

NMR Spectra

for

**Synthesis of Hydrazinoheterocycles from Morita-Baylis-Hillman Adducts of Nitroalkenes with Azodicarboxylates**

Vaijinath Mane, Jyoti Pandey, Narasimham Ayyagari, Chandan Dey, Raju Kale and Irishi N. N. Namboothiri\*

Department of Chemistry, Indian Institute of Technology Bombay, Mumbai 400 076, India

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```

MBHDMAD-1
exp10 PROTON

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file /export/home/+
inn3/2006/Nov06/21-
st/MBH-DMAD-1.fid spin    not used
pw90          pw90    8.500
ACQUISITION alfa   -7.925
sw      6000.0  FLAGS
at      1.589  fl  n
np      23888  in  n
fb      not used dp  y
bs      4     hs  nn
di      1.000  PROCESSING
nt      400   1b  0.10
ct      48    fn  not used
TRANSMITTER DISPLAY
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sfrq   399.883 wp  4824.9
tof     200.0 rfi  3659.0
tpwr    55  rfp  2983.1
pw     4.250  rp  -58.9
DECOUPLER 1p  29.9
dn      C13 PLOT
dof     g   wc  250
dm      nnn sc  0
dmm     c   vs  99
dpwr    51  th  30
dmf    17100 nm  ph

```

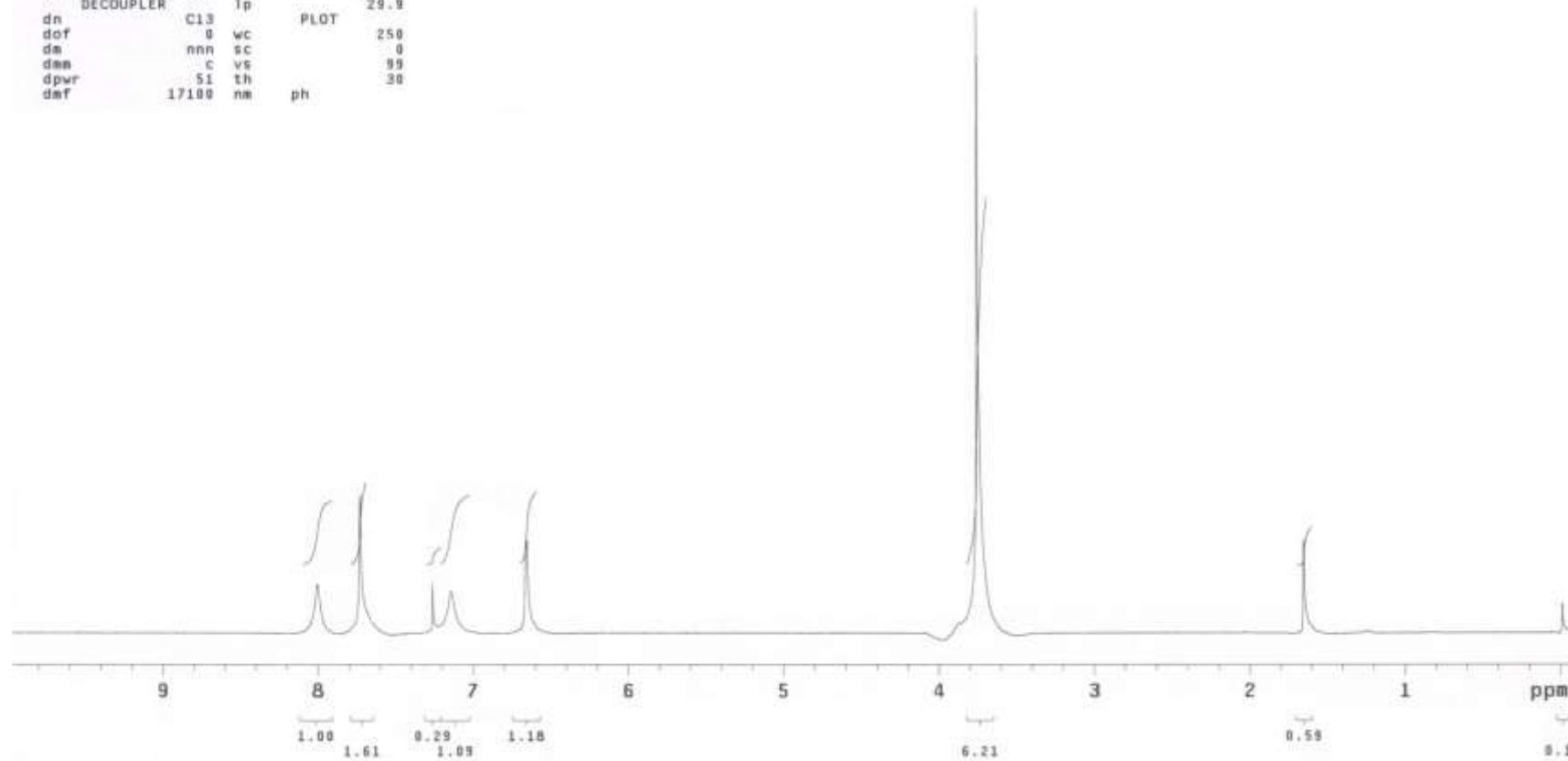
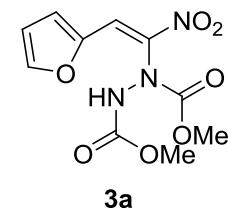
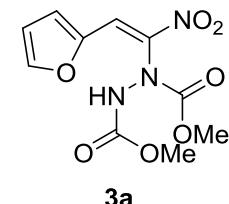


Figure S1. <sup>1</sup>H NMR Spectrum of 3a

QBH-DHAD-1-C13

exp16 CARBON

SAMPLE SPECIAL  
date Dec 1 2006 temp not used  
solvent CDCl<sub>3</sub> gain 34  
file exp spin not used  
ACQUISITION nst 0.008  
sw 25125.6 pw90 14.000  
at 1.199 alfa 20.000  
np 60270 FLAGS  
fb 13800 ll n  
bs 4 in n  
d1 1.000 dp y  
nt 50000 hs nn  
ct 156  
TRANSMITTER lb 2.00  
tn C13 fn not used  
sfrq 100.561 DISPLAY  
tof 1554.3 sp -68.2  
tpwr 56 wp 22153.5  
pw 7.000 rfp 9282.8  
DECOUPLER rfp 7742.3  
dn H1 rp 137.9  
dof 0 lp -336.9  
dmr VVY PLOT  
dmm w wc 250  
dpwr 41 sc 0  
dmr 11900 vs 51  
dmf th 2  
nm ph



3a

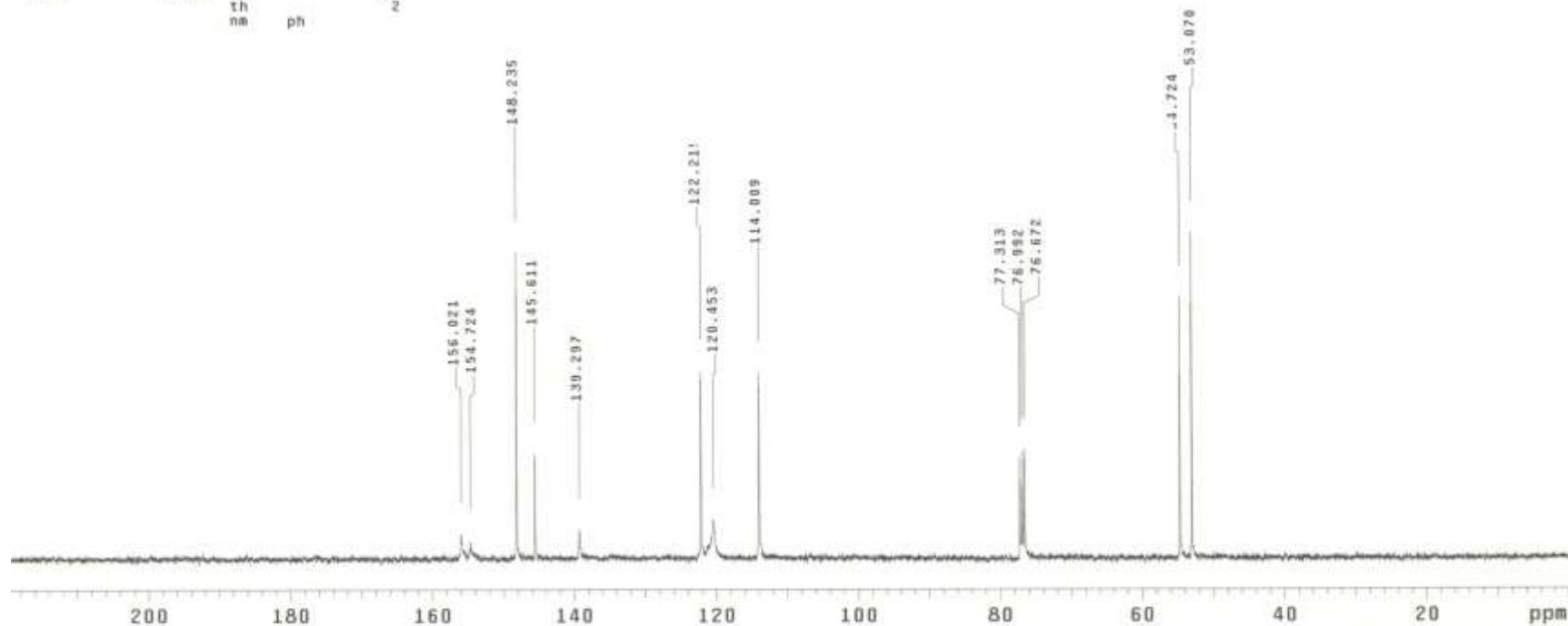


Figure S2. <sup>13</sup>C NMR Spectrum of 3a

Current Data Parameters  
NAME INN-MVD-CINNAMYL DIAD  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

Date 20150327  
Time 12.36  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 12  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 299.8 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
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SP 500.1300125 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

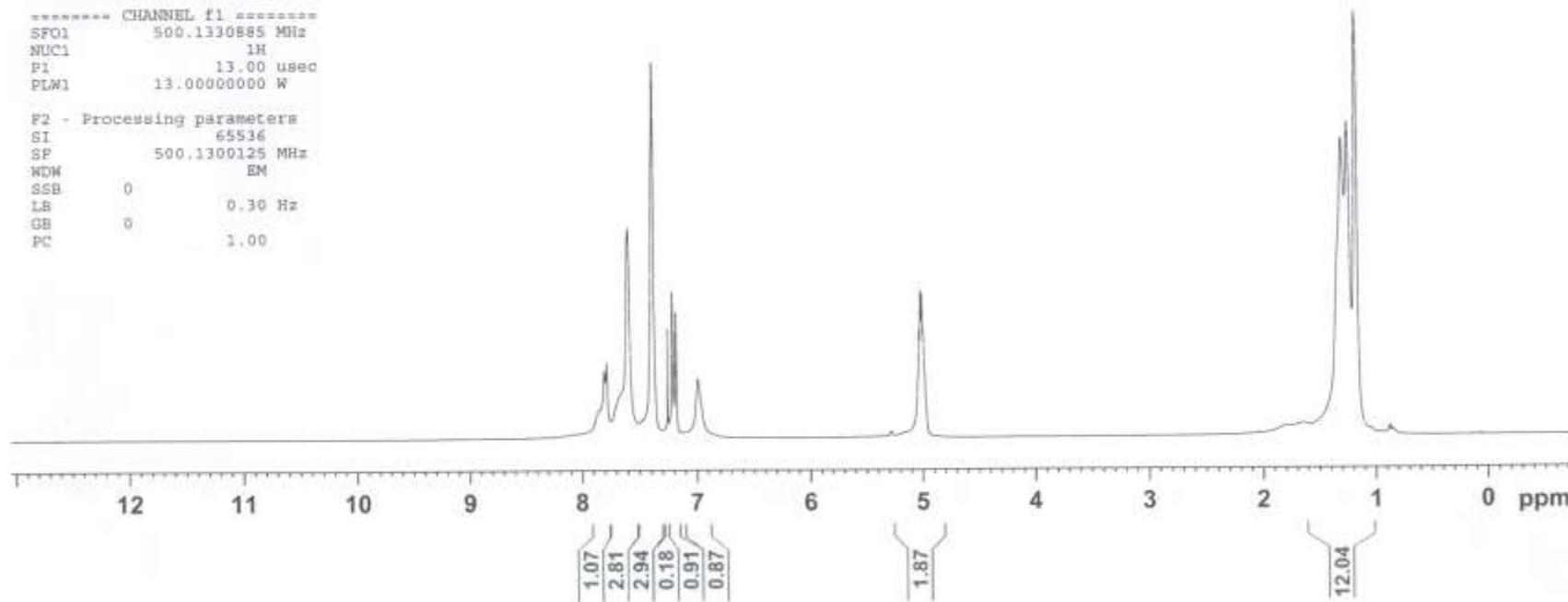
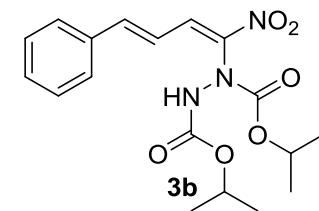


Figure S3.  $^1\text{H}$  NMR Spectrum of **3b**

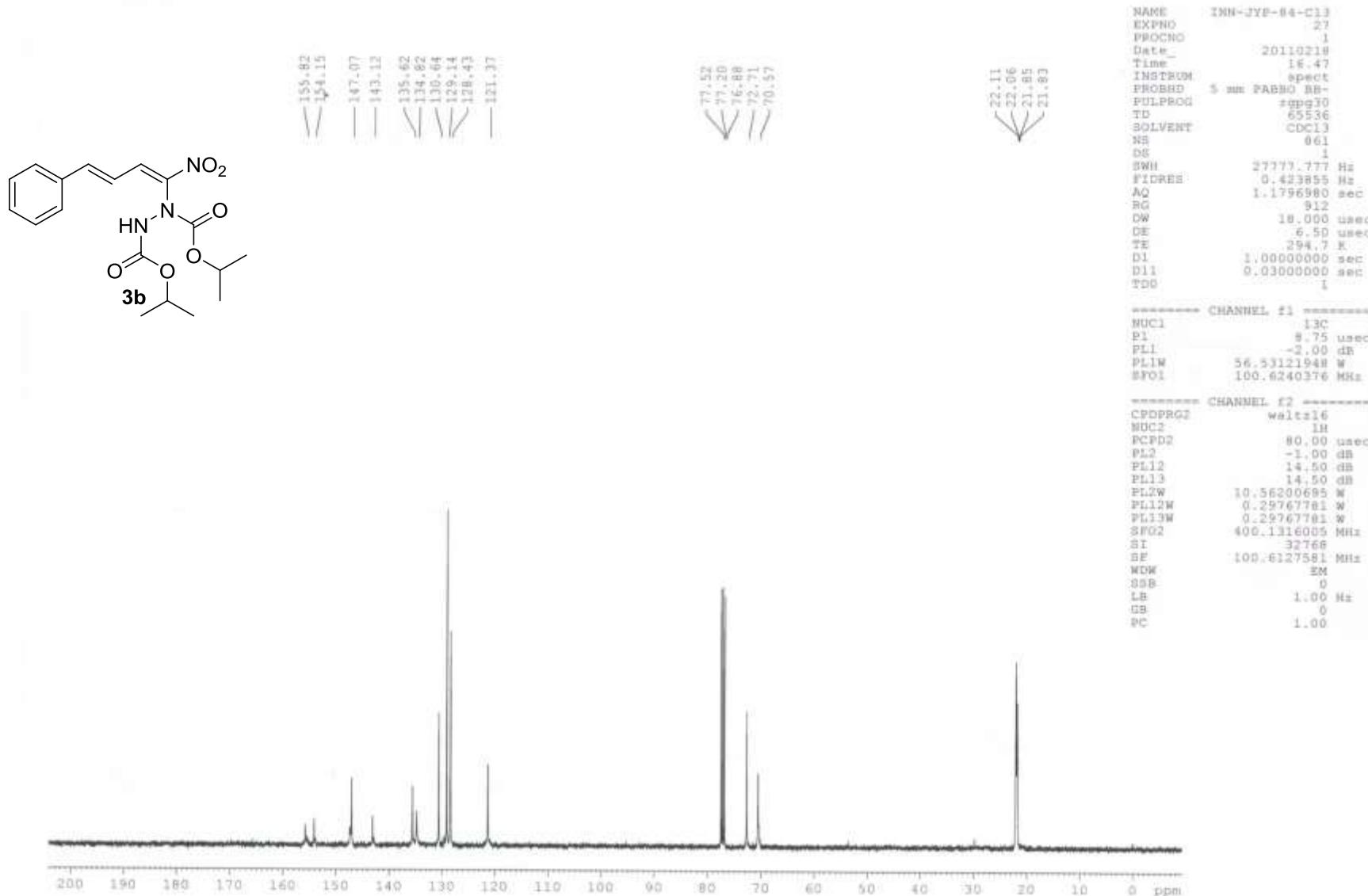


Figure S4.  $^{13}\text{C}$  NMR Spectrum of **3b**

Current Data Parameters  
 NAME INN-RRK-33-1H  
 EXPNO 11  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20131004  
 Time 22:13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 14  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 30.72  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 296.9 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 SFO1 500.1330885 MHz  
 NUC1 1H  
 P1 13.00 usec  
 PIW1 13.0000000 W  
 F2 - Processing parameters  
 SI 65536  
 SF 500.1300000 MHz  
 WDW BM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

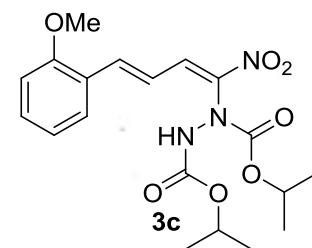
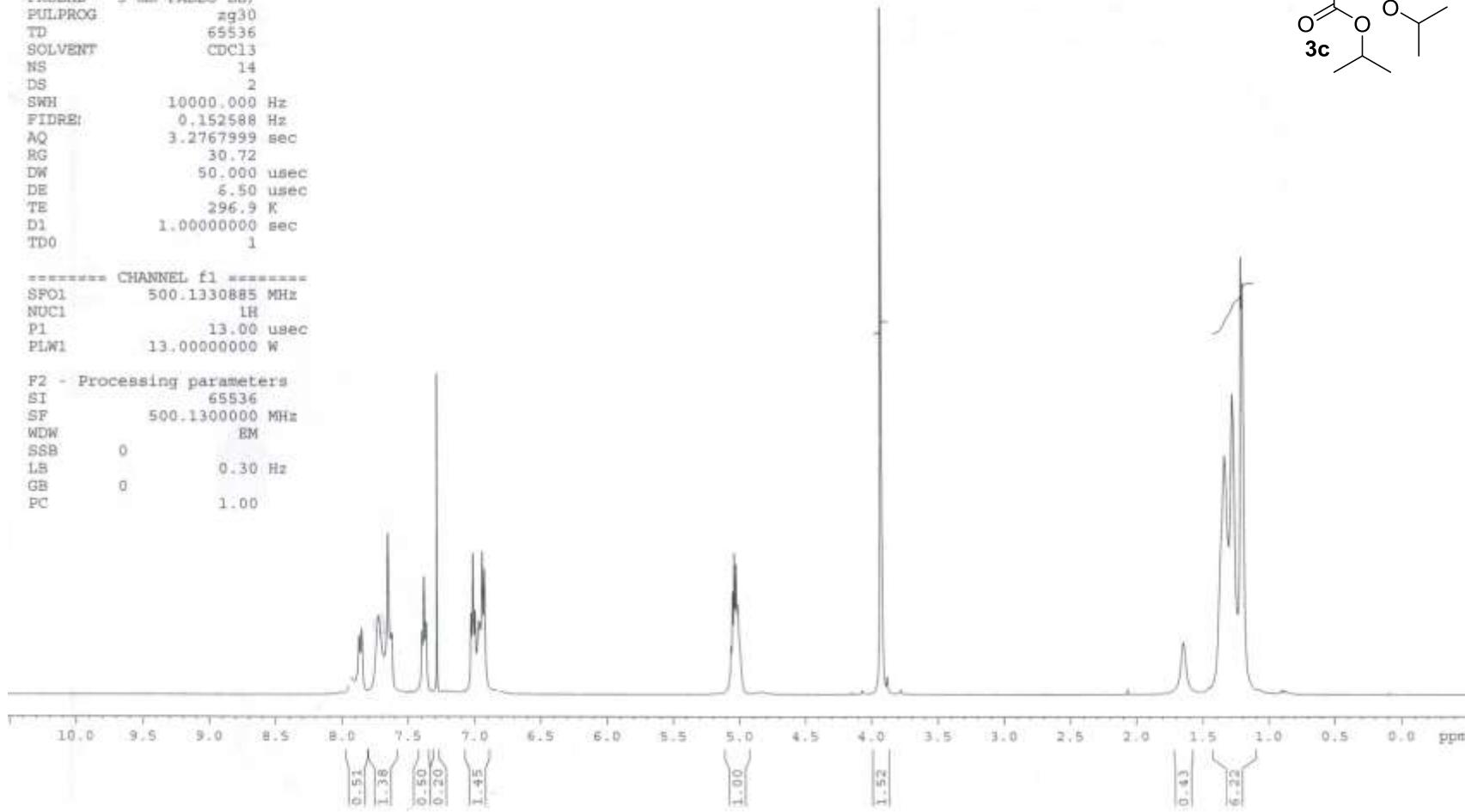


Figure S5.  $^1\text{H}$  NMR Spectrum of **3c**

NAME INN-JYP-103-13C  
 EXPNO 8  
 PROCNO 1  
 Date 20110404  
 Time 10.47  
 INSTRUM spect  
 PROBHD 5 mm zABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 112  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 NUC1 13C  
 F1 8.75 usec  
 PLL1 -2.00 dB  
 PLL1W 56.53121948 W  
 SFO1 100.6228298 MHz

----- CHANNEL f2 -----  
 CPDPRG2 waltz16  
 NUC2 1H  
 ECPD2 80.00 usec  
 PL2 -1.00 dB  
 PLL2 14.50 dB  
 PLL3 14.50 dB  
 PLL2W 10.56200695 W  
 PLL2W 0.29767781 W  
 PLL3W 0.29767781 W  
 SFO2 400.1316005 MHz  
 SI 32768  
 SF 100.6127566 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

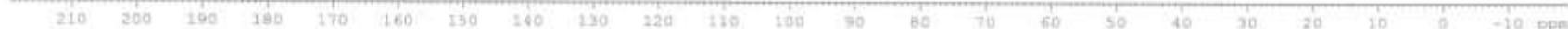


Figure S6.  $^{13}\text{C}$  NMR Spectrum of **3c**

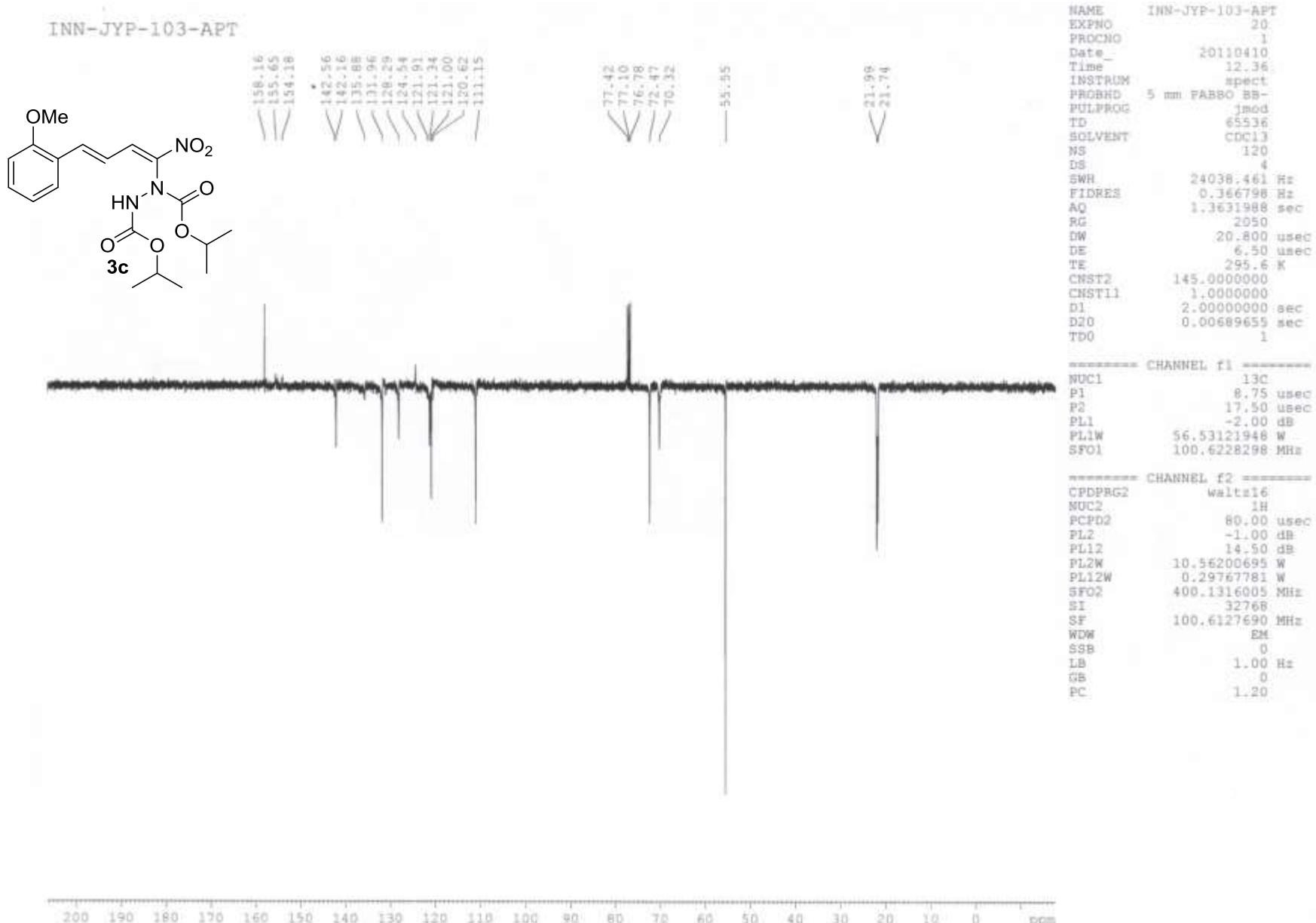


Figure S7.  $^{13}\text{C}$ -APT Spectrum of **3c**

INN-CD-23

Data Collected on: quanta-mercury300  
Archive directory: /export/home/vmrr1/vnmrsys/data  
Sample directory: INN-CD-23\_2008-04-11  
File: gCOSY\_01

Pulse Sequence: gCOSY

Solvent: CDCl<sub>3</sub>

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3273.3 Hz  
2D Width 3273.3 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 299.9475008 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.078 sec  
FT size 2048 x 2048  
Total time 12 min

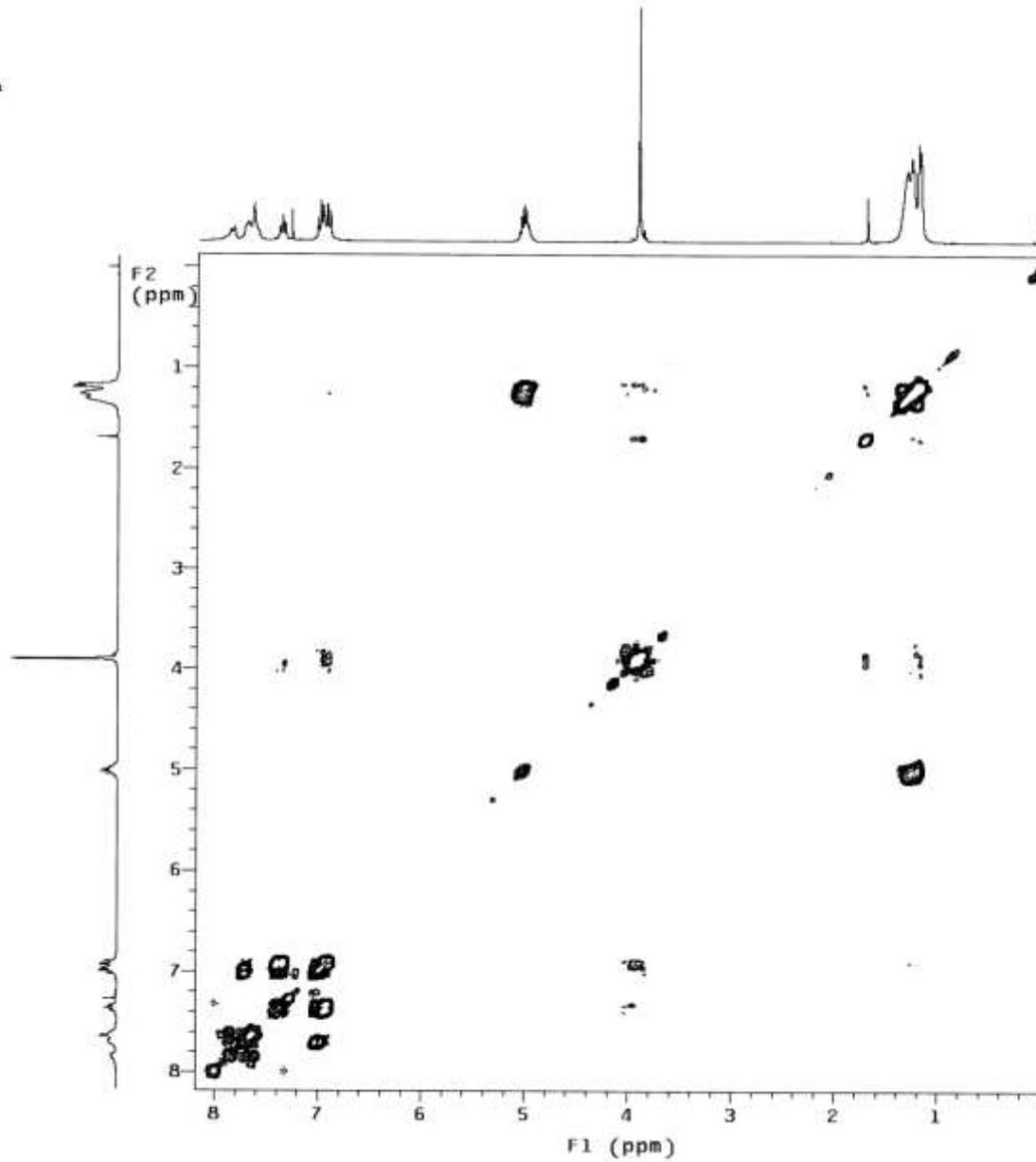
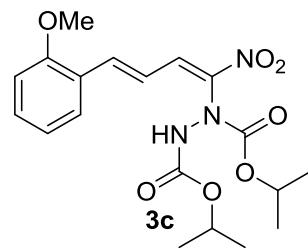


Figure S8. <sup>1</sup>H-<sup>1</sup>H-COSY Spectrum of **3c**

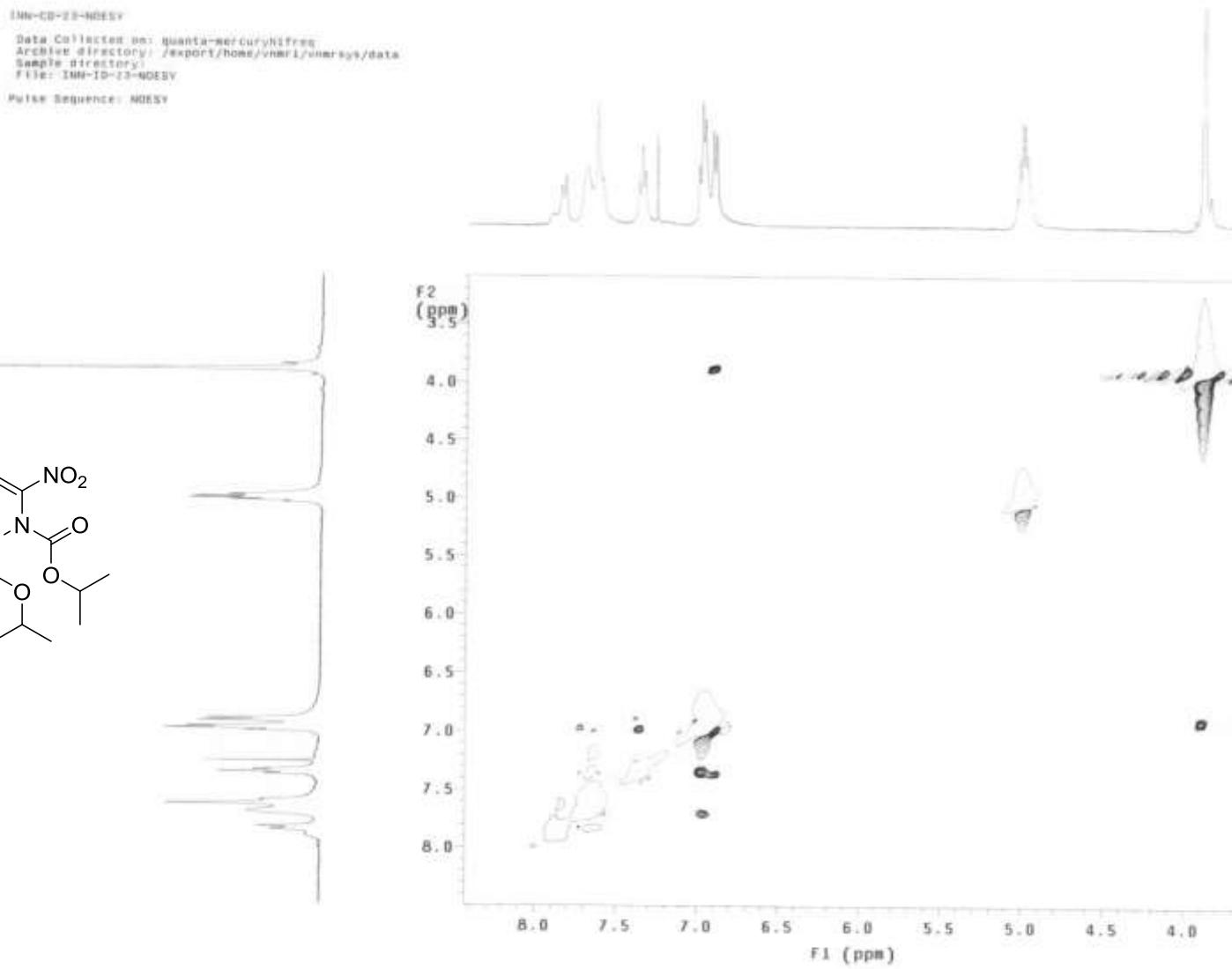


Figure S9.  $^1\text{H}$ - $^1\text{H}$ -NOESY Spectrum of **3c**

INN-CD-24-13C

exp4 s2pu1

SAMPLE SPECIAL  
date Jun 1 2008 temp not used  
solvent CD3OD gain not used  
file /export/home/- spin not used  
inn3/2008/June/1st- hist 0.008  
/INN-CD-24-13C.fid pw90 14.000  
ACQUISITION alfa 20,000  
sw 25125.6 FLAGS  
at 1.199 11 n  
np 60270 in n  
fb 13800 dp y  
bs 2 ns nn  
di 2.000 PROCESSING  
nt 64000 tb 2.00  
ct 18166 fn not used  
TRANSMITTER DISPLAY  
tn C13 sp -151.1  
sfreq 100.561 wp 20535.6  
tof 1558.7 rfp 8850.4  
tpwr 56 rfp 7742.3  
pw 7.000 rp 6.2  
DECOUPLER fp -329.0  
dn H1 PLOT  
dof 0 wc 250  
dm vvv sc 0  
dmm w vs 176  
dpwr 41 th 0  
dnf 11988 nm ph

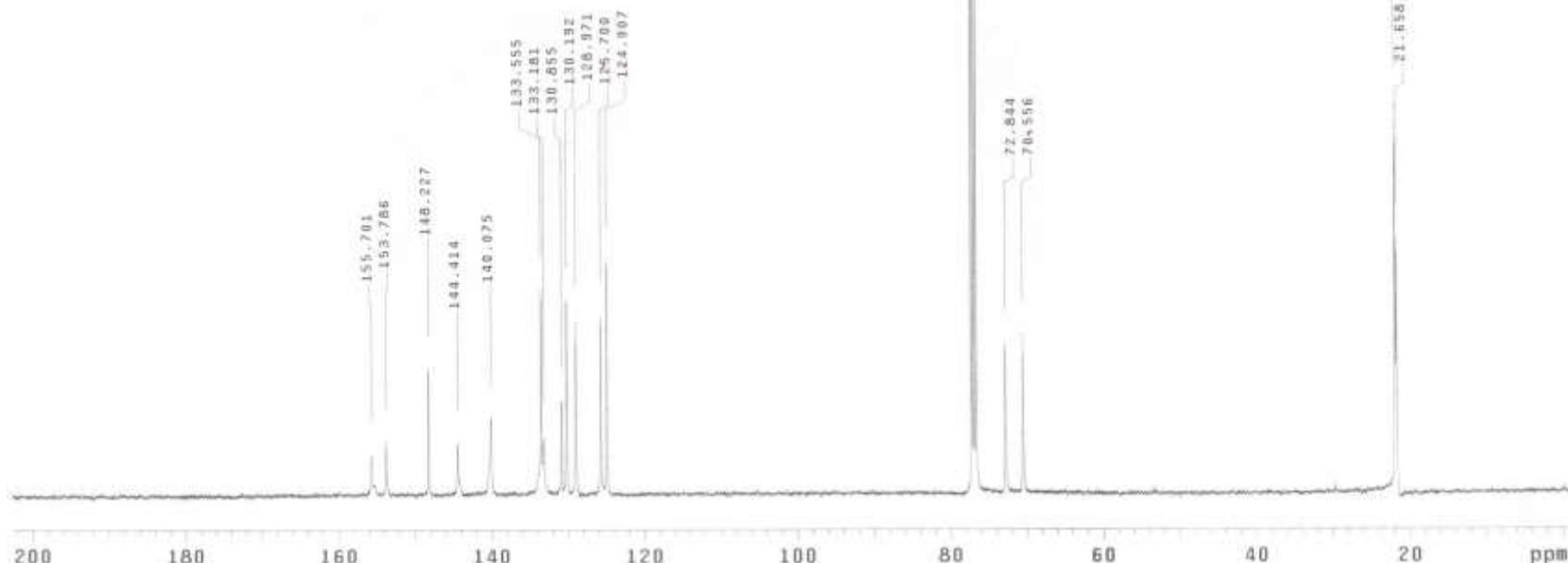
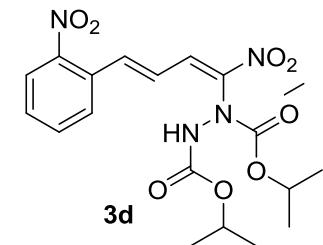


Figure S11.  $^{13}\text{C}$  NMR Spectrum of **3d**



Current Data Parameters  
 NAME INN-MVD-CYCLOHEXANEDIAD-H1  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters

Date 20150331  
 Time 0.38  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 50  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 30.72  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 293.9 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====

SFO1 500.1330885 MHz  
 NUC1 1H  
 P1 13.00 usec  
 PLW1 13.0000000 W

F2 - Processing parameters

SI 65536  
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 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

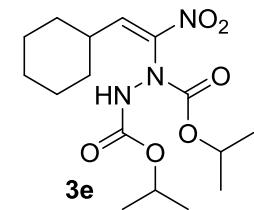
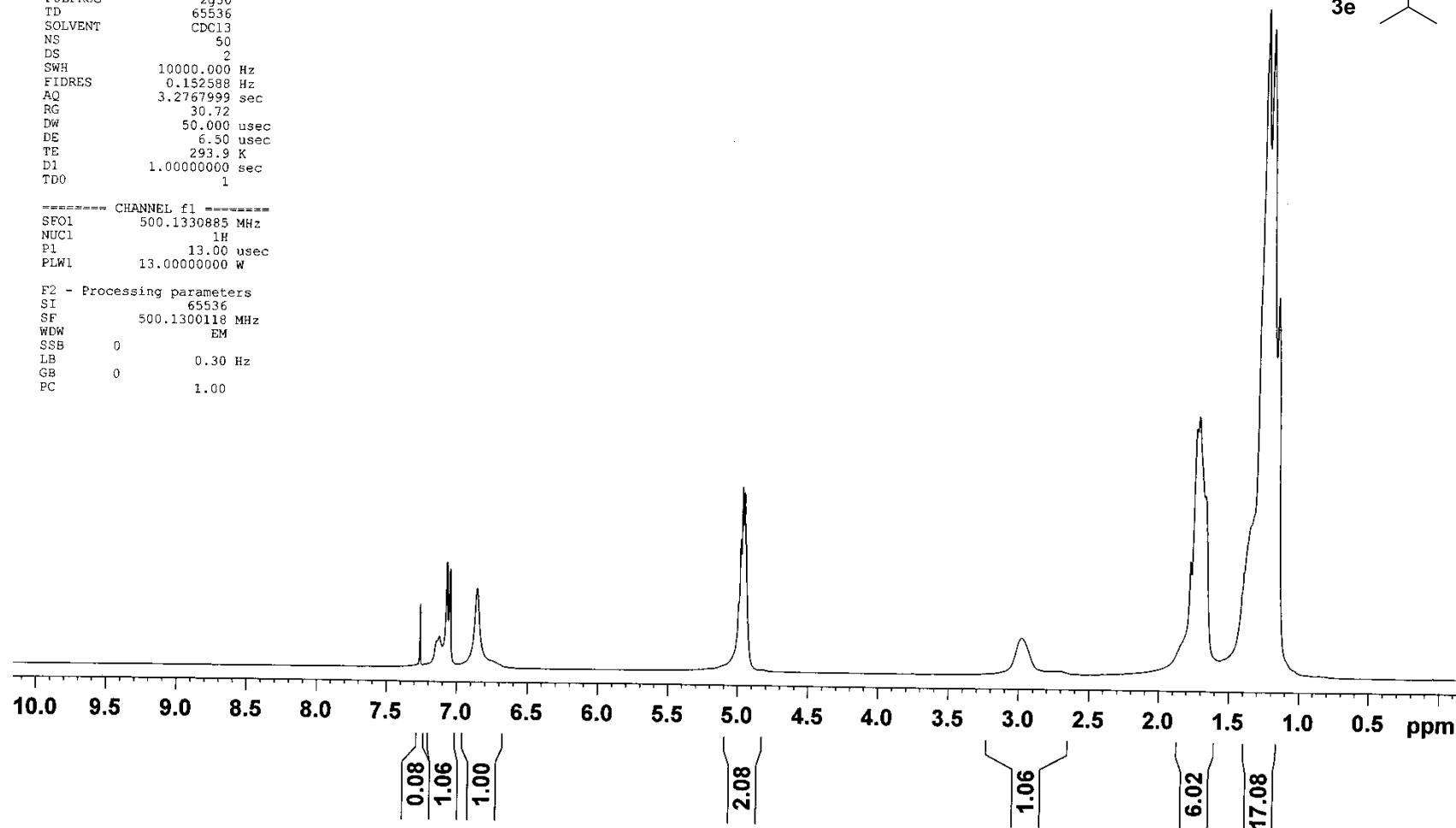


Figure S12. <sup>1</sup>H NMR Spectrum of 3e

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 PROCN0: 1  
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 Time: 13.27  
 INSTRUM: spect  
 PROBHD: 3 mm PABBO BB-  
 PULPROG: zgppg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 104  
 DS: 4  
 SWH: 26041.666 Hz  
 FIDRES: 0.397364 Hz  
 AQ: 1.2583412 sec  
 RG: 2050  
 DW: 19.200 usec  
 DE: 6.50 usec  
 TE: 295.5 K  
 D1: 1.0000000 sec  
 D11: 0.03000000 sec  
 TDO: 1

----- CHANNEL f1 -----  
 NUC1: 13C  
 P1: 8.75 usec  
 PL1: -2.00 dB  
 PL1W: 56.53121948 W  
 SFO1: 100.6238364 MHz

----- CHANNEL f2 -----  
 CPDPRG2: waltz16  
 NUC2: 1H  
 PCPD2: 80.00 usec  
 PL2: -1.00 dB  
 PLL2: 14.50 dB  
 PLL3: 14.50 dB  
 PL2W: 10.56200695 W  
 PL12W: 0.29767781 W  
 PL13W: 0.29767781 W  
 SFO2: 400.1316005 MHz  
 SI: 32768  
 SF: 100.6127564 MHz  
 WDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 PC: 1.40

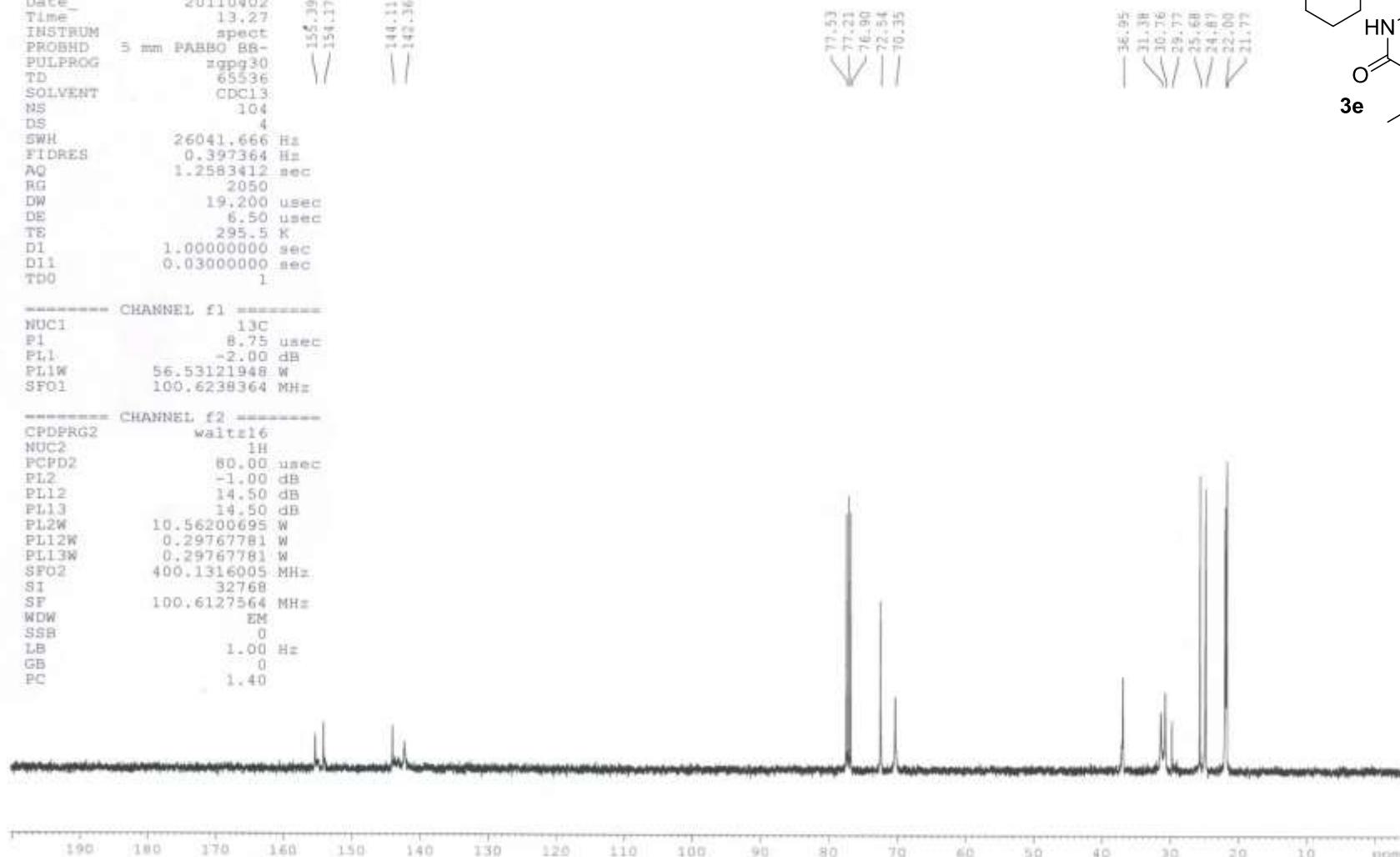


Figure S13.  $^{13}\text{C}$  NMR Spectrum of **3e**

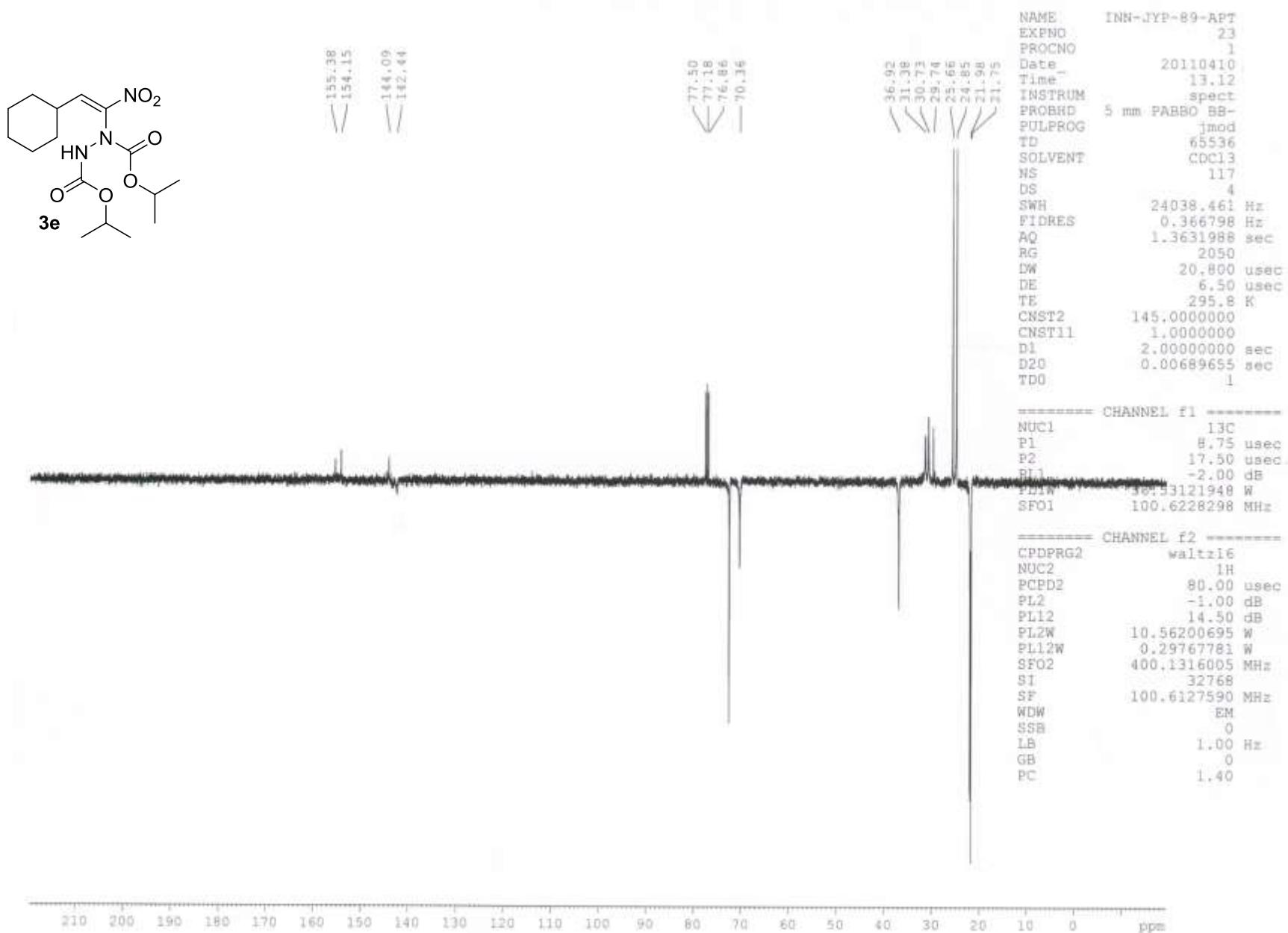


Figure S14.  $^{13}\text{C}$ -APT Spectrum of **3e**

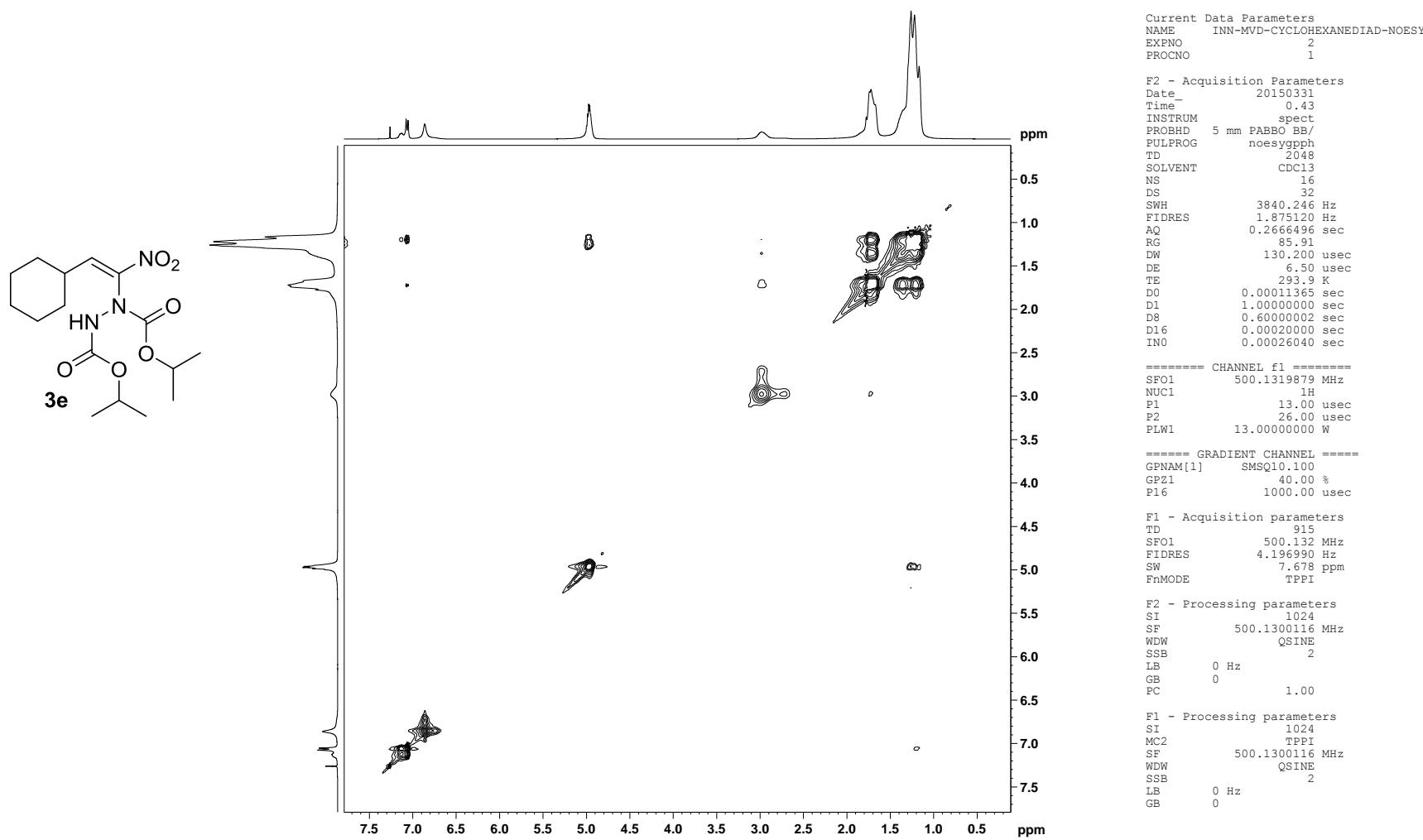


Figure S15.  $^1\text{H}$ - $^1\text{H}$  NOESY Spectrum of **3e**

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 PROCHO: 1  
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 Time: 21:25  
 INSTRUM: spect  
 FROBID: 5 mm PABBO BB-  
 PULPROG: zg30  
 TD: 5474  
 SOLVENT: CDCl3  
 NO: 23  
 OO: 0  
 SWH: 8223.685 Hz  
 FIDRES: 0.151522 Hz  
 AQ: 3.299931 sec  
 RG: 32  
 DW: 60.000 usec  
 DE: 6.50 usec  
 TE: 294.8 °K  
 R1: 3.000000 sec  
 TDO: 1

----- CHANNEL f1 -----  
 NUC1: 1H  
 F1: 14.75 usec  
 PL1: -1.00 dB  
 PL1W: 10.56200695 W  
 FPG1: 400.1324710 MHz  
 SI: 32768  
 SF: 400.1300096 MHz  
 MW0: EM  
 TSSB: 0  
 AB: 0.30 Hz  
 GB: 0  
 PC: 1.00

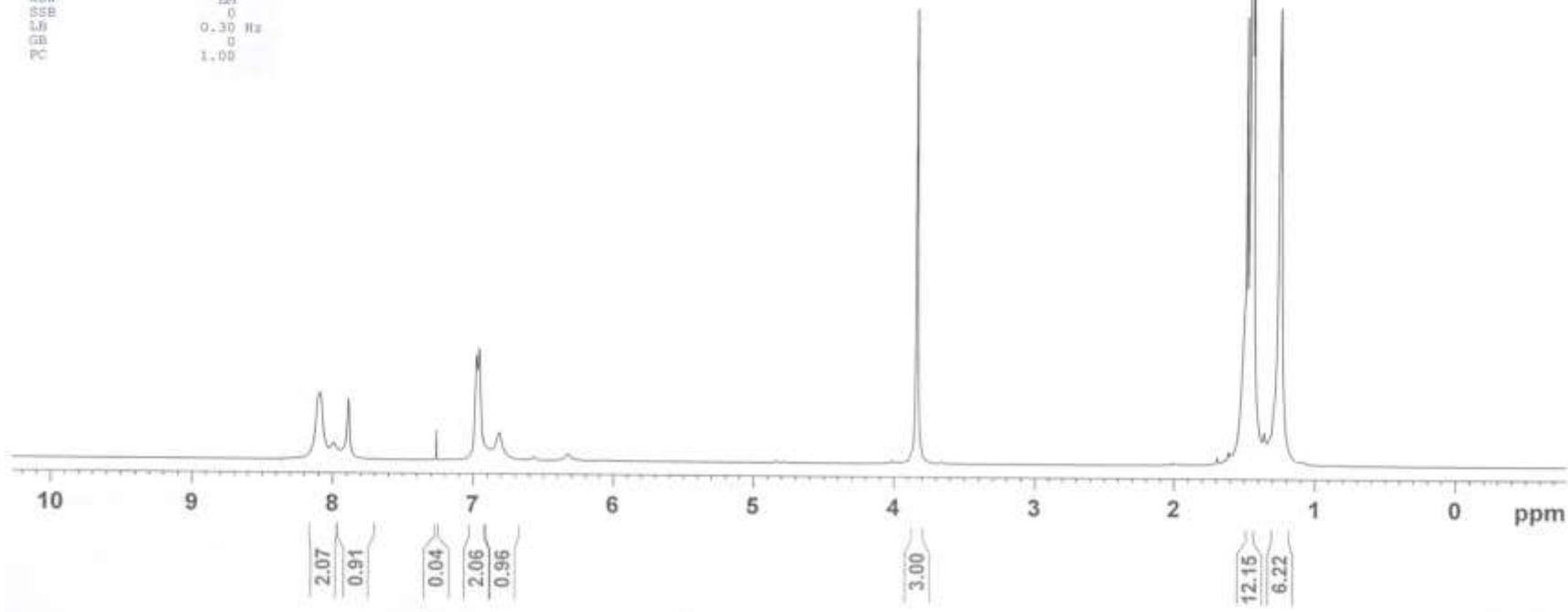
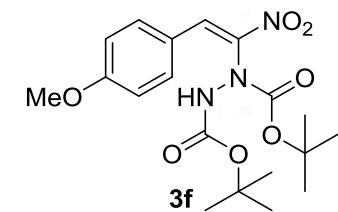
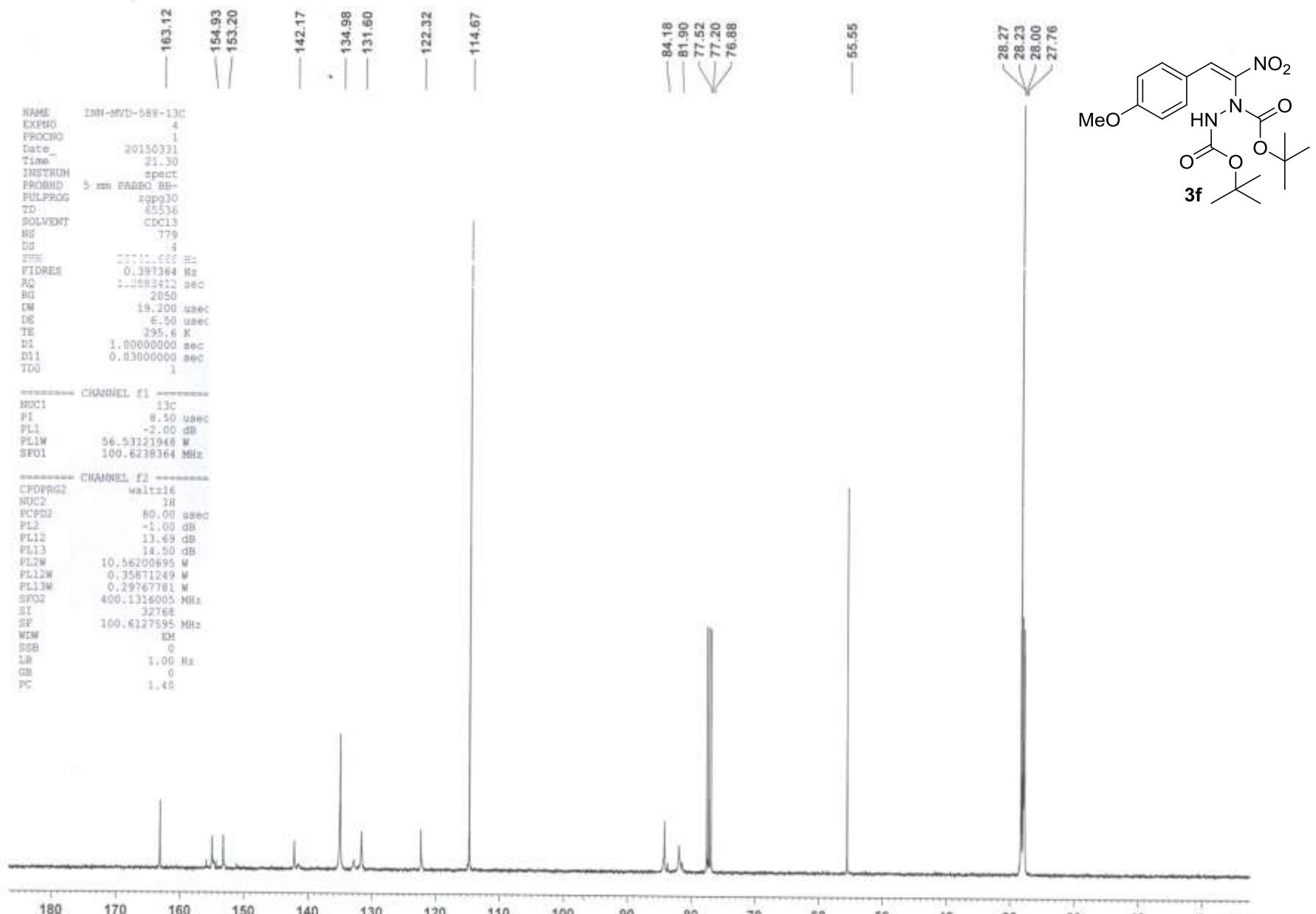


Figure S16.  $^1\text{H}$  NMR Spectrum of **3f**





```

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solvent    CDCl3 dof      nnn
file     exp dm      c
ACQUISITION  daw      200
srg     299.950 daf      50.0
tn      1H temp
at      2.000 PROCESSING
sp      24022 1b      0.10
sw      606.0  fn      not used
fb      not used
bs      2 warr
pw      3.0 wexp
pw      3.0 wbs
tpwrt  60 wnt
di      0 DISPLAY
t0f     900.0 sp      -30.9
nt      1600 wp      3841.1
ct      122 vs      163
clock   n sc      0
gain   2 wc      250
FLAGS   hznm      12.16
t1      n is      22041.89
in      n rfl      651.4
dp      y rfp      0
      y th      2
      ins      1.000
      nm ph

```

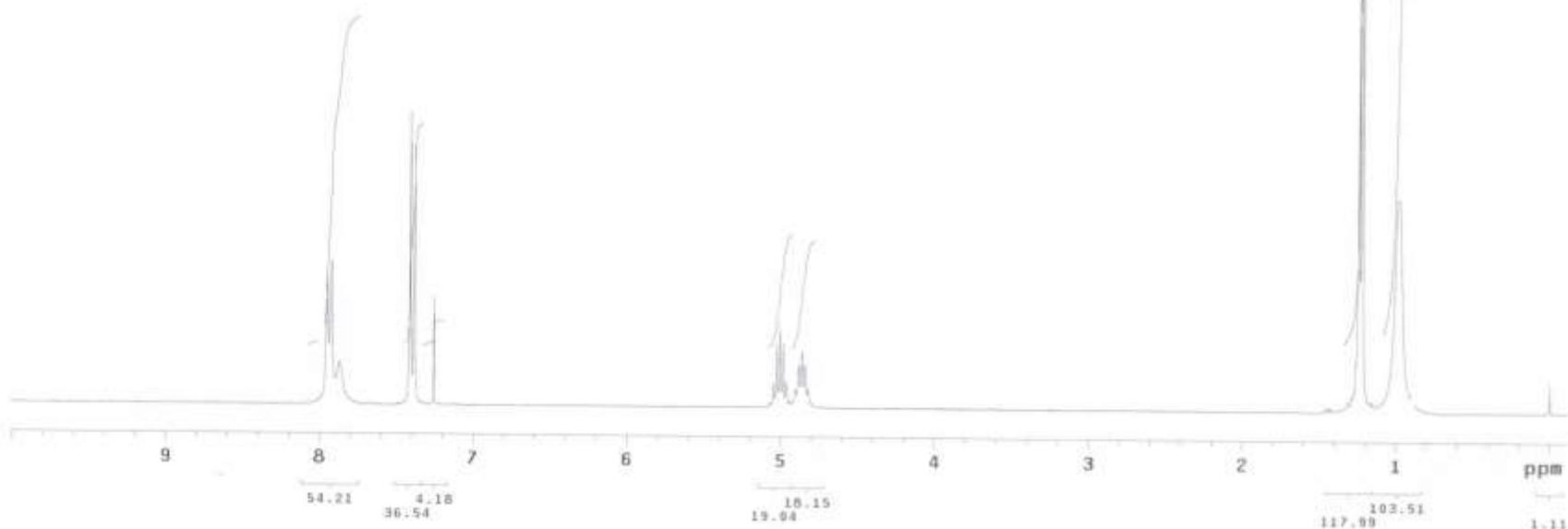
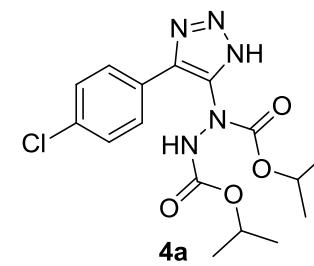


Figure S18.  $^1\text{H}$  NMR Spectrum of **4a**

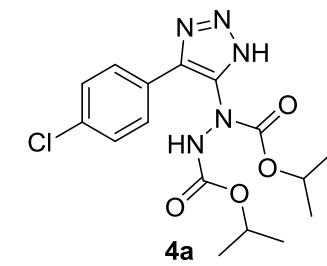
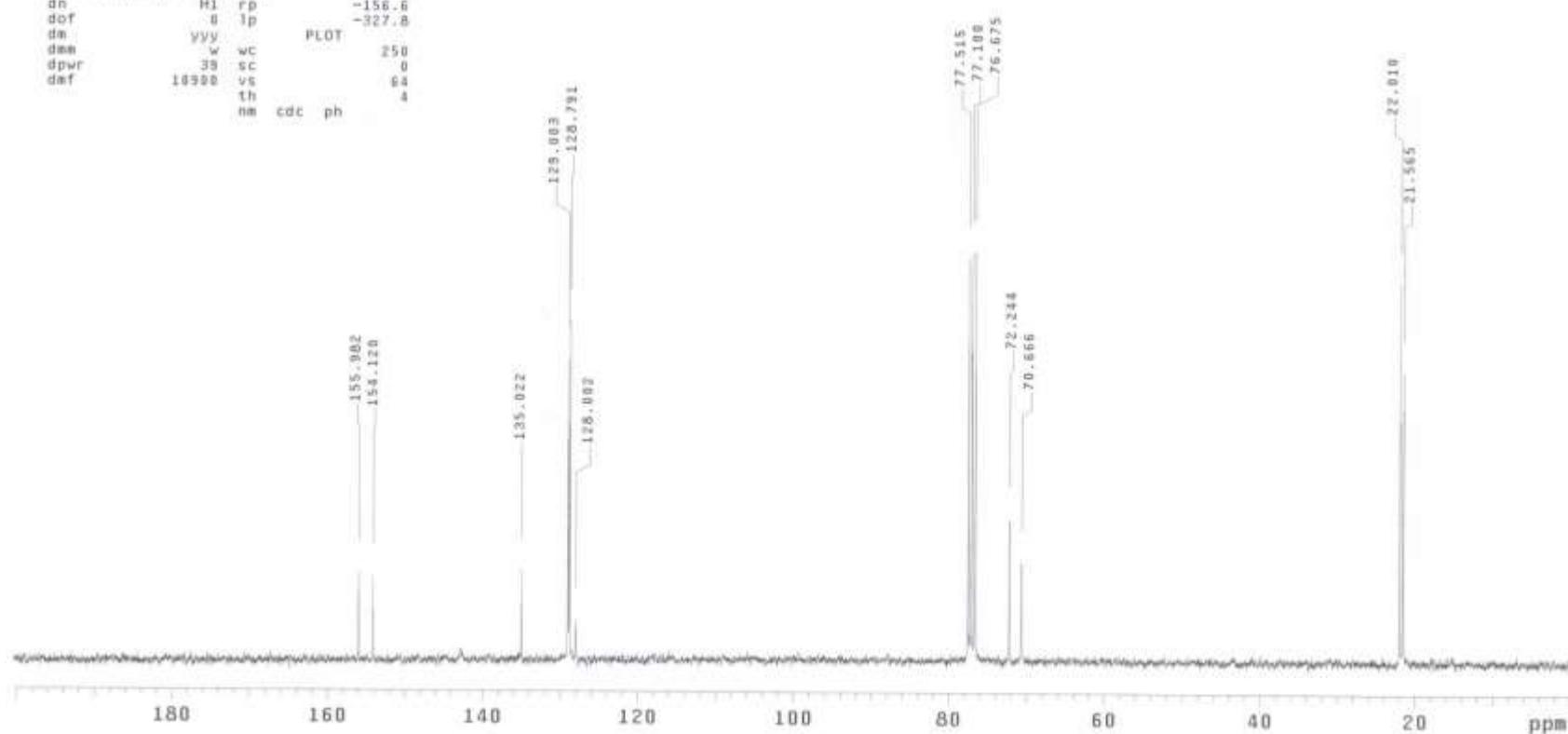
INN-RRK-702

exp3 s2pul

```

SAMPLE          SPECIAL      50.0
date   Feb 18 2014 temp      50.0
solvent    CDCl3  gain       2
file     exp  spin       20
ACQUISITION   hst  0.006
sw      25000.0 pw90    9.500
at      1.288 alfa   20.000
np      64000  FLAGS
fb      13880  f1      n
bs        4  fn      n
di      3.000  dp      y
nt      4800  hs      nn
ct      780  PROCESSING
TRANSMITTER   1b      2.00
tn      C13  fm      not used
sfreq    75.427  DISPLAY
t0f     -2000.0  sp      -36.0
tpwr      59  up      15148.2
pw      4.750 rfp    12773.1
DECOUPLER    H1  rfp    5815.0
dn      39  sc      0
dof      8  tp      -327.8
dm      vyy  PLOT
dmm      w  wc      250
dpwr    10000  vs      64
dmf      th      4
nm  cdc  ph

```

Figure S19.  $^{13}\text{C}$  NMR Spectrum of **4a**

INN-MVD-528

expt s2pu1  
SAMPLE DEC. 8 VT H1  
date May 27 2015 dn -260.4  
solvent CDCl<sub>3</sub> dof nnn  
file /export/home/~dm dmm t  
vnmr1/2015/May/int+ dmm 200  
ernal/INN-Mane/INN- dmt 55.0  
-MVD-528.fid temp 55.0  
ACQUISITION PROCESSING  
streq 299.950 1b 0.10  
tn H1 fm not used  
et 1.395  
sp 23988 wexp  
sw 6000.0 wexp  
fb not used wbs  
hs 2 wint  
pw 3.0 DISPLAY  
pw 3.0 sp -62.7  
tpwr 80 wp 3044.8  
el 0 vt 139  
tof 999.0 sc 0  
nt 32000 uc 250  
ct 638 hzmm 24.02  
atock n ls 13575.88  
gain 2 rfp 64.8  
FLAGS rfp  
tt n th 2  
in n ins 1.000  
dp y nm ph

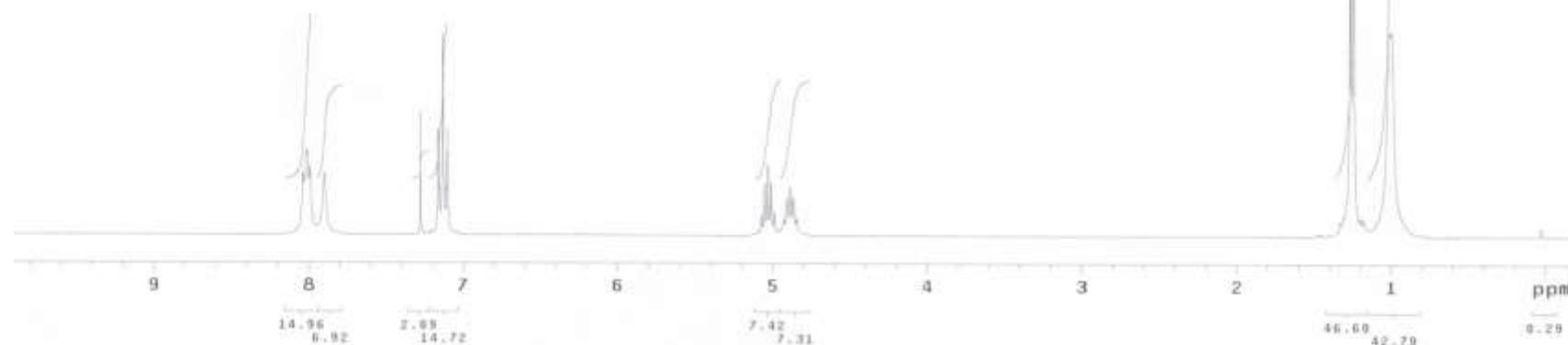
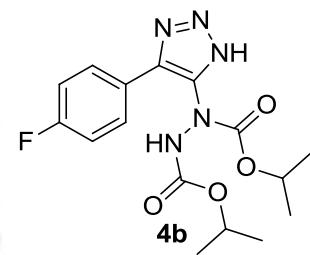


Figure S20. <sup>1</sup>H NMR Spectrum of 4b

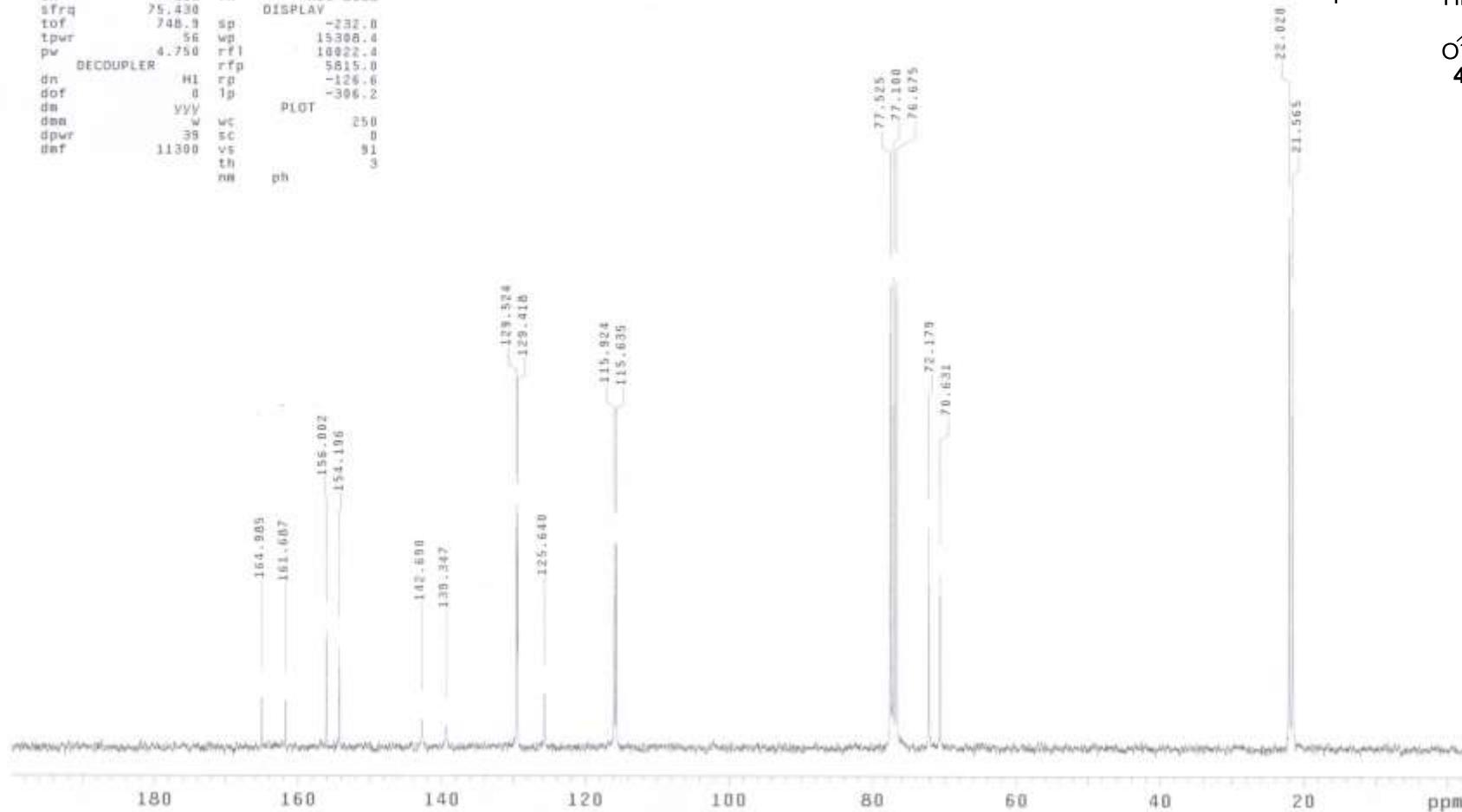
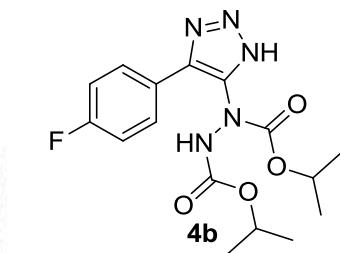
INN-MVD-529

exp3\_s2pul

```

SAMPLE          SPECIAL
date  May 27 2015 temp      55.0
solvent   CDCl3  gain       2
file     exp    spin    not used
ACQUISITION
sw      25000.0 pw90    11.500
at      1.815 alfa   20.000
np      90752   flags
fb      13000   i1      n
bs        4     in      n
di      3.000   dp      y
nt      8000    hs      nn
ct      884    PROCESSING
TRANSMITTER
th      C13   tb      3.00
sfrq    75.430 fn      not used
tof      748.9 sp      -232.0
tpwr    56    up      15398.4
pw      4.750 rfp     18022.4
DECOUPLER
dn      H1    rfp     5815.0
dof      0     lp      -126.6
dm      VVV   wct      250
dss      w    sc      0
dpwr    39    vs      91
dsf      11300 th      3
ns      ph

```

Figure S21.  $^{13}\text{C}$  NMR Spectrum of **4b**

NAME: 218-HVD-529-19F  
 EXPNO: 3  
 PROBHD: 1  
 Date: 20150428  
 TIME: 22:59  
 INSTRUM: spect  
 PROBHD: 5 mm PABBO BB-  
 PULPROG: zfpgn  
 TD: 131072  
 SOLVENT: CDCl3  
 NS: 41  
 DE: 4  
 SWH: 83285.713 Hz  
 FIDRES: 0.681196 Hz  
 AQ: 0.7340532 sec  
 RG: 724  
 DW: 5.600 usec  
 DE: 6.50 usec  
 TE: 296.2 K  
 D1: 1.0000000 sec  
 TDO: 3

===== CHANNEL F1 =====  
 MOC1: 19F  
 PI: 13.00 usec  
 PL1: -3.00 dB  
 PL1M: 17.04036532 W  
 BFO1: 376.4607164 MHz  
 DL: 65326  
 RF: 376.4993660 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.10 Hz  
 GB: 0  
 PC: 1.00

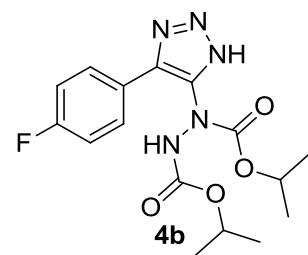


Figure S22.  $^{19}\text{F}$  NMR Spectrum of **4b**

ENH-RRK-701

exp2 sznul

SAMPLE	DEC.	R	VT
date Feb 18 2014	dn		H1
solvent CD3OD	dof	-218.4	
file exp	dm		nnn
ACQUISITION	dmr		a
sfreq 299.951	dif		
tn H1	temp	50.0	
at 2.000			PROCESSING
np 24022	lb	8.10	
sw 6666.0	fn		not used
fb			
bs 2	warr		
pw 3.0	wexp		
prw 3.0	wbs		
tpwr 60	wnt		
di 0			DISPLAY
tof 800.0	sp	-41.7	
nt 1600	wp	3622.2	
ct 318	vs	107	
clock n	sc	0	
gain 2	wc	250	
FLAGS	hzmn	14.49	
il n	rs	10053.40	
in n	rfl	1642.3	
dp y	rfp	992.0	
	th	2	
	tms	1.000	
nmr ph			

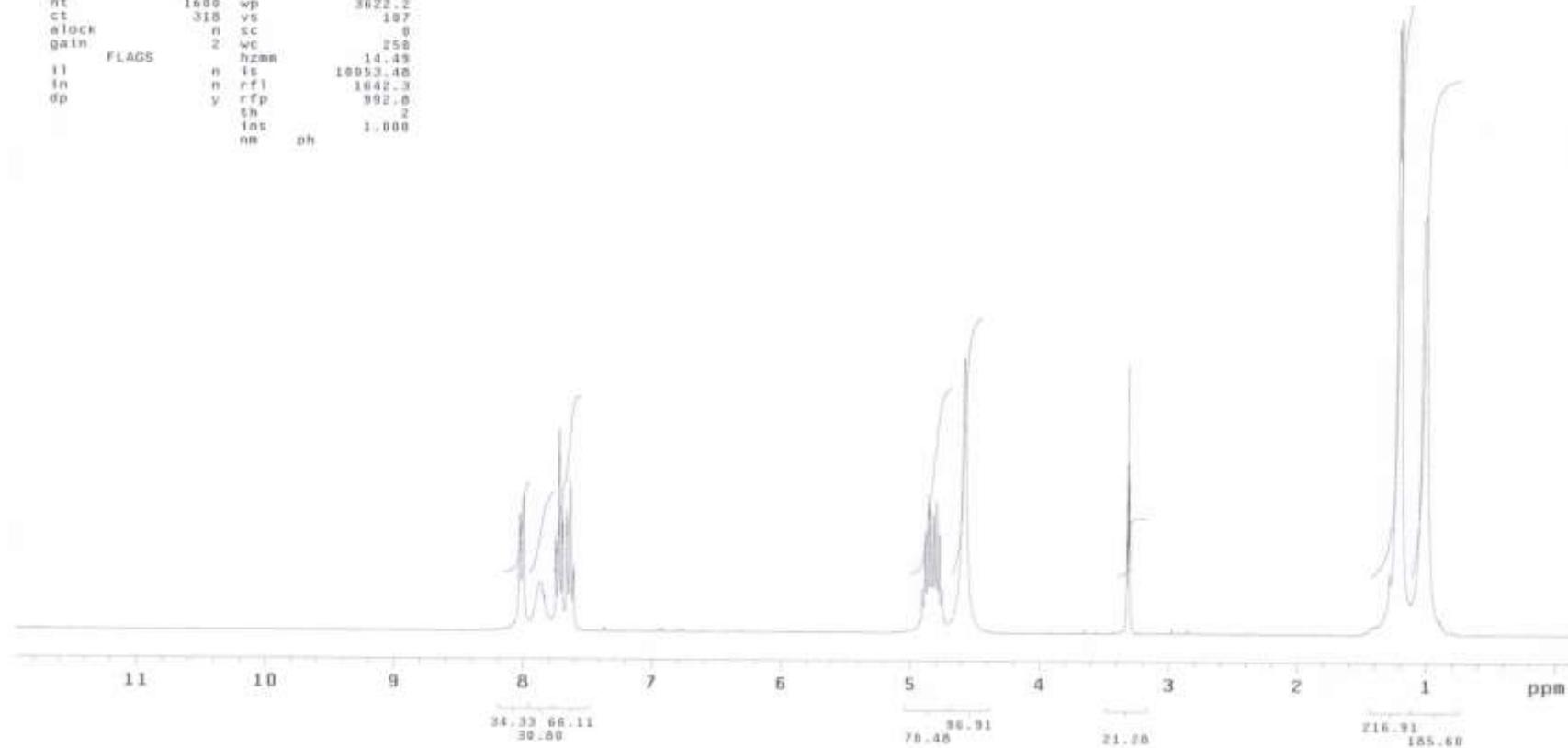
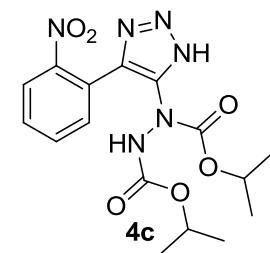


Figure S23.  $^1\text{H}$  NMR Spectrum of **4c**

INN-RRK-701

exp3 s2pul

SAMPLE SPECIAL  
date Feb 18 2014 temp 50.0  
solvent CD3OD gain 20  
file exp spin 20  
ACQUISITION hst 0.006  
sw 25000.0 pw90 9.500  
at 1.288 alfa 20.000  
np 64000 FLAGS  
fo 13800 t1 n  
bs 4 tn n  
dl 3.000 dp y  
nt 1600 hs mn  
ct 556 PROCESSING  
TRANSMITTER tb 3.00  
tn C13 fn not used  
sfrq 75.428 DISPLAY  
tof -2000.0 sp 48.0  
tpwr 59 wp 14964.3  
pw 4.750 rfi 10561.4  
DECOUPLER rfp 3763.2  
dn H1 rp -111.5  
dof 0 1p -335.2  
ds vvv PLOT  
dss w wc 250  
dpwr 39 sc 0  
def 10900 vs 170  
th 4  
nm cdc ph

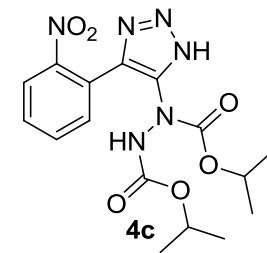
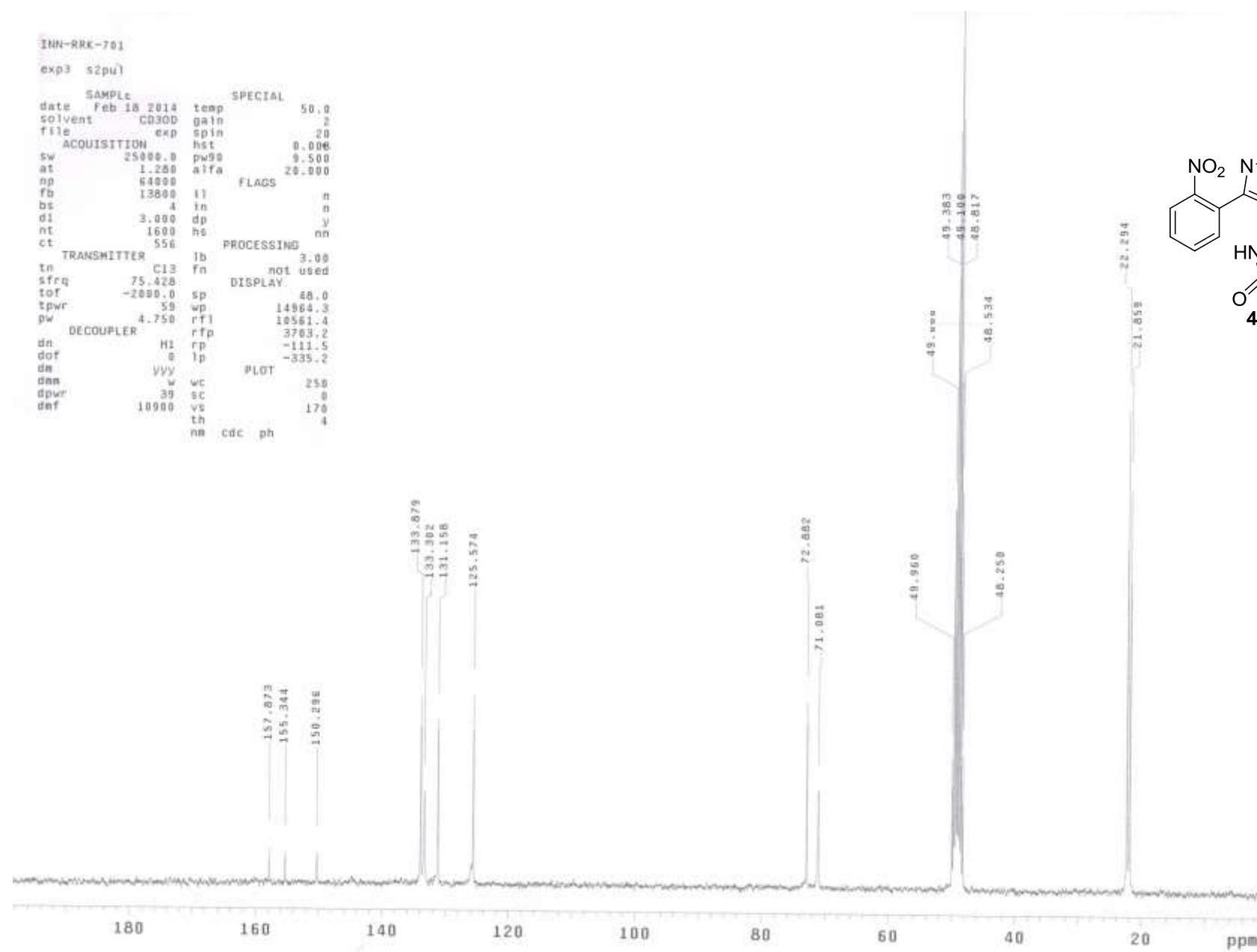


Figure S24.  $^{13}\text{C}$  NMR Spectrum of **4c**

Current Data Parameters  
NAME INN-RKC-G9B-1H@50°C  
EXPNO 18  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140124  
Time 16.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 33086  
SOLVENT CDCl3  
NS 50  
DS 0  
SWH 11029.412 Hz  
FIDRES 0.333356 Hz  
AQ 1.4998987 sec  
RG 30.72  
DW 45.333 usec  
DE 6.50 usec  
TE 323.0 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 500.1335009 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300111 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00

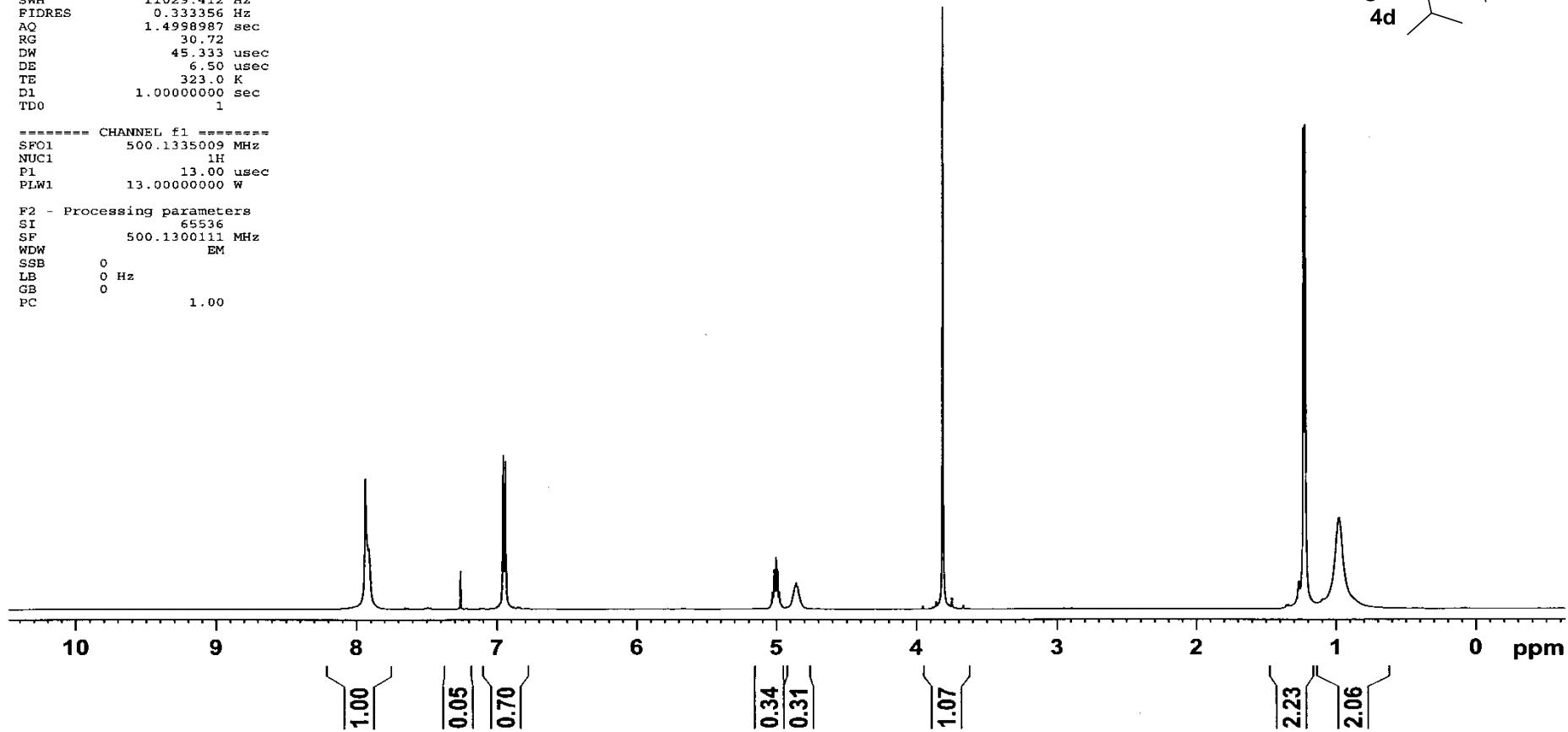
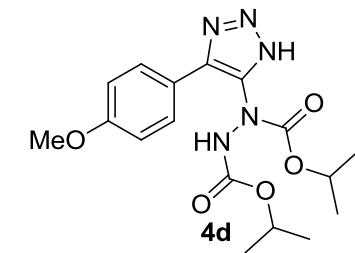


Figure S25.  $^1\text{H}$  NMR Spectrum of **4d**

Current Data Parameters  
NAME INN-RPK-69B-13C@50°C  
EXPNO 20  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140124  
Time 16.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1100  
DS 2  
SWH 34722.223 Hz  
FIDRES 0.529819 Hz  
AQ 0.9437184 sec  
RG 197.27  
DW 14.400 usec  
DE 6.50 usec  
TE 323.1 K  
DI 1.0000000 sec  
D11 0.03000000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 125.7721254 MHz  
NUC1 13C  
PI 8.90 usec  
PLW1 103.00000000 W

----- CHANNEL f2 -----  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 13.0000000 W  
PLW12 0.34327999 W  
PLW13 0.21969999 W

F2 - Processing parameters  
SI 32768  
SF 125.7577593 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

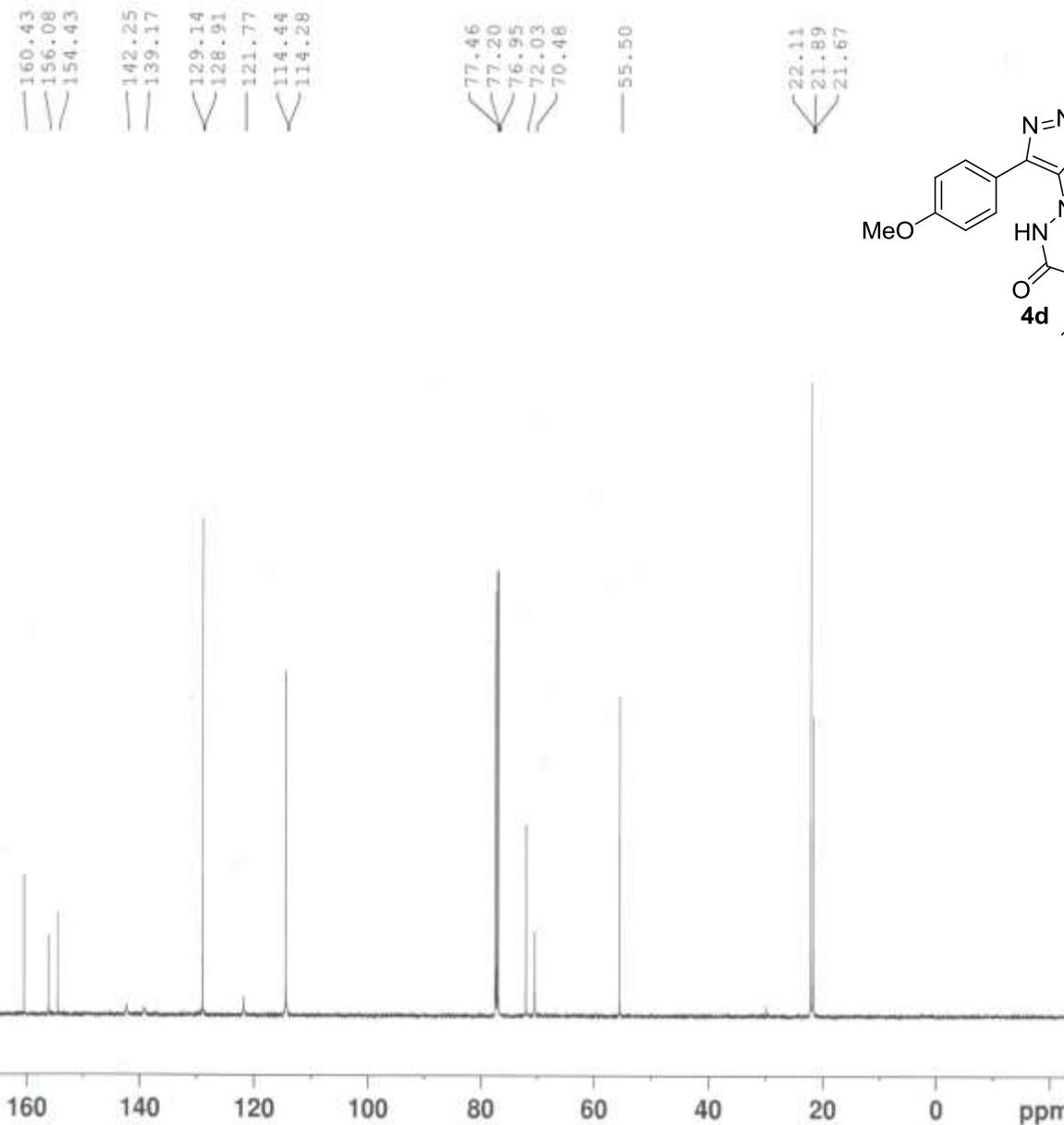


Figure 26.  $^{13}\text{C}$  NMR Spectrum of **4d**

INN-MVO-522 52<sup>3</sup>

exp1 sizepu1  
SAMPLE DEC. & VT  
date May 28 2015 dm H1  
solvent CDCl<sub>3</sub> d6 -268.4  
file /export/home/~dm nnn  
vnmr/2015/May/Int- dnm c  
ernal/INN-Mane/INN- daf 280  
-MVO-522.fid temp 55.0  
ACQUISITION PROCESSING  
sfreq 299.959 fm not used  
tn H1  
at 1.995 warr  
np 23968 wexp  
sw 6000.0 wbs  
fb not used wmt  
bs 2 DISPLAY  
pw 3.0 sp -62.7  
pw 3.0 wp 3844.8  
tpwr 60 vs 157  
di 0 sc 0  
t0f 900.0 wc 250  
nt 3200 hzbb 2.84  
ct 180 ts 6323.99  
alock n rrf 639.7  
gain 2 rfp 0  
FLAGS th 2  
i1 n im 1.000  
in n nm ph  
dp y

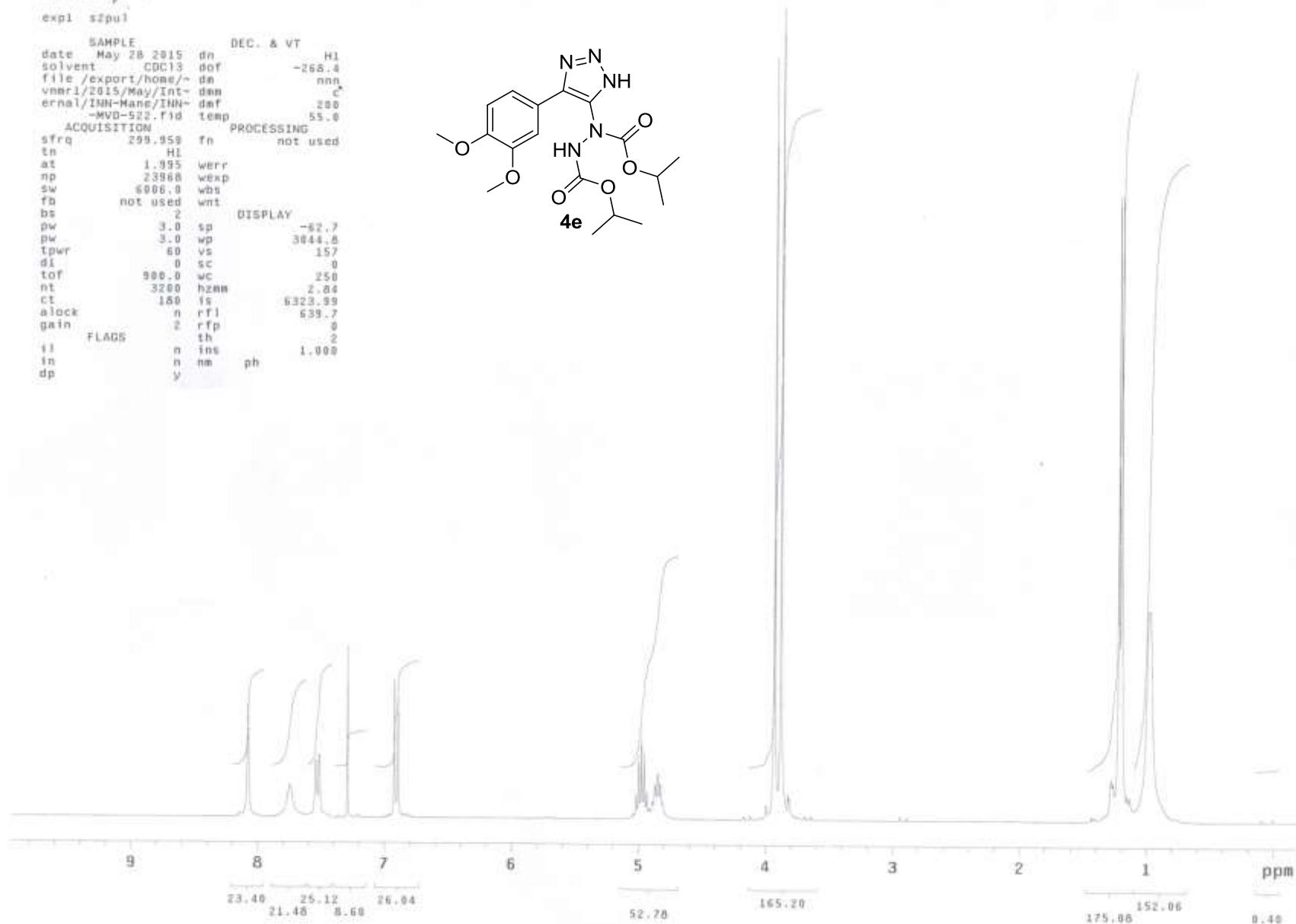
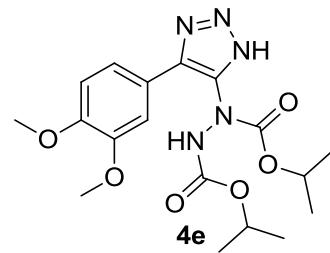


Figure S27. <sup>1</sup>H NMR Spectrum of 4e

INN-MVD-528 S28

exp3 zspout

SAMPLE SPECIAL  
date May 28 2015 temp 55.0  
solvent CDCl<sub>3</sub> gain 2  
file exp spin not used  
ACQUISITION nst 0.008  
sw 2500.0 Hz pw00 11.500  
at 1.815 alfa 20.000  
np 80752 t1 n  
rb 13800 t2 n  
bs 4 in n  
di 3.000 dp y  
nt 1600 hs mn  
ct 528 PROCESSING  
TRANSMITTER 10 3.00  
tn C13 tn not used  
sfreq 75.438 DISPLAY  
tof 748.9 tp -278.9  
tpwr 56 wp 155.38.4  
pw 2.750 rfp 100.38.4  
DECOUPLER rfp 581.5.0  
dn H1 rp -120.6  
dof 0 1p -388.2  
dm VVY PLOT  
dme w wc 250  
dpwr 39 sc 0  
def 11300 vs 99  
nq th 3  
nq ph

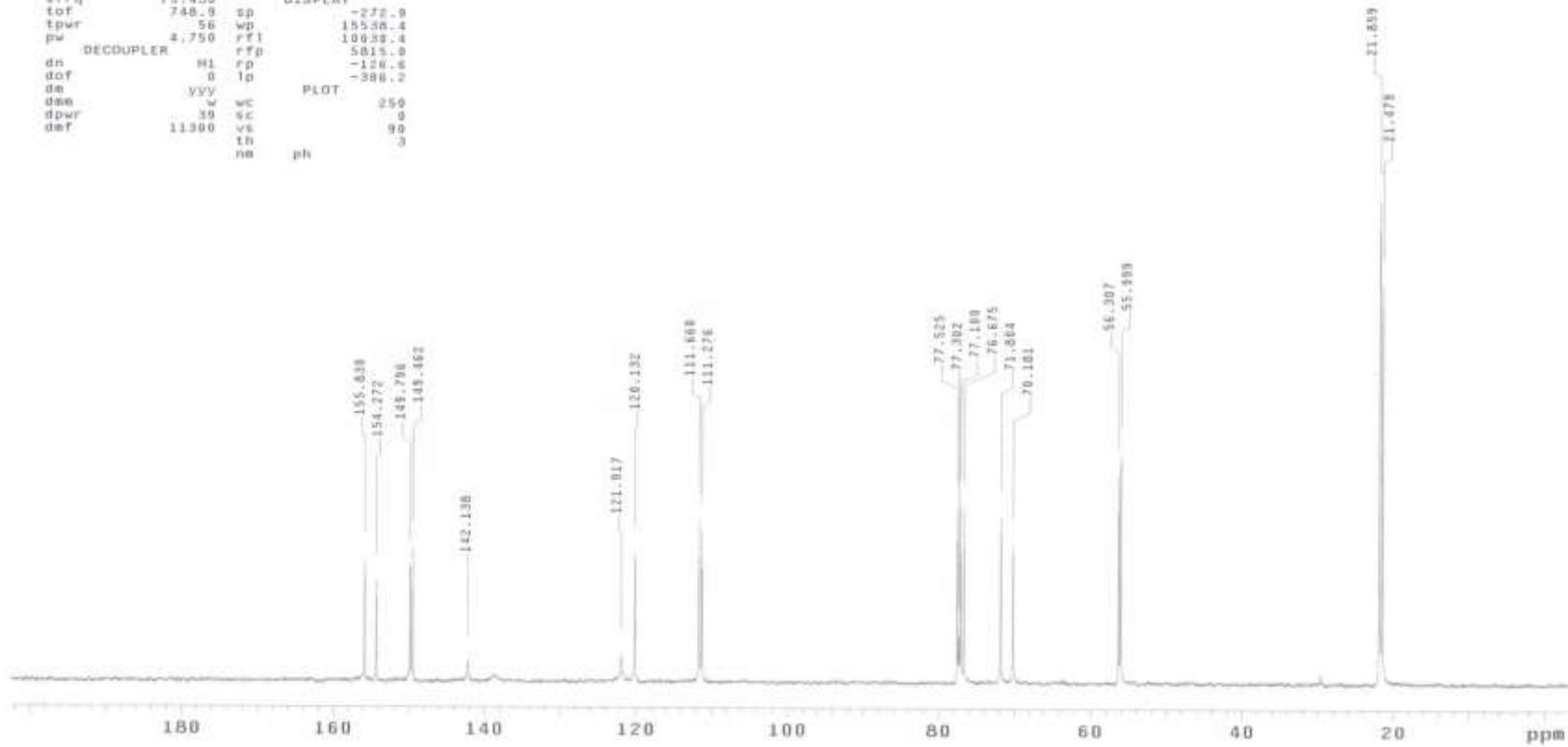
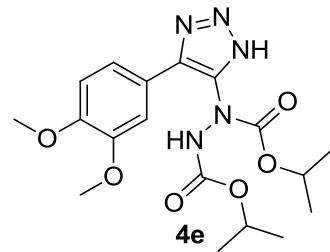


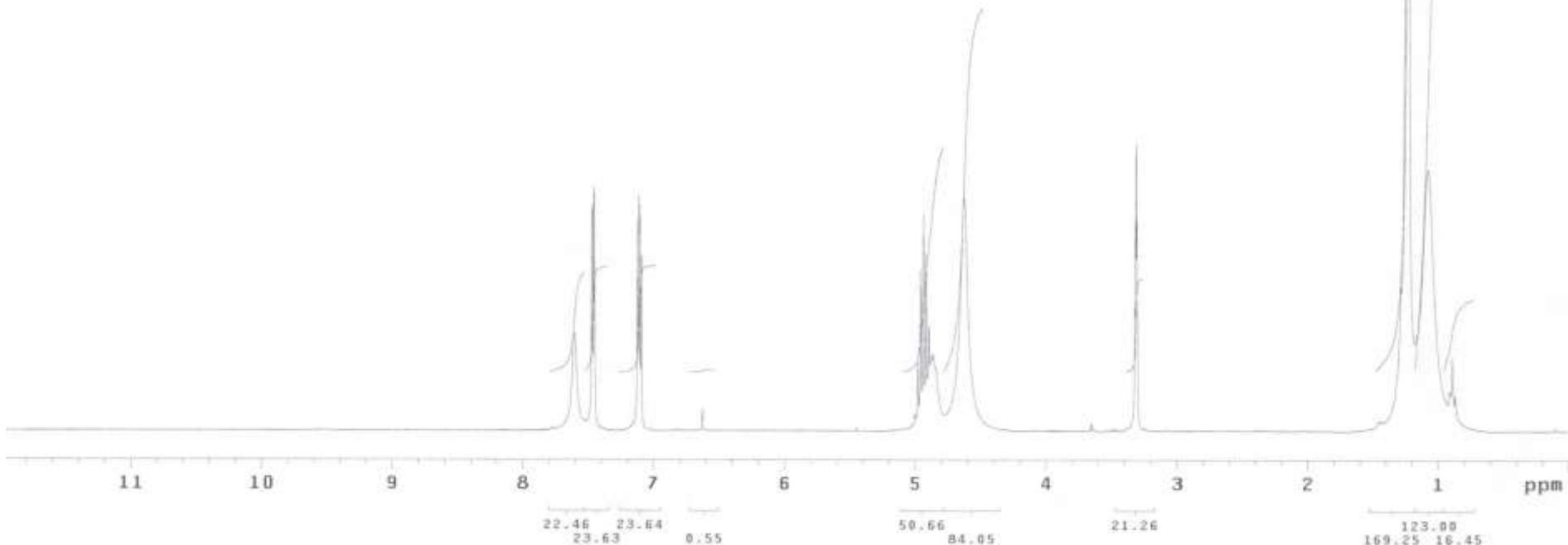
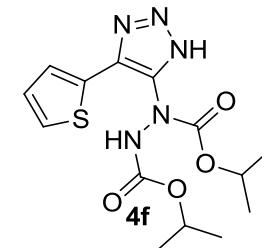
Figure S28. <sup>13</sup>C NMR Spectrum of **4e**

exp2 s2pul

```

SAMPLE           DEC. & VT
date   Feb 19 2014 dn      H1
solvent CD300 dof     -268.4
file    exp dm      nnn
ACQUISITION dmm      c
sfrq   299.951 dmf      200
tn      H1 temp     50.0
at      2.000 PROCESSING
np      24022 fn      not used
sw      6006.0
fb      not used werr
bs      2 wexp
pw      3.0 wbs
pw      3.0 wnt
tpwr   60
di      0 sp      -1.7
t0f    900.0 wp      3605.7
nt      800 vs      155
ct      800 sc      0
alock   n wc      250
gain    2 hzma    14.42
FLAGS
il      n rfl    16301.77
in      n rfp    649.4
dp      y th      2
ins    nm ph      1.000
nm      ph

```

Figure S29.  $^1\text{H}$  NMR Spectrum of **4f**

INN-RRK-705

exp3 s2pul

SAMPLE SPECIAL  
date Feb 19 2014 temp 50.0  
solvent CD3OD gain 2  
file exp spin 20  
ACQUISITION hst 8.000<sup>a</sup>  
sw 25000.0 pw00 3.500  
at 1.280 alfa 20.000  
np 64000 PLADS  
fb 13800 11 n  
bs 4 1n n  
dl 3.000 dp v  
nt 1280 ht nn  
ct 548  
TRANSMITTER 1b 3.00  
tn C13 fn not used  
rfreq 75.428 DISPLAY  
tof -2000.0 sp -58.1  
tpwr 53 wp 15200.2  
pw 4.750 rfi 6858.1  
DECOUPLER rfp 0  
dn H1 rp -116.0  
sof 0 1p -324.7  
dm yyyy PLOT  
dmr w wc 250  
dpwr 39 sc 0  
dmf 10900 vs 334  
nm cdc ph 4

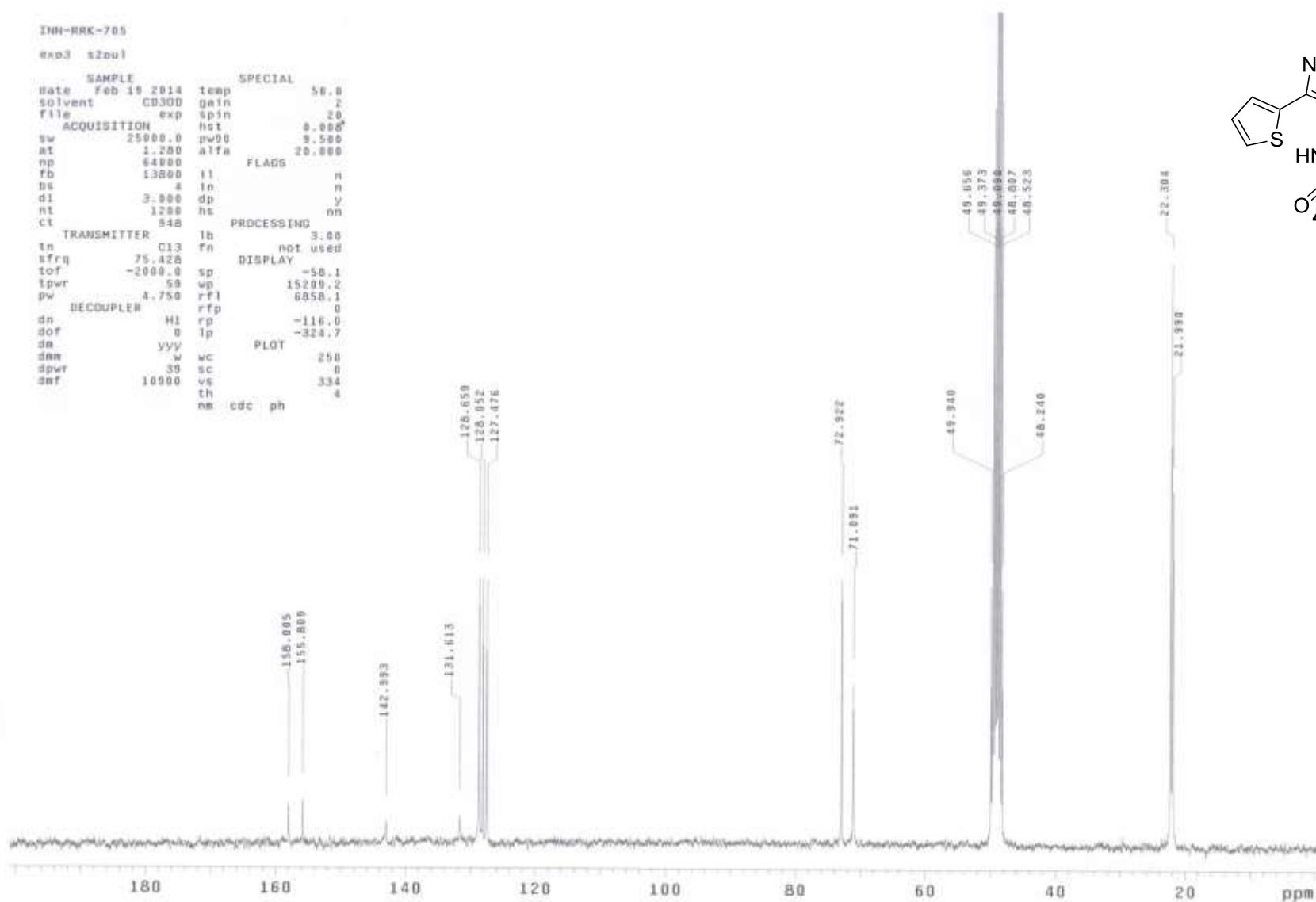
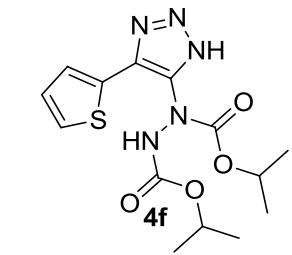


Figure S30. <sup>13</sup>C NMR Spectrum of **4f**



INN-MVD-528

exp1 s2pu1

SAMPLE DEC. & VT  
date May 27 2015 dn H1  
solvent CDCl<sub>3</sub> dof -268.4  
file /export/home/~ds nnn  
vmer1/2015/May/Int- dss c\*  
ernal/INN-Hane/528- dfr 200  
-5deg- fid temp 55.0  
ACQUISITION PROCESSING  
sfrq 299.958 ls 0.10  
in H1 fn not used  
at 1.095  
np 23968 wexp  
sw 6086.0 wexp  
tb not used ws  
bs z wnt  
pw 3.0 DISPLAY  
pw 3.0 sp -56.1  
tpwr 60 wpb 3973.0  
dl 0 vs 154  
t0f 900.0 vc 0  
nt 3200 wc 250  
ct 872 hzam 12.29  
alocs n fs 55259.60  
gain 2 rfi 644.8  
FLAGS rfp 0  
rl n th 2  
in n ins 1.000  
dp y nm ph

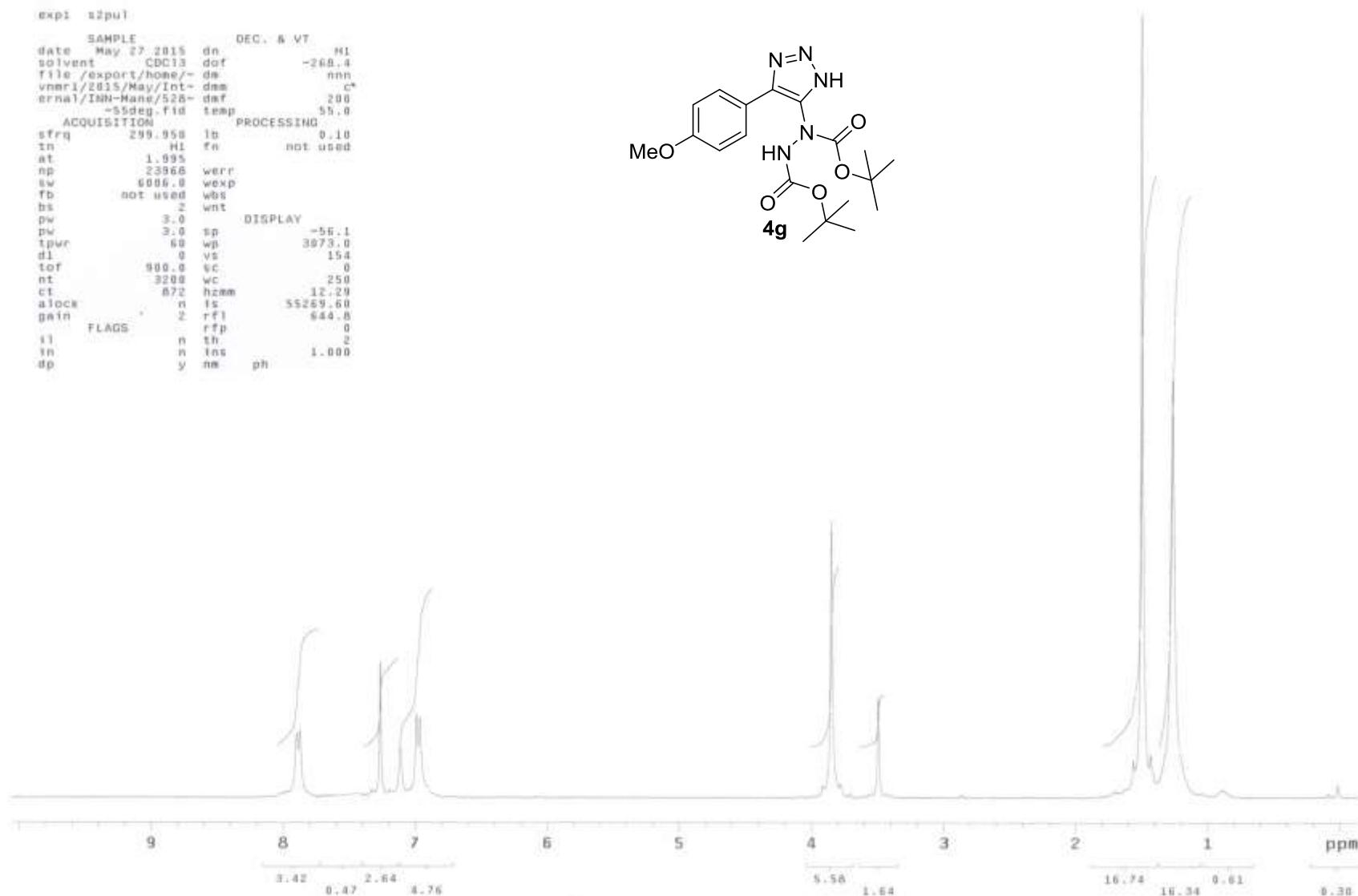
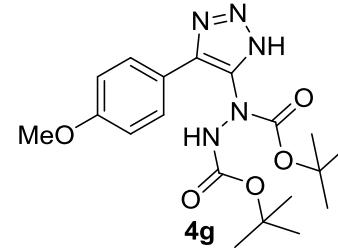
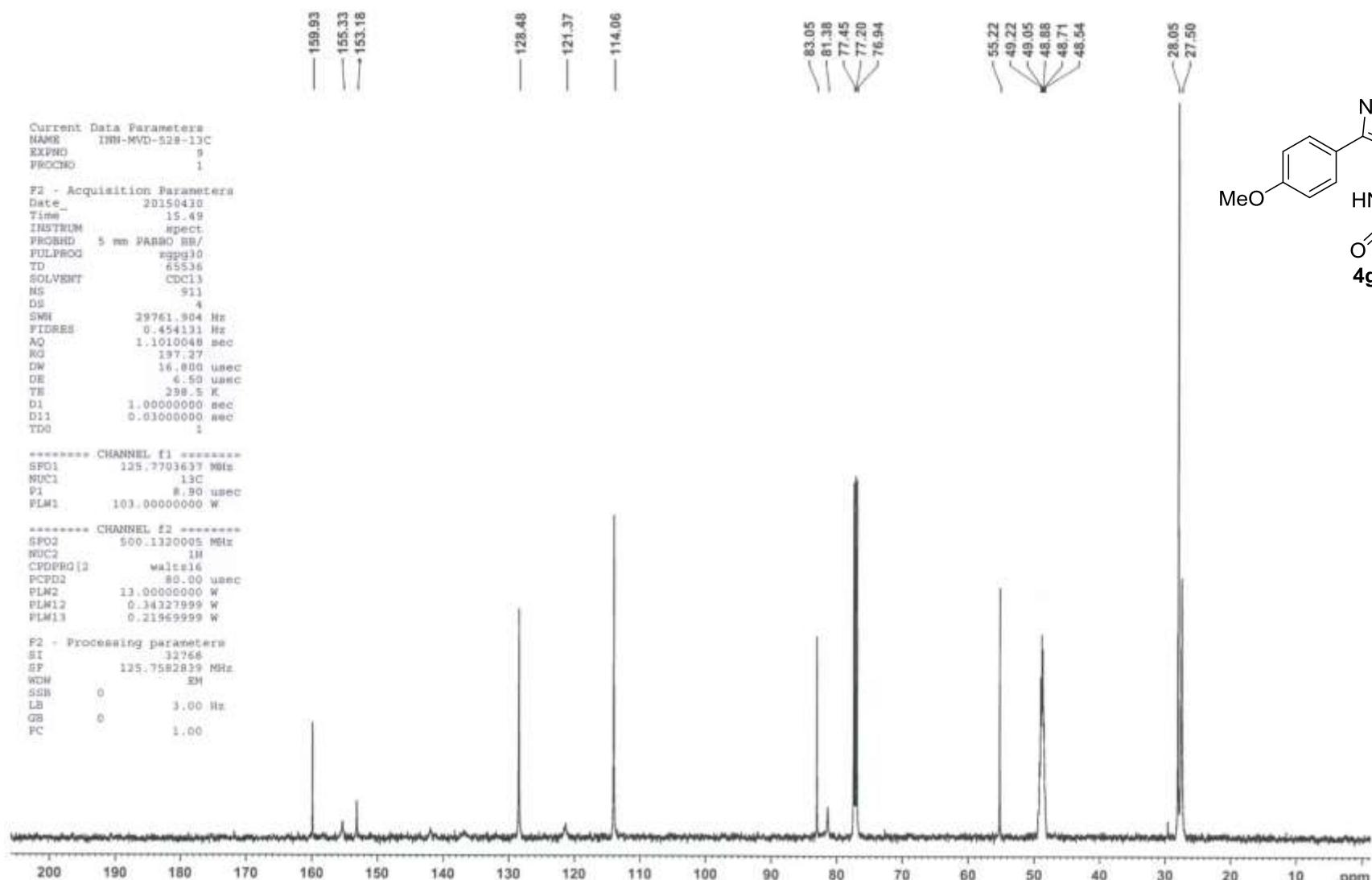


Figure S31. <sup>1</sup>H NMR Spectrum of 4g



Current Data Parameters  
NAME INN-MVD-166-1H  
EXPNO 1  
PROCNO 1  
P2 - Acquisition Parameters  
Date 20131220  
Time 18:03  
INSTRUM spect  
PROBID 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 12  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152586 Hz  
AQ 3.2767999 sec  
RG 48.36  
DW 50.000 usec  
DE 6.50 usec  
TE 295.9 K  
D1 1.0000000 sec  
TDD 1

----- CHANNEL f1 -----  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

P2 - Processing parameters  
SI 65536  
SF 500.1300116 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

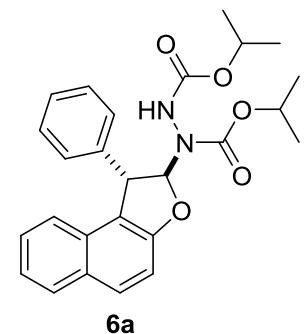
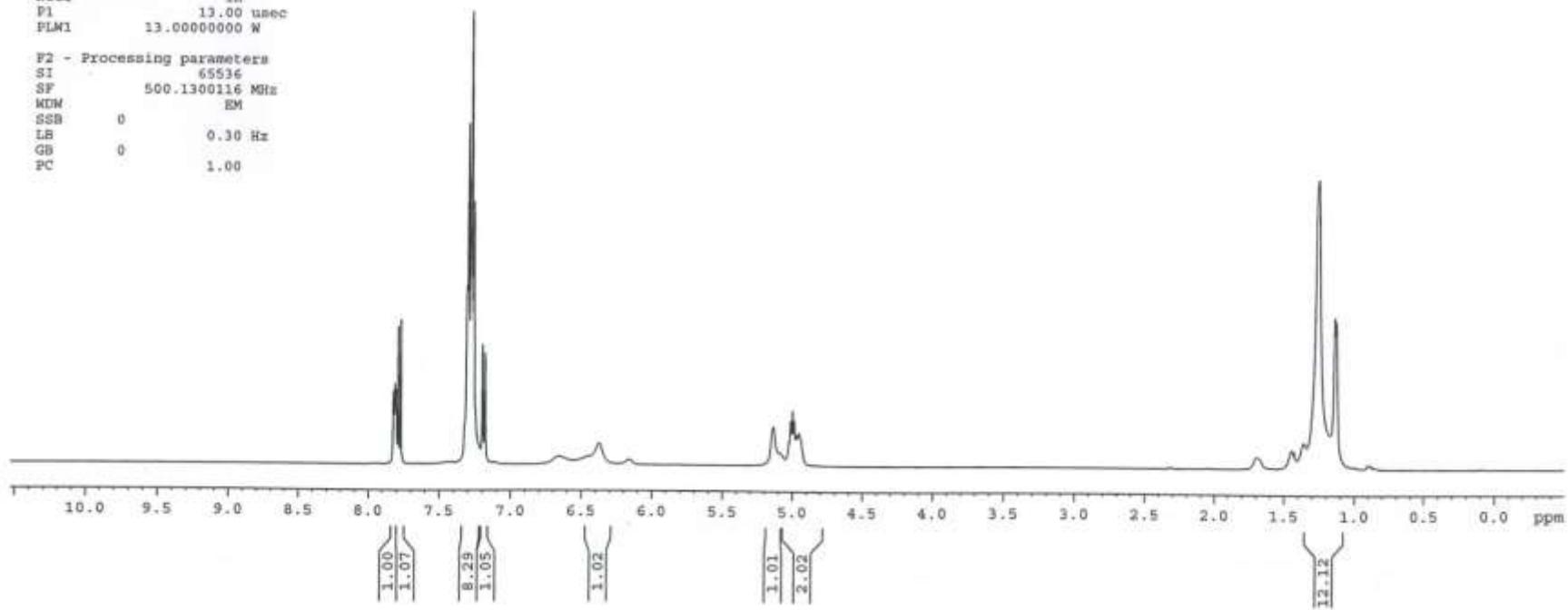


Figure S33.  $^1\text{H}$  NMR Spectrum of **6a**

## INN-MVD-166-13C

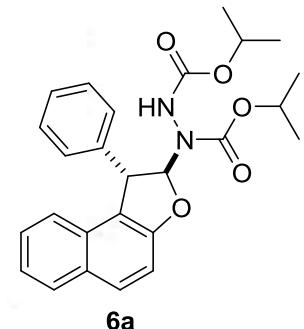
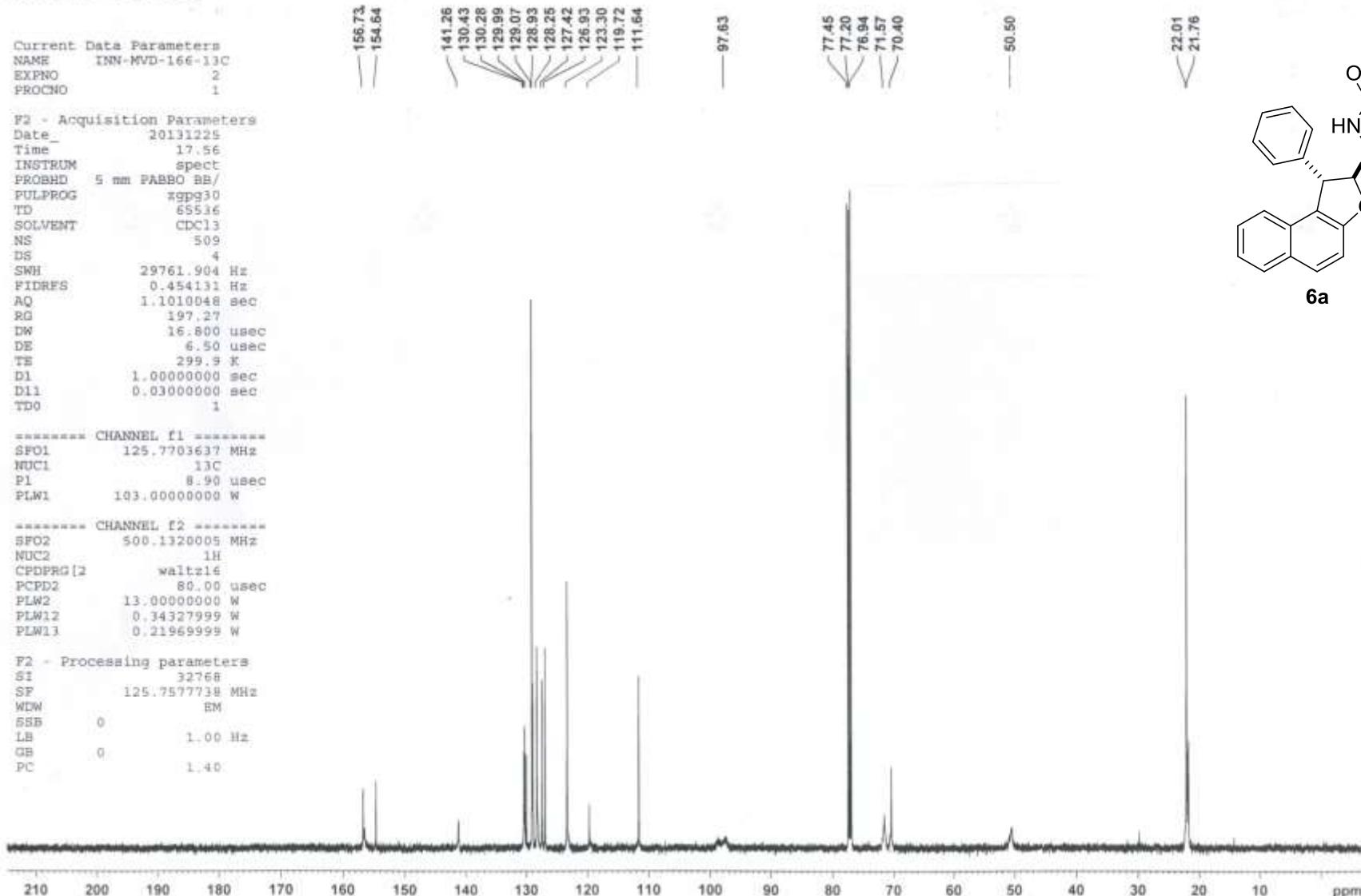
Current Data Parameters  
 NAME INN-MVD-166-13C  
 EXPNO 2  
 PROCHM 1

F2 - Acquisition Parameters  
 Date 20131225  
 Time 17.56  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 509  
 DS 4  
 SWH 29761.904 Hz  
 FIDRES 0.454131 Hz  
 AQ 1.1010048 sec  
 RG 197.27  
 DW 16.800 usec  
 DE 6.50 usec  
 TE 299.9 K  
 D1 1.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 SFO1 125.7703637 MHz  
 NUC1 13C  
 P1 8.90 usec  
 PLW1 103.0000000 W

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 SFO2 500.1320005 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 80.00 usec  
 PLW2 13.0000000 W  
 PLW12 0.34327999 W  
 PLW13 0.21969999 W

F2 - Processing parameters  
 SI 32768  
 SF 125.7577738 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure S34.  $^{13}\text{C}$  NMR Spectrum of **6a**

```

Current Data Parameters
NAME      INN-MVD-164-1H
EXPNO         9
PROCNO        1

P2 - Acquisition Parameters
Date       20131225
Time       21.30
INSTRUM   spect
PROBHD   5 mm PARBO BB/
PULPROG  zg30
TD        65536
SOLVENT    CDCl3
NS         11
DS          2
SWH     10000.000 Hz
FIDRES  0.152588 Hz
AQ     3.2767999 sec
RG        30.72
DW       50.000 usec
DE        6.50 usec
TE       296.9 K
DI     1.0000000 sec
TDO      1

***** CHANNEL f1 *****
SPO1    500.1330865 MHz
NUC1      1H
P1       13.00 usec
PLW1    13.0000000 W

P2 - Processing parameters
SI        65536
SP      500.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB      0
PC       1.00

```

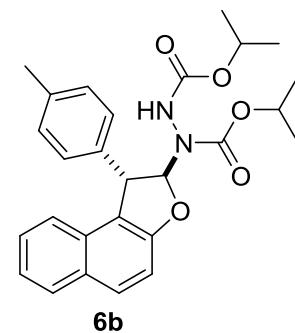
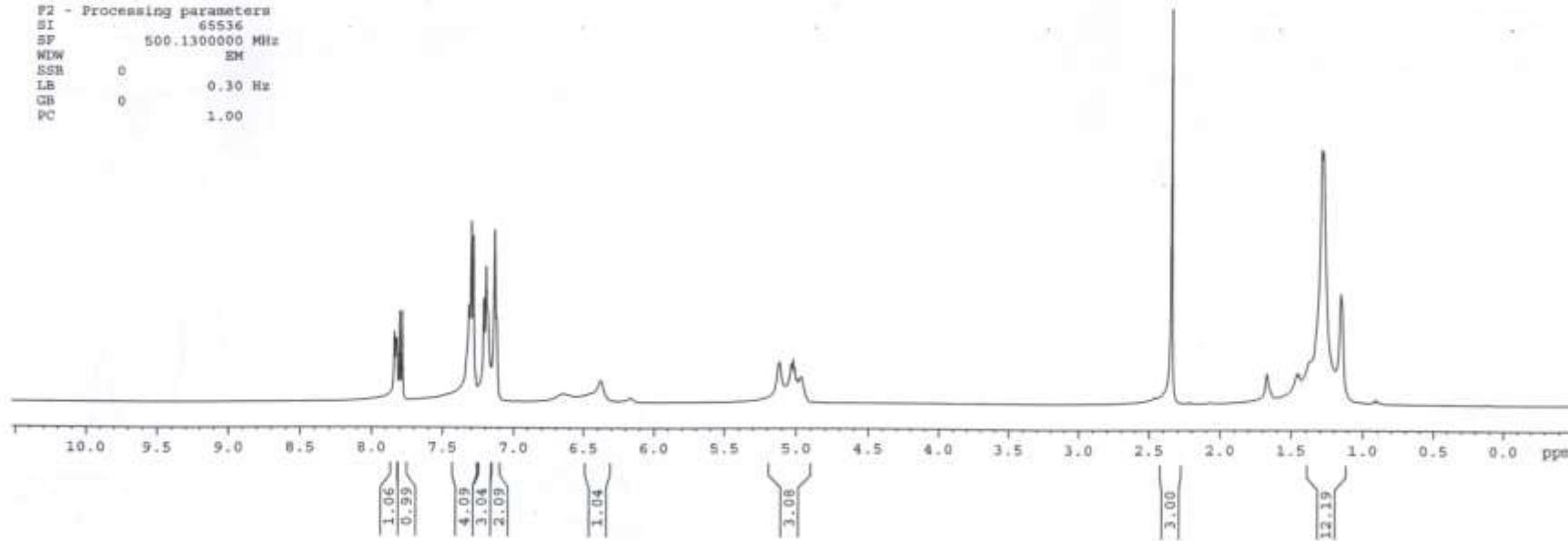
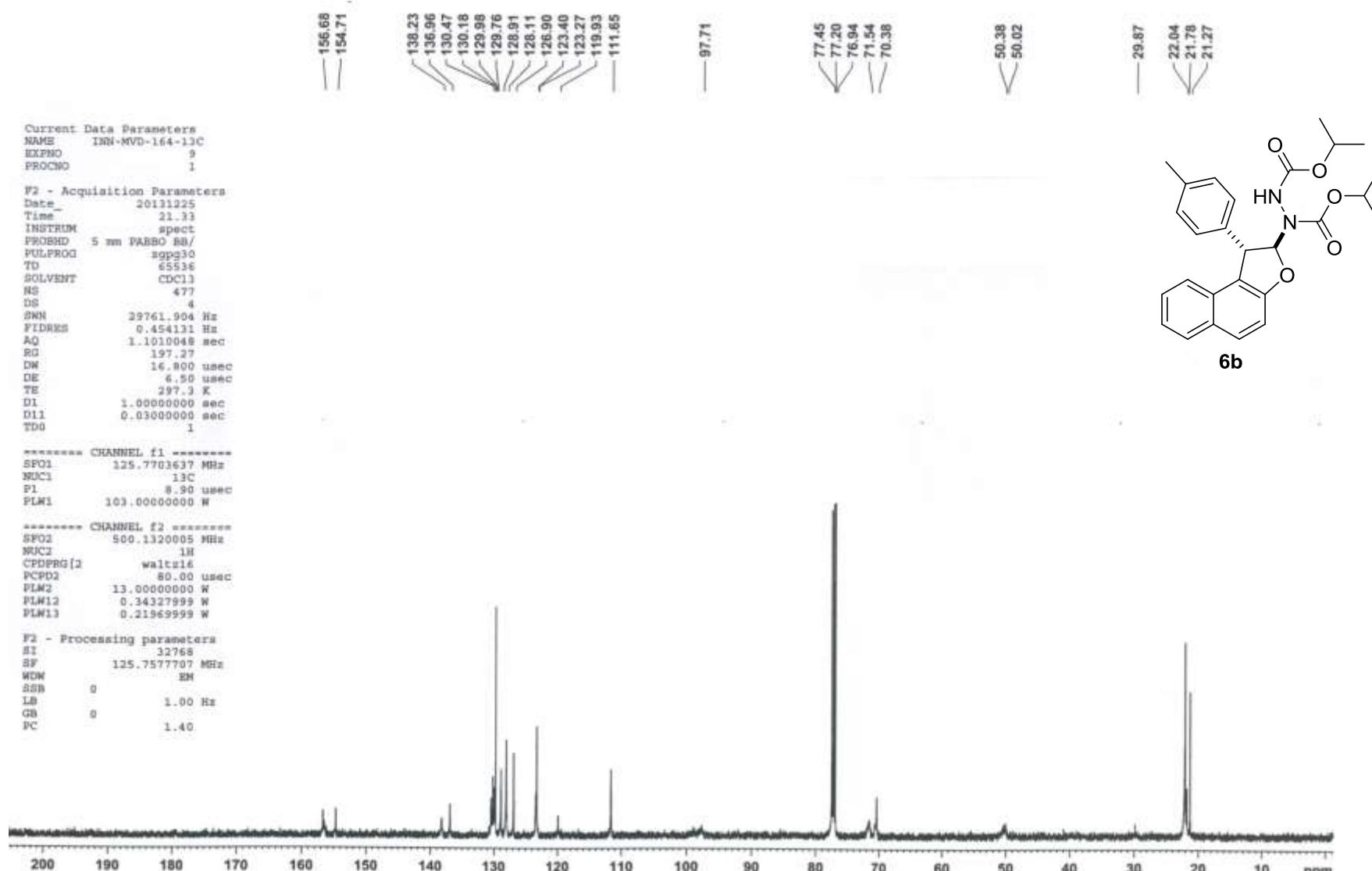


Figure S35.  $^1\text{H}$  NMR Spectrum of **6b**



Current Data Parameters  
 NNAME 1BB-MVD-151-1H  
 EXPNO 5  
 PROCNM 1  
 F2 - Acquisition Parameters  
 Date\_ 20140923  
 Time 15:21  
 INSTRUM spect  
 PROBDS 5 mm PABBO BB/  
 PULPROG zgppsp  
 TD 65536  
 SOLVENT CDCl3  
 MS 12  
 DS 4  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 10.65  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 297.1 K  
 D1 1.0000000 sec  
 D12 0.00002000 sec  
 D16 0.00020000 sec  
 TDO 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 SP01 500.1330885 MHz  
 NUC1 1H  
 P1 13.00 usec  
 P2 26.00 usec  
 P12 2000.00 usec  
 PLW0 0 W  
 PLW1 13.0000000 W  
 SPNAM[1] squar100.1000  
 SPNAM[2] 0.500  
 SPDPFFS1 -2144.93 Hz  
 SPW1 0.00219700 W  
 \*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GPNAME[1] SMMQ10.100  
 GPNAME[2] SMMQ10.100  
 GPR1 31.00 %  
 GPR2 11.00 %  
 P16 1000.00 usec

F2 - Processing parameters  
 SI 65536  
 SF 500.1300245 MHz  
 WDM RM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

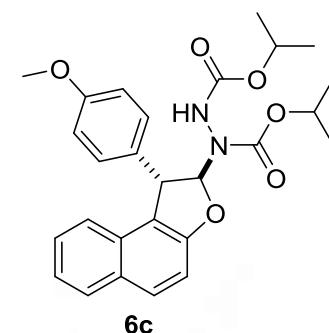
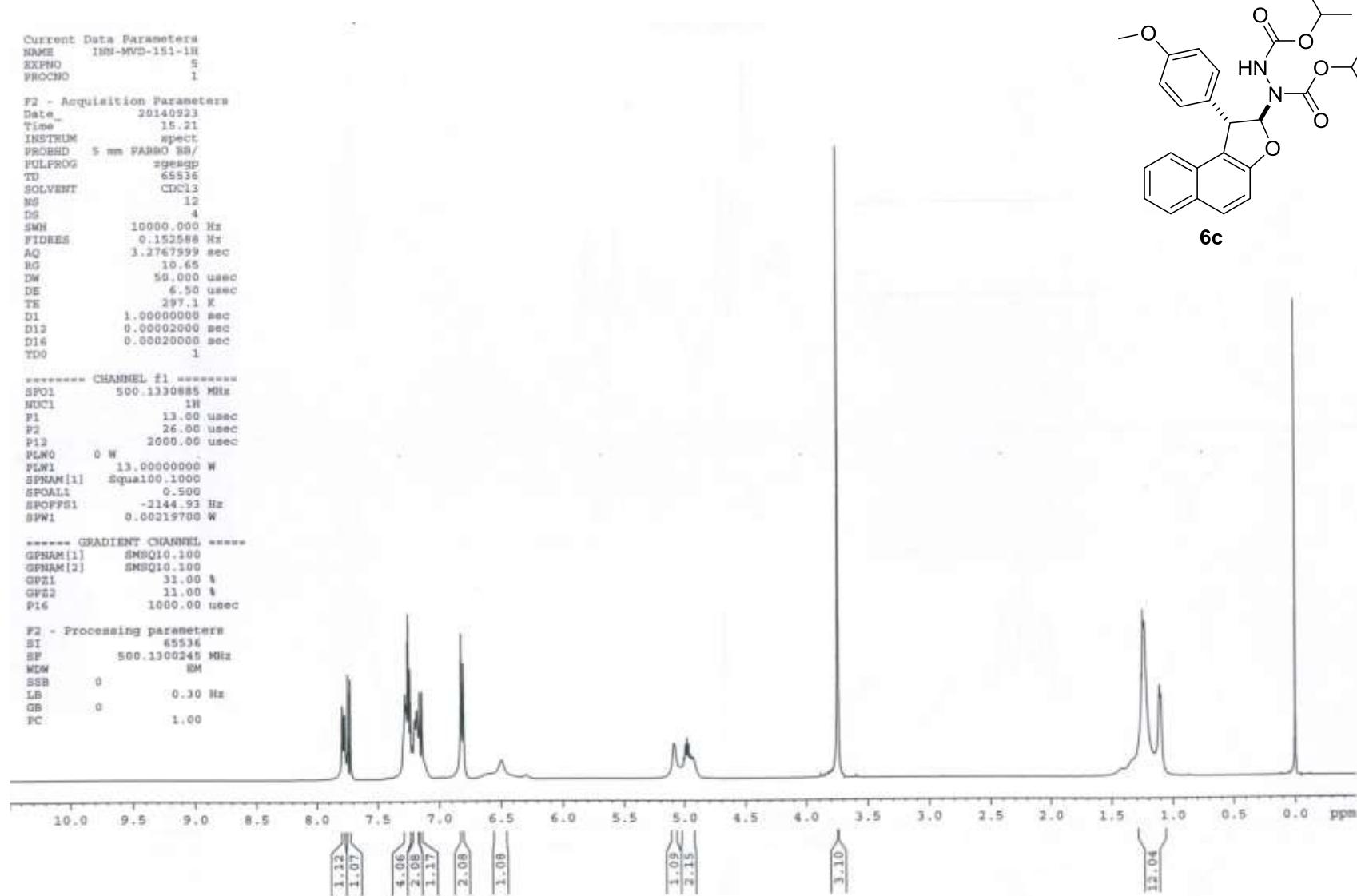
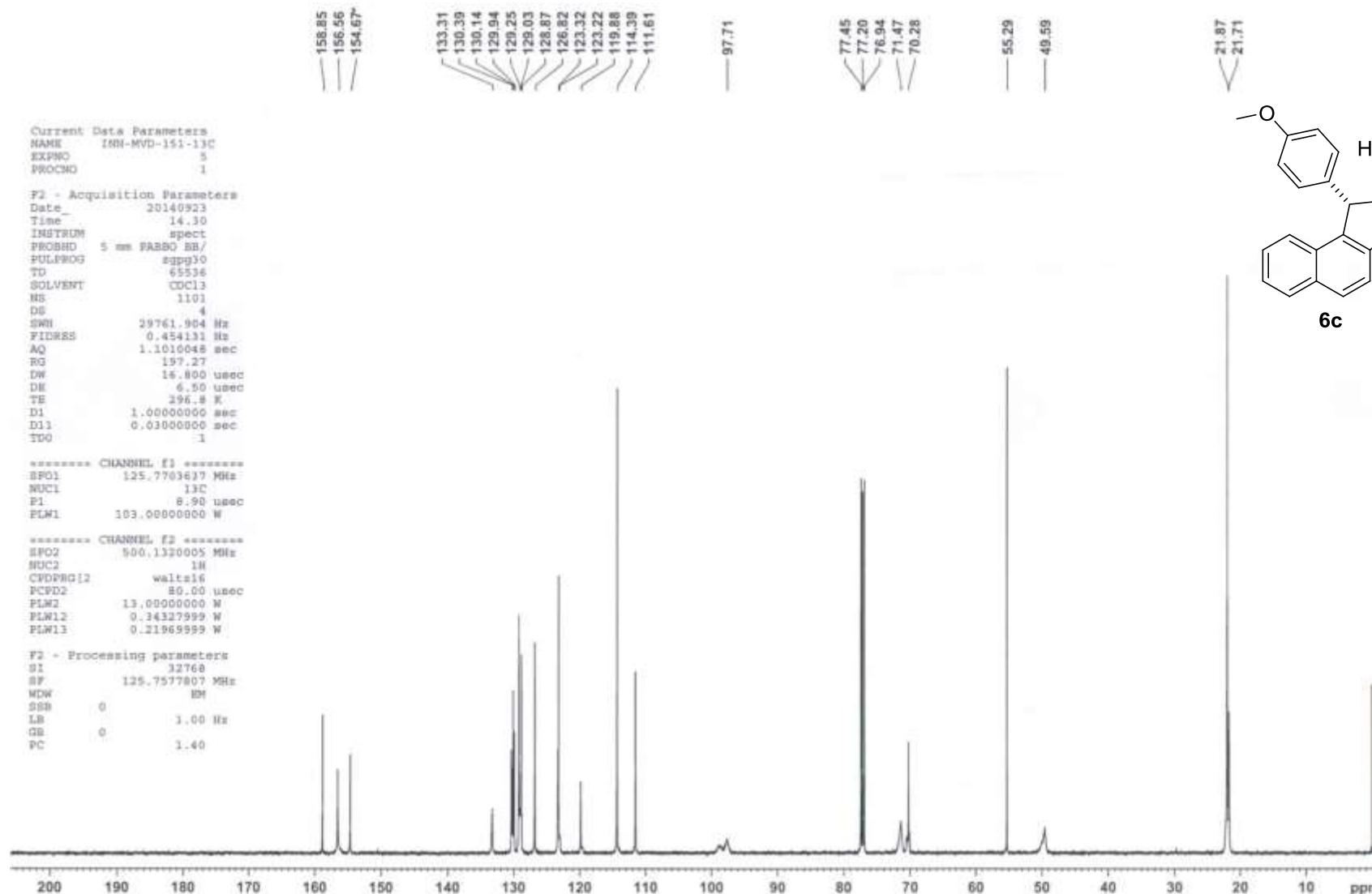
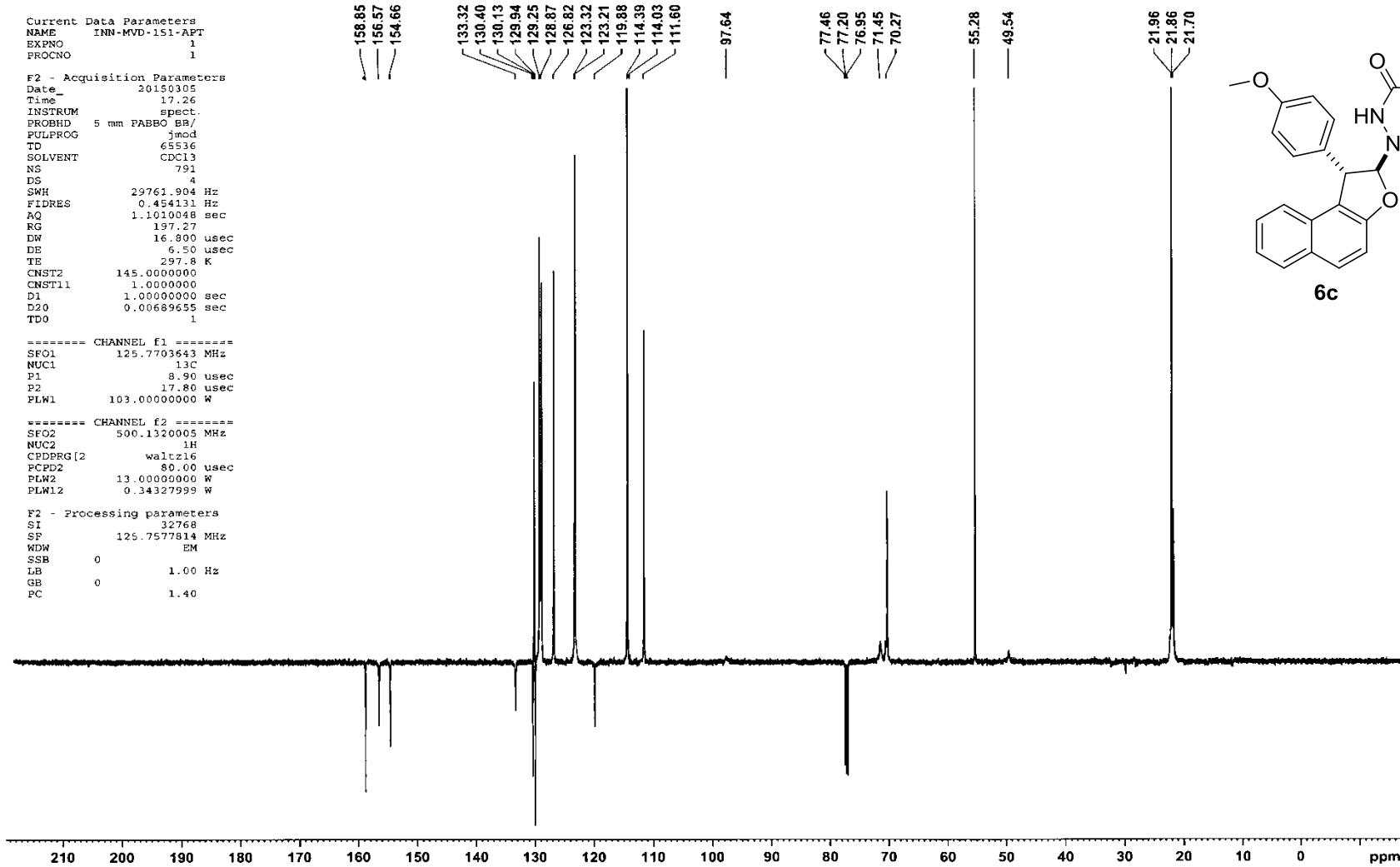


Figure S37.  $^1\text{H}$  NMR Spectrum of **6c**





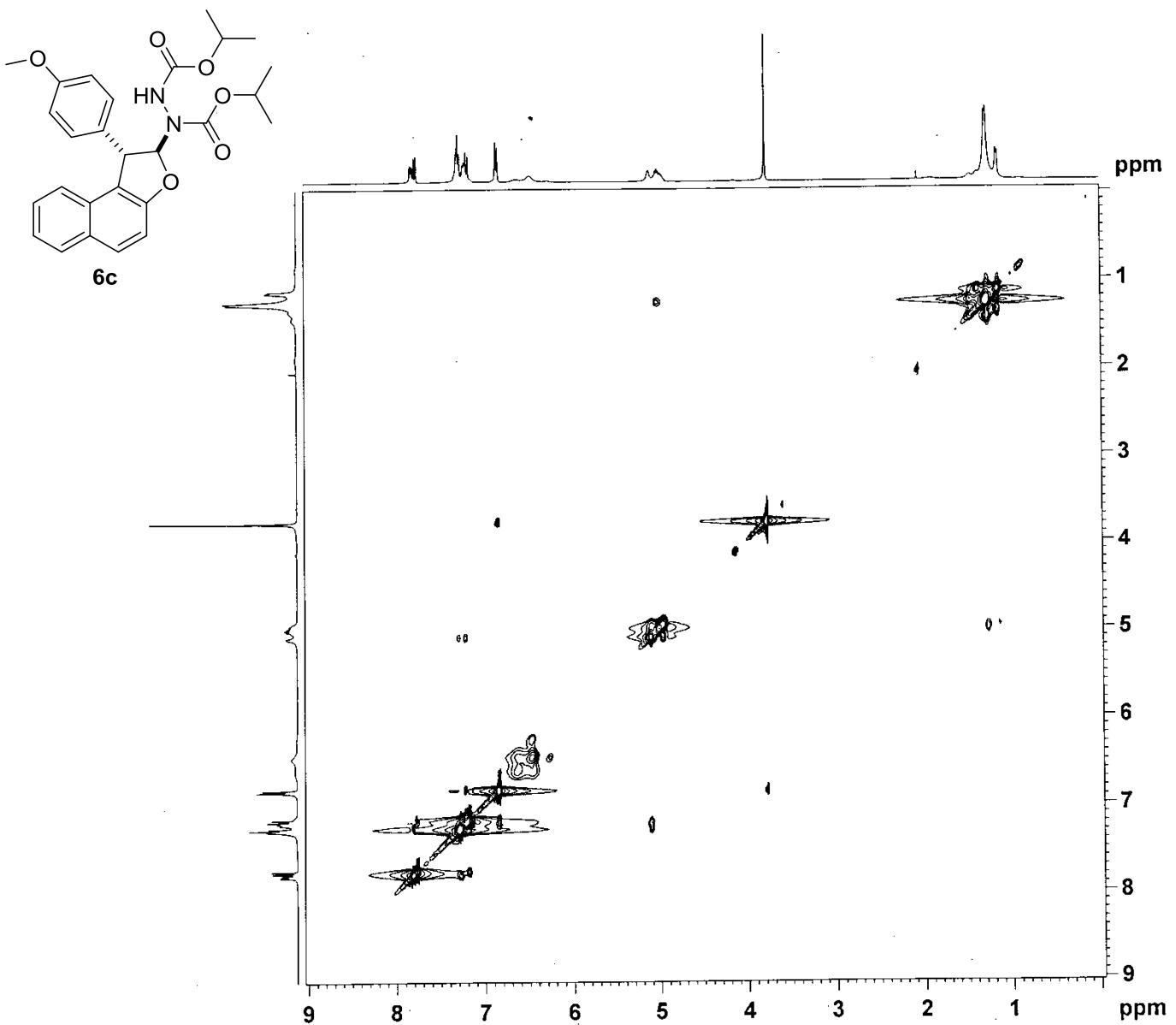


Figure S40.  $^1\text{H}$ - $^1\text{H}$ NNOESY NMRSpectrum of **6c**

Current Data Parameters  
 NAME INN-MVD-151-NOESY  
 EXPNO 7  
 PROCNO 1  
  
 F2 - Acquisition Parameters  
 Date 20151203  
 Time 17.45  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG noesypnshd  
 TD 2048  
 SOLVENT CDCl3  
 NS 32  
 DS 16  
 SWH 3633.721 Hz  
 FIDRES 1.774278 Hz  
 AQ 0.2818048 sec  
 RG 50.8  
 DW 137.600 usec  
 DE 6.50 usec  
 TE 295.6 K  
 D0 0.00012901 sec  
 D1 1.90374398 sec  
 D8 0.60000002 sec  
 IN0 0.00027520 sec  
  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SF01 400.1318090 MHz  
  
 F1 - Acquisition parameters  
 TD 324  
 SF01 400.1318 MHz  
 FIDRES 11.215176 Hz  
 SW 9.081 ppm  
 FmMode States-TPPI  
  
 F2 - Processing parameters  
 SI 2048  
 SF 400.1300095 MHz  
 WDW QSINE  
 SSB 2  
 LB 0 Hz  
 GB 0  
 PC 1.00  
  
 F1 - Processing parameters  
 SI 512  
 MC2 States-TPPI  
 SF 400.1300095 MHz  
 WDW QSINE  
 SSB 2  
 LB 0 Hz  
 GB 0

Current Data Parameters  
NAME INN-MVD-157-1H  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20140921  
Time 17.25  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 20  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 298.5 K  
D1 1.0000000 sec  
TDO 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
SP01 500.1330885 MHz  
NUC1 1H  
PI 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300053 MHz  
WDW DM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

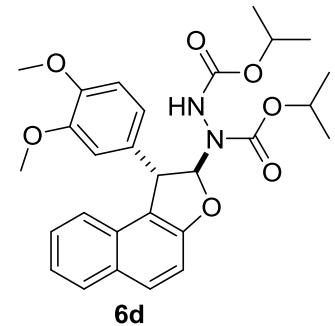
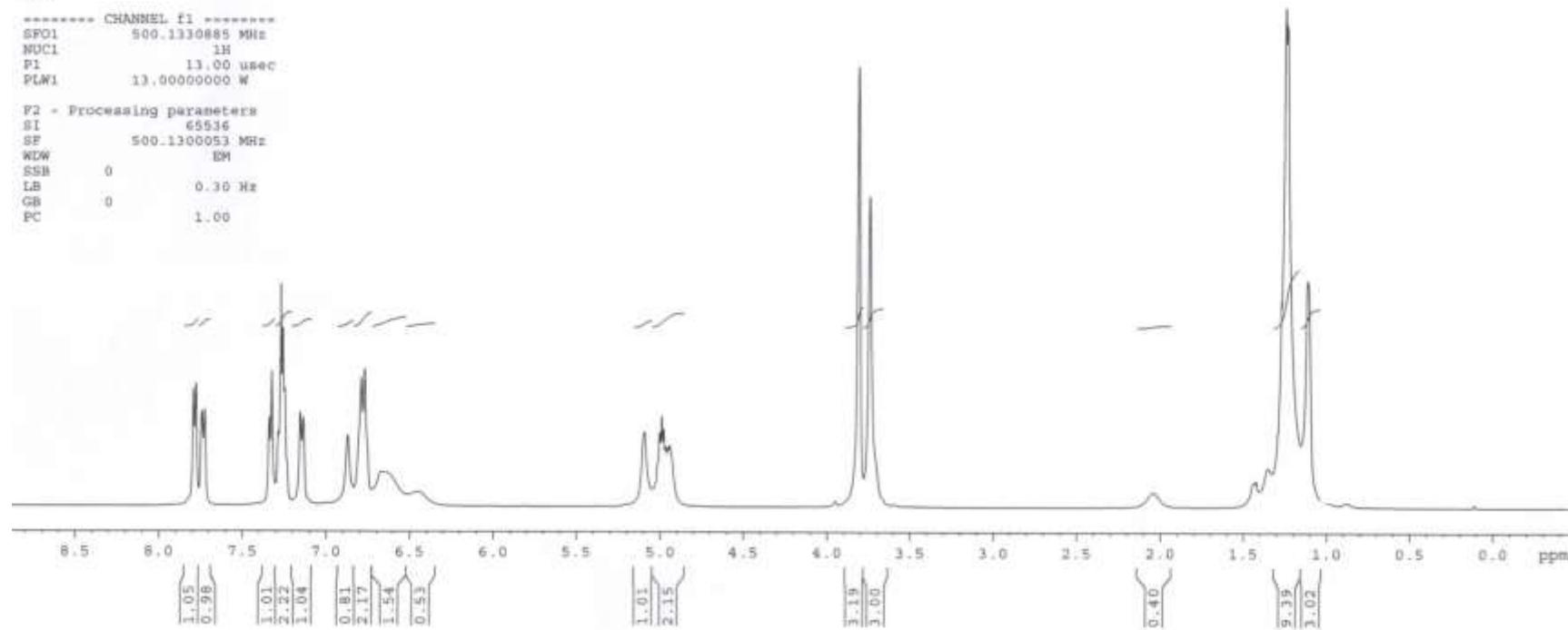


Figure S41. <sup>1</sup>H NMR Spectrum of **6d**

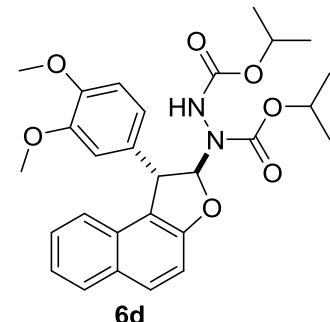
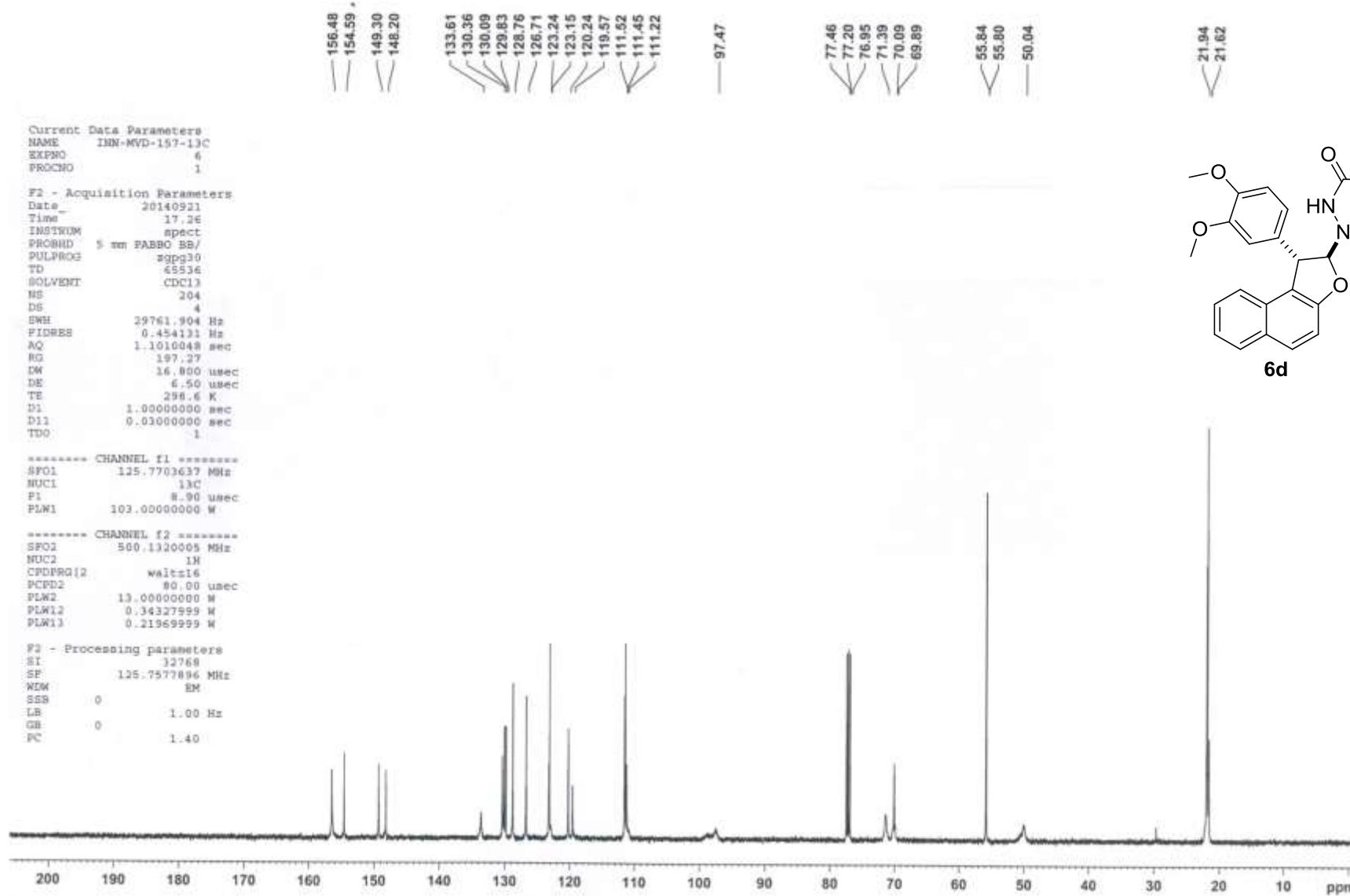


Figure S42.  $^{13}\text{C}$  NMR Spectrum of **6d**

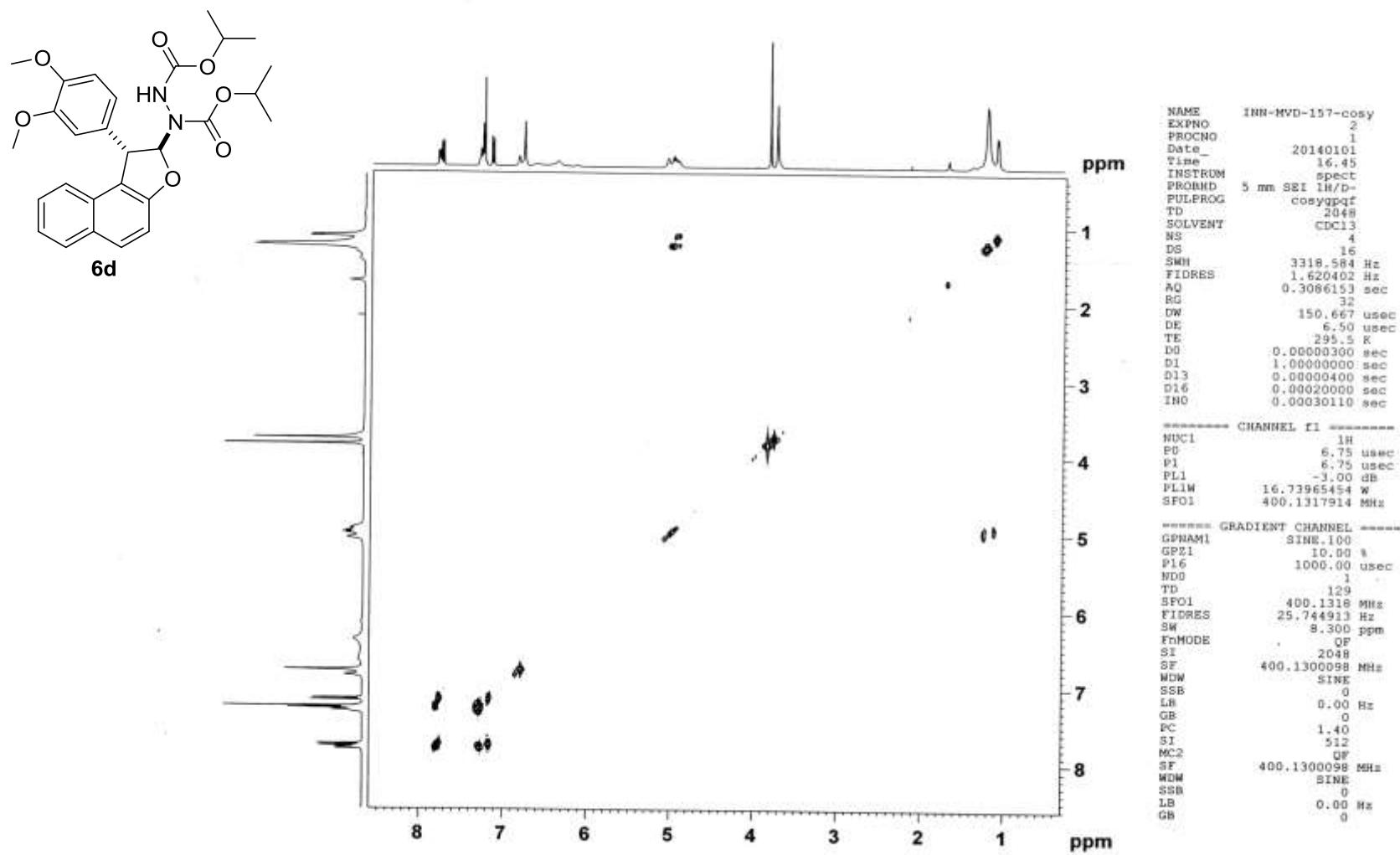


Figure S43.  $^1\text{H}$ - $^1\text{H}$  COSY NMR Spectrum of **6d**

```

Current Data Parameters
NAME      INN-MVD-16J-1H
EXPNO     2
PROCNO    1

P2 - Acquisition Parameters
Date_   20140923
Time    13.28
INSTRUM spect
PROBHD  5 mm PABBO BB/
PULPROG  zg30
TD      65536
SOLVENT  CDCl3
NS      20
DS      2
SWH    10000.000 Hz
FIDRES  0.152588 Hz
AQ      3.2767399 sec
RG      30.72
DW      50.000 usec
DE      6.50 usec
TE      296.1 K
DI      1.0000000 sec
TDO     1

***** CHANNEL f1 *****
SF01    500.1330005 MHz
NUC1    1H
P1      13.00 usec
PLW1   13.00000000 W

P2 - Processing parameters
SI      65536
SF      500.13300119 MHz
WDW    EM
SSB     0
LB      0.30 Hz
GB     0
PC      1.00

```

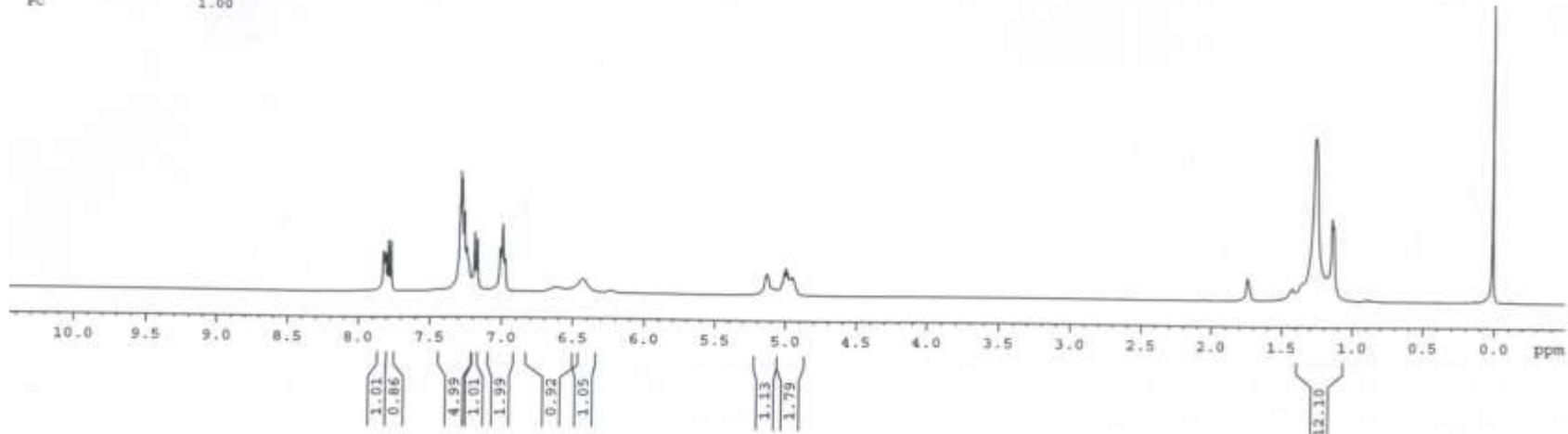
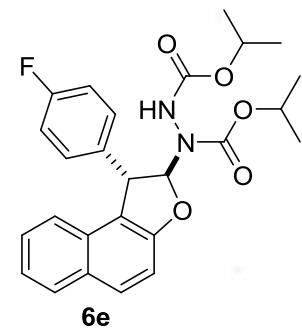


Figure S44.  $^1\text{H}$  NMR Spectrum of **6e**



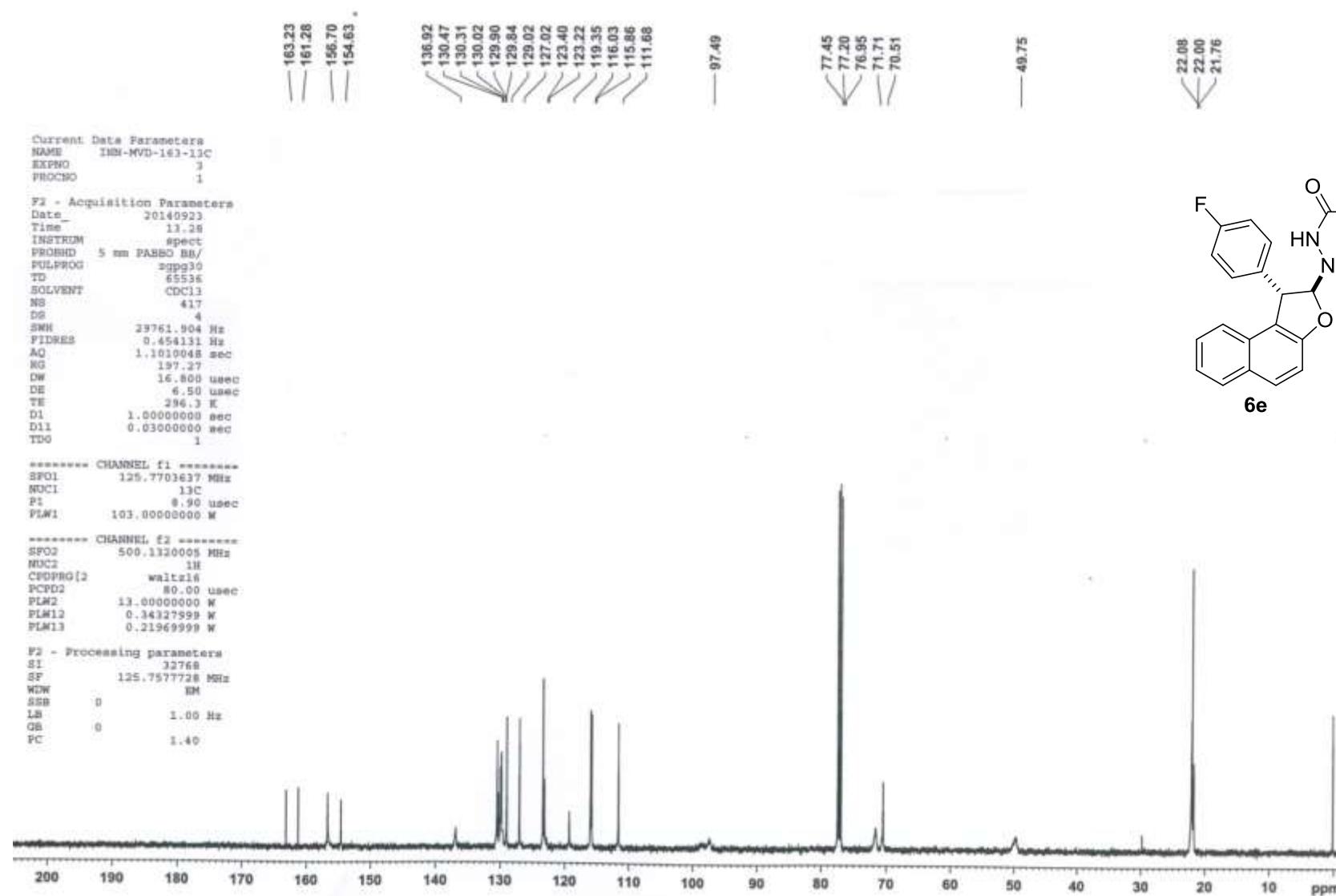


Figure S45. <sup>13</sup>C NMR Spectrum of **6e**

Current Data Parameters  
 NAME INN-MVD-163-19F  
 EXPNO :  
 PROCNO :  
 F2 - Acquisition Parameters  
 Date\_ 20140924  
 Time 19.12  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgflqnd  
 TD 131072  
 SOLVENT CDCl3  
 NS 90  
 DS 4  
 SWH 113636.367 Hz  
 FIDRES 0.866977 Hz  
 AQ 0.5767168 sec  
 RG 197.23  
 DW 4.400 usec  
 DE 6.50 usec  
 TE 297.8 K  
 D1 1.0000000 sec  
 TDO :  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 SPO1 470.5453180 MHz  
 NUC1 19F  
 P1 13.80 usec  
 PLW1 40.0000000 M  
 F2 - Processing parameters  
 SI 65536  
 SF 470.5923770 MHz  
 WMW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

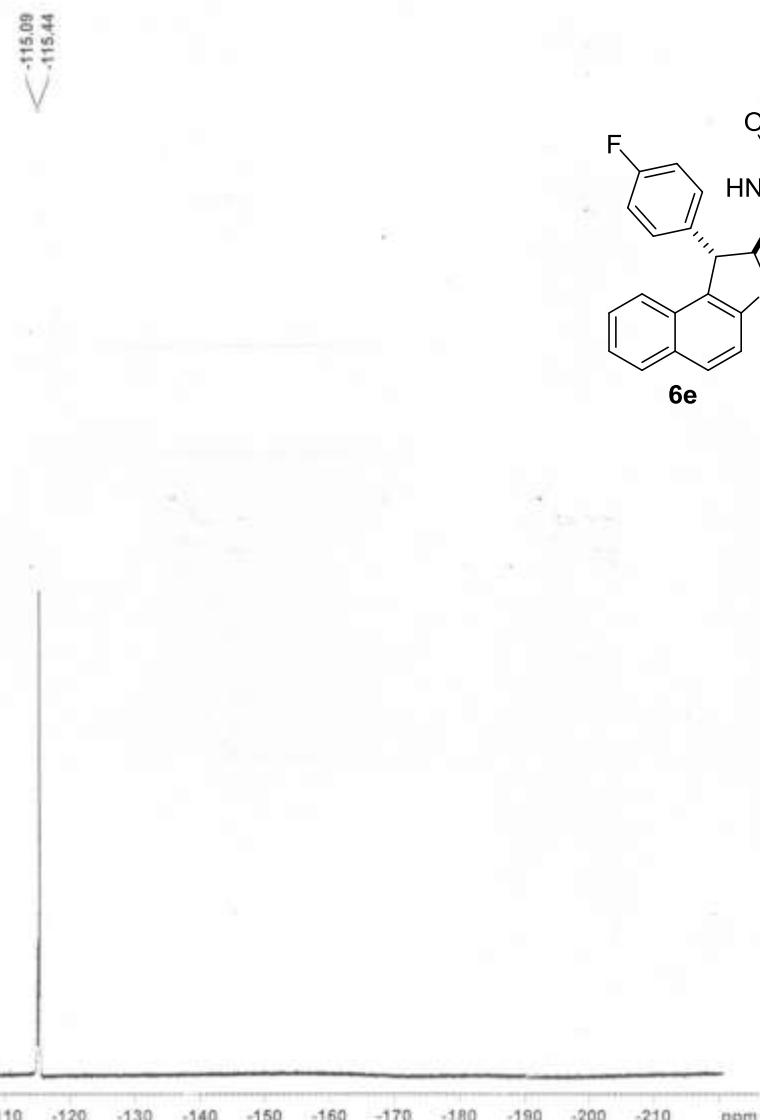


Figure S46.  $^{19}\text{F}$  NMR Spectrum of **6e**

Current Data Parameters  
NAME INN-MVD-153-1H  
EXPNO 1  
PROCNO 1  
P2 - Acquisition Parameters  
Date\_ 20140806  
Time 10:33  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 30  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 294.4 K  
D1 1.0000000 usec  
TDO 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
SF01 500.1130885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 12.0000000 W

P2 - Processing parameters  
SI 65536  
SF 500.11300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
QSB 0  
PC 1.00

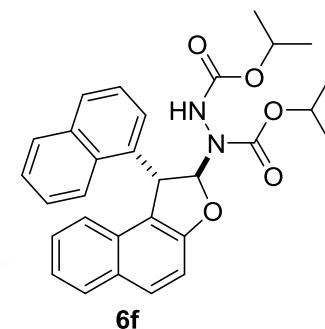
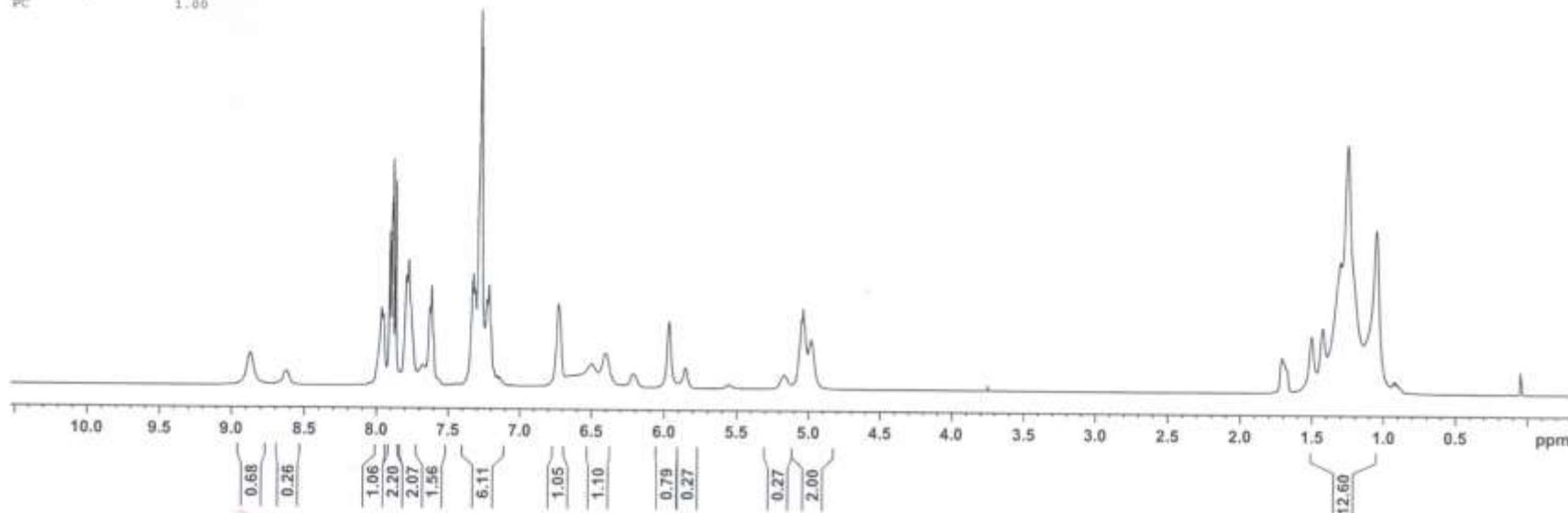
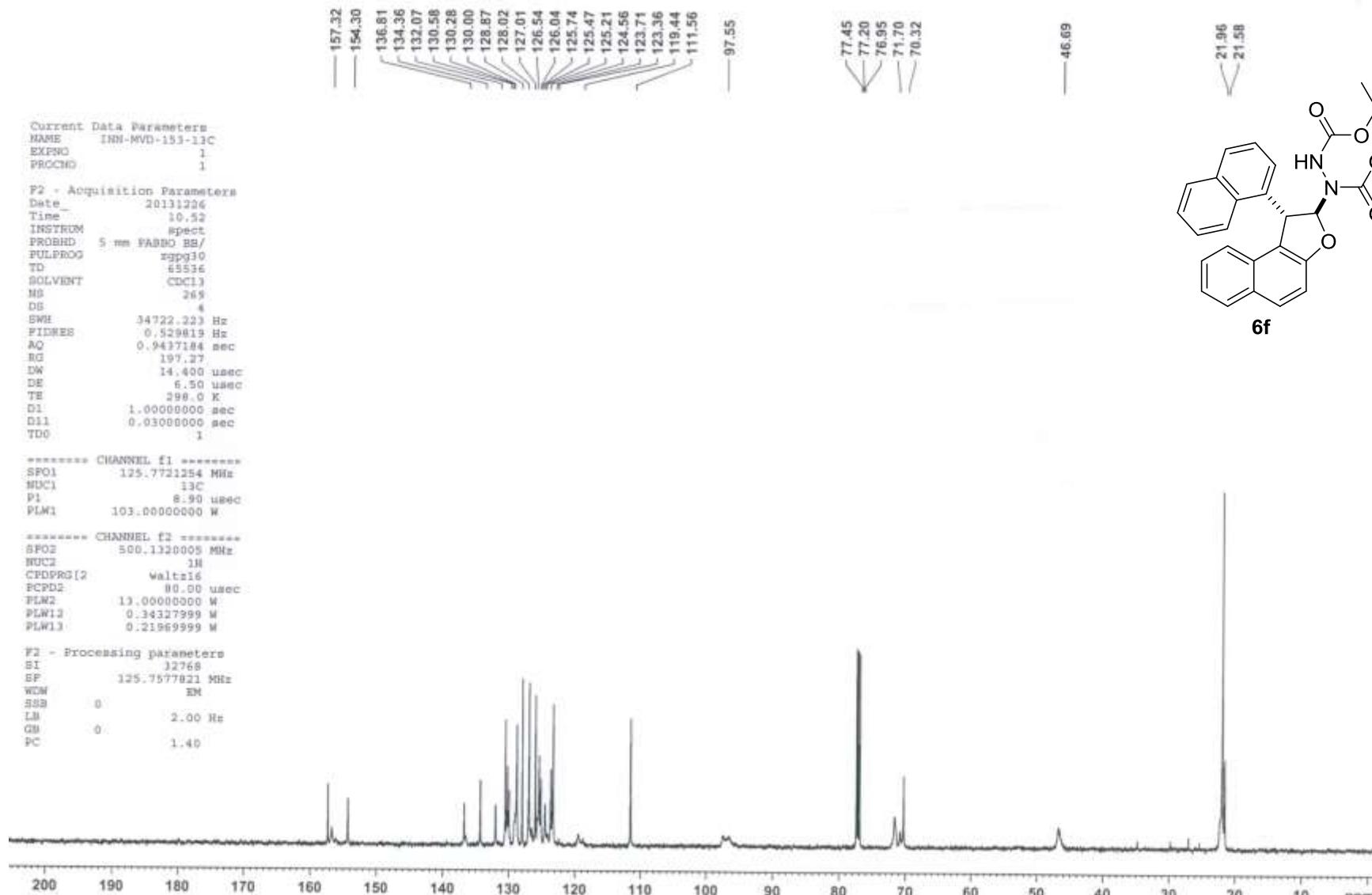


Figure S47.  $^1\text{H}$  NMR Spectrum of **6f**



```

Current Data Parameters
NAME      IMN-MWD-152-1H
EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_   20140921
Time       17:01
INSTRUM  spect
PROBHD  5 mm PABBO BB/
PULPROG  zg3g
TD      65536
SOLVENT   CDCl3
NS          20
DS           2
SW0T    10000.000 Hz
FIDRES  0.152588 Hz
AQ     3.2767999 sec
RG        30.72
DW       50.000 usec
DE        6.50 usec
TE       298.4 K
D1    1.0000000 sec
TD0             1

***** CHANNEL fi *****
SPO1    500.1330085 MHz
NUC1        1H
P1       13.00 usec
PIWI    13.0000000 W

P2 - Processing parameters
SI       65536
SP      500.13300215 MHz
WDW        EM
SSB        0
LB       0.30 Hz
GB        0
PC        1.00

```

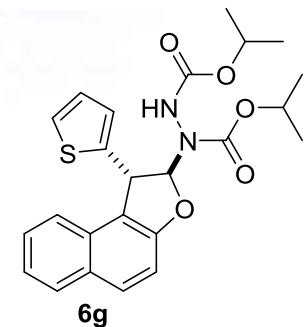
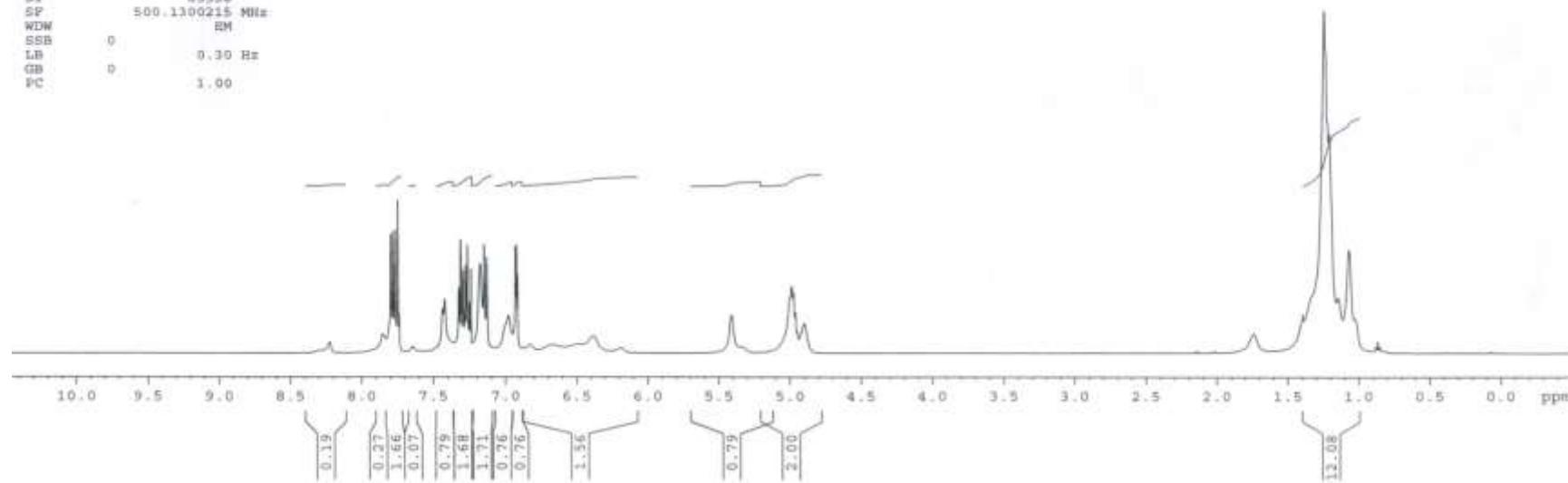


Figure S49.  $^1\text{H}$  NMR Spectrum of **6g**

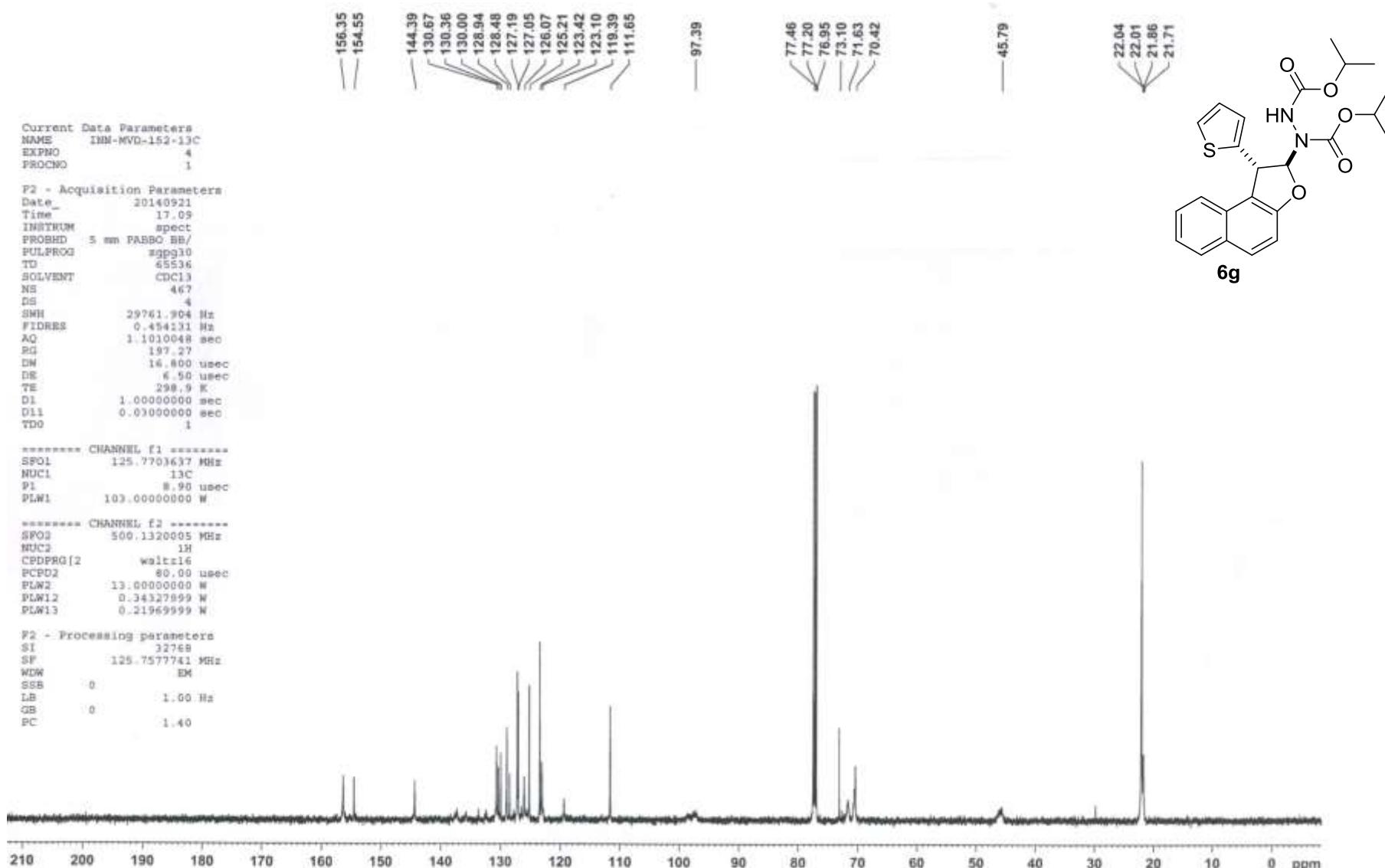


Figure S50. <sup>13</sup>C NMR Spectrum of **6g**

NAME INN-MVD-165-1H  
 EXPNO 7  
 PROCNO 1  
 Date 20131219  
 Time 10.25  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/0-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWR 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 90.5  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.6 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 PI 6.75 usec  
 PLL -3.00 dB  
 PL1W 16.73965454 W  
 SPOL 400.1324710 MHz  
 SI 32768  
 SF 400.1300106 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

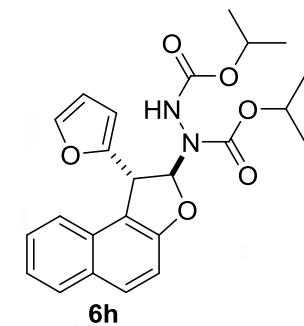
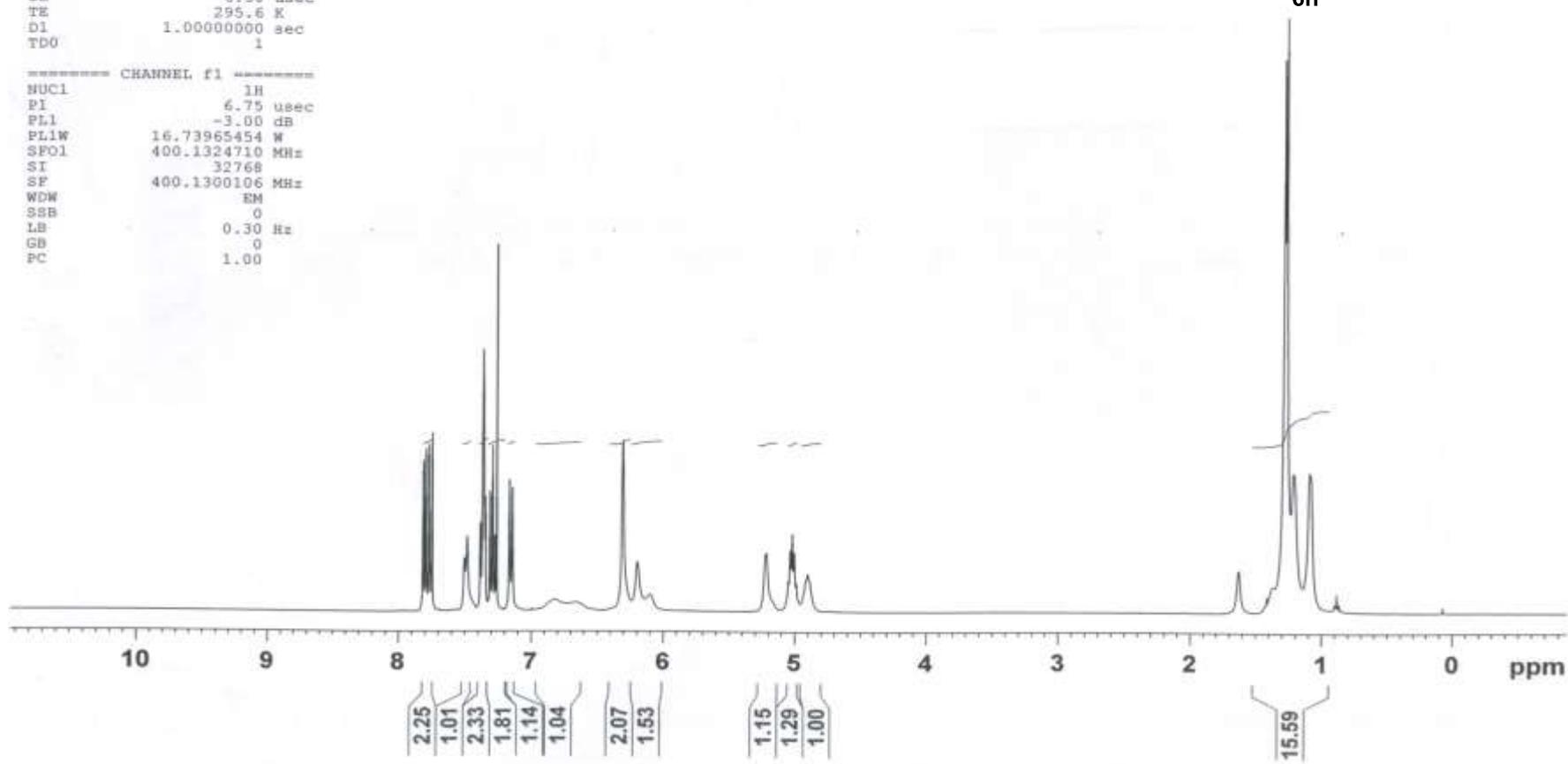
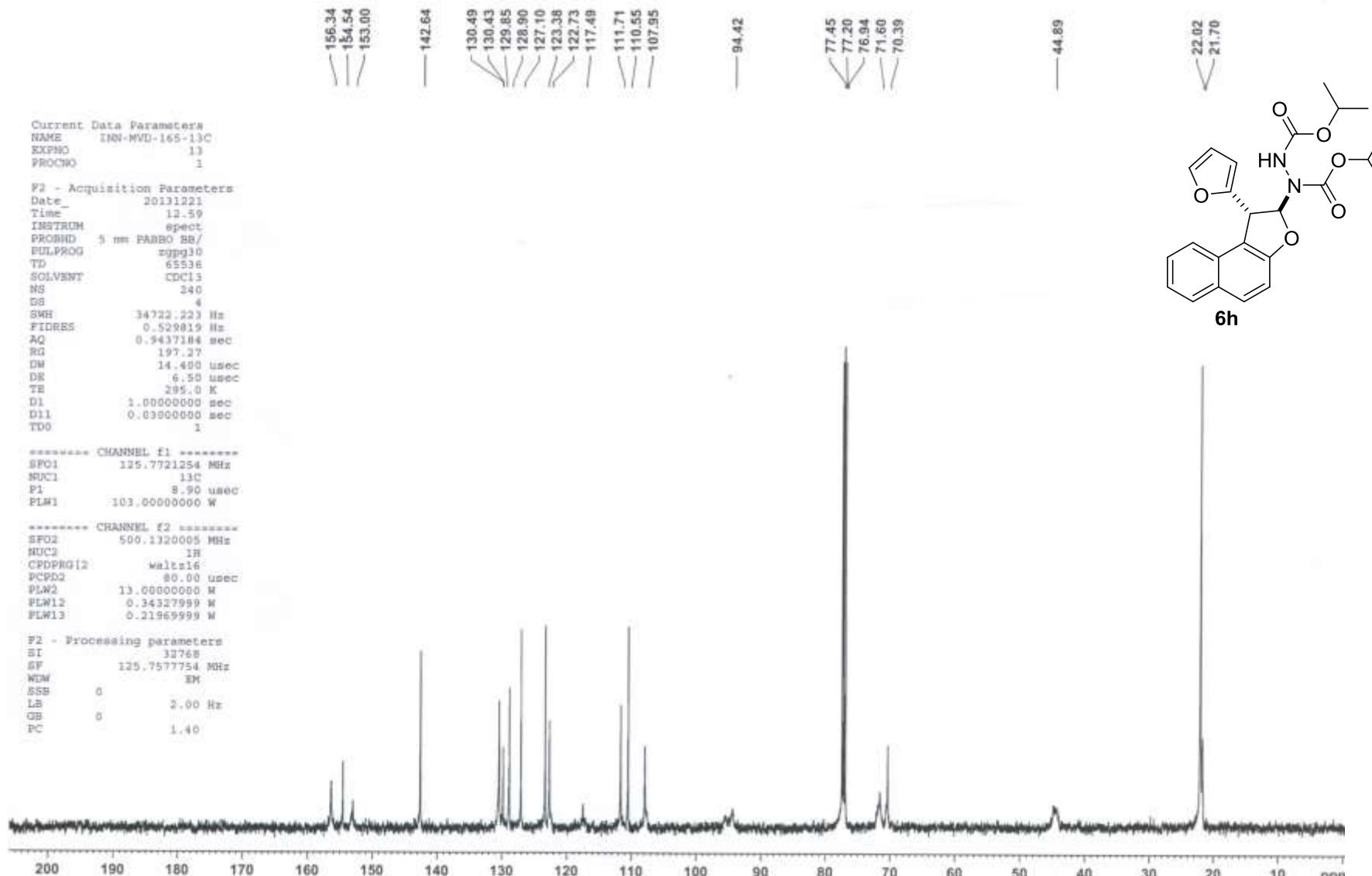


Figure S51. <sup>1</sup>H NMR Spectrum of **6h**



NAME INN-MVD-320-1R  
 EXPNO 1  
 PROCHNO 1  
 Date 20140930  
 Time 18:23  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 54274  
 SOLVENT CDCl3  
 NS 20  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.151522 Hz  
 AQ 3.2999091 sec  
 RG 32  
 DW 60.000 usec  
 DE 6.50 usec  
 TE 297.9 E  
 D1 1.0000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 NOCL 1H  
 F1 14.75 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300268 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

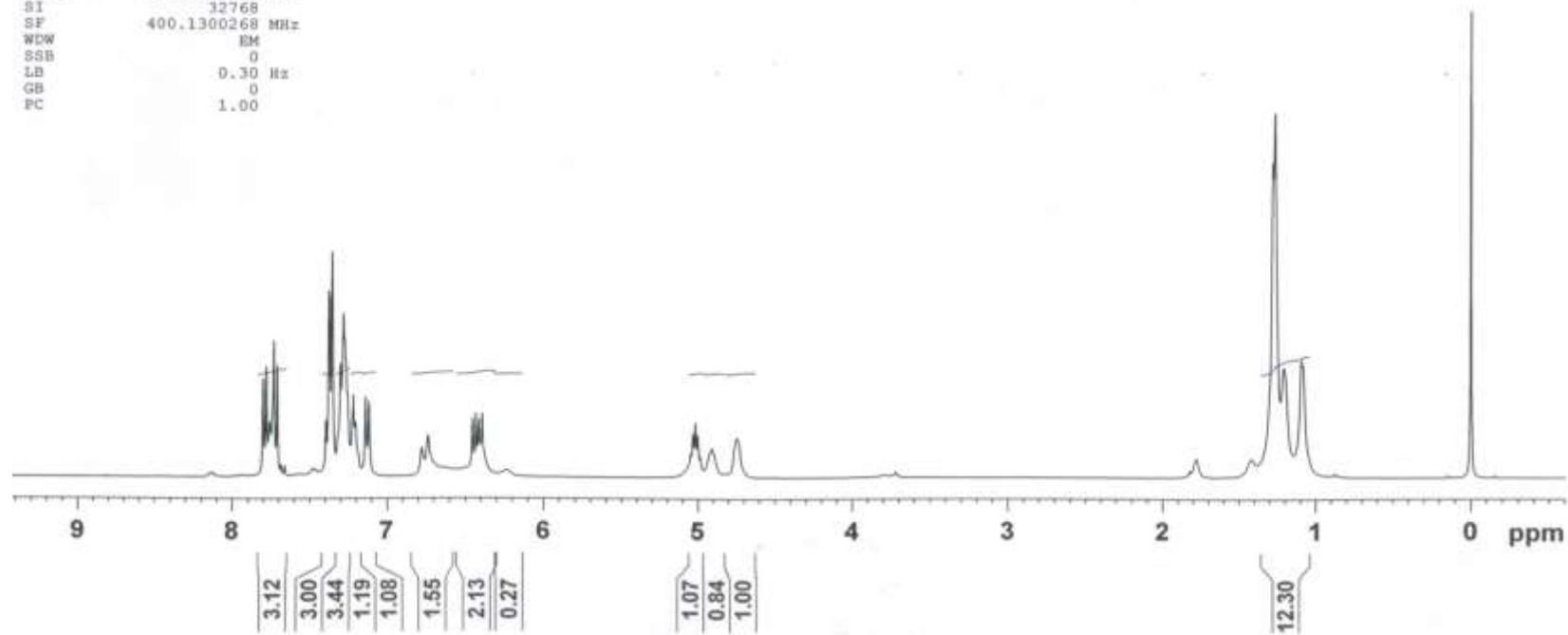
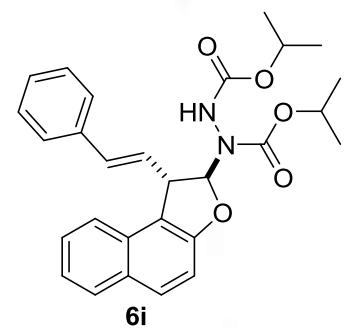
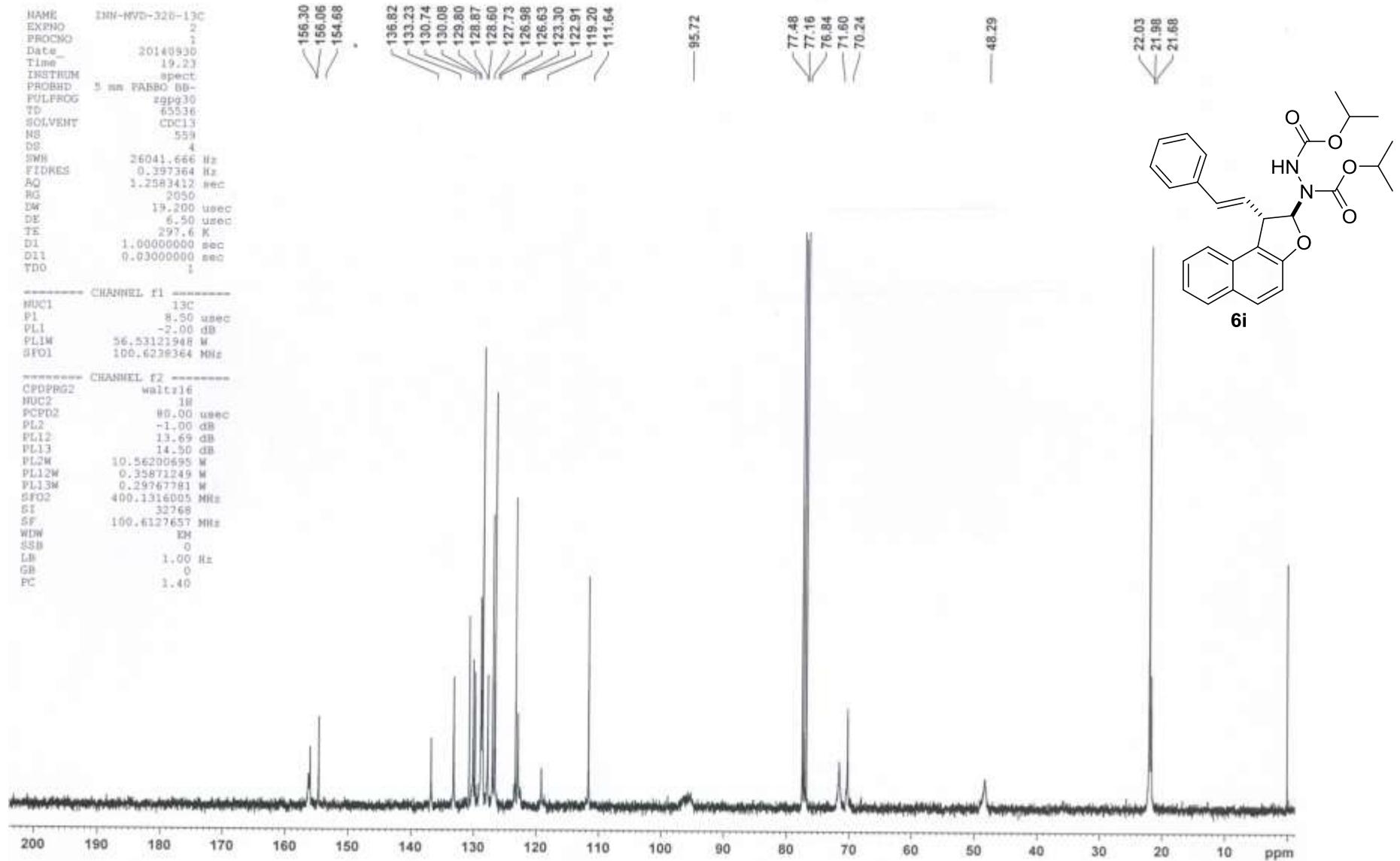


Figure S53.  $^1\text{H}$  NMR Spectrum of **6i**





Current Data Parameters  
 NAME JNN-MVD-155-1H  
 EXPNO 9  
 PROCHD 1  
 P2 - Acquisition Parameters  
 Date 20131225  
 Time 21:53  
 INSTRUM spect  
 PROBHD 5 mm PABBO-BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 30.72 sec  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 296.6 K  
 D1 1.0000000 sec  
 TDD 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 SFO1 500.1330885 MHz  
 NUC1 1H  
 PI 13.00 usec  
 PLWI 13.0000000 W

P2 - Processing parameters  
 SI 65536  
 SF 500.1300000 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

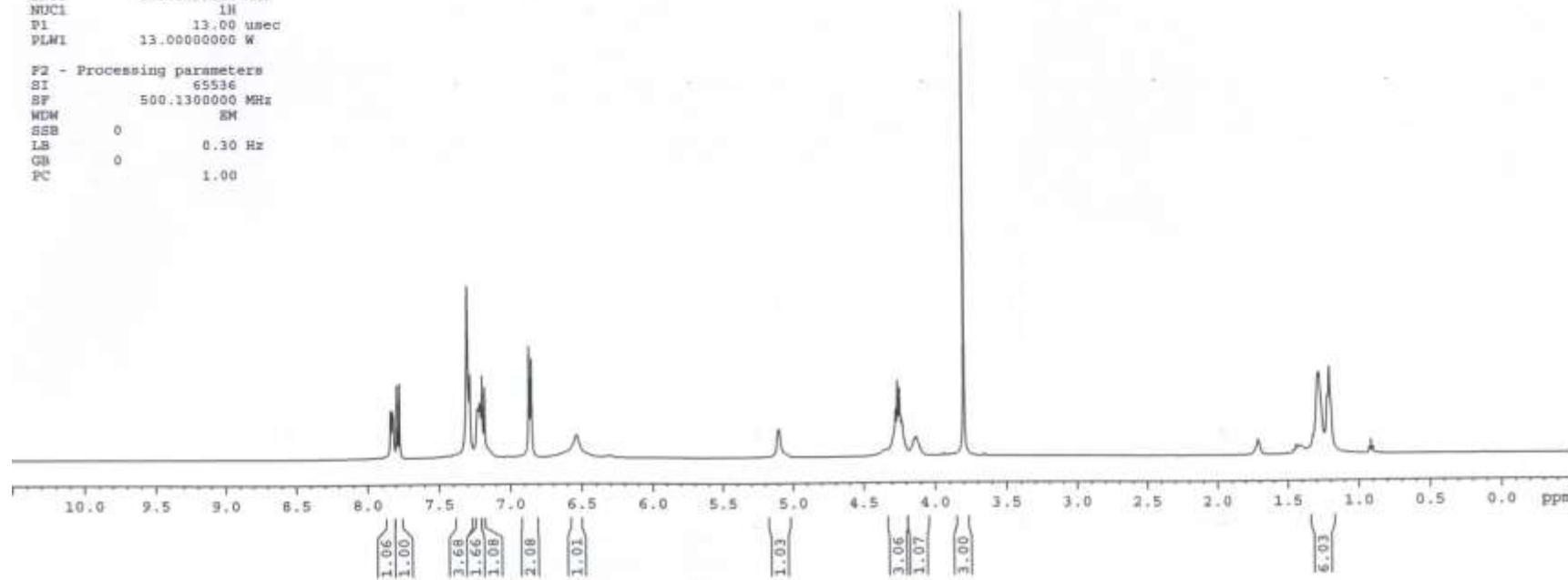
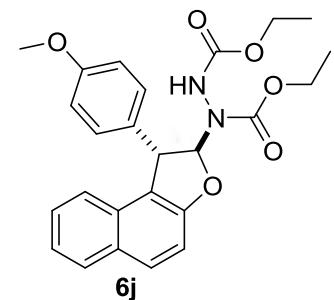


Figure S55.  $^1\text{H}$  NMR Spectrum of **6j**



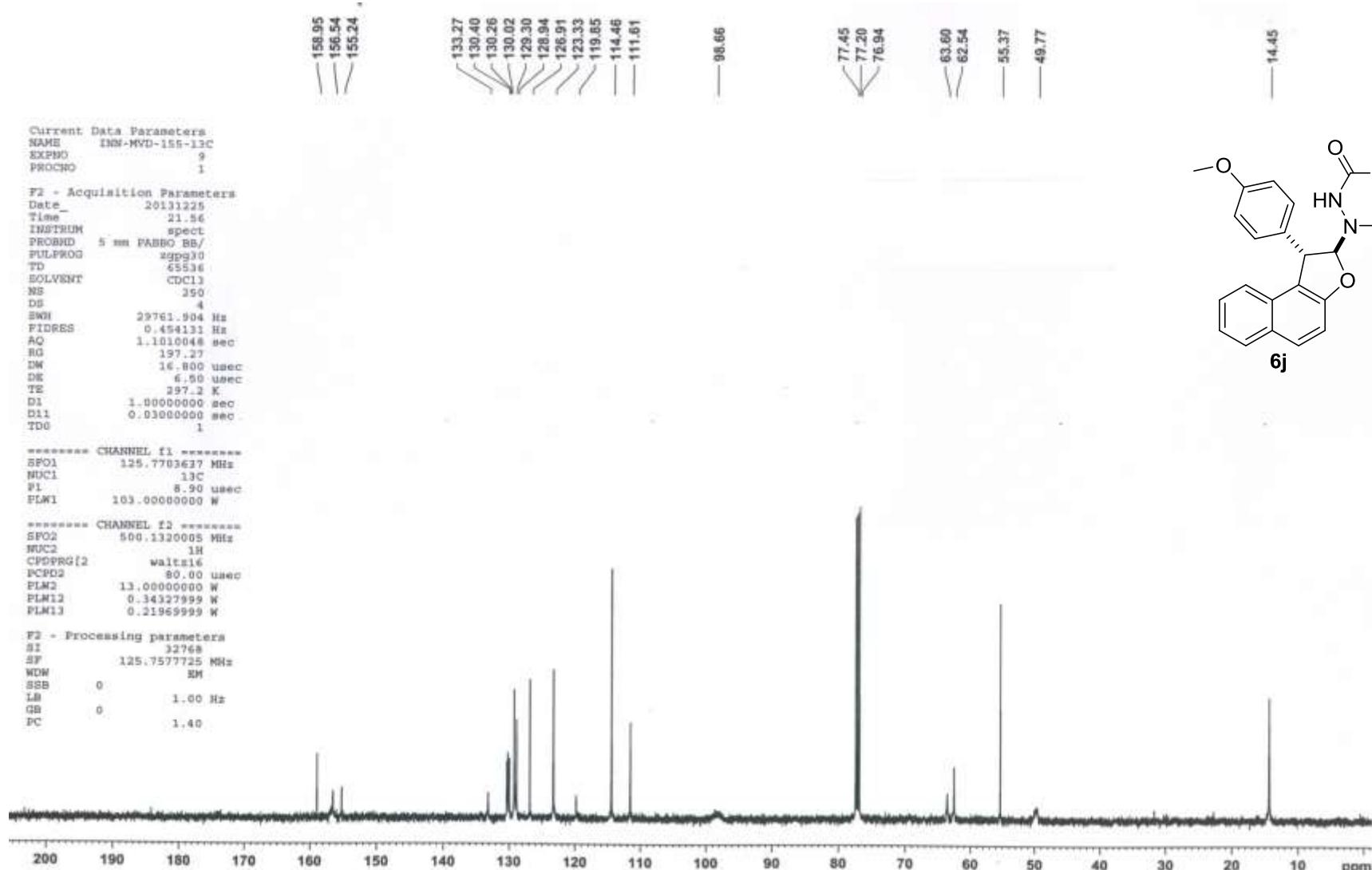


Figure S56.  $^{13}\text{C}$  NMR Spectrum of **6j**

```

NAME      INN-MVD-169-1H
EXPRO        4
PROCNO       1
Date      20141014
Time       18.25
INSTRUM    spect
PROBHD   5 mm PABBO BB-
PULPROG   zgpr
TD        54274
SOLVENT    CDCl3
NS         14
DS          0
SWH       12019.230 Hz
FIDRES    0.221455 Hz
AQ        2.2578485 sec
RG         32
DW        41.600 usec
DE         6.50 usec
TE        297.3 K
D1     1.0000000 sec
D12    0.00002000 sec
TDD          1

```

```

----- CHANNEL f1 -----
NUC1           1H
P1        14.75 usec
PL1        -1.00 dB
PL9        38.23 dB
PL1W    10.56200695 W
PL9W    0.00126109 W
SF01      400.1300279 MHz
SI         32768
SF      400.1300167 MHz
WDW         EM
SSB          0
LB        0.30 Hz
GB          0
PC         1.00

```

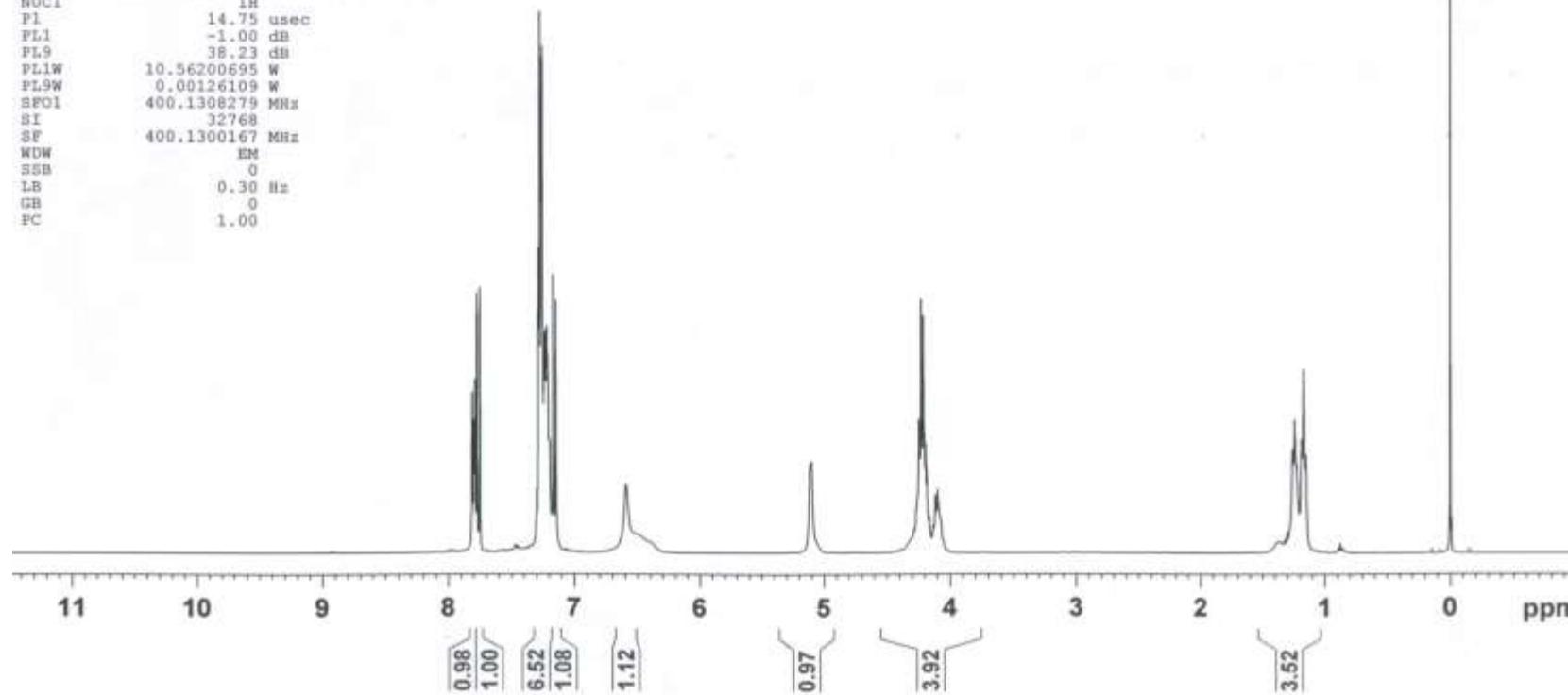
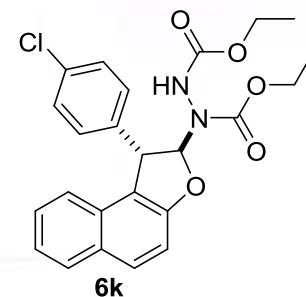


Figure S57. <sup>1</sup>H NMR Spectrum of **6k**



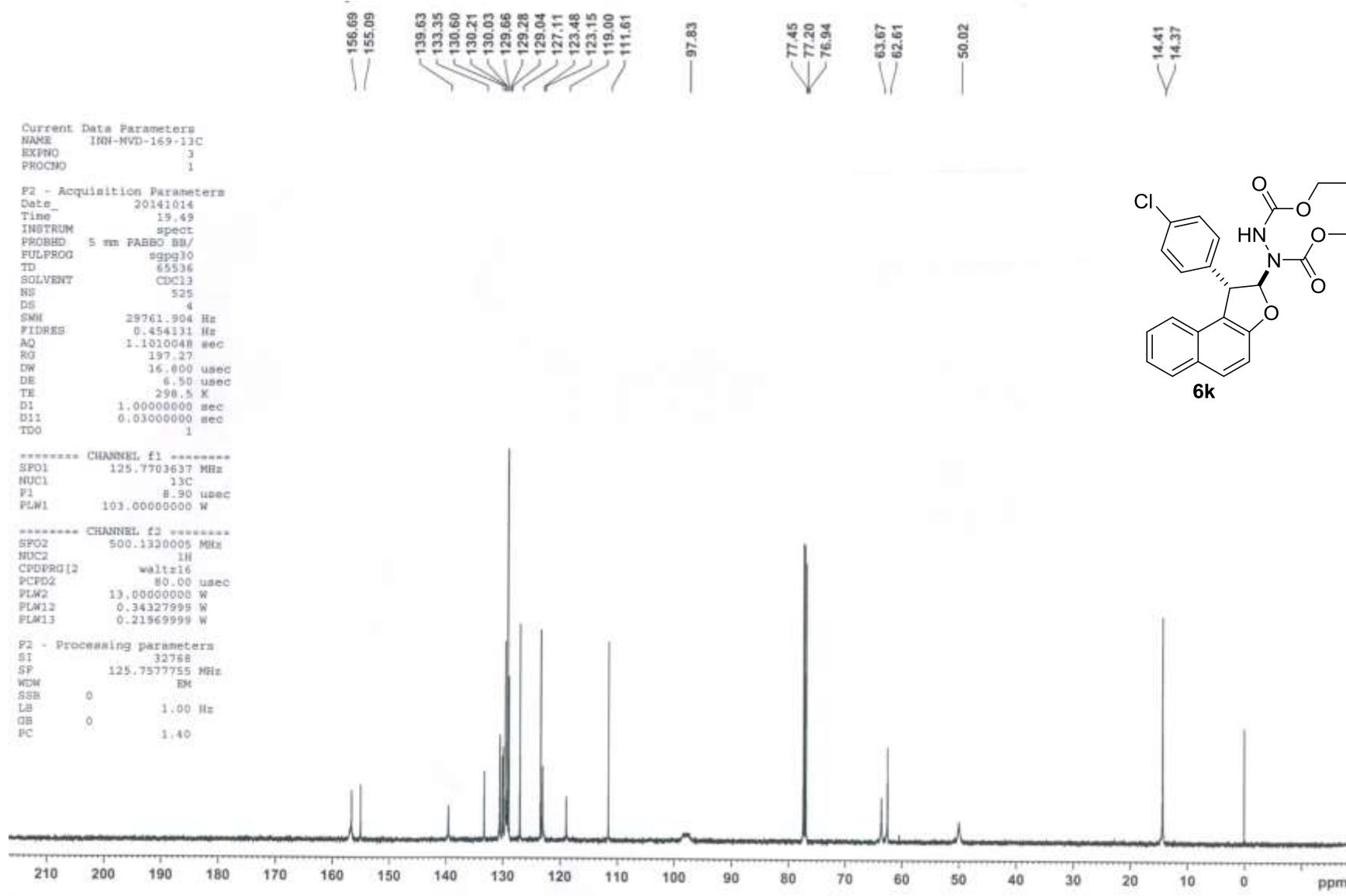


Figure S58. <sup>13</sup>C NMR Spectrum of **6k**

```

Current Data Parameters
NAME      INN-MVD-1H
EXPNO        6
PROCNO       1

P2 - Acquisition Parameters
Date_   20131120
Time     19.33
INSTRUM spect
PROBHDG  5 mm PABBO BB/
PULPROG  zg30
TD        65536
SOLVENT   CDCl3
NS         10
DS          2
SWH       10000.000 Hz
FIDRES   0.152500 Hz
AQ        3.2767999 sec
RG        30.72
DW        50.000 usec
DE        6.50 usec
TE        297.6 K
D1        1.0000000 sec
TD0             1

***** CHANNEL f1 *****
SFO1      500.1330885 MHz
NUC1        1H
P1        13.00 usec
PLW1      13.00000000 W

P2 - Processing parameters
SI        65536
SF        500.13000000 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        1.00

```

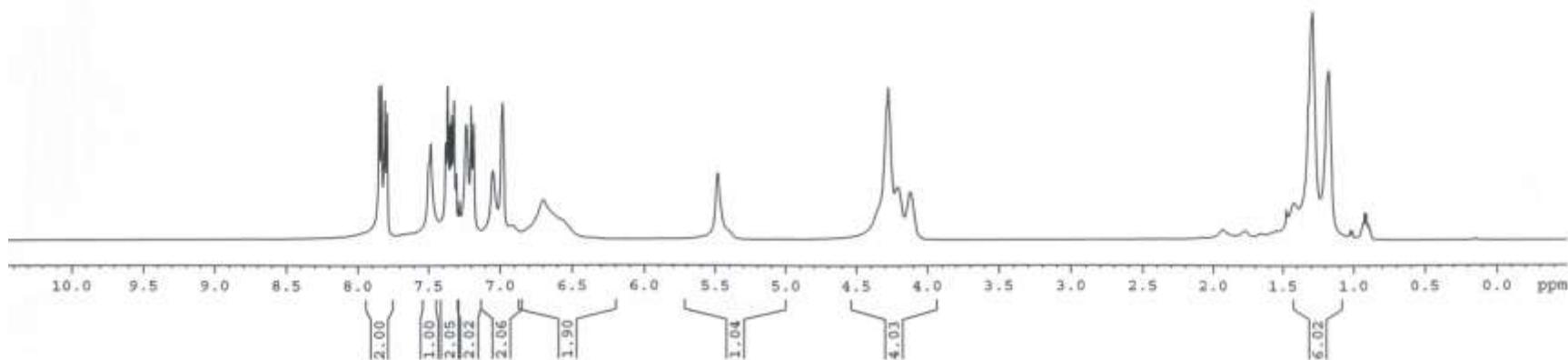
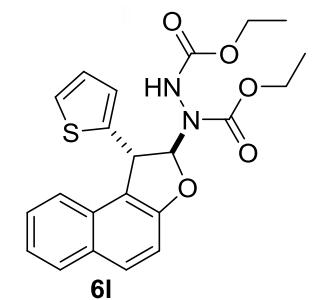


Figure S59.  $^1\text{H}$  NMR Spectrum of **6l**

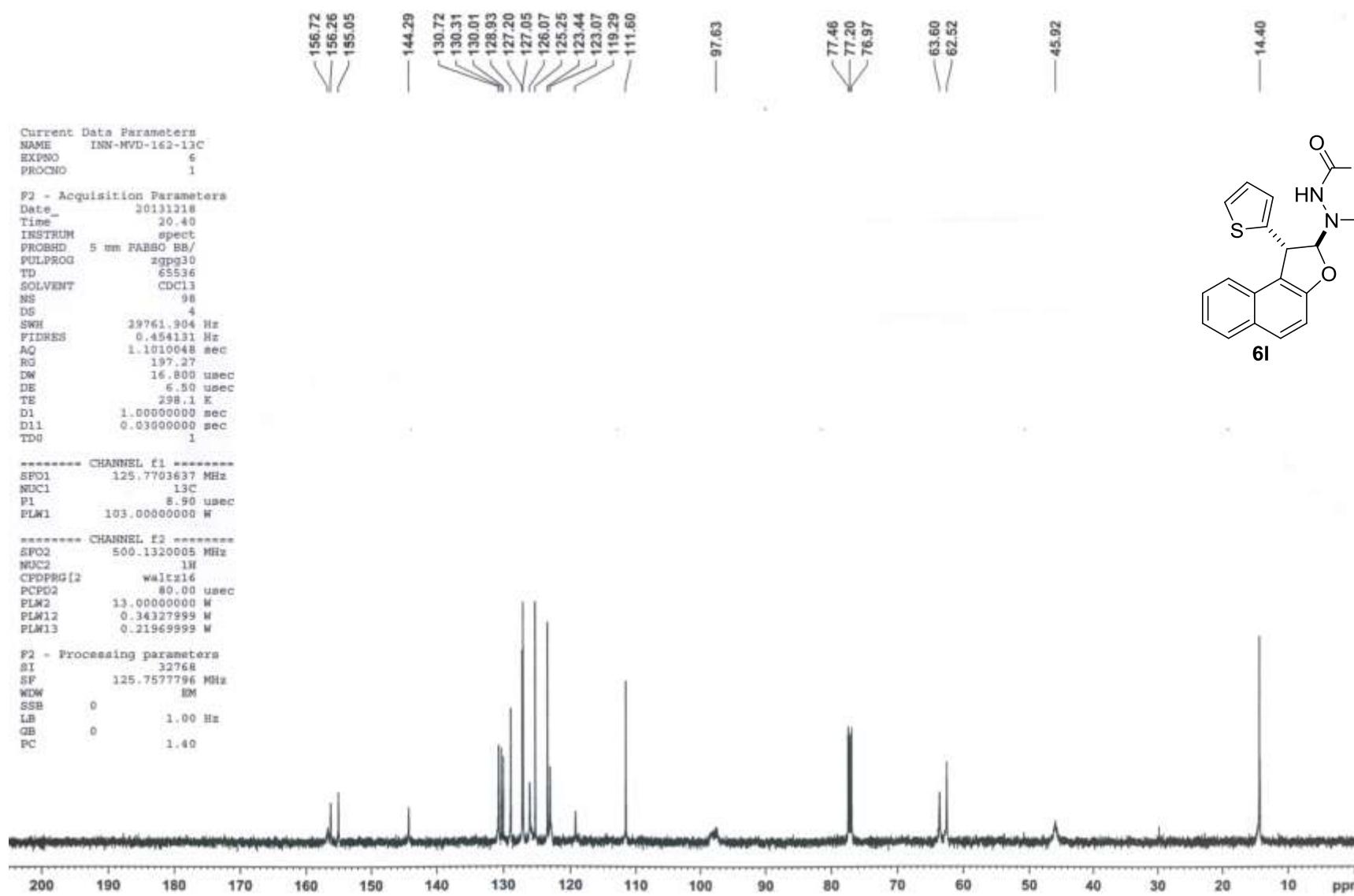
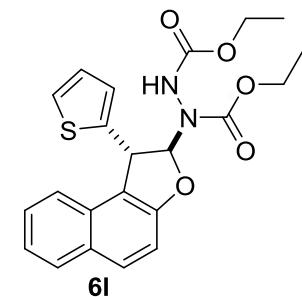


Figure S60.  $^{13}\text{C}$  NMR Spectrum of **6l**



NAME: INN-MVD-444-1H  
 EXPNO: 0  
 PROCHO: 1  
 Date: 20150210  
 Time: 21:29  
 INSTRUM: spect  
 FROBHD: 5 mm PAR80 BB-  
 PULPROG: zg30  
 T1: 54274  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 0  
 SWH: 8223.655 Hz  
 FIDRES: 0.151522 Hz  
 AQ: 3.2999091 sec  
 RG: 32  
 DW: 60.000 usec  
 DE: 6.50 usec  
 TE: 295.9 K  
 D1: 1.0000000 sec  
 TDO: 1

CHANNEL f1 -----  
 HUC1: 1H  
 F1: 14.75 usec  
 PLL: -1.00 dB  
 PL1W: 10.56200695 Hz  
 SP01: 400.1324710 MHz  
 ST: 32768  
 SF: 400.13300095 MHz  
 MDW: EH  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

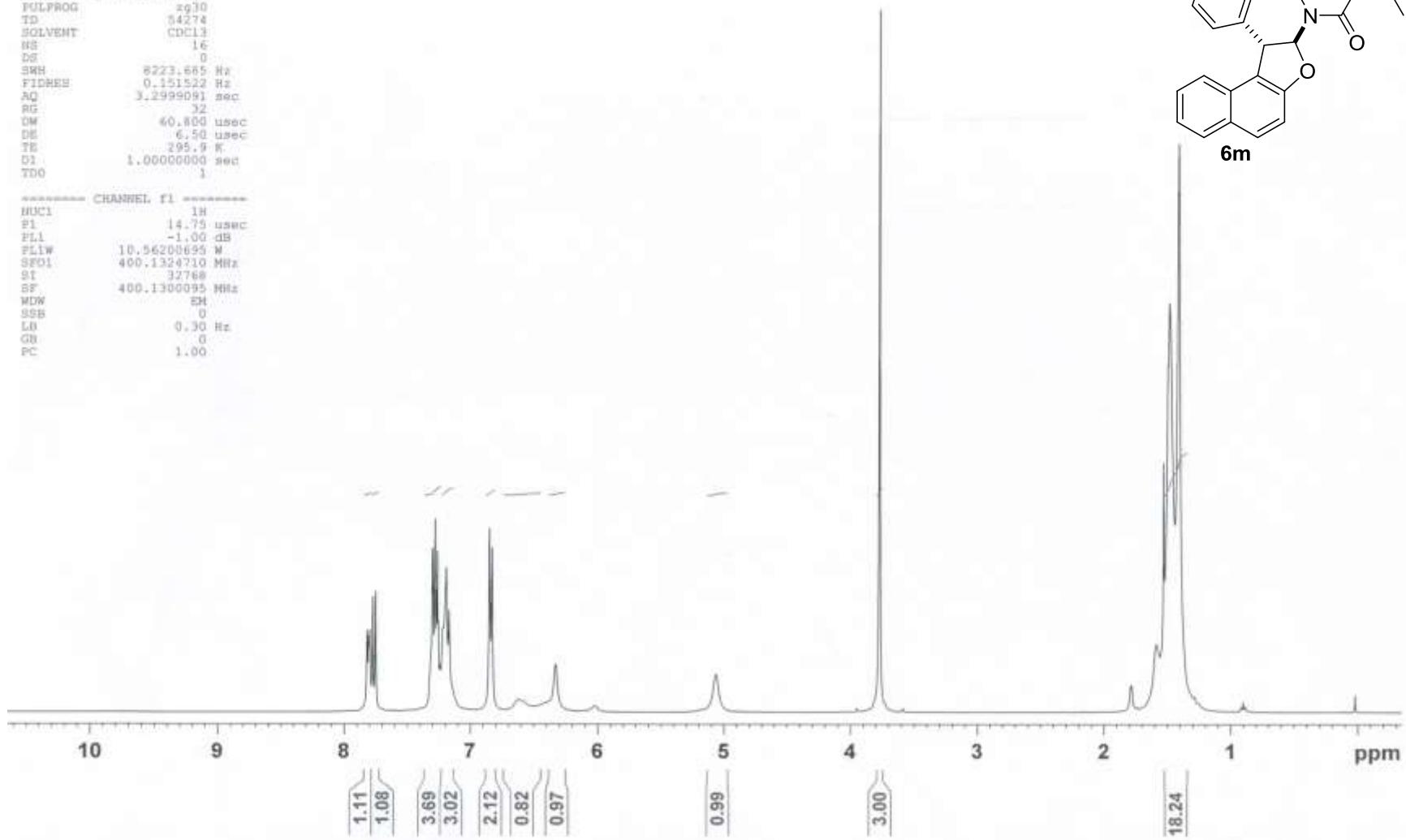
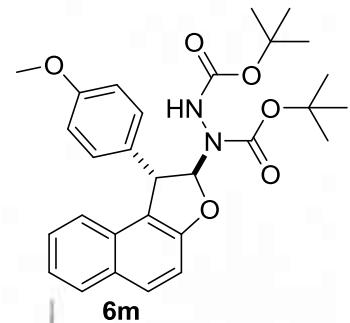


Figure S61.  $^1\text{H}$  NMR Spectrum of **6m**



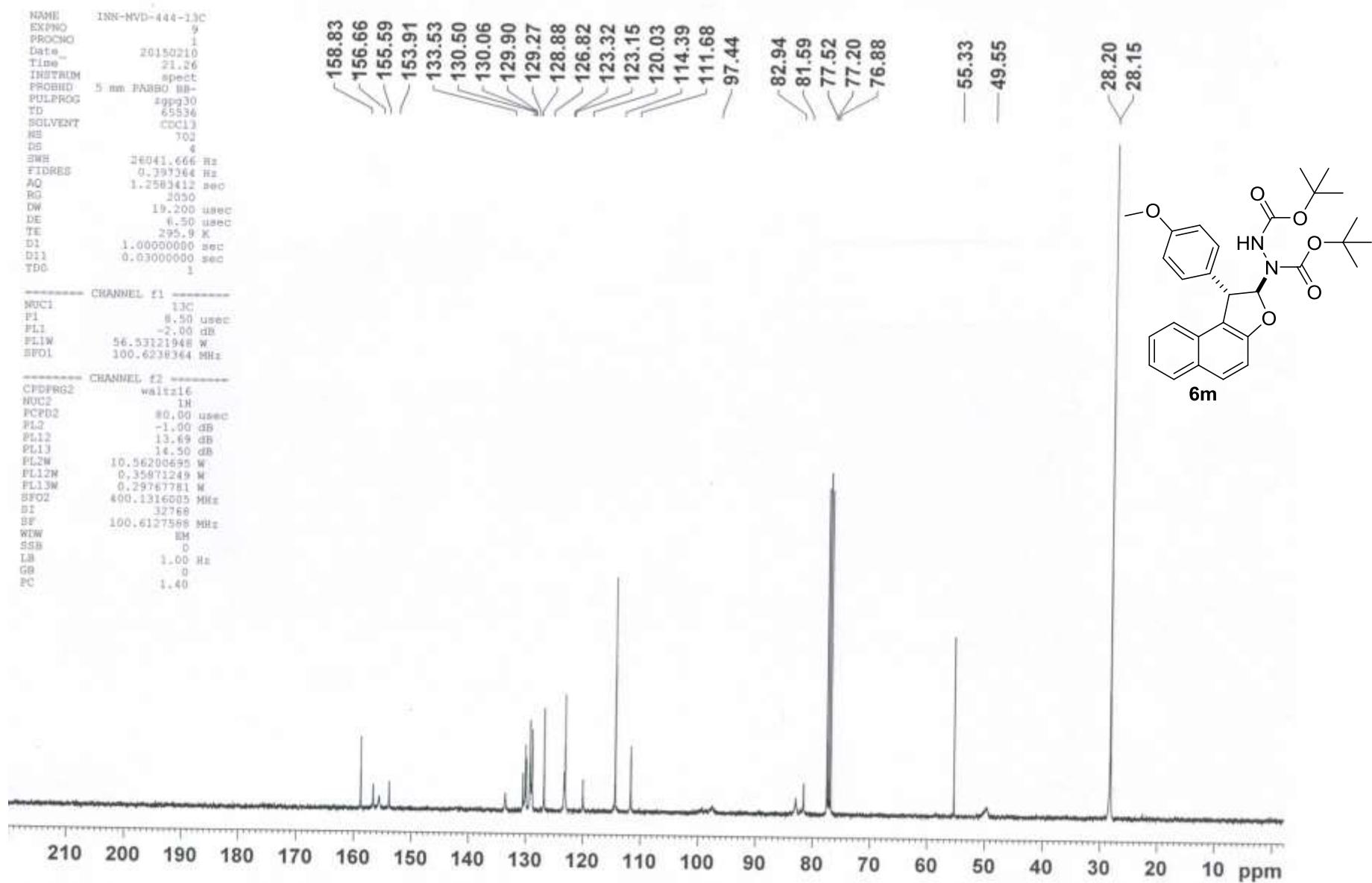


Figure S62.  $^{13}\text{C}$  NMR Spectrum of **6m**

NAME INN-MVD-179-1H  
 EXPNO 1  
 PROCNO 1  
 Date 20140111  
 Time 11.38  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 11  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.1 K  
 D1 1.0000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 6.75 usec  
 PLL -3.00 dB  
 PLLW 16.73969454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300103 MHz  
 WDW RM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

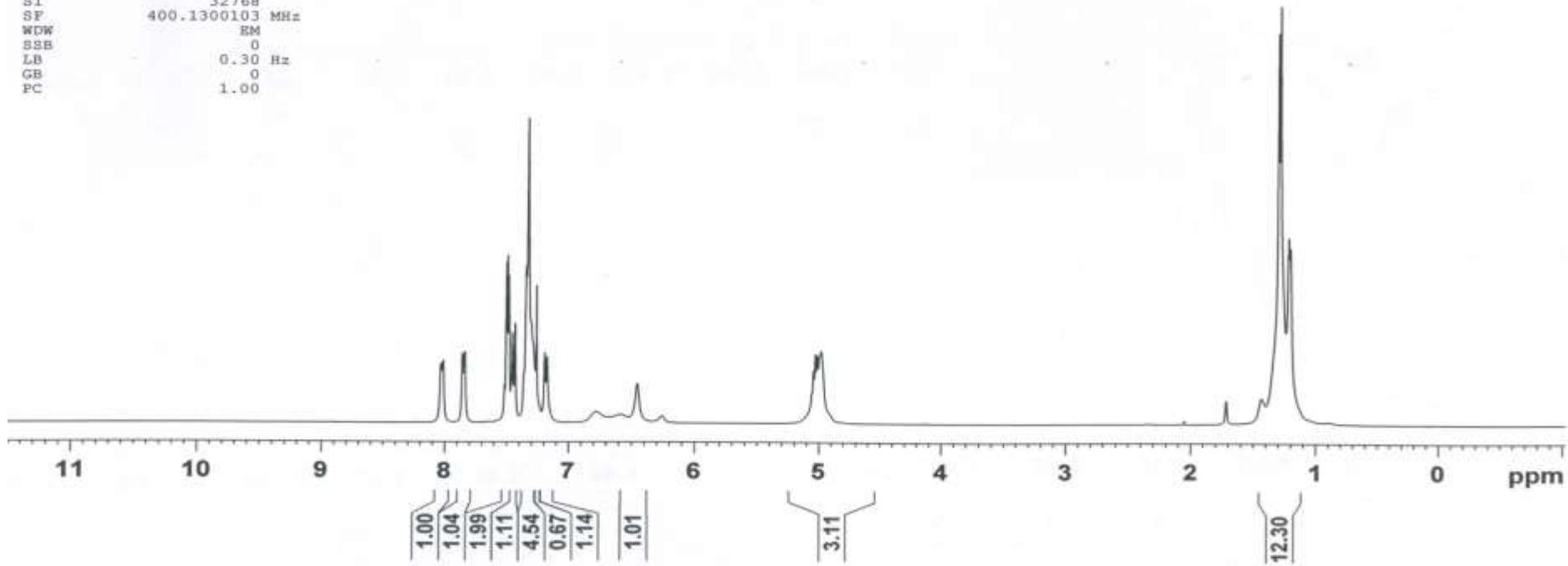
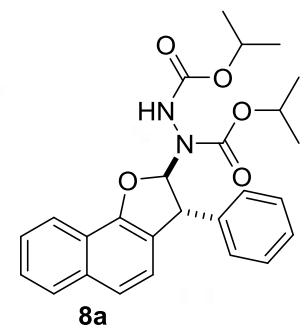
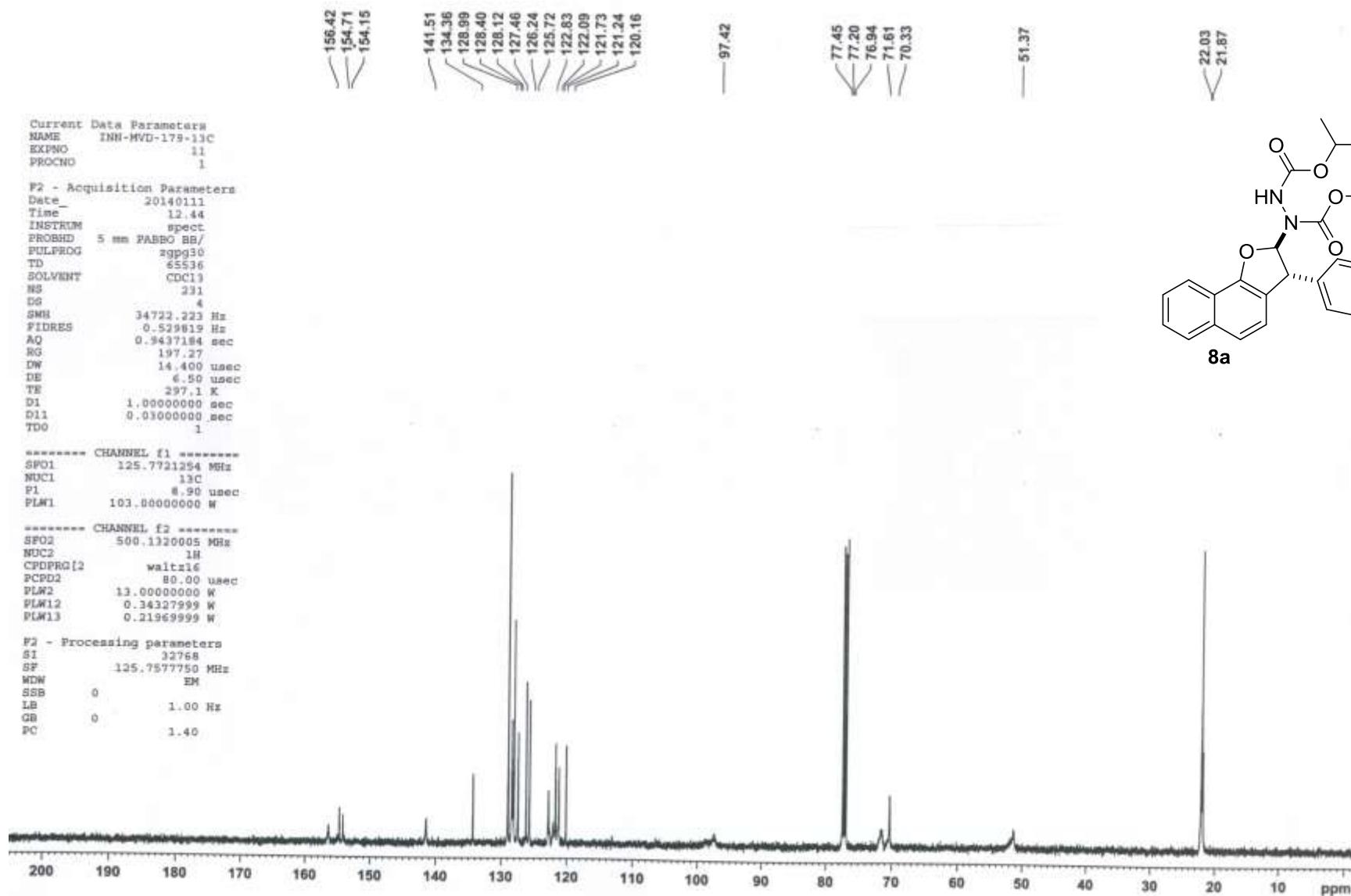


Figure S63.  $^1\text{H}$  NMR Spectrum of **8a**





```

NAME      INN-MVD-154--1H
EXPNO
PROCNO
Date      20140104
Time      0.51
INSTRUM  spect
PROBHD  5 mm SEI 1H/D-
PULPROG  zg30
TD       65536
SOLVENT   CDCl3
NS        14
DS         0
SWH     8223.685 Hz
FIDRES  0.125483 Hz
AQ      3.9846387 sec
RG        90.5
DW       60.800 usec
DE       6.50 usec
TE      296.8 K
D1    1.00000000 sec
TD0             1

```

```

----- CHANNEL f1 -----
NUC1           1H
P1            6.75 usec
PL1          -3.00 dB
P11W      16.73965454 W
SF01      400.1324710 MHz
SI        32768
SF      400.1300102 MHz
WDW           EM
SSB            0
LB          0.30 Hz
GB            0
PC           1.00

```

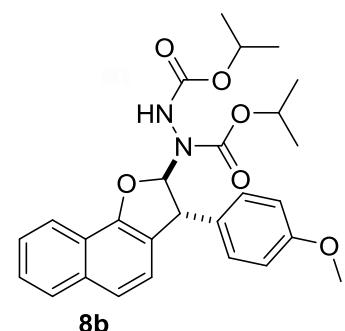
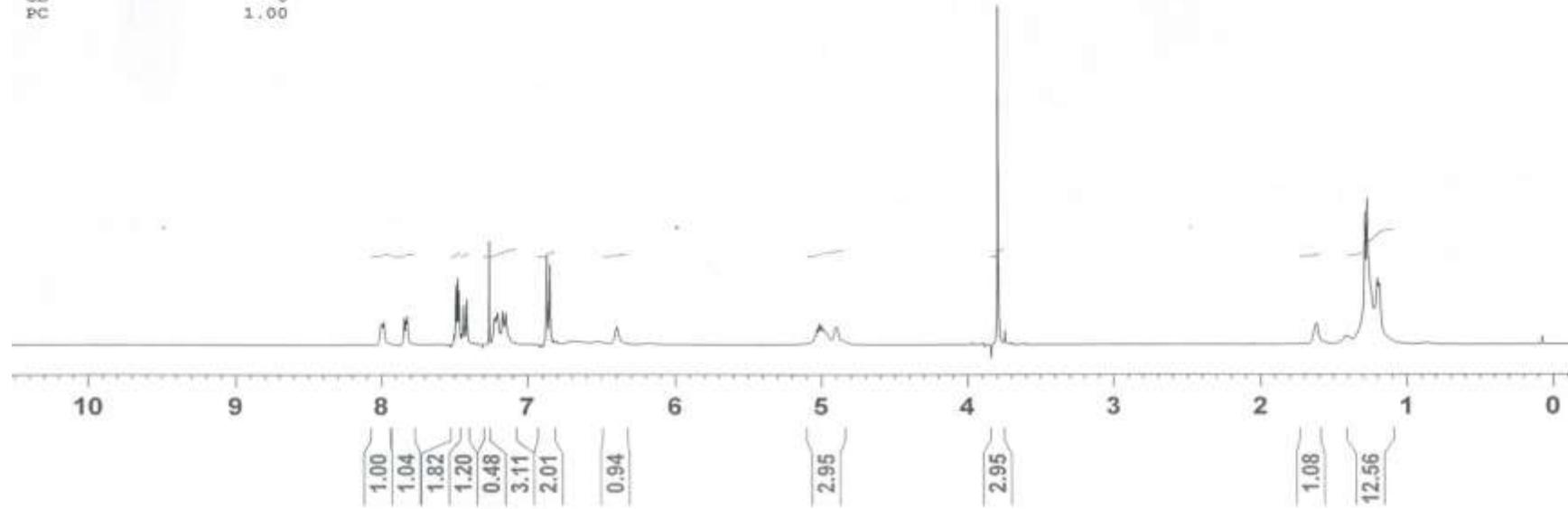
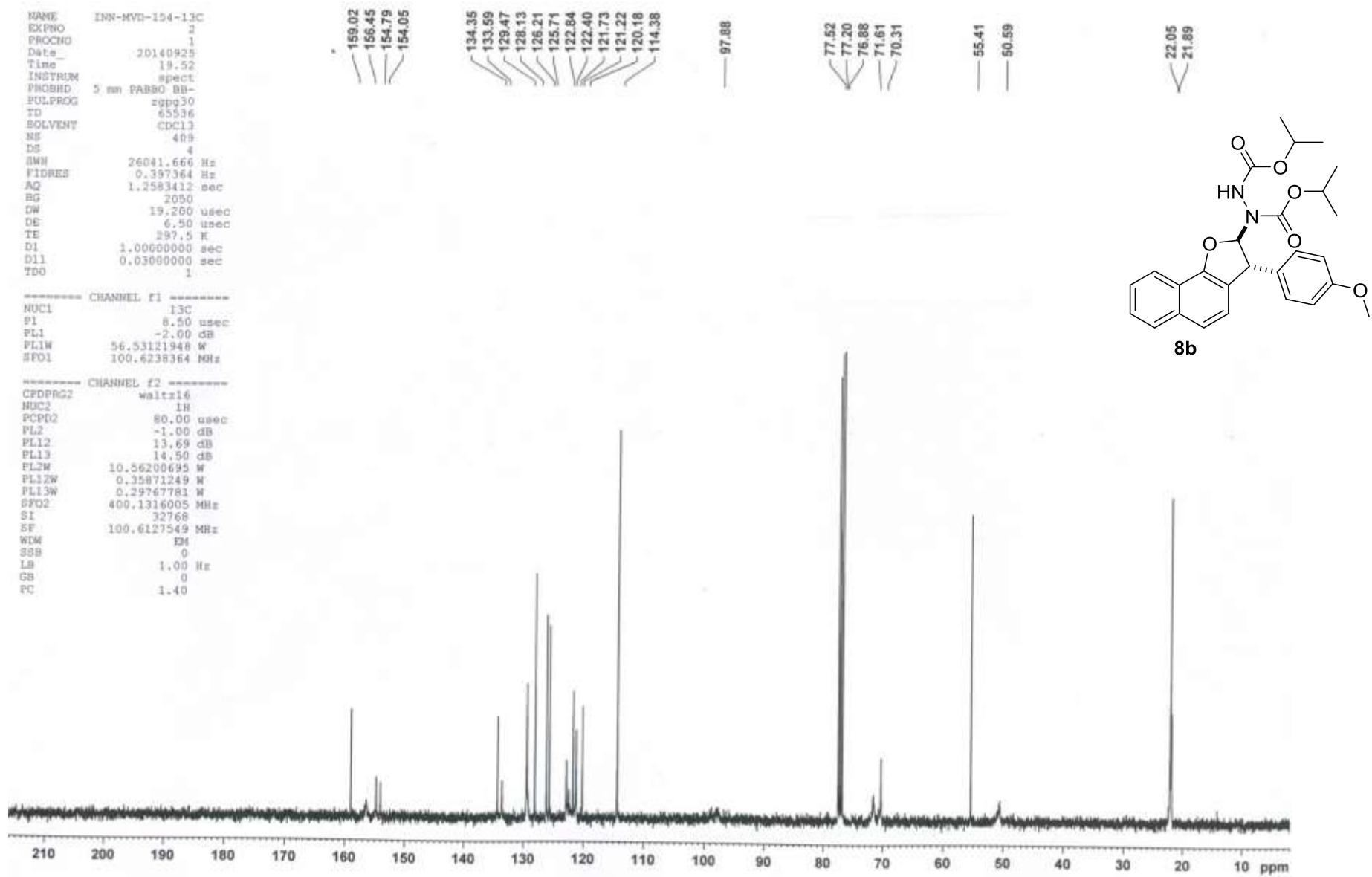


Figure S65.  $^1\text{H}$  NMR Spectrum of **8b**



Current Data Parameters  
NAME INN-MVD-158-1H  
EXPNO 1  
PROCNO 1  
P2 - Acquisition Parameters  
Date 20140921  
Time 16.45  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 19  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 298.2 K  
D1 1.0000000 sec  
TDO 1  
----- CHANNEL f1 -----  
SP01 500.1330885 MHz  
NUC1 1H  
D1 13.00 usec  
PLW1 13.0000000 W  
P2 - Processing parameters  
SI 65536  
SF 500.1300114 MHz  
NDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

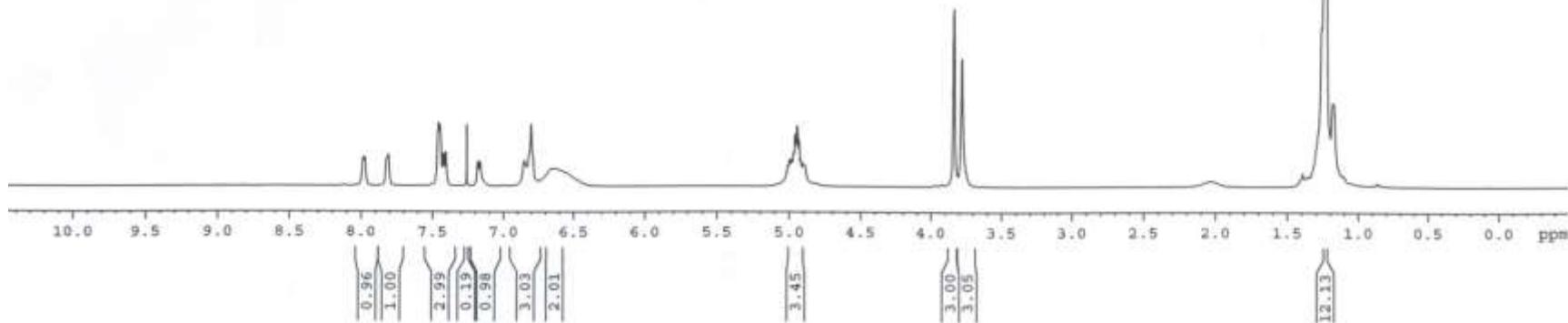
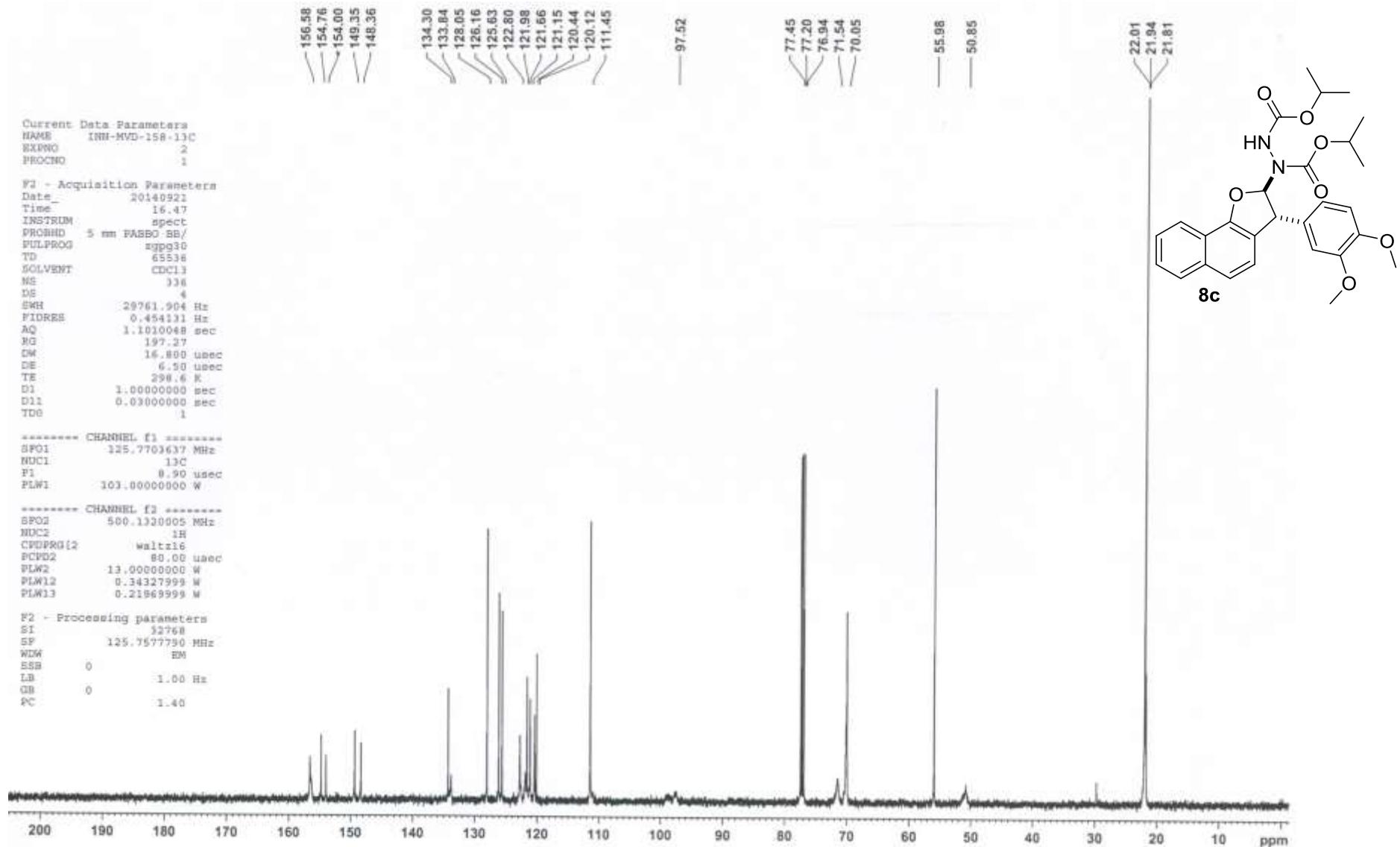


Figure S67. <sup>1</sup>H NMR Spectrum of **8c**



Current Data Parameters  
 NAME INN-MVD-180-1H  
 EXPNO 1  
 PROCMO 1  
 F2 - Acquisition Parameters  
 Date 20140923  
 Time 13.21  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 15  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767955 sec  
 RG 30.72  
 DM 50.000 usec  
 DE 6.50 usec  
 TS 296.1 K  
 D1 1.0000000 sec  
 TDO 1

\*\*\*\*\* CHANNEL F1 \*\*\*\*\*  
 SP01 500.1330885 MHz  
 NUC1 1H  
 PI 13.00 usec  
 PLW1 13.0000000 W

F2 - Processing parameters  
 SI 65536  
 SF 500.1300123 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

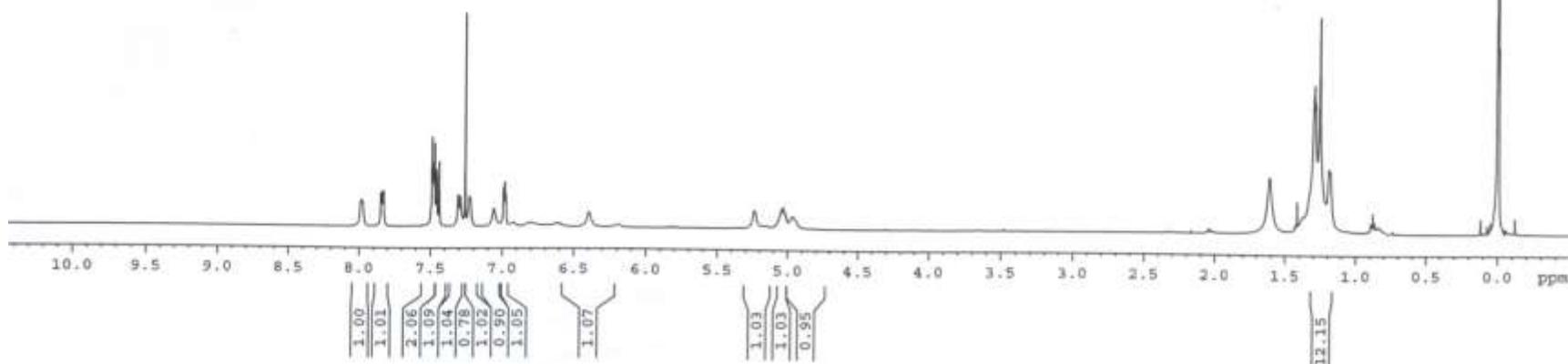
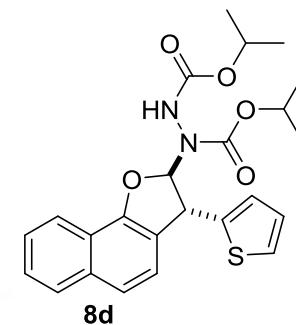


Figure S69.  $^1\text{H}$  NMR Spectrum of **8d**

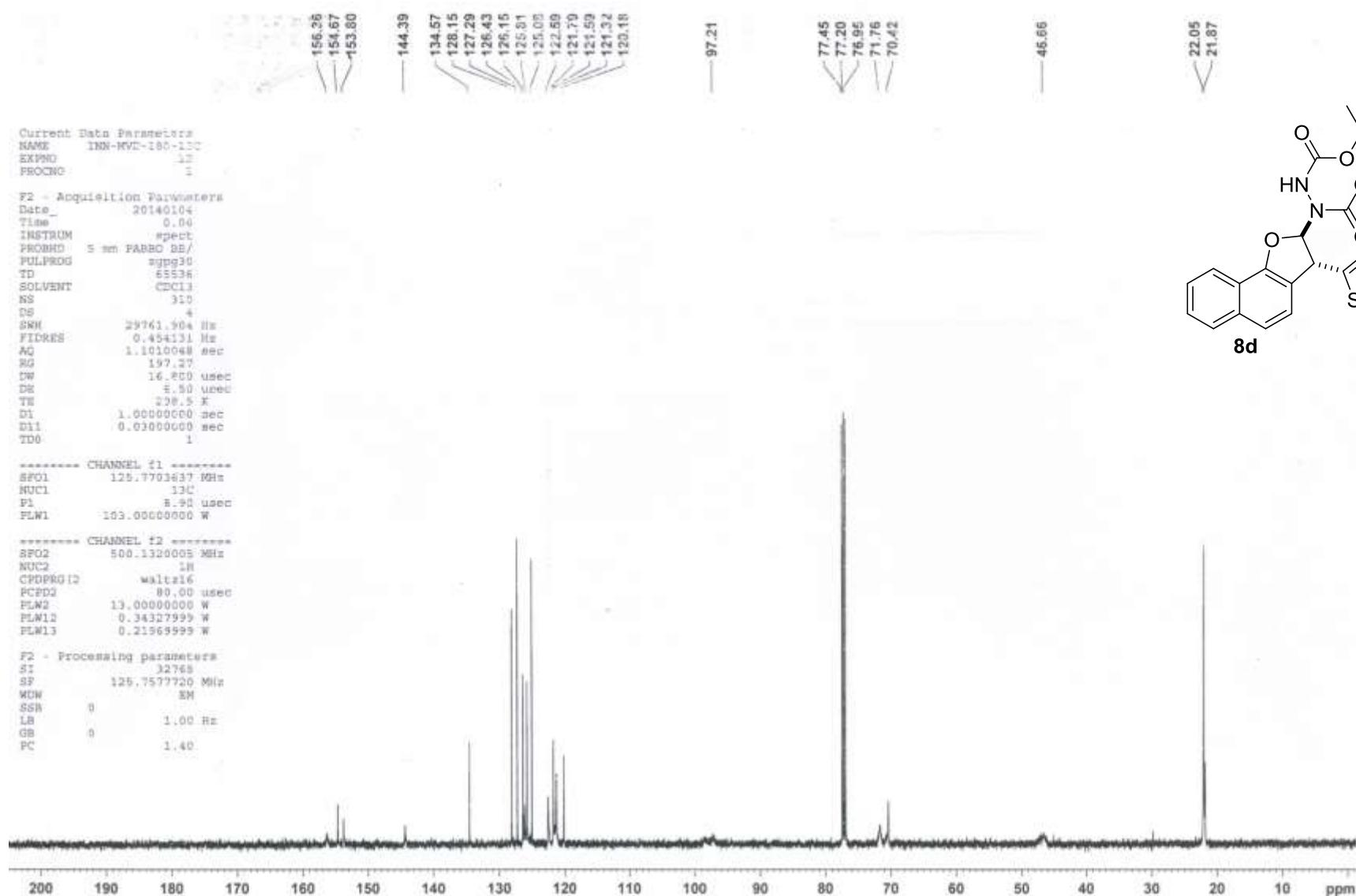


Figure S70. <sup>13</sup>C NMR Spectrum of **8d**

```

NAME      INN-MVD-161-1H
EXPNO             7
PROCNO            1
Date     20141014
Time     18.51
INSTRUM spect
PROBHD  5 mm PABBO BB-
PULPROG zg30
TD      54274
SOLVENT   CDCl3
NS           30
DS            0
SWH       8223.685 Hz
FIDRES   0.151522 Hz
AQ        3.2999991 sec
RG          32
DW       60.000 usec
DE        6.50 usec
TE       297.3 K
D1      1.0000000 sec
TDO          1

```

```

----- CHANNEL f1 -----
NUC1           1H
P1        14.75 usec
PL1        -1.00 dB
PL1W    10.56200695 W
SF01    400.1324710 MHz
SI          32768
SF      400.1300172 MHz
WDW           EM
SSB           0
LB        0.30 Hz
GB           0
PC          1.00

```

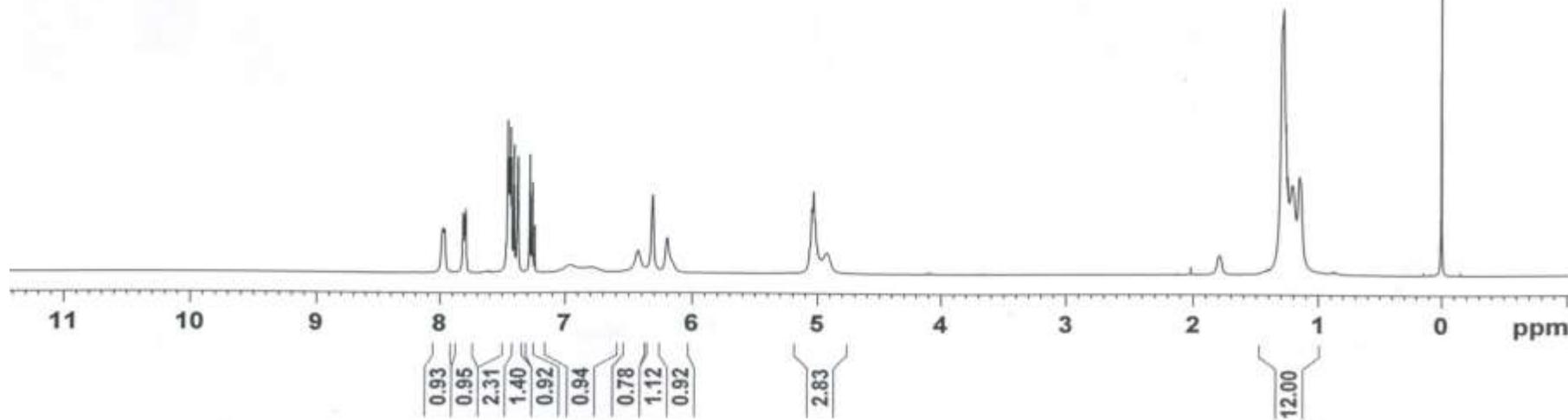
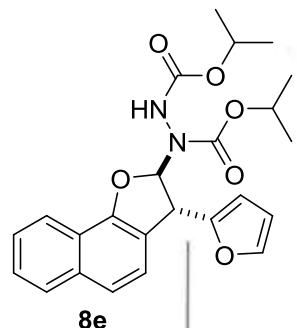


Figure S71.  $^1\text{H}$  NMR Spectrum of **8e**

NAME: ISBN-MVD-181-13C  
 EXPNO: 1  
 PROCN0:  
 Date: 20141014  
 Time: 19.19  
 INSTRUM: spect  
 PROBHD: 5 mm PARBO BB-  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 1025  
 DS: 4  
 SWH: 26041.666 Hz  
 FIDRES: 0.397364 Hz  
 AQ: 1.2583412 sec  
 RG: 2050  
 DW: 19.200 usec  
 DE: 6.50 usec  
 TE: 298.0 K  
 D1: 1.0000000 sec  
 D11: 0.03000000 sec  
 TDD: 1

----- CHANNEL f1 -----

HUC1: 13C  
 P1: 8.50 usec  
 PL1: -2.00 dB  
 PL1W: 56.53121948 W  
 SFQ1: 100.6238364 MHz

----- CHANNEL f2 -----

CPDPRG2: waltz16  
 HUC2: 1H  
 PCPD2: 80.00 usec  
 PL2: -1.00 dB  
 PL12: 13.69 dB  
 PL13: 14.50 dB  
 PL2W: 10.56200695 W  
 PL12W: 0.35871249 W  
 PL13W: 0.29767781 W  
 SFQ2: 400.1316005 MHz  
 SI: 32768  
 SF: 100.6127544 MHz  
 WDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 FC: 1.40

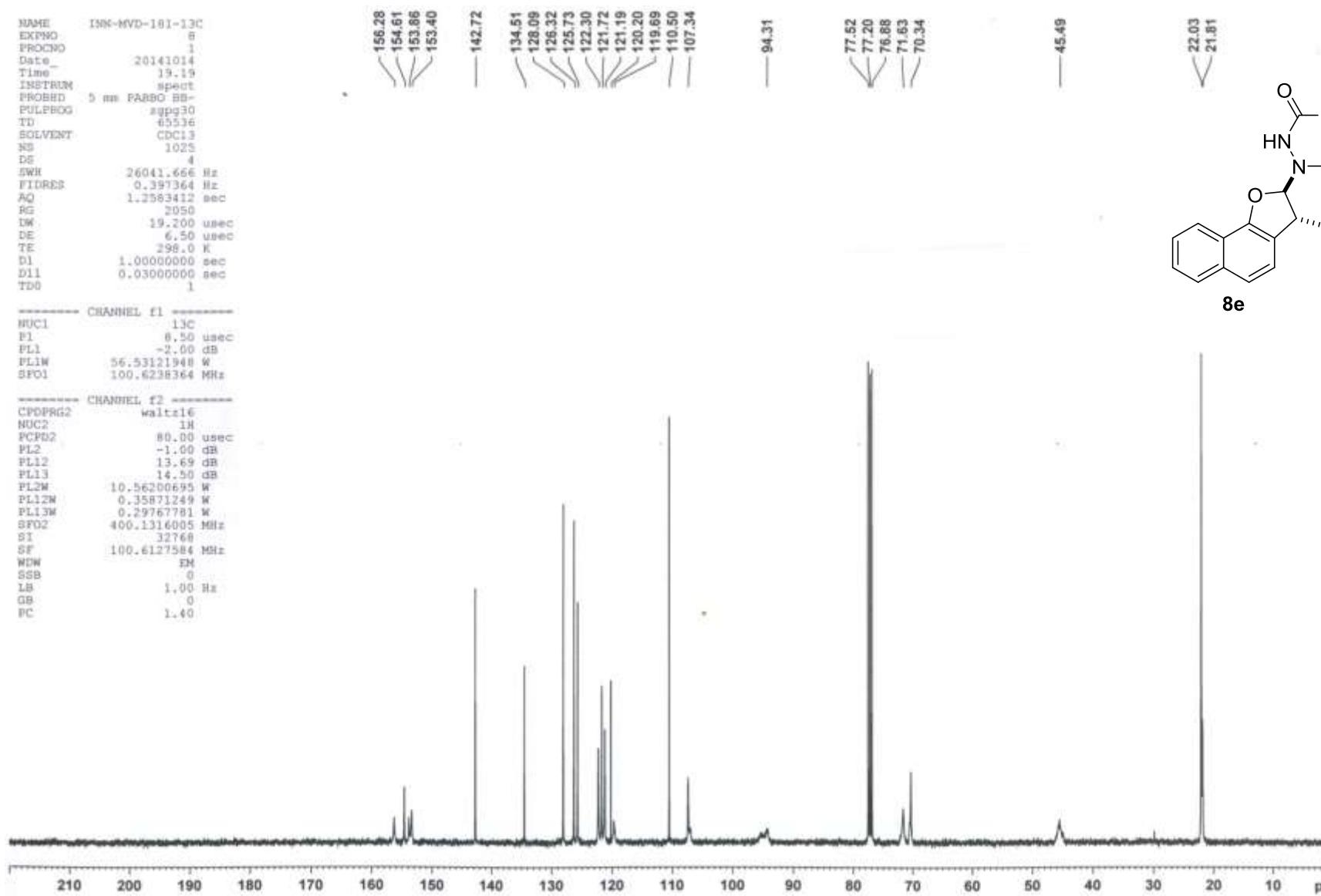


Figure S72.  $^{13}\text{C}$  NMR Spectrum of **8e**

Current Data Parameters  
NAME: IHN-MVD-1H2-1H  
EXPNO: 1  
PROCNO: 1  
  
F2 - Acquisition Parameters  
Date: 20141014  
Time: 19:08  
INSTRUM: spect  
PROBHD: 5 mm PABBO BB/  
PULPROG: zg30  
TD: 65536  
SOLVENT: CDCl3  
NS: 25  
DS: 2  
SWH: 10000.000 Hz  
FIDRES: 0.152598 Hz  
AQ: 3.2767999 sec  
RG: 30.72  
DW: 50.000 usec  
DE: 6.50 usec  
TE: 297.7 K  
D1: 1.0000000 sec  
TDC: 1  
  
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
SFO1: 500.1330085 MHz  
NUC1: 1H  
PI: 13.00 usec  
PL1: 13.0000000 M  
  
F2 - Processing parameters  
ST: 65536  
SF: 500.1330017 MHz  
WDW: EM  
SSB: 0  
LB: 0.10 Hz  
GB: 0  
PC: 1.00

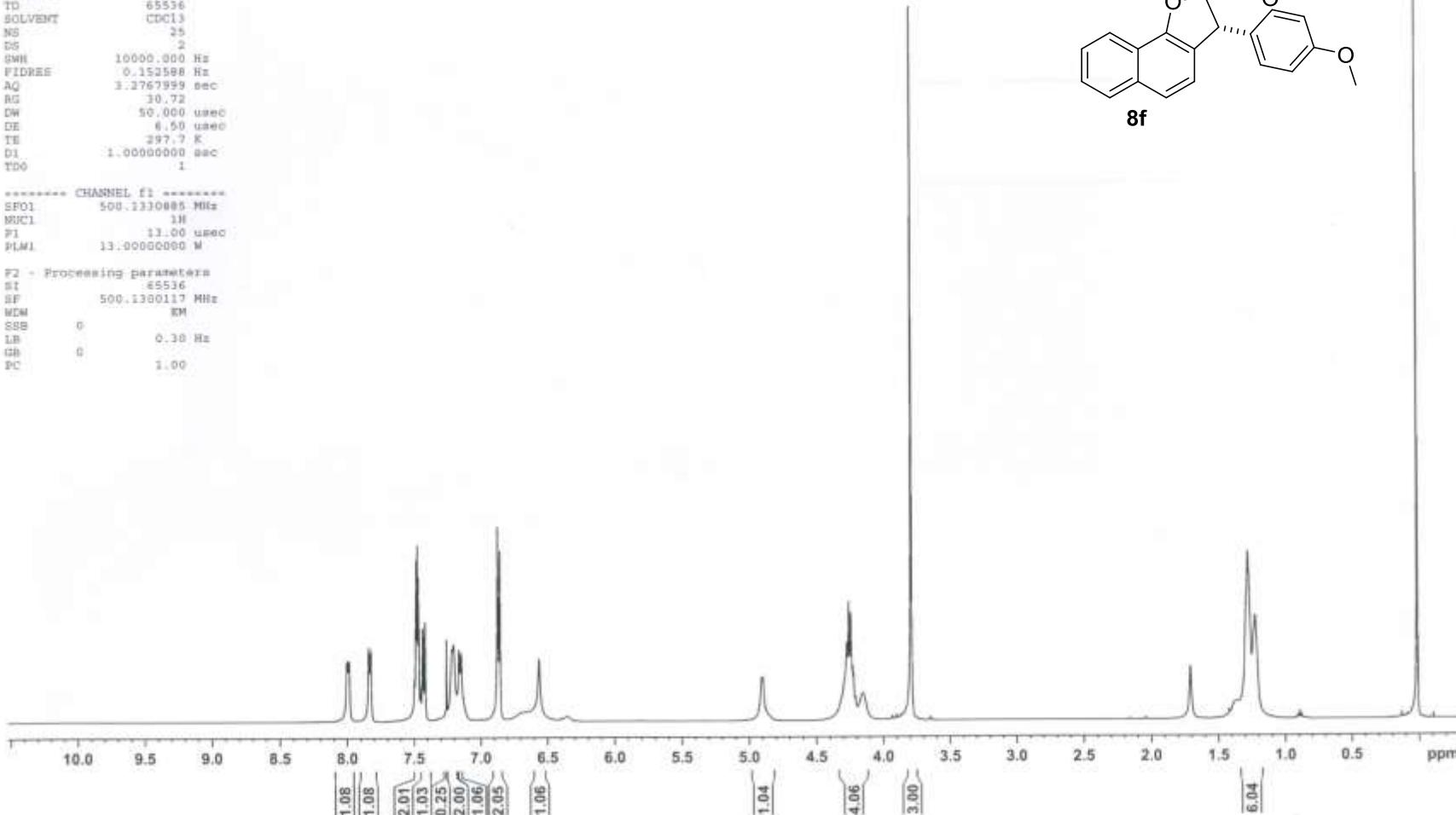


Figure S73.  $^1\text{H}$  NMR Spectrum of **8f**

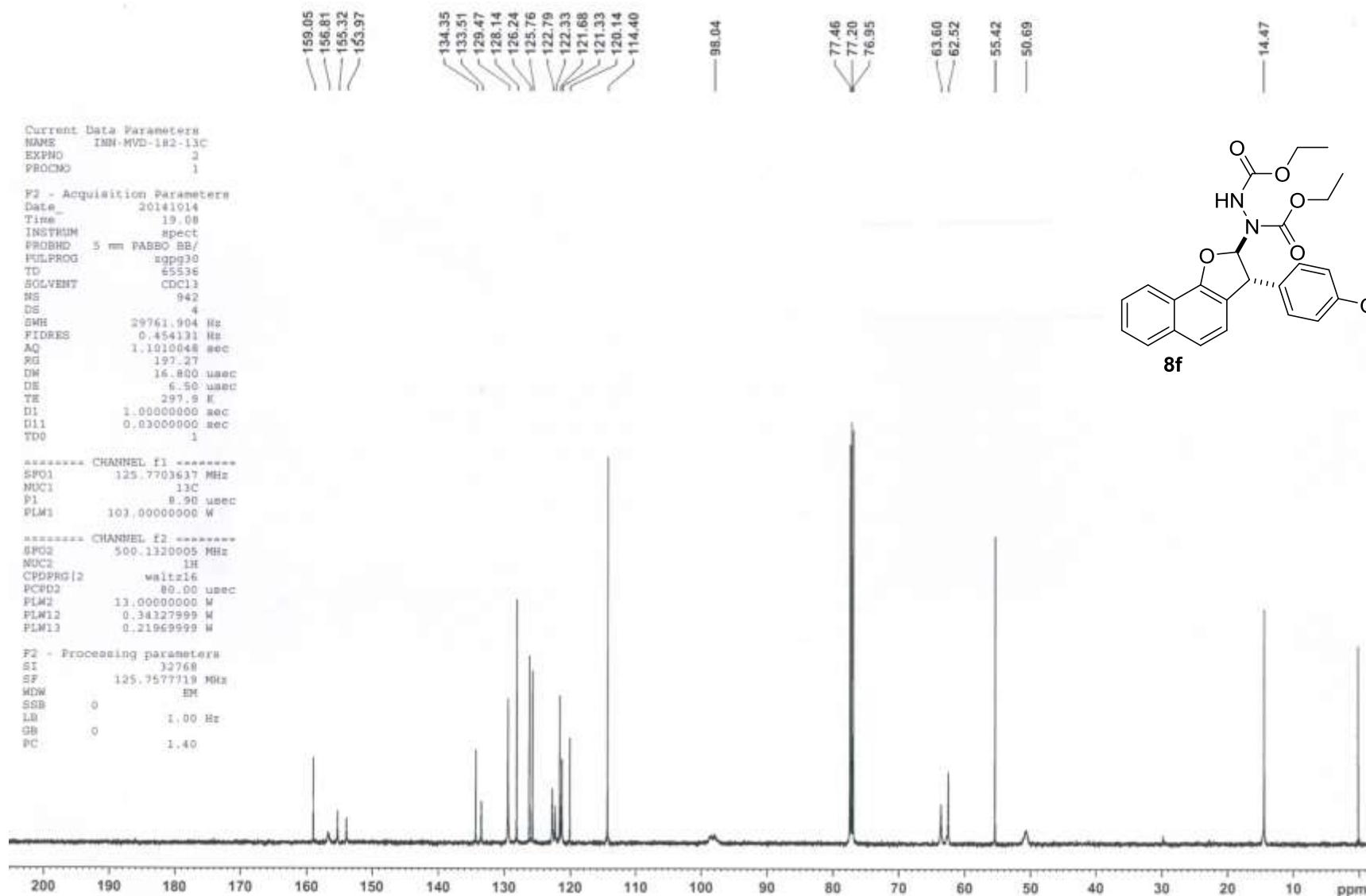


Figure S74. <sup>13</sup>C NMR Spectrum of **8f**

NAME: INN-MVD-173-1H  
 EXPNO: 4  
 PROCHNO: 1  
 Date: 20150305  
 Time: 19.07  
 INSTRUM: spect  
 PROBHD: 5 mm PABBO BB-  
 PULPROG: zg30  
 T1: 54274  
 SOLVENT: CDCl3  
 NS: 30  
 DS: 0  
 SWH: 8223.685 Hz  
 FIDRES: 0.151522 Hz  
 AQ: 3.2999091 sec  
 RG: 32  
 DW: 60.800 usec  
 DE: 6.50 usec  
 TE: 295.7 K  
 D1: 1.0000000 sec  
 TDO: 4

===== CHANNEL F1 =====  
 NUC1: 1H  
 PI: 14.75 usec  
 PL1: -1.00 dB  
 PL1W: 10.56200695 W  
 SP01: 400.1324710 MHz  
 SI: 32768  
 SF: 400.1300097 MHz  
 MDW: 0.00  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

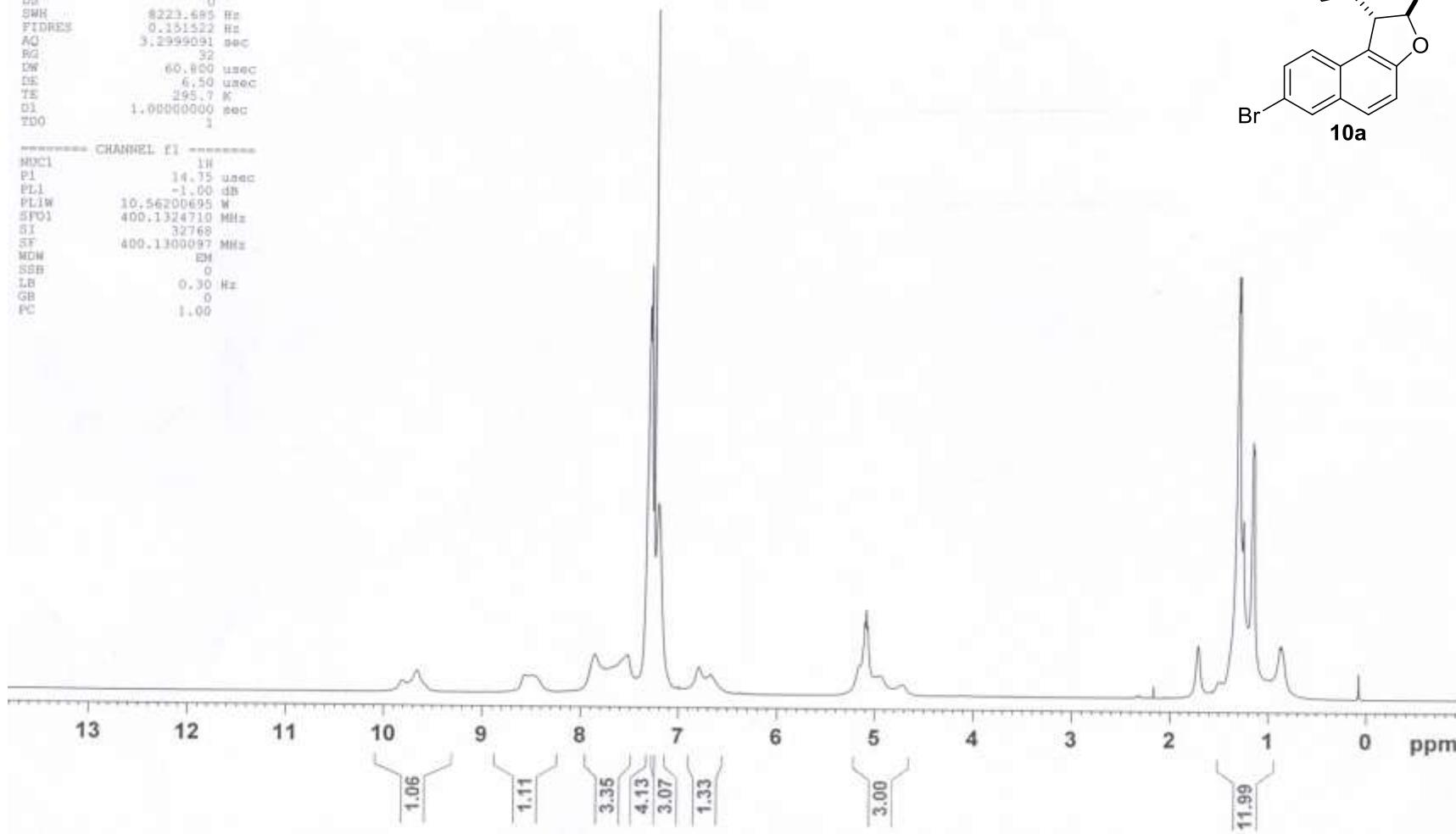
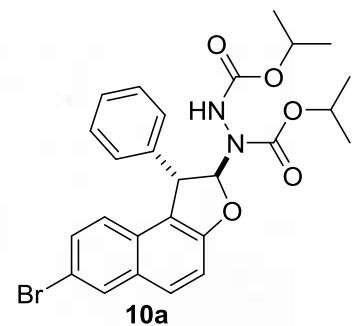
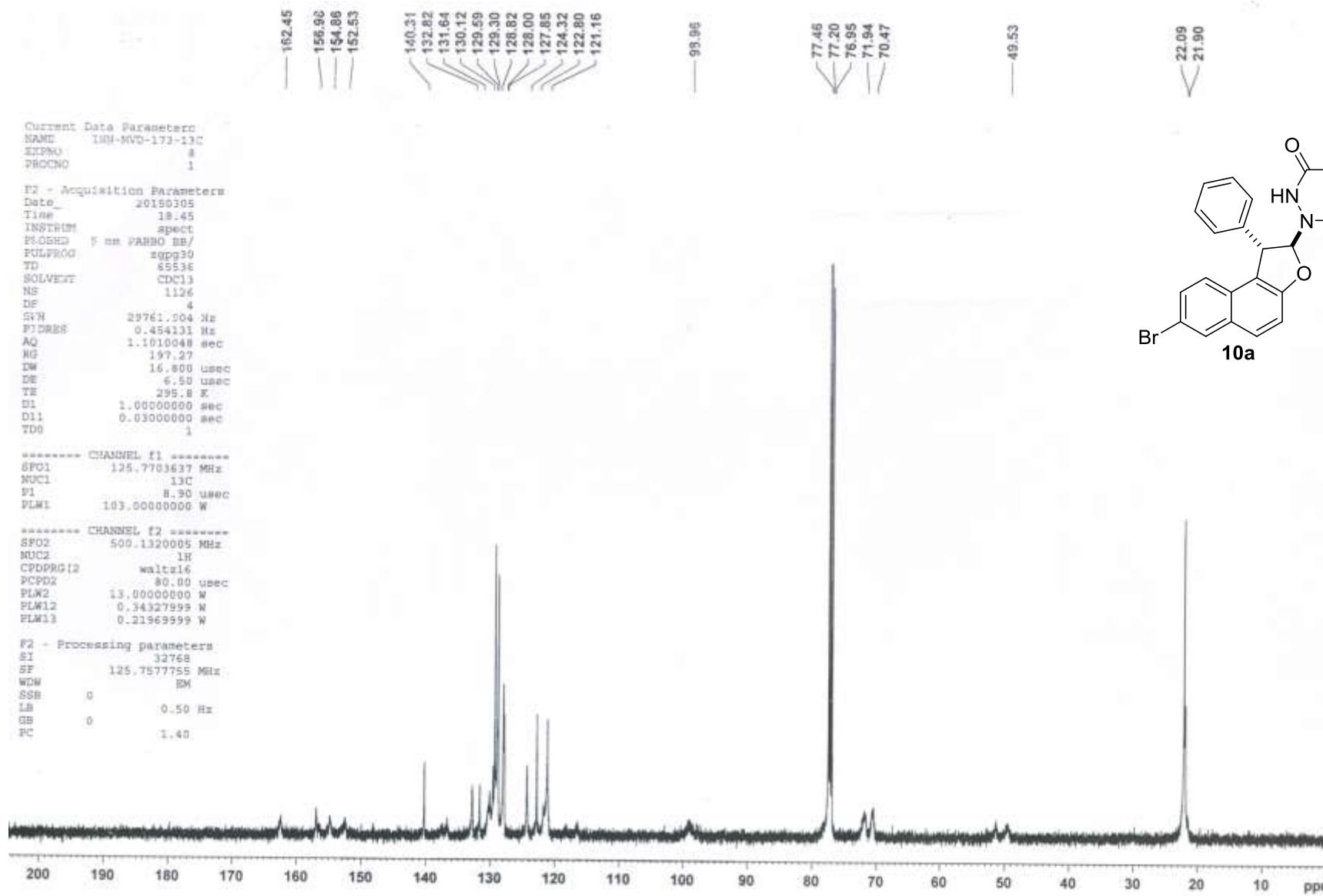


Figure S75.  $^1\text{H}$  NMR Spectrum of **10a**





Current Data Parameters  
NAME INN-MVD-172-1H  
EXPNO 4  
PROCNO 1  
P2 - Acquisition Parameters  
Date 20150307  
Time 16.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 40  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 294.1 K  
D1 1.0000000 sec  
TDO 1

----- CHANNEL f1 -----  
SP01 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

P2 - Processing parameters  
SI 65536  
SF 500.1300122 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

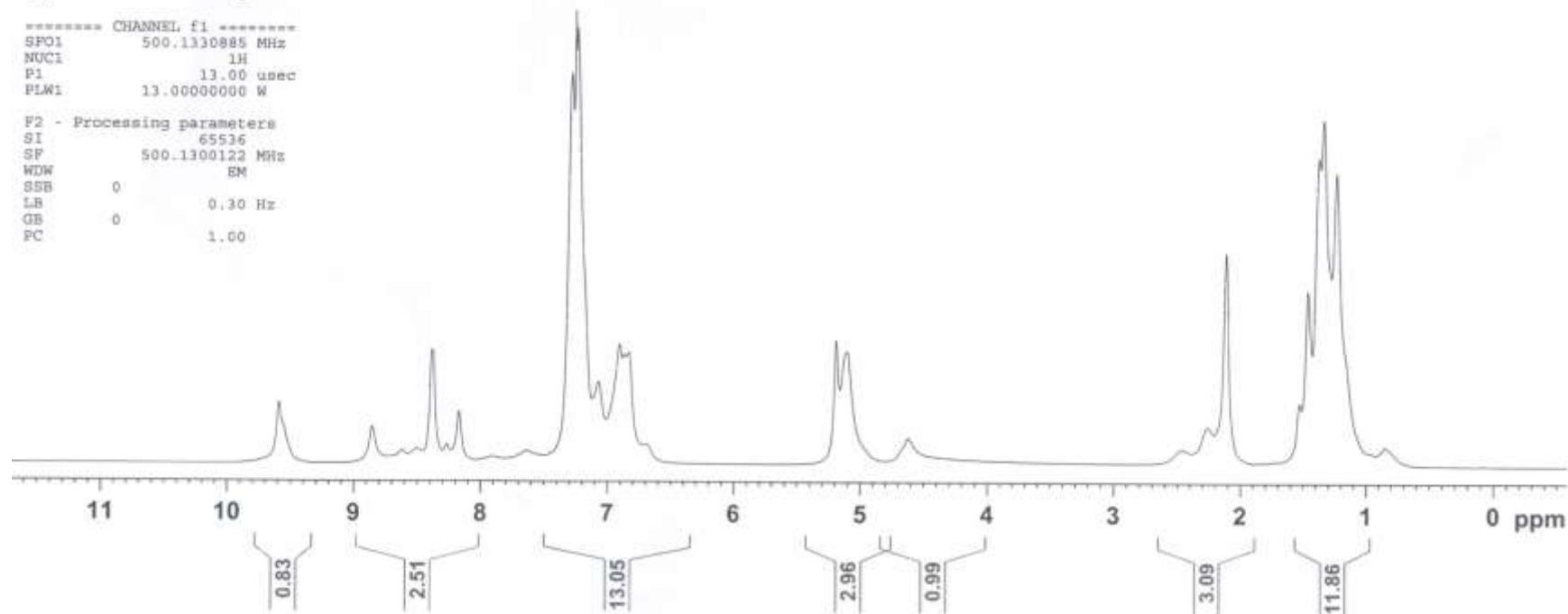
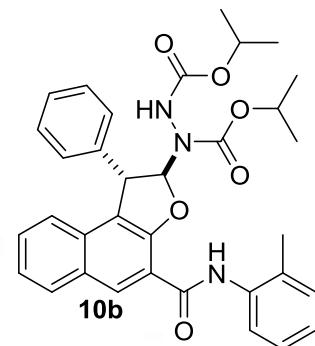


Figure S77.  $^1\text{H}$  NMR Spectrum of **10b**



Current Data Parameters  
NAME INX-MWD-172-188223K  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date 20140910  
Time 15.39  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 50  
DS 2  
SWH 11029.412 Hz  
FIDRES 0.168295 Hz  
AQ 2.9709654 sec  
RG 53.37  
DW 45.333 usec  
DE 6.50 usec  
TB 323.0 K  
D1 1.0000000 sec  
TDO 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
SP01 500.1340010 MHz  
NUC1 1H  
PI 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300211 MHz  
WDW ZR  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

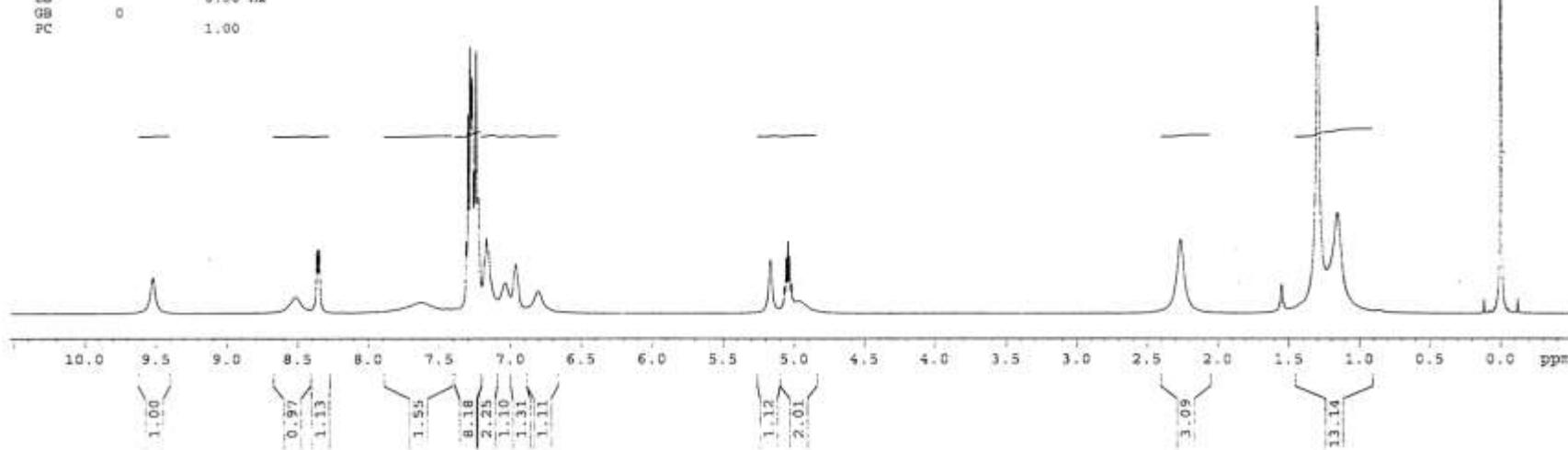
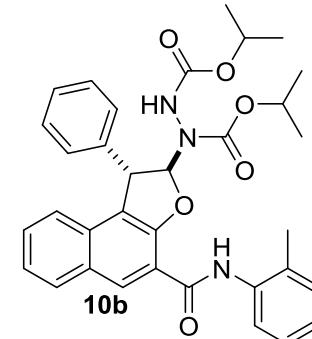


Figure S78.  $^1\text{H}$  NMR Spectrum of **10b** at 323 k

Current Data Parameters  
 NAME INN-MVD-272-1H#333K  
 EXPNO 12  
 PROCN 1  
 P2 - Acquisition Parameters  
 Date 2014001  
 Time 17.23  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 20  
 DS 0  
 SWH 11029.412 Hz  
 FIDRES 0.168295 Hz  
 AQ 2.9709654 sec  
 RG 48.36  
 DW 45.333 usec  
 DS 6.50 usec  
 TZ 333.0 K  
 SL 1.0000000 sec  
 TDO 1  
 ----- CHANNEL f1 -----  
 SFO1 500.1335009 MHz  
 NUC1 1H  
 P1 13.00 usec  
 PLWI 13.0000000 W  
 P2 Processing parameters  
 ST 65536  
 SF 500.1300039 MHz  
 MW1 EM  
 SSB 0  
 TB 0.30 Hz  
 GB 0  
 PC 1.00

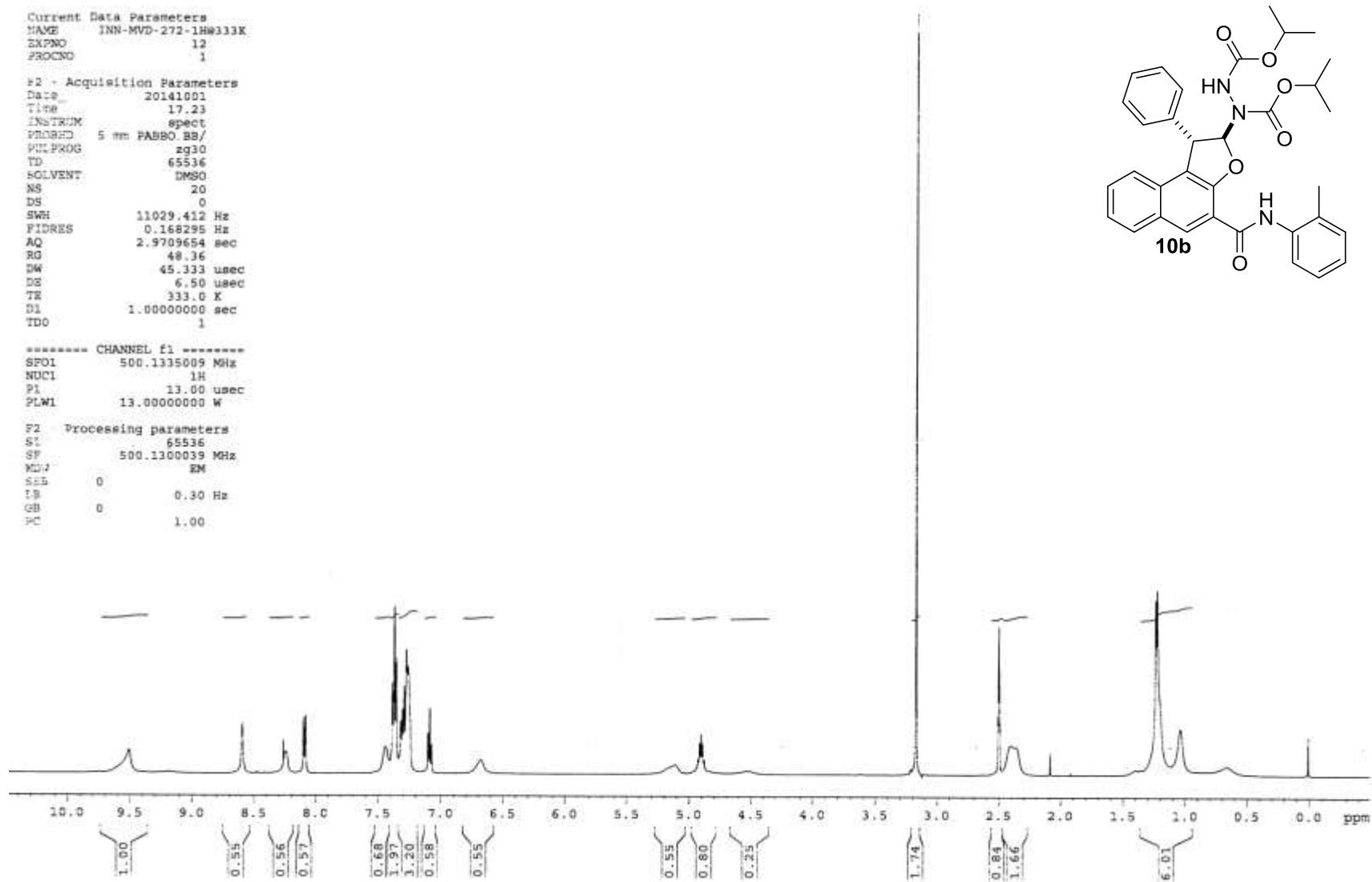


Figure S79. <sup>1</sup>H NMR Spectrum of **10b** at 333 k

```

NAME    IIRN-MVD-172-13C
EXPMO   1
PROCSO   1
Date_   20131120
Time_   10.00
INSTRUM spect
PROBHD  5 mm SEI IN/DB
PULPROG zgpg30
TD      65536
SOLVENT CDC13
NS      3295
DS      4
SW0    24036.461 Hz
FIDRES 0.366798 Hz
AQ     1.3631988 sec
RG      114
DM      20,800 usec
DE      6.50 usec
TE      297.4 K
D1      1.0000000 sec
S11     0.0300000 sec
TBO     t

```

```

----- CHANNEL f1 -----
NUC1          13C
FI            19.00 usec
PLL           -3.00 dB
PL1W          71.16858673 W
SF01          100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2      waitx16
NUC2          IH
PCPD2         80.00 usec
PL2           -3.00 dB
PL1Z          18.48 dB
PL13          19.00 dB
PL2W          16.73965454 W
PL12M         0.11905469 W
PL13M         0.10562007 W
SF02          400.1316005 MHz
S1            32768
SF            100.6127620 MHz
MDW           EH
SSB           0
LB            2.00 Hz
GB           0
PC            1.40

```

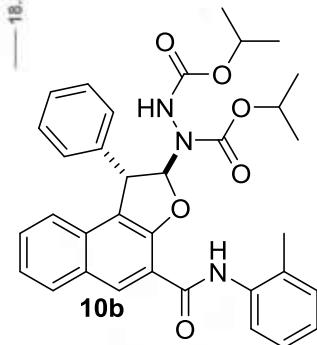
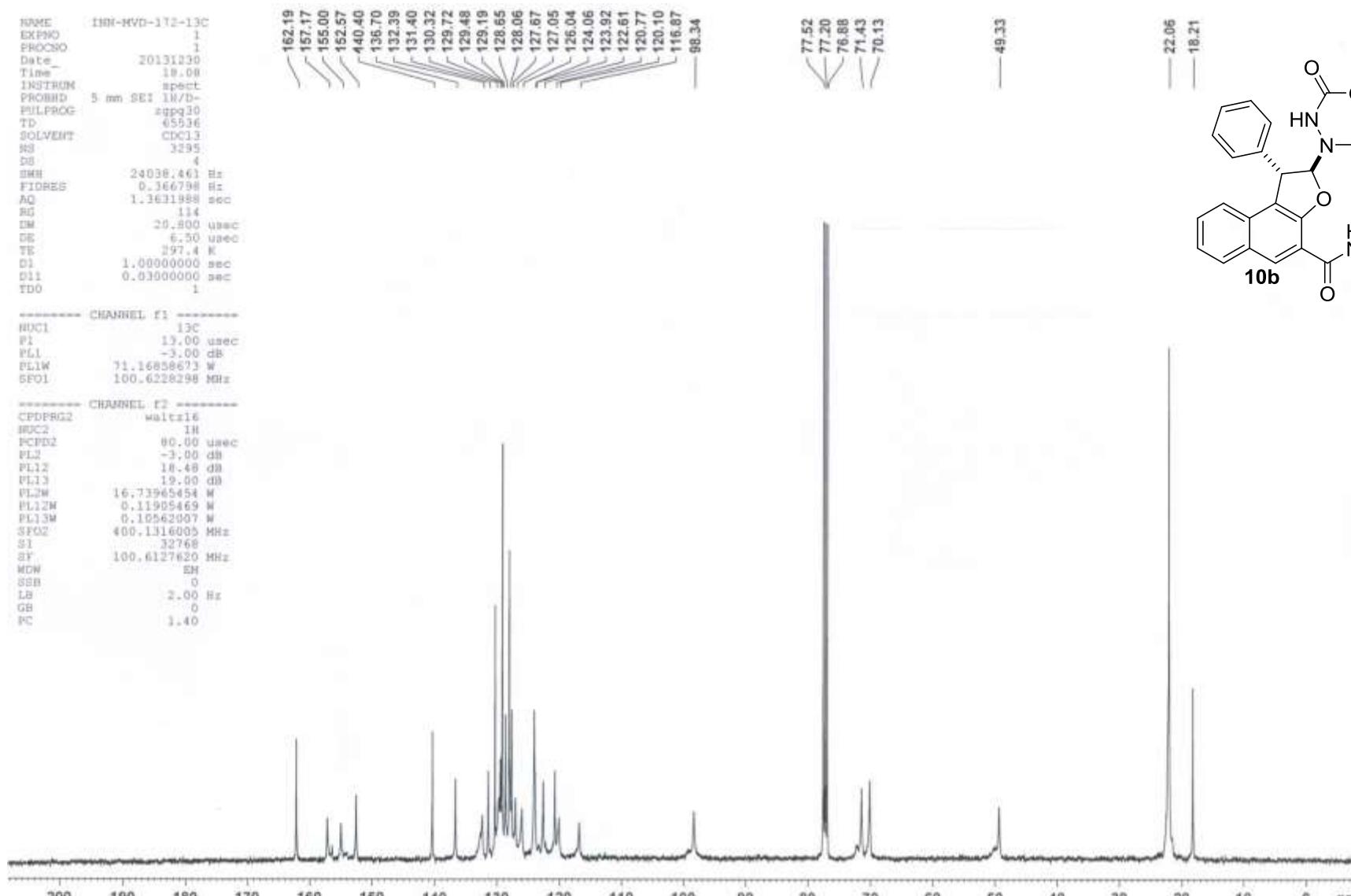


Figure S80.  $^{13}\text{C}$  NMR Spectrum of **10b**

```

Current Data Parameters
NAME    INN-NWD-272-13Cq333
EXPNO          13
PROCNO         1

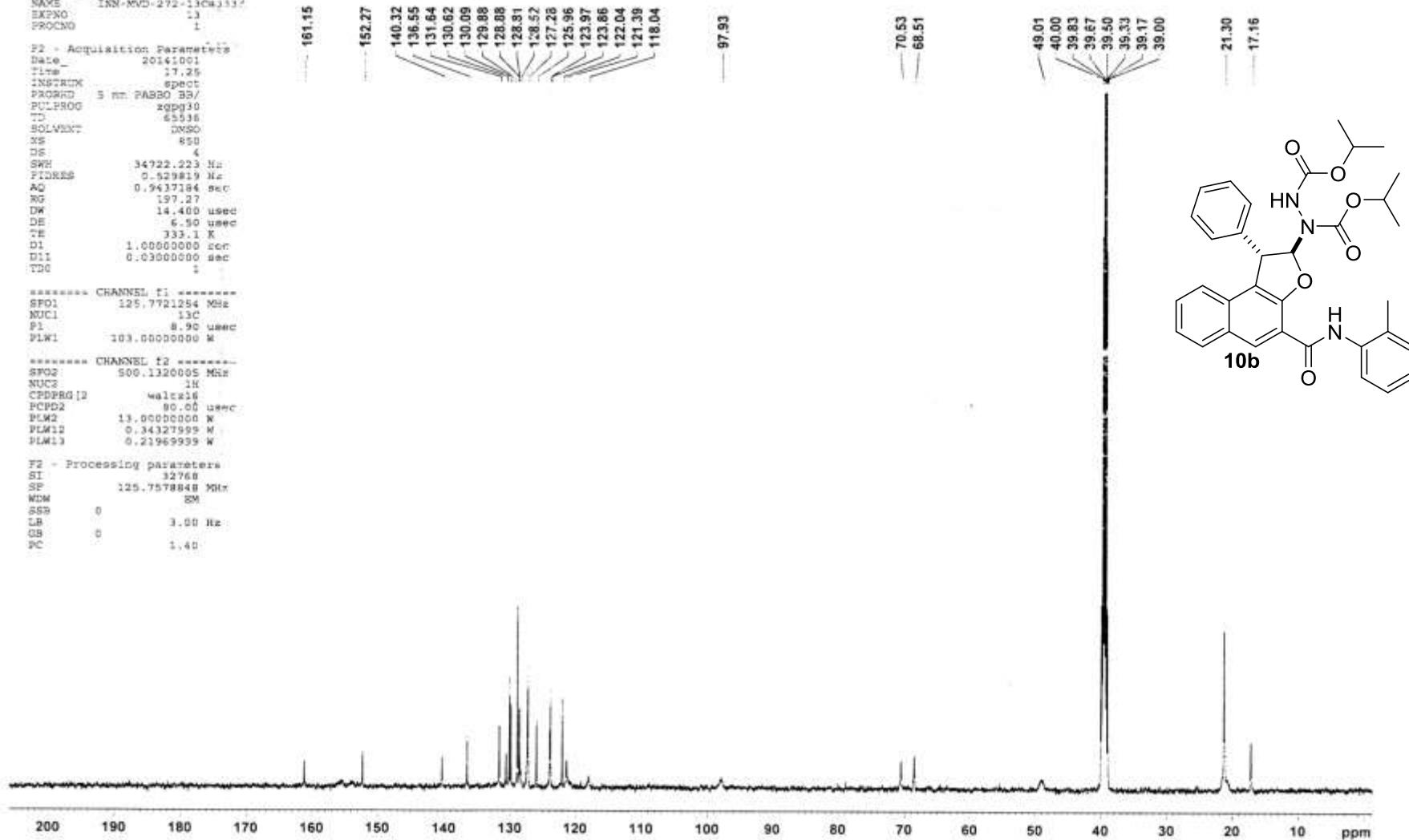
P2 - Acquisition Parameters
Date_   20140101
Time_   17.25
INSTRUM spect
PROBHD  3 mm: PABBO BB/
PULPROG zgpp30
TD      65536
TCDR   1.0000
SOLVENT   DMSO
NS      850
DS       4
SWH     34722.223 Hz
RFINIT  0.529819 Hz
AQ      0.9437184 sec
RG      197.27
DW      14.400 used
DE      6.50 used
TE      333.1 K
DI      1.0000000 sec
D11     0.03000000 sec
TDCS

***** CHANNEL f1 *****
SF01  125.7721254 MHz
NUC1   13C
P1     8.90 usec
PLW1  103.0000000 M

***** CHANNEL f2 *****
SF02  500.1320005 MHz
NUC2   1H
CPDPG1[2]  waltz16
CPDPG2  80.00 usec
PLW2   13.0000000 M
PLW12  0.34327599 M
PLW13  0.21969339 M

F2 - Processing parameters
SI      32768
SP      125.7578848 MHz
WDW    EM
SSB     0
LB      3.00 Hz
GB      0
PC      1.40

```



```

Current Data Parameters
NAME      inn-mvd-147-1h
EXPNO        2
PROCNO        1

P2 - Acquisition Parameters
Date_   20131230
Time    21.33
INSTRUM spect
PROBHD  5 mm PABBO BB/
PULPROG zg30
TD      65536
SOLVENT  CDCl3
NS       6
DS       2
SWH     10000.000 Hz
FIDRES  0.152588 Hz
AQ      3.2767999 sec
RG      30.72
DW      50.000 usec
DE      6.50 usec
TE      298.9 K
D1      1.0000000 sec
TDO      1

***** CHANNEL fi *****
SFO1   500.1330885 MHz
NUC1        1H
P1      13.00 usec
PIW1   13.0000000 W

P2 - Processing parameters
SI      65536
SF      500.1300000 MHz
WDW        EM
SSB      0
LB      0.30 Hz
GB      0
PC      1.00

```

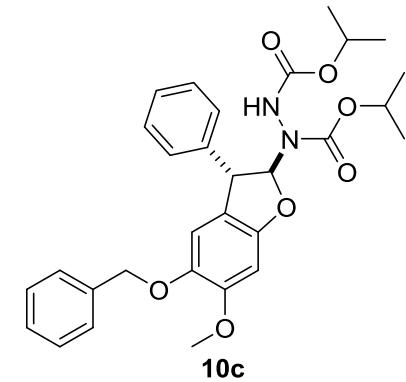
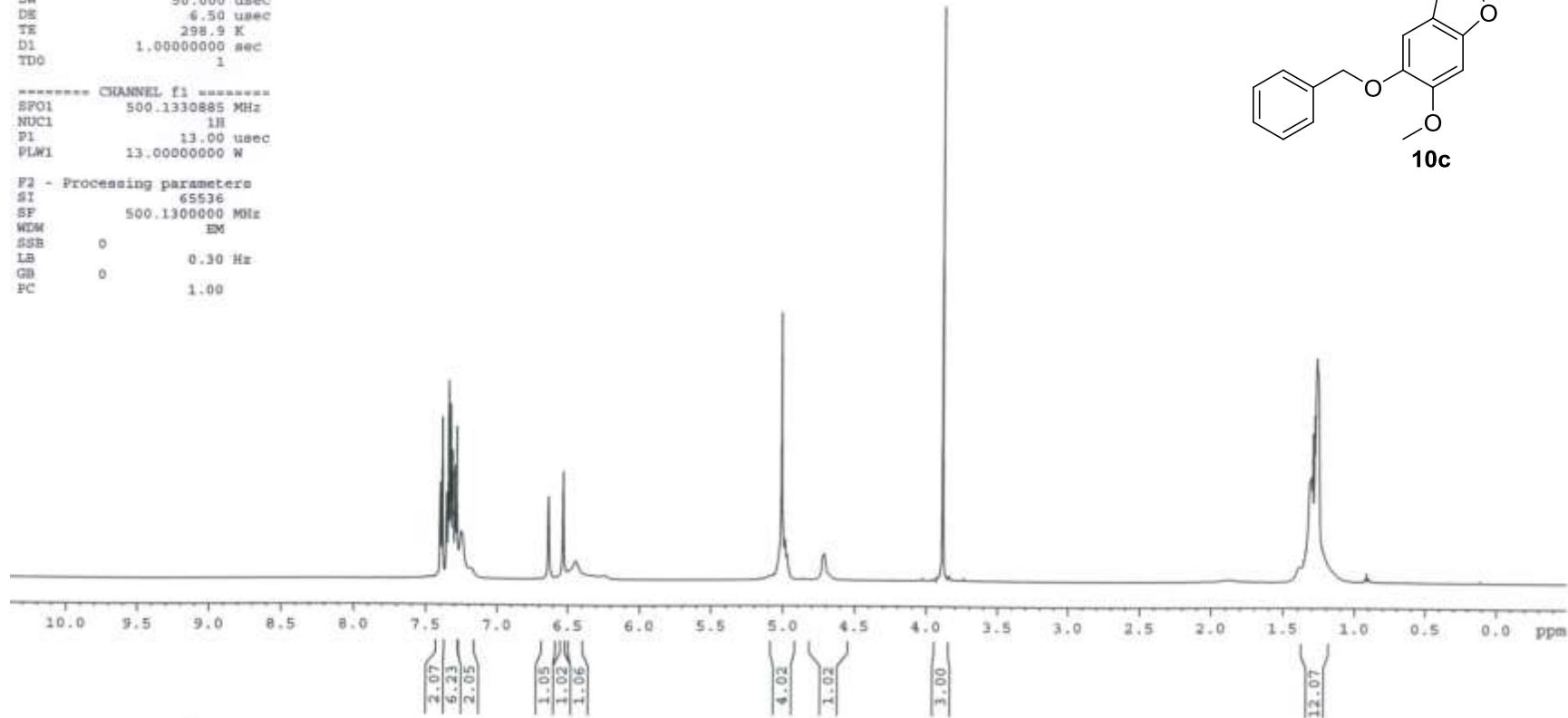


Figure S82. <sup>1</sup>H NMR Spectrum of **10c**

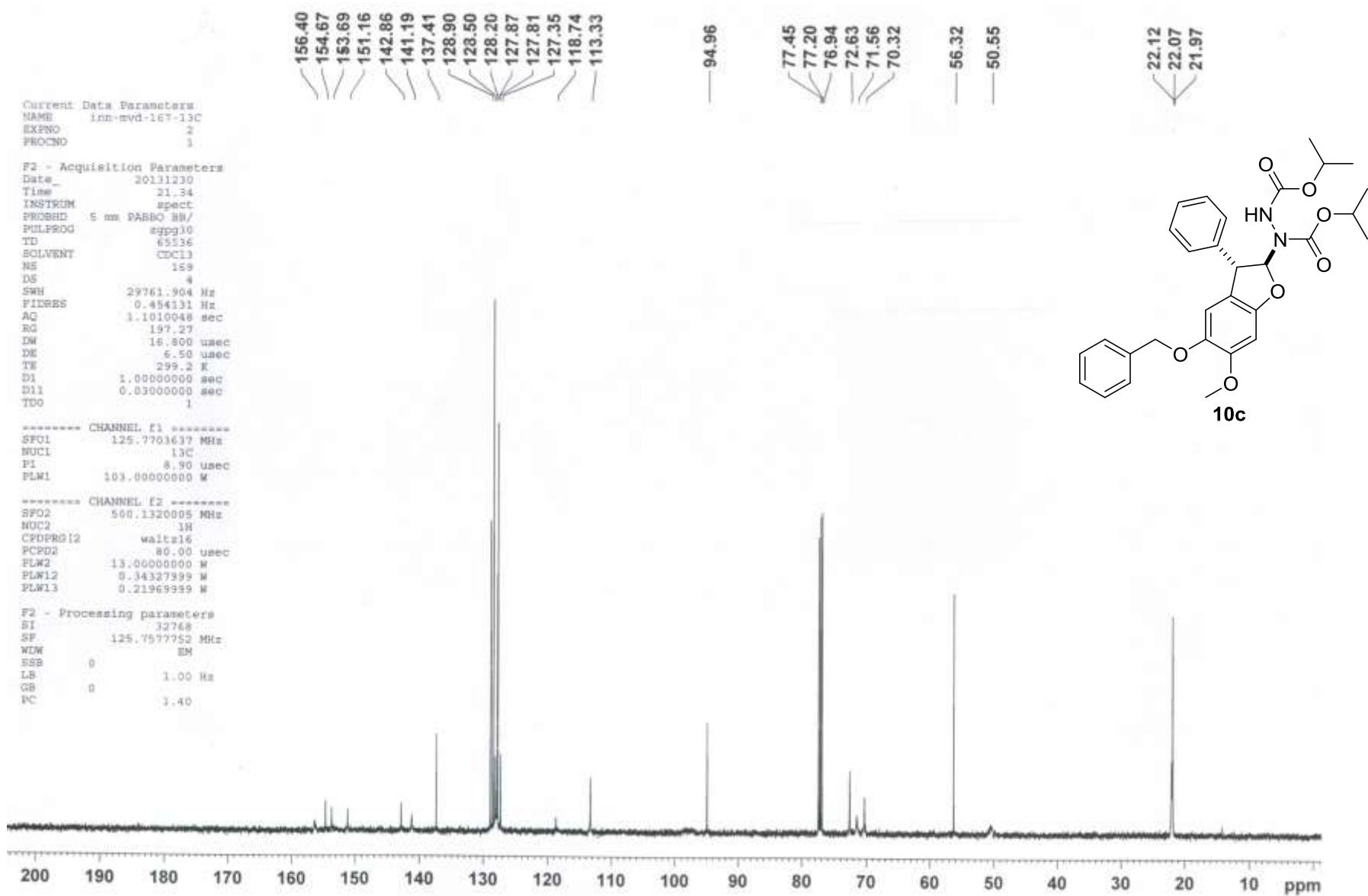


Figure S83. <sup>13</sup>C NMR Spectrum of **10c**