

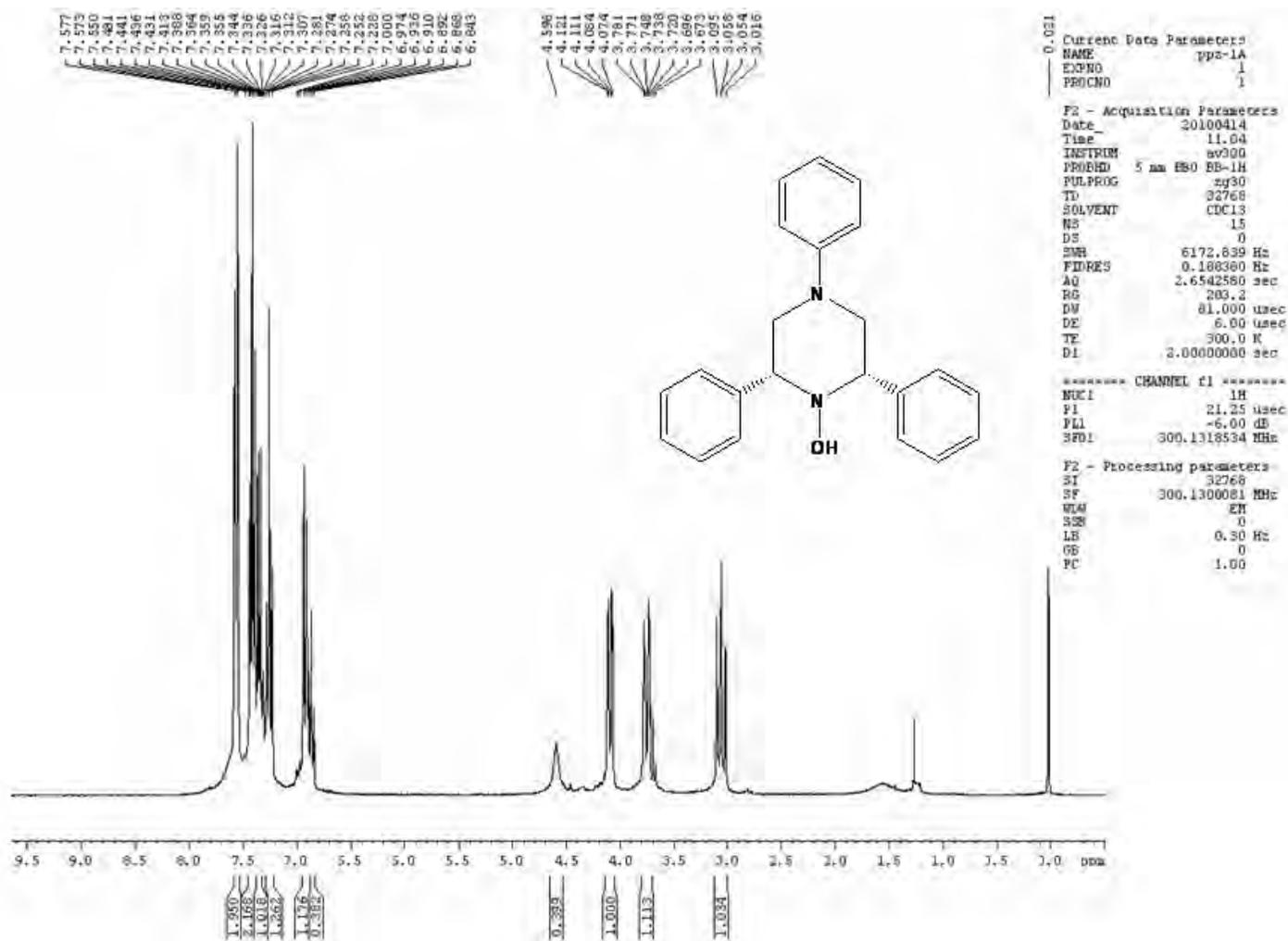
## Supporting Information

### **Synthesis of novel *N*-hydroxy heterocycles *via* intramolecular reductive cyclization of diketoximes by NaBH<sub>3</sub>CN**

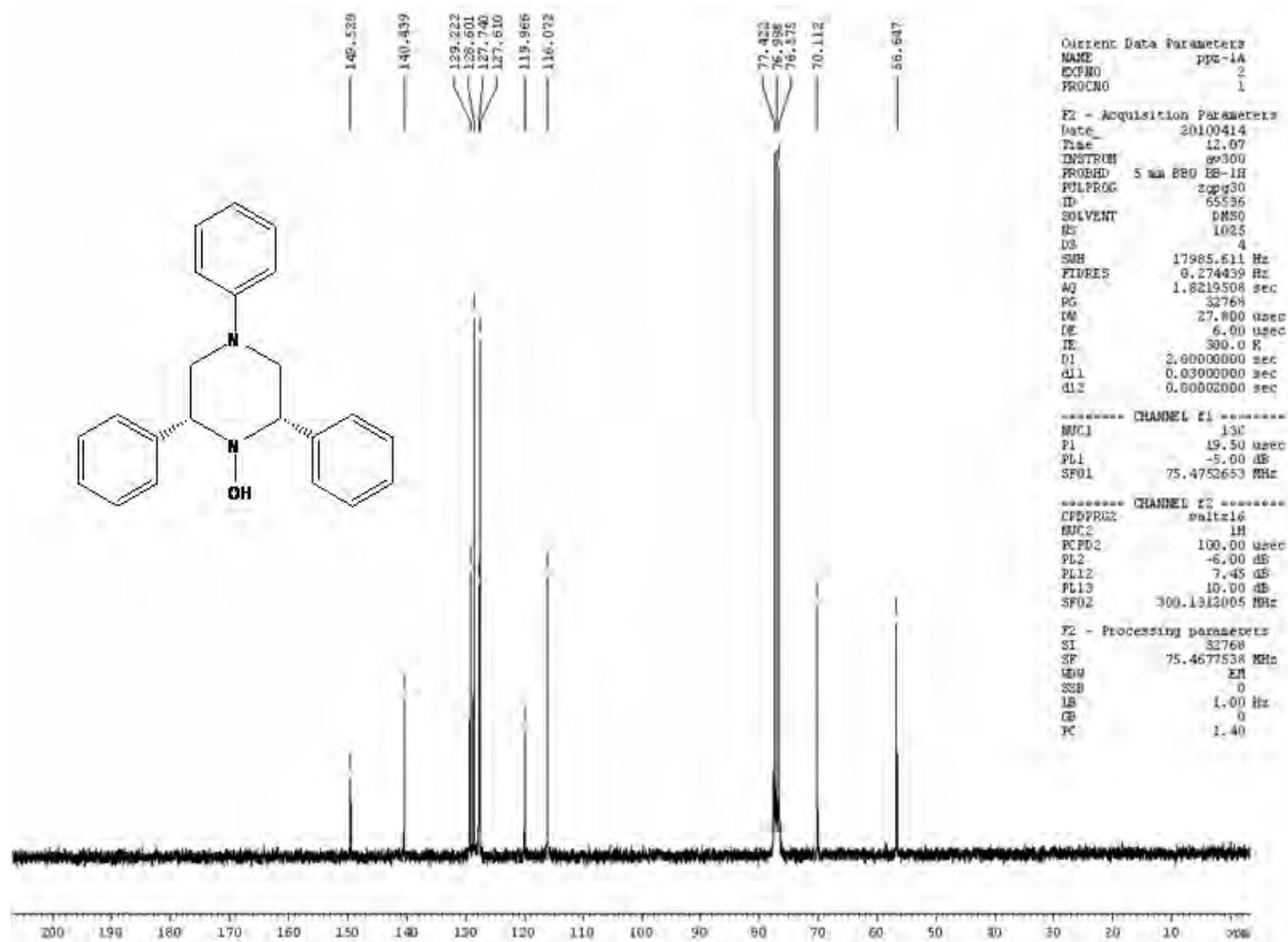
**Muthupandi Nagaraj, Muthusamy Boominathan, Shanmugam Muthusubramanian\*<sup>[a]</sup> and Natamai Bhuvanesh<sup>[b]</sup>**

	<b>Page(s)</b>
Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra .....	2-104

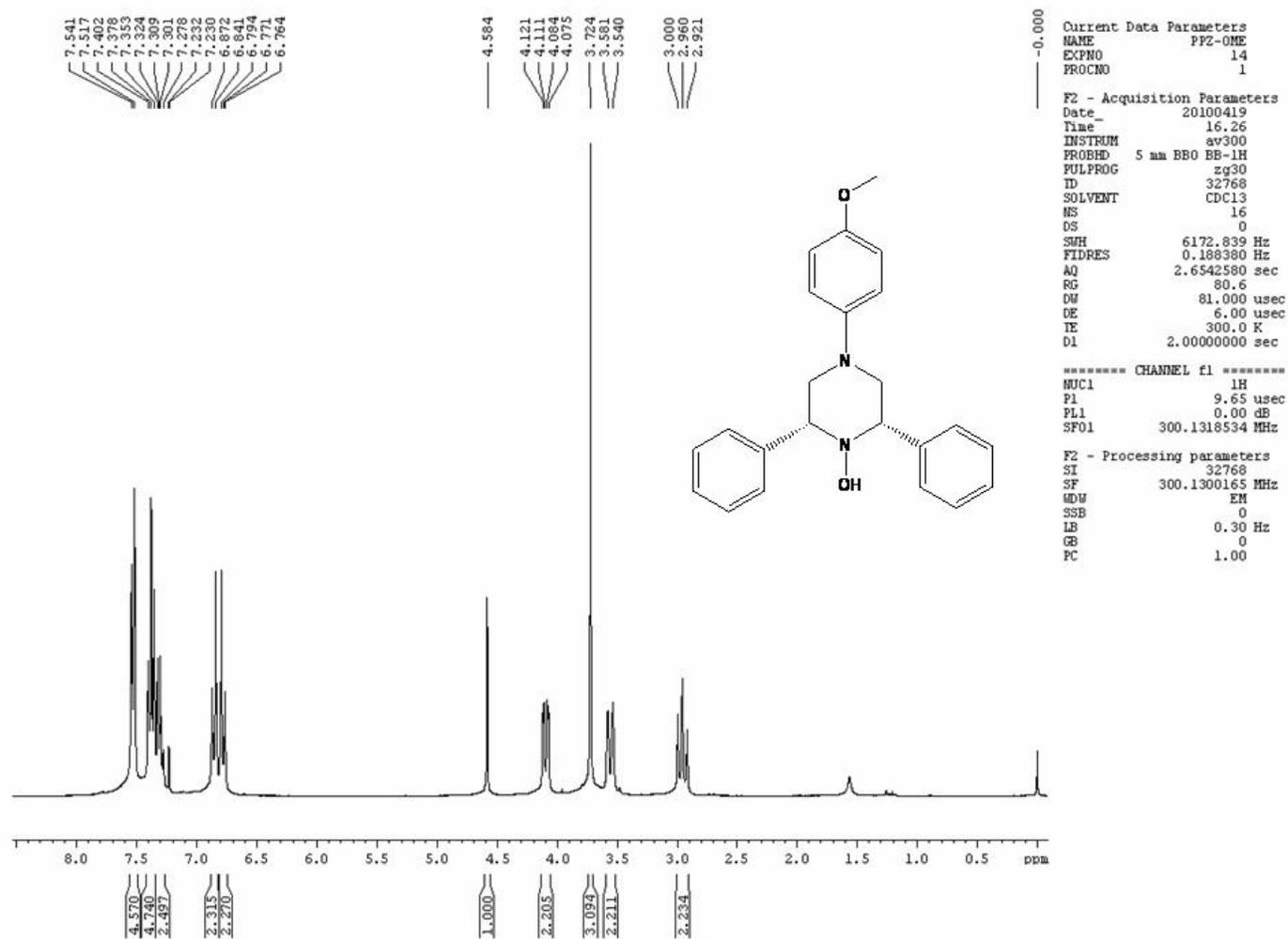
$^1\text{H}$  NMR spectrum of **3a** (300 MHz,  $\text{CDCl}_3$ )



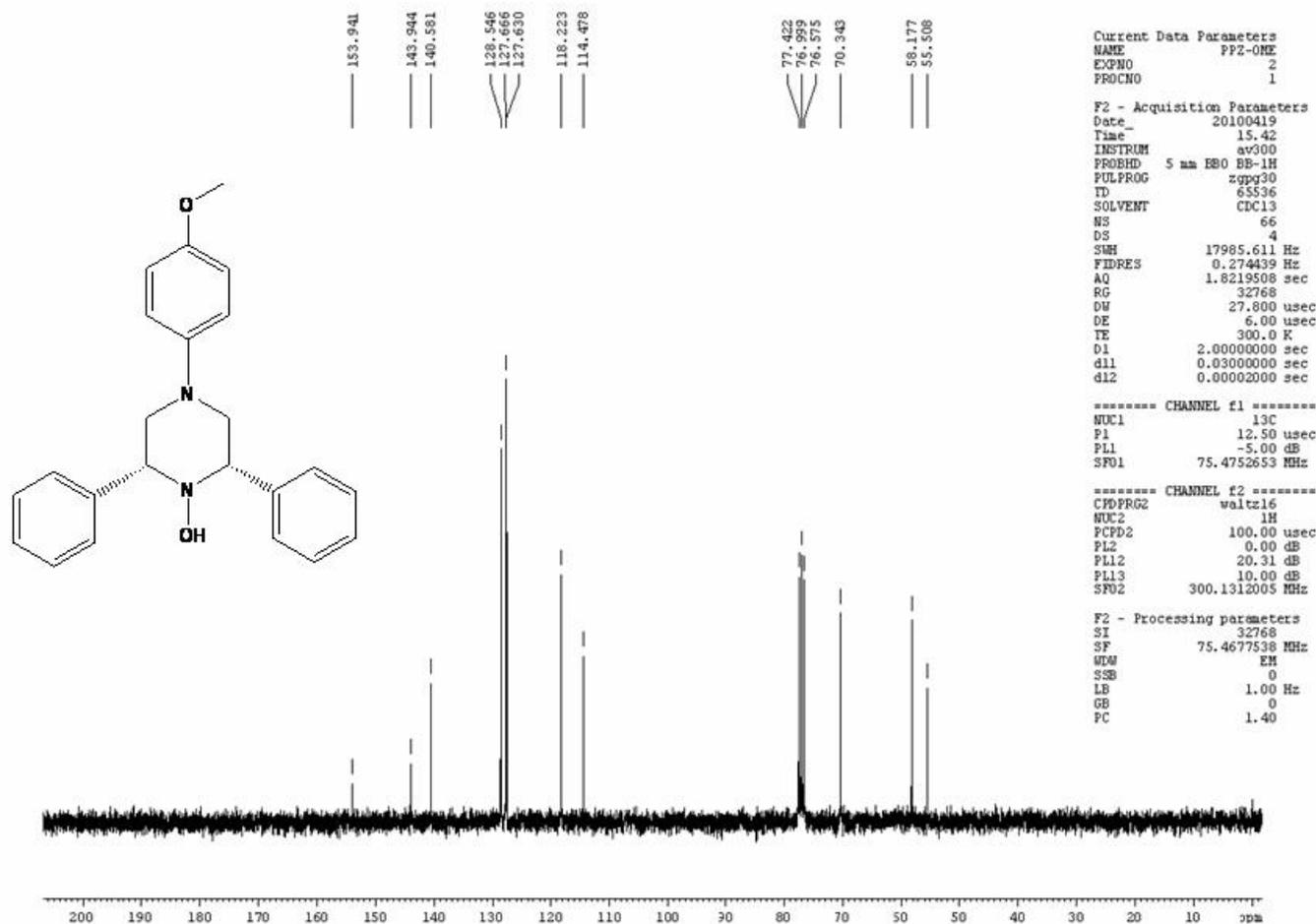
<sup>13</sup>C NMR spectrum of **3a** (75 MHz, CDCl<sub>3</sub>)



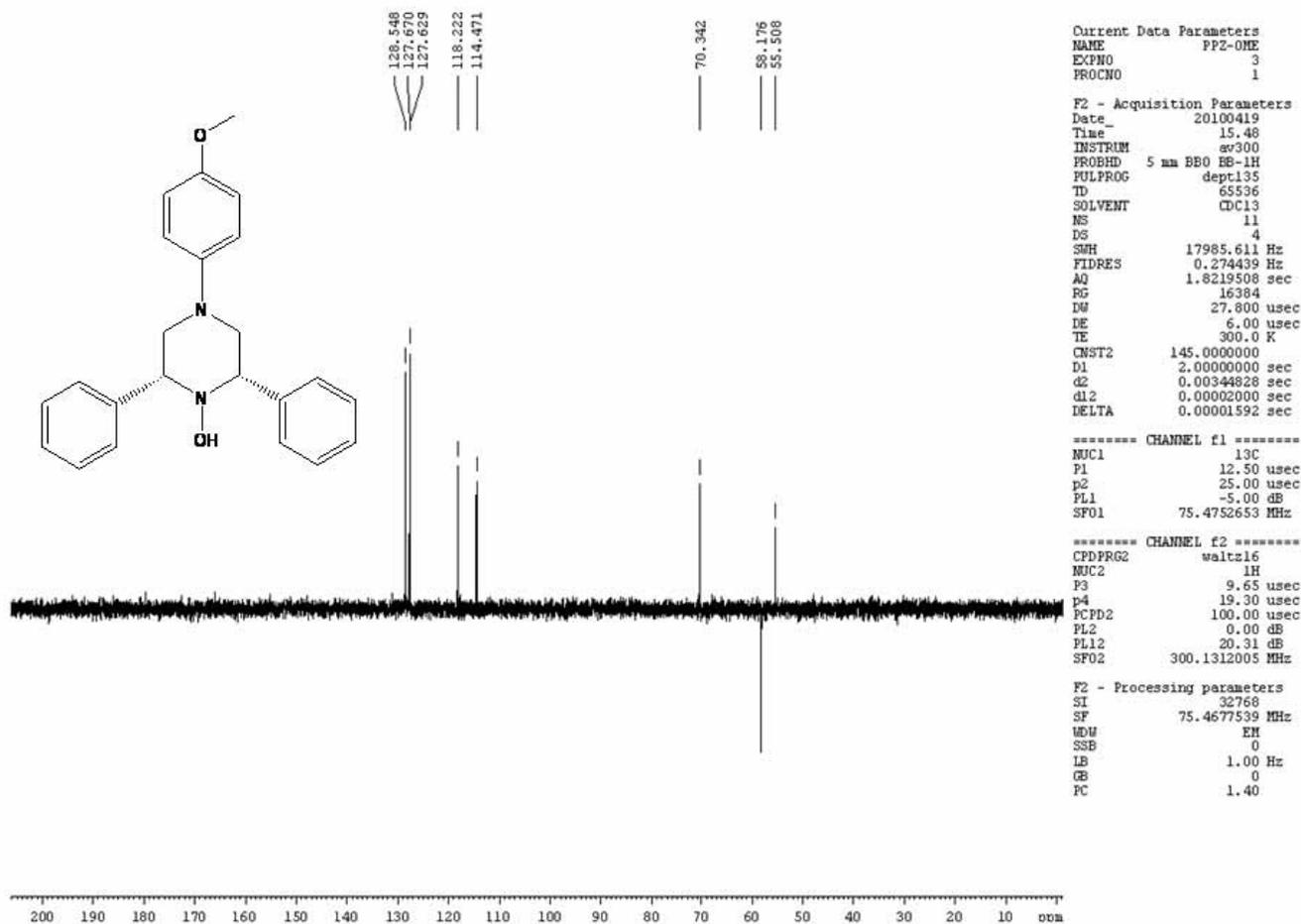
<sup>1</sup>H NMR spectrum of **3b** (300 MHz, CDCl<sub>3</sub>)



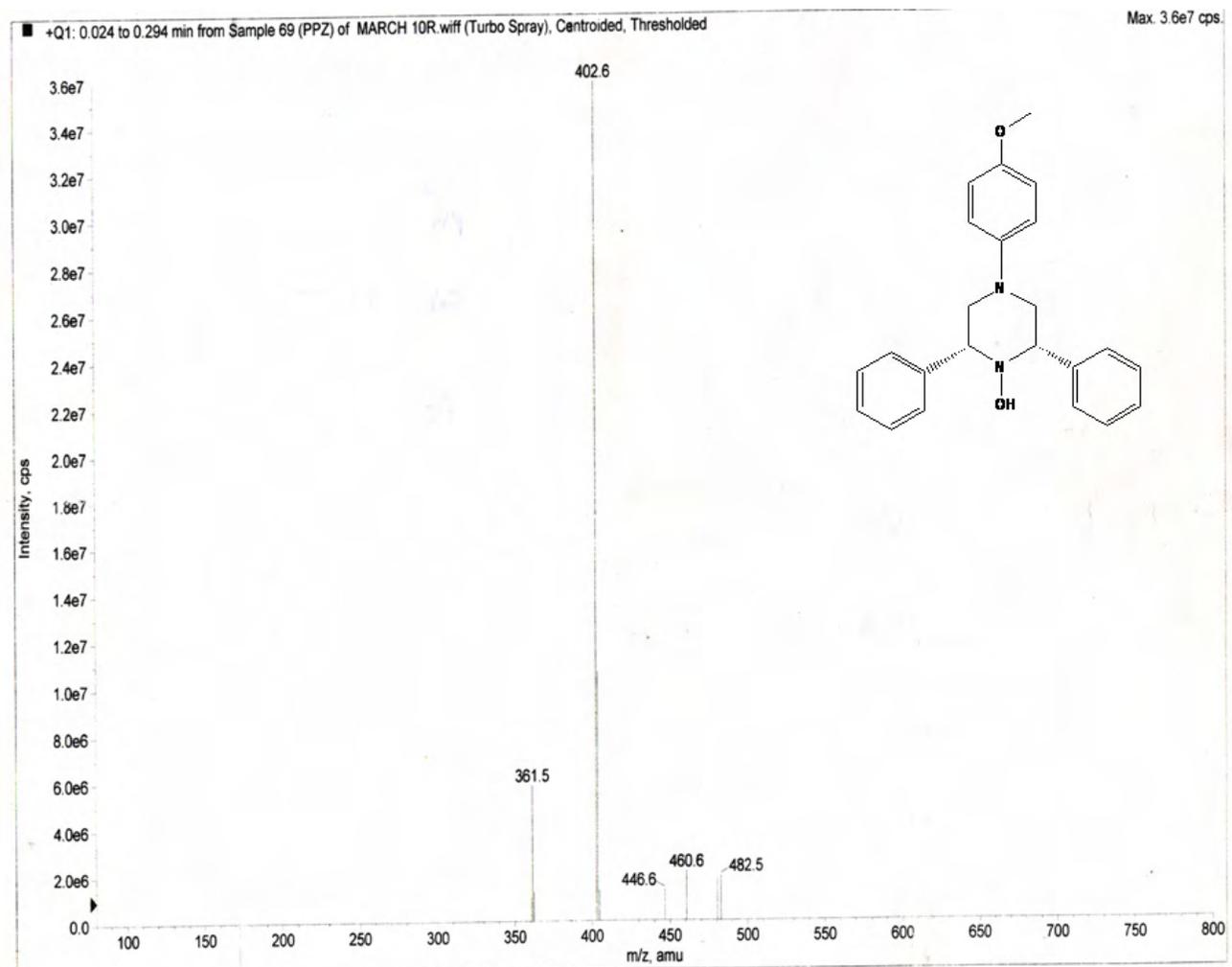
<sup>13</sup>C NMR spectrum of **3b** (75 MHz, CDCl<sub>3</sub>)



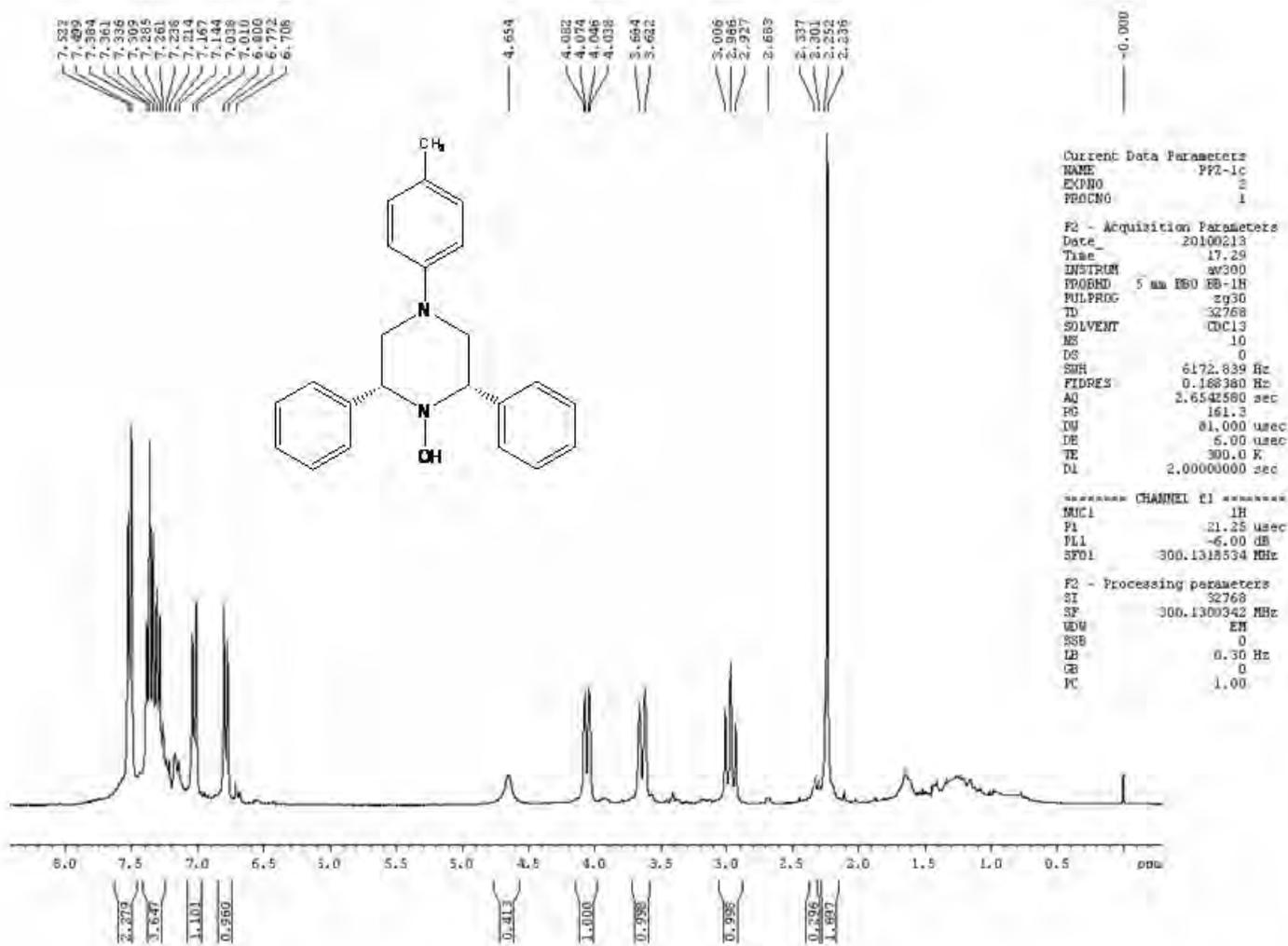
DEPT-135 spectrum of **3b**



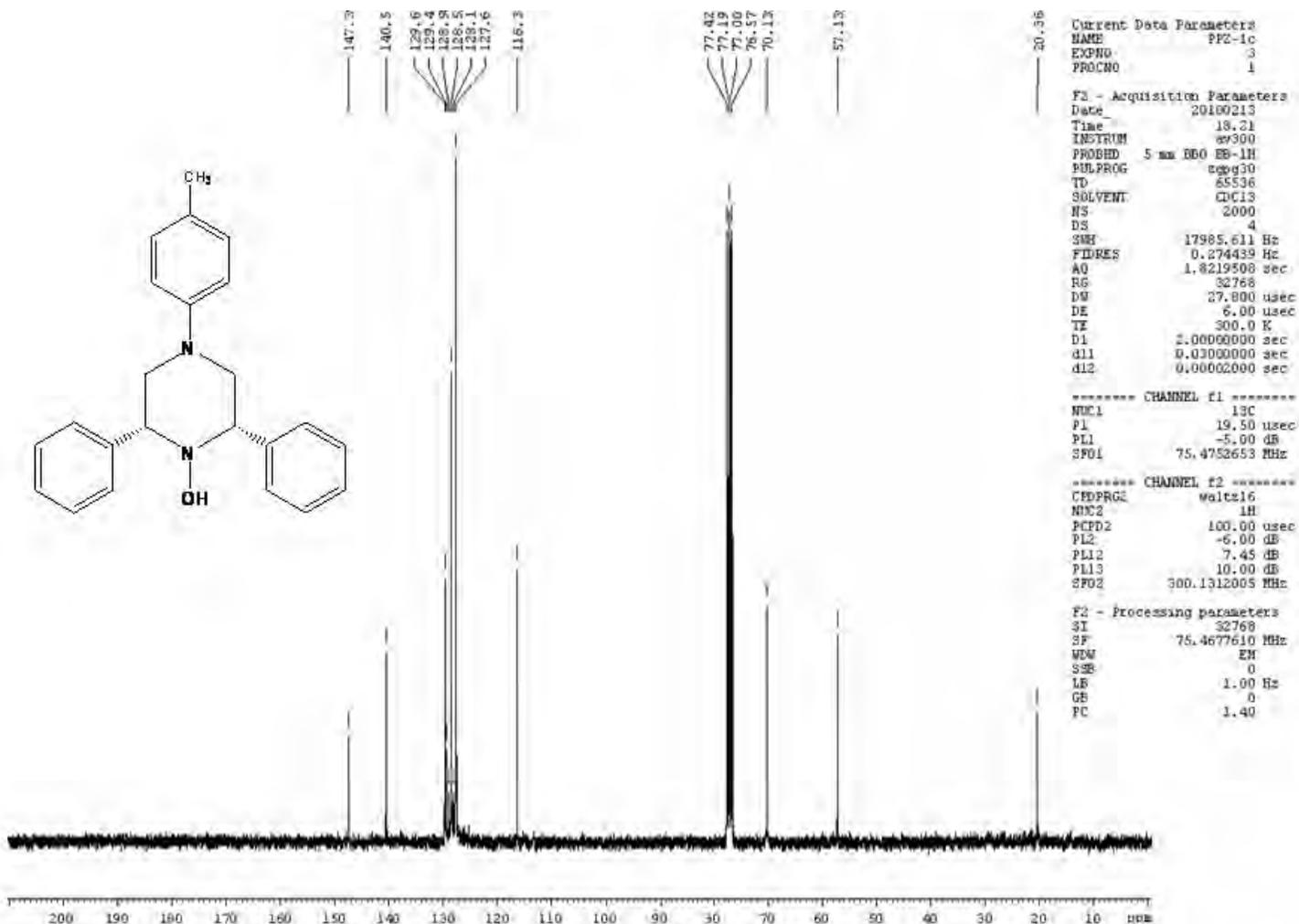
Mass spectrum of **3b**



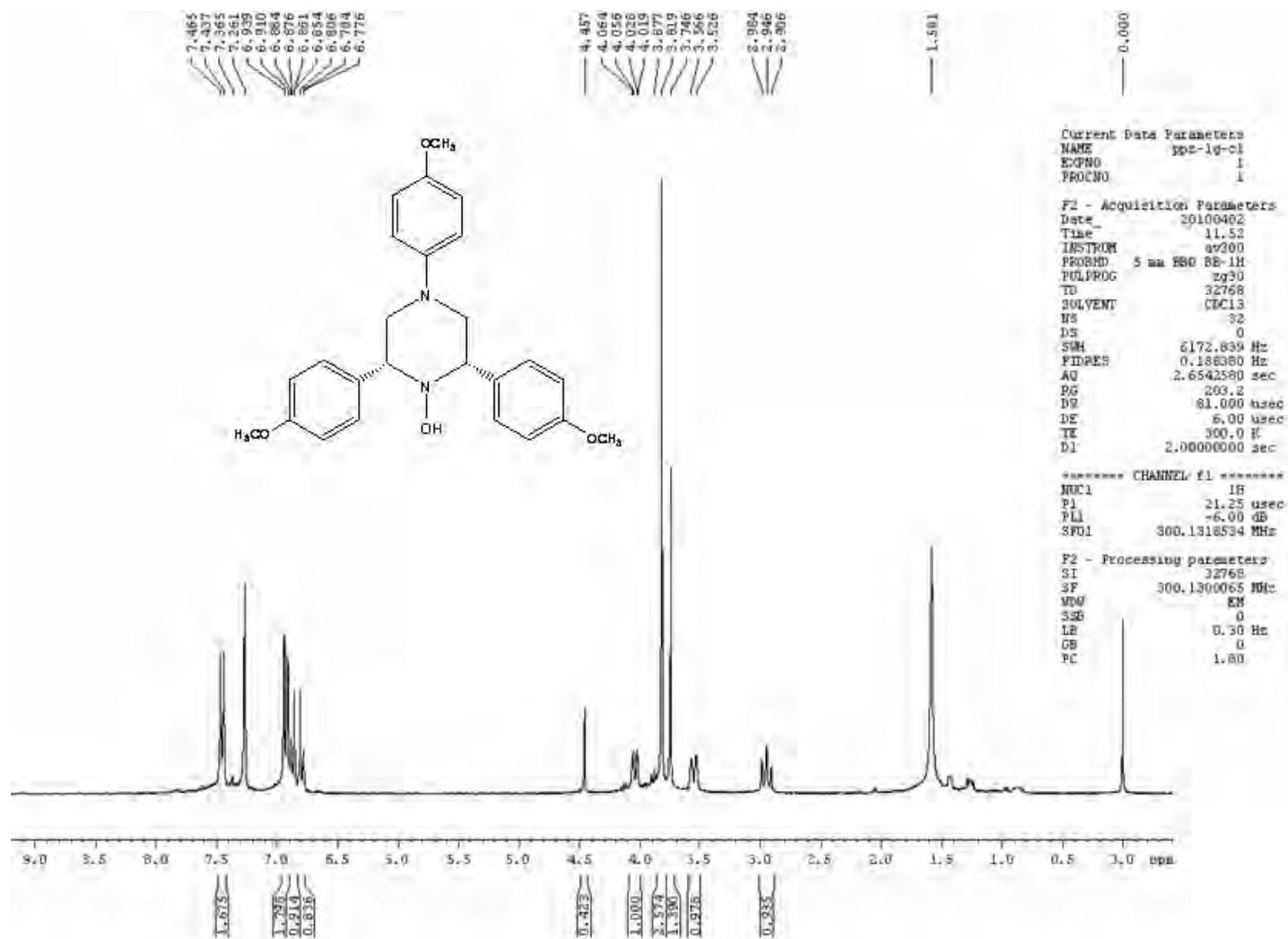
<sup>1</sup>H NMR spectrum of **3c** (300 MHz, CDCl<sub>3</sub>)



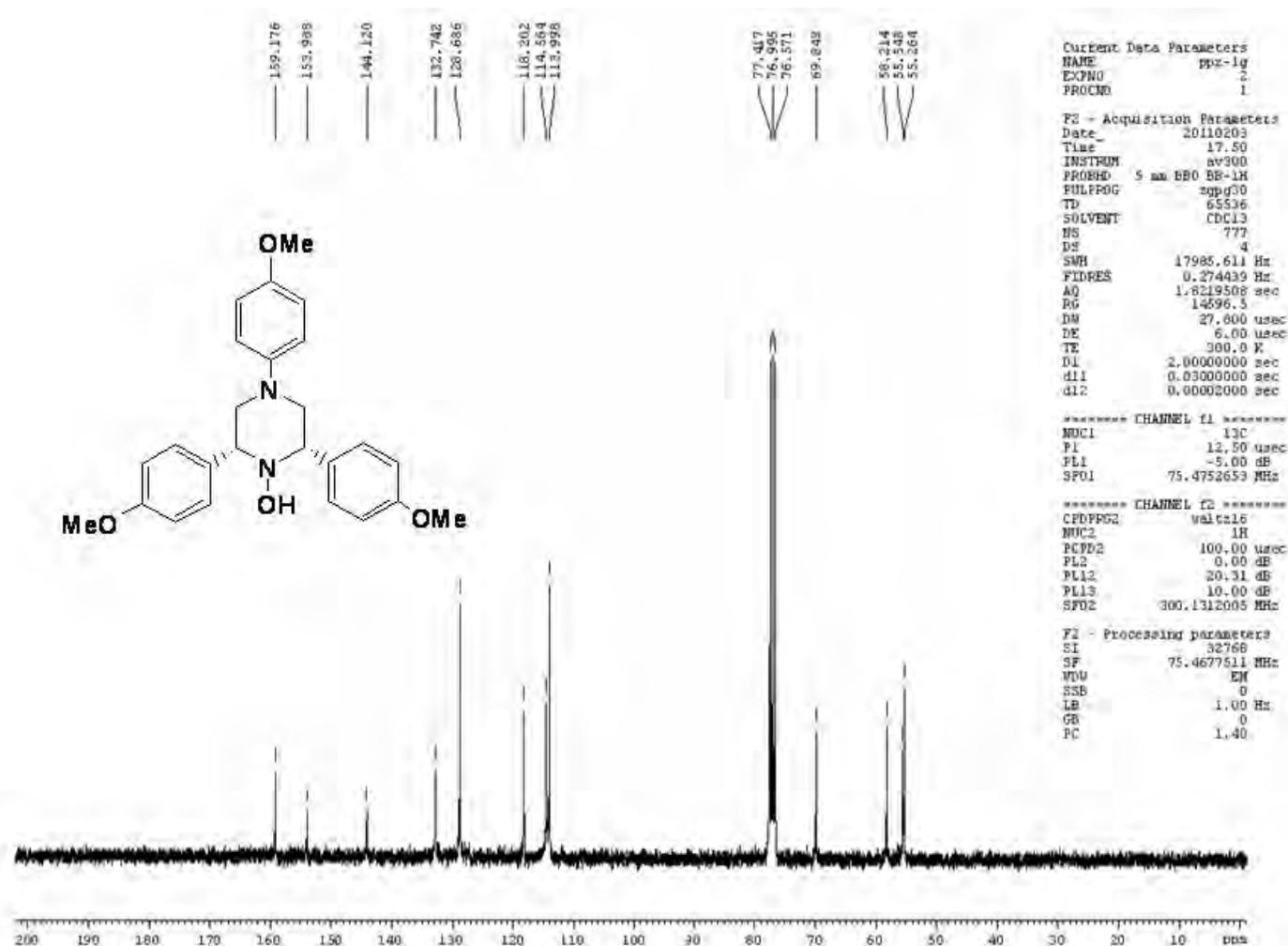
$^{13}\text{C}$  NMR spectrum of **3c** (75 MHz,  $\text{CDCl}_3$ )



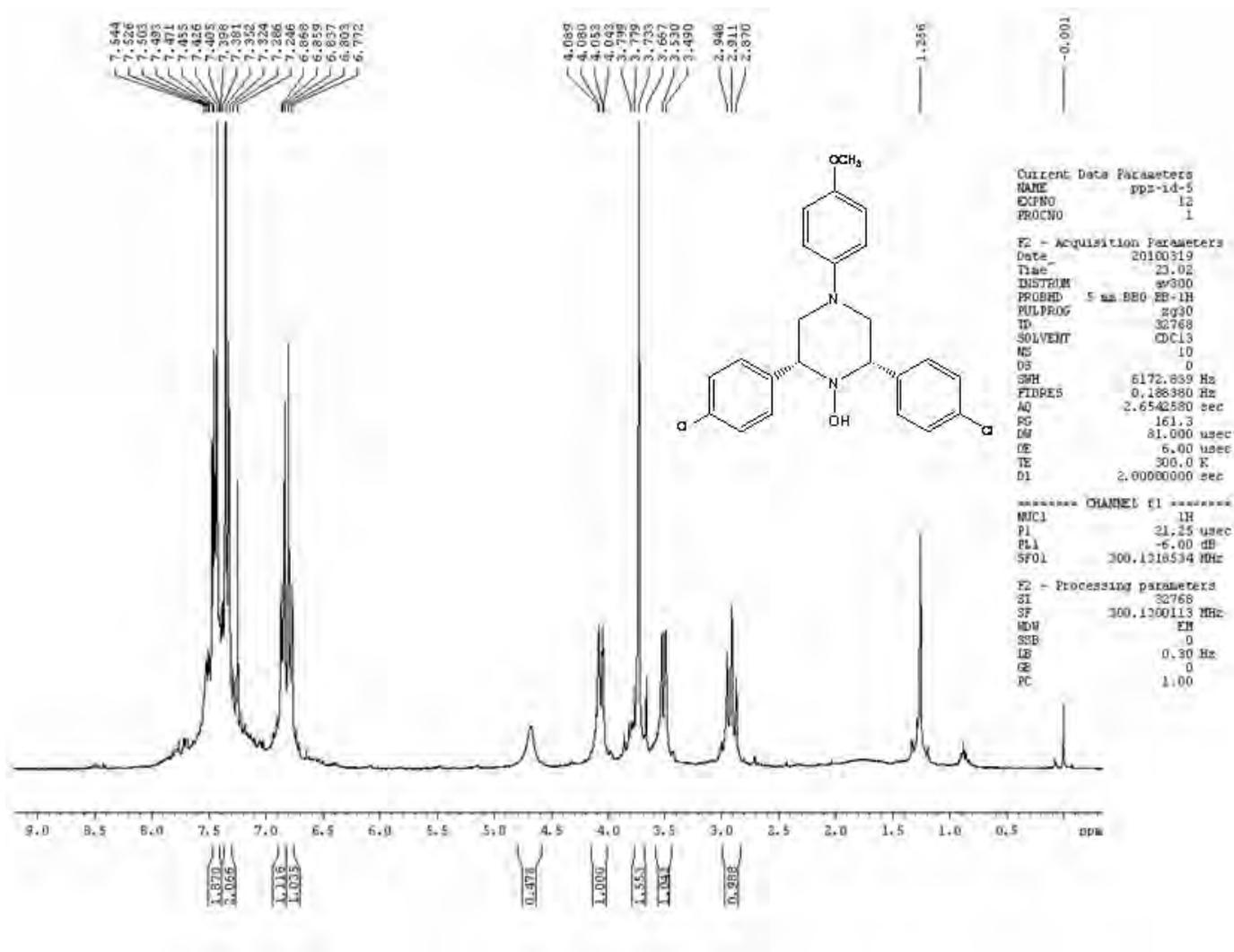
<sup>1</sup>H NMR spectrum of **3d** (300 MHz, CDCl<sub>3</sub>)



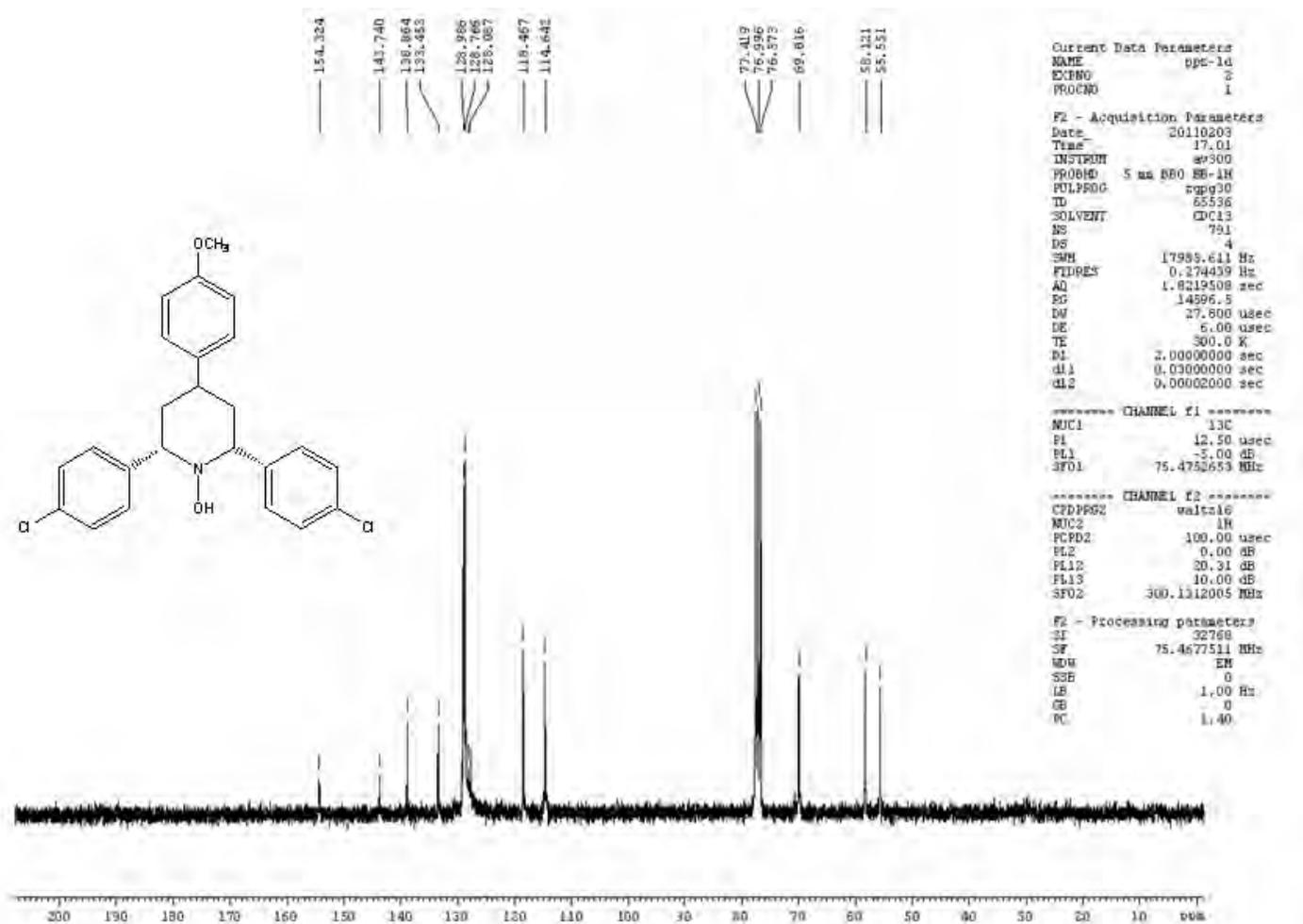
$^{13}\text{C}$  NMR spectrum of **3d** (75 MHz,  $\text{CDCl}_3$ )



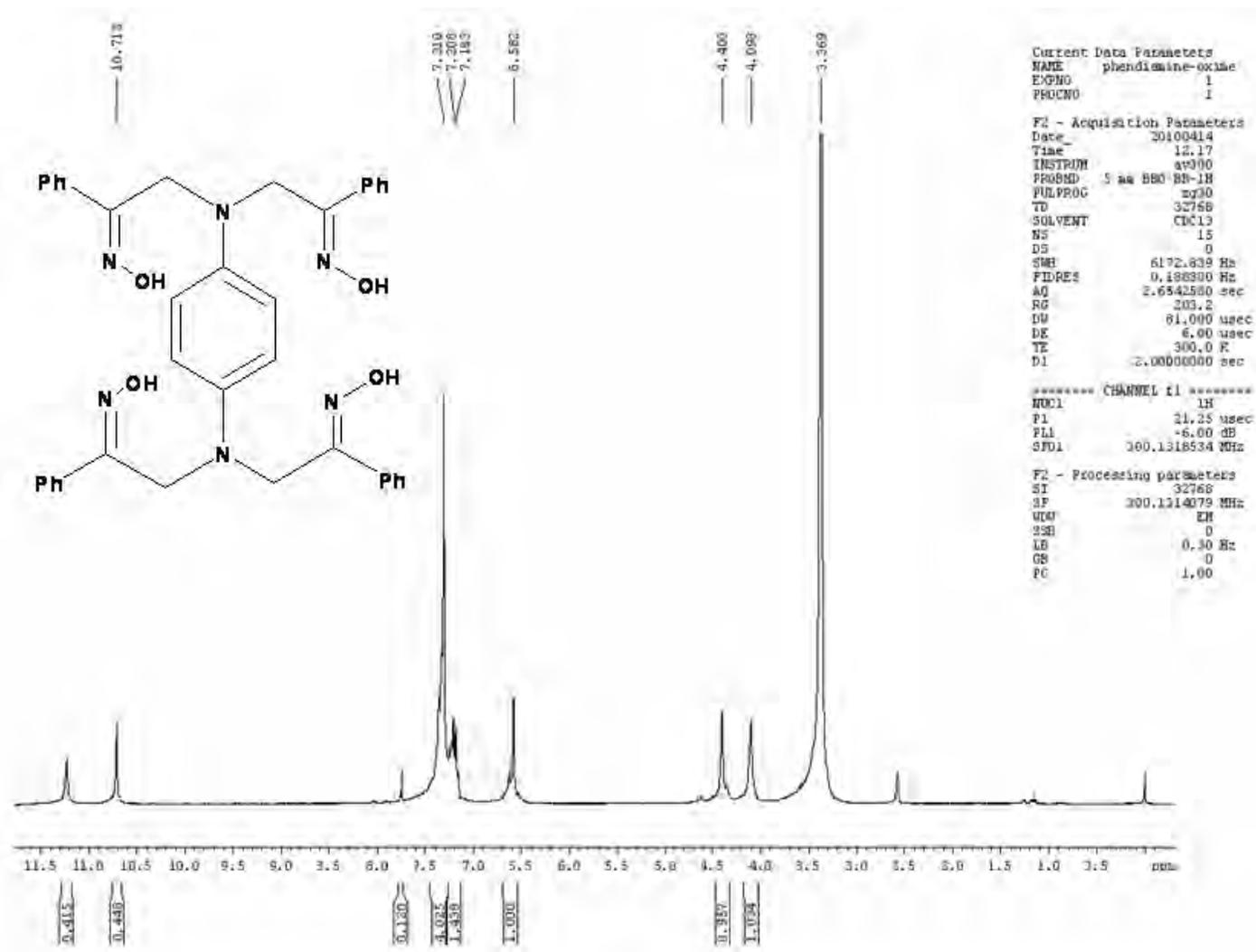
<sup>1</sup>H NMR spectrum of **3e** (300 MHz, CDCl<sub>3</sub>)



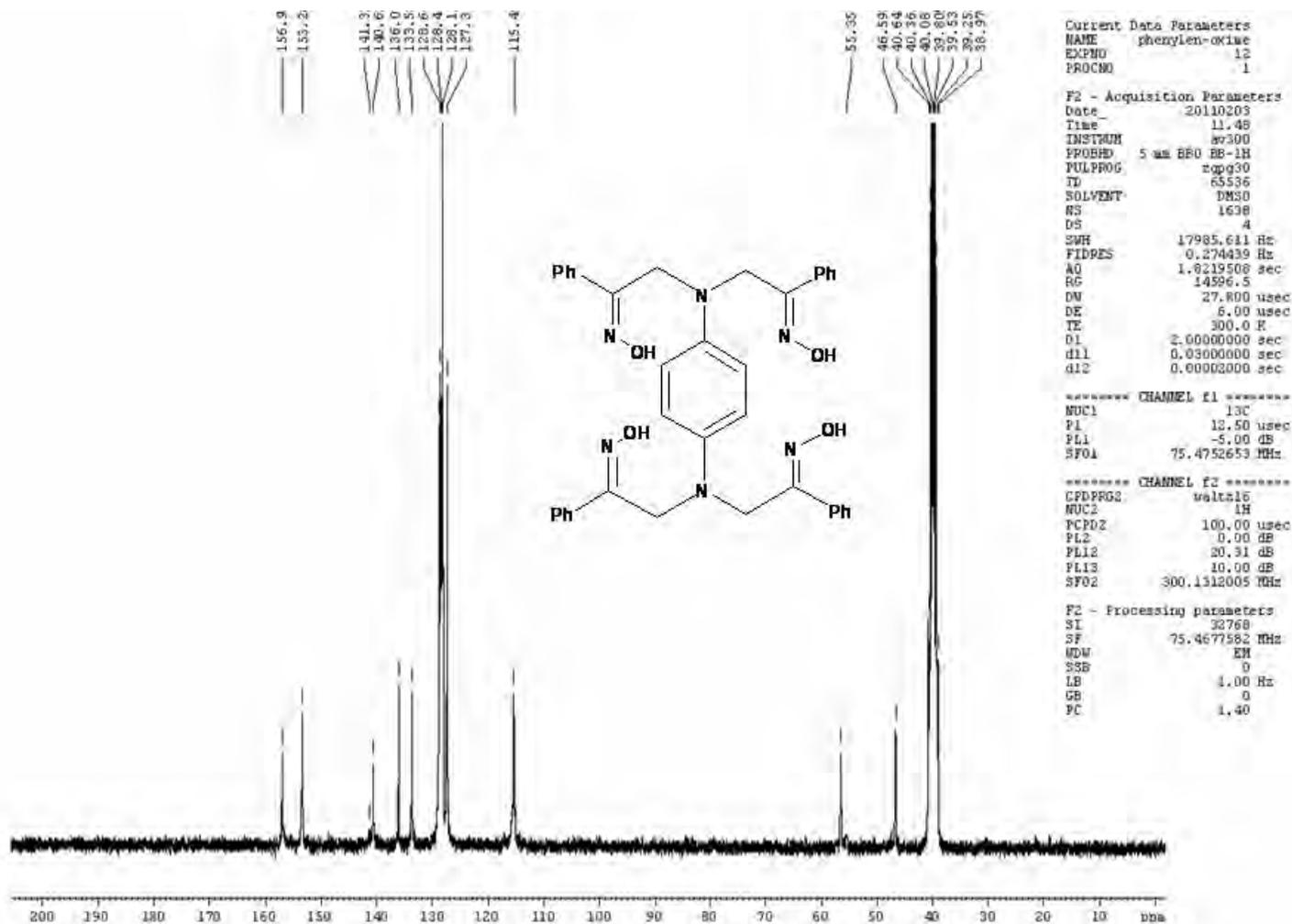
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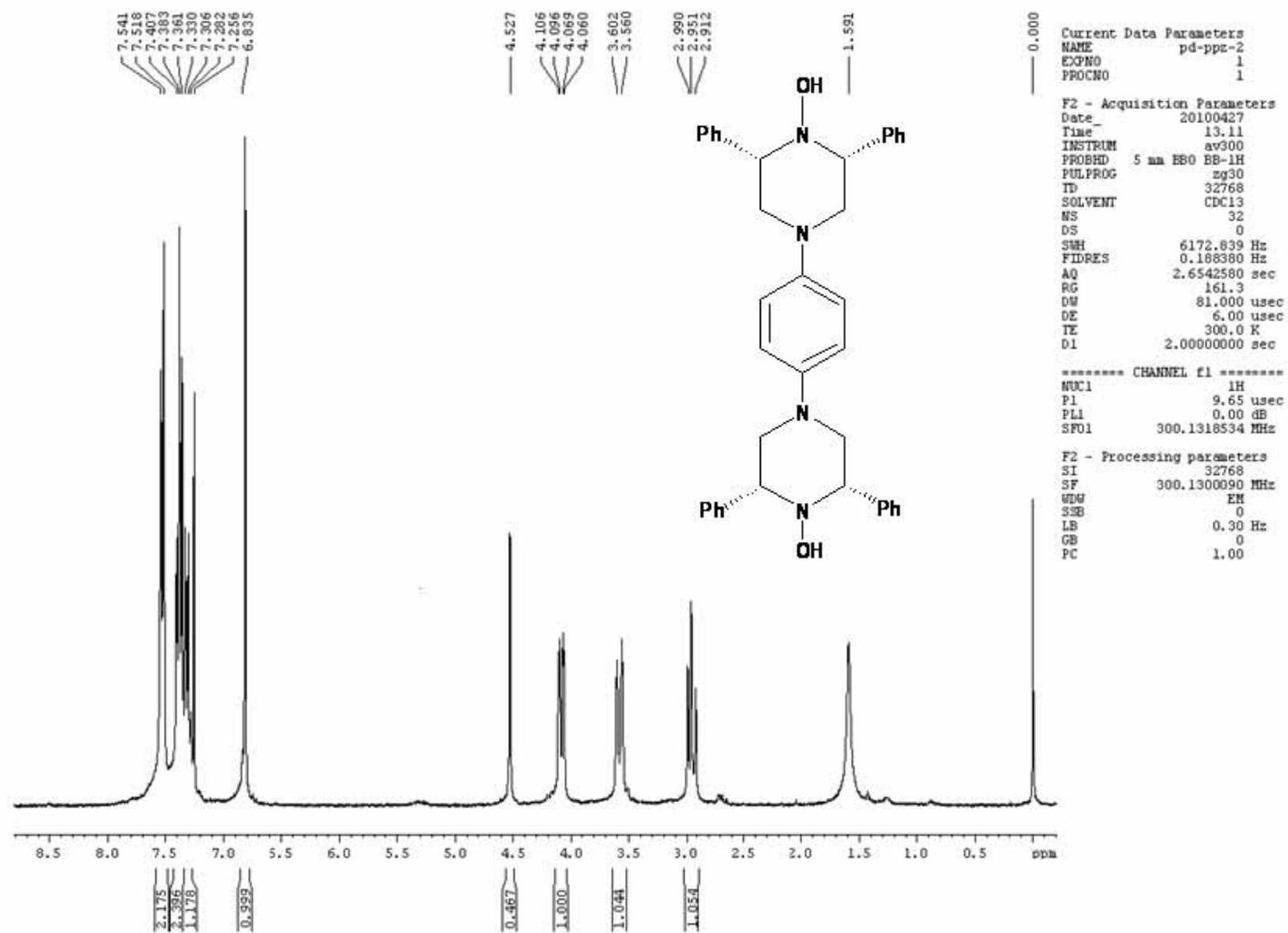
<sup>1</sup>H NMR spectrum of **4** (300 MHz, DMSO-*d*<sub>6</sub>)



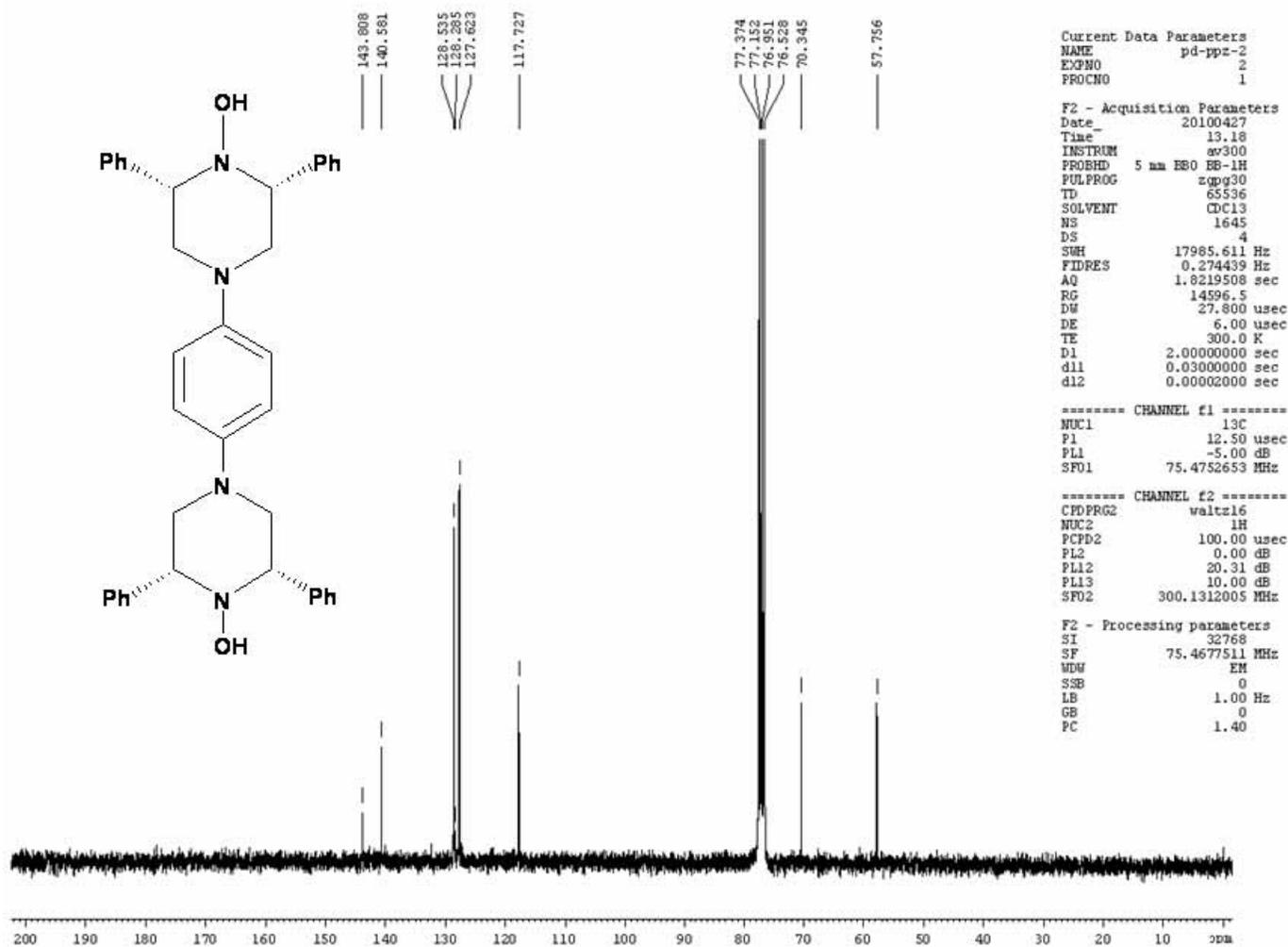
<sup>13</sup>C NMR spectrum of **4** (75 MHz, DMSO-*d*<sub>6</sub>)



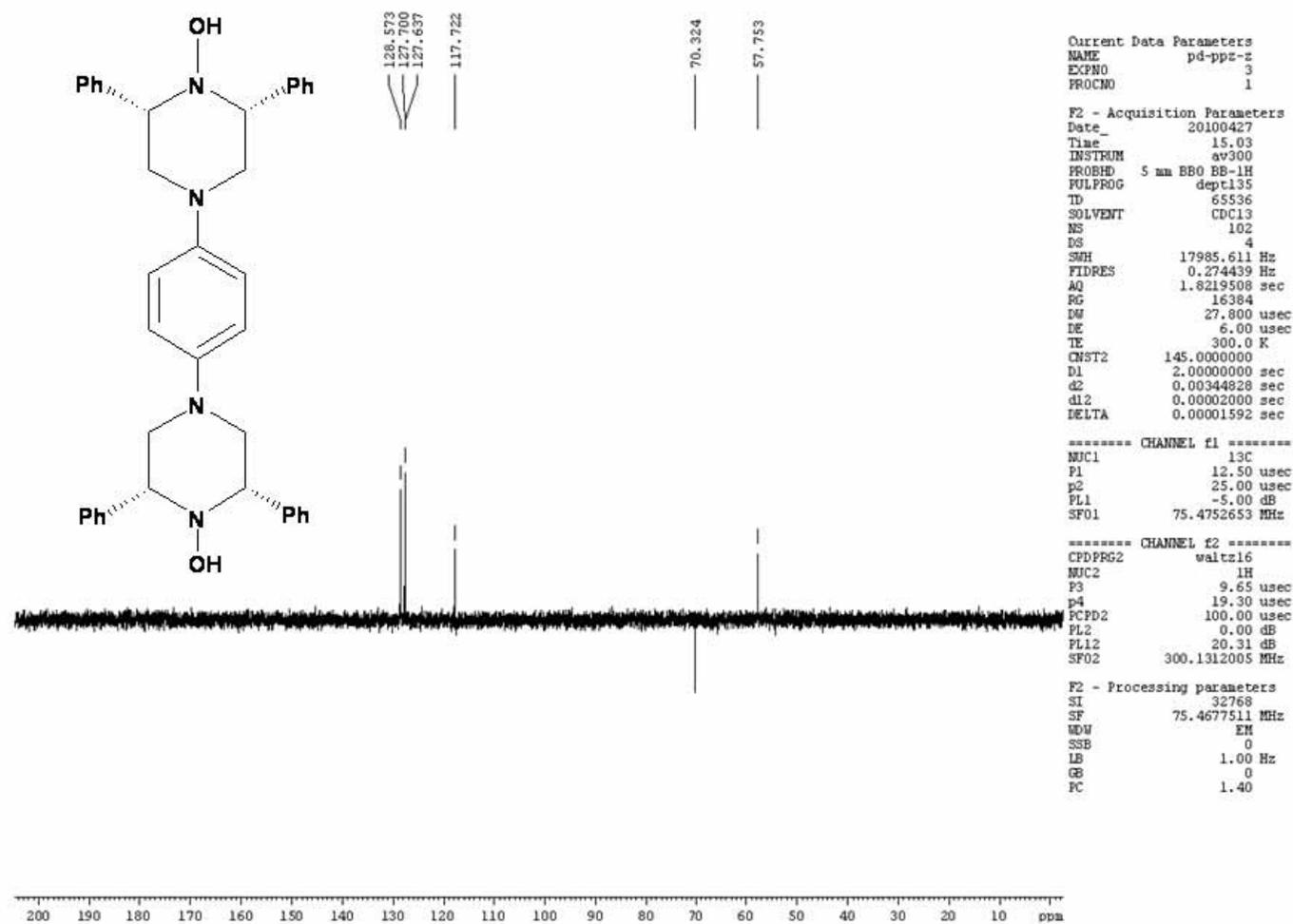
<sup>1</sup>H NMR spectrum of **5** (300 MHz, CDCl<sub>3</sub>)



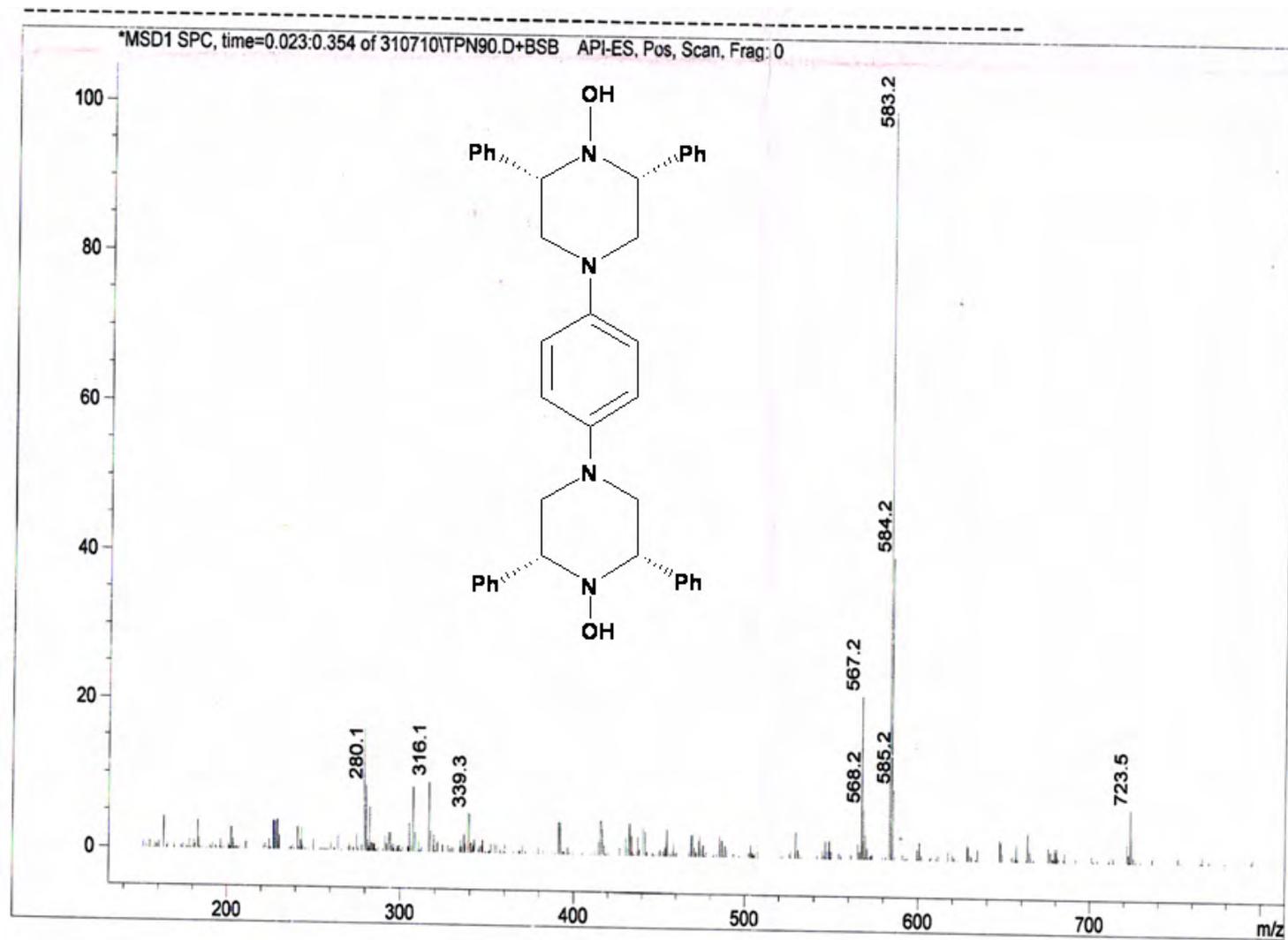
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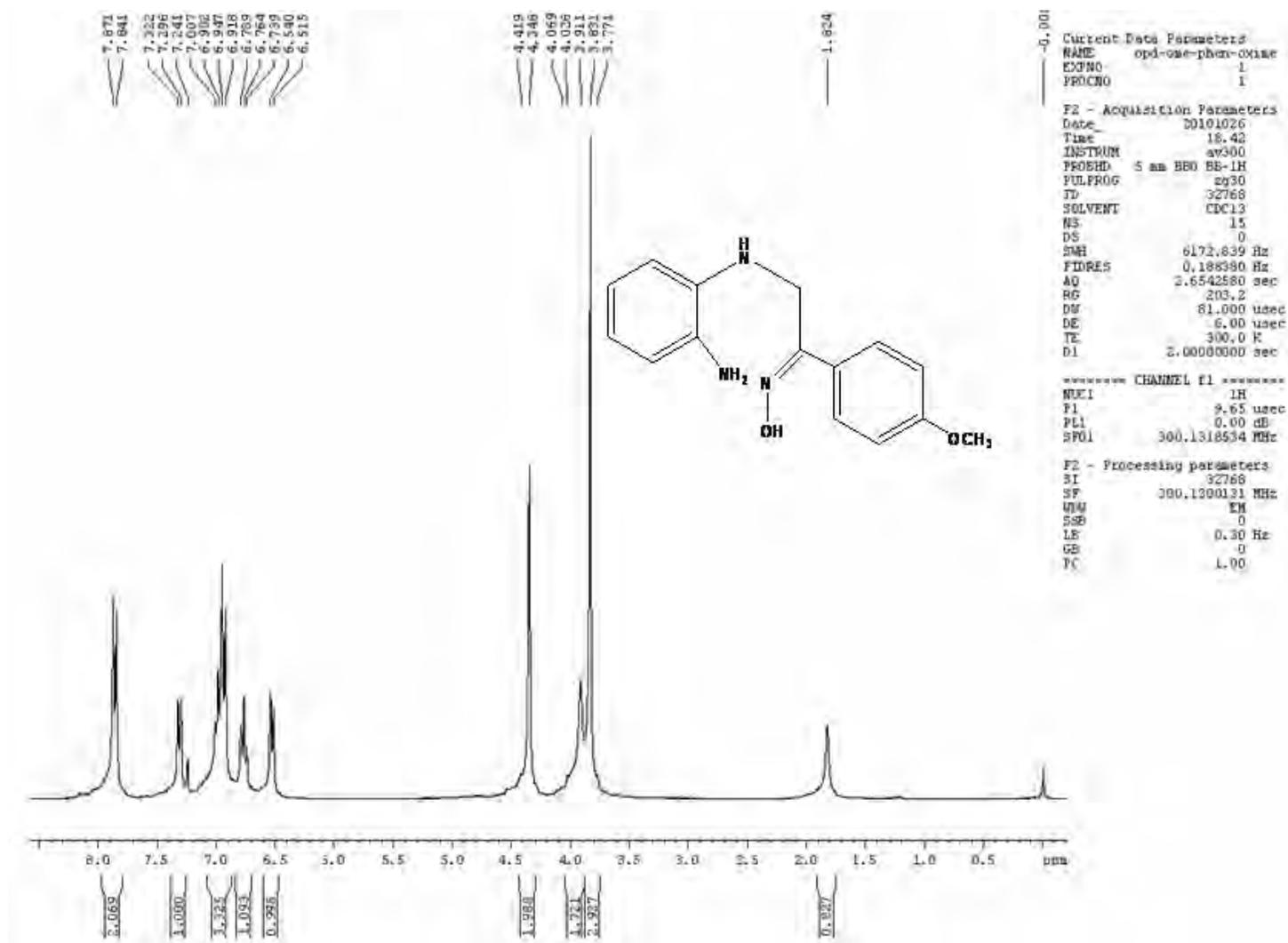
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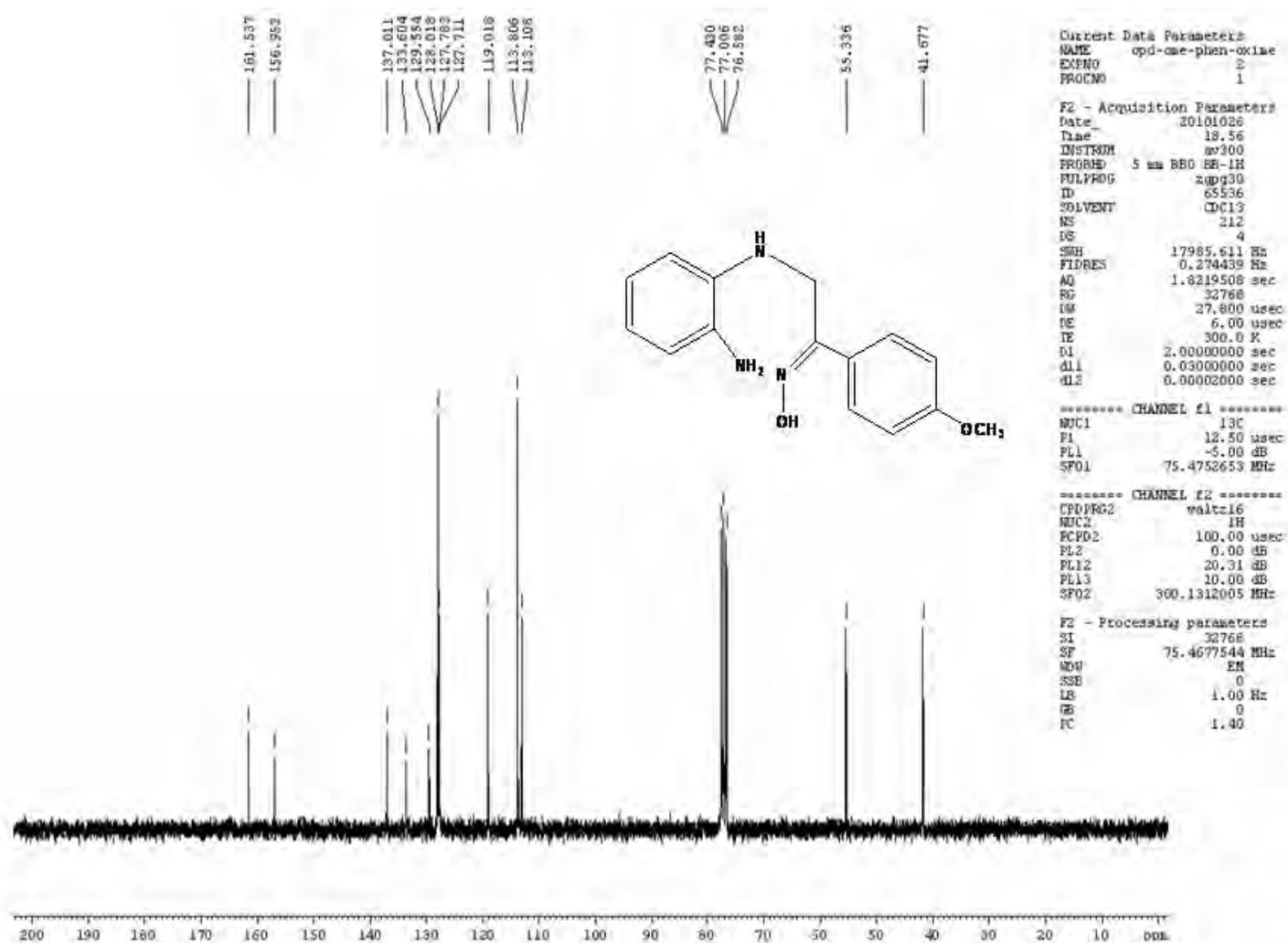
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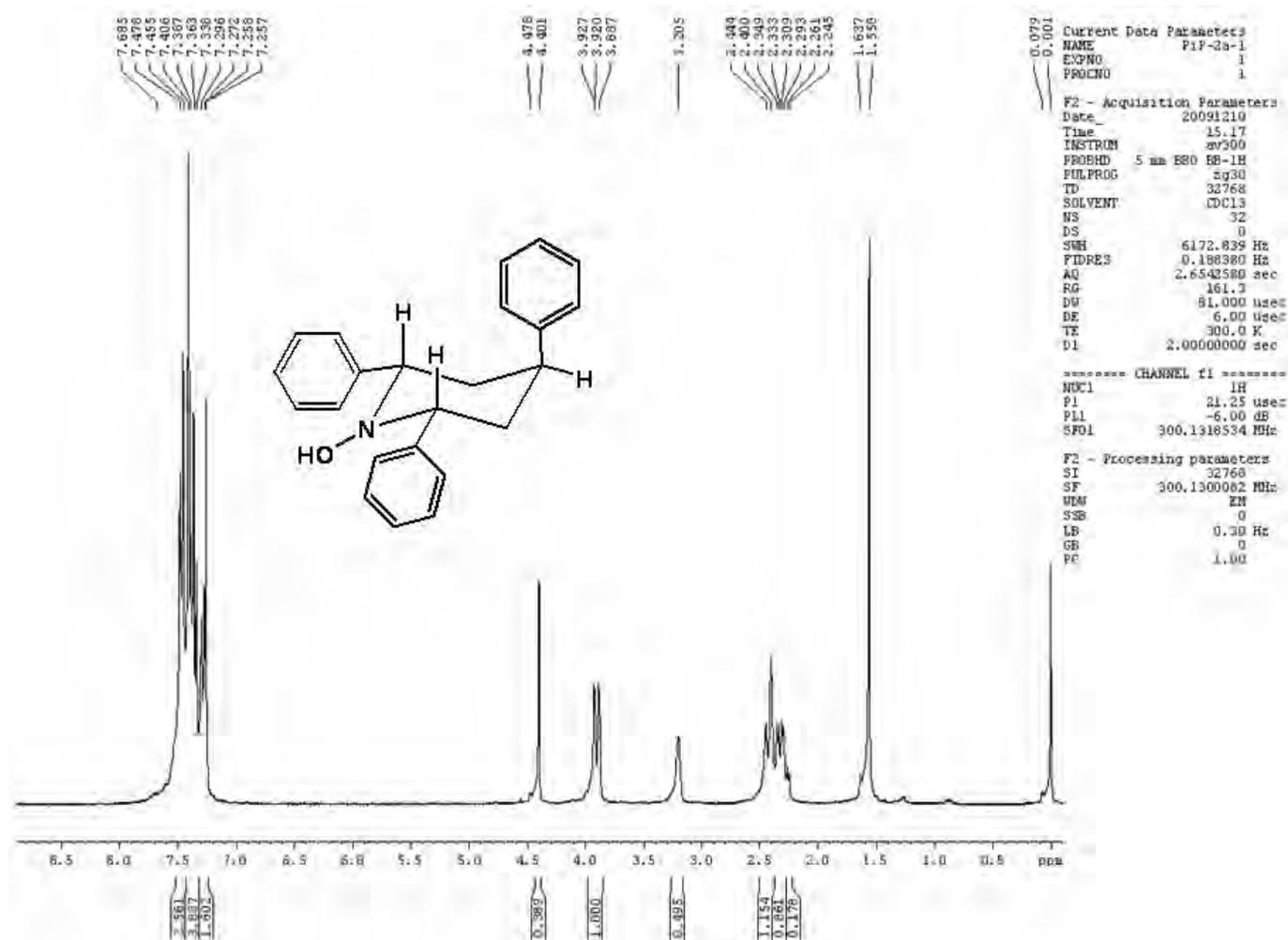
<sup>1</sup>H NMR spectrum of **6** (300 MHz, CDCl<sub>3</sub>)



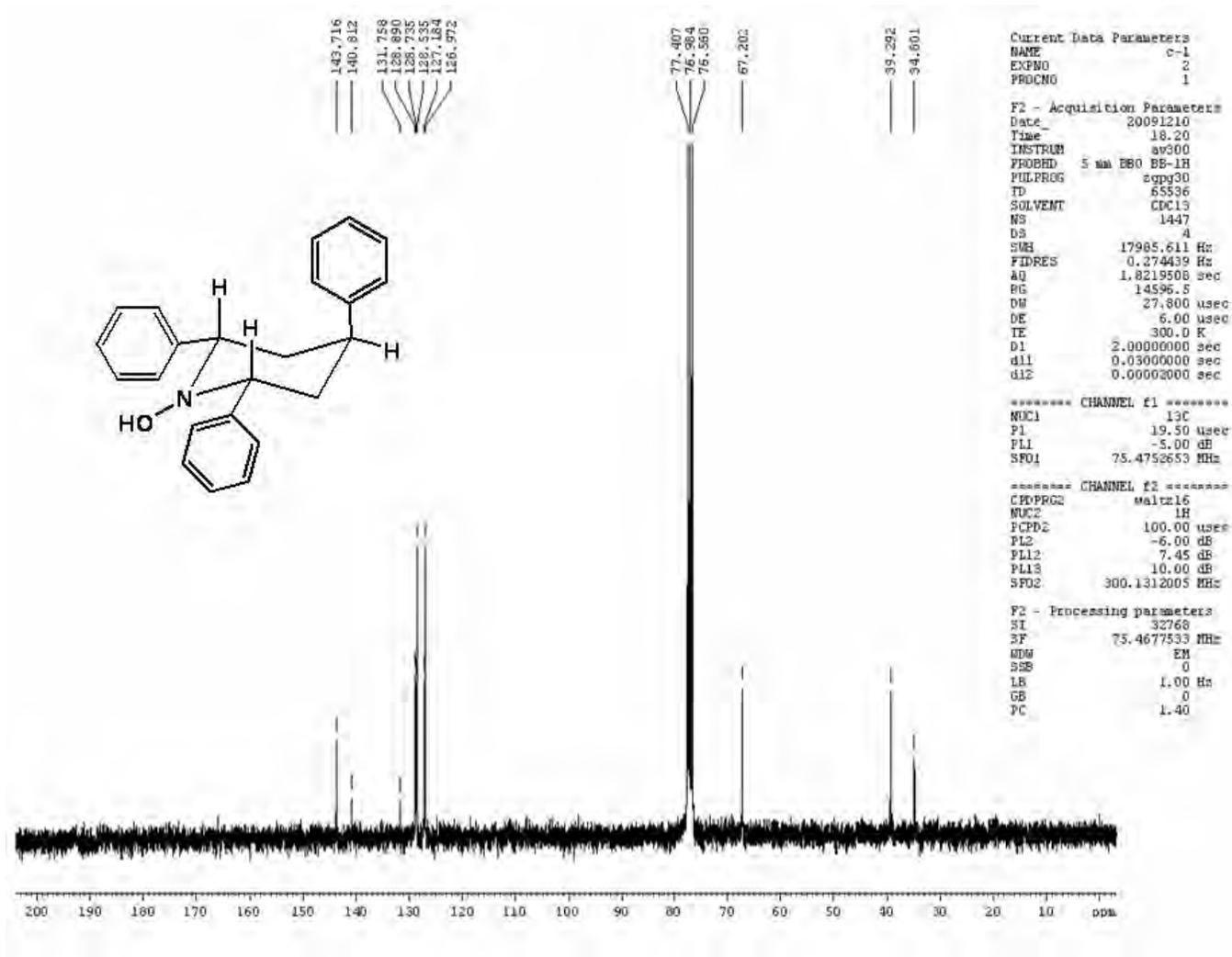
<sup>13</sup>C NMR spectrum of **6** (75 MHz, CDCl<sub>3</sub>)



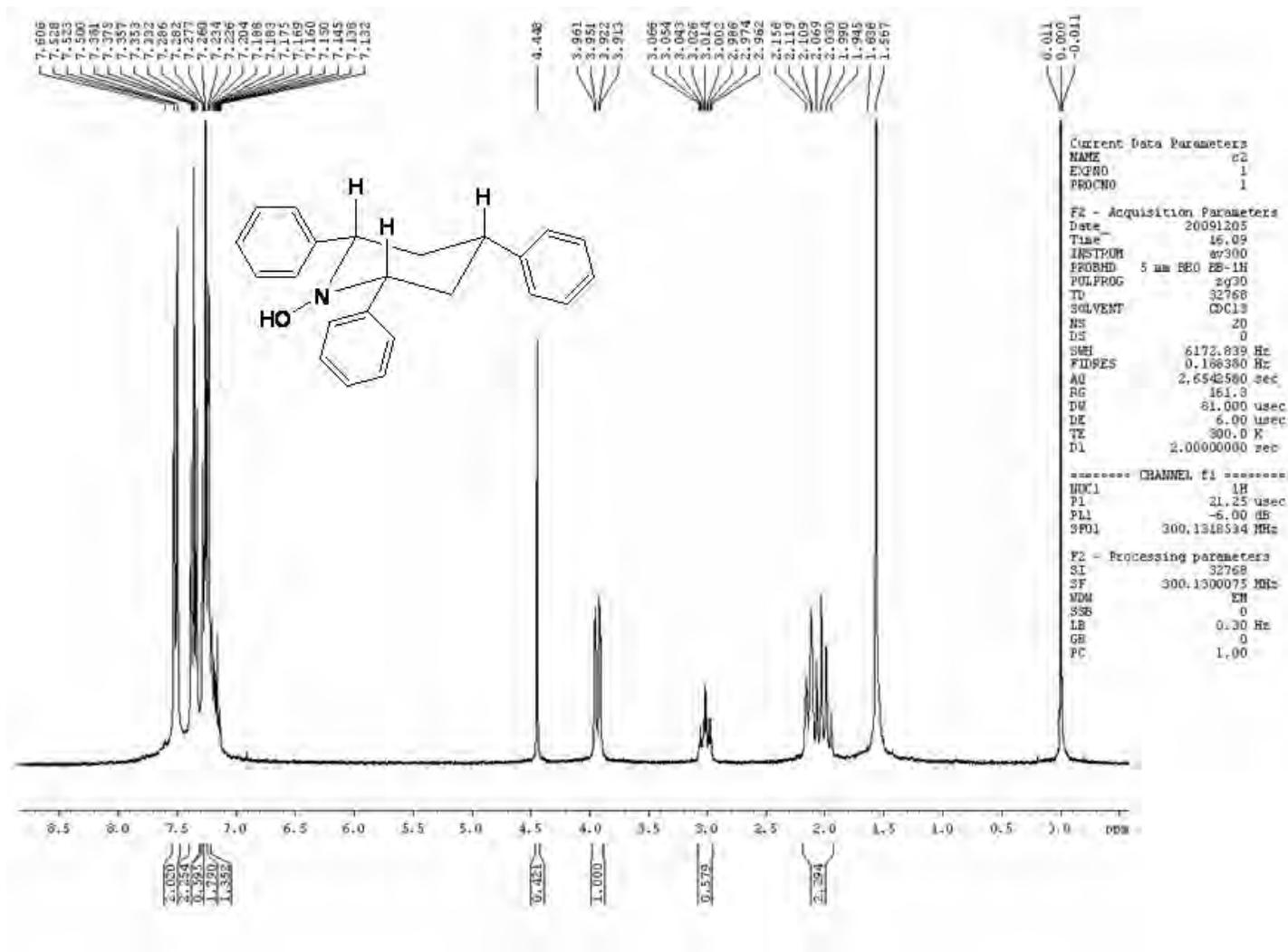
<sup>1</sup>H NMR spectrum of **8a** (300 MHz, CDCl<sub>3</sub>)



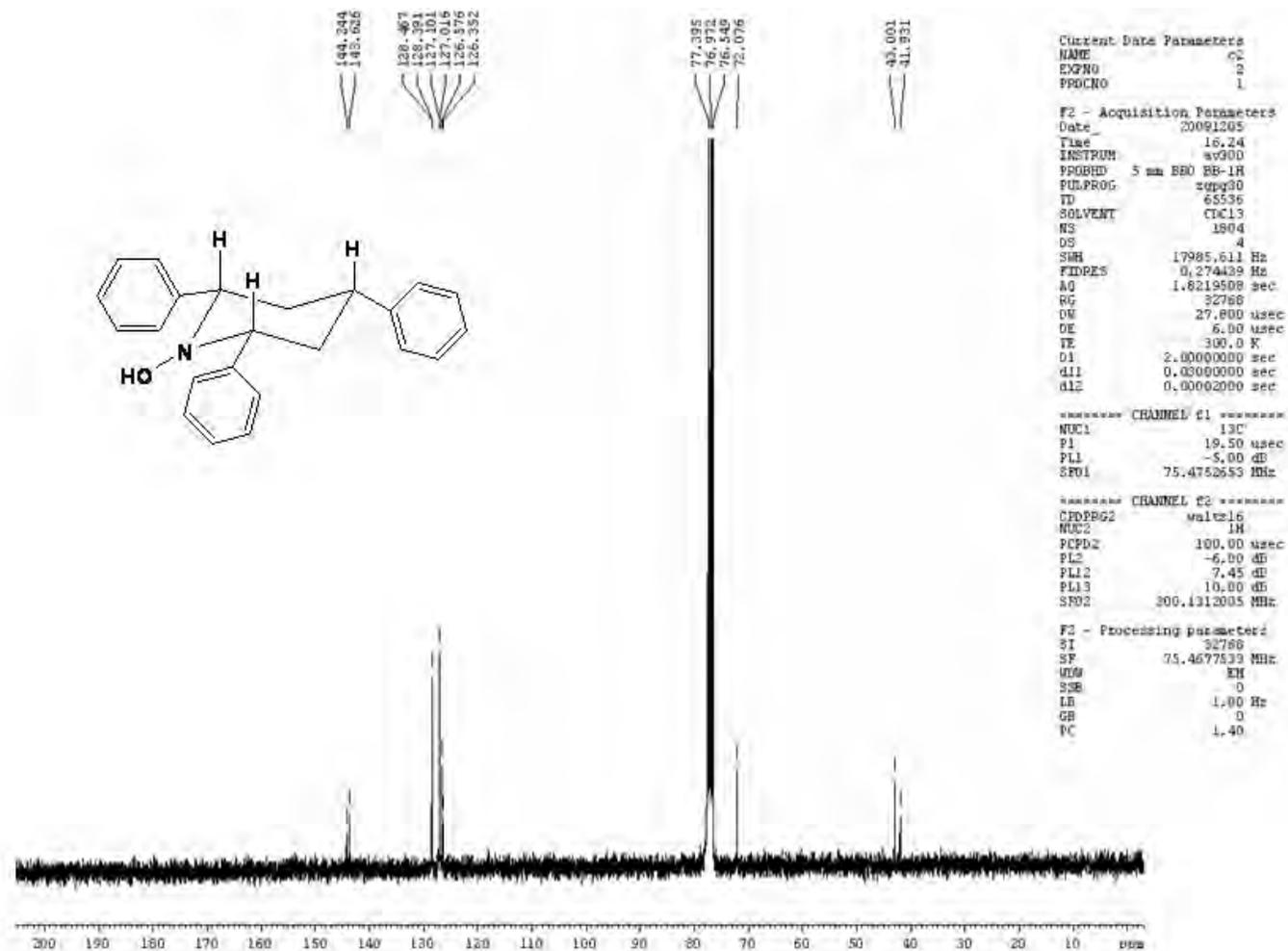
$^{13}\text{C}$  NMR spectrum of **8a** (75 MHz,  $\text{CDCl}_3$ )



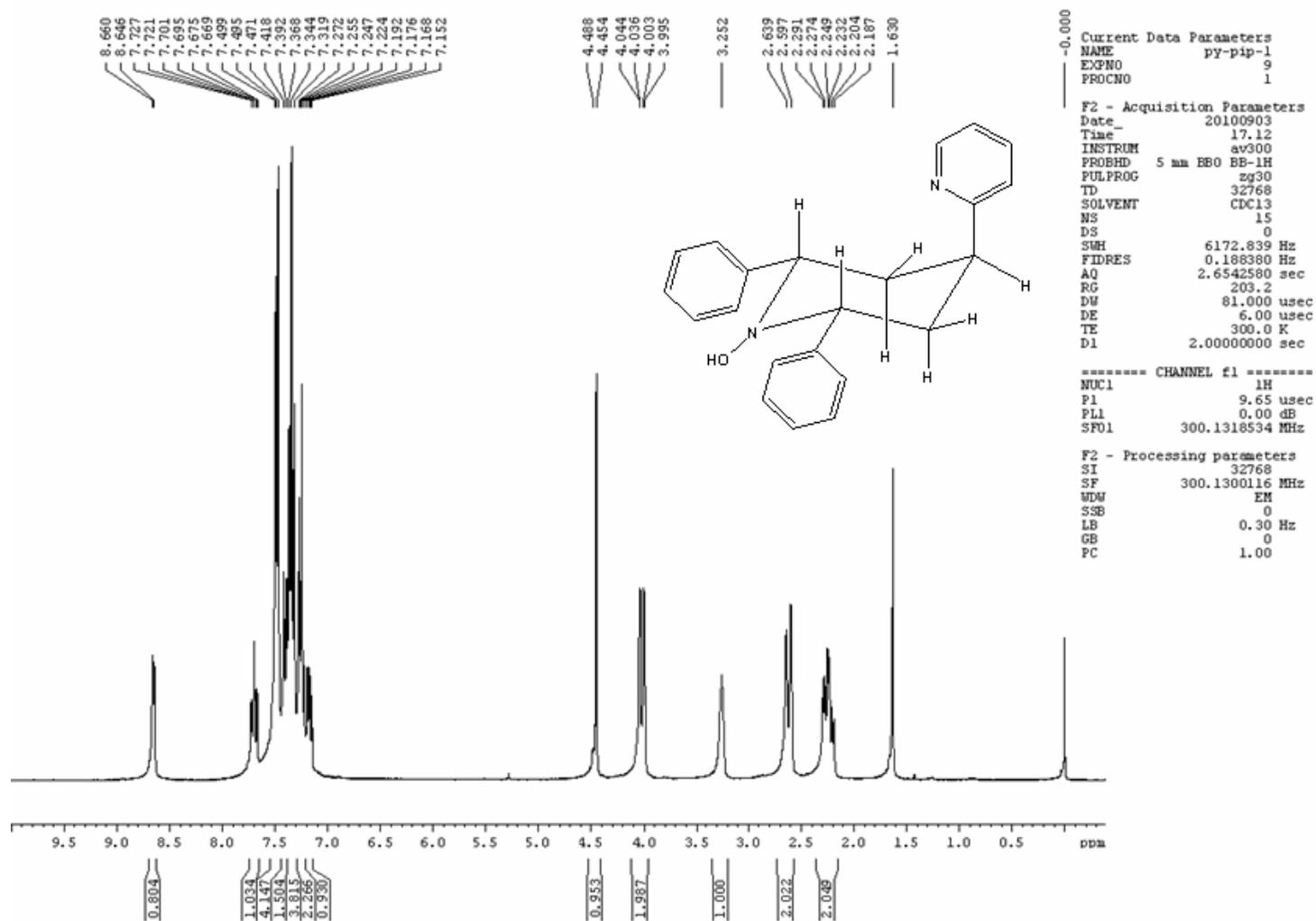
<sup>1</sup>H NMR spectrum of **9a** (300 MHz, CDCl<sub>3</sub>)



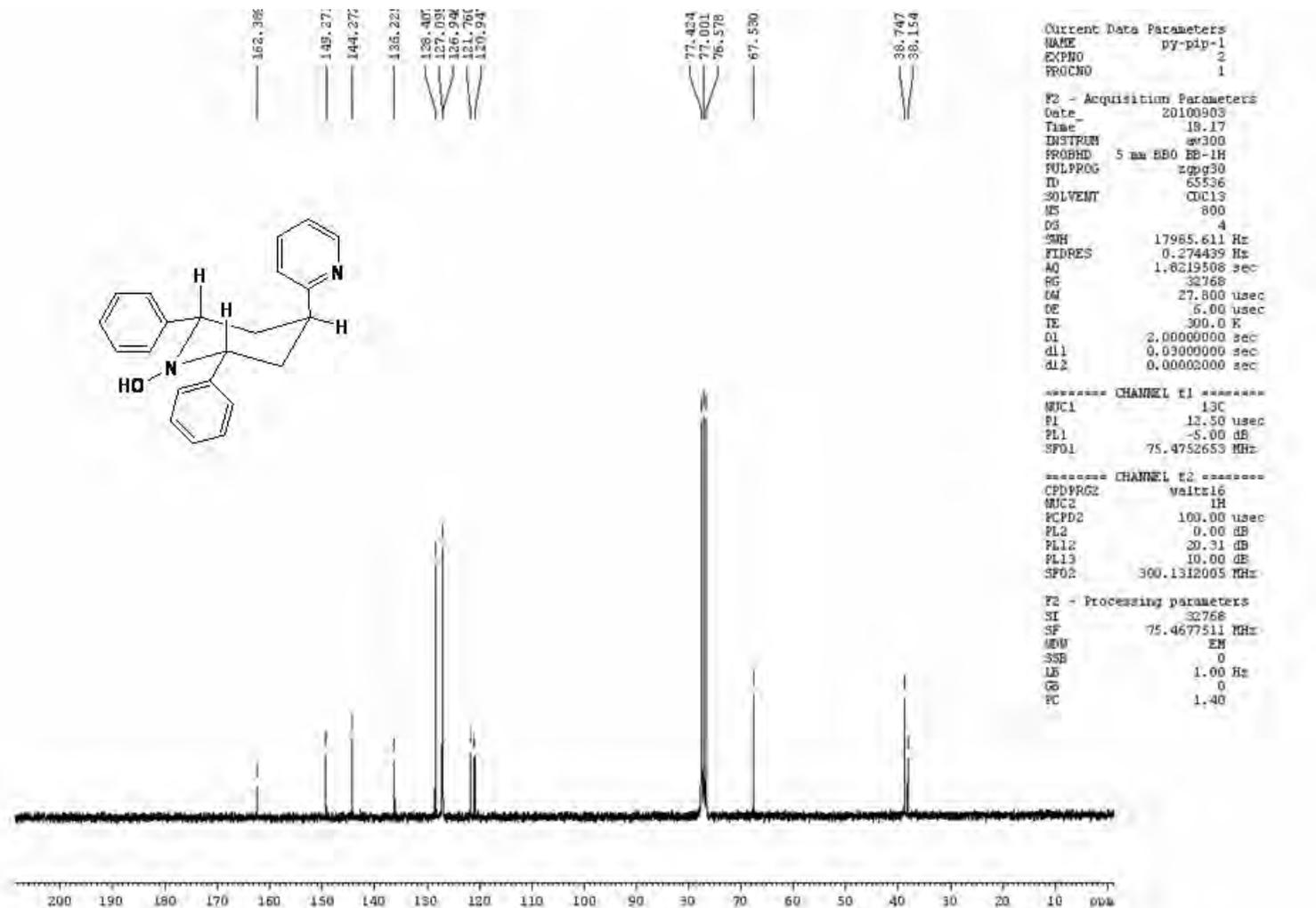
<sup>13</sup>C NMR spectrum of **9a** (75 MHz, CDCl<sub>3</sub>)



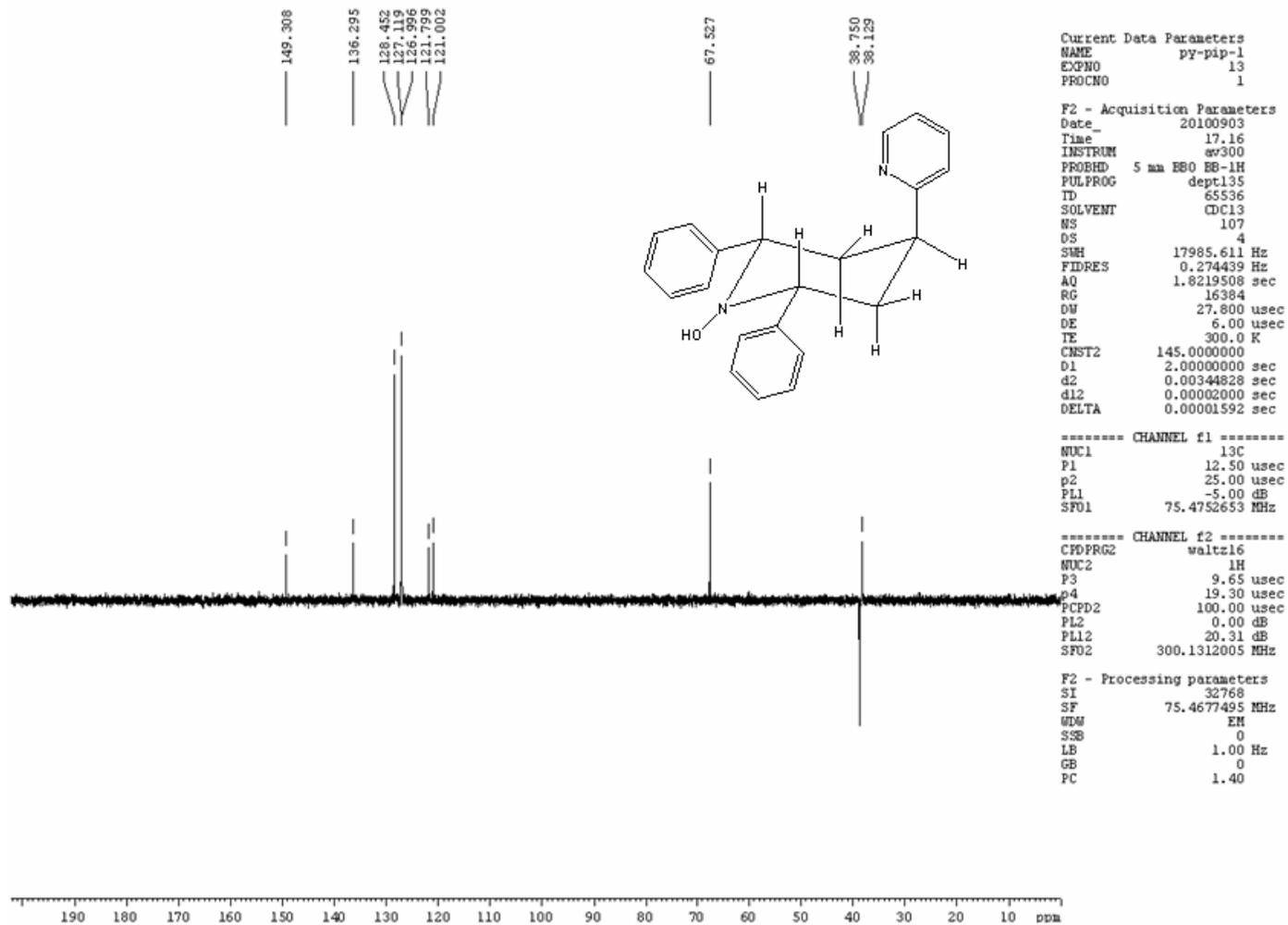
<sup>1</sup>H NMR spectrum of **8b** (300 MHz, CDCl<sub>3</sub>)



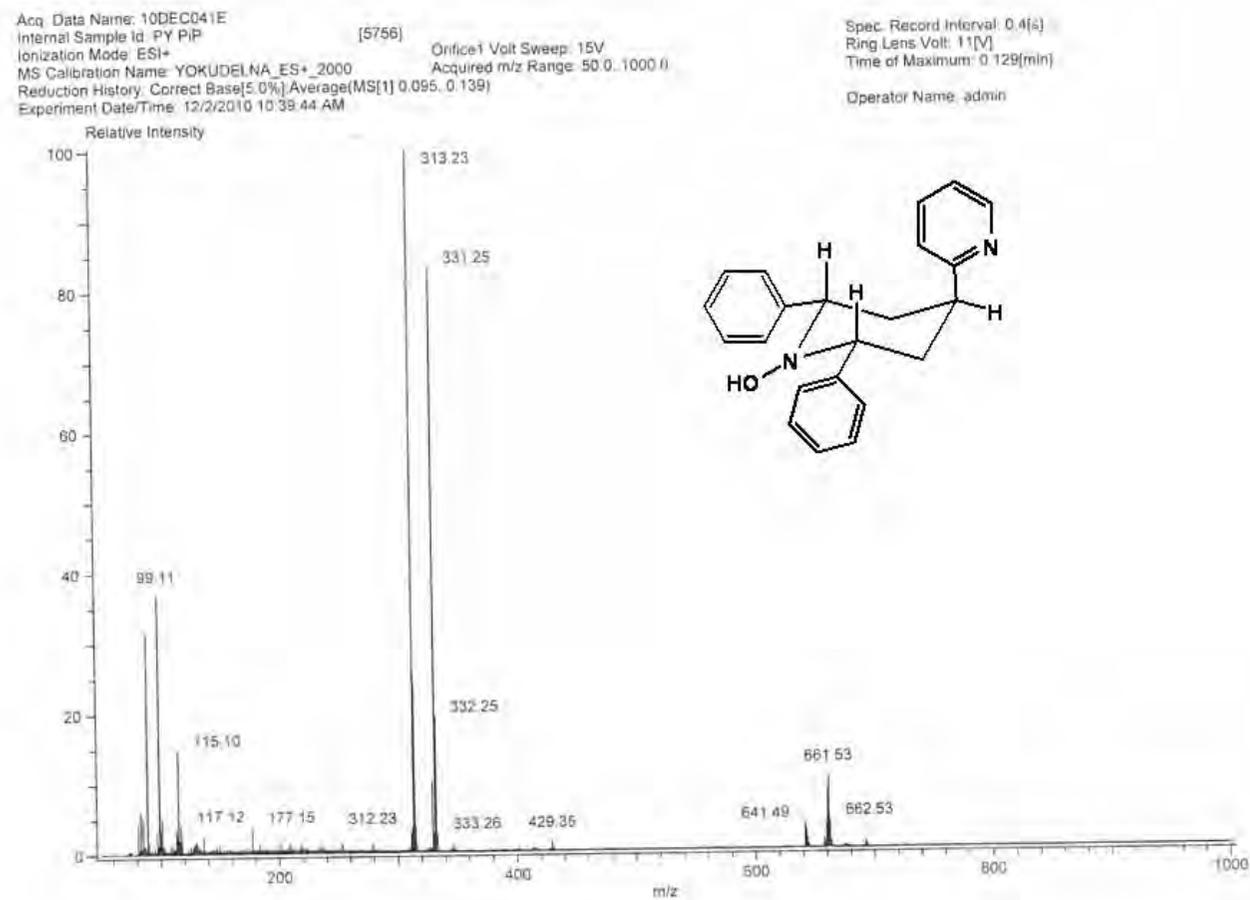
<sup>13</sup>C NMR spectrum of **8b** (75 MHz, CDCl<sub>3</sub>)



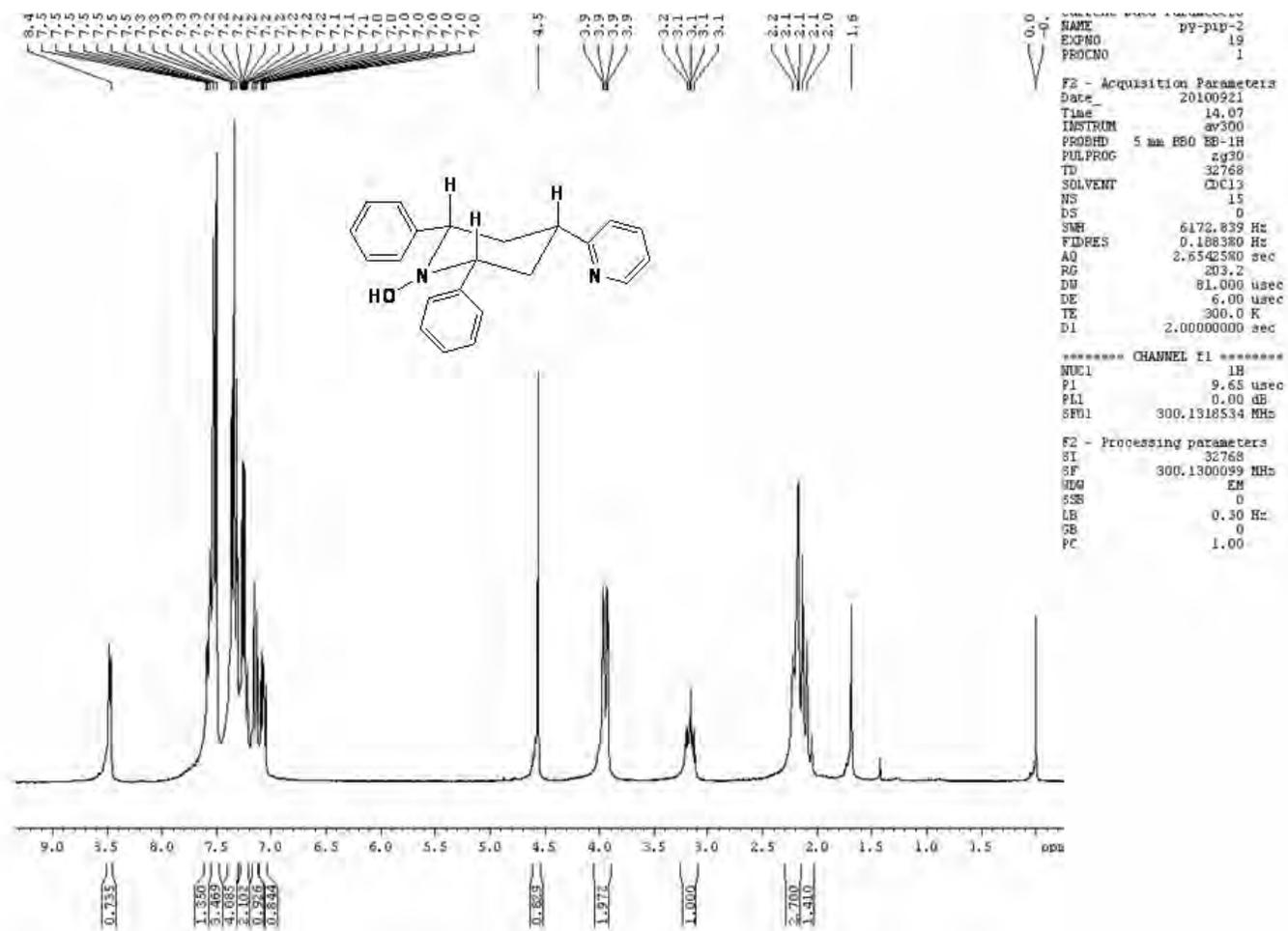
DEPT-135 spectrum of **8b**



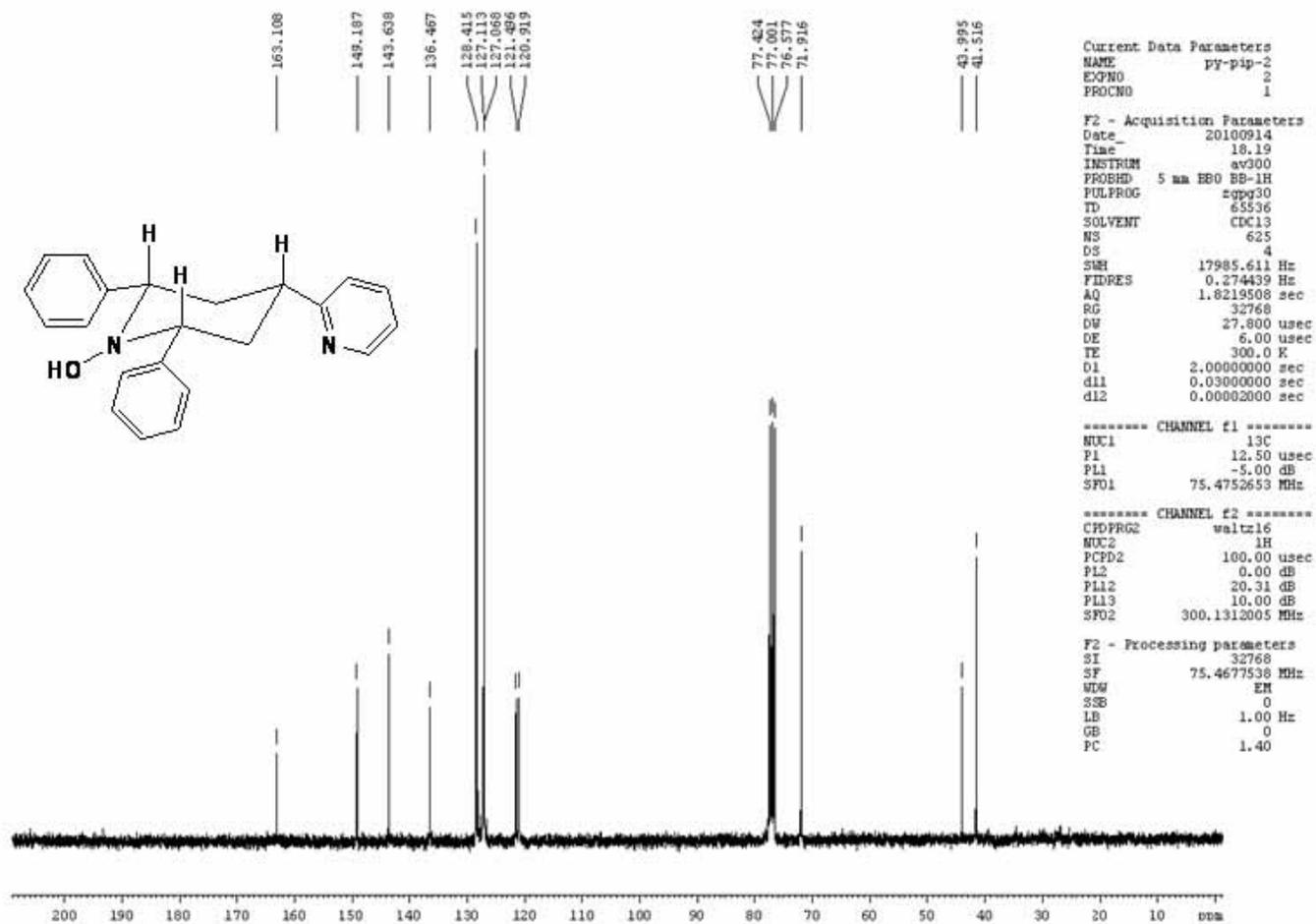
Mass spectrum of **8b**



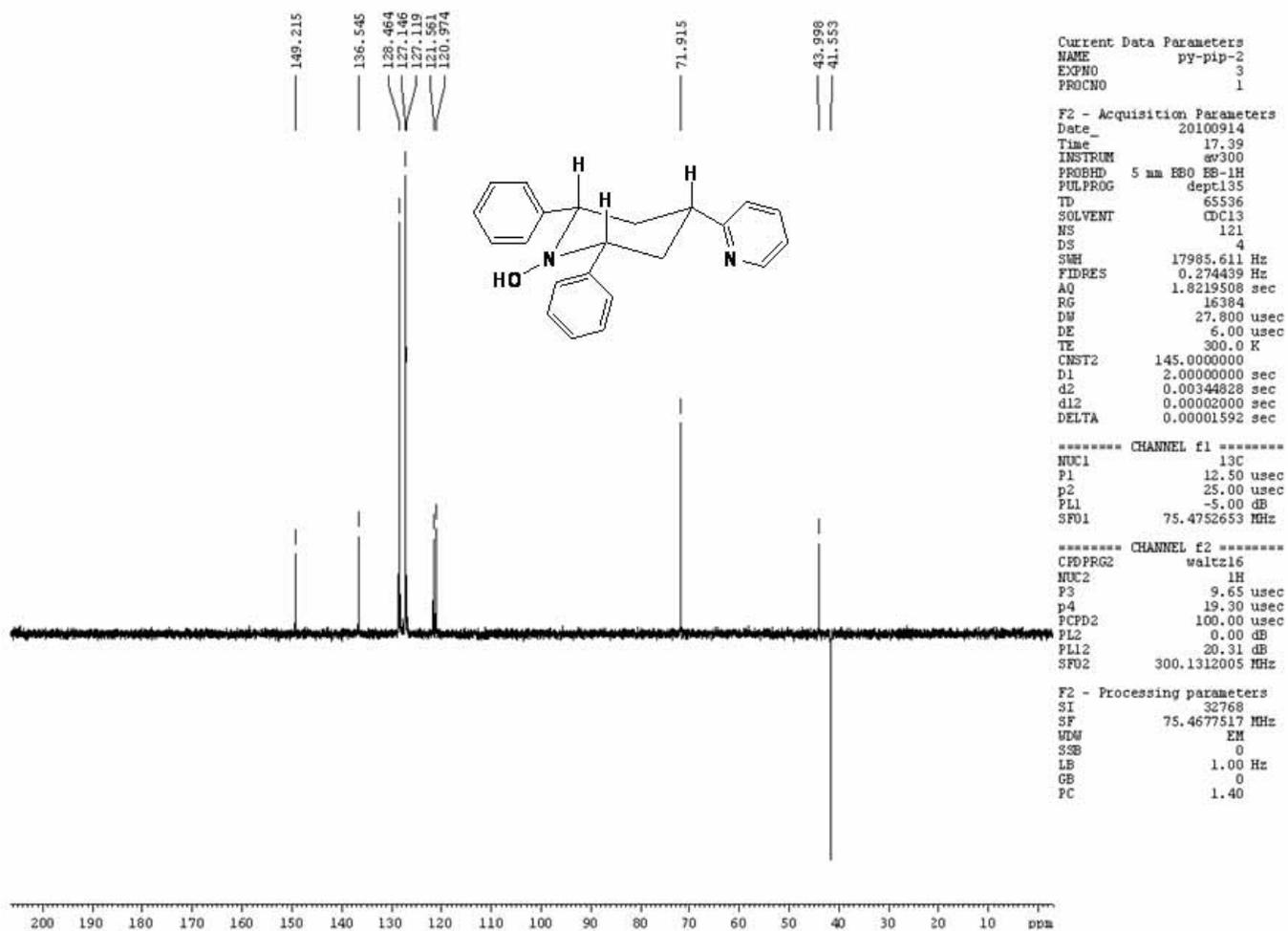
<sup>1</sup>H NMR spectrum of **9b** (300 MHz, CDCl<sub>3</sub>)



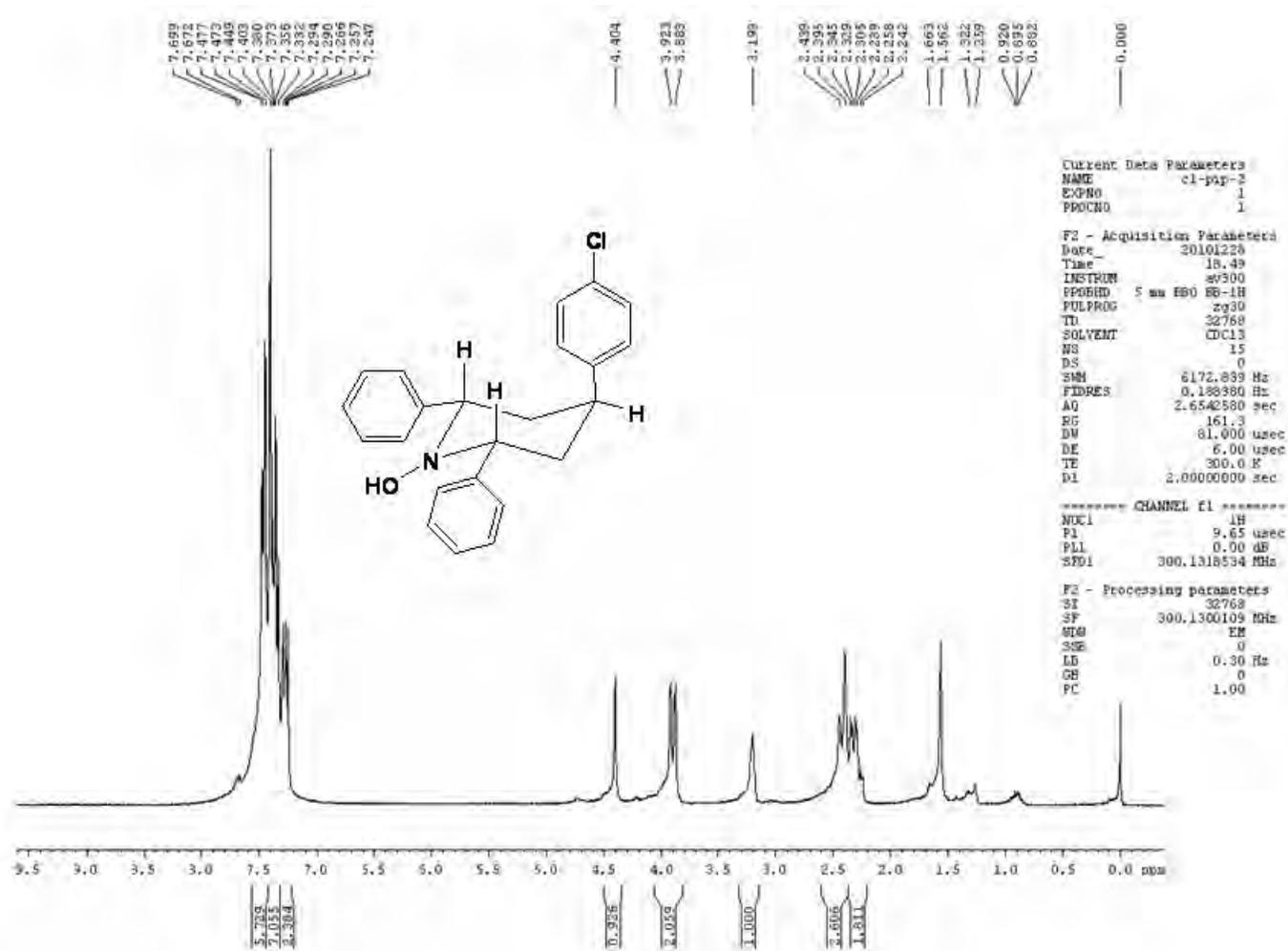
$^{13}\text{C}$  NMR spectrum of **9b** (75 MHz,  $\text{CDCl}_3$ )



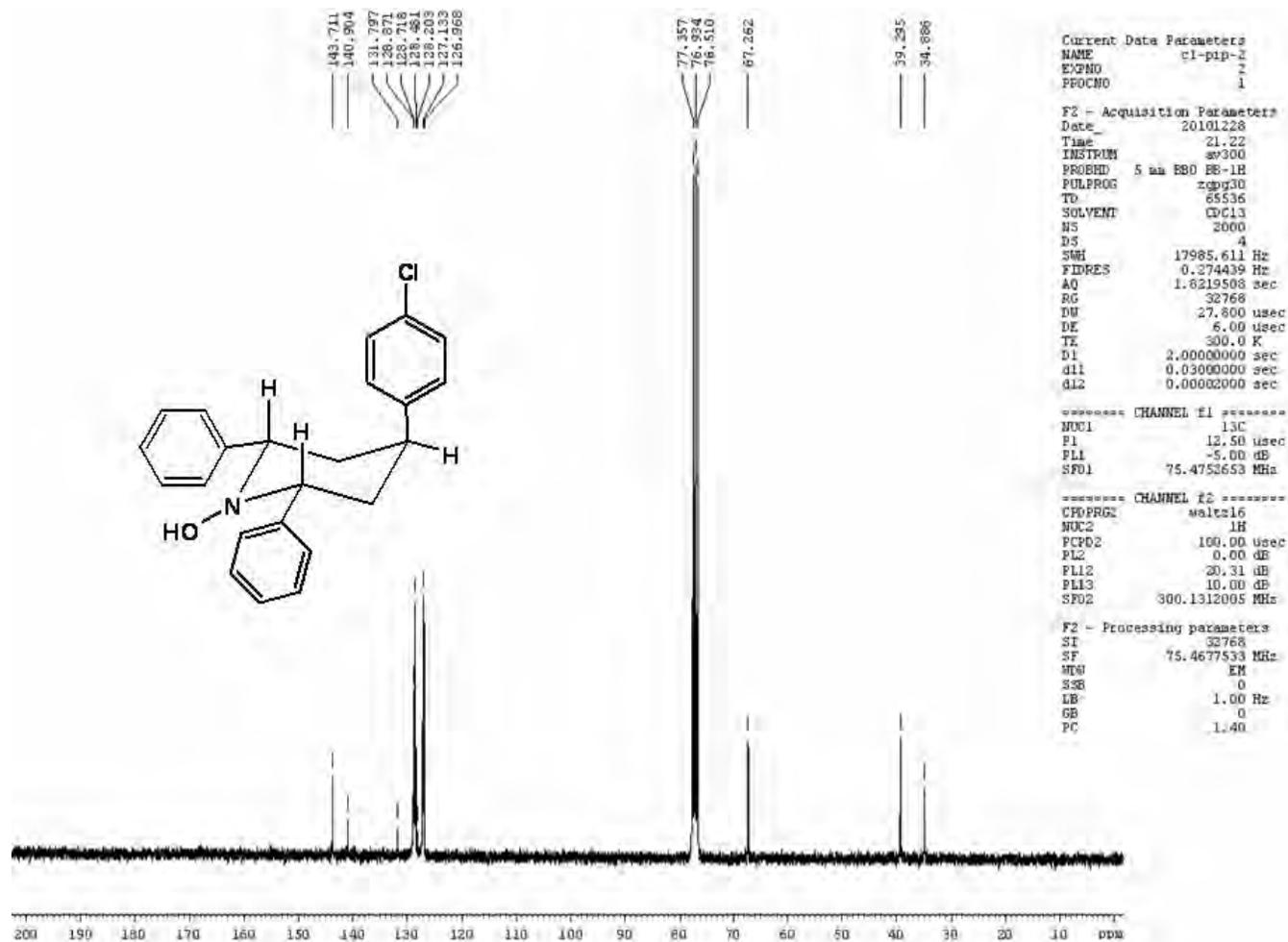
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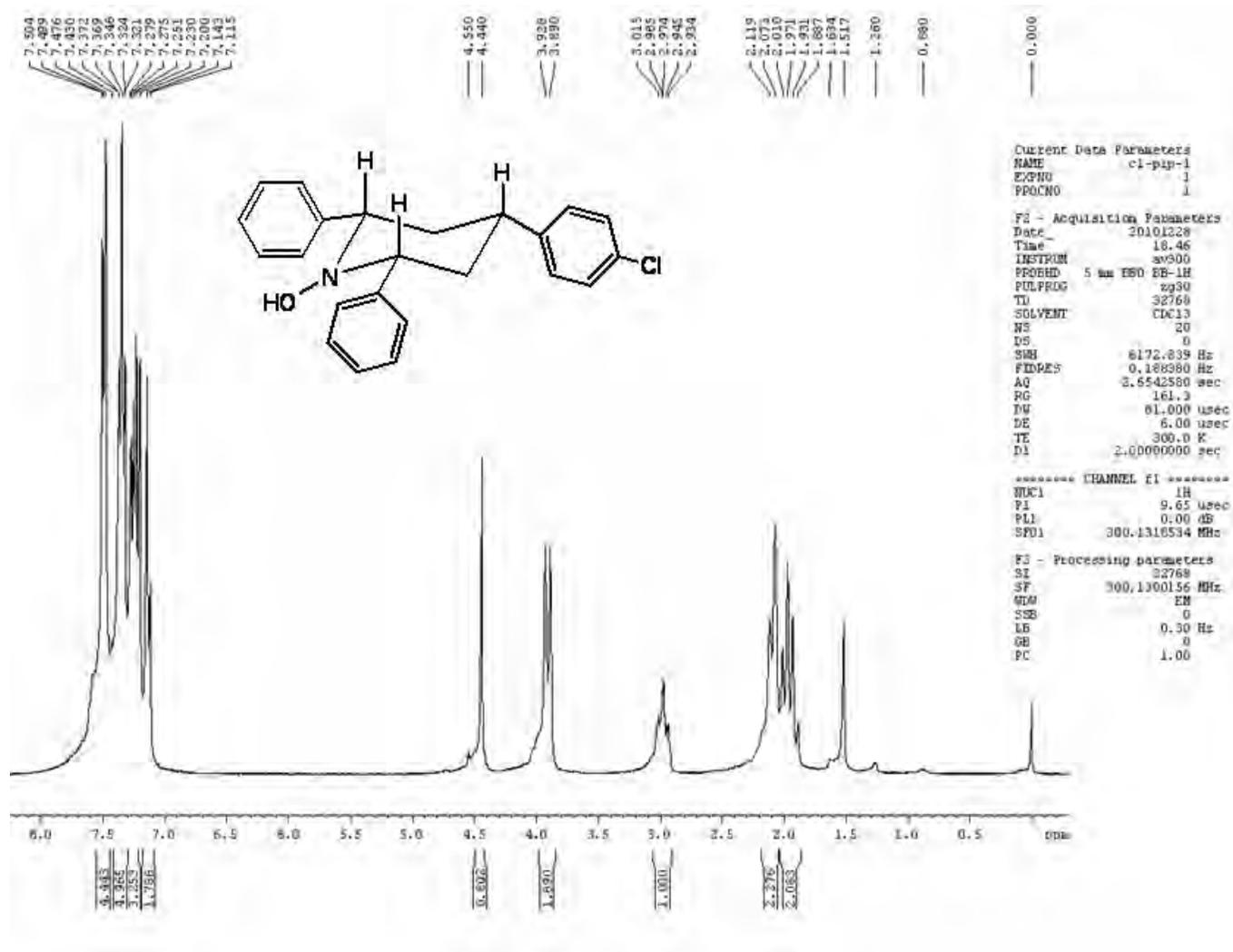
<sup>1</sup>H NMR spectrum of **8c** (300 MHz, CDCl<sub>3</sub>)



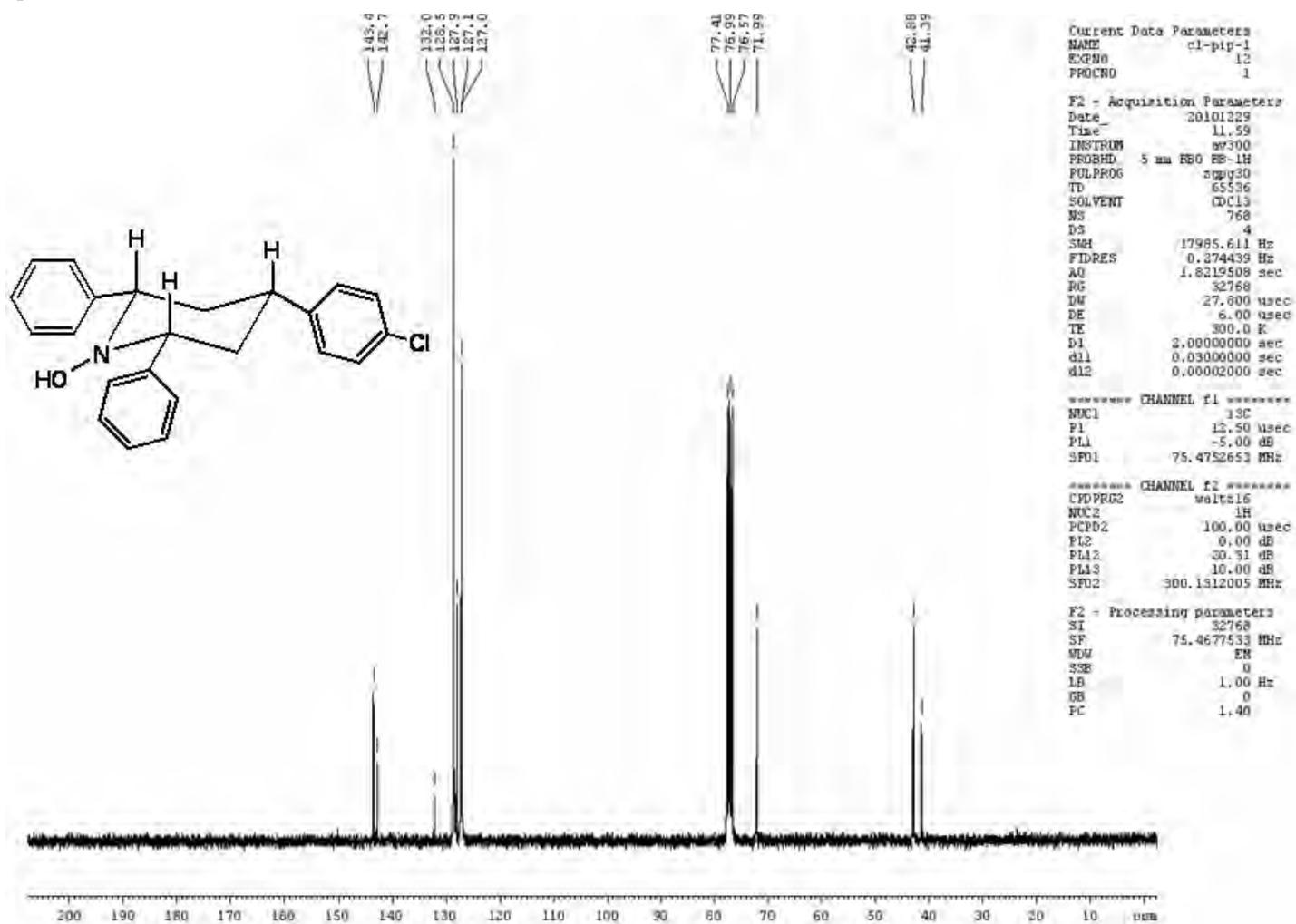
$^{13}\text{C}$  NMR spectrum of **8c** (75 MHz,  $\text{CDCl}_3$ )



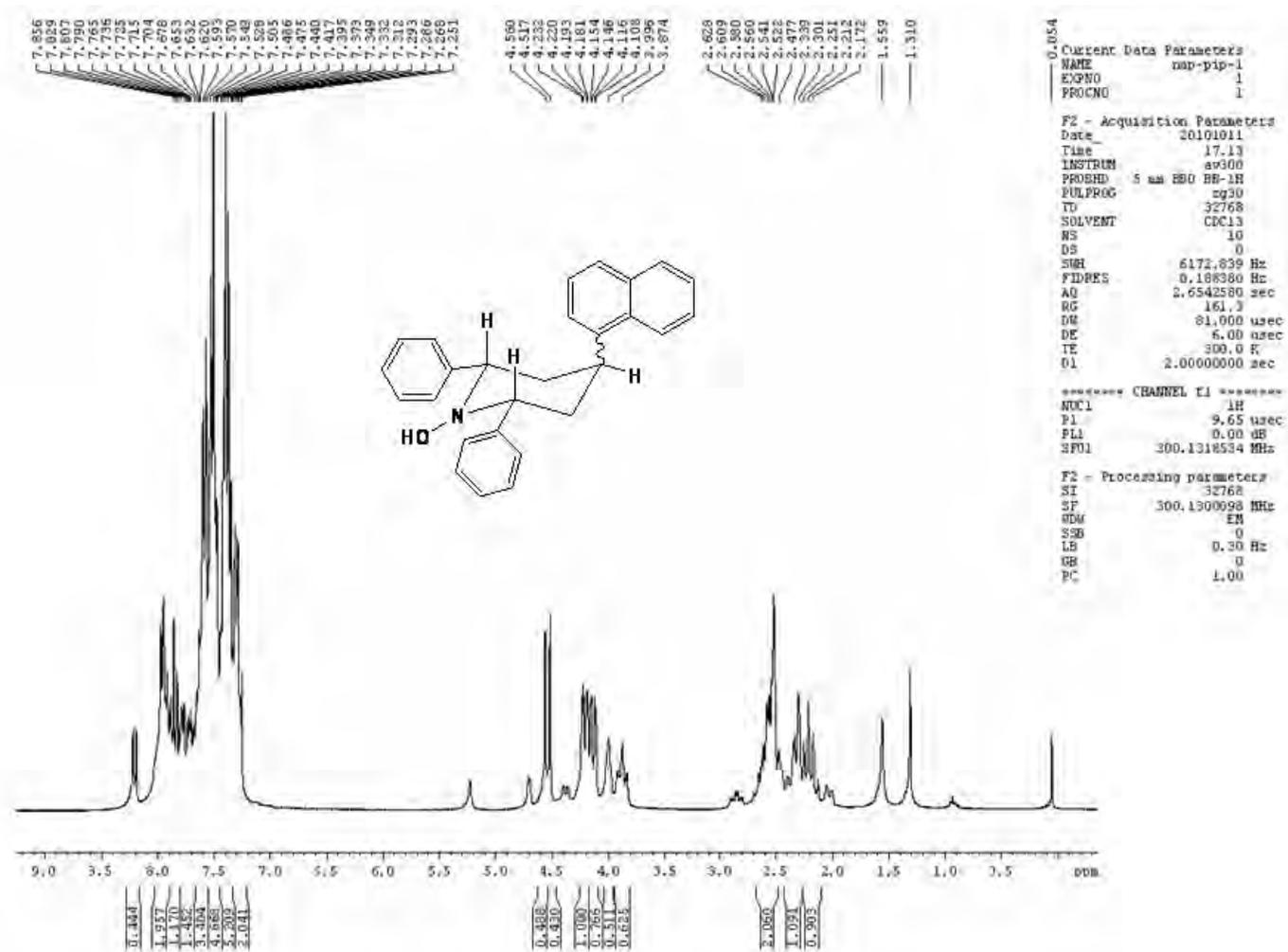
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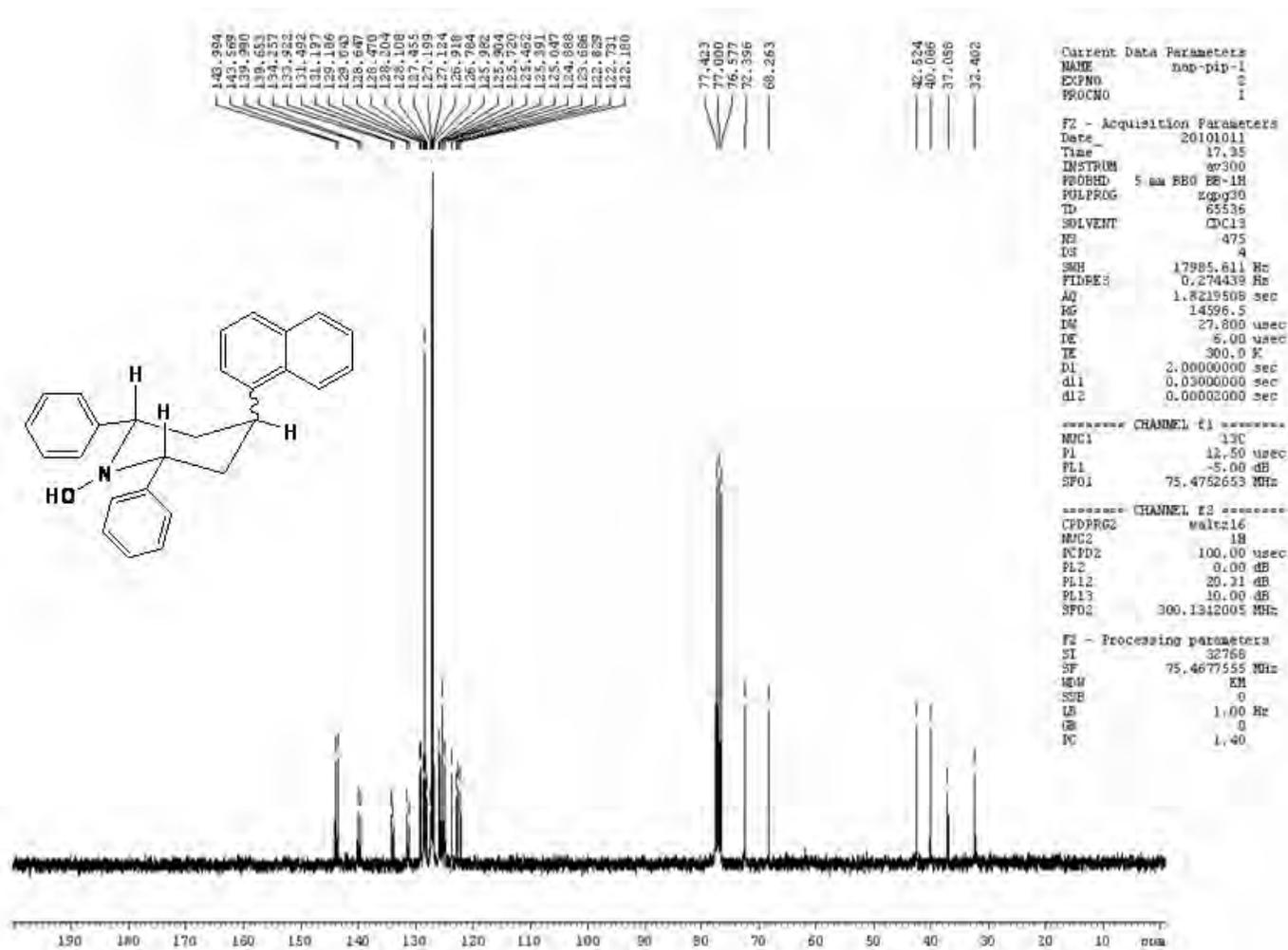
<sup>13</sup>C NMR spectrum of **9c** (75 MHz, CDCl<sub>3</sub>)



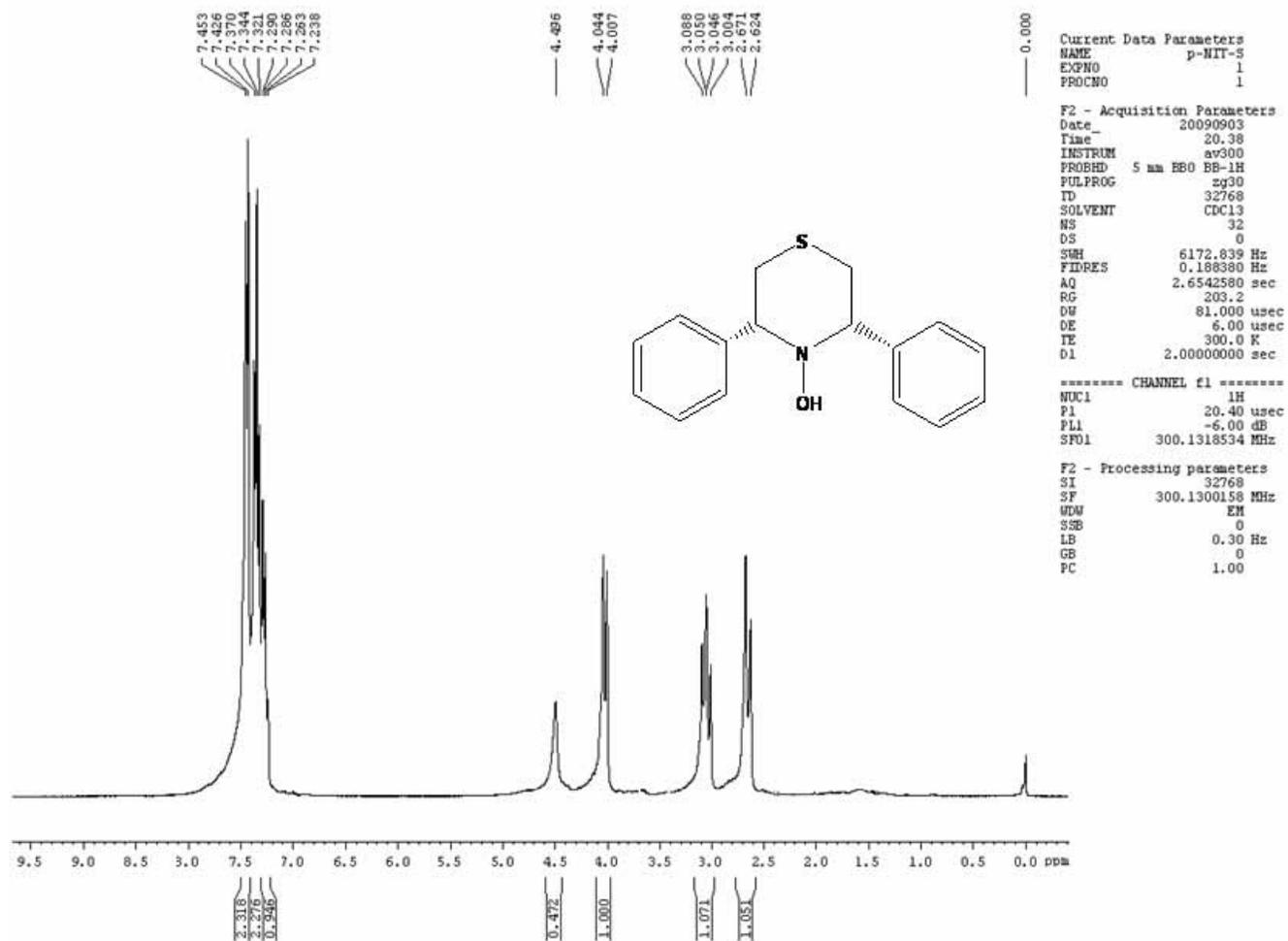
<sup>1</sup>H NMR spectrum of **8d** and **9d** (300 MHz, CDCl<sub>3</sub>)



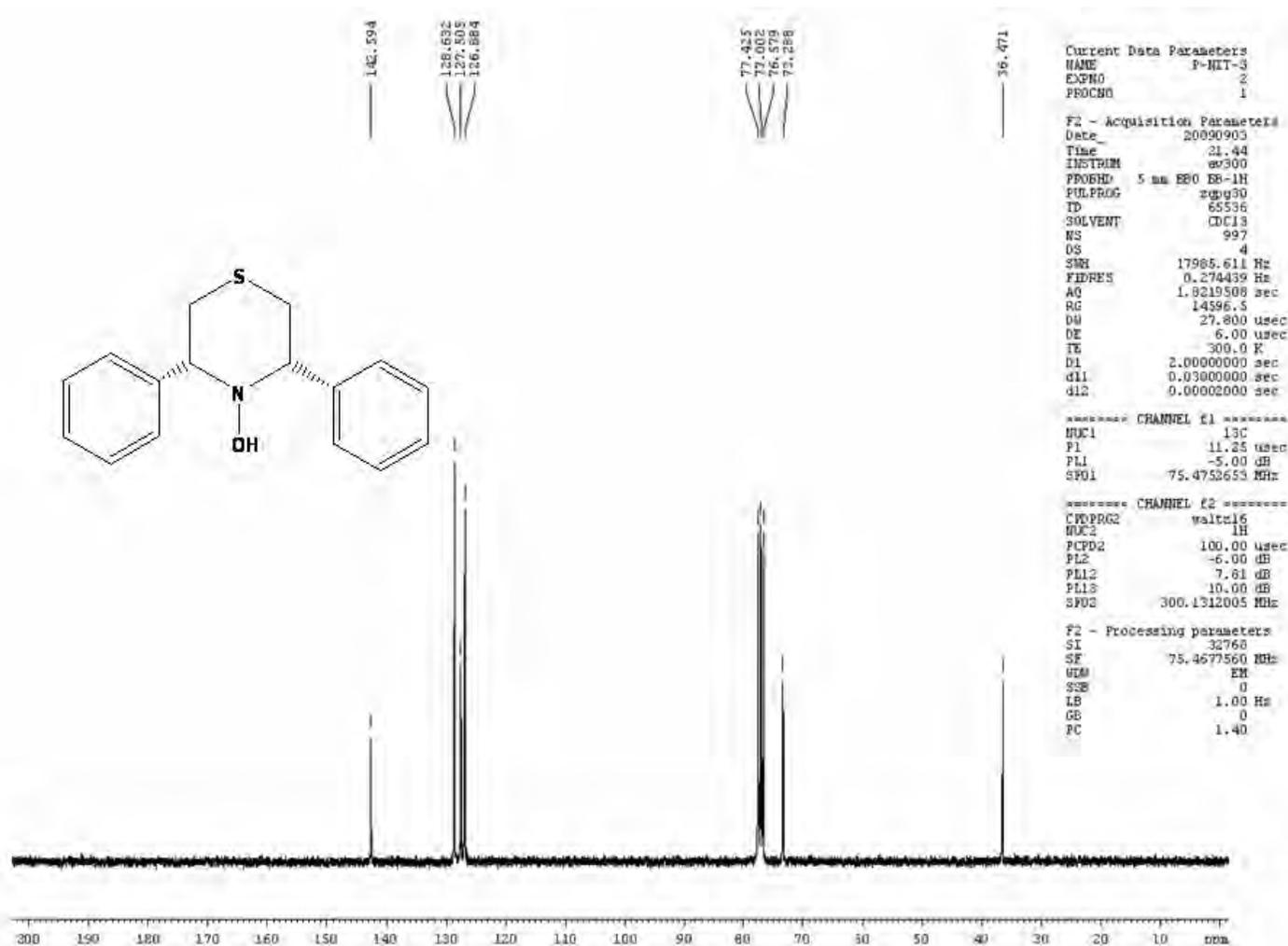
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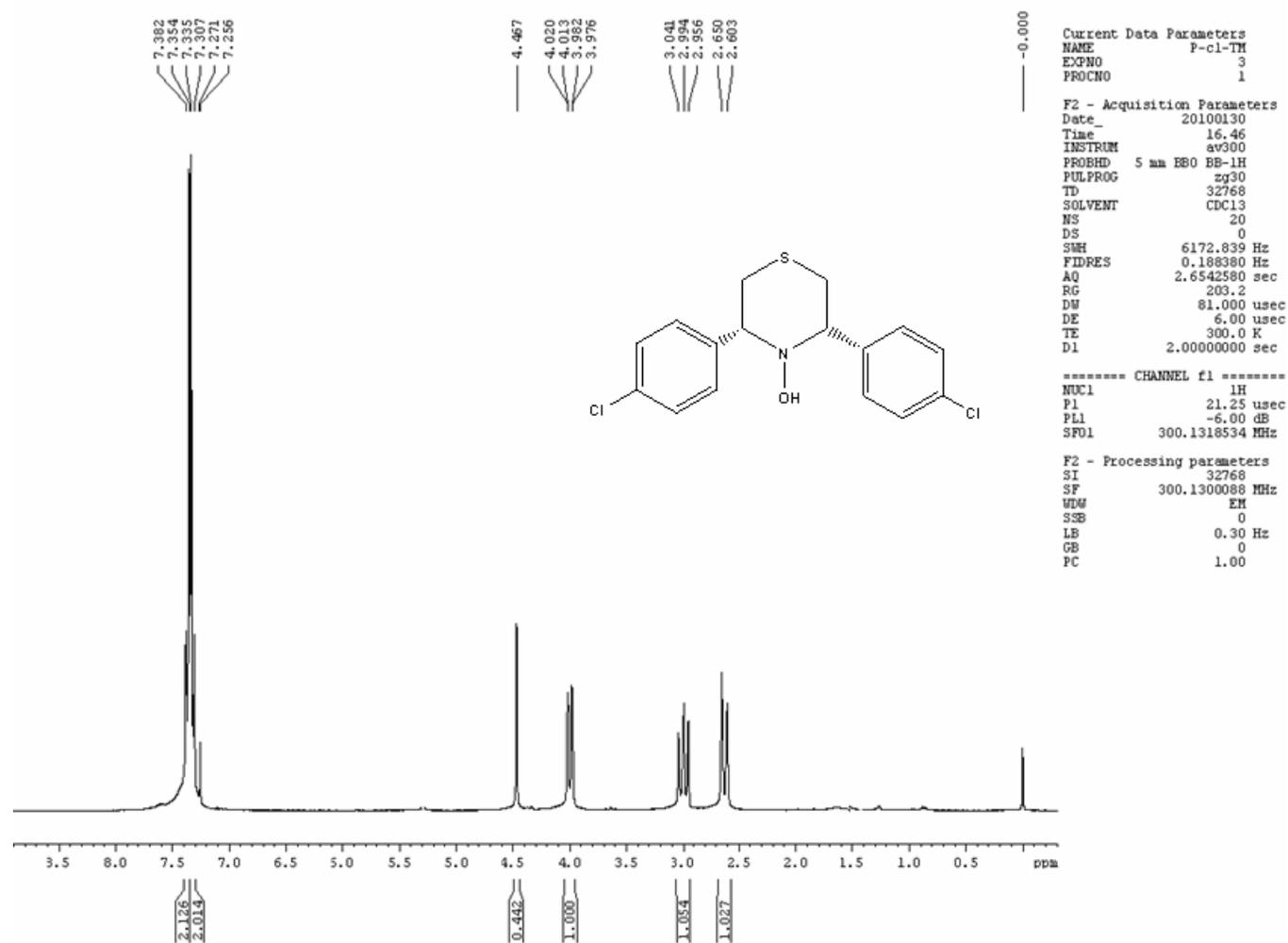
<sup>1</sup>H NMR spectrum of **11a** (300 MHz, CDCl<sub>3</sub>)



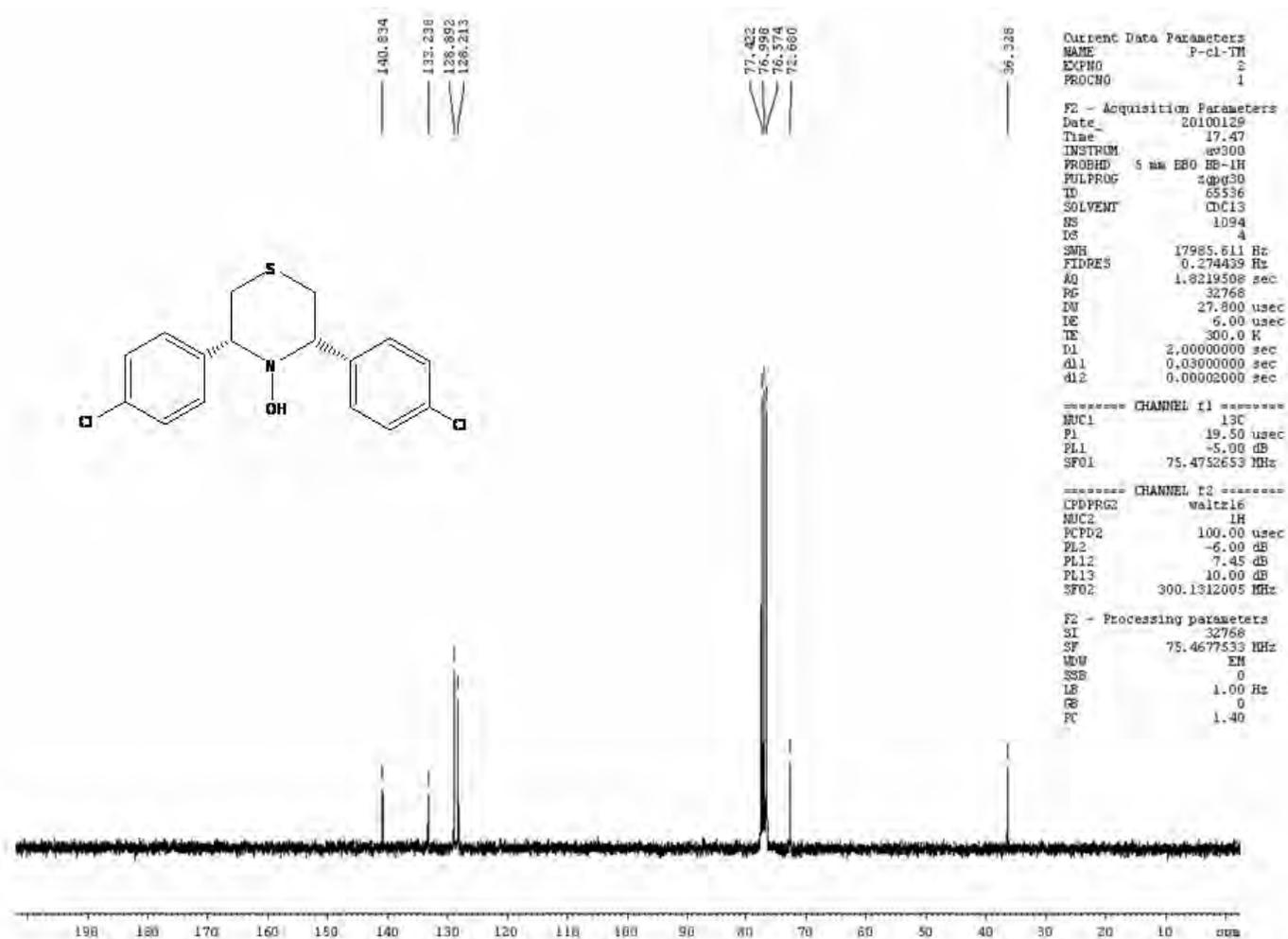
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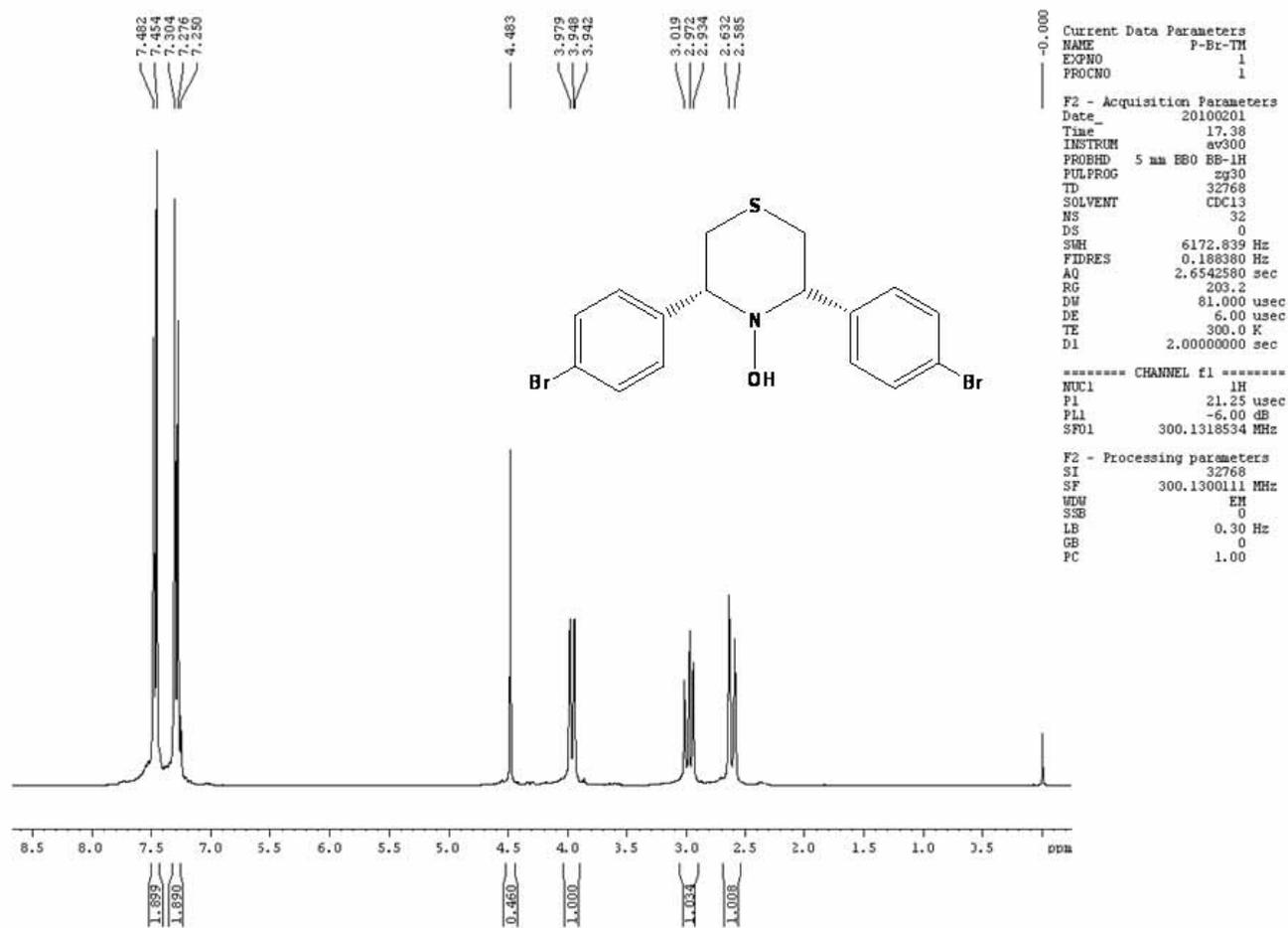
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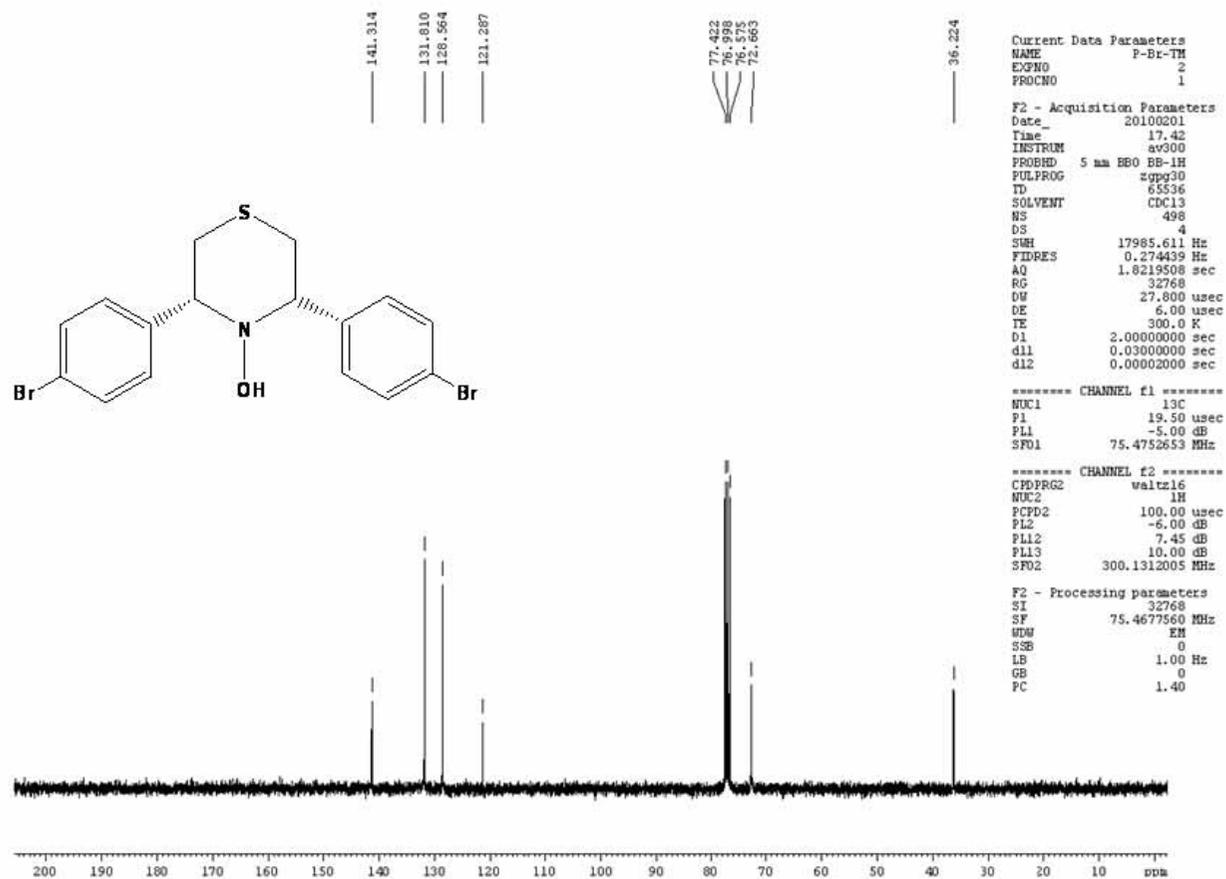
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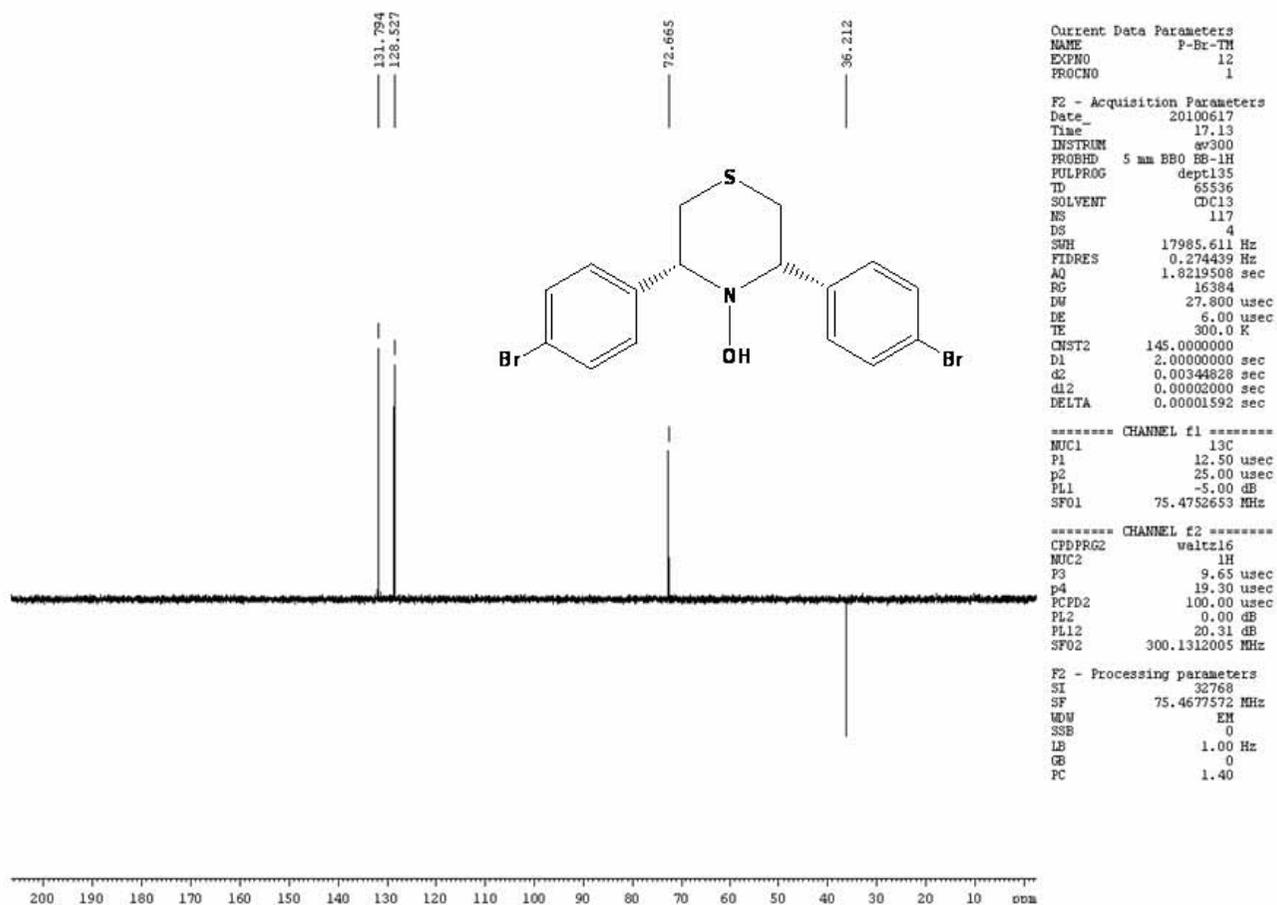
$^1\text{H}$  NMR spectrum of **11c** (300 MHz,  $\text{CDCl}_3$ )



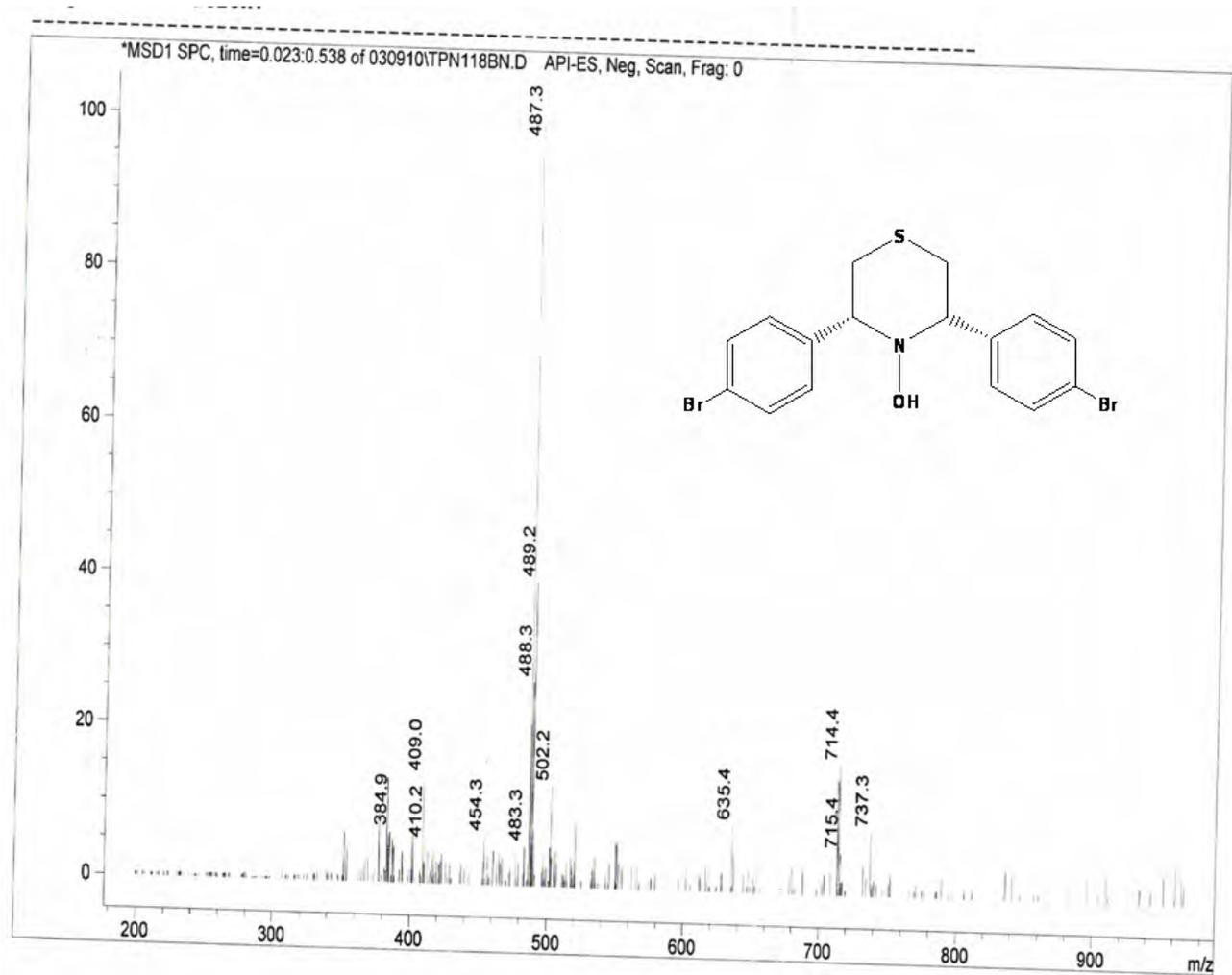
<sup>13</sup>C NMR spectrum of **11c** (75 MHz, CDCl<sub>3</sub>)



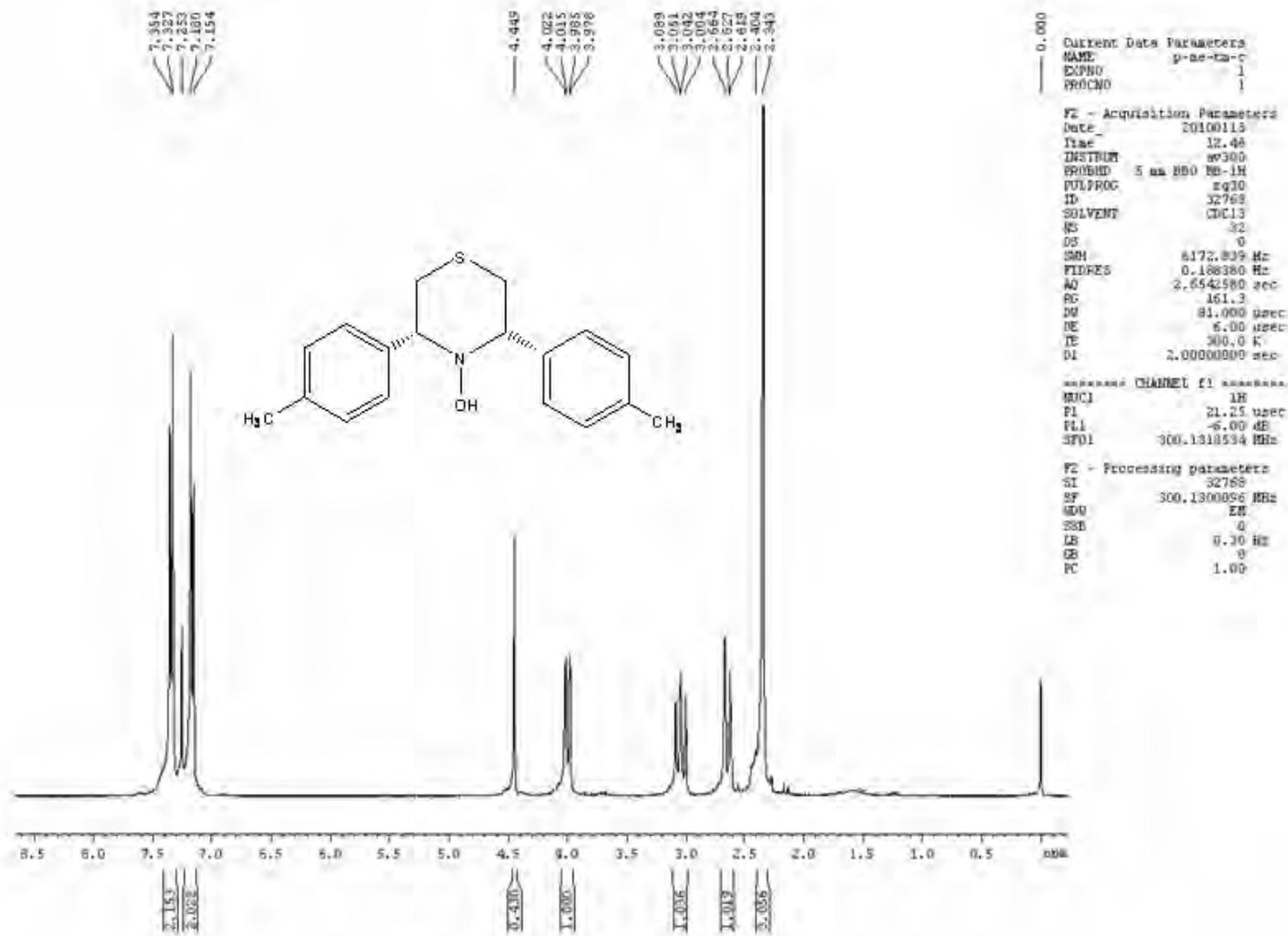
DEPT-135 spectrum of **11c**



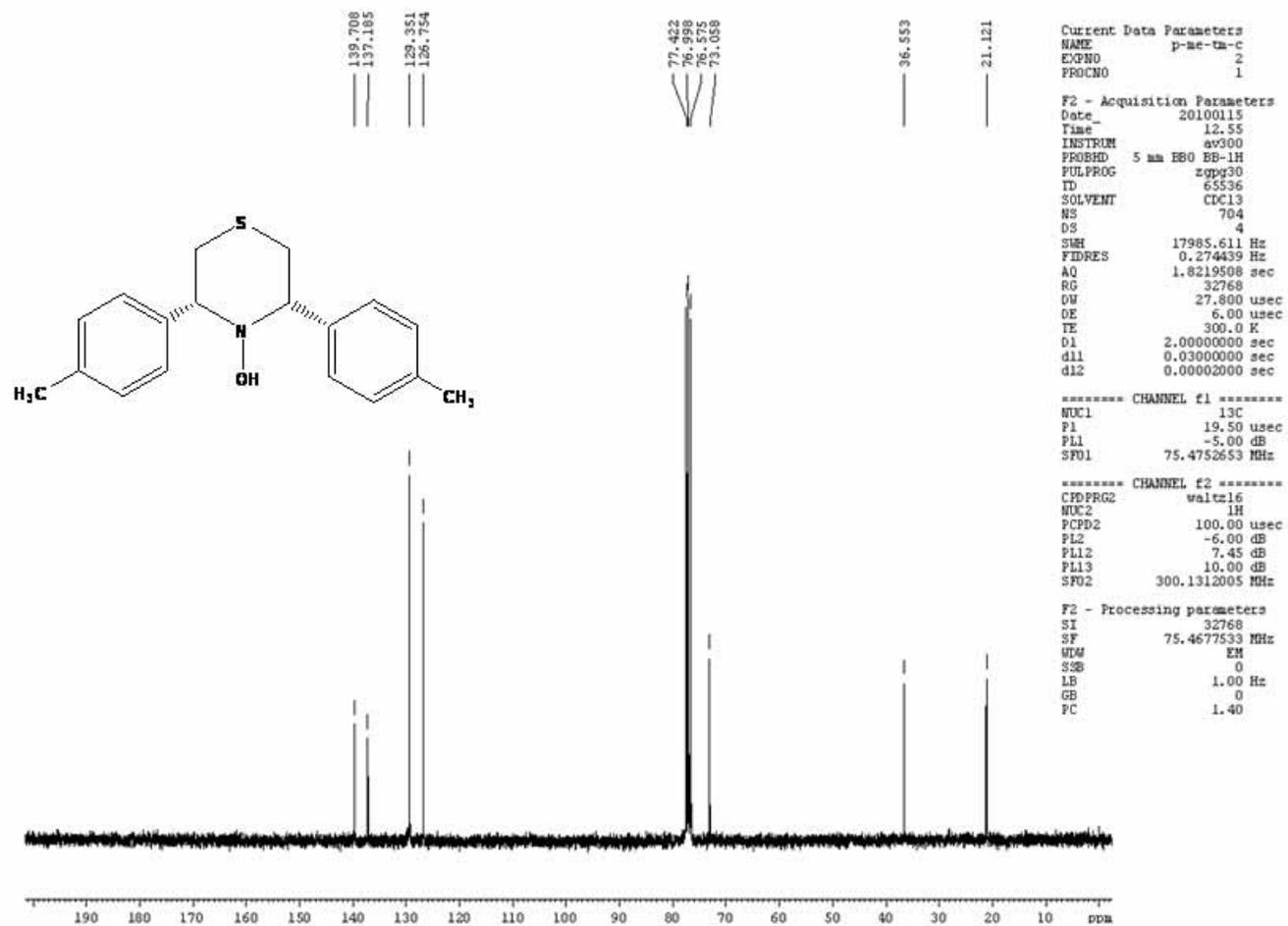
Mass spectrum of **11c**



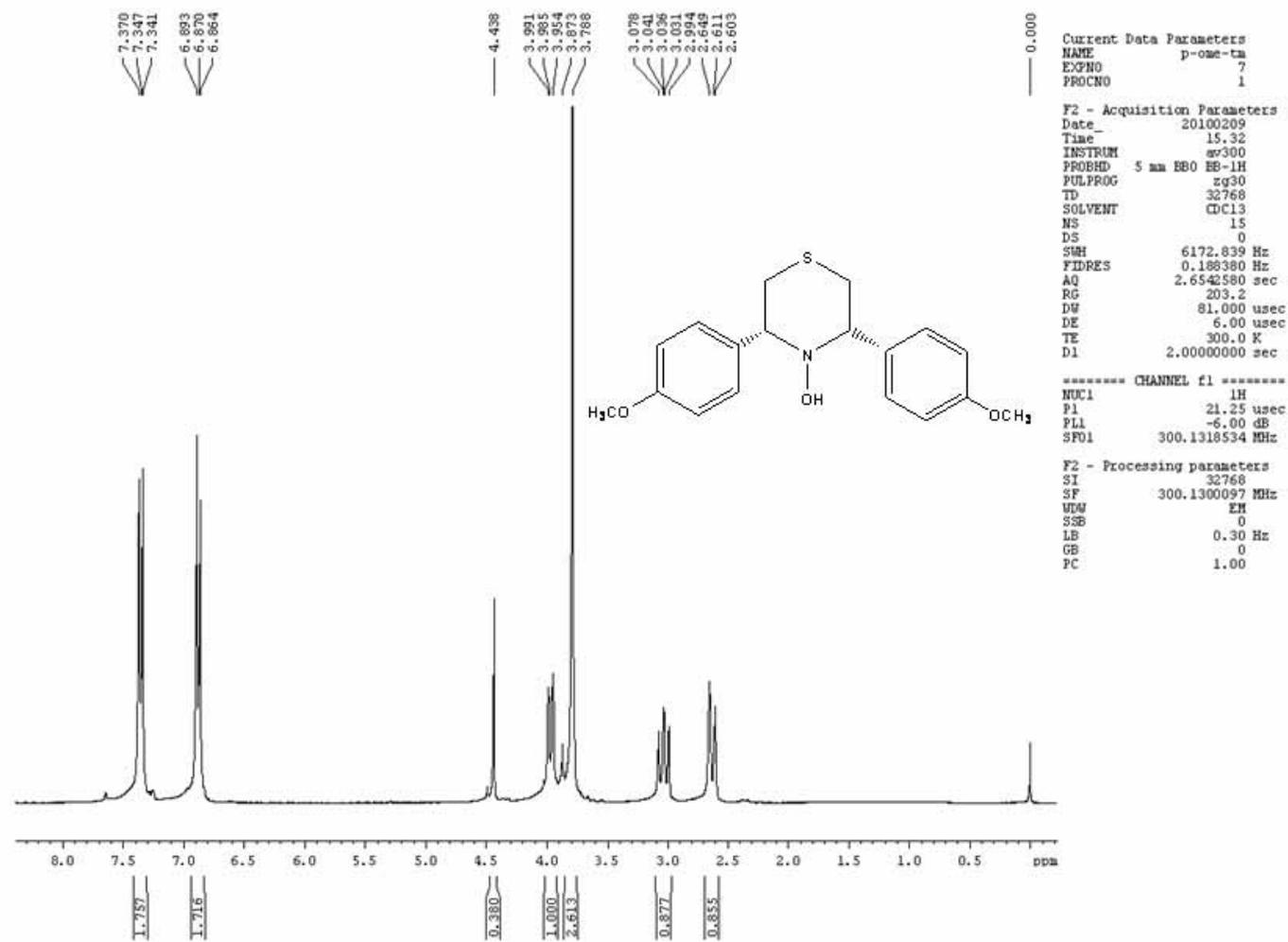
<sup>1</sup>H NMR spectrum of **11d** (300 MHz, CDCl<sub>3</sub>)



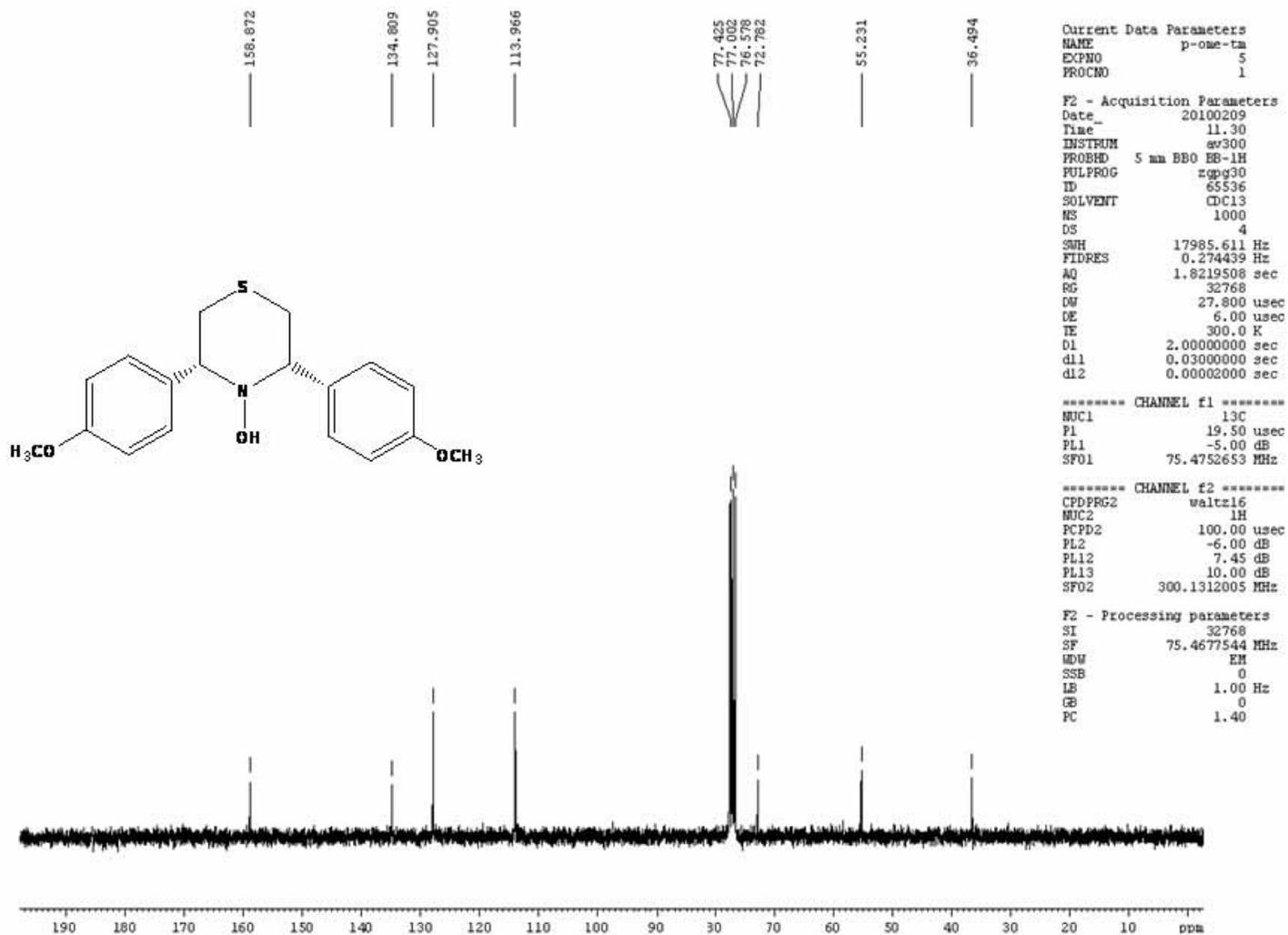
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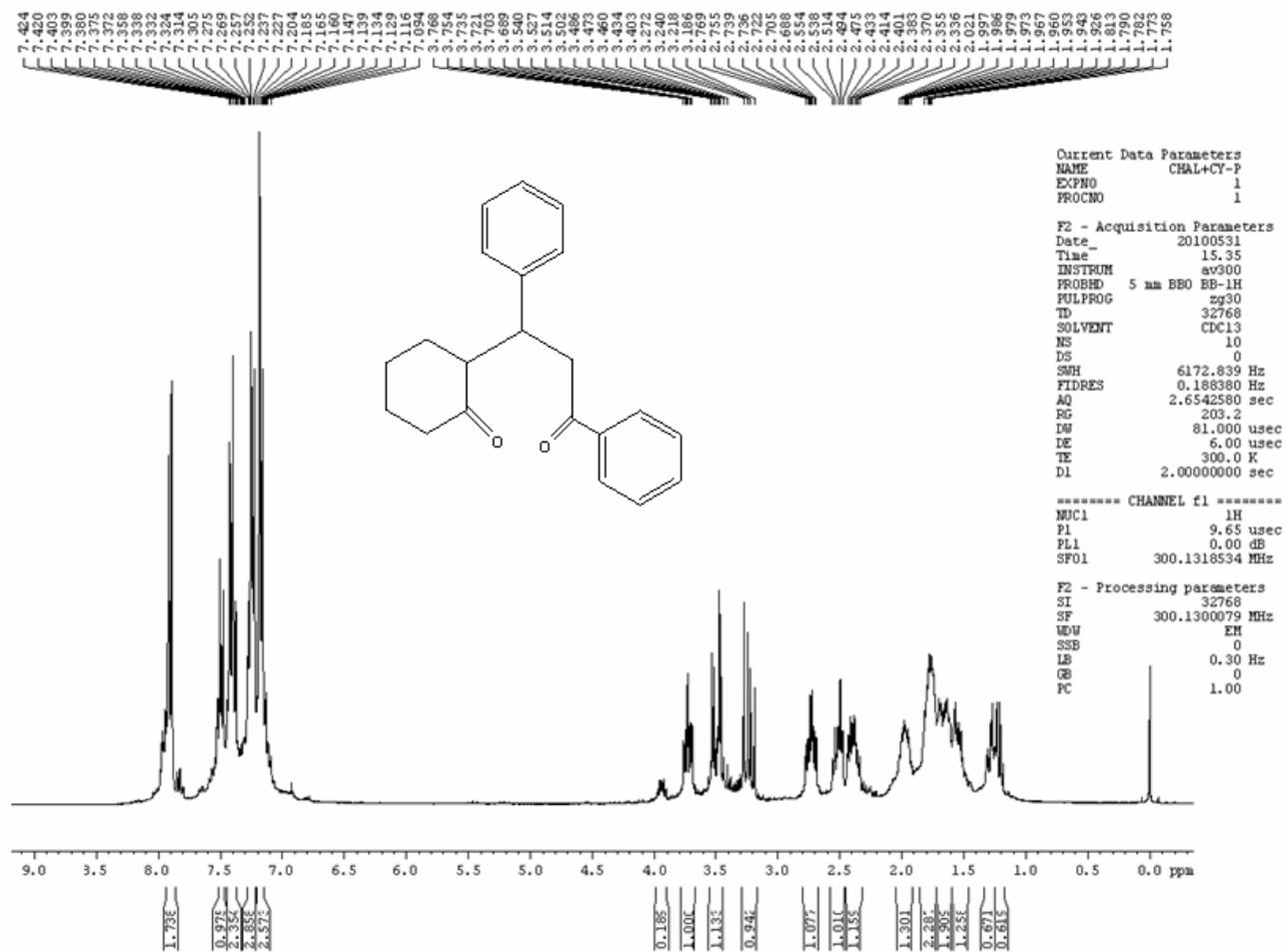
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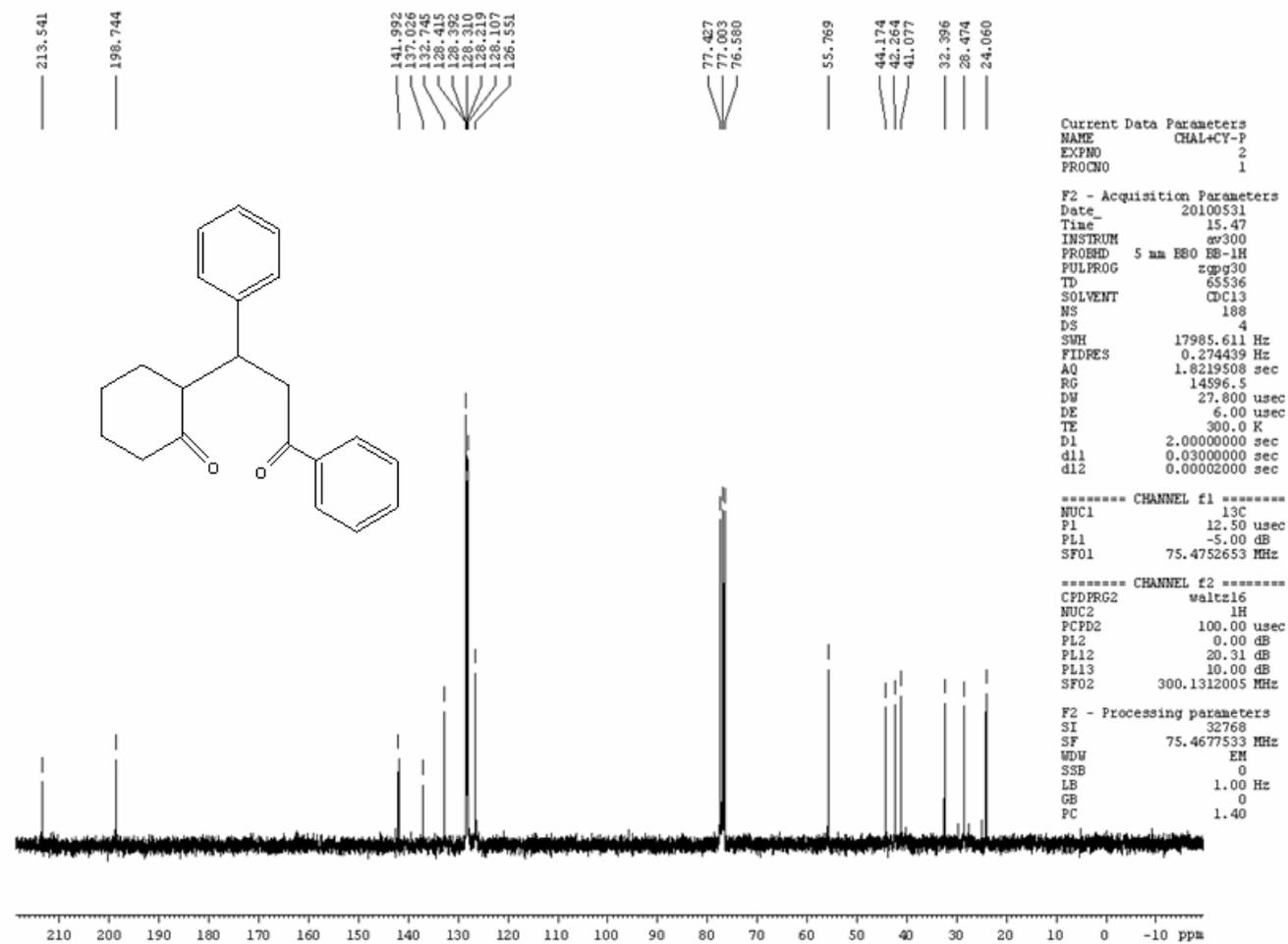
<sup>13</sup>C NMR spectrum of **11e** (75 MHz, CDCl<sub>3</sub>)



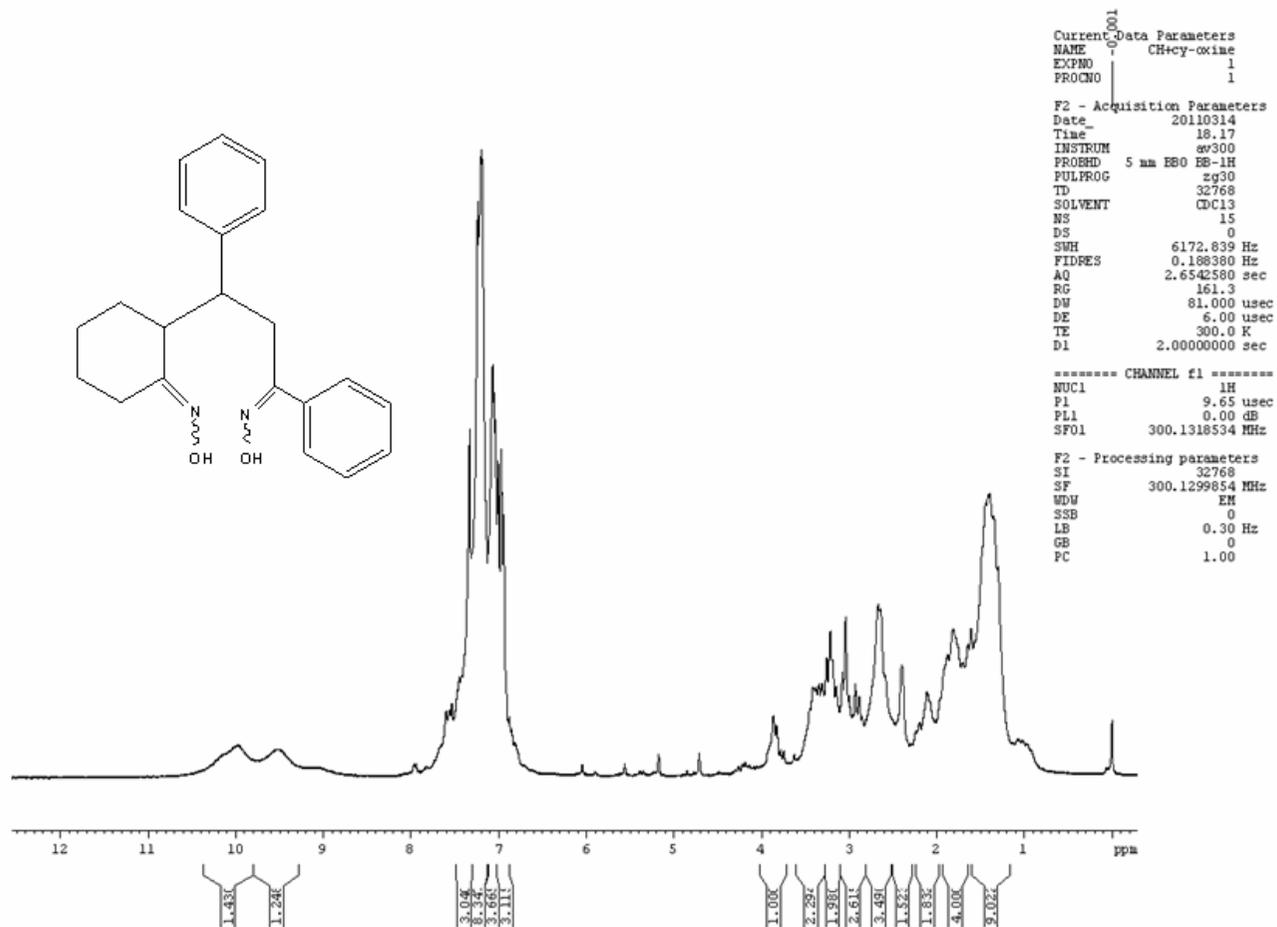
<sup>1</sup>H NMR spectrum of **12a** ketone (300 MHz, CDCl<sub>3</sub>)



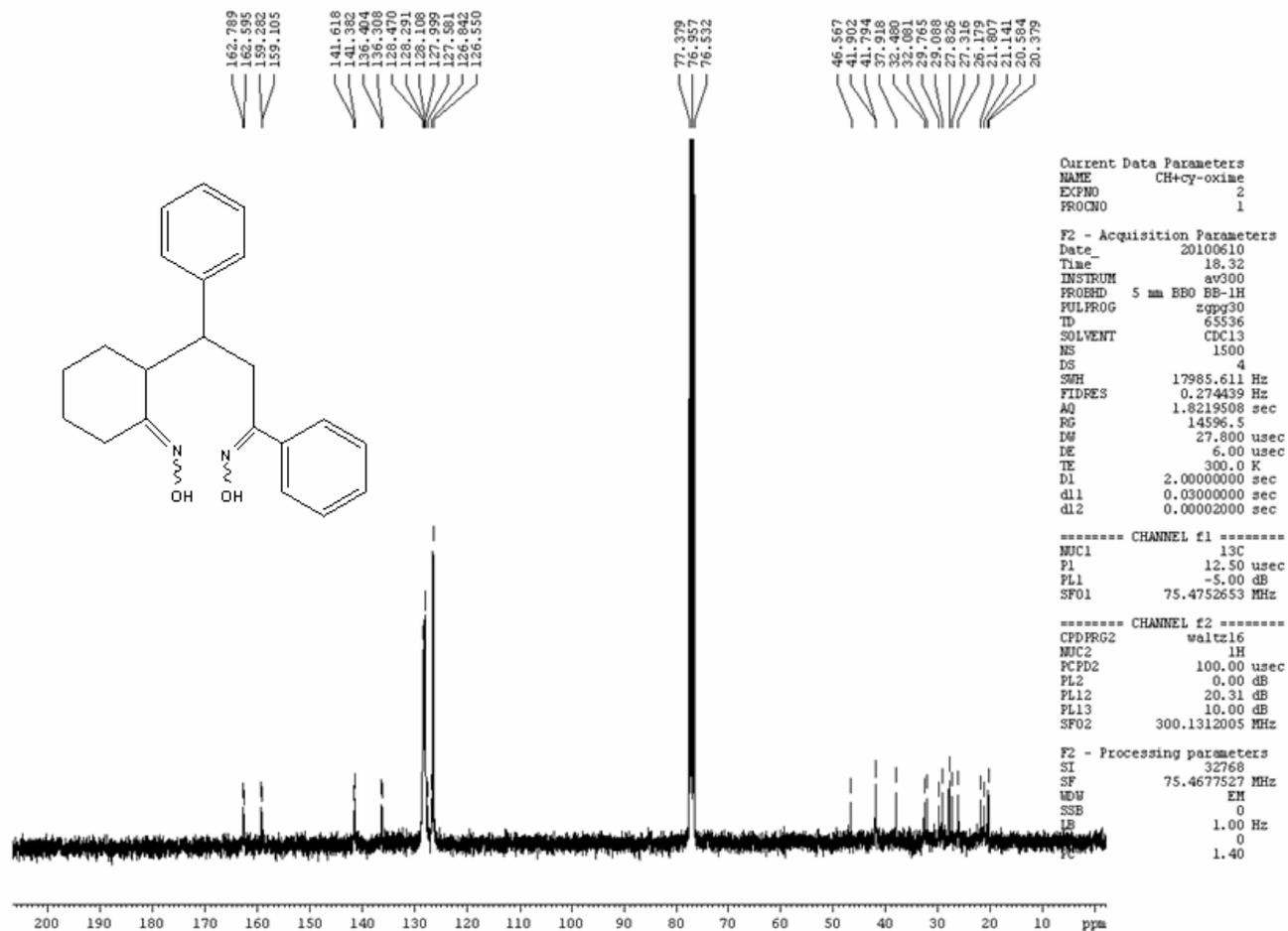
<sup>13</sup>C NMR spectrum of **12a** ketone (75 MHz, CDCl<sub>3</sub>)



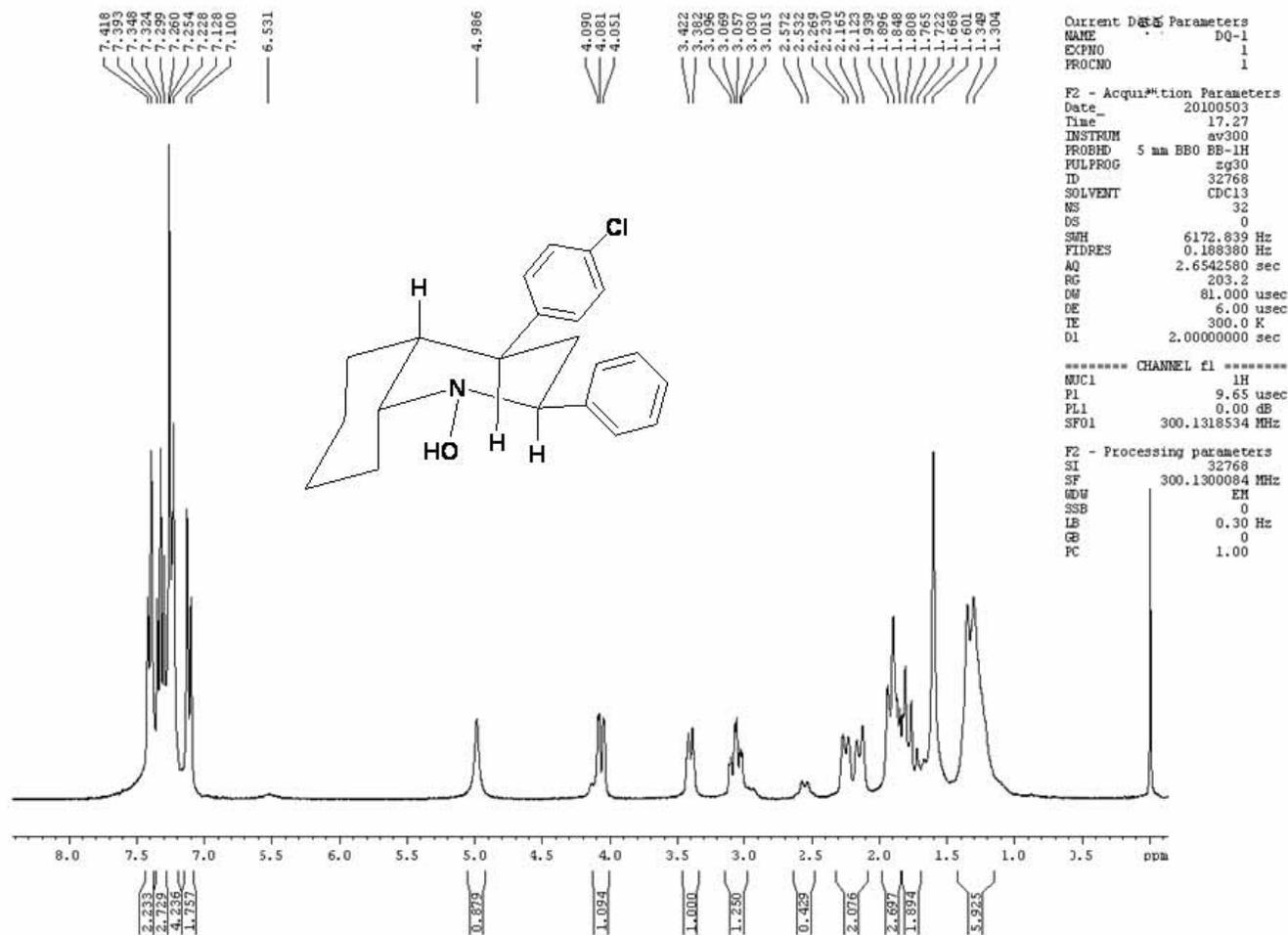
<sup>1</sup>H NMR spectrum of **12a** (300 MHz, CDCl<sub>3</sub>)



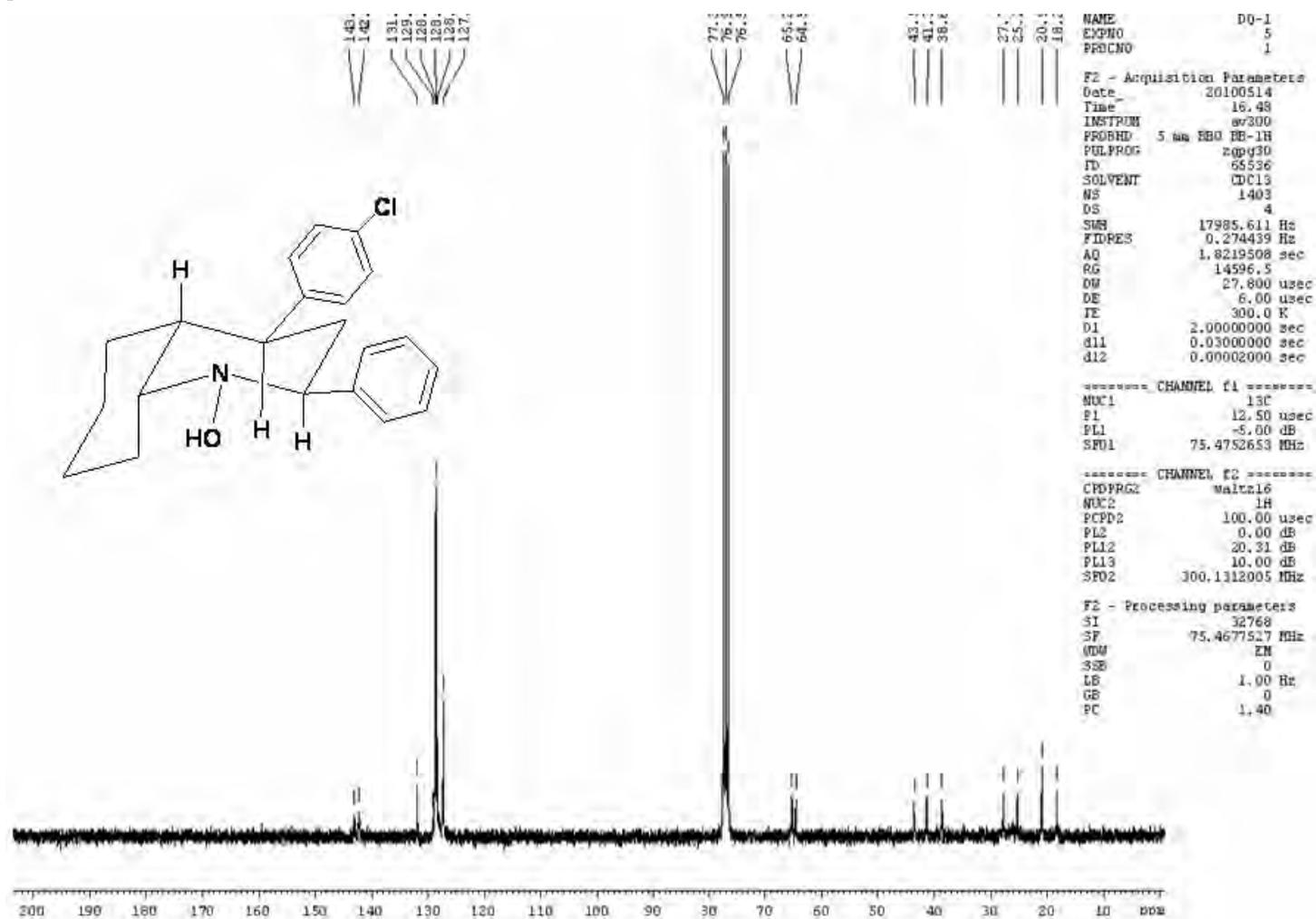
<sup>13</sup>C NMR spectrum of **12a** (75 MHz, CDCl<sub>3</sub>)



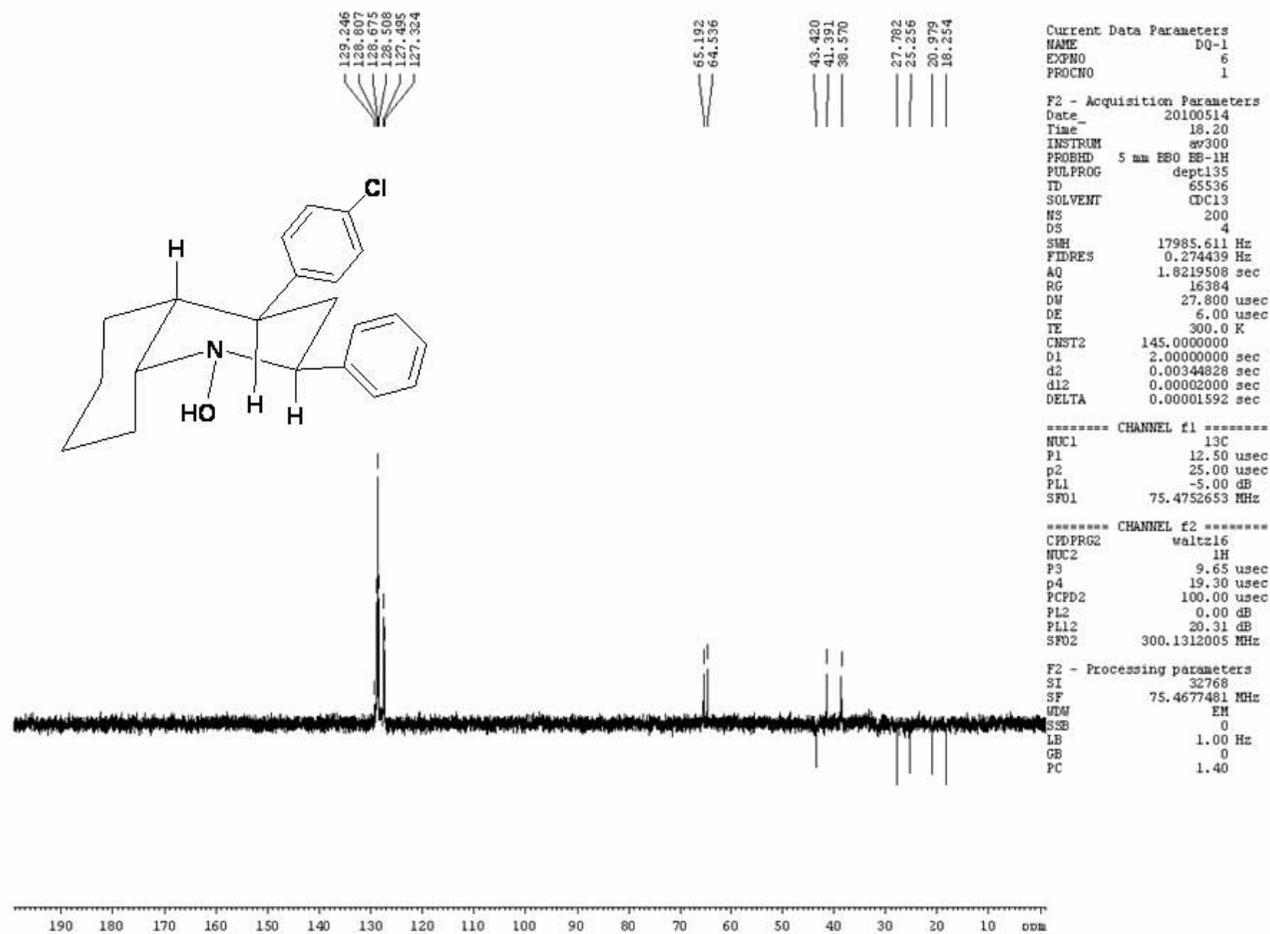
$^1\text{H}$  NMR spectrum of **13b** (300 MHz,  $\text{CDCl}_3$ )



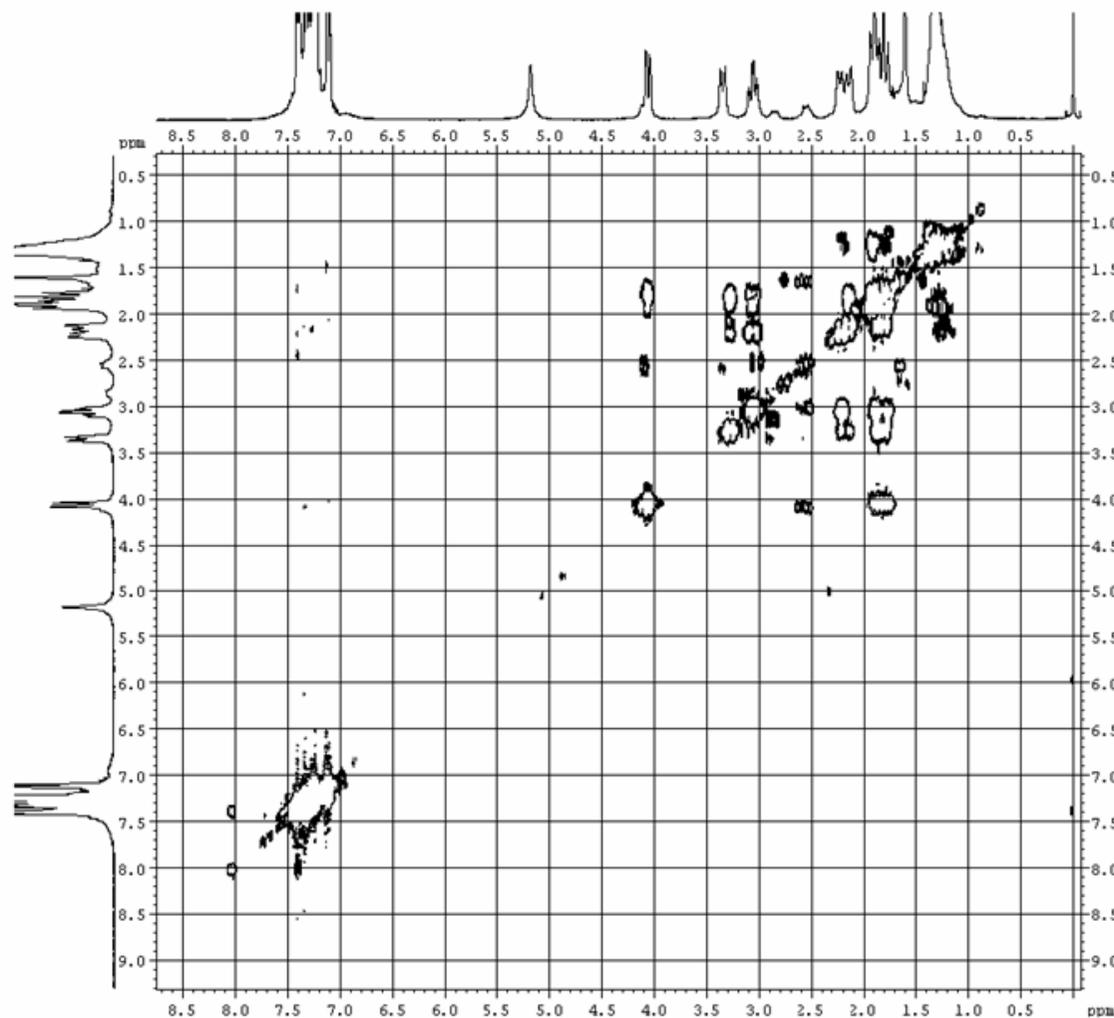
<sup>13</sup>C NMR spectrum of **13b** (75 MHz, CDCl<sub>3</sub>)



DEPT-135 spectrum of **13b**



H-H COSY spectrum of **13b**



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EXPNO 4  
PROCNO 1

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FIDRES 1.956255 Hz  
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DW 124.800 usec  
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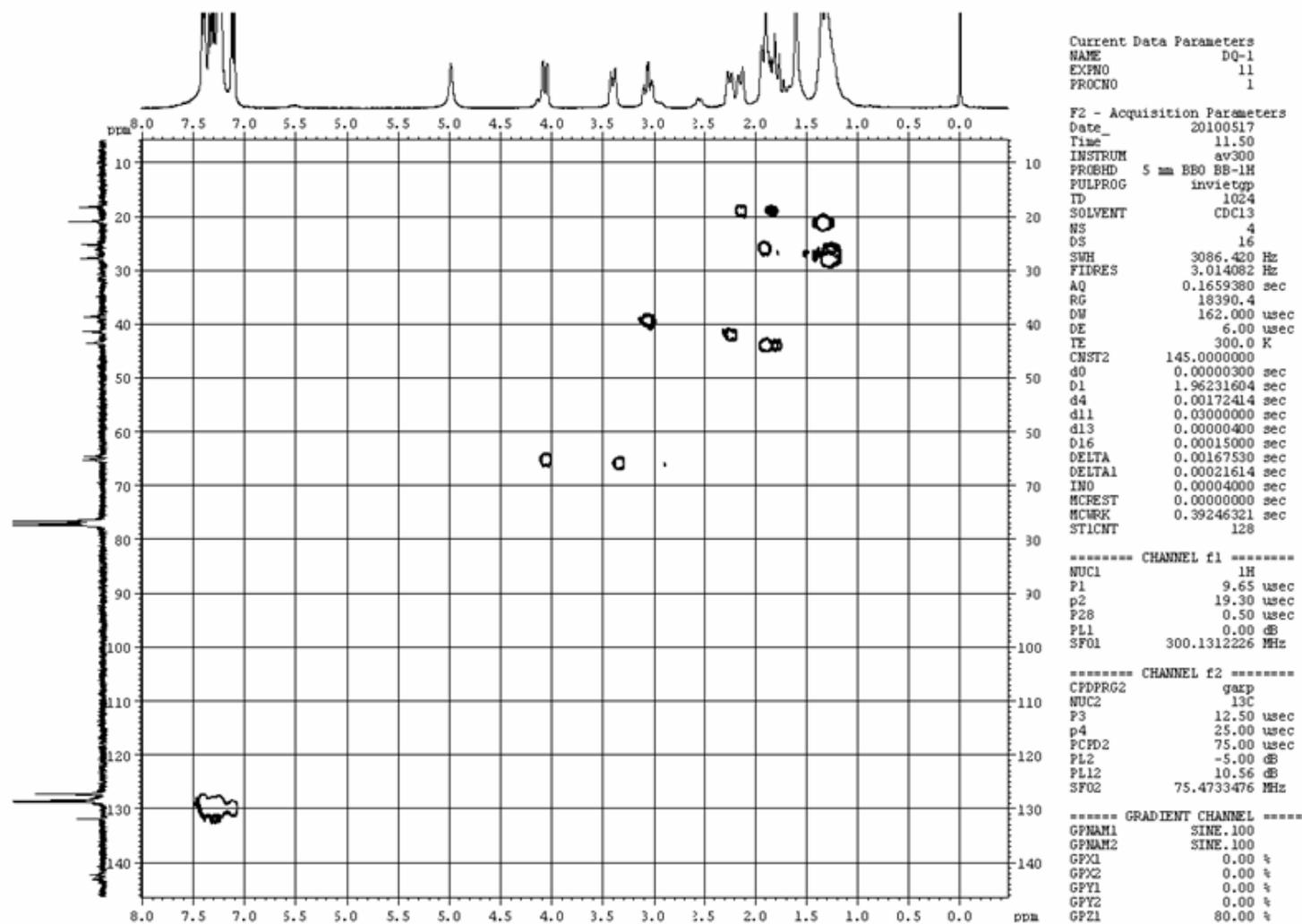
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----- GRADIENT CHANNEL -----  
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GPNAM2 SINE.100  
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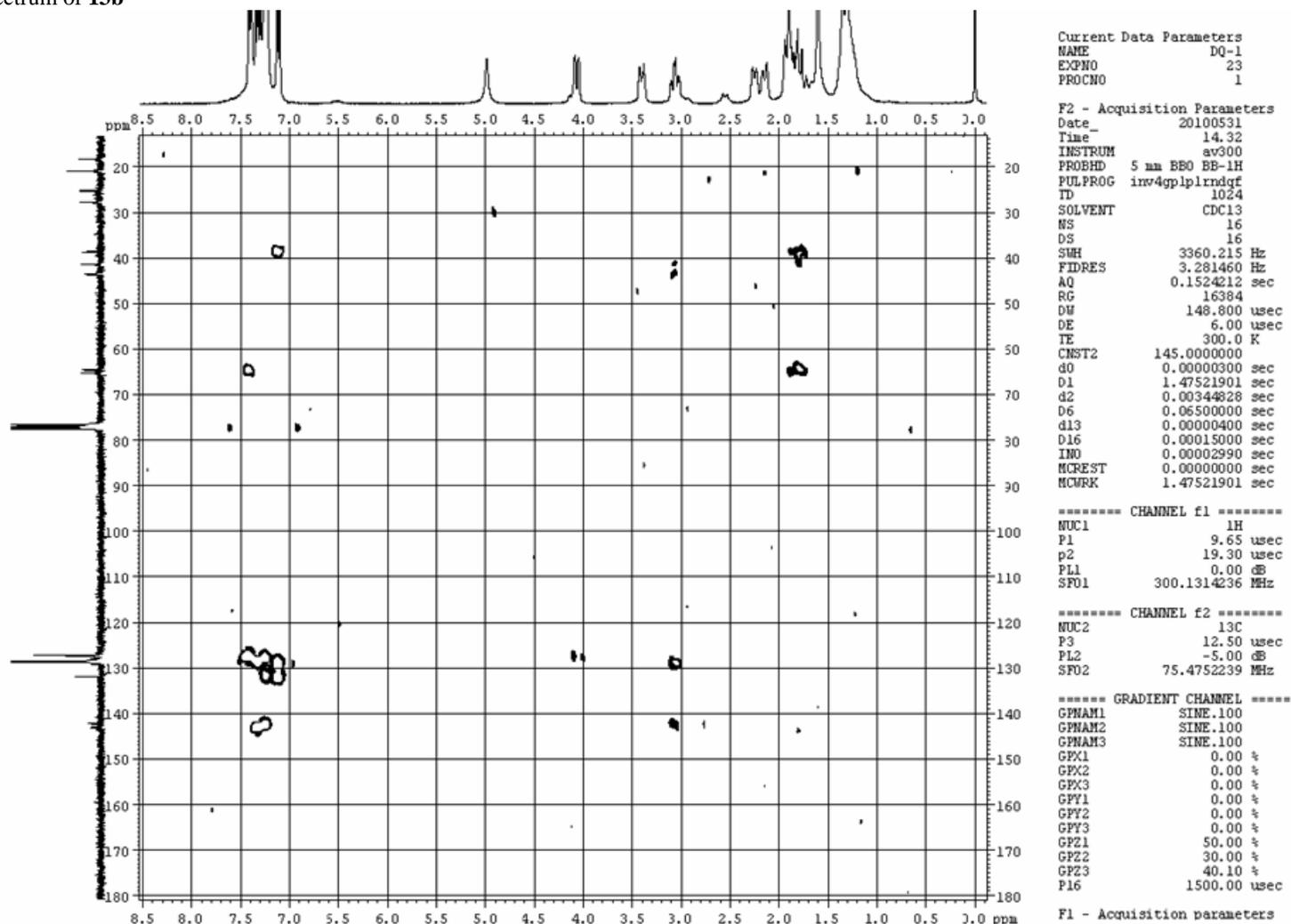
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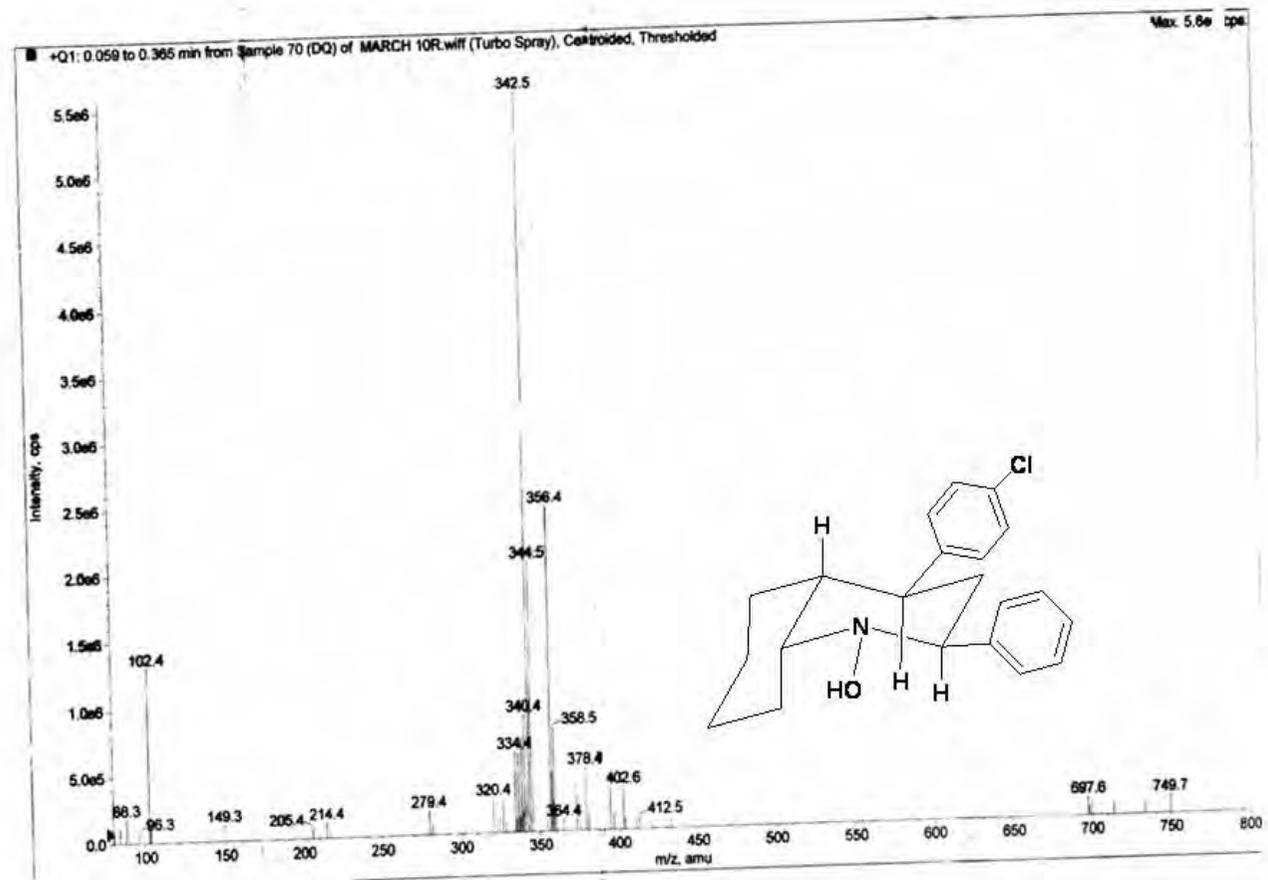
C-H COSY spectrum of **13b**



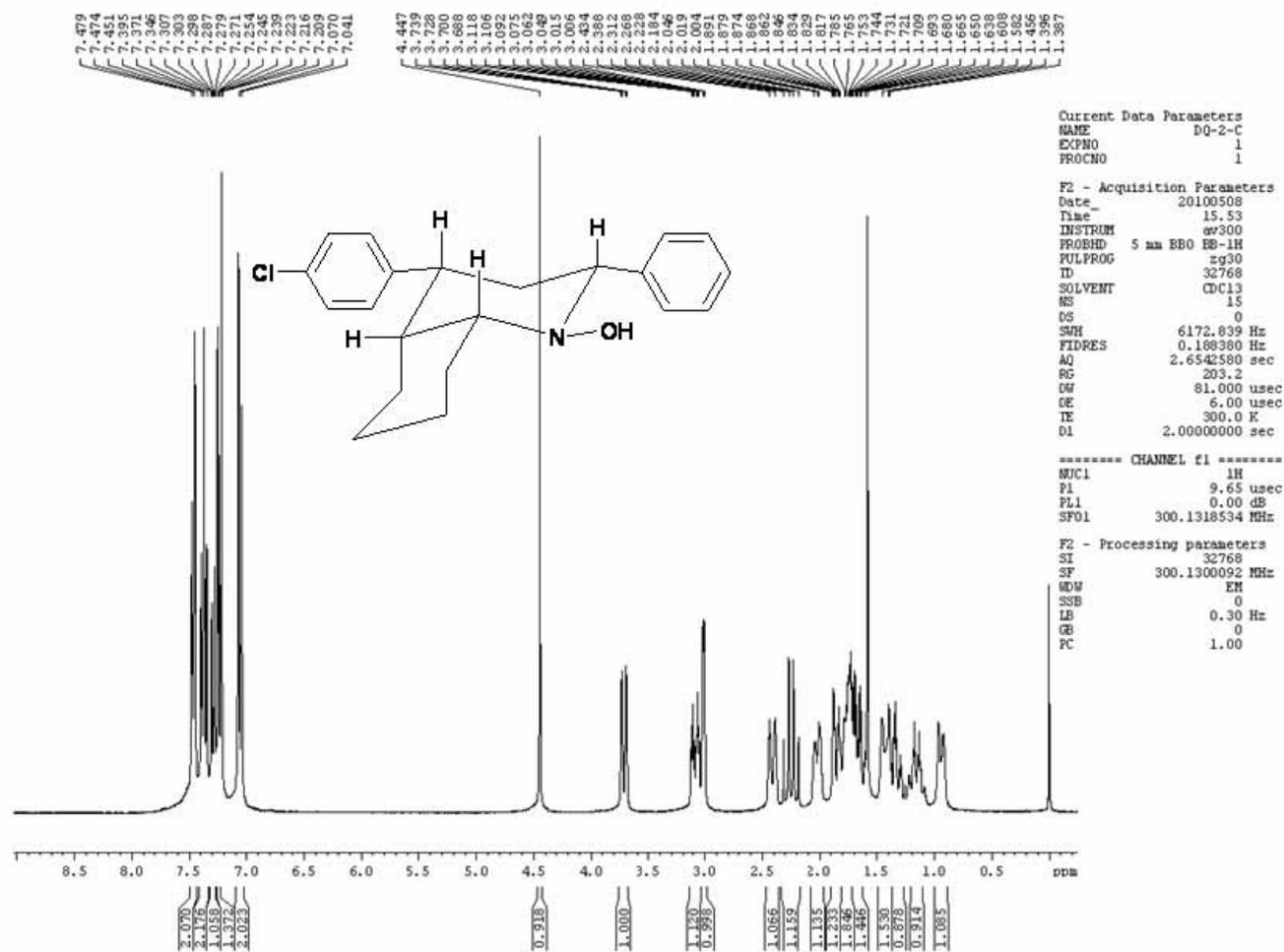
HMBC spectrum of **13b**



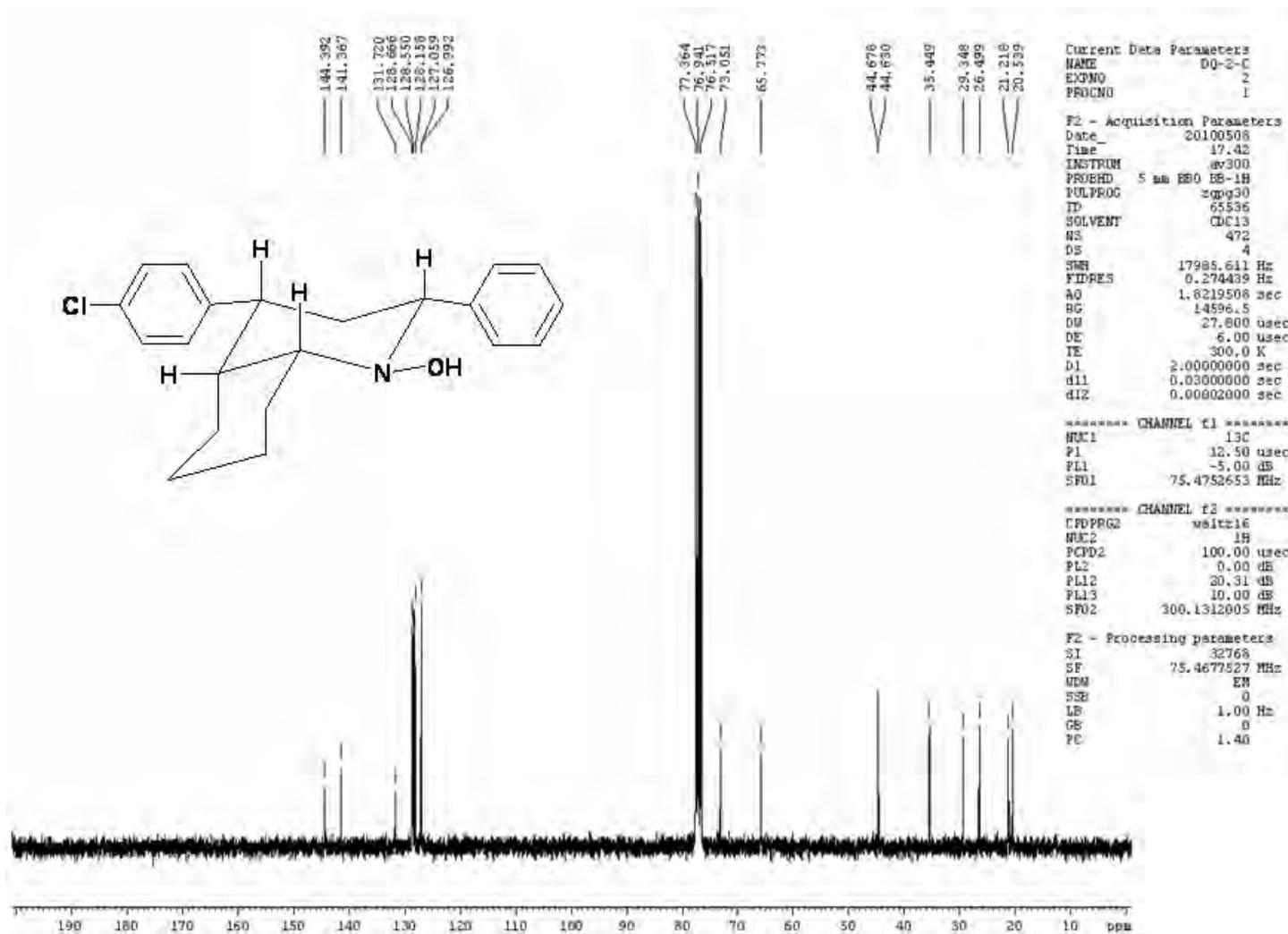
Mass spectrum of **13b**



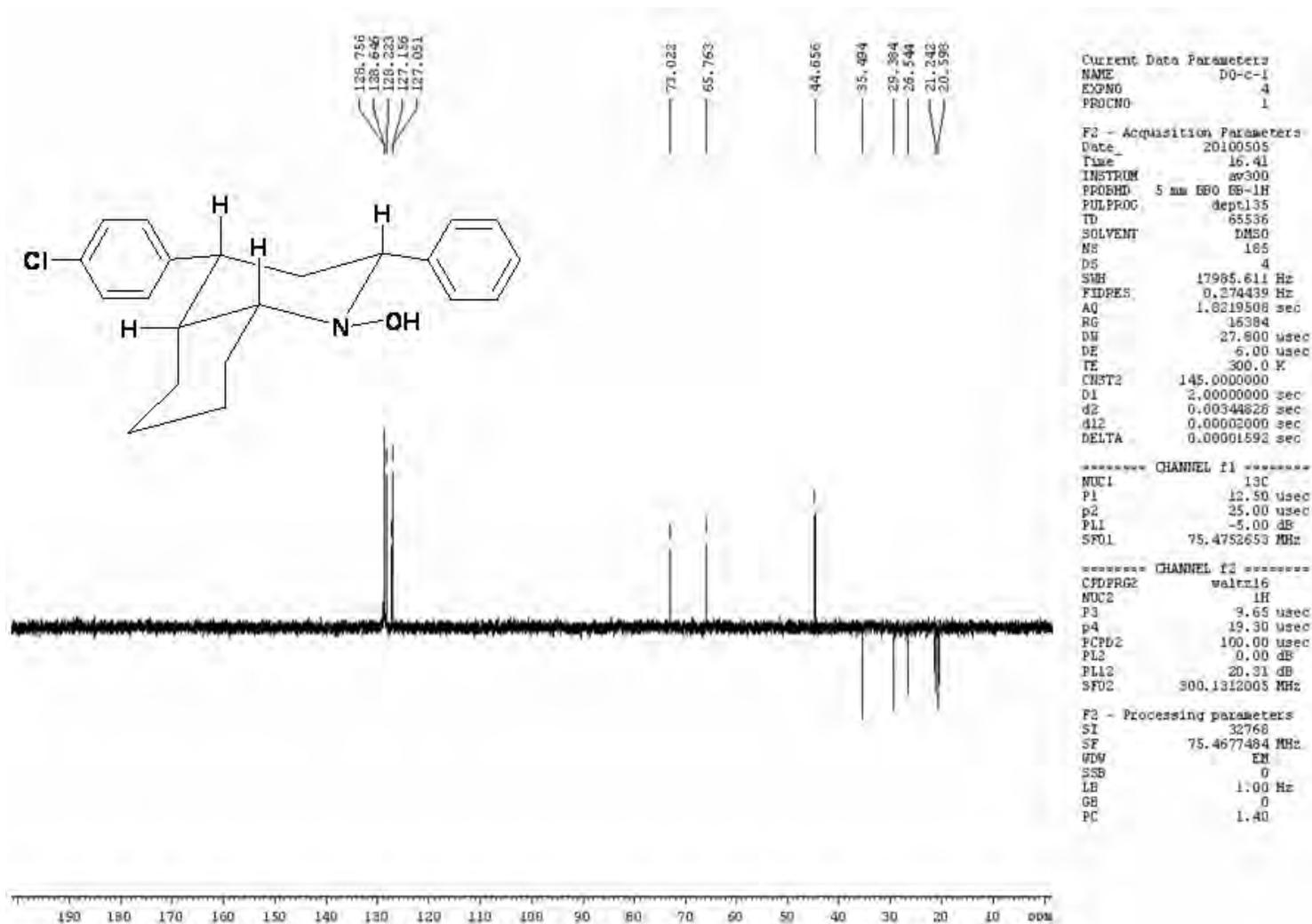
<sup>1</sup>H NMR spectrum of **14b** (300 MHz, CDCl<sub>3</sub>)



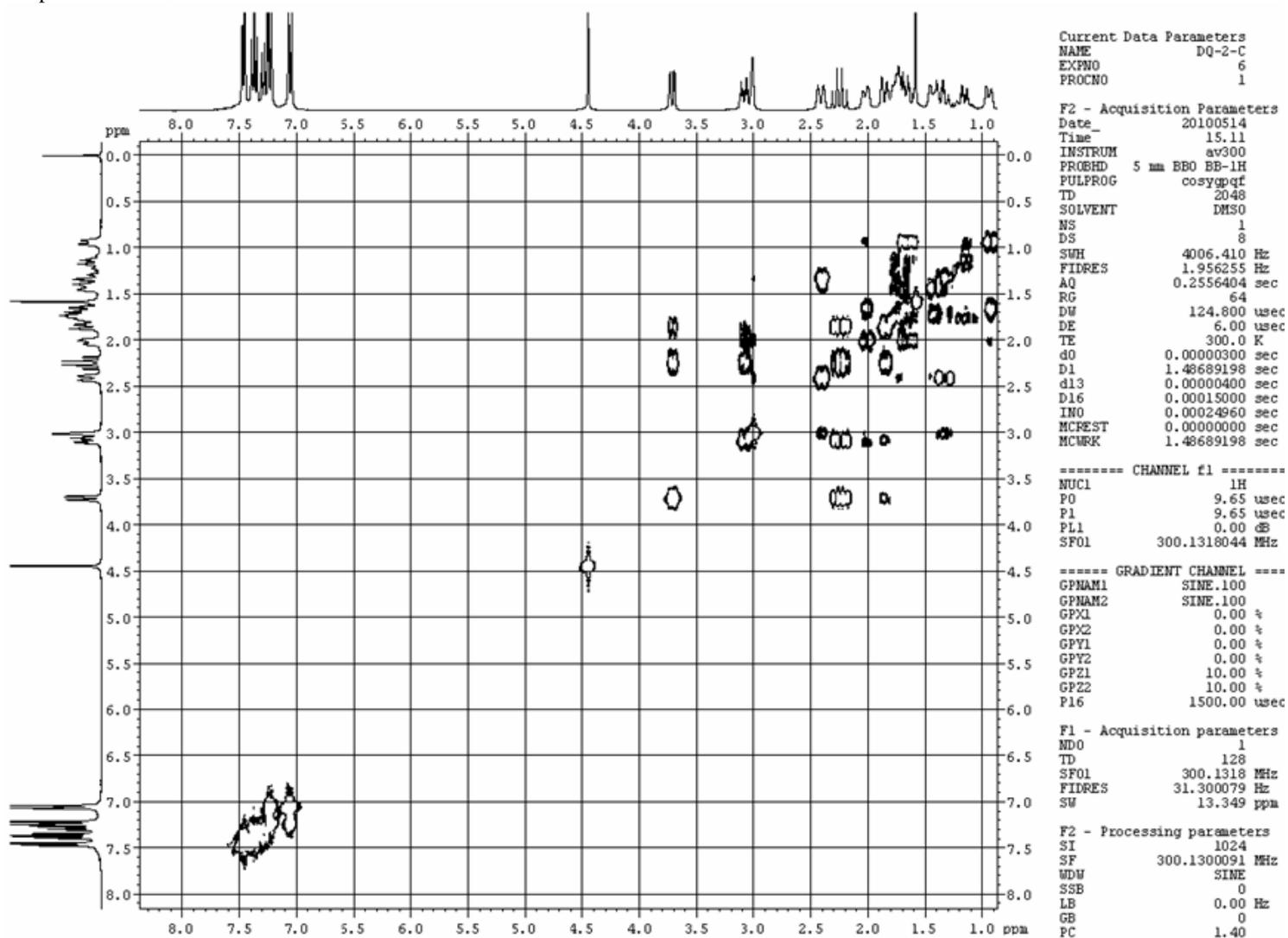
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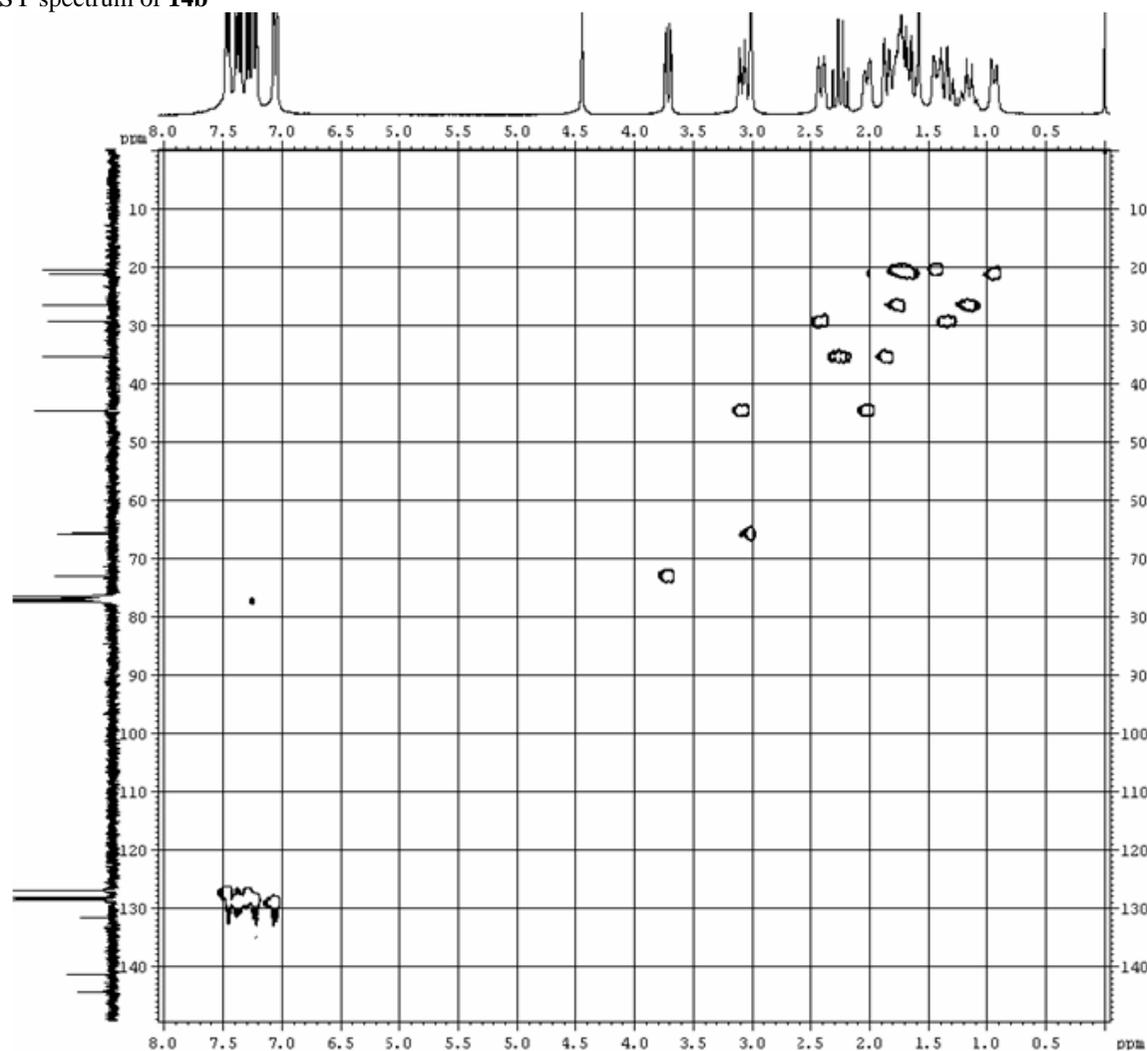
DEPT-135 spectrum of **14b**



H-H COSY spectrum of **14b**



C-H COSY spectrum of **14b**



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PROCNO        1

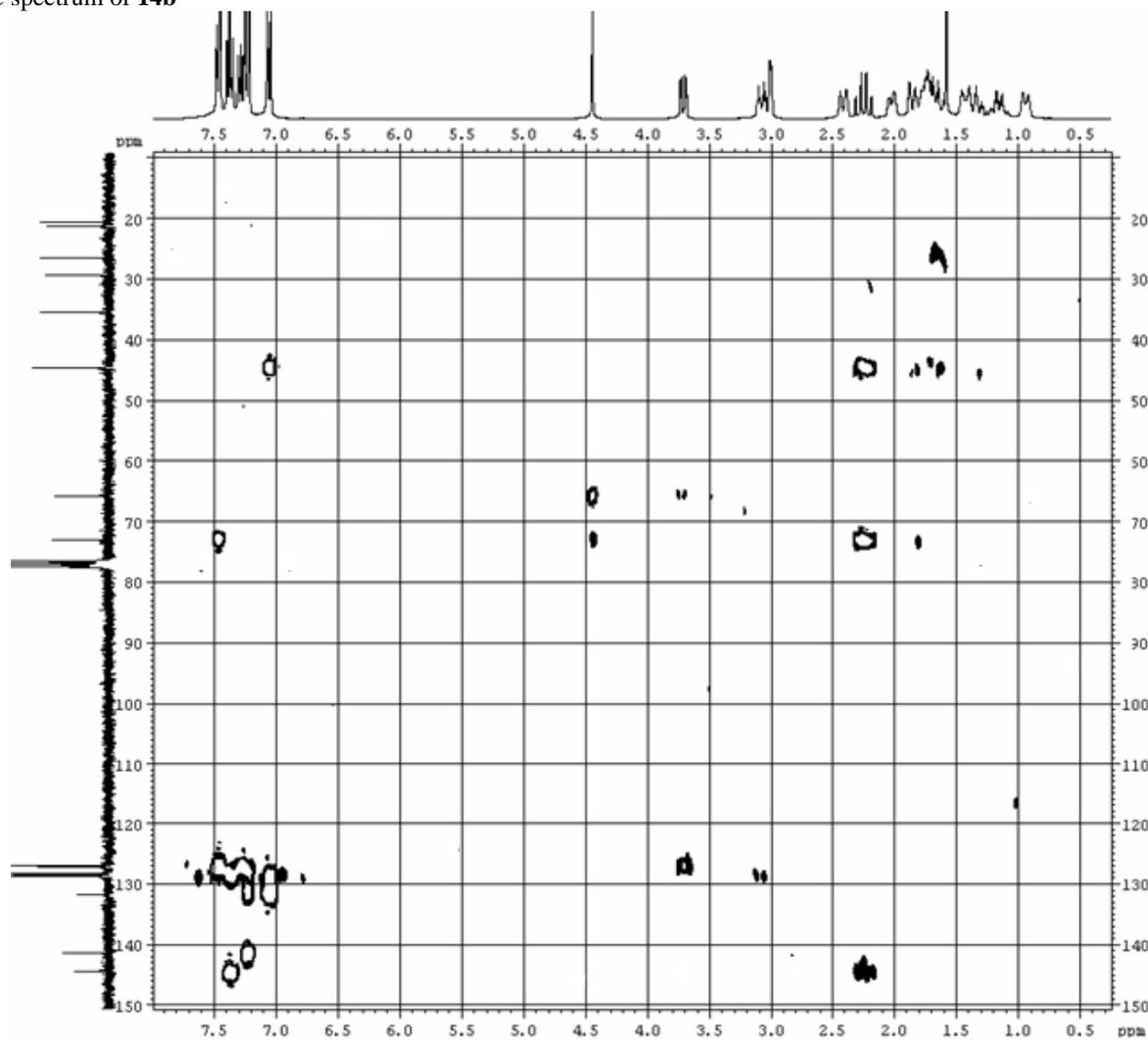
F2 - Acquisition Parameters
Date_         20100517
Time          10.31
INSTRUM       av300
PROBHD        5 mm BBO BB-1H
PULPROG       invietgp
TD            1024
SOLVENT       CDCl3
NS            4
DS            16
SWH           3086.420 Hz
FIDRES        3.014082 Hz
AQ            0.1659380 sec
RG            18390.4
DW            162.000 usec
DE            6.00 usec
TE            300.0 K
CNST2         145.0000000
d0            0.00000300 sec
d1            1.96231604 sec
d4            0.00172414 sec
d11           0.03000000 sec
d13           0.00000400 sec
d16           0.00015000 sec
DELTA         0.00167530 sec
DELTA1        0.00021614 sec
IN0           0.00004000 sec
MCFEST        0.00000000 sec
MCURK         0.39246321 sec
ST1CNT        128

===== CHANNEL f1 =====
NUC1          1H
P1            9.65 usec
p2            19.30 usec
P2S           0.50 usec
PL1           0.00 dB
SF01          300.1312226 MHz

===== CHANNEL f2 =====
CPDPRG2       garp
NUC2          13C
P3            12.50 usec
p4            25.00 usec
PCPD2         75.00 usec
PL2           -5.00 dB
PL12          10.56 dB
SF02          75.4733476 MHz

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPX1          0.00 %
GPX2          0.00 %
GPY1          0.00 %
GPY2          0.00 %
GPZ1          80.00 %
GPZ2          20.10 %
P16           1500.00 usec
```

HMBC spectrum of **14b**



Current Data Parameters  
NAME DQ-2-C  
EXPNO 3  
PROCNO 1

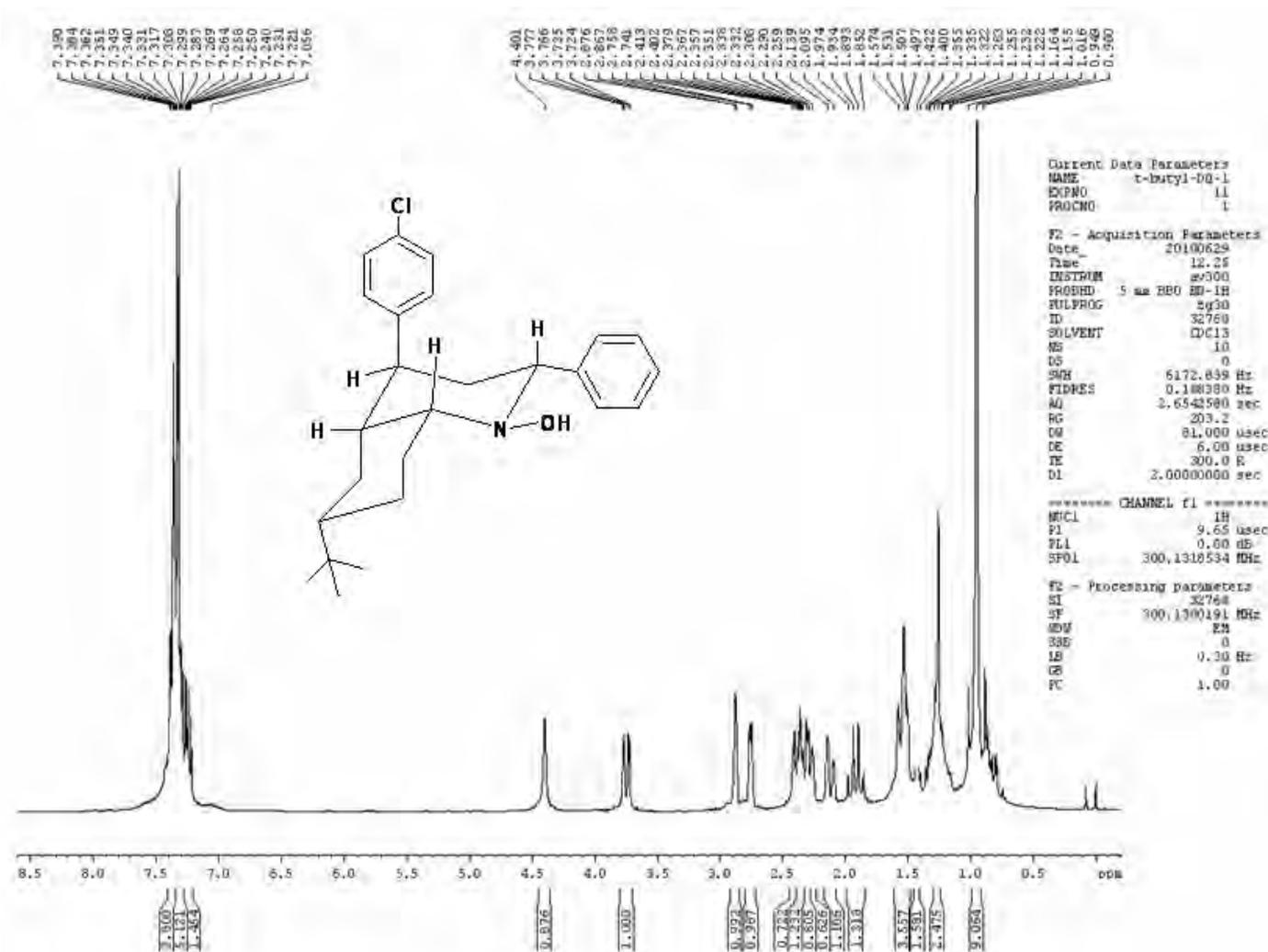
F2 - Acquisition Parameters  
Date\_ 20100514  
Time 14.43  
INSTRUM av300  
PROBHD 5 mm BBO BB-1H  
PULPROG inv4gp1plrndqf  
TD 1024  
SOLVENT CDCl3  
NS 8  
DS 16  
SWH 3360.215 Hz  
FIDRES 3.281460 Hz  
AQ 0.1524212 sec  
RG 16384  
OW 148.800 usec  
OE 6.00 usec  
TE 300.0 K  
CNST2 145.0000000  
d0 0.0000000 sec  
d1 1.47521901 sec  
d2 0.00344628 sec  
d6 0.06500000 sec  
d13 0.00000400 sec  
d16 0.00015000 sec  
IN0 0.00002990 sec  
MCREST 0.00000000 sec  
MCRMK 1.47521901 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
NUC1 1H  
P1 9.65 usec  
p2 19.30 usec  
PL1 0.00 dB  
SF01 300.1314236 MHz

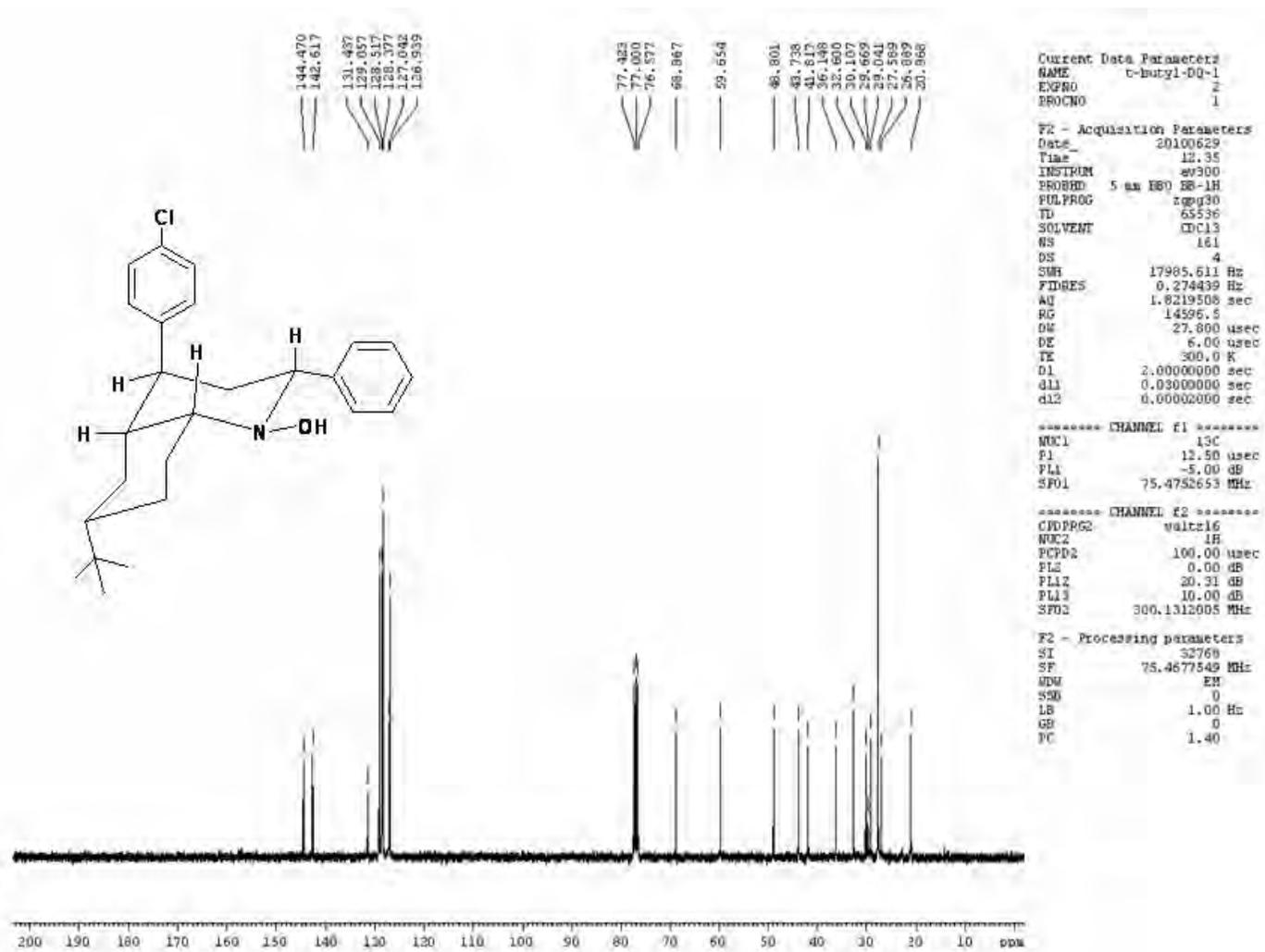
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
NUC2 13C  
P3 12.50 usec  
PL2 -5.00 dB  
SF02 75.4752239 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
GPNAM1 SINE.100  
GPNAM2 SINE.100  
GPNAM3 SINE.100  
GFX1 0.00 %  
GFX2 0.00 %  
GFX3 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPY3 0.00 %  
GPZ1 50.00 %  
GPZ2 30.00 %  
GPZ3 40.10 %  
P16 1500.00 usec

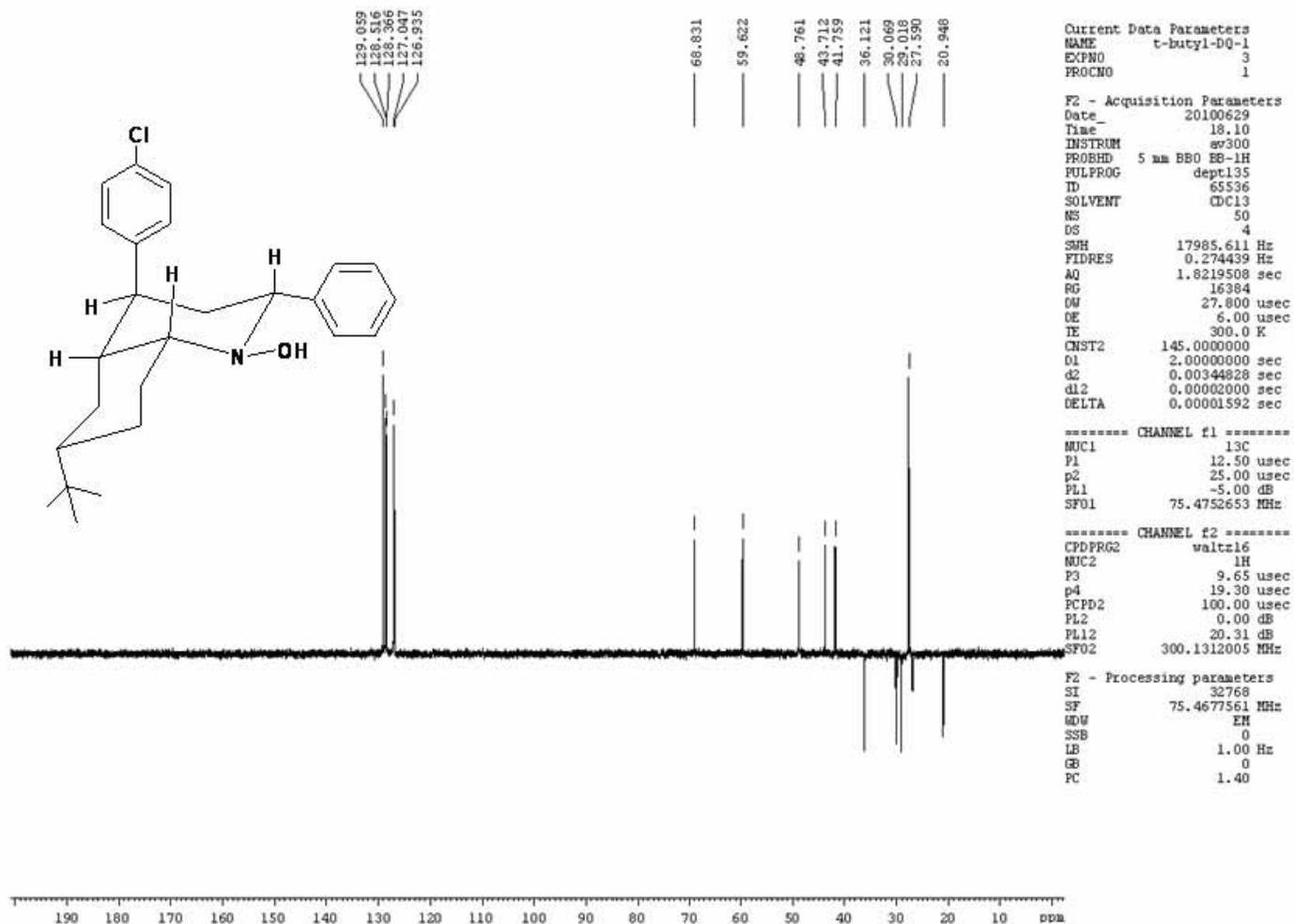
$^1\text{H}$  NMR spectrum of **15c** (300 MHz,  $\text{CDCl}_3$ )



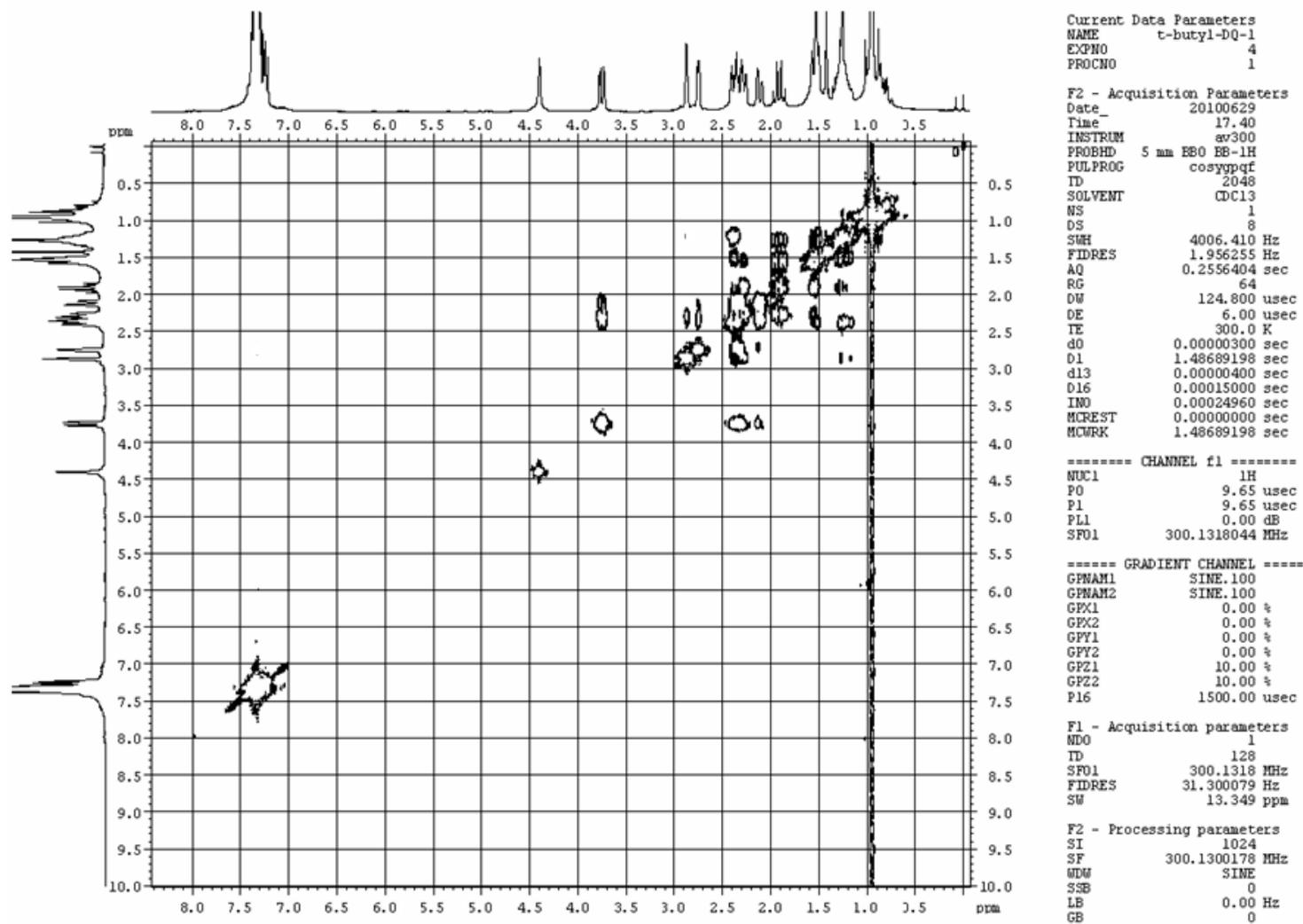
<sup>13</sup>C NMR spectrum of **14b** (75 MHz, CDCl<sub>3</sub>)



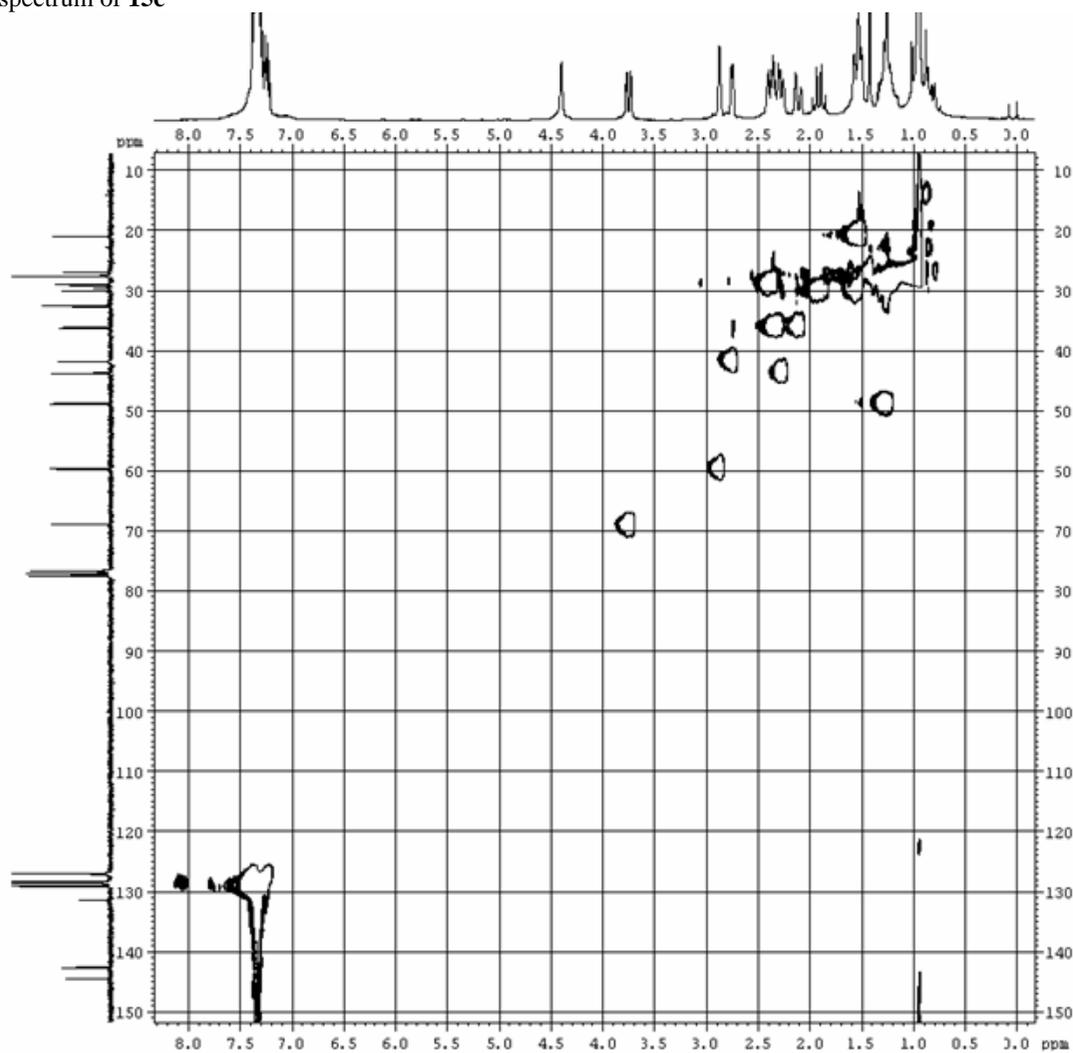
DEPT-135 spectrum of **15c**



H-H COSY spectrum of **15c**



C-H COSY spectrum of **15c**



```
Current Data Parameters
NAME      t-butyl-D0-1
EXPNO    5
PROCNO   1

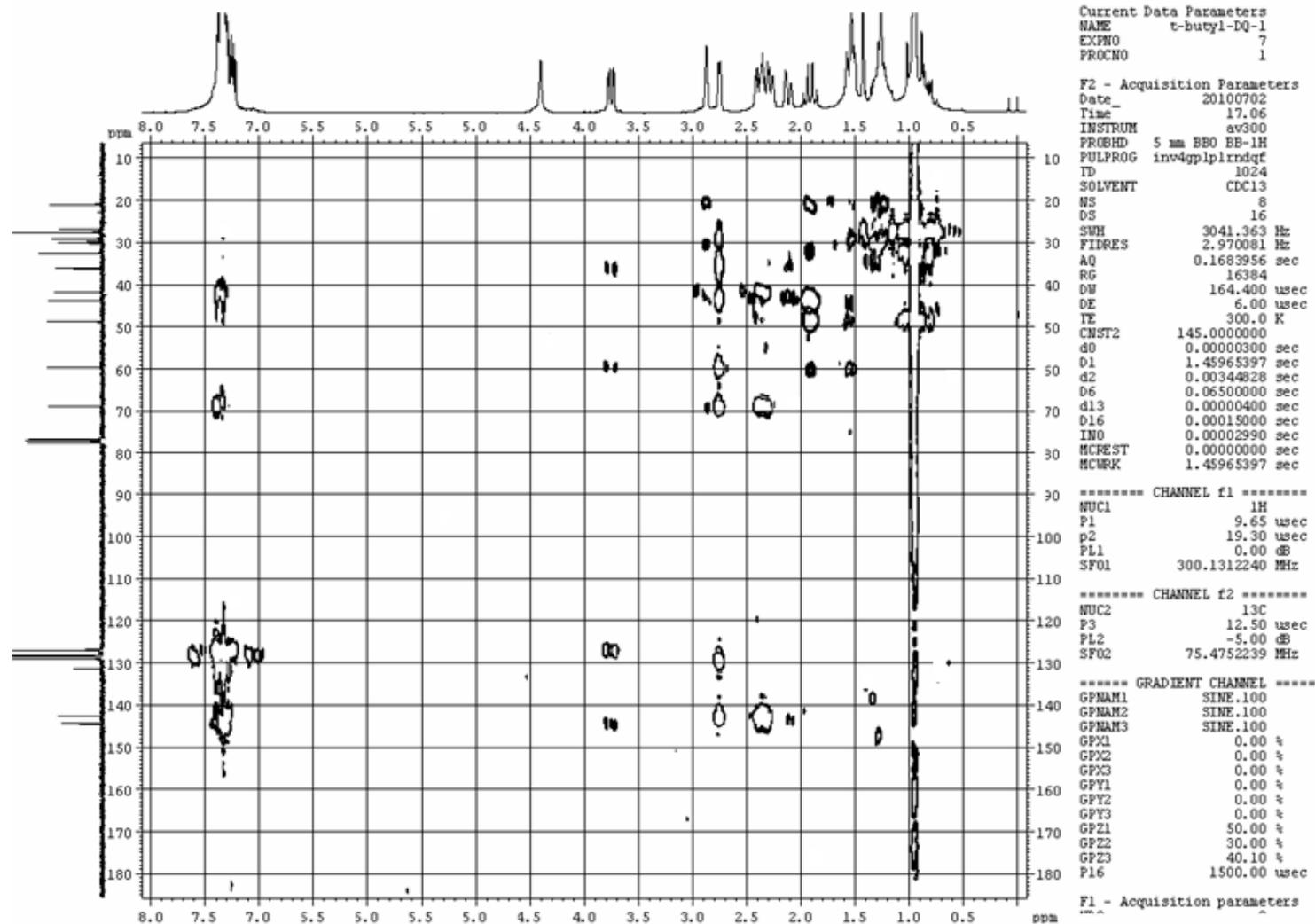
F2 - Acquisition Parameters
Date_    20100629
Time     17.48
INSTRUM  av300
PROBHD   5 mm BBO BB-1H
PULPROG  invietyp
TD       1024
SOLVENT  CDCl3
NS       4
DS       16
SWH      3086.420 Hz
FIDRES   3.014082 Hz
AQ       0.1659380 sec
RG       18390.4
DW       162.000 usec
DE       6.00 usec
TE       300.0 K
CNST2    145.0000000
d0       0.00000300 sec
D1       1.96231604 sec
d4       0.00172414 sec
d11      0.03000000 sec
d13      0.00000400 sec
D16      0.00015000 sec
DELTA    0.00167530 sec
DELTA1   0.00021614 sec
IN0      0.00004000 sec
HCREST   0.00000000 sec
HCHWK    0.39246321 sec
STICNT   128

----- CHANNEL f1 -----
NUC1     1H
P1       9.65 usec
p2       19.30 usec
P28      0.50 usec
PL1      0.00 dB
SF01     300.1312226 MHz

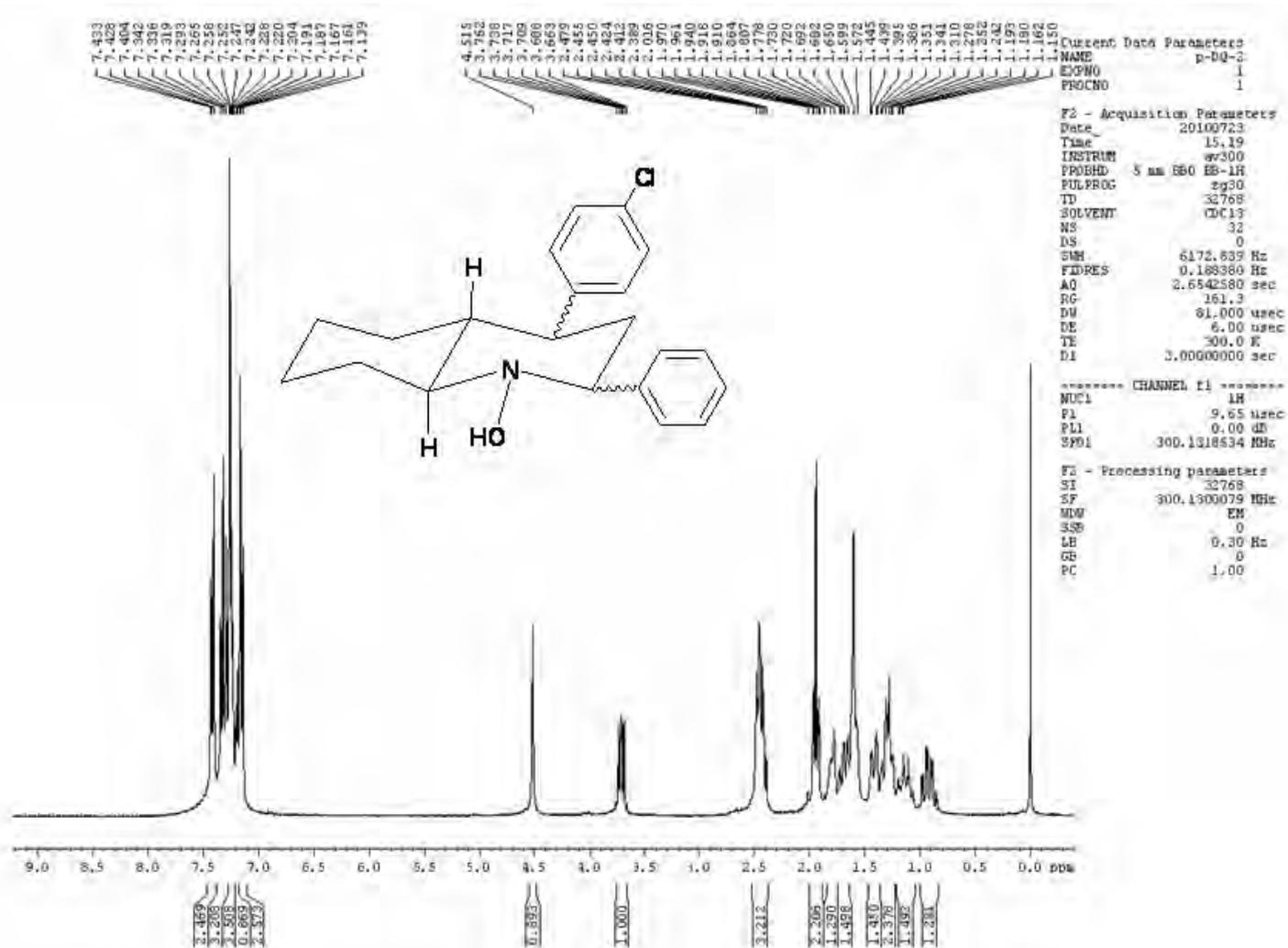
----- CHANNEL f2 -----
CPDPRG2  garp
NUC2     13C
P3       12.50 usec
p4       25.00 usec
PCPD2    75.00 usec
PL2      -5.00 dB
PL12     10.56 dB
SF02     75.4733476 MHz

----- GRADIENT CHANNEL -----
GPNAM1   SINE.100
GPNAM2   SINE.100
GPX1     0.00 %
GPX2     0.00 %
GPY1     0.00 %
GPY2     0.00 %
GPZ1     80.00 %
GPZ2     20.10 %
P16      1500.00 usec
```

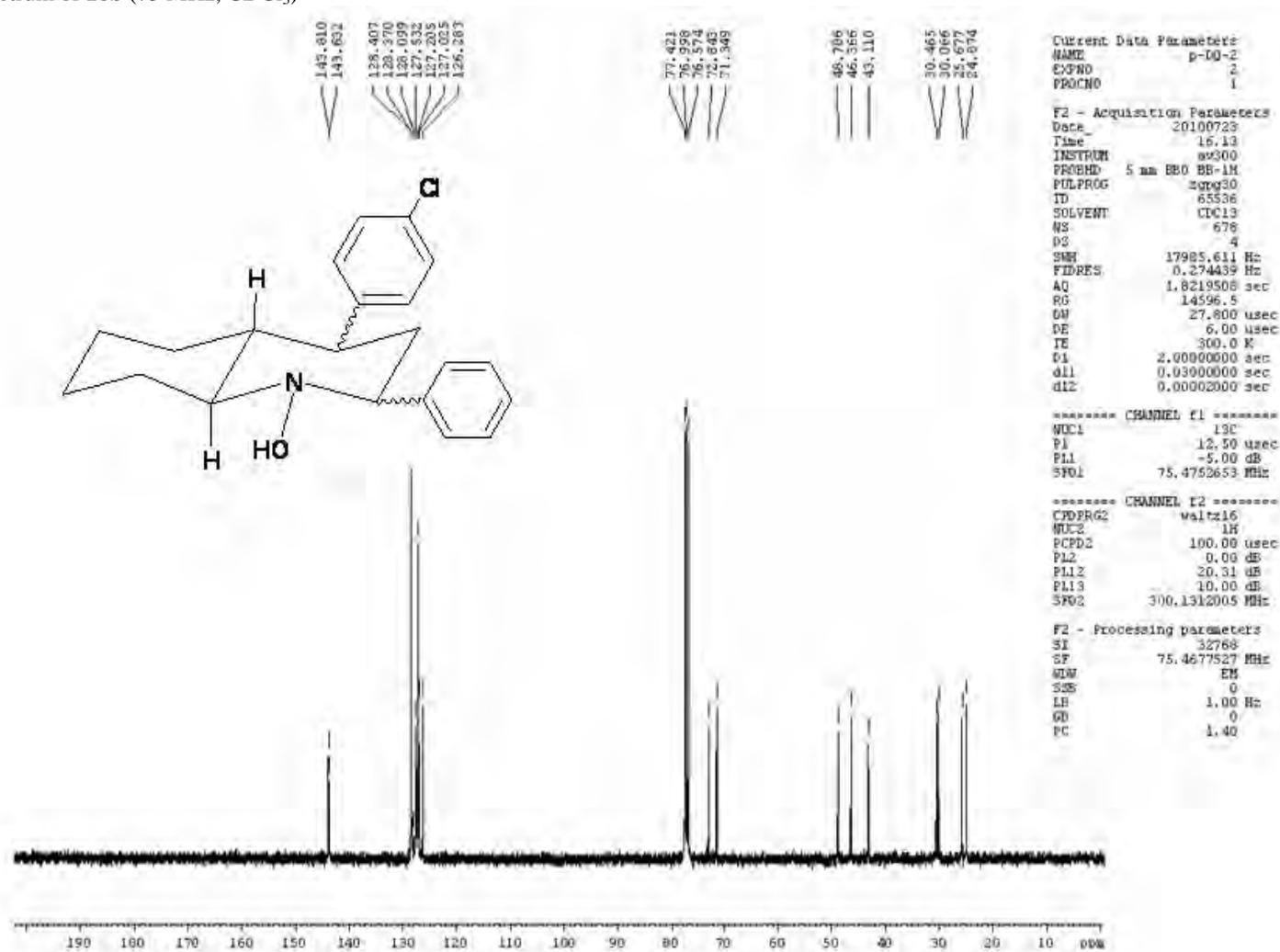
HMBC spectrum of **15c**



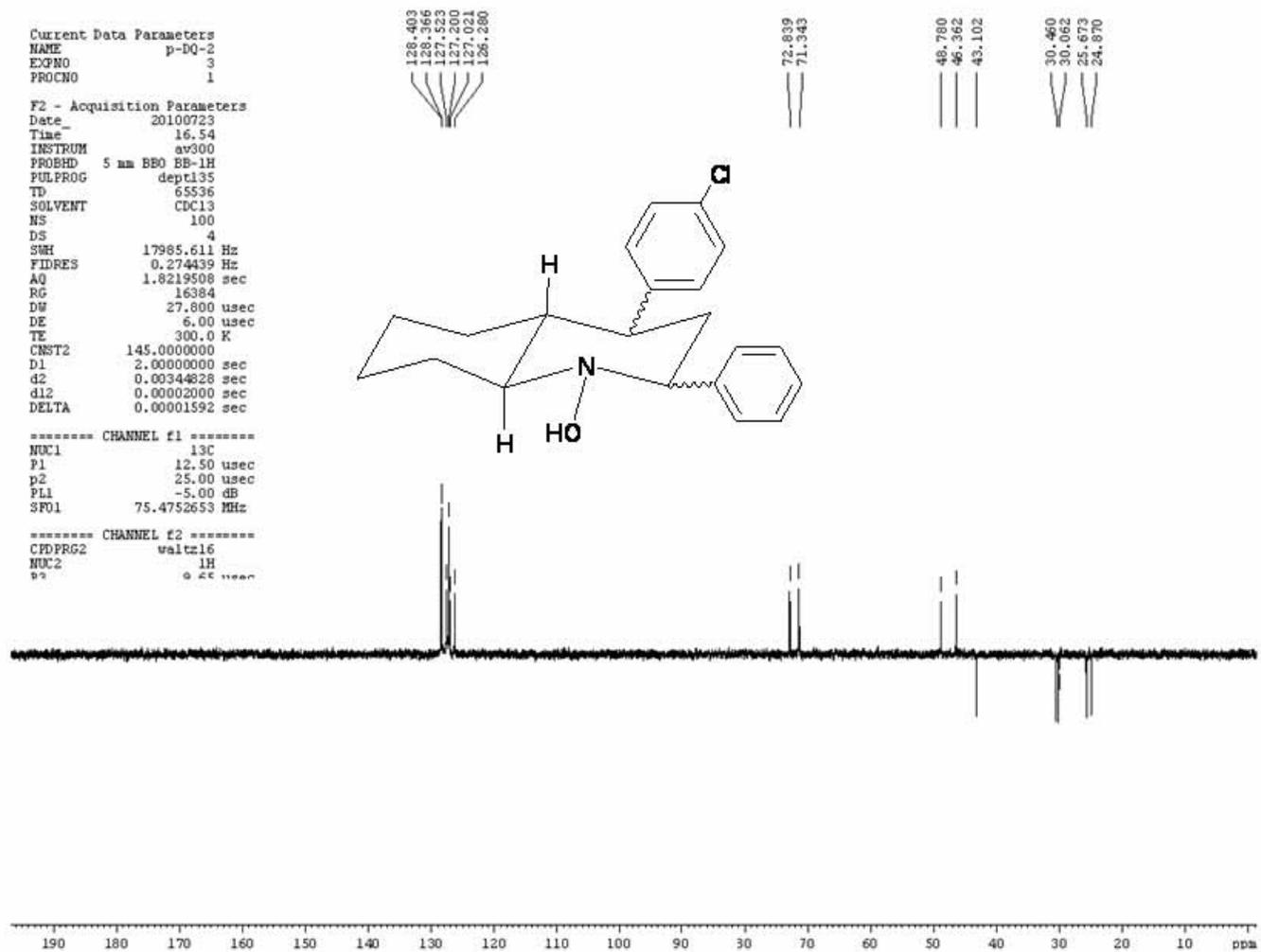
<sup>1</sup>H NMR spectrum of **16b** (300 MHz, CDCl<sub>3</sub>)



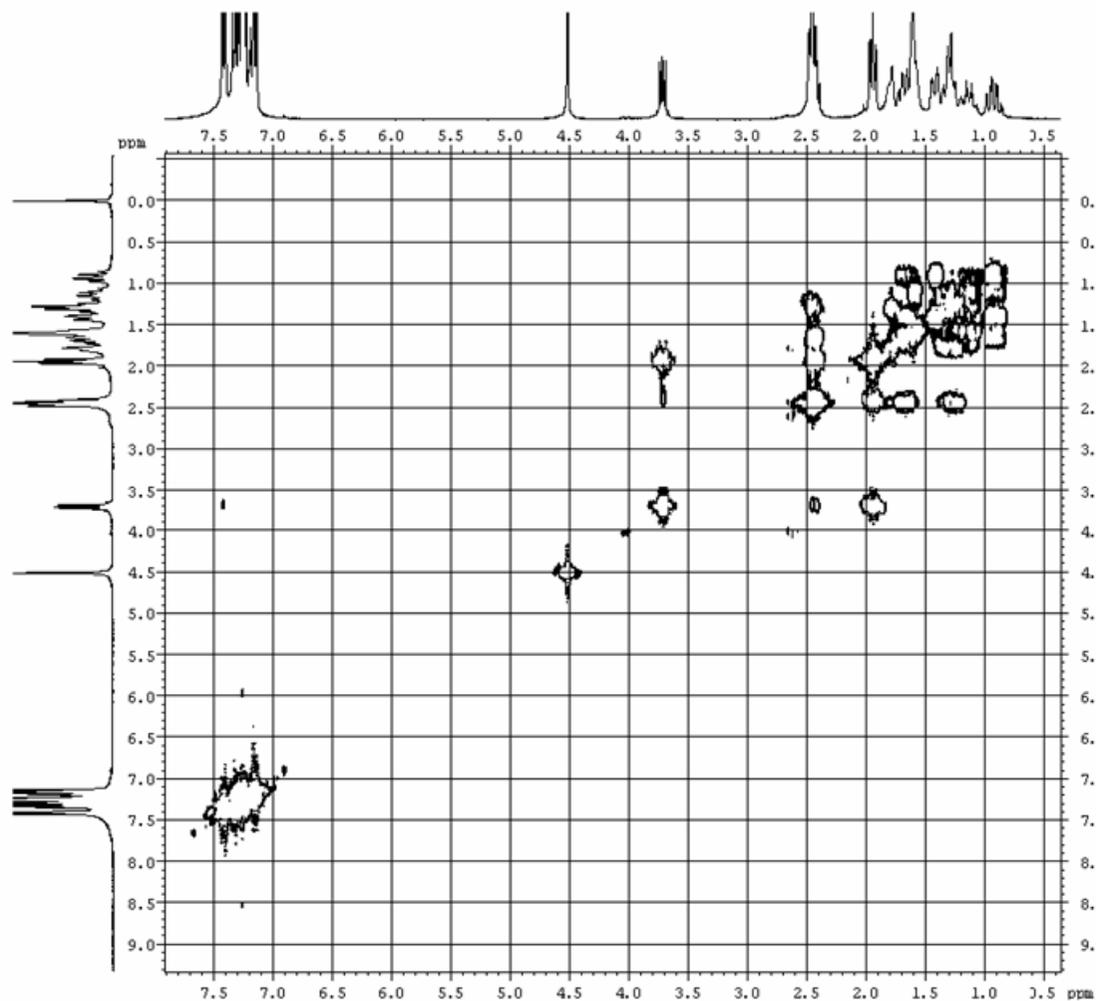
$^{13}\text{C}$  NMR spectrum of **16b** (75 MHz,  $\text{CDCl}_3$ )



DEPT-135 spectrum of **16b**



H-H COSY spectrum of **16b**



```
Current Data Parameters
NAME          p-DQ-2
EXPNO         4
PROCNO        1

F2 - Acquisition Parameters
Date_         20100723
Time          15.23
INSTRUM       av300
PROBHD        5 mm BBO BB-1H
PULPROG       cosyppqf
ID            2048
SOLVENT       DMSO
NS            2
DS            8
SWH           4006.410 Hz
FIDRES        1.956255 Hz
AQ            0.2556404 sec
RG            64
DW            124.800 usec
DE            6.00 usec
TE            300.0 K
d0            0.00000300 sec
d1            1.48689198 sec
d13           0.00000400 sec
d16           0.00015000 sec
dNO          0.00024960 sec
MCREST        0.00000000 sec
MCWRK         1.48689198 sec

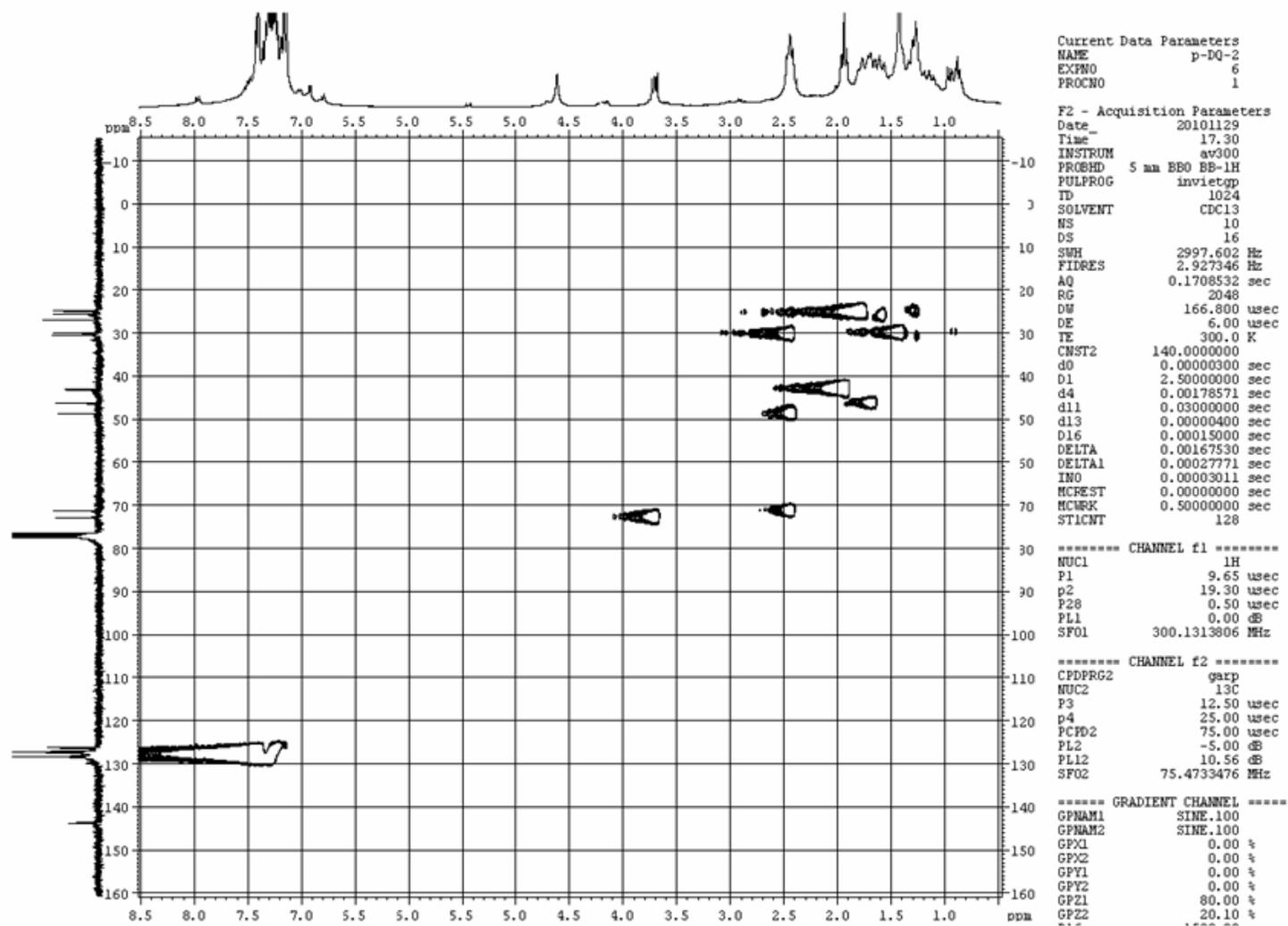
***** CHANNEL f1 *****
NUC1          1H
P0            9.65 usec
P1            9.65 usec
PL1           0.00 dB
SFO1          300.1318044 MHz

***** GRADIENT CHANNEL *****
GPNAM1        SINE.100
GPNAM2        SINE.100
GPX1          0.00 %
GPX2          0.00 %
GPY1          0.00 %
GPY2          0.00 %
GPZ1          10.00 %
GPZ2          10.00 %
PL6           1500.00 usec

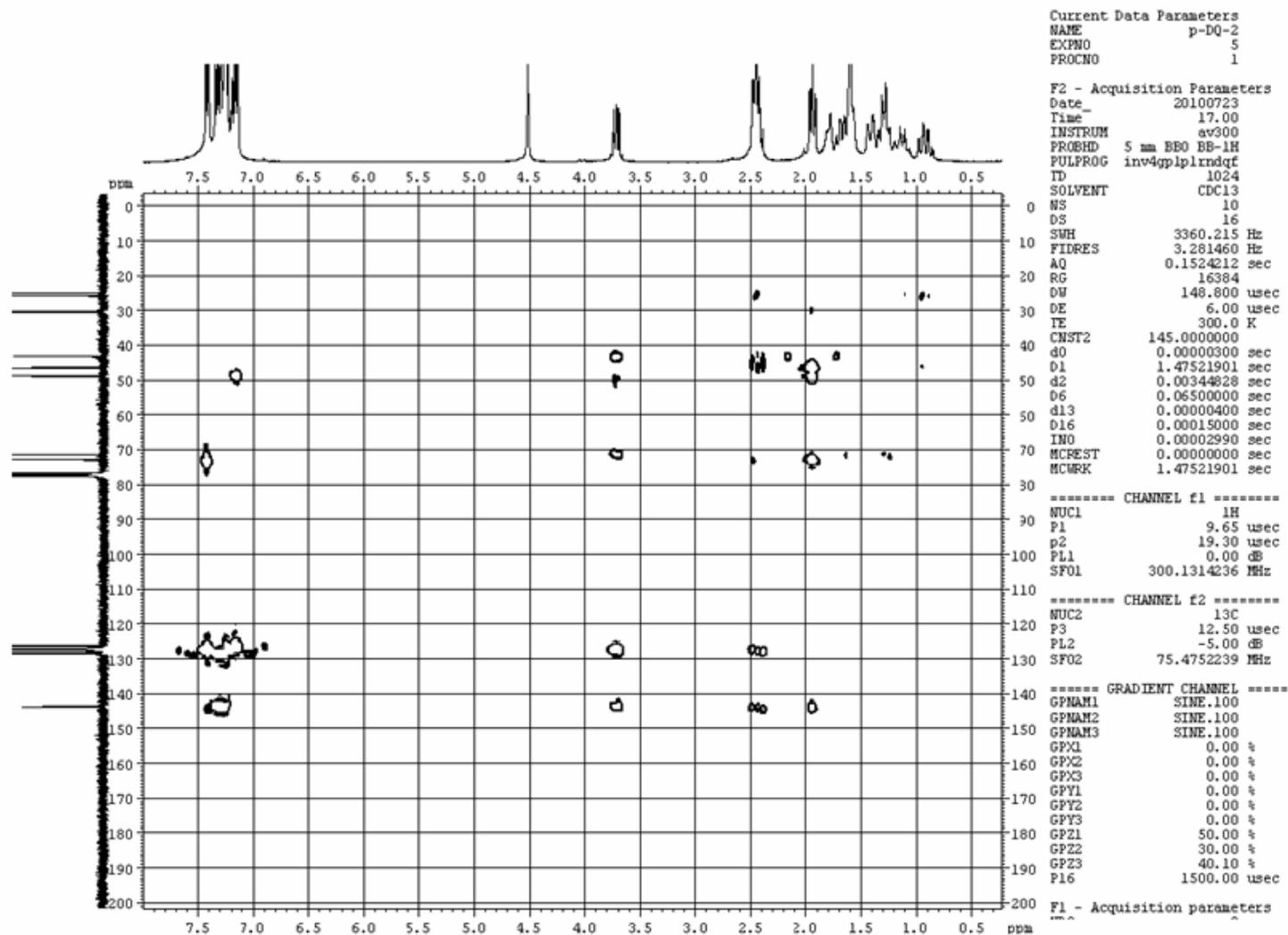
F1 - Acquisition parameters
ND0           1
ID            128
SFO1          300.1318 MHz
FIDRES        31.300079 Hz
SU            13.349 ppm

F2 - Processing parameters
SI            1024
SF            300.1300073 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
```

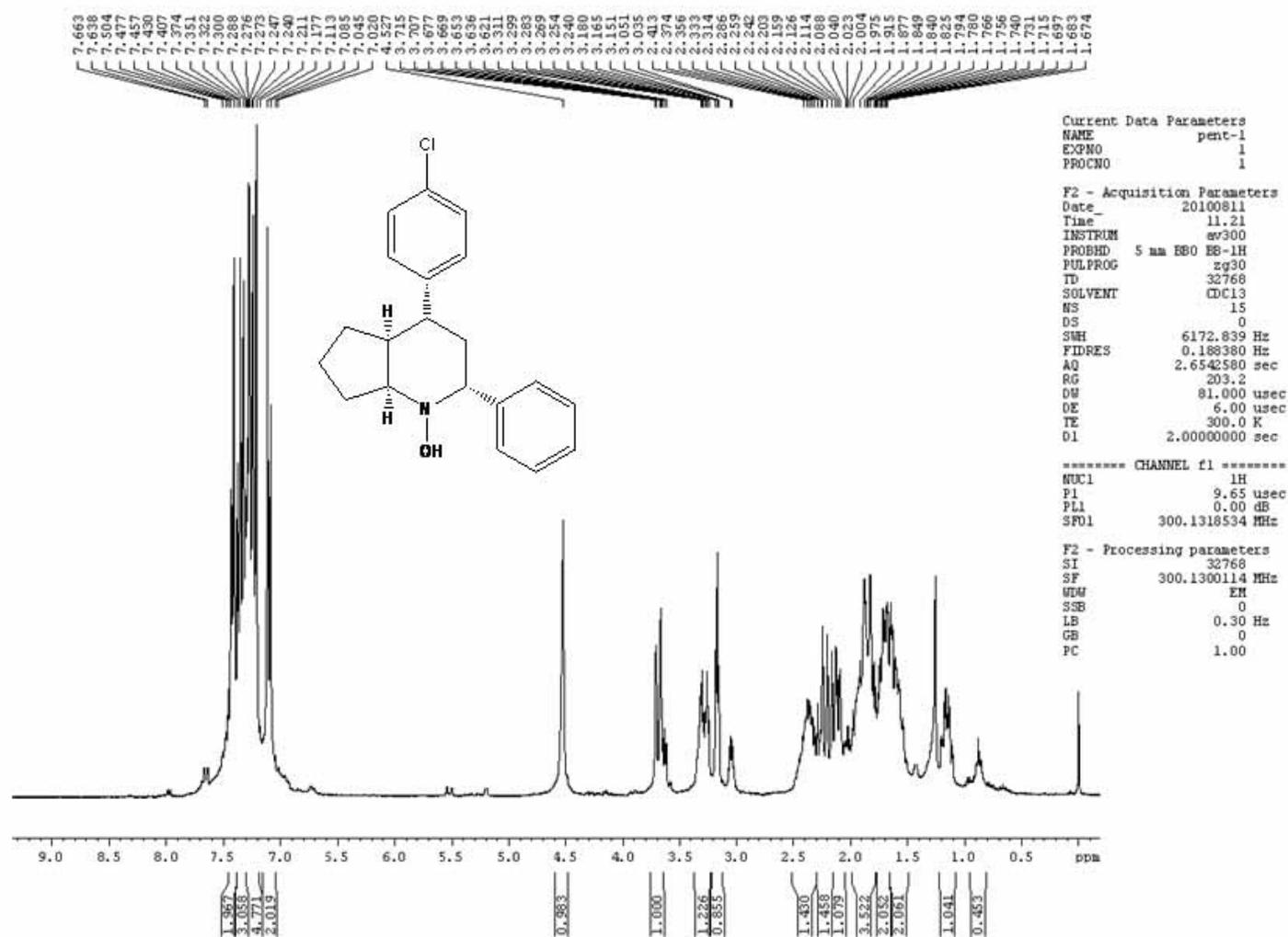
C-H COSY spectrum of **16b**



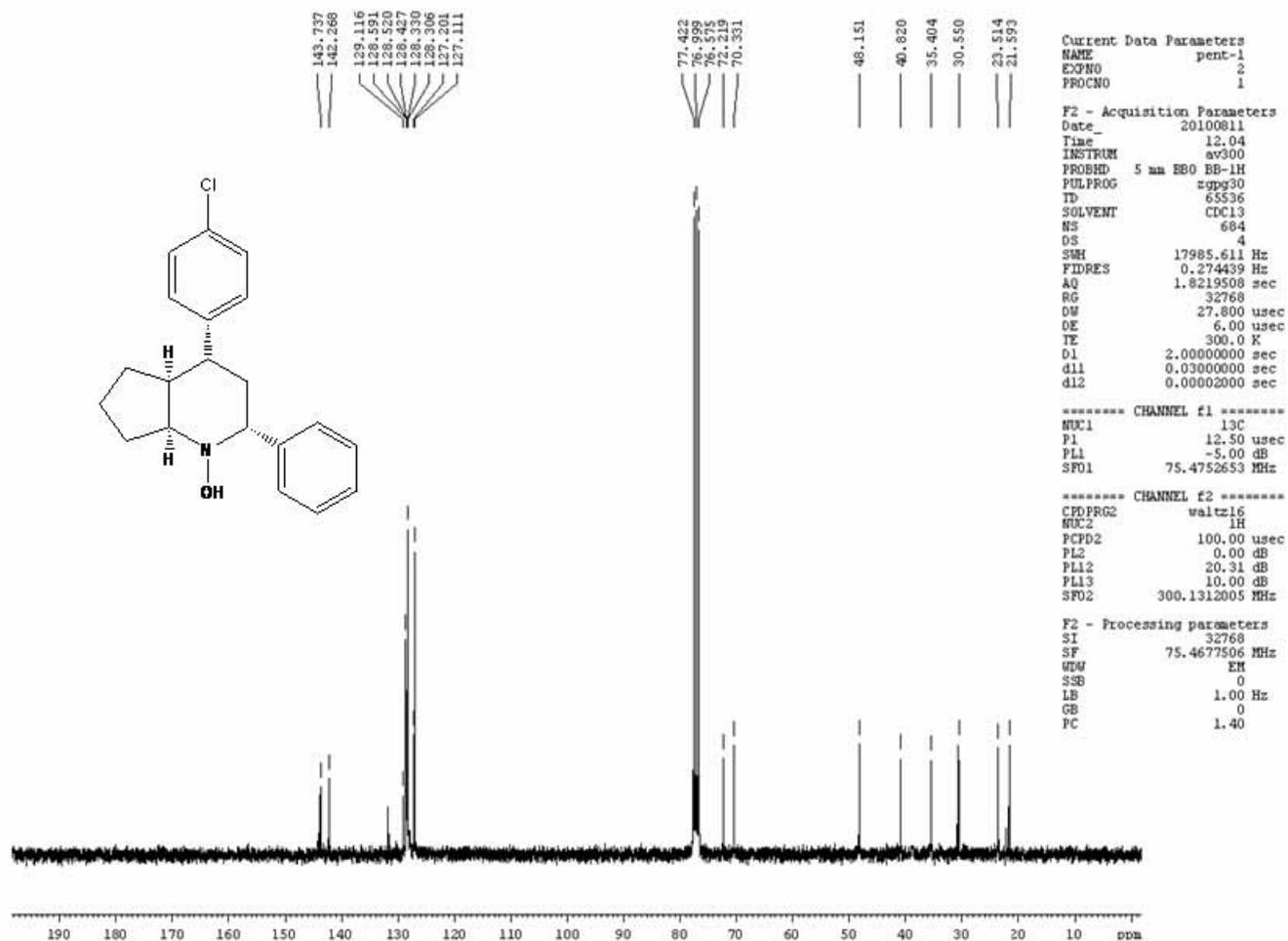
HMBC spectrum of **16b**



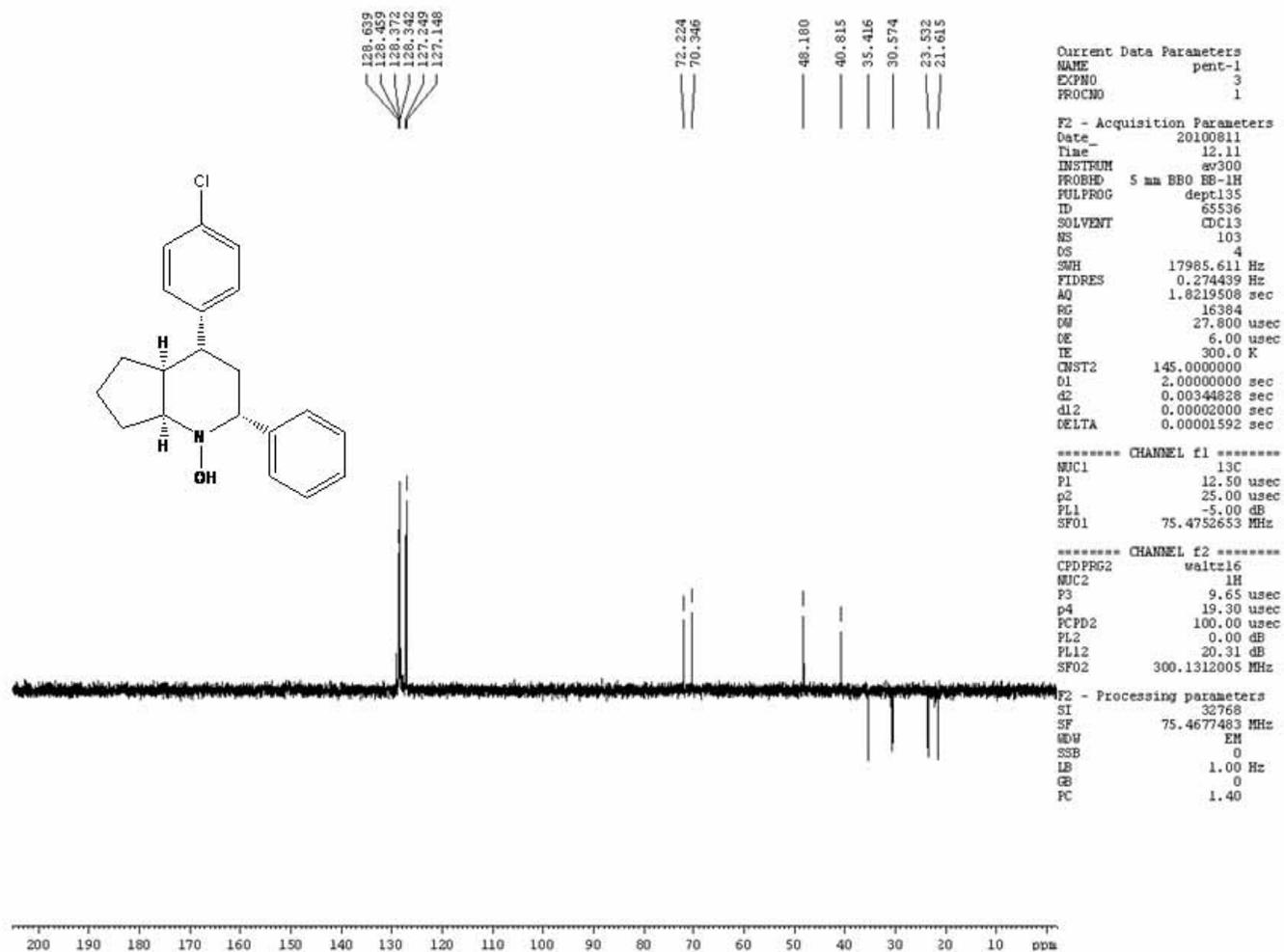
<sup>1</sup>H NMR spectrum of **18** (300 MHz, CDCl<sub>3</sub>)



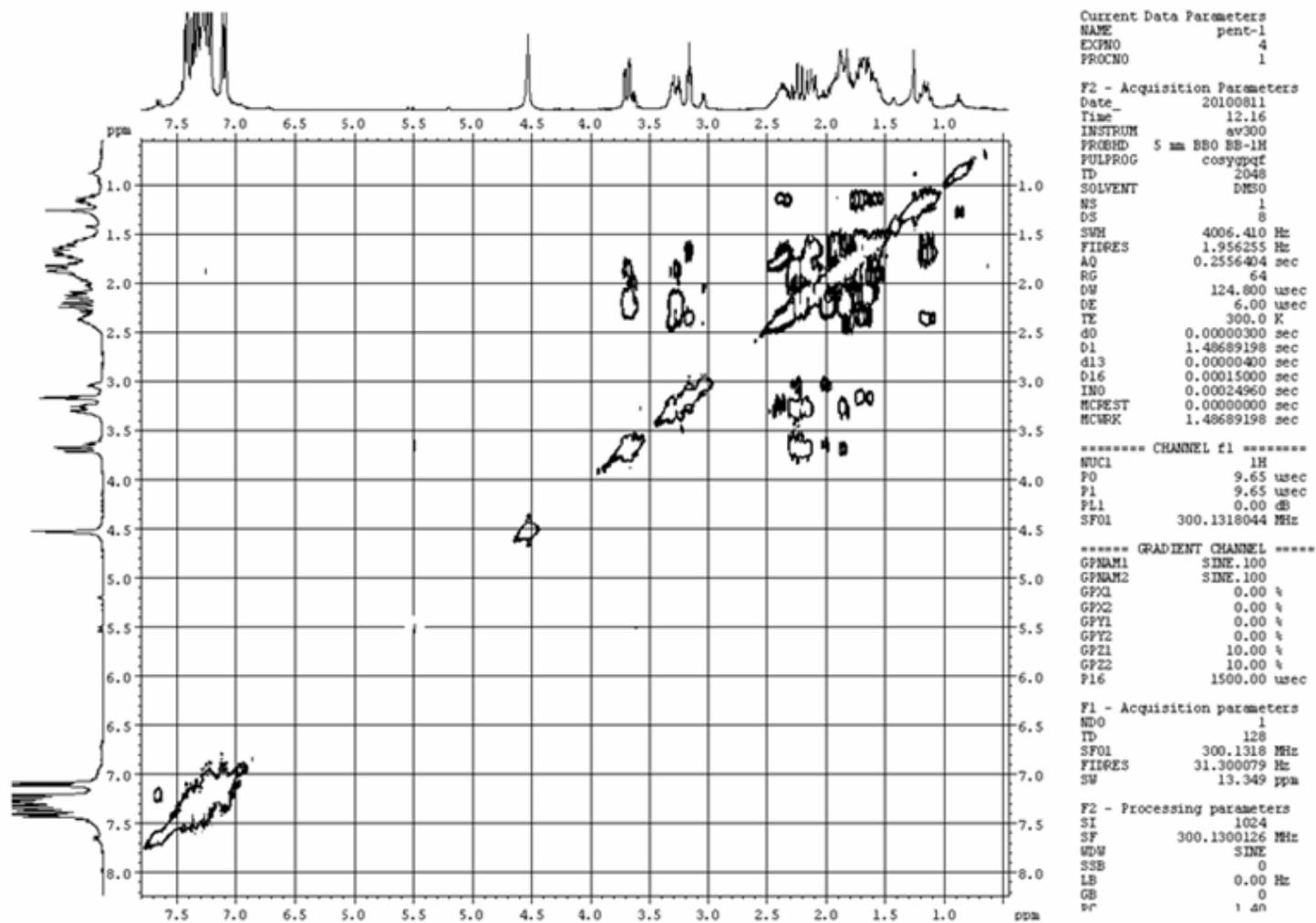
$^{13}\text{C}$  NMR spectrum of **18** (75 MHz,  $\text{CDCl}_3$ )



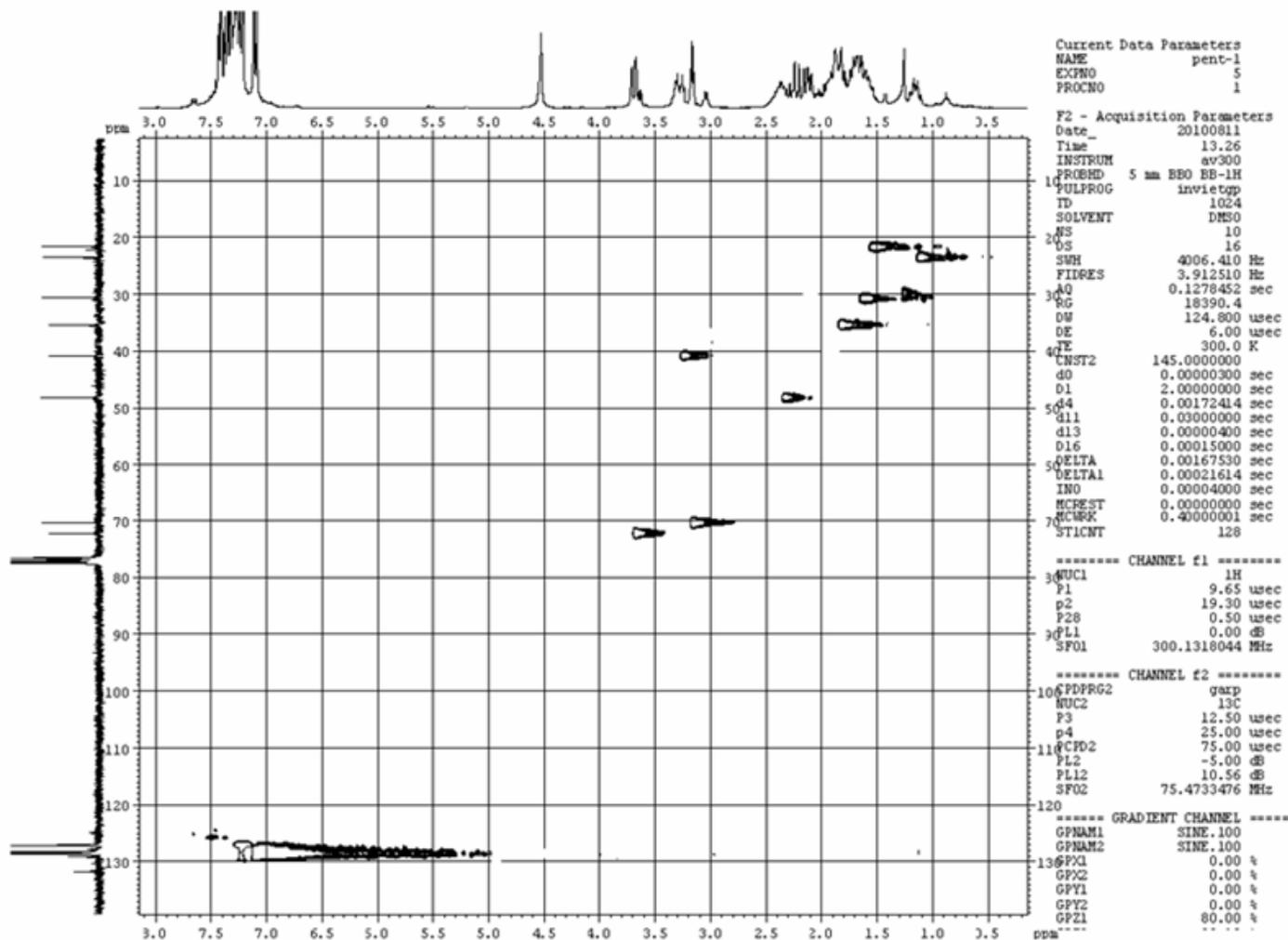
DEPT-135 spectrum of **18**



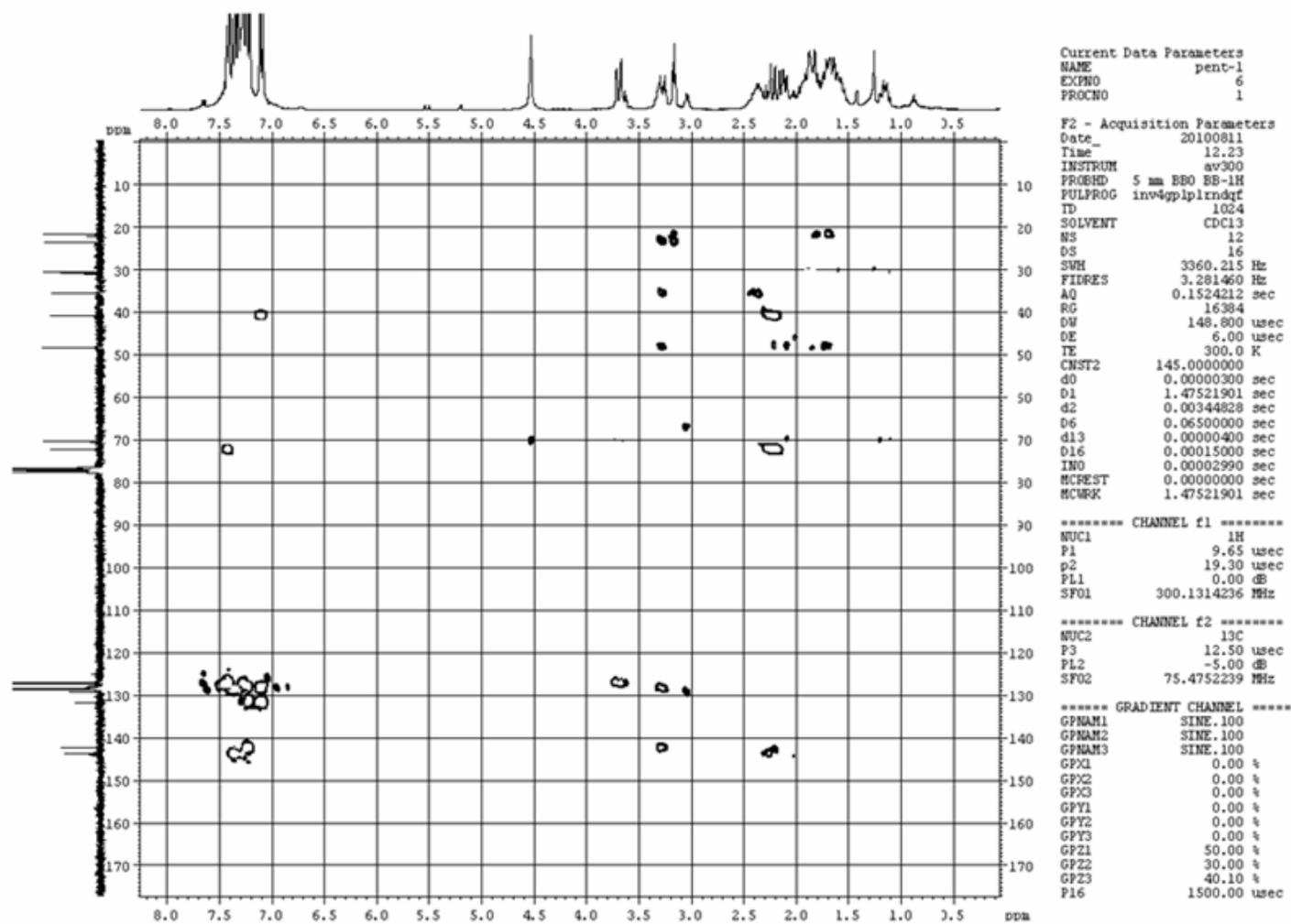
H-H COSY spectrum of **18**



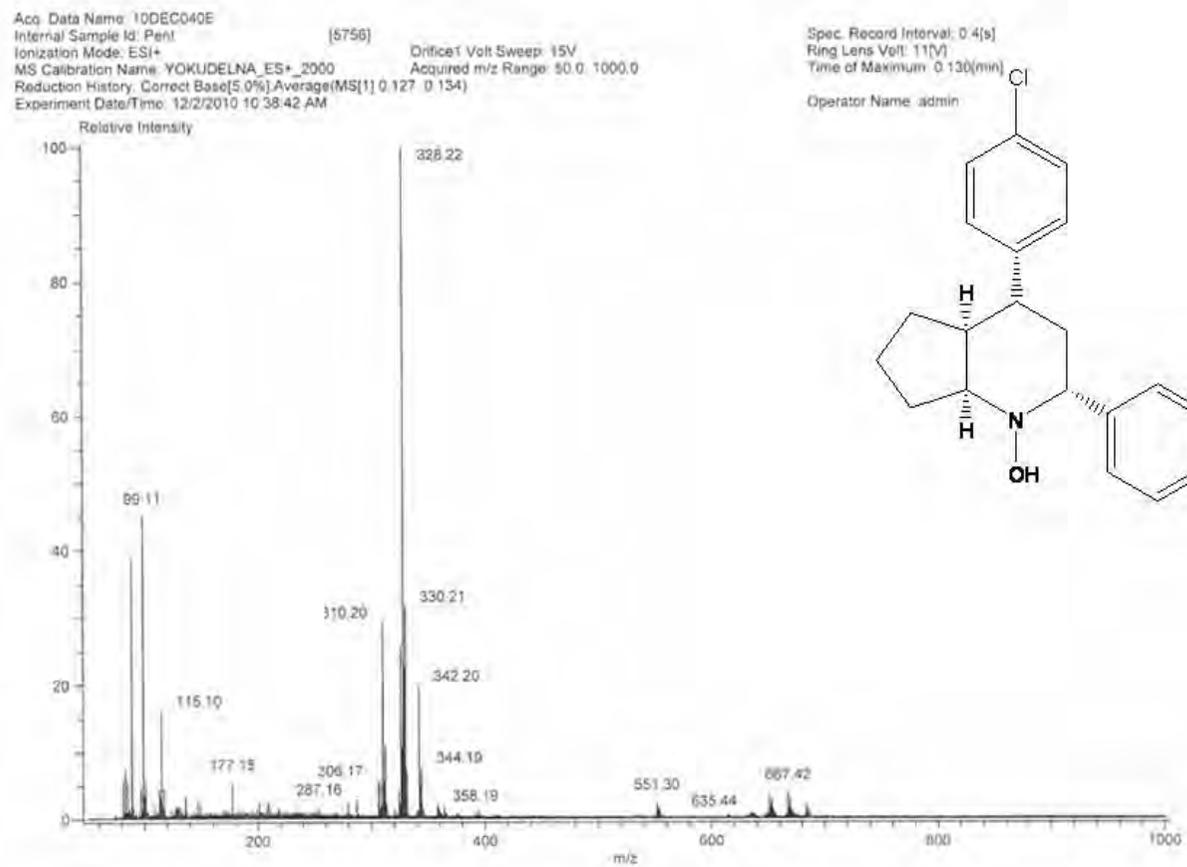
C-H COSY spectrum of **18**



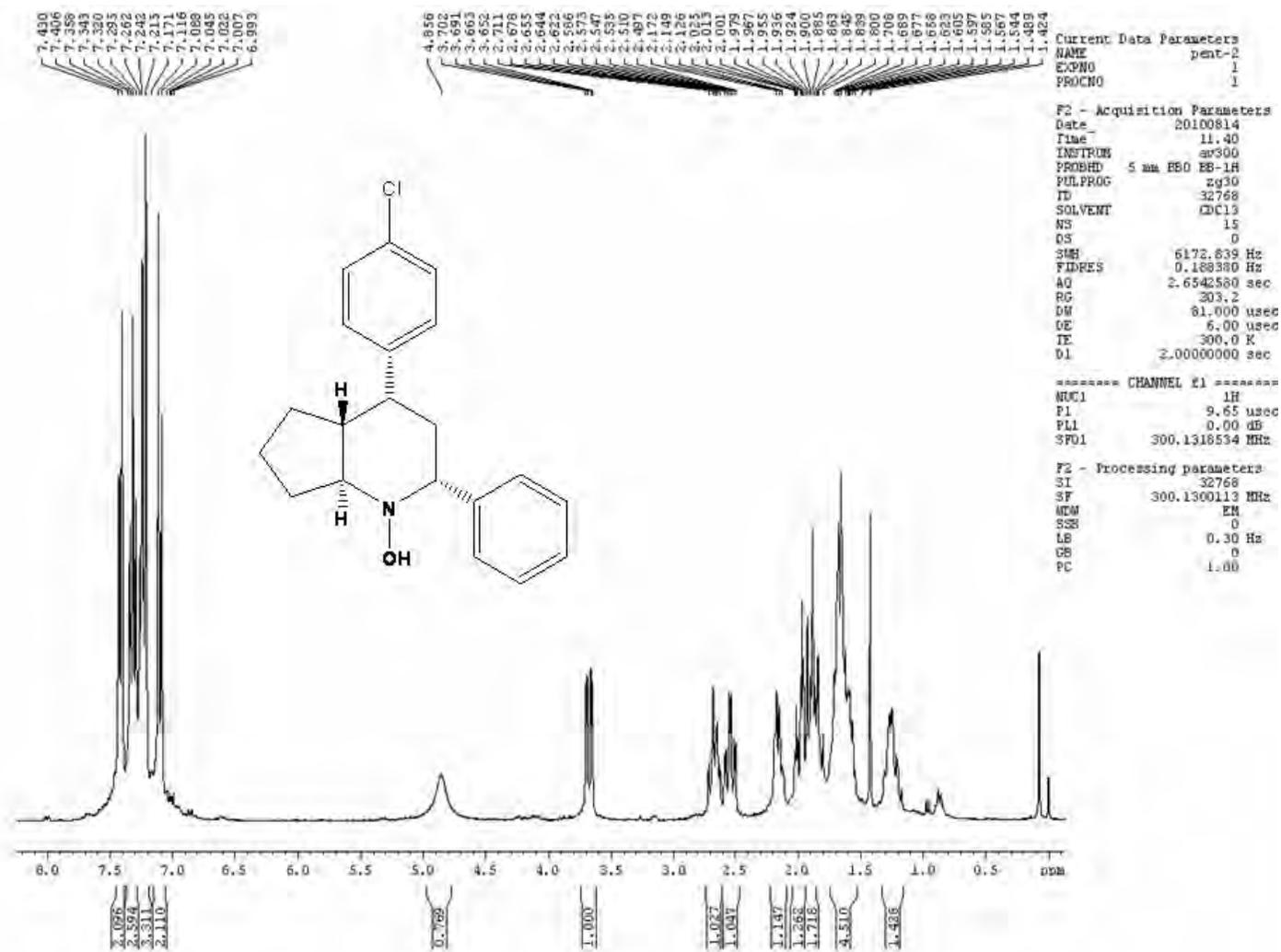
HMBC spectrum of **18**



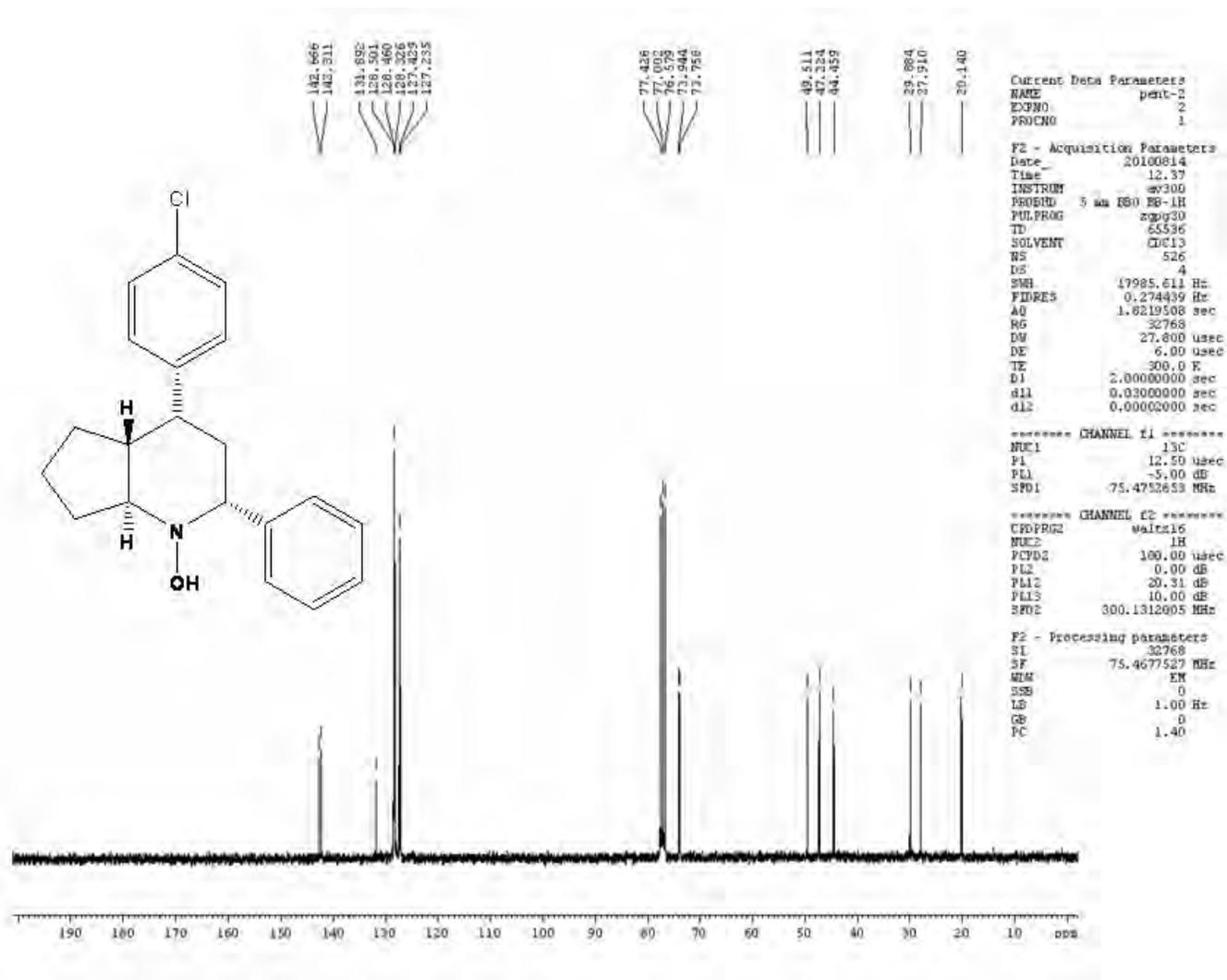
Mass spectrum of **18**



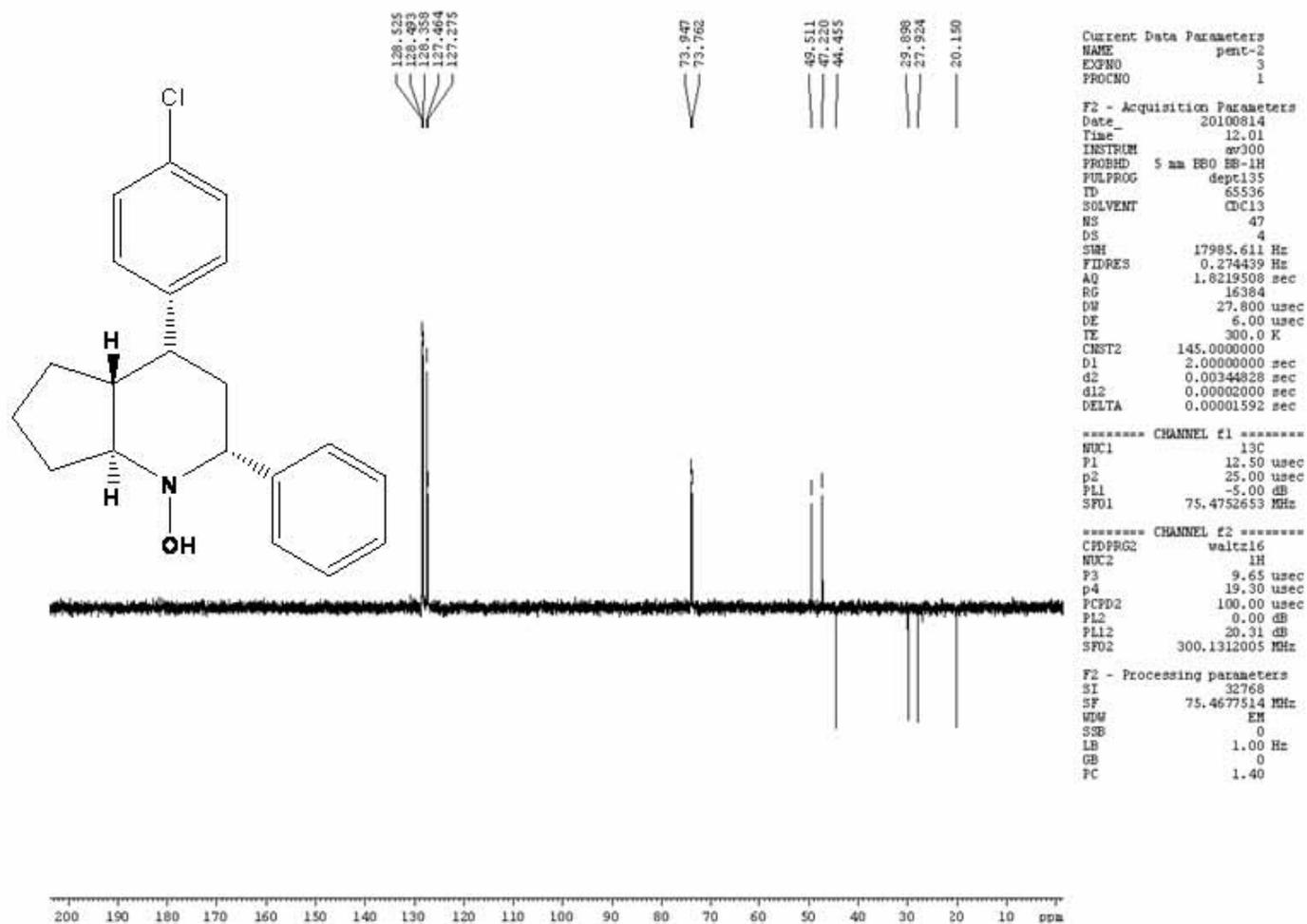
<sup>1</sup>H NMR spectrum of **19** (300 MHz, CDCl<sub>3</sub>)



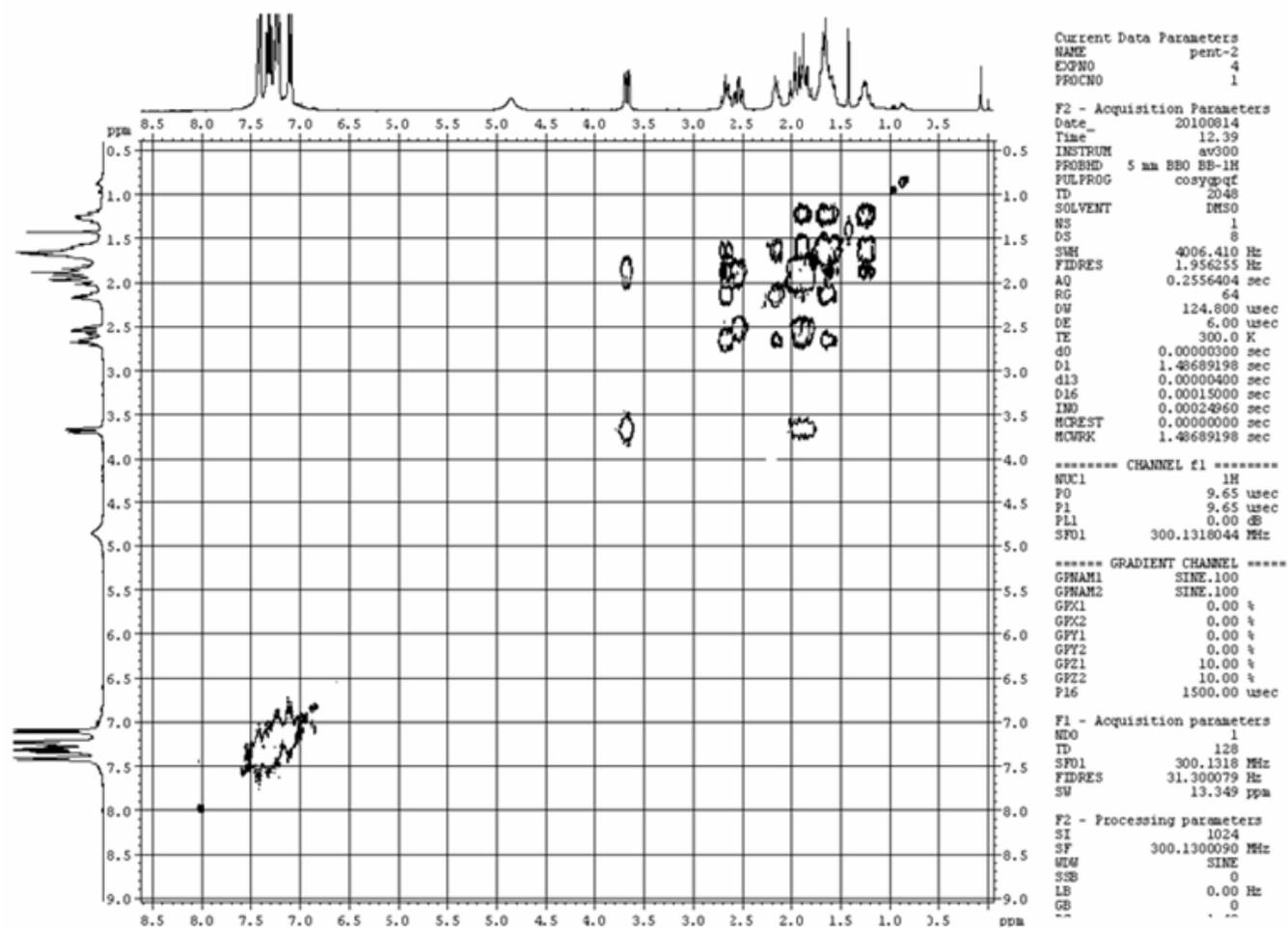
$^{13}\text{C}$  NMR spectrum of **19** (75 MHz,  $\text{CDCl}_3$ )



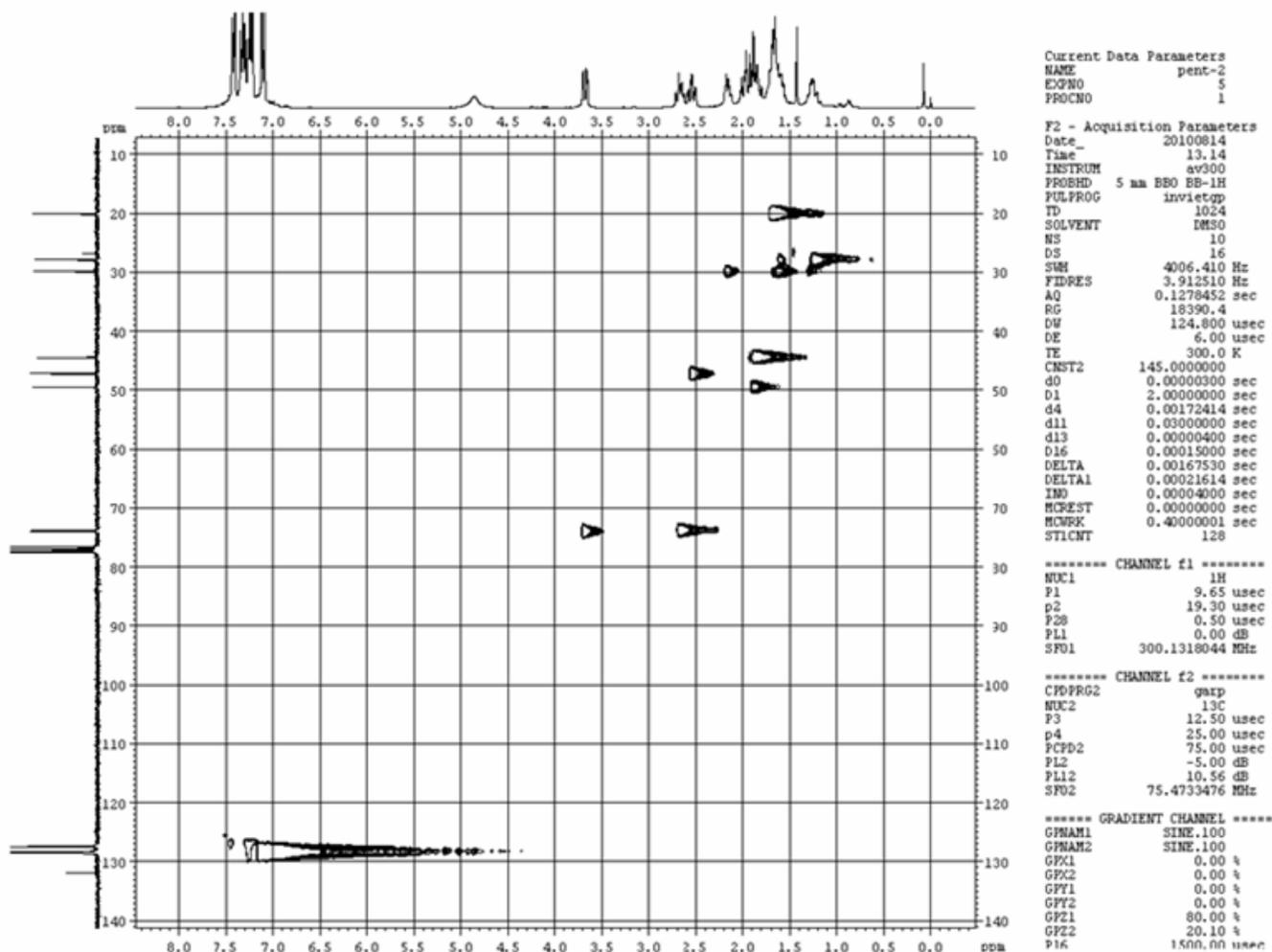
DEPT-135 spectrum of **19**



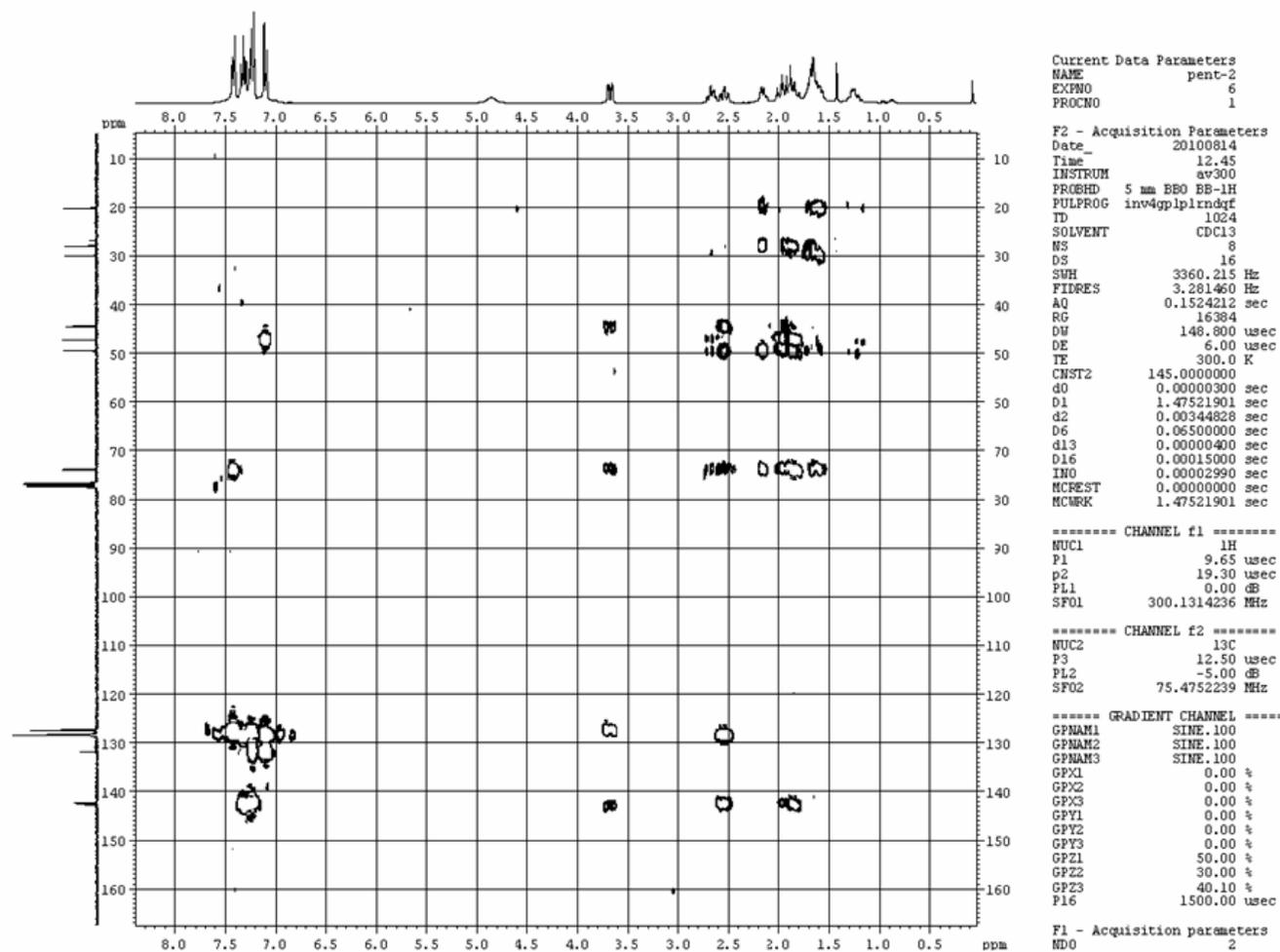
H-H COSY spectrum of **19**



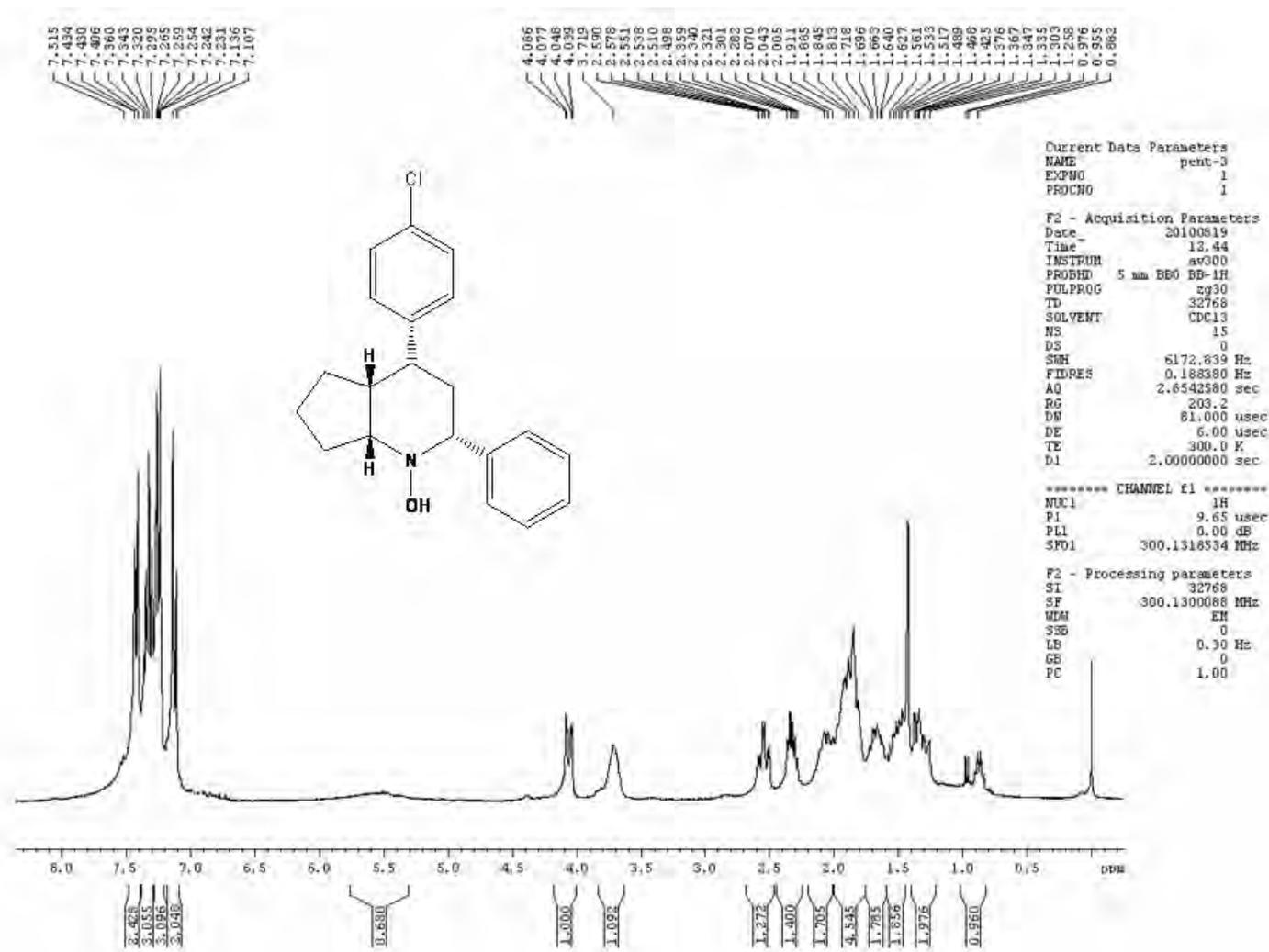
C-H COSY spectrum of **19**



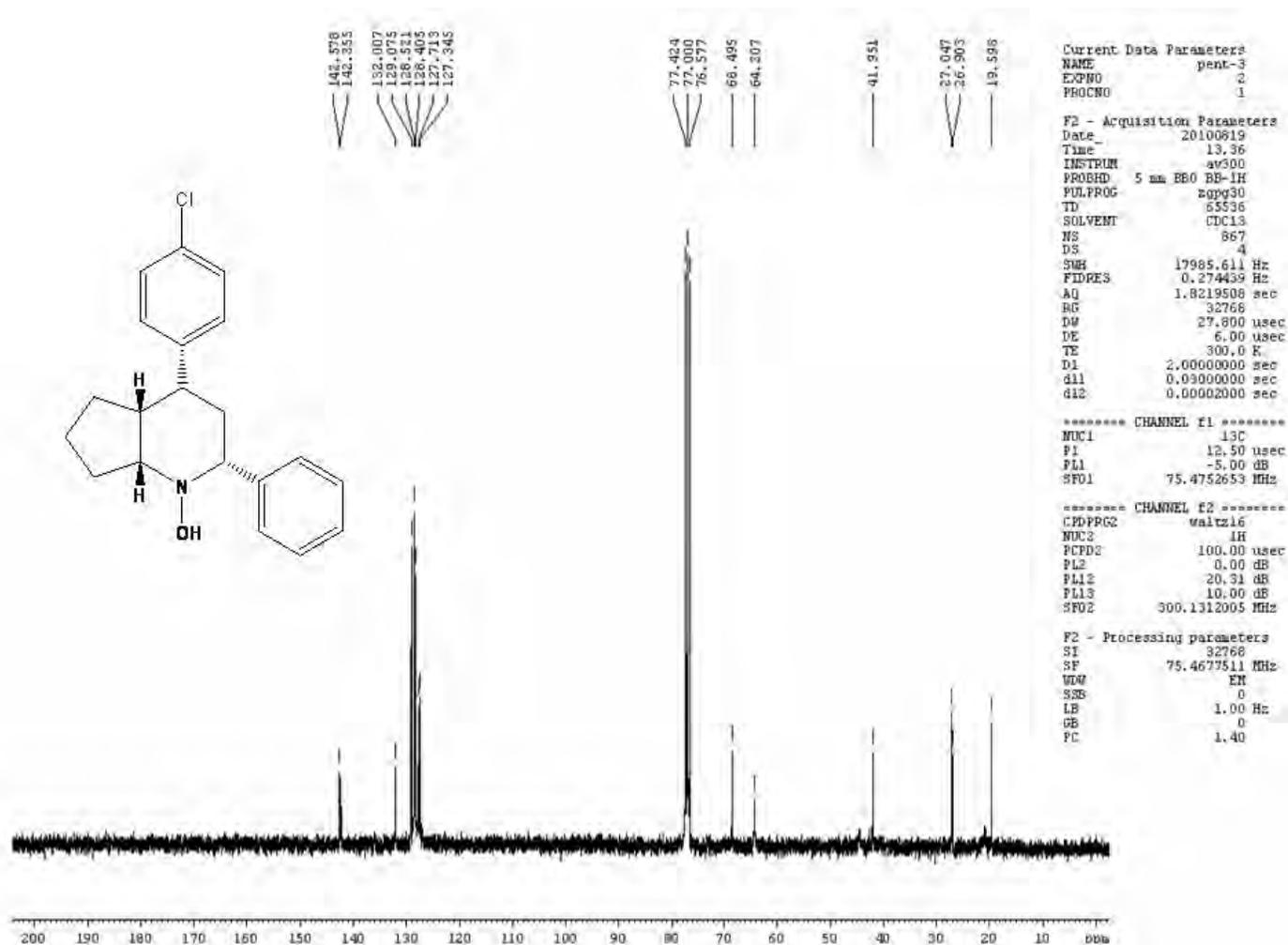
HMBC spectrum of **19**



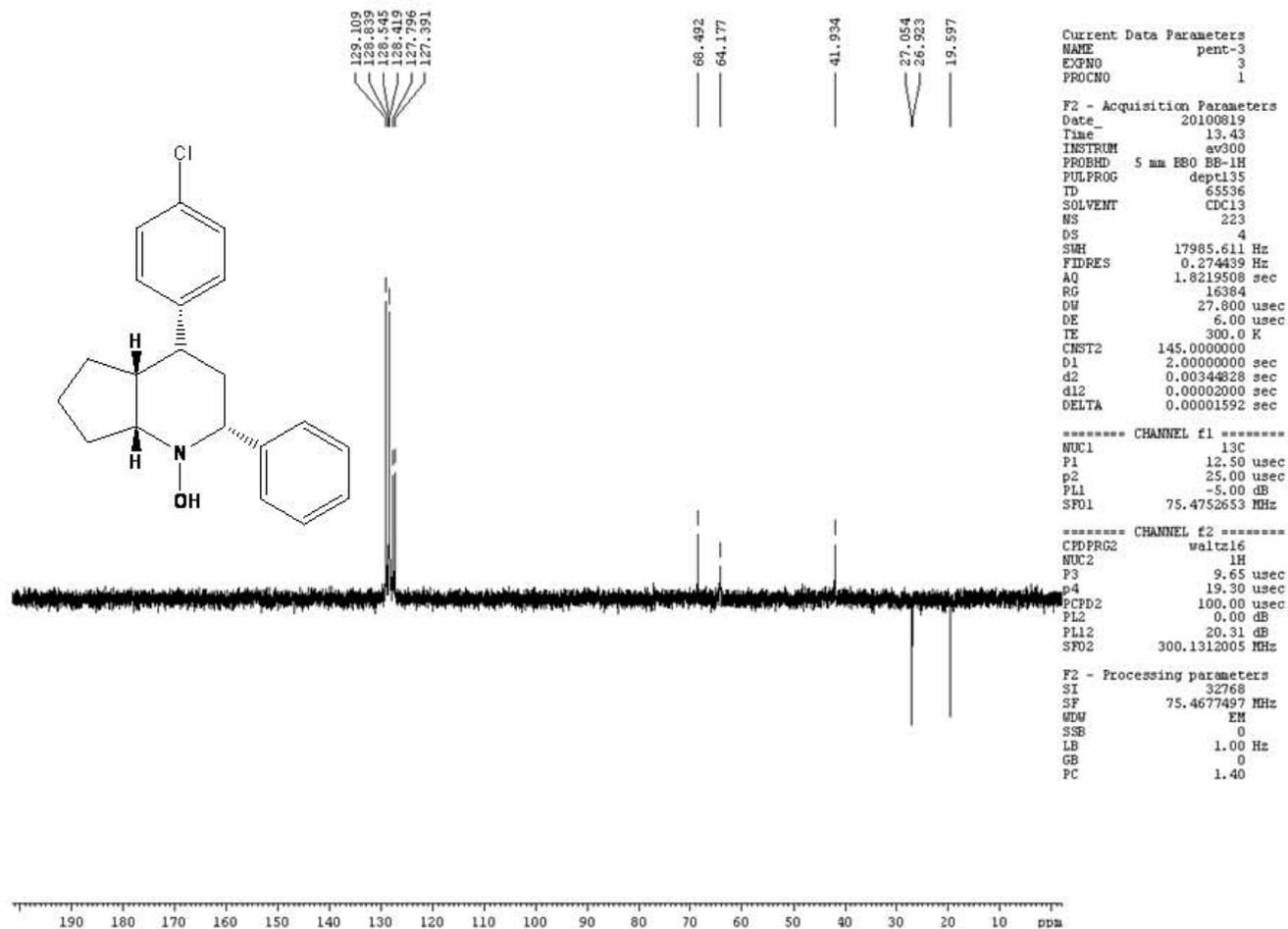
<sup>1</sup>H NMR spectrum of **20** (300 MHz, CDCl<sub>3</sub>)



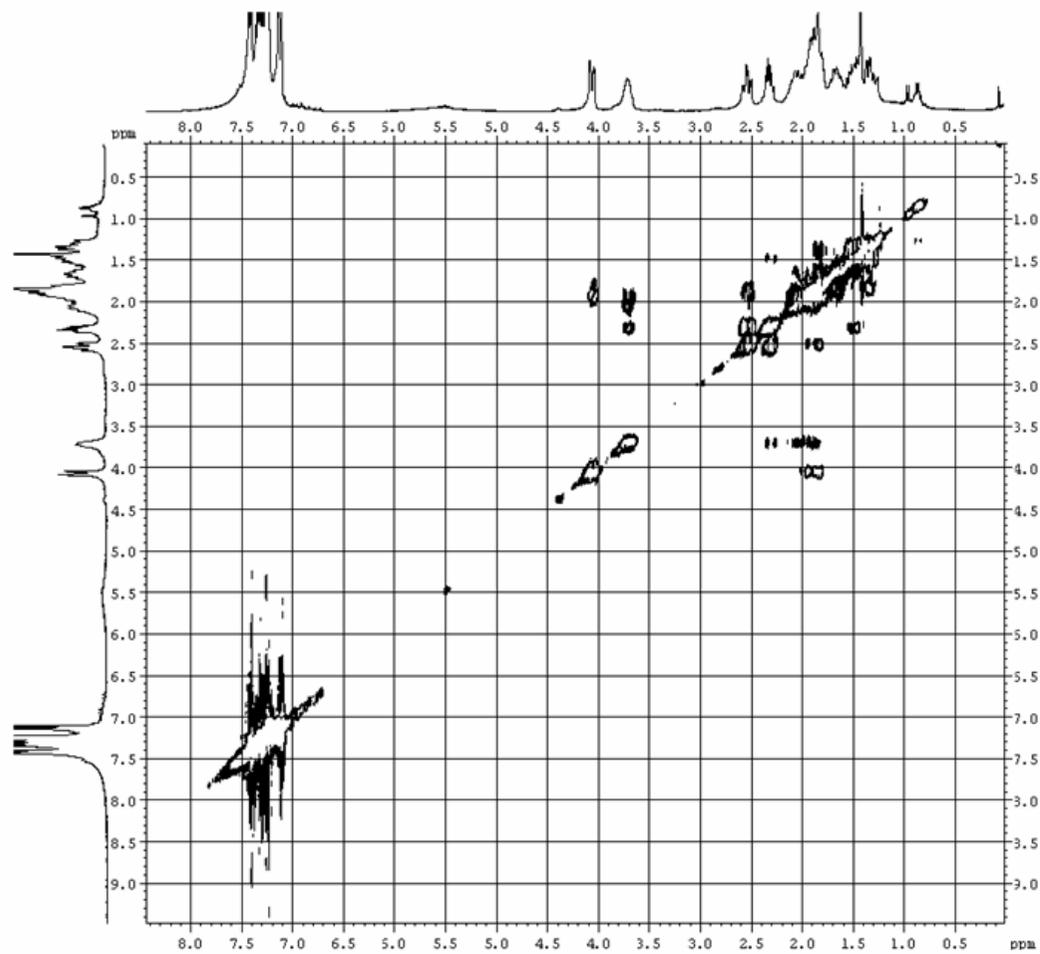
$^{13}\text{C}$  NMR spectrum of **20** (75 MHz,  $\text{CDCl}_3$ )



DEPT-135 spectrum of **20**



H-H COSY spectrum of **20**



```
Current Data Parameters
NAME pent-3
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100819
Time 15.39
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG cosygpgf
TD 2048
SOLVENT DMSO
NS 1
DS 8
SWH 4006.410 Hz
FIDRES 1.956255 Hz
AQ 0.2556404 sec
RG 64
DW 124.800 usec
DE 6.00 usec
TE 300.0 K
d0 0.0000300 sec
D1 1.48689198 sec
d13 0.0000400 sec
D16 0.00015000 sec
IN0 0.00024960 sec
MCREST 0.00000000 sec
MCMRK 1.48689198 sec

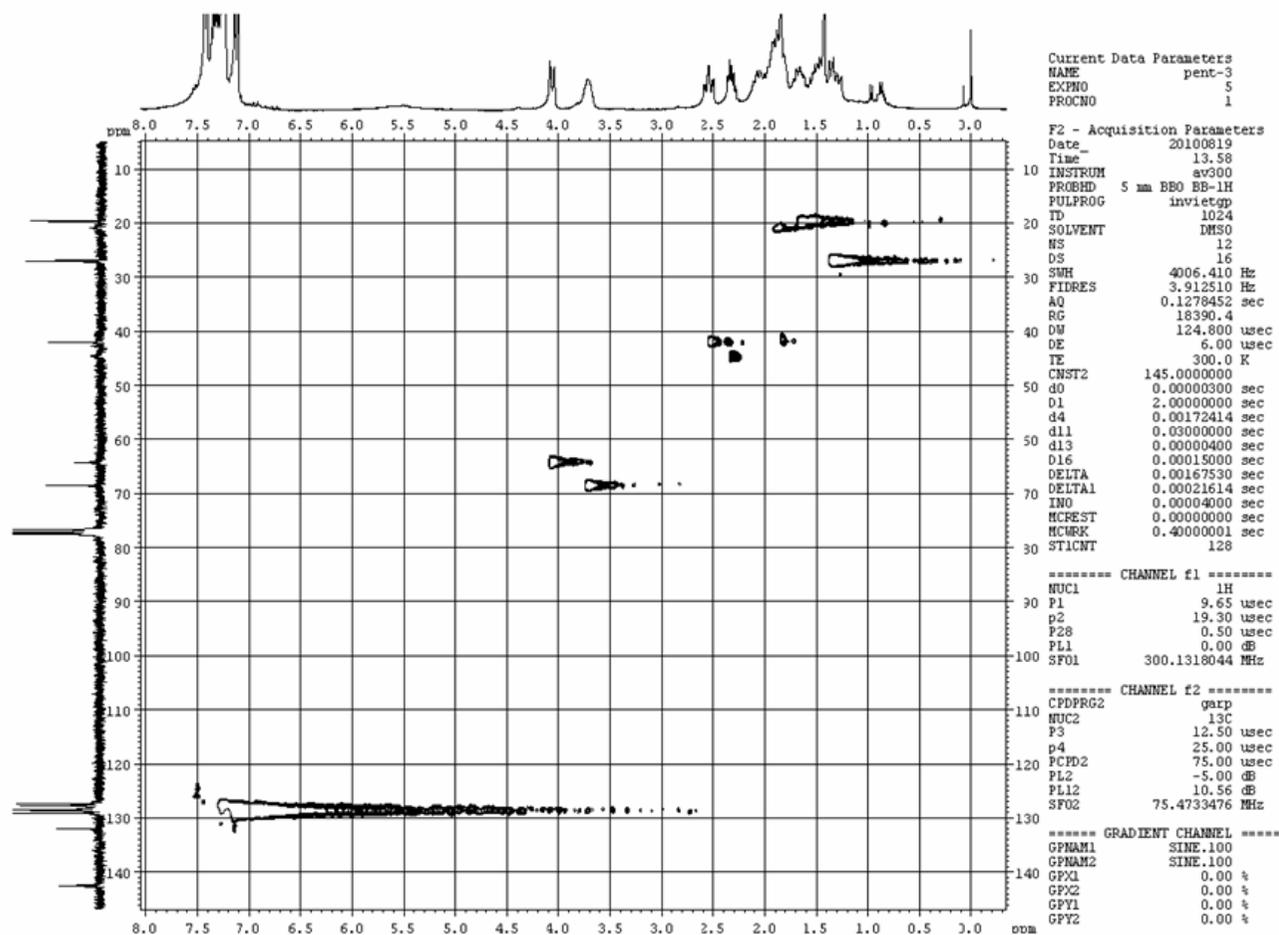
===== CHANNEL f1 =====
NUC1 1H
PO 9.65 usec
P1 9.65 usec
PL1 0.00 dB
SF01 300.1318044 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1500.00 usec

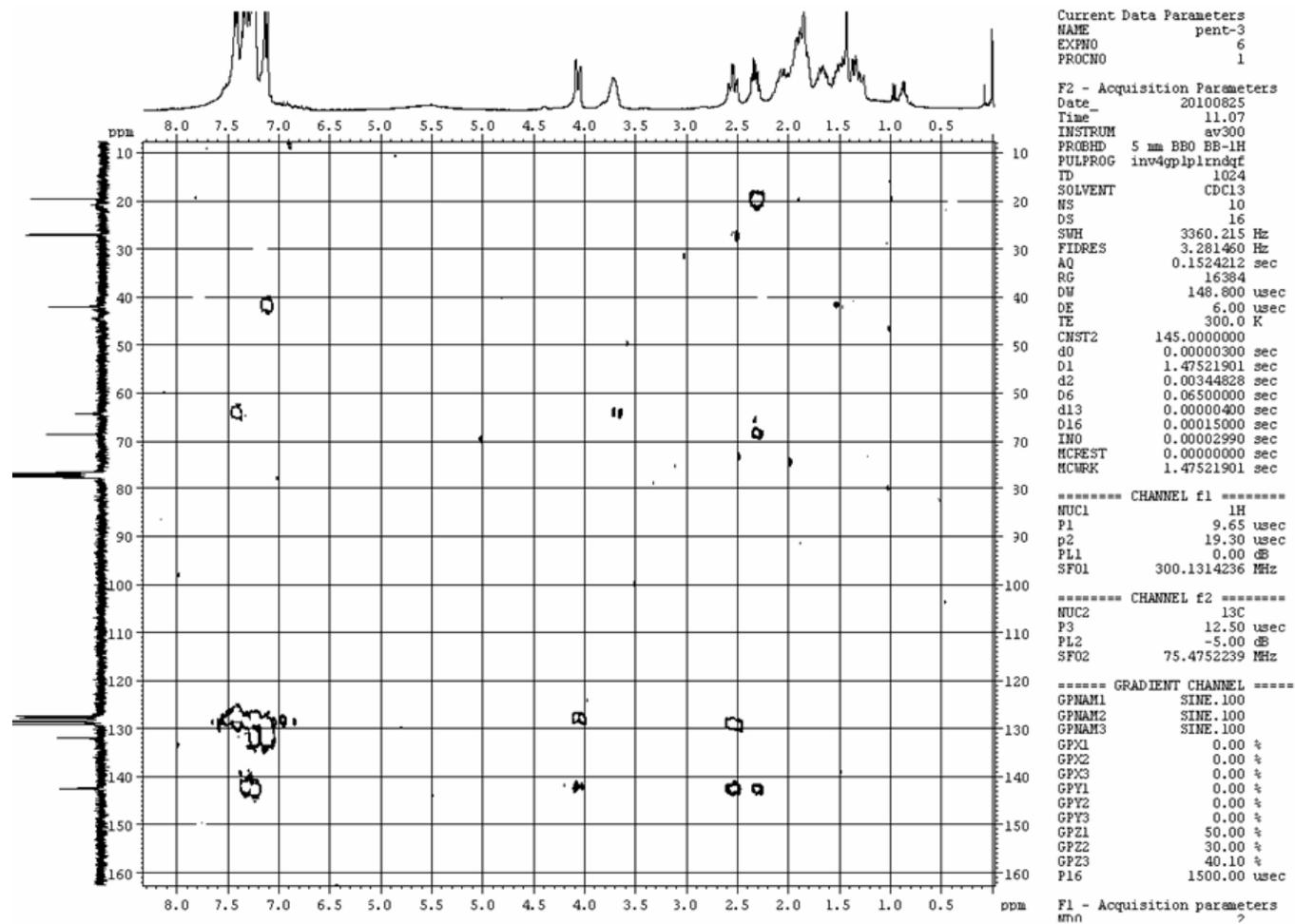
F1 - Acquisition parameters
ND0 1
TD 128
SF01 300.1318 MHz
FIDRES 31.300079 Hz
SW 13.349 ppm

F2 - Processing parameters
SI 1024
SF 300.1300122 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
```

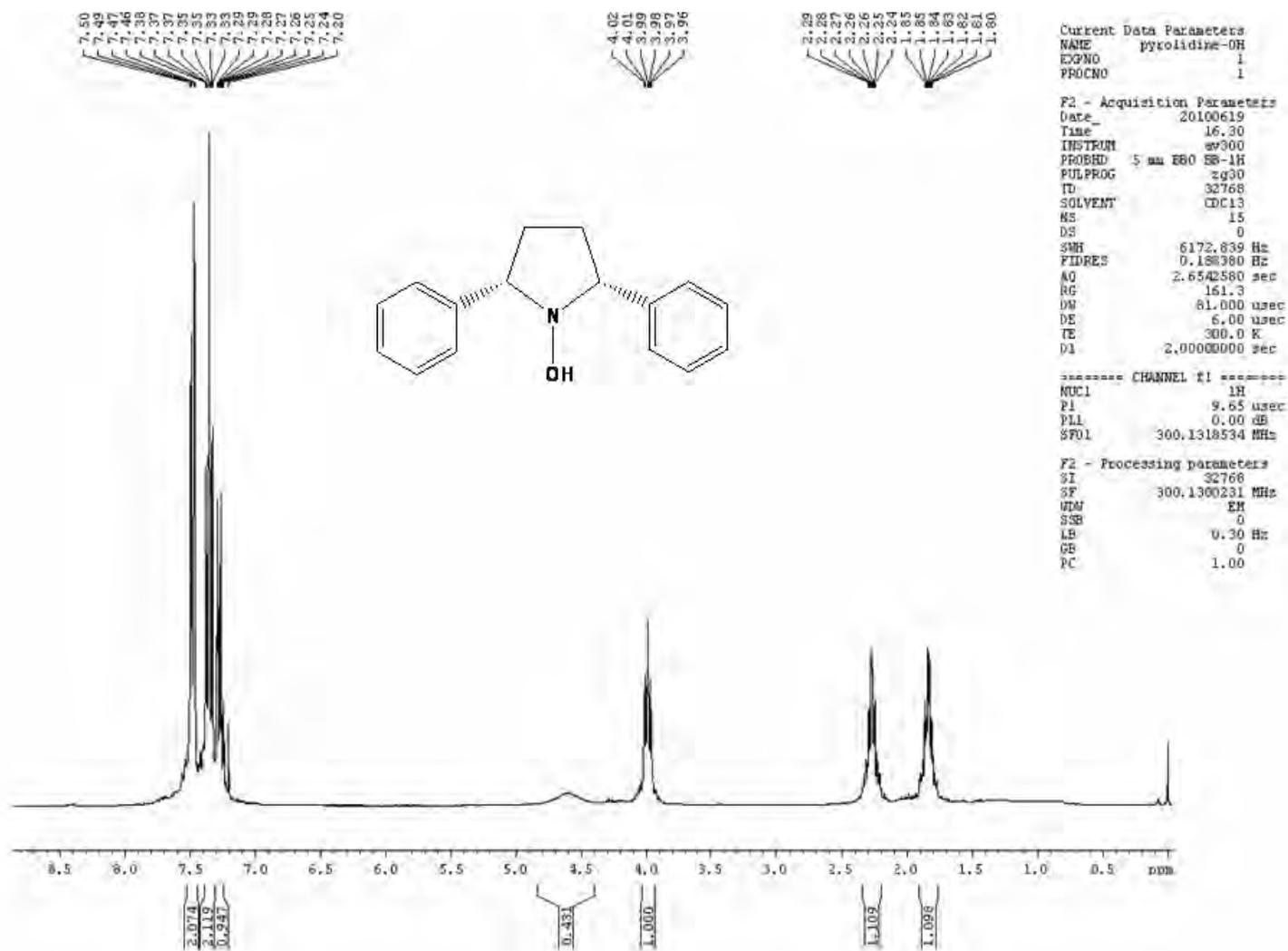
C-H COSY spectrum of **20**



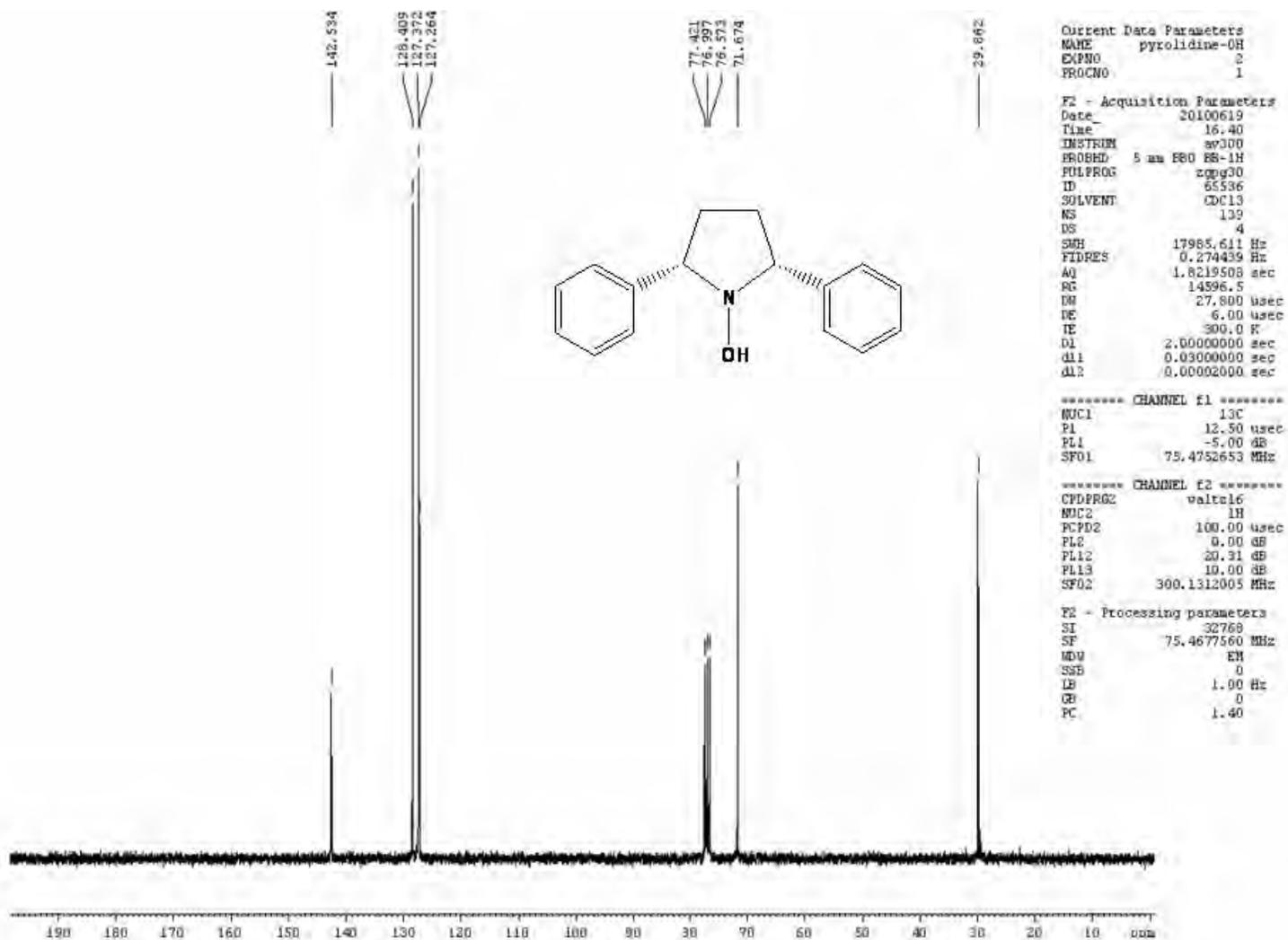
HMBC spectrum of 20



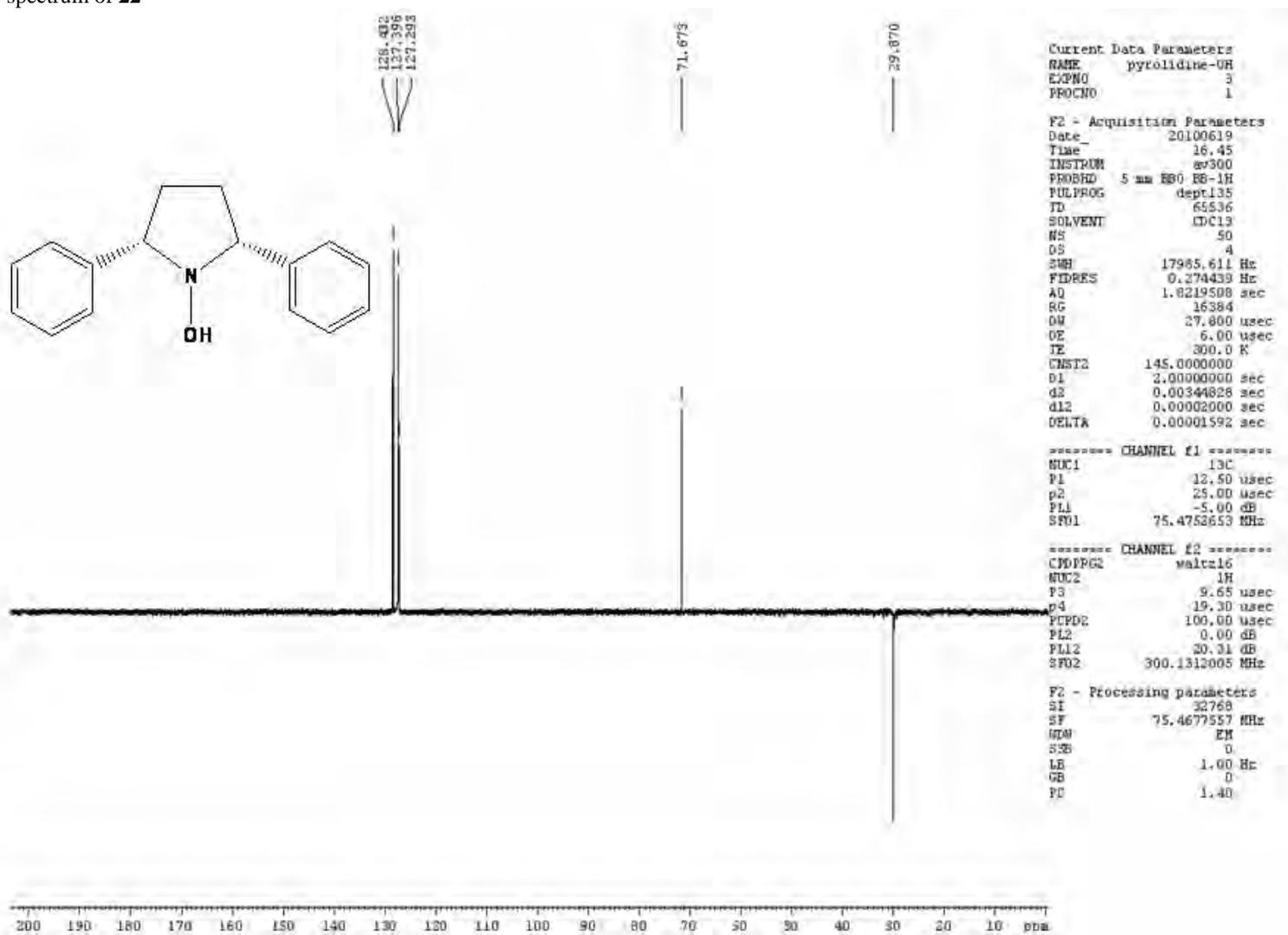
<sup>1</sup>H NMR spectrum of **22** (300 MHz, CDCl<sub>3</sub>)



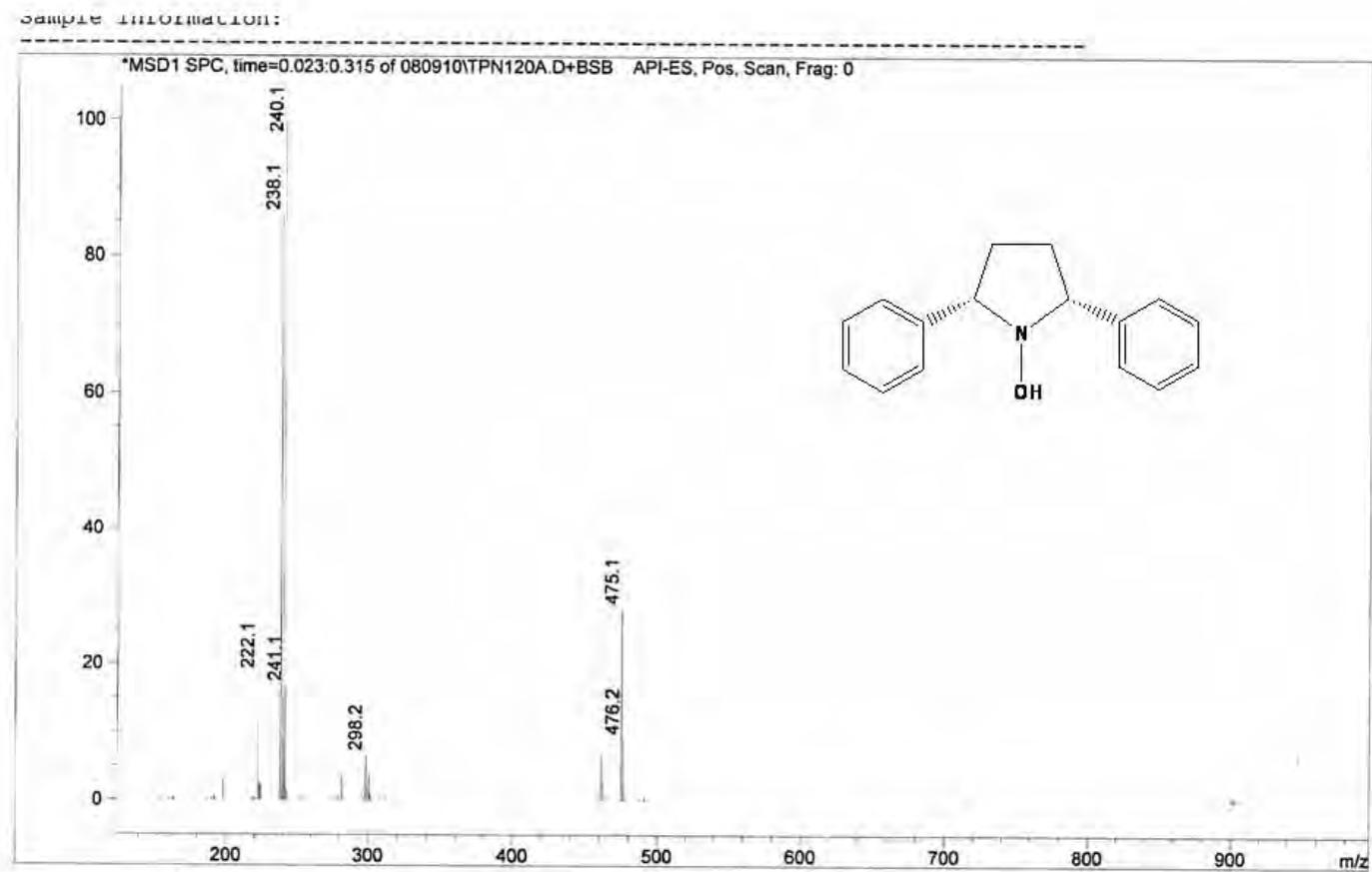
<sup>13</sup>C NMR spectrum of **22** (75 MHz, CDCl<sub>3</sub>)



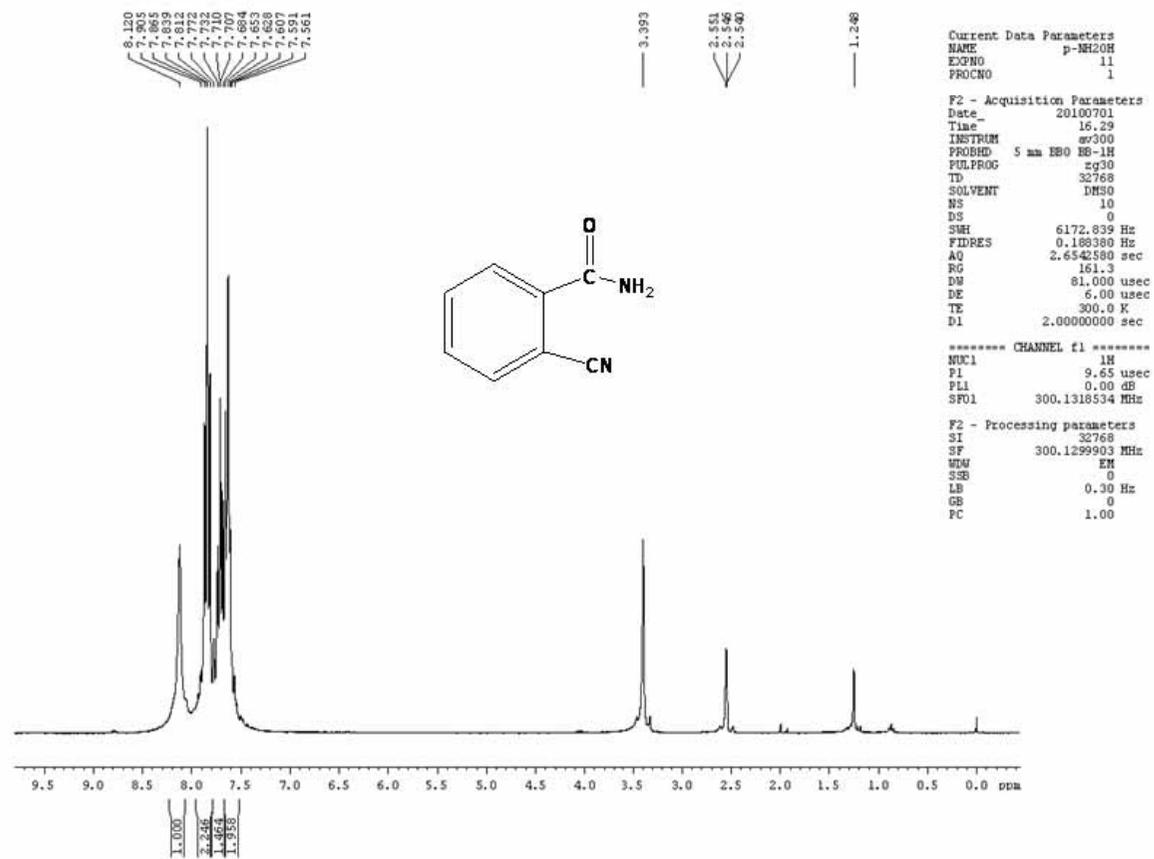
DEPT-135 spectrum of **22**



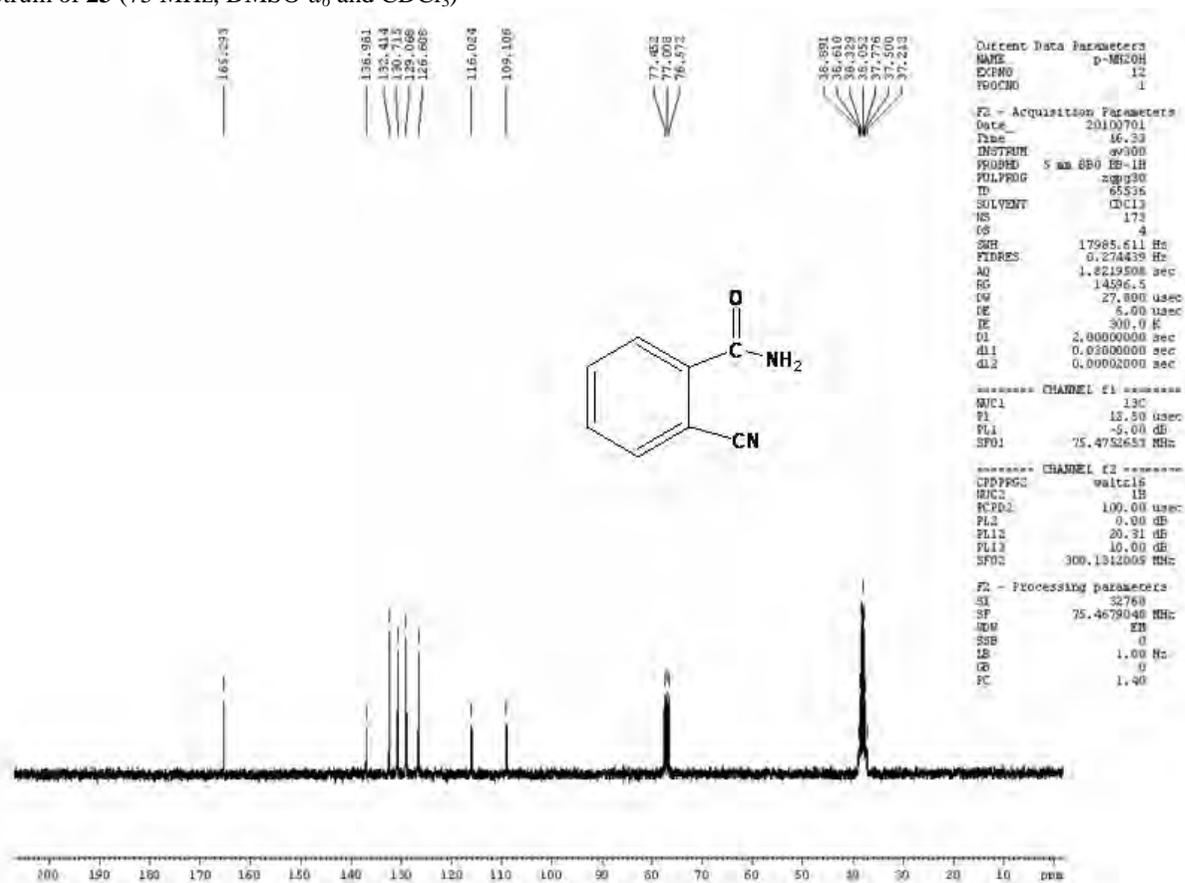
Mass spectrum of **22**



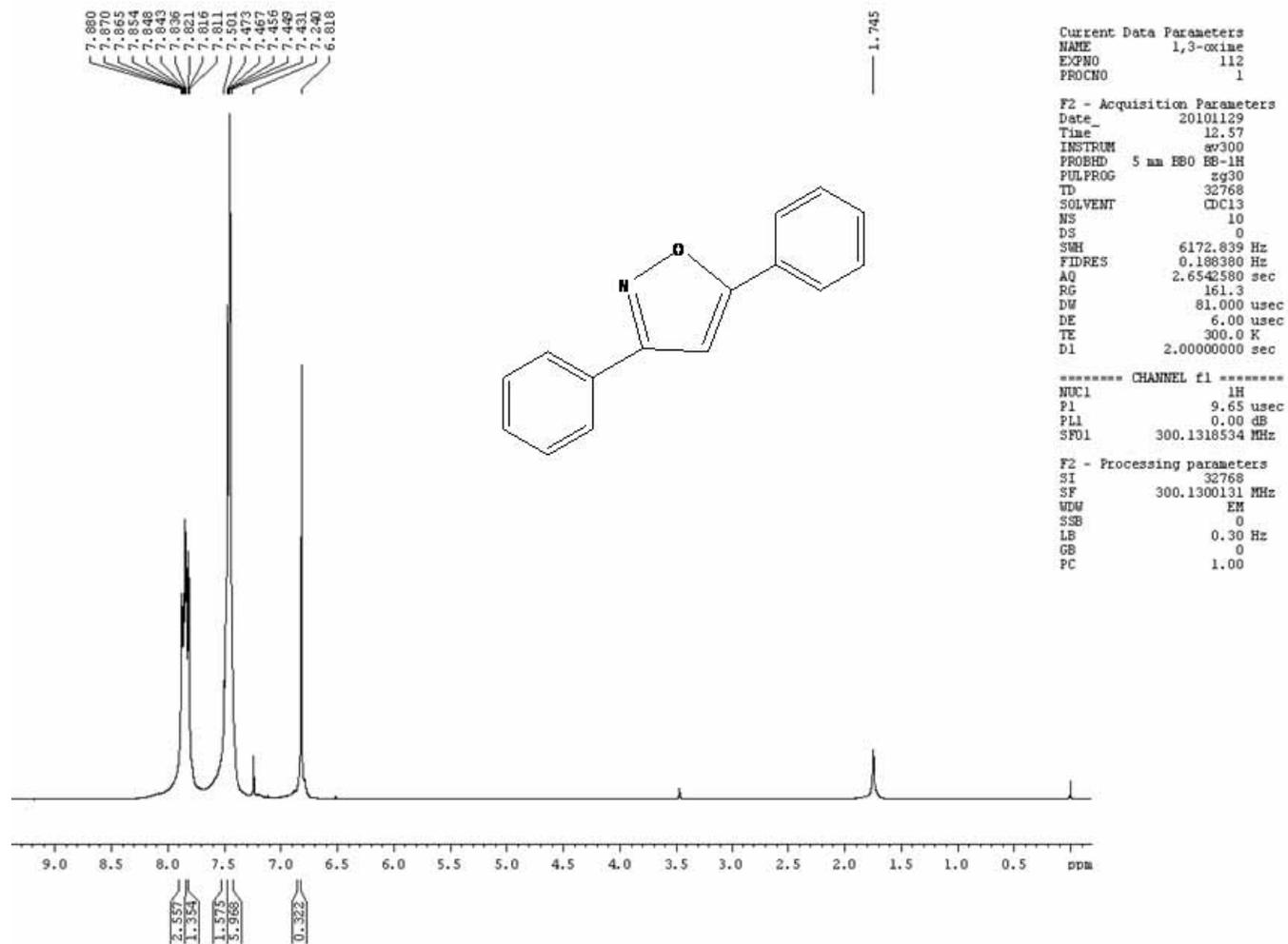
$^1\text{H}$  NMR spectrum of **23** (300 MHz,  $\text{DMSO-}d_6$  and  $\text{CDCl}_3$ )



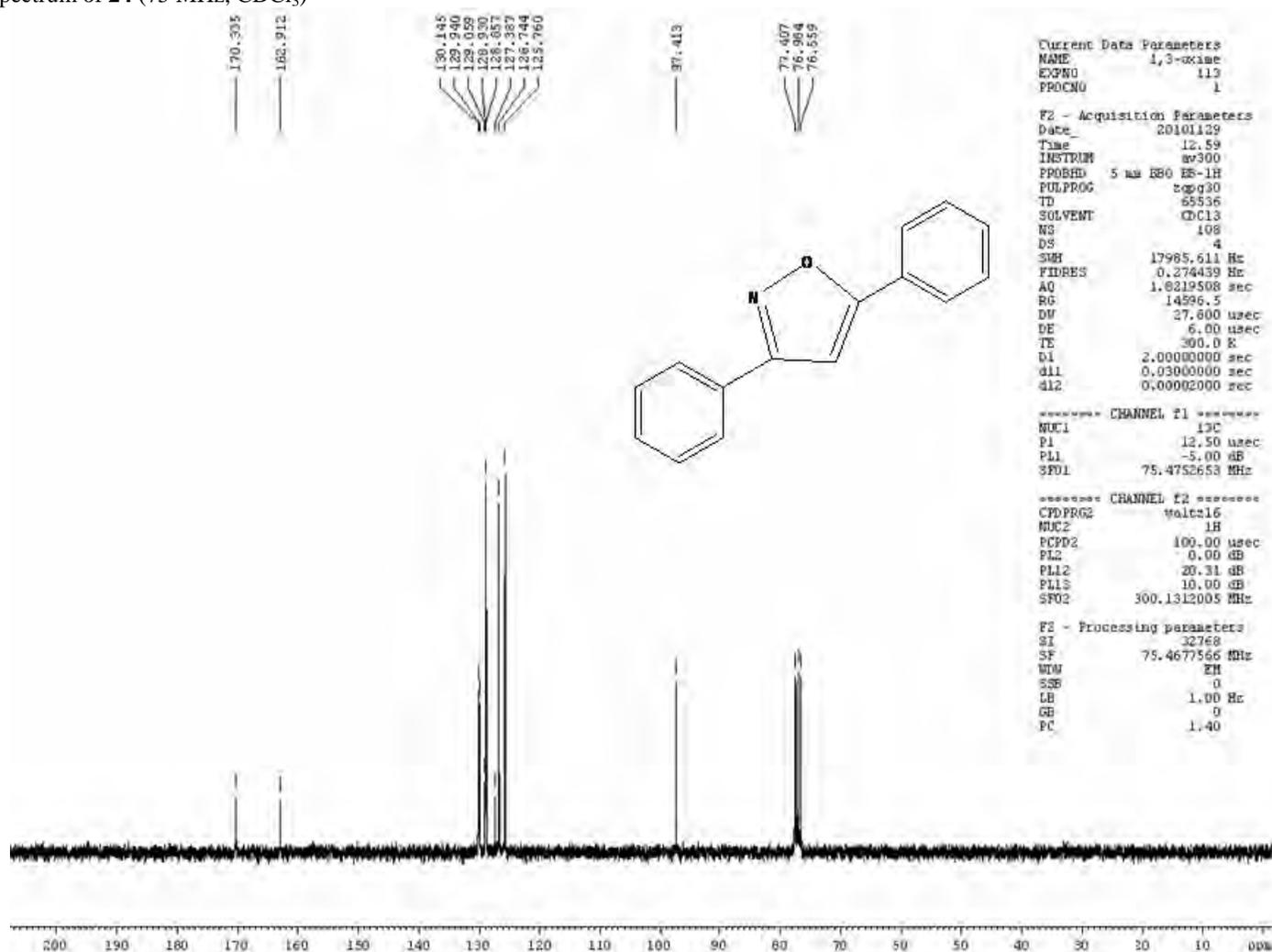
$^{13}\text{C}$  NMR spectrum of **23** (75 MHz, DMSO- $d_6$  and  $\text{CDCl}_3$ )



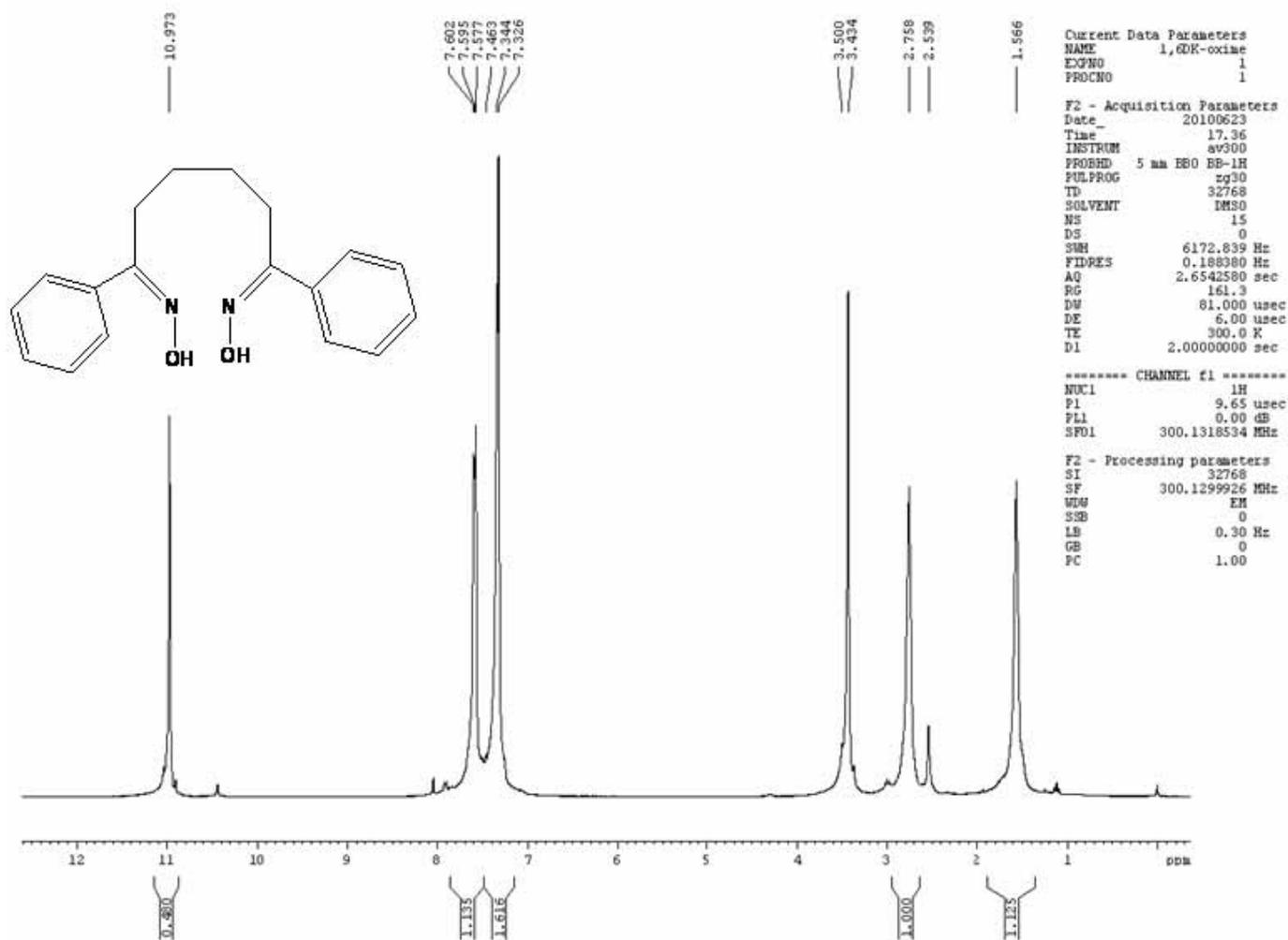
<sup>1</sup>H NMR spectrum of **24** (300 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR spectrum of **24** (75 MHz, CDCl<sub>3</sub>)



<sup>1</sup>H NMR spectrum of **25** (300 MHz, DMSO-*d*<sub>6</sub> and CDCl<sub>3</sub>)



<sup>13</sup>C NMR spectrum of **25** (75 MHz, DMSO-*d*<sub>6</sub> and CDCl<sub>3</sub>)

