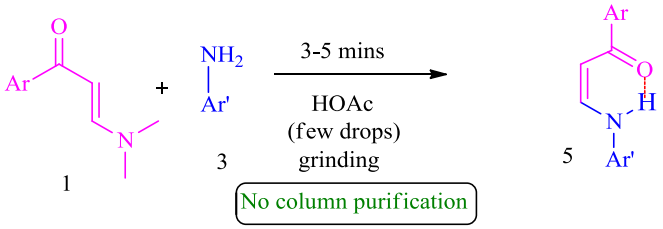
An efficient multi-component regio- and stereoselective synthesis of dihydroindeno[1,2-*b*]pyrroles under grinding

Sivasubramanian Muthusaravanan, Chinnathambi Sasikumar, Balasubramanian Devi bala and Subbu Perumal

Department of Organic Chemistry, School of Chemistry, Madurai Kamaraj University, Madurai 625 021, India

General procedure for the sequential synthesis of 5

A mixture of enaminone 1 (1mmol) and aniline 3 (1mmol) with AcOH (0.1 ml) was ground well in a pestle and mortar at room temperature for 3-5 mins. The reaction progress was monitored by thin layer chromatography. After completion of the reaction, the reaction mixture was poured into crushed ice, the resulting solid filtered off and washed with water to afford (*Z*)-3-(arylamino)-1-arylprop-2-en-1-one



Scheme 3. Synthesis of (*Z*)-1-aryl-3-(arylamino)prop-2-en-1-ones **5**

Table 3. Synthesis of (*Z*)-1-aryl-3-(arylamino)prop-2-en-1-ones s **5**

Entry	Comp.	**Ar** in **1** and **5**	**Ar'** in **3** and **5**	Time (min)	Yield of **5** (%)[a]
1	**5a**	4-MeC_6H_4	4-MeC_6H_4	4	95
2	**5b**	4-MeC_6H_4	4-ClC_6H_4	4	94
3	**5c**	C_6H_5	4-MeOC_6H_4	5	96
4	**5d**	4-MeC_6H_4	4-MeOC_6H_4	5	95
5	**5e**	4-ClC_6H_4	4-MeOC_6H_4	5	96

[a] Yield of after washing with water

(*Z*)-1-*p*-tolyl-3-(*p*-tolylamino)prop-2-en-1-one (5a) Isolated as pale yellow solid. Yield 95% m.p.=162 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 2.32 (s, 3H), 2.41 (s, 3H), 5.60 (d, 1H, *J* = 7.8 Hz), 7.00 (d, 2H, *J* = 8.7 Hz), 7.15 (d, 2H, *J* = 8.1 Hz), 7.24-7.26 (m, 2H), 7.45-7.51 (m, 1H), 7.84 (d, 2H, *J* = 8.4 Hz), 12.12 (bd, NH); ^{13}C NMR (75 MHz, $CDCl_3$) δ_c 20.7, 21.5, 93.2, 116.3, 127.3, 129.1, 130.2, 133.2, 136.6, 137.9, 141.9, 145.0, 190.6 Anal. Calcd for $C_{17}H_{17}NO$ C, 81.24; H, 6.82; N, 5.57 Found C, 81.35; H, 6.75; N, 5.44%

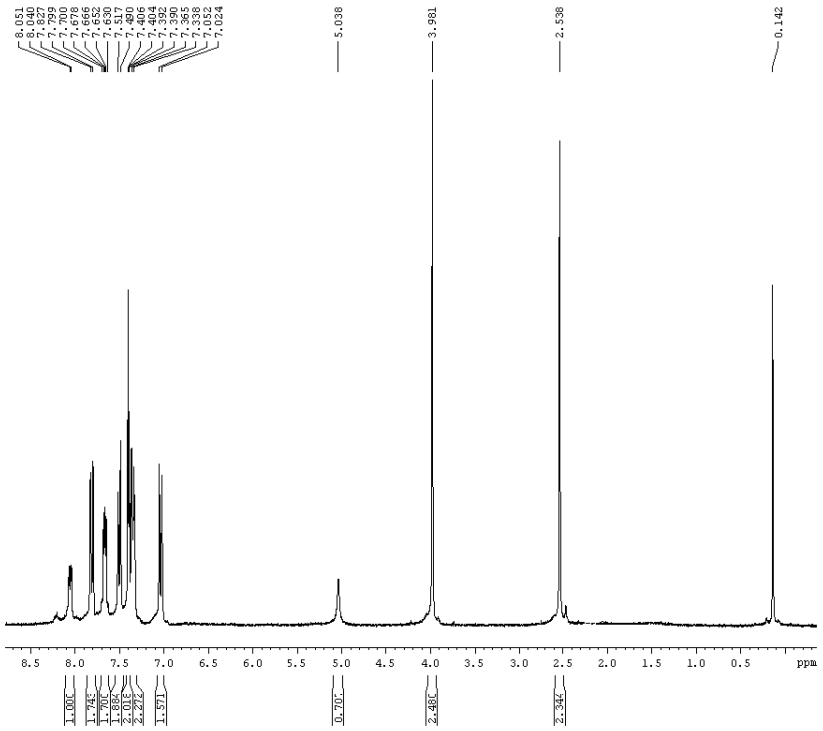
(*Z*)-3-(4-chlorophenylamino)-1-*p*-tolylprop-2-en-1-one (5b) Isolated as pale yellow solid. Yield 94% m.p.=157 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 2.41 (s, 3H), 6.02-6.05 (m, 1H), 7.01-7.04 (m, 2H), 7.24-7.32 (m, 4H), 7.39-7.46 (m, 1H), 7.84 (d, 2H, *J* = 8.1 Hz), 12.10 (bd, NH); ^{13}C NMR (75 MHz, $CDCl_3$) δ_c 21.5, 94.3, 117.4, 127.4, 128.5, 129.2, 129.7, 136.4, 139.1, 142.3, 144.1, 191.1 Anal. Calcd for $C_{16}H_{14}ClNO$ C, 70.72; H, 5.19; N, 5.15 Found C, 70.81; H, 5.25; N, 5.26%

(*Z*)-3-(*p*-methoxyphenylamino)-1-phenylprop-2-en-1-one (5c) Isolated as pale yellow solid. Yield 96% m.p.= 150ºC^{1}H NMR (300 MHz, $CDCl_3$) δ_H 3.80 (s, 3H), 5.98 (d, 1H, *J* = 7.8 Hz), 6.90 (d, 2H, *J* = 8.7 Hz), 7.06 (d, 1H, *J* = 8.7 Hz), 7.41-7.50 (m, 4H), 7.92-7.95 (m, 2H); 12.20 (bd, NH); ^{13}C NMR (75 MHz, $CDCl_3$) δ_c 55.0,92.4, 114.5, 117.4, 126.7, 127.9, 130.8, 133.3, 138.8, 145.3, 155.9, 190.0 Anal. Calcd for $C_{16}H_{15}NO_2C$, 75.87; H, 5.97; N, 5.53 Found C, 75.75; H, 6.04; N, 5.47%

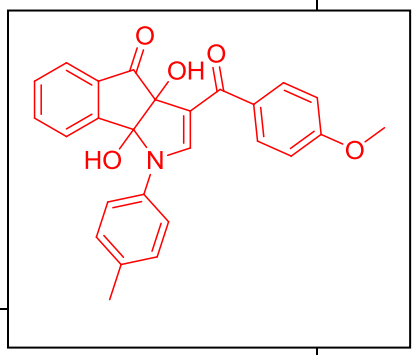
(*Z*)-3-(*p*-methoxyphenylamino)-1-*p*-tolylprop-2-en-1-one (5d) Isolated as pale yellow solid. Yield 95% m.p.=144 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 2.41 (s, 3H), 3.80 (s, 3H), 5.97 (d, 1H, *J* = 7.5 Hz,), 6.88-6.91 (m, 2H), 7.04-7.07 (m, 2H), 7.25 (d, 2H, *J* =7.2 Hz), 7.39-7.45 (m, 1H), 7.84 (d, 2H, *J* = 8.4 Hz); 12.16 (bd, NH); ^{13}C NMR (75 MHz, $CDCl_3$) δ_c 21.5, 55.5, 92.8, 115.0, 117.8, 127.3, 129.1, 133.9, 136.7, 141.9, 145.5, 156.3, 190.4 Anal. Calcd for $C_{17}H_{17}NO_2C$, 76.38; H, 6.41; N, 5.24 Found C, 76.47; H, 6.52; N, 5.15%

(*Z*)-1-(*p*-chlorophenyl)-3-(*p*-methoxyphenylamino)prop-2-en-1-one (5e) Isolated as pale yellow solid. Yield 96% m.p.= 153 ºC ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 3.81 (s, 3H), 5.92 (d,

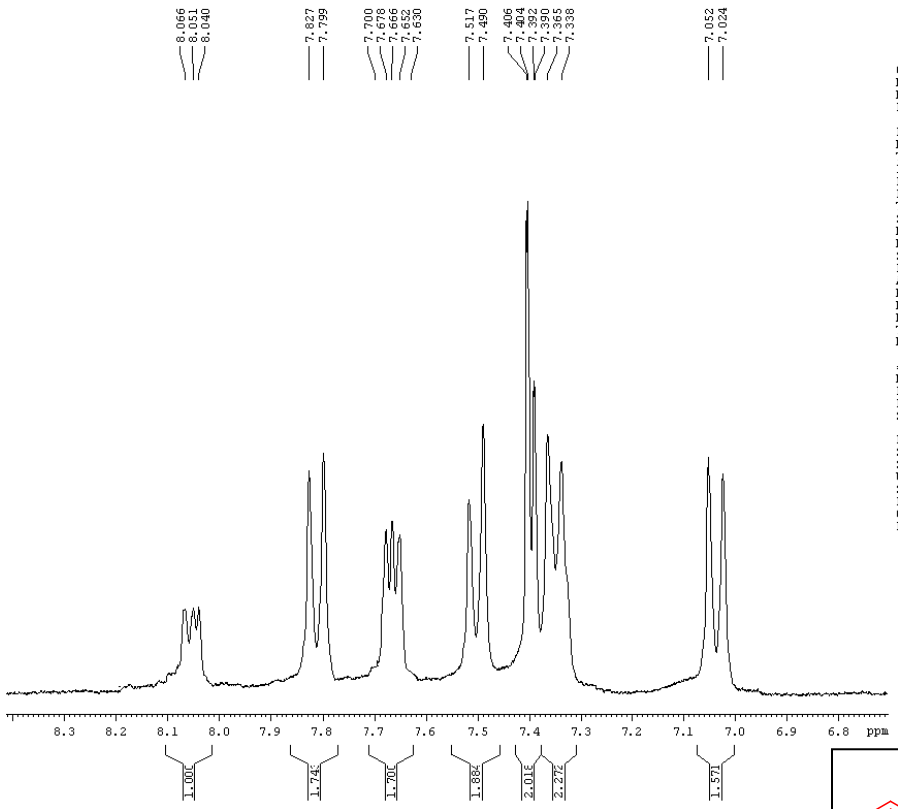
1H, *J* = 7.5 Hz,), 6.88- 6.92 (m, 2H),7.04-7.08 (m, 2H), 7.39-7.48 (m, 3H), 7.85-7.88 (m, 2H), 12.19 (bd, NH); ^{13}C NMR (75 MHz, $CDCl_3$) δ_c 55.6, 92.6, 115.0, 118.0, 128.6, 133.6, 137.5, 137.7, 146.2, 156.5, 189.0 Anal. Calcd for $C_{16}H_{14}ClNO_2$ C, 66.79; H, 4.90; N, 4.87 Found C, 66.67; H, 4.83; N, 4.80%



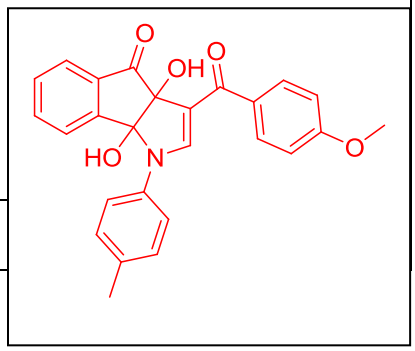

```
Current Data Parameters
NAME      4-ome ek 4-me nin2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131007
Time               0.17
INSTRUM           av300
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   14
DS                    0
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ             2.6542580 sec
RG                456.1
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                9.65 usec
PL1               0.00 dB
SF01       300.1318534 MHz

F2 - Processing parameters
SI                32768
SF         300.1299644 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



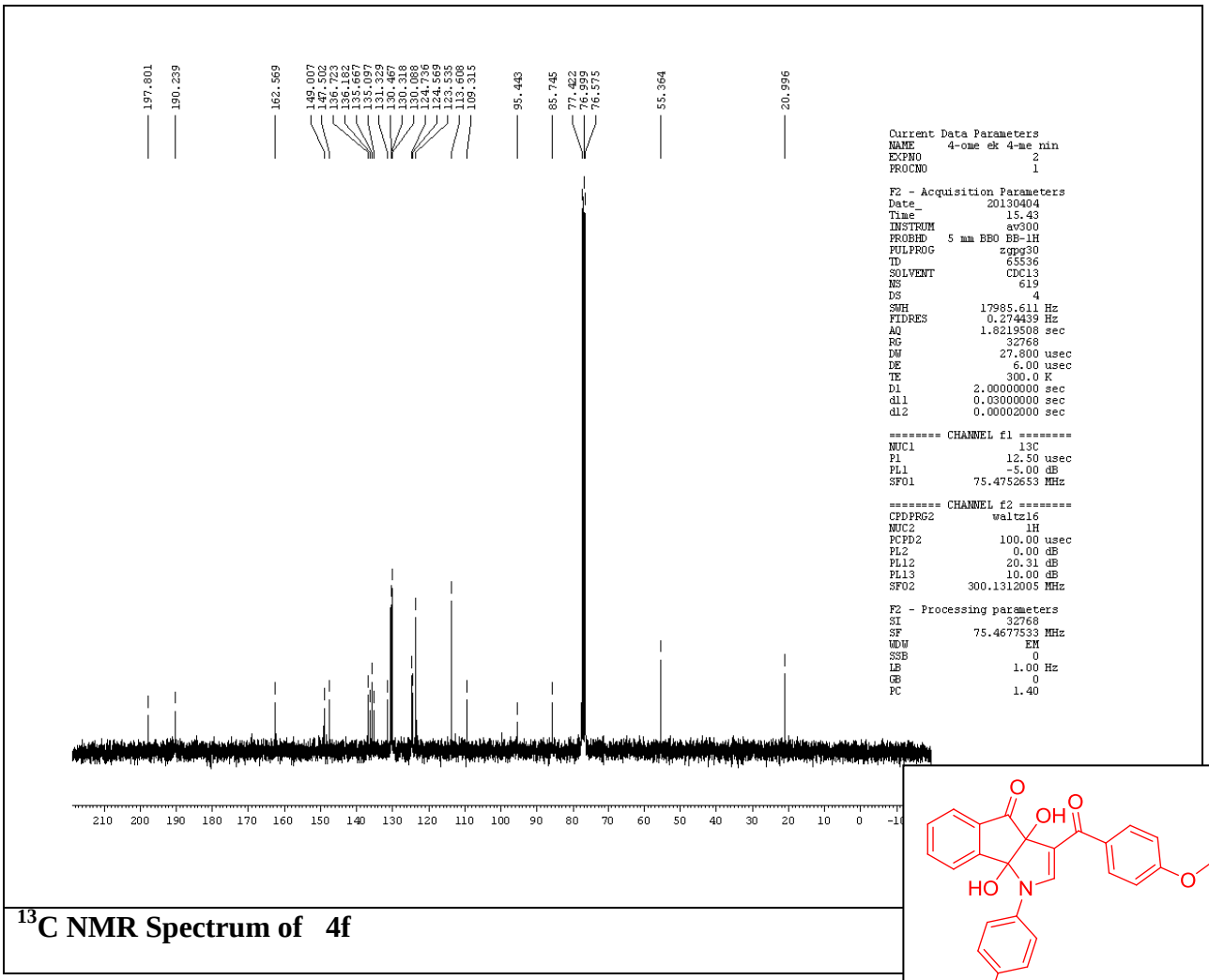
^{1}H NMR Spectrum of 4f



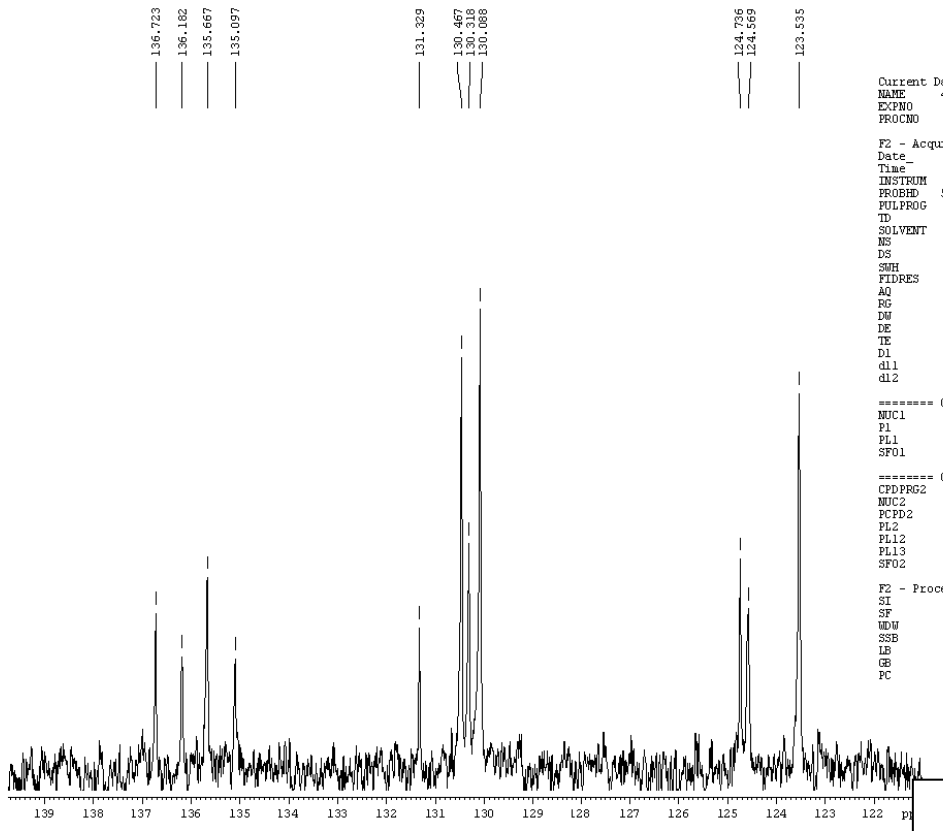

```
Current Data Parameters
NAME      4-ome ek 4-me nin2
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20131007
Time                   0.17
INSTRUM               av300
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       14
DS                        0
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                    456.1
DW                 81.000 usec
DE                  6.00 usec
TE                    300.0 K
D1             2.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.65 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1299644 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



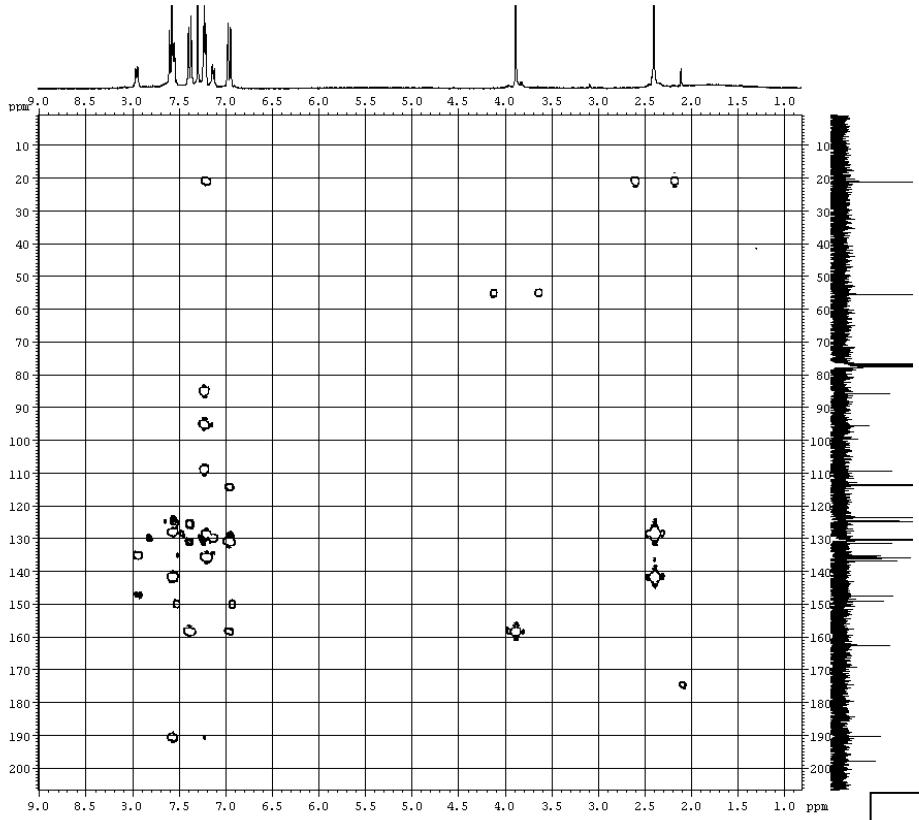
^{1}H NMR Spectrum of 4f



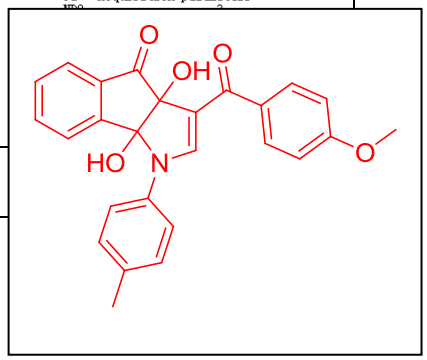
^{13}C NMR Spectrum of 4f



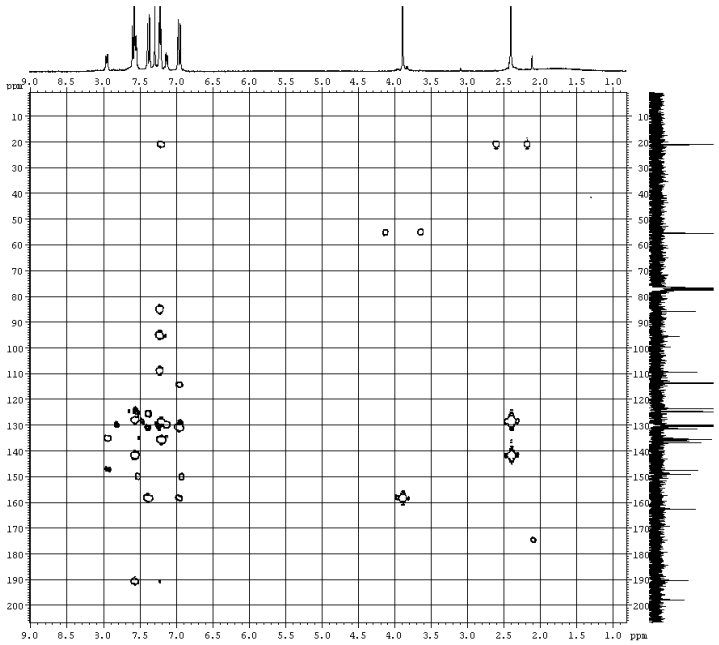
^{13}C NMR Spectrum of 4f

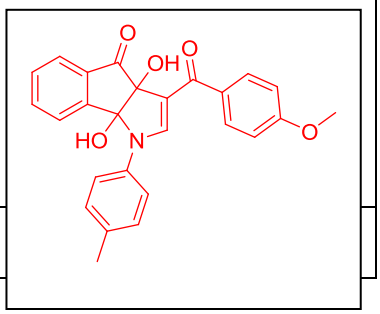


```
Current Data Parameters
NAME      4-ome ek 4-me nin
EXPNO                   55
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130408
Time                 20.01
INSTRUM              av300
PROBHD    5 mm BBO BB-1H
PULPROG  inv4gp1p1rndqf
TD                   1024
SOLVENT              CDCl3
NS                       8
DS                      16
SWH              3360.215 Hz
FIDRES           3.281460 Hz
AQ               0.1524212 sec
RG                   16384
DW                148.800 usec
DE                  6.00 usec
TE                300.0 K
CNST2        145.0000000
d0           0.00000300 sec
D1           1.47521901 sec
d2           0.00344828 sec
D6           0.06500000 sec
d13          0.00000400 sec
D16          0.00015000 sec
IN0          0.00002990 sec
MCREST       0.00000000 sec
MCWRK        1.47521901 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.65 usec
p2                  19.30 usec
PL1                  0.00 dB
SFO1         300.1314236 MHz

======== CHANNEL f2 ========
NUC2                  13C
P3                  12.50 usec
PL2                 -5.00 dB
SFO2          75.4752239 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPNAM3           SINE.100
GPX1                 0.00 %
GPX2                 0.00 %
GPX3                 0.00 %
GPY1                 0.00 %
GPY2                 0.00 %
GPY3                 0.00 %
GPZ1                50.00 %
GPZ2                30.00 %
GPZ3                40.10 %
P16               1500.00 usec

F1 - Acquisition parameters
```

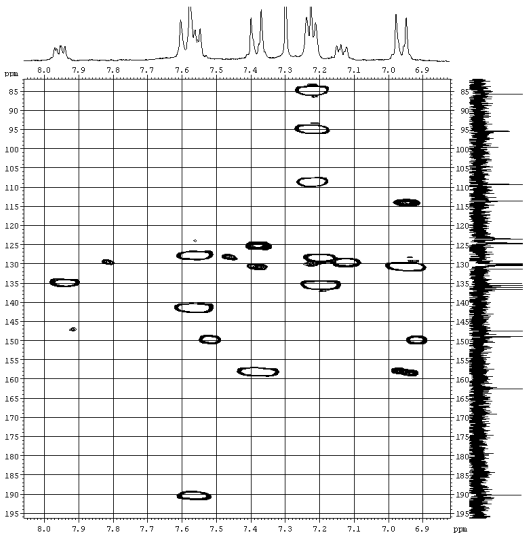


HMBC Spectrum of 4f

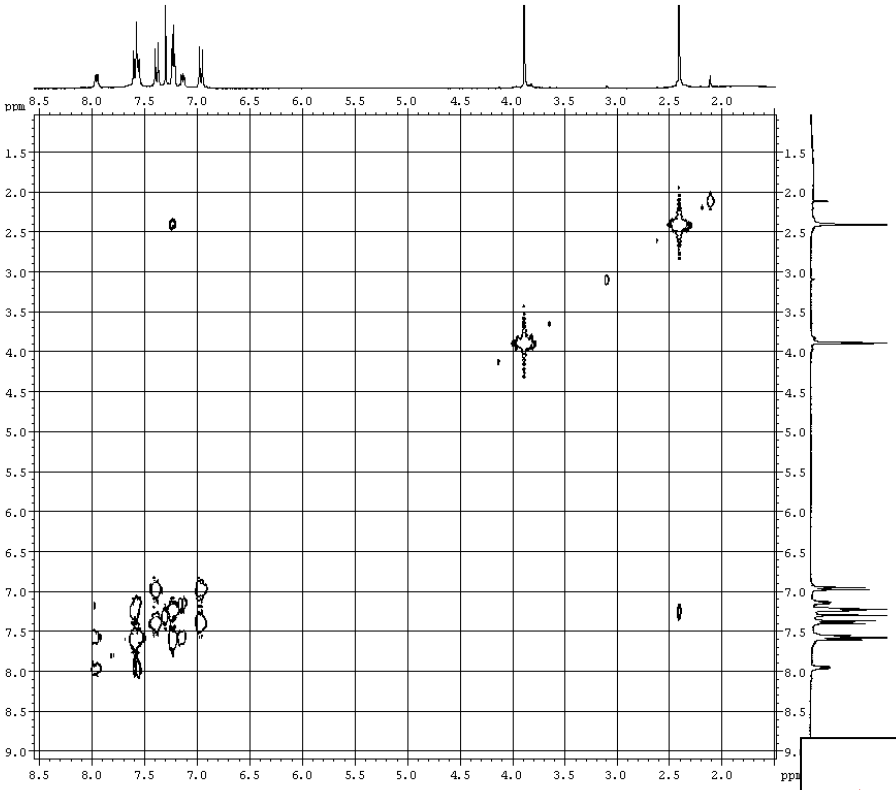




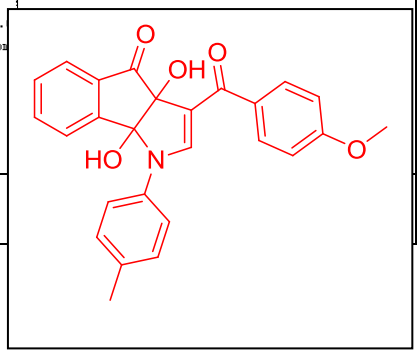
HMBC(expanded) Spectrum of 4f



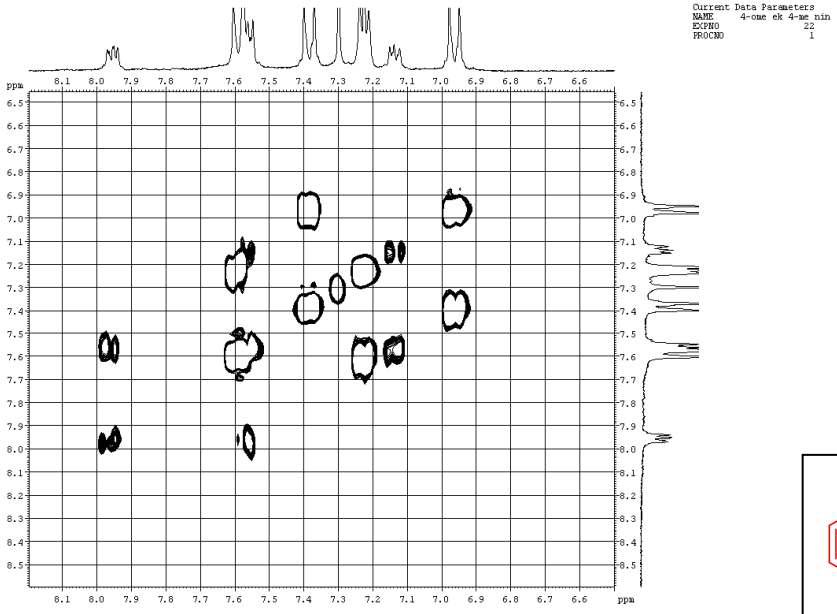
HMBC(expanded) Spectrum of 4f

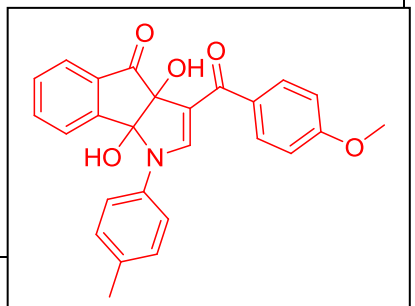


```
Current Data Parameters
NAME        4-ome ek 4-me nin
EXPNO                    22
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130408
Time                 18.45
INSTRUM              av300
PROBHD   5 mm BBO BB-1H
PULPROG          cosygpqf
TD                   2048
SOLVENT              CDCl3
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
IN0            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
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
```

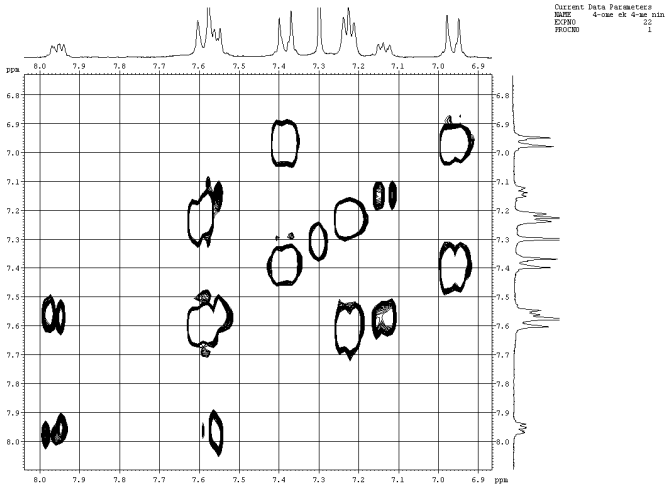


H, H- COSY Spectrum of 4f

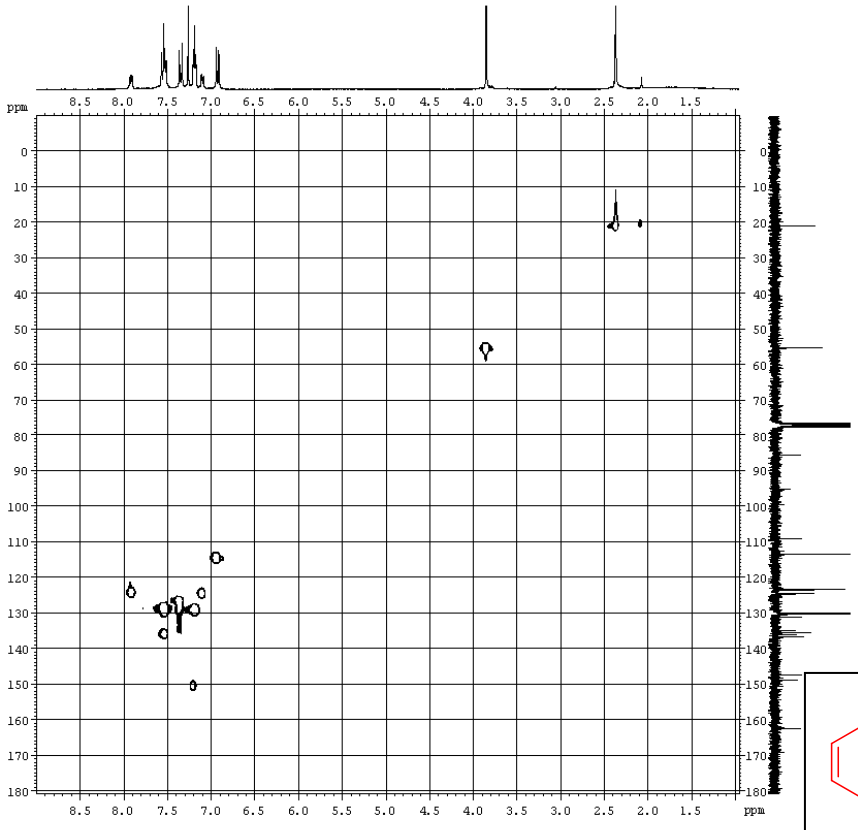




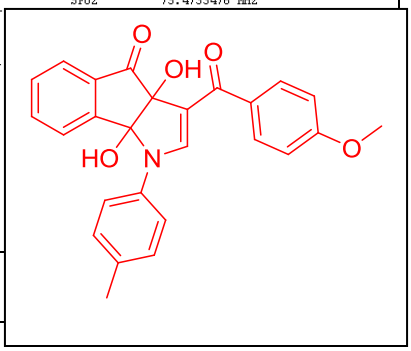
H, H- COSY(expanded) Spectrum of 4f



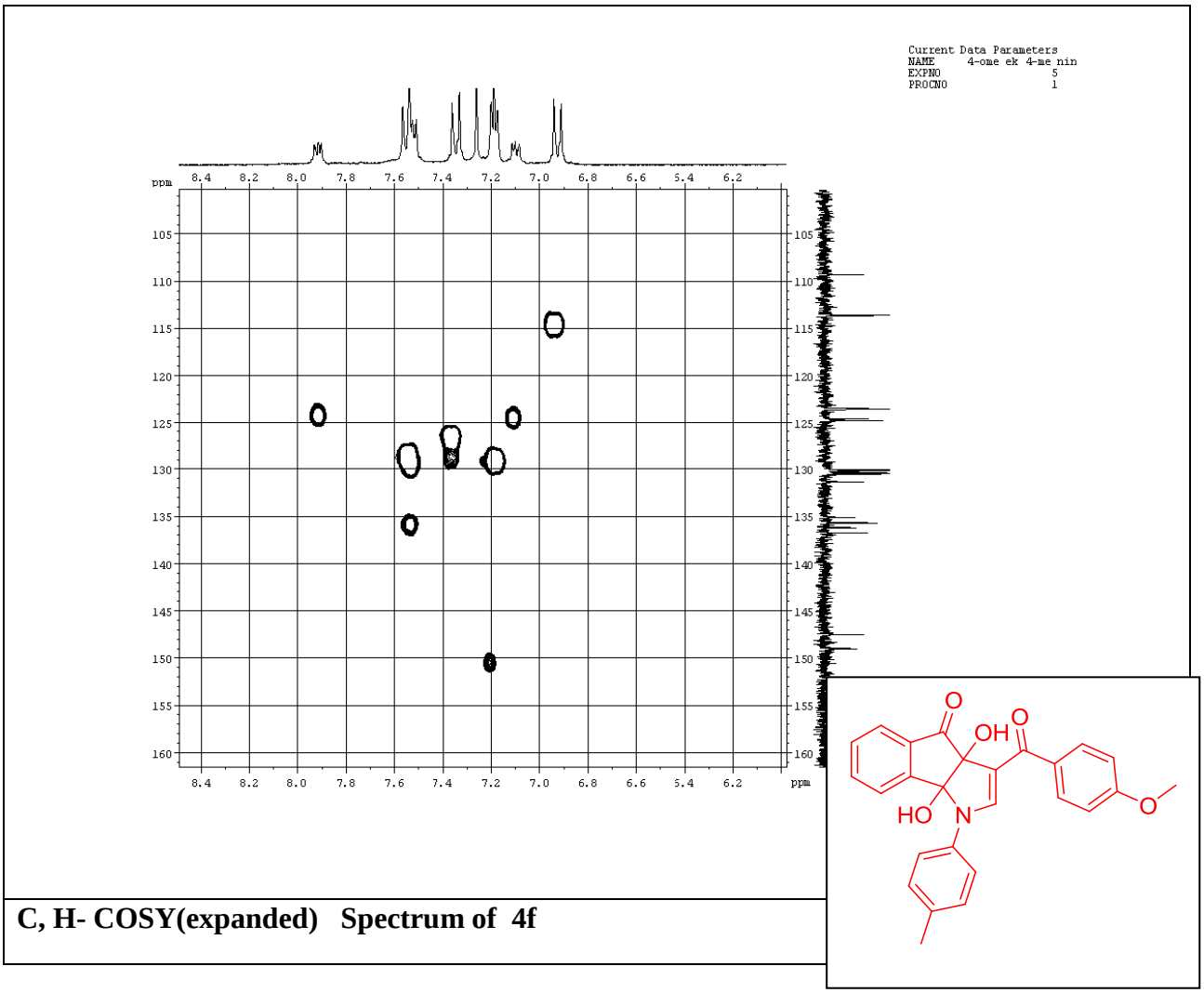
H, H- COSY(expanded) Spectrum of 4f



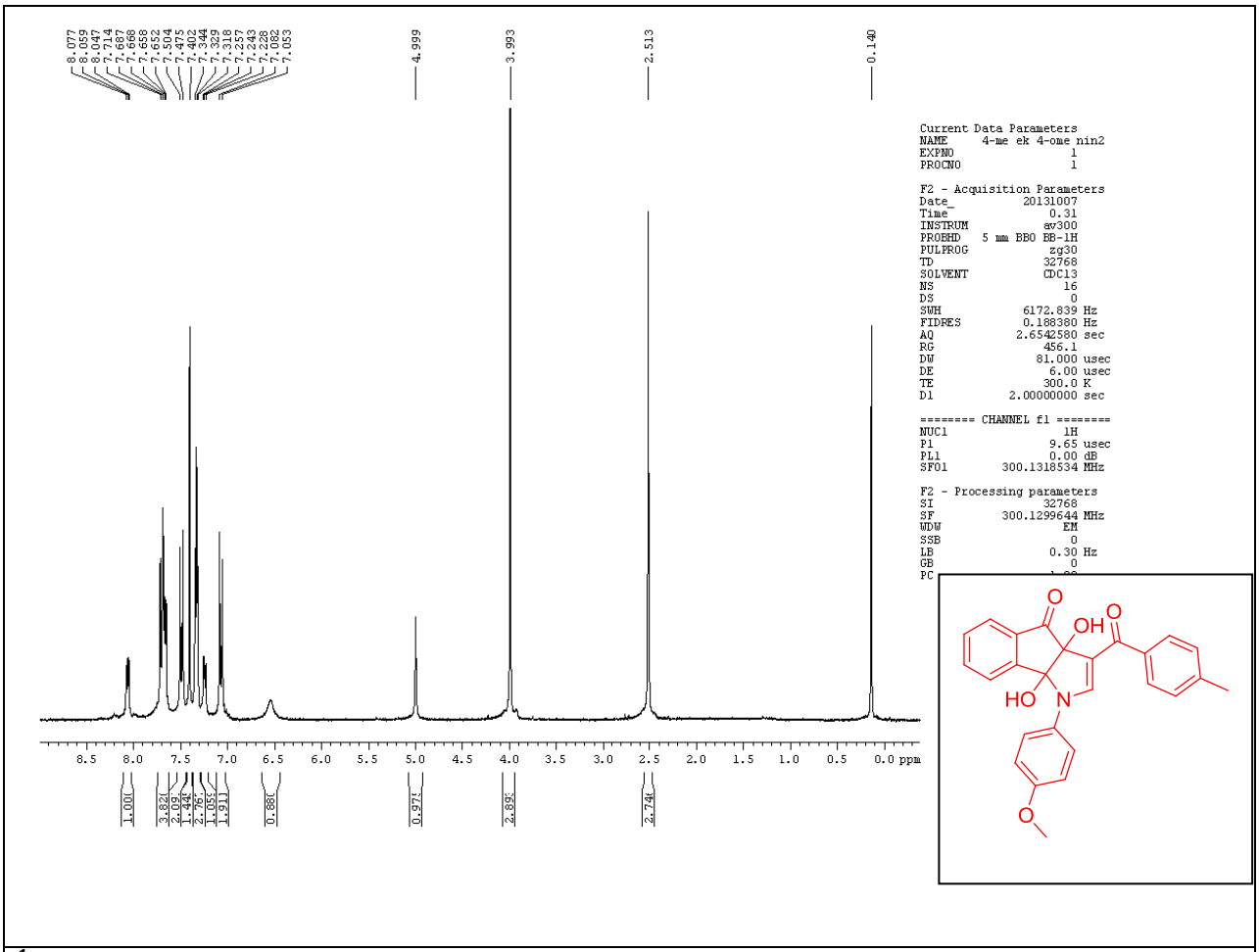
```
Current Data Parameters
NAME      4-ome ek 4-me nin
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_            20130409
Time                10.41
INSTRUM             av300
PROBHD    5 mm BBO BB-1H
PULPROG          invietgp
TD                   1024
SOLVENT             CDCl3
NS                      4
DS                     16
SWH              2997.602 Hz
FIDRES           2.927346 Hz
AQ               0.1708532 sec
RG                    2048
DW                166.800 usec
DE                  6.00 usec
TE                 300.0 K
CNST2           140.0000000
d0               0.00000300 sec
D1               2.50000000 sec
d4               0.00178571 sec
d11              0.03000000 sec
d13              0.00000400 sec
D16              0.00015000 sec
DELTA            0.00167530 sec
DELTA1           0.00027771 sec
INO              0.00003011 sec
MCREST           0.00000000 sec
MCWRK            0.50000000 sec
ST1CNT                  128

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.65 usec
p2                   19.30 usec
P28                   0.50 usec
PL1                   0.00 dB
SFO1          300.1313806 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
SFO2           75.4733476 MHz
```



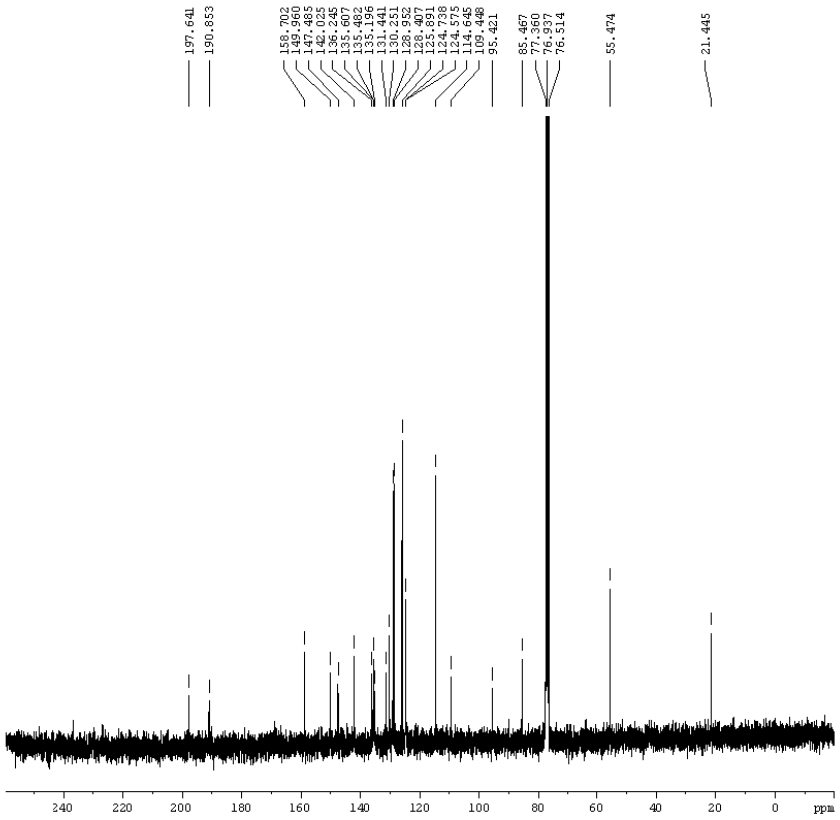
C, H- COSY Spectrum of 4f



C, H- COSY(expanded) Spectrum of 4f

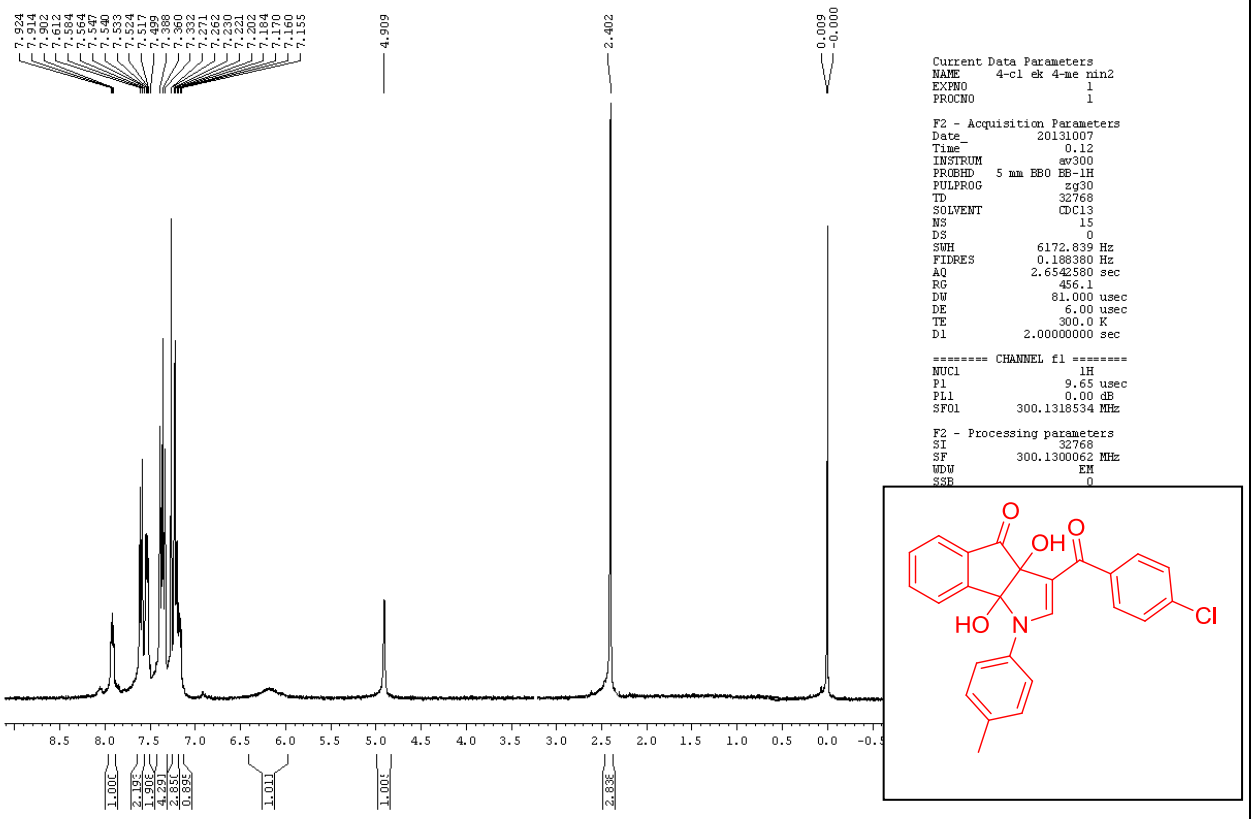


^{1}H NMR Spectrum of 4a

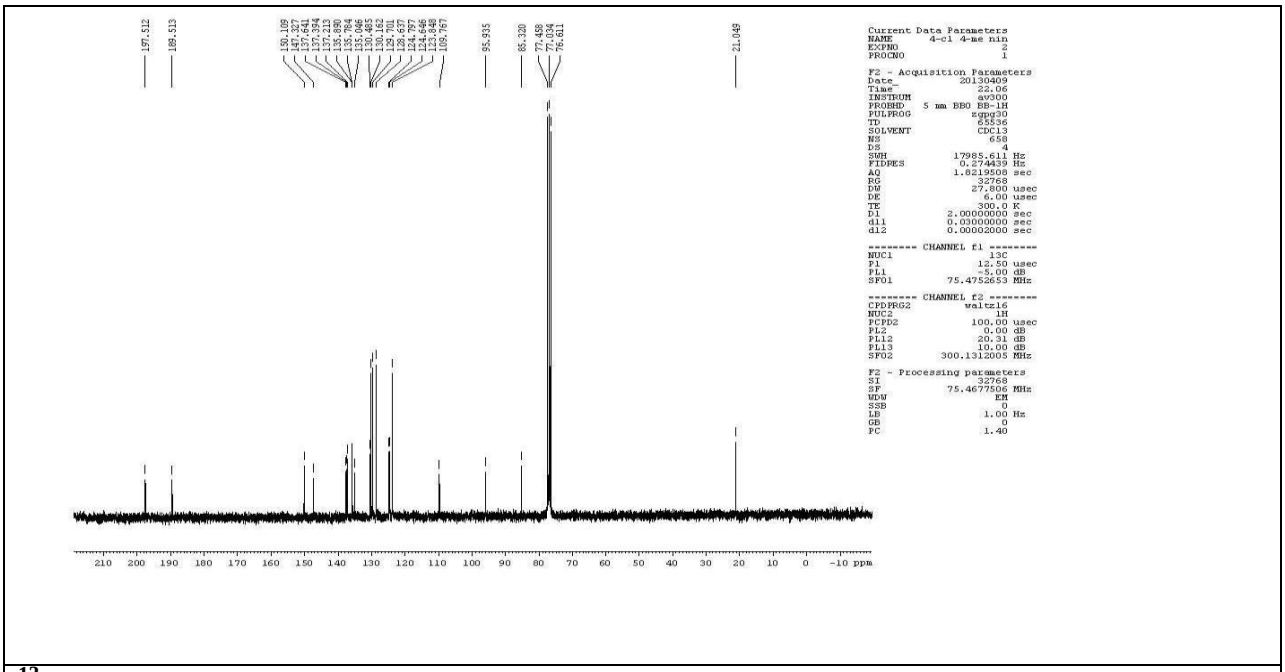



```
Current Data Parameters
NAME                 nin-4a
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130122
Time                 23.05
INSTRUM              av300
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1411
DS                        4
SWH             21097.047 Hz
FIDRES          0.321915 Hz
AQ              1.5532532 sec
RG                    32768
DW                 23.700 usec
DE                  6.00 usec
TE                 300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                -5.00 dB
SFO1         75.4767751 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 0.00 dB
PL12               20.31 dB
PL13               10.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF            75.4677533 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                  1.40
```

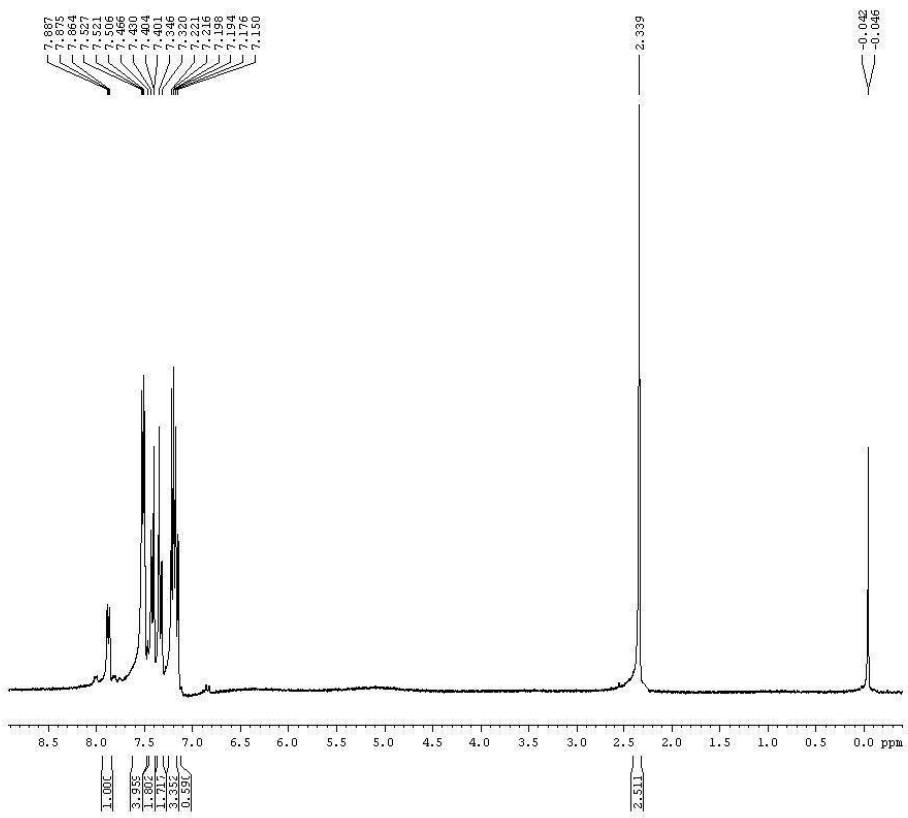
^{13}C NMR Spectrum of 4a



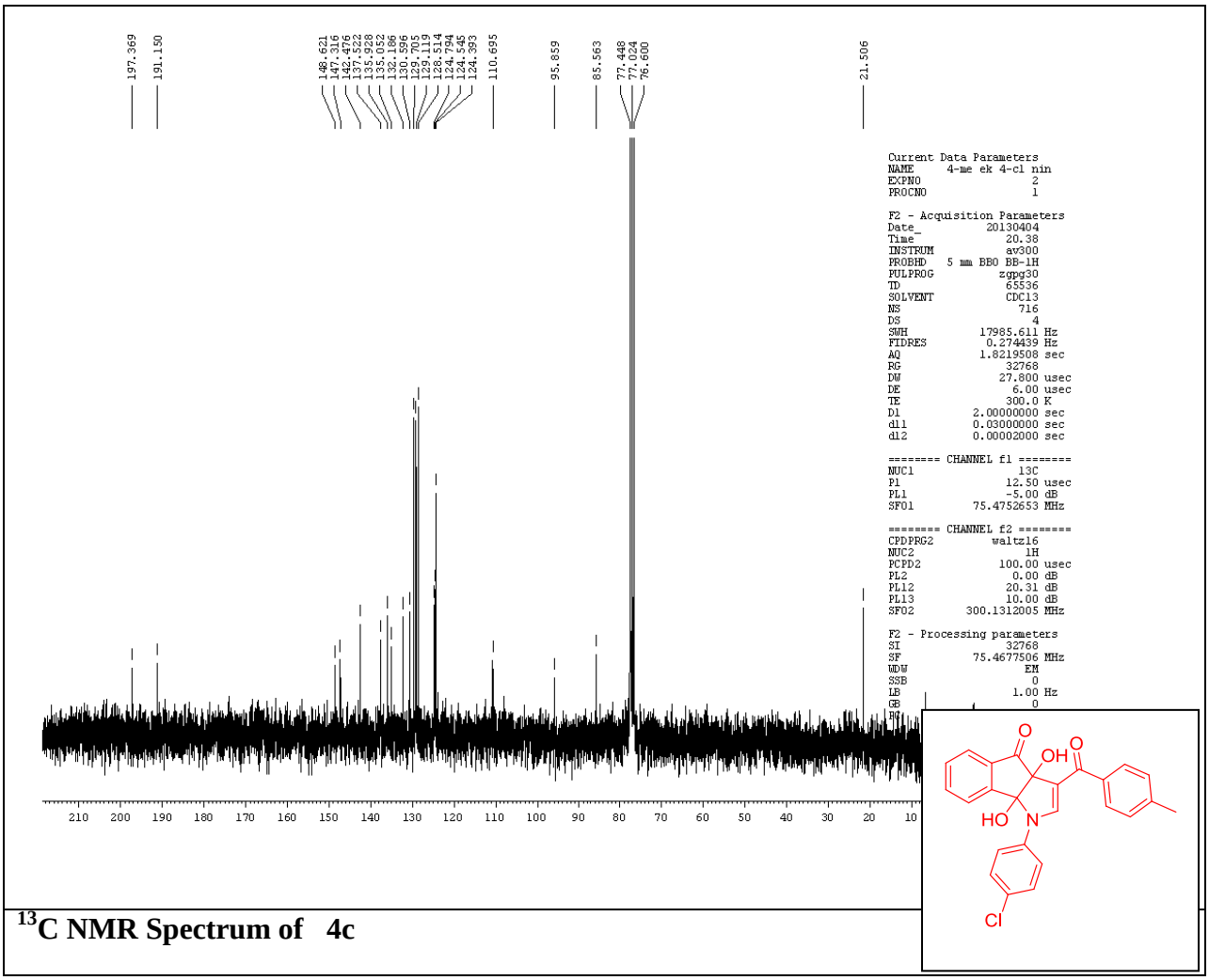
^{1}H NMR Spectrum of 4b



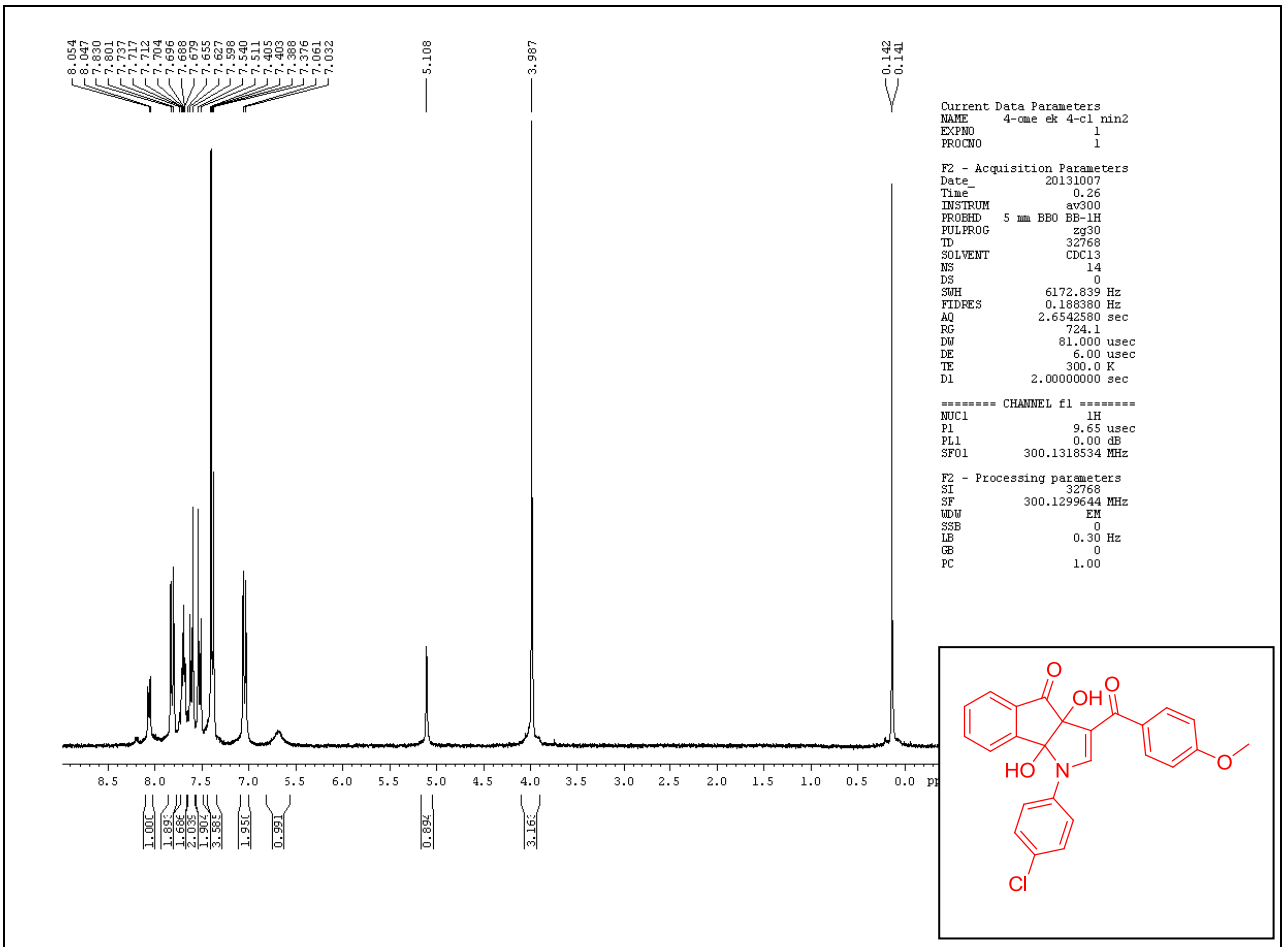
^{13}C NMR Spectrum of 4b



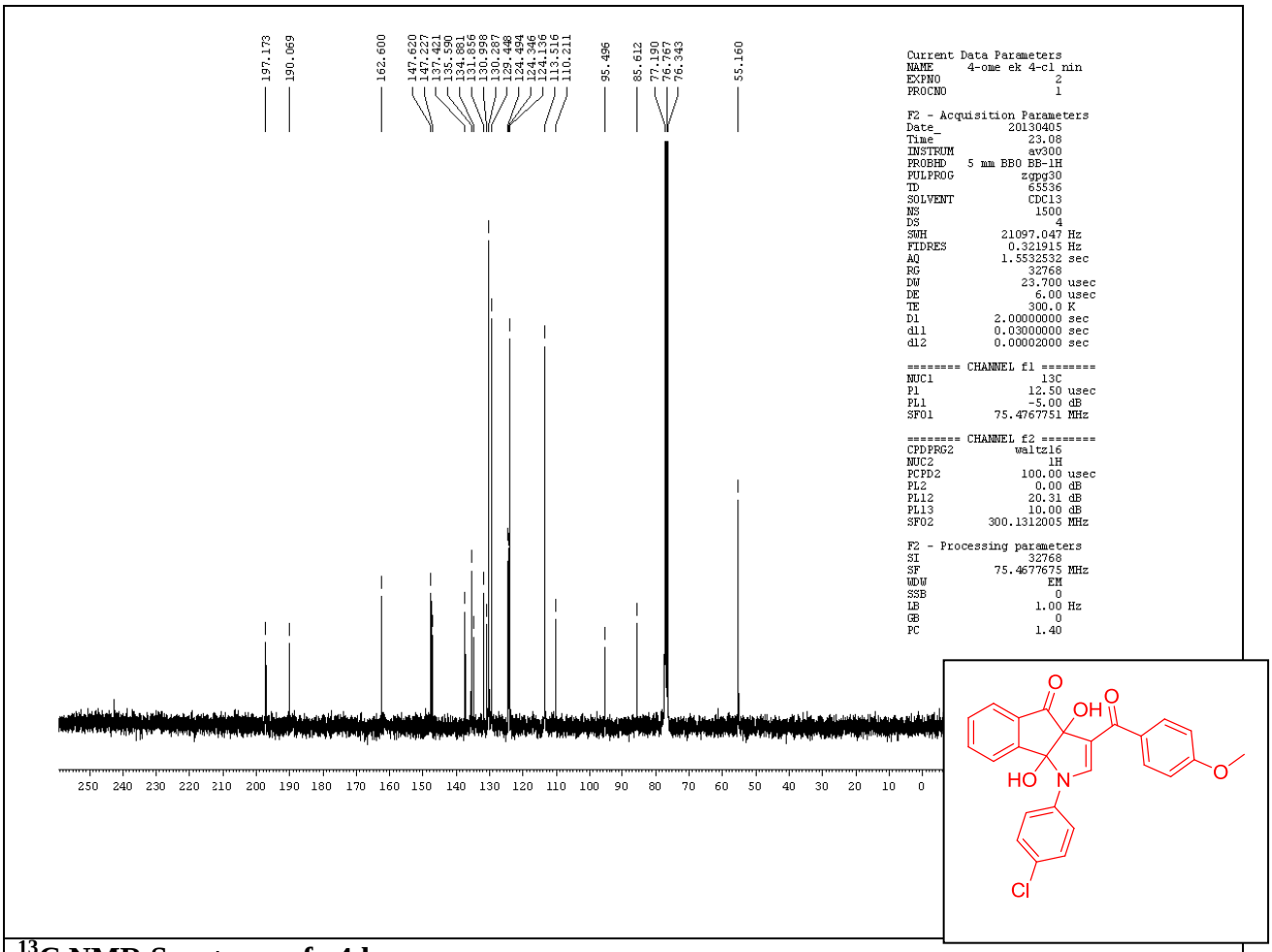
^{1}H NMR Spectrum of 4c



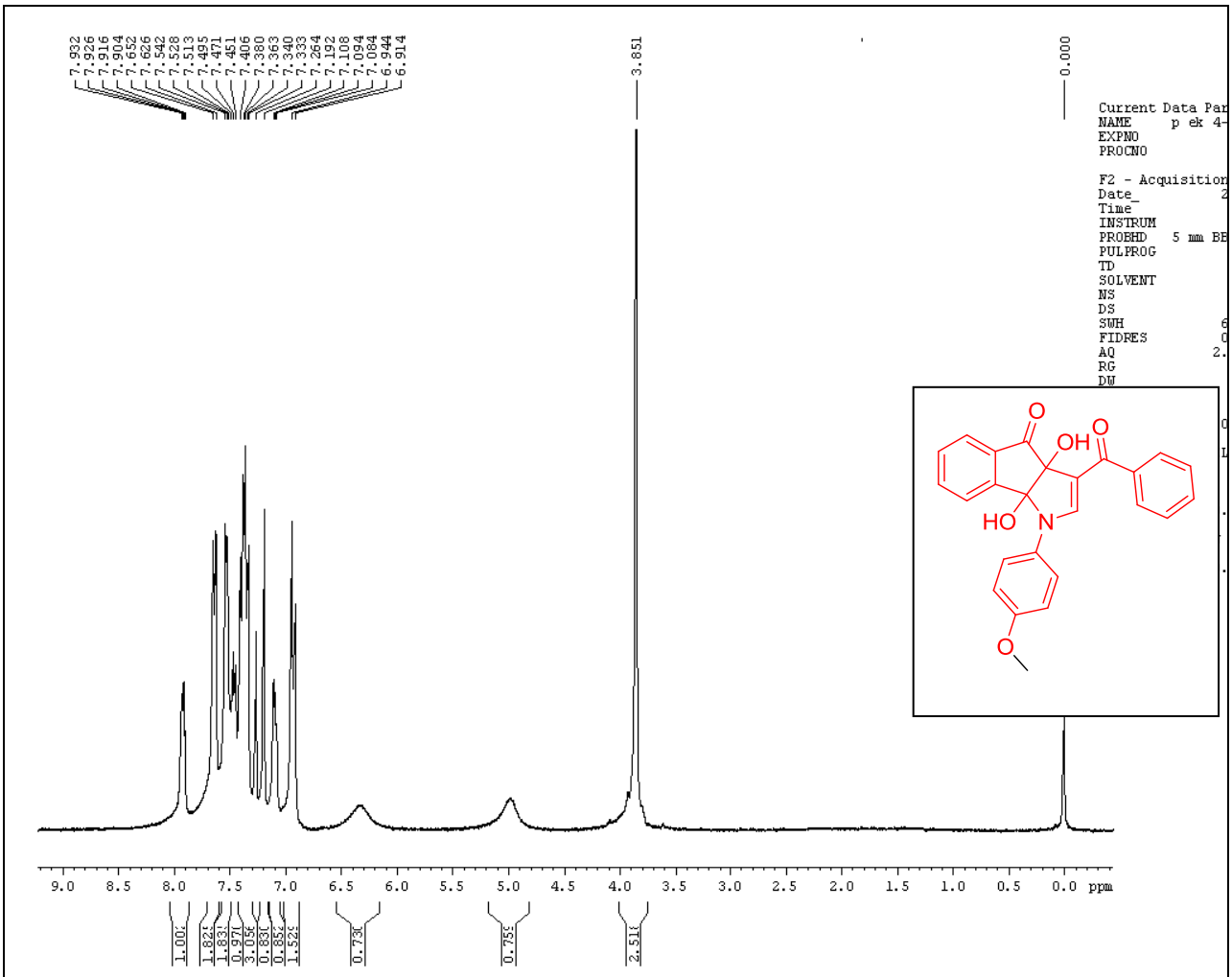
^{13}C NMR Spectrum of 4c



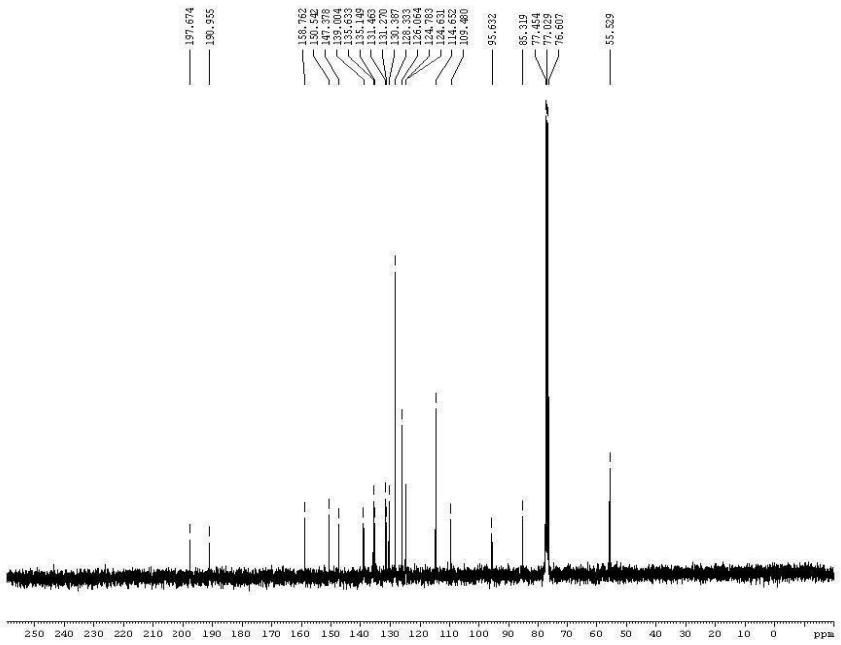
^{1}H NMR Spectrum of 4d



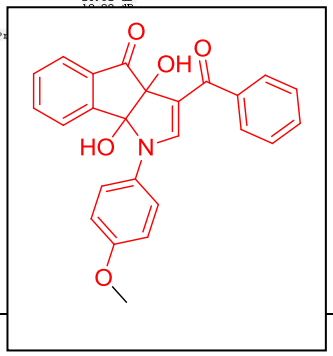
^{13}C NMR Spectrum of 4d



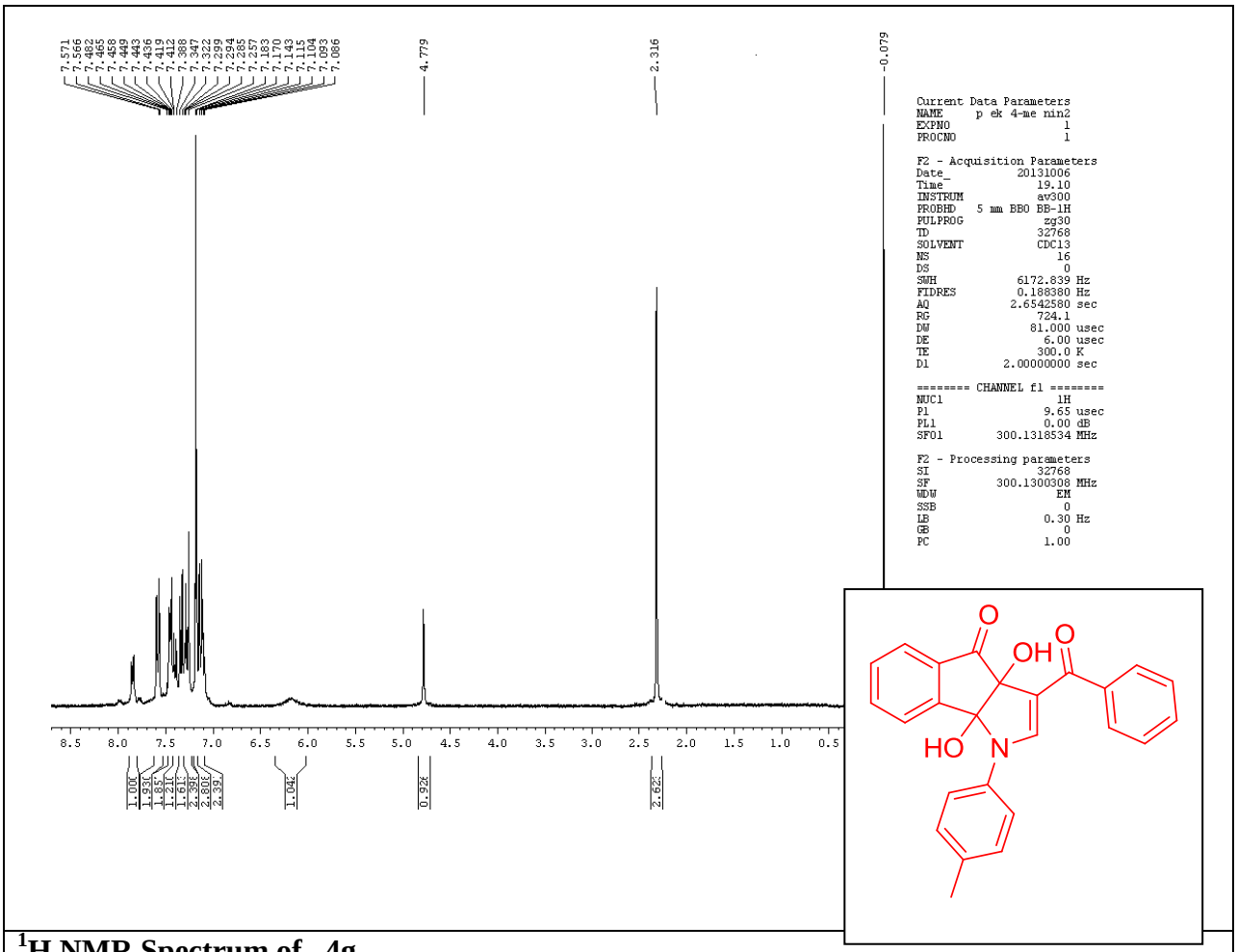
^{1}H NMR Spectrum of 4e



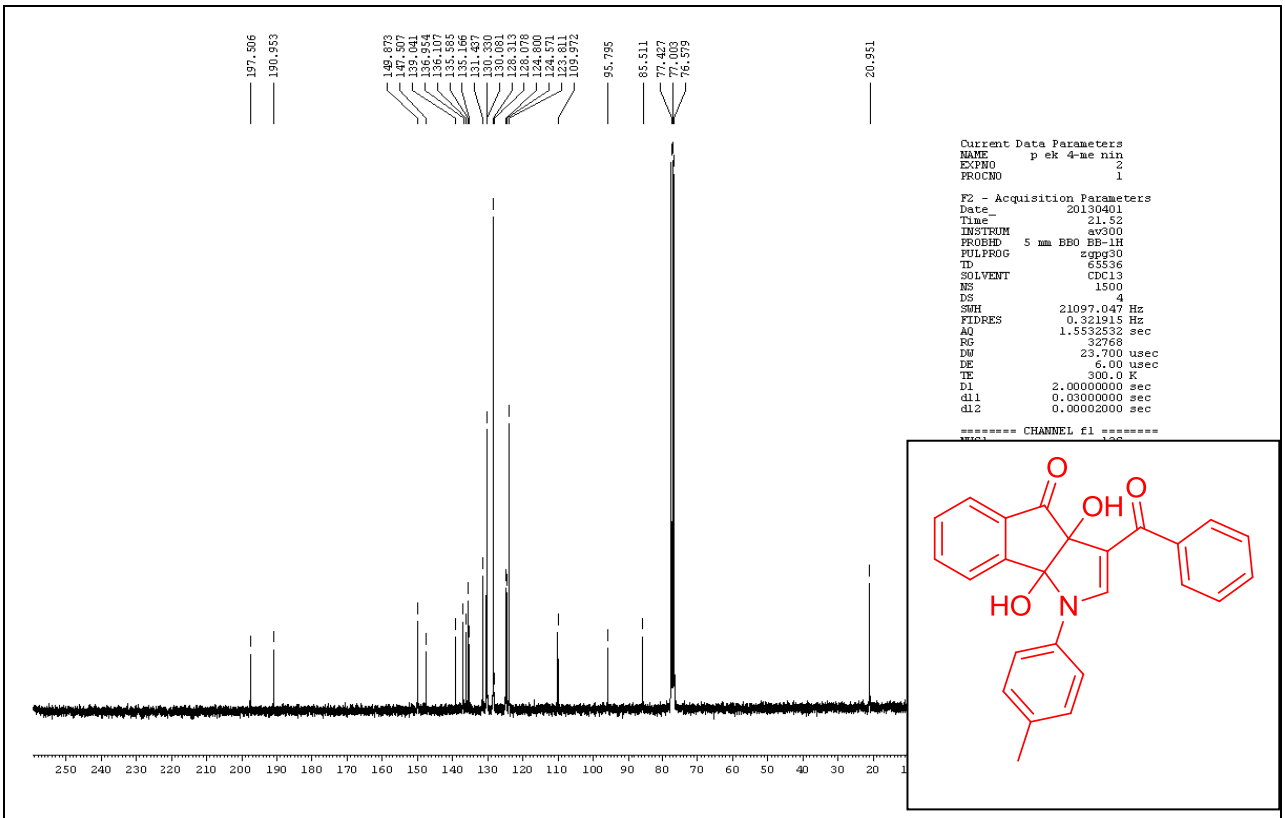

```
Current Data Parameters
NAME      p ek 4-ome nin
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20130405
Time_             16.41
INSTRUM           av300
PROBHD  5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  626
DS                    4
SWH           21097.047 Hz
FIDRES        0.321915 Hz
AQ            1.5532532 sec
RG                32768
DW               23.700 usec
DE                6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1               -5.00 dB
SF01         75.4767751 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2           100.00 usec
PL2               0.00 dB
PL12             20.31 dB
PL13
SF02

F2 - Pr
SI
SF
WDW
SSB
LB
GB
PC
```



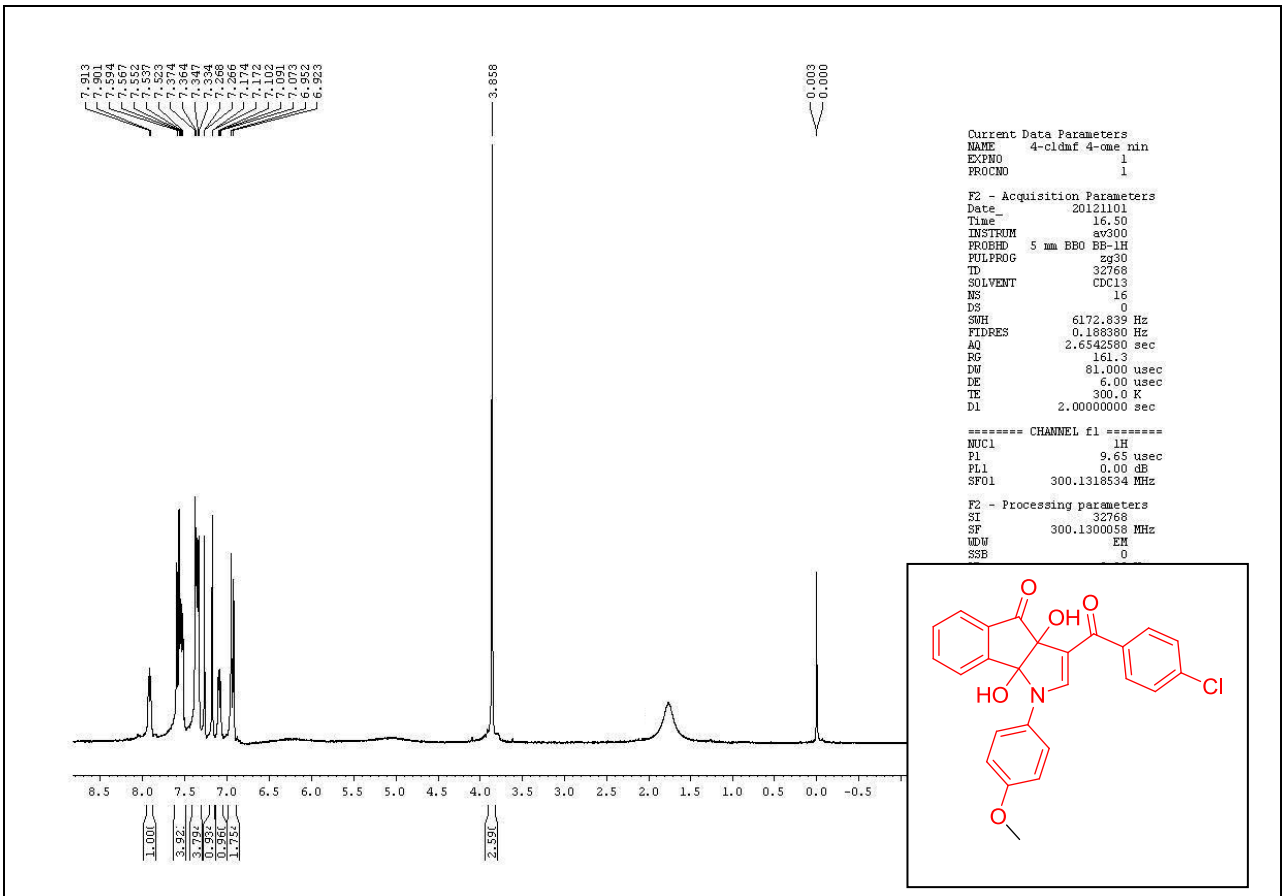
^{13}C NMR Spectrum of 4e



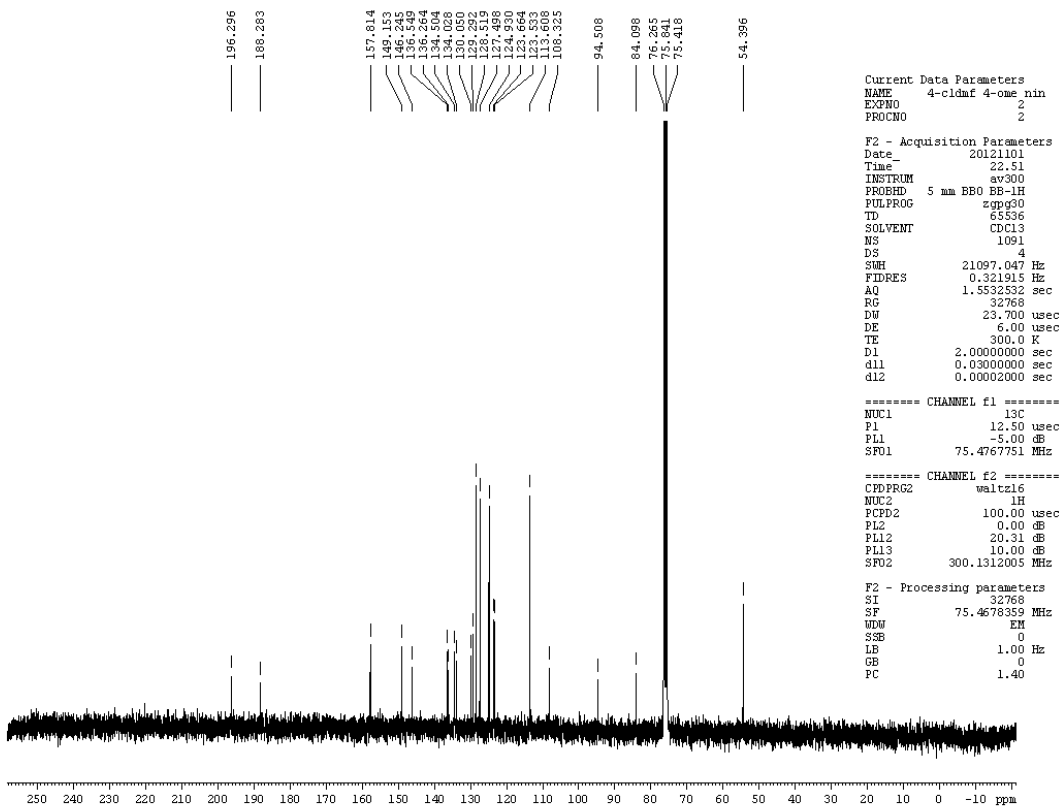
^{1}H NMR Spectrum of 4g



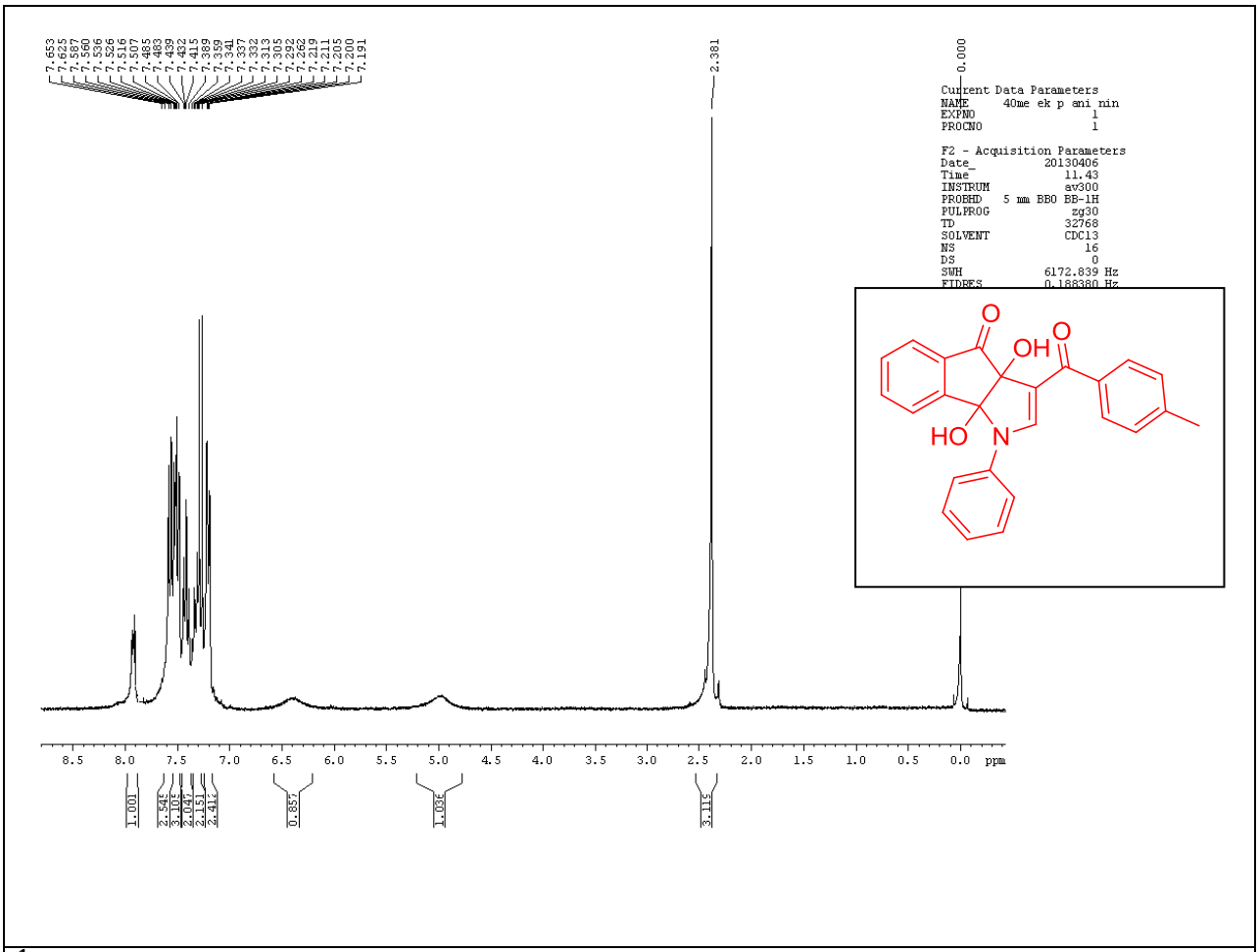
^{13}C NMR Spectrum of 4g



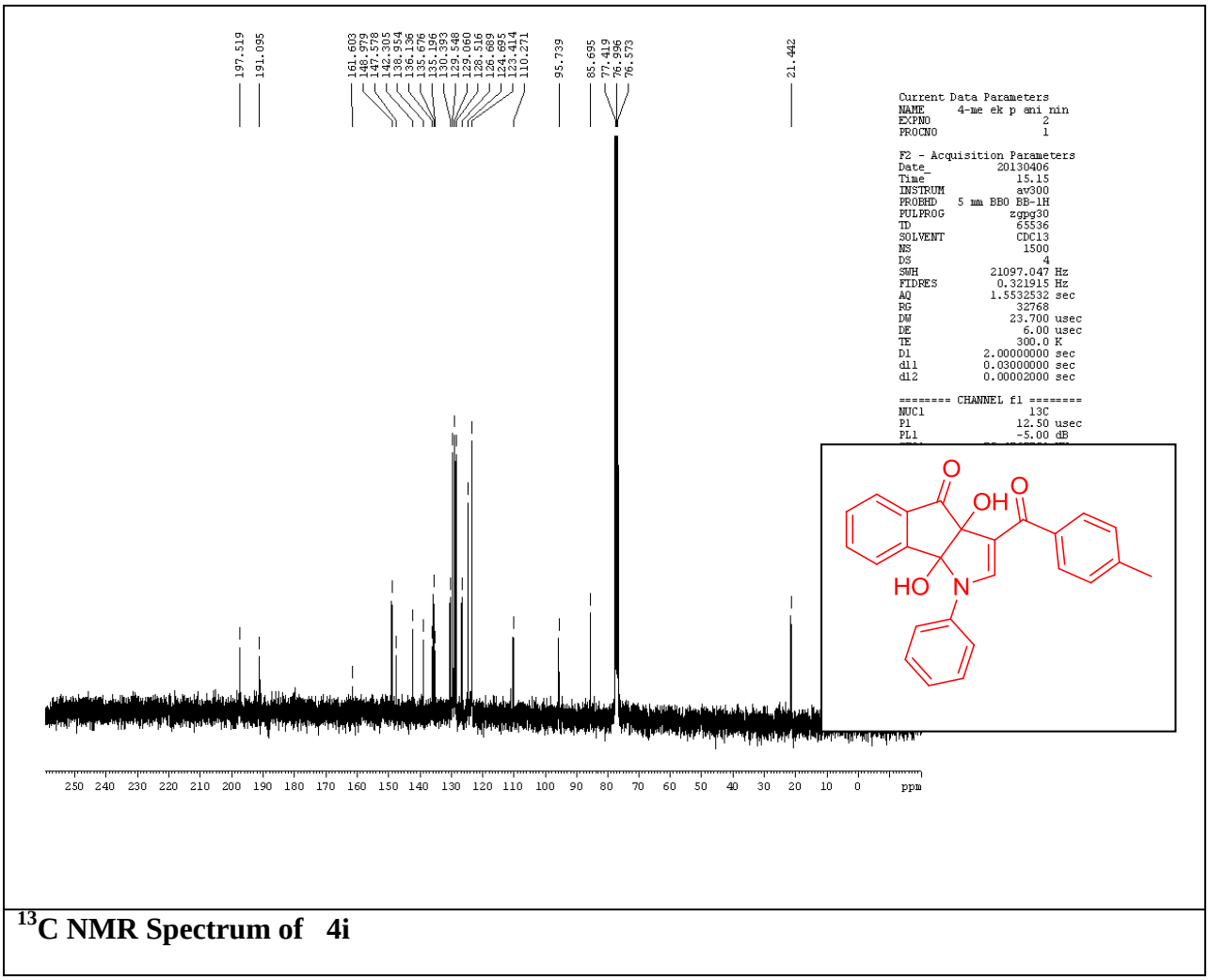
^{1}H NMR Spectrum of 4h



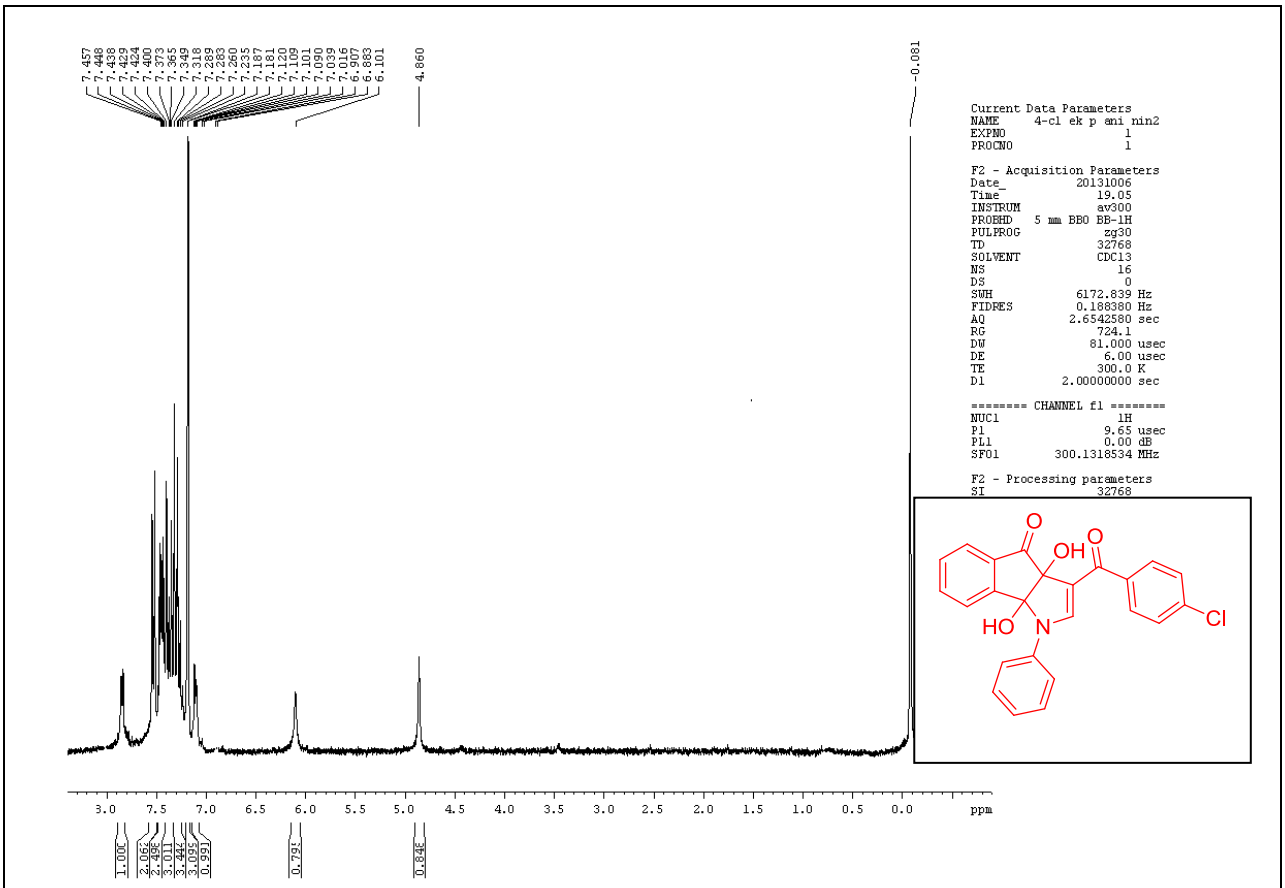
^{13}C NMR Spectrum of 4h



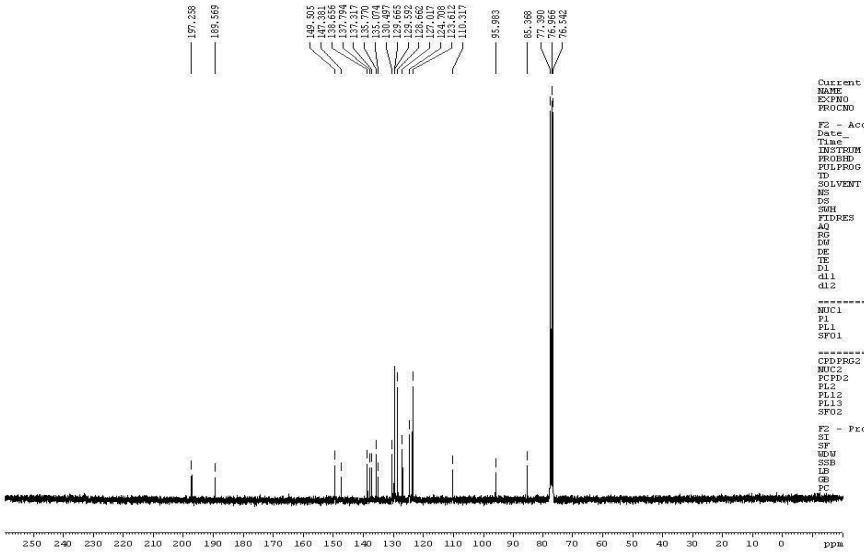
^{1}H NMR Spectrum of 4i

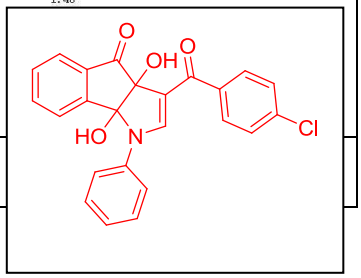


^{13}C NMR Spectrum of 4i

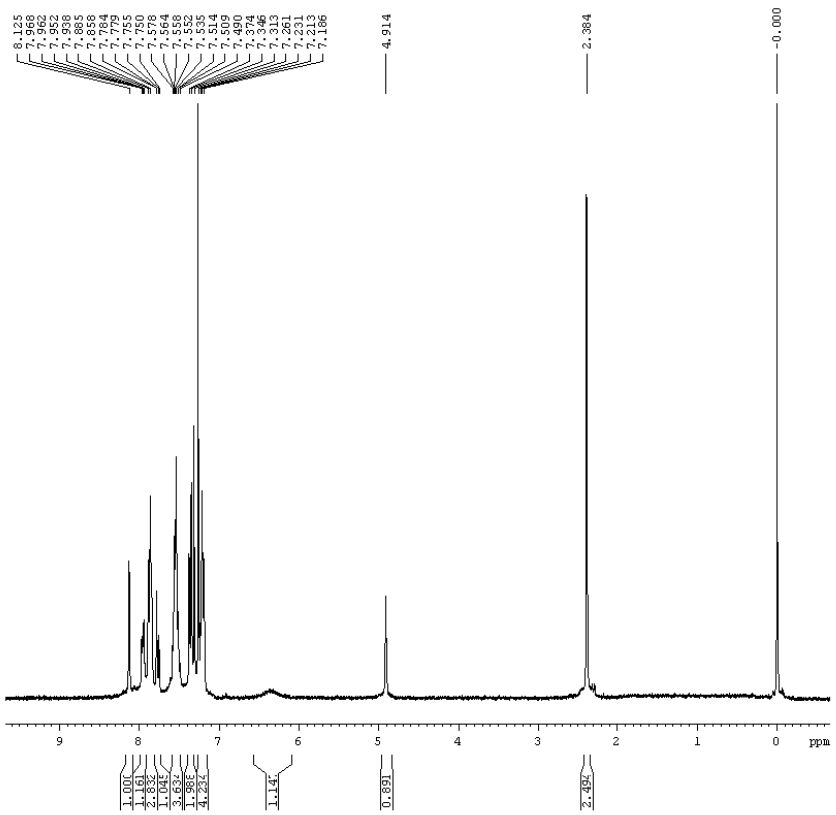


^{1}H NMR Spectrum of 4j

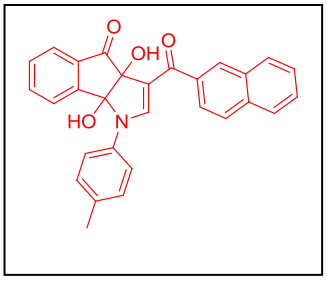




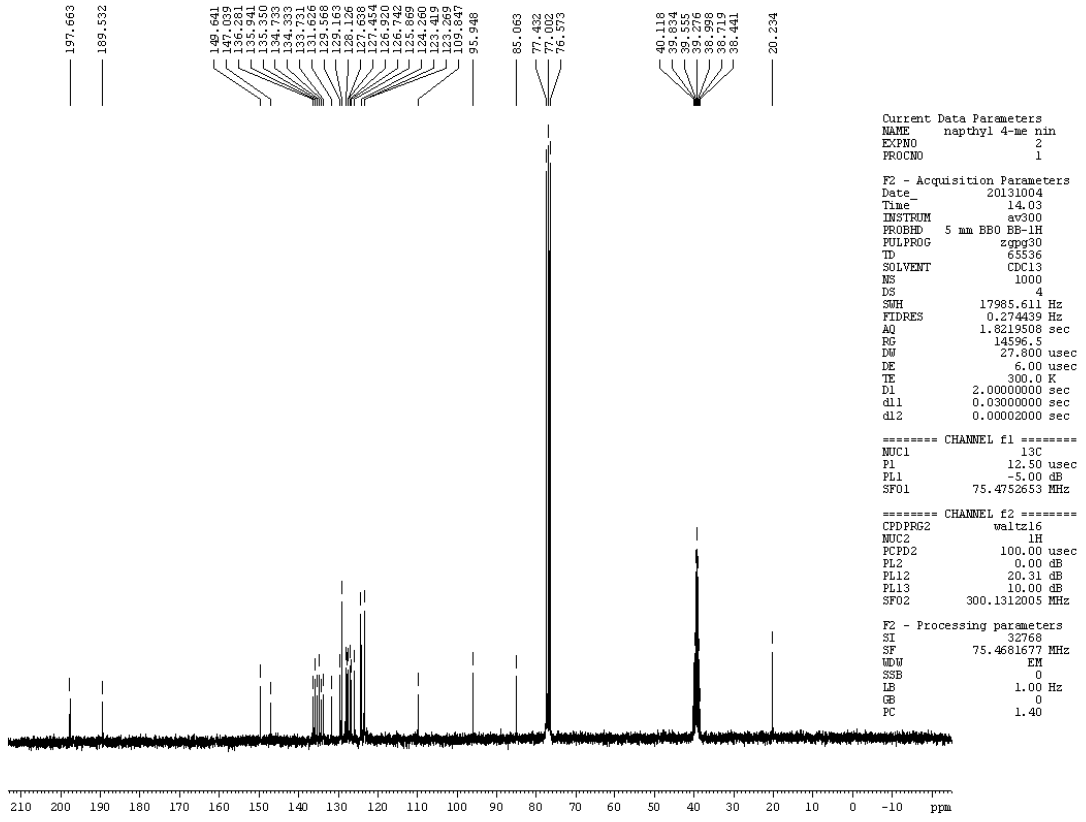
^{13}C NMR Spectrum of 4j



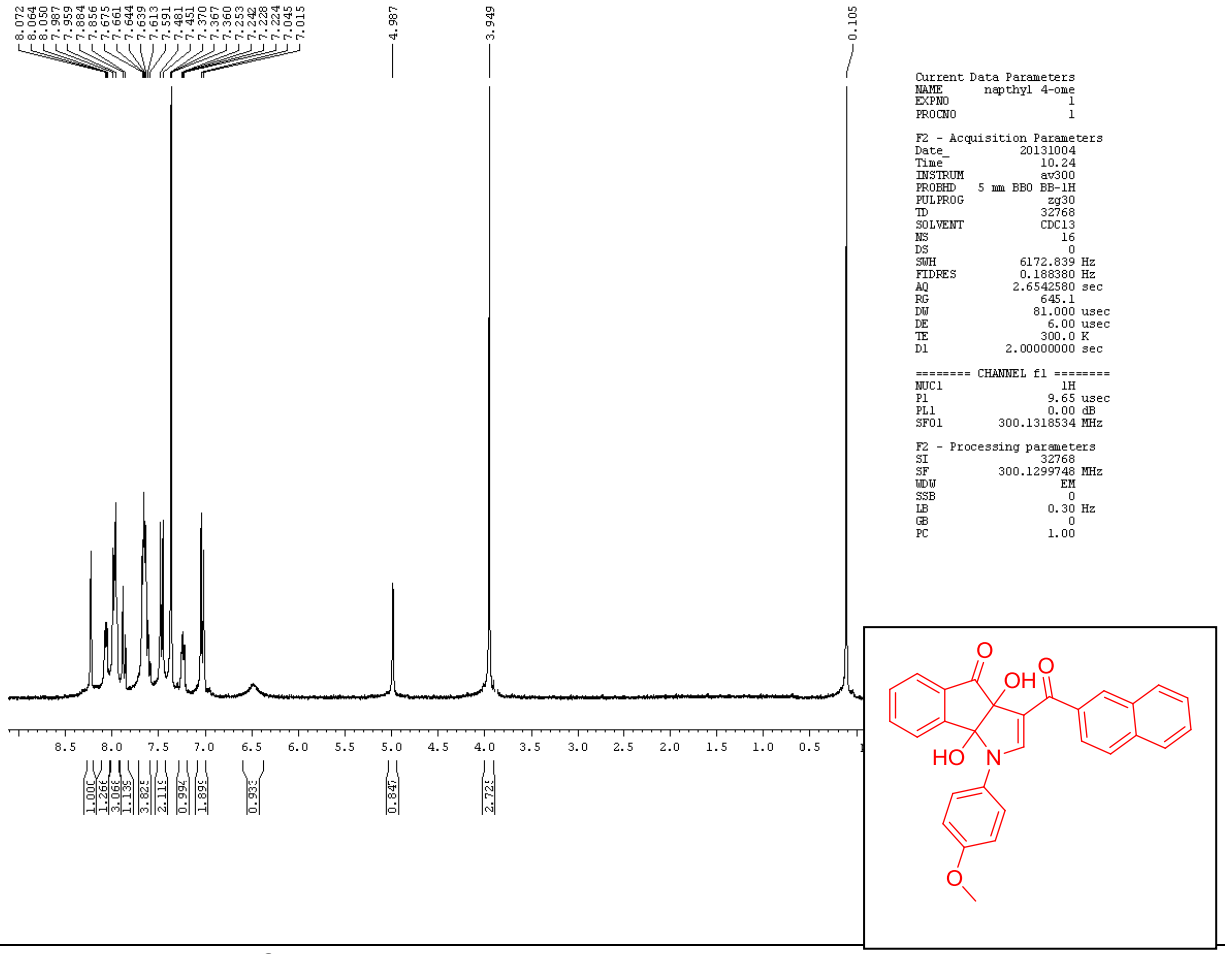

```
Current Data Parameters
NAME        napthyl 4-me
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131004
Time               10.20
INSTRUM            av300
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 724.1
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.65 usec
PL1                0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300088 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



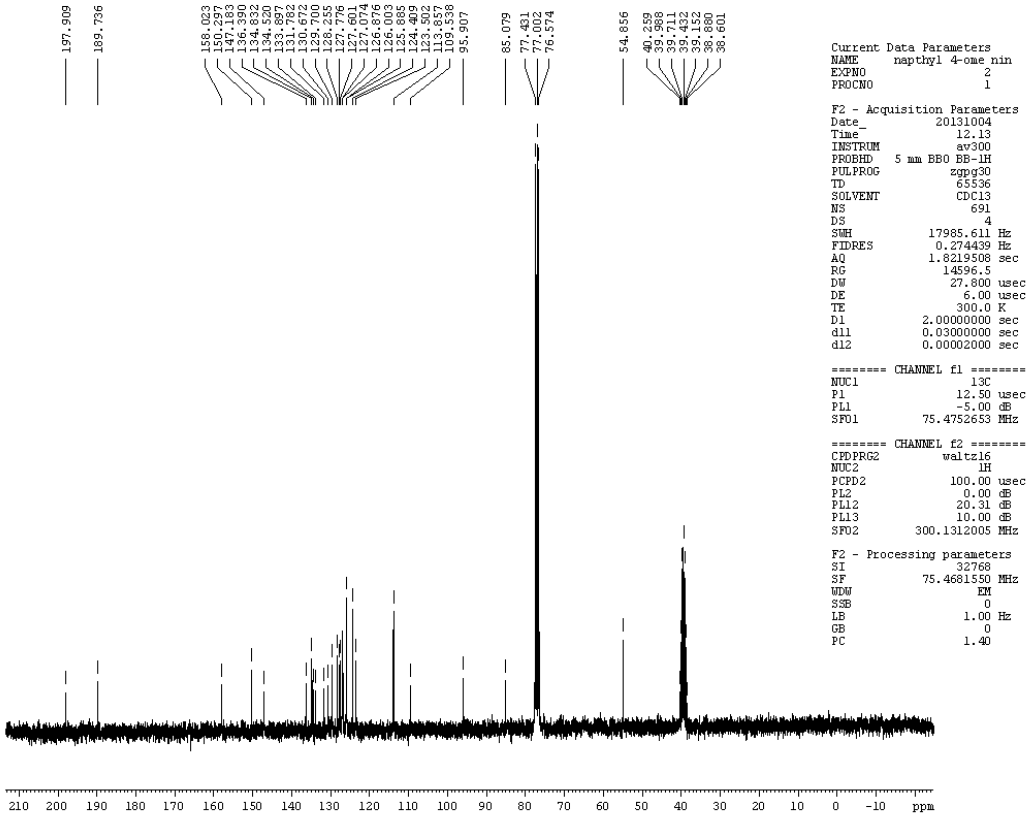
^{1}H NMR Spectrum of 4k



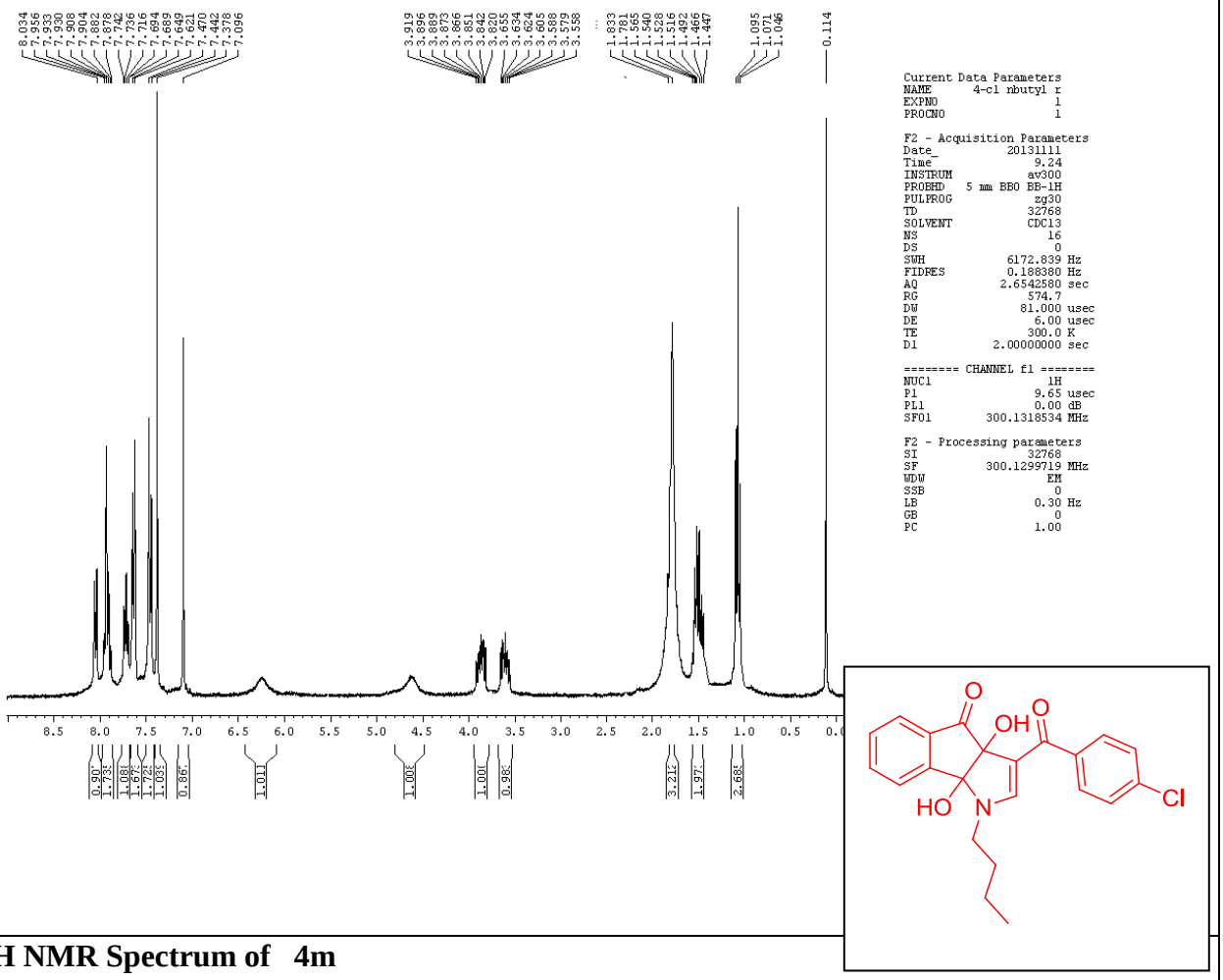
^{13}C NMR Spectrum of 4k



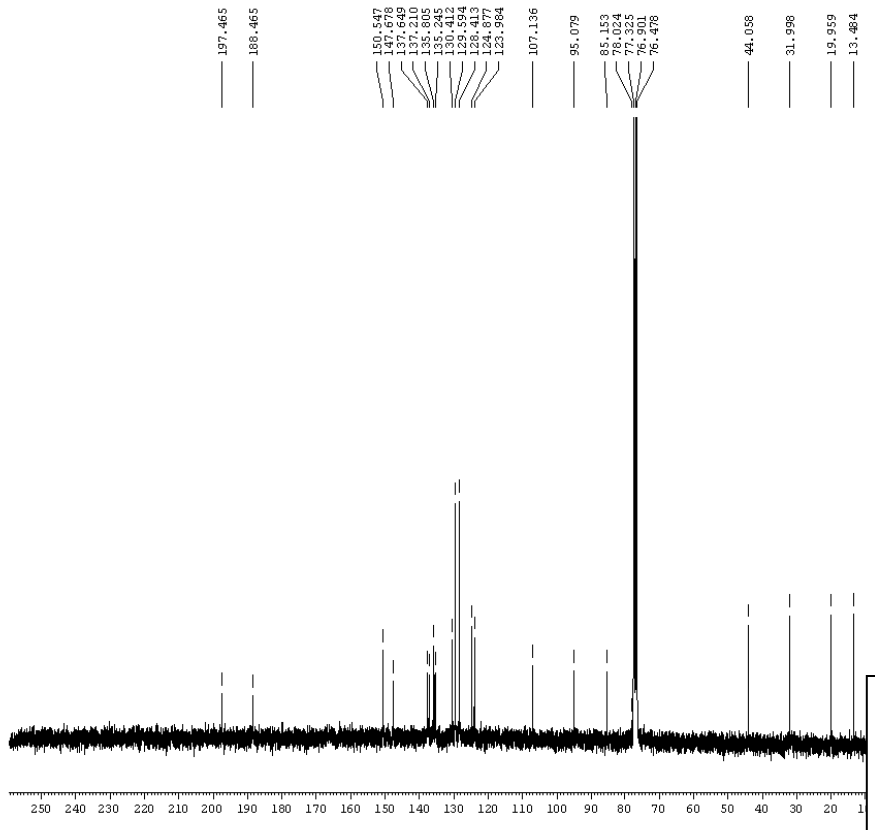
^{1}H NMR Spectrum of 4l



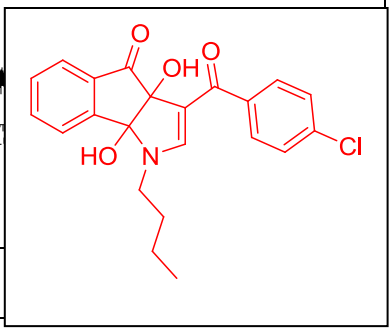
^{13}C NMR Spectrum of 4l



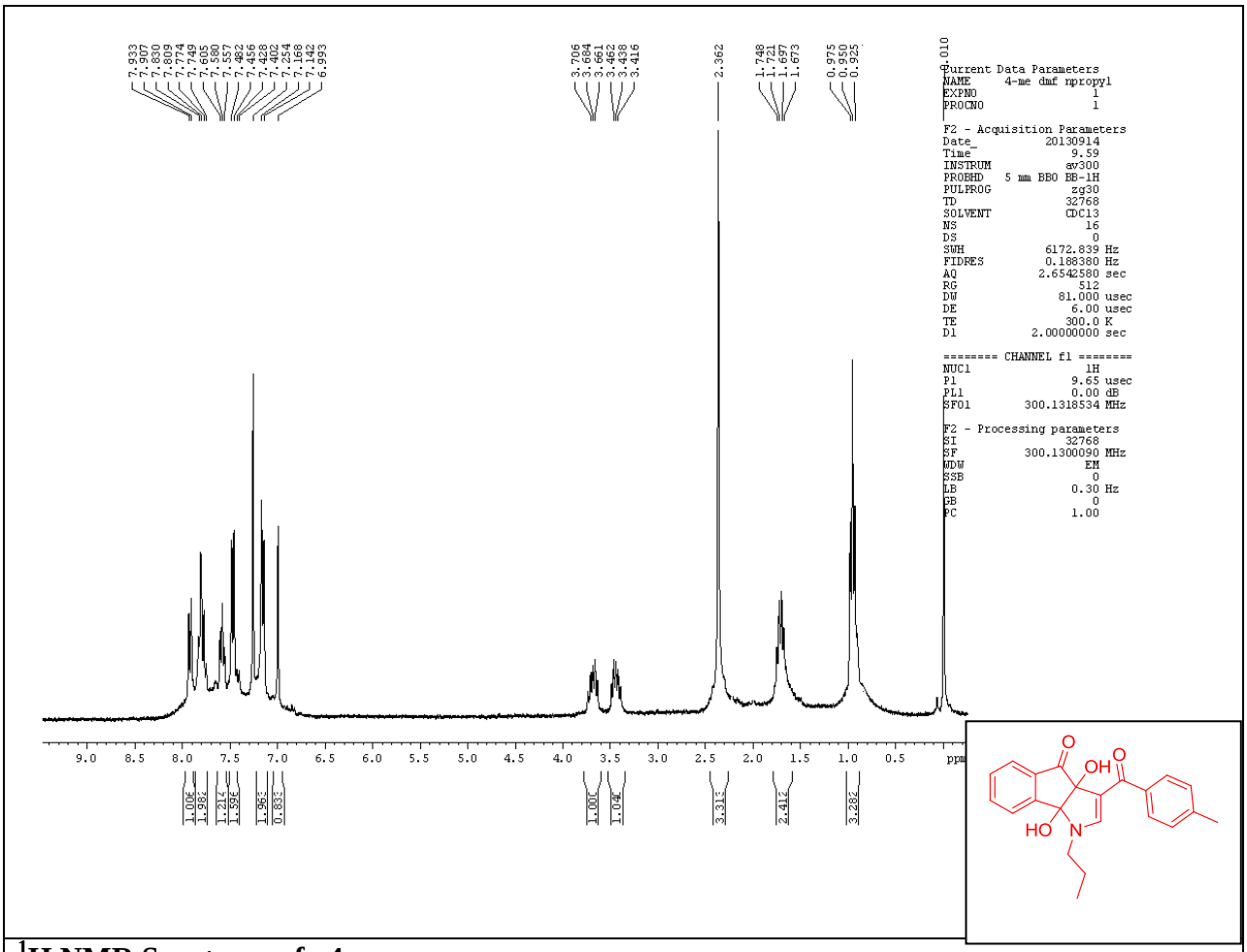
^{1}H NMR Spectrum of 4m



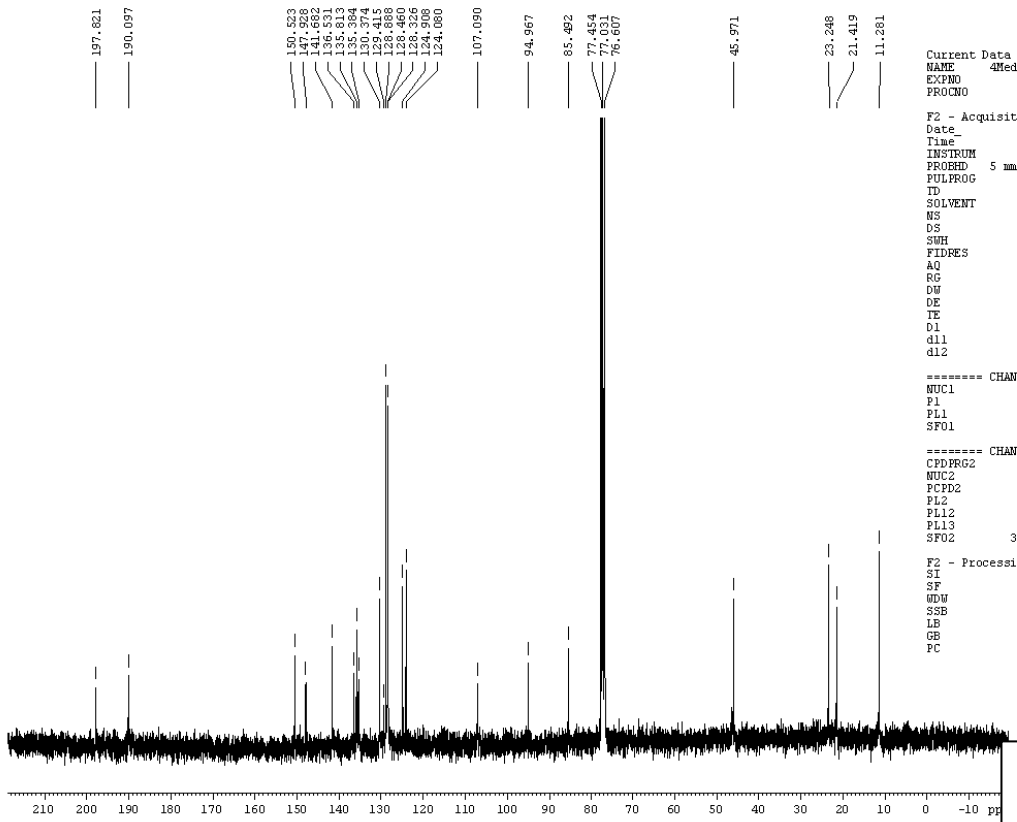

```
Current Data Parameters
NAME          4-cl nbutyl
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130808
Time                10.42
INSTRUM             av300
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                   1974
DS                      4
SWH           21097.047 Hz
FIDRES         0.321915 Hz
AQ             1.5532532 sec
RG               14596.5
DW                23.700 usec
DE                 6.00 usec
TE                300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.50 usec
PL1                 -5.00 dB
SFO1          75.4767751 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 0.00 dB
PL12               20.31 dB
PL13               10.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF            75.4677559 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



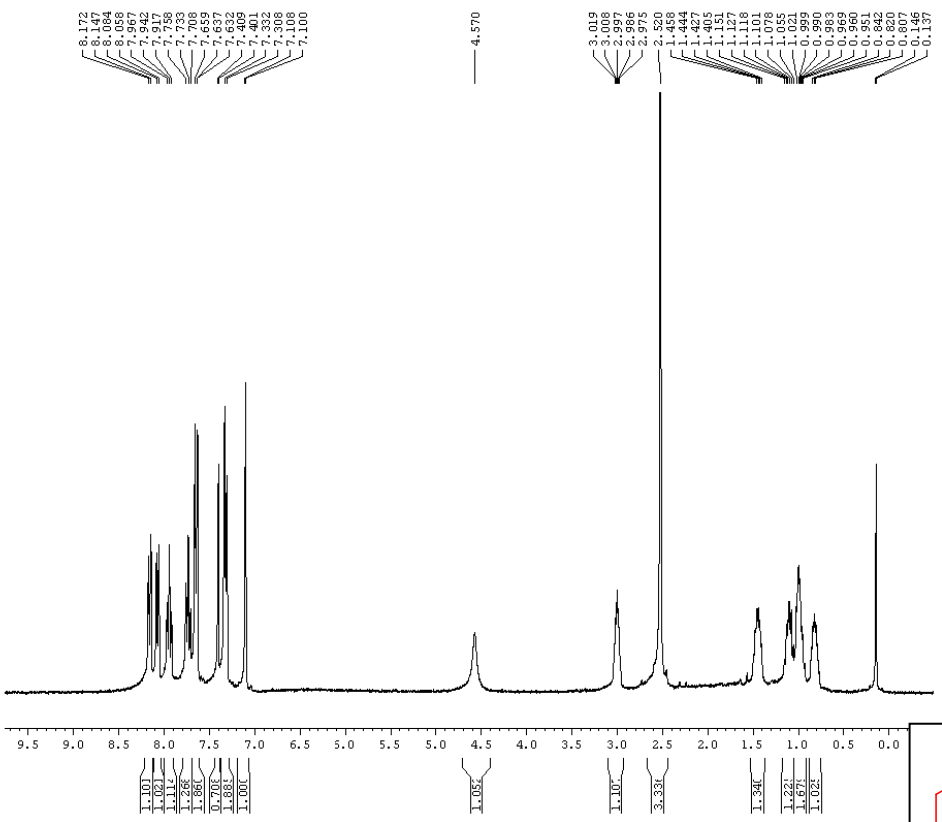
^{13}C NMR Spectrum of 4m



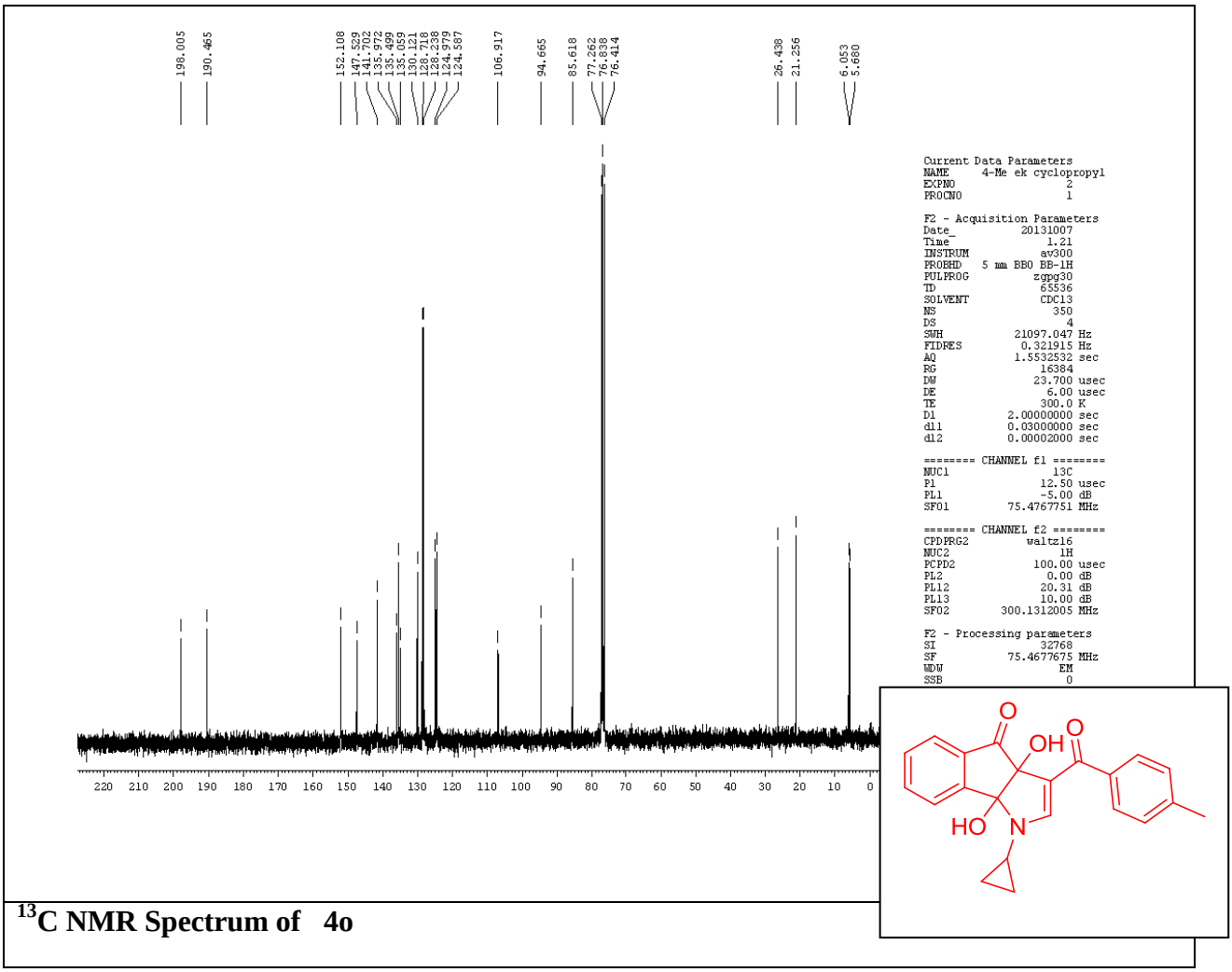
^{1}H NMR Spectrum of 4n



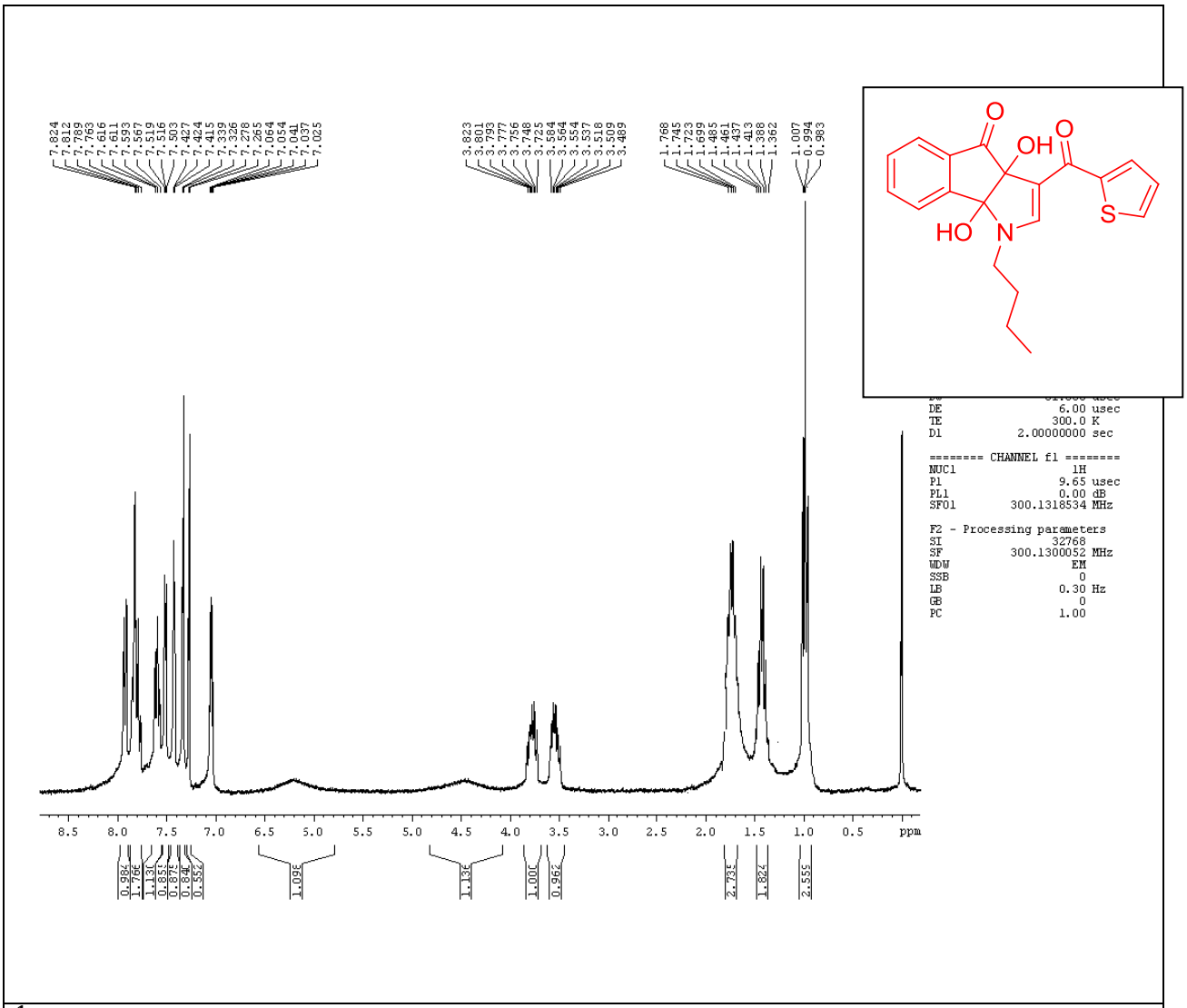
^{13}C NMR Spectrum of 4n



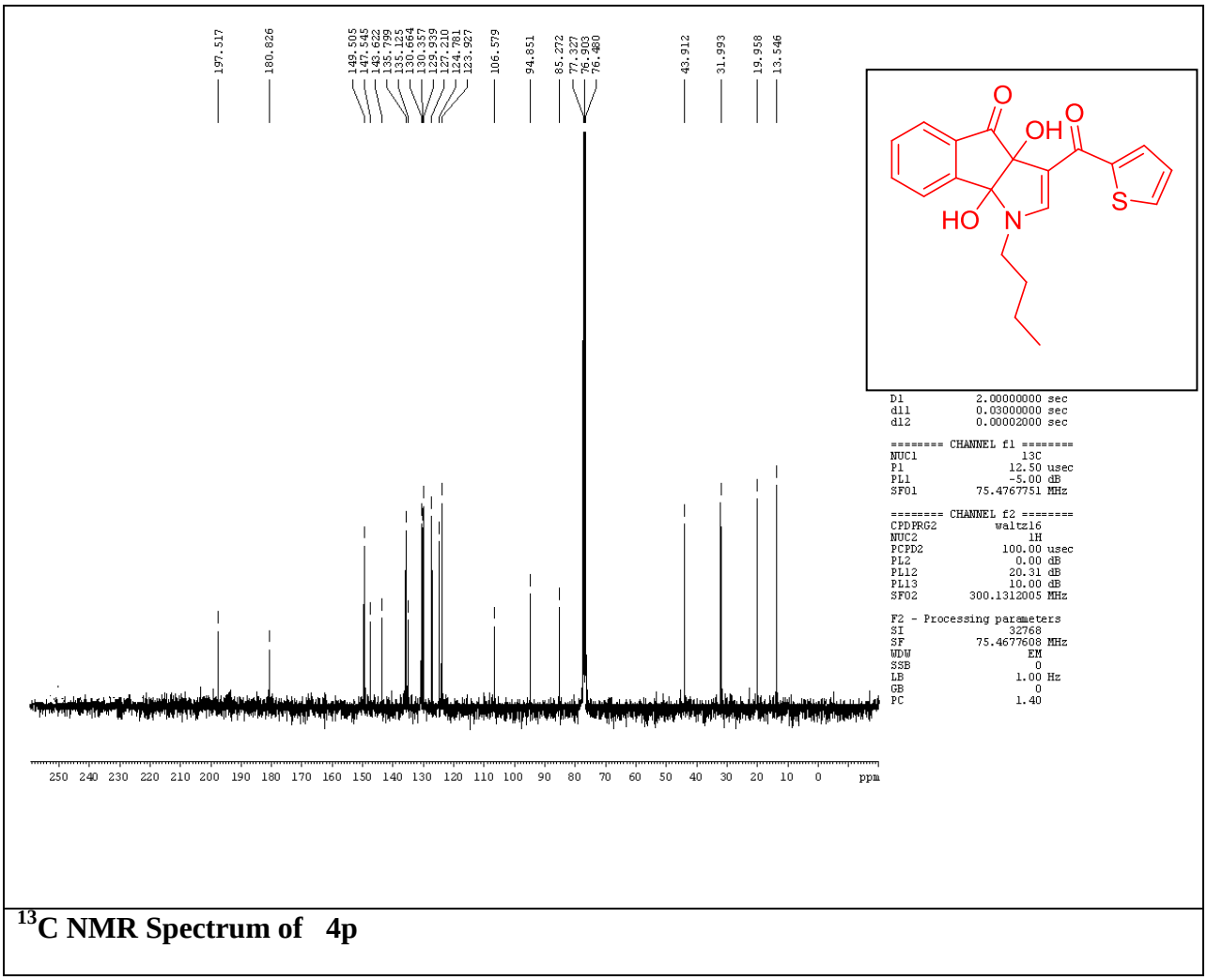
^{1}H NMR Spectrum of 4o



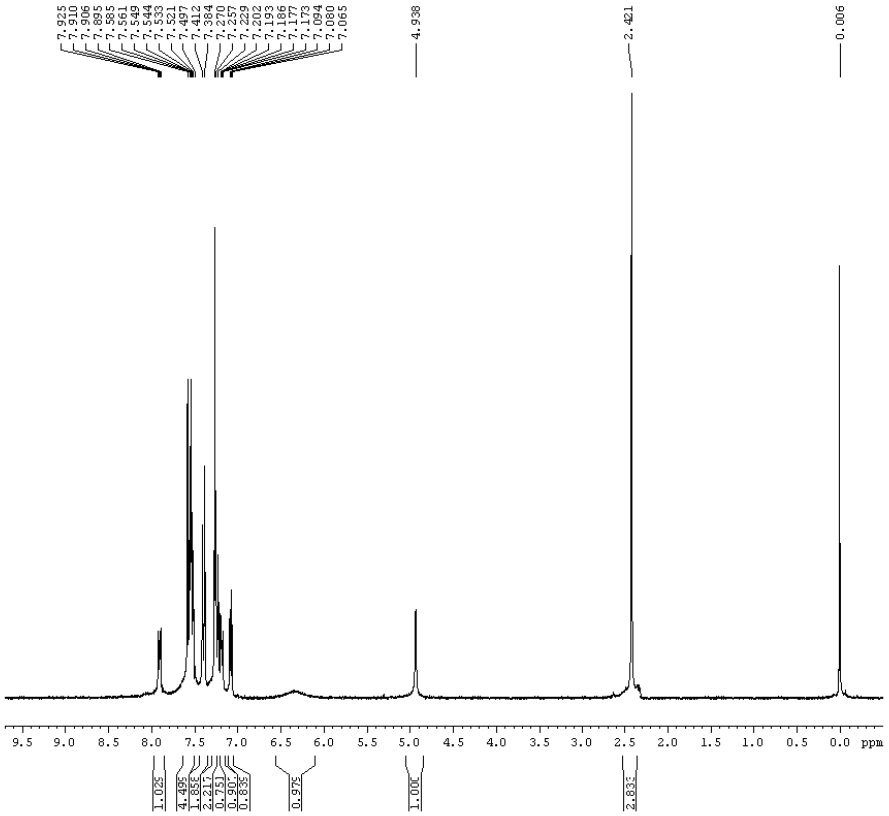
^{13}C NMR Spectrum of 4o



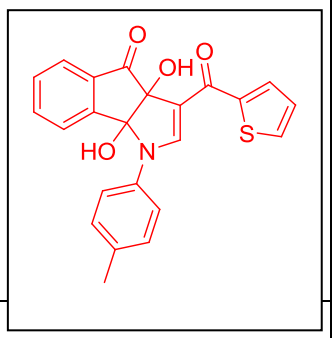
^{1}H NMR Spectrum of 4p



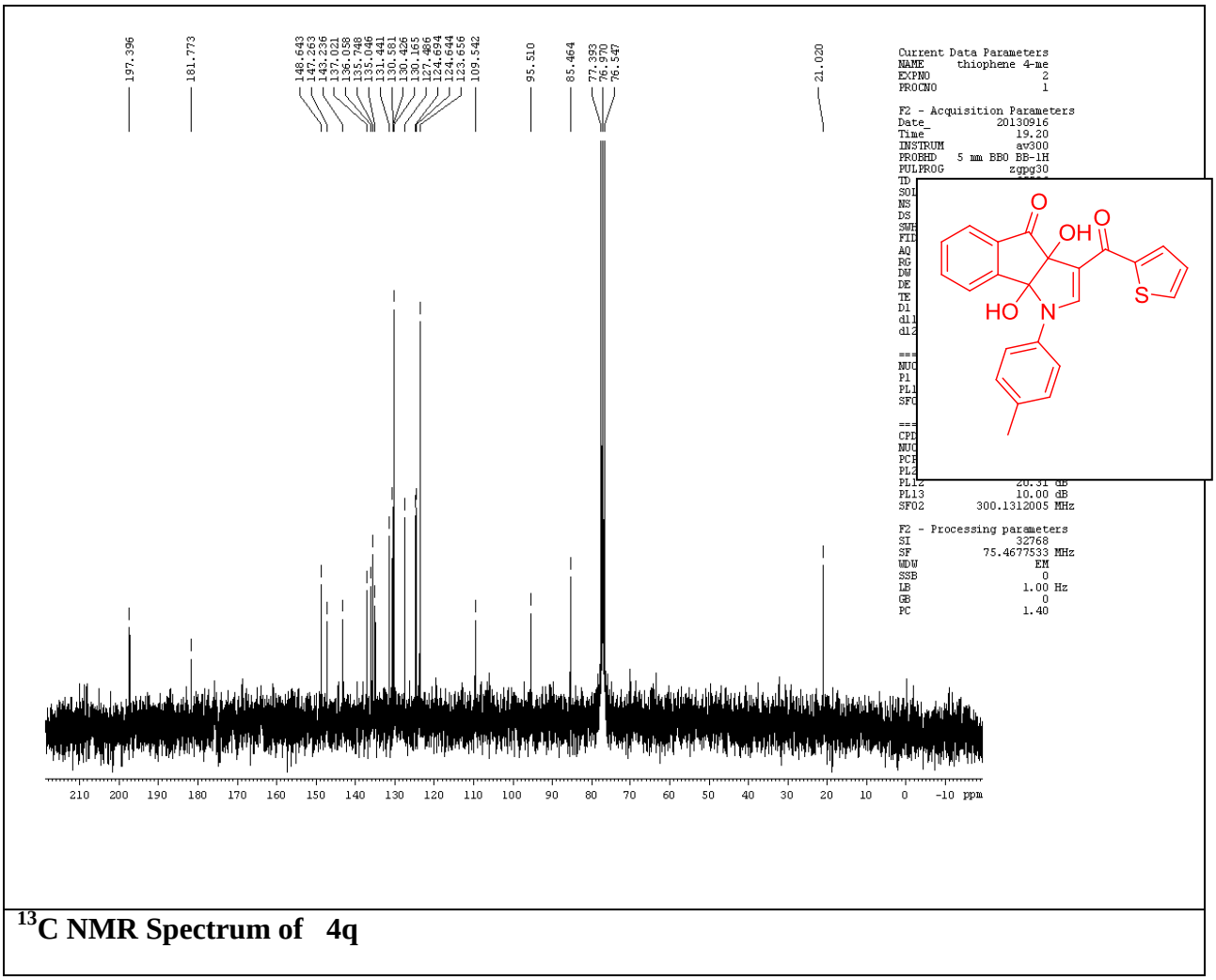
^{13}C NMR Spectrum of 4p



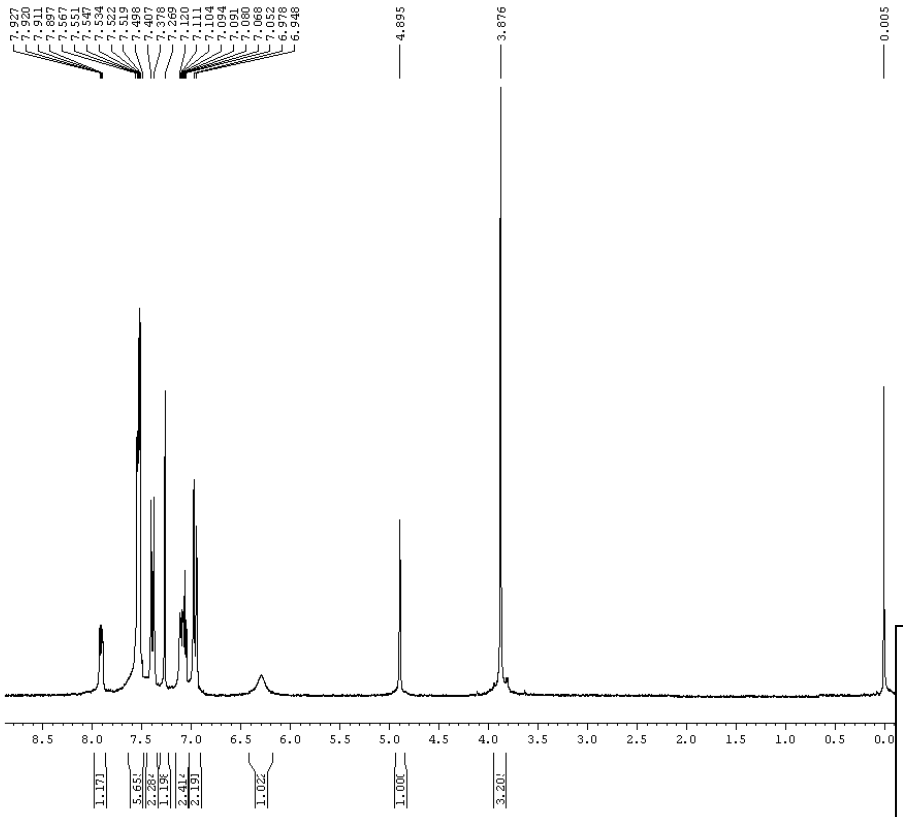

```
Current Data Parameters
NAME       thiphene 4-me
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20130916
Time              16.08
INSTRUM           av300
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    19
DS                     0
SWH            6172.839 Hz
FIDRES            0.188380 Hz
AQ             2.6542580 sec
RG                456.1
DW               81.000 usec
DE                6.00 usec
TE                300.0 K
D1             2.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.65 usec
PL1                0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300041 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



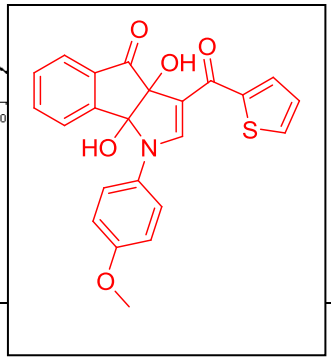
^{1}H NMR Spectrum of 4q



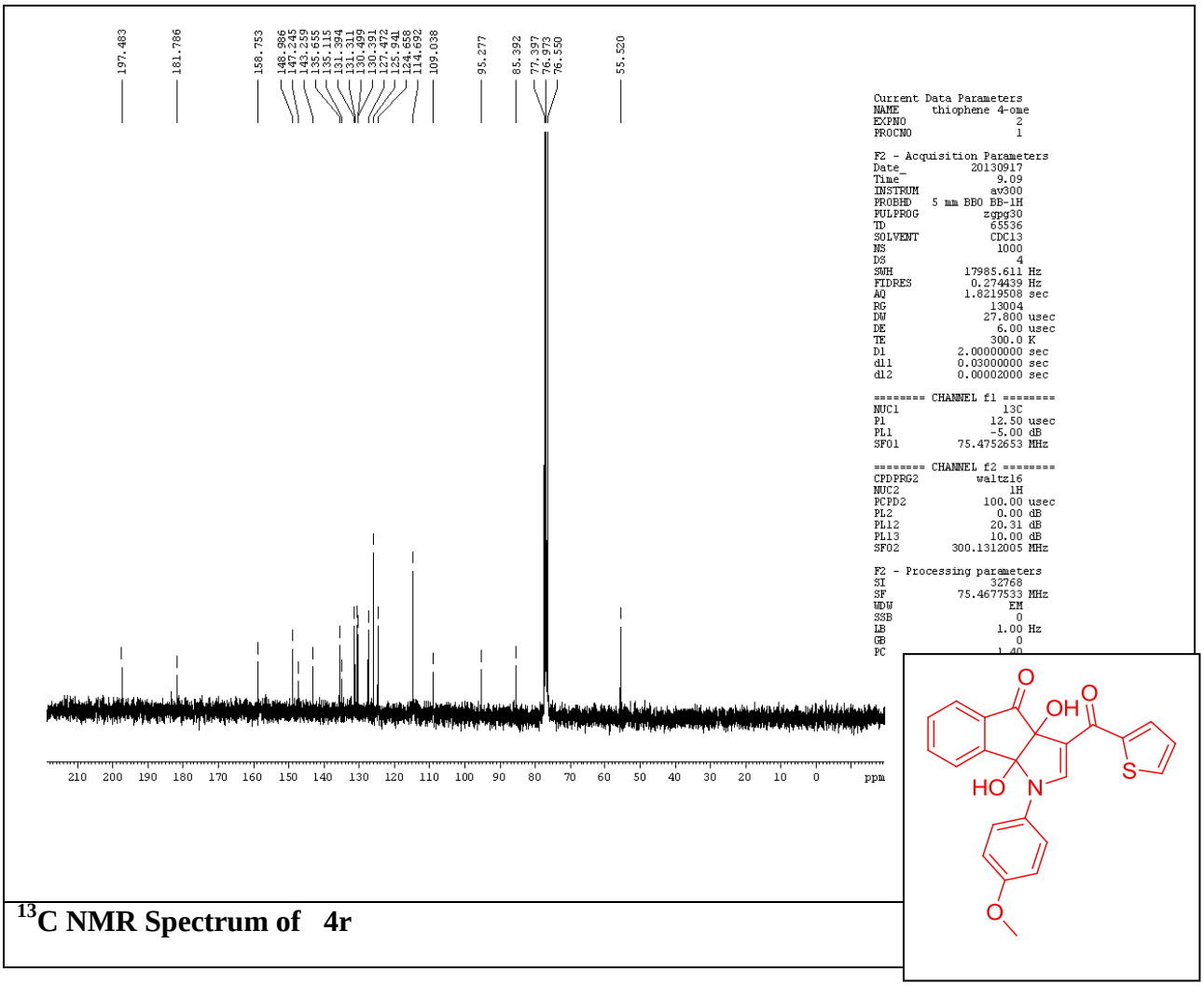
^{13}C NMR Spectrum of 4q



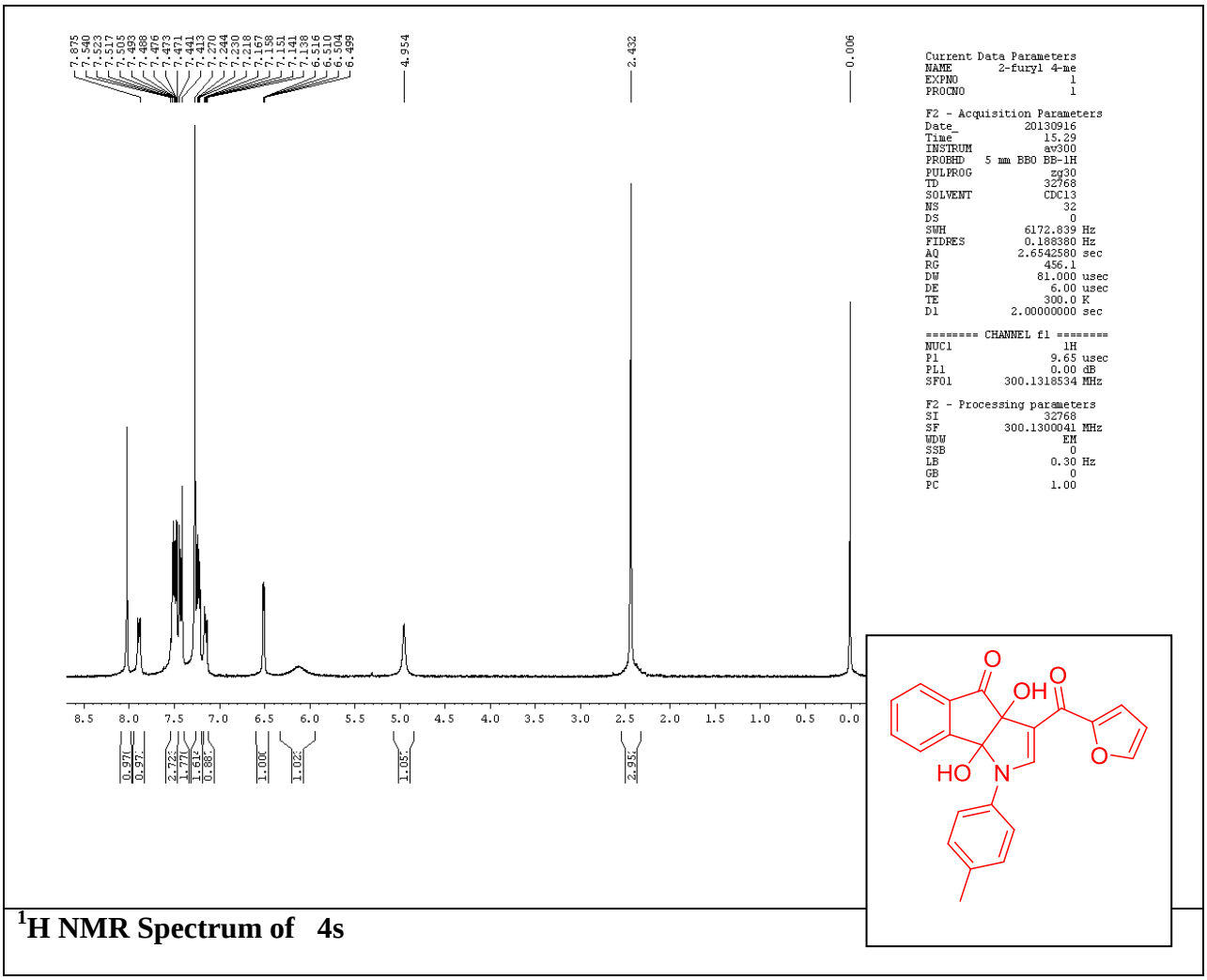

```
Current Data Parameters
NAME      thiophene 4-ome
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130916
Time_              16.17
INSTRUM            av300
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    15
DS                     0
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 456.1
DW                81.000 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.65 usec
PL1                0.00 dB
SF01        300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300041 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



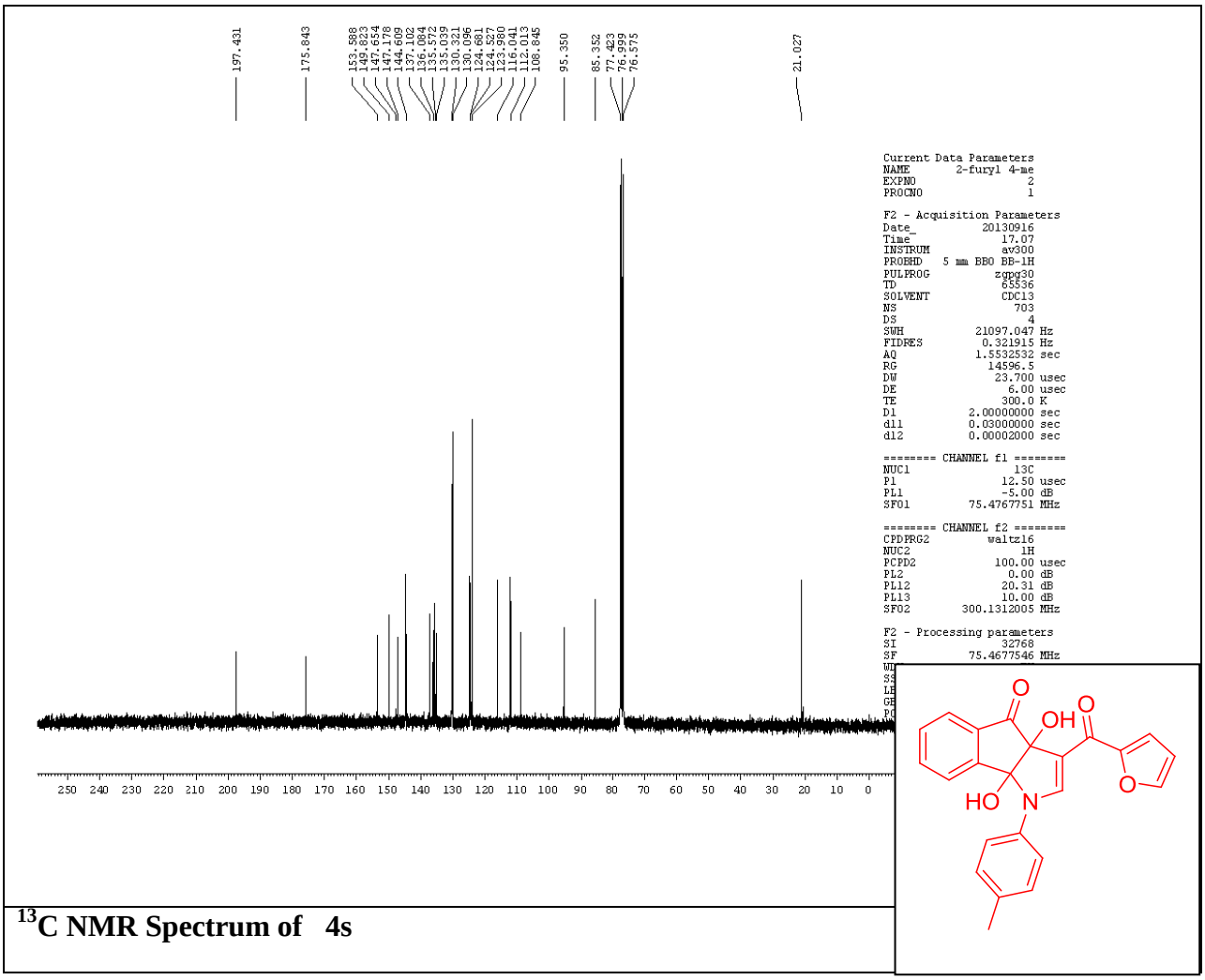
^{1}H NMR Spectrum of 4r



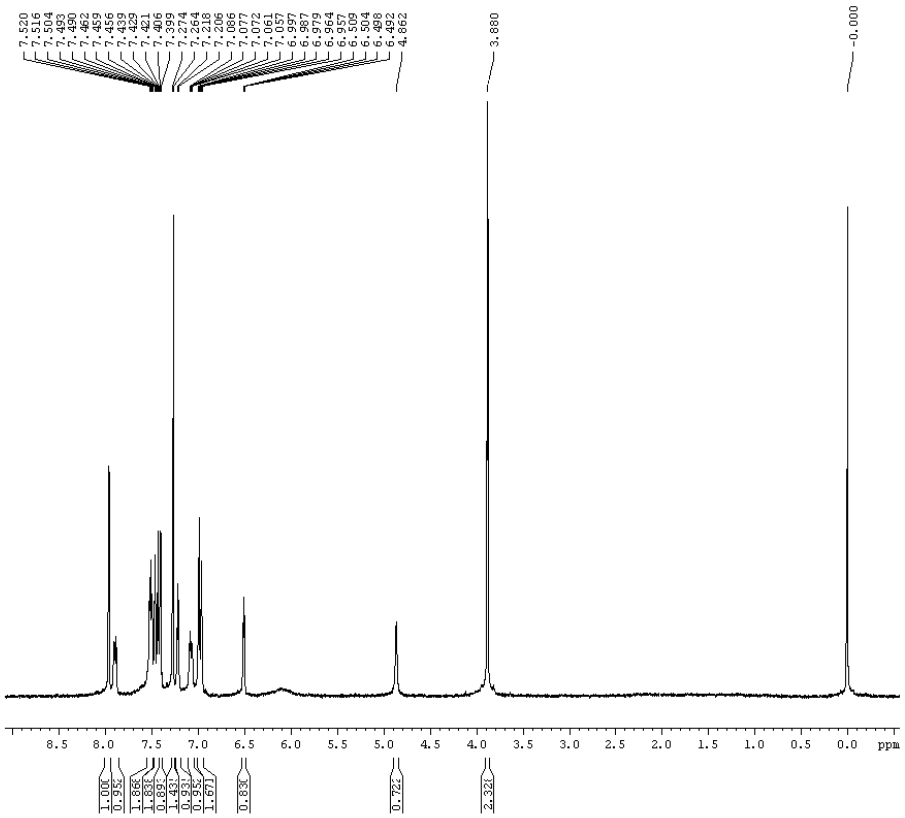
^{13}C NMR Spectrum of 4r



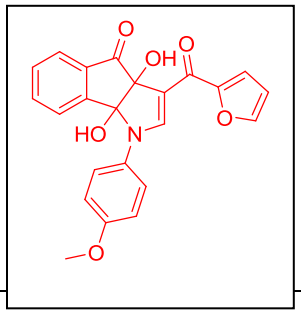
^{1}H NMR Spectrum of 4s



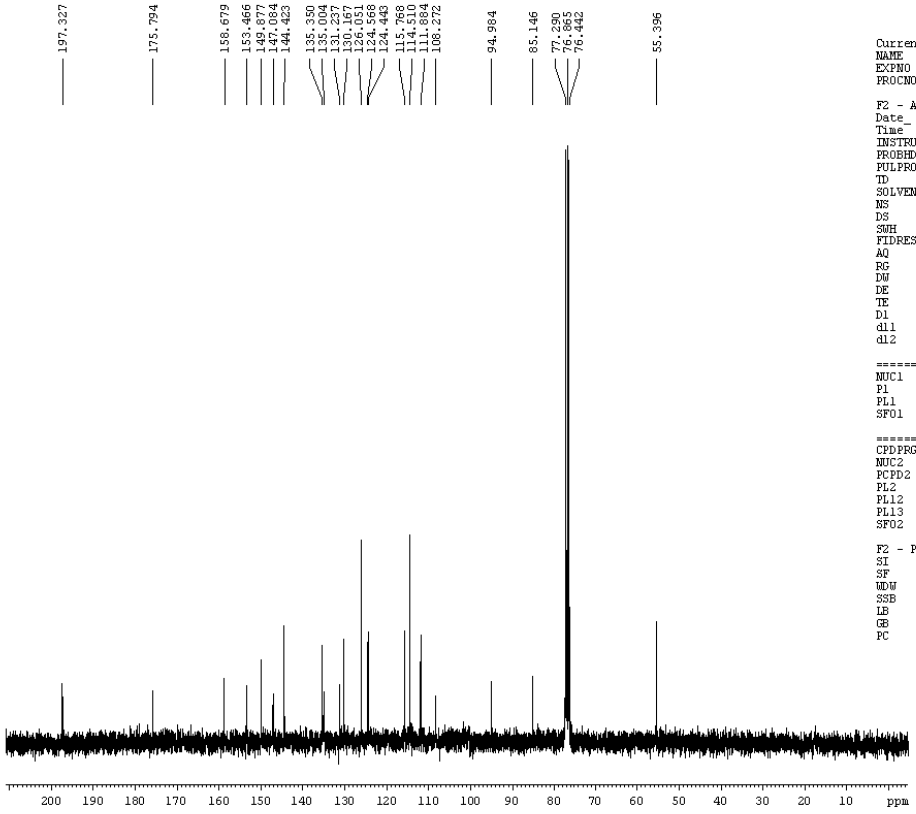
^{13}C NMR Spectrum of 4s



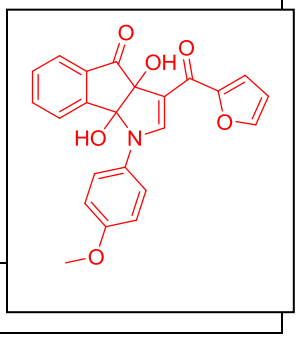

```
Current Data Parameters
NAME      2-furyl 4-methoxy
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20130912
Time                 16.55
INSTRUM              av300
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                   574.7
DW                  81.000 usec
DE                   6.00 usec
TE                  300.0 K
D1              2.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.65 usec
PL1                  0.00 dB
SF01          300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300056 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



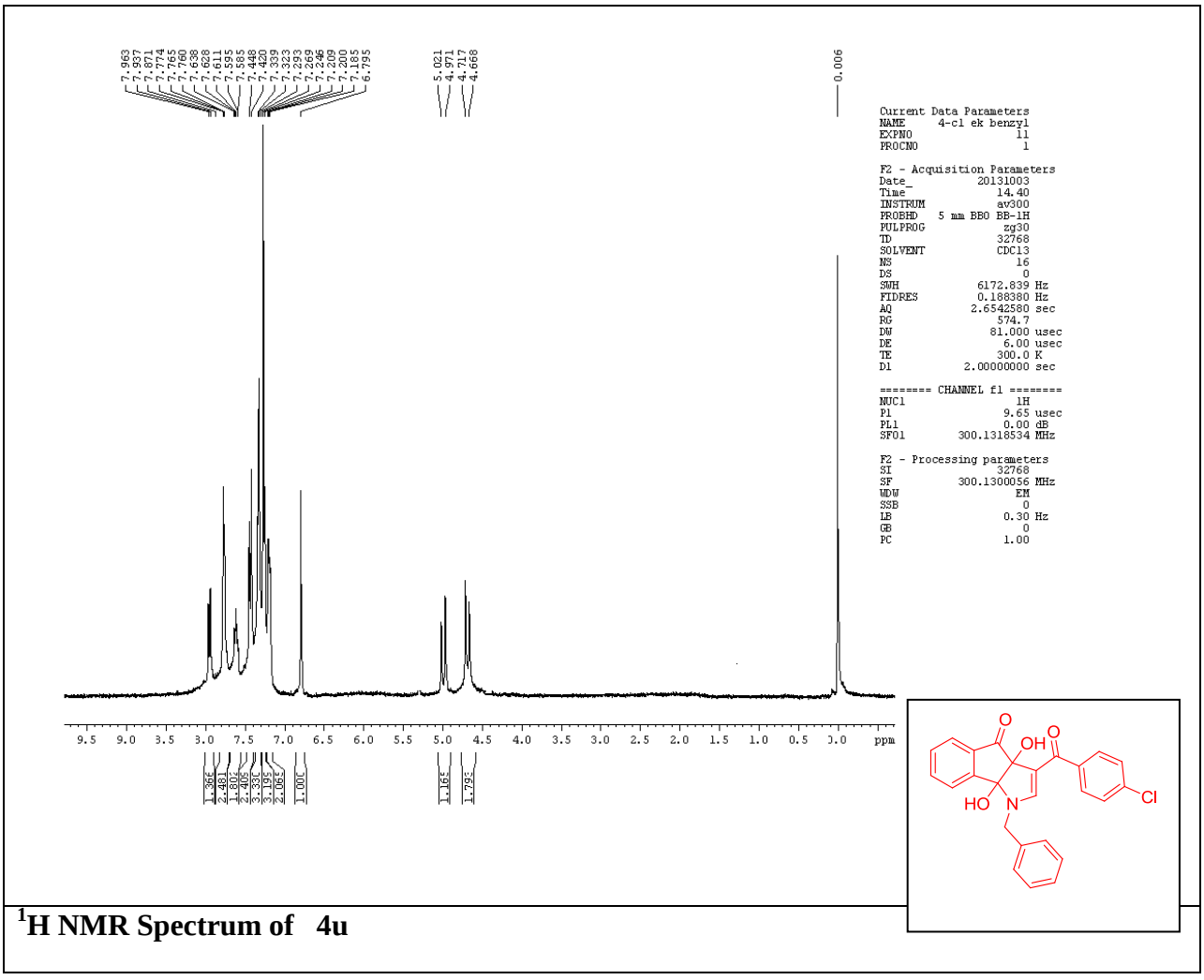
^{1}H NMR Spectrum of 4t



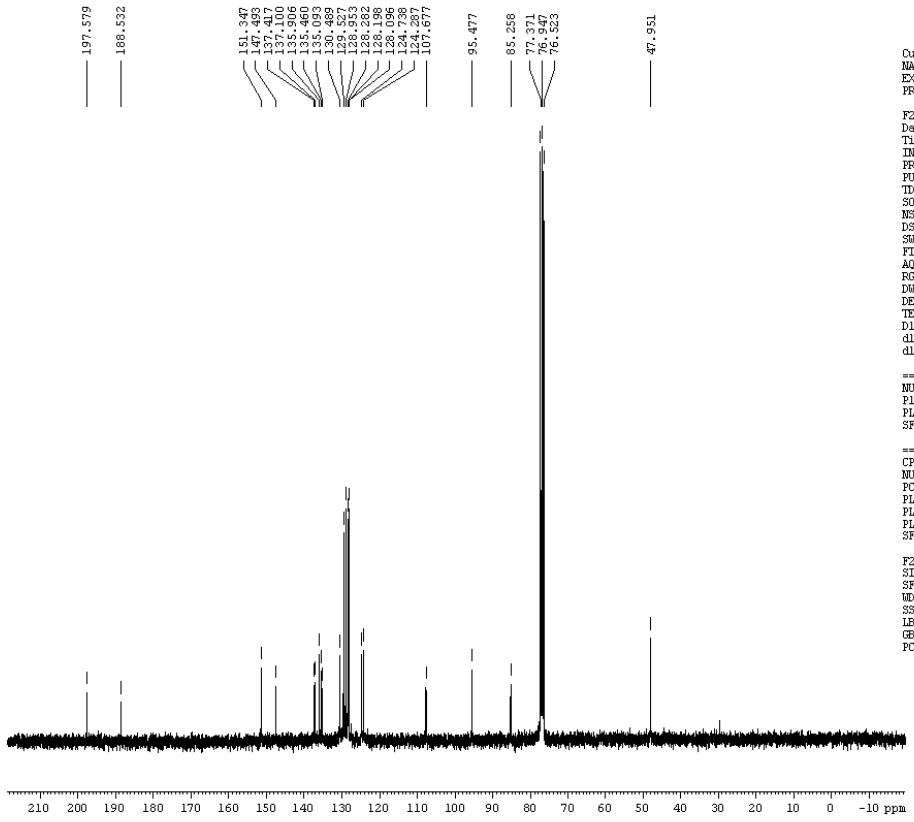

```
Current Data Parameters
NAME       2-furyl 4-ome
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20130916
Time              17.25
INSTRUM             av300
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  321
DS                    4
SWH           21097.047 Hz
FIDRES         0.321915 Hz
AQ             1.5532532 sec
RG               14596.5
DW                23.700 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PL1                -5.00 dB
SFO1          75.4767751 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              20.31 dB
PL13              10.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                  32768
SF            75.4677630 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



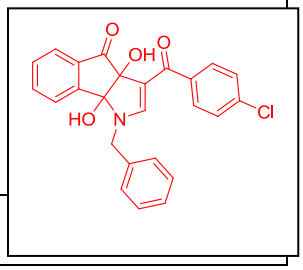
^{13}C NMR Spectrum of 4t



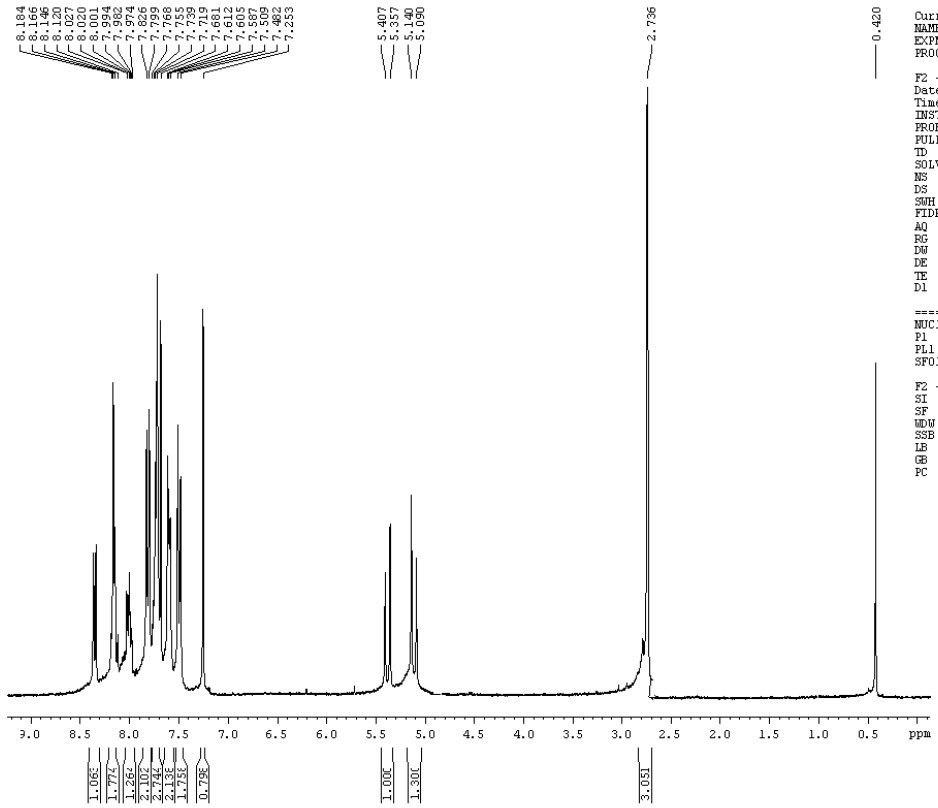
^{1}H NMR Spectrum of 4u

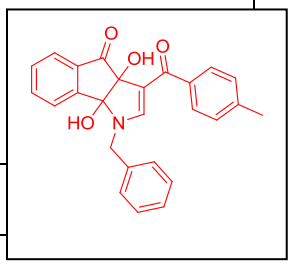



```
Current Data Parameters
NAME      4-cl ek benzyl
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131003
Time              14.57
INSTRUM           av300
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 914
DS                   4
SWH          17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG               14596.5
DW               27.800 usec
DE                6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               12.50 usec
PL1              -5.00 dB
SF01        75.4752653 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2               0.00 dB
PL12             20.31 dB
PL13             10.00 dB
SF02       300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677588 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

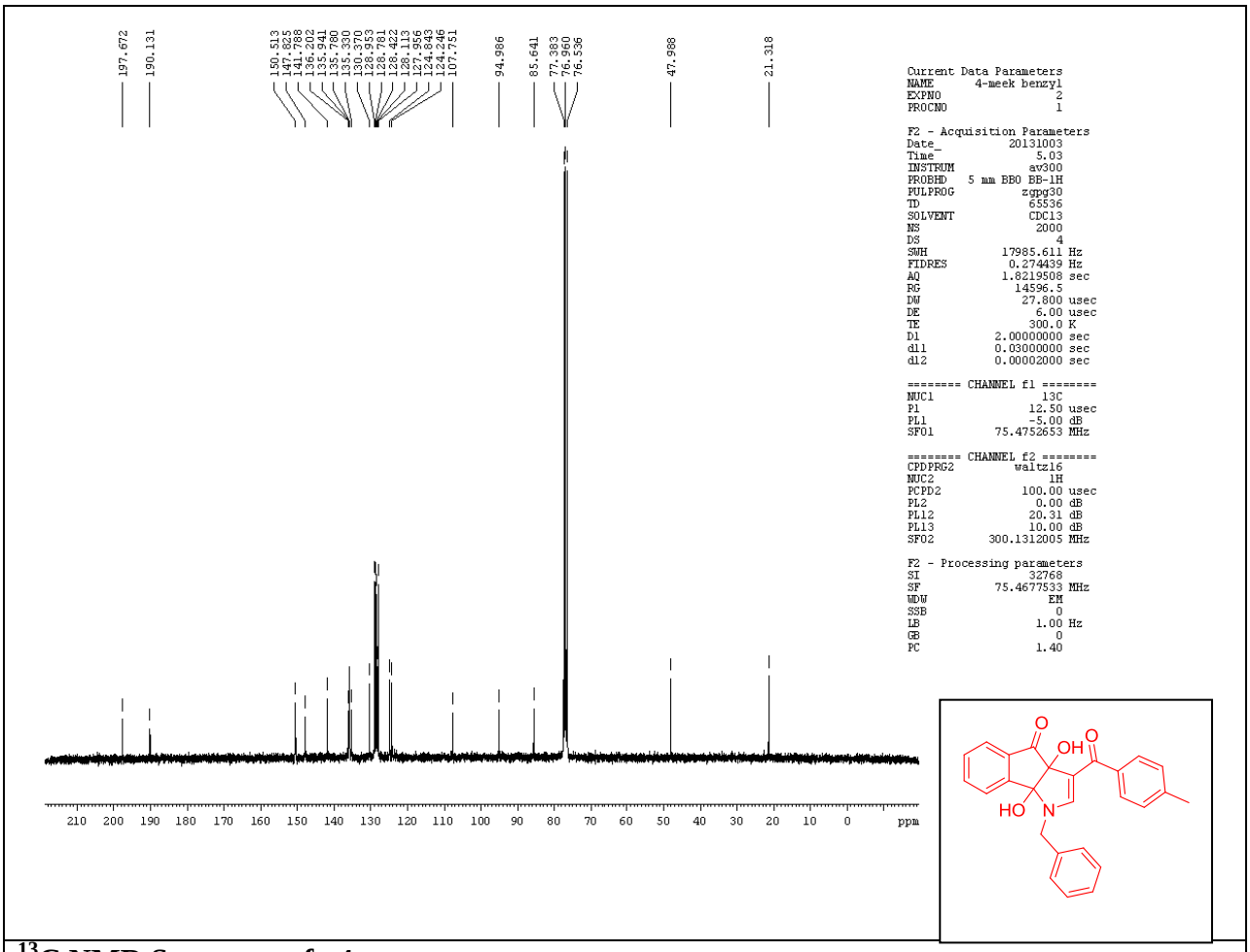


^{13}C NMR Spectrum of 4u

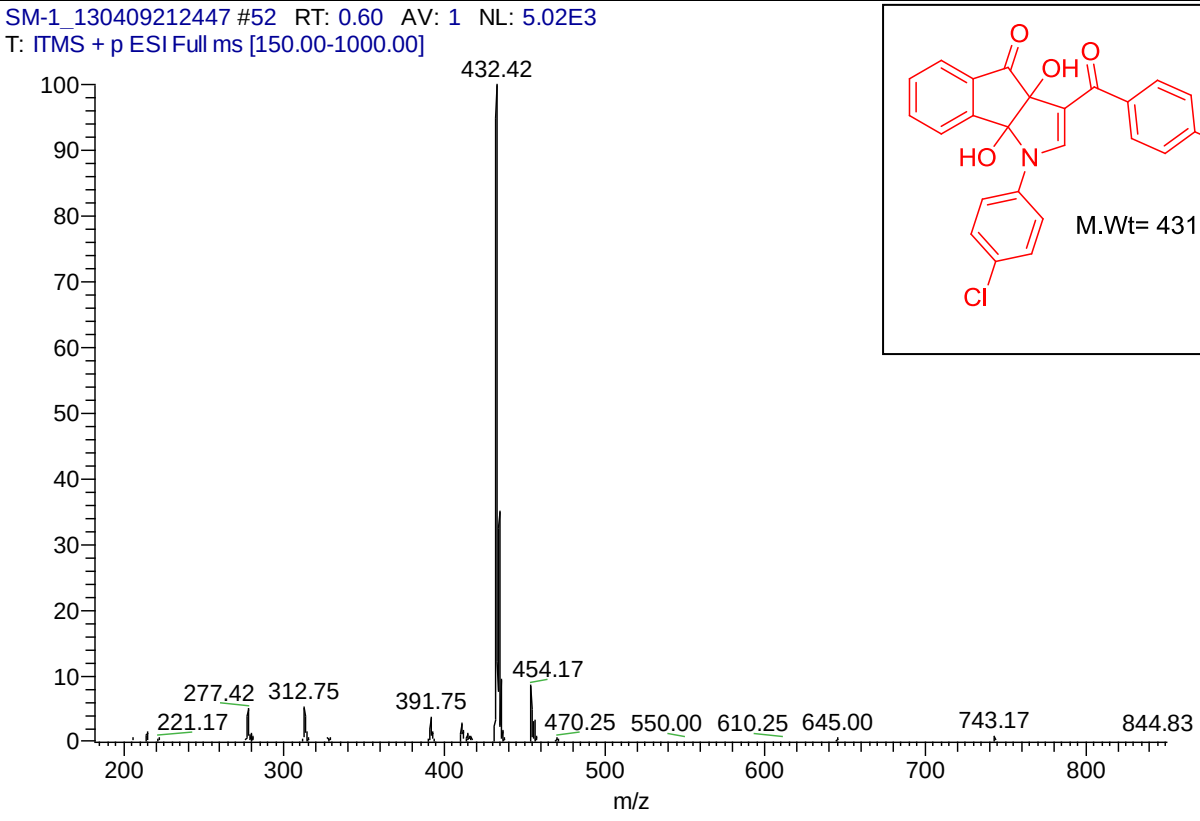




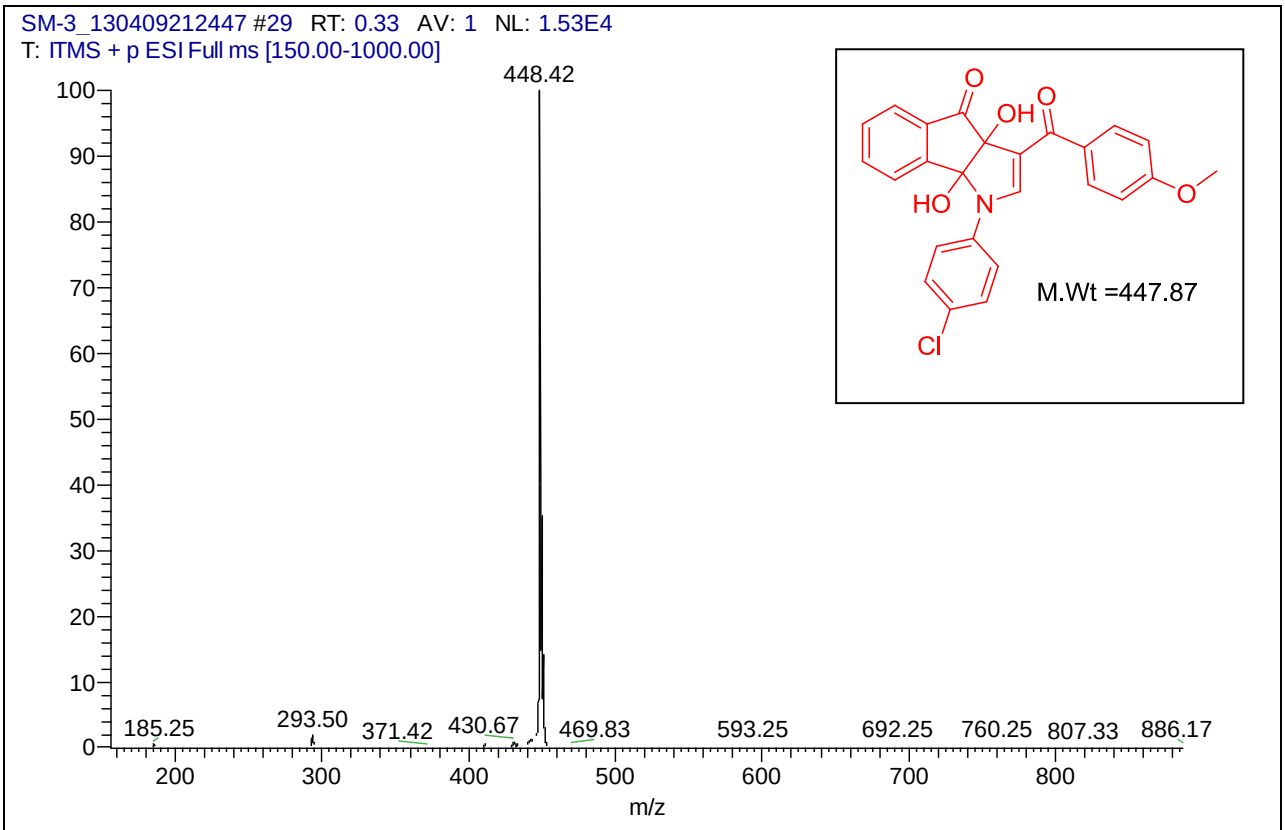
^{1}H NMR Spectrum of 4v



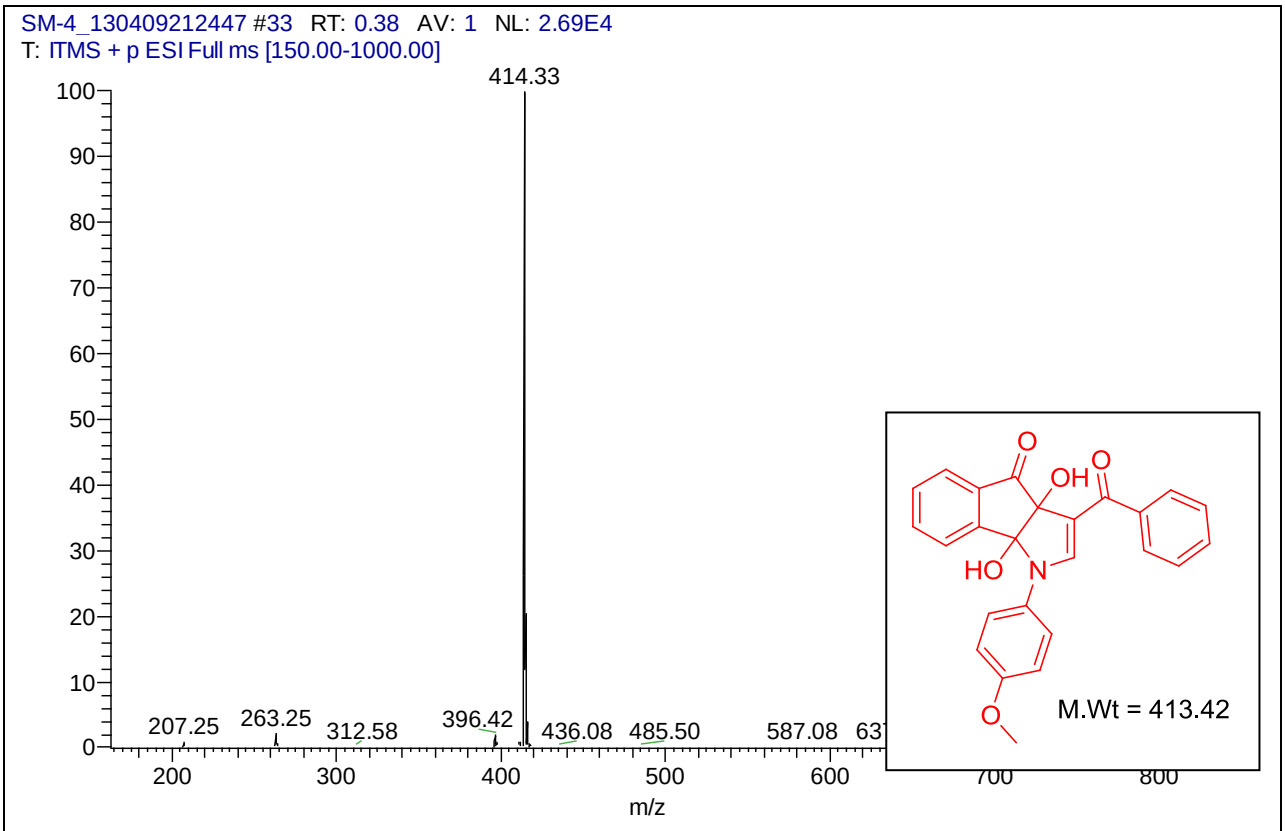
^{13}C NMR Spectrum of 4v



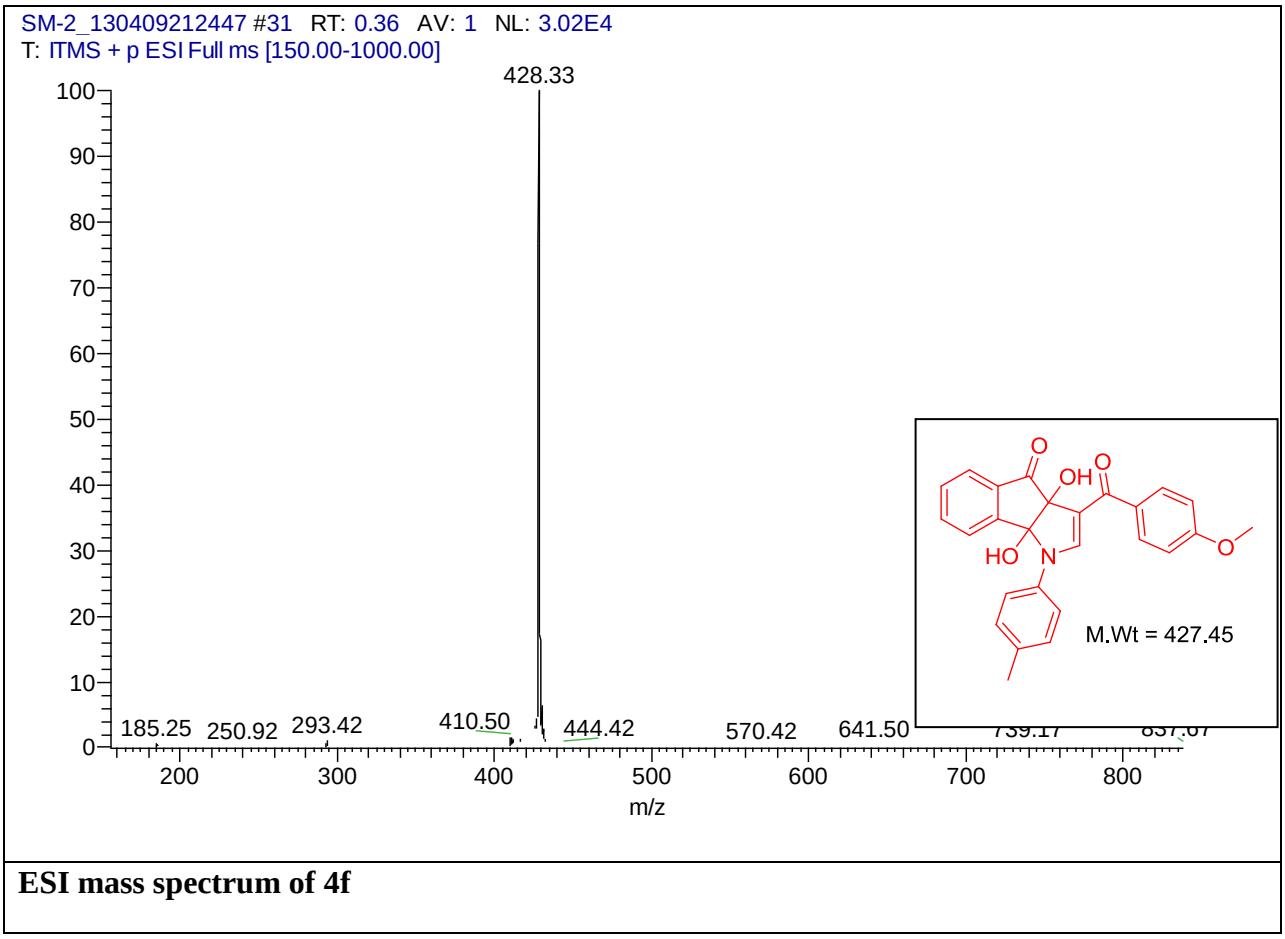
ESI mass spectrum of 4c



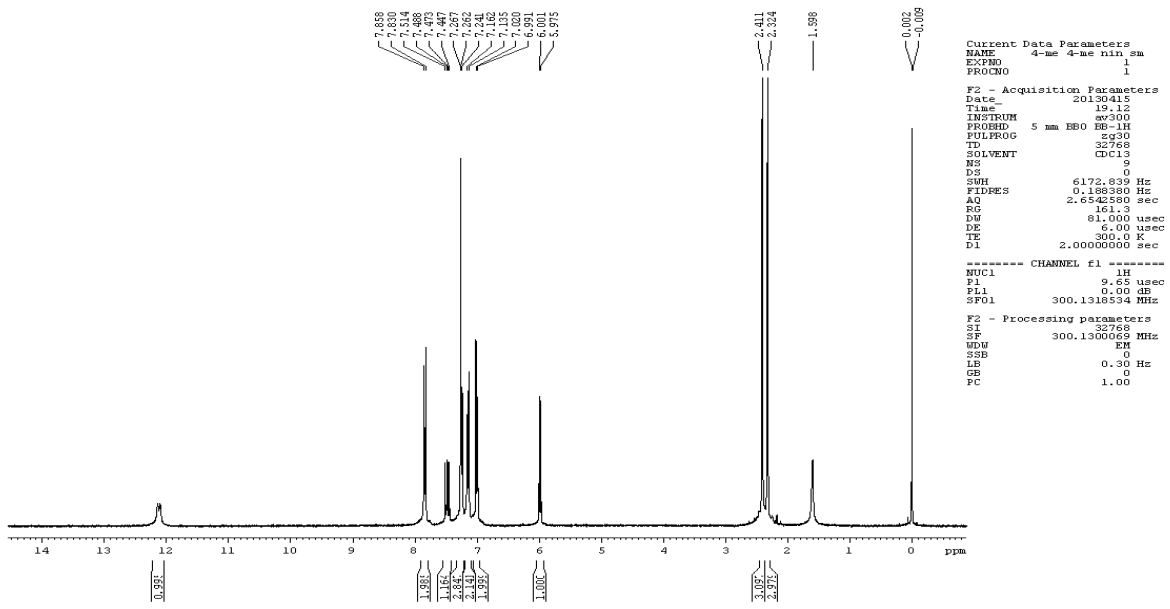
ESI mass spectrum of 4d



ESI mass spectrum of 4e

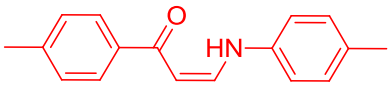


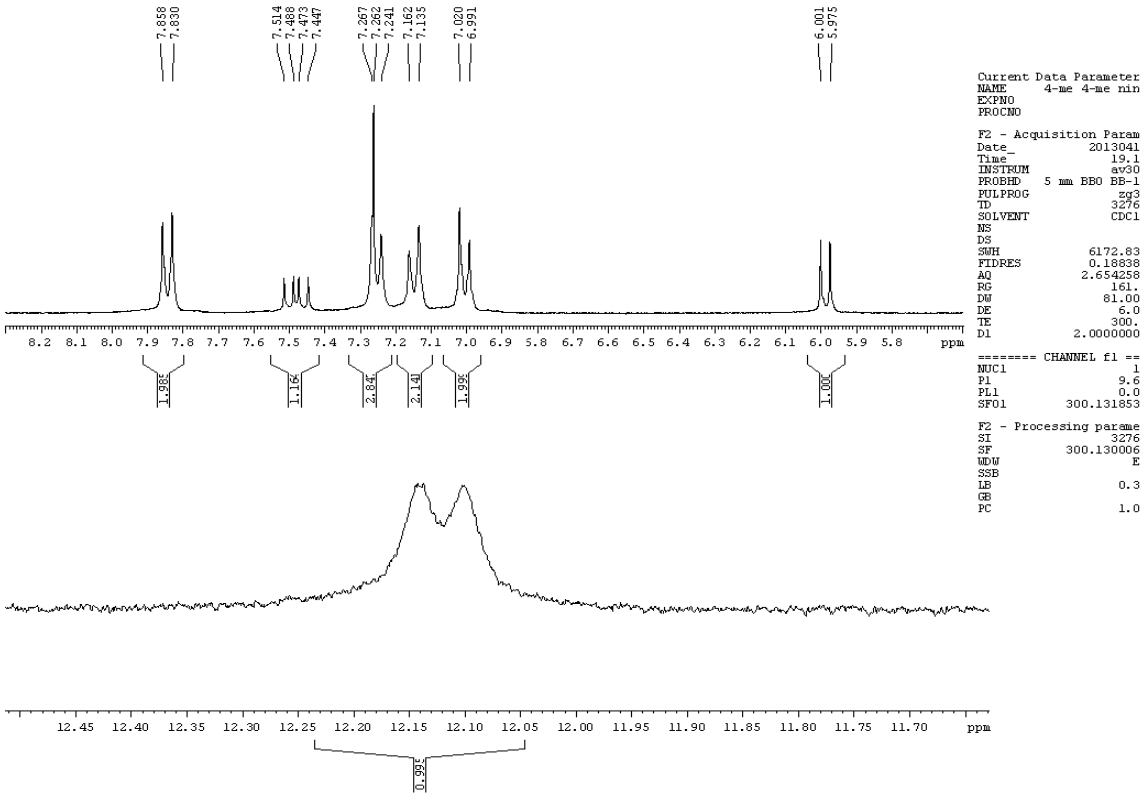
ESI mass spectrum of 4f



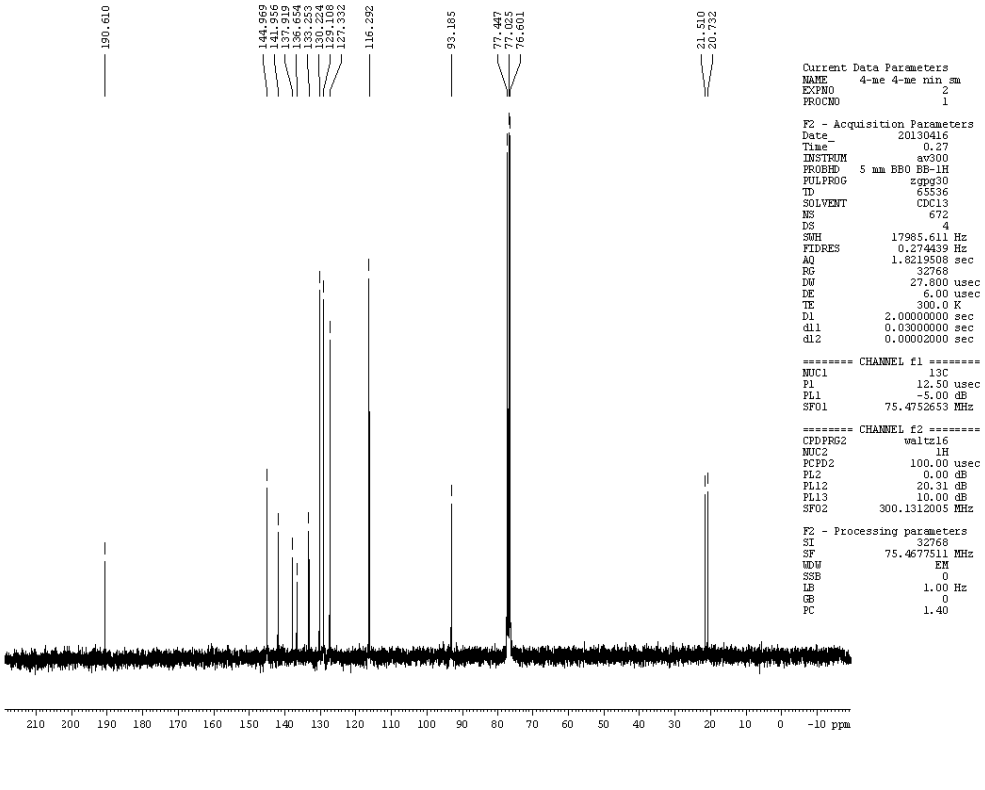

```
Current Data Parameters
NAME      4-me 4-me nin sm
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20130415
Time              19.12
INSTRUM           av300
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   9
DS                   0
SWH           6172.839 Hz
FIDRES        0.188380 Hz
AQ            2.6542580 sec
RG                161.3
DW               81.000 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.65 usec
PL1               0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI               32768
SF          300.1300069 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

^{1}H NMR Spectrum of 5a

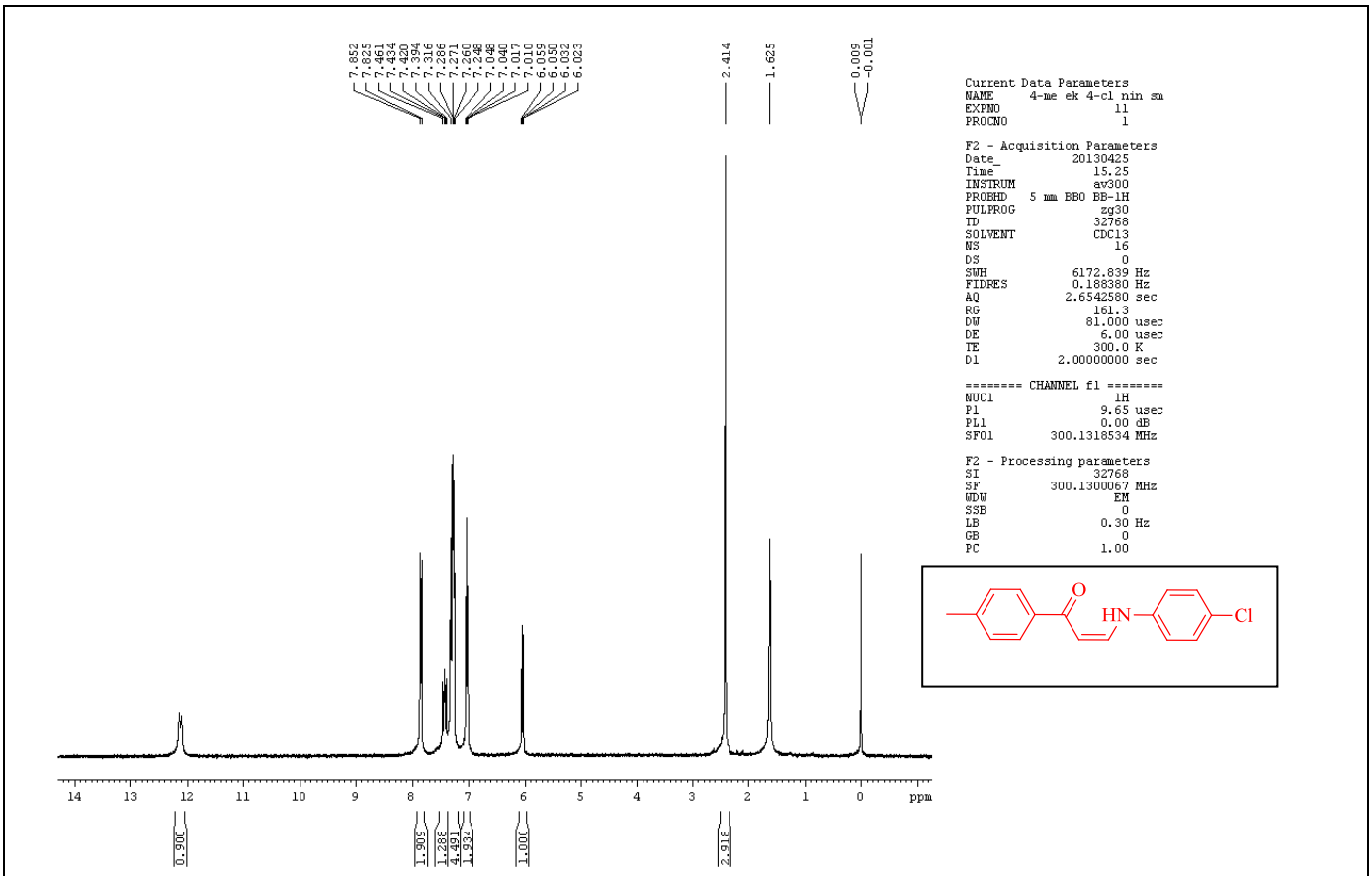




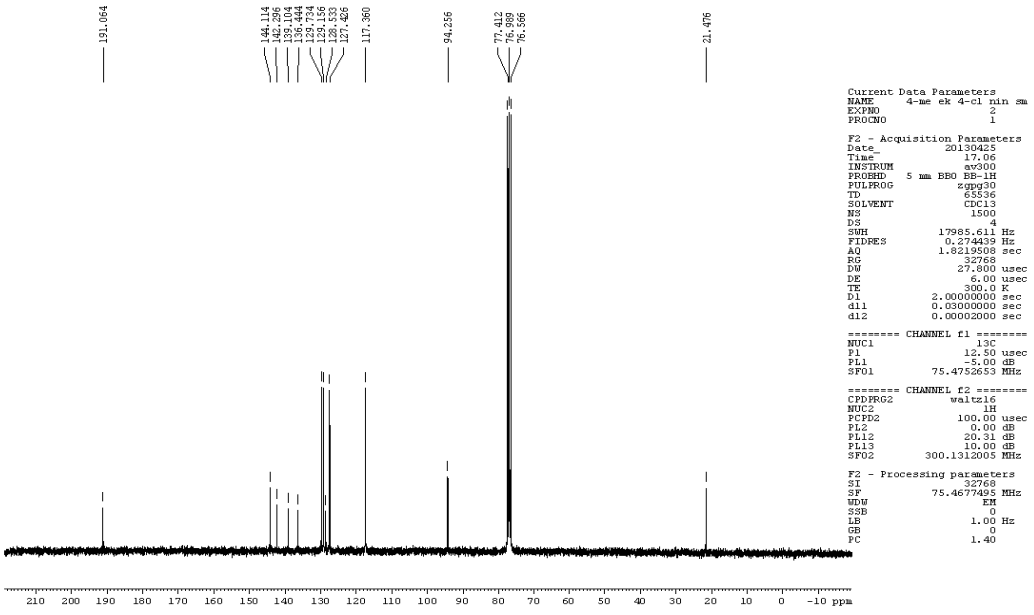
^{1}H NMR (expanded) Spectrum of 5a



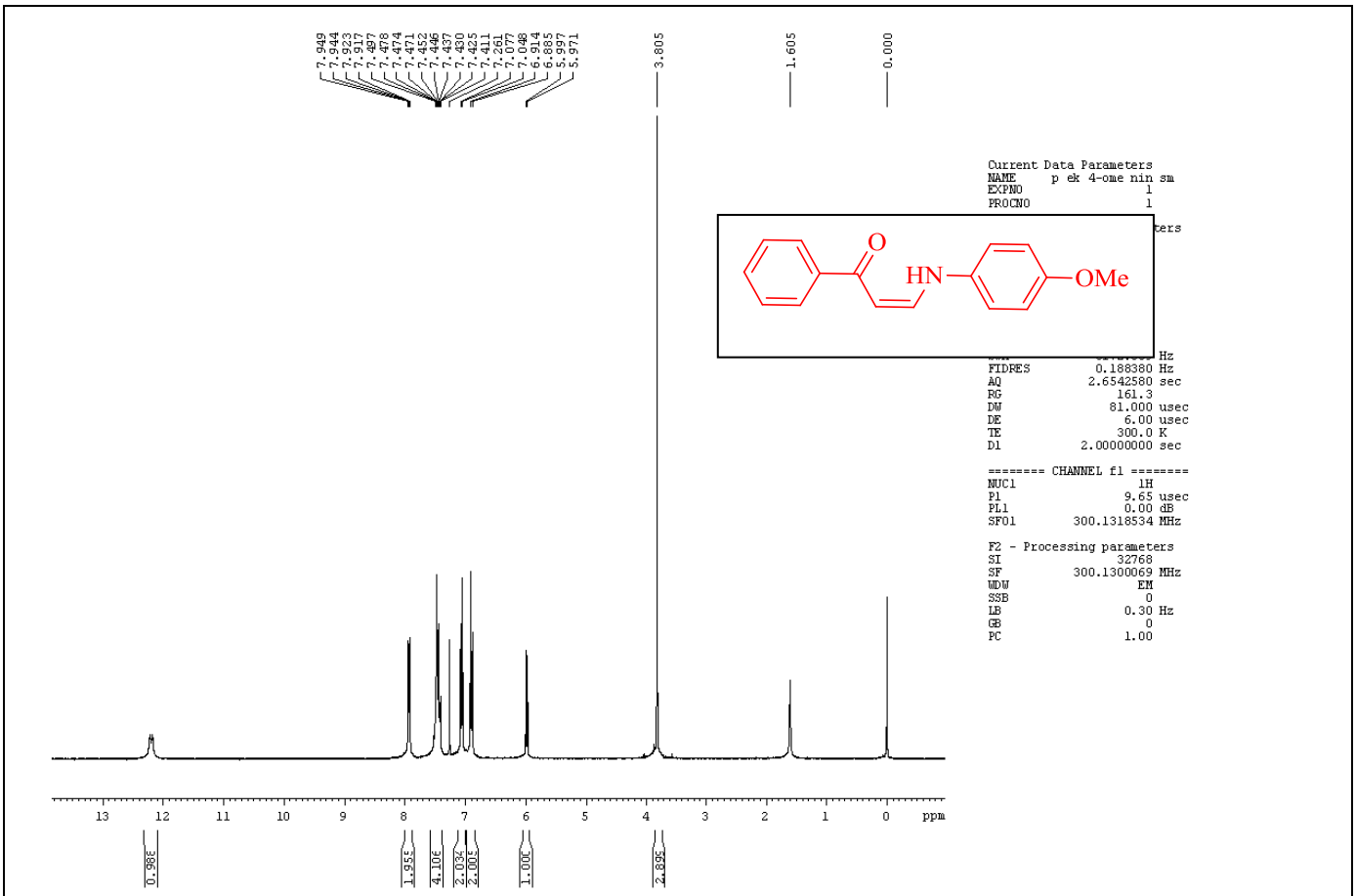
^{13}C NMR Spectrum of 5a



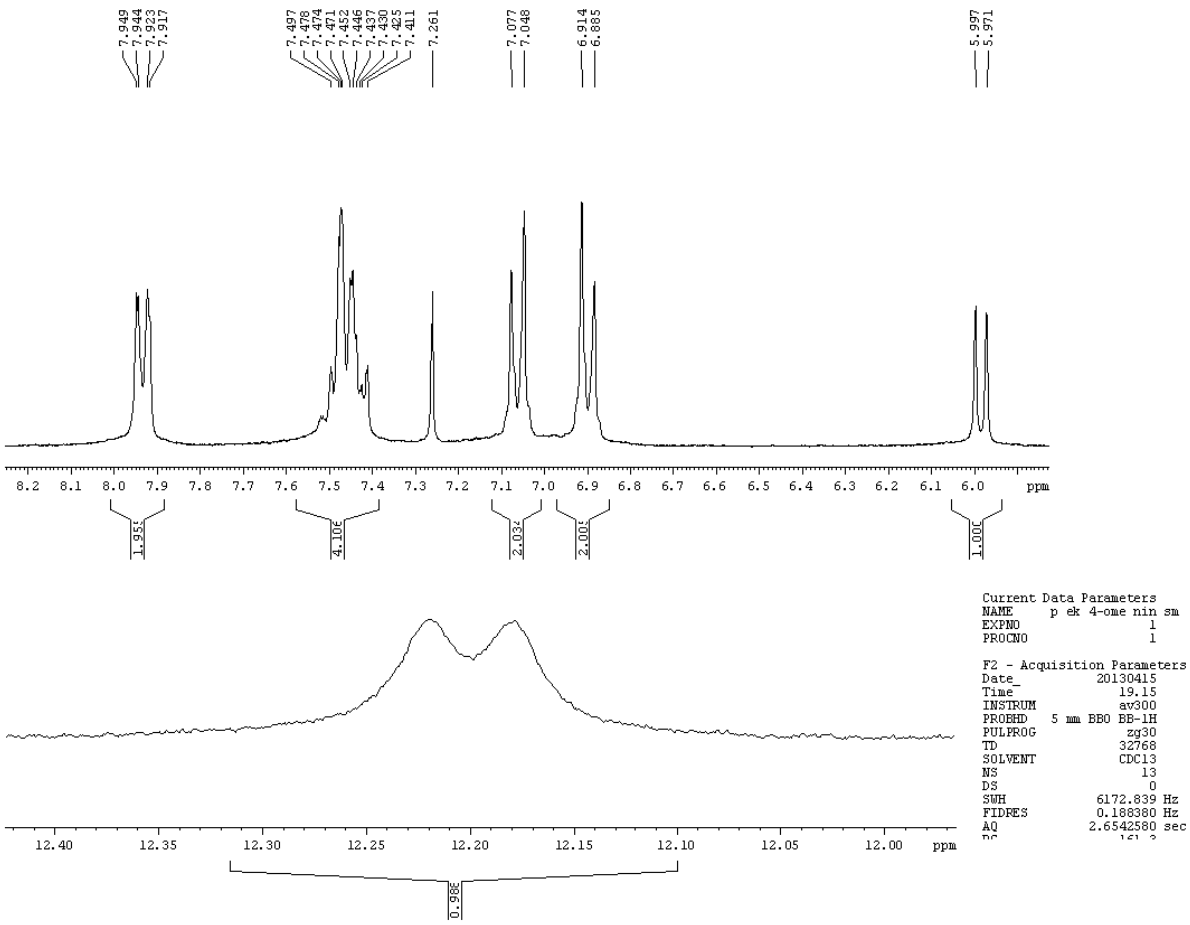
^{1}H NMR Spectrum of 5b



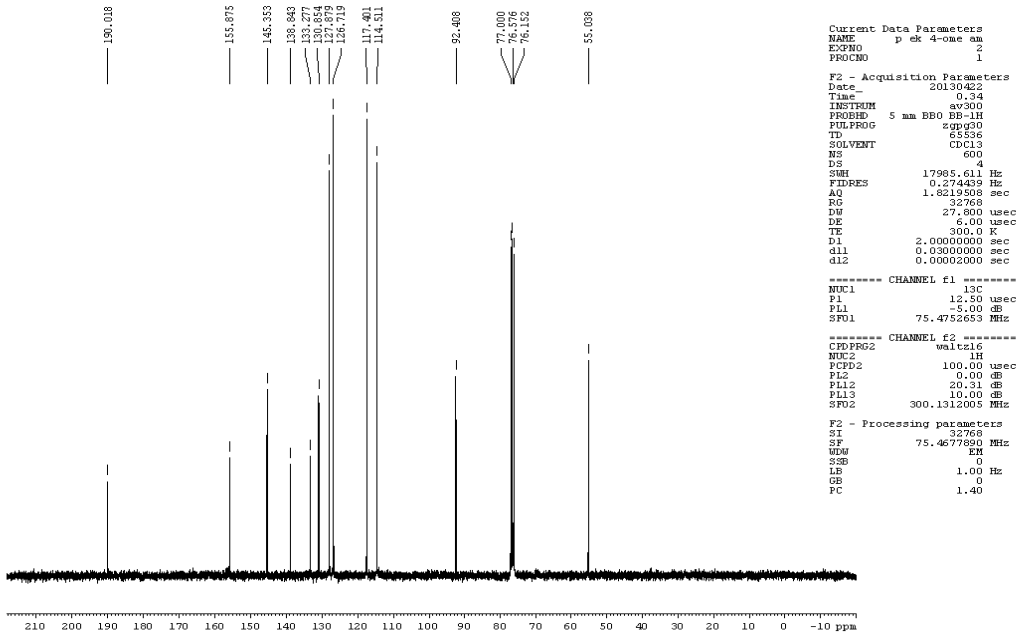
^{13}C NMR Spectrum of 5b



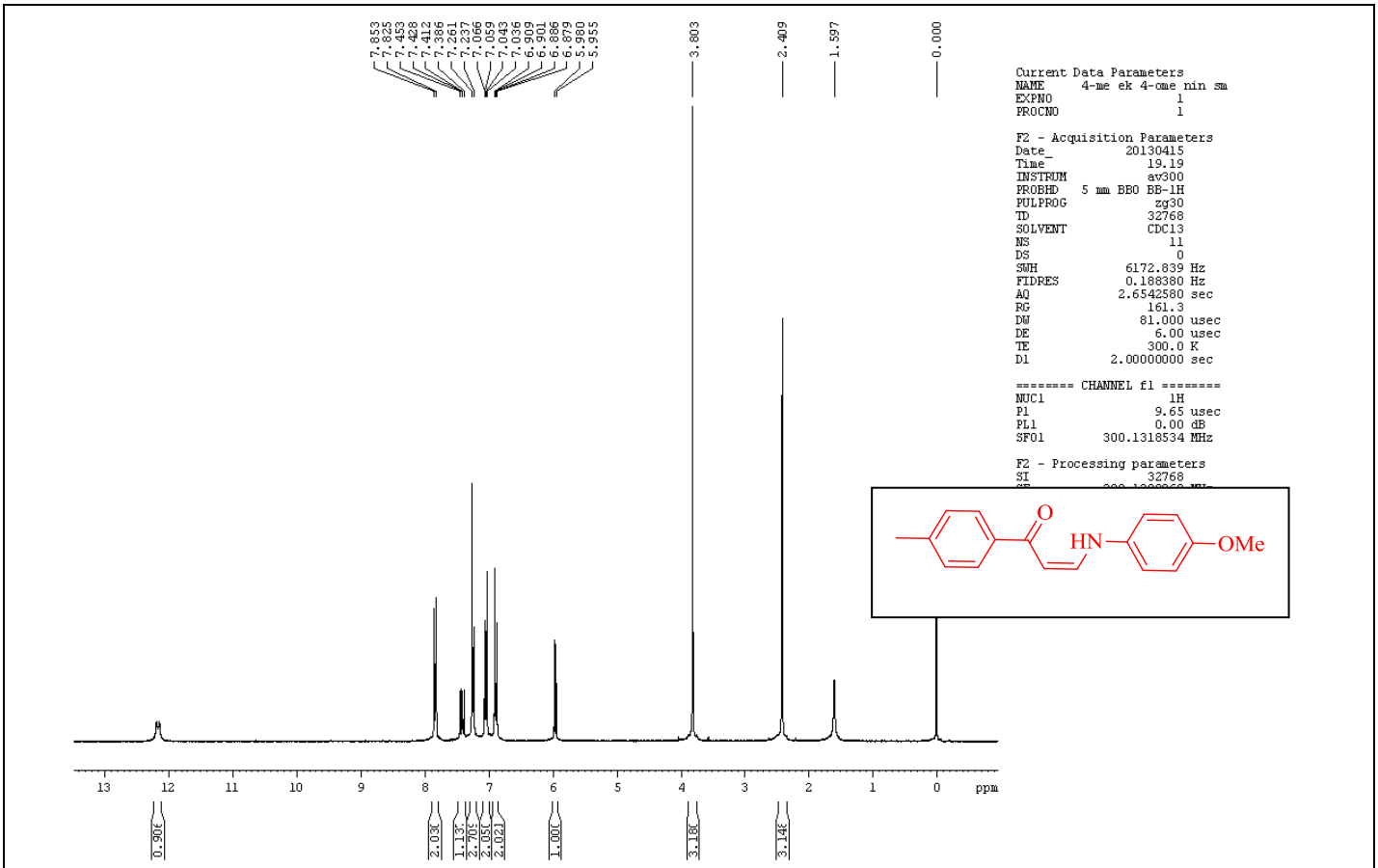
^{1}H NMR Spectrum of 5c



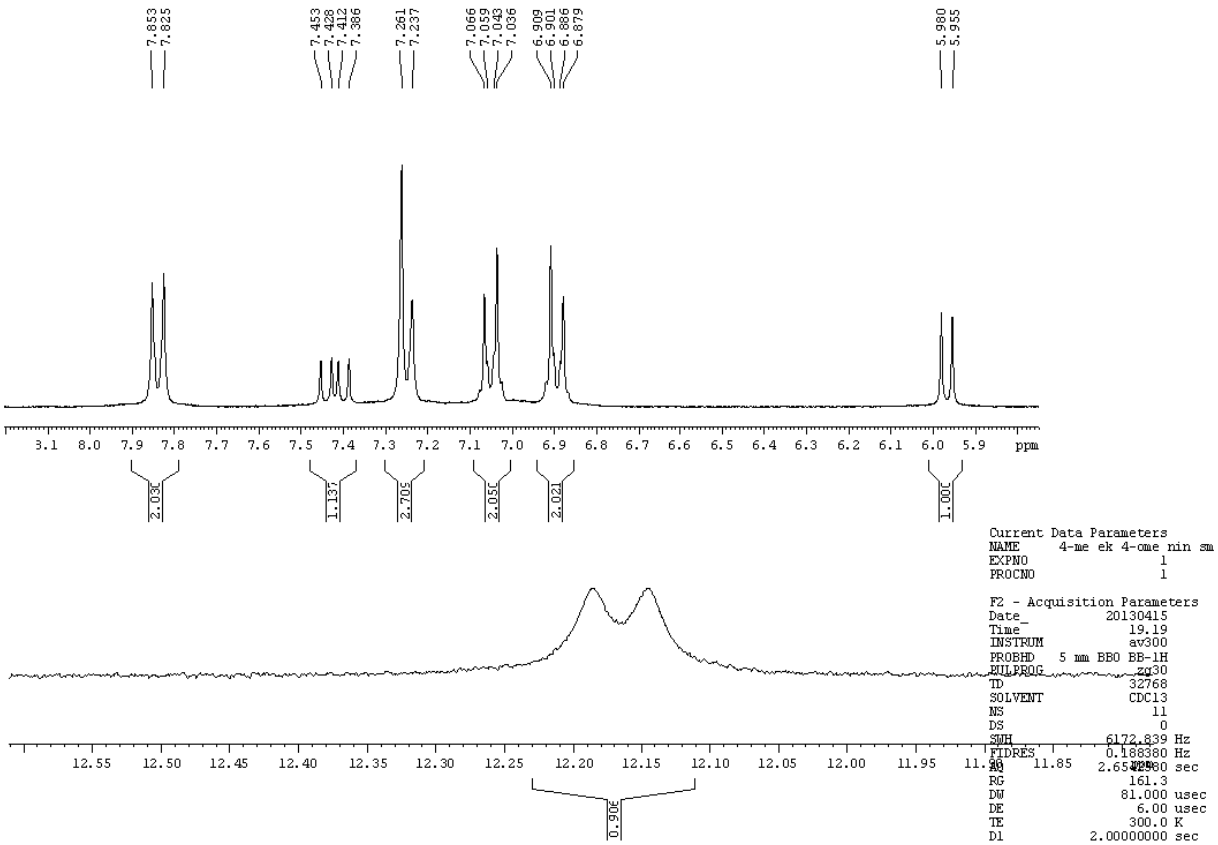
^{1}H NMR (expanded)Spectrum of 5c



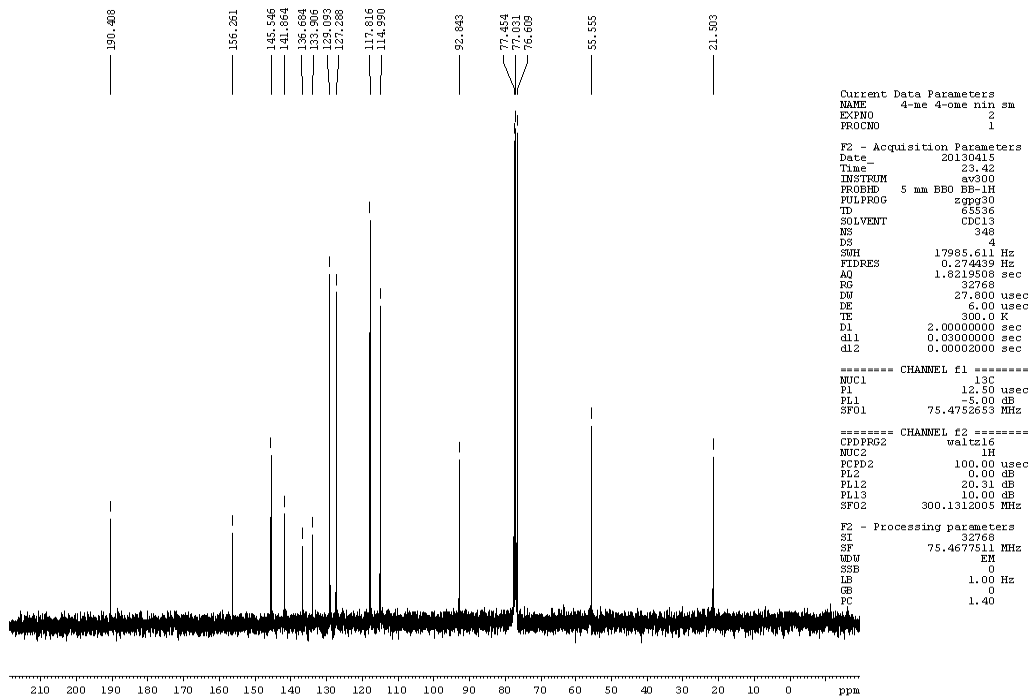
^{13}C NMR Spectrum of 5c



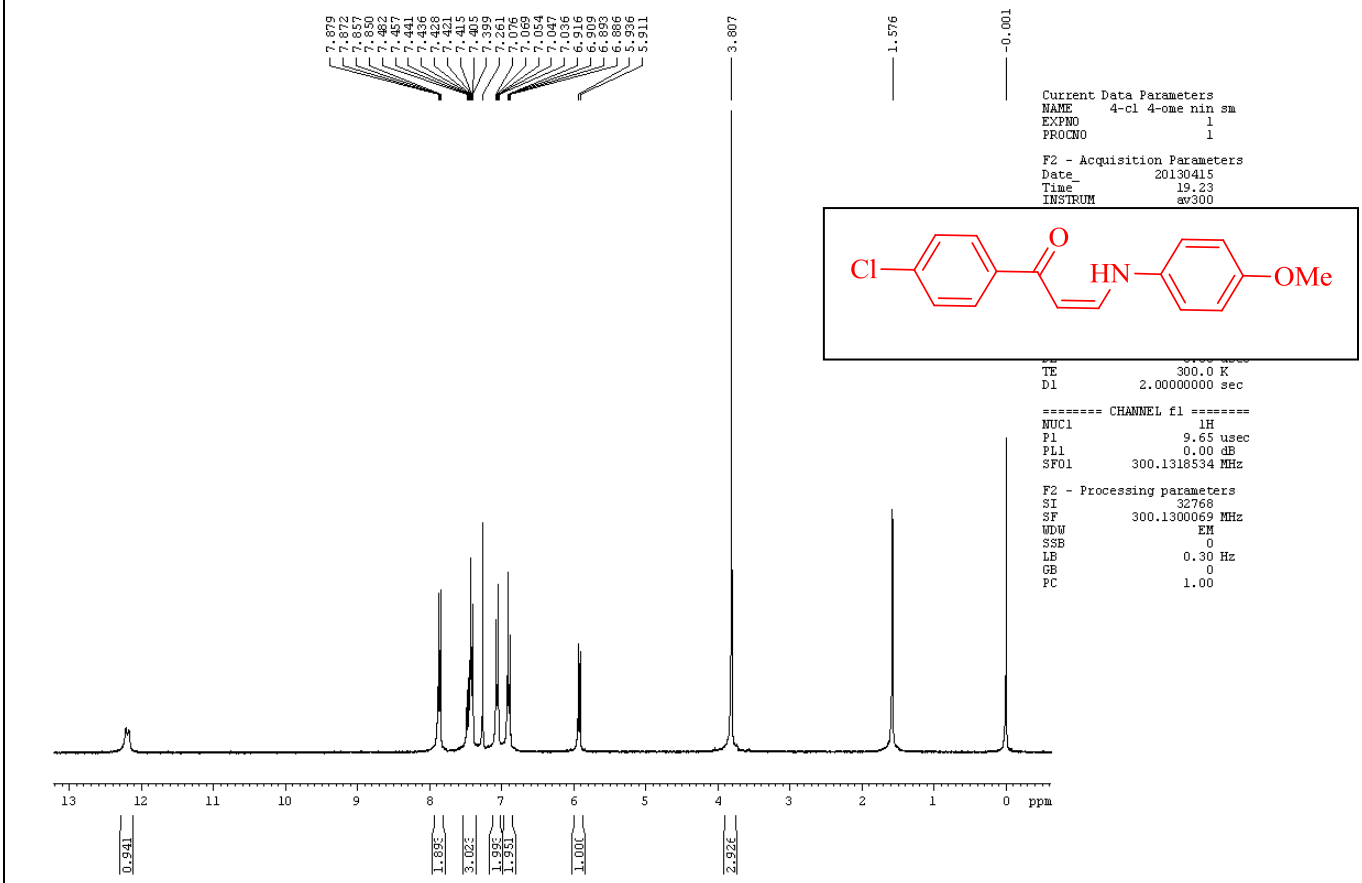
^{1}H NMR Spectrum of 5d



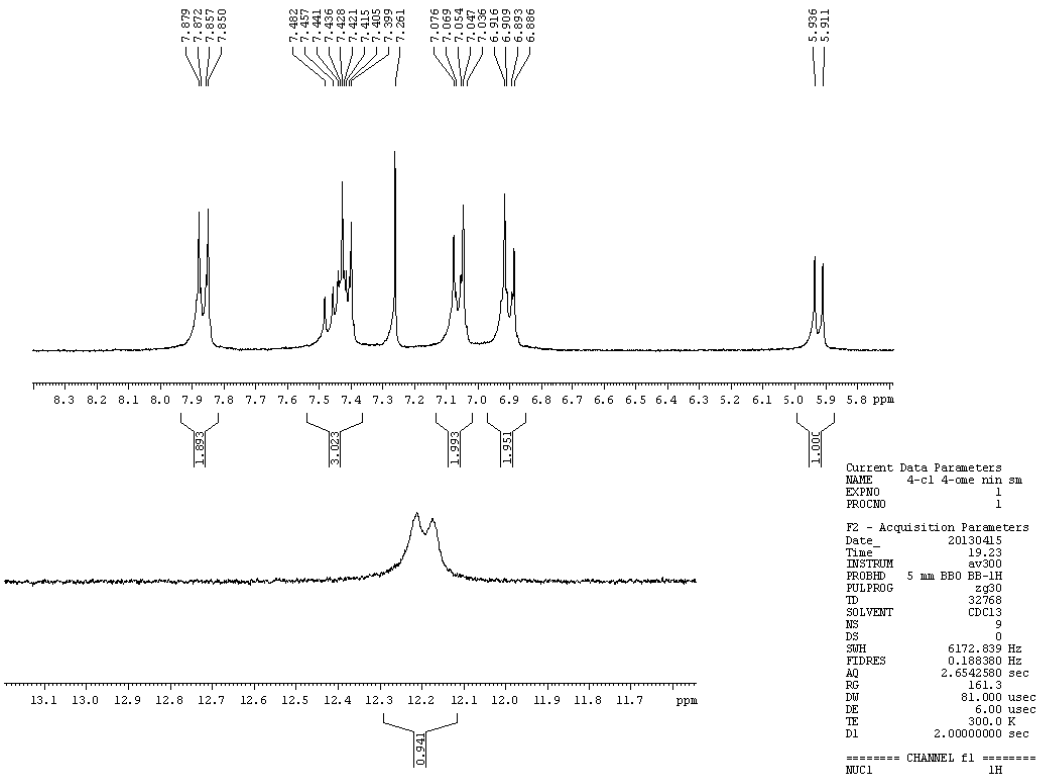
^{1}H NMR (expanded)Spectrum of 5d



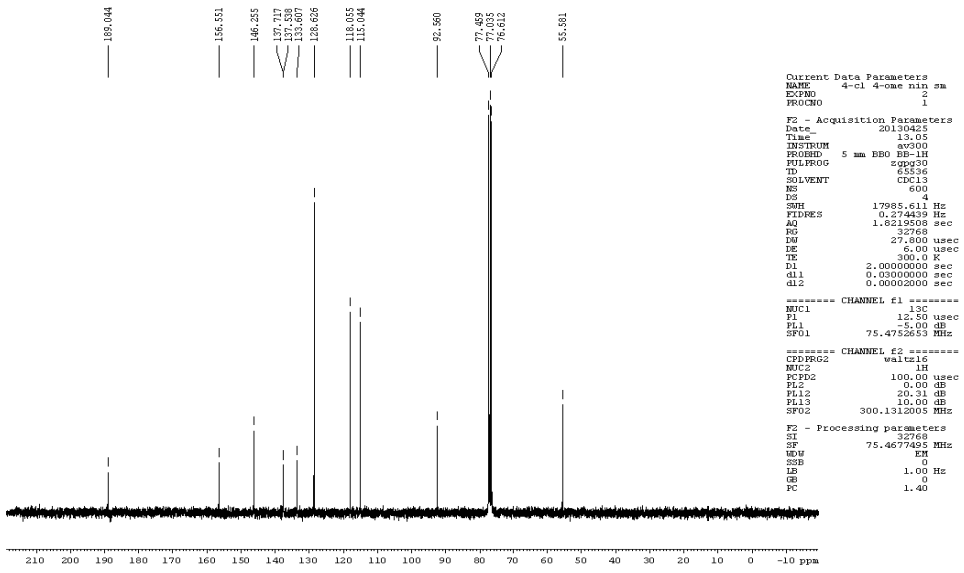
^{13}C NMR Spectrum of 5d



^{1}H NMR Spectrum of 5e



^{1}H NMR (expanded)Spectrum of 5e



^{13}C NMR Spectrum of 5e