

Electronic Supplementary Information (ESI):

Thaixylomolins O–R: Four New limonoids from the Trang mangrove,

Xylocarpus moluccensis

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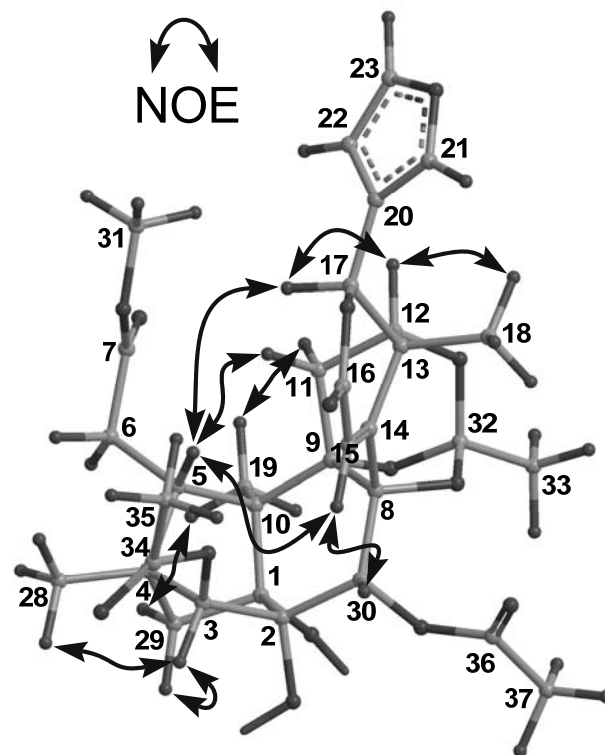


Figure S1. Diagnostic NOE interactions for thaixylomolin P (**2**).

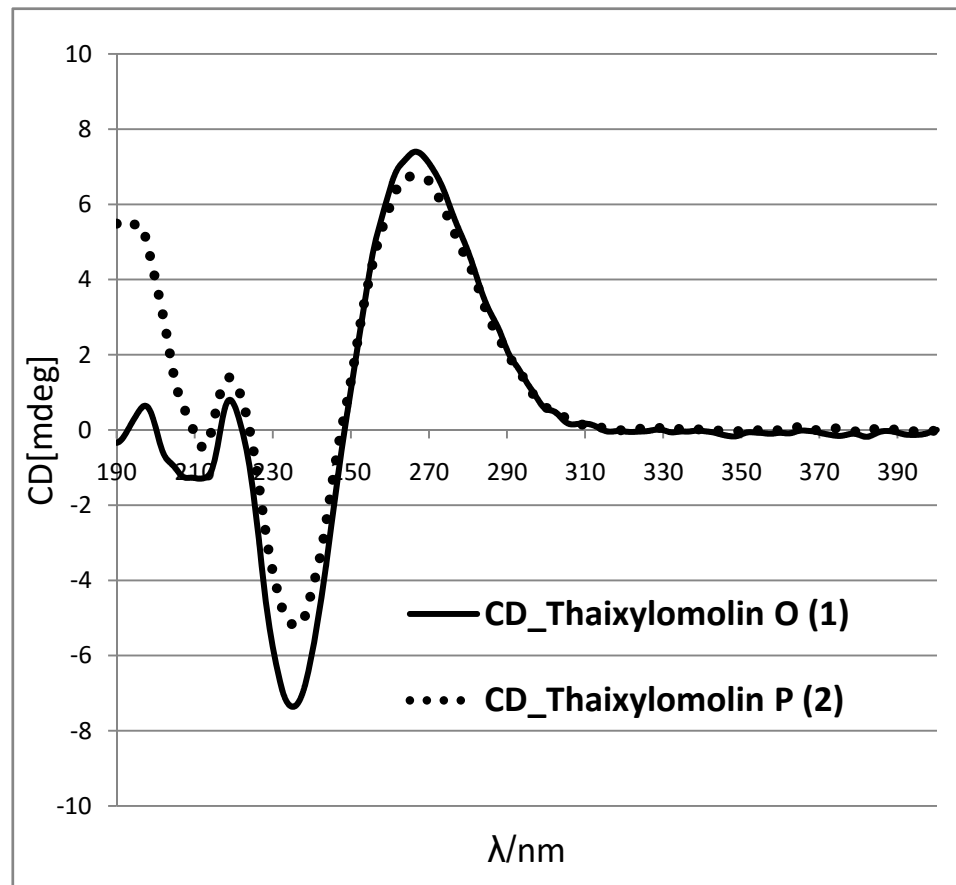


Figure S2. ECD spectra of thaixylomolins O (1) and P (2) measured in MeCN at the concentration of 250 $\mu\text{g/mL}$.

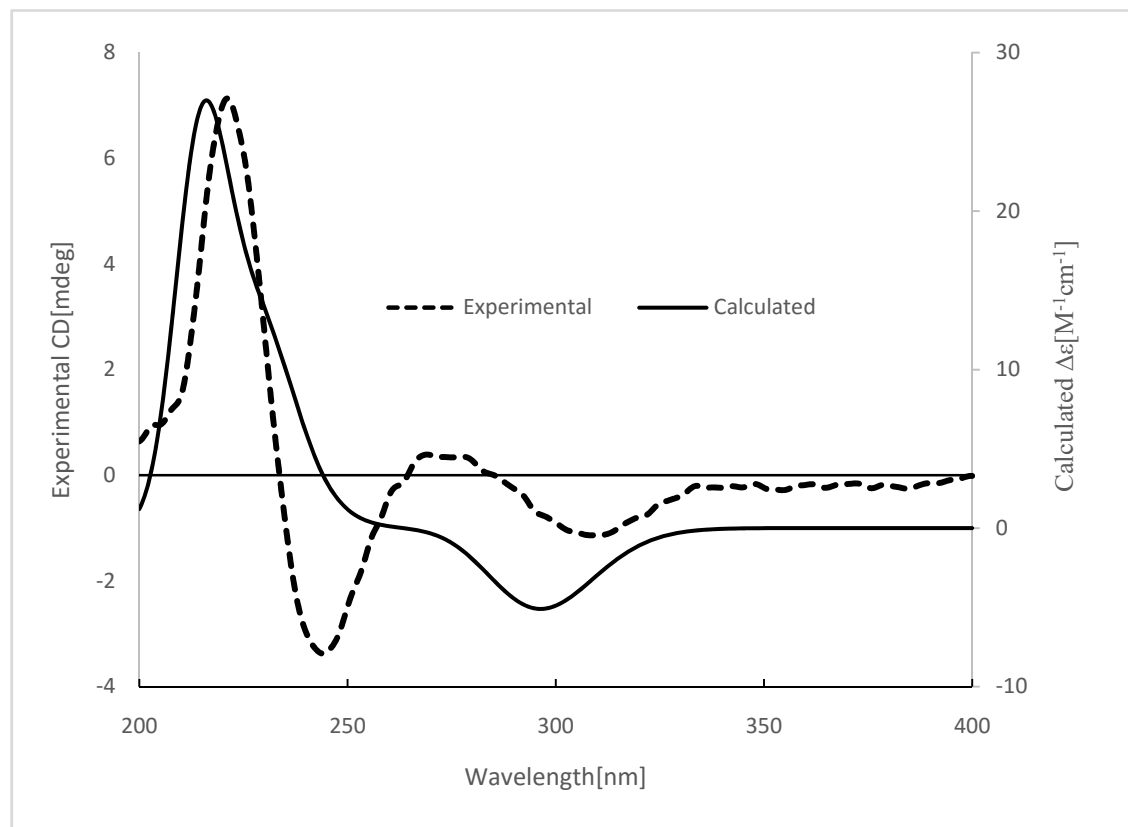


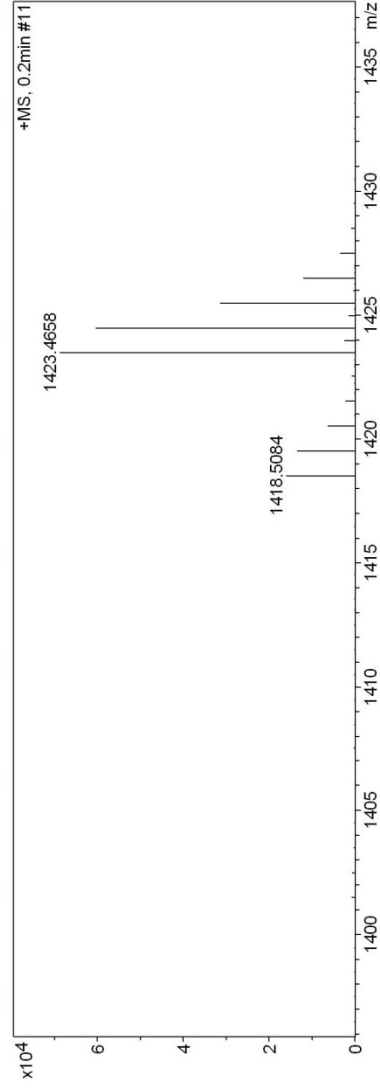
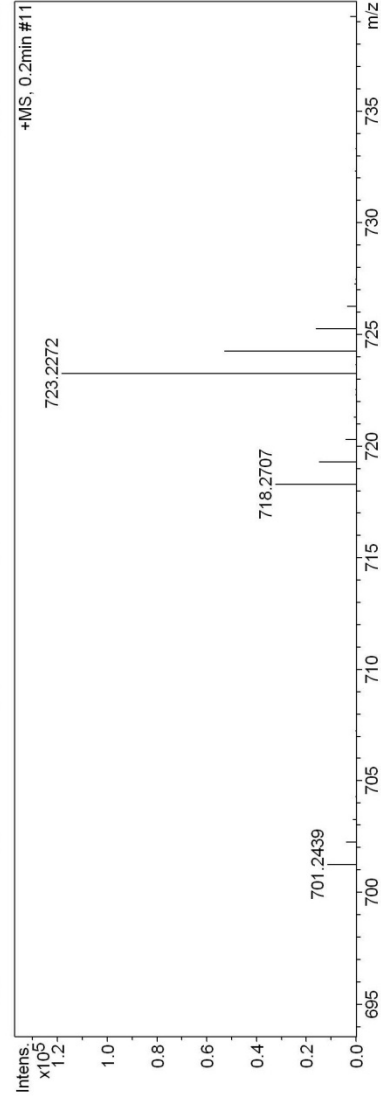
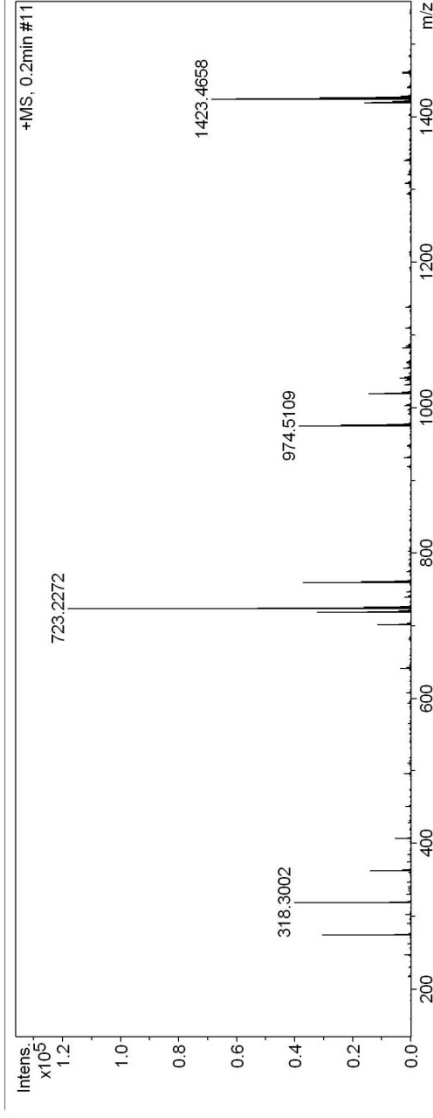
Figure S3. Comparison of the experimental ECD spectrum of thaxylomolin R (**4**) in MeCN with that calculated for 1*R*,5*R*,9*S*,10*R*,13*R*,15*R*,17*R*-**4**.

HR-ESIMS for compound 1

Generic Display Report

Analysis Info

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Sample Name liwanshan_dgy-18_pos Instrument maXis
Comment



HR-ESIMS for compound 1

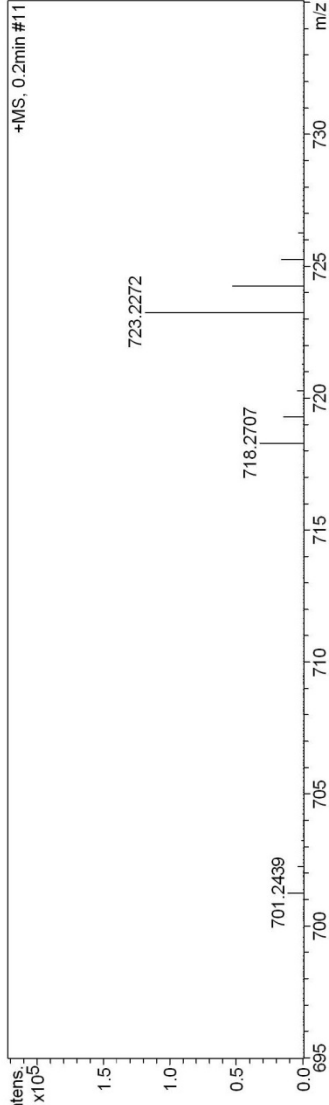
Mass Spectrum SmartFormula Report

Analysis Info

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 Sample Name liwanshan_dgy-18_pos Instrument / Ser# maXis 29
 Comment

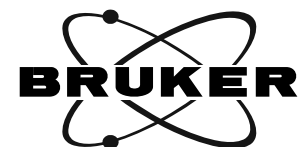
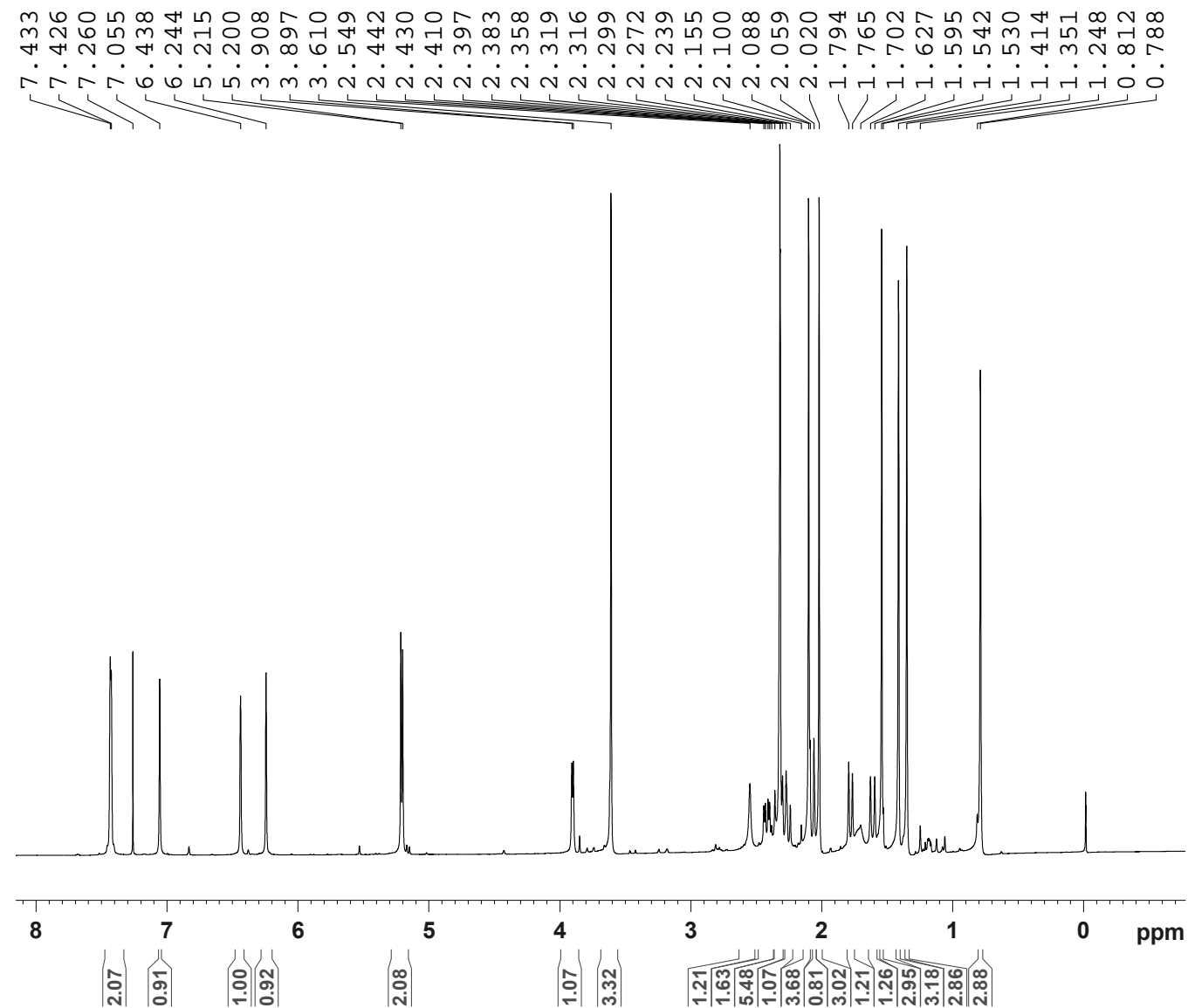
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 Scan Begin 100 m/z -500 V Set Dry Gas 4.0 l/min
 Scan End 2000 m/z 800.0 Vpp Set Divert Valve Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rb	e ⁻ Conf	N-Rule
701.2439	1	C ₃₅ H ₄₁ O ₁₅	100.00	701.2440	0.2	0.1	21.2	15.5	even	ok
723.2272	1	C ₃₅ H ₄₀ NaO ₁₅	71.03	723.2259	-1.7	-1.3	33.3	15.5	even	ok
1423.4658	1	C ₇₀ H ₈₀ NaO ₃₀	11.49	1423.4627	-2.2	-3.1	61.2	30.5	even	ok

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

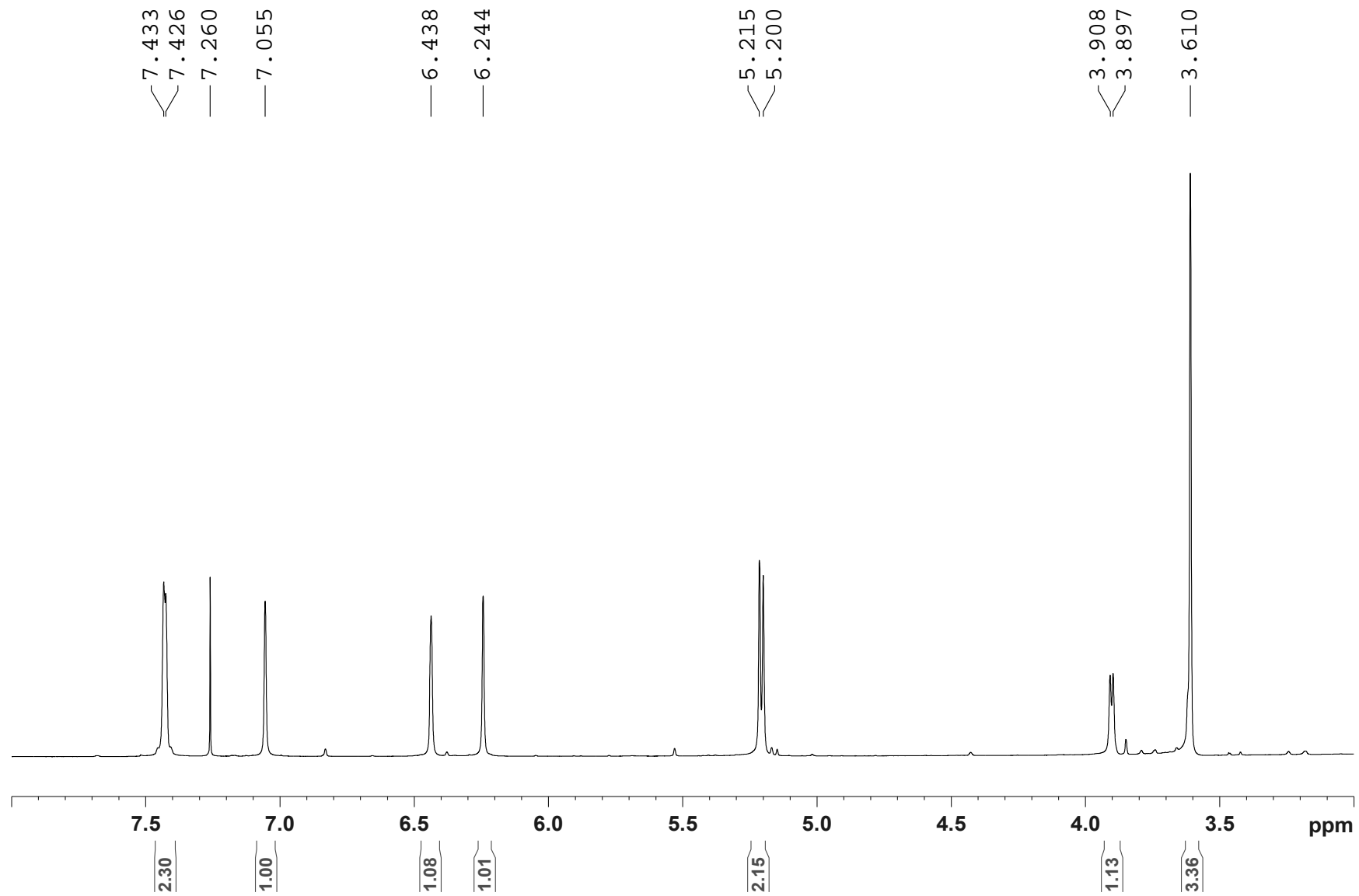


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EXPNO 1
PROCNO 1
Date_ 20151105
Time 5.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 44.55
DW 60.800 usec
DE 10.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

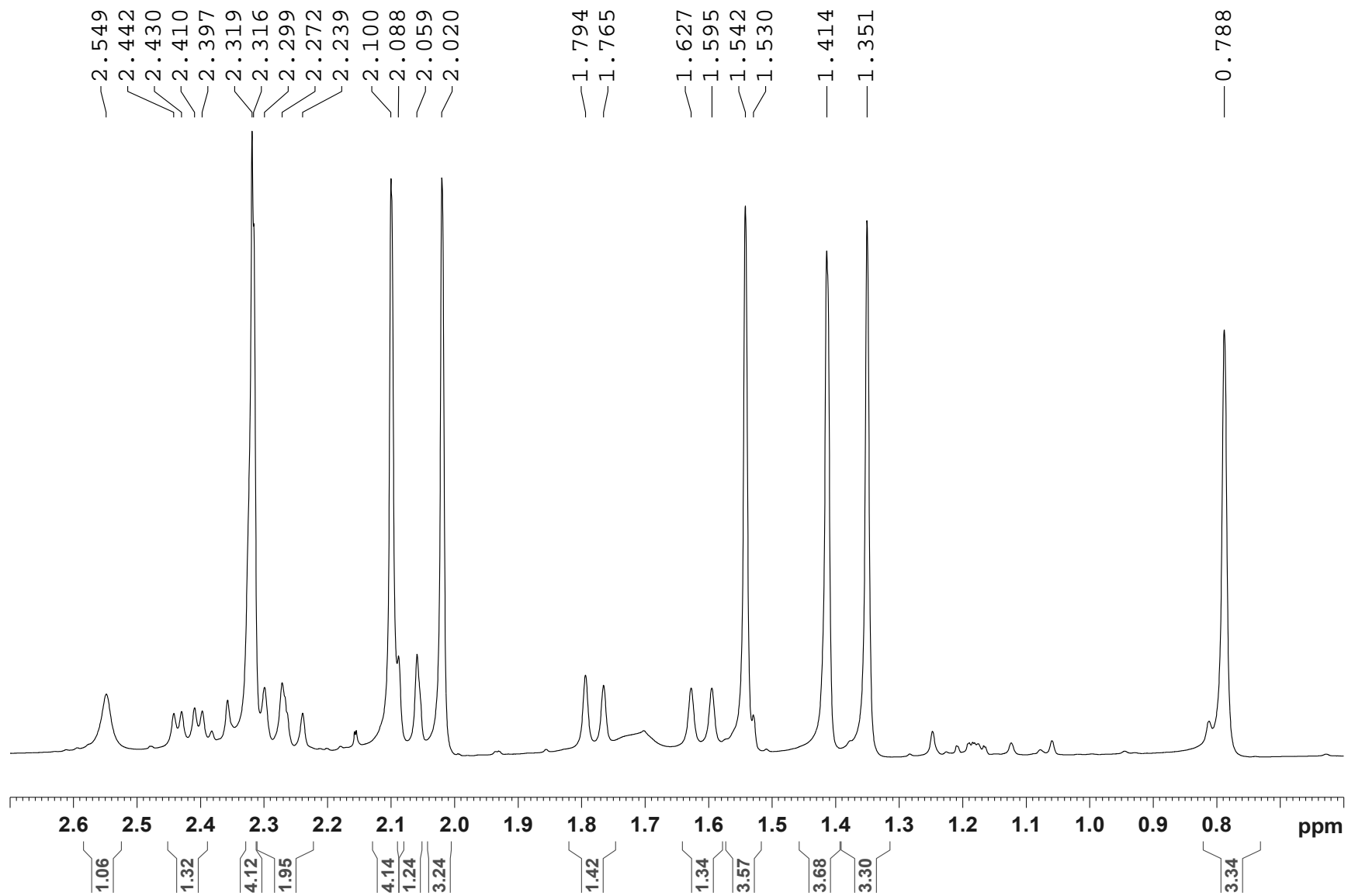
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S10

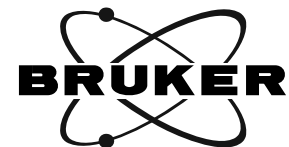
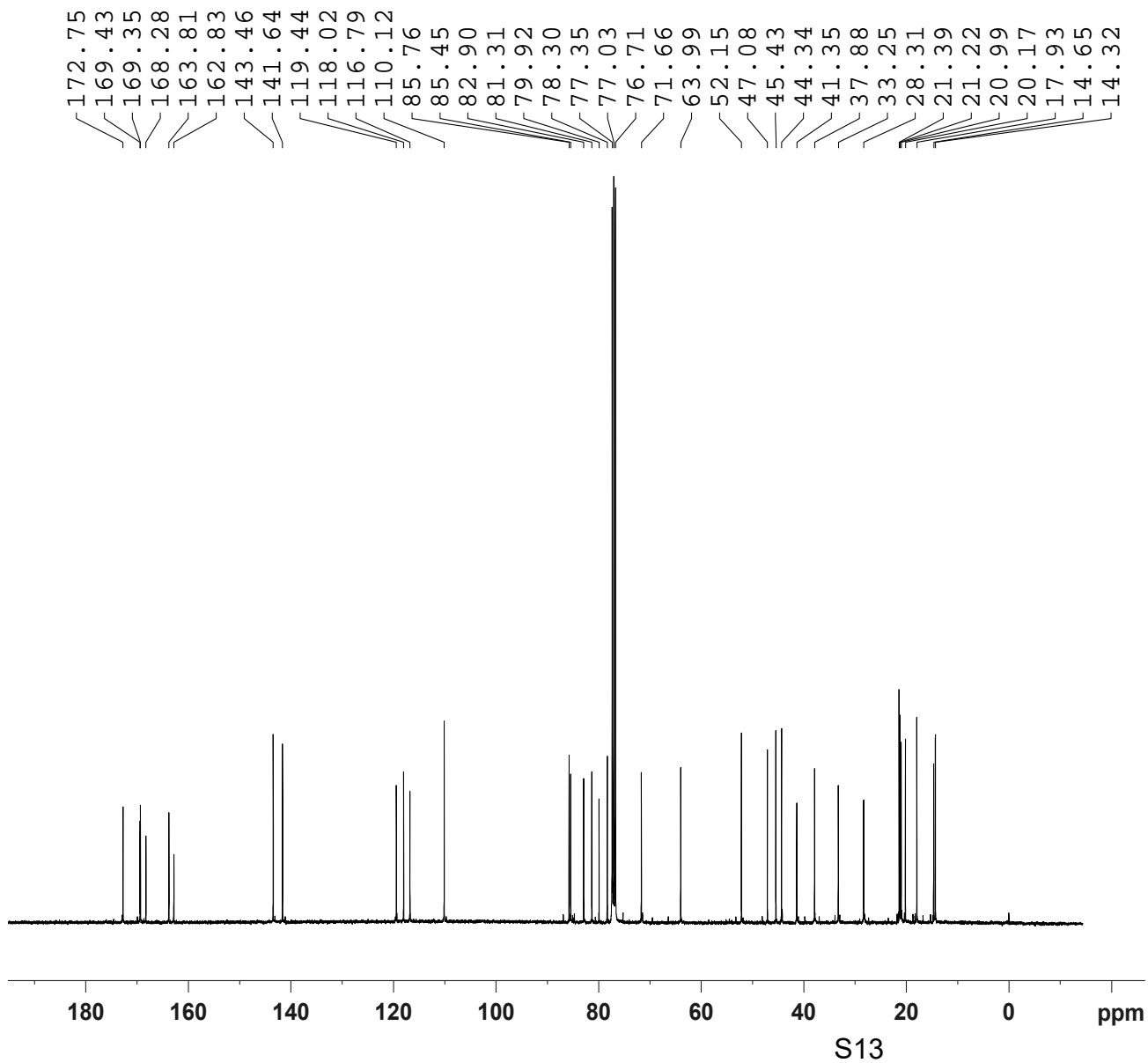
^1H NMR (400 MHz) spectrum of compound **1** in CDCl_3



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



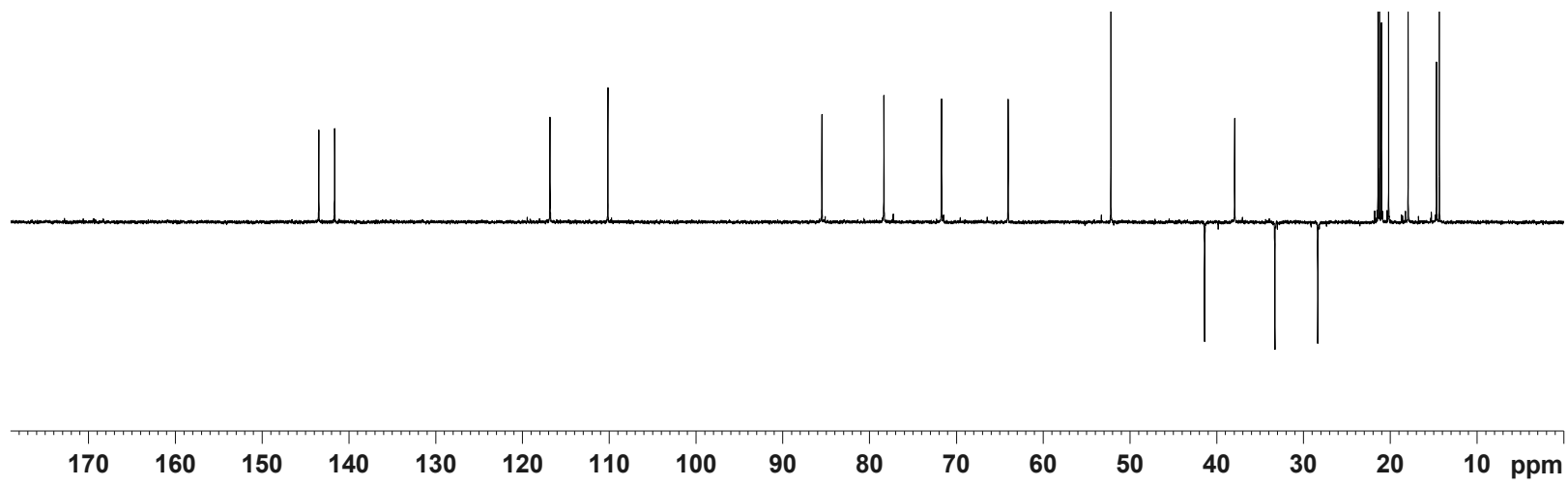
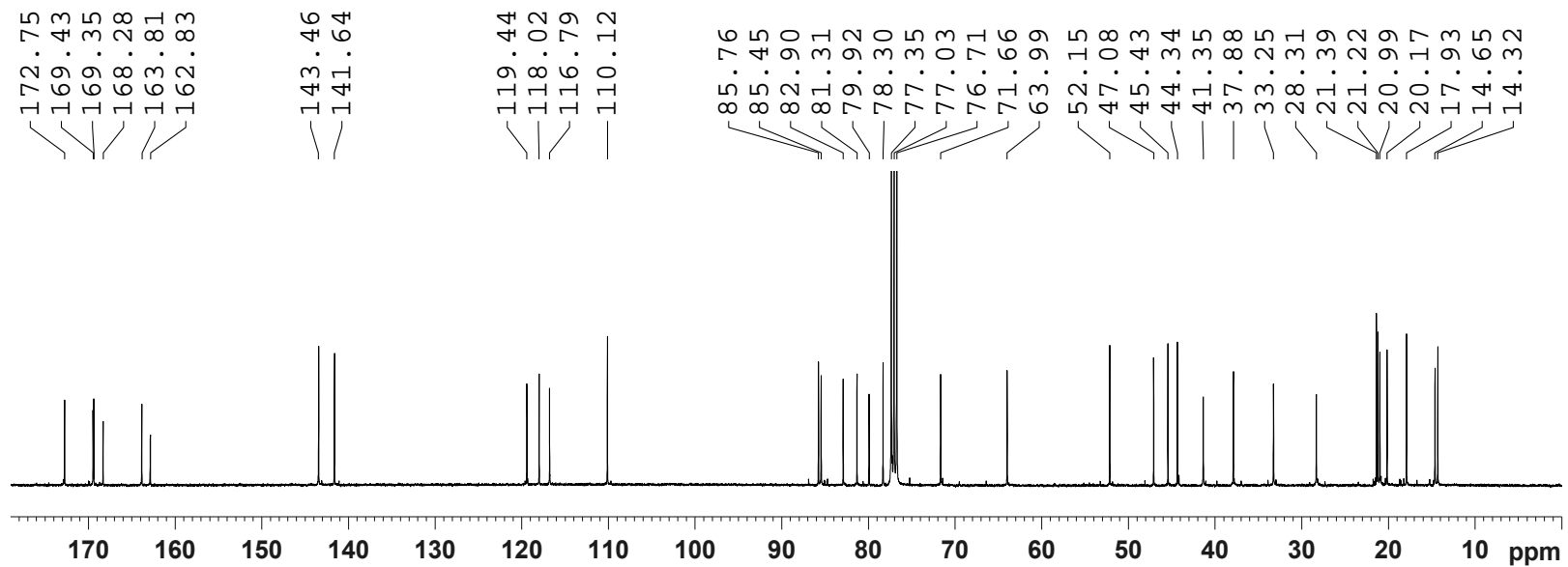
¹³C NMR (100 MHz) spectrum of compound **1** in CDCl₃



NAME dyg-18
EXPNO 2
PROCNO 1
Date_ 20151105
Time 6.22
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

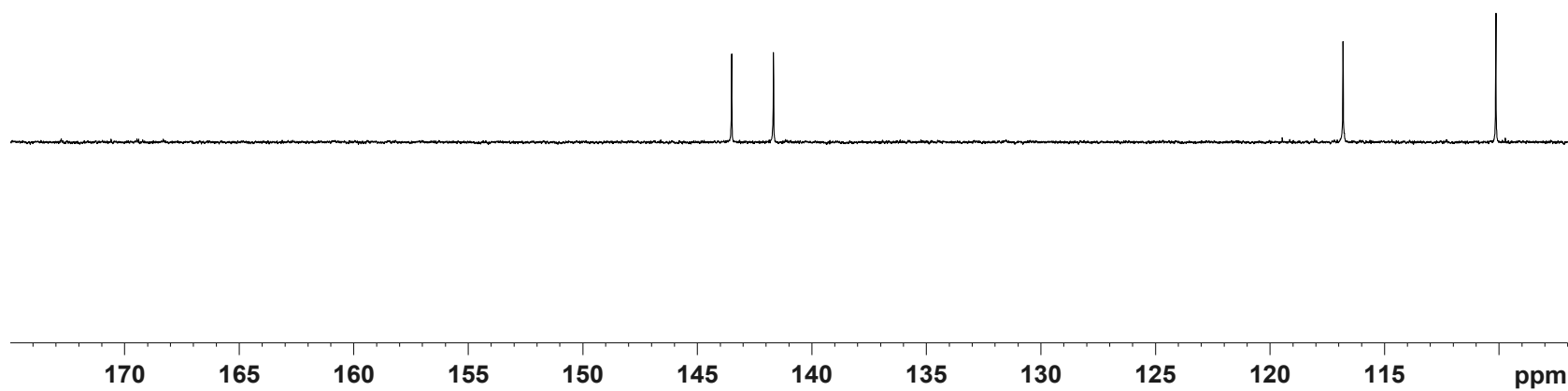
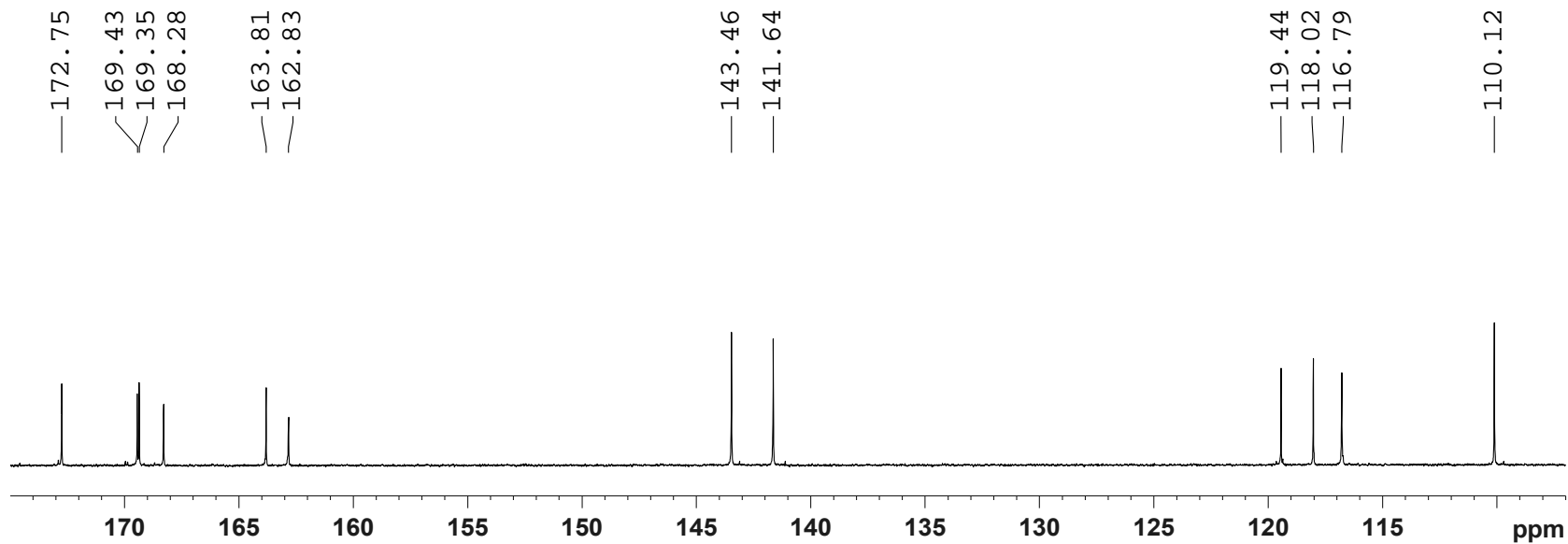
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NUC1 13C
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SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DEPT135 (100 MHz) spectrum of compound **1** in CDCl₃

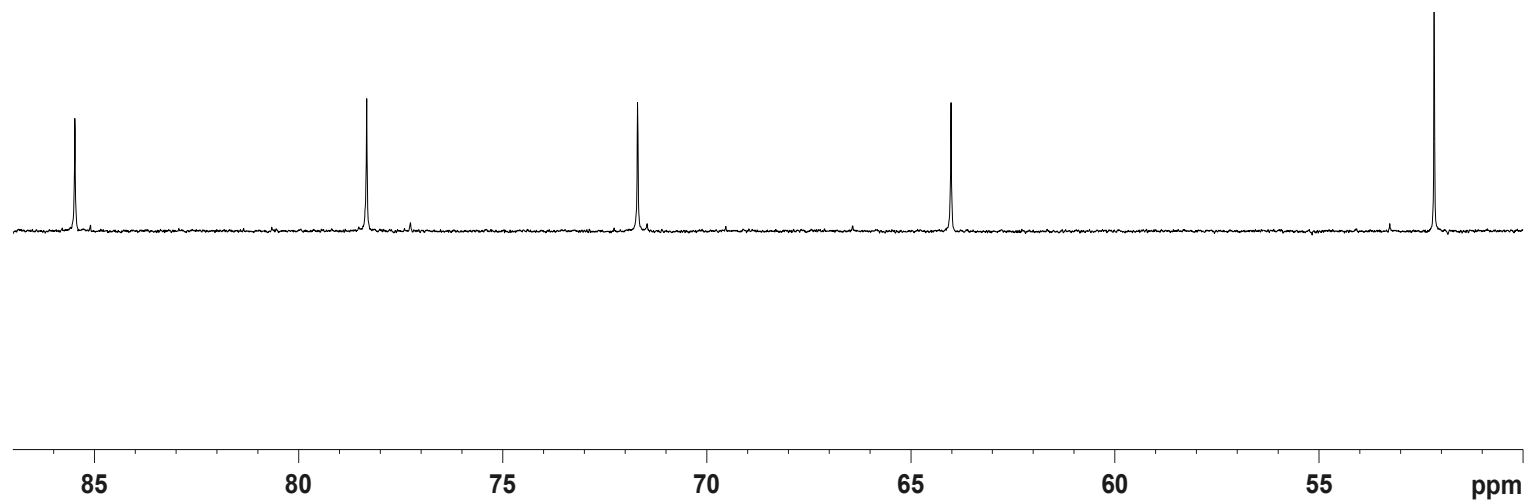
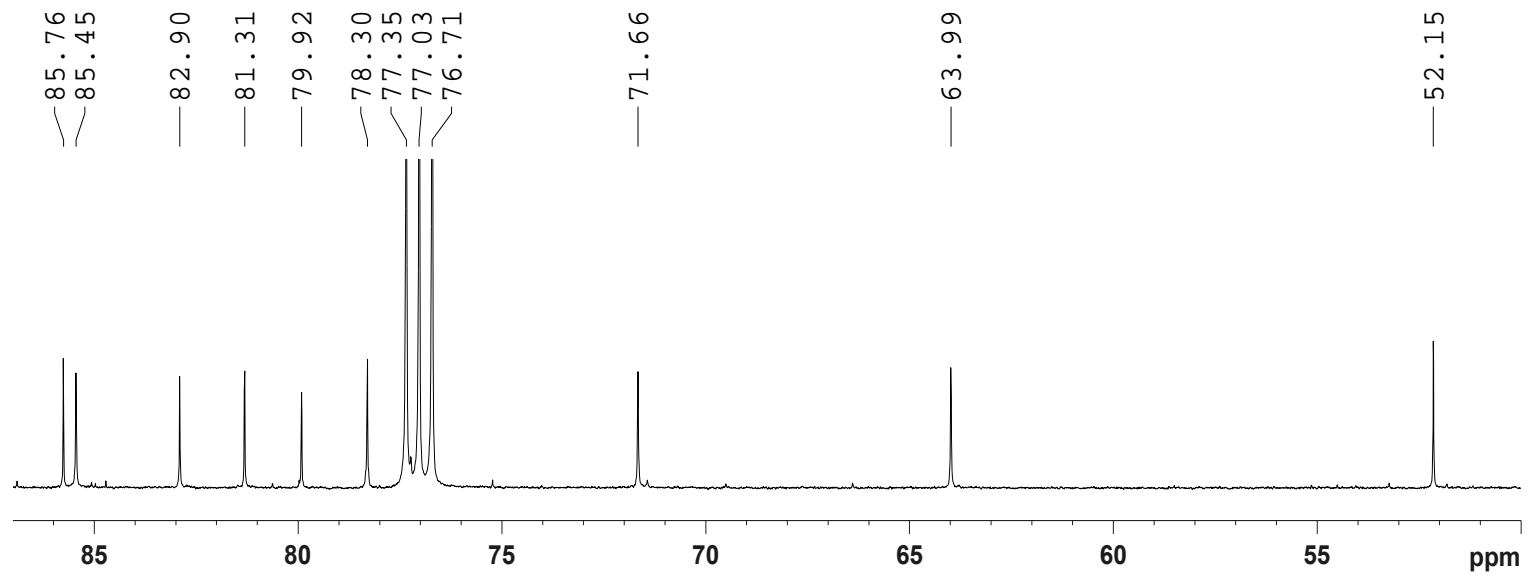


S14

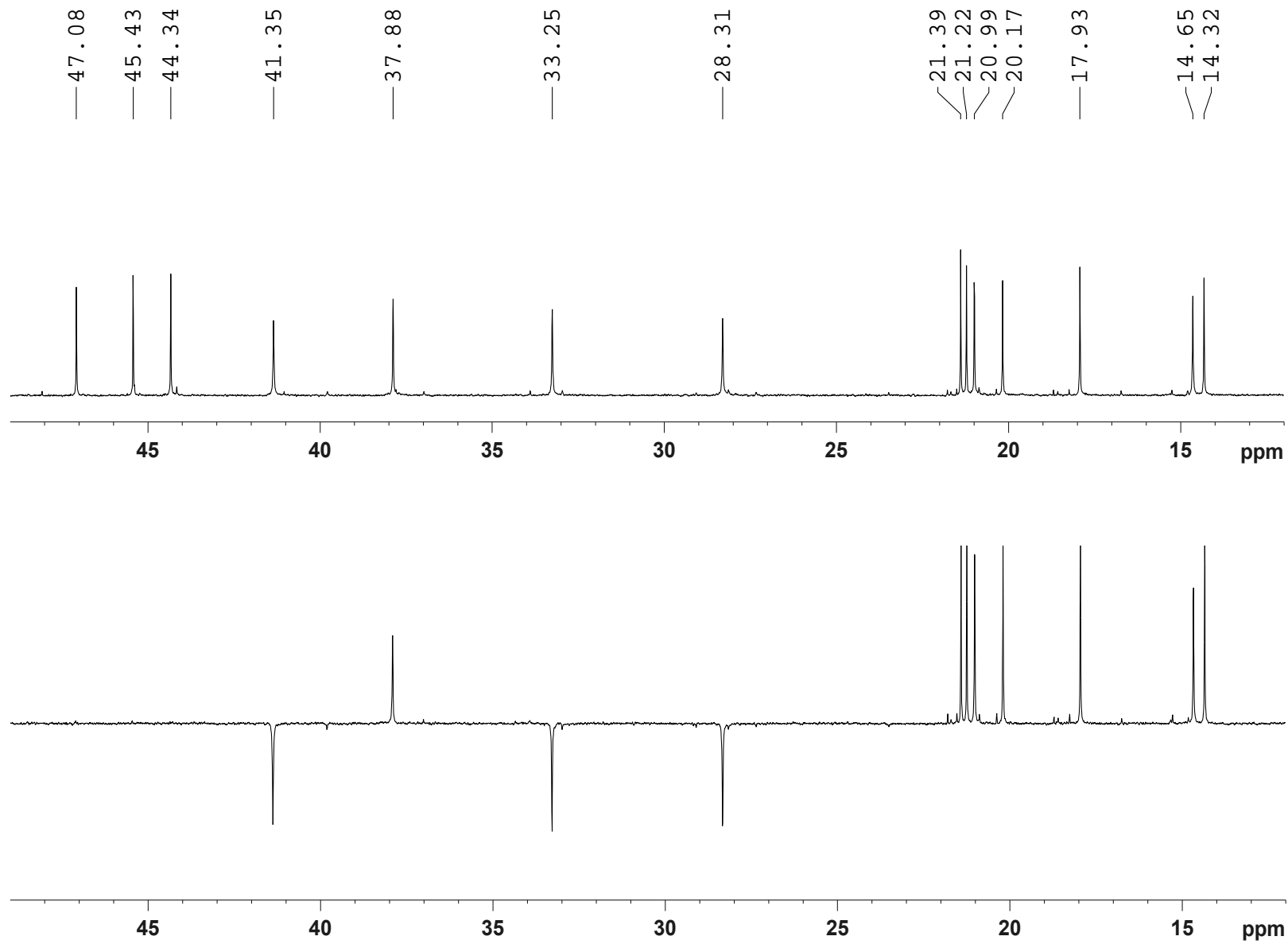
DEPT135 (100 MHz) spectrum of compound **1** in CDCl₃



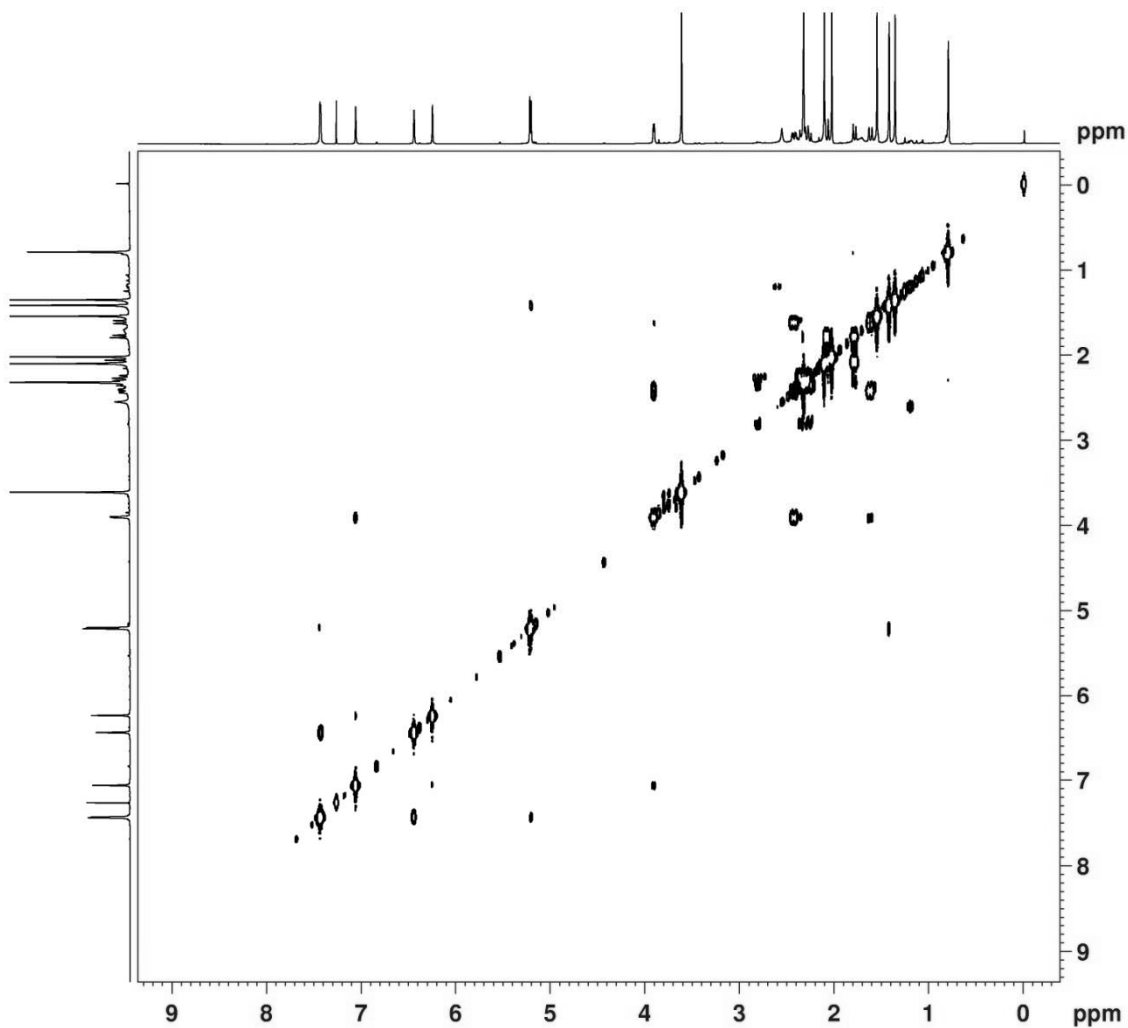
DEPT135 (100 MHz) spectrum of compound **1** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **1** in CDCl₃



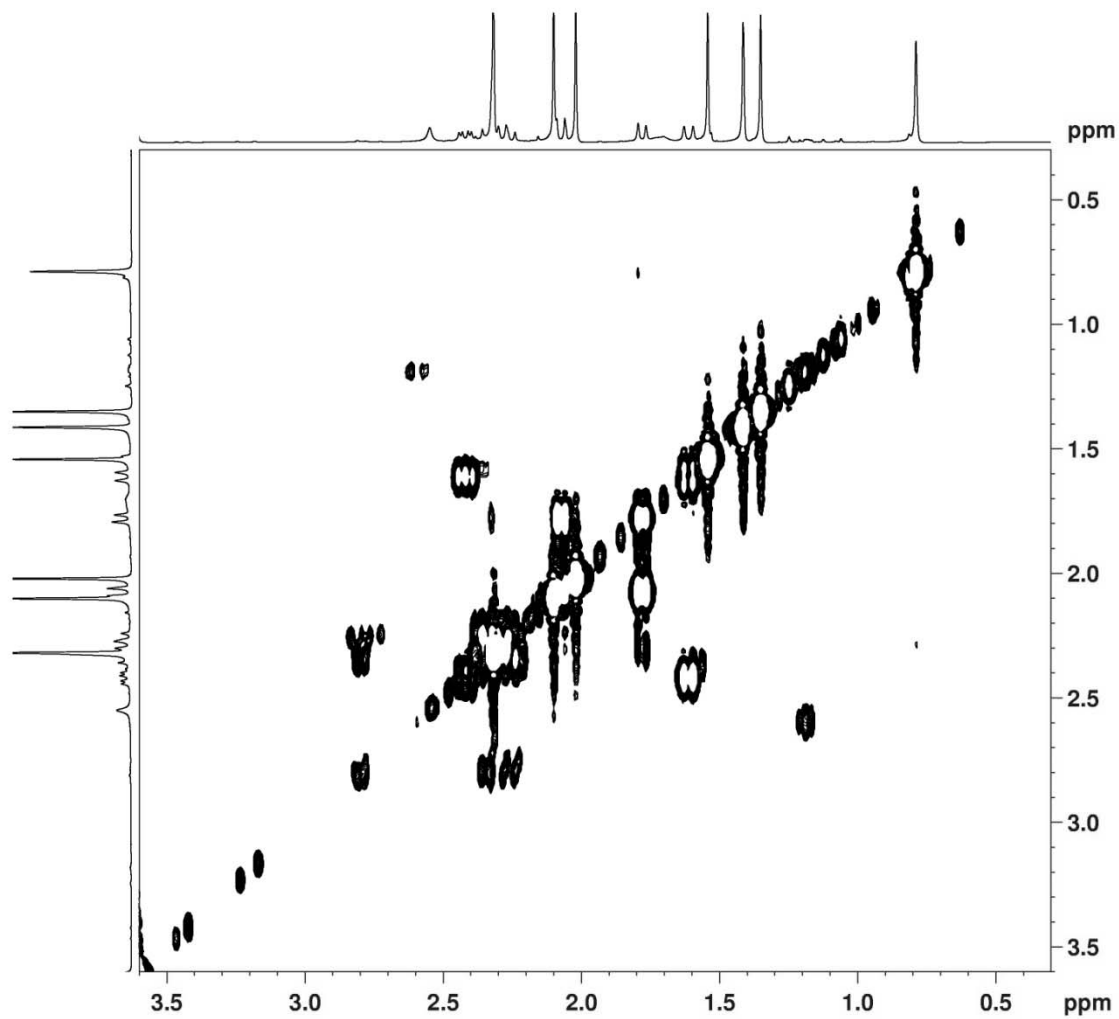
^1H - ^1H COSY (400 MHz) spectrum of compound **1** in CDCl_3



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EXPNO         104
PROCNO        1
Date_         20160208
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PULPROG       cosygpppqf
TD            2048
SOLVENT       CDCl3
NS            32
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 =====
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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
^
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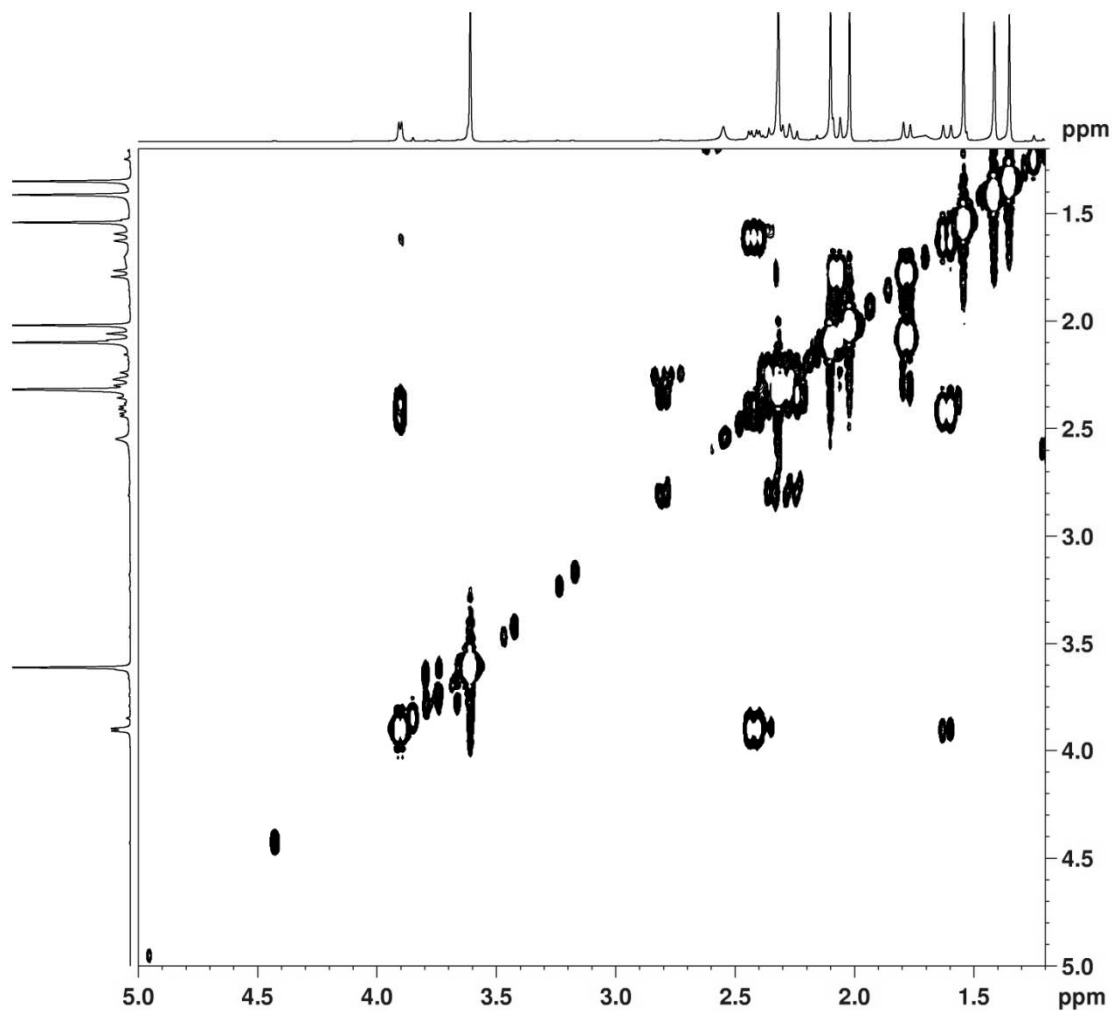
^1H - ^1H COSY (400 MHz) spectrum of compound **1** in CDCl_3



```
NAME          dyg-18
EXPNO         104
PROCNO        1
Date_         20160208
Time          1.46
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            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
FMODE         QF
SI            1024
SF            400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
```

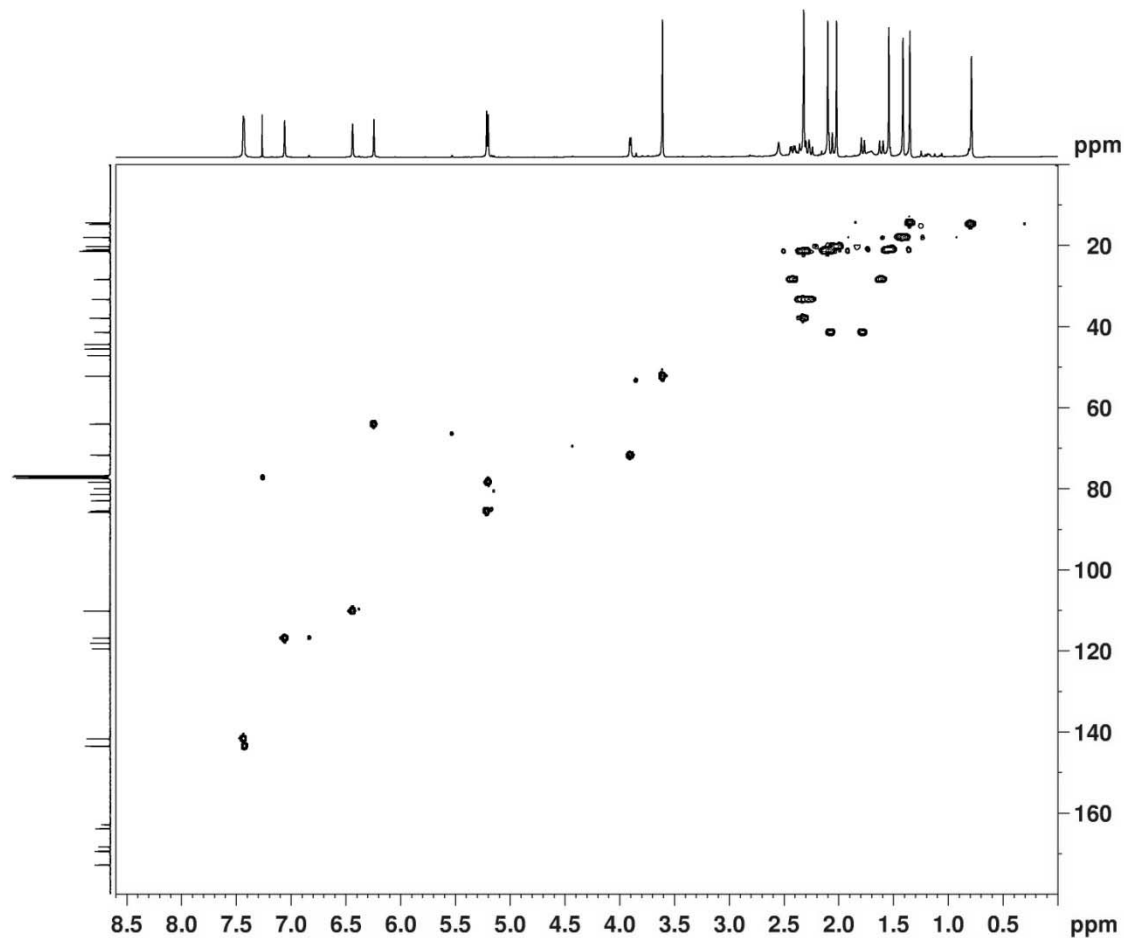
^1H - ^1H COSY (400 MHz) spectrum of compound **1** in CDCl_3



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EXPNO         104
PROCNO        1
Date_         20160208
Time_         1.46
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            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            400.1300098 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
^^
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HSQC (400 MHz) spectrum of compound **1** in CDCl₃

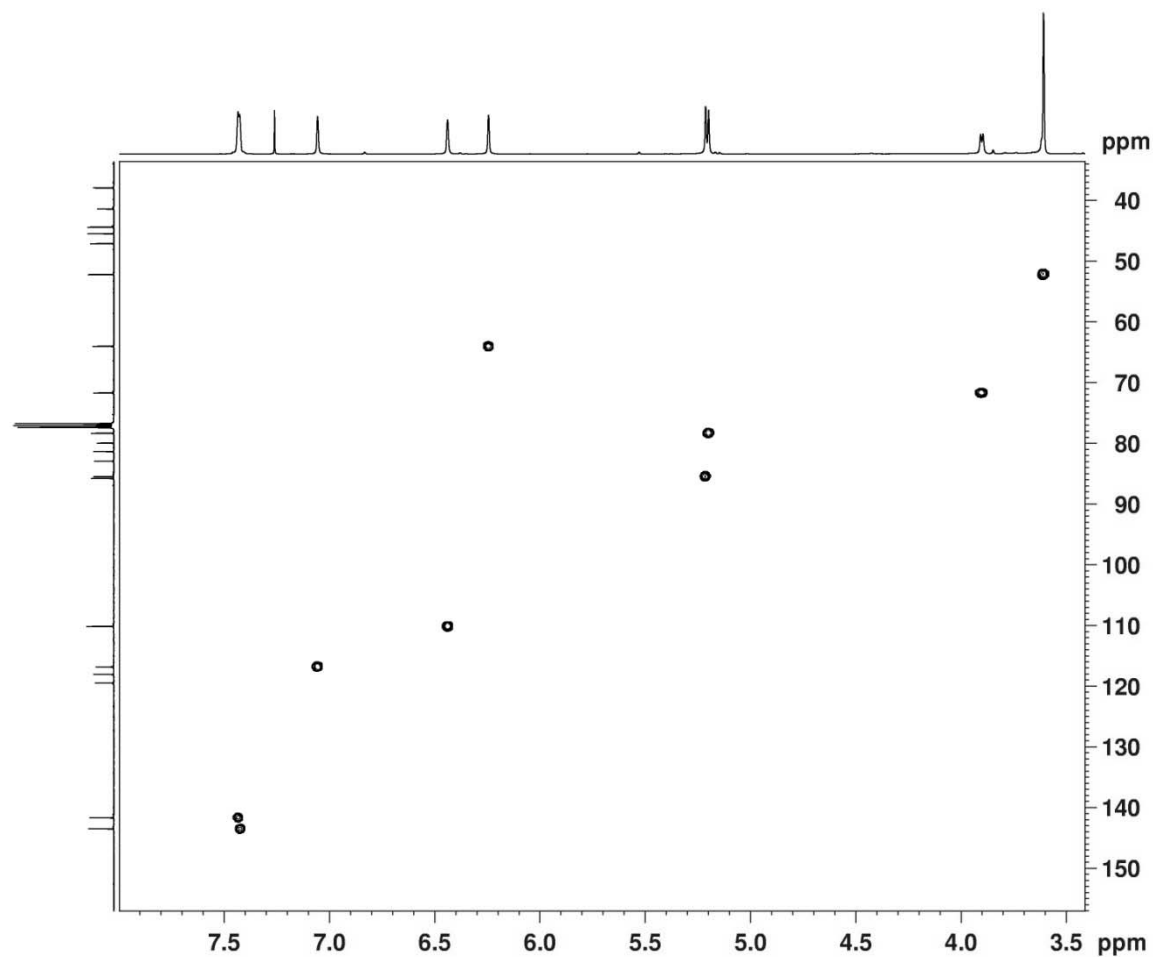


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PROCNO        1
Date_         20160208
Time          4.19
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PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS            32
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 sec
RG            208.5
DW            116.200 nsec
DE            10.00 nsec
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

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NUC1           1H
P1            11.50 nsec
P2            23.00 nsec
P28           0.00 nsec
ND0           2
TD            256
SFO1          100.6233 MHz
FIDRES        93.900238 Hz
SW            238.896 Hz
FrMODE        Echo-Antiecho
SI            1024
SF            400.1300096 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
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WDW           QSINE
  
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HSQC (400 MHz) spectrum of compound **1** in CDCl₃



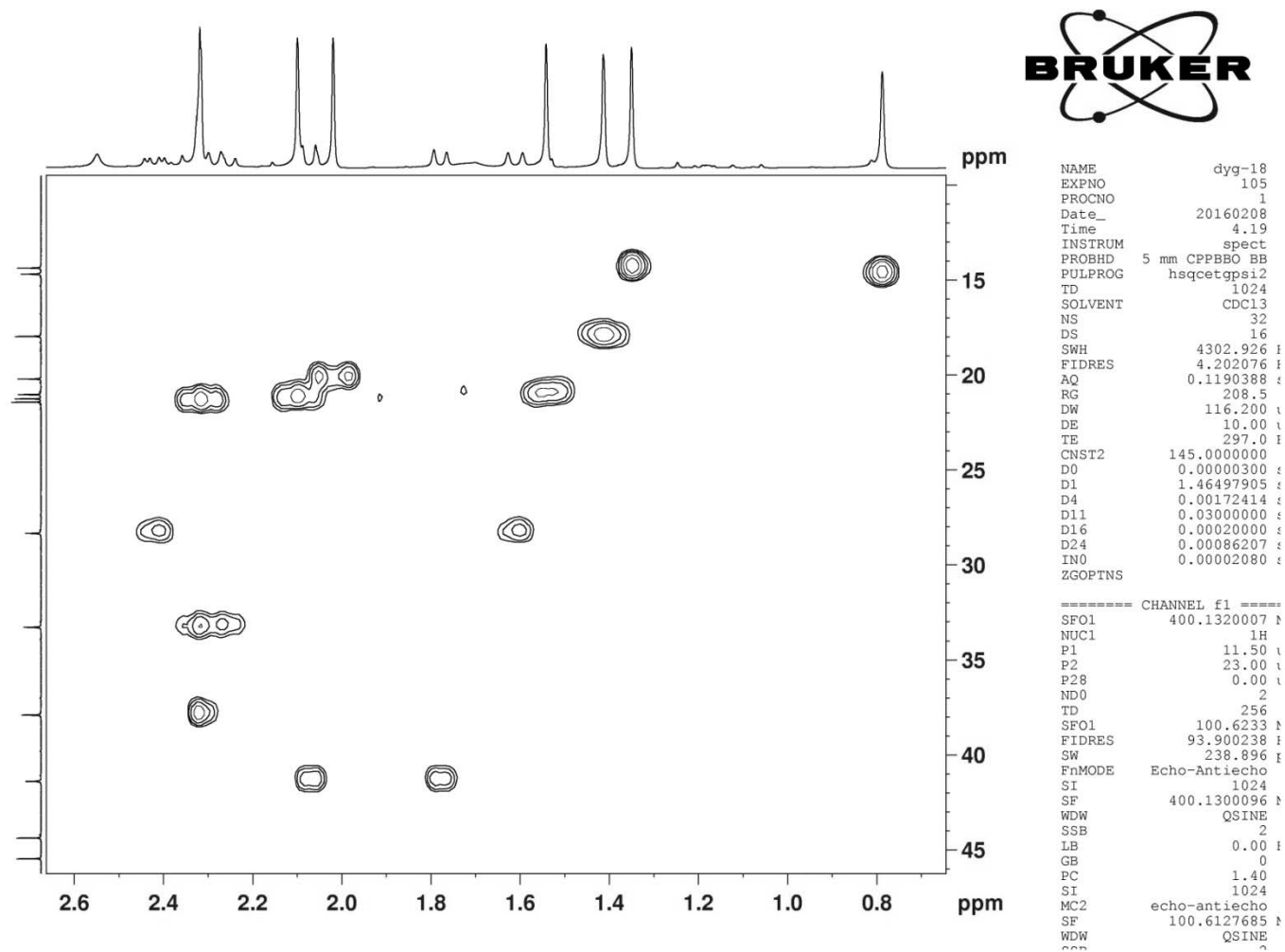
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NAME          dyg-18
EXPNO         105
PROCNO        1
Date_         20160208
Time          4.19
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS            32
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 s
RG            208.5
DW            116.200 us
DE            10.00 us
TE            297.0 K
CNST2         145.0000000
D0            0.00000300 s
D1            1.46497905 s
D4            0.00172414 s
D11           0.03000000 s
D16           0.00020000 s
D24           0.00086207 s
IN0           0.00002080 s
ZGPTNS
    
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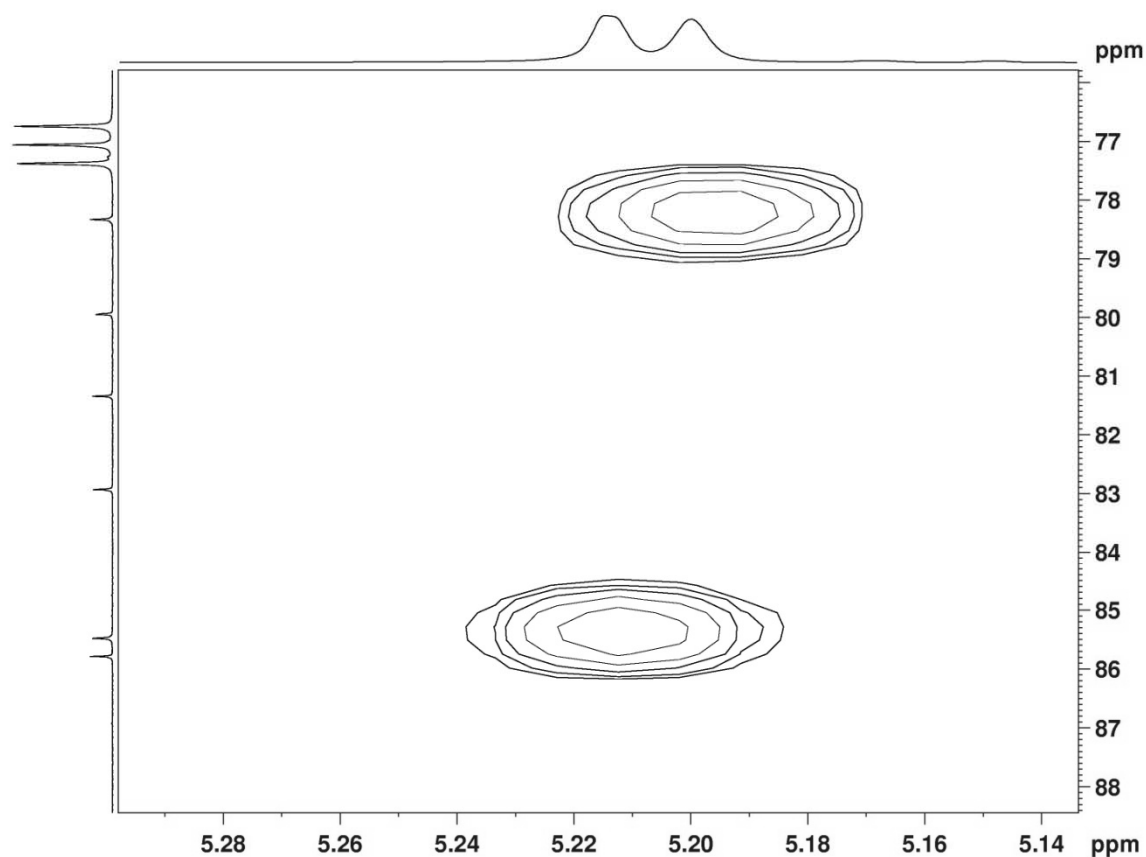
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NUC1          1H
P1            11.50 us
P2            23.00 us
P28           0.00 us
ND0           2
TD            256
SFO1         100.6233 MHz
FIDRES        93.900238 Hz
SW            238.896 Hz
FnMODE        Echo-Antiecho
SI            1024
SF            400.1300096 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            100.6127685 MHz
WDW           QSINE
    
```

HSQC (400 MHz) spectrum of compound **1** in CDCl₃



HSQC (400 MHz) spectrum of compound **1** in CDCl₃

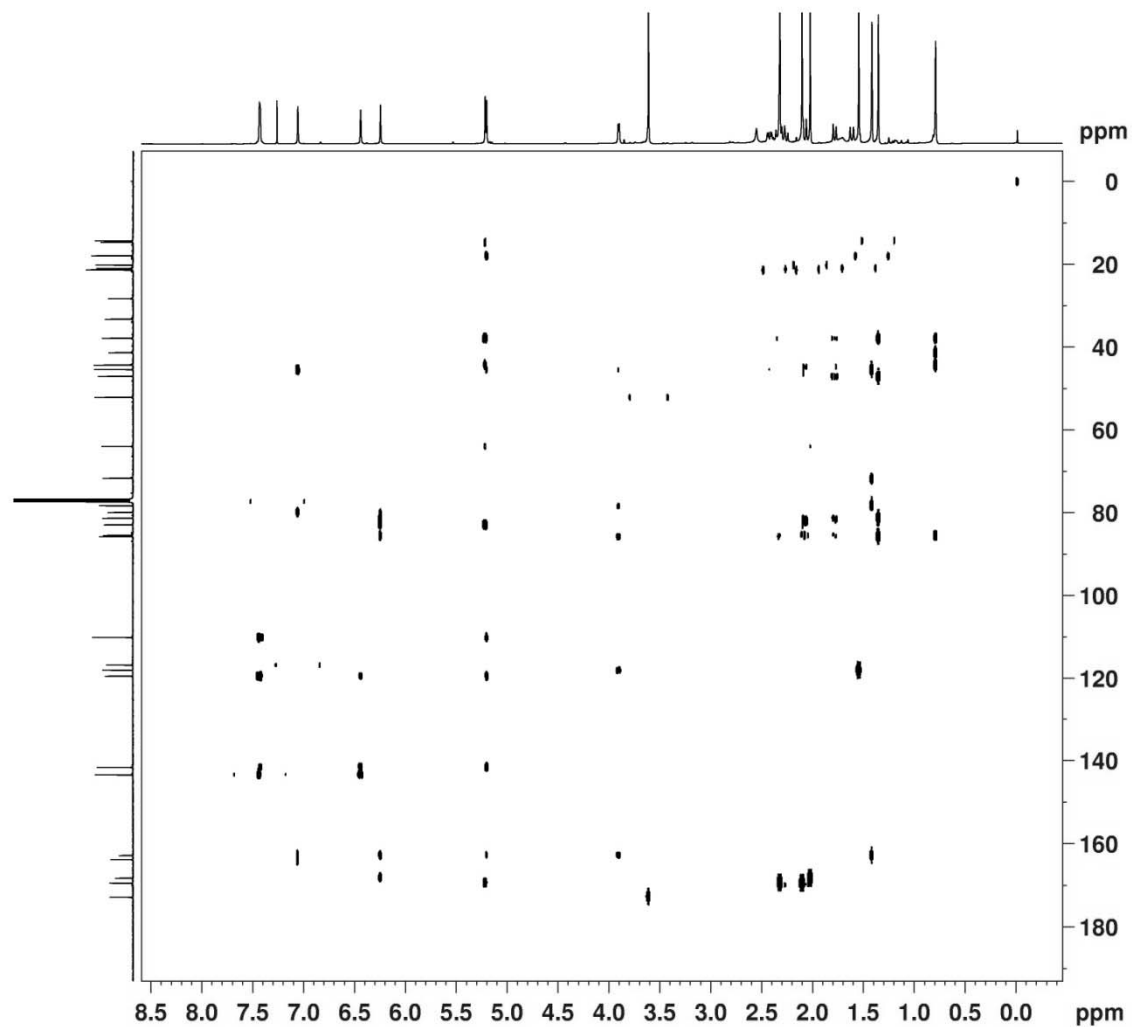


```

NAME          dyg-18
EXPNO         105
PROCNO        1
Date_         20160208
Time          4.19
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS            32
DS            16
SWH           4302.926 MHz
FIDRES        4.202076 MHz
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 =====
SF01          400.1320007 MHz
NUC1           1H
P1             11.50 usec
P2             23.00 usec
P28            0.00 usec
ND0            2
TD             256
SF01          100.6233 MHz
FIDRES        93.900238 MHz
SW             238.896 MHz
FnMODE        Echo-Antiecho
SI            1024
SF            400.1300096 MHz
WDW            QSINE
SSB            2
LB             0.00 MHz
GB             0
PC             1.40
SI            1024
MC2           echo-antiecho
SF            100.6127685 MHz
WDW            QSINE
  
```

HMBC (400 MHz) spectrum of compound **1** in CDCl₃



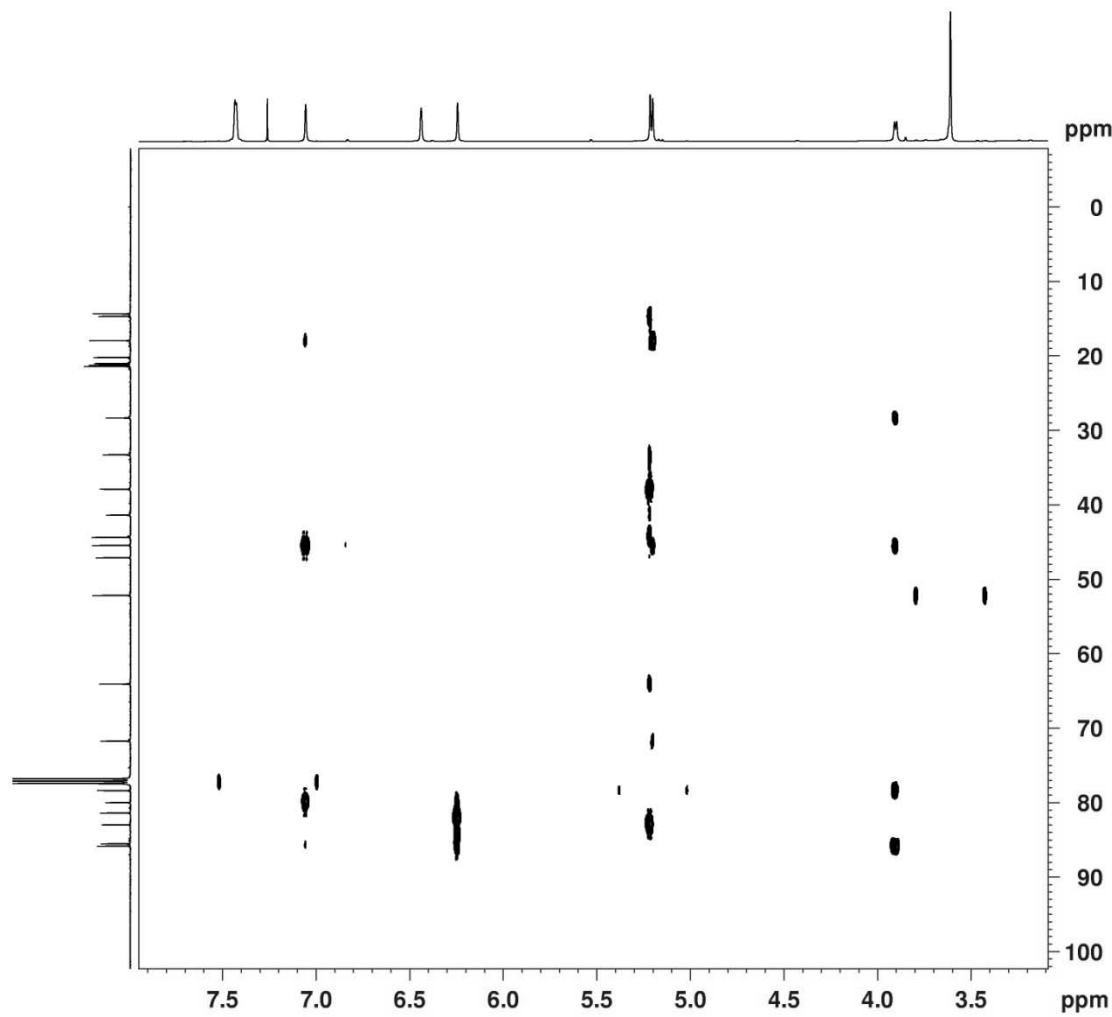
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NAME          dyg-18
EXPNO         106
PROCNO        1
Date_         20160208
Time          8.07
INSTRUM       spect
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PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            256
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.000003000 sec
D1            1.500000000 sec
D2            0.00344828 sec
D6            0.050000000 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
EnMODE        QF
SI            2048
SF            400.1300096 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            100.6127685 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
    
```

HMBC (400 MHz) spectrum of compound **1** in CDCl₃



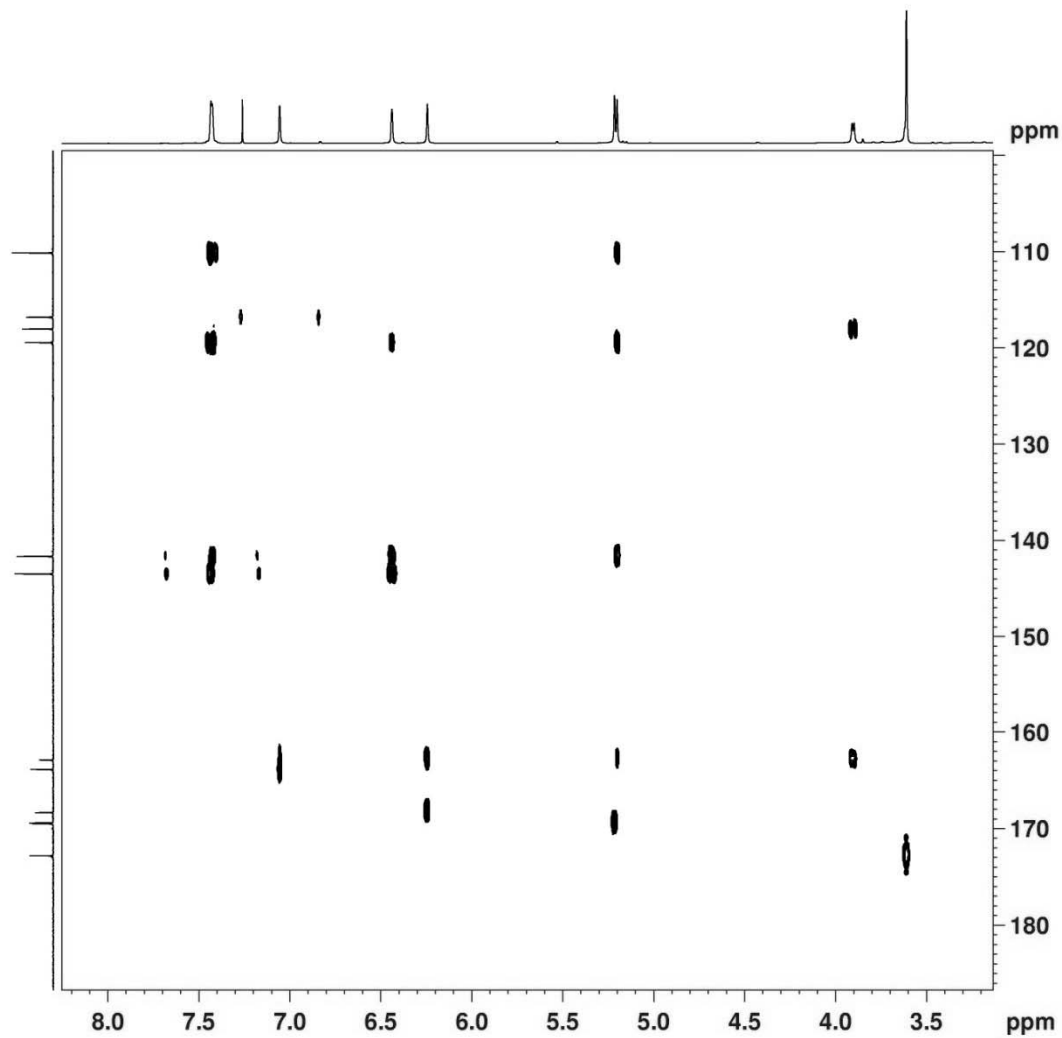
```

NAME          dyg-18
EXPNO         106
PROCNO        1
Date_         20160208
Time          8.07
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            256
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

```

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

HMBC (400 MHz) spectrum of compound **1** in CDCl₃



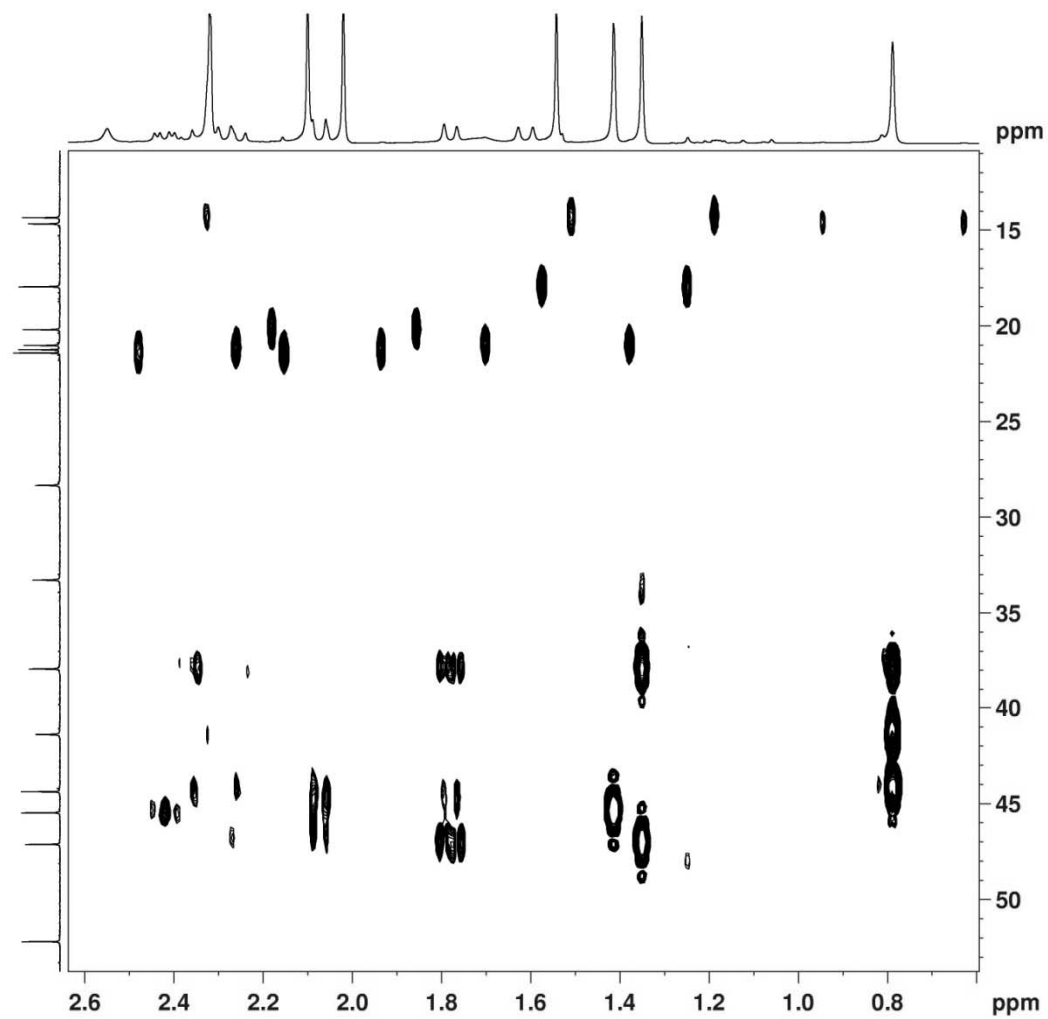
```

NAME          dyg-18
EXPNO         106
PROCNO        1
Date_         20160208
Time          8.07
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            256
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

```

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

HMBC (400 MHz) spectrum of compound **1** in CDCl₃

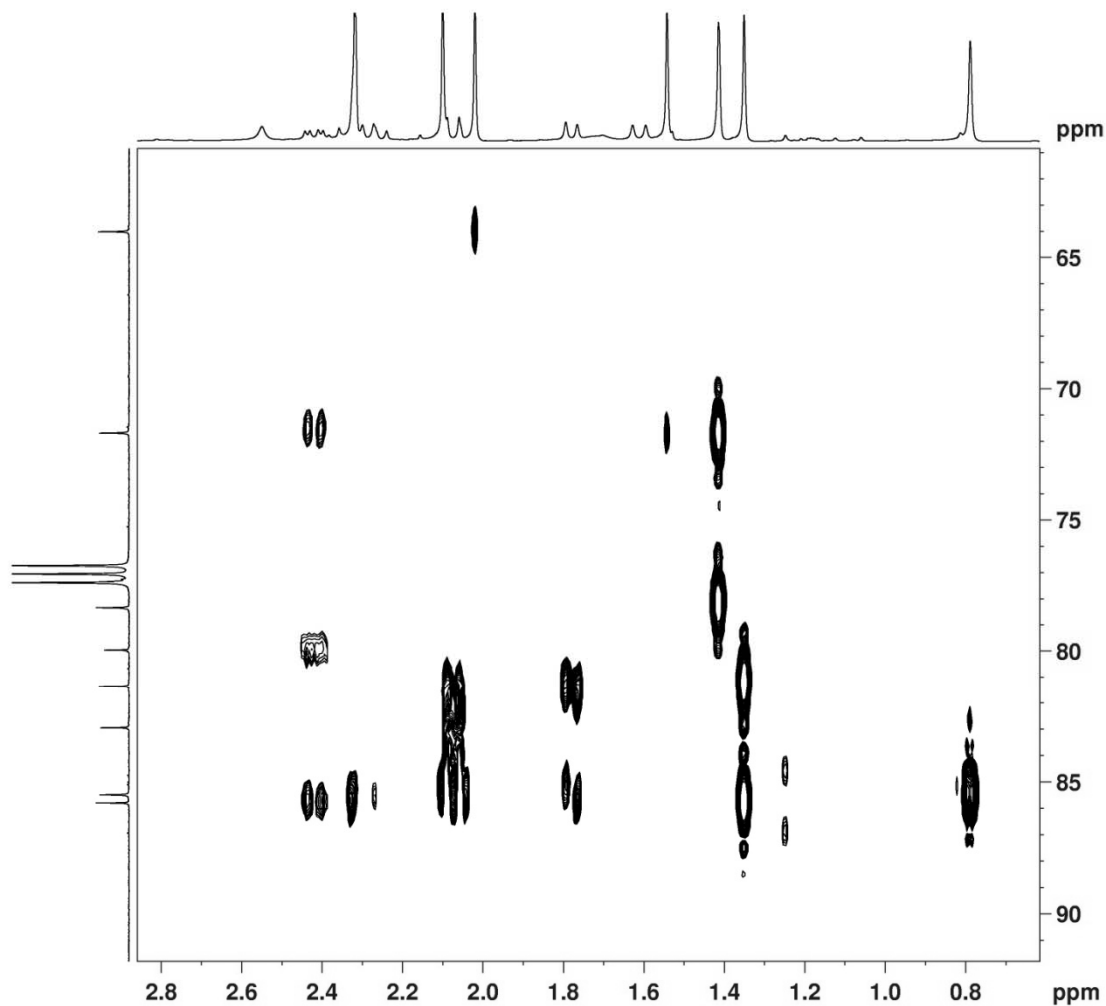


```

NAME          dyg-18
EXPNO         106
PROCNO        1
Date_         20160208
Time          8.07
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            256
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec

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


HMBC (400 MHz) spectrum of compound **1** in CDCl₃



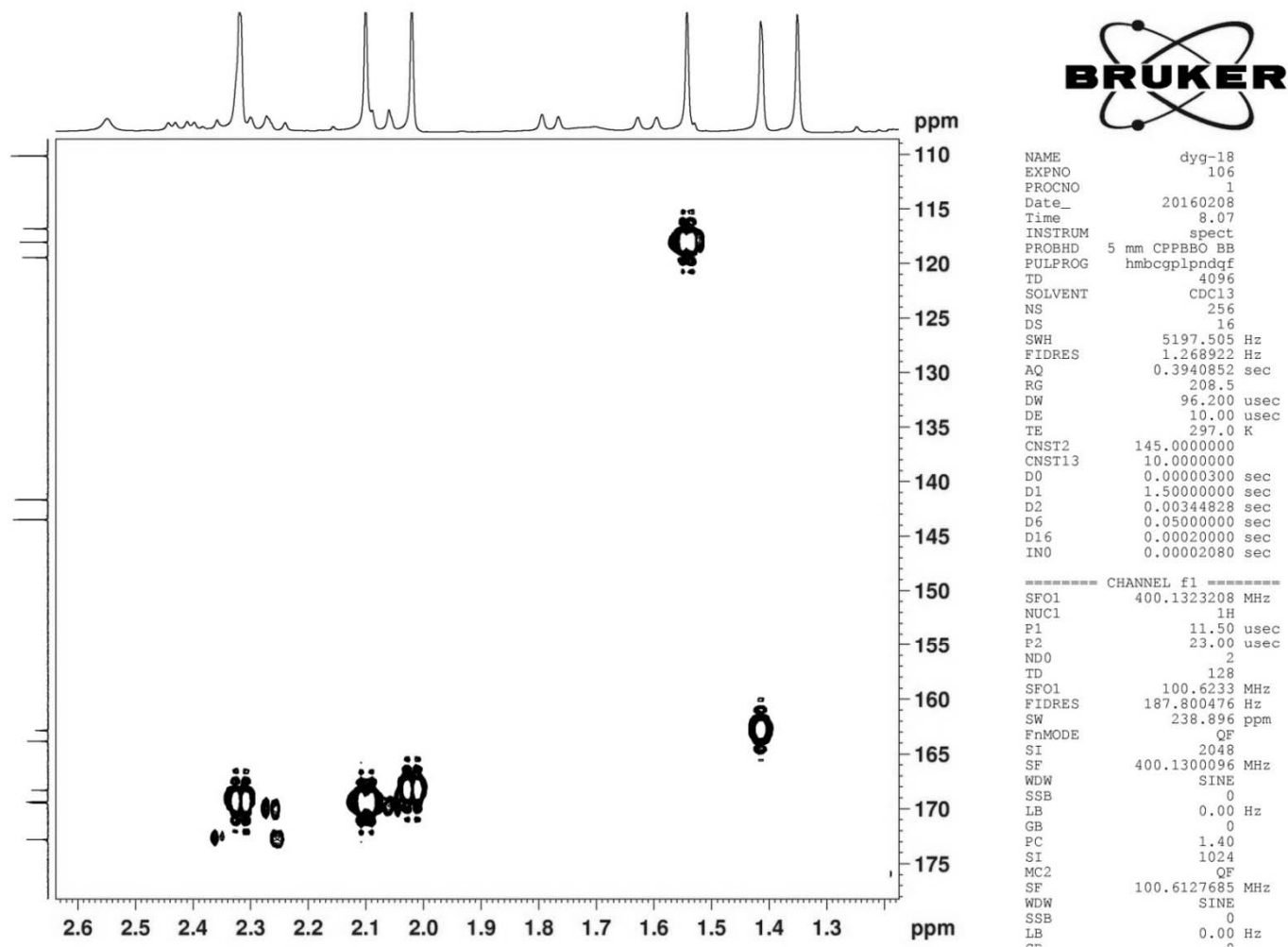
```

NAME          dyg-18
EXPNO         106
PROCNO        1
Date_         20160208
Time          8.07
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            256
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

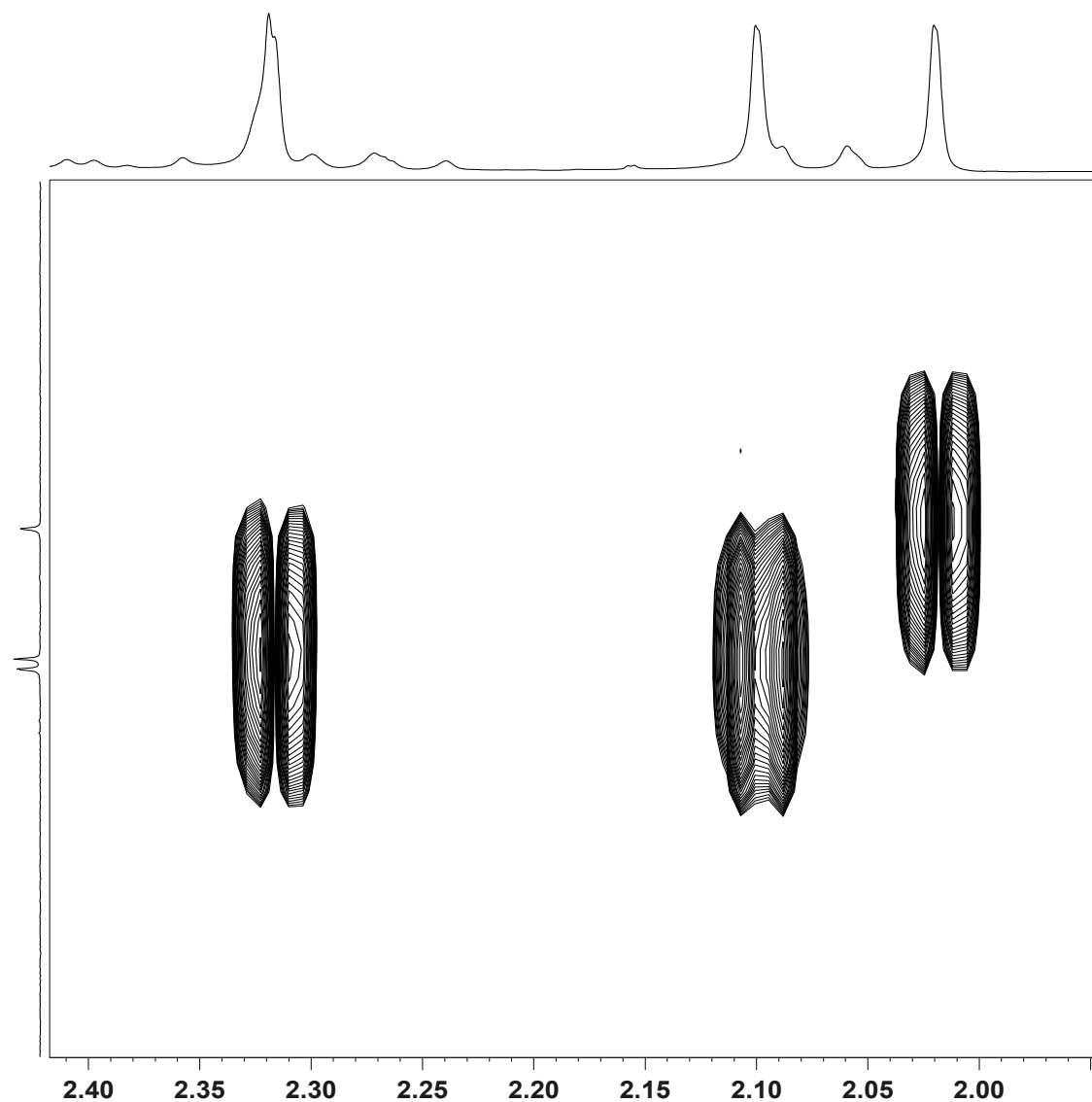
```

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

HMBC (400 MHz) spectrum of compound **1** in CDCl₃

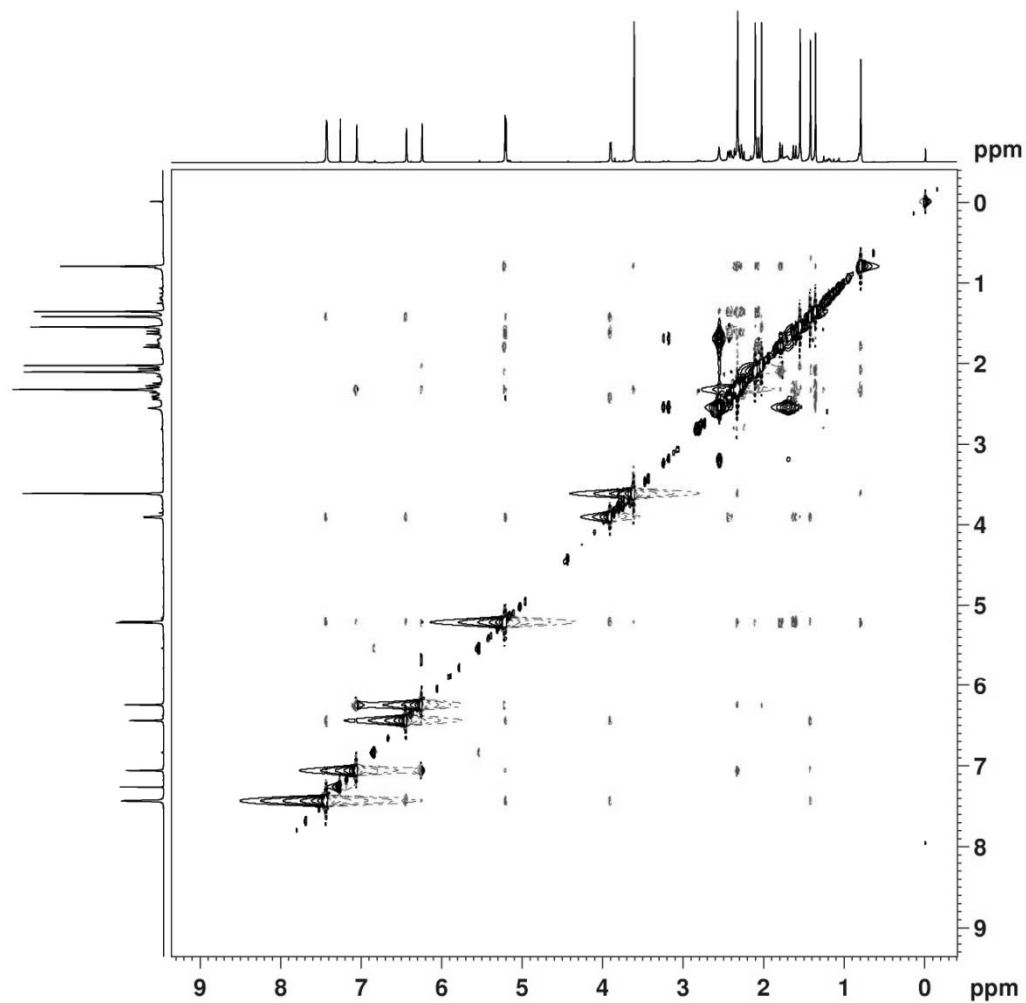


HMBC (400 MHz) spectrum of compound **1** in CDCl₃



NAME	dyg-18
EXPNO	106
PROCNO	1
Date_	20160208
Time	8.07
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	hmbcgp1pndgf
TD	4096
166.0 SOLVENT	CDCl3
NS	256
DS	16
167.0 SWH	5197.505 Hz
FIDRES	1.268922 Hz
AQ	0.3940852 sec
RG	208.5
167.5 DW	96.200 usec
DE	10.00 usec
TE	297.0 K
168.0 CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	1.50000000 sec
168.5 D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
169.0 IN0	0.00002080 sec
===== CHANNEL f1 =====	
SFO1	400.1323208 MHz
169.5 NUC1	1H
P1	11.50 usec
P2	23.00 usec
170.0 ND0	2
TD	128
SFO1	100.6233 MHz
FIDRES	187.800476 Hz
170.5 SW	238.896 ppm
FnMODE	QF
SI	2048
171.0 SF	400.1300096 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
171.5 GB	0
PC	1.40
SI	1024
172.0 MC2	QF
SF	100.6127685 MHz
WDW	SINE
SSB	0
172.5 LB	0.00 Hz
GB	0

NOESY (400 MHz) spectrum of compound **1** in CDCl₃



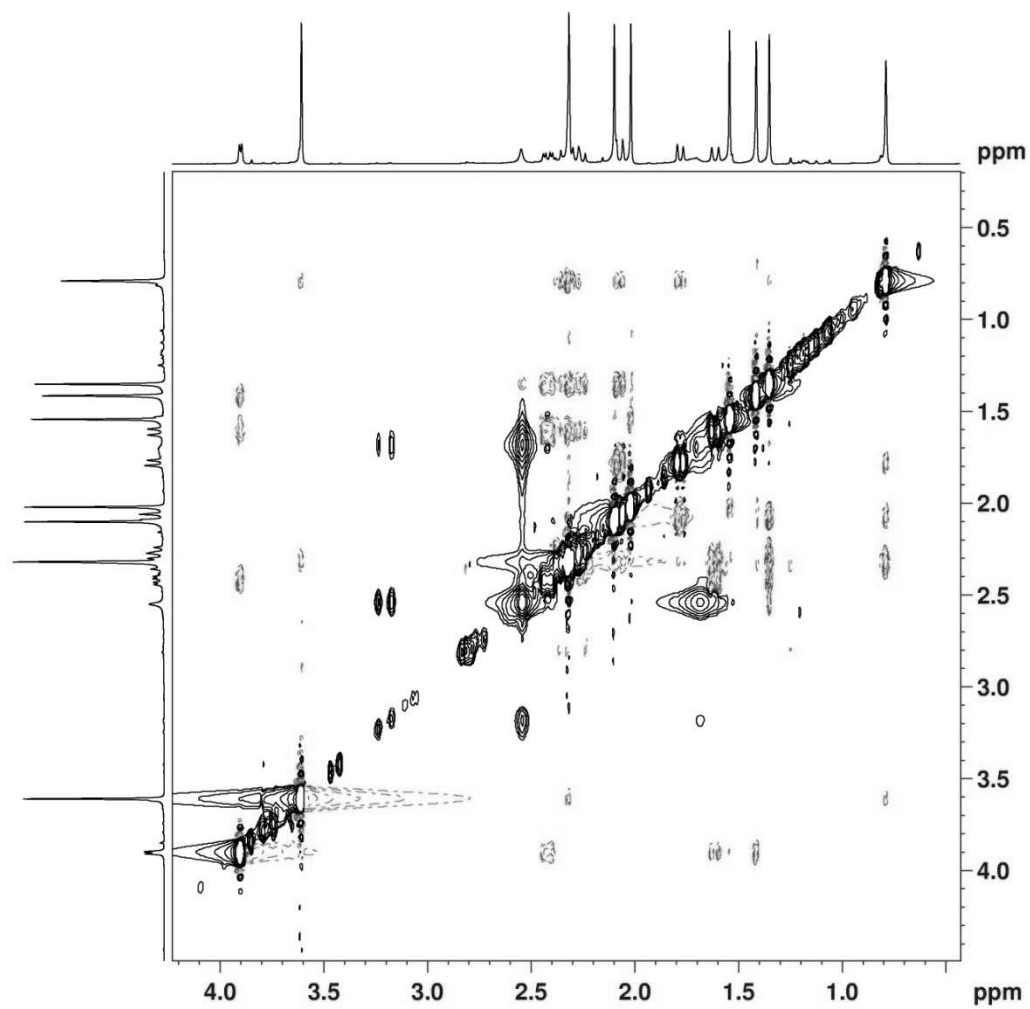
```

NAME          dyg-18
EXPNO         107
PROCNO        1
Date_         20160209
Time          1.51
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 sec
RG            91.64
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00020000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

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

NOESY (400 MHz) spectrum of compound **1** in CDCl₃



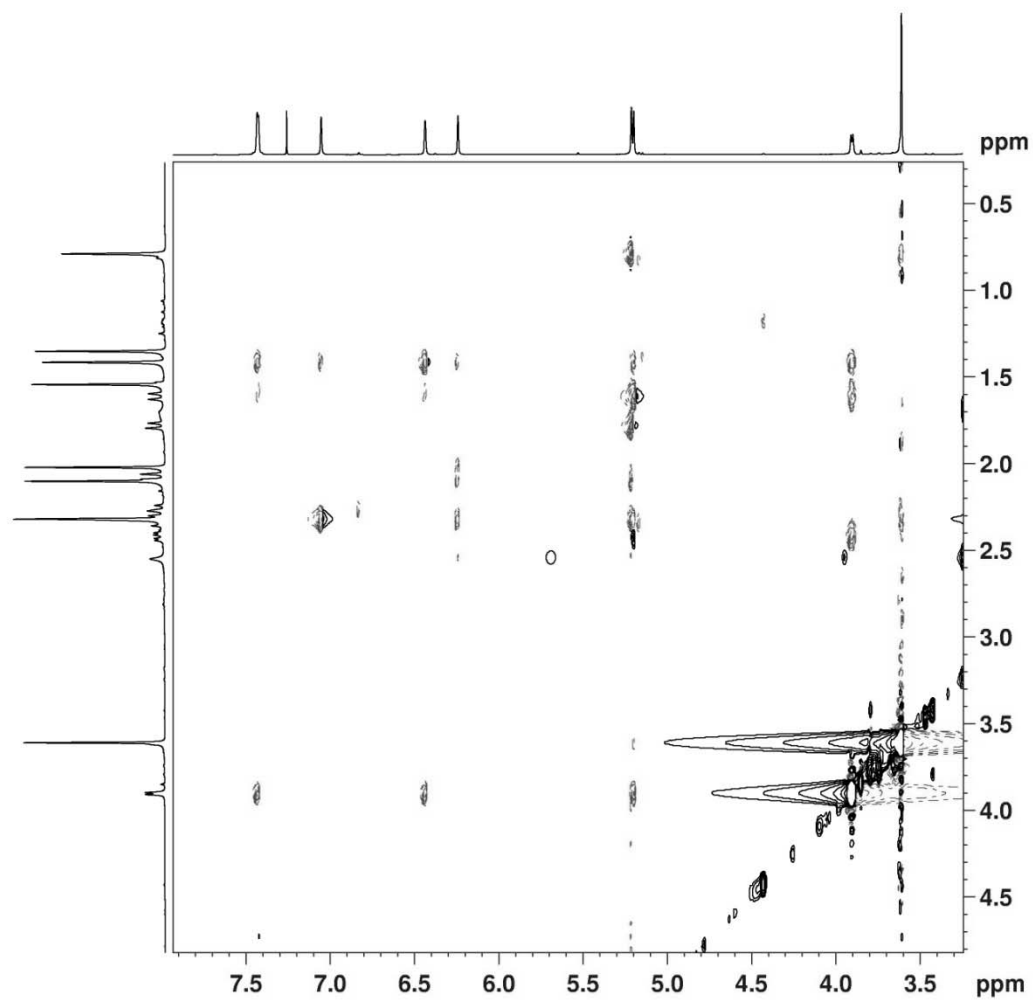
```

NAME          dyg-18
EXPNO         107
PROCNO        1
Date_         20160209
Time          1.51
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpqhpp
TD            2048
SOLVENT       CDCl3
NS            32
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            91.64
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

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

NOESY (400 MHz) spectrum of compound **1** in CDCl₃



```
NAME          dyg-18
EXPNO         107
PROCNO        1
Date_         20160209
Time          1.51
INSTRUM       spect
PROBHD        5 mm CPEBBO BB
PULPROG       noesygpphpp
TD            2048
SOLVENT       CDCl3
NS            32
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            91.64
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
```

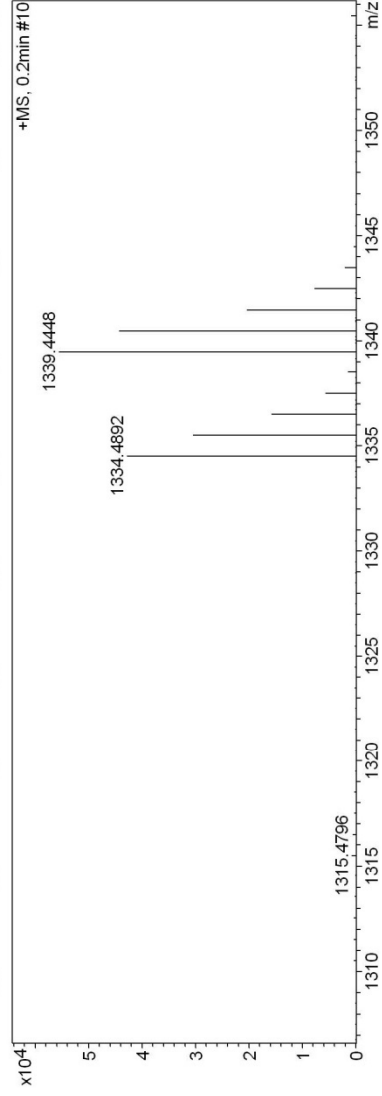
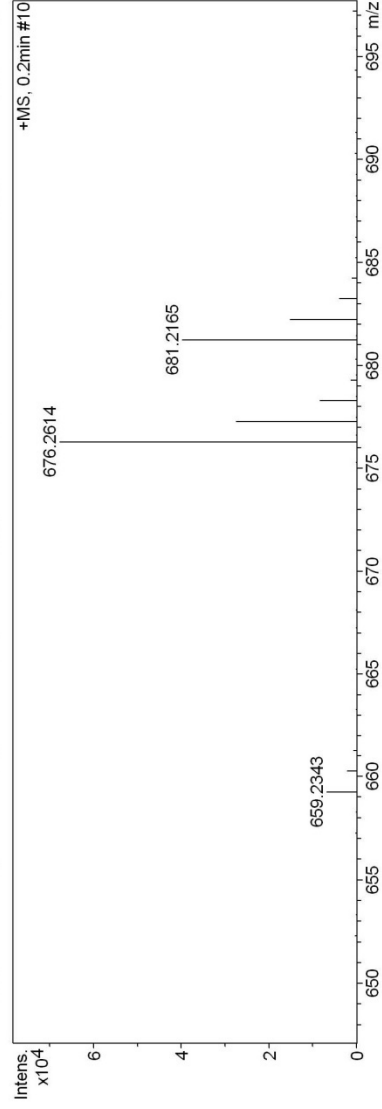
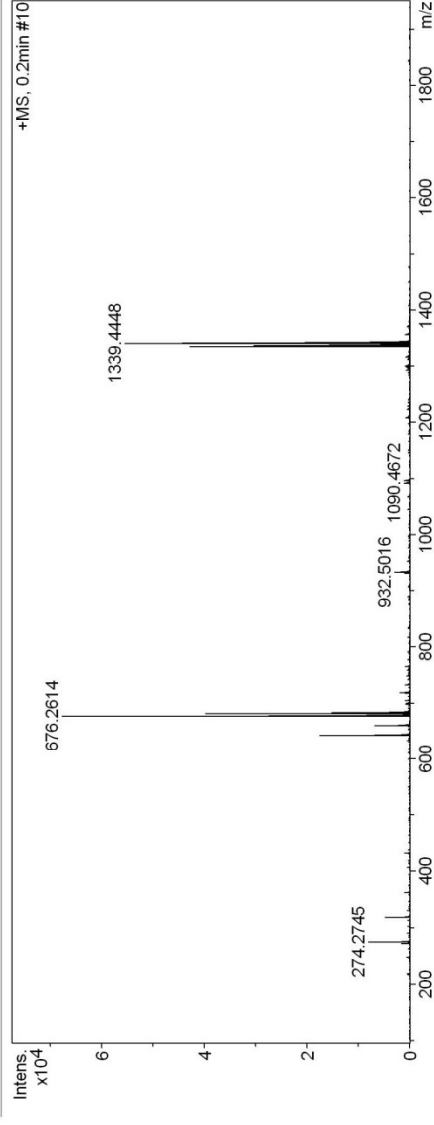
```
----- CHANNEL f1 -----
SFO1          400.1318006 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
P17           2500.00 usec
ND0           1
TD            256
SFO1          400.1318 MHz
FIDRES        15.625000 Hz
SW            9.997 ppm
FnMODE        States-TPPI
SI         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

HR-ESIMS for compound 2

Generic Display Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-3_pos_1_01_1849.d
Method LC_Direct Infusion_pos_100-1000mz.m
Sample Name liwanshan_dgy-3_pos
Comment
Acquisition Date 5/19/2016 8:20:45 AM
Operator SCSIO
Instrument maXis



S35

HR-ESIMS for compound 2

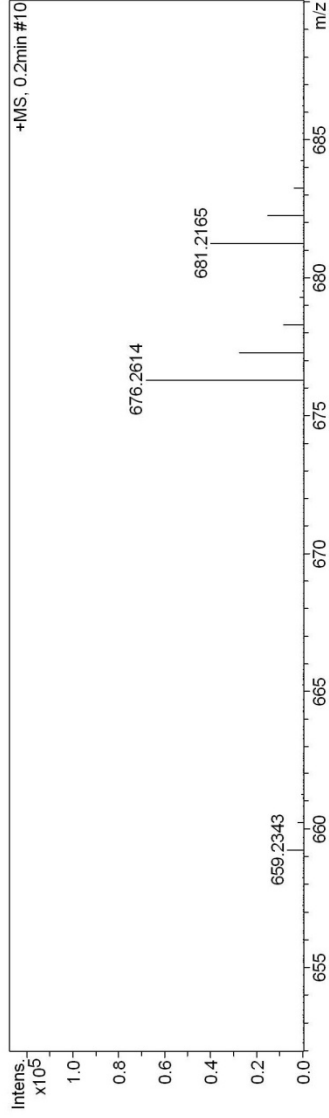
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-3_pos_1_01_1849.d Acquisition Date 5/19/2016 8:20:45 AM
 Method LC_Direct Infusion_pos_100-1000mz.m Operator SCSIO
 Sample Name liwanshan_dgy-3_pos Instrument / Ser# maXis 29
 Comment

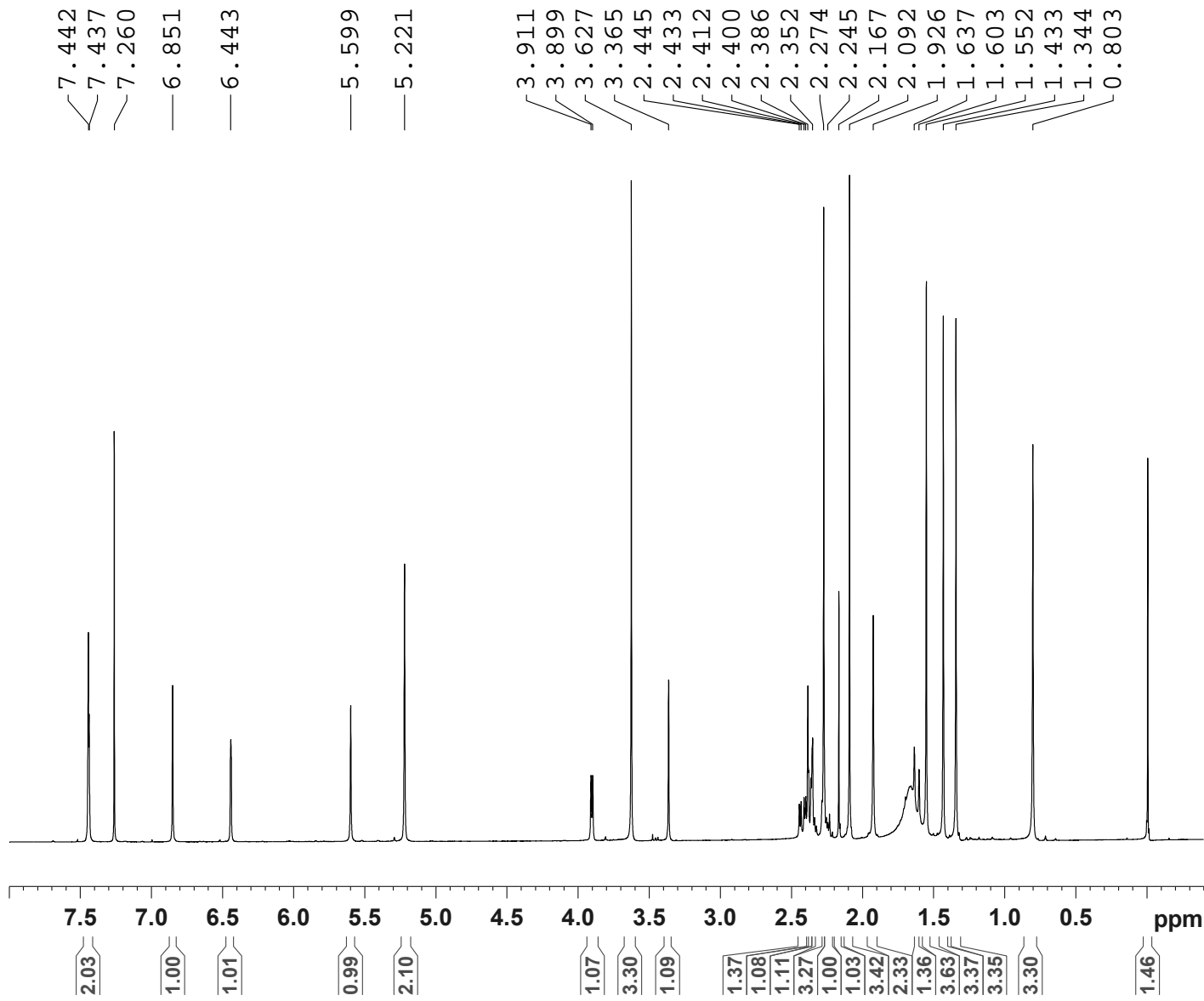
Acquisition Parameter

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



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e ⁻ Conf	N-Rule
659.2343	1	C 33 H 39 O 14	100.00	659.2334	-1.3	-0.9	24.6	14.5	even	ok
681.2165	1	C 33 H 38 Na O 14	100.00	681.2154	-1.6	-1.1	8.6	14.5	even	ok
1339.4448	1	C 66 H 76 Na O 28	10.95	1339.4415	-2.4	-3.2	38.2	28.5	even	ok

¹H NMR (400 MHz) spectrum of compound **2** in CDCl₃



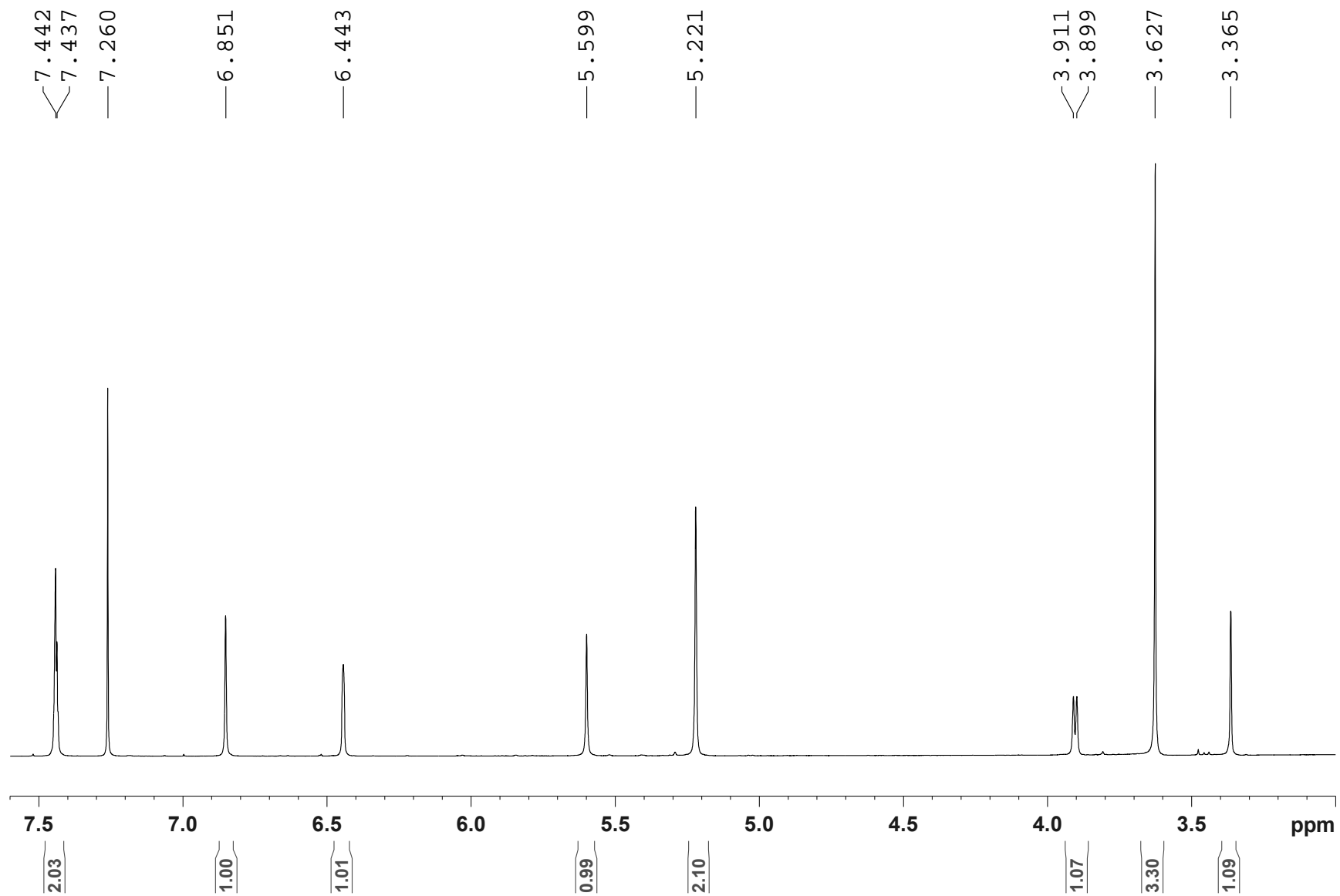
```

NAME          dyg-3
EXPNO         1
PROCNO        1
Date_         20150625
Time          17.42
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 =====
SF01          400.1324710 MHz
NUC1           1H
P1             12.00 usec
SI             65536
SF             400.1300094 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
    
```

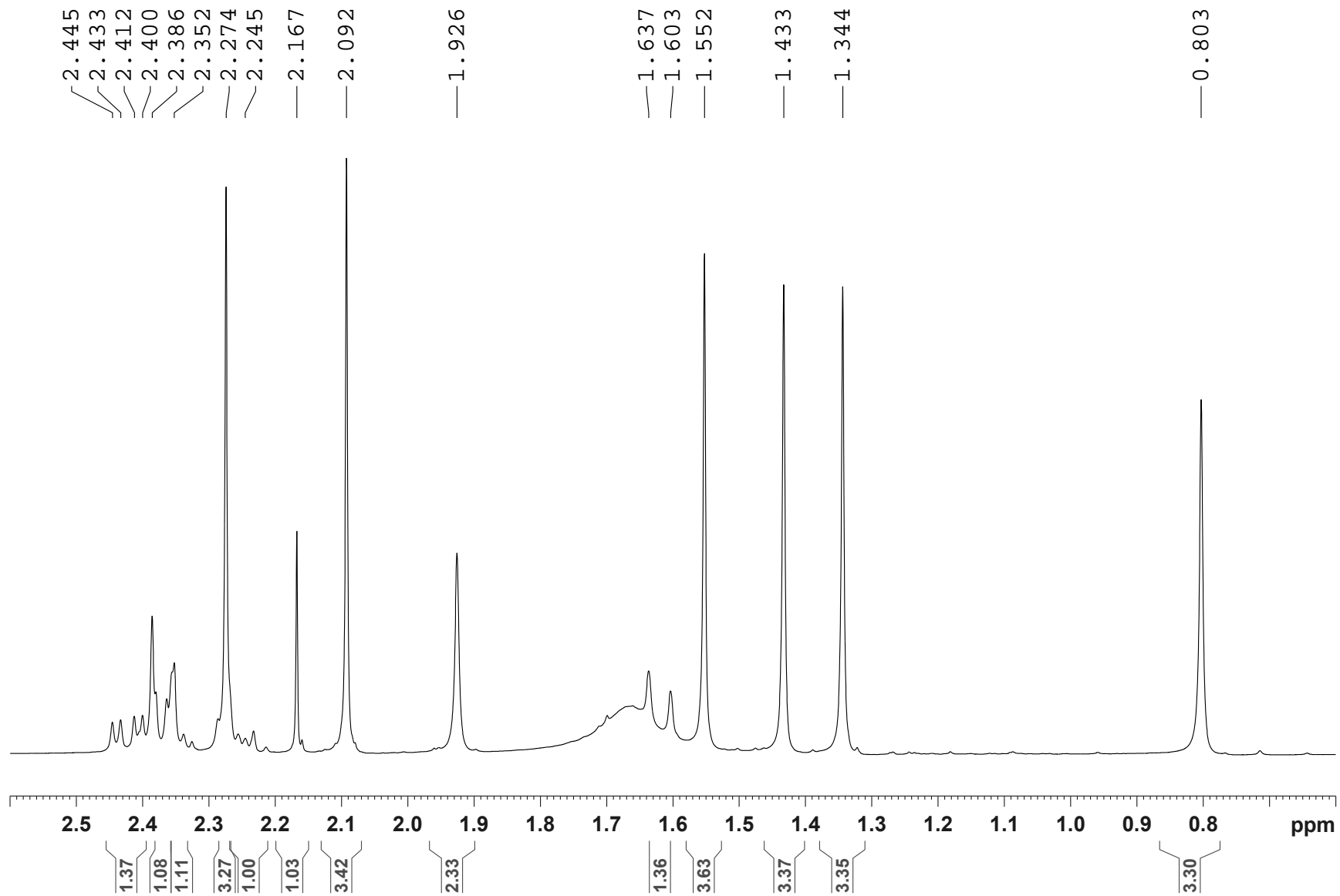
S37

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

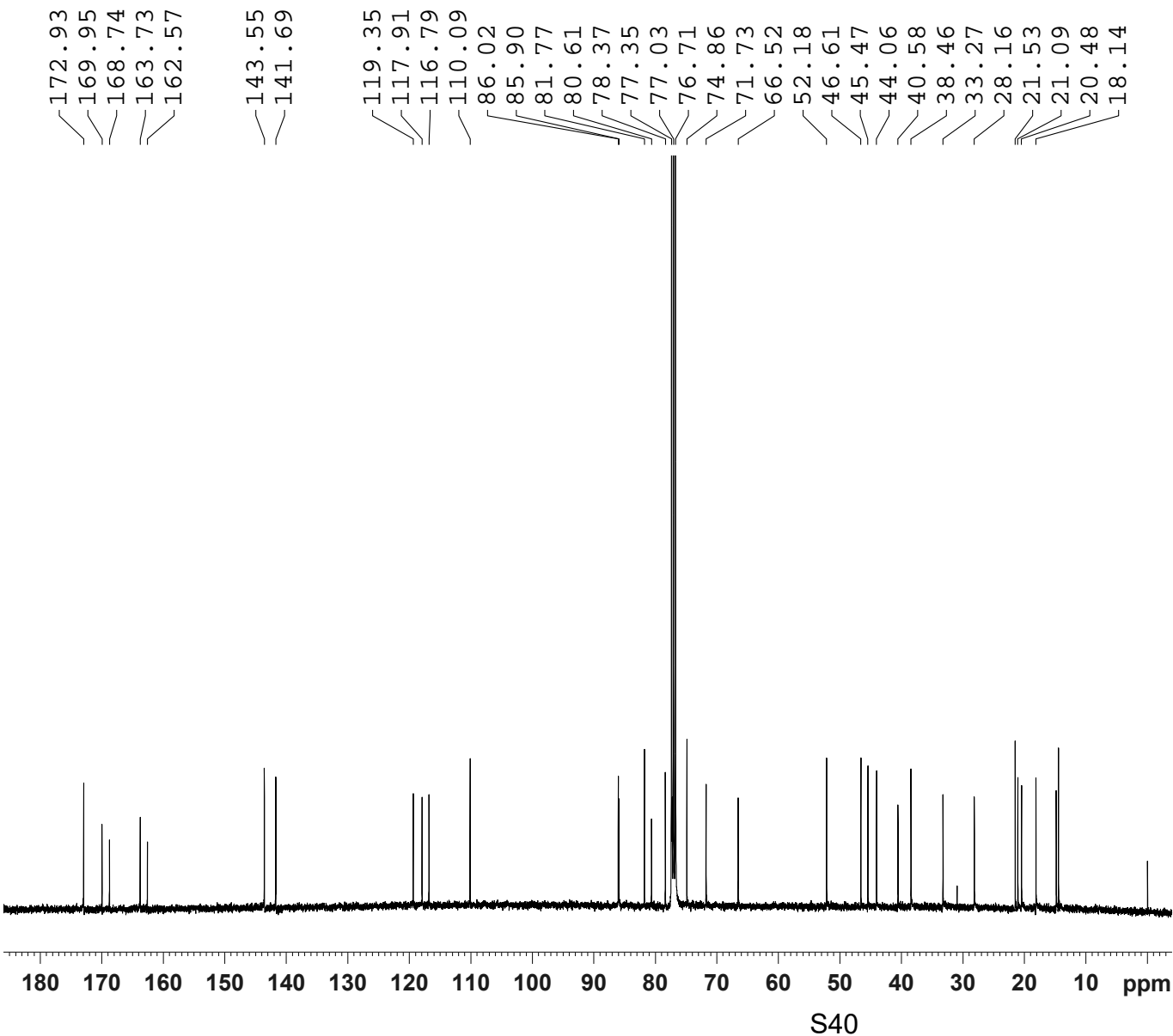


S38

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



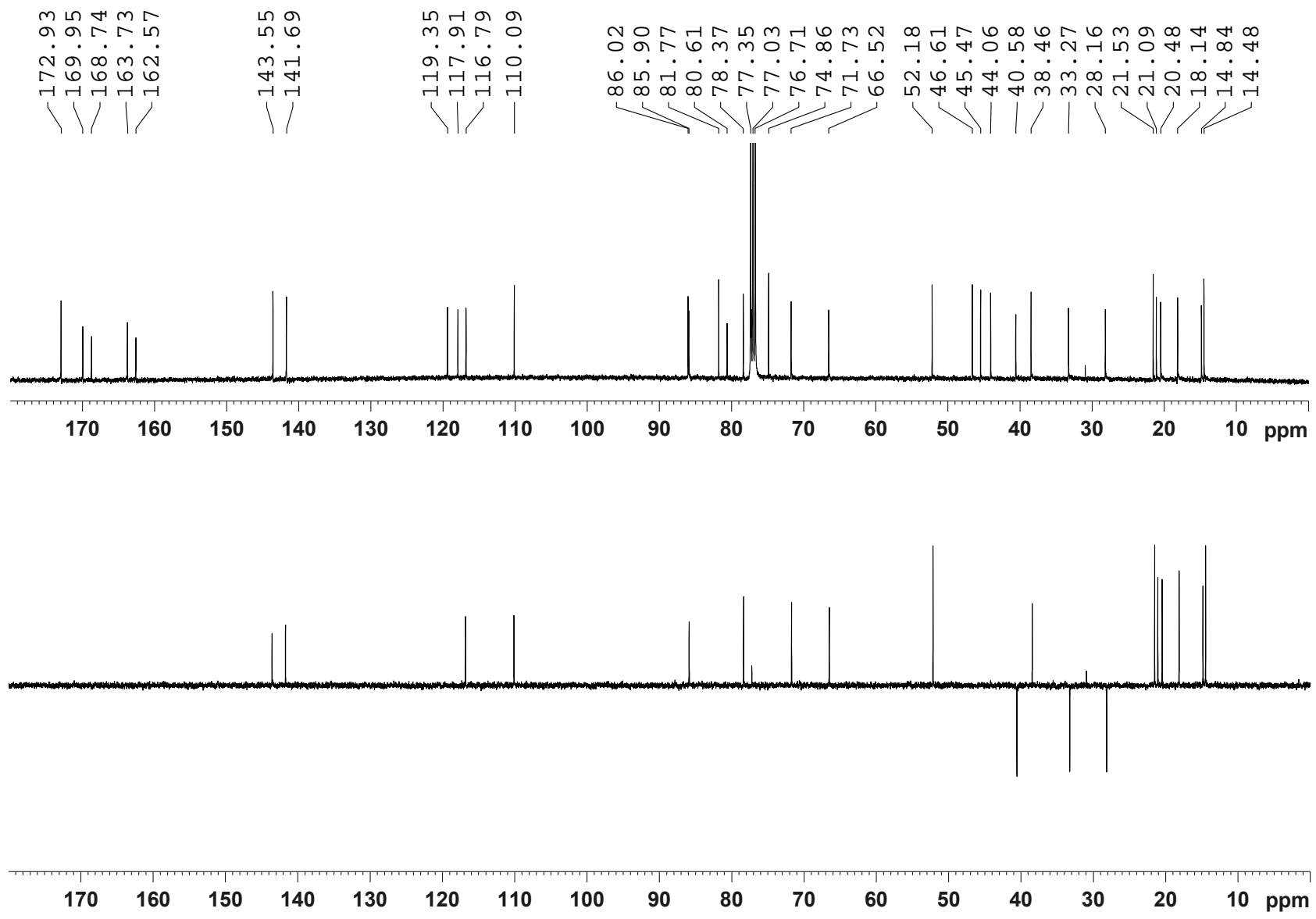
¹³C NMR (100 MHz) spectrum of compound **2** in CDCl₃



```
NAME          dyg-3
EXPNO         2
PROCNO        1
Date_         20150625
Time          18.43
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
```

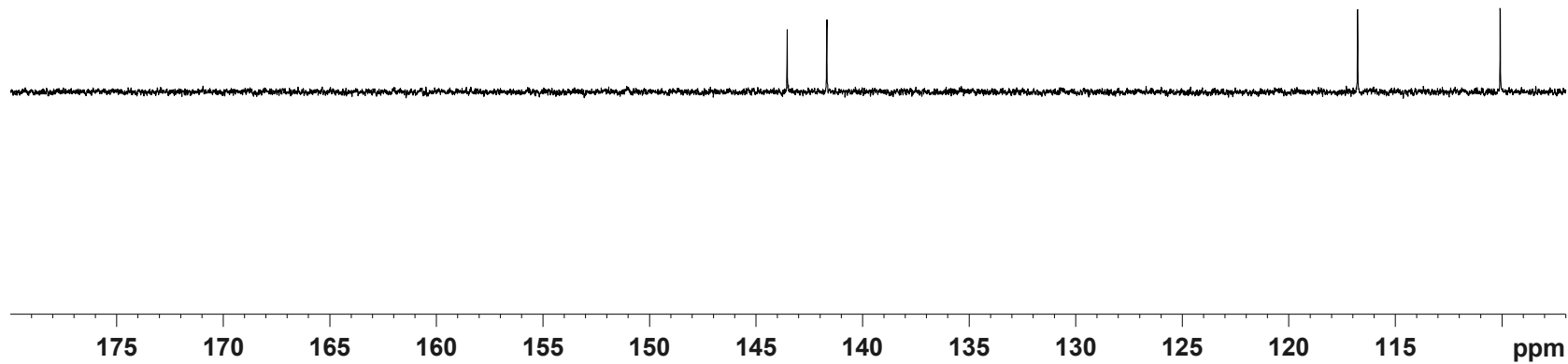
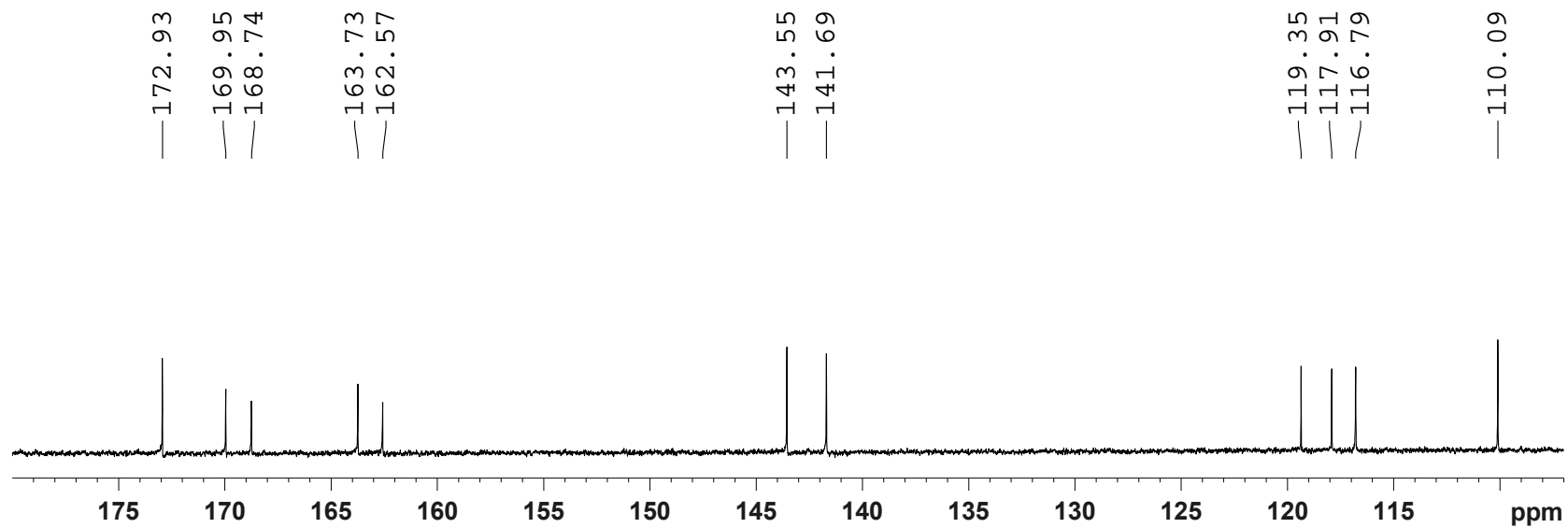
```
===== CHANNEL f1 =====
SFO1          100.6233324 MHz
NUC1          13C
P1            10.00 usec
SI            32768
SF            100.6127685 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

DEPT135 (100 MHz) spectrum of compound **2** in CDCl₃

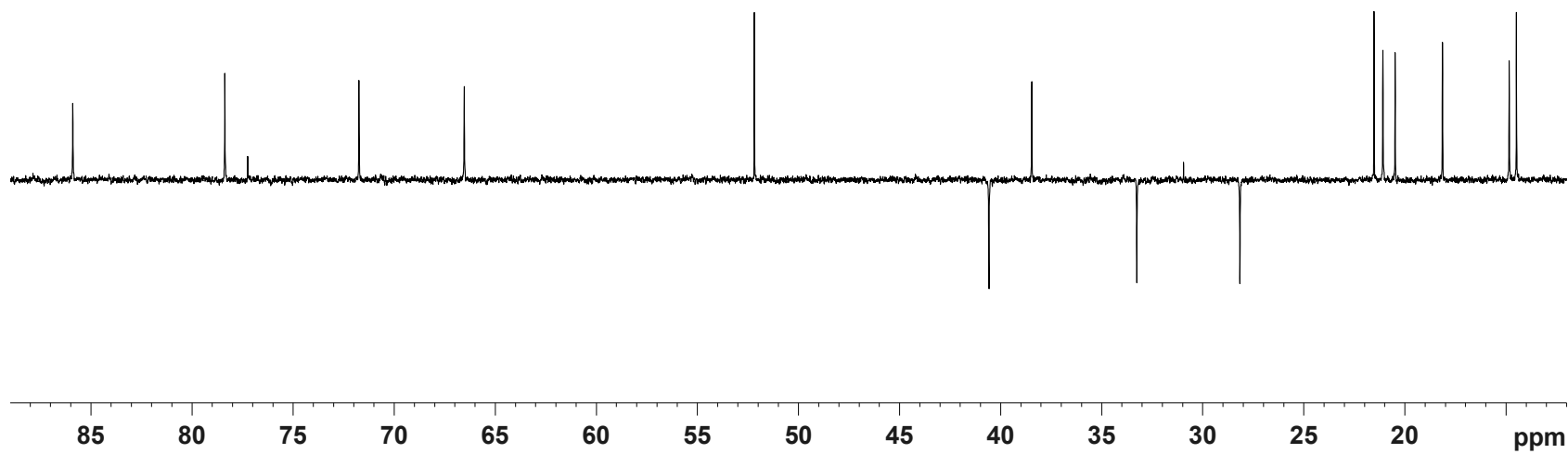
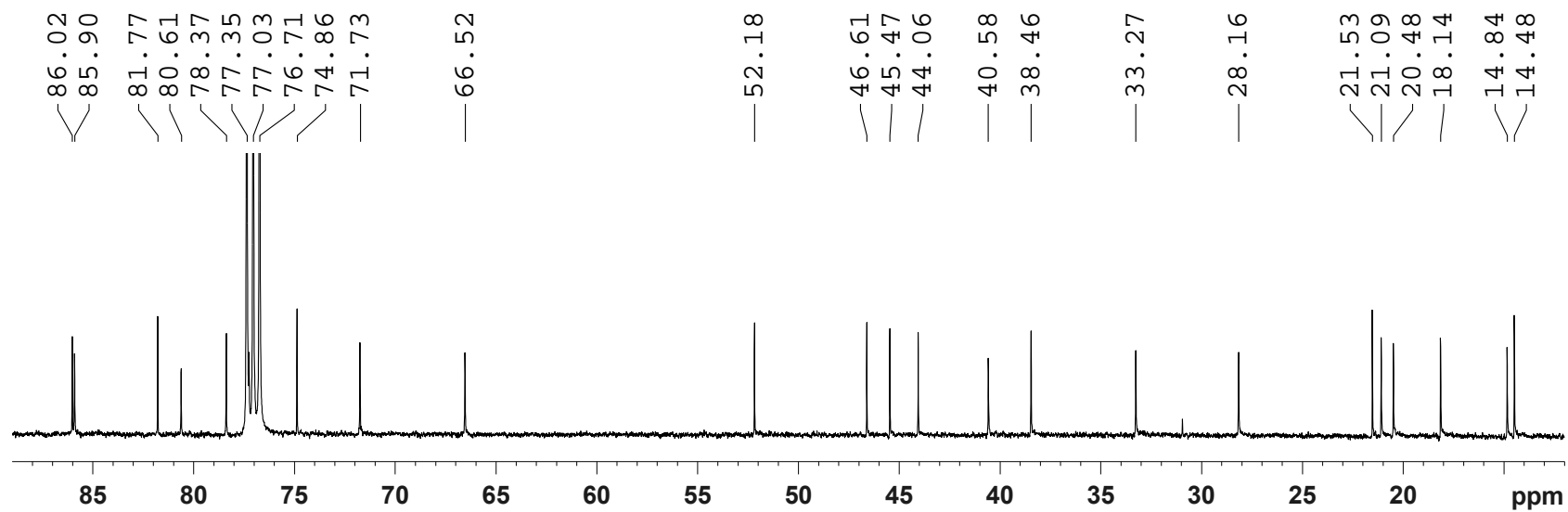


S41

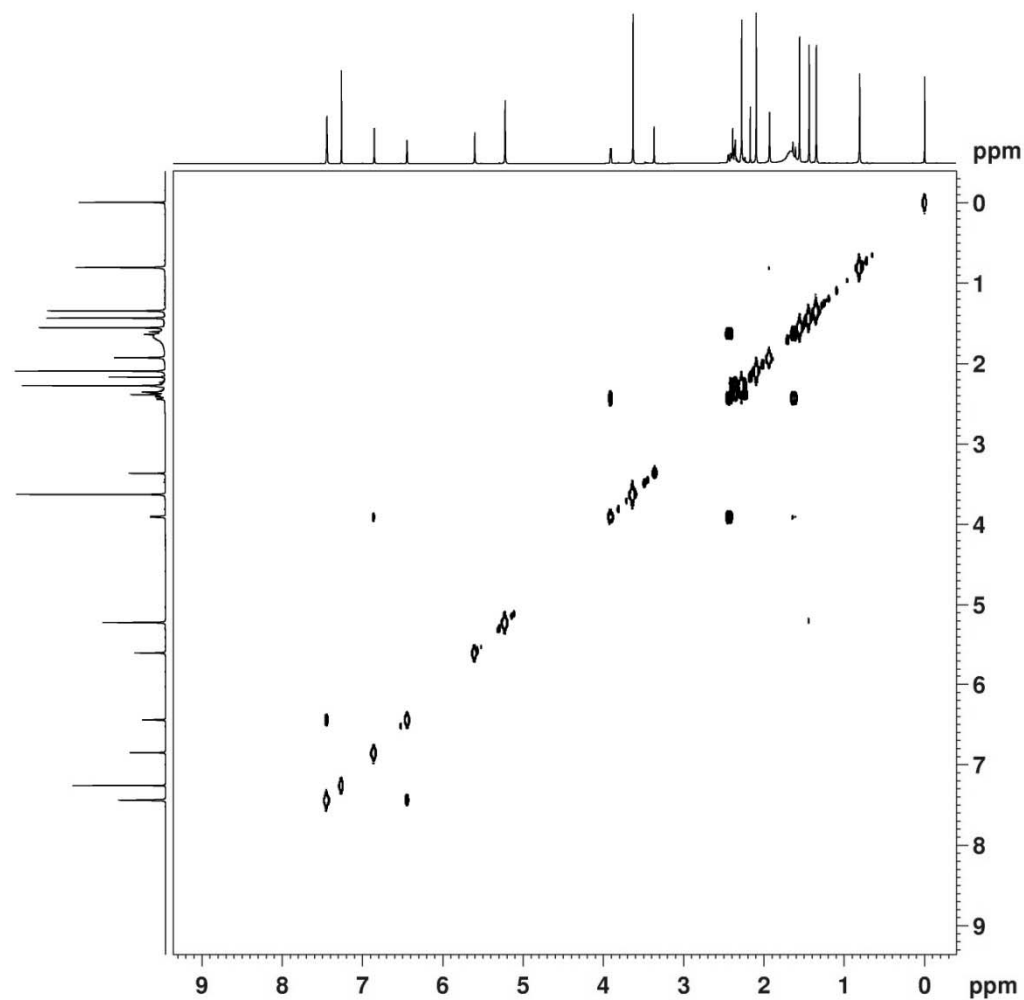
DEPT135 (100 MHz) spectrum of compound **2** in CDCl₃



DEPT135 (100 MHz) spectrum of compound **2** in CDCl₃



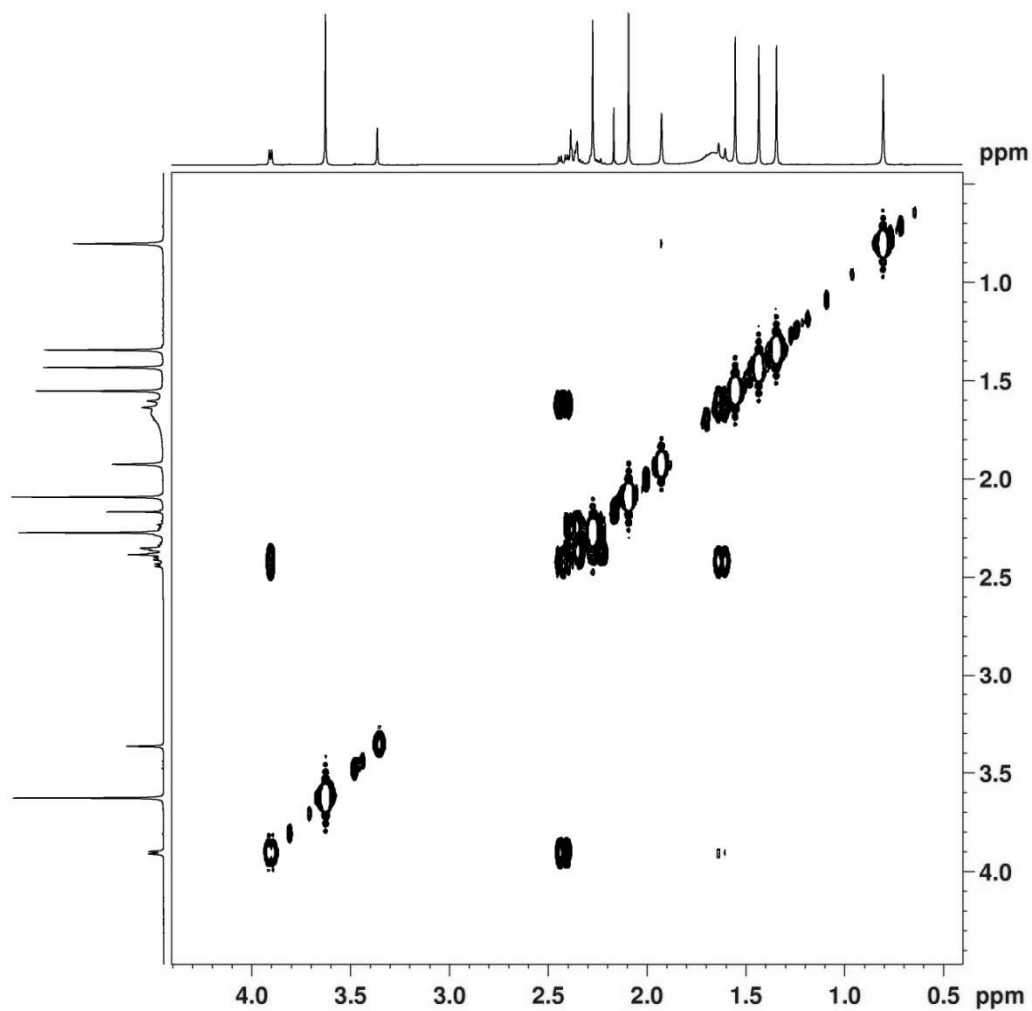
^1H - ^1H COSY (400 MHz) spectrum of compound **2** in CDCl_3



```
NAME          dgy-3
EXPNO         4
PROCNO        1
Date_         20150905
Time          3.57
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       cosygpppqf
TD            2048
SOLVENT       CDCl3
NS            8
DS            8
SWH           3906.250 Hz
FIDRES        1.907349 Hz
AQ            0.2621940 sec
RG            208.5
DW            128.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00000300 sec
D1            1.89678097 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00025600 sec
```

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

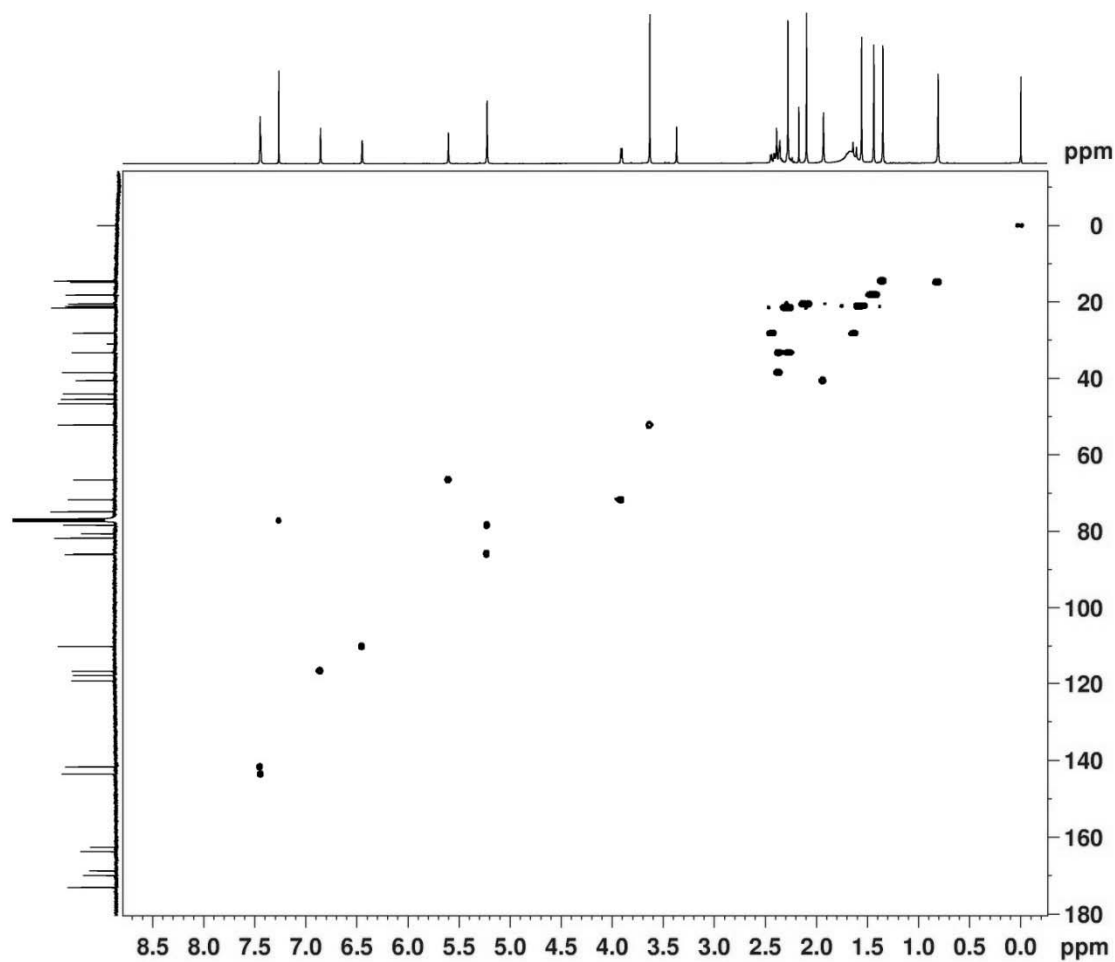

^1H - ^1H COSY (400 MHz) spectrum of compound **2** in CDCl_3



```
NAME          dgy-3
EXPNO          4
PROCNO         1
Date_         20150905
Time          3.57
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
INO           0.00025600 sec
```

```
===== CHANNEL f1 =====
SFO1          400.1318006 MHz
NUC1          1H
P0            12.00 usec
P1            12.00 usec
P17           2500.00 usec
ND0           1
TD            128
SFO1          400.1318 MHz
FIDRES        30.517578 Hz
SW            9.762 ppm
FrMODE        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
```

HSQC (400 MHz) spectrum of compound **2** in CDCl₃



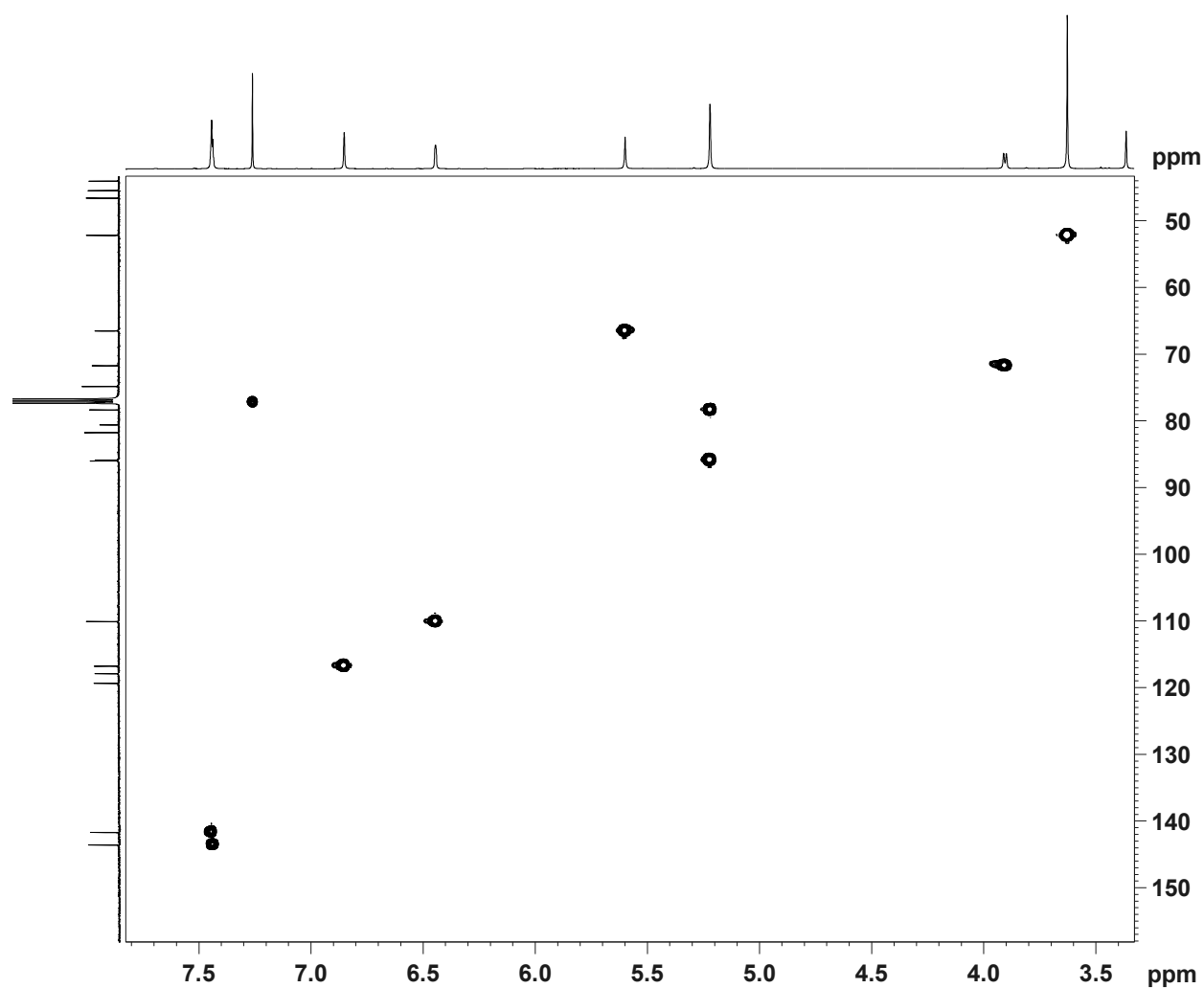
```

NAME          dgy-3
EXPNO         5
PROCNO        1
Date_         20150905
Time          4.37
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgps12
TD            1024
SOLVENT       CDCl3
NS            16
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 sec
RG            208.5
DW            116.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
D0            0.00000300 sec
D1            1.46497905 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00002080 sec
ZGPTNS
    
```

```

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

HSQC (400 MHz) spectrum of compound **2** in CDCl₃



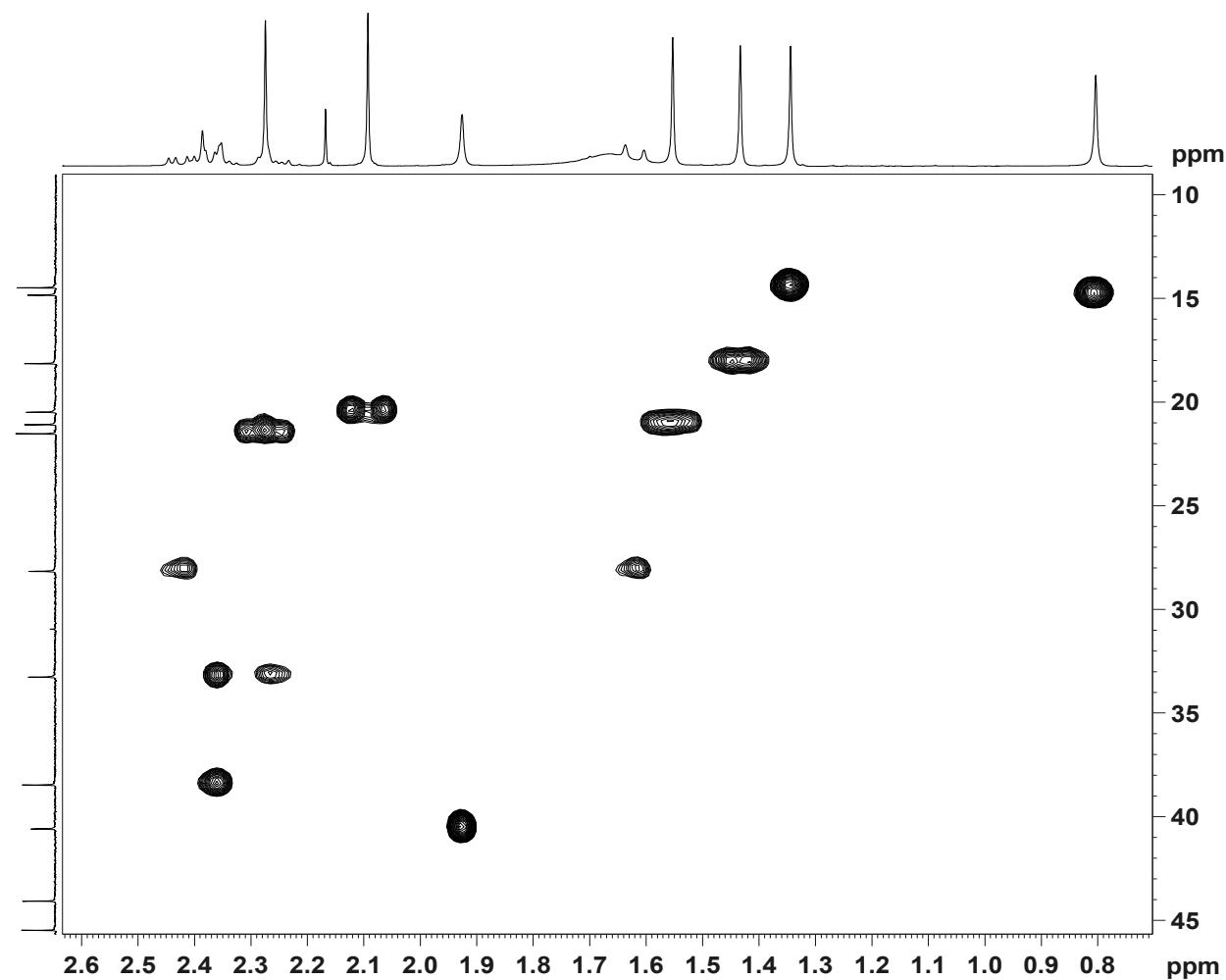
```

NAME                dyg-3
EXPNO                5
PROCNO              1
Date_                20150905
Time                4.37
INSTRUM              spect
PROBHD              5 mm CPPBBO BB
PULPROG              hsqcetgps12
TD                  1024
SOLVENT              CDCl3
NS                   16
DS                   16
SWH                  4302.926 Hz
FIDRES               4.202076 Hz
AQ                   0.1190388 sec
RG                   208.5
DW                   116.200 usec
DE                   10.00 usec
TE                   297.0 K
CNST2                145.0000000
D0                   0.00000300 sec
D1                   1.46497905 sec
D4                   0.00172414 sec
D11                  0.03000000 sec
D16                  0.00020000 sec
D24                  0.00086207 sec
IN0                  0.00002080 sec
ZGPTNS
    
```

```

===== CHANNEL f1 =====
SF01                400.1320007 MHz
NUC1                 1H
P1                   12.00 usec
P2                   24.00 usec
P28                  0.00 usec
ND0                  2
TD                   256
SF01                100.6233 MHz
FIDRES               93.900238 Hz
SW                   238.896 ppm
FnMODE               Echo-Antiecho
SI                   1024
SF                   400.1300079 MHz
WDW                  QSINE
SSB                   2
LB                   0.00 Hz
GB                   0
PC                   1.40
SI                   1024
MC2                  echo-antiecho
SF                   100.6127685 MHz
WDW                  QSINE
SSB                   2
LB                   0.00 Hz
GB                   0
    
```

HSQC (400 MHz) spectrum of compound **2** in CDCl₃



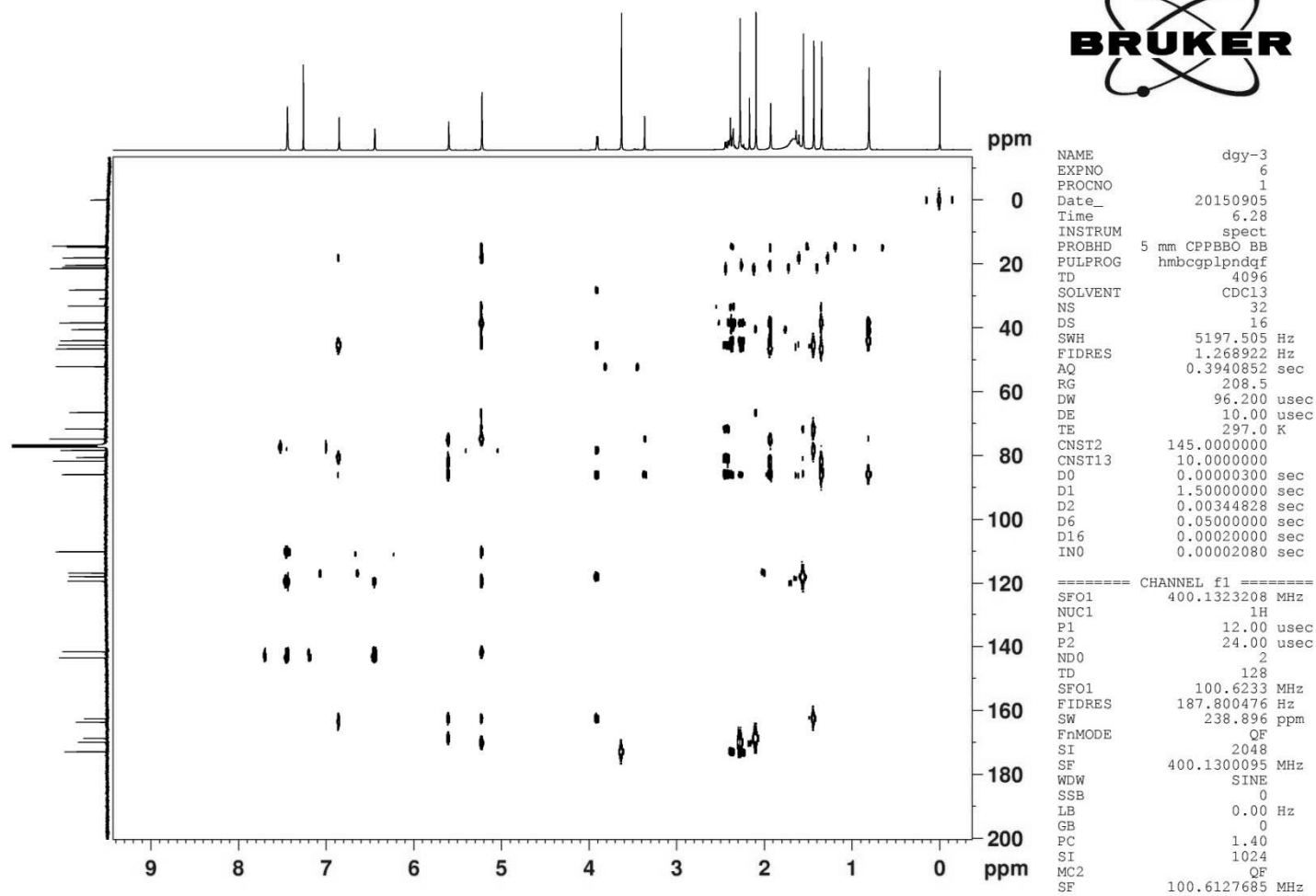
```

NAME          dyg-3
EXPNO         5
PROCNO        1
Date_         20150905
Time          4.37
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
ZGPTNS
    
```

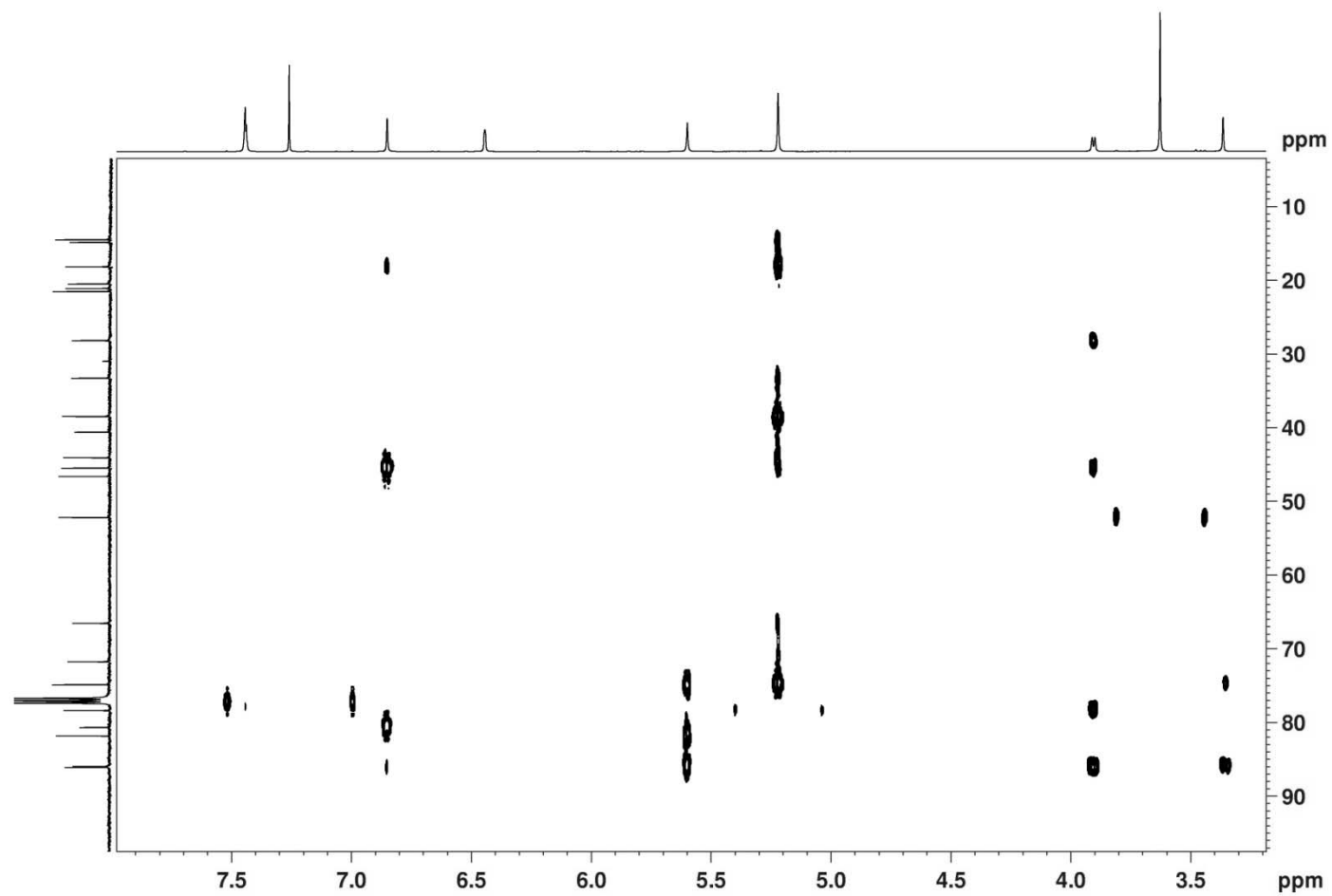
```

===== CHANNEL f1 =====
SF01          400.1320007 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
P28           0.00 usec
ND0           2
TD            256
SF01          100.6233 MHz
FIDRES        93.900238 Hz
SW            238.896 ppm
FnMODE        Echo-Antiecho
SI            1024
SF            400.1300079 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            100.6127685 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

HMBC (400 MHz) spectrum of compound **2** in CDCl₃

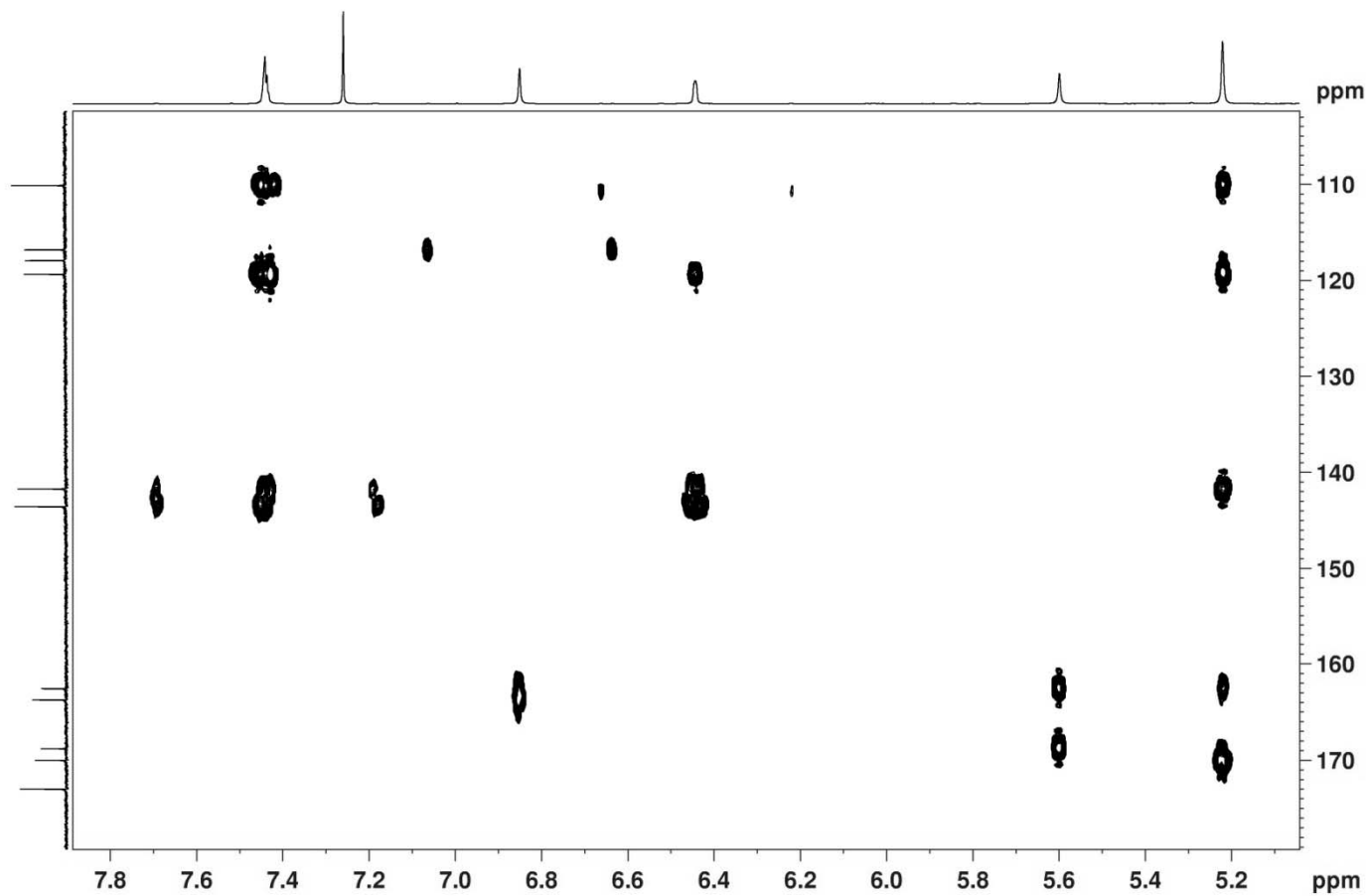


HMBC (400 MHz) spectrum of compound **2** in CDCl₃

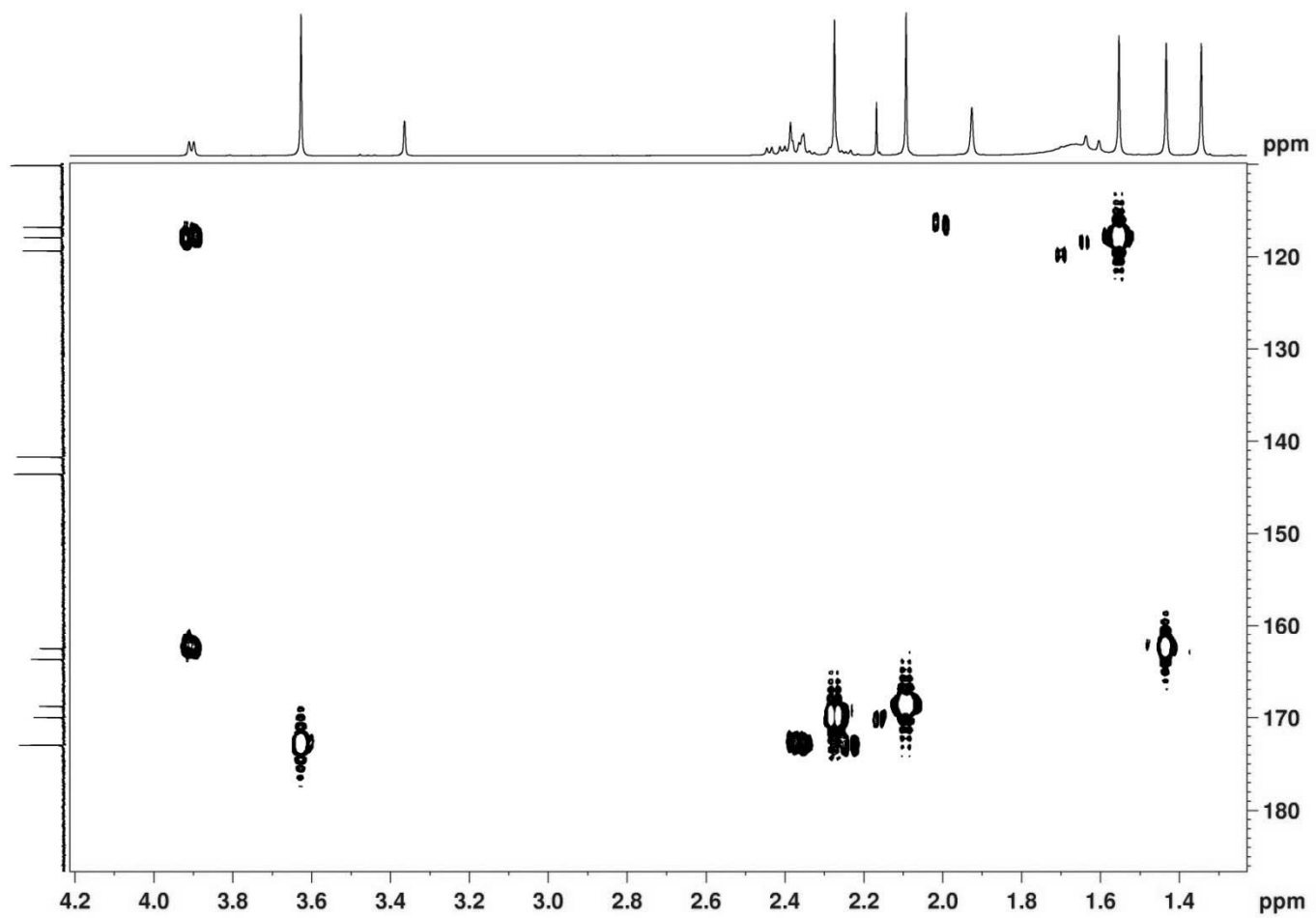


S50

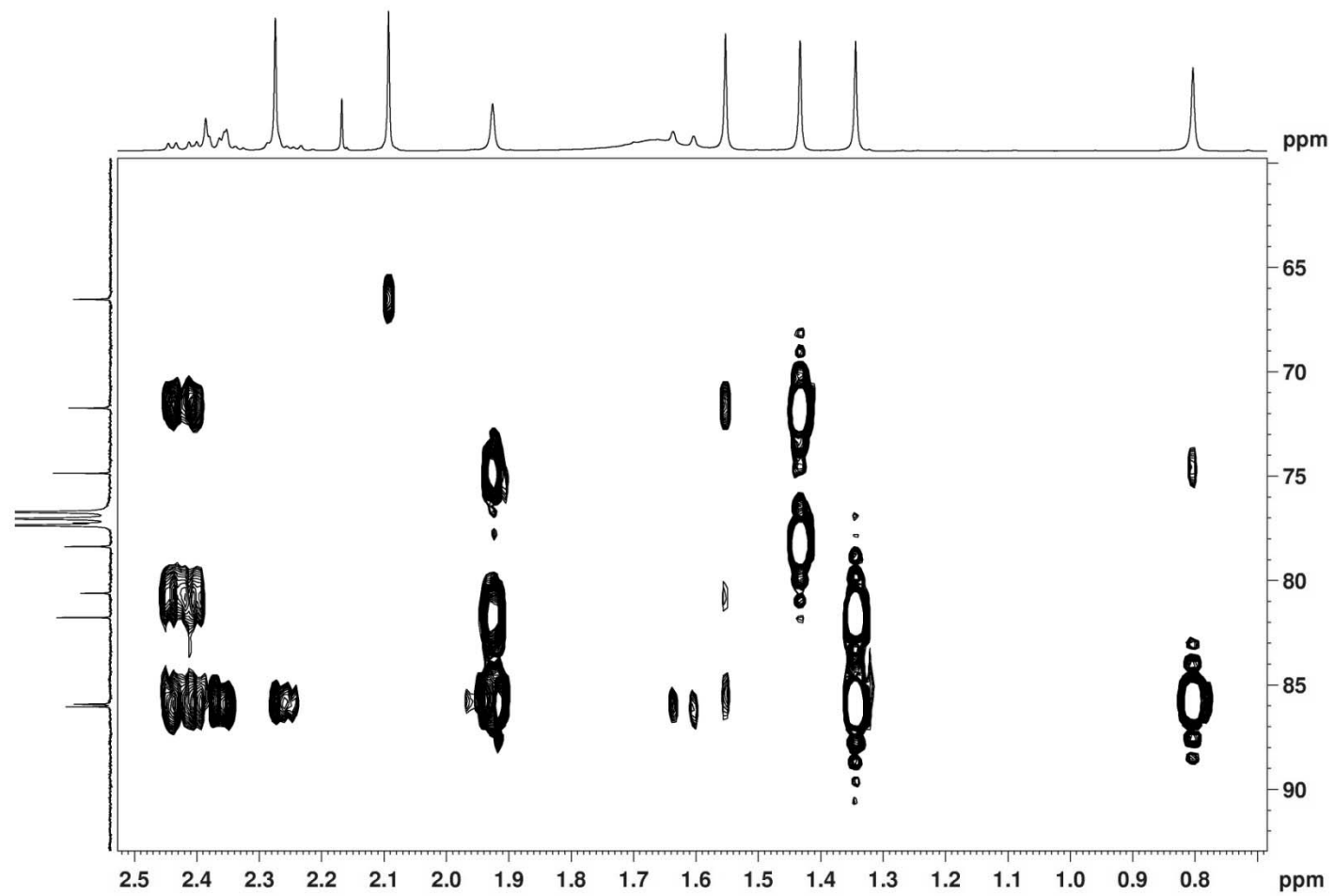
HMBC (400 MHz) spectrum of compound **2** in CDCl₃



HMBC (400 MHz) spectrum of compound **2** in CDCl₃

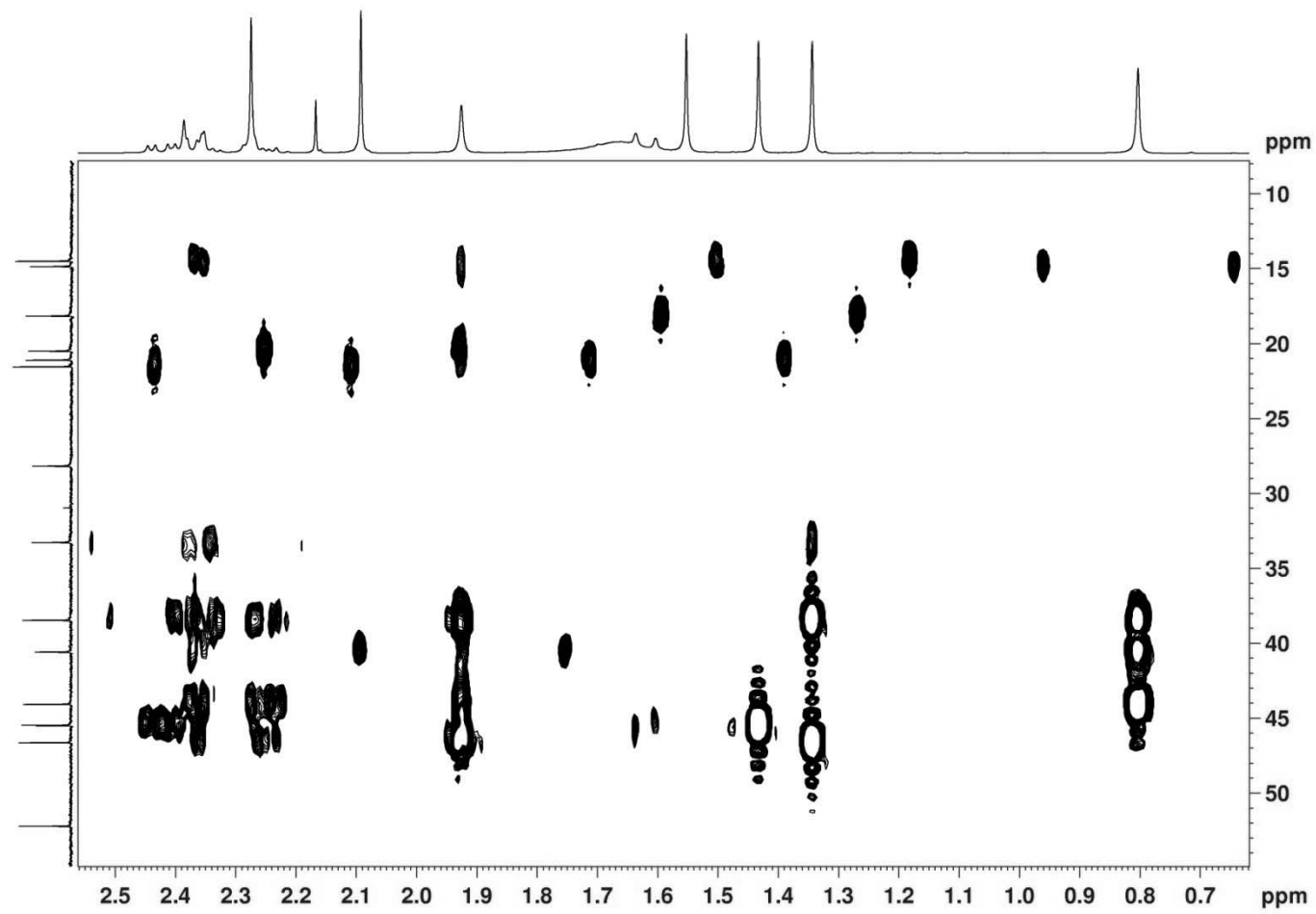


HMBC (400 MHz) spectrum of compound **2** in CDCl₃

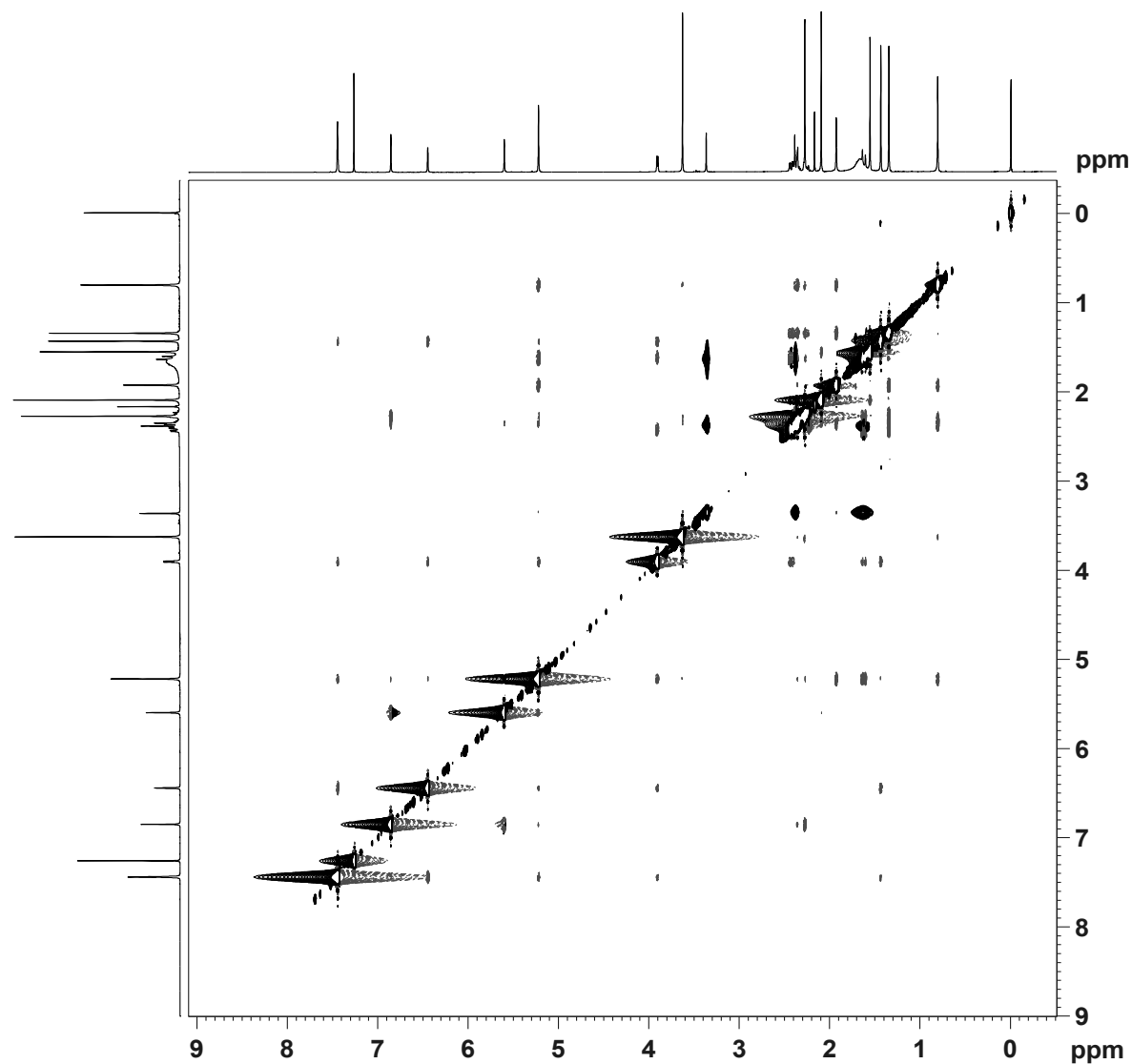
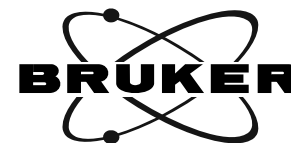


S53

HMBC (400 MHz) spectrum of compound **2** in CDCl₃



NOESY (400 MHz) spectrum of compound **2** in CDCl₃



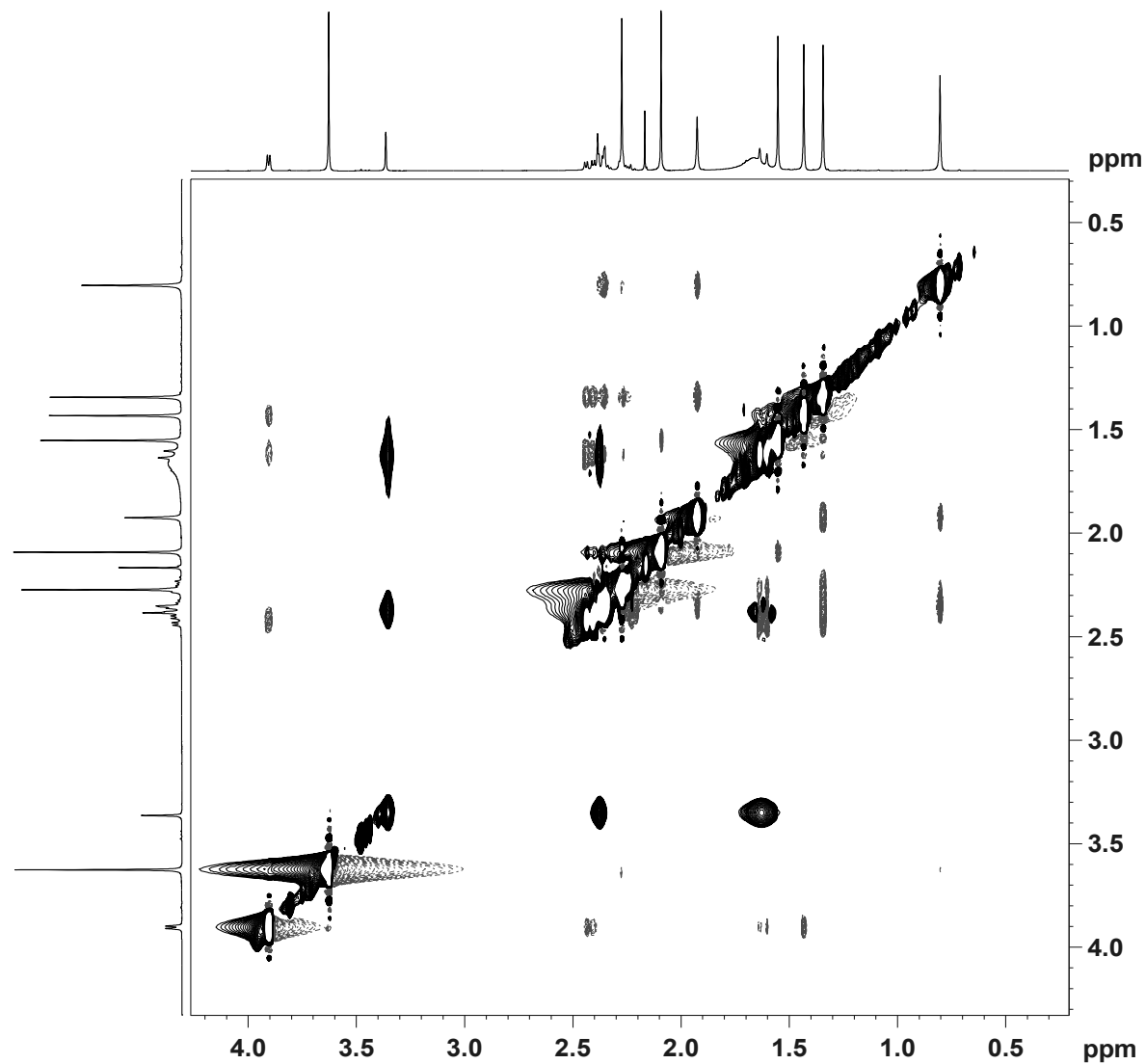
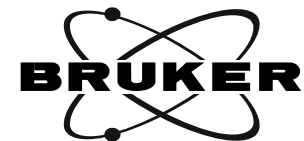
```

NAME          dyg-3
EXPNO         7
PROCNO        1
Date_         20150905
Time          8.44
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 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

===== CHANNEL f1 =====
SFO1         400.1318006 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
P17           2500.00 usec
ND0           1
TD            230
SFO1         400.1318 MHz
FIDRES        17.391304 Hz
SW            9.997 ppm
FnMODE        States-TPPI
SI            1024
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

NOESY (400 MHz) spectrum of compound **2** in CDCl₃



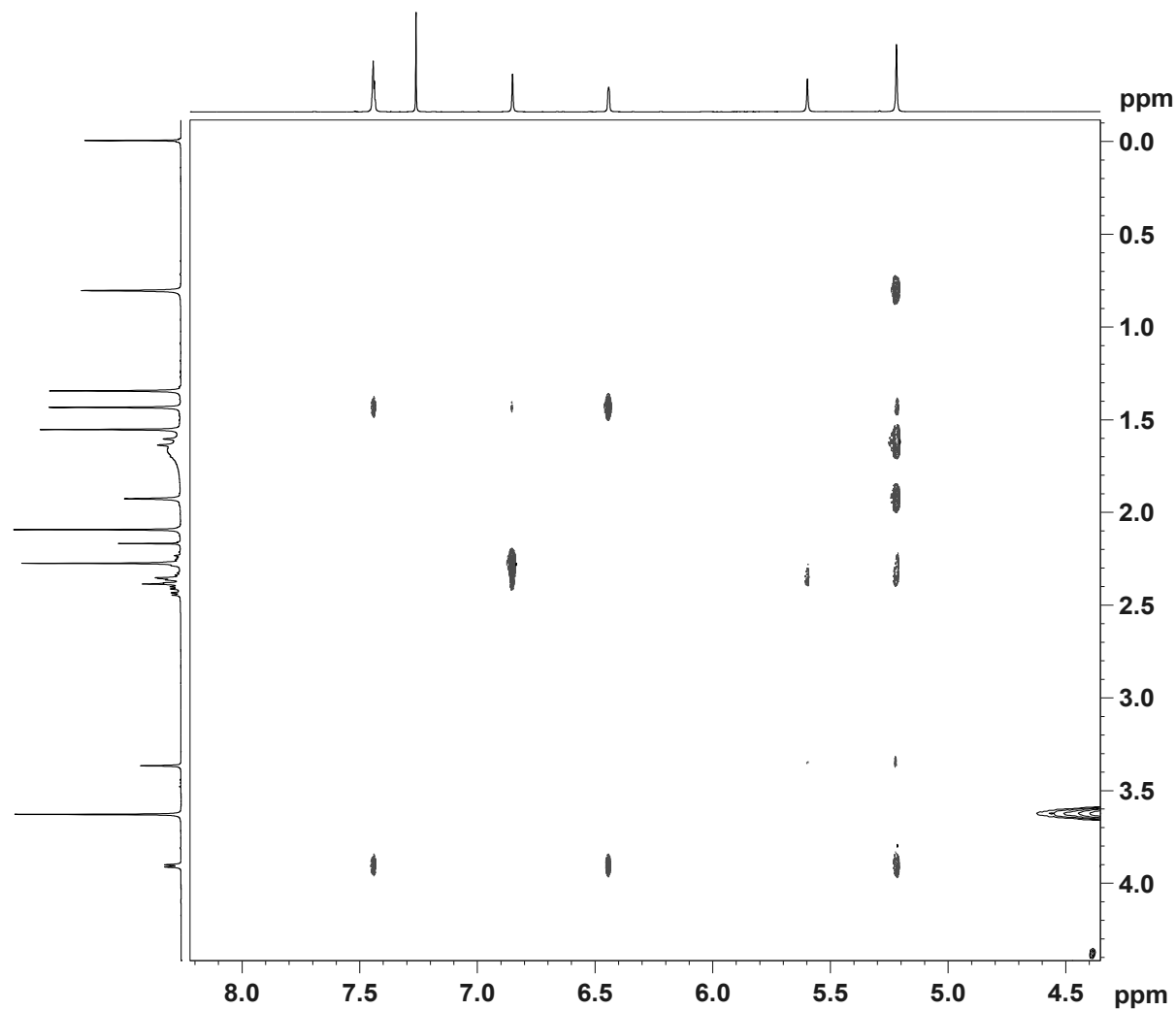
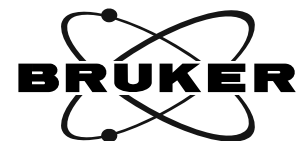
```

NAME          dyg-3
EXPNO         7
PROCNO        1
Date_         20150905
Time          8.44
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 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

===== CHANNEL f1 =====
SF01         400.1318006 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
P17           2500.00 usec
ND0           1
TD            230
SF01         400.1318 MHz
FIDRES        17.391304 Hz
SW            9.997 ppm
FnMODE        States-TPPI
SI            1024
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

NOESY (400 MHz) spectrum of compound **2** in CDCl₃



```

NAME          dyg-3
EXPNO         7
PROCNO       1
Date_        20150905
Time         8.44
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 sec
RG           208.5
DW           125.000 usec
DE           10.00 usec
TE           297.0 K
D0           0.00010972 sec
D1           1.99385595 sec
D8           0.30000001 sec
D11          0.03000000 sec
D12          0.00002000 sec
D16          0.00020000 sec
IN0          0.00025000 sec
    
```

```

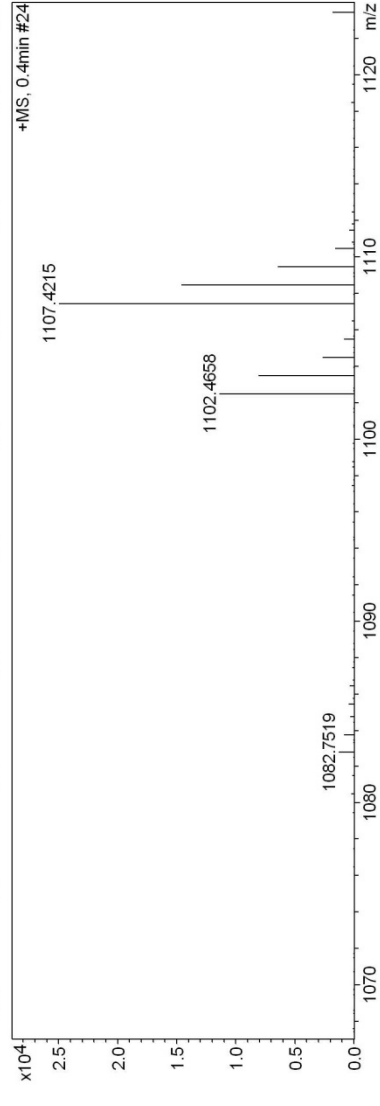
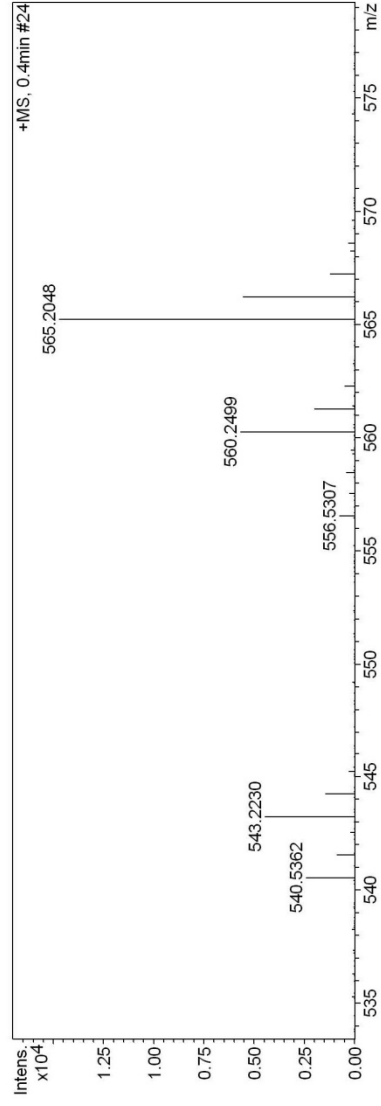
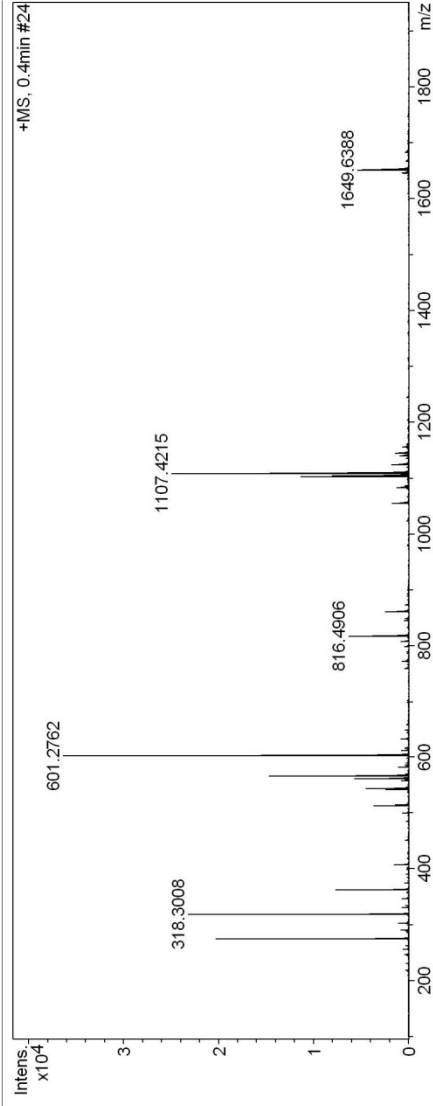
===== CHANNEL f1 =====
SF01       400.1318006 MHz
NUC1        1H
P1          12.00 usec
P2          24.00 usec
P17         2500.00 usec
ND0         1
TD          230
SF01       400.1318 MHz
FIDRES      17.391304 Hz
SW          9.997 ppm
FnMODE     States-TPPI
SI          1024
SF          400.1300098 MHz
WDW         QSINE
SSB         2
LB          0.00 Hz
GB          0
PC          1.00
SI          1024
MC2        States-TPPI
SF          400.1300098 MHz
WDW         QSINE
SSB         2
LB          0.00 Hz
GB          0
    
```

HR-ESIMS for compound 3

Generic Display Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-8_pos_3_01_1851.d
Method LC_Direct_Infusion_pos_100-1000mz.m
Sample Name liwanshan_dgy-8_pos
Comment
Acquisition Date 5/19/2016 8:27:30 AM
Operator SCSIO
Instrument maXis



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HR-ESIMS for compound 3

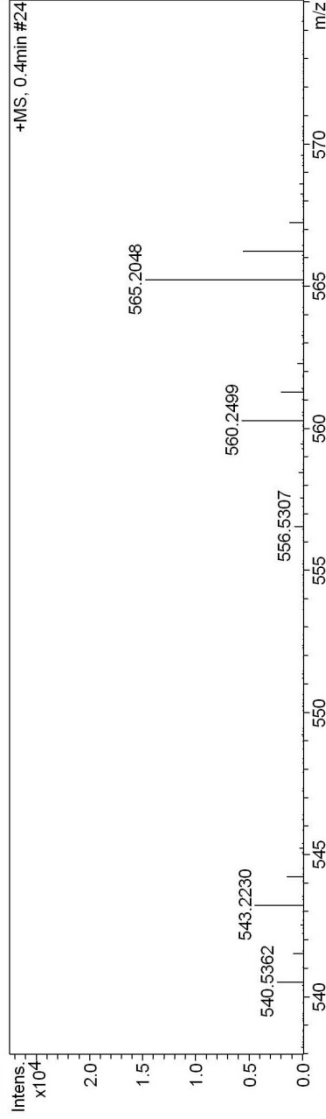
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-8_pos_3_01_1851.d Acquisition Date 5/19/2016 8:27:30 AM
 Method LC_Direct Infusion_pos_100-1000mz.m Operator SCSIO
 Sample Name liwanshan_dgy-8_pos Instrument / Set# maXis 29
 Comment

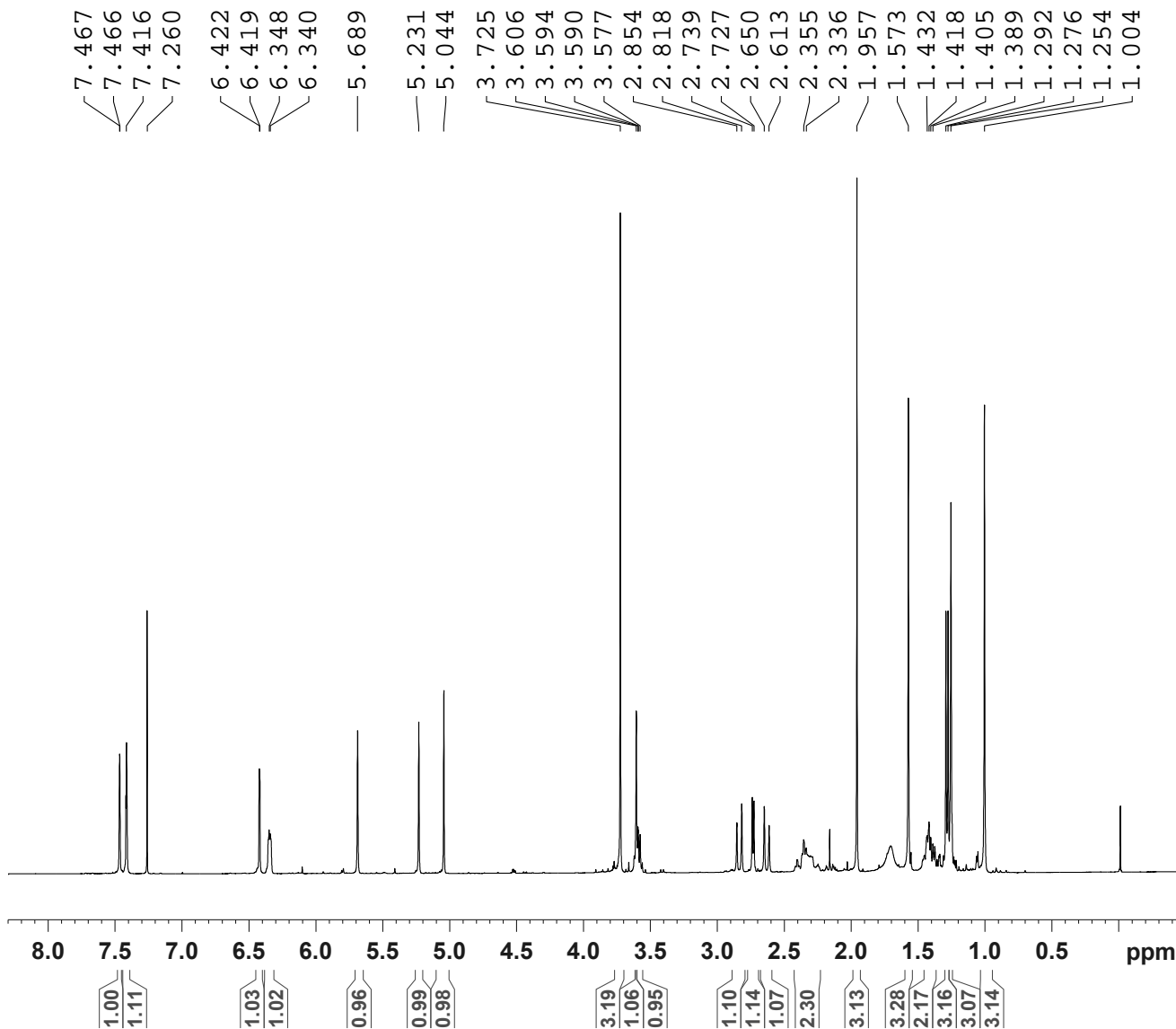
Acquisition Parameter

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



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e ⁻ Conf	N-Rule
543.2230	1	C ₂₉ H ₃₅ O ₁₀	100.00	543.2225	-1.0	-0.6	3.8	12.5	even	ok
565.2048	1	C ₂₉ H ₃₄ NaO ₁₀	100.00	565.2044	-0.6	-0.4	28.8	12.5	even	ok
1107.4215	1	C ₅₈ H ₆₈ NaO ₂₀	55.57	1107.4196	-1.7	-1.9	26.9	24.5	even	ok

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

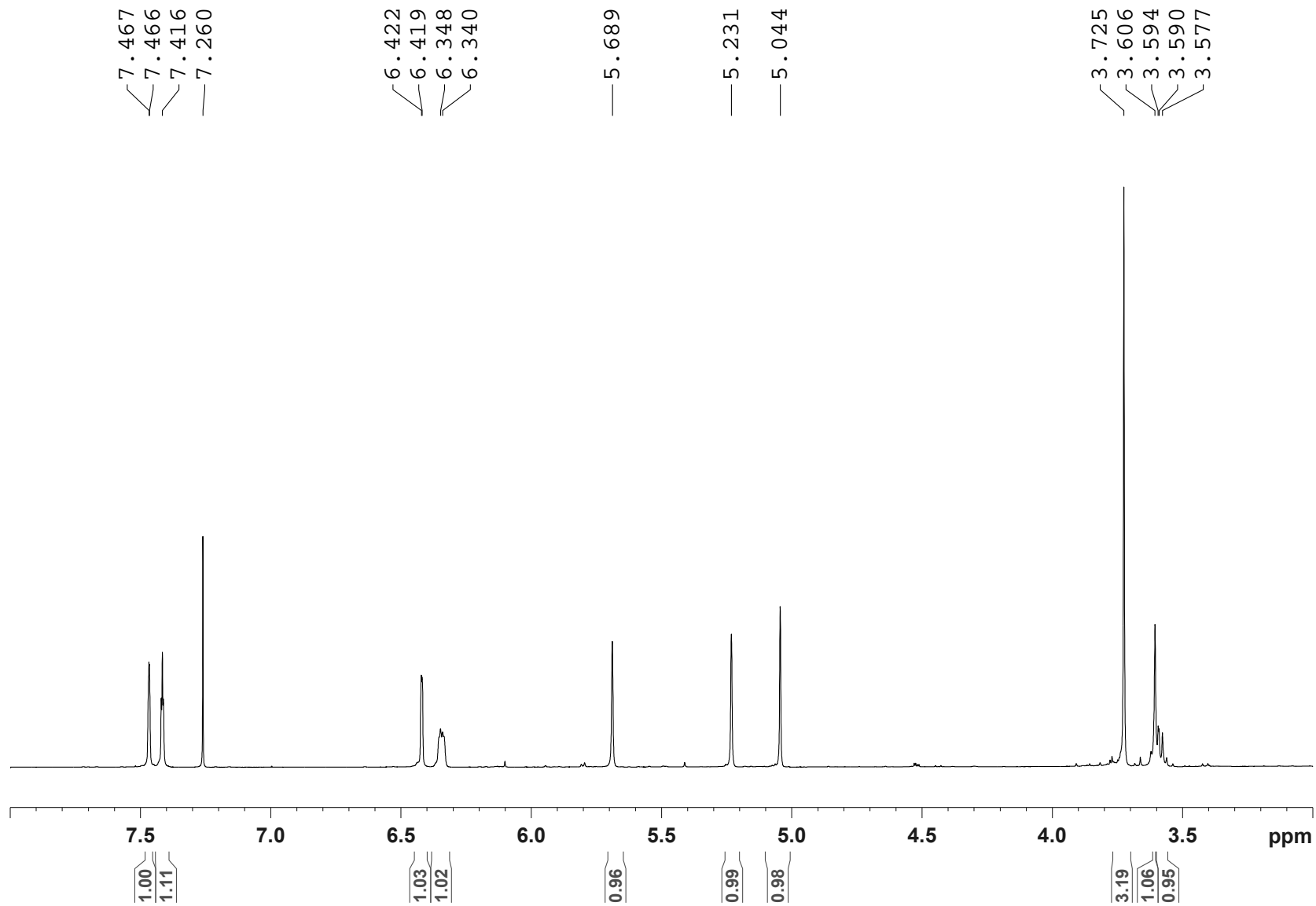


```

NAME                dyg-8
EXPNO                1
PROCNO              1
Date_                20150923
Time                21.44
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                   65.23
DW                   60.800 usec
DE                   10.00 usec
TE                   297.0 K
D1                   1.0000000 sec
TD0                  1

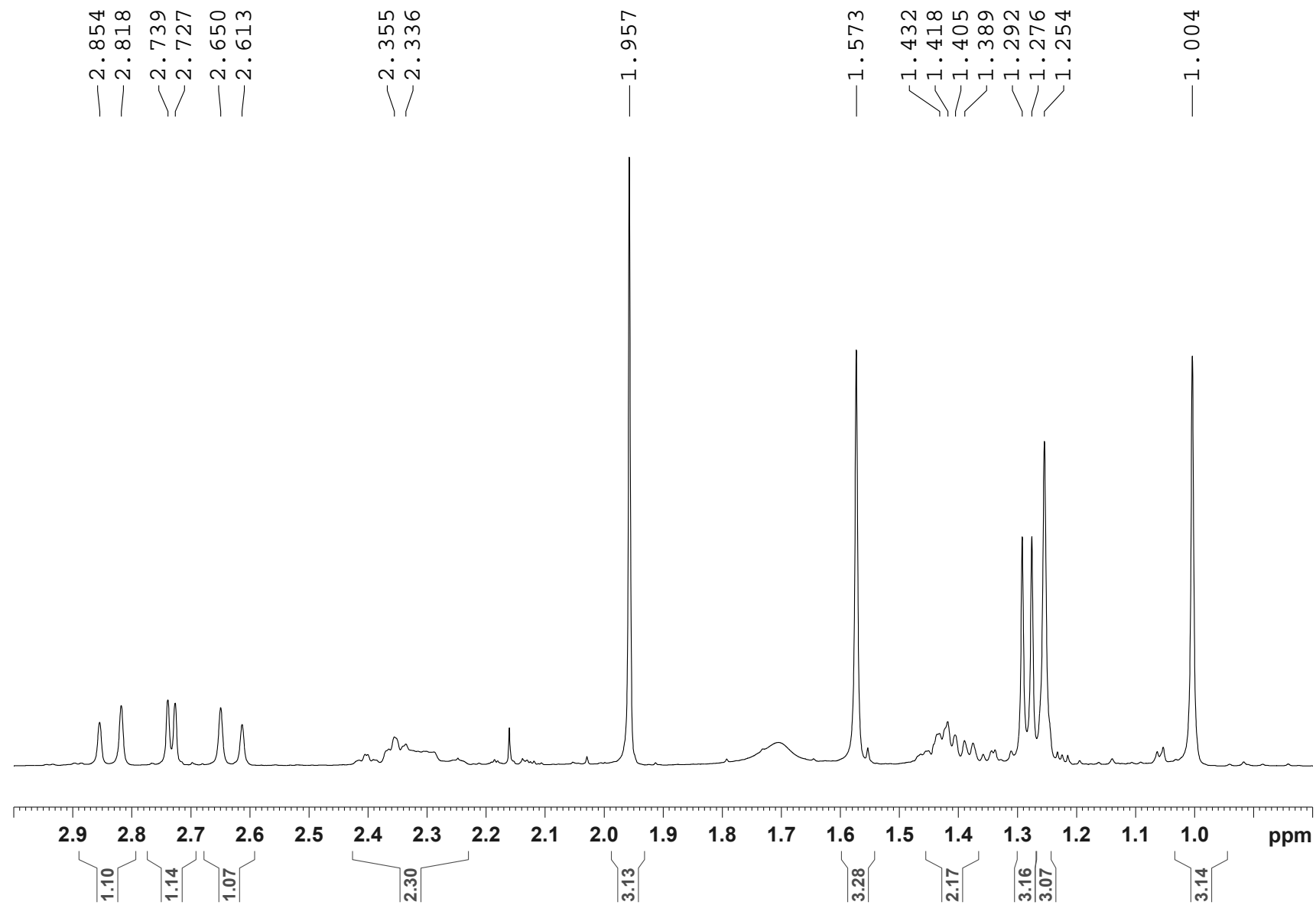
===== CHANNEL f1 =====
SFO1                400.1324710 MHz
NUC1                 1H
P1                   12.00 usec
SI                   65536
SF                   400.1300098 MHz
WDW                   EM
SSB                   0
LB                   0.30 Hz
GB                   0
PC                   1.00
    
```


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

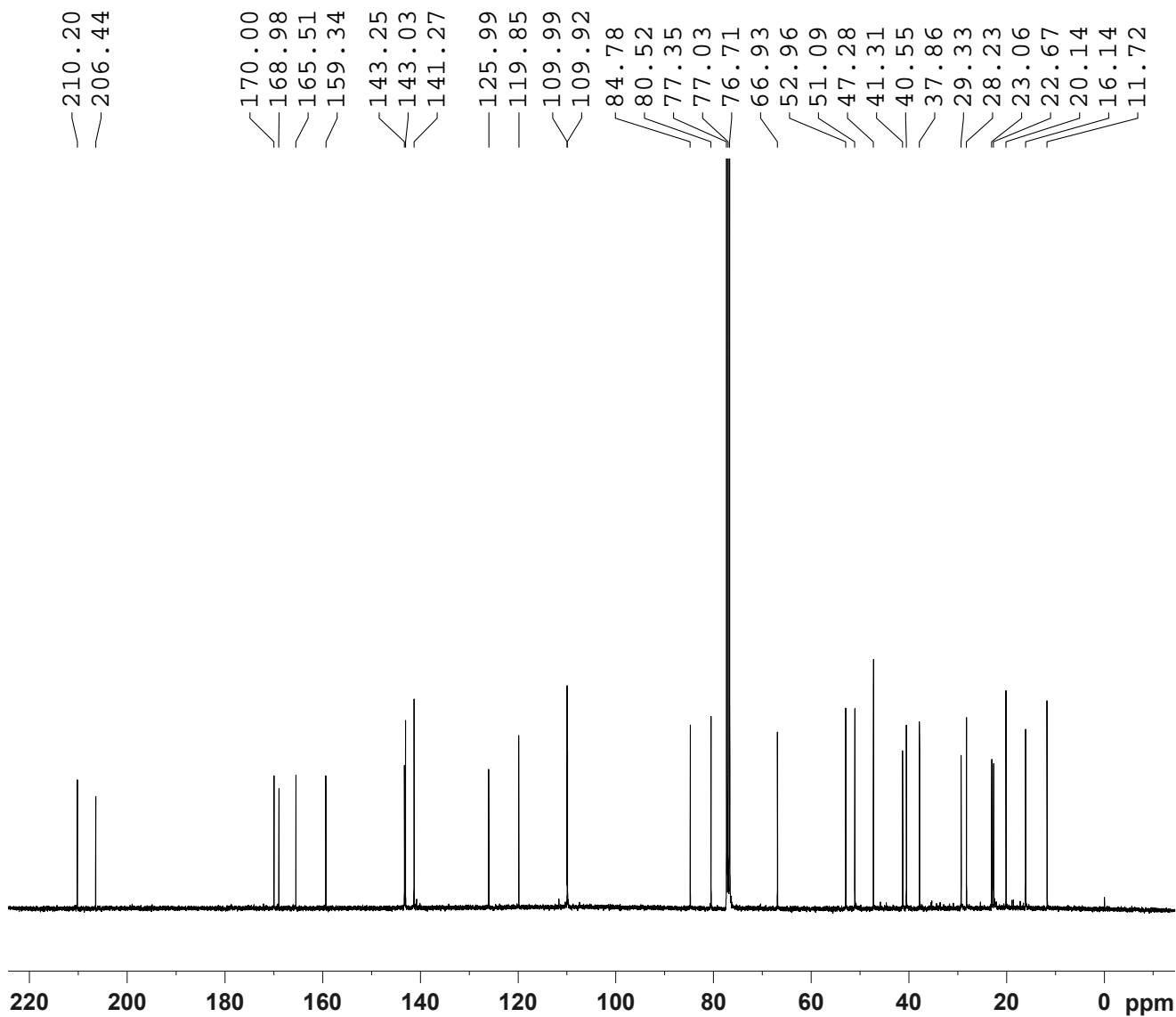
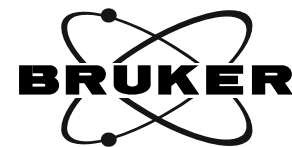


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¹H NMR (400 MHz) spectrum of compound **3** in CDCl₃



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

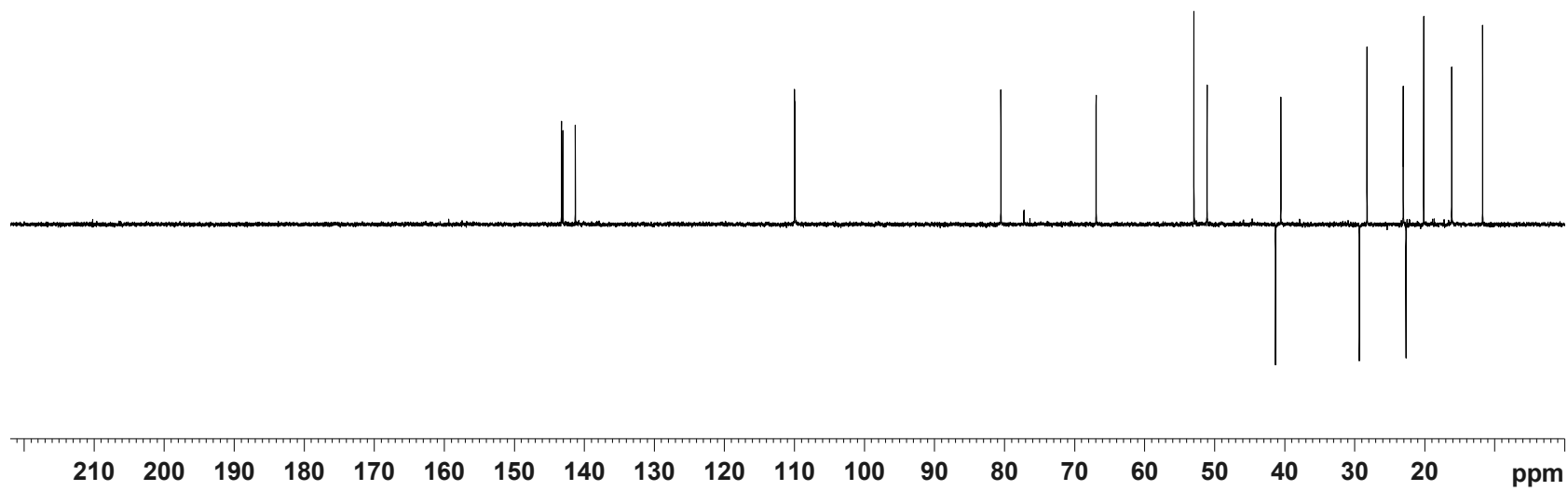
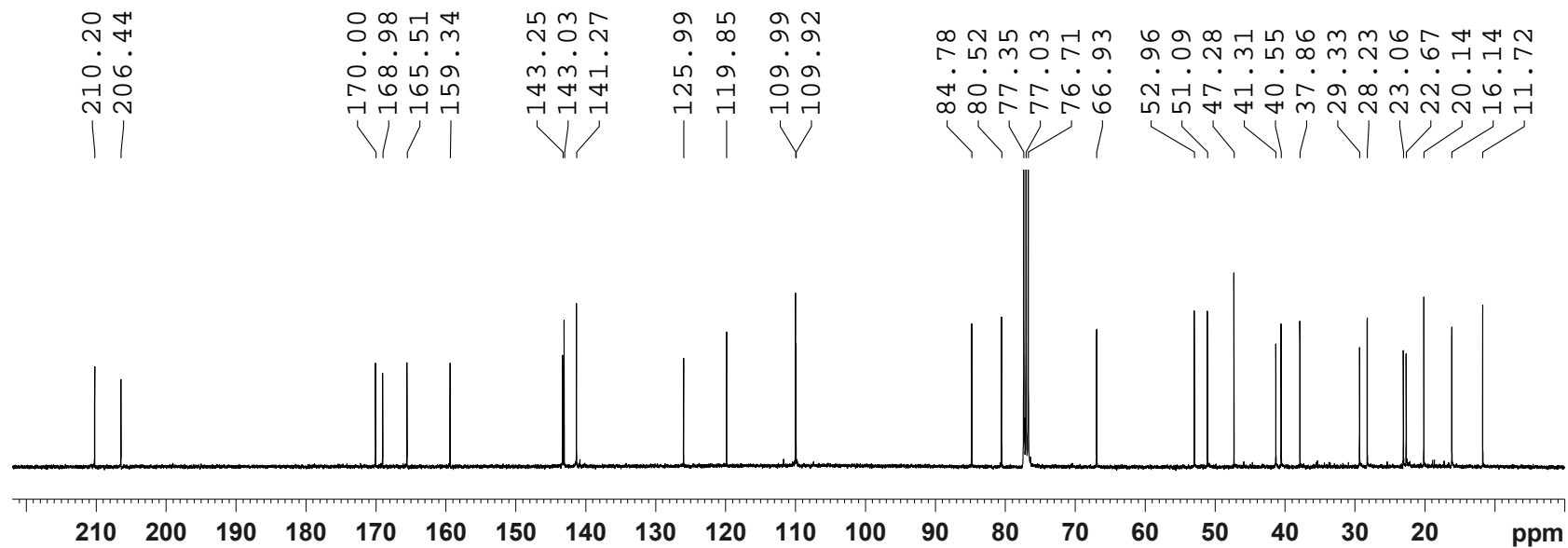


S63

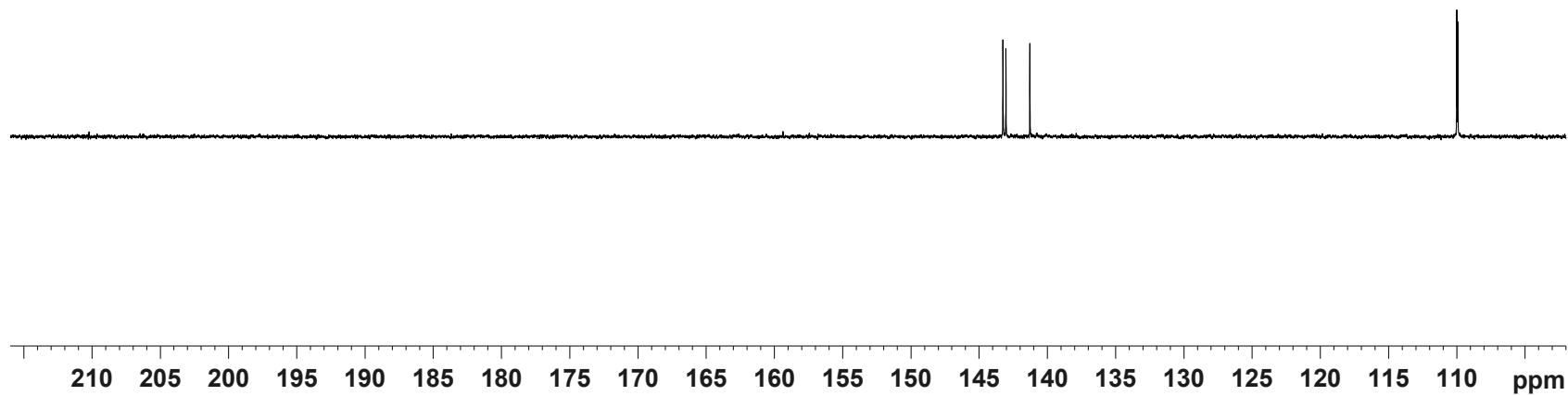
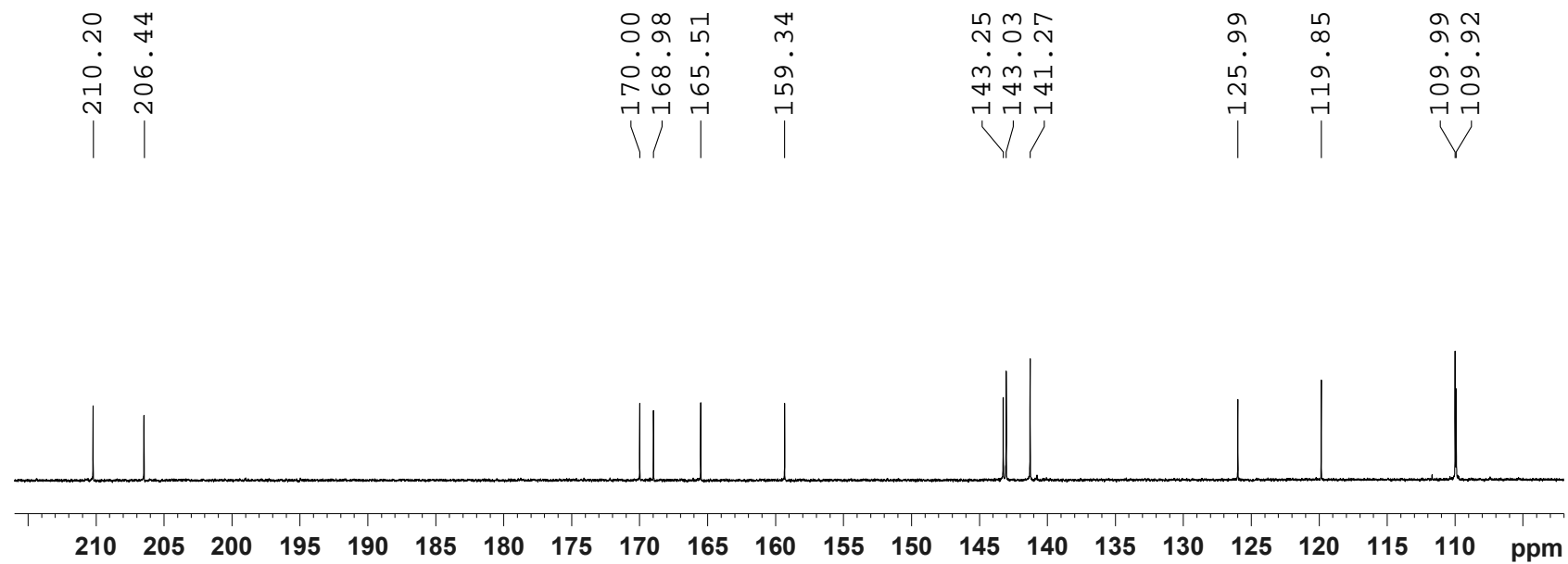
```
NAME          dyg-8
EXPNO         2
PROCNO        1
Date_         20150923
Time_         23.31
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            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            100.6127703 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

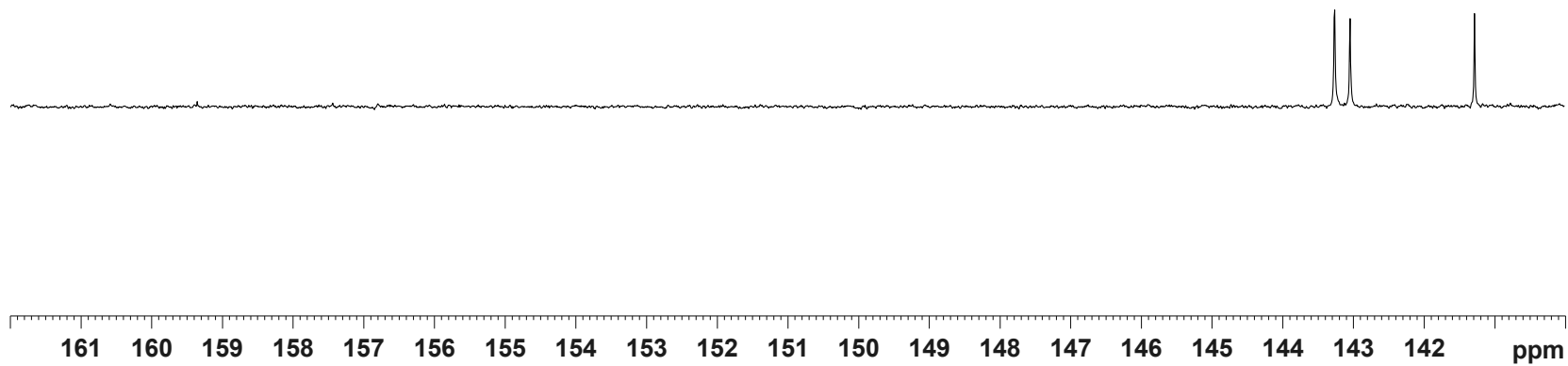
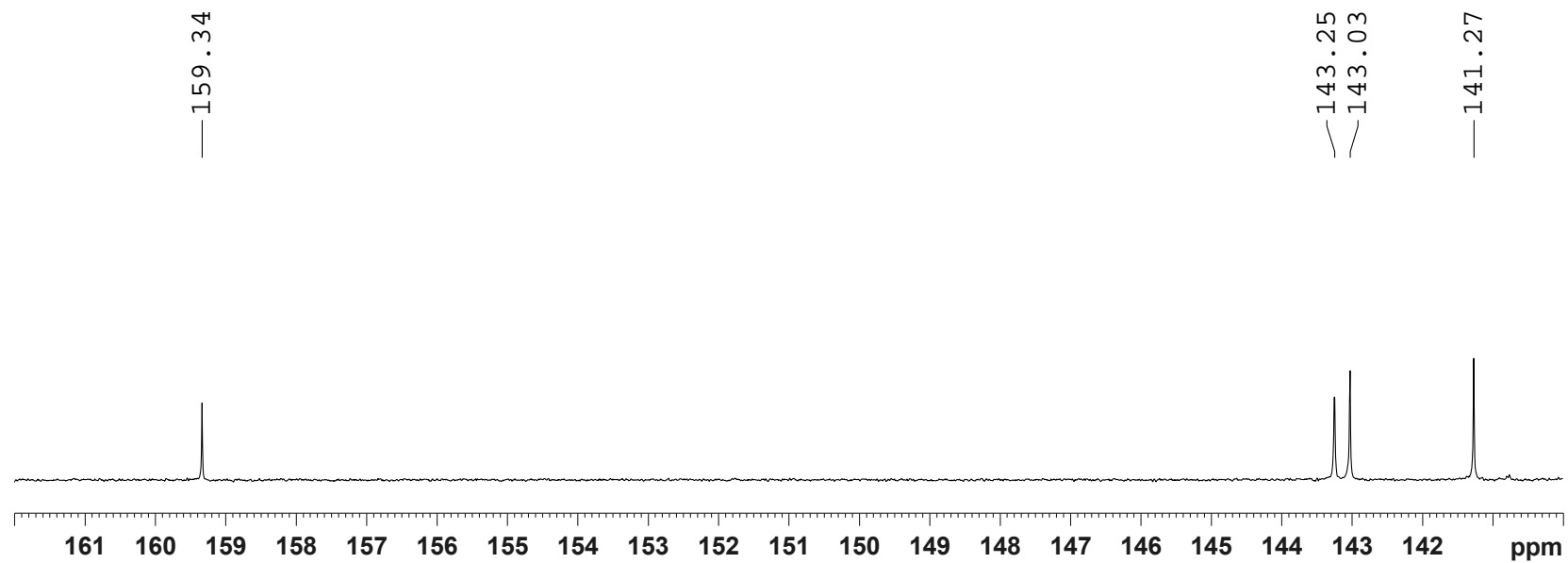
DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



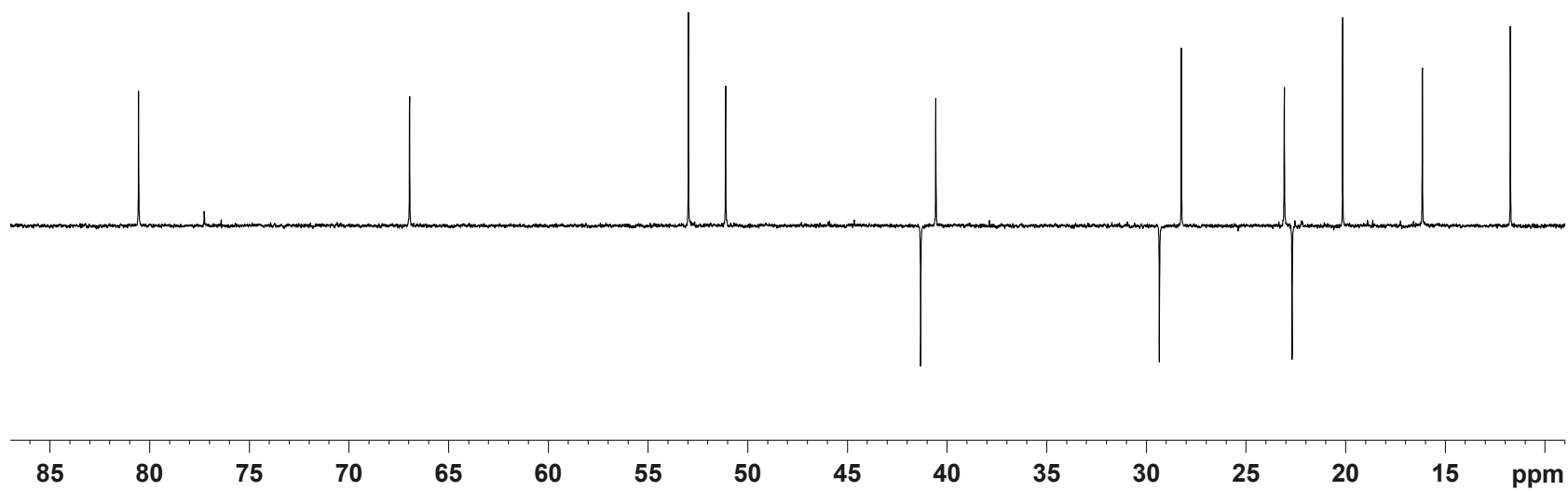
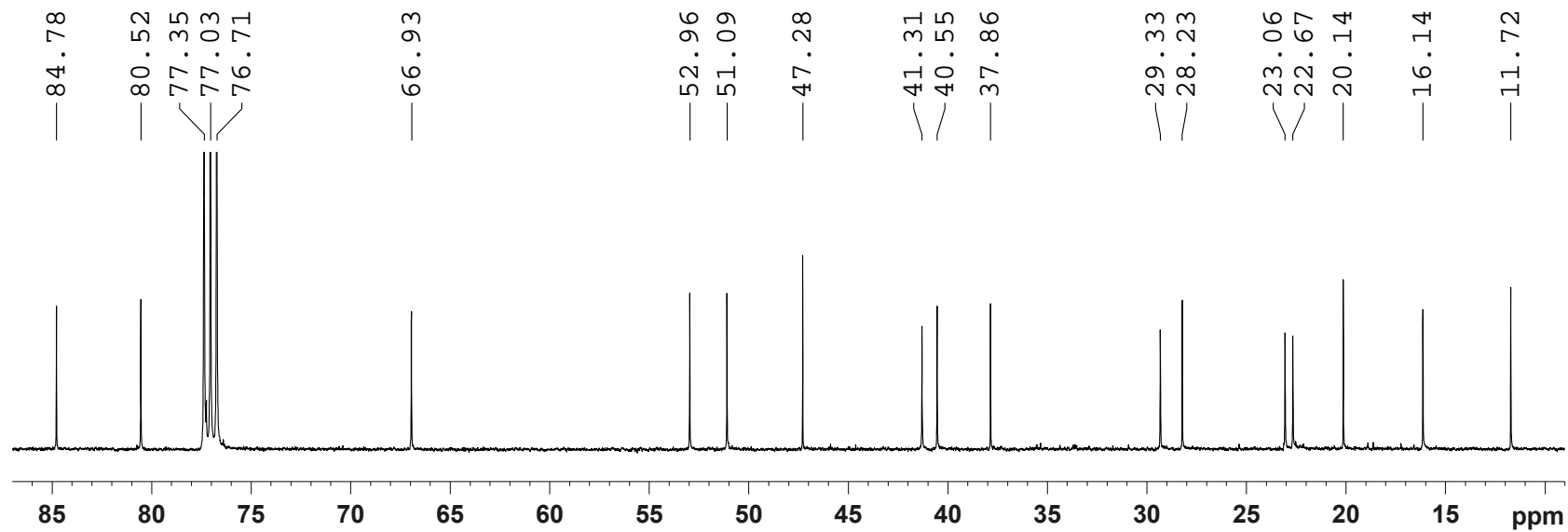
DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



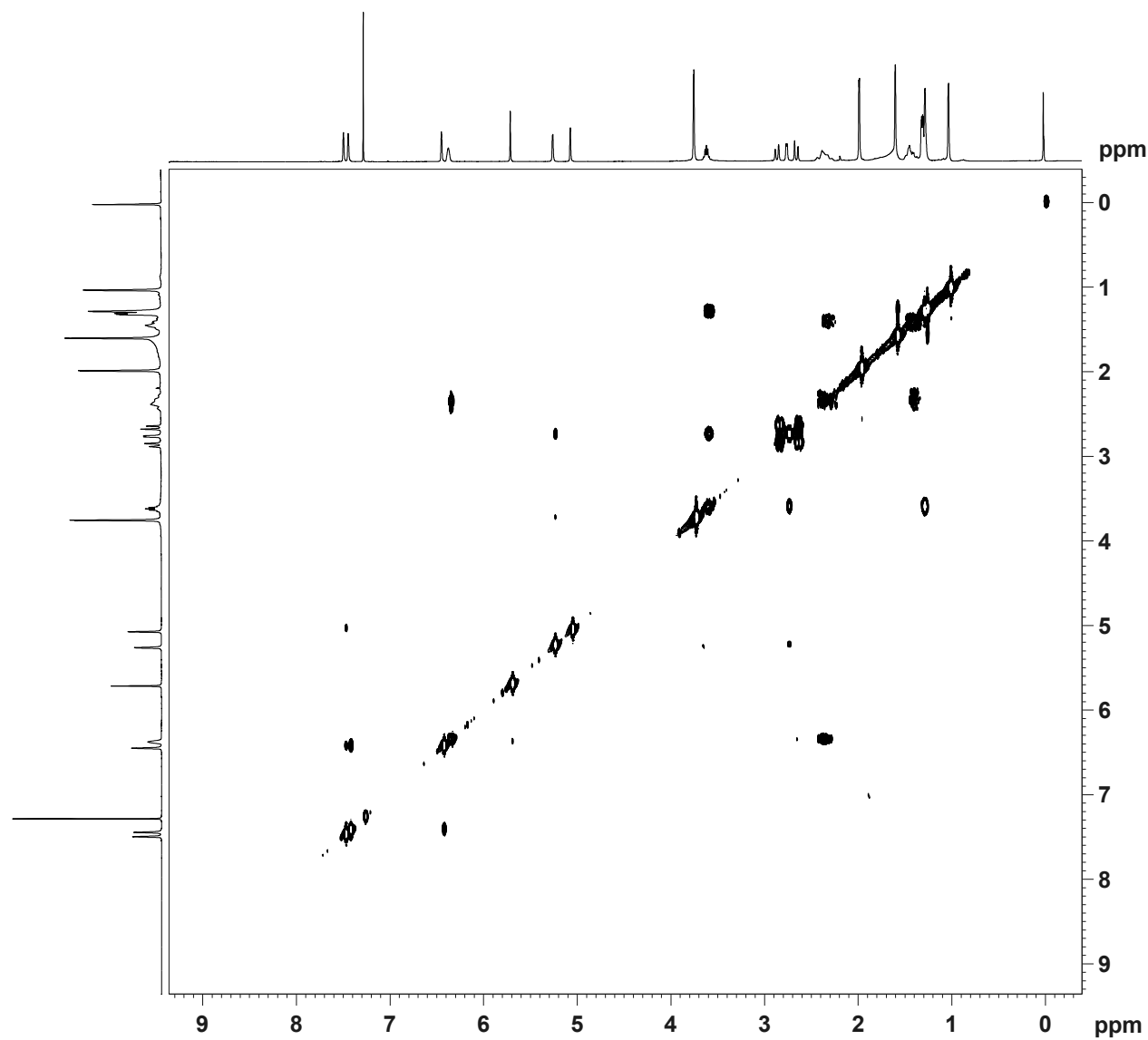
DEPT135 (100 MHz) spectrum of compound **3** in CDCl₃



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



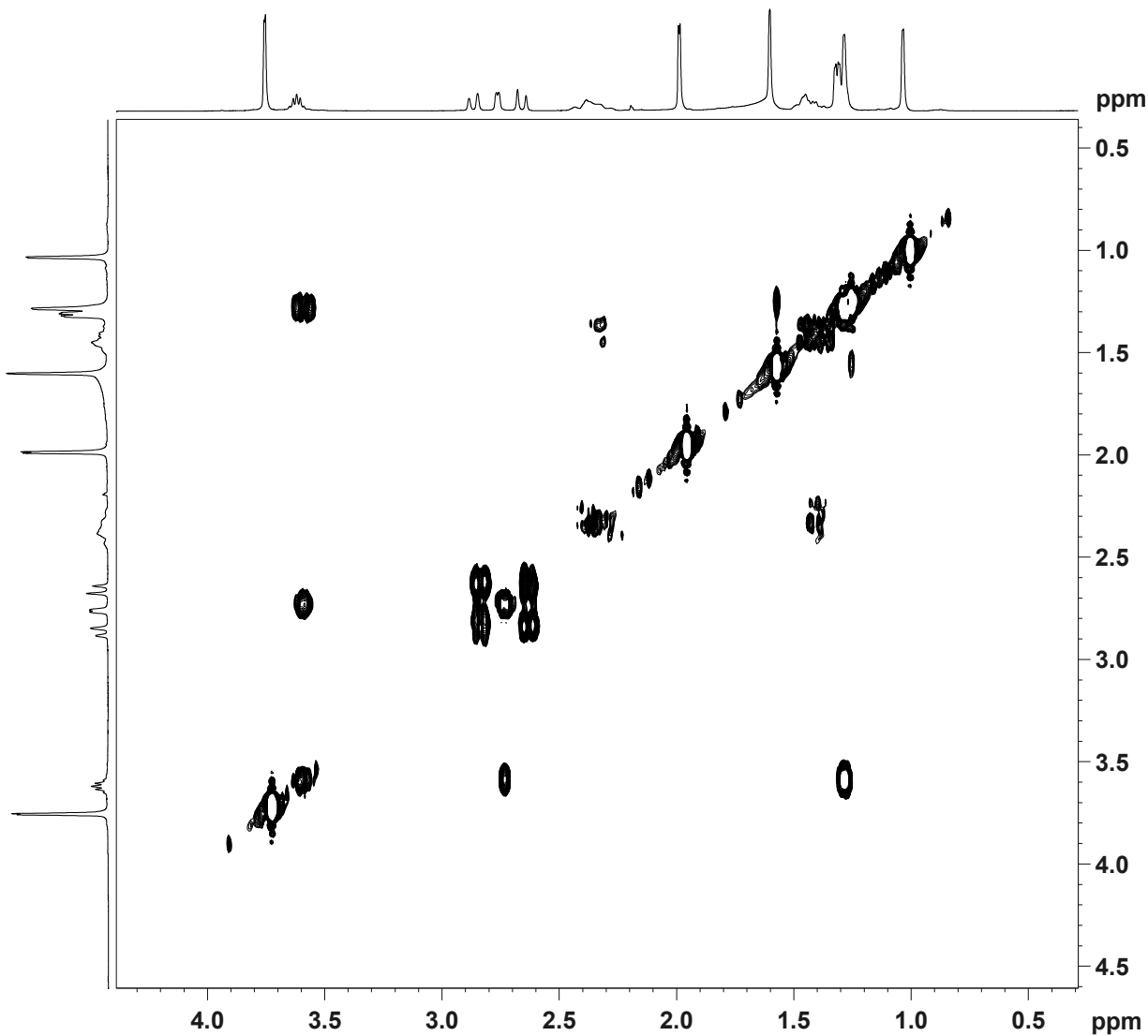
^1H - ^1H COSY (400 MHz) spectrum of compound **3** in CDCl_3



```
NAME          dyg-8-COSY
EXPNO         104
PROCNO        1
Date_         20160528
Time          8.30
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            117.37
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
INO           0.00025600 sec
```

```
===== CHANNEL f1 =====
SF01         400.1318006 MHz
NUC1          1H
P0            11.50 usec
P1            11.50 usec
P17           2500.00 usec
ND0           1
TD            128
SF01         400.1318 MHz
FIDRES        30.517578 Hz
SW            9.762 ppm
FhMODE        QF
SI            1024
SF           400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF           400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
```


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



```
NAME          dyg-8-COSY
EXPNO         104
PROCNO        1
Date_         20160528
Time_         8.30
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            117.37
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
INO           0.00025600 sec
```

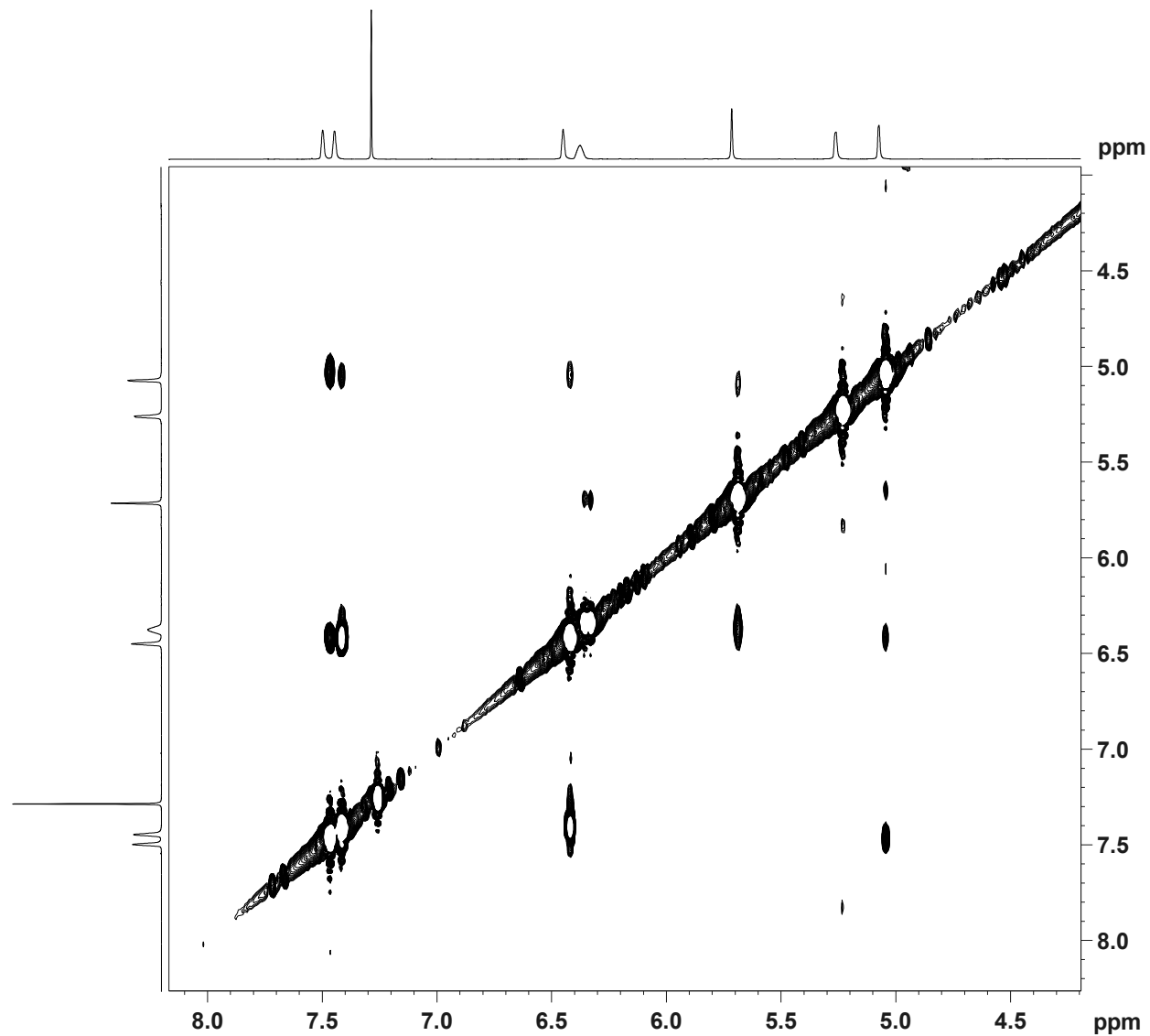
```
===== CHANNEL f1 =====
SF01         400.1318006 MHz
NUC1          1H
P0            11.50 usec
P1            11.50 usec
P17           2500.00 usec
ND0           1
TD            128
SF01         400.1318 MHz
FIDRES        30.517578 Hz
SW            9.762 ppm
FhMODE        QF
SI            1024
SF            400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            400.1300098 MHz
WDW           QSINE
SSB           0
LB            0.00 Hz
GB            0
```

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



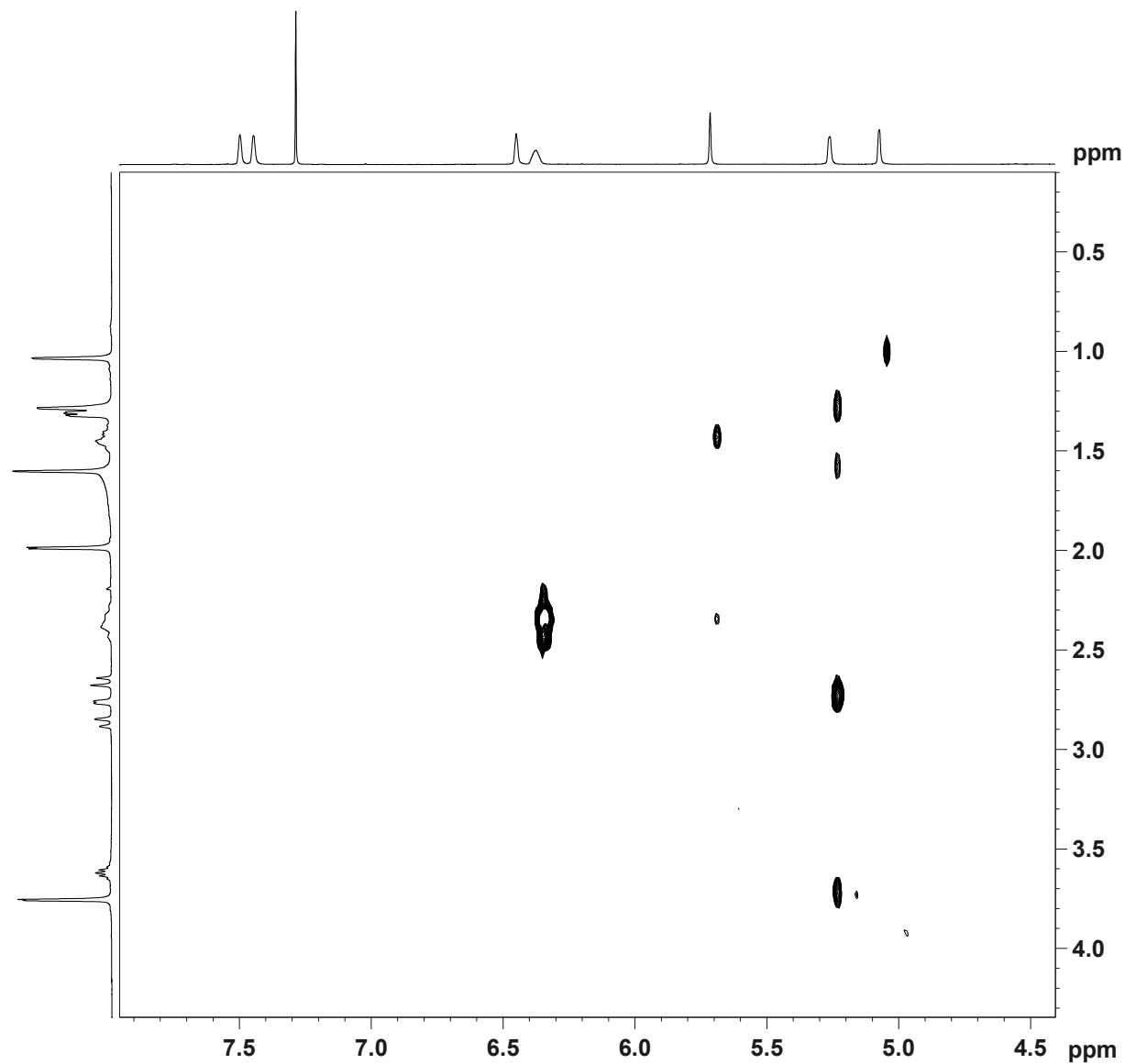
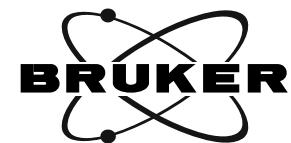
```
NAME      dyg-8-COSY
EXPNO     104
PROCNO    1
Date_     20160528
Time      8.30
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   cosygpppqf
TD        2048
SOLVENT   CDCl3
NS        32
DS        8
SWH       3906.250 Hz
FIDRES    1.907349 Hz
AQ        0.2621940 sec
RG        117.37
DW        128.000 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 =====
SF01    400.1318006 MHz
NUC1     1H
P0       11.50 usec
P1       11.50 usec
P17     2500.00 usec
ND0      1
TD       128
SF01    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
```



S70

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

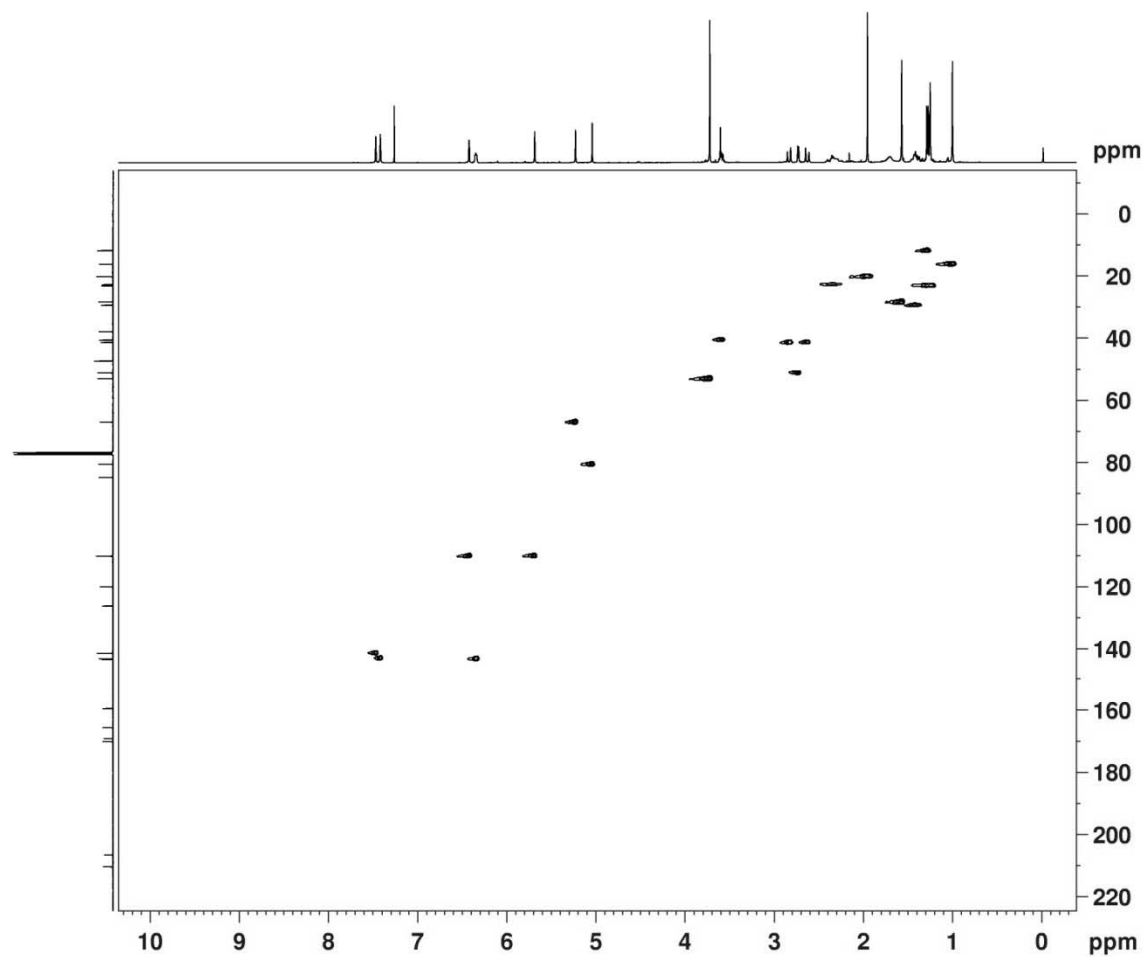


```
NAME          dyg-8-COSY
EXPNO         104
PROCNO        1
Date_         20160528
Time          8.30
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       cosygpppqf
TD            2048
SOLVENT       CDCl3
NS            32
DS            8
SWH           3906.250 Hz
FIDRES        1.907349 Hz
AQ            0.2621940 sec
RG            117.37
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
FhMODE        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
```

S71

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

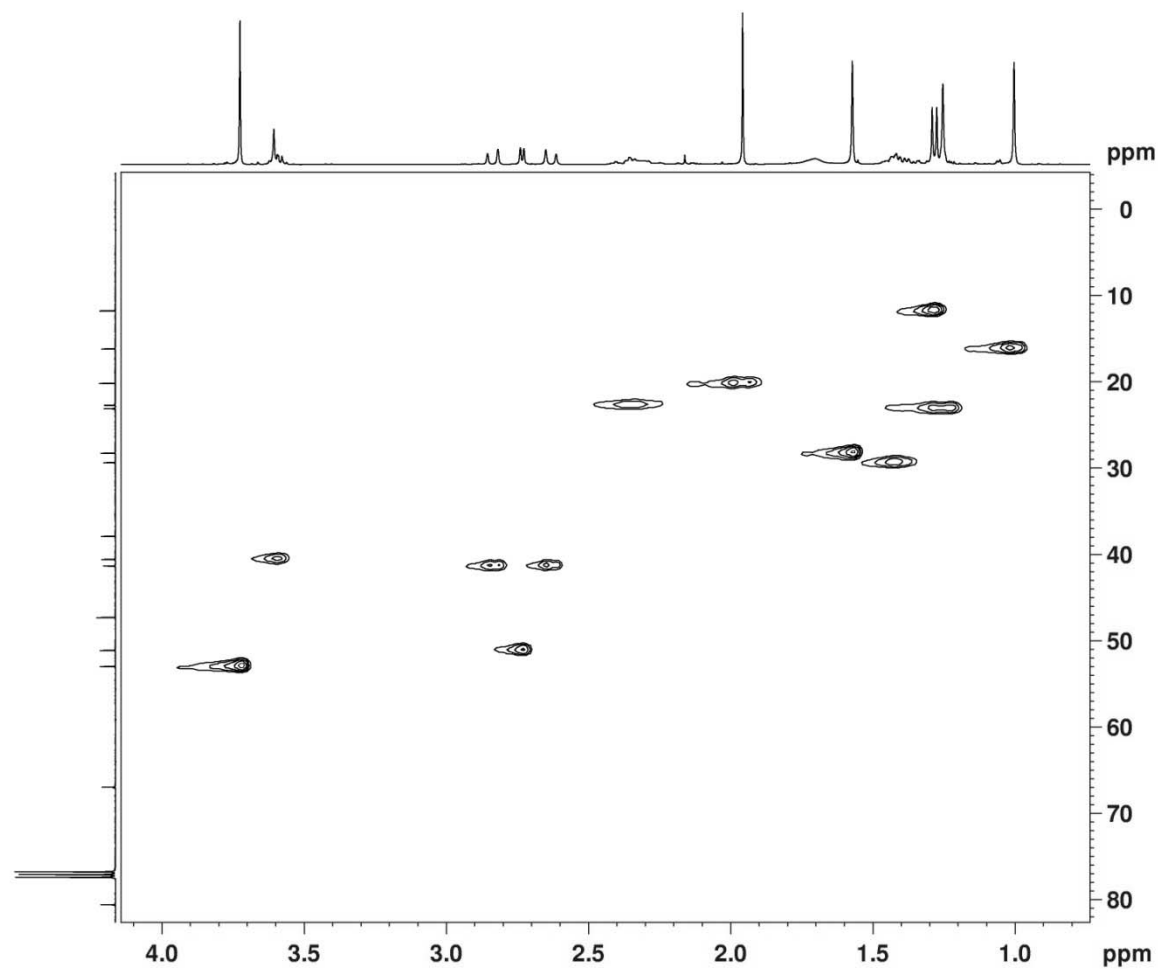


```

NAME          dyg-8
EXPNO         5
PROCNO        1
Date_         20151028
Time          12.35
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgps12
TD            1024
SOLVENT       CDC13
NS            16
DS            16
SWH           4302.926 MHz
FIDRES        4.202076 MHz
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 usec
D1            1.46497905 usec
D4            0.00172414 usec
D11           0.03000000 usec
D16           0.00020000 usec
D24           0.00086207 usec
IN0           0.00002080 usec
ZGPTNS

===== CHANNEL f1 =====
SF01          400.1320007 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
P28           0.00 usec
ND0           2
TD            256
SF01          100.6233 MHz
FIDRES        93.900238 MHz
SW            238.896 MHz
FnMODE        Echo-Antiecho
SI            1024
SF            400.1300079 MHz
WDW           QSINE
SSB           2
LB            0.00 MHz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            100.6127685 MHz
WDW           QSINE
  
```

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



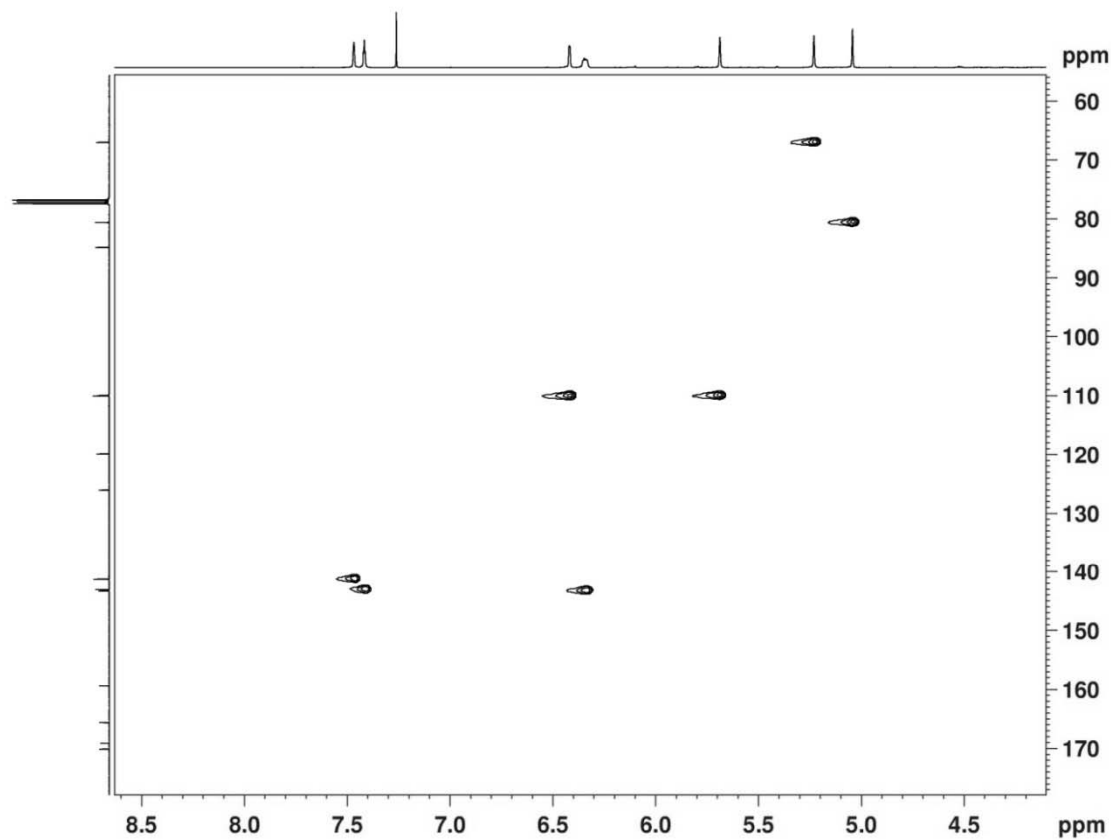
```

NAME          dyg-8
EXPNO         5
PROCNO        1
Date_         20151028
Time          12.35
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS            16
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 sec
RG            208.5
DW            116.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
D0            0.00000300 sec
D1            1.46497905 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IN0           0.00002080 sec
ZGPTNS
    
```

```

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

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

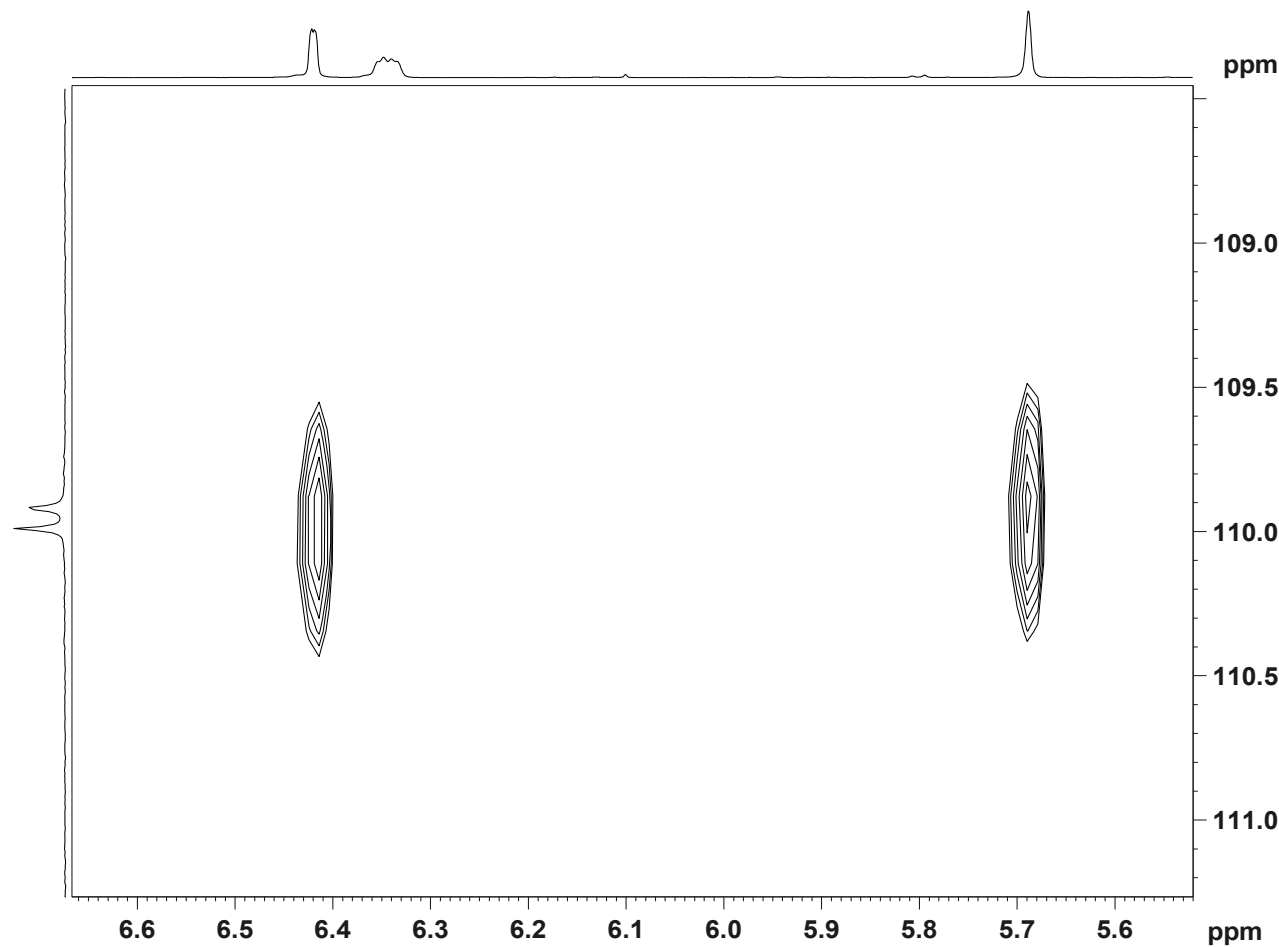


```

NAME          dyg-8
EXPNO         5
PROCNO        1
Date_         20151028
Time          12.35
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetpsi2
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
ZGPTINS

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

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

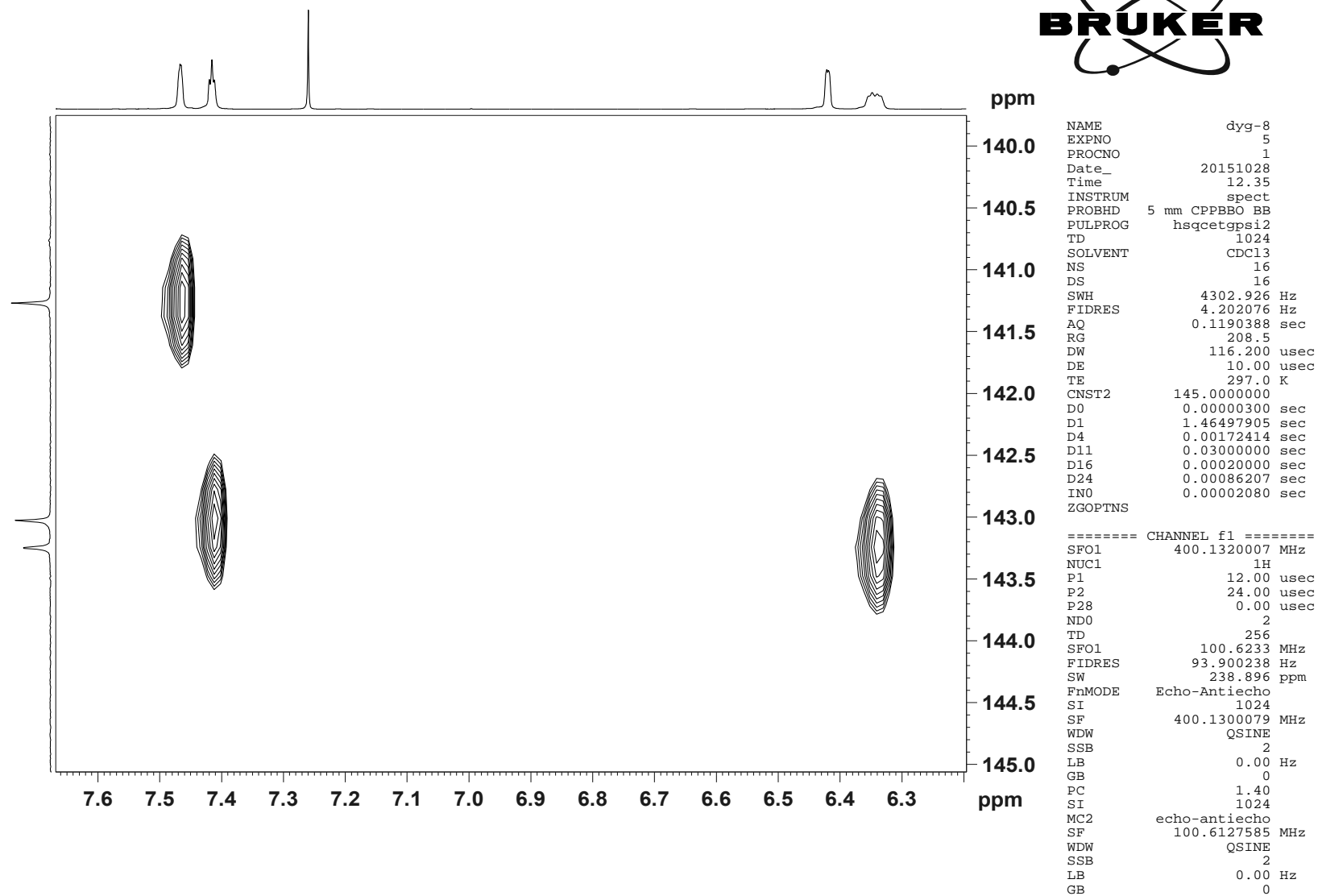
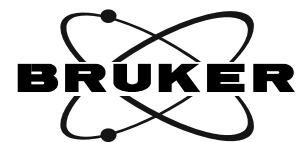


```

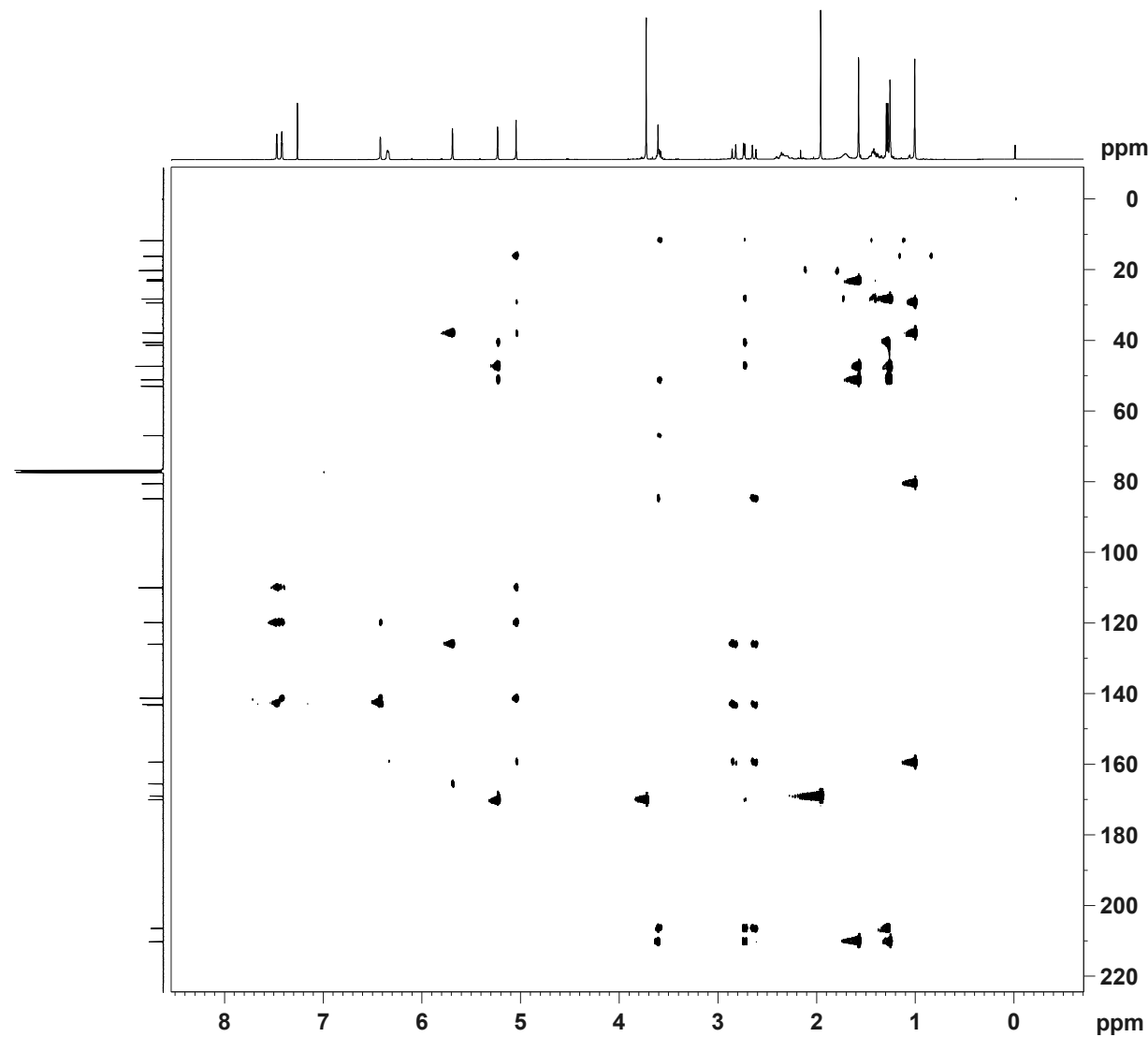
NAME          dyg-8
EXPNO         5
PROCNO        1
Date_         20151028
Time_         12.35
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hsqcetgpsi2
TD            1024
SOLVENT       CDCl3
NS            16
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            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            12.00 usec
P2            24.00 usec
P28           0.00 usec
ND0           2
TD            256
SFO1          100.6233 MHz
FIDRES        93.900238 Hz
SW            238.896 ppm
FnMODE        Echo-Antiecho
SI            1024
SF            400.1300079 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            100.6127585 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

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



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



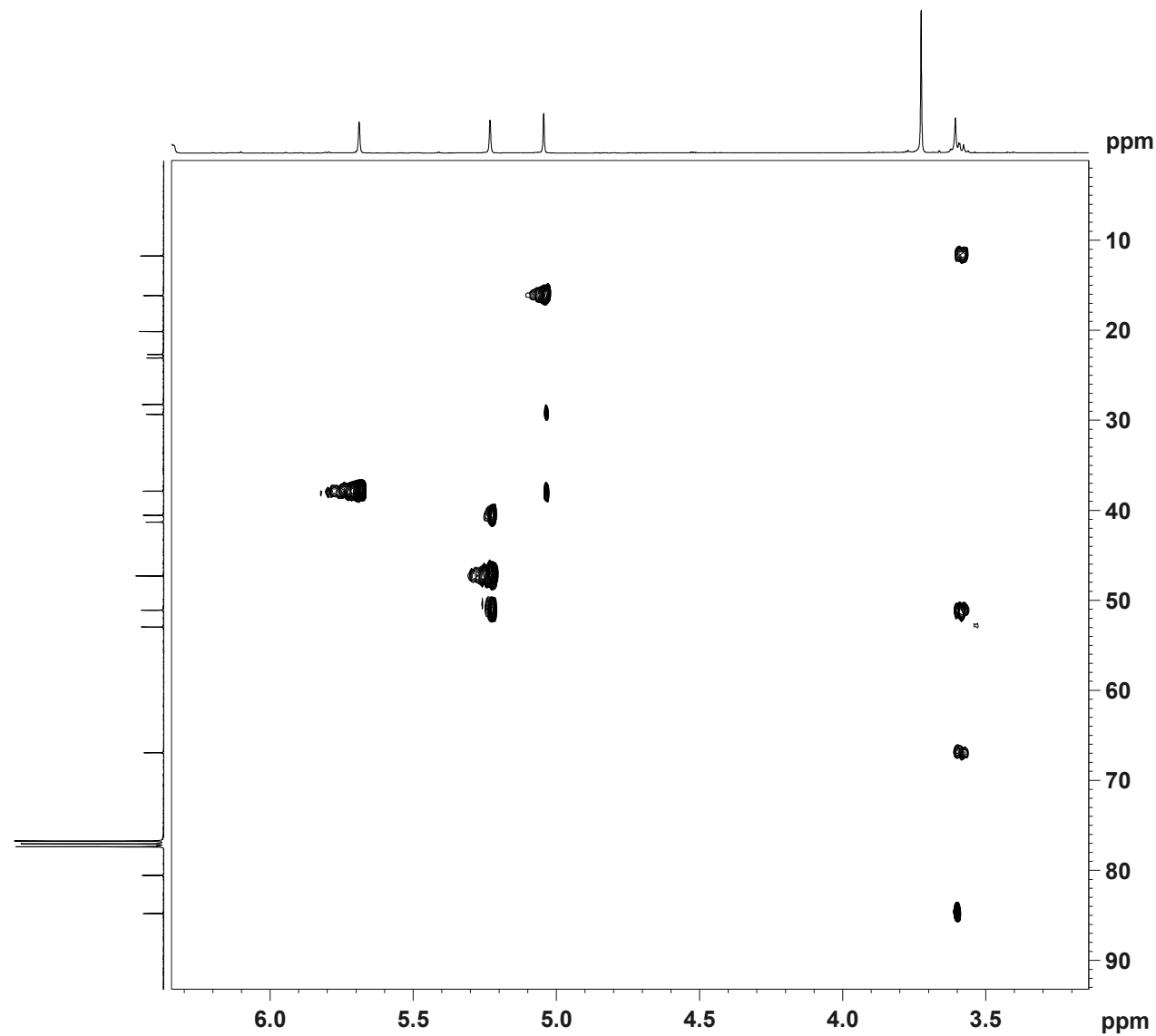
```

NAME          dyg-8
EXPNO         6
PROCNO        1
Date_         20151028
Time          14.27
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

```

===== CHANNEL f1 =====
SFO1          400.1323208 MHz
NUC1           1H
P1             12.00 usec
P2             24.00 usec
ND0            2
TD             128
SFO1          100.6233 MHz
FIDRES         187.800476 Hz
SW             238.896 ppm
FnMODE         QF
SI             2048
SF            400.1300078 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40
SI             1024
MC2            QF
SF            100.6127685 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
    
```

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

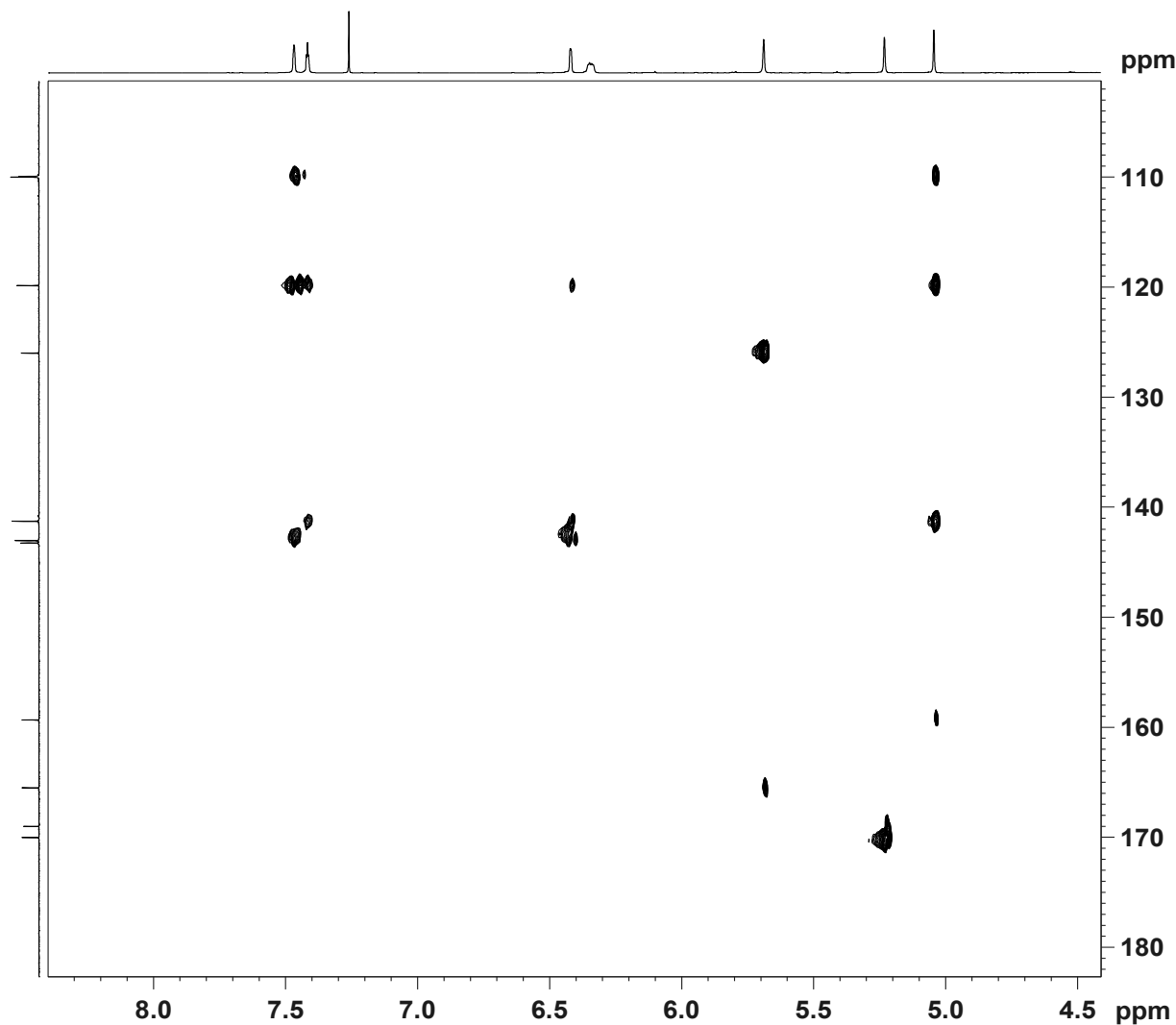


```

NAME          dyg-8
EXPNO         6
PROCNO        1
Date_         20151028
Time          14.27
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDC13
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 =====
SF01          400.1323208 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
ND0           2
TD            128
SF01          100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
FnMODE        QF
SI            2048
SF            400.1300078 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            100.6127685 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

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

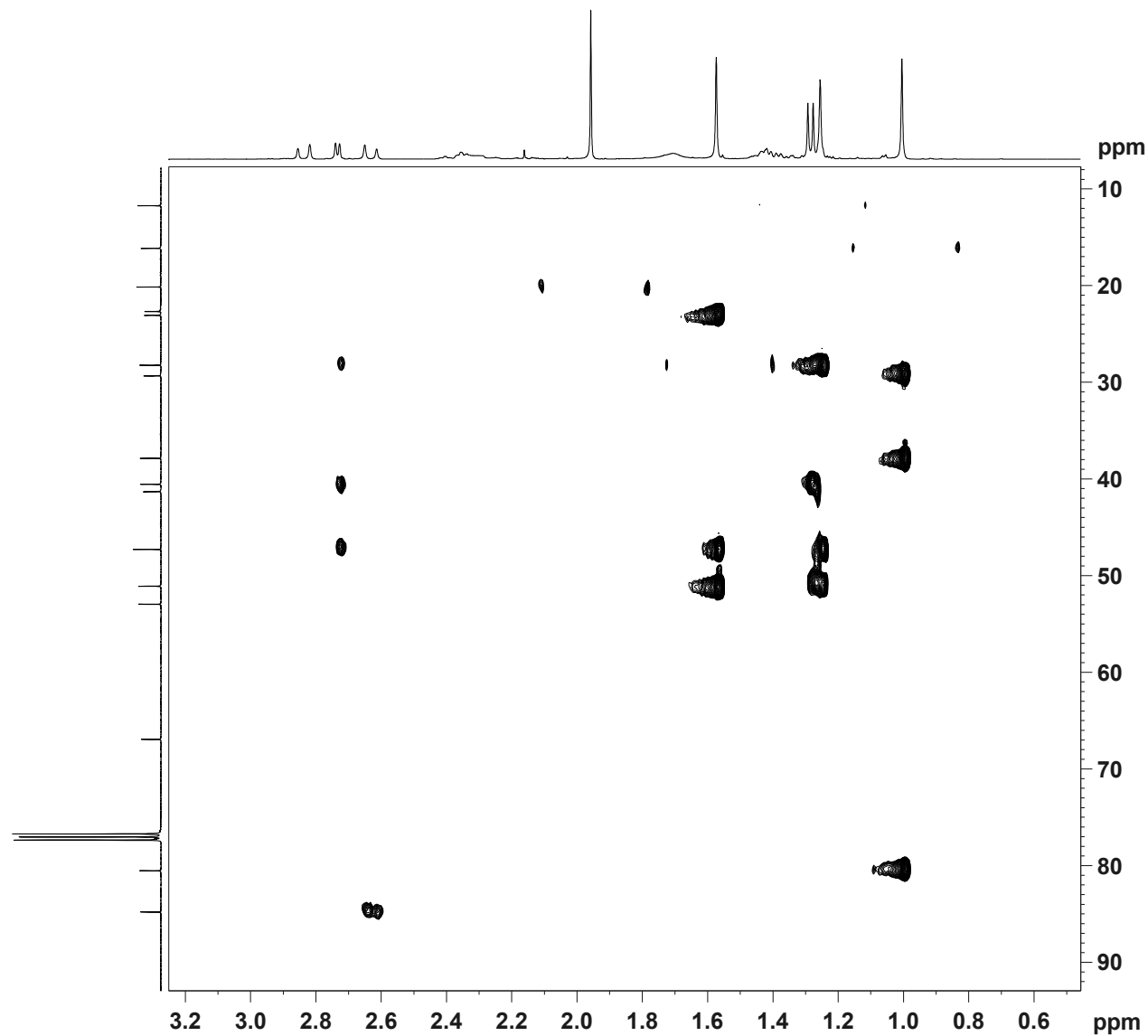


```

NAME                dyg-8
EXPNO                6
PROCNO              1
Date_               20151028
Time                14.27
INSTRUM             spect
PROBHD              5 mm CPPBBO BB
PULPROG             hmbcgp1pndqf
TD                  4096
SOLVENT             CDC13
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                    12.00 usec
P2                    24.00 usec
ND0                    2
TD                    128
SFO1                 100.6233 MHz
FIDRES               187.800476 Hz
SW                   238.896 ppm
FnMODE                QF
SI                    2048
SF                   400.1300078 MHz
WDW                   SINE
SSB                    0
LB                     0.00 Hz
GB                      0
PC                     1.40
SI                    1024
MC2                   QF
SF                   100.6127685 MHz
WDW                   SINE
SSB                    0
LB                     0.00 Hz
GB                      0
    
```

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



```

NAME          dyg-8
EXPNO         6
PROCNO        1
Date_         20151028
Time          14.27
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

```

===== CHANNEL f1 =====
SF01          400.1323208 MHz
NUC1           1H
P1             12.00 usec
P2             24.00 usec
ND0            2
TD             128
SF01          100.6233 MHz
FIDRES         187.800476 Hz
SW             238.896 ppm
FnMODE         QF
SI             2048
SF             400.1300078 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40
SI             1024
MC2            QF
SF             100.6127685 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
    
```

S80

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

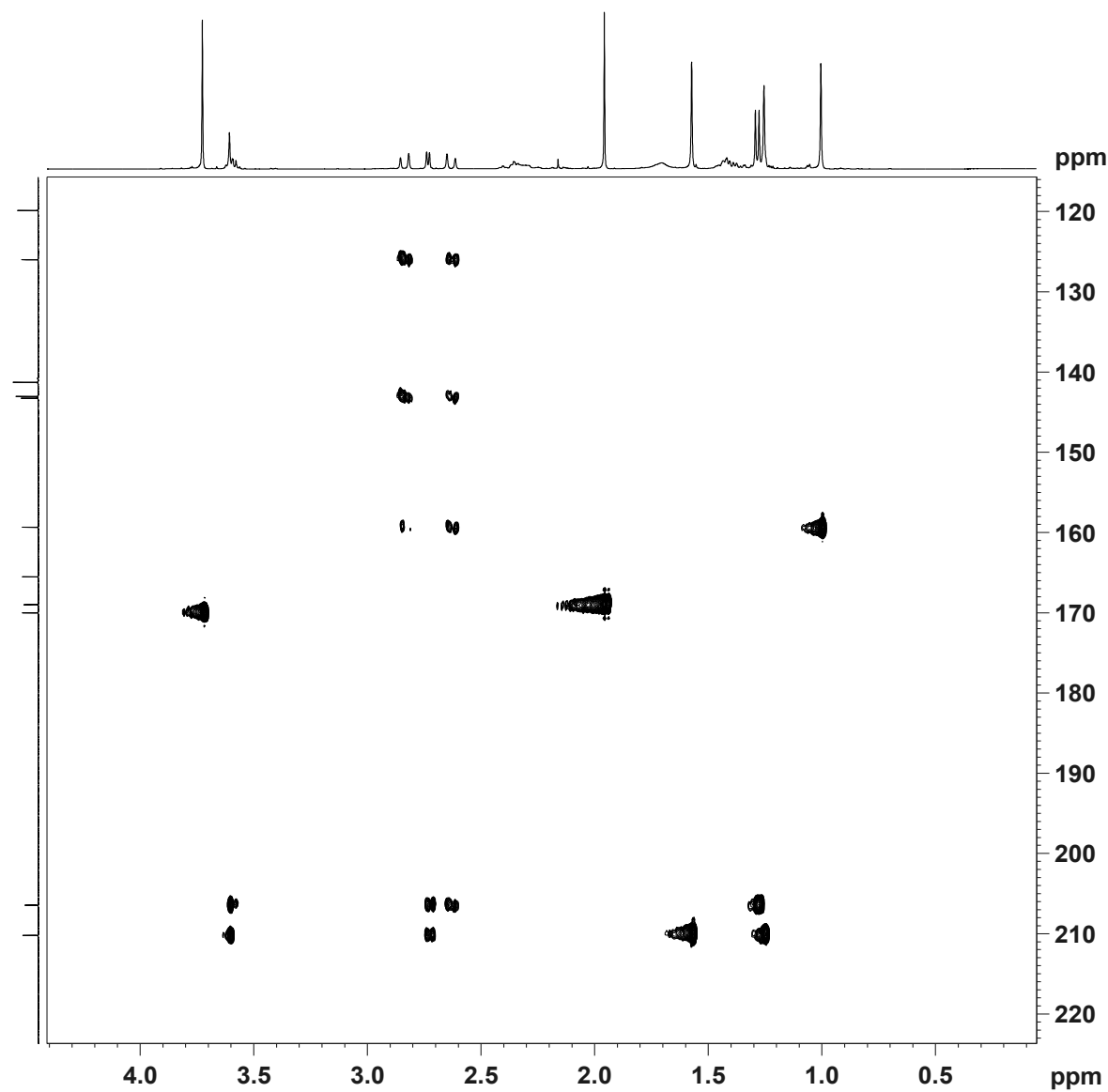


```

NAME          dyg-8
EXPNO         6
PROCNO        1
Date_         20151028
Time_         14.27
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

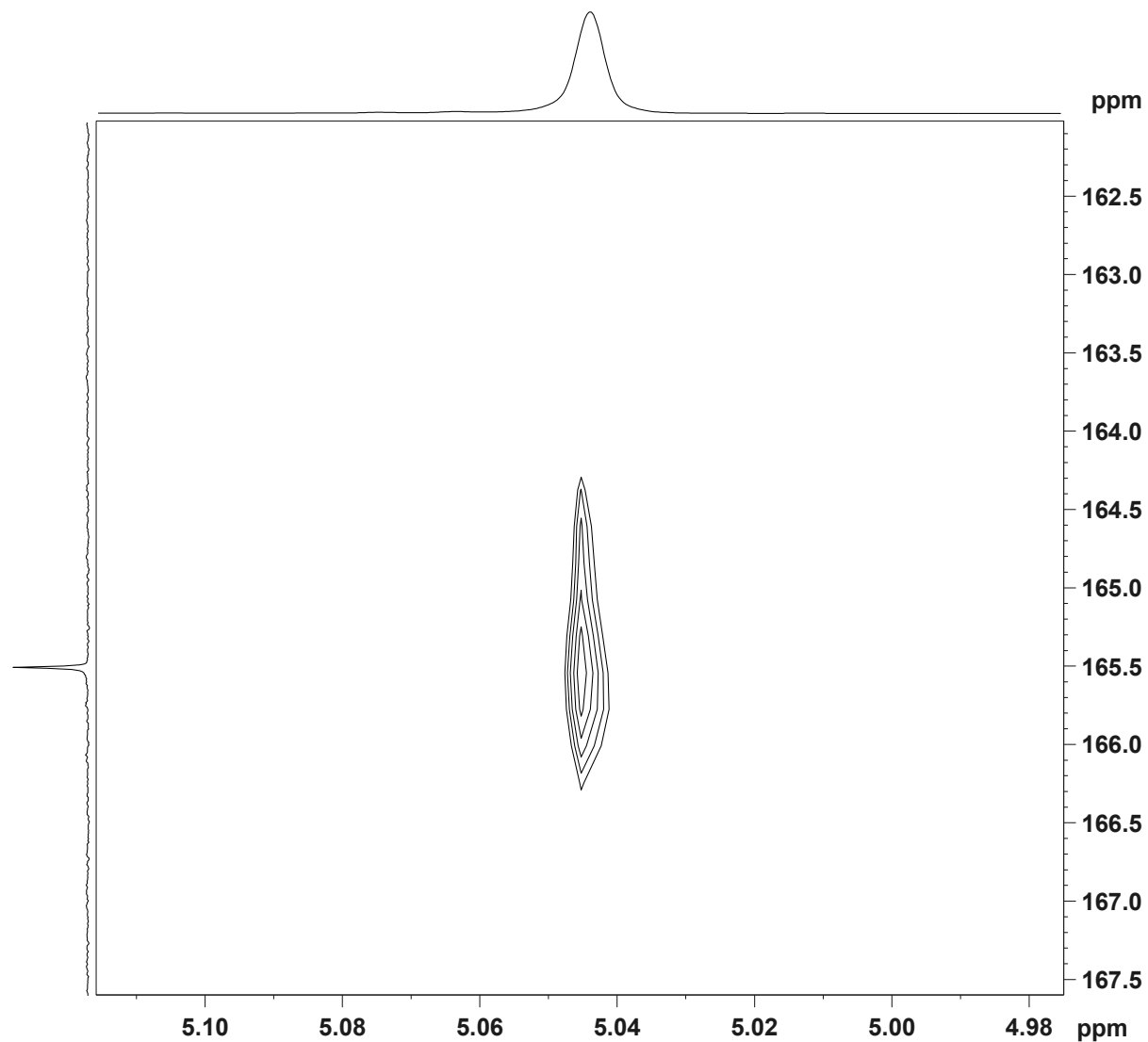
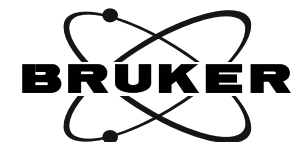
```

===== CHANNEL f1 =====
SF01         400.1323208 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
ND0           2
TD            128
SF01         100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
FnMODE        QF
SI            2048
SF            400.1300078 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            100.6127685 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```



S81

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



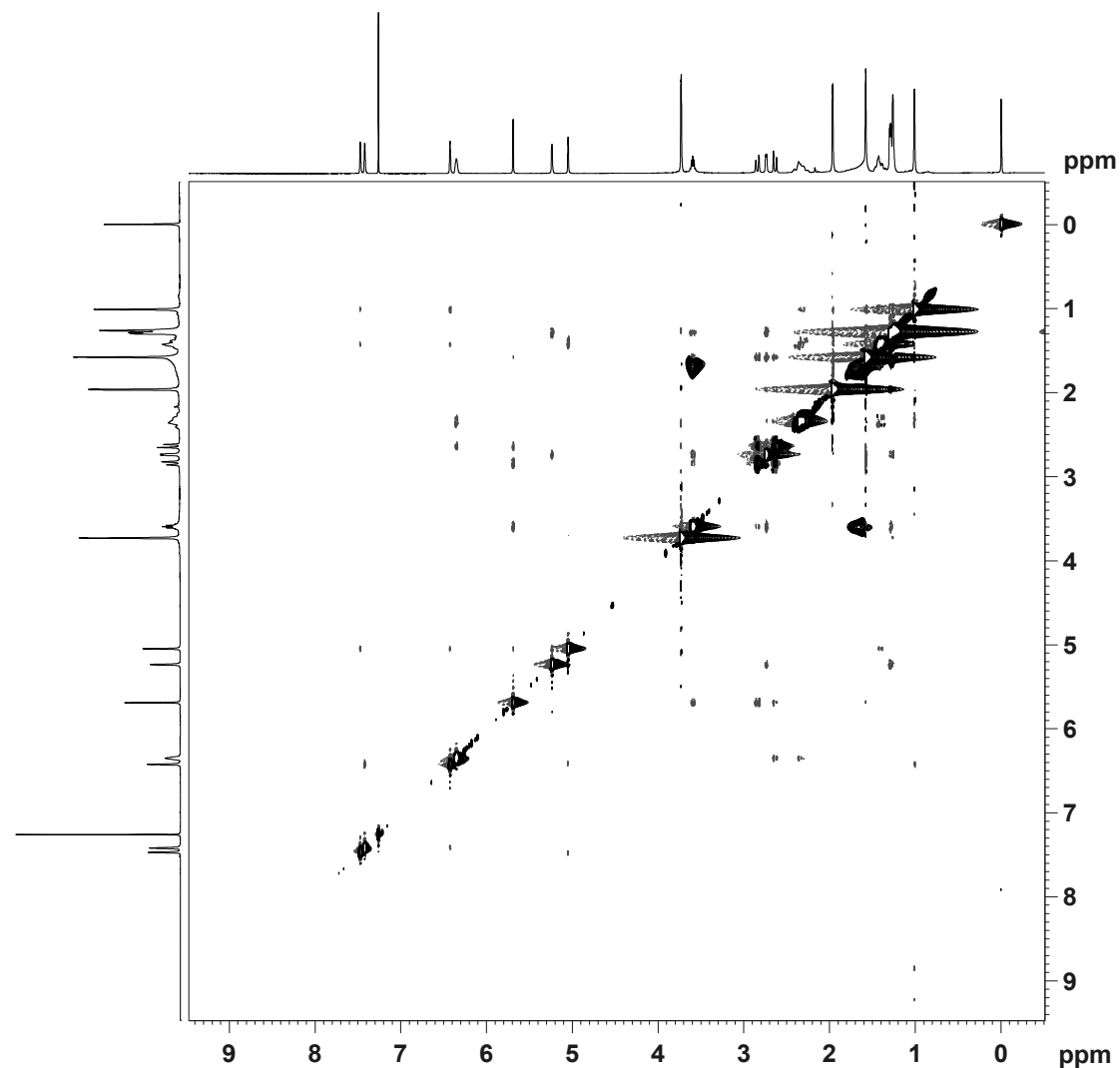
```

NAME          dyg-8
EXPNO         6
PROCNO        1
Date_         20151028
Time          14.27
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
DW            96.200 usec
DE            10.00 usec
TE            297.0 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IN0           0.00002080 sec
    
```

```

===== CHANNEL f1 =====
SFO1          400.1323208 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
ND0           2
TD            128
SFO1          100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
FnMODE        QF
SI            2048
SF            400.1300038 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            100.6127685 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

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



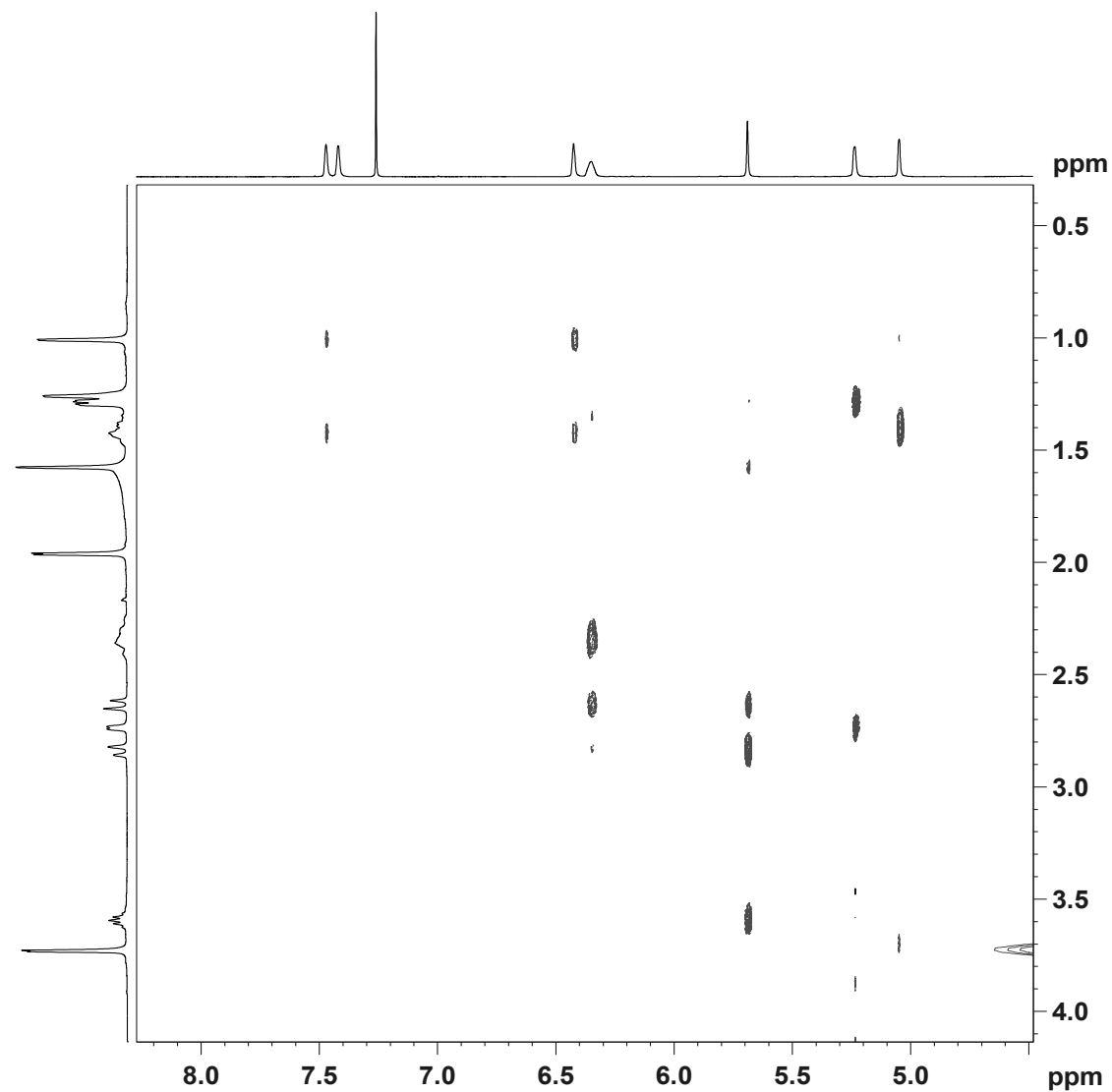
```

NAME          dyg-8-N
EXPNO         103
PROCNO        1
Date_         20160520
Time          18.13
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesyggpphp
TD            2048
SOLVENT       CDCl3
NS            64
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

===== CHANNEL f1 =====
SF01         400.1318006 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
P17           2500.00 usec
ND0           1
TD            256
SF01         400.1318 MHz
FIDRES        15.625000 Hz
SW            9.997 ppm
FnMODE        States-TPPI
SI            1024
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            400.1300098 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

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



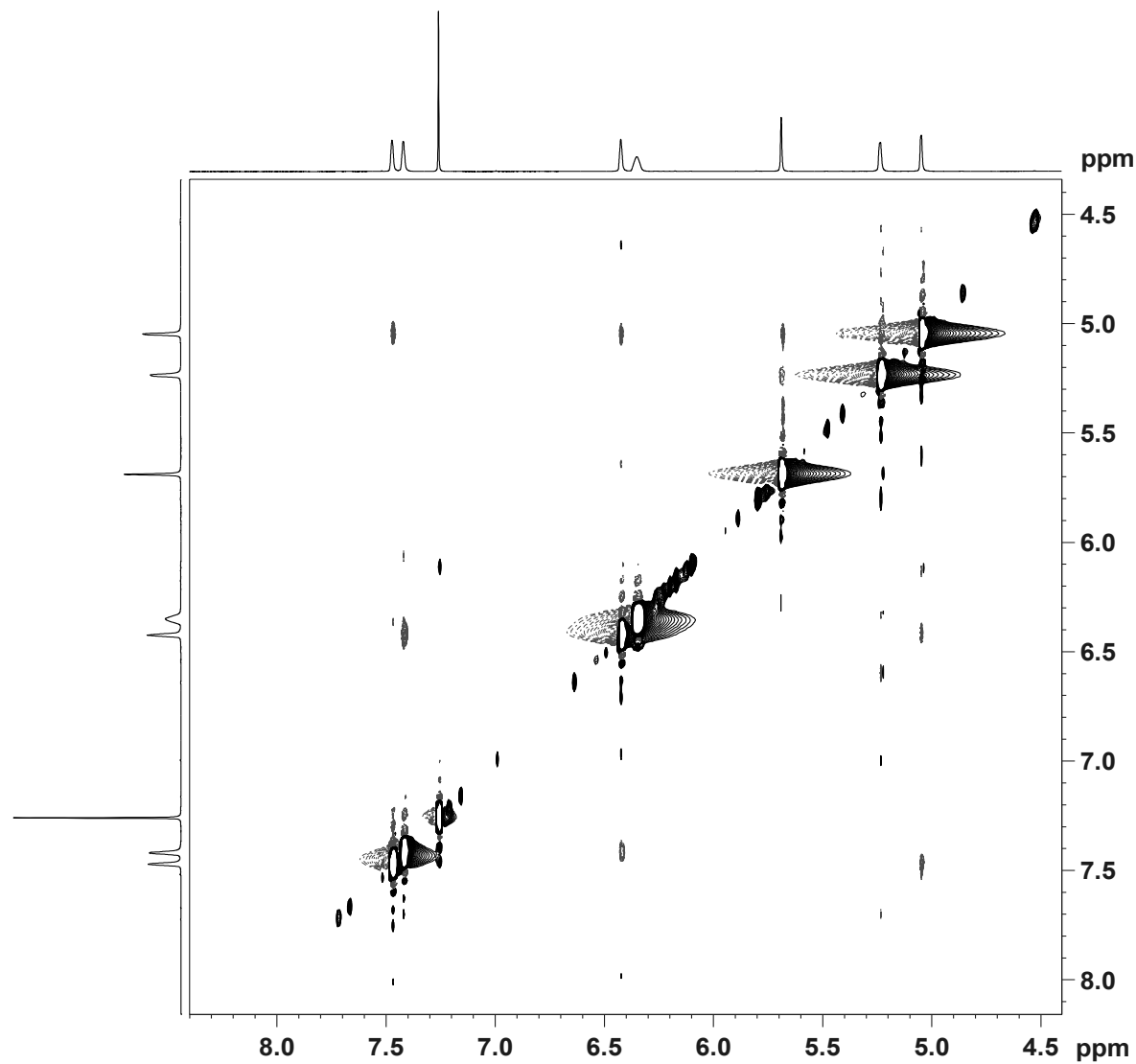
```

NAME          dyg-8-N
EXPNO         103
PROCNO        1
Date_         20160520
Time          18.13
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpphph
TD            2048
SOLVENT       CDCl3
NS            64
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

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


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



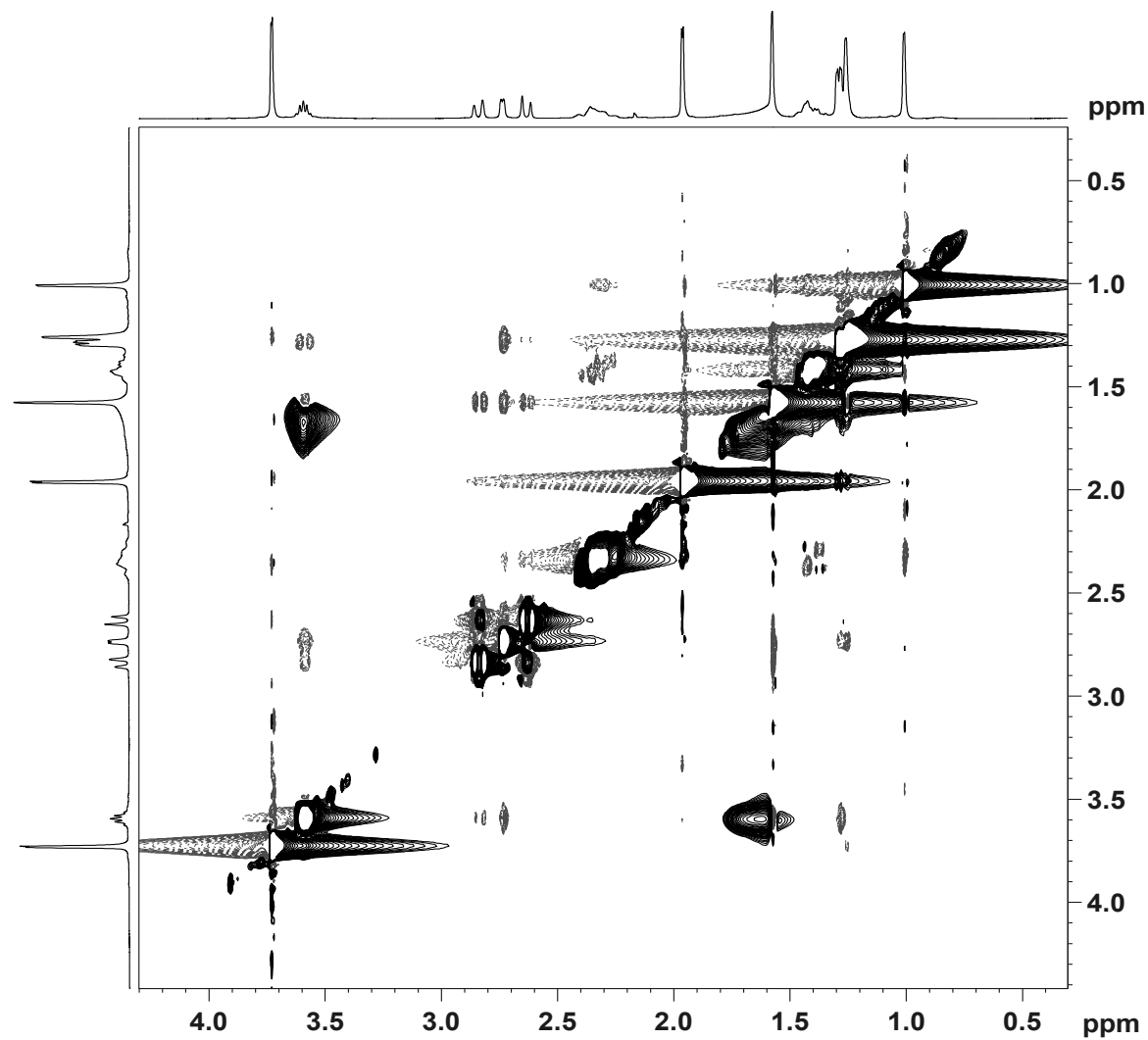
```

NAME          dyg-8-N
EXPNO         103
PROCNO        1
Date_         20160520
Time          18.13
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpphpp
TD            2048
SOLVENT       CDCl3
NS            64
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

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

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



```

NAME          dyg-8-N
EXPNO         103
PROCNO        1
Date_         20160520
Time          18.13
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesyggpphpp
TD            2048
SOLVENT       CDCl3
NS            64
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00011036 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

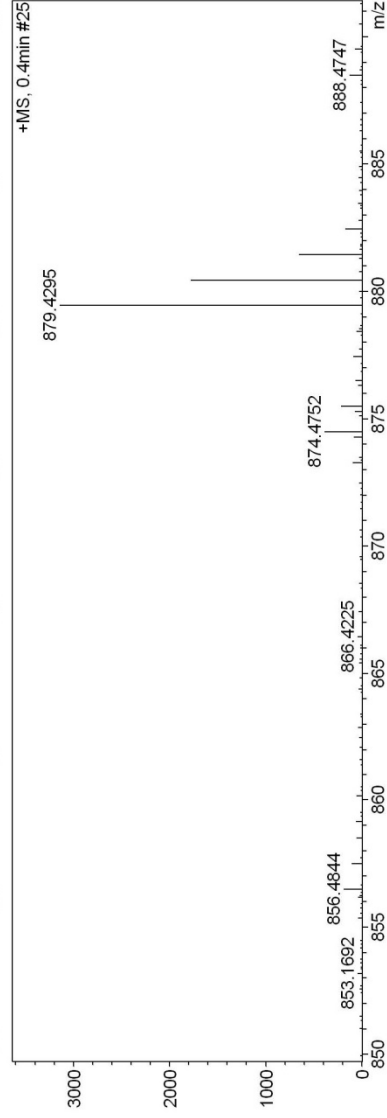
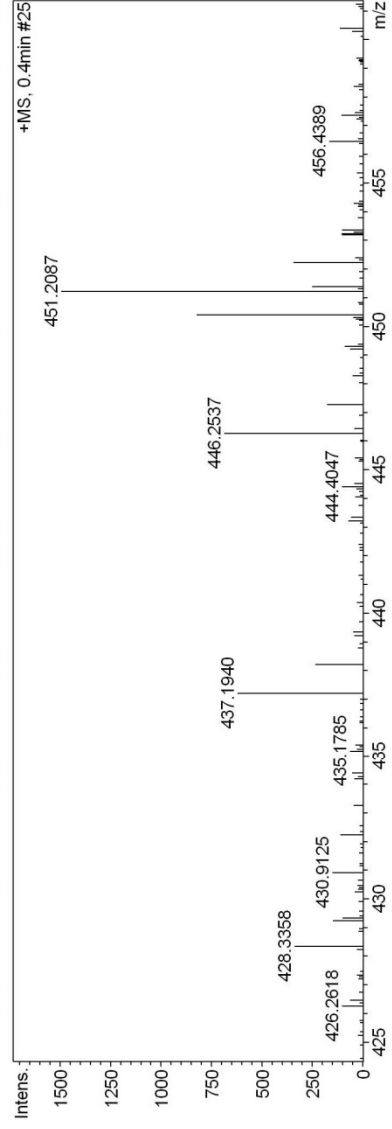
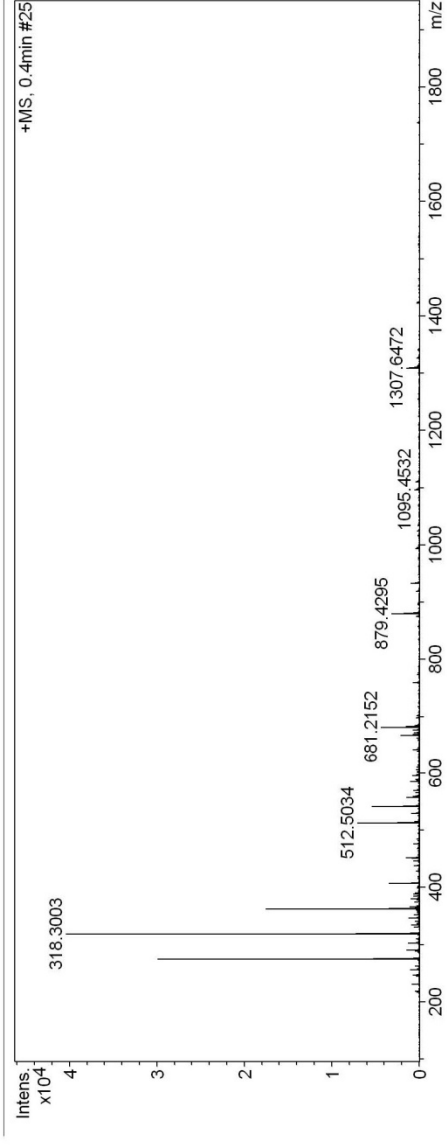
===== CHANNEL f1 =====
SFO1         400.1318006 MHz
NUC1         1H
P1           11.50 usec
P2           23.00 usec
P17          2500.00 usec
ND0          1
TD           256
SFO1         400.1318 MHz
FIDRES       15.625000 Hz
SW           9.997 ppm
FnmODE       States-TPPI
SI           1024
SF      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

HR-ESIMS for compound 4

Generic Display Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-21_pos_8_01_1856.d Acquisition Date 5/19/2016 8:44:40 AM
Method LC_Direct Infusion_pos_100-1000mz.m Operator SCSIO
Sample Name liwanshan_dgy-21_pos Instrument maXis
Comment



S87

HR-ESIMS for compound 4

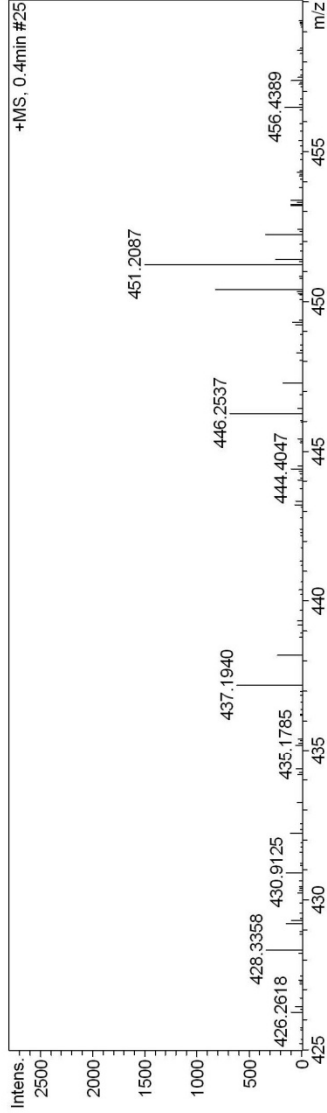
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name D:\Data\MS\data\201605\liwanshan_dgy-21_pos_8_01_1856.d Acquisition Date 5/19/2016 8:44:40 AM
 Method LC_Direct Infusion_pos_100-1000mz.m Operator SCSIO
 Sample Name liwanshan_dgy-21_pos Instrument / Ser# maXis 29
 Comment

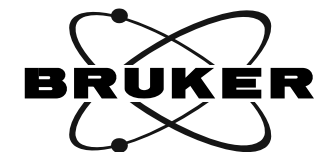
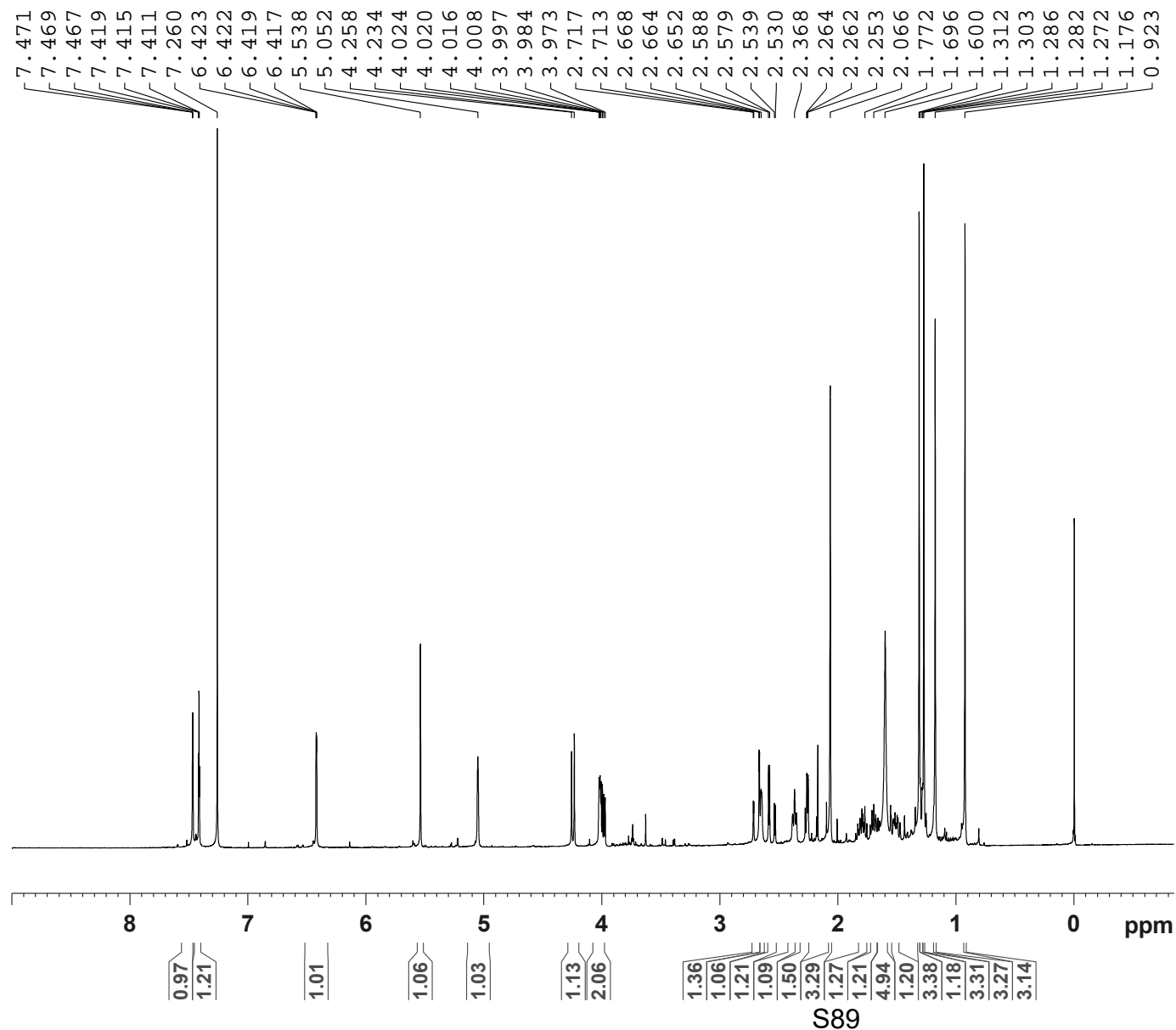
Acquisition Parameter

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



Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdB	e ₋ Conf	N-Rule
429.2271	1	C 25 H 33 O 6	100.00	429.2272	0.3	0.1	38.6	9.5	even	ok
451.2087	1	C 25 H 32 Na O 6	100.00	451.2091	1.0	0.4	28.6	9.5	even	ok
879.4295	1	C 50 H 64 Na O 12	100.00	879.4290	-0.6	-0.5	17.5	18.5	even	ok

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



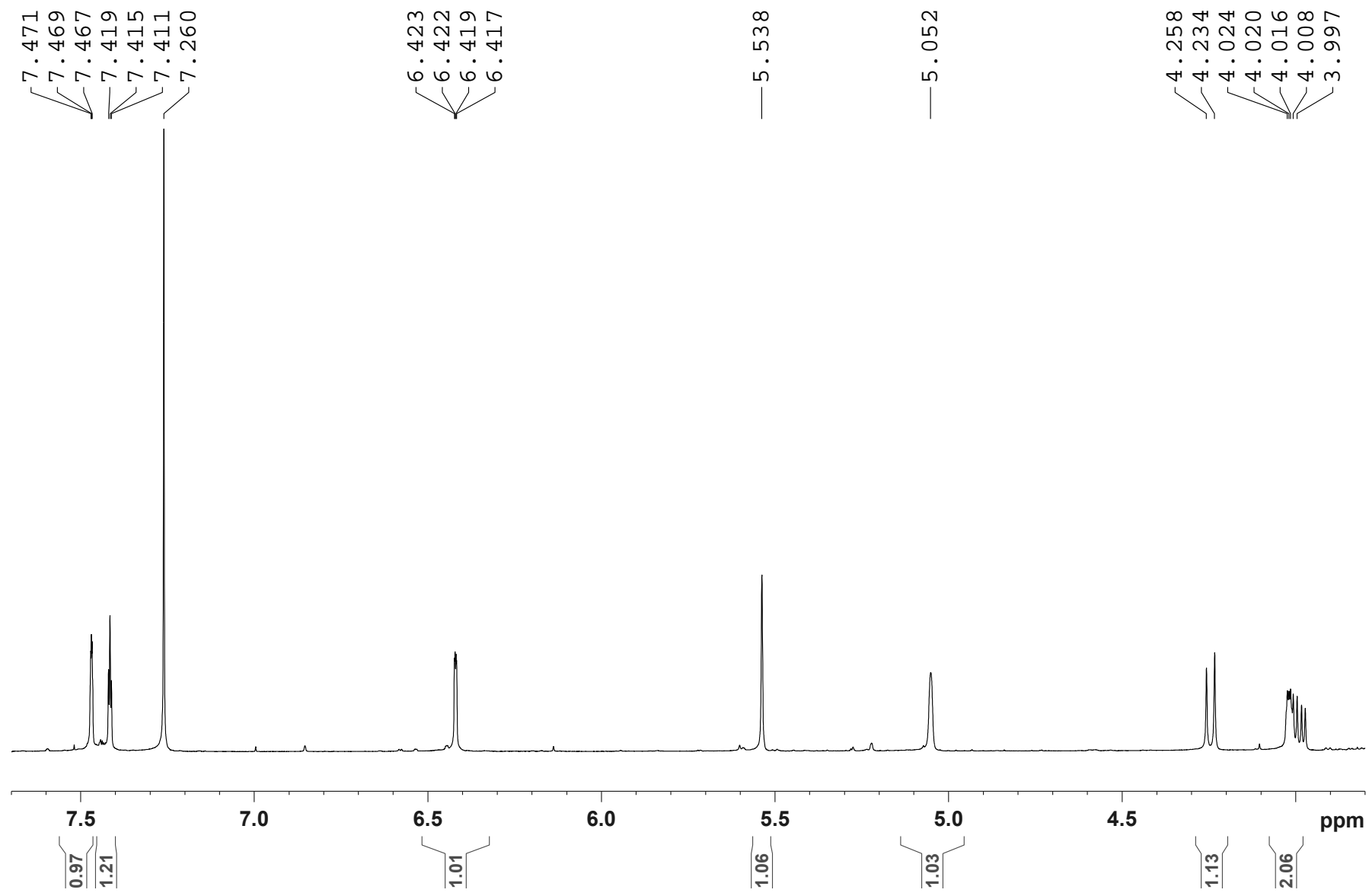
```

NAME          dyg-21
EXPNO         101
PROCNO        1
Date_         20151123
Time          9.34
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
    
```

```

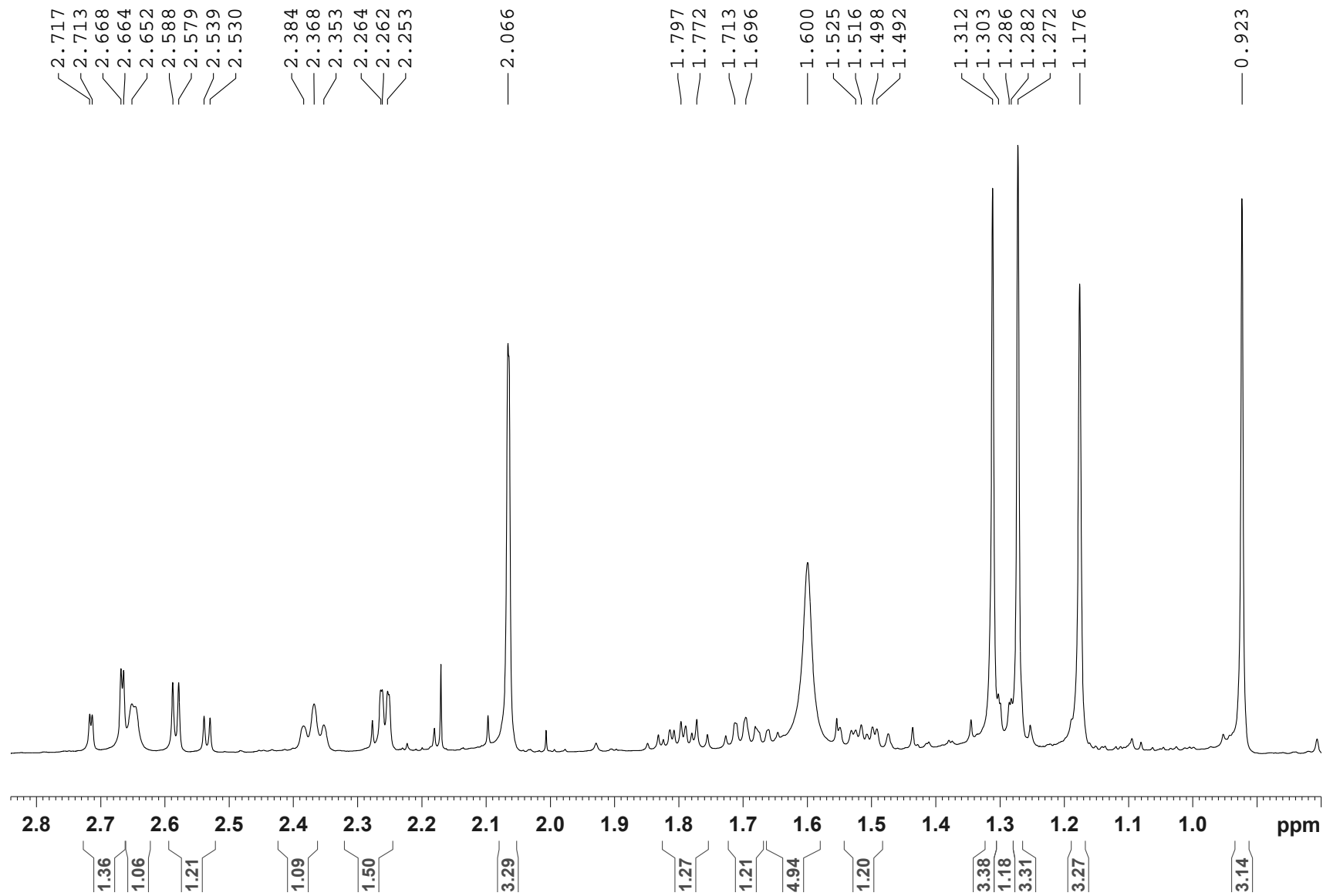
===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1          1H
P1            12.00 usec
SI            65536
SF            400.1300097 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

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

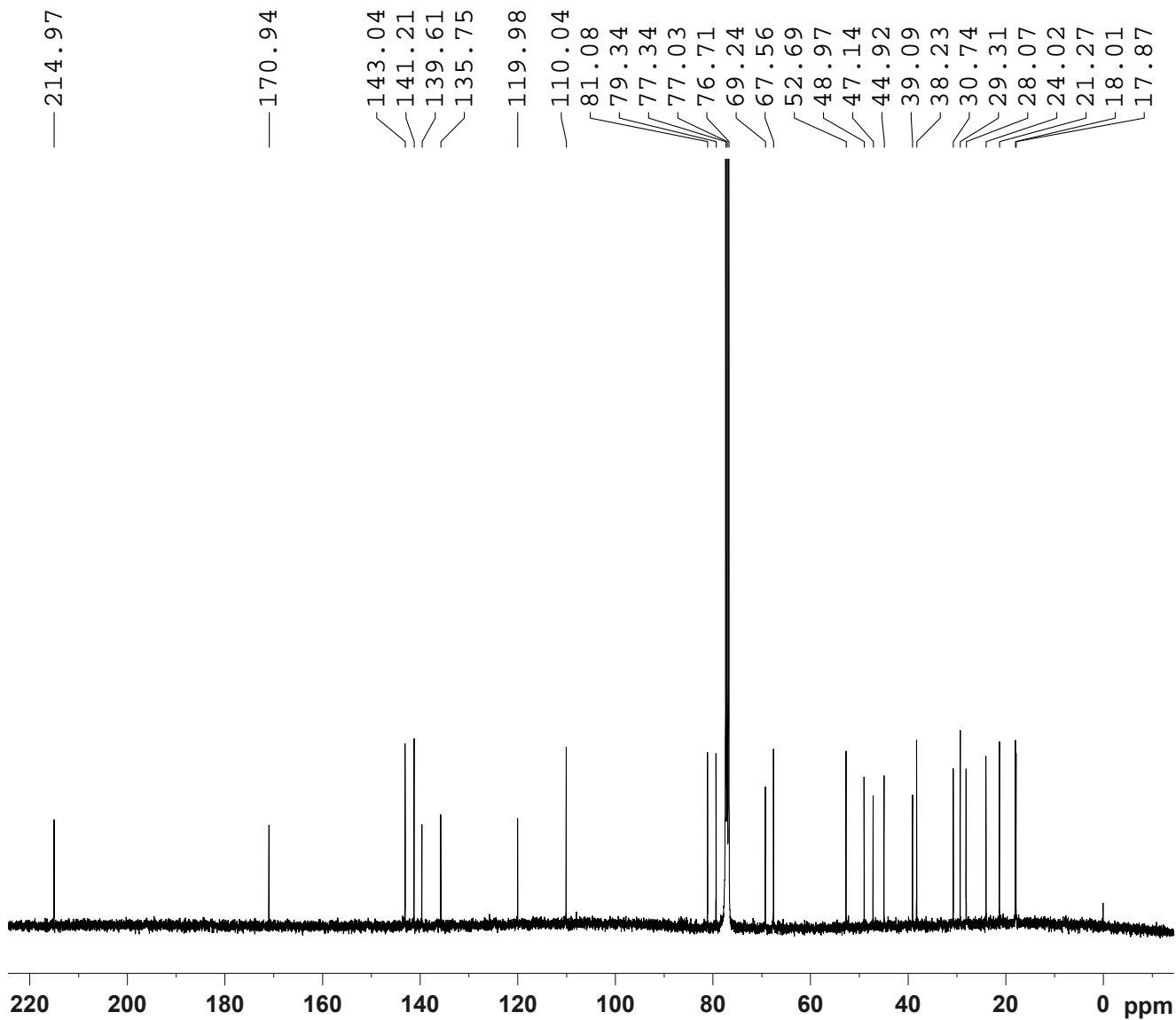


S90

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



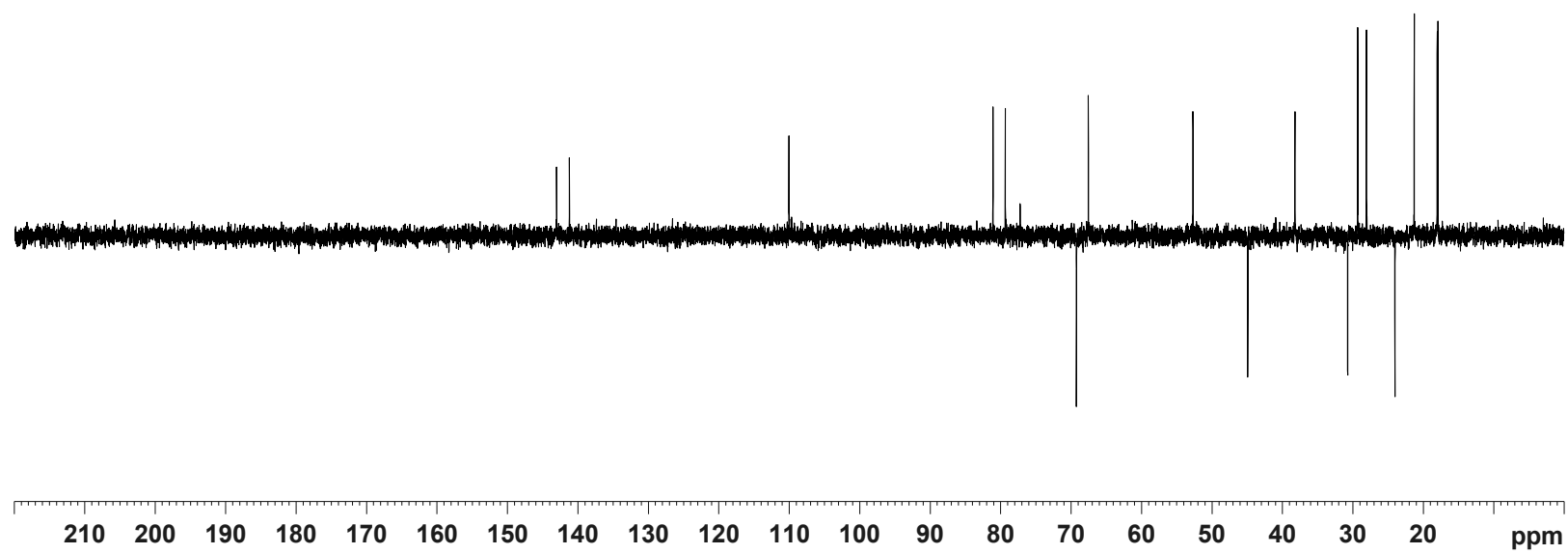
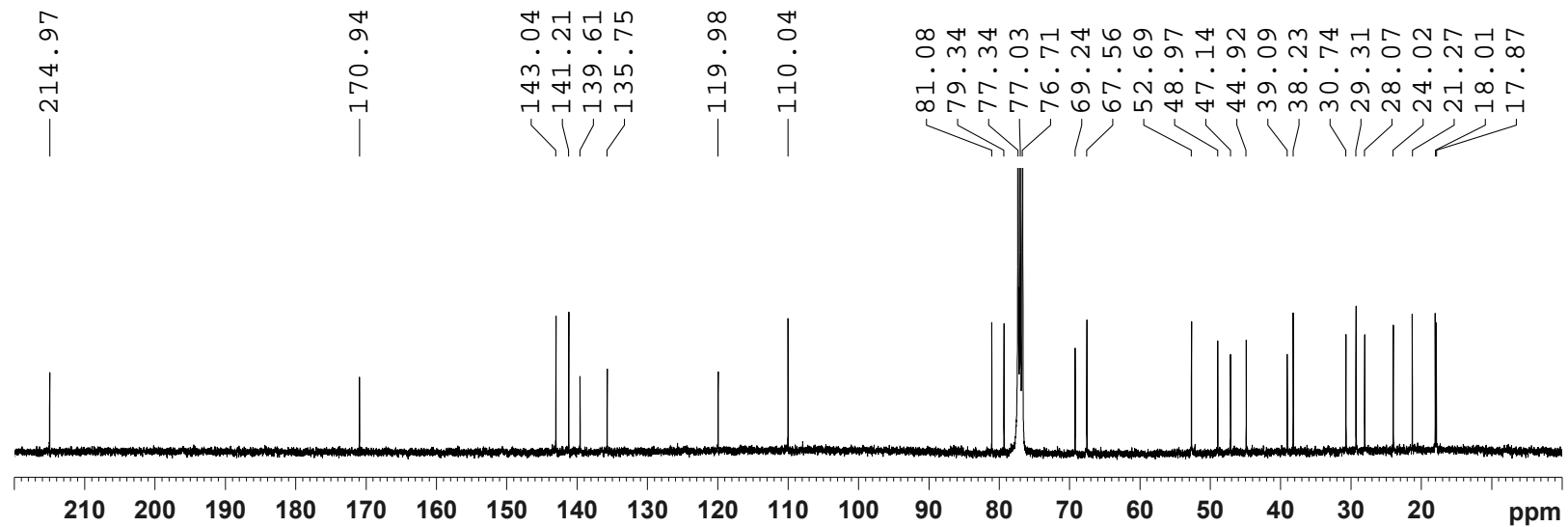
¹³C NMR (100 MHz) spectrum of compound **4** in CDCl₃



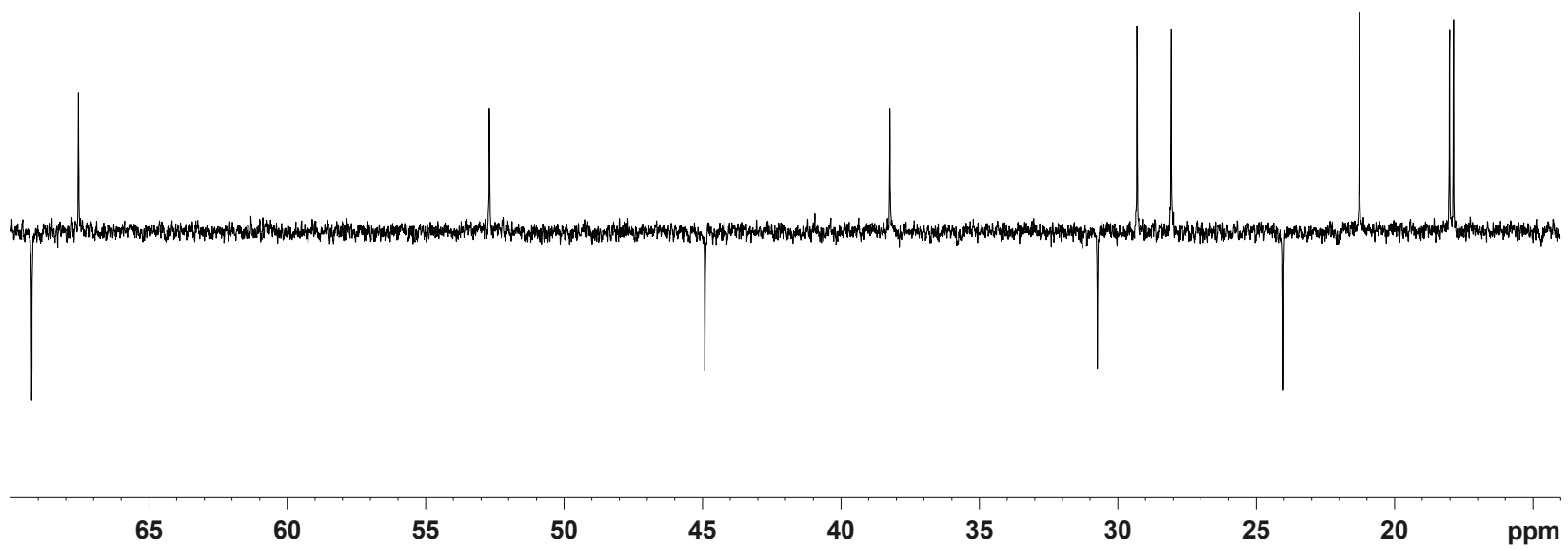
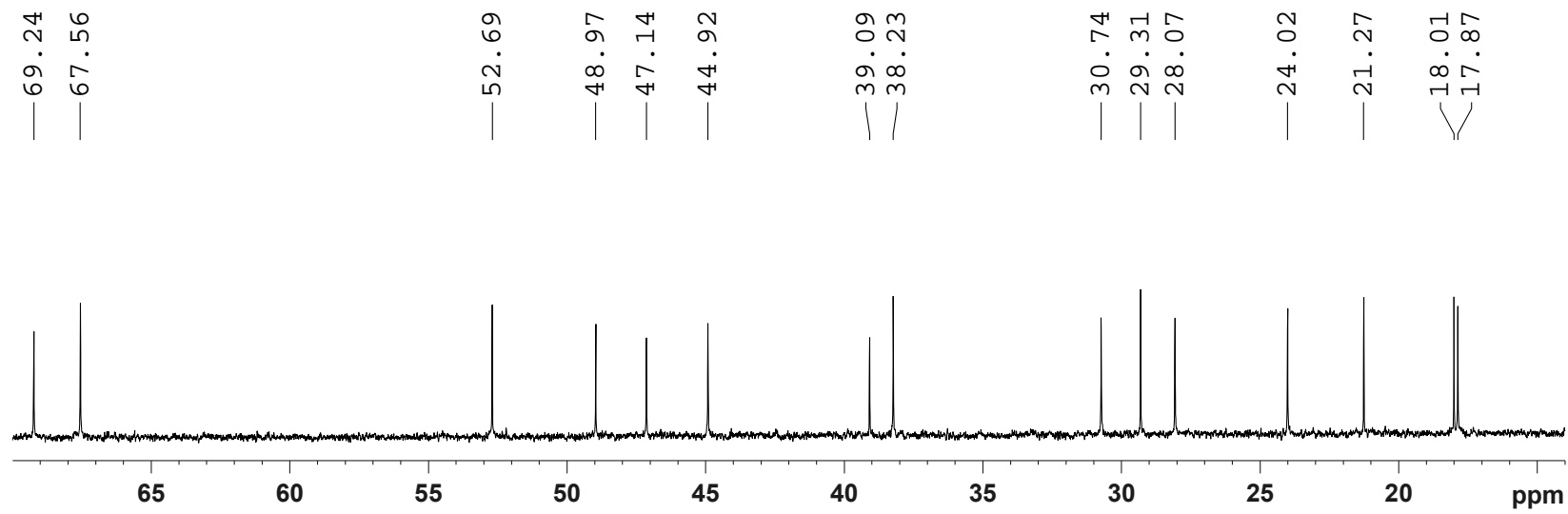
```
NAME          dyg-21
EXPNO         102
PROCNO        1
Date_         20151123
Time          11.45
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1906
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            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 =====
SF01          100.6233324 MHz
NUC1           13C
P1             10.00 usec
SI             32768
SF            100.6127684 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

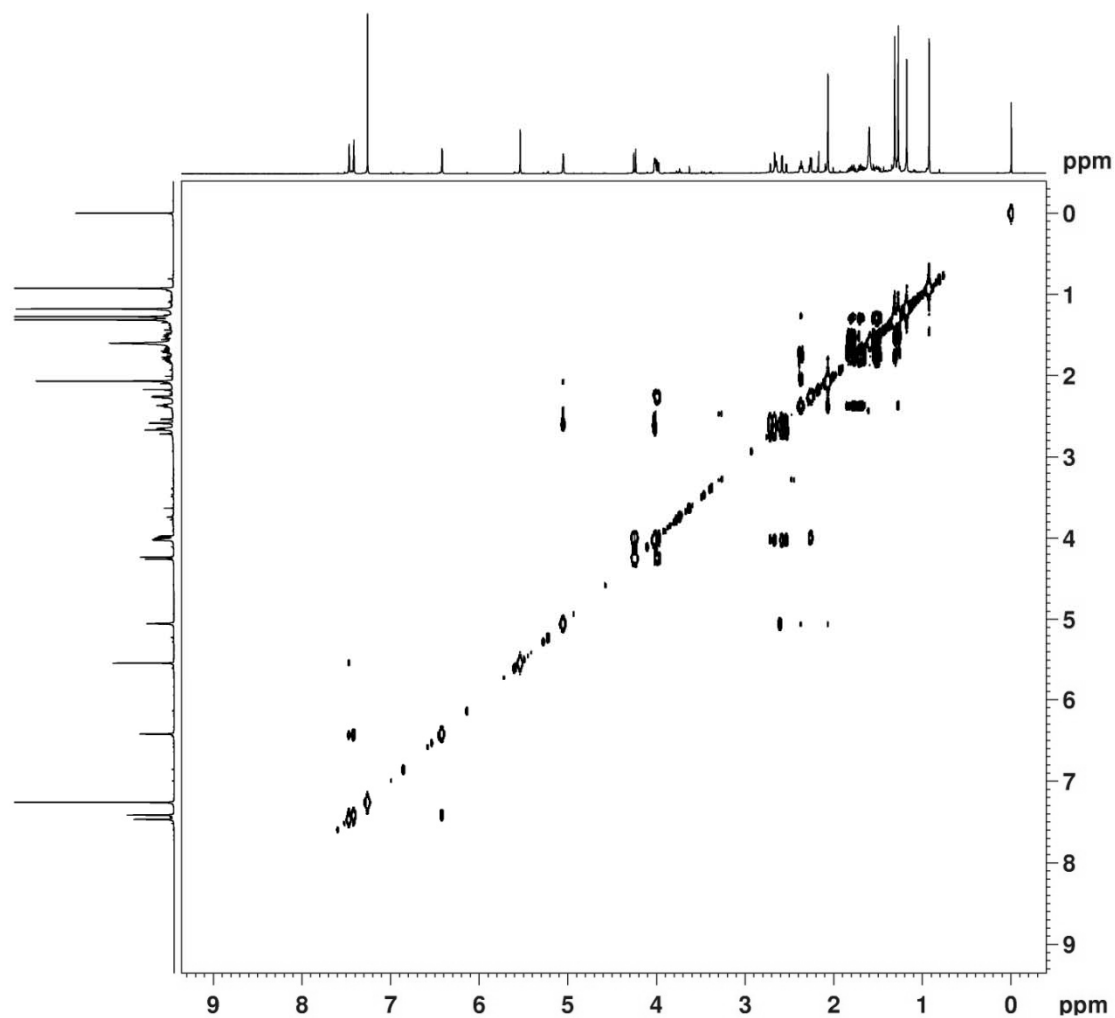

DEPT135 (100 MHz) spectrum of compound **4** in CDCl₃



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



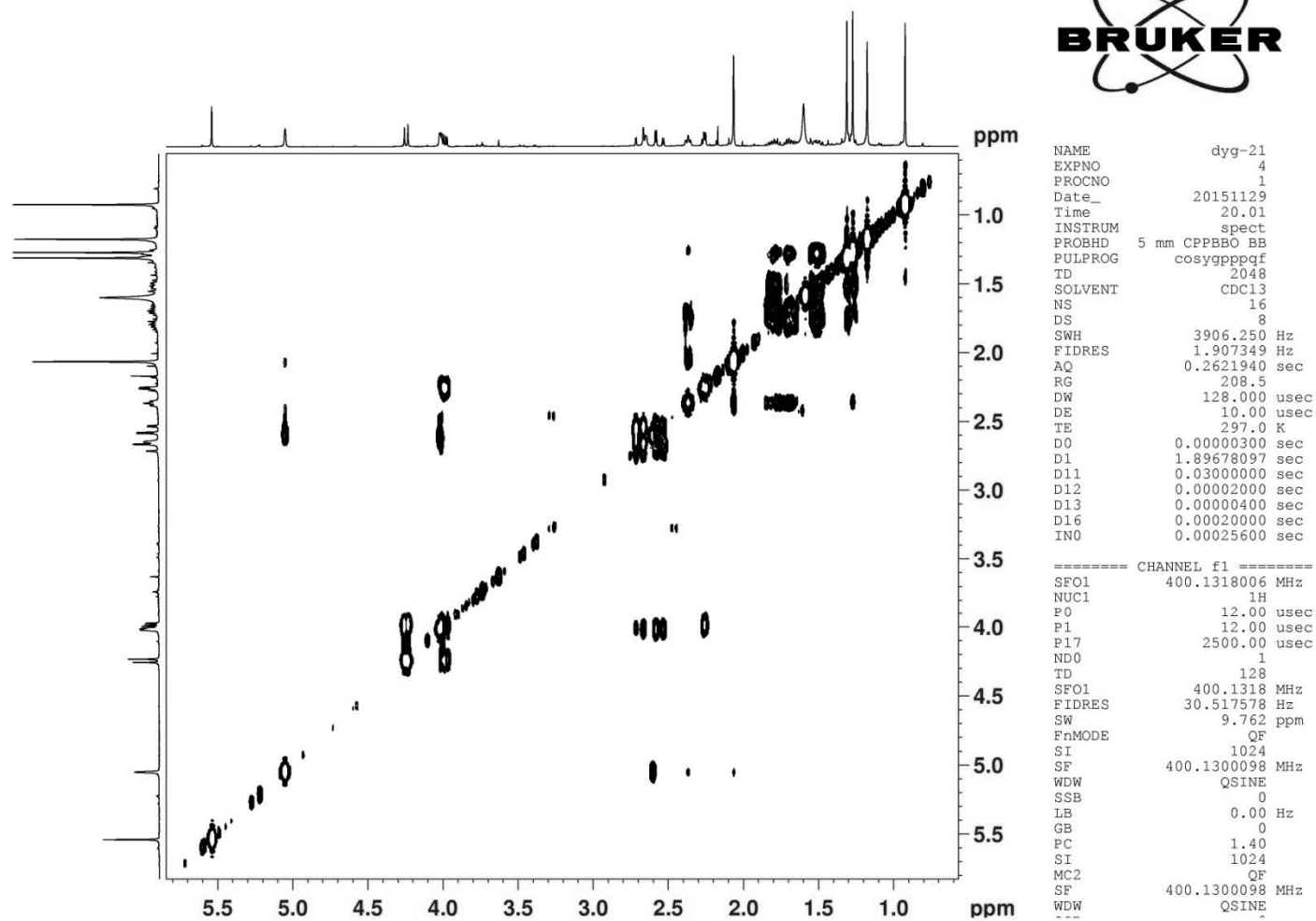
^1H - ^1H COSY (400 MHz) spectrum of compound **4** in CDCl_3



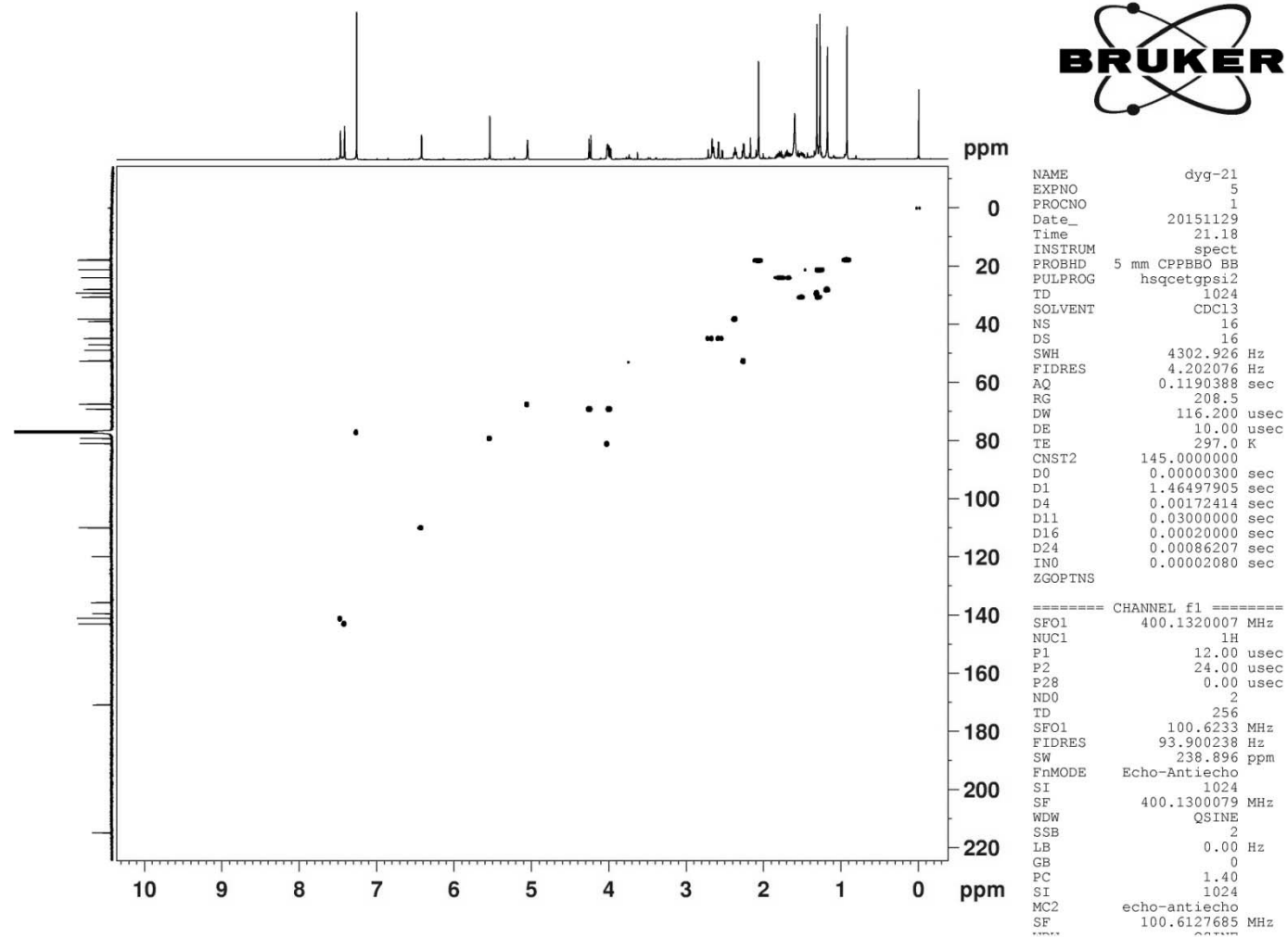
```
NAME          dyg-21
EXPNO         4
PROCNO        1
Date_         20151129
Time          20.01
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       cosygpppqf
TD            2048
SOLVENT       CDCl3
NS            16
DS            8
SWH           3906.250 Hz
FIDRES        1.907349 Hz
AQ            0.2621940 sec
RG            208.5
DW            128.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00000300 sec
D1            1.89678097 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00025600 sec
```

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

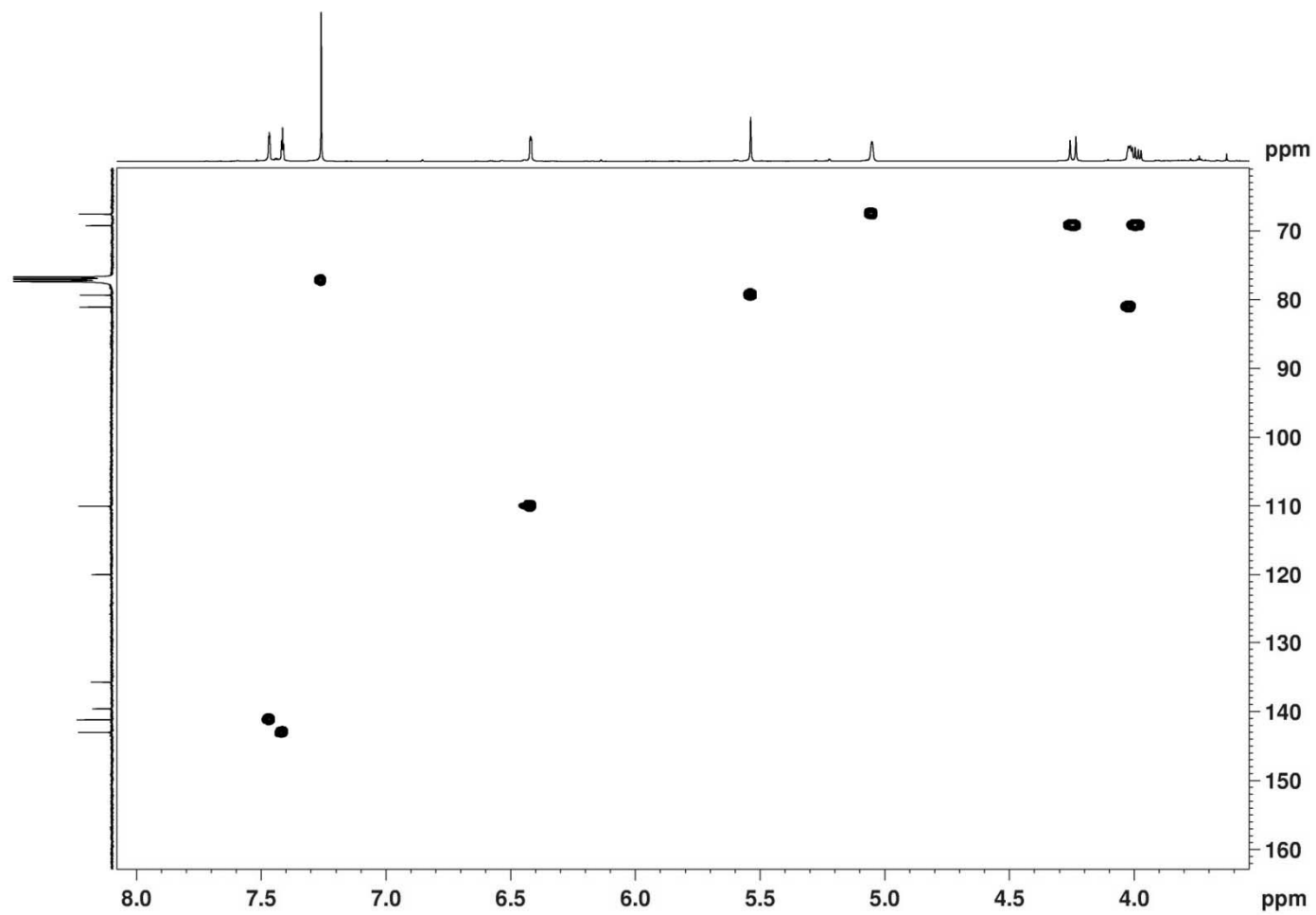
^1H - ^1H COSY (400 MHz) spectrum of compound **4** in CDCl_3



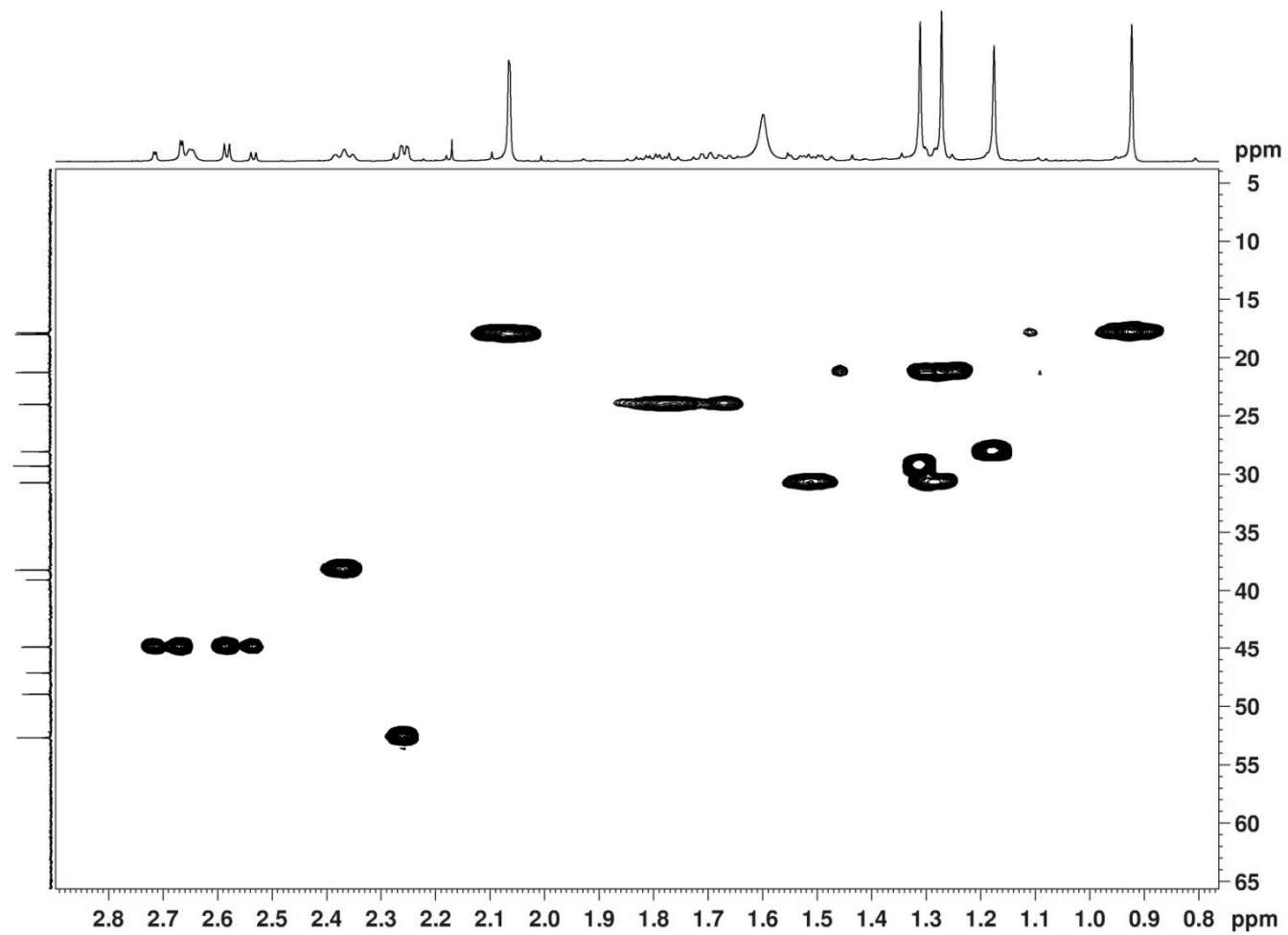
HSQC (400 MHz) spectrum of compound **4** in CDCl₃



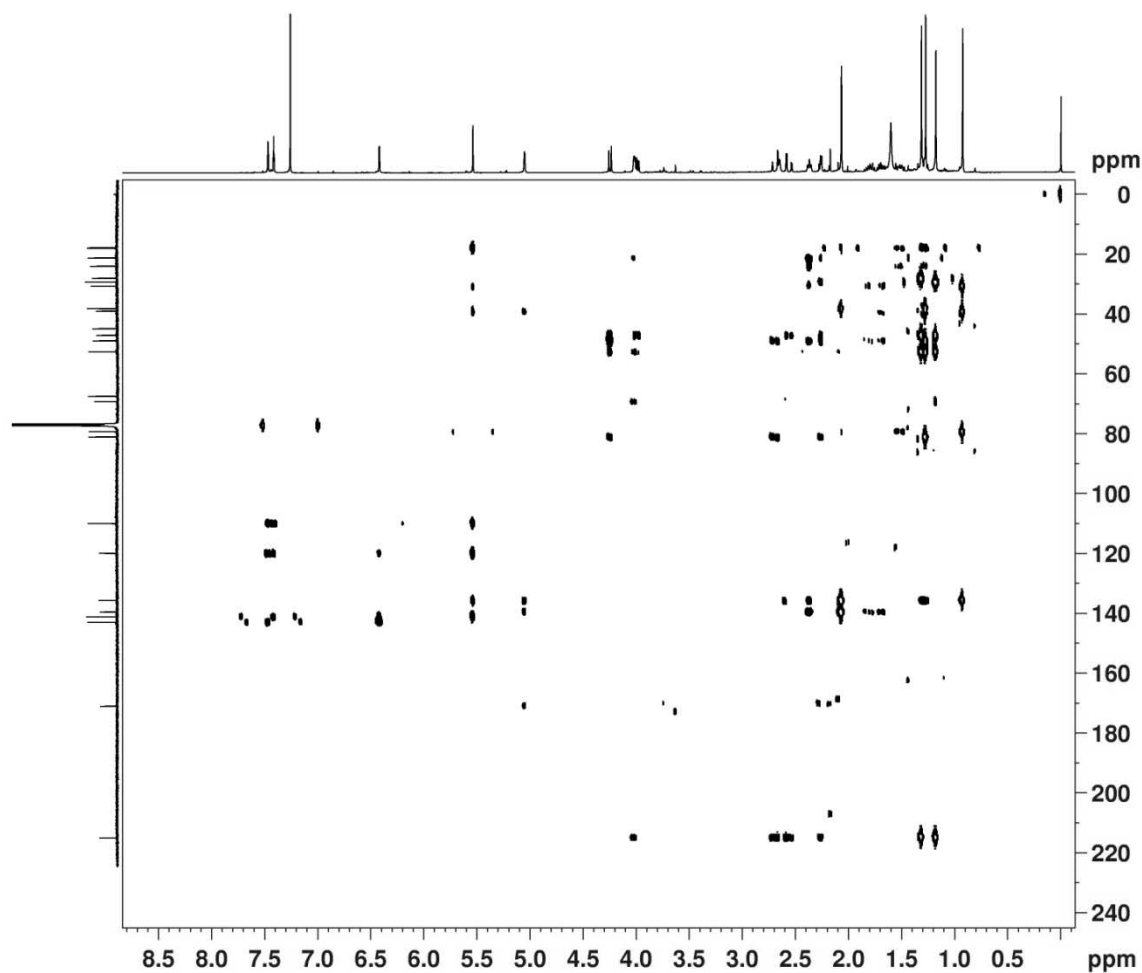
HSQC (400 MHz) spectrum of compound **4** in CDCl₃



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



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



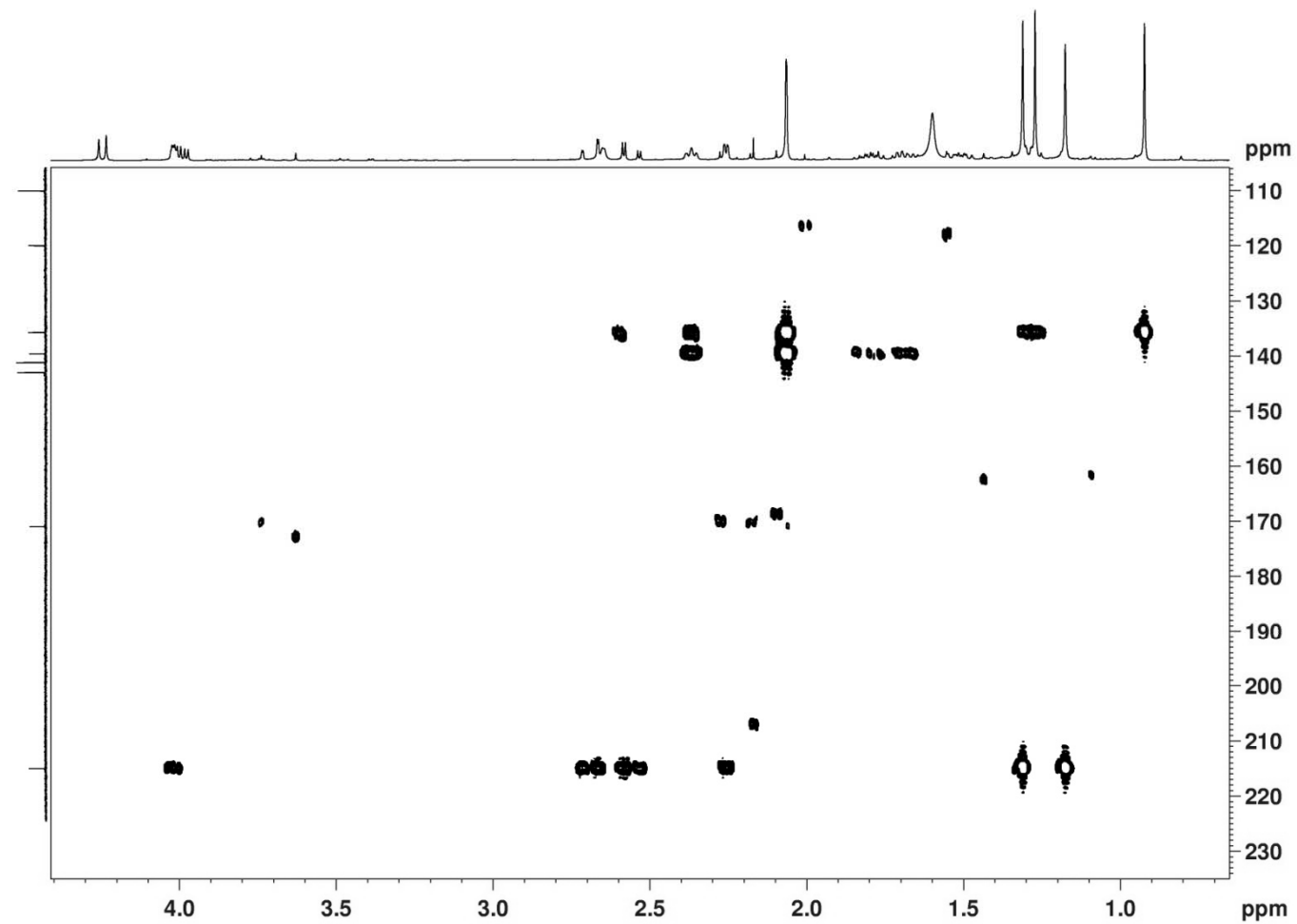
```

NAME      dyg-21
EXPNO     6
PROCNO    1
Date_     20151129
Time      23.12
INSTRUM   spect
PROBHD    5 mm CPPBBO BB
PULPROG   hmbegp1pndqf
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
    
```

```

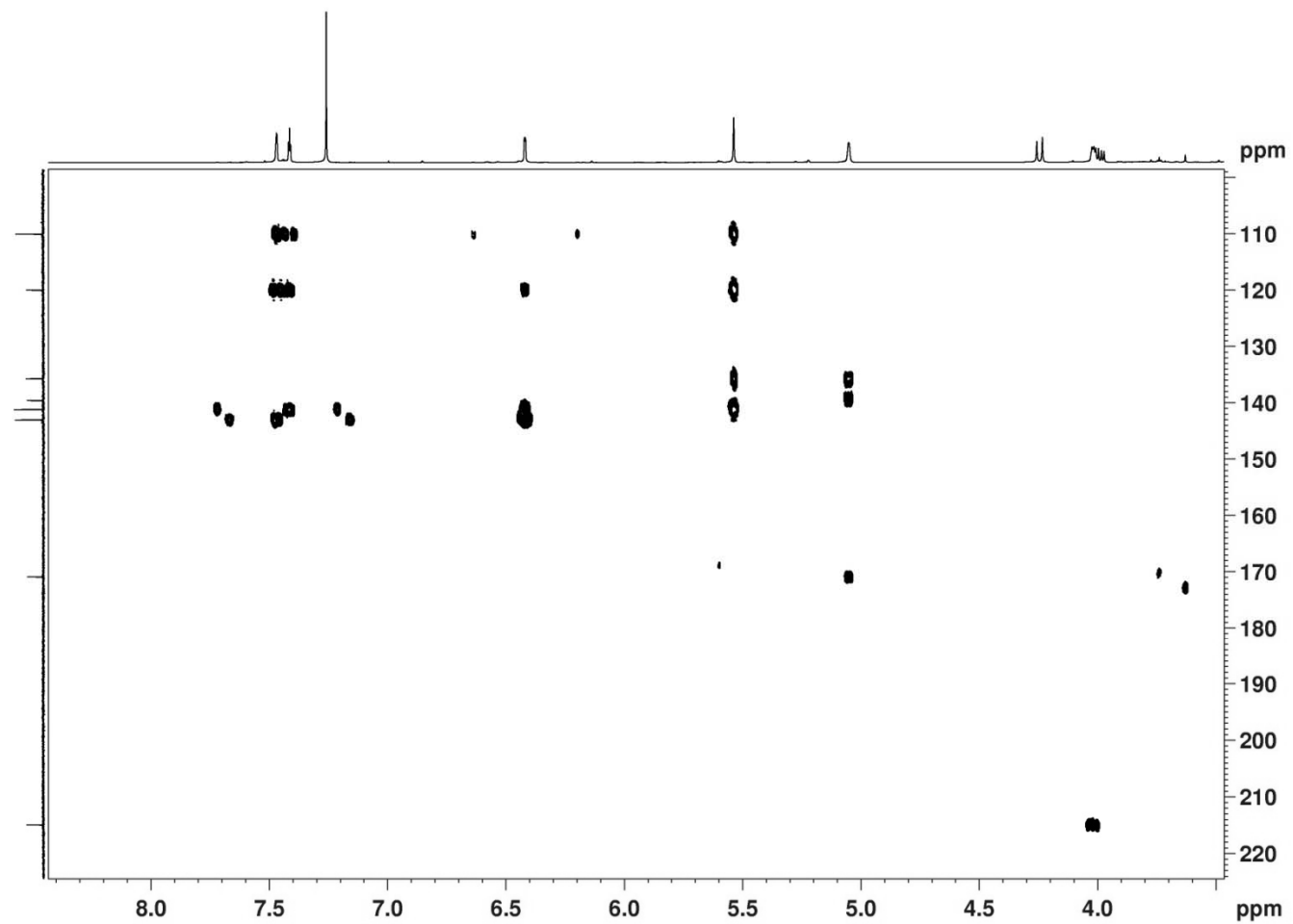
===== CHANNEL f1 =====
SFO1    400.1323208 MHz
NUC1     1H
P1       12.00 usec
P2       24.00 usec
ND0       2
TD        128
SFO1    100.6233 MHz
FIDRES   187.800476 Hz
SW       238.896 ppm
FnMODE   QF
SI        2048
SF       400.1300097 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB         0
PC         1.40
SI        1024
MC2       QF
SF       100.6127685 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB         0
    
```


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



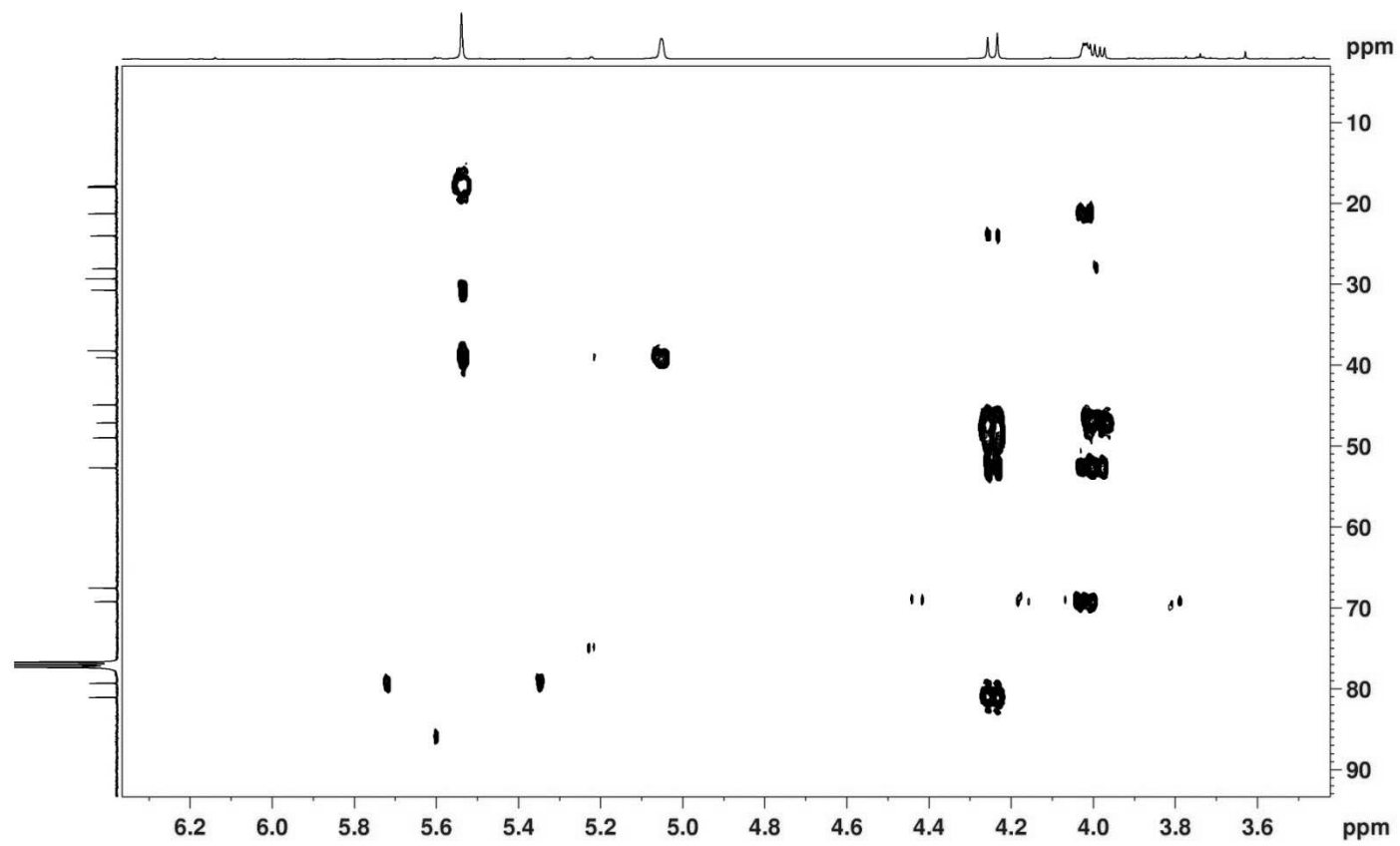
S101

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



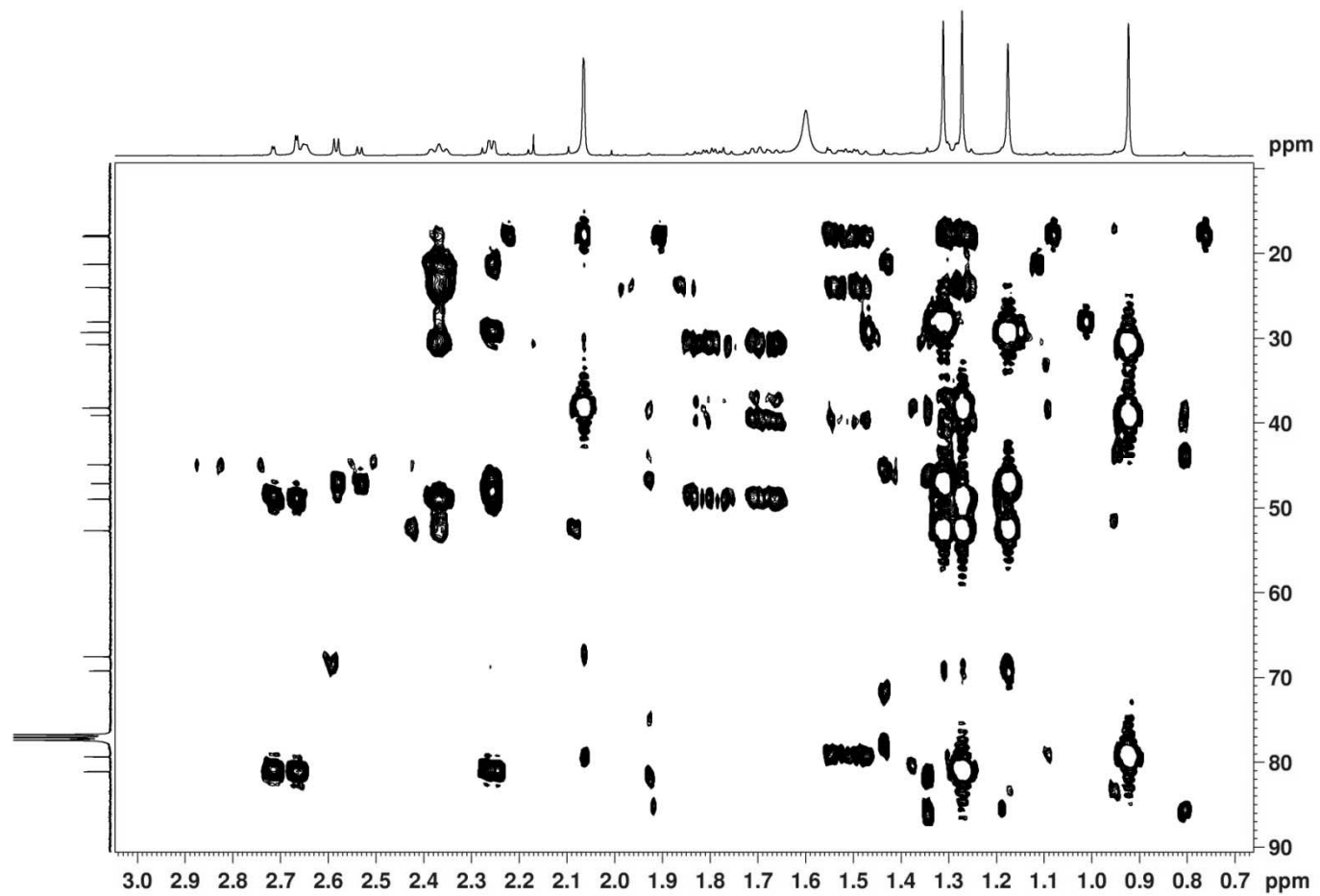
S102

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

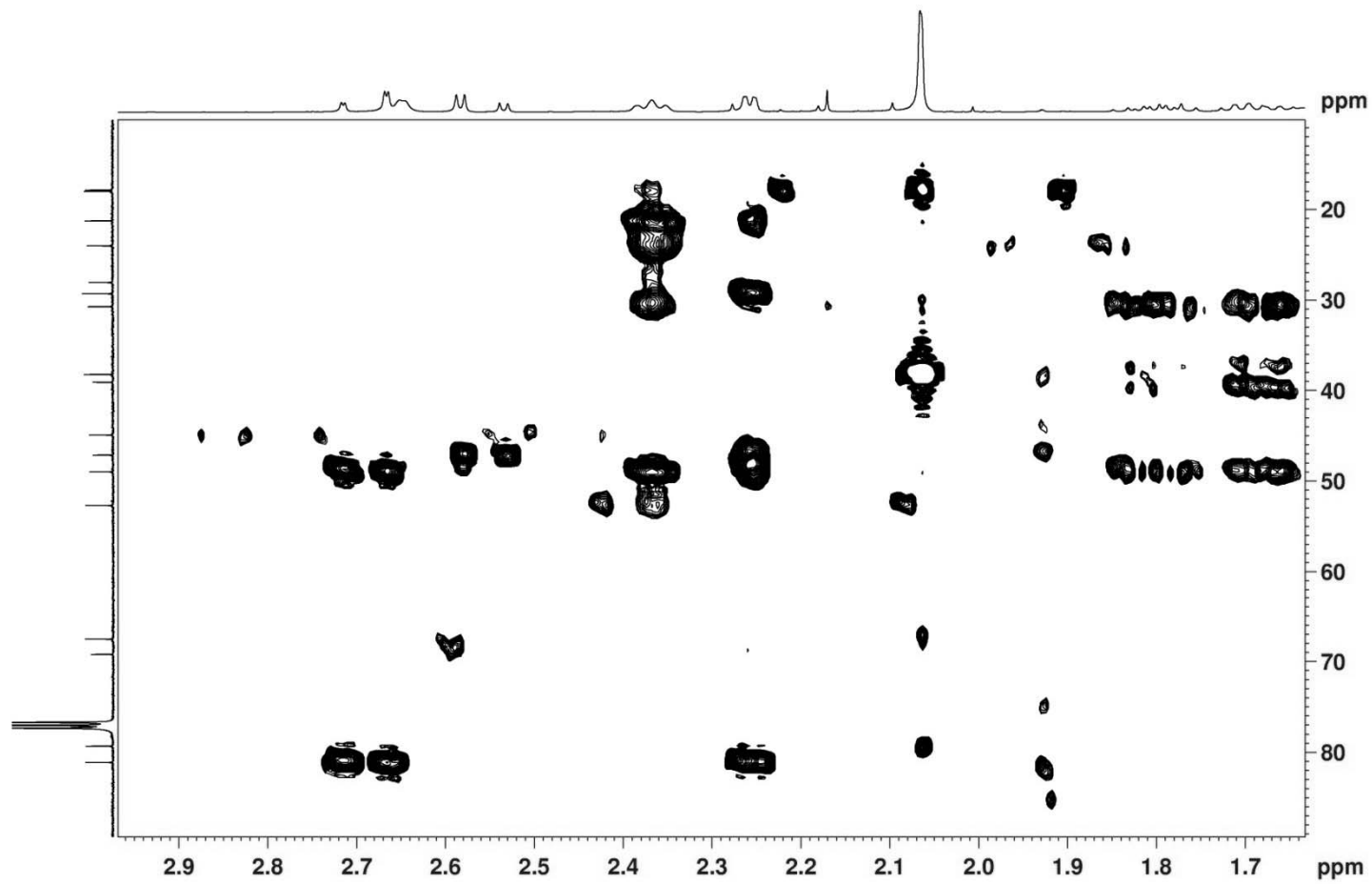


S103

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

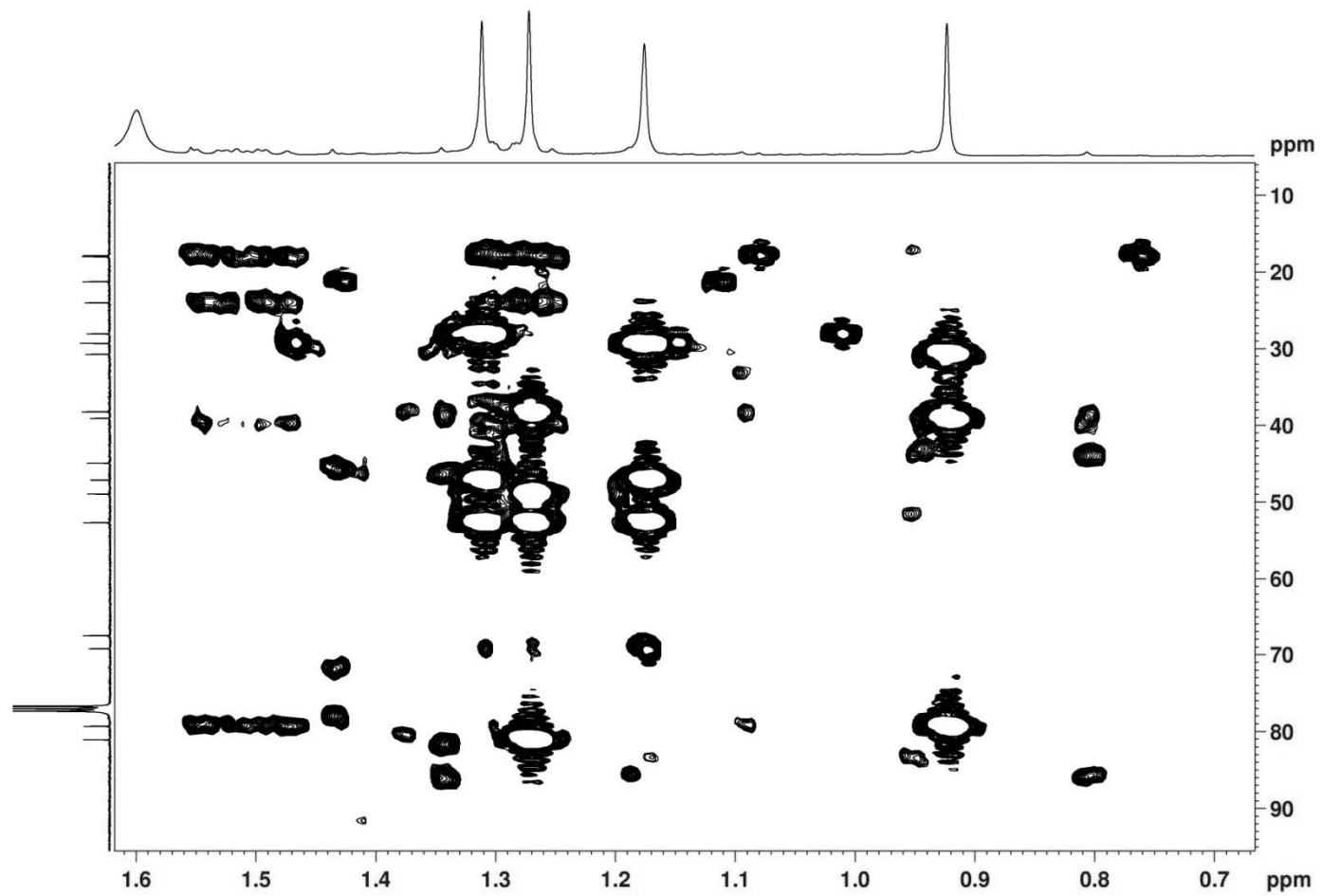


HMBC (400 MHz) spectrum of compound **4** in CDCl₃

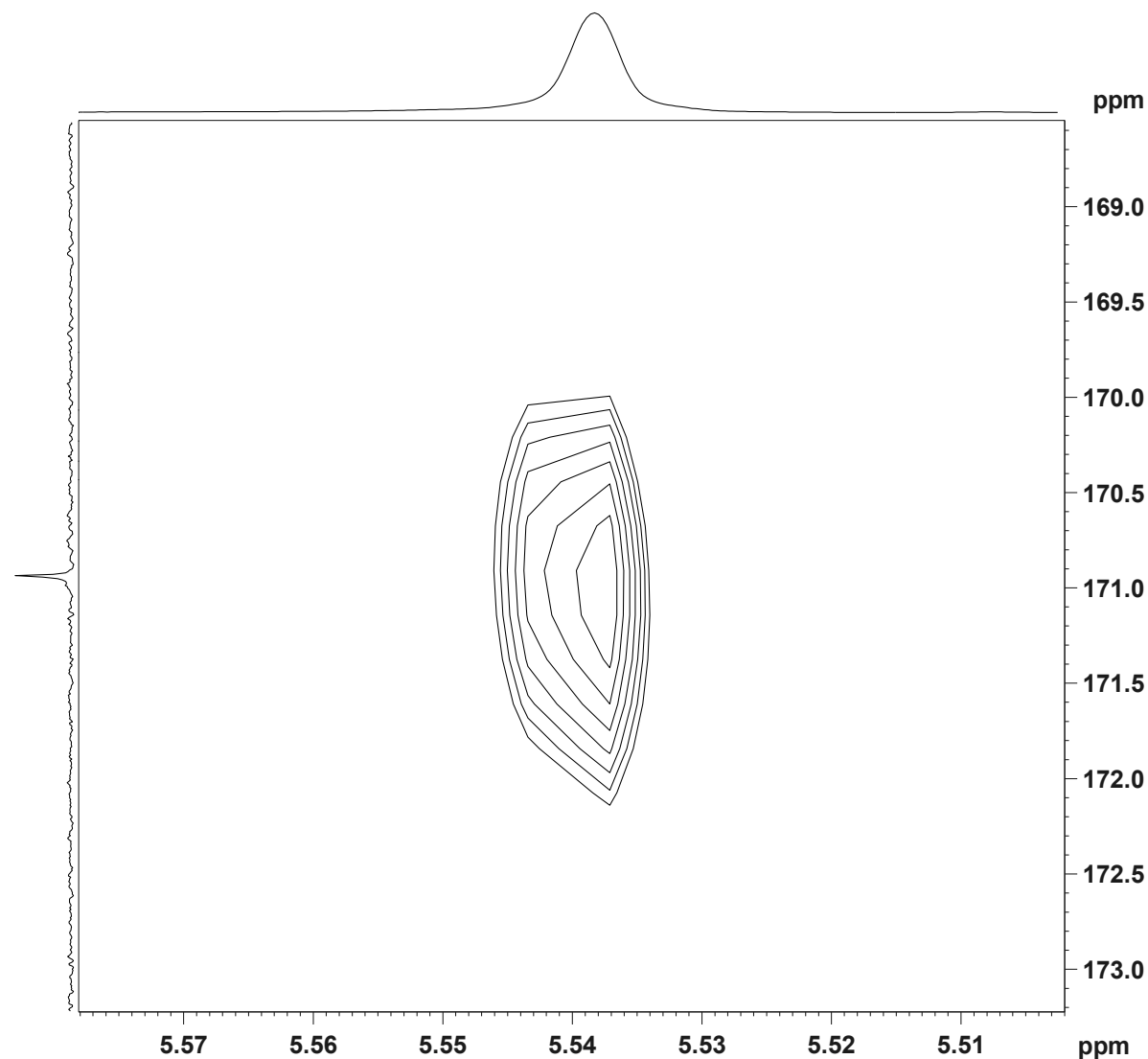
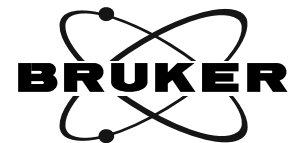


S105

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



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

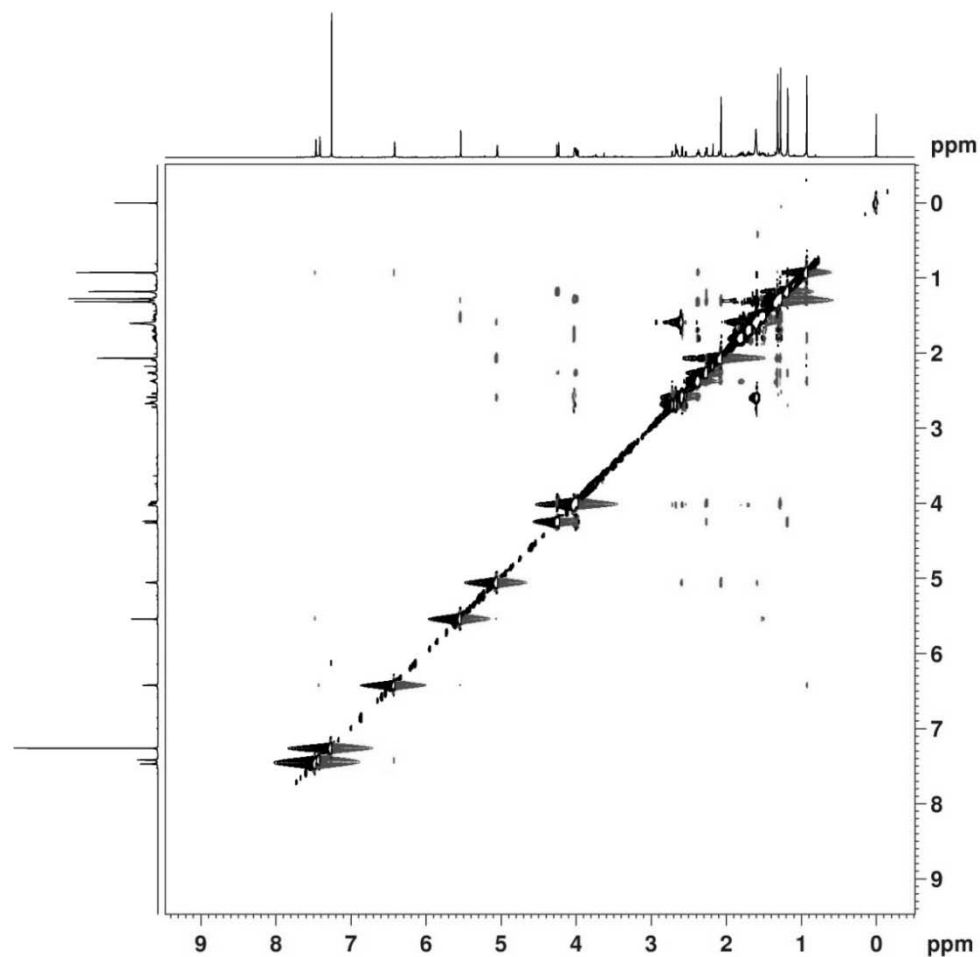


```

NAME          dyg-21
EXPNO         6
PROCNO        1
Date_         20151129
Time          23.12
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
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 =====
SF01          400.1323208 MHz
NUC1           1H
P1             12.00 usec
P2             24.00 usec
ND0            2
TD            128
SF01          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            100.6127685 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

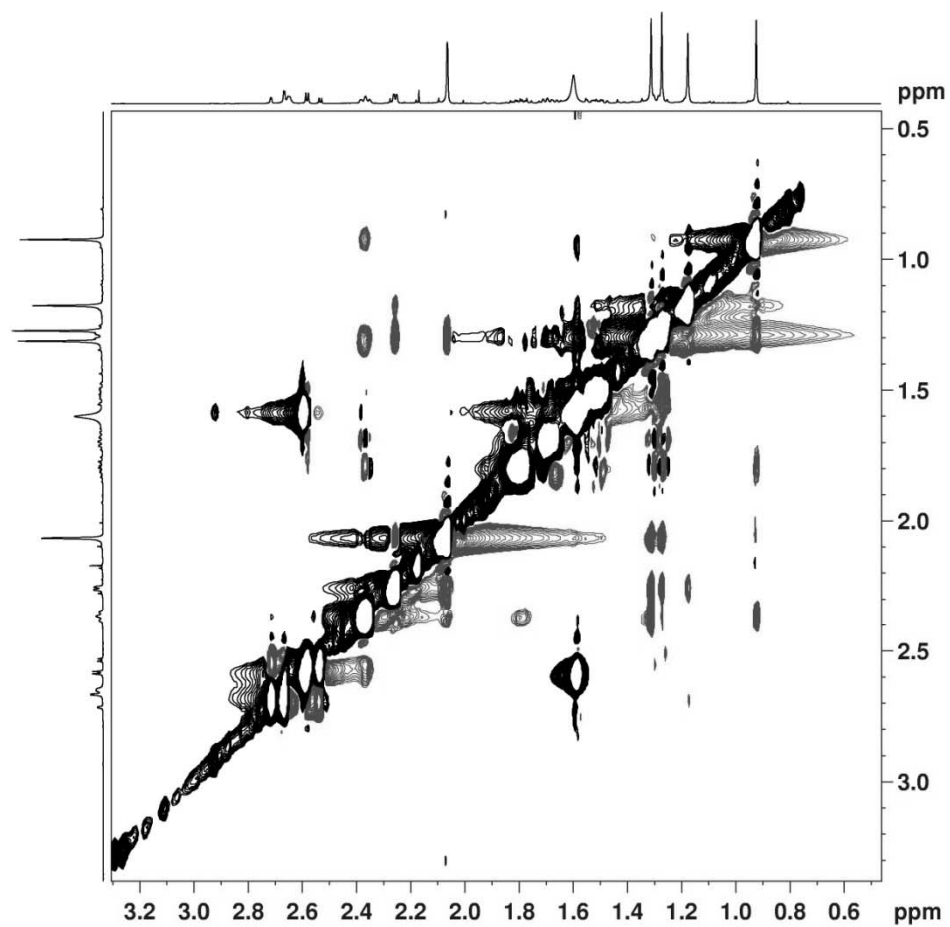
NOESY (400 MHz) spectrum of compound **4** in CDCl₃



```
NAME          dyg-21
EXPNO         7
PROCNO        1
Date_         20151130
Time          8.06
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 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
```

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

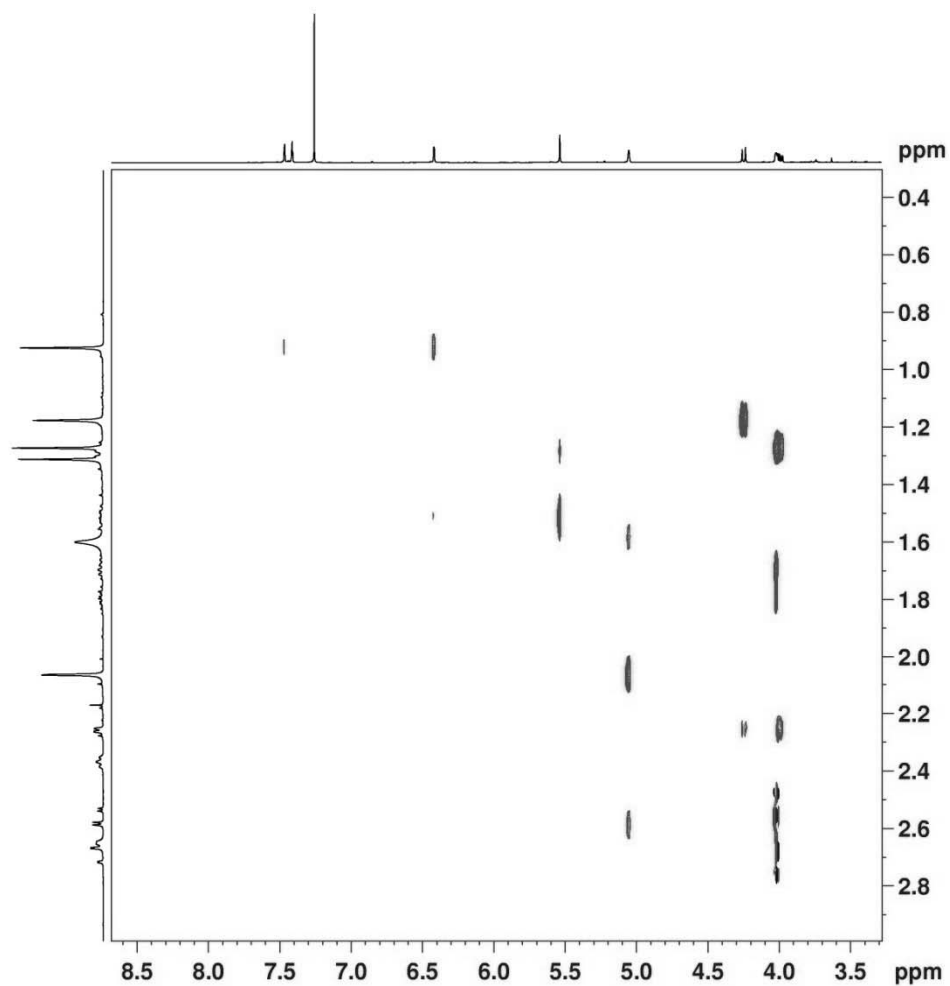

NOESY (400 MHz) spectrum of compound **4** in CDCl₃



```
NAME          dyg-21
EXPNO         7
PROCNO        1
Date_         20151130
Time          8.06
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       noesygpphpg
TD            2048
SOLVENT       CDCl3
NS            32
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
```

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

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



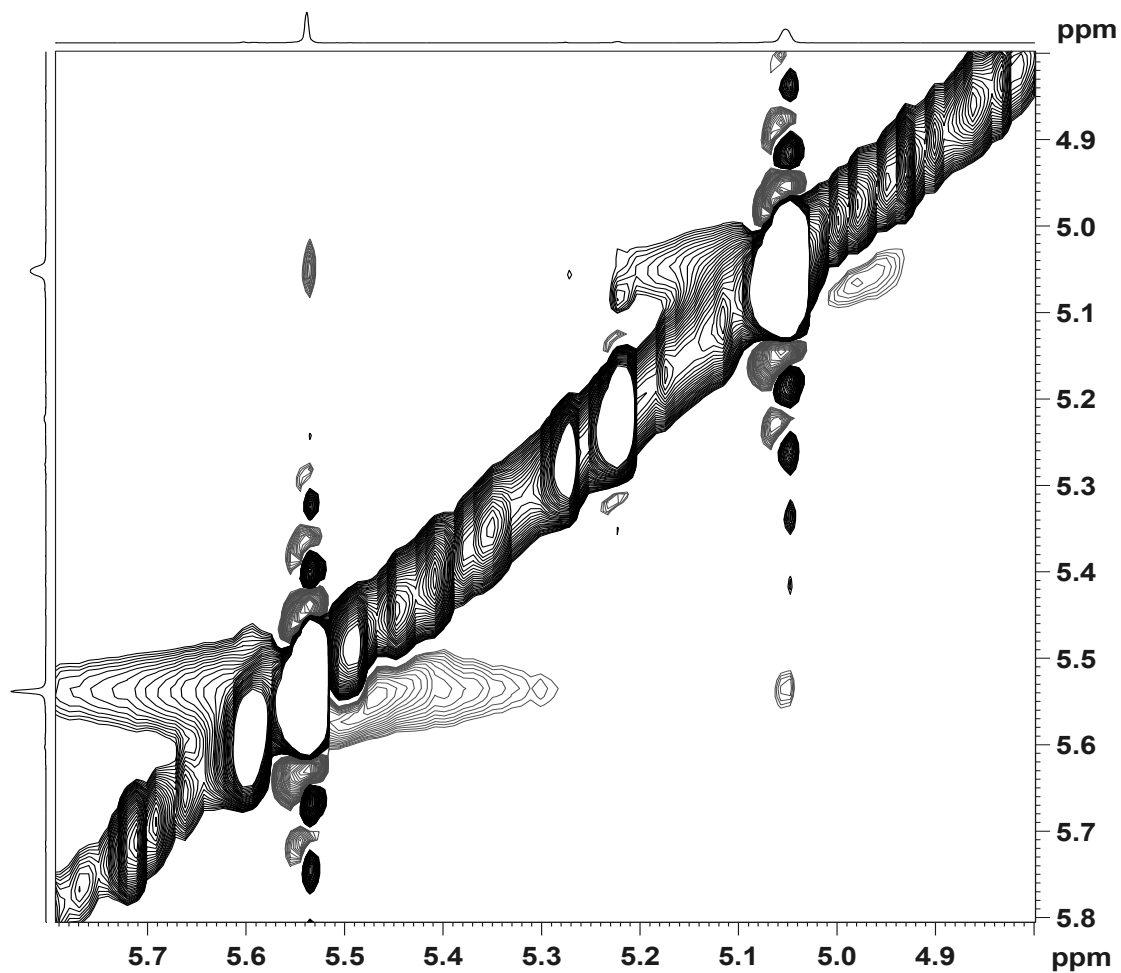
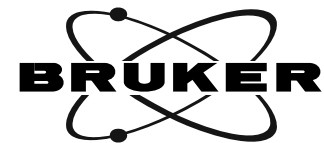
```

NAME          dyg-21
EXPNO         7
PROCNO        1
Date_         20151130
Time          8.06
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 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec
    
```

```

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

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



```

NAME          dyg-21
EXPNO         7
PROCNO        1
Date_         20151130
Time          8.06
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 sec
RG            208.5
DW            125.000 usec
DE            10.00 usec
TE            297.0 K
D0            0.00010972 sec
D1            1.99385595 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00025000 sec

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