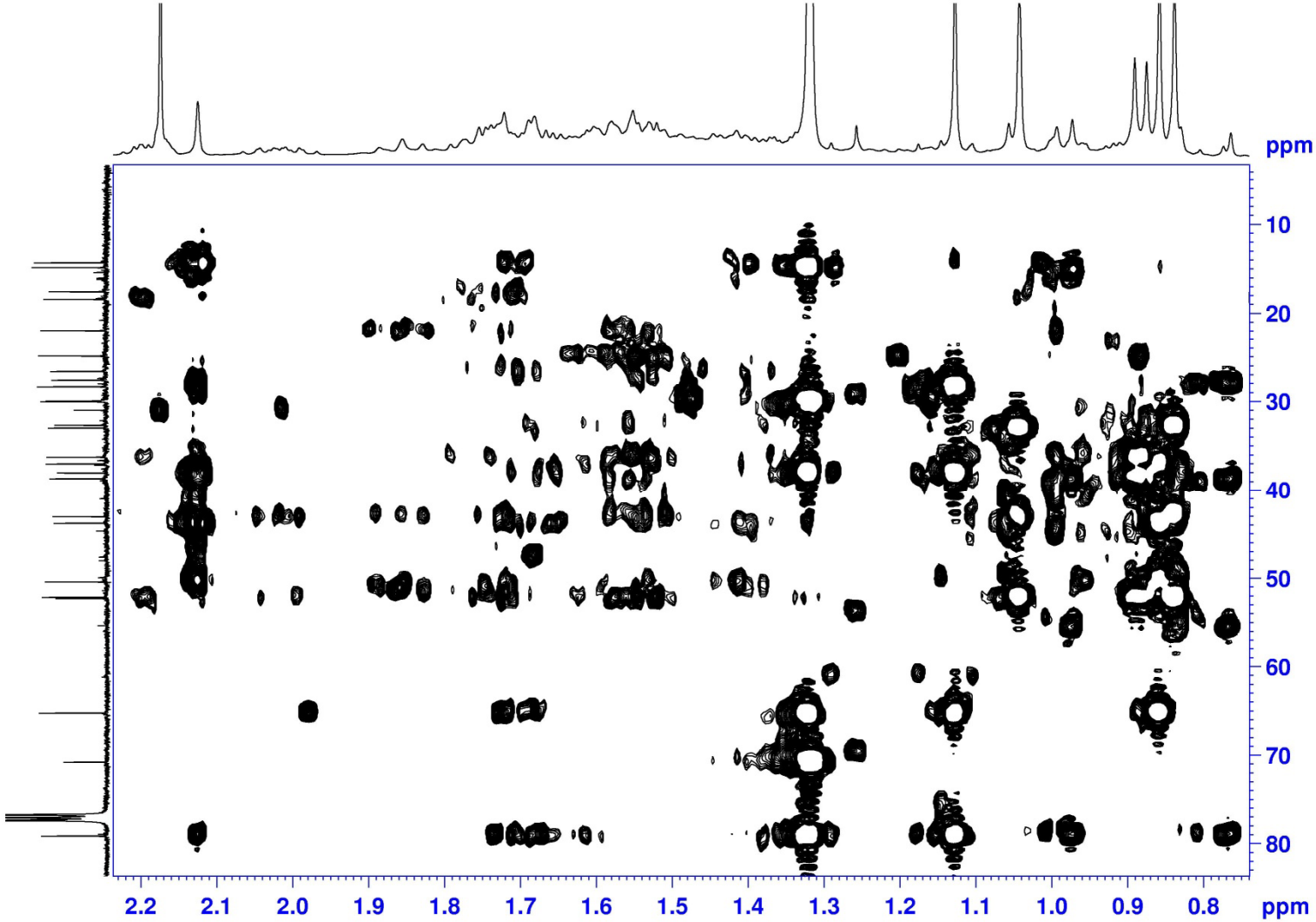
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



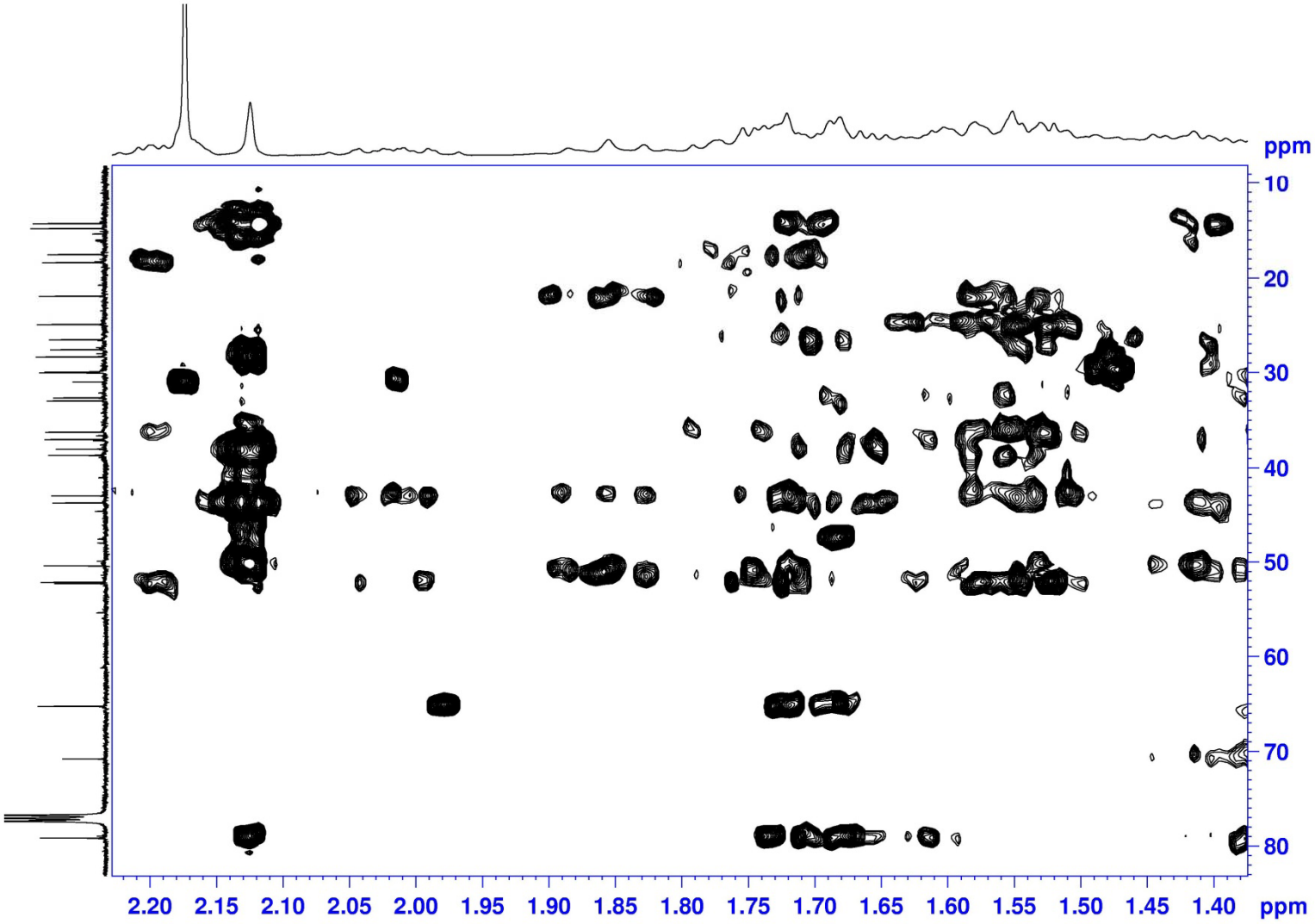
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



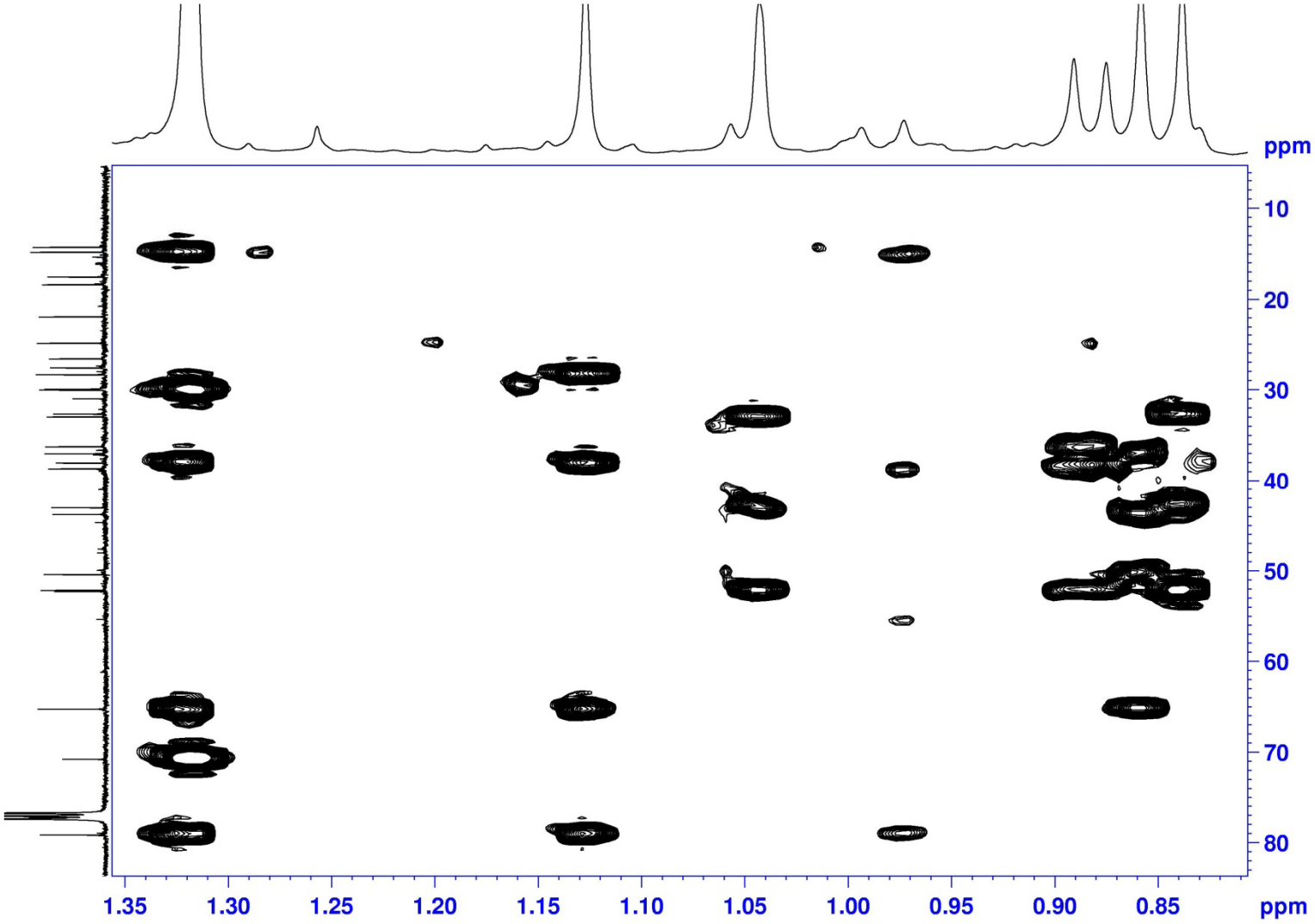
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



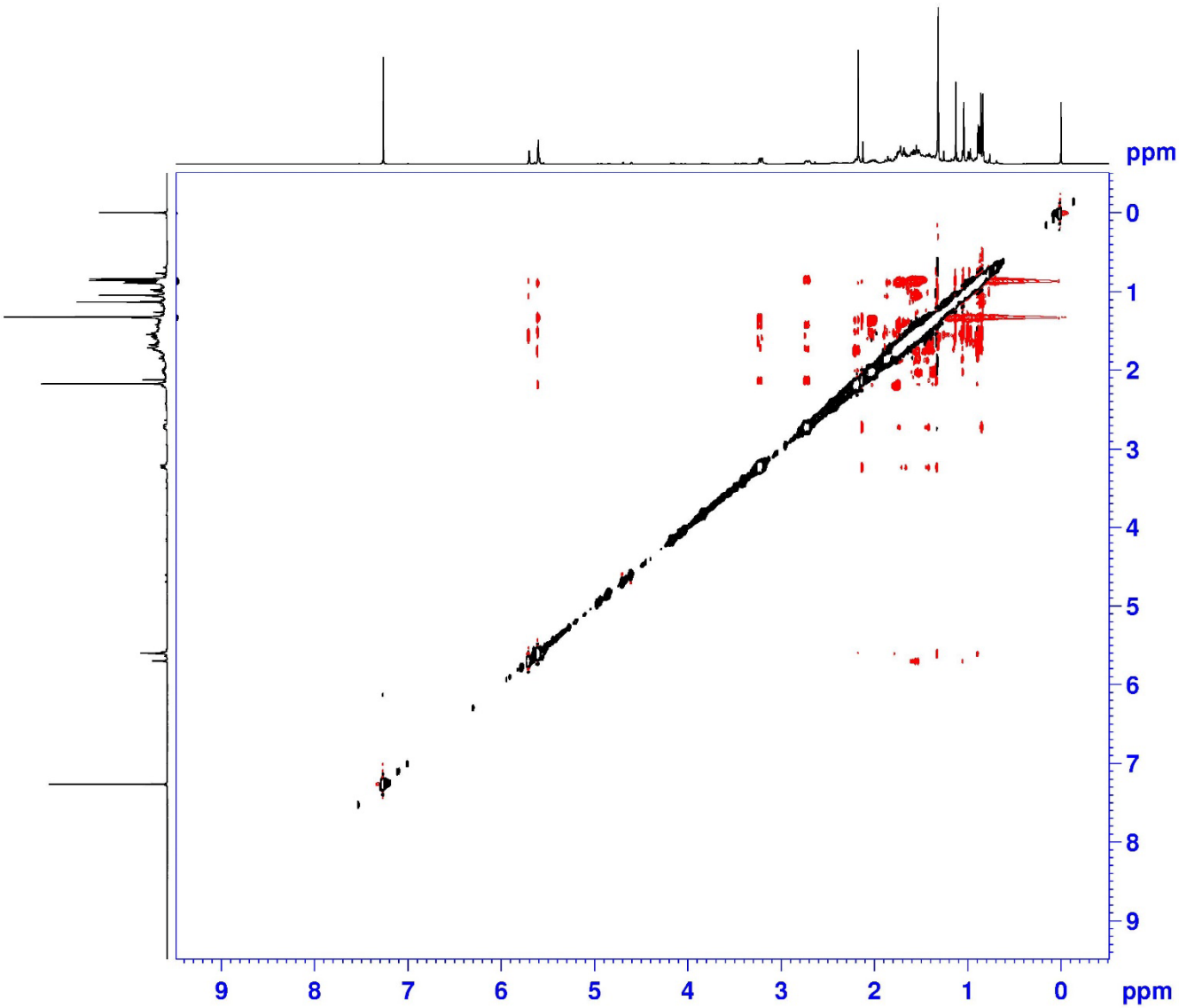
HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **8** in $CDCl_3$



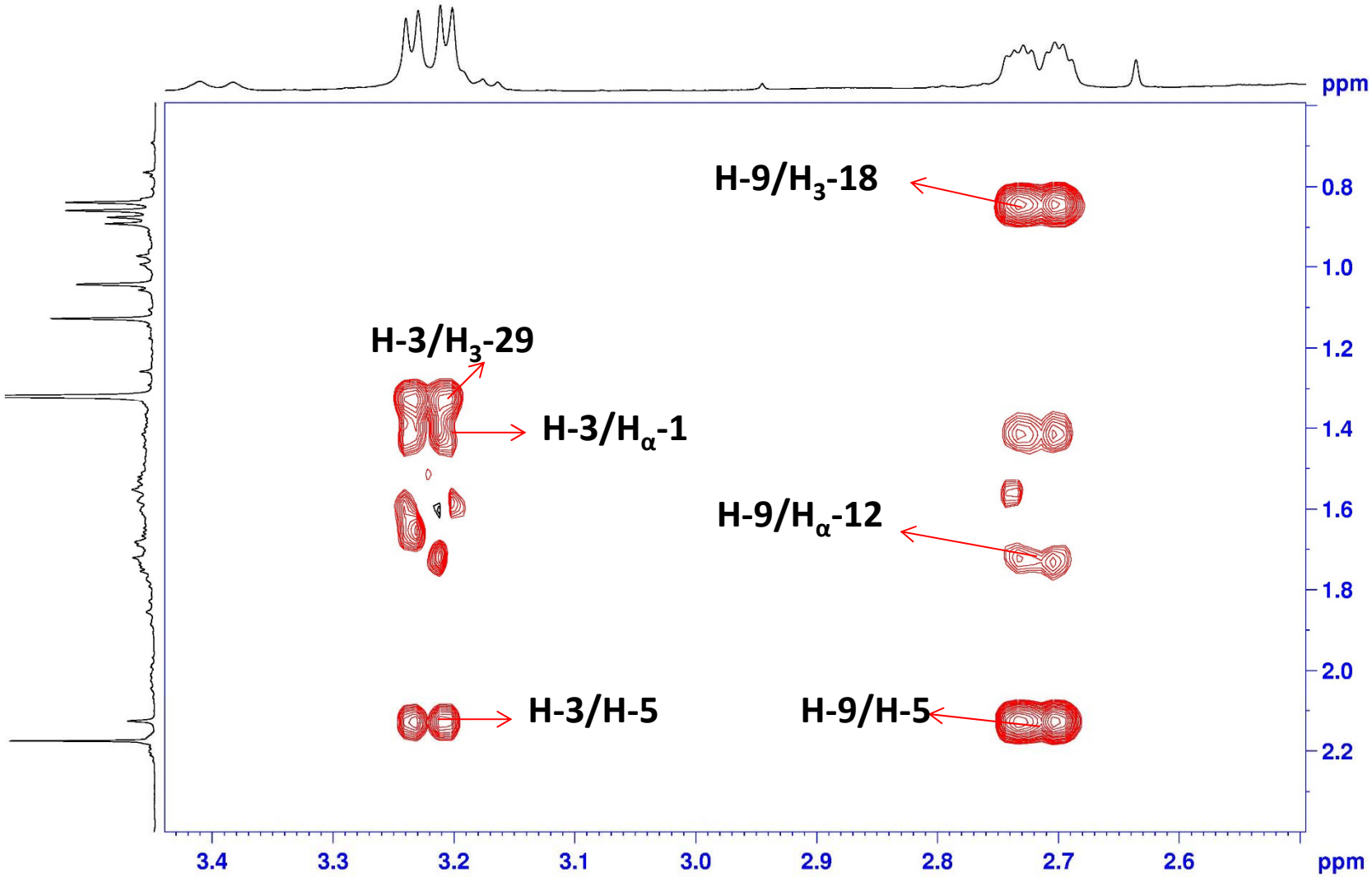
NOESY (400 MHz) spectrum of compound **8** in $CDCl_3$



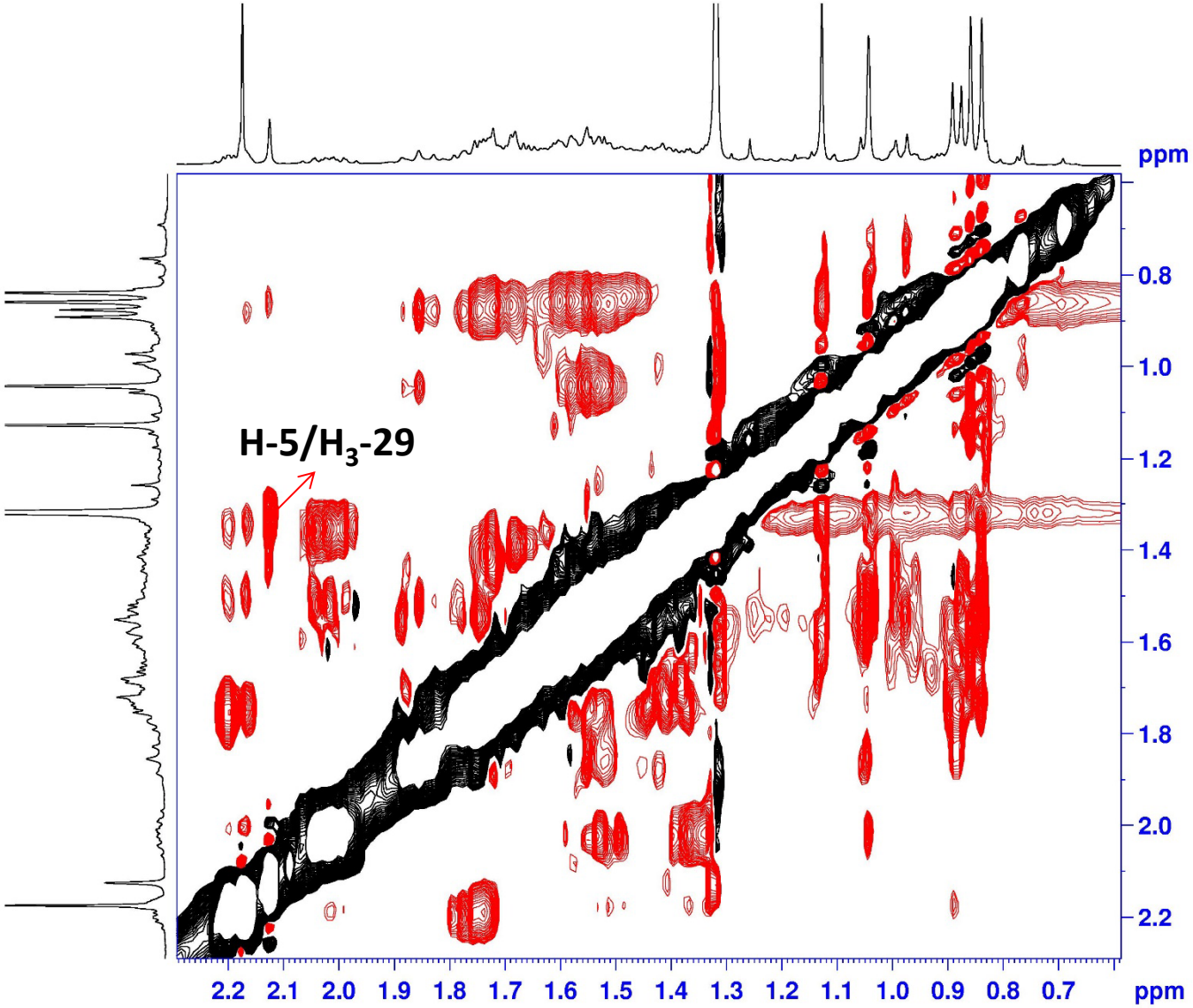

```
NAME          EA3-44-3-1
EXPNO               107
PROCNO                1
Date_          20171127
Time               0.52
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG     noesygpphpp
TD                 2048
SOLVENT           CDCl3
NS                   32
DS                   32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ            0.2560500 se
RG                208.5
DW               125.000 us
DE                 10.00 us
TE                 297.0 K
D0            0.00011036 se
D1            1.99385595 se
D8            0.30000001 se
D11           0.03000000 se
D12           0.00002000 se
D16           0.00020000 se
IN0           0.00025000 se

======== CHANNEL f1 ======
SFO1        400.1318006 MH
NUC1                 1H
P1                11.50 us
P2                23.00 us
P17            2500.00 us
ND0                   1
TD                  256
SFO1           400.1318 MH
FIDRES        15.625000 Hz
SW                9.997 pp
FnMODE      States-TPPI
SI                 1024
SF          400.1300059 MH
WDW               QSINE
SSB                   2
LB                 0.00 Hz
GB                    0
PC                 1.00
SI                 1024
MC2         States-TPPI
SF          400.1300055 MH
WDW               QSINE
SSB                   2
LB                 0.00 Hz
```

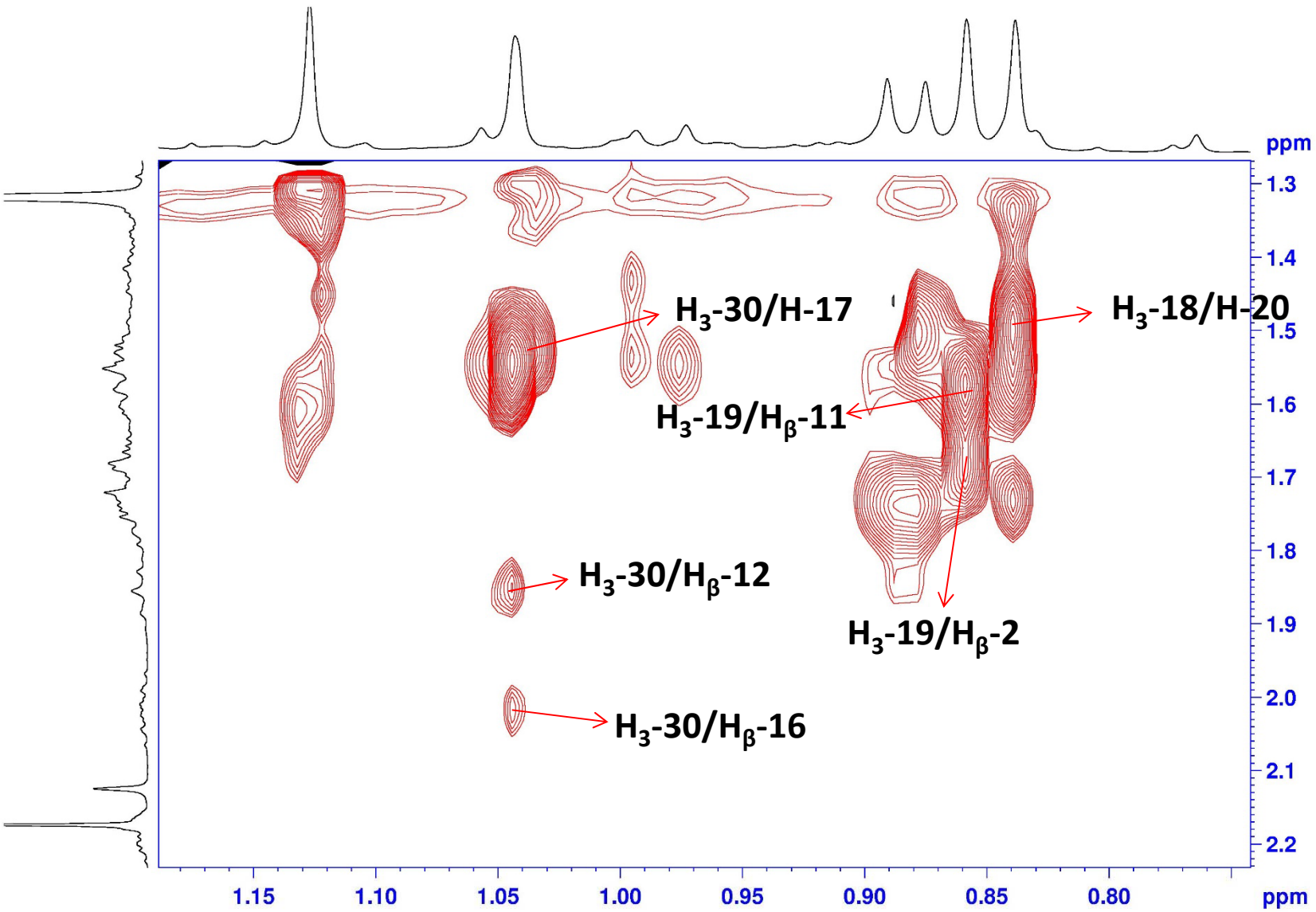
NOESY (400 MHz) spectrum of compound **8** in $CDCl_3$



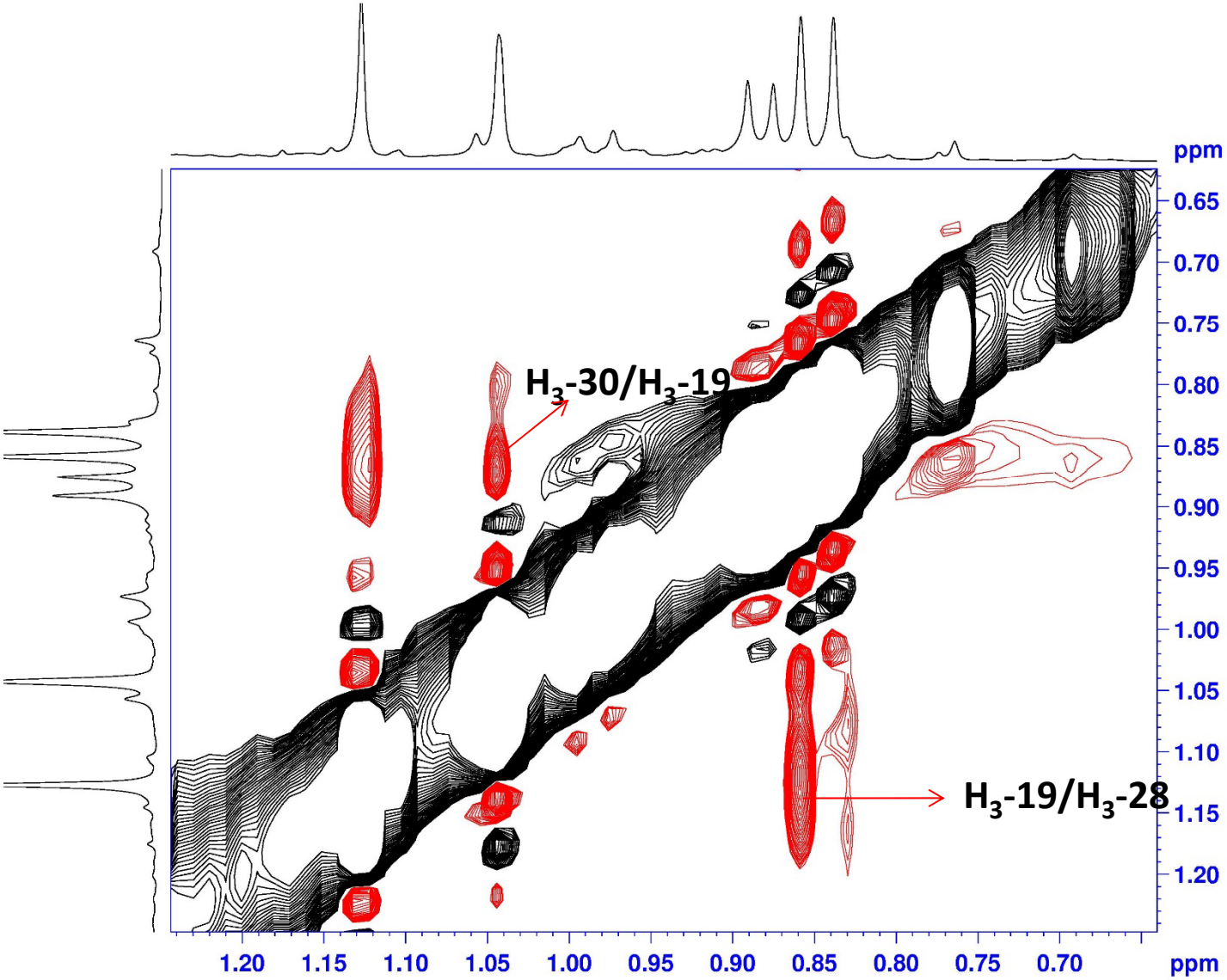
NOESY (400 MHz) spectrum of compound **8** in $CDCl_3$



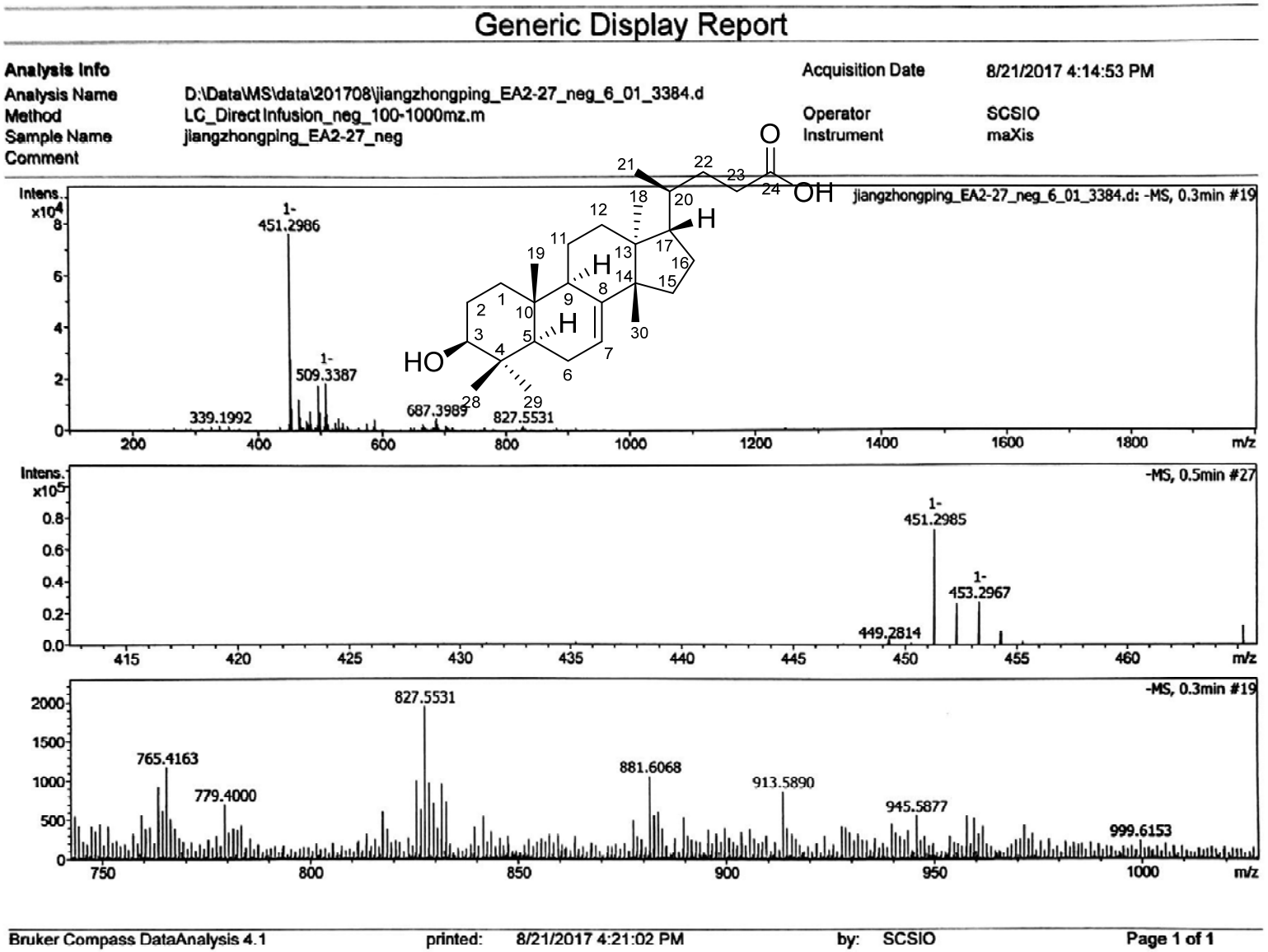
NOESY (400 MHz) spectrum of compound **8** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **8** in $CDCl_3$



HR-ESIMS for compound **9**



HR-ESIMS for compound **9**

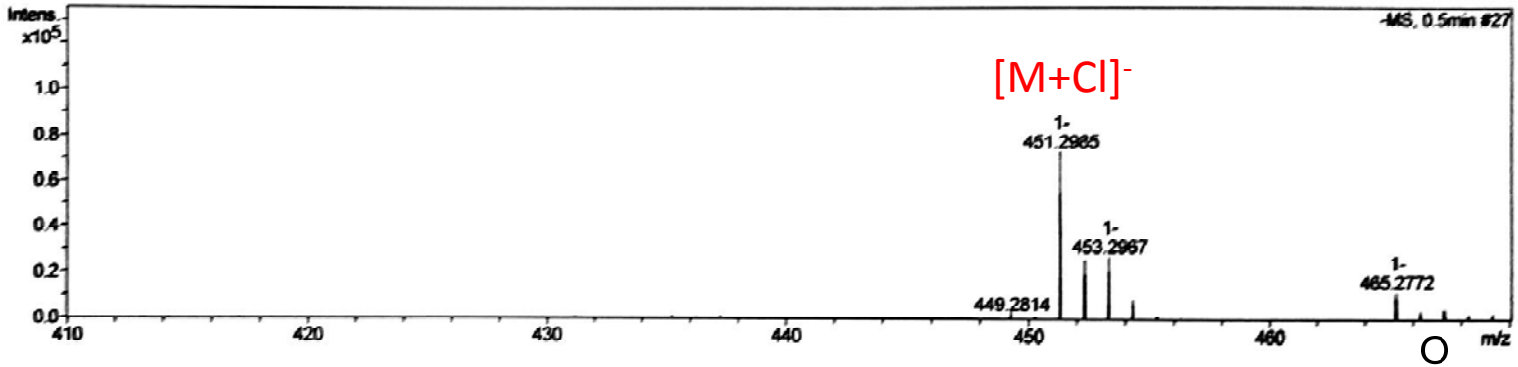
Mass Spectrum SmartFormula Report

Analysis Info

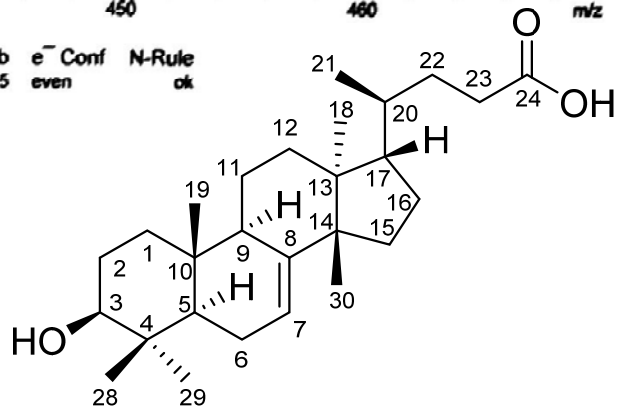
Analysis Name	D:\Data\MS\data\201708\jiangzhongping_EA2-27_neg_6_01_3384.d	Acquisition Date	8/21/2017 4:14:53 PM
Method	LC_Direct Infusion_neg_100-1000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_EA2-27_neg	Instrument	maXis 255552.00029
Comment			

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



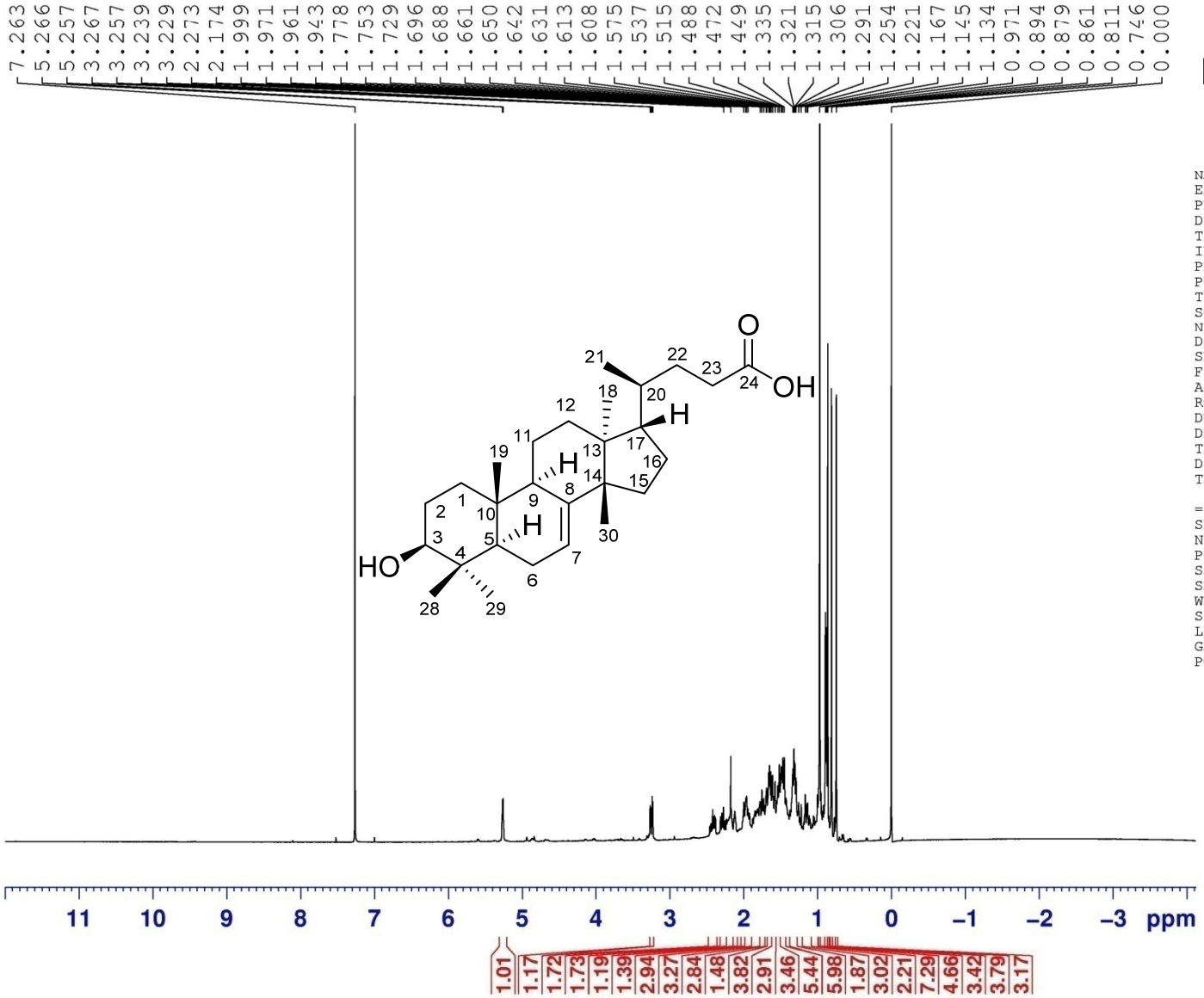
Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
451.298467	1	C27H44ClO3	100.00	451.298447	-0.0	-0.0	25.6	5.5	even	ok



jiangzhongping_EA2-27_neg_6_01_3384.d

Bruker Compass DataAnalysis 4.1 printed: 8/21/2017 4:20:24 PM by: SCSIO Page 1 of 1

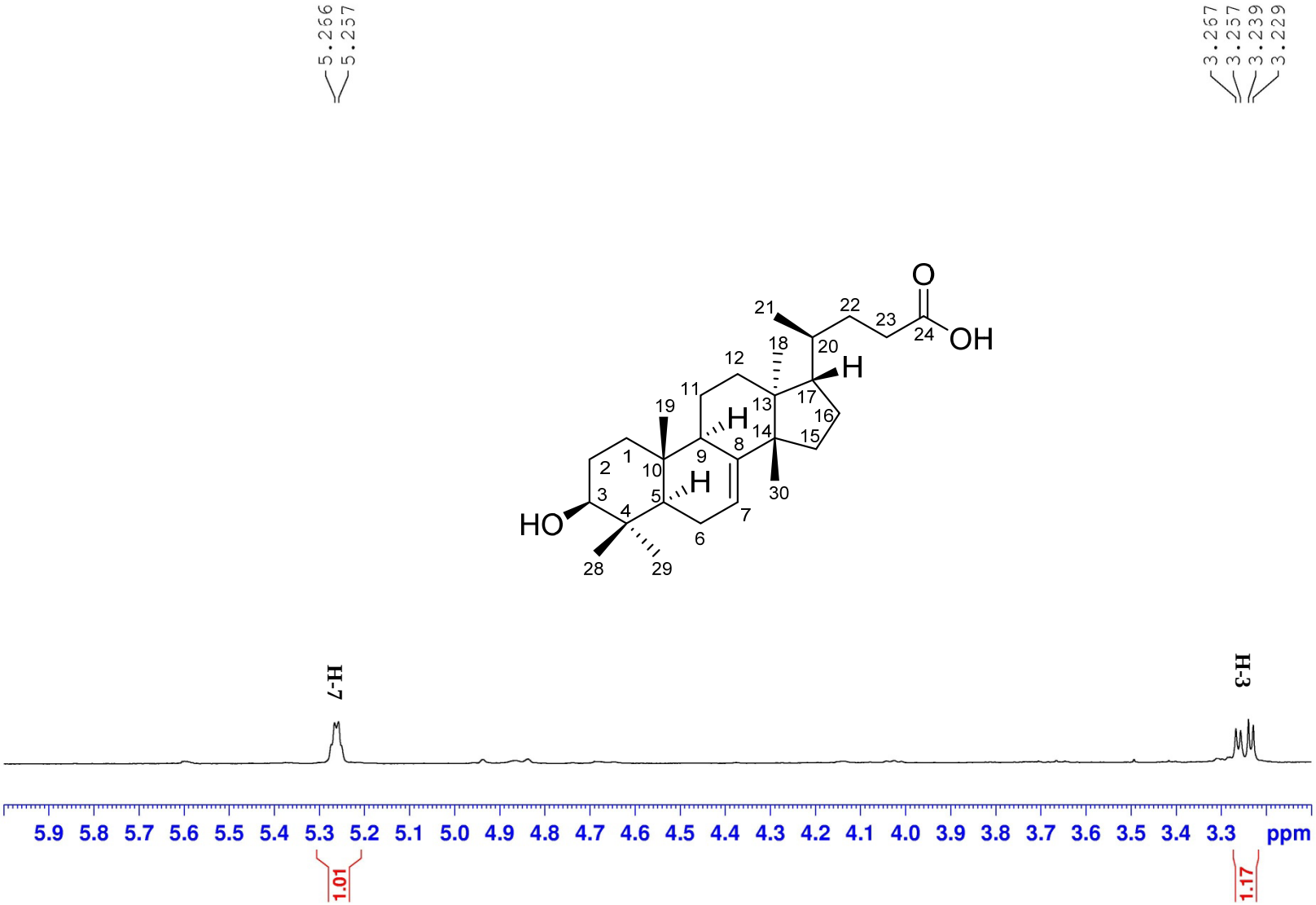
^{1}H NMR (400 MHz) spectrum of compound **9** in $CDCl_3$



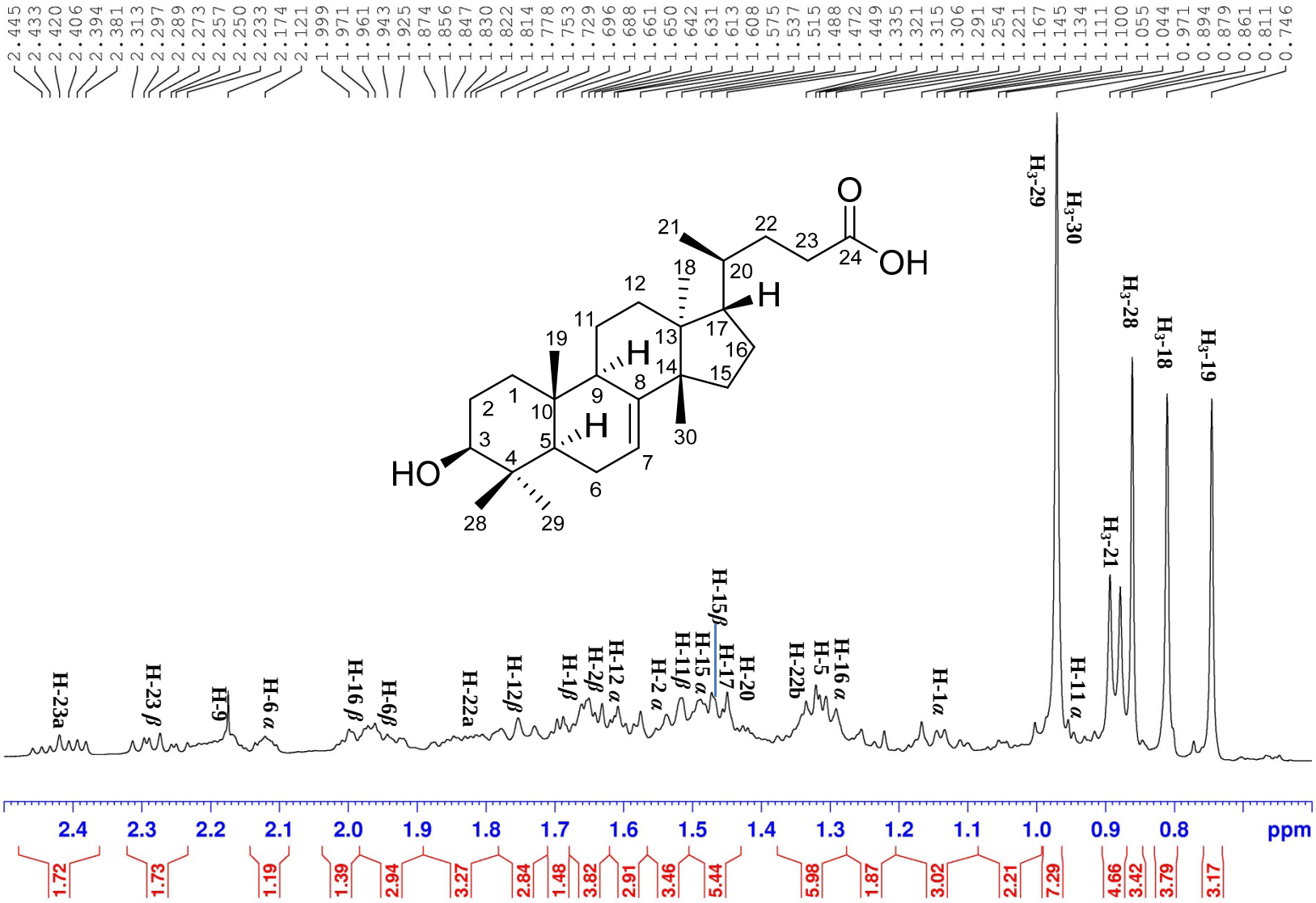

```
NAME        EA2-27(EA2-33-3)
EXPNO                      1
PROCNO                     1
Date_               20170613
Time                    4.00
INSTRUM                spect
PROBHD     5 mm CPPBBO BB
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                    208.5
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                    11.50 usec
SI                    65536
SF               400.1300088 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

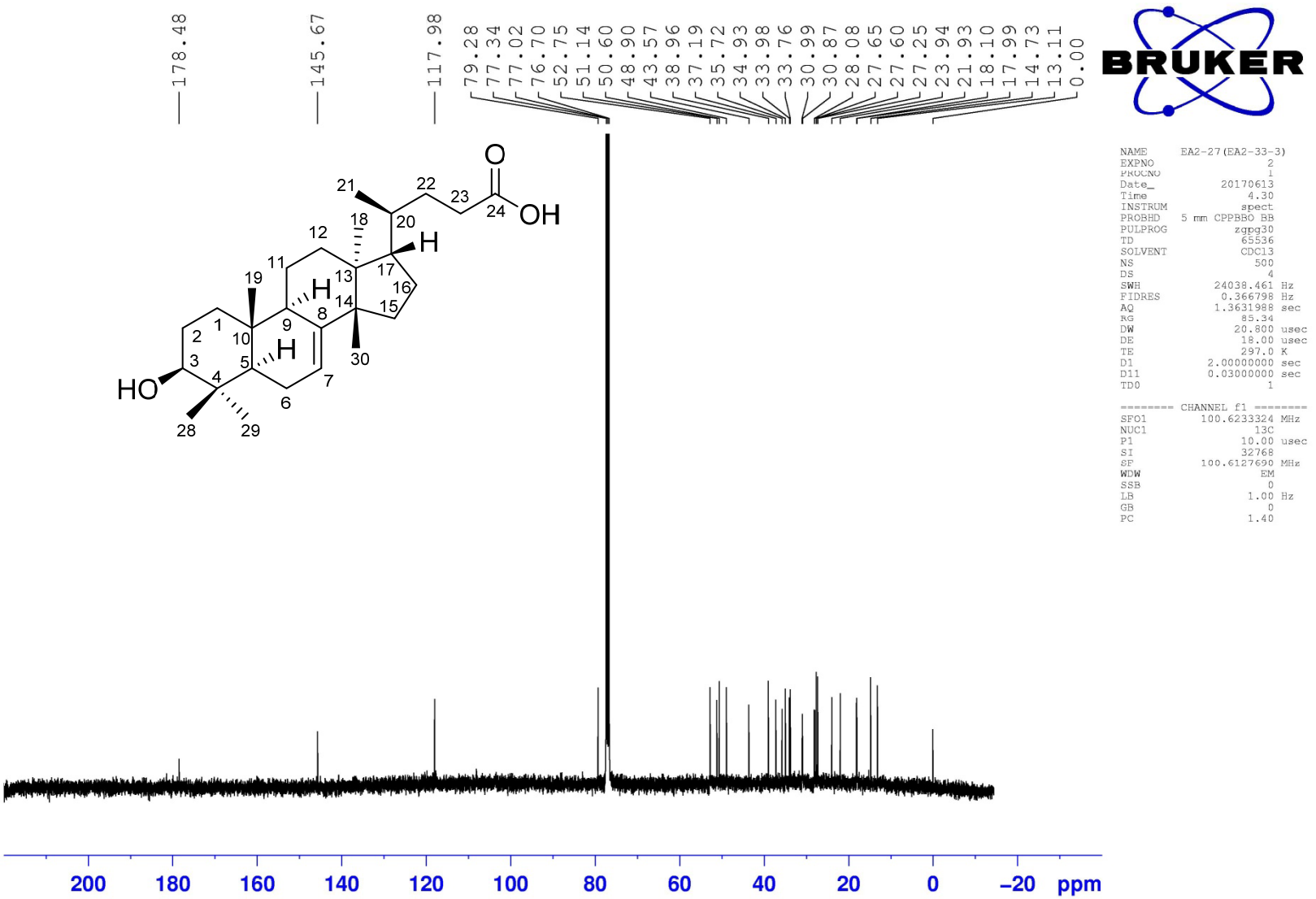
^{1}H NMR (400 MHz) spectrum of compound **9** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **9** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **9** in $CDCl_3$

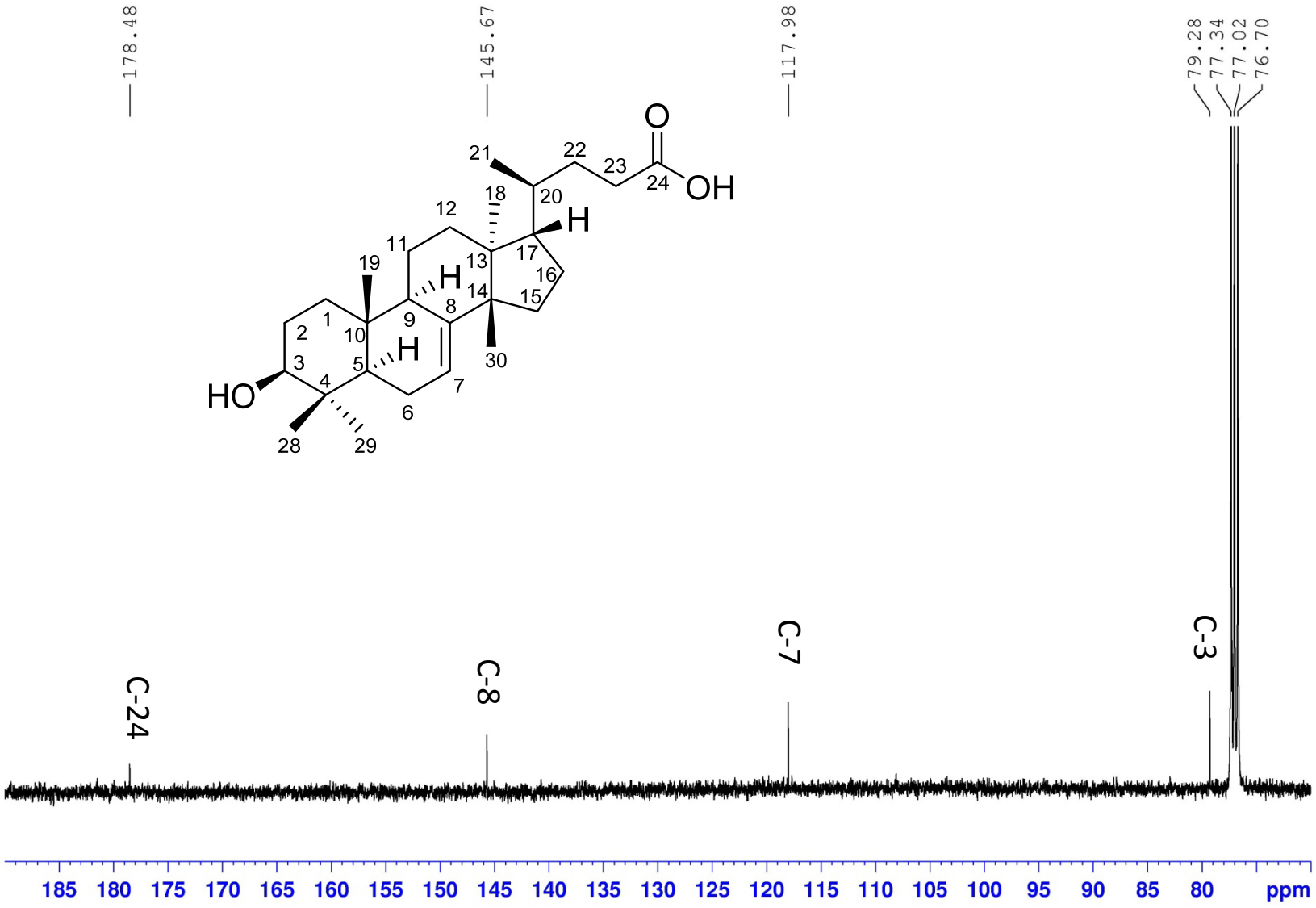


BRUKER

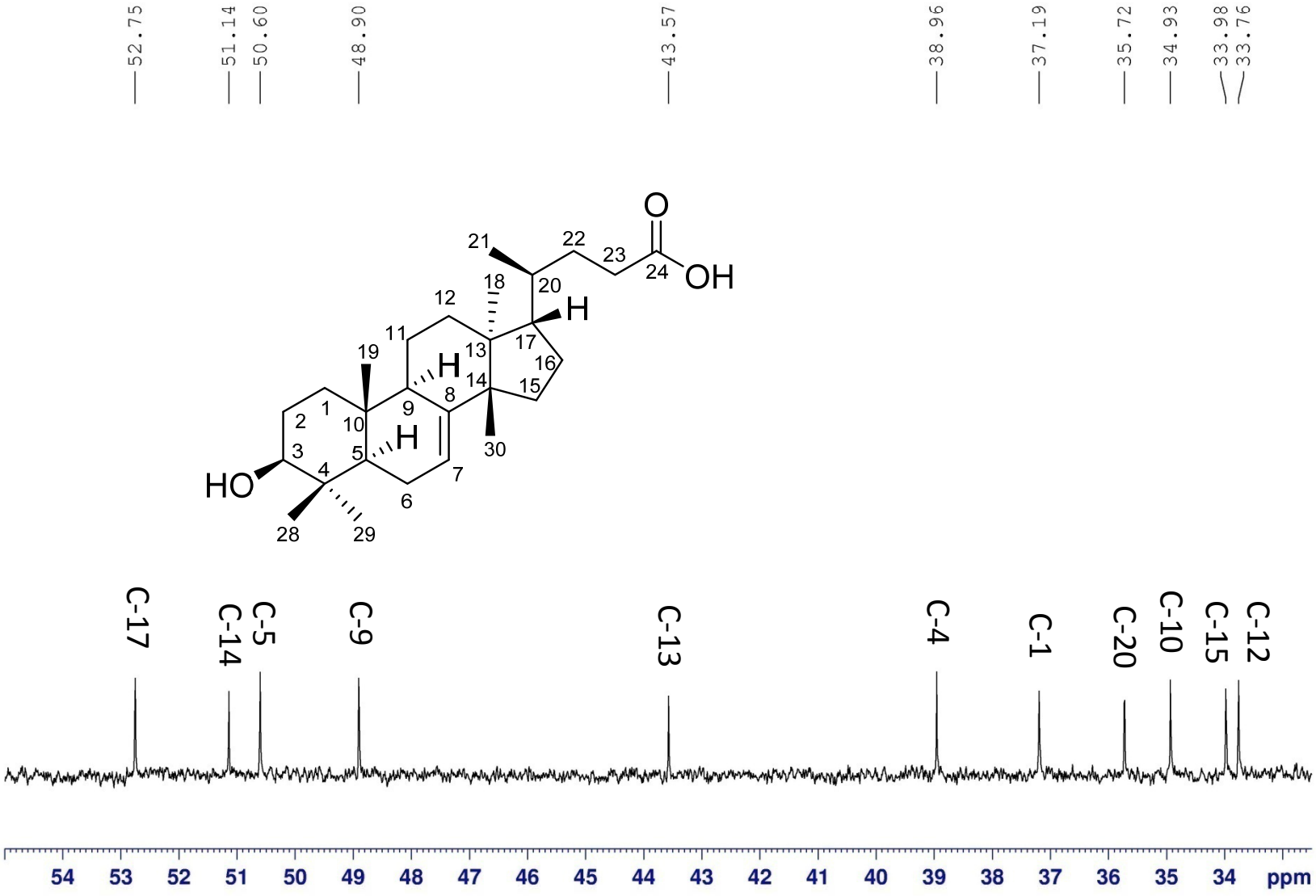
```
NAME        EA2-27(EA2-33-3)
EXPNO                      2
PROCNO                     1
Date_               20170613
Time                    4.30
INSTRUM                spect
PROBHD     5 mm CPPBBO BB
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       500
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631988 sec
RG                      85.34
DW                     20.800 usec
DE                      18.00 usec
TE                      297.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             100.6233324 MHz
NUC1                      13C
P1                      10.00 usec
SI                      32768
SF               100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

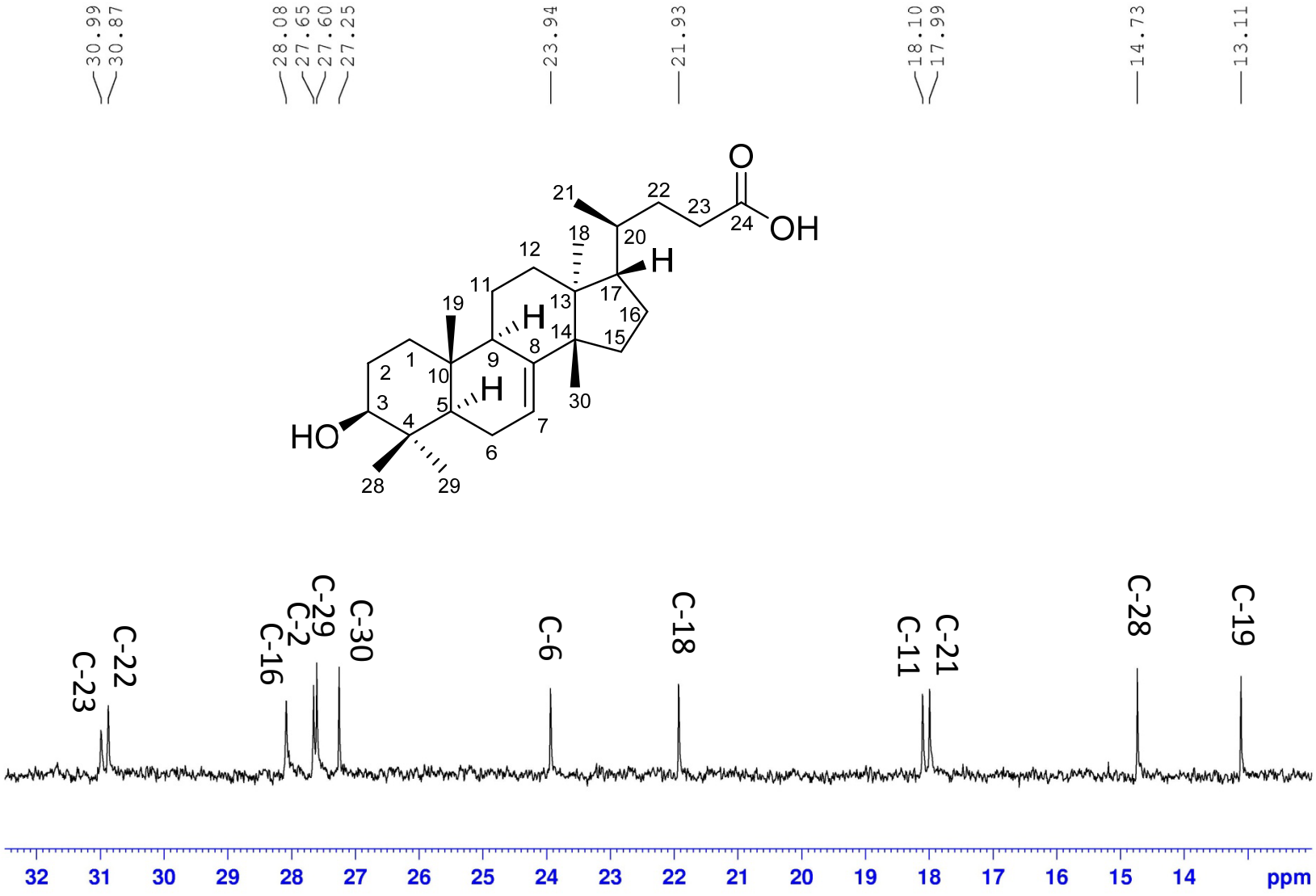
^{13}C NMR (100 MHz) spectrum of compound **9** in $CDCl_3$



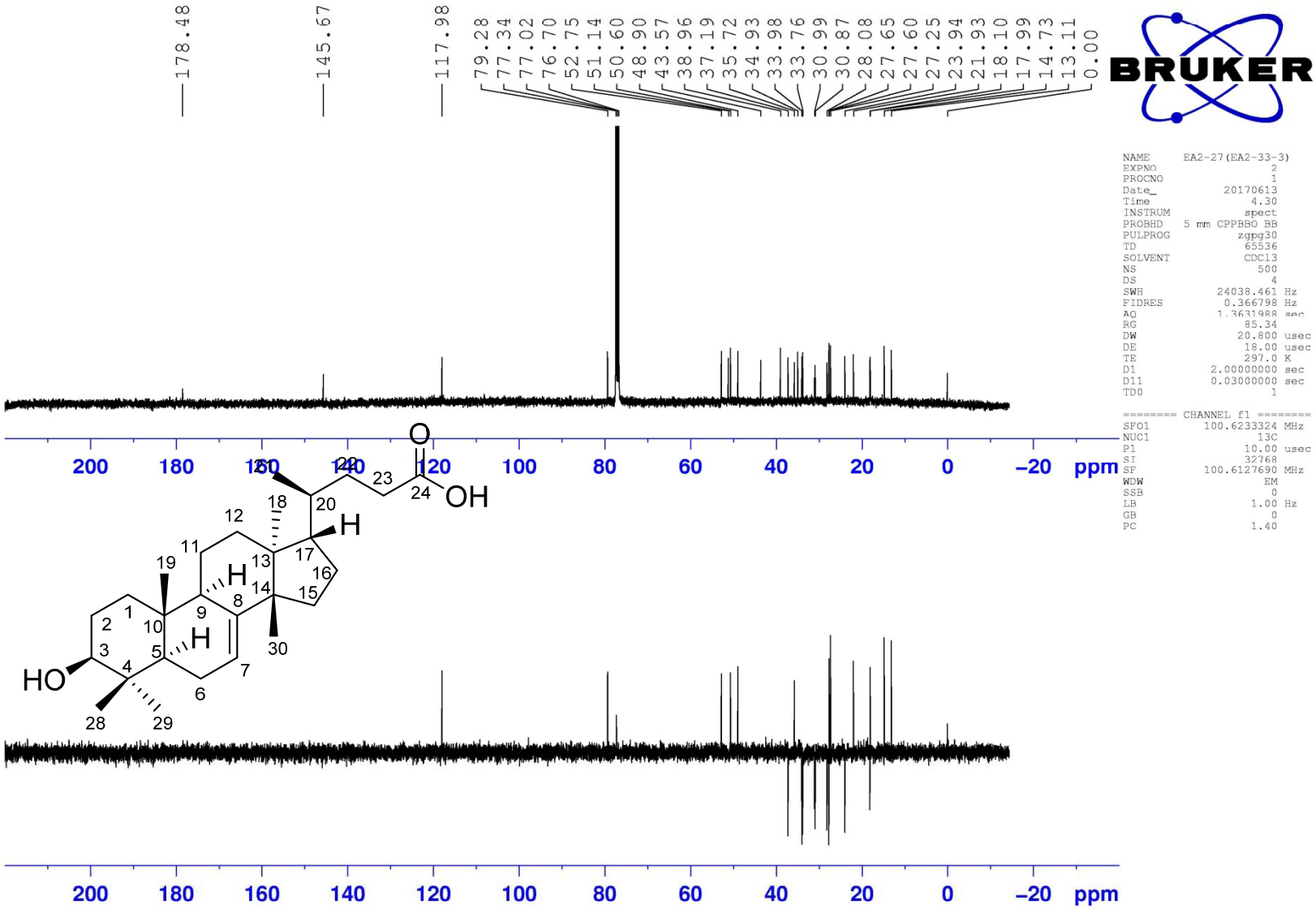
^{13}C NMR (100 MHz) spectrum of compound **9** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **9** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **9** in $CDCl_3$

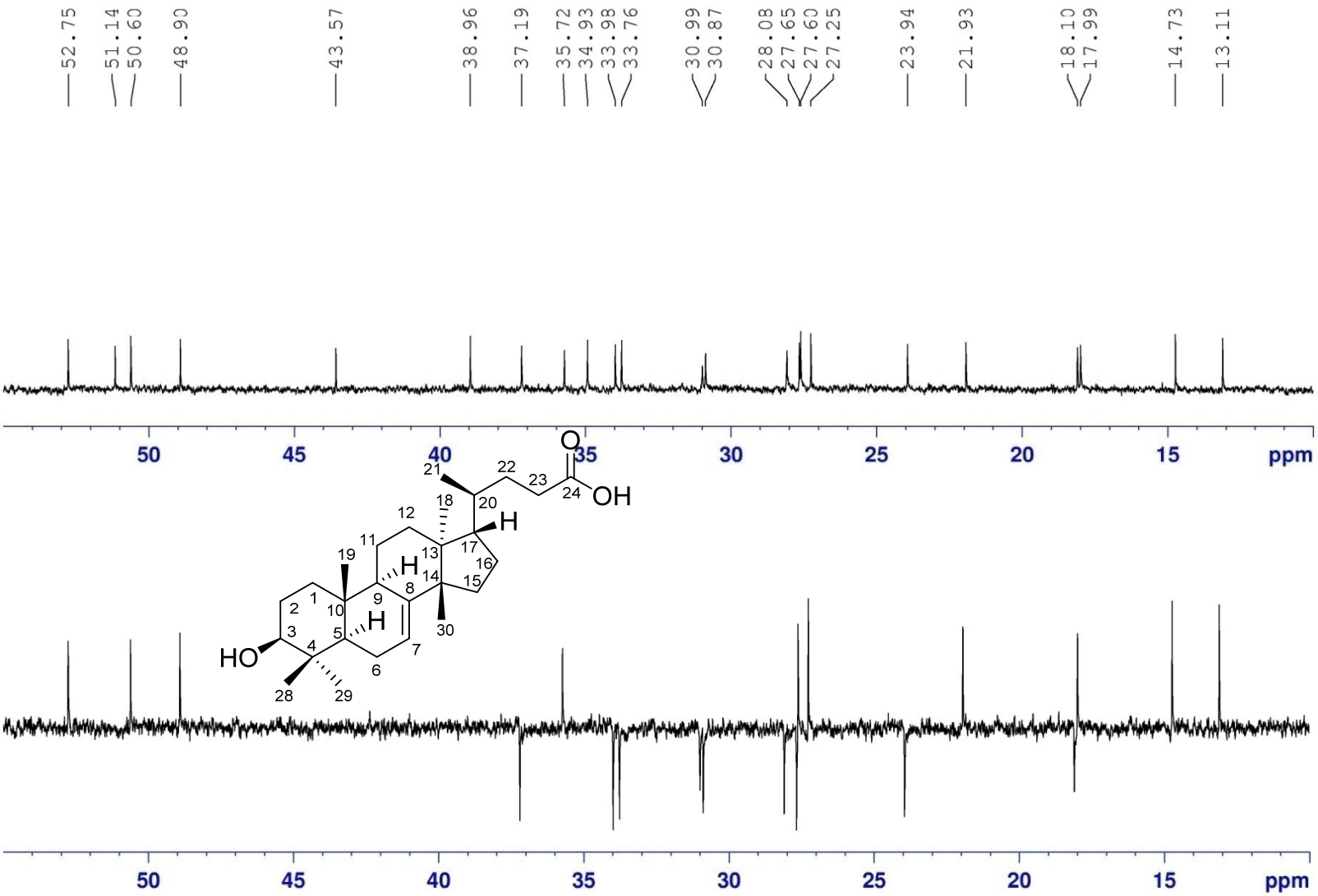


BRUKER

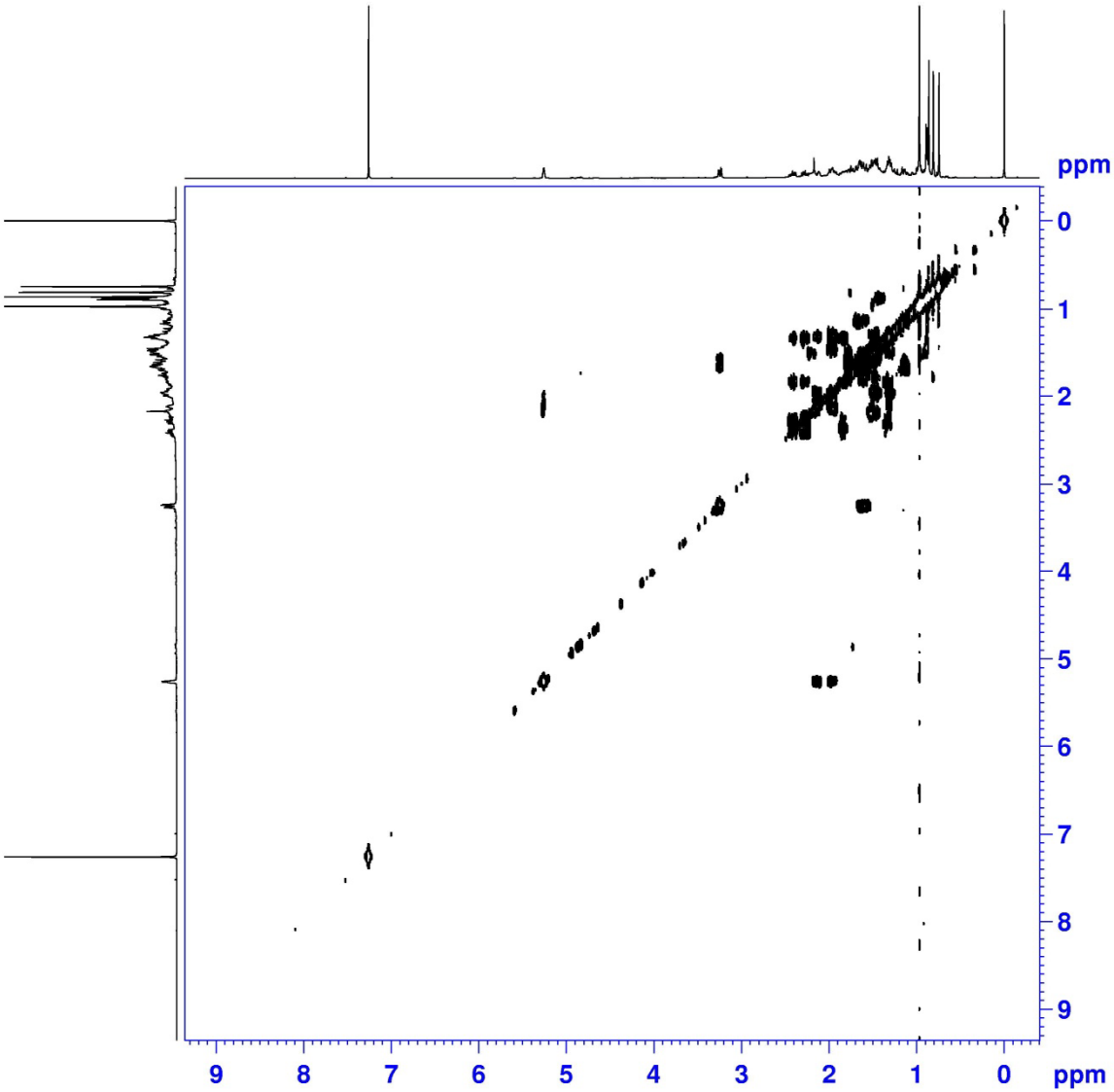
```
NAME        EA2-27(EA2-33-3)
EXPNO                      2
PROCNO                     1
Date_               20170613
Time                     4.30
INSTRUM                 spect
PROBHD       5 mm CPPBBO BB
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        500
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631988 sec
RG                      85.34
DW                     20.800 usec
DE                      18.00 usec
TE                      297.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            100.6233324 MHz
NUC1                     13C
P1                     10.00 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 **9** in $CDCl_3$



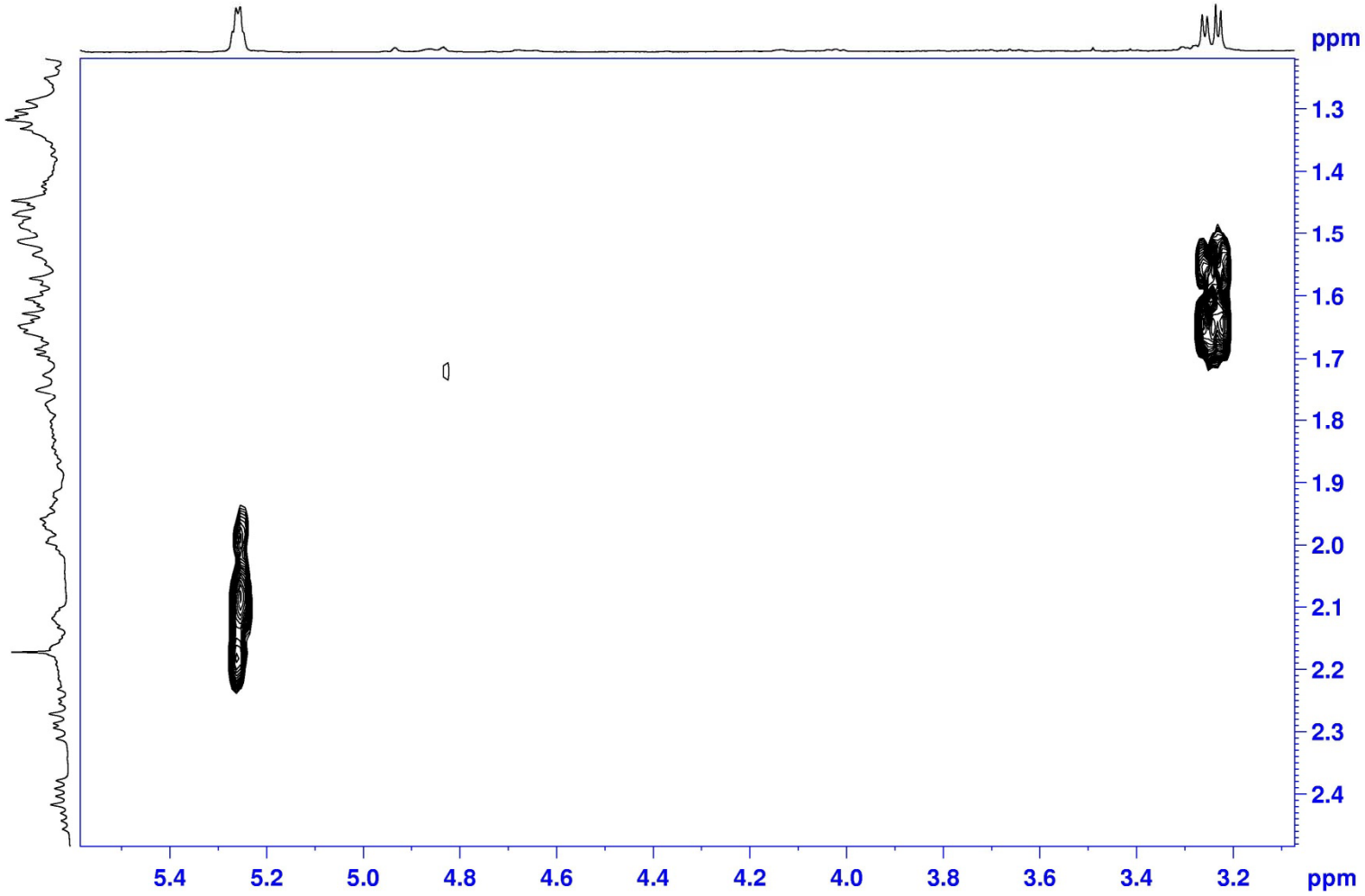
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **9** in $CDCl_3$



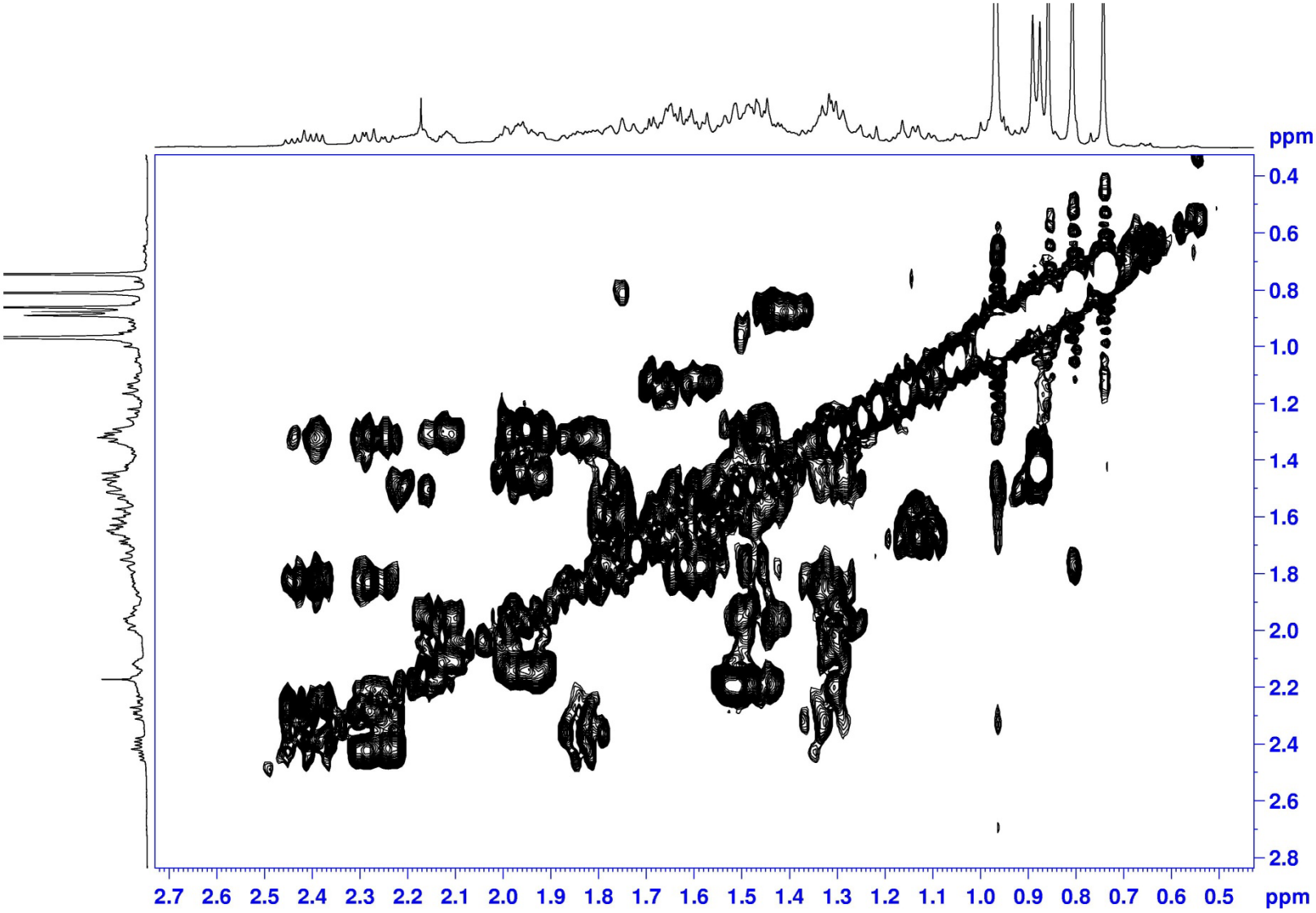

```
NAME      EA2-27(EA2-33-3)
EXPNO                  103
PROCNO                   1
Date_             20170620
Time                 12.26
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG       cosygpppqf
TD                    2048
SOLVENT             CDCl3
NS                      8
DS                      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             400.1300098 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
```

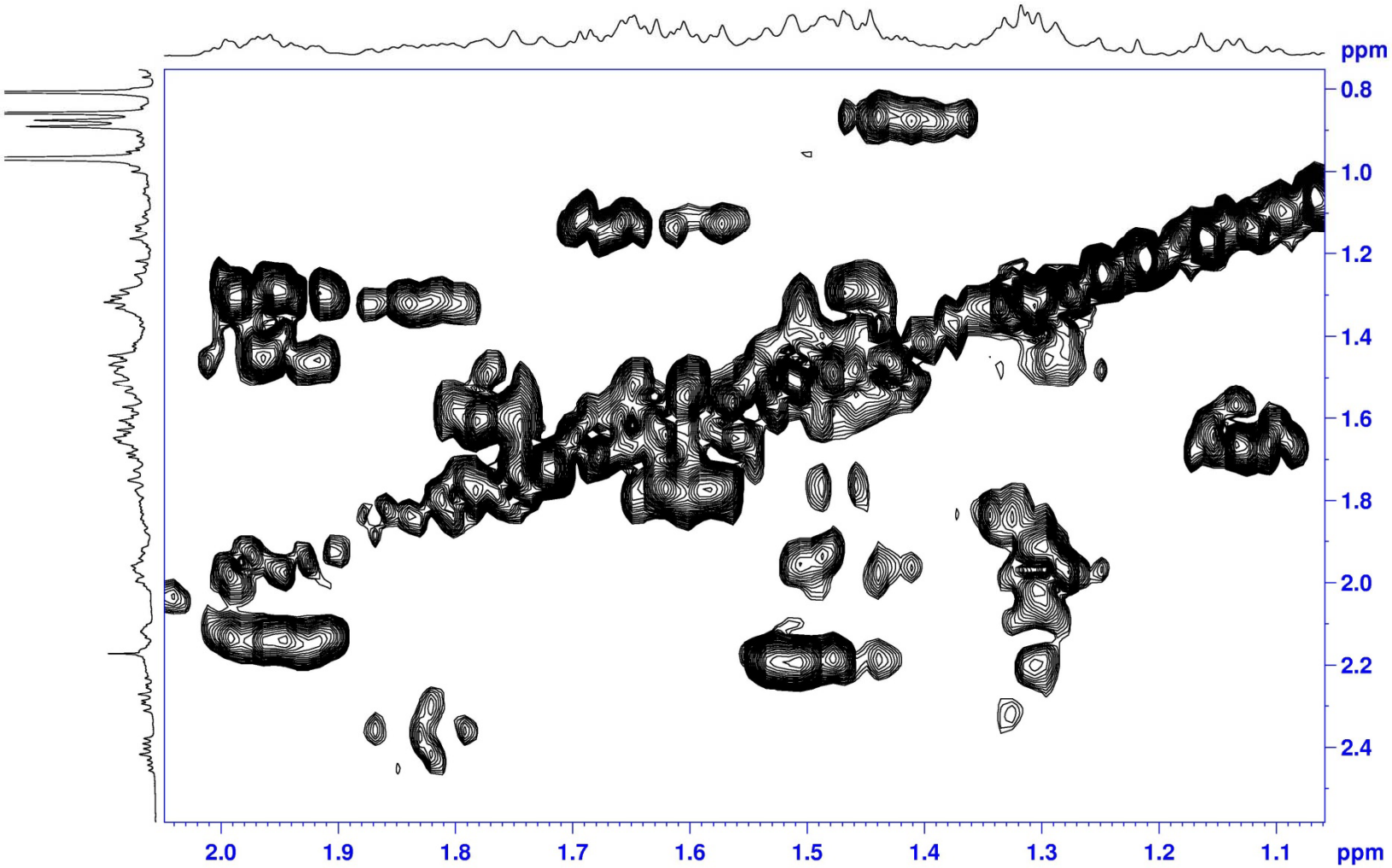
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **9** in $CDCl_3$



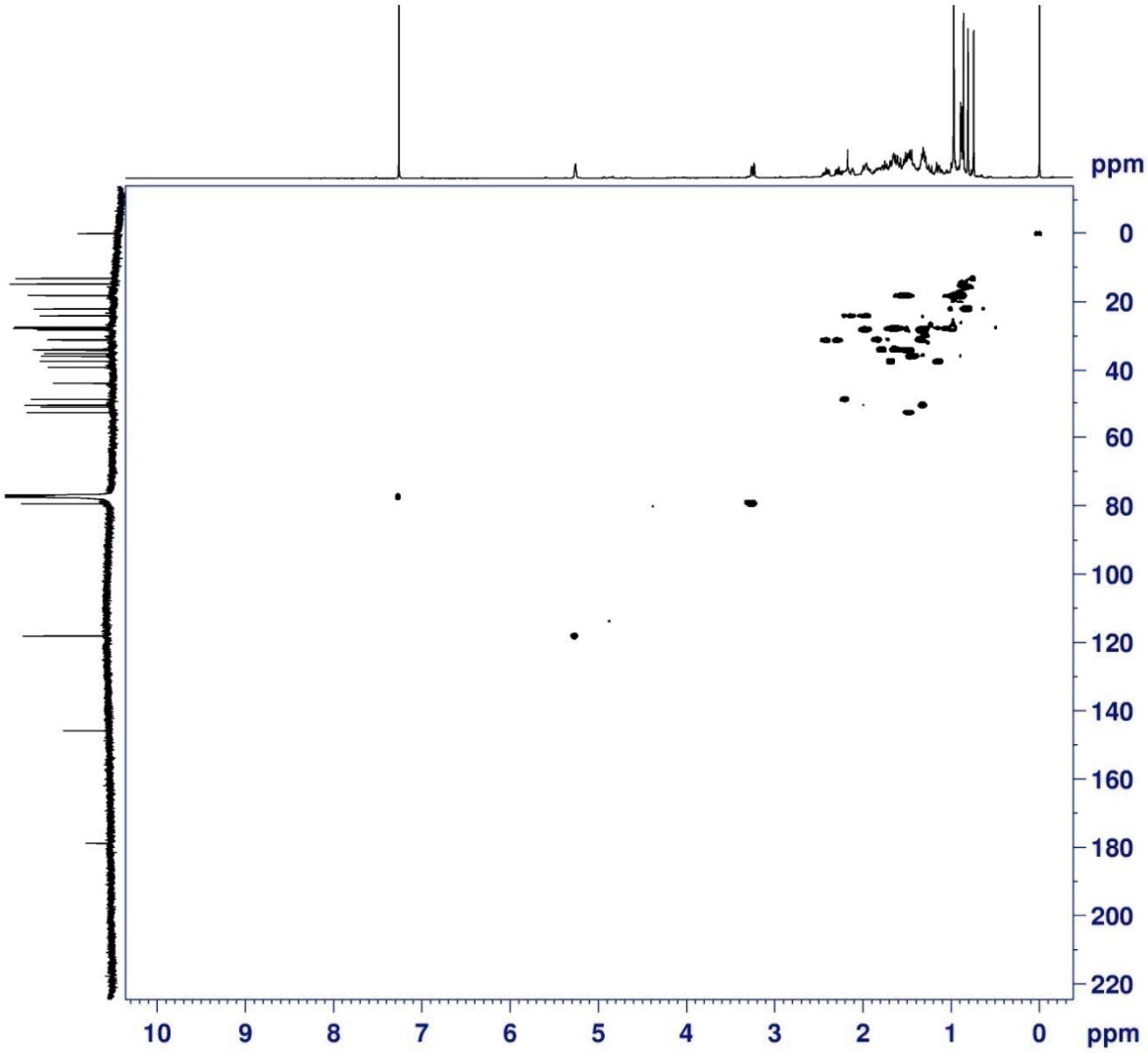
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **9** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **9** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **9** in $CDCl_3$




```
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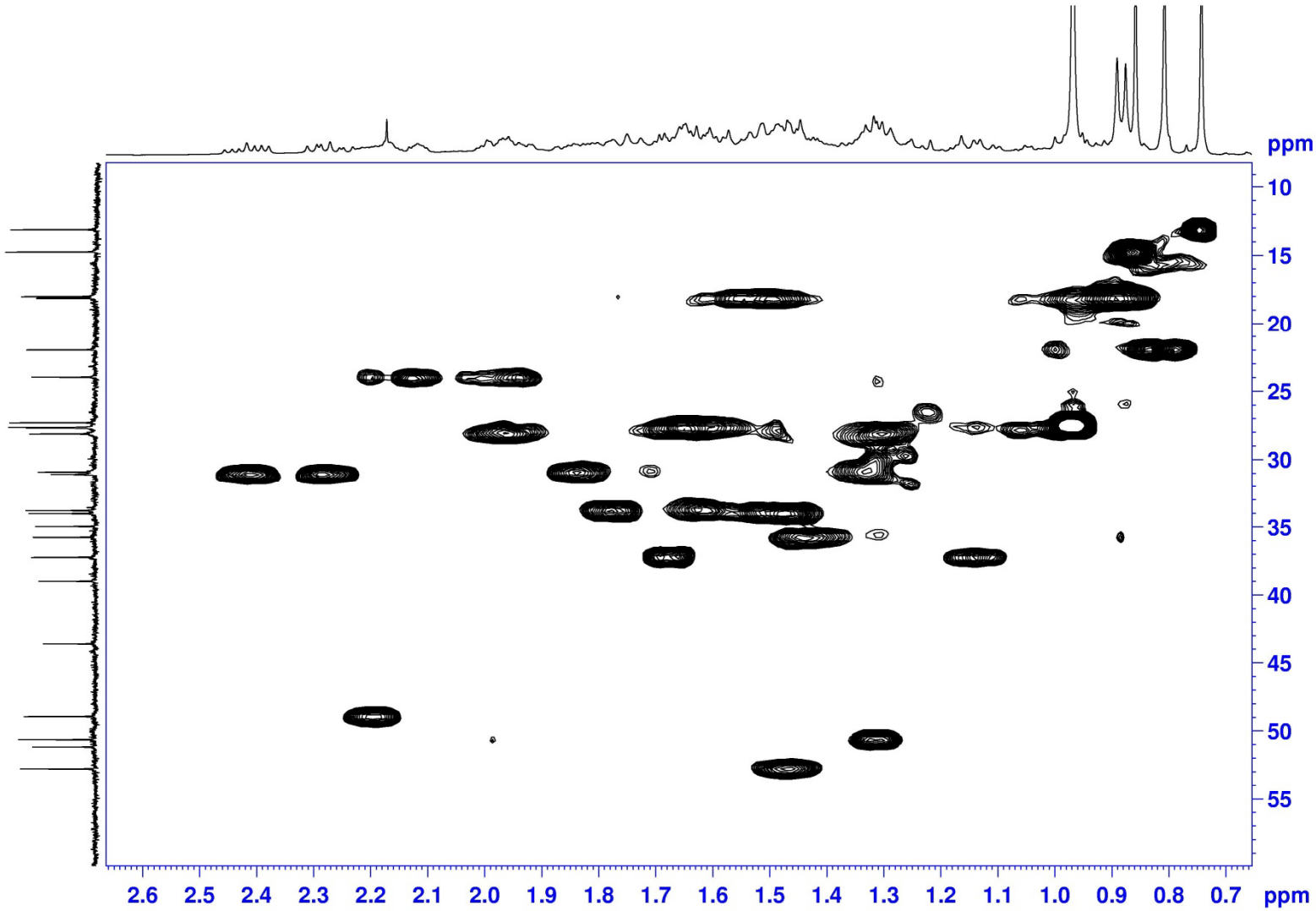
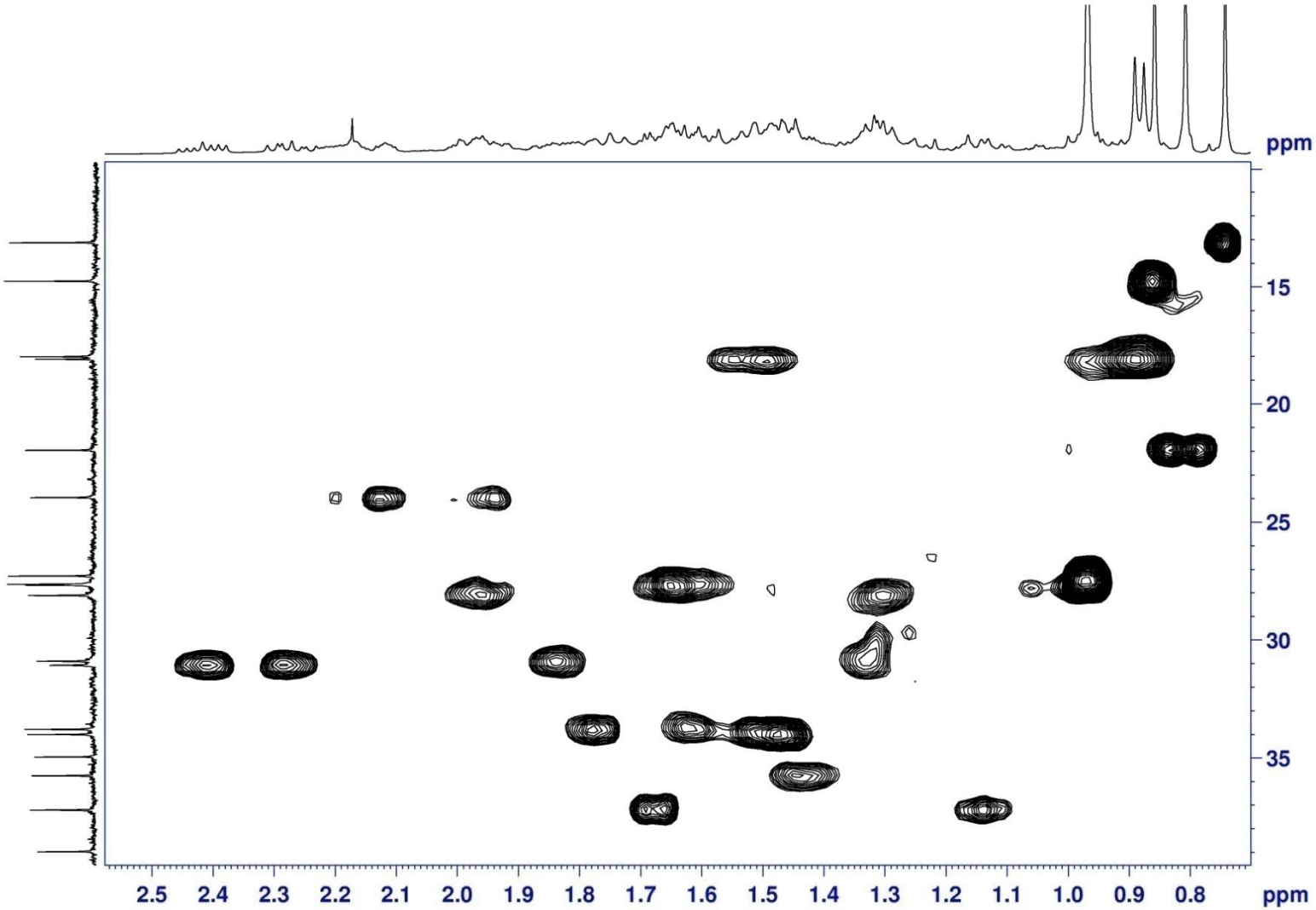
EXPNO                    104
PROCNO                     1
Date_               20170620
Time                   13.06
INSTRUM                spect
PROBHD     5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD                      1024
SOLVENT                CDCl3
NS                        16
DS                        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
ZGOPTNS

======== CHANNEL f1 ========
SFO1              400.1320007 MHz
NUC1                       1H
P1                      11.50 usec
P2                      23.00 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                400.1300068 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40
SI                      1024
MC2            echo-antiecho
SF                 100.6127543 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

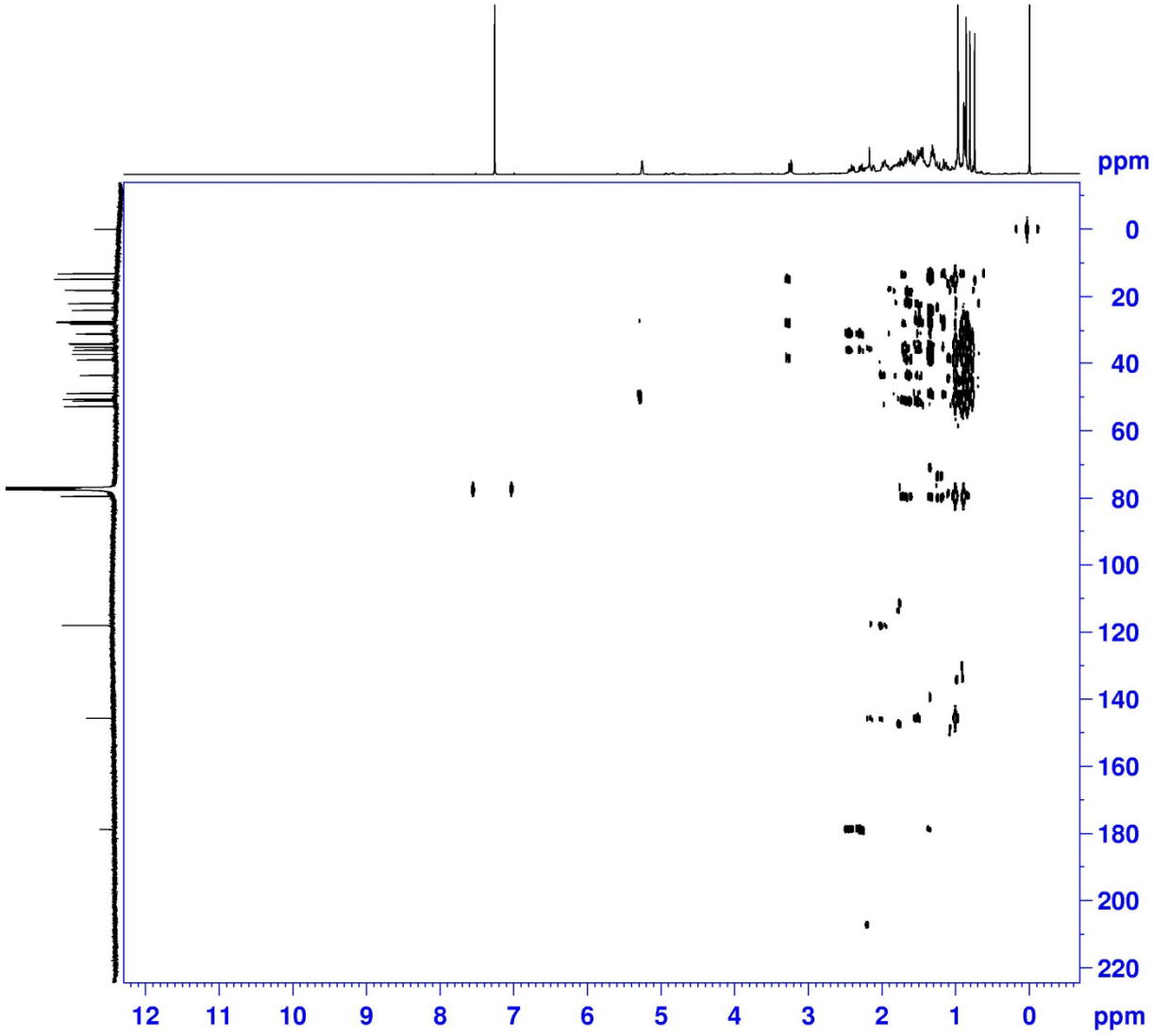
HSQC (400 MHz) spectrum of compound **9** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **9** in $CDCl_3$



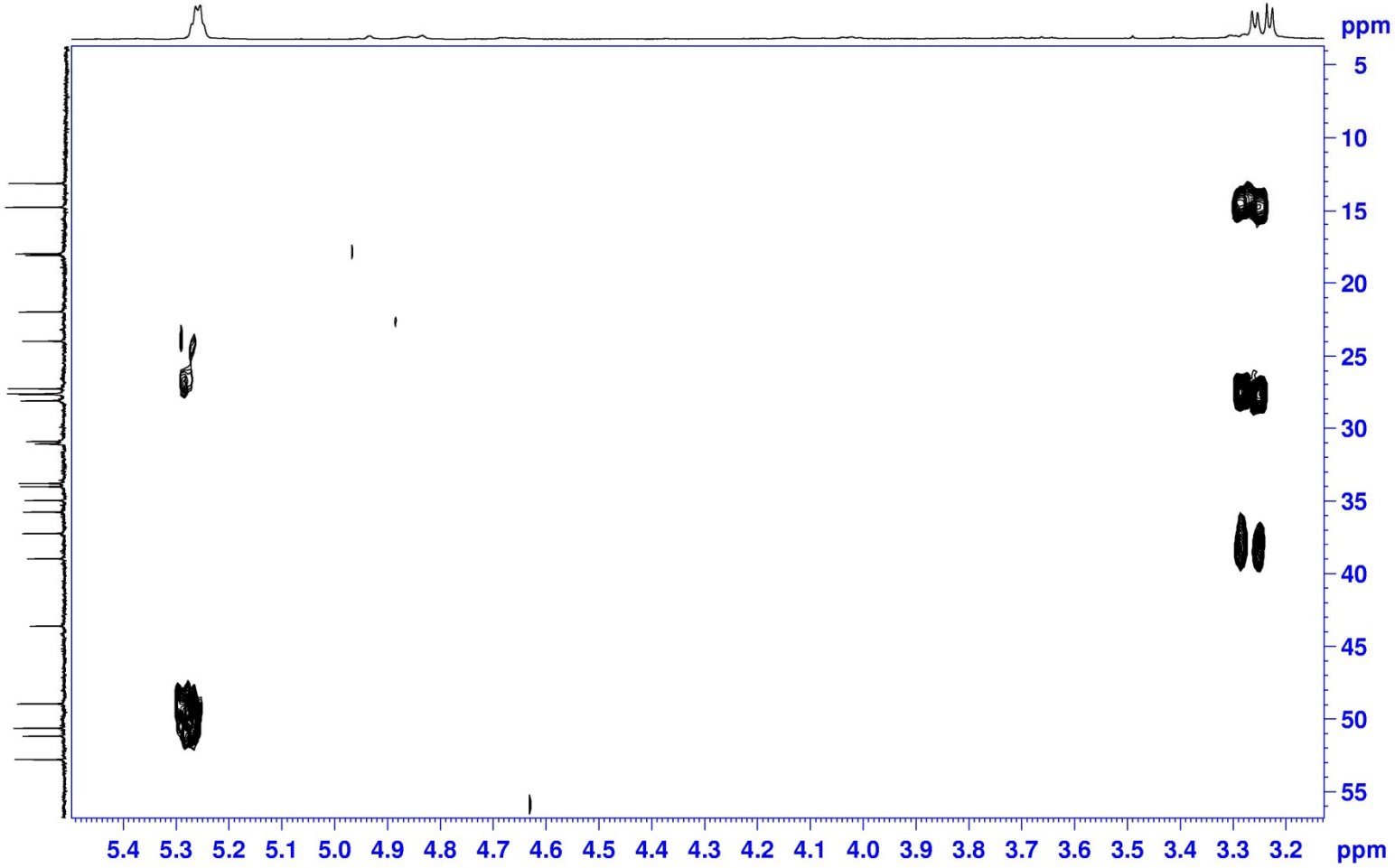
HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



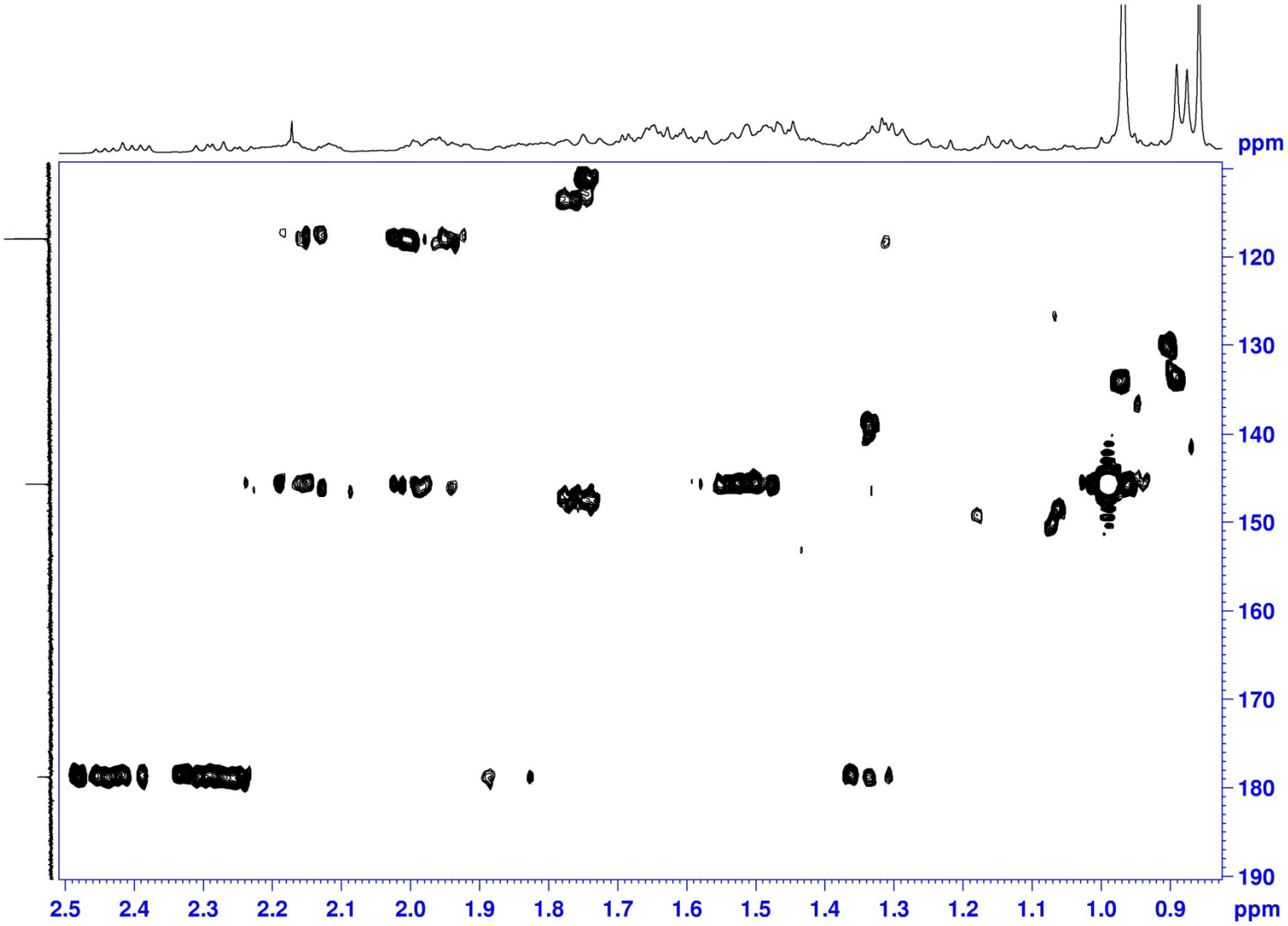

```
NAME        EA2-27(EA2-33-3)
EXPNO                   1006
PROCNO                     1
Date_               20170620
Time                   23.02
INSTRUM                spect
PROBHD     5 mm CPPBBO BB
PULPROG      hmbcgplpndqf
TD                      4096
SOLVENT                CDCl3
NS                        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
FnMODE                     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
```

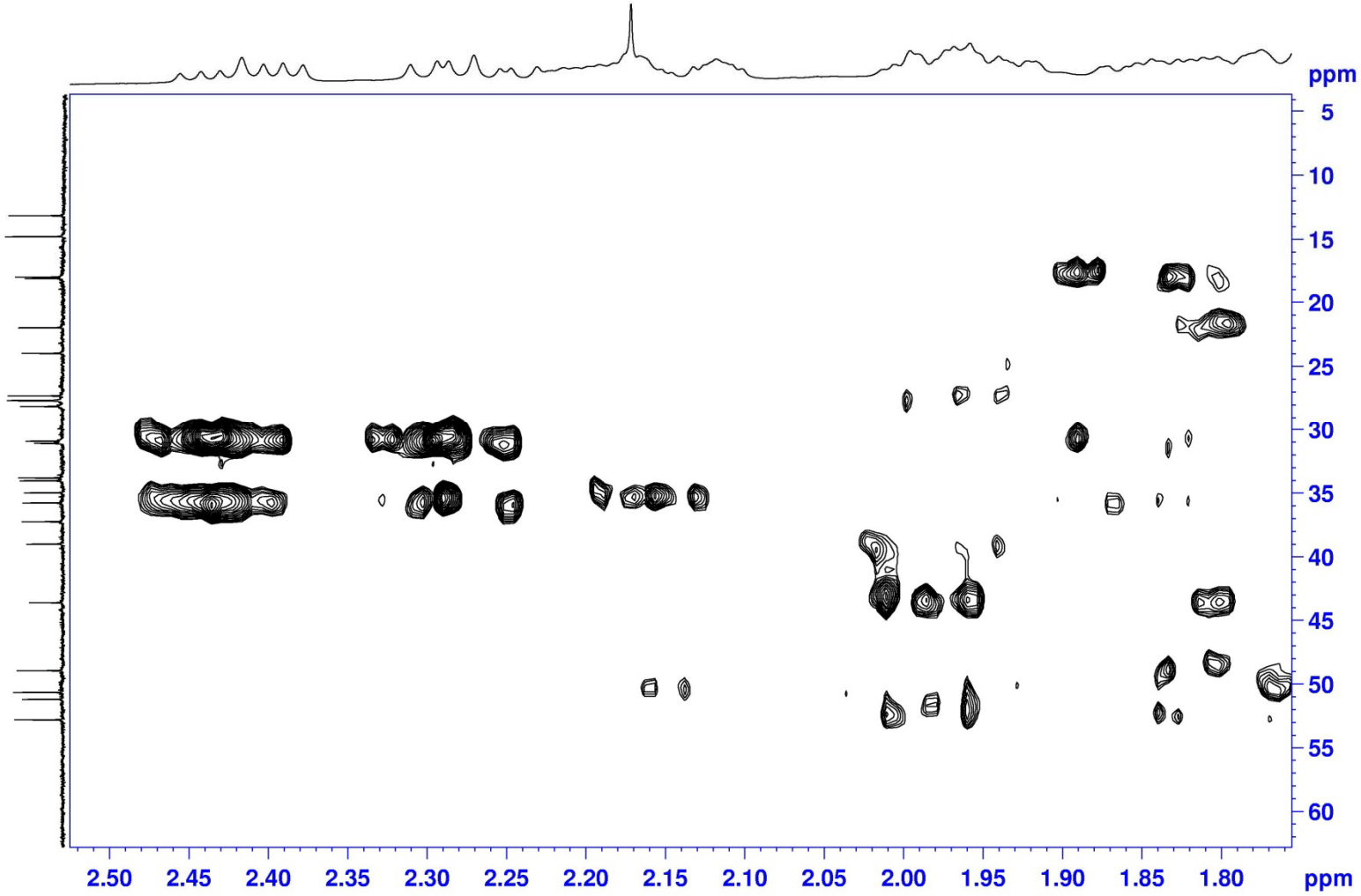
HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



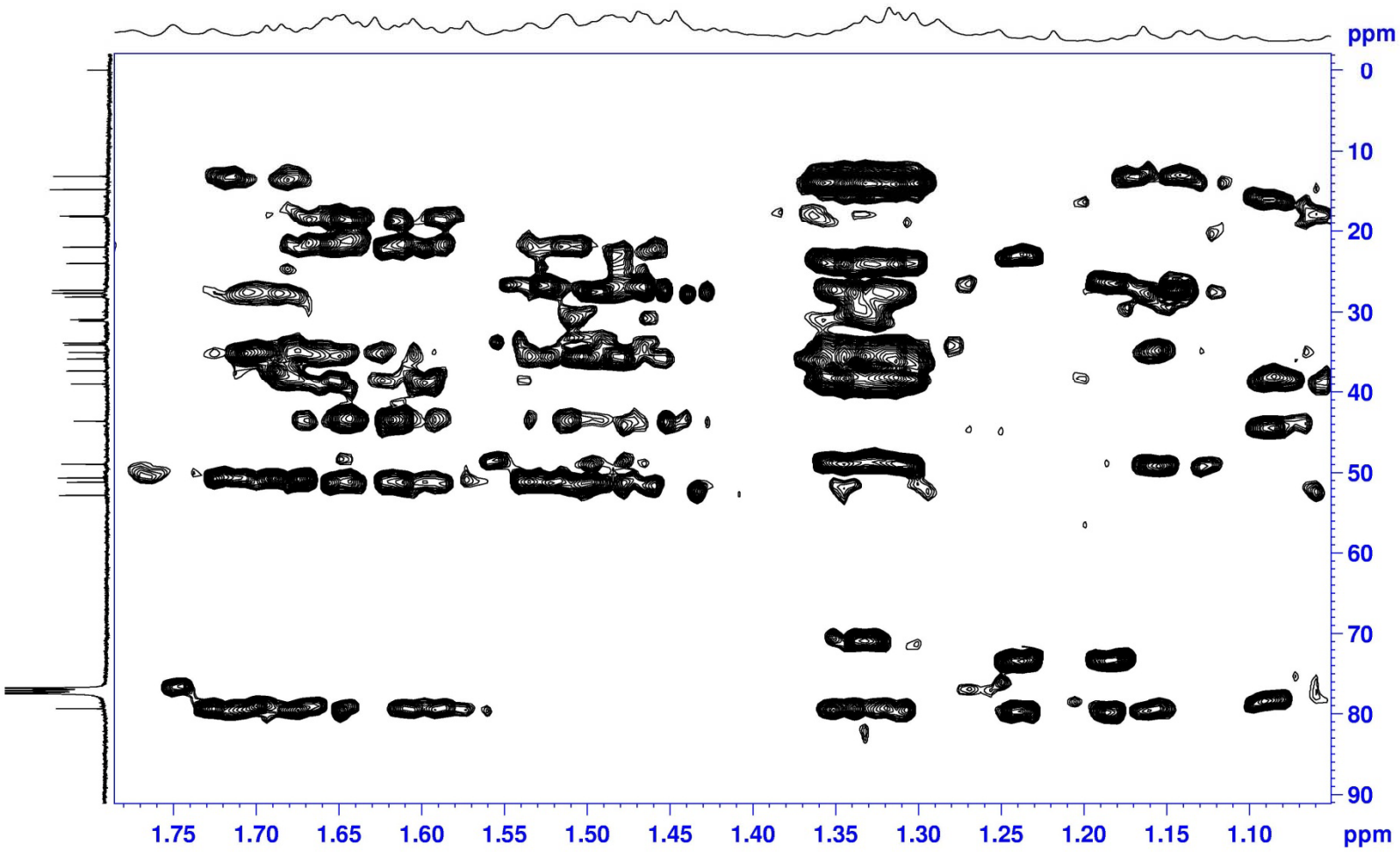
HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



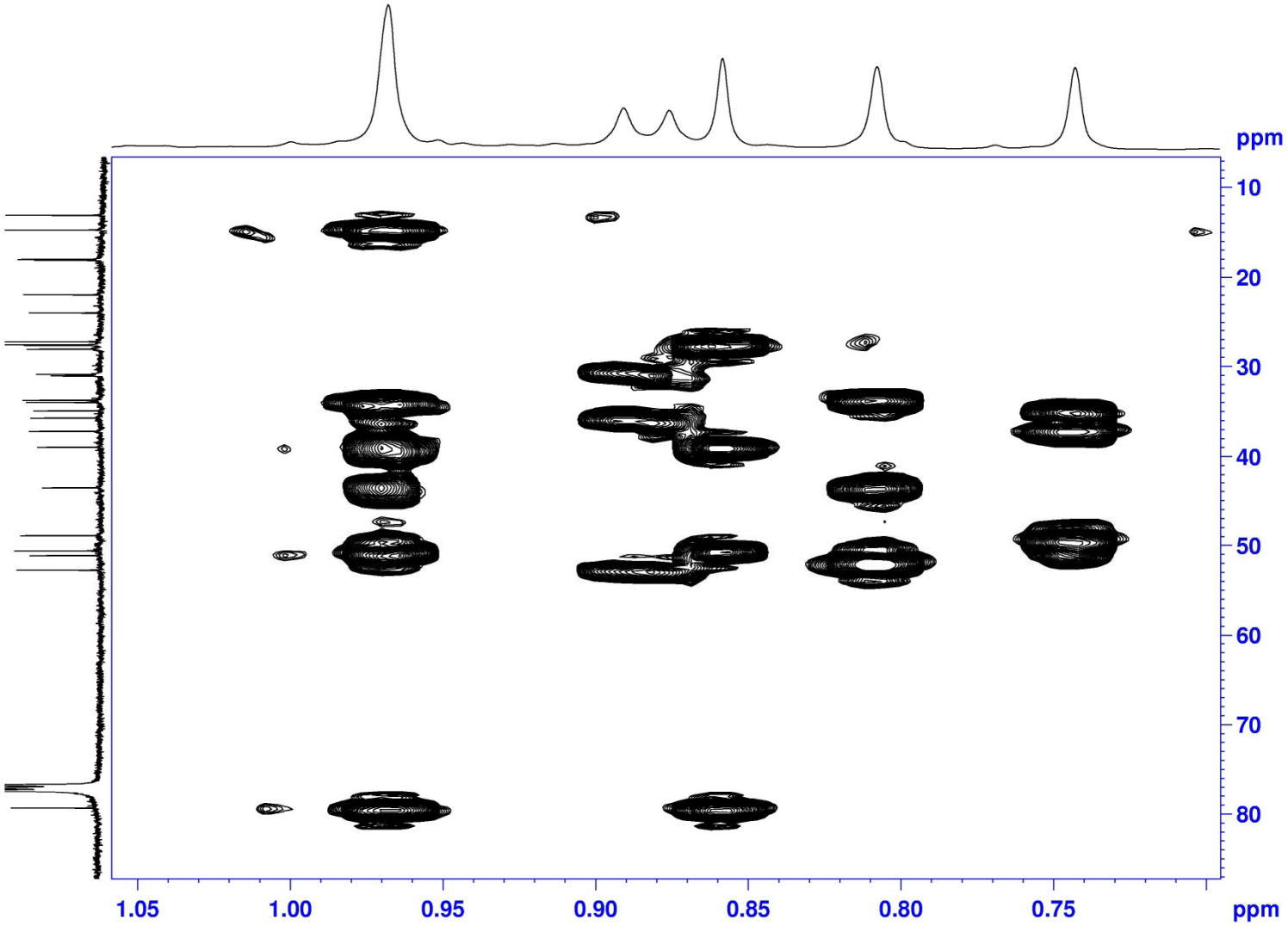
HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



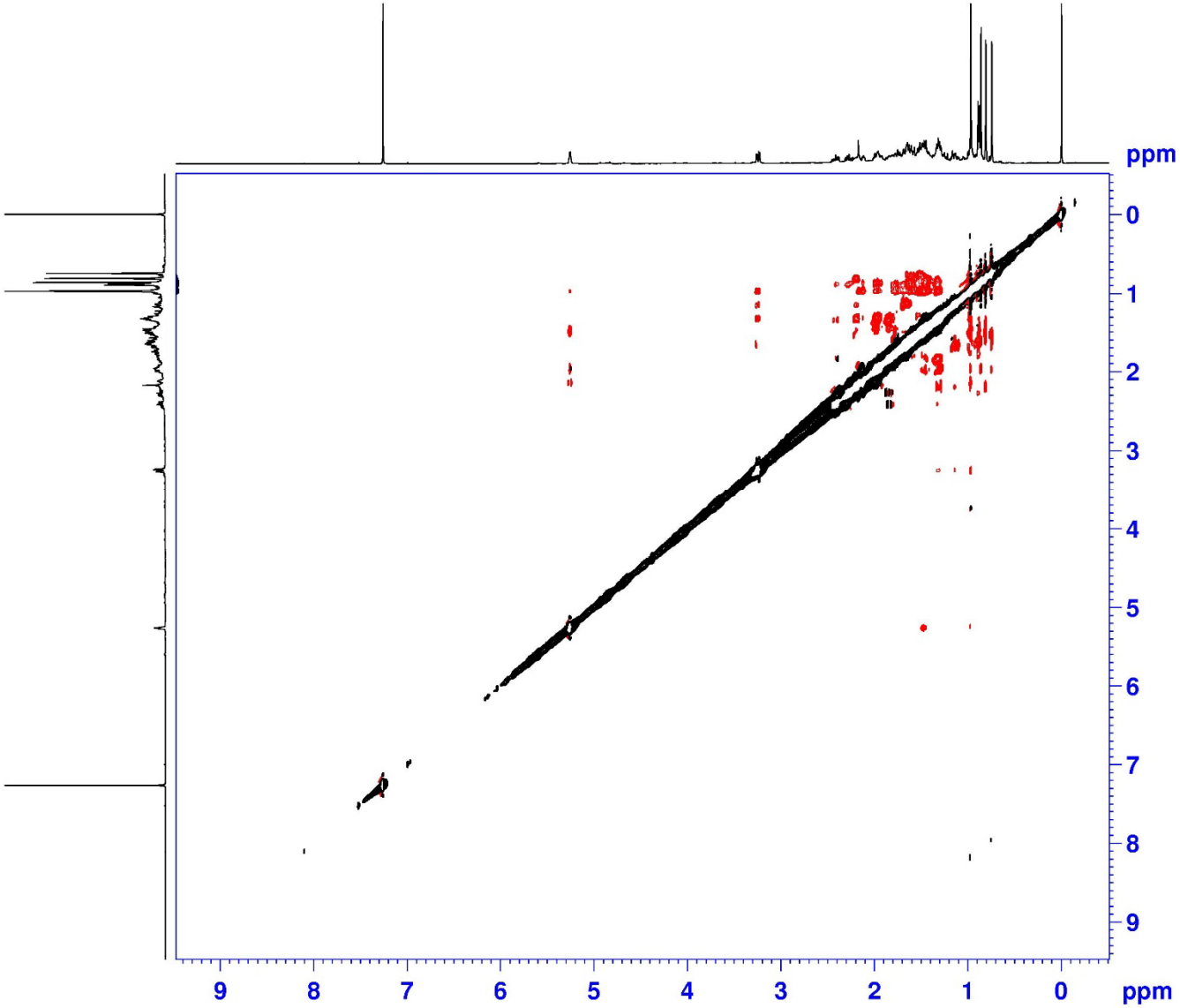
HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **9** in $CDCl_3$



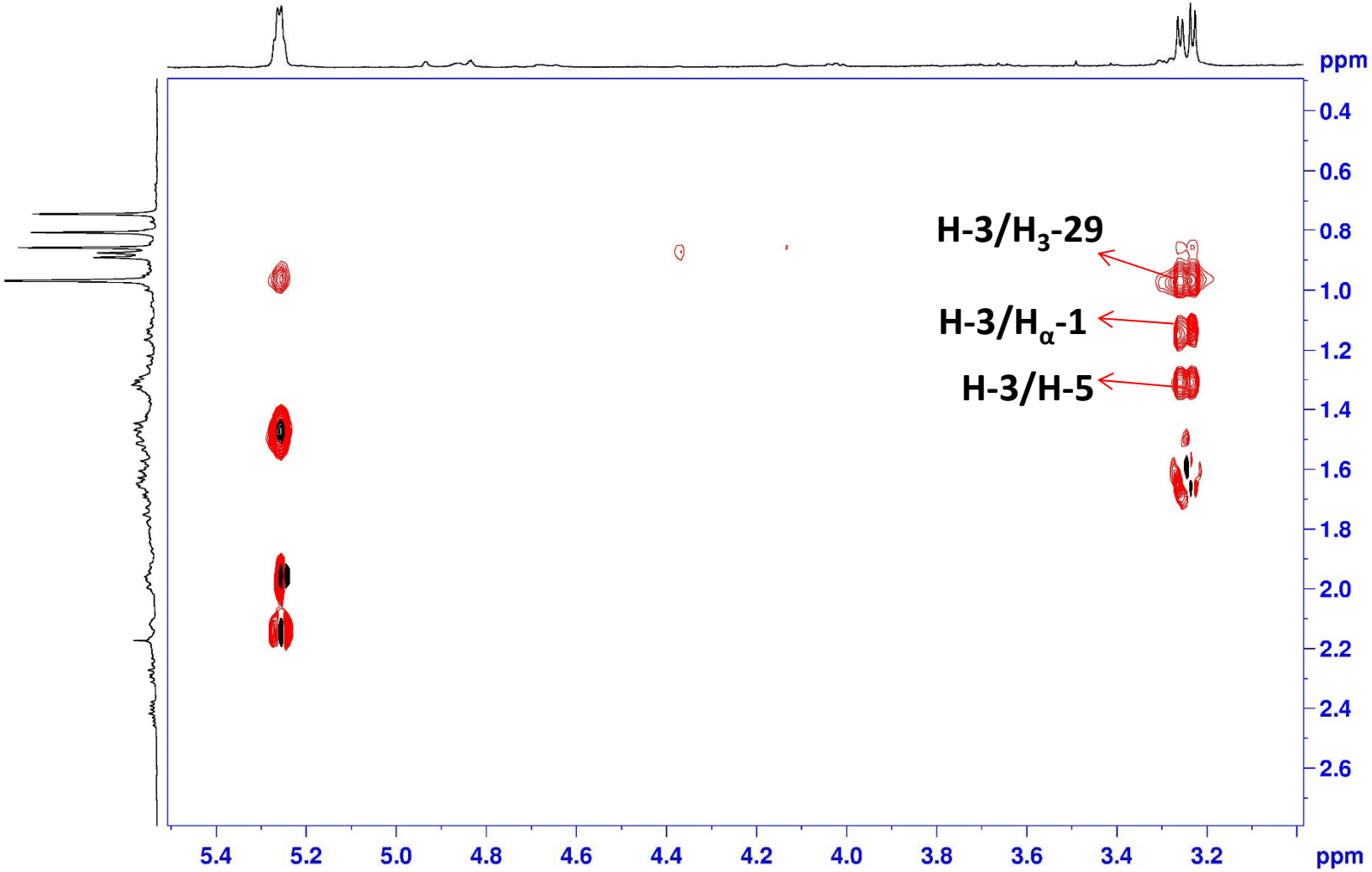
NOESY (400 MHz) spectrum of compound **9** in $CDCl_3$



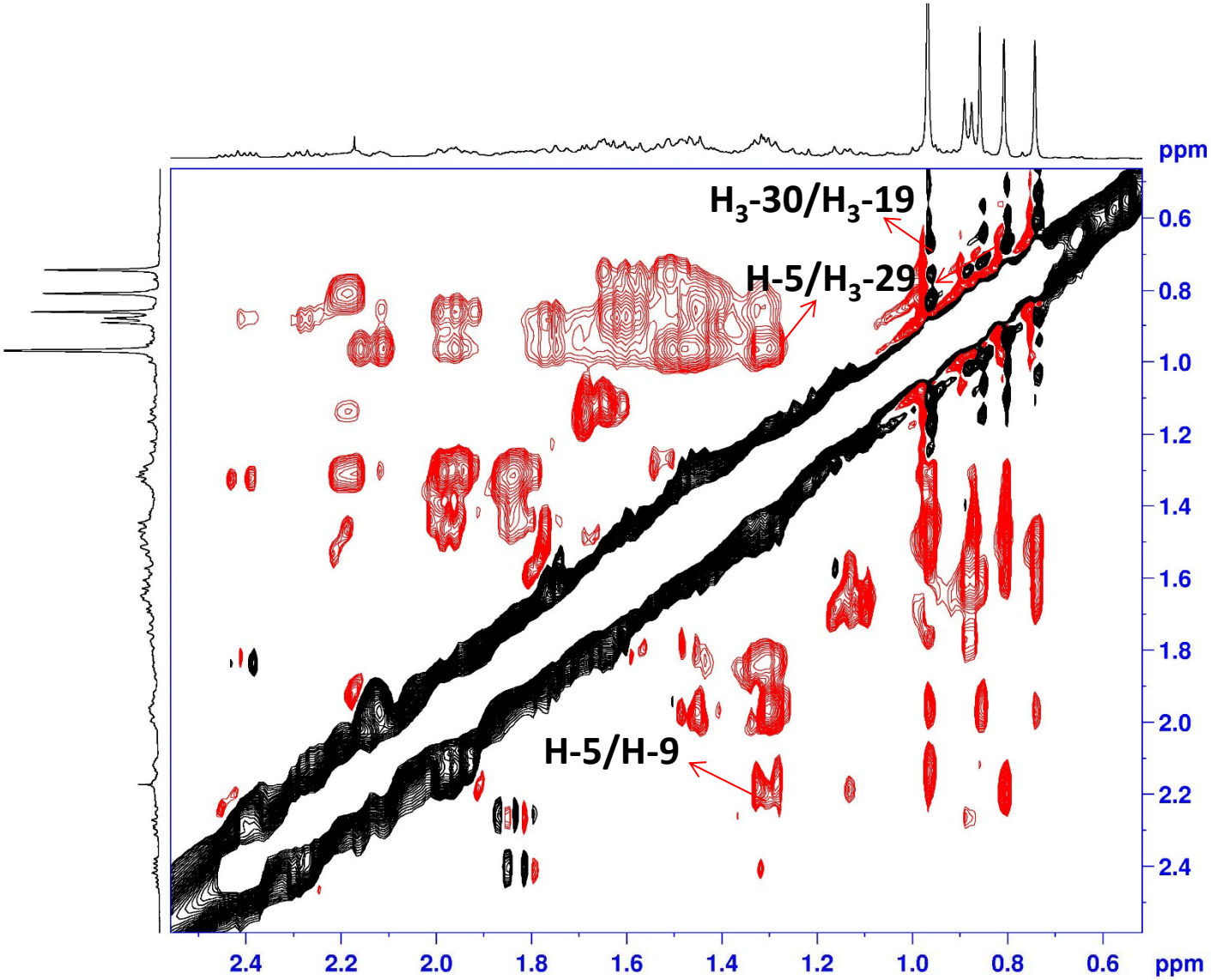

```
NAME        EA2-27(EA2-33-3)
EXPNO                   1007
PROCNO                     1
Date_               20170621
Time                    3.31
INSTRUM                spect
PROBHD     5 mm CPPBBO BB
PULPROG        noesygpphpp
TD                      2048
SOLVENT                CDCl3
NS                         32
DS                         32
SWH                 4000.000 Hz
FIDRES              1.953125 Hz
AQ                 0.2560500 se
RG                     208.5
DW                   125.000 us
DE                     10.00 us
TE                     297.0 K
D0                 0.00011036 se
D1                 1.99385595 se
D8                 0.30000001 se
D11                0.03000000 se
D12                0.00002000 se
D16                0.00020000 se
IN0                0.00025000 se

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

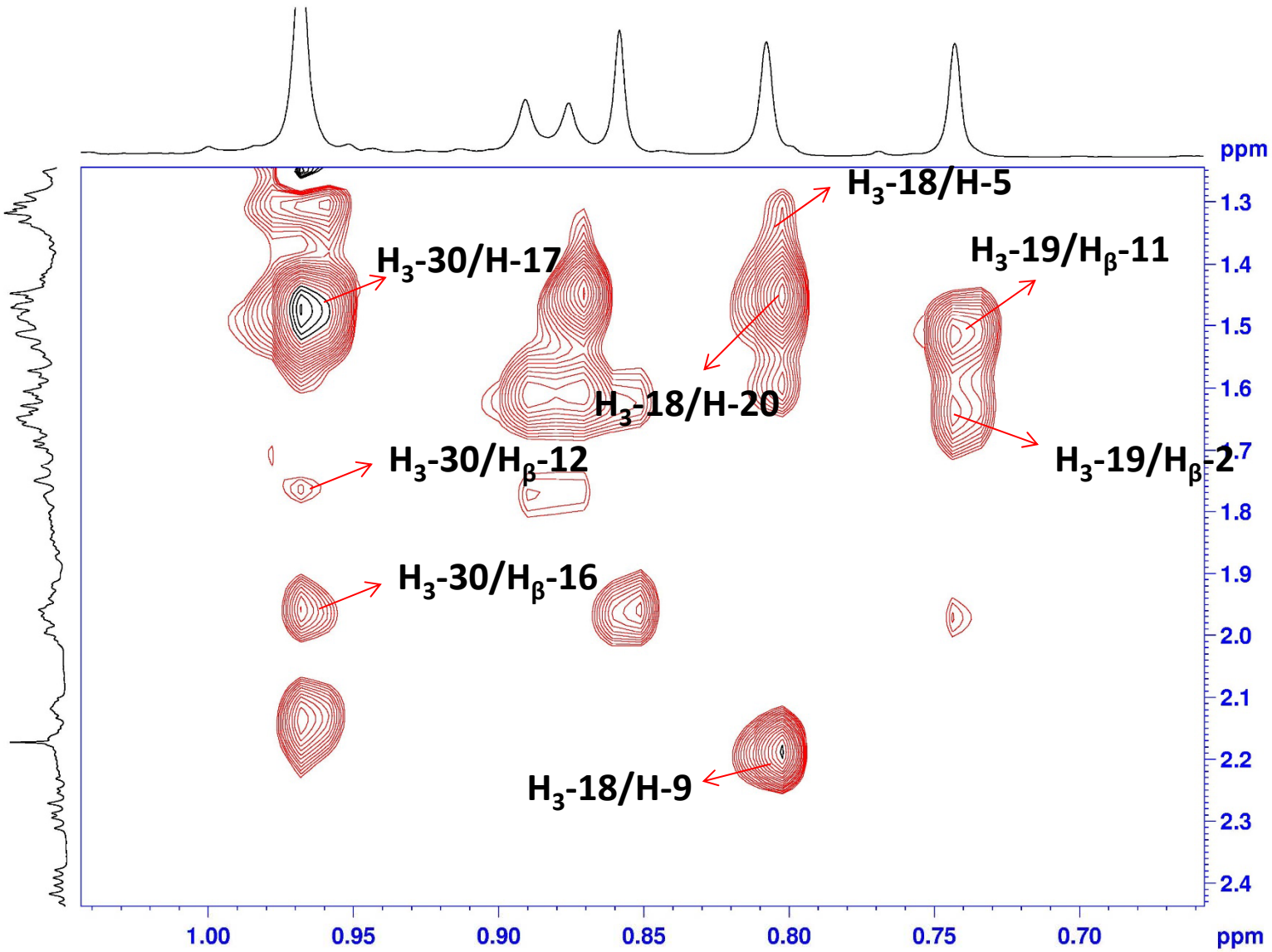
NOESY (400 MHz) spectrum of compound **9** in $CDCl_3$



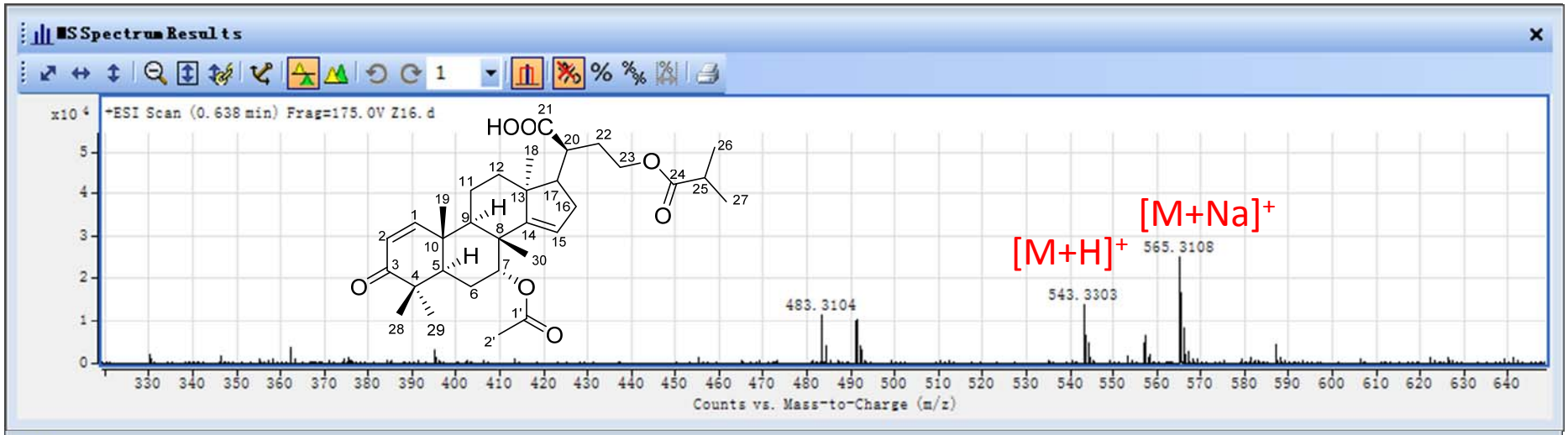
NOESY (400 MHz) spectrum of compound **9** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **9** in $CDCl_3$



HR-ESIMS for compound **10**



MS Formula Results: + Scan (0.638 min)

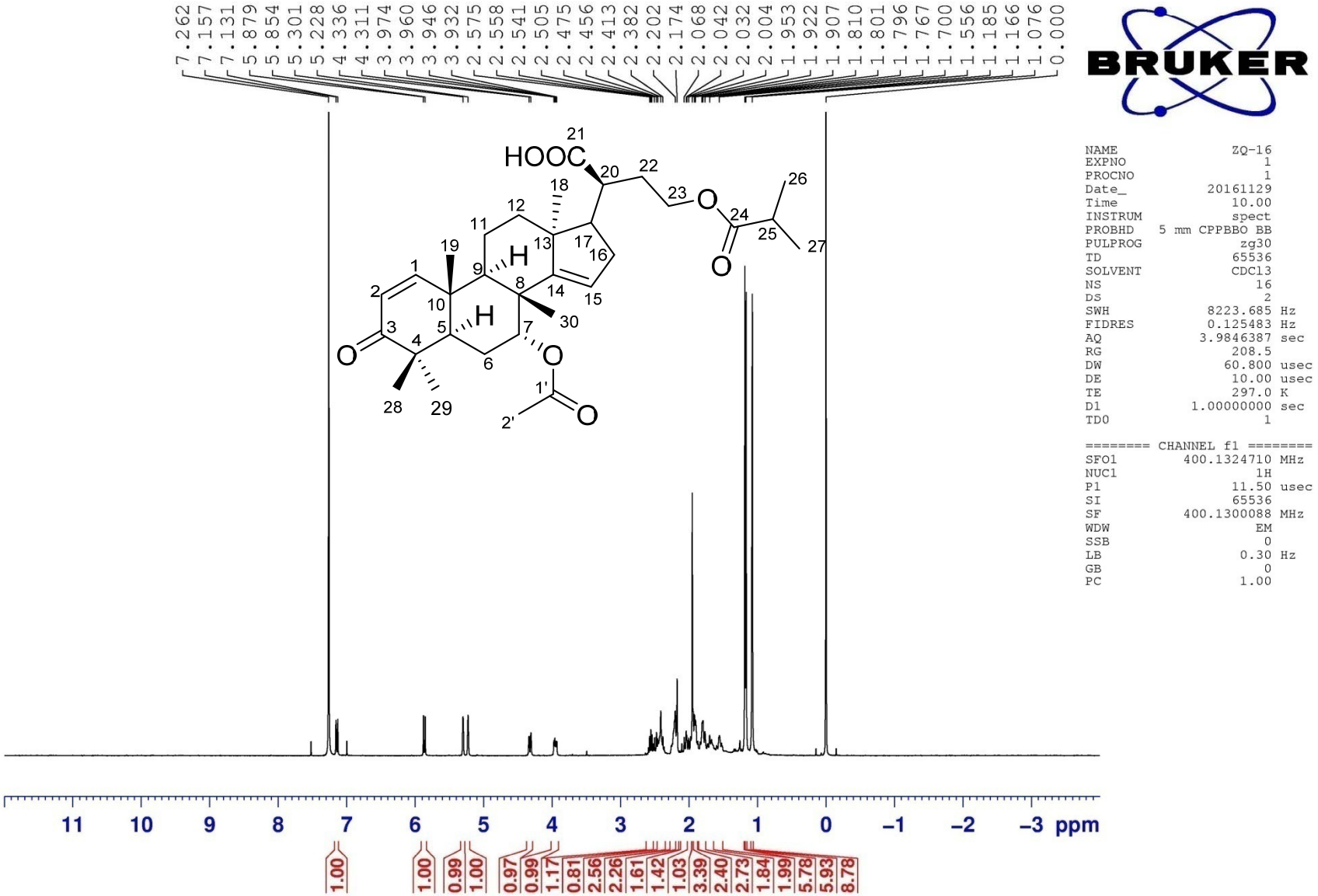
m/z	Ion	Formula	Abundance
543.3303	(M+H)+	C32 H47 O7	13701.3

Best	Formula (M)	Ion Formula	Score	Cross Score	Calc m/z	Diff (ppm)	Mass Match	Abund Match	Spacing Match
✓	C32 H46 O7	C32 H47 O7	94.53		543.3316	2.58	92.57	97.86	94.46

Isotope	Abund Sum%	Calc Abund Sum%	m/z	Calc m/z	Diff (ppm)
1	70.87	69.38	543.3303	543.3316	2.51
2	24.01	24.57	544.3327	544.335	4.31
3	4.26	5.22	545.3389	545.338	-1.66
4	0.86	0.82	546.3466	546.3408	-10.67

Best	Formula (M)	Ion Formula	Score	Cross Score	Calc m/z	Diff (ppm)	Mass Match	Abund Match	Spacing Match
	C39 H42 O2	C39 H43 O2	65.16		543.3258	-8.25	45.53	72.77	95.26
	C26 H51 Cl O9	C26 H52 Cl O9	62.58		543.3294	-1.46	97.56	0.14	67.56

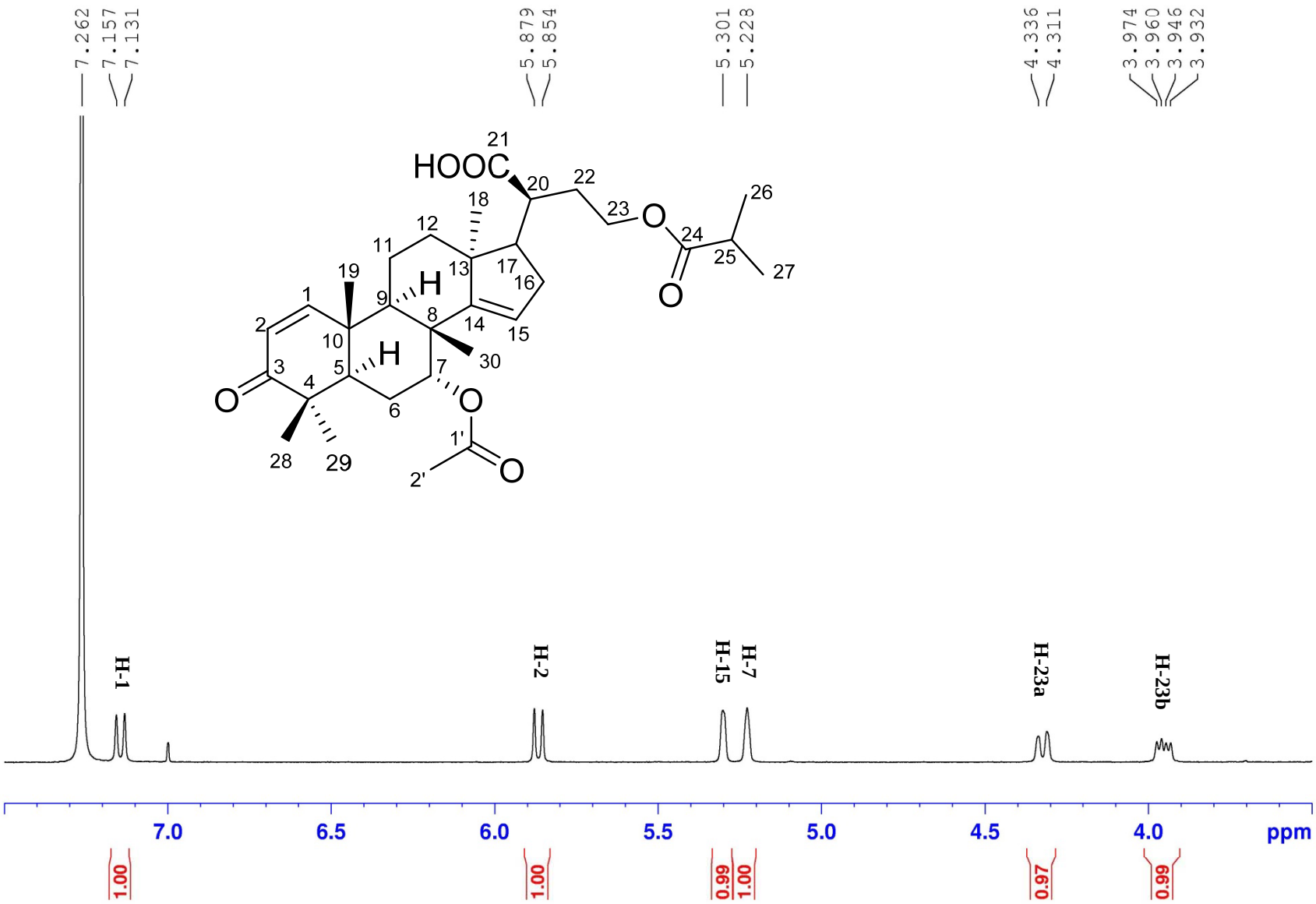
^{1}H NMR (400 MHz) spectrum of compound **10** in $CDCl_3$



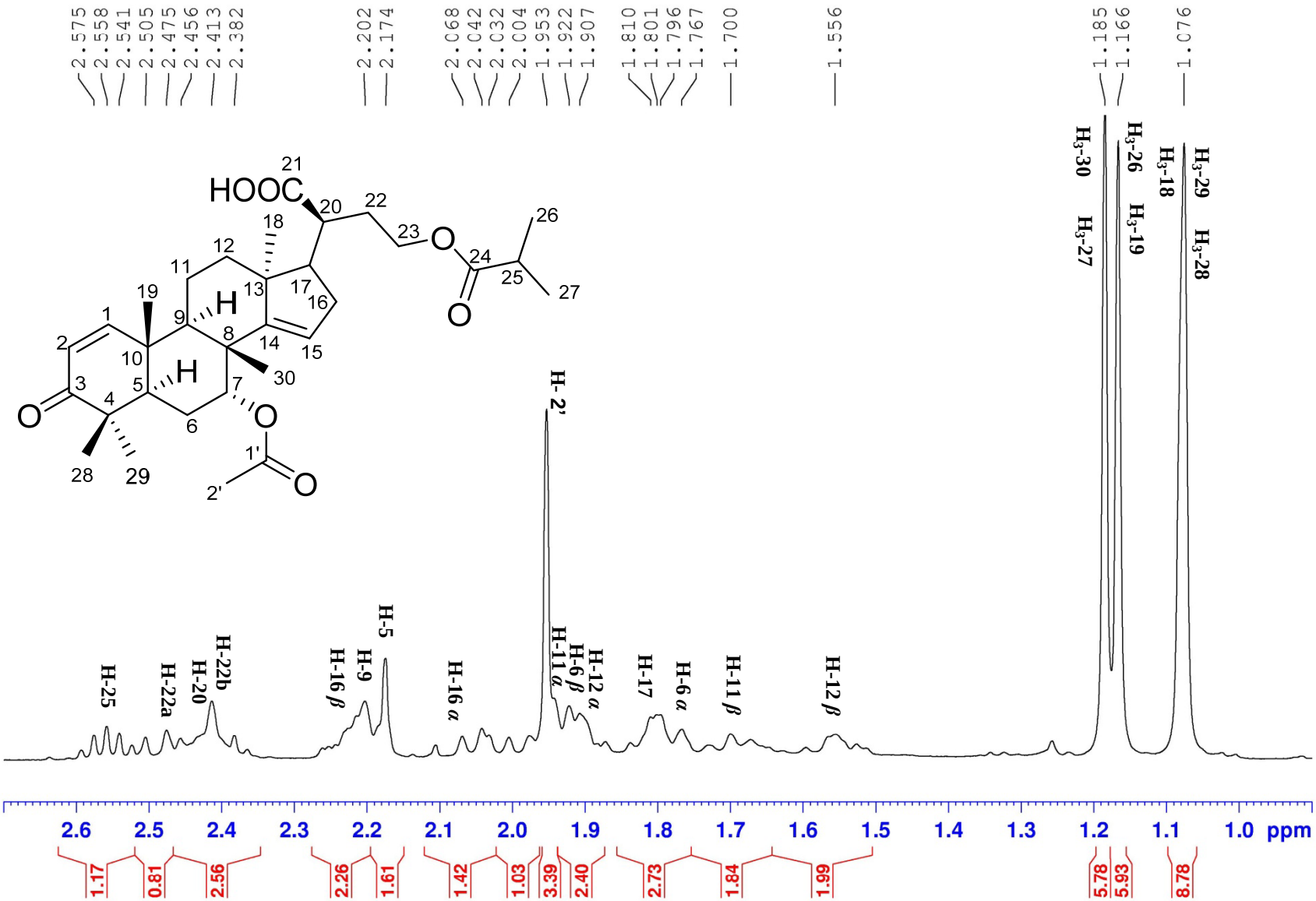

```
NAME              ZQ-16
EXPNO                 1
PROCNO                1
Date_          20161129
Time              10.00
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ           3.9846387 sec
RG               208.5
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               11.50 usec
SI               65536
SF          400.1300088 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

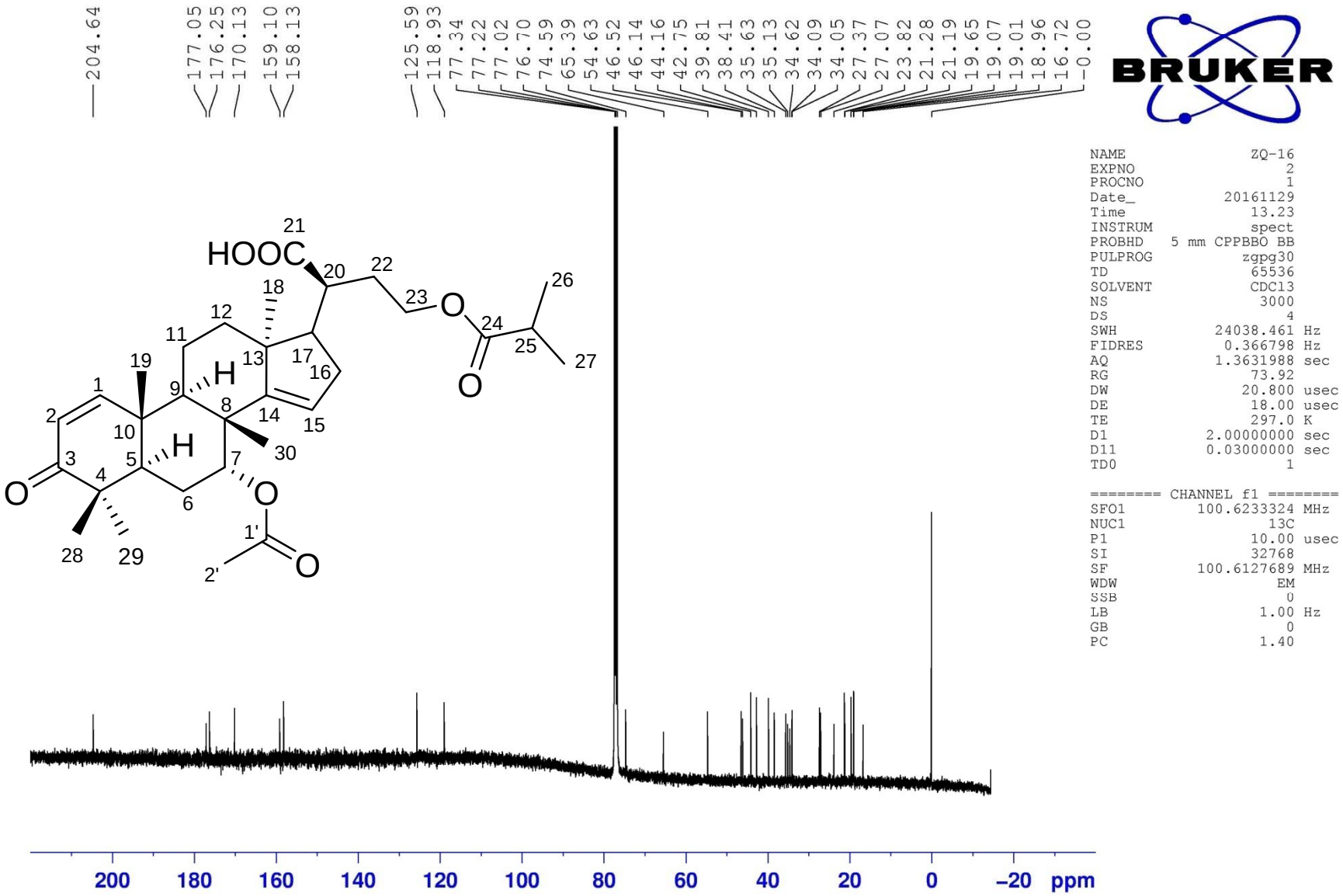
^{1}H NMR (400 MHz) spectrum of compound **10** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **10** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **10** in $CDCl_3$

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NAME                ZQ-16
EXPNO                   2
PROCNO                  1
Date_            20161129
Time                13.23
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   3000
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                  73.92
DW                 20.800 usec
DE                 18.00 usec
TE                 297.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

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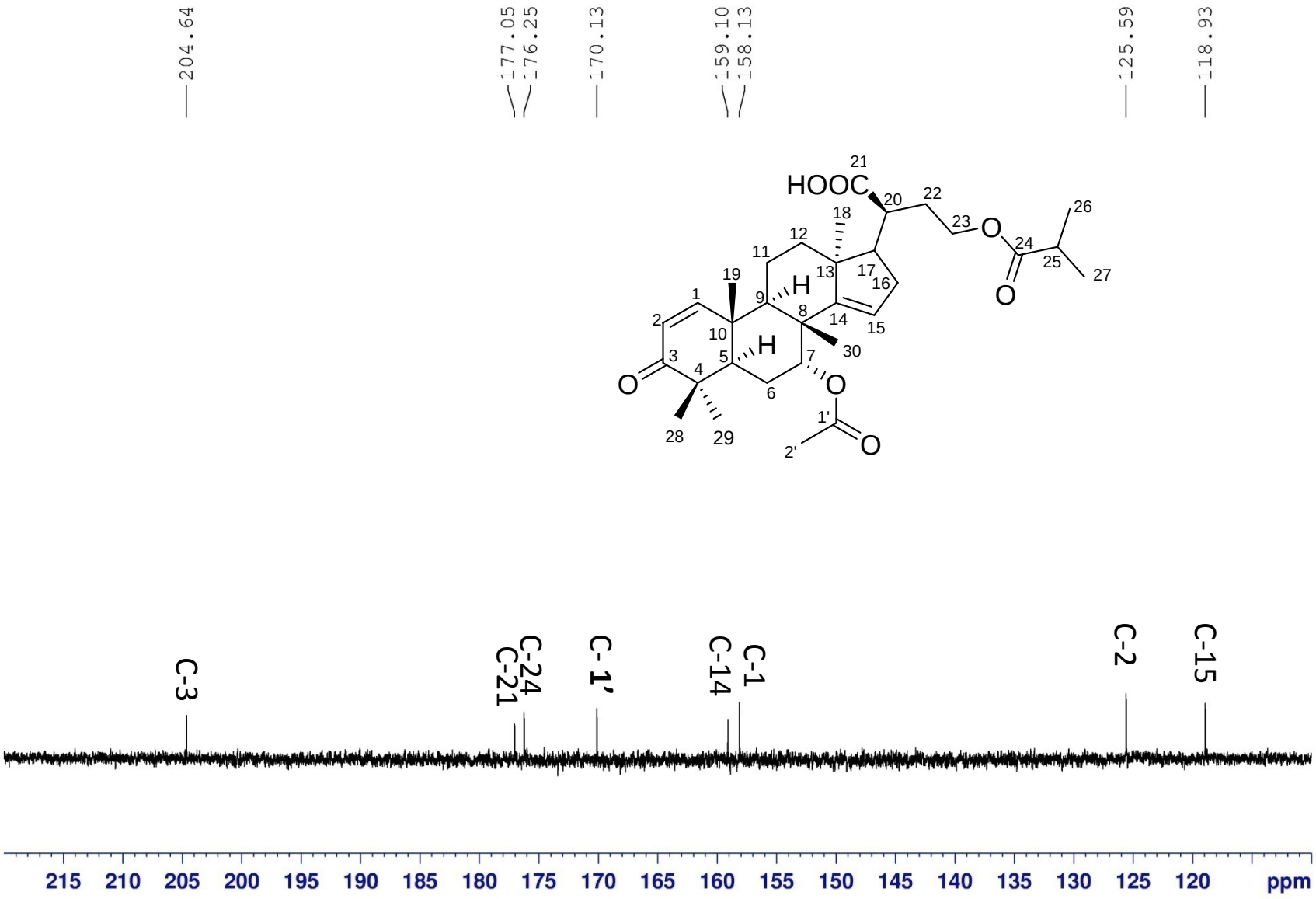
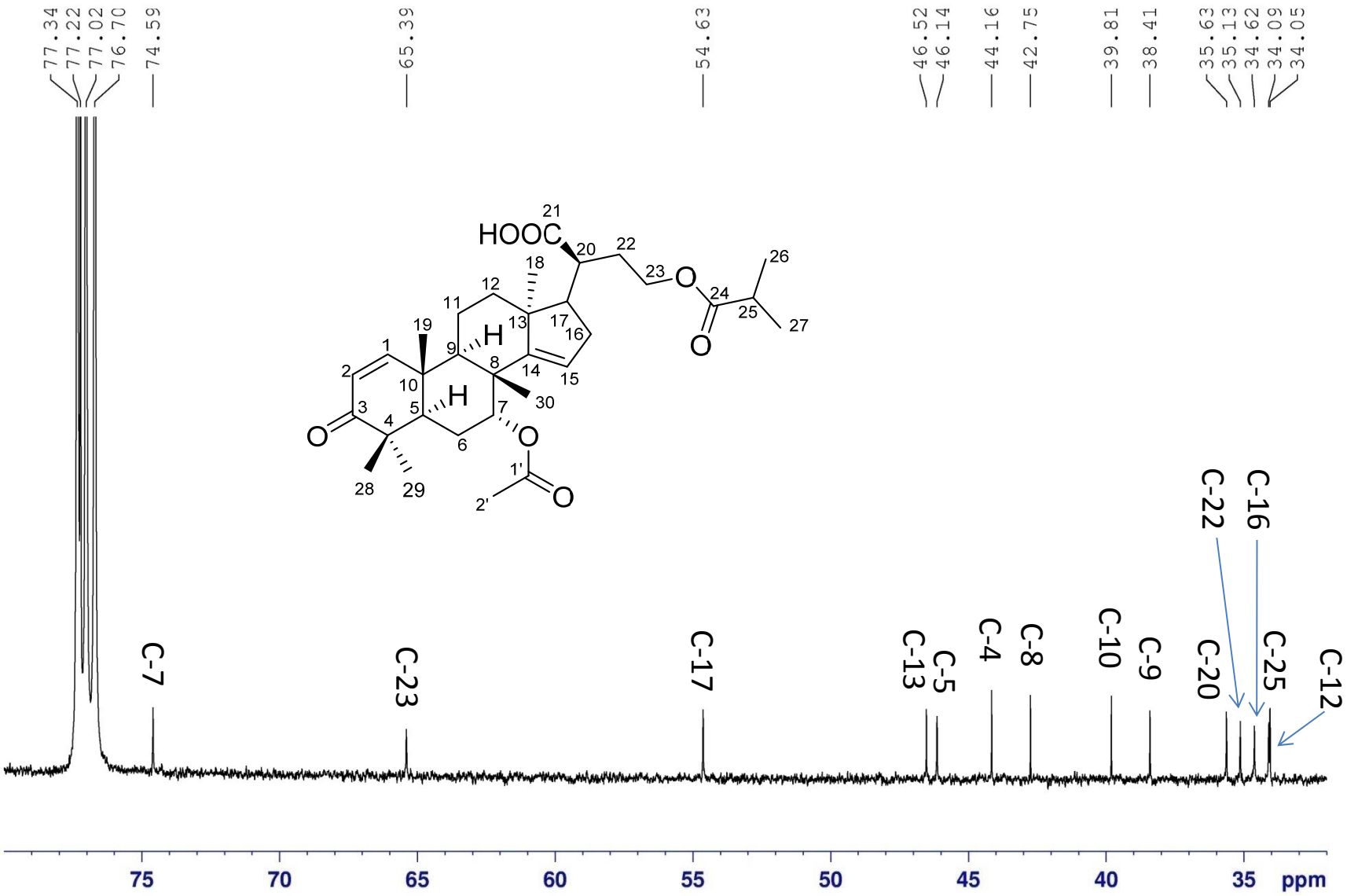
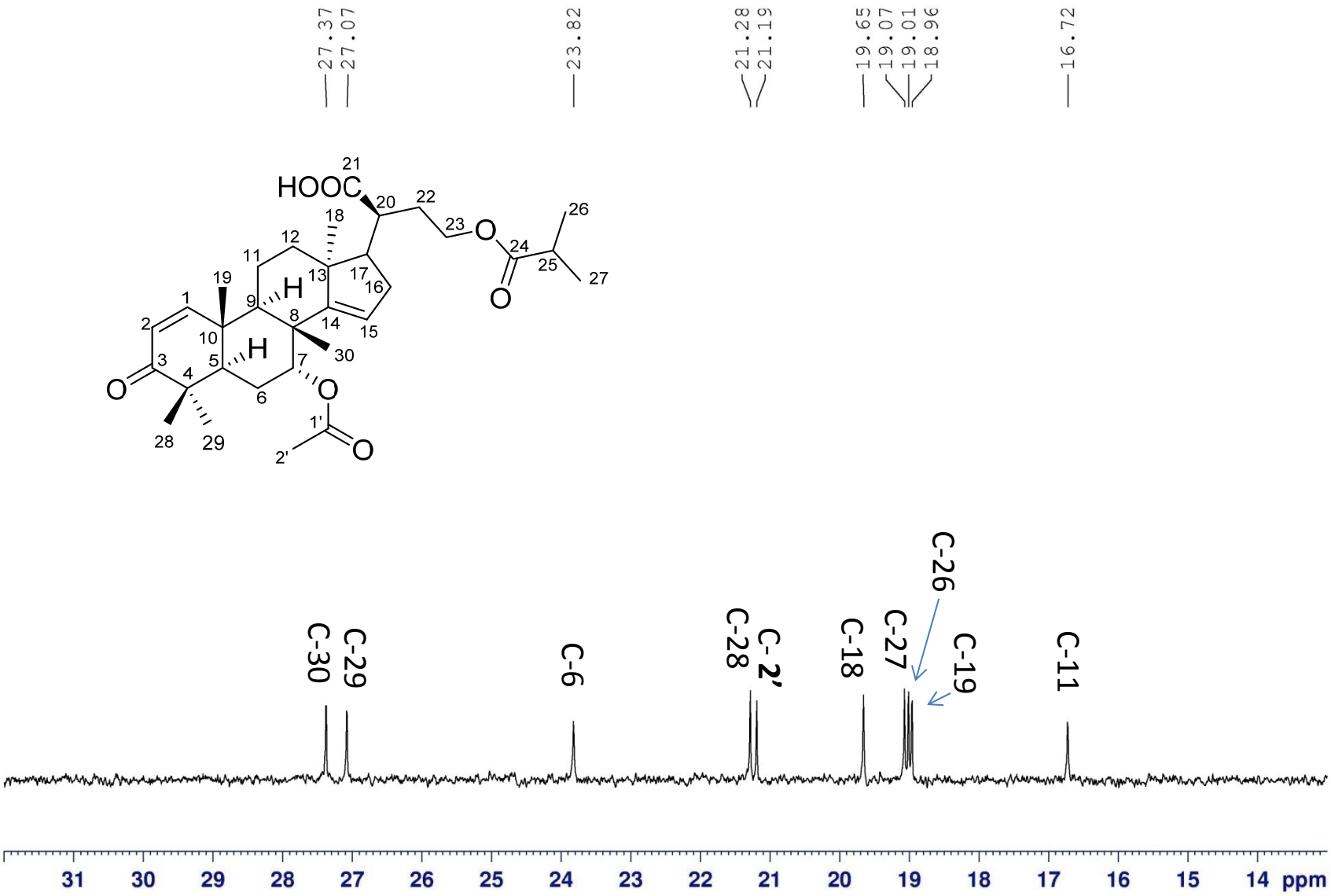

DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$



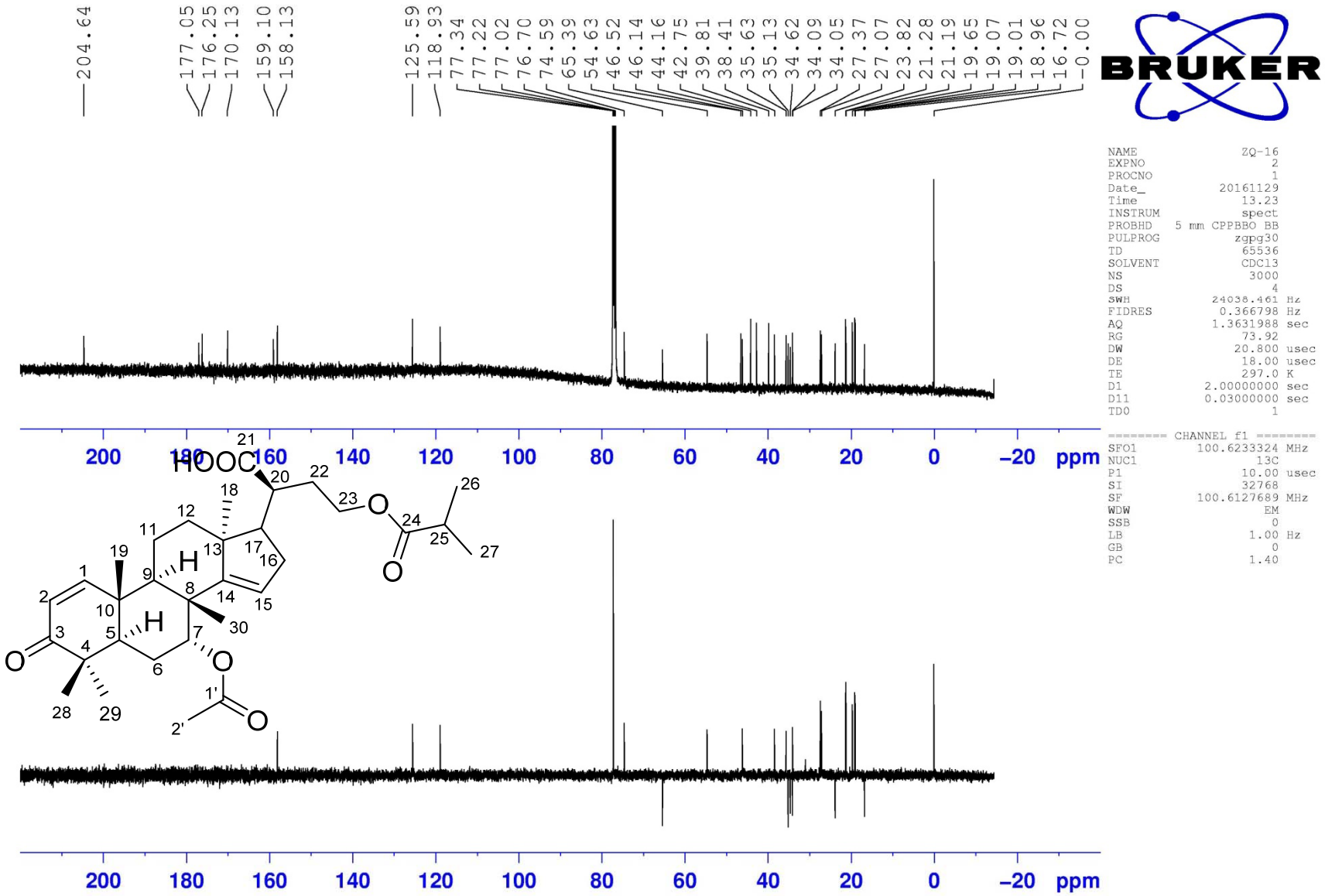
DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$



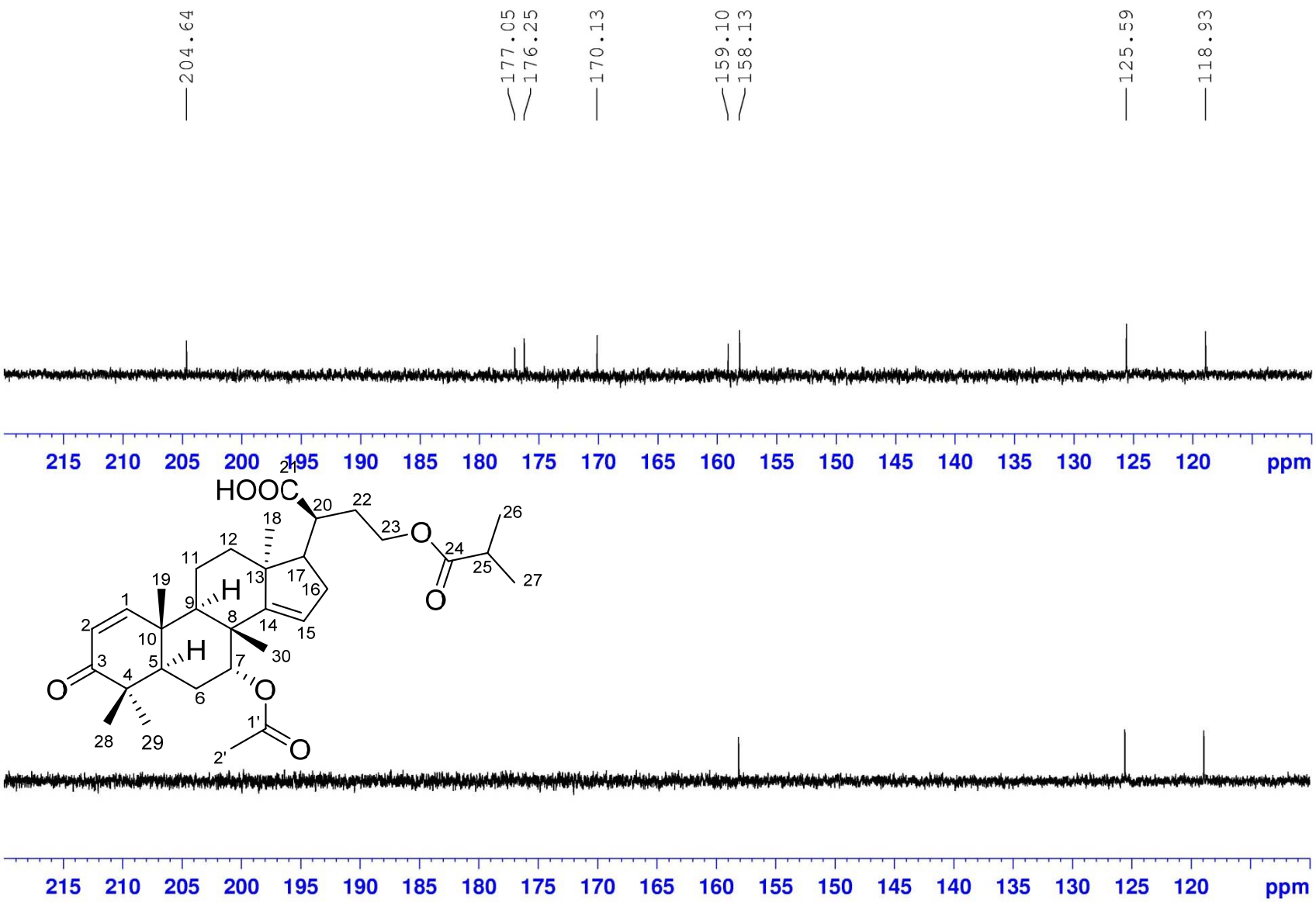
DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$



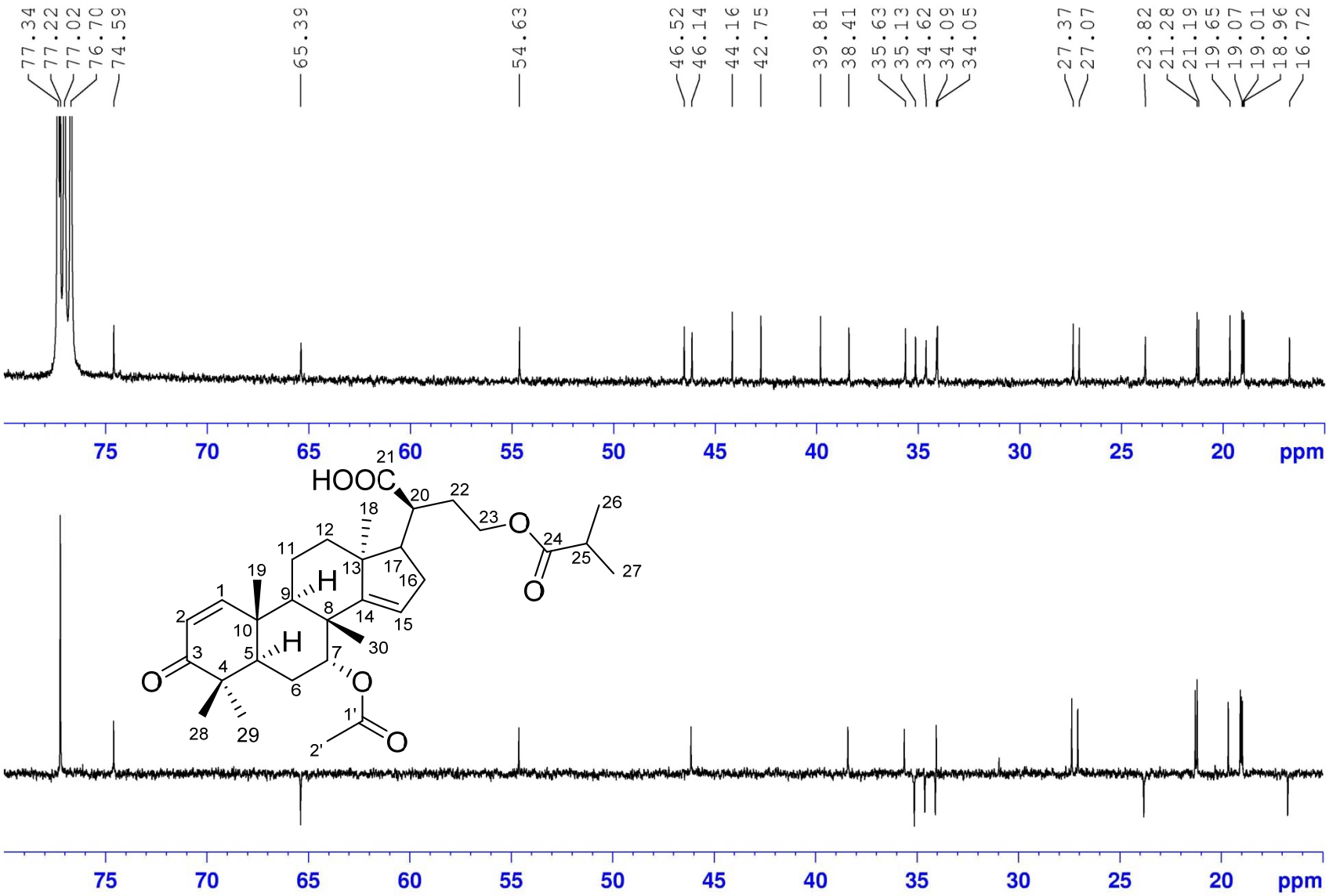
DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$



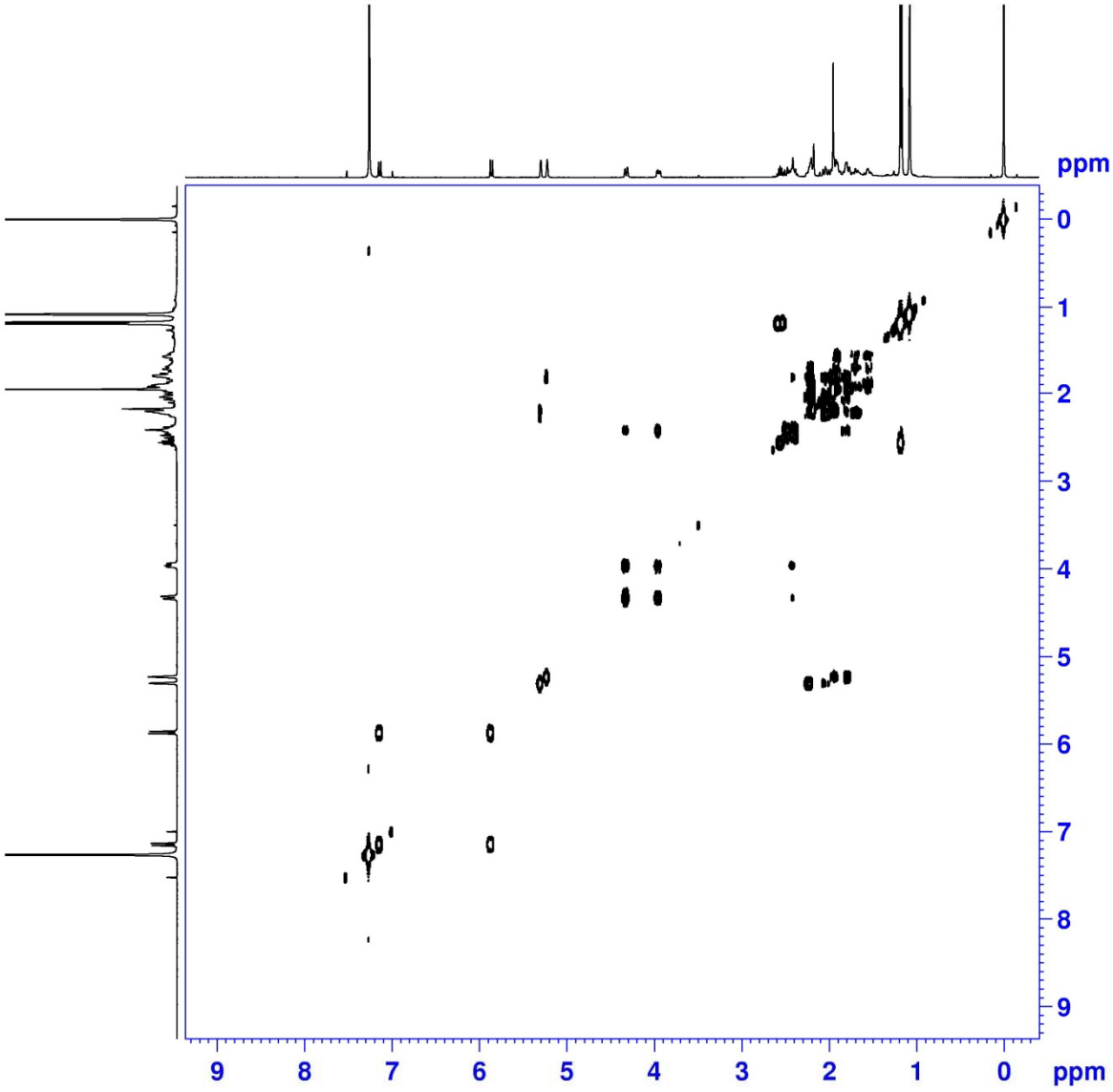
DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **10** in $CDCl_3$

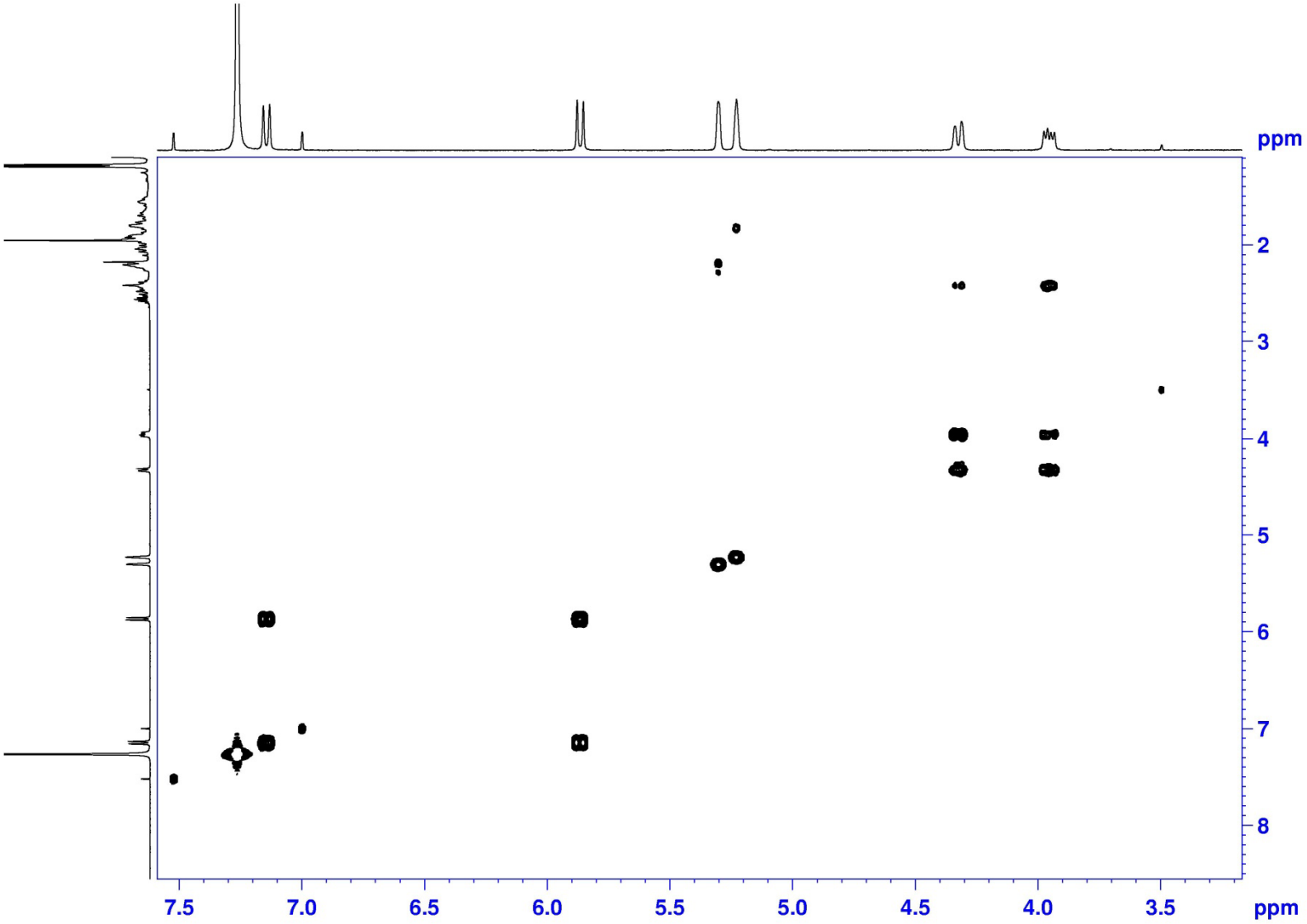


^{1}H-^{1}H COSY (400 MHz) spectrum of compound **10** in $CDCl_3$

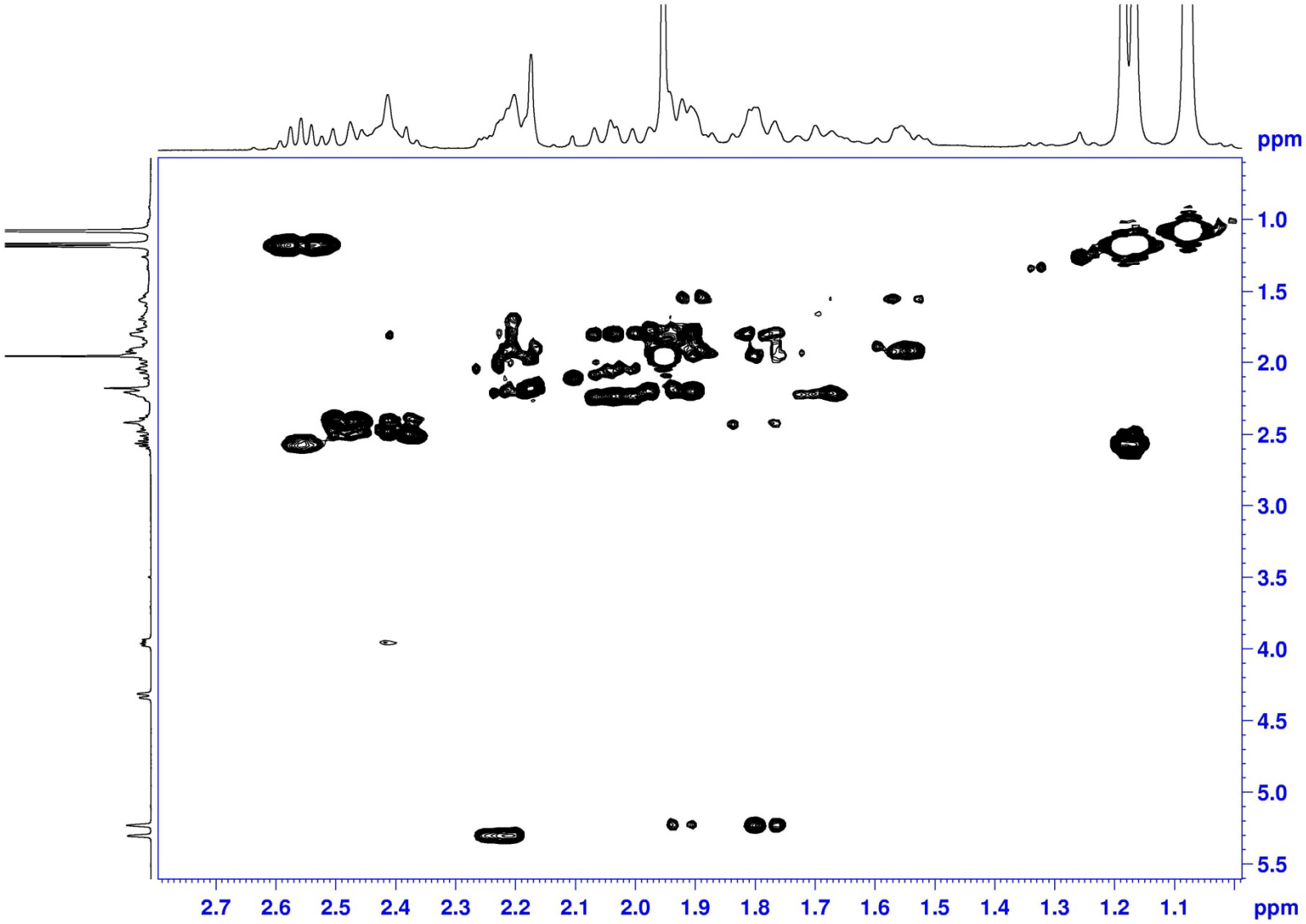



```
NAME                ZQ-16
EXPNO                   4
PROCNO                  1
Date_            20161203
Time                 8.34
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG       cosygpppqf
TD                   2048
SOLVENT             CDCl3
NS                      8
DS                      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            400.1300074 MHz
WDW                 QSINE
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
SI                   1024
MC2                    QF
SF            400.1300054 MHz
WDW                 QSINE
SSB                     0
LB                   0.00 Hz
```

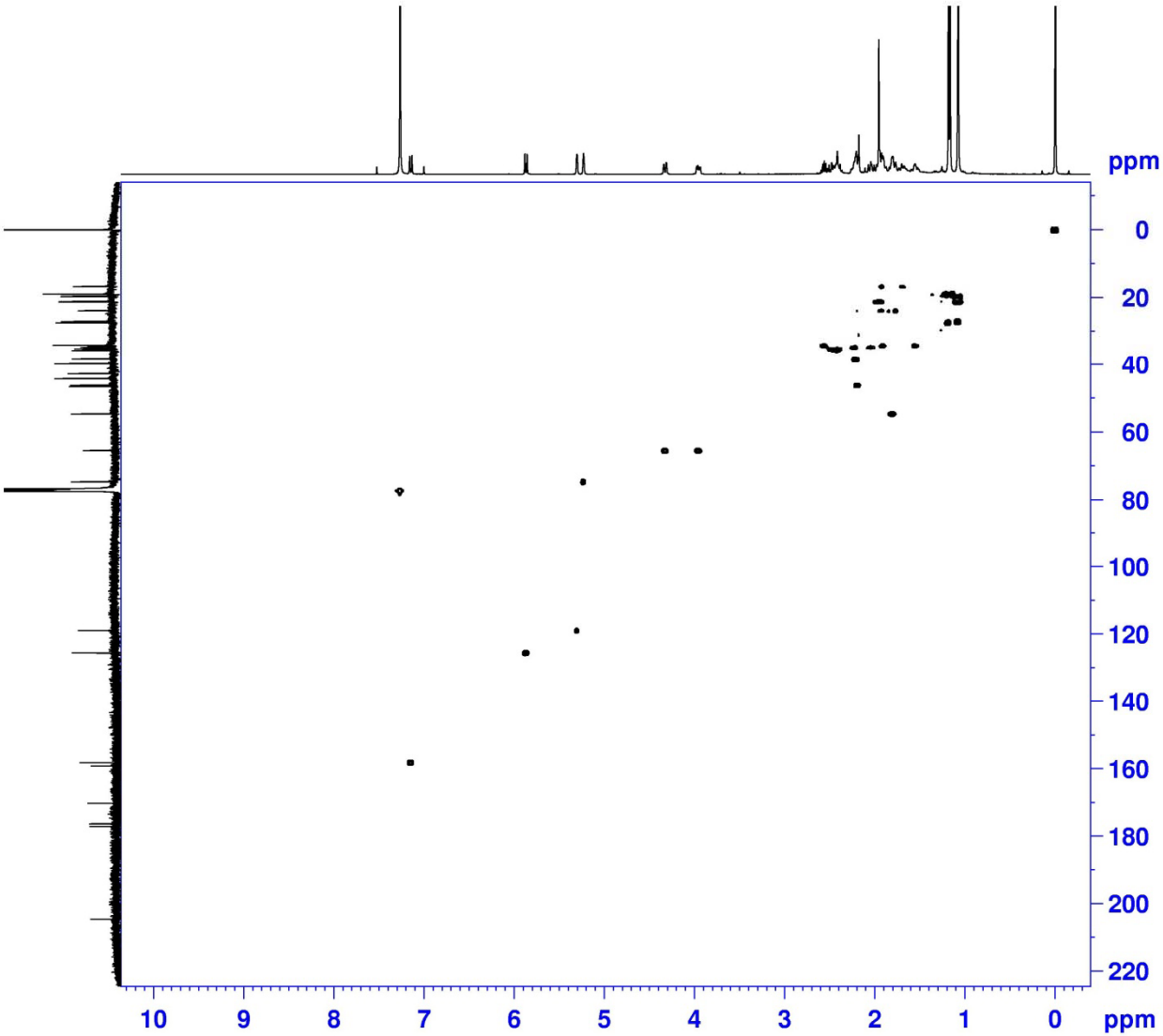
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **10** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **10** in $CDCl_3$

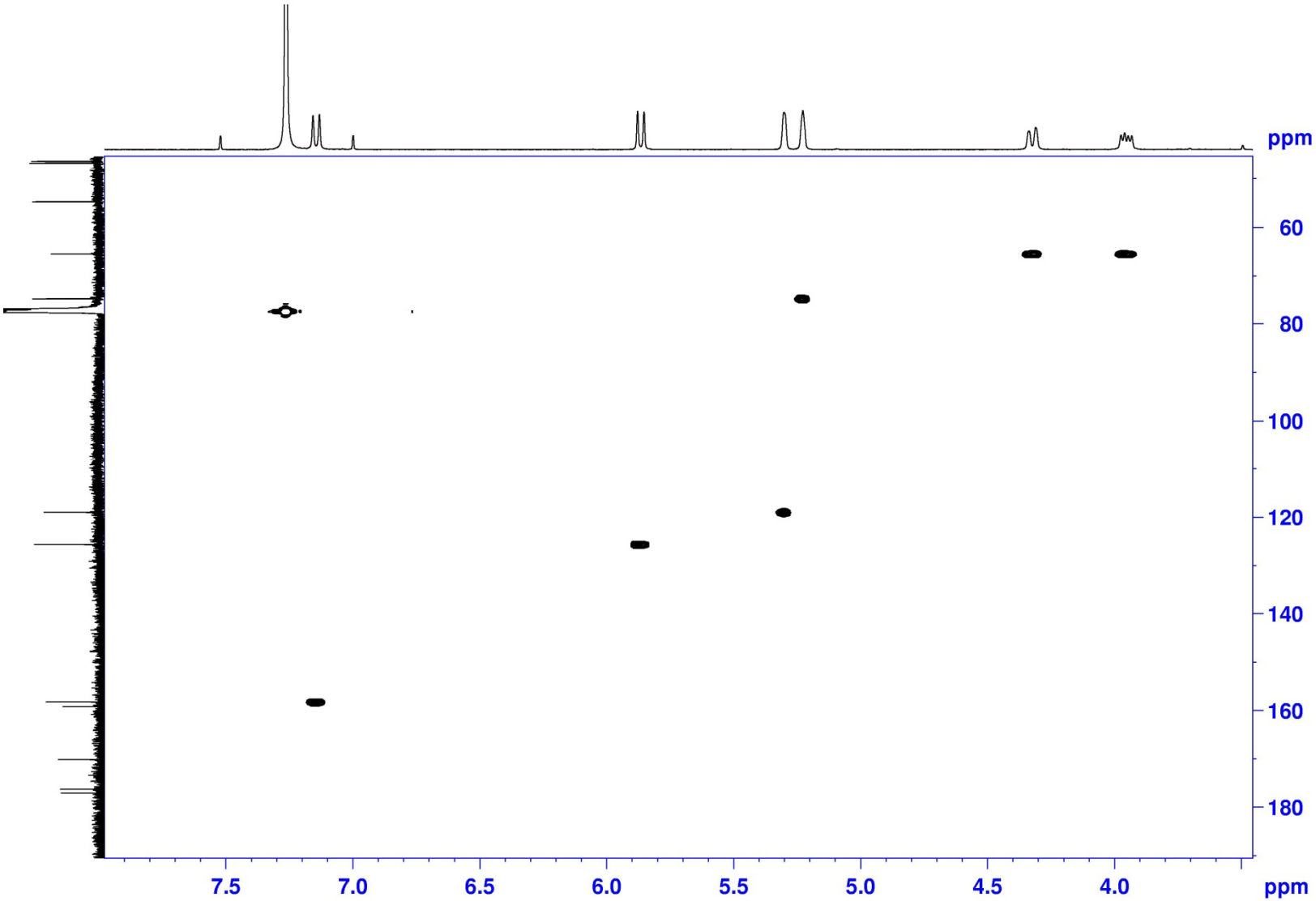


HSQC (400 MHz) spectrum of compound **10** in $CDCl_3$

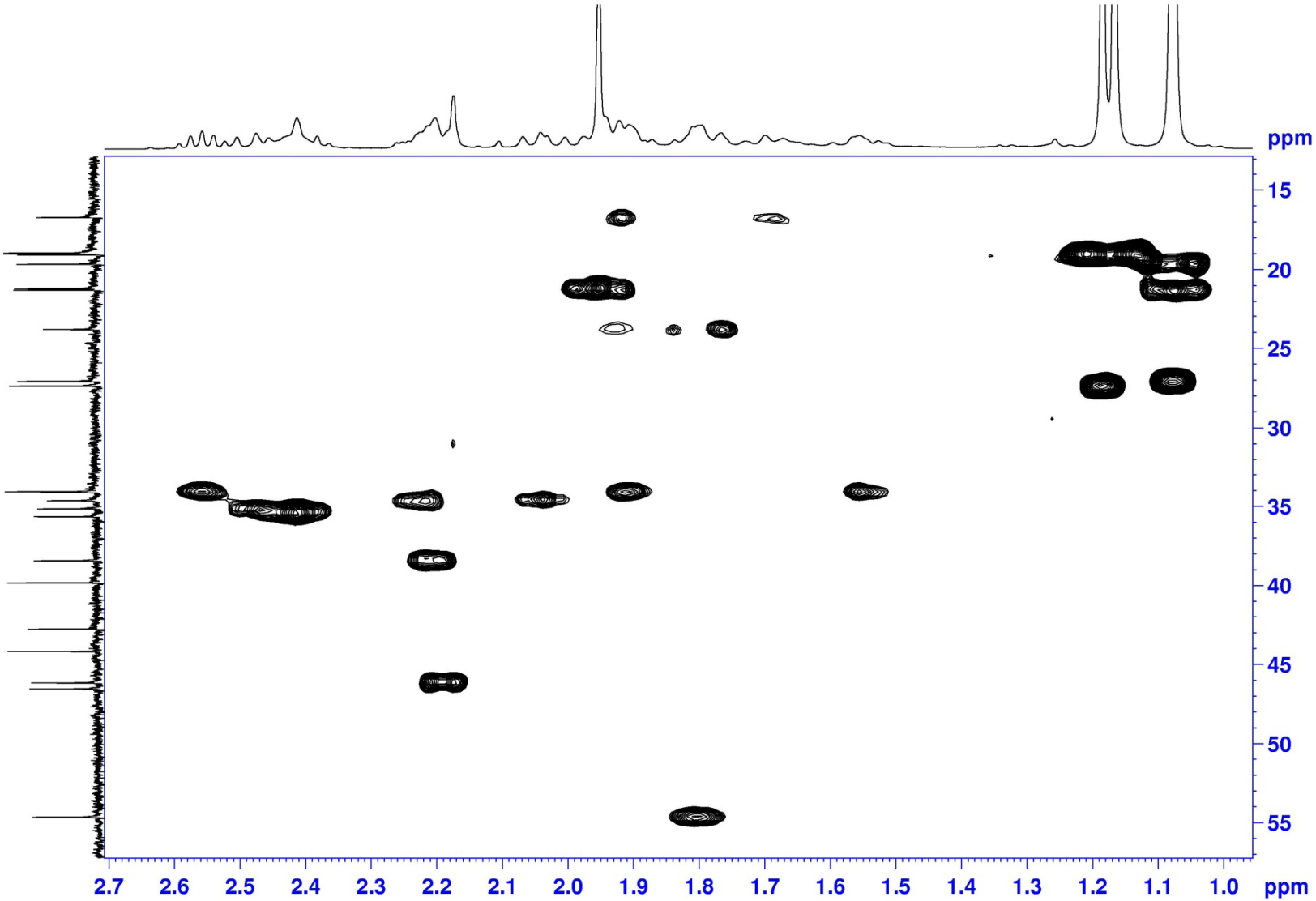



```
NAME                ZQ-16
EXPNO                   5
PROCNO                  1
Date_            20161203
Time                 9.13
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG     hsqcetgpsi2
TD                   1024
SOLVENT             CDCl3
NS                     16
DS                     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
ZGOPTNS

======== CHANNEL f1 ========
SFO1         400.1320007 MHz
NUC1                  1H
P1                 11.50 usec
P2                 23.00 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          400.1300066 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                   1024
MC2        echo-antiecho
SF          100.6127567 MHz
WDW                 QSINE
```

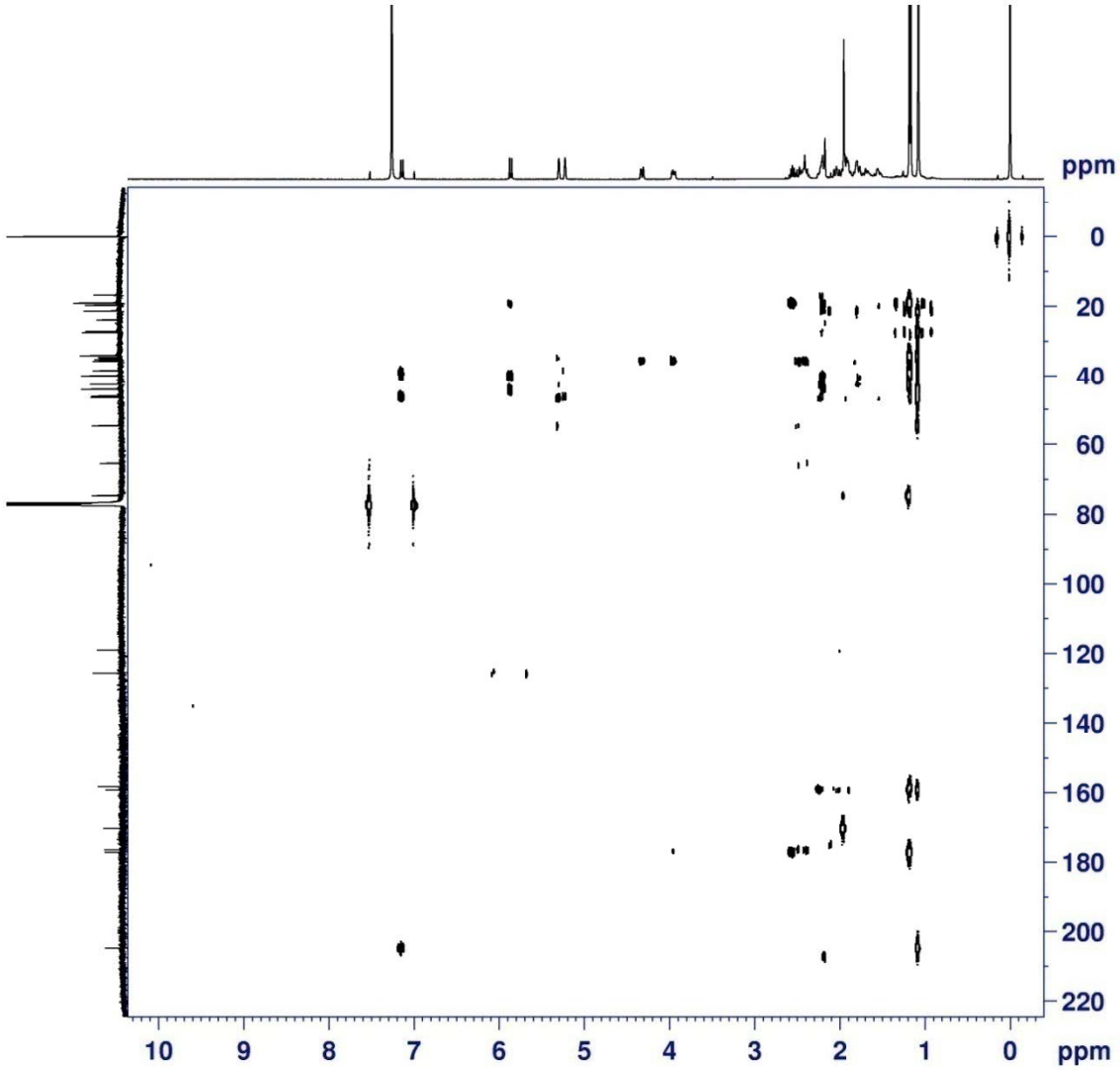
HSQC (400 MHz) spectrum of compound **10** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **10** in $CDCl_3$

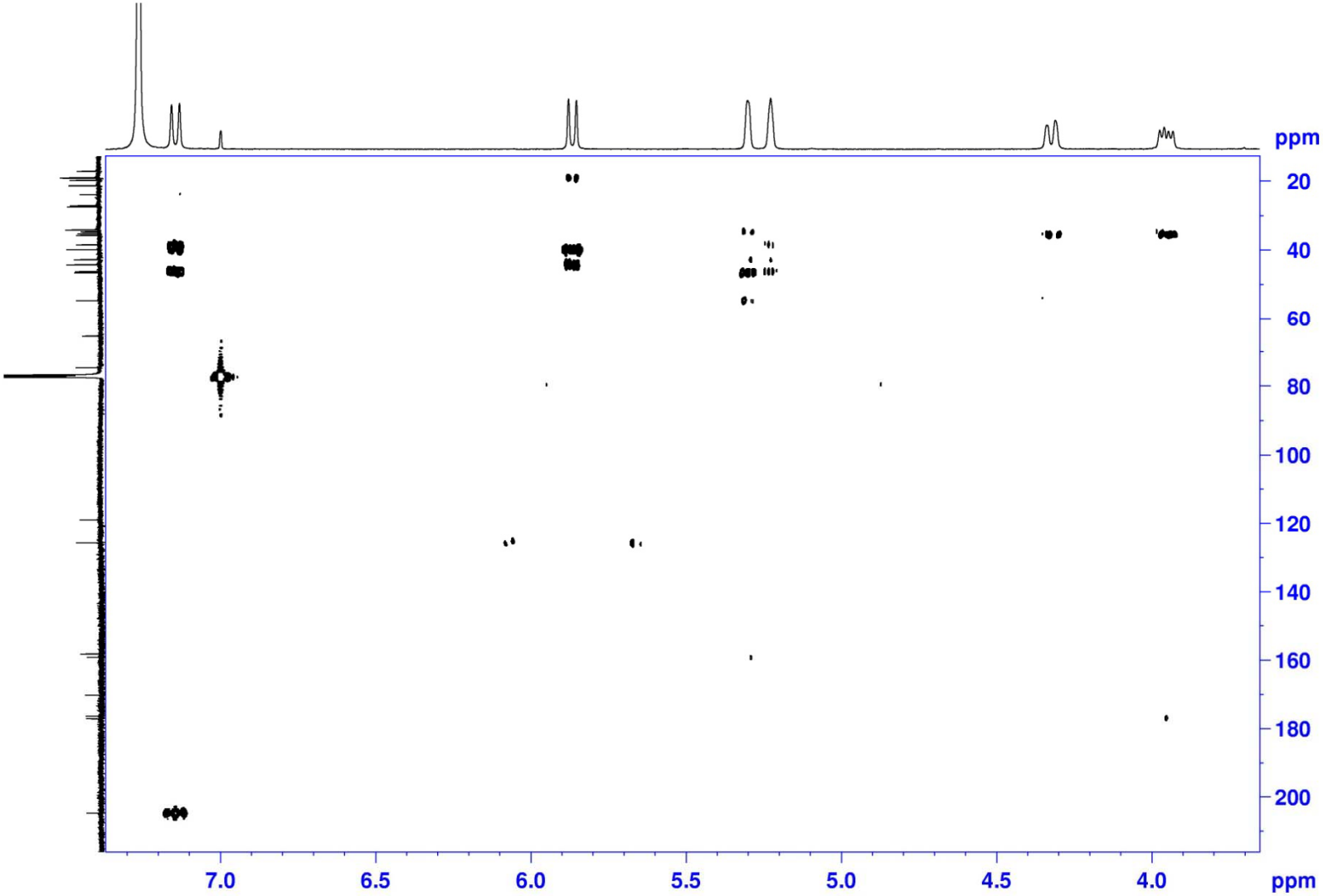


HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$

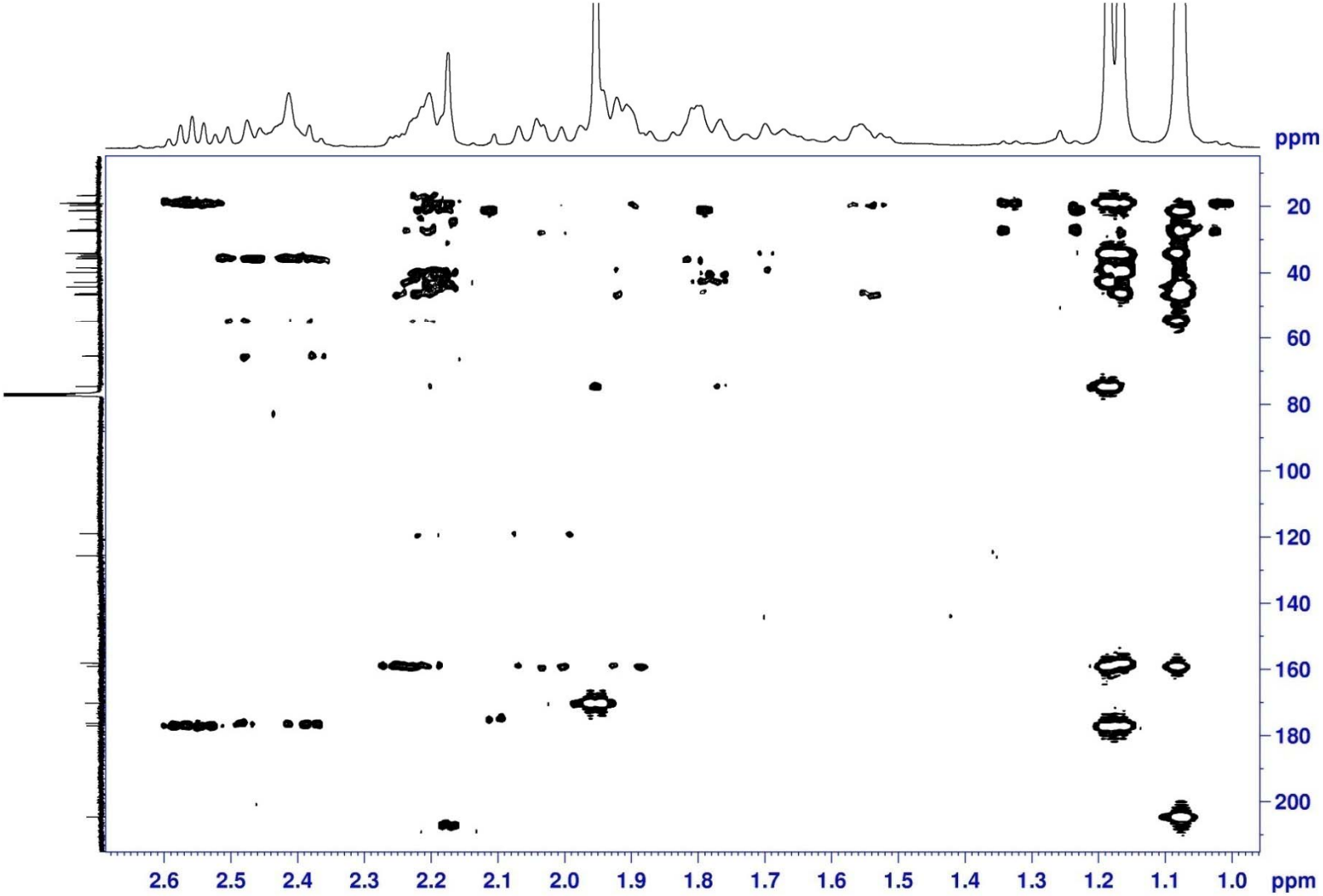



```
NAME                ZQ-16
EXPNO                   6
PROCNO                  1
Date_            20161203
Time                11.04
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG     hmbcgplpndqf
TD                   4096
SOLVENT             CDCl3
NS                     32
DS                     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 ========
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
SI                  2048
SF           400.1300076 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           100.6127600 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
```

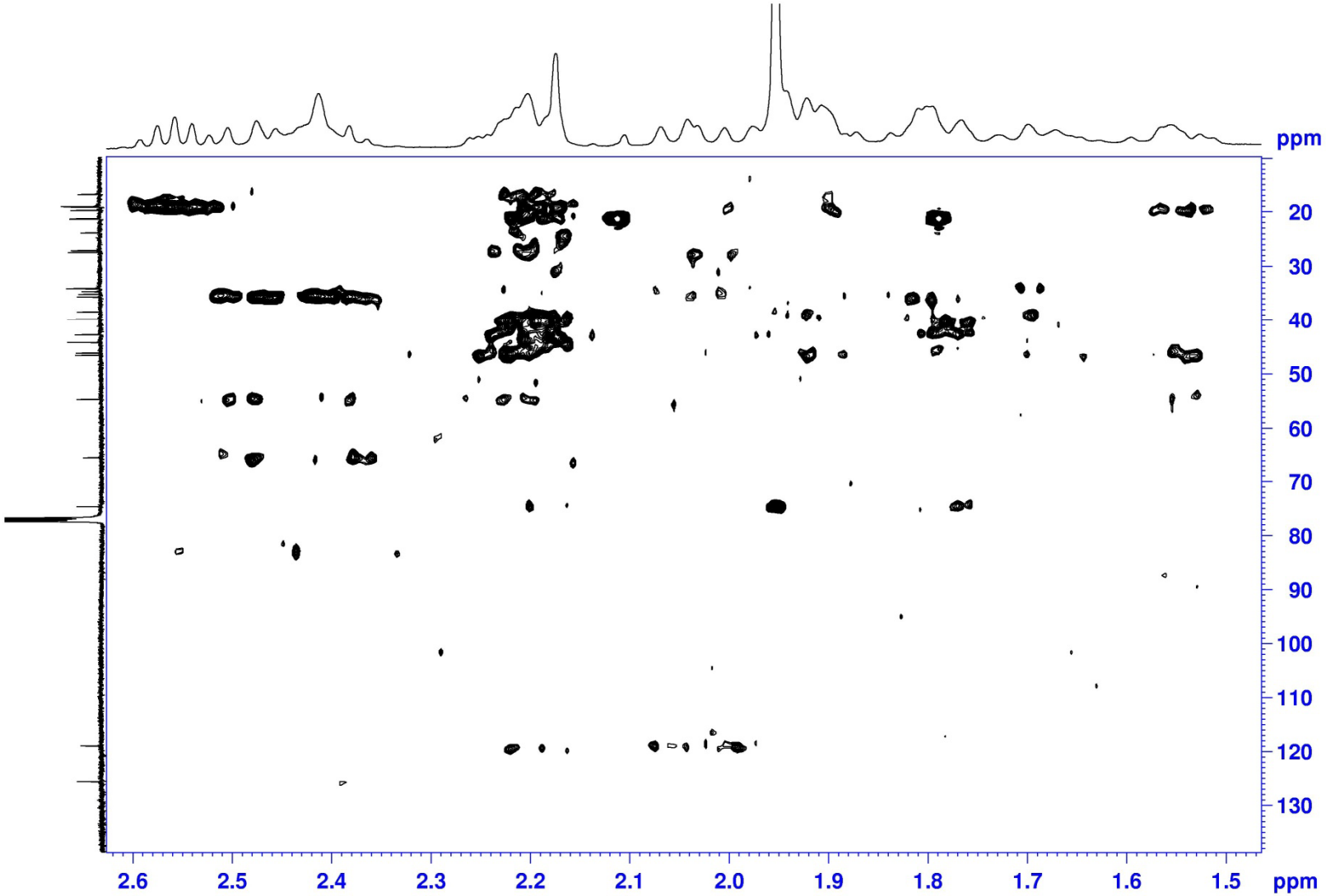
HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



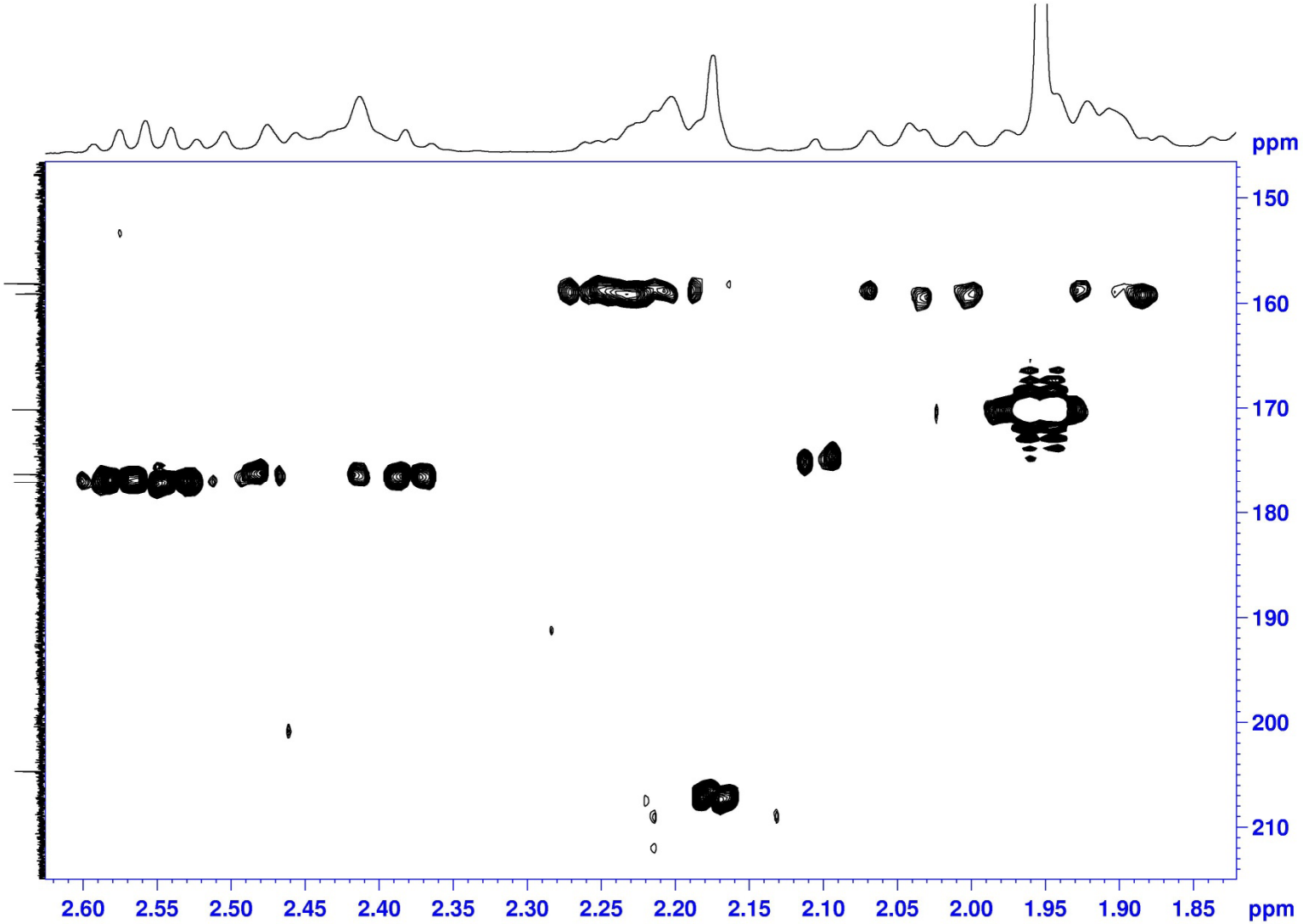
HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



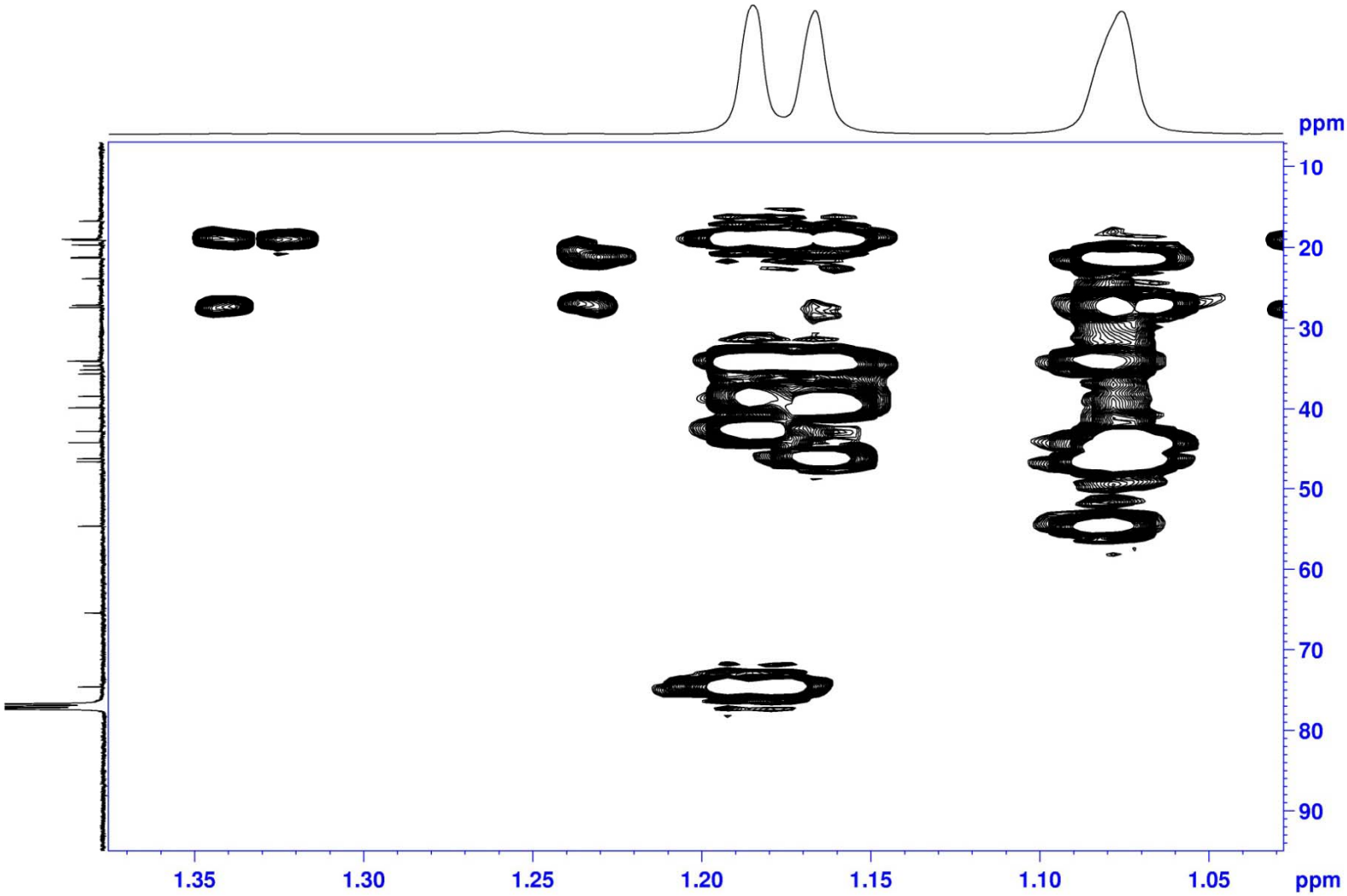
HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



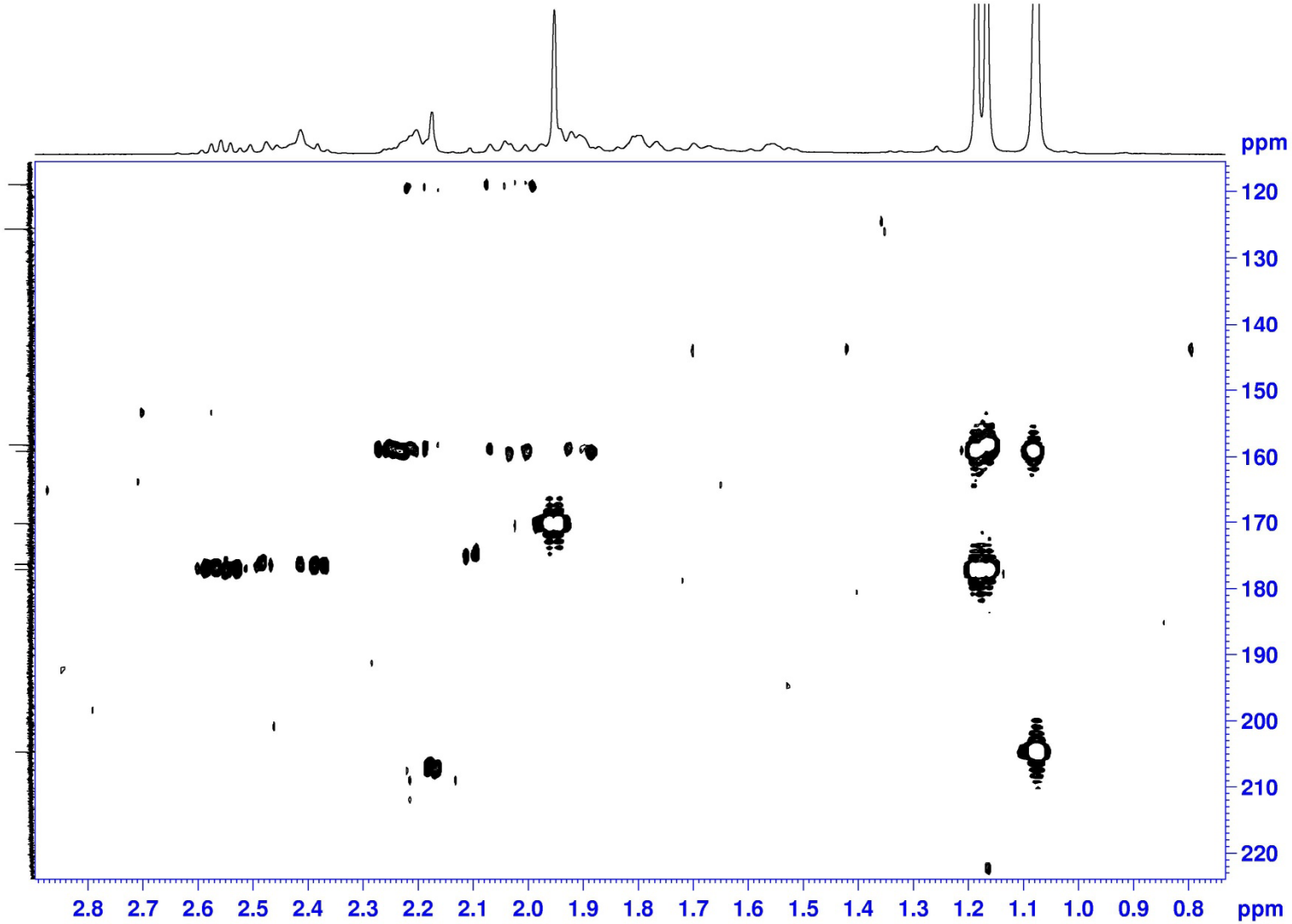
HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



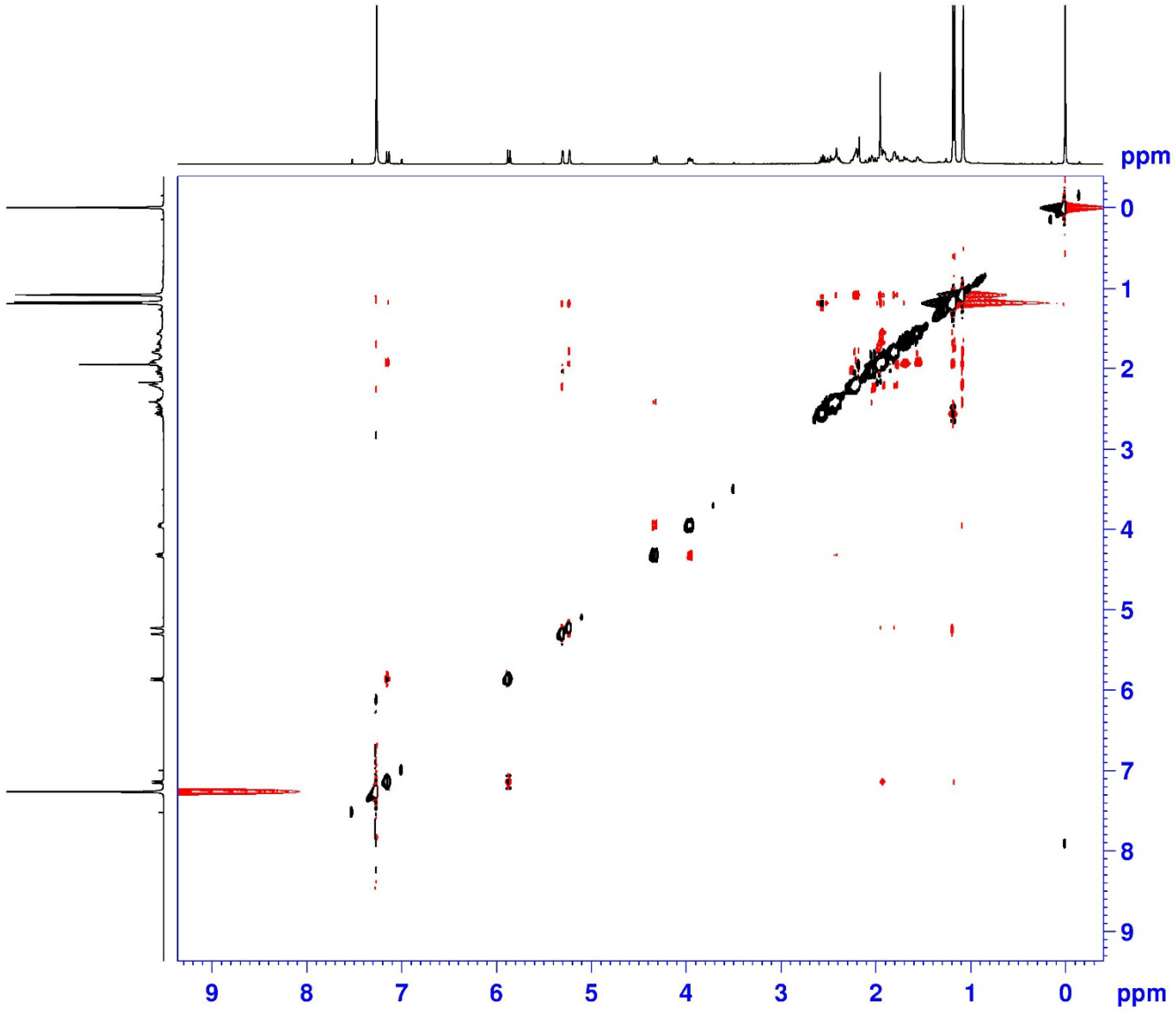
HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **10** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **10** in $CDCl_3$

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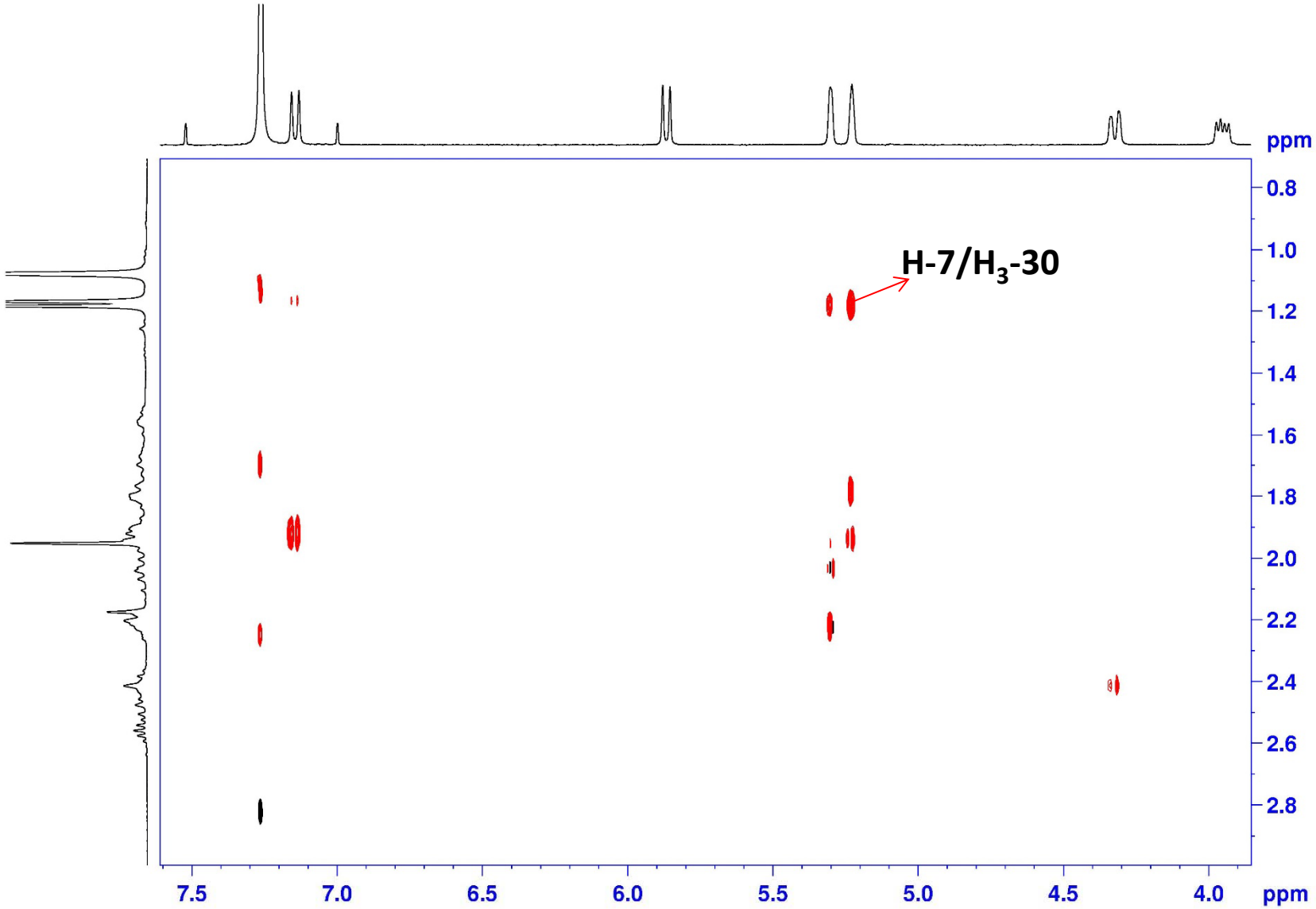
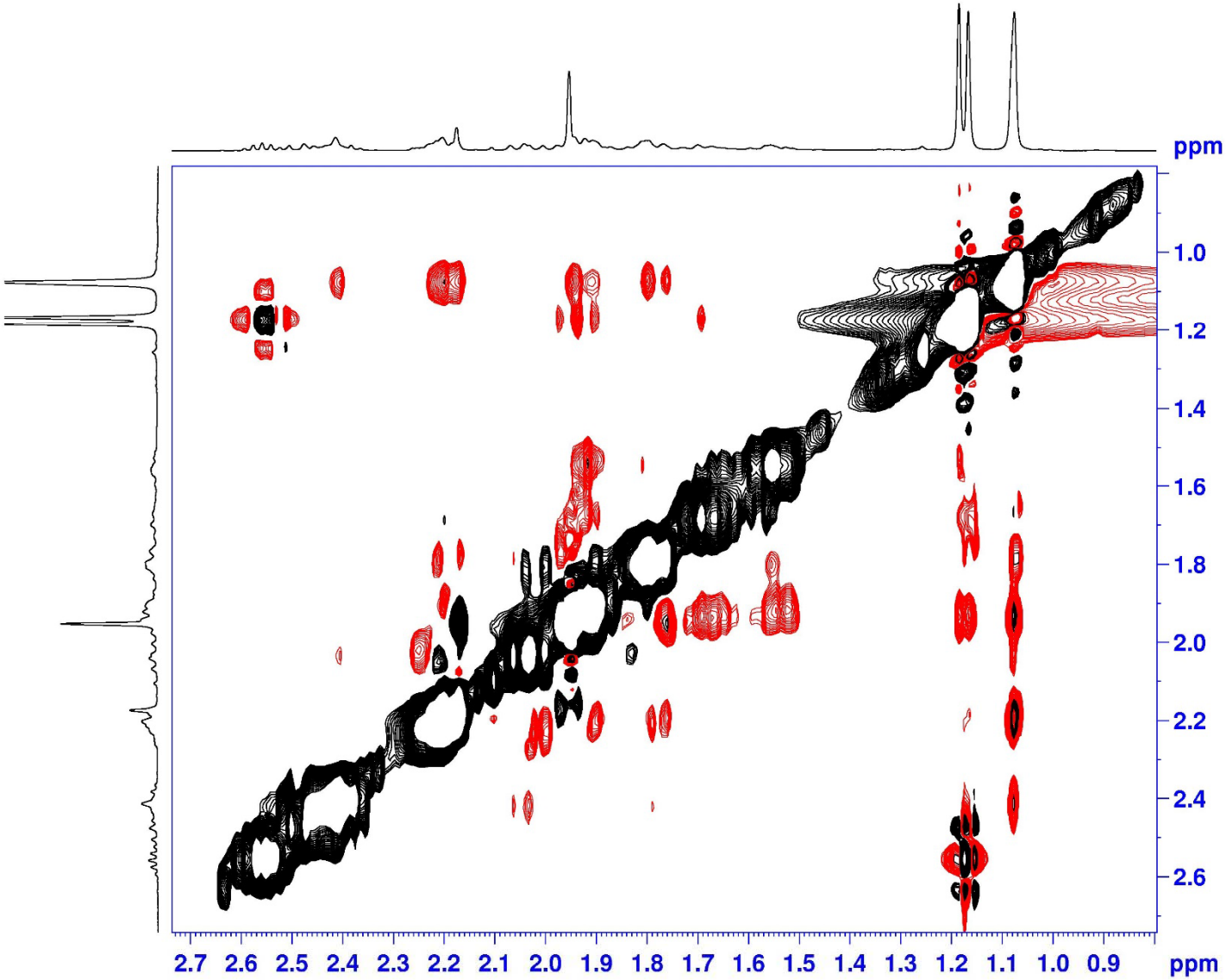
NAME                ZQ-16
EXPNO                   7
PROCNO                  1
Date_            20161203
Time                13.20
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                     16
DS                     32
SWH             4000.000 Hz
FIDRES          1.953125 Hz
AQ             0.2560500 se
RG                 208.5
DW                125.000 us
DE                 10.00 us
TE                 297.0 K
D0             0.00011036 se
D1             1.99385595 se
D8             0.30000001 se
D11            0.03000000 se
D12            0.00002000 se
D16            0.00020000 se
IN0            0.00025000 se

======== CHANNEL f1 ======
SFO1          400.1318006 MH
NUC1                   1H
P1                  11.50 us
P2                  23.00 us
P17               2500.00 us
ND0                     1
TD                    256
SFO1             400.1318 MH
FIDRES          15.625000 Hz
SW                  9.997 pp
FnMODE        States-TPPI
SI                   1024
SF            400.1300061 MH
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00
SI                   1024
MC2           States-TPPI
SF            400.1300088 MH
WDW                 QSINE
SSB                     2
```

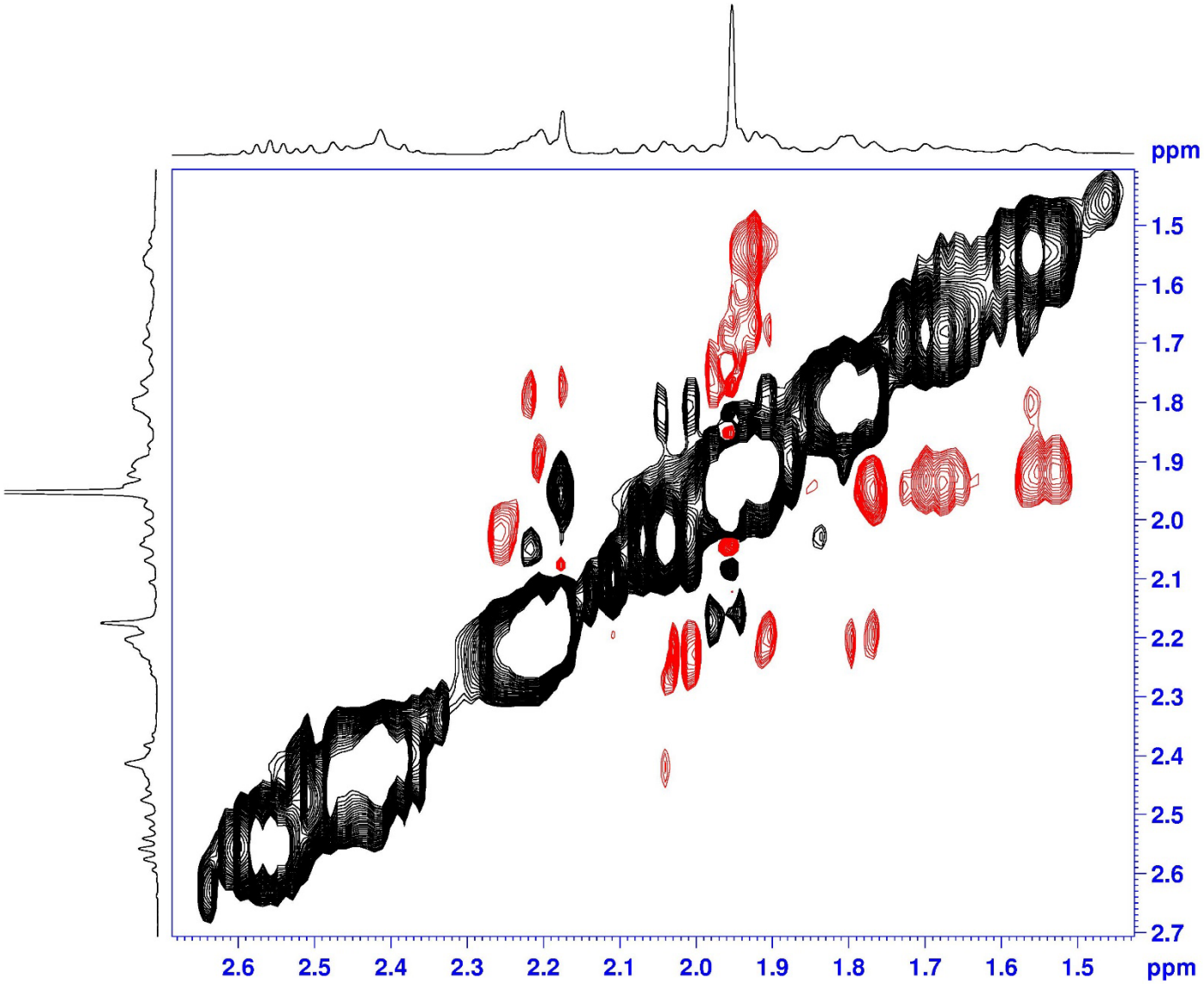
NOESY (400 MHz) spectrum of compound **10** in $CDCl_3$



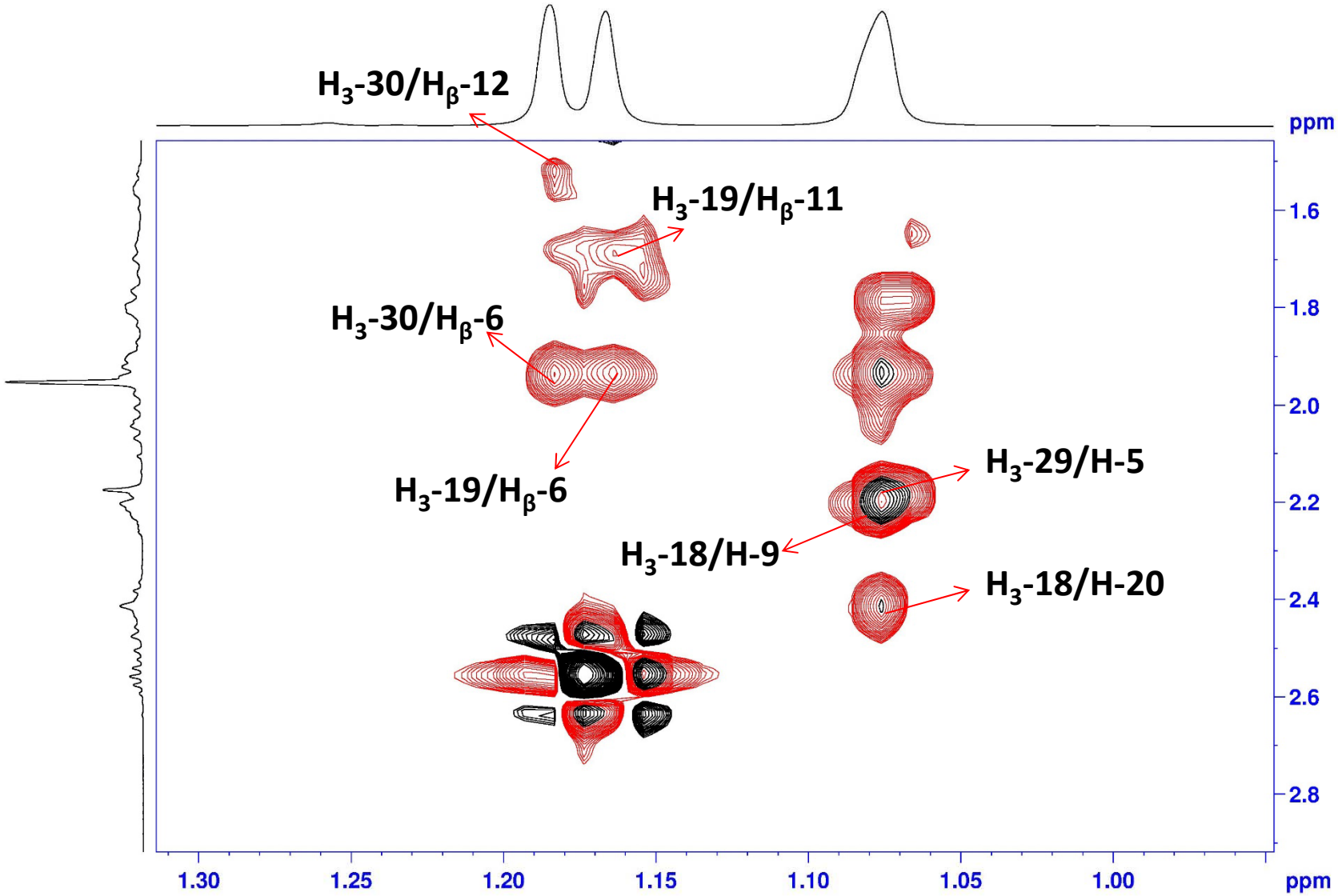
NOESY (400 MHz) spectrum of compound **10** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **10** in $CDCl_3$



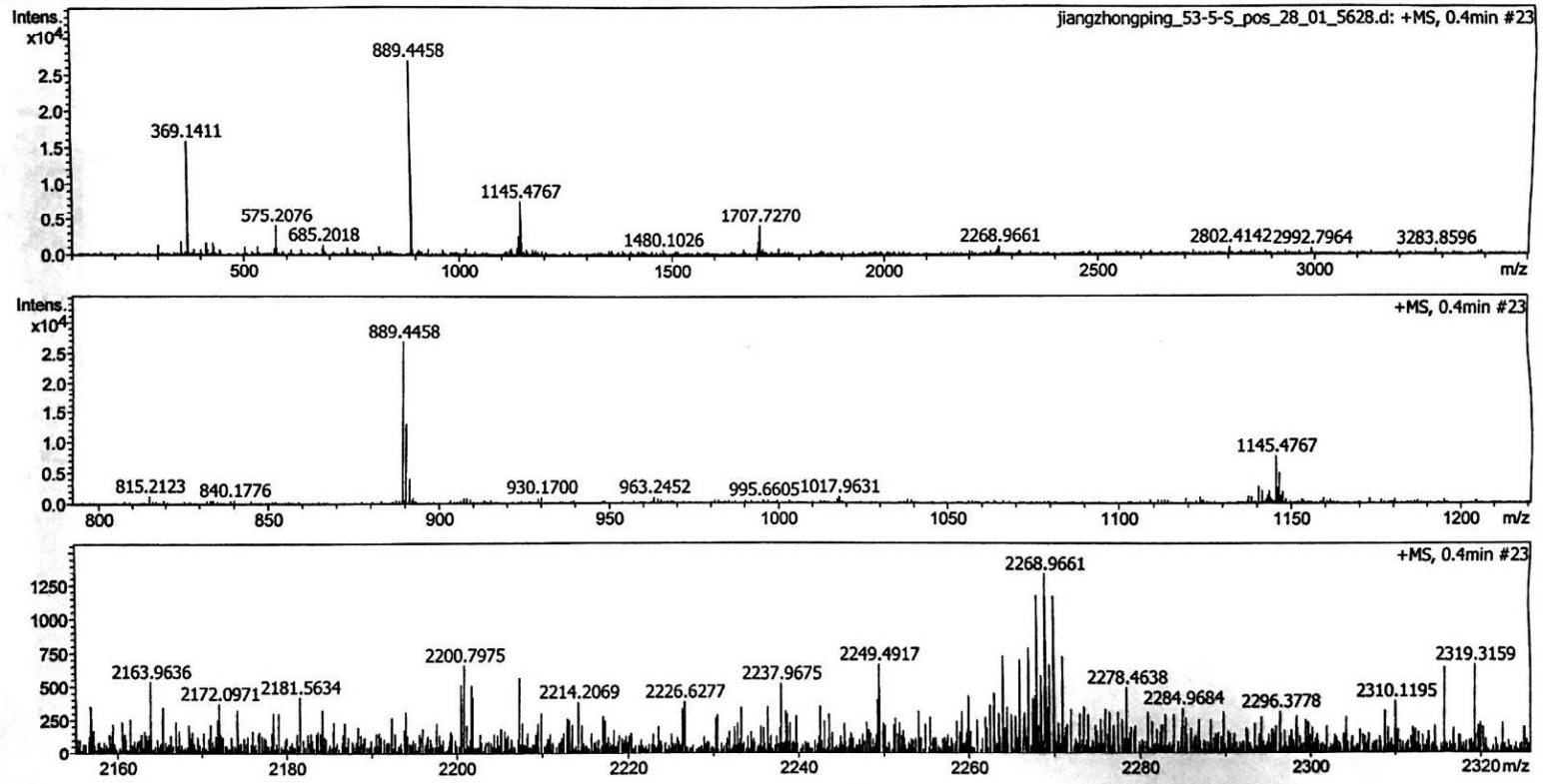
NOESY (400 MHz) spectrum of compound **10** in $CDCl_3$



HR-ESIMS for compound **4s**

Generic Display Report

Analysis Info		Acquisition Date	10/18/2018 3:03:40 PM
Analysis Name	D:\Data\MS\data\201810\jiangzhongping_53-5-S_pos_28_01_5628.d		
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_53-5-S_pos	Instrument	maXis
Comment			



Bruker Compass DataAnalysis 4.1 printed: 10/18/2018 3:09:29 PM by: SCSIO Page 1 of 1

HR-ESIMS for compound **4s**

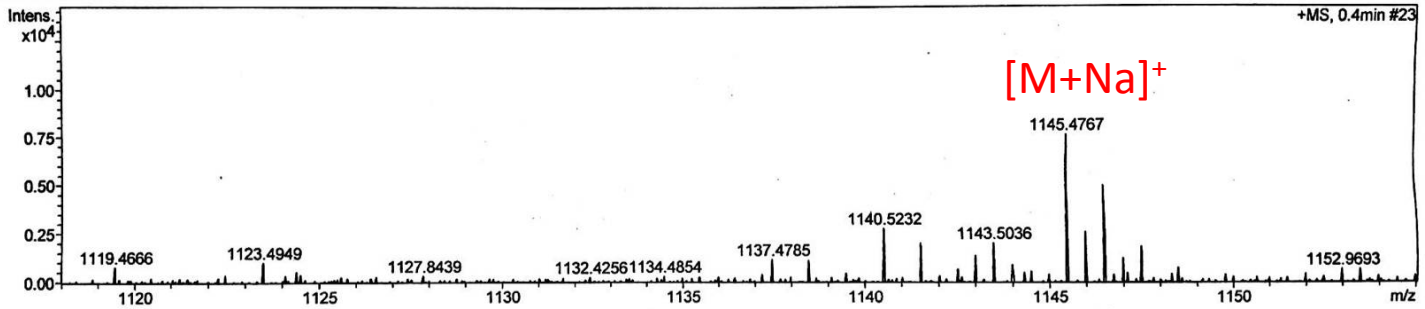
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\MS\data\201810\jiangzhongping_53-5-S_pos_28_01_5628.d	Acquisition Date	10/18/2018 3:03:40 PM
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_53-5-S_pos	Instrument	maXis 255552.00029
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	0 V	Set Dry Gas	4.0 l/min
Scan End	3500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

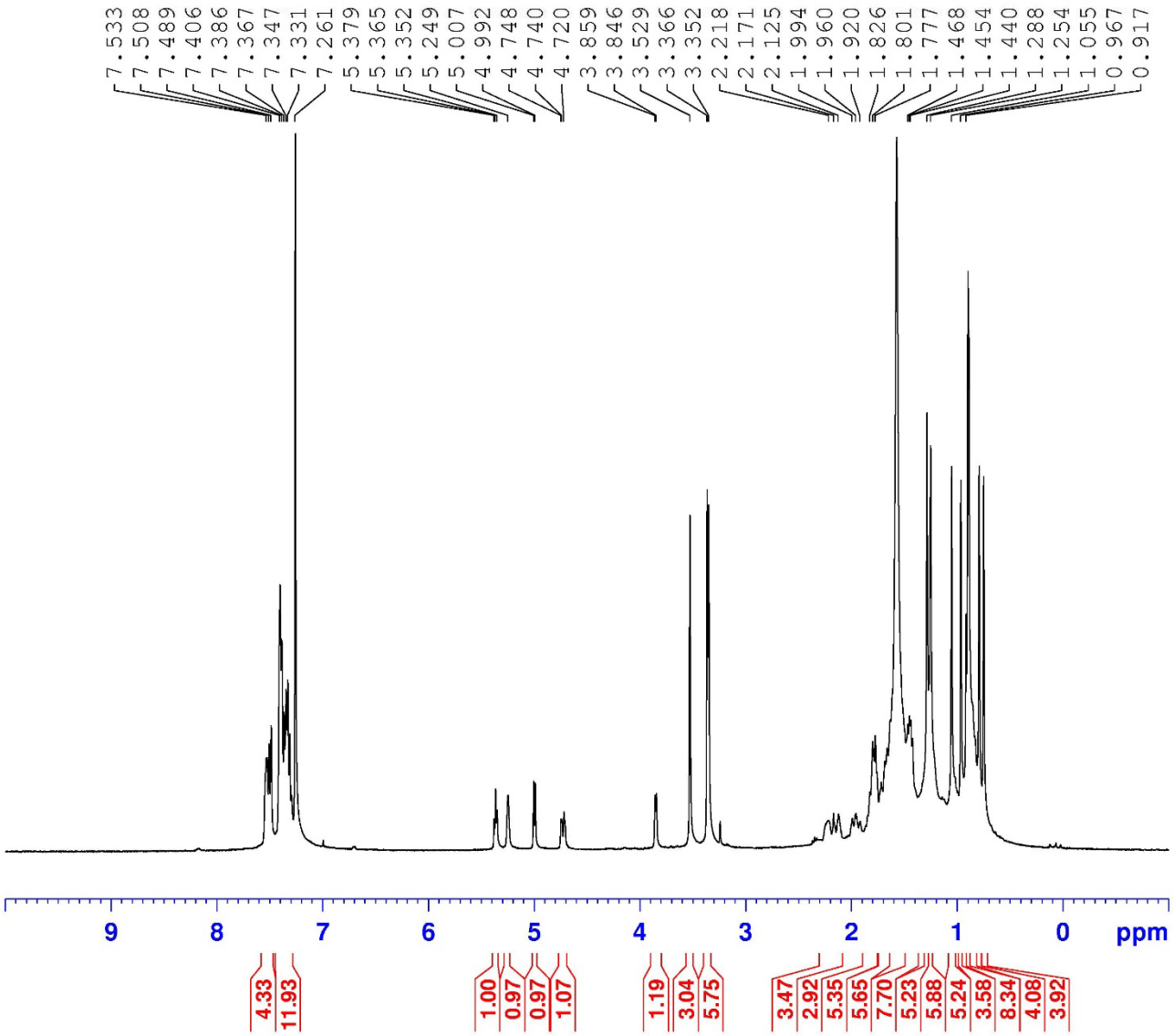


Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
1123.4949	1	C60H72F9O10	100.00	1123.4976	2.4	2.7	121.2	20.5	even	ok
1145.4767	1	C60H71F9NaO10	100.00	1145.4796	-2.5	-2.8	18.1	20.5	even	ok

jiangzhongping_53-5-S_pos_28_01_5628.d

Bruker Compass DataAnalysis 4.1 printed: 10/18/2018 3:08:57 PM by: SCSIO Page 1 of 1

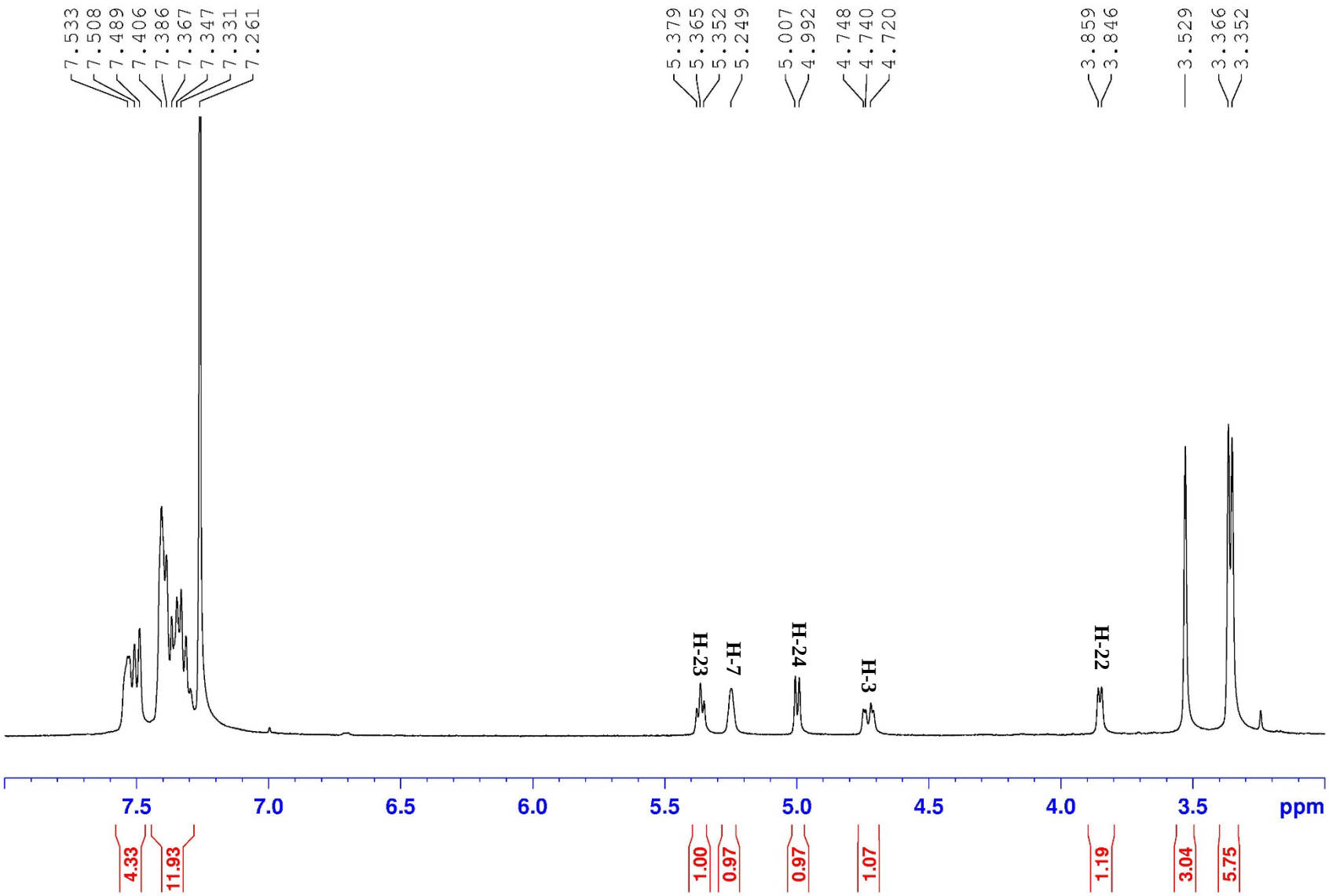
^{1}H NMR (400 MHz) spectrum of compound **4s** in $CDCl_3$



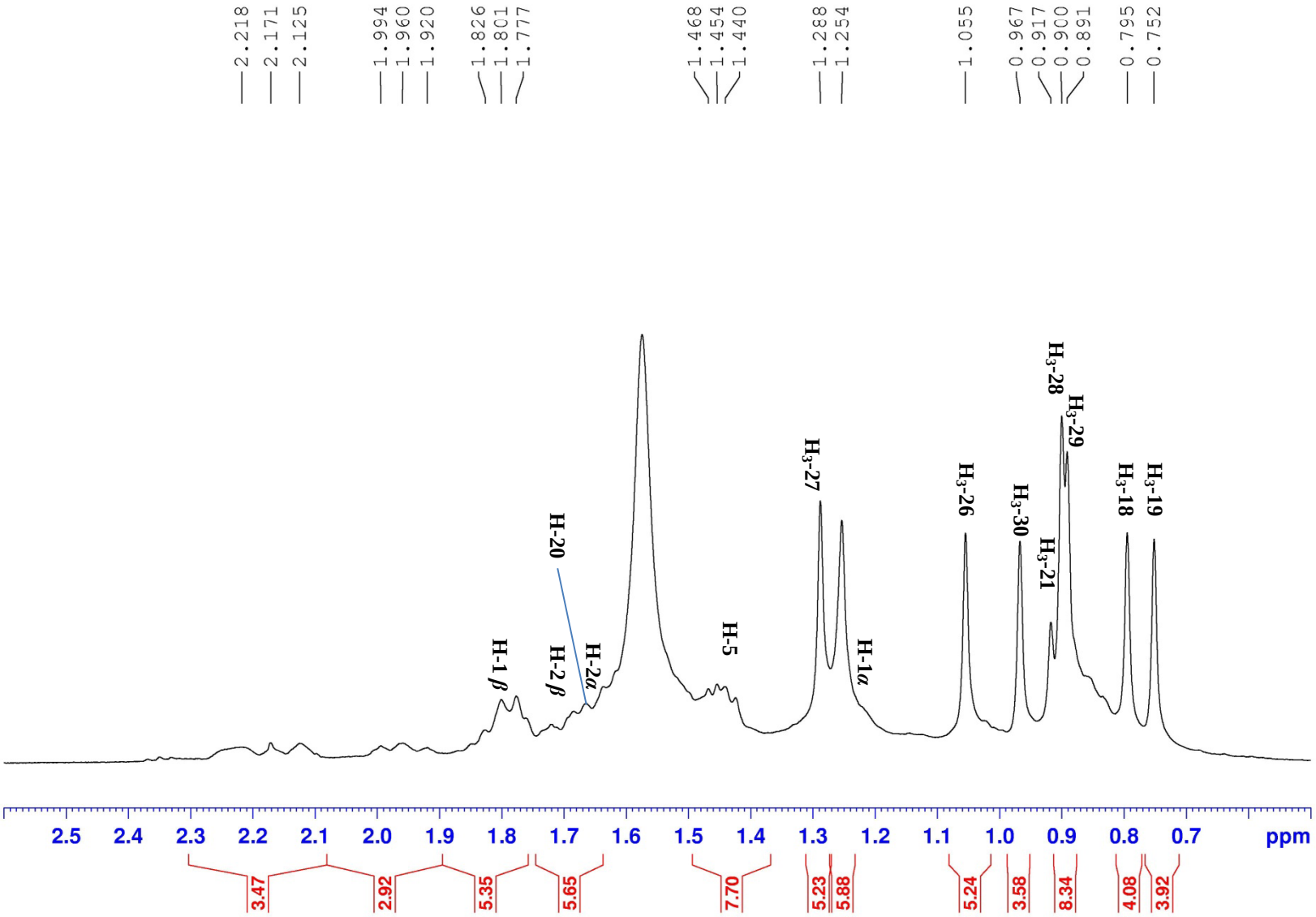

```
NAME      XG-53-5-S-MOSHER
EXPNO                   20
PROCNO                   1
Date_             20180804
Time                 12.29
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                   208.5
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                   11.50 usec
SI                   65536
SF             400.1300098 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

^{1}H NMR (400 MHz) spectrum of compound **4s** in $CDCl_3$



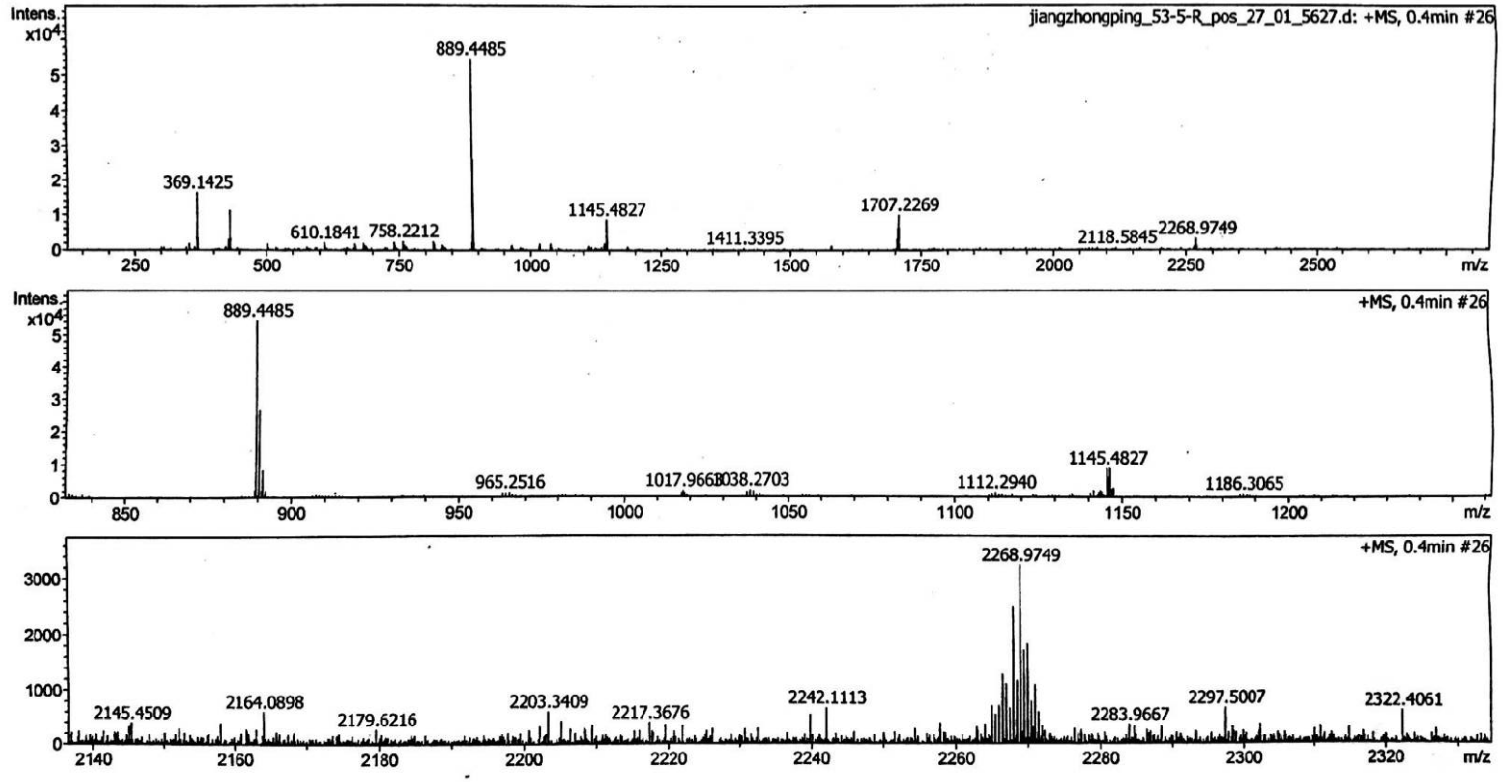
^{1}H NMR (400 MHz) spectrum of compound **4s** in $CDCl_3$



HR-ESIMS for compound **4r**

Generic Display Report

Analysis Info			
Analysis Name	D:\Data\MS\data\201810\jiangzhongping_53-5-R_pos_27_01_5627.d	Acquisition Date	10/18/2018 3:00:09 PM
Method	LC_DirectInfusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_53-5-R_pos	Instrument	maXis
Comment			



Bruker Compass DataAnalysis 4.1 printed: 10/18/2018 3:07:18 PM by: SCSIO Page 1 of 1

HR-ESIMS for compound **4r**

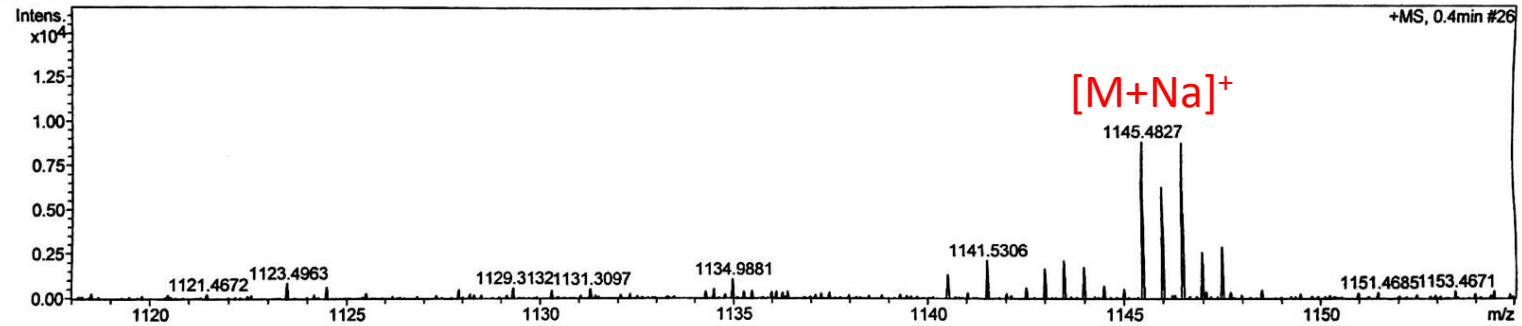
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\MS\data\201810\jiangzhongping_53-5-R_pos_27_01_5627.d	Acquisition Date	10/18/2018 3:00:09 PM
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	jiangzhongping_53-5-R_pos	Instrument	maXis 255552.00029
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	0 V	Set Dry Gas	4.0 l/min
Scan End	3500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

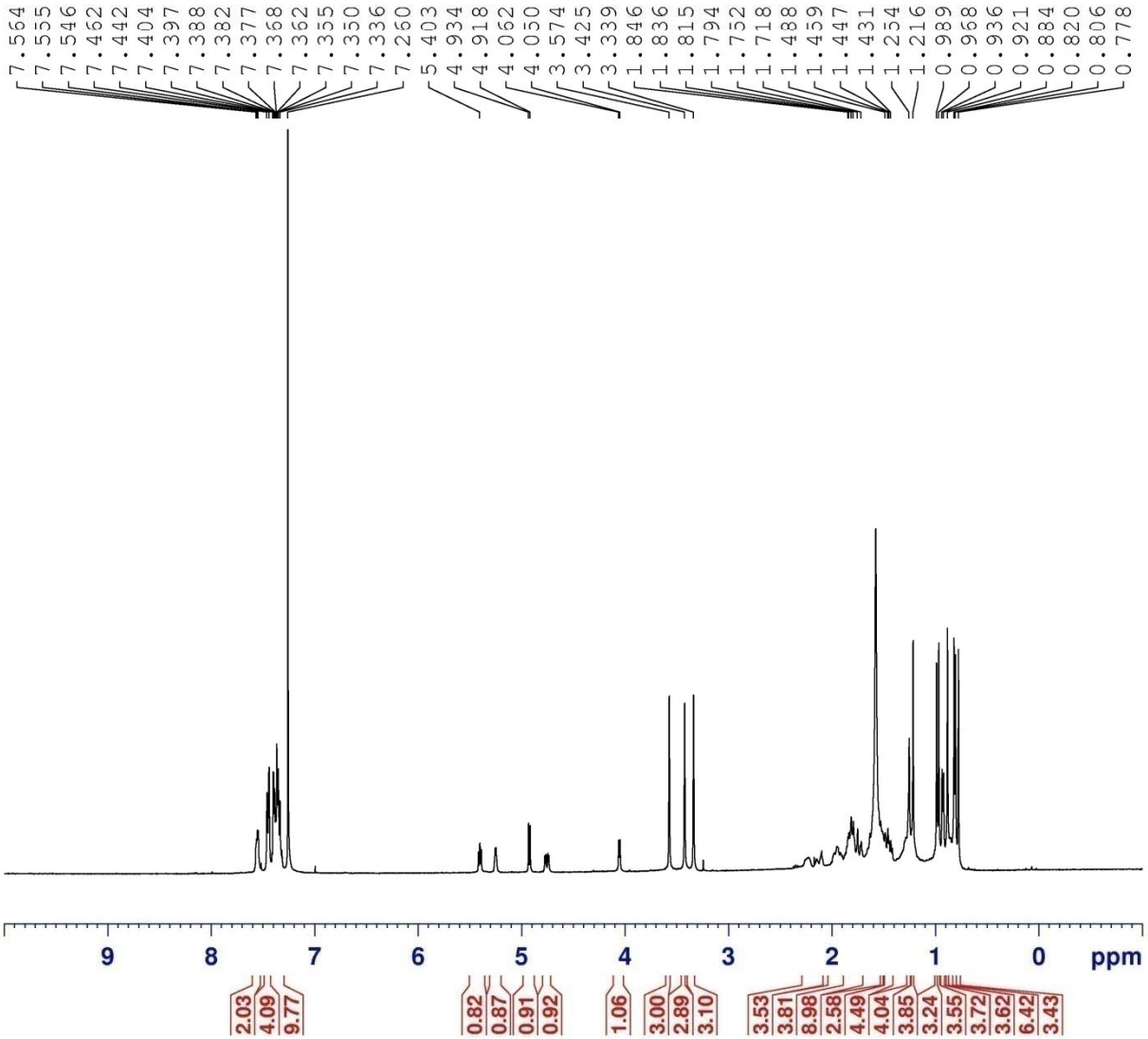


Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
1123.4963	1	C60H72F9O10	100.00	1123.4976	-1.2	-1.3	113.8	20.5	even	ok
1145.4827	1	C60H71F9NaO10	100.00	1145.4796	-2.7	-3.1	154.1	20.5	even	ok

jiangzhongping_53-5-R_pos_27_01_5627.d

Bruker Compass DataAnalysis 4.1 printed: 10/18/2018 3:06:11 PM by: SCSIO Page 1 of 1

^{1}H NMR (400 MHz) spectrum of compound **4r** in $CDCl_3$

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                                   ER
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PROCNO                              1
Date_                        20180728
Time                            21.32
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                              208.5
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                             11.50 usec
SI                              65536
SF                        400.1300098 MHz
WDW                                EM
SSB                                 0
LB                               0.30 Hz
GB                                  0
PC                               1.00
```
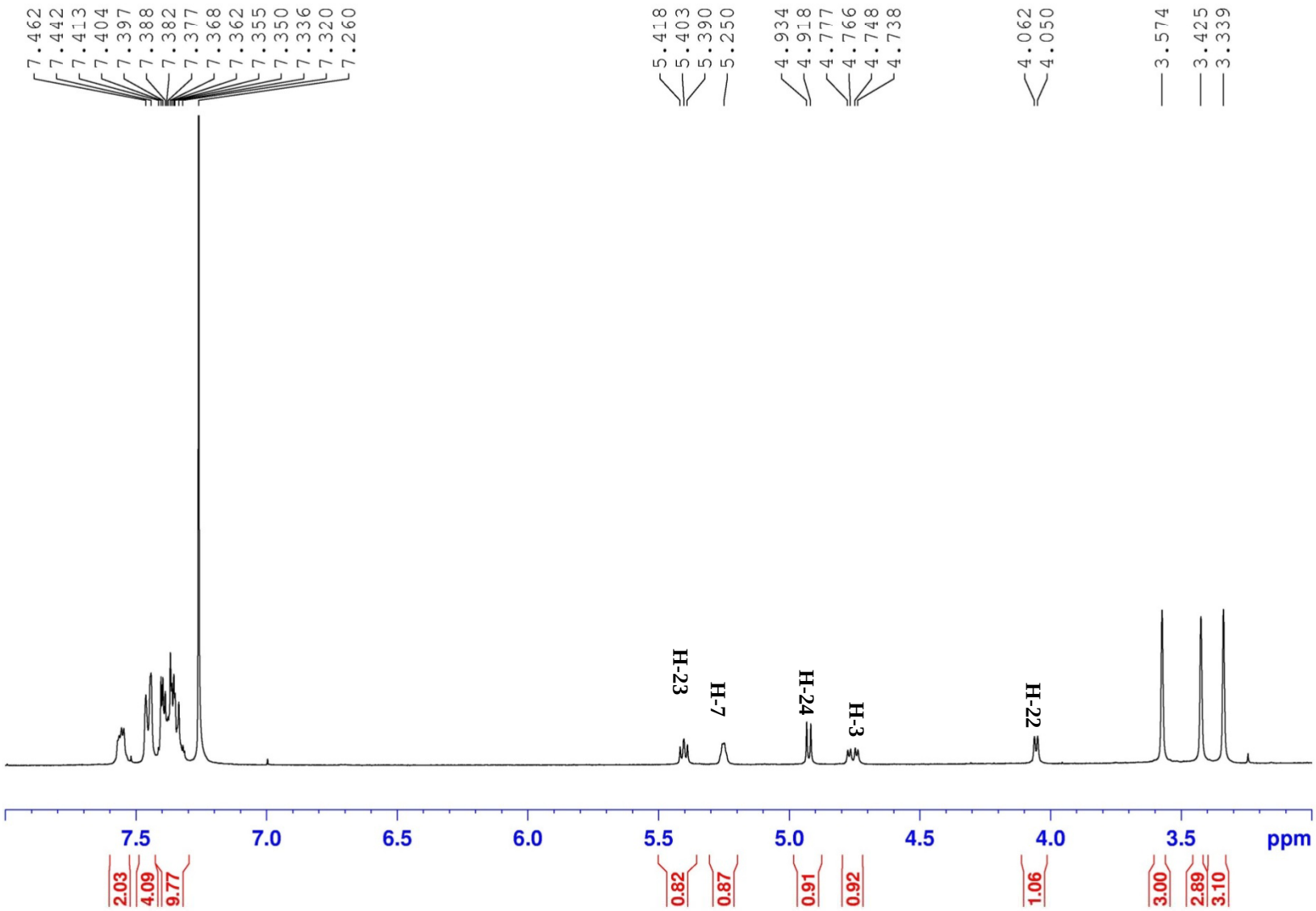
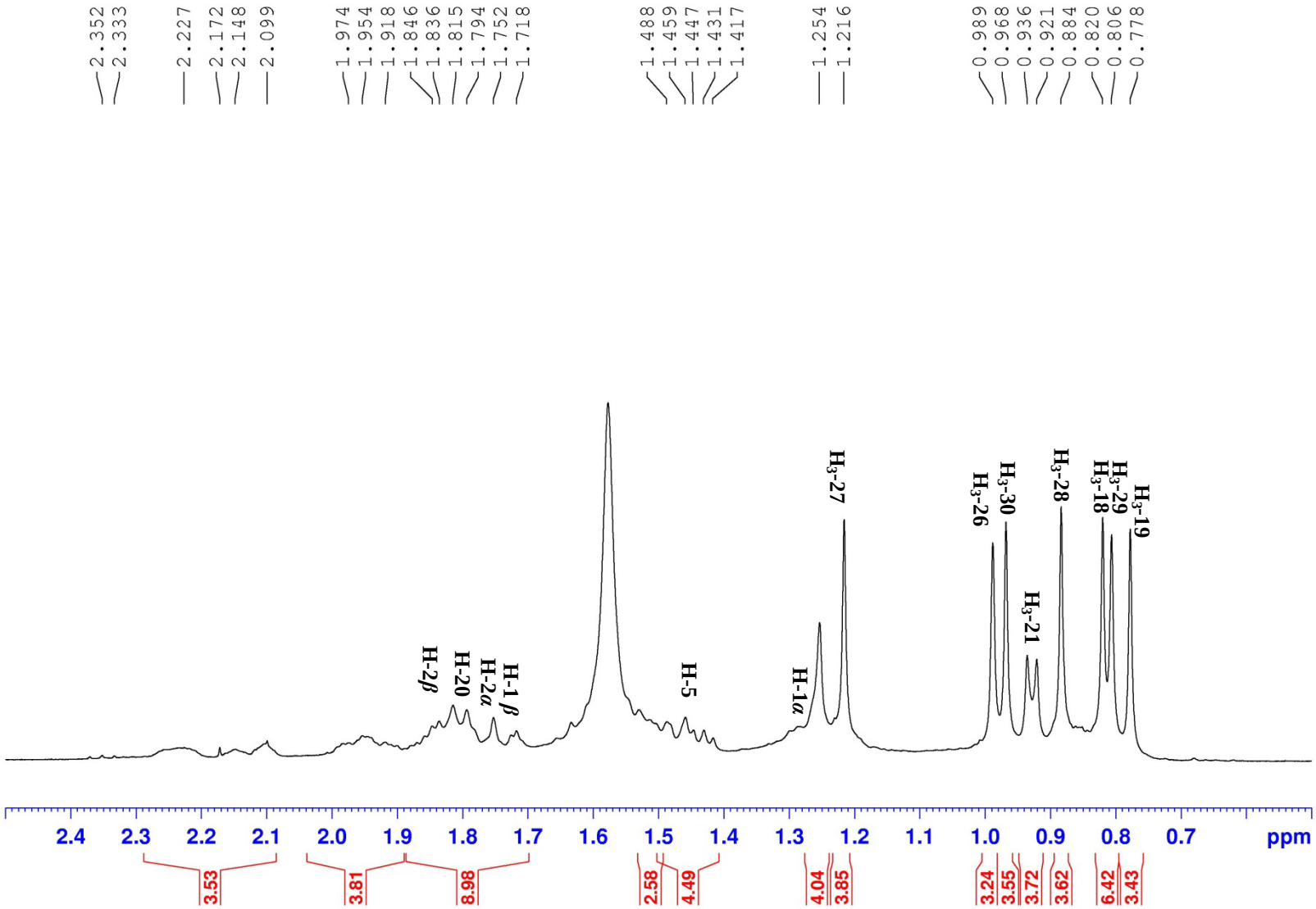

^{1}H NMR (400 MHz) spectrum of compound **4r** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **4r** in $CDCl_3$



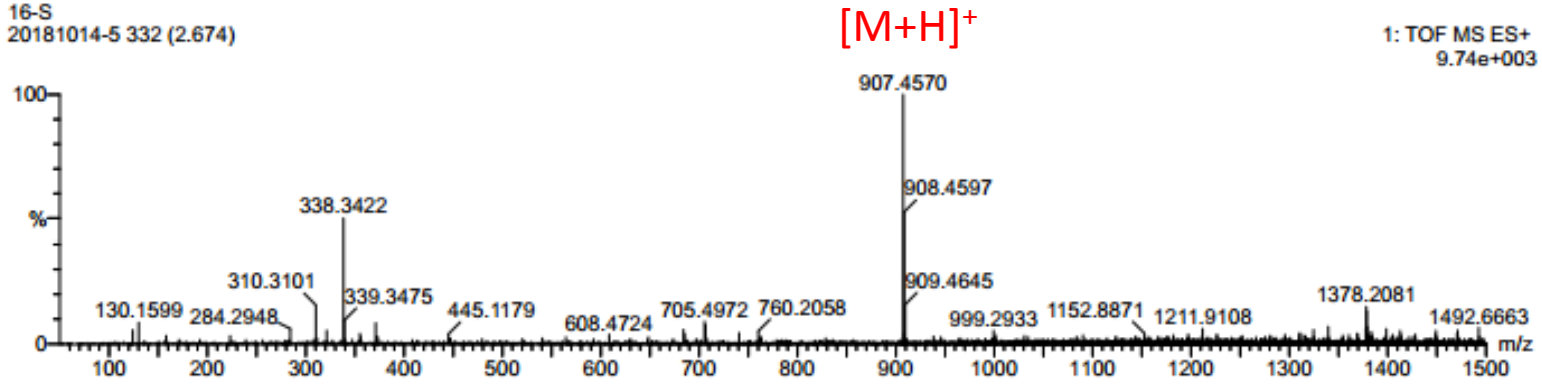
HR-ESIMS for compound **6s**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

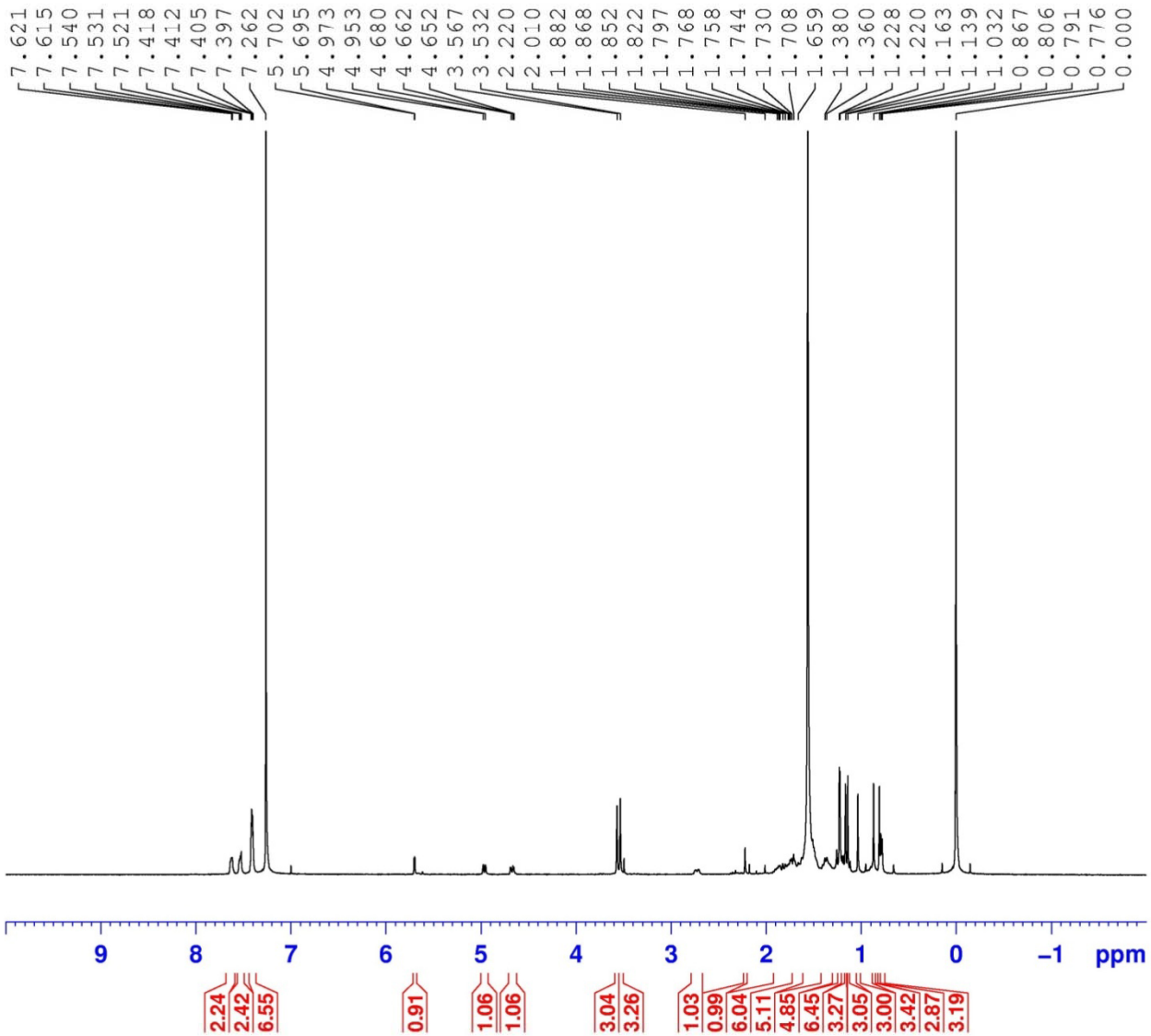
Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-100 O: 0-8 F: 0-6

16-S
20181014-5 332 (2.674)



Minimum:				-1.5				
Maximum:		2.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
907.4570	907.4584	-1.4	-1.5	15.5	142.9	n/a	n/a	C50 H65 O8 F6

^{1}H NMR (400 MHz) spectrum of compound **6s** in $CDCl_3$

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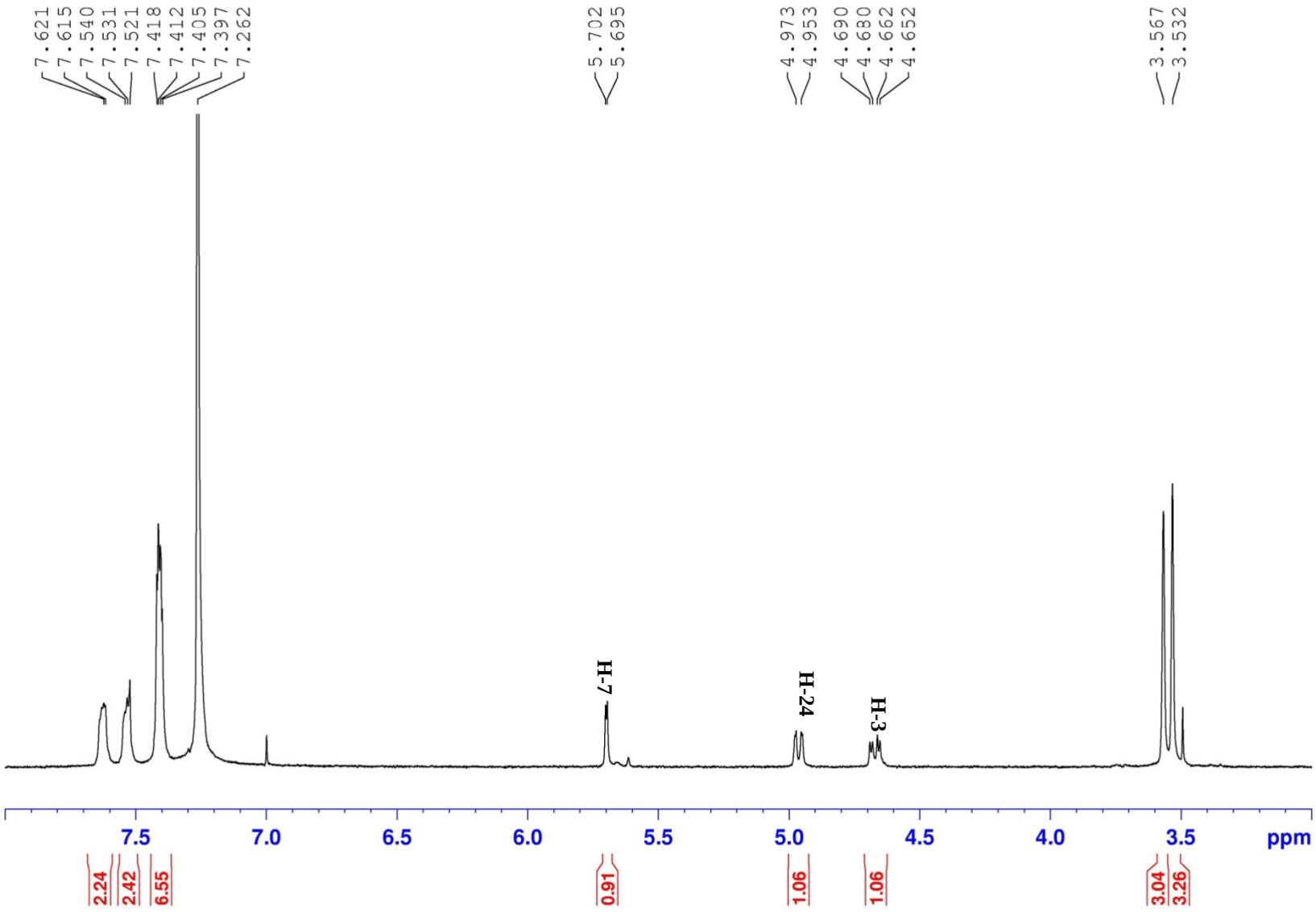
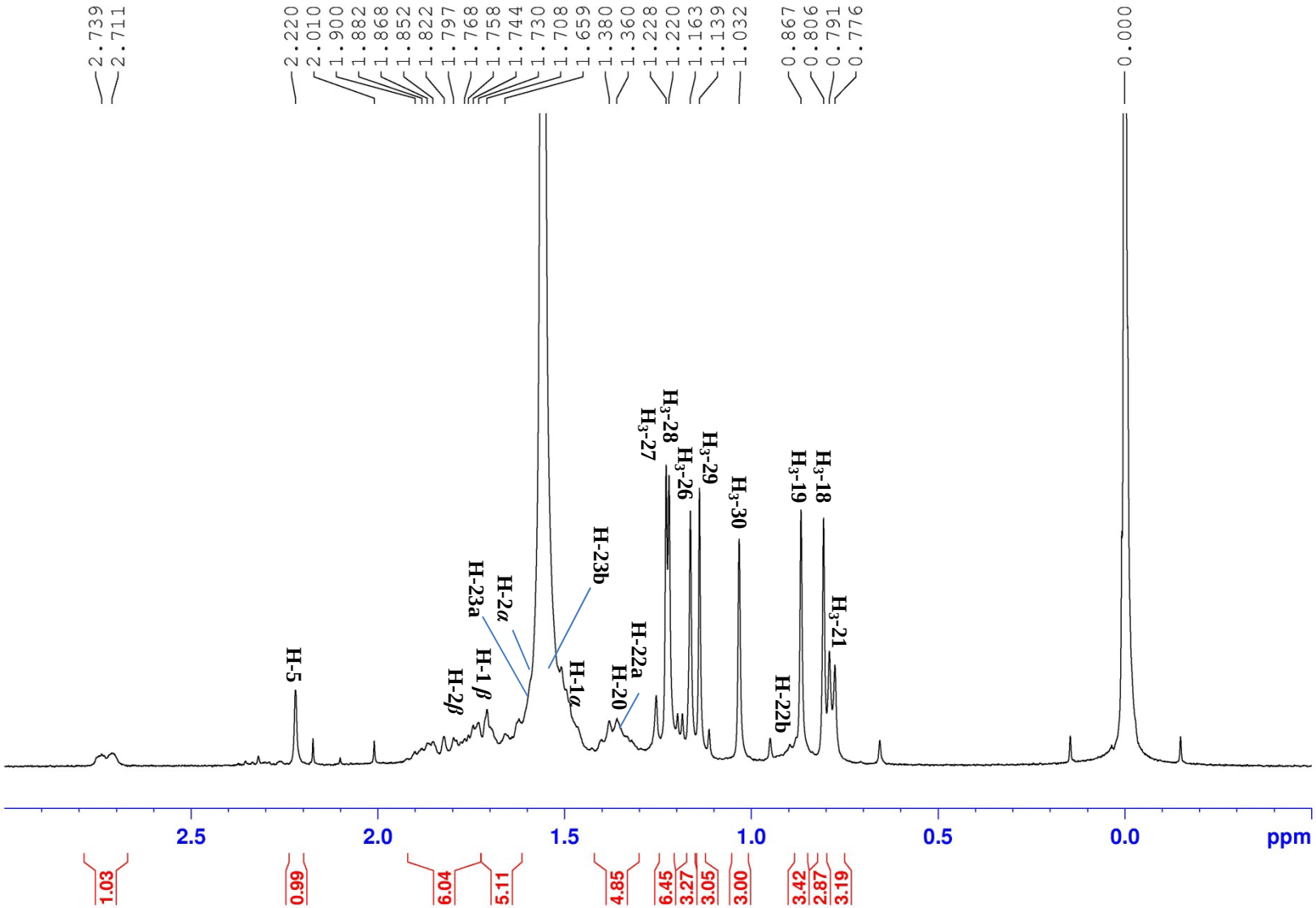
EXPNO                   1
PROCNO                  1
Date_            20170119
Time                23.04
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                  208.5
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                 11.50 usec
SI                 65536
SF            400.1300091 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

^{1}H NMR (400 MHz) spectrum of compound **6s** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **6s** in $CDCl_3$



HR-ESIMS for compound **6r**

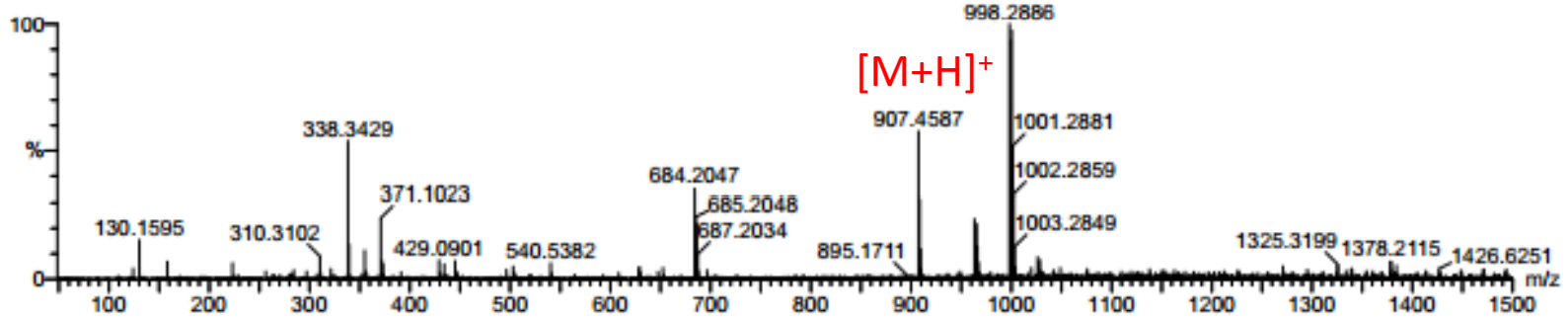
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-100 O: 0-8 F: 0-6

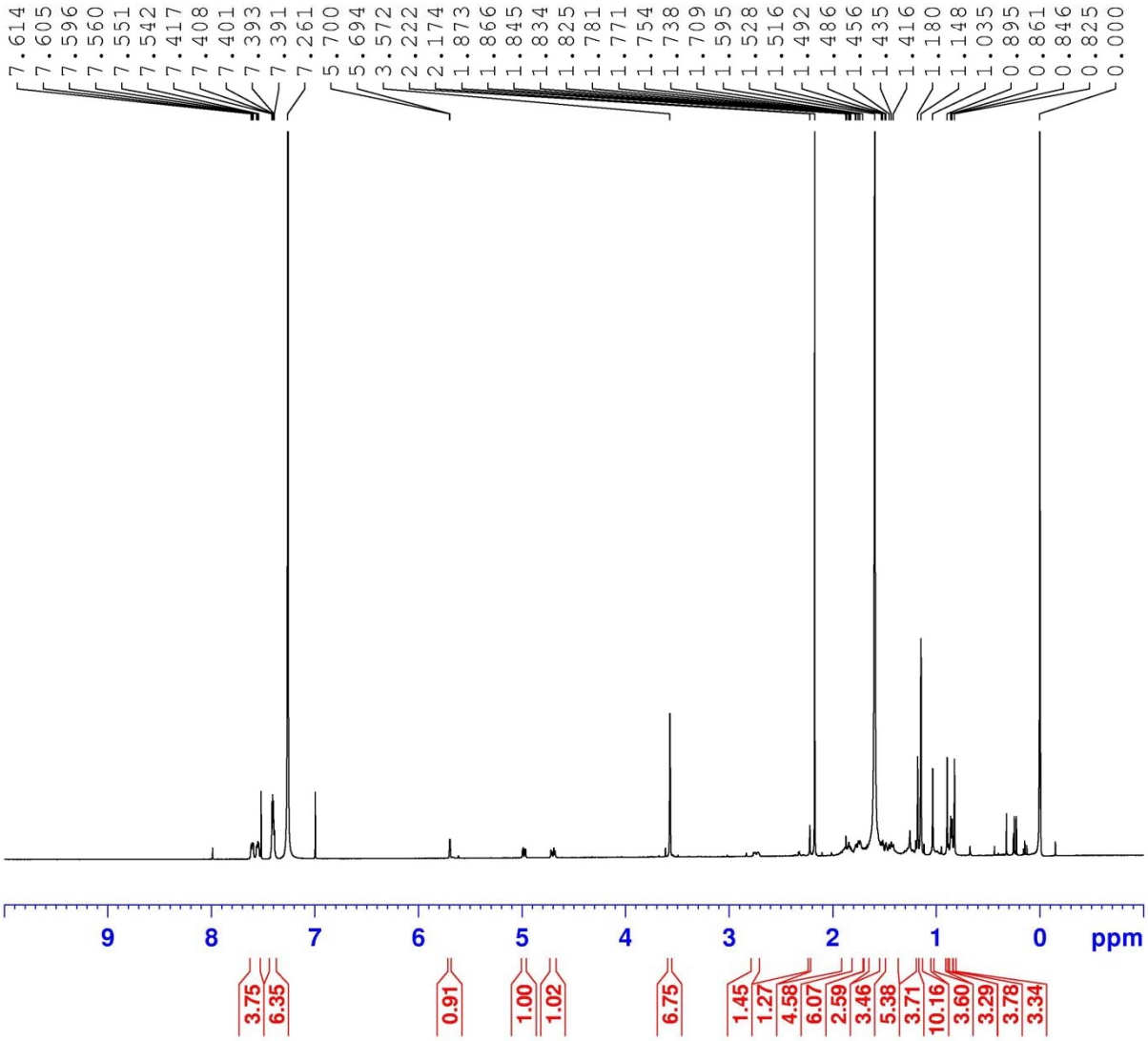
16-R
20181014-4 331 (2.666)

1: TOF MS ES+
1.13e+004



Minimum:				-1.5				
Maximum:		2.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
907.4587	907.4584	0.3	0.3	15.5	157.4	n/a	n/a	C50 H65 O8 F6

^{1}H NMR (400 MHz) spectrum of compound **6r** in $CDCl_3$

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PROCNO                     1
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PROBHD     5 mm CPPBBO BB
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                       128
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                 3.9846387 sec
RG                     208.5
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                    11.50 usec
SI                    65536
SF               400.1300091 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```
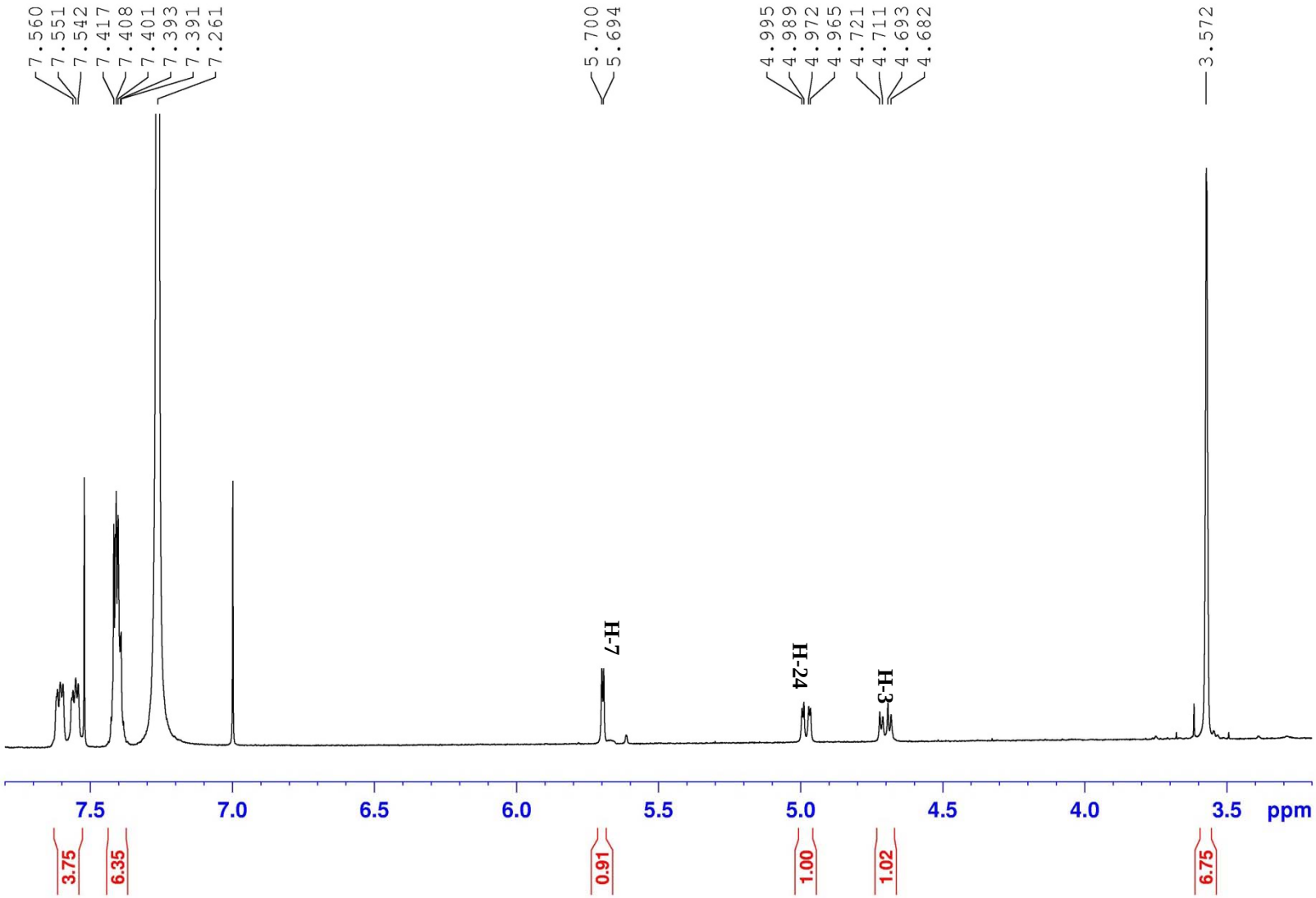
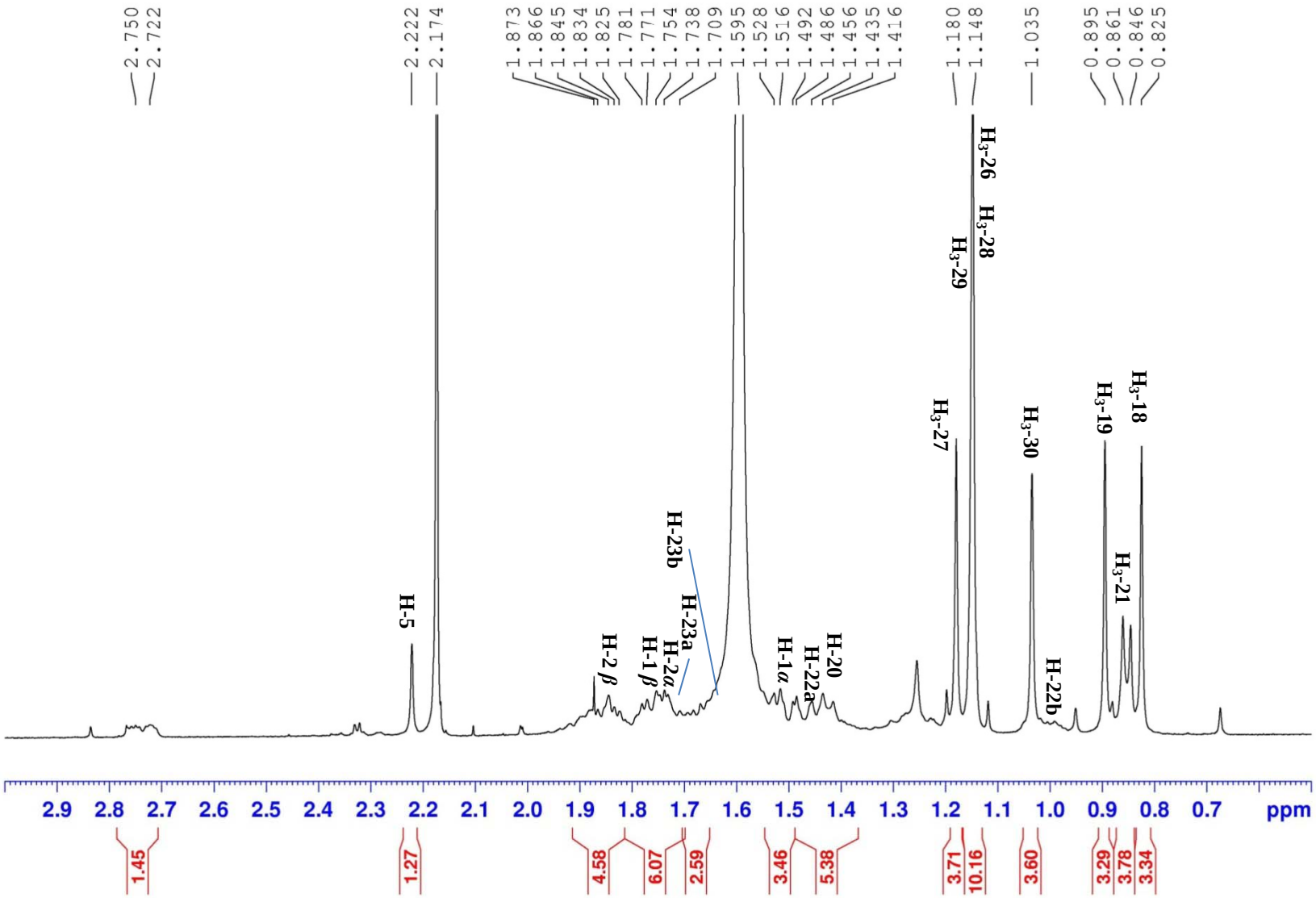

^{1}H NMR (400 MHz) spectrum of compound **6r** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **6r** in $CDCl_3$

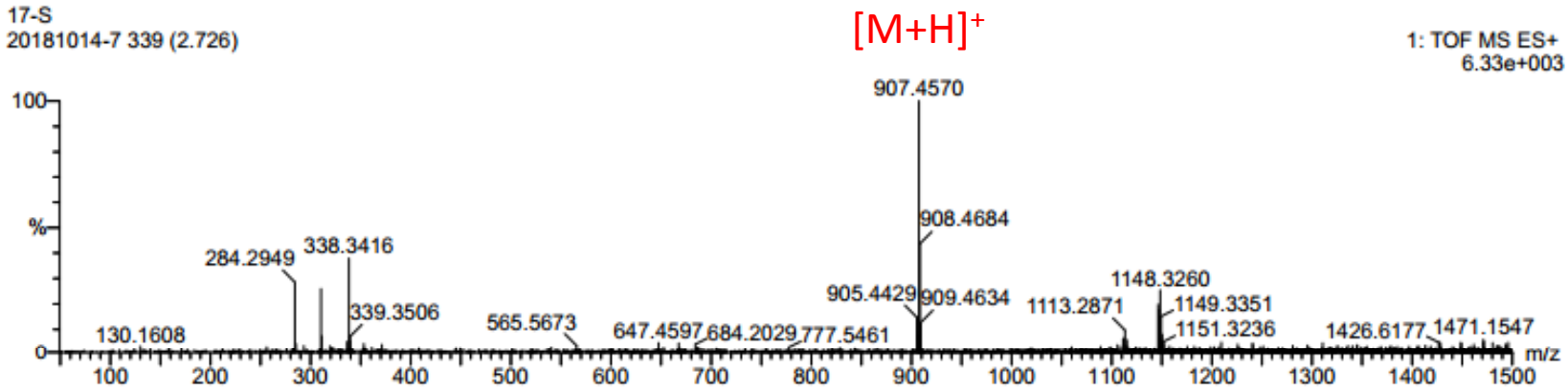


HR-ESIMS for compound **7s**

Elemental Composition Report **Page 1**

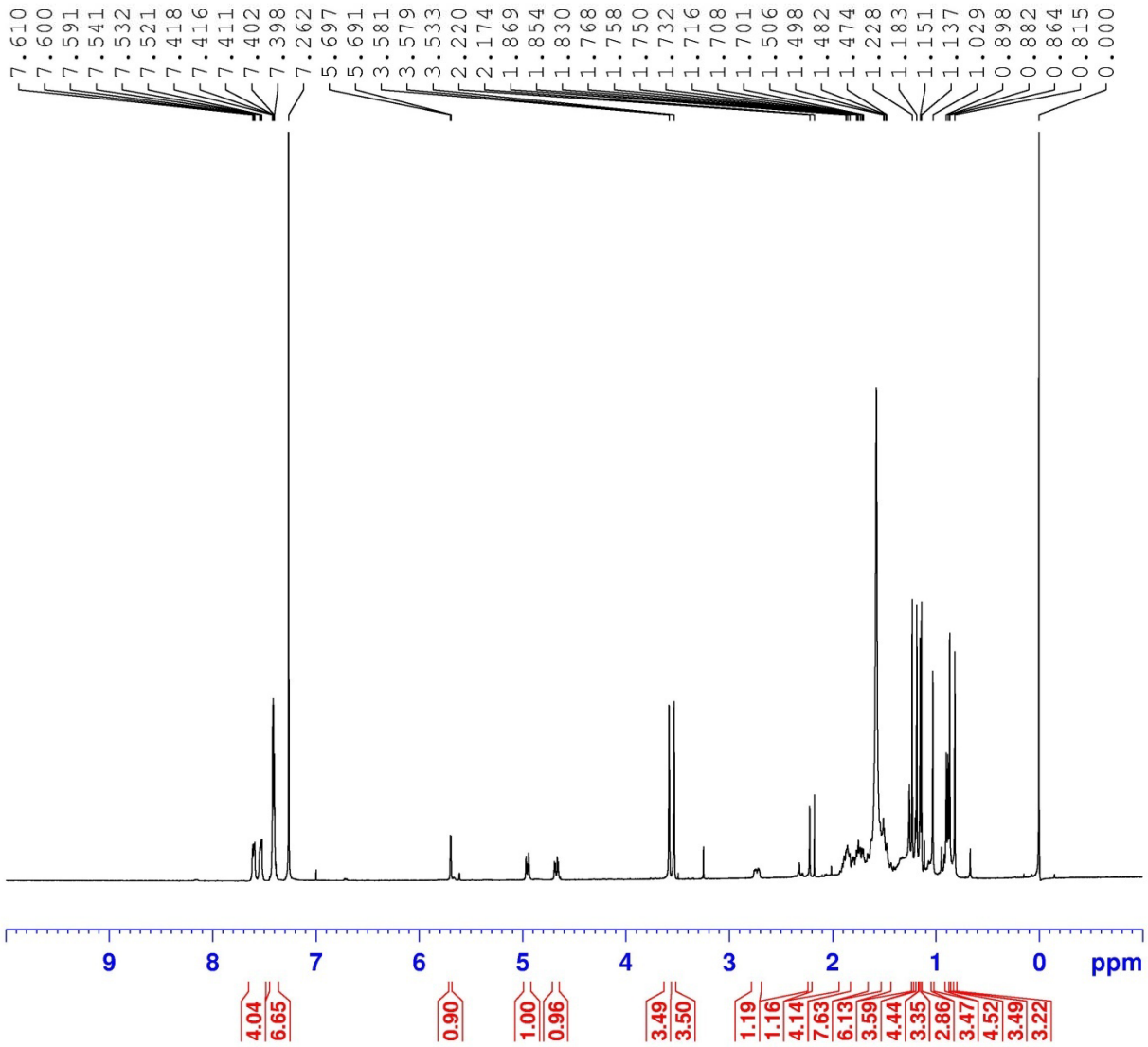
Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-100 O: 0-8 F: 0-6
17-S
20181014-7 339 (2.726)



Minimum:				-1.5				
Maximum:		2.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
907.4570	907.4584	-1.4	-1.5	15.5	120.6	n/a	n/a	C50 H65 O8 F6

^{1}H NMR (400 MHz) spectrum of compound **7s** in $CDCl_3$

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PROCNO                 1
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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                 208.5
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                 11.50 usec
SI                 65536
SF           400.1300089 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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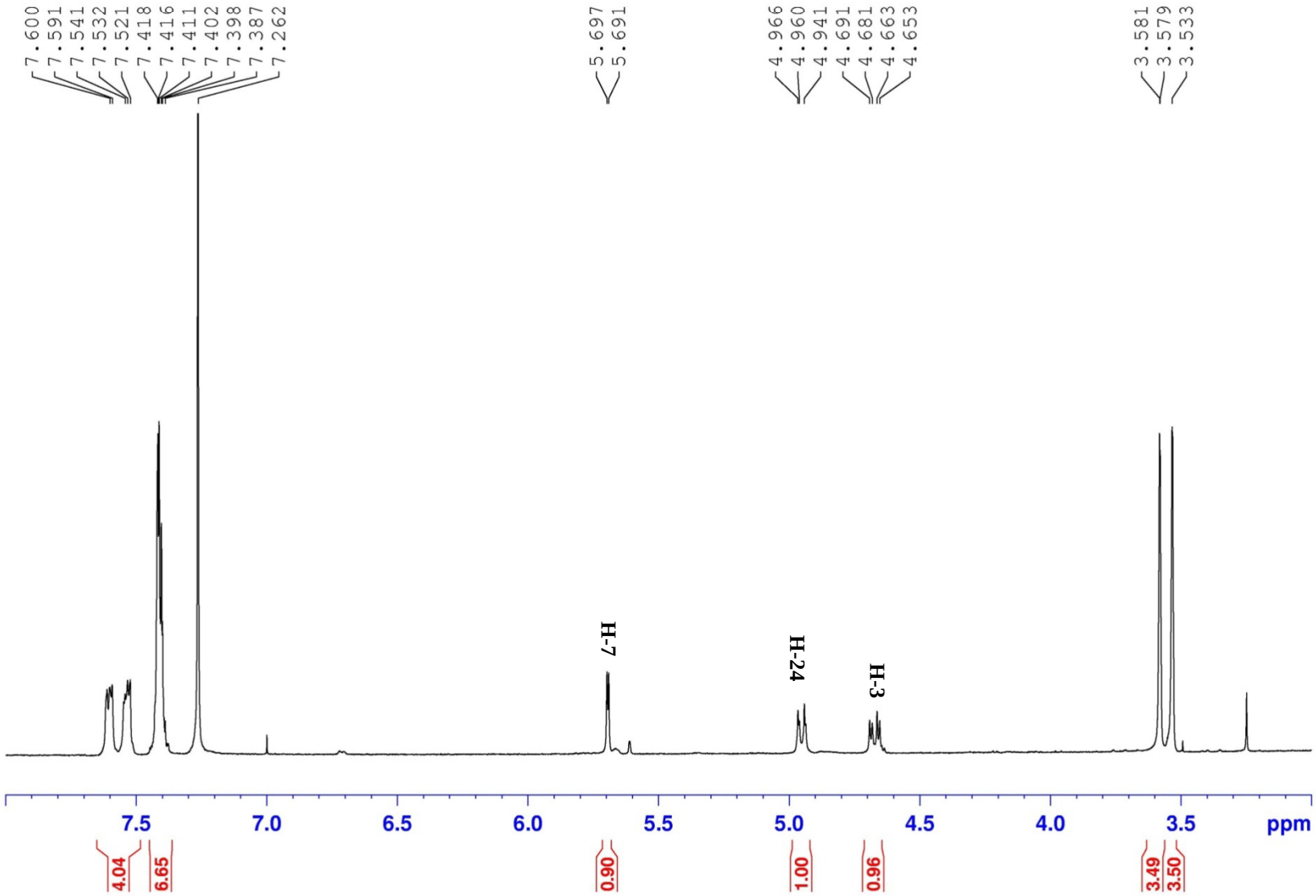
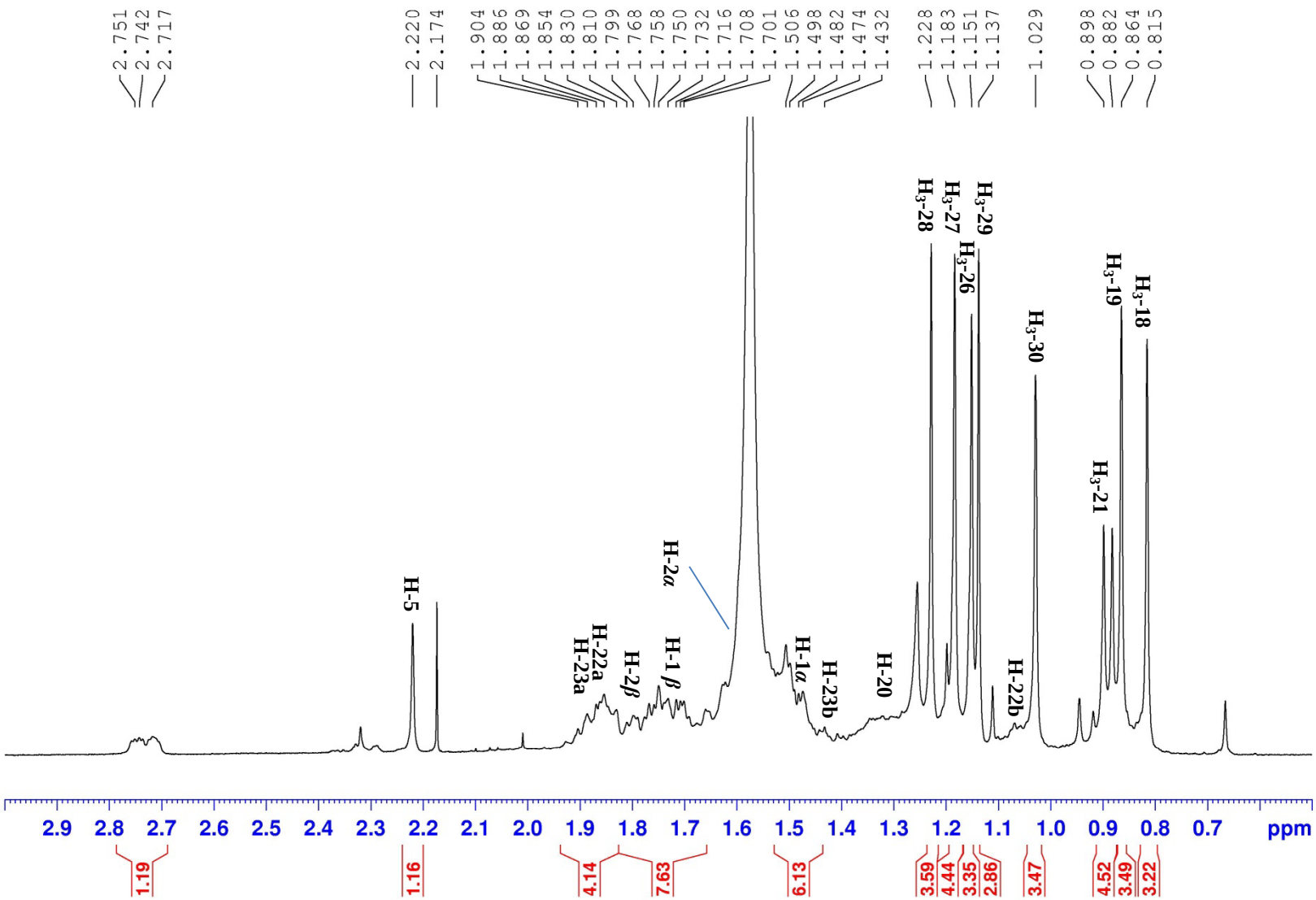
^{1}H NMR (400 MHz) spectrum of compound **7s** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **7s** in $CDCl_3$



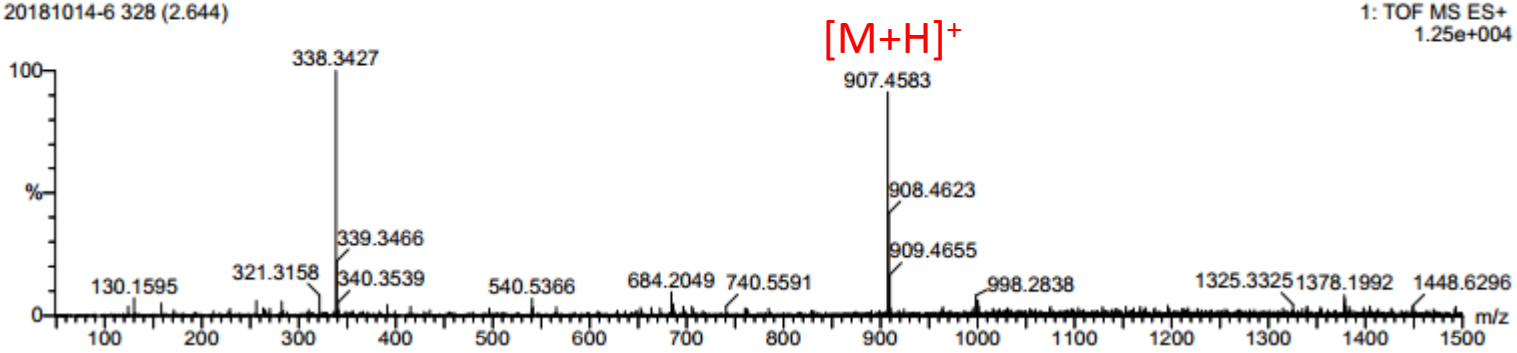
HR-ESIMS for compound **7r**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

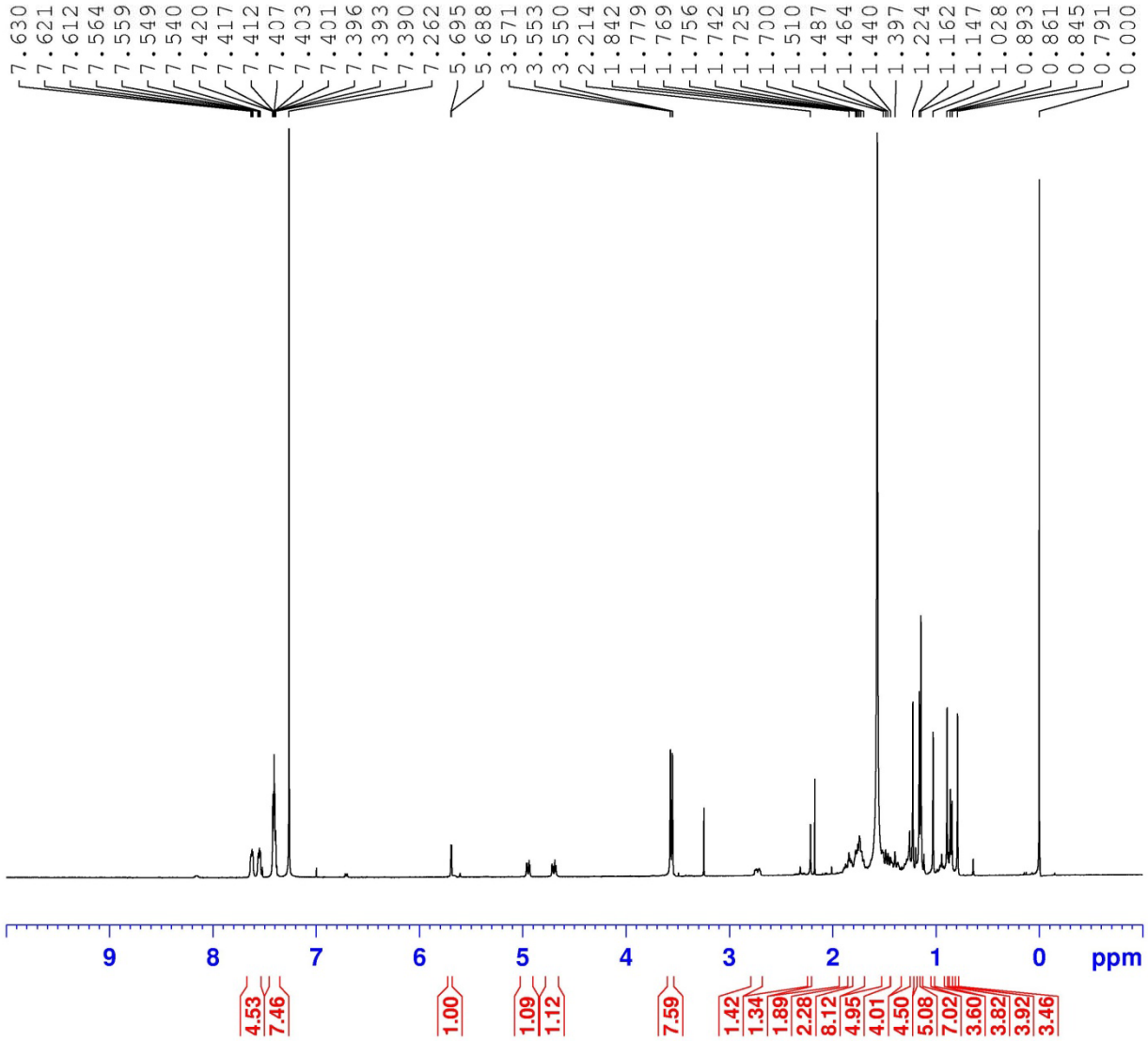
Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-50 H: 0-100 O: 0-8 F: 0-6
17-R
20181014-6 328 (2.644)

1: TOF MS ES+
1.25e+004



Minimum:				-1.5				
Maximum:		2.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
907.4583	907.4584	-0.1	-0.1	15.5	191.0	n/a	n/a	C50 H65 O8 F6

^{1}H NMR (400 MHz) spectrum of compound **7r** in $CDCl_3$

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PROCNO                   1
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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                   208.5
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                   11.50 usec
SI                   65536
SF             400.1300089 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
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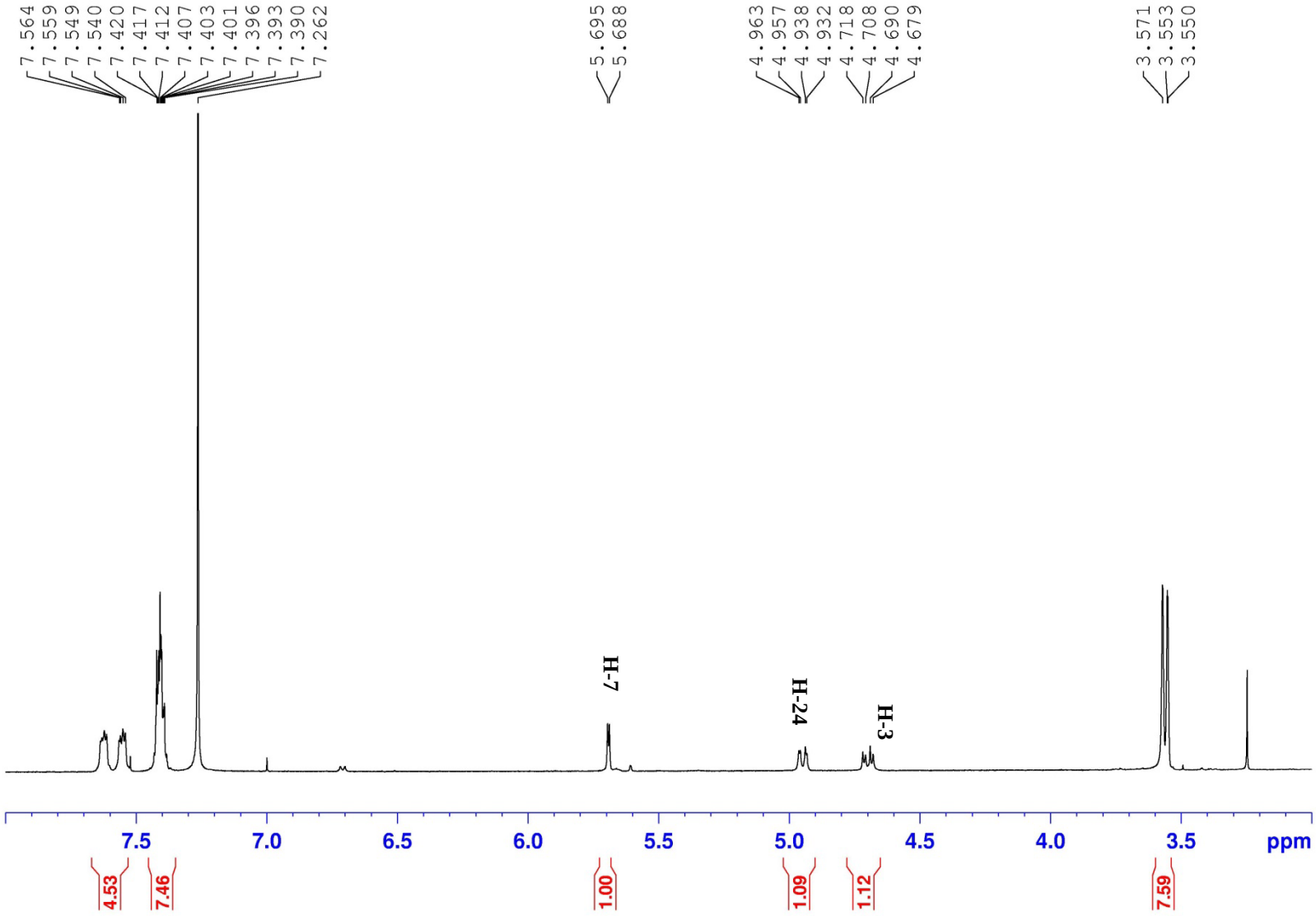
^{1}H NMR (400 MHz) spectrum of compound **7r** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **7r** in $CDCl_3$