

角果木中两个新的 dolabrane 型二萜类化合物

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补充数据

Two New Dolabrane-Type Diterpenes from *Ceriops tagal*

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Supporting data

Supporting Information:

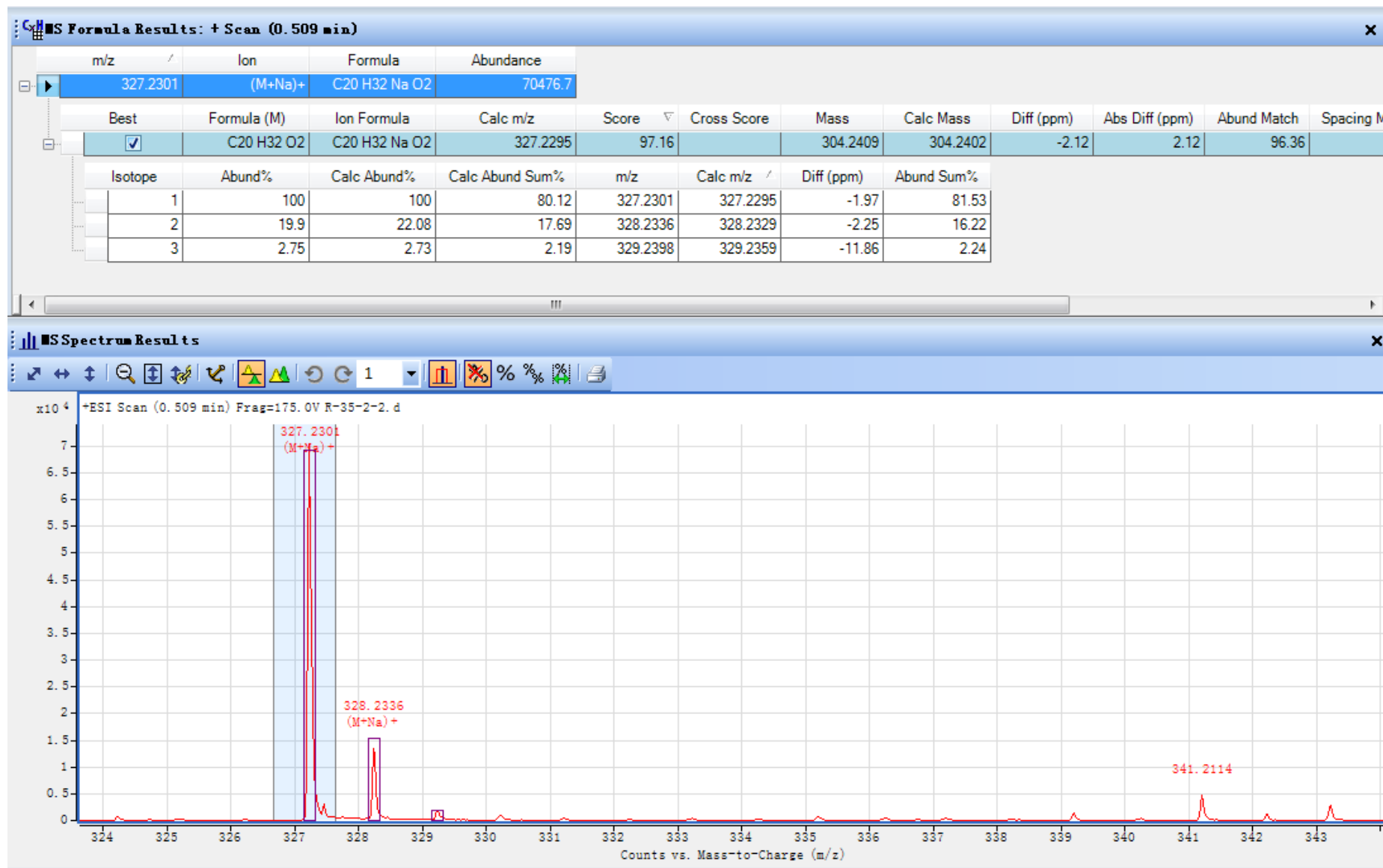
化合物 1 和 2 的 HR-ESIMS, 1D、2D NMR 谱图

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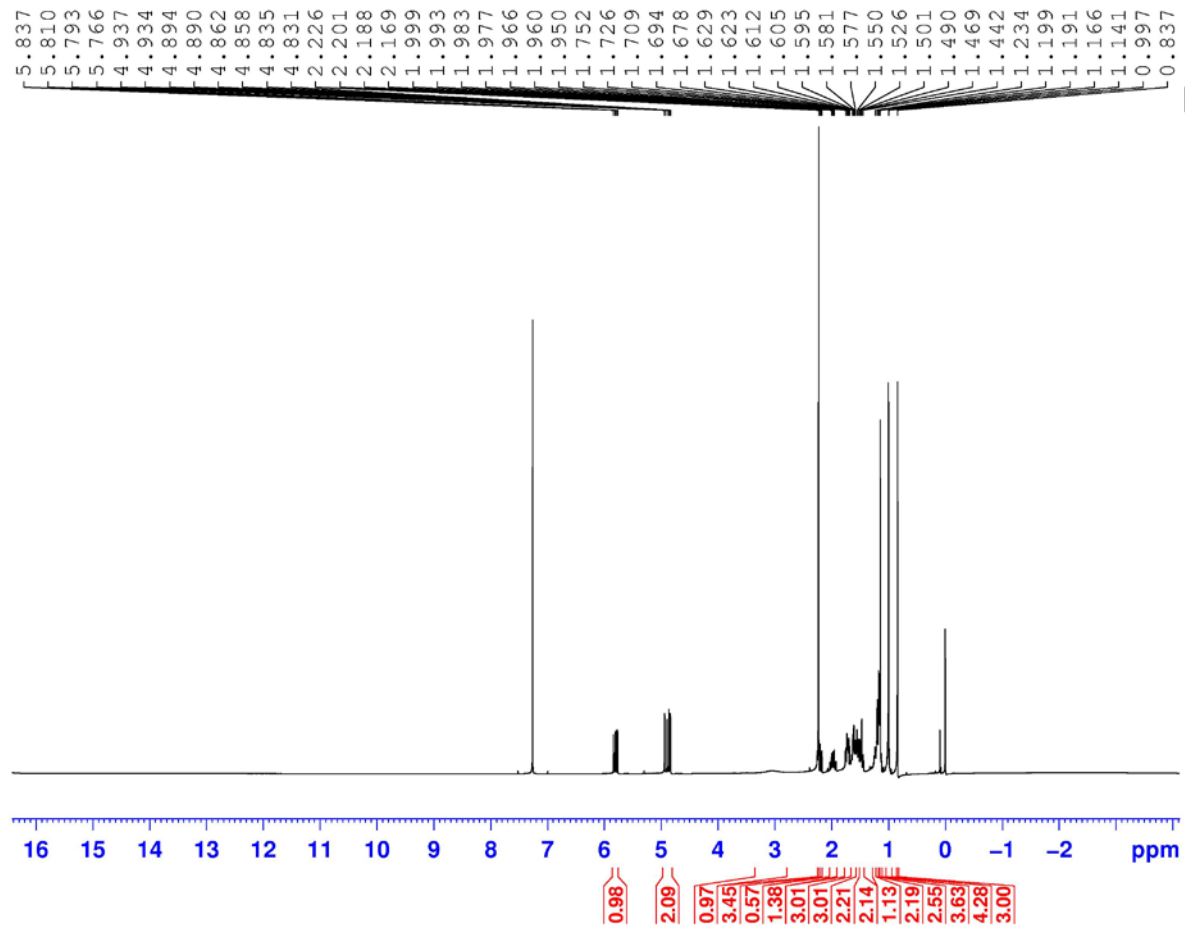
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HR-ESIMS for compound 1



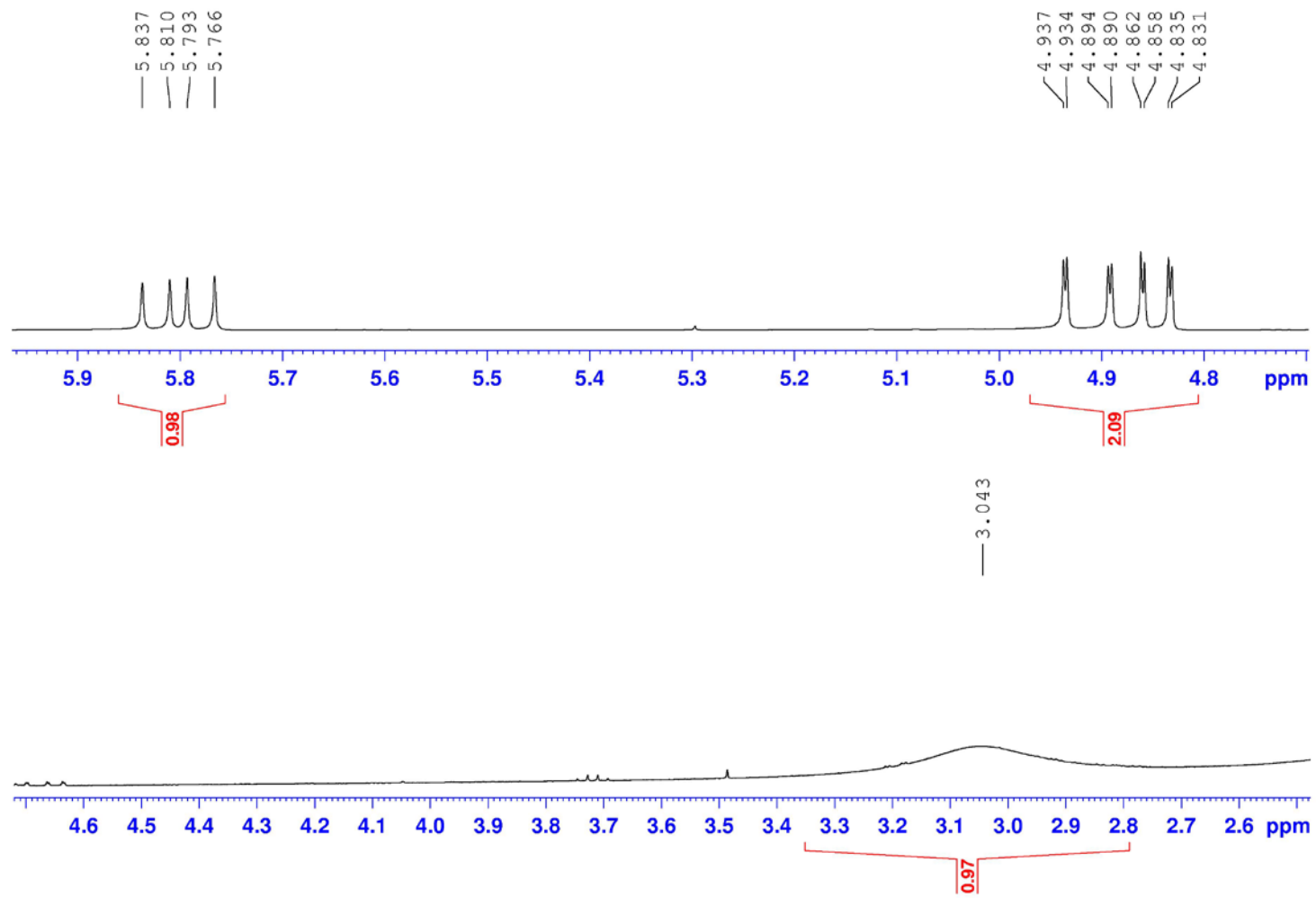
¹H NMR (400 MHz) spectrum of compound 1 in CDCl₃



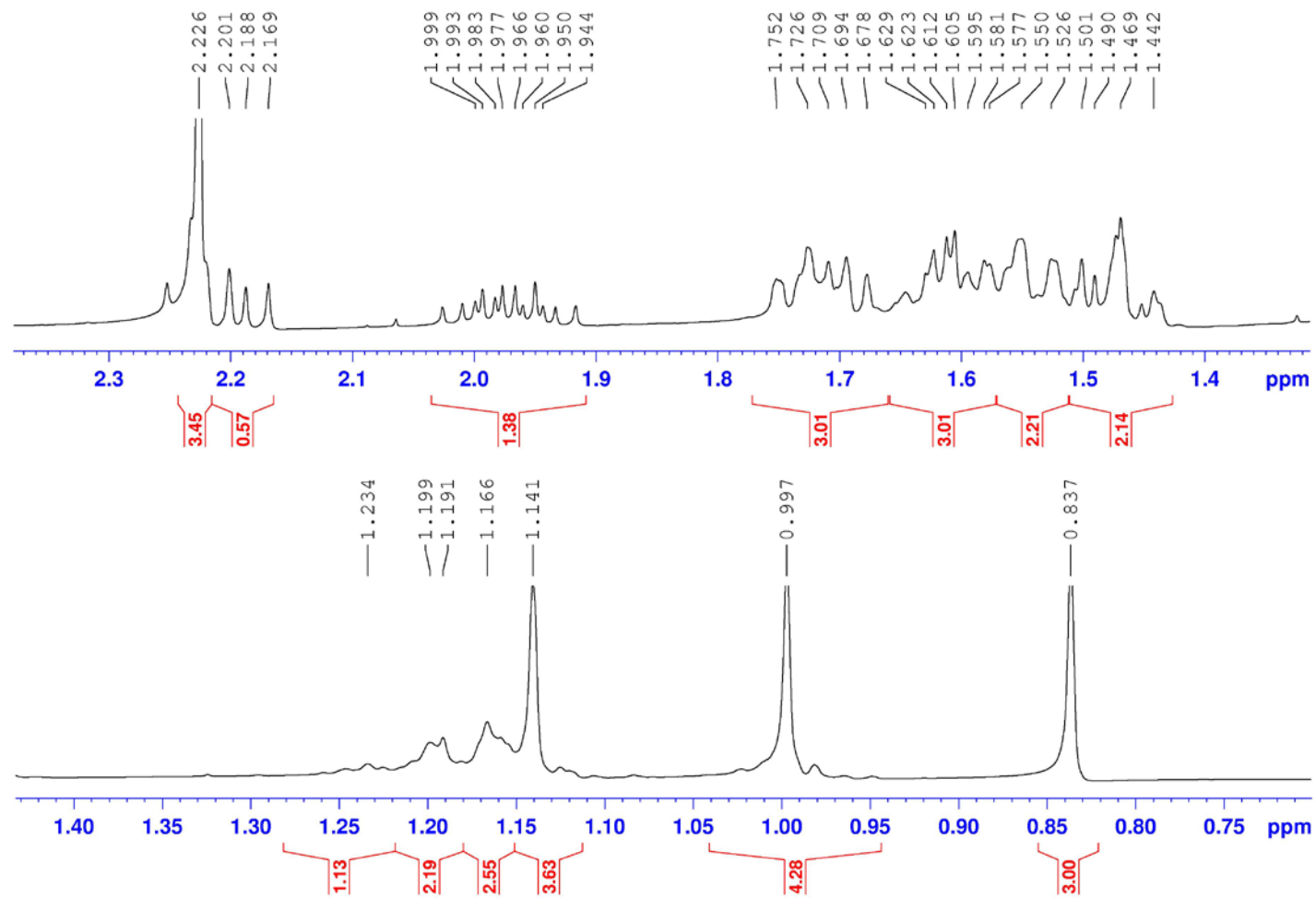
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PROBHD        5 mm CFPBBO BB
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            32
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            117.37
FW           60.800 usec
DE            10.00 usec
TE            297.0 K
D1            1.00000000 sec
TD0           1
===== CHANNEL f1 =====
SF01          400.1324710 MHz
NUC1           1H
P1            11.50 usec
SI            65536
SF            400.1300096 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
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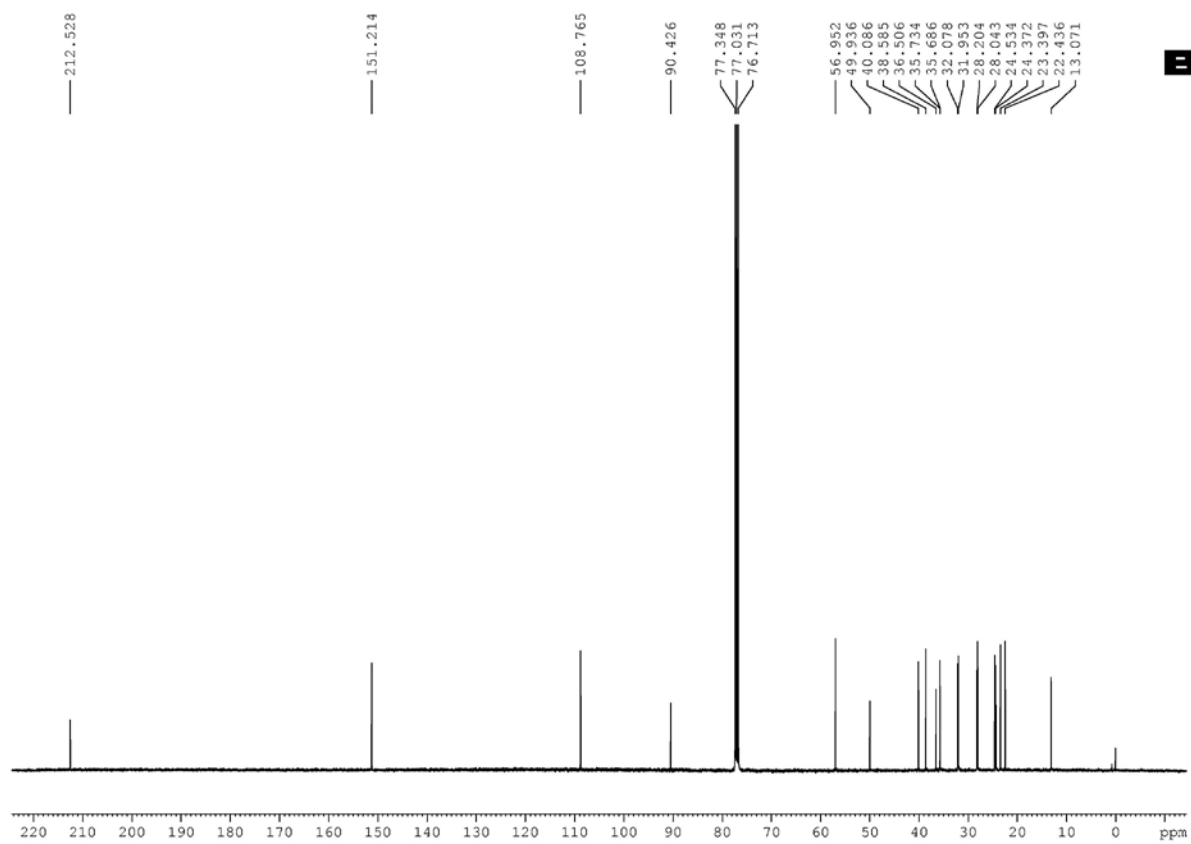
^1H NMR (400 MHz) spectrum of compound 1 in CDCl_3



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



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

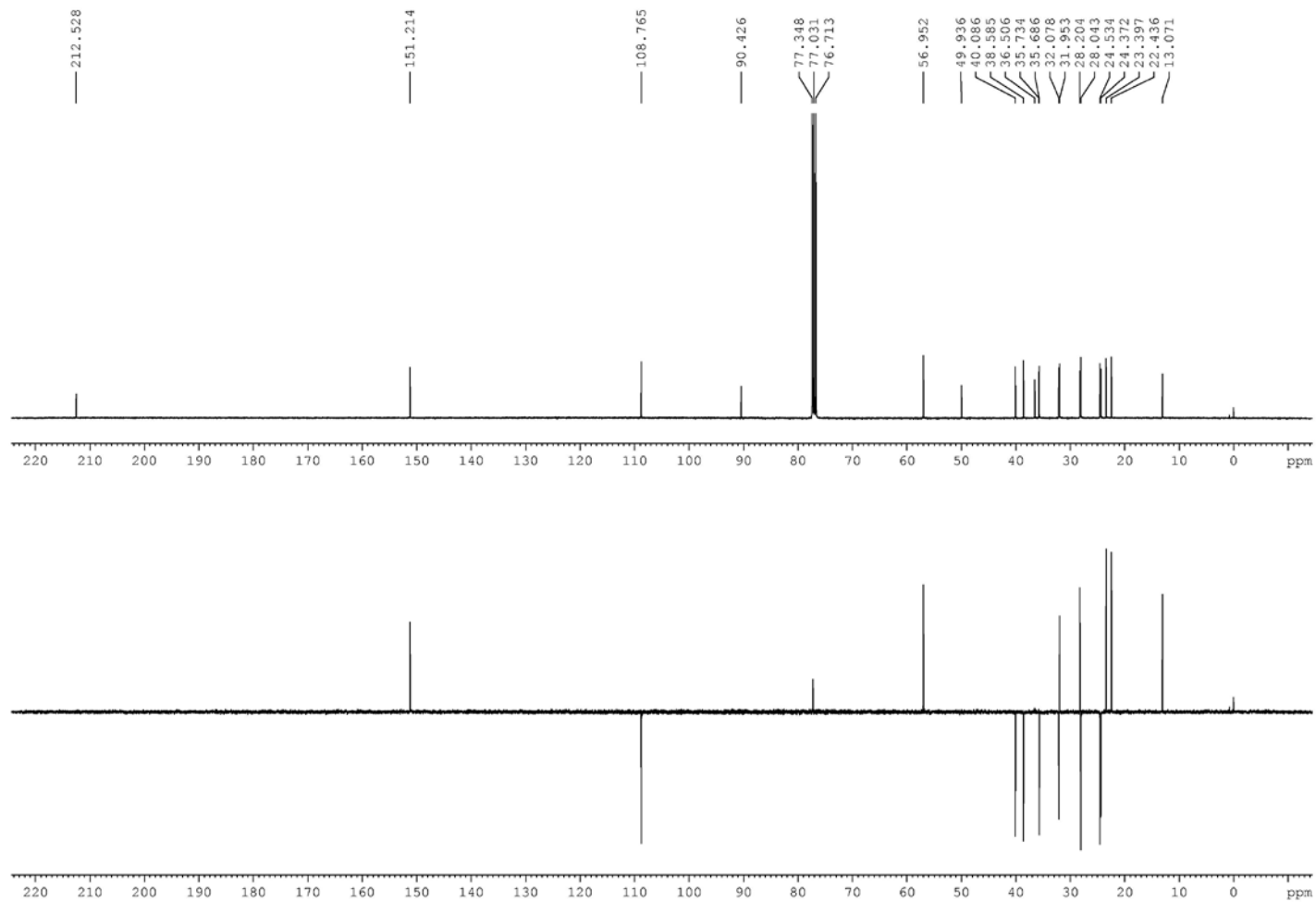


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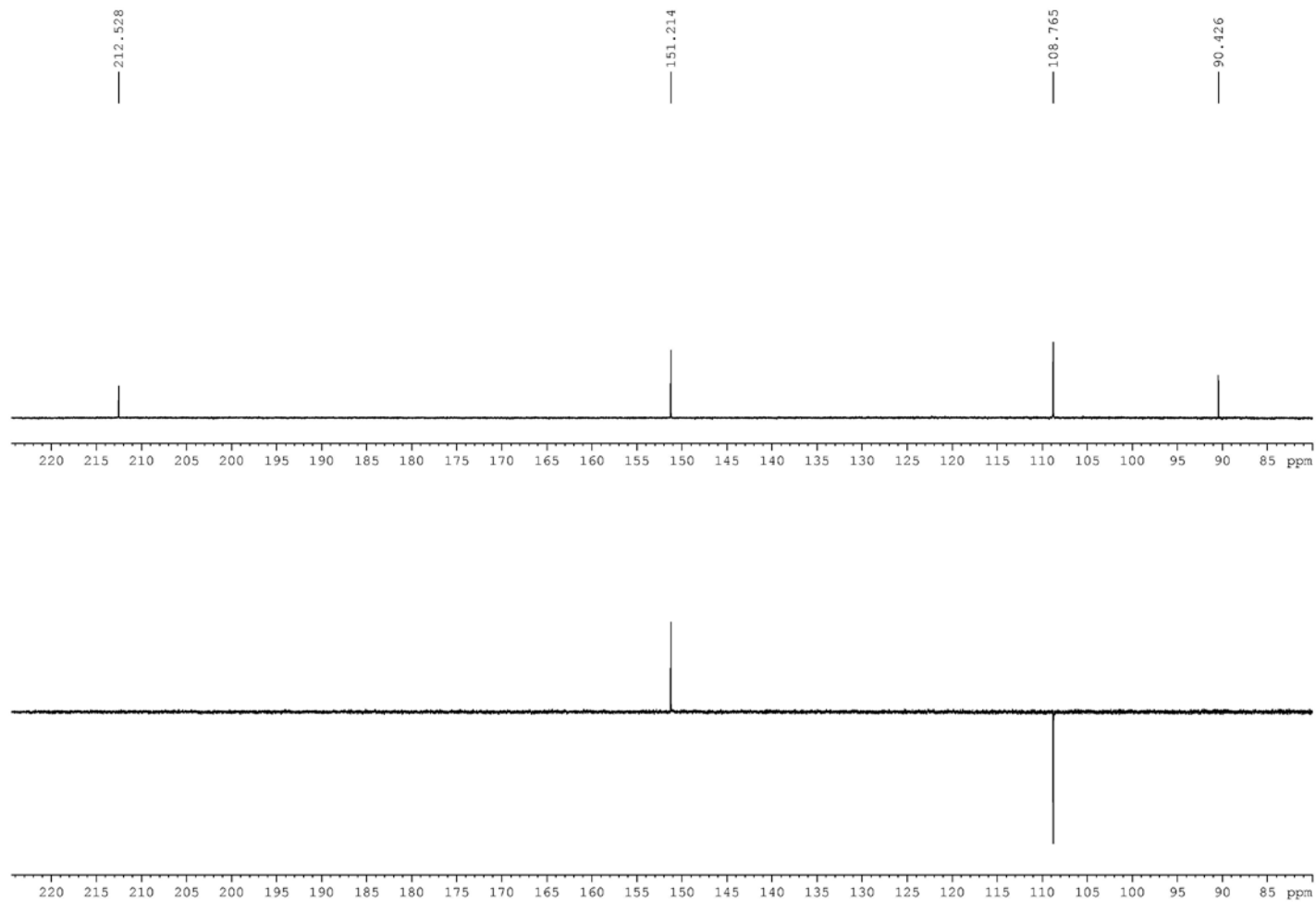
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EXPNO         2
PROCNO        1
Date_         20161015
Time          9.47
INSTRUM       spect
PROBHD        5 mm CPMAS
PULPROG       zgpg30
ID            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            147.94
DW            20.800 usec
DE            18.00 usec
TE            297.0 K
D1            2.0000000 sec
d11           0.0300000 sec
TD0           1

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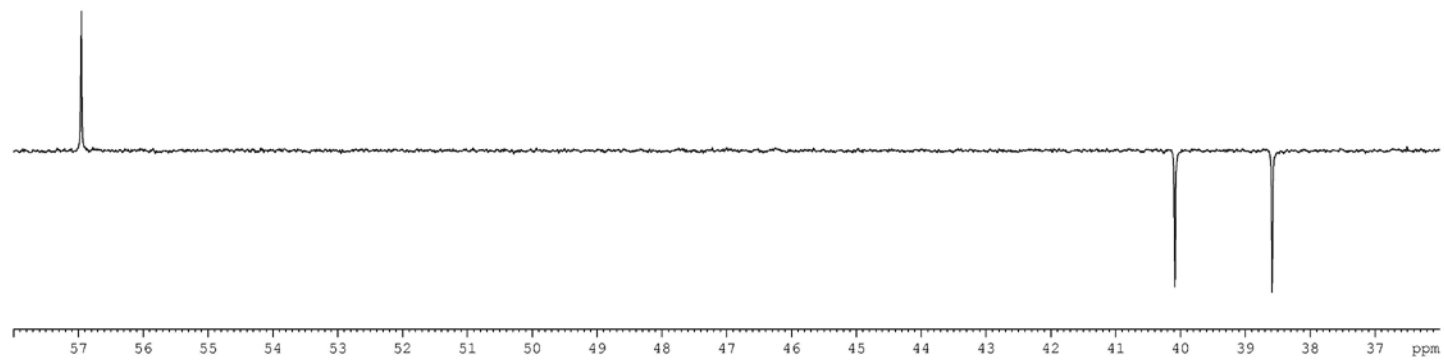
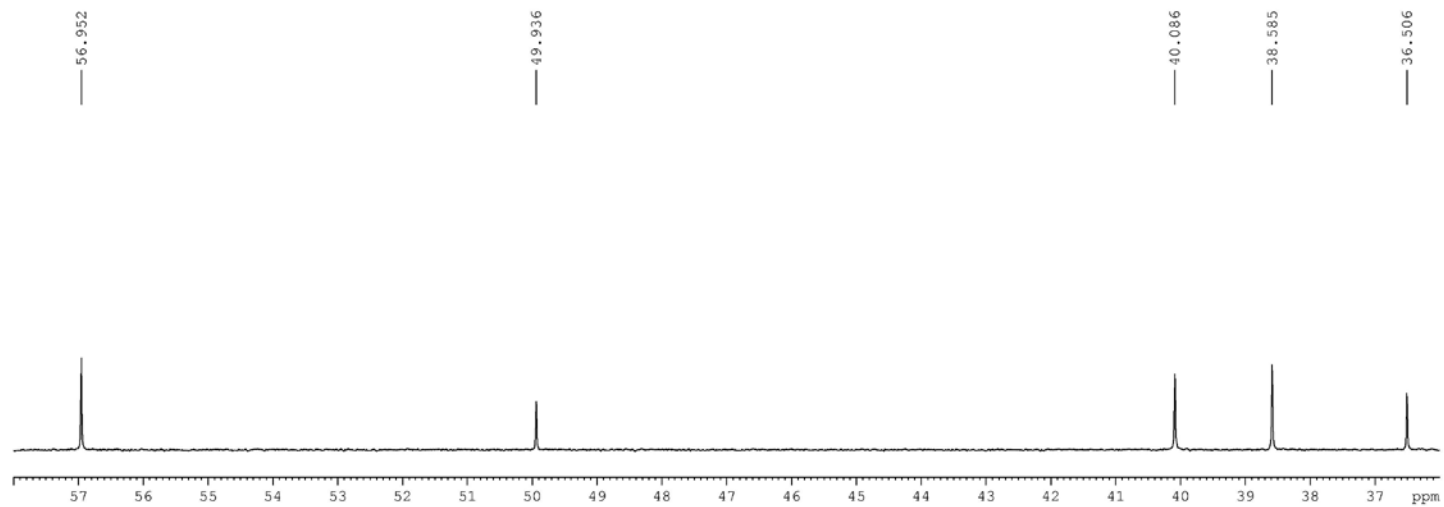

DEPT135 (100 MHz) spectrum of compound 1 in CDCl₃



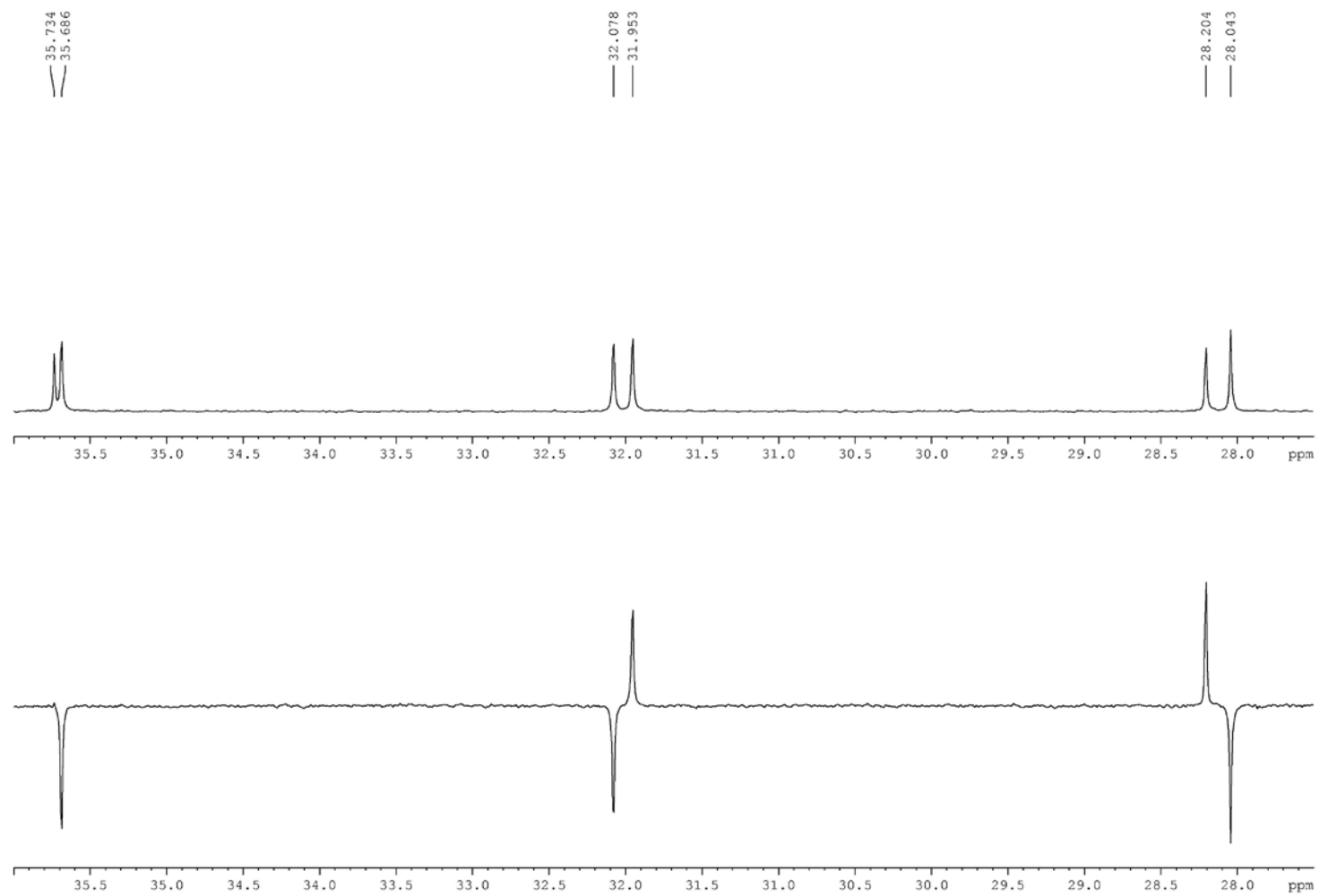
DEPT135 (100 MHz) spectrum of compound 1 in CDCl₃



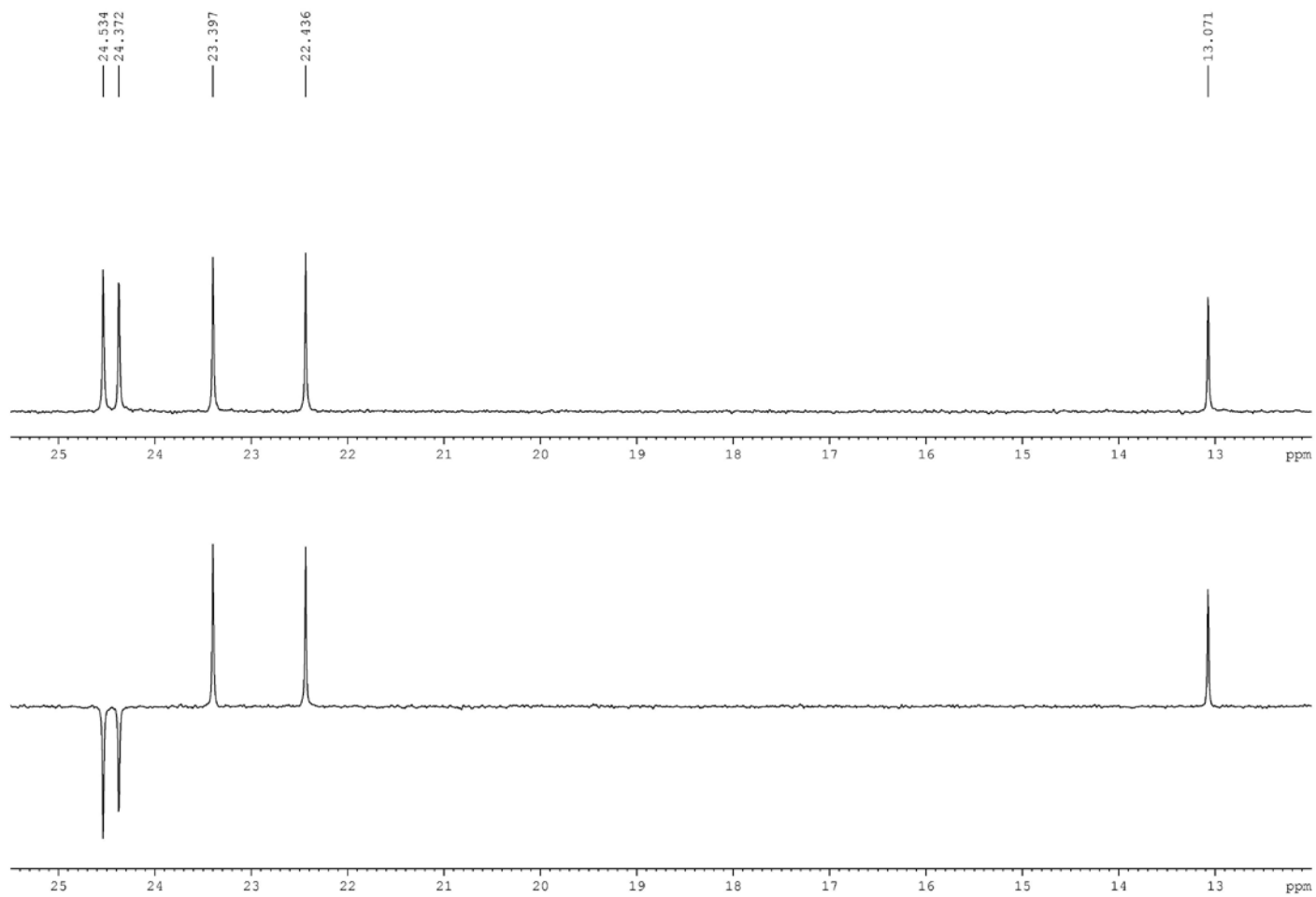
DEPT135 (100 MHz) spectrum of compound 1 in CDCl₃



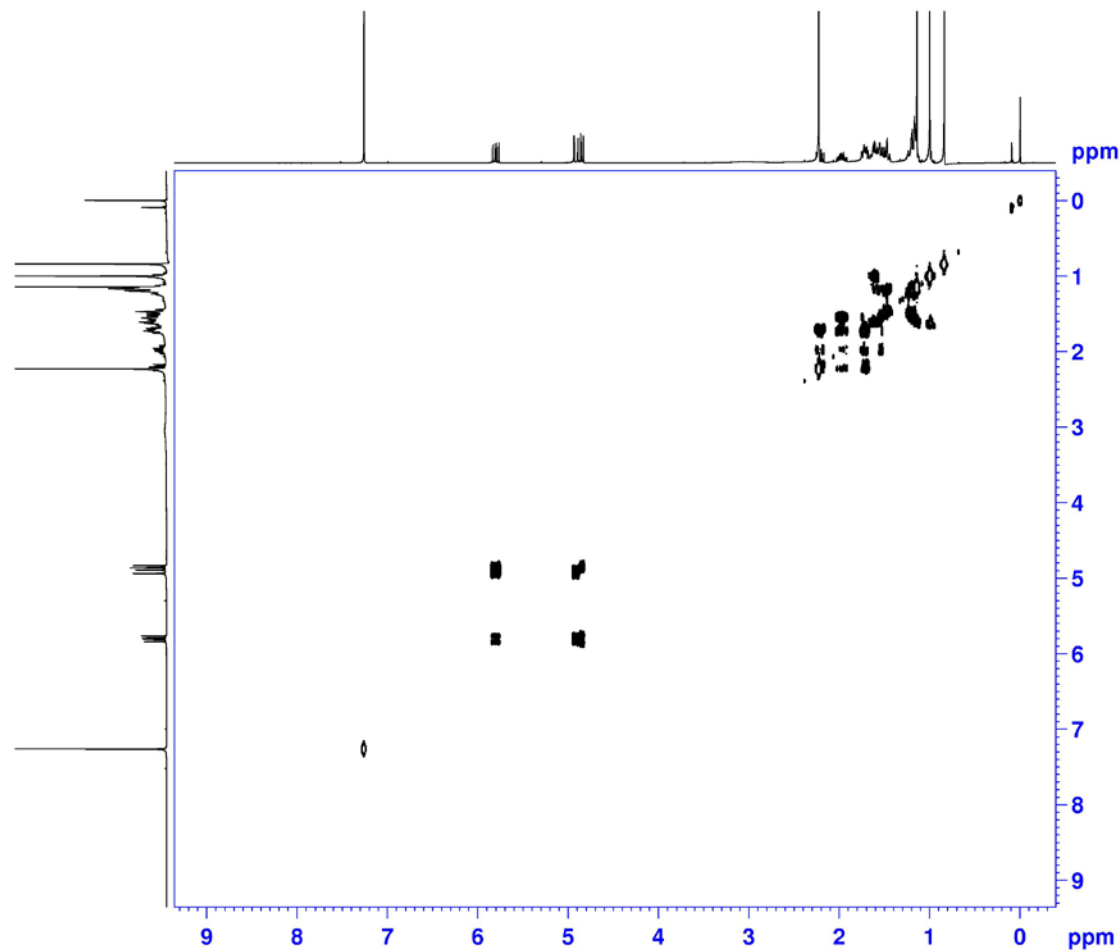
DEPT135 (100 MHz) spectrum of compound 1 in CDCl₃



DEPT135 (100 MHz) spectrum of compound 1 in CDCl₃



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



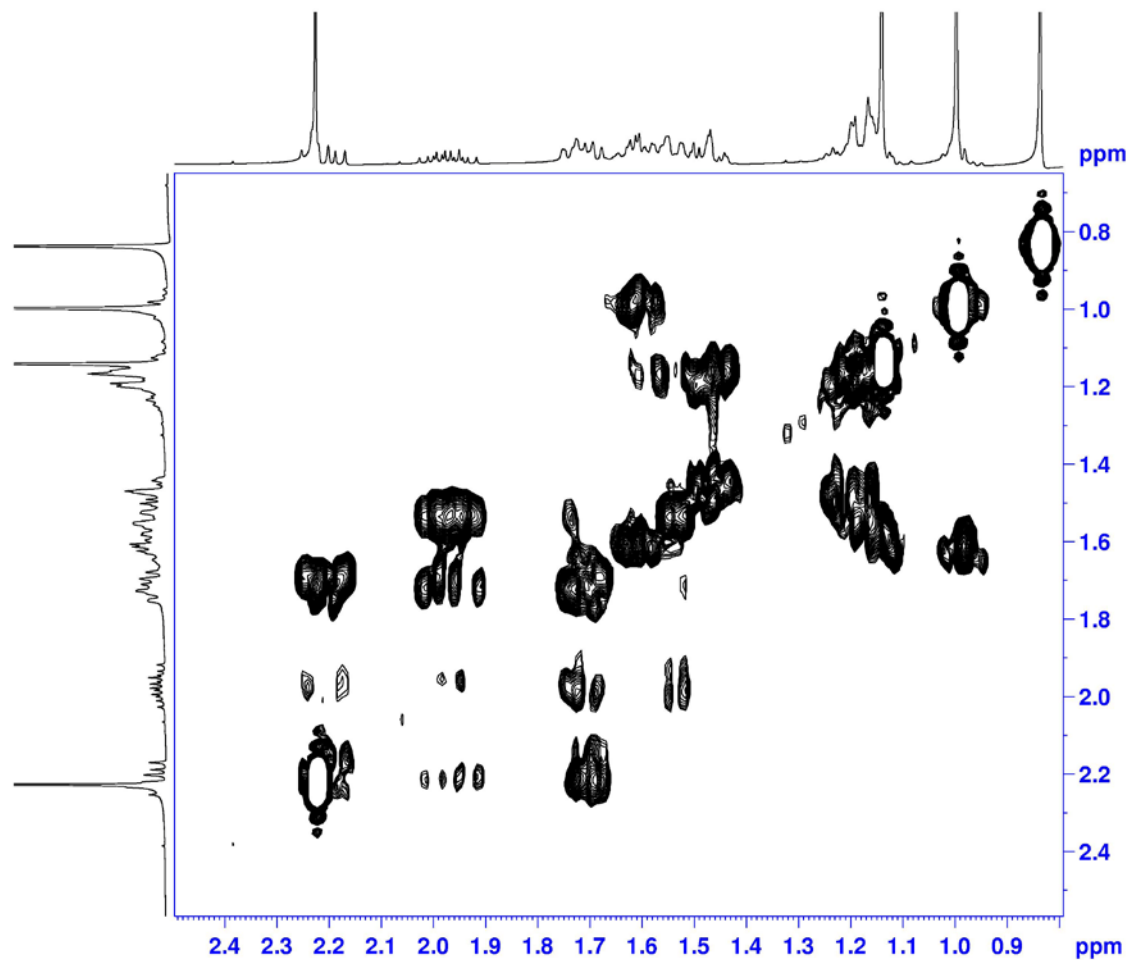
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NAME          R-35-2-2
EXPNO         4
PROCNO        1
Date_         20161017
Time          9.18
INSTRUM       spect
PROBHD        5 mm CPMAS BB
PULPROG       cosypppqf
TD            2048
SOLVENT       CDCl3
NS            8
DS            8
SWH           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            0.0000300 sec
D1            1.89678097 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D15           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
FnmMODE      QF
SI           1024
SF           400.1300998 MHz
WDW          QSI
SSB          0
LB           0.00 Hz
GB           0
PC           1.40
SI           1024
MC2          QF
SF           400.1300998 MHz
WDW          QSI
SSB          0
LB           0.00 Hz
GB           0
    
```

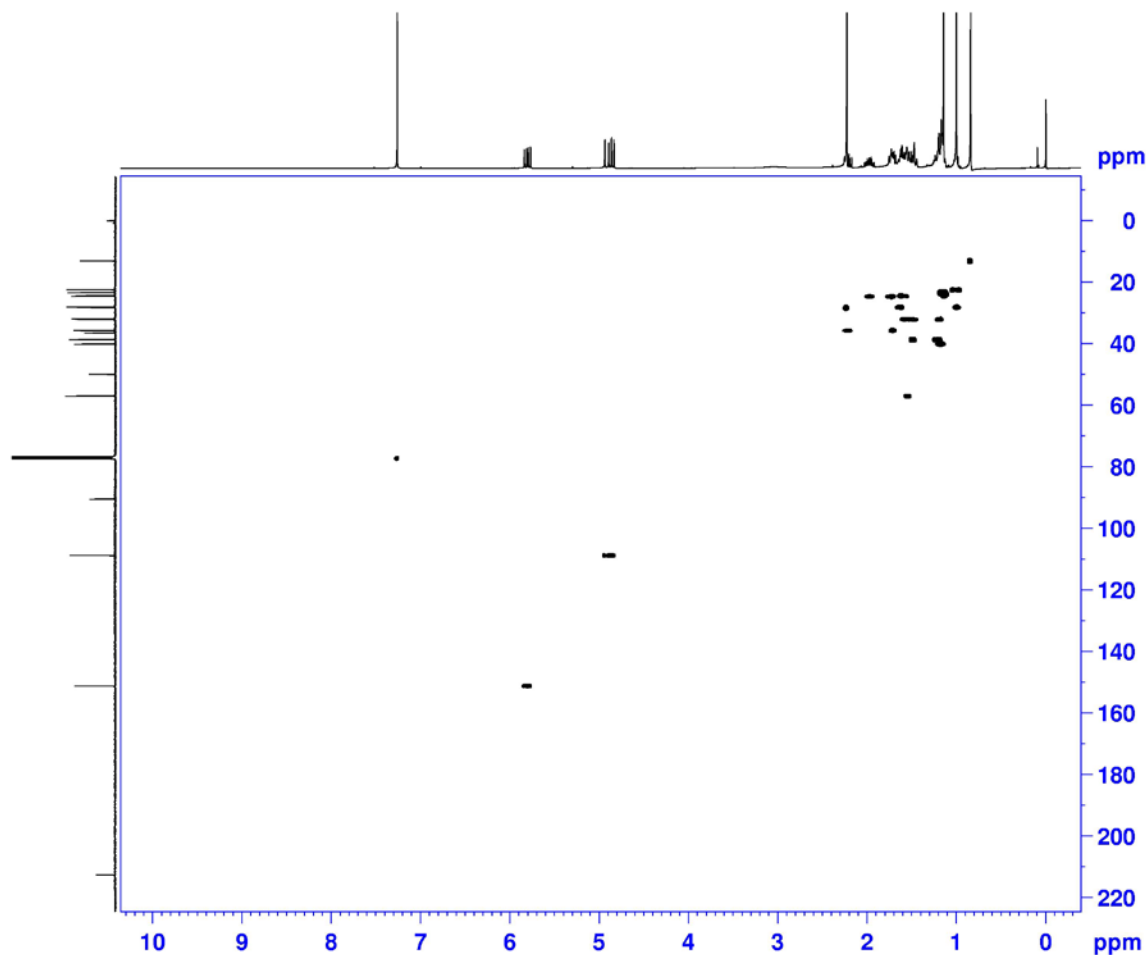
^1H - ^1H COSY (400 MHz) spectrum of compound 1 in CDCl_3



```
NAME R-35-2-2
EXPNO 4
PROCNO 1
Date_ 20161017
Time 9.18
INSTRUM spect
PROBHD 5 mm CPBPR03
PULPROG cosygpppgf
TD 2048
SOLVENT CDCl3
NS 8
DS 8
SWH 3906.250 Hz
FIDRES 1.307349 Hz
AQ 0.2621940 sec
RG 208.5
DW 128.000 usec
DE 10.00 usec
TE 297.0 K
D0 0.0000300 sec
D1 1.59678097 sec
D11 0.33000000 sec
D12 0.00002000 sec
D13 0.00004000 sec
D16 0.00020000 sec
IND 0.00025600 sec
```

```
----- CHANNEL f1 -----
SFO1 400.1318006 MHz
NUC1 1H
P0 11.50 usec
P1 11.50 usec
P17 2500.00 usec
NDO 1
TD 128
SFO1 400.1318 MHz
FIDRES 30.517578 Hz
SW 9.762 ppm
FRMODE QF
SI 1024
SF 400.1300098 MHz
WDW OSINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 400.1300098 MHz
WDW OSINE
SSB 0
LB 0.00 Hz
GB 0
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HSQC (400 MHz) spectrum of compound 1 in CDCl₃

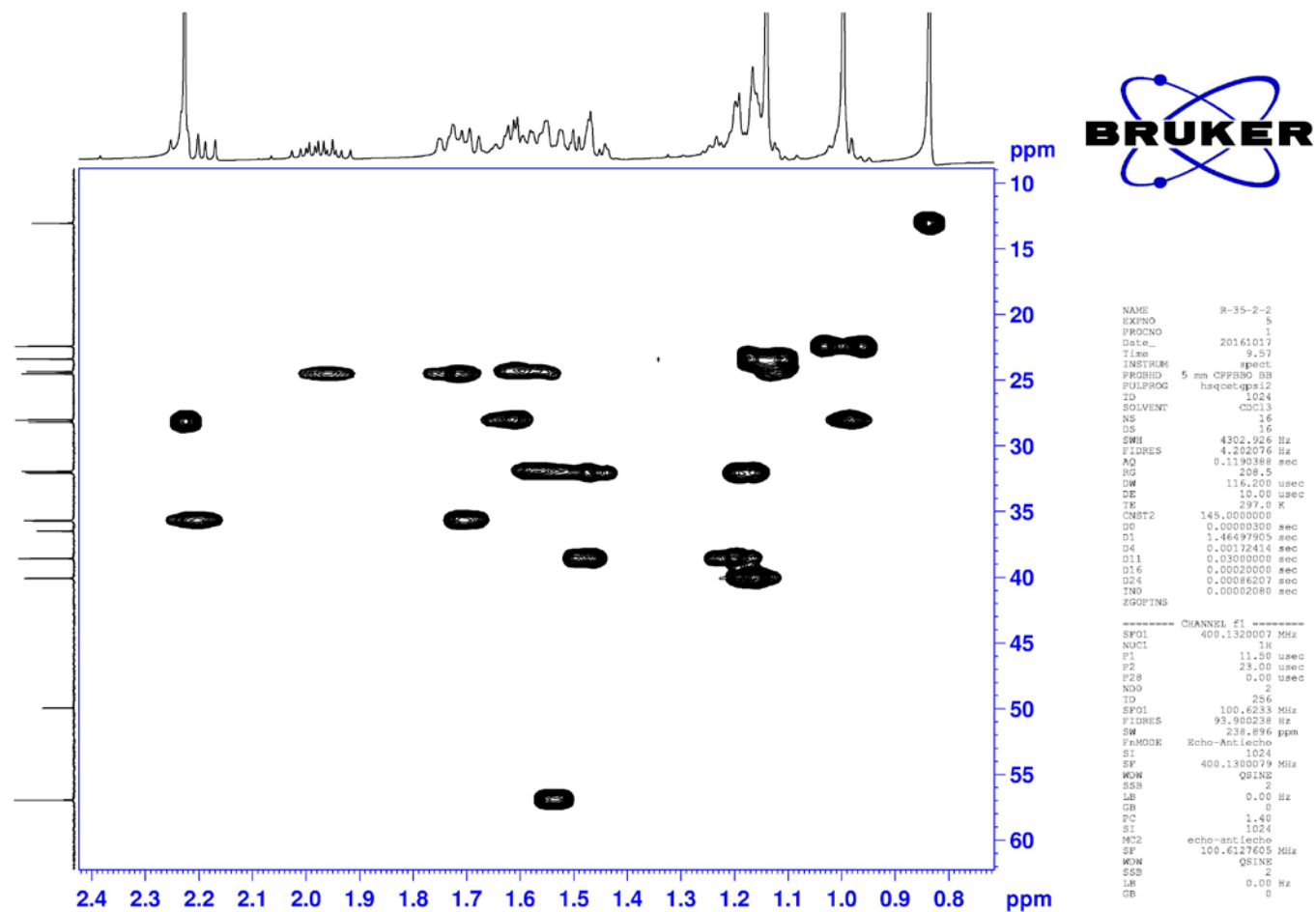


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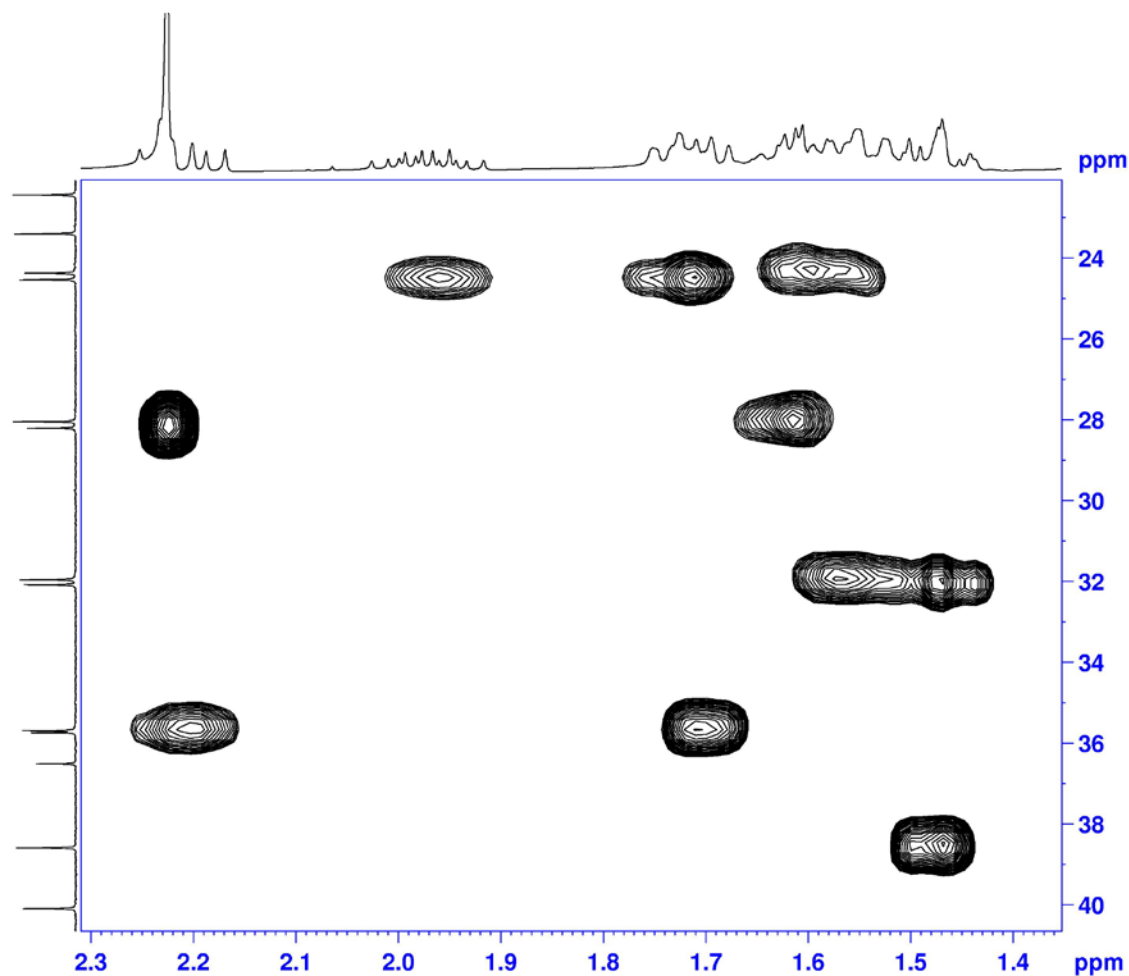
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EXPNO         5
PROCNO        1
Date_         20161017
Time          9.57
INSTRUM       spect
PROBHD        5 mm CPFBBO BB
PULPROG       hsqcetgps12
TD            1024
SOLVENT       CDCl3
NS            16
DS            16
SWH           4302.926 Hz
FIDRES        4.202076 Hz
AQ            0.1190388 sec
RG            208.5
DW            116.200 usec
DE            10.00 usec
TE            297.0 K
CNS12         148.0000000
D0            0.00000300 sec
D1            1.46497905 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.00086207 sec
IND           0.00002080 sec
ZGPG1NS

----- CHANNEL f1 -----
SF01          400.1320007 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
P2B           0.00 usec
ND0           2
TD            256
SF01          100.6233 MHz
FIDRES        93.900238 Hz
DW            236.896 ppm
F1MODE        Echo-AntiLecho
SI            1024
SF            400.1300079 MHz
WDW           QSIINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antilecho
SF            100.6127660 MHz
WDW           QSIINE
SSB           2
LB            0.00 Hz
GB            0
    
```


HSQC (400 MHz) spectrum of compound 1 in CDCl₃



HSQC (400 MHz) spectrum of compound 1 in CDCl₃

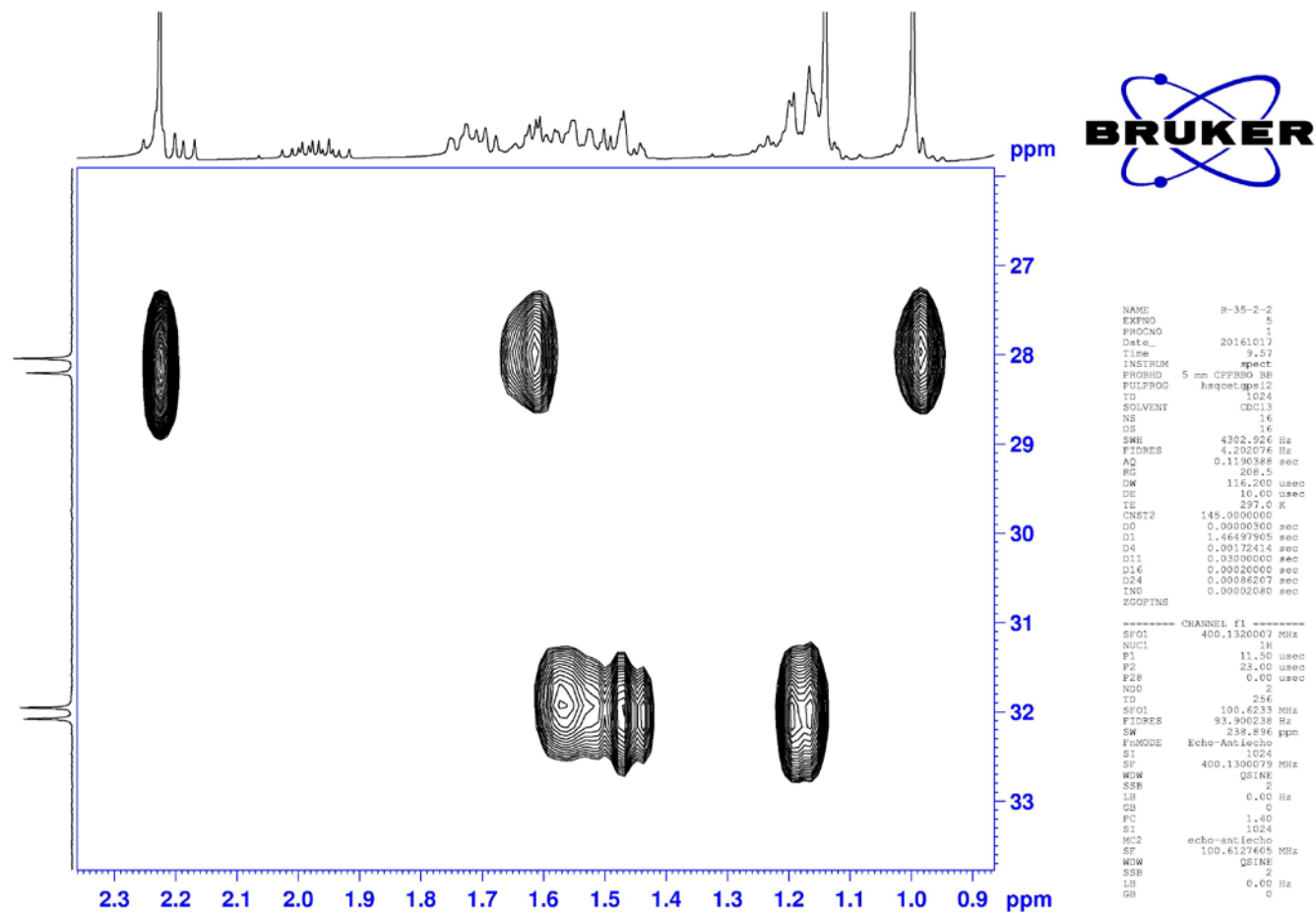


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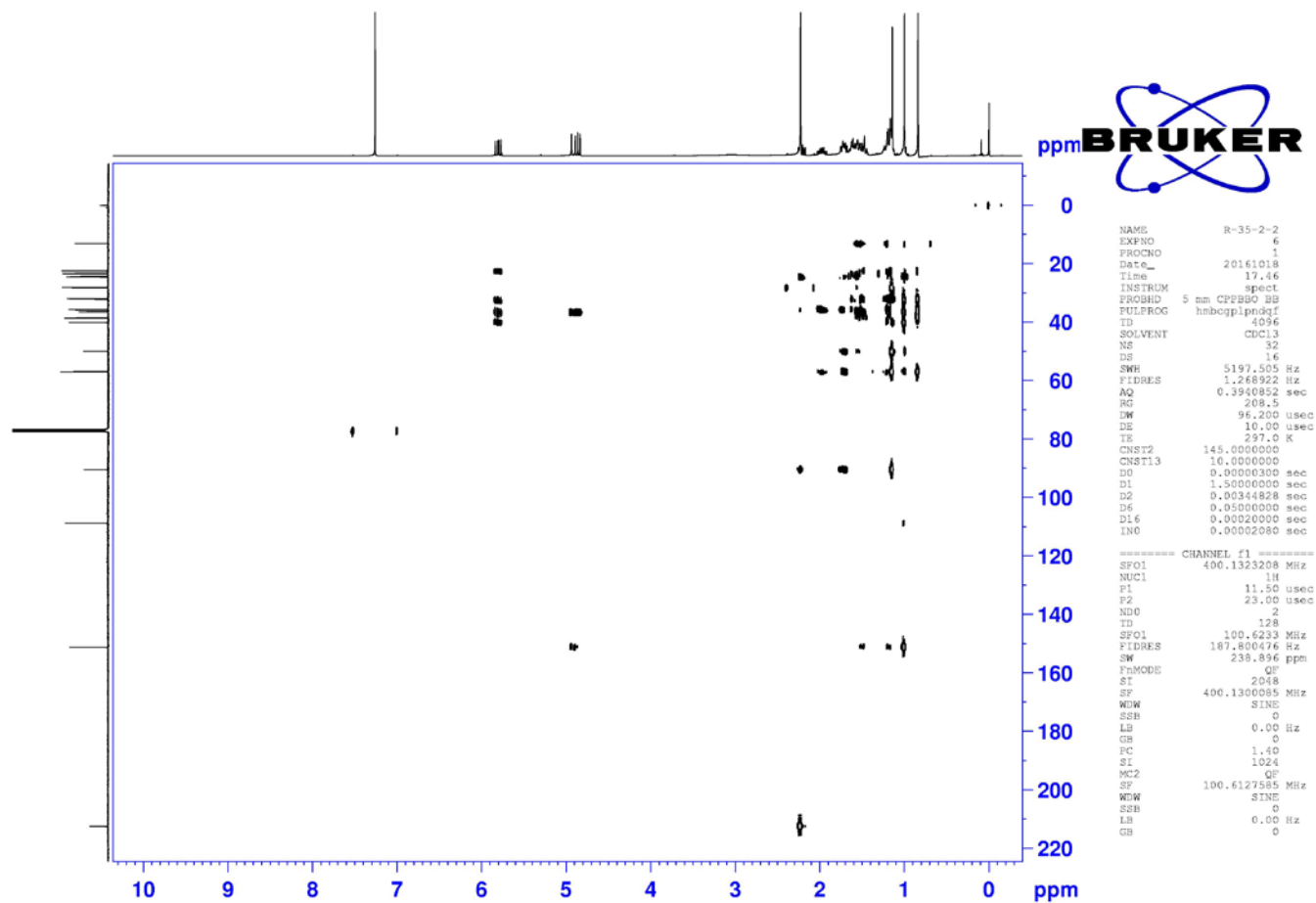
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EXPNO         5
PROCNO        1
Date_         20161017
Time         9.57
INSTRUM       spect
PROBHD        5 mm CPBBO BB
PULPROG       hsqcetgpa12
ID            1024
SOLVENT       CDCl3
NS            16
DS            16
SWH           4302.926 Hz
FIDRES        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
IND           0.00032000 sec
ZGPGTNS

----- CHANNEL f1 -----
SFO1          400.1320007 MHz
NUC1          13C
P1            11.50 usec
P2            23.00 usec
F28           0.00 usec
ND0           2
TD            256
SFO1          100.6283 MHz
FIDRES        93.900238 Hz
SW            238.896 ppm
F1MODE        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            100.6127605 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
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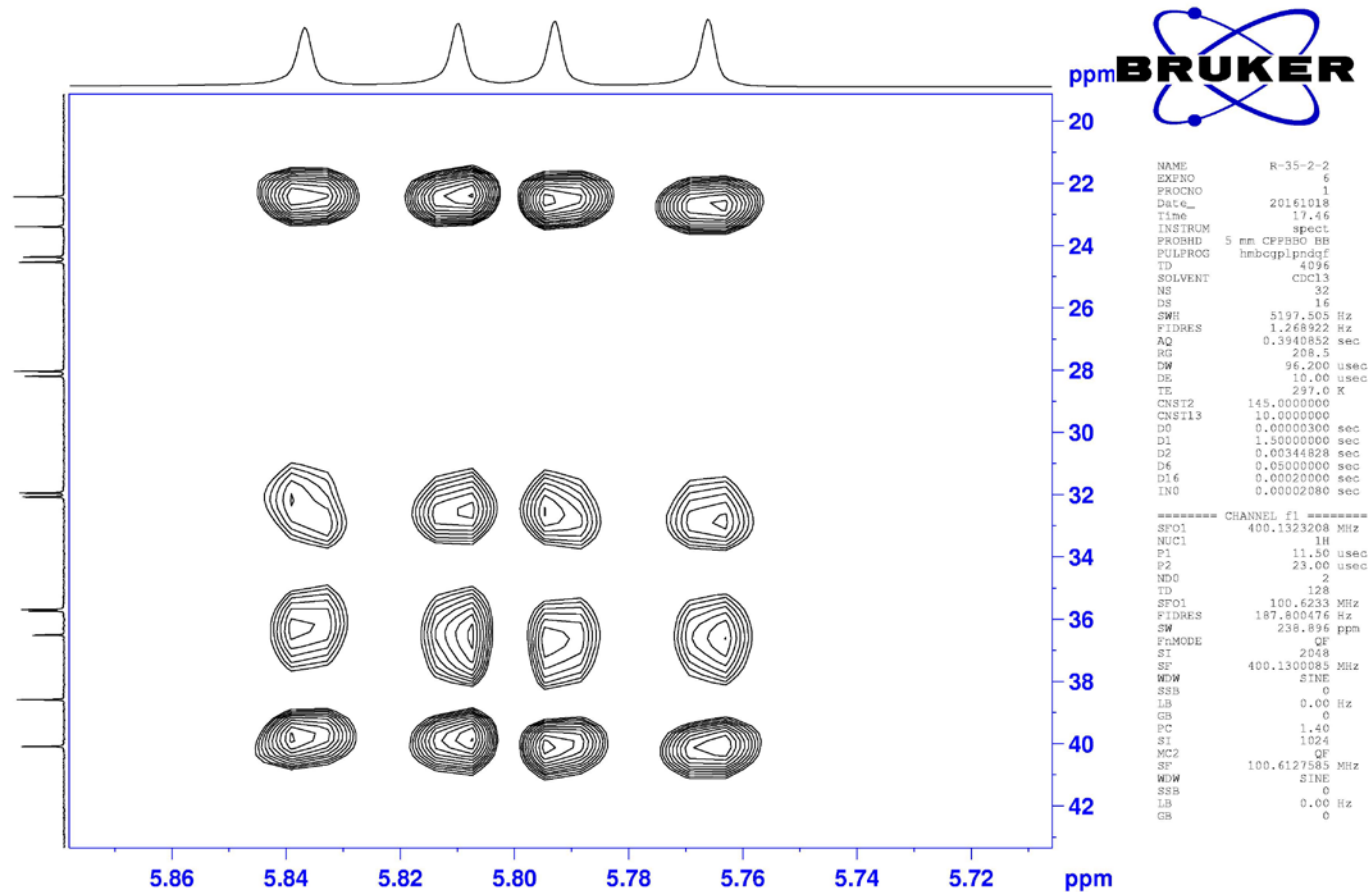
HSQC (400 MHz) spectrum of compound 1 in CDCl₃



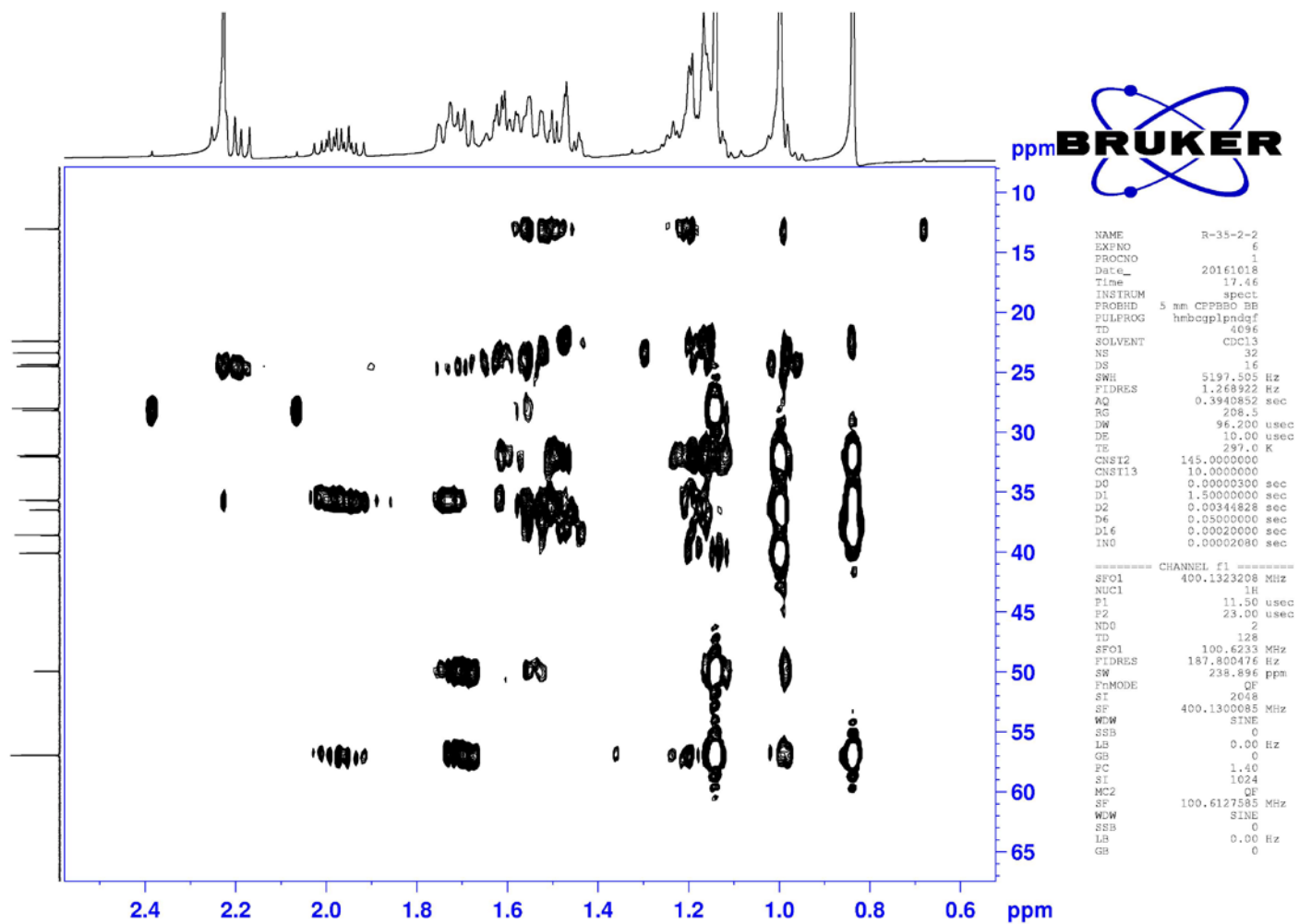
HMBC (400 MHz) spectrum of compound 1 in CDCl₃



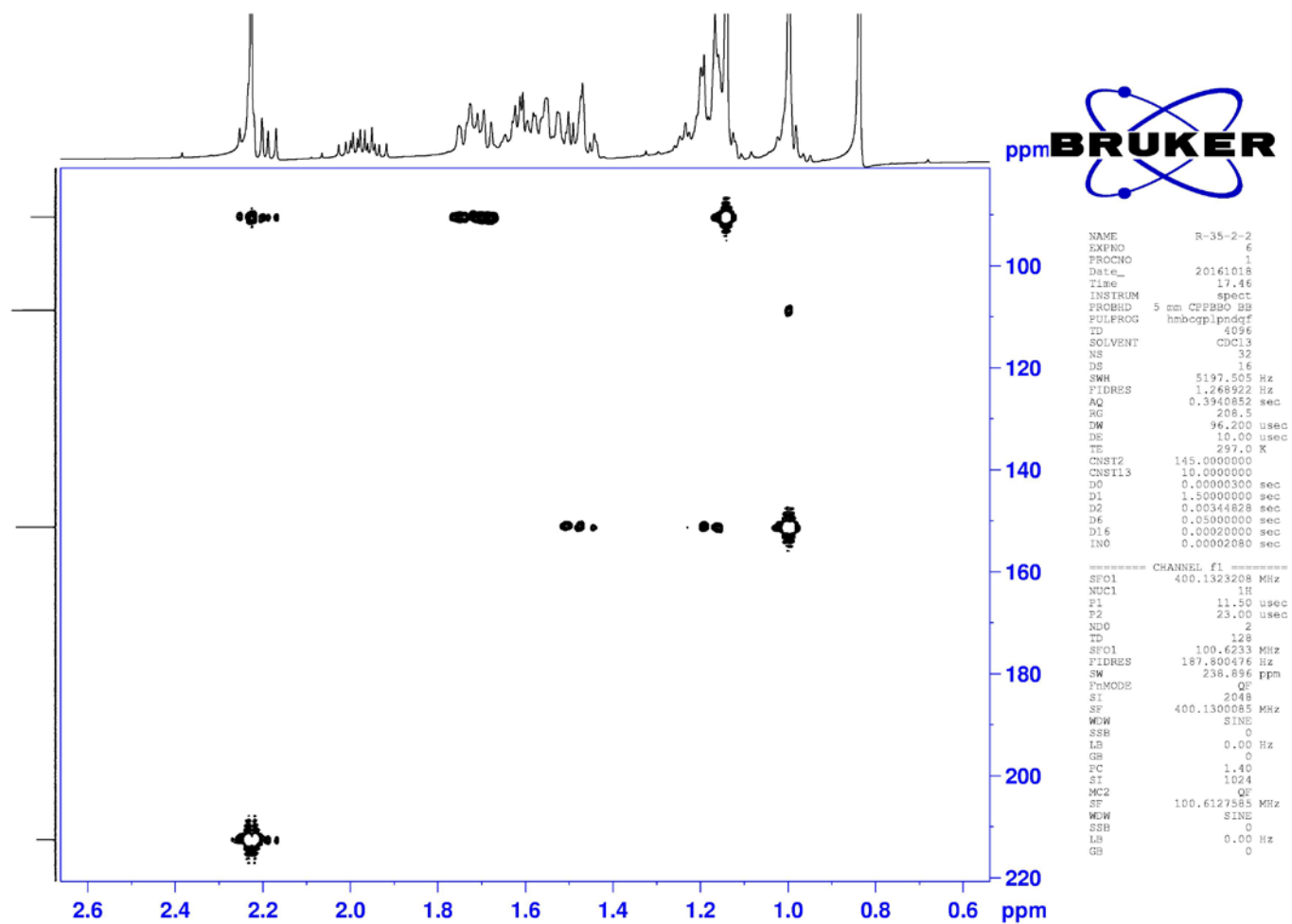
HMBC (400 MHz) spectrum of compound 1 in CDCl₃



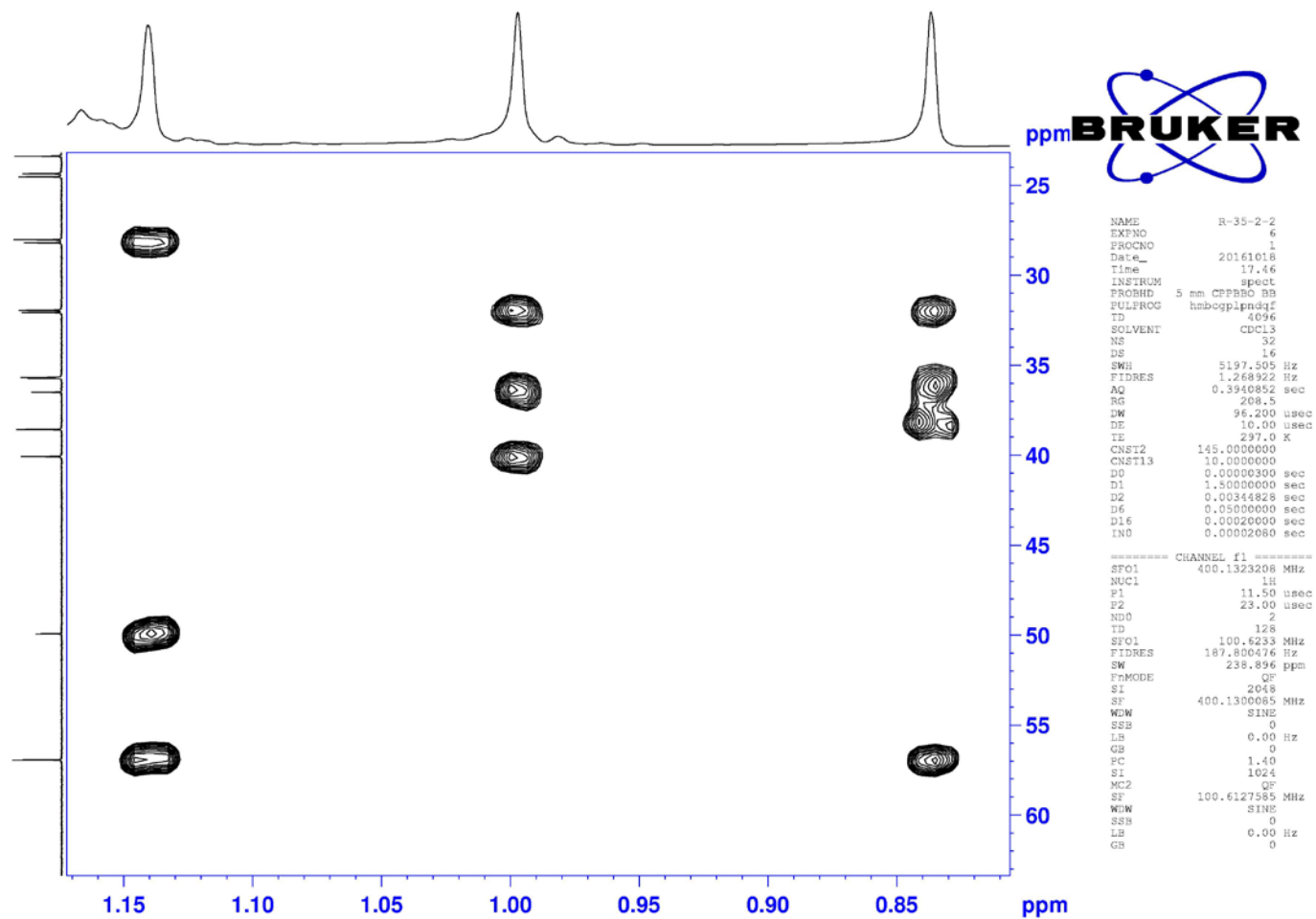
HMBC (400 MHz) spectrum of compound 1 in CDCl₃



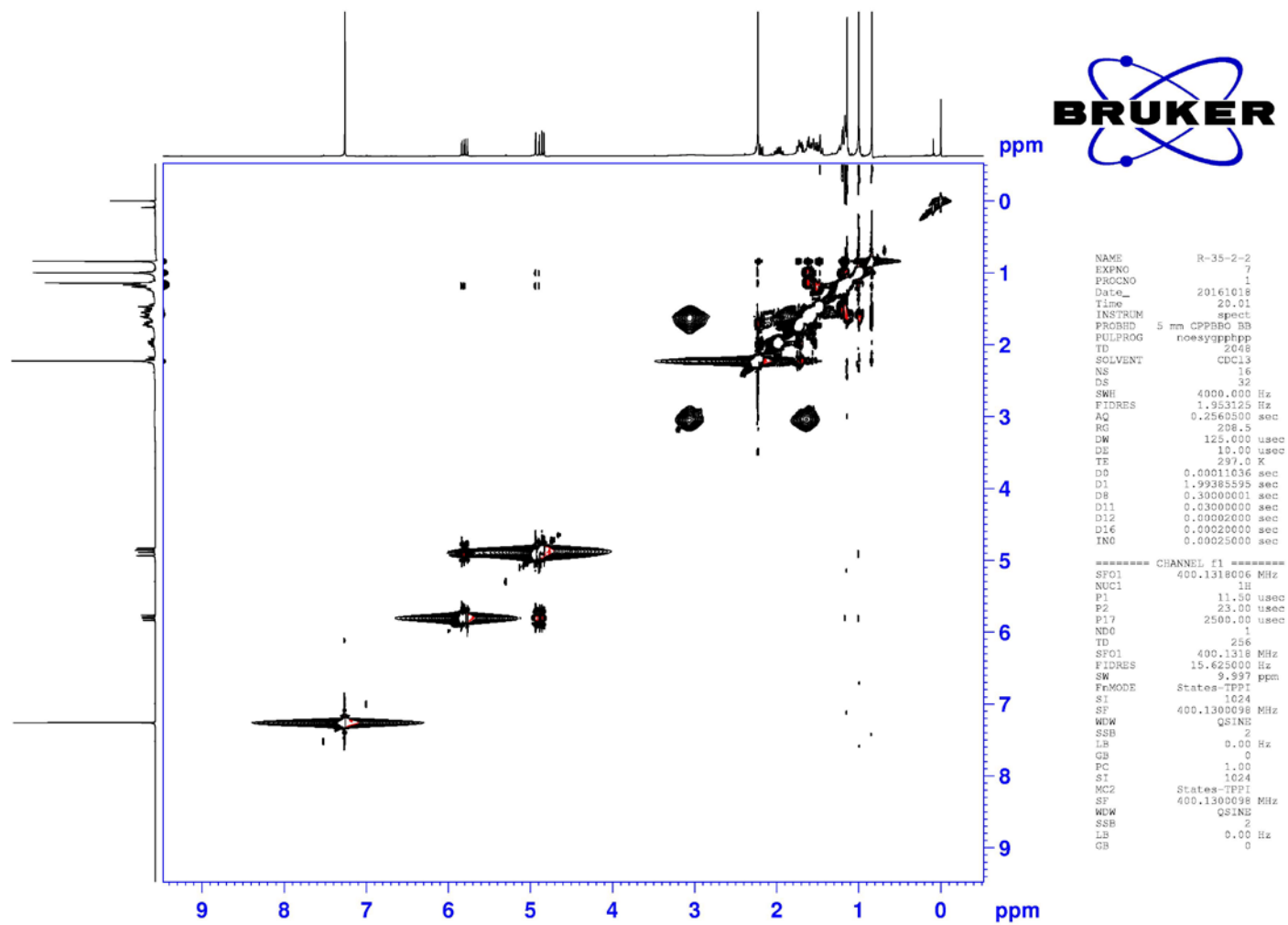
HMBC (400 MHz) spectrum of compound 1 in CDCl₃



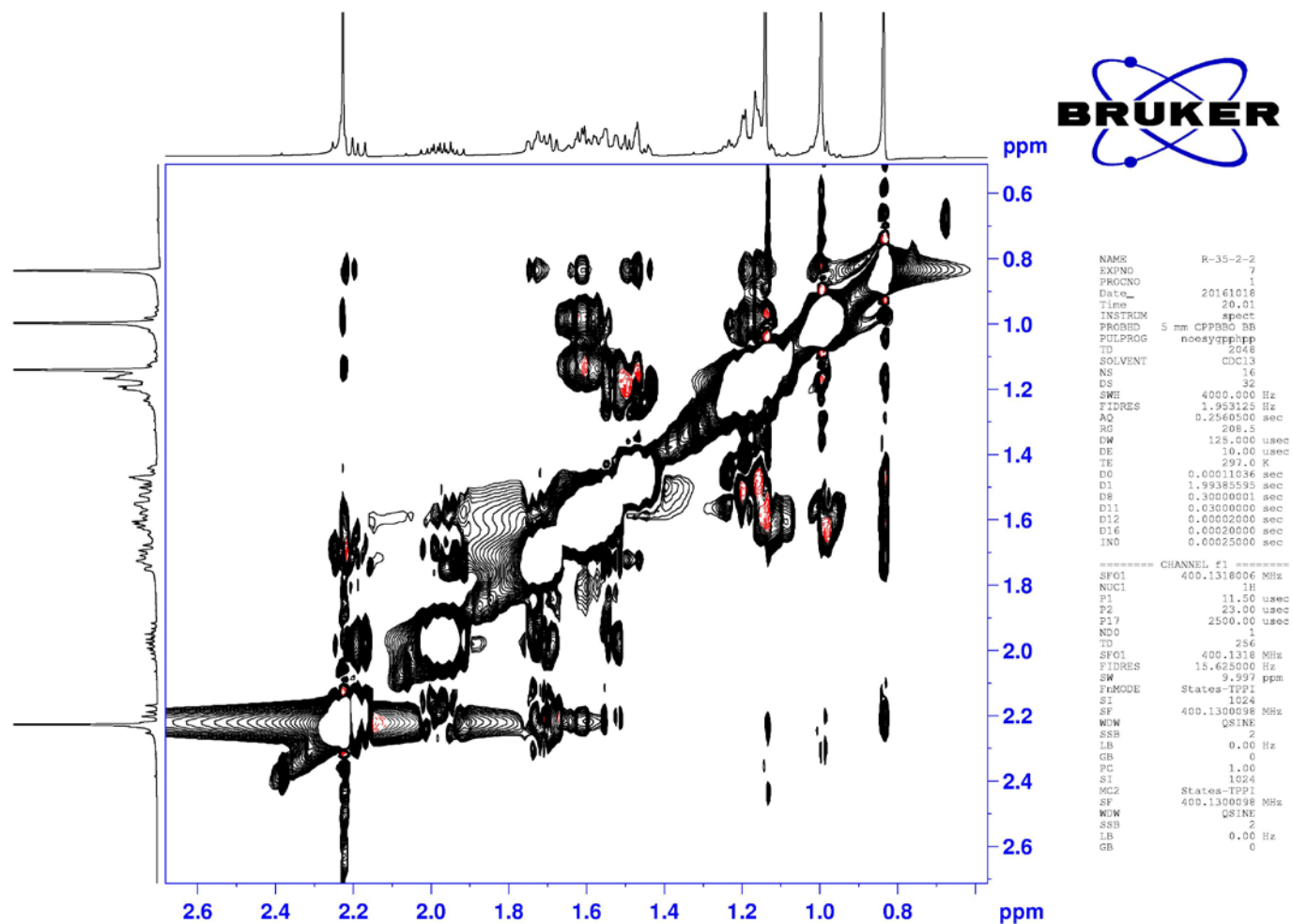
HMBC (400 MHz) spectrum of compound 1 in CDCl₃



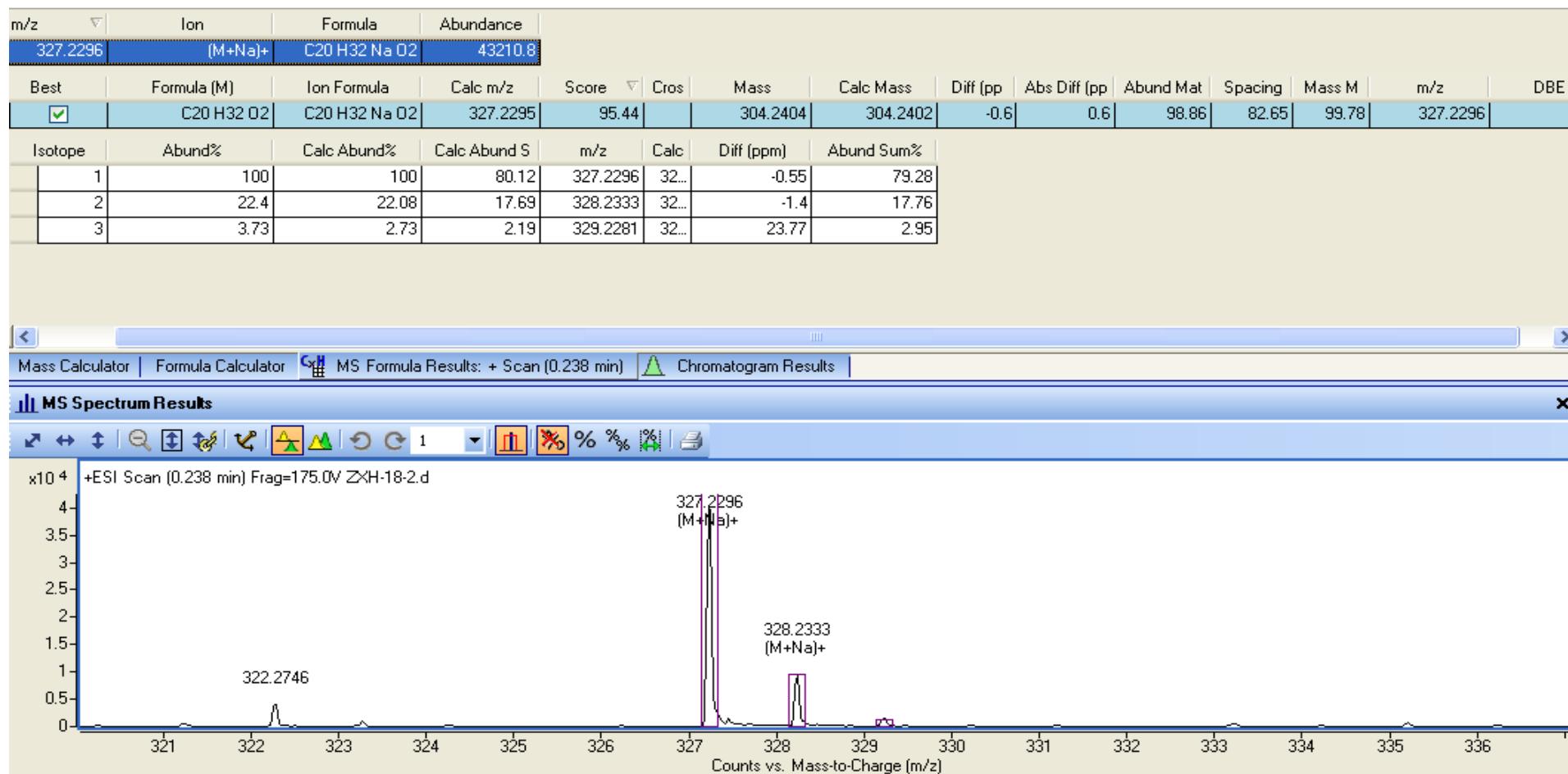
NOESY (400 MHz) spectrum of compound 1 in CDCl₃



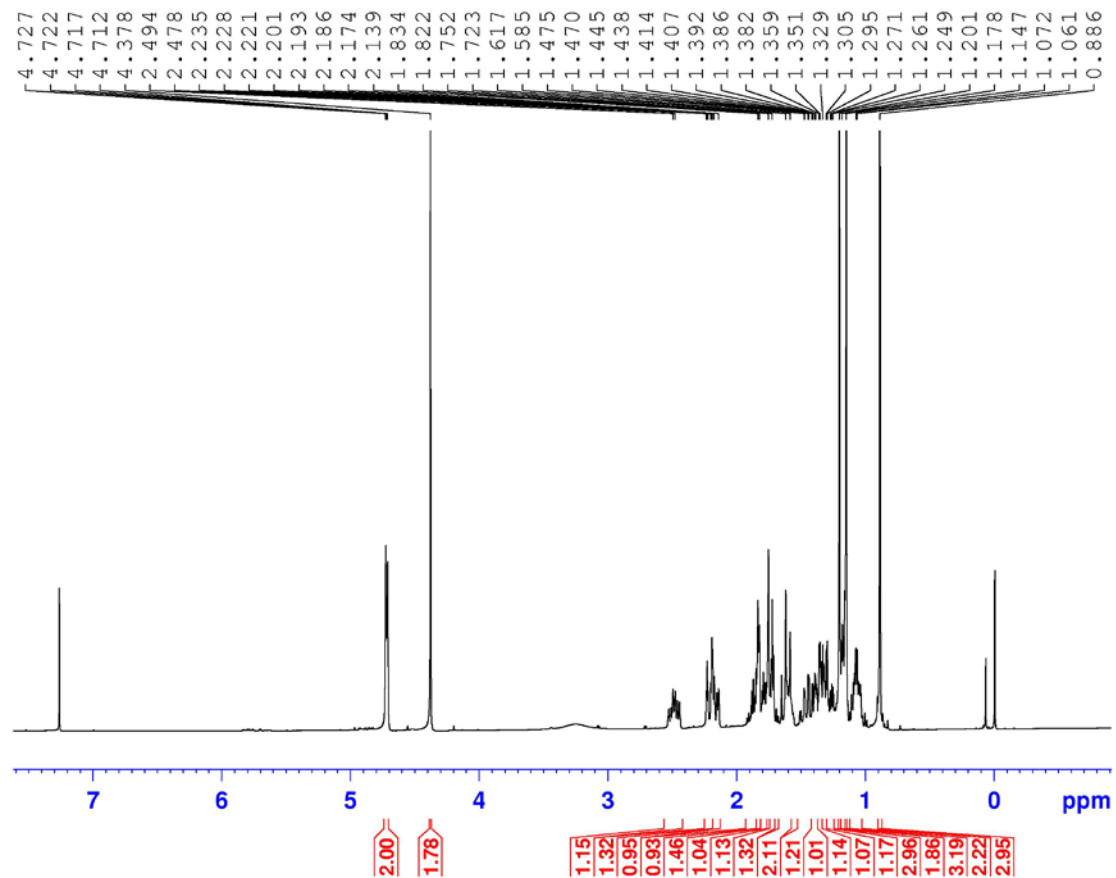
NOESY (400 MHz) spectrum of compound 1 in CDCl₃



HR-ESIMS for compound 2



HR-ESIMS for compound 2

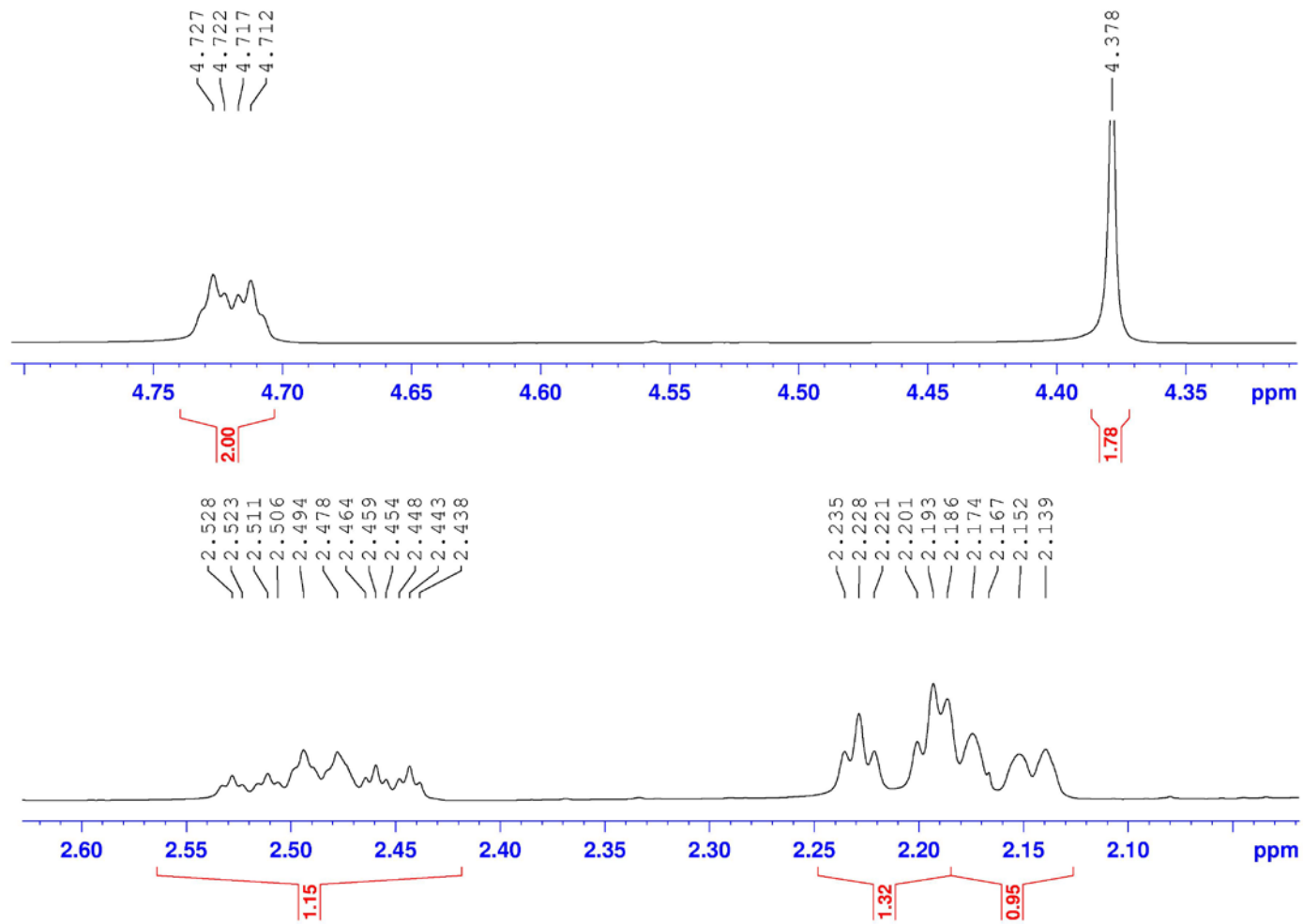


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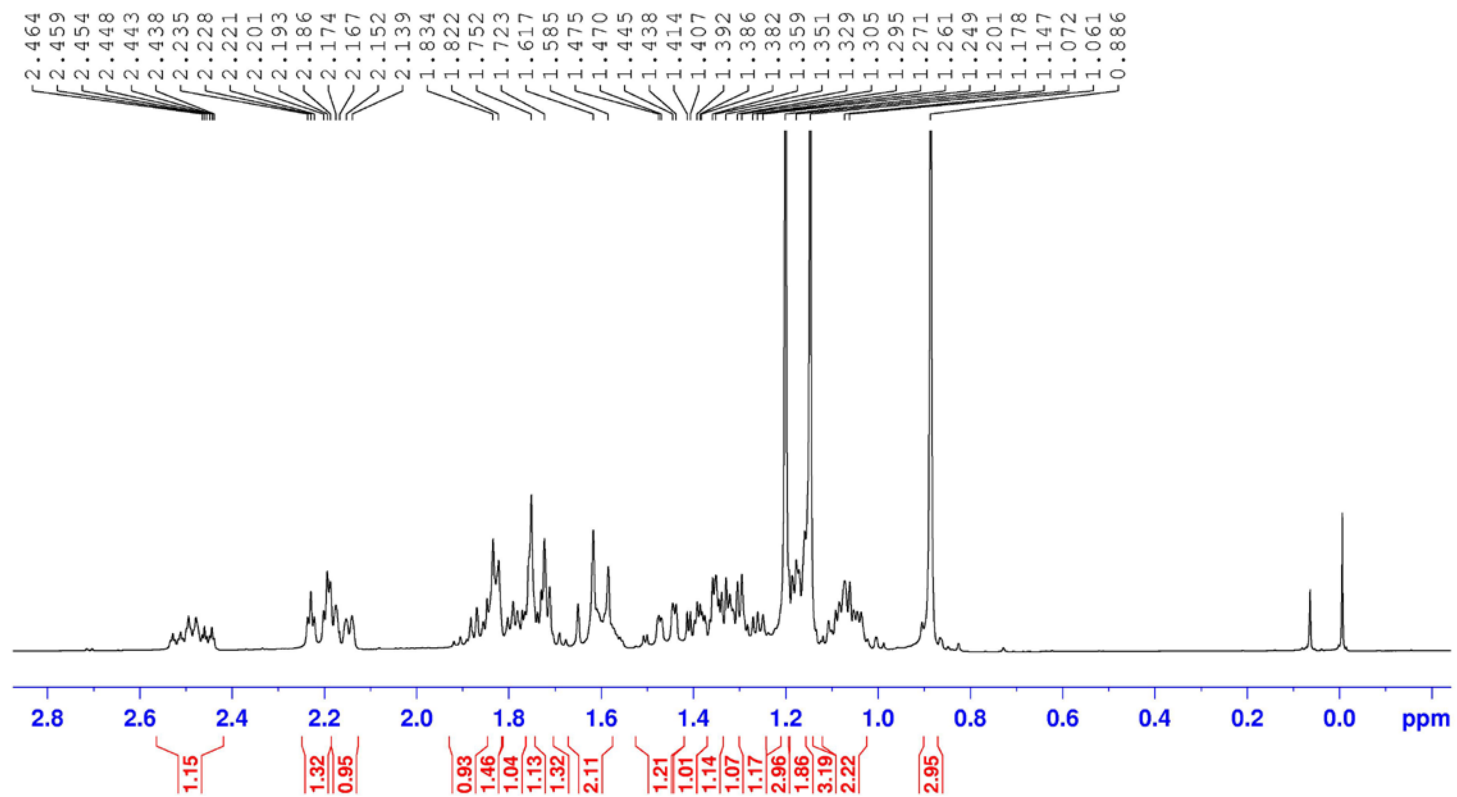
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EXPNO         101
PROCNO        1
Date_         20160113
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PULPROG       zg30
ID            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            51.23
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            400.1300099 MHz
WDW           EM
SSB           0
LB            0.30 Hz
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PC            1.00
    
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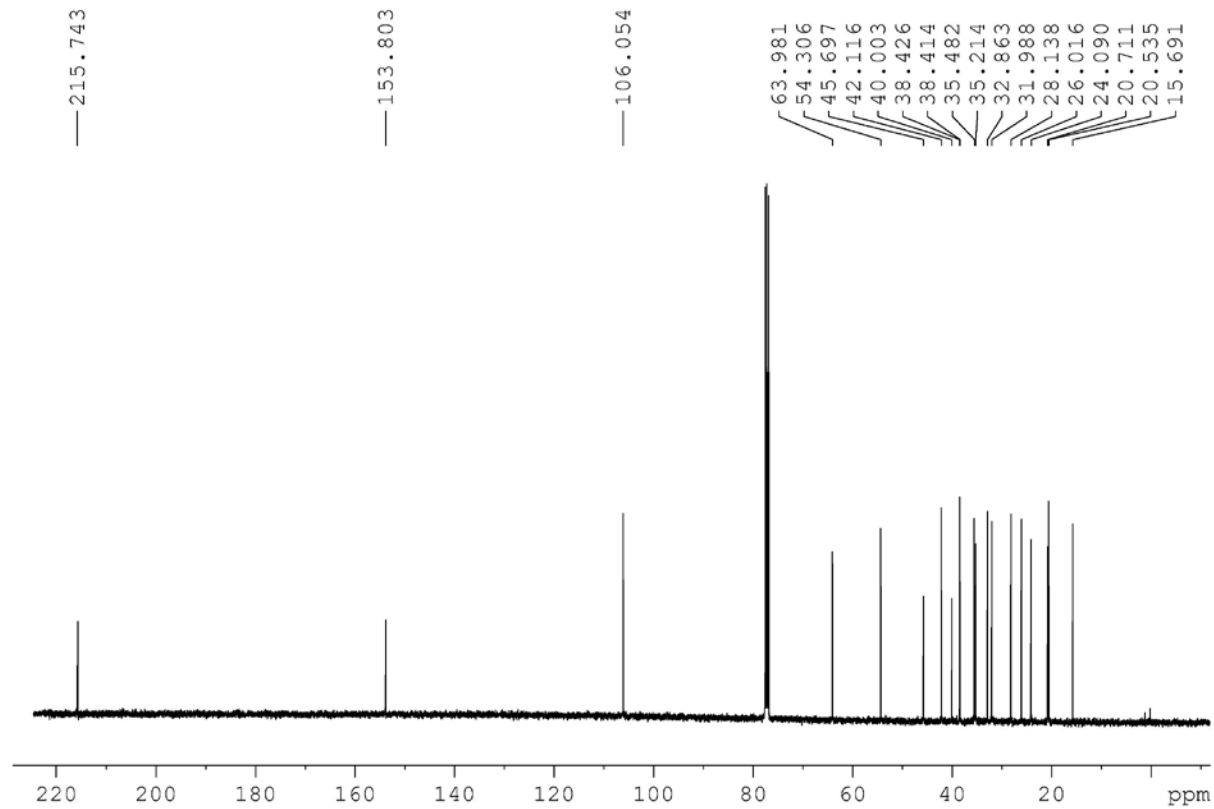
^1H NMR (400 MHz) spectrum of compound 2 in CDCl_3



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

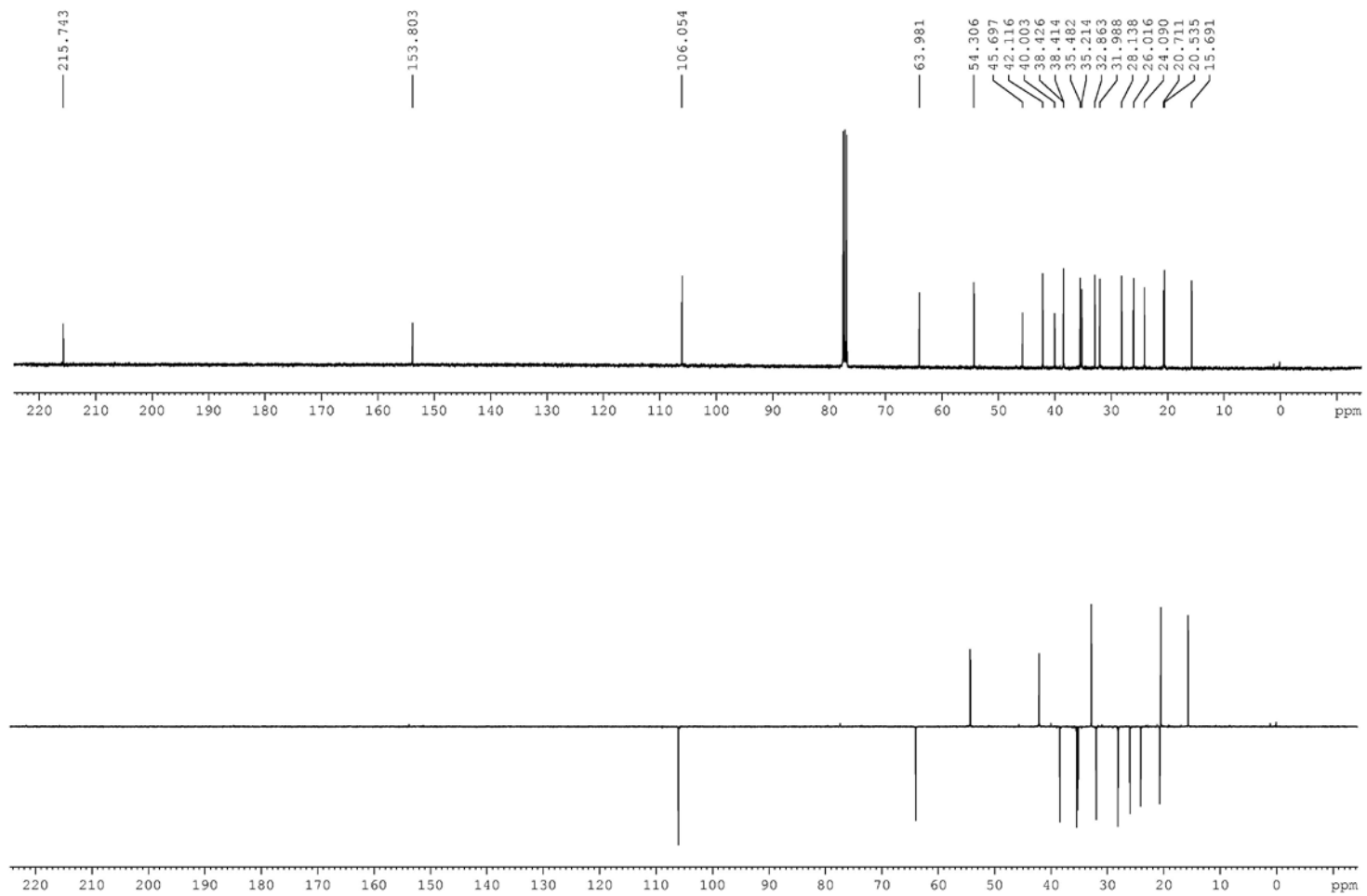


^{13}C NMR (100 MHz) spectrum of compound 2 in CDCl_3

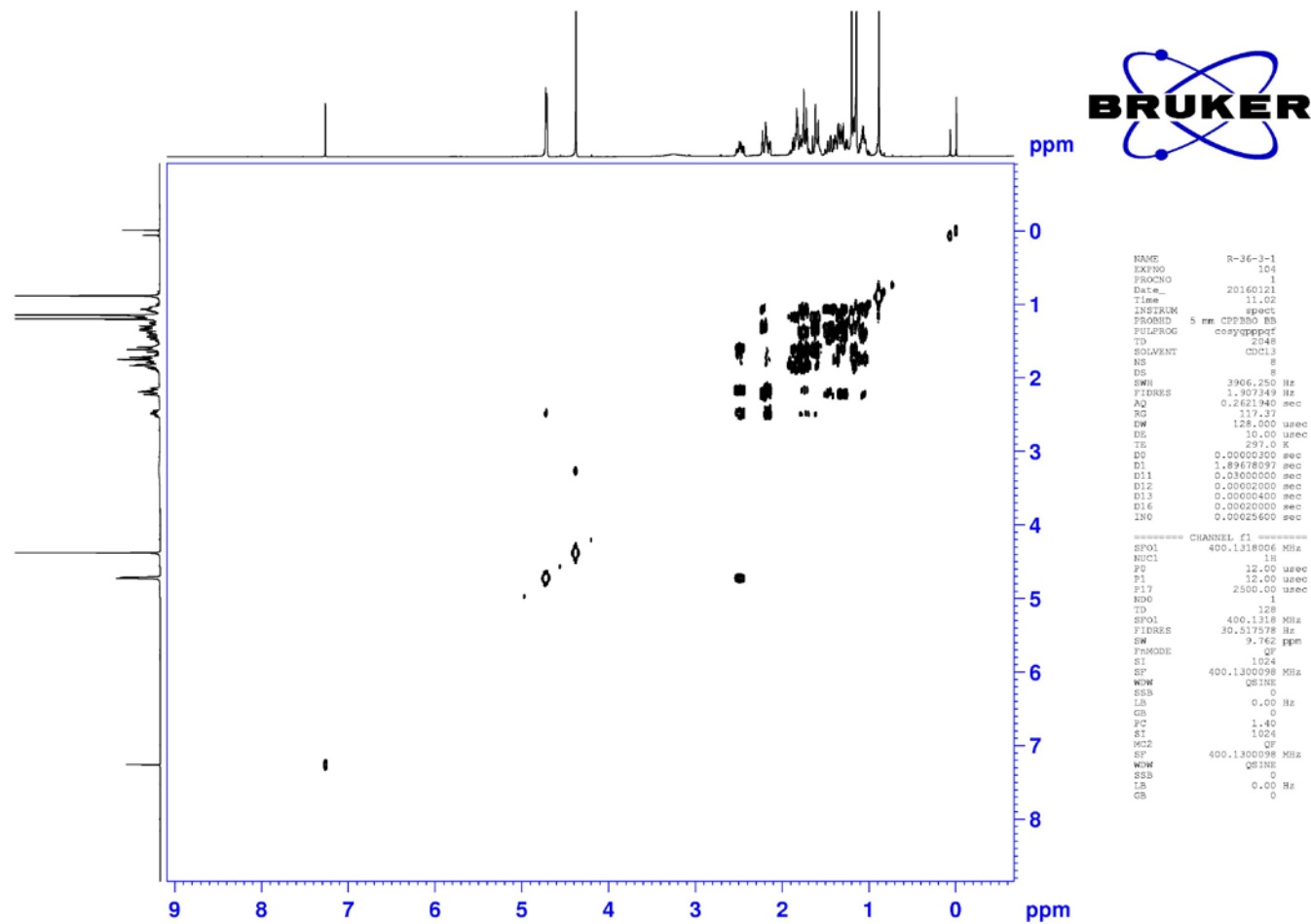


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PROCNO 1
Date_ 20160113
Time 18.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT cdcl3
NS 88
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 147.94
DW 20.800 usec
DE 18.00 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1
===== CHANNEL f1 =====
SFO1 100.6233324 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127563 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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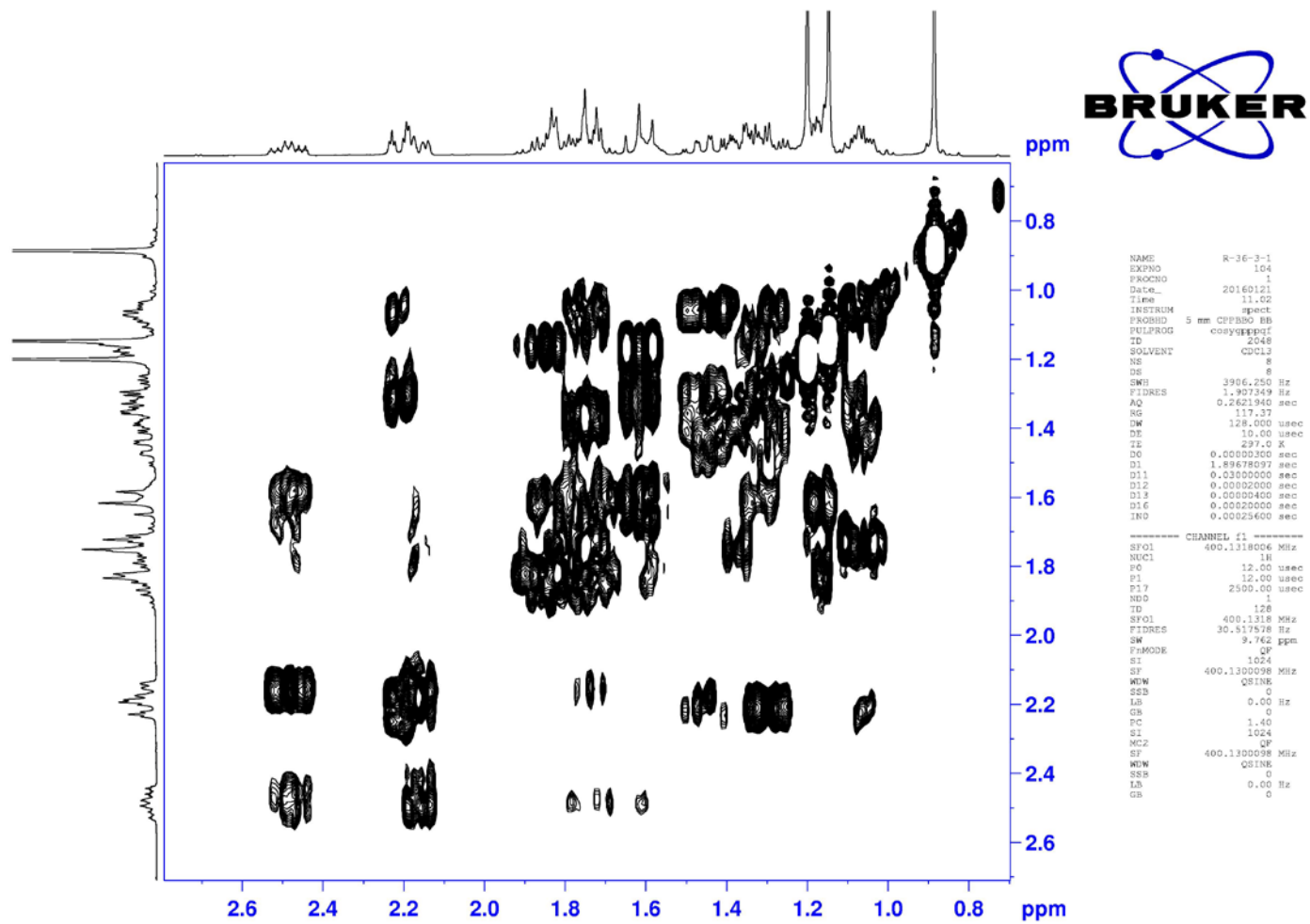
DEPT135 (100 MHz) spectrum of compound 2 in CDCl₃



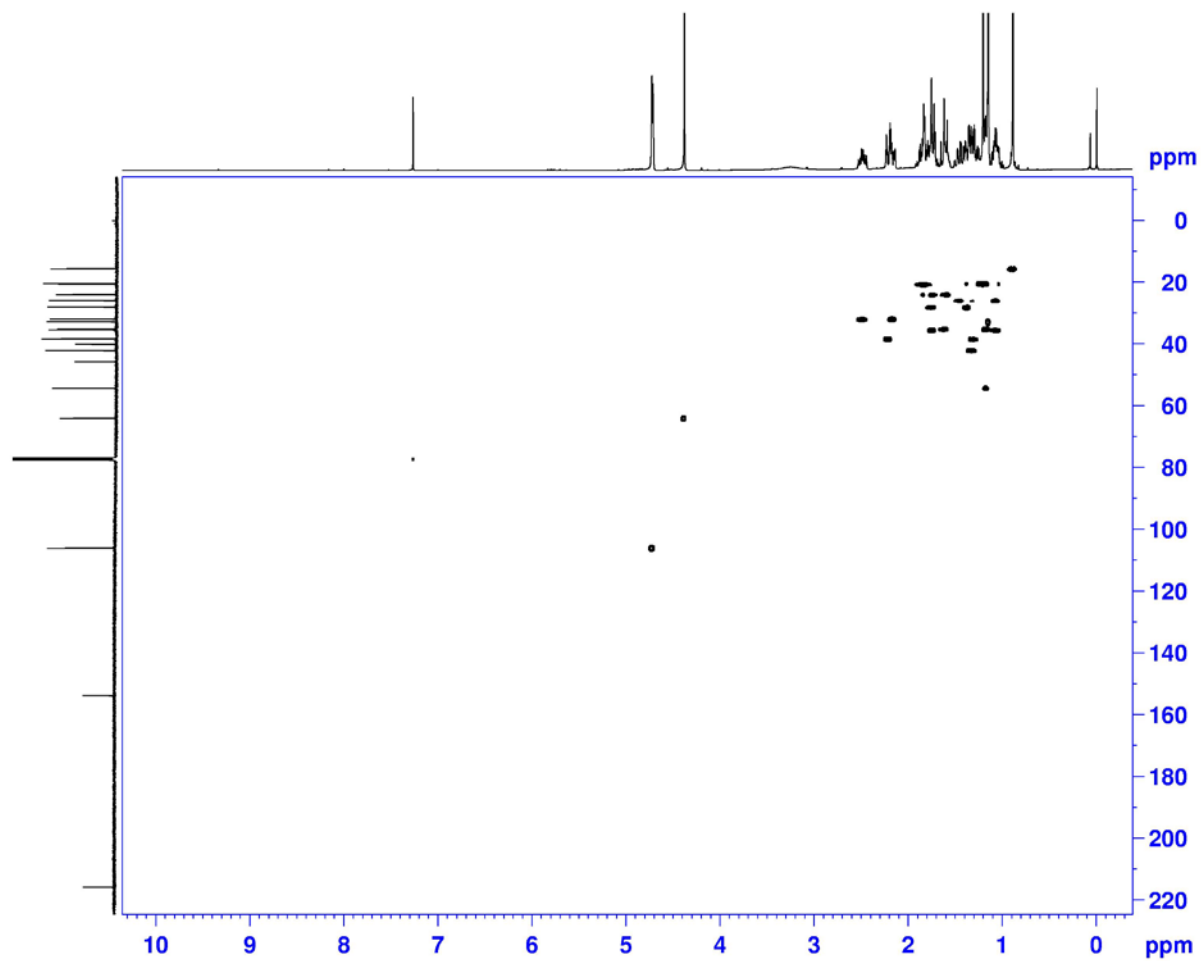
^1H - ^1H COSY (400 MHz) spectrum of compound 2 in CDCl_3



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



HSQC (400 MHz) spectrum of compound 2 in CDCl₃

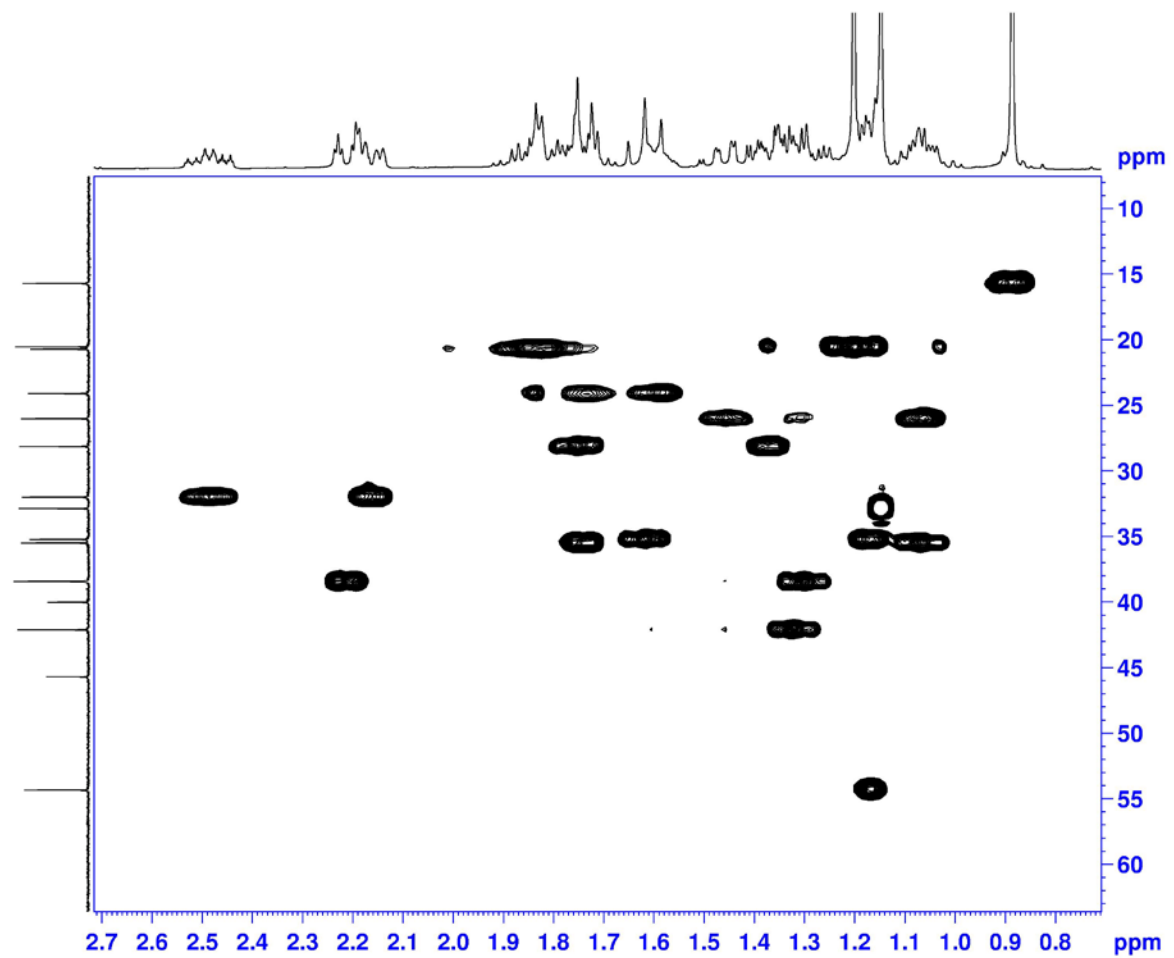


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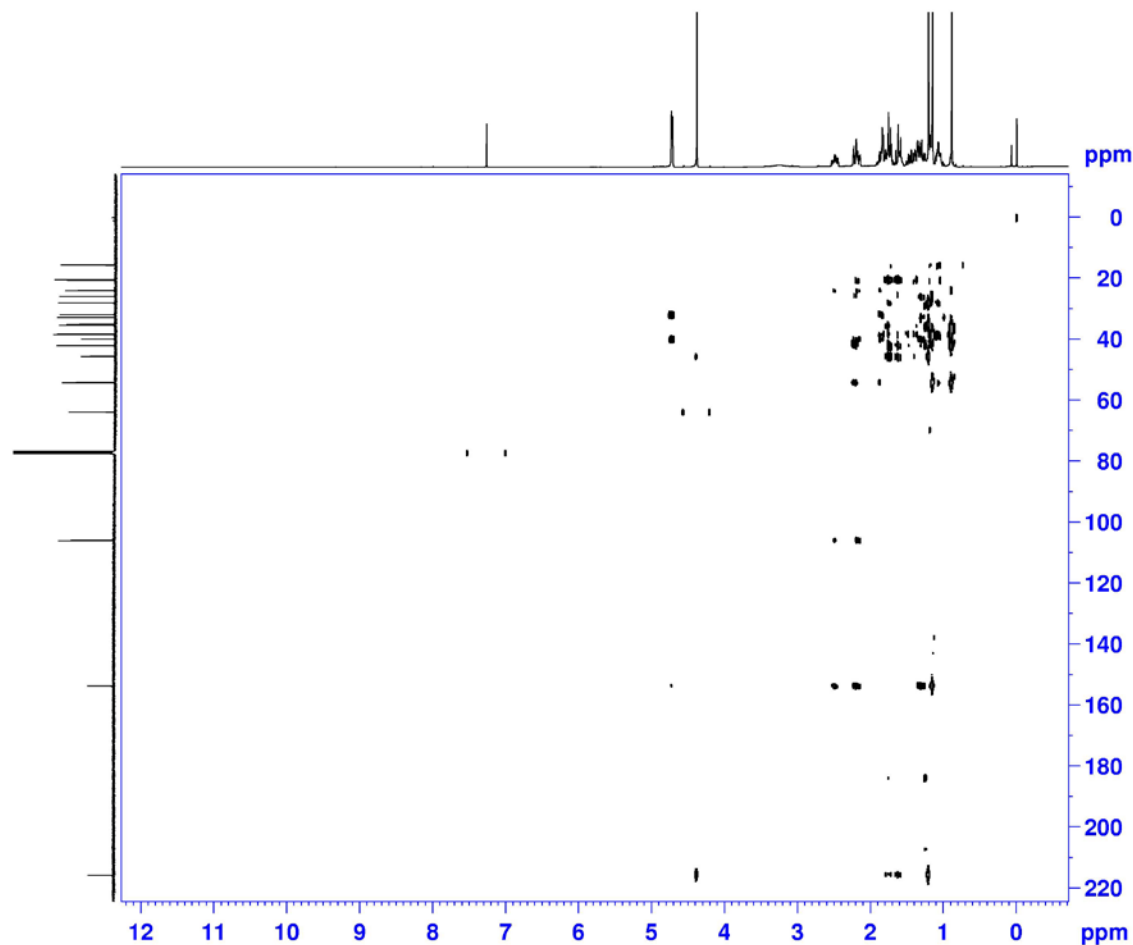
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EXPNO         105
PROCNO        1
Date_         20160121
Time         11.41
INSTRUM       spect
PROBHD        5 mm CPBBD0
PULPROG       haqetcpa12
TD            1024
SOLVENT       CDCl3
NS            16
DS            16
SWE           4302.926 Hz
FIDRES        4.262076 Hz
AQ            0.1190388 sec
RG            298.5
DW            116.200 usec
DE            19.00 usec
TE            297.0 K
CNS12         145.0000000
D0            0.0000300 sec
D1            1.46497905 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D24           0.0008207 sec
IN0           0.00002080 sec
ZG0PTNS

===== CHANNEL f1 =====
SFO1          400.1320007 MHz
NUC1          1H
P1            12.00 usec
P2            24.00 usec
P28           0.00 usec
NDG           2
TD            256
SFO1          100.6233 MHz
FIDRES        93.960238 Hz
SW            238.896 ppm
PRMODE        Echo-Antiecho
SI            1024
SF            400.1300080 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            100.6127475 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
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HSQC (400 MHz) spectrum of compound 2 in CDCl₃



HMBC (400 MHz) spectrum of compound 2 in CDCl₃

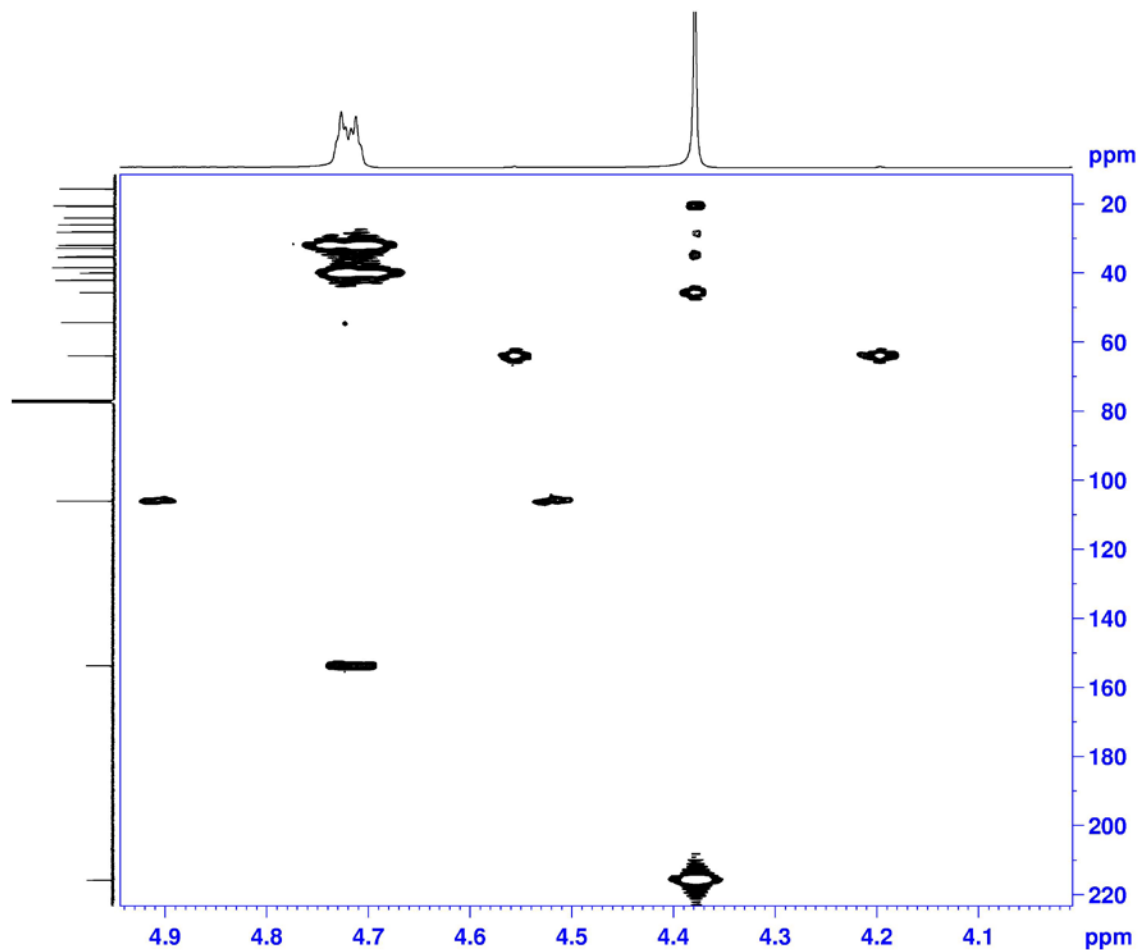


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NAME          R-36-3-1
EXFNO         1006
PROCNO        1
Date_         20160419
Time          20.52
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcgp1pndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
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           0.00620000 sec
IN0           0.00002080 sec

----- CHANNEL f1 -----
SF01          400.1323208 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
ND0           2
TD            128
SF01          100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
FmMODE        QF
SI            2048
SF            400.1300110 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            100.6127565 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
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HMBC (400 MHz) spectrum of compound 2 in CDCl₃

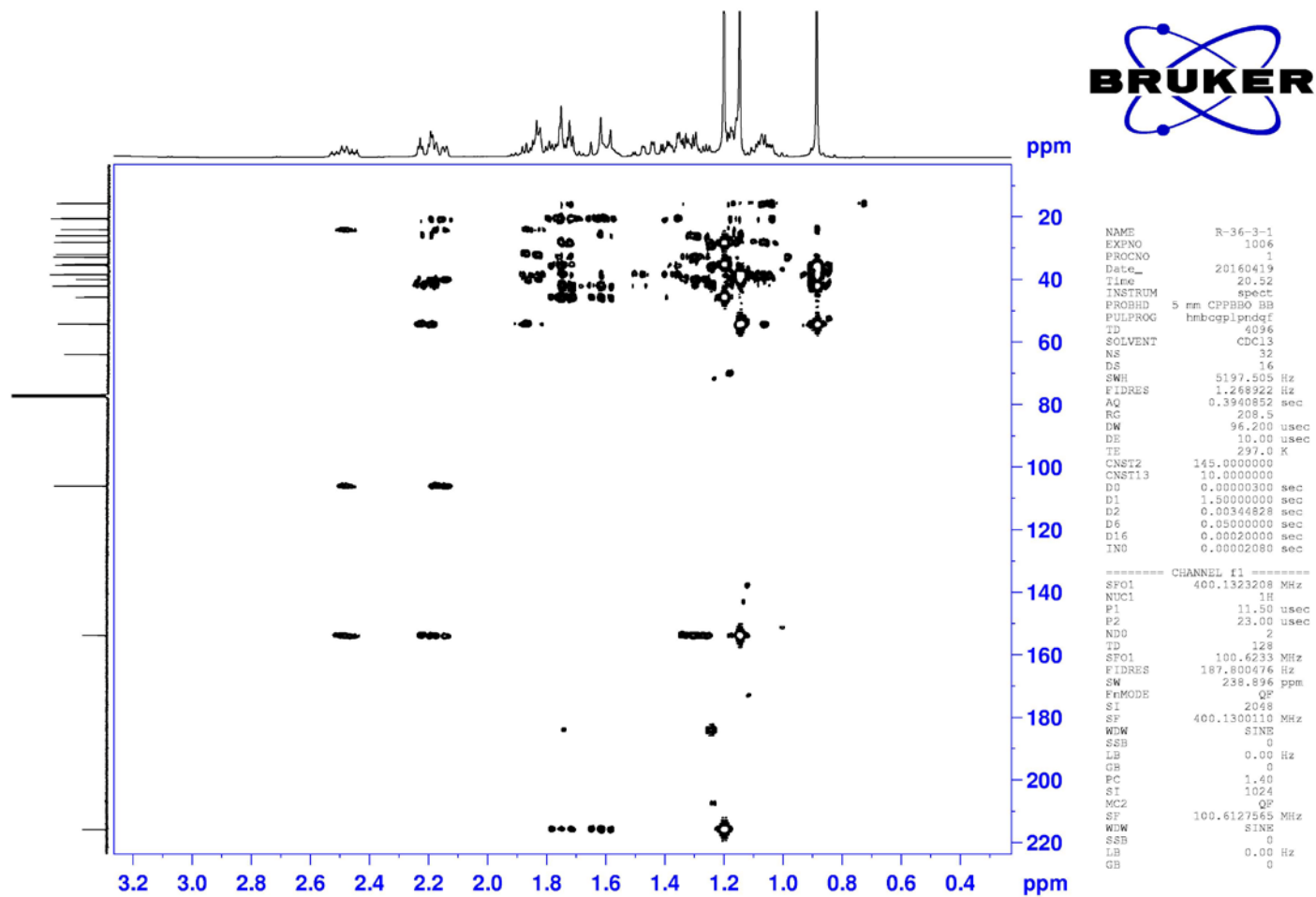


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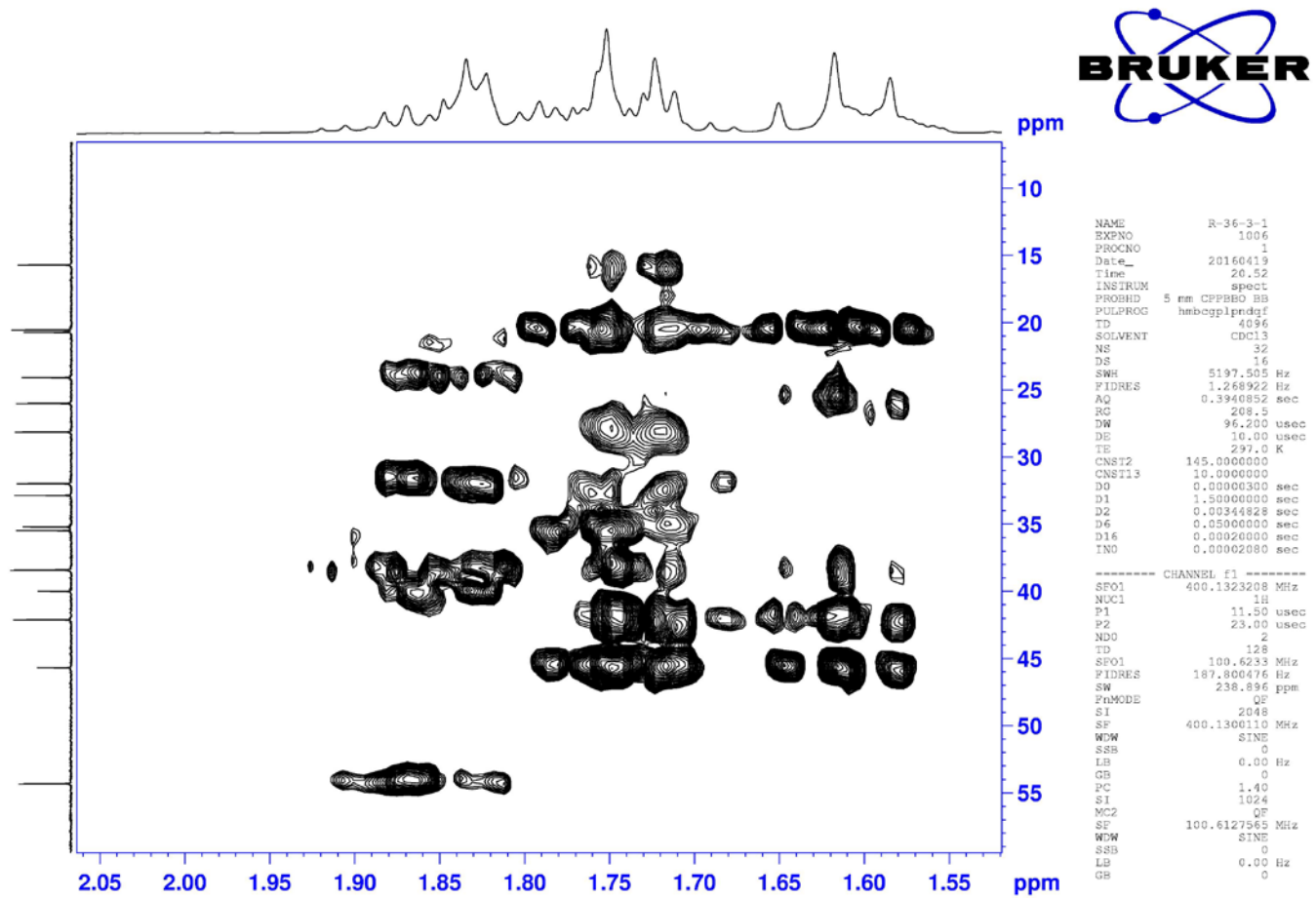
NAME          R-36-3-1
EXPNO         1006
PROCNO        1
Date_         20160419
Time         20.52
INSTRUM       spect
PROBHD        5 mm CPPBBO BB
PULPROG       hmbcggplndqf
TD            4096
SOLVENT       CDCl3
NS            32
DS            16
SWH           5197.505 Hz
FIDRES        1.268922 Hz
AQ            0.3940852 sec
RG            208.5
RW           96.200 usec
DE            10.00 usec
TE            297.0 K
CNS12         145.0000000
CNS13         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            11.50 usec
P2            23.00 usec
ND0           2
TD            128
SF01          100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
P1MODE        QF
S1            2048
SF            400.1300110 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
S1            1024
MC2           QF
SF            100.6127565 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

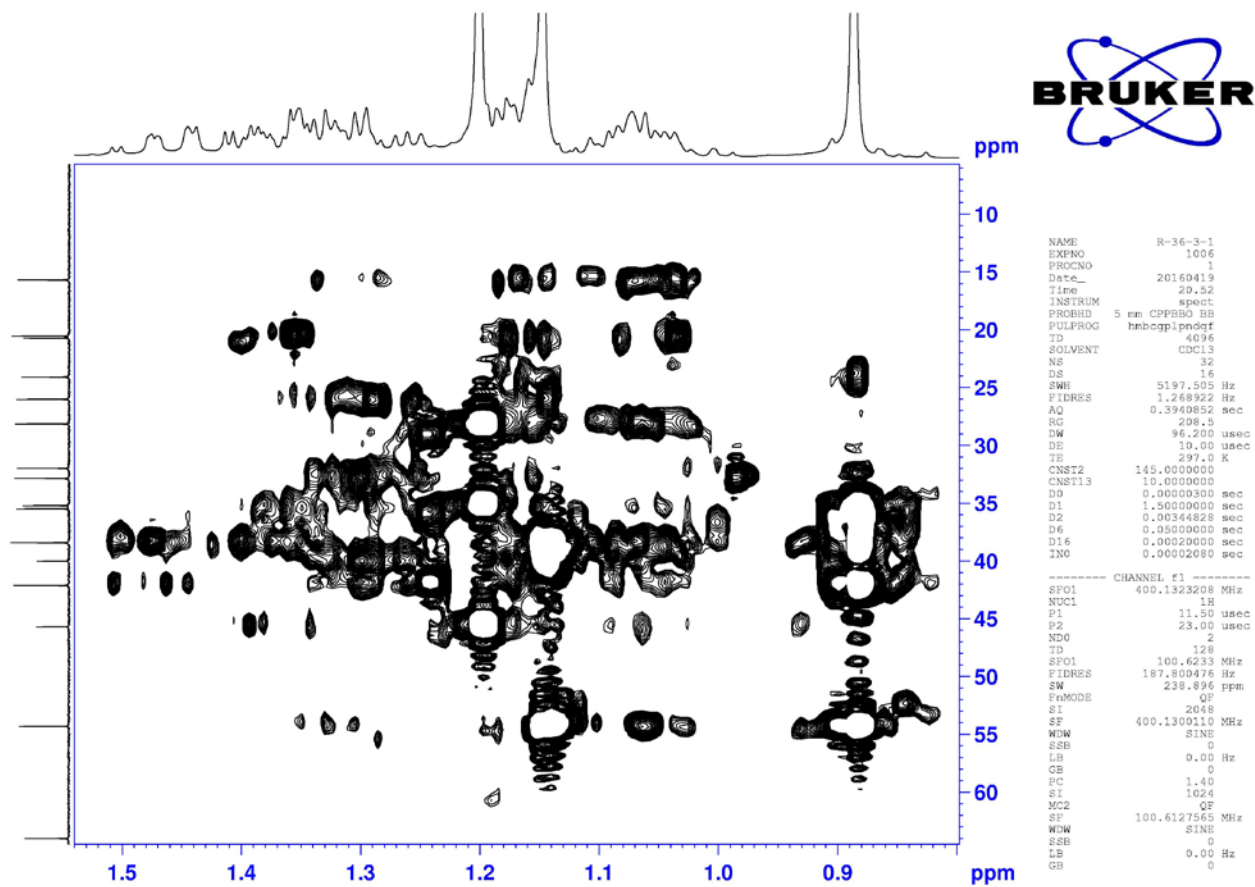
HMBC (400 MHz) spectrum of compound 2 in CDCl₃



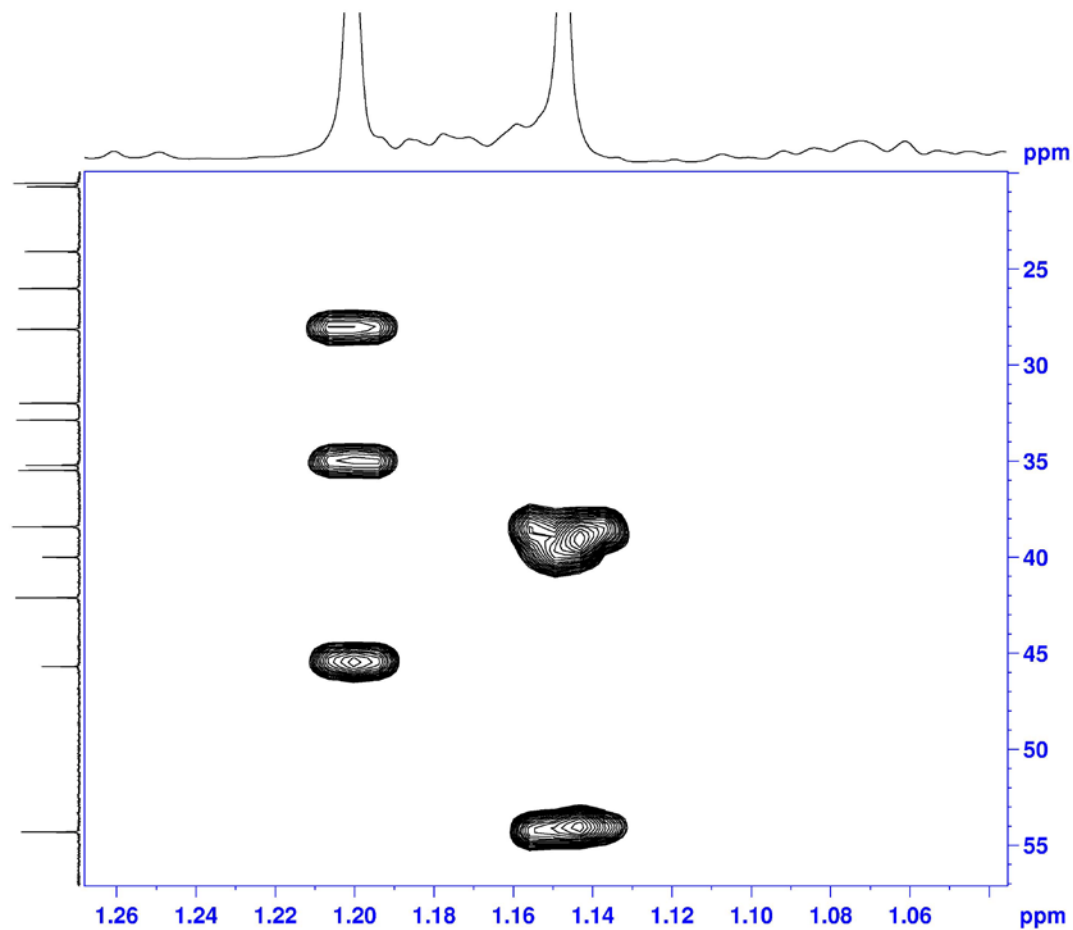
HMBC (400 MHz) spectrum of compound 2 in CDCl₃



HMBC (400 MHz) spectrum of compound 2 in CDCl₃



HMBC (400 MHz) spectrum of compound 2 in CDCl₃

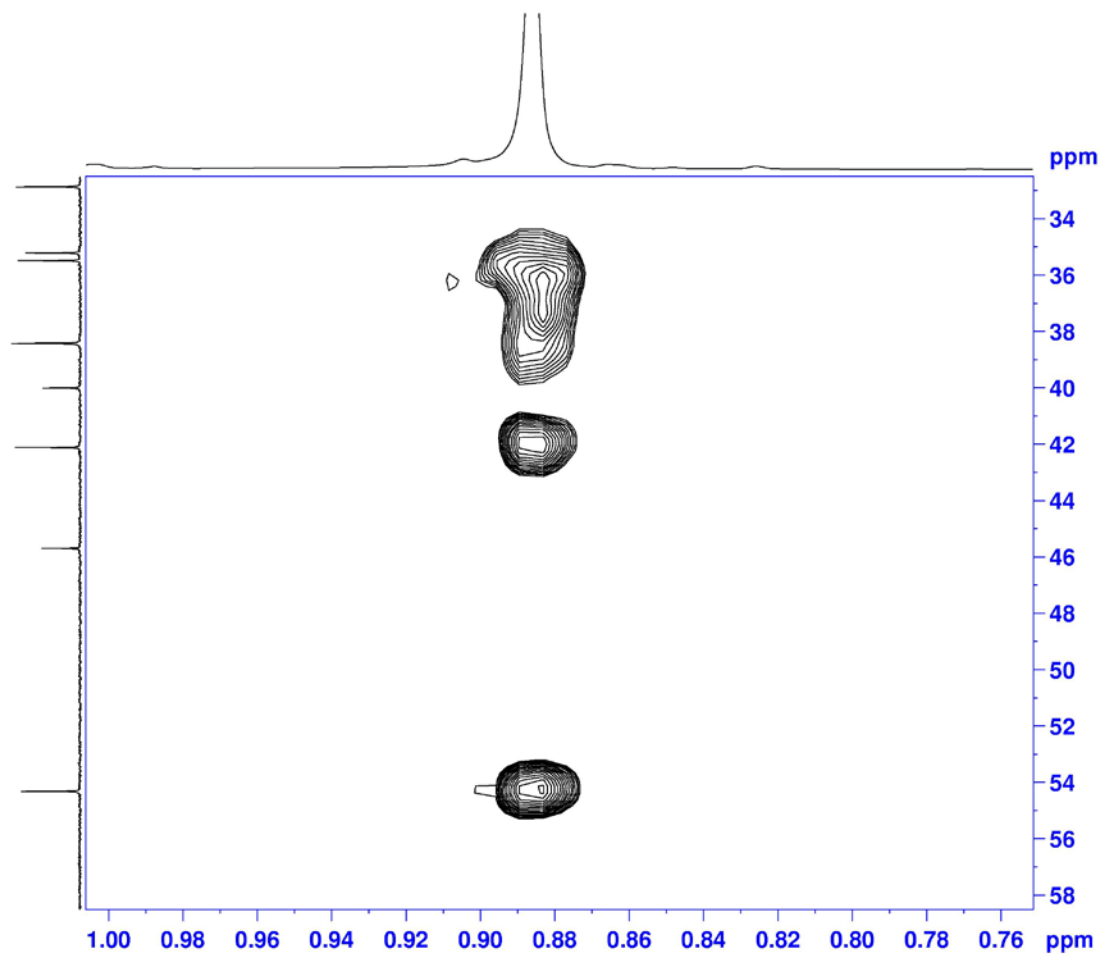


```

NAME R-36-3-1
EXPNO 1006
PROCNO 1
Date_ 20160419
Time 20.52
INSTRUM spect
PROBHD 5 mm CPMAS BB
PULPROG hmbcpg1pndd1
TD 4096
SOLVENT CDCl3
NS 32
DS 16
SWH 5197.505 Hz
FIDRES 1.268922 Hz
AQ 0.3940852 sec
RG 208.5
DM 96.200 usec
DE 10.00 usec
TE 297.0 K
CNS12 145.000000
CNS13 10.000000
DO 0.0000300 sec
D1 1.5000000 sec
D2 0.0034828 sec
D4 0.0500000 sec
D16 0.0022000 sec
TNO 0.00002080 sec

----- CHANNEL f1 -----
SF01 400.1323208 MHz
NUC1 1H
P1 11.50 usec
P2 23.00 usec
ND0 2
TD 128
SF01 100.6233 MHz
FIDRES 187.800476 Hz
SW 238.896 ppm
PRMODE QF
SI 2048
SF 400.1300095 MHz
WDM SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 QF
SF 100.6127075 MHz
WDM SINE
SSB 0
LB 0.00 Hz
GB 0
    
```

HMBC (400 MHz) spectrum of compound 2 in CDCl₃

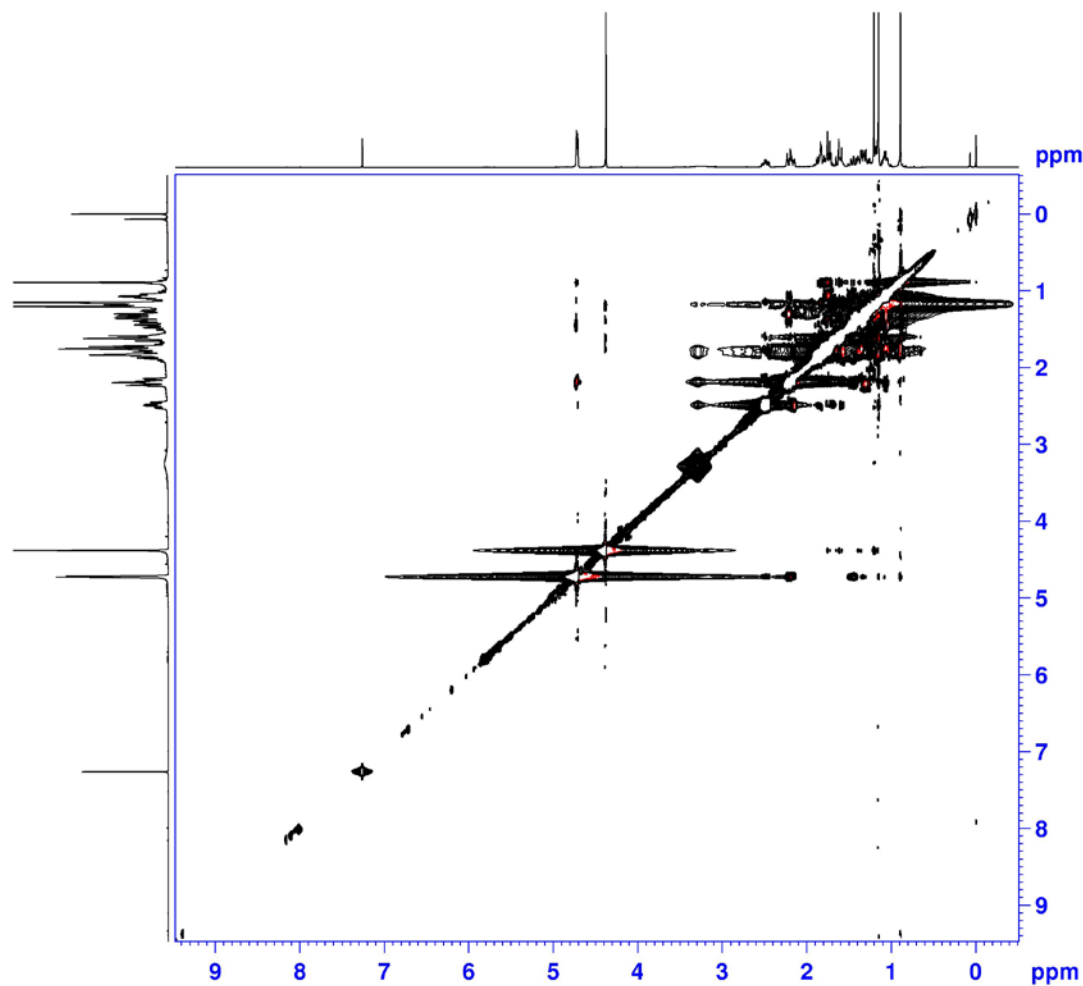


```

NAME          R-36-3-1
EXPNO         1006
PROCNO        1
Date_         20160419
Time         20.52
INSTRUM       spect
PROBHD        5 mm CPP80 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         145.000000
CNST13        10.000000
DO            0.0000000 sec
D1            1.5000000 sec
D2            0.0034828 sec
D6            0.0500000 sec
D16           0.0002000 sec
IN0           0.0000000 sec

----- CHANNEL f1 -----
SFO1          400.1323208 MHz
NUC1          1H
P1            11.50 usec
P2            23.00 usec
NDC           2
TD            128
SFO1          100.6233 MHz
FIDRES        187.800476 Hz
SW            238.896 ppm
FHM00E        GF
SI            2048
SF            400.1300095 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           GF
SF            100.6127575 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

NOESY (400 MHz) spectrum of compound 2 in CDCl₃



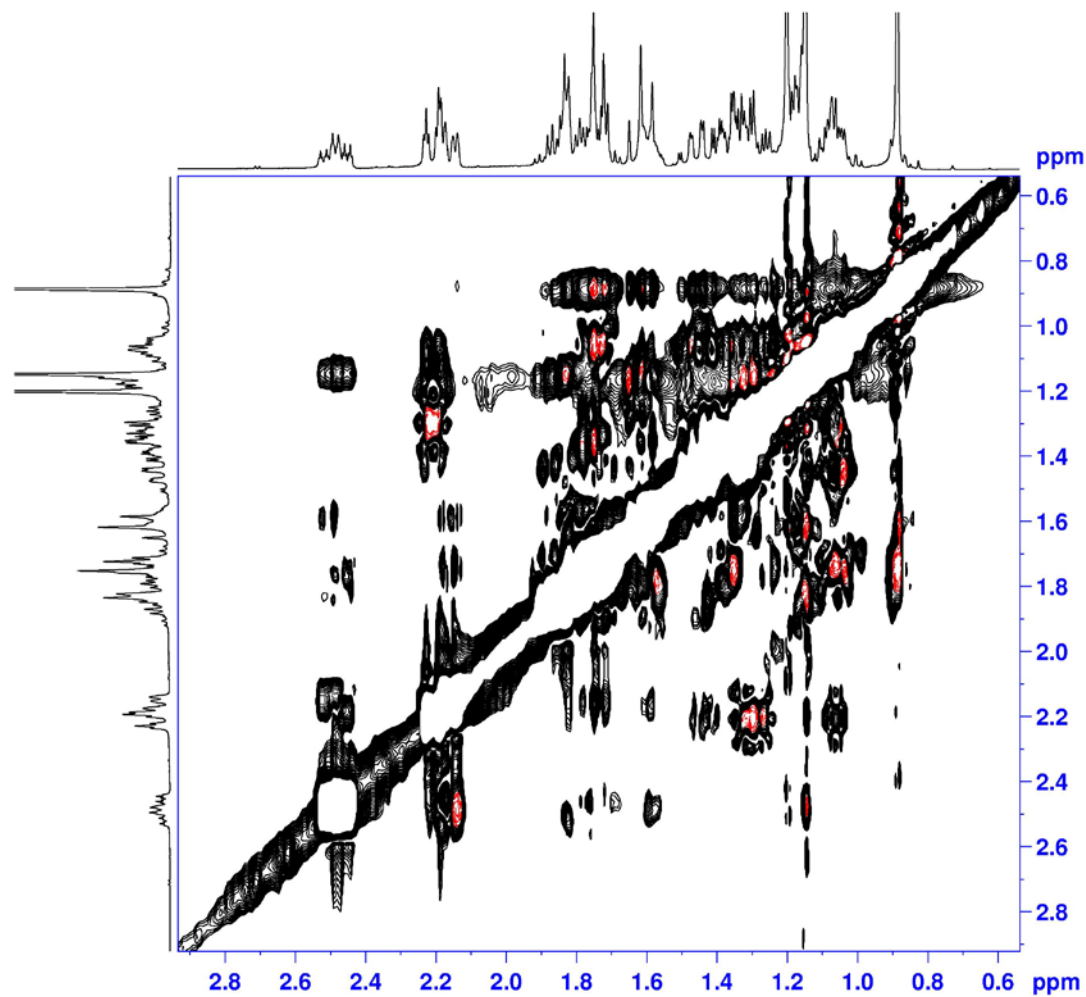
```

NAME          R-36-3-1
EXPNO         107
PROCNO        1
Date_         20160419
Time         23.07
INSTRUM       spect
PROBHD        5 mm CPMAS BB
PULPROG       noesypphpg
TD            2048
SOLVENT       CDCl3
NS            16
DS            32
SWH           4000.000 Hz
FIDRES        1.953125 Hz
AQ            0.2560500 sec
RG            91.64
AW           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
FwMODE        States-TPPI
SI            1024
SF            400.1300098 MHz
WDW           QSIINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MG2           States-TPPI
SF            400.1300098 MHz
WDW           QSIINE
SSB           2
LB            0.00 Hz
GB            0
    
```

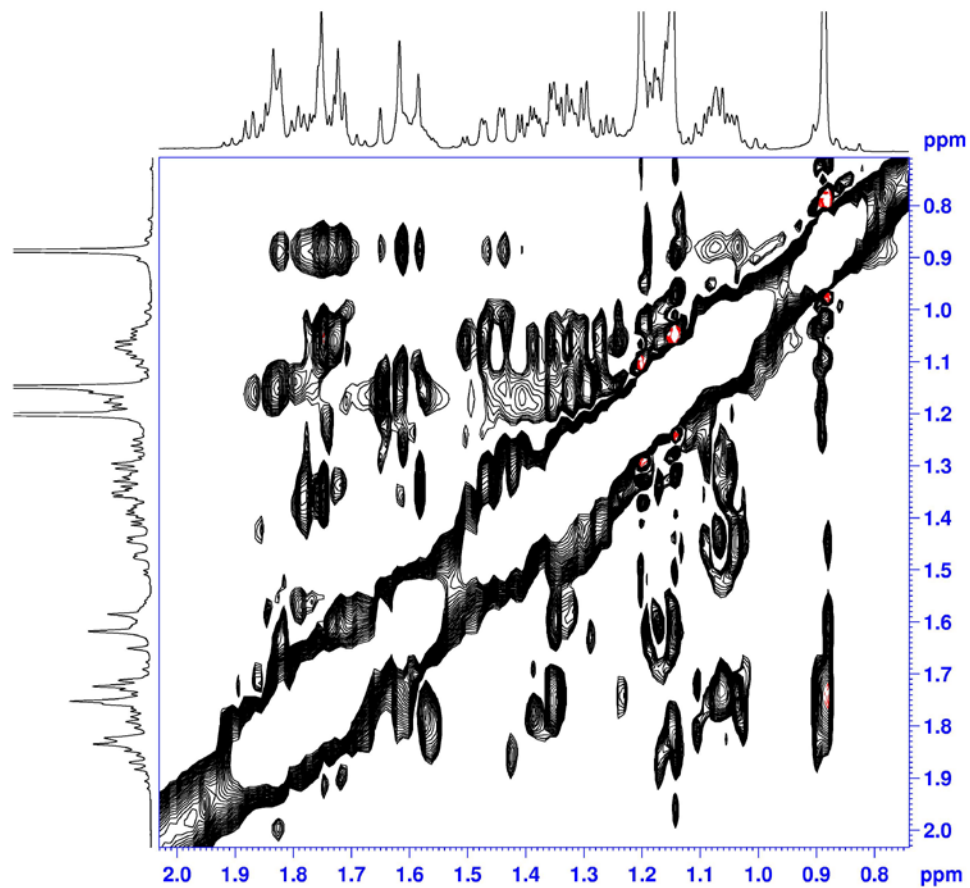
NOESY (400 MHz) spectrum of compound 2 in CDCl₃



```
NAME R-36-3-1
EXPNO 107
PROCNO 1
Date_ 20160419
Time 23.07
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 91.64
DW 125.000 usec
DE 10.00 usec
TE 297.0 K
DO 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 =====
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
FMODE States-TPP1
SI 1024
SF 400.1300098 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPP1
SF 400.1300098 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
```

NOESY (400 MHz) spectrum of compound 2 in CDCl₃



```

NAME           R-36-3-1
EXPNO          107
PROCNO         1
Date_          20160419
Time           23.07
INSTRUM        spect
PROBHD         5 mm CPMAS BB
PULPROG        noesyppp
TD             2048
SOLVENT        CDCl3
NS             16
DS             32
SWH            4000.000 Hz
FIDRES         1.953125 Hz
AQ             0.2560500 sec
RG             91.64
FW            125.000 usec
DE             10.00 usec
TE             297.0 K
D0             0.00011036 sec
D1             1.99385595 sec
D8             0.10000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00025000 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
RG             1
TD             256
SF01           400.1318 MHz
FIDRES         15.625000 Hz
SW             9.997 ppm
FqMODE         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
    
```